INTERNATIONAL
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11466

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Soil quality — Extraction of trace elements soluble in *aqua regia*

Qualité du sol — Extraction des éléments en traces solubles dans l'eau régale

Reference number
ISO 11466:1995(E)

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 11466 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Annex A of this International Standard is for information only.

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Soil quality — Extraction of trace elements soluble in *aqua regia*

WARNING — Digestions with hydrochloric and nitric acid are potentially hazardous and safety glasses or goggles must be used.

The digestion should be carried out in a well-ventilated fume cupboard with the reflux digestion on a temperature-controlled heating apparatus. It is essential to add antibumping granules (or roughened glass beads) both to the blank and the samples to prevent bumping and loss of solution. It is important to maintain gentle reflux, both of the blank and the test samples, to avoid temperature fluctuations which could cause local superheating.

1 Scope

This International Standard specifies a method for the extraction, with *aqua regia,* of trace elements from soils and similar materials, prepared according to ISO 11464, and containing less than about 20 % (m/m) organic carbon according to ISO 10694. Materials containing more than about 20 % (m/m) organic carbon will require treatment with additional nitric acid (see note 8). The resulting solution is suitable for the determination of trace elements using appropriate atomic spectrometric techniques. With high solute concentrations in extract solutions, spectral interferences and background enhancement should be expected.

NOTE 1 *Aqua regia* will not totally dissolve most soils and similar materials, and the efficiency of extraction for particular elements differs from element to element. Such efficiency might also differ for the same element in different matrices. Users of this International Standard should carry out a programme of analysis using reference materials to assure themselves that the method given here is appropriate for their needs. Elements extractable in *aqua regia* cannot, therefore, be decribed as "totals"; conversely they cannot be regarded as the "bio-available" fraction, as the extraction procedure is too vigorous to represent any biological process.

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 the 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 10694:1995, *Soil quality — Determination of organic and total carbon after dry combustion (elementary analysis).*

ISO 11464:1994, *Soil quality — Pretreatment of samples for physico-chemical analyses.*

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

3 Principle

The dried sample (see note 2) is extracted with a hydrochloric/nitric acid mixture by standing for 16 h at room temperature, followed by boiling under reflux for 2 h. The extract is then clarified and made up to volume with nitric acid. The trace metal content of the extract can be determined in accordance with [1] in annex A.

NOTE 2 The soil should be prepared in accordance with ISO 11464. It is common to grind < 2 mm soil to < 150 µm prior to *aqua regia* digestion. Such grinding is designed to

a) give a more homogeneous sample from which a subsample is taken;

b) increase the efficiency of acid attack by increasing the surface area of the particles.

For some soils, experience has shown that there is little difference between the results before and after such grinding (see [2] in annex A). However, it is difficult to predict, with certainty, which soils will behave in this way. This is particularly important when considering soils containing relatively small amounts of potentially toxic elements, compared with materials which are geogenically enriched, or those considerably affected by anthropogenic activities. In both cases the amounts of such elements can be very large. The problem is exacerbated in materials with a wide range of particle size, especially if the element of interest is concentrated in one single size fraction, compared with soils of narrow particle size range. Users should also be aware that relatively little is known about the behaviour of these trace elements, under *aqua regia* extraction, in highly weathered soils such as oxisols and ultisols (see [3] in annex A).

Therefore, the user should verify that the use of ground or unground soil yields results suitable for the purpose of the investigation.

Whether ground or unground soil has been used shall be stated in the test report.

4 Reagents

4.1 General

The reagents used shall meet the purity requirements of the subsequent analysis. Their purity shall be verified by performing a blank test.

4.2 Water

The water used shall comply with grade 2 of ISO 3696, or better.

NOTE 3 Deionized water may be used, providing that it meets the requirements given above. It is recommended that the same batch of water is used throughout a given batch of determinations and that blank determinations are carried out.

4.3 Hydrochloric acid, $c(\text{HCl}) = 12{,}0$ mol/l, $\rho \approx 1{,}19$ g/ml.

4.4 Nitric acid, $c(\text{HNO}_3) = 15{,}8$ mol/l, $\rho \approx 1{,}42$ g/ml.

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

Dilute 32 ml of nitric acid (4.4) with water (4.2) to 1 litre.

5 Apparatus

Clean all the glassware by carefully immersing in warm nitric acid (4.5) for a minimum of 6 h and then rinse with water (4.2).

5.1 Grinding mill, capable of grinding dried soils, sludges and sediments to a size less than 150 µm without contamination by the elements to be determined (see note 2).

NOTE 4 A mill causing little or no contamination is essential, as is adequate cleaning between samples to eliminate memory effects. Both agate and zirconium oxide mills have been found suitable.

5.2 Test sieve, of aperture size 0,150 mm, e.g. test sieve with gauze cloth preferably made from plastics materials, e.g. nylon.

5.3 Desiccator, of nominal volume 2 litres.

5.4 Reaction vessel, of nominal volume 250 ml (see figure 1).

5.5 Reflux condenser, straight-through type, with conical ground-glass joints (see figure 1).

NOTE 5 Water-cooled condensers with a minimum effective length of at least 200 mm have been found suitable. The effective length is the internal surface which is in contact with the cooling water. The overall external length of such condensers is usually at least 365 mm.

5.6 Absorption vessel, non-return type (e.g. as shown in figure 2).

NOTE 6 The absorption vessel is only necessary when mercury is to be determined.

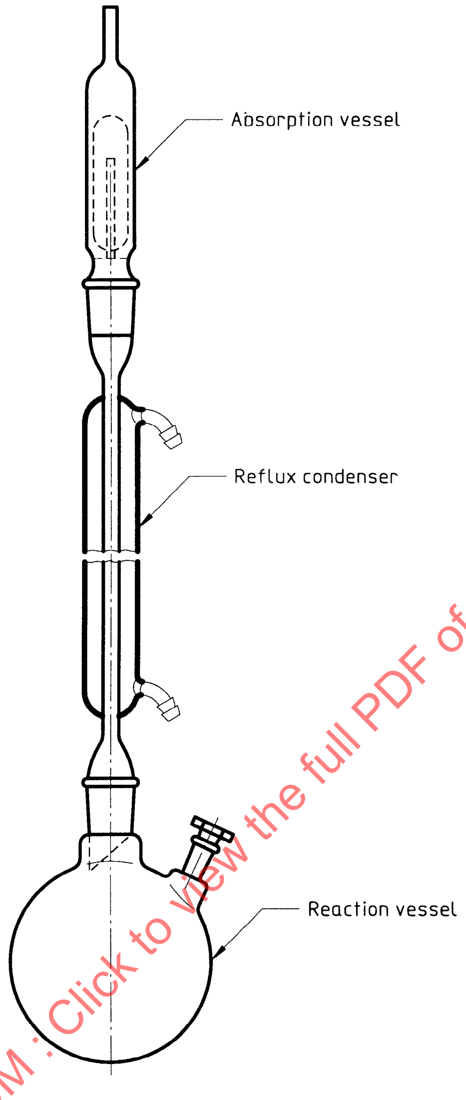

Figure 1 — Reaction vessel, reflux condenser and absorption vessel (assembled)

5.7 Roughened glass beads, diameter 2 mm to 3 mm (or anti-bumping granules).

5.8 Temperature-controlled heating apparatus, capable of heating the contents of the reaction vessel to reflux temperature.

5.9 Funnel, of diameter approximately 110 cm.

5.10 Volumetric flask, of nominal capacity 110 ml.

5.11 Filter papers, cellulose-based ashless types, with a medium pore size of approximately 8 µm and a diameter of 150 mm.

6 Preparation of laboratory sample

Pretreat a representative portion of the air-dried sample according to ISO 11464, or mill and sieve through the 150 µm sieve (5.2), if specified, to give a subsample of approximately 20 g. Use a portion of this subsample to determine the water content in accordance with ISO 11465.

7 Procedure

Weigh approximately 3 g, to the nearest 0,001 g, of the subsample into the 250 ml reaction vessel (5.4). Moisten with about 0,5 ml to 1,0 ml of water (4.2) and add, while mixing, 21 ml of hydrochloric acid (4.3) followed by 7 ml of nitric acid (4.4), drop by drop if necessary, to reduce foaming. Add 15 ml of nitric acid (4.5) to the absorption vessel (5.6). Connect the absorption vessel and condenser (5.5) to the reaction

Approximate dimensions in millimetres

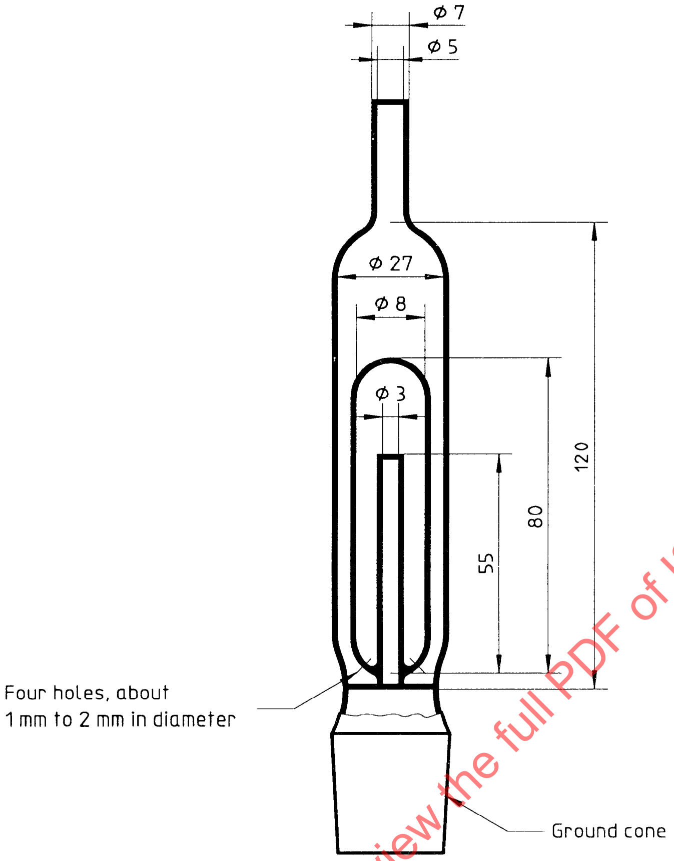

Figure 2 — Example of an absorption vessel

vessel, and allow to stand for 16 h at room temperature to allow for slow oxidation of the organic matter in the soil.

NOTE 7 It is common practice to add the hydrochloric acid directly to the reaction vessel and sample, followed by the nitric acid, and to assume that the *aqua regia* forms in the reaction vessel. However, problems have been encountered with materials rich in carbonates, where a substantial proportion of the hydrochloric acid has been consumed before the nitric acid can be added. Under such circumstances, the correct formation of *aqua regia* is doubtful.

NOTE 8 The amount of *aqua regia* is sufficient only for oxidation of about 0,5 g of organic carbon. If there is any doubt about the amount of carbon present, carry out a determination according to ISO 10694. If there is more than 0,5 g of organic carbon in the 3 g subsample, proceed as follows.

Allow the first reaction with the *aqua regia* to subside. Then add an extra 1 ml of nitric acid (4.4) only to every 0,1 g of organic carbon above 0,5 g. Do not add more than 10 ml of nitric acid at any time, and allow any reaction to subside before proceeding further.

Raise the temperature of the reaction mixture slowly until reflux conditions are reached and maintain for 2 h, ensuring that the condensation zone is lower than 1/3 of the height of the condenser, then allow to cool. Add the contents of the absorption vessel to the reaction vessel, via the condenser, rinsing both the absorption vessel and condenser with a further 10 ml of nitric acid (4.5).

Allow the reaction vessel to stand so that most of any insoluble residue settles out of suspension. Decant the relatively sediment-free supernatant carefully onto a filter paper (5.11), collecting the filtrate in a 100 ml volumetric flask. Allow all the initial filtrate to pass through the filter paper, then wash the insoluble residue onto the filter paper with a minimum of nitric acid (4.5). Collect this filtrate with the first.

NOTE 9 The amount of nitric acid (4.5) used depends on the quantity of residue, but more than 10 ml is rarely necessary.

The extract thus prepared is ready for the determination of trace elements, by an appropriate method, e.g. in accordance with [1] in annex A.

NOTE 10 The flask containing the extract could require the addition of releasing agents, depending on the element(s), of interest, and the spectroscopic method chosen. The flask should not, therefore, be filled to the mark at this stage, until the further steps in the analysis have been decided upon.

8 Test report

The test report can be prepared separately or in conjunction with the test report of the subsequent analytical method.

The test report shall include the following information:

a) a reference to this International Standard;

b) a complete identification of the sample;

c) a statement whether ground or unground soil has been used;

d) any details not specified in this International Standard, or which are optional, as well as any other factor which may have affected the results.

Annex A
(informative)

Bibliography

[1] ISO 11047:—[1], *Soil quality — Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc — Flame and electrothermal atomic absorption spectrometric methods.*

[2] HOUBA, J.V.G., CHARDON, W.J. and ROELSE, K. Influence of grinding of soil on apparent chemical composition (1993). *Commun. Soil Sci. Plant Anal.* **24** (13 and 14), pp.1591-1602.

[3] Soil Survey Staff (1975), Soil taxonomy: a basic system of soil classification for making and interpreting soil surveys. *Agriculture Handbook* No. **436,** Soil Conservation Service, United States Department of Agriculture, Washington DC, USA.

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[1] To be published.

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