Designation: D6802 – 02 (Reapproved 2010)

Test Method for Determination of the Relative Content Of Dissolved Decay Products in Mineral Insulating Oils by Spectrophotometry[1]

This standard is issued under the fixed designation D6802; 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 characterizes by spectrophotometry the relative level of dissolved decay products in mineral insulating oils of petroleum origin. While new oil is almost transparent to a monochromatic beam of light in the visible spectrum, the increasing concentration of dissolved decay products shift the absorbance curve to longer wavelengths.

1.2 This test method is applicable to compare the extent of dissolved decay products for oils in service. It can assess the effectiveness of used or stored oil purification during the reclamation process, as well.

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

1.4 *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 to determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D923 Practices for Sampling Electrical Insulating Liquids

D1524 Test Method for Visual Examination of Used Electrical Insulating Oils of Petroleum Origin in the Field

D3487 Specification for Mineral Insulating Oil Used in Electrical Apparatus

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *aged oil, n*—an oil that no longer complies with the standard specifications for mineral insulating oils used in electrical apparatus according to D3487.

4. Summary of Test Method

4.1 A test specimen of mineral insulating oil is placed in a 10-mm path length glass cuvette, which is installed in an UV-VIS scanning spectrophotometer. The instrument is first zeroed with spectral grade heptane. The absorbance curve of oil is then recorded from 360 to 600 nm. Integration of the area under this curve indicates the numeric value of the dissolved decay products in the oil sample. Because of the high sensitivity of spectral analysis, the deterioration of oil purity can be assessed in the early stages of the decay process.

5. Significance and Use

5.1 The content of dissolved decay products in insulating oils is made up of a variety of compounds, such as peroxides, aldehydes, ketones, and organic acids. Each of them is partially adsorbed on the large surface of paper insulation leading to the premature aging of power transformers. The relative assessment of byproduct formation, therefore, can be used as an indicator of the aging of the mineral oil.

6. Interferences

6.1 The condition of the oil specimen should be clear according to the requirement of Test Method D1524.

6.2 The oil specimen, therefore, should be filtered through 50-μm filter paper.

7. Apparatus

7.1 *Recording UV-Visible Automated Spectrophotometer,* capable of scanning the range between 360 and 600 nm is required. The software should permit the calculation of area under the absorbance curve of the oil specimen.

8. Reagents and Materials

8.1 *Absorption Cuvettes*—To determine the absorbance curve of a mineral insulating oil, two matched glass cuvettes having a path length of 1-cm ± 0.01-cm should be used.

[1] This test method is under the jurisdiction of Committee D27 on Electrical Insulating Liquids and Gasesand 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.

8.2 *Cuvette Filling Device*—A disposable plastic dropper of 2-mL capacity is recommended; however, any other suitable pipette may be used.

8.3 *Petroleum Spirits,* of 60–80°C boiling range.

8.4 *Heptane,* spectral grade.

9. Sampling

9.1 Obtain the oil sample in accordance with Practice D923.

10. Preparation of Apparatus

10.1 Clean the cuvettes thoroughly with petroleum spirits.

10.2 Adjust the automated spectrophotometer in accordance with manufacturer's recommendation.

10.3 Carry out the testing procedure at room temperature (25 ± 5°C).

11. Procedure

11.1 Fill one glass cuvette with heptane; place it in the sample holder and zero the instrument by adjusting it to read zero absorbance.

11.2 Move the heptane-filled cuvette by placing it to the reference position.

11.3 Fill the second glass cuvette with the oil specimen and place it into the sample holder.

11.4 Set the instrument to scan the region from 360 to 600 nm and begin scanning the specimen.

11.5 Display the absorbance curve and set the instrument to calculate the area under the curve.

12. Interpretation of Results

12.1 A relationship exists between the area under the absorbance curve and the total amount of dissolved decay products in mineral insulating oils. New oils usually have a relative area under the curve of less than 25 Abs. × nm.

12.2 The shift of the absorbance curve to longer wavelengths indicates an increased content of dissolved decay products in the oil.

12.3 The shift of the absorbance curve to shorter wavelengths after reclaiming a used or stored oil indicates the selective removal of dissolved decay products.

13. Report

13.1 Identification of oil sample.

13.2 The value of the calculated area under the absorbance curve for the oil specimen from 360 to 600 nm.

13.3 Comparison of this area to the area of typical new oil, which is usually less than 25 Abs. × nm, represents the relative content of dissolved decay products.

14. Precision and Bias

14.1 *Precision*—The precision of this test method has not been investigated through an interlaboratory test program.

14.1.1 *Repeatability*—Repeatability measurements made in one laboratory on three samples resulted in a coefficient of variation of 2.8 %. At the 95 % confidence level, duplicate determinations should agree within 7.8 % of the average of the two results (see Table 1).

14.1.2 *Reproducibility*—No data are available on which to base an estimate of the reproducibility of this test method. An interlaboratory test program will be conducted to develop this data.

14.1.3 *Bias*—No information can be presented on the bias of the procedure for measuring the area under the absorbance curve in this test method, because no materials having an accepted reference value are available.

15. Keywords

15.1 chemical stability; dissolved decay products; insulating oil; oxidation decay; visible spectrum

TABLE 1 Data Used to Develop Precision and Bias Statements for Dissolved Decay Product Area

	Absorbance Area Under the Curve		
Sample	Aged Oil South America	New Oil	Aged Oil North America
	1	2	3
Test 1	450.42	18.55	359.74
Test 2	455.54	18.39	361.03
Test 3	450.05	18.85	382.55
Test 4	443.08	18.85	387.25
Test 5	451.46	18.80	378.36
Average of tests (area)	450.11	18.69	373.79
Variance, $\sigma^2_{(n-1)}$	20.21	0.04	159.75
Standard deviation $\sigma_{(n-1)}$	4.50	0.21	12.64
Coeff. variation, σ/average, %	1.0	1.11	3.38
Number of determinations (n)	5	5	5
Number of DE (n-1)	4	4	4
Grand average, Area	280.86		
Pooled variance, σ^2	60.00		
Pooled standard dev, σ	7.75	Total FE=12	
Pooled coeff var, σ/average, %	2.76		
Repeatability std dev, σ	7.75		
ASTM repeatability 2.83 σ, area	21.92		
Coeff variation, σ/average, %	2.76		
ASTM repeat 2.83 (σ/average), %	7.81		