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Environmental solid matrices — Determination of elements using inductively coupled plasma optical emission spectrometry (ICP-OES)

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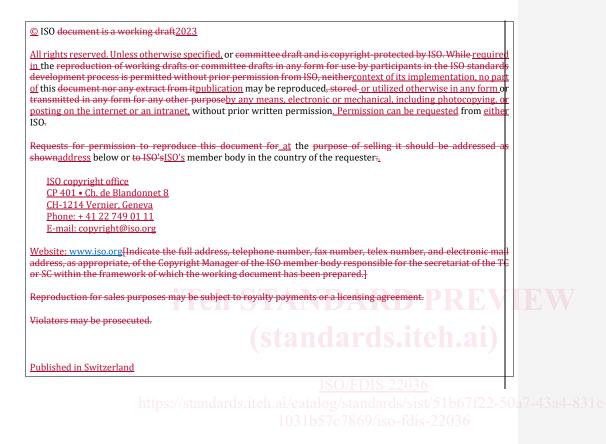   v

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 documentsdocument 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).

Attention is drawnISO draws attention to the possibility that some of the elementsimplementation of this document may beinvolve the subjectuse 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 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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see).

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,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 444, *Environmental characterization,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 22036:2008), which has been technically revised.

The main changes are as follows:

— Thethe content of ISO 22036:2008 and EN 16170:2017 has been merged;

— The scopethe Scope has been widened to include treated biowaste, waste, sludge and sediment;

— Thethe document has been developed parallel with CEN according to the Vienna Agreement;

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— applicable digestion and extraction methods have been updated;

— the text has been editorially 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

This document is applicable and validated for several types of matrices as indicated in Table 1~~Table 1~~ (see Annex A~~Annex A~~ for the results of validation).

Table 1 — Matrices for which this International Standard is applicable and validated

Matrix	Materials used for validation
Sludge	Municipal sludge Industrial sludge Sludge from electronic industry Ink waste sludge Sewage sludge
Biowaste	Compost Composted sludge
Soil	Agricultural soil Sludge amended soils
Waste	City waste incineration fly ash ("oxidised" matrix) City waste incineration bottom ash ("silicate" matrix) Ink waste sludge (organic matrix) Electronic industry sludge ("metallic" matrix) BCR 146R (sewage sludge) BCR 176 (city waste incineration ash)
Sediments	ISE 859 (Sediment from de Bilt / Netherlands)

The choice of calibration method depends on the extractant and can be adapted to the extractant concentration.

Environmental solid matrices — Determination of elements using inductively coupled plasma optical emission spectrometry (ICP-OES)

WARNING — Persons using this document should be familiar with usual laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This document specifies a method for the determination of the following elements in aqua regia, nitric acid or mixture of hydrochloric (HCl), nitric (HNO_3) and tetrafluoroboric (HBF_4)/hydrofluoric (HF) acid digests of soil, treated biowaste, waste, sludge and sediment:

Aluminium (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), bismuth (Bi), boron (B), cadmium (Cd), calcium (Ca), cerium (Ce), chromium (Cr), cobalt (Co), copper (Cu), dysprosium (Dy), erbium (Er), europium (Eu), gallium (Ga), gadolinium (Gd), germanium (Ge), gold (Au), hafnium (Hf), holmium (Ho), indium (In), iridium (Ir), iron (Fe), lanthanum (La), lead (Pb), lithium (Li), lutetium (Lu) magnesium (Mg), manganese (Mn), mercury (Hg), molybdenum (Mo), neodymium (Nd), nickel (Ni), palladium (Pd), phosphorus (P), platinum (Pt), potassium (K), praseodymium (Pr), rhodium (Rh), ruthenium (Ru), samarium (Sm), scandium (Sc), selenium (Se), silicon (Si), silver (Ag), sodium (Na), strontium (Sr), sulfur (S), tantal (Ta), tellurium (Te), terbium (Tb), thallium (Tl), thulium (Tm), thorium (Th), tin (Sn), titanium (Ti), tungsten (W), vanadium (V), yttrium (Y), ytterbium (Yb), zinc (Zn) and zirconium (Zr).

The method is also applicable to other extracts or digests originating from, ~~e.g.~~for example, DTPA extraction, fusion methods or total digestion methods, provided the user has verified the applicability.

The method has been validated for the elements given in Table A.1~~Table A.1~~ (sludge), Table A.2~~Table A.2~~ (compost) and Table A.3~~Table A.3~~ (soil). The method is applicable for other solid matrices and other elements as listed above, provided the user has verified the applicability.

This method is also applicable for the determination of major, minor and trace elements in aqua regia and nitric acid digests and in eluates of construction products (EN 17200[22])~~[19].~~).

NOTE   Construction products include e.g. mineral-based products; bituminous products; metals; wood-based products; plastics and rubbers; sealants and adhesives; paints and coatings.

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
blank calibration solution
solution prepared in the same way as the *calibration solution* (3.3(3.3)) but leaving out the analytes

3.2
blank test solution
solution prepared in the same way as the *test sample solution* (3.10(3.10)) but omitting the test portion

3.3
calibration solution
solution used to calibrate the instrument, prepared from *stock solutions* (3.8(3.8)) by adding acids, buffer, reference element and salts as needed

3.4
instrumental detection limit
lowest concentration that can be detected with a defined statistical probability using a clean instrument and a clean solution

3.5
laboratory sample
sample intended for laboratory inspection or testing

[SOURCE: ISO 11074:2015, 4.3.7, modified — Notes to entry have been removed.]

3.6
linearity
straight-line relationship between the mean result of measurement and the quantity (concentration) of the analyte

3.7
method detection limit
(MDL)
lowest concentration that can be detected using a specific analytical method with a defined statistical probability for defined maximum matrix element concentrations

3.8
stock solution
solution with accurately known analyte concentration(s), prepared from pure chemicals

3.9
test sample
portion of material, resulting from the *laboratory sample* (3.5(3.5)) by means of an appropriate method of sample pretreatment, and having the size (volume/mass) necessary for the desired testing or analysis

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[SOURCE: ISO 11074:2015, 4.3.16]

3.10
test sample solution
solution prepared after extraction or digestion of the *test sample* (3.9(3.9)) according to appropriate specifications

4 Principle

Inductively coupled plasma optical emission spectrometry (ICP-OES) can be used to determine elements in solution. The solution is dispersed by a suitable nebulizer; and the resulting aerosol is transported into the plasma. In a radio-frequency inductively coupled plasma, the solvent is evaporated; the dried salts are then vaporized, dissociated, atomized and ionized. The atoms or ions are excited thermally; and the number of photons emitted during transition to a lower energy level are measured with optical emission spectrometry. The spectra are dispersed by a grating spectrometer; and the intensities of the emission lines are monitored by photosensitive devices. The identification of the element takes place by means of the wavelength of the radiation (energy of photons), while the concentration of the element is proportional to the intensity of the radiation (number of photons). The ICP-OES method can be used to perform multi-element determinations using an optical system.

Annex BAnnex B shows examples of recommended wavelengths and detection limits for one particular instrument. Data given are valid for a synthetical soil matrix (500 mg/l Al, Ca, Fe in 30 ml aqua regia filled up to 100 ml with deionized water) with an optimized instrument. Using other instruments can lead to different detection limits. Adoption of other wavelengths is possible.

This document refers specifically to the use of ICP-OES. Users of this document are advised to operate their laboratories to accepted quality control procedures. Certified reference materials (CRM) should be used to establish the amounts of the relevant elements in in-house reference materials. The latter can be used for routine quality control of the procedures given in this document.

Results shall be established with control charts, for each element, within the laboratory. No result shall be accepted which falls outside an agreed limit. Quality control procedures based on widely accepted statistical techniques shall be used to establish such limits, that these are stable and that no long-term drift is occurring. CRM should be used regularly to maintain the integrity of the in-house reference materials and, thereby, the quality control system.

5 Interferences

5.1 General

The accurate and precise determination of trace element concentrations requires the correction of signal contributions not caused by the analyte of interest ('interferences'). Such interferences can result in both lower and higher results and thus shall be accounted for during analytical method development. 5.25.2 and 5.35.3 characterize possible interferences in ICP-OES and discuss procedures to detect and remedy their influence on the analytical result. Interferences are classified either as spectral or non-spectral.

5.2 Spectral interferences

Spectral interferences result in a change of the analyte instrumental signal, from a (partial) overlap of the analyte emission by emission lines or spectra of other sample constituents (direct spectral interference,

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inter-element or molecular (band) interference), by broad-band, continuous spectra, for example, from recombination of sample constituents, or by spectrally overlapping signals resulting from stray light or spectrally non-resolved molecular emissions.

Spectral interferences result in increased background signals that can obscure a weak analyte emission line completely. Accordingly, using spectrally interfered analyte emission lines can reduce the analytical capabilities and ultimately produce wrong results. Selecting a non-interfering analyte emission line, if available, can normally reduce or avoid deleterious spectral interference effects.

Broad-band spectral background emissions and stray light can normally be accounted for by subtraction of the background signal measured in immediate vicinity to the analyte emission line and extrapolated to the analyte wavelength position ("off-peak" background correction).

Spectral scans of samples compared with single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference."

Such measurements also show if the background signal is best determined based on the interpolation of a measurement on one side or on both sides of the analytical emission line or peak. The position selected for the background-intensity measurement, on one or both sides of the analytical line, is determined by the complexity of the spectrum adjacent to the analyte line. The position used should be as free as possible from spectral interference, and should reflect the same change in background intensity as occurs at the analyte wavelength measured.

For routine measurements, background measurement positions with no spectral off-peak interferences (as e.g. inter-element or molecular band interferences) shall be chosen, ensuring that the background signal measured off-peak is not interfered and allows an accurate background signal determination from its extrapolation to the analytical line wavelength position. If no interference-free off-peak background measurement position can be found, a suitable correction shall be applied to allow background signal determination from extrapolation of an off-peak background signal measurement.

Another possibility to avoid spectral interferences is the use of alternative emission wavelengths for the analyte of interest, if available. Finally, a correction of spectral interferences can also be performed by (typically multi-dimensional) mathematical spectral modelling approaches or (often iterative) correction formulae accounting for inter-element effects. To achieve accurate results with systems employing inter-element correction formulae, analyte and interfering elements shall be measured simultaneously. Spectral interferences that remain undetected and uncorrected lead to wrong positive results for the interfered analyte(s) in the sample under investigation. Table B.1Table B.1 lists recommended and alternative wavelengths of elements.

Modern ICP-OES systems are often equipped with multi-dimensional mathematical spectral modelling algorithms pre-set by the manufacturer for interference correction. Such approaches typically do not require the selection of dedicated wavelength positions for background signal measurements during method development, but instead utilize complete wavelength regions around the analyte emission line for modelling and correction. As with all interference correction techniques, the use of multi-dimensional spectral modelling algorithms requires a careful verification of their effectiveness and of the resulting accuracy, in the sample matrix of interest, to avoid wrong analytical results. This can, for example, be done by analysing matrix-matched samples of known analyte concentration(s), advisably in the range expected for the real samples or required from the analytical task at hand, e.g. the control of limiting values.

5.3 Non-spectral interferences

Apart from the spectral interferences described in 5.25.2, non-spectral interferences can also occur, reducing the analytical accuracy and precision if undetected and uncorrected for. Non-spectral interferences can be subdivided into physical, chemical and memory interferences.

Physical interferences are effects that occur in conjunction with sample transport and nebulization. Differences in sample viscosity or surface tension can result in significant interference effects, especially for samples with high concentrations of acids or dissolved solids. Physical interferences can be reduced by sample dilution, by adjustment of acid concentrations among the samples, by matrix matching or using suitable sample introduction equipment, e.g. 'slurry nebulizers' for samples with high TDS (total dissolved solids). Physical interferences can be corrected for by the application of suitable reference element within the analytical methodology.

The formation of molecular compounds, together with sample evaporation and ionization effects are all examples of chemical interferences. Excluding the easily ionizable elements effect (EIEE) relevant under axial plasma observation, chemical interferences typically do not occur significantly in ICP-OES techniques. However, should chemical interferences still arise, they can normally be minimized by a careful choice of the plasma parameters (e.g. RF power, observation height, nebulizer gas flow rate, 'robust plasma'), by suitably buffering the samples, by matrix-matching or by employing the method of standard additions. In general, chemical interferences are highly dependent on the type of sample matrix and the analyte element(s) of interest.

If large amounts of easily ionizable elements, for example, alkaline or earth-alkaline elements (I. and II. Group of the periodic table), reach the ICP, the plasma ionization equilibrium, i.e. the ratio of neutral atom to ion and electron number densities, can shift, resulting in a changed emission line excitation probability for neutral or ionized analyte atoms. Ultimately, this results in different analyte signals for the same amount of analyte in samples containing different amounts of easily ionizable elements. Especially for the easily ionizable elements themselves, the EIEE leads to substantial calibration function non-linearities that can result in significant analytical errors.

Since the EIEE is a plasma effect, its occurrence is independent from the plasma-viewing orientation (radially of axially) employed. However, its effect on the analytical result is dependent on the plasma-viewing orientation. For an axially viewed plasma, all processes along the analytical channel viewed contribute to the analytical signal; accordingly, the EIEE influence on the measurement results is relevant and shall be accounted and/or corrected for in any case. Possibilities to do so include the use of an ionization buffer (e.g. CsCl) added to all samples, or to employ radial plasma view for the elements affected. For radial view, where only a small plasma region contributes to the measurement signal, the influence of the EIEE on the analytical results can usually be ignored. However, radially viewing the plasma often also results in lower measurement sensitivities. Accordingly, such trade-offs in figures of merit shall be accounted for during any analytical method development.

Memory-Interferences ("memory effects") occur when the analyte signals of the current sample are influenced (usually resulting in positive deviations from the correct value) by the sample(s) measured before. Memory effects can result from sample deposits or accumulation in pump tubing, nebulizer, spray chamber or plasma torch. Occurrence of memory effects is element-specific and often can be reduced by rinsing the complete system thoroughly with a suitable clean rinse solution before the introduction of a new sample. Possible memory effects during analytical measurements shall be assessed and reduced, e.g. by an inter-sample rinse with a clean rinse solution for an appropriate time. The required rinse time is element-specific and shall be determined during method development, usually by defining a maximum