
N Determination in Tyre Rubber according to the Dumas combustion method

Reference: ISO TC 45/SC 2 N2116 Rubber, raw, latex - Determination of nitrogen content by Dumas Combustion method

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



Introduction

Modern pneumatic tyres are made by synthetic or natural rubber, fabric and metal wires with chemical compounds. Pneumatic tyres are used on many types of vehicles, including cars, bicycles, motorcycles, trucks, earthmovers, and aircraft. For tyre manufacturers it is important to determine the content of nitrogen present in the constituent mixture because a high presence of nitrogen adversely affects the performance of the tyre, increasing the incidence of premature failure of it.

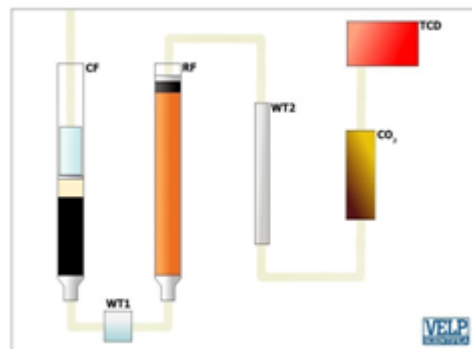
Nitrogen Determination in Tyre Rubber

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 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 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

Cut in pieces the rubber from a car tyre, clean it from fiber and metal parts. Freeze the cut rubber (around -10°C) in order to enhancing the solidification and obtain a better homogenization with a grinder (particle size around 2 mm)

Weigh around 80 mg of tyre rubber directly in a tin foil placed on the balance.

Close carefully the tin foil by hands, obtaining a capsule.

Load it into the autosampler.

Analysis Procedure


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

The TYRE RUBBER method shows the following parameters.

Protein factor: none

O₂ flow rate: 400 ml/min

O₂ factor: 1.8 ml/mg

Press  to start the analysis.

Analysis time: from 3 minutes for one run.

Typical Results on Tyre Rubber

Sample quantity (mg)	Nitrogen %
79.10	0.346
80.00	0.348
79.50	0.332
79.00	0.341
79.40	0.356
79.10	0.340
79.30	0.340
80.00	0.343
79.00	0.342
80.80	0.349
Average ± SD%	0.344± 0.006
RSD% *	1.885
Nitrogen Expected Value: 0.2-0.5%	

(*) RSD% = (Standard Deviation * 100) / Average

Conclusion

The obtained results are reliable and in accordance with the expected value.

The nitrogen content of tyre rubber is in accordance with the specifications of the substance (0.2 < N% < 0.5).

Results have been obtained with the following calibration curve: in a range of 0 – 1.3 mg N with 5 measurements of rice flour standard (%N = 1,38 ± 0,05) (Code A00000235).

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