
**Soil quality — Determination of arsenic,
antimony and selenium in aqua regia
soil extracts with electrothermal or
hydride-generation atomic absorption
spectrometry**

*Qualité du sol — Dosage de l'arsenic, de l'antimoine et du sélénium
dans des extraits du sol à l'eau régale par spectrométrie d'absorption
atomique avec atomisation électrothermique ou génération d'hydrures*



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Published in Switzerland

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 20280 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Soil quality — Determination of arsenic, antimony and selenium in aqua regia soil extracts with electrothermal or hydride-generation atomic absorption spectrometry

WARNING — Certain procedures, reagents and apparatus used in this International Standard pose hazards, especially in connection with concentrated acids, toxic solutions of arsenic (As), antimony (Sb) and selenium (Se), and high-pressure gases. Users should ensure that they are familiar with the safety procedures necessary in such situations, and with any legal requirements (including waste disposal). If in any doubt, seek advice from the competent authorities.

1 Scope

This International Standard specifies methods for the determination of arsenic, antimony and selenium, in an aqua regia extract of soil obtained in accordance with ISO 11466, by electrothermal or hydride-generation atomic absorption spectrometry.

2 Normative references

The following referenced documents are indispensable for the application 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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 11464:2006, *Soil quality — Pretreatment of samples for physico-chemical analysis*

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

ISO 11466:1995, *Soil quality — Extraction of trace elements soluble in aqua regia*

3 Principle

Arsenic, antimony and selenium are extracted from soil samples with aqua regia according to ISO 11466. Arsenic, antimony and selenium are determined by electrothermal atomic absorption spectrometry (ETAAS), in which discrete volumes of sample solution are dispensed into a graphite tube. By increasing the temperature of this graphite tube stepwise, the processes of drying, thermal decomposition of the matrix and thermal dissociation into free atoms occur. The resulting absorption signal should (under optimum conditions) be a sharp symmetrical peak proportional to the element concentration in solution.

Alternatively, arsenic, antimony and selenium are determined by the hydride-generation technique (HGAAS). Arsenic and antimony are first pre-reduced in the aqua regia extract by a mixture of ascorbic acid and potassium iodide. Selenium is pre-reduced by hydrochloric acid at an elevated temperature. After that, the hydride formation occurs by reaction with a sodium borohydride solution. The hydrides are carried from the solution by an argon stream into a heated quartz cell and decomposed at 900 °C and then the atom concentration for arsenic and antimony is measured by atomic absorption spectrometry. There are several commercial systems available where the reaction of hydride formation occurs continuously (continuous flow or

flow injection systems) or stepwise (batch systems). All these techniques can be used, but adaptation of the working steps and reagent concentrations according to the advice of the manufacturer may be necessary. Refer to the manufacturer's recommendation for wavelengths and spectral band widths or see Table 1.

Table 1 — Measurement conditions for arsenic, antimony and selenium

Element	Wavelength nm	Spectral band width nm
Arsenic	193,7	0,7
Antimony	217,6	1,0
Selenium	196,0	1,0

4 Reagents

All reagents shall be of recognised analytical grade. Use demineralised 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 element concentrations that are negligible compared with the lowest concentration to be determined in the sample solutions.

4.1 Hydrochloric acid, $w(\text{HCl}) = 37\%$, $\rho(\text{HCl}) \approx 1,2 \text{ g/ml}$.

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

4.2 Hydrochloric acid, diluted (1 + 9).

Pour 500 ml of water into a 1 000 ml volumetric flask. Add 100 ml of hydrochloric acid (4.1) with caution, mix and fill to the mark with water.

4.3 Nitric acid, $w(\text{HNO}_3) = 65\%$, $\rho(\text{HNO}_3) \approx 1,4 \text{ g/ml}$.

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

4.4 Nitric acid, $c(\text{HNO}_3) = 0,5 \text{ mol/l}$.

Pour 500 ml of water into a 1 000 ml volumetric flask. Add 22 ml of nitric acid (4.3) with caution, mix and fill to the mark with water.

4.5 Aqua regia solution, diluted (1 + 9).

Pour 500 ml of water into a 1 000 ml volumetric flask. Add 75 ml of hydrochloric acid (4.1) and 25 ml of nitric acid (4.3) with caution, mix and fill to the mark with water.

4.6 Palladium/magnesium nitrate modifier solution.

Two sources of modifier solutions can be used:

- commercially available modifier solutions;
- modifier element solutions prepared in the laboratory from pure palladium (e.g. powder) or from stoichiometrically defined and dried salts.

Dissolve 0,30 g of palladium nitrate $[\text{Pd}(\text{NO}_3)_2]$ and 0,36 g of magnesium nitrate $[\text{Mg}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}]$ in a 100 ml volumetric flask, fill to the mark with 0,5 mol/l nitric acid (4.4) and mix.