Designation: D 5554 – 95 (Reapproved 2006)

Standard Test Method for Determination of the Iodine Value of Fats and Oils[1]

This standard is issued under the fixed designation D 5554; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the iodine value of fats and oils.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Significance and Use

2.1 This test method is intended for the determination of the iodine value of fats and oils that do not contain conjugated double bonds. The iodine value is a measure of the unsaturation of fats and oils and is expressed in terms of the number of centigrams of iodine absorbed per gram of sample.

3. Apparatus and Reagents

3.1 *Glass-Stoppered Bottles or Wide Mouth Erlenmeyer Flasks*, 500 mL. Wide mouth bottles or flasks are essential if stirring is done by mechanical means.

3.2 *Glass-Stoppered Volumetric Flasks*, conforming to NIST tolerances[2] and accurately calibrated to contain 1000 mL.

3.3 *Pipette*, 20 mL.

3.4 *Two Pipettes*, 25 mL. One pipette is reserved for use with the standard potassium dichromate solution and shall conform to NIST tolerances[3] and be calibrated to deliver 25 mL.

3.5 *Glacial Acetic Acid*, A.C.S. grade. The permanganate test shall be applied to be sure that this specification is met.

3.5.1 Two millilitres of the glacial acetic acid shall be diluted with 10 mL of distilled water and 0.1 mL of 0.1 *N* $KMnO_4$ shall be added. The pink color shall not be entirely discharged within 2 h.

3.6 *Potassium Iodide*, A.C.S. grade.

3.7 *Chlorine*, 99.8 %. Satisfactory commercial grades are available in cylinders, but this gas must be dried by passing through sulfuric acid (sp. gr. 1.84) before introducing into the iodine solution.

3.7.1 Chlorine may be prepared by allowing hydrochloric acid (sp. gr. 1.19) to drop onto potassium permanganate or on a mixture of potassium permanganate and manganese dioxide. The gas thus generated shall be led through a glass tube into sulfuric acid (sp. gr. 1.84) and then into the iodine solution.

3.8 *Carbon Tetrachloride*, A.C.S. grade.

3.9 *Hydrochloric Acid*, A.C.S. grade, sp. gr. 1.19.

3.10 *Soluble Starch*, of suitable sensitivity.

3.10.1 The test for starch sensitivity shall be made by adding, while stirring, 200 mL of boiling water to a paste made with 1 g of starch in a small amount of water. After placing 5 mL of this solution in 100 mL of water, add 0.05 mL of 0.1 *N* iodine solution. The deep blue color produced must be discharged by 0.05 mL of 0.1 *N* sodium thiosulfate.

3.11 *Potassium Dichromate*, A.C.S. grade, shall be finely ground and dried to constant weight at about 110°C before using. (A standard sample of potassium dichromate with a certificate of analysis may be obtained from NIST.[2] This sample is strongly recommended as the primary standard for this test method. The dichromate should be treated as directed in the certificate of analysis accompanying the sample.)

3.12 *Sodium Thiosulfate*—($Na_2S_2O_3 \cdot 5H_2O$), A.C.S. grade.

3.13 *Iodine*, A.C.S. grade.

3.14 *Potassium Iodide Solution*, prepared by dissolving 150 g in distilled water and making up to 1 L.

3.15 *Starch Indicator Solution*, shall be prepared by making a homogeneous paste of 10 g of soluble starch in cold distilled water and adding to this 1 L of boiling distilled water with rapid stirring; cool. Salicylic acid (1.25 g/L) shall be added to preserve the indicator. If long storage is required, the solution shall be kept in a refrigerator at 4 to 10°C (40 to 50°F). Fresh indicator shall be prepared when the end-point of the titration from blue to colorless fails to be sharp.

3.16 *Standard Potassium Dichromate Solution*, 0.1 *N*, shall be prepared by dissolving 4.9035 g of finely ground and dried potassium dichromate in distilled water in the 1000 mL volumetric flask and making to volume at 25°C.

[1] This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.08 on Fats and Oils. This test method was developed in cooperation with the American Leather Chemists Assn. (Method H 32-1957).

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[2] Available from National Institute of Standards and Technology, (NIST), Gaithersburg, MD 20899.

[3] Wijs Solution (Iodine Monochloride Solution) may be purchased commercially.

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3.17 *Sodium Thiosulfate Solution*, 0.1 *N*, prepared by dissolving 24.8 g of sodium thiosulfate in distilled water and diluting to 1 L.

3.17.1 *Standardization of the Thiosulfate*—Pipette 25 mL of the standard dichromate solution into Erlenmeyer flask or bottle. Add 5 mL of hydrochloric acid, 10 mL of potassium iodide solution and rotate to mix. Allow to stand for 5 min and then add 100 mL of distilled water. Titrate with sodium thiosulfate solution, shaking continuously, until the yellow color has almost disappeared. Add 1 to 2 mL of indicator and continue titration, adding the thiosulfate solution slowly, until the blue color has just disappeared. The strength of the sodium thiosulfate solution is expressed in terms of its normality.

$$\text{Normality of } Na_2S_2O_3 \text{ solution} = \frac{2.5}{\text{mL } Na_2S_2O_3 \text{ solution required}} \quad (1)$$

3.18 *Wijs Solution*[3], prepared by dissolving 13.0 g of iodine in 1 L of glacial acetic acid. Gentle heat may be necessary to promote solution. Cool and remove a small quantity (100 to 200 mL) and set aside in a cool place for future use. Pass dry chlorine gas into the iodine solution until the original titration is not quite doubled. A characteristic color change takes place in the Wijs solution when the desired amount of chlorine has been added. This may be used to assist in judging the end point. A convenient procedure is to add a small excess of chlorine and bring back to the desired titration by addition of some of the original iodine solution that was taken out at the beginning. The original iodine solution and the finished Wijs solution shall both be titrated with $Na_2S_2O_3$ solution as directed in 4.6.

NOTE 1—All Wijs solutions are sensitive to temperature, moisture, and light. Store them in a cool and dark place and never allow them to come to a temperature much above 30°C.

4. Procedure

4.1 Melt the sample, if not already completely liquid, and filter through filter paper to remove any impurities and the last traces of water. (Care shall be taken not to exceed 10° to 15°C above the sample melting point during melting and filtering.) The sample shall be absolutely dry.

4.2 Accurately weigh the sample into a 500-mL flask or bottle to which have been added 20 mL of CCl_4. The weight of sample must be such that there will be an excess of Wijs solution of 50 to 60 % of the amount added, that is, 100 to 150 % of the amount absorbed. Table 1 is a convenient guide for selecting the size of sample.

4.3 Pipette 25 mL of the Wijs solution into the flask containing the sample and swirl to ensure an intimate mixture.

4.4 Prepare at least two blank determinations and conduct with each group of samples simultaneously and similar in all respects.

TABLE 1 Guide for Selecting Sample Size

Iodine Value	Sample Weight, g		Weighing Accuracy, g
	100 % Excess	150 % Excess	
<3	10	10	±0.001
3	10.5760	8.4613	0.005
5	6.3460	5.0770	0.0005
10	3.1730	2.5384	0.0002
20	1.5865	0.8461	0.0002
40	0.7935	0.6346	0.0002
60	0.5288	0.4231	0.0002
80	0.3966	0.3173	0.0001
100	0.3173	0.2538	0.0001
120	0.2644	0.2115	0.0001
140	0.2266	0.1813	0.0001
160	0.1983	0.1587	0.0001
180	0.1762	0.1410	0.0001
200	0.1586	0.1269	0.0001

4.5 Store the flasks in a dark place for 30 min at a temperature of 25 ± 5°C. (Store samples with an iodine value above 150, such as linseed and perilla oils, for 1 h.)

4.6 From storage, remove the flasks and add 20 mL of KI solution followed by 100 mL of distilled water. Titrate the solution with 0.1 *N* $Na_2S_2O_3$ solution, adding gradually and with constant shaking. (Mechanical stirring is very satisfactory for agitating during the addition of thiosulfate.) Continue the titration until the yellow color has almost disappeared, add 1 to 2 mL of starch indicator solution, and continue the titration until the blue color has just disappeared.

5. Calculation

5.1 Calculate the iodine value as follows:

$$\textit{iodine value} = \frac{(B - S) \times N \times 12.69}{\textit{weight of sample}} \quad (2)$$

where:

B = titration of blank,
S = titration of sample, and
N = normality of $Na_2S_2O_3$ solution.

6. Precision and Bias

6.1 This test method is adopted from the procedures of the American Leather Chemists Association where it has long been in use and was approved for publication before the inclusion of precision and bias statements was mandated. The original interlaboratory test data are no longer available. The user is cautioned to verify by the use of reference materials, if available, that the precision and bias (or reproducibility) of this test method is adequate for the contemplated use.

7. Keywords

7.1 fats and oils; iodine value; leather; unsaturation

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