INTERNATIONAL STANDARD

ISO 12989-1

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Carbonaceous materials used in the production of aluminium — Baked anodes and sidewall blocks — Determination of the reactivity to air —

Part 1:
Loss in mass method

Produits carbonés utilisés pour la production de l'aluminium — Anodes et blocs de façade cuits — Détermination de la réactivité à l'air —

Partie 1: Méthode par perte de masse

Reference number
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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 12989 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 12989-1 was prepared by Technical Committee ISO/TC 47, *Chemistry*, Subcommittee SC 7, *Aluminium, cryolite, aluminium fluoride, sodium fluoride, carbonaceous products for the aluminium industry*.

ISO 12989 consists of the following parts, under the general title *Carbonaceous materials used in the production of aluminium — Baked anodes and sidewall blocks — Determination of the reactivity to air*:

— *Part 1: Loss in mass method*

The thermogravimetric method will be the subject of a future part 2 to ISO 12989.

Introduction

The combustion of carbonaceous materials in air leads to undesirable losses that should be minimized in many industrial processes.

The loss of carbonaceous anode material from burning with air is of importance in predicting the behaviour of the anodes during the aluminium reduction process.

Carbonaceous materials used in the production of aluminium — Baked anodes and sidewall blocks — Determination of the reactivity to air —

Part 1: **Loss in mass method**

1 Scope

This part of ISO 12989 specifies a loss-in-mass method for the determination of the reactivity of carbonaceous products to air. The method was developed especially for anodes used in the production of aluminium.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 12989. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 12989 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 8007-2:1999, *Carbonaceous materials used in the production of aluminium — Sampling plans and sampling from individual units — Part 2: Prebaked anodes.*

IEC 60584-2, *Thermocouples — Part 2: Tolerances.*

3 Principle

A cylinder is first heated to 550 °C and then cooled with a gradient of 15 °C/h to (400 ± 1) °C. Frequent agitation is necessary so that the carbon dust caused by the selective burning of the binder matrix can be collected outside the furnace.

Loose particles on the sample are removed using a tumbling-apparatus. The reactivity residue, being the residual body, and the reactivity loss due to burning are reported.

4 Reagents

4.1 Air, bottled or compressed, containing less than 100 µg/g of free water.

5 Apparatus

An appropriate test-apparatus arrangement is shown in Figure 1.

Normal ordinary apparatus and in particular the following:

5.1 Muffle furnace, having the dimensions given in Figure 2 with a vertical, single-zone tube ensuring good, vertical temperature distribution and capable of heating to a maximum temperature of at least 700 °C. The tube shall be made in a refractory steel (austenite) and have an internal diameter of 88,6 mm and a length of 486 mm.

5.2 Sample holder, to support the carbon block in the centre of the furnace with a collection plate incorporated for catching dust falling from the specimens.

5.3 Cam mechanism, motor-driven, which agitates the sample each minute (5 mm fall, 1 r/min).

5.4 Programmable temperature controller, with an integrated digital controller, capable of easily generating and controlling the desired temperature profile (see Figure 3). At least four relays are necessary for the gas and furnace operations.

5.5 Gas-flow meter and pressure control, consisting of a gas-flow meter calibrated for air (p = 0,1 MPa) and a manometer. The gas-flow meter shall have a full-scale flow rate of 250 l/h and shall be accurate to ± 4 %. The gas pressure is adjusted using a valve and the required value, 0,2 MPa, can be controlled by a manometer having a full scale of 0 MPa to 1 MPa.

5.6 Thermocouple, chromel-alumel, K-type, accurate to better than 0,75 % in accordance with IEC 60584-2, 1,6 mm diameter and having a minimum length of 340 mm.

The distance between the upper surface of the anode and the thermocouple shield shall be (10 ± 1,0) mm.

5.7 Tumbling-apparatus, having the following components and assembled as shown in Figure 4.

5.7.1 Electric motor, 90 r/min, 220 V or 110 V, 50/60 Hz.

5.7.2 Two steel cylindrical chambers, with the following dimensions:

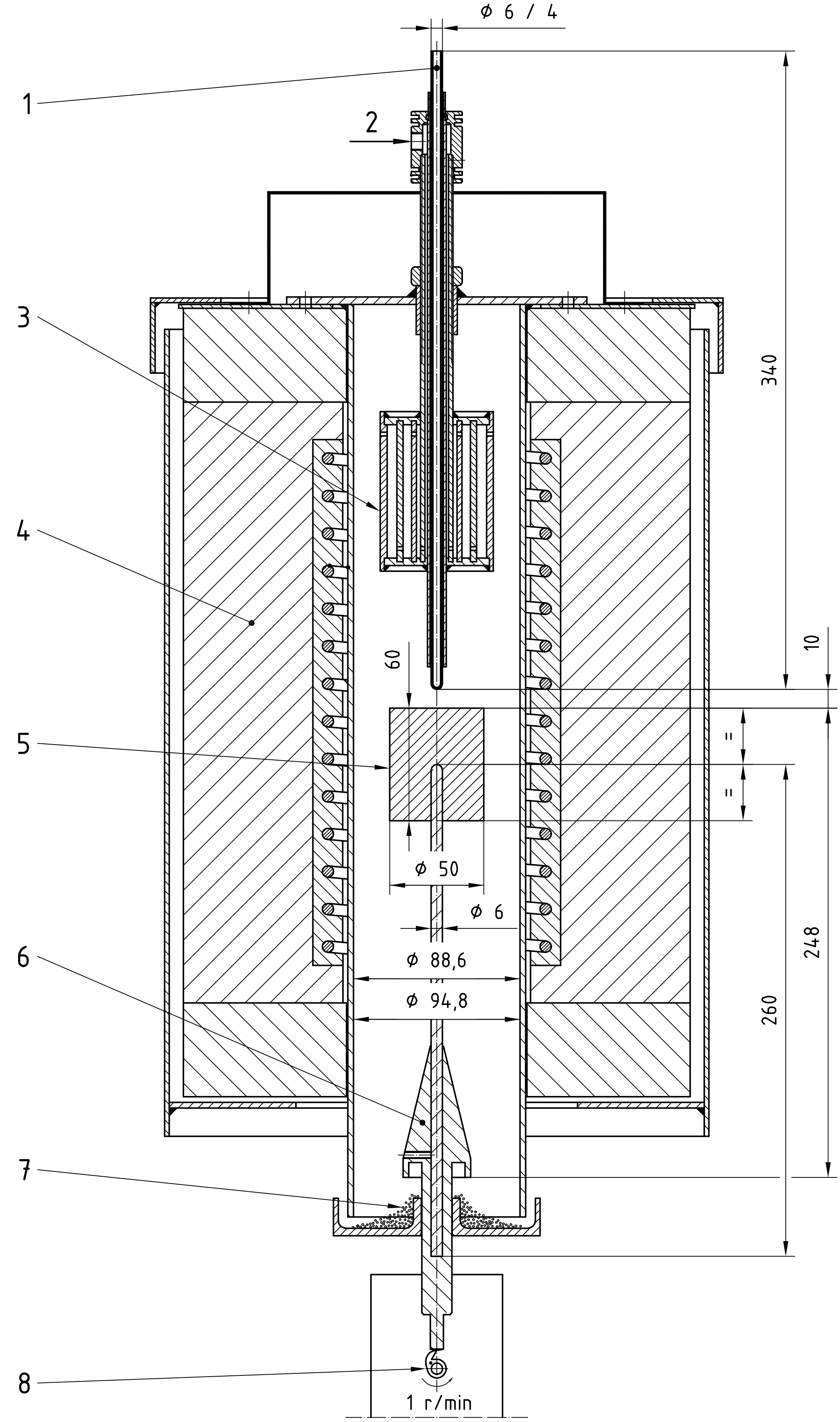
— internal diameter: 69 mm

— internal height: 120 mm

5.7.3 50 steel balls, per chamber (5.8.2), of approximately 6 mm diameter.

5.7.4 Sieve, of 4 mm aperture and pan.

Dimensions in millimetres



Key

1 Thermocouple
2 Air
3 Air preheater
4 Muffle furnace
5 Carbon block test specimen
6 Sample holder
7 Dust
8 Cam mechanism

Figure 1 — Test-apparatus arrangement for the determination of the reactivity of the anodes to air

Dimensions in millimetres

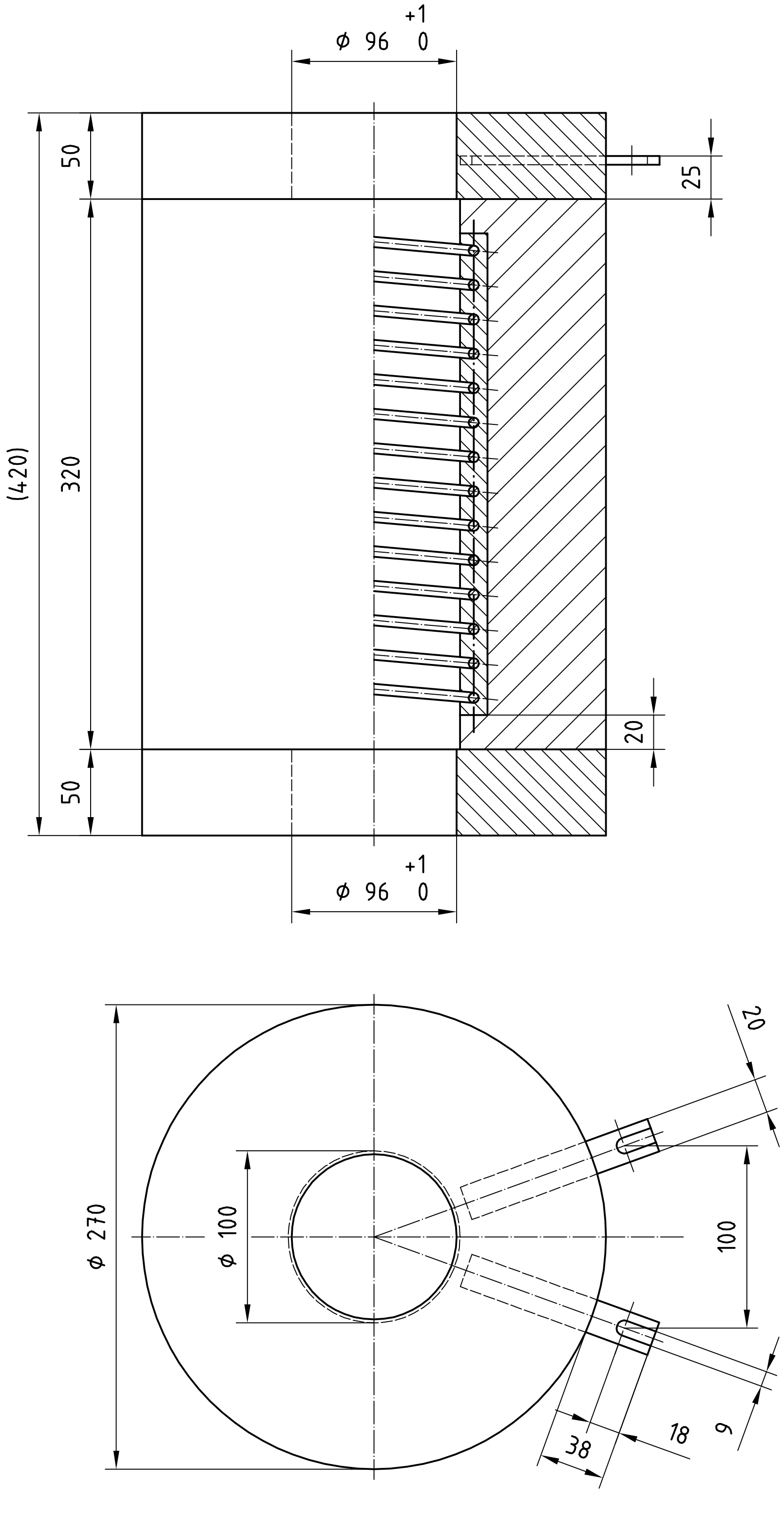
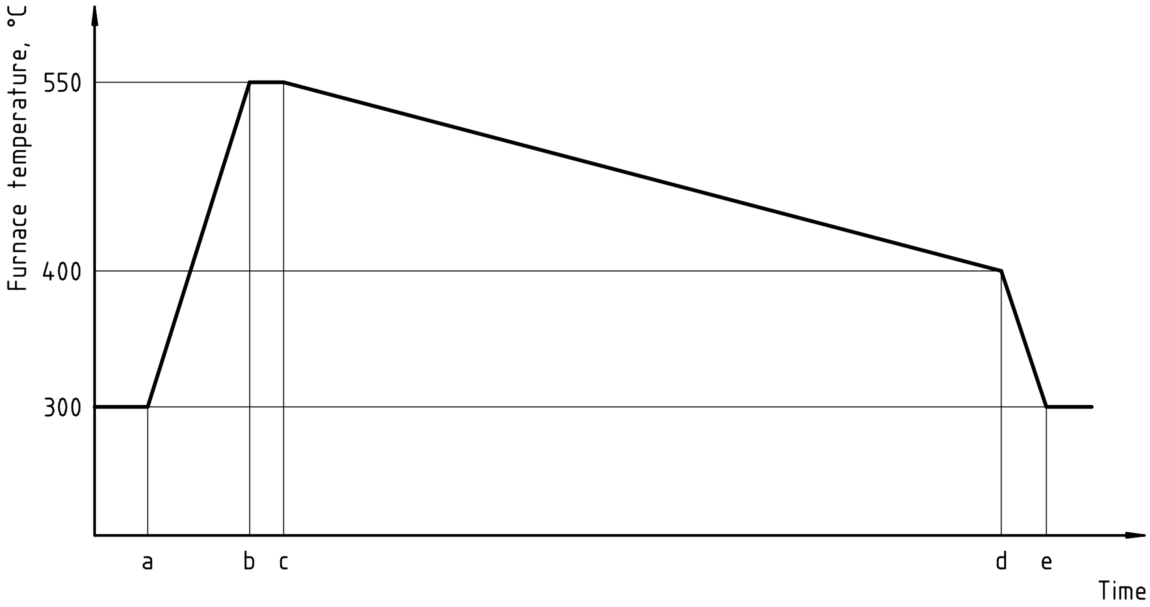


Figure 2 — Dimensions of typical muffle furnace



Key

a Temperature controller on

b Sample in

c Air flowing

d Switch off air

e Remove sample

Figure 3 — Temperature profile and gas operations

6 Sampling

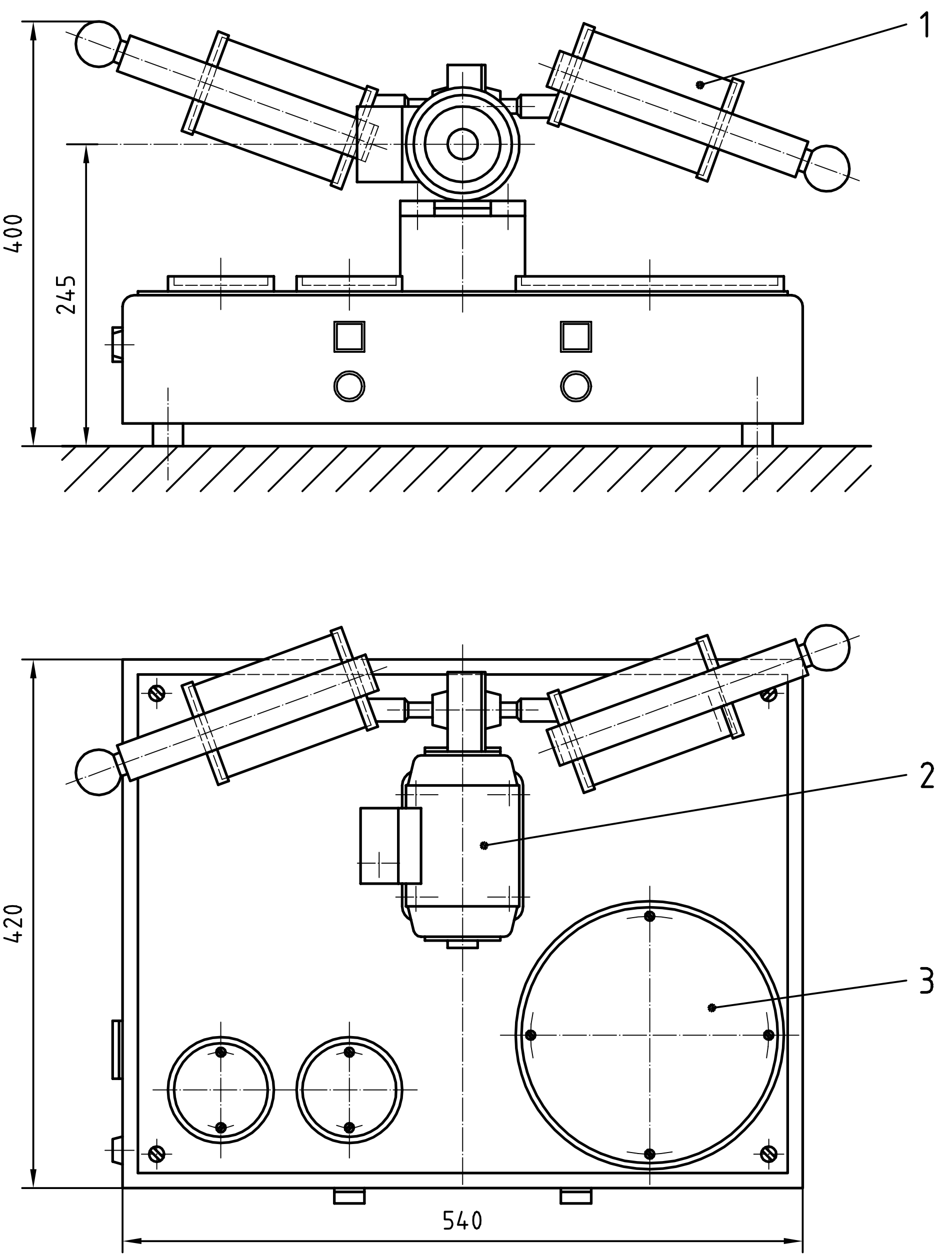
When the test specimen (or core) is to be extracted from a larger body, sampling shall be carried out in accordance with the procedure specified in ISO 8007-2. Alternatively, samples may be prepared in the laboratory, by a bench scale procedure.

7 Preparation of test specimen

Prepare a test specimen with a core diameter of (50 ± 1) mm and a length of (60 ± 1) mm. Dry the specimen at (120 ± 5) °C for 12 h and cool to room temperature.

Drill a hole of 7 mm diameter in the bottom centre of the cylinder to a depth of $(30 \pm 0{,}5)$ mm measured from the top of the cylinder (height of cylinder minus depth of hole). A hard-metal drill bit (diameter 7 mm), with a tip angle of 140° is recommended.

Dimensions in millimetres



Key

1 Chamber

2 Motor

3 Sieve

Figure 4 — Tumbling-apparatus for determining the amount of dust produced

8 Procedure

8.1 Program the temperature controller (5.4) according to the profile illustrated in Figure 3 and as specified in 8.3. The temperature shall not exceed (550 ± 1) °C and the cooling rate shall be 15 °C/h.

8.2 Weigh the initial sample (m_0) to the nearest 0,1 g and record the mass.

8.3 Start the temperature controller. Raise the temperature of the muffle furnace (5.1) to 550°C. Place the test specimen (core) in the furnace. After 30 min, start the air (4.1) flowing through the pre-heater (see Figure 1). Set the gas pressure of the air to 0,2 MPa and adjust the flow meter valve so as to have a flow rate of 200 l/h.

After 10 h, when the temperature of (400 ± 1) °C is reached, switch off the air. Remove the cylinder when the furnace has reached 300 °C and collect the dust from the aluminium plate. Allow the test specimen to cool to room temperature.

8.4 Weigh each test specimen (core) with its associated dust (m_1) to the nearest 0,1 g. Place the test specimen (core) into the tumbling-apparatus chamber (5.7) and allow it to tumble for 20 min. Empty the chamber (5.7.2) into the 4 mm sieve (5.7.4) and remove the balls (5.7.3). Weigh the residual body of the test specimen (core) (m_2) to the nearest 0,1 g.

9 Calculation

The reactivity-to-air parameters, w_{RA} (residue, dust and loss), expressed as a percentage by mass, are given by the formulae :

Residue, w_{RAR}:

$$w_{RAR} = \frac{m_2}{m_0} \times 100$$

Dust, w_{RAD}:

$$w_{RAD} = \frac{m_1 - m_2}{m_0} \times 100$$

Loss, w_{RAL}:

$$w_{RAL} = \frac{m_0 - m_1}{m_0} \times 100$$

where

m_0 is the initial mass, expressed in grams, of the test specimen (core);

m_1 is the mass, expressed in grams, of the residual test specimen (core) and dust;

m_2 is the mass, expressed in grams, of the residual test specimen (core) after tumbling.

Report the result to 0,1 g.

10 Precision

10.1 Repeatability

The repeatability of the test depends strongly on the homogeneity of the tested cores and is therefore difficult to assess.

10.2 Reproducibility

An inter-laboratory trial has allowed an estimate of the reproducibility, R, of the test to be made as a function of the reactivity-to-air (see Figure 5). These reproducibility figures can be used when at least 30 cores are used for determination to characterize a given electrode population.

11 Test report

The test report shall include the following information:

a) an identification of the sample;

b) the method used by reference to this part of ISO 12989, i.e. 12989-1;

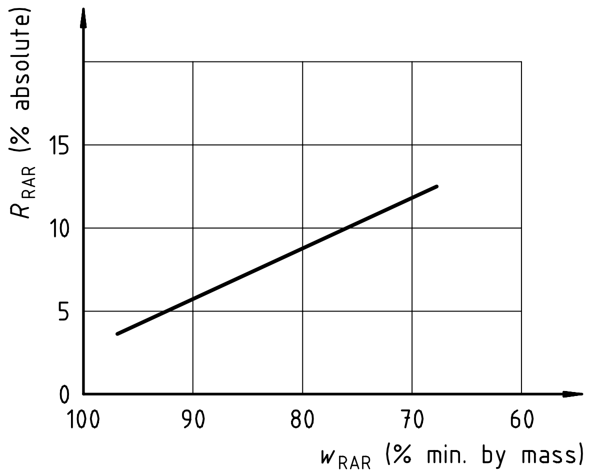
c) the method of sampling used;

d) the date of the test;

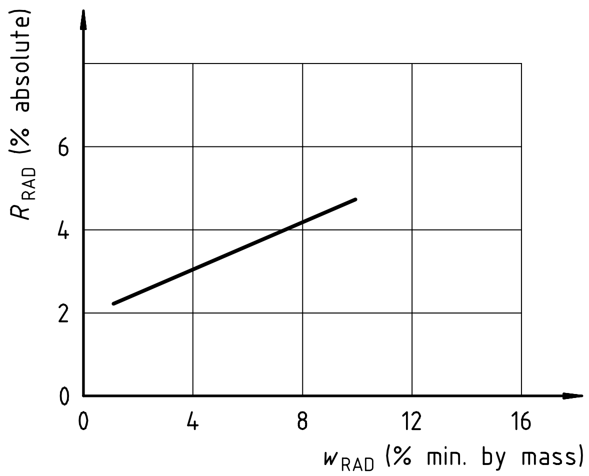
e) the results and the method of expression used;

f) any unusual features noted during the determination;

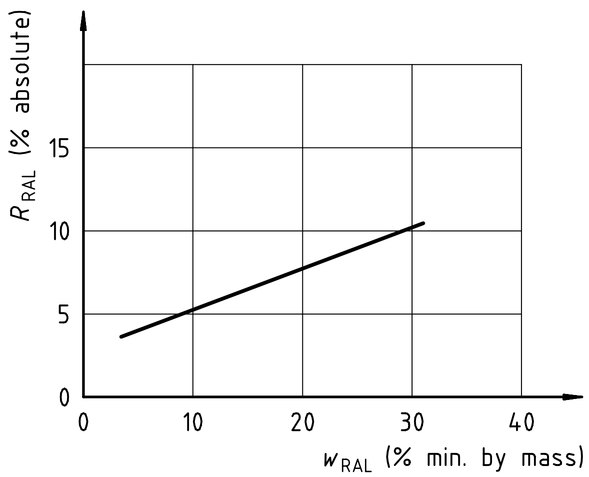
g) any operation not included in this part of ISO 12989 or in the International Standards to which reference is made, or regarded as optional.



a) Estimated reproducibility R_{RAR} as a function of w_{RAR} (reactivity-to-air parameter for residue)



b) Estimated reproducibility R_{RAD} as a function of w_{RAD} (reactivity-to-air parameter for dust)



c) Estimated reproducibility R_{RAL} as a function of w_{RAL} (reactivity-to-air parameter for loss)

Figure 5 — Estimated reproducibility, R_{RA}, as a function of the reactivity-to-air parameter, w_{RA}

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