INTERNATIONAL STANDARD

ISO 3908

Third edition
2009-10-01

Hardmetals — Determination of insoluble (free) carbon — Gravimetric method

Métaux-durs — Dosage du carbone insoluble (libre) — Méthode gravimétrique

Reference number
ISO 3908:2009(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

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

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 3908 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 4, *Sampling and testing methods for hardmetals*.

This third edition cancels and replaces the second edition (ISO 3908:1985), which has been technically revised.

Hardmetals — Determination of insoluble (free) carbon — Gravimetric method

1 Scope

This International Standard specifies a gravimetric method for the determination of the mass fraction of insoluble (free) carbon in carbides and hardmetals.

This method is applicable to

— carbides of hafnium, molybdenum, niobium, tantalum, titanium, vanadium, tungsten and zirconium,

— mixtures of these carbides and binder metals, free of lubricant, and

— all grades of presintered or sintered hardmetals, produced from these carbides,

having a mass fraction of insoluble carbon between 0,02 % and 0,5 %.

2 Normative references

The following referenced documents are indispensable for the application 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 3907:2009, *Hardmetals — Determination of total carbon — Gravimetric method*

3 Principle

Decomposition of the carbides and determination of the insoluble carbon by a gravimetric method.

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

4.1 Nitric acid, $\rho = 1{,}20$ g/ml.

Add 2 000 ml of nitric acid, $\rho = 1{,}42$ g/ml, to 3 000 ml of water.

4.2 Hydrofluoric acid, $\rho = 1{,}12$ g/ml.

5 Apparatus

Ordinary laboratory apparatus and the following.

5.1 Apparatus, as specified in ISO 3907.

5.2 Platinum dish, of capacity 200 ml.

5.3 Filter device: ceramic filter device or bed of suitable refractory fibrous or powder material in a Gooch crucible.

NOTE If necessary, pretreat the refractory material at 800 °C to 1 000 °C under strongly oxidizing conditions for a minimum of 3 h. If pretreated, store it in a desiccator.

5.4 Vacuum filtration assembly.

6 Sampling

6.1 The sample shall be crushed to a powder in a mortar made of a material which does not alter the sample composition. The powder shall pass through a 180 µm sieve.

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

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,01 g, approximately 2,5 g of the test sample.

7.2 Attack

Transfer the test portion (7.1) into the platinum dish (5.2). Add 75 ml of the nitric acid (4.1) and place the dish on a steam bath for 5 min. Add, drop by drop, 10 ml of the hydrofluoric acid (4.2), and leave the dish on the steam bath for about 1 h until complete dissolution is obtained.

Cool the solution to ambient temperature.

CAUTION — Hydrofluoric and nitric acids are very dangerous chemicals. Any contact with these acids or inhalation of their vapours shall be avoided. All operations with these acids shall be carried out in a fume-cupboard with good ventilation.

7.3 Preparation of the Gooch crucible

Insert the ceramic filter device (5.3) into the crucible.

If a refractory material is used, fill the crucible to a depth of approximately 8 mm to 10 mm and press it down so that the residue will be retained on the refractory material and the time of filtering will not be too slow.

7.4 Filtering

Before filtering, add a limited quantity of water to avoid the precipitation of tungstic acid. Filter the contents of the dish (see 7.2) through the filter device (5.3). Then rinse the dish twice with small volumes of water. Be sure that all particles of carbon are transferred to the filter device. Rinse the dish again with water at least twice and then wash the filter device free from acid with hot water (about 500 ml is usually needed).

Remove the wet filter device from the Gooch crucible and transfer the wet filter device into a boat (see ISO 3907:2009, 4.1.5). Dry it at 110 °C.

7.5 Blank test

Carry out two blank tests for each series of determinations.

Prepare the Gooch crucible in accordance with 7.3.

Filter through the filter device (5.3) a mixture of 75 ml of the nitric acid (4.1) and 10 ml of the hydrofluoric acid (4.2) and proceed in accordance with 7.4.

7.6 Determination

Burn the filter device (5.3) in a stream of oxygen in accordance with ISO 3907. Use a furnace with an inner temperature of the tube of approximately 1 200 °C.

8 Expression of results

8.1 Calculation

The mass fraction of carbon , expressed as a percentage, is given by the formula

$$27{,}29 \times \frac{m_2 - m_1}{m_0}$$

where

m_0 is the mass, in grams (g), of the test portion;

m_1 is the mass, in grams (g), of carbon dioxide obtained from the blank test;

m_2 is the mass, in grams (g), of carbon dioxide obtained from the combustion of the test portion;

27,29 is the carbon dioxide to carbon conversion factor, multiplied by 100.

8.2 Tolerances

The deviation between two or three independent determinations shall not exceed the values shown in Table 1.

Table 1 — Tolerances

Total carbon mass fraction %	Range for two determinations %	Range for three determinations %
from 0,02 to 0,1	0,02	0,03
over 0,1 to 0,5	0,04	0,05

8.3 Final result

Report the arithmetical mean of acceptable determinations rounded to the nearest 0,01 % (mass fraction).

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the identification of the test sample;

c) the result obtained;

d) all operations not specified in this International Standard, or regarded as optional;

e) details of any occurrence which may have affected the result.

ICS 77.160

Price based on 4 pages