
**Metallic powders — Determination of
oxygen content by reduction methods —**

Part 3:
Hydrogen-reducible oxygen

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

Partie 3: Oxygène réductible par l'hydrogène



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 4491-3 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 4491-3:1989), clauses 7 and 8 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*

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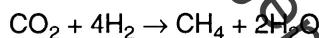
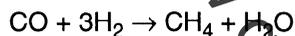
Introduction

The International Standard which the first edition of this part of ISO 4491 (ISO 4491-3:1989) replaced (ISO 4993:1981) described one particular arrangement of apparatus and procedure for the determination of hydrogen-reducible oxygen. However, it has since been established that other schemes will give equally valid results and these are therefore described in this part of ISO 4491.

In addition, the scope of the method has been extended to include powders containing carbon.

If carbon is present in the powder, some metal oxides which may otherwise have been reduced by hydrogen are instead reduced by carbon, producing carbon monoxide or carbon dioxide. These products are not measured by the titration with Karl Fischer reagent which is used to determine the amount of water produced. Consequently a lower result will be obtained for the hydrogen-reducible oxygen content.

This interference is eliminated by passing the gases emerging from the reduction furnace over a catalyst which converts the CO and CO₂ formed into methane and water, in accordance with the following equations:



The conversion reaction is carried out at 380 °C over a nickel catalyst.

NOTE — Certain oxides may be partially reduced by carbon which otherwise would not be reduced by hydrogen. In such cases the interpretation of results should be made with great care [see ISO 4491-1:1989, subclause 4.1.2 d)].

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Metallic powders — Determination of oxygen content by reduction methods —

Part 3: Hydrogen-reducible oxygen

1 Scope

This part of ISO 4491 specifies a method for the determination of the hydrogen-reducible oxygen content of metallic powders containing 0,05 % (m/m) to 3 % (m/m) oxygen.

The method is applicable to unalloyed, partially alloyed or completely alloyed metal powders and also to mixtures of carbides and binder metal. It is not applicable to powders containing lubricants or organic binders.

The method may be extended to powders containing carbon by the use of a special catalytic device.

This part of ISO 4491 shall be read in conjunction with ISO 760 and ISO 4491-1.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 4491. At the time of publication, the editions indicated were 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 editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 760:1978, *Determination of water — Karl Fischer method (General method)*.

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

3 Principle

Pre-treatment of a test portion by drying at low temperature (170 °C) in dry nitrogen or argon.

Reduction in a stream of pure dry hydrogen at a given temperature. Absorption in methanol of the water formed by reaction of oxides with hydrogen. Titration with Karl Fischer reagent, the end-point being determined either visually by the colour change or electrometrically with two electrodes (deadstop end-point).

For powders containing carbon, conversion of the carbon monoxide and carbon dioxide formed to methane and water at 380 °C by means of a nickel catalyst.