

International Standard

ISO 10714

2024-07

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

Aciers et fontes — Détermination des teneurs en phosphore — Méthode spectrophotométrique au phosphomolybdovanadate

Document Preview

ISO 10714·2024

https://standards.iteh.ai/catalog/standards/iso/6ed35a2a-1f5e-427c-b6fd-1caf82c22a0c/iso-10714-2024

Second edition

iTeh Standards (https://standards.iteh.ai) Document Preview

ISO 10714:2024

https://standards.iteh.ai/catalog/standards/iso/6ed35a2a-1f5e-427c-b6fd-1caf82c22a0c/iso-10714-2024



COPYRIGHT PROTECTED DOCUMENT

© ISO 2024

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office CP 401 • Ch. de Blandonnet 8 CH-1214 Vernier, Geneva Phone: +41 22 749 01 11 Email: copyright@iso.org Website: www.iso.org

Published in Switzerland

ISO 10714:2024(en)

Cor	Contents						
Fore	word		iv				
1	Scop	e	1				
2	Norr	native references	1				
3	Tern	as and definitions	1				
4	Scope Normative references Terms and definitions Principle Reagents Apparatus Sampling and sample preparation Procedure 8.1 Test portion 8.2 Blank test 8.3 Determination 8.3.1 Preparation of the test solution 8.3.2 Colour development and extraction 8.3.3 Spectrophotometric measurements 8.4 Establishment of the calibration curve 8.4.1 Preparation of calibration solutions 8.4.2 Spectrophotometric measurements 8.4.3 Plotting the calibration curve Expression of results 9.1 Method of calculation						
5	-						
6	••						
7	Sam	pling and sample preparation	4				
8	8.1 8.2 8.3	Test portion Blank test Determination 8.3.1 Preparation of the test solution 8.3.2 Colour development and extraction 8.3.3 Spectrophotometric measurements Establishment of the calibration curve 8.4.1 Preparation of calibration solutions 8.4.2 Spectrophotometric measurements 8.4.3 Plotting the calibration curve					
9	9.1	Method of calculation	6				
10	Test	Dishment of the calibration curve					
Anne	ex A (in	formative) Additional information on the international interlaboratory test	8				
Anne	ex B (in	formative) Graphical presentation of precision data	9				
Bibli	ograpl	180 10714:2024	.,				

ISO 10714:2024(en)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 459, *ECISS - European Committee for Iron and Steel Standardization*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 10714:1992), which has been technically revised. ISO 10714:2024

The main changes are as follows:

a complete revaluation of the precision data.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

1 Scope

This document specifies a spectrophotometric method for the determination of phosphorus in steel and

The method is applicable to phosphorus contents between 0,001 0 % (mass fraction) and 1,0 % (mass fraction).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, Laboratory glassware — Single-volume pipettes

ISO 1042, Laboratory glassware — One-mark volumetric flasks

ISO 3696, Water for analytical laboratory use — Specification and test methods

ISO 14284, Steel and iron — Sampling and preparation of samples for the determination of chemical composition

3 Terms and definitions

No terms and definitions are listed in this document. 714:2024

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

4 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 presence of perchloric and nitric acids.

Extraction of phosphovanadomolybdate by 4-methyl-2-pentanone in presence of citric acid to complex arsenic.

Spectrophotometric measurements at a wavelength of 355 nm.

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

ISO 10714:2024(en)

portion. Depending on the content of the interfering elements, the application ranges and test portions given in <u>Table 1</u> apply.

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

Table 1 — Application ranges and test portions

	Maximum	content of th % (mass	Test portion	Application range, $\Delta w_{\rm p}$			
As	Hf	Nb	Та	Ti	W	g	% (mass fraction)
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

5 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 (8.2) that the reagents are free from phosphorus or of very low phosphorus content. Whenever necessary, the results shall be corrected accordingly. Reagents giving high blank values (above $10 \mu g$) are unsuitable and shall be discarded.

- **5.1 Hydrochloric acid,** ρ about 1,19 g/ml.
- **5.2** Nitric acid, ρ about 1,40 g/ml.
- **5.3** Nitric acid solution, ρ about 1,40 g/ml, diluted 1 + 4.
- **5.4 Perchloric acid,** ρ about 1,54 g/ml.

WARNING — Perchloric acid vapour can cause explosions in the presence of ammonia, nitrous fumes or organic material in general.

- **5.5 Hydrofluoric acid,** ρ about 1,14 g/ml.
- **5.6 Citric acid,** 500 g/l.

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

5.7 4-Methyl-2-pentanone (isobutyl methyl ketone).

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

5.8 Hexa-ammonium heptamolybdate, 150 g/l.

Dissolve 150 g of hexa-ammonium heptamolybdate tetrahydrate [(NH₄) $_6$ Mo $_7$ O $_2$ 4·4H $_2$ O] in water, dilute to 1 000 ml with water and mix.

This solution shall be freshly prepared each day.

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