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Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content

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National foreword

This British Standard is the UK implementation of EN 15934:2012.

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

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Sludge, treated biowaste, soil and waste - Calculation of dry matter fraction after determination of dry residue or water content

Boues, bio-déchets traités, sols et déchets - Calcul de la teneur en matière sèche par détermination du résidu sec ou de la teneur en eau

Schlamm, behandelter Bioabfall, Boden und Abfall - Berechnung des Trockenmassenanteils nach Bestimmung des Trockenrückstands oder des Wassergehalts

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Foreword

This document (EN 15934:2012) has been prepared by Technical Committee CEN/TC 400 “Project Committee - Horizontal standards in the fields of sludge, biowaste and soil”, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

In case of analysis of solids and sludge water is usually not considered as part of the sample and results are generally related to dry matter, which can be calculated by determination of the dry residue (dry matter fraction). For this purpose, and for the determination of the water content, two methods are described in this European Standard. The choice of the method depends on the type of sample and its content of volatile substances excluding water.

As a result of the validation study, the determination of water content by azeotropic distillation has been replaced by Karl-Fischer-titration. Nevertheless, the distillation may be useful in certain cases. This method is described in Annex B (informative).

This European Standard is applicable and validated for several types of matrices as indicated in Table 1 (see also Annex A for the results of the validation).

Table 1 — Matrices for which this European Standard is applicable and validated

Matrix		Materials used for validation
Sludge	(only method A)	Municipal sludge
Biowaste	(only method A)	Fresh compost
Soil	(only method A)	Sludge amended soil
Waste	(method A and B)	Contaminated soil, Dredged sludge, Nickel sludge, Filter cake, Distillation residue, Drilling emulsion

WARNING — Persons using this European Standard should be familiar with usual laboratory practice. This European Standard 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.

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

1 Scope

This European Standard specifies methods for the calculation of the dry matter fraction of sludge, treated biowaste, soil and waste for which the results of performed analysis are to be calculated to the dry matter basis. Depending on the nature and origin of the sample, the calculation is based on a determination of the dry residue (Method A) or a determination of the water content (Method B). It applies to samples containing more than 1 % (mass fraction) of dry residue or more than 1 % (mass fraction) of water.

Method A applies to sludge, treated biowaste, soil and solid waste. Method B applies to liquid waste and to samples which are suspected or known to contain volatiles except for water.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15002, *Characterization of waste — Preparation of test portions from the laboratory sample*

EN 16179, *Sludge, treated biowaste and soil — Guidance for sample pretreatment*

3 Terms and definitions

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

3.1
dry residue
remaining mass fraction of a sample after a drying process at 105 °C under specified conditions

3.2
water content
mass fraction of water in a sample determined by the method after drying at 105 °C or by Karl-Fischer-titration under specified conditions

3.3
dry matter fraction
mass fraction of a sample excluding water expressed as mass fraction calculated by determination of dry residue or water content

4 Principle

4.1 General

Depending on the origin (sludge, biowaste, soil, waste) and the nature of the sample (liquid, solid or multiphase) either the water content or the dry residue is to be determined. The results from the determination of water content or dry residue are used to calculate the dry matter fraction. In case of multiphase (waste) samples these samples shall be homogenized. If homogenization is not possible, a phase separation according to EN 15002 shall be applicable and the phases are analysed separately.

4.2 Principle of Method A — Drying at 105 °C (dry residue)

The sample is dried to a constant mass at 105 °C. This method applies to solid samples and samples which become solid during the drying process. The method generally applies to soil, sludge and treated biowaste. Solid samples containing amounts of volatiles which are expected to affect the results shall be analysed by Method B.

4.3 Principle of Method B — Direct Karl-Fischer-titration (water content)

The method usually applies to liquid samples and homogenised multiphase samples. It can also be used for solid samples in case the “water content” is of interest and in case volatiles are expected to interfere with the determination according to Method A. The water content of a sample is determined by direct Karl-Fischer-titration and either volumetric or coulometric detection. Liquid samples are directly added to the cell, while solid samples are extracted by use of methanol.

5 Sample preparation

Pretreat the samples according to EN 16179 or EN 15002, if not otherwise specified.

6 Method A — Drying at 105 °C

6.1 General

When performing any kind of analysis on samples, the dry matter fraction shall be determined simultaneously on identical test portions, in order to base all other test results on the determined dry matter content of each individual sample.

6.2 Interferences

The samples can change during the drying process, e.g. by absorption of carbon dioxide in the case of alkaline samples, or of oxygen by reducing substances. Volatile compounds evaporating at 105 °C are measured as water using this procedure.

NOTE For alkaline samples or samples susceptible to oxidation the drying can be carried out in a nitrogen atmosphere.

6.3 Hazards

Flammable or explosive gases may be released in the drying process.

6.4 Apparatus

6.4.1 Drying system thermostatically controlled and capable of maintaining a temperature of (105 ± 5) °C; e.g. drying oven, infrared system, halogen lamp system.

6.4.2 Desiccator with an active drying agent such as silica gel.

6.4.3 Precision balance with an accuracy of 1 mg or better.

6.4.4 Evaporating dish or **crucible**. Temperature tolerant laboratory vessel withstanding 105 °C. Suitable materials are metallic, ceramic, borosilicate glass or quartz.

NOTE 1 If the sample is to be ashed following this procedure, it should be ensured that the material will withstand the elevated temperatures.

NOTE 2 The material of the evaporating dish or crucible should be compatible with the nature of the sample to avoid interferences upon weighing due to possible oxidations.

6.5 Procedure

Place an evaporating dish or crucible (6.4.4) in the drying system (6.4.1) set at (105 ± 5) °C for a minimum of 30 min. After cooling in the desiccator (6.4.2) to ambient temperature, weigh the basin to the nearest 1 mg.

Depending on the expected dry residue, weigh into the evaporating dish or crucible (6.4.4) a suitable amount of material so that the residue obtained has a weight of at least 0,5 g. Weigh the loaded basin to the nearest 1 mg.

Place the evaporating dish or crucible (6.4.4) containing the sample in the drying system (6.4.1) set at (105 ± 5) °C until the residue appears dry, typically overnight.

NOTE For some samples e.g. sludge, there is a risk of formation of a cake surface, which hinders an even drying. To avoid this, a glass rod can be weighed along with the dish. If cake formation occurs during drying, the glass rod is used to stir the sample, to break up the cake. This should be repeated as necessary. The rod should be reweighed afterwards.

After cooling in the desiccator (6.4.2) weigh the evaporating dish or crucible and contents for the first time.

The dry residue shall be regarded as constant if the mass obtained after further 1 h of drying does not differ by more than 0,5 % of the previous value or 2 mg whichever is the greater. Otherwise repeat the drying process.

In case of weight inconstancy after three cycles the drying process may be stopped (after at least 16 h). The result of the last weighing shall be recorded in the test report. A drying period of 16 h is sufficient.

In case of infrared or halogen lamp drying systems the corresponding manufacturer’s instructions apply. The technique of choice shall be recorded in the test report.

Other techniques than oven drying, infrared or halogen lamp drying are allowed, provided they are proven to give comparable results.

6.6 Calculation of results

The dry residue is calculated according to Formula (1):

$$w_{\mathrm{dr}} = \frac{m_{\mathrm{c}} - m_{\mathrm{a}}}{m_{\mathrm{b}} - m_{\mathrm{a}}} \cdot f \qquad (1)$$

where

w_{dr} is the dry residue of the sample, expressed as mass fraction in percent (%) or in grams per kilogram (g/kg);

m_{a} is the mass of the empty dish or crucible, expressed in grams (g);

m_{b} is the mass of the dish or crucible containing the sample, expressed in grams (g);

m_{c} is the mass of the dish or crucible containing the dried sample, expressed in grams (g);

f is the conversion factor f = 100 for expression of results as mass fraction in percent (%) and f = 1 000 for expression of results in grams per kilogram (g/kg).

7 Method B — Direct Karl-Fischer-titration (volumetric/coulometric detection)

7.1 Interferences

Substances like peroxides, oxides, hydroxides, ammonia, carbonates, hydrogen carbonates, disulfites, nitrite, sulfite, thiosulfate, hydrazine and derivatives, iron (II), copper (I), tin (II), silanoles, arsenite, arsenate, selenite, tellurite, phenols, boric compounds, aldehydes, acetone and other reactive ketones may cause interferences. Especially all reductive or oxidative reagents are potentially interfering. In case of concern about interferences results can be verified by azeotropic distillation (Annex B).

Most of the interferences are reduced in case of extraction with methanol before analysis.

7.2 Reagents

All reagents used shall be at least of analytical grade and suitable for their specific purposes. Hygroscopic substances shall be stored in a desiccator.

7.2.1 **Karl-Fischer reagent** as recommended by provider of KF-system.

7.2.2 **Methanol.** The methanol used shall not contain more than a mass fraction of 0,005 % water.

7.2.3 **Toluene** if necessary to solve a sample.

7.3 Apparatus

Usual laboratory glassware and the following equipment shall be used.

7.3.1 **Karl-Fischer-titrator.**

7.3.2 **Shaking device.**

7.3.3 **Weighing pipette.**

7.3.4 **Conical flasks,** 250 ml with ground glass neck and stopper.

7.4 Procedure

7.4.1 Determination of the equivalence factor

Fill the titration vessel (7.3.1) with methanol (7.2.2). Stir without splattering. Titrate with Karl-Fischer reagent (7.2.1) to the end point. Using a weighing pipette (7.3.3) add approximately 80 mg of water (W) with an accuracy of 0,1 %. Titrate to the end point and record the consumption (V_1).

The equivalence factor F is calculated according to Formula (2):

$$F = \frac{W}{V_1} \tag{2}$$

where

F is the equivalence factor, expressed in milligrams of water per millilitre (mg/ml);

W is the quantity of water, expressed in milligrams (mg);

V_1 is the volume of the titration fluid, expressed in millilitres (ml).

The equivalence factor shall be determined weekly.

Instead of water other suitable standards with defined water content are applicable. In case of coulometric detection no titer adjustment is required.

7.4.2 Analysis of liquid samples

Fill the titration vessel (7.3.1) with methanol (7.2.2). Stir without splattering. Titrate with Karl-Fischer reagent (7.2.1) to the end point. Add an appropriate amount of the liquid sample M with an accuracy of 0,1 % (in case of water contents less than 10 % about 1 g of sample is appropriate).

Record the added volume of the titration liquid (V) for the sample.

The water content of the liquid sample is calculated according to Formula (3):

$$w_{\mathrm{W}} = \frac{V \times F}{M \times 1000} \times 100 \tag{3}$$

where

w_{W} is the water content, expressed as mass fraction in percent (%);

V is the added volume of the titration liquid, expressed in millilitres (ml);

F is the equivalence factor (according to Formula (2)), expressed in milligrams water per millilitre (mg/ml);

M is the mass of an aliquot of the sample, expressed in grams (g).

The dry matter fraction is calculated by Formula (6).

7.4.3 Analysis of solid samples

Weigh approximately 10 g solid sample M to 0,1 % accurately into a conical flask (7.3.4). Pipette 50,0 ml methanol (7.2.2). Shake for 10 min in a shaking machine (7.3.2). Fill the titration vessel with methanol (7.2.2). Stir without splattering. Titrate with Karl-Fischer reagent (7.2.1) to the end point. Pipette an appropriate volume (V_{E}) of the extract into the sample vessel (in case of water contents less than 5 % about 10 ml of extract is appropriate).

Note the added volume of titration liquid (V) for the extract. Also perform a blank run. Note the added volume of the titration liquid V_0 for the blank.

The water content of the solid sample, expressed as mass fraction in percent, is calculated according to Formula (4):

$$w_{\mathrm{W}} = \frac{(V - V_0) \times F \times 50}{M \times 1000 \times V_{\mathrm{E}}} \times 100 \tag{4}$$

where

w_{W} is the water content of phase i, expressed as mass fraction in percent (%);

V is the added volume of the titration liquid, expressed in millilitres (ml);

V_0 is the added volume of the titration liquid in blank run, expressed in millilitres (ml);

V_E is the added volume of the extract, expressed in millilitres (ml);

F is the equivalence factor (according to Formula (2)), expressed in milligrams water per millilitre (mg/ml);

M is the mass of an aliquot of the sample, expressed in grams (g).

Calculate the dry matter fraction according to Formula (6).

In case of samples with interfering compounds the manufacturer should be contacted.

8 Calculation of dry matter fraction

The dry matter is calculated according to Formulae (5) or (6):

$$w_{dm} = w_{dr} \tag{5}$$

where

w_{dm} is the dry matter fraction, expressed as mass fraction in percent (%);

w_{dr} is the dry residue, expressed as mass fraction in percent (%)

or

$$w_{dm} = 100 - w_w \tag{6}$$

where

w_{dm} is the dry matter fraction, expressed as mass fraction in percent (%);

w_w is the water content, expressed as mass fraction in percent (%).

9 Precision

The performance characteristics of the methods have been evaluated (see Annex A).

10 Test report

The test report shall contain at least the following information:

a) a reference to this European Standard (EN 15934);

b) complete identification of the sample;

c) the applied method (Method A or Method B)

d) expression of results, according to Clause 8;

e) any details not specified in this European Standard or which are optional, as well as any factor which may have affected the results.

Annex A
(informative)

Repeatability and reproducibility

A.1 Performance characteristics

The data of Method A for sludge, biowaste and soil given in Table A.1 have been established in an European Intercomparison study carried out in 2008. The performance data of Methods A and B for waste given in Table A.1 and Table A.2 have been established in a European interlaboratory comparison study carried out in 2005.

Table A.1 — Performance characteristics Method A – drying at 105 °C – dry residue

Matrix	l	n	n_o	$\bar{\bar{x}}$ %	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
Sludge 1	25	105	3	96,57	0,61	0,63	0,33	0,34
Sludge 2	29	130	2	95,47	0,49	0,51	0,23	0,24
Compost 1	28	125	4	93,72	0,47	0,51	0,14	0,15
Compost 2	29	123	5	91,73	0,54	0,59	0,38	0,42
Soil 4	28	112	4	95,28	0,39	0,41	0,13	0,14
Soil 5	28	106	6	98,39	0,13	0,14	0,08	0,08
Contaminated soil	33	97	12	93,26	0,45	0,48	0,10	0,10
Dredged sludge	32	94	0	66,43	0,66	0,98	0,26	0,39
Nickel sludge	33	98	6	52,95	1,08	2,04	0,34	0,60

Explanation of symbols

l	number of participating laboratories
n	number of analytical results after outlier rejection
n_o	number of outliers
$\bar{\bar{x}}$	total mean of results (without outliers) (mass fraction)
s_R	reproducibility standard deviation
$C_{V,R}$	coefficient of variation of reproducibility
s_r	repeatability standard deviation
$C_{V,r}$	coefficient of variation of repeatability

Table A.2 — Performance characteristics of Method B – Karl-Fischer-titration – water content

Matrix	l	n	o	$\bar{\bar{x}}$	s_R	$C_{V,R}$	s_r	$C_{V,r}$
			%	%		%		%
Contaminated soil	18	54	11	6,44	1,07	16,63	0,23	3,54
Distillation residue	22	66	0	9,04	0,84	9,34	0,20	2,17
Dredged sludge	17	51	18	30,69	3,48	11,32	0,78	2,55
Drilling solution	22	66	0	97,44	1,88	1,93	1,03	1,05
Nickel sludge	18	54	12	38,38	4,95	12,58	0,76	1,92
Waste oil	21	63	14	69,19	1,96	2,83	0,83	1,20

Explanation of symbols

l	number of participating laboratories
n	number of analytical results after outlier rejection
o	outliers in per cent (%)
$\bar{\bar{x}}$	total mean of results (without outliers) (mass fraction)
s_R	reproducibility standard deviation
$C_{V,R}$	coefficient of variation of reproducibility
s_r	repeatability standard deviation
$C_{V,r}$	coefficient of variation of repeatability

A.2 Types of samples and sample preparation

NOTE Aside from the immediate validation data, a validation study contains additional data which may be relevant to the user of this European Standard.

A.2.1 **Sludge 1** and **Sludge 2.** Mixtures of municipal sludge (particle size < 0,5 mm) from waste water treatment plants in North Rhine-Westphalia, Germany.

A.2.2 **Compost 1.** Fresh fine-grained compost (< 2 mm) from Vienna, Austria.

A.2.3 **Compost 2.** Fine-grained compost (< 2 mm) from Germany.

A.2.4 **Soil 4.** Sludge amended, fine-grained soil (< 2 mm) from Hohenheim, Germany.

A.2.5 **Soil 5.** Agricultural, fine-grained soil (< 2 mm) from Reading, United Kingdom.

A.2.6 **Contaminated soil.** The sample was taken from an old industrial site. It was sieved (2 mm) and spiked with activated charcoal (4 %, mass fraction) to ensure practical data for loss on ignition. Afterwards, it was homogenized by shaking in a plastic drum.

A.2.7 **Dredged sludge.** The sample was taken from a stagnant riparian water zone at the river Rhine. In order to obtain a sample ready for homogenisation it was pre-dried at room temperature for about 30 h, sieved (2 mm) and homogenized in a plastic drum.

A.2.8 Nickel sludge. This mixture of inorganic waste material consisted mainly of carbonates resulting from a metallurgical process. The material was passed through a 3 mm sieve. In order to obtain a sample ready for homogenisation it was pre-dried at room temperature overnight and homogenized in a plastic drum.

A.2.9 Drilling emulsion. This sample was an oil-water emulsion that had been used for drilling in metal processing. Laboratory samples were prepared while the sample (about 7 l) was homogenized in a 10 l glass container by vigorous stirring.

A.2.10 Waste oil. This sample was a mixture of waste engine oil and hydraulic fluid, obtained from an oil regeneration plant and homogenized like a drilling solution (A.2.9).

A.2.11 Distillation residue. A mixture of organic compounds was from a pharmaceutical production process. It consisted of low boiling alcohols and ketones, e.g. methanol and acetone.

A.3 Homogeneity and stability

For investigation of homogeneity repeated analyses from eight different sample vessels were performed. Data from homogeneity testing are given in Table A.3.

Stability and in-bottle homogeneity of samples were tested by 3 to 4 repeated analyses from one sample container. Analyses were performed during the experimental phase of the study. Vials a, b, c and d were analyzed at different dates during the validation study. Results showed that all samples were stable.

Table A.3 — Results from homogeneity testing

Sample		Dry residue (105 °C) %	Water content (Karl-Fischer titration) %
Contaminated soil	mean	92,94	—
	standard deviation	0,05	—
	relative standard deviation	0,06	—
Dredged sludge	mean	66,85	—
	standard deviation	0,18	—
	relative standard deviation	0,27	—
Nickel sludge	mean	53,39	—
	standard deviation	0,57	—
	relative standard deviation	1,08	—
Drilling emulsion	mean	—	98,03
	standard deviation	—	0,71
	relative standard deviation	—	0,72
Waste oil	mean	—	69,83
	standard deviation	—	0,22
	relative standard deviation	—	0,31
Distillation residue	mean	—	9,16
	standard deviation	—	0,07
	relative standard deviation	—	0,81

A.4 Dry matter fraction results by different methods

In Table A.4, the calculated dry matter data from the mean value of the different materials are listed. This allows comparing results from different methods. Except for the nickel sludge the data are comparable.

Table A.4 — Calculated dry matter fraction from all different methods

Material	Dry matter fraction (% mass fraction) from		
	Drying at 105 °C	Karl-Fischer-titration	Azeotropic distillation
Contaminated soil	93,3	93,6	93,2
Dredged sludge	66,4	69,3	67,6
Nickel sludge	53,0	61,2	56,8
Drilling emulsion	—	2,5	2,6
Waste oil	—	30,8	30,9
Distillation residue	—	91,0	— [a]

[a] Most laboratories were unable to measure the water content of the distillation residue by azeotropic distillation.

Annex B
(informative)

Azeotropic distillation with toluene

B.1 General

As a result of the validation study the determination of water content by azeotropic distillation has been replaced by Karl-Fischer-titration. Nevertheless, the distillation may be useful in certain cases. See Annex A for data comparison.

B.2 Interferences

Volatile, water-soluble compounds are determined as water. If it is believed that these compounds are present, the distillate may be analysed for water by an appropriate Karl-Fischer method.

B.3 Hazards

Toluene and toluene vapour are toxic and flammable. Avoid inhalation and contact with eye and skin.

B.4 Reagents

B.4.1 Toluene C_7H_8; at least analytical grade.

Other solvents are applicable if they lead to comparable results (e.g. petroleum ether, boiling range: 100 °C to 140 °C).

B.5 Apparatus

Usual laboratory glassware and the following equipment shall be used.

B.5.1 Apparatus for azeotropic distillation

The apparatus consists of a glass distillation vessel 500 ml to 2 000 ml, a heater, a reflux cooler of at least 400 mm length and a scaled distillate vessel. See Figure B.1.

B.5.2 Anti-bumping-agents (e.g. glass pearls, Raschig rings).

B.5.3 Glass rods.

B.6 Procedure

Accurately weigh an aliquot of sample of about (100 ± 1) g into the distillation vessel, and then add 100 ml of toluene (B.4.1). Add glass pearls, Raschig rings etc. if necessary to prevent “bumping”.

Start the distillation and distil until no water is visible in parts of the apparatus except the collection tube and while the water volume in the collection tube changes within 5 min.

Allow the collection tube to cool down to ambient temperature. Push possible water drops down by use of a suitable instrument (e.g. glass rod).

Determine the volume of water by reading the scale.

B.7 Calculation of results

The water content of the sample is calculated according to Formula (B.1):

$$w_{\mathrm{W}} = \frac{V \cdot \delta \cdot f}{m_i} \tag{B.1}$$

where

w_{W} is the water content of the sample, expressed as mass fraction in percent (%) or in grams per kilogram (g/kg);

V is the volume of water in the collection tube, expressed in millilitres (ml);

δ is the density of water, expressed in grams per millilitre (g/ml);

m_i is the mass of sample aliquot i, expressed in grams (g);

f is the conversion factor f = 100 for expression of results as mass fraction in percent (%) and f = 1 000 for expression of results in grams per kilogram (g/kg).

NOTE For the purpose of this European Standard the density of water can be approximated to 1,0 g/ml.

B.8 System performance check

A system check is performed by use of water as a sample or as reference material with known water content. The results have to meet the requirements in Table B.1. Deviations from these requirements may be due to vapour losses, rapid distillation and ambient moisture.

Table B.1 — System performance criteria

Volume of collection tube ml	Volume of water added at 20 °C ml	Recovery at 20 °C ml
5	1	1 ± 0,1
10	1	1 ± 0,1
10	5	5 ± 0,2
25	12	12 ± 0,2

B.9 Performance data

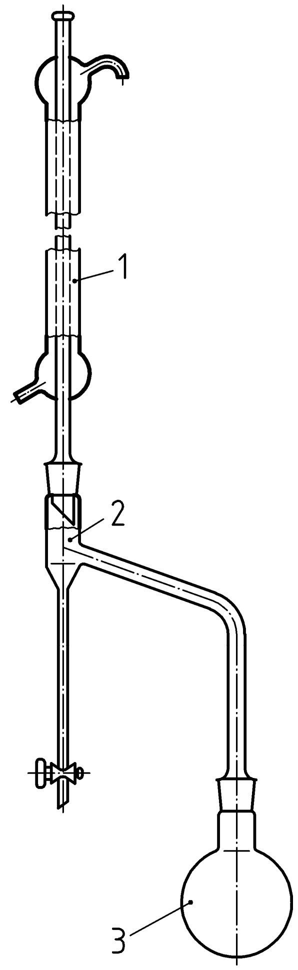
Due to a lack of participants the distillation method did not pass the validation criteria. The available performance data are given in Table B.2 for information.

Table B.2 — Performance data of azeotropic distillation method

Matrix	l	n	o %	$\bar{\bar{x}}$ %	s_R	$C_{V,R}$ %	s_r	$C_{V,r}$ %
Contaminated soil	8	24	0	6,77	1,12	16,52	0,44	6,51
Dredged sludge	8	24	0	32,44	2,29	7,07	1,83	5,65
Drilling emulsion	7	21	14	97,37	2,12	2,18	1,80	1,85
Nickel sludge	7	21	0	43,21	2,73	6,32	1,36	3,14
Waste oil	7	21	14	69,30	2,39	3,44	2,04	2,94

Explanation of symbols

l	number of participating laboratories
n	number of analytical results after outlier rejection
o	outliers in percent (%)
$\bar{\bar{x}}$	total mean of results (without outliers) (mass fraction)
s_R	reproducibility standard deviation
$C_{V,R}$	coefficient of variation of reproducibility
s_r	repeatability standard deviation
$C_{V,r}$	coefficient of variation of repeatability



Key

1 cooler
2 receiver
3 distillation vessel

Figure B.1 — Apparatus for azeotropic distillation

Annex C
(informative)

Summary of general requirements and recommendations

Purpose of this summary is to support the organization of sampling and sample pretreatment processes. The information given should be helpful to prepare a sampling plan.

Table C.1 — General requirements and recommendations

Matrix restrictions	No restrictions.
Typical working range	> 1 % (mass fraction) of dry matter fraction or water content.
Sampling instruments	No restrictions.
Pre-treatment of sample container	Clean and dry; free of solvents.
Sample container	Wide neck amber glass with grounded stopper.
Transport conditions	Dark and cool.
Preservation	Cooling at about 4 °C.
Storage conditions	At 4 °C in the dark not longer than 1 week; longer storage at about –18 °C.
Required amount	About 200 g original sample.
Test portion	About 20 g of the homogenized sample.

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