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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 5158.4-2011

**Metallic powders -- Determination of oxygen content by
reduction methods -- Part 4: Total oxygen by
reduction-extraction
(English Translation)**

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Table of Contents

Foreword...	3
1 Scope...	5
2 Normative references...	5
3 Principle...	5
4 Apparatus and materials...	7
5 Test portion...	7
6 Procedure...	8
7 Expression of results...	9
8 Test report...	9

Foreword

GB/T 5158 "Metallic powders - Determination of oxygen content by reduction methods" is divided into four parts:

- 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.

This Part is Part 4 of GB/T 5158.

This Part is identical to ISO 4491-4:1989 "Metallic powders - Determination of oxygen content by reduction methods - Part 4: Total oxygen by reduction-extraction".

For ease of use, the following editorial modifications have been made to this Part:

- a) Change the word "this International Standard" to "this Part";
- b) Use the decimal separator "." instead of the comma "," as the decimal separator;
- c) Delete the foreword of the international standard.

This Part replaces GB/T 5158.4-2001 "Determination of the total oxygen content of the metal powder reduction-extraction".

Compared with GB/T 5158.4-2001, the main changes in this Part are as follows:

- Delete the ISO foreword;
- Add normative references;
- Revise the parts of the previous standard that differ from ISO 4491-4:1989 TO be consistent.

Annex A of this Part is an informative annex.

This Part was proposed by China Nonferrous Metals Industry Association.

This Part shall be under the jurisdiction of National Nonferrous Metals Standardization Technical Committee.

This Part was drafted by Central South University Powder Metallurgy Research Institute, Shandong Lanyue Technology Co., Ltd., and China Nonferrous Metals
Metallic powders - Determination of oxygen content by
reduction methods - Part 4: Total oxygen by reduction-
extraction

1 Scope

This Part of GB/T 5158 specifies a method for the determination of the total oxygen content of metallic powders in concentrations up to 2 % (mass fraction) 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.

This Part is applicable to all powders of metals, alloys, carbides (hard alloys) 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 shall be used in conjunction with GB/T 5158.1.

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the editions with the dates indicated are applicable to this document. For the undated references, the latest edition (including all the amendments) are applicable to this document.

GB/T 5158.1 Metallic powders - Determination of oxygen content by reduction methods - Part 1: General guidelines (ISO 4491-4:1989, IDT)

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

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

6.1 General

It is not possible to specify the conditions of oxygen determination for each of the various metals, alloys and carbides (hard alloys) 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 optimal 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 (condition) tests on the same sample, increasing the reducing action (i.e. by increasing the temperature or time of reaction) until the measured oxygen content reaches a maximum, constant value. Other test conditions (or 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 verify or calibrate 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.

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