BRITISH STANDARD

BS EN 15560:2009

Fertilizers — Determination of total nitrogen in calcium cyanamide nitrate free

ICS 65.080

BSI British Standards

National foreword

This British Standard is the UK implementation of EN 15560:2009. It supersedes DD CEN/TS 15560:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/37, Fertilisers and related chemicals.

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

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English Version

Fertilizers - Determination of total nitrogen in calcium cyanamide nitrate free

Engrais - Dosage de l'azote total dans la cyanamide calcique exempte de nitrate

Düngemittel - Bestimmung von Gesamtstickstoff in nitratfreiem Kalkstickstoff

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Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 15560:2009) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, 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of an identical text or by endorsement, at the latest by July 2009, and conflicting national standards shall be withdrawn at the latest by July 2009.

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1 Scope

This European Standard specifies a method for the determination of total nitrogen in nitrate-free calcium cyanamide.

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 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

EN 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1:1999 and EN 12944-2:1999 apply.

4 Principle

Digestion in sulfuric acid with the aid of a catalyst. Distillation of the ammonia from an alkaline solution, absorption in an excess of standard sulfuric acid solution and back-titration with standard sodium or potassium hydroxide solution.

5 Reagents

5.1 General

Use only reagents of recognized analytical grade and distilled or demineralized water, free from carbon dioxide and all nitrogenous compounds (grade 3 according to EN ISO 3696:1995).

5.2 Diluted sulfuric acid, mix one volume of sulfuric acid, ρ = 1,84 g/ml, with one volume of water.

5.3 Potassium sulphate, p.a.

5.4 Catalyst

Use 0,3 g to 0,4 g of copper(II)oxide or 0,95 g to 1,25 g of copper(II)sulfate pentahydrate for each determination.

5.5 Sodium hydroxide solution, 30 % mass concentration, of approximately ρ(NaOH) = 1,33 g/ml, ammonia free

5.6 Sulfuric acid, c = 0,05 mol/l.

5.7 Sodium or potassium hydroxide solution, carbonate free, c = 0,1 mol/l.

5.8 Sulfuric acid, c = 0,1 mol/l.

5.9 Sodium or potassium hydroxide solution, carbonate free, c = 0,2 mol/l.

5.10 Sulfuric acid, c = 0,25 mol/l.

5.11 Sodium or potassium hydroxide solution, carbonate free, c = 0,5 mol/l.

5.12 Indicator solutions

5.12.1 Mixed indicator

Solution A: Dissolve 1 g of methyl red in 37 ml of sodium hydroxide solution c = 0,1 mol/l and make up to 1 l with water.

Solution B: Dissolve 1 g of methylene blue in water and make up to 1 l.

Mix one volume of solution A with two volumes of solution B.

This indicator is violet in acid solution, grey in neutral solution and green in alkaline solution. Use 0,5 ml (10 drops) of this indicator solution.

5.12.2 Methyl red indicator solution

Dissolve 0,1 g of methyl red in 50 ml of 95 % ethanol. Make up to 100 ml with water and filter if necessary. This indicator may be used (4 to 5 drops) instead of that specified in 5.12.1.

5.13 Anti-bump granules of pumice stone, washed in hydrochloric acid and calcined.

5.14 Potassium thiocyanate, p.a.

6 Apparatus

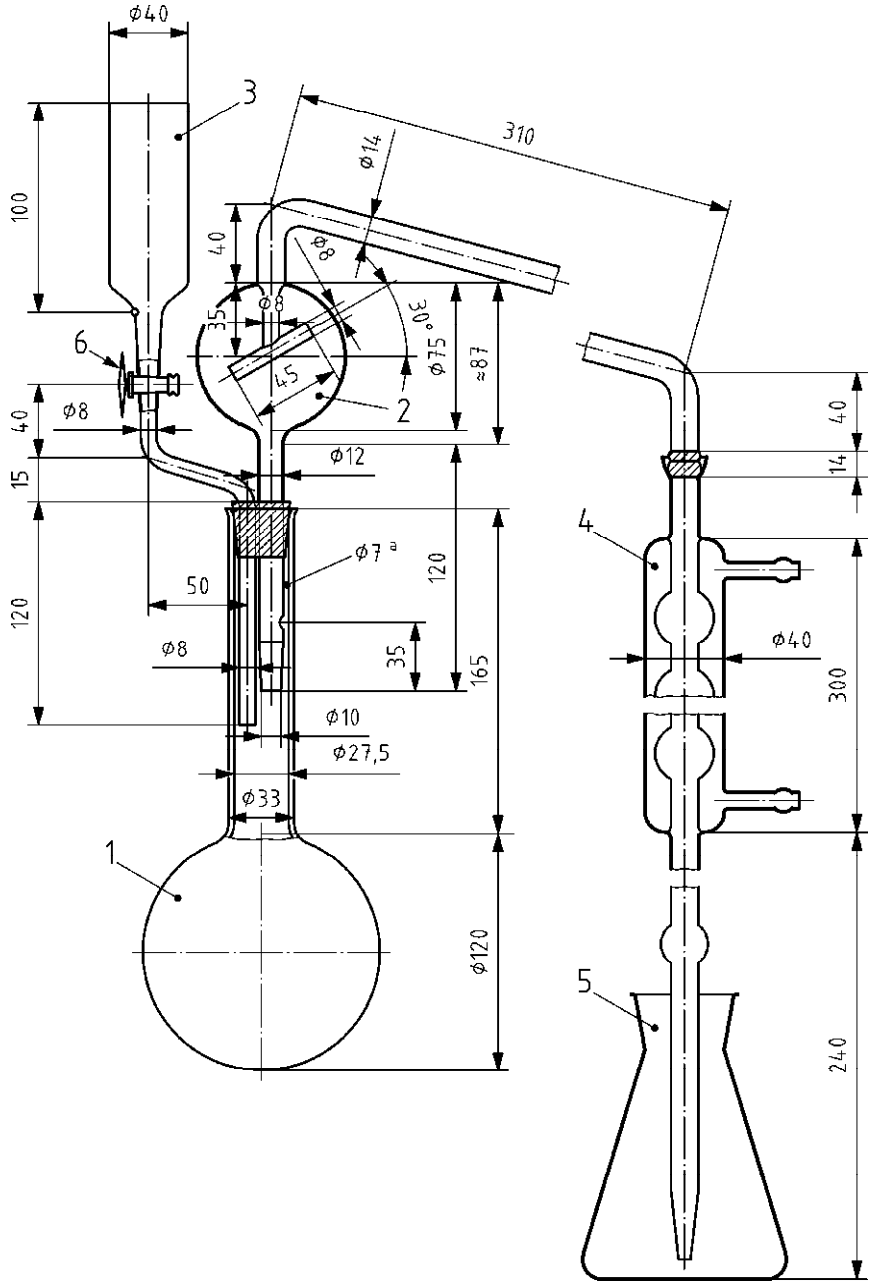
6.1 Distillation apparatus

Consisting of a round-bottomed flask of suitable capacity connected to a condenser by means of a splash head. The equipment is made of borosilicate glass.

NOTE The different types of equipment recommended for this determination are reproduced, showing all the features of construction, in Figures 1, 2, 3 and 4.

An automatic distillation apparatus may also be used, provided that the results are statistically equivalent.

Dimensions in millimetres

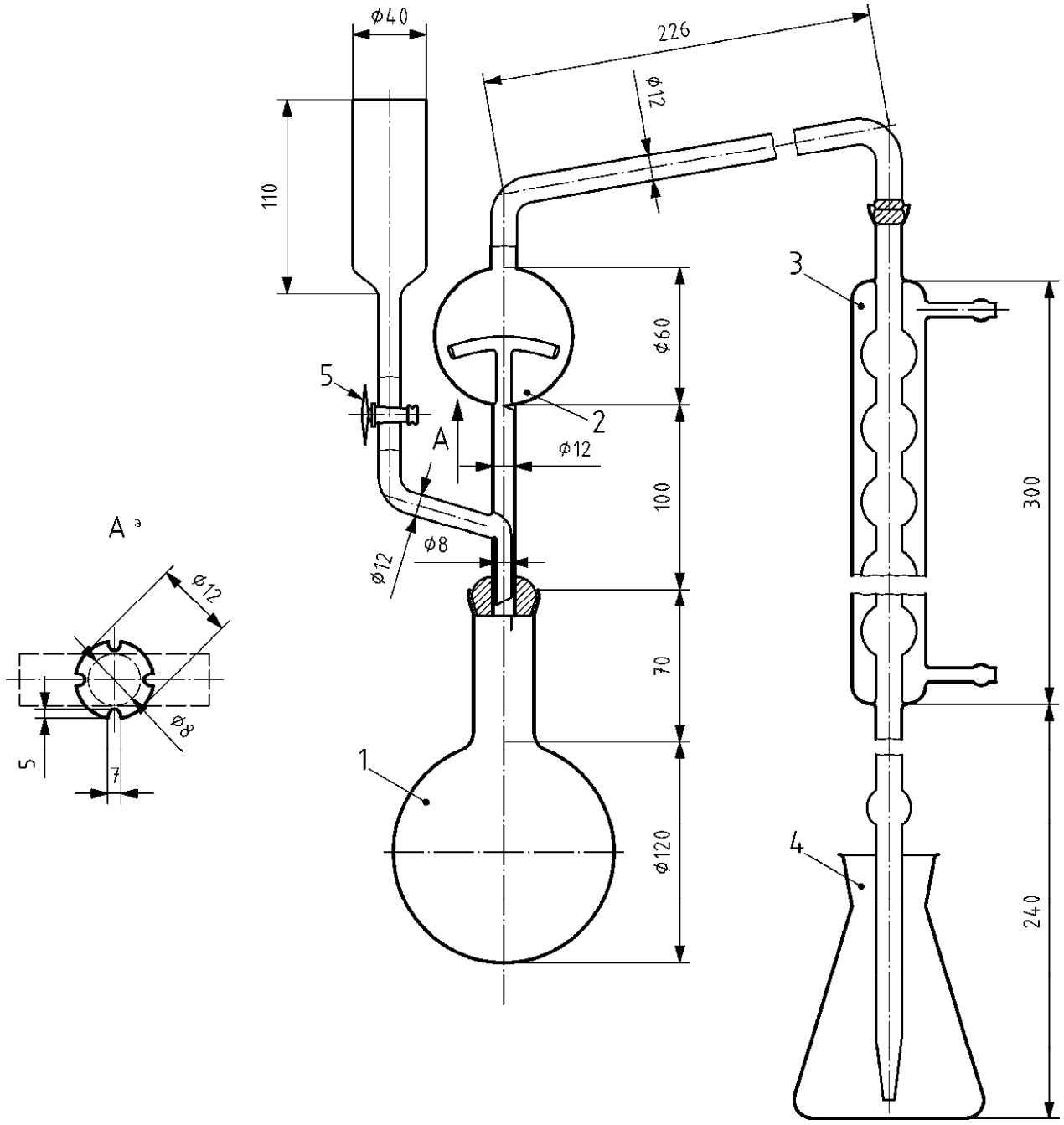


Key

1 round-bottomed, long-necked flask of 1 000 ml capacity
2 distillation tube with a splash head, connected to the condenser by means of a spherical joint (No 18) (the spherical joint for the connection to the condenser may be replaced by an appropriate rubber connection)
3 funnel with a polytetrafluoroethylene (PTFE) tap (6) for the addition of sodium hydroxide (the tap may likewise be replaced by a rubber connection with a clip)
4 six-bulb condenser with spherical joint (No 18) at the entrance, and joined at the issue to a glass extension tube by means of a small rubber connection (when the connection to the distillation tube is effected by means of a rubber tube, the spherical joint may be replaced by a suitable rubber bung)
5 500 ml flask in which the distillate is collected
6 PTFE-tap
[a] hole

Figure 1 — Distillation apparatus 1

Dimensions in millimetres

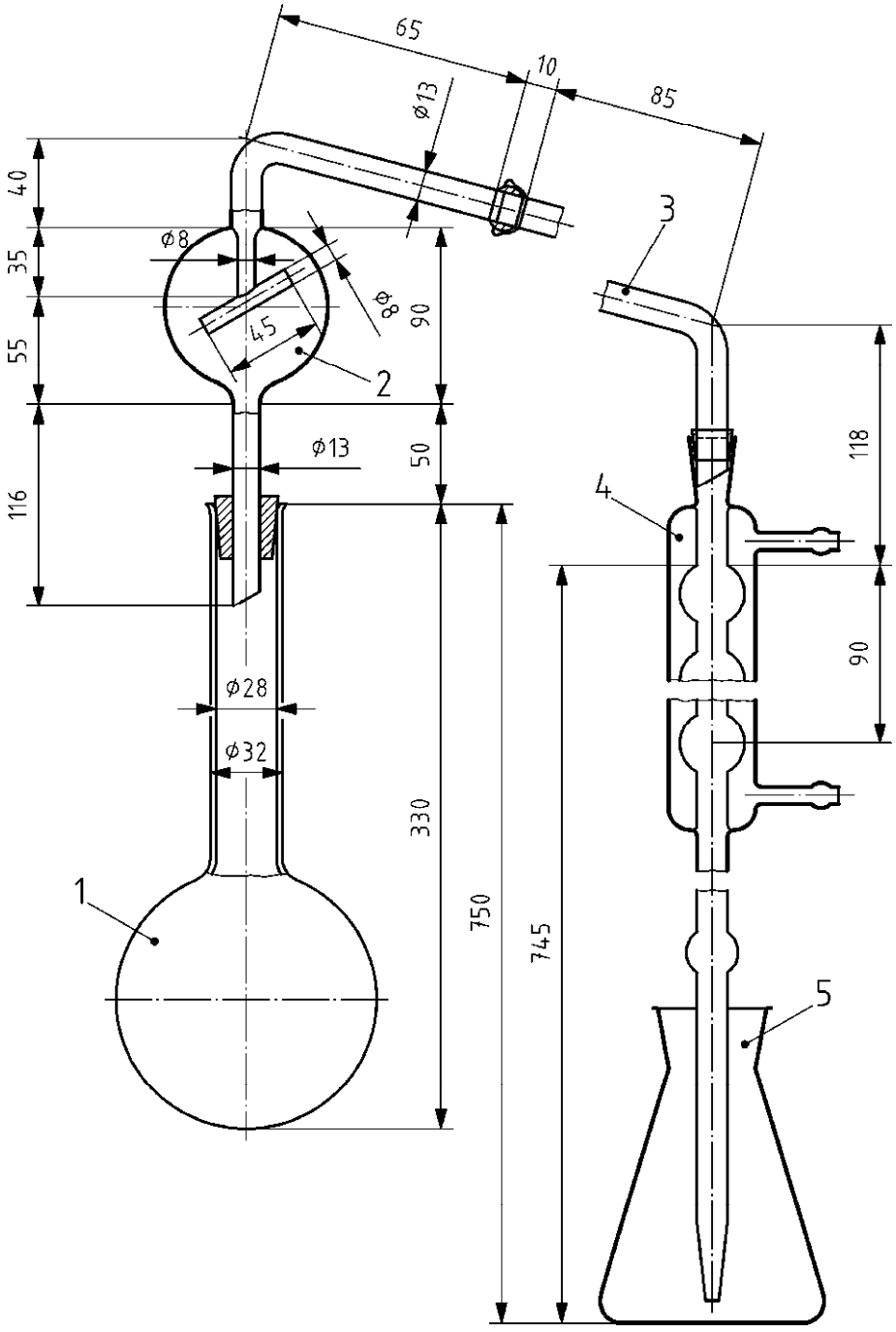


Key

1 round-bottomed, short-necked flask of 1 000 ml capacity with a spherical joint (No 35)
2 distillation tube with a splash head, equipped with a spherical joint (No 35) at the entrance and a spherical joint (No 18) at the issue, connected at the side to a funnel with a polytetrafluoroethylene (PTFE) tap (5) for the addition of sodium hydroxide
3 six-bulb condenser with a spherical joint (No 18) at the entrance and joined at the issue to a glass extension tube by means of a small rubber connection
4 500 ml flask in which the distillate is collected
5 PTFE-tap
[a] enlarged description

Figure 2 — Distillation apparatus 2

Dimensions in millimetres

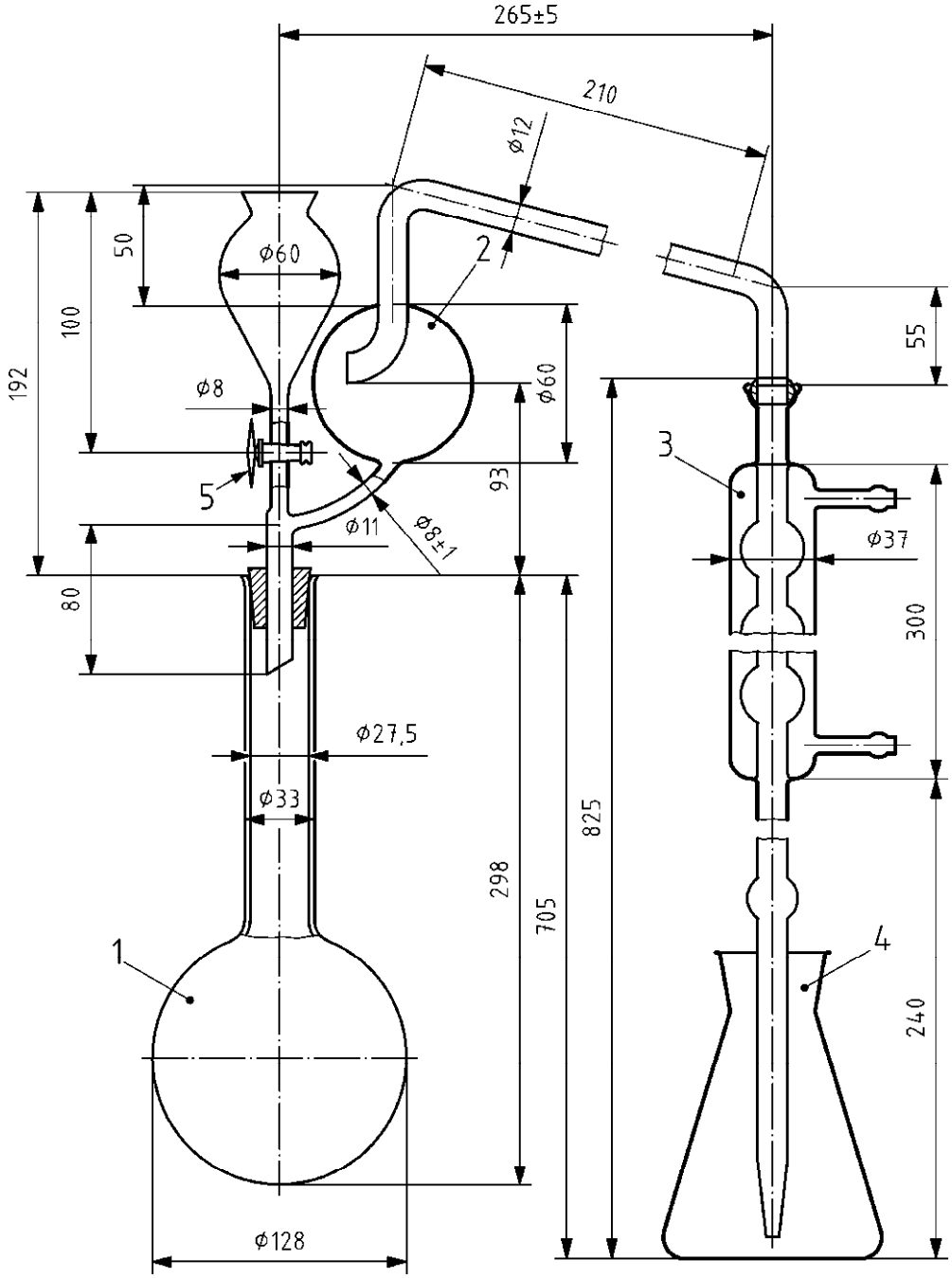


Key

1 round-bottomed, long-necked flask of 750 ml or 1 000 ml capacity with, a bell mouth
2 distillation tube with a splash head and a spherical joint (No 18) at the issue
3 elbow tube with a spherical joint (No 18) at the entrance and a drip cone (the connection to the distillation tube may be effected by means of a rubber tube instead of a spherical joint)
4 six-bulb condenser joined at the issue to a glass extension tube by means of a small rubber connection
5 500 ml flask in which the distillate is collected

Figure 3 — Distillation apparatus 3

Dimensions in millimetres



Key

1 round-bottomed, long-necked flask of 1 000 ml capacity with a bell mouth
2 distillation tube with a splash head and a spherical joint (No 18), at the issue, connected at the side to a funnel with a polytetrafluoroethylene (PTFE) tap (5) for the addition of sodium hydroxide (a suitable rubber bung may be used instead of the spherical joint; the tap may be replaced by a rubber connection with an appropriate clip)
3 six-bulb condenser with a spherical joint (No 18) at the entrance, joined at the issue, by a rubber connection, to a glass extension tube (when the connection to the distillation tube is effected by means of a rubber tube, the spherical joint may be replaced by a suitable rubber bung)
4 500 ml flask for the collection of the distillate
5 PTFE-tap

Figure 4 — Distillation apparatus 4

6.2 Kjeldahl flask, long necked and of suitable capacity.

6.3 Pipettes, of capacity 50 ml, 100 ml and 200 ml.

6.4 Graduated flask, of capacity 250 ml.

7 Sampling and sample preparation

Sampling is not part of the method specified in this Technical Specification. A recommended sampling method is given in EN 1482-1.

Sample preparation shall be carried out in accordance with EN 1482-2.

8 Procedure

8.1 Preparation of the solution

Weigh, to the nearest 0,001 g, 1 g of the sample and place it in the Kjeldahl flask. Add 50 ml of diluted sulfuric acid (5.2), 10 g to 15 g of potassium sulfate (5.3) and the appropriate amount of catalyst (5.4). Heat slowly to drive off the water, boil gently for 2 h, allow to cool and dilute with 100 ml to 150 ml of water. Cool again, transfer quantitatively the suspension to a graduated 250 ml flask, and make up the volume with water, shake and filter through a dry filter into a dry flask.

8.2 Analysis of the solution

According to the variant chosen, place in the receiving flask a measured quantity of standard sulfuric acid as indicated in Table 1, Table 2 or Table 3. Add the appropriate quantity of the chosen indicator solution (5.12.1 or 5.12.2) and, if necessary, water in order to obtain a volume of at least 50 ml. The end of the extension tube of the condenser shall be below the surface of the solution.

With a pipette, transfer, according to the variant chosen, 50 ml, 100 ml or 200 ml of the solution obtained as described in 8.1, and distil the ammonia as described below, adding sufficient NaOH solution (5.5) to ensure a considerable excess. Add water in order to obtain a total volume of about 350 ml and several grains of pumice in order to control the boiling.

Assemble the distillation apparatus, and taking care to avoid any loss of ammonia, add to the contents of the distillation flask 10 ml of concentrated sodium hydroxide solution (5.5). Gradually warm the flask, to avoid boiling vigorously. When boiling commences, distil at the rate of about 100 ml in 10 min to 15 min; the total volume of distillate should be about 250 ml. The condenser shall be regulated so that a continuous flow of condensate is ensured. The distillation should be completed in 30 min to 40 min. When no more ammonia is likely to be evolved, lower the receiving flask so that the tip of the condenser extension is above the surface of the liquid.

Test the subsequent distillate by means of an appropriate reagent to ensure that all the ammonia is completely distilled. Wash the condenser extension with a little water and titrate the surplus acid with the standard solution of sodium or potassium hydroxide prescribed for the variant adopted (see NOTE).

NOTE Standard solutions of different strengths may be used for the back titration provided that the volumes used for the titration do not, as far as possible, exceed 45 ml.

Table 1 — Weighing, dilution and calculation variant a

Declaration **% *N***	**Amount to be weighed** **g**	**Dilution** **ml**	**Solution of sample to be distilled** **ml**	**Expression of the result [a]** **% *N* = (50 – *A*) *F***
0 to 5	10	500	50	$(50 - A) \times 0{,}14$
5 to 10	10	500	25	$(50 - A) \times 0{,}28$
10 to 15	7	500	25	$(50 - A) \times 0{,}40$
15 to 20	5	500	25	$(50 - A) \times 0{,}56$
20 to 40	7	500	10	$(50 - A) \times 1{,}00$
Approximate maximum quantity of nitrogen to be distilled: 50 mg. Sulfuric acid 0,05 mol/l to be placed in the receiving flask: 50 ml. Back titration with NaOH or KOH 0,1 mol/l.				
[a] For the purposes of the equation for expression of the result: — 50 = volume of standard solution of sulfuric acid to be placed in the receiving flask, in millilitres; — *A* = volume of sodium or potassium hydroxide used for the back titration, in millilitres; — *F* = factor comprising the amount weighed, the dilution, the aliquot part of solution of the sample to be distilled and the volumetric equivalent.				

Table 2 — Weighing, dilution and calculation variant b

Declaration **% *N***	**Amount to be weighed** **g**	**Dilution** **ml**	**Solution of sample to be distilled** **ml**	**Expression of the result [a]** **% *N* = (50 – *A*) *F***
0 to 5	10	500	100	$(50 - A) \times 0{,}14$
5 to 10	10	500	50	$(50 - A) \times 0{,}28$
10 to 15	7	500	50	$(50 - A) \times 0{,}40$
15 to 20	5	500	50	$(50 - A) \times 0{,}56$
20 to 40	7	500	20	$(50 - A) \times 1{,}00$
Approximate maximum quantity of nitrogen to be distilled: 100 mg. Sulfuric acid 0,1 mol/l to be placed in the receiving flask: 50 ml. Back titration with NaOH or KOH 0,2 mol/l.				
[a] For the purposes of the equation for expression of the result: — 50 = volume of standard solution of sulfuric acid to be placed in the receiving flask, in millilitres; — *A* = volume of sodium or potassium hydroxide used for the back titration, in millilitres; — *F* = factor comprising the amount weighed, the dilution, the aliquot part of solution of the sample to be distilled and the volumetric equivalent.				

Table 3 — Weighing, dilution and calculation variant c

Declaration % ***N***	**Amount to be weighed** g	**Dilution** ml	**Solution of sample to be distilled** ml	**Expression of the result** [a] % ***N*** = (35 – ***A***) ***F***
0 to 5	10	500	200	$(35 - A) \times 0{,}175$
5 to 10	10	500	100	$(35 - A) \times 0{,}350$
10 to 15	7	500	100	$(35 - A) \times 0{,}500$
15 to 20	5	500	100	$(35 - A) \times 0{,}700$
20 to 40	5	500	50	$(35 - A) \times 1{,}400$
Approximate maximum quantity of nitrogen to be distilled: 200 mg. Sulfuric acid 0,25 mol/l to be placed in the receiving flask: 35 ml. Back titration with NaOH or KOH 0,5 mol/l.				

[a] For the purposes of the equation for expression of the result:

— 35 = volume of standard solution of sulfuric acid to be placed in the receiving flask, in millilitres;

— *A* = volume of sodium or potassium hydroxide used for the back titration, in millilitres;

— *F* = factor comprising the amount weighed, the dilution, the aliquot part of solution of the sample to be distilled and the volumetric equivalent.

8.3 Blank

Carry out a blank test (omitting the sample) under the same conditions and refer to this in the calculation of the final result.

8.4 Control test

Before carrying out the analysis, check that the apparatus is working properly and that the correct technique is applied, using an aliquot part of a standard solution of potassium thiocyanate (5.14), approximating to the concentration of nitrogen in the sample.

9 Calculation and expression of the result

Calculate the *N* content as a percentage mass fraction of the fertilizer as received for analysis according to Table 1 for variant a, Table 2 for variant b or Table 3 for variant c.

10 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) test method used with reference to this European Standard, i.e. EN 15560;

c) test results obtained expressed as percentage mass fraction of nitrogen in the fertilizer;

d) date of sampling and sampling procedure (if known);

e) date when the analysis was finished;

f) all operating details not specified in this document, or regarded as optional, together with details of any incidents that occurred when performing the method which might have influenced the test result(s).

Bibliography

[1] EN 1482-1, Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling

[2] Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers, Official Journal L 304, 21/11/2003, P. 0001-0194, Annex IV, method 2.3.1

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BSI Group
Headquarters 389
Chiswick High Road,
London, W4 4AL, UK
Tel +44 (0)20 8996 9001
Fax +44 (0)20 8996 7001
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