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SIST-TP CEN/TR 10377:2023

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Navodilo za pripravo standardnih postopkov z valovno-disperzno rentgensko fluorescenčno spektrometrijo

Guidelines for the preparation of standard routine methods with wavelength-dispersive X-ray fluorescence spectrometry

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Guidelines for the preparation of standard routine methods with wavelength-dispersive X-ray fluorescence spectrometry

This Technical Report was approved by CEN on 12 June 2023. It has been drawn up by the Technical Committee CEN/TC 459/SC 2.

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

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CEN/TR 10377:2023 (E)**European foreword**

This document (CEN/TR 10377:2023) has been prepared by Technical Committee CEN/TC 459/SC 2 “Methods of chemical analysis for iron and steel”, the secretariat of which is held by SIS.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes CR 10299:1999.

In comparison with the previous edition, the following modifications have been made:

- Conversion of the document from a CEN Report (CR) to a Technical Report (TR);
- Title: reworded;
- Clause 1, “Purpose of the guideline” split in “Introduction” and “Scope”;
- Definition 3.3, deleted;
- Definition 3.4, deleted;
- Definition 3.9, updated;
- Definition 3.10, updated;
- Definition 3.11, updated;
- Definition 3.12, updated;
- Renumbering of Clauses 2, 4, 5, 6, 7, 8, 9 and 10;
- Annex A updated and became “Bibliography”;
- Annex B, became Annex A;
- Annex C, became Annex B;
- Annex D, became Annex C;
- Annex E, withdrawn.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

Introduction

X-ray Fluorescence Spectrometry (XRF) has been used for several decades as an important analytical tool for routine analysis. XRF is characterized by its speed and high precision over wide content ranges. Since the technique in most cases is used as a relative method, its limitations are often connected to the quality of the calibration samples.

The technique is well established and most of its physical properties are well known.

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CEN/TR 10377:2023 (E)**1 Scope**

This document is intended to be used for the analysis of metals and alloys (namely steels), but it can also be applicable to other materials although the sample preparation techniques differ. The purpose of this document is to describe general concepts and the procedures for calibration and analysis by XRF.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

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

**3.1
calibration**

calculation of the best fit of net intensities and contents from a number of calibration samples to a calibration curve

**3.2
recalibration**

calculation of new calibration constants with a few number of samples, selected from the calibration samples

Note 1 to entry: Calibration samples using the apparent contents calculated in 3.1

Note 2 to entry: To compensate for the day-to-day variations of the instrument a set of recalibration samples is measured; either one with a low and one with a high content for each element (two-point recalibration) or one with a high content only for each element (one-point recalibration). The intensities are compared to the initial intensities recorded during the calibration procedure and recalibration coefficients are calculated. Calibration constants are not changed.

**3.3
background equivalent concentration****BEC**

quantity of analyte which, when subjected to excitation, provides a net intensity equal to the spectral background

**3.4
limit of detection****LOD**

minimum content at which the signal generated by a given element can be positively recognised above any background signals with a specified degree of certainty

3.5**lower limit of detection****LLD**

minimum content at which the signal generated by a given element can be positively recognised above any background signals with a specified degree of certainty

Note 1 to entry: The related calculations are based only on the counting statistical error

3.7**limit of quantification****LOQ**

smallest content that can be determined with a specified degree of certainty

3.8**repeatability conditions**

conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time

[SOURCE: ISO 5725-1:1994]

3.9**reproducibility conditions**

conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment

[SOURCE: ISO 5725-1:1994]

3.10**accuracy**

closeness of agreement between test result and accepted reference value

Note 1 to entry: The term accuracy, when applied to a set of test results, involves a combination of random components and a common systematic error or bias component.

[SOURCE: ISO 5725-1:1994]

3.11**trueness**

closeness of agreement between the average value obtained from a large series of test results and an accepted reference value

Note 1 to entry: The measure of trueness is usually expressed in terms of bias.

Note 2 to entry: Trueness has been referred to as “accuracy of the mean”. This usage is not recommended.

[SOURCE: ISO 5725-1:1994]

3.12**sensitivity, S**

difference in intensities between a sample with a high content and one with a low content divided by the difference in content

Note 1 to entry: Sensitivity is expressed as counts per second per percent.

4 Principle

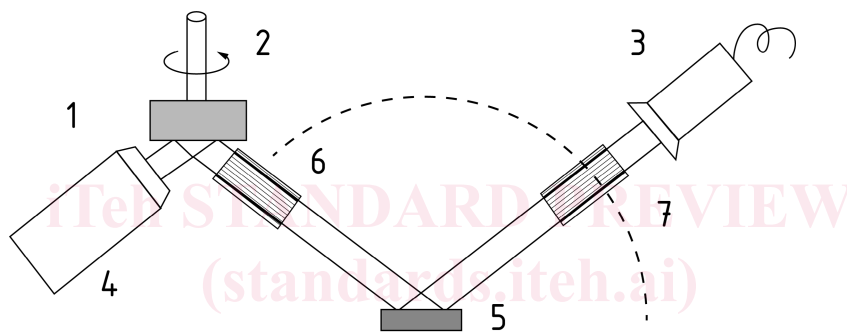
The sample is prepared to a clean uniform surface and then irradiated by an X-ray beam of high energy. The secondary X-rays produced are dispersed by means of crystals and the intensities are measured by detectors at selected characteristic wavelengths. The measuring time is set to reach below a specified statistical counting error.

Contents of the elements are calculated by relating the measured intensities of test samples to calibration curves established with certified reference materials (CRMs) and reference materials (RMs).

5 Instruments

5.1 General

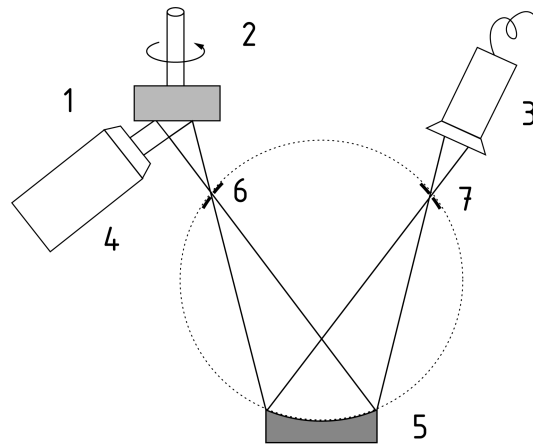
The principle of two different concepts of X-ray fluorescence spectrometer is shown in Figures 1 and 2.



Key

- | | | |
|---|----------------------|---|
| 1 | Sample | https://standards.iteh.ai/catalog/standards/sist/cba1eb08-6780-4796-8b34-4a0c7787a685/sist-tp-cen-tr-10377-2023 |
| 2 | Spinner | |
| 3 | Detector | |
| 4 | Tube | |
| 5 | Crystal | |
| 6 | Primary collimator | |
| 7 | Secondary collimator | |

Figure 1 — Spectrometer geometry of sequential instruments

**Key**

- 1 Sample
- 2 Spinner
- 3 Detector
- 4 Tube
- 5 Crystal
- 6 Source slit
- 7 Detector slit

Figure 2 — Spectrometer geometry of simultaneous instruments

5.2 Tubes

Two different types of X-ray tubes are used: side-window tubes or end-window tubes.

Table 1 gives a comparison of these two types. More favourable measuring conditions are usually obtained for light elements with an end-window tube due to the thinner window.

Table 1 - Comparison of end window and side window tubes

	End window tubes	Side window tubes
Cooling	Two cooling circuits: a) Direct cooling with deionized water; b) Indirect cooling with tap water.	One cooling circuit: Direct cooling with tap water.
Window	Slight thermal stressing: Thinner window.	Greater thermal stressing: Thicker window.
Service life	20 000 h	5 000 h

As target material, different high-purity elements like Rh, Ag, W, Cr or Au are used. For the analysis of steels rhodium is usually used as a multipurpose tube with good excitation conditions for all elements of interest. If possible, it is suggested that the target material (anode) is not made of an element to be determined. The applicability of the usual anode materials is summarized in Table 2.

The X-ray tube produces a continuous spectrum and a characteristic spectra depending on the selected anode material. For optimum excitation, a maximum excitation energy at least two to three times above the corresponding absorption edge of the element line to be determined is recommended.

At present, equipment is available which can be operated with acceleration voltages up to 100 kV and maximum power of 3 kW. The limitations of the apparatus are given either by the high-voltage supply