

Designation: D5555 – 95 (Reapproved 2006)

Standard Test Method for Determination of Free Fatty Acids Contained in Animal, Marine, and Vegetable Fats and Oils Used in Fat Liquors and Stuffing Compounds¹

This standard is issued under the fixed designation D5555; 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 free fatty acid content 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 free fatty acids contained in animal, marine, and vegetable fats and oils for the purpose of quality assurance.

3. Apparatus and Reagents

- $3.1~\mbox{Oil Sample Bottles},\,115~\mbox{or}\,230~\mbox{mL}$ (4 or 8 oz) or 250 mL Erlenmeyer flasks.
- 3.2 Ethyl Alcohol, 95 %. The alcohol shall give a definite, distinct, and sharp end-point with phenolphthalein and shall be neutralized with alkali to a faint but permanent pink color just before using. (Isopropanol, 99 %, may be used as an alternate solvent with crude and refined vegetable oils.)
- 3.3 Phenolphthalein Indicator Solution, $1\ \%$ in $95\ \%$ alcohol.
 - 3.4 Sodium Hydroxide Solution, accurately standardized.

4. Procedure

- 4.1 Use Table 1 to determine the quantities to be used with various ranges of fatty acids.
- 4.2 The sample shall be entirely liquid and well mixed before weighing. Then weigh the designated sample size into

TABLE 1 Suggested Quantity of Material to Test

| Sample, g | Alcohol, mL | Normality of Alkali | Free Fatty Acid Range (%) |
|-----------------|-------------|---------------------|------------------------------|
| 56.4 ± 0.2 | 50 | 0.1 | 0.0 to 0.2 |
| 28.2 ± 0.2 | 50 | 0.1 | 0.2 to 1.0 |
| 7.05 ± 0.05 | 75 | 0.25 | 1.0 to 30.0 |
| 7.05 ± 0.05 | 100 | 0.25 or 1.0 | 30.0 to 50.0 |
| 3.525 ± 0.001 | 100 | 1.0 | 50.0 to 100.0 |

an oil-sample bottle or Erlenmeyer flask; add to that the specified amount of hot, neutralized alcohol and 2 mL of indicator.

4.3 Shaking vigorously, make the titration with alkali to the appearance of the first permanent pink color of the same intensity as that of the neutralized alcohol before adding the sample. The color shall persist for 30 s.

5. Calculation and Report

5.1 Report the free fatty acids in percent, by weight:

% free fatty acids =
$$\frac{\text{mL of } alkali \times N \times 28.2}{\text{weight of sample}}$$
(1)

- 5.2 Reference this test method in the test report.
- 5.3 The free fatty acids are frequently expressed in terms of acid value instead of percent free fatty acids. The acid value is defined as the number of milligrams of KOH necessary to neutralize 1 g of sample. To convert percent free fatty acids (as oleic) to acid value, multiply the former by 1.99.

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; free fatty acids; leather

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 30-1957).

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