Draft Indian Standard

GASSING OF INSULATING LIQUIDS

UNDER ELECTRICAL STRESS AND IONIZATION

(First Revision)

FOREWORD

This *Draft* Indian Standard will be adopted by the Bureau of Indian Standards after the draft finalized on the recommendation of the Fluids for Electrotechnical Applications Sectional Committee will be approved by the Electrotechnical Division Council.

Methods for determination of gassing rate of insulating liquids under stress and ionization under hydrogen and nitrogen environment were earlier covered under IS 12475 (Part 1): 1988 and IS 12475 (Part 2): 1988 respectively. This revision combines both parts of IS 12475 as mentioned above and is being undertaken to bring it in line with latest practices.

Major changes w.r.t the previous edition include: Combining requirements of gassing rate of insulating liquids in hydrogen and nitrogen environment.

The electrical performance of oil impregnated paper insulation used in transformers, cables or capacitors is strongly affected by the presence of small gaseous bubbles which can give rise to partial discharges eventual insulation breakdown, Therefore, gassing properties of insulating liquids, that is, the tendency to absorb or evolve gases have been recognized as factors of major importance to characterize the dielectric liquids used as impregnant in ail paper insulation,

The gassing behaviour of any liquid is primarily a function of its chemistry but change in certain test parameters can modify the result significantly. The transaction from gas absorption to gas evaluation can occur at different temperatures and applied voltage for different oils.

In the preparation of this standard considerable assistance has been taken from IEC 60628:1985 ‘Gassing of insulating liquids under electrical stress and ionization’.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated expressing the result of a test or analysis shall be rounded off in accordance with IS 2: 2022 ‘Rules for rounding of numerical values (revised)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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GASSING OF INSULATING LIQUIDS

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SECTION ONE — GENERAL

1 SCOPE

This draft Indian Standard describes two procedures each using different apparatus to measure the tendency of insulating liquids to evolve or absorb gas when subjected, in cells having specific geometries, to electrical stress of sufficient intensity to cause an electric discharge through a gas phase in which a gas-oil interface is located.

The methods described in this standard are suitable for purchase specifications, general selection of insulating liquids, product development and quality assurance.

WARNING! Attention is called to national regulations associated with the use of high voltage, hydrogen and solvents.

2 GENERAL NOTES ON THE METHODS

2.1 These methods indicate whether insulating liquids are gas absorbing or gas evolving under the test conditions. The gassing behavior of any one insulating liquid is primarily a function of its chemistry but changes in certain test parameters can modify the results significantly.

2.2 These methods can operate under a variety of gas phase, temperature and voltage stress conditions. In order to establish uniform criteria of measurement, specific test conditions are specified which experience has shown to be most informative of the general performance expected from the liquid dielectric in electrical equipment should ionization occur.

At present, however, though it is generally agreed that gas absorbency of the impregnant has a positive effect in minimizing ionization problems in impregnated insulation systems used at high electrical stress, correlation of gassing-cell test results with equipment performance is limited. Engineering judgement is necessary in interpreting the test results in relation to any intended application.

2.3 Both methods, have been originally designed for the range of gassing rates characteristic of mineral insulating liquids. The use of these methods with other liquids may require some adaptations in the dimensions of the test cell.

SECTION TWO — METHOD A

3 OUTLINE OF METHOD

This method determines the gassing tendency of an insulating liquid under a hydrogen atmosphere and expresses the results in terms of gassing rate over a relatively short test period.

After being dried and saturated with hydrogen gas, the insulating liquid and the hydrogen pocket above the liquid are subjected in the specified cell to a radial electrical stress under the following experimental conditions:

voltage:	10 kV;
frequency:	50 Hz or 60 Hz;
temperature:	-80 °C;
test duration:	120 min at 50 Hz or 100 min at 60 Hz

The rate of evolution or absorption of gas resulting from reactions at the gas-oil interface, is calculated as volume per unit of time from changes in pressure with time.

4 APPARATUS

4.1 Gassing-Cell and Gas-Burette Assembly

The gassing-cell illustrated in Fig. 1, with dimensions given in Fig. 2, consists of the following components

a) Cell made of borosilicate glass with a relative permittivity of 5 ± 0.2 at 80 °C measured at a stated frequency (50 Hz or 60 Hz). The part under stress is constructed of 16 ± 0.2 mm inside diameter and 18 ± 0.2 mm outside diameter precision selected light wall tubing according to IS 18236. This cell has an outer electrode (earth) 60 mm high made of solvent-resistant silver paint with a vertical slit for observing the oil level and a copper band for earth connection.
b) Hollow high-voltage electrode made of 10 ± 0.1 mm outside diameter centreless-ground and polished stainless steel seamless tubing No. 11 according to ISO 683/XIII and containing a 1.0 mm stainless steel capillary tubing as a gas passage. The electrode shall be supported and centred by a precision machined 24/29 recessed polytetrafluoroethylene plug.
   A 3.0 mm needle valve (E) with gas inlet is on top of the electrode.

Note— After repeated tests at 80 °C, the shape of the polytetrafluoroethylene plug should be checked because it may deform and no longer be leak-tight.

c) Gas burette (Fig. 1) made of 7 mm outside diameter borosilicate glass tubing with an etched scale (mm), tapered glass joint 10/19 (G) for connecting to the gassing-cell, a by-pass stopcock (D) and three glass bulbs (A, B and C). The correlation between the reading (mm) and the volume (mm3) must be known.

Note — Increased capacity of gas-burette is required for highly gas absorbing liquids.

4.2 Heating Device

A transparent oil bath, preferably filled with silicone liquid, with thermostatic control and liquid circulating system to maintain the bath medium at 80±0.5 °C. The bath may be equipped with suitable supports for holding the gassing-cell and gas burette.

Note — If the level of the oil filling drops below a defined minimum, the high voltage should be disconnected automatically by safety switches. The bath may be provided with an effective circulating cooling system to allow rapid cooling after the test.

4.3 Transparent Safety Shield

Fitted with safety electrical interlock switches to protect the operator from parts under high voltage.

4.4 High-Voltage Transformer

The transformer and its controlling equipment shall be of such size and design that, with a filled gassing-cell in the circuit, the peak factor (ratio of peak value to rms value) of the test voltage shall not differ by more than ±5% from that of a sinusoidal wave while maintaining 10 kV ± 2%.

4.5 Thermometer

Any convenient thermometer for measuring a temperature of 80 ± 0.1 °C (for example, ISO 653 - STL/0.1/60/85).

4.6 Syringe

A convenient glass syringe, volume 10 cm^3.

5 REAGENTS

5.1 Hydrogen with oxygen content less than 10 mm^3/dm^3 and water content less than 2 mm^3/dm^3 from a cylinder with two-stage pressure reducer and a fine flow regulator.

5.2 Dibutyl phthalate, technical grade.

5.3 1,1,1-trichloroethane, technical grade.

5.4 n-heptane, analytical grade

5.5 Silicone vacuum grease.

6 PREPARATION OF THE APPARATUS

General remark:

As the gassing tendency of liquids may be strongly influenced by solvents, it is important that no traces of solvent remain after the cleaning procedure.

6.1 Clean the glass cell by first rinsing it inside and outside with 1,1,1-trichloroethane then with n-heptane. Then, refill the cell with n-heptane and scrub with a stiff brush of polyamide fibres to remove deposits from previous test.

Insert a smaller brush into the tapered joint (G) and scrub out silicone grease, taking care that none of the grease enters the cell. Again rinse with n-heptane and blow dry with clean compressed air.

Check the painted-on silver electrode, and touch up if necessary.

6.2 Clean the hollow electrode by blowing out the capillary tube with clean compressed air, rinsing the oil off the entire electrode with 1,1,1-trichloroethane and wiping off any deposit with tissue paper

Polish the surface of the stainless steel shaft of the electrode with a suitable device, such as a buffing wheel; wipe off the buffing compound carefully with tissue paper moistened with 1,1,1-trichloroethane. Rinse again first with 1,1,1-trichloroethane, then with n-heptane. Blow dry with clean compressed air and complete drying in an oven at 80 °C.

6.3 Apply a light coat of silicone vacuum grease to the stopcock (D) and the standard tapered joint (G) and assemble the glass cell and burette, but do not insert the electrode into the glass cell.

6.4 Fill the burette to the half-full mark with dibutyl phthalate

6.5 Clean the syringe with n-heptane then blow dry with compressed air

7 PROCEDURE

7.1 Filter about 10 cm^3 of the oil sample through a previously dried filter paper and rapidly introduce 5 ± 0.1 cm^3 of the filtered oil into the glass cell by means of the hypodermic syringe.

7.2 Lightly coat the polytetrafluoroethylene plug of the electrode with the test liquid (to act as a gas-seal) and insert the electrode into the glass cell.

7.3 Check the bath temperature, which shall be maintained at 80±0.5 °C during the test.

7.4 Suspend the gassing-cell and gas burette assembly in the oil bath at the level indicated in Figure 1, and connect the lead from the outside electrode to earth.

7.5 Attach the gas inlet and outlet connections. The gas outlet should lead outside the building, either directly or through a fume hood.

7.6 Close the stopcock (D) and open the valve (E) to allow the saturating gas to bubble through the test oil and the burette liquid at a steady rate of 3 dm^3 /h for 60 min.

7.7 Open the stopcock (D) and continue bubbling the saturating gas through the test oil for an additional 5 min.

7.8 After a total of 65 min of gas bubbling, first close the valve (E) and then the stopcock (D), making certain the liquid levels in the two legs of the burette are equal.

7.9 Connect the high-voltage lead to the centre electrode

7.10 Place the transparent safety shield in position and take the burette reading after checking the bath temperature.

7.11 Turn on the high-voltage and adjust to 10 kV.

7.12 Record the time and the burette level and check the observation slit on the outer electrode for onset of the gassing reaction.

7.13 After 10 min, record the burette level.

7.14 After an additional 120 min (if 50 Hz) or 100 min (if 60 Hz) again record the burette level and then turn off the high-voltage.

8 CALCULATION OF THE RESULTS

Calculate the gassing tendency in the presence of hydrogen as follows:

$$G = \left(B_{130\,(or\,100)} - B_{10} \right) K / t$$

where:

G = gassing tendency, in cubic millimeters per minute

$B_{130\,(or\,110)}$ = burette reading, in millimeters, at 130 (or 110) min of test

B10 = burette reading, in millimeters, at 10 min of test

K = burette constant = cubic millimeters per millimeter burette reading

t = test time of computed gassing rate

t (min) = 130-10 = 120 min if 50 Hz

t (min) = 110-10 = 100 min if 60 Hz

Value of G will be positive if gas is evolved and negative if gas is absorbed.

9 NUMBER OF TESTS

Tests should be run in duplicate.

10 REPORT

The report shall include the following:

a) IS 12475 — Method A;
b) gassing tendency (mm^3 /min), mean value of duplicate tests;
c) test voltage;
d) test voltage frequency (50 Hz or 60 Hz);
e) test temperature;
f) test duration;
g) gas phase.

11 PRECISION

Repeatability:

The results should be considered suspect if they differ by more than 0.3 + 0.26 |G| (where |G| is the absolute value of the average of the duplicate results in cubic millimeters per minute).

Note— Results on oils which are about neutral, will not achieve this repeatability

SECTION THREE — METHOD B

12 OUTLINE OF METHOD

This method determines the gassing characteristics of an insulating liquid and expresses the results in terms of the change in gas volume after a specified test period.

After being dried and saturated with nitrogen gas, the insulating liquid and a nitrogen pocket above the liquid are subjected in the specified cell to a radial electrical stress under the following conditions:

a) Voltage: 12 kV;

b) Frequency: 50 Hz or 60 Hz;
c) Temperature: 80 °C;
d) Test duration: 18 h at 50 Hz or 15 h at 60 Hz

The most significant single feature of the cell is the limited gas volume in contact with the liquid. Because of this factor, gaseous species evolved during the early stages of the gassing test can substantially modify the chemical nature of the gas phase and consequently influence both the rates and the net effect of competitive reactions at the oil-gas interface.

The quantity of gas absorbed or evolved is obtained from the observed changes in gas volume.

13 APPARATUS

13.1 Gassing-cell and burette assembly

The gassing-cell shown in Fig. 3, with dimensions as given in Figures 4 and 5, consists of the following components:

13.1.1 Glass cell precision bore (*see* Fig. 4) made of borosilicate glass tubing with permittivity of 5 + 0.2 (at 50 Hz and 80 °C) and dimensions as follows:

a) tube length: 180 ± 1 mm;
b) tube inside diameter: 16 ± 0.02 mm;
c) tube outside diameter: 20.8 + 0.02 mm.

The inner surface is fire-polished, the outside surface ground and machine-polished. The tube is provided with a machine-polished, optically clear plane bottom plate 6 + 0.1 mm thick, fused on square with tube axis and made of glass of the same composition as the tube.

A centring cone groove let into the bottom plate has a base of 4 mm and an angle of 90 ° at the tip of the corner.

The open end of the tube has a small beaded rim.

13.1.2 Outer (high voltage) electrode made of aluminium foil:

a) foil thickness: 0.1 mm;
b) foil width: 110 mm.

The foil is wound around the tube with its edge terminating flush with the edge of the end plate and fixed by any convenient manner (for example, by a suitable plastic adhesive tape).

The high voltage supply is connected to the foil by a braided copper lead with a clamp at the end.

13.1.3 Inner (earth) electrode (*see* Fig. 5) made of free-cutting steel, precision machined and polished to the dimensions shown in Fig. 5.

The centring cone protruding from the upper face of the electrode has a base of 4 mm diameter and an angle of 90 ° at the tip of the cone. The tip is slightly rounded.

All edges are slightly rounded and the electrode surface free from burrs, scratches, or other flaws. The electrode shall be handled with great care and only placed on surfaces covered with filter paper.

O-rings made of suitable resistant material, 11.3 mm inside diameter and 2.4 mm thick should be used to seal the cell.

The electrode is earthed by a braided copper lead with a clamp at the end

Note— Free-cutting steel, cold drawn, recommended limits of alloying constituents:

C: max. 0.13% Si: max. 0.05% P: max. 0.1% Mn: 0.6% to 1.2%
S: 0.18% to 0.25%

13.1.4 Gas-burette, volume 20 cm^3, graduated in 0.1 cm^3, with an etched scale:

a) outside diameter: 13 mm;
b) inside diameter: 11 + 0.5 mm

13.1.5 Connecting hose made of suitable resistant flexible material, preferably fluorinated elastomer, for connecting the inner electrode to the gas-burette:

a) hose length: 150 mm;
b) hose inside diameter: 6 mm;
c) wall thickness: 2 mm.

13.1.6 Capillary tubing made of polyethylene, for introducing the test gas (nitrogen) into the glass cell:

a) tube length: 750 mm;
b) tube inside diameter: 0.4 mm;
c) tube outside diameter: 1.1 mm.

13.1.7 Glass syringe, volume 5 cm^3.

13.1.8 Holding device (*see* Figure 6, page 33) preferably made of resin-bonded laminated paper with polyamide screws or of polymethylmethacrylate resin, for holding the test tube:

a) during the filling and assembly of the whole apparatus in the inverse position,
b) during the test, in the normal position, in the oil bath.

The holding device shall be equipped with guides and a resilient mounting for the gassing cell, with a spring-loaded thrust plate and a retainer to hold it in a correct central position,

and with guides and sockets for the high voltage and earth potential leads. The design shall be such as to withstand a voltage of 20 kV between high voltage and earth sockets.

13.2 *Heating device*
See **4.2**.

13.3 *Transparent safety shield*
See **4.3**.

13.4 *High voltage transformer*

The transformer and its controlling equipment shall be of such size and design that, with a filled gassing-cell in the circuit, the peak factor of the voltage (ratio of peak to rms voltage) shall not differ by more than ±5% from that of a sinusoidal wave while maintaining 12 kV (rms) ± 2%.

13.5 *Thermometer*
Any convenient thermometer for measuring a temperature of 80 ± 0.1 °C (for example, ISO 653-STL/0.1/60/85).

14 REAGENTS

14.1 1,1,1-trichloroethane (technical grade).

14.2 n-heptane (analytical grade).

14.3 Nitrogen, oxygen content less than 10 mm^3/dm^3 and water content less than 2 mm^3/dm^3, from a cylinder with 2-stage pressure reducer and a fine flow regulator.

15 PREPARATION OF APPARATUS

General remark:
As the gassing tendency of liquids may be strongly influenced by solvents, it is important that no traces of solvent remain after the cleaning process.

15.1 Dismantle the gassing-cell and burette.

15.2 Clean the test tube, the inner electrode, the gas-burette and the connecting tube by rinsing them inside and outside first with 1,1,1-trichloroethane then with n- heptane.

Normally, the inside of the test tube shall be scrubbed with a stiff polyamide brush to remove waxy deposits from the previous test. Moreover, it is useful to polish the surface of the inner electrode carefully from time to time with buffing compound and then with tissue paper moistened with 1,1,1-trichloroethane.

Rinse again first with 1,1,1-trichloroethane, then with n-heptane.

Blow dry with dry compressed air and complete drying in an oven at 80 °C.

15.3 Clean the syringe with n-heptane and then blow dry with dry compressed air.

16 PROCEDURE (*see* Fig. 7)

16.1 Wrap the high-voltage electrode round the outside of the glass cell and fix it with suitable adhesive tape. The electrode shall embrace the glass cell tightly and its top edge shall terminate level with the top edge of the end plate of the glass cell.

16.2 Insert the glass cell vertically in the holding device with the open end up.

16.3 Fit the connecting hose on to the gas-burette and the inner electrode and secure it with clamping rings.

Insert the capillary tube into the burette and push it through until it projects out of the mouth of the oil duct of the inner electrode and extends to the electrode.

16.4 Filter about 50 cm^3 of the oil sample through a previously dried filter paper and rapidly introduce 20 cm^3 of the filtered oil into the glass cell.

16.5 Carefully insert the inner electrode to the end plate of the tube in such a way that the oil slowly rises through the oil duct in the inner electrode and the connecting hose into the burette.

The capillary tube (*see* **16.3**) shall extend to the bottom of the test tube.

Clamp the gas-burette to a tripod so that the entire set-up is in a vertical position with the burette at the top and the cell and electrodes at the bottom. Then pour 5 cm^3 of the filtered oil into the burette by means of a syringe.

16.6 Connect the free end of the capillary tube to the nitrogen supply. Before doing this, thoroughly purge the connecting line with nitrogen.

16.7 Saturate the oil with dry nitrogen at a flow rate of 3 dm^3 /h for 1 h and at room temperature.

After that, shut off the nitrogen supply and carefully remove the nitrogen bubbles remaining in the test tube, the oil duct and the connecting hose by repeatedly squeezing the connecting hose.

16.8 Disconnect the burette from the tripod without separating the nitrogen connections.

Clamp the glass cell with the inner electrode to the holding device by means of the retainer and then turn the holding device upside down, keeping the gas-burette vertical. Then clamp the burette to the holding device.

16.9 Record the oil level in the burette.

Inject 3 cm^3 of the nitrogen directly from the nitrogen supply through the capillary tube into the test tube recording the volume injected from the indication on the burette.

Then remove the capillary tube.

16.10 Connect the high-voltage socket (on the holding device) to the high-voltage electrode and the earth potential socket (on the holding device) to the shank of the inner electrode by means of braided copper leads and clamp the latter in position.

16.11 Place the test apparatus in the oil bath at room temperature (*see* note), raise the temperature in about 1 h to the test temperature (80 ± 0.5 °C).

Note— Do not place the test apparatus in a hot bath, otherwise the glass might break.

16.12 About 1 h after the test temperature has been reached, i.e. no further change is noticeable in the oil level, record the oil level in the burette (*a* cm^3, time t_o).

Connect the high-voltage and earth potential leads.

Place the safety shield in position and then apply a voltage of 12 kV.

16.13 After 18 h (if 50 Hz) or 15 h (if 60 Hz) record again the burette level (*b* cm^3, time t_1).

16.14 Disconnect the test voltage, switch off the heater and start the circulating cooling system.

16.15 Before taking the test apparatus out of the oil bath, allow to cool to a temperature below 40°C.

Note — Do not remove the test apparatus from the hot bath, otherwise the glass might break.

17 CALCULATION OF THE RESULTS

Calculate the degree of gassing in the presence of nitrogen according to the following formula:

$$G = (a - b).\frac{p}{101.3}$$

where:
G = gassing tendency, in cubic centimeters
a = burette reading at the beginning of the test (time t_o) in cubic centimeters
b = burette reading at the end of the test (time t_1), in cubic centimeters
p = barometer reading in kPa

Value G will be positive if gas is evolved and negative if gas is absorbed.

Note— The stressed volume in the specified cell is about 10 cm^3. Accordingly, test results can range between -3.0cm^3 and +7.0 cm^3

18 NUMBER OF TESTS

Tests should be run in duplicate.

19 REPORT

The report shall include the following:

a) IS 12475 — Method B;
b) gassing tendency (cm^3), mean value of duplicate tests;
c) test voltage;
d) test voltage frequency (50 Hz or 60 Hz);
e) test temperature;
f) test period;
g) gas phase.

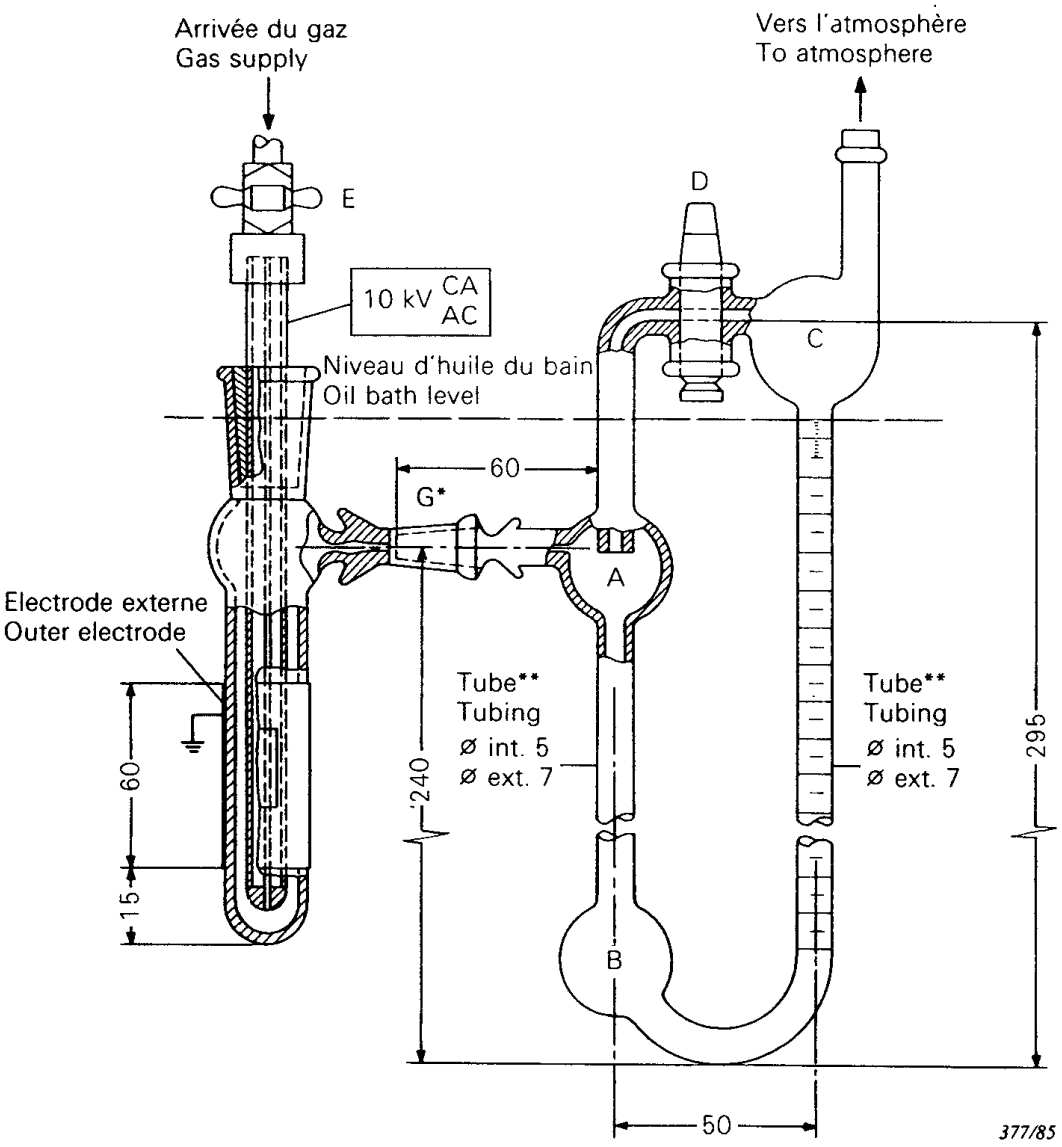
20 PRECISION

Not yet adequately defined.

However, the following figures may be considered indicative for judging the acceptability of result.

Repeatability:
Replicate results by the same operator should not be considered suspect unless they differ by more than 0.5 cm^3.

Reproducibility:
The results submitted by each of two laboratories should not be considered suspect unless they differ by more than 1 cm^3.



Dimensions en millimètres *Dimensions in millimetres*

A, B, C = Ampoules en verre
Glass bulbs

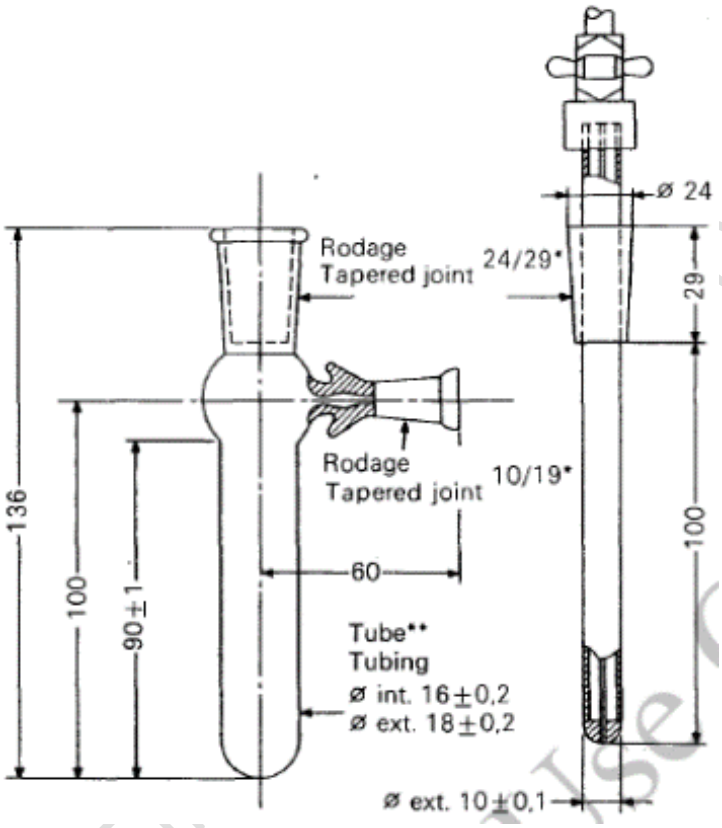
D = Robinet de dérivation
By-pass stopcock

E = Vanne à aiguille
Needle valve

G = Rodage conique en verre 10/19
Tapered glass joint 10/19

* Norme ISO 383
ISO Standard 383

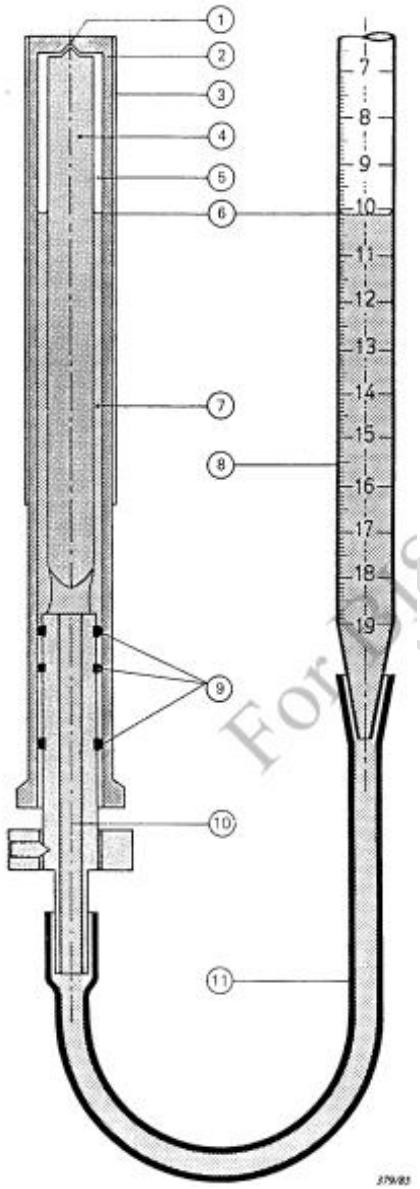
** Tube faible épaisseur suivant la Norme ISO 4803
Light wall tubing complying with ISO Standard 4803



Dimensions in millimetres
A, B, C = Glass Bulbs
D = By-Pass StopCock
E = Needle Valve
G = Tapped Glass Joint 10/19
*ISO Standard 383
**Light wall tubing complying with IS 18236

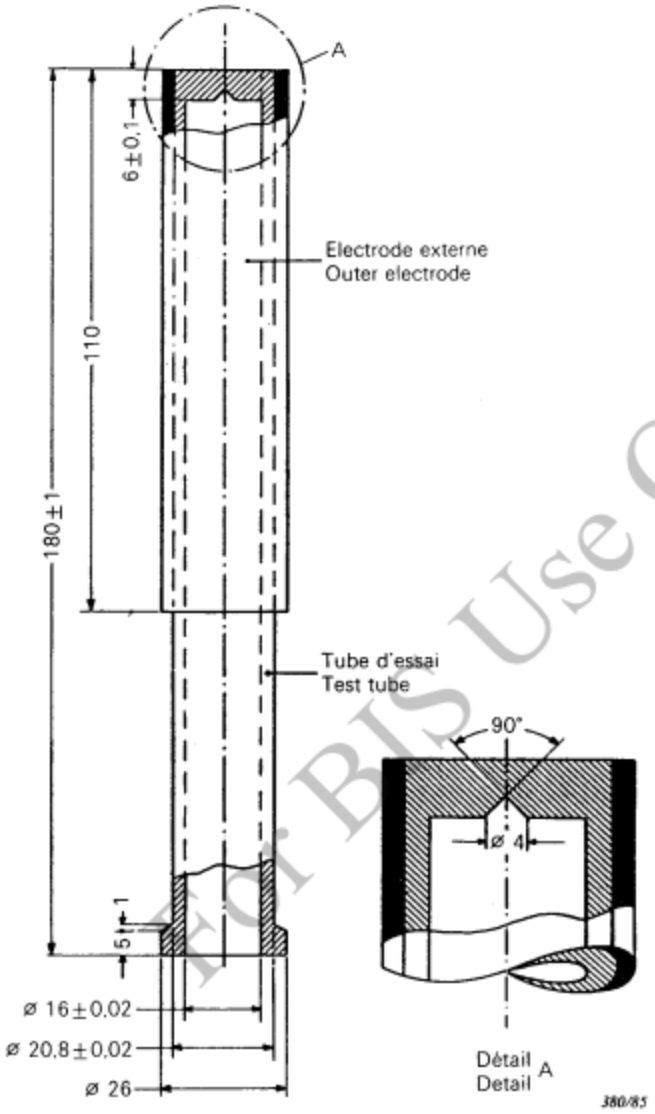
Fig. 1 SCHEMATIC DIAGRAM OF GASSING-CELL AND GAS-BURETTE ASSEMBLY

Fig. 2—DETAILED DIMENSIONS OF THE GLASS CELL AND THE INNER (HIGH-VOLTAGE) ELECTRODE



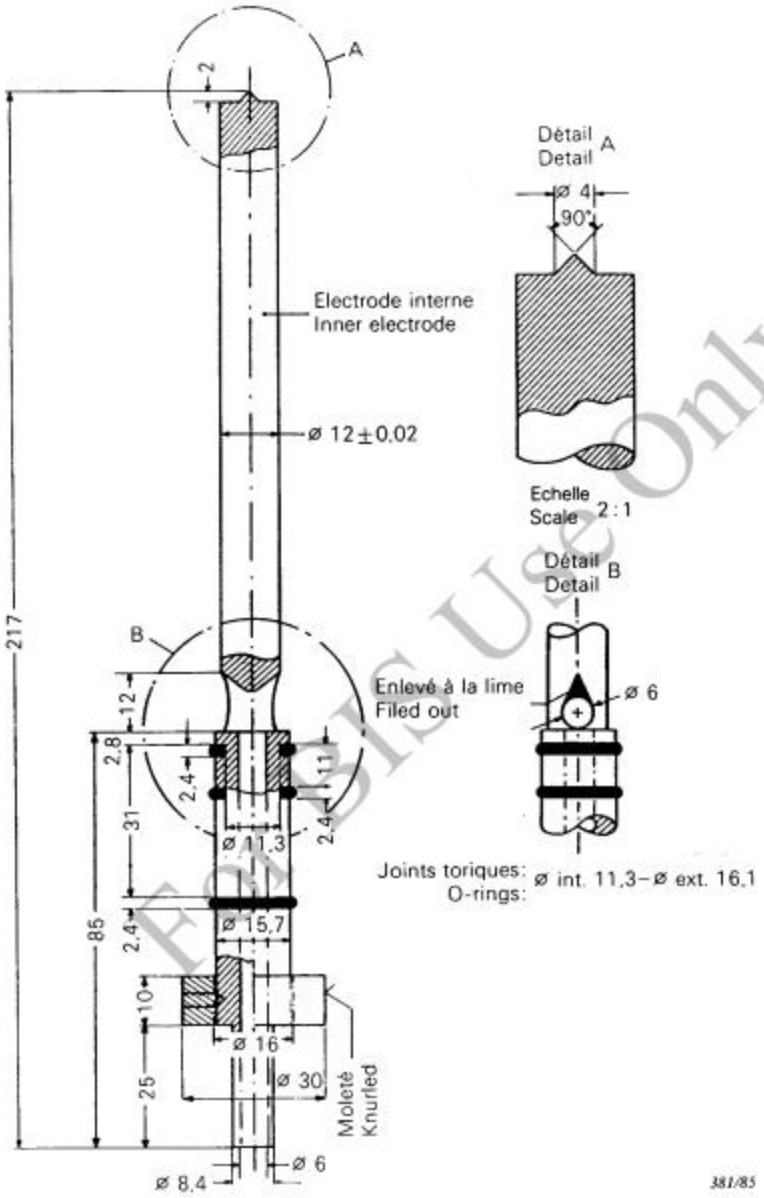
1. Centring Cone
2. Glass cell
3. Outer Electrode (AI foil)
4. Inner Electrode
5. Gas Pocket
6. Oil Level
7. Oil Specimen
8. Burette
9. Sealing rings
10. Oil duct
11. Connection tube

Fig. 3 GASSING-CELL AND BURETTE ASSEMBLY



Dimensions in millimeters

Fig. 4 GASSING-CELL



Dimensions in millimeters

Fig. 5 INNER ELECTRODE

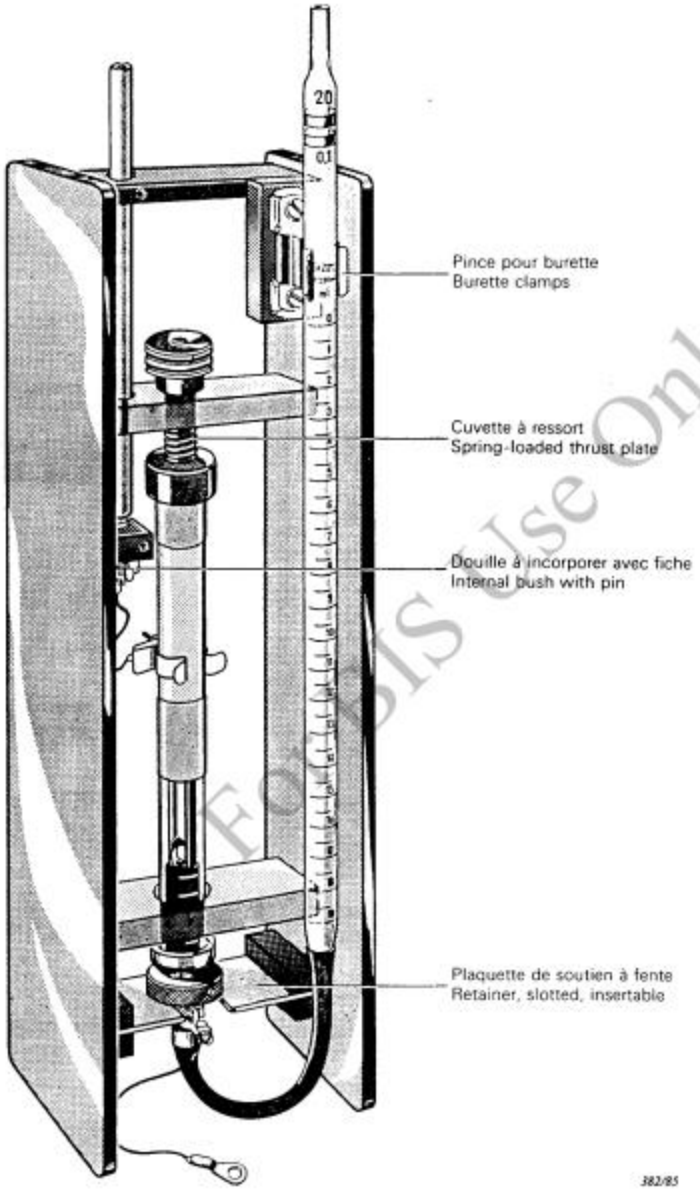


Fig. 6 HOLDING DEVICE, PREFERABLY MADE OF RESIN-BONDED LAMINATED PAPER CONNECTED BY MEANS OF COUNTERSUNK POLYAMIDE SCREWS.

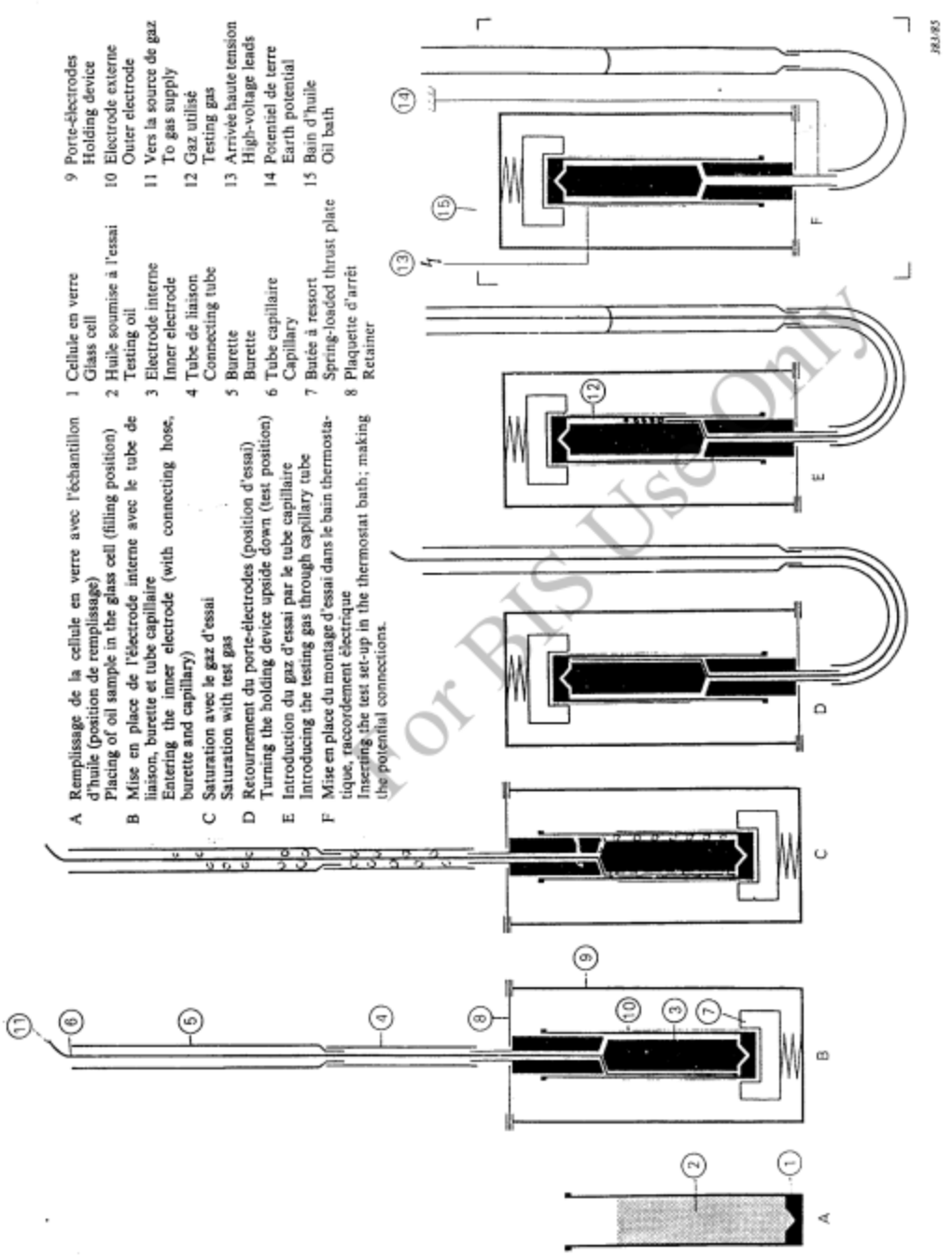


Fig. 7 SCHEMATIC REPRESENTATION OF THE TEST PREPARATIONS (DRAWINGS ARC NOT TO SCALE)