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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Sampling Insulating Liquids for Gas Analysis and Determination of Water Content [1]

This standard is issued under the fixed designation D 3613; 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 practice covers sampling electrical insulating liquids having a viscosity less than 650 cSt at 40°C from apparatus for analysis of their gas content or for measurement of water content, or both. Guide D 117, Test Methods D 1533, and Test Methods D 3612 provide detailed information regarding gas and water testing. Definitions of terms used in this practice may be found in Terminology D 2864.

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.* Specific precautionary statements are given in Sections 6 and 7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 117 Guide to Test Methods and Specifications for Electrical Insulating Oils of Petroleum Origin[2]

D 923 Test Methods for Sampling Electrical Insulating Liquids[2]

D 1533 Test Methods for Water in Insulating Liquids[2]

D 2864 Terminology Relating to Electrical Insulating Liquids and Gases[2]

D 3612 Test Method for Analysis of Gases Dissolved in Electrical Insulating Oils by Gas Chromatography[2]

3. Significance and Use

3.1 A study of gases and moisture contained in insulating oils from transformers and other electrical power apparatus can frequently give an early indication of abnormal behavior of the apparatus, and may indicate appropriate action be taken on the equipment before it suffers greater damage. Specific gas and moisture content can be determined from oil samples for this purpose. Care must be taken in drawing samples to prevent contamination and to ensure a truly representative sample.

4. Apparatus

4.1 *Sample Containers*, may be glass syringes, stainless steel cylinders, or metal cans having flexible sides (to allow for volume changes caused by expansion or contraction of the oil).

4.1.1 *Glass Syringes,* of a suitable size terminated with a luer lock fitting to which is attached a three-way stopcock, should be used. Syringes having precision ground barrels and pistons are preferred.

4.1.2 *Stainless Steel Sampling Cylinders,* equipped with valves on each end may be used for sampling. These cylinders have the disadvantage of not allowing visual inspection of the interior of the cylinder.

4.1.3 *Flexible-Sided Metal Cans,* having screw caps may also be used.

4.2 *Flush Oil Container*—of at least 3.8 L (1 gal) to contain the required flush liquid during preparation and sampling.

4.3 *Clear Poly (Vinyl-Chloride) Tubing*—or any tubing that meets the requirements of this practice that is oil resistant. Size shall be selected to guarantee a gas and liquid tight fit at the sampling port and on the syringe or steel cap. To minimize waste and potential spills a length of 60 cm (24 in.) is suggested and has proven to be acceptable for manipulation of the collection container while attached to the sample port.

5. Cleaning of Apparatus

5.1 A proven method for cleaning sample containers and accessories is the use of a cleaning agent that completely dissolves liquid residue, and shall then be subjected to soap and water cleaning and water rinse followed by a distilled water rinse. See Test Methods D 923 for further details about general cleaning requirements.

5.2 Allow the container to drain for 10 min, and dry in a forced-draft oven at 110°C for not less than 1 h. When the drying period has expired, syringe pistons shall be placed in their barrels, valves closed; stainless steel cylinder valves should be closed; or cans sealed with their screw cap. It is desirable that stainless steel cylinders be evacuated and sealed after cleaning.

5.3 The poly(vinyl chloride) tubing should be replaced with new tubing each time a sample port is used. Cleaning the tubing should be considered impractical.

5.4 *Alternate Stainless Steel Cylinder Cleaning Method*:

5.4.1 Rinse the cylinder with a suitable volatile solvent.

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[1] This practice is under the jurisdiction of ASTM Committee D-27 on Electrical Insulating Liquids and Gases and are the direct responsibility of Subcommittee D27.07 on Physical Test.

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

5.4.2 Allow the cylinder to drain for 10 min with both valves full open, placing the cylinders in a vertical position.

5.4.3 Dry the cylinder by passing dry air through the cylinder until the cylinder is dry. This should involve a volume of air equivalent to at least 55 changes of the cylinder volume during the 15 min period of drying. The air may be dried by passing the air through two drying towers in series, each tower is at least 9 cm diameter and 28 cm in height. Towers filled with 6–16 mesh indicating Silica Gel have been found acceptable.

5.4.4 After 15 min close the cylinder valves and prepare the cylinders for use.

6. Preparation for Sampling

6.1 Check for positive pressure by first observing the pressure vacuum gage, if one is present, then check for positive pressure at a sample outlet by placing a slug of insulating fluid in a piece of clear plastic tubing and attaching it to the sample outlet. While observing the slug of insulating fluid, slowly crack the sample outlet valve open. If the slug moves towards the electrical apparatus, a negative pressure exists. Sampling should be discontinued. If the slug moves away from the electrical apparatus, a positive pressure exists. Samples can be obtained safely. Extreme care should be used in performing this procedure.

6.2 Place a flush-oil container under the main drain valve and remove the security pipe plug from the drain valve. Wipe the inside of the valve and threads with a clean lint-free cloth. Drain at least 1.9 L (2 qt) of fluid into the flush oil container to flush the drain valve and drain valve extension. One of two procedures may then be used to prepare the sample outlet.

6.2.1 *Procedure A*— Install the sample adapter on the drain valve (suitable thread size bushing adapter IPS to ⅜ in. bayonet) with a piece of oil resistant tubing attached. Flush the valve at installed sample adapter, flushing at least one more quart of fluid into the flush oil container before collecting sample.

6.2.2 *Procedure B*— Install the drain valve security plug. Attach oil-resistant tubing to the sample port on the drain valve and flush at least one more quart of fluid into the flush-oil container before collecting the fluid in the sample container.

6.3 This is an alternate procedure for purging the valve when it is not practical to flush oil through the drain valve or a flush container cannot be placed below the valve.

6.3.1 Place a flush oil container under the main drain valve or use absorbent material. Remove the security pipe plug from the drain valve. Wipe the inside of the valve and threads with a clean lint free cloth. Install the drain valve security plug.

6.3.2 Attach a 60-cm (24-in.) length of poly(vinyl chloride) tubing to the sample port on the drain valve and flush a minimum 1.9 L (2 qt) of oil into the flush oil container through the sample port.

7. Sampling Considerations

7.1 When it is necessary to obtain a sample from a piece of energized equipment not hermetically sealed, under no circumstances take the sample by any other means than from an external sampling valve.

7.2 All equipment filled with insulating fluid having a specific gravity less than 1, should be sampled from the bottom valve.

7.3 All equipment filled with insulating fluid having a specific gravity greater than 1, should be sampled at the top of the tank at the 25°C (77°F) liquid level, so that a top sample of the liquid may be obtained.

7.4 Maintain the insulating fluid within the electrical apparatus being sampled at a level that will not reduce the electric strength of the insulation system. Take extreme caution when samples are drawn from electrical apparatus having a small volume of insulating fluid.

7.5 Energized electrical apparatus being sampled must have a positive pressure at the sample outlet, so as not to admit an air bubble into the apparatus during the sampling process.

7.6 Do not sample electrical apparatus if only a drain plug is provided, as it would be difficult to control the flow.

7.7 Samples shall not be taken from energized instrument transformers.

7.8 Care must be taken in drawing samples to prevent contamination and to ensure a representative sample.

7.9 Collection of samples in metal cans will result in atmospheric air contaminating the sample and the loss of gas from the oil into the atmosphere. Test Method D 3612, Table 1, provides coefficients of variation for different gas-in-oil analysis from samples collected in the three containers recommended in this practice.

7.10 New oil resistant tubing shall be used for each apparatus sample port.

7.11 When collecting samples with glass syringes for the determination of dissolved gases or water, or both, the syringe should be filled 80 % full. The filled syringe should contain no air. However, gas-saturated samples will begin to release gases (bubbles) soon after sampling. Do not release any evolved gases (bubbles) since these gases must be included in the dissolved gas analysis. Package the filled syringe as soon as possible to protect the sample from light.

8. Collecting Samples Using Syringe

8.1 Perform the steps in Section 6. Attach the plastic tube to the syringe as shown in Fig. 1.

8.2 Adjust the equipment drain valve or the sample port valve for a gentle flow of fluid through the tubing with the syringe stopcock open (Fig. 1) to permit flushing of the stopcock. Position the handle toward the syringe.

8.2.1 The handle of the plastic stopcock always points to the closed port leaving the other two ports in open communication.

8.3 Turn the stopcock slowly to open the stopcock into the syringe (Fig. 2 handle in line with the flushing port). Allow the fluid to fill the syringe to maximum full mark (shown as 40 in Fig. 2). Immediately close the port into the syringe (Fig. 3 handle toward collection tube). Slowly depress the syringe piston until all the oil is evacuated from the syringe completing

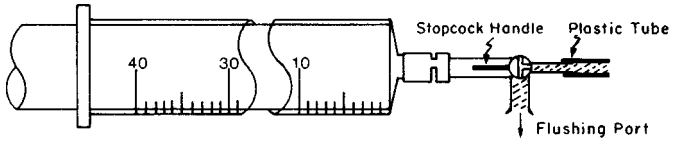

FIG. 1 Stopcock with Two Open Ports

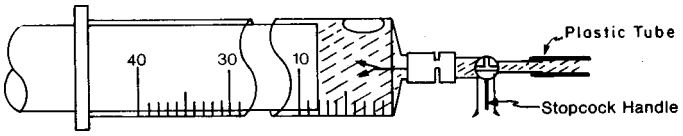

FIG. 2 Stopcock with Handle in Line with Flushing Port

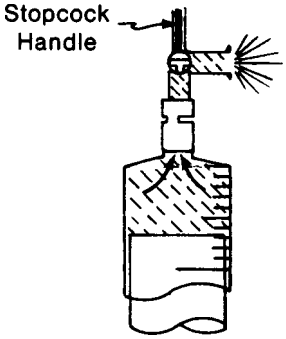

FIG. 3 Stopcock Up, Handle Away from Syringe

the first conditioning. Perform this condition procedure one more time.

8.3.1 If the quantity of oil in the apparatus is small and a visible change in level occurs when filling the syringe, the above conditioning steps may be omitted. A large variance from normal should be considered when evaluating the test results obtained when the syringe conditioning was not used.

8.3.2 If conditions warrant, the sample collection tubing may be removed from the syringe during the time the piston is depressed. In this case the flow of oil shall continue and should be directed into the flush container. Care shall be taken not to contaminate the syringe inlet port.

8.4 Turn the stopcock slowly to open the port to the syringe (Fig. 2 handle in line with the flushing port). Allow 10 mL of fluid to enter the syringe. Immediately close the port to the syringe (Fig. 3 handle toward collection tube).

8.5 With the syringe vertical (Fig. 3), the stopcock up, and the handle away from the syringe, eject any air bubbles and carefully depress the syringe piston far enough to leave 1 to 2 mL of fluid in the syringe. If all of the fluid is evacuated from the syringe, there is a greater chance of an air leak. Close the stopcock, with the handle toward the syringe.

8.6 To eliminate any possibility that air may be entrapped in the valve, let the fluid flow through the flushing port before the valve is turned to allow the syringe to be filled.

8.7 Open the stopcock (Fig. 2), with the handle in line with flushing port. Allow fluid pressure to push the piston back until the syringe is filled to approximately 80 % full. Do not pull the piston manually since this can result in bubble formation.

8.8 Close the stopcock (Fig. 4), with the handle toward syringe. Separate the syringe from the tubing and inspect for air bubbles. If air is present, discharge oil with the syringe vertical (stopcock up) and obtain another sample.

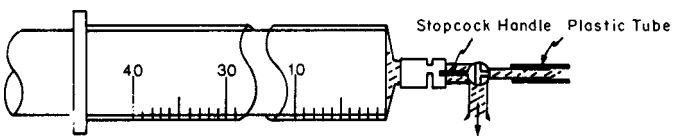

FIG. 4 Stopcock with Handle Towards Syringe

8.9 Remove the sample adapter, if used, and install the security plug with a non-hardening thread sealant.

9. Collecting Samples Using Stainless Steel Cylinder

9.1 Perform the steps in Section 6.

9.2 Hold the steel cylinder in a vertical position. Connect the oil-resistant tubing to the lower valve port on the steel cylinder and connect a short piece approximately 60 cm (24 in.) of clear oil-resistant tubing to the upper valve on the steel cylinder.

9.3 While keeping the cylinder in the vertical position, open the electrical apparatus drain valve or the sample port valve. Open the lower valve on the steel cylinder. Direct the short piece of plastic tubing towards the flush oil container and open the upper valve on the steel cylinder. With all three valves open and the cylinder held in a vertical position (see Fig. 5), flush the cylinder. 1.9 L (2 qt) of fluid should pass through the cylinder into the flush oil container.

9.4 If air bubbles are seen in the plastic tubing, the steel cylinder may be tapped lightly or shaken to dislodge any bubbles inside the cylinder. Flushing with oil should be continued until the flow out of the cylinder is free of any bubbles.

9.5 Tightly close the three valves in the following sequence: first close the upper cylinder valve tightly; then the bottom cylinder valve; followed by the electrical apparatus drain valve or sample port valve. Remove the sample adapter if used, and reinstall the security plug with a non-hardening thread sealant.

9.6 As a final check to determine that the cylinder has been properly filled, shake the cylinder and listen for the motion of the bubbles and the splashing of the oil. If any sound is heard, the cylinder should be drained and the sampling repeated.

10. Collecting Samples Using Flexible-Sided Metal Can

10.1 Perform the steps in Section 6.

10.2 When collecting the sample in a flexible-sided metal can, hold the sample container so that the fluid will run down the sides and limit aeration of the fluid. Partially fill the sample

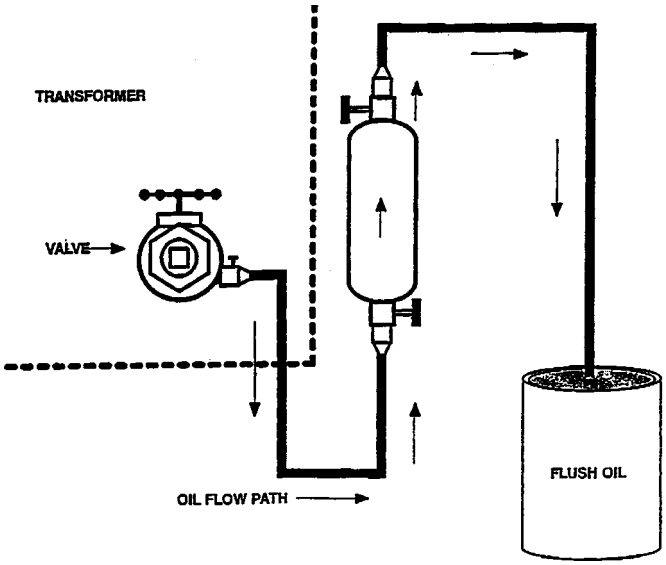

FIG. 5 Sampling with Stainless Steel Sampling Cylinder

container several times and slosh the fluid around to warm (to prevent condensation). Rinse the container, and properly discard the fluid after each rinse. The flow of fluid should be gentle and not interrupted from the start of the flushing of the valve and container to the completion of the final filling of the sample container.

10.3 Obtain the sample by allowing the fluid to flow down the sides of the container or from the bottom up, filling the container to overflowing. Once the container is full, install the cap immediately.

10.4 Close the drain valve, remove the sample adapter, if used, and install the drain valve security plug with a non-hardening thread sealant. The tubing should not be reused. Clean the sample adapter before reusing on any other oil-filled compartments or apparatus.

11. Identification

11.1 Attach a tag or label to each sample container showing the date of sampling, temperature of oil reading the top oil gage if available, and the serial number or identification number of the electrical apparatus.

12. Packaging and Shipping

12.1 Carefully package each container for forwarding to the laboratory for analysis. Do not allow the oil to be exposed to sunlight. Convenient cardboard cartons for storing and transporting syringes can be obtained. Samples should be forwarded to the laboratory as quickly as possible. The samples should not be stored.

13. Keywords

13.1 metal can; gas analysis; insulating liquids; stainless steel cylinder; syringe; water content