INTERNATIONAL STANDARD ISO 3908

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

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

Métaux durs — Détermination du carbone insoluble (libre) — Méthode gravimétrique

First edition — 1976-09-30

UDC 621.762 : 546.26 : 543.21

Ref. No. ISO 3908-1976 (E)

Descriptors : powder metallurgy, sintered products, hardmetals, carbides, chemical analysis, determination of content, carbon, gravimetric analysis.

Price based on 2 pages

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 3908 was drawn up by Technical Committee ISO/TC 119, *Powder metallurgical materials and products*, and was circulated to the Member Bodies in August 1975.

It has been approved by the Member Bodies of the following countries :

Australia	Ireland	Spain
Austria	Italy	Sweden
Brazil	Mexico	Turkey
Canada	Poland	United Kingdom
Czechoslovakia	Portugal	U.S.A.
France	Romania	U.S.S.R.
Germany	South Africa, Rep. of	Yugoslavia

No Member Body expressed disapproval of the document.

Printed in Switzerland

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

1 SCOPE

This International Standard specifies a gravimetric method for the 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 — 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.

5.3 Asbestos, pretreated.

Pretreat fine-fibrous asbestos at 800 to 1 000 °C under strongly oxidizing conditions for a minimum of 3 h. Store it in a desiccator.

CAUTION — Asbestos can be a danger to health and should not be inhaled. All operations with asbestos shall be carried out with care in well-ventilated areas to avoid scattering of the material.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Apparatus specified in ISO 3907.

6.2 Platinum dish, 200 ml.

6.3 Gooch crucible, porcelain or platinum.

NOTE — A ceramic filter may be used instead of the Gooch crucible and asbestos pad.

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 0,18 mm 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 into a platinum dish. 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

Weigh a sufficient quantity of the pretreated asbestos (5.3) to 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 asbestos pad and at the same time filtering will not be too slow.

Moisten the asbestos with water by vacuum filtration. Compact the asbestos pad. Filter 20 to 30 ml of distilled water and check that the asbestos is evenly distributed.

8.4 Filtering

Filter the contents of the dish (see 8.2) through the asbestos pad. Rinse the dish twice with small volumes of distilled water. Use approximately 0,5 g of the pretreated asbestos for removing any insoluble carbon adhering to the dish and add this asbestos to the crucible. Rinse the dish with water at least twice and thereafter wash the pad free from acid with hot distilled water (100 to 200 ml is usually needed).

Transfer the wet asbestos into a boat and 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 asbestos pad 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 asbestos pads obtained (8.4 and 8.5) 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

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

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

Insoluble carbon content %	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

9.3 Final result

Report the arithmetical mean of acceptable determinations rounded to the nearest 0,01 %.

10 TEST REPORT

The test report shall include the following information :

a) reference to this International Standard;

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

c) the result obtained;

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

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