INTERNATIONAL STANDARD

ISO 4491-2

Second edition
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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)

Reference number
ISO 4491-2:1997(E)

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.

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.

International Standard ISO 4491-2 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 2, *Sampling and testing methods for powders (including powders for hardmetals)*.

This second edition cancels and replaces the first edition (ISO 4491-2:1989), table 1 and clause 7 of which have been technically revised.

ISO 4491 consists of the following parts, under the general title *Metallic powders — Determination of oxygen content by reduction methods*:

— *Part 1: General guidelines*

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

— *Part 3: Hydrogen-reducible oxygen*

— *Part 4: Total oxygen by reduction-extraction*

Annex A forms an integral part of this part of ISO 4491.

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Metallic powders — Determination of oxygen content by reduction methods —

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

1 Scope

This part of ISO 4491 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 6.1). It is not applicable to lubricated powders or to mixtures of metal powders.

The results can be influenced by the presence of reducible, oxidizable or volatile metals, metalloids or compounds (see annex A). The results obtained on such powders shall be used with caution and their interpretation shall be subject to agreement between supplier and user.

This part of ISO 4491 shall be read in conjunction with ISO 4491-1.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 4491. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 4491 are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4491-1:1989, *Metallic powders — Determination of oxygen content by reduction methods — Part 1: General guidelines.*

3 Reagents and materials

3.1 Hydrogen, with a maximum oxygen content of 0,005 % (m/m) and a dew point not higher than – 45 °C.

3.2 Nitrogen or **argon**, with a maximum oxygen content of 0,005 % (m/m) and a dew point not higher than – 45 °C.

(See also 6.3, third paragraph.)

4 Apparatus

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

4.1 Laboratory balance, of sufficient capacity, and capable of weighing to an accuracy of 0,1 mg.

4.2 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 (4.5) to within the temperature tolerance stated in table 1.

NOTE — When testing magnetic powders, it is recommended that wire-wound furnaces shall be wound non-inductively.

4.3 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 part of ISO 4491, 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.

4.4 Totally enclosed thermocouple, for example platinum/platinum-rhodium, and an **indicating or recording instrument**, permitting the measurement of temperature with an accuracy 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 in order to ascertain that the temperature of the test sample is in accordance with the values and tolerances specified in table 1.

4.5 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 pretreated 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.

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

5 Sampling

5.1 The powder shall be tested in the as-received condition.

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

5.3 The mass of the test portion shall be approximately 5 g, except that for powders of low apparent density it may be reduced to comply with the requirements of 4.5 and 6.2.

Dimensions in millimetres

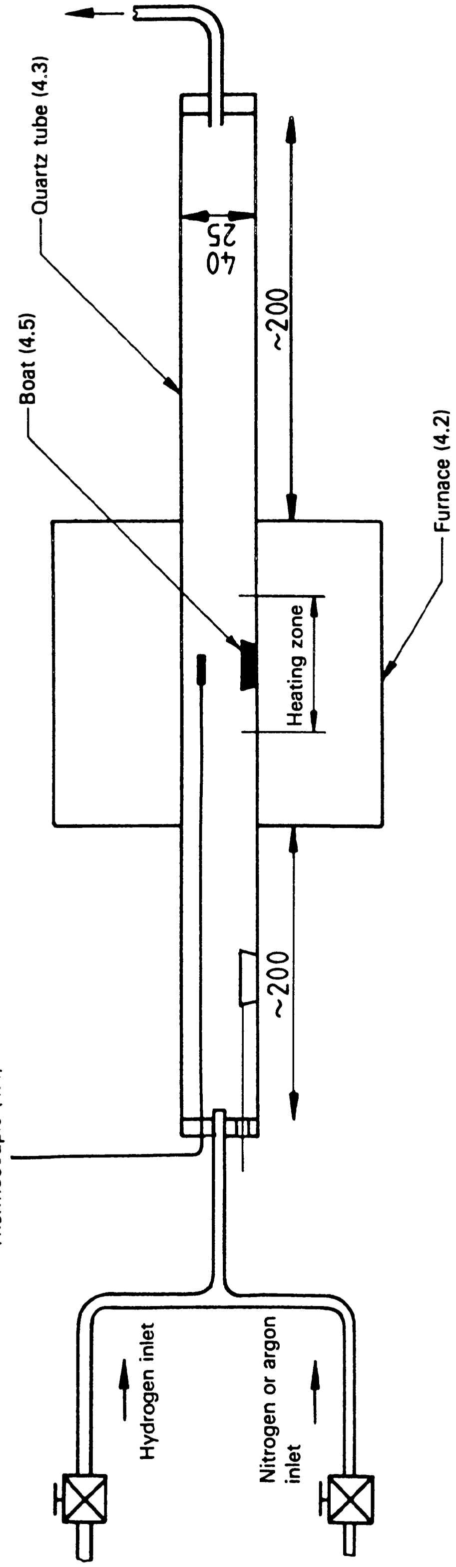

Figure 1 — Diagram of suitable test arrangement

6 Procedure

Carry out two determinations on each test sample.

6.1 Heat the furnace (4.2), with the tube (4.3) 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[1] min
Tin bronze[2]	750 ± 15	30
Tin	425 ± 10	30
Silver	550 ± 10	30
Copper	850 ± 15	30
Copper lead[2]	600 ± 10	10
Leaded bronze[2]	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

1) 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.

2) Results should be interpreted with care. See annex A, clause A.6.

6.2 Weigh the boat (4.5) 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.

6.3 Pass nitrogen (3.2) 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 cooper foil. The copper foil may be used only 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 6.5 and 6.6.)

6.4 Start up the flow of hydrogen (3.1) 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.

6.5 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.

6.6 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.

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

7 Calculation and expression of test results

7.1 The hydrogen loss HL, expressed as a percentage by mass, is given by the formula

$$\mathrm{HL} = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the empty pretreated boat (4.5);

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.

7.2 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

Hydrogen loss % (*m*/*m*)	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

8 Test report

The test report shall include the following information:

a) a reference to this part of ISO 4491;

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 part of ISO 4491, or regarded as optional;

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

Annex A
(normative)

Interpretation of results

A.1 The loss of mass of a powder on hydrogen reduction, commonly called hydrogen loss, is a powder characteristic that has proved useful in the processing of powder metallurgical materials. It was originally considered to be an estimate of the oxygen content of hydrogen-reducible oxides but, with the advent of more complex and alloyed powders, it is now thought that other chemical changes may contribute, positively or negatively, to the measured loss in mass. Thus the following factors should be considered when interpreting experimental results.

A.2 The measured loss of mass does not include oxygen present in the form of oxides such as SiO_2, Al_2O_3, MgO, CaO, BeO and TiO_2, which are not reducible under the test conditions.

A.3 The loss of mass includes any water vapour and/or hydrocarbons present in the powder.

A.4 The loss of mass includes gases which, owing to either adsorption or occlusion, may be present in the powder and which are liberated during heating. The amount of such gases is normally negligible.

A.5 The loss of mass includes elements other than oxygen which are present in the powder and which under the specified test conditions are partially or completely removed, either because they are volatile or because they react with the hydrogen or with oxides present in the powder to form volatile compounds. Examples are carbon, nitrogen, phosphorus and sulfur.

A.6 The loss of mass includes metals which are present in the powder and which under the specified test conditions are volatile and therefore partially or completely removed during the test. Lead, zinc and cadmium are examples of such metals.

A.7 If carbon is present in the powder, the loss of mass during the hydrogen loss test may include oxygen from oxides which under the specified test conditions are reduced by the carbon. Examples of such oxides are Cr_2O_3 and MnO which, when present in carbon-containing steel powders, may be reduced by carbon under the specified test conditions.

A.8 Powders containing manganese and/or chromium, or other elements with a high affinity for oxygen, may be oxidized during the test by the atmosphere or through reduction of less refractory oxides. In extreme cases this can result in a negative figure for hydrogen loss (i.e. an increase of mass during the test).

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