BRITISH STANDARD

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Fertilizers — Determination of total nitrogen in fertilizers containing nitrogen only as nitric, ammoniacal and urea nitrogen by two different methods

ICS 65.080

British Standards

National foreword

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

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Engrais - Détermination de la teneur en azote totale des engrais ne contenant l'azote que sous forme nitrique, ammoniacale et uréique selon deux méthodes différentes

Düngemittel - Bestimmung von Gesamtstickstoff in Düngemitteln mit Stickstoff in Form von Ammonium, Nitrat und Harnstoff unter Anwendung von zwei verschiedenen Verfahren

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Foreword

This European Standard (EN 15750:2009) 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of an identical text or by endorsement, at the latest by April 2010, and conflicting national standards shall be withdrawn at the latest by April 2010.

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Introduction

This document specifies two different methods for the determination of the total nitrogen content in fertilizers. Based on the statistical results of the inter-laboratory tests, obtained with the same samples, the two methods produce equivalent results, and hence can be used both on decision of the user.

1 Scope

This European Standard specifies two different methods (Methods A and B) for the determination of the total nitrogen content in fertilizers. Method A specifies the titrimetric method after distillation according to ISO 5315:1984. Method B specifies a method by reduction of nitrate with iron and tin(II)-chloride.

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 385, *Laboratory glassware — Burettes (ISO 385:2005)*

EN ISO 3696, *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

4.1 Method A – Titrimetric method after distillation according to ISO 5315:1984

Reduction of nitrate to ammonia by chromium powder in acid medium. Conversion of organic and urea nitrogen into ammonium sulfate by digestion with concentrated sulfuric acid in the presence of a catalyst. Distillation of the ammonia from an alkaline solution, absorption in an excess of standard volumetric sulfuric acid solution and back-titration with standard volumetric sodium hydroxide solution in the presence of methyl red or screened methyl red as indicator.

4.2 Method B – Reduction of nitrate with iron and tin(II)-chloride

Reduction of nitrate to ammonia by iron powder and tin chloride in acid medium. Conversion of organic and urea nitrogen into ammonium sulfate by digestion with concentrated sulfuric acid in the presence of a catalyst. Distillation of the ammonia from an alkaline solution, absorption in an excess of standard volumetric sulfuric acid solution and back-titration with standard volumetric sodium hydroxide solution in the presence of an indicator solution.

5 Sampling and sample preparation

Sampling is not part of the methods specified in this European Standard. A recommended sampling method is given in EN 1482-1.

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

6 Method A

6.1 Reagents

Use only reagents of recognized analytical grade having, in particular, low nitrogen contents and distilled or demineralized water (grade 3 according to EN ISO 3696).

6.1.1 Chromium metal, powder of particle size less than or equal to 250 µm.

6.1.2 Aluminium oxide, fused, pumice is suitable.

6.1.3 Anti-foaming agent, for example paraffin wax of melting point not lower than 100 °C, or a silicone.

6.1.4 Ammonium nitrate, dried at 100 °C to constant mass.

6.1.5 Digestion catalyst mixture, finely ground, comprising

- potassium sulfate (K_2SO_4): 1 000 g;
- copper(II) sulfate pentahydrate ($CuSO_4.5H_2O$): 50 g.

6.1.6 Sulfuric acid, concentrated, ρ approximately 1,84 g/ml.

6.1.7 Hydrochloric acid, concentrated, ρ approximately 1,18 g/ml.

6.1.8 Sodium hydroxide, approximately 400 g/l solution.

6.1.9 Sodium hydroxide, standard volumetric solution, c(NaOH) = 0,10 mol/l.

6.1.10 Sulfuric acid, standard volumetric solution, $c(H_2SO_4)$ = 0,25 mol/l.

6.1.11 Sulfuric acid, standard volumetric solution, $c(H_2SO_4)$ = 0,10 mol/l.

6.1.12 Sulfuric acid, standard volumetric solution, $c(H_2SO_4)$ = 0,05 mol/l.

6.1.13 Indicator solution,

use either the screened methyl red solution (6.1.13.1) or the methyl red solution (6.1.13.2).

6.1.13.1 Screened methyl red, ethanolic indicator solution;

mix 50 ml of a 2 g/l ethanolic solution of methyl red with 50 ml of a 1 g/l ethanolic solution of methylene blue.

6.1.13.2 Methyl red, ethanolic indicator solution;

dissolve 0,1 g of methyl red in 50 ml of 95 % (volume fraction) ethanol.

6.1.14 pH indicator paper, wide range.

6.2 Apparatus

Common laboratory equipment and glassware, in particular equipment according to 6.2.1 to 6.2.5.

6.2.1 Digestion apparatus, comprising an 800 ml Kjeldahl flask and a pear-shaped hollow glass stopper.

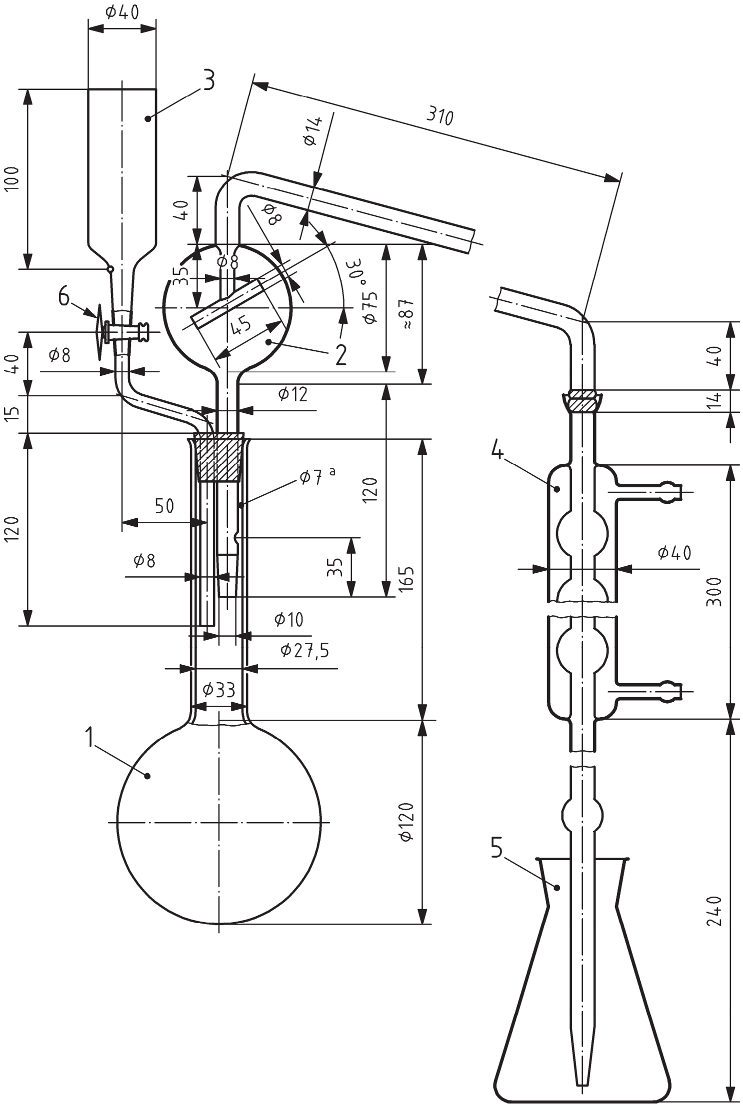
6.2.2 Distillation apparatus

The components of the distillation apparatus may be connected by means of rubber bungs and tubing or by the use of spherical ground glass joints.

Spherical ground glass joints should be held spring clamps to ensure that they are leak tight. Rubber bungs and tubing shall be replaced when they begin to perish or show signs of wear.

Suitable apparatus is illustrated in Figure 1. An automatic distillation apparatus may also be used, provided that the results are statistically equivalent.

Dimensions in millimetres



Key

1 Kjeldahl flask (6.2.1) or round-bottomed, long-necked flask of 1 000 ml capacity

2 distillation tube with a single-bulb 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) (the tap may likewise be replaced by a rubber connection with a clip)

4 seven-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 – Typical distillation apparatus (using a round bottom flask)

6.2.3 Anti-bumping granules, or **anti-bumping device**, consisting of a 100 mm × 5 mm glass rod connected to a 25 mm length of polyethylene tubing.

6.2.4 Two burettes, of capacity 50 ml, according to EN ISO 385, I class A.

6.2.5 Glass beads, of diameter 2 mm to 3 mm.

6.3 Procedure

6.3.1 Test portion

Weight, to the nearest 0,001 g, between 0,5 g and 2,0 g of the test sample, containing not more than 60 mg of nitrate nitrogen and 235 mg of total nitrogen.

6.3.2 Determination

6.3.2.1 Reduction

NOTE 1 This step is not required if nitrate nitrogen is known to be absent.

Transfer the test portion (6.3.1) to the flask (6.2.1) and add sufficient water to make up the total volume to 35 ml. Allow the flask to stand for 10 min with occasional gentle swirling to ensure dissolution of all nitrate salts.

Add 1,2 g of the chromium powder (6.1.1) and 7 ml of the hydrochloric acid solution (6.1.7). Allow the flask to stand for at least 5 min, but not more than 10 min, at ambient temperature.

Place the flask on a heating device in a fume cupboard with the heat input regulated to pass a 7 min to 7,5 min boil test and heat the flask for 4,5 min. Remove from the heat and allow to cool.

NOTE 2 The heat input is that required to bring 250 ml of water at 25 °C to a "rolling" boil in 7 min to 7,5 min.

6.3.2.2 Hydrolysis

NOTE This step can be used instead of the digestion (6.3.2.3) if it is known that the only forms of organic nitrogen present are urea and cyanamide forms.

Stand the flask in a fume cupboard and add 1,5 g of the fused aluminium oxide (6.1.2). Carefully add 25 ml of the sulfuric acid (6.1.6) to the flask. Insert the pear-shaped hollow glass stopper into the neck of the flask and place on a heating device and initially heat until gently boiling. Then adjust the heat input to pass a 7 min to 7,5 min boil test (see 6.3.2.1, NOTE 2).

Continue to heat the flask and contents until dense white fumes of sulfuric acid have been involving for at least 15 min. Allow the flask to cool to room temperature and carefully add 250 ml of water. Allow the flask to cool.

6.3.2.3 Digestion

NOTE This step is necessary only if organic forms of nitrogen other than urea or cyanamide forms are present (see 6.3.2.2) or in the case of fertilizers of unknown composition.

Place the flask in a fume cupboard and add 22 g of the digestion catalyst mixture (6.1.5) and 1,5 g of the fused aluminium oxide (6.1.2). Carefully add 30 ml of the sulfuric acid (6.1.6) to the flask and add 0,5 g of the anti-foaming agent (6.1.3) to reduce foaming. Insert the pear-shaped hollow glass stopper into the neck of the flask and place on a heating device with the heat input adjusted to pass a 7 min to 7,5 min boil test (see 6.3.2.1, NOTE 2).

If considerable foaming occurs, reduce the heat input until this phase is over. Continue to heat the flask and contents until dense white fumes are cleared from the bulb of the flask. Gently swirl the flask and continue digestion for a further 60 min or until the solution is clear, whichever is the longer. Allow the flask to cool to room temperature and carefully add 250 ml of water. Allow the flask to cool.

6.3.2.4 Distillation

If distillation from the round-bottomed flask is preferred, transfer the test portion (6.3.1) or the hydrolysed (6.3.2.2) or digested (6.3.2.3) solution to it quantitatively. Otherwise, place the test portion, or retain the solution, in the Kjeldahl flask. Place the anti-bumping granules or anti-bumping device (6.2.3), in the latter case with the polyethylene in contact with the bottom of the flask, in the flask, and add a few of the glass beads (6.2.5). Assemble the apparatus as shown in Figure 1.

Measure into the receiver (see (5) in Figure 1), according to the expected mass of nitrogen in the test portion, the appropriate volume shown in Table 1 of one of the sulfuric acid solutions (6.1.10, 6.1.11 or 6.1.12).

Table 1 — Expected mass of nitrogen

Expected mass of nitrogen in the test portion mg	Concentration of sulfuric acid solution mol/l	Volume of sulfuric acid solution ml
0 to 30 30 to 50 50 to 65	0,05 (solution 6.1.12)	25,0 40,0 50,0
65 to 80 80 to 100 100 to 125	0,10 (solution 6.1.11)	35,0 40,0 50,0
125 to 170 170 to 200 200 to 235	0,25 (solution 6.1.10)	25,0 30,0 35,0

Add four or five drops of the indicator solution (6.1.13) and place the receiver so that the end of the delivery tube is below the surface of the acid, adding water to the receiver if necessary.

Pour at least 120 ml of the sodium hydroxide solution (6.1.8) or 20 ml if there was neither hydrolysis (6.3.2.2) nor digestion (6.3.2.3), into the dropping funnel and carefully run all but about 2 ml of this solution into the distillation flask. Close the stopcock, leaving the remaining 2 ml in the dropping funnel. Bring the contents of the flask to the boil, increasing the rate of heating progressively until, finally, the contents of the flask are boiling briskly. The contents of the flask shall remain alkaline during the distillation period.

When at least 150 ml of distillate have been collected, partially withdraw the receiver so that the delivery tube rests on the rim of the receiver. Test the subsequent distillate with the pH indicator paper (6.1.14) to ensure that all the ammonia is completely distilled. Remove the source of heat.

Detach the splash head from the condenser and wash the insides of the condenser and expansions bulb with water, collecting the rinsings in the receiver. Also, rinse the outside of the delivery tube and collect the rinsings in the receiver.

6.3.2.5 Titration

Back-titrate the excess of acid with the standard volumetric sodium hydroxide solution (6.1.9) to the neutral colour of the indicator.

6.3.3 Blank test

Carry out a blank test at the same time as the determination, using the same procedure, using the same reagents, but omitting the test portion, and using the 0,05 mol/l standard volumetric sulfuric acid solution (6.1.12).

In the blank test, the poured volume of standard volumetric solution should not exceed 1,0 ml. If the result is greater than 1,0 ml, check the reagents, especially the chromium powder (6.1.1).

6.3.4 Check test

Carry out a periodic check on the efficiency of the apparatus and the accuracy of the method using an aliquot portion of a solution of freshly prepared ammonium nitrate (6.1.4) containing 100 mg of nitrogen. The check shall be made using the same conditions as for the determination and the blank test and using the same indicator.

6.4 Calculation and expression of the results

Calculate the total nitrogen content, w_a, w_b and w_c, expressed as nitrogen (N) as a percentage by mass using Equations (1), (2) and (3):

a) if the 0,05 mol/l sulfuric acid solution (6.1.12) was used:

$$w_a = \frac{[(V_1 - V_2) - (V_3 - V_4)] \times 0{,}140\,1}{m} \tag{1}$$

b) if the 0,10 mol/l sulfuric acid solution (6.1.11) was used:

$$w_b = \frac{[(2V_1 - V_2) - (V_3 - V_4)] \times 0{,}140\,1}{m} \tag{2}$$

c) if the 0,25 mol/l sulfuric acid solution (6.1.10) was used:

$$w_c = \frac{[(5V_1 - V_2) - (V_3 - V_4)] \times 0{,}140\,1}{m} \tag{3}$$

where

V_1 is the volume, in ml, of the sulfuric acid solution (6.1.10, 6.1.11 or 6.1.12 as appropriate) used for the determination;

V_2 is the volume, in ml, of the sodium hydroxide solution (6.1.9) used for the determination;

V_3 is the volume, in ml, of the sulfuric acid solution (6.1.12) used for the blank test;

V_4 is the volume, in ml, of the sodium hydroxide solution (6.1.9) used for the blank test;

m is the mass, in g, of the test portion (6.3.1).

NOTE If the concentrations of the standard volumetric solutions are not exactly as specified in the list of reagents, appropriate corrections should be made.

7 Method B

7.1 Reagents

Use only reagents of recognized analytical grade having, in particular, low nitrogen contents and distilled or demineralized water (grade 3 according to EN ISO 3696).

7.1.1 Iron powder, reduced by hydrogen (ferrum reductum).

A portion of 5 g of the iron powder shall have the capacity to reduce at least 250 mg nitrate.

7.1.2 Tin(II) chloride solution

Dissolve 120 g $SnCl_2 \cdot 2H_2O$ in 400 ml hydrochloric acid (ρ = 1,19 g/ml) and make up the volume to 1 l. The solution shall not be turbid and should be prepared freshly for use.

Before the solution is prepared the tin(II) chloride should be checked for its reduction potential by the following procedure:

- Dissolve 0,5 g $SnCl_2 \cdot 2H_2O$ in 2 ml hydrochloric acid (ρ = 1,19 g/cm^3) and add 50 ml water. Add 5 g potassium-sodium-tartrate ($C_4H_4O_6KNa \cdot 4H_2O$) and mix by stirring. Then, add sodium bicarbonate ($CHNaO_3$) until the solution is alkaline. Check this by a pH indicator paper.
- Titrate this solution with iodine volumetric solution ($c(J_2)$ = 0,05 mol/l) using starch solution as an indicator. 1 ml of the iodine volumetric solution is equal to 0,01128 g $SnCl_2 \cdot 2H_2O$.

7.1.3 Sulfuric acid, concentrated, ρ approximately 1,84 g/ml.

7.1.4 Potassium sulfate, powdered.

7.1.5 Pumice, washed with hydrochloric acid and annealed or glass anti-bumping granules.

7.1.6 Sulfuric acid, standard volumetric solution, $c(1/2\ H_2SO_4)$ = 0,5 mol/l.

7.1.7 Sodium hydroxide solution, approximately 400 g/l.

7.1.8 Sodium hydroxide, standard volumetric solution, $c(NaOH)$ = 0,25 mol/l.

7.1.9 Indicator solution;

dissolve 60 mg methyl red (sodium salt) and 40 mg methylene blue in 100 ml of water.

7.2 Apparatus

Common laboratory equipment and glass ware, in particular equipment according to 7.2.1 to 7.2.2.

7.2.1 Digestion apparatus, with Kjeldahl digestion tubes, long form (approximately 800 ml to 1 000 ml) or an appropriate Kjeldahl digestion flask.

7.2.2 Distillation apparatus, according to Figure 1.

7.3 Procedure

7.3.1 Test portion

Weigh, to the nearest 0,001 g, between 0,5 g to 1,0 g of the test sample, containing not more than 50 mg of nitrate nitrogen.

7.3.2 Determination

7.3.2.1 Reduction

Transfer the test portion (7.3.1) to the digestion tube or flask (7.2.1) and add approximately 5 g iron powder (7.1.1). Carefully add 50 ml tin chloride solution (7.1.2), swirl the flask, allow to stand for 30 min at room temperature with occasional gentle swirling.

Add approximately 30 ml sulfuric acid (7.1.3), 10 g potassium sulfate (7.1.4) and some pieces of pumice or glass anti-bumping granules.

7.3.2.2 Digestion

Place the tube or flask on a heating device in a fume cupboard and start heating it very carefully. If considerable foaming occurs, reduce the heat input until this phase is over. Continue to heat the flask and contents until dense white fumes are cleared from the bulb of the flask. Occasionally gently swirl the flask and continue digestion until the solution is clear. The heat input should be adjusted to such a level that the fumes of sulfuric acid condense in the upper part of the digestion tube or flask. When the solution is clear, heating should be continued for 60 min.

7.3.2.3 Distillation

Allow the flask to cool to room temperature and carefully add water in small portions until the final volume is approximately 350 ml. The solution will become warm when water is added.

If the digestion tube or flask cannot directly be used for distillation, transfer its content quantitatively with water to the flask of the distillation unit. Place some pieces of pumice or anti-bumping granules (7.1.5) in the flask and assemble the distillation apparatus (7.2.2). Measure into the receiver of the distillation apparatus an appropriate volume of sulfuric acid volumetric standard solution (7.1.6) and add water until the end of the delivery tube of the distillation apparatus is 50 mm below the surface of the sulfuric acid.

Pour approximately 100 ml sodium hydroxide solution (7.1.7) into the dropping funnel and carefully run all but about 2 ml of this solution into the distillation flask. Close the stopcock, leaving the remaining 2 ml in the dropping funnel. Bring the content of the flask to the boil, increasing the rate of heating progressively until, finally, the contents of the flask are boiling briskly. The heating should be adjusted to allow distillation of approximately 200 ml of solution within 30 min. Normally under those conditions all of the ammonia has been collected in the receiver after 30 min to 40 min. The contents of the flask shall remain alkaline during the distillation. Remove the heating device when the distillation is complete.

Detach the condenser from the distillation unit and wash the insides of the condenser and expansion bulb with water, collecting the rinsings in the receiver. Also rinse the outside of the delivery tube and collect the rinsings in the receiver.

7.3.2.4 Titration

Add some drops of the indicator solution (7.1.9) and back-titrate the excess of acid with the standard volumetric sodium hydroxide solution (7.1.8) to the (grey) neutral colour of the indicator.

7.3.3 Blank test

Carry out a blank test, at the same time as the determination, using the same procedure, using the same reagents, but omitting the test portion (7.3.1). In the blank test, the poured volume of standard volumetric sulfuric acid solution (7.1.6) should not exceed 0,2 ml. If the result is greater than 0,2 ml, check the reagents.

7.4 Calculation and expression of the results

Calculate the total nitrogen content, expressed as nitrogen (*N*) as a percentage by mass using Equation (4):

$$w_{\mathrm{N}} = \frac{[(V_1 - 0{,}5 \times V_2) - (V_3 - 0{,}5 \times V_4)] \times 0{,}700\,34}{m} \tag{4}$$

where

V_1 is the volume, in millilitres, of the sulfuric acid solution (7.1.6) used for the determination;

V_2 is the volume, in millilitres, of the sodium hydroxide solution (7.1.8) used for the determination;

V_3 is the volume, in millilitres, of the sulfuric acid solution (7.167) used for the blank test;

V_4 is the volume, in millilitres, of the sodium hydroxide solution (7.1.8) used for the blank test;

m is the mass, in grams, of the test portion.

8 Precision of methods A and B

8.1 Inter-laboratory tests

Details of inter-laboratory tests on the precision of the methods A and B are summarized in Annex A. The values derived from this test may not be applicable to concentration ranges and matrices other than those given.

8.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of the cases be greater than the repeatability limits r given in Table 2 (method A) and Table 3 (method B).

8.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of the cases be greater than the reproducibility limits R given in Table 2 (method A) and Table 3 (method B).

Table 2 — Precision data method A

Sample	$\bar{x}$ **(%)**	r **(%)**	R **(%)**
UAN+S	22,1	0,29	1,55

Table 3 — Precision data method B

Sample	$\bar{x}$ **(%)**	r **(%)**	R **(%)**
UAN+S	21,88	0,27	1,32

9 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 (A or B) used with reference to this document, EN 15750;

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) whether the requirement of the repeatability limit has been fulfilled;

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

Annex A
(informative)

Statistical results of the inter-laboratory tests

The precision of the methods was established in 2005 and 2006 by Working Group 7 “Chemical analysis” of CEN/TC 260 “Fertilizers and liming materials” in inter-laboratory tests evaluated in accordance with ISO 5725-1. The statistical results are given in Table A.1.

Table A.1 — Statistical results method A and Method B

Parameter	Sample UAN+S	
	Method A	**Method B**
Number of participating laboratories	12	7
Number of laboratories after elimination of outliers (accepted test results)	12	7
Mean value $\bar{x}$ (%)	22,01	21,88
Repeatability standard deviation s_r (%)	0,10	0,10
Relative Standard Deviation of Repeatability, RSD_r (%)	0,5	0,4
Repeatability limit r (%)	0,29	0,27
Reproducibility standard deviation s_R (%)	0,56	0,47
RSD_R (%)	2,5	2,2
Reproducibility limit R (%)	1,55	1,32

Bibliography

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

[2] ISO 5315:1984, *Fertilizers — Determination of total nitrogen content — Titrimetric method after distillation*

[3] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[4] *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 2.6.2, Annex II

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