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Iron ores — Determination of loss on ignition — Non-oxidised ores

Minerais de fer — Détermination de la perte au feu — Minerais non oxydés

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Foreword

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The committee responsible for this document is ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

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Introduction

The measurement of loss on ignition (LOI) is a technique widely used in the iron ore industry.

Ignition loss is the sum of contributions from the mass loss of volatile compounds such as water vapour, carbon dioxide and sulphides (due to the decomposition of goethite and carbonaceous materials), and the mass gain due to oxidation [Fe(II) to Fe_2O_3]. Its use is complementary to the determination of elemental or oxide concentrations. It serves to allow for an addition of the oxides, generated at the ignition temperature, and the LOI, to arrive at total (oxide + LOI). The determination of LOI is essential in sinter plant and blast furnace balance calculations, as it is used to calculate calcinated elemental concentrations.

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Iron ores — Determination of loss on ignition — Non-oxidised ores

WARNING — This Technical Report may involve hazardous materials, operations and equipment. This Technical Report does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this Technical Report to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This Technical Report describes a gravimetric method for the determination of the loss in mass of non-oxidized iron ores, when ignited at 1 000 °C.

This method is applicable to a concentration range of a mass fraction of −3,0 % to 7,0 % loss on ignition in natural iron ores, iron ore concentrates and agglomerates, and sinters.

The method is not applicable to the following:

a) processed ores containing metallic iron (direct reduced iron);

b) natural or processed ores in which the sulfur content is higher than a mass fraction of 0,2 %;

c) internationally traded ores with combined water greater than 2,5 %.

NOTE 1 Loss on ignition can be used as an estimate of combined water.

NOTE 2 This method is intended for in-house use and is not intended for referee purposes.

2 Principle

A test portion is heated in a muffle furnace at 1 000 °C for 60 min and cooled in a desiccator. The decreased mass of the test portion is then measured.

3 Reagents

During the analysis, use only reagents of recognized analytical grade.

3.1 Silica gel, dried at 105 °C for 4 h.

4 Apparatus

Ordinary laboratory apparatus and the following:

4.1 Silica, **porcelain** or **platinum crucibles**, 15 ml to 25 ml capacity, with lids.

The crucibles should be pre-conditioned in the muffle furnace at 1 000 °C for 60 min. Crucibles and lids should be stored in the desiccator (4.4) prior to use.

If platinum crucibles used for LOI analysis are to be used for flux fusions, platinum ware should be thoroughly cleaned to prevent cross-contamination.

4.2 Balance, capable of reading to the nearest 0,1 mg at the mass load of the crucible.

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4.3 Muffle furnace, capable of maintaining a temperature of 1 000 °C ± 25 °C, with provision for air circulation adequate to prevent water vapour retention.

4.4 Desiccator, of borosilicate glass, 150 mm to 250 mm internal diameter with a vacuum stopcock that will allow the evacuation of air.

The desiccator plate should be metal or ceramic or similar that will not break when in contact with a crucible at 1 000 °C. The rim of the desiccator should be lightly greased with silicon grease or petroleum jelly. 150 g to 200 g of silica gel (3.1) should be replaced daily.

If platinum crucibles are used, metal desiccator plates should not be used.

5 Sampling and samples

5.1 Laboratory sample

For analysis, use a laboratory sample of −100 µm particle size which has been taken and prepared in accordance with ISO 3082.

5.2 Preparation of test samples

Analyses may be performed using either predried test samples (prepared in accordance with ISO 7764).

6 Procedure

6.1 Number of determinations

Carry out the analysis at least in duplicate, using the procedure given in Annex A, independently, on each test sample.

NOTE The expression "independently" means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure are to be carried out either by the same operator at a different time, or by a different operator, including appropriate recalibration in either case.

6.2 Test portion

Taking several increments, weigh to the nearest 0,000 1 g, approximately 2,0 g ± 0,2 g of the test sample (5.2).

6.3 Determination

6.3.1 Pre-treatment of crucible

Using crucible tongs, place the crucible (4.1) into the muffle furnace (4.3) at 1 000 °C for 60 min ± 10 min, ensuring that there is no loose material on the floor of the muffle furnace that could possibly adhere to the crucible.

Remove the crucible from the furnace to a desiccator (4.4), place the lid on the crucible immediately, and cool under vacuum for 30 min. Keep the lid on the crucible throughout the cooling period.

Release the vacuum slowly, then weigh the cooled crucible and lid to the nearest 0,000 1 g (m_1).

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6.3.2 Determination of LOI

Transfer the test portion (6.2) to the crucible and re-weigh the crucible and lid to the nearest 0,000 1 g (m_2).

Place the crucible containing the test portion (m_2) into the muffle furnace (4.3) at 1 000 °C for 60 min ± 10 min.

Remove the crucible from the furnace to a vacuum desiccator (4.4), place the lid on the crucible immediately, and cool under vacuum for 30 min. Keep the lid on the crucible throughout the cooling period.

Release the vacuum slowly, then weigh the cooled crucible and lid to the nearest 0,000 1 g (m_3).

7 Expression of results

7.1 Calculation of loss on ignition

The loss on ignition *LOI*, expressed as a percentage by mass, is calculated Formula (1):

$$LOI\ \%\ (\mathrm{m/m}) = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \qquad (1)$$

where

m_1 is the mass of the crucible and lid, in grams;

m_2 is the mass of the crucible, lid and dry test portion, in grams;

m_3 is the mass of the crucible, lid and test portion after ignition, in grams.

7.2 General treatment of results

7.2.1 Repeatability and permissible tolerances

The precision of this analytical method is expressed by the following formulae (see also Annex A):

$$S_d = 0{,}040 \qquad (2)$$

$$S_L = 0{,}029\,1\,X + 0{,}051\,7 \qquad (3)$$

$$R_d = 0{,}114 \qquad (4)$$

$$P = 0{,}042\,7\,X + 0{,}026\,2 \qquad (5)$$

where

X is the loss on ignition, expressed as a percentage by mass, of the sample;

S_d is the independent duplicate standard deviation;

S_L is the between-laboratories standard deviation;

R_d is the independent duplicate limit;

P is the permissible tolerance.

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7.2.2 Determination of analytical result

Having computed the independent duplicate results according to Formula (2), compare them with the independent duplicate limit (R_d), using the procedure given in Annex A.

7.2.3 Between-laboratories precision

Between-laboratories precision is used to determine the agreement between the final results reported by two laboratories. The assumption is that both laboratories followed the same procedure described in 7.2.2.

Compute the following quantity:

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \tag{6}$$

where

μ_1 is the final result reported by laboratory 1;

μ_2 is the final result reported by laboratory 2;

μ_{12} is the mean of final results.

If $|\mu_1 - \mu_1| \leq P$, the final results are in agreement.

7.2.4 Check for trueness

The trueness of the analytical method should be checked by applying it to a certified reference material (CRM) or a reference material (RM). The procedure is the same as that described above. After confirmation of the precision, the final laboratory result is compared with the reference or certified value A_c. There are two possibilities:

a) $|\mu_c - A_c| \leq C$, in which case the difference between the reported result and the reference/certified value is statistically insignificant.

b) $|\mu_c - A_c| > C$, in which case the difference between the reported result and the reference/certified value is statistically significant.

where

μ_c is the final result for the certified reference material;

A_c is the reference/certified value for the CRM/RM;

C is a value dependent on the type of CRM/RM used.

Certified reference materials used for this purpose should be prepared and certified in accordance with ISO Guide 35.

For a CRM certified by an inter-laboratory test programme:

$$\sigma_L = c\,\mu_c + d$$

$$\sigma_d = a\,\mu_c + b$$

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$$C = 2\sqrt{\sigma_L^2 + \frac{\sigma_d^2}{n} + V\left(A_C\right)}$$

where $V(A_C)$ is the variance of the certified value A_C.

For a CRM certified by only one laboratory:

$$C = 2\sqrt{2\,\sigma_L^2 + \frac{\sigma_d^2}{n}}$$

This type of CRM should be avoided unless it is known to have an unbiased certified value.

7.2.5 Calculation of final result

The final result is the arithmetic mean of the acceptable analytical values for the test sample, or as otherwise determined by the operations provided in Annex A, calculated to four decimal places and rounded off to the second decimal place as follows:

a) where 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;

b) where the figure in the third decimal place is 5 and there is a figure other than 0 in the fourth decimal place, or when the figure in the third decimal place is greater than 5, the figure in the second decimal place is increased by one;

c) where the figure in the third decimal place is 5 and the figure 0 is in the fourth decimal place, 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 should include the following information:

a) the name and address of the testing laboratory;

b) the date of issue of the test report;

c) a reference to this Technical Report, i.e. ISO/TR 18230:2015;

d) the details necessary for the identification of the sample;

e) the analysis result;

f) the reference number of the result;

g) any characteristics noticed during the determination, and any operations not provided in this Technical Report which may have an influence on the result, for either the test sample or the certified reference material(s).

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