INTERNATIONAL STANDARD 4491

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Metallic powders — Determination of loss of mass on hydrogen reduction (hydrogen loss)

Poudres métalliques — Détermination de la perte de masse après réduction par l'hydrogène (perte dans l'hydrogène)

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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.

International Standard ISO 4491 was developed by Technical Committee ISO/TC 119, *Powder metallurgical materials and products*, and was circulated to the member bodies in June 1977.

It has been approved by the member bodies of the following countries :

Australia	Germany	Spain
Austria	Italy	Sweden
Bulgaria	Korea, Rep. of	United Kingdom
Canada	Mexico	U.S.A.
Chile	Poland	U.S.S.R.
Czechoslovakia	Portugal	Yugoslavia
Egypt, Arab Rep. of	Romania	
France	South Africa, Rep. of	

No member body expressed disapproval of the document.

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Metallic powders — Determination of loss of mass on hydrogen reduction (hydrogen loss)

1 SCOPE

This International Standard 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 the annex.)

2 FIELD OF APPLICATION

This test method is applicable to unalloyed, partially alloyed and completely alloyed powders of the metals listed in the table. 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 present (see the annex). The results obtained on such powders shall be used with caution and their interpretation shall be subject to agreement between supplier and user.

3 REAGENTS

The following two gases, with a maximum oxygen content of 0,005 % and a dew point not higher than − 45 °C.

3.1 Hydrogen.

3.2 Nitrogen or **argon.**

4 APPARATUS

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

4.1 Laboratory balance of sufficient capacity, and capable of weighing to an accuracy of ± 0,000 1 g.

4.2 Electrically heated tubular furnace that can be continuously operated at the appropriate temperatures given in the table and that has a control system capable of maintaining the temperature in that part of the tube containing a boat within the temperature tolerance stated in the table.

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

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

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

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.

4.5 Boats, 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 should 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 Cylinders containing 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.

6 PROCEDURE

6.1 Heat the furnace with the tube inserted, to establish the temperature indicated in the table for the metal powder being tested.

TABLE — **Reduction temperatures and times**

Metal powder	Reduction temperature °C	Reduction time min
Tin bronze	775 ± 15	30
Cobalt	1 050 ± 20	60
Copper	875 ± 15	30
Copper lead[1] and Leaded bronze[1]	600 ± 10	10
Iron	1 150 ± 20	60
Alloyed steel	1 150 ± 20	60
Lead[1]	550 ± 10	30
Molybdenum	1 100 ± 20	60
Nickel	1 050 ± 20	60
Tin	550 ± 10	30
Tungsten	1 150 ± 20	60

1) Results should be interpreted with caution. See the annex, clause A.5.

6.2 Weigh the boat to the nearest 0,000 1 g. 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 0,000 1 g.

6.3 Pass the nitrogen through the tube at flow rate corresponding to a gas velocity 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.

NOTES

1 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, wrapped in oxide-free copper foil. The copper foil may be used only when the test temperature exceeds the melting temperature of copper.

2 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 Introduce hydrogen and discontinue the flow of nitrogen. Establish an even flow of hydrogen through the tube, corresponding to a gas velocity 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 the table. During this time, the temperature of the furnace shall be maintained within the prescribed range.

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

6.6 Allow the boat with the 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 test portion to the nearest 0,000 1 g.

7 EXPRESSION OF RESULTS

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

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

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 test portion after the test.

7.2 The maximum permissible difference between the two determinations shall not exceed 0,04 % in absolute value when the hydrogen loss is less than 0,8 %. When the hydrogen loss is equal to or greater than 0,8 %, the difference shall not exceed 5 % of the mean value. The result of each determination shall be calculated to the nearest 0,01 %.

7.3 The hydrogen loss is calculated as the mean of the two results and reported to the nearest 0,02 % for losses up to and including 0,8 % and to the nearest 0,05 % for losses greater than 0,8 %.

8 TEST REPORT

The test report shall include the following information :

a) reference to this International Standard;

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

c) the result obtained;

d) all operations not specified in this International Standard, or regarded as optional;

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

ANNEX

INTERPRETATION OF RESULTS

A.0 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.1 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.2 The loss of mass includes any water vapour and/or hydrocarbons present in the powder.

A.3 The loss of mass includes gases which, owing to either adsorption or occlusion, may be present in the powder and which are liberated during its heating. The amount of such gases is normally negligible.

A.4 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 eliminated, 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 sulphur.

A.5 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 eliminated during the test. Lead, zinc and cadmium are examples of such metals.

A.6 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.7 Powders containing manganese and/or chromium, or other elements with a high affinity for oxygen, may be oxidized during the test from 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).

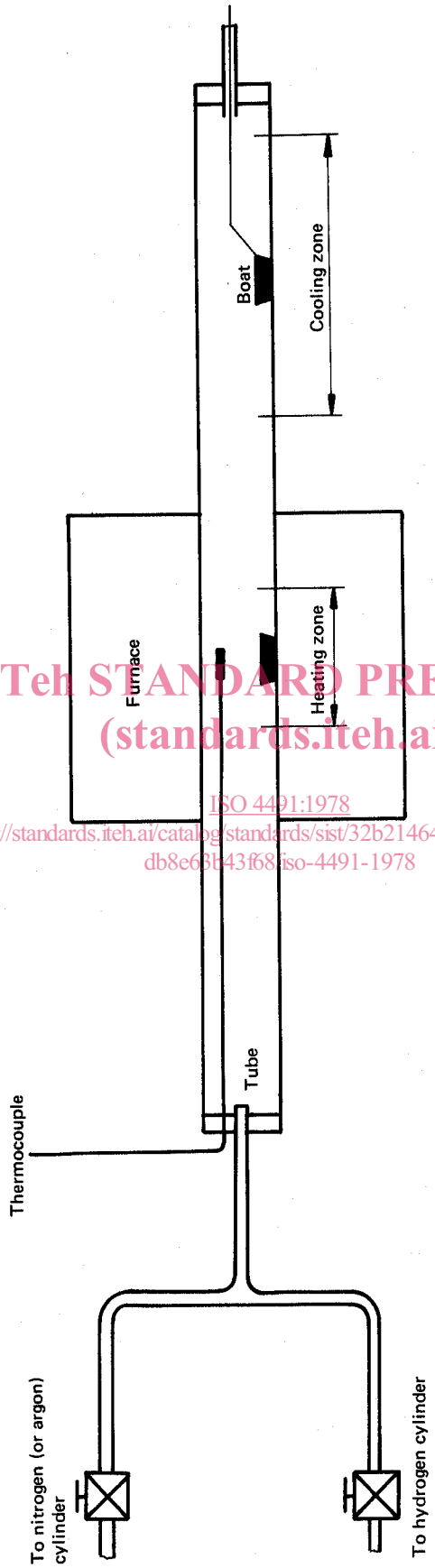

FIGURE — Test arrangement