INTERNATIONAL

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Standard Practices for Examination of Electrical Insulating Oils by Infrared Absorption[1]

This standard is issued under the fixed designation D2144; 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 These practices are to be used for the recording and interpretation of infrared absorption spectra of electrical insulating oils from 4000 cm^{-1} to 400 cm^{-1} (2.5 µm to 25 µm).

NOTE 1—While these practices are specific to ratio recording or optical null double-beam dispersive spectrophotometers, single-beam and HATR (horizontal attenuated total reflectance), Fourier-transform rapid scan infrared spectrophotometers may also be used. By computerized subtraction techniques, ratio methods can be used. Any of these types of equipment may be suitable if they comply with the specifications described in Practice E932.

1.2 Two practices are covered, a Reference Standard Practice and a Differential Practice.

1.3 These practices are designed primarily for use as rapid continuity tests for identifying a shipment of oil from a supplier by comparing its spectrum with that obtained from previous shipments, or with the sample on which approval tests were made. They also may be used for the detection of certain types of contamination in oils, and for the identification of oils in storage or service, by comparison of the spectra of the unknown and known oils. The practices are not intended for the determination of the various constituents of an oil.

1.4 **Warning—**Infrared absorption is a tool of high resolving power. Conclusions as to continuity of oil quality should not be drawn until sufficient data have been accumulated so that the shipment-to-shipment variation is clearly established, for example.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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.*

1.7 *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
E131 Terminology Relating to Molecular Spectroscopy
E168 Practices for General Techniques of Infrared Quantitative Analysis
E932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrometers

3. Terminology

3.1 *Definitions*—For definitions of terms and symbols, refer to Terminology E131.

4. Summary of Practices

4.1 The infrared absorption spectrum may be recorded on the spectrophotometer by either of the two practices outlined below. In both practices differences in wavelength or frequency and intensity of the absorption bands are observed and measured.

4.1.1 *Reference Standard Practice* —An infrared cell filled with the insulating oil test specimen is placed in the sample beam of the spectrophotometer. With the shutter of the reference beam open, the infrared absorption spectrum is recorded over the entire range of the instrument. The absorption spectrum of the test specimen is compared with a reference spectrum obtained with oil from a previous test specimen or the qualification oil.

4.1.2 *Differential Practice*—Two cells having the same sample path length are filled, one with the test specimen and the other with the reference oil. The filled cells are then placed in the paths of the sample and reference beams, respectively,

[1] These practices are under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gases and are 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.

and the differential absorption spectrum recorded. This spectrum is then compared with the reference differential spectrum obtained in a similar manner with the same cells filled with the reference oil.

5. Significance and Use

5.1 The infrared spectrum of an electrical insulating oil is a record of the absorption of infrared energy over a range of wavelengths. The spectrum indicates the general chemical composition of the test specimen.

NOTE 2—The infrared spectrum of a pure chemical compound is probably the most characteristic property of that compound. However, in the case of oils, multicomponent systems are being examined whose spectra are the sum total of all the spectra of the individual components. Because the absorption bands of the components may overlap, the spectrum of the oil is not as sharply defined as that for a single compound. For these reasons, these practices may not in every case be suitable for the quantitative estimation of the components of such a complex mixture as mineral oil.

6. Apparatus

6.1 *Infrared Spectrophotometer* —An infrared spectrophotometer capable of operating within the 4000 cm^{-1} to 400 cm^{-1} (2.5 µm to 25 µm) range in accordance with Practice E932.

6.2 *Absorption Cells*—Three types of cells may be used for measuring the absorbance of electrical insulating oils, namely (*1*) the sealed or fixed liquid cell, (*2*) the variable space cell, and (*3*) the demountable liquid cell. The use of the demountable cell is not recommended for quantitative analysis. Use sealed fixed liquid and demountable liquid cells that meet the requirements of Practices E168. When measuring the absorbance of an oil by the Reference Standard Practice, a sealed or fixed cell having a sample path length of 0.1 mm ± 0.014 mm is recommended. Cells having a fixed path length of 0.2 mm ± 0.028 mm have been found to be acceptable. When the Differential Practice is used, two matched sealed or fixed cells each having a sample path length of 0.050 mm ± 0.007 mm are recommended. Where two matched cells are not available, a variable space cell may be adjusted and used in place of one fixed cell. With spectrophotometers having a range up to 16.7 µm (600 cm^{-1}), liquid cells may be provided with sodium chloride (NaCl) windows. With instruments having a range up to 25 µm (400 cm^{-1}), use liquid cells with potassium bromide (KBr) windows.

6.3 *Cell Filling Device*—Use a glass hypodermic syringe of 2 to 5-mL capacity or other suitable apparatus to fill the liquid cells.

7. Sampling

7.1 Obtain the sample in accordance with Practices D923.

8. Calibration

8.1 Adjust and calibrate the spectrophotometer and cells in accordance with Practice E932.

9. Conditioning

9.1 Store the sample in its original container and shield it from light. Allow the sealed container to stand undisturbed in the room in which the test is to be made for a sufficient period of time to permit the sample to attain room temperature before it is opened.

9.2 Prior to taking specimens of transformer oil or light cable oil, shake the sample container thoroughly and allow it to stand undisturbed for 15 min in order for all air bubbles to be dissipated from the sample. For heavy cable oils, gently tilt or invert the sample container and swirl the fluid several times and then permit it to stand undisturbed for 15 min.

10. Cleaning, Storing, and Filling the Cell

10.1 After the cells have been used, thoroughly rinse them with a suitable reagent grade or functionally equivalent organic solvent such as 2–propanol (isopropyl alcohol) (care should be exercised to keep this solvent as dry as possible), followed by rinsing with a reagent grade or functionally equivalent hydrocarbon solvent, such as petroleum naphtha and store in a desiccator until they are to be used.

10.2 When a cell is to be used, clean it again as described in 10.1 followed by two rinsings with the sample obtained from the middle portion of the fluid. Rinse the cell with a portion of the sample using the hypodermic syringe, which shall also be cleaned prior to use in accordance with 10.1.

10.3 When filling the cell, fill the cleaned and rinsed syringe with about 2 mL of the test specimen. With the cell in the upright position and the TFE-fluorocarbon plugs removed from the ports in the cell, insert the syringe in the lower port and slowly fill the cell by exerting gradual pressure on the syringe plunger. When oil is observed flowing from the top port, lay the cell flat, remove the syringe, plug the lower port tightly, and plug the upper port loosely. (**Warning—**A pocket in some cells may secrete minute quantities of a previous test specimen which may contaminate the current test specimen and cause erroneous results. Where this is suspected, dry the cell out after cleaning and rinsing with a reagent grade or functionally equivalent hydrocarbon solvent, such as petroleum naphtha, and by sweeping it with dry nitrogen applied at a pressure not exceeding 2.5 kPa (20 mm Hg) above ambient.)

11. Procedure—Reference Standard Practice

11.1 Fill a clean sealed or fixed cell having a sample path length of 0.10 mm ± 0.014 mm (or 0.20 mm ± 0.028 mm) with the test specimen as outlined in Section 10 and place the filled cell in the sample beam. Leave the shutter in the reference beam in the open position. Adjust the scanning speed, gain, and other variable controls to the values established for the particular spectrophotometer to provide the desired resolution. Where the instrument is provided with a scale changer, it is recommended that it be used with the 2.5 to 1 ratio in preference to the linear mode in obtaining recordings of the spectra. Record the infrared spectrum over the entire range of the instrument in accordance with Practices E168, using nonlinear absorbance charts.

11.2 Compare the infrared spectrum of the test specimen with the reference spectrum of a test specimen from a previous shipment, or the approved qualification oil, recorded by the same procedure, using the same cell and with the same instrument settings. Comparison can be made by superimposing the two spectra over a viewing light or by testing both test specimens and recording the spectra on the same chart using different colored inks. Software techniques may also be used for this comparison. Note and record any differences in the wavelengths or frequencies of absorption bands and in apparent intensity of these bands. Differences between these spectra can be amplified considerably by using an expanded ordinate scale during the scanning.

11.3 Measurements of the absorbance at specific absorption bands, if required, are made by the base-line method described in Practices E168 and corrected for thickness by expressing the results as absorbance per millimetre.

11.4 When using an FT-IR instrument, scan the atmosphere at least three times with no cell in the instrument and store this averaged spectrum as the background. Place the cell containing the test specimen in the instrument and again scan the spectrum at least three times. The resulting spectrum will be that of the test specimen.

12. Procedure—Differential Practice

12.1 Fill two matched cells with the reference oil, each having a path length of 0.050 mm ± 0.007 mm; insert one cell in the reference beam and the other in the sample beam. Adjust the spectrophotometer as described in 11.1, set the pen position at approximately 50 % transmission at 4000 cm^{-1} (2.5 µm), and record the differential infrared spectrum over the entire range of the instrument, in accordance with Practices E168. Evidences of peaks (positive or negative) will be an indication that the cells are not matched or that the amplifier balance is not properly adjusted.

NOTE 3—Peaks that are below the base line are considered "positive" and those above the base line are "negative."

12.2 When two fixed matched cells having a sample path length of 0.050 mm ± 0.007 mm are not available, a variable cell whose sample path length can be adjusted to equal the path length of the fixed cell may be used. The procedure for adjusting the sample path length of the variable cell is as follows:

12.2.1 Set the variable path length cell to the nominal thickness of the fixed path length liquid cell.

12.2.2 Place the variable and fixed path length cells, both filled with the reference oil, in the paths of the reference and sample beams, respectively.

12.2.3 Close both beams of the spectrophotometer and adjust the electrical balance on the amplifier to no drift on the recorder pen.

12.2.4 Set the pen position to approximately 90 % transmission at 4000 cm^{-1} (2.5 µm).

12.2.5 Record the differential infrared spectrum over the entire range of the instrument in accordance with Practices E168.

12.2.6 Adjust the path length of the variable cell until absorptions due to differences in sample path length are no longer present; then repeat as in 12.2.5.

12.3 With the same two matched cells with which the reference/reference differential spectrum was recorded, fill one with the reference oil and the other with the test specimen and insert them in the paths of the reference and sample beams, respectively. Record the differential infrared spectrum over the entire range of the instrument in accordance with Practices E168, using a nonlinear absorbance chart. Compare the reference/reference differential infrared spectrum obtained in accordance with either 12.1 or 12.2 with the sample/reference differential infrared spectrum of this paragraph. Comparison can be made by recording on the same chart with a different colored ink or by superimposing the two spectra over a viewing light. Note and record any differences in the wavelengths or frequencies of absorption bands and in apparent intensity of these bands.

NOTE 4—This procedure is recommended to ensure that the recording of spurious absorptions due to amplifier drift at zero energy null points are not erroneously assumed to be absorptions induced by differences in composition.

12.4 Measurements of the absorbance per millimetre, if required, shall be made as described in 11.3.

12.5 When using an FT-IR instrument, place the cell containing the reference oil in the instrument and scan the spectrum at least three times. Store the averaged spectrum as the background. Remove the cell from the instrument, empty and clean the cell. Fill the same cell with the test specimen of oil and scan the spectrum at least three times. The resulting spectrum will now be the differential spectrum of the test specimen of oil minus that of the reference specimen of oil.

13. Calculation

13.1 Convert measured absorbances and differences in absorbance and report as absorbance per millimetre in order to correct for variations in the sample path length, within the tolerances prescribed for the cells. Absorbance may not be a linear function of sample path length over a wide range of cell lengths; therefore strictly adhere to the cell sizes and make comparison of absorbance per millimetre measured with different path lengths only with caution. Calculate absorbance per millimetre using the equations given in this section for measurements obtained by either the Reference Standard Practice or the Differential Practice.

13.2 *Reference Standard Practice*—Differences in the absorbance per millimetre at specific absorption bands of spectra obtained from two test specimens of oil shall be expressed as the difference in absorbance, calculated as follows:

$$\text{Difference between absorbance per millimetre}$$
$$\text{of test specimen } S \text{ and test specimen } R \text{ at } \lambda \ \mu\text{m}$$
$$\left[(A_s - A_r)/t\right]\text{mm}^{-1}\ at\left[(10\,000/\lambda)\text{cm}^{-1}\right]$$

where:

A_s = test specimen absorbance at λ µm [(10 000/λ)cm^{-1}] as calculated by the base-line method (Practices E168) at λ_1 and λ_2 boundary points,
A_r = reference oil absorbance at λ µm [(10 000/λ)cm^{-1}] as calculated by the base-line method (Practices E168) at λ_1 and λ_2 boundary points,
λ = wavelength of absorption band, and
t = sample path length of cell used, mm.