



# Standard Test Method for Determination of Metallic Constituents of Tungsten Alloys and Tungsten Hardmetals by X-Ray Fluorescence Spectrometry<sup>1</sup>

This standard is issued under the fixed designation B 890; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method describes a procedure for the determination of the concentration, generally reported as mass percent, of the metallic constituents of tungsten-based alloys and hardmetals utilizing wavelength dispersive X-ray fluorescence spectrometry (XRF). This test method incorporates the preparation of standards using reagent grade metallic oxides, lithium-borate compounds, and fusion techniques. This test method details techniques for preparing representative specimens of both powder and sintered tungsten-based material. This test method is accurate for a wide range of compositions, and can be used for acceptance of material to grade specifications.

1.2 This test method is applicable to mixtures of tungsten or tungsten carbide with additions of refractory metal carbides and binder metals. Table 1 lists the most common elemental constituents and their concentration range. Note that many of these occur as metallic carbides.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials<sup>2</sup>

E 1361 Guide for Correction of Interelement Effects in X-Ray Spectrometric Analysis<sup>3</sup>

### 2.2 ISO Standard:

ISO-4503(E) Hardmetal—Determination of contents of metallic elements by X-ray fluorescence - Fusion method<sup>4</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.06 on Cemented Carbides.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 03.05.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.06.

<sup>4</sup> Available from American National Standards Institute, 11 West 42nd St., 13th Floor, New York, NY 10036.

**TABLE 1 Elemental Constituents and Concentration Range**

Element	Concentration, Mass % (minimum - maximum)
Chromium (Cr)	0.05 - 5.0
Cobalt (Co)	0.05 - 40
Hafnium (Hf)	0.05 - 2.0
Iron (Fe)	0.05 - 2.0
Molybdenum (Fe)	0.05 - 5.0
Nickel (Ni)	0.05 - 30
Niobium (Nb)	0.05 - 15
Tantalum (Ta)	0.05 - 30
Titanium (Ti)	0.05 - 30
Vanadium (V)	0.05 - 2.0

2.3 *Handbook of Chemistry and Physics*,<sup>5</sup> 67th ed

## 3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E 135.

## 4. Summary of Test Method

4.1 A suite of standards which closely match the chemical content of the material to be analyzed are prepared using reagent grade metallic oxides. Test samples are oxidized in a high-temperature furnace open to air. Fused glass specimens are prepared for these standards and for the test samples to be analyzed. These specimens of oxidized tungsten or tungsten carbide alloys are irradiated with an energetic primary X-ray beam. The intensity of the resultant secondary X-rays, characteristic in energy, for each elemental constituent is measured by means of a suitable detector or combination of detectors after diffraction by a Bragg spectrometer. The concentration of each constituent element is calculated by comparison with standard samples which closely match the chemical content of the analyzed material. The calculation may be manual, incorporate a calibration curve, or be performed by a computer program which incorporates correction routines for X-ray absorption and enhancement effects (see Guide E 1361).

## 5. Significance and Use

5.1 This test method allows the determination of the chemical composition of powdered and sintered tungsten-based

<sup>5</sup> CRC Press, Boca Raton, FL, 1987.

hardmetals. This test method is not applicable to material which will not oxidize readily at high temperatures in air, such as tungsten/copper or tungsten/silver alloys.

5.2 This test method specified lithium-borate compounds for the glass fusion material. However, numerous other choices are available. These include other lithium-borate compounds, sodium carbonate and borate mixtures, and others. The methodology specified here is still applicable as long as the same fusion mixture is used for both standards and specimens.

## 6. Interferences

6.1 Errors in XRF-determined compositional values may be encountered due to X-ray enhancement and absorption effects dependent on the elements present and the X-ray line being measured for a specific element. This effect can be reduced by determination of correction factors using appropriate standards and interelement correction routines, manual or computerized.

6.2 Accuracy and precision of the analytical results obtained from molybdenum-containing samples may be rendered unreliable due to the sublimation and evaporation of molybdenum from the material during the oxidation step in specimen preparation.

6.3 Incorporation of the fusion method of specimen preparation will:

6.3.1 Reduce the deleterious influence of particle size effects experienced when analyzing powder materials by varying particle size.

6.3.2 Reduce inhomogenities within a sample.

6.3.3 Improve penetration of X rays.

## 7. Apparatus

7.1 *X-Ray Fluorescence Wavelength Spectrometer*

7.2 *Fluxer*—An automated high-temperature mixing device capable of melting, mixing, and pouring a molten liquid specimen into a proper casting dish, is highly preferred.

7.3 *Analytical Balance*, readability of 0.00001 g.

7.4 *Toploading Balance*, readability of 0.001 g.

7.5 *Ordinary Laboratory Apparatus*.

7.6 *One Pt - 5 % Au Casting Dist* (minimum)

7.7 *One Pt - 5 % Au Crucible* (minimum)

7.8 *Platinum Tipped Tongs*.

7.9 *Weighing Paper*.

7.10 *Chemical Spoon and Scoopula*.

7.11 *Ceramic Combustion Boat*.

7.12 *High Temperature Tube or Muffle Furnace*, open to the atmosphere.

7.13 *Self-adhering Stickers*, ¾ by 1 in.

7.14 *High-Temperature marking pen*

7.15 *Ceramic Mortar and Pestle*.

7.16 *Miniature Mixed*, optional.

## 8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specification of the Committee on Analytical Reagents of the American Chemical Society where

such specifications are available.<sup>6</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Di-lithiumtetraborate* ( $\text{Li}_2\text{B}_4\text{O}_7$ ): *Lithiummetaborate* ( $\text{LiBO}_2$ ), 66 + 34.

8.3 *Lithium Bromide* ( $\text{LiBr}$ ).

8.4 *Metallic Oxide Powder*, highest oxidation state for elements of interest; that is  $\text{Co}_3\text{O}_4$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ ,  $\text{HfO}_2$ ,  $\text{MoO}_3$ ,  $\text{Nb}_2\text{O}_5$ ,  $\text{NiO}$ ,  $\text{Ta}_2\text{O}_5$ ,  $\text{TiO}_2$ ,  $\text{V}_2\text{O}_5$ , and  $\text{WO}_3$ .

**Warning**—Several of the metallic oxides used in this test method are highly toxic and possibly carcinogenic, such as  $\text{Cr}_2\text{O}_3$ ,  $\text{NiO}$ , or  $\text{V}_2\text{O}_5$ . Extreme care should be used at all times when handling this material (especially  $\text{V}_2\text{O}_5$ ). All mixing of standards should be performed in a fume hood. All of the lithium compounds are water-soluble and therefore able to be absorbed into the body by inhalation and possibly by absorption through the skin. This material should be weighed in a fume hood.

8.5 *Nitric Acid* ( $\text{HNO}_3$ ).

## 9. Specimen Preparation

9.1 Prepare specimens of the material to be analyzed by oxidizing, weighing, and fusing starting powders, chips, or crushed sintered hard metal samples.

9.2 Place 3 to 5 g of powdered specimen in a labeled ceramic combustion boat. If a sintered sample is to be analyzed, then the sample must be crushed or pulverized into small pieces or chips must be produced by machining prior to placement in the combustion boat.

9.3 Oxidize the specimen in the heat zone of a high-temperature tube or muffle furnace open to the atmosphere at  $825 \pm 25^\circ\text{C}$ . All specimens must be oxidized.

9.4 When the specimen has been completely oxidized (4 to 6 h), remove from the furnace and allow to cool.

NOTE 1—Complete oxidation of a sintered magnetic tungsten hard metal sample can be checked by testing the cool oxidized chips with a magnet. If any of the chips are still magnetic, recrush the sample and place back in the furnace for further oxidation.

9.5 Pour the specimen onto a clean sheet of paper or into a clean mortar and gently crush with a pestle.

9.6 Transfer the specimen to a labeled specimen vial.

9.7 In a fume hood, weigh out  $15.000 \pm 0.001$  g of the di-lithium tetraborate: lithiummetaborate mixture and  $0.200 \pm 0.001$  g of  $\text{LiBr}$  and transfer to a clean sample vial. This mixture will be referred to as the “fusion mixture.” Seal and store until needed.

NOTE 2—Other fusion materials can be used. See 5.2.

9.8 In a fume hood, transfer the fusion mixture to a platinum crucible immediately prior to weighing of the oxidized sample material.

<sup>6</sup> *Reagent Chemicals, American Chemical Society Specification*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.