INTERNATIONAL STANDARD

ISO 4491-4

Second edition
2013-05-01

Metallic powders — Determination of oxygen content by reduction methods —

Part 4: Total oxygen by reduction-extraction

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

Partie 4: Oxygène total par réduction-extraction

Reference number
ISO 4491-4:2013(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword ... **iv**
Introduction ... **v**
1 Scope ... **1**
2 Normative references ... **1**
3 Principle ... **1**
4 Apparatus and materials ... **2**
5 Test portion ... **2**
6 Procedure ... **3**
6.1 General ... 3
6.2 Blank test and calibration ... 3
6.3 Test ... 4
7 Expression of results ... **4**
7.1 Permissible tolerances ... 4
7.2 Final result ... 4
8 Test report ... **4**
Annex A (informative) **Examples of conditions of extraction for selected metal powders** ... **5**

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.

ISO 4491-4 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 4491-4:1989), of which it constitutes a minor revision.

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

Introduction

The determination of the oxygen content of metallic powders is of the utmost importance in many fields of powder metallurgy.

The standard methods described in ISO 4491-2 and ISO 4491-3 do not give the total oxygen content of the sample, as some oxygen-containing constituents are not reduced by hydrogen.

Therefore, a standard method for the determination of the total oxygen content is needed. The most frequently used method is reduction-extraction. It can be carried out with various commercially available instruments working according to different principles of extraction and measurement.

It should be emphasized that the results of the analysis depend on the type of equipment used and on the test parameters selected. However, as indicated in Clauses 3 to 6, it is always possible, for a given type of metal powder, to optimize the test conditions to obtain reproducible and accurate results with any of the commercially available instruments, provided they are designed for testing the metal powder considered.

It is not possible to standardize one or more particular instruments. However, certain basic points of procedure are recommended for the analysis of metallic powders (see Clause 6).

NOTE The reduction-extraction method is also applicable to nitrogen determination and certain instruments permit simultaneous measurement of oxygen and nitrogen contents. However, the determination of nitrogen is not covered by this International Standard.

Metallic powders — Determination of oxygen content by reduction methods —

Part 4: Total oxygen by reduction-extraction

1 Scope

This part of ISO 4491 specifies a method for the determination of the total oxygen content of metallic powders by reduction-extraction at high temperature.

By agreement, this method is also applicable to the determination of the total oxygen content of sintered metal materials.

The method is applicable to all powders of metals, alloys, carbides, and mixtures thereof which are non-volatile under the test conditions. The sample may be in powder or compact form.

The analysis is carried out on the powder as supplied, but the method is not applicable if the powder contains a lubricant or binder. If such substances are present, the method may be used only if they can first be completely removed by a method not affecting the oxygen content of the powder.

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

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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, *Metallic powders — Determination of oxygen content by reduction methods — Part 1: General guidelines*

3 Principle

A test portion of the sample is heated in a graphite crucible at high temperature, either under vacuum or in a flow of an inert carrier gas. Oxygen in the sample is converted to oxides of carbon. These are extracted and transformed completely to either carbon monoxide or carbon dioxide, which is determined by a suitable gas analysis method.

The methods used in practice to determine the total oxygen content have the following features:

a) Environment in the reaction chamber:

   — Vacuum or

   — flow of inert gas (nitrogen, argon, helium).

b) Graphite crucible:

   — Individual, i.e. used only for one test portion, or

   — cumulative, i.e. the same crucible is used for the analysis of several successive test portions.

c) Reaction medium:

— Dry, i.e. the test portion alone is poured into the graphite crucible, the reduction being carried out in the solid state if the metal being analysed does not melt, or

— metal bath, i.e. in order to accelerate the reduction of certain metals it is advisable to prepare first a bath of a fusible metal (for example platinum, tin, iron, nickel) capable of dissolving both carbon and the metal in the test portion.

d) Heating:

— Continuous, i.e. the test portion is introduced into the crucible previously heated to the reaction temperature, the reduction taking place over a fixed period of time, of the order of several minutes, or

— pulse, i.e. the cold crucible containing the test portion is heated by injecting, over a period of a few seconds, a high-power pulse of energy, reduction taking place very rapidly at the high peak temperature (up to 3 000 °C) which results.

e) Determination of oxygen:

Several methods for measuring either CO or CO_2 are available. In both cases a chemical conversion device is used to ensure that the oxygen to be determined is transformed completely into either CO or CO_2. The analytical methods commonly used are

— volumetric (for carbon monoxide),

— chromatography (for carbon monoxide),

— infrared absorption (for carbon monoxide),

— thermal conductivity (for carbon monoxide and carbon dioxide),

— coulometry (for carbon dioxide).

4 Apparatus and materials

The main elements of an apparatus suitable for determining the oxygen content of a metallic powder are the following:

— crucibles, machined from high purity graphite;

— a device to degas the graphite crucible at high temperature;

— a device to introduce the test portion and degas it under inert gas or in vacuum at ambient temperature;

— a device for gas extraction in accordance with a predetermined temperature cycle;

— a purification train to remove water;

— a measuring device for the determination of the carbon monoxide or carbon dioxide.

The materials needed will depend on the type of equipment used, for example high purity inert gas (helium or argon).

Calibration of the measuring device, when necessary, requires high purity gas, carbon monoxide, carbon dioxide, or certified metallic reference materials.

5 Test portion

The analysis shall be carried out on one or several test portions. The number of test portions required to reach the required precision can be determined by a gauge repeatability and reproducibility study. If a gauge

repeatability and reproducibility study is not made, the analysis shall be carried out on two test portions. Several methods can be used to prepare the test portion prior to its introduction into the apparatus.

a) The test portion is weighed directly into the degassed crucible.

b) A quantity of the powder sample is uniaxially compacted in a small cylindrical die, without any lubricant or binder, under a pressure of 100 MN/mm^2 to 200 MN/mm^2. The mass of the compact is determined.

c) A quantity of the powder sample is enclosed in a small capsule of known weight made of platinum, tin, nickel, or iron-nickel foil of high purity. The whole capsule is weighed. The oxygen content of the foil shall be known or determined previously.

d) In the case of a compact, a suitable fragment of the sample is weighed as the test portion.

All weighings shall be to the nearest 0,1 mg.

A metal foil capsule may be used solely to facilitate the introduction of the sample into the apparatus. In this case, the weight of the capsule shall be kept to a minimum.

Alternatively, the metal of the capsule can constitute the metal bath needed for convenient extraction; in this case, the mass of the capsule is chosen to give the bath/test-portion mass ratio recommended for the particular analysis.

When the graphite crucible is used with a metal bath for several consecutive analyses, it is necessary to degas the bath prior to the beginning of each extraction operation.

The bath/test-portion mass ratio is maintained larger than the recommended minimum value, if necessary, by the periodic introduction of fragments of metal followed by degassing of the bath.

The mass of the test portion shall be selected depending on the sensitivity of the apparatus used and the expected oxygen content. Frequently, a mass between 0,1 g and 1 g is chosen.

6 Procedure

6.1 General

For the reason given in the introduction, it is not possible to specify the conditions of oxygen determination for each of the various metals, alloys, and carbides to be analysed, and for each of the types of apparatus available. It should be noted that, especially when the reduction is carried out in the solid state and with continuous heating, the reaction may be slow and the time for complete reduction of the oxides will depend on the oxygen content.

It is recommended that the optional conditions for testing a given type of material and for a given range of oxygen contents be determined by performing preliminary tests. It is common to make successive tests on the same sample, increasing the reducing action (i.e. by increasing the temperature and/or time of reaction) until the measured oxygen content reaches a maximum constant value. Other parameters (e.g. use of metal bath) may also be varied.

It is strongly advisable to use certified reference materials of the same type as the sample to ensure the correctness of the operating conditions adopted.

6.2 Blank test and calibration

Generally, a blank test is carried out under the same conditions as those selected for the determination, but excluding the test portion.

If necessary, the apparatus is calibrated, or verified to be in correct working order, in accordance with the manufacturer's instructions, generally using pure gases (carbon monoxide, carbon dioxide) or reference materials of certified oxygen content.

6.3 Test

The test is carried out in accordance with the instructions for operating the equipment using the conditions selected (see 6.1). Annex A presents, as examples, conditions of reduction for some metal powders.

7 Expression of results

7.1 Permissible tolerances

The difference between the two determinations shall not exceed the values shown in Table 1.

7.2 Final result

If several determinations were made, the result is calculated as the arithmetical mean value. The result is rounded in accordance with Table 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 method of extraction of lubricant or binder, if relevant;

d) the type of equipment used;

e) all relevant conditions of testing (temperature, time, whether a metal bath or capsule was used, etc.);

f) the final result obtained (see 7.2);

g) details of any operations not specified in this part of ISO 4491, or regarded as optional;

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

Table 1 — Requirements of reported value

Oxygen content, % (*m*/*m*)	**Maximum permissible difference between the two determinations**	**Rounded to the nearest**
≤ 0,005	20 % of the mean value	0,000 5
Over 0,005 to 0,01	10 % of the mean value	0,001
Over 0,01 to 0,02	10 % of the mean value	0,002
Over 0,02 to 0,05	5 % of the mean value	0,002
Over 0,05 to 0,1	5 % of the mean value	0,005
Over 0,1 to 0,2	5 % of the mean value	0,01
Over 0,2 to 0,5	5 % of the mean value	0,02
Over 0,5 to 1,0	5 % of the mean value	0,05
Over 1,0	5 % of the mean value	0,1

Annex A
(informative)

Examples of conditions of extraction for selected metal powders

See Table A.1.

Table A.1 — Examples of conditions of extraction for selected metal powders

Metal powder	Reaction medium	Minimum bath/ test-portion mass ratio	Temperature[a] °C
Iron, steel	Without bath or capsule	-	2 000
Titanium	Nickel bath	12:1	2 100
Titanium, zirconium, and hafnium	Platinum capsule and platinum bath	20:1	2 100
Molybdenum and tungsten	Without bath	-	2 400
Niobium and tantalum	Nickel and tin bath	5:1	2 400
Aluminium	Copper bath	5:1	2 400
Copper	Without bath	-	1 900
Hardmetal mixture	Iron-nickel and tin capsule	4:1	2 400

[a] These temperatures are practical values used with continuous heating (time 1 min to 10 min, depending on gas content). Extraction in a pulse furnace is carried out generally at a temperature in excess of 3 000 °C. Normally, a time between 4 s and 20 s is sufficient for complete reaction.

ICS 77.160
Price based on 5 pages