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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for 2,6-Ditertiary-Butyl Para Cresol and 2,6-Ditertiary-Butyl Phenol in Electrical Insulating Oil by Infrared Absorption [1]

This standard is issued under the fixed designation D 2668; 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 weight percent of 2,6-ditertiary-butyl paracresol and 2,6-ditertiary-butyl phenol in new or used electrical insulating oil in concentrations up to 0.5 % by measuring its absorbance at the specified wavelengths in the infrared spectrum.

1.2 *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. Referenced Documents

2.1 *ASTM Standards:*

D 923 Test Method for Sampling Electrical Insulating Liquids [2]

D 2144 Test Methods for Examination of Electrical Insulating Oils by Infrared Absorption [2]

D 3487 Specification for Mineral-Insulating Oil Used in Electrical Apparatus [2]

3. Significance and Use

3.1 The quantitative determination of 2,6-ditertiary-butyl paracresol and 2,6-ditertiary-butyl phenol in a new electrical insulating oil measures the amount of this material that has been added to the oil as protection against oxidation. In a used oil it measures the amount remaining after oxidation products have reduced its concentration. The test is also suitable for manufacturing control and specification acceptance.

3.2 When an infrared spectrum is obtained of an electrical insulating oil inhibited with either of these compounds there is an increase in absorbance of the spectrum at several wavelengths (or wavenumbers). 2,6 ditertiary-butyl paracresol produces pronounced increases in absorbance at 2.72 µm (3680 cm^{-1}), and 11.63 µm (860 cm^{-1}). 2,6 ditertiary-butyl phenol produces pronounced increases in absorbance at 2.72 µm (3680 cm^{-1}) and 13.33 µm (750 cm^{-1}).

3.3 When making this test on other than a highly oxidized oil or when using a double-beam spectrophotometer, it has been found convenient to obtain the spectrum between 2.5 µm (4000 cm^{-1}) and 2.9 µm (3450 cm^{-1}) because the instrument is compensated for the presence of moisture and the band is not influenced by intermolecular forces (associations). However, when testing a highly oxidized oil or when using a single-beam instrument better results may be obtained if the scan is made between 10.90 µm (918 cm^{-1}) and 14.00 µm (714 cm^{-1}).

3.4 Increased absorption at 11.63 µm (860 cm^{-1}) or 13.33 µm (750 cm^{-1}) or both, will identify the inhibitor as 2,6-ditertiary-butyl paracresol or 2,6-ditertiary-butyl phenol respectively (Note 1).

NOTE 1—The absorbance at 750 cm^{-1} for 2,6-ditertiary-butyl phenol and at 860 cm^{-1} for 2,6-ditertiary-butyl paracresol for equal concentrations will be in the approximate ratio of 2.6 to 1.

4. Apparatus

4.1 With equipment description referring to compliance, the equipment shall be in accordance with Section 6 of Test Methods D 2144.

5. Sampling

5.1 Obtain the sample in accordance with Test Method D 923.

6. Calibration and Standardization

6.1 When the manufacturer of the oil is known and the base oil is available, use it to prepare the standards. For oils of unknown origin use base oils which meet the requirements of Specification D 3487. Some base oils may provide a better match than others and therefore it is desirable to have several available.

6.2 Prepare standards containing between 0.05 and 0.4 weight percent of 2,6-ditertiary-butyl paracresol or 2,6-ditertiary-butyl phenol dissolved in an uninhibited base oil. Obtain a spectrum, at the desired band, of each standard in accordance with Test Methods D 2144. Cells with a standard path length of 0.3 to 1.0 mm are recommended. Other path lengths may be found more suitable for different instruments or particular wave lengths. Other sample path lengths may be used provided the instrument sensitivity can be adjusted to compensate for this change. The dip in the curve for the inhibited oil should provide a distinctive increase in the absorbance at the critical wavelength or frequency (Note 2). Repeat the procedure on each of the standards making three

[1] This test method is under the jurisdiction of ASTM Committee D-27 on Electrical Insulating Liquids and Gases and is the direct responsibility of Subcommittee D27.03 on Analytical Tests.

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[2] *Annual Book of ASTM Standards*, Vol 10.03.

scans on each standard. Record all settings of the spectrophotometer used in obtaining the respective spectra (Note 3).

NOTE 2—Where desired, a chart having a non-linear wavelength scale as the abscissa may be used.

NOTE 3—In making these tests, transmission-scaled charts may be used, but in this case special rulers and nomographs or logarithmic tables will be necessary for determining the intensity measurements.

6.3 The quantitative determination is made from the following equation which is derived from Beer's law:

$$\text{Absorbance} = A - A_o$$

where:

A_o = absorbance units of base oil, and

A = absorbance units of oil containing 2,6-ditertiary-butyl paracresol or 2,6-ditertiary-butyl phenol.

6.4 Designate the point of maximum absorbance of the absorbance band as Point A. Draw a tangent to the spectrum curve and a second line through Point A perpendicular to the absorbance lines, as shown in Fig. 1. Designate the intersection of these two lines as Point A_o. Read the values of absorbance at these points on the charts of the three scans made on each test specimen to the nearest 0.001 absorbance unit (with the aid of a reading glass) and subtract the values of A_o from those of A. When the average of the three values for each of the specially prepared test specimens is plotted against the concentration, a straight line is obtained. The best straight line through the calibration data points should be drawn or determined by linear regression analysis. This is the calibration curve from which the unknown concentration of the 2,6-ditertiary-butyl paracresol or 2,6-ditertiary-butyl phenol in a test specimen may be determined. One such calibration curve is shown as Fig. 2. Fig. 3 and Fig. 4 illustrate sections of differential scans.

6.5 When frequent determinations are made on a routine basis, periodic checks of one or more standards are recommended, since the characteristics of electronics components in spectrophotometers change with time. If the absorbance of the standards differ from the calibration curve by more than the limits given in 8.2, a new curve should be

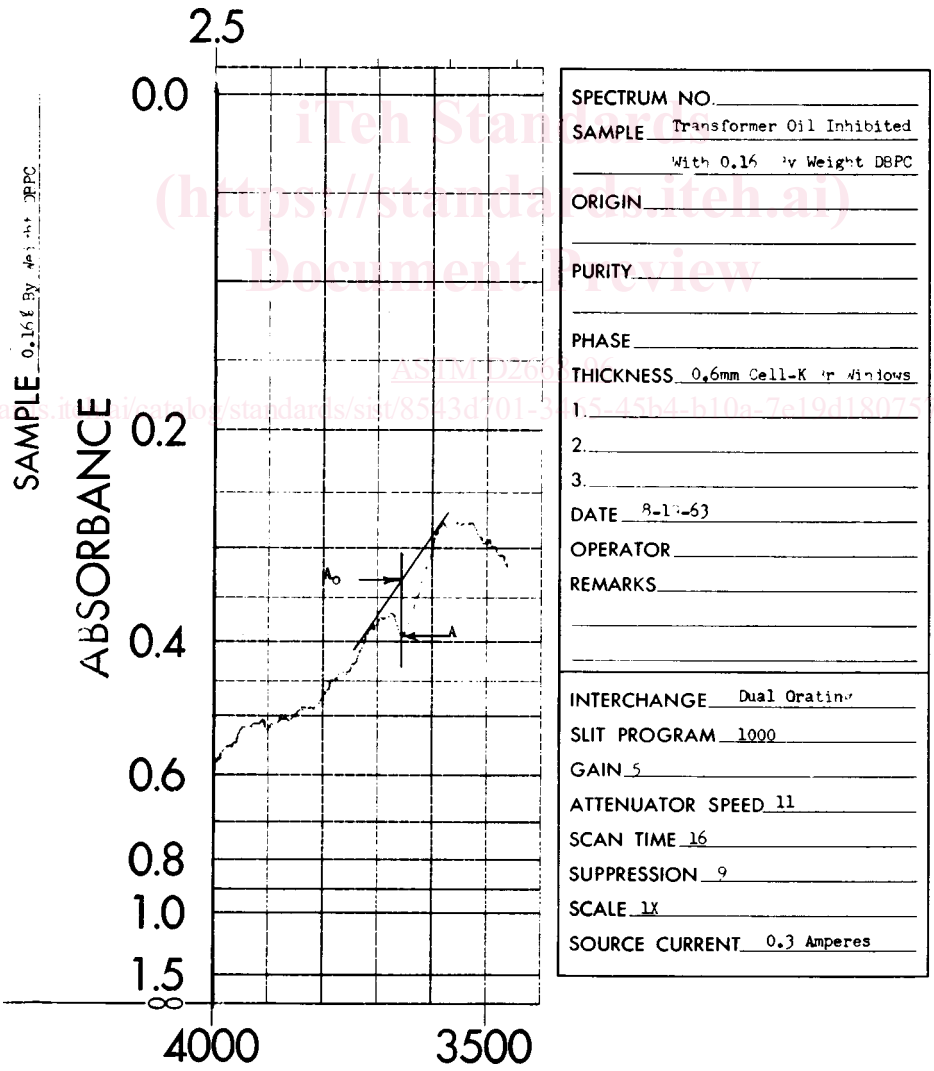

FIG. 1 Spectrum of an Electrical Insulating Oil Inhibited with 2,6-Ditertiary-Butyl Paracresol Showing Location Points A_o and A