



Designation: D 1358 – 86 (Reapproved 1995)^{e1}

Standard Test Method for Spectrophotometric Diene Value of Dehydrated Castor Oil and Its Derivatives¹

This standard is issued under the fixed designation D 1358; 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.

^{e1} NOTE—Unit of measurement statement and Keywords were added editorially in May 1995.

1. Scope

1.1 This test method covers the determination of the spectrophotometric diene value as a measure of the content of conjugated dienoic acids in dehydrated castor oil and its derivatives. Due to the high linoleic acid content in dehydrated castor oil and its derivatives, the absolute conjugated dienoic acid content cannot be determined by this test method.

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.* Specific hazard statements are given in Section 6.

2. Terminology

2.1 *Description of Term Specific to This Standard:*

2.1.1 *spectrophotometric diene value—under the conditions of this test method, the percent of conjugated dienoic acids.*

3. Significance and Use

3.1 In the process of dehydrating castor oil, a double bond is formed in the ricinoleic acid to make either conjugated linoleic or normal linoleic fatty acid segments in the oil. This test method attempts to measure the amount of conjugated unsaturation by means of the absorbance at a specific wavelength which is in the ultraviolet range. Since some absorption is due to linoleic acid, a correction factor is included in the calculation. This test method is only applicable to dehydrated castor oil products.

¹ This test method is under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications, and is the direct responsibility of Subcommittee D01.32 on Drying Oils.

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4. Apparatus

4.1 *Spectrophotometer*—Ultraviolet photoelectric spectrophotometer,² covering a spectral range from 200 to 350 nm, with a wavelength scale readable to 0.1 nm, and equipped with an absorption cell compartment³ for holding 10.00-mm cells.

4.2 *Absorption Cells*, quartz, matched pairs of lengths 10.00 $\pm$ 0.05 mm. The cells in a pair, when filled with water or isooctane, shall match within 0.01 absorbancy unit (Note 1). The cells may be the 10.00-mm nondemountable type made of quartz, or the demountable type consisting of a chemically resistant glass⁴ cell body of outside diameter of about 22 mm with centered-ground glass stopper, threaded metal caps, polished crystalline quartz windows, and cork gaskets.

NOTE 1—If the cells do not match within 0.01 absorbancy unit, they may be filled with water or isooctane and calibrated against each other at the specified wavelength. The necessary correction in absorbancy units is then applied where necessary. Highest precision is obtained by the method of reversing cells. The absorbancy of the solution is read against the solvent blank. The cells are then cleaned and the solvent blank put in the cell previously used for the solution and the solution in the cell previously used for the blank and the absorbancy measured. The two absorbancies are then averaged to obtain A_s , the observed absorbancy. The cells should be rinsed with solvent, water, concentrated nitric acid, water, and solvent, in that sequence, before each reading.

4.3 *Filter Tube*, 810 by 45 mm (32 by 1¾ in.).

4.4 *Cork Stopper* covered with aluminum foil to fit loosely in the top of the filter tube.

4.5 *Dropping Bottle*, stoppered, for weighing the samples.

4.6 *Volumetric Flasks*, glass-stoppered, having capacities of 100 mL, 50 mL, and 25 mL.

² The Beckman Model DU photoelectric quartz spectrophotometer, Catalog No. 2500 DU, Beckman Laboratories, S. Pasadena, CA, has been found satisfactory for this purpose.

³ The cell compartment assembly, as Catalog No. 2510, and the ultraviolet accessory set, as Catalog No. 2501, that includes the No. 2230 hydrogen discharge lamp, the No. 2511 adapter, No. 23-10-10-89 cells, and No. 2504 cover, both available from Beckman Laboratories, S. Pasadena, CA, have been found satisfactory for this purpose.

⁴ Borosilicate glass has been found satisfactory for this purpose.



4.7 *Volumetric Pipets*, precision grade, assorted sizes, for making dilutions when required.

5. Reagents and Materials

5.1 *Silica Gel*, 28 to 200-mesh.⁵

5.2 *Glass Wool*.

5.3 *Isooctane (2,2,4 Trimethylpentane)*—Pure or spectroscopic grades of *isooctane* are satisfactory. Check the absorbancy of a 10-mm layer of the *isooctane* against distilled water at 233 nm. The absorbancy compared with distilled water set at zero absorbancy shall not be more than 0.070 at 233 nm. If the *isooctane* does not meet these requirements, purify it as follows: Place about 90 mm (3½ in.) of glass wool above the lower end of the filter tube. Add about 500 mm (20 in.) of silica gel and about 50 mm (2 in.) of glass wool. Fasten the tube vertically to a ring stand and place a funnel and 2-L amber bottle under the tube. Pour the *isooctane* slowly into the tube, filling about three-fourths full, and allow the *isooctane* to filter through the silica gel. Renew the silica gel in the tube as often as necessary to yield *isooctane* conforming to the transmission limit given above. A constant flow can be maintained by feeding the *isooctane* from a 500-mL glass-stoppered separatory funnel, the tip of which is below the surface of the *isooctane* in the filter tube. The 2-L amber bottle used for storing the purified *isooctane* should be rinsed with a little of the first eluate from the column, and the rinse should be discarded. An aluminum-lined screw cap should be used to close the bottle for storage.

6. Hazards

6.1 *Isooctane* is an extremely flammable solvent with a flash point of – 10°C (10°F). Keep away from heat and open flame. Can react vigorously with reducing materials. See supplier's Material Safety Data Sheet for further information.

7. Calibration of Spectrophotometer

7.1 Operational instructions for spectrophotometers vary with different models. Consult the manufacturer's literature for establishing optimum conditions for the specific instrument used.

⁵ Silica gel available from Davison Chemical Co., Baltimore, MD, as Code 11-08, or the equivalent, has been found satisfactory for this purpose.

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8. Procedure

8.1 Weigh by difference into a 100-mL volumetric flask, to 0.1 mg, 90 to 130 mg of the sample. Add about 75 mL of purified *isooctane*.

8.2 Rotate the flask and warm the contents, if necessary, to dissolve the specimen completely. Cool to room temperature, and allow to stand at least 15 min to attain temperature equilibrium. Dilute to volume with purified solvent and mix thoroughly.

8.3 Make necessary dilutions, in no greater than ten-fold steps, to give a final concentration of about 0.01 g of the sample per litre. Measure the absorbancy of the solution at 233 nm (Note 2). Use a matched cell containing purified solvent only for the blank cell. Take several readings and calculate the mean. The observed absorbancy readings should lie between 0.35 and 0.55; otherwise change the weight or the dilution to give the required reading.

NOTE 2—For highest precision, when making dilutions, allow sufficient time for the solutions to reach room temperature.

9. Calculation and Report

9.1 Calculate the percent of conjugated dienoic acids, C_2 , as follows:

$$C_2, \% = 8.4 [(A_s/bc) - a_o]$$

where:

a_o = 0.07 for esters,

= 0.03 for acid,

A_s = observed absorbancy at 233 nm,

b = cell length, mm, and

c = sample per litre of the final dilution used for the absorption measurement, g.

9.2 Report the percent of conjugated dienoic acids to three significant figures.

10. Precision

10.1 *Repeatability*—Two results obtained by the same operator on the same apparatus should not differ from the mean by more than ± 0.39 .

10.2 *Reproducibility*—Two results obtained by operators in different laboratories should not differ from the mean by more than ± 1.26 .

10.3 *Bias*—Bias has not been determined.

11. Keywords

11.1 dehydrated castor oil; diene value; UV—ultra violet absorption