**Designation: D 6181 – ~~97~~03**

# Standard Test Method for Measurement of Turbidity in Mineral Insulating Oil of Petroleum Origin[1]

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## 1. Scope

1.1 This test method covers the laboratory procedure that ascertains the quantity of suspensions in insulating oils of petroleum origin using a nephelometric measurement technique to determine the fluid's turbidity. This test method is designed to reveal changes that may occur to these oils.

1.2 This test method is applicable for turbidities in the range of 0.1 to 500 Nephelometric Turbidity Units (NTU).

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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 determine the applicability of regulatory limitations prior to use.*

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[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 D27.07 on Physical Test.

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## 2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 923 Test Method for Sampling Electrical Insulating Liquids
D 1533 Test Methods for Water in Insulating Liquids (Karl Fischer Reaction Method)
D 1698 Test Method for Sediments and Soluble Sludge in Service Aged Insulating Oils
D 1889 Test Method for Turbidity of Water
D 4652 Specification for Silicone Fluid Used for Electrical Insulation
D 5180 Test Method for Quantitative Test for Turbidity in Clear Liquids
E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

## 3. Terminology

3.1 *Description of Terms Specific to This Standard:*

3.1.1 *nephelometric turbidity unit (NTU), n*— intensity of light scattered by a known aqueous suspension of formazine. One NTU is the turbidity of a formazine solution produced by mixing 12.5 µg of hydrazine su1fate and 1.25 µg of hexamethylenetetramine in 1 mL of turbidity-free water. See Appendix X1 for preparation instructions.

3.1.2 *turbidity, n*—the reduction of transparency due to the presence of particulate matter.

## 4. Summary of Test Method

4.1 The turbidity is determined by a calibrated, ratio turbidimeter, which measures scattered light at 0.5 π rad (90°) or 0.5 and 1.5 π rad (90 and 270°) angles to the incident beam. These instruments cannot be calibrated accurately in terms of absolute turbidity except in the case of fluids having uniform-size particles that are less than approximately one fifth of the wave length of the incident light. Standards have been prepared by thoroughly mixing suitable amounts of finely divided titanium dioxide into partially polymerized polystyrene. Alternatively, suspension of formazine has been used as a turbidity standard, formed by reacting hydrazine sulfate and hexamethylenetetramine under carefully controlled conditions.[3] Calibrated commercial standards in sealed tubes also are available.

4.2 The test specimen is placed in the cell and its turbidity is measured. The turbidimeter measures the light scattered by suspended solids dispersed within the test specimen. Accuracy and sensitivity of the method is ensured by measuring the turbidity at a wavelength of light where there is little or no absorption of the light by the test specimen. Use of a narrow bandwidth of light reduces interference that may be inherent to ~~these~~ oxidized insulating oils. The narrow bandwidth is achieved through the use of an optical filter to provide light at 600 nm with a 40 nm bandwidth.

4.3 This test method is recommended only for mineral insulating oils and is not intended to be used on other fluids.

## 5. Significance and Use

5.1 This test method uses a ratio turbidimetric optical system to measure the turbidity of insulating oils relative to turbidity standards. Cloudiness or turbidity is attributed to matter whose diameter is approximately 20 % of the wavelength of the incident light. Increasing turbidity signifies increasing transformer fluid contamination, either from external sources or internal chemical reactions (such as oxidation) that produce fine particulate matter. Other turbidity sources, such as water droplets or gas bubbles, are not of interest in this evaluation of insulating oils. The elimination of these interferences is described in 6.2 and 6.6. This test method quantifies changes which may not be apparent to the unaided human eye.

## 6. Interferences

6.1 The ~~increased~~ changed color of the test specimen, as a result of the oxidation process, may cause light absorption at the wave length of the turbidity measurement. This interference is minimized by using light of a narrow bandwidth, which is not absorbed by the test specimen.

~~6.2 Air~~

6.2 Gas bubbles that are entrained in the test specimen will interfere. If ~~air~~ gas bubbles are apparent, measurement should be delayed until all bubbles disappear. This usually will require several minutes.

6.3 Scratches on the test specimen cell can increase the stray light of the optical system resulting in positive interference when performing ultra-low turbidity measurements. The selection of high optical grade cells and coating the outside of the cells with a thin layer of silicone will eliminate this interference.

6.4 The use of matched test specimen cells that are inserted into the measurement instrument in a consistent orientation will result in consistent measurements.

6.5 The turbidity of the sample can be influenced by the temperature of the sample. The test specimen, therefore, always should be analyzed at room temperature between 20 to 30°C.

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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, ~~Vol 10.03.~~ volume information, refer to the standard's Document Summary page on the ASTM website.
~~*Annual Book of ASTM Standards*,~~

[3] For information, see Chevalier, P., "Formazine Standard for Turbidity," Brasserie, Vol 152, 1959, pp.~~θ~~ 132–133.

6.6 When cloudiness in the oil test specimen is visible to the naked eye, it should be tested first for excessive water content in accordance with Test Methods D 1533. If excessive water is confirmed present, a new sample should be obtained for turbidity testing. Moisture contained in the test specimen cells may generate a two-phased test specimen. As a precaution, dry test specimen cells by placing them upright, with caps removed, in an oven at 105°C for 1 h.

## 7. Apparatus

7.1 *Turbidimeter and Cells*,[4] a ratioing turbidimeter equipped with a 600 nm interference filter assembly having the following characteristics:

| | |
|---|---|
| Center Wavelength | 600 ± 6 nm |
| Band Width | 40 ± 8 nm |
| Peak Transmittance | 50 % |

(**Warning**—In this application, the test specimens are often highly colored with products of oxidation. This color will interfere with the turbidity measurements. This interference is the result of the absorption of energy by the colored sample for instruments whose light source emits a broad spectrum of visible radiation. The above wavelength conditions have been found to minimize this interference and provide the most reliable turbidity measurement.)

7.2 *Volumetric Flasks*, Class A, 2 × 100 mL, 1 × 200 mL and 1 × 1 L.

7.3 *Volumetric Pipets*, Class A, 5 mL and 25 mL.

7.4 *Filtration Equipment*, membrane filter equipment with membranes having a pore size of 0.2 µm or less.

7.5 *Degassing Equipment*, such as an ultrasonic bath.

7.6 *Oiling Cloth*, a soft, lint-free cotton cloth for removing excess silicone from the test specimen cell.

## 8. Reagents

8.1 *Water*, deionized water that has been filtered through a 0.2 µm or smaller membrane filter and free of turbidity.

8.2 *Formazine*, primary stock standard solution with a turbidity of 4000 NTU (see Appendix X1 for preparation of formazine solution.

8.3 *Silicone Oil*, a silicone fluid meeting Specification D 4652.

8.4 *Turbidity Standards*, as an alternative to turbidity standard preparation, stable turbidity standards are available from most laboratory supply companies.

## 9. Sampling

9.1 Insulating oils for this test should be sampled in accordance with Test Method D 923.

## 10. Procedure

10.1 Calibrate the turbidimeter according to the manufacturer's directions. Use the calibration standards of 0, 20.0, 200 and 500 NTU prepared as directed in Table 1 from the 4000 NTU stock solution. Calibrate turbidimeter quarterly or as needed.

10.2 Wash test specimen cells with a solvent that will dissolve oil, such as petroleum ether, and remove all oil residues. Wash cells again with water and detergent. Rinse cells three times with filtered, deionized water.

10.3 Place test specimen cells with opening upright and caps removed in an oven at 105°C for 1 h. Cool in a desiccator. Cap immediately when cool to prevent cell contamination.

10.4 Carefully pour the oil into the test specimen cell. Avoid trapping air in the cell. Fill the cell according to instrument manufacturer's directions. The oil level must be such that all the light passes into the test specimen. If ~~air~~ gas bubbles form in the cell upon standing, ~~degassing of the sample~~ their removal is required. ~~Degas the~~ The sample ~~using~~ should be placed in an ultrasonic bath for degassin~~d rg~~. Refill cell with degassed test specimen without bubbles.

10.5 Hold the cell by the cap and wipe it to remove water spots and finger prints.

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[4] A suitable turbidimeter and accessories are obtainable from Hach Company, P.O. Box 389, Loveland, CO 80539.

**TABLE 1 Preparation of Calibration Solutions**

| Calibration Standard Concentration (NTU) | Volumetric Flask to Be Used | Volumetric Pipet to Be Used | Dilution mL Stock Formazine by Filtered Water |
|---|---|---|---|
| Blank | n/a | n/a | none |
| 20.0 NTU | 1 L | 5.00 mL | 5.00 mL to 1 L |
| 200 NTU | 100 mL | 5.00 mL | 5.00 mL to 100 mL |
| 500 NTU | 200 mL | 25.0 mL | 25.0 mL to 200 mL |

10.6 Apply a thin bead of silicone oil from the top of the cell to the bottom of the outside of the cell. Using the oiling cloth, spread the oil uniformly over the surface of the cell. Then, wipe off the excess oil and polish the cell. The polished cell should appear nearly dry with little or no visible oil.

10.7 Insert the cell in the instrument and measure the turbidity according to the instrument manufacturer's instructions.

10.8 Record the instrument's turbidity reading in NTU.

## 11. Report

11.1 Report the following information:

11.1.1 The turbidity of the mineral insulating oil in NTUs.

## 12. Precision and Bias

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

12.2 *Repeatability*:

12.2.1 An estimate of the repeatability has been developed based on data supplied by a single laboratory from two separate test programs. In the first program, conducted in 1996, seven oil samples ranging in turbidity from 0.5 to 6 NTU were tested five times each using the same apparatus and operator (see Table X2.1). In the second program, conducted in 1997, three oil samples ranging in turbidity from 8 to 94 NTU were tested ten times each using the same apparatus and operator (see Table X2.2).

12.2.2 The standard deviations of the test results from these programs were found to be dependent on the level of turbidity measured.

12.2.3 The coefficients of variation (CV %) of test results from these programs were found to be independent of level of test result. The pooled CV % from the combined programs was 0.71 %. While the statistical evidence regarding the appropriateness of combining the data from the two programs is not clear cut, the pooled CV % was used as the basis for the preliminary repeatability estimate below.

12.2.4 *Repeatability*— The 95 % repeatability limit over the range from 0 to 94 NTU is 2.0 % of the test result.

12.3 *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.

12.4 *Bias*—No information can be presented on the bias of the procedure in this test method for measuring the turbidities of insulating oils because no material having an accepted reference value is available.

## 13. Keywords

13.1 insulating oil; oxidation; sludge; stability; turbidity

# APPENDIXES

**(Nonmandatory Information)**

## X1. PREPARATION OF STANDARD FORMAZINE SOLUTION

X1.1 Although turbidity standards are readily available from laboratory supply houses, some people wish to prepare their own standards. The following procedure has been adapted from *Standard Methods for the Examination of Water and Wastewater* , ~~Fourteenth~~ Nineteenth Edition.[5]

X1.2 Prepare turbidity free water by passing distilled water through a membrane filter that removes all particles larger than 0.1 µm. Dissolve 1.000 g of hydrazine sulfate, $(NH_2)_2 \cdot H_2SO_4$, in turbidity-free distilled water and dilute to 100 mL in a volumetric flask. ( **Warning**—Hydrazine sulfate is a carcinogen.) Dissolve 10.00 g hexamethylenetetramine, $(CH_2)_6N_4$, in turbidity-free distilled water and dilute to 100-mL in a volumetric flask. Pipet 25.00 mL of the hydrazine sulfate solution into a 100-mL volumetric flask. Pipet 25.00 mL of the hexamethylenetetramine solution to this same flask. Mix the solutions well and allow to stand for 24 h at 25 ± 3°C. Do not dilute this solution any further. This solution is the formazine solution and the turbidity is, by definition, 4000 NTU. Prepare solutions monthly.

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~~*Annual Book of ASTM Standards*, Vol 14.02.~~

[5] Available from American Public Health Assoc. Sales, PO Box 753, Waldorf, MD 20604–0753.

## X2. DATA USED TO DEVELOP PRECISION AND REPEATABILITY ESTIMATES

X2.1 Data is given from a single laboratory for the measurement of turbidity for several oil samples. The data in Table X2.1 was obtained in 1996 and the data in Table X2.2 was obtained in 1997.

**TABLE X2.1 Turbidity Data Obtained in 1996[A]**

| | Sample | | | | | | |
|---|---|---|---|---|---|---|---|
| Trial | 1 | 2 | 3 | 4 | 5 | 6 | 7 |
| 1 | 0.475 | 1.280 | 1.830 | 2.140 | 2.650 | 3.510 | 6.120 |
| 2 | 0.478 | 1.300 | 1.850 | 2.070 | 2.690 | 3.550 | 6.150 |
| 3 | 0.480 | 1.290 | 1.850 | 2.050 | 2.690 | 3.550 | 6.170 |
| 4 | 0.479 | 1.300 | 1.840 | 2.070 | 2.700 | 3.540 | 6.170 |
| 5 | 0.477 | 1.290 | 1.840 | 2.100 | 2.690 | 3.520 | 6.140 |
| Average | 0.4778 | 1.292 | 1.842 | 2.086 | 2.684 | 3.534 | 6.150 |
| Standard Deviation (SD) | 0.0019 | 0.0084 | 0.0084 | 0.0351 | 0.0195 | 0.0182 | 0.0212 |
| RSD, % | 0.40 | 0.65 | 0.46 | 1.68 | 0.73 | 0.51 | 0.34 |

[A] Results given in NTU for five consecutive runs on the same sample.

**TABLE X2.2 Turbidity Data Obtained in 1997[A]**

| | Sample | | |
|---|---|---|---|
| Trial | 1 | 2 | 3 |
| 1 | 8.25 | 38.0 | 93.9 |
| 2 | 8.19 | 38.5 | 94.3 |
| 3 | 8.31 | 38.5 | 93.8 |
| 4 | 8.16 | 38.4 | 94.2 |
| 5 | 8.18 | 38.6 | 93.8 |
| 6 | 8.31 | 38.3 | 93.8 |
| 7 | 8.24 | 38.6 | 93.5 |
| 8 | 8.25 | 38.8 | 93.9 |
| 9 | 8.37 | 38.8 | 93.8 |
| 10 | 8.23 | 38.5 | 94.4 |
| Average | 8.251 | 38.5 | 93.89 |
| Standard Deviation (SD) | 0.0692 | 0.25 | 0.237 |
| RSD, % | 0.84 | 0.65 | 0.25 |

[A] Results given in NTU for ten consecutive runs on the same sample.