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Purified glycerol for industrial use — Determination of density at 20 °C

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2099 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in March 1971 by the Member Bodies of the following countries:

Austria	India	Thailand
Belgium	Italy	Turkey
Czechoslovakia	Netherlands	United Kingdom
Egypt, Arab Rep. of	New Zealand	U.S.A.
France	Portugal	U.S.S.R.
Germany	South Africa, Rep. of	
Hungary	Switzerland	

No Member Body expressed disapproval of the document.

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Purified glycerol for industrial use – Determination of density at 20 °C

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies methods for the determination of the density at 20 °C of purified glycerol (i.e. free from substances in suspension and crystallized salts) for industrial use. A choice of two pyknometers is provided to carry out this determination; the Gay-Lussac pyknometer, with ordinary or wide neck, and the Reischauer pyknometer with a graduated neck.

2 USE OF THE GAY-LUSSAC PYKNOMETER

2.1 Principle

Determination of the capacity of the pyknometer by measuring the mass of water that it contains at 20 °C.

Measurement of the mass of the volume of sample contained in this pyknometer at 20 °C.

Calculation of the quotient which results from dividing the mass of the sample by the volume of the pyknometer corresponding to this mass, taking into account a buoyancy correction to the apparent masses measured.

2.2 Apparatus

2.2.1 *Gay-Lussac pyknometer,* ordinary, closed by a ground glass stopper fitted with a capillary tube (Figure 1a), or the same pyknometer with a wide neck (Figure 1b) closed by a ground glass stopper fitted with a capillary tube of 2 mm internal diameter.

Capacity : approximately 50 ml.

2.2.2 *Water bath,* capable of being maintained at 20 ± 0.1 °C.

2.3 Procedure

2.3.1 *Calibration of the pyknometer*

Clean and dry the pyknometer (2.2.1). Dab it with a cloth lightly soaked in acetone to disperse any electrostatic charge. After 2 to 3 min weigh it empty but with its stopper, to the nearest milligram. Fill it with distilled water, freshly boiled and then cooled to a temperature slightly below 20 °C, insert the stopper and place the pyknometer in the water bath (2.2.2) for at least 10 min.

While keeping the pyknometer in the water bath, remove the excess liquid so that the level is exactly flush with the end of the capillary tube. Withdraw the pyknometer from the water bath and hold it for 1 min in a stream of water at a temperature below 20 °C. Dry its outside with a lint-free cloth and wipe it with a cloth lightly soaked in acetone. After 2 to 3 min, reweigh the pyknometer to the nearest milligram and calculate, by difference, the apparent mass of the water it contains.

2.3.2 *Determination.*

Empty the pyknometer, rinse it with acetone and dry it. Then fill it to half way up the neck with the laboratory sample, previously brought to a temperature slightly below 20 °C, avoiding the formation of bubbles. Insert the stopper, rinse the outside with distilled water at a temperature slightly below 20 °C and immerse it in the water bath (2.2.2) to a depth half way up the ground portion leaving it there for at least 30 min.

Carry out the operations described in the second paragraph of 2.3.1 to determine the apparent mass of the sample.

2.4 Expression of results

Density of the sample at 20 °C is given, in grams per millilitre, by the formula :

$$\frac{m_2 + A}{m_1 + A} \times \rho_e \qquad \text{... (1)}$$

where

m_1 is the apparent mass, in grams, of water needed to fill the pyknometer at 20 °C;

m_2 is the apparent mass, in grams, of the test portion;

ρ_e is the density of water at 20 °C, 0.998 2 g/ml;

A is the buoyancy correction given by :

$$\rho_a \times m_1$$

ρ_a being the density of air, approximately 0.001 2 g/ml[1];

m_1, the apparent mass, in grams, of water at 20 °C, is approximately equal to its volume V, in millilitres. The difference which results, in the calculation of the formula (1), does not affect the fourth decimal place.

Express the results correct to three places of decimals, the precision of the method being of the order of 0.000 5 g/ml in absolute value.

1) This value varies slightly with atmospheric conditions, but normal variations have a negligible effect on density determinations.

NOTES

1 The density in air at 20 °C, relative to water at 20 °C, in current commercial use, can be obtained by the formula :

$$\frac{m_2}{m_1}$$

2 Formula (1) can also be written

$$\frac{m_2 + A}{V}$$

in which V is the volume, in millilitres, of the pyknometer at 20 °C :

$$V = \frac{m_1 + A}{\rho_e}$$

If it is necessary to carry out numerous determinations, it may be practical not to determine V (and, consequently, m_1) at each measurement but merely to check that these remain constant with time.

3 USE OF THE REISCHAUER PYKNOMETER

3.1 Principle

Calibration of the pyknometer by measuring the masses of water introduced up to the different graduation lines.

Measurement of the mass of the volume of sample contained in the pyknometer at 20 °C up to a certain graduation line.

Calculation of the quotient which results from dividing the mass of the sample by the volume of the pyknometer corresponding to this mass, taking into account a buoyancy correction to the apparent masses measured.

3.2 Apparatus

3.2.1 *Reischauer pyknometer with graduated neck,* closed by a ground glass stopper extended by a capillary tube of about 0.5 mm internal diameter (Figure 2).

Capacity : approximately 50 ml.

3.2.2 *Water bath,* capable of being maintained at 20 ± 0.1 °C.

3.2.3 *Filling device* for the pyknometer.

A choice of two methods is provided :

3.2.3.1 Filling by pressure, by means of a hypodermic syringe with a needle of sufficient size to allow the easy passage of the sample without touching the wall of the graduated neck.

3.2.3.2 Filling under vacuum (Figure 2) by means of :

- a dropping funnel with a thin stem allowing it to be placed in the neck of the pyknometer without touching the wall;
- a Witt's apparatus, with ground glass lid fitted with a rubber stopper drilled with a hole of diameter 1 mm greater than that of the stem of the dropping funnel.

The side tube is connected to a filter pump and is fitted with an adjustable clamp enabling a slightly lower pressure to be created in the interior of the flask, so as to facilitate the flow of the glycerol through the stem of the funnel.

3.3 Procedure

3.3.1 *Calibration of the pyknometer*

Clean and dry the pyknometer (3.2.1). Dab it with a lint-free cloth lightly soaked in acetone to disperse any electrostatic charge. After 2 to 3 min weigh it, empty but with its stopper, to the nearest milligram.

By means of one of the filling devices (3.2.3), introduce, up to one of the lower graduation lines, distilled water freshly boiled and then cooled to a temperature slightly below 20 °C, taking care not to wet the inner wall of the neck above the level of the liquid. Insert the stopper and place the pyknometer in the water bath (3.2.2) for at least 10 min. Read the graduation line corresponding to the water level, withdraw the pyknometer, wipe it and dab it with a cloth lightly soaked in acetone. After 2 to 3 min weigh it to the nearest milligram and calculate, by difference, the apparent mass of the water it contains.

Then add a little water so as to raise the level successively to one-third of the way up the graduation lines, two-thirds of the way up the graduation lines, and then to the top of the graduation lines. For each of these levels, repeat the operations of temperature adjustment, reading and weighing described above.

Calculate the volumes of water at 20 °C, corresponding to the four graduation lines read, according to the following formula :

$$\frac{m_1 + A}{\rho_e} \qquad \text{... (2)}$$

where

m_1 is the apparent mass, in grams, of water at 20 °C obtained respectively for each of the four graduation lines;

ρ_e is the density of water at 20 °C, 0.998 2 g/ml;

A is the buoyancy correction given by :

$$\rho_a \times m_1$$

ρ_a being the density of air, approximately 0.001 2 g/ml[1];

m_1, the apparent mass, in grams, of water at 20 °C, is approximately equal to its volume V in millilitres. The difference which results, in the calculation of the formula (2), does not affect the fourth decimal place.

From the values found, calculate the mean volume contained between two successive graduation lines and the mean volume of the pyknometer up to the first graduation line.

Draw up a calibration table giving the volume of the pyknometer for each graduation line to check that the section of the neck is constant throughout its height.

1) This value varies slightly with atmospheric conditions, but normal variations have a negligible effect on density determinations.

Discard the pyknometer if the neck section is not constant throughout its height.

NOTE — If it is desired to determine the density of the sample at 20 °C in air relative to water at 20 °C, which is common commercial practice in certain countries, it is more convenient to carry out the calibration by making the calculations on apparent masses of water at 20 °C instead of by volume, in this case, without the correction for buoyancy of air. For this purpose calculate, from the mass obtained for each of the four graduation lines read, the mean apparent masses of water at 20 °C, contained respectively

- between two successive graduation lines; and
- in the pyknometer up to the first graduation line.

Draw up a calibration table giving the apparent mass of water, at 20 °C, contained in the pyknometer for each graduation line.

3.3.2 *Determination*

Clean, dry and weigh the pyknometer as described in 3.3.1. Then fill it up to a certain graduation line with the sample, previously brought to a temperature slightly below 20 °C. To carry out this operation, use one of the filling devices (3.2.3) avoiding the formation of air bubbles and the wetting of the inner wall of the neck above the level of the sample. Replace the stopper and place the pyknometer in the water bath (3.2.2) for at least 30 min.

Read the graduation line corresponding to the level of the liquid, withdraw the pyknometer, wipe it and dab it with a cloth lightly soaked in acetone, then weigh it and calculate, by difference, the apparent mass of the test portion.

3.4 Expression of results

Density of the sample at 20 °C is given, in grams per millilitre, by the formula :

$$\frac{m_2 + A}{V} \qquad \text{... (3)}$$

where

m_2 is the apparent mass, in grams, of the test portion;

V is the volume of the pyknometer, in millilitres, corresponding to the graduation line reached by the sample;

A is the buoyancy correction given by :

$$\rho_a \times V$$

ρ_a being the density of air at 20 °C, approximately 0.001 2 g/ml[1].

Express the results correct to three places of decimals, the precision of the method being of the order of 0.000 5 g/ml in absolute value.

NOTE — The density in air at 20 °C, relative to water at 20 °C, in current commercial use, can be obtained by the formula :

$$\frac{m_2}{m_1}$$

where

m_2 is the apparent mass, in grams, of the test portion;

m_1 is the apparent mass, in grams, of the water corresponding to the graduation line reached by the sample.

4 TEST REPORT

The test report shall include the following particulars :

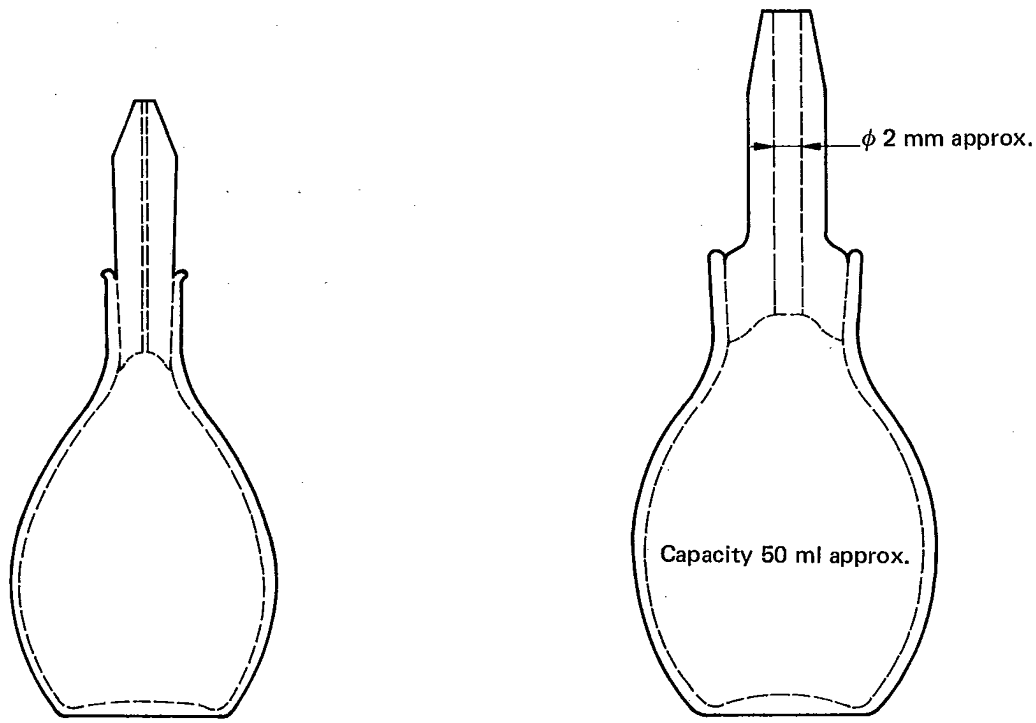
a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.

1) This value varies slightly with atmospheric conditions, but normal variations have a negligible effect on density determinations.



a) Ordinary type

b) Wide-necked type

FIGURE 1 — **Gay-Lussac pyknometer**

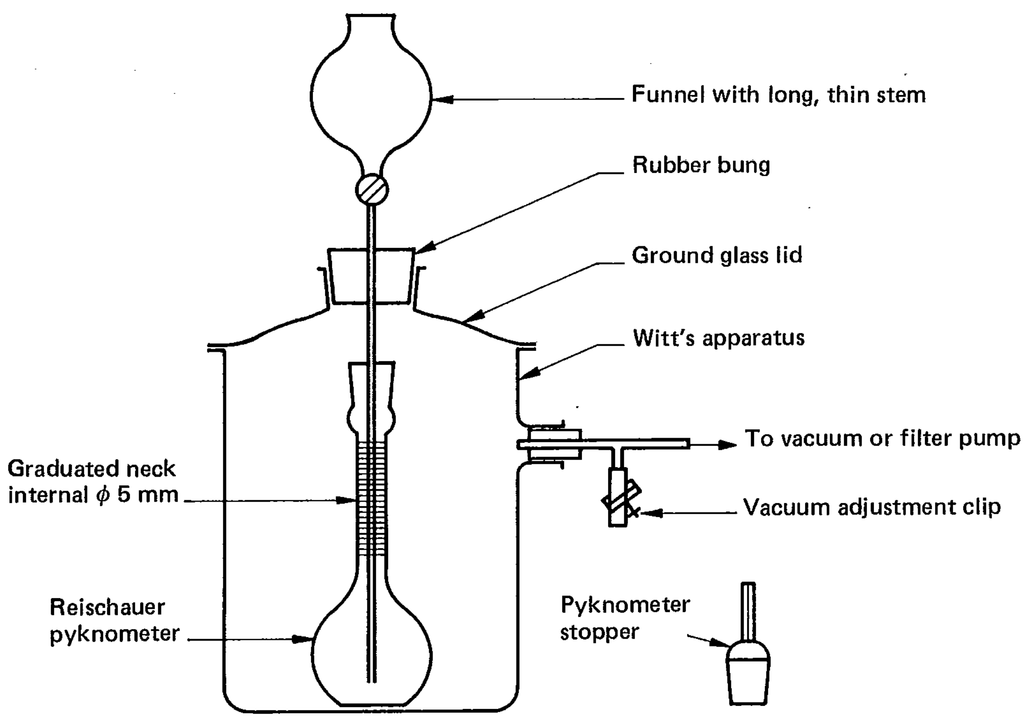


FIGURE 2 — **Device for vacuum-filling the Reischauer pyknometer**