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PREFACE

KIRK-OTHMER

CONCISE ENCYCLOPEDIA OF CHEMICAL TECHNOLOGY

4th EDITION



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KIRK-OTHMER

CONCISE ENCYCLOPEDIA OF CHEMICAL TECHNOLOGY

4th EDITION

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chemistry. The reaction can be used to reduce the boiling point of esters by exchanging a long-chain alcohol group with a short one, eg, methanol, in the analysis of fats, oils, and waxes.

An industrial example of acidolysis is the reaction of poly(vinyl acetate) with butyric acid to form poly(vinyl butyrate). Often a butyric acid-methanol mixture is used and methyl acetate is obtained as a coproduct.

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ESTERS, ORGANIC

Esters are compounds that, on hydrolysis, yield alcohols or phenols and acids according to the equation: $RA + H_2O \rightleftharpoons ROH + HA$, where R is a hydrocarbon fragment and A is the anion portion of an organic acid.

Nomenclature

The names of esters consist of two words that reflect their formation from an alcohol and a carboxylic acid. According to the IUPAC rule, the alkyl or aryl group of the alcohol is cited first followed by the carboxylate group of the acid with the ending -ate replacing the -ic of the acid. For example, $CH_3CH_2COOCH_3$, the methyl ester of propanoic acid, is called methyl propanoate (or methyl propionate, if the trivial name, propionic acid, is used for the carboxylic acid).

Physical Properties

The physical properties of organic esters vary according to the molecular weight of each component. Lower molecular weight esters are colorless, mobile, and highly volatile liquids that usually have pleasant odors. As the molecular weight increases, volatility decreases and the consistency becomes waxy, then solid, and eventually even brittle, often with formation of lustrous crystals. The melting point of an ester is generally lower than that of the corresponding carboxylic acid. However, the boiling point depends on the chain length of the alcohol component and eventually exceeds that of the acid. Lower molecular weight esters are relatively stable when dry and can be distilled without decomposition. Organic esters are generally insoluble in water, but soluble in various organic liquids. Lower esters are themselves good solvents for many organic compounds. The physical properties of commercially important aliphatic and aromatic organic esters are listed in Table 1.

Chemical Properties

The reactions of esters have been reviewed. Because of the large number of possible acid and alcohol moieties, the chemical properties of esters may differ considerably. Only typical reactions applicable to the majority of esters are described in the following sections.

Hydrolysis. Esters are cleaved (hydrolyzed) into an acid and an alcohol through the action of water. This hydrolysis is catalyzed

Table 1. Physical Properties of Some Common Esters

Ester	Mol wt	n_D^{20}	Bp, °C ^a	Freezing point, °C
methyl formate	60.05	1.344	32	-99.8
ethyl formate	74.08	1.3598	54.3	-80
butyl formate	102.13	1.3889	106	-91.9
methyl acetate	74.08	1.3594	57	-98.1
ethyl acetate	88.1	1.3723	77.1	-83.6
vinyl acetate	86.1	1.3959	72.2	-93.2
propyl acetate	102.13	1.3844	101.6	-92.5
isopropyl acetate	102.13	1.3773	90	-73.4
butyl acetate	116.16	1.3951	126	-73.5
isobutyl acetate	116.16	1.3902	117.2	-98.6
sec-butyl acetate	116.16	1.3877	112	
t-butyl acetate	116.16	1.3855	97	
pentyl acetate	130.18	1.4023	149.3	-70.8
isoamyl acetate	130.18	1.4000	142	-78
sec-hexyl acetate	144.22	1.4014	157	0
2-ethylhexyl acetate	172.26	1.4204	199.3	-93
ethylene glycol diacetate	146.14	1.415	191	-31
2-methoxyethyl acetate	118.13	1.4019	145	-65.1
2-ethoxyethyl acetate	132.16	1.4058	156.4	-61.7
2-butoxyethyl acetate	160.12	1.42	187.8	-32
2-(2-ethoxyethoxy)ethyl acetate	176.21	1.423	217.4	-25
2-(2-butoxyethoxy)ethyl acetate	204.27	1.4265	247	-32.2
benzyl acetate	150.18	1.5232	215.5	-51.5
glyceryl triacetate	218.23	1.4296	258	-78
ethyl 3-ethoxypropionate	146.19		165-172	-50
glyceryl tripropionate	260.3	1.4318	176	-58
methyl acrylate	86.09	1.4040	80.5	<-75
ethyl acrylate	100.11	1.4068	99.8	<-72
butyl acrylate	128.17	1.4185	69	-64.6
2-ethylhexyl acrylate	184.28		130 ^f	-90
methyl methacrylate	100.12	1.4119	100	-48
methyl butyrate	102.13	1.3878	102.3	-84.8
ethyl butyrate	116.16	1.4000	121.6	-100.8
butyl butyrate	144.22	1.4075	166.6	-91.5
methyl isobutyrate	102.13	1.3840	92.6	-84.7
ethyl isobutyrate	116.16	1.3870	110	-88
isobutyl isobutyrate	144.22	1.3999	148.7	-80.7
methyl stearate	298.5	1.457	215	40
ethyl stearate	312.52	1.429	213-215	33.7
butyl stearate	340.58		343	27.5
dodecyl stearate	440.8	1.433		28
hexadecyl stearate	496.91	1.441		57
dimethyl maleate	144.13	1.4409	204	
dimethyl oxalate	111.09	1.4096	185	-41
dimethyl adipate	174.2	1.4283	115	10.3
diethyl adipate	202.25	1.4372	245	-19.8
di(2-ethylhexyl) adipate	370.58	1.4472	214	-60
methyl benzoate	136.15	1.517	199.5	-12.5
ethyl benzoate	150.18	1.505	212.9	-34.2
methyl salicylate	152.15	1.536	223.3	-8.6
ethyl salicylate	166.18	1.522	231.5	1.3
dimethyl phthalate	194.19	1.515	282	-2
diethyl phthalate	222.24	1.499	295	-33
dibutyl phthalate	278.35	1.4911	340	-35
di(2-ethylhexyl) phthalate	390.56	1.486	231	-50
dimethyl isophthalate	194.19	1.5168	124	67
dimethyl terephthalate	194.19		288	140
methyl anthranilate	151.17	1.584	132	24
benzyl cinnamate	238.29		244	39
dimethyl carbonate	90.08	1.3682	90	3
diethyl carbonate	118.13	1.3854	127	-43

^a At 101.3 kPa = 760 mm Hg unless otherwise stated.

by acids or bases. The mechanistic aspects of ester hydrolysis have received considerable attention and have been reviewed.

Enzymatic Hydrolysis. Enzymatic hydrolysis has received enormous attention. The enzymes generally employed are lipases from microorganisms, plants, or mammalian liver. The great advantage of the enzymatic process is its high chemo- and stereoselectivity.

Transesterification. When esters are heated with alcohols, acids, or other esters in the presence of a catalyst, the alcohol or acid groups are exchanged. This process is called transesterification. It is accelerated by the presence of a small amount of acid or alkali. Three types of transesterification are known: (1) exchange of alcohol groups (alcoholysis), (2) exchange of acid groups (acidolysis), and (3) ester-ester interchange (see ESTERIFICATION).

Ammonolysis and Aminolysis. Esters and ammonia react to form amides and alcohols.

If primary or secondary amines are used, *N*-substituted amides are formed. This reaction is called aminolysis.

Reduction. Esters can be reduced to alcohols by catalytic hydrogenation using molecular hydrogen or by chemical reduction.

Reaction of Enolate Anions. In the presence of certain bases, eg, sodium alkoxide, an ester having a hydrogen on the α -carbon atom undergoes a wide variety of characteristic enolate reactions. Mechanistically, the base removes a proton from the α -carbon, giving an enolate that then can react with an electrophile.

Grignard and Related Reactions. Esters react with alkyl magnesium halides in a two-stage process to give alcohols. The reaction involves nucleophilic substitution of R^3 or OR^2 and addition of R^3MgX to the carbonyl group. With 1,4-dimagnesium compounds, esters are converted to cyclopentanols. Lactones react with Grignard reagents and give diols as products. Many other organometallic compounds also react with carbonyl.

α -Halo esters react with aldehydes or ketones in the presence of zinc to form β -hydroxy esters. This is known as the Reformatsky reaction.

Preparation of Acyloins. When aliphatic esters are allowed to react with metallic sodium, potassium, or sodium-potassium alloy in inert solvents, acyloins (α -hydroxyketones) are formed.

Pyrolysis. The pyrolysis of simple esters of the formula $RCOOCR^2R^3CHR^4$ to form the free acid and an alkene is a general reaction that is used for producing olefins.

Carbonylation Reaction. The carbonylation of methyl acetate is an important industrial reaction for producing acetic anhydride.

Substitution, Alkylation, and Rearrangement. The reaction of alkaline phenoxides with alkyl *S*-2-(chloro)- or *S*-2-(mesyloxy)propionate gives optically active *R*-2-aryloxyalkanoic acid esters in good chemical and optical yields (>97% ee). The reaction is utilized in the synthesis of several phenoxy herbicides.

Optically active 2-arylalkanoic acid esters have been prepared by Friedel-Crafts alkylation of arenes with optically active esters, such as methyl *S*-2-(chlorosulfonyl)- or *S*-2-(mesyloxy)propionate, in the presence of aluminum chloride.

The Fries rearrangement of phenol esters gives a mixture of 2- and 4-acylphenols. Similarly, enol esters undergo rearrangement to give the corresponding 1,3-diketones.

Occurrence and Preparation

Currently, most of the simple esters used commercially are of synthetic origin, although esters occur naturally in large quantities in fats, oils, and waxes. Microorganisms produce a complex array of compounds containing the ester linkage, ranging from simple esters to macrocyclic lactones, such as erythromycin, which are important because of their antibacterial properties.

Recovery of naturally occurring esters is accomplished by steam distillation, extraction, pressing, or by a combination of these processes. Synthetic esters are generally prepared by reaction of an alcohol with an organic acid in the presence of a catalyst such as sulfuric acid, *p*-toluenesulfonic acid, or methanesulfonic acid.

Stability and Storage

All organic esters are unstable in the presence of acid or base and nucleophiles such as water or alcohols. However, if stored anhydrous, they are stable. Storage vessels can be constructed of steel, aluminum, or other metallic materials, but plastic storage tanks are unsuitable because the highly lipophilic esters can sometimes permeate into the container boundary and soften or even dissolve it.

The properties of flash point, autoignition temperature, and flammable limit should be considered when an ester is to be handled in any fashion.

Health and Safety Factors

Toxicity. The degree of toxicity of organic esters covers a wide range. These toxicities are usually described in terms of threshold limiting values (TLV), or permissible exposure limits (PEL). Both the TLV and the PEL describe the average concentration over an 8-h period to which a worker may be exposed without adverse effects. The lethal dosages for 50% of the exposed animals, LD_{50} s, are also used as an indicator of the relative toxicity. The LD_{50} s of organic esters for small mammals range between 0.4 and 16 g/kg. The TLVs of organic esters range between 5 and 400 ppm.

When ingested or absorbed, organic esters are likely to be hydrolyzed to the corresponding alcohols and carboxylic acids. Therefore the toxicities of the hydrolysis products should also be considered. Some organic esters are highly volatile and can act as asphyxiant or narcotic. Also, skin absorption and inhalation are among the hazards associated with esters that are volatile or have good solvent action. Because of the high solubility of fats and oils in organic esters, prolonged or repeated exposure to skin can cause drying and irritation.

Acetates generally do not cause any physiological effects unless high exposure occurs since they are usually converted into or occur naturally as metabolites. However, large enough exposure to acetate esters can cause narcotic effects.

Propionates and higher aliphatic esters generally become less toxic as the size of the alkyl carboxylate increases.

The acrylate esters are more physiologically hazardous than their saturated homologues. They are usually lachrymators and irritants, and their toxicities decrease with increasing molecular weight.

Among adipates, oxalates, malonates, and succinates, the adipates are the least toxic.

Benzoate esters, like most organic esters, are not very toxic. They are not absorbed through the skin as rapidly as alkyl esters but are more potent physiologically. They are also moderate skin irritants.

The phthalate esters are one of the most widely used classes of organic esters, and fortunately they exhibit low toxicity.

More information on the toxicities of a range of organic esters is available in the literature.

Exposure Limits. The Occupational Safety and Health Act (OSHA) of 1990 lists a multitude of acetates, phthalates, formates, and acrylates along with the corresponding permissible exposure limits and threshold limit values. If there is potential for exposure to an organic ester for which PEL or TLV data has been identified, then an exposure limit lower than that listed

Regulation and Waste

Waste from production of organic esters is usually not a problem since the method of synthesis often involves a carboxylic acid condensation with an alcohol and the only by-product is water. Any organic remnants lost to the process water can usually be biologically degraded. The biochemical oxygen demand (BOD) or chemical oxygen demand (COD) should be measured if biological treatment is used on the process waste from ester production. Organic ester vapor emitted in processing usually can be burned.

Extensive federal environmental regulations exist that govern organic esters as well as many other substances. These regulations must always be consulted for complete information before using large

amounts of organic esters. State and local regulations must also be met, which in some cases are more stringent than federal regulations.

Uses

Organic esters are used as solvents, plasticizers, in resins, plastics, and coatings, as lubricants, in perfumes, flavors, cosmetics, and soap, as surface-active agents, as medicinals, and as herbicides and pesticides.

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ESTROGENS. See HORMONES.

ETHANOIC ACID. See ACETIC ACID AND DERIVATIVES, ACETIC ACID.

ETHANOL

Ethanol or ethyl alcohol, $\text{CH}_3\text{CH}_2\text{OH}$, is one of the most versatile oxygen-containing organic chemicals because of its unique combination of properties as a solvent, a germicide, a beverage, an antifreeze, a fuel, a depressant, and especially as a chemical intermediate for other organic chemicals. The use of fermentation-derived ethanol as an automotive fuel additive to enhance octane and reduce emissions has seen explosive growth since about 1980 so that it now accounts for over 80% of the 6.0×10^9 L ethanol market in the United States.

Physical Properties

A summary of physical properties of ethyl alcohol is presented in Table 1. Detailed information on the vapor pressure, density, and viscosity of ethanol can be obtained from many references. Ethanol forms binary and ternary azeotropes with a wide variety of other organic chemicals.

Chemical Properties

The chemistry of ethyl alcohol is largely that of the hydroxyl group, namely, reactions of dehydration, dehydrogenation, oxidation, and esterification.

Manufacture

Industrial ethyl alcohol can be produced synthetically from ethylene, as a by-product of certain industrial operations, or by the fermentation of sugar, starch, or cellulose. The synthetic route supplies most of the industrial market in the United States.

The synthetic process employing direct hydration has since the early 1970s completely supplanted the old sulfuric acid process in the United States. This process, the catalytic vapor-phase hydration of ethylene, is now practiced by only three U.S. companies: Union Carbide Corporation (UCC), Quantum Chemical Corporation, and Eastman Chemical Company UCC imports crude industrial ethanol, CIE,

Table 1. Physical Properties of Ethanol

Property	Value
freezing point, °C	-114.1
normal boiling point, °C	78.32
density, d_4^{20} , g/mL	0.7893
refractive index, n_D^{20}	1.36143
$\Delta n_D/\Delta t$, 20-30°C, per °C	0.000404
surface tension, at 25°C, mN/m (= dyn/cm)	23.1
viscosity, at 20°C, mPa·s (= cP)	1.17
solubility in water, at 20°C	miscible
heat of combustion, at 25°C, J/g ^a	29676.69
flammable limits in air, vol %	
lower	4.3
upper	19.0
autoignition temperature, °C	423.0
specific heat, at 20°C, J/(g·°C)	2.42
thermal conductivity, at 20°C, W/(m·K)	0.170

^a To convert J to cal, divide by 4.184.

from SADAF (the joint venture of SABIC and Pecten [Shell]) in Saudi Arabia, and refines it to industrial grade.

Other Methods of Preparation. In addition to the direct hydration process, the sulfuric acid process, and fermentation routes to manufacture ethanol, several other processes have been suggested. These include the hydration of ethylene by dilute acids, the hydrolysis of ethyl esters other than sulfates, the hydrogenation of acetaldehyde, and the use of synthesis gas. None of these methods has been successfully implemented on a commercial scale, but the route from synthesis gas has received a great deal of attention since the 1974 oil embargo.

Recovery and Purification

Various distillation and equipment modifications are used to ensure a pure water azeotrope of ethanol (95% by volume ethanol).

Purification schemes have generally emphasized the following techniques: (1) extractive distillation using water reflux to distill a large share of the impurities and concentrate the crude alcohol-water mixture; (2) efficient fractionation to produce approximately 190 proof alcohol; (3) hydrogenation to convert aldehyde impurities to alcohols, together with the use of chemicals such as inorganic bases and sodium sulfite; and (4) ion-exchange resins or azeotropic distillation to dehydrate 190-proof to 200-proof or absolute alcohol.

Manufacturing Costs

The cost of producing industrial ethanol depends on the location of the manufacturing plant; the design, type, and degree of modernization of equipment; the kind of raw material used; the price paid for the raw material; the relative labor costs represented; the scale of production; and the total investment. On a raw material basis, each kilogram of ethanol requires 0.6 kg ethylene, 2 kg sugar, 3.3 kg corn, or 4 kg molasses. If all other cost factors were equivalent, the economics of fermentation ethanol derived from corn would compare favorably with synthetic ethanol if corn prices were 5.5 times less than ethylene on a weight basis. With ethylene at \$0.44/kg, as it was in 1991, corn would have to be about \$0.08/kg (about \$2/bushel). Corn by-products that are produced by some fermentation plants offset the price of corn to a varying degree depending on their market value. It is expected that ethylene will continue to be the primary raw material for industrial ethanol, because during the early to mid-1990s most of the fermentation ethanol will go to fuel due to the impact of the Clean Air Act, and ethylene prices are not expected to significantly increase because of additional ethylene capacity coming onstream (see ETHYLENE).

Shipment

Commercial ethyl alcohol is shipped in railroad tank cars, tank trucks, 208-L (55-gal) and 19-L (5-gal) drums, and in smaller glass or metal

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