



Designation: D 5554 – 95 (Reapproved 2001)

Standard Test Method for Determination of the Iodine Value of Fats and Oils¹

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 approval. 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 tolerances² 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 tolerances³ 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*—($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$), 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.