This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D2668 – 07 (Reapproved 2021)

Standard Test Method for 2,6-*di-tert*-Butyl- *p*-Cresol and 2,6-*di-tert*-Butyl Phenol in Electrical Insulating Oil by Infrared Absorption[1]

This standard is issued under the fixed designation D2668; 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 (DBPC) and 2,6-ditertiary-butyl phenol (DBP) 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D923 Practices for Sampling Electrical Insulating Liquids

D2144 Practices for Examination of Electrical Insulating Oils by Infrared Absorption

D3487 Specification for Mineral Insulating Oil Used in Electrical Apparatus

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 has 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 (3650 cm^{-1}), and 11.63 µm (860 cm^{-1}). 2,6 ditertiary-butyl phenol produces pronounced increases in absorbance at 2.72 µm (3650 cm^{-1}) and 13.42 µm (745 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 13.42 µm (745 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 745 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 Practices D2144. Accordingly, the use of Fourier-transform rapid scan infrared (FTIR) spectrophotometers is permitted by reference to that test method.

5. Sampling

5.1 Obtain the sample in accordance with Practices D923.

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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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 D3487. 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. Alternatively, the range of prepared standards may be increased to 0.5 weight percent if certain oils to be investigated are believed to contain greater amounts of inhibitor. Obtain a spectrum, at the desired band, of each standard in accordance with Practices D2144. 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 3). Repeat the procedure on each of the standards making at least three scans on each standard. (See Note 2) Record all settings of the spectrophotometer used in obtaining the respective spectra (Note 4).

NOTE 2—The current method precision is based on manually determined results where exactly three scans were determined for each standard. Newer instruments are capable of automatically performing scans much more rapidly, which can reduce the variability of results determined. In such cases, it is recommended that the number of scans be increased to statistically compensate for any outliers. Laboratories will need to determine the minimum number of scans that should be used in their instrument standardization and test specimen analyses to satisfy their testing needs.

NOTE 3—Where desired, a chart having a non-linear wavelength scale as the abscissa may be used.

NOTE 4—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. Alternatively, instrument software capable of recording all settings of the spectrophotometer used in obtaining the respective spectra, may be used.

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 *Manual Plotting Routine for Generating Calibration Curve*—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. Figs. 5-7 show FTIR scans of uninhibited, a similar oil with 0.55 % DBPC and the resulting FTIR differential scan.

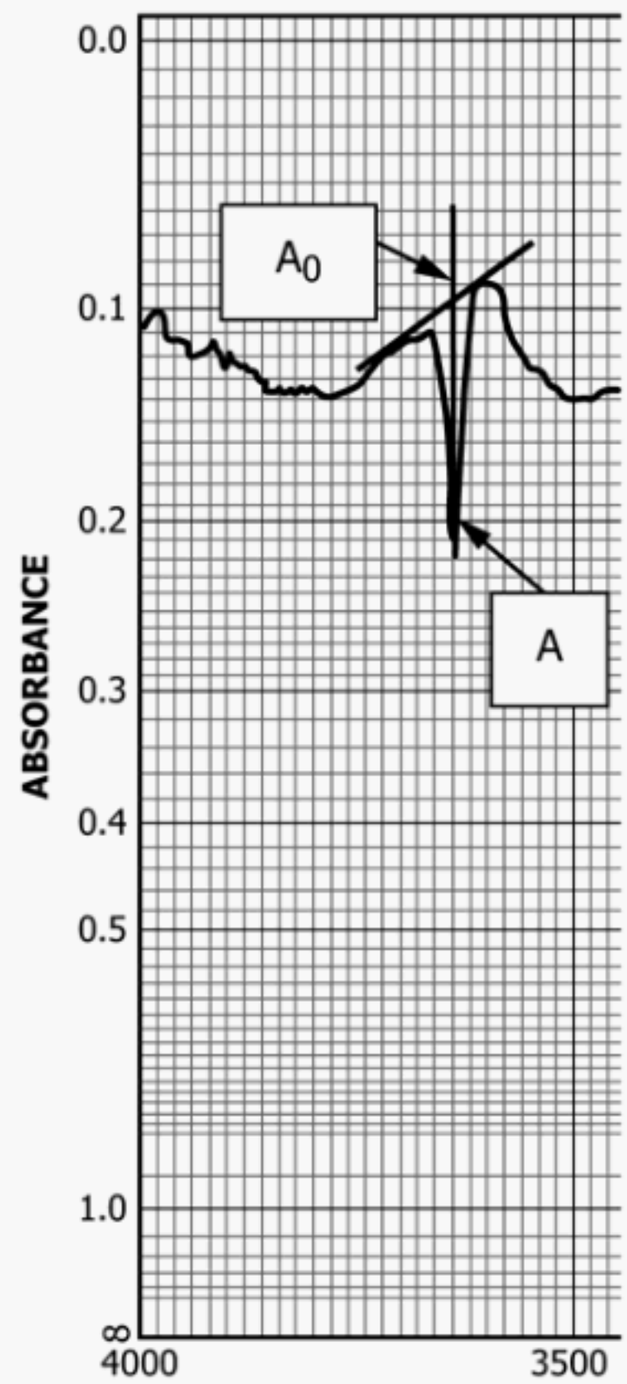


FIG. 1 Spectrum of an Electrical Insulating Oil Inhibited with 2,6-Ditertiary-Butyl Paracresol Showing Location Points A_o and A

6.4.1 *Automatic Plotting Routine for Generating Calibration Curve*—For instruments capable of automatically generating a standard calibration curve, follow manufacturer's instructions. Perform at least three scans for each standard analyzed across the calibration range of interest (see Note 2). Develop a calibration curve which has a minimum correlation value of 0.99 to ensure the linearity of the calibration curve.

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 obtained. Some laboratories have developed tighter limits than these. A new curve should also be obtained whenever there is

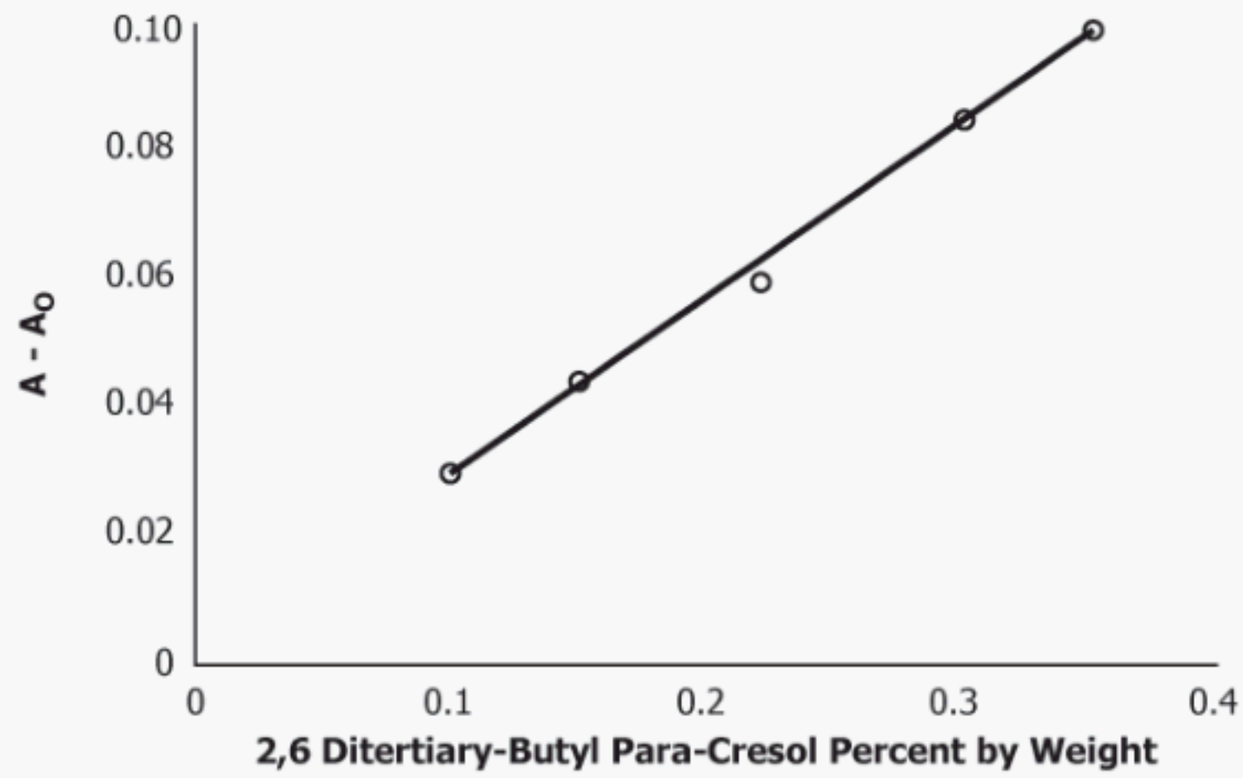


FIG. 2 Calibration Curve for Determining the Percent by Weight of 2,6-Ditertiary-Butyl Paracresol in Electrical Insulating Oil

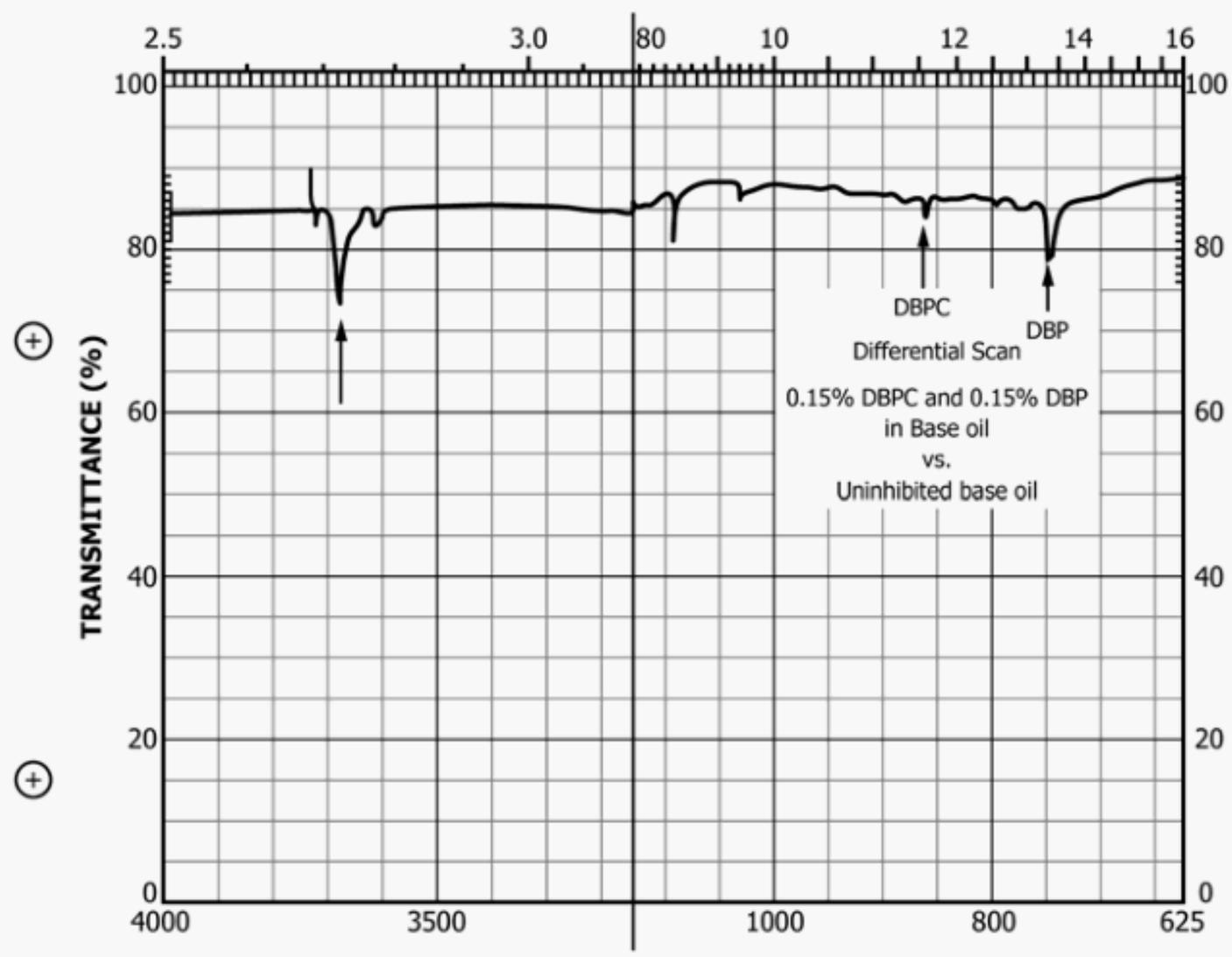


FIG. 3 Section of Differential Scan-A

a change in operating conditions, such as a change in light source, scan speed, and so forth.

NOTE 5—It is recommended that the solutions of known concentration be stored in amber-colored bottles for a period not to exceed one year in order to facilitate a quick check of the characteristics of the spectrophotometer in relation to the calibration curve.

7. Procedure

7.1 Using the quantitative scan mode, make at least three scans (see Note 2) of the test specimen on which the determination of the 2,6-ditertiary-butyl paracresol or 2,6-ditertiary-butyl phenol content is to be made, at the desired band. Do this in accordance with Practices D2144, except that cells having path lengths as specified in 6.2 are preferred. Use the same cell that was used in obtaining the spectra for the calibration curve. For the Differential Method, fill the reference cell with an appropriate base oil free of oxidation inhibitors. Use a spectrophotometer which meets the requirements specified in Section 6 of Practices D2144 and instrumental conditions identical to those used in obtaining the spectra for the calibration curve. In particular, it should be noted that Fourier-transform rapid scan infrared spectrophotometers may also be used and in some cases are preferred for the measurement.

7.2 For routine tests, a single scan of the test specimen is adequate as long as the instrument is capable of meeting the criteria of 8.2 using single scans to replace average values. For referee purposes, use the average of three scans of the test

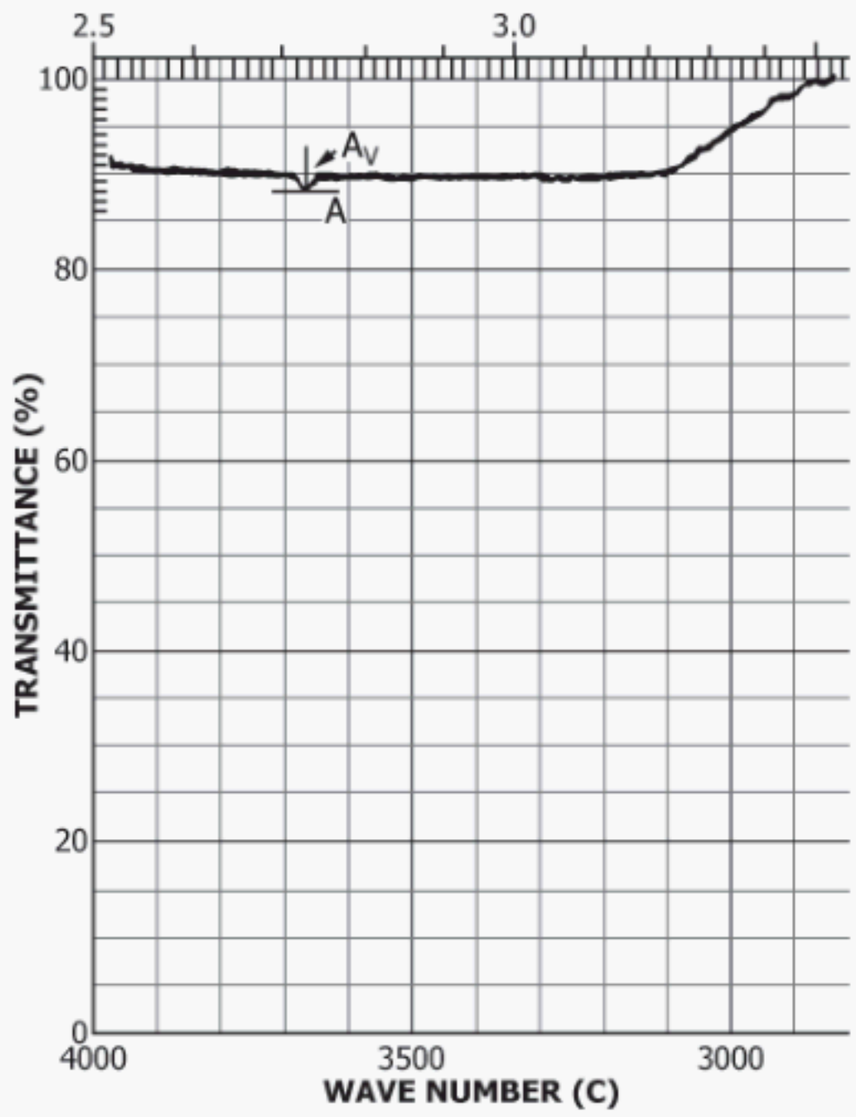
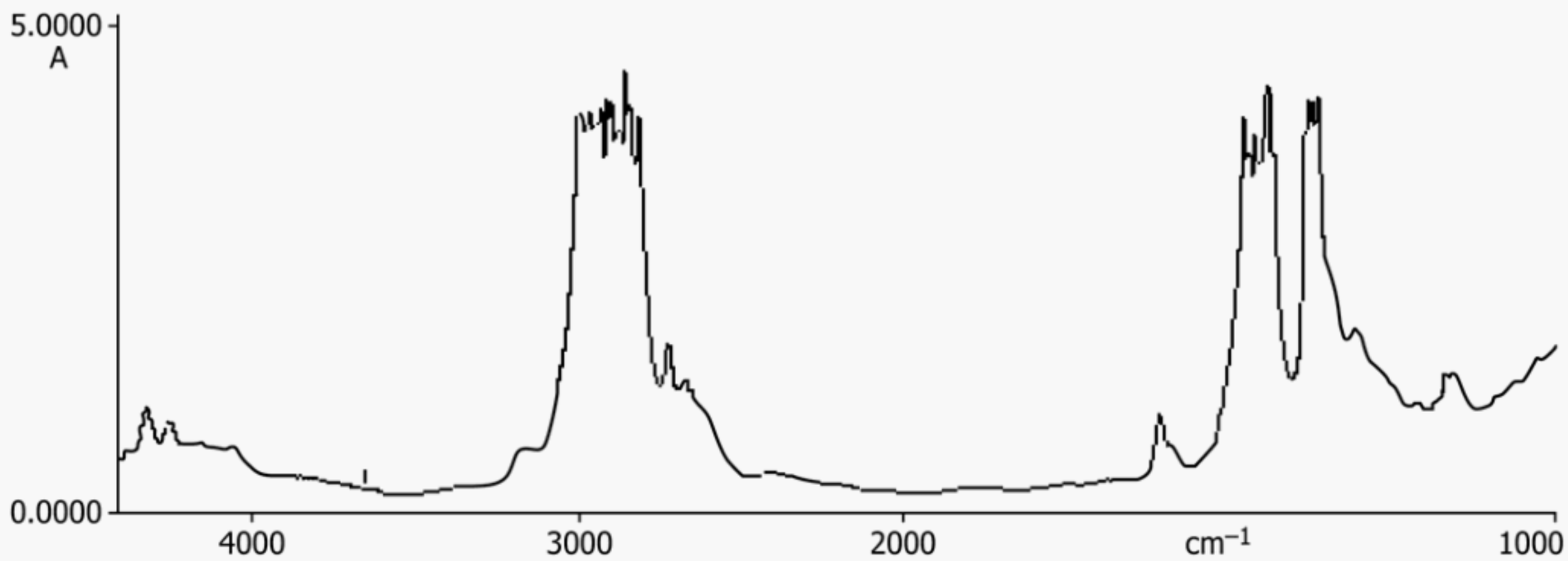


FIG. 4 Section of Differential Scan-B



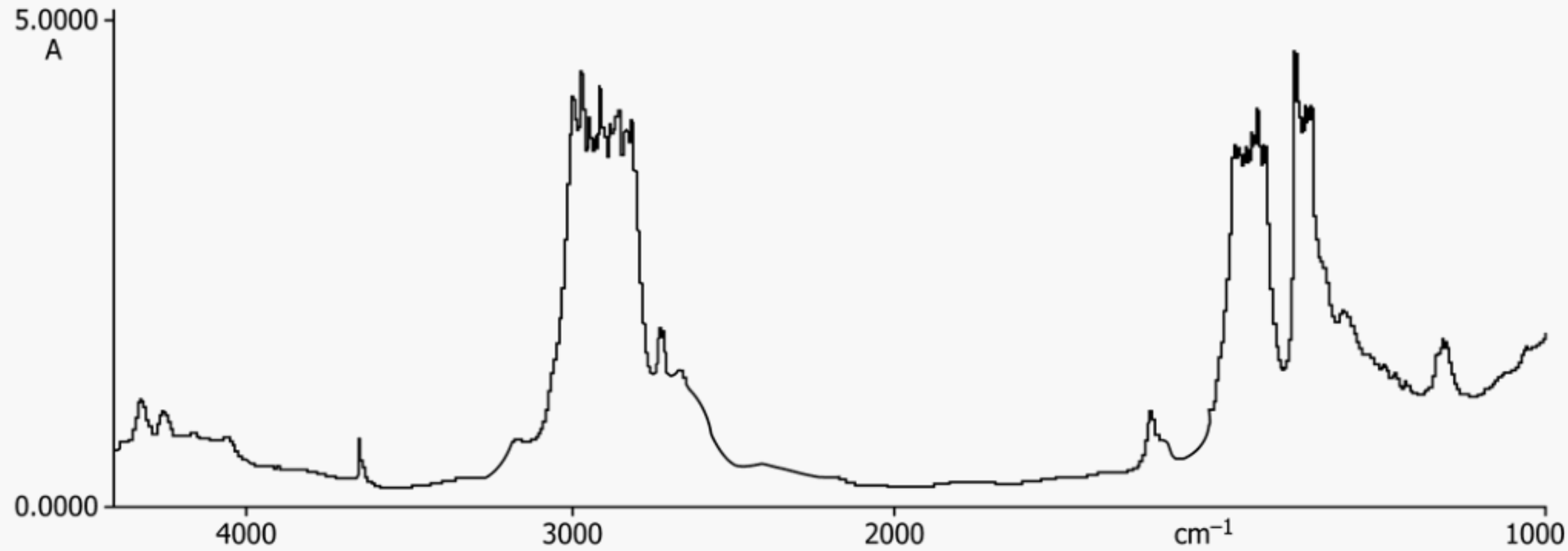
■ Y: 16 scans, 4.0 cm^{-1}, std2, UNINHIBITED VIRGIN TRANS. OIL

FIG. 5 Example of a FTIR Scan of an Uninhibited Transformer Oil

specimen performed manually or the average of the same number of scans used to standardize the instrument for the test specimen, if performed automatically.

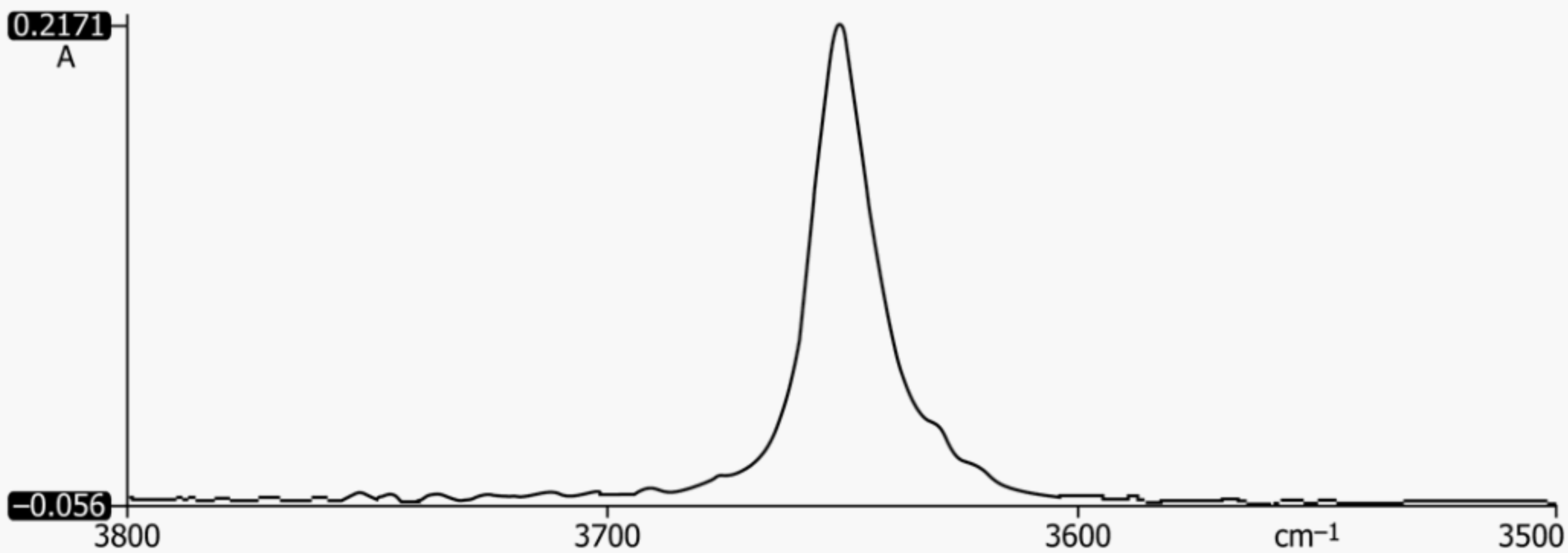
7.3 *Manually Determined Results*—Read the values of absorbance at points A_o and A on each of the three spectra to the nearest 0.001 absorbance unit; obtain the differences and average them. Using this average value, determine the concentration from the calibration curve.

7.4 *Automatically Determined Results*—Using the average value of the absorbance difference between A and A_o that is determined automatically by the instrument for the same number of scans performed on the test specimen as for the standards, determine the concentration of the test specimen from the calibration curve.



Y: 16 scans, 4.0 cm^{-1}, std1, 0.55% DBPC IN TRANS OIL

FIG. 6 Example of a FTIR Scan on a Transformer Oil with 0.55 % DBPC



z: 16 scans, 4.0 cm^{-1}, 0.55% DBPC IN TRANS OIL, diff

FIG. 7 Example of the Differential Scan Showing 0.542 % WT % DBPC Inhibitor

8. Precision and Bias

8.1 *Checking Limits of Range* $(A - A_o)$—Determine the highest and lowest values of $A - A_o$ resulting from the three or more scans and subtract one from the other. If the resulting quantity exceeds 0.010, reject the results as excessively variable and repeat the test.

8.2 *Repeatability*—The difference between two determinations for DBPC and DBP, each obtained from the average of three scans determined manually, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the value of 0.04 % by only one case in twenty.

8.3 *Reproducibility*—The difference between two determinations for DBPC and DBP, each obtained from the average of three scans determined manually, obtained by different operators in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the value of 0.04 % by only one case in twenty.

8.4 No statement is made about bias of this test method for measuring ditertiary-butyl paracresol or ditertiary-butyl phenol as it has not been determined.

9. Keywords

9.1 2,6-ditertiary-butyl paracresol; 2,6-ditertiary-butyl phenol; electrical insulating oil; infrared absorption

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