APPLICATION NOTE

E-EA-K-002-2023/A1

Total Nitrogen determination in Urea – Dumas and Kjeldahl method comparison

Dumas reference: **AOAC 993.13** Nitrogen (Total) in Fertilizers
Kjeldahl reference: **AOAC 955.04** Nitrogen (Total) in Fertilizers

Tested with:
VELP Scientifica NDA 702 Dumas Nitrogen Analyzer (Code F30800080)
DKL 20 Automatic Digestion Unit (Code S30100210)
UDK169 Automatic Kjeldahl Distillation & Titration System (Code F30200165)

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Introduction

Urea has important uses in many fields of industry with an annual production volume of about 170 million tonnes.
It is a crystalline substance widely used as a fertilizer, feed supplement, and starting material for plastics and drugs.
It was first isolated in 1773 by the chemist Hilaire-Marine Rouelle from urine, and the first generally accepted synthesis goes back to 1828.
It has a very high nitrogen content of approximately 46%; it is readily converted to nitrate in the soil, and therefore considered as one of the most concentrated nitrogenous fertilizers.
Although over 85% of Urea is used as fertilizer, it is also widely used in melamine resins production for different industrial sectors.
The importance of having a controlled Nitrogen content in this product is crucial to always keep the same production standards.
Nitrogen determination in fertilizers is commonly carried out using the Kjeldahl method following AOAC 955.04. Here we want to give a comparative of this classic analysis and the Dumas technique elemental analysis, following AOAC 993.13.
Velp provides systems to perform both analyses:

- NDA702 elemental analyser to follow Dumas method
- A system composed of DKL20, SMS scrubber, JP Pump (for automatic digestion) and UDK169 (for distillation and automatic titration)

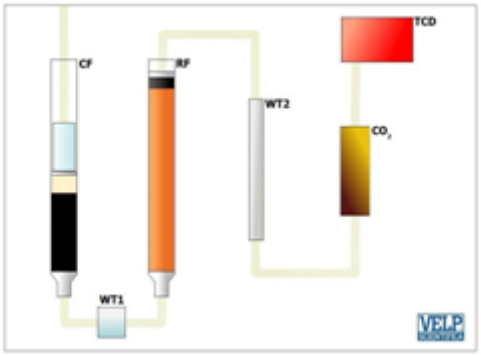
Nitrogen determination in Urea

This application note compares the nitrogen determination in urea by using NDA702 Dumas Nitrogen Analyzer and UDK 169 Automatic Kjeldahl Analyzer.
The specific methods used in this study are briefly summarized below.

Kjeldahl -Dumas: an overview of the methods

The modern Kjeldahl method consists of a procedure of catalytically supported mineralization of organic material in a boiling mixture of sulphuric acid and sulphate salt at digestion temperatures higher than 400 °C. During the process, the organically bonded Nitrogen is converted into ammonium sulphate. Alkalizing the digested solution liberates ammonia which is quantitatively steam distilled and determined by titration.

The Dumas method starts with a combustion furnace (CF) to burn the sample, obtaining elemental compounds.
Water is removed by a first physical trap (WT1 - **DriStep™**), placed after the combustion, and by a second chemical one (WT2). Between the two, the elemental substances passed through a reduction furnace (RF).
The auto-regenerative CO2 adsorbers (CO2) let pass only the elemental Nitrogen that is detected by the **LoGas™** innovative Thermal Conductivity Detector (TCD) with no requirement for a reference gas.
The NDA 702 is controlled via PC through the intuitive **DUMASoft™**.



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Sample Preparation

The samples taken into account are two urea that differs between themselves from the physical form.
One sample is in prills form and the other one has a smaller particle size resulting in microprills.
They have been both analyzed as they are with the two different methods.

Sample	Physical form	Expected Nitrogen
Urea	Prills and microprills	44 - 47%

Kjeldahl Method: DKL 20 and UDK 169 Procedure

Sample Digestion

Put about 0,2g of sample into a 250 ml test tube (Code A00000144), by using the weighing boat (Code CM0486001).
Add in each test tube:

- 2 catalyst tablets KjTabs VCM (code A00000274)
- 2 antifoam tablets KjTabs VS (Code A00000283)
- 20 ml concentrated sulphuric acid (96-98%)

Prepare some blanks with all chemicals and without the sample.
Connect the Digestion Unit to a proper Aspiration Pump (JP code F30620198) and a Fume Neutralization System (SMS Scrubber code F307C0199) to neutralize the acid fumes created during the digestion phase.
Digest the samples, setting the pre-installed Urea method: for 60 minutes at 420 °C.

Distillation and Titration

Let the test tubes cool down at 50-60 °C.
Condition the UDK 169 unit by performing the Automatic checkup in Menu-System and a Wash down.
Distil the samples by selecting the chosen method.
In UDK 169 settings, set as a unit of measure mgN and %N for the final result and as sample quantity “g”.
Distillation &Titration analysis time: from 5 minutes for one test.

Dumas Method - NDA 702 Procedure

Follow the operating manual to start the CN 802 and check that the following parameters are set:
Temperature Combustion reactor (Code A00000158): 1030 °C
Temperature Reduction reactor (Code A00000226): 650 °C
Flow rate MFC1 Helium: 190 ml/min
Flow rate MFC2 Helium: 220 ml/min
Condition the system by testing 2 to 5 empty tin foils (Code A00000153) as checkup and 2 EDTA standards (Code A00000149) to check the calibration curve accuracy.
Fill in the following fields in the database: **Sample name, Weight, Method, Sample type, Calibration number**
Run the analyses using the pre-installed “UREA” method.
Press  to start the analysis.
Analysis time: from 5 minutes for one run.

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Results

	NDA 702		UDK 169	
Sample	**Sample quantity (mg)**	**TN %**	**Sample quantity (g)**	**TN %**
Urea prills	15,625	46,01	0,1949	45,97
	14,996	45,88	0,2030	45,83
	15,347	45,89	0,1994	45,94
	Average ± SD%	**45,93 ± 0,07**	**Average ± SD%**	**45,91 ± 0,08**
Urea microprills	16,453	45,88	0,1945	45,83
	14,859	45,87	0,1882	45,73
	15,475	46,12	0,2129	45,90
	Average ± SD%	**45,96 ± 0.14**	**Average ± SD%**	**45,82 ± 0,09**

Conclusion

Both the method used has excellent repeatability. The accuracy of both systems has been checked with standards before starting the analysis and can therefore be considered compliant. All the data obtained are acceptable and comparable with the expected value.
Even on the elemental analyzer NDA702, it's possible to analyze the sample in prill form without any needing of grinding it.
The difference in results between the two methods can be considered negligible, with a conversion factor very close to one.
NDA702 allows to work with a high throughput since the analysis time is very low, 4-5 minutes, and the operator time on the machine is drastically reduced.
Following the Kjeldahl method you will stick to a well-known procedure, still widely used in Nitrogen determination, with great accuracy and precision. The Velp distillation system UDK 169 has been designed with the highest automation, able to perform distillation and titration simultaneously, increasing productivity.

In addition, connect the **UDK 169** and the **NDA 702** to the exclusive **VELP Ermes Cloud Platform** to improve your laboratory experience.

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