Designation: D2144 – 07

StandardPractices 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 to 400 cm^{-1} (2.5 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 *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:*[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, 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.

[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.

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 to 400 cm^{-1} (2.5 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 ± 0.014 mm is recommended. Cells having a fixed path length of 0.2 ± 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 ± 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 ± 0.014 mm (or 0.20 ± 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