APPLICATION NOTE

M-D-006-2013/A3

N Determination in Cellulose Nitrate

according to the Dumas combustion method

Reference: **Military standardization agreement STANAG No. 4178**

Tested with **VELP Scientifica NDA 701 Dumas Nitrogen Analyzer** (Code F30800070)

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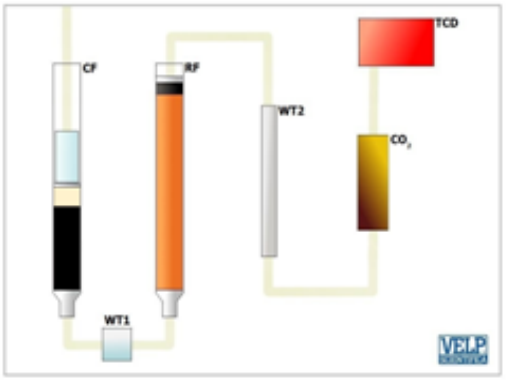
NITROGEN DETERMINATION IN EXPLOSIVES
DUMAS COMBUSTION METHOD

Introduction

Cellulose nitrate, originally known as guncotton, is a highly flammable compound formed by nitrating cellulose through exposure to nitric acid. It was discovered casually in 1846 by C. F. Schönbein, a German-Swiss chemist: while he was working in the kitchen, he spilled a bottle of concentrated nitric acid on the table. After using his wife's cotton apron to mop it up, he hung the apron over the stove to dry, and, as soon as it was dry, there was a flash as the apron exploded. The discovery of cellulose nitrate found several applications in the past, i.e. for cameras flashes but, because of its explosive nature, not all of them of were successful.
Now, it is used as sticky membrane for bio-molecular analysis (Western blot), paints and lacquers, in the tricks of magic and as a propellant in the cartridge pistols and rifles.
One of the most tested parameters is the nitrogen content because cellulose nitrate with a higher nitrogen content (e.g. trinitrocellulose) is used as explosives and those with medium content for preparation of jellies explosive and smokeless powders.

Nitrogen Determination in Cellulose Nitrate - flash cotton type

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 adsorbers (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 701 is controlled via PC through the intuitive **DUMASoft™**.



NDA 701 Preliminary Operations (daily)

Follow the operating manual to start the NDA 701 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

Dry the cellulose nitrate to constant weight in an oven for 8 hours at 50 °C plus 1 hour at 100 °C, before starting the test.
Remove it from the oven and transfer it to a desiccator, containing desiccant, for cooling to room temperature.
Weigh around 30 mg of cellulose nitrate directly in a tin foil placed on the balance.
Close carefully the tin foil by hands, without shocks or frictions, obtaining a capsule.
Load it into the autosampler.

NOTE: *Dry nitrocellulose is flammable and explosive. It cannot be pulverized using a grinder because the heat produced in the mill is sufficient for the ignition of an explosion. It can be inactivated by strong acids or alkali solution.*

Analysis Procedure

Fill the following fields in the database: **Sample name, Weight, Method, Sample type, Calibration number**
The EXPLOSIVES method shows the following parameters.
Protein factor: none
O_2 flow rate: 300 ml/min
O_2 factor: 1.4 ml/mg
Press to start the analysis.
Analysis time: from 3 minutes for one run.

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Typical Results on Cellulose Nitrate – flash cotton type

Sample quantity (mg)	Nitrogen % d.m. (*)
30.90	12.865
30.20	12.830
30.60	12.708
29.60	13.008
30.00	12.852
30.30	12.755
29.70	12.795
30.30	12.912
30.00	12.802
29.70	12.722
Average ± SD%	**12.825 ± 0.091**
RSD% **	**0.707**
Nitrogen Expected Value: > 12.6%	

(*) %N on dry matter
(**) RSD% = (Standard Deviation * 100) / Average

Conclusion

The obtained results are reliable and in accordance with the expected value. The nitrogen content of cellulose nitrate results 12.825 %N d.m., according with the specifications of the substance (N% > 12.6%).
Results have been obtained with the following calibration curve: in a range of 0 - 9 mg N with 5 measurements of EDTA standard (%N = 9,57) (Code A00000149).

Benefits of Dumas combustion method are:

- High productivity, non-stop performance
- Time saving, few minutes required
- Moderate running costs
- Totally unsupervised, fully automated
- Omission of harsh and toxic chemicals
- Eco-friendly, low amount of residues and wastes

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