

VII. ORIENTED OVERGROWTHS OF ORGANIC CRYSTALS

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It has been known for a long time that numerous minerals are able to form oriented overgrowths. As early as 1903, Mugge³¹⁰ was able to report a large number of such examples. Many efforts were made to obtain such overgrowths under artificial conditions in an effort to determine the nature of the phenomenon involved. In 1825, Wakkernagel³¹¹ was able to orient lead nitrate on alum and alum on boracite. Later Frankenheim³¹² obtained oriented overgrowths of a number of inorganic salts on fresh cleavage surfaces of minerals.

The first systematic work on artificially prepared oriented overgrowths was carried out by Barker³¹³. Almost a hundred years later Royer³¹⁴ was to determine the physical basis for overgrowth by applying x-ray methods. Willems³¹⁵ extended these findings to systems of organic compounds.

The number of organic systems showing oriented overgrowths has been, until recently, very limited principally because of the difficulty of preparing suitable surfaces. It is possible, however, to obtain suitable surfaces by utilizing the crystal films formed by thermal methods on a microscope slide³¹⁶. The substance for study is melted and allowed to crystallize between a coverslip and slide. After cooling, the coverslip is removed leaving the crystal film adhering either to the slide or the coverslip. The surfaces thus obtained are suitable for testing as carrier surfaces.

The formation of oriented overgrowths has always been carried out in the past by crystallization from solution. This could not very well be avoided for inorganic substances; however, Sloar and Menzies³¹⁷ recommended that the crystallizing material be brought into contact with the carrier surface by means of vacuum sublimation. It was found in much of the previous work that the choice of solvent used was extremely important in determining whether overgrowths were observed.

Sublimation has been found to be much more suitable than solution experiments as a means of bringing the two components together. The sublimation is carried out by placing a few milligrams of the volatile substance to be adsorbed on a microscope slide. This is surrounded by a glass ring from two to four millimeters high on which is supported the crystalline carrier surface. The entire preparation is then placed on the hot bar at a temperature hot enough to cause sublimation of the volatile component. The experiment may also be carried out in the hot stage so that the condensation of the sublimate may be observed during heating. The ease of sublimation of many organic compounds may be found in the Identification Tables (see page 253).

In those cases where sublimation is not possible the component to be crystallized is dissolved in a solvent having a low dielectric constant. Very small droplets of this solution are then allowed to evaporate to dryness on the crystal surface.

A. Molecular Compounds as Contact Layers

Willems³¹⁸ has shown that the chemistry of formation of oriented overgrowths is related to the formation of molecular compounds. A number of systems have been investigated wherein molecular compounds had been reported in the literature and in nearly all cases oriented overgrowths were obtained. It is quite apparent that the crystal structures of the two components showing overgrowths must bear a definite relationship. Seifert³¹⁹ calls this a

one-dimensional structural analogy. Further x-ray work should be done on the structures of components of systems showing addition compound formation and oriented overgrowths.

It is necessary in these experiments to try several different temperatures before deciding that overgrowths do not exist since many systems will not show overgrowths if the sublimation rate is too high.

Figure 126 shows the oriented overgrowth of quinone on pyrogallol with the corresponding composition diagram. Figure 127 shows the oriented overgrowth of camphor on hydroquinone. Camphor also shows a number of irregular unoriented crystals between the oriented sublimate.

B. Mixed Crystals as Contact Layers

Neuhaus³²⁰ has observed oriented overgrowths as a result of mixed formation. In such systems the strongest tendency toward orientation will be shown in those systems where the two components show solid solutions over their entire composition range according to Types I, II and III of Roozeboom. This does not mean, however, that systems showing Type IV or V cannot show oriented overgrowths³²¹. It is even possible in certain cases for the formation of an oriented overgrowth to initiate growth of the unstable form.

Crystals of p-chloriodobenzene³¹⁶ grow on the carrier surface of p-dichlorobenzene I (Figure 128). p-Chloriodobenzene shows a continuous series of solid solutions with p-dichlorobenzene II according to Type I. According to Willems the contact layer between the carrier surface and the condensing component will be made up of the same phase represented in the composition diagram. In the above case the carrier surface A will change at the contact layer to mixed crystals with gradually increasing concentration of B until finally the crystals are made up of pure B.

When the order of the experiment is reversed so that p-chloriodobenzene becomes the carrier surface, then the oriented overgrowth of p-dichlorobenzene crystallizes in the enantiotropic modification II which is stable below 29°C and which is isomorphous with p-chloriodobenzene.

The oriented overgrowth of pyrene on phenanthrene³²² can be considered to be due to mixed crystal formation since in this system three mixed crystal phases appear; the mixed crystals in the middle concentration range correspond to a stabilized intermediate phase.

In certain cases it is impossible to ascribe the formation of oriented overgrowths to molecular compound or mixed crystal formation. Willems³²³, for example, has observed oriented overgrowths of hexaethylbenzene on chloranil. An oriented overgrowth may therefore be obtained even in those cases where the melt or solution of the two components shows no color attributed to the molecular compound. The crystal front of chloranil will often nucleate growth of hexaethylbenzene in a mixed fusion. The system phenanthrene and hexaethylbenzene found by Willems³²³ to show oriented overgrowths possesses neither molecular compound nor mixed crystal formation.

Although the literature had indicated that oriented overgrowths involving roughly spherical molecules (camphor and camphorlike substances) are very rare³²⁴ a number of new examples have been reported³²⁵. dl-Camphor, d-borneol, isoborneol and bornylchloride form oriented overgrowths with numerous phenols and nitro compounds (Figure 127). Oriented overgrowths of borneol on acid amides have also been found.

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VIII. RESEARCH RESULTS OF MICRO THERMAL ANALYSIS

By applying microscopical methods to thermal analysis, deeper insight into the nature of two-component composition diagrams can be obtained, making it possible to clarify contradictions or controversial points and to correct or complete data obtained by other means.

Quantitative thermal analysis is most productive when contact methods^{1,2} (mixed fusions) are utilized and polymorphism is considered. In many cases, a more complete understanding will thus be achieved concerning the possible solid phases and their relation to one another. Thus, we can see in a mixed fusion that cholesterol, for example, forms a molecular compound⁴ sarcosine when warmed with anhydride. Mixed crystal formation had previously been reported³.

In other cases, for example, when unstable forms or mixed crystals appear in the contact zone, it is only through comparing analyses of mixed fusion preparations of solidified mixtures in various concentrations, or of mixtures of pure components, that the resulting phase diagram can be unequivocally determined (see page 120).

Fusion methods are especially useful with mixed crystal formations and particularly in the investigation of unstable mixed crystal series. It is most advantageous if, during spontaneous solidification of the test mixture, not only the stable modification is formed, but also the unstable form. It is only possible working thus on a micro scale to determine composition diagrams involving unstable polymorphic forms⁵.

A. Two-Component Systems with Mixed Crystal Formation

Dibenzyl, stilbene, azobenzene and diphenyl acetylene are reported in the literature^{6,7} to be completely miscible. It is a question here of isomorphous substitution of the groups $-\text{CH}_2-\text{CH}_2-$, $-\text{CH}=\text{CH}-$, $-\text{N}=\text{N}-$, $-\text{C}\equiv\text{C}-$ ⁸. Thermal analysis shows, however⁹, that dibenzyl is not completely miscible with the stable forms of the three substances with which, until now, it has always been compared crystallographically: instead it is miscible with an unstable, previously unknown, modification II (m.p. 50°C). The two-component systems involving stilbene, azobenzene and diphenyl acetylene form continuous melting curves indicating complete miscibility over the entire composition range, but, contrary to past literature, give a corresponding melting diagram with dibenzyl of two curves due to isodimorphism. The dibenzyl:stilbene system is not a Roozeboom type I system; instead it is a type IV system (Figures 129a and b); the figure on the right is diagramed according to the microscopical results with k actually lying at 3 percent stilbene. The mixed fusion preparation of Plate X, Figure 70, clearly points out two phases (Appendix); in Plate XI, Figure 71, the reaction at the peritectic point is represented.

The dibenzyl:azobenzene system is, instead of Roozeboom type III (Figure 130a), a combination of types III and IV (Figure 130b). The stable form of stilbene, as well as azobenzene and diphenyl acetylene shows solid solutions with an unstable form II of benzalaniline ($-\text{CH}=\text{N}-$); indeed, azobenzene and/or stilbene or diphenyl acetylene is completely miscible (Roozeboom type I) with modification II of benzalaniline which melts at 28°C¹⁰. The melting diagram resulting from isodimorphism corresponds to type V (Figure 131), in good agreement with the literature⁷. In the dibenzyl:benzalaniline system (Figure 132), the two unstable modifications, dibenzyl II and benzalaniline II, crystallize in a mixed preparation with 35 to 65 percent dibenzyl as a stabilized intermediate phase¹¹, with which benzalaniline I forms a eutectic E and with

which dibenzyl I forms the transition point K.

The mixed crystal series of benzalaniline II and dibenzyl II, which corresponds to the intermediate phase, is continuous¹⁰.

The explanation for the deviation in behavior of the unstable form of dibenzyl lies in its nonplanar molecular structure^{12,13}, while stilbene, azobenzene and diphenyl acetylene have planar molecular structures (Figures 33 and 34, page 226). In the unstable dibenzyl modification which arises spontaneously on strong supercooling, the challenge of a planar structure is evidently met since this form shows continuous mixed crystal formation with stilbene, azobenzene and diphenyl acetylene (see also Figures 133a-d).

In addition to the five substances mentioned, further investigations¹⁴ have been made of benzylaniline and hydrazobenzene (representing the -NH-CH₂- and -NH-NH- bridges). Complete miscibility was reported for the systems of hydrazobenzene with dibenzyl, stilbene, diphenyl acetylene and azobenzene. Our microthermal analytical investigation¹⁴ showed that a simple eutectic occurs in all four systems. Similarly, the systems of benzylaniline with stilbene, diphenyl acetylene, azobenzene and benzalaniline which, according to the literature⁷, should show mixed crystal formation (type V), exhibit instead a simple eutectic. However, in the hydrazobenzene:benzylaniline system mixed crystal formation of type IV¹⁴ does occur (Figure 134).

According to the results of microthermal analysis, the seven substances containing -CH₂-CH₂-, -CH=CH-, -N=N-, -C≡C-, -NH-CH₂-, -CH=N- and -NH-NH- can be divided into three groups according to their crystallographic and chemical relationships as follows:

1. -CH=CH-, -C≡C-, -N=N-. The substances of this group, distinguished by their symmetrical structure and by the double or triple bond in the middle showed the greatest degree of crystallographic and chemical similarity that results in continuous mixed crystal series (Figures 133a,b).

2. -CH₂-CH₂- and -CH=N-. These two show isodimorphism with each other as do the substances in the first group. Their unstable forms (II) show complete miscibility (Figure 132). Furthermore, the unstable modifications in group II form continuous mixed crystal series with the stable modifications (I) of group I. Their stable forms possess, however, as is evidenced in one case by free rotation, in the other by the asymmetry around the middle bond, structures which can dissolve only relatively small amounts of the compounds in group I (Figures 129b, 130b, 133c, 131 and 133d).

3. -NH-CH₂-, -NH-NH-. Representative substances of these two types, partly because of their asymmetry and partly because of the single middle bond, are unable to form mixed crystals with representatives of groups I and II, forming instead simple eutectics. However, they do show limited solid solubility relationships with one another (type IV: Figure 134).

Benzalaniline (-NH-CH₂-) shows, as one definite crystallographic and chemical relationship, a strong stabilizing effect on dibenzyl II.

The behavior of several systems of naphthalene groups (naphthalene, α- and β-naphthol, α- and β-naphthylamine) was by Johnson¹⁵, and, later, by Neuhaus^{16,17}, termed "anomalous" mixed crystal formation. Through x-ray investigation of naphthalene, β-naphthol and β-naphthylamine, Neuhaus¹⁶ came to the conclusion that the crystal lattices of these structures are not "isostructural" and therefore the truth of the literature report¹⁷ of complete miscibility in known melting diagrams is to be doubted. Microscopical investigation¹⁸ clarified the understanding of the naphthalene:β-naphthol sys-

tem through the discovery of modification II of β -naphthol, on which rested the literature report¹⁹ of completely miscible mixed crystal formation. The melting diagram in the literature is still correct because of the similarity of the melting points of the two modifications of β -naphthol²⁰. β -naphthol represents the boundary between monotropic and enantiotropic dimorphism, since the transition point coincides with the melting point of both modifications. Neuhaus' investigation of β -naphthol¹⁶ confirmed the presence of form II which is stable at room temperature and only slightly miscible with naphthalene. Since the structure of β -naphthol (form II) shows the existence of double molecules it is assumed that two molecules of naphthalene are replaced by a double molecule of β -naphthol^{21,22}.

Contrary to the literature (Rudolfi and Rheinboldt²⁰), naphthalene and β -naphthylamine are not completely miscible; instead of type III Roozeboom, type IV occurs.

β -naphthol shows with β -naphthylamine a maximum melting solution which is interpreted as an equimolecular compound; this compound is completely miscible with β -naphthol and forms a eutectic with β -naphthylamine (Figure 135).

It should be noted that the prominent branch of the curve of modification I of β -naphthol which, in Figure 135, goes left of β -naphthol over M to E and which yields the form unstable at room temperature is responsible for the stabilization of this modification through the mixed crystal state²⁰. Kremann and Strohschneider²³, as well as Tittus²⁴, report a 2:1 molecular compound (Figure 136), while Hrynakowski²⁵ reports a complete mixed crystal formation of type III (Figure 137).

In further diagrams on page 241, our results are compared with Kremann's²³. The differences can usually be traced to the phenomena of supercooling. Instead of a single molecular compound (Figure 138) between α -naphthol and α -naphthylamine, we found two compounds (Figure 139) and a slight miscibility at the upper end of the α -naphthylamine curve. According to Kremann²³, β -naphthylamine: α -naphthol forms a simple eutectic at 47° (Figure 140). In reality, the eutectic lies at 56°; however, an unstable molecular compound is formed which melts incongruently at 50°²⁰ (Figure 141). According to Kremann²³, β -naphthol: α -naphthylamine forms a 2:3 compound (Figure 142); the actual compound is, however, equimolecular (Figure 143). It is noteworthy that the β -naphthol portion of the melting curve is not formed by modification I, as in the system β -naphthol:naphthalene or β -naphthylamine. Instead it is produced from form II which is unstable at room temperature; the stable form I is absent because of mixed crystal formation.

Concerning the substituted *s*-trinitrobenzenes, the comparative x-ray structural analyses of E. Hertel and G. Romer²⁶ show that with *s*-trinitrobenzenes the substituents Cl, Br, I, OH, NH₂, NO₂, CH₃, OCH₃ and OCH₂ and OC₂H₅ (according to Grimm's rule⁸) do not show the least analogy to the crystal structure of *s*-trinitrobenzene.

Our own investigations, using hot stage microscopy, show isodimorphism in the binary composition diagrams in this series. In these mixed crystal systems, however, the preferred structural type is not, as in the substituted naphthalenes, the stable form; instead, *s*-trinitrobenzene has four modifications in addition to the stable form: I melting at 123.5°; II at 110°, III at 109°, IV at 106° and V at 88°²⁷. In the *s*-trinitrobenzene:picric acid diagram, a eutectic is formed by two crossing isodimorphous mixed crystal series; and indeed stable picric acid I (122°) is completely miscible with *s*-trinitrobenzene III (109°), while stable *s*-trinitrobenzene I (123.5°) yields a continuous mixed crystal series with picric acid III (75°) (Figure 144) (see also page 241). It is notable that modification II of pure *s*-trinitrobenzene (110°) is relatively stable, actually more stable than modifica-

tion III (109°), but in a mixture with picric acid modification II immediately loses its relative stability in favor of modification III. This is especially emphasized because it illustrates the stabilization of unstable forms by minor impurities.

In the picryl chloride: α -trinitrobenzene system, a lattice isostructural with picric acid I occurs as a stabilized intermediate phase (Figure 145).

In the picryl chloride:picric acid system²⁷, the latter likewise controls a great part of the diagram, but is overlapped in the left portion by a stabilized intermediate phase (Figure 146).

In the picryl chloride: α -trinitrotoluene system²⁸, there is extensive miscibility in the stable trinitrotoluene crystal lattice and, to a similar degree, in the lattice of picryl chloride II (61°) (Figure 147). In this system, a molecular compound was found by Titus²⁴, which was identified by Hertel and Römer²⁶ (x-ray diffraction) as a structural analog of trinitrotoluene; in spite of the analogous crystal lattice, no mixed crystal structure should exist according to the above-mentioned authors. The molecular compound was designated as a "new type". In reality, no molecular compound exists; the crystals reported and investigated by Hertel and Römer represent a mixed phase and are, for this reason, structurally analogous with trinitrotoluene (Figure 147)²⁸.

In the trinitrobenzene series, the complete miscibility apparent upon substitution of Cl, Br and CH₃ cannot be explained without further work. The mixed crystal series in the picryl chloride: α -trinitrotoluene system, formed in a trinitrotoluene lattice type, probably has nearly 80% picryl chloride content; the corresponding modification, however, is not known for pure picryl chloride.

In the bromine compound, the stable α -trinitrotoluene lattice type is not detected; on the contrary, it is apparent that the forms described by Hertel and Römer²⁸, as a result of x-ray diffraction to be monoclinic and hexagonal, are present in picryl chloride as two unstable modifications (IV, V)²⁹. The melting diagram corresponds to Roozeboom type V, since the two parallel melting curves of the mixed crystal series of picryl bromide I and II are intersected by the incomplete mixed crystal series of picryl chloride I at the eutectic point (Figure 148).

Brandstätter has found³⁰ another unstable modification of picric acid (III) melting at 101° in mixed fusions with styphnic acid (Figure 149).

Relationships with the substituents of H, OH, Cl and CH₃ on *m*-dinitrobenzene are also evident³¹. Another fact to be noted here is the appearance of stabilized intermediate phases in the systems of dinitrochlorobenzene with, for example, *m*-dinitrobenzene, dinitrotoluene and dinitroaniline (Figures 150, 151 and 152).

According to Brandstätter³³, 1-chloro-2,4-dinitrobenzene and 1-bromo-2,4-dinitrobenzene represent an especially good case of parallel isodimorphism (page 100)³². Four overlapping complete mixed crystal series of type I have been established; a fifth mixed crystal series intersects the two most unstable series; this series, however, has not been completely characterized (Figure 153).

As a test of isomorphism of chloro- and bromo-compounds, the following systems were further analyzed by Brandstätter:³⁴ *p*-dichlorobenzene:*p*-dibromobenzene, *p*-chlorobenzoic acid methyl ester:*p*-bromobenzoic acid methyl ester, and *p*-chloroacetanilide:*p*-bromoacetanilide. The first of these systems shows complete miscibility at room temperature. Since, however, the transition for *p*-dichlorobenzene is enantiotropic, the resulting melting diagram is not of type I; it is, instead, type V (Figure 154). The melting point diagram of the substituted benzoic acid methyl esters shows type IV on

the basis of isodimorphism (Figure 155). The acetanilides are homogeneously miscible, and type I; thus two mixed crystal series are established because of parallel isodimorphism (compare Figure 74) (Figure 156).

The *p*-bromiodobenzene:*p*-diiodobenzene system, described in the literature³⁵ as type Ia (solidification curve with an inflection point), was corrected by Brandstätter³⁶ to type IV on the basis of isodimorphism (Figure 157).

In the pentachlorobenzene group, Brandstätter³⁷ found complete miscibility between most OH-, NH-, CH- and Cl-substituted compounds. The only exception is the hexachlorobenzene:pentachloroaniline system, in which a miscibility gap occurs in the middle of the concentration range. Since the two components are completely miscible with the other members like pentachlorophenol and pentachlorotoluene, respectively, the miscibility gap can only be due to limited miscibility caused by exceeding the tolerance limit of each in the lattice of the other. This is the first such example shown by organic substances.

In the *trans*-cinnamic:hydrocinnamic acid system, there is complete miscibility between the two unstable forms: cinnamic acid II (133°) and hydrocinnamic acid II (35°). This mixed crystal series is the stable phase in the concentration interval between 18 and 78 percent hydrocinnamic acid³⁸ (Figure 158). The crystal structures of both modifications of cinnamic acid³⁹, as well as that of stable hydrocinnamic acid⁴⁰, are known; together, they show no sort of analogy, that is, the two modifications of *trans*-cinnamic acid are as different from one another as they are from the stable hydrocinnamic acid lattice. Bruni and Gorni^{42,43}, who found a continuously rising melting point curve, came, by reason of Boeris⁴³ discovery of distinct crystal forms for both components, to the conclusion that it was very likely a case of isodimorphism. The full explanation of the system, which consisted of the discovery of relationships of the unstable forms of both substances and of their stabilization in mixtures as an intermediate phase, was first arrived at through microthermal analysis³⁸.

Brandstätter⁴⁴ also found stabilized intermediate phases in the two-component systems of phenanthrene:fluorine (Figure 159), phenanthrene:pyrene (Figure 160) and resorcinol:pyrogallol. In none of the three cases, however, was it possible to follow the curved portion of the intermediate phase up to one of the two components. Their mixed crystal natures, however, are well-established.

The two pairs of substances, dianisyl oxide:dianisyl sulfide and diphenylene oxide:diphenylene sulfide, should, according to the literature, be distinctly different⁴⁵. Micro thermal analysis, however, shows that they have analogous mixed crystal formation and, indeed, show isodimorphism⁴⁶ (Figures 79 and 80, page 233).

In one series of 2,4-dinitrophenylhydrazones with ketones and aldehydes, Brandstätter⁴⁷ found a strong tendency to mixed crystal formation. This is significant because 2,4-dinitrophenylhydrazone recently has been used as a test reagent for ketones and aldehydes. Ignorance of its behavior can lead to errors in the capillary method used in mixed melting point determinations; thus, methylbutylketone-2,4-dinitrophenylhydrazone forms a complete mixed crystal series with methylpropylketone-2,4-dinitrophenylhydrazone (type I). Methylisobutylketone-2,4-dinitrophenylhydrazone, as well as methylpropylketone-2,4-dinitrophenylhydrazone with ethylpropylketone-2,4-dinitrophenylhydrazone, always forms such series with type III compounds (page 98). Methylbutylketone-2,4-dinitrophenylhydrazone:methylethylketone-2,4-dinitrophenylhydrazone is a rare case in which the melting point curve for an isomorphous pair has both a maximum and a minimum (Figure 161). The systems of

butyraldehyde-2,4-dinitrophenylhydrazon:propionaldehyde-2,4-dinitrophenylhydrazon and propionaldehyde-2,4-dinitrophenylhydrazon:isobutyraldehyde-2,4-dinitrophenylhydrazon also behave isodimorphously.

Barbituric acid derivatives likewise behave isomorphously or isodimorphously. Brandstätter⁴⁸ found numerous examples of complete mixed crystal series of type I (for example, butylpropylbarbituric acid:ethylbutylbarbituric acid, diallylbarbituric acid:ethylallylbarbituric acid) and, oftener, such systems of type III with a minimum (for example, allylbutylbarbituric acid:ethylbutylbarbituric acid or butylpropylbarbituric acid:allylisopropylbarbituric acid). Isodimorphism has been established with allyldipropylbarbituric acid:allylisobutylbarbituric acid, ethylpropylbarbituric acid:ethylbutylbarbituric acid, allylisobutylbarbituric acid:ethylpropylbarbituric acid and a few other systems. The system allylisopropylbarbituric acid:ethylpropylbarbituric acid, in which a stabilized intermediate phase exists, is illustrated in Figure 85.

Because of frequent mixed crystal formation, identification with the help of mixed melting points is not always reliable since the mixtures show no, or only a slight, melting point depression. Eutectic temperatures, which in many cases definitely prove the identity of the test substance, are likewise unsuitable for barbituric acid derivatives; however, Brandstätter⁴⁹ proved that combining the micro melting point determination with the Koflers glass powder method^{46,50} provided an extraordinarily simple and certain method for investigating the barbituric acid derivatives. Also, simplified quantitative analysis of two-component solid solutions with the help of refractive index of the melt is possible in many cases.

B. Two-Component Systems with a Simple Eutectic or with Molecular Compounds

Microthermal analysis revealed complications caused by polymorphism in a few systems. With the α -, γ - and δ - isomers of the series of hexachlorocyclohexanes, the three possible systems as well as the behavior of polymorphic modifications of γ - and δ -hexachlorocyclohexane were studied⁵¹. Figure 162 shows γ -hexachlorocyclohexane: α -hexachlorocyclohexane. γ -hexachlorocyclohexane has polymorphs; modifications I and II are enantiotropic, their melting points lying extremely close together, m.p. γ II, 113.5°; m.p. γ I is just perceptibly higher so that it must be taken as 113.6°. The transition point at 112.5° is perceptible only in extremely pure products because of its closeness to the melting point. In Figure 163, δ -hexachlorocyclohexane: α -hexachlorocyclohexane is represented. Also, δ -hexachlorocyclohexane is enantiotropically dimorphous, the difference between the melting points being 1° (m.p. I, 139°, m.p. II, 138°) with the transition point at 132°. Heating from room temperature decreases the transition rate of the unstable form I into II. Since spontaneous formation of δ I from the melt is favored and δ I is transformed into II only very sluggishly, especially in mixtures, the lower melting eutectic at 110° is usually observed instead of 112°; in mixtures between 20 and 35 percent α -hexachlorocyclohexane, one frequently finds the final melting point on the lower curve. Because of the small differences in these temperatures, the polymorphic behavior can hardly have practical significance.

In γ -hexachlorocyclohexane: δ -hexachlorocyclohexane (Figure 164), it is difficult to determine the melting point curves because of the behavior of the unstable modifications and their sluggish transformation, which, moreover, is even slower in mixtures. Eutectic temperatures differing up to 9° can be measured in previously completely melted samples, depending on the

combination of polymorphs involved. Also, determination of the primary crystallization point on cooling of melts is difficult for middle concentrations, since sometimes points on the upper curves are measured and sometimes on the lower curves; with a microscope these errors can be avoided since the polymorphic form can be recognized.

Through investigation of veronal (diethylbarbituric acid) in a two-component system, the polymorphic behavior^{52,53} was completely clarified. Of the four modifications, I, 190°; II, 183°; III, 181°; IV, 176°; III and IV behaved enantiotropically with a transition at 135°⁵⁴.

In the systems of veronal with methylacetanilide (Figure 165), dinitrospiperazine (Figure 166) and dimethylpyrone (Figure 167), the melting curves of the stable forms as well as part of the melting curves of the unstable forms could be determined. Enantiotropism of veronal III and IV in the three systems mentioned is evident in the lower value of the eutectic for modification III than for IV. The transition point (135°) is directly observable in a 30 percent methyl acetanilide mixture with veronal. In 2,6-dimethylpyrone: veronal (Figure 167), an incongruently melting equimolecular compound occurs which forms a eutectic with dimethylpyrone at 111° (28 percent veronal) and a transition point at 126° (52 percent veronal).

Veronal behavior in the detection of the eutectic temperature is remarkable: When veronal is allowed to sublime on a coverslip, several modifications arise. If dimethylpyrone is then sublimed onto a slide and if the coverslip is placed on the slide so that the two sublimates touch, a variety of eutectic temperatures can be observed. Phase changes are observed at 88°, 90°, 111° and 126°. Formulation of a melting diagram, with the help of microthermal analysis, clarifies this behavior: 88° and 90° represent the eutectics of veronal IV and III with dimethylpyrone. For modifications I and II of veronal, however, direct determination with dimethylpyrone is unsuccessful since a molecular compound forms whenever the temperature rises above 90°, and 126° is the transition point of the incongruently melting compound⁵⁴.

Investigation of nicotinamide in two-component systems led to the discovery of numerous molecular compounds⁵⁵; thus nicotinamide forms one molecular compound with azelaic acid, glutaric acid, stearic acid and dinitrophenol, two compounds with suberic acid, decamethylene dicarboxylic acid (page 95), adipic acid, sebacic acid and hexadecane dicarboxylic acid, and three compounds with catechol (Figure 168). Polymorphism was detected in some of the molecular compounds.

A series of organic molecular compounds was tested for polymorphism⁵⁶. *s*-Trinitrobenzene forms with fluorene a molecular compound with four modifications, showing a noteworthy analogy to the four above-mentioned modifications of trinitrobenzene (Figure 169). The molecular compound of picric acid with fluorene has two monotropic modifications. The compounds of *s*-trinitrobenzene with naphthalene, phenanthrene (Figure 170) and α -naphthylamine have two enantiotropic forms. The compound of picric acid and phenanthrene is also enantiotropically dimorphous (Figure 171).

With anthracene, picric acid produces an enantiotropic, incongruently melting compound^{56,57}. With α -naphthylamine, picric acid gives two distinct molecular compounds. The equimolecular compound is yellow; the 1:2 compound is orange (Figure 172). Pyridine picrate is enantiotropically dimorphous.

Quehenberger⁵⁸ has recently investigated the formation of many molecular compounds and their polymorphs. He has studied organic acid amides in two-component systems with carboxylic acids and phenols. Nicotinamide with *m*-nitrobenzoic acid shows, for example, three molecular compounds (Figure 173);

with cinnamic acid, a dimorphous molecular compound (Figure 174), and with *p*-cresotic acid, two compounds of which one (1:3) is dimorphous. Furthermore, benzidine forms a dimorphous compound with cinnamic acid and, with *p*-nitrobenzoic acid methyl ester, two incongruently melting compounds of which the equimolecular compound is found to be dimorphous. Melting curves of unstable modifications can usually be determined experimentally with suitable mixtures. A series of further molecular compounds is recorded in Tables 5 and 6.

TABLE 5
a) Acid amides

Substance pair	Melting point of the molecular compound	Substance pair	Melting point of the molecular compound
<u>Benzamide:</u>		<u>Propionamide:</u>	
<i>o</i> -cresotinic acid	120	<i>m</i> -nitrobenzoic acid	74
<i>p</i> -cresotinic acid	116	<i>p</i> -nitrophenol	58
<i>m</i> -nitrobenzoic acid	137	<u>Salicylamide:</u>	
picric acid	111	<i>o</i> -cresotinic acid	136
resorcinol	88	<i>p</i> -cresotinic acid	117
salicylic acid	118	<i>m</i> -nitrobenzoic acid	135
<u>Nicotinamide:</u>		picric acid	106
<i>o</i> -cresotinic acid	144	<u><i>n</i>-Butyramide:</u>	
hexadecane		catechol	35
dicarbonic acid	129, 130.5	<i>m</i> -nitrobenzoic acid	84
<i>p</i> -nitrophenol	110, 115		
picric acid	129.5, 189		
salicylic acid	136.5		

Thanks to the cold stage⁶², the behavior of organic two-component systems with constituents which are liquid at room temperature was also investigated. With catechol, *p*-chloroacetophenone forms a dimorphous molecular compound with melting points of 26° and 14° (Figure 175).

C. Melting Diagrams with Inflection Points or Immiscible Melt Phases

In one series of organic substance pairs, melting curves exhibited an inflection instead of a continuously rising curve^{63,64}.

The existence of inflections can be traced to many different causes. One frequently given is the proximity of a miscibility gap to the liquid phase (ML) so that after supercooling of the melt, the temperature is lower than the critical solution temperature (CST) and separation into two liquid phases results. Inflections often form if, unlike the normal ratio with liquid components, the ratio of the molar heat of fusion Q to the absolute melting temperature T_0 , i.e., Q/T_0 , is under about 3.5. Other reasons are a negative heat of mixing or compound formation in the melt^{63,64}.

In several organic two-component systems, the inflection shows that the

TABLE 6
b) Amines

Substance pair	Melting point of the molecular compound	Substance pair	Melting point of the molecular compound
<u>Benzidine:</u>		<u>o-Phenylenediamine:</u>	
aspirin	252	catechol	84.5
azelaic acid	114, 114.5	o-cresotinic acid	128
benzoic acid	103	p-cresotinic acid	112, 117, 123
catechol	148	p-nitrophenol	88
o-cresotinic acid	138, 151	resorcinol	50
hexadecane		salicylic acid	116
dicarboxylic acid	124	cinnamic acid	95.5
hippuric acid	146		
suberic acid	124	<u>p-Phenylenediamine:</u>	
lactophenin	115	azelaic acid	112
α -naphthol	98	catechol	108
β -naphthol	132.5, 171	o-cresotinic acid	142, 148
m-nitrobenzoic acid	158.5, 160.5	p-cresotinic acid	172
o-nitrobenzoic acid	189	hexadecane	
m-nitrobenzaldehyde	129	dicarboxylic acid	130
o-nitrobenzaldehyde	214	m-nitrobenzoic acid	136, 158.5, 163
p-nitrobenzoic acid		p-nitrophenol	122.5, 139
methyl ester	89, 102	resorcinol	116
p-nitrophenol	151	salicylic acid	137
phenylacetic acid	126	cinnamic acid	125, 128
pyrogallol	143		
m-oxybenzaldehyde	270	<u>Thiosinamine:</u>	
m-oxybenzoic acid	151	picric acid	162
resorcinol	143		
salicylic acid	161		
tetradecane			
dicarboxylic acid	122		
s-trinitrobenzene	131		
x-trinitrotoluene	99		
o-vanillin	212		
cinnamic acid	113, 109		

proximity of a miscibility gap to the liquid phase could be experimentally determined⁶⁵.

To test a substance pair with an inflection curve for a liquid phase lying under the melting curve, a mixed fusion is melted completely and transferred immediately to the hot stage, which is heated to just a few degrees below the expected CST. Excessive cooling of the preparation should be avoided to retard spontaneous crystallization. If a liquid phase exists, two layers separate after a short time; upon further warming, up to the CST, the two layers again mix completely. Crystal films of uniform composition are employed to determine particular points on the melting diagrams.

Using the microscope, one can also observe the behavior of unstable mod-

ifications and investigate their melting curves relative to their behavior with immiscible melts.

In all of the systems with inflection points described below⁶⁵, turbidity occurs on rapid cooling of the mixtures before spontaneous crystallization begins; this is caused by separation into two liquid phases. On reheating the microscopical preparation, the separated droplets redissolve at one of the concentration-dependent temperatures corresponding to the limits of the particular liquid phase. Naturally these temperatures are lower than the primary recrystallization temperatures of the corresponding mixtures.

In naphthalene:resorcinol and naphthalene:hydroquinone, Kremann and Janetzky⁶³ discovered inflection curves which they explained through the hypothesis of a compound in the melt. According to the rule of Kremann and Rodini⁶⁶, meta- and para- disubstitution products, rather than ortho-products, should favor molecular compounds; thus naphthalene should form molecular compounds with resorcinol or hydroquinone, but not with catechol. Upon reexamination, immiscible melts were easily detected in a supercooled mixed fusion preparation of naphthalene:resorcinol in which the critical solution temperature (CST) lay at 93°, only 4° below the point of inflection. In Figure 176, in which the melting curve was drawn according to the data of Kremann and Janetzky⁶³, the extent of the liquid phase (ML) is marked by a broken line. In the naphthalene:hydroquinone system, however, no liquid phase could be detected in the supercooled state, since hydroquinone is extremely difficult to supercool even in mixtures. Also for naphthalene:catechol, during supercooling at room temperature, no separation of the melt into two phases occurs.

In two-component mixtures with acetamide, the liquid phase and the behavior of the unstable modifications can be observed. Acetamide I crystallizes spontaneously from the melt, as does the unstable modification which melts at 70°. The latter should also remain unchanged upon warming, according to the rules, up to its melting point. It is therefore possible to determine the behavior of both the stable form I (80°) and the unstable form II (70°). Since acetamide is hygroscopic, the powdered mixture must be kept in a desiccator.

An especially good example of inflection curves, because of its adjacent miscibility gap, is the acetamide: α -trinitrotoluene system (Figure 177). Here the two inflection curves can be determined one above the other. In the stable acetamide system an inflection curve is formed by the branch (I) emanating in the direction E_I of Figure 177; in the unstable acetamide system, the branch of the trinitrotoluene (TNT) curve to the right of E_{II} of Figure 177 shows an inflection area. The CST reaches to 69°, slightly below the inflection portion of the TNT curve. The two eutectics are, as it were, pushed aside by the immiscible phases so that they attain completely opposite concentrations; E_{II} = 65°, 24% TNT and E_I = 73°, 82% TNT⁶⁵.

The acetamide:p-nitrophenetol system is intermediate between a stable inflection curve and a stable immiscible melt system, inasmuch as the stable modification of acetamide forms a diagram with an inflection curve with p-nitrophenetol, but in the system of unstable acetamide II with p-nitrophenetol, immiscible melts appear (Figure 178).

Figure 179 shows the acetamide:benzil system, in which the immiscible melts curve is cut by the curved branch of benzil. The liquid phase extends from 28 to 77% benzil at 88°; the critical solution temperature is 94°. The latter can be demonstrated very easily in a mixed fusion if it is warmed carefully to above 88°. The meniscus appearing at 88° disappears at 94°. As long as the diffusion has not reached a broader zone of mixing, two-

fold melting can be discerned at first by the differing refractive indices (see also Plate IX, Figure 61 [Appendix]).

Figure 180 shows a much wider temperature range for the immiscible melts in the system acetamide: β -naphthol methyl ether (nerolin). Here the eutectics of the two acetamide modifications lie on opposite sides, that is, E_{II} at 66°, 14% nerolin (NE), E_I at 71° and 97% NE. The immiscible melt phases are apparent for modification II above 70°; for modification I above 76°; CST = 130°.

Acetamide shows wide immiscibility gaps, with CST above 200°, with many aromatic substances like acenaphthene, fluorene, naphthalene, azobenzene, diformyl, dibenzyl, stilbene, diphenylacetylene, betol, benzalazine and others. In these cases, the CST can no longer be determined since, over 200°, partial decomposition frequently sets in so that the range of immiscibility again increases toward higher temperatures.

In the acetamide:acenaphthene system, the liquid phase reaches from 12 to 97.5% acenaphthene. E_I = 79°, 5.5% acenaphthene; E_{II} = 69°, 4% acenaphthene.

In these cases it is advantageous, in studying the liquid phase, to use L. Kofler's method which depends on determining mixture composition by refractive index of the melt⁶⁷ (page 239, Figure 124).

Mixed fusions^{46,68} were most recently applied by Fredga and Matell^{69,70} in determining the configuration of optically active substances. Because of the simplicity, as well as the small sample needed, they found the method very useful. The principle of investigation depends on one given by Timmermans⁷¹, and especially on a method worked out by Fredga⁷² which is based on the following properties of optical isomers.

It is known that equimolecular quantities of optical isomers form either a molecular compound, i.e., racemic compound; mixed crystals, i.e., a racemic solution; or a eutectic, i.e., a racemic mixture. Racemic compound formation is so common that it must be regarded as normal. A genuine compound can be recognized in the melting diagram, like every other molecular compound, through a more or less clearly marked maximum at 50% in relation to which the two eutectic points are symmetrical (Figure 181). If a small structural change is made in one of the two components, the new component pair, in many cases, also form a molecular compound⁷³. The resulting melting diagram again shows a maximum at 50 mole percent, but the two eutectics are no longer symmetrical.

The configuration of an optically active substance can be determined microthermally as follows: A pair of isomers, d- and l-B, are chosen in which the configuration of each is known and the molecular structure differs only very slightly from A. With the help of melting diagrams, the systems A:d-B and A:l-B are determined. If the A:d-B system shows a molecular compound in a 1:1 ratio, whereas the A:l-B system shows only a simple eutectic, the A has an opposite configuration to d-B; therefore it is l-A.

Since the conditions for addition compound formation are still not completely understood and since even mixed crystal formation can be questioned, decisions were possible with the classical methods only after complete melting diagrams had been drawn. With a mixed fusion^{46,68}, however, the question of whether a molecular compound exists is answered by a single microscopical preparation and, moreover, with very little substance. Furthermore, to decide if the compound is 1:1 one need only observe melting of a weighed 1:1 mixture previously melted and resolidified. If the compound is 1:1 the mixture will behave like a pure compound; if not 1:1, evidence of a eutectic will be apparent.

As an example of a quickly recognized configuration, Fredga and Matell⁶⁹ name the pair α -(1-naphthoxy-propionic acid) and α -(2-naphthoxy-propionic acid). Contrary to all conjecture, the two, thought to have identical configurations, showed a compound in the mixed fusion and therefore proved opposite configurations; the melting diagram confirmed this.

Matell⁷⁰ emphasizes the interesting formation of addition compounds (pseudo racemic compounds) between homologous alkylsuccinic acids (Table a). Pseudo racemates can also occur if CH_2 groups cause the structure difference.

In further work Matell⁷⁴ reports on the investigation to determine the configurations of s-propylmercapto succinic acid and s-methylmercaptoethyl succinic acid in which mixed fusions were again used because it was simple, timesaving and certain (Figure 182).

TABLE a

Molecular compounds between alkylsuccinic acids
(+ indicates molecular compound)

	CH_3^-	C_2H_5^-	C_3H_7^-	C_4H_9^-	$\text{C}_5\text{H}_{11}^-$	$\text{C}_6\text{H}_{13}^-$
CH_3^-	Rac.	+	+	+	-	-
C_2H_5^-	+	Rac.	+	+	-	-
C_3H_7^-	+	+	Rac.	+	-	-
C_4H_9^-	+	+	+	Rac.	+	-
$\text{C}_5\text{H}_{11}^-$	-	-	-	+	Rac.	Rac.
$\text{C}_6\text{H}_{13}^-$	-	-	-	-	+	Rac.

IX. CRYSTALLIZATION OF SUPERCOOLED MIXED FUSIONS

A. Quasi-Eutectic Sycrystallization

1. Two-Component Mixtures

Separation of crystals from any liquid mixture depends on the concentration and temperature. Normal separation occurs, then, at a particular concentration and temperature if supercooling is avoided.

In Figure 183, the melting or saturation curves aE and Eb separate the range of melt S, unsaturated in both components, from the lower left and right ranges which are unsaturated, in some regions, in one component⁷⁵. Upon cooling of a melt N (concentration below the eutectic) to the temperature A', the component A therefore crystallizes when the corresponding state of supersaturation is reached. The composition of the melt is displaced toward a higher concentration of B; the change proceeds as the temperature decreases along the curve portion a'E. At E, the eutectic point, the melt is saturated in both components. If the temperature now decreases further, both components will separate simultaneously. Solidification of mixture N proceeds, therefore, in two steps: primary separation of A at the temperature a' followed by secondary separation of the remaining melt with the eutectic composition at E. On the other side of the diagram, at a concentration above the eutectic, solidification proceeds analogously except that component B crystallizes first. Only mixtures having the eutectic concentrations separate upon cooling, in one single act of crystallization, as "eutectics".

Eutectic crystallization begins at a definite point and progresses in a homogeneous crystallization front consisting of lamellar, granular or spherulitic arrangements of mixed crystals of A and B. They are eutectic grains⁷⁶, eutectic colonies⁷⁷ or eutectic fibers⁷⁸. Simultaneous separation of components (eutectic sycrystallization⁷⁹) in a common crystallization front leads to a similar arrangement, designated a eutectic structure. Similar regular structures^{78,80} with eutectic grains are present, according to the x-ray work of Straumanis and Brackss⁷⁸, in eutectic mixtures of cadmium and zinc, tin and bismuth, as well as zinc and tin.

The eutectic structures of other substances, i.e., the approximately parallel arrangement of the component crystals cannot, as a rule, be traced to the crystallographic structures, but instead depends on the orientation of the faster growing crystal faces. It should therefore be interpreted as a regular structure caused by alternate orientation of both components in a eutectic crystallization. Since metal lattice structures frequently display relatively close relationships, regular orientation in eutectic crystallization is the rule with them. The "degree of relationship", which leads to mutual seed crystal induction and to oriented arrangement of both components frequently does not depend on the chemical relationship, however, as the very broadened appearance of oriented growth shows (page 124).

During microscopical analysis of the structures of crystallized mixtures, one finds, assuming typical equilibrium conditions, one portion of a component as a primary precipitate, with the remainder, as well as the second component, entirely in the eutectic. But a mixture of the combination at the eutectic point shows no primary separation, instead consisting only of the eutectic. From structural analysis, therefore, conversely the concentration of an existing two-component crystallization can be determined.

Upon supercooling of a mixed melt, there is a deviation from the normal precipitation procedure. It is of essential importance whether the heat of

positions on either side crystallize as quasi-eutectics. In these systems the "range of constant CV" surrounds the eutectic point. This anomaly is the least frequently encountered.

Type C represents a combination of types A and B in which, for a small degree of supercooling, the system shows type A but on further supercooling becomes type B, so that now the "range of constant CV" again surrounds the eutectic point. Except for A, type C is most frequently found. Naturally all variations exist between the three types.

Figure 186 contrasts the three kinds of anomalous solidification with normal behavior.

Figures 187-190 show the SB's of several type A systems; Figures 191 and 192 show two systems with SB's of type B; Figures 193 and 194 of type C.

Figures 195 and 196 (Plate XIV) show quasi-eutectic syncrystallizates of two distinctly different habits. Acenaphthene and sulfonal separate in parallel threads fastened together in bundles. Antipyrène and sulfonal form dendritic syncrystallizates with foliated "eutectic kernels".

Since our results show that the relative behavior of the CV is the conclusive factor in the separation of supercooled mixtures, certain restricted hypotheses and predictions concerning the occurrence of one of the three types of SB can be made. Of special significance are, on the one hand, the specific CV or a substance and, on the other hand, the concentration ratio of the eutectic mixture. If one chooses, therefore, to mix a substance A having a greater CV with a component having a lesser CV but a much higher melting point, then a type A with a much stronger deviation of the SB from the eutectic point is to be expected. In this case, diffusion conditions are better for the specifically greater CV of the lower-melting component A, since in systems of great melting point difference the eutectic point, as a rule, lies in an area of high concentration of the lower-melting component. Thus, partners can easily be found for substances with specifically greater CV's, for example, azobenzene, naphthalene or diphenyl, which, in mixtures, give SB's of type A with strong deviation. Figure 197 shows such a case with diphenyl:acetanilide ($T=66^\circ$, 10.5% acetanilide) (see also page 151).

If, however, A (for example, aceto-*m*-toluidine) has a very small specific CV then, in spite of the great lateral region of the eutectic point, type B is possible (Figure 198). All of the diagrams shown here have the lower-melting component A on the left and the higher-melting B on the right.

With a melting point difference that is not too large and a specifically large CV of both components, the SB is narrow and deviates slightly from the eutectic point, for example azobenzene:diphenyl of Figure 199. Above all, a large specific CV of both components is a deterrent to anomalous separation; this phenomenon can be understood by running isotherms for CV's of primary and secondary crystallization (see page 150). There are ratios, however, for which the properties of most metals are at least approximately comparable. In a few of the organic systems investigated, notably fluorene:stilbene, crystallization is independent of supercooling (to about 15°) insofar as supercooled eutectic mixtures always crystallize simultaneously and almost no broadening in the sense of an SB is observed. Stronger supercooling could not be studied because of the spontaneously occurring unstable modification of stilbene.

An example of the behavior of the SB upon the occurrence of a molecular compound⁸⁸ is α -trinitrotoluene:anesthesin (Figure 200).

The kind of anomalous eutectic designated type A has also long been known with metals; in Dix's investigation⁹⁰ of the eutectic of Al-Al Fe the phenomenon was called "displacement of the eutectic point"⁸⁵. Other such eu-

tections are Fe-C (graphite), Al-Si, Zn-Zn₃Sb₂ and Pb-Ag⁸⁵. In all of these there is a decided melting point difference, hence the eutectic lies in high-concentration areas of the lower-melting components. Ratios exist, therefore, which are comparable to those in Figure 197 in relation to the eutectic point. Rosenhain and Tucker⁹¹ found a eutectic structure in a small concentration interval in the Sn-Pb eutectic. In perlite (the ferrite:cementite eutectic) a pure eutectic structure also occurs in a concentration range which widens with supercooling.

The conclusion drawn from the supercooling phenomena of metals, that anomalous eutectic crystallization occurs more easily the more different the relative proportions of the two components in the eutectic, is therefore also valid for organic substances. However, this anomaly does not depend only on "the stronger nucleation potential of one of the two components" for the eutectic mixture of such a system precipitates equally well on supercooling whether the one component crystallizes primarily or the crystals of both components occur simultaneously. Because of the greater CV of one component in type A systems that component reaches the sycrystalline state and separates first.

It is assumed that the SB deviation observed with organic mixtures and also their specific behavior with other substances are significant, especially with substances which incline toward supercooling, for example, the silicates. This viewpoint could throw some light on known varying composition of eutectic mixtures of quartz and feldspar in granites. Gackel found⁹⁴, upon investigating granite from Ringebaker and Bornholm and from Lysekit in Sweden, that this was the case with good development of the eutectic structure in varied proportions of quartz and feldspar, from 20 to 38.5% quartz. The order of magnitude of this interval compares as well with organic substances.

Anomalous separation in salt solutions upon supercooling can likewise be observed simply with a cooling stage⁶². The behavior of supercooling salt solutions is completely analogous to the anomalous behavior of organic substances. On the one hand, deflection of the quasi-eutectic sycrystallization region of the eutectic concentration could be observed (type A according to Kofler)⁸⁷; on the other hand, halo formation (page 152).

During crystallization experiments it is easily shown that ice has an especially higher CV than the salt. In aqueous systems in which the eutectic point lies in an area of high water concentration, in analogy to organic substance behavior, strong deviation of the quasi-eutectic region is to be expected with higher salt concentration. This can be seen in the aqueous systems of ammonium chloride, potassium nitrate and sodium nitrate. The eutectic mixture is allowed to crystallize, and cooled further. Then the chief portion of the crystallize is melted with a warm spatula and again cooled. The remaining crystals begin to grow, so that one can follow the solidification at distinct degrees of supercooling. With the eutectic of potassium nitrate:water ($E = -3^\circ$, 17.7 g in 100 g of water), a strong primary growth of ice crystals can already be seen at -5°C . In this case it is difficult to hold the temperature so near the eutectic that genuine eutectic crystallization occurs and ice does not form immediately. In the ammonium chloride:water solution the ratios are similar. In the sodium nitrate:water system the unusually small CV of sodium nitrate leads, in spite of the favorable eutectic concentration ratio, to type A sycrystallization.

2. Three-Component Mixtures

In three-component systems the presence of a ternary SB is limited by

the behavior of the binary SB. The three types A, B and C described above can, however, also be recognized here. Figures 201a and 201b show the ratios in the three-component system azobenzene:acenaphthene:benzil. The ternary point coordinates are 28% benzil, 46% azobenzene, 26% acenaphthene and $39^{\circ 95}$. The binary SB extends into the three-component system as a mountainlike region in which the ridges of the binary eutectic crystallization curve are already formed, while the basal surfaces, before the region of constant CV has been reached, depend on the degree of supercooling.

The region in which the binary SB's overlap corresponds to the ternary SB. In this case, it is in shape of a horn, the point of which is the ternary eutectic point.

Seven distinct crystallization regions divide the concentration diagram of Figure 201a. In spaces I, III and IV, the adjacent components have the greatest CV at all degrees of supercooling, so that these solidify first on nucleating with the ternary mixture. If one nucleates, for example, a mixture of the combination a in region IV, Figure 201a, with the ternary mixture, acenaphthene crystallizes first. The composition of the remaining melt then moves in the direction of the arrow into region IV, approximately opposite a'. At this concentration, a new, two phase crystallize develops, after which the concentration of the remaining melt moves toward region VII. As the third crystallization, the ternary mixture separates together. In the concentration diagram of Figure 201a, the SB's are lowered to 20° upon supercooling; in Figure 201b with less supercooling, only to 50° . The ternary SB lies outside the eutectic concentration, opposite the lower concentration of benzil, i.e., type A.

2,4-Dinitrophenol:acetanilide:benzil is also a type A system. In Figure 202, the SB's are designated upon supercooling to 20° with solid lines; upon supercooling to 50° with dotted lines. The ternary point with the coordinates 45% benzil, 33% dinitrophenol, 22% acetanilide and 60° lies, according to our investigation, outside the ternary SB⁸⁷.

As was already mentioned, Tammann and Botschwar⁸⁶ have said the "range of constant KC" surrounds the eutectic point, so that the two-component systems they investigated, acetanilide:2, 4-dinitrophenol and azobenzene:benzil, as well as the three-component system, acetanilide:2, 4-dinitrophenol:benzil, were types B or C. This is only partially correct. According to our investigation, azobenzene:benzil, as well as the three-component system of Figure 202, belong to type A. The error can, for the most part, be traced to the fact that Tammann and Botschwar had incorrect values for the eutectic point, because the eutectic azobenzene:benzil lies, not at 45% benzil, but instead at 38%; in the three-component system the ternary point coordinates are not 25% benzil, 45% dinitrophenol and 30% acetanilide but, instead, 45% benzil, 33% dinitrophenol and 22% acetanilide.

B. The Isotherms of Crystallization Velocity During Supercooling

If, in a binary system, one measures the CV of the first separating component in distinct mixtures below the eutectic temperature, one obtains in the concentration-CV diagrams two curved portions intersecting at the eutectic. The isotherm of primary crystallization appears, therefore, at the eutectic temperature as a uniform curve with a zero value at the eutectic point⁸⁶. The position of the latter in relation to both the concentration and the temperature limits the degree of decrease and/or rise of the isotherm.

If one withdraws still more heat during the resulting primary crystallization at the eutectic temperature, the remaining melt solidifies in a secondary act of crystallization as the eutectic. The isotherm of the eutectic crystallization is then a horizontal line which touches the isotherm of the

CV of primary crystallization at the eutectic point.

Upon solidification of supercooled mixtures, this relationship of the isotherm to primary and secondary crystallization changes. Both isotherms can then be seen to originate from two branches⁹⁶.

The isotherms for the CV of primary crystallization (primary isotherm) have, at the highest point with the pure components, the CV of the corresponding components at the chosen degree of supercooling. The zero point for both components no longer lies at the eutectic concentration, but rather — in agreement with the degree of supercooling — for the component A at the saturation curve Ea' of Figure 184, for the component B at the curved portion Eb'. Since this point in relation to the type of primary crystals lies, for the time being, on the other side of the eutectic concentration, it must cut both curved portions.

Figure 203 shows acetanilide:2,4-dinitrophenol (eutectic 78°, 53% dinitrophenol); Figure 203a shows the quasi-eutectic syncrystallization branch (SB); the isotherms of primary and secondary crystallization for various degrees of supercooling are shown in b, c and d. In Figure 203c, the two primary isotherms continue farther than the broken curves and terminate at 60% dinitrophenol, one of the eutectic concentrations.

The isotherms for the CV of secondary crystallization (secondary isotherm) show divided curved portions which overlap in Figures 203b, c and d, approaching a similar height.

Every primary isotherm intersects the corresponding secondary isotherm. Between m and n, upon simultaneous nucleation with both components, there will be only one crystallization front. Therefore, the curved portion m-n represents the isotherm of quasi-eutectic crystallization. The broken line extrapolation of the primary isotherm of Figure 203a can be determined experimentally, then, if the supercooled melt concerned is nucleated with only one of the two components.

The quasi-eutectic isotherm has a minimum at the concentration where it intersects the two primary isotherms. This concentration diverges, as a rule, from the eutectic composition: in Figure 203a, the place of ultimate minimum-concentration is shown by a dot-and-dash line.

In anesthesin:benzil (eutectic 64°, 52% benzil) (Figure 204), the minimum-concentration diverges relatively little from the eutectic concentration; the SB deviation is much greater, as is that of the minimum-concentration area in azobenzene:benzil (eutectic 52°, 38% benzil); Figure 205a shows an SB corresponding to a type A. The greater anomaly of this system is expressed in Figures 205b and c: the length of the secondary isotherm branch is greater than in the two systems of Figures 203 and 204. The course of the quasi-eutectic isotherm m-n is most unsymmetrical, since on the azobenzene side the secondary crystallization grows very slowly and on the benzil side it grows very rapidly. In this latter portion, the isotherms for quasi-eutectic and primary crystallization can no longer be differentiated. During crystallization apparently only a single crystallization front moves; the two processes, however, run in rapid succession, and, indeed, secondary separation immediately follows primary separation. The coupling is forcibly regulated because in this region secondary crystallization still has the greater crystallization velocity without, however, — because of the composition — being able to overtake the primary crystallization front. The coupled crystallization makes determination of the syncrystallization range limits difficult.

Azobenzene:acetanilide (eutectic 66°, 6.5% acetanilide) is an extreme case. The two components were chosen so that, on supercooling, the CV's ex-

hibit the greatest possible difference (page 148); the high azobenzene (melting point 68°) CV reaches a maximum at $20-30^{\circ}$ supercooling, 640 mm^{86} . The eutectic point — nearly lost by the relatively large melting point difference — lies at 93.5% azobenzene. The addition of acetanilide causes a higher CV and better diffusion conditions since, upon supercooling, the azobenzene CV still surpasses the CV of acetanilide in over-eutectic mixtures so that a type A occurs with strong syncrystallization range deviation (Figure 206a). Figure 206b shows the primary isotherm at the eutectic temperature. Even with a small supercooling of 3° , that is, to 63° , the SB (Figure 206c) is strongly drawn towards the higher acetanilide concentration; the minimum crystallization velocity of the syncrystallization region lies at 13.5%, and therefore moves to the right in contrast with the eutectic concentration around 7%. When the system is supercooled to 60° , the minimum appears to be shifted to 22% acetanilide. The great anomaly is apparent in the especially rapid decline of the secondary crystallization velocity; the intersection point n, at about 40% acetanilide with 3° of supercooling, shifts with 6° of supercooling to above 80%.

With their measurements of the CV in supercooled mixtures, Tammann and Botschwar⁸⁶ also observed that in certain concentration ranges the primary crystallization velocity sinks below the secondary and that, furthermore, the quasi-eutectic crystallization velocity exhibits a minimum. But the two authors misplaced the minimum in the eutectic concentration and also erred in their idea that the quasi-eutectic region surrounds the eutectic point.

C. Halo Formation

Whereas quasi-eutectic syncrystallization of a supercooled mixed melt occurs in the presence of both components, the anomaly of "halo formation", likewise known with metals, depends on the absence of seed crystals of one of the two components; the position of the SB also plays an essential role.

The halo phenomenon was explained by Jiraud through supercooling⁹⁸. Lamplough and Scott⁹⁹, however, reject supercooling theories on the basis of their experiments. Tammann and Botschwar¹⁰⁰, on the other hand, found the basis for these authors' views unsound. When Tammann reduced the concentration, during crystallization, of the melt next to the primary crystals of the component with a smaller diffusion velocity, the second component formed a halo.

Further investigations by Botschwar and Gorew¹⁰¹ led to the conclusion that only one component could form a ring around the second. The two authors state, concerning the series of metals Bi, Sb, Cd, Pb, Sn, that only those on the left (lower melting) form a halo around those on the left.

If one considers the eutectic mixture of Figure 207 with supercooling to e and nucleation with the component A, the following thought presents itself. A can crystallize until the remaining melt has reached, along the horizontal, the concentration m. It was assumed that the right limiting curve of the SB coincided with the saturation curve Ea'. (For the experimental determination of saturation curves, see Kofler¹⁰².) Therefore, the separation of A is ended by lowering the concentration of A to m. If, however, the mixture is now seeded with B, then, since m still lies in the SB, both components immediately crystallize as a quasi-eutectic. The structure shows, therefore, the primary crystals of A lying in an apparently eutectic crystallizate. The same crystallization effect is achieved if the mixture, which has been supercooled to e, is immediately seeded with both components. Then A crystallizes in each case, because of its rapidly climbing CV, before B until the remain-

ing melt has reached the concentration of SB.

In the same region of the SB, halo formation then occurs; however, only if a eutectic melt supercooled to e has been seeded primarily with $B^{82.89}$. Upon crystallization of B, the remaining melt moves to the left, towards s on the saturation curve bEb' . Nucleation with A produces a concentration at which A displays a much greater CV than B; therefore, A very quickly separates. Since the environment of the B crystals has been much enriched in A, a halo of A is formed around the primarily precipitated B crystals. The syn-crystalline solidification of the remaining melt then causes the concentration to return to the right to the SB (r of Figure 207).

The proportions are then especially favorable for formation of a noteworthy halo of A around the primary crystals of B if an over-eutectic mixture (about N of Figure 207) is quickly supercooled to n and primary crystallization of B occurs. Here the concentration of the remaining melt again shifts to the left, towards p on bEb' . Because of the increased difference between the original concentration and the concentration of the remaining melt, halo formation becomes especially prominent.

The behavior in the last case, for example in azobenzene: α -naphthol, can be clearly observed in the analogous concentration halo if one waits for primary crystallization of α -naphthol with an over-eutectic mixture after supercooling. In this case, spontaneous crystallization occurs regularly in over-eutectic mixtures. In other cases, in which spontaneous crystallization is absent, one can force primary crystallization of surplus components in a simple way so that the over-eutectic mixture does not fuse entirely but instead forms over the eutectic temperature only while there are residual crystals. Upon cooling to the desired temperature, this residue forms mostly excellent primary crystals around which halo formation can easily be observed upon injection with the second component. An example is azobenzene:trional.

Figures 208 and 209 (Table XV) show the crystallization procedures of over-eutectic mixtures; Figure 208 shows a normal precipitation procedure; Figure 209, anomalous crystallization. In both cases, it is a question of whether there are analogous, over-eutectic mixtures, as is evident from the additional melting diagrams 208a and 209a.

In normal precipitation, the eutectic crystallizate (Figures 208c, d) follows the primary crystallization of B (Figure 208b).

Anomalous separation of an analogous over-eutectic mixture is shown in Figures 209b, c and d with a mixture of 65% trional with azobenzene. The mixture is heated to 65° , then quickly cooled to 35° . The remaining crystals of B (trional) form fine rhombs. The edge of the microscopical preparation is immediately seeded with both components. Since the selected concentration lies within the SB, both components separate quasi-eutectically (Figure 209b, right). The crystallization front progresses to the region around the primary crystals which is impoverished in B, therefore rich in A; since, however, in all concentrations (lying left of the SB), the CV of A is greater than that of B, A crystallizes first, forming a "halo" around B. Then, if the concentration of the remaining melt again shifts to the right, quasi-eutectic front again proceeds uniformly (Figure 209d).

Under certain conditions, then, a halo of B can form around A as well as of A around B. It is assumed that the corresponding limits of the S of the saturation curves aEa' and bEb' (Figure 210) are maintained. If, for example, a eutectic mixture E is supercooled to e and seeded with A, then A precipitates during displacement of the remaining melt over point 1 and beyond, towards point 2 on the saturation curve aEa' . Subsequent seeding by B causes a greater CV for G; the net result is halo formation by B around A, followed

by syncrystallization. If that same mixture at e is first seeded with B, then the solidification can proceed entirely analogously, except that it is laterally transposed since the concentration of the remaining melt is first displaced towards 3 and, at a maximum, towards 4. Nucleation of A at this point causes a greater CV for A; therefore a halo of A next forms around B followed only afterward by the syncrystallization. From this illustration we can see that with type B for both components, the possibility arises that either can form a halo around the other under certain circumstances. Two conditions, however, are necessary. First, the crystallization cannot always be arbitrarily conducted, because crystallization of one of the components is usually strongly favored and can hardly be suppressed by supercooling, so that, practically speaking, even here only one component can form a halo around the other. Second, with type B also, one of the SB limits usually lies in the region of the saturation limits so that, as with type A, halo formation by only one component is possible.

With type C the type of halo formation depends, naturally, on the degree of supercooling.

If one considers all the above-described conditions for halo formation, then this phenomenon depends upon the direction in which the concentration of the remaining melt is displaced, according to the way in which primary crystallization began in relation to the SB. Halo formation occurs, then, only if the concentration of the remaining melt moves, upon primary crystallization, away from the SB.

Returning to Botschwar and Corew's list of metals¹⁰¹, Bi, Sb, Cd, Pb, Sn, in which a halo was formed only around the metals on the left (page 152), the following considerations present themselves regarding eutectic point location.

According to the investigations of numerous authors, continuous solid solubility occurs in the system of Bi/Sb, so that this case does not belong here. (Since, upon solidification of mixtures of this system, strong equilibrium displacement was observed due to extraordinarily sluggish diffusion, the halo formation observed by Botschwar is probably traceable to layer crystal formation.)

In the Cd/Sb system there is an important melting point difference with the eutectic point at 92% Cd¹⁰³. Since this eutectic forms between the intermediate phase Cd/Sb and cadmium, the question must be whether the primary crystals, surrounded by a cadmium halo are, not pure antimony crystals, but, instead, Cd/Sb (a stable proportion assumed). With Pb/Sb, a melting point difference¹⁰³ is indeed missing; however, the eutectic point here is strongly one-sided, at 82.5% Pb¹⁰³. In the latter case, the right branch of the melting curve exhibits an inflection point. Also in the systems of Pb/Cd and Cd/Sb, the component present in a large quantity in the eutectic has, because the eutectic point has a lateral location due to better diffusion conditions, the greater chance of growing quickly in mixtures, that is, to form a type A of SB which gives the most favorable conditions for halo formation.

Also in the Pb/Sn system (eutectic at 61.5% Sn), the proportion is such that a type ASB with a displacement towards a Pb-rich mixture is probable; therefore tin can form a halo around primary lead crystals.

The zinc halo shown by Scheil⁸⁵ around the primary ϵ crystals in photomicrographs of the Zn/Mn system, as well as the zinc halo around primary Mg-Zn crystals in Zn/Mn system, arranged itself equally informally in the representations obtained with organic mixtures, the halo of Al_3Mg_2 around primary aluminum crystals¹⁰⁴ of the eutectic Al- Al_3Mg_2 can be explained in the same way.

As was explained above, halo formation is assumed to be based on the fact that the component with the greater CV under the given restrictions (which frequently exist in greater concentrations) is absent at first, which in general is the least probable case, not to mention that the nucleation velocity of crystal formation with metals is generally very great. It is obvious that this can partially be traced to the fact that halo formation with metals is known in only a limited number of systems.

Also in the Al/Si system (eutectic at 11.6-11.7% Si¹⁰⁵), an SB of type A is to be expected with a diversion to higher concentrations of the silicon. Corresponding to this is the photomicrograph, shown in Fuss' book¹⁰⁵, of an over-eutectic, 20% Si mixture. Distinct aluminum halos are seen here around the gray polygon of silicon, the aluminum also projecting in skeletal crystals in the surrounding area. Through the skeletal growth, ground specimens of aluminum crystals from certain places also appear to be primary.

The circumstances which lead to halo formation signify strong coarsening of the crystals for the structures of the mixtures in question. If one considers in this respect, for example, the diagram of Figure 197 with a small degree of supercooling (at about 70°), he sees that the finest syncrystallizate distribution of components occurs from about 30 to 45% acetanilide if seed crystals of both components are present. If, however, acetanilide crystallizes primarily, then, depending on the strong over-eutectic mixture ratio, a considerable shift of the remaining melt concentration to the left soon occurs, which, with diphenol crystallization, leads to decided halo formation. The particle size of a primary solidification product thus formed exceeds that of a syncrystallizate of the same combination many hundreds to a thousand times.

It should still be mentioned that eutectic anomalies can occur even without supercooling, if for any reason a common crystallization front is not developed, for example, with very great quantity differences in the eutectic mixture or with very slow crystallization⁹⁸.

In the first case, the anomaly is connected with the particle size required for a certain type of supercooling. If, for example, the eutectic lies at 5% B, a eutectic lamellar pair must be constructed so that a thin layer of B is accompanied by a layer of A 20 times as thick. Since lamellar width depends on cooling speed, and indirectly on diffusion speed, then, if restrictions for the lamellar width of B are met, A forms no new seed crystals but instead the A-component precipitated with B deposits on the previously existing A-crystals. It is likely that with rapid cooling decreased diffusion velocity (if no other anomalies occur), the stable, fine lamellar structure will itself form a true eutectic structure.

During crystallization of aqueous salt solutions, besides the above-mentioned anomaly, quasi-eutectic syncrystallization with preferred type A occurrence as well as limited stationary halo formation can be conducted experimentally and followed on the cold stage⁶². Since, in these cases, water nearly always has the greater CV, so that the SB diverges to higher salt concentrations, in over-eutectic mixtures ice forms halos around primary crystals of the concerned salt or — more frequently — a hydrate is easily attained.

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XI.* EXERCISES

The exercises are intended to illustrate previously discussed phenomena to demonstrate various techniques. Simple examples have been selected so that each phenomenon appears without simultaneous changes of other kinds. This is done so the beginner will not be confused by the complex changes common in many systems. Most organic substances show, at or near the melting point, not one but several striking metamorphoses.

Since the exercises proceed from simple to more complicated systems, it is recommended that they be studied in the sequence presented. In exercises illustrated by several examples, the process is described in detail for only the first example. Therefore, if one selects the second example in a given exercise he will have to read the description for the first sample. Readily available substances have been chosen as often as possible to make it easy to follow all of the procedures given. The rheostat resistance should be adjusted at the beginning of each experiment, as mentioned in the general chapter, so the rate of temperature increase is about 4°C per minute in the melting point region. This precaution must be carefully observed throughout all of the exercises. To simplify matters, the directions will refer to adjusting the heating rate for an expected melting point at a given temperature. This will mean that the rheostat should be set to give a heating rate of 4° per minute at that temperature.

The microscopical preparation is placed in the slide-moving device of the heating stage and covered with the glass "bridge". The apparatus is always then sealed with the round glass cover plate.

The proper quantity of substance is essential for the success of many of the exercises. Although some notations of this sort are included, the beginner may have difficulty selecting the best quantity. If an experiment fails, repetition is recommended with a different quantity. Impurities often cause melting point variations; hence slight differences from the given values should be expected.

It may be well to point out here some obvious points of technique. Slides and coverslips should be scrupulously clean and forceps should be used to place coverslips on slides and slides on the hot stage. Fingerprints on slides and coverslips naturally affect the preparation, and fingerprints on the hot stage, especially at high temperatures, may cause corrosion.

One should indicate, when giving melting point, not only whether the value is corrected or uncorrected but also if it is a capillary or a micro-melting point. The unmodified designation "melting point" or "melting point (uncorr.*)" should indicate an uncorrected capillary value. Other self-explanatory designations are "melting point (corr.*)" and "micromelting point". The latter is ordinarily presumed to be corrected unless it is otherwise specified.

Many of the experiments are conducted with ordinary white light, others with crossed polars. Polars, when used, usually lie beneath the stage in a rotating filter holder, while the analyzer is placed on the ocular as a cap. If the microscope has a vertical tube, the vibration directions of the polar with respect to the microscope axes are unimportant; to cross the polars, the polars are rotated with respect to each other until the field of view appears black. If one again wishes to work in ordinary light, either one of the two polars is removed. If the microscope has an inclined bodytube, the rotation directions must be oriented north-south and east-west; with these microscopes, the polarizer is usually fixed in the rotating filter holder and the analyzer is rotated to give a darkfield.

* Chapter X, Optical Crystallography, is not translated per se; for it is readily available in other texts.

1. Simple Melting

This exercise covers two examples of the relatively rare class of substances which show no change under the microscope upon the melting point. These examples are given to permit the experimenter to concentrate on the apparatus and on microdetermination of the melting point rather than on the changes before and during melting. For this reason, the descriptions of the procedure with the first example and the observations under the microscope are more detailed than in the later exercises.

Novatophane K, melting point 59°C

One of two small grains of novatophane K are placed on the slide and covered with a coverslip. The grains are crushed into a fine powder by pressure on the coverslip. Move the coverslip back and forth with a finger wrapped in a small doeskin patch, a clean cloth (or a pencil eraser — translator) to achieve better distribution. The preparation then should show, under the microscope, separate crystal fragments of irregular contours and various sizes.

Adjust the heating rate according to an expected melting point of approximately 60°.

No change is noted with novatophane K below the melting point. The smallest particles begin to melt at about 58°C depending on the purity. Of course, these particles are so small that the transition from solid to melt is not easy to recognize. As the temperature continues to rise, however, somewhat larger particles are observed to melt. With the largest crystal fragments, melting starts at the edges. The angles become rounded and, as melting proceeds, droplets form in which residues of the original crystals are visible. These residues diminish and finally disappear at about 59.5°C.

The remaining droplets may have various appearances. The smaller drops are dome-shaped. On focusing up and down, they show a broad, dark border which widens when the tube is raised as the center brightens due to lens action. The larger drops fill the narrow vertical space between the slide and the coverslip. These drops are surrounded by a sharp black rim which can be circular or irregular. It is possible to observe the last crystal residues melting only in the interior of the larger drops.

If, in this or later exercises, the observed melting point is higher than that given, the error may be due to too large a sample. The melting points listed have been determined on optimum amounts of material so that the same melting points will be obtained by the equilibrium method. The above-mentioned melting point, 59°C, was determined by this latter method. In the continuous heating procedure, the novatophane melting point lags about 1°C.

Novocaine nitrate, melting point 102-103°C

The preparation is made as directed above for novatophane and differs little in appearance from it. In addition, the behavior during heating is similar to novatophane except that the melting point range is even larger. The first visible melting is at 102°C; however, the main mass melts at 103°C.

2. Sublimation

The most frequently observed change during heating of organic substances on the hot stage is sublimation. Although most substances sublime, subli-

mates vary as widely in form and appearance as sublimation temperatures (with relation to the melting point) do in magnitude. Rapidly multiplying condensation drops nearly always form next to the crystallized sublimate, especially near the melting point.

The substance is prepared by crushing it between two slides. The top slide is then replaced with a clean coverslip so that the underside of the coverslip remains reasonably free of the original substance. Thus, it is easier to recognize the first appearance of sublimate. To see formation of sublimate watch the preparation in a region where there is not too much substance in the field of view.

The sublimate forms on the underside of the coverslip, where it may be observed most simply with a sharply focused microscope. Focusing on the lower surface is usually easy because of impurities such as small particles or fibers etc. on the undersurface. On the other hand, if the coverslip is completely clean, focus on the original crystals, then slowly focus upward as far as necessary to be sure to have passed the lower surface of the coverslip. Repeat this procedure at short intervals, paying attention to the entire field of vision. The inexperienced observer will first notice the sublimed crystals only after they have reached considerable size, and the temperature will be much higher than that mentioned. Having once observed the crystals, however, one finds it much easier to detect them earlier in later experiments.

The sublimation temperature is not as exact or useful as other phase changes, but it is useful in the overall description. The first sublimate appearance depends on the quantity and particle size of the primary substances and on other, not entirely reproducible, circumstances. Therefore, sublimation temperatures are rounded to the nearest multiple of five in the examples and also in the tables.

The first appearance of the sublimate is also highly dependent on the rate of temperature increase. The resistance at the beginning of the experiment should be set so the temperature increases approximately 4° per minute in the melting point region.

More rapid heating will cause the first visible sublimate to appear only at a higher temperature than that indicated in the exercises. Conversely, slower heating will make the first sublimate appear earlier.

Naphthalene, melting point 80.5°C

Commercial naphthalene usually shows irregular large and small lumps after grinding. The first evidence of sublimation is apparent at about 45°C . Three, four, five and six-sided tablets form, partly on the coverslip surface, partly in the intervening space. They may appear feathery or swordlike. As heating continues, fast-growing swordlike growths appear. Before reaching the melting point, the original crystals usually completely sublime. The microscopical preparation does not resemble the original preparation in any way after sublimation. Pure naphthalene melts sharply at 80.5°C .

Observation of the sublimation process for naphthalene is sometimes disturbed by movement of the coverslip. This is probably due to the very rapid vaporization of naphthalene, which raises the coverslip slightly so that it slides sideways.

Anesthesin, melting point 90.5°C

In contrast to naphthalene, anesthesin does not sublime strongly; most

of the original crystals still remain at the melting point.

The sublimate, first formed at 75°C, grows into various shapes; often these are platelike, transparent squares or rectangles with fringed edges. In addition, small dark crystal grains appear. All grow constantly up to the melting point. At 85°C, drops also condense on the coverslip soon covering the entire field of view except in the immediate vicinity of the crystals. As heating continues and the melting temperature is reached, the original crystals melt first, followed by the sublimate a fraction of a degree later.

The melting of anesthesin (Figure 6) is not directly comparable to the procedure described here. Because the crystals in the figure were obtained by longer microsublimation, the original crystals have completely disappeared.

3. The Equilibrium Method of Melting Point Determination

A larger sample is used for observation of the equilibrium melting point so that larger drops of melt may be obtained and residual crystals may be more readily observed. Heating is regulated, as with the continuous melting point determination, according to the expected melting point.

The equilibrium melting point may, of course, be low because of impurities in the sample. Besides the correct melting point for the pure compound, the deviations to be expected with the usual commercial samples are mentioned for the following examples.

(Translator's note: It is important to measure the equilibrium melting point with nearly all of the sample melted. Especially with impure samples, widely different melting points are obtained depending on the percentage of the sample melted.)

Phenacetin, melting point 135°C

The preparation used for observing the equilibrium melting point must be rather large. It is heated as usual but heating is stopped as soon as melting starts. The temperature, of course, will then rise for a short time, remain constant and then fall. The crystal residues in the melting drops will grow smaller, then remain unchanged for a short time and finally start to grow slowly. During melting, the edges and corners of the crystals become rounded. As soon as cooling begins, the corners and edges become straight and sharp. The crystals then grow faster and faster in prisms and rods, and soon the melt is completely crystallized. The equilibrium point is taken as the temperature at which the crystals have stopped melting and growth has not begun again. This temperature is exactly 135°C for phenacetin.

It is not easy for the beginner to control the heating so that the equilibrium between crystals and melt is observed. At first the heat will probably be cut off too late. Unless heating is stopped at the very beginning of melting, all of the crystals will melt before the temperature begins to fall. If this occurs, the experiment is a failure. If melting is intercepted in time and if the residual crystals begin to grow slowly again as the temperature begins to fall, the experiment is completely successful. With some practice there should be no difficulty catching the equilibrium. As soon as the crystals begin to grow again, heating is resumed. The crystals will grow for a while, then begin to melt again; at that instant the heat should be turned off. Therefore the heat must be on or off, respectively, when the crystals start to grow or to melt.

This experiment can be repeated several times with the same preparation to obtain more accurate values but not indefinitely because anesthesin decom-

poses to some extent during long heating. Consequently, the melting point slowly decreases.

The temperature can also be regulated in another way. If heating is stopped too late and it appears that the sample may melt completely, the glass plate can be removed and one can cool the preparation by blowing on it.

On the other hand, the temperature can be increased more rapidly if the resistance is set for a higher temperature for a short time. Such manipulations are inviting to the impatient worker and can be applied as needed; however, they endanger precise temperature determination and are not advised for careful workers. It is much better to become accustomed to the above-mentioned procedure in which the equilibrium temperature is regulated only by turning the current on and off without changing the resistance and without moving the glass plate.

It is advisable to compare the melting points obtained by continuous heating with those obtained by the equilibrium method. This is done by starting with a new preparation of phenacetin, containing a little less substance than is required for the equilibrium melting point determination described above for novatophane and novocaine nitrate. As soon as the small splinters melt and the edges and corners on the larger ones are curved, the reading is taken. If the determination is carried out properly, a value of 135°C will be obtained, just as with the equilibrium method.

Benzil, melting point 95°C

The equilibrium melting point for benzil should be obtained on a crystal film. For this purpose a quantity, somewhat larger than that used for the previous melting point, is thoroughly melted between slide and coverslip, then solidified on a cold metal plate. If crystals do not form spontaneously, scratch the edge of the coverslip with a dissecting needle.

The sample is heated in the usual way; the equilibrium melting point is best observed between crossed polars. With polars, which are not fixed on the microscope as a rule, one first tests their position, then rotates the analyzer until the field of view is totally dark. The crystal film of benzil then shows polarization colors, usually yellow. At the beginning of melting, the anisotropic crystals stand out very noticeably against the dark background (melt).

Istizin (1,8-dihydroxyanthraquinone), melting point 194°C

Istizin is a relatively high-melting substance, in contrast to the two previous examples. Because istizin sublimes strongly below the melting point, even more material than usual must be used to determine the equilibrium melting point. During cooling, the unmelted seed crystals grow as thick prisms similar to the crystals formed during the last stages of sublimation.

Since istizin's melting temperature is so high, the temperature drops faster after the current is cut off than it does from the lower melting temperatures of phenacetin and benzil. Therefore, melting should be allowed to progress somewhat farther before the current is cut off. Conversely, the temperature does not rise as rapidly and the current should, therefore, be turned on the instant the crystals begin to grow.

4. Solidification of the Melt

With many materials the melt crystallizes spontaneously during cooling,

sometimes several degrees below the melting point and often as much as 10-20°C below. If no crystals form within this range, it is advisable to touch the edge of the coverslip with a needle, whereupon crystallization often results. (It may be necessary to touch the needle to crystals of that substance before seeding the melt — translator's note). The crystals then grow, usually radiating from the point of seeding. The fineness of the crystals generally depends on the cooling rate. Slow cooling is achieved by allowing the preparation to remain on the hot stage after melting with the heat disconnected but with the glass plate and bridge removed. Fast cooling is accomplished by removing the melted preparation and placing it inverted on a cold surface, preferably a metal plate. Quick cooling generally favors development of unstable polymorphic forms.

β-naphthol, melting point 122°C

Crystallization from the melt is observed by cooling the preparation immediately after complete melting, the round cover plate of the hot stage and the glass bridge are removed and the apparatus is allowed to cool spontaneously. β-Naphthol melt crystallizes several degrees below the melting point, though not always at the same temperature; temperatures between 112 and 118°C are usually observed. Nucleation and crystallization take place rapidly throughout the entire field of vision, including even isolated droplets of melt.

The appearance after solidification varies according to the size of the original melt droplets. Each small droplet will solidify to a circular aggregate of partly irregular and partly radiating structures. Large droplets which completely, or almost completely, fill the entire microscope field solidify as compact radiating crystal complexes. Transverse shrinkage cracks appear during cooling and become more and more apparent on further cooling.

If the crystallized preparation is replaced on the hot stage at an elevated temperature, small platelike crystals of sublimate form between the solidified drops.

8-Hydroxyquinoline, melting point 74°C

This preparation should originally be made up with a number of scattered crystals so that, when they melt, many separate droplets will be visible in the field of view.

During heating, some of the substance sublimes as needles, prisms and small droplets. Immediately after the entire preparation melts, heating is stopped and the circular glass plate and bridge are removed. The preparation is then observed as the apparatus cools. Solidification generally occurs between 45 and 65°C. Crystallization usually proceeds from one or more needles, starting at the edge and gradually including the entire preparation. The growth rate of the needles varies depending on the mass of the melted substance they encounter. These variations are easily observed when the visual area includes many melted drops and the entire free space between is filled with small condensation droplets. In the region of the condensation drops, the needles and rods advance slowly. Directly before each advancing crystal, a drop-free area forms as the droplets evaporate because of their higher vapor pressure. It is often fascinating to watch crystals growing towards specific droplets to see if the drops will completely evaporate before they are seeded by the advancing crystal. When such a growing crystal contacts a bigger drop, it grows very rapidly through the entire drop. The

crystal aggregate thus formed is usually smaller than the original drop (due to shrinkage) so that some needles and rods extend from the side opposite that through which they entered and continue to grow through the preparation. The rods and needles do not always grow straight; they are often curved since they depend on the size and distribution of the available drops.

Occasionally a needle may seed a large drop in such a way that numerous needles fan out from the contact point. The needles advance in a relatively even front with no one crystal growing more rapidly or to a greater size than the others. The resulting crystal aggregate then resembles spherulites except that the crystallization nucleus is at the drop periphery.

Nicotinamide, melting point 129°C

Nicotinamide is an example of solidification of a melt into spherulites. The first observation should be in ordinary light, the second between crossed polars.

Enough substance should be used so that the melt completely fills the space between slide and coverslip. Melt all of the substance on the hot stage (or another heating apparatus) and soak up the excess melt with filter paper simply by pressing small pieces of filter paper to the edge of the coverslip. Keep the preparation melted on the hot stage at 50-55°C; in white light one soon sees, in certain places, spherulites forming from the aggregates which arise from round, fine needles radiating from a center.

Solidification between crossed polars is much more distinct. With both polars, a completely melted preparation appears black; however, one soon observes numerous anisotropic seed crystals which grow into distinctly formed spherulites. The resulting aggregate is called a spherulitic mosaic.

5. Hydrates

a) Warming in air

Oxalic acid, melting point 190-191°C

The escape of the water of crystallization from oxalic acid dihydrate under the microscope should be easily apparent. To best observe this process, be sure the microscopical preparation of the substance is not too finely divided so that some larger crystals and crystal fragments remain.

If the electrical resistance corresponds to a heating rate of 4°C/minute at the melting point of anhydrous oxalic acid 190°C, the water of crystallization escapes between 60 and 90°C. At this point, the previously clear crystals become turbid and opaque, next brownish and finally black. The phenomenon begins in one or more places at the margin or within a crystal and fans out. The progressing fronts meet, overlap and soon cover the entire crystal.

After the water of crystallization escapes, polycrystalline anhydrous aggregates remain in the external form of the hydrate crystals (called anhydrous pseudomorphs of the hydrate — translator). Upon closer inspection, one sees that the margins are covered with fine needles; these are anhydrous oxalic acid crystals.

With slower heating, the water of crystallization escapes earlier. If, for example, one holds the hydrate crystals at 50°C for ten minutes, almost all of the water of crystallization escapes.

Citric acid, melting point 151-154°C

The crystals should not be ground too finely. When the preparation is held at 60°C, one observes the clearest possible crystal fragments. Upon further warming, some of the hydrate crystals melt at 70-73°C, the others being transformed with structural changes and increasing turbidity into anhydrous aggregates. The turbidity here is not so great as with oxalic acid, however, and the crystals are not completely opaque. The resulting drops of melt lose water by evaporation and crystallize when they touch a solid anhydrous crystal. This is best induced by sliding the coverslip with a spatula. The melt begins, upon contact with the crystals, to crystallize immediately but slowly; the process is quite apparent with crossed polars. With further heating, the melting point of anhydrous citric acid, 151-154°C, is reached.

b) Heating in paraffin oil

Heating in paraffin oil prevents premature escape of the water of crystallization. After several crystals have been crushed between slide and coverslip a drop of paraffin oil is introduced at the edge of the coverslip.

When warmed in paraffin oil, oxalic acid dihydrate remains unchanged to the congruent melting point; the equilibrium melting point can be observed at 100.5°C, whereupon solid 6-sided grains arise as the preparation cools. On heating and after melting, the drops remain clearly recognizable due to their insolubility in paraffin oil. Vapor evolution usually then begins immediately because, over 100°C, the vapor pressure of the water in the aqueous oxalic acid solution rises rapidly.

Aluminum nitrate, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$

The mostly solid crystals are ground in the usual way. Several clear crystal fragments are placed in the middle of the field of view and heated slowly. The resistance is set for about 70°C. At 60°, part of the water of crystallization escapes, whereupon new gray-looking centers of a lower hydrate appear and grow slowly until finally the original crystals are completely opaque. The heat is removed and the preparation is cooled to about 60°C.

In a second experiment, one places the hydrate crystals in paraffin oil and again heats the preparation on the hot stage. The hydrate crystals now melt incongruently at 75°C and new, doubly refracting, prismatic crystals separate.

Sodium bromide dihydrate

A small sample is first observed between crossed polars. The dihydrate is anisotropic; anhydrous sodium bromide, on the other hand, is isotropic. Pure sodium bromide dihydrate releases water in air at 30°C so that many preparations are no longer dihydrates but already anhydrous. The hydrate can be regained by dissolving the sodium bromide in a small test tube with a small amount of water (sodium bromide is readily soluble) and using a capillary to place a drop of the concentrated solution on the slide. (Recrystallizing directly from a drop on a slide is quicker and easier — translator). Crystallization can be hastened, if necessary, by scratching the slide with a drawn-out glass rod tip. When the crystals are large enough, the remaining

solution is soaked up with filter paper and the preparation is dried by pressing it with small hardened pieces of filter paper. The paraffin oil is then added and covered with a coverslip.

Freshly prepared sodium bromide dihydrate crystals are usually six-sided (Figure 14) with relatively low birefringence. After checking with crossed polars that a doubly refractive dihydrate is indeed present, one then proceeds with one polar only to observe the melting. The crystals begin to melt at 51°C, whereupon new isotropic cubes simultaneously separate out; one can convince himself of their isotropic nature with crossed polars.

If sodium bromide is not completely pure, the water of crystallization does not escape so easily; the dihydrate then remains in air up to 51° without losing water and melts incongruently at this temperature with separation of anhydrous cubes.

6. Refractive Index of the Melt

The skilled microscopist will find it easy to determine the refractive index of the melt. Anyone not familiar with the microscope, however, must be patient and follow the exercises carefully.

In the first experiment, the beginner will be unable to see the glass flakes over a temperature range of several degrees, so that it might seem impossible to read the temperature more precisely than $\pm 1-2^\circ\text{C}$. However, repeating the experiment several times will make an exact reading of the temperature range easier. This initial difficulty was considered when the exercises were planned; it is therefore advisable to work through each carefully, one after another. Good illumination, i.e., axial light, is of paramount importance in achieving good contrast for the glass particles.

The sample is prepared by covering the glass powder with the test substance on the slide. A small quantity of glass powder is placed on a slide and covered with the test substance, and the two are mixed with a needle or by movement of the coverslip (with a pencil eraser — translator).

After working through the following examples and acquiring the necessary practice, one should be able to reproduce the given temperatures within, at most, $\pm 1^\circ\text{C}$.

The Becke Line (see page 30)

The first experiment illustrates the behavior of the Becke line. Make a microscopical preparation with somewhat more novatophane than usual and a very small amount of 1.6598 glass powder. More powder is used than for a simple melting point determination; in fact, enough so that, after melting, several large drops containing numerous glass flakes remain. A large accumulation of glass flakes would interfere with accurate observations and should be avoided.

The hot stage must be heated to 70°C and the temperature should be kept constant between 65 and 75°C during the brief experiment. Place the microscopical preparation on the hot stage, wait until novatophane melts at 59°C and find a large drop containing some isolated glass flakes. The glass flakes are clearly visible since their refractive index differs from that of the surrounding melt. The first step, then, is to determine if the glass flakes have a higher or lower refractive index than the melt. This is possible if one observes the Becke line, the bright line which moves in and out of the glass flakes when the microscope is focused above and below the particles. The condenser diaphragm should be almost completely closed if one is to

observe the Becke line clearly and sharply. At this point, when the microscope bodytube is raised, the Becke line moves from the melt into the glass flake; when the body tube is lowered, the Becke line will move from the flakes into the melt. Since the bright line always moves towards the higher refractive index when the bodytube is raised, the flakes in this case have a higher refractive index than the melt (Figure 18).

The glass flakes are yellowish to orange, as is the Becke line. Frequently the flake margins are bluish. This, of course, depends on the relative dispersion of the melt and particles. A red filter in the filter holder of the microscope sometimes improves visibility of the Becke line. In red light, the Becke line appears as a bright line rather than a colored line although it behaves as described above, that is, it moves towards the center of the glass flakes when the tube is raised.

For the following experiments, use the red filter described on page 32.

Novatophane K, melting point 59°C

Refractive indices, 1.6353 at 93-94°C; 1.6483 at 63-64°C

Place a microscopical preparation of novatophane and glass powder 1.6353 on the hot stage. Cover with the glass plate and adjust the heating rate for a final temperature of 90°C. As soon as the novatophane melts completely, remove the preparation and select a flat drop with only a few glass flakes. The condenser diaphragm must be completely closed during this operation. In white light the glass flakes again seem bordered with color. For further observation, select a sharp-edged particle isolated from foreign matter and from the edge of the drop.

When the bodytube is raised, white light reveals a bright, slightly bluish line moving from the glass particle to the surrounding melt. When the tube is lowered, the line moves from the melt into the glass particle. Notice the intensities on various edges; thin edges give a fainter line than thick edges and, therefore, are less suited to observing the Becke line. Thin particles, consequently, appear to have the same refractive index as the melt over a greater temperature interval than coarse glass flakes.

After finding a suitable portion of the preparation, cover it with the bridge and the glass plate, adjust the stage for slow heating and place the red filter in the microscope filter holder. In red light, the glass particles are less visible but the bright Becke line will be seen clearly as it moves outside the particles when the microscope is focused upward. The melt has the higher refractive index at this temperature (<93°C). The fine focus adjustment is now manipulated constantly so that the Becke line can be observed moving in and out. The Becke line becomes steadily fainter as the refractive index of the melt approaches that of the glass particles. The diminishing contrast makes it difficult to keep the particles in view. If a glass particle should be lost, it can be found by removing the red filter for an instant, because the particle is nearly always visible in white light due to the dispersion difference.

The particle disappears completely at 93°C, but reappears at 94°C in red light. Above 94°C, the melt has the lower index; hence the Becke line moves from the melt towards the glass particle when the bodytube is raised. In the beginning, it is not too easy to define the temperature range in which the glass disappears to 1°C. Instead the particles may seem invisible over several degrees. Further practice and better choice of glass particle (large and chunky) will soon yield more precise results.

The glass powder used may cause small deviations from the temperature indicated above and described in the general chapter. This may be partially due to the form of the powder and partially to nonuniformity of refractive index of the glass. It is, therefore, advisable to repeat the procedure with several different particles in case of divergent results. (Consistently different results indicate impure novatophane — translator.) It should be emphasized that the equilibrium temperature is usually read following a temperature increase, even during determinations below the melting point. In this case, the temperature is allowed to fall until the melt has the higher index. Then the heat is again turned on and the equilibrium temperature is determined. Only in exceptional cases, expressly mentioned in the tables, is the equilibrium temperature determined during cooling.

A second experiment with novatophane is possible with the higher index glass powder 1.6483. This experiment is, in some ways, more difficult for the beginner than that with the glass powder 1.6353 because refractive index identity occurs just above the melting point. The resistance is adjusted for a melting point of 60°C. After the novatophane is completely melted, the heat is turned off and the temperature is allowed to drop to 55°C. When the bodytube is raised, the Becke line moves toward the higher refractive index melt. When heating is resumed, the Becke line grows weaker and weaker, finally disappearing between 63 and 64°C. Above 64°C, the line reappears again, but moves into the glass particles when the tube is raised.

Acetanilide, melting point 115°C

Refractive indices, 1.5204 at 136-137°C; 1.5299 at 109-111°C

The sample is prepared just as it was in the preceding example. After melting, it can be seen that the Becke line moves into the higher refractive index melt when the tube is raised. With further temperature rise, one can clearly see the dispersion phenomenon of colored margins; thus, one can always find selected glass flakes by removing the red filter. The equilibrium temperature of index identity with the red filter is 136-137°C.

With glass powder 1.5299, dispersion colors are especially prominent in white light. After melting, at about 120°C, two colored Becke lines can be seen clearly, a blue one moving into the melt and a red one moving into the glass flakes on focusing higher. The brightness of both halos appears about identical at this temperature. If a red filter is inserted, however, it is obvious that the glass flakes have the higher index. The preparation should now be cooled to about 108°C and reheated at a normal rate. Identical refractive indices are reached, with a red filter, at 109-111°C.

Mannitol, melting point 166°C

Refractive index, 1.4842 at 190°C

A sample of mannitol is mixed with glass powder 1.4842 and melted. The glass particles are clearly visible in the mannitol melt without colored borders even in white light. With the red filter, the refractive indices are equal at 190°C which means that somewhat below 190°C the glass disappears and reappears again at 190.5°C. In this experiment, the glass flakes must be carefully watched since they cannot be found by removal of the red filter as with novatophane. The fact that the glass powder 1.4842 shows no colored borders in the mannitol melt indicates that melt and glass have identical

dispersion curves. In this refractive index determination, it is immaterial whether one uses white or red light, because one obtains the same temperature value in either case.

Observation of dispersion

Novatophane K, melting point 59°C

With glass powder 1.6483, novatophane shows strong dispersion in white light. Immediately after novatophane melts, the glass particles with colored edges are clearly visible in white light. When the bodytube is raised, a bright somewhat blue line moves from the glass towards the melt, while an orange-red line moves towards the glass. When the bodytube is lowered, the movements are reversed. As the temperature increases, the color shades at the edges change. The light blue line moving towards the melt at the beginning becomes duller and bluer while the orange-red line becomes brighter and more orange. Between these two lines, a greenish zone appears at both the high and the low setting of the bodytube. At 75°C both the blue and the orange line appear uniform in intensity for several degrees. Beginning at 80°C and above, the intensity of the orange line is, without doubt, greater than that of the blue line. At the match temperature, the orange line moves towards the inside of the particle as the blue line moves towards the outside when the bodytube is raised. The reason for this phenomenon, as was mentioned in the general test on page 31 is to be found in the difference in dispersion of the melt and of the glass. Because of this difference, uniformity of the refractive index never occurs during heating at the same temperature for all colors. The relative intensities of the various colored bands also depend on the dispersion of the two substances and on the temperature. Since the judgment of relative intensities of two different colors is not very precise, it is much better to use monochromatic light.

Through the use of the red filter, all colors but red are eliminated. Since, however, it is impossible to obtain an absolutely monochromatic red glass filter, the glass particles will never be completely transparent in the melt if very strong dispersion differences exist. Instead, at a definite temperature there is a sudden reversal in movement of the Becke line despite the lack of disappearance of the glass particles. Azobenzene is an example of a system of this type.

Azobenzene, melting point 68°C

Refractive index, 1.6346 at 77°C

Azobenzene is orange in the solid as well as the liquid state. With white light, the glass particles 1.6346 appear greenish-yellow with red edges in the orange-yellow azobenzene melt. When the red filter is inserted immediately after melting, the Becke line moves from the glass towards the melt. During further heating, this line diminishes in brightness, as in other experiments, but the glass particles do not disappear completely in the melt. The strong difference of dispersion is, therefore, noticeable despite the red filter.

The sudden change in brightness, clearly visible with novatophane in white light, occurs with azobenzene in a very narrow wavelength range of the red filter over a small temperature interval at 77°C. When the bodytube is raised at a temperature just below 77°C, the Becke line moves from the glass

to the melt and reverses just above 77°C. This change is sharply visible even without complete disappearance of the glass particles.

7. Eutectic Temperature

In the determination of the eutectic temperature (E.T.), the resistance is set so that the rate of temperature increase is about 4°C per minute in the eutectic melting point range.

Benzamide, melting point 128°C; acetanilide, melting point 115°C, E.T. 82°C

Benzamide and acetanilide (in the amount necessary to half fill a lancet needle) are ground between two slides. From this mixture a microscopical preparation is made with somewhat more substance than is usually used in a melting point determination. Under the microscope, a location is sought where small and large crystal fragments can be observed next to one another. Melting begins at 82°C. Since distribution of the two substances through simple grinding is not entirely homogeneous, the distribution of each in the melted mass is also irregular. The only important matter, however, is to observe the beginning of melting, i.e., the eutectic temperature. With further heating more and more melt forms.

Acetanilide, melting point 115°C, and phenacetin, melting point 135°C, E.T. 90°C

The mixture and the microscopical preparation are produced in the same way as for the previous example. The beginning of melting, the eutectic temperature, is at 90°C.

Acetylsalicylic acid (aspirin), melting point 130-136°C, and acetanilide, melting point 115°C, E.T. 81°C

This example shows that even substances which do not melt sharply and then only with decomposition have good, reproducible eutectic temperatures which are often very useful for identification.

8. Absorption of the Eutectic Melt

Phenacetin, melting point 135°C; sulfonal, melting point 126.5°C; E.T. 106°C, composition 60% phenacetin

Prepare a mixture of about 70% phenacetin, 30% sulfonal and check the eutectic temperature (106°C). Then, place on a microscope slide a piece of hardened filter paper, e.g., Schleicher-Schüll 575, approximately 18 x 18 mm, and on it place a reasonably thick layer of the test mixture. Over the sample, place a second slide crossed with respect to the first. Meanwhile, the hot stage should be heated with the resistance set for a melting point at 135°C. When the temperature reaches about 105°C, place the preparation on the hot stage but omit the round glass plate. At about 110°C the first appearance of melt soaking into the filter will appear on the filter paper as translucent region around the crystals. Now, press the upper slide firmly onto the substance, then lift it together with the attached crystalline substance. Replace the filter paper with a new one and replace the slide. As

soon as spots are again visible on the filter paper, exchange the paper again in the same way. Repeat until no further spots appear on the filter paper even with increasing temperature. This will occur at approximately 130°C when the paper has been exchanged about ten times. As the temperature rises further, leave the preparation on the hot stage until translucent spots again appear on the filter paper; the thermometer will then show about 140°C. Remove the slide quickly. The attached crystals are pure phenacetin, as is proved by their melting point, 135°C.

The temperature values indicated by the thermometer are somewhat high during absorption because the round glass plate is not in place and because the filter paper acts as an insulating layer.

9. Polymorphism

Unstable modification in sublimate

Istizin, melting points: 194°C (I); about 183°C (II)

Make a preparation with as much istizin as would be required for a continuous melting point determination of a volatile substance and set the resistance for a melting point of 194°C.

The first sublimate will appear between 110 and 130°C. It will not be easy in the beginning to distinguish between the original needles and the sublimate. The sublimate appears in two forms: lemon-colored flakes of the unstable modification (melting point 183°C) and yellow-gold rods of the stable form (melting point 194°C). Usually the platelike crystals of the unstable modification either sublime or transform before they reach the melting point. The plates can be observed to grow smaller and smaller because of their high vapor pressure. They finally sublime completely while the stable rods grow continuously in number and size.

When a plate touches a rod, it transforms suddenly to the stable modification. This is clearly noticeable because the previously bright yellow plate turns dark at the point of contact. These pseudomorphs melt with the stable rods at 194°C.

Eldoral (piperidine ethyl barbituric acid),
melting points: 217°C (I); 210°C (II)

Eldoral is allowed to sublime at 170–180°C or a hot bar from a micro beaker to a preheated coverslip. The unstable modification produces fine-angled aggregate which is partly matted, partly gathered in sheets. It is better to wait until a copious sublimate has developed. The preparation is kept on the hot stage at about 120–150°C and again heated quickly (resistance set for 4°C/min. at 220°C). Upon close scrutiny of the preparation one frequently finds, usually next to the fine stems, individual stable shiny grains, often 6-sided. If no such grains can be seen the preparation is heated further. At about 150°C, new stable grains form in many places while the needles generally diminish. With rapid heating, however, it is always possible to melt the unstable needles at 210°C before complete volatilization, while the stable shiny grains melt at 217°C.

Equilibrium melting point of the unstable modification.

Acetamide, melting points: 80°C (I); 70°C (II)

The equilibrium melting point can be determined as well with the unstable as with the stable modification of acetamide. The determinations are carried out between crossed polars.

The unstable modification is obtained most readily by completely melting the substance, between slide and coverslip, to a liquid film. The preparation is then allowed to solidify spontaneously on a cold metal block. During heating on the hot stage, the equilibrium melting point can be determined at 70°C.

After the melting point of form II has been ascertained, the preparation is heated above about 70°C so that all of the substance melts. The edge of the coverslip is then touched with a dissecting needle which has previously been brought in contact with the original substance. It is now immediately seen that, from the point of seeding, new stemlike crystals of the stable phase grow. With further heating, an equilibrium melting point can be determined at 80°C.

Polymorphic transformations

The following experiments are made between crossed polars unless otherwise directed. The birefringent crystalline areas contrast with the isotropic melt so that the transformations are more easily recognized because of the changes in polarization colors.

For some experiments in this and especially in the following chapter, it is advantageous to have a second polarizing microscope in addition to the hot stage microscope. Much time can also be gained by having a small hot plate set constantly at a temperature of 180-200°C. The hot bar, of course, is most suitable for all temperatures from 50-250°C.

In the following experiments, enough substance should be used so that the melting and subsequent solidification fill the entire space between slide and coverslip.

Veronal, melting points: 190°C (I); 183°C (II);
181°C (III); 176°C (IV)

Only modifications I, II and III are obtained when veronal is crystallized from the melt.

A crystalline preparation of veronal is completely melted, then quickly placed on a cold metal block. It is advisable to overheat the preparation slightly and to avoid touching the edge of the coverslip during cooling since otherwise the stable modification frequently crystallizes out. If these precautions are taken, modification II crystallizes on rapid cooling. The preparation is placed in the hot stage at about 90°C and observed between crossed polars.

The large rods with transverse shrinkage cracks transform to modification II at 110°C. The resulting preparation appears as a multicolored, spotted crystalline layer. A transformation into the stable modification I begins at about 150°C from a number of separate nuclei. The large crystals that form are less strongly doubly refractive, so that the polarization colors are gray or black.

Modification IV can be produced most easily in the following way. Modification III is produced by melting and rapid cooling. Then the coverslip is lifted from the slide and replaced loosely. A small drop of 96% alcohol is allowed to flow in. After about half a minute new nuclei form; these are of two types: the first are pyramidal dense grains of modification II (183°C); the second are rectangles, with a variety of interference colors, of modifi-

cation IV which frequently show twin striations. Gradually the original modification III dissolves entirely and II and IV remain in the preparation (with some isolated stalks of I). During evaporation it can be seen that the double pyramids of modification II are more stable than the rectangles of IV because the rectangles partially dissolve, the corners and edges becoming rounded, while the double pyramids of II enlarge. From watching the solution process, we can conclude that modification III (181°C), arising from the melt at room temperature, is more soluble and therefore more unstable than IV (176°C). This confirms enantiotropism between modifications III and IV with a transition point at 135°C.

Enantiotropism

Trinitro-t-butylxylene, melting points: 114°C (I) = 107°C (II); III unstable

Melt a preparation completely and cool on a metal block. Coarse spherulites of modification I, unstable at room temperature, crystallize. Place the preparation in the hot stage and observe with polarized light, first at room temperature, then as the temperature rises with the resistance set for 85°C. At room temperature, one observes the separate growth of new, large-surfaced, frequently striped, colored crystals which arise from form II, which is stable at room temperature. At first, the new crystals grow slowly, then, with increasing temperature, they quickly grow back through the original spherulites. At about 80°C the transformation slows, then comes to a standstill at 84°C, the transition point; with further warming, the crystals revert to the stable form I. The transformation process can be reversed at will by alternately heating above or cooling below 84°C.

Modification III changes rapidly to I at room temperature. If one examines a preparation closely enough during solidification, one can frequently follow the transformation from III to I. One can also recognize the point in which II is first formed, even after the transformation to the rearrangement structure.

2,4,6-tribromophenol, melting points: 94°C (I); 92°C (II)

A crystal film is produced in the usual way, through rapid melting and solidification. The result is a radiating aggregate of the high-temperature form I. After about five minutes, clusters of form II, stable at room temperature, appear in separate areas. If such clusters do not appear for a long time, some of the original substance should be used as seed crystals at the edge of the coverslip. As the preparation is heated on the hot stage in the normal way, the growth of form II, stable at room temperature, can be followed easily. During heating, many new clusters form. If the temperature nears 80°C, the transition front of II progresses more and more slowly until it stops completely at 84°C. At this transition temperature, the two crystal phases are in equilibrium. Upon further heating, the transformation is reversed, form II being reconverted to the original form I. This usually happens very rapidly so that it is not always possible to determine the melting point of form II at 92°C.

10. Melting Point and Refractive Index Determination of Highly Volatile Substances

Determining the melting point for strongly volatile substances can be

difficult for many reasons. First, in many cases the entire substance evaporates before it reaches the melting point and, second, precipitation of the sublimate on the coverslip can make visibility very poor.

In many cases these annoyances can be avoided if two precautions are taken. First, more substance than usual is used, about as much as is necessary for a refractive index determination; large coverslips are also used, 21 x 26 mm or even larger. In many cases it is favorable if the volatile substance is allowed to sublime from a small dish or ring with about a 15-mm diameter in a thick mass onto the large coverslip. To attain uniform thickness, the coverslip should be placed on the support. It is also favorable to begin with the preparation about 20°C below the expected melting point. If camphor is treated in this way and the preparation is placed on the stage at about 190°C, some melt usually soon appears which moves here and there on the pavementlike aggregate. If enough substance is present, it is possible to observe the melting point at 206-208°C even with rapid evaporation. The heat-induced sublimate precipitates only at the side of the bridge when larger coverslips are used and therefore causes fewer visibility problems.

One determines the eutectic with salophen by using camphor in a crystal film. Mix and grind salophen with camphor in the usual way, cover a large sample with a coverslip, melt it quickly on the hot stage and allow it to solidify. To be certain of having the stable form of salophen, seed the melt with the original powder at the edge of the preparation. In the film preparation, the eutectic melting can be easily recognized at 153°C.

The second possibility, on the one hand, for a melting point determination and, on the other hand, for a refractive index determination with glass powder, is the silicone chamber. The chamber ring described on page 16 with an 8-mm bore is coated on the underside with semisolid silicone gum and a 12-mm coverslip is pressed into the dent provided in the silicone layer. One then piles a small heap of the test substance on the coverslip, about as much as for the refractive index determination. The prepared brass ring, together with the silicone mass, is now pressed down on the slide so that the substance remains as near the center as possible under the ring opening. The ring, still under pressure, is turned on the slide so that the silicone mass forms a completely closed zone which can be easily seen because the air bubbles in it are easily visible. The preparation is now placed in the movable slide holder in the hot stage and a coverslip is loosely placed in the dent. This procedure eliminates the glass bridge.

As an example, let us determine the refractive index of anthracene. The melting point of anthracene can be determined directly under a large coverslip. However, the usual method of refractive index determination is difficult because of the volatility of the anthracene; therefore the silicone chamber is used. In the usual way, some of the glass powder 1.6126 is scattered in the middle of a slide together with a large quantity of anthracene. The previously prepared chamber ring (with a coverslip) is pressed on the slide and the ring is turned to test whether the silicone seal is complete. This entire preparation is placed on the movable portion of the hot stage, which has been heated to 220°C. The chamber ring is closed above with a coverslip and the entire apparatus is covered with a glass plate. The temperature is raised (resistance at 240°C!) and the melting of anthracene is observed. After finding a glass flake in the usual way, one can determine the equilibrium temperature at 244-246°C without being the least troubled by the volatility.

11a. Melting Points and Micro Melting Points on the Hot Bar

The hot bar must first be standardized for room temperature variation. Phenacetin is sprinkled on the appropriate area of the hot bar and the sliding pointer is set at the melt limit. Then the room temperature indicator (the rider over the scale portion of the sliding pointer — translator) is slid to 135°C, the known melting point of phenacetin. Acetanilide is then sprinkled on the bar and its melting point is determined through movement of the pointer without, however, any change in the rider's position. With pure acetanilide, a sharp melting point is obtained at 115°C.

If one now wishes to determine the melting point of an unknown substance, one sprinkles the fine powder of the substance (coarse-grained preparations are first ground between two coverslips) in very small quantities in the middle of the hot bar and then, depending on whether the substance melts or remains crystalline, sprinkles a good bit of the substance either above or below the first place. In the interval in which the melting point is found to lie, it is best to sprinkle additional substance in a strip. This is most easily accomplished as one places the substance, ground between two slides (frequently pulverization is necessary), on the hot bar by slipping one slide off over the other. The melting limit and its sharpness can be seen especially easily in a strip.

If the melting point of the substance being tested lies above or below the range included between the melting points of phenacetin and acetanilide, it is advisable, for greatest accuracy, to standardize the bar in the corresponding range with other test substances. The hot bar should be cleaned quickly. If there is no danger of mixing, it is better not to clean it after each determination because the accuracy is lowered for a few minutes after each wiping.

Acetylsalicylic acid (aspirin) is an example of an unstable substance. Sprinkle the powdered substance on the hot bar and take a reading after ten seconds. One usually finds a melting point of 143°C, often less. After about one minute, the melting limit is 140°C, after two minutes 137°C. If the preparation already melted below 140°C at the beginning, the melting point sinks below 130°C after two minutes.

A further example of an especially unstable substance is picrolonic acid. Melting begins above 150°C but the melting limit quickly recedes to lower temperatures — after the usual ten seconds to about 140°C, after twenty seconds to about 130°C. Further decrease is slower, so that after thirty seconds the temperature remains at about 125°C. The melt soon becomes dark brown.

11b. Mixed Melting Point

To determine a mixed melting point one proceeds in a different way. First scatter a long strip of the first substance on the hot bar and determine the melting point. Leave the pointer at the melting limit and scatter some of the second test substance over the still solid particles of the first standard substance. The mixed particles soon begin to melt to the right of the pointer, at lower temperatures. In another kind of determination both substances are placed on the hot bar in long parallel lengthwise strips which must not touch, and the melting points of standard and test substance are compared directly. Then the still solid particles of both substances are mixed with a needle and one watches to see whether the test substance melts at a lower temperature.

This procedure should be carried out with urea and phenacetin. Each substance is carefully sprinkled on the hot bar in a strip about 0.5 to 1 cm wide and the melting points are compared. It is clear that urea melts only slightly below phenacetin. Then the needle is used to mix the particles of both substances which are still solid below 135°C and the lowering of the melting point is observed.

There is a third way to determine a mixed melting point: the substances are finely ground together between two slides, then the melting point of the mixture is determined.

12. Mixed Fusion for Thermal Analysis

It is advisable to draw a temperature-composition diagram for each example similar to the appropriate general diagram in Chapter 5 and to insert in each the melting points, eutectic temperatures etc. This will make the mixed fusion much easier to understand and will develop familiarity with the composition diagram.

- a) Simple eutectic (Figures 49 and 50).
Acetanilide, melting point 115°C;
benzil, melting point 95°C;
eutectic temperature 78°C.

Place about a half microspatula point of acetanilide on a microscope slide at the edge of a square coverslip (15 x 15 mm is best) and melt it on a hot plate at 120°C. As soon as half the space between coverslip and slide is filled with melt, chill the preparation on a cold metal block.

Now place the same amount of benzil at the edge of the coverslip, not on the completely free side but on one of the two sides near the acetanilide (b of Figure 48). When the preparation is heated to about 100°C, the benzil will flow along the solidified acetanilide and fill the remaining free space. The benzil melt is then also chilled on a cold metal block.

The hot bar is especially convenient for preparing a mixed fusion; place the preparation with coverslip and the small heap of substance below the melting temperature range and slide it slowly to a higher temperature until the substance melts and flows between slide and coverslip. After solidifying the preparation on a cold block, proceed similarly with the second substance. The preparation can be more quickly prepared in this way than is possible on the hot stage. (An alcohol lamp is even faster — translator.)

In a mixed fusion preparation thus prepared, the zone of mixing of the two substances can be seen easily even with the naked eye. The preparation is laid on the hot stage, the zone of mixing is brought into the field of view, the heat is switched on and the corresponding resistance is set at the expected eutectic temperature of about 80°C. Observations with a mixed fusion preparation are always between crossed polars because then the melt appears as a dark line in strong contrast with the anisotropic crystals (Figures 49 and 50). Melting begins in the zone of mixing at 78°C; a dark line forms between the two substances and widens towards benzil with further temperature increase. At about 94°C, shortly before the pure benzil melts, the heat is shut off, the round glass plate is removed and the preparation is allowed to cool slowly. The benzil again begins to grow and approach the acetanilide, and the zone of mixing finally crystallizes.

- b) The components form a molecular compound (Figures 53 and 54). *p*-Nitrophenol, melting point 113°C; benzamide, melting point 128°C; $E_1 = 84^\circ\text{C}$, $E_2 = 91^\circ\text{C}$; melting point of the molecular addition compound, 98°C.

The microscopical preparation is made in the same way as above by melting the higher melting benzamide first, then the *p*-nitrophenol. The zone of mixing should be much wider with these components (by remelting several times — translator) and the molecular compound is then clearly observed. Place the contact preparation on the hot stage at about 75°C and reheat it slowly with the resistance set for a melting point of about 100°C.

At 84°C the eutectic melt appears between *p*-nitrophenol and the addition compound as a dark strip which widens as the temperature rises. The second eutectic, between the molecular compound and benzamide, melts at 91°C. The molecular compound is then apparent as a birefringent strip between the two melted eutectics. It dissolves (melts) slowly, disappearing entirely at 98°C.

- c) The components form two molecular compounds, M_1 and M_2 (Figures 55 and 56). Decamethylenedicarboxylic acid, melting point 129°C; nicotinamide, melting point 129°C; $E_1 = 119^\circ\text{C}$, $M_1 = 125^\circ\text{C}$, $E_2 = 122^\circ\text{C}$, $E_2 = 122^\circ\text{C}$, $M_2 = 123^\circ\text{C}$, $E_3 = 117^\circ\text{C}$.

Prepare the mixed fusion as follows:

Set the hot stage at about 125°C and place on the stage a slide holding a small quantity of the two components, each at opposite edges of the coverslip. At this temperature the two substances melt and run under the coverslip. After about half a minute, remove the preparation from the hot stage, place it on a cool surface and allow it to crystallize. Meanwhile, cool the stage to 80°C, replace the preparation and reheat it slowly with the resistance set for about 130°C. On heating, a polymorphic transformation occurs between 80 and 100°C as the nicotinamide changes to the stable modification. On continued heating, the eutectic between nicotinamide and M_2 melts at E_3 (117°C). Almost immediately thereafter at 118°C, E_1 , the eutectic between decamethylenedicarboxylic acid and M_2 ; next the eutectic between the two molecular compounds melts at 122°C, only a few degrees below the melting points of M_1 (125°C) and M_2 (123°C). Heating must not be too rapid if one wishes to detect and record all seven melting points.

Hydroquinone, melting point 172.5°C; antipyrène, melting point 111.5°C;
 $E_1 = 102^\circ\text{C}$, $E_2 = 118^\circ\text{C}$, $E_3 = 120^\circ\text{C}$ (Figure 57, Plate IX)

Completely melt the hydroquinone under one-half of the coverslip at about 180°C, then solidify it through cooling. Add the antipyrène at about 120°C. Since antipyrène crystallizes sluggishly, it is a good idea to scratch the coverslip edge with a dissecting needle. Leave the preparation on the hot stage or hot bar for a time at 30–50°C, and gradually the zone of mixing will also solidify.

When the entire preparation has crystallized, set the resistance for 110–120°C and heat the preparation. At 102°C the first eutectic melts, forming a dark strip between antipyrène and the adjoining molecular compound which widens until the melting point of antipyrène is reached at 111.5°C.

The two other eutectics melt shortly, one at 118°C, the other at 120°C. At first, the two molecular compounds remain as birefringent strips; then they too melt, one after the other.

- d) An incongruently melting molecular compound (Figure 58).
Carbazole, melting point 244°C; α -trinitrotoluene, melting point 81°C.

Incongruently melting compounds form most readily between components whose melting points differ strongly as in this case. The mixed fusion is produced in the following way. Place a slide with a small amount of carbazole, covered with a coverslip, at about 250°C on the hot stage and melt it quickly. After the melting, place the preparation on a wood support; the melt solidifies immediately.

Since the preparation remains hot for a long time, trinitrotoluene can be melted at the edge of the coverslip so that it flows into the free space.

This preparation is laid once more at 180°C on the hot stage or the hot bar for about three minutes (to allow diffusion to widen the zone of mixing — translator). Then it is transferred to the cold microscope and, as quickly as possible, the edge of the carbazole is observed. After a few seconds, the molecular compound grows along the carbazole front. If trinitrotoluene does not crystallize shortly, scratch the coverslip edge.

Now the preparation is warmed in the hot stage; at 77°C, the eutectic between trinitrotoluene and the molecular compound melts. No second eutectic appears with further heating. Instead the molecular compound melts slowly, disappearing completely at 135°C. Frequently, the molecular compound appears to melt below 135°C due to rapid trinitrotoluene diffusion. The correct melting point, however, can be determined in each place that the molecular compound has grown over the carbazole. To see this better, one should heat the preparation above 135°C so that the molecular compound is definitely melted away. Then, after shutting off the heat, place one or two metal cubes next to the preparation and observe the carbazole crystallization front. After a short time, the long rods of the molecular compound reach down in many places; they grow partly in the melt, but in several places they also grow over the carbazole crystals clinging to the surface. Now heat the preparation further (after removing the metal cubes) and observe each place where superposition has occurred. The superposition consisting of the molecular compound melts at 135°C.

The three-phase equilibrium at the transition temperature is investigated in an equimolecular mixture of carbazole and trinitrotoluene. A sample of this mixture is quickly melted between slide and coverslip, then allowed to solidify. Upon heating the sample to 135°C, one sees the addition compound melt while carbazole flakes precipitate. Try to find a place where the carbazole crystals are already present and observe the three-phase equilibrium between the molecular compound, the carbazole crystals and the melt at 135°C. Where no carbazole remains, the molecular compound "overheats" above the transition temperature due to a lack of carbazole nuclei, since the true congruent, though unstable, melting point of the molecular compound lies rather high above the peritectic.

- e) Miscibility gap of the liquid phase.
Acetamide, melting point 80°C; benzil, melting point 95°C
(Figures 60 and 61).

In a spontaneously solidifying mixed fusion, acetamide is nearly always in the unstable modification II (70°C). With heating (set the resistance at about 70°C!) the eutectic between acetamide II and benzil melts at 67°C; acetamide II melts at 70°C. Now, set the resistance for about 90°C; at 88°C a liquid:liquid meniscus appears near the benzil front. Upon further heating to 93°C, this dark line between the two liquid phases vanishes. Above this critical solution temperature (CST), the two melts are again miscible in all proportions. After the fine dark line disappears, the two liquid phases can still be recognized since their refractive indices differ and at first only a very narrow strip mixes completely. With further heating, complete equilibrium soon occurs. If one again turns off the heat, the sharp separation line suddenly reappears at 93°C. If the preparation has been above 93°C for some time and then cools rapidly, many immiscible droplets form near the sharp limiting line of the two liquids since diffusion cannot progress so rapidly.

To test the behavior of the stable form of acetamide, inoculate the corresponding edge of the coverslip before heating. Now the eutectic lies at 77°C. The behavior on further heating is, of course, unchanged.

f) Isomorphism: type I (Figure 64).

Azobenzene, melting point 68°C; stilbene, melting point 125°C.

A mixed fusion preparation is partially melted so that the stilbene remains solid; the preparation is placed on the hot stage at 70°C and the crystallization front is observed. Cooling is partially effected by blowing and partially by a small cool block laid next to the preparation.

The jagged stilbene front soon grows into the azobenzene melt without any phase change. After complete solidification, no line is discernible between the two substances; the plates or rods which grow from the stilbene into the azobenzene show uniform extinction and no differences. When the polarizer is removed, one can see the yellow color of the azobenzene. The experiment is often disturbed by general crystallization (nucleation of the azobenzene), but it is easily repeated.

When the solidified preparation is heated, melting begins on the azobenzene side and continues across to the stilbene, corresponding to a type I Roozeboom diagram.

g) Isomorphism: type III Roozeboom (Figure 65).

Butylethylbarbituric acid (soneryl), melting point 126°C;
allylbutylbarbituric acid (idobutal), melting point 129°C;
minimum melting solid solution 120°C.

Place a sample of idobutal under the edge of a small coverslip and place it on the hot stage maintained at just over 130°C. As soon as the melt fills about half of the space under the coverslip, add a sample of soneryl at the open edge of the coverslip so that it flows into the remaining space. Then place a cooling block beside the slide on the idobutal side, simultaneously feeding in seed crystals of idobutal with a needle. Below 120°C the crystal-front grows toward the zone of mixing. On further cooling, the crystals grow with greatly diminished speed through the mixing zone into the soneryl melt. The velocity then increases again. If the hot stage is reheated after complete crystallization, a minimum melting composition shows at 120°C as a dark strip between crossed polars in the zone of mixing. Diphenylacetylene (60°C)

and azobenzene (68°C) also form a type III mixed crystal series.

- h) Isomorphism: type IV Roozeboom (Figure 66).
 Stilbene, melting point 126°C; dibenzyl, melting point 53°C.

Quickly melt a mixed fusion and place the stilbene side on a metal block. As soon as crystallization begins, transfer the preparation to the hot stage at 50°C. The crystal-front of stilbene is the same as it was with azobenzene, with sharp toothlike protuberances. This front does not continue to grow as such but fast-growing crystals of dibenzyl appear from several different nuclei. After complete solidification, again heat the preparation; melting begins in the pure dibenzyl.

The peritectic reaction (page 214) can be observed easily in a five to eight percent stilbene-containing mixture. The mixture is melted between slide and coverslip and solidified on a metal block. Mixed crystals of the dibenzyl type appear; the small melting curve portion of these lies to the left in Figure 129b and reaches to K. The prepared mixture lies (in the schematic Figure 69) between K and P. If the preparation is now heated slowly, somewhat below 54°C the mixed crystals begin to melt corresponding to the low, solid curve. Soon afterwards, at 55°C, at the peritectic temperature KPS, diamonds of the mixed stilbene-type crystals appear (the right curve branch of the diagram, Figure 129b, from K upwards to the right). Figure 71 illustrates the process. As soon as the diamonds appear, blow on the preparation to cool it, after which the equilibrium condition of the three phases will appear, namely of the dibenzyl type (rods), the stilbene type (diamonds) and the melt. If the preparation is very slowly cooled further, the reverse procedure is observed; the diamonds begin to dissolve (above to the right), while the rods of the dibenzyl type grow. As a rule, the peritectic reaction does not proceed to the end with the usual cooling rate, because through the rapid growth of the dibenzyl crystals the stilbene diamonds are soon completely enclosed, so that no reciprocal action is possible with the melt. The complete result of cooling can be reached only if one cools the preparation extremely slowly.

- i) Isomorphism: type V Roozeboom resulting from isodimorphism. Benzalaniline, melting point 51°C; azobenzene, melting point 68°C; E = 40°C (Figure 131), page 240.

Completely melt a mixed fusion of azobenzene with benzalaniline on the hot stage. Turn off the heat and, just below 68°C, seed the azobenzene by touching the melt at edge of the coverslip with an inoculated needle. When the preparation is cooled below 28°C, azobenzene grows isomorphously into the melt of benzalaniline. This benzalaniline modification is unstable but isomorphous with azobenzene (type I Roozeboom); if a transformation does not result spontaneously, the crystals, resulting from the isomorphous growth into benzalaniline, melt at 28°C. Upon cooling again, the azobenzene is again allowed to growth into the benzalaniline melt and the transformation into the stable modification of benzalaniline is induced with a needle holding crystals of stable benzalaniline. This is easily observed and proceeds quickly. With subsequent heating, one sees that the transformation progresses still further into the mixing zone, until the eutectic strip melts at 40°C. Therefore, a type V Roozeboom system is shown by the transformation of unstable benzalaniline into the stable form.

13. Quantitative Determination of Two-Component Mixturesa) Using glass powders

Quantitative analysis with glass powders depends on the fact that the refractive index of liquid mixtures is usually a straight line relationship. To test this, use a mixture of m-xylene (63°C) and phenacetin (135°C). With glass powder 1.5101, the indices will be identical for m-xylene at 79.5°C and for phenacetin at 134.5°C; connect the two points with a straight line (page 113). The equilibrium temperatures of all mixtures will lie on the straight line joining these two points.

First melt a mixture of 30% phenacetin, 70% xylene on a small aluminum foil on the hot bar quickly, then allow it to solidify. Pulverize the mixture between two slides, mix it with glass powder 1.5101 and determine the equilibrium temperature. Conduct the same experiment with a 50% and a 75% phenacetine mixture. The three temperatures are 96°C, 107°C and 121°C.

b) Microscopical determination of the primary crystallization point

This should be determined using a benzil:acetanilide system. The behavior of both substances in a mixed fusion is first observed, the simple eutectic is melted at 78°C and the morphology of both crystallizes is examined simultaneously. Between crossed polars, benzil is yellow while acetanilide, on the contrary, shows numerous interference colors. Two mixtures are prepared, one with 20% acetanilide and one with 40% acetanilide. The 20% mixture is first melted on a slide (on the hot bar) quickly and a coverslip is laid on the melted drops. If too much melt is present, quickly press a filter paper on the hot preparation. The preparation solidifies quickly (through inoculation, if necessary) and then is heated on the hot stage. At 78°C, the eutectic melts and clear homogeneous rods, yellow between crossed polars, remain; these are easily recognized as benzil crystals because of their morphology observed in the mixed fusion. If the preparation was correctly produced, the remaining crystals of benzil are uniformly distributed over the entire field of view. The preparation is heated further and the last crystals are seen to melt at 85°C.

The 40% mixture is prepared in a similar way and heated on the hot stage. After the eutectic melts at 78°C, rods remain, this time with various interference colors so that they can be recognized as acetanilide. In this mixture, melting ends at 84°C. The two final melting points, therefore, lie on different sides of the eutectic point. The diagram of this system is shown in Figure 189 on page 247. The eutectic point is at 33% acetanilide.

c) Thermal analysis on the hot bar

Besides the acetanilide:benzil mixtures already prepared (20 and 40% acetanilide), two other mixtures are prepared of 10% and 60% acetanilide. The determination on the hot bar is conducted as follows. The 20% mixture is first melted on an aluminum foil (about 15 x 20 mm; a corner is turned up so that the platelet can be grasped more easily with the forceps) 20°C higher than the expected melting point, then placed on a cold aluminum block. After solidification, most of the sample is scraped off the foil, then crushed and pulverized between two slides. The hot bar is calibrated with benzil (melting point 95°C). Then the powdered mixture is stripped off over the edge of

the slide; three zones can be seen on the hot bar surface. At the high temperature end (completely melted), in the middle, turbid droplets remain and below this the substance is still solid (Figure 125). The limit between the completely and partially melted powder, the clear melt limit, is easily recognized; often in the middle it passes through one of the melt drops. The limit between the turbid droplets and the solid substance is the eutectic temperature, 78°C. At the clear melt limit, one obtains 85°C with a 20% mixture, 90°C with a 10% mixture and 97°C with a 60% mixture. Since the eutectic temperature is also known, the diagrams can be drawn with satisfactory accuracy, the eutectic concentration lying at 33% acetanilide.

During these determinations it is advisable not to clean the hot bar after each test, but to apply several mixtures next to one another in a narrow strip since too much wiping disturbs the temperature equilibrium.

*15. Liquid Crystals

p-Azoxyanisole, melting point 135°C

A sample is melted between slide and coverslip, then transferred to a cold microscope stage. It immediately forms a liquid crystalline layer with irregular extinction. After several seconds, the transformation enters the solid phase, where rods grow quickly. A second transformation follows a few seconds later.

When the hot stage has been heated to 118°C, one observes the transformation of the solid crystals into the liquid crystalline mass. One can verify that the doubly refractive mass occurring above 18°C is really liquid by constantly observing it while pressing on the coverslip. From the change in retardation and extinction with pressure, it is obvious that a liquid exists. The transition at 135°C from liquid crystals to an optically isotropic melt is very sharp. It is, therefore, necessary that the temperature climb very slowly. With the last melt and the first occurrence of liquid crystals, one sees doubly refracting spheres, which flow together upon cooling and soon unite in a liquid crystalline film between slide and coverslip. Between crossed polars, the spheres show a black cross; they are comparable in appearance to crystalline spherulites in which the fine needle crystals are arranged radially. Liquid crystal spheres are made up of elongated molecules oriented perpendicular to the sphere surfaces.

Molecular compound between cholesterol (melting point 148°C) and cetyl alcohol (melting point 51.5°C)

The mixed fusion is allowed to remain completely melted for a few minutes in order to obtain a broader mixing zone. Then the preparation is placed on the hot stage at 50°C. Cholesterol crystallizes first in radiating aggregates, which show partial twisting of the needle units; soon, doubly refractive tiny spheres appear on the cholesterol front; like other spherulites, they show a black cross. Many of these tiny balls come together in a tubelike, elongated structure, part of which shows wormlike motion (strong magnification is frequently an advantage here!). These weak doubly refracting forms, called liquid crystals, convert in places into a solid phase. When the temperature falls below 50°C, cetyl alcohol crystallizes as a doubly

*Translator's note: There is no Section 14 in the German text.

refracting feltlike aggregate. If the preparation is again heated a little after complete solidification, one sees melting of the eutectic between cetyl alcohol and the liquid crystalline compound. The compound now appears, as the drops flow together, as a doubly refracting mass with irregular extinction. During further heating and melting, the liquid nature of the doubly refracting mass is clearly evident because of its movement.

16. Crystal Optics Determination with a Polarizing Microscope

a) Angular measurement on azobenzene

Useful crystals for this measurement are easily produced as follows. Dissolve azobenzene in alcohol on the hot bar (about 70°C) in a small glass tube and use a capillary to place a large drop of the solution on a cold slide. The capillary should be left in the drop until crystallization begins at the edge. Provide nuclei by stirring the preparation for uniform distribution and spreading the solution with a horizontal capillary. There must be enough solution so that total evaporation of the alcohol does not occur during this manipulation. Now allow the preparation to evaporate to a few drops. It is advisable to quickly produce more of the preparation. If the crystals are very small, more solution must be dropped on. As a rule, chiefly rhombs are produced, followed by hexagons and several other forms. Find a good rhomb and measure the pointed angle with the polarizing microscope. For this purpose, first center the objective (or stage) with the two screws provided. Bring the vertex of the angle on the crystal to be measured to the cross hairs; after reading the angular position of the stage, place the second edge parallel to the same cross hair and again note the angular position. The difference on subtraction should be the angle for the azobenzene rhombus, 61°. This rhombus corresponds to the basal pinacoid of a monoclinic crystal.

Save the preparation for future determinations.

b) Determination of the extinction direction

In the above-mentioned preparation of azobenzene, find a six-sided crystal; these crystals usually are flattened and the long edges appear as dark borders. Now focus on the dark edges of such a crystal between crossed polars and turn the crystal to complete darkness (extinction). Remove the analyzer and note that the crystal edge is now parallel to a cross hair; the crystals therefore have straight (parallel) extinction. The rhombuses, on the other hand, lie symmetrical to the cross hairs at extinction, i.e., they have symmetrical extinction. The six-sided crystals are rhombuses, in which the ends are truncated by a pair of edges.

The rodlike urea crystals produced from water also have straight extinctions.

To observe oblique extinctions, one can recrystallize benzamide. Dissolve the benzamide in 96% alcohol in a small dish and heat it on the hot bar to about 80°C. With a capillary, transfer a drop of the solution to a slide and spread the solution with a horizontal capillary until crystallization begins. Allow the alcohol to evaporate and examine the preparation with the polarizing microscope. It is advisable to prepare between five and eight preparations in this way. If the solution does not spread as a thin film the slide must first be cleaned with acetone, carbon tetrachloride, chloroform or

the like.

Benzamide forms monoclinic crystals. As a rule, most of the crystals are rectangular with straight extinction. One also finds a rather larger number of rhombs, which extinguish obliquely. To clear the preparation so that it can be examined easily, allow a drop of xylene or bromobenzene to flow between slide and coverslip. With the analyzer out, place the long edges of the rhombs parallel to the north-south cross hair and read off the angular position. Cross the polars and bring the crystals to extinction. The extinction angle is about 47 or 43° according to the direction in which the crystal was turned. The extinction must be measured several times and averaged. The benzamide preparation can also be used for the next investigation.

c) Twin crystals

Besides rectangles and rhombs in the benzamide preparation, one finds simple twins with a re-entrant angle on one side. The twin plane is a pinacoid; since, however, most of the crystals lie on the prism face, extinction of the twin individuals is not symmetrical with the cross hairs.

Twin lamination can be observed in modification IV of veronal, which was already mentioned because of its polymorphism.

d) Determination of slow and fast vibration directions

In the azobenzene preparation which was used for the investigation of angle measurements, the vibration directions can be located. Find a thin rhomb with gray-blue polarization colors (somewhat modified by the intrinsic color of azobenzene) and bring the shorter diagonal, which has the obtuse angle, into the 45° position, that is, parallel to a line running from the upper left to the lower right. Insert the first-order red gypsum plate in the bodytube compensator slot provided. This changes the polarization color of the rhombs from gray to blue, which signifies an increase in the retardation, and indicates this is the additive position. If the crystal is now rotated 90° , so that the bisectrix of the acute angle is parallel to the vibration direction of the slow component of the gypsum plate, a decrease of first-order red to yellow results (subtractive position).

Urea crystals (optically uniaxial) show the slow vibration direction to their lengths.

e) Pleochroism

Choose about six rhombs in the azobenzene preparations and rotate them with the analyzer out (polarizer in); the colors of the rhombs will be seen to change from orange or brick red (depending on the thickness) to citron yellow. Which of the two vibration directions corresponds to the brick red and citron yellow colors can be decided only if one knows the vibration direction of the polarizer. This is usually determined with the more easily removed analyzer. The analyzer is held close to the eye as one looks towards a reflected highlight. As the analyzer is rotated the reflecting surface appears now brighter, now darker. Since reflected light from a horizontal surface vibrates horizontally, it appears light if the vibration direction of the analyzer is likewise horizontal. This direction should be marked on the analyzer if this has not already been done. The polarizer must vibrate perpendicular to the analyzer if darkness prevails upon insertion of the analyzer.

zer. If, for example, the vibration direction of the polarizer lies north-south the azobenzene crystals can be placed so that they appear citron yellow, corresponding also to a north-south vibration direction for the azobenzene. In this way it can be determined that the *alpha* direction is citron yellow, the *gamma* direction brick red.

f) Determination of the indices of anisotropic crystals

With optically anisotropic crystals, the refractive index for each vibration direction must be individually determined. This is done with the polarizer (without analyzer), and it is important to know the vibration direction, the determination of which was described above. To observe the difference of refractive index as a function of vibration direction, use Nipagin A. Cover the pulverized sample on the slide with a coverslip and allow alcohol to flow in. Heat the preparation moderately so that a residue remains, then transfer the preparation to a cold microscope stage. Prisms grow on cooling. Upon rotation of the stage, these prisms can be seen to vary in contrast; some are, therefore, much higher and some are somewhat lower in refractive index than the surrounding solution.

As a second example, take a benzamide preparation, let a drop of chlorobenzene ($n = 1.53$) flow between the slide and coverslip, and look for rhomb crystals. Rotate the crystals, with the analyzer inserted, first into one then into the other extinction position. In each extinction position remove the analyzer; observe that in one of the two positions the crystals practically disappear. Reproducible results are impossible in this case for two reasons: first, benzamide dissolves slowly in the index liquid; and second, the crystals do not always lie in exactly the same position. One often sees that the index liquid has a slightly higher refractive index while in other cases the crystal has a slightly higher refractive index.

To continue determination of refractive indices for the benzamide crystals, it should now be ascertained that one of the indices lies above 1.66. This can be done by immersing the crystals in α -bromonaphthalene. One then sees clearly that, if the γ' direction is parallel to the vibration direction of polarizer, the crystal has a slightly higher index than the medium (slightly lower above 24°C). Further determinations can be carried out on the rectangular crystals. By immersing them in iodobenzene (1.62), one can see that α' lies only slightly above 1.62.

Look for a thinner crystal with lower interference colors, preferably yellow or red of the first order (most of the crystals are thick and do not show a variety of interference colors, only white of higher order), and determine the vibration directions. Since the crystal elongation is not always the same, the vibration direction parallel to the longitudinal and cross directions varies. The subtraction position, corresponding to the γ -direction of the crystal, is usually easier to recognize than the addition position.

g) Conoscopic observations

As an example of optically uniaxial crystals, use malonamide, in which the stable modification crystallizes tetragonally above 166°C. Melt the substance on the hot bar at 180-185°C. Since malonamide decomposes upon melting, gas bubbles form, so that the corresponding crystal film is strongly dispersed. Examine the resulting crystals between crossed polars and find a crystal which remains dark when the stage is rotated. These crystals lie on a basal pinacoid and show a centered uniaxial interference figure. Orient

the chosen crystal in the center of the field of view, arrange the condenser for highest NA, use the highest NA (at least the 4 mm "high dry") objective and focus on the surface of the crystal. Test whether the centering of the stage is exact, and center if necessary. Then remove the ocular or insert the Bertrand and observe the uniaxial interference figure — a black cross which remains the same with turning, with one or more colored rings in the field of view.

Testing the optical character is now very simple. Insert the first-order red compensator in the provided slot in the 45° position. In this case the quadrants lying in the insertion direction are blue (left above and right below), the two other quadrants yellow. This condition indicates an optically negative crystal.

The flake crystals of iodoform are also optically uniaxial.

As an example of optically biaxial crystals, use mica. Adjust the microscope as above and again remove the ocular or insert the Bertrand lens. As the stage is rotated, one can observe that two black curved brushes close in a cross and then open again. If one turns the preparation from the position of the closed black cross through 45° , one sees the two axial poles of the dark parabolic brushes. The plane connecting the two axial poles is the optic axial plane. With the closed cross, the optic axial plane can be adjusted from front to back or from right to left. The position is recognized immediately from the behavior as the preparation is rotated slightly.

The optical character can again be tested with a first order red compensator. If one observes the interference figure in the 45° position with the optic axial plane running upper left lower right, upon inserting the compensator the region on the concave side of the parabola is blue; mica is therefore optically negative. In an optically positive crystal, the color at that point would be yellow.

Frequently crystals show off-center interference figures. We now again take the crystals of benzamide and focus the conoscopic figure for a right-angled crystal. An optic axis of a biaxial crystal appears. The single dark brush is almost straight as is always the case if the optic axial angle is near 90° . If one rotates the crystal he sees that the optic axial plane is parallel to the longitudinal direction of the rectangle. It lies in the symmetry plane of the monoclinic crystal. The direction perpendicular to the axial plane, that is the cross direction, therefore corresponds to the optic normal β . The emergence of an optic axis can also be demonstrated in fragments of sugar candy.

XII. Explanation of the Identification Tables

It is necessary for everyone who uses the Identification Tables to read the following explanation first. Compiled after many hours of practice and experience, it is a collection of simple but important advice essential in facilitating the use of the constants and indices quoted in the table. Reading this advice helps to ensure good results.

First of all, the substances in the tables were those readily obtainable to us or were readily available through cooperating laboratories. The tables also include a series of new drugs. Most of the substances for the tables were examined in Innsbruck; only the data for some of the sulfonamides were taken from the publications of Dr. Reimers¹, who cooperated by releasing the translations from the original Danish and afterwards determined the necessary eutectic temperatures. The tables will be extended as rapidly as possible in the future.

In the index of substances, the commercial names as well as the chemical symbols and the structural formulas were added as far as they were known. The double bond in the benzene ring is always expressed with double lines; hydrogenated benzene rings, therefore, have only single lines.

Temperature data for the melting point and in the "special characteristics" columns were determined with the standard 4° per minute temperature rise in the vicinity of the melting point (page 5). Our variable resistor is marked for the different temperature ranges (page 3). If a slower or faster temperature increase is desirable for observation of particular reactions, this is noted.

The melting points given in our tables do not always agree completely with those in the literature²⁻⁴. Many times differences of several degrees occur. In some cases the melting points are lower, in others higher than those in the literature. On one hand, this is due to the degree of purity of the substance investigated; on the other hand, to the uniqueness of microscopical melting point determinations which, especially with heat-sensitive substances, use much smaller samples.

In microscopical melting point determination, judgment of the purity of a substance is obviously much more sensitive than it would be in any other sort of melting point determination. It has been shown that even preparations which had been painstakingly purified through repeated recrystallization, sublimation etc. still showed a melting interval of 12°C instead of a sharp melting point upon microscopical observation (page 44). With commercial substances designated "purest" or "for analysis", there is usually a melting interval of two or more degrees. For this reason, one should be careful in a melting point determination to ascertain the "equilibrium condition" with the last crystal residue, *i.e.*, 99% melted, since this comes closest to the exact value (page 11). Moreover, because impurities are seldom distributed uniformly — as seen, microscopically — melting point deviations are found (page 44).

Furthermore, in microscopical studies one must recognize the fact that during long storage many organic substances decompose to some extent in light or in air. Even storage in dark glass in closed bottles cannot completely stop decomposition of many substances. Frequently the decomposition is recognizable as a color change, a pure white substance often becoming brownish or gray. In many cases, dark saturated colors even occur; o-aminophenol, for example, becomes dark red through surface decomposition.

In some substances one can recognize the impurity — whether it was in the substance originally or whether it is due to decomposition — because

after melting crumbly masses remain in the drops. In these cases, the substance must definitely be purified; sublimation is usually sufficient; if it is not, the substance must be recrystallized from solution (page 46).

Many of the substances used for the tables are very pure, so that their melting points agree with the best in the literature. However, we have not always required the greatest possible degree of purity. In general, this depends on the substances. Some, which can be purified only through very time-consuming and troublesome precautions, come in a less pure commercial form and may not even be marked "purest" or "for scientific use". Also, many purified substances "for analysis" contain small quantities of isomers, which are unimportant in chemical analysis but can be recognized by their behavior during melting. In general, we have satisfied ourselves with a degree of purity which corresponds to "purest" or "for analysis". This purity can usually be obtained rapidly for the small amount of substance necessary in microscopical investigations through simple or repeated sublimation (page 11), through filtering with suction (page 47) or through filtering with a small adsorption column (page 47). It is expressly emphasized that our table is intended primarily as an aid in identification and only secondarily as a "melting point table". We do not deny, however, that melting points of very pure products are stated in cases where these were at our disposal. Thus, for example, the values for the fatty acids are from extremely pure preparations; these values were obtained from Dr. Stenhagen, Upsala.

The reproducibility of the constants given in the tables does not suffer relative to purity if one observes the following. As the melting point of the substance, one should take, as was already mentioned, the value determined as the equilibrium temperature of the last crystal to melt. In determining the eutectic temperature, one pays attention not only to individual drops in separate places but also to that temperature at which the chief mass begins to melt; the latter is the eutectic melting point for the two major components. Refractive index of the melt is, on the one hand, generally not so sensitive to small impurities as the melting point; on the other hand, the differences in index between substances with close melting points are usually so large that the reproducibility as well as the difference is easily guaranteed from the outset. It must be emphasized that the purity of substances with low melting points (below about 40°C) is usually especially bad, so that the values in our table may be slightly higher than those of most test substances.

In a series of cases in which the literature melting points are lower than the micromelting points, the reason — at least most of the time — is the uniqueness of the procedure. In many cases, for example, with phenols, amides, urea and urea derivatives, the hot stage method involves much faster heating than in capillary tubes, thus preventing excessive lowering of the melting points due to decomposition. Still, in seeking to identify substances, one must always consider the possibility of a melting point depression due to decomposition during heating. It is advisable to repeat the melting point determination as follows, especially with high-melting substances (over 200°C). Make a microscopical preparation in the usual way, first, however, covering it with a bridge and glass plate; lay the preparation on the heated hot stage only about 15-20°C below the previously determined melting point. In such cases one must, therefore, take even more than the usual care with substances in the Identification Tables with higher melting points.

In special cases, particularly with high melting substances, the melting point can be determined only difficultly or not at all in the customary slide

-coverslip preparation because the substance completely sublimates before melting (this is noted in the table). With some substances, such as caffeine or lophine, the difficulty can be avoided simply by using larger coverslips (for example 21 x 28 mm). If enough substance is available a crystallized film can be used with advantage, for example, with camphor or anthracene.

Crystal films are also often convenient in determining eutectic temperatures. Thus, the eutectics with borneol can be detected without difficulty in a crystal film because the borneol at the edge of the coverslip soon sublimates and the residual crystals concentrate sufficiently. A complete seal may be attained by surrounding the coverslip with a cement, as described on page 16), although this is usually not sufficiently resistant at higher temperatures.

One can also form a seal with a melted second component; Brandstätter, for example, used mannite to enclose a camphor preparation. An effective seal can often be obtained by coating the coverslip edge with silicone gum.

The "silicone chamber" (page 16) offers further sealing possibilities. For identification procedures, use a brass ring with a small central bore (6-7 mm). Coat the entire underside of the ring with semisolid silicon and press a coverslip onto it (12 x 12 mm). More sample must be used than for the usual melting point determination — about as much as for a refractive index determination with a glass powder. The substance is laid in the middle of the slide and the underside of the ring is pressed on the slide around the test substance. A seal is obtained by pressing the ring onto the slide with rotation. The heating space is then closed off with the glass plate. The bridge is not used in this case. The temperatures obtained with this method differ only slightly from those obtained with the usual methods. Up to about 300°C, the values obtained in the silicone chamber are usually slightly lower (1-2°C). For identification purposes, these differences are within the range of error which results from the variation in purity.

Direct sealing in a silicone mass is possible with only a few substances because so many materials dissolve in silicone at high temperatures.

A very simple kind of sealing is obtained with a "salt chamber". Here one uses a eutectic mixture of potassium nitrate and sodium nitrite (55% KNO_3 and 45% NaNO_2) which melts at the eutectic temperature of 141°C. The salt mixture is melted on a slide on the hot bench and a coverslip, coated with the substance to be investigated, is pressed down as near the center as possible. The substance can be applied to the coverslip either through sublimation or simply by pressing on it. In either case, one must be sure that the substance is completely surrounded by the salt mass. The use of the salt chamber presupposes that the substances being investigated are insoluble in the salt mixture. This is the case for many aromatic compounds; and, for example, even small crystals of α -hexachlorocyclohexane surrounded by the salt mass remain unchanged up to their melting point (158°C). If a somewhat larger quantity of the test substance is enclosed in the salt melt, evaporation occurs, mostly at the edge of the substance mass, forming a rather large halo around the residual crystals. It is usually possible, however, without special difficulty to heat these residual crystals to their melting point with concurrent equilibrium (for example, dioxamide and β -hexachlorocyclohexane). Other substances, for example, barbituric acid derivatives, are rather soluble in the salt mixture, so that they disappear before the correct melting temperature is reached. Thus, occasionally, lower melting points may be simulated. The values usually obtained in the salt chamber are several degrees higher because the low wettability of the glass prevents the salt melt from flowing apart into a thin film, hence the preparation is thicker. In

themselves, however, the values have good reproducibility.

Substances for which determination of the melting point in the salt chamber is necessary and possible are referred to in the "special characteristics" column. Besides these determinations, performed with the help of the slide-cover slip preparation, one can also perform melting point or refractive index determinations in Fischer's microcell⁵ or in capillary tubes like those used for the molecular weight determination (page 59). In either case the substance is heated in a sealed tube under the microscope. The microcells are commercially available or they can be easily prepared by compressing the ends of heating glass capillaries. During the melting point determination, the microcell lies in a slit on a metal plate (page 59) for better heat transfer. When the common melting point capillary is used, it is also necessary to lay it in a metal block; this block has a trough in which the capillary tube is placed and a window through which the tube can be observed in transmitted light.

Frequently the tables have two eutectic temperatures for which the following standard compounds are used as second components with the test substance depending on its melting point.

<u>For melting points from:</u>	<u>Standard second components</u>
20 to 100°C	azobenzene, benzil
100 to 120°C	benzil, acetanilide
120 to 140°C	acetanilide, phenacetin
140 to 170°C	phenacetin, benzanilide
170 to 190°C	benzanilide, salophen
190 to 340°C*	salophen, dicyandiamide

*above 240°C a third component, phenolphthalein, is usually used — translator.

A second value in parentheses, for example with sulfathiazole, is the eutectic temperature for an unstable modification. These values are noted only if the behavior must be reckoned with during the customary determination (see page 41). Several lesser eutectic temperatures are given in the tables with a dash on both sides, for example, in the case of thiourea with benzanilide -153-; 153°C here represents only an average value; variations are often observed. If a eutectic melts sluggishly "tr." appears next to the temperature value (page 41). If a low degree of purity, frequently evidenced by discoloration, is observed in a substance during the melting point determination, the substance must be purified before the eutectic or the refractive index is determined (page 47). During eutectic temperature determination, one must also reckon with the possibility of a strongly volatile substance which sublimes completely before the eutectic temperature is reached, so that no depression can be determined. One can provide for this possibility in different ways. One can use the sublimate of the volatile substance which can then be clearly differentiated from the other substance (Table VI, Figure 20), one can use considerably more of the volatile substance, or one can quickly produce a crystal film from whatever mixture he chooses (page 6).

For hydrates, which melt before reaching the eutectic temperature (see page 18), the melting temperature is noted beside the eutectic temperature in parentheses, for example, for citric acid ($+H_2O:58^\circ C$). In this (unusual) case, the sample resolidifies with further heating and later the anhydrous

eutectic melts.

For the refractive index determination (abbrev.: Br. Ind.), the glass powder is usually sought which at the moment of melting of a substance has somewhat lower index than the melt, so that equality of index first appears above the melting temperature.

In exceptional cases, the refractive index was determined below the melting point. This was necessary when a determination over the melting point was impossible because of too great volatility or decomposition. It is only possible when the melt regularly supercools below the melting point without solidifying. If gas bubbles form during melting, the preparation should be quickly cooled with a metal cube before the investigation proceeds any further. When possible, the index was determined with a second glass and even with the next lower refracting one, so that two refractive indices are reported in the tables with the corresponding temperatures. (The temperature coefficient of index can then be calculated — translator.) It is superfluous in many cases to use two glass powders, as we already have shown. (It may not be superfluous when the tables contain additional compounds — translator.)

If the refractive index is found in our tables for only one glass, the determination could probably not be carried out with the next lower glass, usually due to decomposition or to high volatility.

When two glasses, for example 1.5000-1.5101, are given without temperatures, this means that the refractive index lies between those two glass powders immediately after melting and that exact determinations were not possible (usually due to decomposition). These approximate data are most important in identification, as the agreement of values for the substance in question with those for adjacent substances in the tables often shows.

For several unstable substances, the refractive indices were determined with the help of a "mixing procedure" (page 35), i.e., for a 50% mixture with, say, salophen. For all unstable substances which do not allow even an approximate determination between two glasses, we plan to determine the indices by this method and to use these values in the tables, inasmuch as no other final values are possible for the pure substance. For the present, we have made these determinations with a limited number of substances, especially the alkaloids and their salts, and, therefore, the values are not given in a specific column, but rather under "special characteristics". For morphine (melting point 245-225°C), the statement "with salophen $n = 1.5700$ at 156-157°C, 1.5611 at 186-187°C," means: in a mixture with salophen in a 1:1 ratio, one observes equal refractive index with the glass $n = 1.5700$ at 156-157°C, and with the glass 1.5611 at 186-187°C.

For a series of substances, usually high melting, which decompose appreciably on melting, properties of the crystals like shape, angles, extinction and the refractive indices were given in the Identification Tables to compensate for the lack of constants determinable for the stable substances. The refractive index liquids used are listed in the small table below; the gap between α -bromonaphthalene ($n = 1.66$) and methylene iodide ($n = 1.74$) is annoyingly noticeable; in general, however, the index liquids used are sufficient for identification.

Refractive index liquids

Alcohol	1.37	Nitrobenzene	1.55
Chloroform	1.44	Bromobenzene	1.56
Carbon tetrachloride	1.46	<i>o</i> -Toluidine	1.57
Castor oil	1.48	Aniline	1.59
Xylene (or benzene)	1.50	Iodobenzene	1.62
Cedar oil	1.51	α -Chloronaphthalene	1.64
Monochlorobenzene	1.53	α -Bromonaphthalene	1.66
Clove oil	1.54	Methylene iodide	1.74

Compiling the last column, "special characteristics", is difficult. The processes before, during and after melting are so characteristic of many substances that the expert often recognizes the substance before it reaches the melting point. The entire variety of these phenomena cannot, however, be condensed briefly in a table. The description must be similar to a personal description of a journey, in which it is impossible to give all of the details, but which nevertheless is helpful to someone who must recognize the route. The easier the description, the broader and more comprehensively it can be written. Then, too, even though the rate of heating is always the same, the appearances before melting are not always identical. Even the same preparation appears different in different places.

From the total number of characteristics, we have chosen several identifying ones which usually serve as the most important clues to identification. The intrinsic color, if one is present, is noted first. No mention of color means that the substance is colorless or has only minimum color. If a color is due to decomposition, this is noted, as was mentioned above, and next to it the best purification method is given to quickly supply the small amount of pure substance necessary for a microscopical investigation.

In many cases, simple sublimation suffices; this is best done on the hot bar, as were all of the sublimation examples in the tables (page 21). If one uses the hot stage for sublimation, one should choose temperatures 5-10°C below the stated values.

To prepare a sublimate, place a small glass dish or a slide bearing a little heap of the substance on the hot bar, lay a glass or metal ring on it (chromed or nicked brass ring about 17-18 mm in diameter and 2 mm high) and cover it with a coverslip or a slide. To produce larger crystals more suitable for the melting point determination, it is better to first heat the coverslip, since a thick layer of small crystals usually forms on cold coverslips due to rapid condensation. This hinders observation of the processes during warming. The amount of sublimate obtained depends on the specific behavior of the substance, the temperature, the permanence of the sublimate, the temperature difference etc. (page 12). For the melting point determination, it is necessary to use a large amount of substance only if the material is especially volatile (for example, *p*-aminophenol).

For the refractive index of the melt, somewhat more substance is necessary; it is simplest to use the results of two sublimations together. To do this, one first collects a sublimate on a slide, then sprinkles glass powder on it and covers it with a coverslip also coated with sublimate.

If the substance has a striking odor, "odor" is noted, usually without further specification.

The above-mentioned observations are followed by notations of characteristic sublimation appearances during heating. The accompanying temperature is that at which the first sublimate appears during heating at the usual

rate, chosen to correspond to the expected melting point. So that the reader can understand the notations used here, which characterize the habit of crystals obtained either through sublimation or in equilibrium, the following explanations should be given.

Grains are crystals whose diameter is approximately the same in all directions. They can be cubes or polyhedra, but frequently appear entirely irregular. Most of these grains are very different. Their larger representatives are often well-developed crystals; the smallest grains are so tiny that it is difficult to distinguish them from droplets.

By flakes, we mean thin crystals greatly developed in one plane. Their outline can be easily discerned; they are found in regular geometric figures — squares, rectangles, triangles, trapezoids and rhombs — or in entirely irregular forms. Thicker formations are also spoken of as tablets.

Prisms are crystals whose diameter in one direction is two or three times that in the two other directions. They are, therefore, distinguished from the grains by greater growth in one direction. Very long prisms are called rods.

Crystals are called needles if they are much longer than they are thick. They can be short or very long. Many needles are so thin that they appear only as dark streaks in transmitted light and may be very ribbonlike. Wide needles can taper to a very fine point or can show a blunt termination. Many are straight, others curved.

Several different crystal habits may be shown. Grainlike crystals frequently form at equilibrium; upon cooling, the crystals often grow in one direction, so that the grains quickly turn into rods. Besides the typical forms and the transitions between them, very different forms can also be found in the microscopical preparation; these sometimes can be compared to anything at all; for example, many crystal forms can reasonably be called swords.

The crystals are not always separate but often occur in complexes called crystal aggregates. Most grains can usually be distinguished very easily according to the substance or to the mode of origin. One speaks of large- or fine-grained aggregates. Flakes frequently lie partially over one another, forming chainlike or rosettelike aggregates. Crystal rods or needles form clustered, star-shaped, sheaflike or radiating aggregates.

Besides the crystallized sublimate, there are always many distinct liquid drops. Their number usually increases greatly near the melting point, a fact which can be helpful in determining the melting point. Since the formation of condensation drops is an almost obligatory phenomenon, it was omitted in "special characteristics". An exception is the substance for which no crystallized sublimate forms; in this case, one finds the remark, for example, "droplets at 120°C".

Also given under "special characteristics" are the equilibrium melting points of all substances which can be obtained without difficulty and the form of the corresponding crystals.

The phrase "sinkt ab" means that the temperature must be allowed to decrease in order to maintain equilibrium between the crystal residue and the melt. Upon reproducing the equilibrium, one finds a steadily lower temperature. This phenomenon is manifested in substances which melt with decomposition (see page 18).

After complete melting of a preparation, it can often be observed during cooling that the melt is strongly "supercooled"; with higher-melting substances, the expression "solidified to a glass" is used because the viscosity upon strong supercooling is so great that the consistency is "solid". One

can often see cracks form in the isotropic and structureless "glass". Many melts which solidify to a glass can be transformed into crystals; for these substances, one finds next to the remark "solidifies to a glass" the note: "Crystallizes on heating". The crystal habit of the solidification product is stated for spontaneous crystallization of the melt.

For a hydrate, turbidity upon warming is noted together with the corresponding temperature. In addition, almost all of the hydrates can be recognized as such by melting in paraffin oil; several hydrates melt congruently in paraffin oil (for example, oxalic acid dihydrate), but many melt incongruently with the separation of lower hydrate or anhydrous crystals (page 18). The temperatures noted for these processes are generally very characteristic, so that they are valuable in identification. Incongruent melting is usually very sluggish, which is connected with the temperature difference of the transition point and of the final congruent, however unstable, melting point (page 96). For this reason, an interval is usually given. However, even the congruent melting points are not always sharp. This is usually due to the fact that free water is present due to absorption or to capillary action. Steam usually first escapes from a paraffin preparation at 105-110°C, but often first much later; in cases where it is doubtful whether a hydrate exists at all, it is advisable to heat the preparation to at least 130°C.

The melting point in paraffin oil is very easily carried out and, since they often yield characteristic results, they should always be performed when they are noted. Several hydrate crystals are crushed on a slide, mixed with a few drops of paraffin oil, then covered with a coverslip. Clear fragments should be chosen for observation. If the hydrate melts congruently in oil, clear drops form which look like oil drops in water. With incongruent melting, the rather rapid crystallization of the anhydrous substance (or lower hydrate) can be observed. For determination of the exact transition point, see page 96).

At higher temperatures, the deposit of paraffin droplets on the bridge or on the glass plate can markedly decrease visibility. It is usually unnecessary, however, to repeatedly wipe off the glass plate, because, after a certain time, the droplets on the glass flow into a film, after which sight is again clear. If this does not occur, the underside of the glass plate can be coated with a thin layer of paraffin oil.

Polymorphism is noted only with the remark "dimorphism" or "polymorphism". In most of these cases, the melting point of the unstable modification is also given. It should be pointed out, however, that in many cases of "polymorphism" unstable forms can be produced by special procedures as described in the text. When a transition point is given for an enantiotropic substance, this can usually be determined only if one places a preparation containing both modifications on the hot stage at a temperature near the transition point.

If a substance in the usual commercial or laboratory preparation is in the unstable modification, which frequently happens, this modification is indicated in italics, for example "2-phenyl quinoline-4-carboxylic acid, II, 213°C; *III*, 196°C". All commercial preparations of this acid by any other name are modification II.

Diethyl barbituric acid (vernonal) has four modifications (so far — translator): I, 190°C, II, 183°C, III, 181°C, IV, 176°C. All substances called veronal, as well as pharmaceutical preparations under other names, are modification II, 183°C. In melting point determinations both in capillary tubes and under the microscope, the unstable modification transforms to the stable one, so that in the usual procedure with a crystal powder only the

melting point of the stable form can be observed. It is possible, however, that crystals of modification IV (176°C) may melt at 176°C as determined with both the hot stage and capillary tubes. With atophan, on the other hand, modification II is so stable that normally only its melting point, 213°C, is observed rather than I, 218°C. In the tables, the melting point given in the first column is that observed with the usual working procedure, so that with veronal it is for the stable modification, 190°C, and with atophan it is that of modification II, 213°C, not 218°C. Only when two or more melting points occur with the usual working conditions are these also given in the first column in parentheses.

For high-melting substances, hints are included in the "special characteristics" for the refractive index of the melt determination (Br. Ind.) because these often melt with decomposition, as is evidenced by a brown color, gas evolution or both. It has been shown in many such cases that an approximate determination, though difficult, is possible and can prove very useful for comparison with neighboring substances. In several cases the temperature interval at which the refractive index of the melt and the glass powder is similar is greater than usual, that is, more than 1-2°C. In other cases, it can only be stated that the index of the melt immediately after melting is lower than that of a certain glass, but higher than that of the next glass. Such characteristics can often be determined, together with very strong gas evolution due to decomposition. Since these processes, together with refractive index determination, are always reproducible when standardized working methods are used, they are often very useful in identification as one can imagine due to the similarity of melting point data for some neighboring substances.

Of course, the refractive index determined on the melt is not as valuable as a characteristic constant for the substance. As far as such measurements can be reproduced, however, the results were included as helpful values in the tables. Since the edge of the drop usually decomposes more quickly than the center, it is advisable in all cases to observe glass particles which lie as close as possible to the center of the large drops.

In several cases, the refractive index changes rapidly during decomposition, so that a substance which at the moment of melting has a lower index than a certain glass powder may be higher after one or two minutes. In such cases this has been noted. The numbers appearing with the refractive index data, then, usually refer to the melt after it has decomposed for one to two minutes.

In determining the refractive index of substances in which the melt develops bubbles as a result of decomposition, one must proceed as follows. Above all, the preparation with the glass powder must be completely melted as quickly as possible. This is accomplished by placing the preparation on the hot stage at a temperature 8-10°C or more above the melting point. The glass cover plate should always be used, but it is often more convenient to omit the bridge during very strong bubble formation because the bubbles can be dispersed from the field of view by pressing on the coverslip with a lancet needle. One must lift off the cover plate quickly; the bridge hinders this. If, even through the described method of operation, the temperature measurement is impaired, it is frequently sufficient to estimate the refractive index difference between the glass powder and the melt.

The occurrence of many bubbles in a melt is not always due to decomposition. Often they are only air bubbles which have not escaped. This happens if the substance is a fine powder and the resulting melt is very viscous, for example with sugar. This is noted for the substances concerned.

If the refractive indices are given for substances which are usually hydrates in the original preparation, there are two ways in which damage by the release of water can be limited. The simplest is to allow the substance to dry on the hot bench at about 110-130°C for some time before determining the refractive index. If, however, one works as quickly as possible, one can mix the hydrate with the glass powder and lay the preparation on the hot stage during warming without a coverslip. Thus, the resulting bubbles can escape; a coverslip is first placed on the preparation at about 160°C. One can also use a procedure like that described above for an unstable substance and place the preparation with a coverslip on the heated hot stage above the melting point. The coverslip can be placed on the preparation only when gas evolution has ceased.

Test procedure: During identification of organic substances, the following methods should be used. First, one must decide whether a striking odor is present and whether any color of the melt is characteristic or is due to decomposition or impurities. In the case of impurities, substances usually show mixed colors. (The taste can also be characteristic.) (Good God! — translator.) In each case, one first determines the melting point on the hot bar. If none is available, it is advisable to first determine the melting point approximately with a rather rapid temperature rise. If one suspects secondary discoloration, he should attempt to sublime the preparation in the manner described on page 13. Choose a sublimation temperature on the hot bar about 10-20°C below the melting point. On the slide, of course, the temperature is a few degrees lower. We should emphasize that this sublimation temperature is not always the best since the degree of volatility is also important. If, with a substance showing mixed colors and a very unsharp melting point, one obtains only droplets upon sublimation, it is better to purify by absorption (page 47) or by recrystallization (page 47). All further investigations are carried out with only the purified substance.

The melting point of the test substance is next determined at a normal heating rate in the hot stage microscope in order to clearly observe the special characteristics. The resistance can be properly adjusted only after the initial test, be it on the hot bar or by approximate determination on the hot stage. Naturally, for substances of mixed colors, one immediately uses the crystals obtained through sublimation.

At the same time, determine the eutectic temperatures with the standard second components. Immediately after determining the eutectic temperatures (page 39), compare the three results — the melting point and the two eutectic temperatures — with those in the tables. This procedure is best because the melting points often shows greater deviations than the eutectic temperatures. Frequently, the diagnosis is given from just these three values but one should never neglect to confirm the data by determining the refractive index of the melt. Only if this value agrees with the others for a substance is the result certain. It is also possible that the test substance is not included in the tables, but has a melting point and eutectic temperatures near those of a substance that is in the tables. Since, however, it is hardly likely that a substance missing from the tables will show, besides the other three values, a corresponding refractive index, the refractive index is included with the other constants as a certain means of identification.

For substances melting above 240°C, the column for refractive indices of the melt was replaced by the temperature of a third eutectic, with phenolphthalein. This was only done, however, in cases where decomposition of the melt made refractive index determination impossible. With the other substances which melted above 240°C, the refractive index values are given in

the "special characteristics" column.

For crystal optics investigations, it is necessary to produce good crystals through sublimation or recrystallization. The kind of crystals used in doubtful cases is noted in the "special characteristics" column. The temperatures given for substances which sublime are for the hot bar; with the hot stage, 5-10°C lower temperatures should be used. Crystallization from a solvent is usually carried out as described in the examples or in the following way. One places a small amount of the substance on a slide, lays the preparation at the cool end of the hot bar and mixes it with one or more drops of the solvent. One then slides the preparation to higher temperatures, using alcohol at about 90°C, water at about 110°C. After a few seconds, one transfers the preparation, still without a coverslip, cautiously to a cold support and allows the solvent to partially evaporate. The residual solution, mostly colored with contaminating substances, is soaked up with filter paper and the preparation is dried by pressing it with hardened filter paper.

The conoscopic behavior of the test substances is referred to in only a few cases, since achieving an identification should not depend on the use of a "polarizing microscope". This information can, however, facilitate the diagnosis in doubtful cases. The refractive indices for anisotropic crystals can also be determined with no further adjustment with a fixed object stage — assuming that ocular cross hairs are present — if the crystals are placed parallel to the cross hairs by hand. The sign of elongation can also be determined with a fixed stage if the preparation is carefully placed in the 45° position by hand.

The use of a home-made rotating stage is mentioned on page 6 ; it is a metal disc, furnished with a central bore, which is placed in the hot stage and can rotate in it. The mechanical slide-mover, together with its accompanying mount, must naturally be removed first. The 2-mm thick metal disc must fit easily in the outer frame of the hot stage ring so that the centering is preserved as well as possible during turning. By means of two metal knobs and/or screw heads, which are placed on the edge of the disc at a 90° angle, each turn can be executed easily. The decentering which thus occurs is usually so slight that the crystal remains in focus and the optical properties can be observed without delaying adjustments. If a low temperature of matching refractive index is detected, the temperature effect of the metal disc must next be tested on the stage with a standard.

With the rotating metal disc, the refractive indices of anisotropic crystals as well as their vibration directions can be determined almost as easily as on a microscope with a rotating stage.

Conoscopic investigations cannot be carried out on the hot stage because of the thickness of the stage.

ABBREVIATIONS IN THE TABLES

Aggr.	aggregate	Mod.	modification
Alk.	alcohol	Mol. Vbg.	molecular compound
anisotr.	optically anisotropic	monotr.	monotropic
A. W.	axial angle	neg.	negative
Best.	determination	opt.	optical
Br. Ind.	refractive index	Paraf.	paraffin oil
d.	the, through	pos.	positive
doppelbr.	doubly refracting	Prod.	product
enant.	enatiotropic	R. T.	room temperature
eutekt.	eutectic	Schmz.	melt
ev.	eventual	Spharol.	spherulites, spherulitic
F.	melting point	Strassenpfl.	street pavement
Flücht.	volatile, volatility	Subl.	sublimate, to sublime
Flüss.	liquid, liquidity	teilw.	partially
Gasblasenblg.	gas bubble formation	tr.	sluggish
Gleichgew.	equilibrium	u.	and
homog.	congruent	unt.	under
Hydr.	hydrate	Up.	transition point
inhomog.	incongruent	Vbg.	compound
isotr.	optically	Verfl.	evaporation
K. G.	crystallization velocity (CV)	vielfl.	polyhedral
l. lösl.	readily soluble	Zers.	decomposition
Luftbl.	air bubbles		

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m-Oxybenzaldehyd	104	Pentachloranilin	232
p-Oxybenzaldehyd	117	Pentachlorphenol	190
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m-Oxybenzoesäure	202	Pentadecansäure	62,5
p-Oxybenzoesäure	214	Pentaerythrit	256
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Phenol- β -d-galaktosid+ $\frac{1}{2}$ H ₂ O	155	Phthalanil	209
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Phenol- β -d-glucosid+2H ₂ O	173	Phthalimid	235
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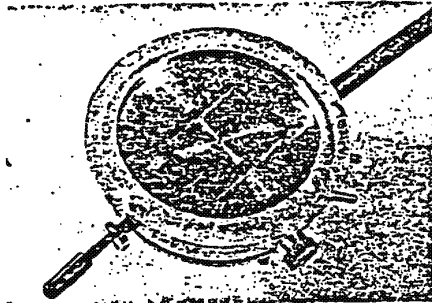


Figure 1 Kofler hot stage with slide manipulator (thermometer model).

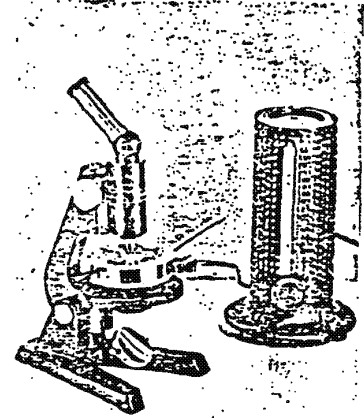


Figure 2 Kofler hot stage microscope.

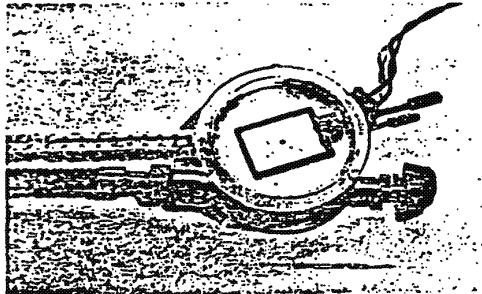


Figure 3 Kofler cold stage.

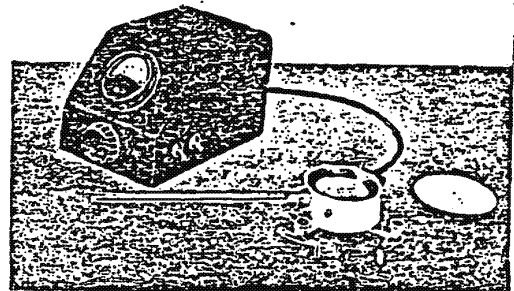


Figure 4 Kofler high temperature stage.

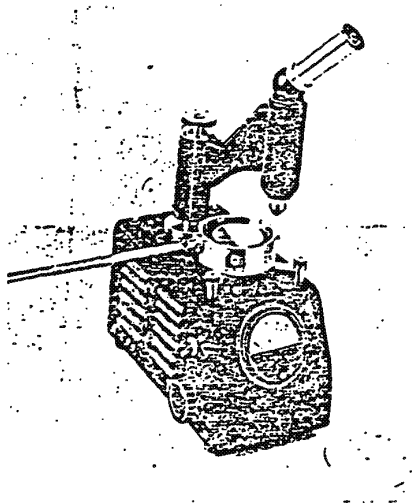


Figure 5 Kofler hot stage microscope with swing-out bodytube.

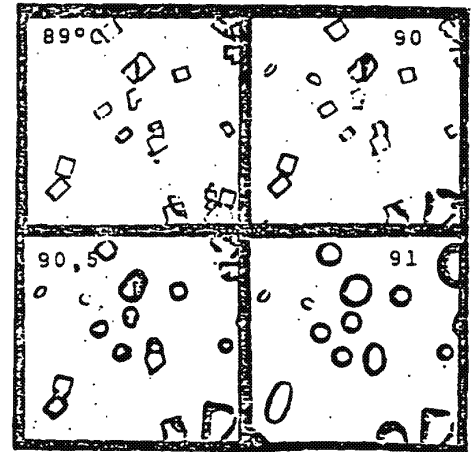


Figure 6 Continuous heating melting point for Anaesthesin.



Figure 7 Equilibrium melting point for cardiazol.

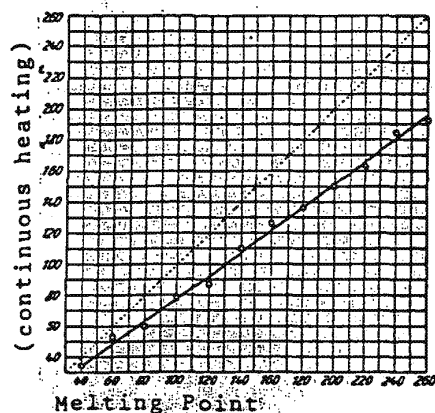


Figure 9 Sublimation temperatures.



Figure 10 Growth spirals for Borneol.



Figure 11 Layer growth for Naphthalin.

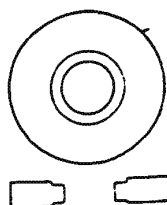
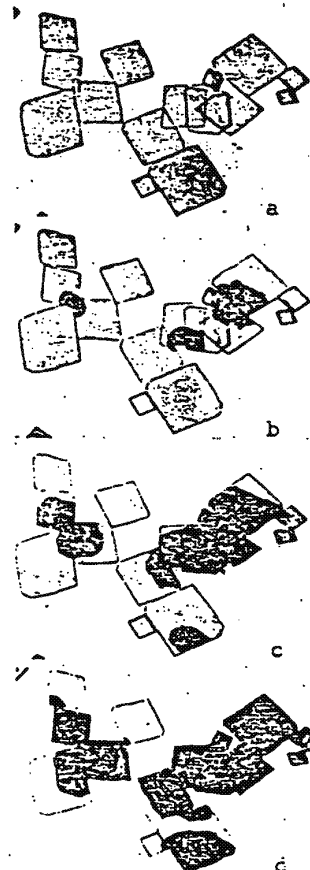


Figure 12 Silicon cell (actual size).

Figure 13 Dehydration of phloroglucinol hydrate.



Figure 8 Crystalline sublimate and condensed droplets; the growing crystals nucleate some droplets and grow at the expense of the more volatile droplets thru the vapor phase.



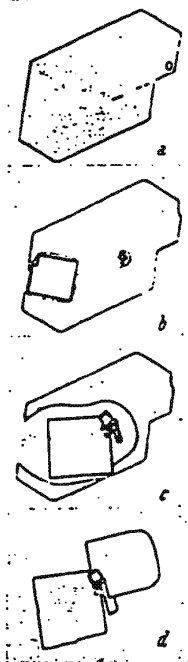


Figure 14 Incongruent melting of $\text{NaBr} \cdot 2\text{H}_2\text{O}$ with crystallization of anhydrous NaBr.

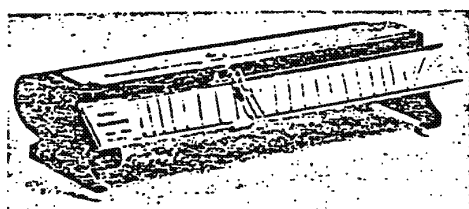


Figure 15 Kofler hot bar (50° - 260°).

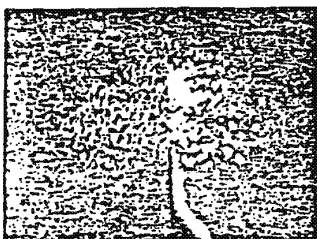


Figure 16 Melting point with the hot bar; the point shows the demarcation between crystals and melt.

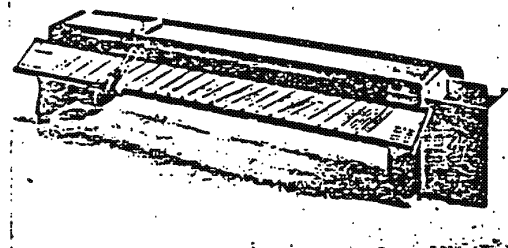


Figure 17 Hot bar for lower temperatures (10° - 210°).

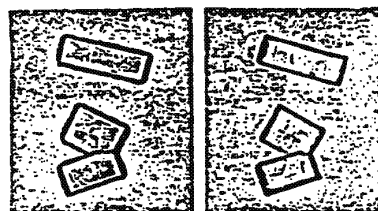


Figure 18 Crystals with higher refractive index than the liquid:
a) on focusing higher,
b) on focusing lower.

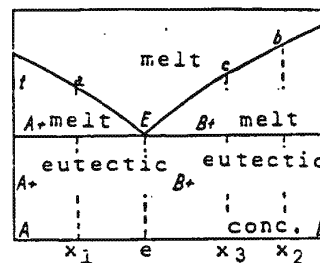


Figure 19 Simple eutectic composition diagram.

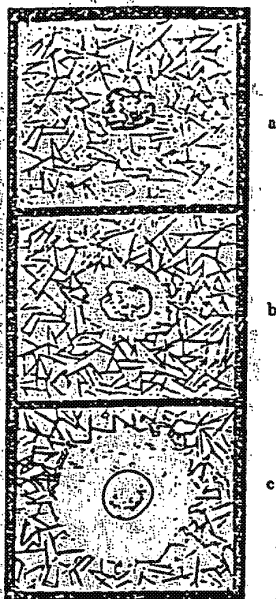


Figure 20 "Mixed melting point" between two different substances.

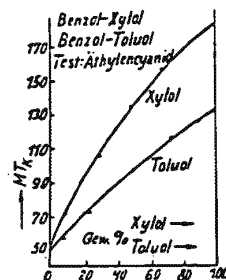


Figure 22 CST for benzene: xylene and benzene: toluene with ethylene cyanide (after Fischer).

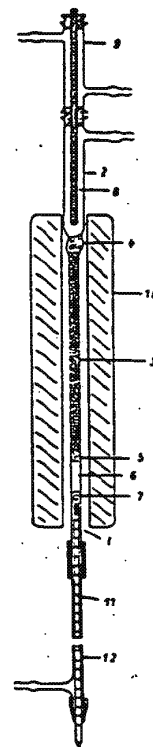


Figure 21 Apparatus for adsorption-sublimation.

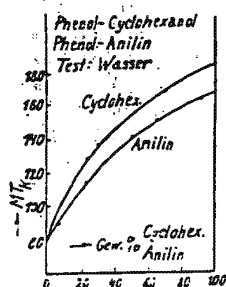


Figure 23 CST for phenol: cyclohexanol and phenol: aniline with water (after Fischer).

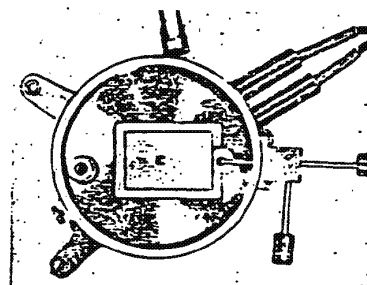


Figure 24 Hot stage with metal block for molecular weight determination.

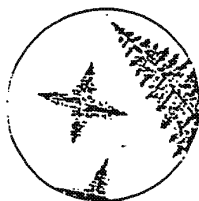


Figure 25 First crystals of camphor during molecular weight determination.

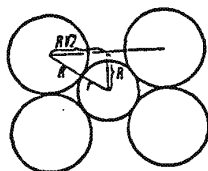


Figure 26 Coordination number of 8.

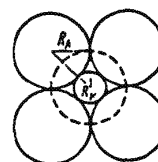


Figure 27 Coordination number of 6.

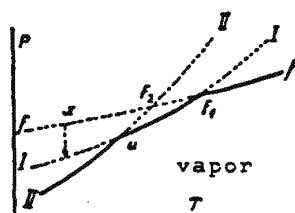


Figure 28 Vapor pressures curves for Enantiotropy.

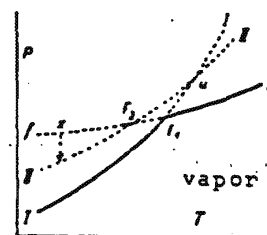


Figure 29 Vapor pressures curves for Monotropy.

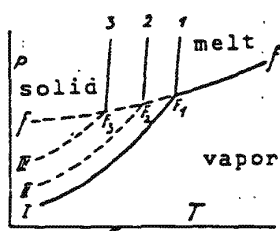


Figure 30 Vapor pressure and melting point curves for different polymorphs.

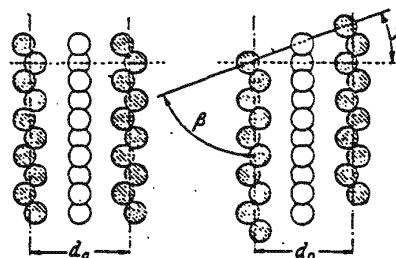


Figure 32 Model for crystal lattice showing a possible change due to polymorphism.

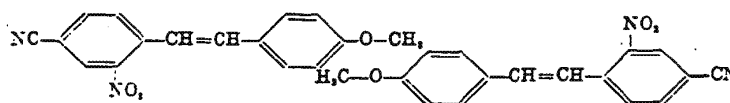


Figure 31 p-Cyan-o-nitro-p'-methoxystilbene.

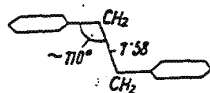


Figure 33 Dibenzyl molecule.



Figure 37 The central spherulites transform, on warming, the unstable form.

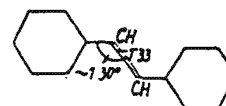


Figure 34 Stilbene molecule.



Figure 35 Three polymorphic forms of Luminal during crystallization from the melt.



Figure 36 Two polymorphs of Benzalazine.

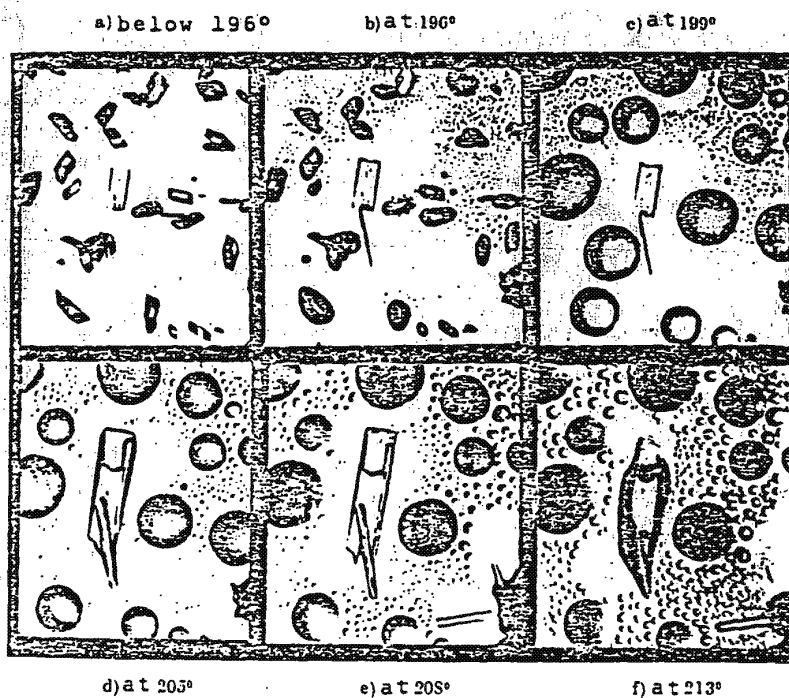


Figure 38 Atophane:
a) oblique-angled unstable form III (np. 196°) and a right-angled crystal of form II (np. 213°); b) and c) melting of form III; d) and e) growth of form II thru sublimation and f) melting of form II.

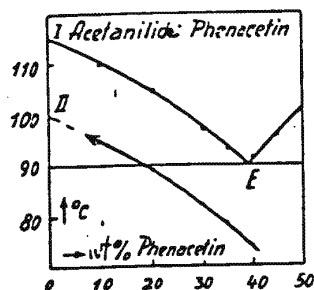


Figure 39 Melting point for acetanilide II at 100° obtained by extrapolation of melting point curve in 2-component system with phenacetin.

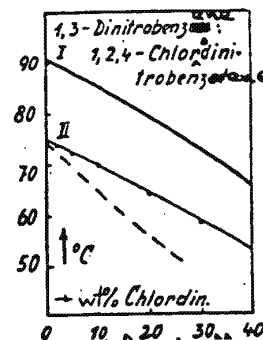


Figure 40 Melting point for 1,3-Dinitrophenol II by extrapolation of curve in a two component system with 1,2,4-chlorodinitrobenzene.

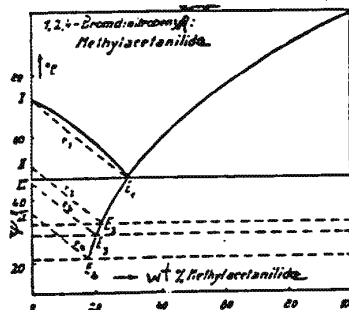


Figure 41 Indirect melting point determination by use of parallel melting point curves from eutectic points, r_1 , r_2 , r_3 , r_4 , for 1-Brom-2,4-dinitrobenzene: Methylacetanilide.

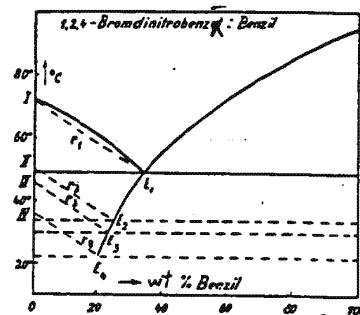


Figure 42 Similarly for 1-Brom-2,4-dinitrobenzene: Benzil.

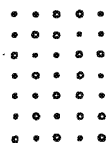


Figure 43 Statistical distribution in mixed crystals.

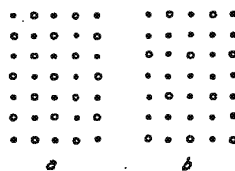


Figure 44 Ordered distribution in mixed crystals.



Figure 45 Interstitial solid solution.

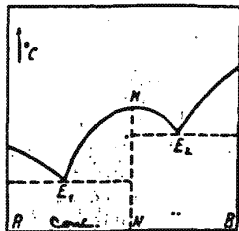


Figure 46 Schematic addition compound composition diagram.

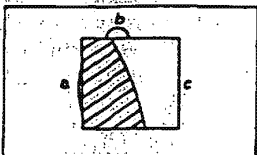


Figure 48 Mixed fusion (contact prep).



Figure 50 Diphanyl:
acetanilide mixed fusion
a) below the eutectic
temperature
b) above the eutectic
temperature.

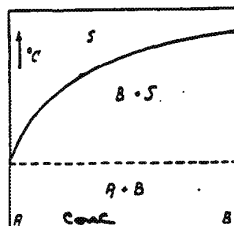


Figure 51 Phase regions
in portion of a eutectic
system.

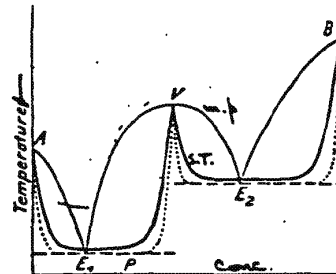


Figure 47 Sintering
temperature (S.T.)
according to A. Stock.

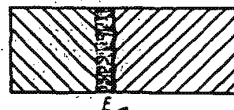
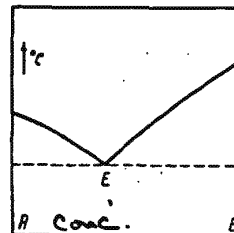


Figure 49 Schematic
simple eutectic diagram
and corresponding mixed
fusion during heating to
just above the eutectic
melting point.

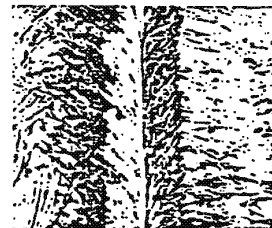


Figure 52 Phenanthrene:
2,4-dinitrophenol mixed
fusion with molecular
addition compound.

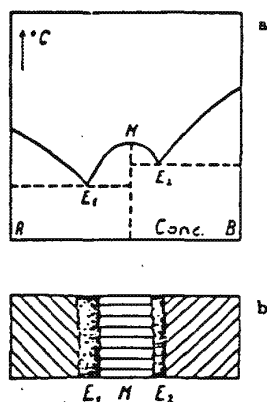


Figure 53 Schematic molecular addition compound system and corresponding mixed fusion during heating just above E_2 .

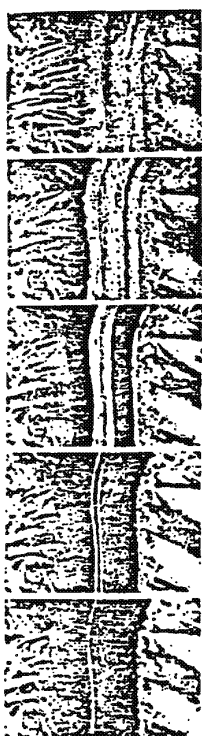


Figure 56 Two molecular addition compounds of decamethylene dicarboxylic acid: nicotinic acid amide.



Figure 57 Two molecular addition compounds of antipyrone: hydroquinone.

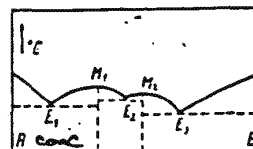


Figure 55 Composition diagram for nicotinic acid amide: decamethylene dicarboxylic acid showing two molecular addition compounds.



Figure 54 2,4-Dinitrotoluene: Naphthalene mixed fusion with 1:1 molecular addition compound.

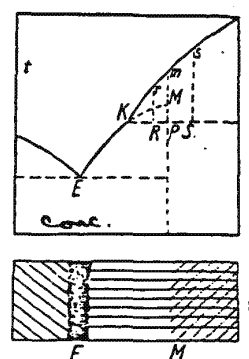


Figure 58 Schematic peritectic system showing an incongruently melting addition compound and the corresponding mixed fusion just above the eutectic melting point.

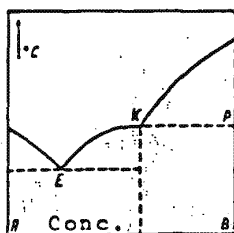


Figure 59 The limiting case; between a congruently and an incongruently melting addition compound; the peritectic point K is at the melting point of the addition compound.

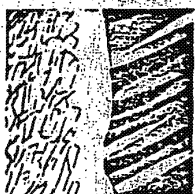


Figure 61 Immiscible liquid phases in the system acetamide: benzil.

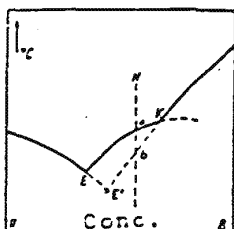


Figure 63 Unstable equilibrium in the absence of nucleation of the stable addition compound.

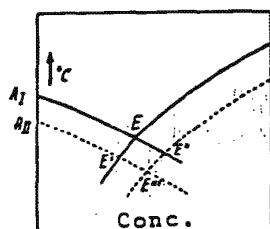


Figure 62 Eutectic systems between stable and unstable polymorphs.

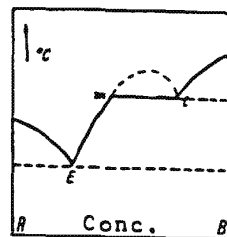


Figure 60 Schematic composition diagram for a binary system melting to give two immiscible liquids s_1 , s_2 and the corresponding mixed fusion at a temperature just above mn .

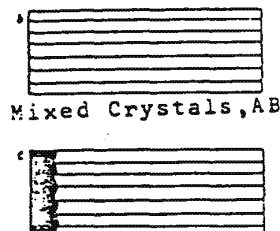
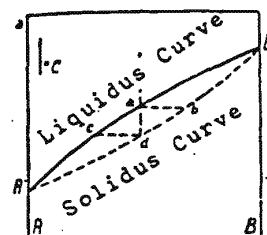
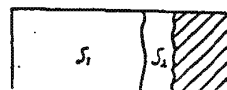
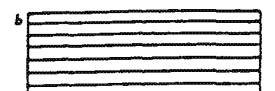
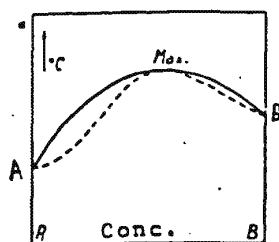


Figure 64 Roozeboom Type I isomorphism; schematic composition diagram and corresponding mixed fusion at a temperature just above A .



Mixed Crystals, AB

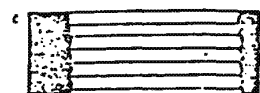


Figure 65 Roozeboom Type II isomorphism; schematic composition diagram and corresponding mixed fusion at a temperature just above B.

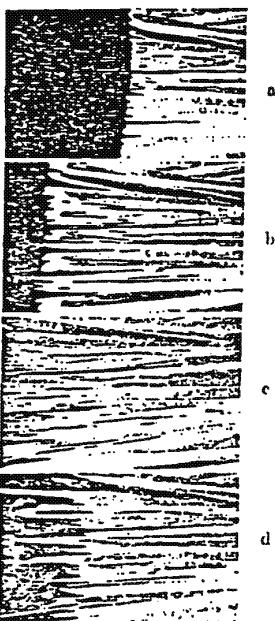
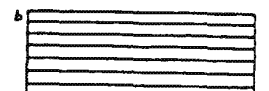
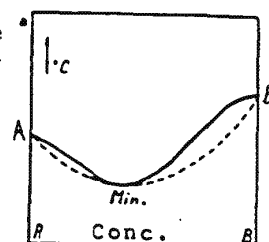
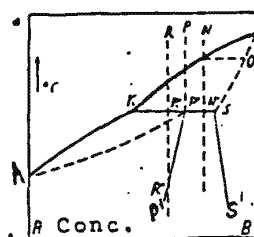
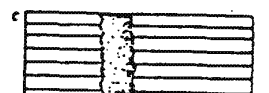


Figure 67 Type I Roozeboom system for propyl-3-methylbutyl: m-butyl-propylbarbituric acid.

Figure 66 Roozeboom Type III isomorphism; schematic composition diagram and corresponding mixed fusion at a temperature just above min.



Mixed Crystals, AB



+A Mixed +B Mixed Crystals Crystals

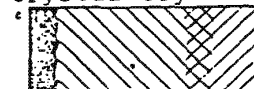


Figure 69 Schematic Partial solid solubility composition diagram (Type IV, Roozeboom) with the corresponding mixed fusion at a temperature just above A.

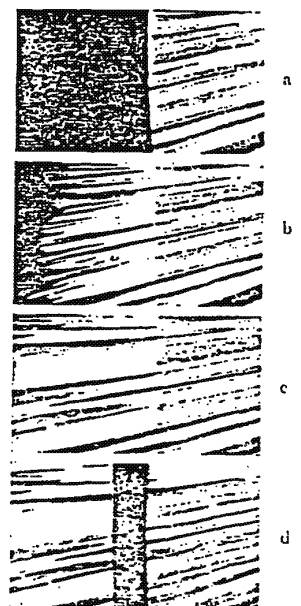


Figure 68 Type III Roozeboom system for allylbutyl: dipropylbarbituric acid.



Figure 70 Mixed fusion for dibenzyl: stilbene showing type IV Roozeboom.



Figure 71 Peritectic reaction in the system dibenzyl: stilbene.

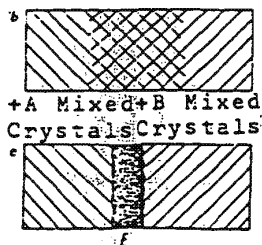
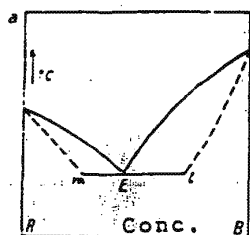


Figure 72 Schematic Partial solid solubility composition diagram (Roozeboom Type V) with the corresponding mixed fusion at a temperature just above m-E-1.

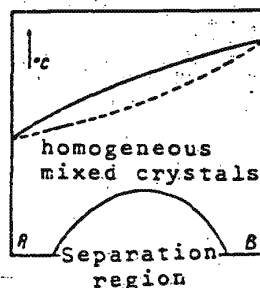


Figure 73 Separation of mixed crystals at low temperatures in a solid solution system.

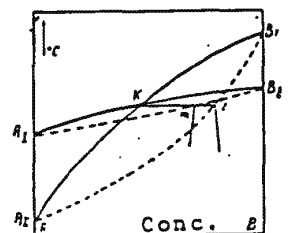
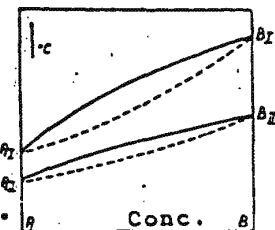


Figure 75 Crossed isodimorphism; Type IV Roozeboom.

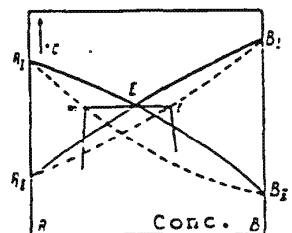


Figure 76 Crossed isodimorphism; Type V Roozeboom.

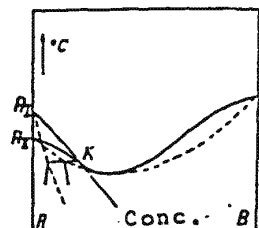


Figure 77 Isodimorphism of dibenzyl: azobenzene.

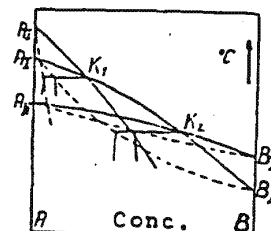


Figure 78 Three intersecting mixed crystal series.

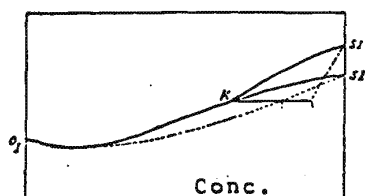


Figure 79 Isomorphism in the system diphenylene oxide: diphenylene sulfide.

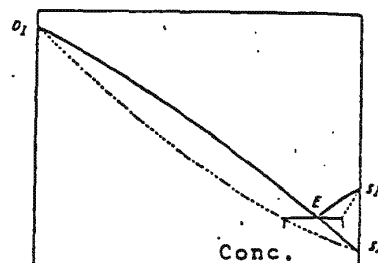


Figure 80 Isomorphism in the system dianisyl oxide: dianisyl sulfide.

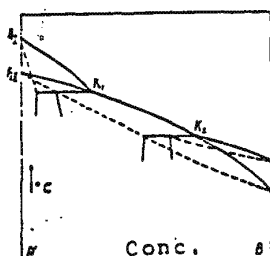


Figure 81 Stabilized intermediate phase with two peritectic points.

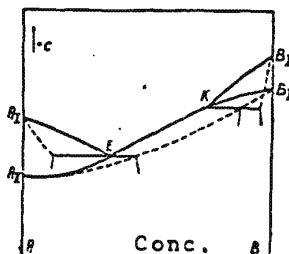


Figure 82 Stabilized intermediate phase with one eutectic and one peritectic.

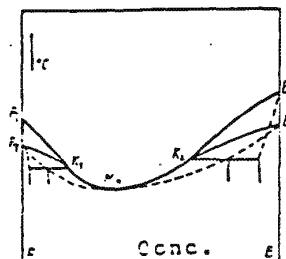


Figure 83 Stabilized intermediate phase with a minimum.

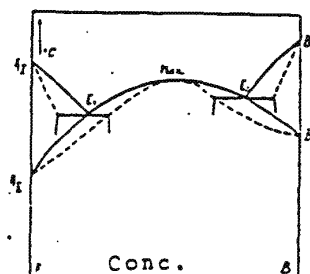


Figure 84 Stabilized intermediate phase with a maximum.

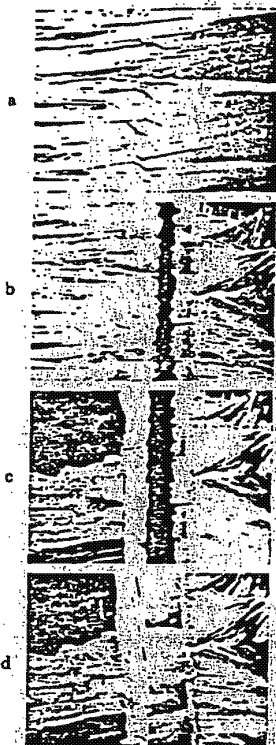


Figure 85 Mixed fusion between allylisopropyl-: ethylpropylbarbituric acid.

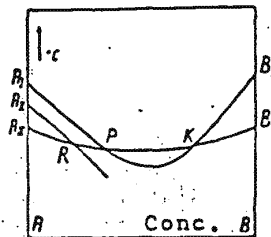


Figure 86 Composition diagram corresponding to Figure 85.

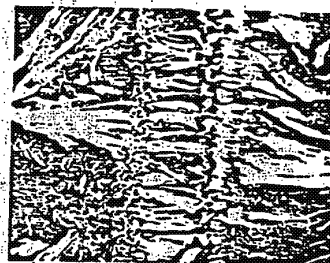


Figure 87 The stabilized intermediate phase for palmitic-: stearic acid.

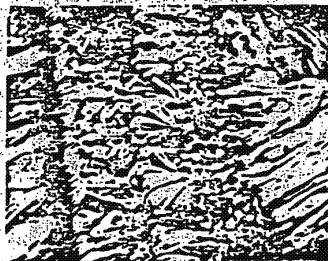


Figure 88 Two stabilized intermediate phases for pentadecane-: stearic acid.



Figure 89 Homogeneous mixed crystals of NaNO_3 : KNO_3 (80% NaNO_3).

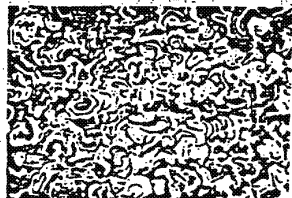


Figure 90 Rearrangement of rings and spirals during melting of NaNO_3 : KNO_3 .



Figure 91 Primary dendrites on slow cooling.



Figure 92 Changes in dendrites on further cooling.



Figure 93 Zoned crystals.



Figure 94 Beginning of melting of zoned crystals with formation of spirals.



Figure 95 Progress of melting.

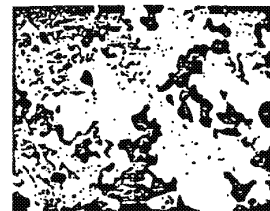


Figure 96 Homogenization after four hours heating at 220-225°.

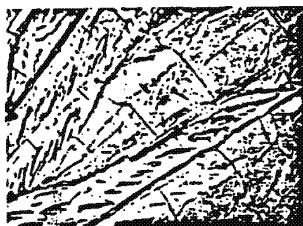


Figure 97 Uniform mixed crystals of phenanthrene:anthracene.

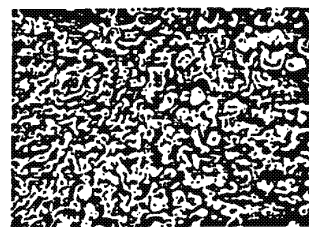


Figure 98 Rearrangement of spirals and rings during melting.

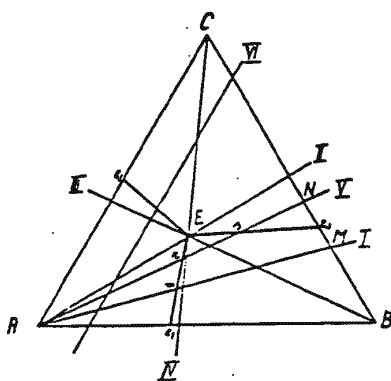


Figure 99 Projection of 3-dimensional melting point diagram on the concentration plane.

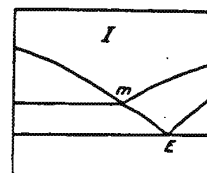


Figure 100 Typical cross-section through the 3-dimensional diagram (I in Figure 99).

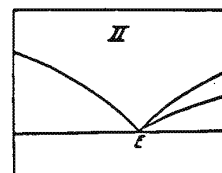


Figure 101 Typical cross-section through the 3-dimensional diagram (II in Figure 99).

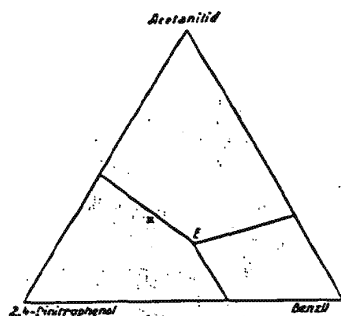


Figure 102 Three-Component composition diagram 2,4-dinitrophenol: acetanilide: benzil.

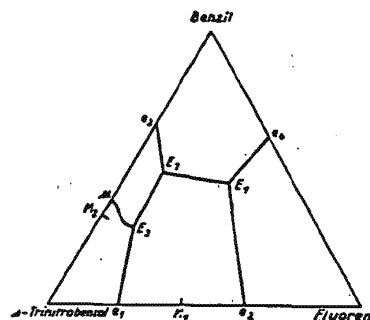


Figure 103 Three-Component composition diagram s-trinitrobenzene: benzil: fluorene.

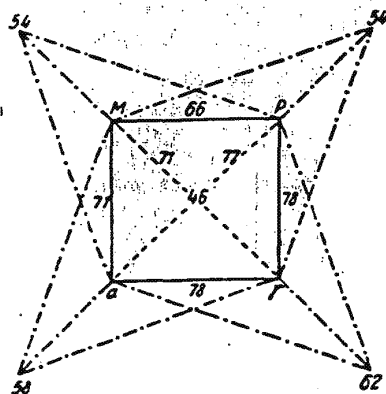


Figure 104 Eutectic melting points of 2-, 3-, and 4-component mixtures of m-nitrophenol(M), p-nitrophenol(P), 2,5-dinitrophenol(d) and 2,4-dinitrophenol(γ).

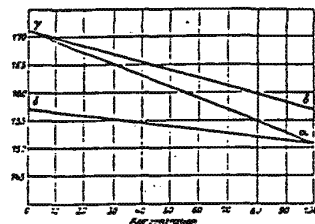


Figure 106 Refractive index of the melt of α-, γ-, and δ-hexachlorocyclohexane mixtures (glass powder 1.5101).

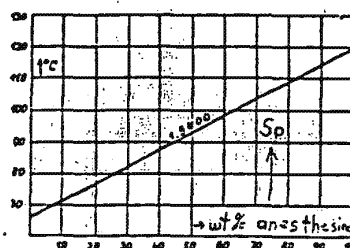


Figure 105 Refractive index of the melt in the system cycloform: anesthesine with glass powder 1.5400.

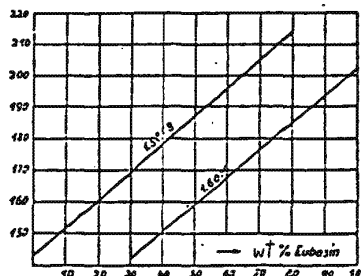


Figure 107 Refractive index of the melt of mixtures of uliron: eubasin with glass powders 1.5898 and 1.6010.

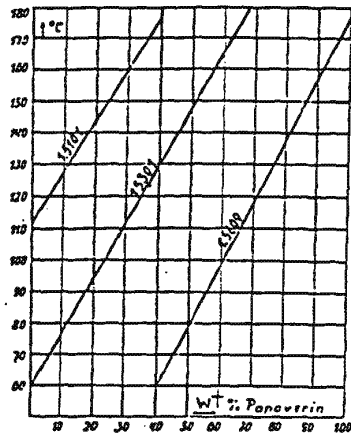


Figure 108 Atropine:
papaverin.

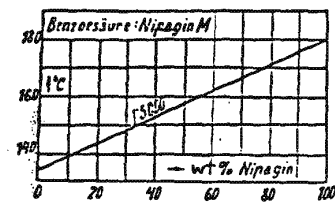


Figure 110 Benzoic acid:
nipagin M.

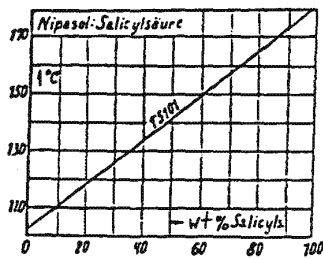


Figure 112 Nipasol:
salicylic acid.

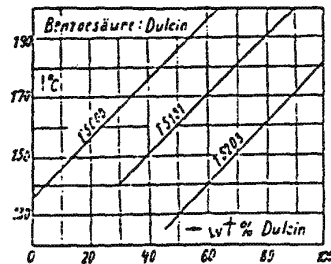


Figure 114 Benzoic acid:
dulcin.

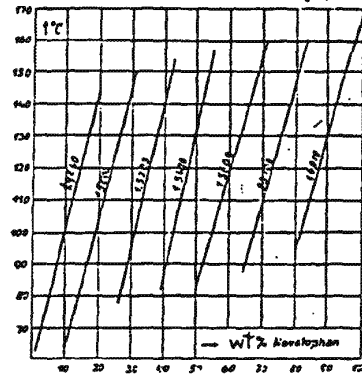


Figure 109 Trional:
novatophanek.

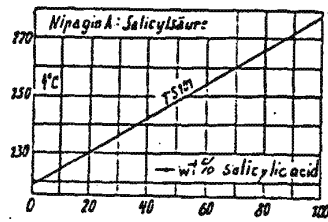


Figure 111 Nipagin A:
Salicylic acid.

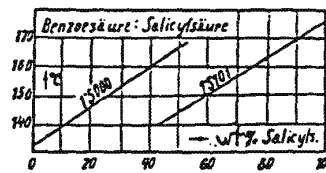


Figure 113 Benzoic acid:
salicylic acid.

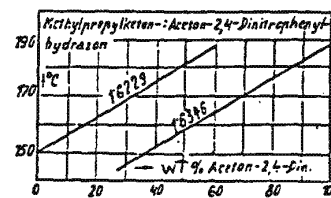


Figure 115 Methylpropyl-
ketone: acetone 2,4-
dinitrophenylhydrazone.

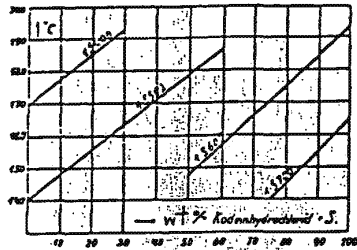


Figure 116 Ephedrine:
Codeine hydrochloride
(all mixed with equal
weights of salphen).

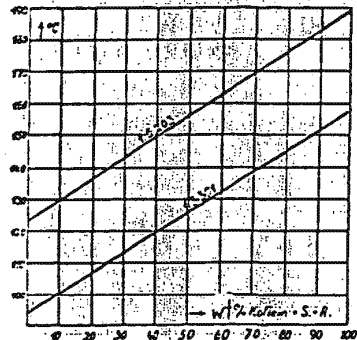


Figure 118 Cardiazole:
Caffeine (all mixed with
salphen and acetanilide).

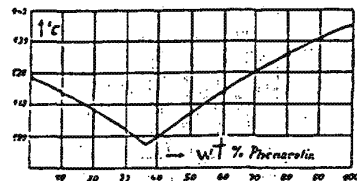


Figure 120 Eutectic
composition diagram for
Lactophenine: phenactin.

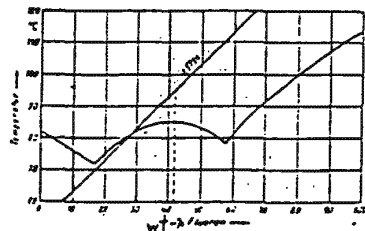


Figure 122 Mixed fusion
of α -trinitrotoluene:
fluorene with admixed
glass powder 1.5794.

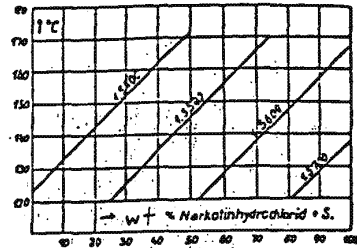


Figure 117 Aल्पine hydro-
chloride: narcotine
hydrochloride (all mixed
with equal weights of
salphen).

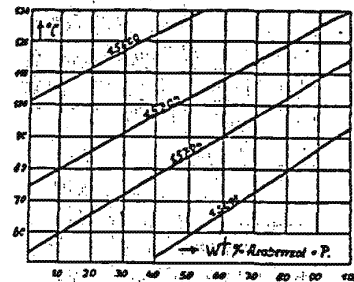


Figure 119 Thiosinamine:
azobenzene (all mixed
with phenylethyl urea).

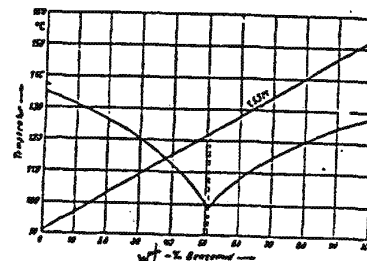


Figure 121 Mixed fusion
of phenacetin: benzamide
with admixed glass pow-
der 1.5301.

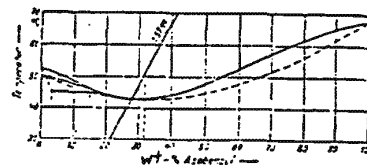


Figure 123 Dibenzyl:
azobenzene with glass
powder 1.5794.

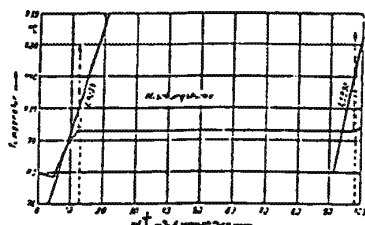


Figure 124 Acetamide:
acenaphthene with glass
powders, 1.4339 and
1.5898.

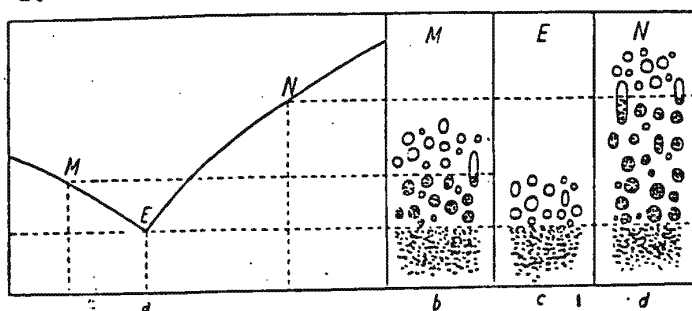


Figure 125 Schematic
eutectic diagram and
corresponding behavior
on the hot bar.



Figure 126 Oriented
overgrowths of qui-
none on pyrogallol.



Figure 127 Oriented
overgrowths of cam-
phor on hydroquinone.

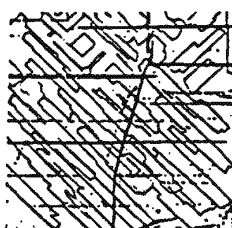


Figure 128 Oriented over-
growths of hexaethylben-
zene on chloranil.

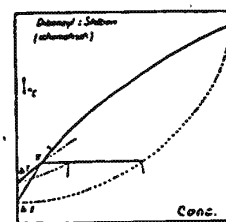
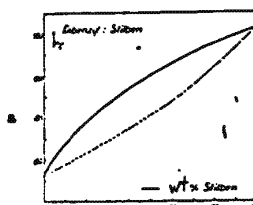


Figure 129 a) Dibenzyl:
stilbene according to the
literature. b) same ac-
cording to Kofler and
Brandstätter.

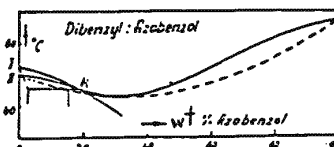
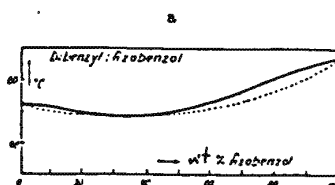


Figure 130 a) Dibenzyl:
azobenzene according to
the literature. b) same
according to Kofler and
Brandstätter.

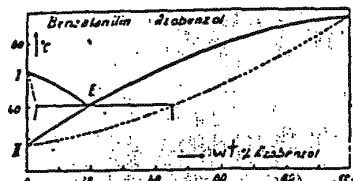


Figure 131 Azobenzene: benzalaniline (Brändstätter).

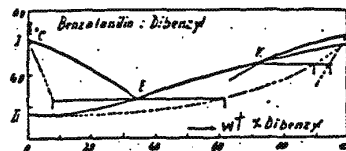
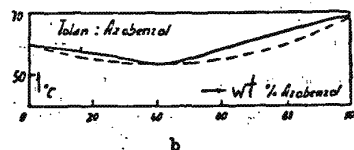
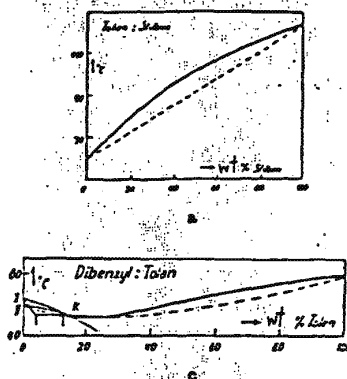
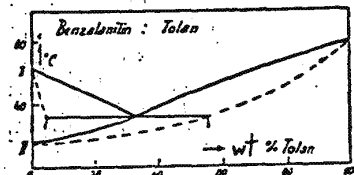


Figure 132 Dibenzyl: benzalaniline with stabilized intermediate phase (Brandstätter).



b



d

Figure 133 Tolane in two-component systems with a) stilbene, b) azobenzene c) dibenzyl and d) benzalaniline (Brandstätter).

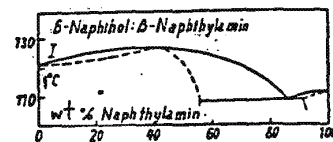


Figure 135 β -Naphthol: β -naphthylamine (Kofler and Brandstätter).

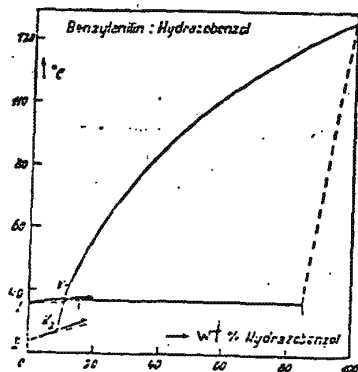


Figure 134 Benzalaniline: hydrazobenzene (Brändstätter).

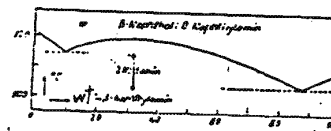


Figure 136 β -Naphthol: β -naphthylamine (Kremann)



Figure 137 β -Naphthol: β -naphthylamine (Krynakowsky).

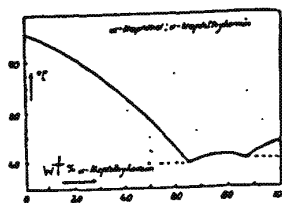


Figure 138 α -Naphthol:
 α -naphthylamine (liter-
ature).

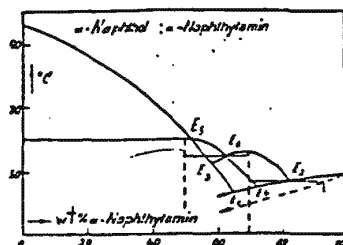


Figure 139 α -Naphthol:
 α -naphthylamine (Kofler
and Brandstätter).

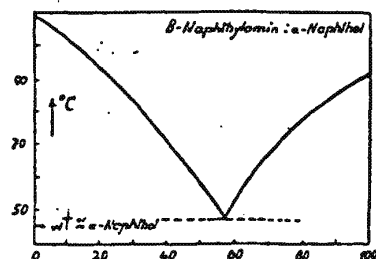


Figure 140 α -Naphthol:
 β -naphthylamine (liter-
ature).

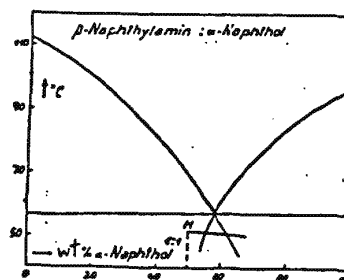


Figure 141 α -Naphthol:
 β -naphthylamine (Kofler
and Brandstätter).

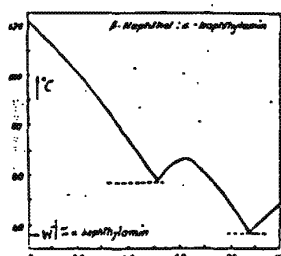


Figure 142 β -Naphthol:
 α -naphthylamine (Kremann)

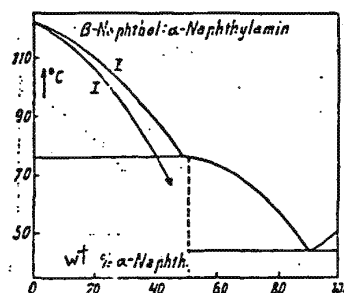


Figure 143 β -Naphthol:
 α -naphthylamine (Kofler
and Brandstätter).

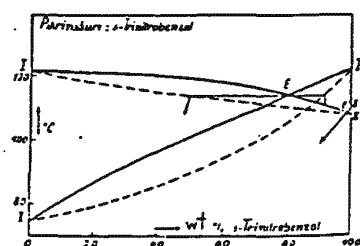
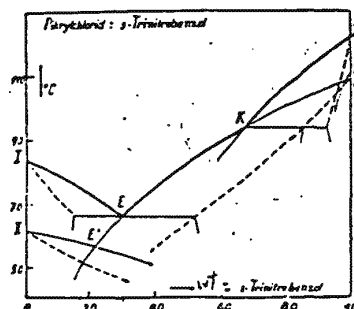


Figure 144 Roozeboom
Type V solid solution
system-picric acid: s-
trinitrobenzene.

Figure 145 Stabilized
intermediate phase for
picrylchloride: s-
trinitrobenzene.



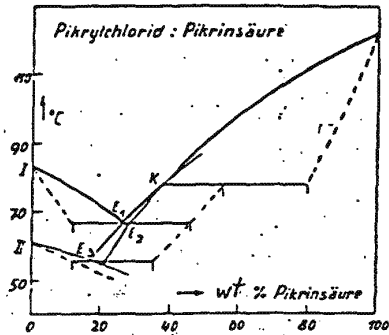


Figure 146 Stabilized intermediate phase for picrylchloride: picric acid.

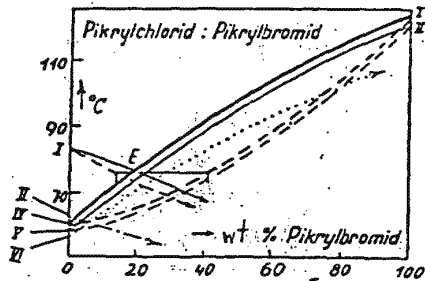


Figure 148 Picrylchloride:
picrylbromide.

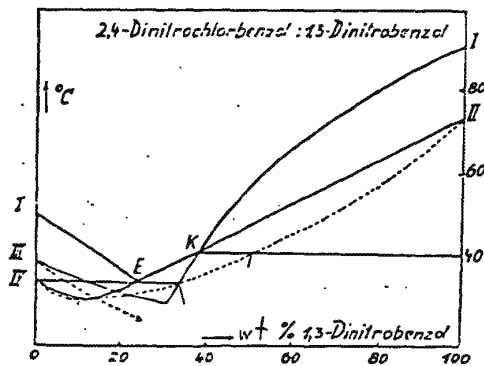


Figure 150 Stabilized
intermediate phase for
2,4-dinitrochlorobenzene:
1,3-dinitrobenzene.

Figure 152 Stabilized
intermediate phase for
2,4-dinitrochlorbenzene:
2,4-dinitroaniline.

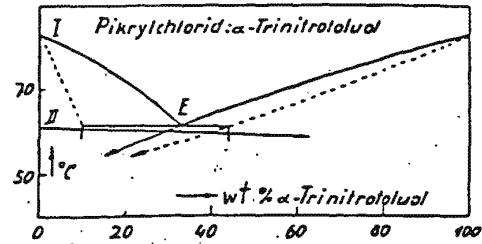


Figure 147 Picrylchloride;
s-trinitrotoluene (Kofler
and Brandstätter).

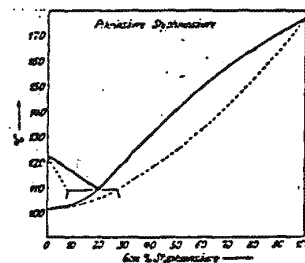


Figure 149 Picric acid;
styphnic acid.

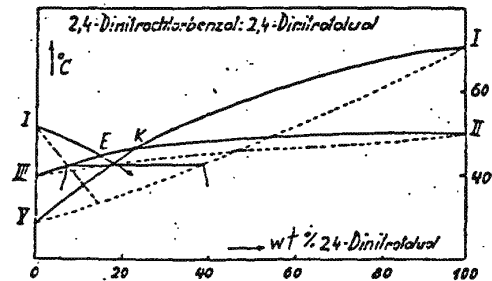
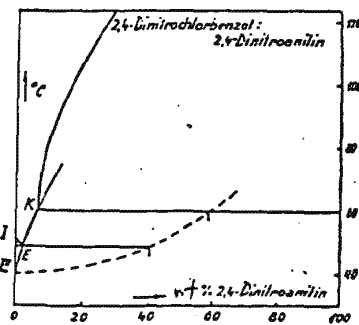


Figure 151 Stabilized
intermediate phase for
2,4-dinitrochlorobenzene:
2,4-dinitrotoluene.



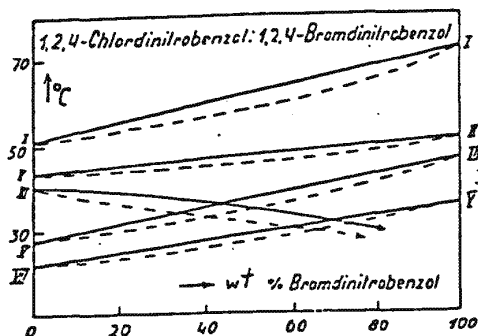


Figure 153 Parallel Isomorphism for 1,2,4-bromdinitrobenzene (all type I Roozeboom).

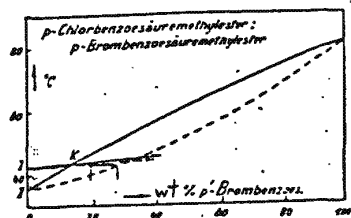


Figure 155 Roozeboom Type I isodimorphism for p-chlor- and p-brom benzoic acid methyl ester

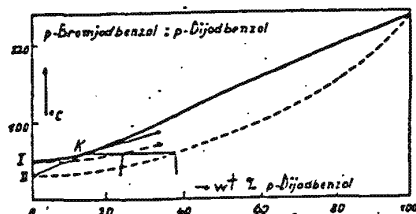


Figure 157 Roozeboom type IV isodimorphism for p-bromido- and p-dijodobenzene.

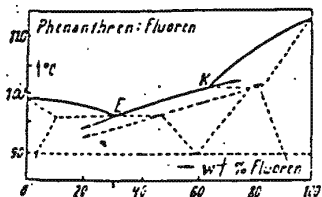


Figure 159 Stabilized intermediate phase for phenanthrene: fluorene.

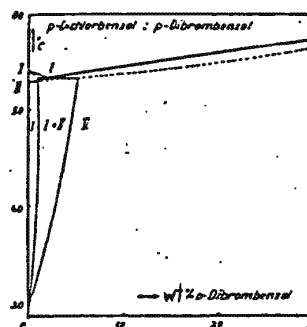


Figure 154 Roozeboom Type I isodimorphism for p-dichlorbenzene: p-dibrombenzene.

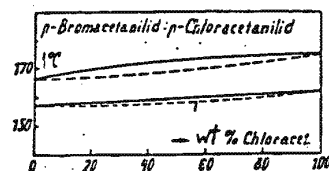


Figure 156 Parallel isodimorphism for p-chlor- and p-brom acetanilide.

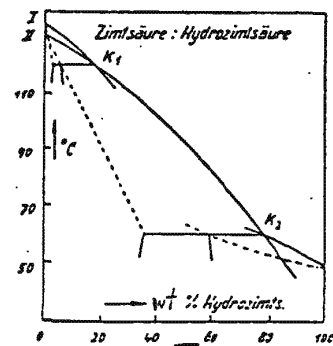


Figure 158 Stabilized intermediate phase for cinnamic acid: hydro-cinnamic acid.

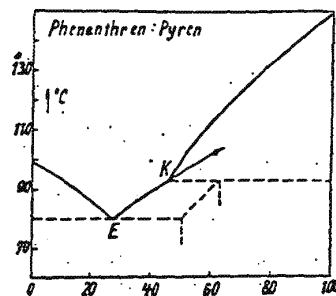


Figure 160 Stabilized intermediate phase for phenanthrene: pyrene.

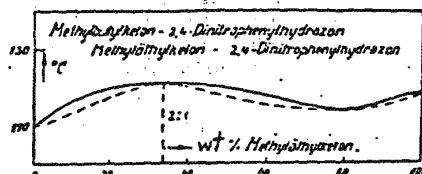


Figure 161 Continuous solid solubility with maximum and minimum melting mixtures for methylbutylketone: methylethylketone-2,4-dinitrophenylhydrazone.

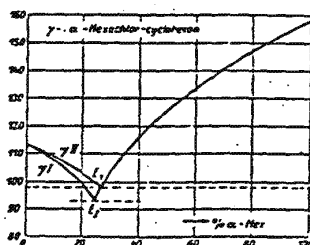


Figure 162 Binary composition diagram for γ-α-hexachlorocyclohexane.

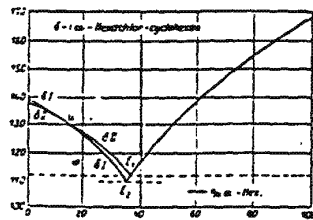


Figure 163 Binary composition diagram for δ-α-hexachlorocyclohexane.

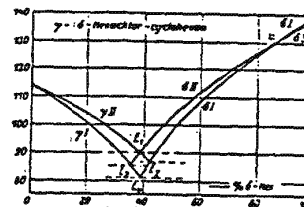


Figure 164 Binary composition diagram for γ-δ-hexachlorocyclohexane.

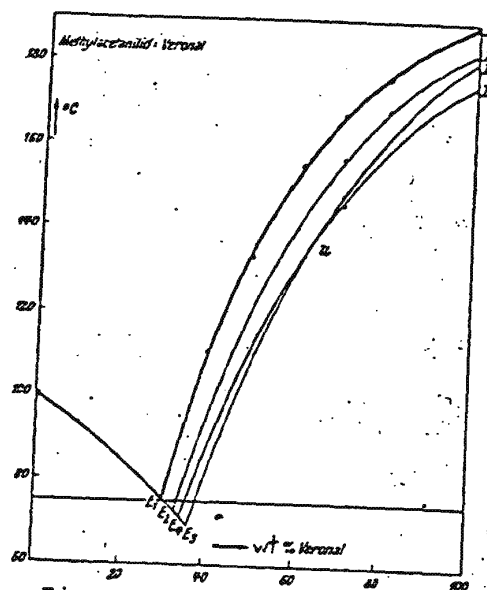


Figure 165 Methylacetanilid: veronal.

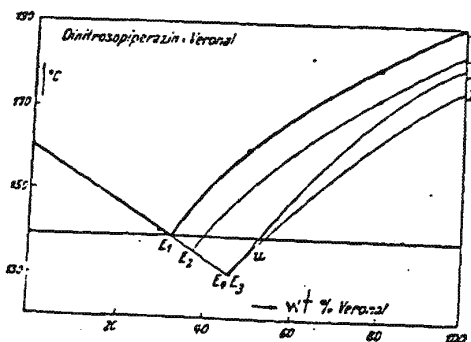


Figure 166 Dinitroperazine: veronal.

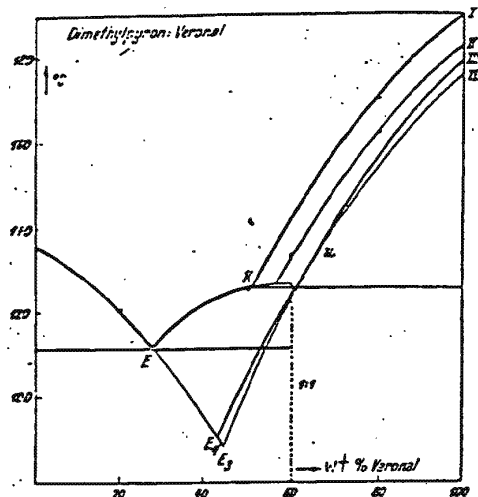


Figure 167 2,6-Diethylpyrone: veronal.

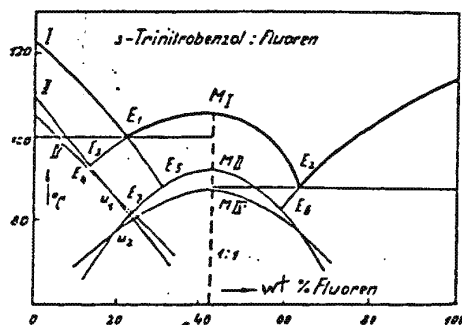


Figure 169 s-Trinitrobenzene: fluorene.

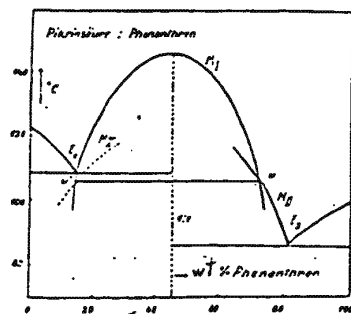


Figure 171 Picric acid: phenanthrene.

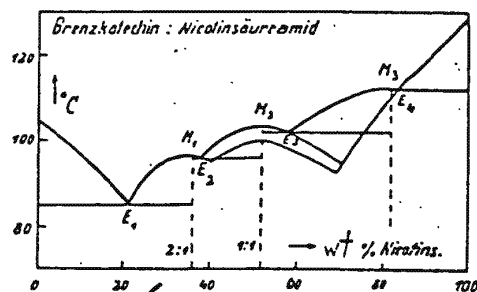


Figure 168 Pyrocatechol: nicotinic acid amide.

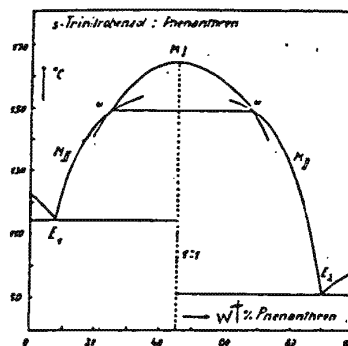


Figure 170 s-Trinitrobenzene: phenanthrene.

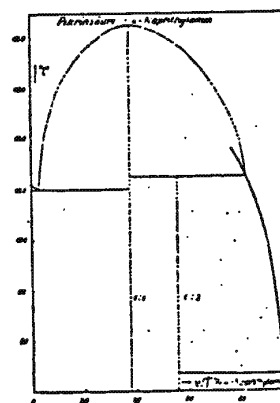


Figure 172 Picric acid: α-naphthylamine.

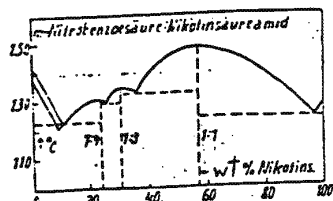


Figure 173 m-Nitrobenzoic acid: nicotinic acid amide.

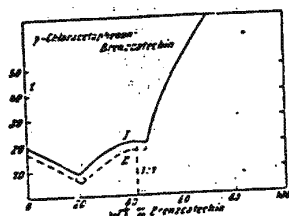


Figure 175 p-chloroacetophenone: pyrocatechol.

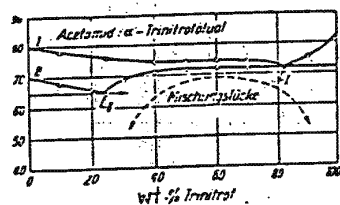


Figure 177 Both modifications of acetamide show an inflection in the composition diagram with s-trinitrotoluene.

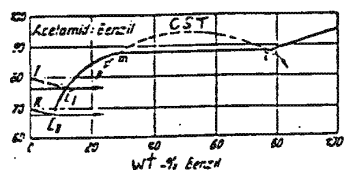


Figure 179 Acetamide: benzil

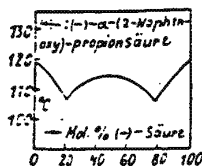


Figure 181 True racemate (+)-(-)-α(2-naphthoxy)-propionic acid.

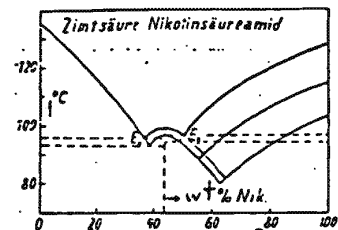


Figure 174 Cinnamic acid: nicotinic acid amide.

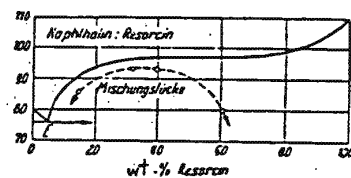


Figure 176 Melting point curve for naphthalene: resorcinol showing an inflection.

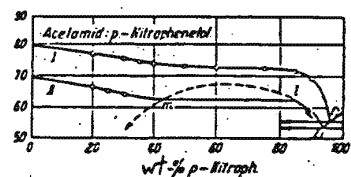


Figure 178 Acetamide I shows an inflection in the melting point curve with p-nitrophenetole; Form II shows two immiscible liquid phases.

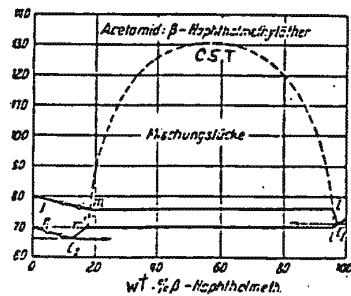


Figure 180 Acetamide: β-naphthol methyl ether.

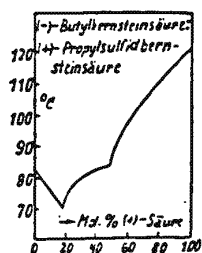


Figure 182 Pseudo racemate (+)-propylsulfide succinic acid: (-)-butyl succinic acid.

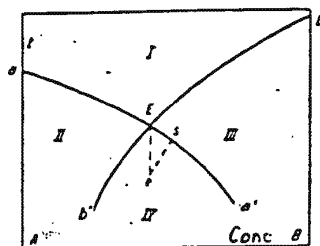


Figure 184 Abnormal phase separation in simple eutectic systems due to supercooling.

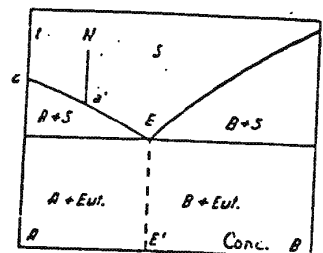


Figure 183 Normal phase separation in simple eutectic systems.

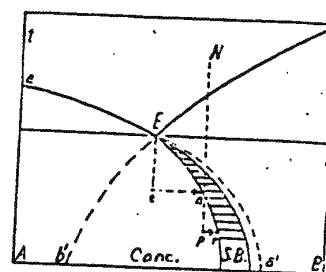


Figure 185 Quasi-eutectic syncrystallization region: type A.

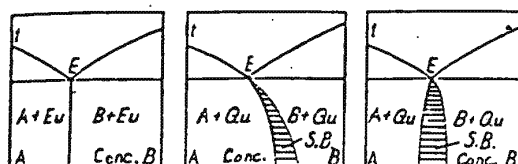


Figure 186 Comparison of normal phase regions in binary systems with different types of quasi-eutectic syncrystallization.

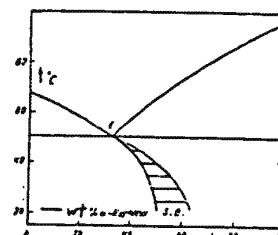


Figure 187 Azobenzene: α -naphthol.

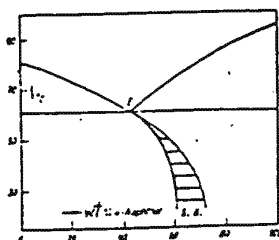


Figure 188 Naphthalene: α -naphthol.

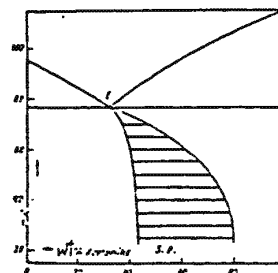


Figure 189 Benzil: acetanilide.

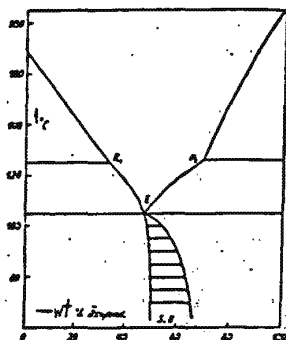


Figure 190 Ammonium nitrate: ethylene diamine dinitrate.

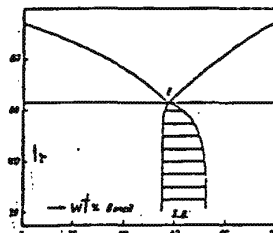


Figure 191 Acenaphthene: Benzil.

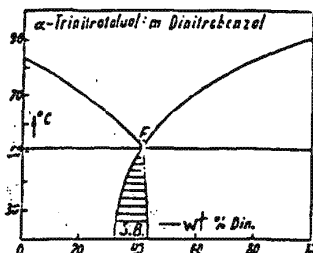


Figure 192 a-Trinitrotoluene: m-dinitrobenzene.

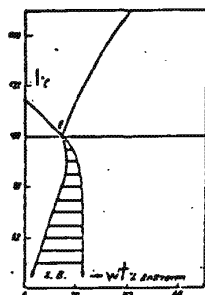


Figure 193 Anthracene: 2,4-dinitrophenol.

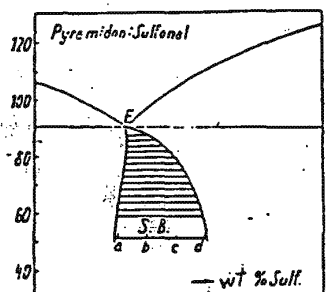


Figure 194 Pyramidon: sulfonal.



Figure 195 Quasi-eutectic syncrystallization of acenaphthene with 40% sulfonal.



Figure 196 Quasi-eutectic syncrystallization of antipyrine with 50% sulfonal.

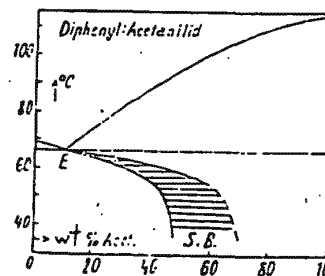


Figure 197 Type A quasi-eutectic syncrystallization for diphenyl: acetanilide.

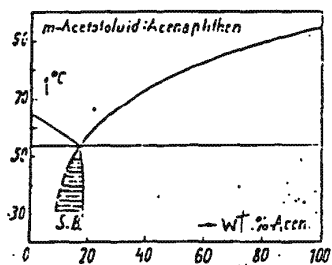


Figure 198 Type B quasi-eutectic syncrystallization for Acer-m-toluidide acenaphthene.

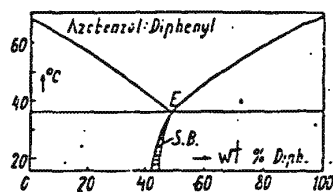


Figure 199 Narrow
syncrystallization region
for azobenzene: diphenyl.

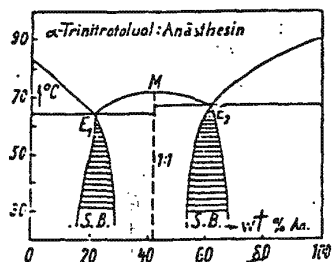


Figure 200 Syn-crystal-
lization regions for the
addition compound system
of s-trinitrotoluene:
anaesthesine.

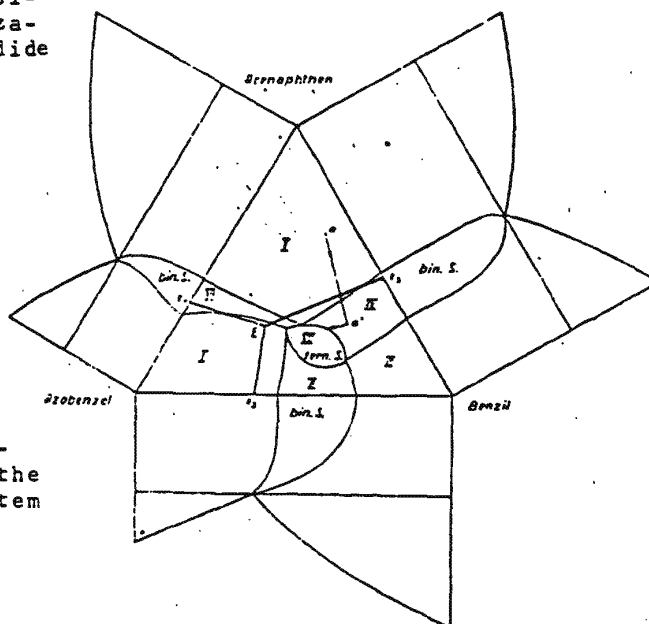


Figure 201a. Syncrystallization regions in the ternary system azobenzene: acenaphthacene: benzil on supercooling 20°C.

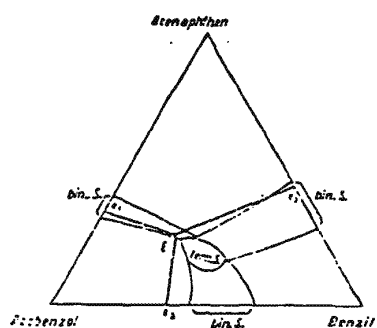


Figure 201b. Syncrystallization regions in the ternary system acenaphthacene: azobenzene: benzil at 50°C.

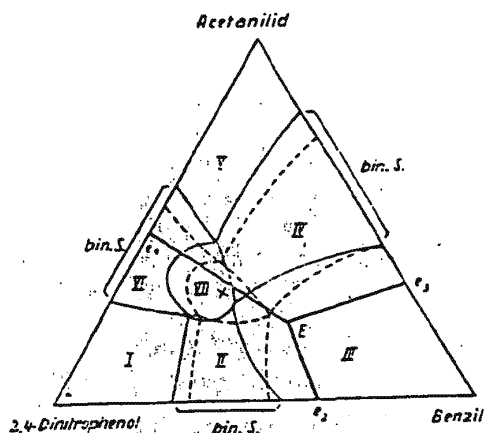


Figure 202. Syncrystallization regions in the ternary system 2,4-dinitrophenol: acetanilide: benzil supercooled 20°C at 50°C; x=the ternary eutectic composition according to Tammann.

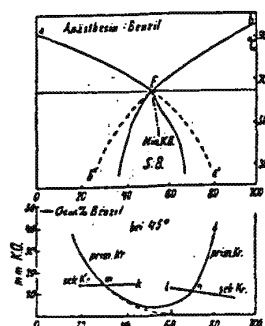


Figure 204 Isothermal crystallization velocities of the primary and secondary crystal growths on supercooling 45°C for anaesthesine: benzil.

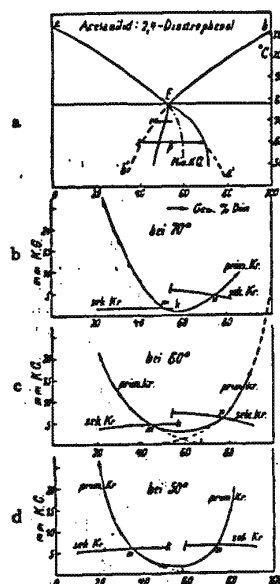


Figure 203 Isothermal crystallization velocities of the primary and secondary crystal growths on supercooling to 70°, 60° and 50°C for acetanilide: 2,4-dinitrophenol.

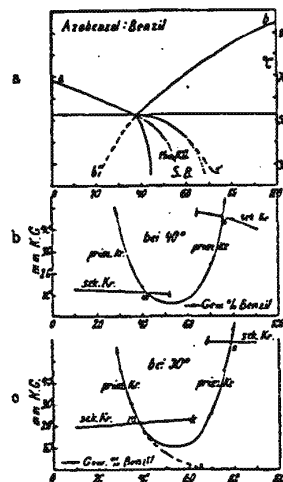


Figure 205 Isothermal
crystallization velocities on
supercooling to 40° and 30° C
for azobenzene: benzil.

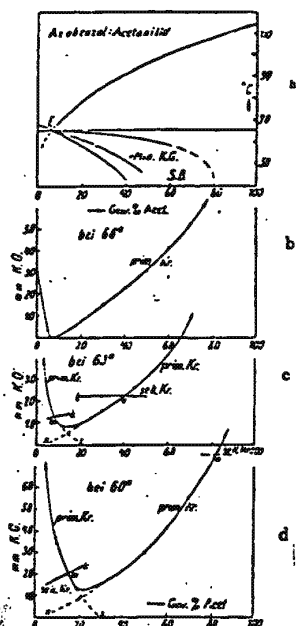


Figure 206 Isothermal crystallization velocities on supercooling to 63° and 60°C for azobenzene: acetanilide.

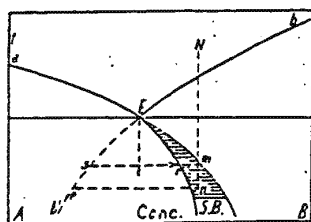


Figure 207 Conditions favorable to the formation of crystal halos.

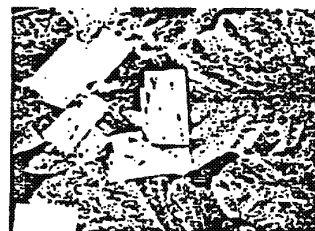
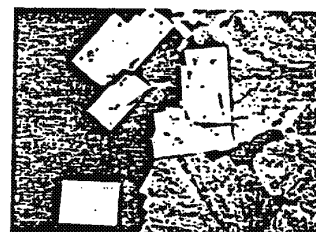
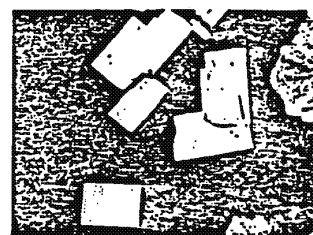
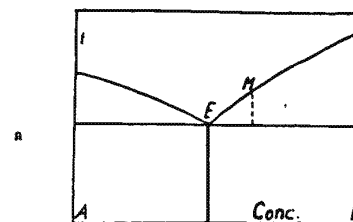


Figure 208 Normal crystallization of a non-eutectic mixture.

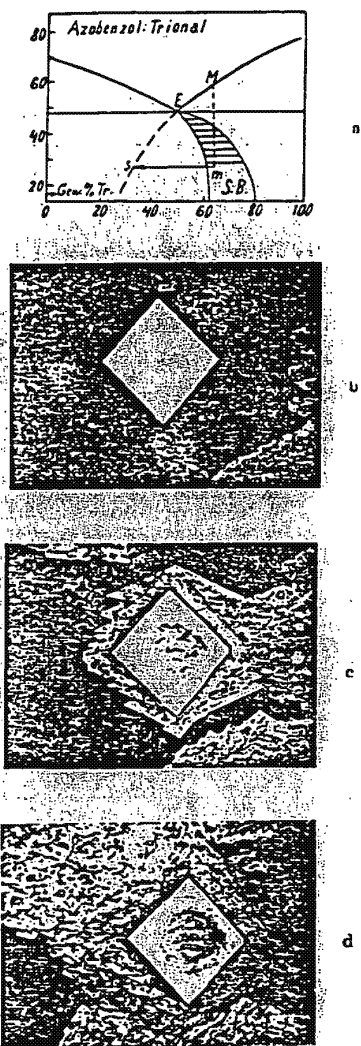


Figure 209 Halo formation during crystallization of a supercooled non-eutectic mixture of azobenzene: trional.

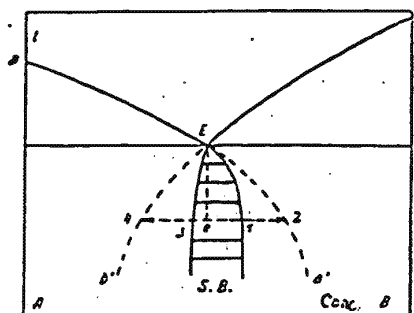


Figure 210 Halo formation of type B.

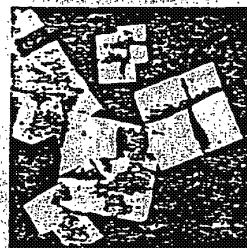


Figure 211 g-Strophanthine (quabain) decahydrate.



Figure 212 g-Strophanthine (quabain) octahydrate.

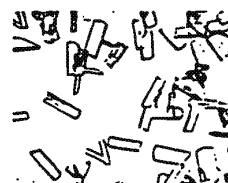
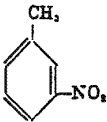
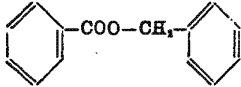
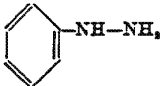
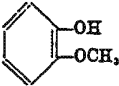
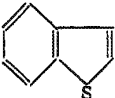
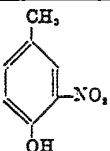
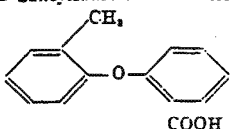
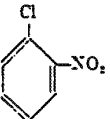
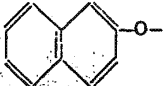
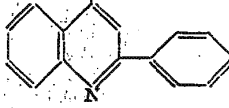
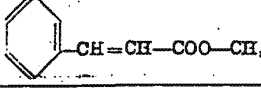
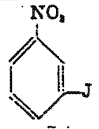
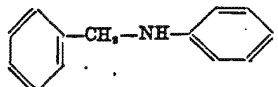
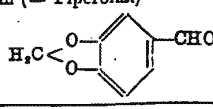
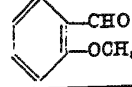
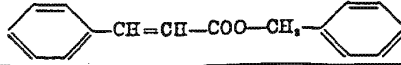
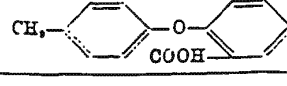
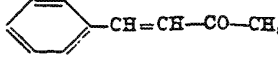
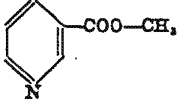
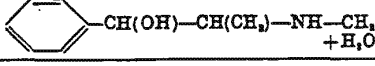
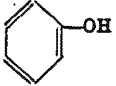
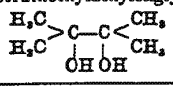
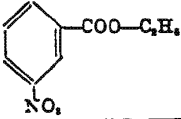
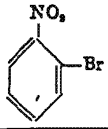
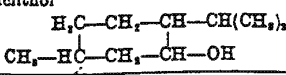
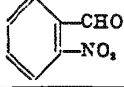
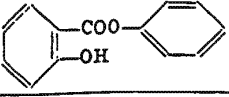
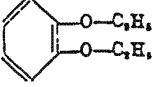


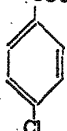
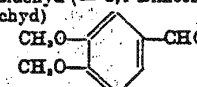
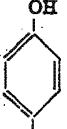
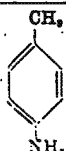
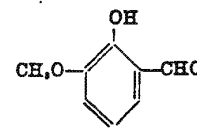
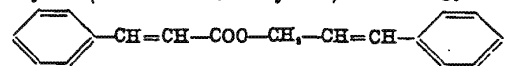
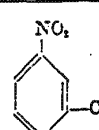
Figure 213 g-Strophanthine (quabain) dihydrate from NaCl solution.

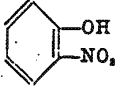
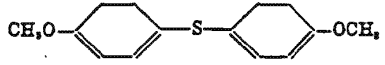
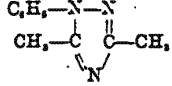
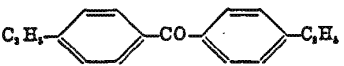
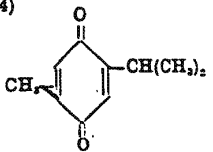
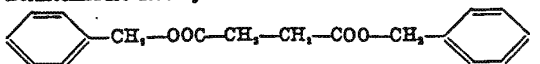
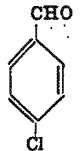
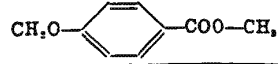
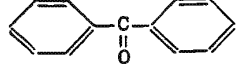
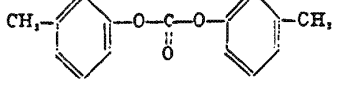
XIV. Identifizierungs-Tabelle

F. °C	Substanz	Eutekt. Temp. mit β -Naphthol-äthyläther	Temp. mit Benzol	Glas-pulver	Temp. °C	Besondere Kennzeichen
14	m-Nitrotoluol 	-3	-18	1,5403 1,5502	28-30 9-10	Gleichgew.: lange Spindeln, Stengel. In Wasser nur sehr wenig löslich.
19	Benzoesäure-benzylester 	3	-16	1,5700 1,5611	14 35	Kristallisiert äußerst trüg, meist erst d. Kratzen bei etwa -20° bis -30°. Gleichgew. sehr trüg: derbe Körner, Prismen. In Wasser unlöslich.
16-19	Phenylhydrazin 	8	-4	1,5611	20-22	Häufig orangerot verfärbt. Gleichgew.: sechseckige Prismen, Platten, Stengel. Verändert sich an der Luft zu feinkristallinen Krusten (am Rand des Deckglases), die zwischen 30-40° schmelzen.
Azobenzol Benzil						
27	p-Bromtoluol 	13	20	1,5403 1,5299	35-37 50-52	Geruch. Erstarrt in Platten.
28,5	Guajacol (= Brenzcatechinmonomethyl-äther) 	18.	17	1,5301 1,5204	41 62-64	Geruch. Gleichgew.: Prismen. Schmelze stark unterkühlbar. Dimorph. (6).
31	Thionaphthen 	16	18	1,6231 1,6126	46 65-66	Geruch. Gleichgew.: Gelappte Blättchen.
32	3-Nitro-4-oxytoluol 	19	22	1,5700	38-40	Gelb. Geruch. Gleichgew. trüg: lange, zugespitzte Stengel, häufig Zwillinge.
33	o-Kresalol (= Salicylsäure-o-kresoläther) 	flüssig	flüssig	1,5700 1,5611	41 62-63	Gleichgew.: Körner. Schmelze unterkühlbar.
33	o-Chlornitrobenzol 	20	22	1,5501 1,5403	46-47 65-66	Hellgelb. Bittermandelgeruch. Gleichgew.: unregelmäßige Kristalle. Schmelze unterkühlbar, bei Druck auf das Deckglas Sphärolithe.

F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
34	2-Methylnaphthalin 	16 22	1,5897	59	Geruch. Beim Auflegen sublimieren sofort Blättchen. Gleichgew.: Platten.
35	β -Naphtholäthyläther (= Nerolin Neu) 	22 27	1,5897 1,5704	45—46 67—68 2,2	Ab 25° Körner. Gleichgew.: bandförmige Kristalle.
36	Atochinol (= 2-Phenyl-cinchoninsäureallylester) 	12 17	1,0483 1,6353	28—29 55—57 2,7	Gleichgew. trüg.: Körner, Prismen. Schmelze unterkühlbar; kristallisiert sehr langsam in Rosetten.
36	Zimtsäure-methylester 	21 26	1,5011 1,5502	41—42 63—64 2,2	Gleichgew.: Stengel.
36	m-Jodnitrobenzol 	20 25	1,6353 1,6231	43—44 66—67 2,2	Geruch. Gelblich; wenn bräunlich verfärbt, absaugen. Gleichgew. trüg.: Prismen. Schmelze unterkühlbar.
36	Benzylanilin 	25 23	1,6011	43—44	Ab 30° Tröpfchen. Gleichgew.: Körner und Blättchen. Dimorph. (7).
36	Heliotropin (= Piperonal) 	26 26	1,5795 1,5700	44—45 67—68 2,3	Häufig bräunlich verfärbt. Absaugen! Gleichgew.: Körner, Stengel.
37	Salicylaldehydmethyläther 	23 34	1,5502 1,5403	37 60 2,2	Gleichgew.: Körner, Prismen. Schmelze unterkühlbar.
38	Zimtsäure-benzylester 	22 27	1,5897 1,5795	46—47 69—70 2,2	Geruch. Gleichgew.: Platten. Erstarrt zu feinstrahligem Sphärolithen.
39	p-Kresolol (= Salicylsäure-p-kresoläther) 	23 27	1,5700 1,5601? 1,5600	44—45 60—67 2,2	Geruch. Ab 25° Tröpfchen. Gleichgew.: Körner, Platten.
40	Benzalacetone 	24 28	1,5795 1,5700	45—46 64—65 2,2	Geruch nach Cumarin und Rhabarber. Bei gelblicher Verfärbung umkristallisieren aus Alkohol. Gleichgew. trüg.: Rechtecke, Prismen. Schmelze unterkühlbar. Dimorph. II 30° (8).

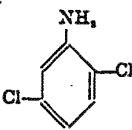
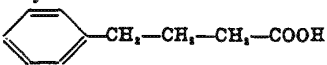
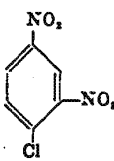
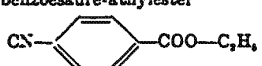
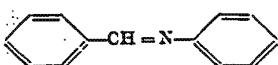
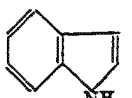
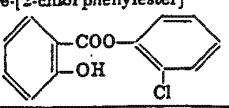
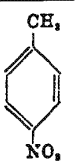
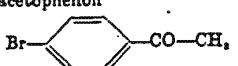
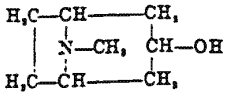
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
40	Nicotinsäure-methylester (Pyridin-β-carbonsäure-methylester) 	15 30	1,5002 1,4936	53—55 69—71	Ab 25° Körner, Blättchen, Nadeln. Gleichgew.: Balken, Tafeln. Erstarrt sphärolithisch.
40	l-Ephedrin  + H ₂ O	31 33	1,5204 1,5101	43 63	Schmilzt als Hydrat langsam unter Hüpfen der Kristalle. Gleichgew.: Körner und Prismen.
40	Phenol 	25 19	1,5403 1,5299	35—36 82—84	Geruch. Ab 25° Nadeln. Gleichgew.: Körner und Balken.
36—41	Pinakon (= Tetramethyläthylenglykol) 	34 31	1,4339	40—41	Naphthalinähnlicher Geruch. Ab 30° Balken, Blättchen. Gleichgew.: Nadeln.
41	m-Nitrobenzoesäure-äthylester 	26 26	1,5299 1,5101	48—49 71—72	Gleichgew.: sechseckige, rechteckige Blättchen und Stengel.
41	Hexamethylenglykol HO-CH ₂ -[CH ₂] ₄ -CH ₂ -OH	39 38	1,4339	80—90	Ab 30° Blättchen. Gleichgew.: Körner.
41	o-Bromnitrobenzol 	25 27	1,5700 1,5785	60—62 42—44	Gelb. Ab 30° Tröpfchen. Gleichgew.: derbe Prismen. Erstarrt strahlig.
41,5	n-Tridecansäure CH ₃ -[CH ₂] ₁₁ -COOH	33 39	1,4339	45	Über 32° Umwandlung. Schmelzpunkt meist unscharf, dann Eutektikum 2° tiefer. Gleichgew.: gelappte Platten, Stengel. Enantiotrop dimorph (9).
42	l-Menthol 	32 37	1,4339	87—88	Geruch. Ab 35° kurze, feine Nadelchen. Gleichgew.: unregelmäßige Formen.
42,5	o-Nitrobenzaldehyd 	31 31	1,5611 1,5502	50—51 73—74	Hellgelb. Gleichgew.: spindelförmige Kristalle. Dimorph. Schmelze erstarrt meist zu Sphärolithen von II 39° (10).
42,5	Salol (= Salicylsäure-phenylester) 	26 29	1,5700 1,5611	57—60 80—81	Ab 38° Tröpfchen. Gleichgew.: Stengel Rauten. Erstarrt erst bei starkem Abkühlen. Polymorph (11, 12).
43	Brenzcatechin-diäthyläther 	20 32	1,5502 1,4936	40 51—53	Geruch. Gleichgew.: derbe Prismen und Platten. Schmelze unterkühlbar.

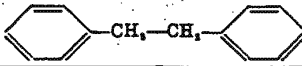
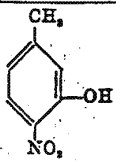
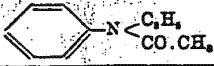
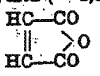
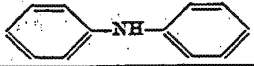
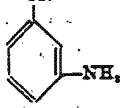
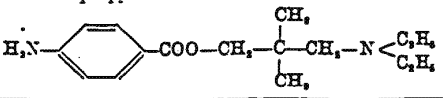
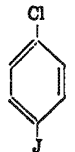
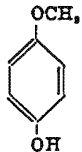
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
43	p-Chlorbenzoesäure-methylester 	26	31	1,5299 1,5204	34—35 55—56 21 Geruch. Ab 35° Körner. Gleichgew.: Körner, Prismen. Erstarrt sphäro- lithisch.
43	1,9-Nonandiol $\text{HO}-\text{CH}_2-[\text{CH}_2]_7-\text{CH}_2-\text{OH}$	38	39	1,4339— 1,4584	Geruch. Gleichgew. trägt: spitze Rauten, Linsen, Schuppen.
43	Veratrumaldehyd (= 3,4-Dimethoxy- benzaldehyd) 	28	29	1,5700 1,5611	40—42 62—64 27 Geruch nach bitteren Mandeln. Gleich- gew. trägt: Platten. Erstarrt langsam.
43	p-Chlorphenol 	flüssig	flüssig	1,5502 1,5403	56—58 81—83 25 Geruch. Ab 25° Tröpfchen.
44	p-Aminotoluol (= p-Toluidin) 	28	27	1,5502 1,5403	47 65—66 16 Ab 35° Blättchen. Gleichgew.: unregelmäßige Blättchen.
44	Laurinsäure $\text{CH}_3-[\text{CH}_2]_{10}-\text{COOH}$	35	40	<1,4339	F. meist unscharf, dann Eutektikum 2° tiefer. Gleichgew.: rundliche Blättchen und Spindeln.
44	o-Vanillin 	30 tr.	29	1,5700 1,5611	42—44 64—65 22 Vanillegeruch. Ab 30° Nadeln. Gleich- gew.: Nadeln.
44	Styracin (= Zimtsäure-cinnamylester) 	30	34	1,6011 1,5897	53—59 83 24 Leichter Zimtgeruch. Gleichgew. etwas trägt: Balken.
44,5	Elaidinsäure $\text{CH}_3-[\text{CH}_2]_7-\text{CH}=\text{CH}-[\text{CH}_2]_7-\text{COOH}$	38	42	1,4339	88—90 Gleichgew.: Rhomboide und Stengel.
45	m-Chlornitrobenzol 	30	31	1,5502 1,5403	56—58 76—78 13 Bittermandelgeruch. Gleichgew.: Pris- men, Balken. Dimorph. Schmelze unter- kühlbar (13).
45	p-Aminobenzylcyanid 	38	36	1,5611 1,5502	67—68 89—90 22 Gelb. Gleichgew.: Prismen, Platten.

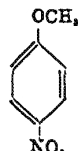
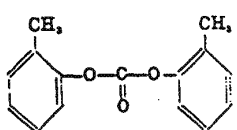
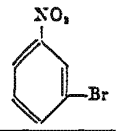
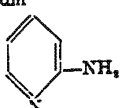
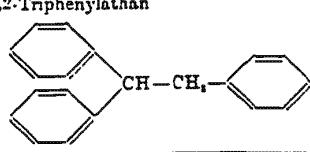
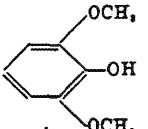
F. ° C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. ° C	Besondere Kennzeichen
45	o-Nitrophenol 	31 39	1,5700 1,5611	57—58 77—78 26	Gelb. Ab 40° Stengel und Spieß. Gleichgew.: spindelförmige Kristalle.
45	Dianisylsulfid 	28 34	1,6011 1,5898	58—59 85—86 27	Gleichgew.: Platten. Dimorph (14).
46	1-Phenyl-3,5-dimethyl-1,2,4-triazol 	flüssig flüssig	1,5502 1,5403	40—41 65—66 25	Ab 40° Tröpfchen. Gleichgew.: Körner. Dimorph.
47	p,p'-Diäthylbenzophenon 	28 35	1,5700 1,5611	47—49 68—69 21	Ab 35° Körner. Gleichgew.: Prismen und Körner.
47	Thymochinon (= 2-Methyl-5-isopropylbenzoquinon-1,4) 	25 33	1,5000	53—54	Gelb. Geruch. Ab 30° Nadeln, Stäbchen. Gleichgew.: Balken, Prismen. Erstarrt sphärolithisch.
47	Bernsteinsäure-dibenzylester 	34 38	1,5204 1,5101	68—70 98—98 22	Gleichgew.: Platten. Erstarrt sphärolithisch.
48	p-Chlorbenzaldehyd 	28 32	1,5502— 1,5611		Ab 30° Blättchen, Körner, Nadeln, Stengel. Gleichgew.: zugespitzte Stengel, Balken. Zersetzung und Abscheidung hochschmelzender Nadeln (über 170°).
46—48	Alival (= 1,2-Dioxy-3-jodpropan) $\text{CH}_2\text{J}-\text{CH}(\text{OH})-\text{CH}_2\text{OH}$	42 44	1,5611	73—74	Gleichgew.: Spindeln. Schmelze unterkühlbar.
48	Anisäure-methylester (= p-Methoxybenzoesäuremethylester) 	28 33	1,5203 1,5101	56—57 70—81	Geruch. Ab 35° Nadeln. Gleichgew.: Körner und Balken.
48	Benzophenon 	29 34	1,5897 1,5795	53 68—69 12	Ab 40° Tröpfchen. Gleichgew.: Körner. Schmelze unterkühlbar. Polymorph (11, 12).
48	m-Kresolcarbonat 	29 31	1,5299 1,5204	51 77—78 27	Geruch. Schmilzt und kristallisiert sehr träg. Schmelze unterkühlbar.

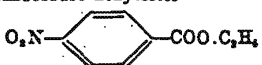
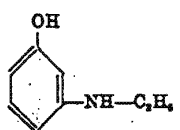
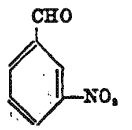
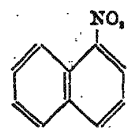
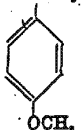
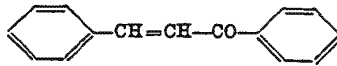
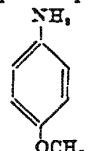
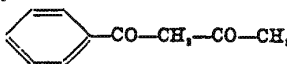
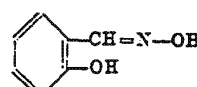
F. °C	Substanz	Eutekt. Temp. mit: Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
49	Urethan (= Carbamidsäureäthylester) 	41	42	<1,4339	Ab 40° Stengel, Spieße. Gleichgew.: Körner, Platten.
49	α -Naphthylamin 	33	28	1,6598	63—64 Ab 45° Stäbchen. Gleichgew.: lange Prismen. Polymorph II 32°, III 20° (15).
49	o-Aminodiphenyl 	27	32	1,6231 1,6126	49—50 Ab 40° wenig Körner. Gleichgew. trög: 70—71 Tafeln. Erstarrt in breitstrahligen 21 Sphärolithen.
49	Bromessigsäure $\text{CH}_2\text{Br}-\text{COOH}$	36	33	1,4683 1,4584	75 Ab 30° Blättchen, Körner. Gleichgew.: 99 Platten. Sinkt ab. Stechender Geruch. Dimorph (16).
49	Formanilid 	38	39	1,5700 1,5611	57—59 Ab 40° Nadeln. Gleichgew.: Tafeln, 79—81 Prismen. Erstarrt langsam sphäro- 22 lithisch. Dimorph (17).
49	Hydrozimtsäure 	32	38	1,5101	53—54 Ab 40° Tröpfchen. Gleichgew.: Stengel. Bei raschem Erstarren feinstrahlig. Dimorph (18, 19).
49	o-Nitrobenzylchlorid 	32	35	1,5611 1,5602	45—46 Ab 30° Körner. Gleichgew.: Balken, 69—70 Blättchen. Hautreizend. 24
49,5	Cetylalkohol $\text{CH}_3-(\text{CH}_2)_{14}-\text{CH}_2\text{OH}$	42	46	1,4339	68—70 Gleichgew. etwas trög: gelappte For- men.
50	Phenylurethan (= Euphorin = Carbanil- säureäthylester) 	35	36	1,5204 1,5101	57—58 Ab 45° Stäbchen. Gleichgew.: Balken 82—83 und Prismen. 25
50	o-Bromacetophenon 	34	36	1,5700 1,5611	54—55 Geruch. Gleichgew.: Platten. Schmelze 76—78 unterkühlbar, bei Erwärmen grob- 27 strahlige Kristalle.
50	Thymol 	31	30	1,5101 1,5000	44—45 Ab 40° Körner, Rauten und Sechsecke. 67—69 Gleichgew.: Rauten und Prismen. 28
50	N-Benzoylpiperidin 	23	30	1,5403 1,5299	57—58 Gleichgew.: Prismen. Erstarrt erst nach 80—82 Impfen zu Platten und breiten Strahlen. 29
50	β -Bromnaphthalin 	26	35	1,6231 1,6128	71—73 Ab 30° runde und gelappte Blättchen. 88—89 Gleichgew.: dünne Blättchen. 17

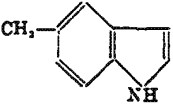
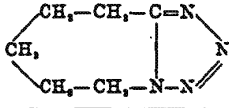
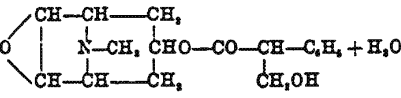
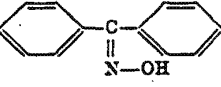
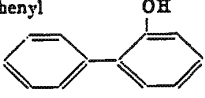
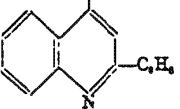
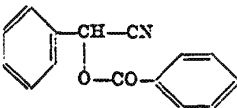
Körner, Mikro-Kristalle

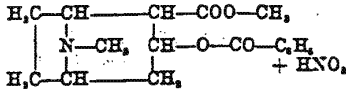
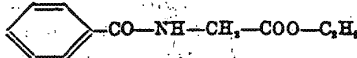
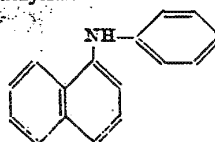
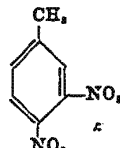
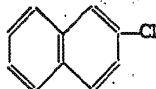
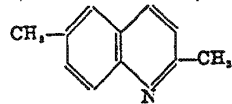
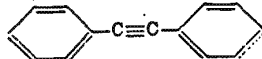
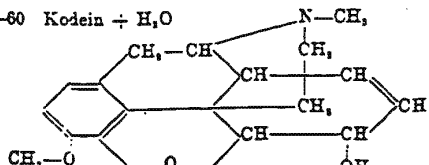
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulvor	Temp. °C	Besondere Kennzeichen
50	2,6-Dichloranilin 	30 29	1,5785 1,5700	66—68 87—89 21	Geruch. Ab 40° Nadeln, Körner. Gleichgew.: Balken. Erstarrt zu feinstrahligen Sphärolithen.
50	γ -Phenylbuttersäure 	30 35	1,5101 1,5110	47—48 70—71	Gleichgew.: Körner und Platten.
51	4-Chlor-1,3-dinitrobenzol 	31 34	1,5785 1,5700	62—63 83—85 21	Gleichgew.: vielfächige, kurze Prismen. Polymorph II 43°, III 40°, IV 35°, V 28°, VI 27°, VII 22° (20).
51	p-Cyanbenzoesäure-Äthylester 	28 35	1,5204 1,5101	42—43 65—66 23	Hellorange. Gleichgew.: hantelförmige Kristalle, Stengel.
52	Benzalanilin 	38 39	1,6231 1,6126	67—68 88 26	Gleichgew.: Körner. Erstarrt sphärolithisch. Dimorph II 25° (21).
52	Indol 	27 25	1,6011	75—74	Ab 40° Stengel, Prismen. Gleichgew.: gelappte Kristalle. Mit Pikrinsäure rote Verbindung.
52	Salicylsäure-[2-chlorphenylester] 	30 37	1,5785 1,5700	55 77 22	Geruch. Gleichgew. sehr träg: Körner.
52	p-Nitrotoluol 	29 34	1,5299 1,5204	64 83 19	Bittermandelgeruch. Hellgelb. Ab 30° Blättchen, Prismen, Nadeln. Gleichgew.: Balken. Dimorph (12, 22).
52	p-Bromacetophenon 	31 37	1,5611 1,5502	62—64 84—87 23	Geruch. Ab 40° Blättchen. Gleichgew.: Tafeln.
48—52	Tropin 	38 41	1,4936	50—60	Gleichgew. sehr träg: Stengel. Brechungsindex schwankend wegen teilweiser Zersetzung. Hygroskopisch. Zerfließt oft schon unter 40°. In Paraffin F. 52°.
52.5	Pentadecansäure $\text{CH}_3-(\text{CH}_2)_{13}-\text{COOH}$	42 49	1,4339	57	Über 48° Umwandlung. F. meist unscharf, dann Eutektikum 2° tiefer. Gleichgew.: gelappte Platten, Stengel. Enantiotrop dimorph Up. 46° (12, 23).

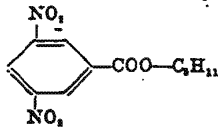
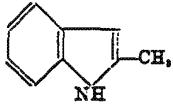
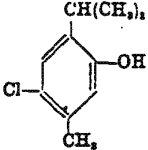
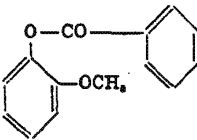
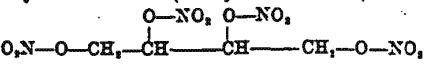
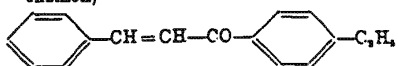
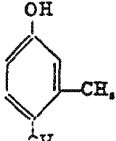
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
53	Dibenzyl 	43 41	1,5502	51	Ab 35° Körner, Blättchen. Gleichgew.: Körner. Schmelze erstarrt sphärolithisch. Dimorph II 50° (sehr unbeständig) (24, 25).
53	4-Nitro-3-oxytoluid 	24 24	1,5700 1,5611	53 72 19	Gelbbraun. Geruch. Gleichgew. trüg: Körner.
53	N-Äthylacetanilid 	31 36	1,5101 1,5000	50—51 72—73 22	Ab 40° Körner. Gleichgew.: Blättchen, Tafeln. Erstarrt sphärolithisch.
53	Maleinsäureanhydrid (= 2,5-Dioxofurandihydrid) 	46 43	1,4584 1,4339	46—48 105—108	Ab 30° Prismen, Nadeln. Gleichgew.: Prismen, Spieße. Erstarrt sphärolithisch.
53,5	Diphenylamin 	33 34	1,6231 1,6128	63—64 82—83 19	Gleichgew.: rechtwinkelige Tafeln. Dimorph II 53° (26).
54	m-Aminobenzonitril 	43 41	1,5795 1,5700	57—58 79—80 22	Häufig oberflächlich violett verfärbt. Ab 40° Tröpfchen. Gleichgew.: Prismen.
54	p-Dichlorbenzol 	30 40	1,5299 1,5204	49—51 69—72 21	Geruch. Ab 40° Spieße und unregelmäßige Blättchen. Gleichgew.: Körner. Enantiotrop dimorph II 53,5°. Up. 29° (27, 12).
54	p-Aminobenzoessäure-β,β-dimethyl-γ-diäthylamino-propylester 	39 43	1,5204 1,5101	69—70 93—95 24	Gleichgew. sehr trüg: Körner, Prismen.
54	Myristinsäure $\text{CH}_3-(\text{CH}_2)_{11}-\text{COOH}$	44 50	1,4339	53	F. meist unscharf, dann Eutektikum 2° tiefer. Gleichgew.: gelappte Platten und Spindeln.
54	p-Chlorjodbenzol 	30 38	1,6126 1,6011	55 76 21	Geruch. Ältere Präparate bräunlich verfärbt. Gleichgew.: Blättchen, Körner.
54	Hydrochinonmonomethyläther 	39 37	1,5433 1,5299	44—47 70—73 26	Ab 40° Tröpfchen, die zu Rhomboiden erstarren. Gleichgew.: Rhomboide, Sechsecke, Stengel.

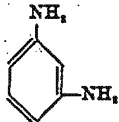
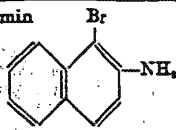
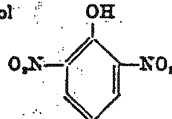
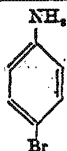
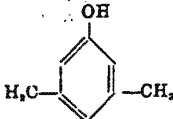
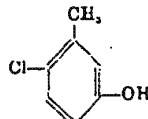
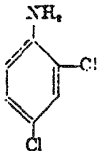
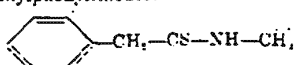
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
54	Carbamidsäuremethylester 	50	50	<1,4339	Ab 40° lange Nadeln, Balken. Gleichgew.: Blättchen, Balken.
54	p-Nitroanisol 	33	37	1,5611 1,5502	68—70 92 Geruch. Ab 35° Nadeln, Blättchen. Gleichgew.: Prismen, Platten. Erstarrt sphärolithisch.
53—55	Trimyristin $\text{H}_2\text{C}-\text{O}-\text{CO}-(\text{CH}_2)_{11}-\text{CH}_2$ $\text{HC}-\text{O}-\text{CO}-(\text{CH}_2)_{11}-\text{CH}_2$ $\text{H}_2\text{C}-\text{O}-\text{CO}-(\text{CH}_2)_{11}-\text{CH}_2$	45	49	1,4339	94—96 Schmelze erstarrt bei langsamem Abkühlen zu feinstrahligen Sphärolithen. Bei raschem Abkühlen feinkörnig.
55	o-Kresolcarbonat 	44	39	1,5203	70—71 Ab 45° Tröpfchen. Gleichgew.: Körner, Balken. Erstarrt sphärolithisch.
55	m-Bromnitrobenzol 	33	35	1,5794 1,5700	54—55 72—73 Geruch. Ab 40° viereckige Blättchen. Gleichgew.: Körner, Blättchen. Erstarrt sphärolithisch. Dimorph (28, 16, 12).
56	Trichloressigsäure CCl_3-COOH	flüssig	flüssig	1,4584	62—63 Stechender Geruch. Zerfließlich. Erstarrt sphärolithisch. Dimorph (29).
56	2-Aminopyridin 	45	43	1,5700 1,5611	64—65 84—86 Geruch. Ab 45° Rauten. Gleichgew.: rechteckige Platten.
56	1,1,2-Triphenyläthan 	34	43	1,5795 1,5611	72—73 84—86 Gleichgew. trüg.: Prismen und Körner. Schmelze erstarrt schwer zu feinen Nadeln.
56	Pyrogallol-1,3-dimethyläther 	36	38	1,5299 1,5204	67—68 88—89 Geruch. Wenn gelbbraun verfärbt, absaugen. Ab 50° wenig Körner. Gleichgew.: Körner, Prismen.

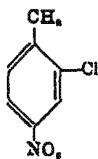
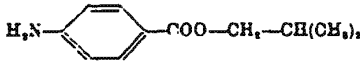
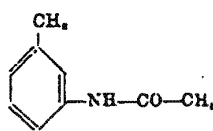
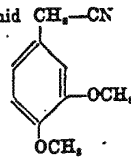
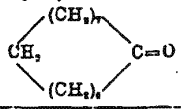
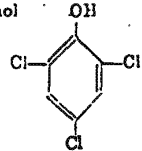
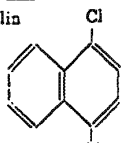
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen	
56	p-Nitrobenzoesäure-äthylester 	41	40	1,5101 1,5204	74—76 51—52 22	Ab 35° Körner, Vierecke, Blättchen. Gleichgew.: Platten, Rechtecke, Prismen.
55—57	Chloralhydrat $\text{CCl}_3\text{—CH(OH)}_2$	43	41	1,4842— 1,4683		Geruch. Ab 35° Körner, Blättchen. Gleichgew.: Körner. Erstarrt zu grobstrahligen Sphärolithen.
57	m-Äthylaminophenol 	45	46	1,5795 1,5700	34—38 60—61 26	Ältere Präparate schwarz verfärbt, Umkristallisation aus Wasser, Aceton und Tierkohle; an Luft und Licht bald bräunlich verfärbt. Ab 40° Tröpfchen. Gleichgew. trüg: Körner, Spindeln. Schmelze bald bräunlich. Unterkühlbar. Bei Impfen sehr träge Kristallisation.
57	m-Nitrobenzaldehyd 	38	49	1,5611 1,5502	67—68 90—92 22	Cremefarben. Gleichgew.: dreieckige Blättchen, dann Spindeln. Dimorph II 51 (10).
57	α-Nitronaphthalin 	34	40	1,6231 1,6126	70 93—94 24	Hellgelb. Ab 40° Stäbchen. Gleichgew.: Stengel und Spindeln. Erstarrt sphärolithisch in zwei Modifikationen: II 52° derbestrahlig, I feinstrahlig (28, 16, 12).
57	1,4-Dimethoxybenzol (= Hydrochinon-dimethyläther) 	34	42	1,5000 1,4936	69—71 82—84	Ab 30° Platten, Sechsecke, Spieße. Gleichgew.: Platten, Spieße. Erstarrt in Platten oder Strahlen.
57	Benzalacetophenon 	37	42	1,6353 1,6231	65—75 85—88 16	Ab 45° Körner. Schmilzt meist sehr trüg. Erstarrte Schmelze zwischen gekreuzten Nicols blau.
57	p-Anisidin (= p-Aminophenolmethyläther) 	42	42	1,5611 1,5502	48—49 70—71 22	Ab 50° vereinzelte Körner, Gleichgew.: Prismen. Erstarrt sphärolithisch. Dimorph II 55° (30).
57	Benzoylacetone 	36	43	1,5700 1,5611	61—63 80—81 12	Geruch. Ab 45° Tröpfchen. Gleichgew.: Nadeln. Erstarrt sphärolithisch.
58	Salicylaldoxim 	36	38	1,5795 1,5700	68—70 92—94 24	Geruch. Gleichgew.: Prismen und unregelmäßige Kristalle. Erstarrt sphärolithisch.

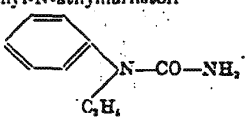
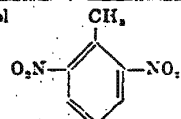
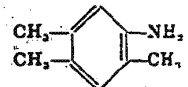
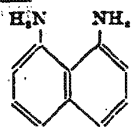
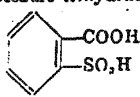
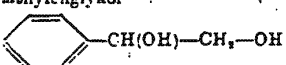
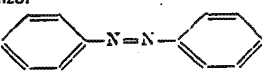
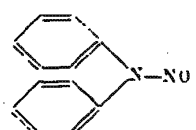
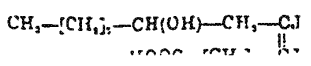
F.°C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
59	5-Methylindol 	34 32	1,5897 1,5795	60 84 2.4	Geruch. Ab 30° Nadeln, Stäbchen. Gleichgew.: schwach anisotrope Nadeln und Balken. Schmelze erstarrt sphärolithisch.
59	Cardiazol (= Pontamethylantetrazol) 	34 36	1,4936 1,4842	60—63 91—93	Ab 45° Tröpfchen. Gleichgew.: sechseckige Blättchen oder Prismen.
59	Scopolamin inakt. + H ₂ O 	37 39	1,5299	60—65	Schmilzt und kristallisiert sehr trög. Brechungsindex durch Wasserabgabe erschwert.
59	Acetophenonoxim 	34 42	1,5502 1,5403	77—79 98—100 2.1	Ab 30° Tröpfchen. Gleichgew.: Balken. Erstarrt zu Sphärol. I. und II. Letzt. werden sofort umgewandelt.
59	dl-α-Bromstearinsäure $\text{CH}_3-(\text{CH}_2)_{11}-\text{CHBr}-\text{COOH}$	34 tr. 40	1,6353 1,6231	67 87 2.5	Gleichgew.: Balken. Erstarrt sphärolithisch. Polymorph II 66°, III 62° (30).
59	p-Nitrophenetol 	33 42	1,5403 1,5299	77—79 98—99 2.1	Geruch. Ab 40° Körner. Gleichgew.: Tafeln, Prismen. Erstarrt zu Blättern und Tafeln.
59	Octadecylalkohol $\text{CH}_3-(\text{CH}_2)_{16}-\text{CH}_2-\text{OH}$	50 57	1,4339	69—70	Gleichgew.: Nadeln und schwach anisotrope Blättchen. Enantiotrop dimorph. Up. 48° (31, 12).
59	o-Oxydiphenyl 	30 31	1,6011 1,5897	63—64 84—85 2.1	Ab 45° Nadeln und Körner. Gleichgew.: Platten. Erstarrt zu Sphärolithen.
59	Novatophan K (= 2-Phenylcinchoninsäure-methylester) 	34 43	1,6463 1,6353	63—64 91—93 2.2	Gleichgew. trög: Körner. Schmelze erstarrt sehr langsam zu Sphärolithen mit F. 61,5° (32).
59	Benzoylmandelsäurenitril 	40 45	1,5502 1,5403	58—60 80—82 2.2	Gleichgew.: Stengel und Prismen.

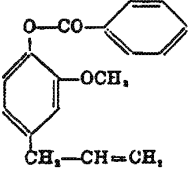
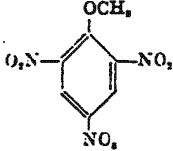
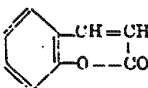
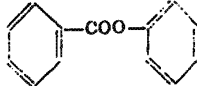
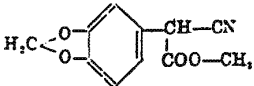
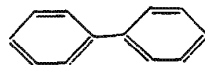
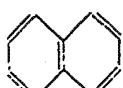
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen	
55—59 (55—75)	Cocainnitrat 	39	46	1,5204	76—77	Schmilzt trög. Gleichgew.: Rechtecke. In einzelnen Tropfen fallen sekundär kurze Nadeln aus. Manchmal schon Zersetzung bei Erwärmen und F. 55° bis 75°; in Paraffin bleibt F. 55° bis 59°.
59,5 (62,5)	Voluntal (=Carbamidsäure-[β,β,β-trichlor-äthylester]) $\text{H}_2\text{N}-\text{CO}-\text{OCH}_2-\text{CCl}_3$	46	45	1,4842	51—54	Ab 45° winzige vierseitige Kristalle. Gleichgew.: Rechtecke der instabilen Modifikation 59,5°, Stengel der stabilen Modifikation 62,5°. Polymorph II 60°, III 59,5° (33).
59,5	Hippursäureäthylester 	47	48	1,5101 1,5000	78—79 102—104	Gleichgew.: Prismen. Erstarrt langsam zu feinstrahligen Sphärolithen.
55—60	α,α,β-Trichlorbuttersäure $\text{CH}_3-\text{CHCl}-\text{CCl}_2-\text{COOH}$	flüssig	flüssig	1,4683 1,4584	67 90	Geruch. Schmilzt trög. Schmelze erstarrt zu Sphärolithen.
60	N-Phenyl-α-naphthylamin 	34	37	1,6715 1,6598	69 91	Geruch. Wenn rotbraun verfärbt, absaugen. Schmilzt meist unscharf. Gleichgew.: Körner, Stengel. Schmelze unterkühlbar.
60	3,4-Dinitrotoluol 	35	32	1,5502 1,5403	49—50 73—74	Gleichgew. Prismen. Erstarrt sphärolithisch.
60	β-Chlornaphthalin 	29	37	1,6011	71—72	Geruch. Ab 35° Blättchen. Gleichgew.: Platten. Erstarrt zu flächigen Aggregaten.
60	2,6-Dimethylninolin 	32	40	1,5795 1,5700	64—66 85—88	Unreine Präparate oft gelb. Reinigen durch Absaugen, größere Kristalle ev. mit Alkohol abwaschen. Ab 30° Körner. Gleichgew.: Stengel. Erstarrt spontan zu II (bunt); bei Erwärmen Umwandlung in I (grau oder gelb) oder F. II 53° (Gleichgew.: Körner, Prismen).
60	Tolan (= Diphenylacetylen) 	53	43	1,6231 1,6126	72 88—91	Ab 50° Körner. Gleichgew.: Körner.
55—60	Kodein + H ₂ O 	55 tr.	55 tr.	vgl. Kodein wasserfrei 156°		Beim Schmelzen scheiden sich wasserfreie Kristalle ab. In Paraffin ab 52° inhomogenes Schmelzen und Abscheidung wasserfreier Kristalle oder homogenes Schmelzen der Hydratkristalle bei 80°.

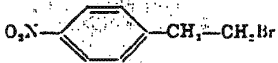
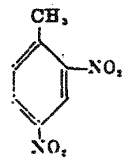
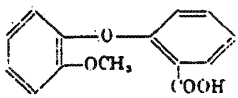
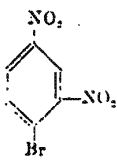
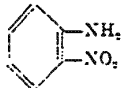
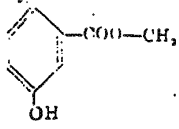
F. °C	Substanz	Eutekt. Temp. mit		Glas-	Temp.	Besondere Kennzeichen
		Azobenzol	Benzil	pulver	°C	
60	3,5-Dinitrobenzoesäure-isoamylester 	31	42	1,5204 1,5101	47 80 33	Schmelze erstarrt zu breitstrahligen Aggregaten.
60	2-Methylindol 	38	38	1,6011 1,5897	35 80 45	Geruch. Häufig rötlich verfärbt. Absaugen oder Adsorption durch Al ₂ O ₃ mit Chloroform. Gleichgew.: Platten, Prismen. Erstarrt sphärolithisch.
61	4-Chlorthymol 	33	34	1,5204 1,5101	67-68 89-91 27	Geruch. Ab 45° wenig Blättchen, Körner. Gleichgew.: Körner, Prismen.
61	Benzosol (= Guajacolbenzoat) 	38	43	1,5502 1,5403	80 105-107 26	Ab 45° kleine Stäbchen. Gleichgew.: Körner. Erstarrt zu strahligen grauen Aggregaten, die sich beim Erwärmen teilweise buntfleckig umlagern.
61	Erythroltetranitrat (= Erythrittetranitrat) 	57	52	1,4683	60-63	Gleichgew.: Körner, Stengel, Balken. In der Schmelze Tropfenbildung. Erstarrt sphärolithisch.
61	Carbamidsäurepropylester $H_2N-COO-C_2H_5$	50	52	<1,4339		Ab 40° Nadeln. Gleichgew.: Nadeln, Balken. Erstarrt sphärolithisch. Dimorph. Mit zehn Prozent Novatophan $n = 1,4339$ bei 83-86°.
61	Acetoxim (= Acetonoxim) $(CH_3)_2C=N-OH$	41	50	<1,4339		Ab 25° reichlich Körner, Sechsecke, später wabenartig. Gleichgew.: Körner, Prismen. Erstarrt strahlig.
61,5	Heptadecansäure $CH_3-(CH_2)_{15}-COOH$	61	59	1,4339	65	Über 56° Umwandlung. F. meist unscharf, dann Eutektikum 2° tiefer. Gleichgew.: gelappte Platten; Brechungsindex durch Erstarren erschwert.
62	Monochloressigsäure $CH_2Cl-COOH$	46	41	1,4339	55-56	Ab 40° schön ausgebildete Rauten. Gleichgew. sehr trüg: Körner. Polymorph (22, 12).
62,5	p'-Äthylbenzalacetophenon (= p'-Äthylchalkon) 	39	45	1,6126 1,6011	74-75 98-100 24	Gleichgew.: Platten, Prismen.
62,5	o-Xylenol (= 3,4-Dimethylphenol) 	42	43	1,5203 1,5101	71-72 97-98 24	Geruch. Ab 45° Körner. Gleichgew.: Körner, Prismen. Schmelze erstarrt palmwedelartig oder sphärolithisch.

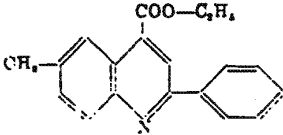
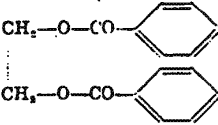
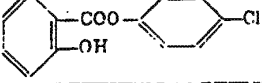
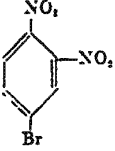
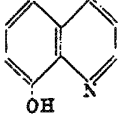
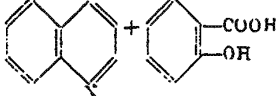
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen	
63	m-Phenylendiamin 	59	55	1,6231 1,6126	70—77 105 25	Ab 45° feine Tröpfchen. Gleichgew.: Prismen. Luft- und lichtempfindlich; Verfärbung.
63	1-Brom-2-naphthylamin 	38	38	1,6715 1,6598	95—96 119—121 24	Ab 40° Nadeln. Gleichgew.: Nadeln, Balken. Erstarrt sphärolithisch.
63	Palmitinsäure $\text{CH}_3-(\text{CH}_2)_{14}-\text{COOH}$	62	60	1,4339	62	F. meist unscharf, dann Eutektikum etwa 2° tiefer. Gleichgew.: Rauten, gelappte Platten. Brechungsindex durch Erstarren erschwert.
63,5	2,6-Dinitrophenol 	43	44	1,6011 1,5997	59—61 82—83 23	Gelb. Tröpfchen. Schmelze erstarrt sphärolithisch. Dimorph II 54° (32).
64	p-Bromanilin 	39	37	1,6011 1,5897	66—68 90—92 24	Ab 50° Körner. Gleichgew.: Rechtecke, Rhomboide, Prismen.
64	m-Xylenol (= 3,5-Dimethylphenol) 	41	40	1,5101	79—80	Ab 50° Nadeln. Gleichgew.: Stengel. Dimorph. Wenige Sekunden nach Erstarren der Schmelze auch makroskopisch sichtbare Umwandlung. II 55° (vgl. Übungsbeispiele) (10).
64	Carbamidsäureisocamylester $\text{H}_2\text{N}-\text{COO}-\text{CH}_2-\text{CH}_2-\text{CH}(\text{CH}_3)_2$	47	52	<1,4339		Wenn gelblich verfärbt, absaugen. Ab 45° schwach anisotrope, gelappte Blättchen und Stengel. Gleichgew.: Platten. 1 Teil Benzil und 2 Teile Carb. mit 1,4584 bei 91—93°.
64	6-Chlor-m-kresol 	37	31	1,5299 1,5204	106—108 117—119 "	Geruch. Ab 45° einzelne Körner. Gleichgew.: Körner.
64	2,4-Dichloranilin 	39	40	1,5700 1,5611	86—88 107—109 21	Ab 40° Nadeln und Balken. Gleichgew.: Balken. Erstarrt sphärolithisch.
64	N-Methylphenylthioacetamid 	47	49	1,6011 1,5997	75—77 99—102 25	Ab 50° Tröpfchen. Gleichgew.: Prismen, Balken. Dimorph.

F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
65	2-Chlor-4-nitro-toluol 	33	44	1,5502 1,5403	64 — Ab 45° Stengel, Körner. Gleichgew.: 84 — Balken. 2°
65	Cycloforin (= p-Aminobenzoesäure- isobutyl ester) 	44	45	1,5403 1,6290	65—66 ! Ab 50° Körnchen, Nadeln und Blätt- 88—89 chen. Gleichgew.: Körner und Blätt- 23 chen. Schmelze erstarrt zu Sphäro- lithenmosaik.
65	Acet-m-toluidid 	43	47	1,5403	71—72 ! Ab 40° Tröpfchen, wenig Körnchen. Gleichgew. trög: rundliche Blättchen. Polymorph II 51° (Gleichgew.: spitze Rauten). Schmelze unterkühlbar. Ge- ringe Kristallisationsgeschwindigkeit u. Keimzahl.
62—65	Tripalmitin $\begin{array}{l} \text{H}_2\text{C}-\text{O}-\text{CO}-(\text{CH}_2)_{14}-\text{CH}_3 \\ \\ \text{H}-\text{C}-\text{O}-\text{CO}-(\text{CH}_2)_{14}-\text{CH}_3 \\ \\ \text{H}_2\text{C}-\text{O}-\text{CO}-(\text{CH}_2)_{14}-\text{CH}_3 \end{array}$	52	56	1,4330	95—98 Schmilzt meist unscharf. Erstarrt fein- körnig.
65	3,4-Dimethoxybenzylcyanid 	47	51	1,5204 1,5101	70—71 Ab 45° Nadeln, Stengel. Gleichgew.: 95—96 Balken. Erstarrt sphärolithisch. 25
65	Exalton (= Cyclopentadecanon) 	20	35	1,4584	78—80 Geruch. Ab 30° Tröpfchen. Gleichgew.: rundliche Körnchen oder gelappte Blättchen. Schmelze erstarrt strau- pflasterartig.
66	2,4,6-Trichlorphenol 	29	35	1,5700 1,5611	86—88 Phenolartiger Geruch. Ab 40° Nadeln, 85 Stengel. Gleichgew.: Spieße, Prismen, 18 Stengel. Schmelze erstarrt sphäro- lithisch. Polymorph.
66	Neuronal (= Diäthylbromacetamid) $\begin{array}{l} \text{C}_2\text{H}_5 \\ \\ \text{C}_2\text{H}_5 > \text{CBr}-\text{CO}-\text{NH}_2 \end{array}$	45	49	1,4842 1,4683	83 Geruch. Ab 50° Blättchen. Gleichgew.: 99 Körner. Erstarrt sphärolithisch.
66	1,4-Dichlornaphthalin 	40	53	1,6231 1,6126	64—66 Ab 50° Nadeln, Körner. Gleichgew.: 87—89 Nadeln, Balken. Erstarrt sphärolithisch. 23

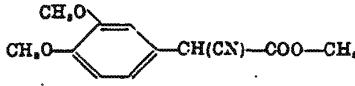
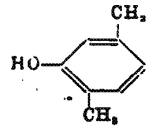
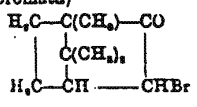
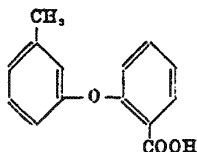
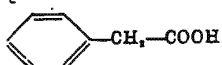
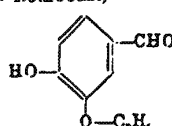
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
66	N-Phenyl-N-äthylharnstoff 	39 46	1,5299 1,5204	74—75 101—102 27	Gleichgew.: Körner. Schmelze erstarrt sphärolithisch.
66	Carbamidsäureisobutylester $H_2N-COO-CH_2-CH(CH_3)_2$	51 55	<1,4339		Ab 35° Blättchen, Spieße. Gleichgew.: Blättchen, Platten. Erstarrt sphärolithisch. Brechungsindex von 1 Teil Benzil und 2 Teilen Carb. mit 1,4584 bei 54—80°.
66	2,6-Dinitrotoluol 	41 42	1,5502 1,6403	55—58 79—81 23	Ab 55° Körner, Stäbchen. Gleichgew.: Prismen, Körner. Erstarrt palmwedelartig. Enantiotrop dimorph II 58°, Up. 38° (16, 14).
66	Pseudocoumidin (= 2, 4, 5-Trimethylanilin) 	41 46	1,5299 1,5204	77—78 90—100	Stechender Geruch. Ab 40° Nadeln. Gleichgew.: Nadeln. Polymorph II 62°, III 61° (30).
66	1,8-Naphthylendiamin 	43 40	1,6877	76—80	Häufig braun verfärbt. Umkristallisieren aus verdünntem Alkohol mit Aktivkohle. Gleichgew.: Balken. Schmelze bald bräunlich. Erstarrt strahlig.
64—67	o-Sulfohensäure-trihydrat  + 3 H ₂ O	55 tr. 60 tr.	1,5101	90—95	Schmilzt trög zwischen 64° und 67°. Gleichgew.: Sechsecke, Rauten, Prismen. Manchmal später wasserfreie Stengel. F. 135° (s. dort).
67	Phenyläthylenglykol 	57 tr. 60	1,5299 1,5204	76—77 99—100 22	Gleichgew.: Balken. Schmelze zäh. Erstarrt zu Sphärolithen.
68	Azobenzol 	68 50	1,6483 1,6353	51 76 25	Orangerot. Ab 45° Rauten. Gleichgew.: Körner, Balken.
68	Diphenylnitrosamin 	40 46	1,5897 1,5795	78—79 102—103 21	Gelb. Aromatischer Geruch. Ab 53° wenig Körner. Gleichgew.: Körner, Balken.
68	Dijodyl (= Ricinostearolsäuredijodid) $CH_3-(CH_2)_{17}-CH(OH)-CH_2-CI$ 	55 63	1,5299 1,5204	84—86 105—107 21	Substanz feinnadelig. Gleichgew.: Nadeln. Schmelze unterkühlbar. Nach Kratzen des Deckglasrandes zuerst feine Nadeln, dann träge Kristallisation zu

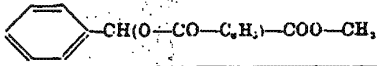
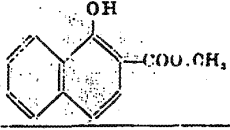
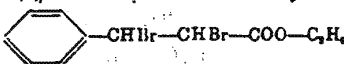
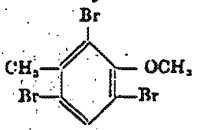
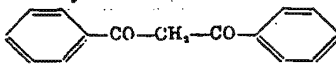
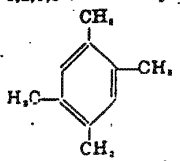
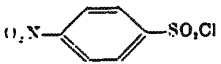
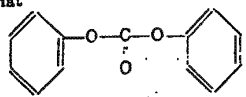
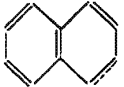
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
68	Eugenolbenzoat 	41	51	1,5403 1,5299	92—93 114—115 22 Gleichgew.: Körner, Prismen. Erstarrt sphärolithisch.
68,5	2,4,6-Trinitroanisol 	48	48	1,5700 1,5611	70—71 84 23 Hellgelb. Gleichgew.: kurze Prismen.
68,5	Nonadecansäure $\text{CH}_3-(\text{CH}_2)_{17}-\text{COOH}$	56	85	1,4339	73 Über 64° Umwandlung. F. meist un- scharf. Gleichgew.: Stengel und Platten. Enantiotrop dimorph (34, 12).
69	Cumarin 	44	47	1,6011	63—64 Heugeruch. Gleichgew.: viereckige Blättchen. In Sublimaten oft auch Blättchen mit F. 64,5°. Polymorph II 64,5°, III 55° (35, 10).
69	Benzoesäure-phenylester 	43	48	1,5502 1,5403	79—80 101—102 22 Gleichgew.: abgeschrägte Stengel. Poly- morph II 56,5°, III (10).
69	3,4-Methylenoxyphenyl-cyanessigsäure- methylester 	50	55	1,5101 1,5000	75—76 98—97 Ab 60° Tröpfchen. Schmelze unterkühl- bar.
67—70	Helenin (= Alantolnaton) $\text{C}_{14}\text{H}_{22}\text{O}_2$	39	42	1,5101 1,5000	77—78 103—105 24 Geruch. Schmilzt sehr trög. Schmelze unterkühlbar.
70	Diphenyl 	38	48	1,5785	84—85 Ab 50° lappige Kristalle und Prismen. Gleichgew.: gelappte Platten.
70	α,α-Dipyridyl 	38	46	1,6011 1,5897	54 77—79 21 Ab 45° Körner, Prismen. Gleichgew.: Prismen und Körner.

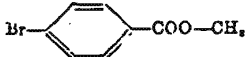
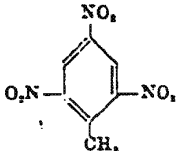
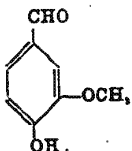
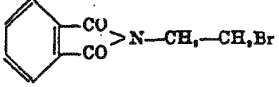
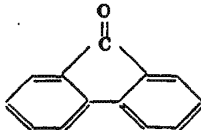
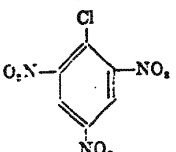
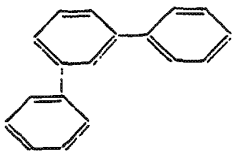
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen	
70	2-[p-Nitrophenyl]-1-bromäthan 	41	48	1,5700 1,5611	69—70 88—89 19	Ab 55° Tröpfchen. Gleichgew.: Prismen.
70	Stearinsäure $\text{CH}_3-(\text{CH}_2)_{16}-\text{COOH}$	57	67	1,4339	69—70	Meist F. unscharf und Eutektikum etwa 2° tiefer. Gleichgew.: gelappte Platten. Brechungsindex durch Er- starren erschwert.
71 (78)	Thiosinamin (= Allylthioharnstoff) $\text{CH}_2=\text{CH}-\text{CH}_2-\text{NH}-\text{CS}-\text{NH}_2$	66	64	1,6011 1,5898	68—69 97—98 29	Gleichgew.: Prismen. Erstarrt meist zu stabiler Modifikation mit F. 78°. Enan- tiotrop polymorph II 78°, III 70° (33).
71	p-Chloranilin 	42	42	1,5811 1,5502	89—90 107—110 19	Geruch. Ab 40° Körner. Gleichgew.: Körner, Prismen.
71	2,4-Dinitrotoluol 	39	46	1,5502 1,5403	85—86 106—108 21	Ab 54° Körner, Stäbchen. Gleichgew.: Balken. Erstarrt sphärolithisch.
72	(= Salicylsäure-guajacoläther) 	41	50	1,5611 1,5502	79 100—102 22	Ab 55° Körnchen. Gleichgew.: Körner. Schmelze erstarrt sphärolithisch.
72	4-Brom-1,3-dinitrobenzol 	42	49	1,5897 1,5795	91—93 113—116 23	Gelb. Ab 50° wenig Körner. Gleichgew.: Balken, Körner. Erstarrt sphärolithisch. Polymorph II 51°, III 46°, IV 41°, V 36° (20).
72	o-Nitroanilin 	50	50	1,6353 1,6231	92—94 115—114 21	Gelb. Ab 60° spärlich Nadeln. Gleich- gew.: Prismen, Balken. Erstarrt zu Sphärolithen. Dimorph II 68° (14).
72	m-Oxybenzoesäure-methylester 	52	53	1,5299 1,5204	79 103 24	Ab 65° winzige Blättchen, Körner und Stäbchen. Gleichgew.: Körner, Prismen. Erstarrt sphärolithisch. Dimorph II 66°.
73	Nerolin (= β-Naphtholmethylether) 	45	52	1,5897 1,5795	79—80 101—102 22	Blütengeruch. Ab 50° unregelmäßige Blättchen. Erstarrt zu grobstrahligen Sphärolithen.

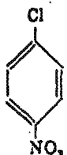
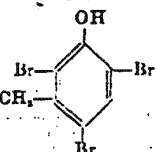
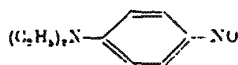
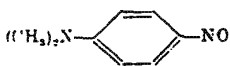
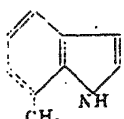
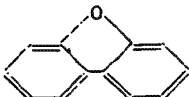
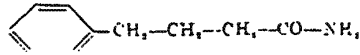
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
73	Diphenylcarbamidsäure-äthylester (= Diphenylurethan) $(C_6H_5)_2N-COO-C_2H_5$	44	51	1,5502 1,5403	65—66 87—88 Ab 55° Tröpfchen. Gleichgew.: Körner, Prismen. Erstarrt sphärolithisch. Dimorph II 66°.
73	Novonal (= Diäthylallylacetamid) $(CH_2=CH-CH_2-C(C_2H_5)_2-CO-NH_2)$	52	60	1,4584	76—77 Ab 50° feine Nadeln. Gleichgew.: Nadeln und Stengel
73	Dekamethylenglykol $HOCH_2-(CH_2)_8-CH_2OH$	64	67	1,4339	101—103 Ab 55° Tröpfchen. Gleichgew. trög: Stengel und Prismen.
73	p-Dimethylamino-benzaldehyd $(CH_3)_2N-C_6H_4-CHO$	42	49	1,6231	82—84 Ab 70° Tröpfchen. Gleichgew.: sechs- und vierckige, dann vielseitige Tafeln.
71—73	0-Methyl-2-phenylchinolin-4-carbonsäure-äthylester (= Neocinchophen) 	41	55	1,6231 1,6128	67—68 87—88 Schmelze erstarrt glasig, beim Erwärmen nur teilweise kristallin.
73	Glykoldibenzoat 	46	55	1,5289 1,5204	81—82 103—105 Gleichgew.: Balken, Platten.
73	Salicylsäure [4-chlorphenylester] 	40	50	1,5795 1,5700	64—65 87—88 Geruch. Ab 60° Stäbchen. Gleichgew.: Balken und Stengel.
73	4-Brom-1,2-dinitrobenzol 	36	36	1,6011 1,5897	69—70 83 Aromatischer Geruch. Gleichgew.: Körner, Prismen. Dimorph (20).
74	8-Oxychinolin 	47	54	1,6131	82—84 Geruch. Ab 50° Nadeln. Gleichgew.: derbe Balken. Polymorph II 65°, III 58°, IV 39,5° (36).
70—74	Chinolinosalicylat 	47	56	1,5897— 1,6011	— Geruch. Ab 50° Nadeln. Während des Schmelzens Kristalle bräunlich trüb. Schmelze farblos.

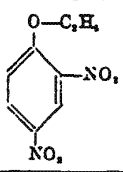
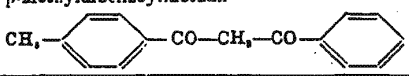
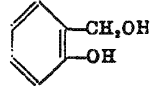
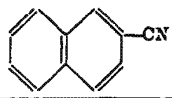
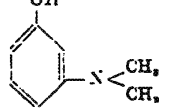
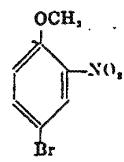
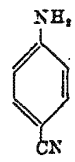
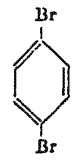
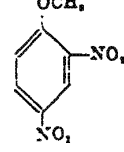
F. °C	Substanz	Entsch. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
74	Pellidol (= 4'-Diacetylamino-2,3'-dimethyl- azobenzol)	43	52	1,8011 1,5897	85—86 111—112 26 Gelbrot. Gleichgew.: Körner. Schmelze braungelb.
74	Formyldiphenylamin	42	47	1,5795 1,5700	90—91 110—111 26 Geruch. Ab 60° Nadeln, Blättchen. Gleichgew.: Stengel, Balken.
74	p-Aminobenzoesäure-propylester (= Propäsin)	50	54	1,5000 1,5504	93—94 71—74 Ab 70° Tröpfchen. Gleichgew.: Prismen. Erstarrt sphärolithisch.
74	Pentabromacetone CHBr5-CO-CBr5	37	47	1,6131	70 Ab 60° Tröpfchen. Gleichgew.: Körner, kurze Prismen. Erstarrt sphärolithisch.
74	p,p'-Dioxy-ditandekamethylenäther	41	55	1,5299 1,5204	79—81 109—110 30 Gleichgew.: Stengel und Platten. Er- starrt zu Sphärolithen.
74	3,5-Dinitrobenzoesäure-propylester	37	50	1,5204 1,5101	73 97—98 25 Gleichgew.: Prismen und Stengel.
74,5	Äthantetracarbonsäure-tetraäthylester	51	57	<1,4339	Ab 70° Nadeln. Gleichgew.: Stengel und Spieße.
74,5	Phthalid	50	54	1,5403 1,5299	84—86 101—103 17 Geruch beim Erhitzen. Ab 60° Körner. Blättchen. Gleichgew.: Körner, Pris- men. Erstarrt sphärolithisch. Dimorph: II 66° (18, 28, 12).
75	1-[p-Methoxyphenyl]-1-phenyläthylen	45	57	1,5700 1,5611	96—98 116—118 20 Ab 60° wenig Körner. Gleichgew.: Tafeln. Erstarrt sphärolithisch.
75	p-Nitrobenzoylchlorid	32	42	1,5502— 1,5011	Geruch. Ab 40° Körner. Stäbchen. Gleichgew.: Prismen, Spieße.

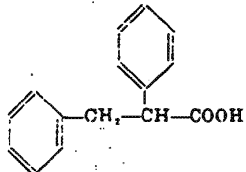
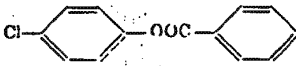
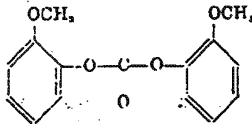
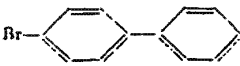
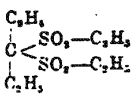
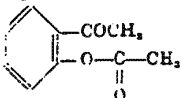
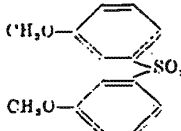
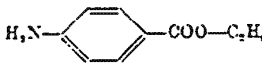
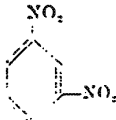
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen	
75	Josen (= α -Dihydrophylloladen) Tetraacycl. Diterpen $C_{20}H_{32}$	48	68	1,5000 1,4936	76 95	Ab 50° Tröpfchen. Gleichgew. träg: Körner, Prismen.
75	3,4-Dimethoxyphenyl-oxanessigsäure- methylester 	54	59	1,5000 1,4936	84—86 97—98	Ab 60° Kristallgrieß. Gleichgew.: Pris- men, Körner. Erstarrt erst nach länge- rem Liegen zu Sphärolithen.
75	p-Xylenol (= 2,5-Dimethylphenol) 	47	49	1,5101 1,5000	78—80 97—98	Geruch. Ab 40° Nadeln und Spieße. Gleichgew.: Balken, Spieße. Erstarrt sphärolithisch.
75,5	Arachinsäure $CH_3-(CH_2)_8-COOH$	60	73	1,4339	77	F. meist unscharf. Gleichgew.: gelappte Platten, Spieße.
76	Trional (= Methylsulfonal) $CH_3-C(SO_2-CH_3)_2$	49	55	1,4840 1,4883	58—62 103—105	Ab 65° rechtwinkelige Blättchen. Gleich- gew.: Rechtecke. Dimorph II 56° (wird durch Stößen induziert) (8).
76	3-Brom-d-campher (= Camphora monobromata) 	39	49	1,4936 1,4842	71—72 96—98	Campher-Geruch. Ab 65° Spieße, Sten- gel. Gleichgew.: abgeschrägte Stengel und Spieße.
76	Compral (= Pyramidon + Volantal) $C_{11}H_{17}ON_3 + NH_4-COO-CH_2-CCl_3$	57	58	1,5299 1,5204	67 93—94 27	Gleichgew.: Prismen. Erstarrt glasig.
76	m-Kresolol (= Salicylsäure-m-kresoläther) 	45	55	1,5611 1,5502	63—70 90—91 22	Ab 50° Körner, Nadeln. Gleichgew. Platten. Erstarrt sphärolithisch.
76,5	Tribenzoin (= Glycerintribenzoat) $H_2C-O-CO-C_6H_5$ $H-C-O-CO-C_6H_5$ $H_2C-O-CO-C_6H_5$	47	58	1,5302 1,5403	66—68 90—92 24	Gleichgew. sehr träg: Platten, Körner. Erstarrt glasig.
77	Phenyllessigsäure 	48	57	1,5000 1,4936	95—98 110—112	Geruch. Ab 60° gelappte Blättchen. Gleichgew.: große Rauten, Sechsecke und Stengel.
77	4-Oxy-3-äthoxybenzaldehyd (= „Äthyl- vanillin“, = Bourbonal) 	52	58	1,5611 1,5502	75 96 21	Vanillegeruch. Ab 55° Blättchen, Stäb- chen. Gleichgew.: Prismen, Körner. Erstarrt körnig. Polymorph II 74°, III 65°, IV 60° (37).

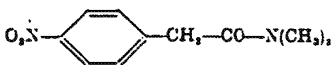
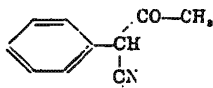
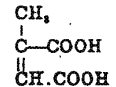
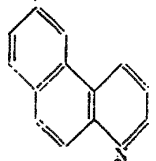
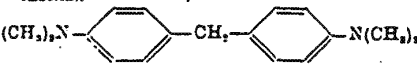
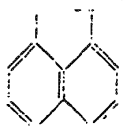
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
77	Benzoylmandelsäure-methylester 	60 57	1,5299 1,5204	78—79 99—100 21	Ab 60° Tröpfchen und wenige Körnchen. Gleichgew.: Prismen, Körner. Erstarrt glasig, bei Erwärmen große Kristalle.
78	Lactamid (= Milchsäureamid) $\text{CH}_3\text{—CHOH—CO—NH}_2$	66 73	1,4584	69	Geruch. Ab 60° Tröpfchen. Gleichgew.: Körner, Prismen. Erstarrt zu Sphärolithen.
75	1-Oxynaphthoesäure-2-methylester 	45 57	1,0011 1,5897	81—83 108—109 27	Ab 55° wenig Körner. Gleichgew.: Platten, Körner. Dimorph.
78	dl,α,β-Dibromhydrozimsäure-äthylester 	47 58	1,5204 1,5101	118—120 139—141 21	Gleichgew.: Blättchen, Tafeln. Erstarrt zu Sphärolithen. Dimorph.
78	2,4,6-Tribrom-3-methoxytoluol 	42 61	1,0011 1,5897	76—77 99—101 23	Ab 50° Körner, Nadeln. Gleichgew.: Stengel.
78	Dibenzoylmethan 	55 57	1,6353	99—100	Gleichgew.: Prismen, Platten. Erstarrt zu gefiederten Sphärolithen. Dimorph II 71° (38).
79	Propionamid $\text{CH}_3\text{—CH}_2\text{—CO—NH}_2$	66 71	<1,4339		Ab 60° große, übereinandergelagerte Platten. Gleichgew.: große, dünne Platten.
80	Durol (= 1,2,4,5-Tetramethylbenzol) 	46 64	1,4683 1,4842	108—110 78—80	Ab 35° Körner, Prismen; reichlich Sublimat. Gleichgew.: Prismen, Platten. Erstarrt strahlig.
80	p-Nitrobenzolsulfonchlorid 	43 49	1,5611 1,5302	72—73 96 23	Geruch. Gleichgew.: Prismen, Balken.
80	Hedonal (= Methylpropylcarbinol-carbamate) $\text{C}_2\text{H}_5\text{—CH—O—CO—NH}_2$ CH_3	53 63	<1,4339		Ab 50° Nadeln, Stengel. Gleichgew.: Nadeln, Stengel.
80	Acetamid $\text{CH}_3\text{—CO—NH}_2$	66 77	1,4339	58	Geruch. Ab 50° kurze Prismen, Nadeln. Stengel. Gleichgew.: Prismen, Balken. Dimorph II 70° (28, 12).
80	Diphenylcarbonat 	49 56	1,5299 1,5204	68—69 92—93 24	Geruch. Ab 50° Nadeln. Gleichgew.: Balken, Spieße. Erstarrt fächerförmig.
80,5	Naphthalin 	42 52	1,5897 1,5795	79 100—102 22	Geruch. Ab 45° schiefwinkelige Blättchen, Spieße. Gleichgew.: Körner und Prismen (vgl. Übungsbeispiele).

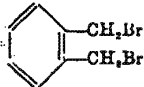
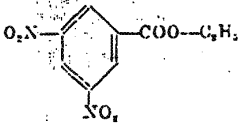
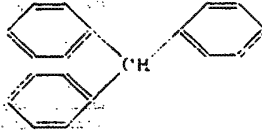
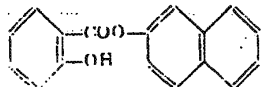
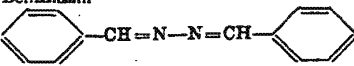
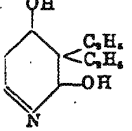
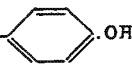
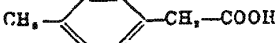
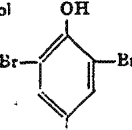
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
81	α -Naphthylurethan (= α -Naphthyl- carbamidsäureäthylester) <chem>NC(=O)OCC</chem> 	53 50	1,5705 1,5700	84-85 104-106 20	Ab 75° Körner, Nadeln. Gleichgew. trüg: Prismen. Erstarrt sphärolithisch. Poly- morph II 80°, III 78° (30).
81	p-Brombenzoesäure-methylester <chem>BrC1=CC=C(C=C1)C(=O)OC</chem> 	48 56	1,5290 1,5204	85-87 105-106 20	Geruch nach Aas. Ab 50° Stäbchen, Spicße. Gleichgew.: Balken.
81	2,4,6-Trinitrotoluol (= Trötyl) <chem>Cc1c([N+](=O)[O-])cc([N+](=O)[O-])cc1[N+](=O)[O-]</chem> 	(51) (47)	1,5811 1,5502	91-93 114-115 23	Ab 70° Tröpfchen, wenige Stäbchen. Gleichgew.: Prismen. Erstarrt sphäro- lithisch.
81.5	Vanillin <chem>COc1cc(O)ccc1C=O</chem> 	55 64	1,5700 1,5611	94-98 114 13	Geruch. Ab 70° Sechsecke, Prismen. Gleichgew.: Körner, Prismen.
83	N-[p-Bromäthyl]-phthalimid <chem>BrCCN1C(=O)c2ccccc2C1=O</chem> 	47 tr. 58	1,5700 1,5611	93-94 114-116 21	Ab 60° kleine Körner, vereinzelte Nadeln. Gleichgew. trüg: Prismen, Körner, Platten. Erstarrt sphäroli- thisch.
83	Fluorenon <chem>O=C1c2ccccc2c3ccccc13</chem> 	53 55	1,6353 1,6231	93-97 118-121 24	Gelb. Ab 75° Tröpfchen. Gleichgew.: sechseckige Blättchen. Polymorph II 72°, III 54°, IV 52° (32).
83	2,4,6-Trinitrochlorbenzol (= Pikrylchlorid) <chem>Clc1c([N+](=O)[O-])cc([N+](=O)[O-])cc1[N+](=O)[O-]</chem> 	45 51	1,5795 1,5700	91-92 114-115 23	Ab 70° Körner. Gleichgew.: Prismen, Platten. Polymorph II 81°, III 60°, IV 60°, V 58°, VI 56° (39, 40).
83	1,3-Diphenylbenzol <chem>c1ccc(cc1)-c2ccccc2-c3ccccc3</chem> 	40 50	1,6353 1,6231	77-79 102-104 25	Ab 65° wenig Nadeln. Gleichgew.: Nadeln, Stengel. Wenn Rückstand, umkristallisieren aus Alkohol. Erstarrt in zweierlei Sphärolithen: I strahlig, II blättrig mit F. 65-68°.

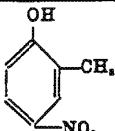
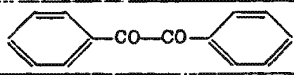
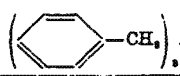
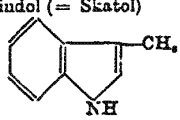
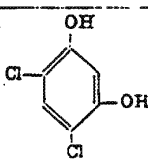
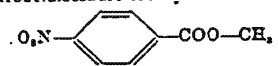
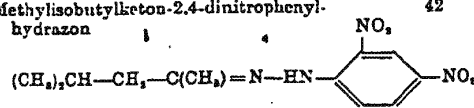
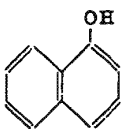
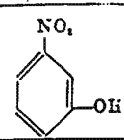
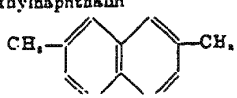
F.°C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
83	p-Chlornitrobenzol 	39 49	1,5502 1,5400	81—82 101—103 20	Bittermandelgeruch. Ab 40° Körner, Spieße. Gleichgew.: Prismen, Stengel.
83	Trigenin (= Butyrylchlorhydrat + Pyramidon) $\text{CH}_3\text{—CH—CCl}_2\text{—CH(OH)—} + \text{C}_{12}\text{H}_{17}\text{N}_3\text{O}$ 	50 tr. 64 tr.	1,5101	107—109	Geruch. Ab 65° Nadeln. Gleichgew.: Nadeln. Aus Schmelze spitze Stengel, kleine Kristallisationsgeschwindigkeit; wenn zwei Minuten ohne Deckglas auf 100° erwärmt, Zersetzung. Restschmelze erstarrt in Sphärolithen von Pyramidon.
83	2,4,6-Tribrom-5-methylphenol 	47 —70—	1,6353	82	Ab 60° Nadeln. Gleichgew.: Nadeln, Stengel. Erstarrt zu dichtverfilzten Nadeln.
84	Acetessigsäureanilid $\text{CH}_3\text{—CO—CH}_2\text{—CO—HN—}$ 	63 66	1,5403 1,5290	85—89 108—110 22	Gleichgew. träg: Prismen. Erstarrt zu Sphärolithenmosaik. Polymorph II 61°, III unbeständig (32).
84	p-Nitrosodiäthylanilin $(\text{C}_2\text{H}_5)_2\text{N—}$ 	50 57	>1,6877		Grün. Absaugen meist notwendig oder umkristallisieren aus Alkohol mit Kohle. Ab 70° Körner. Gleichgew.: Sechsecke, Rhomboide. Erstarrt sphärolithisch. Polymorph. (30) Bei 80° sublimieren langsam bunte Rechtecke III 76°, graugrüne Rechtecke und Körner II 81°, grüne schiefwinkelige Kristalle und lange Stengel I 84°.
85	p-Nitrosodimethylanilin $(\text{CH}_3)_2\text{N—}$ 	49 55	>1,6877		Grün. Eventuell umkristallisieren aus Alkohol mit Kohle. Ab 75° Tröpfchen. Gleichgew.: Stengel mit sehr spitzen Enden. Bei 80° sublimieren langsam viele Tröpfchen, dazwischen isolierte graugrüne Körner und Rechtecke III 76°. Aus Tröpfchen Aggregate II 80° und schiefwinkelige Kristalle I 85°.
85	7-Methylindol 	45 45	1,5397 1,5793	76—77 100—101 24	Geruch. Ab 45° gelappte Blättchen, Spieße. Gleichgew.: Platten.
85	Diphenylenoxyd 	43 55	1,6126 1,6011	79 105 26	Geruch. Ab 45° Körner, Stäbchen. Gleichgew.: gelappte Blättchen. Erstarrt palmwedelartig.
85	γ-Phenylbuttersäureamid $\text{CH}_2\text{=CH—CH}_2\text{—CH}_2\text{—CO—NH—}$ 	63 70	1,5204 1,5101	87—88 110—111 23	Gleichgew.: Viereckige Platten. Erstarrt sphärolithisch.

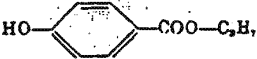
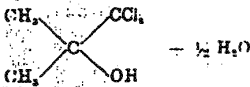
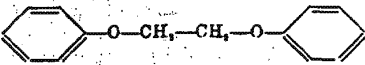
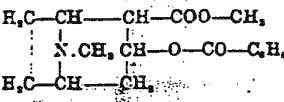
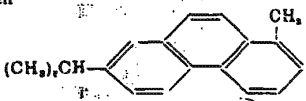
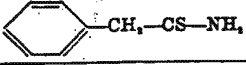
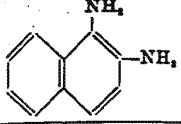
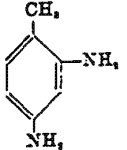
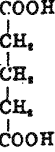
P. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
55,5	2,4-Dinitrophenetol 	44 55	1,5502 1,5403	90—91 114—115 24	Gelb. Ab 70° feine Nadeln. Gleichgew.: schöne Stengel mit sehr spitzen Enden.
85	p-Methyldibenzoylmethan 	49 60	1,6229 1,6128	112—114 129—130 17	Ab 70° Körner. Gleichgew.: Rechtecke, Prismen. Erstarrt zu Sphärolithen- mosaik. Polymorph II 64°.
85,5	Salicylalkohol (= Saligenin) 	62 67	1,5403 1,5299	95—97 120 24	Ab 65° dünne Rauten, vier- und sechs- eckige Blättchen. Gleichgew.: Rauten, Vierecke, Sechsecke. Erstarrt zu fein- strahligen Sphärolithen.
86	β-Naphthyleyanid 	55 63	1,5897 1,5795	76—78 100—101 24	Geruch. Ab 75° Tröpfchen.
87	m-Dimethylaminophenol 	56 61	1,5611 1,5502	85—86 108—110 23	Oberfläche nach längerem Liegen dun- kelviolett. Umsublimieren bei 80°. Ab 60° Körner, Blättchen. Gleichgew.: Prismen, Balken; oft Streifung. Er- starrt sphärolithisch.
87	2-Nitro-4-bromanisol 	40 58	1,5700 1,5611	79—81 98—100 19	Ab 70° wenig Nadeln. Gleichgew.: Stengel, Prismen. Erstarrt sphäro- lithisch.
87	p-Aminobenzonitril 	58 60	1,5897 1,5795	100—101 125 24	Ab 70° Körner, Blättchen. Gleichgew.: Prismen, Körner.
87,5	p-Dibrombenzol 	44 61	1,5700 1,5611	94—97 114 18	Ab 40° Körner, Blättchen, Nadeln, Stengel, Prismen. Gleichgew.: Platten und Prismen.
88	2,4-Dinitroanisol 	47 54	1,5700 1,5611	85—88 113—114 27	Gelb. Gleichgew.: schiefwinkelige, lange prismatische Kristalle.

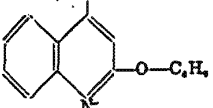
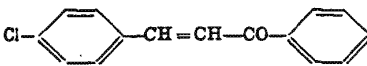
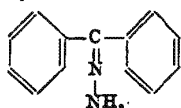
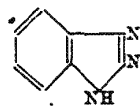
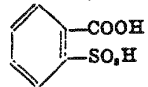
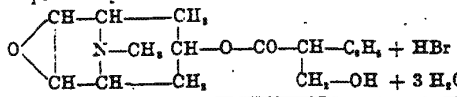
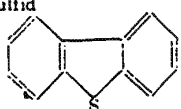
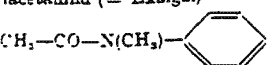
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
85—88	<i>o</i> -Phenylhydrozimtsäure 	51 tr.	61	1,5403 1,5299	108—109 125—127 Gleichgew. träg: Platten. Dimorph. 17
88	<i>p</i> -Chlorphenolbenzoat 	48	63	1,5502 1,5403	102—103 125—127 Ab 75° Körner. Nadeln. Gleichgew.: Platten. 23
88	Guajacolcarbonat 	53	64	1,5204 1,5101	109—110 130—133 Gleichgew.: Körner. 22
89	<i>p</i> -Bromidiphenyl 	58	60	1,6126 1,6011	89—91 112—115 Geruch. Ab 50° Blättchen. Gleichgew.: Platten. 24
89	Tetronal 	51	61	1,4683 1,4594	112 140—142 Ab 70° Blättchen. Stengel. Gleichgew.: Prismen, Stengel. Erstarrt zu Sphärolithen.
90	<i>o</i> -Acetoxynacetophenon 	58	68	1,4936 1,4742	86—87 109—110 Ab 50° Körner. Gleichgew.: Körner und Platten. Erstarrt zu breitstrahligen Sphärolithen.
90	Bromacetamid $\text{CH}_2\text{Br}-\text{CO}-\text{NH}_2$	65	76	1,5101	98—100 Ab 50° Nadeln. Balken. Gleichgew.: Balken. Erstarrt zu Sphärolithen.
90	2,3'-Dimethoxydiphenylsulfon 	54	65	1,5700 1,5611	84 109—111 Gleichgew.: Prismen, Stengel. Erstarrt zuerst glasig, später sphärolithisch. 26
90,5	Anästhesin (= <i>p</i> -Aminobenzoesäure-äthyl-ester) 	56	63	1,5302 1,5403	99 119 Ab 75° kleine, rechteckige Blättchen. Gleichgew.: Prismen. 20
91	<i>m</i> -Dinitrobenzol 	44	53	1,5502 1,5403	94—95 115—116 Ab 60° Nadeln. Gleichgew.: Balken, Prismen. Trimorph (14). 21

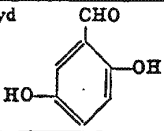
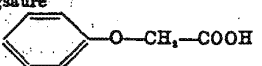
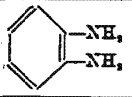
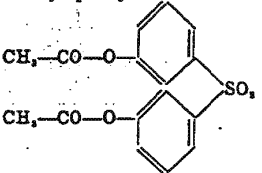
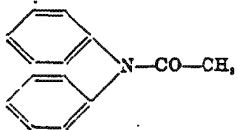
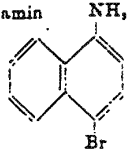
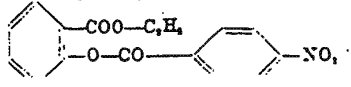
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
p 91	p-Bromjodbenzol 	46 53	1,6231 1,6126	90 111 21	Ab 35° Blättchen, Stäbchen, Körner. Gleichgew.: Körner, Prismen. Dimorph (41).
7 91	Butyrylchloralhydrat $\text{CH}_3\text{---CHCl---CCl}_2\text{---CH(OH)}_2$	61 —70—	<1,4339		Geruch. Verflüchtigt ohne Sublimation. Bei rascher Abkühlung flüchtige Kristallisation mit F. 70—75°. In Paraffin homogenes Schmelzen 72—75°.
o 91	p-Nitrophenyl-acetdimethylamid 	57 63	1,5403 1,5299	92 115—116 24	Ab 70° Körnchen. Gleichgew.: Blättchen, Prismen.
7/ 91	α-Phenyl-acetessigsäurenitril 	60 68	1,5101 1,5000	92—94 109—110 17	Ab 70° Körner und Blättchen. Gleichgew. träg: Platten. Schmelze erstarrt zu kleinen Sphärolithen.
2 92	Citraconsäure 	67 83	<1,4339		Beißender, unangenehmer Geruch. Ab 80° Tröpfchen. Zersetzung.
7 92	Euchinin $\text{C}_{20}\text{H}_{21}\text{ON}_2 \cdot (\text{---O---})_n \cdot \text{C}_2\text{H}_5$	47 63	1,5403	105—106	Ab 65° Nadeln. Gleichgew.: Nadeln, Stengel. Erstarrt glasig.
7 92 ^a	Salipyrin (= Antipyrinum salicylicum) $\text{C}_{11}\text{H}_{11}\text{ON}_2 + \text{C}_7\text{H}_5\text{O}_2$	52 67	1,5611 1,5502	99—100 125—126 12	Ab 75° Tröpfchen. Gleichgew.: Prismen.
5 92	β-Naphthochinolin (= 5,6-Benzochinolin) 	47 58	1,6715 1,6598	84—85 110—111	Aromatischer Geruch. Ab 60° gelappte Blättchen und kurze Spieße. Gleichgew.: unregelmäßige Blättchen.
92	Tetramethyl-4,4'-diamino-diphenylmethan 	47 60	1,5795 1,5700	92—93 110 17	Ab 80° Tröpfchen. Gleichgew.: Körner. Erstarrt glasig oder sphärolithisch.
92	Traubensäuremonoäthylester $\text{C}_6\text{H}_5\text{---COO---CH(OH)---CH(OH)---COOH}$	67 81	1,4339	86—90	Ab 70° einzelne Körner. Gleichgew.: Platten. Erstarrt zu Sphärolithen. Dimorph.
f 92	Äthylharnstoff $\text{C}_2\text{H}_5\text{---NH---CO---NH}_2$	67 83 tr.	1,4584 1,4339	77—78 150—151	Ab 60° Nadeln, Körner. Gleichgew.: Stäbchen. Erstarrt sphärolithisch.
85—93	d-Sorbit (wasserhaltig) $\text{HOCH}_2\text{---[CH(OH)]}_4\text{---CH}_2\text{OH} + \frac{1}{2}\text{H}_2\text{O}$	67 92	1,5101	88—91	Süß. Schmilzt träg. Schmelze zäh, daher verbleiben darin Dampfblasen. In Paraffin F. 83—88°, wasserfrei F. 110° (Trocknen!).
93	Acenaphthylene 	35 47	1,0483 1,0353	81 104—105 24	Gelb. Schwacher Geruch. Ab 45° verschiedenartige Blättchen und Körner. Gleichgew.: Platten.

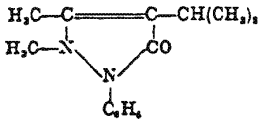
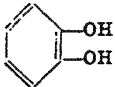
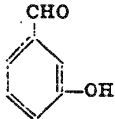
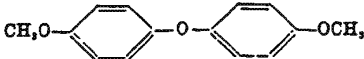
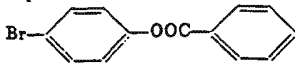
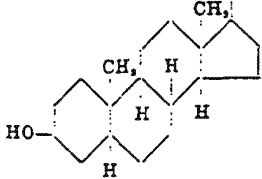
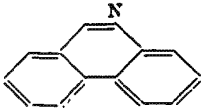
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
93	<i>o,o'</i> -Dibrom- <i>o</i> -xylol 	48 tr. 58	1,5795 1,5700	115—117 136—137 21	Geruch; tränenarregend. Ab 40° Tröpfchen. Gleichgew.: Körner. Erstarrt sphärolithisch.
93	Carbamidsäureisopropylester $H_2N-COO-CH(CH_3)_2$	61 73	<1,4339		Ab 35° Nadeln, Balken. Gleichgew.: Nadeln, Balken. Erstarrt sphärolithisch.
93	3,5-Dinitrobenzoesäure-äthylester 	44 58	1,5204	93—94	Ab 60° feine Nadeln, Körner. Gleichgew.: Stengel. Erstarrt sphärolithisch.
94	Triphenylmethan 	47 61	1,5897	84—85	Ab 80° Nadeln, Körner. Gleichgew.: Prismen. Erstarrt zu Sphärolithen. Dimorph (42, 16, 12).
94	Betol (= Salicylsäure- β -naphthylester) 	49 62	1,6126 1,6011	97—98 124—126 27	Gleichgew. trüg: Prismen. Erstarrt zu Sphärolithen. Polymorph (43, 12).
94	Benzalazin 	48 63	1,6353 1,6231	98—99 116—118 18	Gelb. Ab 45° Körner. Gleichgew.: Körner, Prismen. Dimorph II (30).
94	3,3-Diäthyl-2,4-dioxytetrahydropyridin 	62 71	1,5101	93—95	Ab 70° Körner, Stengel. Gleichgew.: Prismen und Stengel.
94	Aconaphthen 	47 63	1,6011 1,5997	101—102 121—122 20	Ab 50° Nadeln, Spieße und Prismen. Gleichgew.: Prismen. Erstarrt zu strahligen Aggregaten.
94	<i>p</i> -tert.-Amyl-phenol $C_6H_5-C(CH_3)_2-CH_2-OH$ 	47 58	1,4936 1,4842	93—95 113—114	Geruch. Ab 45° Nadeln. Gleichgew.: Spieße. Erstarrt zu feinstahligen Sphärolithen.
94	<i>p</i> -Methylphenylacessigsäure $CH_3-C_6H_4-CH_2-COOH$ 	57 69	1,4936 1,4842	103—104 126—127	Ab 70° Nadeln, kurze Spieße. Gleichgew.: Prismen. Bei unreinen Präparaten kann F. höher sein.
94	2,4,6-Tribromphenol 	48 56	1,6231 1,6126	111—113 128—131 15	Geruch. Ab 50° Nadeln. Gleichgew.: Stengel, Spieße. Enantiotrop dimorph II 92°, Up. 84° (32).

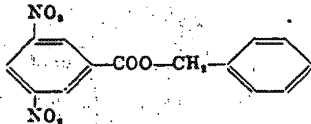
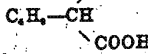
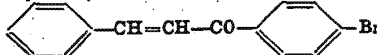
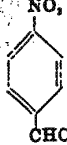
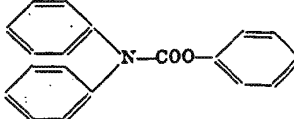
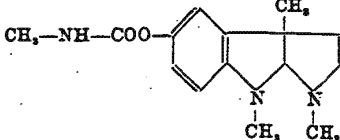
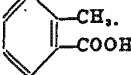
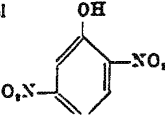
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
95	4-Nitro-2-methylphenol 	35 58	1,5897 1,5795	94—95 115—116 21	Gleichgew.: Körner, Balken. Erstarrt zu Sphärolithen von II 86° mit Gleichgew.: Nadeln.
95	Benzil 	50 95	1,5700 1,5611	96—98 117—118 21	Hellgelb. Ab 85° Nadeln, Stengel. Gleichgew.: Stengel mit spitzen Enden. Polymorph II 41,5°, III etwa 27° (36).
95	Tribenzylamin 	44 64	1,5502 1,5403	112 134—135 23	Ab 75° winzige Körner. Gleichgew.: Platten, Prismen. Erstarrt zu feinstrahligen Sphärolithen. Bei Erwärmen Umlagerung.
95	Tetrabromkohlenstoff CBr ₄	54 40	1,5898	111—113	Ab 30° starke Sublimation von gezackten Blättchen und Rosetten, ab 46° isotrop wabenartig (12).
95	3-Methylindol (= Skatol) 	43 44	1,5700 1,5611	100 121 21	Geruch. Ab 60° gelappte Blättchen. Gleichgew.: dünne, gelappte Platten. Erstarrt flächenhaft.
95	4,6-Dichlorresorcin 	50 —46—	1,5700	95—97	Oft braun verfärbt. Umsublimieren bei 90°. Ab 55° Nadeln. Schmilzt trög. Aus Schmelze lange Nadeln.
96	p-Nitrobenzoesäure-methylester 	51 63	1,5101 1,5000	111—112 130 8	Hellgelb. Ab 70° einzelne Rauten. Gleichgew.: Rauten.
96	Methylisobutylketon-2,4-dinitrophenyl- hydrazon 	42 60	1,6126	111	Orange. Ab 40° Körner, Blättchen. Gleichgew.: Körner. Erstarrt sphärolithisch.
96	α-Naphthol 	50 47	1,6231 1,6126	86—87 107—109 21	Ab 80° langgestreckte, rechteckige Kristalle. Gleichgew.: Rechtecke.
96	Tetramethyl-4,4'-diaminobenzhydrol (= Michlers Hydrol) [(CH ₃) ₂ N-C ₆ H ₄] ₂ CH-OH	71 80	1,5397 1,5795	110—112 147—149 3	Gleichgew.: Platten. Erstarrt sphärolithisch. Dimorph. Bei Mischen mit Benzil vorübergehend blaugrün.
96,5	m-Nitrophenol 	57 59	1,5502 1,5403	124 145—147 22	Gelb. Ab 65° schiefwinkelige Stäbchen und Prismen, vieleckige Körner. Gleichgew.: Körner.
97	2,7-Dimethylnaphthalin 	51 65	1,5611 1,5502	106 126 20	Ab 45° unregelmäßige Blättchen und Stäbchen. Gleichgew.: unregelmäßige Blättchen. Bei unreinen Präparaten kann F. höher sein.

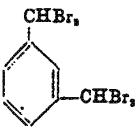
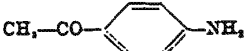
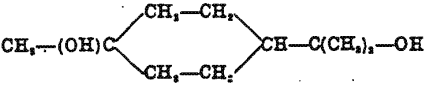
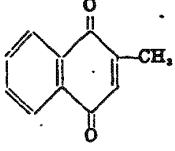
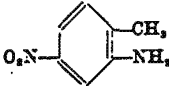
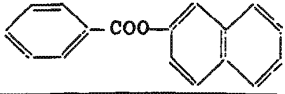
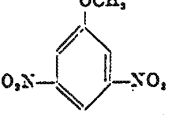
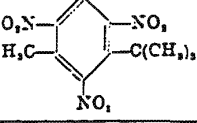
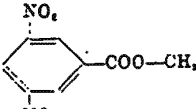
F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
4 97	Nipasol (= p-Oxy-benzoesäure-propyl- ester) 	59	72	1,5101 1,5000	98—100 122—123 Ab etwa 90° Tröpfchen. Gleichgew.: Prismen.
5 97	β,β,β-Trichlor-tert.-butylalkohol (= Acetonchloroform = Chlorbutol = = Chloreton)  + ½ H₂O	33	35	1,4339	135—140 Campherartiger Geruch. Ab 25° Stäb- chen, Blättchen, Straßenpflaster. Star- ke Sublimation. Gleichgew.: Platten. Das Halhydrat schmilzt unter Wasser bei 78° (44).
6 98	Glykoldiphenyläther 	55	60	1,5209 1,5211	107—109 130—131 Ab 60° Blättchen. Gleichgew. Blätt- chen.
7 98	Cocain 	47	62	1,5000 1,4936	103—105 121—123 Ab 70° Körner, Nadeln. Gleichgew. trög: Körner. Erstarrt glasig, bei Erwärmen sphärolithisch.
8 98	Roten 	41	59	1,0231	112—114 Ab 60° Nadeln. Gleichgew.: breite Linsen, Blättchen.
9 98	Phenylthioacetamid 	62	72	1,6131 1,6011	105—108 129—131 Ab 65° Nadeln. Gleichgew.: Balken.
10 98	1,2-Naphthylendiamin 	61	58	Zersetzung	Meist nahezu schwarz verfärbt (licht- empfindlich!). Umkristallisieren in ver- dünntem Alkohol mit Kohle; daraus dünne, seidige Blättchen, die sich bald verändern. Schmilzt unter Zersetzung und Braunfärbung. In Indexflüssig- keiten löslich.
11 99	Phenanthren 	46	60	1,0598 1,0483	98 126 Ab 60° dünne Blättchen. Gleichgew.: gelappte Scheiben.
12 99	m-Toluyldiamin (= 2,4-Diaminotoluol) 	62	66	1,5897 1,5795	104—105 126—127 Ab 60° Körner, Blättchen, Nadeln. Gleichgew.: Körner und Prismen. Er- starrt sphärolithisch.
13 99	Glutarsäure 	66	84	1,4339	98—100 Geruch. Ab 80° Umwandlung wie ein Schleier. Ab 85° gebogene Stäbchen. Gleichgew.: Körner, Prismen. Enantio- trop dimorph. Up. 80° (45).

F. °C	Substanz	Eutekt. Temp. mit Azobenzol Benzil	Glas- pulver	Temp. °C	Besondere Kennzeichen
4 99	2-Butyloxychinolin-4-carbonsäure-[β-diäthyl- amino-äthylamid]-hydrochlorid (= Percain = Cineainhydrochlorid) $\text{CO}-\text{NH}-\text{CH}_2-\text{CH}_2-\text{N}(\text{C}_2\text{H}_5)_2$  $+ \text{HCl}$	63 74	1,5502 1,5403	101—102 131—132 30	Gleichgew. trüg: Nadeln, Platten.
5 99	p-Chlorchalkon (= p-Chlorbenzalaceto- phenon) 	54 69	1,6231 1,6126	106—108 127—128 2-1	Ab 90° spärliche Blättchen. Gleichgew.: gestreckte Sechsecke und Stengel.
6 99	Benzophenonhydrazon 	54 67	1,6011 1,5897	104—106 127—129 23	Ab 80° Körner, Prismen. Gleichgew.: Körner, Platten. Erstarrt zu feinstrah- ligen Sphärolithen.
7 99	Benzotriazol (= Aziminobenzol) 	67 74	1,5795 1,5700	114—115 139—140 25	Ab 70° Nadeln. Gleichgew.: lange Nadeln, Stengel. Erstarrt sphäroli- thisch. Polymorph II 97°, III 85°, IV 83°, V 68° (32).
90—100	o-Sulfobenzoesäure, teilw. H ₂ O 	Vgl. bei 135°			Schmilzt ab 60° und kristallisiert wieder. In einzelnen Tropfen Auflösen der neuen Kristalle bei etwa 100°; größere Aggregate F. 135°. Schmilzt in Paraffin bei 65—67° homogen.
90—100	Scopolaminhydrobromid + 3 H ₂ O  $\text{C}_{17}\text{H}_{21}\text{O}_4\text{N} + \text{HCl} + \text{H}_2\text{O}$	Vgl. Scopolaminhydrobromid bei 194—197°			Schmilzt unter Abscheiden wasserfreier Kristalle. In Paraffin homogenes Schmelzen bei 90° Nachher manchmal wasserfreie Körner.
90—100	Scopolaminhydrochlorid + H ₂ O $\text{C}_{17}\text{H}_{21}\text{O}_4\text{N} + \text{HCl} + \text{H}_2\text{O}$	Vgl. Scopolaminhydrochlorid bei 190—205°			Schmilzt unter Abscheiden wasserfreier Kristalle. In Paraffin inhomogenes Schmelzen bei 70° unter Abscheiden von wasserfreien Prismen, die sich bei etwa 100° lösen.
1 100	Diphenylsulfid 	Benzil Acetanilid 63 88	1,6598 1,6482	99 125 26	Ab 55° Nadeln, kleine Balken. Gleich- gew.: Nadeln, Prismen. Dimorph II 96° (14).
2 100	Homatropin (= Mandelsäure-tropinester) $\text{CH}_2-\text{CH}-\text{CH}_2$ $\text{N}-\text{CH}_2-\text{CH}-\text{O}-\text{CO}-\text{CH}(\text{OH})-\text{C}_6\text{H}_5$ $\text{CH}_2-\text{CH}-\text{CH}_2$	73 79	1,5204 1,5101	92—93 116—117 27	Gleichgew. trüg: Körner, Prismen. Erstarrt glasig.
3 100	N-Methylacetanilid (= Exalgin) $\text{CH}_3-\text{CO}-\text{N}(\text{CH}_3)-\text{C}_6\text{H}_5$ 	63 58	1,4937 1,4840	113 130—131	Ab 60° Körner, Prismen und quadra- tische Blättchen. Gleichgew.: Prismen, Körner. Erstarrt sphärolithisch.

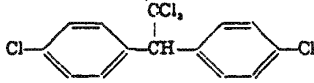
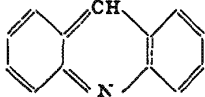
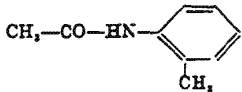
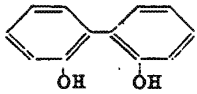
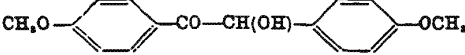
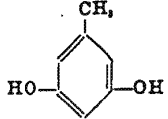
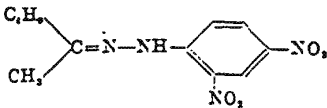
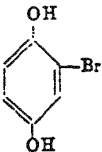
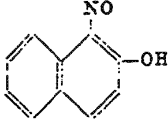
F.°C	Substanz	Eutekt. Temp. mit Benzil	Temp. mit Acetanilid	Glaspulver	Temp. °C	Besondere Kennzeichen
4 100	2,5-Dioxybenzaldehyd 	69	58	1,5897	100—105	Gelb. Ältere Präparate schmutziggelb, Umsublimieren bei 90°. Ab 80° Blättchen, Nadeln. Gleichgew.: Stengel, Tafeln. Brechungsindex wegen Verfärbung erschwert. Erstarrt zu stabilen und instabilen Sphärolithen.
100,5	α -Chlorcrotonsäure $\text{CH}_3\text{—CH=CCl—COOH}$	68	51	1,4339 1,4534		Eigenartiger Geruch. Ab 45° Stäbchen, Blättchen. Gleichgew.: breite Prismen, Balken. Verflüchtigt. Erstarrt zu grobstrahligen Sphärolithen.
101	Phenoxycessigsäure 	72	55	1,5101 1,5000	96 114—115	Geruch. Ab 60° Stäbchen. Gleichgew.: Stengel, Prismen. Erstarrt zu Sphärolithen.
7 102	o-Phenylendiamin 	57	79	1,5897	116—118	Ab 80° kurze Prismen, rechteckige Blättchen. Schmelze wird rasch gelb. Brechungsindex wegen Verfärbung schwierig.
8 102	Adonit $\text{HO—CH}_2\text{—[CH(OH)]}_n\text{—CH}_2\text{—OH}$	94	100	1,4937 1,4840	111—112 145—146	Gleichgew. träg: Prismen. Erstarrt glasig.
9 102	3,3'-Diacetoxydiphenylsulfon 	72	82	1,5403 1,5301	93—94 118—119 25	Gleichgew.: Prismen, Stengel. Erstarrt glasig.
102	Maleinsäureanhydrid-Addukt der 8,10-Heptadecadien-1-carbonsäure HC—CO $\parallel$ $> \text{O} + \text{CH}_2\text{—[CH}_2\text{]}_n\text{—CH=CH—CH=CH—[CH}_2\text{]}_m\text{—COOH}$ HC—CO	74	75	1,4683 1,4594	94—95 120—122	Ab 55° Nadeln, Körner. Gleichgew.: Nadeln, Stengel. Schmelze erstarrt milchig trüb. Dimorph II 67°.
103	Novocainnitrat $\text{H}_2\text{N—C}_6\text{H}_4\text{—COO—CH}_2\text{—CH}_2\text{—N(C}_2\text{H}_5)_3 + \text{HNO}_3$	93	73	1,5502	113—115	Gleichgew.: Körner. Erstarrt glasig.
2 103	Acetyldiphenylamin 	67	68	1,5011 1,5502	100 127—128 25	Ab 75° Körner, sechseckige Blättchen. Gleichgew.: Körner. Erstarrt zu feinstrahligen Sphärolithen. Polymorph II 87°, III 86° (32).
3 103	Methylharnstoff $\text{CH}_3\text{—NH—CO—NH}_2$	82	72	1,4683 1,4594	75—76 106—109	Ab 80° Stäbchen. Gleichgew.: Körner, Prismen. Erstarrt sphärolithisch. Dimorph II 101° (32).
4 103	4-Brom-1-naphthylamin 	63	79	1,6715	110	Ab 80° Nadeln. Gleichgew.: Balken, Nadeln. Sinkt ab. Erstarrt zu Sphärolithen.
5 103	p-Nitrobenzoyl-salicylsäureäthylester 	72	86	1,5403 1,5299	109—110 134—136 25	Ab 80° kleine Körner. Gleichgew.: Körner, Prismen.

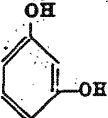
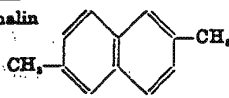
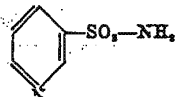
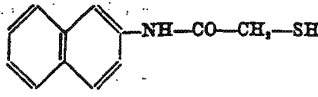
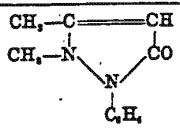
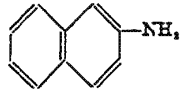
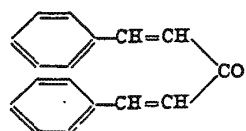
F. °C	Substanz	Eutekt. Temp. mit Benzil	Temp. mit Acetanilid	Glaspulver	Temp. °C	Besondere Kennzeichen
103,5	4-Isopropyl-antipyrin 	67	55	1,5403 1,5299	94—95 116—117 27	Ab 80° Nadeln. Gleichgew. Stengel und Nadeln. Erstarrt zu Sphärolithen verschiedener Modifikationen. Bei Erwärmen Umwandlung.
100—104	Lävulose (= d-Fructose) HOCH ₂ —CO—[CH(OH)] ₃ —CH ₂ OH	94	97	1,5101 1,5000	108—110 135—137 27	Geringe Doppelbrechung. Erweicht langsam. Erstarrt glasig.
104	Pimelinsäure HOOC—[CH ₂] ₆ —COOH	82	69	1,4339	106—107	Geruch. Gleichgew.: Körner, Prismen, Blättchen. Enantiotrop dimorph Up. 77° (46).
104	Brenzcatechin 	61	37	1,5403 1,5290	112—114 128—129 16	Ab 75° Prismen, vierseitige Blättchen. Gleichgew.: vierseitige Blättchen. Polymorph (17).
104	m-Oxybenzaldehyd 	74	61	1,5611 1,5502	100—101 124—125 24	Geruch. Ab 80° Blättchen, Nadeln, Stengel. Gleichgew.: Prismen, Körner. Erstarrt sphärolithisch.
104	Dianisylloxid 	72	39	1,5403 1,5299	105—107 126—127 21	Ab 80° Blättchen. Gleichgew.: Platten, Stengel. Erstarrt sphärolithisch.
104	p-Bromphenolbenzoat 	70	38	1,5611 1,5502	119—121 140—142 21	Ab 83° Körner, Nadeln. Gleichgew. trüg. Platten.
103—105	Sparteinsulfat C ₁₅ H ₂₁ N ₃ + H ₂ SO ₄ + 5 H ₂ O	80	47	1,5299 1,5204	105 123—125 19	Zwischen 60° und 80° entweicht Wasser unt. Trübung. In Paraffin teilweise inhomogenes Schmelzen bei 74°, teils homogenes Schmelzen bei 81°. Vollständig wasserfrei F. 140°.
105	Koprosterin (= cis,cis,trans,trans Dihydrocholesterin) 	80	38	1,4936 1,4842	93—94 123—124	Gleichgew.: Balken, Stengel. Erstarrt glasig. Bei Erwärmen Sphärolithe.
105	Phenanthridin 	69	50	1,6398 1,6483	105—106 128—129 23	Ab 75° Nadeln, Stäbchen. Gleichgew.: Platten, Prismen. Erstarrt sphärolithisch. Wenn unrein Faserdrilling und F. höher.

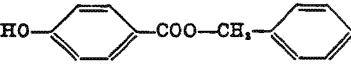
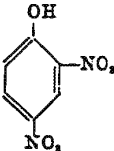
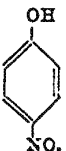
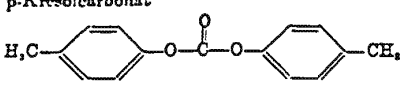
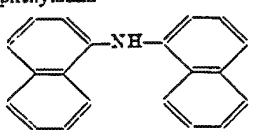
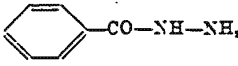
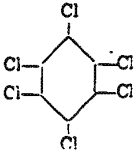
F. °C	Substanz	Eutekt. Temp. mit Benzil	Temp. mit Acetanilid	Glaspulver	Temp. °C	Besondere Kennzeichen
105	2,3-Dimethylnaphthalin (= Guajen) 	65	90	1,5700 1,5611	107 123 11p	Ab 65° gelappte Blättchen, Spieße. Gleichgew.: Platten.
105	3,5-Dinitrobenzoesäure-benzylester 	70	85	1,5611 1,5602	107—108 123 15	Gleichgew.: Stengel, Spieße. Erstarrt sphärolithisch.
105	Butylmalonsäure (= Pentan-2,3-dicarbon-säure) 	77	46	<1,4339		Geruch. Gleichgew.: Prismen. Erstarrt zu Sphärolithen.
105	p'-Bromchalkon (= p'-Brombenzalaceto-phenon) 	72	86	1,6353 1,6231	119—121 139—141 20	Ab 90° Nadeln. Gleichgew.: Balken.
105,5	p-Nitrobenzaldehyd 	68	82	1,5403 1,5299	122 138—139 17	Bittermandelgeruch. A 75° feinkörnige und kurzprismatische Kristalle. Gleichgew.: Stengel, Balken. Dimorph II 104,5° (10).
106	Diphenylcarbamidsäure-phenylester 	67	80	1,5700 1,5611	110—111 133 22	Ab 90° feinkörnige Sublimate. Gleichgew.: Körner, Prismen. Erstarrt glasig oder nach Berührung sphärolithisch.
106	Physostigmin (= Eserin) 	72	71	1,5403 1,5299	83 111—112 29	Gleichgew.: Körner, Prismen. Erstarrt glasig.
106	Chloralurethan (= Uralin) $\text{CCl}_3\text{—CH(OH)—NH—COO—C}_6\text{H}_5$	75 tr.	65 tr.	1,4584 1,4339	108—110 152—153	Geruch. Ab 70° Blättchen, Körner. Gleichgew.: Balken, Prismen. Erstarrt zu Sphärolithen.
106	o-Toluylsäure 	76	76	1,5101 1,5000	117—118 136—137	Ab 60° Nadeln, Spieße. Gleichgew.: Stengel, Balken. Erstarrt sphärolithisch.
106	2,5-Dinitrophenol 	67	75	1,5700	119—120	Gelb. Gleichgew. trägt: Körner und Blättchen.

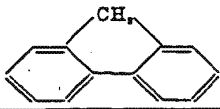
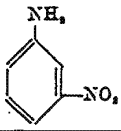
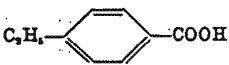
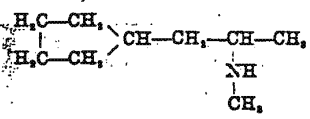
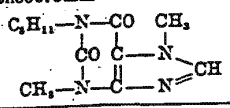
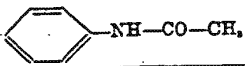
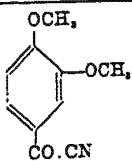
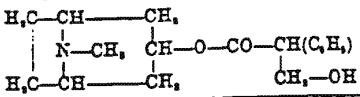
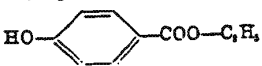
F. °C	Substanz	Eutekt. Temp. mit Benzil	Temp. mit Acetanilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
106	$\omega, \omega, \omega', \omega'$ -Tetrabrom-m-xylol 	68	87	1,6353 1,6231	97 118 9-1	Ab 70° Nadeln. Körner. Gleichgew.: Prismen, Körner. Erstarrt sphärolithisch.
106	p-Aminoacetophenon 	74	76	1,6011 1,5897	117—118 136—136 18	Gelb. Ab 80° Körner, Blättchen. Gleichgew.: Blättchen, Prismen, Körner. Erstarrt sphärolithisch.
108	cis-Terpin (= 1,8-Terpin) 	82	78	1,4584 1,4339	88 144—145	Ab 80° Blättchen, Prismen. Ab 70° lagern sich die Kristalle zu weniger doppelbrechenden um mit F. 106°, da meist Terpinhydrat vorliegt. Gleichgew.: Körner, Rechtecke. In Paraffin schmelzen ursprüngliche Kristalle bei 116—118°, Dampfblasen.
106	2-Methyl-1,4-naphthochinon 	71	83	1,5700 1,5611	109—110 130 20	Gelb. Ab 65° Nadeln. Wenn F. unscharf, umsublimieren bei 100°. Kein Gleichgew. Aus Rest kristallisieren erst bei 103° zwilliggestreifte Balken.
106,5	Azelainsäure $\text{HOOC}-(\text{CH}_2)_4-\text{COOH}$	90	77	etwa 1,4339		Gleichgew.: Körner, Prismen. Dimorph (47).
107	4-Nitro-2-aminotoluol 	67	79	1,5795 1,5700	113 134 21	Gelb. Ab 100° Körner, Blättchen. Gleichgew.: Prismen, Balken. Erstarrt teils grobstrahlig sphärolithisch, teils regellos.
107	Benzonaphthol (= Benzoesäure- β -naphthylester) 	72	91	1,6011 1,5897	102—106 130—131 26	Gleichgew.: Spieße und schiefwinkelige Prismen. Erstarrt sphärolithisch. Polymorph (48).
107	3,5-Dinitroanisol 	68	85	1,5403 1,5299	113—115 132—133 19	Ab 90° Körner, Prismen, Blättchen. Gleichgew.: Nadeln, Spieße. Erstarrt in Nadeln.
107 (114)	2,4,6-Trinitro-5-tert.-butyl-m-xylol (= Xylolmoschus) 	72	93	1,5000 1,4936	121—123 137—139	Parfumergeruch. Ab 80° Körner. Gleichgew.: Prismen, Körner. Erstarrt sphärolithisch. Trimorph I 114° und II 107° enantiotrop; III unbeständig (14).
107	3,5-Dinitrobenzoesäure-methylester 	66	81	1,5204	127—128	Ab 75° Nadeln, Spieße. Gleichgew.: Stengel, Prismen.

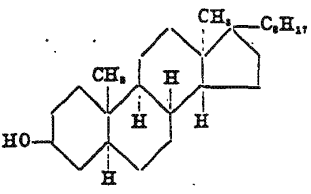
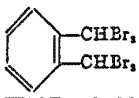
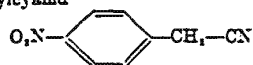
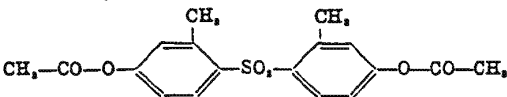
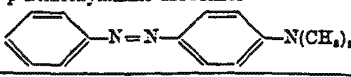
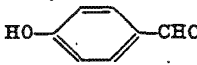
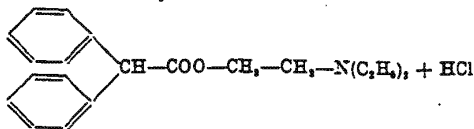
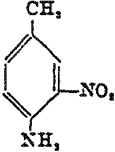
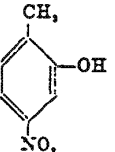
F. °C	Substanz	Eutekt. Temp. mit Benzil	Temp. mit Acetanilid	Glaspulver	Temp. °C	Besondere Kennzeichen
107	Hyoscyamin (= 1-Tropasäuretropinester) <chem>CC1(CCN(C1)COC(=O)C2=CC=CC=C2)O</chem>	77	84	1,5101	114—118	Ab 100° winzige Nadeln. Erstarrt glasig, bei Erwärmen feinnadelige Kristallmasse.
108	N-Methylolbenzamid <chem>CC1=CC=CC=C1C(=O)NCO</chem>	88	94	1,5504 1,5403	95—96 122—123	Ab 90° Tröpfchen. Gleichgew. trägt Prismen, Körner. 27
108	Diäthylstilböstrol-dipropionat <chem>CCOC(=O)C1=CC=CC=C1C(C=CC1=CC=CC=C1)C(=O)OCC</chem>	71	86	1,5000 1,4937	114—116 129—131	Gleichgew.: Platten. Erstarrt sphärolithisch.
108	Pyramidon <chem>CC1(C)N(C)C(=O)C2=CC=CC=C12</chem>	69	60	1,5403 1,5299	117—120 138—140	Ab 80° Nadeln, Blättchen, Balken. Gleichgew.: Balken. Erstarrt zu Sphärolithen von Modifikation II, bei Erwärmen Umwandlung von vielen Punkten aus. 20
108	Benzoylperoxyd <chem>O=C(Oc1ccccc1)Oc2ccccc2</chem>	75	90	1,5700	81—84	Ab 80° Tröpfchen, kleine Körner und wenig Nadeln. Gleichgew.: Körner (rhomboidische Doppelpyramiden); nach Schmelzen sofort Gasblasen.
108	N,N'-Dimethylharnstoff <chem>CN(C)C(=O)NC(C)=O</chem>	79	60	1,4339	118—119	Ab 60° Nadeln, Stengel. Gleichgew.: Stengel, Prismen. Erstarrt zu Sphärolithen.
2 108	2-Oxypyridin <chem>O=C1C=CC=CC=N1</chem>	79	69	1,5611 1,5502	114—115 137—138	Ab 100° briefkuvertartige Sublimat, Körner. Gleichgew.: Körner. Erstarrt oft zu II F. 95°. 22
3 109	Phenyl-β-naphthylamin <chem>Nc1ccc2ccccc2c1</chem>	65	85	1,6598 1,6483	123—124 146—147	Ab 95° Stäbchen. Gleichgew.: Körner, Prismen. Erstarrt zu Sphärolithen. 23
4 109	9,10-Dihydroanthracen <chem>C1=CC=C2C(=C1)C=C3C=CC=CC3C2</chem>	70	92	1,5700 1,5611	114—116 136—138	Ab 50° Körner, Nadeln. Gleichgew.: Rechtecke, Prismen. 22
5 109	p-Oxyacetophenon <chem>CC(=O)c1ccc(O)cc1</chem>	78	65	1,5611 1,5502	114—116 134—135	Ab 70° Nadeln, Blättchen. Gleichgew.: Platten, Balken. Erstarrt sphärolithisch. 20
6 109	Fluoranthren <chem>c1ccc2c(c1)c3ccccc3c2</chem>	63	* 85	1,6715	149	Gelb. Ab 70° Nadeln. Gleichgew.: Prismen, Stengel, Körnchen. Erstarrt zu grobstrahligen Sphärolithen.

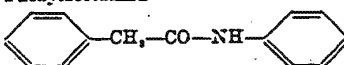
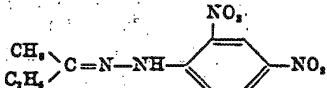
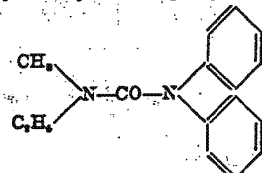
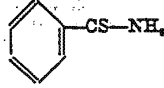
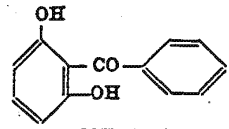
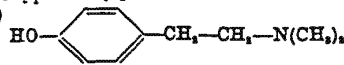
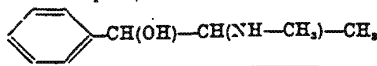
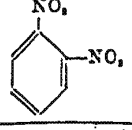
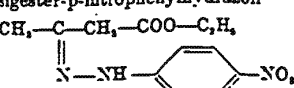
F.°C	Substanz	Eutekt. Temp. mit Benzil	Temp. mit Acetanilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
7 109	p,p'-Dichlor-diphenyl-trichloräthan (= DDT = Neocid) 	66	93	1,5795 1,5700	111—112 122—123 11	Ab 95° Tröpfchen. Gleichgew.: Nadeln, Prismen. Erstarrt schwer zu Sphärolithen. Dimorph II 95° (32).
8 110	Acridin 	67	83	1,6715 1,6598	135 155—158 20	Weiß bis gelb. Ab 70° kleine Stäbchen. Gleichgew.: Balken. Polymorph II 110°, III 109,5°, IV 199°, V 106° (25).
9 110	Acet-o-toluidid 	77	74	1,5101 1,5000	124—126 151—152 27	Ab 70° Nadeln, Blättchen. Gleichgew.: Stengel. Erstarrt zu Sphärolithen.
10 110	o,o-Diphenol (= 2,2'-Dioxydiphenyl) 	65	46	1,5897 1,5795	124—125 143—144 19	Ab 60° Körnchen, Rauten. Gleichgew.: Körner, Rauten, Prismen. Erstarrt glasig, bei Erwärmen langsam Platten. In Wasser wenig löslich, daraus schöne Sechsecke.
110	Anisoin (= 4,4'-Dimethoxy-benzoin) 	78	88	1,5700 1,5611	127 109 18	Gleichgew.: Stengel, Nadeln. Erstarrt glasig, bei Erwärmen sphärolithisch.
2 110	Orcin (= 3,5-Dioxytoluol = 5-Methyl-resorcin)  + H ₂ O	67	25	1,5403— 1,5299		Hydratkristalle trüben sich ab 45° oder F. 58°. Gleichgew.: Körner. In Paraffin inhomogenes Schmelzen bei 56° und Abscheidung wasserfreier Stengel und Körner.
3 110	Methylbutylketon-2,4-dinitrophenyl-hydraxon) 	73	87 tr.	1,6011 1,5897	127 155 22	Orange. Ab 90° spärliche Nadeln. Gleichgew.: Platten. Körner. Erstarrt sphärolithisch. Trimorph (40).
7 110	Bromhydrochinon 	64	44	1,5897	112	Ab 90° Tröpfchen. Schmilzt trög. Gleichgew.: Körner; erstarrt sphärolithisch.
106—110	α-Nitroso-β-naphthol 	73	85	Zersetzung		Orange, oft braun verfärbt. Umsublimieren bei 100°. Ab 80° Nadeln. Körner, vierseitige kurze Prismen. Gleichgew.: Körner, Prismen, Spieße. Aus Schmelze feinfaserige Sphärolithe II 89°. Umwandlung durch Impfen, bei Umsublimieren Nadeln von II.

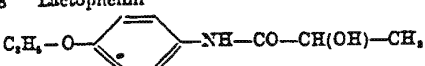
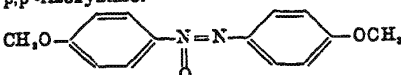
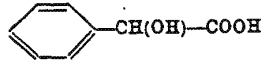
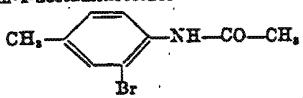
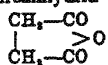
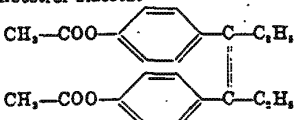
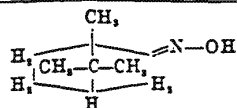
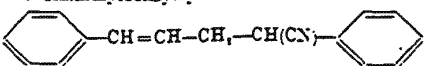
F. °C	Substanz	Eutekt. Temp. mit Benzil	Temp. mit Acetanilid	Glaspulver	Temp. °C	Besondere Kennzeichen
6 110	Nipagin M instabil	Vgl. bei 127°				Instabile Modifikation sublimiert in Stengeln, Rhomboiden, Sechsecken. Aus Schmelze instabile Sphärolithe, bei Erwärmen Umwandlung.
108—110 d-Sorbit (= Sionon) wasserfrei		Siehe 93° (Hydrat)				
108—110	Abasin (= N-Acetyl-N'-bromdiäthylacetyl-harnstoff) $\text{CH}_3-\text{CO}-\text{NH}-\text{C}(=\text{O})-\text{NH}-\text{CO}-\text{CBr}(\text{C}_2\text{H}_5)_2$	76	77	1,4842 1,4683	110—111 144—146	Aromatischer Geruch. Ab 85° Blättchen, Stäbchen. Gleichgew.: Balken. Erstarrt sphärolithisch.
2 110,5	Resorcin 	68	33	1,5502 1,5403	121—123 149—151 28	Ab 90° Körner. Gleichgew.: Stengel, Polymorph II 108°, III 107°, IV und V sind unbeständig (33). I und II enantiotrop.
1 111	2,6-Dimethylnaphthalin 	72	95	1,5611	106	Bei Erwärmen Geruch nach Anis. Ab 50° Blättchen, starke Sublimation. Gleichgew.: gelappte Platten. Pikrat (im Kontaktpreparat) F. 142°.
1 111	Pyridin-3-sulfamid 	90	81	1,5502 1,5403	93—95 124 30	Gleichgew.: Körner. Erstarrt sphärolithisch.
2 111	Thionalid (= Thioglykolsäure-β-naphthylamid) 	76	78	1,6483 1,6353	107—108 142—143 35	Gleichgew. sehr träg: Platten. Erstarrt grobstrahlig sphärolithisch.
3 111,5	Antipyrin 	72	51	1,5611 1,5502	120—122 144—147 25	Ab 85° Nadeln, Stengel, Blättchen. Gleichgew.: Körner. Erstarrt sehr langsam zu Sphärolithen. Trimorph (50).
4 112	Dehydracetsäure $\text{CH}_3-\text{CO}-\text{CH}=\text{CO}-\text{CH}_3$ $\text{CO}-\text{O}-\text{C}(=\text{O})-\text{CH}_3$	66	61	1,5101	123—125	Ab 60° Stäbchen, Körner. Gleichgew.: kurze Prismen. Dimorph II 111° (32). Erstarrt grobkörnig.
5 112	Äthylmalonsäure $\text{C}_2\text{H}_5-\text{CH}(\text{COOH})_2$	86	50	<1,4339		Ab 90° Nadeln, Blättchen, Stengel. Gleichgew.: Rauten, Balken, Prismen.
96 112	β-Naphthylamin 	66	82	1,6353 1,6231	125—126 147—148 27	Häufig rötlich verfärbt. Ab 60° übereinandergelagerte Blättchen. Gleichgew.: unregelmäßige Blättchen.
97 112	Dibenzylidenacetone 	65	75	1,6353 1,6231	155—126 147—150	Hellgelb. Gleichgew.: Körner, Platten. Erstarrt feinnadelig.

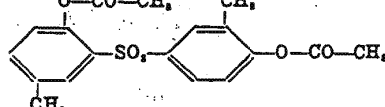
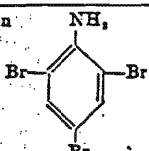
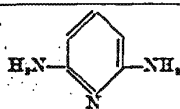
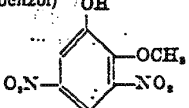
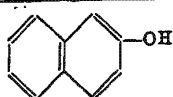
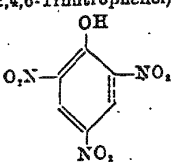
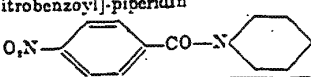
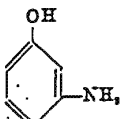
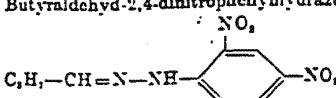
F. °C	Substanz	Eutekt. Temp. mit Benzil	Temp. mit Acetanilid	Glaspulver	Temp. °C	Besondere Kennzeichen
8 112	N,N'-Diäthylharnstoff $C_2H_5-NH-CO-NH-C_2H_5$	81	63	<1,4339		Ab 35° Nadeln, zum Teil gebogen. Reichlich Sublimat. Gleichgew.: Stengel, Prismen. Erstarrt sphärolithisch.
7 112	Nipabenzyl (= p-Oxybenzoesäurebenzylester) 	76	70	1,5611 1,5502	110—112 136—137 20	Ab 70° kleine Körner. Gleichgew.: Körner, Balken. Polymorph II 107°, III 104°, IV 102° (51). Erstarrt sehr langsam zu feinstrahligen Sphärolithen.
110—113	Mannit hexanitrat $O_6N-O-CH_2-[CH(O-NO_2)]_4-CH_2-O-NO_2$	78	53		Zersetzung	Ab 100° feine Nadeln, schmilzt unter Zersetzung, Gasblasen. In Paraffin inhomogenes Schmelzen bei 95—98° unt. Abscheidung von Nadeln, Gasblasen.
1 113	2,4-Dinitrophenol 	62	79	1,5897 1,5795	105—106 125—127 20	Gelb. Ab etwa 60° Körner, Blättchen. Gleichgew.: Stengel, Balken. Erstarrt fächerartig.
2 113	p-Nitrophenol 	64	30	1,5897 1,5795	117—119 137—138 20	Gelb. Ab 75° Prismen, Stengel. Gleichgew.: Körner. Polymorph (52, 53, 12).
3 113	p-Kresolcarbonat 	71	90	1,5000	125	Geruch. Ab 60° Nadeln, Spieße. Gleichgew.: Balken. Erstarrt zu Sphärolithen.
4 113	p-Chinon (= 1,4-Benzochinon) 	67	80	1,4936	121—123	Dunkelgelb, häufig verfärbt. Umsublimieren bei 90°. Ab 35° Blättchen, Stäbchen, Stengel. Gleichgew.: Stengel, Balken. Erstarrt zu großen Balken.
5 113	Di-σ-naphthylamin 	68	92	1,6715 1,6598	161—162 175—177 24	Gleichgew.: Prismen, Körner. Erstarrt in Körnern.
6 113	Benzhydrazid (= Benzoesäurehydrazid) 	81 tr.	82 tr.	1,5611 1,5502	122 147—148 26	Geruch. Ab 50° Körner, später Nadeln, Blättchen. Gleichgew.: Balken, Prismen.
7 113,5	γ-Hexachloreyclohexan (= Gammexan) 	64	88	1,5101	171	Ab 85° Prismen. Gleichgew.: Prismen. Trimorph II 113,4°, III unbeständig (54). Erstarrt zu Strahlen von I (grau), zerfällt bald zu schräg gestellten Lamellen von III. Mod. II bei R.T. beständig (bunt).

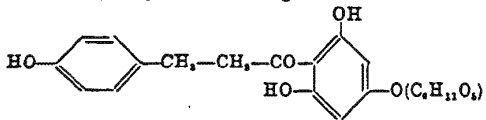
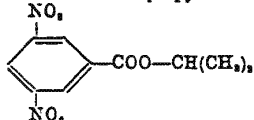
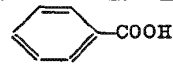
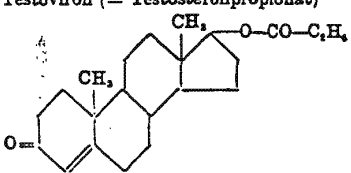
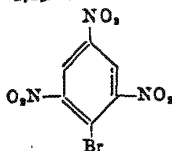
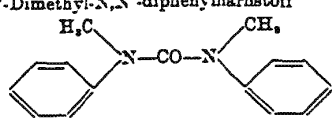
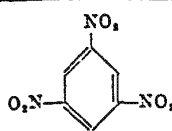
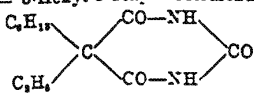
F. °C	Substanz	Eutekt. Temp. mit Benzil	Acetanilid	Glaspulver	Temp. °C	Besondere Kennzeichen
3 114	Fluoren 	68	93	1,5897	133	Ab 35° blättchenförmige, gelappte Blättchen. Gleichgew.: gelappte Platten.
9 114	Xylolmoschus	Siehe 107°				Modifikation I aus der Schmelze in breitstrahligen Sphärolithen. Nach einiger Zeit Umwandlung in gestreifte, bunte Aggregate II 107°.
2 114	5-Äthyl-5-[1-methylbutyl]-barbitursäure (= Pentobarbital)	Vgl. bei 129°				
1 114	m-Nitroanilin 	73	72	1,5795 1,5700	116—117 136 19	Gelb. Ab 80° Blättchen. Körner, Prismen. Gleichgew.: Tafeln. Erstarrt sphärolithisch.
2 114	p-Äthylbenzoesäure 	—72—	—75—	1,5101 1,5000	109—110 128—130 19	Ab 70° Körner, Blättchen, später Straßenpflaster. Gleichgew.: Prismen, Platten. Erstarrt zu Platten und Balken.
3 114	Clopane-hydrochlorid (= Cyclopentamin-hydrochlorid)  <i>1-cyclopentyl-methyl-2-propylamine</i>	81	—30—	1,4683 1,4584	121—123 155—157	Ab 100° Tröpfchen. Gleichgew.: Nadeln Stengel.
110—115	1-1-Amyltheobromin 	60	77 tr.	1,5101 1,5000	110—112 137—138 25	Gleichgew.: Körner, Prismen. Dimorph.
115	Acetanilid (= Antifebrin) 	78	115	1,5299 1,5204	109—111 136—137 27	Ab 60° Körner, später reichlich Nadeln und Prismen. Gleichgew.: Körner. Dimorph II 100° (32). Aus Schmelze II und sofortige Umwandlung.
115	Cholesterinacetat $C_{27}H_{48}(—O—CO—CH_3)$	80	100	1,4683 1,4584	144—147 171—173	Zwischen 70° und 80° Trübung durch enantiotrope Umwandlung. Gleichgew.: unregelmäßige Kristalle. Erstarrt sphärolithisch. Polymorph II 9°, III, IV und V kristallinflüssig (52, 55).
115	Veratrumssäurenitril 	74	87	1,5502 1,5403	123—124 139—141 16	Gelb. Ab 80° Nadeln. Stengel. Gleichgew.: Nadeln, Stengel. Erstarrt sphärolithisch.
116	Atropin 	82	89	1,5101 1,5000	111—112 134—136 27	Gleichgew. träg: Rauten.
9 116	Nipagin A (= p-Oxybenzoesäure-äthylester) 	76	73	1,5101 1,5000	117—118 139—140 27	Ab 85° Körner, sehr dicht. Gleichgew.: derbe Prismen.

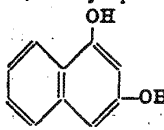
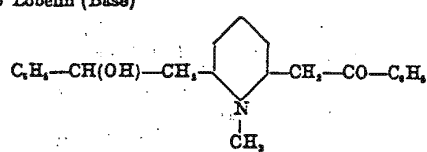
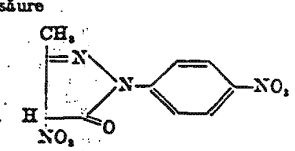
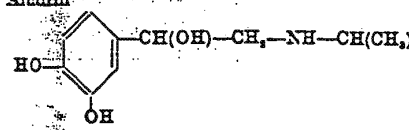
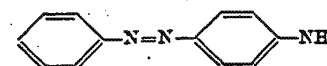
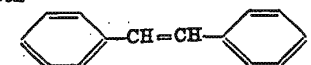
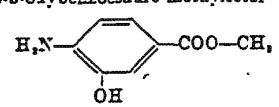
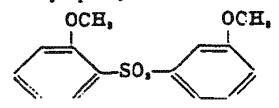
F. °C	Substanz	Eutekt. Temp. mit Benzil	Temp. mit Acetanilid	Glaspulver	Temp. °C	Besondere Kennzeichen
116	Pseudokoprosterin (= Epikoprosterin) 	83	109	1,4842 1,4683	124 162	Ab 100° kleine Nadelchen. Gleichgew. trág: Nadelbüschel. Erstarrt glasig.
116	$\omega, \omega, \omega', \omega'$ -Tetrabrom-o-xylol 	66	92	1,6353 1,6331 1,6321	110 134 24	Aromatischer Geruch. Ab 70° Nadeln, Spieße. Polymorph II 102°, III 98° (32).
116	p-Nitrobenzylecyanid 	70 tr.	79 tr.	1,5204 1,5101	135—138 159—160 24	Ab 60° Tröpfchen. Gleichgew.: Balken, Platten. Erstarrt breitstrahlig.
116	Butyramid $C_4H_9-CO-NH_2$	88	78	<1,4339		Geruch nach Buttersäure. Ab 40° verschiedenartige Sublimate. Gleichgew.: Balken, Platten. Erstarrt zu feinststrahligen Sphärolithen.
116	4,4'-Diacetoxy-2,2'-dimethyldiphenylsulfon 	73	86	1,5403 1,5299	108—109 129 23	Ab 60° wenig kleine Körner. Gleichgew. trág: Körner, Prismen. Erstarrt glasig.
117	p-Dimethylamino-azobenzol 	70	97	1,6877	etwa 200	Gelb. Ab 100° Nadeln, Blättchen. Gleichgew.: Rhomboide, Stengel. Erstarrt zu Sphärolithen. Polymorph (16).
117	p-Oxybenzaldehyd 	82	70	1,5795 1,5700	119—121 142 22	Ab 80° Nadeln, Prismen, Körner. Gleichgew.: Prismen, Körner. Erstarrt sphärolithisch.
117	Trasentin (= Diphenylacetyl-diäthylaminoäthanol-hydrochlorid) 	77	51	1,5502 1,5403	91—93 120—122 29	Bei etwa 100° teilweises Schmelzen und Wiederauskristallisieren. In Paraffin inhomogenes Schmelzen bei 94—96°.
117	3-Nitro-4-aminotoluol 	67	76	1,6126	115	Orangerot. Ab 65° Körner. Schmilzt trág. Gleichgew.: Körner, Prismen.
117	5-Nitro-2-methylphenol 	52	66	1,5502 1,5403	124—125 145—146 21	Ab 90° Körner, Blättchen. Gleichgew.: Körner, Prismen. Erstarrt zu grobstrahligen Sphärolithen.

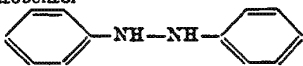
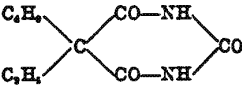
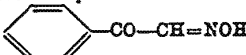
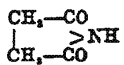
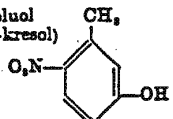
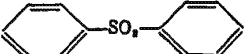
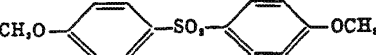
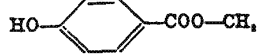
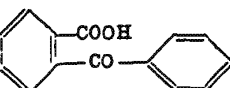
F: °C	Substanz	Eutekt. Temp. mit Benzil	Temp. mit Acetanilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
0 117	Phenylacetanilid 	77	81	1,5700—1,5611	110—111 133	Ab 80° Stäbchen, später Spieße. Gleichgew.: Stengel. Erstarrt zu Sphärolithen-mosaik.
1 117	N-Äthyl-N-butyl-N'-acetylharnstoff $\begin{matrix} \text{C}_2\text{H}_5 \\ \text{C}_4\text{H}_9 \end{matrix} \text{N} \text{---} \text{CO} \text{---} \text{NH} \text{---} \text{CO} \text{---} \text{CH}_3$	85	90	<1,4339		Ab 80° Nadeln. Gleichgew.: Nadeln. Erstarrt sphärolithisch.
2 117	Methyläthylketon-2,4-dinitrophenylhydrazon 	68	88	1,6353 1,6231	124 146—147	Orange. Ab 80° Stäbchen. Körner. Gleichgew.: Körner, Platten. Erstarrt sphärolithisch. Dimorph (49).
3 118	N-Methyl-N-äthyl-N',N'-diphenylharnstoff 	71	55	1,5403 1,5299	107—108 125—126	Ab 70° Körner, Tröpfchen. Gleichgew.: Körner, Prismen.
115—118	ThioBenzamid 	74	75	1,0483	131—132	Ab 90° Nadeln; bei Erwärmen stechender Geruch. Gleichgew.: Nadeln. Schmelze gelb. Erstarrt zu feinfaserigen Sphärolithen.
5 118	o-Benzoylresorcin 	77	94	1,5611 1,5502	122—124 148—150	Nur einzelne Tröpfchen. Gleichgew.: Rhomboide (beinahe Rechtecke), Prismen. Erstarrt zu kleinen (grauen) Sphärolithen.
116—118	Terpinhydrat	Siehe cis-Terpin				Ab 60° Blättchen; ab 70° Umlagerung zu cis-Terpin. F. 106; unter Paraffin F. 116—118°.
7 118	Hordenin (= [4-Oxyphenäthyl]-dimethylamin) 	84	93	1,5101 1,5299	118—120 125—126	Ab 90° Körner, Platten. Gleichgew.: Körner, Tafeln.
3 118	d-Pseudoephedrin 	82	92	1,4942	124—126	Ab 75° Blättchen, Prismen. Gleichgew.: Tafeln. Erstarrt sphärolithisch.
9 118	o-Dinitrobenzol 	72	86	1,5204 1,5101	125—128 147—150	Ab 60° Körner, Blättchen. Gleichgew.: Prismen. Erstarrt sphärolithisch.
10 118	Acetessigester-p-nitrophenylhydrazon 	77	83	1,6126	108—109	Gelb. Ab 95° Tröpfchen. Gleichgew.: Körner, Prismen.

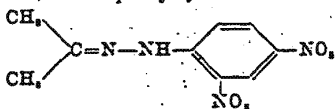
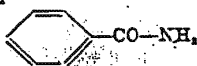
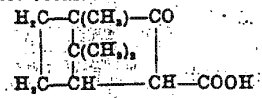
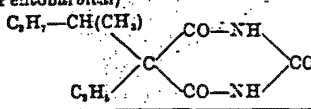
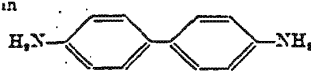
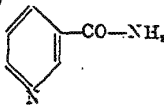
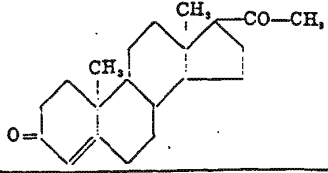
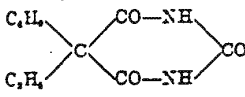
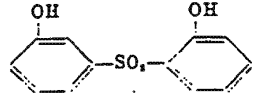
F. °C	Substanz	Eutekt. Temp. mit Benzil	Temp. mit Acetanilid	Glaspulver	Temp. °C	Besondere Kennzeichen
77 118	Lactophenin 	83	82	1,5101 1,5000	132—133 156 23	Ab 110° Nadeln, Blättchen, Stengel. Gleichgew.: Körner, Prismen. Erstarrt feinstrahlig sphärolitisch.
12 118	p,p'-Azoxyanisol 	79	98	1,6231 1,6126	154—158 172—175 17	Gelb. Bei 118° flüssige Krystalle, die bei 136° klar schmelzen (gelkreuzte Nicols). Aus Schmelze flüssige Kristalle, dann zweimal Umwandlung (12).
43 119	Chloracetamid <chem>CH3Cl-CO-NH2</chem>	87	87	1,4584— 1,4339		Ab 45° Prismen, Stengel, sehr reichlich. Gleichgew.: Balken. Erstarrt grobstrahlig.
4 119	dl-Mandelsäure 	84	86	1,5204 1,5101	103—106 137—138 23	Ab 90° Prismen, Rauten. Gleichgew.: Sechsecke, Prismen. Erstarrt zu feinkristallinen Sphärolithen von II 108° bei Erwärmen breitstrahlig I.
15 119	3-Brom-4-acetaminotoluol 	76	78	1,5403 1,5299	123—124 144—146 21	Ab 75° Nadeln, Spieße. Gleichgew.: Balken, Nadeln. Erstarrt zu Sphärolithen.
6 119,5	Erythrit <chem>HOCH2-[CH(OH)]2-CH2OH</chem>	93	108	1,4683 1,4584	135—136 169—170	Ab 110° Körner. Gleichgew.: Körner. Erstarrt sphärolithisch. Dimorph (28, 12).
7 120	Bernsteinsäureanhydrid 	87	103	<1,4339		Ab 55° Körner, sehr starke Sublimation. Gleichgew.: Prismen, Balken. Erstarrt zu feinstrahligen Sphärolithen.
8 120	Diäthylstilböstrol-diacetat 	94	104	1,5101 1,5000	114—115 136—137 27	Gleichgew.: Platten. Erstarrt sphärolithisch.
9 120	Acetylthioharnstoff <chem>CH3-CO-NH-CS-NH2</chem>	88	104	1,6011 1,5997	136—137 160—162 24	Ab 80° Blättchen, Körner. Gleichgew.: Tafeln. Erstarrt langsam zu Sphärolithen. Dimorph (14).
10 120	Adalin (= Bromdiäthyl-acetylharnstoff) <chem>(C2H5)2CBr-CO-NH-CO-NH2</chem>	89	102	1,4842	117—119	Ab 70° Nadeln, Stengel, Balken. Gleichgew.: Prismen, Balken. Erstarrt sphärolithisch. Polymorph (14).
57 120	d-Campheroxim 	87	101	1,4584	135	Camphergeruch. Ab 50° Körner, kurze Prismen, ab 110° wabenartig, bei 115° isotrop (Analysator ausschalten!). Gleichgew.: Platten.
22 120	Cyanacetamid <chem>CN-CH2-CO-NH2</chem>	97	112	1,4339— 1,4584		Ab 80° Körner, Blättchen. Gleichgew.: Prismen, Körner.
53 120	α-Cinnamylbenzylcyanid 	97	102	1,6715 1,6598	135—136 152—153 17	Gelb. Ab 80° Körner, Blättchen. Gleichgew.: Prismen, Körner.

F. °C	Substanz	Eutekt. Temp. mit Acetanilid	Temp. mit Phenacetin	Glaspulver	Temp. °C	Besondere Kennzeichen
121	β,β,β -Trichlormilchsäure $\text{CCl}_3\text{—CH(OH)—COOH}$	27	48	1,4683 1,4584	133—134 154—166	Geruch. Wenn bräunlich verfärbt, ab-saugen. Ab 80° Körner, Nadeln. Schmilzt trög. Gleichgew.: Nadeln, Balken. Dimorph (14).
121	2,4'-Diacetoxy-5,3'-dimethyldiphenylsulfon 	90	104	1,5299 1,5204	114—116 139—140	Gleichgew.: Körner, Prismen. 25
121	2,4,6-Tribromanilin 	97	109	1,6353 1,6231	131 151	Ab 75° lange Nadeln und Stengel. Gleichgew.: Balken, Stengel. Erstarrt sphärolithisch. 20
121	2,6-Diaminopyridin 	84	98	1,6126 1,6011	119—120 144—146	Geruch. Ab 65° Nadeln, Blättchen. Gleichgew.: Balken, Platten. Erstarrt unregelmäßig oder sphärolithisch. 25
121	3,5-Dinitroguajacol (= 3,5-Dinitro-1-oxo-2-methoxybenzol) 	84	97	1,5795	122—125	Gelb. Ab 100° Stäbchen, Nadeln. Gleichgew.: Stengel, Prismen. Dimorph II 114° (verfilzte Nadeln).
110—122	Chininacetat $\text{C}_{20}\text{H}_{24}\text{O}_4\text{N}_2 + \text{C}_2\text{H}_4\text{O}_2$	74 tr.	—75—	1,5611 1,5502	108 129	Schmelze erstarrt glasig. 21
122	β -Naphthol 	69	69	1,6011 1,5897	135—136 159—160	Ab 90° dünne, gelppte Blättchen. Gleichgew.: Spindeln, Stengel. Aus Schmelze Modifikation I, geht beim Abkühlen langsam in II über (56). 24
122	Pikrinsäure (= 2,4,6-Trinitrophenol) 	71	87	1,6011 1,5897	120 145—146	Gelb. Ab 115° Sublimation. Gleichgew.: Balken. Schmelze gelb. Trimorph II 101°, III 75° (57, 59). 25
122	N-[p-Nitrobenzoyl]-piperidin 	77	95	1,5299 1,5204	137—138 162—164	Ab 90° Körner, Nadeln. Gleichgew.: Balken, Tafeln. Erstarrt feinstrahlig sphärolithisch. 22
122	m-Aminophenol 	82	94	1,5795 1,5700	122—125 147—148	Ab 65° Körner, Nadeln. Gleichgew.: Balken, Prismen. Erstarrt sphärolithisch. 24
122	Butyraldehyd-2,4-dinitrophenylhydrazon 	86	95	1,6353	117	Gelb. Ab 100° Körner. Gleichgew.: Platten, Balken. Dimorph II 110° (49).

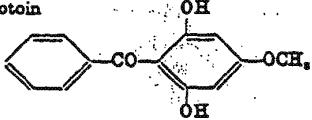
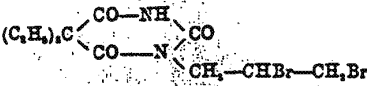
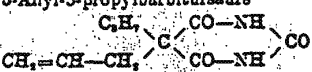
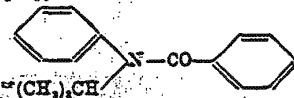
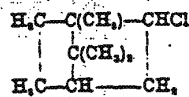
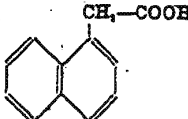
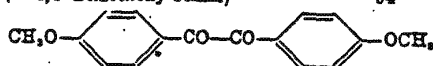
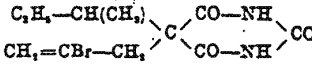
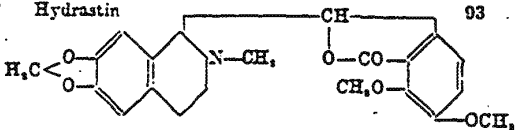
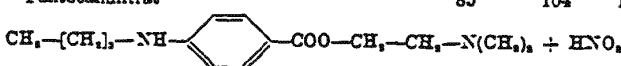
F. °C	Substanz	Eutekt. Temp. mit Acet-anilid	Temp. mit Phen-acetin	Glas-pulver	Temp. °C	Besondere Kennzeichen
120—122	Phloridzin + 2 H ₂ O (= Phloretin glucosid) 	82	99	1,5502	140—155	Schmilzt inhomogen unter Abscheidung wasserfreier Kristalle mit F. 170—180°. Schmilzt trög, Schlieren. Brechungsindex nicht genau bestimmbar.
122	3,5-Dinitrobenzoesäure-isopropylester 	92	105	1,4938	125—126	Ab 90° Nadeln, Körnchen. Gleichgew.: Stengel.
122,5	Benzoesäure 	76	88	1,5000 1,4938	134—135 148—149	Ab 60° Nadeln, Stengel, Spieße, schiefwinkelige Blättchen. Gleichgew.: Balken.
123	Jodoform CHJ ₃	101	114	Zersetzung		Geruch. Gelb. Ab 70° sechseckige Blättchen. Gleichgew.: Blättchen; sinkt ab. Schmelze dunkelbraun. Opt. einachs. negativ. Brechungsindex über 1,74 (in Methyljodid löslich).
123	Testoviron (= Testosteronpropionat) 	70	90	1,5101 1,5000	94 116—117 23	Gleichgew.: Körner, Prismen. Erstarrt glasig; bei Erwärmen Sphärolithe aus II und rasche Umwandlung.
123	Pikrylbromid (= 2,4,6-Trinitro-brombenzol) 	83	100	1,5785 1,5700	133—135 153—155 20	Ab 90° Körner, sechseckige Blättchen. Gleichgew.: Blättchen. Erstarrt sphärolithisch. Dimorph II 121° (40).
123	N,N'-Dimethyl-N,N'-diphenylharnstoff 	84	99	1,5299 1,5104	138—140 161—163	Ab 80° Körner, Prismen. Gleichgew.: Balken. Erstarrt feinfasrig.
123,5	s-Trinitrobenzol 	79	99	1,5611 1,5502	128—130 149—150 21	Hellgelb. Ab 75° Körner, Stengel. Blättchen. Gleichgew.: Prismen, Platten. Polymorph II 110°, III 109°, IV 106°, V 88° (39).
124	Hebaral (= 5-Hexyl-5-äthylbarbitursäure) 	84	102	1,4584	134—136	Ab 100° kleine Nadeln. Gleichgew.: Nadeln. Erstarrt glasig.
124	Chloralformamid CCl ₃ -CH(OH)-NH-CHO	79	92	1,4842	116—118	Ab 55° Körner, Blättchen. Erstarrt wabenartig. Bei Brechungsindex rasch arbeiten, da bald Zersetzung und Gasblasen.

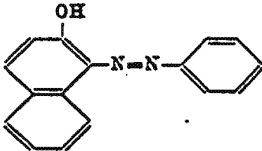
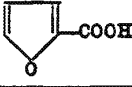
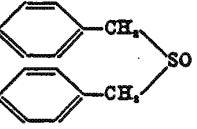
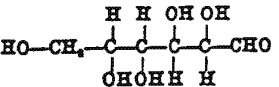
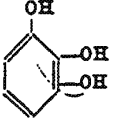
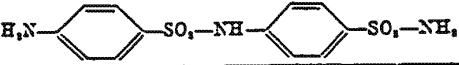
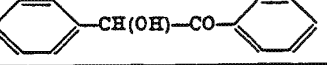
F. °C	Substanz	Eutekt. Acet-anilid	Temp. mit Phen-acotin	Glas-pulver	Temp. °C	Besondere Kennzeichen
5 124	Naphthoresorcin (= 1,3-Dioxynaphthalin) 	47	56	1,6483	106—108	Rötlichbraun, ab 100° Tröpfchen, schmilzt und kristallisiert sehr träg. Schmelze bald braun, für Brechungsindex durch Absaugen reinigen.
115—125	Lobelin (Base) 	88	—110—	1,5403 1,5299	114—116 137—139	Erstarrt glasig, beim Auflegen auf den warmen Heiztisch büschelförmig.
7 125	Pikrolonsäure 	88	99	Zersetzung		Gelb. Ab 115° „Kochen“ in Kristallen. Allmähliches Schmelzen und Zersetzen mit Abscheidung von Blättchen und Nadeln mit F. über 200°. Aus Alkohol: Rechtecke, Achtecke; gerade Auslöschung. $\alpha' = 1.74$ bei 28°, $\gamma' > 1.74$. Auf der Heizbank F. 150° nach zehn Sekunden, 140° nach 20 Sekunden. Schmelze braun.
120—125	Aludrin 	103 tr.	118 tr.	1,5611	105—108	Ab 90° wenig Körner. Erweicht langsam. Schmelze zäh. Brechungsindex wegen Gasblasen erschwert.
9 125	p-Aminoazobenzol 	85	97	1,6877	183—199	Orange. Ab 95° Nadeln, Stäbchen. Gleichgew.: Balken. Dimorph (16, 12).
180 125	Stilben 	97	108	1,6126 1,6011	116 139 23	Ab 70° Körner, Blättchen. Gleichgew.: Blättchen, Rauten.
45 125	Äthylidendiurethan $\text{CH}_3\text{—CH(NH—COO—C}_2\text{H}_5)_2$	80	104	<1,4339		Ab 80° Nadeln, Stäbchen. Gleichgew.: Spieße, Nadeln. Erstarrt zu kleinen feinfaserigen Sphärolithen mit γ' radial.
452 125	Tetradecandicarbonsäure $\text{HOOC—[CH}_2\text{]}_{12}\text{—COOH}$	97	110	<1,4339		Schmilzt meist unscharf. Gleichgew.: Linsen, Platten, Bänder, strauchartige Aggregate. Wenig unterkühlbar. Mit Nikotinsäureamid zwei Molekülverbindungen, F. 129°, F. 129,5°. Erstarrt zu feinkörnigen Aggregaten, dazwischen schmale Rauten.
453 125 483	4-Amino-3-oxypbenzoesäure-methylester 	75	89	1,5204 1,5101	136—138 153—154 17	Ab 90° Körner, Blättchen. Gleichgew.: Blättchen, Prismen. Erstarrt zu Sphärolithen. Polymorph II 115°, III 107°.
454 125	2,3'-Dimethoxydiphenylsulfon 	94	106	1,5611 1,5502	114—115 139—140 25	Gleichgew. träg: Prismen. Erstarrt sphärolithisch.

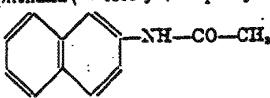
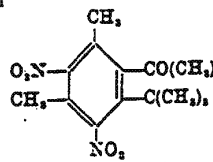
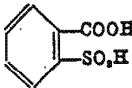
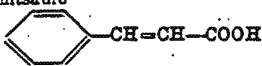
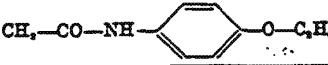
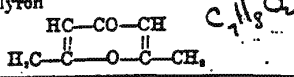
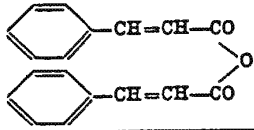
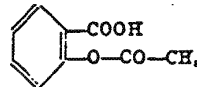
F. °C	Substanz	Eutekt. Acet-anilid	Temp. mit Phen-acetin	Glas-pulver	Temp. °C	Besondere Kennzeichen
5 126	Hydrazobenzol 	88	101	1,6011 1,5897	104—105 135—137 31	Ab 80° sechseckige Blättchen. Alte Präparate oberflächlich gelb und F. tiefer.
6 126	Soneryl (= Neonal = 5-Butyl-5-äthyl-barbitursäure) 	80	99	1,4683 1,4584	136—137 153	Ab 60° Nadeln. Gleichgew.: Balken, Nadeln.
7 126	ω-Isonitroso-acetophenon 	77	88	1,5403— 1,5502		Ab 85° Körner, Prismen, Nadeln. Gleichgew.: Balken, sinkt ab. Erstarrt in feinstrahligem Sphärolithen.
8 126	Succinimid 	97	105	1,4842 1,4683	102—104 139—140	Ab 65° Körner, wenig Stäbchen und Blättchen. Gleichgew.: Körner. Erstarrt sphärolithisch. Dimorph II 124° (59, 60).
9 126	Hexadecandicarbonsäure $\text{HOOC}-(\text{CH}_2)_{14}-\text{COOH}$	99	112	<1,4339		Gleichgew. trägt: Platten (graue Interferenzfarbe), Spieße. In Platten bei Raumtemperatur viele parallele Quersprünge. Bei raschem Erstarren feinkristallin. Mit Nikotinsäureamid zwei Molekülverbindungen, F. 129,5°, F. 132°.
10 126	6-Nitro-3-oxy-toluol (= 4-Nitro-m-kresol) 	58	72	1,5611 1,5700	140 122—123 1	Ältere Präparate braun, Umsublimieren bei 100°. Unangenehmer Geruch. Ab 90° Stäbchen, Blättchen. Gleichgew.: Körner, Platten. Erstarrt sphärolithisch.
126.5	Sulfonal (= Bis-Äthylsulfon-dimethyl-methan) $(\text{CH}_3)_2\text{C}(\text{SO}_2-\text{C}_2\text{H}_5)_2$	91	107	1,4584 1,4339	122—124 200—204	Ab 80° Körner, rechteckige und quadratische Blättchen. Gleichgew.: Körner, Prismen.
2 127	Diphenylsulfon 	89	104	1,5611 1,5502	119—121 142—143 23	Ab 70° Stäbchen, Rhomboide. Gleichgew.: Rhomboide, Platten. Erstarrt sphärolithisch. Polymorph (61, 12).
3 127	4,4'-Dimethoxydiphenylsulfon 	91	106	1,5700 1,5611	118—120 139—140 21	Erstarrt meist glasig, bei Erwärmen kristallin.
4 127	Nipagin M (= p-Oxybenzoesäure-methylester) 	74	87	1,5204 1,5101	132—133 156—163 54	Ab 90° Körner. Gleichgew.: Körner. Polymorph II 116°, III 110°, IV 110°, V 100°, VI 106° (10).
5 127	p-Bromnitrobenzol 	91	105	1,5502 1,5403	139—140 159—160 20	Bittermandelgeruch. Ab 65° Nadeln, Spieße. Gleichgew.: Spieße, Stäbchen. Erstarrt sphärolithisch.
6 127	o-Benzoylbenzoesäure 	64	82	1,5700 1,5611	135—138 159—160 23	Ab 80° wenig Blättchen, Körner. Gleichgew.: Blättchen, Körner. Unterkühlbar. Bei Erwärmen ab 80° träge Kristallisation. Sphärolithe zweier Modifikationen II 123° (S).

F. °C	Substanz	Eutekt. Acet-anilid	Temp. mit Phen-acetin	Glas-pulver	Temp. °C	Besondere Kennzeichen
128	Aceton-2,4-dinitrophenylhydrazon 	92	105	1,6553 1,6231	156 179 2.2	Orange. Ab 75° Körner, später Nadeln. Gleichgew.: Platten. Erstarrt sphärolithisch.
125	Benzamid 	82	98	1,5403 1,5299	129 152—153	Ab 95° Blättchen. Gleichgew.: Stengel, Balken. Dimorph (62, 28).
128	d-Camphocarbonsäure 	70	84	1,4584	110—115	Ab 90° Körner. Ab 115° Gasblasen. Für Brechungsindex bei 130° auflegen, nach Schmelzen rasch mit Metallwürfeln abkühlen.
129	5-Äthyl-5-[1-methylbutyl]-barbitursäure (= Pentobarbital) 	85	102	1,4683 1,4584	136—138 161—162	Ab 70° spärliche Sublimation. Gleichgew.: Spieße, Balken. Erstarrt trüg. Dimorph II 114° (63).
129	p-Dijodbenzol 	99	112	1,0483	143—145	Ältere Präparate grau. Ab 70° Stäbchen, Blättchen. Gleichgew.: schiefwinkliger Balken. Umsublimieren bei 110°. Brechungsindex durch Flüchtigkeit erschwert.
129	Donzidin 	88	98	1,6598 1,0483	143 171—173 2.9	Gleichgew.: Balken, Prismen. Wiedererstarrte Schmelztropfen F. 125°, 121°. Polymorph (64).
129	Nicotinsäureamid (= Pyridin-β-carbonsäureamid) 	89	106	1,5502 1,5403	130 166 2.6	Ab 90° wenig Nadeln, Stäbchen. Gleichgew.: Stengel, Balken. Erstarrt sphärolithisch. Polymorph II 116°, III 113°, IV 111°, V 110°, VI 105° (65).
129	Progesteron (= Proluton) 	73	94	1,5204 1,5101	122—123 152—154 2.0	Ab 100° Tröpfchen. Gleichgew.: Körner. Erstarrt sphärolithisch. Dimorph (66).
129	Idobutal (= 5-Allyl-5-butylbarbitursäure) 	85	99	1,4683 1,4584	146 169	Ab 100° Nadeln, Stengel. Gleichgew.: Nadeln, Stengel. Erstarrt bei Raumtemperatur schwer zu Sphärolithen. Polymorph (63).
129	o',m-Dioxydiphenylsulfon 	68	81	1,5795 1,5700	146—147 172—173 2.5	Ab 100° Stäbchen, Blättchen. Gleichgew. trüg. Stengel. Erstarrt glasig.

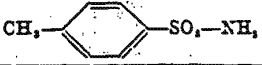
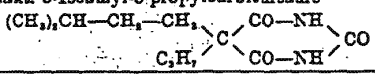
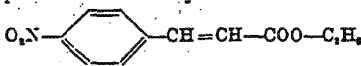
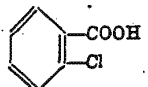
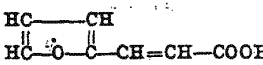
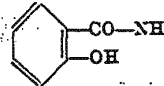
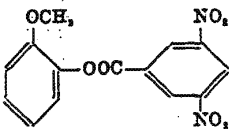
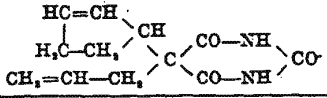
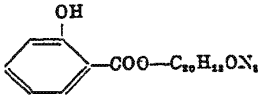
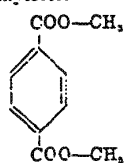
F. °C	Substanz	Eutekt. Acet-anilid	Temp. mit Phen-acetin	Glas-pulver	Temp. °C	Besondere Kennzeichen
129	1-Phenyl-3-methyl-pyrazolon $\begin{array}{c} \text{CH}_3-\text{C}=\text{CH} \\ \diagup \quad \diagdown \\ \text{HN} \quad \text{C}=\text{O} \\ \quad \quad \quad \text{N} \\ \quad \quad \quad \text{C}_6\text{H}_5 \end{array}$	82	95	1,5611 1,5502	121—122 139—140 17	Ab 100° Blättchen, Stäbchen. Gleichgew. trüg: Körner, Stäbchen.
129	Dekamethylendicarbonsäure HOOC-(CH ₂) ₈ -COOH	95	107	<1,4339		Gleichgew.: unregelmäßige Formen. Bildet mit Nikotinsäureamid zwei Molekülverbindungen, F. 123° und 125° (Kontaktpräparat).
129	Hexaäthylbenzol $\begin{array}{c} \text{C}_6\text{H}_2 \\ \diagup \quad \diagdown \\ \text{H}_3\text{C}_2 \quad \text{C}_2\text{H}_5 \\ \diagdown \quad \diagup \\ \text{H}_3\text{C}_2 \quad \text{C}_2\text{H}_5 \\ \diagup \quad \diagdown \\ \text{C}_2\text{H}_5 \end{array}$	110	127	1,4683 1,4584	144—146 165—168	Ab 69° Nadeln, Rhomboide, Stengel; dichte Sublimat. Gleichgew.: Prismen, Balken. Erstarrt strahlig.
125—129	Optochinbase + H ₂ O (= O(Ch)-Äthylhydrocyprein) $\begin{array}{c} \text{CH} \\ \diagup \quad \diagdown \\ \text{C}_6\text{H}_5-\text{HC} \quad \text{CH}_2 \\ \diagdown \quad \diagup \\ \text{H}_3\text{C} \quad \text{CH}_2 \\ \diagup \quad \diagdown \\ \text{N} \quad \text{CH} \end{array}$	76 tr. 96 tr.	70 tr. 112 tr.	1,5611	109—112	Ab 76° teils homogenes teils inhomogenes Schmelzen, später entweicht Wasser allmählich. Schmelze sehr zäh mit vielen Blasen.
127—129	4-Toluolsulfonyl-β-naphthylamid $\text{CH}_3-\text{C}_6\text{H}_4-\text{SO}_2-\text{NH}-\text{C}_{10}\text{H}_7$	84	98	1,6126 1,6011	111—113 143—144 32	Schmilzt trüg, Schmelze leicht rosa. Erstarrt glasig.
129,5	β-Acetylphenylhydrazin $\text{C}_6\text{H}_5-\text{NH}-\text{NH}-\text{CO}-\text{CH}_3$	88	102	1,5299	142	Ab 100° Nadeln, Blättchen. Gleichgew.: Körner, Spieße. Dimorph.
129,5	Piperin $\text{H}_3\text{C}-\text{O}-\text{C}_6\text{H}_4-\text{CH}=\text{CH}-\text{CH}=\text{CH}-\text{CO}-\text{N}-\text{C}_5\text{H}_{10}$	82	101	1,6231 1,6126	129 147—148 19	Gelb. Geruch. Gleichgew.: Körner. Erstarrt teils glasig, teils kristallin.
130	5-Jod-2-aminopyridin $\text{J}-\text{C}_5\text{H}_3\text{N}_2$	90	101	1,6598 1,6483	98—99 124—125 26	Hellgelb. Ab 80° Blättchen. Gleichgew.: Blättchen, oft schiefwinklig. Erstarrt sphärolithisch.
130	p-Acetanisidid $\text{CH}_3-\text{O}-\text{C}_6\text{H}_4-\text{NH}-\text{CO}-\text{CH}_3$	86	112	1,5299 1,5204	118 137—140 21	Ab 105° Rechtecke, Stengel. Gleichgew.: Rechtecke, Prismen, Platten. Erstarrt sphärolithisch.
130	o-Tolidin (= 3,3'-Dimethylbenzidin) $\text{H}_2\text{N}-\text{C}_6\text{H}_3(\text{CH}_3)_2-\text{C}_6\text{H}_3(\text{CH}_3)_2-\text{NH}_2$	91	104	1,6353 1,6231	135—138 160—162 24	Ab 103° Körner, Nadeln. Gleichgew.: Prismen, Blättchen.

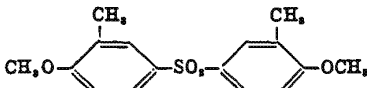
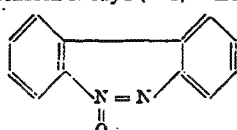
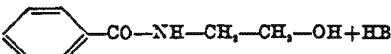
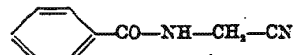
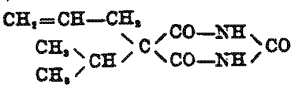
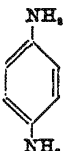
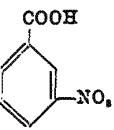
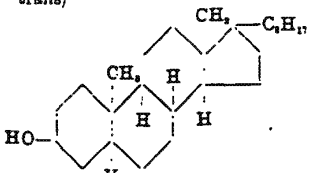
F. °C	Substanz	Eutekt. Temp. mit Acet-anilid	Temp. mit Phen-acetin	Glas-pulver	Temp. °C	Besondere Kennzeichen
130	Cotoin 	71	86	1,6011 1,5897	137 159 22	Gelb. Ab 120° Körner, Stengel, Prismen. Gleichgew.: Prismen, Körner.
131	3-[β,γ-Dibrompropyl]-5-diäthyl-barbitursäure 	94	108	1,5101 1,5000	132 167 35	Schmelze erstarrt glasig, bei Erwärmen Strahlenbüschel.
131	5-Allyl-5-propylbarbitursäure 	85	102	1,5101 1,5000	140 167 27	Ab 115° Nadeln, Blättchen. Gleichgew.: trüg: griesartig. Schmelze erstarrt glasig.
131	dl-Äpfelsäure HOOC-CH ₂ -CH(OH)-COOH	85	104	1,4584	114—115	Gleichgew. sehr trüg: sechseckige oder rhomboidische Kristalle.
131	N-Isopropyl-benzanilid 	93	108	1,5299 1,5204	139—140 162—164 23	Ab 90° Nadeln, Blättchen. Gleichgew.: Stengel.
128—131	Bornylchlorid 	92	111	1,4339— 1,4584		Camphergeruch. Ab 30° isotrop, ge-lappte Sterne, Spieße, Sicheln: starke Sublimation. Straßenpflaster. Gleich-gew.: Körner, gelappte Formen. Eutek-tische Temperatur mit Sublimaten bestimmen.
131	α-Naphthyllessigsäure 	71	86	1,5795 1,5700	133—134 155—156 22	Ab 35° Körner, Stäbchen. Gleichgew.: Körner. Erstarrt sphärolithisch.
131	Anisil (= 4,4'-Dimethoxy-benzil) 	94	111	1,5795 1,5700	134—136 154—156 20	Gelb. Gleichgew.: Stengel, Balken. Er-starrt zu Sphärolithen.
132	Pernocton (= 5-sec.-Butyl-5-[β-bromallyl]-barbitursäure) 	81	101	1,5101 1,5000	133—140 163—165 20	Ab 116° Tröpfchen. Erstarrt bei län-gerem Liegen.
132	Hydrastin 	93	106	1,5611 1,5502	131—134 159—162 25	Gleichgew.: Tafeln. Schmelze gelb, er-starrt glasig.
132	Pantocainnitrat 	85	104	1,5299	143	Gleichgew.: Körner, trüg. Dimorph.
132	Thiodiglykolsäure HOOC-CH ₂ -S-CH ₂ -COOH	77	93	1,4936 1,4842	125—126 165—166	Widerlicher Geruch. Ab 100° Blättchen, Körner. Gleichgew.: Körner, Prismen. Erstarrt zu Sphärolithen.

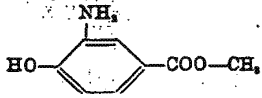
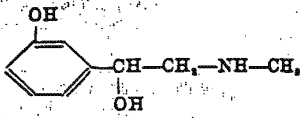
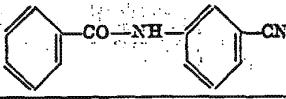
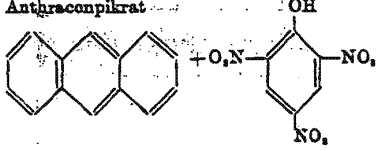
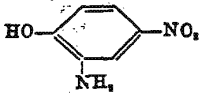
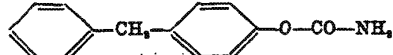
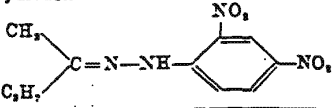
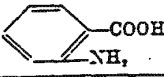
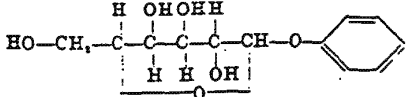
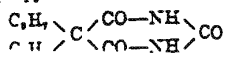
F.°C	Substanz	Eutekt. Acet- anilid	Temp. mit Phen- acetin	Glas- pulver	Temp. °C	Besondere Kennzeichen
132	Sudan I (= 1-Benzolazo-2-naphthol) 	102	114	—	—	Rot. Ab 105° Nadeln. Gleichgew.: Nadeln. Erstarrt sphärolithisch. Dimorph. Brechungsindex wegen Farbe unmöglich.
132	Brenzschleimsäure 	69	87	1,4842— 1,4783—	— 1,46%	Ab 60° Nadeln, Körner, Prismen, Blättchen. Gleichgew.: Blättchen, Stengel. Brechungsindex wegen Zersetzung und Flüchtigkeit erschwert.
133	Sebacinsäure $\text{HOOC}-(\text{CH}_2)_8-\text{COOH}$	91	106	<1,4339	—	F. meist unscharf. Gleichgew.: Spindeln, bandförmige Kristalle. Schmelze wenig unterkühlbar. Graue, flächig-strahlige Sphärolithe.
133	Pentaacetyl-β-d-glucose $\text{C}_6\text{H}_7\text{O}(\text{O}-\text{CO}-\text{CH}_3)_5$	98	112	<1,4339	—	Ab 100° Nadelchen. Gleichgew.: Stengel. Erstarrt glasig. Bei Erwärmen kristallin.
133	Dibenzylsulfoxyd 	79	99	1,5700	etwa 140 sinkt ab	Gelblich. Ab 110° Blättchen, Spieße. Gleichgew.: Balken, Spieße.
128—133	d-Mannose 	114 tr.	127 tr.	1,5101	136—139	F. trög. Schmelze zäh, daher bleiben viele Luftblasen. Erstarrt glasig.
133,5	Pyrogallol (= 1,2,3-Trioxybenzol) 	59	71	1,5611 1,5602	133—135 156—157	Ab 100° Blättchen, Balken. Gleichgew.: viereckige Blättchen.
110—134	Chinacinnamylat $\text{C}_{18}\text{H}_{15}\text{O}_2\text{N}_3 + \text{C}_6\text{H}_5\text{O}_2$	76	79	1,5397 1,5775 1,5795	107—109 137—138 20	Bei 120° schmilzt Hauptmasse. Erstarrt glasig. In Paraffin ab 90° wenig Dampfblasen, zwischen 90—100° inhomogenes Schmelzen unter Abscheidung von Nadeln, die sich bei 134° auflösen.
112—134	Cliron C (= p-Sulfanilylamino-benzolsulfonamid) 80 	80	95	1,6231 1,6126	137—139 170—173 30	Schmilzt langsam. Gleichgew.: kein Wachsen. Erstarrt glasig (Reimers [87]). Schmilzt in Paraffin erst ab 125° sehr trög. ab 130° Gasblasen.
134	Benzoin 	96	110	1,5592 1,5403	132—135 154 20	Ab 90° Nadeln, Stengel, Prismen. Erstarrt feinstrahlig sphärolithisch. Polymorph (68).
134	Sorbinsäure $\text{CH}_3-\text{CH}=\text{CH}-\text{CH}=\text{CH}-\text{COOH}$	85	102	1,4842	134—136	Säuerlicher Geruch. Ab 60° Nadeln, Körner, Prismen. Gleichgew. sinkt ab: Körner. Brechungsindex wegen Flüchtigkeit erschwert. Unreine Präparate keine verfärbt. Umsublimation bei 120°.

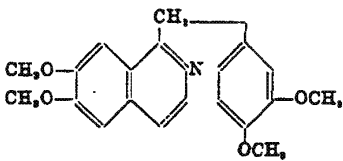
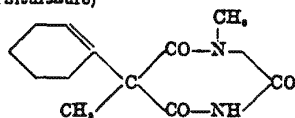
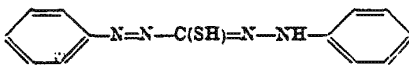
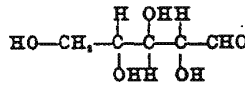
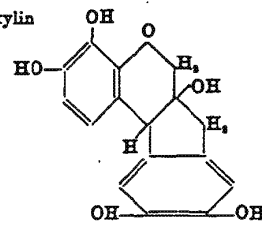
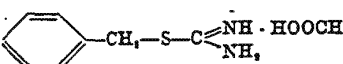
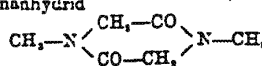
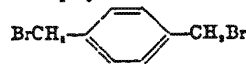
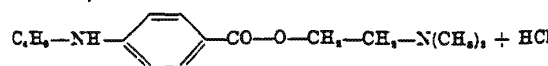
F. °C	Substanz	Eutekt. Temp. mit Acetanilid	Temp. mit Phenacetin	Glaspulver	Temp. °C	Besondere Kennzeichen
134	β -Acetnaphthalid (= Acetyl- β -naphthylamin) 	87	102	1,6011 1,5897	146—147 170—172 24	Ab 90° Nadeln, Blättchen. Gleichgew.: Blättchen, Balken. Erstarrt sphärolithisch. Polymorph II 133°, III 128°.
134	4,6-Dinitro-1,5-dimethyl-3-tert.-butyl-2-acetylbenzol 	94	109 tr.	1,4936 1,4842	133 153—155	Ab 90° Nadeln, Körner. Gleichgew.: Blättchen, Balken. Erstarrt sphärolithisch. Dimorph.
132—135	Raffinose (= Melitose) $C_{12}H_{22}O_{11} + 5 H_2O$	114	130	1,5204 1,5299	166—168 129—131 37	Beim Schmelzen Gasblasen. Schmelze sehr zäh. Erstarrt glasig. Bei 110° aufgelegt, vollständiges Schmelzen des Hydrates. Schmilzt in Paraffin homogen bei 78—80°. Für Brechungsindex bei 110° trocknen.
135	o-Sulfobenzoesäure 	32	127	1,5299 1,5204	125—127 158—159 33	Schmilzt als Hydrat 64—67°; kristallisiert manchmal wieder aus und schmilzt dann bei 135° (nur wenn scharf getrocknet, F. 140°). Eutektische Temperatur mit Acetanilid gilt für Hydrat. In Paraffin homogenes Schmelzen bei 64—67° (siehe dort).
135	Harnstoff $H_2N-CO-NH_2$	102	125	1,4683	156	Ab 110° Spieße. Gleichgew.: Körner, Prismen.
135	(trans)-Zimtsäure 	84	93	1,5611 1,5502	136 156—157 21	Ab 100° Rauten, quadratische Blättchen. Gleichgew.: Blättchen. Dimorph II 133° (60, 70).
135	Phenacetin $CH_3-CO-NH-C_6H_4-O-C_2H_5$ 	90	135	1,5101 1,5000	134—135 156—157 22	Ab 100° Nadeln, Blättchen, Balken. Gleichgew.: Rechtecke, Balken. Polymorph II 129° (71). Aus Schmelze II, bei Erwärmen Umwandlung.
130—135	p-Aminosalicylsäure	Siehe 143—148°				Schmilzt unter starker Zersetzung und Gasblasen.
135	2,6-Dimethylpyron $H_3C-C(=O)-O-C(=O)-CH_3$ 	68	91	1,4683	146—147	Ab 60° Stengel, Nadeln, kurze Prismen. Gleichgew.: sechseckige Blättchen und Rauten. Erstarrt zu verfilzten Kristallen.
135,5	Zimtsäureanhydrid 	70	96	1,5897 1,5795	141—142 164—165 23	Ab 100° kleine Körner. Gleichgew.: Prismen.
136	Malonsäure (= Methandicarbonsäure) $\begin{array}{c} \text{COOH} \\ \\ \text{CH}_2 \\ \\ \text{COOH} \end{array}$	62	84	<1,4339		Ab 100° Nadeln, Prismen, Rauten. Prismen und Rauten schmelzen teils bei 130—132°; erstarren oft wieder.
130—136	Acetylsalicylsäure (= Aspirin) 	81	97	1,4842— 1,4936		Ab 105° Nadeln, Stengel. Gleichgew. trüg: Balken; teilweise Zersetzung. Auf Heizbank F. 143, sinkt ab (72).

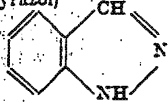
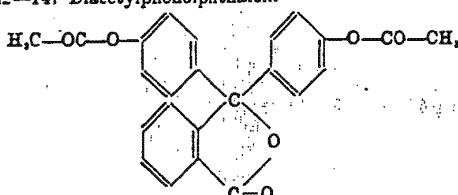
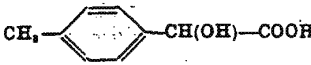
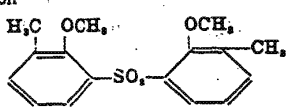
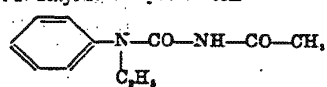
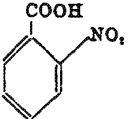
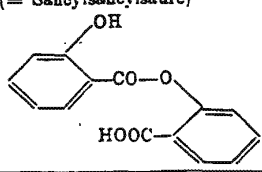
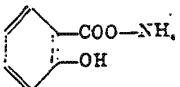
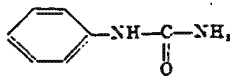
F. °C	Substanz	Eutekt. Temp. mit Acetanilid	Temp. mit Phenacetin	Glaspulver	Temp. °C	Besondere Kennzeichen
≡ 136	Isobutyryl-p-phenetidid <chem>CC(C)C(=O)Nc1ccc(OCC)cc1</chem>	85	104	1,4836 1,4842	129—131 150—151	Ab 90° Nadeln. Gleichgew.: Stengel. Erstarrt sphärolithisch. Schmelze über 140° rosa.
≡ 136	Phenylpropioisäure <chem>c1ccccc1C#CC(=O)O</chem>	53	71	1,5403	142—146	Ab 85° Körner, Nadeln, Spieße. Gleichgew.: Spieße. Schmelze gelblich. Erstarrt sphärolithisch.
≡ 136	d-Galaktonsäure-γ-lacton <chem>OCC(O)C(O)C(O)C(=O)O</chem>	109	128	1,5000 1,4936	124—128 144—146	Gleichgew.: Körner, Tafeln. Schmelze erstarrt glasig.
≡ 136	Sitosterin <chem>CC(C)[C@H](CCCC(C)C)[C@H]1CC[C@@H]2[C@@]1(CC[C@H]3[C@H]2CC=C4[C@@]3(CC[C@@H](C4)O)C)C</chem>	105	115	1,4842	146—148	Gleichgew.: Kleinkristalle. Erstarrt sphärolithisch.
≡ 136	Resorcyraldehyd (= 2,4-Dioxybenzaldehyd) <chem>Oc1cc(O)cc(C=O)cc1</chem>	70	84	1,6011— 1,5897		Ab 100° Körner, Plättchen. Gleichgew.: Balken. Erstarrt feinstrahlig sphärolithisch.
≡ 136	p,p'-Azoxyanisol <chem>c1ccc(cc1)Oc2ccc(cc2)Oc3ccccc3</chem>	siehe 118°				Wird bei 118° kristallinflüssig und bei 136° isotrop.
≡ 137	1,6-Dioxynaphthalin <chem>Oc1ccc2cc(O)ccc2cc1</chem>	50 tr.	64 tr.	1,6483	110—113	Ältere Präparate wegen oberflächlicher Zersetzung schwarz. Umsublimieren bei 125° oder Adsorptionsäule (Alkohol und Al ₂ O ₃). Ab 105° Nadeln, Körner. Gleichgew.: Prismen, Körner.
≡ 138	2,4-Dinitro-α-naphthol <chem>Oc1ccc2cc([N+](=O)[O-])ccc2cc1[N+](=O)[O-]</chem>	93	109	1,6598	138—142	Gelb. Ab 120° Nadeln. Gleichgew.: Nadeln. Unreine Präparate beim Schmelzen viele Blasen. Umsublimieren bei 120°. Brechungsindex durch teilweise Zersetzung erschwert.
≡ 135 (139)	δ-Hexachlorcyclohexan <chem>ClC1(Cl)CC(Cl)(Cl)CC1(Cl)Cl</chem>	78	92	1,5101	157	Geruch. Ab 80° anisotrope Stäbchen, Körner, Blättchen mit F. 138° (Gleichgew.: Spieße) und isotrope Sechsecke, Stengel mit F. 139° (Gleichgew.: Prismen). Enantiotrop dimorph Up. 132° (64).
≡ 139	Sandoptal (= 5-Isobutyl-5-allylbarbitursäure) <chem>CC(C)C(=O)Nc1ccc(C=C)cc1C(=O)N</chem>	92	107	1,4842	100—112	Ab 100° kurze, dicht gelagerte Nadeln. Gleichgew.: kurze Stengel.
135—139	S-Benzyl-thiuroniumacetat <chem>CC(=O)Nc1ccc(CS=C(N)N)cc1</chem>	104	119	—		Zwischen 70—80° Trübung und Umlagerung zu körnigen Aggregaten. Ab 110° Nadeln, Blättchen. Geruch beim Erhitzen. Schmilzt unter Zersetzung und Abscheidung von Körnern und Tropfen. In Paraffin bei 100—110° Umlagerung und Blasenbildung. Aus Benzol + Alkohol kleine lange, flache Rechtecke mit γ' längs; gerade Auslöschung; γ' wenig über 1,62, α' = 1,57.

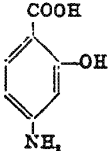
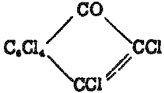
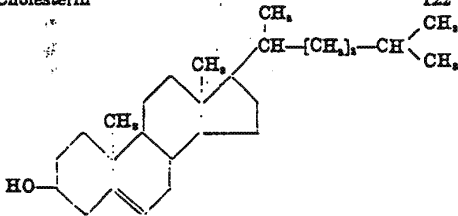
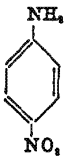
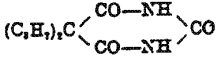
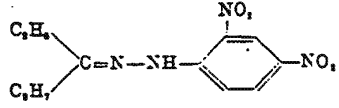
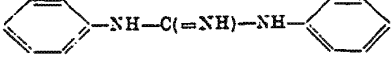
F. °C	Substanz	Eutekt. Acet-anilid	Temp. mit Phen-acetin	Glas-pulver	Temp. °C	Besondere Kennzeichen
139	p-Toluolsulfonsäuresamid 	83	99	1,5204 1,5101	143—145 171—173	Ab 90° Körner, später Blättchen. Rauten. Gleichgew.: Platten. Dimorph. 28
135—139	inakt. 5-Isosämyl-5-propylbarbitursäure $(\text{CH}_3)_2\text{CH}-\text{CH}_2-\text{CH}_2-\text{C}(\text{C}_2\text{H}_5)_2$ 	89	107	1,4584	131—132	Ab 90° Nadeln. Blättchen. Schmilzt trüg. Dimorph.
139	p-Nitrozimtsäure-äthylester 	97	111	1,5403 1,5299	136 158	Hellgelb. Geruch. Ab 85° Stäbchen. Blättchen. Gleichgew.: Körner, Prismen, Rauten. Erstarrt sphärolithisch oder palmwedelartig.
140	o-Chlorbenzoesäure 	73	90	1,5204 1,5101	146—148 166—169	Ab 90° breite Nadeln, Balken. Gleichgew.: Balken, Körner. Eutekt. mit Benzanilid 110°. 21
140	Furfuracrylsäure (= β-Furyl-(2)-acrylsäure) 	102	123	1,5403	155—157	Ab 80° Nadeln, Balken. Gleichgew.: Nadeln, Balken. Erstarrt sphärolithisch. Brechungsindex durch rasches Verdampfen erschwert (leichter in Silikonkammer).
140	Salicylamid 	102	117	1,5502 1,5403	151—152 172—173	Ab 120° Nadeln. Gleichgew.: Balken, Nadeln. Erstarrt sphärolithisch. Polymorph II 136°, III 133°, IV 131° (S). 21
140	3,5-Dinitrobenzoesäure-[2-methoxyphenylester] 	112	118	1,5403	161—162	Ab 115° feine Nadeln. Gleichgew.: Prismen, Balken.
140	5-Cyclopentenyl-5-äthylbarbitursäure 	108	91	1,5204 1,5101	122—123 151—152	Ab 105° Nadeln, verfilzte Nadelbüschel. Gleichgew.: Nadeln, Balken. Polymorph. 29
141	Salochinin (= Salicylsäure-chininester)  C ₂₇ H ₂₃ O ₄ N	121	128	1,5700 1,5611	140—141 167—169	Gleichgew.: Nadeln. Erstarrt glasig. 27
141	Korksäure (= Suberinsäure) HOOC-[CH ₂] ₈ -COOH	109	128	<1,4339		Ab 110° Nadeln, Blättchen, Körner. Gleichgew.: gelpappte Blättchen. Polymorph II 138°, III unbeständig, enantiotrop (26). Aus Schmelze I, Umwandlung in II oder Zerfall in Lamellen III
141	Terephthalsäure-dimethylester 	117	126	1,4683	148—151	Ab 65° lange Nadeln, Blättchen, Körner. Gleichgew.: Stengel, Platten. Erstarrt zu langen Stengeln, Spießen und Platten. Brechungsindex durch Flüchtigkeit erschwert.

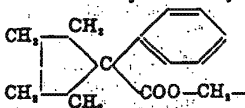
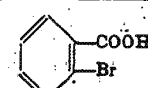
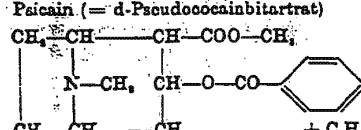
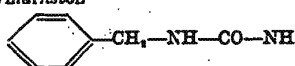
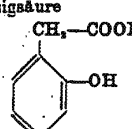
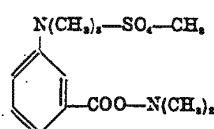
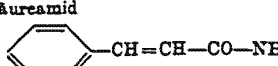
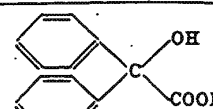
F. °C	Substanz	Eutekt. Temp. mit Phenacetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
≡ 141	4,4'-Dimethoxy-3,3'-dimethyldiphenyl-sulfon 	112	119	1,5611 1,5502	121—122 145—146	Gleichgew.: Körner, verschiedenartige Kristalle. Erstarrt glasig, bei Erwärmen palmwedelartig.
≡ 141 (128)	Phenazon-N-oxyd (= 2,2'-Azoxy-diphenyl) 	99	107 (130)	1,6877	170—172	Ab 100° Nadeln, Stengel. Teilweises Schmelzen 128°, teilweise Umwandlung. Gleichgew.: Prismen, Nadeln, zugespitzte Stengel. Erstarrt sphärolithisch, manchmal auch Sphärolithe II, bei Erwärmen Umwandlung. Aus Alkohol Nadeln und Stengel von II.
≡ 141	β-Benzaminoäthanolbromhydrat 	107	130	1,6611	129—130	Gleichgew.: Prismen, Platten. Dimorph.
≡ 142	Benzoylaminoacetonitril 	107	121	1,5204 1,5101	139—140 163—164	Ab 120° Blättchen, Nadeln. Gleichgew.: Balken, Blättchen.
≡ 142	Numal (= 5-Allyl-5-isopropylbarbitursäure) 	108	123	1,4842	134—136	Ab 80° Körner, später Nadeln. Gleichgew.: lange Nadeln, Spieße, Prismen. Polymorph II 139°, III 133°, IV 130° (63).
≡ 142	p-Phenylendiamin 	109	123	1,5795	154—156	Ab 80° kurze Prismen, sechseckige Blättchen, Platten. Gleichgew.: Körner. Schmelze rot bis dunkelbraun.
≡ 142	Trichloracetamid CCl ₃ -CO-NH ₂	96	113	1,4683	135—138	Würziger Geruch. Ab 45° vierseitige Blättchen. Sehr flüchtig. Vollständige Umlagerung. Gleichgew.: vierseitige Platten. Erstarrt strahlig.
≡ 142	Pentaerythritetranitrat C(CH ₂ -O-NO ₂) ₄	124	133 tr.	1,4584	115	Ab 115° Nadeln, Körner. Gleichgew.: Stengel. Erstarrt sphärolithisch.
142,5	m-Nitrobenzoesäure 	83	108	1,5403 1,5299	138—139 157—158	Ab 100° gebogene Fäden, rechteckige Blättchen (nicht genau 90°), Spieße. Blättchen schmelzen bei 135°. Polymorph II 135°, III 135°, IV 134° (33).
≡ 142,5	Dihydrocholesterin (trans, trans, trans, trans) 	121	129	1,4842 1,4683	125—126 167—168	Ab 90° Stäbchen. Gleichgew.: Stengel.

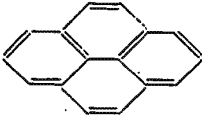
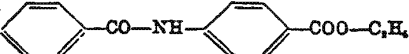
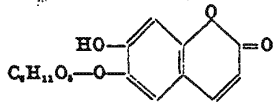
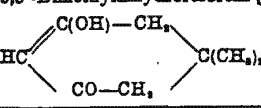
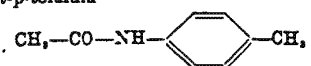
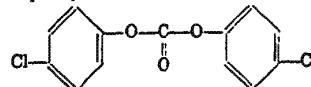
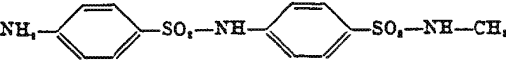
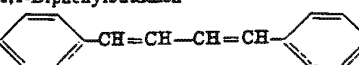
F. °C	Substanz	Eutekt. Temp. mit Phenacetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
143	Orthoform Neu (= m-Amino-p-oxybenzoesäuremethylester) 	105 tr.	120	1,5611 1,5502	146—149 169—171 21	Ab 100° Körner. Gleichgew.: Körner. Schmelze bald braun, erstarrt glasig. Polymorph. Orthoform + H ₂ O F. 52° bis 60°.
135—143	Oxyharnstoff $\text{H}_2\text{N}-\text{CO}-\text{NH}-\text{OH}$	106	127	Zersetzung		Ab 105° Körner, Blättchen, Rauten. Schmilzt unter starker Gasblasenbildg.
141—143	Adrianol (= 1-[m-Oxyphenyl]-2-methyl-aminoäthanol) 	110	135	1,5700 1,5511 1,5511	88—90 130—132 42	Gleichgew.: Prismen, Tafeln. Erstarrt glaig.
143	m-Benzamino-benzonitril 	104	121	1,5897 1,5795	139 159—160 21	Schmelze erstarrt teils sphärolithisch, teils glasig.
141—143	Anthraconpikrat 	101	114	—	—	Bräunlichrot. Ab 110° Nadeln, Blättchen. Schmilzt 141—143° inhomogen unter Abscheidung von Rauten und Sechsecken (Anthracen). Gleichgew.: Stengel. Bei 130° sublimieren Rauten und pleochroitische Stengel (bläulich-schwarz und rot).
143	4-Nitro-2-aminophenol 	86	111	1,6353	140—143	Gelbbraun. Ab 125° Nadeln, Spieße. Schmelze orange. Brechungsindex durch teilweise Zersetzung erschwert.
144	Eutolan (= p-Oxydiphenylmethan-carbaminsäureester) 	91	117	1,5403— 1,5502		Ab 110° rechtwinkelige Blättchen, Stäbchen. Gleichgew.: Rechtecke, sinkt ab. Zersetzung unter Abscheidung hochschmelzender Stengel.
144	Methylpropylketon-2,4-dinitrophenyl-hydrazon 	116	121	1,6011	161	Orange. Ab 90° Körner, Nadeln, Stäbchen. Gleichgew.: Körner, Balken.
145	o-Aminobenzoessäure (= Anthranilsäure) 	98	122	1,5700 1,5611	145—147 165—168 21	Ab 90° Rauten, Prismen, Spieße. Gleichgew.: lange Nadeln und Spieße.
145	Phenol-a-d-galaktosid + H ₂ O 	117	132	1,5204 1,5101	164—166 194—196 22	Ab 40° entweicht Wasser unt. Trübung. Ab 120° Nadeln und Nadelbüschel. Gleichgew.: Nadeln. Erstarrt glasig, bei Erwärmen kristallin.
146	5-Äthyl-5-propylbarbitursäure 	108	124	1,4683 1,4534	140—143 164—165	Ab 95° Nadeln, Blättchen, Körnchen. Gleichgew.: Stäbchen, Körner. Polymorph II 132°, III 112° (63).

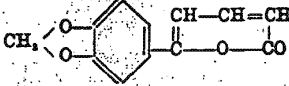
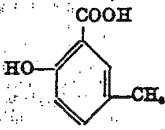
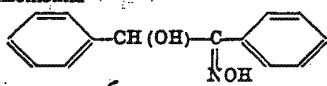
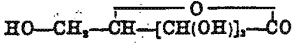
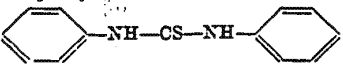
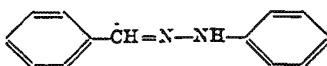
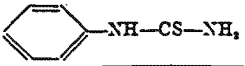
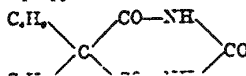
F. °C	Substanz	Eutekt. Temp. mit Phen-acetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
≠ 146	Papaverin 	116	122	1,5700 1,5611	156—167 177—178 f/	Ab 130° Tröpfchen, wenig kleine Blättchen. Gleichgew.: Prismen, Blättchen. Erstarrt glasig.
≠ 146	Evipan (= 3-Methyl-5-cyclohexenyl-5-methyl-barbitursäure) 	114	123	1,5000 1,4936	138—140 156—158	Ab 110° kurze Nadeln. Gleichgew.: Stengel.
141—147	Diphenylthiocarbazon (= Dithizon) 	120	132	—	—	Schwarz. Schmilzt trög. Schmelze teils braungrün, teils schwarz. Ätherlösung grün.
143—147	d-Xylose 	130	140	1,5000 1,4936	127 150—152	Ab 130° Tröpfchen. Erstarrt glasig.
144—147	Hämatoxylin  + 3 H ₂ O	102	126	Zersetzung	—	125—135° entweicht Wasser unt. Trübung. Schmelze gelbrosa. Gasblasen. In Paraffin Dampfblasen ab 120°; ab 140° Schmelzen und Zersetzung unter Abscheidung hochschmelzender Kristalle. Aus Wasser (Lösung bald rosa) viel. Körner und rechteckige Prismen mit α' zw. 1,62 und 1,66 γ' zw. 1,66 und 1,74.
144—147	S-Benzyl-thiuroniumformiat 	120	135	—	—	Bei Erwärmen schwefeliger Geruch. Ab 110° stellenweise Blättchen. Schmilzt unter Zersetzung. Aus Benzol + Alkohol gestreckte Sechsecke mit schiefer Auslösung. α' zwischen 1,59 und 1,62, γ' > 1,66.
≠ 147	Sarkosinanhydrid 	100	106	1,4683 1,4594	156 181	Ab 80° schöne Körner, starke Sublimation. Gleichgew.: Körner. Erstarrt sphärolithisch.
≠ 147	ω,ω'-Dibrom-p-xytol 	119	128	1,5611 1,5502	150—152 173—175 22	Scharfer Geruch, tränenenerregend. Ab 70° Körner, Blättchen. Gleichgew.: Balken. Erstarrt sphärolithisch.
147 (139)	Pantocain (= 2-Dimethylaminoäthyl-p-butylaminobenzoester-hydrochlorid) 	105	116 tr.	1,5502 1,5403	133—135 161—163 24	Gleichgew.: Platten, Blättchen. Enantiotrop polymorph II 139°, III 130° (74). Manche Präparate enthalten II, dann Schmelzbeginn 139°. Erstarrt glasig.

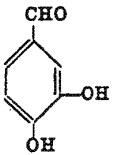
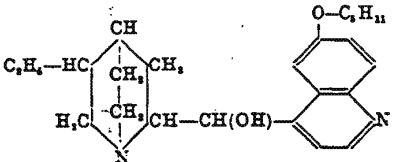
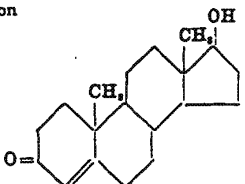
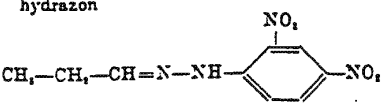
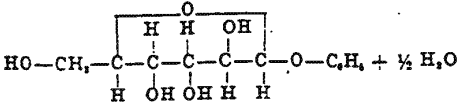
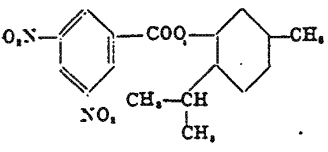
F. °C	Substanz	Eutekt. Phen-acetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
147	Indazol (= Benzopyrazol) 	101	118	1,5511 1,5502	156—158 180—181 24	Ab 100° Nadeln, Stäbchen. Gleichgew.: Balken. Erstarrt sphärolitisch.
142—147	Diäcetylphenolphthalein 	110	116	1,5502 1,5403	142 169—170 25	Gleichgew. sehr trüg; sechs- oder acht-seitige, schwach anisotrope Prismen; ferner Leisten und Prismen mit höheren Interferenzfarben. Erstarrt glasig.
147	p-Methylmandelsäure 	96	124	1,5000 1,4936	146—148 166—168	Ab 100° Blättchen, Nadeln, Prismen. Erstarrt glasig oder sphärolitisch.
147	2,2'-Dimethoxy-3,3'-dimethyldiphenyl-sulfon 	113	122	1,5403 1,5299	134—137 157—159 27	Ab 100° Tröpfchen. Gleichgew.: Prismen, Blättchen. Erstarrt sphärolitisch.
147	N-Phenyl-N-äthyl-N'-acetylarnstoff 	117	131	1,5000 1,4937	157—159 170—172	Ab 100° verfilzte Nadeln. Gleichgew.: Körner, kleine Prismen, sinkt sehr rasch ab. Erstarrt auf warmem Heistisch zu Sphärolithen. Polymorph.
147	o-Nitrobenzoesäure 	83	110	1,5101	174—176	Ab 70° Körner, Blättchen, Prismen. Gleichgew.: Platten, Stengel. In ursprünglichen Platten und Sublimaten ab 120° Umwandlung. Enantiotrop polymorph (73).
145—143	Diplosal (= Salicylsalicylsäure) 	102	124	1,5611 1,5502	139 176 27	Ab 80° Körner, Nadeln. Erstarrt glasig.
145—148	Ammoniumsalicylat 	85	109	1,5502	120—125	Ab 50° Blättchen, Prismen, Stengel. Gleichgew.: Stengel. In Schmelze Gasblasen. Erstarrt sphärolitisch. Polymorph. Bei 135° aufgelegt (großes Deckglas!), sublimieren Nadeln, die von Rhomboiden umgewandelt werden. mit F. 145°. Stengel 145°.
148	Phenylarnstoff 	105	124	1,5611 1,5502	131—132 161—162 20	Ab 110° Körner, vierseitige Blättchen. Gleichgew.: Körner, Prismen. Erstarrt sphärolitisch.

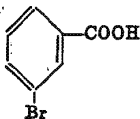
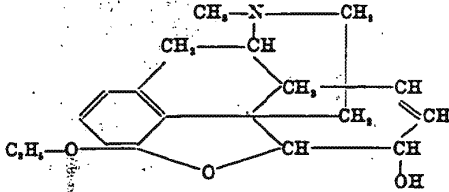
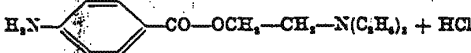
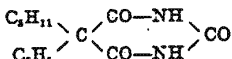
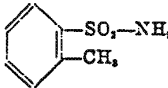
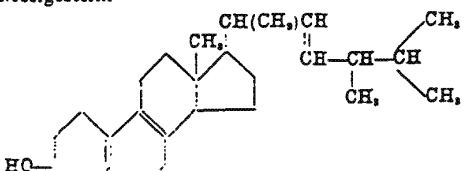
F. °C	Substanz	Eutekt. Temp. mit Phenacetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
145—148	Chininhydrochlorid + 2 H ₂ O $C_{20}H_{24}O_4N_2 + HCl + 2 H_2O$	96	111	1,5785	174—176	Zwischen 90—100° entweicht Wasser. Erweicht langsam. Schmelze sehr zäh. In Paraffin schmilzt Hydrat bei 104° homogen. Mit Phenacetin $n = 1,5502$ bei 106—103°, 1,5403 bei 137—140°.
146—148	p-Aminosalicylsäure 	120	130	Zersetzung		Beim Erwärmen Trübung. Ab 125° Tröpfchen, ab 140° Gasblasen in Kristallen (bei älteren Präparaten ab 130° und F. 130—135°). Schmilzt unter heftiger Gasblasenbildung. Restschmelze erstarrt strahlig mit F. 118—121° (m-Aminophenol siehe dort).
148	Hexachlorinden-(1)-on-(3) (= Perchlorindon) 	117	127	1,6353 1,6231	145—148 172—174	Gelb. Ab 120° Nadeln, einzelne Prismen. Gleichgew.: Sechsecke, Prismen, sinkt ab. Dimorph. Erstarrt zu II (kleinkristallin), bei Erwärmen F. II 123° oder Umwandlung.
148	Cholesterin 	122	133	1,4842	145—147	Ab 130° feine Tröpfchen. Gleichgew.: Rechtecke, Spieße. Schmelze erstarrt strahlig, häufig Faserdrilling, niedere Interferenzfarben.
148	p-Nitroanilin 	105	120	1,6598 1,6483	163—155 178—180	Gelb. Ab 120° Körner, vierseitige Blättchen. Gleichgew.: Balken und Spieße.
148	Proponal (= 5,5-Dipropylbarbitursäure) 	109	123	1,4584	163—155	Ab 110° Nadeln, Nadelbüschel, Balken. Gleichgew.: Balken. Dimorph II 146° (63).
148	Äthylpropylketon-2,4-dinitrophenylhydrazon 	118	124	1,5397 1,5795	167—169 188—189	Orange. Ab 75° Körner, später Blättchen. Gleichgew.: Balken. Erstarrt sphärolithisch.
148	N,N'-Diphenylguanidin 	108	123	1,6011 1,5897	146—148 170—173	Carbolgeruch. Ab 100° Nadeln, Körner. Gleichgew. Nadeln, Prismen. Erstarrt glasig, bei Erwärmen Sphärolithe.
145—148	Traubenzucker (= Dextrose = d-Glucose) $HO-CH_2-\overset{H}{\underset{ }{C}}-\overset{H}{\underset{ }{C}}-\overset{OH}{\underset{ }{C}}-\overset{H}{\underset{ }{C}}-CHO + H_2O$	133	145	1,5101 1,5000	142—145 178—179	Ab 70° allmähliches Entweichen von Wasser. Kristalle lagern sich zu körnigen oder stengeligen Aggregaten um. Schmelze zäh. In Paraffin sehr träge, inhomogenes Schmelzen 75—80° unter

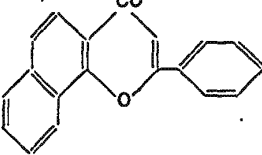
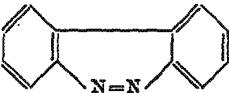
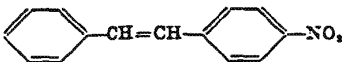
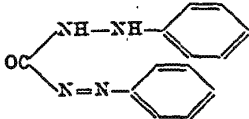
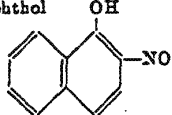
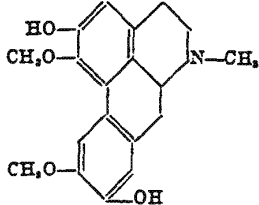
F. °C	Substanz	Eutekt. Phen-acetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
148,5	Parpanit (= 1-Phenylcyclopentancarbonsäure-2-diäthylaminoäthylesterhydrochlorid)  + HCl	97	105	1,4936 1,4842	176—177 ($\frac{1}{2}$)	Ab 100° Nadeln, Prismen. Gleichgew.: Platten, Spieße. Enantiotrop dimorph II 145° (8).
149	o-Brombenzoesäure 	90	115	1,5403 1,5299	156—157 177—179 $\frac{2}{1}$	Ab 100° Nadeln. Gleichgew.: Nadeln, Balken. Erstarrt sphärolithisch.
147—149	Hyoscyaminhydrobromid (= 1-Tropasäure-tropinester-hydrobromid) $C_{17}H_{21}O_4N + HBr$	106	122	1,5403	159—160	Schmilzt langsam. Gleichgew. sehr träg: Körner, Prismen.
149	p-Nitrobenzonitril 	110	123	1,5204 1,5101	151—153 173—175 $\frac{2}{2}$	Ab 80° Blättchen, Nadeln. Gleichgew.: Körner, Prismen.
149	Paicain (= d-Pseudococainbitartrat)  + $C_6H_8O_4$	121	134	1,5101 1,5000	142 172—174 $\frac{5}{1}$	Ab 100° Körner, Blättchen. Erstarrt glasig. In Paraffin homogenes Schmelzen bei 120—125°.
149	Benzylharnstoff 	109*	124	1,5299 1,5204	137—139 172—174 $\frac{3}{5}$	Ab 105° Nadeln. Gleichgew.: Balken. Erstarrt grobstrahlig sphärolithisch.
149	o-Oxyphenyllessigsäure 	94	—122—	1,5101— 1,5000		Ab 120° Körner, Prismen, Stengel. Bei Erwärmen stechender Geruch. Gleichgew.: Prismen. Erstarrt spontan zu II 82°, durch Impfen Umwandlung. Bei Schmelze Gasblasen.
149	Prostigmin 	103	115	1,4936 1,4842	139—141 177—179	Ab 110° Körner. Gleichgew. träg: Körner, Prismen. Erstarrt glasig. Durch Impfen bei Raumtemperatur nur strangförmige Kristalle am Rande des Tropfens; bei Erwärmen kristallisiert ab 40° ein neuer Ring. Ab 70° wächst fasergebrilltes Aggregat nach innen.
149	Zimtsäureamid 	110	123	1,5700 1,5611	164—166 186—188 $\frac{2}{2}$	Ab 90° Körner, Prismen, Balken. Gleichgew.: Balken. Erstarrt sphärolithisch. Polymorph.
142—150	Chininhydrobromid + H_2O $C_{20}H_{24}O_4N_2 + HBr + H_2O$	96	117 tr.	1,6011 1,5897	137—140 177—180 $\frac{4}{0}$	Erstarrt glasig. In Paraffin bei 75° inhomogenes Schmelzen unter Abscheidung wasserfreier Nadeln, zum Teil homogenes Schmelzen ab 110° unter mäßiger Dampfblasenbildung.
145—150	Benzilsäure 	94	115	1,5502— 1,5611		Geruch. Ab 100° Stäbchen. Gleichgew.: Stengel. Erstarrt glasig. Bei Erwärmen Rosetten. Brechungsindex unmittelbar nach Schmelzen 1,5502, steigt rasch, besonders am Rande des Tropfens.

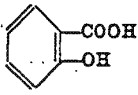
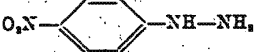
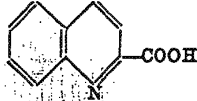
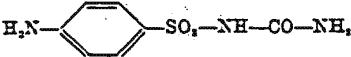
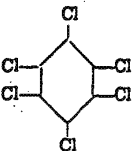
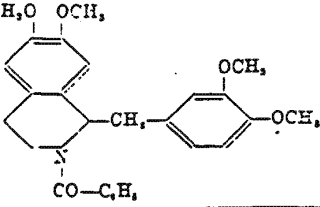
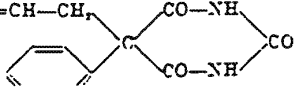
F. °C	Substanz	Eutekt. Temp. mit Phenacetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
150	Pyren 	113	129	1,6877	175—178	Geruch. Gelb. Ab 90° Rauten, Körner, kurze Prismen. Gleichgew.: Körner, Rauten, Rechtecke, Prismen. Erstarrt sphärolithisch, bei Erwärmen palmwedelartig. Dimorph (75).
160	4-Oxypyridin  $N + H_2O$	101 (+ H_2O : 55)	118 (+ H_2O : 55)	1,5795 1,5700	137—140 164—167	Schmelzen des Hydrates 62—65°, kristallisiert wieder aus. Ab 110° Tröpfchen. Gleichgew.: Körner. Erstarrt sphärolithisch. 27
150	4-Benzaminobenzoesäure-äthylester 	116	—130—	1,5611 1,5502	156—158 176—177	Gleichgew.: Nadeln. Erstarrt sphärolithisch. Dimorph. 26
140—150	Colchicin $C_{21}H_{25}O_6N$	95	105	Zersetzung		60—90° leichte Trübung der Kristalle. Schmelzest. Mit Phenacetin $n = 1,5611$ bei 121—123°. Leicht löslich in Alkohol, Aceton und Essigester. Aus letzterem flache Stengel (träge Kristallisation) mit exzentrischem Austritt einer Achse, A.-E. quer.
148—150	Äsculin (= Äsculetin-6-glucosid)  $C_{21}H_{21}O_{11}$	123	140	1,6011 1,5897	148—150 165—167	Erweicht ab 148°. Erstarrt glasig. Wässrige Lösung fluoresziert blau. 37
148—150	H_2 -Trastinhydrochlorid $C_{21}H_{21}O_6N + HCl$	99	111	Zersetzung		Erweicht langsam zu zähflüssiger Schmelze. Mit Phenacetin $n = 1,5403$ bei 128—131°.
146—150	5,5'-Dimethyldihydroresorcin (= Dimedon) 	109	128	1,4842	125—130	Ab 90° Blättchen, Rechtecke, Stäbchen. Gleichgew.: Rechtecke, Prismen; sinkt rasch ab. Schmelze bald gelb. Erstarrt sphärolithisch, bei Erwärmen Umlagerung zu körniger Struktur. Brechungsindex steigt an.
151	Acet-p-toluidid 	109	121	1,5101 1,5000	143—144 167—168	Ab 90° Körner, Prismen, Blättchen. Gleichgew.: Balken. Erstarrt sphärolithisch. Dimorph II 145° (32). 34
151	p-Chlorphenylcarbonat 	119	130	1,5101 1,5000	168—169 191—192	Ab 105° Nadeln, Spieße. Gleichgew.: Stengel. 33
151	Neo-Uliron (= 4-Sulfanilylamino-benzol-sulfonsäure-(1-methylamid) 	109	128	1,6011 1,5898	150—151 187—188	Schmilzt teilweise bei 146°, wandelt sich teilweise in rechtwinkelige und schiefwinkelige Kristalle um. Erstarrt glasig. 37
152	Adipinsäure $HOOC-(CH_2)_4-COOH$	113	134	<1,4339		Ab 110° körnige, spitze Rauten, Stäbchen, Prismen. Reichliche Sublimat. Gleichgew.: Balken, Spieße.
152	1,4-Diphenylbutadien 	122	134	1,6353 1,6231	172—173 184—186	Ab 90° Körner, Prismen, Blättchen. Gleichgew.: Platten. 12

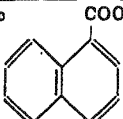
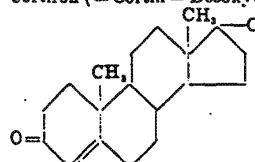
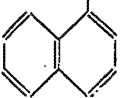
F. °C	Substanz	Eutekt. Temp. mit Phenacetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
152	Paracotoin (= 6-[3,4-Methylenedioxy-phenyl]-cumalin) 	115	121	1,6231 1,6126	158—158 179—181 3	Ab 105° Nadeln. Gleichgew.: Platten. Schmelze gelb. Polymorph II 147°, III unbeständig (30).
152	α -Bromisovalerylharnstoff (= Brom-ural) $(CH_3)_3CH-CHBr-CO-NH-CO-NH_2$	111	126	1,4683	165—167	Ab 110° Nadeln, Blättchen, Spieße und Büschel. Gleichgew.: verfilzte Nadeln und Spieße. Erstarrt zu verfilzten Kristallen. Polymorph II 148°, III 143° (4,70).
142—152	Harnstoffnitrat $H_2N-CO-NH_2 + HNO_3$	114	141	Zersetzung		Ab 100° Blättchen, Stäbchen. Schmilzt mit sehr starker Gasblasenbildung. In Wasser leicht löslich, daraus derbe Prismen, stahlgrau (gekrenzte Nicols) mit I. Bisektrix, kleiner Achsenwinkel negativ. Brechungsindex der grauen Kristalle wenig über 1,62.
152	p-Kresotinsäure (= 6-Oxy-m-tolylsäure = 6-Oxy-3-methylbenzoesäure) 	95	122	1,5101	162—164	Ab 70° Nadeln, Stengel. Ab 130° starke Verflüchtigung. Gleichgew.: Stengel. Für Brechungsindex großes Deckglas! Erstarrt sphärolithisch.
145—153	Verätrin $C_{11}H_{10}O_2N$	101	111 tr.	1,5204 1,5101	126—128 161—163	Nicosenregend. Erweicht langsam zu zäher Schmelze. Erstarrt glasig.
151—153	α -Benzoinoxim 	113	133	1,5611	147—149	Ab 120° Körner, Stäbchen. Gleichgew.: Körner. Erstarrt glasig, bei Erwärmen sphärolithisch.
140—153	d-Gluconsäure- δ -lacton 	129	141	1,5000 1,4937	145 165—166	Schmilzt trög, erstarrt glasig.
153	N,N'-Diphenylthioharnstoff 	110	133	1,6126 1,6011	155 167	Ab 130° winzige Nadeln und Blättchen.
148—153	Benzylidenphenylhydrazin 	108	124 tr.	1,6231	155—160	Häufig rot verfärbt. Umsublimieren bei 140° (eventuell umkristallisieren mit Adsorbens). Ab 95° Nadeln, später Balken. Gleichgew.: Nadeln, Balken. Sinkt ab. Brechungsindex wegen Gasblasen erschwert.
148—154	Hyoscyaminhydrochlorid $C_{17}H_{21}O_2N + HCl$	105	116 tr.	1,5299	150—151	Gleichgew. trög: Körner, Blättchen. Erstarrt glasig. Schmilzt in Paraffin 63° inhomogen mit Rückstand feiner wasserfreier Kristalle.
154	Phenylthioharnstoff 	111	133	1,6353— 1,6231		Ab 100° wenig Nadeln, später Körner. Gleichgew.: Körner, Prismen, sinkt ab. Schmelze erstarrt glasig, bei Erwärmen sphärolithisch.
154	5-Ethyl-5-propylbarbitursäure 	112	129	1,4584	143—144	Ab 105° Nadeln, Stengel. Gleichgew.: Stengel, Balken. Erstarrt sphärolithisch.

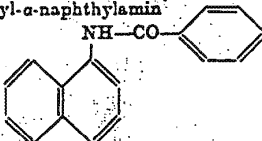
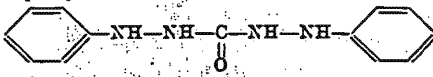
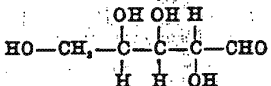
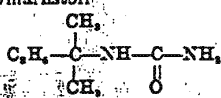
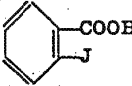
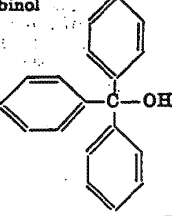
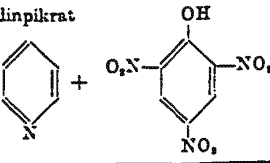
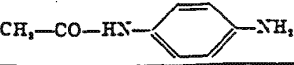
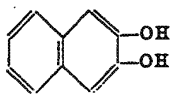
F. °C	Substanz	Eutekt. Temp. mit Phen-acetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
154	Dipropionamid (C ₃ H ₇ -CO) ₂ NH	116	129	1,4339		Geruch nach Buttersäure. Ab 80° Nadeln, später starke Verflüchtigung. Sublimate wie Watte. Gleichgew.: Nadeln, Balken. Erstarrt sphärolithisch.
154	3,4-Dioxybenzaldehyd (= Protocatechu-aldehyd) 	98	124	1,6011	148—152	Ab 110° Nadeln, flache Stengel, Rechtecke. Gleichgew.: Spieße, Balken. Schmelze bald bräunlich. Erstarrt sphärolithisch.
151—154	Citronensäure $\text{HO}-\text{C}(\text{CH}_3-\text{COOH})_2-\text{COOH} + \text{H}_2\text{O}$ $\text{C}_6\text{H}_8\text{O}_7$	108 (+H ₂ O: 58)	146 (+H ₂ O: 58)	1,4584	170—173	Zwischen 60—70° entweicht Wasser. Teilweises Schmelzen. In Paraffin teils inhomogenes Schmelzen ab 36°, teils homogenes Schmelzen ab 73°. Blasen erschweren Brechungsindex.
153—155	Eucupin (= Isoamyl-dihydrocuprein) 	122	132	1,5299 1,5204	141 170 29	Ab 130° feine Nadeln.
155	Testosteron 	107	117	1,5204 1,5101	155—156 185—186 20	Ab 130° wenig Körner, Stäbchen. Gleichgew.: Körner. Erstarrt glasig.
155	Propionaldehyd-2,4-Dinitrophenyl-hydrazon 	114	122	1,6353 1,6231	152 172 20	Ziegelrot. Ab 110° Körner, Nadeln. Gleichgew.: Körner. Erstarrt büschelförmig. Dimorph II 135°.
155	Phenol-β-d-galaktosid + ½ H ₂ O 	122	142	1,5204 1,5101	175—177 203—205 25	Ab 100° Trübung. Ab 115° Nadeln. Gleichgew.: Nadeln.
155	3,5-Dinitrobenzoesäure-1-menthylester 	122	134	1,4083	191—192	Ab 115° feine Nadeln. Gleichgew.: Stengel. Dimorph.

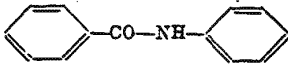
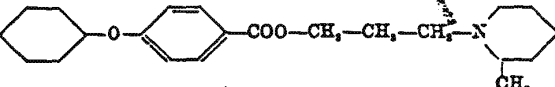
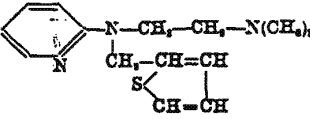
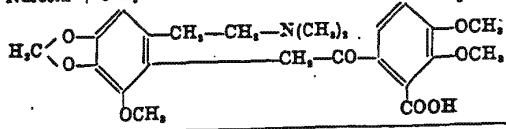
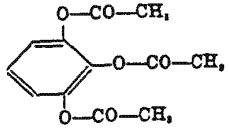
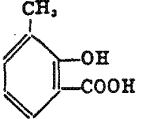
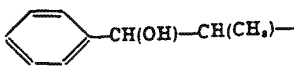
F. °C	Substanz	Eutekt. Phen-acetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
140—155	Camphan	—112—	—110—	—	—	Camphergeruch. Ab 30° isotrope Sterne, Spieße, später wabenartig. F. mit großem Deckglas oder Silikonkammer, oft schon ab 120° Flüssigkeit. Reinigen auch durch Umsublimieren schwierig. Isotrop, n bei Raumtemperatur zwischen 1,50 und 1,515.
155,5	m-Brombenzoesäure 	103	126	1,5299 1,5204	174—176 197—199 23	Ab 105° Nadeln. Gleichgew.: Spieße.
149—156	Dionin (= Äthylmorphinhydrochlorid)  + HCl + 2 H ₂ O	96	108 tr.	1,5795	141—144	Zwischen 110—120° entweicht Wasser. Zerfließt ab 145° langsam zu zäher Schmelze. In Paraffin homogenes Schmelzen bei 118—122°, dann Dampfblasen.
156	Novocainhydrochlorid (= Procainhydrochlorid = 4-Aminobenzoessäure-β-diäthylaminoäthylester) + HCl 	114	129	1,5611 1,5502	159—161 192—195 21	Ab 135° Tröpfchen. Gleichgew.: Prismen und große Kristallblöcke. Erstarrt glasig.
156	Kodein (= Morphin-monomethyläther) C ₁₈ H ₂₁ O ₃ N	117	126	1,5403 1,5299	175—177 194—196	Ab 70° Körner, später Nadeln. Prismen. Gleichgew.: Körner. Kodeinhydrat schmilzt inhomogen ab 55—60° (vgl. dort).
156	Amytal (= 5-Isoamyl-5-äthylbarbitursäure) 	112	128	1,4584 1,4939	144 217—220	Ab 100° Tröpfchen. Ab 145° träge Schmelzen. Gleichzeitig entstehen Nadeln, Spieße, Stengel mit Gleichgew. bei 156° (63).
156	o-Toluolsulfonsäureamid 	107	127	1,5204 1,5101	163—165 189—192 27	Ab 70° Körner. Gleichgew.: Platten. Erstarrt strahlig.
150—157	Eupaverin + H ₂ O	Vgl. Eupaverin wasserfrei bei 210—230°				In Paraffin homogenes Schmelzen zwischen 145—155° bei mäßiger Blasenbildung, teilweise wasserfreie Kristalle.
157	Neoergosterin 	122	135	1,5000 1,4937	169 188	Ab 130° Nadeln, Spieße. Gleichgew.: Stengel, Spieße. Erstarrt sphärolithisch oder feinkristallin. Dimorph.

F. °C	Substanz	Eutekt. Temp. mit Phenacetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
157	α -Naphthoflavin (= Chlortest=7,8-Benzoflavin) 	116	120	1,6598 1,6483	167—168 180—192	Rosa. Ab 130° Blättchen. Gleichgew.: Körner, Rauten. Erstarrt sphärolithisch oder verfilzt.
157	Phenazon 	112	121	1,6483	160—162	Gelb. Ab 90° Körner, Prismen, Rhomboide. Gleichgew.: Rhomboide, Prismen. Erstarrt strahlig.
157	4-Nitrostilben 	121	132	1,6483 1,6353	165—167 196—199	Gelb. Ab 100° Nadeln, Körner, Prismen. Gleichgew.: Prismen, Balken. Schmelze gelb. Erstarrt sphärolithisch.
155—158	Diphenylcarbazon 	117 tr.	132 tr.	Zersetzung		Ab 140° Tröpfchen. Schmelze bräunlich. Zersetzung und Gasblasenbildung, wobei vierseitige Platten und Körner ausfallen. Reagens auf Hg. Ursubstanz dünne Blättchen (teilweise sechseitig), α' zwischen 1,53 und 1,55; γ' zwischen 1,59 und 1,62.
150—158	β -Nitroso- α -naphthol 	111	129	Zersetzung		Gelb, ältere Präparate braungrün. Ab 130° gelbe Nadeln, Stengel. Schmilzt trög, mit Zersetzung und Braunfärbung. Bei 130° sublimieren moosbüschelartige Aggregate und Prismen mit α' (längs) etwa 1,55, γ' > 1,74.
150—158	Tartronsäure $\text{HOOC}-\text{CH}(\text{OH})-\text{CO}(\text{OH}) + \frac{1}{2} \text{H}_2\text{O}$ $\text{C}_3\text{H}_4\text{O}_5$	102	140tr.	—	—	Ab 130° Rechtecke, Rhomboide (wenn Substanz feucht, absaugen). Ab 140° Gasblasen aus Kristallen, schmilzt trög mit „Kochen“ in Kristallen. In sublimierten Rechtecken α' etwas größer 1,50, γ' etwa 1,57. Mit Anilin oder o-Toluidin neue stengelige Kristalle.
151—158	Boldin (= 2,6-Dioxy-3,5-dimethoxy- aporphin) 	116	130	Zersetzung		Braun. Schmelze braun. Erstarrt glasig. Mit Phenacetin $n = 1,5700$ bei 113° bis 115°, 1,5611 bei 124—126°. In Alkohol leicht löslich; glasiger Rückstand gibt mit Aceton Körner, Bisphenole, Rauten. In Rauten lange Diagonale γ' , α' zwischen 1,44 und 1,46; $\gamma' > 1,66$.
153—158	Chininbisulfat $\text{C}_{20}\text{H}_{24}\text{O}_4\text{N}_2 + \text{H}_2\text{SO}_4 + 7 \text{H}_2\text{O}$	108	151 tr.	1,5700	140—145	Ab 60° entweicht Wasser unt. Trübung. Einzelne Hydratkristalle schmelzen bei 90°. Erweicht langsam. Schmelze gelb und zäh. Mit Phenacetin $n = 1,5301$ bei 143—146°, $n = 1,5203$ bei 167—170°. In Paraffin homogenes Schmelzen bei 73°, dann wasserfreie Nadeln.

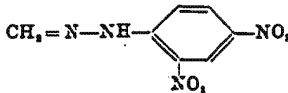
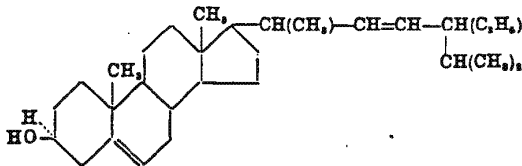
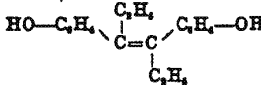
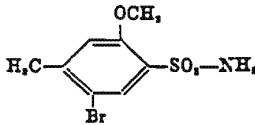
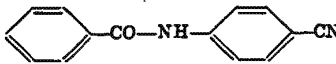
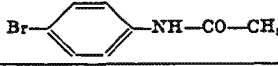
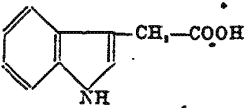
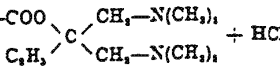
F.°C	Substanz	Eutekt. Temp. mit Phenacetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
158	Salicylsäure (= o-Oxybenzoesäure) 	91	121	1,5204 1,5101	155—157 175—177 20	Ab 65° Nadeln, Balken mit schiefen Enden, kurze Prismen. Stark flüchtig. Gleichgew.: Balken.
158	N-Allyl-N-butyl-N'-acetylarnstoff $\begin{array}{c} \text{C}_2\text{H}_5 \\ \text{C}_2\text{H}_5 \end{array} \text{N} - \text{CO} - \text{NH} - \text{CO} - \text{CH}_3$	120	137	<1,4339		Ab 120° feine Nadeln. Gleichgew.: Nadeln. Erstarrt sphärolithisch.
158	p-Nitrophenylhydrazin 	114	130	>1,8877		Orangerot. Ab 120° Dreiecke, ausgezackte Trapeze, Stengel. Gleichgew.: Balken, sinkt ab. Gasblasen. Schmelze orangegeb.
158	Chinaldinsäure (= Chinolin-2-carbonsäure) 	110	126	1,5397 1,5705	160—161 176—177 16	Ab 120° Körner, Stäbchen, gekrümmte Nadeln. Gleichgew.: Körner, Prismen. Erstarrt teilweise sphärolithisch.
156—168	Sulfanilylarnstoff (= Euvernil) 	123	150	1,6126— 1,6231		Ab 90° Kristalle dunkler, ab 150° langsames Erweichen. Reichlich Gasblasen. Erstarrt glasig (Markovic).
158	α-Hexachlorcyclohexan 	121	134	1,5101	151	Ab 80° Blättchen mit rhomboidischem Umriß, Prismen. Gleichgew.: Prismen.
159	Phenol-α-d-glucosid + H2O $\begin{array}{c} \text{H} \quad \text{H} \quad \text{OH} \quad \text{H} \\ \quad \quad \quad \\ \text{HO}-\text{CH}_2-\text{C}-\text{C}-\text{C}-\text{C}-\text{CH}-\text{O}-\text{C}_6\text{H}_5 \\ \quad \quad \quad \\ \text{OHH} \quad \text{OH} \quad \text{O} \end{array} + \text{H}_2\text{O}$	127	146	1,5204 1,5101	165—167 185—187 50	Ab 125° Nadeln. Gleichgew.: Nadeln. Dimorph.
159	N-Benzoyltetrahydropapaverin 	122	134 tr.	1,5611 1,5502	143—145 168—171 20	Ab 100° kurze Nadeln. Gleichgew. trög: Nadeln. Erstarrt glasig, bei Erwärmen sphärolithisch. Dimorph.
159	5-Allyl-5-phenylbarbitursäure 	108	130	1,5403 1,5299	158—160 181—183 20	Ab 140° Nadeln, Stengel. Gleichgew. sinkt ab: Prismen, Stengel. Erstarrt glasig, bei Erwärmen palmwedelartig.

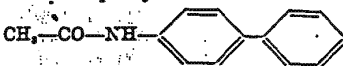
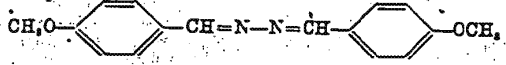
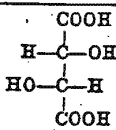
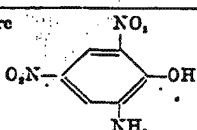
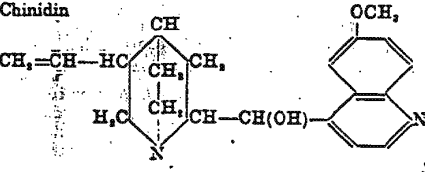
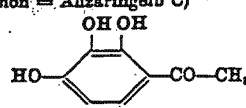
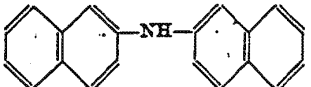
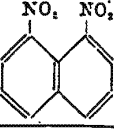
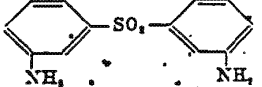
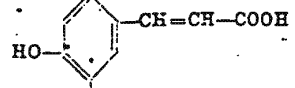
F. °C	Substanz	Eutekt. Phen-acetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
-150—160	Tricarballoylsäure $\begin{array}{c} \text{CH}_2\text{—COOH} \\ \\ \text{CH—COOH} \\ \\ \text{CH}_2\text{—COOH} \end{array}$	108	138 tr.	1,4339— 1,4584		Schmilzt trüg. Gleichgew. trüg: tonnenförmige Prismen.
Verfl. 150—160	Metalddehyd (C ₂ H ₂ O) _x	133 Harnstoff 132	156 Salicylsäure 154			Ab 105° Körner, Stengel, bei 150—160° vollkommene Verflüchtigung. Bei 120° sublimieren Stengel mit α' (längs) 1,50 bei 30—32°.
160	Dormin (= 5-Äthyl-5-allylbarbitursäure) $\begin{array}{c} \text{C}_2\text{H}_5 \quad \text{CO—NH} \\ \diagdown \quad \diagup \\ \text{C} \\ \diagup \quad \diagdown \\ \text{C}_2\text{H}_5 \quad \text{CO—NH} \end{array} \text{CO}$	115	134	1,4683	175—178	Ab 110° Körner, Rauten. Gleichgew.: Körner, sinkt ab. Erstarrt sphärolithisch. Dimorph II 129° (63).
160	β-Propionylphenylhydrazin $\text{C}_2\text{H}_5\text{—CO—NH—NH—}$ 	115	130	1,5101 1,5000	157—159 179—180	Ab 100° Körnchen, Blättchen. Gleichgew.: Stengel, Blättchen. 22
160	N,N'-Dinitrosopiperazin $\begin{array}{c} \text{ON—N} \quad \text{CH}_2\text{—CH}_2 \\ \diagdown \quad \diagup \\ \text{CH}_2\text{—CH}_2 \quad \text{N—NO} \end{array}$	117	127	1,5000 1,4936	159—160 178—177	Hellgelb. Ab 70° Körner, Blättchen. Starke Sublimation. Umlagerung zu Straßenpflaster. Gleichgew.: Platten.
160	4-Jodantipyrin (= Jodopyrin) $\begin{array}{c} \text{H}_2\text{C—C=CH} \\ \quad \quad \\ \text{H}_2\text{C—N} \quad \text{N—CO} \\ \quad \quad \\ \text{C}_6\text{H}_5 \end{array}$	107	115	1,6011 1,5997	160—161 174—176	Ab 150° Tröpfchen. Gleichgew.: Balken, Prismen. 24
156—181	Hydroxylaminhydrochlorid NH ₂ OH + HCl	128	146	Zersetzung		Ab 100° Körner, Blättchen. Zersetzung und Gasblasen. In Wasser leicht löslich. Aus Alk. Stengel und gestreckte Sechsecke mit gerader Auslöschung. γ' halbiert Winkel 103°; α' etwa 1,62, γ' zwischen 1,62 und 1,66.
161	α-Naphthoesäure 	105	120	1,5795	179—181	Ab 120° Körner, Balken. Gleichgew.: Prismen. Wenn F. unscharf, absaugen.
161	dl-Brombernsteinsäure HOOC—CH ₂ —CHBr—COOH	88	122	Zersetzung		Ab 130° Nadeln, Stäbchen. Bei und nach dem Schmelzen entstehen in Tropfen und als Sublimate reichlich Nadeln, Spieße und Stengel, die bis 230° nicht schmelzen, meist über 200° Verflüchtigung.
161	Cortiron (= Cortin = Desoxycorticosteronacetat)  $\text{CH}_3\text{—CO—CH}_2\text{—O—CO—CH}_3$	111	115	1,5000	166—187	Ab 140° wenig Nadeln. Gleichgew. trüg: Nadeln, Stengel. Erstarrt glasig, bei Erwärmen sphärolithisch.
161	α-Acetonaphthalid (= Acetyl-α-naphthylamin)  NH—CO—CH_3	113	123	1,5795 1,5700	166—167 189—191	Ab 80° Nadeln. Gleichgew.: Nadeln. Erstarrt muschelg. 25

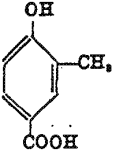
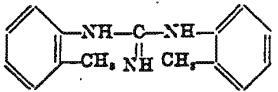
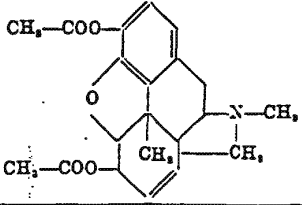
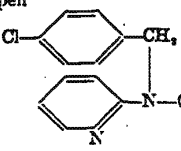
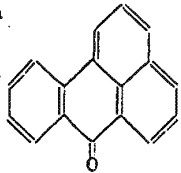
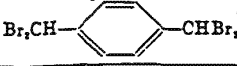
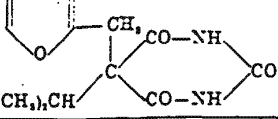
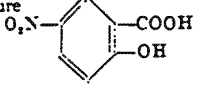
F. °C	Substanz	Eutekt. Temp. mit Phenacetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
161	Benzoyl- α -naphthylamin 	174	129 tr.	1,6126 1,6011	106—198 223—224	Ab 130° Nadeln. Gleichgew.: Nadeln. Erstarrt feinstrahlig sphärolithisch. 27
159—162	Diphenylcarbazid 	117	134	1,5795 1,5700	154—166 177—179	Ab 145° Tröpfchen. Erstarrt glasig, bei 130° aufgelegt, sehr träge Kristallisation, abgeschrägte Platten und Stengel. 23
157—162	l-Arabinose 	134	158	1,4936	163—164	Ab 160° Tröpfchen. Erstarrt glasig.
162	tert. Amylharnstoff 	114	132	<1,4339		Ab 100° Stäbchen, Spieße, Prismen. Gleichgew.: Balken, Prismen.
162	o-Jodbenzoesäure 	99	125	1,5795 1,5700	169—170 189—190	Ab 110° Körner, Prismen. Gleichgew.: Prismen. Erstarrt sphärolithisch. 26
162,5	Triphenylcarbinol 	117	133	1,5611 1,5502	170—172 193—195	Ab 120° Körner. Gleichgew.: Prismen. 23
163	Arecolinhydrochlorid $\begin{array}{c} \text{H}_2\text{C}-\text{CH}=\text{C}-\text{CO}_2-\text{CH}_3 \\ \text{H}_2\text{C}-\text{N}(\text{CH}_3)-\text{CH}_3 \end{array} + \text{HCl}$	100	116	1,5000 1,4936	147—148 182—183	Ab 125° Prismen, Vierecke. Hydrat schmilzt ab 55°, kristallisiert wieder. Gleichgew.: Prismen. In Paraffin inhomogenes Schmelzen bei 58° unter Abscheidung wasserfreier Stengel.
163	Pyridinpikrat 	117	—128—	Zersetzung		Gelb. Ab 115° Körnchen. Gleichgew.: kleine Prismen, sinkt ab. Schmelze gelb. Aus Alkohol lange Stengel, Rauten, Körner. Die Stengel löschen schief aus (80). α' (längs) 1,53—1,55, $\gamma' > 1,74$.
163	p-Aminoacetanilid 	119	134	1,5700 1,5611	153 174—175	Meist hellrosa verfärbt. Ab 130° Körner. Gleichgew.: Prismen, Körner. Erstarrt glasig, bei Erwärmen feinstrahlig sphärolithisch. Dimorph II 141° (32). 23
163	2,3-Dioxynaphthalin 	75	109	1,6011	185—190	Ab 95° Blättchen, Nadeln. Gleichgew.: Blättchen, Körner. Brechungsindex rasch bestimmen, bald Zersetzung.

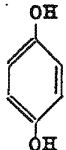
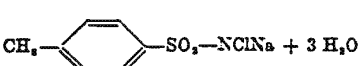
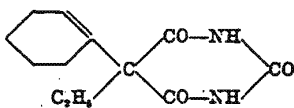
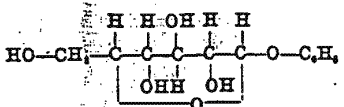
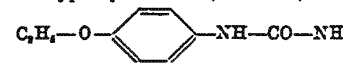
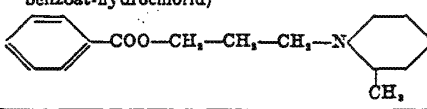
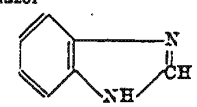
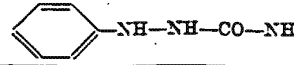
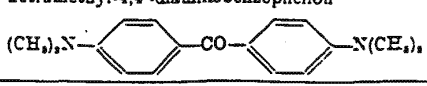
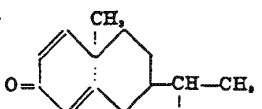
F. °C	Substanz	Eutekt. Temp. mit Phenacetin	Temp. mit Benz. anilid	Glaspulver	Temp. °C	Besondere Kennzeichen
163	Benzanilid 	114	163	1,5700 1,5611	168—169 190—192	Ab 100° Nadeln, Blättchen. Gleichgew.: verschiedenartige Kristalle. Erstarrt sphärolithisch.
163,5	Allypinnitrat $\begin{array}{c} \text{C}_6\text{H}_5-\text{COO} \\ \text{C}_6\text{H}_5 \end{array} \text{C} \begin{array}{l} \text{CH}_2-\text{N}(\text{CH}_3)_2 \\ \text{CH}_2-\text{N}(\text{CH}_3)_2 \end{array} + \text{HNO}_3$	103	115	1,4936— 1,4842		Ab 140° Nadeln. Gleichgew.: Körner, Platten. Schmelze bald braun.
155—164	Coffeincitrat $\text{C}_8\text{H}_{10}\text{O}_7\text{N}_4 + \text{C}_6\text{H}_8\text{O}_6$	118	136 tr.	1,5204 1,5101	114—116 146—148	Ab 120° Nadeln, Spieße, Körner. Erstarrt glasig.
155—164	5-sec. Amyl-5-[β-bromallyl]-barbitursäure $\begin{array}{c} \text{C}_5\text{H}_7-\text{CH}(\text{CH}_3) \\ \text{C}_5\text{H}_7\text{Br} \end{array} \text{C} \begin{array}{l} \text{CO-NH} \\ \text{CO-NH} \end{array} \text{CO}$	115	132	1,5000 1,4936	150 167	Ab 130° Tröpfchen. Schmelze trög, erstarrt glasig.
164	Surfacain 	115	133	1,5000 1,4936	167 193—195	Gleichgew.: klein kristallin. Erstarrt glasig.
164	Histadyl (= Thenylpyraminhydrochlorid)  + HCl	105	117	1,5611	171—173	Ab 130° Körner, Blättchen. Gleichgew.: Prismen.
155—165	Narcein + 3 H ₂ O 	112	126	1,5502— 1,5611		Bei 70—90° entweicht Wasser unt. Trübung. Schmelze gelb. Bei stärkerem Erwärmen entstehen in der Schmelze Stäbchen und Körner. Mit Phenacetin n = 1,5403 bei 128—130°, 1,5301 bei 148—149°.
154—165	Gelsemin $\text{C}_{22}\text{H}_{22}\text{O}_8\text{N}_2$	113	125	Zersetzung		Ab 120° Nadeln. Langsames Entweichen von Blasen. Später Zersetzung. Mit Phenacetin n = 1,5794 bei 118—122°, 1,5700 bei 143—146°.
165	Arbutin instabil.	160	135	Vgl. bei 200°		
165	Pyrogalloltriacetat (= 1,2,3-Triacetoxybenzol) 	124	137	1,4339	179—181	Ab 120° Körner. Gleichgew.: Straßenpflaster. Erstarrt sphärolithisch.
165	o-Kresotinsäure (= 2-Oxy-m-toluyisäure) 	98	127	1,5101— 1,5000		Geruch. Ab 80° Nadeln, büschelig angeordnet. Sublimiert stark. Gleichgew.: Stengel. Erstarrt sphärolithisch. Mit Nikotinsäureamid zwei Molekülverbindungen. F ₁ 124°, F ₂ 140° (Kontaktpräparat).
165	rac. Pseudoephedrinhydrochlorid  + HCl	116	136	1,5204	175—178	Ab 130° Nadeln. Gleichgew.: Balken. Stengel. Dimorph.

F.° C.	Substanz	Eutekt. Temp. mit Phenacetin	Temp. mit Benz. anilid	Glaspulver	Temp. °C	Besondere Kennzeichen
165	1,2-Benzanthracen 	118	129	1,6877	184—186	Ab 95° Stäbchen, Körner. Gleichgew.: Platten.
165	Acetaldehyd-2,4-dinitrophenylhydrazon $\text{CH}_3-\text{CH}=\text{N}-\text{NH}-\text{C}_6\text{H}_3(\text{NO}_2)_2$ 	120	131	1,6483 1,6353	161 185	Gelb. Ab 110° Körner, Blättchen, Nadeln. Gleichgew.: Balken.
165	p-Oxydiphenyl 	98	120	1,5700	175—177	Ab 90° Nadeln, meist in Bündeln; stark flüchtig (großes Deckglas!). Gleichgew.: lange Nadeln.
164—166	Chinasäure $\text{HO}-\text{CH}-\text{CH}_2-\text{C}(\text{OH})-\text{COOH}$ $\text{HO}-\text{CH}-\text{CH}(\text{OH})-\text{CH}_3$ <i>C₄H₆O₆</i>	131	159	1,4936	158—162	Schmelze wirft Blasen und erstarrt nur schwer. Gleichgew.: Prismen.
166	N,N'-Di-o-tolylthioharnstoff 	135	153	1,5502	174—176 steigt rasch an	Geruch. Ab 140° Nadeln. Zwischen 162° und 168° teilweises Schmelzen mit Gleichgew.: Stengel. In einzelnen Kristallen ab 160° „Kochen“; plötzliches Auskristallisieren von Plättchen und Stengeln mit F. zwischen 172—180°. Auf der Heizbank F. 181° (nur Zersetzungsprod.). Aus Schmelze Rosetten F. 146° und derbe Prismen F. 152°.
166	Hexamethylbenzol 	127	144	1,4083	175—180	Ab 55° Körner, Prismen, Rhomboide. Eutektische Temperatur mit Sublimat bestimmen! Dichte Sublimat. Straßenpflaster. Gleichgew.: Balken, Stengel, Prismen. Erstarrt zu Balkenwerk. Brechungsindex wegen Flüchtigkeit erschwert.
166	p-Aminobenzolsulfonamid (= Prontalbin) $\text{NH}_2-\text{C}_6\text{H}_4-\text{SO}_2-\text{NH}_2$	118	143	1,5897 1,5795	146—147 173—180	Ab 140° Blättchen, Körner. Gleichgew.: Körner. Dimorph.
166	Mannit $\text{HO}-\text{CH}_2-\text{C}(\text{H})(\text{OH})-\text{C}(\text{H})(\text{OH})-\text{C}(\text{H})(\text{OH})-\text{CH}_2\text{OH}$ <i>OH OH H</i>	133	161	1,4936 1,4942	156—157 190	Gleichgew.: Balken, Nadeln. Erstarrt zu Sphärolithen. Dimorph (77, 12).
166 (170)	Malonamid $\text{H}_2\text{C}(\text{CO}-\text{NH}_2)_2$	131	165	1,4842	182—185	Ab 110° Körner. Gleichgew.: Prismen. Gasblasen. Erstarrt in Platten mit F. 170°. Polymorph II 166°, III 165° (32).
167	4,4'-Diacetoxy-3-nitrodiphenylsulfon $\text{CH}_3-\text{CO}-\text{O}-\text{C}_6\text{H}_3(\text{NO}_2)-\text{SO}_2-\text{C}_6\text{H}_3(\text{O}-\text{CO}-\text{CH}_3)_2$ 	122	133	1,5204 1,5101	157—159 177—178	Ab 150° wenig Blättchen. Gleichgew.: Balken. Erstarrt teils glasig, teils sphärolithisch.
167	3,4,5-Trijodnitrobenzol 	122	135	>1,6877		Gelb. Geruch. Körner und Stäbchen ab 110°. Gleichgew.: Körner. Bei 160° sublimieren schöne Körner mit drei-, sechs- oder rechteckigem Umriß. Kristalle in Brechungsindexflüssigkeit löslich.

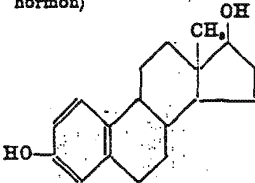
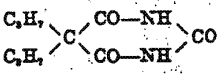
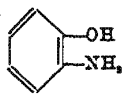
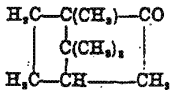
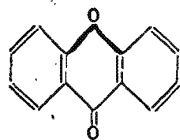
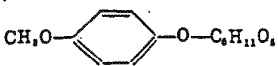
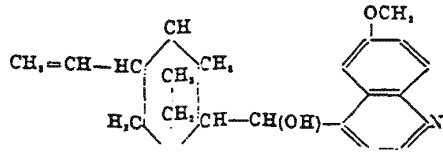
F. °C	Substanz	Eutekt. Phen-acetin	Temp. mit Benz-anilid	Glas-pulver	Temp. °C	Besondere Kennzeichen
167	Formaldehyd-2,4-dinitrophenylhydrazon 	120	132	1,6483	190	Gelb. Ab 95° Nadeln. Gleichgew.: Balken. Erstarrt sphärolithisch.
167	Stigmasterin 	128	147	1,4339— 1,4584		Ab 120° Nadeln, Körner. Gleichgew.: Körner, Stengel. Erstarrt sphärolithisch oder feinkristallin.
168	Diäthylstilböstrol (= 4,4'-Dioxy-α,β-diäthylstilben) 	123	138	1,5299	185—188	Ab 120° Nadeln, Stengel. Gleichgew.: Nadeln, Stengel. F. auf Heizbank 173°, sinkt nach 30 Sekunden auf 168° ab.
169	2-Methoxy-4-methyl-5-brombenzol-sulfonsäureamid 	113	129	1,5502 1,5403	161—162 182—183	Ab 110° Nadeln, Stengel. Gleichgew.: Nadeln, Stengel. Erstarrt sphärolithisch.
169	p-Benzamino-benzonitril 	116	133 tr.	1,5897 1,5895 1,5775	173—174 197—199	Ab 130° Nadeln, Spieße, Blättchen. Gleichgew. trägt: Prismen, Stengel.
169	p-Bromacetanilid 	116	129	1,5403 1,5299	177—179 197	Geruch. Ab 100° kleine Nadeln. Gleichgew.: Prismen, Platten.
168—170	β-Indolylleisigsäure (= Neoauxin) 	103	130	Zersetzung		Unangenehmer Geruch. Ab 110° Blättchen, Rauten, Stäbchen. Schmelze und Braunfärbung unter Gasblasenbildung. Gleichgew.: Rauten, sinkt rasch ab. Brechungsindex der Rauten 1,62 bis 1,66; in Anilin löslich unter Ausfall von Nadeln und Stengeln.
165—170	Kodeinhydrochlorid + 2 H ₂ O C ₁₇ H ₁₉ O ₅ N + HCl + 2 H ₂ O	Vgl. bei 260—270°				
		Benz-anilid	Salophen			
170	Allypinhydrochlorid C ₆ H ₅ -COO-C(CH ₂ -N(CH ₃) ₃) ₂ + HCl 	107	119	1,4936— 1,4842		Geruch. Ab 80—90° entweicht Wasser unt. Trübung. Ab 125° Körner. Gleichgew.: Körner. Erstarrt glasig. In Paraffin homogenes Schmelzen bei 90° mit Gleichgew.: Körner.
170	Atropinhydrochlorid C ₁₇ H ₂₁ O ₄ N + HCl	104	120	Zersetzung		Ab 120° Körner. Gleichgew.: Körner. Sinkt rasch ab. Beim Auflösen bei 130° vollständiges Schmelzen und Wiederauskristallisieren.

F. °C	Substanz	Eutekt. Temp. mit Benz.-anilid	Temp. Salophen	Glaspulver	Temp. °C	Besondere Kennzeichen
170	4-Acetaminodiphenyl 	131	147	1,5795 1,5700	177—178 198—199	Ab 100° Nadeln. Gleichgew.: Nadeln. Erstarrt zu Sphärolithenmosaik. Dimorph.
170	Anisaldazin (= Bis-[p-methoxyphenyl]-azimethylen) 	142	157	1,6131 1,6011	185—186 206—207	Gelb. Ab 90° Körner, Stengel. Gleichgew.: Prismen, Balken. $C_{10}H_{10}O_2N_2$
170	d-Weinsäure 	109	154	1,4683	148—150	Ab 155° viele Tröpfchen. Gleichgew.: Prismen. In Schmelze Gasblasen.
169—171	Pikramidsäure 	134	141	1,6231	165—170	Orange, ältere Präparate braun. Ab 180° Rauten, Körner, Prismen. Erstarrt derbkristallin. Für Brechungsindex bei 180° umsublimieren. Wegen Zersetzung und Braunfärbung erschwert.
171	Chinidin 	140	153	1,5611 1,5502	180—182 186—188	Ab 160° Nadeln. Gleichgew.: Nadeln, Stengel. Erstarrt glasig. Aus Alkohol Addukt mit 1 Mol. Kristallalkohol.
171	Gallacetophenon (= 2,3,4-Trioxycetophenon = Alizarin gelb C) 	136	140	1,5511	178	Ab 90° Körner, Prismen, Blättchen. Gleichgew.: Körner, Stengel. Erstarrt sphärolithisch. Bei längerem Erwärmen Zersetzung.
171	Di-β-naphthylamin 	141	156	1,6715 1,6598	200—202 224—226	Ab 150° Körner, Blättchen. Gleichgew.: Blättchen. Erstarrt sphärolithisch.
171	1,8-Dinitronaphthalin 	122 tr.	135 tr.	1,6011 1,5897	163—164 191—192	Bei 120—125° Umwandlung der Ursubstanz. Sublimiert in Nadeln. Gleichgew.: Platten. Erstarrt sphärolithisch. Polymorph II 153°, III unbeständig (30).
171	3,3'-Diaminodiphenylsulfon 	139		1,6231 1,6128	150—151 178—179	Ab 70° Körner. Gleichgew.: Prismen, Körner.
171	Ferulasäure (= m-Methoxy-p-oxyzimtsäure) 	139	146 tr.	1,5795	76	Ab 130° Prismen, Körner, Blättchen. Gleichgew.: Balken. Dimorph II 143° (8).

F. °C	Substanz	Eutekt. Benz- anilid	Temp. mit Salophen	Glas- pulver	Temp. °C	Besondere Kennzeichen
165—171	4-Oxy-m-toluylsäure 	128	141	1,5101	165—168	Ab 100° Nadeln, sehr flüchtig. Gleichgew.: Prismen, Spieße, Gasblasen. Erstarrt sphärolithisch. Mit Anilin sofort neue Nadeln, nach einer Minute feinkristalliner wolkiger Niederschlag.
152—172	N,N'-Di-o-tolyl-guanidin 	127	119 tr.	1,5700	162—163	Ab 100° Nadeln, später Spieße. Gleichgew.: Spieße. Polymorph.
170—172	Heroin (= Diacetylmorphin) 	135	122	1,5101 1,5000	166—170 189 20	Ab 150° vierseitige Blättchen. Gleichgew.: Spieße, Blättchen. Erstarrt teils glasig, teils kristallin.
169—172	Helmitol (= Anhydromethylencitronensäures Hexamethylentetramin) $(\text{CH}_3)_6\text{N}_4 + \text{H}_2\text{C} \begin{array}{c} \diagup \text{O} \diagdown \\ \diagdown \text{O} \diagup \end{array} \text{C} \begin{array}{c} \text{CH}_3\text{—COOH} \\ \text{CH}_3\text{—COOH} \end{array}$	158	161 tr.	Zersetzung		Zersetzung unter Bildung von Gasblasen. In Wasser l. löslich. Daraus Prismen und Linsen mit schiefer Auslöschung. γ' etwa 1,59, α' zwischen 1,57 u. 1,59. Bei Übersättigung Sphärolithe.
172	Synopen  $\text{N—CH}_2\text{—CH}_2\text{—N}(\text{CH}_3)_2 + \text{HCl}$	118	134	1,5611 1,5503	166—167 195—196	Ab 130° Stengel, Prismen. Gleichgew.: Stengel, Prismen.
172	Benzanthron 	134	153	1,6877	198—200	Gelb. Ab 120° Stäbchen. Körner. Gleichgew. träg: Stäbchen, Prismen. Erstarrt sphärolithisch.
172	$\omega,\omega,\omega',\omega'$ -Tetrabrom-p-xylol 	138	155	1,6011 1,5897	177—178 207—208 20	Geruch. Ab 100° Nadeln, Stäbchen, oft Büschel. Starke Sublimation. Gleichgew.: Körner, Prismen. Erstarrt sphärolithisch.
172	Dormovit (= 5-Furfuryl-5-isopropylbarbitursäure) 	137	154	1,4936 1,4842	157—159 187	Ab 120° Nadeln. Stengel. Gleichgew.: Stengel. Erstarrt glasig.
172	3,5-Dinitrosalicylsäure 	108	113 tr.	1,5795 1,5700	169—170 190—191 21	Ab 150° Nadeln. Gleichgew.: Nadeln, Stengel. Dimorph.

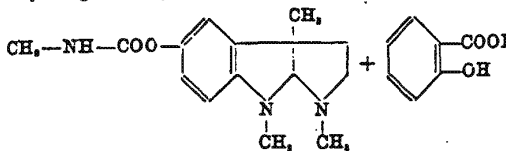
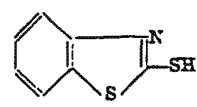
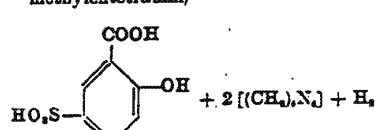
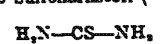
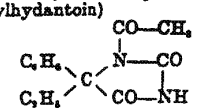
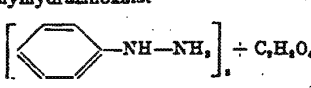
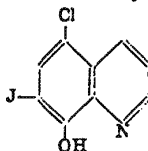
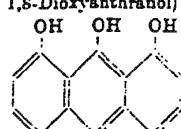
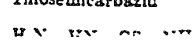
F. °C	Substanz	Eutekt. Temp. mit Benz-anilid	Temp. mit Salophen	Glas-pulvor	Temp. °C	Besondere Kennzeichen
172,5	Hydrochinon (= 1,4-Dioxybenzol) 	125	139	1,5204	197—198	Ab 95° Stengel. Rauten. Gleichgew.: Stengel. Dimorph (79, 78, 12). Stabile Modifikation geringe Doppelbrechung.
171—173	Chloramin  $\text{CH}_3\text{---C}_6\text{H}_4\text{---SO}_2\text{---NClNa} + 3 \text{H}_2\text{O}$	152	132	Zersetzung		Ab 55° entweicht Wasser und Trübung. Schmilzt unter Zersetzung. Erstarrt glasig. In Paraffininhomogenes Schmelzen 78° unter Bildung wasserfreier Kristalle (wenig Schmelze, daher wie Umwandlung). Aus Wasser lange Stengel und rhomboidische Körner, schiefe Auslöschung.
173	Phanodorm (= 5-Cyclohexenyl-5-äthyl-barbitursäure)  C_6H_9	136	151	1,5000 1,4936	170 189—191	Ab 130° Nadeln, Stengel, Prismen. Gleichgew.: Stengel, Prismen, Rauten.
173	Phenol-β-D-glucosid + 2 H ₂ O  $\text{HO---CH}_2\text{---C---C---C---C---C---O---C}_6\text{H}_5$ OHH OH	154	154	1,5101 1,5000	192—194 219—221	Ab 125° Nadeln. Gleichgew.: Nadeln. Erstarrt sphärolithisch.
173	p-Äthoxyphenylharnstoff (= Dulcin)  $\text{C}_2\text{H}_5\text{---O---C}_6\text{H}_4\text{---NH---CO---NH}_2$	140	153	1,5204	181—182	Ab 125° Nadeln, später Stäbchen. Gleichgew.: Platten, sinkt ab. Erstarrt breitstrahlig sphärolithisch. Dimorph.
173	Metycain (= di-[2-Methylpiperidino]-propylbenzoat-hydrochlorid)  CH_3 + HCl	116	130	1,5000	184—186	Ab 115° Nadeln. Gleichgew.: Nadeln, sinkt ab. Erstarrt glasig, bei Erwärmen kristallin.
173	Benzimidazol 	126	140	1,5700 1,5011	169—171 197	Ab 80° Körner, später dichte Sublimat. Gleichgew.: Körner. Erstarrt sphärolithisch.
172—174	1-Phenylsemicarbazid  $\text{C}_6\text{H}_5\text{---NH---NH---CO---NH}_2$	142	156	1,5611 1,5502	157—158 183—187	Ab 135° Nadeln, ab 165° verflühtes Netzwerk. Gleichgew. trägt: Stengel.
172—174	Tetramethyl-4,4'-diaminobenzophenon  $(\text{CH}_3)_2\text{N---C}_6\text{H}_4\text{---CO---C}_6\text{H}_4\text{---N}(\text{CH}_3)_2$	129	149	1,6353 1,6231	180—181 206—208	Hellgelb. Gleichgew.: Körner
174	Santonin  CH_3	122	143	1,5000 1,4936	176—177 193—194	Ab 120° Nadeln, Blättchen. Schmelzbeginn oft ab 100°. Gleichgew.: Prismen, gebogene Spieße. Dimorph (77, 12).

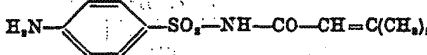
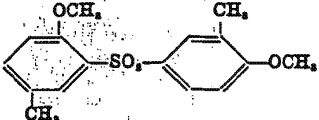
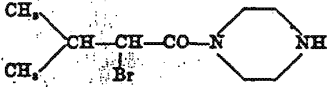
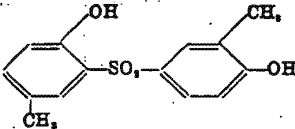
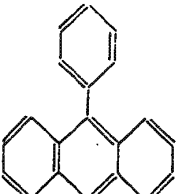
F. °C	Substanz	Eutekt. Temp. mit Benz-anilid	Salophen	Glas-pulver	Temp. °C	Besondere Kennzeichen
174	Medomin (= 5-Cycloheptenyl-5-äthylbarbitursäure) $\begin{array}{c} \text{CH}_3-\text{CH}_2-\text{CH} \\ \\ \text{CH}_2-\text{CH}_2-\text{CH}_2 \\ \diagdown \\ \text{C}=\text{C} \\ / \quad \backslash \\ \text{CO}-\text{NH} \quad \text{CO}-\text{NH} \\ \qquad \quad \\ \text{C}_6\text{H}_5 \end{array}$	143	159	1,4936 1,4842	176—178 201—203	Ab 70° Körner, Nadeln, Platten. Gleichgew.: Platten.
174	p-Dinitrobenzol NO ₂ , NO ₂	132	146	1,5101	186—188	Farblos bis hellgelb. Ab 90° wenig Nadeln, Stengel, Körner, Prismen. Gleichgew.: derbe Balken.
174	Narkotin 	131	152	1,5403	165—169	Ab 165° Tröpfchen. Gleichgew.: Prismen. Brechungsindex wegen Zersetzung schwierig. Mit Phenacetin n = 1,5611 bei 114—115°, 1,5700 bei 130—140°.
174	Dial (= 5,5-Diallylbarbitursäure) $\begin{array}{c} \text{CH}_2=\text{CH}-\text{CH}_2 \\ \\ \text{CH}_2=\text{CH}-\text{CH}_2 \\ \diagdown \quad \diagup \\ \text{C} \\ / \quad \backslash \\ \text{CO}-\text{NH} \quad \text{CO}-\text{NH} \\ \backslash \quad / \\ \text{CO} \end{array}$	142	156	1,4842	158—162	Ab 130° Körner, Prismen. Sublimiert dicht. Erstarrt sphärolithisch. Gleichgew.: Körner, Brechungsindex steigt bald an.
174	Pervitin (= N-Methyl-α-methyl-β-phenyl-äthylaminhydrochlorid) + HCl	132	145	1,5101 1,5000	157—159 192—194	Ab 125° Nadeln, Körner. Gleichgew.: Balken, Tafeln. Erstarrt zu grobstrahligem Sphärolithen.
175	Luminal (= 5-Phenyl-5-äthylbarbitursäure) $\begin{array}{c} \text{C}_6\text{H}_5 \\ \\ \text{C} \\ / \quad \backslash \\ \text{CO}-\text{NH} \quad \text{CO}-\text{NH} \\ \qquad \quad \\ \text{C}_2\text{H}_5 \end{array}$	137	153	1,5403 1,5301	151—152 176—179	Ab 135° Nadeln, Rauten. Gleichgew.: Nadeln, Rauten. Schmelze erstarrt glasig. Bei Erwärmen langsam Sphärolithe verschiedener Modifikationen. II 167°, III 160°, IV 157°, V 155° (SO, S).
175	Semicarbazidhydrochlorid $\text{H}_2\text{N}-\text{CO}-\text{NH}-\text{NH}_2 + \text{HCl}$	162	166	Zersetzung		Ab 140° feine Tröpfchen, ab 150° sehr dicht. Ab 165° Gasblasen in Kristallen. Beim Schmelzen Gasblasen und Zersetzung mit feinkristallinem Rückstand, der bis 200° nicht schmilzt. In Wasser löslich, daraus derbe Stengel. β längs, Achsenebene quer, Achsenwinkel mittelgroß, negativ α < 1,53, β zwischen 1,62 und 1,66.
157—175	Oreoselon 	120	131	1,5795	162—165	Ab 110° Nadeln. Gleichgew. sehr träg: Nadeln, Stengel. Aus Schmelze I und II 157°. Brechungsindex steigt rasch an (F. von Bruchhausen und H. Hoffmann).
165—175	Hydrochinin 	153 (÷ H ₂ O: 130 tr.)	165 (÷ H ₂ O: 130 tr.)	1,5302 1,5403	165—167 194—196	Ab 125° und 135° schmelzen einzelne Hydratkristalle. Später meist Wiederauskristallisieren. Ab 135° Nadeln und winzige Körner. Erstarrt glasig.

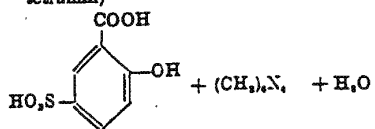
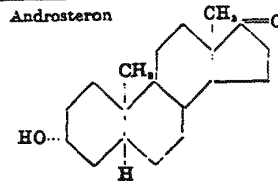
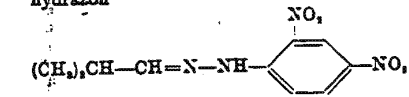
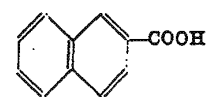
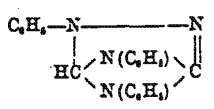
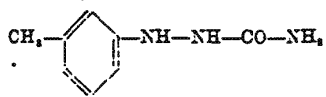
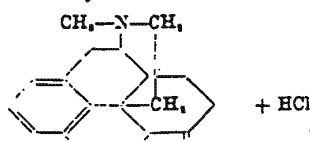
F. °C.	Substanz	Eutekt. Temp. mit Benz-anlid	Temp. mit Salophen	Glas-pulver	Temp. °C	Besondere Kennzeichen
175	Oestradiol (trans) (= Dihydro-a-follikel-hormon) 	135	151	1,5502 1,5403	154—156 182—189 20	Ab 110° Stäbchen. Gleichgew.: Stäbchen, Stengel, Blättchen. Erstarrt glasig.
175	5-Isopropyl-5-propylbarbitursäure 	143	160	1,4684	167—168	Ab 120° Nadeln, Stengel. Gleichgew.: Balken. Erstarrt zu Sphärolithen. Dimorph II etwa 155°.
175	o-Aminophenol 	133	149	1,5502	175—178	Ältere Präparate durch oberflächliche Zersetzung rotbraun. Umsublimieren bei 160°. Ab 90° quadratische Blättchen. Gleichgew.: rechteckige Tafeln. Brechungsindex wegen baldiger Braunfärbung erschwert.
175	Diisobutyramid (CH ₃) ₂ CH—CO—NH—CO—CH(CH ₃) ₂	189	153	<1,4339		Geruch nach Buttersäure. Ab 60° Nadeln, starke Sublimation. Gleichgew.: Stengel, Balken (großes Deckglas). Brechungsindex leichter in Silikonkammer.
174—176	Campher 	94	119	<1,4439		Geruch. Ab 35° rosettenartige Sublimation, später wabenartig. Verflüchtigt rasch. Bestimmung der eutektischen Temperatur im Sublimat. Brechungsindex in der Silikonkammer. Bei Raumtemperatur anisotrop, ab etwa 80° isotrop. In Indexflüssigkeit löslich. Bei Raumtemperatur Brechungsindex zwischen 1,50 und 1,51.
176	Xanthon (= Dibenzo-γ-pyrone) 	131	148	1,5897	181—182	Ab 100° Nadeln, Stäbchen. Starke Sublimation. Gleichgew.: Stengel, Spieße. Polymorph.
176	Methylarbutin 	129	153	1,5101	187—189	Bei 160,5° teilweises Schmelzen der instabiler. Modifikation und Kristallisation der stabilen. Gleichgew.: Nadeln, Stengel. Erstarrt glasig, beim Erwärmen Sphärolithe. Dimorph II 160,5° (81).
176	Chinin 	142	155	1,5611 1,5502	168—169 192—193	Ab 125° Nadeln, Nadelbüschel. Gleichgew.: Nadeln, Balken. Erstarrt glasig oder sphärolithisch. Chininhydrat ähnlich. Bei 120° aufgelegt schmilzt es und kristallisiert bald wieder aus.

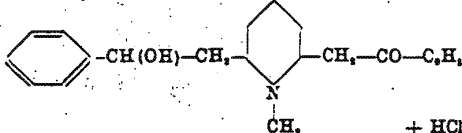
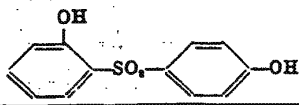
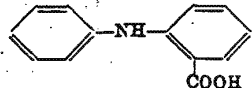
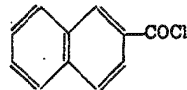
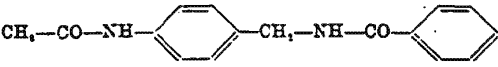
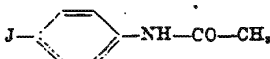
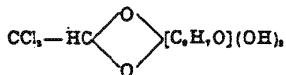
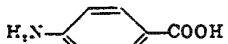
F. °C	Substanz	Eutekt. Benz-anilid	Temp. mit Salophen	Glas-pulver	Temp. °C	Besondere Kennzeichen
176	4-Benzalamino-antipyrin $\begin{array}{c} \text{CH}_3-\text{C}=\text{C}-\text{N}=\text{CH}-\text{C}_6\text{H}_5 \\ \quad \quad \\ \text{CH}_3-\text{N} \quad \quad \text{CO} \\ \\ \text{C}_6\text{H}_5 \end{array}$	131	150	1,6126 1,6011	179—181 202—203 23	Hellgelb. Ab 135° Nadeln, Körner. Gleichgew.: Balken.
176	4,4'-Dibromazorybenzol $\text{Br}-\text{C}_6\text{H}_4-\text{N}=\text{N}-\text{C}_6\text{H}_4-\text{Br}$	146	162	1,6483 1,6353	178—190 205—207 27	Orange. Ab 65° Körner, Nadeln. Gleichgew.: Balken, Blättchen. Dimorph.
172—177	Marbadal (= Badiionalsaures Marfanil) $\begin{array}{c} \text{H}_2\text{N}-\text{C}_6\text{H}_4-\text{S}(=\text{O})_2-\text{N}=\text{CS}-\text{NH}_2 \\ \\ \text{O} \end{array} \quad \begin{array}{c} \text{H} \\ \\ \text{H}_2\text{N}-\text{CH}_2-\text{C}_6\text{H}_4-\text{S}(=\text{O})_2-\text{NH}_2 \\ \\ \text{O} \end{array}$	156	164	Zersetzung		Schmilzt unscharf mit Gasblasen. Kein Gleichgew. In Alkohol wenig löslich; daraus sechseckige Blättchen mit gerader Auslöschung. Bismetrisches Achsenbild, Austritt einer Bisektrix. Achsenabstände quer, Achsenwinkel groß. In Sechsecken β längs (etwa 1,62), $\gamma > 1,66$.
160—177	Genatropinhydrochlorid $\begin{array}{c} \text{CH}_3-\text{CH}-\text{CH}_3 \\ \quad \quad \\ \text{O}=\text{N}-\text{CH}_2-\text{CH}-\text{O}-\text{CO}-\text{CH}-\text{CH}_2-\text{OH} \\ \quad \quad \quad \quad \\ \text{CH}_3 \quad \quad \text{C}_6\text{H}_5 \end{array}$	120	126	1,5204— 1,5299		Ab 120° Körner. Gleichgew.: Körner, sinkt ab. Erstarrt glasig. Mit Salophen $n = 1,5611$ bei 117—118°, 1,5502 bei 151—152°.
174—177	Acotin (= Dianisyl-phenetyl-guanidinhydrochlorid) $(\text{CH}_3\text{O}-\text{C}_6\text{H}_4-\text{NH})_2\text{C}=\text{N}-\text{C}_6\text{H}_4-\text{O}-\text{C}_6\text{H}_5 + \text{HCl}$	106	124	1,5795 1,5700	181—182 207—209 26	Erstarrt zum Teil kristallin, zum Teil glasig.
177	Arecolinhydrobromid $\begin{array}{c} \text{H}_2\text{C}-\text{CH}=\text{C}-\text{COO}-\text{CH}_3 \\ \quad \quad \\ \text{H}_2\text{C}-\text{N}(\text{CH}_3)-\text{CH}_3 \end{array} + \text{HBr}$	129	144	1,5299 1,5204	139—140 179—180 40	Ab 165° kleine Nadeln. Gleichgew.: Körner. Erstarrt glasig.
177	m-Kresotinsäure (= 2-Oxy-p-toluylsäure = 4-Methylsalicylsäure) $\begin{array}{c} \text{CH}_3 \\ \\ \text{C}_6\text{H}_3(\text{OH})_2 \\ \\ \text{COOH} \end{array}$	133	145	1,5000	195—197	Geruch. Ab 100° Nadeln, Stäbchen, Blättchen, später Umlagerung. Sehr flüchtig. Gleichgew.: Körner, Prismen. Erstarrt stengelig.
177	Styphninsäure (= 2,4,6-Trinitroresorcin) $\begin{array}{c} \text{OH} \\ \\ \text{O}_2\text{N}-\text{C}_6\text{H}_2(\text{NO}_2)_3-\text{OH} \\ \\ \text{NO}_2 \end{array}$	122	132	1,6011	173	Ab 130° vier- und sechseckige Blättchen, Körner, Stengel. Gleichgew.: Prismen. Schmelze gelb. Dimorph II 165,5° (82, 83).
177	Pyridin-Quecksilberdoppelsalz $\text{Pyridin} + \text{HgCl}_2 + \text{HCl}$	150	162	1,6483	211—212	Ab 120° feine Nadeln. Gleichgew.: Prismen, Spieße.
176—178	Brucin (Base) $\text{C}_{21}\text{H}_{20}\text{O}_8\text{N}_4 + 4 \text{H}_2\text{O}$	127	149	1,5700— 1,5795		Sehr bitter! Erstarrt glasig. Bei 140° aufgelegt, sofort Schmelzen des Hydrats und wasserfreie Kristalle. Mit Phenacetin $n = 1,5794$ bei 142—144°, 1,5700 bei 169—171°. In Paraffin schmelzen klare Hydratkristalle 80—90° inhomogen (Nadeln); die trüben werden ab 105° zu Blättchen umgelagert.

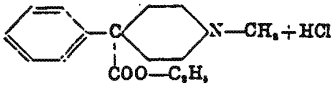
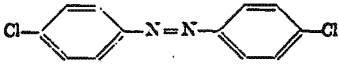
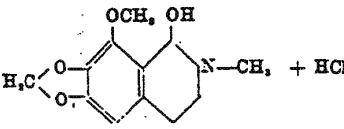
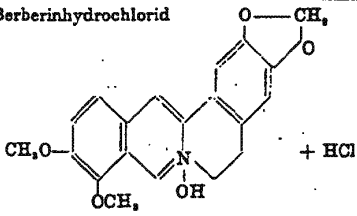
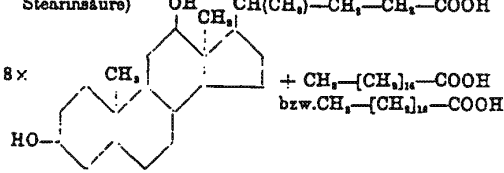
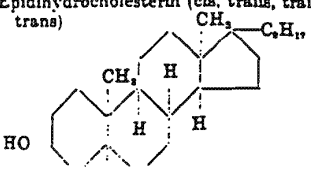
26

F. °C	Substanz	Eutekt. Temp. mit Benz-anilid	Temp. mit Salophen	Glas-pulver	Temp. °C	Besondere Kennzeichen
179	Physostigminsalicylat 	138	157	1,5299— 1,5403		Ab 150° Körner, Stengel. Gleichgew.: Körner; Balken. Erstarrt glasig. Mit Salophen $n = 1,5611$ bei 129—132°, 1,5502 bei 159—161°. Schmelze braun.
2 179	2-Mercaptobenzthiazol 	130	145	>1,6877		Geruch. Ab 110° Nadeln, Körner. Gleichgew.: Platten. Erstarrt breitstrahlig. Brechungsindex auch bei 220° > 1,0877.
170—180	Neohexal (= sec. 5-Sulfosalicylsäures Hexamethylen-tetramin) 	160	170	1,5611	150—160	Schmilzt unt. Gasblasenblg. Schmelze gelb, erstarrt glasig. Brechungsindex wegen Zersetzung schwer bestimmbar. In Paraffin inhomogenes Schmelzen bei 110°.
176—180	Sulfoharnstoff (= Thioharnstoff) 	—153—	170	1,6483	137—138	Ab 120° Körner, kurze Spieße. Kein Gleichgew., Reste wachsen erst ab etwa 175°.
177—180	Acetylnirvanol (= N-Acetyl-5-äthyl-5-phenylhydantoin) 	136	155	1,5000 1,4937	179—181 195—196	Ab 140° Stengel, Rhomben, Prismen. Gleichgew.: Prismen.
176—181	Phenylhydrazinoxalat 	155 tr.	163 tr.	Zersetzung		Ab 90° Nadeln, später Blättchen. Aus Wasser umkristallisiert, dünne, große Rauten, symmetrische Auslöschung. Im stumpfen Winkel γ' (etwa 1,66), α' wenig > 1,53.
180—181	Vioform (= 7-Jod-5-chlor-8-oxychinolin) 	144	163	1,6598	182—185	Ab 100° kleine Nadeln, allmählich sublimiert die ganze Substanz. Schmelze rasch braun. Brechungsindex wegen Zersetzung erschwert.
181	Cignolin (= 1,8-Dioxyanthranol) 	140	156	1,6231	185—188	Gelb. Ab 120° Stäbchen, Körner, reichlich Sublimate. Gleichgew.: Körner, Blättchen. Schmelze bald verfärbt. Brechungsindex steigt rasch an.
181	Thiosemicarbazid 	157	175	1,6598— 1,6715		Ab 120° Nadeln. Schmilzt unter Zersetzung mit Gasblasen, daher Bre-

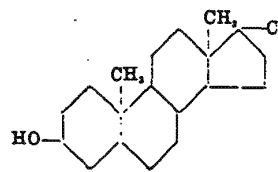
F. °C	Substanz	Eutekt. Temp. mit Benz-anilid	Temp. mit Salophen	Glas-pulver	Temp. °C	Besondere Kennzeichen
181	p-Aminobenzolsulfonsäure-β,β-dimethyl-acrylamid (= Irgamid) 	140	158	1,5611 1,5502	166—168 183—194	Ab 175° Tröpfchen. Gleichgew. sehr trüg: Prismen. Erstarrt mosaikartig (Markovic). 27
181	2,4'-Dimethoxy-5,3'-dimethyldiphenylsulfon 	137	157	1,5299 1,5204	186—187 208—210	Ab 160° Nadeln, Blättchen. Gleichgew.: Stengel, Prismen. Erstarrt sphärolithisch. 22
183	N-(α-Bromisovaleryl)-piperazid 	132	152	1,4938— 1,5000		Ab 130° lange Nadeln. Schmelze wird bald braun.
180—183	g-Strophanthin (= Ousbain) $C_{20}H_{24}O_8 + 8 H_2O$	146	169	1,5403 1,5299	178—180 218—220	Schmilzt unter langsamem Erweichen. In Paraffin schmelzen Hydrate bei 92° inhomogen. In Methylalkohol leicht löslich. Rückstand gibt mit Wasser isotrope Quadrate und anisotrope Vierecke mit „Fensterkreuz“ (92).
183	Phenylisopropylmethylaminpikrolonat C_6H_5 CH_3 $N-CH(CH_3)_2 + C_{10}H_8O_4N_2$	135	154	1,6126 1,6011	163—164 200—203	Gelb. Gleichgew.: Prismen, Körner. Erstarrt glasig bei Erwärmen sphärolithisch. Wegen Zersetzung bzw. Erstarrung Brechungsindex erschwert. 38
183	2,4'-Dioxy-5,3'-dimethyldiphenylsulfon 	130	163	1,5611 1,5502	169—170 183—194	Ab 120° Blättchen, Stäbchen, Körner. Gleichgew.: Prismen, Körner. Erstarrt teils sphärolithisch, teils glasig. Dimorph II 161°. 24
183	Jodival (= α-Jodisovalerylharnstoff) $(CH_3)_2CH-CH_2-CO-NH-CO-NH_2$	141	162	>1,6877		Ab 120° Nadeln, Stäbchen. Gleichgew. trüg: Stäbchen. Schmelze gelb, bald braun.
183 (176)	Veronal	Siehe F. 190°				Modifikation II 183° und IV 176° entstehen häufig bei Sublimation aus unreinem Material. Modifikation II pyramidenartig, IV flächenhaft, meist Rechtecke mit Zwillingsstreifung (87).
183.5	Noctal (= 5-Isopropyl-5-[β-bromallyl]-barbitursäure) $(CH_3)_2CH$ $CH_2=CH-CH_2$ C $CO-NH$ $CO-NH$ CO	142	158	1,5000 1,4937	180—182 197—198	Ab 160° wenig Blättchen, Nadeln. Gleichgew.: Körner. Polymorph II 180°, III 179° (33).
184	9-Phenylacridin 	137 tr.	150 tr.	1,6483 1,5353	196—197 224—226	Hellgelb. Ab 125° Körner, Blättchen. Gleichgew.: Körner. Erstarrt sphärolithisch. Polymorph II 177°, III 175° (30). 23

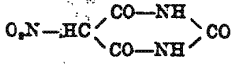
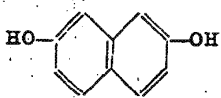
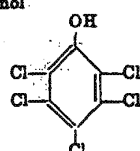
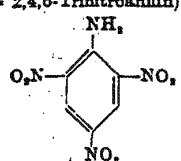
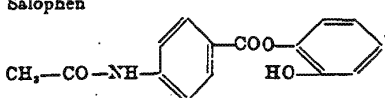
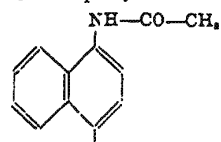
F. °C	Substanz	Entekt. Benz-anilid	Temp. mit Salophen	Glas-pulver	Temp. °C	Besondere Kennzeichen
184	Hexal (= 5-Sulfosalicylaures Hexamethylen-tetramin)  $\text{HO}_2\text{S}-\text{C}_6\text{H}_3(\text{OH})-\text{COOH} + (\text{CH}_2)_6\text{N}_4 + \text{H}_2\text{O}$	121 tr.	180	Zersetzung		Zwischen 110° und 120° einzelne Schmelztropfen. Schmilzt unter Zersetzung und Gasblasen. Aus Alkohol kleine Prismen (langsam Verdunsten) und lange Stengel (rasches Verdunsten). α' (länge) etwa 1,50, γ' zwischen 1,62 und 1,68.
184	Androsteron 	133	154	1,4842	187	Geruch, beim Erhitzen stärker. Ab 103° Nadeln, Körner. Gleichgew.: Körner. Erstarrt sphärolithisch.
184	Albucid (= p-Aminobenzolsulfonacetamid) $\text{NH}_2-\text{C}_6\text{H}_4-\text{SO}_2-\text{NH}-\text{CO}-\text{CH}_3$	142	161	1,5611— 1,5502		Ab 160° Körner. Gleichgew.: Körner, Prismen, sinkt rasch ab. Erstarrt glasig.
184	Isobutyraldehyd-2,4-dinitrophenyl-hydraxon  $(\text{CH}_3)_2\text{CH}-\text{CH}=\text{N}-\text{NH}-\text{C}_6\text{H}_3(\text{NO}_2)_2$	137	162	1,5897	189	Orange. Ab 115° Körner, später Nadeln, Prismen. Gleichgew.: Körner, Balken. Erstarrt sphärolithisch.
184	akt. Pseudoephedrinhydrochlorid $\text{C}_6\text{H}_5-\text{CH}(\text{OH})-\text{CH}(\text{NH}-\text{CH}_3)-\text{CH}_2-\text{HCl}$	144 tr.	156	1,5204 1,5101	173—175 212—214 39	Ab 160° Nadeln, später Blättchen. Gleichgew.: Blättchen.
184	β -Naphthoesäure 	141	156	1,5795	195—198	Ab 95° Nadeln, Blättchen. Gleichgew.: Balken, Tafeln. Erstarrt sphärolithisch. Wenn Rückstand, umsublimieren bei 170°. Brechungsindex steigt an.
184	Nitron 	139	150	1,6598	188—192	Gelb. Gleichgew.: Nadeln, Körner, sinkt ab. Schmelze rasch braun. Brechungsindexbestimmung erschwert.
190—184	Maretin (= 1-m-Tolylsemicarbazid) 	145	161	1,5403	172—173	Ab 170° unregelmäßige Blättchen. Erstarrt glasig.
178—185	Guanidinhydrochlorid $\text{NH}_2-\text{C}(\text{NH}_2)=\text{NH}-\text{HCl}$	160	174	1,5204 1,5101	182—184 214—244	Gleichgew.: netzartige Gebilde. Erstarrt sphärolithisch.
175—185	Dikodidhydrochlorid  $+ \text{HCl}$	121	147 tr.	1,5700— 1,5611		Ab 165° Erweichen. Erstarrt glasig. Bei 140° aufgelegt, sofortiges Schmelzen des Hydrates. Brechungsindex schwierig. Mit Salophen $n = 1,5794$ bei 120—122° 1,5700 bei 150—152°.

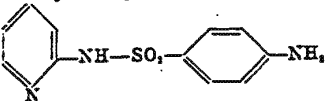
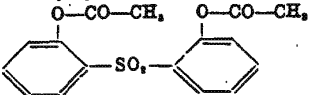
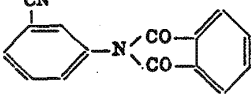
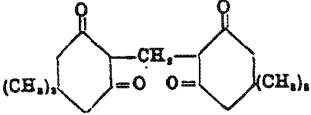
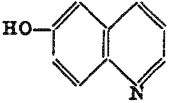
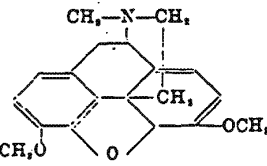
F. °C	Substanz	Eutekt. Benz-anilid	Temp. mit Salophen	Glas-pulver	Temp. °C	Besondere Kennzeichen
175—185	Lobelinhydrochlorid 	136	145	1,5502—1,5403		Schmilzt unter Zersetzung und Braunfärbung. Mit Salophen $n_D = 1,5611$ bei 147—149°; 1,5502 bei 170—171°.
180—185	Inulin (C ₆ H ₁₀ O ₅) _x	160 tr.	182		Zersetzung	Erweicht langsam unter Zersetzung und Gasblasen. Isotrop. Brechungsindex der festen Substanz 1,53 bei 28°.
185	Anissäure (= p-Methoxybenzoesäure) 	143	157	1,6000	197—199	Ab 100° Körner, Nadeln, Spieße. Gleichgew.: Balken. Erstarrt sphärolithisch. Brechungsindex wegen Flüchtigkeit erschwert.
185	o,p'-Dioxydiphenylsulfon 	131	148	1,5700 1,5611	190 215	Ab 155° Körner, Stäbchen, einzelne vierseitige Blättchen, die wieder wegsublimieren. Gleichgew.: Körner, Prismen. Erstarrt glasig oder sphärolithisch. Dimorph.
185	p-Aminophenol 	148	159		Zersetzung	Ältere Präparate bräunlich verfärbt. Umsublimieren bei 150°. Ab 125° Körner, Prismen. Ab etwa 170° Braunfärbung. Gleichgew.: Körner, Prismen. Schmelze dunkelbraun. Erstarrt sphärolithisch. Bei Sublimation Sechsecke und Rauten (Kantenwinkel 44°, γ' in langer Diagonale); α' zwischen 1,62 und 1,66, γ' > 1,74.
186	N-Phenylanthranilsäure (= N-Phenyl-o-aminobenzoesäure) 	141	158	1,5897	183—184	Hellgelb. Geruch. Ab 125° Stäbchen. Gleichgew.: Körner, Prismen. Schmelze gelblich, erstarrt federförmig oder sphärolithisch.
186	β-Naphthoylechlorid 	147	157	1,5795 1,5700	194 213	Ab 110° Nadeln, meist gekrümmt. Gleichgew.: Stengel. Erstarrt sphärolithisch.
186	[4-Acetaminobenzyl]-benzoylamin 	140	168	1,5611 1,5502	188—188 210—211	Gelblich. Gleichgew.: Nadeln. Erstarrt teils glasig, teils sphärolithisch.
186	p-Jod-acetanilid 	135	155	1,5795 1,5700	183—184 200—202	Ab 100° Nadeln, Körner. Gleichgew.: Körner, Prismen. Erstarrt sphärolithisch.
186	α-Chloralose 	145	161	1,4842—1,4083		Ab 100° Nadeln. Gleichgew.: Stengel, Spieße. Erstarrt sphärolithisch. Ursubstanz (oder Kristalle aus Alkohol) schwach anisotrope Stengel, α' längs, α' = 1,57 bei 30—35°.
187	p-Amino-benzoesäure 	140	183	1,5795	193	Ab 110° Nadeln, Stäbchen. Gleichgew.: Prismen, Spieße, sinkt ab, später Gasblasen.

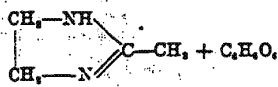
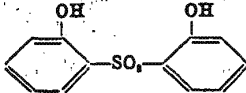
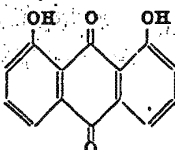
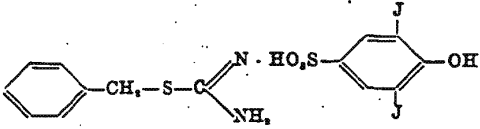
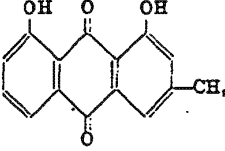
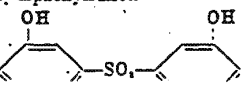
F. °C	Substanz	Eutekt. Temp. mit Benz-anilid	Temp. mit Salophen	Glas-pulver	Temp. °C	Besondere Kennzeichen
187	1-Methyl-4-phenyl-piperidin-4-carbon-säure-äthylesterhydrochlorid (= Dolan-tin = Petidinhydrochlorid) 	113	133	1,5204 1,5299	135—133 98—96	Ab 110° Körner, Blättchen, Rauten. Gleichgew.: Sechsecke, Körner, Spin-deln. Sinkt ab. Gasblasen. Erstarrt glasig, bei Erwärmen sphärolithisch. Dimorph (83). Brechungsindex bei sinkender Temperatur bestimmen!
187	β, β -Dinaphthyl 	152	168	1,6598 1,6483	194—195 219—221	Ab 120° Körner, Blättchen. Gleichgew.: Blättchen. Erstarrt sphärolithisch. Di-morph.
187	4,4'-Dichlorazobenzol 	149	167 tr.	1,6011 1,5897	205—207 227—229	Gelb. Ab 100° Nadeln. Gleichgew.: Tafeln. Aus Benzol kristallisiert limit 27° F. 168°; kristallisiert dann wieder aus.
187	Aristochin (= Dichinincarbonat) (C ₂₂ H ₂₁ O ₇ N ₂) ₂ CO	146	167	1,5502 1,5403	188—190 212—214	Gleichgew. sehr trög: unregelmäßige Formen. Erstarrt glasig.
182—188	Cotarninhydrochlorid (= Stypticin) 	118	137	Zersetzung		Gelb. Zwischen 70° und 90° entweicht Wasser unt. Trübung. Schmelze rot-braun, starke Gasentwicklung. Mit Salophen n = 1,6010 bei 93—96°, 1,5898 bei 133—136°. Schmilzt in Paraffin in-homogen 70—72°. In Wasser leicht lös-lich. Stengel α' (längs) etwa 1,50. Hygroscopisch.
182—188	Berberinhydrochlorid 	136	127	Zersetzung		Gelb. Ab etwa 150° verfärben sich die Kristalle. Schmilzt trög unter Zer-setzung und Gasblasen. Schmelze rot-braun. Aus Alkohol Nadeln und Nadel-büschel. α' längs, $\gamma' > 1,74$, α' etwa 1,53 (Brechungsindexbest. mit starker Ver-größerung!).
186—188	Aconitinnitrat C ₃₄ H ₄₇ O ₁₁ N + HNO ₃	140	158	1,5000— 1,5101		Erweicht unter Gelbfärbung und Gas-entwicklung.
188	Choleinsäure (= Addukt aus 8 Mol. Des-oxycholsäure + 1 Mol. Palmitin- bzw. Stearinsäure) 	152	163	1,4842	206—208	Gleichgew.: Stäbchen, Körner. Er-starrt glasig.
188	Epidihydrocholesterin (cis, trans, trans, trans) 	154	175	1,4584	197—198	Ab 130° Nadeln, Blättchen, Körner. Gleichgew.: Balken, Spieße. Erstarrt sphärolithisch oder verfilzt.

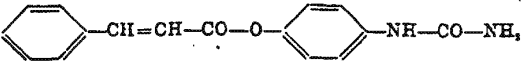
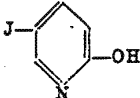
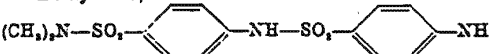
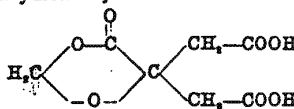
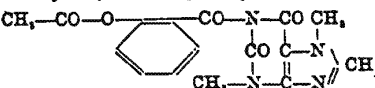
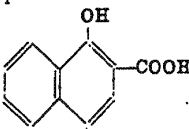
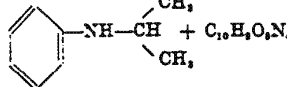
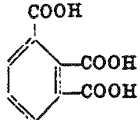
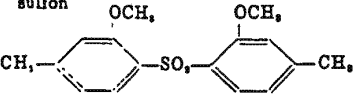
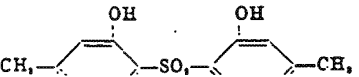
F. °C	Substanz	Eutekt. Benz-anilid	Temp. mit Salophen	Glas-pulver	Temp. °C	Besondere Kennzeichen
188	Bernsteinsäure <chem>HOOC-CH2-CH2-COOH</chem>	150	161	<1,4339		Ab 110° Körner, Nadeln. Sublimiert sehr dicht. Gleichgew.: lange Stengel, sinkt ab. Enantiotrop dimorph (59, 85). Bei 166° sublimieren Spieße und flache Aggregate mit rhomboidischen Stengeln, die sich im Winkel 86° überqueren, $\alpha' = 1,44$. In Anilin löslich mit Ausfallen langer Nadeln.
188	4-Methylumbelliferon (= 7-Oxy-4-methyl-cumarin) <chem>O=C1C=CC(=C(C=C1)OC(=O)C)O</chem> + H ₂ O	138	155	1,6011 1,5897	192—195 214—216 21	Ab 70—90° entweicht Wasser unter Trübung. Ab 140° kleine Vierecke, Körner, Prismen. Erstarrt sphärolithisch. Aus verdünntem Alkohol Hydratstengel und wasserfreie Körner. Hydrat in Paraffin zwischen 90—100° Umlagerung in körnige Aggregate.
188	2-[p-Aminobenzolsulfamino]-5-äthylthiadiazol (= Globucid) <chem>CC1=NC(=NS1)NC(=O)S(=O)(=O)c2ccc(N)cc2</chem>	145	164	1,6011 1,6126	191—192 158—159	Ab 180° Tröpfchen. Gleichgew.: Körner. Erstarrt glasig (Markovic).
183—189	Sympatol (= 1-[p-Oxyphenyl]-2-methyl-aminoäthanol-tartrat) 2x <chem>OC1=CC=C(C=C1)C(CO)CNC</chem> + C ₄ H ₆ O ₄	166 tr.	177	etwa 1,5700		Schmilzt unter Gelbfärbung und Gasblasen. Erstarrt glasig. Unmittelbar nach Schmelzen (<1,5700, nach zwei Minuten > 1,5700. Mit Salophen $n = 1,5611$ bei 151—155°.
189	Dulcit <chem>OC(CO)C(CO)C(CO)CO</chem>	162	174	1,4842 1,4683	188 239	Gleichgew.: Körner, Prismen. Erstarrt glasig, bei Erwärmen körnig und sphärolithisch.
185—190	Paracodinbitartrat (= Dihydrokodeinbi-tartrat) <chem>CC12C3C(C1OC2=CC=C3C4C(C(=O)O)C(CO)C(CO)C4=O)O</chem> + <chem>OC(=O)C(O)C(O)C(=O)O</chem>	158	170	1,5502— 1,5403		Ab 185° Tröpfchen. Gasblasen. Erstarrt glasig. Mit Salophen $n = 1,5611$ bei 145—147°, 1,5502 bei 173—175°.
185—190	1,5-Naphthylendiamin <chem>Nc1ccc2cc(N)ccc2c1</chem>	145	160	1,6483— 1,6353		Meist rötlichgrau verfärbt. Umsublimieren bei 170°. Ab 130° Nadeln, Stengel. Gleichgew.: Nadeln. Schmelze rasch braun. Erstarrt sphärolithisch. Sublimierte Stengel α' längs, α' wenig unter 1,74, $\gamma' > 1,74$ (eutektische Temperatur mit Dicyandiamid 176°).
184—190	N,N'-Di-p-tolylthioharnstoff <chem>Cc1ccc(NC(=S)Nc2ccc(C)cc2)cc1</chem>	168	185	1,5700	185—188	Ab 150° Körner, Prismen. Ab 135° Trübung der Kristalle. Gleichgew.: zugespitzte Säulen. Brechungsindex sinkt ab. Flüchtigkeit erschwert F. und Brechungsindex (großes Deckglas). Bei Schmelzen Geruch nach Anis. Erstarrt

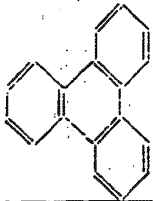
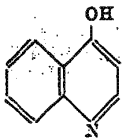
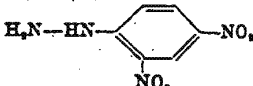
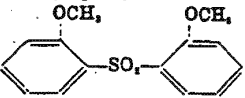
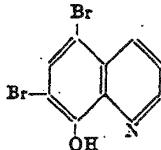
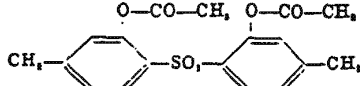
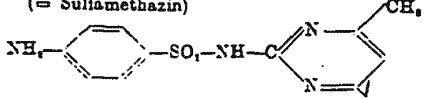
F. °C	Substanz	Eutekt. Temp. mit Benz-anilid	Salophen	Glas-pulver	Temp. °C	Besondere Kennzeichen
185—190	l-Ascorbinsäure (= Vitamin C = Cebion) $\text{HO}-\text{CH}_2-\overset{\text{H}}{\underset{\text{OH}}{\text{C}}}-\overset{\text{H}}{\underset{\text{OH}}{\text{C}}}-\overset{\text{O}}{\text{C}}=\text{C}-\text{CO}$	169	174	1,5101—1,5204		Ab 160° Tröpfchen, ab 170° Gelbfärbung. Beim Schmelzen Gasblasen. Für Brechungsindexbest. bei 195° auflegen und rasch beobachten.
185—190	Rohrzucker (= Saccharose) $\text{HOCH}_2-[\text{CH}(\text{OH})]_4-\text{CH}_2-\text{O}-\overset{\text{OH}}{\underset{\text{CH}_2\text{OH}}{\text{C}}}-[\text{CH}(\text{OH})]_4-\text{CH}_2\text{OH}$	163	173 tr.	1,5101 1,6000	172—174 205—208	Gleichgew. sehr trüg. Prismen. Erstarrt glasig. Brechungsindexbest. durch Gasblasen erschwert. 34
190	p-Acetaminobenzolsulfonamino-acetonitril $\text{CH}_3-\text{CO}-\text{NH}-\text{C}_6\text{H}_4-\text{SO}_2-\text{NH}-\text{CH}_2-\text{CN}$	149	169	1,5403	184	Gleichgew.: Platten, Spieße. Schmelze wird braun.
190	p-Aminobenzolsulfonylguanidin-monohydrat (= Sulfaguanidin) $\text{H}_2\text{N}-\text{C}_6\text{H}_4-\text{SO}_2-\text{NH}-\text{C}(=\text{NH})=\text{NH}_2 + \text{H}_2\text{O}$	169	168	1,6126	171—173	Ab 90° Kristalle meist trüb. Gleichgew.: Prismen, Körner. Erstarrt glasig, bei Erwärmen kristallin. In Paraffin Gasblasen ab 120°; einige Kristalle schmelzen homogen 130—135°, andere trüben sich.
190	Ephetonin (= racem. Ephedrinhydrochlorid) $\text{C}_6\text{H}_5-\text{CH}(\text{OH})-\text{CH}(\text{CH}_3)-\text{NH}-\text{CH}_2 + \text{HCl}$	162	165 tr.	1,5204 1,5101	178—180 214—217	Ab 100° quadratische Blättchen. Gleichgew.: Prismen, Blättchen. 39
188—190	Veronal (= 5,5-Diäthylbarbitursäure) $(\text{C}_2\text{H}_5)_2\text{C} \begin{matrix} \text{CO}-\text{NH} \\ \text{CO}-\text{NH} \end{matrix} \text{CO}$	163	172	1,4584	182—184	Ab etwa 100° Körner, häufig Umwandlung. Gleichgew.: Stengel. Polymorph II 183°, III 181°, IV 176° (87, 86, 10). Aus Schmelze spontan III, bei Erwärmen zweimal Umwandlung (siehe Übungsbeispiele).
190	Hippursäure (= N-Benzoylaminocessigsäure) $\text{C}_6\text{H}_5-\text{CO}-\text{NH}-\text{CH}_2-\text{COOH}$	183	141	1,5101	200	Ab 165° kurze Prismen, Stengel. Schmelze wird über 190° rosa, dann dunkler.
190	Lithocholsäure (= 3-Oxycholensäure) $\text{CH}_3-\text{CH}(\text{CH}_3)-\text{CH}_2-\text{CH}_2-\text{COOH}$  $\text{C}_{27}\text{H}_{46}\text{O}_3$	169	170	1,4936 1,4842	177—178 200—202	Ab 150° Tröpfchen. Gleichgew. trüg. Körner, sinkt ab. Erstarrt glasig.
190	d-Campfersäure $\text{H}_3\text{C}-\overset{\text{COOH}}{\underset{\text{C}(\text{CH}_3)_2}{\text{C}}}-\text{CH}_2-\text{CH}(\text{CH}_3)-\text{COOH}$	160	163	1,4339	207—209	Ab 130° Körner, später Blättchen, Spieße. Gleichgew.: Körner, Spieße. Erstarrt sphärolithisch. Trimorph. Brechungsindex steigt rasch an.
190	Dijodoform (= Äthylenperjodid = Tetra-jodäthylen) $\text{J}_2\text{C}=\text{CJ}_2$	178	166	>1,8877		Gelb. Zwischen 70° und 85° Umwandlung. Ab 135° Körner, Rauten, Prismen. Straßenpflaster. Gleichgew.: Rhomboide, Platten. Erstarrt sphärolithisch.

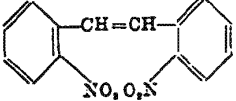
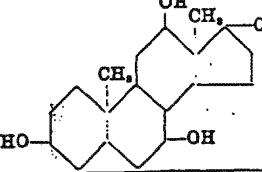
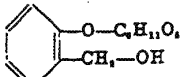
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
185—190	Dilitursäure (= 5-Nitrobarbitursäure) 	161	180	Zersetzung		Schmilzt mit heftigen Gasblasen und Bräunung. Aus heißgesättigter Wasserlösung bei langsamem Abkühlen Hydratstengel. In Paraffin Umlagerung zu wasserfreien Kristallen bei 120—125°, teilweise früher.
190	2,7-Dioxynaphthalin 	150	160	1,6126	198—202	Ab 110° Körner, gelappte Blättchen, Nadeln, flache Stengel. Erstes Schmelzen 185°, auskristallisieren und zweites Schmelzen 190°. Gleichgew.: Rauten, Platten. Bei 170° aufgelegt viele Nadeln und flache Stengel II, ab 180° träge Umwandlung oder F. II 185°. Teilweise Zersetzung. Erstarrt zu Platten und Strahlen. (Eutektische Temperatur Benzanilid 122°).
Verfl. 180—190 (195)	Hexachloräthan $\text{CCl}_3-\text{CCl}_3$	135	163	Verflüchtigung		Sublimation ab 30°. Bei etwa 50° Umwandlung der Ursubstanz. Reichlich Sublimata. Körner, Prismen, Spieße, die über 75° isotrop werden. Zweiter Up. 46°. Verflüchtigung 180—190°. F. in Silikonkammer 195°.
190	Pentachlorphenol 	133	186	1,5502 1,5403	205—206 226—227	Ab 70° Nadeln, Spieße. Ab 130° wabenartig. Gleichgew.: Platten. Erstarrt zu Platten. Bei Erwärmen beißender Geruch.
190	Pikrazid (= 2,4,6-Trinitroanilin) 	149 tr.	173	1,6483	183—189	Dunkelgelb. Ältere Präparate braun. Umsublimieren bei 180° und kleiner Abstand (Deckglas direkt auf die Substanz legen). Ab 130° Körner, Rauten. Gleichgew.: Rauten, Prismen.
190	Salophen 	190	176	1,5502 1,5403	189 215—216 21	Ab 150° Nadeln, Blättchen. Gleichgew.: Körner, Prismen, Balken. Polymorph II 162°, III 160° (30).
189—191	Cocainhydrochlorid $\text{C}_{17}\text{H}_{21}\text{O}_4\text{N} + \text{HCl}$	165	138	1,5101— 1,5204		Ab 170° Nadeln, Rechtecke, Prismen. Beim Schmelzen Gasblasen. Mit Salophen $n = 1,5611$ bei 117—118°, 1,5502 bei 146—147°.
188—191	Oxalsäure $\text{COOH} + 2 \text{H}_2\text{O}$ COOH	132 (+ 2 H ₂ O: 85°)	177	Zersetzung		Ab 60—90° entweicht Wasser unter Trübung. Ab 75° Spieße, Prismen. Ab 110° rapide Sublimation. F. unter heftiger Gasblasenentwicklung. In Paraffin schmilzt das Dihydrat homogen 100,5°.
191	N-Acetyl-4-brom-1-naphthylamin 	168	179	1,5897 1,5795	208—210 231—233 23	Ab 150° Nadeln. Gleichgew.: Nadeln. Erstarrt zu feinstrahligen Sphärolithen.

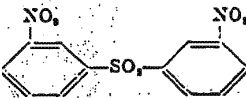
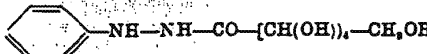
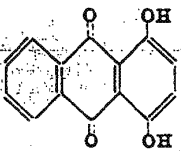
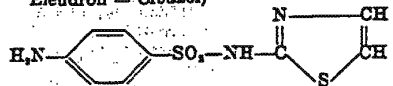
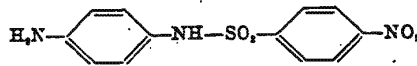
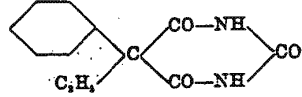
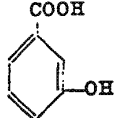
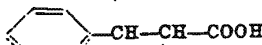
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
192	2-Sulfanilylaminopyridin (= Eubasin) 	169	173	1,6126 1,6011	170—171 201—202 3)	Ab 180° Tröpfchen, seltener kleine Kristalle. Gleichgew.: schiefe- und rechtwinkelige Kristalle. Erstarrt glasig, kristallisiert langsam bei Erwärmen. Polymorph II 179°, III 174° (Reimers) (67).
190—192	Atropinsulfat $2 C_{17}H_{21}O_4N + H_2SO_4 + H_2O$	152 (+H ₂ O: 120°)	100 tr.	1,5204— 1,5101		Schmilzt trög. Erstarrt glasig. Bei 160° aufgelegt schmelzen einzelne Hydratkristalle. Mit Salophen n = 1,5502 bei 134—139°, 1,5403 bei 165—167°. In Paraffin Dampfblasen ab 145° ohne Schmelzen.
192	2,2'-Diacetoxydiphenylsulfon 	168	180	1,5101 1,5000	187—188 210—211 23	Ab 170° Körner. Gleichgew.: Körner, Prismen.
192	m-Phthalimidobenzonitril 	160	190	1,5700 1,5611	197—199 217—211 20	Ab 130° Nadeln, Blättchen. Gleichgew.: Stengel, Balken.
192	Formaldomedon 	168	186	1,4683— 1,4584		Ab 100° Nadeln, Blättchen. Gleichgew.: Balken, Stengel.
188—193	2-[N ⁴ -Succinylsulfanilamino]-thiazol-monohydrat (= Sulfasuxidin) $HOOC-CH_2-CH_2-CO-NH-C_6H_4-SO_2-NH-C(=S)CH=CH_2 + H_2O$	173	140 tr.	1,6011	188—189	Kristalle ab 120° in mikrokristalline Aggregate umgewandelt. Erstarrt glasig. In Paraffin Gasblasen ab 120° (Markovic).
193	6-Oxychinolin 	152	185	1,6126	192—196	Wenn bräunlich verfärbt, umsublimieren bei 170°. Ab 125° Nadeln, schlanke Linsen, Vierecke, Tonnen, Dreiecke. Gleichgew.: Prismen, Sechsecke, Linsen. Schmelze gelb. Erstarrt sphärolithisch.
180—193	Thebainhydrochlorid $C_{18}H_{21}O_2N + HCl$	129 tr.	131	1,6126— 1,6231		Ab 65° Trübung einzelner Kristalle. Erstarrt glasig. Mit Salophen n = 1,6011 bei 85—87°, 1,5898 bei 119—124°. Schmelze sehr zäh mit Schlieren und Gasblasen. Wird bald braun.
193	Thebain 	165	186	1,5403— 1,5502		Ab 150° Quadrate, Rechtecke, Körner. Gleichgew.: vierseitige Blättchen. Bei Wiederholung Zersetzung. Mit Salophen n = 1,5898 bei 124—127°, 1,5794 bei 150—151.
193	Sedormid (= N-Allyl-N-isopropyl-N'-acetyl-harnstoff) $CH_2=CH-CH_2-CH_2-N-CO-NH-CO-CH_3$	169	186	<1,4339		Ab 100° Nadeln, später verfüzte Stengel und Balken. Gleichgew.: Stengel, Balken.

F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyan- diamid	Glas- pulver	Temp. °C	Besondere Kennzeichen
190—194	Narkotinhydrochlorid $C_{17}H_{21}O_2N + HCl$	125	106	Zersetzung		Ab 180° Nadeln. Mit Salophen n = 1,5700 bei 137—139°, 1,5611 bei 168° bis 170°. In Paraffin ab 110° Gasblasen und Trübung. Paraffinpräparat bei 150° aufgelegt inhomogenes Schmelzen mit sofortiger heftiger Blasenbildung.
190—194	Lysidinbitartrat 	182	133	1,4842— 1,4936		Schmilzt unter Gasblasen und baldiger Bräunung. In Wasser leicht löslich. Aus verdünntem Alkohol Trapeze u. Rhomboides; in Trapezen α' parallel d. Basis α' 1,53—1,55, γ' 1,56—1,57.
194	o,o'-Dioxydiphenylsulfon 	166	177	1,5502 1,5403	196—197 221—222 25	Ab 150° Körner, Prismen. Gleichgew.: Körner. Erstarrt zu Sphärolithen. Bei Erwärmen Umwandlung in derbe Prismen. Dimorph.
190—194	Acetbadional $CH_3-CO-NH-C_6H_4-SO_2-NH-CS-NH_2$	—119—	—170—	1,6011	183—185	Schmilzt mit mäßiger Gasblasenbildung. Brechungsindex trotzdem gut reproduzierbar.
194	1,8-Dioxyanthrachinon (= Istizin = Chrysazin) 	160	191	1,6598	217	Orange-gelb. Nadeln, Blättchen. Ab 120° Platten. Erstarrt zu derbstrahligem Aggregaten. Dimorph II etwa 183°.
192—194	S-Benzyl-thiuronium-3,5-dijod-4-oxybenzol-sulfonat 	168	169	—		Bei Erwärmen schwefeliger Geruch. Ab 170° wenig Körner. Gleichgew.: Prismen. Bei Umkristallisieren aus Benzol/Alkohol zweiaxige Quader (nahezu Würfel) und Rauten mit < 90°, schiefe Auslöschung (etwa 35°). α' wenig kleiner 1,66, γ' > 1,74.
180—195	Hyoseyaminsulfat (= l-Tropasäuretropin-ester-sulfat) $2 C_{17}H_{21}O_2N + H_2SO_4 + 2 H_2O$	146	105 tr.	1,5101— 1,5204		Beginnt bei etwa 110° zu erweichen. Schmilzt trög. Mit Salophen n = 1,5502 bei 128—135°, 1,5403 bei 157—164°. In Paraffin bei 130° aufgelegt, teilweises Schmelzen unter Dampfblasenblg.
180—195	Aconitin (= Acetyl-benzoyl-aconin) $C_{29}H_{41}$ $\begin{cases} N-CH_3 \\ (OH)_2 \\ (O-CH_3)_2 \\ O-CO-CH_3 \\ O-CO-C_6H_5 \end{cases}$	163	177	1,5000— 1,5101		Erweicht unter Gelbfärbung und Gasblasen. Mit Salophen n = 1,5403 bei 143—145°, 1,5399 bei 167—169°. Ursubstanz gestreckte Sechsecke, α' längs. In Indexflüssigkeiten löslich.
195	Chrysophansäure 	162	192	1,6598	184—188	Orange-gelb. Ab 110° Rauten (< 80°), Spicße, Nadeln. Gleichgew.: Rhomboides, Prismen. In erstarrter Schmelze hellgelbe stabile und orange instabile (II 190°) Sphärolithe.
195	m,m'-Dioxydiphenylsulfon 	147	154	1,5700 1,5611	211—212 243—244 52	Ab 170° Körnchen. Gleichgew.: Körner. Erstarrt sphärolithisch.

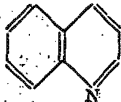
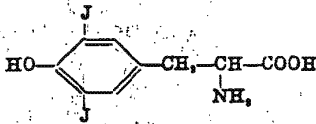
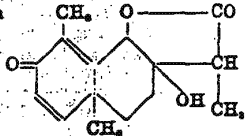
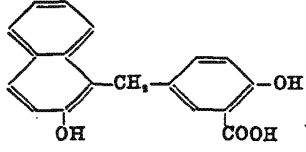
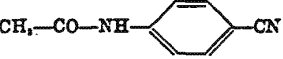
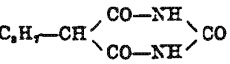
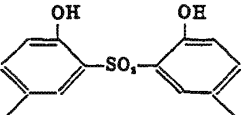
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
190—195	Elbon (= Cinnamoyl-p-oxyphenyl-harnstoff) 	Vgl. bei 270—275°				
190—195	5-Jod-2-oxypyridin 	161	162	1,6011— 1,6126		Ab 70° Kristalle dunkler. Ab 110° Stäbchen, Prismen. Sublimiert stark. Gleichgew. trägt Körner. Erstarrt sphärolithisch. Brechungsindex wegen Flüchtigkeit erschwert.
195	Uliron (= 4-Sulfanilylamino-benzolsulfonsäure-(1)-dimethylamid) 	163	176	1,5795 1,5700	173—176 200—203 27	Gleichgew.: Prismen. Schmelztropfen erstarrten glasig (Reimers) (67).
185—195	Anhydromethylencitronensäure 	169	133	1,4564— 1,4683		Ab 150° Tröpfchen. Schmilzt unter Zersetzung und Gasblasen. In Anilin löslich unter Abscheidung sphärolitischer Aggregate.
190—196	Theacylon (= O-Acetylsalicyl-theobromin) 	196	167	1,5502 1,5403	161—162 190—192 29	Ab 180° Tröpfchen. Erstarrt glasig. Bei längerem Erhitzen Zersetzung.
192—196	1-Oxy-2-naphthoesäure 	159	163	1,5795— 1,5897		Scharfer Geruch. Ab 130° Körner, starke Sublimation. Schmilzt unter Gasblasen und Verfärbung. Brechungsindex schwierig, steigt mit Braunfärbung. Bei 170° sublimieren Körner, Stengel, polare fünfseitige Blättchen. α' (Symmetrieebene) = 1,46, γ' = 1,66.
196	Phenylisopropylaminpikrolonat 	163	168	1,6126	205—208	Gelb. Ab 165° Blättchen, Körner, Nadeln. Gleichgew.: Prismen, sinkt ab.
196	Benzol-1,2,3-tricarbonsäure (= Hemimellitsäure) 	160 tr.	132 tr.	1,5299	192—194	Ab 50° entweicht Wasser unt. Trübung. Ab 160° Nadeln. Über 180° Umwandlung in Anhydrid und teilweises Schmelzen mit Umwandlung in große Körner. Gleichgew.: Körner, Prismen. Erstarrt sphärolithisch. In Paraffin inhomogenes Schmelzen des Hydrates 95—98°. Für Brechungsindex bei 140° trocken.
194—196	2,2'-Dimethoxy-4,4'-dimethyldiphenylsulfon 	161	179	1,5299 1,5204	186 209—211 24	Ab 120° Prismen, Nadeln. Gleichgew.: Körner, Prismen. In erstarrter Schmelze bei Erwärmen Umwandlung.
197	2,2'-Dioxy-4,4'-dimethyldiphenylsulfon 	155	172 tr.	1,5299 1,5403	213 191 27	Ab 100° Körner, Prismen. Ab 185° Schmelzen und Umwandlungen. Gleichgew.: Prismen. Dimorph.

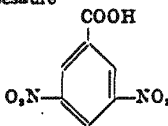
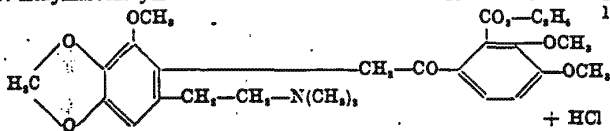
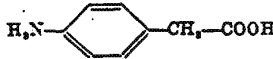
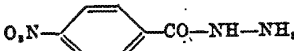
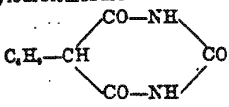
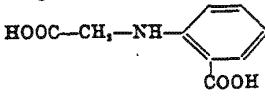
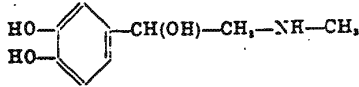
F. °C	Substanz	Eutekt. Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
194—197	Scopolaminhydrobromid $C_{17}H_{21}O_4N + HBr + 3 H_2O$	95 tr.	92 tr.	1,5502	172—176	Ab 90° schmilzt Hydrat, teilweise neue wasserfreie Kristalle. Bei 160° aufgelegt, totales Schmelzen, nach einer Minute wasserfreie Stengel. Für Brechungsindex bei 190° auflegen. Schäumen abwarten. Schmelze gelbbraun. Erstarrt glasig. In Paraffin homogenes Schmelzen 90°.
197	Anilinhydrochlorid  + HCl	108	123 tr.	1,5502— 1,5403		Ab 100° Körner, Stäbchen, später Straßenpflaster. Gleichgew.: Körner, Prismen. Brechungsindex durch große Flüchtigkeit erschwert (großes Deckglas). Bei 130° sublimieren anisotrope Dreiecke, Sechsecke, Scheiben.
198	Triphenylen 	170	197	1,6715 1,6593	197—200 224—226 26	Ab 125° Nadeln, Stengel. Gleichgew.: Stengel. Mit Dicyandiamid Mischungslücke der flüssigen Phasen. Erstarrt strahlig. Brechungsindex steigt an.
195—198	4-Oxychinolin 	160	168	—		Häufig bräunlich verfärbt. Umasublimieren bei 180°. Ab 140° Blättchen, Rechtecke, Prismen. Schmelze bald braun. Gleichgew.: Prismen, sinkt ab. Bei 180° sublimieren spitze Dreiecke (meist zwei an der Schmalseite verwachsen) und kleine Tonnen mit a' in der Symmetrieebene. a' zwischen 1,46 und 1,48, γ' über 1,74.
196—198	2,4-Dinitrophenylhydrazin 	162	174	1,6598— 1,6715		Orange mit violetter Schimmer. Ältere Präparate braun. Ab 150° Rhomboide. Schmelze dunkelorange. Gasblasen
198	2,2'-Dimethoxydiphenylsulfon 	—170—	188	1,5299 1,5204	198 222—224 25	Ab 140° Nadeln, Stengel. Gleichgew.: Prismen, Stengel. Erstarrt sphärolithisch. Brechungsindex wegen Flüchtigkeit erschwert.
198	5,7-Dibrom-8-oxychinolin 	171	196	1,6231	205—208	Ab 130° Nadeln, Prismen, Körner. Gleichgew.: Prismen, Stengel, sinkt ab. Brechungsindex wegen Flüchtigkeit erschwert.
192	2,2'-Diäcetoxy-4,4'-dimethyldiphenylsulfon 	156	—165—	1,5101 1,5000	185—186 205 19	Ab 160° Tröpfchen. Bei 185° Schmelzen und Wiedererstarren. Gleichgew.: bei 192° Prismen, Körner. Andere Kristalle Gleichgew.: 199° gelappte Kristalle. Erstarrt sphärolithisch. Polymorph II 192°, III 185°.
199	2-Sulfanilylamino-3,5-dimethylpyrimidin (= Sulfamethazin) 	167	172	1,5700 1,5795	186—187 165 27	Ab 190° kleine Körner, Nadeln, Prismen. Gleichgew.: Körner, sinkt ab. Erstarrt glasig (Markovic).

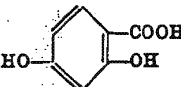
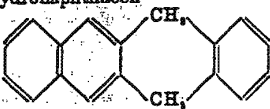
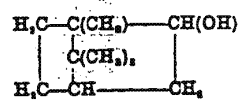
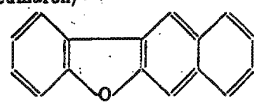
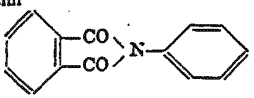
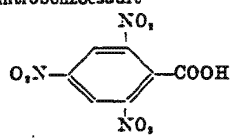
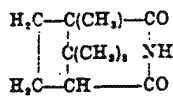
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
199	Nirvanol (= 5-Phenyl-5-äthylhydantoin) $\begin{array}{c} \text{C}_6\text{H}_5 \\ \diagup \\ \text{C} \\ \diagdown \\ \text{C}_2\text{H}_5 \end{array} \begin{array}{l} \text{CO}-\text{NH} \\ \text{NH}-\text{CO} \end{array}$	167	175	1,5101 1,5000	203—205 231—233	Ab 160° wenig Spindeln, Spieße. Gleichgew. trüg: Balken, Spieße. Erstarrt glasig. Bei Erwärmen Strahlenbüschel.
199	2,2'-Dinitrostilben 	170	195	1,5897 1,5795	195—196 222—223	Hellgelb. Ab 130° Körner, Nadeln. Stengel. Gleichgew.: Balken.
197—199	Larocain (= p-Aminobenzoessäure-β,β-dimethyl-γ-diäthylaminopropylesterhydrochlorid) $\text{NH}_2-\text{C}_6\text{H}_4-\text{CO}_2-\text{CH}_2-\text{C}(\text{CH}_3)_2-\text{CH}_2-\text{N}(\text{C}_2\text{H}_5)_3 + \text{HCl}$	156	125	1,5403 1,5299	182—183 212—213	Ab 160° Nadeln, Keile, Dreiecke, Spindeln. Gleichgew. trüg: polare Prismen, Platten.
198—200	Cholsäure (= 3,7,12-Trioxycholansäure) 	167	163	1,5000	210—211	Ab 180° Tröpfchen. Nach Schmelzbeginn feinkörnige Masse. Erstarrt glasig.
198—200	p-Methoxy-acetophenon-azin $\text{CH}_3\text{O}-\text{C}_6\text{H}_4-\text{C}(\text{CH}_3)=\text{N}-\text{N}=\text{C}(\text{CH}_3)-\text{C}_6\text{H}_4-\text{OCH}_3$	174	197	1,5611	214—215	Gelb. Ab 130° Nadeln, Stengel, Körner. Gleichgew.: Stengel, Prismen. Erstarrt sphärolithisch.
180—200	Narceinhydrochlorid $\text{C}_{22}\text{H}_{27}\text{O}_7\text{N} + \text{HCl}$	189 tr	158	1,5411— 1,5700		Ab 155° teilweises Schmelzen, sehr trüg unter Gelbfärbung. Erstarrt glasig. Mit Salophen n = 1,5794 bei 102—103°, 1,5700 bei 142—144°. In Paraffin inhomogenes Schmelzen bei 98—100° unter Abscheidung vieler Körner. Gasblasen.
198—200	S-Benzyl-thiuronium-4-sulfosalicylat $\text{C}_6\text{H}_5-\text{CH}_2-\text{S}-\text{C}(\text{NH}_2)=\text{NH} \cdot \text{HO}-\text{S}-\text{C}_6\text{H}_3(\text{OH})(\text{COOH})$	172	141	—		Beim Erwärmen schwefeliger Geruch. Schmilzt unter Zersetzung und Gasblasen. Mit Anilin zunächst Ausfall grauer Linsen, nach ein bis zwei Minuten langnadelige, dichte Sphärolithe.
195—200	d-Glutaminsäure $\text{COOH}-\text{CH}_2-\text{CH}_2-\text{CH}(\text{NH}_2)-\text{COOH}$	167	169	1,4683	195—190	Geruch. Ab 175° einzelne Kristalle braun. Ab 190° Tröpfchen. Schmilzt trüg unter Gasblasen. Brechungsindex nach einer Minute an Restschmelze bei sinkender Temperatur bestimmen.
198—200	Pilocarpinhydrochlorid $\text{C}_8\text{H}_9-\text{CH}(\text{CO}-\text{O}-\text{CH}_3)-\text{CH}_2-\text{C}(\text{CH}_3)=\text{N}-\text{N}=\text{C}(\text{CH}_3)-\text{CH} + \text{HCl}$	153	101	1,5000 1,4936	211—212 233—236	Ab 140° Körner, Blättchen, Nadeln. Gleichgew.: Körner.
200	Arbutin $\text{HO}-\text{C}_6\text{H}_4-\text{O}-\text{C}_8\text{H}_{11}\text{O}_4$	166	156	1,5403 1,5299	168—169 198—199	Ab 65° entweicht Wasser. Bisweilen Schmelzen bei 165°, dann Wiedererstarren. Dimorph. II 165° (81).
200	Salicin (= Saligeninglucosid) 	173	160	1,5204 1,5101	199—202 233—235	Gleichgew.: Rechteckige Kristalle. Erstarrt glasig. Sehr bitter.

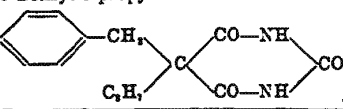
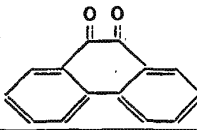
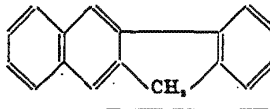
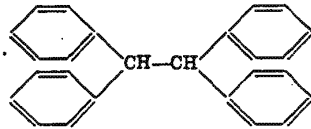
F. °C.	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C.	Besondere Kennzeichen
etwa 200 (214)	N,N'-Dimethyloxamid $\text{CH}_3\text{—NH—CO—CO—NH—CH}_3$	168	174	<1,4339		Geruch. Ab 60° Stäbchen. Verflüchtigung etwa bei 200°, unter großem Deckglas F. 214°. (Für Brechungsindex großes Deckglas!)
201	3,3'-Dinitrodiphenylsulfon 	172	196	1,5611 1,5502	198—199 223—224 25	Ab 170° Blättchen. Gleichgew.: vierseitige Platten. Erstarrt sphärolitisch.
198—201	Pikrotoxin $\text{C}_{10}\text{H}_{10}\text{O}_5 + \text{C}_{10}\text{H}_{10}\text{O}_7$	161	168	etwa 1,4842		Ab 170° Tröpfchen. Gleichgew.: Stengel, Balken. Schmelze braun. Ursubstanz Nadeln, γ' längs, γ' zwischen 1,57 und 1,59; α' zwischen 1,53 und 1,55.
199—201	Gluconsäurephenylhydrazid 	177	168	1,5299— 1,5403		Schmilzt unter Gelbfärbung und geringer Gasblasenbildung.
201	Chinizarin (= 1,4-Dioxyanthrachinon) 	186	201	>1,6715		Rotgelb. Ab 145° Sublimat. Zwischen 180—190° intensive Rotfärbung. Gleichgew.: Rauten, Prismen. Enantiotrop dimorph I rot 201°, II orange 195° (10).
201	Sulfathiazol (= 2-Sulfanilylaminothiazol = Eleudron = Cibazol) 	172 (159)	167 (152)	1,6463— 1,6353		Ab etwa 160° teilweise Umwandlung, teilweise F. 174° (Gleichgew.: Stengel, Blättchen, oft sechseckig). Gleichgew. bei 201°: schiefwinkelige Stengel, Blättchen (Reimers) (67).
202	N-[4-Nitrobenzolsulfonyl]-p-phenylendiamin 	167	179	1,5795— 1,5897		Gelb. Ab 180° Blättchen. Gleichgew.: Prismen, Platten. Schmelze wird gelb und braun.
202	5-Äthyl-5-cyclohexylbarbitursäure 	168	168	1,4683	215	Ab 140° Körner. Gleichgew. träg: Körner, Platten. Dimorph. Brechungsindex durch Flüchtigkeit erschwert.
202	m-Oxybenzoesäure 	156	148	1,5101— 1,5204		Ab 120° Stäbchen. Gleichgew.: Stengel, Balken. Erstarrt zu Sphärolithen. Sehr flüchtig. Bei 190° sublimiert instabile Modifikation (F. 193°) in längsgestreiften Rechtecken (α' unter 1,44, γ' über 1,74) und kurzen Leisten (α' und γ' über 1,75); ferner stabile Stengel mit β (längs) zwischen 1,62 und 1,66 (Austritt opt. Achse).
200—202	α,β -Dibromhydrozimtsäure (= inakt. Zimtsäuredibromid) 	155	155	1,5299— 1,5403		Ab 120° Blättchen, Körner, Prismen. Schmilzt unter Gasblasenbildung.

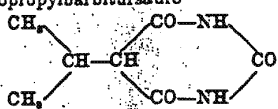
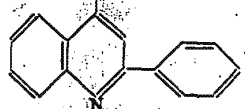
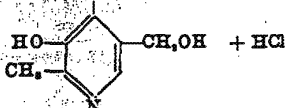
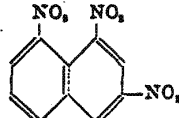
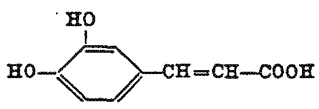
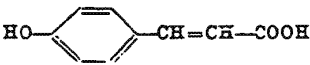
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
202	Mesaconsäure $\begin{array}{c} \text{CH}_3 \\ \\ \text{C}-\text{COOH} \\ \\ \text{HOOC}-\text{CH} \end{array}$	160	130 tr.	1,4339	220—225	Ab 85° Blättchen, Stengel. Gleichgew.: Balken. Schmelze bald braun. Dimorph. Für Brechungsindex Präparat erst bei 220° auflegen.
200—203	Benzoyl-l-ekgonin $\begin{array}{c} \text{CH}_3-\text{CH}-\text{CH}-\text{COOH} \\ \quad \quad \\ \text{N}-\text{CH}_2 \quad \text{CH}-\text{O}-\text{CO}-\text{C}_6\text{H}_5 \\ \quad \quad \\ \text{CH}_3-\text{CH}-\text{CH}_3 \end{array} + \text{H}_2\text{O}$	144	164	1,5101	198	Ab 70° schmelzen einzelne Hydratkristalle, andere trüb. Ab 175° Rechtecke, kurze Prismen. Polymorph II 181°, III 135°, IV 105° (10). In Paraffin homogenes Schmelzen bei 90° mit Gleichgew.: Stengel, seltener inhomogenes Schmelzen bei 85°.
203	Protocatechusäure (= 3,4-Dioxybenzoesäure) $\begin{array}{c} \text{COOH} \\ \\ \text{C}_6\text{H}_2 \\ \quad \\ \text{HO} \quad \text{OH} \end{array} + \text{H}_2\text{O}$	149	150	Zersetzung		Zwischen 95—120° entweicht Wasser unt. Trübung. Ab 145° vielseitige Blättchen. Schmilzt unter Gasblasen und leichter Gelbfärbung. In Paraffin Dampfblasen ab 110°, bei 120°—125° Umlagerung. In Wasser erhitzt; bei Abkühlen zuerst lange Stengel, dann Hydrat in prismatischen Rhomboiden (113°) mit schiefer Auslöschung (20°).
203	m-Nitrozimtsäure $\begin{array}{c} \text{C}_6\text{H}_4 \\ \\ \text{NO}_2 \end{array} - \text{CH}=\text{CH}-\text{COOH}$	162	160	1,5502	212—214	Ab 115° Nadeln, starke Sublimation Gleichgew.: Körner, Stengel. Schmelze gelblich. Brechungsindex wegen Flüchtigkeit erschwert.
203	p-Nitrobenzamid $\text{O}_2\text{N}-\text{C}_6\text{H}_4-\text{CO}-\text{NH}_2$	164	172	1,5403 1,5299	211 234—235	Hellgelb. Ab 150° Körner, Prismen, Blättchen. Gleichgew.: Prismen, Körner. Erstarrt sphärolithisch.
195—204	Doryl (= Carbaminoyl-cholinchlorid) $\text{NH}_2-\text{CO}-\text{O}-\text{CH}_2-\text{CH}_2-\text{N}(\text{CH}_3)_3\text{Cl}$	173	108 tr.	1,4842— 1,4937		Aminartiger Geruch. Ab 160° Trübung. Gleichgew. trüg: Prismen, sinkt ab. Schmilzt unter Gasblasen. Schmelze zäh, gelblich; erstarrt glasig, bei Erwärmen kristallin.
204	Cinchonidin $\begin{array}{c} \text{CH} \\ \\ \text{CH}_3=\text{CH}-\text{HC} \begin{array}{c} \text{CH}_3 \\ \\ \text{CH}_2 \\ \\ \text{H}_3\text{C} \end{array} \text{CH}-\text{CH}(\text{OH})-\text{C}_8\text{H}_6\text{N} \end{array}$	166	193	1,5403— 1,5502		Ab 180° Stäbchen, Körner, Blättchen. Gleichgew. trüg: Körner. Mit Salophen $n = 1,5700$ bei 151—154°, 1,5502 bei 185—187°.
204	Benzoesäure-[p-bromanilid] $\text{Br}-\text{C}_6\text{H}_4-\text{NH}-\text{CO}-\text{C}_6\text{H}_5$	167	196	1,5795 1,5700	201—203 220—221	Ab 140° Nadeln, Stengel, Blättchen. Gleichgew. trüg: Stengel, Nadeln.
204	Isatin $\begin{array}{c} \text{C}_6\text{H}_5 \\ \\ \text{C}=\text{O} \\ \\ \text{NH} \end{array}$	157	161	1,5795 1,5700	204—206 223—229	Ziegelrot. Ab 160° kurze Prismen. Gleichgew.: Prismen.
204	Ipral (= 5-Äthyl-5-isopropylbarbitursäure) $\begin{array}{c} \text{CH}_3 \\ \\ \text{CH} \\ \\ \text{CH}_3 \end{array} \text{C} \begin{array}{c} \text{CO}-\text{NH} \\ \\ \text{CO}-\text{NH} \end{array} \text{CO}$	165	180	1,4584— 1,4339		Ab 140° Nadeln, Spieße. Gleichgew.: Balken, Nadeln.
199—204	Ferropyrin (= 3 Antipyrin + 2 FeCl ₃) $3 \text{C}_{11}\text{H}_{11}\text{ON}_3 + 2 \text{FeCl}_3$	166	162	—		Bräunlichrote Aufhellung der Körner ab 190°. Schmelze zuerst orange, wird blutrot, bei weiterem Erhitzen Bildung gefiederter Sterne in der Schmelze. Mit Wasser am Objektträger erhitzt, Zersetzung unter Abscheidung von Tropfen, die entzündlich explodieren.

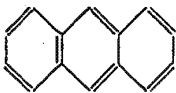
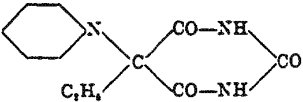
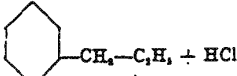
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
204	Chinolinpikrat  + C ₇ H ₅ O ₄ N ₃	164	165	1,6482	218	Gelb. Unangenehmer Geruch. Ab 130° Körner, rhomboid. Prismen. Gleichgew.: Spieße und Nadeln.
204	2,6-Dijodtyrosin (= Jodgorgosäure=Dityrin) 	180	170	—	—	Während des Erwärms Trübung. Ab 185° Tropfen und Körner. Schmilzt unter Gasblasen und Braunfärbung. In Paraffin ab 120° Gasblasen, bei 150° sehr heftig. Teilweises Schmelzen unter Zersetzung ab 160°. Ursubstanz schwach anisotrop mit n zwischen 1,66 und 1,74.
200—204	Artemisin 	156	169	1,5000	200—225	Ab 135° Nadeln, Stäbchen, Körner. Gleichgew.: Prismen, Stengel. Schmelze gelblich, erstarrt glasig.
190—205	Chinidinsulfat 2 C ₂₁ H ₂₈ O ₄ N ₂ + H ₂ SO ₄ + 2 H ₂ O	158	146	1,5502— 1,5611	—	Schmilzt sehr trög. Schmelze bald braun, zeigt Schlieren.
190—205	Scopolaminhydrochlorid C ₁₇ H ₂₁ O ₄ N + HCl + 2 H ₂ O	95 tr.	90 tr.	1,5204— 1,5299	—	Ab 90° Schmelzen des Hydrates, später wasserfreie Kristalle, ab 150° Nadeln, Stengel. Schmelze braun. Mit Salophen n = 1,5611 bei 125—128°, 1,5502 bei 158° bis 161°. In Paraffin teils homogenes Schmelzen, teils inhomogenes Schmelzen bei 75—80°.
190—205	Brucinhydrochlorid C ₂₈ H ₂₈ O ₄ N ₂ + HCl	156 tr.	126 tr.	1,8011	180—185	Schmilzt sehr zäh, viele Luftblasen. Schmelze langsam bräunlich. Erstarrt glasig. Mit Salophen n = 1,5794 bei 174—176°.
200—205	Episcarin (= β-Oxynaphthyl-p-oxy-m-toluylsäure) 	161	187	1,8353— 1,8483	—	Ab 180° Nadelbüschel. Ab 185° beginnt Verfärbung. Schmelze bald braun, besonders am Rand der Tropfen.
205	p-Acetaminobenzonitril 	162	178	1,5204 1,5101	221—222 224—246	Gelb. Ab 100° Nadeln. Körner, Blättchen. Gleichgew.: Balken, Blättchen.
205	5-Propylbarbitursäure 	169	165	1,4584	219	Ab 140° Nadeln, Blättchen. Gleichgew.: Stengel, Spieße. Erstarrt sphärolithisch.
206	2,2'-Dioxy-5,5'-dimethyldiphenylsulfon 	—150—	—195—	1,5299 1,5203	207 228—230 228	Ab 100° Prismen, Körner. Gleichgew. trög. Spindeln, Prismen. Erstarrt glasig, bei Erwärmen kristallin.

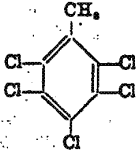
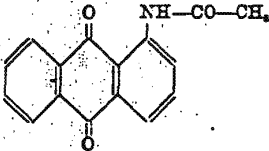
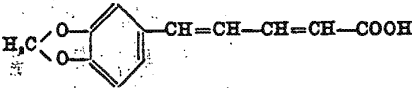
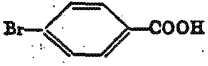
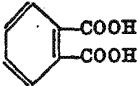
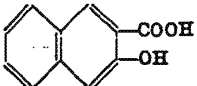
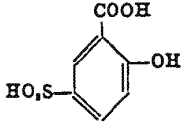
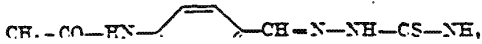
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
208	2-Methylantracen 	173	205	1,6126	225—228	Manchmal hellgelb verfärbt. Umsublimieren bei 200° (dann die ersten Fraktionen oft niedrigerer F.). Ab 115° Blättchen, gelappte Platten. Gleichgew.: rundliche Scheiben und Platten. Mit Pikrinsäure rotbraune Verbindung (Kontaktpräparat). Brechungsindex 1,6353 etwas unterhalb F.
206,5	3,5-Dinitrobenzoesäure 	142	133	1,5403	185	Hellgelb. Ab 150° Stengel, Blättchen, Körner. Gleichgew.: sehr große Prismen.
195—207	Cevadin $C_{17}H_{20}O_4N + H_2O$	170	190	1,4936 1,4842	182—185 210—213	Niesenerregend. Ab 100° entweicht Wasser unt. Trübung. Einzelne Kristalle schmelzen als Hydrat. Erstarrt glasig.
195—207	Äthylnarceinhydrochlorid 	157	142	1,5502— 1,5403		Ab 190° einzelne Nadeln. Schmelze gelb, erstarrt glasig. Mit Salophen n = 1,5611 bei 133—135°, 1,5502 bei 162—164°.
198—207	p-Aminophenyllessigsäure 	163	153	etwa 1,5403		Ab 150° Körner, Spieße. Schmilzt unter Gelbfärbung und Gasblasen. Für Brechungsindex bei 215° auflegen, unmittelbar nach dem Schmelzen < 1,5403, nach einer Minute > 1,5403.
196—208	Cinchonidinsulfat $2 C_{20}H_{21}ON_3 + H_2SO_4 + 3 H_2O$	178	186	1,5502— 1,5611		Ab 40° entweicht Wasser unt. Trübung. Schmelze bald braun. Mit Salophen n = 1,5611 bei 173—176°, 1,5700 bei 144—147°. In Paraffin Dampfblasen ab 110° und Trübung einzelner Kristalle, andere bleiben klar.
200—208	p-Nitrobenzhydrazid 	166	164	1,5502— 1,5611		Ab 140° Blättchen. Schmilzt unter Bräunung und Gasblasen. Bei 200° sublimieren Nadeln mit rautenförmigem Querschnitt (bei stehenden Nadeln). α (längs) etwa 1,48, β unter, γ über 1,74.
200—208	5-Butylbarbitursäure 	164	—155—	1,4584— 1,4683		Ab 130° Blättchen, Spieße. Gleichgew.: Prismen, Platten. Schmelze hellgelb. Erstarrt sphärolithisch. Dimorph.
202—208	Anilinessigsäure-o-carbonsäure  $C_7H_7O_4N$	171 tr.	149 tr.	Zersetzung		Gelblich. Ab 180° Körner. Schmilzt unter Zersetzung und Gasblasen. Aus heißer alkoholischer Lösung kleine rundliche, durch vier Kerben kreuzartige Scheiben, fast isotrop. n = zwischen 1,59 und 1,62 (starke Vergr.).
203—208	Adrenalin (= Suprarenin) 	163	180	Zersetzung		Ab 180° Bräunung. Zersetzung. Mit Chlorhydrat Verbindung F. 160°. Ursubstanz kleine, gestreckte sechseckige Blättchen, oft schuppig überlagert. α' längs, α' zwischen 1,55 und 1,57, γ' wenig unter 1,62 (starke Vergr.); pinselartige Aggregate. Wirkt pupillen-erweiternd.
204—208	Aspidospermin $C_{17}H_{21}O_4N$	165	203	1,5101— 1,5204		Gleichgew. träg: Stengel, Spieße, sinkt ab. Schmelze gelb. Mit Salophen n = 1,5502

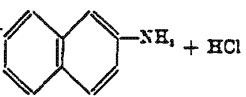
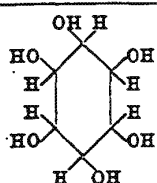
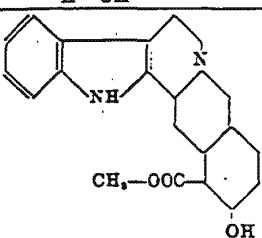
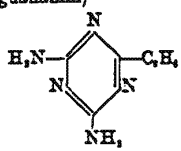
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
205—208	Chinintartrat $2 C_{10}H_{11}O_2N_2 + C_6H_8O_4 + 2 H_2O$	175	168	1,5502—1,5611		Schmelze bald braun. In Paraffin Beginn der Zersetzung ab 180° unter Gasblasen und langsamem Schmelzen. Aus Alkohol Stengel mit schiefer Auslöschung. γ' längs wenig unter 1,60, α' wenig über 1,59.
204—208	β -Resorcyllsäure (= 2,4-Dioxybenzoesäure) $+ 3 H_2O$ 	180	151	Zersetzung		Zwischen 70—100° entweicht Wasser und Trübung. Ab 120° Körner. Schmilzt unt. Gasblasenbgl. Aus Wasser polare Prismen und Rhomboide des Hydrates. In Paraffin ab 90° allmähliche Umlagerung unter Dampfblasen. Bei 130° aufgelegt (in Paraffin) rapide Umlagerung unter Blasen, seltener homogener F. 140—145°.
208	N,N-Diäthyl-N'-acetylarnstoff $(C_2H_5)_2N-CO-NH-CO-CH_3$	175	189	<1,4339		Ab 90° reichlich Nadeln, die sich verfilzen. Gleichgew.: Nadeln, Spieße. Erstarrt sphärolithisch. Polymorph.
208	9,10-Dihydronaphthacen 	174 tr.	197	1,8011 1,6897	208—210 220—223 13	Ab 140° Nadeln, Prismen. Gleichgew.: Balken.
208	Borneol (= Borneocampher) 	154 (136)	—	<1,4339		Campherartiger Geruch. Ab 40° sechsstrahlige Blättchen, Spieße. F. wegen Flüchtigkeit schwierig (großes Deckglas!). Eutektische Temperatur mit Salophen im Film bestimmen (instabiles Sal. 130°!). Brechungsindex in Silikonkammer. Ab 140° sublimieren isotrope Sterne mit n zwischen 1,51 und 1,53).
208	2,3-Benzdiphenylenoxyd (= 2,3,5,6-Dibenzocumaron)  $C_{16}H_{10}O$	172	207	1,6353	225—228	Ab 130° Blättchen, später Sicheln, Spieße, gelpappte Platten. Gleichgew.: rundliche Scheiben, Platten. Erstarrt zu gemaserten Platten. Mit Pikrinsäure gelbe Verbindung (Kontaktpräparat!).
209	Phthalanil 	164	200	1,5502 1,5403	216—218 241—243 25	Ab 120° Körner. Gleichgew.: Körner. Polymorph II 204°, III 175° (30).
190—210	2,4,6-Trinitrobenzoesäure 	143	135	1,5101—1,5204		Gelblich. Ab 160° Tröpfchen. Schmilzt unter Gasblasen. Schmelze bald braun.
Verfl. 190—210 (255)	Camphersäureimid 	145	166	<1,4339		Ab 60° Körner, sechsstrahlige Sterne; dichte Sublimate, später Straßenspflaster. Unter großem Deckglas oder in Silikonkammer F. 255°. F. der Sublimate nicht immer gleich. Gleichgew.: rundliche, gelpappte isotrope Kristalle. Erstarrte Schmelze und Sublimate unter 94° anisotrop.
200—210	Traubensäure (= dl-Weinsäure) $HOOC-CH(OH)-CH(OH)-COOH$	181	135	1,5000	65—70	Ab 180° Tröpfchen. Schmilzt trüg. Gasblasen. Erstarrt glasig. Aus Wasser kristallisieren Hydratstengel, die in Paraffin bei 78° inhomogen schmelzen unter Abscheidung wasserfreier Prismen. Für Brechungsindex rasch mit

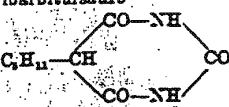
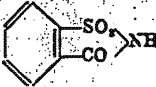
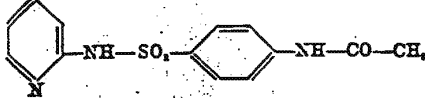
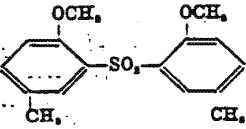
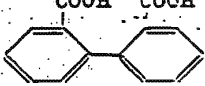
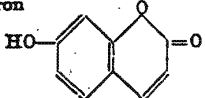
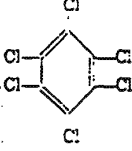
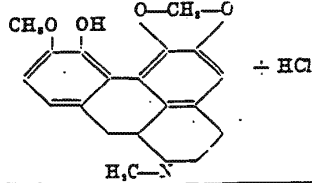
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
210	Oxamidsäure $\text{H}_2\text{N}-\text{CO}-\text{COOH}$	180	141	Zersetzung		Ab 140° Blättchen, Körner, Stäbchen. Schmilzt unter heftiger Gasblasenbildung und Gelbfärbung. Nach ein bis zwei Minuten Abscheidung von Stäbchen. Bei 120° sublimieren kleine Körner und ausgefranzte Spindeln; letztere längs $\gamma' = 1,59$ (starke Vergrößerung!)
210	5-Benzyl-5-propylbarbitursäure 	169	188 tr.	1,4683	228	Ab 140° Stäbchen. Gleichgew.: Stengel, Körner. Erstarrt zu Sphärolithen.
210—211	Sarkosin (= Methylaminoessigsäure) $\text{CH}_3-\text{NH}-\text{CH}_2-\text{COOH}$	178	136	1,4842	155—160	Ab 150° schöne achtförmige Körner, ähnlich Doppelpyramiden. Schmilzt unter starker Gasblasenbildung und leichter Braunfärbung. Für Brechungsindex nach Durchschmelzen rasch mit Metallwürfeln abkühlen.
210—211	Phenanthrenchinon 	159	182	1,6353—1,6231		Orange. Ab 125° Stengel und Sechsecke. Blättchen schmelzen teilweise bei 207°. Gleichgew.: Balken, Spieße, Blättchen. Dimorph II 207° (10).
210—212	Dicyandiamid $\text{H}_2\text{N}-\text{C}(=\text{NH})-\text{NH}-\text{CN}$	175	210	1,5502	194—196	Ab 175° Nadeln, Prismen, Blättchen. Gleichgew.: Prismen, Körner, sinkt ab.
200—212	Hordeninsulfat $2 [\text{HO}-\text{C}_6\text{H}_4-\text{CH}_2-\text{CH}_2-\text{N}(\text{CH}_3)_2] + \text{H}_2\text{SO}_4 + \text{H}_2\text{O}$	177	159	1,5204	212—216	Schmilzt träg, einzelne Gasblasen. Hydratkristalle schmelzen in Paraffin bei 130°—135° homogen, zum Teil inhomogen.
212	2,3-Benzofluoren 	175	205	1,6353	211—212	Ab 100° Körner, Blättchen, Nadeln; sublimiert stark. Gleichgew.: Körner, Blättchen. Dimorph. Leicht überhitzte Schmelze erstarrt flächenhaft mit sofortiger Umwandlung.
212	1,1,2,2-Tetraphenyläthan 	176	206	1,5403 1,5299	218—220 237—240 2.	Ab 180° starke Sublimation von Nadeln. Gleichgew.: Stengel.
212	Coniinhydrobromid  $\text{CH}_2-\text{CH}_2-\text{CH}_3 + \text{HBr}$	154	134	1,4683	221	Geruch. Ab 180° Nadeln. Gleichgew.: Stengel.
210—213	Kaliumxanthogenat $\text{C}_2\text{H}_5-\text{O}-\text{CS}-\text{SK}$	144 tr.	132	Zersetzung		Farblos oder hellgelb. Ab etwa 160° Kristalle dunkel. Schmilzt unter Gasblasen und Knoblauchgeruch. Schmelze hellbraun. In Wasser leicht löslich. Daraus meist gebündelte Stengel mit

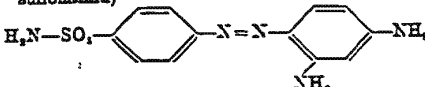
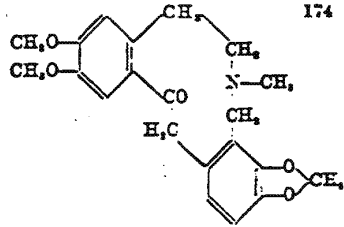
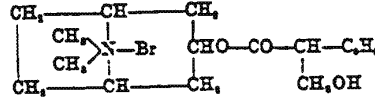
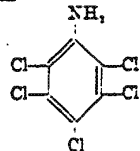
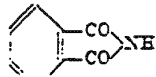
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
213	5-Isopropylbarbitursäure 	173	175	1,5484	225—230	Ab 130° Blättchen, Stäbchen. Gleichgew.: Stengel, Rauten. Erstarrt sphärolithisch. Brechungsindex durch Zersetzung erschwert.
213	Atophan (= 2-Phenylchinolin-4-carbonsäure) 	171	175	1,6353 1,6231	218—219 242—244 24	Ab 165° Nadeln, Balken, einzeln und in Büscheln. Gleichgew.: Balken in grün-gelber Schmelze. Polymorph II 213°, III 196° (87).
207—214	Adermin-hydrochlorid (= Vitamin B ₆) 	183	151	1,5897— 1,6011		Ab 160° Tröpfchen, aus denen Blättchen und gebogene Nadeln wachsen. Schmilzt unter Gasblasenbildung und Braunfärbung. Brechungsindex erschwert.
214	p-Oxybenzoesäure 	156	154	1,5299	220—223	Ab 130° Körner, Prismen. Gleichgew.: Körner, Spieße. Schmelze erstarrt glasig.
214	Guanidinnitrat $H_2N-C(=NH)-NH_2 + HNO_3$	183	152	1,4683— 1,4584		Beim Schmelzen Gasblasen. Gleichgew. trüg: Körner. Erstarrt zu grobem Mo-saik mit zum Teil isotropen Kristallen, optisch einachsigt, negativ.
215	1,3,8-Trinitronaphthalin 	163	197	1,6011 1,5897	216 239 23	Ab 200° kurze Prismen. Gleichgew.: Prismen, Balken, Stengel. Schmelze gelb. Erstarrt glasig.
180—215	Chininbihydrochlorid $C_{20}H_{28}O_4N_2 + 2 HCl + 2 H_2O$	152 tr.	122 tr.	Zersetzung		Erweicht langsam. Schmelze braun. Erstarrt glasig. Mit Salophen n = 1,6010 bei 103—105°, 1,5898 bei 146° bis 149°. Wässrige Lösung gibt nur glasi-gen Rückstand. Aus Alkohol (zwischen Objektträger und Deckglas) Nadeln mit a' längs (etwa 1,55); sie zerfließen bald nach Absaugen des Alkohols.
190—215	3,4-Dioxyzimtsäure (= Kaffeesäure) 	168	159	Zersetzung		Gelb. Ab 175° Tröpfchen. Schmilzt unter Gasblasen und Braunfärbung. In Paraffin ab 160° Gasblasen in Kristallen. Aus Alkohol kleine, ausgefrante Pri-smen (Lösungsmitteladdukt, in Paraffin Umlagerung ab 70°) und kleine, breite Linsen und Rosetten, schiefl auslöschend. γ' über 1,74.
205—215	Emetindihydrochlorid $C_{20}H_{24}O_4N_2 + 2 HCl + 6 H_2O$	152	130	Zersetzung		Erweicht sehr langsam zu zäher gelber Schmelze. Erstarrt glasig. Bei 150° auf-gelegt, teilweises Schmelzen d. Hydrates. Mit Salophen n = 1,5700 bei 152—155°, 1,5611 bei 181—184°. In Paraffin schmilzt das Hydrat trüg bei 80—90° zu zäher Masse.
206—215	p-Cumarsäure (= trans 4-Oxyzimtsäure) 	165	152	1,5700— 1,5795		Ab 160° Prismen. In der Schmelze Gas-entwicklung. Aus Alkohol 2 Modifikatio-nen: Sphärolithe und Stengel II, bei Erwärmen Umwandlung, Rauten und rechteckige Prismen I, letztere (meist

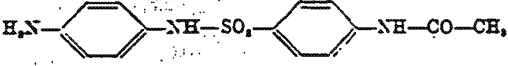
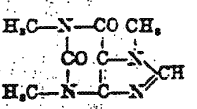
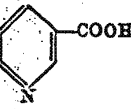
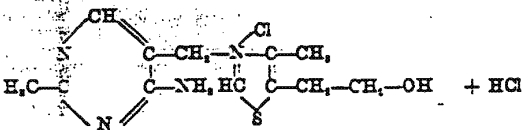
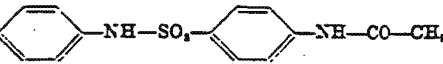
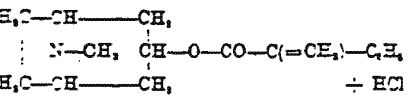
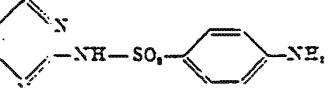
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyan-diamid	Glas-pulver	Temp. °C	Besondere Kennzeichen
215	Isoborneol $\begin{array}{c} \text{H}_3\text{C}-\text{C}(\text{CH}_3)-\text{CH}-\text{OH} \\ \quad \\ \text{C}(\text{CH}_3)_2 \\ \quad \\ \text{H}_3\text{C}-\text{CH}-\text{CH}_3 \end{array}$	-146— (125)	-185—			Camphergeruch. Ab 40° isotrope Sterne, Sechsecke, später wabenartig. Eutekt. Temperatur im Film bestimmen, (mit instab. Salophen 125°). Brechungsindex: der isotropen Kristalle etwa 1,51 (in Zedernöl löslich).
215	Diacetyl-tetrabrombrenzcatechin $\begin{array}{c} \text{Br} \quad \text{OOC}-\text{CH}_3 \\ \quad \\ \text{C}_6\text{H}_2 \\ \quad \\ \text{Br} \quad \text{Br} \end{array}$	169	148	1,5403	237	Ab 180° Blättchen, Rauten, Körner. Gleichgew.: Prismen. Erstarrt sphärolithisch. Eventuell umkristallisieren aus Chloroform + Alkohol mit Kohle.
215	Anthracen 	175	205	1,6231 1,6126	218—220 240—242	Ab 100° verschiedene Sublimat, später Straßenpflaster. Mit Pikrinsäure rote Verbindung, (sublimiert in Stengeln bei 130°). Brechungsindex mit großem Deckglas oder in Silikonkammer.
206—216	Milchzucker (= Lactose) $\begin{array}{c} \text{HO}-\text{CH}_2-\text{CH}-[\text{CHOH}]_4-\text{CH} \\ \quad \quad \\ \text{O} \quad \text{O} \quad \text{O} \\ \quad \quad \\ \text{HO}-\text{CH}_2-\text{CH}-\text{CH}-[\text{CHOH}]_4-\text{CH}-\text{OH} \end{array}$	187	163	1,5000— 1,5101		Schmilzt unter Gelbfärbung und Gasblasen. Erstarrt glasig.
193—216	Spasminhydrochlorid (= Dibenzylsuccinat + HCl) $\text{C}_6\text{H}_5-\text{CH}_2-\text{OOC}-\text{CH}_2-\text{CH}_2-\text{COO}-\text{CH}_2-\text{C}_6\text{H}_5 + \text{HCl}$	168	162	Zersetzung		Ab 180° Tröpfchen, schmilzt trög. Bräunung. Mit Salophen n = 1,6128 bei 93—95°, 1,6011 bei 127—129°. Aus Alkohol vielflächige Körner und Prismen. In Nitrobenzol erhitzt bei Abkühlen sechseckige Prismen mit a' Länge nahe 1,55, (γ' über 1,74).
216	N'-Äthylsulfanilamid $\text{C}_2\text{H}_5-\text{NH}-\text{C}_6\text{H}_4-\text{SO}_2-\text{NH}_2$	179	174	1,5502	208—208	Ab etwa 205° Körner, Stäbchen. Gleichgew.: rechtwinkelige Stengel und Prismen. Schmelze kristallisiert fast glasig. Bei etwa 180° Umwandlung mit F. 198°; nach neuer Umwandlung 206°. Selten erneute Umwandlung (Reimers) (67).
215—217	Homatropinhydrobromid $\begin{array}{c} \text{H}_3\text{C}-\text{CH}-\text{CH}_3 \\ \quad \\ \text{N}-\text{CH}_2 \quad \text{CH}-\text{O}-\text{CO}-\text{CH}(\text{OH})-\text{C}_2\text{H}_5 \\ \quad \\ \text{H}_3\text{C}-\text{CH}-\text{CH}_3 \end{array} + \text{HBr}$	172	147	1,5502— 1,5403		Ab 200° Tröpfchen. Schmilzt unter leichter Braunfärbung. Erstarrt glasig. Mit Salophen n = 1,5502 bei 150—151°, 1,5403 bei 178—180°.
217	Eldoral (= 5-Piperidino-5-äthylbarbitursäure) 	156	189	1,4842	204—205	Ab 140° Stengel, Prismen, Körner. Gleichgew.: Körner. Dimorph II 210° (siehe Übungsbeispiele).
208—218	Cinchoninhydrochlorid $\text{C}_{20}\text{H}_{21}\text{ON}_3 + \text{HCl} + 2 \text{H}_2\text{O}$	132	119	1,5700— 1,5611		Ab 50° entweicht Wasser. Teilweises Schmelzen bei 160—170°. Ab 170° an Kristallen kleine stengelartige Auswüchse. Reichlich Tröpfchen. Schmelze gelbbraun. In Paraffin ab 120° Dampfblasen und Trübung oder Auffaserung.
215—218	Coniinhydrochlorid 	155	122	1,4584	179—182	Ab 130° feine Nadeln pinsel- und sternartig, später verfilzt. Gleichgew.: Prismen, Balken. Braunfärbung.

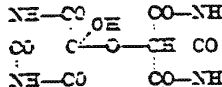
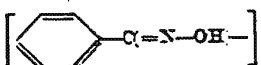
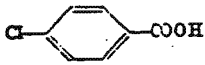
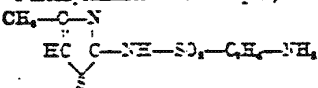
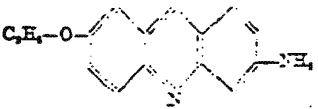
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyan- diamid	Glas- pulver	Temp. °C	Besondere Kennzeichen
220	Pentachlortoluol 	177	181	1,5204— 1,5299		Ab 70° Nadeln, später Straßenpflaster. Ab 170° starke Verflüchtigung. Schmilzt unt. Gasblasenbgl. Brechungsindex in Silikonkammer. Bei 170° sublimieren lange Stengel. Anflachen abgeschragten Stengeln α' (Länge) etwa 1,55 (löslich), γ' 1,74.
220	1-Acetylaminoanthrachinon 	176 tr.	201 tr.	1,6128— 1,6231		Gelb. Ab 150° Nadeln. Gleichgew.: Nadeln. Balken. Erstarrt sphärolithisch. Bei Rückstand umkristallisieren oder umsublimieren bei 210—215° Sublimiert in zugespitzten Nadeln oder „zerknitterten“ Bändern, quer γ' größer 1,74, α' 1,56—1,57.
215—221	Piperinsäure 	172	169	1,6011— 1,6128		Gelblich. Geruch. Ab 130° Stäbchen, Körner. Sublimiert dicht. Schmilzt trög, Braunfärbung und Gasblasen.
Verflücht. über 220 (265)	p-Brombenzoesäure 	175 tr.	184	1,4938	275—278	Ab 80° Nadeln, gebogene Blättchen. F. und Brechungsindex nur unter großem Deckglas oder in Silikonkammer. Gleichgew.: Platten, Stengel.
220—222	o-Phthalsäure 	171	145	1,4842— 1,4883		Ab 160° schöne Körner und Prismen. Schmilzt mit heftiger Gasblasenbgl. Bei Wiederholung sinkt F. wegen Umwandlung in Anhydrid. Brechungsindex nur unter großem Deckglas oder in Silikonkammer (im geschlossenen Rohr F. 191°). Aus Wasser am Objektträger Rauten (Kantenwinkel 93°). Auslöschung fast symmetrisch. γ' > 1,74, α' etwa 1,46. In Anilin löslich unter Abscheidung von Nadeln und Linsen.
222	3-Oxy-2-naphthoesäure 	160	153	1,5795— 1,5700		Gelb. Scharfer Geruch. Ab 175° Körner. Gleichgew.: Körner. Erstarrt sphärolithisch. Dimorph.
224	5-Sulfosalicylsäure 	126 (+H ₂ O: 100)	160 (+H ₂ O: 100)	1,5403— 1,5502		Zwischen 100—110° teilweises Schmelzen des Hydrats und Wiedererstarren. Bei 160° neuerliches Schmelzen und sofortige Wiederkristallisation. Bei Schmelzen Gasblasen. In Paraffin homogenes Schmelzen bei 105—110°. Für Brechungsindex bei 120° trocknen Brechungsindex gleich nach Schmelzen < 1,5403, nach zwei Minuten > 1,5502 steigt weiter.
220—224	Conteben 	187	193	1,6598— 1,6715		Schmilzt unter Gasblasenbildung und Braunfärbung.

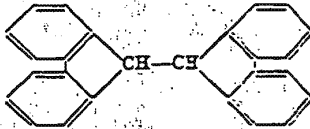
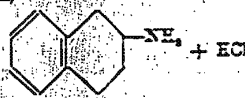
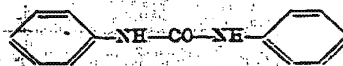
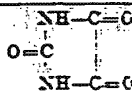
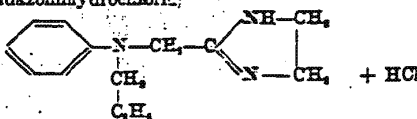
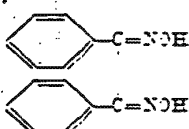
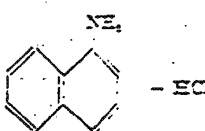
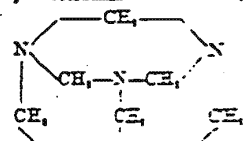
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
224	Diäthylmalonamid $\begin{array}{c} \text{C}_2\text{H}_5 \\ \diagdown \\ \text{C} \\ \diagup \\ \text{C}_2\text{H}_5 \end{array} \begin{array}{l} \text{CO-NH}_2 \\ \text{CO-NH}_2 \end{array}$	181	177	1,4339— 1,4584		Ab 120° Nadeln. Körner. Starke Sublimation. Gleichgew.: Körner, Balken. Erstarrt sphärolithisch. Brechungsindexbestimmung wegen Flüchtigkeit erschwert.
213—225 Verflücht. (250— 258)	β-Naphthylaminhydrochlorid 	174	143	Zersetzung		Ab 90° Sechsecke, sehr starke Sublimation. Über 200° Umwandlung in Rosetten und Stengel (sehr charakteristisch). Enantiotroper Up. 188°. Schmelze düsterrötlich. Schmilzt unter Zersetzung mit Ausscheidung anisotroper Quadrate. Unter großem Deckglas oder in Silikonkammer F. 250—258°.
222—225	Meso-inosit 	179	173	1,5204	218—221	Ab 140° Körner, Prismen. Schmilzt unter Verfärbung und Gasblasen. Brechungsindex wegen vieler kleiner Blasen erschwert.
215—225	Yohimbin 	175	198	1,5611— 1,5700		Schmilzt unter Zersetzung. Schmelze orange. Mit Salophen n = 1,5700 bei 139—142°, 1,5611 bei 170—173°. Gleich nach Schmelzen Brechungsindex <1,5611, nach einer Minute >1,5611.
215—225	Schleimsäure $\begin{array}{ccccccc} \text{OHH} & \text{H} & \text{OH} & & & & \\ & & & & & & \\ \text{HOOC}-\text{C}-\text{C}-\text{C}-\text{C}-\text{COOH} \\ & & & & & & \\ \text{H} & \text{OH} & \text{OH} & \text{H} & & & \end{array}$	192	171	1,4842— 1,4936		Schmilzt trög unter Zersetzung und Gasblasen. Brechungsindex wegen heftiger Gasblasenbildg. erschwert. Gleich nach dem Schmelzen <1,4842, steigt rasch an.
225	2,4-Diamino-6-phenyl-1,3,5-triazin (= Benzoguanamin) 	175	202	Zersetzung		Ab 150° Körner, Rechtecke, Prismen. Gleichgew.: Rechtecke, Prismen. Bei 210° sublimieren kleine rechtwinkelige Prismen. α' etwa 1,66, γ > 1,74. Wenn in Schmelze Rückstände, umsublimieren bei 210°.
226	Rutonal (= 5-Methyl-5-phenylbarbitursäure) $\begin{array}{c} \text{C}_6\text{H}_5 \\ \diagdown \\ \text{C} \\ \diagup \\ \text{CH}_3 \end{array} \begin{array}{l} \text{CO-NH} \\ \text{CO-NH} \end{array} \text{CO}$	175	183	1,5204 1,5101	234 258—259 25	Ab 150° Nadeln, Stengel, Blättchen. Gleichgew.: Stengel, Spieße, Vierecke. Erstarrt sphärolithisch. Dimorph II 200° (90).
224—226	Novalgin (= 1-Phenyl-2,3-dimethylpyrazolon-4-methylaminomethansulfonsaures Natrium) $\begin{array}{c} \text{CH}_3-\text{C}=\text{C}-\text{N}(\text{CH}_3)-\text{CH}_2-\text{SO}_3\text{Na} \\ \\ \text{CH}_3-\text{N}-\text{N}(\text{C}_6\text{H}_5)-\text{CO} \end{array} + \text{H}_2\text{O}$	160	140 tr.	1,5403— 1,5502		Ab 100° entweicht Wasser. unter Trübung. Schmilzt unter Gasblasenbildg. und Braunfärbung. Nach Schmelzen Brechungsindex > 1,5403, nach einer Minute <1,5403. In Paraffin Dampfblasen ab 150°. In Wasser leicht löslich, aus übersättigter Lösung flächige Kristalle oder Sphärolithe.
225—227	Ergotin $\text{C}_{20}\text{H}_{20}\text{O}_4\text{N}_2$	180	195	Zersetzung		Schmelze dunkelbraun. Aus Aceton oder Alkohol lange Stengel (rhombisch) mit c längs. c = 1,575, β (quer) = 1,590

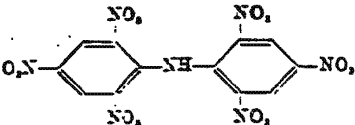
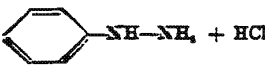
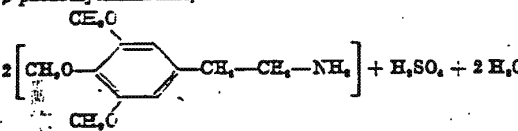
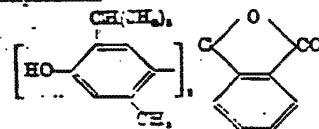
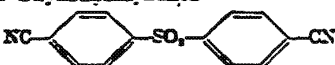
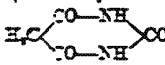
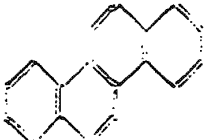
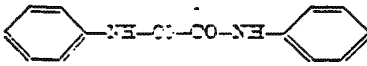
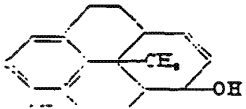
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyandiamid	Glaspulver	Temp. °C	Besondere Kennzeichen
227	Adipinsäureamid $\text{NH}_2\text{—CO—(CH}_2)_4\text{—CO—NH}_2$	183	171	1,4339— 1,4584		Ab 180° Nadeln, Stengel. Gleichgew.: Prismen, sinkt ab. Erstarrt zu strahligen Aggregaten. In Alkohol kaum löslich (Stäbchen, Körner).
226—228	5-Amylbarbitursäure 	176	179	1,4339— 1,4584		Ab 140° Blättchen, anfangs Rauten, später verzerrt. Gleichgew.: Platten; flüchtig. Erstarrt zu Platten. Aus Alkohol am Objektträger Sechsecke und Rosetten.
228	Saccharin (= o-Benzoesäuresulfonid) 	165	161	1,5204 1,5101	244 275 31	Ab 170° Sechsecke, Rauten, Prismen. Später Straßenpflaster. Gleichgew.: Sechsecke, Stengel.
228	2-[N'-Acetyl-sulfanilylamino]-pyridin 	183	186	1,5795 1,5700	216—217 241—242 25	Ab etwa 215° kleine schiefwinklige Kristalle. Erstarrt glasig. Kristallisiert langsam bei Erwärmen, F. 212°, dann Umwandlung und F. 225° (Zeimers) (67).
228	2,2'-Dimethoxy-5,5'-dimethyl-diphenyl-sulfon 	176	204	1,5101 1,5000	219—221 240—241 21	Ab 150° Blättchen, Körner. Gleichgew.: Prismen, Körner.
228	Diphenylsäure 	167	145	1,5502 1,5403	217—219 244—246 27	Ab 180° Blättchen, Körner. Gleichgew.: rechteckige Blättchen. Polymorph II 224°, III 212° (32).
228	Umbelliferon 	160	180	1,5997— 1,6126		Häufig rosa verfärbt. Umsublimieren bei 220°. Ab 160° Nadeln, Blättchen. Bei 175° teilweises Schmelzen. Gleichgew.: Platten, Nadeln. Brechungsindex auch mit großem Deckglas oder in Silikonkammer schwierig.
228	Hexachlorbenzol 	179	Verfl.	1,5299	250—255	Geruch. Ab 80° Nadeln, Stäbchen. Dichte Sublimation, später Straßenpflaster. Schmilzt unter rascher Verflüchtigung. Brechungsindex auch mit großem Deckglas oder Silikonkammer schwierig. Bei 160° sublimieren Stengel, oft stark gekrümmt, α' (längs) etwa 1,55, γ' > 1,74.
230	Bulbocapninhydrochlorid 	177	173	Zersetzung		Ab 200° Prismen, ab 230° schmelzen einige Kristalle zu braunen Tropfen. Allmähliche Zersetzung und Verkohlung. Kristallreste bis über 300°. Aus Alkohol Nadeln, längs α' (= β) etwas über 1,66, γ' > 1,74.
225—229	Allantoin $\text{NH}_2\text{—CO—NH—CH—NH}_2$ CO	169	157	Zersetzung		Ab 200° Trübung, ab 225° Kochen in Kristallen, Bräunung. Aus heißer Wass. Lösung Rechtecke. α' 1,59—1,62.

F.°C	Substanz	Erstarrt Salophen	Temp. mit Dextran-diazid	Glas-pulver	Temp. °C	Besondere Kennzeichen
225—230	Homatropinhydrochlorid $C_{10}H_{11}O_2N + HCl$	169	127	1,5203— 1,5101		Ab 170° Blättchen, sphärolithisch. Gleichgew. trüg: Sechsecke und Leisten. Erstarrt glasig. Mit Salophen $n = 1,5502$ bei 160—161°, 1,5403 bei 178—180°.
223—230	Prontosil (= 2,4-Diaminoazobenzol-4'-sulfonamid) 	180	193	Zersetzung		Rot. Ab 215° nimmt die Durchsichtigkeit zu. Schmilzt unscharf. Gleichgew.: sehr trüg; schiefwinkelige Platten. Schmelze rotbraun. Gasblasen. Aus Aceton rote Rechtecke I, gelbe dendritische Aggregate II.
215—230	Kryptopin 	174	202	1,5403	219—221	Ab 195° kurze Prismen. Gleichgew. wegen Zersetzung nur kurz einstellbar. Mit Salophen $n = 1,5611$ bei 164—166°, 1,5502 bei 192—194°.
226—230	Atropinbrommethyleat 	166	125	1,5403— 1,5502		Erstarrt glasig. Mit Salophen $n = 1,5700$ bei 118—120°, 1,5611 bei 152—154°.
220—230	d-Arginin $H_2N-C(=NH)-NH-(CH_2)_3-CH(NH_2)-COOH$	164	167	Zersetzung		Zwischen 80° und 90° entweicht Wasser, schmilzt unter reichlichen Gasblasen und Braunfärbung. In Paraffin inhomogenes Schmelzen bei 101° (wenig Schmelze).
220—230	Semioxamazid $H_2N-CO-CO-NH-NH_2$	186	194	1,5101— 1,5204		Ab 160° Nadeln, Nadelbüschel. Dichte Sublimata. Schmilzt mit heftiger Gasblasenbgl. Brechungsindex sehr schwer bestimmbar, weil rasch Abscheidung reichlicher krümeliger Massen. Bei 200° sublimieren Stengel und lange Rhomboide mit α' etwa 1,48, γ' über 1,74.
232	Pentachloranilin 	166 tr.	208	1,5502	246—244	Ab 80° Nadeln, ab 170° Straßenpflaster. Verflüchtigt. Schmelze erstarrt zu dünnen Platten. Brechungsindexbestimmung mit großem Deckglas oder Silikonkammer bei sinkender Temperatur (schwierig). Bei 210° sublimieren lange Stengel; an flachen, schräg begrenzten Stengeln schiefe Auslöschung (5°); α' zwischen 1,53 und 1,55, $\gamma' > 1,74$.
234	Dehydrocholeinsäure (= 3,7,10-Trikochoholsäure) $C_{26}H_{44}O_4$	170	174	1,4842— 1,4936		Ab 190° Tröpfchen. Gleichgew.: Nadeln, sinkt ab. Ursubstanz flache Stengel mit α' zwischen 1,53 und 1,57, γ' zwischen 1,55 und 1,57.
200—235	Hydrastininhydrochlorid $C_{11}H_{11}O_2N + HCl$	128	88	Zersetzung		Schmilzt unter Braunfärbung. Mit Salophen $n = 1,5794$ bei 122°—124°, 1,5700 bei 150°—152°.
235	Phthalimid 	170	182 tr.	1,5204	242—244	Ab 110° Körner, Prismen, Platten. Starke Sublimation. Brechungsindex nur in Silikonkammer möglich. Bei 150° sublimieren Rechtecke mit α' etwa 1,53

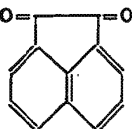
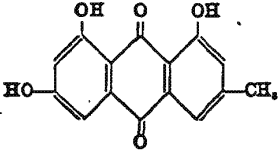
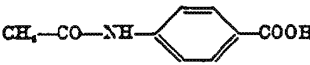
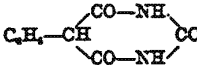
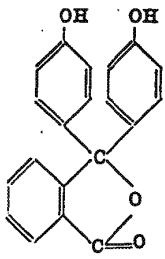
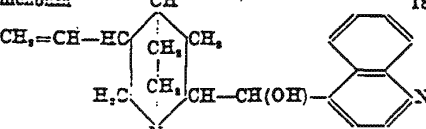
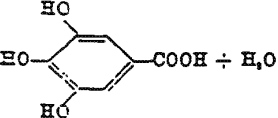
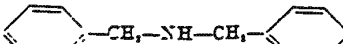
F. °C	Substanz	Eutekt. Temp. mit Salophen	Temp. mit Dicyan- diamid	Glas- pulver	Temp. °C	Besondere Kennzeichen
235	N-[4-Acetaminobenzolsulfonyl]-p-phenylendiamin 	184	191	1,5700—1,5811		Ab 180° wenig Nadeln, Spieße. Gleichgew.: Stengel, Prismen. Schmelze violett.
220—235	Koefeinphosphat $C_{10}H_{14}O_8N + H_3PO_4$	186	144	1,5811—1,5502		Schmilzt unter Gelbfärbung und Gasblasenbg. Mit Salophen $n=1,5811$ bei 168—170°, 1,5502 bei 201—203°.
236	Coffein 	163	—173—	1,4936	~~263—264~~	Ab 95° Nadeln, Spieße, Balken; später Straßenpflaster. Gleichgew.: Körner, Stengel.
237	Nicotinsäure (= Pyridin-β-carbonsäure) 	180 tr.	—150—	1,4936	240—241	Ab 115° Blättchen, Nadeln, Stengel. Starke Sublimation. Gleichgew.: zugespitzte Platten (großes Deckglas).
232—237	Ascorbin (= Vitamin B ₁)-hydrochlorid 	184	142	Zersetzung		Geruch. Ab 225—230° braune Verfärbung. Schmilzt langsam unt. Gasblasenbg. Schmelze braun. Leicht löslich in Wasser; aus verdünntem Alkohol lange Nadeln, schiefe Auslöschung 18—20°, γ' längs.
237	3-[4-Acetaminobenzolsulfamino]-benzonitril CN 	180	195	1,5611—1,5502	130 155—156 26	Gleichgew.: Prismen, Körner. Erstarrt glasig, bei Erwärmen sphärolithisch oder fächerartig.
238	Dimethylglyoxim $H_3C-C=N-OH$ $H_3C-C=N-OH$	179	187	<1,4339		Ab 80° Nadeln, Stengel, ab 230° rasche Verflüchtigung. Für F. großes Deckglas. Schmilzt unter Zersetzung und Braunfärbung. Aus Alkohol Rhomboide und geriefte Körner, zuletzt Nadeln. In geriefen Körnern α' kleiner 1,44, γ' (längs der Riefen) > 1,74. Gefärbte Metallkomplexe, Ni-Salz rot.
238—240	Apoatropinhydrochlorid 	187	147	1,5403—1,5299		Ab 220° Spieße, Stengel. Schmelze gelb bis braun, erstarrt glasig. Mit Salophen $n=1,5611$ bei 146—152°, 1,5502 bei 176—179°.
238—240	Sulfamethyldiazin (= 2-Sulfamethylamino-4-methylpyrimidin = Sulfamerazin = Sepacid) CH ₃ 	174	179	1,5011—1,5502		Ab etwa 100° Umwandlung. Teilweises Schmelzen bei 208°. Schmilzt unscharf bei 236—241°. Gleichgew.: Rauten. Erstarrt glasig (Reimers) (67).

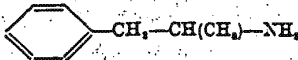
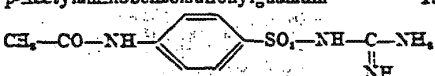
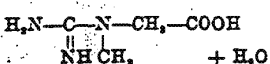
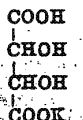
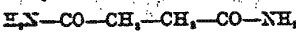
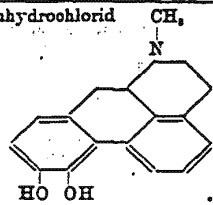
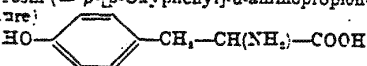
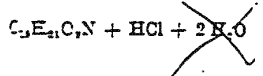
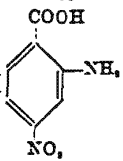
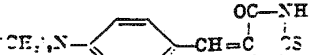
F. °C	Substanz	Eutekt. Temp. mit Salophen	Dicyan- diamid	Phenol- phtalein	Besondere Kennzeichen
241	p-Nitrobenzoesäure 	158	155 tr.	222	Ab 120° Nadeln, Blättchen, Stengel. Dichte Sublimation. Gleichgew.: Blättchen, Stengel. Dimorph (59). Brechungsindex der Schmelze zwischen 1,6000 und 1,4937.
235—242	Alloxantin 	187	204	190	Oft rötlich verfärbt. Ab 140° entweicht Wasser. Ab 200° Körner, Rhomboide. Schmilzt unter Gasblasen und Braunfärbung. In Paraffin ab 110° Dampfblasen. Bei 128—130° teilweise inhomogenes Schmelzen unter Braunfärbung. Bei 180° sublimieren kleine Rechtecke und Rhomboide. α' in Rechtecken zwischen 1,65 und 1,56, γ' in Rhomboiden > 1,66. In Anilin leicht löslich.
242	α-Benzoldioxim 	178	188	239	Ab 220° Nadeln, ausgezackte Stengel. Schmilzt unter Zersetzung und Gasblasen. Schmelze braun. Bei 230° sublimieren ausgezackte, schwach anisotrope Stengel, schiefe Auslöschung. γ' längs, α' wenig über 1,66.
243	p-Chlorbenzoesäure 	173	193 (178)	225	Ab 70° Nadeln, Prismen, Blättchen. Später Straßenpflaster. Ab 220° stark flüchtig. Gleichgew.: Rauten. Braunfärbung und Zersetzung. Bei 200° sublimieren Rhomboide mit schiefer Auslöschung 28°. α' etwa 1,44, γ > 1,66.
240—244	Hydrazinsulfat: $\text{NH}_2\text{--NH}_2 + \text{H}_2\text{SO}_4$	100	139	220	Ab 220° zerspringen einzelne Kristalle. Schmilzt unter Braunfärbung und Gasblasen. In Paraffin ab 240° reichlich Gasblasen in den Kristallen. Aus heißer Wasserlösung rechtwinkelige und sechseckige Prismen mit gerader Auslöschung, dünne Sechsecke mit schiefer Auslöschung. Durch Anilin zersetzt und verfärbt. In o-Toluidin Zersetzung und Tropfenbildung. Alle Brechungsindices unter 1,62.
220—244	Brucininsäure $\text{C}_{22}\text{H}_{20}\text{O}_4\text{N}_4 + \text{HNO}_3$	157	130	192	Bitter. Ab 185° reichlich Tröpfchen, ab 200° kleine nadelige Auswüchse an der Substanz. Schmelze dunkelrotbraun. Gasblasen. Aus Wasser kurze, aus Alkohol mittelgroße Prismen, die sich bald trüben. In Paraffin ab 80° Gasblasen. Einzelne Kristalle erst Umlagerung ab 130°, Prismen aus Wasser α' zwischen 1,51—1,53, γ' > 1,66.
244	Sulfamethylisobazol (= 2-Sulfamethylamino-4-methylisobazol = Ultraseptil) 	181	185	216	Ab etwa 60° Umwandlung. Ab etwa 230° kleine Nadeln, Stengel, Blätter. Gleichgew.: spitze Prismen. Erstarrt glasig (Reimers) (67). Brechungsindex der Schmelze zwischen 1,6229 und 1,6128.
230—245	Rivanol 	170	162 tr.	188	Gelb. Ab 200° viele Tröpfchen. Schmilzt unter Braunfärbung und Zersetzung. Bei Verdunsten alkoholischer Lösung anisotrope Massen, die ab 110° erweichen und langsam Scheiben bilden bis Mosaik. Bei 130° aufgelegt, wird anisotrope Flüssigkeit sofort isotrop und scheidet skelettierte Rauten ab mit α' in kurzer Diagonale.
240—245	Digitonin Merrick (310) (Digitalis-Saponein)	185	100 tr.	222 tr.	Ab 170° Tröpfchen. Sintert bei 240—245° zu zähen Tropfen mit anisotropen Massen, die sich ab 290° zu lösen beginnen. Bei 310° alles isotrop. Gasblasen.
245	Carbazol = Triphenylimid 	175	204	222	Ab 130° verschiedene Blättchen. Dichte Sublimation. Gleichgew.: Straßenpflaster. Brechungsindex der Schmelze 1,539; bei 254—255° (Silikonkammer). Bei 150° sublimieren gelappte Sterne und unregelmäßige Platten mit α' etwa 1,59, γ' etwa 1,74.
245	Optochlohydrochlorid $\text{C}_{17}\text{H}_{21}\text{O}_2\text{N}_2 + \text{HCl}$	162	147	185	Ab 175° Körner, Blättchen. Schmilzt unter mäßigen Gasblasen und Braunfärbung. Brechungsindex der Schmelze zwischen 1,5000 und 1,5000.

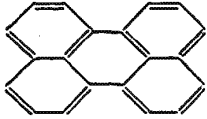
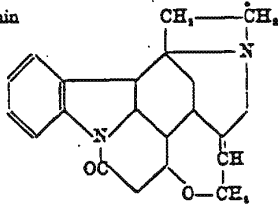
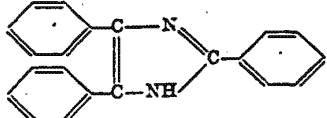
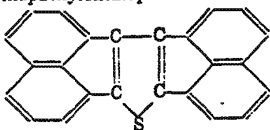
F. °C	Substanz	Eutekt. Temp. mit Salo-phen	Temp. mit Dicyan-diamid	Phenol-phthalein	Besondere Kennzeichen
246	α, β -Bis-diphenylenäthan (= Difluorenyl) 	186	208	238	Ab 135° Nadeln, Prismen. Gleichgew.: Balken, Spieße. Erstarrt sphärolithisch, meist instabile Modifikation mit sofortiger Umwandlung. Brechungsindex der Schmelze 1,6010 bei 246°, 1,5898 bei 271—275°.
240—246	Diacetyldioxyphenylisatin (= Isacen) $C_{14}H_{10}O_4N$	184	192	—	Ab 200° Tröpfchen. Schmelze gelblich. Erstarrt glasig.
246	Tetrahydro- β -naphthylaminhydrochlorid (= Termin) 	180	160	—	Ab 150° Nadeln, Blättchen, später verfilzte Aggregate. Gleichgew.: Balken, Prismen, sinkt rasch ab. Schmelze braun. Aus kaltem Alkohol Rauten (50°), Sechsecke oder flächige Aggregate. In Rauten α' in langer Diagonale, α' etwas über 1,57, γ' kleiner 1,62. (In Anilin löslich.) Aus heißem Alkohol Rechtecke mit α' etwa 1,62, γ' etwa 1,66.
247	p,p'-Dioxydiphenylsulfon 	184	177	218	Ab 200° Nadeln, Stäbchen. Gleichgew.: Balken. Erstarrt sphärolithisch. Brechungsindex der Schmelze 1,5700 bei 252—254°, 1,5611 bei 277°.
247	Carbanilid (= N,N'-Diphenylcarbazon) 	177	201	214	Ab 150° Nadeln, Prismen, Körner. Gleichgew.: Stengel, Nadeln. Starks Sublimation erschwert Gleichgew. und Brechungsindex, 1,5403 bei 245° bis 250° (Silikonkammer oder großes Deckglas). Aus heißem Alkohol Stengel, α' immer längs, etwas höher 1,62, γ' in breiteren Stengeln > 1,74.
240—248	Parabansäure 	—180—	208	235	Ab 140° Nadeln, Körner, Blättchen. Gasblasen. Brechungsindex der Schmelze 1,5101, 1,5204. Aus Alkohol ausgezogene Kristalle, γ' (Länge) 1,66—1,74, α' etwa 1,63.
248	Antistin (= 2-[N-Phenyl-N-benzylaminomethyl]-imidazolinhydrochlorid) 	169	151	162	Ab 190° Prismen, Nadeln. Schmelze bald braun. Brechungsindex der Schmelze 1,5611 bei 245—247°.
249	α -Diphenylglyoxim 	180	190	236	Ab 180° Nadeln, Spieße. Schmilzt unter Braunfärbung und Gasblasen. Brechungsindex der Schmelze zwischen 1,5502 und 1,5611 wegen Gasblasen erschwert; sinkt ab. Bei 240° sublimieren dünne, skelettierte Platten mit α' und γ' über 1,66 (< 1,74). Aus Alkohol Rauten, α' und γ' ebenso.
240—250	α -Naphthylaminhydrochlorid 	180	145	210	Meist rötlich verfärbt. Geruch. Ab 110° Blättchen, Stengel. Schmilzt unter Verflüchtigung und Braunfärbung. In Silikonkammer häufig ab 230° (nicht immer!) Umwandlung in Spieße mit F. 270° bis 275° und Verflüchtigung. Bei 160° sublimieren gestreifte Rechtecke, α' längs. Brechungsindex zwischen 1,66 und 1,74. Dicke Rechtecke mit Austritt erster Bisektrix, kleiner Achsenwinkel. Starke Dispersion.
Verfl. 250	Hexamethylentetramin 	176	175	193	Ab 80° isotrope Körner, meist sechseckig. Später Straßengpflaster. Verflüchtigt bis 250° vollständig. Brechungsindex der isotropen Körner 1,82 bei 76—77° (ohne runde Deckplatte bestimmen!).

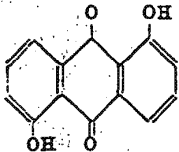
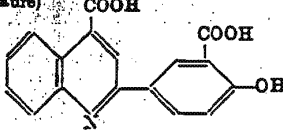
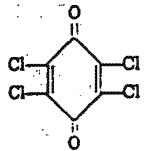
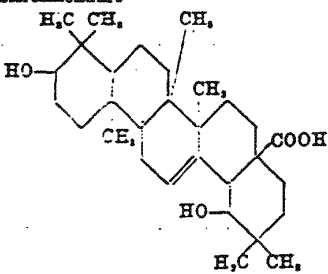
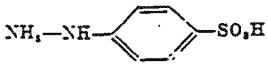
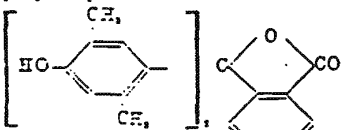
F. °C	Struktur	Eutekt. Temp. mit Salophen	Temp. mit Dicyan- diamid	Phenol- phthalein	Besondere Kennzeichen
246—250	2,4,6,2',4',6'-Hexanitrodiphenylamin 	171	180 tr.	235	Gelb. Ab 200° Körner. Schmilzt unter Braunfärbung und Gasblasen. Tropfenbildung stört bei Eutektikum mit Phenolph. Brechungsindex der Schmelze zwischen 1,6126 und 1,6231, wegen Gasblasen erschwert.
240—250	Phenylhydrazinhydrochlorid 	185	141	230	Geruch. Ab 120° starke Sublimation. Blättchen. Schmilzt unter Gelbfärbung und Gasblasenbgl. Größere Mengen verkohlen. Bei 160° sublimieren Rosetten und vier- und sechseckige Platten mit gerader Auslöschung. Kantenwinkel 87°, Länge α'. Brechungsindex der Platten zwischen 1,59 und 1,62.
230—250	Mescalinsäure (= 3,4,5-Trimethoxy-β-phenylthylaminosäure) 	163 tr.	130 tr.	155 tr.	Ab 125° entweicht Wasser unt. Trübung einzelner Kristalle. Bei 180° Schmelzen und teilweise Wiederkristallisation. Zwischen 220—230° Schmelzen der Körner und Auskristallisieren von Platten. Schmelze bräunlich. Brechungsindex der Schmelze zwischen 1,4683 und 1,4642. Mit Salophen n = 1,5403 bei 151—154°, 1,5391 bei 177—188°. In Paraffin ab 160° Dampfblasen. 165—168° inhomogenes Schmelzen.
253	Trimorphphthalein 	174 tr.	204	225	Ab 210° Körner, Prismen. Gleichgew.: Platten, Balken, sinkt ab. Schmelze gelb, erstarrt glasig. Brechungsindex der Schmelze 1,5203 bei 239—241°, 1,5101 bei 256—258°.
250—253	4,4'-Dicyanodiphenylsulfon 	185	196	225	Erstarrt teils sphärolithisch, teils feinstrahlig. Dimorph. Brechungsindex der Schmelze 1,5700 bei 248°, 1,5611 bei 277°.
252—254	Barbitinsäure (= Malonylbarbitoff) 	187	205	240	Ab 180° Körner. Schmilzt unter Zersetzung mit Bildung vieler feiner Körner. Bei 200° sublimieren unregelmäßige Körner, Dreiecke, Knäuel-Aggregate. Brechungsindex zwischen 1,66 und 1,74.
254	Carycen 	184	205 tr.	237	Gelb. Ab 170° unregelmäßige Blättchen, dichte Sublimate. Gleichgew.: runde Platten. Erstarrt zu Platten. Brechungsindex der Schmelze 1,6433 bei 275—277°.
254	Oxanilid 	185	206	231	Ab 125° Nadeln, Rechtecke. Gleichgew.: Platten, Balken. Starke Flüchtigkeit. Brechungsindex 1,5299 bei 271—273°, 1,5403 bei 248—250°. Bei 200° sublimieren dünne Rechtecke, γ' längs, γ' wenig < 1,74.
250—255	Martand (= 4-Aminomethyl-2-methyl-sulfonamidothiophenol = Merudin) 	154 tr.	151	225	Ab 165° Körner, Stengel, große Tropfen (stören bei Eutektikum); lebhafte Interferenzfarbe. Schmilzt trög unter Zersetzung. Brechungsindex zwischen 1,5700 und 1,5611. Nach einigen Minuten fallen drei-, vier- oder sechsstrahlige isotrope Sternchen aus.
245—255	Morphin 	156	174	255 tr.	Zwischen 115—140° entweicht Wasser unt. Trübung. Ab 175° Körner, Stengel. Schmilzt unter Zersetzung. Schmelze braun. Mit Salophen n = 1,5700 bei 156—157°, 1,5611 bei 186—187°. In Paraffin ab 153° Gasblasen, ab 160° reichlich Gasblasen; aus Kristallen neue Blättchen ohne Schmelze. Für Eutektikum trocknen!

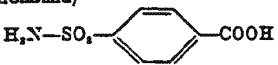
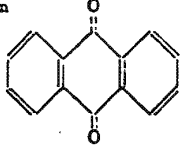
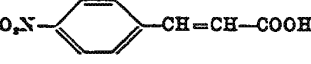
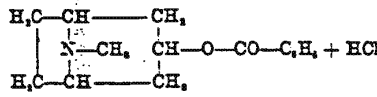
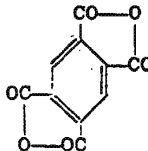
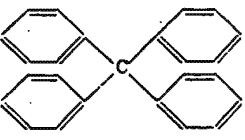
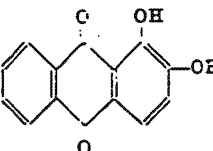
F.C	Substanz	Eutekt. Temp. mit Salophen	Dicyan- diamid	Phenol- phthalein	Besondere Kennzeichen
250—253	Paracodinhydrochlorid (= Dihydrokodesinhydrochlorid) 	165	131	—	Ab 180° Körner, Prismen. Ab 230° Tröpfchen und Zusammenfließen; daher F. schwer zu bestimmen. Schmelze bald braun. Brechungsindex zwischen 1,5502 und 1,5611. Mit Salophen $n = 1,5700$ bei 155—158°, 1,5611 bei 184—186°.
255	α -Aminoanthrachinon 	176	207	222	Rot. Ab 145° Nadeln, Stäbchen, reichlich Sublimata. Gleichgew.: Stengel, Balken. Brechungsindex zwischen 1,6483 und 1,6598. Ursubstanz flache Nadeln, α' längs (zwischen 1,59 und 1,62, mit Rotfilter), $\gamma' > 1,74$.
250—253	4,4'-Dimethoxydiphenylsulfon-2,2'-dicarbonsäure 	184	184	—	Ab 230° Nadeln, Stengel. Gleichgew.: Stengel. Erstarrt glasig. Bei Erwärmen sphärolithisch. Brechungsindex 1,5502 bei 254—257°.
256	Pentaerythrit 	160	174	188	Ab 140° Körner, Blättchen, später Straßenpflaster. Gleichgew.: gelpappte Blättchen. Bei höherer Temperatur Gasblasenblg. Brechungsindex der Schmelze 1,4339 bei 290—295°.
257	Tetrachlorphthalsäureanhydrid 	173	184	233	Ab 150° Nadeln, Stengel, später Straßenpflaster. Starke Sublimation. Geruch. Erstarrt federförmig. Dimorph. Mit Dicyandiamid Braunfärbung. Brechungsindex der Schmelze zwischen 1,5301 und 1,5203. Bei 200° sublimieren Prismen und Stengel. Achsenebene quer, meist Austritt einer optischen Achse. In Anilin löslich unter Abscheidung winziger Sphärolithe.
247—257	Hykokoll (= Aminoessigsäure = Glycin) 	190	189	245	Ab 70° Rhomboide, Stäbchen, Prismen. Schmilzt unter Braunfärbung. Aus Wasser gerade auslöschende Prismen mit Brechungsindex quer kleiner, längs größer 1,55. Ferner Rhomboide (auch bei Sublimaten) mit schiefer Auslöschung 18° (= α'), Kantenwinkel 112°.
254—257	5'-p-Aminobenzol-sulfonamino]-pyrimidin (= Sulfadiazin) 	184	195	227	Ab 170° Körner, Prismen, Blättchen. Schmilzt trög unter Zersetzung, Gasblasen und Gelbfärbung. Erstarrt glasig. Bei 250° sublimieren Stäbchen, meist leicht geknickt, α' (längs) wenig über 1,60, γ' wenig unter 1,74. Aus heißem Wasser Stengel. β längs (opt. Achse exzentrisch).
225—230	Eccepal (= 7,8-Dihydro-14-oxykodesinhydrochlorid) 	147 tr.	148 tr.	180 tr.	Ab 70° entweicht Wasser unt. Trübung. Ab 150° aus Kristallen Nadeln und Blättchen. Schmelze zäh und bräunlich. Brechungsindex der Schmelze zwischen 1,5502 und 1,5611; reichlich Gasblasen. Mit Salophen $n = 1,5700$ bei 157—160°, 1,5611 bei 192—195°. Über 200° aufgelegt Schmelzen und Kristallisation strahliger Aggregate. In Paraffin ab 82° sehr trübes inhomogenes Schmelzen.

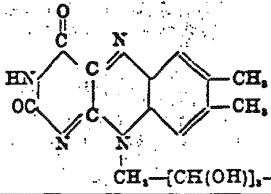
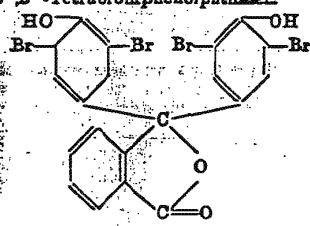
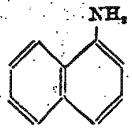
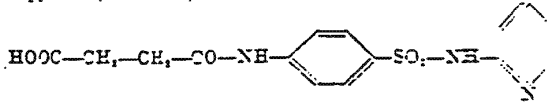
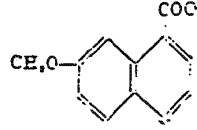
F.°C	Substanz	Eutekt. Temp. mit			Besondere Kennzeichen
		Salophen	Dicyandiamid	Phenolphthalein	
261	Acenaphthenchinon 	177	193	217	Gelb. Häufig schmutzig verfärbt. Umsublimieren bei 250°. Brechungsindex 1,5795 bei 272—276°, 1,5897 bei 248—252°. Ab 100° reichlich Nadeln. Gleichgew.: Nadeln, Spieße. Schmelze braun. Erstarrt zu Platten. Mit etwas Phenylhydrazin leicht erwärmt orange Nadeln.
262	Emodin (= Frangula-emodin) 	174	201	234	Orange. Ab 160° Nadeln, Stäbchen, Stengel. Gleichgew.: Stengel. Erstarrt sphärolithisch. Polymorph II 258°, III unbeständig. Brechungsindex 1,6715 bei 255—260°.
257—262	Chinidinhydrochlorid $C_{20}H_{21}O_2N_2 + HCl + H_2O$	176	127	190	Ab 90° entweicht Wasser unt. Trübung. Ab 150° teilweises Schmelzen und Wiederkristallisation. Ab 180° Schmelze dunkelbraun. Mit Salophen n = 1,5898 bei 112—114°, 1,5794 bei 143—146°. In Paraffin bei 130—135° Umlagerung zu wasserfreien Kristallen.
262	p-Acetamino-benzoesäure 	181	183	234	Ab 150° Nadeln, später Prismen. Gleichgew.: Prismen, sinkt ab. Bald Gasblasen. Aus Alkohol Rhomboide, Stengel, Rosetten. In Rhomboiden schiefe Auslöschung (30°), α' etwa 1,59, γ' > 1,74.
263	5-Phenylbarbitursäure 	186	—185—	—232—	Ab 220° Blättchen. Gleichgew. trägt: Blättchen, Prismen. Schmelze gelb. Erstarrt sphärolithisch. Brechungsindex zwischen 1,5203 und 1,5301. Aus Alkohol + Aceton Stäbchen, Länge α' = 1,53, γ' > 1,66 (starke Vergrößerung!)
263	Phenolphthalein 	174	200	263	Ab 230° Fünfecke, Stäbchen, Rosetten. Gleichgew.: Fünfecke, Platten, Spieße. Erstarrt langsam, breitstrahlig. Brechungsindex 1,5794 bei 244—246°, 1,5700 bei 275°.
264	Cinchonin 	180	206	175	Ab 180° Stengel. Gleichgew.: Stengel. Brechungsindex der Schmelze zwischen 1,5301 und 1,5203. Mit Salophen n = 1,5611 bei 167—168°, 1,5502 bei 192—193°.
258—265	Gallussäure (= 3,4,5-Trioxibenzoessäure) 	173	181	238	Ab 90—110° entweicht Wasser unt. Trübung. Ab 210° Prismen, Körner. F. unter Zersetzung und Gasblasen. Dimorph (im Sublimat) II 230° (10). Mit Fe Cl, blauschwarzer Niederschlag. In Paraffin ab 115° Gasblasen, ab 125° Umlagerung ohne Schmelze.
etwa 265	Dibenzylaminhydrochlorid 	175	166	190	Ab 120° Nadeln, Blättchen. F. wegen Flüchtigkeit nur bei Auflegen über 250° möglich. F. in Silikonkammer 266°, sinkt stark ab. Brechungsindex in Silikonkammer 1,5204 bei 268—270°. Aus Alkohol

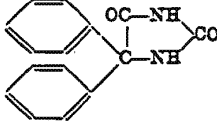
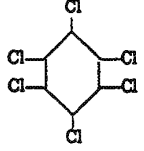
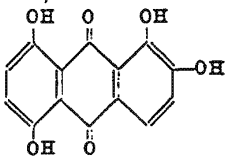
F.°C	Substanz	Erweich. Temp. mit			Besondere Kennzeichen
		Salophen	Dicyan- diamid	Phenol- phthalein	
250—265	Benzedrin (= Amphetamin)-hydrochlorid  + HCl	150	171	228 tr.	Ab 100° Körner. Schmilzt unter starker Gasblasenbildung und Braunfärbung. Leicht löslich in Wasser und Alkohol. Aus Alkohol unregelmäßige Körner, 1. Bisektrix (kleiner Achsenwinkel, positiv). Brechungsindex etwas unter 1,55.
263—266 (232)	p-Acetylaminobenzolsulfonylguanidin 	187 tr.	192	—	Ab 140—170° Trübung der Kristalle. Teilweises Schmelzen bei 232°. Ab 250° Tröpfchen. Zersetzung. Erstarrt glasig. In Paraffin Gasblasen ab 115°. Dimorph II 232° (Markovic). Brechungsindex zwischen 1,5611 und 1,5700.
258—265	Kreatin  + H2O	155	191	212	Ab 230° Stengel, Balken. Schmilzt unter Zersetzung. Gasblasen. Bei 200° aufgelegt, teilweises Schmelzen, teilweise Trübung. In Paraffin entweicht Wasser zwischen 110—120°. Ab 230° neue Gasblasen und Ausscheidung von Stengeln mit F. 280° bis 290°. Aus Wasser lange Rechtecke, α' längs < 1,55, γ' etwas > 1,59, ferner Stengel mit flachem Dach, α' längs.
260—268	Kasumbitartrat 	158	194	260	Ab 260° Gasblasen. Schmilzt unter heftiger Gasblasenblg. Aus Wasser sechseckige Prismen, Leisten, Vierecke (92) mit symmetrischer Auslöschung und Achtecke (Austritt einer Bisektrix). α' = β etwa 1,55, γ' zwischen 1,59 und 1,62 (mit Rotfilter höher).
265—270	Succinamid (= Bernsteinsäurediamid) 	150	186	228	Geruch. Zwischen 70—80° teils Schmelzen, teils Trübung. Ab 260° Körner, Rhomboid (fast Rechtecke), Stengel. Ab 260° in Kristallen Gasblasen und Flüssigkeit. Gleichgew.: Rechtecke. Schmilzt mit Gasblasenblg. und Braunfärbung. Brechungsindex zwischen 1,4339 und 1,4584.
220—270	Apomorphinhydrochlorid  + HCl	150	167	212	Ab 220—270° Trübung der Kristalle, die dann grau werden und zu schwarzen Tropfen schmelzen. Mit Salophen n = 1,6011 bei 159—161°, 1,5897 bei 192—194°. Nadeln mit γ (längs) zwischen 1,66 und 1,74, β wenig über 1,66.
260—270	Tyrosin (= β-[p-Oxyphenyl]-α-aminopropionsäure) 	154	196	245	Ab 250° Bräunung. Schmilzt trög unter Gasblasen und Bräunung. Aus Wasser feine Nadeln mit γ' längs, α' < 1,55, γ' > 1,59. Mit α-Naphthylurethan Verbindung mit F. 206°.
260—270	Kodeinhydrochlorid 	149	121	—165—	Teilweises Schmelzen bei 165—170°. Ab 230° Braunfärbung. Mit Salophen n = 1,5700 bei 166; 1,5611 bei 196—199°. Aus verdünntem Alkohol Stäbchen und Prismen, die in Paraffin ab 130° Gasblasen bilden unter Abscheidung neuer Kristalle oder bei 140—145° homogen schmelzen.
262—272	4-Nitro-2-aminobenzoessäure 	177	150	232	Orange. Ab 150° Nadeln, Stengel. Bei 260° leichtes Kochen in den Kristallen. Schmilzt unter Zersetzung und Verflüchtigung. Bei 250° sublimieren kleine Rechtecke und monosymmetrische Blättchen wie Schmetterlingschuppen. α' (längs) etwa 1,52, γ > 1,66.
262—272	p-Dimethylaminobenzyliden-rhodanin 	155	234	246	Erdbeerrot. Ab 200° Nadeln, Körner. Schmilzt unter Zersetzung. Schmelze rotbraun. Brechungsindex der Schmelze > 1,6577.

F. °C	Substanz	Eutekt. Temp. mit			Besondere Kennzeichen
		Salophen	Dicyan- diamid	Phenol- phthalein	
274	Theophyllin (= Theocin) $\begin{array}{c} \text{H}_2\text{C}-\text{N}-\text{CO} \\ \quad \quad \\ \text{CO} \quad \text{C}-\text{NH} \\ \quad \quad \\ \text{H}_2\text{C}-\text{N}-\text{C}=\text{N} \end{array} \begin{array}{c} \diagup \\ \diagdown \end{array} \text{CH}$	180	182	222	Zwischen 70—80° entweicht Wasser unt. Trübung. Ab 140° Nadeln, Blättchen, Prismen. Dichte Sublimat. Gleichgew.: Prismen, Stengel. Dimorph II 271° (74). Brechungsindex 1,5203 bei 286°.
270—275	Elbon (= Cinnamoyl-p-oxyphenylkarnstoff) $\text{C}_6\text{H}_5-\text{CH}=\text{CH}-\text{COO}-\text{C}_6\text{H}_4-\text{NH}-\text{CO}-\text{NH}_2$	169	175	186	Schmilzt bei 190—195°, kristallisiert später wieder und schmilzt bei 270—275° unter Gelbfärbung. Brechungsindex 1,5700 bei 263—265°.
270—275	Perylen 	190	210	246	Orange. Ab 180° gelbe Rauten (fast Quadrate mit symmetrischer Auslöschung), gelappte Platten; starke Sublimation. Ab 250° teilweises Schmelzen unter Braunfärbung. Gleichgew.: Platten, Stengel. Schmelze orangefarbig bis braun. In sublimierten Rauten α' wenig über 1,66, γ' über 1,74.
270—275	Strychnin 	180	201	208	Ab 220° schöne Prismen, Körner. Ab 260° Braunfärbung. Schmelze braun. Gasblasen. Mit Salophen n = 1,5898 bei 149—153°, 1,5700 bei 173—178°. Mit Chlorwasser Niederschlag von Monochlorstrychnin.
277	Lophin (= 2,4,5-Triphenylimidazol) 	165	206	230	Ab 220° Nadeln, Stengel. Gleichgew.: schiefwinklige Stengel. Sehr flüchtig (großes Deckglas). Brechungsindex 1,5898 bei 234°.
273	peri-Dinaphthylthiophen 	180	206	252	Rotbraun. Ab 240° feine lange Nadeln, später flache Stengel. Gleichgew.: Stengel, Prismen. Schmelze braunrot. Bei 260° sublimieren gebogene pleochroitische Stengel und flache Stengel mit γ' (quer) größer 1,74, α' zwischen 1,62 und 1,65.
275—278	β-Eucain (= O-Benzoyl-vinyldiaceton-alkaminhydrochlorid) $\begin{array}{c} \text{O}-\text{CO}-\text{C}_6\text{H}_5 \\ \\ \text{HC}=\text{C}(\text{CH}_3)-\text{CH}_2-\text{N}^+\text{H}(\text{CH}_2)_3 \\ \\ \text{H}_2\text{C} \end{array} + \text{HCl}$	177	169	194	Ab 170° Nadeln. Starke Sublimation. Schmilzt unter mäßiger Gasblasenbildung. Erstarrt glasig. Brechungsindex zwischen 1,4633 und 1,4842; durch Flüchtigkeit erschwert. Aus Alkohol Rauten, symmetrische Auslöschung. γ' längs. Sublimiert bei 240° in flachen Stengeln, α' wenig > 1,50, γ' wenig < 1,62.
275—280	Berberinsulfat $\text{C}_{20}\text{H}_{21}\text{O}_4\text{N} + \text{H}_2\text{SO}_4$	180	139	205	Gelb. Ab 200° Bräunung. Schmelze dunkelschmutzgrün. Mit Salophen n = 1,6128 bei 169—172°, 1,6011 bei 199—204°. Aus verdünntem Alkohol Nadeln mit γ' (quer) über 1,74, α' etwa 1,50.
280—282	Mekonsäure $\begin{array}{c} \text{HC}-\text{CO}-\text{C}-\text{OH} \\ \quad \quad \\ \text{HOOC}-\text{C}-\text{O}-\text{C}-\text{COOH} \end{array} + 3 \text{H}_2\text{O}$	171	202	245	Zwischen 90—110° entweicht Wasser. Ab 260° Zersetzung und Braunfärbung. Ab 270° Verflüchtigung oder Verkohlung. Bei raschem Erhitzen Schmelzen unter heftigem Aufbrausen und Verkohlung. Aus Mischung mit Phenolphthalein sublimieren ab 210° neue, große Körner und dicke Rauten. In Benzol bei 165° Schmelze.

F.°C	Substanz	Eutekt. Temp. mit Salo- phen	Dicyan- diamid	Phenol- phthalein	Besondere Kennzeichen
265—280	Yohimbinhydrochlorid $C_{21}H_{23}O_2N_2 \div HCl$	183	193	248	Ab 250° Tröpfchen. Kristalle schon vor dem Schmelzen braun. Schmelze zäh, braun, viel Gasblasen. Aus Alkohol unregelmäßige und ovale Blättchen mit γ' längs. Wässrige Lösung gibt mit Na_2CO_3 Flocken und Nadeln von Yohimbin, F. 230—235°.
280	1,5-Dioxyanthrachinon (= Anthrarufin) 	180	103 tr.	246	Gelb-orange. Wenn schmutziggelb verfärbt, Umsublimieren bei 270°. Ab 120° Nadeln, Körner, Blättchen. F. durch Flüchtigkeit erschwert. Schmelze orange. Brechungsindex in Silikonkammer 1,6231 bei 276—278°.
270—280	Hexophaan (= 2-[4-Oxy-3-carboxyphenyl]-cinchoninsäure)  + H ₂ O	189	172	255	Schmilzt unter Braunfärbung und Gasblasen. Aus Wasser kleine rechteckige Blättchen mit α' längs, α' etwa 1,51, γ' > 1,66 (starke Vergrößerung).
Verfl. 280 (290— 295)	Chloranil (= Tetrachlorchinon) 	182	195	228	Gelbe Blättchen. Ab 100° Rauten, Sechsecke, Spieße, Kreissegmente (eutektische Temperatur mit Sublimaten bestimmen!). Totale Verflüchtigung bei 270—280°. In Silikonkammer F. 290—295° unter Zersetzung. In Anilin löslich unter Abscheidung kleiner bräunlicher Rechtecke und farblosere sechseckiger Tafeln. In o-Toluidin blauschwarze Lösung.
278—280	Dipropylaminhydrochlorid $(C_3H_7)_3NH \div HCl$	162	114	138	Ab 140° isotrope Quadrate, an den vier Ecken Skelettkristalle, ferner anisotrope Körner und Leisten. Vollständige Umlagerung. Straßenpflaster. Sehr flüchtig (großes Deckglas!). Brechungsindex der Schmelze < 1,4339. Bei Eutektikum mit Phenolphthalein viele Nadeln.
283	Siaresinolsäure 	188	209	—225—	Ab 180° Trübung der Kristalle. Ab 230° Tröpfchen. Ab etwa 250° an Kristallen nadelige Auswüchse wie Igel. Schmilzt unter Gasblasen und Gelbfärbung. Brechungsindex der Schmelze zwischen 1,4683 und 1,4842.
280—285	Phenylhydrazin-4-sulfonsäure 	179	163	—225—	Manchmal bräunlich verfärbt. Ab 110° Tröpfchen. Schmilzt unter Gasblasenbildung. Erstarrt glasig. Aus Wasser gerade auslöschende Spindeln und Prismen mit α' längs, α' etwa 1,59, ferner graue schmale Spindeln mit schiefer Auslöschung.
280—285	p-Xylenolphthalein 	185	208	244	Ab 220° Körner, kurze Prismen. Schmilzt unter Zersetzung und Braunfärbung. Brechungsindex der Schmelze etwa 1,5403, ändert sich mit Bräunung.

F. °C	Substanz	Eutect. Temp. mit Salophen	Dicyan- diamid	Phenol- phthalein	Besondere Kennzeichen
254—286	p-Sulfamid-benzoesäure (= Benzoesäure-p-sulfonamid) 	184	183	246	Ab 210° Stengel, Spieße, Prismen. Gleichgew.: Tafeln; sinkt ab. Erstarrt glasig. Brechungsindex der Schmelze zwischen 1,5403 und 1,5502.
256	Anthrachinon 	183	206	238	Hellgelb. Ab 150° feine Nadeln. Gleichgew.: Balken, Platten. Sublimiert sehr stark. Brechungsindex der Schmelze 1,5611 bei 280—282° (großes Deckglas).
256	p-Nitrozimtsäure 	182	193	232	Ab 160° rechteckige Stäbchen, Stengel, Prismen. Gleichgew.: Prismen, Platten, sinkt ab. Schmelze wird braun. Bei 240° sublimieren dünne Rechtecke, monosymmetrische Fünf- oder Sechsecke mit α' in Symmetrieebene. α' etwa 1,53, γ' über 1,74.
230—288	Guanidinsulfat $2 [H_2N-C(=NH)-NH_2] + H_2SO_4 + \frac{1}{2} H_2O$	179	181	256 tr.	Im Handelspräparat neben isotropen Hemihydratkr. oft anisotrope wasserfreie Kristalle. Ab 100° einzelne Tröpfchen. Bei etwa 130° Zerspringen der Kristalle. Ab 230° wenig kleine Körner. Schmilzt mit Gasblasenbgl. In Wasser löslich. In Paraffin ab 110° Umwandlung des isotropen Hydrats in anisotrope Kristalle.
235—288	Tropacocainhydrochlorid 	168	141	165 tr.	Ab 160° Prismen, allmählich reichlich Sublimat. Schmilzt mit Gasblasenbgl. und leichter Braunfärbung. Mit Salophen $n = 1,5611$ bei 122—124°, 1,5502 bei 153—155°. Sublimiert bei 240° in Aggregaten aus blattartigen und prismatischen Kristallen mit α' unter 1,55 (in o-Toluidin und Anilin löslich), γ' zwischen 1,62 und 1,66.
233	Pyromellitsäuredianhydrid 	181	175 tr.	229	Ab 200° schöne große Polyeder (instabile Modifikation); später Verflüchtigung oder Umwandlung durch neue gelappte Kristalle. Gleichgew.: Balken, Spieße. In Paraffin starke Gasentwicklung ab 130°. Neuerliche Gasentwicklung ab 250°, bei 260—265° Schmelzen zu braunen Tropfen.
238	Tetraphenylmethan 	183 tr.	203	256	Ab 190° Nadeln, Stengel, starke Sublimation. Gleichgew.: Balken, Stengel. Brechungsindex der Schmelze 1,5299 bei 302—305°; wegen Flüchtigkeit erschwert.
232	Alizarin (= 1,2-Dicyanthrachinon) 	179	200	242	Dunkelorange, meist braun verfärbt. Umsublimieren bei 260°. Ab 170° Nadeln und Stengel. Gleichgew.: Spindeln, Spieße. Baldige Zersetzung. Erstarrt sphärolithisch. Für Brechungsindex Sublimat verwenden! Brechungsindex der Schmelze 1,6126—1,6231.
etwa 220	Fumarsäure $HOOC-CH=CH-COOH$	182	152	250	Ab 110° Körner, Nadeln, Balken. Gegen 290° Verflüchtigung und Verfärbung (Zersetzung). Bei 210° sublimieren Stengel, zum Teil nicht einheitlich auslöschend, zum Teil schief auslöschend. α' wenig > 1,56, γ' wenig < 1,53. In Toluidin löslich unter

F. °C	Substanz	Erwärmen Temp. mit			Besondere Kennzeichen
		Salol	Diphenyl- diamid	Phenol- phthalein	
280—290	Lactoflavin (= Vitamin B ₂)  $\text{CH}_2\text{—}[\text{CH}(\text{OH})]_3\text{—CH}_2\text{OH}$	189	191	248	Gelb. Ab 260° wenig Nadeln. Schmilzt unter starker Zersetzung mit Gasblasen. Lösung intensiv gelb, grüne Fluoreszenz, die durch Mineralsäuren oder Alkali verschwindet.
280—290	l-Isosucin (= α-Amino-β-methylvaleriansäure) $\text{C}_6\text{H}_5\text{—CH}(\text{CH}_3)\text{—CH}(\text{NH}_2)\text{—COOH}$	183	186 tr.	248	Ab 170° Blättchen. F. durch Flüchtigkeit erschwert (Silikonkammer!). Starke Gasblasenbildung schon bei Eutektikum mit Phenolphthalein. Aus verdünntem Alkohol gestreckte Sechsecke mit γ' längs. γ' etwa 1,56, α' < 1,55.
280—290	β-Dextrin (= Hexamyllose) $(\text{C}_6\text{H}_{10}\text{O}_5)_n + \text{H}_2\text{O}$	190	191	260	Zwischen 80—100° entweicht Wasser. Ab etwa 250° Verfärbung. Schmilzt unter starkem Aufblähen. In Paraffin ab 90° Blasen. Durch Jod braun. Aus Wasser Prismen und Rechtecke (grau) mit n zwischen 1,50 und 1,51.
270—290	3',5',3'',5''-Tetrabromphenolphthalein 	181	207	248	Ab 250° zahlreiche sichtstörende Tröpfchen, später Körner. Bei langsamem Erwärmen mäßige Zersetzung. Schmelze gelb. Gleichgew. trög: Körner, Sechsecke, Prismen. Brechungsindex der Schmelze 1,6126 bei 275—280°.
285—293	α-Naphtylaminhydrobromid  + HBr	179	134	225	Manchmal schmutziggelb verfärbt. Umsublimieren bei 200°. Ab 120° Nadeln. Starke Sublimation. Bei Erwärmen widerlicher Geruch. Schmilzt trög unter Braunfärbung. Bei 160° aus Schälchen (3 mm Abstand) sublimieren zwei Modifikationen: instabile Rosetten und stabile Nadeln und Nadelsterne. Auf warmem Heiztisch Umwandlung oder Verflüchtigung von II.
293	4,4'-Bis-acetamino-diphenylsulfon (= Rodilon) $\text{CH}_3\text{—CO—NH—C}_6\text{H}_4\text{—SO}_2\text{—C}_6\text{H}_4\text{—NH—CO—CH}_3$	187	204	—	Ab 120—130° teilweises Schmelzen einzelner Kristalle. Ab 265° Tröpfchen. Schmilzt unter Zersetzung und Braunfärbung (Markovic). Brechungsindex der Schmelze zwischen 1,5502 und 1,5611.
296 (197—199)	2-[p-Succinylaminobenzolsulfamino]-pyridin (= Soranil) 	186	196	—	Die instabile Modifikation schmilzt zuerst bei 197—199°, nach Erkalten und Erwärmen kristallisiert stabile Modifikation mit F. 296°. Dimorph II 197—199° (Markovic).
275—295	Strychninhydrochlorid $\text{C}_{21}\text{H}_{22}\text{N}_4 + \text{HCl} + 2\text{H}_2\text{O}$	188	181	—190	Ab 70° entweicht Wasser unt. Trübung. Ab 175° einzelne Körner, Blättchen, ab 220° starke Sublimation. Schmilzt unter Zersetzung. In Paraffin ab 130° Dampfblasen. Totale Umwandlung 150—170°.
286—295	Chininsäure (= 6-Methoxychinolin-4-carbonsäure) 	182 tr.	—184	240	Ab 190° Körner, Stengel, Prismen. Ab 250° Verflüchtigung oder Schmelzen der ersten Sublimat. Bildung neuer Prismen (Rechtecke). F. vom Erhitzungsstempo abhängig. Aus Alkohol Nadeln mit α' (längs) zwischen 1,46 und 1,48, γ' über 1,66.

F. °C	Substanz	Eutekt. Temp. mit			Besondere Kennzeichen
		Salophen	Dicyan- diamid	Phenol- phthalein	
298	5,5-Diphenylhydantoin (= Difhydan) 	182	203	—	Ab 180° Nadeln. Gleichgew.: Stengel, Prismen. Brechungsindex der Schmelze zwischen 1,5301 und 1,5203.
299	d-Valin $(\text{CH}_3)_2\text{CH}-\text{CH}(\text{NH}_2)-\text{COOH}$	182 tr.	188 tr.	243	Ab 170° Nadeln, später Blättchen. F. nur bei Auflegen über 290° möglich (großes Deckglas). Bei Schmelzen Gasblasen. Aus verdünntem Alkohol Rhomboide, schiefe Auslöschung (2°). α' wenig kleiner 1,59.
285—300	Morphinhydrochlorid $\text{C}_{17}\text{H}_{19}\text{O}_2\text{N} + \text{HCl} + 3 \text{H}_2\text{O}$	186	175	236	F. nur bei raschem Erhitzen, sonst Verkohlung. Ab 210° Körner, Prismen. In Paraffin inhomogenes Schmelzen 88—90°. Mischung aus einem Teil Morphin-HCl mit vier Teilen Zucker gibt in Schwefelsäure Rotfärbung.
290—300	α -Dextrin (= Tetramylose) $(\text{C}_6\text{H}_{10}\text{O}_5)_n + \text{H}_2\text{O}$	188	198	258	Zwischen 90 und 130° entweicht Wasser. Schmilzt unter Aufblähen. In Paraffin Umwandlung in wasserfreie Kristalle nur allmählich. Gasblasen.
Verfl. 260—300 (310)	β -Hexachlorcyclohexan 	181	208	249	Beim Erwärmen Geruch. Ab 140° dreiseitige Körner (Oktaeder), später Straßpflaster. Verflüchtigung trotz großen Deckglases bei 280—300°, in Silikonkammer F. 310° (korr.). Bei 200° sublimieren (ohne Abstand) isotrope Körner mit $n = 1,68$ bei 38°; mit Abstand sublimieren anisotrope Sechsecke, baldige Umwandlung (54).
300—310	d-Valin $(\text{CH}_3)_2\text{CH}-\text{CH}(\text{NH}_2)-\text{COOH}$	185 tr.	190 tr.	252	Ab 190° Nadeln, Blättchen, starke Sublimation. F. wegen Flüchtigkeit nur bei Auflegen über 290° möglich, bei Schmelzen Gasblasen. Aus verdünntem Alkohol lange Sechsecke. γ' (längs) wenig > 1,59, α' höher 1,55 (Unterschied von Isoleucin).
280—310	Strychninnitrat $\text{C}_{21}\text{H}_{22}\text{O}_4\text{N}_2 + \text{HNO}_3$	175	160	208	Ab 230° vierseitige Blättchen. Ab 250° Braunfärbung. 1 cm³ wässrige Lösung (1:99) mit 0,5 cm³ $\text{K}_2\text{Cr}_2\text{O}_7$ -Lösung gelbe Kristalle. Aus heißem Wasser lange, flache Stengel. Gerade Auslöschung. α' (längs) wenig < 1,62, γ' > 1,66.
313—316	1,2,5,8-Tetraoxyanthrachinon (= Chinalizarin) 	188	207	258	Rot. Ab 180° lange Nadeln, Büschel. Stark flüchtig. Nadeln schiefe Auslöschung. Pleochroitisch. α hellorange, β und γ braunrot. Teilweise Zwillinge.
300—320	Diisobutylaminhydrobromid $[(\text{CH}_3)_2\text{CH}-\text{CH}_2]_2\text{NH} + \text{HBr}$	164 tr.	154	190	Ab 190° Nadeln, Blättchen, sehr starke Sublimation. Schmilzt unter Zersetzung mit Gasblasen. Ursubstanz schwach anisotrope Blättchen mit $n = 1,53$ bei 33—35°.
323	Natriumacetat + 3 H ₂ O $\text{CH}_3\text{COONa} + 3 \text{H}_2\text{O}$	182	180 (63)	142	Ab 50° entweicht Wasser unt. Trübung. Einzelne Kristalle schmelzen inhomogen etwa 60°. Über 65° aufgelegt, sofortiges Schmelzen unter Ausfall schwach anisotroper Stengel. Mischung mit Phenolphthalein bei Erwärmen violett. Hydrat schmilzt in Paraffin homogen 55—58° (Gleichgew.: Prismen). Getrocknetes Salz bildet mit wenig Wasser

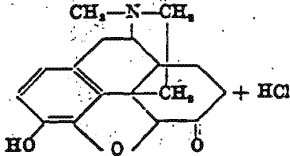
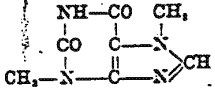
F. °C	Substanz	Eutekt. Temp. mit			Besondere Kennzeichen
		Salophen	Dicyan- diamid	Phenol- phthalein	
300—330	Äthylendiaminhydrochlorid $\text{H}_2\text{N}-\text{CH}_2-\text{CH}_2-\text{NH}_2 + 2 \text{HCl}$	190	167	244	Ab 140—160° Trübung der Kristalle und Hüpfen. Ab 190° Rechtecke, Dreiecke, Stäbchen, Rosetten. In Paraffin ab 140—160° Zerspringen mit Umwandlung.
325—350	Naphthacen (= Tetracen) 	186	205	257	Gelb. Ab 185° Blättchen; sehr flüchtig. Schmilzt unter Braunfärbung und Zersetzung. Sublimiert in Blättchen. $\alpha' = 1,66$ bei 80—100°, $\gamma' > 1,74$. Pleochroitisch. α hellgelb, γ bräunlichgelb.
300—330	Dilaudidhydrochlorid (= Dihydromorphinonhydrochlorid) 	185	133	232 tr.	Braunfärbung und langsame Verkohlung. Ursubstanz schwach anisotrope Körner. $n = 1,66$ bei 45—48° (mit Rotfilter). Starke Dispersion.
320—340	Theobromin 	185	197	245	Ab 150° Körner, Platten. Bei 200° starke Sublimation. Bei Schmelzen Braunfärbung und Verkohlung. Sublimiert in trüben Rauten, Körnern und Stäbchen mit Brechungsindex $> 1,74$, selten Rhomboide mit α' etwa 1,44.

Plate I

Figure 1.
Kofler hot stage with
slide manipulator
(thermometer model).

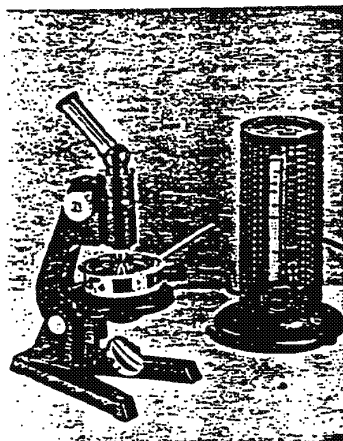
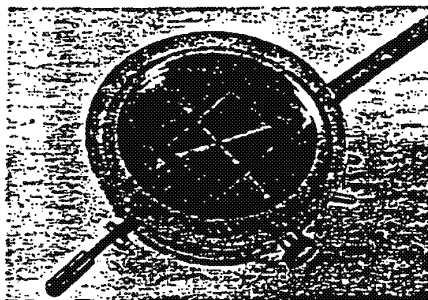
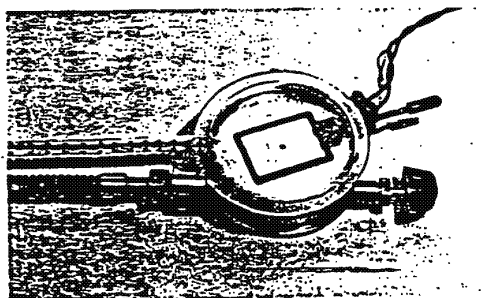


Figure 2.
Kofler hot stage
microscope.

Figure 3.
Kofler cold stage



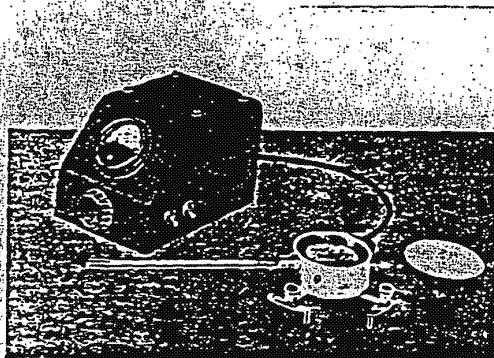


Figure 4.
Kofler high
temperature stage.

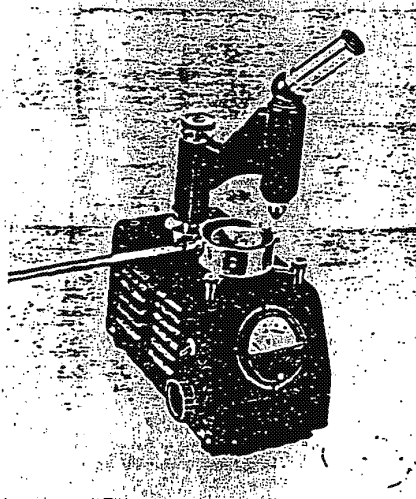


Figure 5.
Kofler hot stage
microscope with
swing-out bodytube.

Plate III

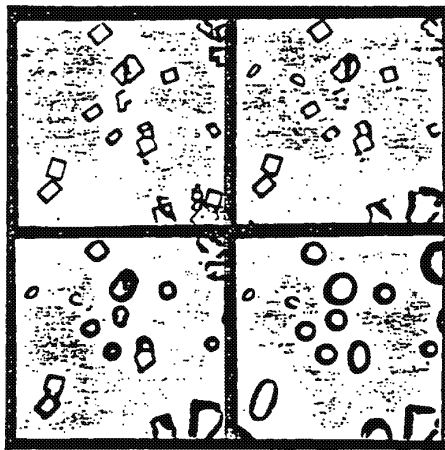


Figure 6.
Continuous heating
melting point for
anaesthesin.



Figure 7.
Equilibrium melting
point for cardiazol.



Figure 8.
Crystalline sublimate and
condensed droplets; the growing
crystals nucleate some droplets
and grow at the expense of the
more volatile droplets thru the
vapor phase.



Figure 10.
Growth spirals for borneol.



Figure 11.
Layer growth for naphthalin.

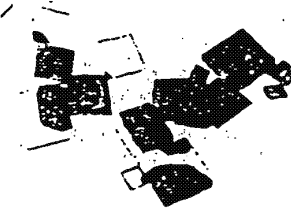
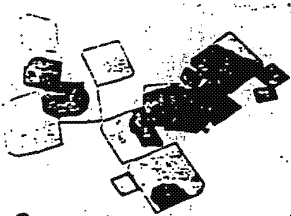
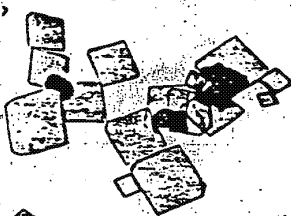
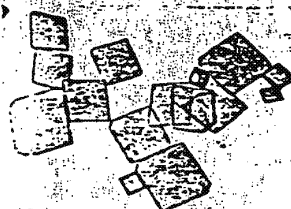


Figure 13.
Dehydration of phloroglucinol
hydrate.



Figure 14.
d Incongruent melting of $\text{BaBr} \cdot 2\text{H}_2\text{O}$
with crystallization of
anhydrous NaBr.

Plate V

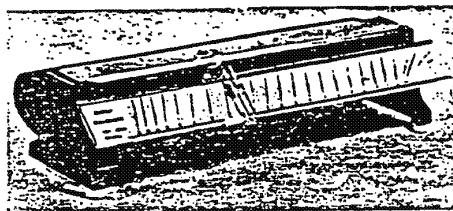


Figure 15.
Kofler hot bar (50° - 260°).

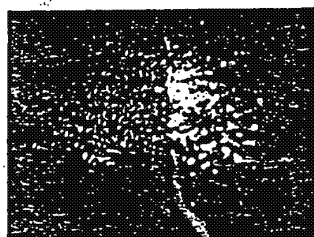


Figure 16.
Melting point with the
hot bar; the point
shows the demarcation
between crystals and melt.

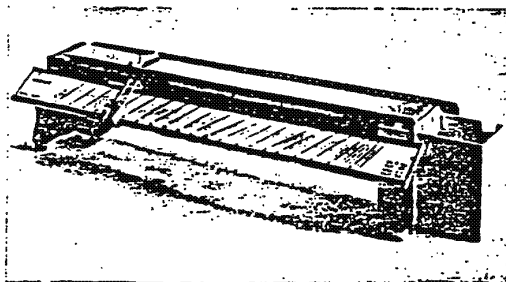


Figure 17.
Hot bar for lower
temperatures (10° - 210°).

Plate VI

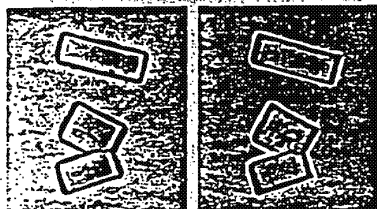


Figure 18.
Crystals with higher refractive index than the liquid:
a) on focusing higher
b) on focusing lower.

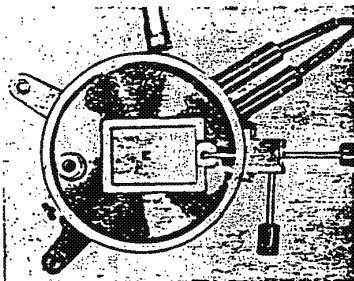


Figure 24.
Hot stage with metal block for molecular weight determination.

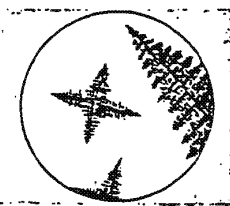


Figure 25.
First crystals of camphor during molecular weight determination.

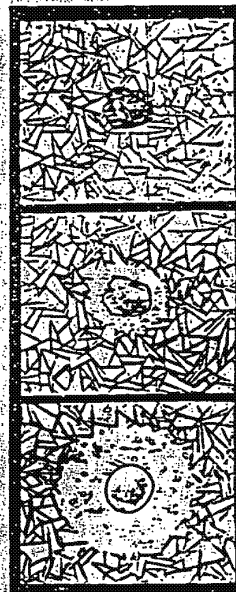


Figure 20.
"Mixed melting point"
between two different substances.



Figure 35.
Three polymorphic forms of luminal during crystallization from the melt.

Plate VII



Figure 36.
Two polymorphs of
benzalazine.



Figure 37.
The central spherulites
transform, on warming,
the unstable form.

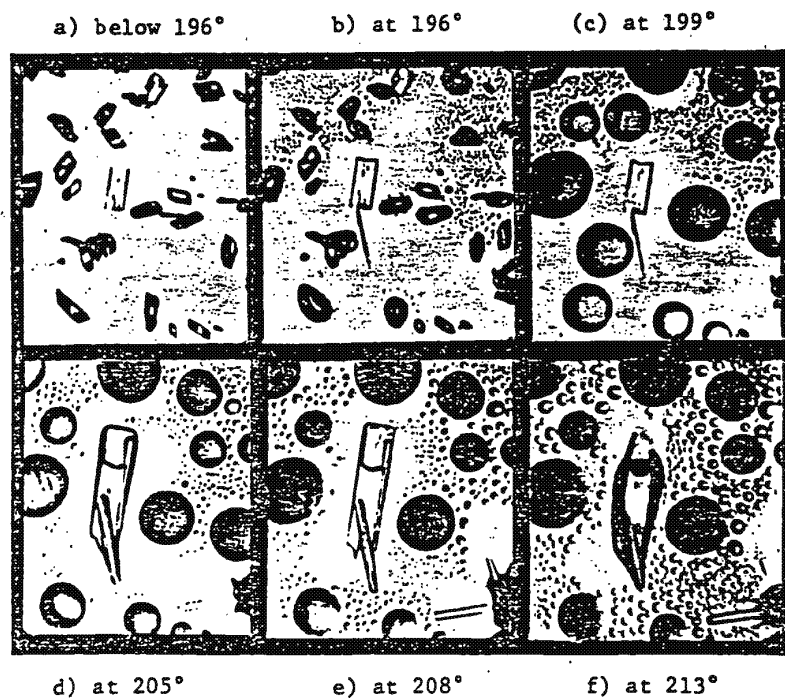


Figure 38. Atophane:
a) oblique-angled unstable
form II (np. 196°) and a right-
angled crystal of form II
(np. 213°); b) and c) melting
of form III; d) and e) growth
of form II through sublimation
and f) melting of form II.

Plate VIII

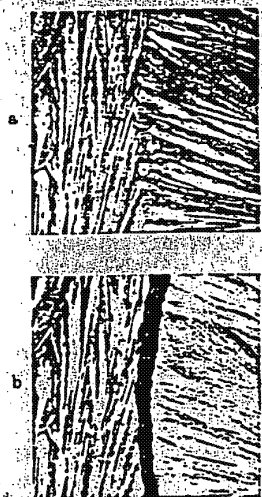


Figure 50. Diphanyl:
acetanilide mixed fusion
a) below the eutectic
temperature b) above
the eutectic temperature

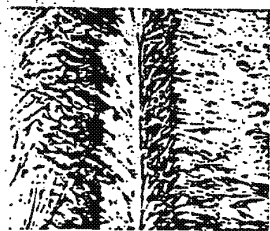


Figure 52. Phenanthrene:
2,4-dinitrophenol mixed
fusion with molecular
addition compound.

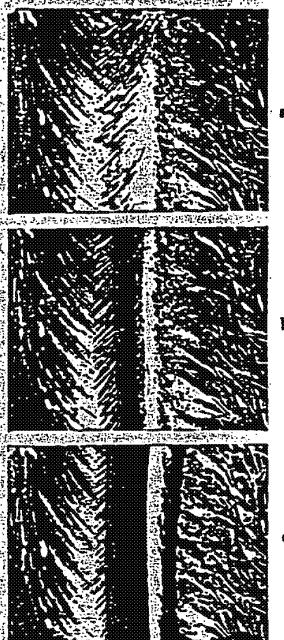


Figure 54.
2,4-dinitrotoluene:
naphthalene mixed fusion
with 1:1 molecular
addition compound.

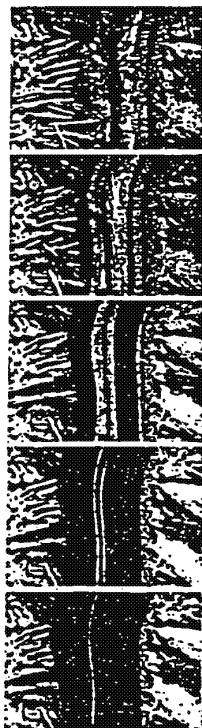


Figure 56.
Two molecular addition
compounds of decamethylene.
dicarboxylic acid: nicotinic
acid amide.



Figure 57.
Two molecular addition
compounds of antipyrene:
hydroquinone.



Figure 61.
Immiscible liquid
phases in the system
acetamide: benzil.



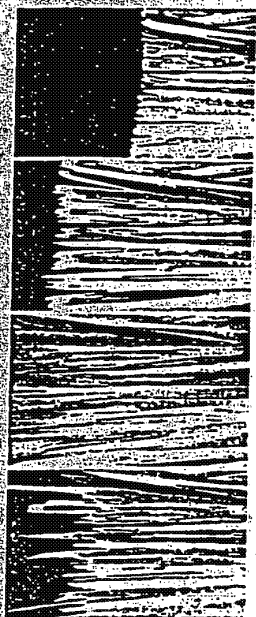


Figure 67. Type I
Roozeboom system for
propyl-3-methylbutyl:
m-butylpropylbarbituric
acid.

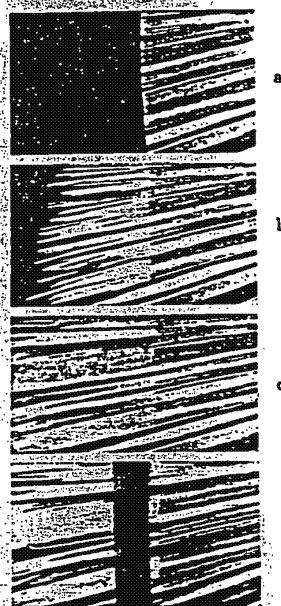


Figure 68. Type III
Roozeboom system for
allylbutyl:dipropyl
barbituric acid.



Figure 70.
Mixed fusion for
dibenzyl: stilbene.



Figure 71. Peritectic reaction in the system dibenzyl: stilbene.



Figure 87.
The stabilized
intermediate phase
for palmitic: stearic
acid.

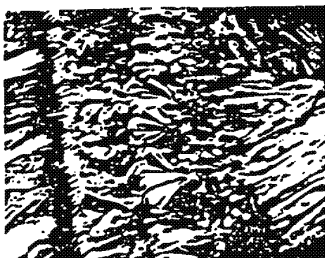


Figure 88.
Two stabilized
intermediate phases
for pentadecane:
stearic acid.

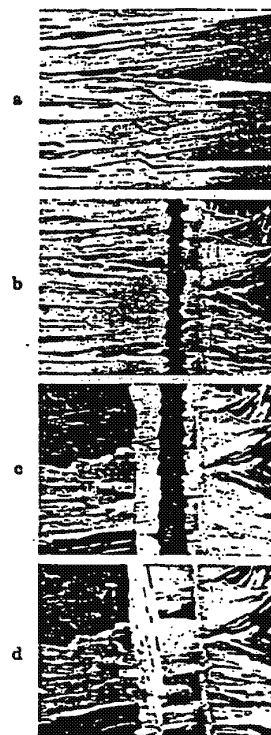


Figure 85.
Mixed fusion between
allylisopropyl:
ethylpropylbarbituric acid.

Plate XII



Figure 89. Homogeneous mixed crystals of NaNO_3 : KNO_3 (80% NaNO_3).



Figure 90. Rearrangement of rings and spirals during melting of NaNO_3 : KNO_3 .



Figure 91. Primary dendrites on slow cooling.

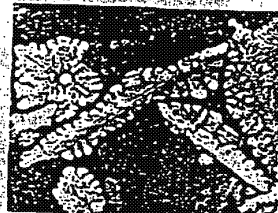


Figure 92. Changes in dendrites on further cooling.



Figure 93. Zoned crystals.



Figure 94. Beginning of melting of zoned crystals with formation of spirals.

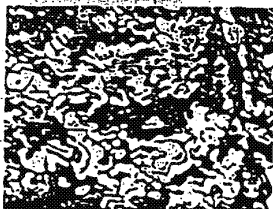


Figure 95. Progress of melting.

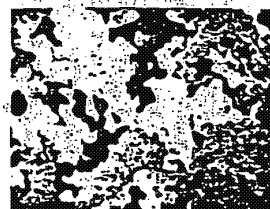


Figure 96. Homogenization after four hours heating at $220-225^\circ$.

Plate XIII



Figure 97. Uniform mixed crystals of phenanthrene: anthracene.

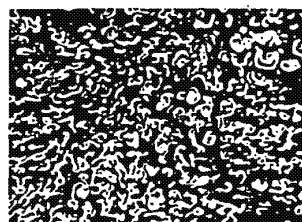


Figure 98. Rearrangement of spirals and rings during melting.



Figure 126. Oriented overgrowths of quinone on pyrogallol.



Figure 127. Oriented overgrowths of camphor on hydroquinone.

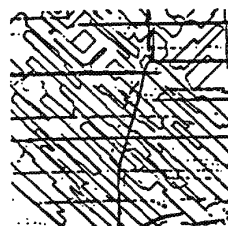


Figure 128. Oriented overgrowths of hexaethylbenzene on chloranil.

Plate XIV



Figure 195. Quasi-eutectic
syncrystallization of
acenaphthene with 40% sulfonal.

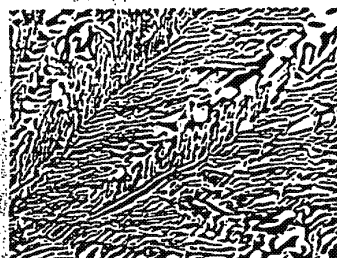


Figure 196. Quasi-eutectic
syncrystallization of
antipyrène with 50% sulfonal.

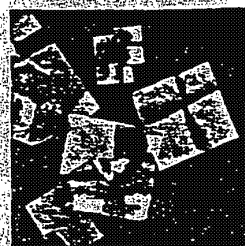


Figure 211. g-Strophanthine
(quabain) decahydrate.



Figure 212. g-Strophanthine
(quabain) octahydrate.



Figure 213. g-Strophanthine
(quabain) dihydrate from
NaCl solution.

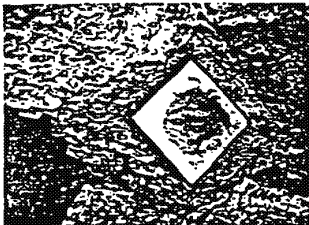
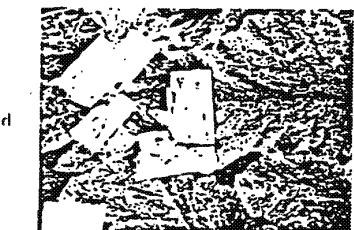
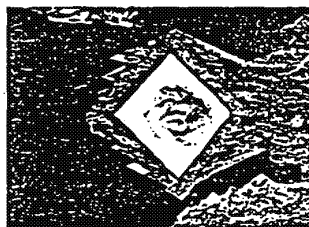
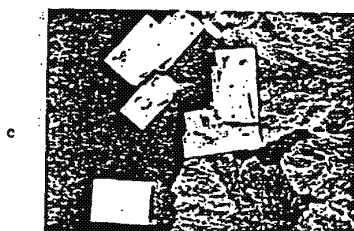
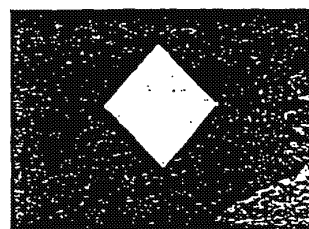
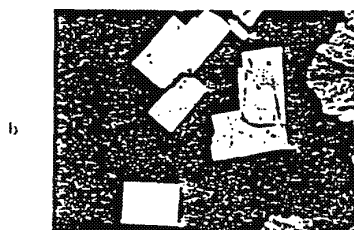
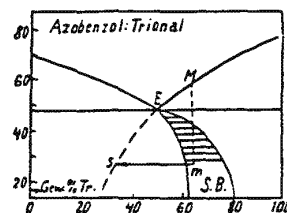
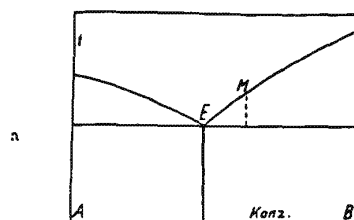


Figure 208. Normal crystallization of a non-eutectic mixture.

Figure 209. Halo formation during crystallization of a supercooled non-eutectic mixture of azobenzene: trianol.

Plate XVI

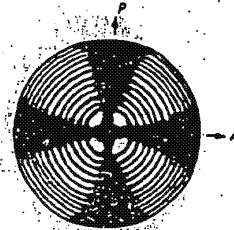


Figure 243. Conoscopic image of a uniaxial crystal.

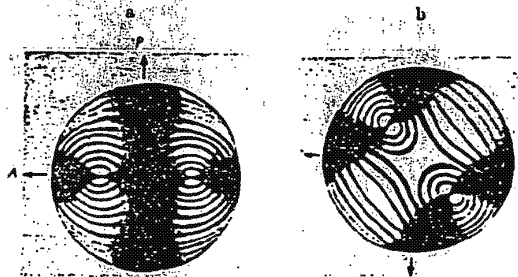


Figure 245. Conoscopic image of a biaxial crystal. a) Extinction position b) 45° from extinction.

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