

Characterization of sludges — Determination of trace elements and phosphorus — Aqua regia extraction methods

The European Standard EN 13346:2000 has the status of a
British Standard

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National foreword

This British Standard is the official English language version of EN 13346:2000.

The UK participation in its preparation was entrusted to Technical Committee EH/5, Sludge characterization, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Further information

Users of this standard are informed that the UK submitted some substantial comments at the final voting stage of the draft EN for the following reasons.

1) The scope fails to address the question of whether this method is appropriate for all of the types of sludges in the scope of CEN/TC 308. For example, is it applicable to sludges containing large amounts of lime? Hence, modify the scope to answer the question of applicability to the scope of the TC.

2) The following note should be added to the scope:
“The validation for this standard has only been carried out using materials as described in annex A. For other sludges, the user should validate the method using recovery and reproducibility tests”.

3) The final phrase, “and sludge products,” in the first sentence of the scope should be removed as it is inappropriate.

4) This is not a single standard since there are 4 alternative methods of extraction and 3 alternative methods of pretreatment. The test report in clause 12 should therefore specify which of the 12 permutations has been used. Therefore, either rewrite c) as “describe which method of pretreatment and which method of digestion was used” or modify the document so that there is only one standard method against which others may be benchmarked.

5) In annex D, the data presented for Method D with reference materials appear to condemn this method as unfit since it is almost invariably outside the 95% confidence of the reference sample. Therefore, suggest deleting Method D.

6) It is recommended that an additional brief section on the use of adequate AQC as per CEN/TC 223 methods be added:
“The analysis described in this standard should be carried out within a quality control system utilizing analytical quality control measures. Where performance data are absent for the sample type, recovery and reproducibility data should be obtained as a minimum”.

This British Standard, having been prepared under the direction of the Health and Environment Sector Committee, was published under the authority of the Standards Committee and comes into effect on 15 October 2000

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Cross-references

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 29 and a back cover.

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English version

Characterization of sludges - Determination of trace elements and phosphorus - Aqua regia extraction methods

Caractérisation des boues - Détermination des éléments
traces et du phosphore - Méthodes d'extraction à l'eau
régale

Charakterisierung von Schlämmen - Bestimmung von
Spurenelementen und Phosphor - Extraktionsverfahren mit
Königswasser

This European Standard was approved by CEN on 5 August 2000.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.



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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 308, Characterization of sludges, the Secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2001, and conflicting national standards shall be withdrawn at the latest by February 2001.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

The annexes A to D are informative.

Introduction

Elements extractable in aqua regia cannot be described as "total"; conversely they cannot be regarded as the "bio-available" fraction, as the extraction procedure is too vigorous to represent all biological processes.

One extraction method is proposed, using aqua regia. Four heating processes are described, they use the same reagents therefore the same principle of chemical reaction.

Method A is a vigorous digestion procedure allowing larger test portions.

Method B is a fast procedure carried out in a single vessel, smaller test portions are used.

Methods C and D are microwave heating procedures using closed and open systems respectively.

1 Scope

This standard specifies methods for the extraction, with aqua regia, of trace elements and phosphorus from sludges and sludge products. The resulting solution is suitable for the determination of As, Cd, Cr, Cu, Hg, Ni, Pb, Se, Zn and P using spectrometric techniques.

NOTE 1 With high solute concentrations in extract solutions, spectral interferences and background enhancement should be expected.

NOTE 2 This method can be used for other elements except that portion of the elements bonded to silica.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 1233, *Water quality - Determination of chromium - Atomic absorption spectrometric methods*.

EN 1483, *Water quality - Determination of mercury*.

EN 12880, *Characterization of sludges - Determination of dry residue and water content*.

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods* (ISO 3696:1987).

EN ISO 5961, *Water quality - Determination of cadmium by atomic absorption spectrometry* (ISO 5961:1994).

EN ISO 11885, *Water quality - Determination of 33 elements by inductively coupled plasma atomic emission spectroscopy* (ISO 11885:1996).

EN ISO 11969, *Water quality - Determination of arsenic - Atomic absorption spectrometric method (hydride technique)* (ISO 11969:1996).

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*.

ISO 8288, *Water quality - Determination of cobalt, nickel, copper, zinc, cadmium and lead - Flame atomic absorption spectrometric methods*.

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1

aqua regia

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

3.2

dry mass (dry matter)

the mass of solids obtained after the specified drying process. It is expressed as grams or kilograms [EN 12880]

4 Principle

The laboratory sample is prepared in order to obtain a representative test portion which is extracted with aqua regia according to one of the following heating procedures:

- boiling for about 2 h with a reflux condenser, followed by filtration and making up to volume in a volumetric flask (method A);
- boiling for about 15 min in a calibrated glass tube, making up to volume in the same tube, allowing to settle and then decanting the supernatant solution for analysis (method B);
- microwave digestion for about 20 min in a closed vessel followed by filtration and making up to volume in a volumetric flask (method C);
- microwave digestion for about 30 min in an open vessel followed by filtration and making up to volume in a volumetric flask (method D).

NOTE Other heating procedures can be used providing they demonstrate equivalent performance.

5 Reagents

5.1 General

The reagents used shall be of sufficient purity so as not to introduce a significant error into the subsequent analysis. A reagent blank comprising the same quantity of acids shall be taken through the entire procedure with each batch of analysis.

5.2 Water

Deionized or distilled water used shall conform to purity grade 2 of EN ISO 3696.

5.3 Hydrochloric acid, solution $c(\text{HCl}) = 12,0 \text{ mol/l}$.

About 37 % (m/m), $\rho \approx 1,18 \text{ g/ml}$.

5.4 Nitric acid, solution, $c(\text{HNO}_3) = 15,8 \text{ mol/l}$.

About 65 % (m/m), $\rho \approx 1,42 \text{ g/ml}$.

5.5 Nitric acid solution, about 1 % (V/V) for cleaning.

Take 10 ml of nitric acid (5.4) and dilute to 1 l in a flask with water. This dilute nitric acid shall be used to clean the glassware.

5.6 n-Dodecane or other suitable antifoaming agents (for addition in method B).

6 Apparatus

6.1 General

All glassware and plasticsware shall be adequately cleaned and stored in order to avoid any contamination.

NOTE It can be necessary to periodically clean the reaction vessel with a suitable surfactant to remove stubborn deposits.

6.2 Reaction vessels

- a) glass flask, 250 ml for example (for method A);
- b) calibrated borosilicate glass tube 50 ml, for example, with a glass or polypropylene stopper (for method B);
- c) plastics vessel (at least 100 ml) made of perfluoroalkoxy polymer (PFA) or tetrafluoromethoxyl polymer (TFM) equipped with a system capable of controlling overpressure in order to avoid explosion of the vessel (for method C);
- d) digestion vessel made of borosilicate glass with a volume of at least 50 ml (for method D).

NOTE Quartz vessels can be used instead of glass vessels.

6.3 Reflux condenser

Adaptable to reaction vessels used in methods A or D.

6.4 Heating devices

- a) a heating mantle or aluminium block heater (for methods A and B respectively);
- b) a microwave oven with programmable power to control fast exothermic reaction and closed microwave vessels equipped with a safety system for method C. A domestic microwave oven is not allowed;
- c) a microwave oven with a programmer module (for method D).

NOTE For example an apparatus with microwave frequencies 2,45 GHz, electronic automatic power control which can be pilot-operated from 10 % to 99 % with 5 % increment for maximal power released of 200 W and a pump for reagent.

6.5 Filter papers

Filter paper cellulose based, hardened and resistant to aqua regia.

6.6 Volumetric flasks, capacity 25 ml, 50 ml or 100 ml.

6.7 Analytical balance, with an accuracy of 1 mg or better.

6.8 Boiling aids, anti bumping granules or glass beads, diameter 2 mm to 3 mm, acid washed (for methods A and B).

7 Preparation of the laboratory sample

Weigh a representative amount of the wet sample, to give a sufficient dry mass for the method used, in a porcelain dish and prepare it according to EN 12880. Take the dried sample and grind with contaminant free pestle and mortar or mill. Keep the sample under dry conditions.

NOTE Other methods of dried sample preparation are presented in annex A.