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Metallic powders — Determination of oxygen content by reduction methods —

Part 2: Loss of mass on hydrogen reduction (hydrogen loss)

Poudres métalliques — Dosage de l'oxygène par les méthodes de réduction —

Partie 2: Perte de masse par réduction dans l'hydrogène (perte dans l'hydrogène)

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 119 *Powder metallurgy*, Subcommittee SC 2, *Sampling and testing methods for sintered metal materials (excluding hardmetals),* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/SS M11, *Powder metallurgy,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 4491-2:1997), which has been technically revised.

The main changes compared to the previous edition are as follows:

— adding of precision statement.

A list of all parts in the ISO 4491 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Metallic powders — Determination of oxygen content by reduction methods —

Part 2: Loss of mass on hydrogen reduction (hydrogen loss)

1 Scope

This document specifies a method for the determination of the relative loss of mass which a metallic powder undergoes when heated in a stream of pure dry hydrogen under specified conditions.

The purpose of this test is to evaluate a chemical powder characteristic which is of importance to the powder metallurgical industry. The test is not intended as a means for the determination of the content of specific elements (see Annex A and ISO 4491-1).

The test method is applicable to unalloyed, partially alloyed and completely alloyed powders of the metals listed in Table 1 (see 7.2.1). It is not applicable to lubricated powders or to mixtures of metal powders.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Reagents and materials

Hydrogen, with a maximum oxygen content of 0,005 % mass fraction and a dew point not higher than -45 °C.

Nitrogen or argon, with a maximum oxygen content of 0,005 % mass fraction and a dew point not higher than -45 °C (see also 7.2.3, third paragraph).

5 Apparatus

5.1 General

An example of suitable test arrangement is shown schematically in Figure 1.

5.2 Balance

Laboratory balance, of sufficient capacity, and capable of weighing to ±0,1 mg.

5.3 Furnace

Electrically heated tubular furnace, that can be continuously operated at the appropriate temperatures given in Table 1 and that has a control system capable of maintaining the temperature in that part of the tube containing the boat (5.6) to within the temperature tolerance stated in Table 1.

When testing magnetic powders, it is recommended that wire-wound furnaces are wound non-inductively.

5.4 Gas-tight tube

Gas-tight tube, of quartz or refractory material (for example dense alumina). The inside diameter of the tube shall be between 25 mm and 40 mm and its length such that it extends about 200 mm beyond each end of the furnace.

When a large number of hydrogen loss determinations is to be carried out, a larger furnace than that described in this document, and one which permits several test portions to be tested simultaneously, may be used, provided that the temperature and time conditions shown in Table 1 are fulfilled and the results obtained are in agreement with those obtained when the test is carried out with the preferred apparatus.

5.5 Thermocouple

Totally enclosed thermocouple, for example platinum/platinum-rhodium, and an indicating or recording instrument, permitting the measurement of temperature with maximum permissible error of 5 °C.

If for some reason it is desirable to place the thermocouple outside the reduction tube, this is acceptable. But in this case, a preliminary calibration shall be made with a second thermocouple placed inside the tube to ascertain that the temperature of the test sample is in accordance with the values and tolerances specified in Table 1.

5.6 Boat

Boat, preferably of high-alumina ceramic with a polished surface. Other materials, for example quartz and nickel, may be used when test conditions allow. The boat shall be of such dimensions, for example 75 mm long and 12 mm wide, that the thickness of the powder, when uniformly distributed, does not exceed 3 mm.

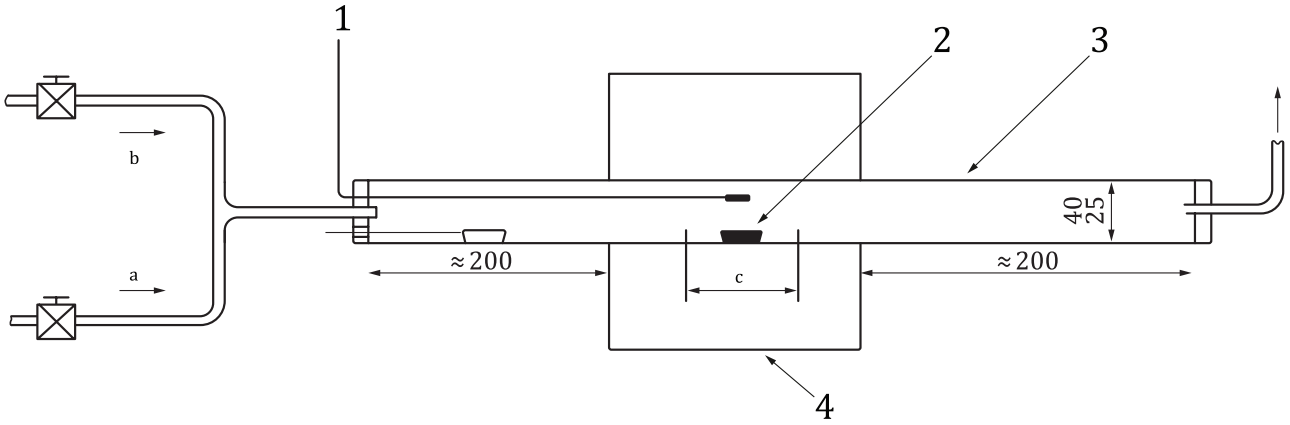
New boats shall be pre-treated in a stream of hydrogen at the test temperature and stored in a desiccator.

A boat may be used more than once, provided that it is always used for testing the same metal powder or type thereof and provided that it is carefully cleaned by mechanical means between determinations and stored in a desiccator.

5.7 Supply unit

Supply unit for hydrogen and either nitrogen or argon, with pressure gauges and flow meters to control the flow of gas.

Dimensions in millimetres



Key

1 thermocouple (5.5)
2 boat (5.6)
3 quartz tube (5.4)
4 furnace (5.3)

a Nitrogen or argon inlet.
b Hydrogen inlet.
c Heating zone.

Figure 1 — Diagram of suitable test arrangement

6 Sampling

The powder shall be tested in as-received condition.

The loss in mass shall be determined on two test portions.

The mass of the test portion shall be approximately 5 g, except for powders of low apparent density it may be reduced to comply with the requirements of 5.6 and 7.2.2.

7 Procedure

7.1 General

Carry out two determinations on each test sample.

7.2 Test procedure

7.2.1 Heat the furnace (5.3), with the tube (5.4) inserted, to the temperature indicated in Table 1 for the metal powder being tested.

Table 1 — Reduction temperatures and times

Metal powder	Reduction temperature °C	Reduction time[a] min
Tin bronze[b]	750 ± 15	30
Tin	425 ± 10	30

a) These reduction times are given for guidance purposes only. Shorter times may be applied provided that for each apparatus and for each type of powder experience has shown them to be sufficient to guarantee the completion of the hydrogen loss reactions.

b) Results should be interpreted with care. See A.6.

Table 1 *(continued)*

Metal powder	Reduction temperature ° C	Reduction time[a] min
Silver	550 ± 10	30
Copper	850 ± 15	30
Copper lead[b]	600 ± 10	10
Leaded bronze[b]	600 ± 10	10
Iron and steel	1 100 ± 20	60
Cobalt	1 000 ± 20	60
Nickel	1 000 ± 20	60
Tungsten	1 000 ± 20	60
Molybdenum	1 100 ± 20	60
Rhenium	1 150 ± 20	60

a) These reduction times are given for guidance purposes only. Shorter times may be applied provided that for each apparatus and for each type of powder experience has shown them to be sufficient to guarantee the completion of the hydrogen loss reactions.

b) Results should be interpreted with care. See A.6.

7.2.2 Weigh the boat (5.6) to the nearest 0,1 mg. Distribute the test portion throughout the boat to a uniform depth not exceeding 3 mm. Weigh the boat with the test portion to the nearest 0,1 mg.

7.2.3 Pass nitrogen (see Clause 4) through the tube at a flow rate corresponding to a gas speed of at least 25 mm/s, as measured in the cooling zone of the tube, for a period of at least 1 min. Insert the boat containing the test portion in the tube and move it until it is at the centre of the uniform-temperature zone of the furnace. The boat shall be moved sufficiently slowly to prevent ejection of powder as a result of a high rate of gas evolution. Continue the flow of nitrogen for 1 min.

If difficulties are experienced in preventing ejection of powder from the boat, the powder may be pressed (without addition of lubricant) to form a low density compact, or, if such a compact has a very low green strength, it may be wrapped in oxide-free copper foil. The copper foil can only be used when the test temperature exceeds the melting temperature of copper.

When testing powders that are susceptible to combination with nitrogen (for example chromium-containing alloy steel powder), the purging operations shall be carried out with argon instead of nitrogen (see 7.2.5 and 7.2.6).

7.2.4 Start up the flow of hydrogen (see Clause 4) and discontinue the flow of nitrogen. Establish an even flow of hydrogen through the tube, corresponding to a gas speed of at least 25 mm/s in the cold zone of the tube. This is equivalent to approximately 50 l/h for a tube of 25 mm diameter and approximately 110 l/h for a tube of 40 mm diameter. Maintain the flow of hydrogen for the period of time indicated in Table 1. During this time, maintain the temperature of the furnace within the prescribed range.

7.2.5 At the end of the prescribed time, start up the flow of nitrogen again and discontinue the flow of hydrogen. Withdraw the boat after 2 min to 3 min to the cool part of the tube beyond the end of the furnace.

7.2.6 Allow the boat with the reduced test portion to cool in the nitrogen atmosphere to below 35 °C, remove it from the tube and permit it to cool to ambient temperature in a desiccator.

7.2.7 Weigh the boat with the reduced test portion to the nearest 0,1 mg.

8 Calculation and expression of test results

The hydrogen loss f_{HL}, expressed as a mass fraction in percent, is given by Formula (1):

$$f_{HL} = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (1)$$

where

f_{HL} is the hydrogen loss expressed as a mass fraction in percent;

m_1 is the mass, in grams, of the empty pre-treated boat (5.6);

m_2 is the mass, in grams, of the boat with the test portion before the test;

m_3 is the mass, in grams, of the boat with the reduced test portion after test.

The mean of the two determinations is calculated. This calculation and the expression of the test results are carried out according to the rules shown in Table 2.

Table 2 — Calculation of mean value

Hydrogen loss % (mass fraction)	**Calculation of test results to the nearest** %	**Maximum permissible difference between two determinations**	**Expression of results to the nearest** %
≤ 0,2	0,01	max. 0,01 % (absolute value)	0,01
> 0,2 ≤ 0,5	0,01	max. 5 % of mean	0,02
> 0,5 ≤ 1,0	0,01	max. 5 % of mean	0,05
> 1,0	0,01	max. 5 % of mean	0,1

9 Precision

Four different types of metal powders, see Table 3, were in included in the inter-laboratory study to develop this precision statement.

Table 3 — Type of powders included in the inter-laboratory study

Powder type
Pure atomized iron powder
Sponge iron powder
Water atomized copper powder
Tungsten powder

In Table 4, the repeatability and reproducibility are presented as one standard deviation.

Table 4 — Repeatability and reproducibility as standard deviations

Tested powder	**Level** Average H_2-loss %	**Repeatability** (S_r) Standard deviation %	**Reproducibility** (S_R) Standard deviation %
Pure atomized iron powder	0,09	0,006	0,019
Sponge iron powder	0,15	0,019	0,035
Water atomized copper powder	0,18	0,020	0,037
Tungsten powder	0,08	0,012	0,014

The difference between two test results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability limit (*r*), see Table 5, on average not more than once in 20 cases in the normal and correct operation of the method.

Test results on identical test material reported by two laboratories will differ by more than the reproducibility limit (*R*), see Table 5, on average not more than once in 20 cases in the normal and correct operation of the method

Table 5 — Repeatability and Reproducibility, difference between two tests at 95 % probability level

Tested powder	**Level** Average H_2-loss %	**Repeatability** Limit *(r)* %	**Reproducibility** Limit *(R)* %
Pure atomized iron powder	0,09	0,017	0,053
Sponge iron powder	0,15	0,052	0,097
Water atomized copper powder	0,18	0,057	0,103
Tungsten powder	0,08	0,033	0,039

The precision data were determined from an experiment organized and analysed in accordance with ISO 5725-2 in year 2020 where each of the powders included in the investigation was analysed two times by 7 laboratories.

The laboratories participating in the study were following the test procedure described in ISO 4491-2. All participating laboratories used their own test equipment (furnace, scale and boats) for the study.

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 4491-2:2023;

b) all details necessary for the identification of the test sample;

c) the test results obtained;

d) details of any operations not specified in this document, or regarded as optional;

e) details of any occurrence which may have affected the results

f) the date of the test.