BRITISH STANDARD

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Characterization of waste — Calculation of dry matter by determination of dry residue or water content

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ICS 13.030.01

British Standards

National foreword

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Characterization of waste - Calculation of dry matter by determination of dry residue or water content

Caractérisation des déchets - Calcul de la teneur en matière sèche par détermination du résidu sec ou de la teneur en eau

Charakterisierung von Abfällen - Berechnung der Trockenmasse durch Bestimmung des Trockenrückstandes oder des Wassergehaltes

This European Standard was approved by CEN on 25 November 2006.

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Foreword

This document (EN 14346:2006) has been prepared by Technical Committee CEN/TC 292 “Characterization of waste”, the secretariat of which is held by NEN.

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of an identical text or by endorsement, at the latest by June 2007, and conflicting national standards shall be withdrawn at the latest by June 2007.

Anyone dealing with waste and sludge analysis should be aware of the typical risks of that kind of material irrespective of the parameter to be determined. Waste and sludge samples may contain hazardous (e.g. toxic, reactive, flammable, infectious) substances, which can be liable to biological and/or chemical reaction. Consequently these samples should be handled with special care. Gases which may be produced by microbiological or chemical activity are potentially flammable and will pressurise sealed bottles. Bursting bottles are likely to result in hazardous shrapnel, dust and/or aerosol. National regulations should be followed with respect to all hazards associated with this method.

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

Introduction

In case of waste analysis water is usually not considered as part of the sample and results are generally related to dry matter, which can be calculated from the water content or the dry residue. For this purpose 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).

1 Scope

This European Standard specifies methods for the calculation of the dry matter of samples for which the results of performed analysis are to be calculated to the dry matter basis. Depending on the nature 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 % (*m/m*) of dry residue or more than 1 % (*m/m*) of water.

2 Normative references

The following referenced documents are indispensable for the application of this document. 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*

3 Terms and definitions

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

NOTE These definitions only apply to waste. In standards dealing with other matrices other terms and definitions exist for technical reasons.

3.1
dry residue
w_{dr}
remaining mass fraction of a sample after a drying process at 105 °C as specified in this European Standard

3.2
water content
w_{w}
mass fraction of water in a sample determined by Karl-Fischer-titration as specified in this European Standard

3.3
dry matter
w_{dm}
mass fraction of a sample excluding water expressed as a percentage by mass calculated by determination of dry residue or water content according to this European Standard

4 Principle

4.1 General

Depending on 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. In case of multiphase 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 in an oven at (105 ± 3) °C. This method applies to solid samples and samples which become solid during the drying process. 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 — Karl-Fischer-titration (water content)

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. The method usually applies to liquid samples and homogenised multiphase samples. It could 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.

5 Sample preparation and storage

Specification of sample preparation is not included in this European Standard. As the dry matter content is calculated in order to convert obtained results for a sample to the dry matter basis, the determination of dry residue or water content shall be carried out on the identical portions of the same test sample. By that the sample preparation is as specified in the actual analysis standard.

Storage of samples may cause changes as e.g. uptake or liberation of water, carbon dioxide and other volatiles. If it is not possible to carry out the determination of dry residue or water content parallel to the analyses, samples shall be stored in air-tight containers. Biologically active samples shall be stored at about 4 °C and analysed within 3 days.

6 Method A — Drying at 105 °C

6.1 General

When carrying out one or more analysis on a sample, the dry matter shall be determined on an identical test portion in parallel in order to recalculate the results of the other analysis to the dry matter basis.

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 could be carried out in a nitrogen atmosphere.

6.3 Hazards

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

6.4 Equipment

6.4.1 Drying system thermostatically controlled and capable of maintaining a temperature of (105 ± 3) °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 Analytical 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 or ceramic.

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 waste 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 ± 3) °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 (m_a).

Depending on the expected dry residue, weigh into the evaporating dish or crucible 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 (m_b).

Place the evaporating dish or crucible (6.4.4) containing the sample in the drying system (6.4.1) set at (105 ± 3) °C until the residue appears dry, typically overnight.

NOTE 1 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, (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.

Otherwise repeat the drying process.

NOTE 2 In case of weight inconstancy after three cycles the drying process may be stopped (after at least 12 h). The result of the last weighing should be recorded in the test report.

In case of infrared or halogen lamp drying systems the corresponding manufacturer's instructions apply. The technique of choice has to be noted in the test report.

Other techniques than oven drying (infrared/halogen) are allowed, provided they are proven to give comparable results.

6.6 Calculation of results

The dry residue, expressed as mass fraction in per cent or in grams per kilogram, is calculated according to Equation (1):

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

where

w_{dr} is the dry residue of the sample, expressed as mass fraction in per cent [%] 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 per cent [%] and $f = 1\ 000$ for expression of results in grams per kilogram [g/kg].

The analysis is performed at least in duplicate; the mean value is calculated and reported.

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 interfere. 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 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 Equipment

Usual laboratory glassware and the following equipment shall be used.

7.3.1 Apparatus suitable for Karl-Fischer-titration.

7.3.2 Shaking apparatus.

7.3.3 Weighing pipette.

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

7.4 Procedure

7.4.1 Determination of the equivalence factor

Fill a clean and dry 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 by use of a weighing pipette (7.3.3) approximately 80 mg of water (W) with an accuracy of 0,1 %. Titrate to the end point and note the consumption (V_1).

The equivalence factor F, expressed as mass concentration in milligrams per millilitre, is calculated according to Equation (2):

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

where

F is the equivalence factor, expressed in milligrams 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

Treat the liquid sample as follows:

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).

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

The water content of the liquid sample, expressed as mass fraction in per cent, is calculated according to Equation (3):

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

where

w_{W} is the water content, expressed as mass fraction in per cent [%];

V is the added volume of the titration liquid, expressed in millilitres [ml];

F is the equivalence factor (according to Equation (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 is to be calculated by Equation (6).

7.4.3 Analysis of solid samples

Treat the solid phases as follows:

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 per cent, is calculated according to Equation (4):

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

where

w_{W} is the water content of phase i, expressed as mass fraction in per cent [%];

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 Equation (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 according to Equation (6), Clause 9.

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

8 Performance characteristics

The performance data of Methods A and B given in Table 1 and Table 2 have been established in a European Intercomparison study on 6 samples of waste and sludge carried out in 2005.

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

Sample	**O** %	p	N	x % w/w	s_R	S_R %	s_r	S_r %
Contaminated soil	12	33	97	93,26	0,45	0,48	0,10	0,10
Dredged sludge	0	32	94	66,43	0,66	0,98	0,26	0,39
Nickel sludge	6	33	98	52,95	1,08	2,04	0,34	0,60

p	Number of laboratories
N	Number of observed values
O	Percentage of outliers
x	Mean value
s_R	Estimate of the reproducibility standard deviation
s_r	Estimate of the repeatability standard deviation
S_R	Estimate of the relative reproducibility standard deviation expressed in per cent
S_r	Estimate of the relative repeatability standard deviation expressed in per cent

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

Sample	**O** %	p	N	x % w/w	s_R	S_R %	s_r	S_r %
Contaminated soil	11	18	54	6,44	1,07	16,63	0,23	3,54
Distillation residue	0	22	66	9,04	0,84	9,34	0,20	2,17
Dredged sludge	18	17	51	30,69	3,48	11,32	0,78	2,55
Drilling solution	0	22	66	97,44	1,88	1,93	1,03	1,05
Nickel sludge	12	18	54	38,38	4,95	12,58	0,76	1,92
Waste oil	14	21	63	69,19	1,96	2,83	0,83	1,20

p	Number of laboratories
N	Number of observed values
O	Percentage of outliers
x	Mean value
s_R	Estimate of the reproducibility standard deviation
s_r	Estimate of the repeatability standard deviation
S_R	Estimate of the relative reproducibility standard deviation expressed in per cent
S_r	Estimate of the relative repeatability standard deviation expressed in per cent

9 Calculation of dry matter

The dry matter, expressed as mass fraction in per cent or in grams per kilogram, is calculated according to Equations (5) or (6):

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

where

w_{dm} is the dry matter, expressed as mass fraction in per cent [%] or in grams per kilogram [g/kg];

w_{dr} is the dry residue, expressed as mass fraction in per cent [%] or in grams per kilogram [g/kg];

or:

$$w_{\mathrm{dm}} = 100 - w_{\mathrm{w}} \qquad (6)$$

where

w_{dm} is the dry matter, expressed as mass fraction in per cent [%] or in grams per kilogram [g/kg];

w_{w} is the water content, expressed as mass fraction in per cent [%] or in grams per kilogram [g/kg].

10 Test report

The test report shall contain at least the following details:

a) reference to this European Standard, i.e. EN 14346 and the method (A or B) used;

b) all data which are necessary to identify the sample;

c) results according to methods A and/or B and the calculated dry matter according to Clause 9;

d) details of all procedural steps which deviate from this standard together with all circumstances that may have influenced the results;

e) type and technique of drying procedure.

Annex A
(informative)

Additional validation data

A.1 General

Except for direct validation data a validation study contains a number of additional data which may be of relevance for the users of this European Standard.

A.2 Type of samples and sample preparation

a) Contaminated soil

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

b) 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.

c) 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.

d) Drilling solution

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.

e) Waste oil

This sample was from an oil regeneration plant. It was a mixture of waste engine oil and hydraulic fluid. It was homogenized in the same way as in the case of the drilling solution.

f) 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.1.

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.1 — 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 results by different methods

In Table A.2 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.2 — Calculated dry matter from all different methods

Material	Dry matter [% *w/w*] 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.

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

B.5 Equipment

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, rasching 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, Rasching 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, expressed as mass fraction in per cent or grams per kilogram, is calculated according to Equation (B.1):

$$w_{\mathrm{W}} = \frac{V \times \delta \times f}{m_{\mathrm{i}}} \tag{B.1}$$

where

w_{W} is the water content of the sample, expressed as mass fraction in per cent [%] 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, expressed in grams [g];

f is the conversion factor $f = 100$ for expression of results as mass fraction in per cent [%] 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 may 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

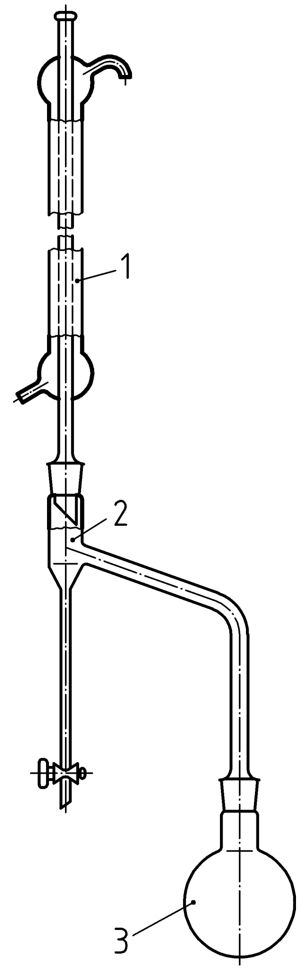
B.9 Performance data

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

Sample	**O** %	p	N	x % w/w	s_R	S_R %	s_r	S_r %
Contaminated soil	0	8	24	6,77	1,12	16,52	0,44	6,51
Dredged sludge	0	8	24	32,44	2,29	7,07	1,83	5,65
Drilling solution	14	7	21	97,37	2,12	2,18	1,80	1,85
Nickel sludge	0	7	21	43,21	2,73	6,32	1,36	3,14
Waste oil	14	7	21	69,30	2,39	3,44	2,04	2,94

p	Number of laboratories
N	Number of observed values
O	Percentage of outliers
x	Mean value
s_R	Estimate of the reproducibility standard deviation
s_r	Estimate of the repeatability standard deviation
S_R	Estimate of the relative reproducibility standard deviation expressed in per cent
S_r	Estimate of the relative repeatability standard deviation expressed in per cent



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 pre-treatment processes. The information given should be helpful to prepare a sampling plan.

Requirements not mentioned in this European Standard are considered as recommendations.

Table C.1

Matrix restrictions	No restrictions.
Typical working range	> 1 % (*m/m*) of dry matter 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.

blank

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