International Standard ISO 7627/4

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

Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 4: Determination of molybdenum, titanium and vanadium in contents from 0,01 to 0,5 % (m/m)

Métaux-durs — Analyse chimique par spectrométrie d'absorption atomique dans la flamme — Partie 4: Dosage du molybdène, du titane et du vanadium à des teneurs comprises entre 0,01 *et* 0,5 % (m/m)

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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.

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It has been approved by the member bodies of the following countries:

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No member body expressed disapproval of the document.

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Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 4: Determination of molybdenum, titanium and vanadium in contents from 0,01 to 0,5 % (*m/m*)

1 Scope and field of application

This part of ISO 7627 specifies the method to be used for the determination of molybdenum, titanium and vanadium contents of hardmetals within the range 0,01 to 0,5 % (*m/m*) by flame atomic absorption spectrometry.

General requirements concerning the field of application, principle, interfering elements, apparatus, sampling and test report are given in ISO 7627/1.

2 Reference

ISO 7627/1, *Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 1: General requirements.*

3 Reagents

3.1 Hydrofluoric acid, ϱ 1,12 g/ml.

3.2 Nitric acid, ϱ 1,42 g/ml.

3.3 Ammonium fluoride, 0,1 g/ml solution.

3.4 Caesium chloride, 0,01 g/ml solution.

3.5 High-purity stock solutions, for calibration purposes for each element to be determined, containing 1,000 g of the element per litre.

NOTE — This value is understood to establish a maximum limit of 1,000 5 g and a minimum limit of 0,999 5 g.

4 Procedure

4.1 Test portion

Weigh, to the nearest 0,001 g, approximately 1 g of the test sample. Transfer it to a 100 ml polytetrafluorethylene beaker or a beaker of other suitable material. Cover the beaker.

4.2 Dissolution of the test portion

Add 10 ml of water, 5 ml of the hydrofluoric acid (3.1). and then 5 ml of the nitric acid (3.2), drop by drop, to the beaker containing the test portion (4.1) and heat gently until the test portion is completely dissolved. Add 10 ml of the caesium chloride solution (3.4) and 10 ml of the ammonium fluoride solution (3.3). Then transfer the solution totally to a 100 ml polypropylene one-mark volumetric flask and dilute to the mark.

4.3 Dilution volume

Prepare the relevant dilution volume for the analysis according to table 1 as follows:

4.3.1 Dilution volume 100 ml: use the solution in 4.2.

4.3.2 The concentration of the solution may be reduced by a factor of 10 for instruments of higher sensitivity by transferring 10 ml of the solution in 4.2 to a 100 ml polypropylene one-mark volumetric flask. Add 10 ml of the caesium chloride solution (3.4). Add 10 ml of the ammonium fluoride solution (3.3) and dilute to the mark.

4.4 Preparation of calibration and blank solutions

4.4.1 Prepare at least six solutions according to 4.2 with a matrix composition as similar as possible to the test portion to be analysed, but without making up to volume. Then add increasing volumes of properly diluted stock solutions of the elements to be determined according to the concentration ranges to be covered. Make up to 100 ml and mix.

4.4.2 Also prepare calibration solutions with a diluted matrix in accordance with 4.3 for dilution volumes of 1 000 ml, if necessary.

4.4.3 Prepare one or more blank solutions (see 4.4.1) without the addition of the relevant element to be determined.