Characterization of Compost by the Thermo Scientific FLASH 2000 Elemental Analyzer

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

Application Note 42197

Key Words
Compost, CHNS/O, Heat Value, TOC

Introduction

Production of compost is one of the most efficient ways of recycling waste. Due to the heterogeneity of the material used, accurate control of parameters such as nitrogen, total carbon, total organic carbon (TOC) and sulfur content in the recycled waste is a key factor to ensure the quality of the final product. High quality composts can then be used as fertilizers in agronomy. Using elemental analysis and heat value determination, it is possible to establish whether the compost to be burned is able to maintain the incinerator temperature or if it has to be mixed with other materials.

The importance of compost testing has grown dramatically in recent years and many of the classical methods are now no longer suitable for routine analysis. Organic elemental analysis has always been a traditional method to characterize these materials and it is very important to have an accurate and precise technique which provides fast analysis with excellent reproducibility.

Thermo Scientific FLASH 2000 CHNS/O analyzer

The Thermo Scientific™ FLASH 2000 CHNS/O analyzer (Figure 1) was evaluated for quantitative determination of carbon, nitrogen, hydrogen, sulfur and oxygen in compost samples. The system, which is based on the dynamic combustion of the sample, provides automatic CHNS determination in a single analysis run and oxygen determination by pyrolysis in a second run without extra cost.

The fully automatic procedure allows unattended analysis of up to 125 samples without matrix effect, from a few mg to large sample sizes. The inherent flexibility of the instrument permits analysis of NC in less than 5 minutes, simultaneous CHNS in less than 10 minutes or S only in less than 4 minutes, and analysis of oxygen in less than 5 minutes depending on laboratory requirements. The same analytical conditions can also be used for the determination of TOC, analyzing the sample after acidification to eliminate the carbonates.

Thermo SCIENTIFIC

Method

The CHNS analyzer configuration operates according to the dynamic flash combustion of the sample. The sample is 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 is finally detected by a thermal conductivity detector (TCD) (Figure 2).

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 (carbon monoxide) which is then chromatographically separated from other combustion products and finally detected by a thermal conductivity detector (Figure 2).

A complete report, including the option for calculating the relative heat value is automatically generated by the Thermo Scientific™ Eager Xperience dedicated data handling software and displayed at the end of the analytical routine.

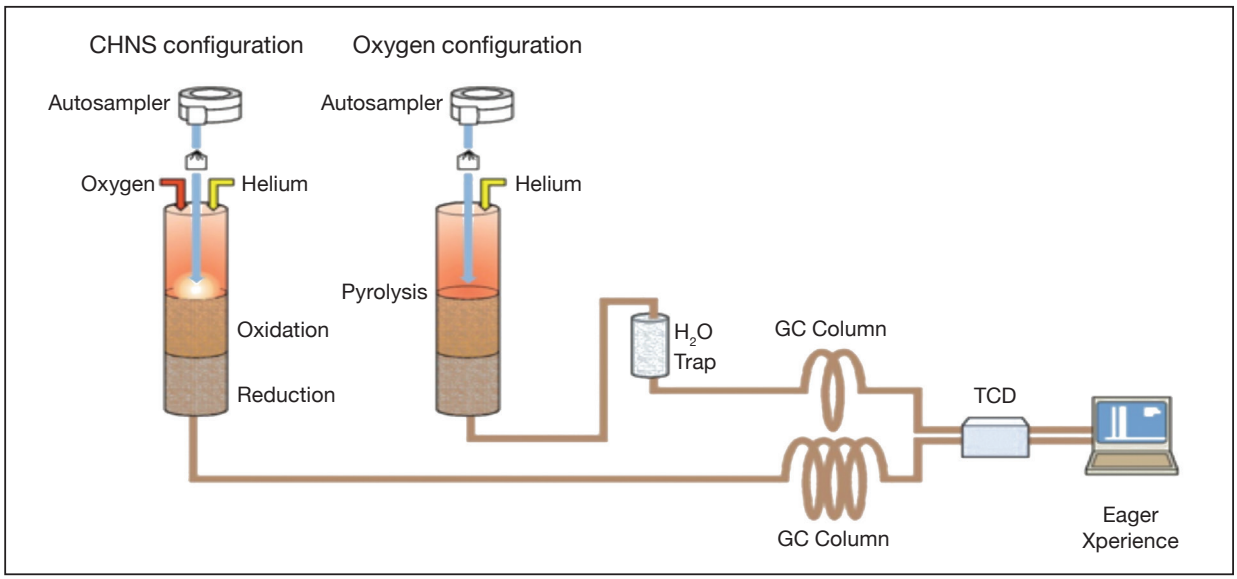

Figure 2 – FLASH 2000 CHNS/O Layout

Results

Different compost samples were chosen to evaluate the CHNS and oxygen configurations. The samples were homogenized using a rotor speed mill and a ball mill. BBOT* (2 - 3 mg) was used as standard to calibrate the system in CHNS configuration, while Benzoic acid was used as standard in oxygen configurations.

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

Table 1 shows the data obtained by CHNS/O analysis. Samples were analyzed three times to evaluate the reproducibility of the system.

Table 1 – CHNS/O data of compost samples.

Sample	N %	C %	H %	S %	O %
1	1.500	40.874	5.683	0.510	34.482
	1.568	40.882	5.702	0.507	34.098
	1.552	40.770	5.664	0.471	34.083
2	1.864	40.037	5.224	0.590	31.157
	1.879	39.604	5.158	0.567	31.395
	1.823	39.938	5.193	0.592	31.504
3	2.196	39.724	5.137	0.560	31.425
	2.201	39.674	5.177	0.564	31.820
	2.176	39.680	5.109	0.597	31.112
4	2.296	41.400	5.620	0.660	32.798
	2.243	41.783	5.532	0.665	32.425
	2.297	41.399	5.611	0.688	32.352
5	2.237	41.858	5.553	0.710	31.673
	2.205	41.802	5.518	0.705	31.387
	2.283	41.607	5.478	0.714	31.475

Table 2 shows the CHNS/O data and the heat value calculation of a compost sample. The Heat Value is calculated automatically by the Eager Xperience software using the modified Dulong-Petit equation (Characterizing Fuels, Journal of Coal Quality, Vol 3, 1982).

Table 2 - CHNS/O and Heat value data of a compost sample

Element	N %	C %	H %	S %	O %	G.H.V.	N.H.V.
Data	13.0884	36.7927	5.1549	1.9008	30.8515	3525.02	3559.00
	13.2374	36.9949	5.2545	1.9000	30.8594	3524.68	3558.67
	12.8570	36.7172	5.1442	1.9242			
Average %	13.0609	36.8349	5.1845	1.9083	30.8554	3524.85	3558.84
RSD %	1.4676	0.3898	1.1733	0.7204	0.0181	0.0068	0.0074

Figure 5 shows the sample pre-treatment for the determination of total organic carbon. The sample is weighed in a silver capsule and introduced via the sample holder plate. The acidification with Chlorhydric acid 1:1 eliminates the inorganic carbon as carbon dioxide. Dry in the electric heater at 65°C and close the capsule for sample analysis.

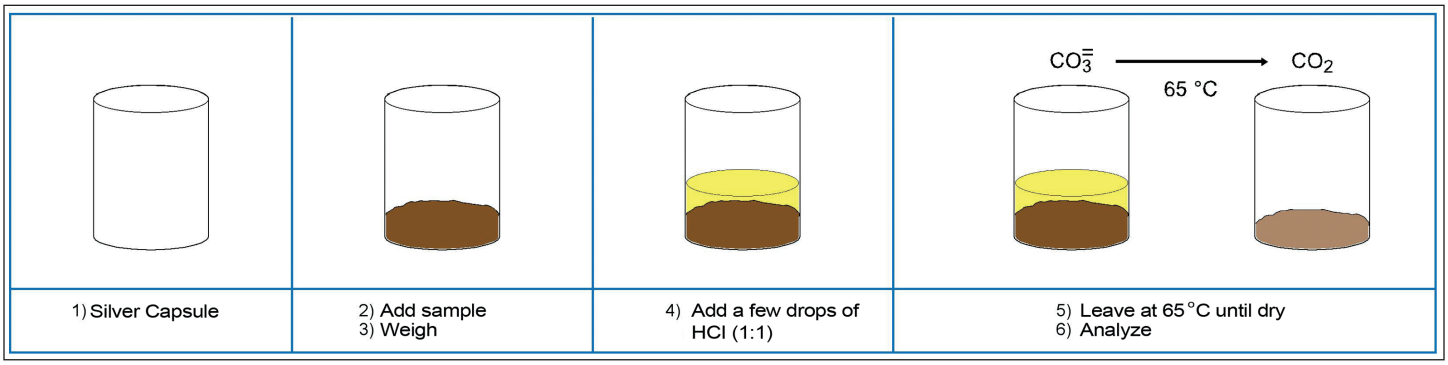

Figure 5 – Sample pre-treatment for TOC determination

Table 3 shows the data of total nitrogen (TN), total carbon (TC) and total organic carbon (TOC) on compost samples. Both analyses (TC and TOC) were performed consecutively using the same analytical conditions.

Table 3 - TN, TC and TOC in compost samples.

Sample	N %	TC %	TOC %
1	0.26 0.25	11.02 11.19	3.02 3.06
2	0.80 0.80	10.55 10.42	2.08 2.02
3	1.12 1.11	19.17 19.16	4.26 4.23

Conclusion

All data were obtained with good reproducibility and no matrix effect was observed when changing the sample. The advantage of the FLASH 2000 analyzer lies in the possibility to perform CHNS determination in a single run, oxygen determination in a second run, total organic carbon without any modification of the analytical conditions and finally to automatically calculate the relative Heat Value.

The FLASH 2000 elemental analyzer copes with all the stringent requirements of modern laboratories such as flexibility, accuracy, reproducibility, high productivity and low cost of analysis.

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

Thermo SCIENTIFIC
Part of Thermo Fisher Scientific

AN42197_E 01/14C