

Total Nitrogen Determination in Liquid Fertilizers

Dumas and Kjeldahl method comparison

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

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

Tested with:

VELP Scientifica DKL 20 Automatic Digestion Unit (S30100210)

VELP Scientifica UDK 159 Automatic Distillation Unit (F30200155)

VELP Scientifica KS 1000 Scrubber (F307A0660)

VELP Scientifica NDA 702 Dumas Nitrogen Elemental Analyzer (F30800080)



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.

One of the most important macroelements for the plants is nitrogen.

Kjeldahl determination is the commonly accepted method of nitrogen determination, based on traditional wet chemistry.

An alternative to the classical Kjeldahl method is the Dumas combustion technique, innovative dry chemistry, easy to use and highly accurate.

Both the techniques are officially approved for the determination of the nitrogen content in fertilizer.

Nitrogen Determination in Fertilizer

This application note compares the nitrogen determination in different samples of fertilizer by using NDA 702 Dumas Nitrogen Analyzer and UDK 159 Automatic Kjeldahl Analyzer.

The specific methods used in this study are summarized briefly here.

Kjeldahl method

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

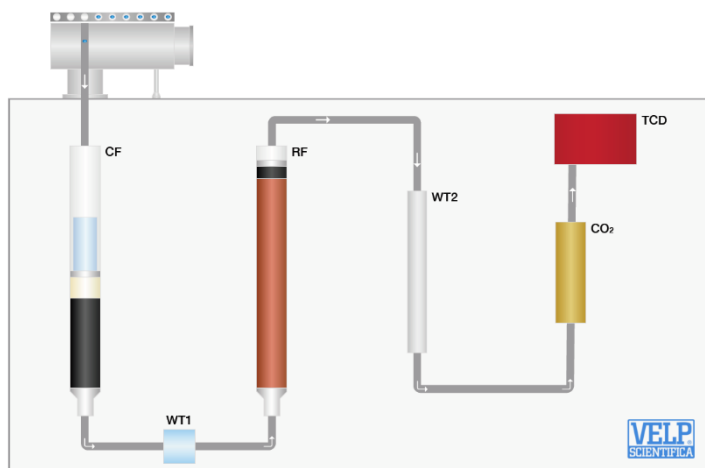
Dumas method

The Dumas method starts with a combustion step, in the 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 a second chemical one (WT2). Before the second trap, the elemental substances passed through a reduction furnace (RF).

The auto-regenerative CO₂ absorbers (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™**.



Samples

Four liquid fertilizers with different content of nitrogen have been analyzed.

Sample	Expected Nitrogen content
Fertilizer 1	3.24 %
Fertilizer 2	1.24 %
Fertilizer 3	30.60 %
Fertilizer 4	2.49 %

Kjeldahl analysis

1. Sample Digestion

Weigh about 0,5/1 gram in a nitrogen-free weighing boat (Code CM0486000) and transfer in a test tube.

In each test tube add:

- 1 Catalyst Tablets KjTabs VCT (code A00000276) (5g K₂SO₄+ 0,15g CuSO₄ x 5 H₂O + 0,15g TiO₂)
- 20 ml concentrated sulfuric acid (96-98%) containing 1g Salicylic acid

Shake until thoroughly mixed and let stand, with occasional shaking, for at least 30min.

Prepare some blanks with all chemicals and without sample.

Connect the Digestion Unit to the KS 1000 Scrubber to neutralize the acid fumes created during digestion phase.

Digest the samples for 90 minutes at 420 °C, setting a customized method on the DKL Digestion units according to the method "AOAC Official Method 955.04".

2. Distillation and Titration

Let the test tubes cool down to 50-60 °C.

Condition the UDK Distillation units by performing the Automatic Check up in Menu-System and a Wash down.

Distill the samples setting the following parameters on the customized method on UDK 159.

- H₂O (dilution water): 50 ml
- NaOH (32%): 70 ml
- Vreceiver™ (A00000316): 30 ml
- H₂SO₄ (0.1 N) as titrant solution
- Steam power: 50%

Distillation & Titration analysis time: from 4 minutes for one test.

Dumas analysis

NDA 702 Preliminary Operations (daily)

Follow the operating manual to start the Elemental analyzers units 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 He: 190 ml/min

Flow rate MFC2 He: 220 ml/min

Condition the system by testing 2 EDTA standard (Code A00000149) and 3 to 5 empty tin foils (Code A00000153) as Check up. Verify the calibration curve with one or more tests as Standard by testing the same standard used for the curve creation.

Sample Preparation (NDA 702)

Weigh around 50-200 mg of sample depending on the expected value of nitrogen in a tin foil directly on the analytical balance, adding 50-125 mg of Super-Absorbent Powder (Code A00000317).

Close the tin foil, obtaining a capsule.

Load the capsule into the autosampler.

Analysis Procedure (NDA 702)

Fill the following fields in the database of the software DumaSoft™: **Sample name, Weight, Method, Sample type, Calibration number**

Setting a personalized method with the following parameters:

Protein factor: 0 **O₂ flow rate**: 200 ml/min **O₂ factor**: 1.0 ml/mg

Press  to start the analysis.

Analysis time: from 3 minutes for one run.

Results have been obtained with the calibration curve: in a range of 0 - 7mg N with 7 measurements of EDTA standard (%N = 9.59) (Code A00000149). The data obtained are included in the tolerance admitted by the EDTA certificate.

Typical Results on Liquid Fertilizers

The table below shows the nitrogen results, obtained by NDA702 Dumas unit and Velp Kjeldahl system.

Dumas Technique		Kjeldahl Technique	
Fertilizer 1 (mg)	Nitrogen %	Fertilizer 1 (mg)	Nitrogen %
109.1	3.280	533.7	3.219
116.0	3.232	519.9	3.228
101.9	3.237	509.9	3.226
133.2	3.238	538.9	3.252
Average ± SD%	3.246 ± 0.022		3.231 ± 0.014
RSD% *	0.688%		0.444%

Fertilizer 2 (mg)	Nitrogen %	Fertilizer 2 (mg)	Nitrogen %
224.5	1.233	1074.2	1.233
212.1	1.235	1007.6	1.241
215.0	1.249	997.1	1.235
208.8	1.245	1230.4	1.259
Average ± SD%	1.241 ± 0.008		1.242 ± 0.012
RSD% *	0.623%		0.953%

Fertilizer 3 (mg)	Nitrogen %	Fertilizer 3 (mg)	Nitrogen %
26.6	30.522	151.3	30.449
14.6	30.460	171.2	30.029
46.8	30.081	155.9	30.506
24.5	30.451	151.8	30.270
Average ± SD%	30.379 ± 0.201		30.314 ± 0.215
RSD% *	0.661%		0.709%

Fertilizer 4 (mg)	Nitrogen %	Fertilizer 4 (mg)	Nitrogen %
147.6	2.495	1048.0	2.492
150.2	2.510	1010.0	2.509
109.1	2.493	991.8	2.485
163.2	2.484	970.7	2.502
Average ± SD%	2.495 ± 0.011		2.497 ± 0.011
RSD% *	0.432%		0.428%



N/PROTEIN DETERMINATION IN LIQUID FERTILIZERS KJELDAHL AND DUMAS METHOD

Conclusions

VELP is a leading company designing and manufacturing instruments for Nitrogen determination with the traditional Kjeldahl method and the innovative Dumas combustion method.

The obtained values fell within the expected nitrogen range, demonstrating the high performances of both VELP equipments, Kjeldahl system and Dumas analyzer NDA 702.

Excellent repeatability is ensured with both techniques, as demonstrated by low RSD values.

NDA 702 Dumas combustion apparatus with high productivity and non-stop performances is indeed ideal for high throughput, being fully automated and requiring just 3-4 minutes per analysis.

VELP Kjeldahl system using genuine catalyst tablets KJTabs™, is a robust solution for nitrogen determination offering versatility to adjust to every throughput requirement.

In conclusion both techniques are efficient and capable of analyzing the liquid fertilizer sample with high accuracy and repeatability.

VELP offers a wide range of solutions for nitrogen determination in environmental field. Just find the best alternative according to your needs.

VELP solutions

Tested with	Other VELP solutions				
DKL 20	DKL 8	DKL 12	DK 6	DK 8	DK 20
UDK 159	UDK 129	UDK 139	UDK 149	UDK169	
NDA 702					