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Hardmetals — Determination of total carbon — Gravimetric method

Métaux-durs — Dosage du carbone total — Méthode gravimétrique

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Foreword

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ISO 3907 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 4, *Sampling and testing methods for hardmetal*.

This third edition cancels and replaces the second edition (ISO 3907:1985), which has been technically revised.

Hardmetals — Determination of total carbon — Gravimetric method

1 Scope

This International Standard specifies a gravimetric method for the determination of the mass fraction of total carbon in carbides and hardmetals.

This method is applicable to

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

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

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

having a mass fraction of total carbon exceeding 4 %.

2 Principle

Oxidation of carbon to carbon dioxide at a high temperature in a stream of pure oxygen, with the addition of a flux, if necessary.

Absorption of the carbon dioxide, carried by oxygen, by Ascarite[1)] in a tared bulb. Determination of the increase in mass of the Ascarite[1)] which corresponds to the quantity of carbon dioxide formed.

$$C + O_2 \rightarrow CO_2$$

$$2\,NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O$$

3 Reagents

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

3.1 Oxygen, with a limitation of carbon-containing impurities of $\leqslant$ 0,6 ml of carbon per cubic metre of oxygen.

1) Ascarite is the trade name of a product supplied by Arthur H. Thomas Co. This information is given for the convenience of users of the International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

3.2 Magnesium perchlorate, anhydrous.

CAUTION — To prevent the possibility of explosion, contact of this reagent with organic materials must be avoided, especially when discarding it.

3.3 Flux, for example tin metal, copper metal or oxide, iron metal.

3.4 Ascarite[1)].

4 Apparatus

Ordinary laboratory apparatus and the following.

4.1 Apparatus, consisting of an electric furnace with a combustion tube, a purification train and a system to absorb carbon dioxide. If it is necessary to obtain oxygen of adequate purity, an oxygen purification train may also be used.

Successive parts of the apparatus shall be joined together with connecting tubes forming an airtight seal.

The apparatus is shown schematically in Figure 1.

4.1.1 Source of oxygen (3.1), with a pressure-regulating valve.

4.1.2 Flow meter.

4.1.3 Electric furnace, capable of operation at up to 1 350 °C, with a suitable device for temperature control.

4.1.4 Combustion tube, made of a non-porous refractory material. The internal diameter of the tube should be 18 mm to 30 mm and its length should be at least 650 mm, so that the ends of the tube do not reach a temperature higher than 60 °C during the operation.

4.1.5 Boat, made of a refractory material, pretreated in an oxygen stream at the test temperature for 10 min or alternatively at 800 °C to 1 000 °C for 1 h.

The boat shall be of suitable dimensions, for example of length 80 mm to 100 mm, width 12 mm to 14 mm and depth 8 mm to 9 mm.

The pretreated boats shall be kept in a desiccator. The ground surfaces of the desiccator and its lid shall not be greased.

4.1.6 Plug of silica wool.

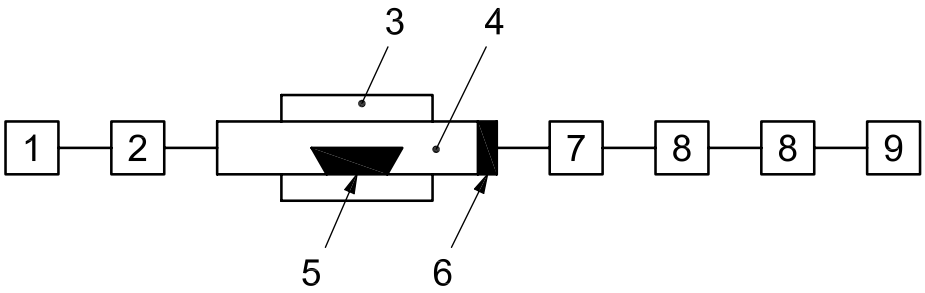
4.1.7 Drying bulb, containing anhydrous magnesium perchlorate (3.2).

4.1.8 Absorption bulbs, containing Ascarite[1)] (3.4) and a small amount of anhydrous magnesium perchlorate (3.2).

An example of an absorption bulb is shown in Figure 2.

4.1.9 Additional absorption bulb, facing the opposite way to the absorption bulb in 4.1.8 (see Figure 1, item reference 8) to avoid introduction of carbon dioxide and moisture from the air.

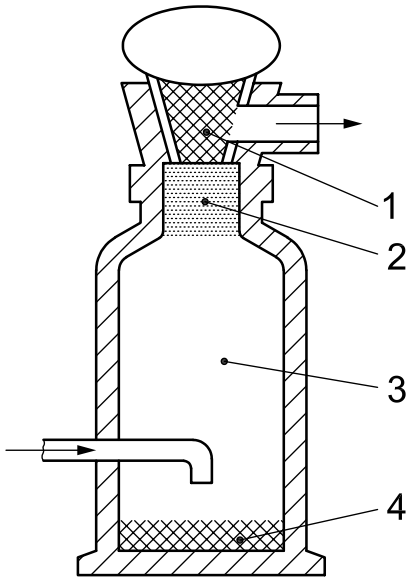
4.2 Hook, made from heat-resisting metal wire with a mass fraction of carbon less than 0,05 %. Its diameter should be approximately 3 mm and its length should be 500 mm to 600 mm.



Key

1 source of oxygen
2 flow meter
3 electric furnace
4 combustion tube
5 boat
6 plug of silica wool
7 drying bulb
8 absorption bulbs
9 additional absorption bulb

Figure 1 — Apparatus



Key

1 glass wool in stopper
2 anhydrous magnesium perchlorate (3.2)
3 Ascarite[1)] (3.4), not too compressed
4 bottom layer of glass wool to protect inside of tube

Figure 2 — Absorption bulb

5 Sampling

5.1 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.

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

6 Procedure

6.1 General

Check the temperature in the combustion zone (1 200 °C to 1 350 °C, and not less than 1 300 °C if chromium carbide is present), the gastightness of the apparatus and the efficiency of the oxygen purification. Pass oxygen through the apparatus for 10 min to 15 min at a rate of 300 cm^3/min to 500 cm^3/min depending on the diameter of the tube used. Then disconnect the absorption bulbs (see Figure 1, item reference 8), weigh them at ambient temperature and place them in position again.

6.2 Test portion

The mass of the test portion (m_0) shall be such that it contains approximately 0,03 g of carbon, and shall be determined to the nearest 0,000 1 g.

If necessary, add 0,2 g to 1 g of the flux (3.3) to the test portion.

6.3 Blank test

Carry out the blank test by combustion (proceed as described in 6.4 and 6.5) in the presence of the quantity of flux used in the analysis, and carefully determine the increase in mass (m_1) of the absorption bulbs.

6.4 Combustion

Open the combustion tube at the oxygen inlet end and, using the hook (4.2), place the boat (see Figure 1, item reference 5) containing the test portion (6.2) in the centre of the heated zone of the tube. Quickly close the tube and immediately pass a stream of oxygen at a rate of 300 cm^3/min to 500 cm^3/min, depending on the diameter of the tube used. Continue to pass oxygen for 10 min to 20 min, so that the carbon dioxide is completely removed from the combustion tube and the purifying bulb.

6.5 Determination

Close the tap of the absorption bulbs (see Figure 1, item reference 8) and immediately remove the bulbs from the apparatus. After 5 min, weigh the bulbs to the nearest 0,000 1 g. It is recommended that the fused mass in the boat be visually examined to verify that combustion has been completed. The increase in mass represents the carbon dioxide absorbed (m_2).

7 Expression of results

7.1 Calculation

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

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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 the carbon dioxide obtained from the combustion of the test portion;

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

7.2 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 4 to 10	0,05	0,06
over 10	0,07	0,08

7.3 Final result

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

8 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.

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