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TECHNICAL NOTE

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High Automation of Thermo Scientific Flash*Smart* CHN/O Analyzer using the MultiValve Control (MVC) Module

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Key Words
Accuracy, Automation, CHN, Flash Combustion, Organic Chemistry, Oxygen, Unattended Analysis

Goal

To demonstrate the performance of the MultiValve Control (MVC) Module on the Flash*Smart* Elemental Analyzer for CHN/O determinations.

Introduction

Carbon, nitrogen and hydrogen determination, by combustion analysis, and oxygen determination by pyrolysis are commonly used for the characterization of raw and final products in pharmaceutical, cosmetics, universities and material industries for quality control and R&D purposes. The use of an accurate and automated analytical technique, allowing the fast analysis with excellent reproducibility, is however essential.

The Thermo Scientific™ Flash*Smart*™ Elemental Analyzer (Figure 1) is equipped with two totally independent furnaces allowing the installation of two analytical circuits that can be used sequentially and are completely automated through the Thermo Scientific™ MultiValve Control (MVC) Module (Figure 2).

Figure 1. Thermo Scientific Flash*Smart* Analyzer.

Each analytical circuit accepts its own autosampler. In this way the system copes effortlessly with the laboratory requirements such as accuracy, day to day reproducibility and high sample throughput.

The MVC Module ensures very low helium consumption by switching from helium to nitrogen or argon gas, when the instrument is in Stand-By Mode. In this way the cost of analysis significantly reduced.

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Figure 2. Thermo Scientific MultiValve Control Module.

Methods

For CHN determination, the Flash*Smart* Analyzer operates with the dynamic flash combustion of the sample. Samples are weighed in tin containers and introduced into the combustion reactor (left furnace) from the Thermo Scientific™ MAS Plus Autosampler with oxygen. After combustion, the resulted gases are conveyed by a helium flow to the catalyst, a layer filled with copper and silvered cobaltous-cobaltic oxide, then swept through a GC column that provides the separation of the combustion gases. Finally, they are detected by a Thermal Conductivity Detector (TCD). The total run time is about 7 minutes. (Figure 3).

For oxygen determination, the system operates in pyrolysis mode. Samples are weighed in silver containers and introduced into the pyrolysis reactor (right furnace) from the MAS Plus Autosampler. The reactor contains nickel coated carbon maintained 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 3). A complete report is automatically generated by the Thermo Scientific™ Eager*Smart* Data Handling Software and displayed at the end of the analysis.

Analytical Conditions	
Reactor Temperature	950 °C
GC Oven Temperature	75 °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	480 sec

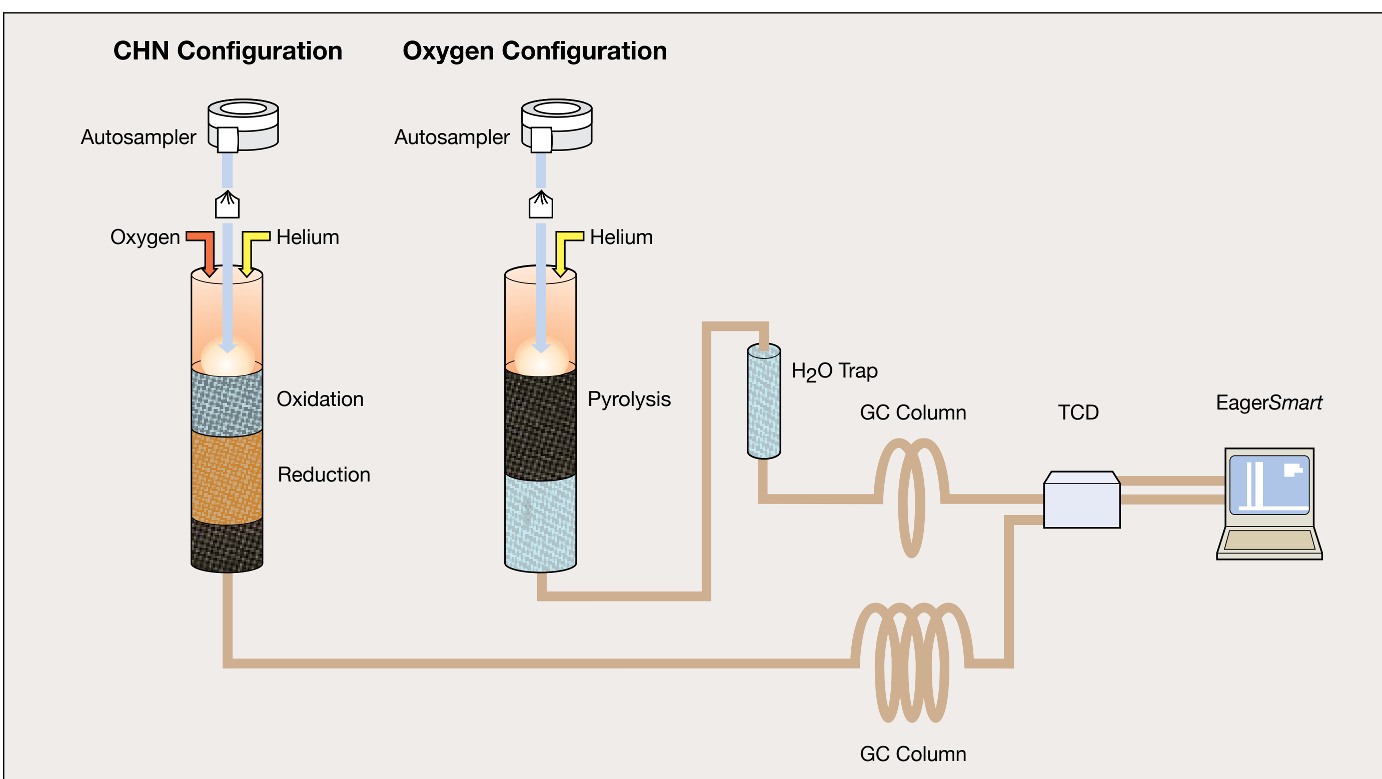

Figure 3. Flash*Smart* CHN/O configuration.

The pneumatic circuits for CHN and oxygen determination are setup simultaneously in the same system which allows the MVC Module to automatically switch between the reactors through the Eager*Smart* Software without any operational action by the user. Figure 4 shows the internal parts of the Flash*Smart* Analyzer.

Figure 4. Flash*Smart* CHN and Oxygen internal circuits.

The Eager*Smart* Data Handling Software page managing the MVC module. Figure 5 shows in the lower part how to switch from left to right furnace to pass from CHNS determination by combustion to oxygen analysis by pyrolysis. The upper of the page indicates how to switch from helium carrier gas to nitrogen or argon gas when the instrument is not used for analysis.

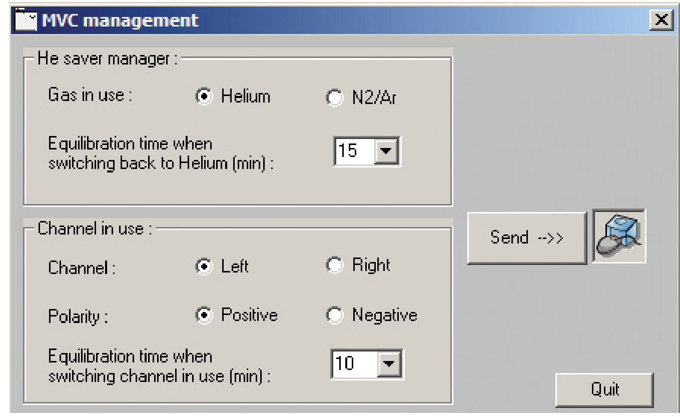

Figure 5. The MVC Module Management page on the Eager*Smart* Data Handling Software.

Results

Typical analytical tests were performed for CHN and oxygen configuration during several non-consecutive days, to evaluate the repeatability, accuracy and the stability of the system when the configuration is switched from CHN to oxygen, and vice versa. The experimental data obtained were compared with the theoretical values and the acceptable range according to the technical specification of the system.

At the end of each day the instrument remains in Stand-By Mode to reduce the consumption of helium. The Auto-Ready function was activated through the Eager*Smart* Data Handling Software to automatically wake-up the Analyzer following day and prepare for analysis.

On day 1, the instrument was calibrated for CHN and for oxygen with Acetanilide standard, using K factor as the calibration method. Then three runs of CEDFNI (cyclohexanone 2,4-dinitrophenylhydrazone) for CHN and Acetanilide for oxygen determination were performed as unknown to verify the calibration.

Table 1 shows the sequence of analysis for CHN determination. Table 2 shows the sequence of analysis for oxygen determination.

The theoretical values and the acceptable range according to the technical specification of the system of CEDFNI are 20.14 N% (±0.20), 51.79 C% (± 0.30), 5.07 H% (±0.10), while for Acetanilide the value is 11.84 O% (±0.12).

Table 1. CHN sequence of analysis for day 1.

Run	Sample name	Day/Month	Injection Time	Type	Weight (mg)	N%	C%	H%
1	Tin container	16/02	15:58	Blank				
2	Acetanilide	16/02	16:07	By-Pass				
						Theoretical values		
3	Acetanilide	16/02	16:15	STD	2.491	10.36	71.09	6.71
4	Acetanilide	16/02	16:23	STD	2.733	10.36	71.09	6.71
5	Acetanilide	16/02	16:32	STD	3.007	10.36	71.09	6.71
						Experimental data		
6	CEDFNI	16/02	16:40	UNK	2.528	19.98	51.86	5.05
7	CEDFNI	16/02	16:49	UNK	2.464	19.95	51.88	5.05
8	CEDFNI	16/02	16:57	UNK	2.542	19.95	51.84	5.05
						Theoretical values		
	CEDFNI					20.14	51.79	5.07
						Accepted range (±)		
	CEDFNI					0.20	0.30	0.10

Table 2. Oxygen sequence of analysis for day 1.

Run	Sample name	Day/Month	Injection Time	Type	Weight (mg)	O%	
1	Silver container	16/02	17:08	Blank			
2	Acetanilide	16/02	17:16	By-Pass			
						Theoretical values	Accepted range
3	Acetanilide	16/02	17:24	STD	1.995	11.84	±0.12
4	Acetanilide	16/02	17:33	STD	1.603	11.84	±0.12
5	Acetanilide	16/02	17:41	STD	1.810	11.84	±0.12
						Experimental data	
6	Acetanilide	16/02	17:50	UNK	1.732	11.77	
7	Acetanilide	16/02	17:58	UNK	1.687	11.84	
8	Acetanilide	16/02	18:06	UNK	1.789	11.81	

Acetanilide and CEDFNI for CHN, and Acetanilide for oxygen determination were analyzed as unknown without recalibration of the instrument, in four series each day with each series run in duplicate across 40 days. After each series of CHN, the instrument was automatically switched to oxygen determination using the MVC Module. Afterwards, the instrument returned to CHN determination. The switching between CHN and O configurations was performed to evaluate the data and the stability of the system. The Analyzer was stable and ready for analysis in only ten minutes after switching configurations.

Table 3 shows the CHN average data obtained, while Table 4 shows the oxygen average data. In each configuration, 208 analyses were performed. All results are acceptable and according to the specification of the instrument, indicating no effect due to configuration switching using the MVC Module from combustion to pyrolysis, or vice versa confirming the stability of the Flash*Smart* Analyzer.

Table 3. CHN average data.

Day / Month	Standard	No. Runs	N%	RSD%	C%	RSD%	H%	RSD%
17/02	Acetanilide	8	10.40	0.59	71.06	0.16	6.70	0.44
	CEDFNI	8	20.20	0.30	51.85	0.14	5.07	0.27
18/02	Acetanilide	8	10.35	0.63	71.18	0.19	6.73	0.43
	CEDFNI	8	20.17	0.23	51.91	0.14	5.08	0.24
19/02	Acetanilide	8	10.32	0.39	71.13	0.18	6.72	0.40
	CEDFNI	8	20.14	0.48	51.90	0.12	5.08	0.41
22/02	Acetanilide	8	10.39	1.08	71.27	0.69	6.78	0.77
	CEDFNI	8	20.24	0.46	51.86	0.09	5.09	0.13
23/02	Acetanilide	8	10.37	0.49	71.18	0.11	6.75	0.38
	CEDFNI	8	20.22	0.63	51.91	0.10	5.09	0.31
24/02	Acetanilide	8	10.34	0.51	71.12	0.22	6.74	0.56
	CEDFNI	8	20.08	0.28	51.97	0.13	5.08	0.29
25/02	Acetanilide	8	10.38	0.51	71.30	0.08	6.73	0.42
	CEDFNI	8	20.16	0.54	51.99	0.20	5.08	0.22
26/02	Acetanilide	8	10.37	0.51	71.30	0.24	6.71	0.38
	CEDFNI	8	20.09	0.49	51.97	0.25	5.07	0.34
23/03	Acetanilide	8	10.43	0.28	71.25	0.16	6.74	0.67
	CEDFNI	8	20.24	0.26	52.06	0.17	5.08	0.49
24/03	Acetanilide	32	10.42	0.32	71.02	0.15	6.71	0.37
	CEDFNI	32	20.16	0.36	51.79	0.12	5.06	0.25

Table 4. Oxygen average data.

Day / Month	Standard	No. Runs	O%	RSD%
17/02	Acetanilide	16	11.82	0.28
18/02	Acetanilide	16	11.86	0.37
19/02	Acetanilide	16	11.85	0.29
22/02	Acetanilide	16	11.88	0.33
23/02	Acetanilide	16	11.86	0.31
24/02	Acetanilide	16	11.89	0.28
25/02	Acetanilide	16	11.87	0.36
26/02	Acetanilide	16	11.84	0.34
23/03	Acetanilide	16	11.88	0.45
24/03	Acetanilide	64	11.86	0.36

To evaluate the linearity of the system, pure organic compounds with a range of CHN/O amounts were chosen. Instrument calibration was performed with Acetanilide (10.36 N%. 71.09 C%, 6.71 H%, 11.84 O%), Atropine (4.84 N%, 70.56 C%, 8.01 H%, 16.59 O%) and CEDFNI (20.14 N%, 51.79 C%, 5.07 H%, 23.00 O%) standards (STD) using Linear Fit as the calibration method. For CHN determination, the standard weight was 2-3 mg, while for oxygen determination the weight was 1-1.5 mg.

After the CHN sequence, the instrument switched to oxygen configuration and the oxygen sequence of analyses was performed. The switch is automated by the Eager*Smart* Data Handling Software.

Pure organic standards in a large range of concentration were selected and analyzed as unknown (UNK) to evaluate the calibration, repeatability and accuracy of the data obtained.

Table 5 shows the theoretical percentages of the pure organic standards analyzed as unknown and the accepted range according to the technical specification of the system. Table 6 shows the experimental data obtained. Each standard was analyzed in triplicate.

All data are acceptable and no effect was observed when changing the sample or switching between configurations using the MVC Module.

Table 5. Theoretical values and accepted range of pure organic standards.

Standard	Nitrogen		Carbon		Hydrogen		Oxygen	
	%	Range (±)	%	Range (±)	%	Range (±)	%	Range (±)
Acetanilide	10.36	0.10	71.09	0.30	6.71	0.10	11.84	0.12
Aspartic Acid	10.52	0.10	36.09	0.28	5.30	0.08	48.08	0.30
Atropine	4.84	0.07	70.56	0.30	8.01	0.10	16.59	0.18
BBOT*	6.51	0.10	72.53	0.30	6.09	0.10	7.43	0.10
Benzoic Acid	N/A	N/A	68.85	0.30	4.95	0.08	26.20	0.25
CEDFNI**	20.14	0.20	51.79	0.30	5.07	0.08	23.00	0.22
Methionine	9.39	0.10	40.25	0.30	7.43	0.10	21.45	0.20
L-Cystine	11.66	0.12	29.99	0.28	5.03	0.08	26.63	0.25
Nicotinamide	22.94	0.22	59.01	0.30	4.95	0.08	13.10	0.14
Sulfanilamide	16.27	0.16	41.84	0.30	4.68	0.07	18.58	0.20

* BBOT: 2,5-Bis (5-tert-butyl-benzoxazol-2-yl) thiophene
**CEDFNI: cyclohexanone 2,4-dinitrophenylhydrazone

Table 6. Repeatability and accuracy of pure organic standards.

Standard	Nitrogen		Carbon		Hydrogen		Oxygen	
	%	RSD%	%	RSD%	%	RSD%	%	RSD%
Acetanilide	10.41 10.35 10.44	0.44	71.09 71.34 71.21	0.18	6.73 6.74 6.75	0.15	11.89 11.81 11.81	0.39
Aspartic Acid	10.45 10.45 10.44	0.06	36.18 36.26 36.24	0.11	5.31 5.33 5.33	0.22	47.99 48.12 48.05	0.13
Atropine	4.91 4.88 4.89	0.31	70.60 70.73 70.62	0.10	8.04 8.04 8.04	0.00	16.73 16.63 16.67	0.30
BBOT	6.54 6.52 6.56	0.31	72.65 72.64 72.63	0.01	6.12 6.09 6.02	0.84	7.41 7.38 7.42	0.28
Benzoic Acid	NA	NA	68.99 68.83 69.01	0.14	4.91 4.99 4.96	0.81	26.32 26.20 26.26	0.23
CEDFNI	20.15 20.11 20.07	0.20	51.87 51.90 52.06	0.20	5.09 5.09 5.10	0.11	22.85 23.00 22.82	0.42
Methionine	9.40 9.46 9.44	0.32	40.38 40.42 40.26	0.34	7.48 7.50 7.48	0.15	21.46 21.41 21.38	0.19
L-Cystine	11.61 11.64 11.62	0.13	29.93 29.93 29.91	0.04	5.10 5.09 5.10	0.11	26.60 26.76 26.68	0.11
Nicotinamide	23.02 22.88 22.93	0.31	59.06 59.07 59.08	0.02	4.97 4.97 4.97	0.00	13.03 12.98 12.97	0.24
Sulfanilamide	16.17 16.21 16.24	0.21	41.77 41.85 41.91	0.16	4.69 4.68 4.70	0.21	18.52 18.52 18.48	0.14

Additionally, two tests were performed to show the accuracy and repeatability of organic compounds with high nitrogen and carbon content. For high nitrogen determination, 2-3 mg of Urea standard (46.65 N%, 20 C%, 6.71 H%) was analyzed in triplicate as unknown.

The calibration was performed with 2.5-3 mg of Imidazole standard (41.15 N%, 52.93 C%, 5.92 H%) using K factor as the calibration method. Table 7 shows the experimental CHN data of Urea in comparison to the theoretical values and the acceptable range. All data are inside the technical specification of the system and show excellent accuracy and precision.

Table 7. CHN data of Urea (46.65 N%, 20 C%, 6.71 H%).

Run	W (mg)	Nitrogen			Carbon			Hydrogen		
		%	RSD%	Acceptable range	%	RSD%	Acceptable range	%	RSD%	Acceptable range
1	2.545	46.79			20.15			6.71		
2	2.422	46.66	0.15	46.36 – 46.96	20.09	0.23	19.80 – 20.20	6.74	0.22	6.61 – 6.81
3	2.194	46.69			20.18			6.73		

For high carbon determination, the calibration were performed with 2-3 mg of Acetanilide (10.36 N%, 71.09 C%, 6.71 H%) using K factor as the calibration method. Following, about 2 mg of different high carbon content standards were analyzed in triplicate as unknown.

Table 8 shows the CHN theoretical percentages of the standards analyzed as unknown and the acceptable range. Table 9 shows the experimental data obtained which are inside the technical specification of the system and demonstrate excellent accuracy and precision of the Analyzer.

Table 8. CHN theoretical data of high carbon content standards.

Standard	Nitrogen		Carbon		Hydrogen	
	%	Acceptable range	%	Acceptable range	%	Acceptable range
Tocopherol Nicotinate	2.61	± 0.07	78.46	± 0.30	9.97	± 0.10
Polyethylene	N/A	N/A	85.70	± 0.30	14.30	± 0.15
Polystyrene	N/A	N/A	92.30	± 0.30	7.70	± 0.10
Antracene	N/A	N/A	94.34	± 0.30	5.66	± 0.09
Fluorene	N/A	N/A	93.81	± 0.30	6.03	± 0.10

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Table 9. CHN experimental data of high carbon content standards.

Run	Standard	Nitrogen		Carbon		Hydrogen	
		%	RSD%	%	RSD%	%	RSD%
1	Tocopherol Nicotinate	2.59	1.33	78.57	0.22	10.01	0.10
2		2.65		78.25		10.02	
3		2.59		78.53		10.03	
4	Polyethylene	N/A	N/A	85.89	0.11	14.26	0.33
5		N/A		85.82		14.35	
6		N/A		85.70		14.33	
7	Polystyrene	N/A	N/A	92.16	0.04	7.74	0.33
8		N/A		92.22		7.72	
9		N/A		92.17		7.77	
10	Antracene	N/A	N/A	94.22	0.12	5.67	0.18
11		N/A		94.43		5.66	
12		N/A		94.24		5.65	
13	Fluorene	N/A	N/A	93.84	0.11	6.05	0.19
14		N/A		93.87		6.07	
15		N/A		94.03		6.07	

Conclusion

The Flash*Smart* Analyzer is the optimal solution for the analysis of CHN/O 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 switch from the left channel to the right channel, or vice versa.
- Reduced helium (or argon) consumption by switching from helium (or argon) to nitrogen or argon when the system is in Stand-By Mode.
- Optionally insert, using the Eager*Smart* Data Handling Software, an external command, for example an actuator for a gas sampling valve.

The dual analytical configuration capability using the MVC Module allows you to:

- Automatically and rapidly switch from one configuration to another, increasing laboratory productivity.
- 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 by the Eager*Smart* Data Handling Software.

The all-in-one Flash*Smart* Analyzer hardware, autosamplers and software can be used for other combinations such CHNS/O, CHN/S, CHNS/CHNS, CHN/CHN, NC/S, N-Protein/S, etc. This can be achieved by only changing the consumables as the hardware and software are complete, illustrating the all-in-one nature of the Analyzer.

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