SWEDISH STANDARDS INSTITUTE

SVENSK STANDARD
SS-EN ISO 10714

Fastställd 2002-08-16

Utgåva 1

Järn och stål – Bestämning av fosforhalt – Spektrometrisk metod med fosforvanadomolybdat
(ISO 10714:1992)

Steel and iron – Determination of phosphorus content – Phosphovanadomolybdate spectrometric method
(ISO 10714:1992)

ICS 77.040.30

Språk: engelska

Tryckt i september 2002

Europastandarden EN ISO 10714:2002 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN ISO 10714:2002.

The European Standard EN ISO 10714:2002 has the status of a Swedish Standard. This document contains the official English version of EN ISO 10714:2002.

Dokumentet består av 13 sidor.

Upplysningar om **sakinnehållet** i standarden lämnas av SIS, Swedish Standards Institute, tel 08 - 555 520 00.

Standarder kan beställas hos SIS Förlag AB som även lämnar **allmänna upplysningar** om svensk och utländsk standard.
Postadress: SIS Förlag AB, 118 80 STOCKHOLM
Telefon: 08 - 555 523 10. *Telefax*: 08 - 555 523 11
E-post: sis.sales@sis.se. *Internet*: www.sisforlag.se

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 10714

July 2002

ICS 77.040.30

English version

Steel and iron - Determination of phophorus content - Phosphovanadomolybdate spectrophotometric method (ISO 10714:1992)

Aciers et fontes - Dosage du phosphore - Méthode par spectrophotométrie au phosphovanadomolybdate (ISO 10714:1992)

Bestimmung des Phosphorgehaltes - Fotometrische Bestimmung - Vanadatomolybdatophosphat-Verfahren (ISO 10714:1992)

This European Standard was approved by CEN on 29 May 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN ISO 10714:2002 E

Foreword

The text of the International Standard from Technical Committee ISO/TC 17 "Steel" of the International Organization for Standardization (ISO) has been taken over as a European Standard by Technical Committee ECISS/TC 20 "Methods of chemical analysis of ferrous products", the secretariat of which is held by SIS.

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 January 2003, and conflicting national standards shall be withdrawn at the latest by January 2003.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 10714:1992 has been approved by CEN as a European Standard without any modifications.

NOTE Normative references to International Standards are listed in annex ZA (normative).

Steel and iron — Determination of phosphorus content — Phosphovanadomolybdate spectrophotometric method

1 Scope

This International Standard specifies a spectrophotometric method for the determination of phosphorus in steel and iron with the following limitations.

The method is applicable to phosphorus contents between 0,001 0 % (*m*/*m*) and 1,0 % (*m*/*m*).

Arsenic, hafnium, niobium, tantalum, titanium, and tungsten interfere in determining phosphorus, but the interferences can be partially overcome by formation of complexes and use of small quantities of test portion. Depending on the concentration of the interfering elements, the application ranges and test portions given in table 1 apply.

The lower end of the application range can only be reached in test samples with low contents of the interfering elements.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 377-2:1989, *Selection and preparation of samples and test pieces of wrought steels — Part 2: Samples for the determination of the chemical composition.*

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

Table 1

Maximum content of the interfering elements, % (*m*/*m*)						Test portion	Application range, Δw_P
As	Hf	Nb	Ta	Ti	W	g	% (*m*/*m*)
0,05	0,1	1	0,1	2	2	1,0	0,001 to 0,010
0,2	0,5	5	0,5	10	8	0,25	0,005 to 0,040
0,5	1,5	10	1,0	25	25	0,10	0,010 to 0,100
0,2	0,5	5	0,5	10	8	0,25	0,100 to 1,00

3 Principle

Dissolution of a test portion in an oxidizing acid mixture.

Fuming with perchloric acid and removal of chromium as volatile chromyl chloride.

Complexing of silicon and the refractory elements with hydrofluoric acid and complexing of the excess of hydrofluoric acid with orthoboric acid.

Conversion of phosphorus to phosphovanadomolybdate in perchloric and nitric acid solution.

Extraction of phosphovanadomolybdate by 4-methyl-2-pentanone with citric acid present to complex arsenic.

Spectrophotometric measurement at a wavelength of 355 nm.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 2 water as specified in ISO 3696.

Verify by blank tests (7.2) that the relevant reagents are free from phosphorus or of very low phosphorus content. Whenever necessary, the results shall be corrected accordingly. Grades giving high blank values (above 10 µg) are unsuitable and shall be discarded.

4.1 Hydrochloric acid, ρ about 1,19 g/ml.

4.2 Nitric acid, ρ about 1,40 g/ml.

4.3 Nitric acid, ρ about 1,40 g/ml, diluted 1 + 4.

4.4 Perchloric acid, ρ about 1,54 g/ml.

4.5 Hydrofluoric acid, 40 % (m/m), ρ about 1,14 g/ml.

4.6 Citric acid, solution.

Dissolve 500 g of citric acid monohydrate ($H_8C_6O_7.H_2O$) in water, dilute to 1 000 ml and mix.

4.7 4-Methyl-2-pentanone (isobutyl methyl ketone).

The same batch of 4-methyl-2-pentanone shall be used for analysing a series of samples.

4.8 Hexa-ammonium heptamolybdate, solution.

Dissolve 150 g of hexa-ammonium heptamolybdate tetrahydrate [$(NH_4)_6Mo_7O_{24}.4H_2O$] in water, dilute to 1 000 ml and mix.

This solution shall be freshly prepared each day.

High and unstable blank values might be attributable to this reagent in particular. In such a case, change to another batch.

4.9 Ammonium metavanadate, solution.

Dissolve 2,5 g of ammonium metavanadate (NH_4VO_3) in water, dilute to 1 000 ml and mix.

4.10 Sodium nitrite, solution.

Dissolve 50 g of sodium nitrite ($NaNO_2$) in water, dilute to 1 000 ml and mix.

4.11 Tetrafluoroboric acid, solution.

Dissolve 75 g of orthoboric acid (H_3BO_3) in 600 ml of water in a plastic beaker. Add 50 ml of hydrofluoric acid (4.5), dilute to 1 000 ml with water and mix. The solution can be gently heated if the boric acid tends to precipitate.

Keep the solution in a plastic bottle.

4.12 Phosphorus, standard solutions.

4.12.1 Stock solution, corresponding to 1 g of P per litre.

Weigh, to the nearest 0,000 1 g, 4,393 6 g of potassium dihydrogen orthophosphate (KH_2PO_4) previously dried to constant mass at 110 °C and cooled in a desiccator.

Transfer to a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

1 ml of this stock solution contains 1 mg of P.

4.12.2 Standard solution, corresponding to 10 mg of P per litre.

Transfer 10,0 ml of the stock solution (4.12.1) to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

Prepare this standard solution just before use.

1 ml of this standard solution contains 10 µg of P.

5 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 385-1, ISO 648 or ISO 1042 as appropriate.

Ordinary laboratory apparatus, and

5.1 Spectrophotometer, equipped to measure absorbance at a wavelength of 355 nm.

Using the slitwidth recommended by the manufac-