INTERNATIONAL STANDARD 901

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Aluminium oxide primarily used for the production of aluminium — Determination of absolute density — Pyknometer method

Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Détermination de la masse volumique — Méthode pycnométrique

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47 has reviewed ISO Recommendation R 901 and found it technically suitable for transformation. International Standard ISO 901 therefore replaces ISO Recommendation R 901-1968 to which it is technically identical.

ISO Recommendation R 901 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 901 into an International Standard :

Egypt, Arab Rep. of

Printed in Switzerland

Aluminium oxide primarily used for the production of aluminium — Determination of absolute density — Pyknometer method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a pyknometer method for the determination of the absolute density of aluminium oxide primarily used for the production of aluminium.

2 REFERENCES

ISO 802, *Aluminium oxide primarily used for the production of aluminium — Preparation and storage of test samples.*

ISO 2927, *Aluminium oxide primarily used for the production of aluminium — Sampling.*

3 PRINCIPLE

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

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Xylene, ρ 0,860 to 0,865 g/ml, distilling between 138 and 144 °C.

NOTE — To ensure absence of a volatile fraction, the xylene shall distil within the given range.

4.2 Ethanol, 95 % (*V/V*) solution, ρ approximately 0,81 g/ml.

4.3 Diethyl ether, ρ approximately 0,715 g/ml.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Pyknometer (see figure 1), consisting of

5.1.1 Flask (A), of capacity approximately 25 ml, fitted with a side arm with a ground glass cover (F).

5.1.2 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.

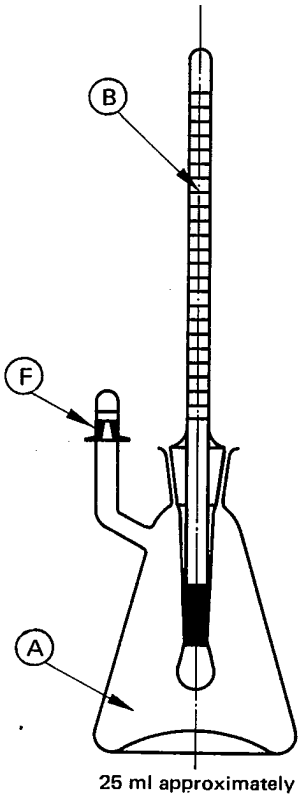

FIGURE 1 — **Pyknometer (5.1)**

5.2 Degassing and filling apparatus (see figure 2), consisting of a tap funnel (C) of capacity about 50 ml, fitted with a side arm, with a tap (D) for connecting to the vacuum pump (5.4). This can be fitted to the pyknometer flask (A) by means of a conical ground glass joint (E).

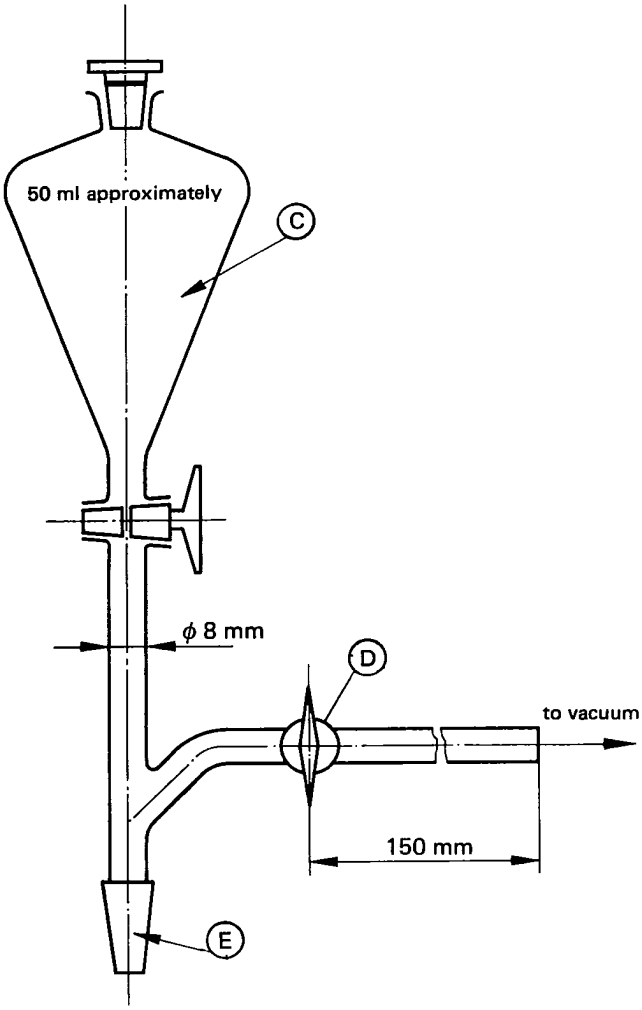

FIGURE 2 — **Degassing apparatus (5.2)**

5.3 Water bath, capable of being controlled at 20 ± 0,1 °C.

5.4 Vacuum pump, capable of giving a vacuum below 1,33 kPa (10 mmHg).

5.5 Mercury manometer.

6 PROCEDURE

6.1 General

6.1.1 Always weigh the pyknometer with the thermometer and the side tube cover in position.

6.1.2 Always weigh to the nearest 0,000 1 g.

6.1.3 When the pyknometer contains liquid, stabilize its temperature at 20 ± 0,1 °C.

6.2 Determination

6.2.1 *Determination of the mass of the pyknometer*

Wash the pyknometer (5.1), including its accessories, with lukewarm chromic-sulphuric acid, taking all necessary precautions. Thoroughly rinse, first with tap water, then with distilled water and ethanol (4.2) and finally with diethyl ether (4.3).

Thoroughly dry the apparatus and weigh.

Let m_0 be the mass, in grams, of the dry pyknometer.

6.2.2 *Determination of the volume of the pyknometer*

Fill the pyknometer (5.1) with distilled water and connect via the degassing apparatus (5.2) to the vacuum pump (5.4), the mercury manometer (5.5) 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 min. 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 (5.2) and put the thermometer (B) in position.

Stabilize the temperature of the pyknometer in the water bath (5.3), 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, in millimetres, of the pyknometer, is given by the formula

$$V = \frac{(m_1 - m_0)}{0{,}998\ 2}$$

where

m_0 is the mass, in grams, of the dry pyknometer;

m_1 is the mass, in grams, of the pyknometer filled with degassed distilled water;

0,998 2 is the absolute density of water, in grams per millilitre, at 20 °C.

6.2.3 *Determination of the absolute density of xylene*

Fill the pyknometer (5.1) with xylene (4.1) after having determined its mass as in 6.2.1. Carry out the procedure specified in 6.2.2, i.e. degas, stabilize the temperature at 20 ± 0,1 °C in the water bath (5.3), fill, cool and finally weigh. The weighing shall be carried out quickly to avoid loss of xylene by evaporation.

The mass m_x, in grams, of xylene equivalent to the volume V of the pyknometer is given by the formula

$$m_x = m_2 - m_0$$

where

m_0 is the mass, in grams, of the dry pyknometer;

m_2 is the mass, in grams, of the pyknometer full of xylene.

The absolute density ρ_x, in grams per millilitre, of xylene is given by the formula

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

where

m_x is the mass, in grams, of xylene equivalent to the volume V of the pyknometer;

V is the volume, in millilitres, of the pyknometer.

6.2.4 *Test portion*

Transfer to the flask of the pyknometer (5.1), previously weighed in accordance with 6.2.1, about 10 g of the test sample, dried at 300 °C (see 3.3 of ISO 802) and weigh. The mass m_e, in grams, of the test portion is given by the formula

$$m_e = m_3 - m_0$$

where

m_0 is the mass, in grams, of the dry pyknometer;

m_3 is the mass, in grams, of the pyknometer and test portion.

6.2.5 *Determination of the absolute density of the aluminium oxide*

Moisten the ground glass joints of the pyknometer containing the test portion (6.2.4) with a little xylene (4.1) and insert the degassing apparatus (5.2). Connect the latter to the vacuum produced by the pump (5.4) and controlled by means of the mercury manometer (5.5). Close the side tube with its cover (F), gently open tap (D) and apply vacuum for 15 min. 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 any air bubbles adhering to the walls.

NOTE — When the test portion is placed with the xylene in the pyknometer, the expulsion of air trapped in the aluminium oxide covered with xylene is facilitated by the use of high-frequency (for example, 120 kHz) vibrations.

Then fill the pyknometer with the xylene (4.1) up to the ground glass joint and insert the thermometer (B).

Stabilize the temperature of the pyknometer in the water bath (5.3), 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 thoroughly and weigh the pyknometer quickly, because of the tendency for xylene to evaporate.

The total mass m_{e+x}, in grams, of the test portion and additional xylene required to fill the pyknometer is given by the formula

$$m_{e+x} = m_4 - m_0$$

where

m_0 is the mass, in grams, of the dry pyknometer;

m_4 is the mass, in grams, of the pyknometer containing the test portion and filled with xylene.

7 EXPRESSION OF RESULTS

The absolute density ρ, in grams per millilitre, of aluminium oxide is given by the formula

$$\frac{m_e}{(m_e + m_x) \cdot m_{e+x}} \times \rho_x$$

where

m_e is the mass, in grams, of the test portion (6.2.4);

m_x is the mass, in grams, of xylene required to fill the pyknometer volume V;

m_{e+x} is the mass, in grams, of the test portion and of the additional xylene required to fill the pyknometer volume V;

ρ_x is the absolute density, in grams per millilitre, of xylene.

Indicate the absolute density to three places of decimals, two of which should be significant figures.

8 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 in the International Standards to which reference is made, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO ALUMINIUM OXIDE PRIMARILY USED FOR THE PRODUCTION OF ALUMINIUM

ISO 802 – Preparation and storage of test samples.

ISO 803 – Determination of loss of mass at 300 °C (conventional moisture).

ISO 804 – Preparation of solution for analysis – Method by alkaline fusion.

ISO 805 – Determination of iron content – 1,10-Phenanthroline photometric method.

ISO 806 – Determination of loss of mass at 1 000 and 1 200 °C.

ISO 900 – Determination of titanium content – Diantipyrylmethane photometric method.

ISO 901 – Determination of absolute density – Pyknometer method.

ISO 902 – Measurement of the angle of repose.

ISO 903 – Determination of untamped density.

ISO 1232 – Determination of silica content – Reduced molybdosilicate spectrophotometric method.

ISO 1617 – Determination of sodium content – Flame emission spectrophotometric method.

ISO 1618 – Determination of vanadium content – *N*-Benzoyl-*N*-phenylhydroxylamine photometric method.

ISO 2069 – Determination of calcium content – Flame atomic absorption method.

ISO/R 2070 – Determination of calcium content – Spectrophotometric method using naphthalhydroxamic acid.

ISO 2071 – Determination of zinc content – Flame atomic absorption method.

ISO/R 2072 – Determination of zinc content – PAN photometric method.

ISO 2073 – Preparation of solution for analysis – Method by hydrochloric acid attack under pressure.

ISO 2828 – Determination of fluorine content – Alizarin complexone and lanthanum chloride spectrophotometric method.

ISO 2829 – Determination of phosphorus content – Reduced phosphomolybdate spectrophotometric method.

ISO 2865 – Determination of boron content – Curcumin spectrophotometric method.

ISO 2926 – Particle size analysis – Sieving method.

ISO 2927 – Sampling.

ISO 2961 – Determination of an adsorption index.

ISO 3390 – Determination of manganese content – Flame atomic absorption method.