BRITISH STANDARD

BS EN
12880:2000

Characterization of sludges — Determination of dry residue and water content

The European Standard EN 12880:2000 has the status of a British Standard

ICS 13.030.20

National foreword

This British Standard is the official English language version of EN 12880:2000.

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) In annex A, it is essential that the description of the samples used in the inter-laboratory comparison is given. The date that the laboratory inter-comparison was carried out should also be given.

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 reproducibility tests".

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

4) In clause **5**, suggest adding the following phrase: "This is an empirical method where the result is defined by the methodology. The test is usually employed as a process control parameter with emphasis on the detection of significant changes".

5) In the section on Analytical Quality Control (AQC), it is recommended that an additional brief section be added on the use of adequate AQC as per CEN/TC223 methods:
"The analysis described in this standard should be carried out within a quality control system utilizing Analyical Quality Control measures. Where performance data are absent for the sample type, minimum reproducibility data should be obtained".

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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Amendments issued since publication

Amd. No.	Date	Comments

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

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Summary of pages

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 12880

August 2000

ICS 13.030.20

English version

Characterization of sludges - Determination of dry residue and water content

Caractérisation des boues - Détermination de la teneur en matière sèche et de la teneur en eau

Charakterisierung von Schlämmen - Bestimmung des Trockenrückstandes und des Wassergehalts

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

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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.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 12880:2000 E

Contents

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 annex A is informative.

1 Scope

This European Standard specifies a method for the determination of dry residue and water content of sludges and sludge products.

This method is applicable to the determination of dry residue and water content of sludges which include liquid, paste-like or solid matter.

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 12879, *Characterization of sludges – Determination of the loss on ignition of dry mass.*

3 Terms and definitions

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

3.1
dry residue
the dry mass portion of the sludge obtained after the specified drying process. It is expressed as percent or in grams per kilogram

3.2
water content
the mass portion of water present in the sludge. It is determined as a loss of mass under defined conditions after the specified drying process. It is expressed as percent or as grams per kilogram

3.3
dry mass (dry matter)
the mass of solids obtained after the specified drying process. It is expressed as grams or kilograms

3.4
constant mass
constant mass is reached when, during the drying process the difference between two successive weighings of the sample, first heated, then cooled to room temperature and with an interval of 1 h between them, does not exceed 0,5 % (m/m) of the last determined mass or 2 mg, whichever is the greater

4 Principle

Sludge samples are dried to constant mass in an oven at (105 ± 5) °C. The difference in mass before and after the drying process is used to calculate the dry residue and the water content.

5 Interferences

During storage, sludge samples can be subject to change (e.g. uptake or liberation of water, carbon dioxide and other substances) which is liable to falsify the analytical result. The samples can also change chemically during the drying process (e.g. by absorption of carbon dioxide in the case of basic samples or of oxygen caused by reducing substances).

NOTE 1 When determining the water content, volatile substances (such as organic solvents or substances deriving from the decomposition of organic or inorganic substances) are also included either completely or partially. In the case of sludges containing considerable amounts of water it is expedient to evaporate the greater part of the water carefully on a water bath in order to avoid loss of substance by splashing.

NOTE 2 In the case of sludges with a high solids content (e.g. dry residue $w_{dr} \geq 30$ %) there is the risk of water still remaining trapped in the sludge after drying.

6 Hazards

6.1 General

Samples are liable to ferment and usually contain harmful micro-organisms. It is essential to keep them away from any food or drink, and to protect any cuts. Bursting glass bottles containing sludge can produce micro-organism contaminated shrapnel. Plastic bottles can also burst and produce a hazardous spray and aerosol.

6.2 Storage

It is not advisable to store samples in the open laboratory. If samples are to be stored, store them between 0 °C and 4 °C.

6.3 Handling

Cleanliness when working is essential. When handling sludge samples, it is necessary to wear gloves, a face and eye protection, and sufficient body protection to guard against bottles bursting. The gas evolved is usually flammable, so all equipment used in the vicinity must be flame proof to avoid any source of ignition.

7 Apparatus

7.1 Drying oven, thermostatically controlled with forced air ventilation and capable of maintaining a temperature of (105 ± 5) °C.

7.2 Desiccator, with an active drying agent such as silica gel or diphosphorus pentoxide.

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

7.4 Evaporating dish or crucible. Temperature tolerant laboratory vessel withstanding 105 °C for dry residue analysis or 550 °C if further analysis of loss on ignition is required (see EN 12879). Suitable materials are porcelain, silica, platinum.

8 Procedure

Place an empty evaporating dish or crucible (7.4) in the drying oven (7.1) set at 105 °C for a minimum of 30 minutes. After cooling in the desiccator (7.2) to ambient temperature, weigh the basin to the nearest 1 mg, m_a.

If the same crucible is to be used for a subsequent loss on ignition measurement (see EN 12879), it shall be pre-ignited at 550 °C for a minimum of 30 min.

Depending on the expected water content, weigh into the evaporating dish or crucible a suitable amount of sludge, m_b, so that the dry matter obtained has a mass of at least 0,5 g.

Place the evaporating dish or crucible (7.4) containing the sample in the drying oven (7.1) set at 105 °C until the residue appears dry, typically overnight.

NOTE 1 There is a risk of formation of a cake surface. As a result of this cake formation, an even drying is hindered. To avoid this, a glass rod can be weighed along with the crucible. If cake formation occurs during drying, the glass rod is used to stir the sludge to break up the cake and put the liquid surface into contact with the hot air. This is repeated as necessary.

After cooling in the desiccator (7.2), weigh the evaporating dish or crucible and contents for the first time, m_c.

The dry residue ($m_c - m_a$) shall be regarded as constant, if the mass obtained after a further one hour of drying does not differ by more than 0,5 % of the previous value or 2 mg whichever is the greater (3.4).

Otherwise, repeat drying until constant mass has been reached.

NOTE 2 In cases where even after the third drying process it is not possible to obtain a constant value, record the value determined after at least 12 h and note this in the test report.

9 Expression of results

9.1 Calculation

Calculate the dry residue (w_{dr}) or the water content (w_w) expressed as percentage by mass or grams per kilogram using the following equations:

$$w_{dr} = \frac{(m_c - m_a)}{(m_b - m_a)} \times f \quad (1)$$

$$w_w = \frac{(m_b - m_c)}{(m_b - m_a)} \times f \quad (2)$$

where

w_{dr} is the dry residue of the sludge sample, in percent or grams per kilogram;

w_w is the water content of the sludge sample, in percent or grams per kilogram;

m_a is the mass of the empty dish or crucible in grams;

m_b is the mass of the dish or crucible containing the sludge sample in grams;

m_c is the mass of the dish or crucible containing the sludge dry matter in grams;

f is the conversion factor $f = 100$ for expression of results in percents and factor $f = 1\,000$ for expression of grams per kilogram.

Values shall be rounded to the nearest 0,1 % or alternatively to the nearest 1 g/kg.

9.2 Precision data

See annex A.

10 Test report

The test report shall contain the following information:

a) reference to this European Standard;

b) all information necessary for complete identification of the sludge sample;

c) details of sample pretreatment, if carried out;

d) results of the determination according to clause 9;

e) any detail not specified in this European Standard and any other factor which may have affected the results.

Annex A
(informative)

Performance data of the interlaboratory comparison

The data below were determined within the frame of a joint interlaboratory comparison for the parameters "Kjeldahl nitrogen" (EN 13342), "loss on ignition of dry mass" (EN 12879) and pH-value (EN 12176) and for this standard "dry residue and water content".

Table A.1 — Precision data for dry residue

Sample	l	n	NAP %	$\overline{X}$ %	σ_R %	VC_R %	σ_r %	VC_r %
Sludge 1	4	15	0	28,2	2,329	8,26	1,939	6,88
Sludge 2	4	15	0	27,9	0,63	2,26	0,423	1,52
Sludge 3	8	23	25	8,29	1,256	15,15	0,334	4,03
Sludge 4	8	23	25	9,32	0,916	9,83	0,308	3,30
Sludge 5	3	13	0	4,11	0,1	2,43	0,041	1,00
Sludge 6	3	13	0	2,88	0,167	5,80	0,065	2,26
Sludge 7	7	-	0	4,21	0,277	6,58	0,109	2,59
Sludge 8	7	-	0	3,49	0,606	17,36	0,151	4,33

l is the number of laboratories;

n is the number of values;

NAP is the percentage of outliers;

$\overline{X}$ is the overall mean;

σ_R is the standard deviation of reproducibility;

VC_R is the coefficient of variation of reproducibility;

σ_r is the standard deviation of repeatability;

VC_r is the coefficient of variation repeatability.

Bibliography

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

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