Characterization of Carbon Black with the Thermo Scientific FLASH 2000 Organic Elemental Analyzer

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Overview

Purpose: Characterization of carbon black by Organic Elemental Analysis.

Methods: Samples were analyzed using an elemental analyzer with automated autosampler.

Results: CHNS/O data are shown to assess the performance of the FLASH 2000 OEA.

Introduction

Carbon black is a material produced by the incomplete combustion of heavy petroleum products such as coal tar, ethylene cracking tar, and a small amount from vegetable oil. It is mainly used as a reinforcing filler in tires and other rubber products while in plastics, paints, and inks carbon black is used as a color pigment.

In a typical production process of carbon black the elemental content is periodically monitored and tested for quality control, in particular carbon quantification. The total carbon content is required for the calculation of carbon dioxide emissions and it can be also used in calculations to estimate yield of the process.

The Thermo Scientific™ FLASH™ 2000 CHNS/O Analyzer (Figure 1), which operates with dynamic combustion of the sample (Dumas method), provides automated and simultaneous CHNS determination in a single analysis run and it provides oxygen determination by pyrolysis in a consequent run. The dedicated Thermo Scientific™ Eager Xperience Data Handling Software calculates automatically the Gross Heat and Net Heat Values (GHV and NHV), both expressed in kcal/kg, and the CO_2 Emission Trade data.

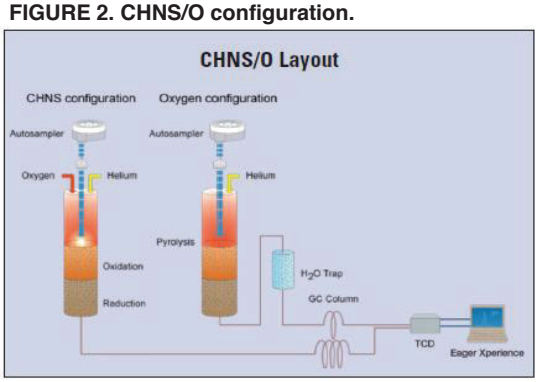
Methods

For CHNS determination, samples are weighed in tin capsules and introduced into the combustion reactor via the Thermo Scientific MAS 200R Autosampler. After combustion the resulted 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 (TCD) (Figure 2).

For oxygen determination, samples are weighed in silver capsules and introduced into the pyrolysis chamber via the MAS 200R Autosampler. The reactor contains nickel coated carbon at 1060°C. The oxygen present in the sample, combined with the carbon, forms carbon monoxide which is then chromatographically separated from other products and detected by the TCD detector (Figure 2).

FIGURE 1. FLASH 2000 OEA CHNS/O Analyzer.

FIGURE 2. CHNS/O configuration.



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Results

Different types of carbon black samples were analyzed according to the different element content, in order to demonstrate the performance of the system and show the repeatability (without any matrix effect).

To verify the complete combustion of high containing carbon samples, carbon determination was evaluated in a Carbon Mesoporous Reference Material (99.95 C%, Aldrich No. 699624). The system was calibrated with BBOT* as standard, 2,5-Bis (5-tert-butyl-benzoxazol-2-yl) thiophene.

Table 1 shows the data obtained with good repeatability. The average is comparable with the expected value, indicating the complete oxidation of the sample.

**BBOT: 2,5-Bis (5-tert-butyl-benzoxazol-2-yl) thiophene*

TABLE 1. Carbon determination.

C%	Av. C%	RSD%
99.91	99.71	0.143
99.58		
99.86		
99.71		
99.68		
99.69		
99.82		
99.72		
99.69		
99.41		

Table 2 shows the repeatability of CHNS determination of carbon black samples while Table 3 shows the oxygen data and the GHV and NHV values (both expressed in kcal/kg), and the CO_2 Emission Trade data calculated automatically by the Eager Xperience Data Handling Software. For CHNS, carbon black samples were run in two series of analyses. In each series samples were analyzed in duplicate, the statistical data showed in the table is the average of the four determinations.

TABLE 2. CHNS data of carbon black samples.

Sample	N %	RSD %	C %	RSD %	H %	RSD %	S %	RSD %
A	0.188 0.185 0.186 0.186	0.676	96.559 96.734 96.817 96.706	0.111	0.300 0.295 0.304 0.308	1.843	0.829 0.848 0.868 0.868	2.193
B	0.273 0.277 0.272 0.273	0.810	96.178 95.899 95.351 96.079	0.159	0.267 0.280 0.272 0.277	2.086	0.823 0.814 0.815 0.817	0.493
C	0.239 0.229 0.239 0.232	2.155	95.768 96.126 95.826 95.700	0.196	0.343 0.350 0.341 0.328	2.697	1.016 1.020 1.019 1.013	0.311
D	0.148 0.146 0.149 0.152	1.681	97.073 97.066 96.966 97.146	0.076	0.282 0.285 0.292 0.286	1.465	1.273 1.267 1.256 1.261	0.583

The performance of the FLASH 2000 CHNS/O Analyzer was evaluated through the comparison of the repeatability of the CHN data obtained in comparison to the ASTM D5373 requirements showed in Table 4.

The precision of the FLASH 2000 OEA for CHN determination was evaluated from the results showed in Table 2. Table 5 shows the difference (Diff.) calculated between both data of each series of analyses. All differences are acceptable and less than the Repeatability Limit indicates in the Official Method indicating the good homogeneity and the complete combustion of the samples.

TABLE 3. Oxygen data, Heat Values and CO_2 value of carbon black samples.

Sample	O %	RSD %	GHV (kcal/kg)	RSD %	NHV (kcal/kg)	RSD %	CO_2 E.T.	RSD %
A	1.149 1.158 1.152	0.397	8005 8005 8005	0.003	7990 7989 7990	0.003	105.96 105.96 105.96	0.000
B	1.661 1.679 1.661	0.623	7915 7916 7915	0.006	7901 7901 7901	0.006	106.37 106.38 106.37	0.005
C	1.597 1.597 1.602	0.181	7933 7934 7934	0.002	7916 7916 7916	0.002	106.00 106.00 106.01	0.005
D	0.624 0.612 0.616	0.990	8062 8063 8063	0.003	8048 8048 8048	0.003	105.59 105.58 105.58	0.006

TABLE 4. Concentration Range and Limit of Repeatability accepted by ASTM D5373.

Element	Concentration range (%)	Repeatability Limit r
Carbon	48.6 to 90.6	0.64
Hydrogen	0.14 to 5.16	0.16
Nitrogen	0.69 to 1.57	0.11

Repeatability Limit (r): the value below which the absolute difference between two test results calculated to a dry basis of separate and consecutive test determinations, carried out on the same sample, in the same laboratory, by the same operator, using the same apparatus.

TABLE 5. CHN data of carbon black samples according to ASTM D5373 requirements.

Sample	Serie	N %	Diff.	C %	Diff.	H %	Diff.
A	1	0.188	0.003	96.559	0.175	0.300	0.005
		0.185		96.734		0.295	
	2	0.186	0.000	96.817	0.111	0.304	0.004
		0.186		96.706		0.308	
B	1	0.273	0.004	96.178	0.279	0.267	0.013
		0.277		95.889		0.280	
	2	0.272	0.001	95.851	0.228	0.272	0.005
		0.273		96.079		0.277	

The Thermo Scientific FLASH 2000 Elemental Analyzer proved to be a valuable solution for the elemental analysis of carbon black in terms of:

- Accuracy, repeatability and sensitivity of results.
- Its automation and high speed of analysis allow efficient analysis and help reduce overall operational costs.
- All data were obtained according to the ASTM D5373 method.
  No matrix effect was observed when changing the sample, indicating complete combustion.

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