Designation: D7150 – 13

Standard Test Method for the Determination of Gassing Characteristics of Insulating Liquids Under Thermal Stress[1]

This standard is issued under the fixed designation D7150; 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 describes the procedures to determine the gassing characteristics due to thermal stress at 120°C of insulating liquids specifically and without the influence of other electrical apparatus materials or electrical stresses. This test method was primarily designed for insulating mineral oil. It can be applied to other insulating liquids in which dissolved gas-in-oil analysis (Test Method D3612) is commonly performed.

1.2 This test method is particularly suited for detection of the phenomenon sometimes known as "stray gassing" and is also referred to in CIGRE TF11 B39.

1.3 This test method is performed on transformer insulating liquids to determine the propensity of the oil to produce certain gases such as hydrogen and hydrocarbons at low temperatures.

1.4 This test method details two procedures:

1.5 Method A describes the procedure for determining the gassing characteristics of insulating liquids, at 120°C for 164 h.

1.6 Method B describes the procedure for processing the insulating liquid through an attapulgite clay column to remove organic contaminants and other reactive groups that may influence the gassing behavior of an insulating liquid, which is suspected of being contaminated. This procedure applies to both new and used insulating liquids.

1.7 The values stated in SI units are to be regarded as standard. English units are used when there is no metric equivalent.

1.8 *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]

D1933 Specification for Nitrogen Gas as an Electrical Insulating Material

D3612 Test Method for Analysis of Gases Dissolved in Electrical Insulating Oil by Gas Chromatography

2.2 *IEEE Document:*[3]

C 57.104 IEEE Guide for the Interpretation of Gases Generated in Oil-Immersed Transformers, 2008

2.3 *IEC Documents:*[4]

IEC 60599 Mineral oil-impregnated electrical equipment in service – Guide to the interpretation of dissolved and free gases analysis, 2007

IEC 61464 Guide for the interpretation of dissolved gas analysis (DGA) in bushings where oil is the impregnating medium of the main insulation (generally paper), 1998

CIGRE TF11 B39 Gas formation tendency test for mineral transformer oils, 2002

3. Terminology

3.1 *Definitions:*

3.1.1 *stray gassing, n*—the production of gases in an insulating liquid due to heating, contamination or in combination.

3.1.2 *Fuller's Earth, n*—highly adsorbent clay-like substance consisting mainly of hydrated aluminum silicates with the main minerals being montmorillonite, kaolinite, attapulgite and palygorskite.

4. Summary of Test Method

4.1 *Method A*—Insulating liquid is filtered through a mixed cellulose ester filter. A portion of the test specimen is sparged for 30 min with dry air. A test specimen is then placed into a glass syringe, capped and aged at 120 ± 2°C for 164 h. The test is run in duplicate. The other portion of the test specimen is

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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 D27.03 on Analytical Tests.

Current edition approved Dec. 1, 2013. Published January 2014. Originally approved in 2005. Last previous edition approved in 2005 as D7150-05. DOI: 10.1520/D7150-13.

[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.

[3] Available from the Institute of Electrical and Electronic Engineers, Inc, (IEEE), 445 Hoes Lane, Piscataway, NJ 08854; www.ieee.org

[4] Available from the International Electrotechnical Commission, 3, rue de Varembé, P.O. Box 131 CH-1211, Geneva 20, Switzerland; www.iec.ch

sparged for 30 min with dry nitrogen. A test specimen is then placed into a glass syringe, capped and aged at 120°C ± 2°C for 164 h. The test is run in duplicate. After the test specimens have cooled, dissolved gas-in-oil analysis is then performed according to Test Method D3612 or IEC Method 60599.

4.2 *Method B*—Insulating oil is passed through a heated (60 to 70°C) Fuller's earth column at a rate of 3 to 5 mL per minute. The insulating liquid is contacted with the Fuller's earth at a ratio of 1 g clay to 33 mL (range: 30 to 35 mL) of insulating liquid. The insulating liquid is collected and subjected to the testing as outlined in 4.1.

5. Significance and Use

5.1 Generation of combustible gases is used to determine the condition of oil-filled electrical apparatus. Many years of empirical evidence has yielded guidelines such as those given in IEEE C57.104, IEC 60599 and IEC 61464. Industry experience has shown that electric and thermal faults in oil-filled electrical apparatus are the usual sources that generate gases. Experience has shown that some of the gases could form in the oil due to thermal stress or as a result of contamination, without any other influences.

5.2 Some transformer oils subjected to thermal stress and oils that contain certain types of contamination may produce specific gases at lower temperatures than normally expected for their generation and hence, falsely indicate abnormal operation of the electrical apparatus. Some new oils have produced large amounts of gases, especially hydrogen, without the influence of other electrical apparatus materials or electrical stresses. This renders interpretation of the dissolved gas analysis more complicated.

5.3 Heating for 164 h has been found to be sufficient to reach a stable and characteristic gassing pattern.

5.4 This method uses both dry air and dry nitrogen as the sparging gas. This is to reflect either an electrical apparatus preservation system that allows oxygen to contact the oil or one that is sealed from the outside atmosphere. Oils sparged with air generally produce much more hydrogen as a percentage of the total combustible gas content as compared to oils sparged with nitrogen as these produce more hydrocarbons in relation to hydrogen.

6. Reagents and Materials

6.1 *Mixed Cellulose Ester Membrane Filter,* consisting of either 1 or 1.2 µm pore size, of correct diameter to fit in the filtering apparatus listed in 6.2.

6.2 *Vacuum Filtering Apparatus,* consisting of funnel reservoir (250 mL or larger), clamp, base, stopper and receiving flask. The 47 mm size is the type most often used.

6.3 *Dry Nitrogen,* meeting the requirements of Specification D1933, Type III with the following exception: the total hydrocarbon content must be <0.5 µL/L. This type of gas is sometimes referred to as Ultra-High Purity (UHP).

6.4 *Dry Air,* meeting the following requirements: 20 to 22 % oxygen, <3 µL/L water, and <1 µL/L total hydrocarbons. This type of gas is sometimes referred to as Zero Grade.

6.5 *Ovens,* forced-draft, adjustable to 120 ± 2°C and a drying oven, convection or forced-draft, or both, adjustable to 100 ± 5°C.

6.6 *Syringes,* glass, either 30 or 50 mL, either matched plunger and barrel or precision ground to 0.006 ± 0.001 mm maximum spacing between the inside of the barrel to the outside of the plunger for both the 30 mL and 50 mL syringes.

6.7 *Female-Luer-to-Closed-End-Adapter* , nickel-plated brass.

6.8 *Fuller's Earth (clay),* virgin material sized at 30/60 mesh.

7. Method A

7.1 If the sample has visible particles, filter 225 mL of insulating liquid through a 1 or 1.2-µm filter. Discard the first 25 mL. Collect the remainder in a flask that has been cleaned, rinsed with distilled water and dried for 4 h at 100 ± 5°C. Flasks that have been prepared beforehand are acceptable as long as all openings have been covered with aluminum foil. Alternatively, remove the plunger from a glass syringe and secure the tip of the barrel with a metal female luer-to-closed-end adapter. Place the barrel in a vertical position so that the large opening of the barrel is facing up. Place 25 mL of insulating liquid in a 30 mL syringe or 40 mL of oil in a 50 mL syringe. For flasks, use 100 mL.

7.2 Sparge enough of the filtered insulating liquid to conduct the testing with dry air for 30 ± 3 min. Sparge the air through the liquid at an approximate flow rate of 200 mL per minute. Typical setups are shown in Fig. 1 and Fig. 2.

7.3 If not already prepared in the syringes, immediately fill two (2) glass syringes (either 30- or 50-mL) with the sparged insulating liquid (25 mL in the 30-mL syringe; 40 mL in the 50-mL syringe). Make sure the syringe is filled with enough test specimen to conduct a dissolved gas-in-oil analysis. The barrel and plunger of each glass syringe must be well-fitted to avoid air ingress at the junction of the plunger and barrel seal as the test specimen is being heated during the test procedure.

7.4 Immediately remove any trapped bubbles from the syringe as described in Practice D3612 and seal the syringe with a female-luer-to-closed-end adapter if not already done so. Do not use plastic stopcocks as they tend to leak and melt during the heating phase of the procedure.

7.5 Sparge more insulating liquid as described in 7.1 through 7.2, but with dry nitrogen for 30 ± 3 min. The nitrogen is sparged through the insulating liquid at an approximate flow rate of 200 mL per minute.

7.6 Repeat the procedures listed in 7.3 and 7.4 for the nitrogen-sparged insulating liquid.

7.7 Place the syringes with the test specimens laying down in the oven with the leur fitting slightly above horizontal. Heat all the test specimens at 120 ± 2°C for 164 h ± 15 min. Remove the test specimens from the oven, allow to cool for at least 1 h.

7.8 After the test specimens have been allowed to cool (Note 1), remove the female-luer-to-closed-end adapter and

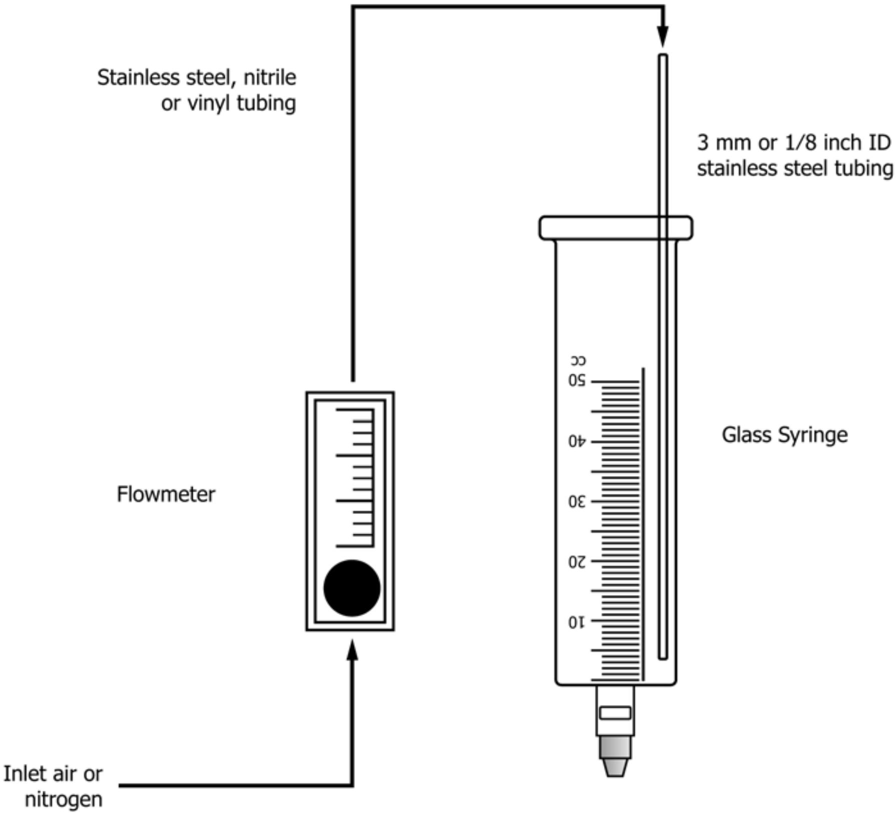

FIG. 1 Diagram of Sparging Setup with Syringe

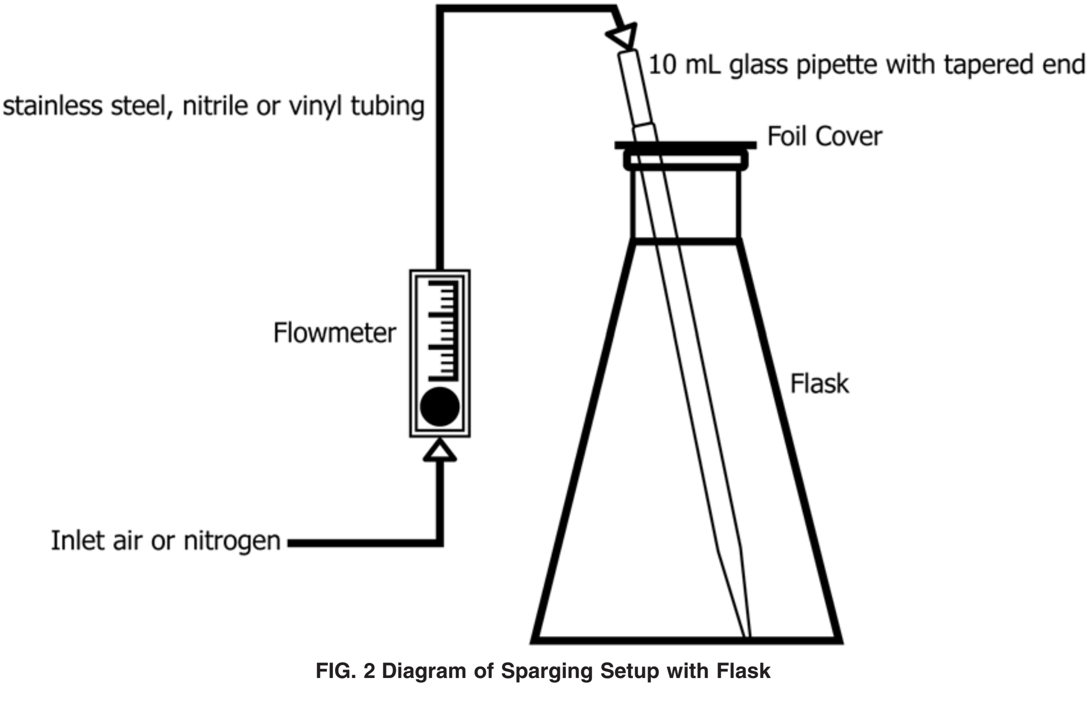

FIG. 2 Diagram of Sparging Setup with Flask

replace with normal stopcocks. Expel a small amount of oil (2-5 mL) from the stopcock to remove any air that may have entered into the syringe when changing out the female-luer-to-closed-end adapter.

NOTE 1—Periodically check for air ingress during the cooling of the syringe. In those cases where it is visible, it is advisable to perform the test again.

7.9 Perform dissolved gas-in-oil analysis (Note 2) on all four test specimens according to Test Method D3612 or IEC 60599. Whichever Test Method D3612 method is employed (A, B, or C) or IEC 60599, the following minimum detection limits must be met:

Hydrogen:	5 µL/L
Hydrocarbon gases:	1 µL/L
Carbon oxides:	25 µL/L
Atmospheric gases:	50 µL/L

NOTE 2—It is not usually necessary to conduct DGA analysis on the sparged samples after sparging but prior to heating to establish a baseline. Laboratory testing has shown that sparging samples containing up to 50 µL/L of acetylene will fully remove or significantly reduce all combustible gases.

7.10 The test results are to conform to the following:

Nitrogen and Oxygen:	±12 % of the average of the two analyses
Hydrocarbon Gases:	±10 % of the average of the two analyses and ±2 ppm
Hydrogen:	±10 % of the average of the two analyses and ±3 ppm

7.11 If the test results do not conform to the provisions provided in 7.10, the results are to be discarded and the test repeated.

7.12 Acetylene should not be detected. If acetylene is detected, then the results should be considered suspect and the process reviewed.

7.13 Report the results as detailed in Section 9.

8. Method B

8.1 Prepare a stainless steel column such as the one shown in Fig. 3. Alternatively, construct a glass column (a vacuum filtering apparatus of 47 mm has worked well) using partial vacuum to pull the oil through as opposed to being pressurized.

8.2 Fill the column with clay filtering medium with a mesh size of 30/60 so that the final ratio is 1 g of clay to 30 to 35 mL of insulating liquid. The clay can be preheated if desired (recommended range is 80 to 100°C).

8.3 Place the appropriate volume of insulating liquid in the sample reservoir to achieve the correct clay to liquid ratio. No less than 275 mL of insulating liquid is to be used as the clay will absorb some of the insulating liquid and not all of it is recoverable.

8.4 Heat the clay and insulating liquid with an external heating tape so that the exiting temperature of the insulating liquid is between 60 and 70°C.

8.5 The rate at which the insulating liquid is passed through the column is 3 to 5 mL per minute.

8.6 Collect the eluent from the clay-filled column in a flask that meets the requirements of 7.1.

8.7 Continue with the testing procedure as detailed in 7.2 through 7.11.

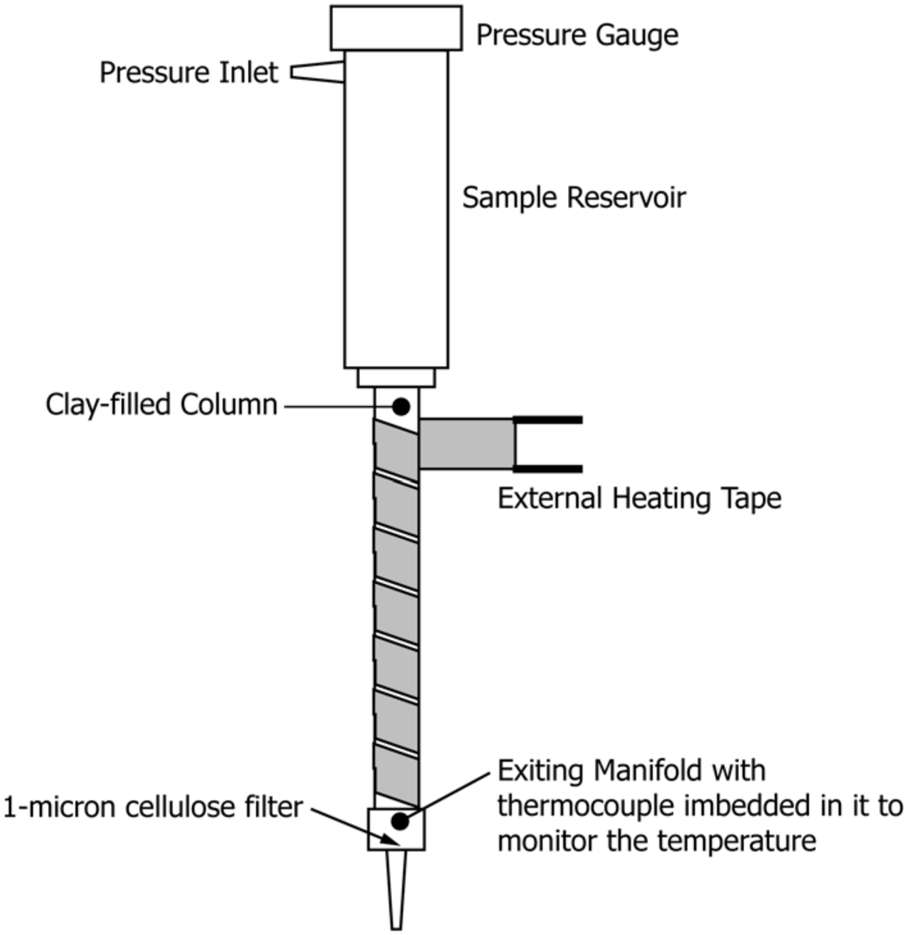

FIG. 3 Diagram of Clay Treatment Column

8.8 Report the results as detailed in Section 9.

9. Report

9.1 Report the following information:

9.1.1 Sample Identification.

9.1.2 Identify the sample as "As Received" or "After Being Processed by Clay Treatment".

9.1.3 Sparging gas.

9.1.4 Volume concentration in the insulating liquid, of each component gas, expressed in µL/L at STP (0°C and 760 torr).

10. Precision and Bias

10.1 *Precision*—Because this test method uses the analytical procedures in Test Method D3612, refer to the precision statements for that test method.

10.2 *Bias*—Because this test method uses the analytical procedures in Test Method D3612, refer to the bias statements for that test method.

11. Keywords

11.1 combustible gases; DGA; dissolved gases; dissolved gas-in-oil; insulating oil; low temperature gassing characteristics; stray gassing; transformer oil, clay treatment

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