APPLICATION NOTE

E-EMA-002-2021/A2

CHNS/O Determination in rubber samples – material characterization

Reference method**:**

ASTM D5291: Test method for Instrumental determination of Carbon, Hydrogen and Nitrogen in petroleum products and lubricants.

DIN 51724-3: Solid mineral fuels - Determination of sulfur - Part 3: Instrumental methods

ASTM D 5373: Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke.

Tested with **VELP Scientifica EMA 502 Elemental Analyzer**

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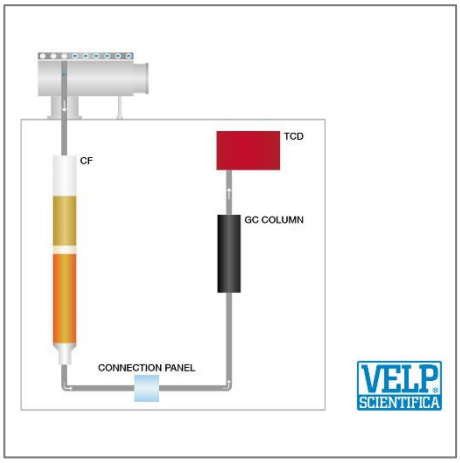
Introduction

Viton® is a brand name from DuPont for its synthetic rubber and fluoropolymer elastomer, resistant to extreme temperatures and harsh chemicals. It is commonly used in O-rings, gaskets, and fuel hoses. This family of elastomers comprises copolymers or terpolymers of tetrafluoroethylene (TFE), hexafluoropropylene (HFP) and vinylidene fluoride (VDF or VF2), vinylidene fluoride (VDF) and hexafluoropropylene (HFP) as well as perfluoromethylvinylether (PMVE) containing specialty polymers.
It is important to ensure that a polymer with the required formulation is used in a particular application. To enable this, analytical techniques, that can positively identify fluoroelastomer types, are required. **Combustion and pyrolysis** are useful techniques for **characterizing this complex family of materials**.

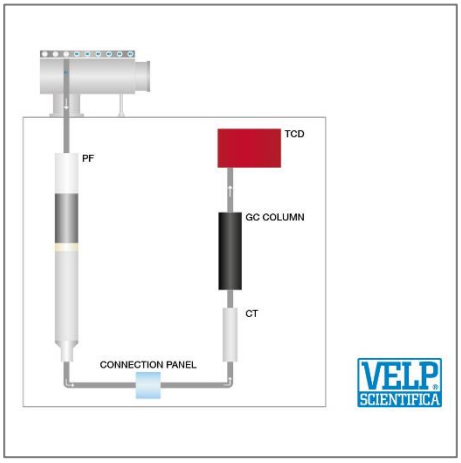
CHNS determination in rubber

The **CHNS** analysis starts with the combustion of the sample inside the VELP combustion furnace at a temperature higher than 1000°C to obtain elemental compounds.
VELP Vcopper™, a formulation of highly active copper powder, is placed in the lower part of the reactor helps the reduction of NO_x into N_2.
The gas stream reaches the gas-chromatographic column and components are separated flowing out of the column with different retention times.
The innovative **LoGas™** Thermal Conductivity Detector (TCD), with no requirement for a reference gas, enables the detection of all gases content.
The EMA 502 is controlled via PC through the intuitive **EMASoft™**.
The analysis is completed in few minutes.



Oxygen determination in rubber

The sample is purged with carrier and conveyed to the reactor where pyrolysis happens.
The sample is converted into its elemental compounds at a high temperature (>1000°C).
The carrier with elemental gases reaches the chemical trap where all impurities are absorbed.
The flow reaches the gas-chromatographic column and then the TCD (Thermal conductivity detector) that enables the quantification of oxygen as carbon monoxide in few minutes.



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EMA 502 Preliminary Operations (daily)

Follow the operating manual to start the EMA 502 and check that the following parameters are set:

Temperature CHNS reactor (Code A00000443): 1030 °C
Temperature GC Column Oven: 55 °C
Flow rate MFC1 He: 120 ml/min
Flow rate MFC2 He: 140 ml/min

Condition the system by testing 2 Sulphanilic acid standard (Code A00000434) and 2 to 3 empty tin foils (Code A00000436) as checkup. Verify the calibration curves with one or more tests as Standard by testing the same standard used for the curve's creation.

Follow the operating manual to start the EMA 502 and check that the following parameters are set:

Temperature O reactor (Code A00000444): 1060 °C
Temperature GC Column Oven: 55 °C
Flow rate MFC1 He: 140 ml/min
Flow rate MFC2 He: 160 ml/min

Condition the system by testing 2 EDTA standard (Code A00000149) and 2 to 3 empty tin foils (Code A00000436) as checkup. Verify the calibration curves with one or more tests as Standard by testing the same standard used for the curve's creation.

Sample Preparation

Viton samples:	Expected range values declared on the certificate:
Reference soil	C%=0.09 - 0.13% S%=0.09 - 0.13%

The sample has been analyzed in small pieces.

Analysis Procedure

The complete analysis must be carried out in two different steps: CHNS configuration and Oxygen configuration.

CHNS Analysis Procedure

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

Create a new customizable method with the following parameters:
O_2 flow rate: 300 ml/min
O_2 factor: 1.4 ml/mg
Min Oxy volume: 10 ml
Press to start the analysis.
Analysis time from 12 minutes for one run.

Oxygen Analysis Procedure

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

Method: pyrolysis
Press to start the analysis.
Analysis time from 6 minutes for one run.

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CHNS Results on different Viton samples

CHNS Calibration: the CHNS results have been obtained with the calibration curve using the certified standard Sulphanilic acid (code A00000434) (C% = 41.58 – H%= 4.04 – N%= 8.08 – S%= 18.48), using a range of 0.4 – 4.0 mg of Sulphanilic acid.

Oxygen Calibration: the Oxygen results have been obtained with the calibration curve using the certified standard EDTA (code A00000149) (O% = 43.8), using a range of 0.4 – 4.0 mg of EDTA.

The tables below show the %C, %H, %N, %S and %O results obtained by the **EMASoft™** using 3 mg of sample.

Sample	Carbon %	Hydrogen %	Oxygen %
Viton A	49.1638	1.6155	17.3478
	49.1248	1.6308	17.3388
	49.2205	1.6198	17.2124
	49.1930	1.6073	17.2384
Average %	**49.1755**	**1.6183**	**17.2843**
RSD %	**0.07**	**0.52**	**0.34**

Sample	Carbon %	Hydrogen %	Nitrogen %	Oxygen %
Viton B	38.5705	1.8777	0.3135	17.5291
	38.6497	1.8471	0.3153	17.6940
	38.6204	1.8427	0.3108	17.6742
	38.5948	1.8637	0.3144	17.5155
Average %	**38.6089**	**1.8578**	**0.3135**	**17.6033**
RSD %	**0.07**	**0.75**	**0.53**	**0.46**

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Sample	Carbon %	Hydrogen %	Sulfur %	Oxygen %
Viton C	26.3374	1.0962	2.5927	20.3712
	26.4305	1.0795	2.5701	20.3910
	26.4734	1.0991	2.5421	20.2663
	26.3752	1.0216	2.5311	20.3182
Average %	**26.4041**	**1.0741**	**2.5590**	**20.3367**
RSD %	**0.19**	**2.91**	**0.94**	**0.24**

Conclusion

The **EMA 502 Elemental Analyzer CHNS-O** is a reliable solution for the determination of carbon, hydrogen, nitrogen, sulfur and oxygen aimed at the characterization of different rubber samples.

The main advantages of VELP CHNS-O elemental analyzer:

- Precision and reproducibility with the LOD of 0.001 mgN with Helium and the RSD of 0.2% (Sulphanilic acid)
- Low cost per analysis thanks to innovative technology and genuine consumables
- Fast set up of the instrument
- Easy maintenance
- Intuitive software User Interface
- Connectivity to VELP Ermes Cloud Platform for remote Application and Service support, remote access to the instruments, alerts and notifications and much more
- Software upgrade with the optional 21 CFR Part 11 Package for Pharmaceutical, Cosmetic and Food industry laboratories that require compliance with FDA regulation

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