Application Note 42213

Thermo Scientific FLASH 2000 CHNS Analyzer: Stability, Linearity, Repeatability and Accuracy

Dr. Liliana Krotz, OEA Product Specialist, and Dr. Guido Giazzi, OEA Product Manager,
Thermo Fisher Scientific, Milan, Italy

Key Words
Accuracy, automated and unattended analysis, CHNS, flash combustion, organic chemistry, precision

Introduction

The chemical characterization of organic compounds plays a very important role in all the processes of synthesis, separation, purification and their structural identification both for research and quality control purposes in organic chemistry and pharmaceuticals. Determination of organic elemental composition is periodically monitored for the characterization of these materials. For fast analysis with excellent reproducibility, it is essential to have an accurate and precise technique, preferably automatic and easy to use.

The Thermo Scientific™ FLASH 2000 CHNS analyzer (Figure 1), which is based on the dynamic combustion of the sample, allows quantitative determination of carbon, nitrogen, hydrogen and sulfur in a single run. The system copes effortlessly with the wide array of modern laboratory requirements such as accuracy, reproducibility and low cost per analysis.

Figure 2. FPD Detector

Figure 1. FLASH 2000 Analyzer

Methods

For CHNS determinations, the elemental analyzer operates according to the dynamic flash combustion of the sample. Samples are weighed in a tin capsule and introduced into the combustion reactor via the Thermo Scientific™ MAS™ 200R autosampler together with a proper amount of oxygen. After combustion, the resultant gases are carried by a helium flow to a layer filled with copper, then swept through a GC column that separates the combustion gases, and are finally detected by a thermal conductivity detector (TCD) (Figure 3). Total run time is 10 minutes. A complete report is automatically generated by the Thermo Scientific™ Eager Xperience data handling software and is displayed at the end of the analysis. The dedicated software automatically calculates the minimum formula.

Thermo SCIENTIFIC

Figure 3. CHNS configuration

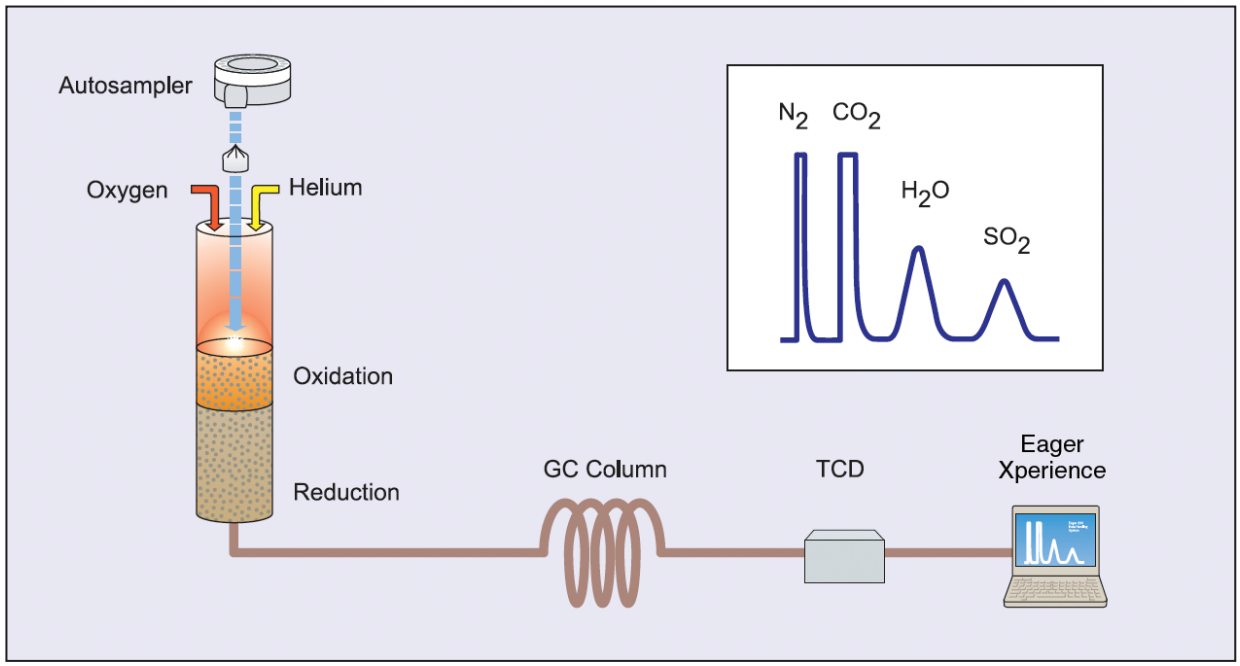

Table 1. Sequence of Methionine analyses

N %	C %	H %	S %
9.355	40.399	7.437	21.237
9.391	40.154	7.381	21.261
9.383	40.185	7.404	21.318
9.402	40.300	7.451	21.485
9.373	40.292	7.434	21.269
9.394	40.164	7.440	21.471
9.365	40.112	7.442	21.306
9.376	40.204	7.424	21.559
9.435	40.294	7.426	21.477
9.429	40.498	7.462	21.388
9.402	40.232	7.453	21.407
9.387	40.134	7.418	21.365
9.414	40.061	7.393	21.348
9.439	40.096	7.419	21.400
9.439	40.368	7.470	21.556
9.440	40.255	7.450	21.421
9.427	40.221	7.442	21.349
9.420	40.233	7.426	21.534
9.427	40.182	7.428	21.253
9.391	40.156	7.403	21.229
9.439	40.266	7.420	21.265
9.499	40.424	7.464	21.288
9.376	40.071	7.414	21.422
9.498	40.429	7.449	21.365
9.422	40.098	7.402	21.267
9.429	40.178	7.393	21.411
9.488	40.407	7.444	21.518
9.475	40.196	7.432	21.217
9.497	40.356	7.468	21.492
9.484	40.393	7.457	21.589

Results

To validate the system, pure organic compounds with different concentrations were chosen.

Table 1 shows the CHNS data of 30 analyses of methionine whilst Table 2 shows the statistical results. Instrument calibration was performed with Cystine (11.66 %N, 29.99 %C, 5.03 %H, 26.69 %S), Sulphanilamide (16.27 %N, 41.84 %C, 4.68 %H, 18.62 %S) and BBOT* (6.51 %N, 72.53 %C, 6.09 %H, 7.44 %S) using Linear Regression as calibration method. The weight was 2 – 3 mg and the analyses were performed with the addition of Vanadium Pentoxide for a complete conversion of sulfur. The test points out the accuracy of the data and the stability of the system.

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

Table 2. Statistical results

Element	Theoretical	Experimetal	RSD %
	Value %	Average value %	
Nitrogen	9.39	9.42	0.44
Carbon	40.25	40.24	0.29
Hydrogen	7.43	7.40	0.32
Sulfur	21.49	21.38	0.52

Table 3 shows a typical sequence to evaluate the performance of the instrument using K factor as calibration method. Methionine (9.39 %N, 40.25 %C, 7.43 %H, 21.49 %S) was used as standard, while Sulfanilamide (16.27 %N, 41.84 %C, 4.68 %H, 18.62 %S) was analyzed as unknown to check the calibration. All data obtained are according to the technical specifications of the system (acceptable range). Table 4 shows the theoretical data, the acceptable range, the experimental values and the relative standard deviation of sulfanilammide.

Table 3. Typical CHNS sequence

Sample Name	Type	Weight (mg)	N %	C %	H %	S %
Methionine	Standard	2.326	9.39	40.25	7.43	21.49
Methionine	Standard	2.485	9.39	40.25	7.43	21.49
Methionine	Standard	2.684	9.39	40.25	7.43	21.49
Sulfanilamide	Unknown	2.468	16.26	41.75	4.66	18.68
Sulfanilamide	Unknown	2.530	16.24	41.85	4.65	18.72
Sulfanilamide	Unknown	2.479	16.24	41.80	4.65	18.71

Table 4. Statistical results of sulfanilamide

Element	Theoretical data %	Acceptable Range	Experimental average data %	RSD %
Nitrogen	16.27	16.11 – 16.43	16.25	0.071
Carbon	41.84	41.54 – 42.14	41.80	0.120
Hydrogen	4.68	4.61 – 4.75	4.65	0.124
Sulfur	18.62	18.42 – 18.82	18.70	0.274

Table 5 shows the experimental CHNS data obtained of different organic compounds in a large range of concentrations to demonstrate the complete combustion of the samples. Each compound was analyzed five times to show good repeatability. All results obtained are comparable with the theoretical values (Table 6) indicating the complete conversion of the elements without any memory effect when changing the matrix. The data obtained are inside the technical specification of the system and are shown in Table 6.

Table 5. CHNS data of organic compounds

Organic compound	Experimental data							
	N %	RSD %	C %	RSD %	H %	RSD %	S %	RSD %
4-(2-hidroxyethyl)-piperazine-1-propanesulfonic acid (> 99% purity)	11.09 11.07 11.08	0.09	42.79 42.87 42.89	0.12	7.95 7.89 7.93	0.38	12.73 12.71 12.67	0.24
4-Amine-N-tiazolilbencenesulfonamide (Sulfatiazol, ≥ 98% purity)	16.33 16.30 16.29	0.13	42.01 41.99 42.03	0.05	3.53 3.52 3.52	0.16	25.03 24.98 25.01	0.10
DL-homocystine (≥ 99% purity)	10.34 10.39 10.36	0.24	35.73 35.56 35.64	0.24	6.01 6.01 6.00	0.10	23.86 23.79 23.81	0.15
Taurine (≥ 99% purity)	11.13 11.14 11.13	0.05	19.22 19.21 19.21	0.03	5.62 5.63 5.63	0.10	25.61 25.59 25.60	0.04
N-acetiltiourea (99% purity)	23.68 23.66 23.69	0.06	30.47 30.50 30.49	0.05	5.09 5.08 5.09	0.01	27.05 26.99 27.01	0.11

Table 6. Theoretical CHNS data of organic compounds and technical specification range (±)

Organic compound	Theoretical values and acceptable range (±)							
	N %	Range	C %	Range	H %	Range	S %	Range
4-(2-hidroxyethyl)-piperazine-1-propanesulfonic acid (>99% purity)	11.04	0.11	42.58	0.30	7.89	0.10	12.61	0.13
4-Amine-N-tiazolylbencenesulfonamide (Sulfatiazol, ≥98% purity)	16.28	0.16	41.87	0.30	3.48	0.05	24.81	0.24
DL-homocystine (≥99% purity)	10.38	0.10	35.60	0.28	5.93	0.09	23.73	0.23
Taurine (≥99% purity)	11.13	0.11	19.08	0.20	5.57	0.08	25.44	0.24
N-acetiltiourea (99% purity)	23.58	0.23	30.31	0.28	5.05	0.08	26.95	0.25

Table 7 shows the experimental CHNS data of 3 - 4 mg of halogen-containing compounds (BCR standards) and the theoretical values (T.V.). Calibration was performed with 2-3 mg BBOT using K factor as calibration method. Samples were analyzed with the addition of Vanadium Pentoxide.

Table 7. CHNS of BCR standards

Standard	Halogen	Nitrogen			Carbon			Hydrogen			Sulfur		
Name	%	Av. %	RSD %	T.V.	Av. %	RSD %	T.V.	Av. %	RSD %	T.V.	Av. %	RSD %	T.V.
BCR 71 *	9.18 Cl 20.68 Br	10.70 10.63 10.64	0.35	10.80	40.18 39.98 40.02	0.26	40.40	2.37 2.35 2.34	0.65	2.37	8.36 8.31 8.30	0.39	8.30
BCR 72 **	8.12 Cl 29.28 I	9.64 9.58 9.57	0.39	9.65	36.12 36.14 36.13	0.03	36.10	2.11 2.10 2.11	0.03	2.10	7.41 7.43 7.43	0.16	7.35

*BCR 71: N-(4-bromophenyl)-N'(2-chloro-4-nitrophenyl)thiourea **BCR 72: N-(2-chloro-4-phenyl)-N'-(4-iodophenyl)thiourea

Table 8 shows the sulfur determination of NIST Reference Materials. Calibration was performed with 2-3 mg BBOT using K factor as calibration method. Standard and Reference Materials were analyzed with the addition of Vanadium Pentoxide. The sulfur NIST value is certified for montana soil while for tomato and peach leaves the sulfur value is provided for information only.

Table 8. Sulfur data of NIST Reference Materials.

Montana Soil SRM 2711		Tomato Leaves SRM 1573a		Peach Leaves SRM 1547	
Weight (mg)	S %	Weight (mg)	S %	Weight (mg)	S %
12.136	0.0469	4.325	0.9779	4.546	0.1929
15.267	0.0433	4.982	0.9899	4.728	0.1907
10.824	0.0443	4.877	0.9642	4.091	0.1898
14.981	0.0427	4.367	0.9848	4.998	0.1913
15.139	0.0442	4.903	0.9983	4.482	0.1874
14.776	0.0422	4.255	0.9816	4.538	0.1973
13.645	0.0428	4.609	0.9938	4.906	0.1881
14.873	0.0440	4.025	0.9970	4.779	0.1908
11.229	0.0429	4.786	0.9776	4.965	0.1916
14.549	0.0458	4.865	0.9805	4.183	0.1980
Average	0.0439	Average	0.9845	Average	0.1918
RSD %	3.3753	RSD %	1.0648	RSD %	1.8188
NIST value	0.042±0.001	NIST value	0.96	NIST value	0.20

Conclusion

The FLASH 2000 analyzer is the best solution for the simultaneous determination of CHNS in a single run by combustion, showing excellent reproducibility, accuracy and precision.

No memory effect was observed when changing the type of sample, indicating complete combustion and detection of the elements.

With the addition of the easily coupled FPD (flame photometric detector), it is possible to analyze low trace sulfur (5 – 10 ppm), increasing the sensitivity in sulfur determination (Figure 2).

www.thermoscientific.com

COMPANY WITH QUALITY MANAGEMENT SYSTEM CERTIFIED BY DNV
= ISO 9001:2008 =
Thermo Fisher Scientific S.p.A. Milan, Italy is ISO 9001:2008 Certified.

Africa +43 1 333 50 34 0
Australia +61 3 9757 4300
Austria +43 810 282 206
Belgium +32 53 73 42 41
Canada +1 800 530 8447
China 800 810 5118 (free call domestic)
400 650 5118
Denmark +45 70 23 62 60
Europe-Other +43 1 333 50 34 0
Finland +358 9 3291 0200
France +33 1 60 92 48 00
Germany +49 6103 408 1014
India +91 22 6742 9494
Italy +39 02 950 591
Japan +81 45 453 9100
Latin America +1 561 688 8700
Middle East +43 1 333 50 34 0
Netherlands +31 76 579 55 55
New Zealand +64 9 980 6700
Norway +46 8 556 468 00
Russia/CIS +43 1 333 50 34 0
Singapore +65 6289 1190
Spain +34 914 845 965
Sweden +46 8 556 468 00
Switzerland +41 61 716 77 00
UK +44 1442 233555
USA +1 800 532 4752

AN42213_E 05/14C

Thermo SCIENTIFIC
A Thermo Fisher Scientific Brand