SIST

SLOVENSKI STANDARD
oSIST prEN ISO 22036:2022

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Tla, obdelani biološki odpadki in blato - Določevanje elementov z optično emisijsko spektrometrijo z induktivno sklopljeno plazmo (ICP/OES) (ISO/DIS 22036:2022)

Soil, treated biowaste and sludge - Determination of elements using inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO/DIS 22036:2022)

Boden, behandelter Bioabfall und Schlamm– Bestimmung von Elementen mittels optischer Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-OES) (ISO/DIS 22036:2022)

Sols, bio-déchets traités et boues - Dosage d'éléments par spectroscopie d'émission optique avec plasma induit par haute fréquence (ICP-OES) (ISO/DIS 22036:2022)

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Soil, treated biowaste and sludge — Determination of elements using inductively coupled plasma optical emission spectrometry (ICP-OES)

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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 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 document will supersede EN 16170:2017 and ISO 22036:2008.

Introduction

This document is applicable and validated for several types of matrices as indicated in Table 1 (see 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
Biowaste	Compost
Soil	Soil

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Soil, treated biowaste and sludge — 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 digest or extraction solutions of soil, treated biowaste and sludge:

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), gallium (Ga), indium (In), iron (Fe), lanthanum (La), lead (Pb), lithium (Li), magnesium (Mg), manganese (Mn), mercury (Hg), molybdenum (Mo), neodymium (Nd), nickel (Ni), phosphorus (P), potassium (K), praseodymium (Pr), samarium (Sm), scandium (Sc), selenium (Se), silicon (Si), silver (Ag), sodium (Na), strontium (Sr), sulfur (S), tellurium (Te), thallium (Tl), thorium (Th), tin (Sn), titanium (Ti), tungsten (W), vanadium (V), zinc (Zn) and zirconium (Zr).

This multi-element determination method is applicable to extracts obtained with nitric acid or aqua regia. The method is also applicable for other extracts or digests originating from e.g. DTPA extraction, fusion methods or total digestion methods, provided the user has verified the applicability[1-8].

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

The method has been validated for the elements given in [Annex A](). The method is applicable for the other elements listed above, provided the user has verified the applicability.

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.

EN ISO 11074, *Soil quality — Vocabulary*

3 Terms and definitions

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

ISO and IEC maintain terminological 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 but leaving out the analytes

3.2
blank test solution
solution prepared in the same way as the test sample solution but omitting the test portion

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

3.4
instrument 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: EN ISO 11074:2015, 4.3.7]

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

3.7
method detection limit
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 (6.4)

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

[SOURCE: EN ISO 11074:2015, 4.3.16]

3.10
test sample solution
solution prepared after extraction or digestion of the test sample 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.

Table B.1 in Annex 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 International Standard refers specifically to the use of ICP-OES. Users of this International Standard 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 International Standard.

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. Certified Reference Materials should be used regularly to maintain the integrity of the in-house reference materials and, thereby, the quality control system.

5 Interferences

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 have to be accounted for during analytical method development. The following paragraphs characterize possible interferences in ICP-OES and discuss procedures to detect and remedy their influence on the analytical result. Interferences are classified either as *non-spectral* or *spectral*.

5.1 Spectral interferences

Spectral interferences often synonymously called *matrix effects*. Spectral interferences result in a change of the analyte signal, from a (partial) overlap of the analyte emission by emission lines or spectra of other sample constituents (direct spectral interference, inter-element or molecular (band) interference), by broad-band, continuous spectra, e.g. 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 even 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. The alternative choice of a non-interfered 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). Additionally, measurements of the sample spectrum and comparison to single element spectra in the relevant wavelength regions can often indicate if alternative analyte emission lines might be better suited in the sample matrix at hand, due to lesser or no spectral interference at the alternative wavelength position.

Such measurements also show if the background signal is best determined based on the interpolation of a measurement on one side of the analytical emission line, or by interpolating the signal determined at two background locations, on either side of the analytical peak. Therefore, the wavelength position(s) or region(s) chosen for background signal determination in an analytical method is/are ultimately determined by the structure and complexity of the sample emission spectrum in the vicinity of the analytical emission line.

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For routine measurements, background measurement positions with no spectral off-peak interferences (as e.g. inter-element or molecular band interferences) have to 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 has to 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 effected by (typically multi-dimensional) mathematical spectral modelling approaches or (often iterative) correction equations accounting for inter-element effects. To achieve accurate results with systems employing inter-element correction equations, analyte and interfering elements have to 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.1 lists recommended wavelengths of elements and common spectral interferences could be identified.

Modern ICP-OES systems are often equipped with manufacturer pre-set multi-dimensional mathematical spectral modelling algorithms 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, also 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 e.g. be done by analyzing 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.

5.2   Non-spectral interferences

Apart from the *spectral* interferences described before, also non-spectral interferences can 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 by the use of suitable sample introduction equipment, e.g. 'slurry nebulizers' for samples with high TDS. 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', etc.), 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.

Easily Ionizable Elements Effect (EIEE): If large amounts of easily ionizable elements, e.g. 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.