



DRAFT International Standard

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

ICS: 13.080.10

ISO/DIS 18227

ISO/TC 190/SC 3

Secretariat: DIN

Voting begins on:
2024-09-23

Voting terminates on:
2024-12-16

iTeh Standards
(<https://standards.iteh.ai>)
Document Preview

[oSIST prEN ISO 18227:2024](https://standards.iteh.ai/catalog/standards/sist/0e4c47d1-f122-4c25-a5b6-7267e1301425/osist-pren-iso-18227-2024)

<https://standards.iteh.ai/catalog/standards/sist/0e4c47d1-f122-4c25-a5b6-7267e1301425/osist-pren-iso-18227-2024>

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 18227:2024(en)

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENTS AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

© ISO 2024

ISO/DIS 18227:2024(en)

iTeh Standards (<https://standards.itih.ai>) Document Preview

[oSIST prEN ISO 18227:2024](https://standards.itih.ai/catalog/standards/sist/0e4c47d1-f122-4c25-a5b6-7267e1301425/osist-pren-iso-18227-2024)

<https://standards.itih.ai/catalog/standards/sist/0e4c47d1-f122-4c25-a5b6-7267e1301425/osist-pren-iso-18227-2024>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2024

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

ISO/DIS 18227:2024(en)

Contents

	Page
Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Safety remarks	3
5 Principle	3
6 Apparatus	3
7 Reagents	4
8 Interferences and sources of error	5
9 Sample preparation	5
9.1 General.....	5
9.2 Drying and determination of dry mass.....	5
9.3 Preparation of pressed pellet.....	6
9.4 Preparation of fused beads.....	6
10 Procedure	7
10.1 Analytical measurement conditions.....	7
10.1.1 Wavelength dispersive instruments.....	7
10.1.2 Intensities and background corrections.....	7
10.1.3 Counting time.....	7
10.1.4 Energy dispersive instruments.....	7
10.1.5 Intensities and background corrections.....	7
10.2 Calibration.....	8
10.2.1 General.....	8
10.2.2 General calibration procedure.....	8
10.2.3 Internal standard correction using Compton (incoherent) scattering method.....	8
10.2.4 Fundamental parameter approach.....	9
10.2.5 Fundamental or theoretical influence coefficient method.....	9
10.2.6 Empirical alpha correction.....	10
10.2.7 Calibration procedure for trace elements using the pressed pellet method.....	10
10.2.8 Calibration procedure for major and minor oxides using the fused bead method.....	12
10.3 Analysis of the samples.....	13
11 Quality control	13
11.1 Drift correction procedure.....	13
11.2 Blank test.....	13
11.3 Reference materials.....	13
12 Calculation of the result	14
13 Test report	14
Annex A (informative) Semi-quantitative screening analysis of waste, sludge and soil samples	15
Annex B (informative) Examples for operational steps of the sample preparation for soil and waste samples	18
Annex C (informative) Suggested analytical lines, crystals and operating conditions	23
Annex D (informative) List of reference materials applicable for XRF analysis	25
Annex E (informative) Validation	26
Bibliography	36

ISO/DIS 18227:2024(en)

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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO [had/had not] received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190, Soil quality, Subcommittee SC 3, Chemical and physical characterization.

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

The main changes compared to the previous edition 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.

ISO/DIS 18227:2024(en)

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, 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 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 material 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.

iTeh Standards
(<https://standards.iteh.ai>)
Document Preview

[oSIST prEN ISO 18227:2024](#)

<https://standards.iteh.ai/catalog/standards/sist/0e4c47d1-f122-4c25-a5b6-7267e1301425/osist-pren-iso-18227-2024>

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

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

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 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 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.

ISO/DIS 18227:2024(en)**3.6****drift correction monitor**

physically stable sample used to correct for instrumental drift

3.7**emitted sample X-rays**

radiation emitted by 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

Note 1 to entry: This is expressed in units of 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 5725-2:2019, x.xx]

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

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

ISO/DIS 18227:2024(en)

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 European/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 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 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 (such as source filters, secondary targets, polarizing targets, collimators, focussing 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). 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.

ISO/DIS 18227:2024(en)

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 °C and 1 250 °C.

6.8 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.

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

7 Reagents

The reagents mentioned are used as carrier material.

oSIST prEN ISO 18227:2024

7.1 Binder: liquid or solid binder free of analytes of interest. <https://standards.iteh.ai/> <https://standards.iteh.ai/standards/iso/18227/2024-05/iso-18227-2024-a5b6-7267e1301425/osist-pren-iso-18227-2024>

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

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

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

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