International Standard 6606

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Aluminium ores — Determination of loss of mass at 1 075 °C — Gravimetric method

Minerais alumineux — Détermination de la perte de masse à 1 075 °C — Méthode gravimétrique

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Aluminium ores — Determination of loss of mass at 1 075 °C — Gravimetric method

0 Introduction

Aluminium ores when heated undergo a loss of mass. At temperatures up to 110 °C the loss is due to hygroscopic moisture. At higher temperatures the loss of mass is due largely to the dissociation of hydroxides and oxyhydroxides of aluminium and iron and also, to a lesser degree, to the dissociation of minor constituents.

The total loss of mass is a function of the temperature and time of heating. There are no conditions at which the loss represents exclusively the water (hygroscopic and combined) in the sample.

The temperature chosen for the test, 1 075 °C, is a compromise between such factors as furnace suitability and the absorption of water by the sample during cooling.

The value obtained for loss on ignition is calculated on the basis of a dried sample.

The test portion is heated initially at 375 ± 25 °C and finally at 1 075 ± 25 °C and the loss of mass determined. The crucible is covered with a loose-fitting lid after the test portion is placed in the crucible and this is left in position throughout all the subsequent operations. The presence of the lid contributes to more reproducible results by preventing the random effects of small pieces of furnace lining falling into the crucible during heating and does not interfere with the maintenance of oxidizing conditions in the crucible.

1 Scope and field of application

This International Standard specifies a gravimetric method for the determination of the loss of mass of analytical samples of aluminium ores when heated to constant mass at 1 075 °C. The method is applicable to all aluminium ores having loss of mass values in the range of 10 to 30 %.

2 Reference

ISO 8557, *Aluminium ores — Determination of hygroscopic moisture of analytical samples — Gravimetric method.*

3 Principle

Heating of a test portion in a tared crucible in a furnace at 375 ± 25 °C for 10 min. Transfer of the covered crucible to a second furnace at 1 075 ± 25 °C and heating to constant mass. Correction of the resultant loss in mass for the original hygroscopic moisture.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Dishes, of inert material of sufficient size to take the required quantity of sample at a layer density of 5 mg/mm^2.

4.2 Platinum crucible, approximately 30 mm top diameter, 20 mm bottom diameter and 35 mm deep, with matching platinum lid.

4.3 Electric furnaces, capable of being controlled at 375 ± 25 °C and at 1 075 ± 25 °C, and having provision for a flow of air through the heated cavity.

4.4 Balance, capable of being read to 0,000 1 g.

4.5 Desiccator, containing either fresh magnesium perchlorate or activated alumina as desiccant.

NOTES

1 Activated alumina should be activated by heating at 300 ± 10 °C overnight.

2 When discarding magnesium perchlorate, flush down the sink using copious quantities of water.

5 Sampling and samples

5.1 Samples

Laboratory samples shall be taken and crushed to pass a 150 µm test sieve, in accordance with the methods specified in the relevant standards.[1].

1) Where no International Standards exist, the relevant standards shall be the national standards. Two International Standards on this subject are currently in preparation : ISO 6137, *Aluminium ores — Method of sampling,* and ISO 6140, *Aluminium ores — Preparation of samples.*

5.2 Preparation of the test sample

Take approximately 10 g of the laboratory sample and place in a flat dish (4.1). Spread the sample evenly to give a layer density of approximately 5 mg/mm^2 and allow to equilibrate with the laboratory atmosphere for a minimum of 2 h.

6 Procedure

6.1 Number of determinations

Carry out the determination in duplicate on each ore sample.

NOTE — The principle of carrying out a blank determination and a check test does not apply to this method.

6.2 Preparation of crucible and test portion

Heat the platinum crucible (4.2) and lid in the furnace (4.3) controlled at 1 075 ± 25 °C for 15 min. Remove the covered crucible from the furnace and place in the desiccator (4.5) to cool. Weigh the crucible and lid, to the nearest 0,000 2 g, as rapidly as possible after they have cooled to ambient temperature and within 1 h of placing them in the desiccator.

Add approximately 1 g (± 0,01 g) of the test sample to the platinum crucible, distribute it evenly over the bottom of the crucible, replace the lid and weigh the crucible, lid and contents to the nearest 0,000 2 g. Record the mass of the test portion (m_1).

At the same time, weigh test portions for the determination of hygroscopic moisture by the procedure specified in ISO 8557.

6.3 Determination of loss on ignition

Place the covered crucible and contents, with the lid slightly displaced, in the furnace (4.3) controlled at 375 ± 25 °C and heat for 10 ± 1 min.

Transfer the partially covered crucible and contents to the furnace controlled at 1 075 ± 25 °C and heat for 60 ± 2 min.

Remove the covered crucible and contents from the furnace, ensuring that the lid is now fully in position, and place in the desiccator to cool. Weigh the covered crucible and contents, to the nearest 0,000 2 g, as rapidly as possible after they have cooled to ambient temperature and within 1 h after they have been placed in the desiccator.

NOTE — Prior to each weighing, inspect the outside of the crucible lid and brush if necessary.

Return the covered crucible and contents to the furnace controlled at 1 075 ± 25 °C and heat for a further 30 ± 2 min. Allow to cool in the desiccator and re-weigh as soon as possible after the crucible and contents have cooled to ambient temperature and within 1 h of placing them in the desiccator.

If the difference between the weighings after the first and second heatings at 1 075 °C exceeds 0,000 5 g, repeat the heating, cooling and weighing steps, until this requirement is met.

Use the minimum mass of the crucible, lid and contents to calculate the minimum mass of the heated sample (m_2).

7 Expression of results

7.1 Calculation of the loss on ignition

Calculate the loss on ignition (LOI), expressed as a percentage by mass, using the formula

$$\text{LOI} = \left[\frac{100\,(m_1 - m_2)}{m_1} - H \right] \times \frac{100}{100 - H}$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the test portion after heating;

H is the hygroscopic moisture content, as a percentage by mass, of the equilibrated sample.

7.2 General treatment of results

7.2.1 Precision

A planned trial of the method was carried out by nine countries with a total of sixteen laboratories participating. Two determinations were carried out by each analyst on each of four samples. From the results obtained, the repeatability, reproducibility and reproducibility index have been calculated and are presented in the following table.

Table — Precision data for loss of mass determinations

Sample	Mean loss in mass at 1 075 °C % (m/m)	Components of standard deviation		Reproducibility index
		s_w	s_b	$2s$
MT/12/12	14,47	0,064	0,215	0,45
MT/12/4	25,24	0,050	0,126	0,27
MT/12/1	26,43	0,090	0,101	0,27
MT/12/9	27,53	0,078	0,195	0,42

where

s_w is the within-laboratory standard deviation;

s_b is the between-laboratories standard deviation.

7.2.2 Acceptance of analytical values

The analytical value for the test sample shall be accepted when the difference between the two values for the test sample does not exceed 2.77 s_w as calculated from the appropriate value of s_w given in the table.

When the range (absolute difference) of the two values for the test sample is greater than 2,77 s_w, additional determinations shall be carried out on the test sample.

7.2.3 Calculation of final result

The final result is the arithmetical mean of the values for the test portions calculated to the fourth decimal place and rounded off to the second decimal place as follows :

1) When the figure in the third decimal place is less than 5, it is discarded and the figure in the second decimal place is kept unchanged.

2) When the figure in the third decimal place is 5 and there are figures other than 0 in the fourth decimal places, or when the figure in the third decimal place is greater than 5, the figure in the second decimal place is increased by one.

3) When the figure in the third decimal place is 5 and there are no figures other than 0 in the fourth decimal places, the 5 is discarded and the figure in the second decimal place is kept unchanged if it is 0, 2, 4, 6 or 8 and is increased by one if it is 1, 3, 5, 7 or 9.

8 Test report

The test report shall include the following information :

a) details necessary for the identification of the sample;

b) reference to this International Standard;

c) the result of the analysis;

d) reference number of the result;

e) any characteristics noticed during the determination and any operations not specified in this International Standard which may have had an influence on the result.

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