APPLICATION NOTE 42490

Elemental Analysis: Nitrogen and simultaneous CHNS determination of Indian tea by Dumas method

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Goal

To assess the performance of the elemental analyzer using helium and argon as carrier gas in alternative to the Kjeldahl method and CHNS determination of Indian tea analysis.

Introduction

Indian tea (such as Assam tea and Darjeeling tea) is commercialized globally and has made India one of the largest tea producers in the world.

For research and quality control of tea, elemental analysis gives important information: from the type, plant age, leaf age, leaf position and to soils nutrient. By the determination of nitrogen content in tea leaves, the tea tree growth and nitrogen nutrition in tea leaves can be monitored as well as fertilization methods can be optimized. Traditionally, the Kjeldahl method was used to determine nitrogen and consequently protein content.

Also carbon, hydrogen and sulfur determination can be used to characterize tea. For the determination of carbon, hydrogen, nitrogen and sulfur the Dumas method, based on combustion, can be utilized. The capabilities of the Dumas method (combustion method) for the determination of nitrogen have been greatly improved to make faster, safer and more reliable than the traditional Kjeldahl method.

The Thermo Scientific™ Flash*Smart*™ Elemental Analyzer (Figure 1), based on the dynamic flash combustion of the sample, meets a wide array of requirements of laboratories such as accuracy, day by day reproducibility and high sample throughput. The Flash*Smart* EA uses helium as carrier gas, which ensures high sensitivity.

Figure 1. Thermo Scientific Flash*Smart* Elemental Analyzer

Considering the need for cost efficiencies and the likely increase in helium gas cost, due to its possible shortage, an alternative for the carrier gas, is needed. Argon, which is readily available, can be used as alternative to helium in the Flash*Smart* EA.

The Flash*Smart* EA allows also the simultaneous determination of nitrogen, carbon, hydrogen and sulfur by combustion method using the same system with a specific single combustion-reduction reactor for the complete characterization of all type of matrices.

Methods

The Elemental Analyzer operates according to the dynamic flash combustion of the sample. The sample is weighed in tin containers and introduced into the combustion reactor via the Thermo Scientific™ MAS Plus Autosampler with oxygen.

For nitrogen determination, after combustion, the produced gases are carried by a helium or argon 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) (Figure 2). A complete report is automatically generated by the Thermo Scientific™ Eager*Smart*™ Data Handling Software and displayed at the end of the analysis. The Eager*Smart* Data Handling Software controls all analytical parameters of the instrument including the oxygen flow and the timing of oxygen injection. It calculates automatically the amount of oxygen, relative to the sample matrix and sample weight, through the proprietary Thermo Scientific™ OxyTune Function ensuring the complete combustion of the sample. Through this optimization also decreases the cost per analysis by not wasting oxygen or consuming the copper unnecessarily. Figure 3 shows the OxyTune Categories.

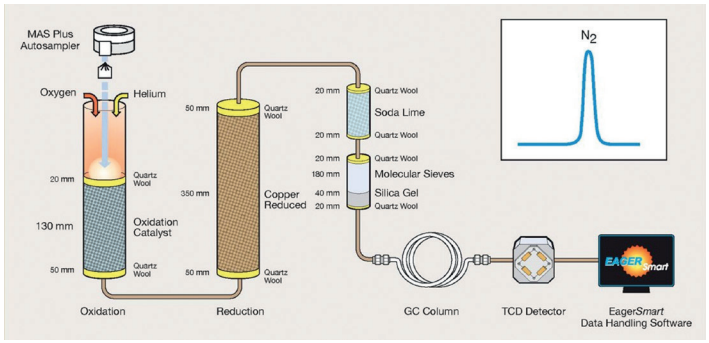

Figure 2. Nitrogen configuration

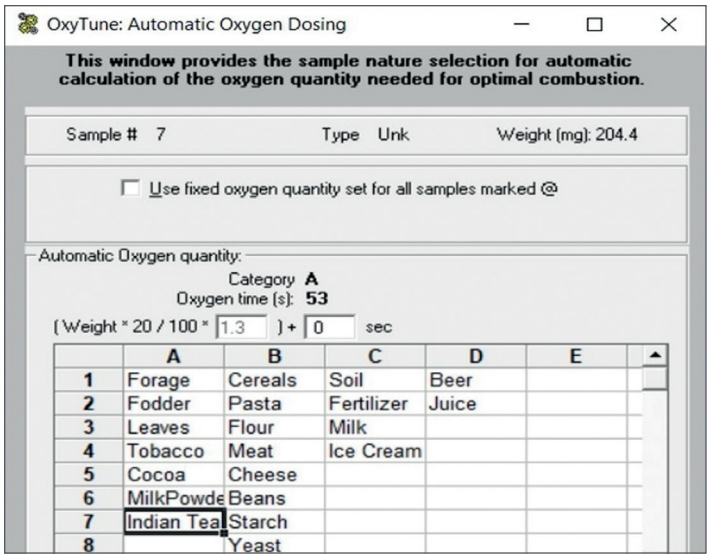

Figure 3. OxyTune Eager*Smart* Data Handling Software window

For CHNS determination, after combustion, the produced gases are carried in a helium carrier gas to a layer filled with copper. The analyte then enters the GC column, which separates the produced gases before detection by a Thermal Conductivity Detector (TCD) (Figure 4). For weight percent determination a complete report is automatically generated by the Thermo Scientific™ Eager*Smart* Data Handling Software and displayed at the end of the analysis.

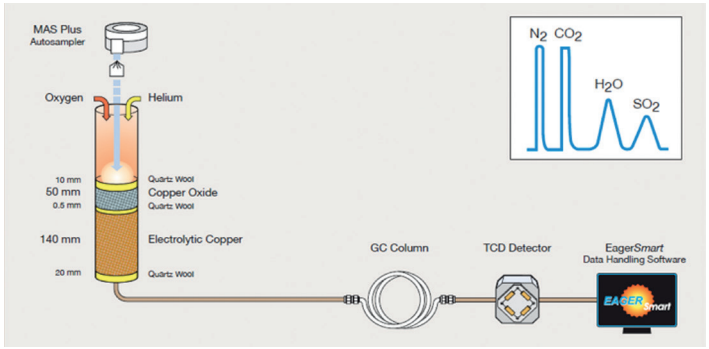

Figure 4. CHNS configuration

Results

Seven Indian tea samples were analyzed to demonstrate the performance of the Flash*Smart* Elemental Analyzer using helium and argon as carrier gas for nitrogen determination. All the tea samples mostly used for breakfast; only green tea sometimes used for medicine. The Indian tea samples analyzed come from different part of India: Maharashtra, North India Utter Pradesh, Gujarat, South and North India, Assam-West Bengal, North India, Madhya Pradesh. The Indian tea samples were dried and homogenized by a ball mill.

For nitrogen configuration, the instrument calibration was performed with aspartic acid standard (10.52 N%) using K factor as calibration method. The calibration was evaluated by the analysis of aspartic acid as unknown before and after the samples. The data obtained fall within the technical specification of the system for aspartic acid (theoretical 10.52 N%, accepted range 10.42–10.62 N%). Table 1 shows the sample weight, the standard and the OxyTune Category when helium or argon is used as carrier gas.

Table 2 shows the nitrogen data obtained using helium and argon as carrier gas. Each sample was analyzed three times. The data are comparable and the repeatability is more than acceptable giving in both cases a RSD% less than 2% as Official Methods requirements.

Table 2. Nitrogen data of Indian tea samples using helium and argon carrier gas

Tea sample	Indian region of provenience	Helium carrier gas		Argon carrier gas	
		N%	Std. Dev. RSD%	N%	Std. Dev. RSD%
1	Maharashtra	3.42 3.43 3.41	0.006 0.17	3.39 3.40 3.40	0.006 0.17
2	North India Utter Pradesh	3.54 3.55 3.53	0.010 0.28	3.52 3.50 3.53	0.015 0.43
3	Gujarat	3.88 3.86 3.84	0.020 0.52	3.81 3.84 3.80	0.021 0.54
4	South and North	3.87 3.86 3.90	0.021 0.54	3.82 3.88 3.83	0.032 0.84
5	Assam, West Bengal	3.71 3.69 3.70	0.010 0.27	3.65 3.71 3.68	0.030 0.81
6	North	3.68 3.67 3.66	0.010 0.27	3.68 3.67 3.65	0.015 0.42
7	Madhya Pradesh	3.66 3.63 3.63	0.017 0.47	3.62 3.63 3.61	0.010 0.28

Table 1. Samples, standard and OxyTune information

Tea sample	Indian provenience	Helium carrier gas			Argon carrier gas		
		Weight (mg)	OxyTune category	Calibration	Weight (mg)	OxyTune category	Calibration
1	Maharashtra	190–200	A	Aspartic acid 50–70 mg	120–130	A	Aspartic acid 60–80 mg
2	North India Utter Pradesh	160–180	A		60–80	A	
3	Gujarat	190–210	A		80–90	A	
4	South and North	180–200	A		90–100	A	
5	Assam, West Bengal	150–190	A		70–90	A	
6	North	180–190	A		70–80	A	
7	Madhya Pradesh	180–190	A		60–70	A	

At last the data of nitrogen, carbon, hydrogen and sulfur obtained simultaneously by combustion method is shown in Table 3. The calibration was performed with 2–3 mg BBOT standard (2,5-Bis (5-ter-butyl-benzoxazol-2-yl) thiophene) using K factor as calibration method. The calibration was evaluated by the analysis of BBOT and aspartic acid as unknown. Table 3 shows the CHNS data of the samples analyzed three times each sample, sample weight 3–4 mg.

Table 3. CHNS data of Indian tea samples

Tea sample	Nitrogen		Carbon		Hydrogen		Sulfur	
	N%	Std.Dev. RSD%	C%	Std.Dev. RSD%	H%	Std.Dev. RSD%	S%	Std.Dev. RSD%
1	3.40 3.39 3.40	0.006 0.17	47.00 46.89 47.10	0.105 0.22	5.87 5.83 5.74	0.067 1.14	0.190 0.198 0.195	0.004 2.08
2	3.54 3.55 3.57	0.015 0.43	47.33 47.20 47.26	0.065 0.14	5.75 5.69 5.74	0.032 0.56	0.200 0.200 0.207	0.004 1.99
3	3.83 3.87 3.84	0.021 0.54	47.09 47.04 47.06	0.025 0.05	5.62 5.54 5.65	0.057 1.01	0.200 0.196 0.197	0.002 1.05
4	3.88 3.88 3.85	0.017 0.45	46.16 46.08 46.13	0.040 0.09	5.86 5.78 5.77	0.049 0.85	0.196 0.201 0.200	0.003 1.32
5	3.73 3.73 3.71	0.011 0.31	47.00 47.35 47.49	0.252 0.53	5.74 5.71 5.63	0.057 0.99	0.203 0.201 0.207	0.003 1.50
6	3.67 3.67 3.69	0.011 0.31	46.87 47.04 46.97	0.085 0.18	5.69 5.68 5.66	0.015 0.27	0.200 0.201 0.203	0.001 0.76
7	3.64 3.63 3.65	0.010 0.27	47.42 47.23 47.31	0.095 0.20	5.77 5.74 5.72	0.025 0.44	0.196 0.200 0.199	0.002 1.05

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Conclusions

For nitrogen determination the Flash*Smart* Elemental Analyzer, based on the combustion method (Dumas), offers advantages over the Kjeldahl Method in terms of automation, ease of use and cost per sample.

Considering the need for an alternative carrier gas, the Flash*Smart* Elemental Analyzer can perform nitrogen analysis without matrix effect using argon as carrier gas. The nitrogen data obtained are comparable with those obtained using helium as carrier gas. The RSD% obtained was less than 2% of the performance requirements of the Official Methods.

Good repeatability was also obtained for CHNS determination and the nitrogen values are comparable with those obtained using the nitrogen configuration.

No memory effect was observed, indicating complete combustion and detection of the element independent of the sample matrix.

The application showed that the Dumas Method meets manufacturers and laboratories requirements, including compliance to official methods.

The Dumas Combustion method has been approved and adopted by several Official Organizations such as ASBC, AOAC, AACC, AOCS, IDF, IFFO and ISO.

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