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Iron ores — Determination of the moisture content of a lot

Minerais de fer — Détermination de l'humidité d'un lot

ICS: 73.060.10

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

This document was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 1, *Sampling*.

This fifth edition cancels and replaces the fourth edition (ISO 3087:2011), which has been technically revised.

The main technical changes to this edition are as follows:

— Confirmation that the existing two 105 °C moisture determination methods are to serve as reference methods;

— Allowing for alternative moisture determination methods if they can be shown to result in equivalent moisture contents as the reference methods;

— Changing weighing device readability requirement from 0,05 % to 0,01 % equivalent of test portion mass;

— Revising clause 9: Calculation and expression of results;

— Updating Annex D with new example reports.

Introduction

Currently, large tonnages of iron ore are traded internationally and a small error in the measured moisture content [mass fraction (%)] of a lot has a considerable effect on the commercial transaction. The correct determination of moisture content of a lot is, therefore, a matter of importance for both the purchaser and the vendor.

This International Standard does not address the determination of the hygroscopic moisture content of a test sample for chemical analysis. If the hygroscopic moisture content is required to be determined, reference should be made to ISO 2596:2006, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods*.

Iron ores — Determination of the moisture content of a lot

1 Scope

This International Standard specifies a method for the determination of the moisture content of a lot of iron ore. This method is applicable to all iron ores, whether natural or processed.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 apply.

4 Principle

Dry the test portion in air at 105 °C and measure the loss in mass. Express the moisture content as the mass loss relative to the original mass of the sample as a mass fraction (%).

5 Apparatus

5.1 Drying pan, with a smooth surface, free from contamination and capable of accommodating the specified quantity of a test portion in a layer of nominal thickness not greater than 31,5 mm.

5.2 Drying oven, equipped with a temperature indicator and control apparatus capable of regulating the temperature at any point in the oven at 105 °C ± 5 °C and so designed as to maintain this temperature with a current of air to ensure efficient drying but without any loss of sample, and fitted with a fan that allows for both the circulation and change of air.

5.3 Weighing device, with readability equivalent to at least 0,01 % of the Table 1 minimum test portion mass.

The capacity of the weighing device shall be enough for the combined mass of the drying pan and the initial mass of the test portion.

6 Samples

Test samples which have been taken and prepared in accordance with ISO 3082 shall be used. The mass of a test portion, in relation to its nominal top size, is specified in Table 1, in accordance with ISO 3082-2017, clause 10.6.

The nominal top size of the moisture test sample shall be 31,5 mm or less. Samples with a nominal top size greater than 31,5 mm shall be crushed prior to extraction of test samples for moisture determination. When it is difficult to conduct crushing and dividing owing to a sample being adhesive or excessively wet, the sample may be partially dried in accordance with the procedure in Annex A.

Table 1 — Minimum mass of test portion

Nominal top size of test portion		Minimum mass of test portion
mm		kg
Over	up to and including	
22,4	31,5	10
10,0	22,4	5
—	10,0	1

7 Procedure

7.1 Number of moisture measurements

Carry out one moisture measurement per test portion on the number of test portions specified in Table 2, according to the conditions of preparation of the test sample.

Table 2 — Number of test portions

Preparation of test sample	Number of partial samples per lot	Number of test portions to be tested
From gross sample	—	4 per gross sample
From partial sample	2	4 per partial sample
	3 to 7	2 minimum per partial sample
	≥ 8	1 minimum per partial sample
From increment	—	1 minimum per increment

In order to minimize losses of moisture to the atmosphere, it is necessary to perform all the initial weighings of the test portions as quickly as possible after obtaining those test portions.

7.2 Reference methods

7.2.1 General

The moisture determination shall be conducted in accordance with 7.2.2 or, optionally, for ores with 8 % or more of combined water in accordance with 7.2.3.

7.2.2 Normal reference method

a) Spread the test portion in a layer of nominal thickness not greater than 31,5 mm in the tared drying pan (5.1) and determine the total mass immediately. Record the total mass, the mass of the drying pan, the initial mass of the test portion (m_1) and the numerical value of 0,05 % of m_1.

b) Place the drying pan with the test portion in the drying oven (5.2) set at 105 °C, and maintain this temperature for not less than 4 h. Remove the drying pan with the test portion from the drying oven and weigh it immediately while still hot in order to minimize any reabsorption of moisture. Otherwise, weigh the test portion after cooling in air in a container having a close-fitting airtight lid. In each case, report the method of weighing.

c) Once more, place the drying pan with the test portion in the drying oven, heat for a further 1 h, and then repeat the weighing.

d) Repeat the procedure described in item c) until the difference in mass between subsequent measurements becomes 0,05 % or less of the initial mass of the test portion.

NOTE 1 The weighing device should be protected from the influence of heat.

NOTE 2 To reduce drying time, it is recommended that layer-thickness of the sample be kept as low as possible. It should be specified for particular ores by check experiments carried out beforehand.

NOTE 3 For convenience, the test portion of mass 10 kg for ore of particle size less than 31,5 mm may be divided into two portions, each of which is subjected to moisture measurement. In calculating the results, the mean of the two values of initial mass and the mean of the two values of the drying loss in mass should be used.

7.2.3 Optional reference method for ores of high combined water content

For ores containing 8 % or more combined water, the following procedure may be applied.

a) Spread the test portion in a layer of nominal thickness not greater than 31,5 mm in the tared drying pan (5.1) and determine the total mass immediately. Record the total mass, the mass of the drying pan and the initial mass of the test portion (m_1).

b) Place the drying pan with the test portion in the drying oven (5.2) set at 105 °C and maintain this temperature for not less than 24 h. Remove the drying pan with the test portion from the drying oven and weigh it immediately while still hot in order to minimize any reabsorption of moisture. Otherwise, weigh the test portion after cooling in air in a container having a close-fitting airtight lid. Record the total mass after drying. In each case, report the method of weighing.

NOTE The notes in 7.2.2 apply to this subclause

7.3 Alternative methods

7.3.1 General

Alternative methods refer to the potential use of different technology, such as ovens that do not rely on convection drying alone or to procedures that potentially differ in terms of oven temperature (up to 140 °C) and time from what is described in clause 7.2, but which do not deviate from the conditions stipulated in Clause 6 and Clause 7.1 (minimum mass of test portion and number of portions to be tested).

Such alternative methods or modifications for determining the moisture content are allowed where check experiments carried out beforehand, demonstrate satisfactorily to all parties concerned, that an equivalent moisture result is obtained when compared to the applicable reference method specified in 7.2. This demonstration shall include ISO 3086 bias testing using a relevant bias of no more than 0,05 % moisture and

where the confidence interval includes the zero. Further, the precision of the alternative method needs to be demonstrated by ISO 3085 to be the same or better than achieved by the applicable reference method.

If the sample is subsequently to be utilized for multiple use, the chemistry, LOI and size distribution of the dried remains shall be checked by means of ISO 3086 to ensure they are not different to dried remains from equivalent reference moisture testing methods. If any changes are identified, the alternative method must be discarded.

7.3.2 Adjustment of drying time only

In the case where an alternative method changes only the drying times specified in 7.2.2 in order to eliminate repeat weighings for verification of constant mass, then it is only necessary to demonstrate equivalence of precision and moisture result.

NOTE The notes in 7.2.2 apply to this subclause

8 Verification

Regular checking of apparatus and procedures is essential to verify the test results. Checks shall be carried out prior to the commencement of a routine test in accordance with this International Standard and at regular intervals thereafter. The frequency of checking is a matter for each laboratory to determine. A detailed record of all verification activities shall be maintained for the following items:

a) Sprinkled water measurement

— volumenometer;

b) Rainfall measurement

— rain gauge;

c) Moisture test

— oven temperature/temperature regulation;

— circulation and change of air in oven;

— weighing device.

9 Calculation and expression of results

9.1 Test portion

The result of the determination of the moisture content, w_i, expressed as a mass fraction (%), for each test portion, is given by Formula (1) and reported to the second decimal place.

$$w_i = \frac{m_1 - m_2}{m_1} \times 100 \quad (1)$$

where

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

m_2 is the mass, in grams, of the test portion after drying.

An illustration of moisture determination of a random test portion is shown in an example test report in Annex D, Table D.1.

9.2 Lot

The moisture content of a lot is given by one of the Formulas (2) to (6) as the occasion may demand, and reported to the first decimal place.

Sprinkled water and/or rainwater over iron ore during loading and/or unloading operation, in other words moisture added after/before the point where sampling has taken place, shall be corrected according to the procedure specified in Annex B.

9.2.1 When moisture determination is conducted on the gross sample from the lot, the moisture of the lot is determined as follows.

When the range of the four test results does not exceed 1,3r as given in Table 3, the arithmetic mean, $\bar{w}$, of the four results shall be the moisture content, expressed as a mass fraction (%), of the lot as given by Formula (2).

$$\bar{w} = \frac{w_1 + w_2 + w_3 + w_4}{4} \tag{2}$$

where w_1, w_2, w_3 and w_4 are the results of the determinations of the moisture contents, expressed as a mass fraction (%), on each of the four test portions.

When the range of the four test results exceeds 1,3r as given in Table 3, the median shall be taken as the moisture content of the lot. The median of four test results is defined as the mean of the two non-extreme test results.

Table 3 — Repeatability limit of moisture determination on the gross sample

Average of moisture content $\bar{w}$ mass fraction (%)	Repeatability limit r [a] mass fraction (%)	Repeatability limit 1,3r mass fraction (%)
$\bar{w} \leq 3$	0,20	0,26
$3 < \bar{w} \leq 6$	0,25	0,33
$6 < \bar{w}$	0,31	0,40

[a] The theoretical background of the repeatability limit is shown in Annex C.

An illustration of moisture determination of a gross sampled lot with four test portions, where the range of the four moistures do not exceed 1,3r, is shown in an example test report in Annex D, Table D.2.

An illustration of moisture determination of a gross sampled lot with four test portions, where the range of the four moistures exceed 1,3r, is shown in an example test report in Annex D, Table D.3.

9.2.2 When mass-basis sampling has been performed and moisture determination is conducted on each partial sample, the weighted mean, $\overline{\overline{w}}$, of the results from all the partial samples, considering the number of increments for each partial sample, shall be the moisture content, expressed as a mass fraction (%), of the lot, as given by Formula (3).