

TECHNICAL SPECIFICATION
SPÉCIFICATION TECHNIQUE
TECHNISCHE SPEZIFIKATION

CEN/TS 17770

April 2022

ICS 65.080

English Version

**Organic and organo-mineral fertilizers - Determination of
the total content of specific elements by ICP-AES after
digestion by aqua regia**

Engrais organiques et organo-minéraux -
Détermination de la teneur totale en éléments
spécifiques par ICP-AES après digestion à l'eau régale

Organische und organisch-mineralische Düngemittel -
Bestimmung des Gesamtgehaltes spezifischer
Elemente durch ICP-AES nach Aufschluss durch
Königswasser

This Technical Specification (CEN/TS) was approved by CEN on 13 March 2022 for provisional application.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (CEN/TS 17770:2022) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

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CEN/TS 17770:2022 (E)**Introduction**

This document is part of a modular approach and concerns the analytical measurement step. “Modular” means that a test standard concerns a specific step in assessing a property and not the whole chain of measurements. Inductively coupled plasma atomic emission spectrometry (ICP-AES) is nowadays widely used and a well-established method in many laboratories.

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1 Scope

This document specifies a method for the determination of elements in organic fertilizers and organo-mineral fertilizers digests using inductively coupled plasma-atomic emission spectrometry (ICP-AES).

NOTE Alternatively, inductively coupled plasma mass spectrometry (ICP-MS) can be used for the measurement if the user proves that the method gives the same results.

This method is applicable to aqua regia digests prepared according to CEN/TS 17768 for the determination of P, K, Ca, Mg, Na, S, B, Co, Cu, Fe, Mn, Mo, Zn, As, Cd, Cr, Ni, Pb by ICP-AES. The method can be used for the determination of other elements, 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 11885:2009, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885:2007)*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

4 Principle

The method is based on the inductively coupled plasma atomic emission spectrometry (ICP-AES) measurement of the concentration of elements in organic fertilizers and organo-mineral fertilizers in aqua regia digests. The elements are determined after an appropriate dilution of the digest, if necessary. The solution is dispersed by a suitable nebulizer of the ICP-AES instrument and the resulting aerosol is transported into the plasma torch. Element specific emission spectra are produced by a radiofrequency inductively coupled argon plasma where atoms or ions are excited at high temperature. The emission spectra are dispersed by a spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Multi-element determinations using sequential or simultaneous optical systems and axial, radial or dual viewing of the plasma may be used.

5 Interferences

5.1 General

Interferences and matrix effects shall be recognized and appropriate measures to minimize them shall be made. There are several types of interferences, see 5.2 to 5.6.

5.2 Spectral interferences

Spectral interferences are due to incomplete isolation of the radiation emitted by the analyte from other detected radiation sources. Spectral interferences are caused by the background emission from continuous or recombination phenomena, by stray light which causes background increase or overlap of a spectral line from another element, or by the unresolved overlap of molecular band spectra. Background

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emission and stray light can usually be compensated for by subtracting the background emission measured adjacent to the analyte wavelength peak. To correct a sloping background shift background correction points on each side of the peak are used. The increase of background is more intensive with axial-view instruments. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. A spectral line overlap usually leads to the choice of an alternative line. If this is not possible, mathematical correction procedures (e.g. inter-element correction technique or multi-component spectral fitting) can be used to compensate for the interference. These correction procedures are usually a part of the instrument software.

5.3 Transport interferences

Transport interferences are caused by differences in the properties between the sample solutions and the calibration solutions (viscosity, surface tension, density, dissolved solid content, type and concentration of acids). As a consequence, the supply of solution to the nebuliser, the efficiency of nebulisation and the droplet size distribution of the aerosol is altered, resulting in a change of sensitivity. Errors due to these interferences can be overcome by dilution of the solutions, by matrix matching, by standard addition or by internal standard.

5.4 Excitation interferences

Excitation interferences are attributed to a change in the excitation conditions in the plasma, especially by the presence of easily ionisable elements. The interference depends on the operating conditions of the plasma (e.g. power, sample introduction, gas flowrate or observation height) and differ from element to element. Improvement of the plasma conditions can therefore reduce excitation interferences. Other possibilities are dilution of the solutions, matrix matching or the standard addition technique.

5.5 Chemical interferences

Chemical interferences are not significant with the ICP-AES technique, but if observed, they can be minimized by a careful selection of operating conditions (e.g. radio frequency power, observation position, gas flow rate and so forth).

5.6 Memory interferences

Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. This type of interference can be caused by sample deposits or the accumulation in pump tubing, nebulizer, spray chamber or plasma torch. The possibility of memory interferences should be recognized within an analytical run and suitable rinse steps and rinse times should be used.

6 Reagents

6.1 General

All reagents including water shall be of recognized analytical grade and they shall have negligible concentration of the element to be determined if compared to the lowest concentration of that element in the sample solution.

6.2 Nitric acid, substance concentration $c(\text{HNO}_3) \approx 14,3 \text{ mol/l}$; mass concentration $\rho \approx 1,4 \text{ g/ml}$.

6.2.1 Diluted nitric acid solution, $c(\text{HNO}_3) = 5 \text{ mol/l}$.

Add 350 ml of nitric acid (6.2) to 650 ml of water.

6.3 Hydrochloric acid, $c(\text{HCl}) \approx 12 \text{ mol/l}$; $\rho \approx 1,18 \text{ g/ml}$.

6.4 Mixed acid solution, 0,8 mol/l nitric acid and 1,8 mol/l hydrochloric acid.

Add 150 ml of hydrochloric acid (6.3) and 56 ml nitric acid (6.2) to 800 ml of water.

6.5 Standard stock solutions, concentration of 1 000 mg/l for each element.

6.5.1 General

Both single-element stock solutions and multi-element stock solutions with adequate specification, stating the acid used and the preparation technique, are commercially available. Multi-element stock solutions are usually available at the individual mass concentration *equal to* 100 mg/l for each element. These solutions are considered to be stable for more than one year, but in reference to guaranteed stability, the recommendations of the manufacturer should be considered. Alternatively, the stock solutions may be prepared by the dissolution of high purity metals.

6.5.2 Standard solution, concentration of 100 mg/l of element.

Use commercially available solution of this concentration for each element or pipette, 10 ml of the appropriate element stock solution (6.5) into a 100 ml volumetric flask. Add 10 ml of diluted nitric acid (6.2.1), fill to the mark with water and mix well. This solution is used to prepare spiked test solutions and calibration solutions.

6.5.3 Standard solution, concentration of 10 mg/l of element.

Pipette 10 ml of standard solution (6.5.2) into a 100 ml volumetric flask. Add 10 ml of diluted nitric acid (6.2.1), fill to the mark with water and mix well. This solution is used to prepare calibration solutions e.g. for cadmium.

6.6 Argon, purity 99,995 % or better.

7 Apparatus

7.1 Common laboratory glassware.

7.2 Inductively coupled plasma atomic emission spectrometer.

WARNING – It is essential that the manufacturer's safety instructions are strictly observed when using this apparatus.

The inductively coupled plasma atomic-emission spectrometer consists of a sample introduction system, the plasma as an excitation source, an optical system, a detector and a computer with suitable software. The sample is transported by the introduction system (rotation tube pump, nebuliser and a spray chamber) to the plasma torch. Around the torch a water-cooled RF coil is placed. A frequency of 27 MHz to 56 MHz with a power of 600 W to 2 000 W is usually used. The emission from the plasma can be observed either from the side (radial) or from the torch central symmetrical axis (axial). Axial viewing gives more signal intensity due to the increased observation path length of the normal analytical zone of the plasma, but an increase of interference is also commonly observed. Spectral lines are measured and registered either in a sequential or a simultaneous manner.

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8 Procedure

8.1 Preparation of test and blank solution

Aqua regia digests are usually measured using ICP-AES directly with calibration solutions of the same final concentration of aqua regia. The aqua regia extracts may also be used for the determination of other elements.

A blank test is prepared for the measurement following the same procedure as for samples.

If dilution is necessary, dilute an aliquot portion of the extract in one or more steps so that the final concentration of the element to be determined is approximately in the middle of the given calibration range (8.2). In the final diluting step fill to the mark with the mixed acid solution (6.4) and mix well. Prepare a diluted blank solution by pipetting a blank solution and dilute in the same way as the test solution.

8.2 Preparation of the calibration solutions

Individual calibration series for each element or mixed series may be used. For cadmium calibration standards, pipette volumes of 0 ml; 0,5 ml; 1 ml; 2 ml and 5 ml of the standard solution (6.5.3) into five 100 ml volumetric flasks respectively. For arsenic, copper, chromium, lead, nickel and zinc calibration standards pipette volumes of 0 ml; 1 ml; 2 ml; 5 ml and 10 ml of the standard solution (6.5.2) into the same five 100 ml volumetric flasks respectively.

Fill the volumetric flasks to the mark with the mixed acid solution (6.4).

The mass concentrations of cadmium in the calibration solutions are: 0 mg/l; 0,05 mg/l; 0,1 mg/l; 0,2 mg/l and 0,5 mg/l. The mass concentrations of arsenic, copper, chromium, lead, nickel and zinc in the calibration solutions are: 0 mg/l; 1 mg/l; 2 mg/l; 5 mg/l and 10 mg/l respectively.

Individual sets of calibration solutions for each element may be used instead of the mixed calibration solutions for all elements. If necessary, calibration solutions of higher or lower concentrations may be prepared in the same way.

NOTE 1 Small differences in acid concentration between sample digests and calibration solutions do not affect the measurement.

NOTE 2 It is possible to calibrate the instrument for higher concentrations of the elements if the calibration curve is linear.

8.3 Measurement

8.3.1 Instrument conditions

Due to differences between various kinds of instruments, no detailed instructions can be given to operate the specific instrument. The instruction provided by the manufacturer for waiting time, instrument stability, gas flows, plasma conditions, nebuliser conditions, sample uptake rate, etc. should be followed.

Examples of conditions for two types of instruments are given in Table 1.

The software of the instrument is used to calculate concentrations of the elements in the individual test solutions. All test solutions, blanks and calibration solutions are measured under the same optimized conditions using background correction and other suitable steps to eliminate or minimize interferences.