
**Soil, treated biowaste, sludge and
waste — Digestion of aqua regia
soluble fractions of elements**

*Sols, biodéchets traités, boues et déchets — Digestion des éléments
solubles dans l'eau régale*



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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 444, *Environmental characterization of solid matrices*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Regarding the comparability of the procedure described in this document with those of the other standards mentioned above the next remarks can be made:

- This document describes the digestion of solid samples with aqua regia.
- Differences in the procedures of the different standards are small. An important difference between the reflux procedures as described in ISO 11466 and EN 13657 and EN 16174 concerns the waiting time after addition of the acid to the sample, before the digestion starts. ISO 11466 specifies a waiting time of 16 h, both European standards state that the digestion can start after the first strong reactions have ceased. In validation work it was proven that the difference between 2 h and 16 h of waiting was negligible, therefore this document follows the approach of EN 13657 and EN 16174.
- The heating block procedure was added to the reflux and microwave digestion procedures. The procedure was adopted from the Dutch standard NEN 6961, which specifies a boiling time of 2 h to 4 h. This document specifies a boiling time of 2 h.

The methods specified in this document are providing multi-element aqua regia digestion techniques for soil, treated biowaste, sludge and waste prior to analysis. It is known that the digestion of environmental samples with aqua regia will not necessarily lead to complete element recoveries, and that the extract from a test sample may not reflect the total concentrations of the target analytes. However, for most environmental applications the result obtained based upon digestion methods specified in this document are considered to be fit for the intended purpose.

This document is validated for several types of matrices as indicated in [Table 1](#).

Table 1 — Matrices for which this document is validated

Matrix	Materials used in the validation test
Sludge	Municipal sludge Industrial sludge Sludge from electronic industry Ink waste sludge Sewage sludge
Biowaste (Method A)	Compost Composted sludge
Soil	Agricultural soil Sludge amended soils
Waste	City waste incineration fly ash ("oxidised" matrix) City waste incineration bottom ash ("silicate" matrix) Ink waste sludge (organic matrix) Electronic industry sludge ("metallic" matrix) BCR 146R (sewage sludge) BCR 176 (city waste incineration ash)

WARNING — Persons using this document should be familiar with usual laboratory practice. Some of the reagents used in this document are highly corrosive and very toxic. Safety precautions are absolutely necessary, not only due to the strong corrosive reagents, but also to the high temperature and high pressure.

The use of laboratory-grade microwave equipment with isolated and corrosion resistant safety devices is required. Domestic (kitchen) type microwave ovens shall not be used, as corrosion by acid vapours may compromise the function of the safety devices and prevent the microwave

magnetron from shutting off when the door is open, which could result in operator exposure to hazardous levels of microwave energy.

All procedures should be performed in a fume hood or in closed force-ventilated equipment. By the use of strong oxidising reagents, the formation of explosive organic intermediates is possible, especially when dealing with samples with a high organic content. Do not open pressurized vessels before they have cooled down. Avoid contact with the chemicals and the gaseous reaction products.

IMPORTANT — It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

Soil, treated biowaste, sludge and waste — Digestion of aqua regia soluble fractions of elements

1 Scope

This document specifies two methods for digestion of soil, treated biowaste, sludge and waste by the use of an aqua regia digestion.

Digestion with aqua regia will not necessarily accomplish total decomposition of the sample. The extracted analyte concentrations may not necessarily reflect the total content in the sample but represent the aqua regia soluble metals under the condition of this test procedure. It is generally agreed that for environmental analysis purposes, the results are fit for the intended purpose to protect the environment.

This document is applicable for the following elements:

Aluminium (Al), antimony (Sb), arsenic (As), barium (Ba), beryllium (Be), boron (B), cadmium (Cd), calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), mercury (Hg), molybdenum (Mo), nickel (Ni), phosphorus (P), potassium (K), selenium (Se), silver (Ag), sodium (Na), strontium (Sr), sulfur (S), tellurium (Te), thallium (Tl), tin (Sn), titanium (Ti), vanadium (V), and zinc (Zn).

This document can also be applied for the digestion of other elements, provided the user has verified the applicability.

2 Normative references

There are no normative references in this document.

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 <http://www.electropedia.org/>

3.1

aqua regia

digestion (3.2) solution obtained by mixing 1 volume of nitric acid (mass fraction of 65 % to 70 %) and 3 volumes of hydrochloric acid (mass fraction of 35 % to 37 %)

Note 1 to entry: These mass percentages agree with the concentrations of 6.2 and 6.3.

3.2

digestion

mineralization of the organic matter of a sample and dissolution of its mineral part, more or less completely, when reacting with a reagent mixture

3.3

dry residue

dry matter expressed as a percentage by mass after drying at $105\text{ °C} \pm 5\text{ °C}$ to the constancy of weight