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Fertilizers and soil conditioners — Determination of total nitrogen by combustion

Matières fertilisantes — Détermination de l'azote total par combustion

ICS: 65.080

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Fertilizers and soil conditioners — Determination of total nitrogen by combustion

1 Scope

This International Standard establishes a method for the determination of total nitrogen content in all nitrogen containing fertilizers by combustion method.

2 Principle

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

3 Normative References

The following referenced 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.

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ISO 3696:1995, *Water for Analytical Laboratory Use- Specification and Test Methods*

ISO/DIS 8157, *Fertilizers and Soil Conditioners-Vocabulary*

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ISO 22241-2:2006, *(E), Annex B, Determination of Urea Content by Total Nitrogen*

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

4 Apparatus

4.1 Automatic nitrogen analyser, based on combustion methods.

4.2 Analytical balance.

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

4.3 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.

4.4 Customary chemically resistant glass.

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5 Chemicals

5.1 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, cellulose.

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

5.2 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 (for example 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 suited for calibration purposes.

5.3 Oxygen, min. 99,999 % O_2.

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

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

6 Procedure

6.1 General

Samples must be properly prepared to account for small test portions and potential non-uniformity of some fertilizer products. 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.

6.2 Reference curve

Perform calibration as required for the specific type of analyser and according to the respective operation manuals (for example, after replacement of the combustion tube, reagent or similar) by performing measurements as described in 6.4. Weigh in an appropriate amount of standard substances repeatedly as appropriate for the respective types of apparatus to obtain a reference curve.

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6.3 Inspection and calibration

Use an appropriate standard substance or in-house secondary stanadard 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.

6.4 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 (3) single determinations.

7 Results

7.1 Calculation

Use blank samples to background correct prior to analysing 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 percent nitrogen for each sample using the apparatus specific software.

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7.2 Expression of Results

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

8 Precision

Sample ID	# Data Sets	# of replicates	%N by Composition	Avg of data (%N)	Recovery	Repeatibility (r)	Repeatibility 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

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Additional data for the evaluation of total nitrogen by combustion with fertilizers is available in AOAC method 933.13.

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*	Solid	Ammonium, Nitrate, Phosphorous, Potassium
Magruder 170411*	Solid	Ammonium, Nitrate, Urea, Phosphorous, Potassium, Sulfur
Multicut NPK-23	Solid	Ammonium, Nitrate, Phosphorous, Potassium, Sulfur
* Samples contain ammonium and phosphate as mono-ammonium phosphate and di-ammonium phosphate.		

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