SLOVENSKI STANDARD
oSIST prEN 10351:2009

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Kemična analiza železovih zlitin - Analiza z direktno spektrometrijo optične emisije z induktivno sklopljeno plazmo malolegiranih jekel - Določevanje Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (skupni) in Sn [rutinska metoda]

Chemical analysis of ferrous materials - Inductively coupled plasma optical emission spectrometric analysis of low alloyed steels - Determination of Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (total) and Sn [Routine method]

Analyse chimique des matériaux sidérurgiques - Analyse des aciers faiblement alliés par spectrométrie d'émission optique avec source à plasma induit - Détermination de Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (total) et Sn [Méthode de routine]

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Page

Foreword

This document (prEN 10351:2009) has been prepared by Technical Committee ECISS/TC 20 “Methods of chemical analysis of ferrous products”, the secretariat of which is held by SIS.

This document is currently submitted to the CEN Enquiry.

1 Scope

This document specifies an inductively coupled plasma emission spectrometry routine method for the analysis of unalloyed and low alloyed steels, whose iron content should be at least 95 %.

This method is applicable to the elements listed in Table 1 within the ranges shown.

Table 1 — Application ranges

Element	Mass fraction %	
	min.	max.
Mn	0,005	2,00
P	0,005	0,05
Cu	0,005	0,80
Ni	0,010	2,00
Cr	0,010	1,60
Mo	0,005	0,80
V	0,002	0,40
Co	0,002	0,10
Al (total)	0,020	0,30
Sn	0,001	0,10

In all cases, the ranges specified can be extended or adapted (after validation) for the determination of other mass fractions, provided that the iron content in the samples under concern still is above 95 %.

Other elements may be included. However such elements and their mass fractions should be carefully checked, taking into account the eventual interferences, the sensitivity, the resolution and the linearity criteria for each instrument and each wavelength.

Depending also on the sensitivity of each instrument, suitable dilutions of the calibration and the test sample solutions may be necessary.

Moreover, even if the method described is a "multi elemental" method it is not absolutely necessary to carry out the determination of all the elements of its scope simultaneously: the measurement conditions have to be optimised by each laboratory, depending on the performances of each apparatus available.

NOTE 1 The accuracy of the method is unsatisfactory for phosphorus contents from 0,05 to 0,1 %.

NOTE 2 The trueness of the method couldn't be checked for vanadium contents below 0,05 %.

NOTE 3 The precision of the method is unsatisfactory for aluminium (total) contents below 0,02 %.

2 Normative references

This working draft incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to, or revisions of any of these publications apply to this working draft only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred applies (including amendments)

EN ISO 14284, *Steel and iron — sampling and preparation of samples for the determination of chemical composition.*

ISO 648, *Laboratory glassware — One mark pipettes.*

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

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

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method.*

CEN/TR 10345:2008, *Guideline for statistical data treatment of inter laboratory tests for validation of analytical methods.*

3 Principle

Dissolution of a test portion with nitric and hydrochloric acids. Filtration and ignition of the acid insoluble residue. Removal of silica with hydrofluoric acid. Fusion of the residue with a mixture of orthoboric acid and potassium carbonate, dissolution of the melt with acid and addition of this solution to the reserved filtrate.

After suitable dilution and, if necessary, addition of an internal reference element, nebulisation of the solution into an inductively coupled plasma emission spectrometer and measurement of the intensity of the emitted light (including, eventually, that of the internal reference element).

4 Reagents

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

The same reagents should be used for the preparation of calibration solutions and of sample solutions.

4.1 Hydrochloric acid, HCl (ρ_{20} = 1,19 g/ml)

4.2 Hydrochloric acid, solution 1 + 1

Add 500 ml of hydrochloric acid (4.1) to 500 ml of water.

4.3 Nitric acid, HNO_3 (ρ_{20} = 1,33 g/ml)

4.4 Nitric acid, solution 1 + 1

Add 500 ml of nitric acid (4.3) to 500 ml of water.

4.5 Hydrofluoric acid, HF (ρ_{20} = 1,13 g/ml)

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water, apply a topical gel containing 2,5 % (mass fraction) calcium gluconate, and seek immediate medical treatment.