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Elemental Analysis: NCS characterization of fertilizers

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Keywords

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Goal

This application notes reports data of different solid and liquid fertilizer samples to show the accuracy of the Flash*Smart* Elemental Analyzer for samples with large range of element concentrations.

Introduction

Fertilizers are chemically characterized for research and quality control purposes in agronomy.

Nitrogen and carbon determination in soils enables the evaluation of organic matter and the calculation of the needed amount of fertilizer since characterizing nitrogen and carbon provides information on nutritional elements of the organic matter. When a lack of sulfur is determined, growth conditions of vegetables is negatively affected, and consequently the quality of the protein that they produce. For this reason the demand for the determination of nitrogen, carbon and sulfur in fertilizers and in agricultural water resulting from the use of fertilizer has increased. It is therefore very important to have an accurate and automated analytical technique, to perform fast analysis with excellent reproducibility.

The Thermo Scientific™ Flash*Smart*™ Elemental Analyzer (Figure 1) based on the dynamic combustion method (modified Dumas method), provides automated nitrogen, carbon and sulfur determination without the use of hazardous chemicals. The Flash*Smart*™ EA enables to run determinations at high and low levels of concentrations for solid and liquid samples in one single system.

This note presents data on NCS determination of fertilizers to show the performance of the Flash*Smart* Elemental Analyzer.

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Methods

The Flash*Smart* EA operates according to the dynamic flash combustion of the sample. Samples are weighed in tin containers and introduced into the combustion reactor via the Thermo Scientific™ MAS Plus Autosampler or they can be directly injected by a syringe via the Thermo Scientific™ AS 1310 Liquid Autosampler, in both cases with oxygen.

Solid samples were homogenized by a ball mill while liquid samples were analyzed without pre-treatment.

For nitrogen only determination, after combustion, the resultant gases are carried by a helium flow to a second reactor filled with copper, then swept through CO_2 and H_2O traps, a GC column and finally detected by a Thermal Conductivity Detector (TCD). Total run time less than five minutes (Figure 2).

For simultaneous NCS or for sulfur only determination, after combustion, the gases are carried by a helium flow to a layer filled with copper, then swept through a water trap, a GC column that provides the separation of the combustion gases, and finally, detected by a Thermal Conductivity Detector. Total run time is 5 – 10 min. (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.

Results

This paper presents data of different solid and liquid fertilizer samples analyzed several times. Liquid samples were homogenized by a ball mill while liquid samples were analyzed without pre-treatment.

Table 1 shows the nitrogen determination of solid fertilizer samples. Instrument calibration was performed with approximately 100 mg of urea (46.65 N%). Urea was analyzed also as an unknown sample to check the accuracy and precision of the system. The weight used for fertilizers was 4 – 5 mg for ammonium nitrate, 100 - 120 mg for potassium nitrate and 250 - 270 mg for the mix.

Figure 1. Thermo Scientific Flash*Smart* Elemental Analyzer.

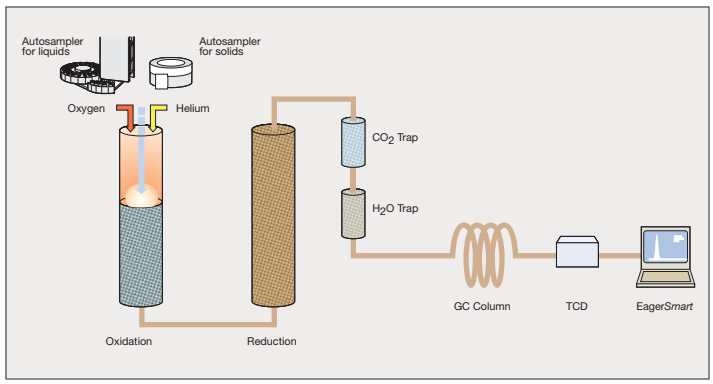

Figure 2. Nitrogen determination.

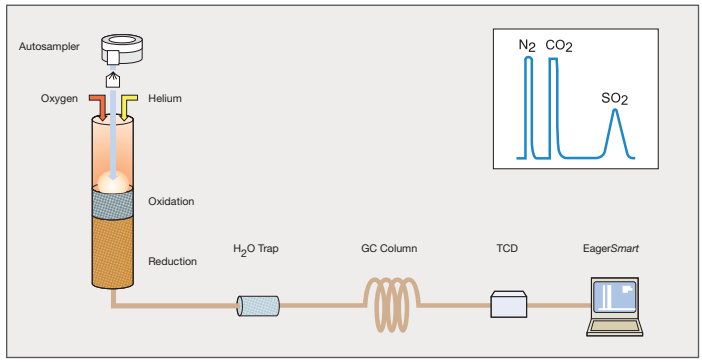

Figure 3. NCS or sulfur determination.

Table 1. Nitrogen determination in solid fertilizers.

Sample	N%	RSD%
Urea Thermo Scientific Standard 46.67 N%	46.59 - 46.56 - 46.56	0.268
Potassium nitrate KNO_3	13.80 - 13.82 - 13.94	0.561
Ammonium nitrate NH_4NO_3	34.93 – 34.93 – 34.93	0.009
Urea and ammonium sulphate $(NH_4)_2SO_4$ mix	34.56 – 34.71 – 34.78	0.324
Fertilizer mix A	17.55 – 17.51 – 17.55	0.147
Fertilizer mix B	15.44 – 15.42 – 15.57	0.524
Fertilizer mix C	39.055 - 39.131 - 38.895	0.309
Fertilizer mix D	38.765 - 38.821 - 38.729	0.121

Table 2 shows the nitrogen determination of liquid fertilizer samples. Samples were analyzed by liquid injection at 100 µl. Instrument calibration was performed with 100 ul of urea water solutions (0.3 and 7 N%).

Table 2. Nitrogen determination in liquid fertilizers.

Sample	N%	RSD%	Sample	N%	RSD%
1	0.072 0.073 0.072	1.519	6	2.789 2.849 2.830	1.075
2	0.220 0.218 0.220	0.607	7	7.133 7.103 7.148	0.324
3	0.310 0.316 0.316	1.103	8	10.197 10.160 10.167	0.193

Table 3 shows the nitrogen and carbon determination of solid fertilizers. To calibrate the system, 4 - 5 mg of aspartic acid (10.52 N%, 36.09 C%) was used and samples were weighed at 5 - 6 mg.

Table 3. Nitrogen and carbon determination in solid fertilizers.

Sample	Nitrogen		Carbon	
Name	%	RSD%	%	RSD%
1	3.876 3.869 3.891	0.274	29.837 30.476 29.813	1.250
2	17.873 17.807 17.874	0.215	0.231 0.227 0.228	0.910
3	33.466 33.505 33.550	0.125	0.038 0.037 0.039	1.860

Table 4 shows the nitrogen and carbon determination of liquid fertilizers. To calibrate the system 4 - 5 mg of aspartic acid (10.52 N%, 36.09 C%) was used and samples were weighed at 10 - 15 mg adsorbed on approximately 10 mg of Chromosorb® WAW 30-60 mesh.

Table 4. Nitrogen and carbon determination in liquid fertilizers.

Sample	N%	RSD%	C%	RSD%
1	0.7881 0.7752	0.2412	0.4552 0.4675	1.8852
2	1.6335 1.6329	0.0260	0.0133 0.0122	6.1005
3	4.0065 3.9530	0.9506	0.0416 0.0404	2.0696
4	4.0576 4.0105	0.8256	0.3475 0.3552	1.5497
5	0.6753 0.6883	1.3483	0.4791 0.4799	0.1180

Table 5 shows the NCS data of solid fertilizer samples. The calibration was performed with 2 - 3 mg of BBOT*, Nicotinamide, Cystine and Urea, and the sample weight used was 3 - 4 mg.

** BBOT (6.51 N%, 72.53 C%, 7.44 S%), Nicotinamide (22.94 N%, 59.01 C%), Cystine (11.66 N%, 29.99 C%, 26.69 S%) and Urea (46.65 N%, 20 C%)*

Table 5. Simultaneous NCS determination in solid fertilizers.

Sample	N%	RSD%	C%	RSD%	S%	RSD%
1	10.538	0.230	4.583	1.821	0.218	1.862
	10.582		4.523		0.222	
	10.578		4.688		0.214	
2	24.613	0.247	0.221	0.686	1.232	0.126
	24.732		0.223		1.234	
	24.698		0.220		1.231	
3	25.500	0.094	0.046	3.901	0.085	0.713
	25.538		0.042		0.084	
	25.546		0.044		0.085	
4	20.632	0.305	0.943	0.411	23.939	0.115
	20.755		0.936		23.986	
	20.669		0.939		23.987	

Table 6 shows the sulfur data of solid samples. The calibration was performed with 2 - 3 mg of BBOT* (7.44 S%) and the sample weight used was 2 - 4 mg.

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

Table 6. Sulfur determination in solid fertilizers.

Fertilizer 1		Fertilizer 2		Fertilizer 3	
S%	RSD%	S%	RSD%	S%	RSD%
2.740	0.735	3.616	0.857	4.873	0.638
2.698		3.614		4.849	
2.722		3.664		4.860	
2.749		3.623		4.810	
2.738		3.681		4.803	

Conclusions

All data were obtained with a good reproducibility and no matrix effect was observed when changing from solid to liquid samples.

The Flash*Smart* EA enables to perform simultaneous NCS determination in a single run, and consequently sulfur, with no hardware modification required.

Nitrogen only determination can be performed by increasing the sample weight and changing the configuration.

The Flash*Smart* EA allows to obtain automated analysis with lower cost per sample, while obtaining excellent accuracy and reproducible data.

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