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# CS Determination in coal samples by flash combustion

Reference method:

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

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

**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**



## Introduction

**Coal** is mainly composed by **carbon**, which is the useful element for energy purposes; other components in coal are water, rock residues, volatile matter and **sulfur** compounds.

**Sulfur** is present basically in two inorganic forms, pyritic sulfur ( $\text{FeS}_2$ ) and sulfates ( $\text{Na}_2\text{SO}_4$ ,  $\text{CaSO}_4$ ,  $\text{FeSO}_4$ ), and many organic forms such as sulfides, mercaptans, bisulfides, etc. Typically, coal contains anywhere from 0.2 to 5 % sulfur by dry weight. When coal is burned, sulfur will be released in the form of sulfide and  $\text{H}_2\text{S}$ , which then reacts with  $\text{O}_2$ , water and other substances to change into sulfuric acid; this will impact water environment, acidify the soil and do great harm to plants and human health.

Thus, to define the quality of the product it is important to know the percentage of **carbon**, as well as the amount of **sulfur**.

## CHNS determination in coal and coke

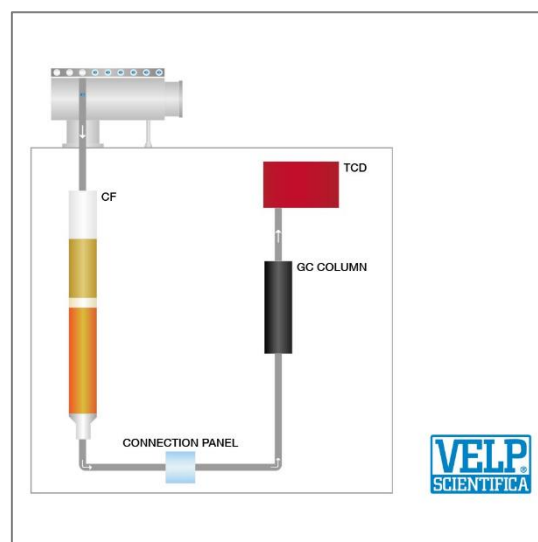
The **CHNS** analysis starts with the combustion of the sample inside the VELP combustion furnace at a temperature higher than  $1000^\circ\text{C}$  to obtain elemental compounds.

**VELP Vcopper™**, a formulation of highly active copper powder, is placed in the lower part of the reactor helps the reduction of  $\text{NO}_x$  into  $\text{N}_2$ .

The gas stream reaches the gas-chromatographic column and flows out of the column after component separation 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.



## 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):  $1030^\circ\text{C}$

**Temperature GC Column Oven**:  $55^\circ\text{C}$

**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.

## Sample Preparation

Reference coal samples:

Reference coal 1:

Reference coal 2:

Expected range values declared on the certificate:

$\text{S}\% = 0.35 \pm 0.03\%$

$\text{S}\% = 1.41 \pm 0.03\%$

The sample has been analyzed as is.

## 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<sub>2</sub> flow rate:** 400 ml/min

**O<sub>2</sub> factor:** 2.0 ml/mg

Min Oxy volume: 10 ml

Press  to start the analysis.

Analysis time: from 12 minutes per sample.

## Typical CHNS Results on soil samples

**CHNS Calibration:** the CHNS results have been obtained with the calibration curve using the certified standard AR4019 (C% = 0.11 - S%= 0.11), using a range of 15 – 50 mg of AR4019.

The data obtained are included in the tolerance admitted by the certificate of analysis for Carbon and Sulphur content (declared parameters). The table below shows the %C, %S, obtained by the **EMASoft™** using using 5-6 mg of sample.

Sample	C%	S%
Reference coal 1	89.4678	0.3449
	89.4739	0.3464
	89.0314	0.3578
	88.5053	0.3415
	89.4180	0.3367
	89.2310	0.3570
Average %	89.1880	0.3474
RSD %	0.38	2.00

Sample	C%	S%
Reference coal 2	92.4155	1.4023
	93.4889	1.4039
	94.0130	1.4079
	94.1025	1.3897
	91.2840	1.4124
	93.0145	1.4116
Average %	93.0531	1.4046
RSD %	1.05	0.54

## Conclusion

The **EMA 502 Elemental Analyzer CHNS-O** is a reliable solution for the determination of carbon and sulfur in order to assess the quality of coal 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