SLOVENSKI STANDARD
oSIST prEN ISO 4491-2:2022

01-julij-2022

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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) (ISO/DIS 4491-2:20022)

Ta slovenski standard je istoveten z: **prEN ISO 4491-2**

ICS:

77.160	Metalurgija prahov	Powder metallurgy

oSIST prEN ISO 4491-2:2022 **en,fr,de**

DRAFT INTERNATIONAL STANDARD

ISO/DIS 4491-2

ISO/TC **119**/SC **2** Secretariat: **SIS**

Voting begins on: **2022-05-23**

Voting terminates on: **2022-08-15**

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)

ICS: 77.160

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 4491-2:2022(E)

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Published in Switzerland

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Foreword

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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)*,

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

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 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.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 4491-1:1989, *Metallic powders — Determination of oxygen content by reduction methods — Part 1: General guidelines*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological 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 % (m/m) and a dew point not higher than -45 °C.

Nitrogen or argon, with a maximum oxygen content of 0,005 % (m/m) 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 an accuracy of ±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 shall be 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 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 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.

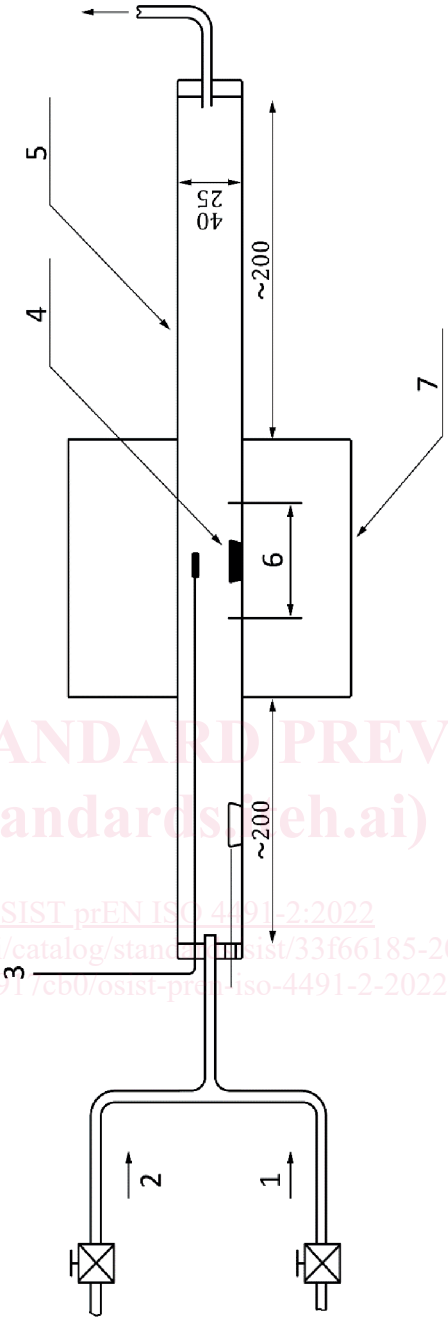
New boats shall be pretreated in a stream of hydrogen at the test temperature and stored in a desiccator.

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.

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 mm



Key

1	nitrogen or argon inlet	5	quartz tube (5.4)
2	hydrogen inlet	6	heating zone
3	thermocouple (5.5)	7	furnace (5.3)
4	boat (5.6)		

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 7.3.

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[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.

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 (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 cooper foil. The copper foil may be used only when the test temperature exceeds the melting temperature of copper.