International Standard 8006

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Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Determination of ash

Produits carbonés utilisés pour la production de l'aluminium — Brai pour électrodes — Détermination du taux de cendres

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Foreword

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Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Determination of ash

1 Scope and field of application

This International Standard specifies a method for the determination of ash in the pitch for electrodes used in the production of aluminium.

The method is applicable to products (tar pitch, petroleum pitch, etc.) producing a quantity of ash greater than or equal to 0,05 % (*m/m*).

If it is required to express the result on the dry product, carry out the water determination by azeotropic distillation as specified in ISO 5939. Refer the result of the determination of ash obtained on the product as such, to the dry product.

NOTE — This ash may be utilised for subsequent elemental analysis.

2 References

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

ISO 5939, *Carbonaceous materials for the production of aluminium. Determination of water content — Azeotropic distillation (Dean and Stark).*

ISO 6257, *Carbonaceous materials for the production of aluminium — Pitch for electrodes — Sampling.*

3 Principle

Heating of a test portion in a tared platinum crucible, in a furnace at 700 °C, under oxidizing and specified conditions, to constant mass.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Platinum crucible, low form, of capacity approximately 50 ml.

4.2 Electric furnace, capable of being controlled at 700 ± 10 °C and provided with air circulation.

4.3 Electric oven, capable of being controlled at 150 ± 5 °C.

5 Sampling and samples

Sample in accordance with ISO 6257.

6 Procedure

6.1 Test portion

Heat the crucible (4.1) for 1 h in the furnace (4.2), controlled at 700 ± 10 °C. Allow the crucible to cool, first in air to about 100 and 150 °C, and then in a desiccator charged with diphosphorus pentaoxide, to room temperature. Weigh the crucible to the nearest 0,000 2 g (m_1).

Weigh into the crucible, to the nearest 0,000 2 g, approximately 5 g of the test sample (see clause 5) (m_0).

6.2 Determination

Place the crucible containing the test portion (6.1) into the electric furnace (4.2) set at a temperature close to but not exceeding 300 ± 10 °C. Increase heating following the heating rule indicated below.

In periods of not less than 30 min each, but which may, without prejudice, be increased to approximately 60 min, raise the temperature at a uniform rate first to 350 ± 10 °C, then to 400 ± 10 °C and finally to 450 ± 10 °C, inspecting regularly to ensure that no mechanical loss of the test portion occurs due to over-vigorous volatilization. If losses are observed, repeat the determination on another test portion and following the procedure specified below.

If the water content of the sample is high, place the crucible containing the test portion (6.1) in the electric oven (4.3), controlled at 150 ± 5 °C, and allow to stand at this temperature for about 2 h. Remove the crucible and its content from the oven and place it directly in the electric furnace (4.2), controlled at 300 ± 10 °C. Continue heating following the heating rule already specified.

If even in such conditions some losses of the test portion are observed (over-vigorous volatilization), reduce the rate of heating, in the range 300 to 450 °C by suitably increasing the relevant periods of heating.

Raise the temperature of the furnace to 700 ± 10 °C and heat the crucible and contents at this temperature until the ashing process appears to be complete (conveniently overnight).

Remove the crucible and contents, allow to cool, first in air to about 100 to 150 °C and then in a desiccator, charged with diphosphorus pentaoxide, to room temperature. Weigh the crucible and contents to the nearest 0,000 2 g.

Transfer the crucible and contents to the furnace maintained at 700 ± 10 °C and heat for 30 min. Cool and weigh as specified in the previous paragraph. Repeat the heating, cooling and weighing operations until the mass of the crucible and contents does not differ by more than 0,000 2 g on two consecutive weighings.

NOTES

1 The furnace (4.2) should be located in a well-ventilated fume cupboard.

2 The number of tests which may be carried out simultaneously is dependent on the performance characteristics of the furnace used. Particular attention should be paid to the temperature profile within the furnace in positioning the crucibles for a multiple determination.

7 Expression of results

7.1 Method of calculation

The ash, determined on the normal product, x, expressed as a percentage by mass, is given by the equation

$$x = (m_2 - m_1) \times \frac{100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (6.1);

m_1 is the mass, in grams, of the empty crucible;

m_2 is the mass, in grams, of the crucible containing the ash.

Report the result to the nearest 0,01 % (m/m).

7.2 Precision

The repeatability and reproducibility were calculated according to ISO 5725.

From the data obtained a connection is observed between both repeatability and reproducibility and the average value (see the figure).

The graph shows, as abscissae, the average values as percentages, obtained for each level, and as ordinates the corresponding values, as percentages, of repeatability and reproducibility.

Calculate these values for each level using the two linear equations:

$$r = 0{,}012\,6 + 0{,}081\,7\,\bar{x}$$

$$R = 0{,}001\,26 + 0{,}427\,\bar{x}$$

8 Test report

The test report shall include the following particulars:

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

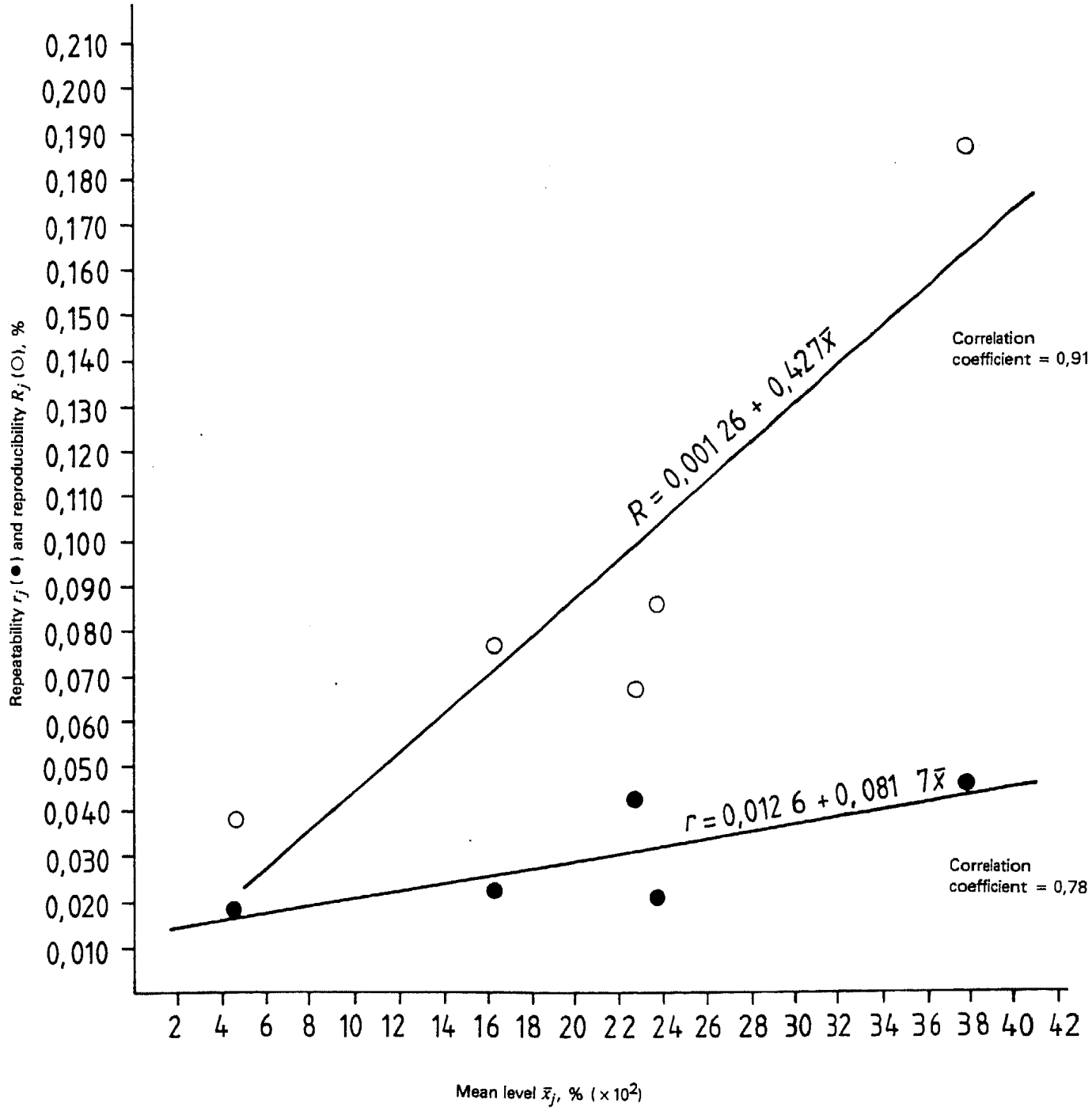

Figure — Graph of repeatability r_j and of reproducibility R_j as a function of mean level $\bar{x}_j$