



BSI Standards Publication

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

National foreword

This British Standard is the UK implementation of EN ISO 18227:2025. It is identical to ISO 18227:2025. It supersedes BS EN 15309:2007 and BS ISO 18227:2014, which are withdrawn.

The UK participation in its preparation was entrusted to Technical Committee EH/4, Soil quality.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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Feststoffe in der Umwelt - Bestimmung der
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Röntgenfluoreszenz (ISO 18227:2025)

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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

European foreword

This document (EN ISO 18227:2025) has been prepared by Technical Committee ISO/TC 190 "Soil quality" in collaboration with Technical Committee CEN/TC 444 "Environmental characterization of solid matrices" the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2026, and conflicting national standards shall be withdrawn at the latest by June 2026.

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Endorsement notice

The text of ISO 18227:2025 has been approved by CEN as EN ISO 18227:2025 without any modification.

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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 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.

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.

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.

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

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.

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 Analytical balance, readable and accurate to 0,001 g.

6.3 Drying oven, thermostatically controlled and capable of maintaining a temperature of $(105 \pm 5) ^\circ\text{C}$.

6.4 Grinding mill, capable of grinding dried materials to a required particle size without contaminating the samples with compounds to be determined, preferable with walls made of agate, corundum or zircon.

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

6.6 Aluminium cup: supporting backing cup for pressed pellets.

6.7 Fusion apparatus: electric, gas or high frequency induction furnace that can be heated up to a fixed temperature of between 1 000 $^\circ\text{C}$ and 1 250 $^\circ\text{C}$.

6.8 Fusion crucibles: crucibles made of non-wetting platinum alloy (with a mass fraction of 95 % Pt and a mass fraction of 5 % Au is suitable).

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

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

6.9 Casting moulds: non-wetting platinum alloy (with a mass fraction of 95 % Pt and a mass fraction of 5 % Au 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 types 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 (e.g. 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.

7.3 Helium, purity $\geq 99,996$ % by volume.

7.4 Nitrogen, purity $\geq 99,996$ % by volume.

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 shall be corrected.

Spectral artefacts, e.g. escape peaks, sum peaks, pulse pile up lines, dead time, and continuous radiation 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. For quantitative analysis, the same sample preparation procedure is applied to both the standards and the samples to be analysed.

9 Sample preparation

9.1 General

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 shall be prepared. The pressed pellet method should be applied for the quantification of trace elements and shall be applied for the quantification of volatile elements, and the fused bead method should be applied 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 shall be used. If not otherwise specified, pre-treatment and preparation of test portions should be carried out according to the appropriate clauses of e.g. ISO 11464, EN 15002 or EN 16179. The particle size of the sample can strongly affect the precision of the measurement. The particle size should preferably be smaller than 150 µm.

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

If not otherwise specified, the determination of the dry mass should be carried out according to e.g. ISO 11465 or EN 15934.

9.3 Preparation of pressed pellet

After drying (6.3) and milling or grinding the sample (6.4), a pellet is prepared in the pellet press (6.5). Before pressing, the sample shall be mixed and homogenized with a binder (7.1) in a ratio of sampler:binder of 10:1 by mass. For the preparation of 40 mm in diameter pellets, approximately 10,0 g of sample is taken; for 32 mm in diameter pellets, approximately 4,5 g of sample is required. The amount of binder in the pellet shall be taken into account for the dilution factor. The sample should be pressed in an aluminium cup (6.6) 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 (6.3) and milling or grinding the sample (6.4), a fused bead is prepared using the fusion apparatus (6.7).

Ignite the sample at $1\,025\text{ °C} \pm 25\text{ °C}$ until constant mass is reached. Determine the loss on ignition at the chosen temperature to correct for volatile elements or compounds being released during ignition of the sample, or both.

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

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 mass. For the preparation of 40 mm in diameter beads, approximately 1,6 g of ignited sample is taken; for 32 mm in diameter beads, approximately 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.

Non-ignited material can be used to prepare beads but, nevertheless, loss of ignition shall be determined and shall 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 4 Different dilution factors can be used.

After fusion in a platinum-gold crucible (6.8) the melt is poured into a casting mould (6.9) to make a bead.

Beads can deteriorate because of adverse temperature and humidity conditions, so they should be stored in desiccators.

10 Procedure

10.1 Analytical measurement conditions

10.1.1 Wavelength dispersive instruments

The suggested analytical lines and operating conditions are given in [Table C.1](#) of [Annex C](#). 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.

10.1.2 Intensities and background corrections

For the determination of trace elements, the measured intensities shall 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 as in [Formula \(1\)](#):

$$I = I_p - I_b \quad (1)$$

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.

10.1.3 Counting time

The minimum counting time is the time necessary to achieve an uncertainty (2σ %), 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 \(2\)](#):

$$t = \frac{100}{2\sigma \%} \cdot \frac{1}{\sqrt{I_p - I_b}} \quad (2)$$

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.

10.1.4 Energy dispersive instruments

The suggested analytical lines and operating conditions are given in [Table C.2](#) of [Annex C](#). 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.

10.1.5 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.

10.2 Calibration

10.2.1 General

The calibration procedure is similar for energy dispersive and wavelength dispersive techniques. In general calibration is established by using matrix-adapted reference materials. The calibration equations and inter-element corrections are calculated by the software of the instrument. An accuracy check is performed with CRMs or samples with known composition.