International Standard

ISO 13196

# Soil quality — Screening soils for selected elements by energy-dispersive X-ray fluorescence spectrometry using a handheld or portable instrument

*Qualité du sol — Diagnostic rapide d'une sélection d'éléments dans les sols à l'aide d'un spectromètre de fluorescence X à dispersion d'énergie de type mobile ou pistolet*

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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).

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This second edition cancels and replaces the first edition (ISO 13196:2013), which has been technically revised.

The main changes are as follows:

— the details of measurement options have been made clearer;

— suitable materials for the equipment for sampling and sample preparation are indicated;

— more information is provided for users of this document on requirements for:

  — sieve;

  — sample cup;

  — basic operation of XRF spectrometers;

  — safety instructions;

  — sample preparation.

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 spectrometry (XRF) using battery or active source-powered handheld or portable equipment is a quick method for the determination of total elemental compositions of soil samples. Unlike laboratory analyses by inductively coupled plasma optical emission spectroscopy (ICP-OES) and atomic absorption spectroscopy (AAS), XRF needs no digestion step to prepare a test solution to be analysed. Consequently, handheld or portable equipment of energy dispersive XRF (ED-XRF) is suitable for the rapid on-site determination of selected elements, mainly heavy metals, in screening processes. When performing analyses at a site, it can be important to have information on the presence of an element (qualitative analysis) and also to obtain results from semi-quantitative analysis. Typical elements that can be detected and measured are Cr, As, Se, Cd, Hg and Pb, depending on the instrument (the elements validated for XRF detection and measurement are listed in Note 3 in Clause 1). In these situations, factory pre-set calibrations are used. For quantitative results, complementary analysis by alternative means is needed.

An ED-XRF exercise can comprise a single determination at one location, in accordance with the guidance in this document, several determinations, or a large number of determinations.

Where XRF analysers are being used to assess concentrations of soil contaminants which are harmful to humans or the environment, or both, there can be applicable national regulations with frameworks of standards, guidance and codes of practice for such investigations.

This document does not aim to provide a strategy, tactics or methodology for environmental investigations, or human health assessments of potentially contaminated land or soil, nor does it provide any such strategies for the assessment of mineral resources.

Adherence to this document does not demonstrate compliance with any national contaminated land investigation regulations.

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# Soil quality — Screening soils for selected elements by energy-dispersive X-ray fluorescence spectrometry using a handheld or portable instrument

**WARNING — Soil samples can contain toxic contaminants. Avoid direct contact of soil samples with exposed parts of the body. Appropriate measures shall be taken to avoid ingestion and inhalation.**

**Exposure to X-rays can give rise to radiation damage throughout the body as well an increased risk of cancer. XRF spectrometers are usually required to comply with national regulations. Those using, managing or supervising the use of such equipment are usually required to be qualified to do so in accordance with national regulations.**

**The XRF spectrometer to be used in accordance with this document shall employ a fail-safe function to prevent the operator and the public from inadvertent exposure to the X-ray beams. XRF users should engage a radiation protection officer to look at their proposed activity with the XRF spectrometer and provide informed advice on the safety implications of those proposals.**

**For in-situ (including strictly in-situ) analysis, a safe working area or controlled area should be established by signs and barriers, if necessary, in order to ensure bystanders are kept at a safe distance.**

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## 1 Scope

This document specifies the procedure for screening soils for selected elements using handheld or portable equipment for energy dispersive X-ray fluorescence spectrometry (ED-XRF). It covers the application of this screening method to obtain qualitative or semi-quantitative data to assist decisions on a sampling strategy for detailed assessment of soil quality employing laboratory analytical chemical methods.

NOTE 1 Screening methods generally provide qualitative or semi-quantitative concentration values that are indicative of concentration values, although occasionally they can give quantitative results under specific or limited conditions.

NOTE 2 The greater the effort applied to the pretreatment of soil samples, the better the analytical results that can be expected (see e.g. Reference [19]).

This document does not explicitly specify elements for which it is applicable, since the applicability depends on the performance of the apparatus and the objective of the screening. The elements which can be determined are limited by the performance of the instrument used, the concentrations of particular elements present in the soil, and the requirements of the investigation in terms of the minimum concentrations of concern (e.g. guideline value).

NOTE 3 The XRF measurements of As, Cd, Co, Cr, Cu, Hg, Mo, Ni, Pb, Sb, Sn, V and Zn were validated as described in Annex A.

NOTE 4 Annex B provides examples of when screening with a handheld ED-XRF spectrometer and a portable ED-XRF spectrometer can be useful.

This document does not provide guidance on how to use the equipment to provide quantitative data for use in detailed site assessments. This document does not cover how the results of multiple determinations are synthesized to address the objectives of an ED-XRF determination.

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## 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 12404, *Soil and waste — Guidance on the selection and application of screening methods*

IEC 62495, *Nuclear instrumentation — Portable X-ray fluorescence analysis equipment utilizing a miniature X-ray tube*

## 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**
**X-ray fluorescence spectrometer**
**XRF spectrometer**
spectrometer to observe X-ray fluorescence emitted from elements for analysis

Note 1 to entry: In this document, X-ray fluorescence spectrometer (XRF spectrometer) means energy dispersive X-ray fluorescence spectrometer (ED-XRF spectrometer).

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**3.2**
**handheld XRF spectrometer**
*XRF spectrometer* (3.1) which can be used for in-situ analysis (e.g. *strictly in-situ measurement* (3.11), *in-situ measurement* (3.12)) by handheld operation

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Note 1 to entry: Handheld XRF spectrometers are applicable to both in-situ measurements (including strictly in-situ measurements) and measurements with sampling or *post-sampling measurements* (3.13) where post-sampling measurement means application of determination methods including XRF to samples which are collected and pre-treated, if needed, at a site or in a laboratory.

Note 2 to entry: When applying an XRF spectrometer to samples at a spot just after collecting them from the ground thereat, the operation is still in-situ measurement.

**3.3**
**portable XRF spectrometer**
XRF spectrometer for samples taken out of a spot at a site, which can be transported to the site

Note 1 to entry: Portable XRF spectrometers are applicable to *in-situ measurements* (3.12) and to *post-sampling measurements* (3.13).

Note 2 to entry: Handheld XRF spectrometers (3.2) can be used at the bench top. However, the device works for handheld operation as designated in 3.2.

**3.4**
**fundamental parameter approach**
method to obtain element composition through successive approximation of the theoretical X-ray fluorescence intensities to the measured X-ray fluorescence intensities

Note 1 to entry: The calculation of the theoretical X-ray fluorescence intensities is carried out based on assumed element composition, theoretical parameters and pre-determined sensitivity coefficients of the XRF spectrometer.

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**3.5**
**screening**
application of any analytical *semi-quantitative* (3.8) method for exploratory analysis

[SOURCE: ISO 12404:2021, 3.1]

**3.6**
**screening method**
method which is used (often on site) to quickly explore a given area including target parameter distribution or to test a set of samples and obtain data on sample characteristics

Note 1 to entry: It is not necessarily directly comparable with *reference methods* (3.7).

[SOURCE: ISO 12404:2021, 3.2]

**3.7**
**reference method**
method which is performed in accordance with national or international standards

[SOURCE: ISO 12404:2021, 3.3]

**3.8**
**semi-quantitative**
approximate and comparative rather than absolutely quantitative

**3.9**
**semi-quantitative analysis**
data analysis method that provides approximate and comparative measurements rather than absolute quantification within a single experiment

Note 1 to entry: It combines elements of qualitative and quantitative analysis, allowing for the interpretation of numerical values that reflect the degree or extent of a particular characteristic within a sample. Unlike fully quantitative methods, which yield results that can be directly compared across different experiments, *semi-quantitative* (3.8) methods provide information that is meaningful primarily within the context of a single experiment or study.

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**3.10**
**qualitative analysis**
data analysis method that focuses on detecting or identifying constituent elements without providing specific concentrations

**3.11**
**strictly in-situ measurement**
directly observing the ground surface with a handheld device

**3.12**
**in-situ measurement**
observing at the sampling location samples collected from the ground, with a handheld or portable device

**3.13**
**post-sampling measurement**
observing, with a handheld or portable device, samples which are collected from the ground and then taken to another place (e.g. sampling stations or laboratories) after collection

Note 1 to entry: On-site measurement includes *strictly in-situ measurements* (3.11) and *in-situ measurements* (3.12) as well as post-sampling measurement when the measurement is carried out at the site but at a place different from the sample collection spot after collection (e.g. sampling stations at a site).

Note 2 to entry: As strictly in-situ and in-situ measurements are carried out applying an XRF device to the surface of a target ground spot and to a sample collected therefrom immediately after collection, they are in other words surface measurement and spot measurement, respectively.

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