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Title: Draft BS ISO 4653 Ferronickel — Determination of carbon, sulphur, silicon, phosphorus, nickel, cobalt, chromium, and copper contents by Spark Emission Spectrometry.

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Introduction

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

For international standards, comments will be reviewed by the relevant UK national committee before sending the consensus UK vote and comments to the international committee, which will then decide appropriate action. If the international standard is approved, it is usual for the text to be published as a British Standard.

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Please indicate whether you consider the UK should submit a negative (with supporting technical reasons) or positive vote on this draft. Please indicate if you are aware of any reason why this draft standard should not be published as a British Standard.

Submission of Comments

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Template for comments and secretariat observations

Date: xx/xx/20xx	Document: ISO/DIS xxxx
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1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/ Figure/Table/Note	Type of comment	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	



DRAFT International Standard

ISO/DIS 4653

Ferronickel — Determination of carbon, sulphur, silicon, phosphorus, nickel, cobalt, chromium, and copper contents by Spark Emission Spectrometry

ICS: 77.120.40; 77.100

ISO/TC 155

Secretariat: **AFNOR**

Voting begins on:
2025-11-25

Voting terminates on:
2026-02-17

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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 documents 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).

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This document was prepared by Technical Committee ISO/TC 155, Nickel and nickel alloys.

Ferronickel — Determination of carbon, sulphur, silicon, phosphorus, nickel, cobalt, chromium, and copper contents by Spark Emission Spectrometry

1 Scope

This document specifies a routine method for the determination of carbon, sulphur, silicon, phosphorus, nickel, cobalt, chromium and copper contents in ferronickels by spark optical emission spectrometry (S-OES). This method cannot be used for arbitration analysis.

The method is applicable to the content ranges (mass fraction) given in [Table 1](#).

Table 1 — Content ranges

Element	Content range % (mass fraction)
Carbon	0,030 to 2,5
Sulphur	0,018 to 0,27
Silicon	0,13 to 4,2
Phosphorus	0,007 to 0,04
Nickel	14,3 to 48,5
Cobalt	0,13 to 2,2
Chromium	0,08 to 2,3
Copper	0,019 to 0,26

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 8049, *Ferronickel shot — Sampling for analysis*

ISO 8050, *Ferronickel ingots or pieces — Sampling for analysis*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 reference material RM

material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to befit for its intended use in a measurement process

[SOURCE: ISO GUIDE 30:1992/Amd.1:2008, definition 2.1]

3.2

certified reference material

CRM

reference material (RM) characterized by a metrologically valid procedure for one or more specified properties, accompanied by an RM certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

[SOURCE: ISO GUIDE 30:1992/Amd.1:2008, definition 2.2]

3.3

test sample

representative quantity of material for testing purposes

3.4

drift control samples

series of homogeneous materials that contain all the elements which have been calibrated and that cover the low, mid and high points of the calibration range for each element, used to detect variations over time in these points

Note 1 to entry: Drift control samples can also be used for statistical process control (SPC) of the instrument.

3.5

recalibration samples

samples at both low and high points of the calibration ranges used to recalibrate the spectrometer

Note 1 to entry: These samples are measured during the calibration procedure and the intensities obtained are stored in the computer according to the manufacturer's instructions.

Note 2 to entry: No chemical analyses are necessary, but the homogeneity of these samples should be carefully evaluated.

3.6

type recalibration samples

reference samples or internally developed standard samples, with the same matrix and comparable composition as the test samples

3.7

quality control sample

sample with known composition which is analysed in the same way as the test sample to check the trueness of the analytical results

4 Principle

Measurement of the intensity of the radiation, whose wavelength is characteristic of each element, generated by a spark resulting from the application of an electrical discharge between the sample, as one electrode, and an inert counter-electrode.

The content of each element is determined by relating the measured intensities of test samples to calibration curves prepared from reference materials.

5 Apparatus

5.1 Optical emission spectrometer

Spectrometer with spark source capable of measuring the intensities of the optical radiation emitted at specific wavelengths by the elements present in the material.

The wavelengths generally used are given in [Annex A](#).

5.2 Apparatus for sample surface preparation

The most common surface preparation techniques for ferronickel are milling and grinding or turning, provided that the composition of the surface shall not be influenced.

The lathe, milling machine or any other machines used for surface preparation shall be able to produce a surface that conforms to the requirements of [7.1](#).

WARNING — The appropriate safety recommendations for the use of mechanical apparatus shall be observed. These operations shall be carried out only by properly trained personnel wearing appropriate personal protective equipment.

6 Sampling

Sampling shall be carried out in accordance with ISO 8049 or ISO 8050 as appropriate.

The sample needs to be sufficiently homogeneous with regards to the spark impacts. The measuring surface should be free of imperfections.

7 Procedure

7.1 Surface preparation

7.1.1 The surface of the sample must be prepared so that it is sufficiently flat and smooth to tighten the sample chamber and must be reasonably free of contaminants, pores, cracks, inclusions and shrinkage cavities that could otherwise affect the analytical results.

In order to avoid variation of results due to the influence of surface finish, the same method of preparation shall be used for all samples.

To avoid cross-contamination between different sample materials, all relevant components of the machine shall be thoroughly cleaned before use or separate tools shall be used.

Once the surface has been prepared, avoid any contamination, for example fingerprints.

Measurements shall be carried out soon after surface preparation. Samples should be stored, until the time of later measurement, in a desiccator.

7.1.2 The milling, grinding or turning shall be carried out at a suitable speed to avoid undue heating of the sample surface, which might otherwise lead to bias in analysis.

Any lubricants (e.g. propanol) used shall be selected to ensure that they do not affect the analytical result.

7.2 Calibration procedures

7.2.1 General

7.2.1.1 Calibration process

The calibration process is subdivided into calibration and drift compensation by recalibration.

7.2.1.2 Range of calibration

The range of calibration for an element shall extend well below the minimum content reported in the list of alloys composition and above the maximum content reported in the same list, taking into account that the lowest limit should be at least equal to the quantification limit.

7.2.1.3 Number of sparks on calibration samples

The number of sparks carried out on each certified reference material (CRM) or reference material (RM) for calibration shall be not less than four. The spark areas shall be distributed over the prepared surface. Centre and border of the sample have to be avoided. All measurements shall be examined; if any measurement is obviously defective, further sparks shall be carried out to obtain the minimum four acceptable measurements. The average of the four acceptable measurements is used for calibration.

The influence of temperature is very important for direct measurement methods. The sample should be cooled to ambient temperature between each spark.

7.2.2 Calibration

The calibration of the spectrometer is carried out by using a series of certified reference materials (CRMs) or reference materials (RMs) which have the same or at least similar matrix and metallurgical structure as the samples to be analysed, in order to calculate the calibration functions from which the analysis of test samples can be obtained. The content range of the certified reference materials used shall cover that of all the samples to be analysed within each specific analytical program. For each element in each CRM or RM, the mean intensity is correlated to the corresponding certified content and a regression is calculated.

Calibration CRMs or RMs should span the composition ranges and types of materials expected. Extrapolation should be avoided. It is recommended that the number of calibrations RMs used for each curve be twice the number of coefficients to be determined by regression. This includes the curve parameters and any correction coefficients. Minimally, there should be at least one more data point than the number of coefficients or constants in the equation. The proportion of CRM in relation to RM in the analytical curve must be at least 60 %. It is recommended that reference material for calibration curve (RM) have metrological traceability to primary methods.

For each element in every reference material, a regression curve is generated by relating the measured intensity to the certified concentration. The calibration functions are usually stored within a computer, connected to the spectrometer. These calibration functions are 1st or 2nd order polynomials. Verification of the quality of the adjustment is done by means of the regression residuals graph, explained in [B.1](#), and if possible, by a normal probability plot of the regression residuals. Abnormal behavior of the points indicates the necessity of exclusion and/or inclusion of new CRM or RM; the choice of other calibration functions and adjustment in the concentration range, for instance. When the calibration range of any particular element is large, two calibration curves covering different parts of that range may be used.

The calibration shall be in accordance with the spectrometer manufacturer's instruction manual.

The trueness of the analytical procedure is checked by measuring a set of certified reference materials or—if not available—a set of reference materials not used in the calibration. These reference materials shall cover at least the low, mid and high points of the calibration range for each element. The difference between the analysis value of each element and the certified value should not exceed $\pm 2\sigma$ (σ is the uncertainty or standard deviation of the certified value of each element of the standard sample). If the measurement results do not match the certified values, the cause shall be identified and the calibration curve shall be redrawn or optimized.

Some theoretical principles related to calibration are provided in [Annex B](#) for reference.

7.2.3 Recalibration

Drifts of the spectrometer readings shall be corrected using a recalibration procedure as described in the manufacturer's instruction manual. Recalibrations can be done either for all analytical channels (global recalibration), or only for individual analytical channels (selective recalibration).

Recalibration can either be done periodically or due to a deviation from statistical process control (SPC) limits (see [7.3.4](#)). When a periodical recalibration procedure is used, the period depends on the stability of