N/Protein Determination in Rapeseeds

Dumas and Kjeldahl method comparison

Kjeldahl reference: **AOAC 2001.11** Protein (Crude) in Animal Feed, Forage (Plant Tissue), Grain, and Oilseeds; **REG CE 152/2009; EN ISO 5983-2:2009** Animal feeding stuffs

Dumas reference: **AOAC 992.23** Crude Protein in Cereal Grains and Oilseeds, **ISO 16634-1:2008** Oilseeds and animal feeding stuffs

Tested with **VELP Scientifica DKL 20 Automatic Kjeldahl Digestion Unit** (Code S30100210)
VELP Scientifica UDK 169 Automatic Kjeldahl Analyzer with AutoKjel Autosampler (Code S30200160) and
VELP Scientifica NDA 702 Dual Carrier Gas Dumas Nitrogen Analyzer (Code F30800080)

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Introduction

Rapeseed meal, called canola meal in North America, Australia and some other countries, is a high protein-containing material that can be used as a feed for livestock and poultry. Typical meal contains a little less than 40% of protein; however it also contains about 12% of crude fiber. Thus it is used mostly in ruminant feeding. The protein value of rapeseed meal is higher than that of the majority of other vegetable proteins containing both lysine and sulfur amino acids, and its worldwide production is second only to soybean meal.
Both Kjeldahl and Dumas techniques are officially approved for the determination of the protein content in seeds.
Performances of VELP Kjeldahl system and Dumas unit were evaluated by participating in the proficiency testing program organized by BIPEA (Bureau Interprofessionnel d'Etudes Analytiques).
The obtained results (as % Protein) were compared with the BIPEA assigned values.

Protein Determination in BIPEA sample Rapeseeds

This application note compares the nitrogen/protein determination in rapeseeds by using NDA702 Dumas Nitrogen Analyzer and UDK169 Automatic Kjeldahl Analyzer with AutoKjel Autosampler.

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

Kjeldahl method

The modern 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.

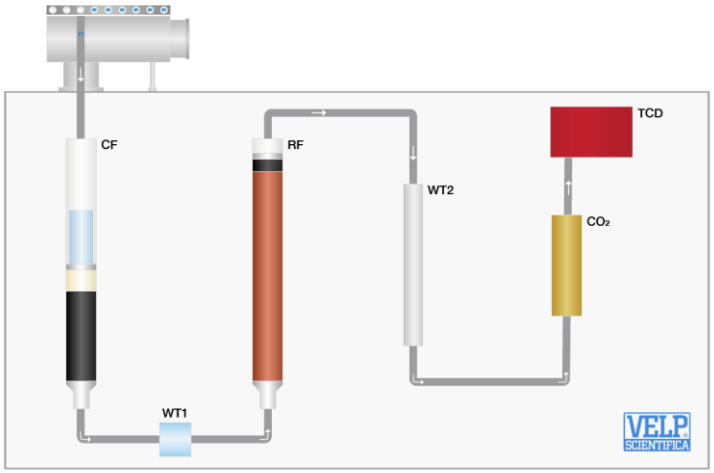
Dumas method

The Dumas method starts with a combustion 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). Between the two, the elemental substances passed through a reduction furnace (RF).

The auto-regenerative CO_2 absorbers (CO_2) 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

BIPEA Rapeseeds	ID: 11-0413-0058
Dumas protein:	assigned value: 19.1 ± 0.3
Kjeldahl protein:	assigned value: 18.9 ± 0.3

Sample has been grinded by using a laboratory grinder (particle size 1 mm).

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Kjeldahl analysis

1. Sample Digestion

Weight about 0.8 grams in a nitrogen-free weighing boat (Code CM0486000) and transfer in a test tube.
In each test tube add:

- 2 catalyst tablets VCM (code A00000274; 3.5 g K_2SO_4, 0.1 g $CuSO_4$ $5H_20$ Missouri)
- 2 antifoam tablets VS (Code A00000283)
- 20 ml concentrated sulfuric acid (96-98%)
- 5 ml hydrogen peroxyde (H_2O_2)

Prepare some blanks with all chemicals and without 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 digestion phase.
Digest the sample for 40 minutes at 300 °C plus 90 minutes at 420 °C, according to the method “Oats, barley, corn, rice, rye” (n°9 on DKL 20).

2. Distillation and Titration

Let the test tubes cool down to 50-60 °C.
Condition the UDK 169 unit by performing the Automatic Check up in Menu-System and a Wash down.
Distill the samples selecting the predefined methods n°9.

- H_2O (dilution water): 50 ml
- H_2SO_4 (0.2 N) as titrant solution
- NaOH (32%): 70 ml
- Protein factor: 6.25
- Vreceiver™ (A00000316): 30 ml

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

Dumas analysis

NDA 702 Preliminary Operations (daily)

Follow the operating manual to start the NDA 702 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 EDTA, used for the curve creation.

Sample Preparation (NDA 702)

Weigh around 150 mg of sample in a tin foil directly on the analytical balance.
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**
The “SEED MEAL” method shows the following parameters:
Protein factor: 6.25
O_2 flow rate: 400 ml/min **O_2 factor**: 1.6 ml/mg
Press to start the analysis.
Analysis time: from 3 minutes for one run.
Results have been obtained with the following calibration curve: in a range of 0 - 7 mg N with 7 measurements of EDTA standard (%N = 9.57) (Code A00000149). The data obtained are included in the tolerance admitted by the EDTA certificate.

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Results on BIPEA Rapeseeds samples

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

Technique	Sample quantity (mg)	Nitrogen %	Protein %
Dumas	149.10	3.071	19.194
	149.30	3.042	19.010
	151.20	3.036	18.976
	150.60	3.020	18.874
	149.50	3.035	18.970
	Average ± SD%	**3.041 ± 0.019**	**19.005 ± 0.117**
	RSD% *	**0.616**	**0.617**
Kjeldahl	863.4	2.955	18.469
	708.1	2.968	18.550
	821.3	3.002	18.763
	798.1	3.001	18.756
	832.4	2.964	18.525
	Average ± SD%	**2.978 ± 0.022**	**18.613 ± 0.137**
	RSD% *	**0.738**	**0.738**
Dumas assigned value:19.1 ± 0.3 % P			
Kjeldahl assigned value: 18.9 ± 0.3 % P			

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 determination of Nitrogen and Protein in rapeseeds fell within the expected protein range indicated by BIPEA, demonstrating the high performances of both VELP equipment, UDK 169 Distillation Unit and NDA 702 Dumas analyzer.

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 protein determination in food and feed field.

The complete procedure was verified by using 5 ml of glycine standard solution (3%) containing 28 mg of nitrogen, as reference substance.

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