Designation: D 6786 – 02

Standard Test Method for Particle Count in Mineral Insulating Oil Using Automatic Optical Particle Counters[1]

This standard is issued under the fixed designation D 6786; 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 particle concentration and particle size distribution in mineral insulating oil. It is suitable for testing oils having a viscosity of 6 to 20 cSt at 40°C. The test method is specific to liquid automatic particle analyzers that use the light extinction principle.

1.2 *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:*

D 923 Practices for Sampling Electrical Insulating Liquids[2]

2.2 *ISO Standards:*

4406:1999 Hydraulic Fluid Power—Fluids—Method for Coding the Level of Contamination by Solid Particles[3]

11171:1999 Hydraulic Fluid Power—Calibration of Automatic Particle Counters for Liquids[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *coincidence*—the presence of more than one particle in the sensing zone of a particle analyzer at the same time, causing mis-sizing and mis-counting of the particle present. The coincidence limit of the counter is determined by the maximum acceptable concentration of particles in the sensing zone and is supplied by the instrument manufacturer.

3.1.2 *concentration limit*—a direct function of coincidence and electronic saturation. The concentration limit of the system is determined by the maximum acceptable concentration of particles in the given sample and is supplied by the instrument manufacturer.

3.1.3 *electronic saturation level*—particle concentration at which the electronic circuitry of the analyzer ceases to function properly due to excessive counting rates.

3.1.4 *light extinction*—the reduction in intensity of a light beam passing through the sensing zone of a particle analyzer, caused by the absorption and/or scattering of the light by particles. Synonyms: light obscuration, light interruption, light blockage.

4. Summary of Test Method

4.1 Samples are taken in particle-clean bottles that are dedicated to particle analysis. The sample bottle is agitated to redistribute particles in the oil, then immediately placed in an automatic particle counter, where the number of particles and their size distribution are determined by the light extinction principle.

4.2 As particles pass through the sensing zone of the instrument, the quantity of light reaching the detector is obscured. This signal is translated to an equivalent projected area diameter based on calibration with a NIST-traceable fluid (ISO Medium Test Dust suspension).

5. Significance and Use

5.1 Particles in insulating oil can have a detrimental effect on the dielectric properties of the fluid, depending on the size, concentration, and nature of the particles. The source of these particles can be external contaminants, oil degradation by-products, or internal materials such as metals, carbon, or cellulose fibers.

5.2 Particle counts provide a general degree of contamination level and may be useful in accessing the condition of specific types of electrical equipment. Particle counts can also be used to determine filtering effectiveness when processing oil.

5.3 If more specific knowledge of the nature of the particles is needed, other tests such as metals analysis or fiber identification and counting must be performed.

6. Interferences

6.1 Dirty environmental conditions and poor handling techniques can easily contaminate the sample and/or test specimen. Care must be taken to ensure test results are not biased by introduced particles.

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[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 D27.07 on Physical Tests.

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[2] *Annual Book of ASTM Standards*, Vol 10.03.

[3] Available from American National Standards Institute, 11 West 42nd Street, New York, NY 10036.

6.2 Air bubbles in the oil may be counted as particles giving false positive readings. Mixing or agitating the sample introduces bubbles into the oil, but these readily dissipate and generally do not interfere with insulating oil particle counts.

6.3 Suspended or free water in the oil will generally be counted as particles. Normally, *free* water concentrations below 10 ppm will not interfere.

6.4 Excessive concentrations of particles in the oil will cause coincidence and/or electronic saturation errors. Limits are determined by ISO 11171 and are generally supplied by the instrument manufacturer. These errors may be avoided by diluting the sample with particle-clean dilution oil or particle-clean solvent.

6.5 Odd-shaped particles and fibers may be improperly analyzed, depending on their orientation as they pass through the sensing zone of the instrument.

7. Apparatus

7.1 *Automatic Particle Counter*, liquid optical particle counter based on the light extinction principle. The instrument shall be capable of recording the size and number of particles as they pass across the detector. The particle counter shall include a bottle sampling apparatus that automatically delivers a predetermined volume of specimen at a controlled flow rate to the sensing zone of the analyzer.

7.2 *Mechanical Shaker*, paint shaker, table shaker, or other mechanical device to vigorously agitate sample bottles.

8. Materials

8.1 *Particle-clean Bottles*, recommended sample containers are cylindrical bottles made of polypropylene, polystyrene, PET, or glass with flat bottoms, fitted with a suitable non-shedding threaded cap. Bottles should be at least 100-mL capacity. The bottles shall meet the cleanliness criteria of contributing less than 1 % of the total particles expected in the cleanest sample.

8.2 *Particle-clean Solvent*, petroleum spirits, hexane, kerosene, or other suitable solvent, filtered through a 0.45 µm membrane filter.

8.3 *Calibration Fluid*, suspension of ISO Medium Test Dust in oil or hydraulic fluid, either a primary sample obtained directly from NIST[4] (SRM 2806) or a secondary sample prepared in accordance to ISO 11171 and traceable to NIST.

8.4 *Dilution Oil*, insulating oil that has been filtered to contain fewer than 1 % of the total particles expected in the cleanest sample.

9. Sampling

9.1 Refer to Practice D 923 for precautions for sampling from energized electrical equipment.

9.2 Proper sampling is crucial to particulate analysis. The following guidelines are offered to ensure representative sampling and to preserve sample integrity:

9.2.1 Wipe the sample port outlet with a clean lint-free towel. Open the sample valve and flush about 3 times the flow path volume of oil into a container.

9.2.2 While flushing, uncap the sample bottle and retain the cap in one hand and the bottle in the other. Without adjusting the valve or disturbing sample flow, fill the bottle to about 80 % capacity. Do not completely fill the bottle since the headspace will be needed for sample agitation.

9.2.3 Cap the sample bottle, then close the sample valve.

10. Calibration

10.1 Calibration of the instrument shall be with a NIST-traceable calibration fluid in accordance with ISO 11171.

10.2 Calibrate the instrument annually, unless experience justifies longer or shorter intervals.

10.3 Interim calibration checks should be made regularly by using a particle count standard fluid prepared using the procedures of ISO 11171. The check values should be within 10 % of the standard values.

11. Procedure

11.1 If necessary, wipe the outside of the sample bottle with a clean lint-free towel.

11.2 Agitate the sample bottle vigorously for 2-5 min to redistribute particles, the length of time depending on the effectiveness of the method of agitation. Use hand shaking or a mechanical shaker. Do not use a magnetic stir bar or any other device that comes in contact with the oil. Do not use an ultrasonic bath as this may break up the large particles.

11.3 Allow a few seconds for gas bubbles to escape or apply vacuum to the bottle until bubbles dissipate.

11.4 Immediately analyze the sample with the automatic particle counter according to the manufacturer's operating instructions. Usually the analyzer is flushed with a specimen of the oil prior to testing. After the initial flush, 2-3 runs on each sample are recommended.

11.5 When particle count runs are finished, flush the instrument with particle-clean solvent or particle-clean oil in accordance with the instrument manufacturer's recommendations.

12. Report

12.1 Report the average of the particle count runs as the cumulative number of particles per mL >4, >6, >10, >14, >21, >38, and >70 µm (c). The "(c)" after the size indicates that the particle counter was calibrated using ISO 11170. These particle sizes correspond approximately to >2, >5, >10, >15, >25, >50, and >100 µm for particle counters that were calibrated with the old ISO 4402 calibration standard. If only one particle count run is analyzed, report the results of the single run.

12.2 Optionally, also report the ISO solid contaminant code corresponding to the number of particles per mL >4, 6, and 14 µm (c), as prescribed in ISO 4406:1999.

13. Precision and Bias

13.1 Precision and bias statements based on interlaboratory round robin testing will be determined within five years of adoption of this standard method.

14. Keywords

14.1 optical particle counter; particle count; particulate contamination

[4] Available from National Institute of Standards and Technology, 100 Bureau Drive, Gaithersburg, MD 20899.