International Standard 3908

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

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

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

1 Scope

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

2 Field of application

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,

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

having an insoluble carbon content between 0,02 and 0,5 % (m/m).

3 Reference

ISO 3907, *Hardmetals — Determination of total carbon content — Gravimetric method.*

4 Principle

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

5 Reagents

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

5.1 Nitric acid, ϱ 1,20 g/ml.

Add 2 000 ml of nitric acid, ϱ 1,42 g/ml, to 3 000 ml of water.

5.2 Hydrofluoric acid, ϱ 1,12 g/ml.

6 Apparatus

Ordinary laboratory apparatus and

6.1 Apparatus specified in ISO 3907.

6.2 Platinum dish, of capacity 200 ml.

6.3 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 to 1 000 °C under strongly oxidizing conditions for a minimum of 3 h. Store it in a desiccator, if pretreated.

6.4 Vacuum filtration assembly.

7 Sampling

7.1 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 a 180 µm sieve.

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

8 Procedure

8.1 Test portion

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

8.2 Attack

Transfer the test portion (8.1) into the platinum dish (6.2). Add 75 ml of the nitric acid (5.1) and place the dish on a steam bath for 5 min. Add, drop by drop, 10 ml of the hydrofluoric acid (5.2), 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 must be avoided. All operations with these acids shall be carried out in a fume-cupboard with good ventilation.

8.3 Preparation of the Gooch crucible

Insert the ceramic filter (6.3) into the crucible.

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

8.4 Filtering

Before filtering add a limited quantity of water to avoid the precipitation of tungstic acid. Filter the contents of the dish (see 8.2) through the filter device (6.3). Rinse the dish twice with small volumes of water. Be sure that all particles of carbon are transferred to the filtering device. Rinse the dish again with water at least twice and thereafter 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 sub-clause 5.1 of ISO 3907). Dry it at 110 °C.

8.5 Blank test

Carry out two blank tests with each series of determinations.

Prepare the Gooch crucible according to 8.3.

Filter through the filter device (6.3) a mixture of 75 ml of the nitric acid (5.1) and 10 ml of the hydrofluoric acid (5.2) and proceed according to 8.4.

8.6 Determination

Burn the filter device (6.3) 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.

9 Expression of results

9.1 Calculation

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

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

where

m_0 is the mass, in grams, of the test portion;

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

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

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

9.2 Tolerances

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

Total carbon content % (m/m)	Range for two determinations % (m/m)	Range for three determinations % (m/m)
from 0,02 to 0,1	0,02	0,03
over 0,1 to 0,5	0,04	0,05

9.3 Final result

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

10 Test report

The test report shall include the following information :

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.