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Water quality — Digestion for the determination of selected elements in water —

Part 1:
Aqua regia digestion

Qualité de l'eau — Digestion pour la détermination de certains éléments dans l'eau —

Partie 1: Digestion à l'eau régale

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

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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 3.

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 part of ISO 15587 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15587-1 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical method*.

ISO 15587 consists of the following parts, under the general title *Water quality — Digestion for the determination of selected elements in water*:

- *Part 1:* Aqua regia *digestion*
- *Part 2: Nitric acid digestion*

Annexes A to E of this part of ISO 15587 are for information only.

Water quality — Digestion for the determination of selected elements in water —

Part 1:
Aqua regia digestion

WARNING — Persons using this part of ISO 15587 should be familiar with normal laboratory practice. This part of ISO 15587 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This part of ISO 15587 specifies a method for extracting trace elements from a water sample using *aqua regia* as a digestion agent. The method is applicable to all types of waters with a suspended solids mass concentration of less than 20 g/l and a mass concentration of total organic carbon (TOC) expressed as carbon of less than 5 g/l.

The *aqua regia* digestion method is empirical and it might not release elements completely. However, for most environmental applications, the result is fit for purpose.

Aqua regia digestion is suitable for the release of: Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, Ni, P, Pb, Sb, Se, Sn, Sr, Tl, V, Zn. It is not suitable for the digestion of refractory compounds such as SiO_2, TiO_2 and Al_2O_3. The presence of chloride in the digestion solution may limit the application of analytical techniques.

The method is generic and may be implemented using a wide variety of equipment provided

- the digestion composition is unchanged,
- the digestion temperature is known, and
- the digestion duration is in accordance with this temperature.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 15587. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 15587 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 5667-3:1994, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples*

3 Terms and definitions

For the purposes of this part of ISO 15587, the following terms and definitions apply.

3.1

aqua regia

solution obtained by mixing one volume of nitric acid with three volumes of hydrochloric acid

3.2

wet digestion

treatment with reactive liquid chemicals with the aim of bringing analytes into solution so as to be suitable for subsequent analysis

NOTE The degree to which analytes are brought into solution suitable for subsequent analysis may depend on the principle of the subsequent analysis. Some analytical methods require the element in solution to be independent of its chemical state, others require a specific valence or a specific ionic species, for instance hydride atomic spectrometry, photometry and electrochemical methods. For the latter, special attention should be paid to the presence of chemically stable organometals [5].

4 Principle

The test portion is digested with *aqua regia* at a defined temperature. The temperature is at least the boiling point (103 °C at 101,3 kPa) and at most 175 °C. At the boiling point (101,3 kPa), the minimum duration to release the digestible fraction is 120 min. The maximum duration is set at four times the minimum duration at that temperature. By convention, both the required minimum and maximum duration is assumed to halve with every 15 °C increase in temperature above the boiling point (see Figure 1).

After digestion the digest is clarified, if necessary.

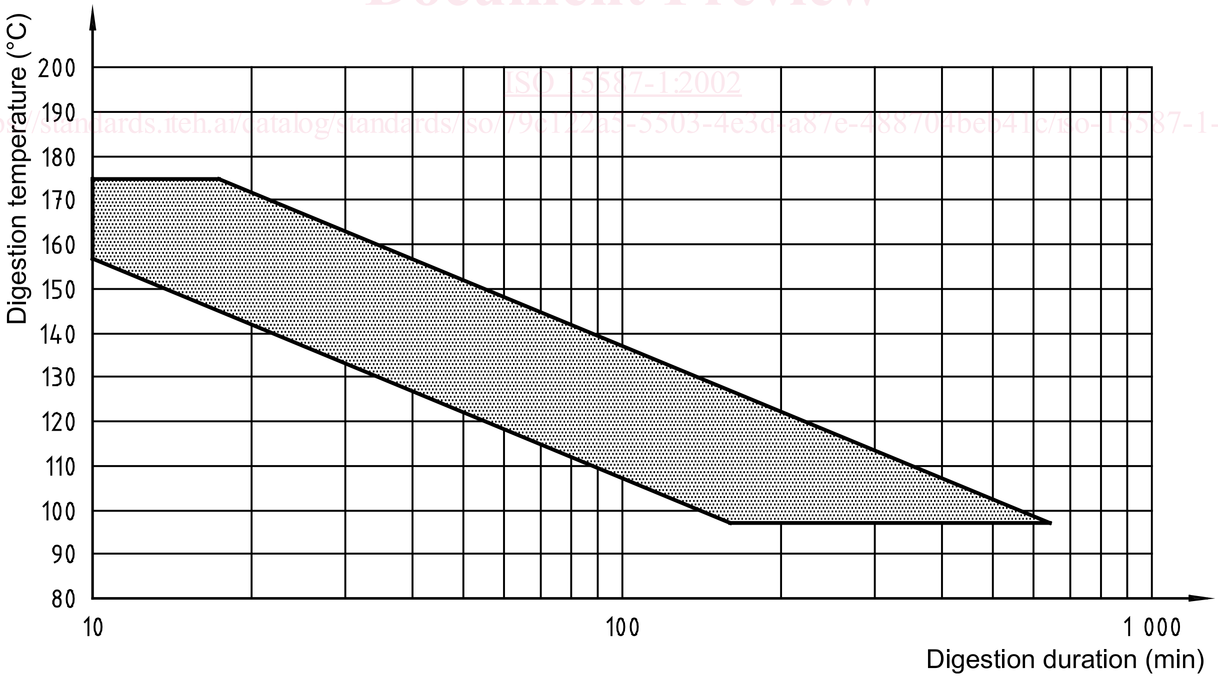

Figure 1 — Range of permitted digestion temperature and duration

5 Reagents

During analysis, use only reagents of recognized analytical grade that meet the purity requirements of the subsequent analysis. Verify their purity by performing a blank test.

5.1 Water, complying with grade 1 of ISO 3696. Grade 2 may be used if it meets the purity requirements for the subsequent analysis.

5.2 Hydrochloric acid, $c(\text{HCl}) = 12{,}0$ mol/l, $\rho = 1{,}2$ kg/l, approximately 37 % by mass.

5.3 Nitric acid, $c(\text{HNO}_3) = 15{,}8$ mol/l, $\rho = 1{,}4$ kg/l.

Nitric acid is available both as $\rho(\text{HNO}_3) = 1{,}40$ kg/l, approximately 65 % by mass and $\rho(\text{HNO}_3) = 1{,}42$ kg/l, approximately 69 % by mass. Both are suitable.

5.4 Antifoaming agent, for instance *n*-dodecane ($C_{12}H_{26}$).

6 Apparatus

6.1 Digestion vessel, temperature- and pressure-resistant and capable of containing the mixture of sample and digest solution.

If a closed vessel is applied, its inner wall shall be inert and shall not release substances to the digest in excess of the purity requirements of the subsequent analysis.

6.2 Vapour recovery system, capable of recovering the evaporated digest during open digestion (e.g. reflux condenser) and designed to minimize losses.

Materials in contact with the vapour shall be inert and shall meet the purity requirements of the subsequent analysis.

6.3 Volatile species trap, in an open digestion system capable of trapping one or more volatile measurement species which may pass through the vapour recovery system (6.2).

This trap may comprise condensation equipment or equipment to absorb these species in nitric acid or *aqua regia*. Materials in contact with the vapour shall be inert and shall meet the purity requirements of the subsequent analysis.

6.4 Heating device, capable of maintaining the target temperature for the required time period.

6.5 Filter paper, cellulose-based ashless type, with a median pore size adapted to the apparatus used for analysis.

It shall not be able to release substances to the digest in excess of the purity requirements of the subsequent analysis and shall not absorb substances from the digest beyond the level of negligible impact on trueness of the analytical result.

6.6 Anti-bumping granules or **roughened glass beads**, having a diameter of 2 mm to 3 mm and acid-washed [for instance with warm nitric acid (5.3) diluted to 10 % by volume with water (5.1)].

6.7 Volumetric flask, usually of nominal capacity of 50 ml or 100 ml.

6.8 Graduated pipettes or **dispensers**, of appropriate volume for the task.

7 Sampling

Handle and preserve the water sample in accordance with ISO 5667-3.

The digestion procedure (see clause 8) is based on a test portion of 25,0 ml ± 0,1 ml taken from a well-shaken homogeneous water sample. Any test portion larger than 20 ml is allowed, provided that the volume of *aqua regia* is modified proportionally.

If suspended solids make it impossible to deliver a representative test portion of a known volume, deliver the test portion otherwise, for instance by weighing. Calculate the volume from the determination of its mass, possibly corrected for the mass and density of solids. Report the uncertainty of the volume if it is higher than 0,1 ml for a volume of 25 ml.

To lower the limit of detection, the preserved sample may be concentrated by evaporation. Evaporation is carried out after addition of nitric acid (5.3) and before addition of hydrochloric acid (5.2). However, the concentrated water sample shall meet the requirements of clause 1. Evaporation is not allowed if volatile analyte species might be present.

8 Procedure

8.1 Preparation

Carefully acid-wash digestion equipment in contact with the digestion solution and volumetric ware [for instance with warm nitric acid (5.3) diluted to 10 % by volume with water (5.1)] and rinse with water (5.1).

Add a test portion of 25,0 ml ± 0,1 ml to the digestion vessel (6.1).

Add 6,0 ml ± 0,1 ml of hydrochloric acid (5.2) followed by 2,0 ml ± 0,1 ml of nitric acid (5.3). The boiling point of this mixture is equal to 103 °C.

The volume ratio sample to *aqua regia* is equal to approximately three. A volume ratio up to four is allowed.

If excessive foaming occurs, add one or more drops of antifoaming agent (5.4).

Swirl and allow the mixture to stand until any visible reaction has stopped.

If the digestion vessel is of the open type, proceed with 8.2. If not, proceed with 8.3.

8.2 Digestion in an open system

Connect the digestion vessel (6.1) to the vapour recovery system (6.2).

Connect the volatile species trap (6.3) so as to prevent volatile measurement species from passing through the vapour recovery system.

Place the digestion vessel in the heating device (6.4).

Increase the temperature to the boiling point.

Maintain boiling for at least 120 min. As the boiling point decreases with altitude, add 20 min to the minimum duration for every 1 000 m above sea level. The maximum allowed duration equals four times the minimum duration.

Once the digestion period is completed, allow the digestion vessel to cool.

If the volatile species trap (6.3) is used, add the contents to the digestion vessel.

Rinse the trap and/or the condenser with water (5.1) and decant the washings. Collect the water in the digestion vessel.

Dismount the digestion vessel from the digestion system.

Proceed with 8.4.

Examples of digestion in an open system are given in annexes A and B.

8.3 Digestion in a closed system

Cap the digestion vessel (6.1) and weigh it.

Connect the digestion vessel to the microwave equipment or place it onto the heating device (6.4).

Increase the temperature of the digestion mixture slowly to temperature T_d between the boiling point (103 °C at 101,3 kPa) and 175 °C.

Maintain digestion for a period Δt of at least 10 min and at most 480 min (101,3 kPa).

Given the digestion period Δt, expressed in minutes, the digestion temperature T_d, expressed in degrees Celsius, shall meet the following condition:

$$206{,}6 - 21{,}64 \times \ln(\Delta t) < T_\mathrm{d} < 236{,}6 - 21{,}64 \times \ln(\Delta t) \qquad (1)$$

NOTE 1 Boundary conditions in equation (1) reflect the assumption of clause 4 on release kinetics and duration time.

The permitted domain of digestion temperature and duration is shown in Figure 1.

Once the digestion period is completed, allow the digestion vessel to cool.

Weigh the digestion vessel and accept the digest if the mass loss is consistent with the mass loss of a well documented reference material known to lead to acceptable results.

Uncap and vent the digestion vessel in a fume hood.

Examples of digestion in a closed system are given in annexes C and D.

NOTE 2 Several microwave units control power rather than temperature. A procedure to establish the relationship between microwave power and temperature is given in C.6.

8.4 Sample transfer

Decant the sample into a suitable acid-cleaned volumetric flask (6.7). In order to transfer the analytes quantitatively, rinse the digestion vessel (6.1) with water (5.1) and decant the washings. Collect the water in the same volumetric flask.

If the transfer of particles that might interfere with the subsequent analysis cannot be avoided by simple decantation, decant the digest through a filter paper (6.5), collecting the filtrate in a suitable acid-cleaned volumetric flask (6.7). Wash the digestion vessel with water (5.1) and decant the washings through the filter paper (6.5). Collect the filtrate in the same volumetric flask.

Fill the volumetric flask to the mark after addition of reagents required for subsequent sample handling and analysis.

The extract is ready for determination.

Examples of performance checks are given in annex E.

9 Test report

The test report can be prepared separately or in conjunction with the test report of the subsequent analytical method. Either of these shall include the following information:

a) a reference to this part of ISO 15587, i.e. ISO 15587-1;

b) the identity of the water sample;

c) the volume of the test portion and its uncertainty if higher than 0,5 %;

d) the conditions of digestion;

e) any deviation from this method.