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

## 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.

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.

International Standard ISO 3908 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*.

ISO 3908 was first published in 1976. This second edition cancels and replaces the first edition, of which it constitutes a technical revision.

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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, $\rho$ 1,20 g/ml.

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

### 5.2 Hydrofluoric acid, $\rho$ 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  $\mu$ m 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.**