International Standard

ISO 4491-3

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

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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 document 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 powders (including powders for hardmetals)*, in collaboration with the European Committee for Standardization (CEN), in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 4491-3:1997), of which it constitutes a minor revision.

The main changes are as follows:

— normative references in Clause 2 have been updated;

— editorial revisions and alignment with current ISO drafting directives.

A list of all parts in the ISO 4491 series can be found on the ISO website.

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.

Introduction

This document is intended to be used for the determination of hydrogen reducible oxygen content of metallic powders, including powders containing carbon.

If carbon is present in the powder, some metal oxides which can 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 $C0_2$ formed into methane and water, in accordance with the following equations:

$$CO + 3H_2 \rightarrow CH_4 + H_2O$$

$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O$$

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

Certain oxides can 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 4491-1:2023, 5.1.2 d).

Metallic powders — Determination of oxygen content by reduction methods —

Part 3: Hydrogen-reducible oxygen

1 Scope

This document specifies a method for the determination of the hydrogen-reducible oxygen content of metallic powders containing mass percentage of 0,05 % to 3 % oxygen.

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

This document can be extended to powders containing carbon by the use of a special catalytic device. This document is intended to be used in conjunction with ISO 760 and ISO 4491-1.

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 760, *Determination of water — Karl Fischer method (General method)*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology 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 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). ISO 760 and ISO 4491-1 shall be used in conjunction with this document.

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