

High Productivity of Thermo Scientific FlashSmart CHNS/CHNS Analyzer through the MultiValve Control (MVC) Module

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Key Words

Accuracy, Automation, CHNS, Flash Combustion,
Organic Chemistry, Productivity, Unattended Analysis

Goal

To demonstrate the performance of the the FlashSmart Elemental Analyzer with the MultiValve Control (MVC) Module.

Introduction

Carbon, nitrogen, hydrogen and sulfur determination by combustion analysis are used in the characterization of raw and final products in pharmaceutical, cosmetics, universities and material industries for quality control and R&D purposes. The use of accurate and automated analytical techniques allow fast analysis with an excellent reproducibility whilst meeting laboratory demands for high productivity and low cost per analysis. The Thermo Scientific™ FlashSmart™ Elemental Analyzer (Figure 1) is equipped with two independent furnaces that allow the installation of two analytical circuits. These independent circuits can be controlled automatically through the Thermo Scientific™ MultiValve Control (MVC) Module (Figure 2). This unique Module allows the Analyzer to be set-up for ultimate productivity by having an automated CHNS/CHNS configuration in one system. Each analytical circuit can receive its own autosampler.



Figure 1. Thermo Scientific FlashSmart Elemental Analyzer.

The system copes effortlessly with the wide array of laboratory requirements such as accuracy, day to day reproducibility and high sample throughput. The MVC Module also ensures low helium consumption by switching from helium to nitrogen or argon gas, when the instrument is in Stand-By Mode. Consequently, the cost of analysis is reduced significantly.



Figure 2. Thermo Scientific MVC Module.

Methods

For CHNS determination, the FlashSmart Analyzer operates with the dynamic flash combustion of the sample. Samples are weighed in tin containers and introduced into the combustion reactor from the Thermo Scientific™ MAS Plus Autosampler with oxygen. 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). The total run time is less than 10 min (Figure 3). A complete report is automatically generated by the Thermo Scientific™ EagerSmart™ Data Handling Software and displayed at the end of the analysis.

Analytical Conditions	
Reactor Temperature	950 °C
Oven Temperature	65 °C
Helium Carrier Flow	140 ml/min
Helium Reference Flow	100 ml/min
Oxygen Flow	250 ml/min
Oxygen Injection Time	5 sec
Sample Delay	12 sec
Total Run Time	600 sec

Both pneumatic circuits for CHNS determination are preset in the system in order to pass automatically from one to the other through the MultiValve Control (MVC) Module, which is managed by the dedicated EagerSmart Data Handling Software without any operational action by the user.

The EagerSmart Data Handling Software MVC Module management page allows you to control the MVC Module (Figure 4). In the lower section of the page, you can switch from the left to the right furnace to pass from CHNS analysis on the left furnace to CHNS analysis on the right furnace. In the upper section of the page, you can switch from helium carrier gas to nitrogen or argon gas when the instrument is not used for analysis.

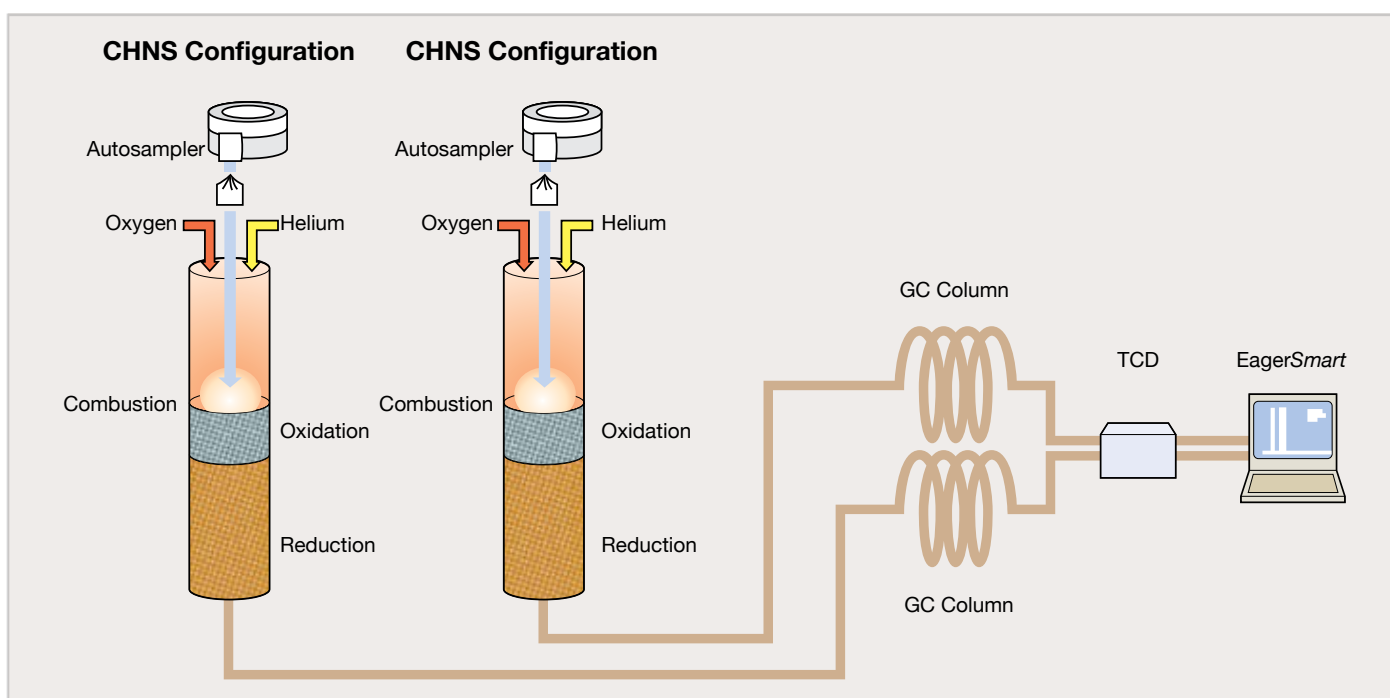


Figure 3. FlashSmart CHNS/CHNS configuration.

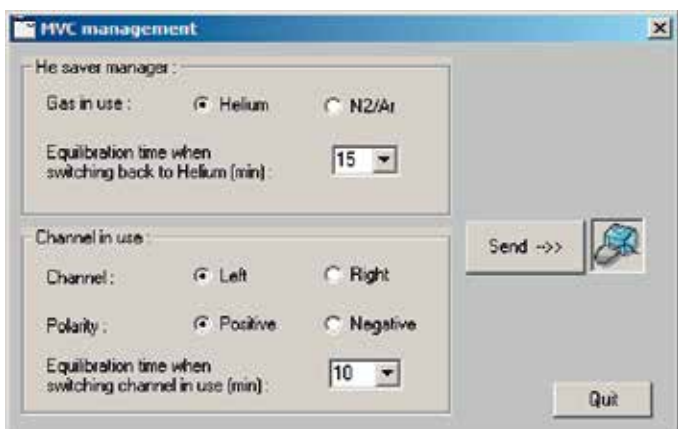


Figure 4. The MVC Module Management page on the EagerSmart Data Handling Software.

Results

Typical analytical tests were performed for CHNS configuration on both reactors during several non-consecutive days to evaluate the repeatability and accuracy as the stability of the Analyzer when the configuration is switched from left to right and vice versa. The experimental data were compared with the theoretical values and the acceptable range according to the technical specification of the system. At the end of every day, the instrument was placed into Stand-By Mode to reduce helium consumption. The Auto-Ready

Function was activated in the EagerSmart Data Handling Software for the automatic wake-up of the system, which prepared the system for analysis the next morning.

In day 1, the instrument was calibrated for CHNS with methionine standard, using K factor as the calibration method. Then, three runs of sulfanilamide were analyzed as unknown to check the calibration on each reactor.

Table 1 shows the sequence of analysis for CHNS determination for the left reactor while Table 2 shows the sequence for the right reactor. The theoretical values and the acceptable range according to the technical specification of the system of sulfanilamide are 16.27 ± 0.16 N%, 41.84 ± 0.30 C%, 4.68 ± 0.07 H% and 18.62 ± 0.2 S%.

Methionine and sulfanilamide were analyzed as unknowns, without recalibration of the instrument, in four series across 5 days with each series run in duplicate. After each series of CHNS on left reactor, the instrument automatically switched to the right reactor via the MVC Module to perform the analysis on the right reactor.

Table 1. CHNS sequence of analysis for day 1 on left reactor.

Run	Sample name	Day/ Month	Injection time	Type	Weight (mg)	N %	C %	H %	S %
1	Tin capsule	13/04	13:13	Blank					
2	Methionine	13/04	13:25	By-Pass					
						Theoretical values			
3	Methionine	13/04	13:37	STD	2.523	9.39	40.25	7.43	21.49
4	Methionine	13/04	13:49	STD	2.176	9.39	40.25	7.43	21.49
5	Methionine	13/04	14:01	STD	2.600	9.39	40.25	7.43	21.49
						Experimental data			
6	Sulfanilamide	13/04	14:13	UNK	3.056	16.22	41.64	4.67	18.61
7	Sulfanilamide	13/04	14:25	UNK	3.334	16.40	41.88	4.69	18.70
8	Sulfanilamide	13/04	14:37	UNK	3.211	16.32	41.77	4.66	18.56
						Theoretical values			
Sulfanilamide						16.27	41.84	4.68	18.62
						Accepted range (\pm)			
						0.16	0.30	0.07	0.20

Table 2. CHNS sequence of analysis for day 1 on right reactor.

Run	Sample name	Day/ Month	Injection time	Type	Weight (mg)	N %	C %	H %	S %
1	Tin capsule	13/04	15:12	Blank					
2	Methionine	13/04	15:24	By-Pass					
						Theoretical values			
3	Methionine	13/04	15:36	STD	2.576	9.39	40.25	7.43	21.49
4	Methionine	13/04	15:48	STD	2.367	9.39	40.25	7.43	21.49
5	Methionine	13/04	16:00	STD	2.762	9.39	40.25	7.43	21.49
						Experimental data			
6	Sulfanilamide	13/04	16:12	UNK	3.162	16.16	41.62	4.64	18.72
7	Sulfanilamide	13/04	16:25	UNK	2.937	16.19	41.86	4.69	18.58
8	Sulfanilamide	13/04	16:37	UNK	3.113	16.22	41.68	4.62	18.76
						Theoretical values			
Sulfanilamide						16.27	41.84	4.68	18.62
						Accepted range (±)			
Sulfanilamide						0.16	0.30	0.07	0.20

Once the analysis was completed, the MVC Module switched back to the left reactor and Stand-By Mode was activated overnight. These analyses were performed to evaluate the data and the stability of the system: the system reached stability in less than 10 min after switching. Table 3 shows the CHNS average data on the left reactor during the switching days, while Table

4 shows the data on the right reactor. The data of 80 sequential analyses from each reactor fall within the acceptable range and are according to the specification of the instrument. This indicates that there is no effect after the switch between reactors confirming the stability of the FlashSmart Analyzer. The data obtained demonstrate the repeatability of the system.

Table 3. CHNS average data on the left reactor.

Day/ Month	Sample name	Runs	N %	RSD %	C %	RSD %	H %	RSD %	S %	RSD %
14/04	Methionine	8	9.37	0.35	40.30	0.13	7.42	4.67	21.49	18.64
	Sulfanilamide	8	16.21	0.58	41.72	0.31	4.67	0.55	18.68	0.58
15/04	Methionine	8	9.36	0.41	40.42	0.24	7.40	0.47	21.49	0.50
	Sulfanilamide	8	16.27	0.31	41.85	0.21	4.68	0.56	18.70	0.33
18/04	Methionine	8	9.36	0.35	40.35	0.24	7.44	0.75	21.54	0.44
	Sulfanilamide	8	16.27	0.45	41.85	0.19	4.69	0.59	18.70	0.26
19/04	Methionine	8	9.35	0.37	40.32	0.26	7.43	0.83	21.49	0.50
	Sulfanilamide	8	16.28	0.40	41.76	0.13	4.65	0.13	18.64	0.39
20/04	Methionine	8	9.33	0.28	40.31	0.15	7.41	0.47	21.44	0.29
	Sulfanilamide	8	16.23	0.51	41.68	0.19	4.64	0.36	18.60	0.42

Table 4. CHNS average data on the right reactor.

Day/ Month	Sample name	Runs	N %	RSD %	C %	RSD %	H %	RSD %	S %	RSD %
14/04	Methionine	8	9.39	0.25	40.32	0.18	7.42	4.66	21.48	18.59
	Sulfanilamide	8	16.19	0.19	41.69	0.26	4.67	0.46	18.68	0.40
15/04	Methionine	8	9.35	0.33	40.28	0.28	7.43	0.65	21.48	0.21
	Sulfanilamide	8	16.23	0.75	41.63	0.13	4.69	0.48	18.65	0.24
18/04	Methionine	8	9.41	0.22	40.29	0.23	7.46	0.61	21.40	1.04
	Sulfanilamide	8	16.38	0.20	41.72	0.37	4.69	0.56	18.62	0.47
19/04	Methionine	8	9.40	0.64	40.20	0.43	7.38	1.07	21.53	0.41
	Sulfanilamide	8	16.25	0.32	41.81	0.38	4.65	0.31	18.67	0.45
20/04	Methionine	8	9.39	0.55	40.36	0.28	7.43	0.68	21.53	0.58
	Sulfanilamide	8	16.24	0.22	41.78	0.24	4.65	0.51	18.68	0.47

Conclusion

The all-in-one Thermo Scientific FlashSmart Analyzer is the optimal solution for the analysis of CHNS/CHNS in terms of accuracy, reproducibility, automation, speed of analysis and cost per analysis. All data showed were obtained with an acceptable repeatability and no matrix effect was observed when changing the configuration.

The MultiValve Control (MVC) Module performs the following functions:

- Automated or manual control of two MAS Plus Autosamplers.
- Automated switching from the left channel to the right channel, or vice versa.
- Reduced helium consumption by switching to nitrogen or argon gases when the instrument is in Stand-By Mode.
- Optional insertion, by the EagerSmart Data Handling Software, of an external command, for example, an actuator for a gas sampling valve.
- Auto-Ready: return automatically to helium as carrier gas if the system remained with nitrogen or argon gases in Stand-By Mode.

The double analytical configuration capability allows you to:

- Automatically switch between configurations increasing the productivity of the laboratory.
- Gain continuous operation of the system by using one reactor for CHNS on the left furnace and one reactor on the right furnace.
- Fully control the workflow using the EagerSmart Data Handling Software.

Thanks to the modularity of the all-in-one FlashSmart Analyzer, the same hardware, autosamplers and software can be readily used for other configurations such as CHN/O, CHN/S, CHNS/O, CHNS/CHNS, CHN/CHN, NC/S, N-Protein/S and more. This can mainly be realized by changing the consumables as the Analyzer and software are complete, illustrating the all-in-one nature of the Analyzer.

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