BRITISH STANDARD

BS EN
12879:2000

Characterization of sludges — Determination of the loss on ignition of dry mass

The European Standard EN 12879:2000 has the status of a British Standard

ICS 13.030.20

National foreword

This British Standard is the official English language version of EN 12879:2000.

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

ISBN 0 580 36377 5

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 12879

August 2000

ICS 13.030.20

English version

Characterization of sludges - Determination of the loss on ignition of dry mass

Caractérisation des boues - Détermination de la perte au feu de la matière sèche

Charakterisierung von Schlämmen - Bestimmung des Glühverlustes der Trockenmasse

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.

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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 12879:2000 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 the loss on ignition of dry mass of sludges and sludge products at 550 °C after the dry residues have been determined in accordance with the method of EN 12880.

NOTE It should be noted that inorganic substances or decomposition products (e.g. H_2O, CO_2, SO_2, O_2) are released or absorbed and some inorganic substances are volatile under the reaction conditions.

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

3 Terms and definitions

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

3.1
loss on ignition
the portion of mass escaping as gas as a result of the ignition of the dry mass of a sludge under specified conditions. The loss on ignition is related to the dry mass and expressed in percent

3.2
residue on ignition
the portion of mass remaining after the ignition of the dry mass of a sludge under specified conditions. The residue on ignition is related to the dry mass and expressed in percent

3.3
dry mass (dry matter)
the mass of solids obtained after the specified drying process. It is expressed as grams or kilograms [EN 12880]

3.4
constant mass
constant mass is reached when, during the ignition process, the difference between two successive weighings of the sample, first heated, then cooled to room temperature and with an interval of 1 hour between them, is within 0,5 % (*m/m*) of the last determined mass or 2 mg whichever is the greater

4 Principle

Samples of dried sludge are heated in a furnace at (550 ± 25) °C. The difference in mass before and after the ignition process is used to calculate the loss on ignition.

5 Interferences

There are normally no interferences with the loss on ignition determination. However, for many purposes this determination is used as an assessment of the organic part of the sludge dry mass. In this case the loss of volatile inorganic substances can occur giving high results.

NOTE 1 All sewage sludges contain iron in varying quantities and most of it will be present as ferrous iron. During ignition iron (II) will be oxidized to iron (III) with a possible increase in mass and therefore a reduction in the loss on ignition.

NOTE 2 If a sludge has been conditioned with lime, the calcium hydroxide or calcium oxide present will be available to combine with sulfur oxides liberated during ignition or with carbon dioxide formed during ignition decreasing the loss on ignition value.

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 advisable not 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, 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 **Crucible**, (50 to 70) mm in diameter, suitable for ignition at 550 °C, e.g. porcelain, silica or platinum.

7.2 **Muffle furnace** or equivalent equipment, capable of maintaining a temperature of (550 ± 25) °C.

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

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

8 Procedure

If the determination of loss on ignition is carried out in the same crucible as the determination of dry residue, refer to EN 12880 for the initial crucible weighing. If not, the sample is a representative portion of the dry mass obtained according to EN 12880. All the necessary precautions shall be taken to avoid absorption of atmospheric humidity by the sample until it is weighed.

Place a crucible (7.1) in the furnace (7.2) and heat at (550 ± 25) °C for at least 30 min. After cooling in the desiccator (7.3) to ambient temperature, weigh to the nearest 1 mg, (m_a).

Into the crucible weigh 0,5 g to 5 g of the dried sludge to the nearest 1 mg, (m_b), and heat in the furnace (7.2) at (550 ± 25) °C for at least 60 min.

NOTE 1 If the dry mass has a high organic content, losses can occur as a result of rapid ignition or deflagration of the sample. In this case heat the sample slowly until ignition.

Place the hot crucible containing the residue on ignition in the desiccator and leave to cool.

NOTE 2 If black particles are still present (some organic substances burn only slowly at 550 °C), wet the residue using ammonium nitrate solution prepared by dissolving 10 g of analytical grade ammonium nitrate ($NH_4 NO_3$) in 100 ml of distilled water. After repeated drying, slowly heat (in order to avoid losses by deflagration) the residue to ignition.

After cooling in the desiccator to ambient temperature, weigh the crucible containing the dry residue to the nearest 1 mg (m_c).

Weighing is carried out immediately after removal of the crucible from the desiccator and the weighing operation is completed as quickly as possible. The mass of the residue on ignition and therefore the loss on ignition shall be regarded as constant if the mass obtained after a further half-hour period of ignition at 550 °C in the preheated furnace, ($m_c - m_a$), does not differ by more than 0,5 % of the previous value or 2 mg whichever is the greater (3.4). Otherwise repeat ignition until constant mass has been reached.

NOTE 3 In cases when even after the third heating to 550 °C it is not possible to obtain constant mass, record the value determined as the mean of at least three replicates measured after 1 hour stored in a desiccator. This should be reported together with the result.

9 Expressions of results

9.1 Calculation

The loss on ignition of the dry mass of a sludge expressed in percent shall be calculated from equation (1):

$$w_V = \frac{(m_b - m_c)}{(m_b - m_a)} \times 100 \quad (1)$$

The residue on ignition of the dry mass of a sludge expressed in percent shall be calculated from equation (2):

$$w_R = 100 - w_V \quad (2)$$

where

w_V is the loss on ignition of the dry mass of a sludge or sediment, in percent;

w_R is the residue on ignition of the dry mass of a sludge or sediment, in percent;

m_a is the mass of the empty crucible, in grams;

m_b is the mass of the crucible containing the dry mass, in grams;

m_c is the mass of the crucible containing the ignited dry mass, in grams.

The results shall be rounded to the nearest 0,1 %.

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) results of the determination according to clause 9;

d) 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 below data were determined within the frame of a joint interlaboratory comparison for the parameters "Kjeldahl nitrogen" (EN 13342), "dry residue and water content" (EN 12880) and pH-value (EN 12176) and for this standard "loss on ignition of dry mass".

Table A.1 — Precision data for loss on ignition of dry mass

Sample	l	n	NAP	$\overline{X}$	σ_R	VC_R	σ_r	VC_r
			%	%	%	%	%	%
1	4	15	0	63,08	0,642	1,02	0,26	0,41
2	4	15	0	90,1	0,105	0,12	0,105	0,12
3	8	23	25	68,1	1,287	1,89	0,305	0,45
4	8	23	25	79,8	0,707	0,89	0,33	0,41
5	3	13	0	73,9	1,707	2,31	0,613	0,83
6	3	13	0	61,5	1,99	3,24	0,341	0,55
7	7	-	0	59,02	1,346	2,28	0,506	0,86
8	7	-	0	46,65	2,541	5,45	1,625	3,48

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.

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