
A new frontier of insect flours: Nitrogen and Protein Determination Dumas and Kjeldahl method comparison

Kjeldahl reference: **AOAC 2001.11-2005** Protein (crude) in animal feed, Forage (plant)

Dumas reference: **AOAC 990.03** Protein (Crude) in Animal Feed Combustion Method

Tested with:

VELP Scientifica NDA 702 Dumas Nitrogen Analyzer (Code F30800080)

DKL 20 Kjeldahl Digestion Unit (Code S30100210)

KS 1000 Scrubber (code F307A0660)

UDK 169 Automatic Kjeldahl Nitrogen Protein Analyzer (Code F30200165)



Introduction

The trend of using edible insects to produce foods is growing. Thus, the food industry needs to find analytical methods and instruments to assess their potential. Insects have interesting nutritional characteristics, not to mention the relationship between breeding costs, time and yield. For these reasons, a very rapid expansion of its use in human nutrition is expected as early as the second half of the 21st century. With a typically "powder" consistency, insect flour is usually dark brown in colour. In addition, since 24 January 2023, the European Union has also adapted its food production regulation to the availability of cricket flour as a new ingredient (COMMISSION IMPLEMENTING REGULATION (EU) 2023/5). Most insects intended for human consumption contain very little fat and, moreover, have a **protein concentration that is close to 60%** of the total mass and has a high biological value. The protein content in foods is often determined on the basis of the total nitrogen content. Kjeldahl or Dumas methods are universally applied for nitrogen determination.

Velp provides instruments for both Dumas and Kjeldahl methods: the NDA 702 elemental analyser to follow the Dumas method, and a system composed of DKL 20, KS 1000 Scrubber and UDK 169 (for distillation and automatic titration) for Kjeldahl.

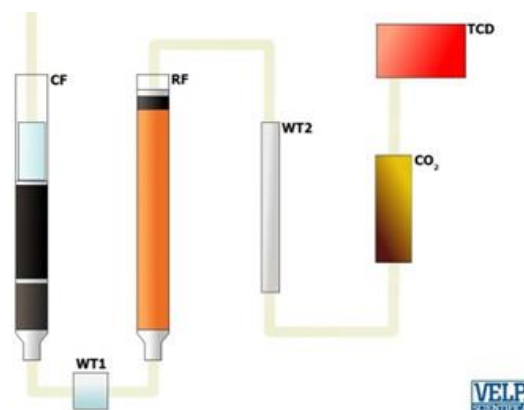
Dumas method

The elemental analysis 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 pass 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™**.



Kjeldahl method

The 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.

Sample Preparation

The samples, three different insect flours, look like a fine powder and they do not require to be homogenized before the analysis.

Dumas analysis

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 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 standard (Code A00000149) to check the calibration curve accuracy.

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

Method: Feed for animals, dry

Sample weight: ~ 50mg

Protein factor: 6.25

Press  to start the analysis

Analysis time: from 4 minutes for one run.

Results have been obtained with the following calibration curve: in a range of 1 – 8 mg N with 7 measurements of EDTA standard (N% = 9.47) (Code A00000149)

Kjeldahl Method: DKL 20 and UDK 169 Procedure

Sample Digestion

Put about 0.5g of sample into a 250 ml test tube (Code A00000144). 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 a few blanks with all chemicals and without samples.

Connect the Digestion Unit to KS1000 Scrubber (cod. F307A0660) to neutralize the acid fumes created during the digestion phase.

Digest the samples, setting up the following ramps: heat for 30 min at 300°C and 60 min at 420°C

Distillation and Titration

Let the test tubes cool down to 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 method “**Cereal and feed**” into the software.

In UDK 169 settings, set as a unit of measure mgN and %N for the final result and as sample quantity “g”.

Distillation and titration analysis time: from 5 minutes for one test.

The protein factor 6.25 was used to calculate the protein factor

Results on insect flour

Dumas – NDA702

	Nitrogen%*	RSD%**	Protein%*	RSD%**
Insect flour 1	9.226 ± 0.018	0.190	57.660 ± 0.110	0.190
Insect flour 2	9.328 ± 0.062	0.669	58.302 ± 0.390	0.669
Insect flour 3	8.458 ± 0.066	0.779	52.865 ± 0.412	0.779

(*) Average ± standard deviation

(**) RSD% = Relative Standard Deviation

Kjeldahl – UDK169

	Nitrogen%*	RSD%**	Protein%*	RSD%**
Insect flour 1	9.194 ± 0.029	0.315	57.463 ± 0.182	0.317
Insect flour 2	9.326 ± 0.058	0.622	58.290 ± 0.360	0.618
Insect flour 3	8.418 ± 0.028	0.333	52.614 ± 0.117	0.336

(*) Average ± standard deviation

(**) RSD% = Relative Standard Deviation

Conclusion

The accuracy of both methods adopted has been checked with standards before starting the analysis and can be therefore considered compliant. All the data obtained are acceptable and comparable with the expected value.

The NDA 702 can easily and quickly determine the nitrogen content on insect flours. The results are reliable and comparable to the Kjeldahl ones. The repeatability of the results is likewise very good (RSD ≤ 0.8 %) and the data confirm the complete combustion of the sample with no memory effect observed.

The NDA 702 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. VELP Scientifica NDA 702 Dumas Nitrogen Analyser is the perfect response to simple, fast and precise nitrogen/protein determination.

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.

The results prove the validity of the two methods (Kjeldahl and Dumas).

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

Contact the **VELP Analytical Team** to learn more about Nitrogen and Protein Determination in insect flour.