

International Standard



4493

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

● Metallic powders — Determination of hydrogen reducible oxygen content

Poudres métalliques — Détermination de la teneur en oxygène réductible par l'hydrogène

First edition — 1981-08-15

UDC 621.762 : 669-492.2 : 543

Ref. No. ISO 4493-1981 (E)

Descriptors : pulverulent products, metallic powder, tests, determination of content, hydrogen, oxygen, Karl Fischer reagent

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4493 was developed by Technical Committee ISO/TC 119, *Powder metallurgy*, and was circulated to the member bodies in November 1979.

It has been approved by the member bodies of the following countries :

Austria	Italy	Spain
Bulgaria	Japan	Sweden
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Czechoslovakia	Poland	USA
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India	South Africa, Rep. of	

The member body of the following country expressed disapproval of the document on technical grounds :

China

Metallic powders — Determination of hydrogen reducible oxygen content

1 Scope and field of application

This International Standard specifies a method for the determination of the hydrogen reducible oxygen content of metallic powders free of admixed pressing lubricant.

The method is applicable to the metal powders listed in table 2 and for the determination of oxygen contents between 0,05 and 3 % (*m/m*). It is not applicable to the metal powders in the upper part of table 2 when they contain carbon.¹⁾

2 Reference

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

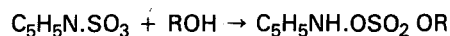
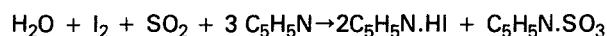
3 Principle

3.1 Heating a test portion in a stream of pure and dry hydrogen. Absorption of the water, formed by reaction of the metal oxide with the hydrogen, in methanol, and titration with Karl Fischer reagent. Determination of the end-point electrometrically, using the dead-stop technique.

3.2 If the powder contains moisture, the test portion is heated to 150 °C in dry nitrogen before the reduction in hydrogen is carried out; otherwise, the moisture would react with the Karl Fischer reagent.

3.3 As the apparatus may not be perfectly gas tight, and as the dried hydrogen and nitrogen may still contain very small amounts of water and oxygen, a blank test is carried out.

3.4 The determination of water by the Karl Fischer method is based on the fact that iodine and sulphur dioxide, in a pyridine/methanol or pyridine/2-methoxyethanol mixture, react with water according to the equations



The Karl Fischer reagent consists of iodine, sulphur dioxide and pyridine dissolved in methanol or 2-methoxyethanol (ethylene glycol monomethyl ether). The end-point is reached when all the water has reacted and free iodine appears in the solution, i.e. when the pale yellow solution turns to orange red (visual end-point).

ISO 760 describes in detail the preparation and standardization of the Karl Fischer reagent and the different types of equipment. The procedure given in this International Standard is only sufficient to ensure that the method can be understood.

4 Reagents

4.1 Methanol, water free.

4.2 Karl Fischer reagent, equivalent to 3 to 5 mg of oxygen per millilitre.

WARNING — Karl Fischer reagent contains four toxic compounds: iodine, sulphur dioxide, pyridine and methanol. It is important to avoid direct contact and especially inhalation. Following accidental spillage, rinse with plenty of water.

1) If carbon is present, some metal oxides may be reduced by the carbon, producing carbon monoxide or carbon dioxide. These oxides may otherwise have been reduced by the hydrogen. If this carbon reduction occurs, a lower result will be obtained for the hydrogen reducible oxygen content. In such cases the method may be used possibly with some modifications by agreement between the parties concerned. One solution is to convert the carbon monoxide or dioxide to methane and water by passing the gases over activated nickel at a temperature of 350 °C.

The titre of the Karl Fischer reagent may be determined by either of the following methods :

- a) addition of 20 to 30 mg of water, weighed to the nearest 0,1 mg;
- b) addition of 100 to 300 mg, weighed to the nearest 0,1 mg, of sodium tartrate dihydrate [certified material containing theoretically 15,66 % (m/m) of water, corresponding to 13,92 % (m/m) of oxygen], ground to fine powder and dried at 155 ± 5 °C to constant mass.

The titre shall be calculated to the nearest 0,01 mg of oxygen per millilitre.

NOTE — See ISO 760 for methods of standardization.

4.3 Hydrogen, having a maximum oxygen content of 0,005 % and a dew point not exceeding - 45 °C.

4.4 Nitrogen, having a maximum oxygen content of 0,005 % and a dew point not exceeding - 45 °C. Argon may be substituted for nitrogen.

4.5 Desiccant : granular anhydrous aluminium sodium silicate, activated silica gel, or magnesium perchlorate.

5 Apparatus

NOTE — The arrangement of the apparatus is shown schematically in figure 1.

Ordinary laboratory apparatus and

5.1 Source of hydrogen (A), fitted with a pressure regulating valve, a flow control valve, and a flow meter.

5.2 Catalytic purifier (B), for the hydrogen.

5.3 Source of nitrogen (C), fitted with a pressure regulating valve, a flow control valve, and a flow meter.

5.4 Gas selecting valve (D).

5.5 Gas drying unit (E), containing one of the desiccants specified in 4.5.

5.6 Reduction tube (F), gas tight, made of quartz, closed at one end, having a diameter of 27 to 30 mm and a length of about 400 mm; it also comprises two quartz tubes of diameter 5 to 6 mm and lengths 60 to 80 mm and 200 to 240 mm, respectively, arranged as shown in figure 2.

5.7 Two furnaces (G), one for drying the test portion and one for reduction, with temperature control systems capable of maintaining the temperature in the part of the tube containing the boat within the specified temperature tolerances.

If available, one furnace combining both these functions may be used.

5.8 Boats (H), preferably high alumina ceramic, with a polished surface and of a suitable size such that no more than half is filled by the test portion. These boats shall be conditioned in hydrogen at 900 to 1 100 °C for at least 1 h and stored in a desiccator before use.

5.9 Automatic burette (J), of capacity 25 ml and with a fine point, graduated in 0,05 ml divisions, and protected from atmospheric moisture by a guard tube filled with one of the desiccants specified in 4.5.

5.10 Titration flask (K), comprising a 300 ml conical flask containing platinum electrodes and a magnetic stirrer.

5.11 End-point detector (L).

A circuit diagram of an electrometric end-point detector, utilizing the dead-stop technique, is shown in figure 3.

NOTE — Items (J), (K) and (L) are given as examples with which satisfactory results have been obtained. Modifications to these or to any commercially available Karl Fischer titration equipment may be made, provided that the operating requirements are fulfilled (see ISO 760).

5.12 Stop-watch, for determination of the titration end-point.

5.13 Timer.

5.14 Blower (for example a hair-dryer), for cooling the tube.

6 Sampling

6.1 The powder shall be tested in the as-received condition.

6.2 The analysis shall be carried out on two test portions.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,000 1 g, a mass of test portion, according to the expected hydrogen reducible oxygen content, as indicated in table 1.

Table 1

Expected hydrogen reducible oxygen content % (m/m)	Mass of test portion g
from 0,05 to 0,5	5
from 0,5 to 2,0	2
from 2,0 to 3,0	



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