INTERNATIONAL STANDARD 4941

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Steels and cast irons — Determination of molybdenum content — Photometric method

Aciers et fontes — Dosage du molybdène — Méthode photométrique

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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.

International Standard ISO 4941 was developed by Technical Committee ISO/TC 17, *Steel*, and was circulated to the member bodies in August 1976.

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

Austria	India	Romania
Belgium	Iran	Spain
Brazil	Ireland	Sweden
Bulgaria	Italy	Switzerland
Canada	Japan	Turkey
Finland	Korea, Rep. of	United Kingdom
France	Mexico	U.S.A.
Germany	Netherlands	U.S.S.R.
Hungary	Portugal	Yugoslavia

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Australia
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Steels and cast irons — Determination of molybdenum content — Photometric method

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1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method for the determination of the molybdenum content of steels and cast irons.

The method is applicable to steels and cast irons having molybdenum contents between 0,003 and 9 % (*m/m*).

Vanadium and tungsten interfere with the measurement if, because of their contents, the V/Mo ratio is greater than 16 or the W/Mo ratio is greater than 8.

NOTE — Greater V/Mo or W/Mo ratios (up to 300) may, however, be permitted, but in such cases it is necessary to carry out the measurement very quickly after the extraction.

2 REFERENCE

ISO/R 377, *Selection and preparation of samples and test pieces for wrought steel.*

3 PRINCIPLE

Dissolution of a test portion in an appropriate mixture of acids and decomposition of the carbides by oxidation. Quantitative formation of a coloured compound of molybdenum, in the presence of thiocyanate, iron(II) and/or copper(II) ions and extraction of this compound using *n*-butyl acetate.

Photometric measurement of the coloured compound at a wavelength of about 470 nm.

NOTE — When the conditions of the procedure are respected, the coefficient of molecular absorption is 18 930 ± 60.

4 REAGENTS

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

4.1 Iron, in flake or powder form, with a molybdenum content less than 0,005 % and free from tungsten and vanadium.

4.2 *n*-Butyl acetate.

4.3 Nitric acid, ρ about 1,40 g/ml, about 14 M solution.

4.4 Hydrochloric acid, ρ about 1,19 g/ml, about 12 M solution.

4.5 Hydrochloric acid, about 9 M solution (3 + 1).

4.6 Hydrochloric acid, about 6 M solution (1 + 1).

4.7 Acid mixture I.

To 1 volume of the nitric acid (4.3) add 2 volumes of the hydrochloric acid (4.4) and mix well.

Prepare the solution immediately before use.

4.8 Acid mixture II.

Add 150 ml of phosphoric acid, ρ about 1,70 g/ml, to 300 ml of water, and add this diluted acid to 360 ml of perchloric acid, ρ about 1,67 g/ml. Dilute to 1 000 ml with water and mix.

NOTE — In the preparation of this acid mixture, the 360 ml of perchloric acid, ρ about 1,67 g/ml, may be replaced by 150 ml of sulphuric acid, ρ about 1,84 g/ml.

4.9 Ascorbic acid, 100 g/l solution.

Prepare this solution at the moment of use.

4.10 Ammonium thiocyanate, 320 g/l solution.

Store this solution away from light.

4.11 Copper(II), solution corresponding to 70 mg of Cu(II) per litre in a hydrochloric medium about 1,5 M.

Dissolve 0,188 g of copper(II) chloride dihydrate ($CuCl_2.2H_2O$) or 0,275 g of copper(II) sulphate pentahydrate ($CuSO_4.5H_2O$) in 125 ml of the hydrochloric acid (4.4). Make up the volume to 1 000 ml with water and mix.

4.12 Tin(II) copper(II) chloride, solution in a hydrochloric medium about 2 M.

Dissolve 80 g of tin(II) chloride in 155 ml of the hydrochloric acid (4.4). Add 100 ml of the copper(II) solution (4.11). Make up the volume to 1 000 ml with water and mix.

Prepare the solution immediately before use.