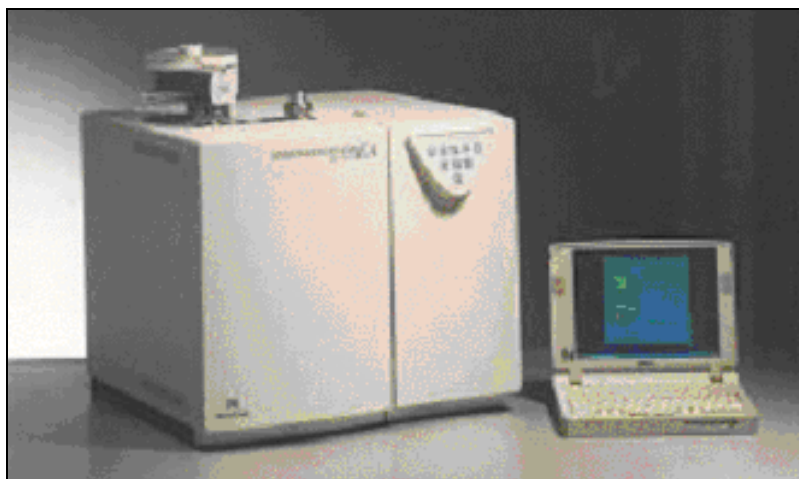


## AN 641

### CHNS/O analysis in Carbon Fiber by Flash 2000 Analyzer

OEA Team

- π Automated, unattended analysis
- π No matrix effect
- π Reliable results
- π Instrument flexibility



#### Introduction

Organic elemental analysis has always been a traditional method to characterize these materials.

The importance of this technique lies in the possibility to perform Carbon, Nitrogen, Hydrogen and Sulfur determination of these types of samples in a single analysis run, and the Oxygen determination in a second run.

In this light the Flash 2000 CHNS/O Analyzer is the most reliable instrument for elemental analysis and copes with all requirements of modern laboratories such as accuracy, reproducibility and low cost per analysis.

#### Description of the analytical method

The Analyzer as CHNS configuration operates according to the dynamic flash combustion of the sample. The sample is weighed in tin capsule and introduced into the combustion reactor via the MAS 200R Autosampler together with a proper amount of oxygen.

After combustion, the produced gases are carried by a helium flow to a layer filled with copper, then swept through a GC column that provides the separation of the combustion gases and finally detected by a thermal conductivity detector. Total run time less than 10 minutes.

For oxygen determination, the system operates in pyrolysis mode. Samples placed in silver containers are dropped into the pyrolysis chamber, which is maintained at 1060°C and contains nickel-coated carbon. The oxygen in the sample combined with carbon forms CO that is then chromatographically separated from other combustion products. Similar to CHNS determination, the analyzed gas is available for further quantitative analysis

A complete CHNS/O report is automatically generated by the Eager 300 data handling software package and displayed at the end of the analytical routine.

## Analytical Conditions

T left tube: 900°C (for CHNS analysis)

T right tube: 1060°C (for O analysis)

T oven: 65°C

Carrier Flow: 130 ml/min

Reference Flow: 100 ml/min

Oxygen Flow: 250 ml/min

(OFF for Oxygen analysis)

Oxygen Injection End: 5 sec

(0 sec for Oxygen analysis)

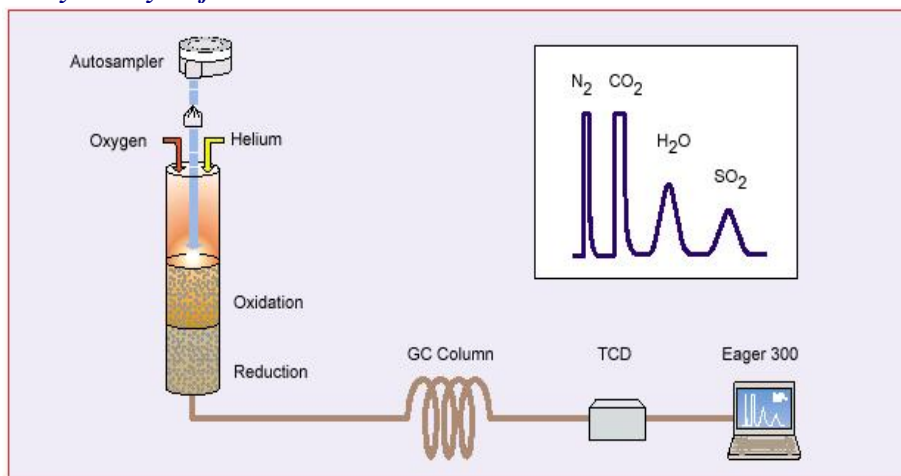
Sample Delay Time: 12 sec

Run time: 720 sec

(300 sec for Oxygen analysis)

Sample weight: 2-3 mg

## Analytical layout for CHNS determination



## Results

For CHNS analysis the instrument calibration was performed with Anthracene (94.34 C%, 5.66 H%) and BBOT (6.51 N%, 72.53 C%, 6.09 H%, 7.44 S%) as standards and K factor as calibration method. The standards selected cover with the range of concentration from the samples.

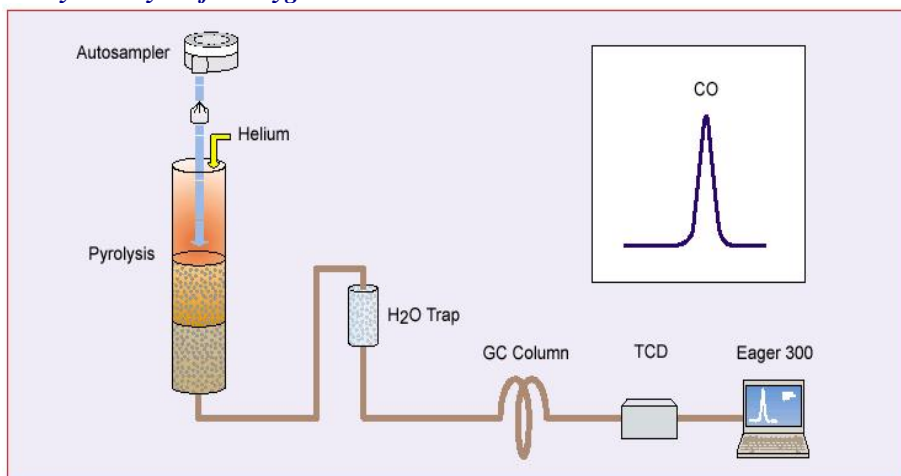
For Oxygen analysis was used Acetanilide (11.84 O%) as standard and K factor as calibration method.

Table 1 shows CHN data of two different Carbon fiber samples

Table 2 shows CHNS/O data of three different Rayon based Carbon fiber.

All data were obtained with an excellent reproducibility and no matrix effect was observed when changing sample.

## Analytical layout for Oxygen determination



**Table 1 – CHN determination of Rayon based Carbon fiber**

Sample	N %	RSD%	C %	RSD%	H %	RSD%
1	20.746	1.282	60.077	0.134	3.611	2.532
	21.236		60.103		3.422	
	21.288		60.020		3.473	
	21.326		59.921		3.427	
2	6.254	1.024	91.426	0.291	0.362	4.155
	6.376		91.946		0.335	
	6.353		91.584		0.340	

**Table 2 – CHNS/O determination of Rayon based Carbon fiber**

Sample	N %	RSD%	C %	RSD%	H %	RSD%	S %	RSD %	O %	RSD %
1	0.2642	0.8992	89.7354	0.4139	0.8072	0.7032	0.1483	2.2773	7.3561	0.3502
	0.2682		90.4785		0.7961		0.1418		7.3995	
	0.2685		90.1653		0.8034		0.1441		7.3536	
2	0.5011	0.5902	90.8000	0.3477	0.2884	0.8518	0.2817	1.1888	4.5824	0.8298
	0.5034		91.3435		0.2924		0.2780		4.5087	
	0.5070		90.7914		0.2879		0.2751		4.5598	
3	0.0299	2.7602	98.4037	0.2967	0.0267	4.4944	0.0641	2.3962	0.2654	1.0382
	0.0311		98.9710		0.0279		0.0611		0.2679	
	0.0295		98.8140		0.0255		0.0626		0.2624	

OEA Team is constituted of: Liliana Krotz, Andrea Cadoppi, Luigi Ragaglia and Guido Giuzzi.