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Fertilizers and soil conditioners — Determination of total nitrogen by combustion

Engrais et amendements — Détermination de l'azote total par combustion

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Foreword

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This document was prepared by Technical Committee ISO/TC 134, *Fertilizers, soil conditioners and beneficial substances*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Fertilizers and soil conditioners — Determination of total nitrogen by combustion

1 Scope

This document specifies a method for the determination of total nitrogen content in all nitrogen containing fertilizers by combustion method.

NOTE 1 The presence of non-nutritive sources of nitrogen (e.g. chelating agents) causes positive bias to samples being analysed for nutritive nitrogen content. The non-nutritive nitrogen content is subtracted from the total nitrogen value to determine the nutritive nitrogen content.

NOTE 2 Common internationally traded fertilizers were evaluated for total nitrogen by combustion analysis in the ring-study in this document. While the fertilizers analysed in this international ring study were mineral fertilizers, previous studies have shown that total nitrogen by combustion is suitable for use with many non-mineral, nitrogen-containing fertilizers.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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

ISO 14820-2:2016, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

The sample is combusted at a high temperature of 900 °C or above in the presence of oxygen. Following the reduction of formed nitrogen oxides to elemental nitrogen and the removal of any interfering products of combustion, nitrogen is measured with a thermal-conductivity detector.

5 Apparatus

5.1 **Automatic nitrogen analyser**, based on combustion methods.

5.2 **Analytical balance.**

The accuracy of the balance is a function of the analyser used and the required weighed portions. The resolution should be 0,1 % or better of the weighed portion.

5.3 Auxiliary devices for sample preparation, for example:

— tweezers with a blunt tip;

— micro-spatula with a flattened tip;

— pipette.

The pipette is recommended for weighing in and thus does not need to be calibrated. It is important, however, to obtain a good droplet size (small droplets). Fixed-volume pipettes or pipettes with an adjustable volume in the range from 10 µl to 1 000 µl or single-trip Pasteur pipettes with a fine tip may also be used.

5.4 Customary chemically resistant glass.

6 Chemicals

6.1 Auxiliary combustion agent and other equipment, appropriate for use with the selected nitrogen analyser.

The following materials are merely examples. Other or similar materials may be used as required, depending on the system that is available:

— tin capsule or similar sample containers;

— auxiliary combustion agent, non-nitrogenous saccharide, such as sucrose or cellulose;

— absorbing agent for liquids, non-nitrogenous, such as magnesium oxide or diatomaceous earth.

6.2 Standard substances for nitrogen determination, preferably with certified nitrogen content.

EXAMPLE Suitable standard substances include: ethylenediamine tetraacetic acid (EDTA), nicotinic acid amide, ammonium nitrate, aspartic acid and nicotinic acid.

Low-biuret urea of adequate purity (e.g. crystalline ultra pure or analytical) or other such standard substances recommended by and available from the equipment manufacturer may also be used. Certified standard substances should be preferred.

NOTE Liquid standard substances (e.g. urea solutions) are not suitable for calibration purposes.

6.3 Oxygen, min. 99,999 % O_2.

6.4 Other ultrapure gases, if required to operate the nitrogen analyser, such as helium, min. 99,999 %.

6.5 Other reagents or auxiliary agents, as required by the equipment.

7 Procedure

7.1 General

Fertilizers and soil conditioners should fit the definitions found in ISO 8157. Samples shall be properly prepared to account for small test portions and potential non-uniformity of some fertilizer products in accordance with ISO 14820-2:2016. Liquids should be well mixed and solids should be homogenized and well mixed. A sample that is not sufficiently homogeneous can increase the variability in the results obtained by this method. It is recommended that replicate analysis of each sample be performed to evaluate the homogeneity of the lab samples. Further processing of the non-homogeneous lab samples may be required if the instrumental results show high variability. Replicate values of ±0,20 %*N* have

been obtained for the same sample material when analysed in succession when the sample has been sufficiently prepared.

Different types of apparatus are available on the market. Operation and performance criteria should be based on the respective operation manuals.

7.2 Reference curve

Perform calibration as required for the specific type of analyser and according to the respective operation manuals (e.g. after replacement of the combustion tube, reagent or similar) by performing measurements as described in 7.4. Weigh in an appropriate amount of standard substances repeatedly as appropriate for the respective types of apparatus to obtain a reference curve.

7.3 Inspection and calibration

Use an appropriate standard substance or in-house secondary standard to review the good working order of the apparatus and the reference curve. Preferably, a certified standard should be used.

Frequency of inspection is a function of the analyser used. Use of quality control charts is also advised to monitor trends in instrument performance.

7.4 Measurement

Weigh a portion of the sample in a suitable holder as specified for the type of nitrogen analyser used. The amount should be such that the absolute amount of nitrogen is in the middle range of the reference curve.

Use approximately the threefold amount of combustion agent.

Enter the required data (weighed portion, sample identification) into the analyser (or a control computer) depending on the type of apparatus. Feed the weighed-in sample to the analyser and start combustion. Perform at least three single determinations.

8 Results

8.1 Calculation

Use blank samples to background correct prior to analysing the calibration standards and calculating the calibration curve. Use the apparatus-specific program to calculate the reference curve or the drift correction for the samples. Calculate per cent nitrogen for each sample using the apparatus-specific software.

8.2 Expression of results

Report the mean value for the replicates of the sample provided that each replicate is in good agreement. Express the results as per cent nitrogen.

9 Precision

Grubbs and Cochran tests were used to identify outliers. The recovery for each sample was calculated for samples with noted %N values. Repeatability (r), reproducibility (R), standard deviation of repeatability (s_r) and standard deviation of reproducibility (s_R) are listed in Table 1. Table 2 lists the composition and physical state of each fertilizer used in the ring-study.

Additional data for the evaluation of total nitrogen by combustion with fertilizers is available in AOAC Official Method 993.13[3]. Data for the determination of total nitrogen in urea by combustion is available in ISO 22241-2:2019, Annex B.

Table 1 — Statistical data from the analysis of ring-study samples

Sample ID	Number of data sets	Number of replicates	%*N* by composition	Average of data (%*N*)	Recovery	Repeatability (*r*)	Repeatability standard deviation (s_r)	Reproducibility (*R*)	Reproducibility standard deviation (s_R)
Urea	11	22	46,6	46,40	99,6	0,338	0,120	0,617	0,220
UAN	15	30	31,9	32,32	101,3	0,331	0,118	1,245	0,445
Ammonium sulfate	10	20	21	21,14	100,7	0,168	0,060	0,242	0,087
Ammonium thiosulfate solution	12	24	12	11,82	98,5	0,141	0,051	0,523	0,187
Magruder 170711	13	26	6,54	6,99	106,9	0,374	0,133	0,535	0,191
Magruder 170411	13	26	19,60	19,82	101,1	0,209	0,075	0,450	0,161
Multicut NPK-23	15	30	22,90	22,86	99,8	0,225	0,081	1,260	0,450
Average						0,255	0,091	0,696	0,249

Table 2 — Ring-study fertilizer components and physical states

Sample ID	Physical state	Components
Urea	Solid	Urea
UAN 32	Liquid	Urea, ammonium, nitrate
Ammonium sulfate	Solid	Ammonium, sulfate
Ammonium thiosulfate solution	Liquid	Ammonium, thiosulfate
Magruder 170711[a]	Solid	Ammonium, nitrate, phosphorous, potassium
Magruder 170411[a]	Solid	Ammonium, nitrate, urea, phosphorous, potassium, sulfur
Multicut NPK-23	Solid	Ammonium, nitrate, phosphorous, potassium, sulfur

[a] Samples contain ammonium and phosphate as mono-ammonium phosphate and di-ammonium phosphate.

Bibliography

[1] ISO 8157, *Fertilizers and soil conditioners — Vocabulary*

[2] ISO 22241-2:2019, *Diesel engines — NOx reduction agent AUS 32 — Part 2: Test methods*

[3] AOAC Official Method 993.13, *Nitrogen (Total) in Fertilizers, Combustion method*

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