

Characterization of different forms of Nitrogen in Liquid fertilizers with UDK169 Automatic distillation and titration system

AOAC 920.03 Nitrogen (Ammoniacal) in Fertilizers: Magnesium Oxide Method

AOAC 941.04 Urea and ammoniacal nitrogen: Urease Method

AOAC 892.01 Nitrogen (Ammoniacal and Nitrate) in Fertilizers: Devarda Method

Tested with **VELP Scientifica UDK169 Automatic Kjeldahl Distillation and Titration System** (Code F30200165)



Introduction

A fertilizer is any type of organic or inorganic substance that contains nutrients, macroelements or microelements, in forms assailable by plants, to maintain or increase the content of these elements in the soil, improve the quality of the substrate at the nutritional level, stimulate the vegetative growth of the plants, etc.

Fertilizers play an important role in improving the productivity of farming. Solid and liquid fertilizers are both widely used in the agriculture industry, with the latter gaining up because of their easiness of use.

Liquid fertilizers have many advantages over traditional granular fertilizers. They are able to carry with them different sources of nitrogen in one product, helping to prevent leaching and ensuring at the same time nutrients immediately available in soil.

Different forms of nitrogen are taken up by the plants in different ways, therefore characterizing the type of nitrogen contained in fertilizers is crucial to develop the most efficient fertilizing program.

Here we are going to describe the determination of Ammonia Nitrogen, Urea Nitrogen and Nitrate Nitrogen in liquid fertilizers.

Determination of Ammonia nitrogen (AOAC 920.03)

This method describes the procedure for the determination of ammoniacal nitrogen using Magnesium oxide.

Adding an excess of magnesium oxide will promote a mild alkaline pH, liberation ammonia from any ammonium salts in solution. The sample is then distilled in a steam flow and collected into an acid solution with indicators, ready for the titration step. In UDK169 the steps of distillation and titration are automatic and simultaneous.

Determination of Nitrate and Ammonia nitrogen (AOAC 892.01)

This method describes the procedure for the determination of nitrogen and ammoniacal nitrogen using Devarda's alloy.

When a solution of nitrate ions and Devarda's alloy is mixed with aqueous sodium hydroxide, the mixture gently liberates ammonia gas. This conversion under the form of ammonia, requires some minutes. The sample is then distilled in a steam flow and collected into an acid solution with indicators, ready for the titration step. In UDK169 the steps of distillation and titration are automatic and simultaneous. The results obtained will be comprehensive of the ammonia nitrogen too, so it has to be subtracted afterwards.

Determination of Urea and Ammonia nitrogen (AOAC 941.04)

This method describes the procedure for the determination of urea and ammonia nitrogen using urease solution.

A urease solution is able to catalyse the hydrolysis of urea nitrogen, freeing up ammonia, available to be distilled and determined by titration. The distillation and titration are taking place automatically and simultaneously in the system. The results obtained will be comprehensive of the ammonia nitrogen too, so it has to be subtracted afterwards.

Samples

Three different liquid fertilizer formulation have been tested:

Sample	Expected Ammonia Nitrogen	Expected Nitrate Nitrogen	Expected Urea Nitrogen
Liquid fertilizer 1	1,9 – 2,2%	< 0,1%	< 0,1%
Liquid fertilizer 2	6,0 – 6,3%	0,2 – 0,4%	0,5 – 0,7%
Liquid fertilizer 3	0,3 – 0,4%	4,9 – 5,2%	< 0,1%

Reagent and equipment

- Magnesium oxide
- Vreceiver (Code A00000411)
- Sulfuric acid 0.2 N
- Deionized water
- UDK169 (Code F30200165)
- Devarda's alloy
- Sodium Hydroxide 32 %
- Urease solution (1% commercial urease solution neutralized with HCl 0,1N)
- CaCl₂ solution (prepared adding 25g of CaCl₂ to 100ml of deionized water)

Sample Preparation

Mix the sample thoroughly before using and take the portion needed for the analysis under stirring to ensure the best representativity of the sample.

Distillation and titration

Create three different methods for the forms of Nitrogen described.

Once the methods are created, they will be recorded and be selected to run the specific analysis.

In the methods menu, add the methods selecting the following parameters:

Ammonia Nitrogen

- H₂O: 50 ml
- NaOH: 0 ml
- Receiving solution: 30 ml
- Steam power: 50 %
- Pause: 0 min

Ammonia and Nitrate Nitrogen

- H₂O: 10 ml
- NaOH: 50 ml
- Receiving solution: 30 ml
- Steam power: 50 %
- Pause: 12 min

Ammonia and Urea Nitrogen

- H₂O: 50 ml
- NaOH: 50 ml
- Receiving solution: 30 ml
- Steam power: 50 %
- Pause: 0 min

Set No automatic distillation residues discharge at the end of the distillation.

Results

The urea and nitrate methods will include ammonia nitrogen in the results; therefore, ammonia nitrogen has to be subtracted. (For the values of the singles species refer to table 4).

Tab. 1 Ammonia Nitrogen – AOAC 920.03		
Sample	Sample quantity (g)	N %
Liquid fertilizer 1	1,0131	2,00
	1,0191	2,01
	1,0336	2,02
	Average ± SD%	2,01 ± 0,01
Liquid fertilizer 2	0,5170	6,18
	0,5232	6,17
	0,5126	6,17
	Average ± SD%	6,17 ± 0,01
Liquid fertilizer 3	3,0171	0,34
	3,0696	0,34
	3,0255	0,34
	Average ± SD%	0,34 ± 0,00

Tab. 2 Ammonia and Nitrate Nitrogen – AOAC 892.01		
Sample	Sample quantity (g)	N %
Liquid fertilizer 1	1,0196	2,00
	1,0174	2,02
	1,0093	2,03
	Average ± SD%	2,02 ± 0,01
Liquid fertilizer 2	0,5196	6,41
	0,5142	6,42
	0,5082	6,48
	Average ± SD%	6,44 ± 0,04
Liquid fertilizer 3	0,7135	5,43
	0,7317	5,35
	0,7221	5,43
	Average ± SD%	5,40 ± 0,05

Tab. 3 Ammonia and Urea Nitrogen – AOAC 941.04

Sample	Sample quantity (g)	N %
Liquid fertilizer 1	1,0326	2,05
	1,0275	2,04
	1,0168	2,05
	Average ± SD%	2,05 ± 0,00
Liquid fertilizer 2	1,0342	6,70
	1,0255	6,69
	1,0210	6,69
	Average ± SD%	6,70 ± 0,00
Liquid fertilizer 3	1,0107	0,35
	1,0017	0,34
	1,0037	0,33
	Average ± SD%	0,34 ± 0,01

Tab. 4 Results of the single species

Sample	Ammonia Nitrogen	Nitrate Nitrogen (Ammonia and Nitrate – Ammonia)	Urea Nitrogen (Ammonia and Urea – Ammonia)
Liquid fertilizer 1	2,01%	0,01%	0,04%
Liquid fertilizer 2	6,17%	0,27%	0,53%
Liquid fertilizer 3	0,34%	5,06%	0,00%

Conclusion

The results obtained show an excellent reproducibility and always fall within the range expected by the samples.

The urea and nitrate methods will include ammonia nitrogen in the results; therefore, ammonia nitrogen has to be determined separately and then subtracted.

The **UDK 169** is an **automatic system** able to perform **distillation and titration simultaneously and fully automatically**. The unit is available to be connected to an autosampler, ensuring the best productivity for laboratories.

If an autosampler is not required, the **UDK 159** is able to reach the same performances with the same procedure.

The results above show the good recovery and precision of the machine, being able to perform an analysis in just about 8 minutes.