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Hårdmetall – Bestämning av olösligt (fritt) kolinnehåll – Gravimetrisk metod (ISO 3908:2009)

Hardmetals – Determination of insoluble (free) carbon – Gravimetric method (ISO 3908:2009)

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Denna standard ersätter SS-EN 23908, utgåva 1.

The European Standard EN ISO 3908:2009 has the status of a Swedish Standard. This document contains the official English version of EN ISO 3908:2009.

This standard supersedes the Swedish Standard SS-EN 23908, edition 1.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 3908

October 2009

ICS 77.160

Supersedes EN 23908:1993

English Version

Hardmetals - Determination of insoluble (free) carbon - Gravimetric method (ISO 3908:2009)

Métaux-durs - Dosage du carbone insoluble (libre) - Méthode gravimétrique (ISO 3908:2009)

Hartmetalle - Bestimmung des unlöslichen (freien) Kohlenstoffgehaltes - Gravimetrisches Verfahren (ISO 3908:2009)

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Foreword

This document (EN ISO 3908:2009) has been prepared by Technical Committee ISO/TC 119 "Powder metallurgy".

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2010, and conflicting national standards shall be withdrawn at the latest by April 2010.

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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.

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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.