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Environmental solid matrices — Determination of elemental composition by X-ray fluorescence spectrometry

Matrices solides environnementales — Détermination de la composition élémentaire par spectrométrie de fluorescence X

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Foreword

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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 190, *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 444, *Environmental characterization of solid matrices,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 18227:2014), which has been technically revised.

The main changes are as follows:

— the contents of the two almost identical standards ISO 18277:2014 and EN 15309:2007 have been combined;

— normative references have been revised.

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.

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Introduction

X-ray fluorescence (XRF) spectrometry is a fast and reliable method for the quantitative analysis of the total content of certain elements within different matrices.

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 should be considered, such as the matrices to be analysed, elements to be determined, detection limits required and the measuring time. The quality of the results depends on the element to be determined and on the surrounding matrix.

Due to the wide range of matrix compositions and the lack of suitable reference materials in the case of inhomogeneous matrices such as waste, it is generally difficult to set up a calibration with matrix- matched reference materials.

Therefore, this document describes two different procedures:

— a quantitative analytical procedure required for homogeneous solid waste, soil and soil-like material, where the calibration is based on matrix-matched standards;

— an optional XRF screening method for solid and liquid material as waste, sludge and soil in Annex A which provides a total element characterization at a semi-quantitative level, where the calibration is based on matrix-independent calibration curves, previously set up by the manufacturer.

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Environmental solid matrices — Determination of elemental composition by X-ray fluorescence spectrometry

1 Scope

This document specifies the procedure for a quantitative determination of major and trace element concentrations in homogeneous solid waste, soil, soil-like material and sludge by energy dispersive X-ray fluorescence (EDXRF) spectrometry or wavelength dispersive X-ray fluorescence (WDXRF) spectrometry using a calibration with matrix-matched standards.

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, Se, Br, Rb, Sr, Y, Zr, Nb, Mo, Ag, Cd, Sn, Sb, Te, I, Cs, Ba, Ta, W, Hg, Tl, Pb, Bi, Th and U. Concentration levels between a mass fraction of approximately 0,000 1 % and 100 % can be determined depending on the element and the instrument used.

An optional XRF screening method for solid and liquid material as waste, sludge and soil is added in Annex A which provides a total element characterization at a semi-quantitative level, where the calibration is based on matrix-independent calibration curves, previously set up by the manufacturer.

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 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/

3.1
absorption edge
jump of the mass absorption coefficient at a specific wavelength or energy

3.2
analytical line
specific characteristic X-ray spectral line of the atom or ion of the analyte used for determination of the analyte content

3.3
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

3.4
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.

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3.5
drift correction monitor
physically stable sample used to correct for instrumental drift

3.6
fused bead
analyte sample prepared by dissolution in a flux

3.7
liquid sample
analyte sample submitted as a solution for direct measurement in the sample cup

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

Note 1 to entry: This 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
powder sample
analyte sample submitted as a powder for direct measurement in the sample cup

3.10
precision
closeness of agreement between independent test results obtained under stipulated conditions

Note 1 to entry: Precision depends only on the distribution of random errors and does not relate to the true value or the specified value.

Note 2 to entry: The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results. Less precision is reflected by a larger standard deviation.

Note 3 to entry: Quantitative measures of precision depend critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme conditions.

[SOURCE: ISO 3534-2:2006, 3.3.4]

3.11
pressed pellet
analyte sample prepared by pressing milled material into a disk

3.12
primary X-ray
X-ray by which the sample is radiated

3.13
quality control sample
stable sample with known contents used to monitor instrument and calibration performance

EXAMPLE Certified reference material (CRM).

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

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4 Safety remarks

Anyone dealing with waste and sludge analysis shall be aware of the typical risks that this kind of material presents irrespective of the parameter to be determined. Waste and sludge samples can contain hazardous substances, e.g. toxic, reactive, flammable, and infectious substances, which could potentially undergo either biological or chemical reaction, or both. Consequently, these samples should be handled with special care. The gases that can be produced by microbiological or chemical activity are potentially flammable and pressurize sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. The user shall be aware of national regulations applicable to hazard handling.

The user shall be aware of 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 as defined in national regulations.

5 Principle

After a suitable preparation, if necessary, the sample is introduced into an 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 either pure reagents or series of internal or reference materials, or both, provided they meet all the requirements of the relevant preparation technique.

6 Apparatus

6.1 X-ray fluorescence spectrometer, which shall be able to analyse the elements listed in 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 a number of components:

— primary X-ray source, an X-ray tube with a high voltage generator;

— a sample holder;

— detector unit including electronic equipment;

— source modifiers to modify the shape or intensity of the source spectrum or the beam shape (e.g. source filters, secondary targets, polarizing targets, collimators, focussing optics).

The detector unit is different for WDXRF and for EDXRF spectrometers. WDXRF spectrometers take advantage of the dispersion of the emitted radiation by scattering by a crystal or a synthetic multilayer. The detector should not 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). The spectrometer is capable to measure under vacuum, helium-atmosphere (7.3) or nitrogen- atmosphere (7.4).

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.

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