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Soil quality — Determination of elemental composition by X-ray fluorescence

Qualité du sol — Détermination de la composition élémentaire par 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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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

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Introduction

X-ray fluorescence 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 have to be considered, such as the matrices to be analysed, the elements to be determined, the detection limits required, and the measuring times. 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 International Standard describes two different procedures:

- a quantitative analytical procedure for homogeneous solid waste, soil, and soil-like material in the normative part. The calibration is based on matrix-matched standards;
- an XRF screening method for solid and liquid materials as waste, sludge, and soil in Annex A which provides a total element characterization at a semi-quantitative level. The calibration is based on matrix-independent calibration curves, previously set up by the manufacturer.

The technical content of this International Standard is identical with the European Standard EN 15309:2007.

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Soil quality — Determination of elemental composition by X-ray fluorescence

1 Scope

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

This International Standard 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 approximately 0,000 1 % and 100 % can be determined depending on the element and the instrument used.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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ISO 11464, *Soil quality — Pretreatment of samples for physico-chemical analysis*

ISO 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

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ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

EN 14346:2006, *Characterization of waste — Calculation of dry matter by determination of dry residue or water content*

EN 15002:2006, *Characterization of waste — Preparation of test portions from the laboratory sample*

3 Terms and definitions

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

NOTE See References [11] and [14] for non-specified terms.

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

3.2
absorption of X-rays
loss of intensity of X-rays by an isotropic and homogenous material as described by the Bouger-Lambert law

3.3
analytical line
specific characteristic X-ray spectral line of the atom or ion of the analyte used for the determination of the analyte content

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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 strike 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 monitor

physically stable sample used to correct for instrumental drift

3.7
emitted sample X-ray

radiation emitted by a sample consisting of X-ray fluorescence radiation and scattered primary X-rays

3.8
fused bead

analyte sample prepared by dissolution in a flux

3.9
liquid sample

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

3.10
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: 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.

3.11
polarized excitation X-ray spectrometer

energy dispersive X-ray spectrometer where the excitation is performed by polarized radiation and the emitted X-ray fluorescence radiation is detected along the direction of polarization

3.12
powder sample

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

3.13
precision

closeness of agreement of results obtained by applying the method several times under prescribed conditions

[SOURCE: ISO 5725-2:1994]

3.14
pressed pellet

analyte sample prepared by pressing milled material into a disk

3.15
primary X-ray

X-ray by which the sample is radiated

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3.16
quality control sample

stable sample with known contents, e.g. certified reference material (CRM), used to monitor instrument and calibration performance

3.17
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

Anyone dealing with waste and sludge analysis has to 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 e.g. toxic, reactive, flammable, and infectious substances, which could potentially undergo biological and/or chemical reaction. Consequently, it is recommended that 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. National regulations should be followed with respect to all hazards associated with this method.

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.

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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 pure reagents and/or series of internal or reference materials providing they meet all the requirements of the relevant preparation technique.

6 Apparatus

6.1 X-ray fluorescence spectrometer, shall be able to analyse the elements according to the scope of this International Standard.

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 (such as 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 scattering 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).

6.2 Mill, preferable with walls made of agate, corundum, or zircon.

6.3 Pellet preparation equipment, manual or automatic pellet press, capable of providing a pressure of at least 100 kN.

6.4 Aluminium cup, supporting backing cup for pressed pellets.

6.5 Fusion apparatus, electric, gas, or high-frequency induction furnace that can be heated up to a fixed temperature of between 1 050 °C and 1 250 °C.

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6.6 Fusion crucibles, crucibles made of non-wetting platinum alloy (Pt 95 %; Au 5 % is suitable).

Lids, if used, shall be made from platinum alloy.

NOTE Certain metal sulphides (so called platinum poisons) affect the platinum crucibles in which the sample is melted.

6.7 Casting moulds, non-wetting platinum alloy (Pt 95 %; Au 5 % is suitable).

7 Reagents

The reagents mentioned are used as carrier material.

7.1 Binder, liquid or solid binder free of analytes of interest.

Solid materials can contain a certain amount of moisture, which shall be compensated for.

NOTE Different type of binders can be used. A binder commonly used is wax.

7.2 Flux, solid flux free of analytes of interest.

Solid materials can contain a certain amount of moisture, which shall be compensated for (see ISO 12677 for compensation for moisture in flux).

NOTE Different type of fluxes can be used. Fluxes commonly used are lithium metaborate, lithium tetraborate, or mixtures of both.

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8 Interferences and sources of error

The container in which the sample is delivered and stored can be a source of error. Its material shall be chosen according to the elements to be determined.

NOTE Elemental Hg can penetrate polyethylene walls very rapidly in both directions. In the case of glass containers, contamination can be observed for some elements, e.g. Al, As, Ba, Ce, K, Na, and Pb.

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 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.

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

Particle size effects can be reduced by milling the sample, and both particle size and mineralogical effects can be eliminated by preparing bead samples. It is vital for quantitative analysis that the same sample preparation procedure is applied to both the standards and the samples to be analysed.

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9 Sample preparation

9.1 General

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In analysis by XRF spectrometry, the sample preparation step is crucial as the quality of the sample preparation strongly influences the accuracy of the results.

For quantitative analysis of solid samples, pressed pellets or fused beads have to be prepared. The application of the pressed pellet method is recommended for the quantification of trace elements and mandatory for the quantification of volatile elements, and the fused bead method for the determination of non-volatile major and minor elements.

NOTE 1 The preparation of fused beads eliminates effects due to particle size and mineralogy.

The conditions of the preparation of fused beads shall be adapted to the matrix properties. Otherwise, the preparation of fused beads can be difficult or can cause problems in case of waste-like matrices such as sludges.

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

NOTE 2 Depending on the sample type, other sample preparation methods can be applied according to Annex B.

For precise quantitative measurements, homogeneous and representative test portions are necessary. Pre-treatment and preparation of test portions shall be carried out according to the appropriate clauses of ISO 11464 and EN 15002. The particle size of the sample can strongly affect the precision of the measurement. The particle size should preferably be smaller than 150 µm.

NOTE 3 Particle size smaller than 80 µm is recommended for the analysis of low atomic mass elements when using the pressed pellet method.

9.2 Drying and determination of dry mass

Prepare and dry the sample according to ISO 11464 or EN 15002. Determine the dry mass according to ISO 11465 or prEN 14346.

9.3 Preparation of pressed pellet

After drying and milling or grinding the sample, a pellet is prepared in the pellet press (6.3). Before pressing, the sample shall be mixed and homogenized with a binder (7.1) in a ratio of sample:binder of 10:1 by weight. For the preparation of 40 mm in diameter pellets, about 10,0 g of sample is taken; for 32 mm in diameter pellets, about 4,5 g of sample is required. The amount of binder in the pellet shall be taken into account for the dilution factor. It is recommended to press the sample in an aluminium cup (6.4) as support.

NOTE 1 Different types of binders can be used. A binder commonly used is wax. In the case of a liquid binder, the pellet is placed in an oven to evaporate organic solvent.

NOTE 2 Different dilution factors can be used.

9.4 Preparation of fused beads

After drying and milling or grinding the sample, a fused bead is prepared using the fusion apparatus (6.5).

Ignite the sample at 1 025 °C ± 25 °C until constant mass is reached. Determine the loss on ignition at the chosen temperature to correct for volatile elements and/or compounds being released during ignition of the sample.

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NOTE 1 The ignition temperature can vary depending on the sample matrix.

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Because of the wide applicability of the fused bead technique, various fluxes and modes of calibration are permitted providing they have been demonstrated to be able to meet certain criteria of reproducibility, sensitivity, and accuracy.

For application of alkaline fusion technique (e.g. selection of flux, fusion temperature, and additives), ISO 14869-2 or CEN/TR 15018 should be used.

NOTE 2 Fluxes commonly used are lithium metaborate, lithium tetraborate, or mixtures of both.

NOTE 3 Loss of volatile elements, e.g. As, Br, Cd, Cl, Hg, I, S, Sb, Se, and Tl, can occur during the fusion process. Also, Cu can be volatile if a bromide-releasing agent is used.

The flux (7.2) is added to the ignited material in a dilution ratio of sample:flux of 1:5 by weight. For the preparation of 40 mm in diameter beads, about 1,6 g of ignited sample is taken; for 32 mm in diameter beads, about 0,8 g of ignited sample is required. The amount of flux in the bead shall be taken into account for the dilution factor. The same sample preparation procedure and ratio of sample to flux shall be used for samples and standards. The beads produced should be visually homogeneous and transparent.

NOTE 4 Non-ignited material can be used to prepare beads but, nevertheless, loss of ignition needs to be determined and needs to be taken into account in the calculation of the results. It should be noted that non-ignited material can contain compounds that can damage the platinum crucibles during fusion.

NOTE 5 Different dilution factors can be used.

After fusion in a platinum-gold crucible (6.6) the melt is poured into a casting mould (6.7) to make a bead.

Beads can deteriorate because of adverse temperature and humidity conditions, so it is recommended that beads are stored in desiccators.

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10 Procedure

10.1 Analytical measurement conditions

10.1.1 Wavelength dispersive instruments

The analytical lines to be used and the suggested operating conditions are given in Table C.1. The settings are strongly dependent on the spectrometer configuration, e.g. the type of X-ray tube (Rh, Cr), tube power, available crystals, and type of collimators.

10.1.1.1 Intensities and background corrections

For the determination of trace elements, the measured intensities have to be background-corrected. The measured background positions should be free of spectral line interferences. The net peak intensity I, expressed as the number of counts per second of the element of interest, is calculated as the difference between the measured peak intensity of the element and the background intensity:

$$I = I_p - I_b \tag{1}$$

where

I_p is the count rate of the element i, expressed as the number of counts per second;

I_b is the background count rate of the element i, expressed as the number of counts per second.

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10.1.1.2 Counting time

The minimum counting time is the time necessary to achieve an uncertainty ($2\sigma_{\%}$), which is less than the desired precision of the measurement. Choose a reference material with a concentration level in the middle of the working range and measure the count rate. The counting time for each element can be calculated according to Formula (2):

$$t = \left(\frac{100}{2\sigma_{\%}} . \frac{1}{\sqrt{I_p} - \sqrt{I_b}} \right)^2 \tag{2}$$

where

t is the total counting time for the peaks and background, in seconds;

$2\sigma_{\%}$ is the relative target precision at a confidence level of 95 %, expressed as percentage.

10.1.2 Energy dispersive instruments

The analytical lines to be used and the suggested operating conditions are given in Table C.2. The settings are strongly dependent on the spectrometer configuration, e.g. type of X-ray tube (Rh, Pd), tube power, available targets, and type of filters.

Intensities and background corrections

Deconvolution of the spectra and background correction are needed when analysing the samples with overlapping lines. Usually, XRF instruments are supplied with a specific software module for that purpose.

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