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R 901

ALUMINIUM OXIDE PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

DETERMINATION OF ABSOLUTE DENSITY

PYKNOMETER METHOD

1st EDITION

December 1968

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BRIEF HISTORY

The ISO Recommendation R 901, *Aluminium oxide primarily used for the production of aluminium – Determination of absolute density – Pyknometer method*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question by the Technical Committee began in 1962 and led, in 1966, to the adoption of a Draft ISO Recommendation.

In May 1967, this Draft ISO Recommendation (No. 1171) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Israel	South Africa, Rep. of
Belgium	Italy	Spain
Bulgaria	Japan	Sweden
Canada	Korea, Dem. P. Rep. of	Switzerland
Czechoslovakia	Korea, Rep. of	Thailand
France	Netherlands	Turkey
Germany	New Zealand	U.A.R.
Hungary	Norway	United Kingdom
India	Poland	U.S.A.
Iran	Portugal	U.S.S.R.
Ireland	Romania	Yugoslavia

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in December 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 901 December 1968

ALUMINIUM OXIDE PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

DETERMINATION OF ABSOLUTE DENSITY

PYKNOMETER METHOD

1. SCOPE

This ISO Recommendation describes a method for the determination of the absolute density of aluminium oxide primarily used for the production of aluminium.

2. PRINCIPLE

Determination by a pyknometer method of the absolute density of aluminium oxide after complete degasification.

3. REAGENTS

3.1 *Xylene*, $d = 0.860$ to 0.865, distilling between 138 and 144 °C (see clause 7.1).

3.2 *Ethanol*, 95 % (V/V) solution, $d = 0.81$ approximately.

3.3 *Diethyl ether*, $d = 0.715$ approximately.

4. APPARATUS

Ordinary laboratory apparatus and

4.1 *Pyknometer* (see Fig. 1) consisting of

(*a*) *flask* (A), approximately 25 ml capacity, fitted with a side arm with a ground-glass cover (F);

(*b*) *thermometer* (B), covering the range 15 to 25 °C graduated in intervals of 0.1 °C, which can be fitted to the flask by a ground-glass joint.

4.2 *Degassing and filling apparatus*, (see Fig. 2), consisting of a tap funnel (C) of about 50 ml capacity, fitted with a side arm, with a tap (D) for connecting to a vacuum pump. This can be fitted to the pyknometer flask (A) by means of a conical ground-glass joint (E).

4.3 *Water bath*, controlled at a temperature of 20 ± 0.1 °C.

4.4 *Vacuum pump*, capable of giving a vacuum below 10 mmHg.

4.5 *Mercury manometer.*

5. PROCEDURE

General instructions

(1) Always weigh the pyknometer with the thermometer and the side tube cover in position.

(2) Always weigh to the nearest 0.1 mg.

(3) When the pyknometer contains liquid, stabilize its temperature at 20 ± 0.1 °C.

5.1 Determination

5.1.1 *Determination of the mass of the pyknometer.* Wash the pyknometer (4.1) including its accessories, with a lukewarm mixture of sulphuric acid and chromium trioxide. Thoroughly rinse, first with tap water, then with distilled water and ethanol (3.2) and finally with diethyl ether (3.3). Thoroughly dry the apparatus and weigh.

Let M_0 be the mass, in grammes, of the dry pyknometer.

5.1.2 *Determination of the volume of the pyknometer.* Fill the pyknometer (4.1) with distilled water and connect via the degassing apparatus (4.2) to the vacuum pump, the mercury manometer (4.5) being inserted to control the vacuum. Close the side tube of the pyknometer with its cover (F) and slowly open tap (D) and apply the vacuum for approximately 15 minutes. Occasionally tap the walls of the pyknometer to facilitate the release of any air bubbles. Restore atmospheric pressure in the pyknometer, disconnect it from the degassing apparatus (4.2) and put the thermometer (B) in position.

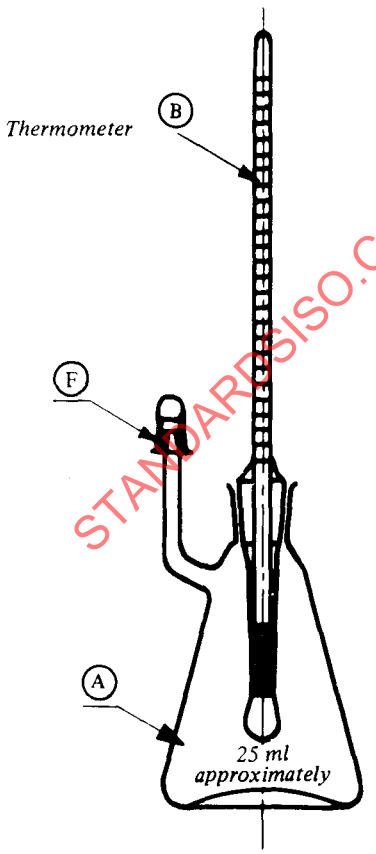

FIG. 1 – **Pyknometer**

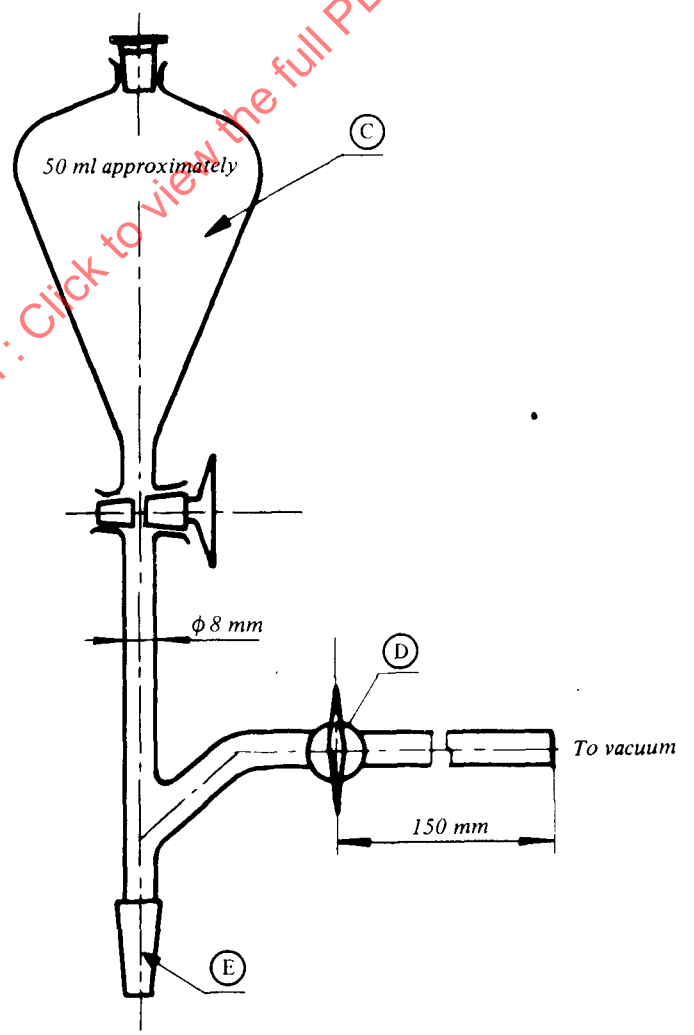

FIG. 2 – **Degassing apparatus**

Stabilize the temperature of the pyknometer in the water bath previously adjusted to 20 ± 0.1 °C. Completely fill the side tube with water, using a length of narrow glass tubing. Remove the pyknometer from the water bath, cool slightly under running cold water and close the side tube with its ground-glass cover (F). Carefully dry the pyknometer and weigh.

The volume V of the pyknometer, in millilitres, is given by the following formula :

$$V = \frac{(M_1 - M_0)}{0.9982}$$

where

M_1 is the mass, in grammes, of the pyknometer filled with degassed distilled water,

M_0 is the mass, in grammes, of the dry pyknometer (see clause 5.1.1),

0.9982 is the absolute density of water (g/ml) at 20 °C.

5.1.3 *Determination of the absolute density of xylene*. Fill the pyknometer (4.1) with xylene (3.1) after having determined its mass as in clause 5.1.1. Carry out the procedure given in clause 5.1.2, i.e. degas, stabilize the temperature at 20 ± 0.1 °C in the water bath, fill, cool, and finally weigh. The weighing should be carried out quickly to avoid loss of xylene by evaporation.

The mass M_x of xylene equivalent to the volume V of the pyknometer is given by the following formula :

$$M_x = (M_2 - M_0)$$

where

M_2 is the mass, in grammes, of the pyknometer full of xylene,

M_0 is the mass, in grammes, of the dry pyknometer (see clause 5.1.1).

The absolute density (g/ml) of xylene (ρ_x) is given by the formula :

$$\rho_x = \frac{M_x}{V}$$

where

M_x is the mass, in grammes, of xylene equivalent to the volume V of the pyknometer,

V is the volume, in millilitres, of the pyknometer (see clause 5.1.2).

5.1.4 *Test portion*. Transfer to the flask of the pyknometer (4.1), previously weighed in accordance with clause 5.1.1, about 10 g of dry test sample* and weigh. The mass of the test portion (M_e) is given by the following formula :

$$M_e = (M - M_0)$$

where

M is the mass, in grammes, of the pyknometer and test portion,

M_0 is the mass, in grammes, of the dry pyknometer (see clause 5.1.1).

* See ISO Recommendation R 802, *Aluminium oxide primarily used for the production of aluminium – Preparation and storage of test samples*, clause 2.3.

5.1.5 *Determination of the absolute density of the aluminium oxide*. Moisten the ground-glass joints of the pyknometer containing the test portion (5.1.4) with a little xylene (3.1) and insert the degassing apparatus (4.2). Connect the latter to the vacuum produced by the pump (4.4) and controlled by means of the mercury manometer (4.5). Close the side tube with its cover (F), gently open tap (D) and apply vacuum for 15 minutes. Close tap (D) and slowly run in xylene from the tap funnel (E) until the test portion is just covered. Carefully re-open tap (D), still connected to the vacuum pump, and occasionally tap the walls of the pyknometer to facilitate the release of air bubbles.

Then fill the pyknometer with xylene (3.1) up to the ground-glass joint and insert the thermometer (B).

Stabilize the temperature of the pyknometer in the water bath (4.3) previously adjusted to 20 ± 0.1 °C. Completely fill the side arm with xylene, using a length of narrow glass tubing. Remove the pyknometer from the water bath, cool slightly under running cold water and close the side arm with its ground-glass cover (F). Dry and weigh the pyknometer quickly, because of the tendency for xylene to evaporate.

The total mass of the test portion and additional xylene required to fill the pyknometer ($M_{(e+x')}$) is given by the following formula :

$$M_{(e+x')} = (M_3 - M_0)$$

where

M_3 is the mass, in grammes, of the pyknometer containing the test portion and filled with xylene,

M_0 is the mass, in grammes, of the dry pyknometer (see clause 5.1.1).

6. EXPRESSION OF RESULTS

The absolute density (ρ) of aluminium oxide, expressed in grammes per millilitre, is given by the following formula :

$$\frac{M_e}{(M_e + M_x) - M_{(e+x')}} \times \rho_x$$

where

M_e is the mass, in grammes, of the test portion (5.1.4),

M_x is the mass, in grammes, of xylene to fill the pyknometer volume (V),

$M_{(e+x')}$ is the mass, in grammes, of the test portion and of the additional xylene required to fill the pyknometer volume (V),

ρ_x is the absolute density of xylene (see clause 5.1.3).

NOTE. – Indicate the absolute density with three decimals, two of which should be significant figures.