



SLOVENSKI STANDARD
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Trdna alternativna goriva - Določevanje elementne sestave z rentgensko fluorescenco (ISO/DIS 22940:2020)

Solid recovered fuels - Determination of elemental composition by X-ray fluorescence (ISO/DIS 22940:2020)

Feste Sekundärbrennstoffe - Bestimmung der Elementzusammensetzung durch Röntgenfluoreszenz (ISO/DIS 22940:2020)

Combustibles solides de recuperation - Détermination de la composition élémentaire par fluorescence de rayons X (ISO/DIS 22940:2020)

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75.160.10 Trda goriva

Solid fuels

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Solid recovered fuels — Determination of elemental composition by X-ray fluorescence

Combustibles solides de recuperation — Détermination de la composition élémentaire par fluorescence de rayons X

ICS: 75.160.10

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Foreword

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This document was prepared by Technical Committee ISO/TC 300, *Solid Recovered Fuels*.

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Introduction

X-ray fluorescence spectrometry can be used as a fast method for a qualitative overview of ash forming elements and impurities. When calibration is based on reference materials or on matrix-matched homogeneous solid recovered fuel samples with known content, X-ray fluorescence spectrometry can be used for a quantitative analysis of the total content of the specified elements within different solid recovered fuels.

The quality of the results obtained depends very closely on the type of instrument used, e.g. bench top or high performance, energy dispersive or wavelength dispersive instruments. When selecting a specific instrument, several factors have to be considered, such as the matrices to be analysed, elements to be determined, detection limits required and the measuring time.

Due to the wide range of matrix compositions and the lack of suitable reference materials in the case of solid recovered fuels from various origin, it is generally difficult to set up a calibration with matrix-matched reference materials. Therefore, it is important to use several homogenized solid recovered fuel samples with properties that sufficiently match the matrices of interest and whose content has been derived by independent measurement techniques, e.g. total digestion of solid recovered fuels and characterization of major and minor elements by measurement of digestion solutions with ICP-MS or ICP-OES or by other techniques like elemental analysis using combustion technology on sulfur or by combustion and ion chromatographic determination for chlorine.

Therefore, this document describes two different procedures:

- 1) Quantitative analytical procedure for major elements of solid recovered fuels. The calibration is based on different reference materials and solid recovered fuel samples with known content.

The elements described as major elements of solid recovered fuels are in fact major elements of the fuel ashes more than of the fuels. The determination of these elements may be helpful to predict the melting behaviour and slagging of the ashes. Moreover, contamination of fuel with sand or soil is indicated by high values of several elements.

- 2) Total element characterization at a semi-quantitative level for major and minor elements of solid recovered fuels. The calibration is based on matrix-independent calibration curves, previously set up by the manufacturer.

In general, the sensitivity of X-ray fluorescence is not sufficient for a determination of the content of minor elements (trace metals) in solid recovered fuels. However, determination of minor elements after calibration with solid recovered fuel samples with known content or at a semi-quantitative level based on matrix-independent calibration curves could be used to collect data for higher sample numbers taking into account lower achievable precision. Therefore, it may be used to reveal excessive contents of minor elements in solid recovered fuels.

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Solid recovered fuels — Determination of elemental composition by X-ray fluorescence

1 Scope

This document specifies the procedure for a determination of major and minor element concentrations in solid recovered fuel material by energy dispersive X-ray fluorescence (EDXRF) spectrometry or wavelength dispersive X-ray fluorescence (WDXRF) spectrometry using a calibration with solid recovered fuel reference materials or solid recovered fuel samples with known content. A semi-quantitative determination may be carried out using matrix independent standards.

X-ray fluorescence spectrometry may be used as a fast method for a qualitative overview of elements and impurities and after suitable calibration it is very useful for determining major elements or even minor elements (except Hg) in order to quickly identify increased concentrations of minor elements in solid recovered fuels (e.g. during SRF-production).

This document is applicable for the following elements: Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Br, Mo, Cd, Sb, Sn, Tl and Pb. Concentrations from approximately 0,000 1 % and above can be determined depending on the element, the calibration materials used and the instrument used.

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 21637:—,¹⁾ *Solid recovered Fuels — Terminology, definitions and descriptions*

ISO 21646:—,²⁾ *Solid recovered fuels — Sample preparation*

ISO 21660-3:—,³⁾ *Solid recovered fuels — Determination of moisture content using the oven dry method — Part 3: Moisture in general analysis sample*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 21637:— and the following apply.

3.1

absorption edge

abrupt change in mass absorption coefficient at a specific wavelength or energy

3.2

absorption of X-rays

loss of intensity of X-rays through absorption by an isotropic and homogenous material as described by the Beer-Lambert law

1) Under preparation. (Stage at the time of publication ISO/FDIS 21637.)

2) Under preparation. (Stage at the time of publication ISO/CD 21646)

3) Under preparation. (Stage at the time of publication ISO/DIS 21660-3.)

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3.3

analytical line

specific characteristic X-ray spectral line of the atom or ion of the analyte used for determination of the analyte content

3.4

continuous radiation

electromagnetic radiation produced by the acceleration of a charged particle, such as an electron, when deflected by another charged particle, such as an atomic nucleus

3.5

compton-line

spectral line due to incoherent scattering (Compton-effect), occurring when the incident X-ray photon strikes an atom without promoting fluorescence

Note 1 to entry: Energy is lost in the collision and therefore, the resulting scattered X-ray photon is of lower energy than the incident X-ray photon.

3.6

drift correction monitors

physically stable samples used to correct for instrumental drift

3.7

emitted sample X-rays

radiation emitted by sample consisting of *X-ray fluorescence radiation* (3.14) and scattered *primary X-rays* (3.12)

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3.8

mass absorption coefficient

constant describing the fractional decrease in the intensity of a beam of X-radiation as it passes through an absorbing medium

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Note 1 to entry: It is expressed in cm^2/g .

Note 2 to entry: The mass absorption coefficient is a function of the wavelength of the absorbed radiation and the atomic number of the absorbing element.

3.9

polarised excitation X-ray spectrometer

energy dispersive X-ray spectrometer where the excitation is performed by polarised radiation and the emitted *X-ray fluorescence radiation* (3.14) is detected along the direction of polarisation

3.10

powder sample

analyte sample submitted as a powder for direct measurement in the sample cup

3.11

pressed pellet

analyte sample prepared by pressing milled material into a disk

3.12

primary X-rays

X-rays by which the sample is radiated

3.13

quality control sample

stable sample with known contents, e.g. (certified) reference material (CRM) or homogenized solid recovered fuel samples from known origin which contents have been derived by independent analysis used to monitor instrument and calibration performance

3.14**X-ray fluorescence radiation**

emission of characteristic X-rays from a sample that has been bombarded by high-energy X-rays or gamma rays

4 Safety remarks

The X-ray fluorescence spectrometer shall comply with international and national regulations relevant to radiation protection.

The person responsible for managing or supervising the operation of X-ray equipment shall provide evidence of his knowledge of radiation protection according to national regulations.

5 Symbols and abbreviated terms**5.1 Symbols**

Al Aluminium

As Arsenic

Br Bromine

Ca Calcium

Cd Cadmium

Cl Chlorine

Co Cobalt

Cr Chromium

Cu Copper

Fe Iron

K Potassium

Mg Magnesium

Mn Manganese

Mo Molybdenum

Na Sodium

Ni Nickel

P Phosphorus

Pb Lead

S Sulphur

Sb Antimony

Si Silicon

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Sn	Tin
Ti	Titanium
Tl	Thallium
V	Vanadium
Zn	Zinc

5.2 Abbreviated terms

EDXRF	Energy dispersive X-ray fluorescence
MCA	Multi-Channel Analyser
WDXRF	Wavelength dispersive X-ray fluorescence

6 Principle

After a suitable preparation, the sample is introduced into a XRF-spectrometer and excited by primary X-rays. The intensities of the secondary fluorescent energy lines specific for each element are measured and the elemental composition of the sample is determined by reference to previously established calibration graphs or equations and applying corrections for inter-element effects. The calibration equations and inter-element corrections are established using pure reagents and/or series of internal or reference materials provided that they meet all the requirements of the relevant preparation technique.

7 Apparatus

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7.1 X-ray fluorescence spectrometer shall be able to analyse the elements according to the scope of this document. The following types of X-ray fluorescence spectrometers are applicable:

- energy dispersive X-ray fluorescence (EDXRF) spectrometer that achieves the dispersion of the emitted X-ray fluorescence radiation by an energy dispersive detector;
- wavelength dispersive X-ray fluorescence (WDXRF) spectrometer that achieves the dispersion of the emitted X-ray fluorescence radiation by diffraction by a crystal or a synthetic multilayer.

The spectrometer consists of the following components:

- primary X-ray source, an X-ray tube with a high voltage generator;
- sample holder;
- detector unit including electronic equipment;
- source modifiers to modify the shape or intensity of the source spectrum or the beam shape (like source filters, secondary targets, polarizing targets, collimators, focusing optics, etc.).

The detector unit is different for WDXRF and for EDXRF spectrometers. WDXRF spectrometers take advantage of the dispersion of the emitted radiation by diffraction by a crystal or a synthetic multilayer. The detector does not need to be capable of energy discrimination. EDXRF spectrometers use an energy dispersive detector. Pulses of current from the detector, which are a measure of the energy of the incoming X-rays, are segregated into channels according to energy using a Multi-Channel Analyser (MCA).

NOTE 1 The use of a high-energy X-ray tube increases the potential for losses of volatile analytes from samples by heating in the spectrometer during analysis.

NOTE 2 The new generation of EDXRF spectrometers takes advantage of the polarizing target theory resulting in a significant decrease of the background scattering, and therefore lower limits of detection can be achieved (comparable to WDXRF).

7.2 Pellet press, capable of providing a pressure of at least 30 kN. The pellet press may be a cold press or a hot mould press, operating at temperatures not exceeding 180 °C.

8 Interferences and sources of error

Interferences in X-ray fluorescence spectrometry are due to spectral line overlaps, matrix effects, spectral artefacts and particle size or mineralogical effects.

Spectral line overlaps occur when an analytical line cannot be resolved from the line of a different element. Corrections for these interferences are made using the algorithms provided with the instrument software.

Matrix effects occur when the X-ray fluorescence radiation from the analyte element is absorbed or enhanced by other elements in the sample before it reaches the detector. In the case of complex matrices, these effects generally have to be corrected. The correction procedure depends on the X-ray fluorescence spectrometry system (EDXRF or WDXRF) and the apparatus type itself.

Spectral artefacts, e.g. escape peaks, sum peaks, pulse pile up lines, dead time, Bremsstrahlung correction, are accounted for by the provided instrument software. Spectral artefacts differ for energy dispersive and wavelength dispersive XRF spectrometry.

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9 Sample preparation (standards.iteh.ai)

9.1 Preparation principles oSIST prEN ISO 22940:2020

The sample preparation is very critical for XRF analysis of solid recovered fuels. The quality of sample preparation strongly influences the accuracy of the results. The following different options exist:

- For quantitative analysis of solid recovered fuel samples, the preparation of pressed pellets from prepared general analysis sample material is recommended.
- For semi-quantitative analysis of solid recovered fuels, the general analysis material may be used directly (in powder form) and concerning samples of solid recovered fuel pellets, the original pellets may be used directly without any sample preparation. It may be used to provide fast basic information about the approximate composition of a sample. Similar results may be obtained using portable XRF instruments for field analysis.

For a given calibration, the same preparation method shall be used throughout, for both samples and standards.

For precise quantitative measurements, homogeneous and representative test portions are necessary. The nominal top size of the material shall be 0,5 mm or less, according to ISO 21646:—.

9.2 Drying of general analysis sample material

Dry a sufficient amount of general analysis sample material according to ISO 21660-3:— immediately before pressing pellets for XRF-analysis.

NOTE Concerning some XRF-instruments, the applied vacuum will dry the general analysis sample material during the determination giving the same results as if the sample was previously dried.