
Soil quality — Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc in aqua regia extracts of soil — Flame and electrothermal atomic absorption spectrometric methods

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Qualité du sol — Dosage du cadmium, chrome, cobalt, cuivre, plomb, manganèse, nickel et zinc dans les extraits de sol à l'eau régale — Méthodes par spectrométrie d'absorption atomique avec flamme et atomisation électrothermique

ISO 11047:1998

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11047 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

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Annex A of this International Standard is for information only.

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Soil quality — Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc in aqua regia extracts of soil — Flame and electrothermal atomic absorption spectrometric methods

WARNING — The procedures in this International Standard should be carried out by competent, trained persons. Some of the techniques and reagents, including the use of equipment, are potentially very dangerous. Users of this International Standard who are not thoroughly familiar with the potential dangers and related safe practices should take professional advice **BEFORE** commencing any operation.

1 Scope

This International Standard specifies two methods for the determination, by atomic absorption spectrometry, of one or more of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc, in aqua regia extracts of soil obtained in accordance with ISO 11466. The choice of method for any element depends on the amount of that element expected to be in a sample, and both methods might be needed to cover all the elements in one sample. The methods are applicable when the extractable element content is above or below the amount given in table 1 (as milligrams per kilogram, dry matter), as appropriate. The two methods are described separately as follows:

Method A – Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc by flame atomic absorption spectrometry. <https://standards.iteh.ai/catalog/standards/sist/0c26271c-2975-495d-931c-bfb9014495ca/iso-11047-1998>

Method B – Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc by electrothermal atomic absorption spectrometry.

Many of the procedures and reagents are common to both methods, and to the different elements within each method. Users are advised, however, to read the whole standard carefully before embarking on any of the procedures.

Table 1 — Indicative limits of soil contents (mg/kg dry matter) for elements extractable in aqua regia in accordance with ISO 11446

Element	Method A (flame AAS)	Method B (electrothermal AAS)
Cadmium	> 2	< 2
Chromium	> 12	< 12
Cobalt	> 12	< 12
Copper	> 5	< 5
Lead	> 15	< 15
Manganese	> 2	< 2
Nickel	> 12	< 12
Zinc	> 2	< 2

NOTE 1 Indicative limits given in table 1 are appropriate to the equipment and techniques available at the time of publication of this International Standard. Technical improvements may change this position in the future.

NOTE 2 This International Standard refers specifically to the use of atomic absorption spectrometry. 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 should be established with control charts, for each element, within the laboratory. No result should be accepted which falls outside an agreed limit. Quality control procedures based on a widely accepted statistical technique should be used to establish such limit, to ensure 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.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 11465:1993, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

ISO 11466: 1994, *Soil quality — Extraction of trace elements soluble in aqua regia*

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3 Method A — Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc by flame atomic absorption spectrometry

3.1 Principle

The method is based on the atomic absorption spectrometric measurement of the concentration of the element in an aqua regia extract of the sample, prepared in accordance with ISO 11466, using the instrumental conditions given in table 2.

Table 2 — General analytical conditions for flame atomic absorption spectrometry

Element	Wavelength (nm)	Flame type	Lanthanum chloride	Main Interferences	Background correction
Cadmium	228,8	Oxidizing air/ acetylene	No	Fe	Deuterium
Chromium	357,9	Reducing air/ acetylene or acetylene/N ₂ O	Yes No	Fe, Al Ca, Mg	Halogen
Cobalt	240,7	Oxidizing air/ acetylene	No		Deuterium
Copper	324,8	Oxidizing air/ acetylene	No		Deuterium
Lead	217,0	Oxidizing air/ acetylene	No		Deuterium
Manganese	279,5	Oxidizing air/ acetylene or acetylene/N ₂ O	Yes No	Fe, Si	Deuterium
Nickel	232,0	Oxidizing air/ acetylene	No	Fe	Deuterium
Zinc	213,9	Oxidizing air/ acetylene	No		Deuterium

NOTE The wavelengths given are the most sensitive. Interferences are generally lower if the nitrous oxide flame is used for determination of chromium and manganese. Users should be aware that small changes in gas volume ratios can have significant effects on the intensity of the analytical signal and can also change the linearity of the instrument response. Also difference in acid strength, which will vary slightly from digest to digest, can have a measurable effect on some elements with some instruments especially if background correction is not used. Users should, therefore, familiarize themselves with these aspects of their instrument's performance.

3.2 Reagents

All reagents shall be of recognized analytical grade. Use deionized water or water distilled from an all-glass apparatus, conforming to grade 2 of ISO 3696.

The water used for blank determinations and for preparing reagents and standard solutions shall have a concentration of the element to be determined that is negligible compared with the lowest concentration of that element in the sample solutions.

3.2.1 Hydrochloric acid, 37 %; $\rho \sim 1,18$ g/ml

The same batch of hydrochloric acid shall be used throughout the procedure.

3.2.2 Nitric acid, 65 %; $\rho \sim 1,42$ g/ml

The same batch of nitric acid shall be used throughout the procedure.

3.2.3 Nitric acid, diluted 1 + 3 (V/V)

Add 250 ml of nitric acid (3.2.2) to 500 ml of water in a 1000 ml volumetric flask and fill to the mark with water.

3.2.4 Sulfuric acid, 98 %; $\rho \sim 1,84$ g/ml.

The same batch of sulfuric acid shall be used throughout the procedure.

3.2.5 Sulfuric acid, diluted 1 + 9 (V/V).

Add slowly with swirling or stirring (magnetic stirrer bar) 100 ml of sulfuric acid (3.2.4) to 700 ml of water in a 1000 ml volumetric flask, mix, cool and fill to the mark with water.

3.2.6 Lanthanum chloride solution, 37 g/l lanthanum.

Dissolve 100 g of lanthanum(II) chloride, $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$, in 700 ml water. Then quantitatively transfer it to a 1000 ml volumetric flask and fill to the mark with water.

3.2.7 Blank solution without lanthanum – for calibration

Dilute 210 ml of hydrochloric acid (3.2.1) and 70 ml of nitric acid (3.2.2) with 500 ml water in a 1000 ml volumetric flask and fill to the mark with water.

3.2.8 Blank solution with lanthanum – for calibration

Dilute 210 ml of hydrochloric acid (3.2.1) and 70 ml of nitric acid (3.2.2) with 500 ml water in a 1000 ml volumetric flask. Add 100 ml lanthanum chloride solution (3.2.6) and fill to the mark with water.

3.2.9 Acetone

3.3 Preparation of stock and standard solutions of individual elements

The stock solutions of all the elements shall be replaced after a maximum of one year, but the standard solutions shall be renewed monthly as a minimum. If stock solutions are prepared directly from metals, care needs to be taken to ensure that the metal used is free of surface oxide layers. Stock solutions of metals are available commercially, and may be used, but their use should be controlled by appropriate laboratory procedures which meet accepted good laboratory practice. Records shall be kept of such control data.

3.3.1 Cadmium solutions

WARNING — Cadmium is highly toxic. Safety measures shall be taken to avoid ingestion.

3.3.1.1 Cadmium, stock solution corresponding to 1000 mg/l of cadmium.

Weigh, to the nearest $\pm 0,0002$ g, approximately 1,0000 g of cadmium metal (minimum purity 99,5 %) and dilute it in a covered 250 ml glass beaker with 40 ml of nitric acid (3.2.3). Then add 100 ml of water. Boil to expel nitrous fumes, cool, transfer to a 1000 ml volumetric flask and fill to the mark with water.

3.3.1.2 Cadmium, standard solution corresponding to 20 mg/l of cadmium.

Pipette 20,00 ml of the stock cadmium solution (3.3.1.1) into a 1000 ml volumetric flask. Add 20 ml of nitric acid (3.2.3) and fill to the mark with water.

3.3.2 Chromium solutions

3.3.2.1 Chromium, stock solution corresponding to 1000 mg/l of chromium.

Dissolve $2,8290 \text{ g} \pm 0,0002 \text{ g}$ of potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$, dried at 130°C for 24 h, in a covered 400 ml glass beaker with 40 ml of water. Add 5 ml of sulfuric acid (3.2.4), cool, transfer to a 1000 ml volumetric flask and fill to the mark with water.

3.3.2.2 Chromium, standard solution corresponding to 20 mg/l of chromium.

Pipette 20,00 ml of the stock chromium solution (3.3.2.1) into a 1000 ml volumetric flask. Add 20 ml of nitric acid (3.2.3) and fill to the mark with water.

3.3.3 Cobalt solutions

3.3.3.1 Cobalt, stock solution corresponding to 1000 mg/l of cobalt.

Weigh, to the nearest $\pm 0,0002$ g, approximately 1,0000 g of cobalt metal (minimum purity 99,5 %) and dissolve it in a covered 250 ml glass beaker with 10 ml of nitric acid (3.2.3) and 10 ml of hydrochloric acid (3.2.1). Then add 100 ml of water, boil to expel nitrous fumes, cool, transfer to a 1000 ml volumetric flask and fill to the mark with water.

3.3.3.2 Cobalt, standard solution corresponding to 20 mg/l of cobalt.

Pipette 20,00 ml of the stock cobalt solution (3.3.3.1) into a 1000 ml volumetric flask, add 20 ml of nitric acid (3.2.3) and fill to the mark with water.

3.3.4 Copper solutions

3.3.4.1 Copper, stock solution corresponding to 1000 mg/l of copper.

Weigh, to the nearest $\pm 0,0002$ g, approximately 1,0000 g of copper metal (minimum purity 99,5 %) and dissolve it in a covered 250 ml glass beaker with 40 ml of nitric acid (3.2.3). Then add 100 ml of water, boil to expel nitrous fumes, cool, transfer to a 1000 ml volumetric flask and fill to the mark with water.

3.3.4.2 Copper, standard solution corresponding to 20 mg/l of copper.

Pipette 20,00 ml of the stock copper solution (3.3.4.1) into a 1000 ml volumetric flask. Add 20 ml of nitric acid (3.2.3) and fill to the mark with water.

3.3.5 Lead solutions

3.3.5.1 Lead, stock solution corresponding to 1000 mg/l of lead.

Weigh, to the nearest $\pm 0,0002$ g, approximately 1,0000 g of lead metal (minimum purity 99,5 %) and dissolve it in a covered 250 ml glass beaker with 40 ml of nitric acid (3.2.3). Then add 100 ml of water, boil to expel nitrous fumes, cool, transfer to a 1000 ml volumetric flask and fill to the mark with water.

3.3.5.2 Lead, standard solution corresponding to 20 mg/l of lead.

Pipette 20,00 ml of the stock lead solution (3.3.5.1) into a 1000 ml volumetric flask. Add 20 ml of nitric acid (3.2.3) and fill to the mark with water.

3.3.6 Manganese solutions

3.3.6.1 Manganese, stock solution corresponding to 1000 mg/l of manganese.

Clean manganese metal by transferring several grams of electrolytic manganese (minimum purity 99,5 %) to a 250 ml glass beaker containing about 150 ml of dilute sulfuric acid (3.2.5). Stir and allow the manganese to settle for several minutes. Decant, wash several times with water and finally with acetone (3.2.9). Decant the surplus acetone, dry the metal for 2 min at 105 °C and cool in a desiccator.

Weigh, to the nearest $\pm 0,0002$ g, approximately 1,0000 g of such cleaned manganese metal and dissolve it in a covered 250 ml glass beaker with 20 ml of hydrochloric acid (3.2.1) and 20 ml of nitric acid (3.2.3). Then add 100 ml of water, boil to expel nitrous fumes, cool, transfer to a 1000 ml volumetric flask and fill to the mark with water.

3.3.6.2 Manganese, standard solution corresponding to 20 mg/l of manganese.

Pipette 20,00 ml of the stock manganese solution (3.3.6.1) into a 1000 ml volumetric flask. Add 20 ml of nitric acid (3.2.3) and fill to the mark with water.

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3.3.7 Nickel solutions

3.3.7.1 Nickel, stock solution corresponding to 1000 mg/l of nickel.

Weigh, to the nearest $\pm 0,0002$ g, approximately 1,0000 g of nickel metal (minimum purity 99,5 %) and dissolve it in a covered 250 ml glass beaker with 10 ml of hydrochloric acid (3.2.1) and 10 ml of nitric acid (3.2.3). Then add 100 ml of water, boil to expel nitrous fumes, cool, transfer to a 1000 ml volumetric flask and fill to the mark with water.

3.3.7.2 Nickel, standard solution corresponding to 20 mg/l of nickel.

Pipette 20,00 ml of the stock nickel solution (3.3.7.1) into a 1000 ml volumetric flask. Add 20 ml of nitric acid (3.2.3) and fill to the mark with water.

3.3.8 Zinc solutions

3.3.8.1 Zinc, stock solution corresponding to 1000 mg/l of zinc.

Weigh, to the nearest $\pm 0,0002$ g, approximately 1,0000 g of zinc metal (minimum purity 99,5 %) and dissolve it in a covered 250 ml glass beaker with 40 ml of nitric acid (3.2.3). Then add 100 ml of water, boil to expel nitrous fumes, cool, transfer to a 1000 ml volumetric flask and fill to the mark with water.

3.3.8.2 Zinc, standard solution corresponding to 20 mg/l of zinc.

Pipette 20,00 ml of the stock zinc solution (3.3.8.1) into a 1000 ml volumetric flask. Add 20 ml of nitric acid (3.2.3) and fill to the mark with water.

3.4 Apparatus

3.4.1 Ordinary laboratory apparatus

All glassware shall be cleaned carefully before trace element determinations, e.g. by immersion in warm 5 % (V/V) aqueous nitric acid solution for a minimum of 6 h, followed by rinsing with water before use. The nitric acid shall be replaced each week.

NOTE It is convenient to keep separate sets of glassware for the determinations given in this International Standard, in order to reduce the possibility of within-laboratory contamination. Similarly, it may be convenient to carry out the acid cleaning step overnight.

3.4.2 Atomic absorption spectrometer

This shall be equipped with: a hollow cathode lamp or an electrode-less discharge lamp appropriate to the element of interest (operated at the current recommended for the lamp by the instrument manufacturer), a background correction system, a burner suitable for an air/acetylene or nitrous oxide/acetylene flame (operated according to the manufacturer's instructions). Deuterium background correction is the minimum technical specification acceptable for background correction for measurement wavelengths below 350 nm and a halogen lamp for measurement wavelengths above 350 nm. Other systems (e.g. Zeeman polarization, Smith-Hieftje) are equally acceptable and, in certain circumstances, can be superior.

WARNING — It is essential that the manufacturer's safety recommendations are strictly observed when using these flames.

3.5 Procedure

3.5.1 Test portion

Use aqua regia extracts of soil in accordance with ISO 11466.

3.5.2 Blank test

Carry out a blank test at the same time as the extraction with aqua regia using cleaned quartz sand instead of the soil sample and follow the same procedure, using the same quantities of all the reagents for the determination.

3.5.3 Preparation of the calibration solutions

Before each batch of determinations, prepare from the 20 mg/l element standard solution (see 3.3) at least five calibration solutions covering the range of concentrations to be determined, as below. Fresh calibration solutions for all these elements shall be prepared at least monthly.

3.5.3.1 Cadmium calibration solutions

Pipette 1,00 ml, 2,00 ml, 4,00 ml, 6,00 ml, 8,00 ml, 10,00 ml of cadmium standard solution (3.3.1.2) into a series of 100 ml volumetric flasks. Add to each flask 21 ml of hydrochloric acid (3.2.1) and 7 ml of nitric acid (3.2.2). Dilute to the mark with water and mix well.

These solutions correspond to cadmium concentrations of 0,2 mg/l, 0,4 mg/l, 0,8 mg/l, 1,2 mg/l, 1,6 mg/l and 2,0 mg/l, respectively.

3.5.3.2 Chromium calibration solutions

Pipette 5,00 ml, 10,00 ml, 20,00 ml, 30,00 ml and 40,00 ml portions of chromium standard solution (3.3.2.2) into a series of 100 ml volumetric flasks. Add to each flask 21 ml of hydrochloric acid (3.2.1) and 7 ml of nitric acid (3.2.2). Dilute to the mark with water and mix well.

For measurement with an air/acetylene flame, add 10 ml of lanthanum chloride solution (3.2.6) before diluting to the mark.

These solutions correspond to chromium concentrations of 1,0 mg/l, 2,0 mg/l, 4,0 mg/l, 6,0 mg/l and 8,0 mg/l, respectively.

3.5.3.3 Cobalt calibration solution

Pipette 5,00 ml, 10,00 ml, 20,00 ml, 30,00 ml and 40,00 ml portions of cobalt standard solution (3.3.3.2) into a series of 100 ml volumetric flasks. Add to each flask 21 ml of hydrochloric acid (3.2.1) and 7 ml of nitric acid (3.2.2). Dilute to the mark with water and mix well.

These solutions correspond to cobalt concentrations of 1,0 mg/l, 2,0 mg/l, 4,0 mg/l, 6,0 mg/l and 8,0 mg/l, respectively.

3.5.3.4 Copper calibration solutions

Pipette 5,00 ml, 10,00 ml, 20,00 ml, 30,00 ml and 40,00 ml portions of copper standard solution (3.3.4.2) into a series of 100 ml volumetric flasks. Add to each flask 21 ml of hydrochloric acid (3.2.1) and 7 ml of nitric acid (3.2.2). Dilute to the mark with water and mix well.

These solutions correspond to copper concentrations of 1,0 mg/l, 2,0 mg/l, 4,0 mg/l, 6,0 mg/l and 8,0 mg/l, respectively.

3.5.3.5 Lead calibration solutions

Pipette 5,00 ml, 10,00 ml, 20,00 ml, 30,00 ml and 40,00 ml portions of lead standard solution (3.3.5.2) into a series of 100 ml volumetric flasks. Add to each flask 21 ml of hydrochloric acid (3.2.1) and 7 ml of nitric acid (3.2.2). Dilute to the mark with water and mix well.

These solutions correspond to lead concentrations of 1,0 mg/l, 2,0 mg/l, 4,0 mg/l, 6,0 mg/l and 8,0 mg/l, respectively.