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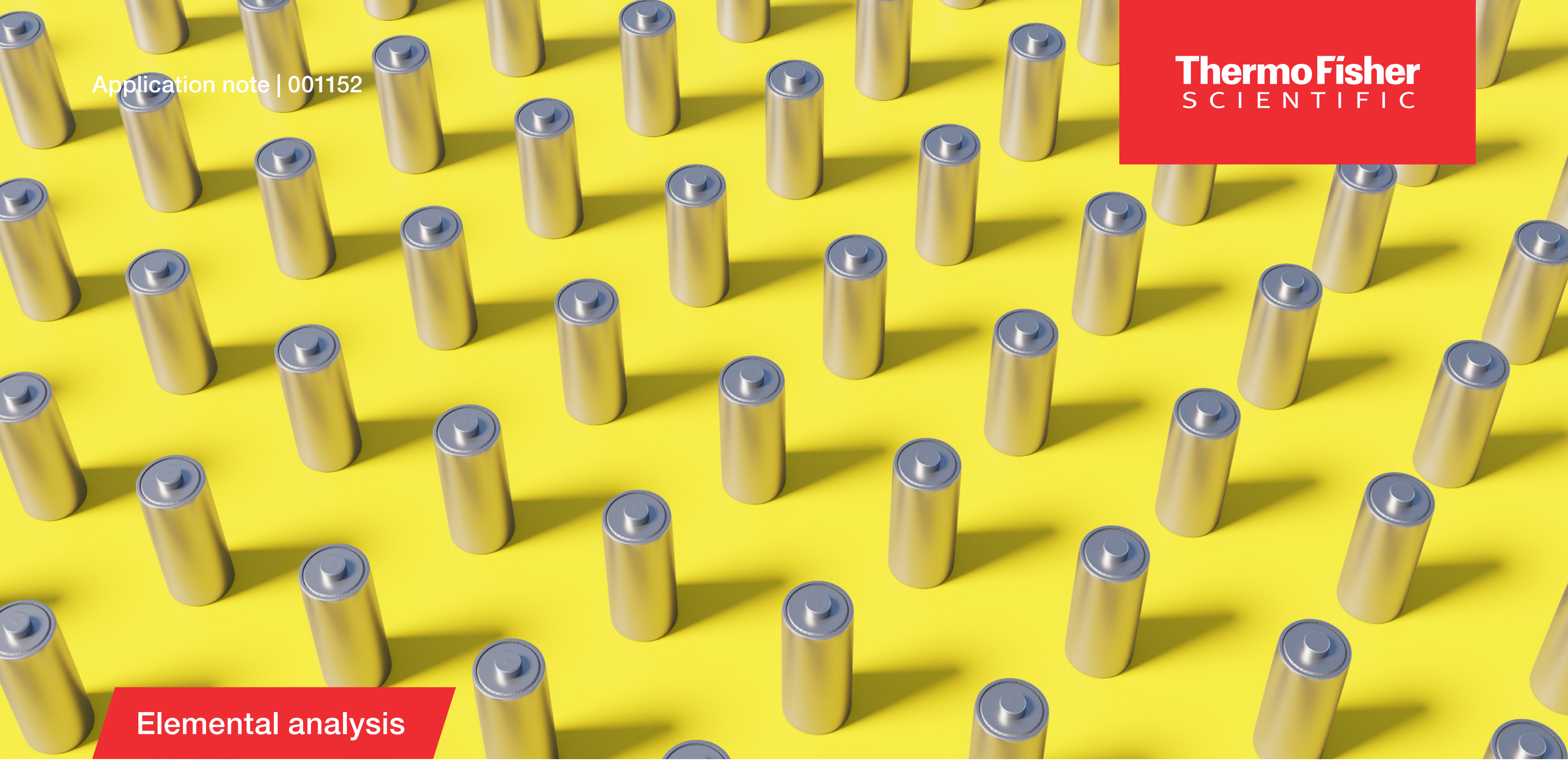

Elemental analysis

Characterization of battery samples by the Flash*Smart* Elemental Analyzer

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Keywords
Batteries, carbon, combustion, graphite, sulfur, quality control

Goal
Demonstrate accurate and precise CHNS determination in battery samples using the Thermo Scientific™ *FlashSmart*™ Elemental Analyzer

Introduction
During the development and production of batteries, quality control (QC) of raw, intermediate and finished products is required to assure high quality and performance, and also safety. A simple and fast way to monitor elemental composition of these products for QC is by using the Flash*Smart* Elemental Analyzer (EA). Elemental characterization by Flash*Smart* EA mainly focuses on two elements: **carbon and sulfur**. Carbon and sulfur elemental determination allows accessing information on the purity of material which is critical as changes in production can cause the introduction of impurities and ultimately impact the lifetime and energy storage capacity of the battery.

The electrochemical properties of $LiFePO_4$ cathodes with different carbon contents are analyzed to determine the role of carbon as conductive additive, whereas as lead paste exhausted batteries are analyzed for sulfur content. Carbon and sulfur determination are also performed on graphite material used in negative electrode production.

Method
The Flash*Smart* Elemental Analyzer (Fig. 1) uses dynamic flash combustion to perform quantitative elemental analysis, offering the flexibility to perform CHNS analysis in a single run.

Figure 1. Thermo Scientific Flash*Smart* Elemental Analyzer

For simultaneous NCS and CHNS determination, samples are weighed in tin containers and introduced into the combustion reactor via the Thermo Scientific™ MAS Plus Autosampler. After combustion, the produced gases are separated in a GC column, and detected by a Thermal Conductivity Detector (TCD) (Figure 2). For weight percent determination of CHNS a complete report is automatically generated by the Thermo Scientific™ Eager*Smart*™ Data Handling Software and displayed at the end of the analysis.

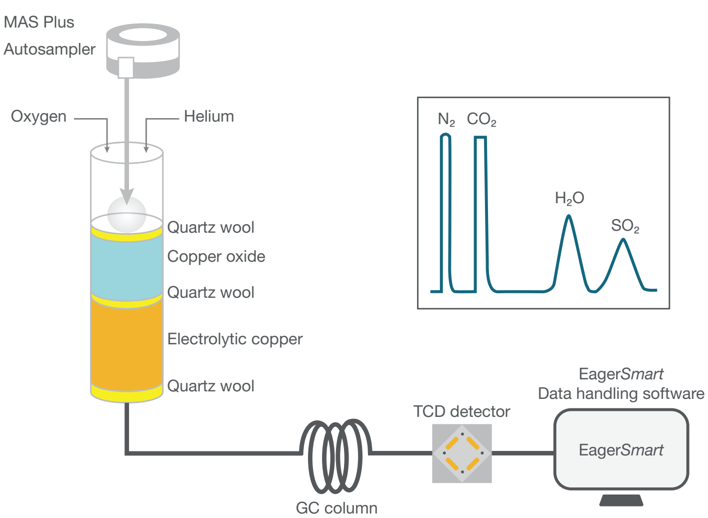

Figure 2. CHNS configuration

Results

The analysis of different batteries and graphite samples were performed to demonstrate the performance of the Flash*Smart* EA for CHNS wt% determination.

Carbon data of $LiFePO_4$ batteries

The capacity of the $LiFePO_4$ cathode increases as conductive additive content (carbon black or mixture of carbon black and graphite) is increased. Therefore, accurate and precise carbon content determination, as demonstrated in Table 1, is critical for QC. The calibration was performed with aspartic acid standard using K factor as calibration method. The sample weight was approx. 8-9 mg.

Table 1. Carbon data of $LiFePO_4$ batteries

Sample $LiFePO_4$ battery	C%	RSD%
1	4.54	0.77
	4.59	
2	7.57	0.47
	7.52	
3	4.82	0.15
	4.81	
4	5.16	0.69
	5.11	

Sulfur data of lead paste ($PbSO_4$, PbO_2, Pb) exhausted batteries

Sulfur content determination is important because sulfur affects the lifetime of the battery and the reduction of battery capacity. Table 2 demonstrates accuracy and precision of sulfur weight% determination in lead paste exhausted batteries. The calibration was performed with 2,5-Bis-(5-tert-butylbenzoxazol-2-yl)-thiophen (BBOT) standard using K factor as calibration method. The sample weight was 2-6 mg depending on the sample.

Table 2. Sulfur data of lead paste ($PbSO_4$, PbO_2, Pb) exhausted batteries

Sample $PbSO_4$, PbO_2, Pb exhausted battery	S%	RSD%
1	6.23	0.43
	6.22	
	6.18	
2	1.67	0.92
	1.66	
	1.64	
3	7.18	0.29
	7.19	
	7.22	
4	0.778	0.97
	0.772	
	0.762	
5	1.74	0.67
	1.72	
	1.72	

CHNS determination by TCD detector for QC of graphite

For lithium-ion batteries, the negative electrode (anode) material is generally made from graphite powder. Elemental analysis is used for C and S determination of graphite material in batteries for assessing material purity. Table 3 demonstrates how in a single run, 4 elements (CHNS) can be determined with high precision.

Graphite samples were homogenized by a ball mill. For CHNS determination, the calibration was performed with 2 – 3 mg BBOT standard using K factor as calibration method; the sample weight was 1.8 – 2.2 mg; to obtain complete combustion the oxygen flow was 300 ml/min and the oxygen injection time 13 seconds.

Conclusions

The Flash*Smart* Elemental Analyzer, based on the combustion (modified Dumas method), determines CHNS of battery material in a wide range from low to high content and without the use of sample digestion or toxic chemicals, which is normally required by traditional methods. Simultaneous CHNS or NCS determination can be easily done in a single run, with support of dedicated Eager*Smart* Data Handling Software that provides you with customized reports of your data. The Flash*Smart* Elemental Analyzer demonstrates excellent repeatability, reproducibility, accuracy, and precision in the analysis of different battery material in a cost-efficient and simple way.

Table 3. CHNS determination of graphite samples

Sample Graphite	N%	RSD%	C%	RSD%	H%	RSD%	S%	RSD%
1	0.0700	3.05	97.52	0.05	0.0200	8.98	0.0600	4.91
	0.0722		97.51		0.0199		0.0658	
	0.0744		97.60		0.0170		0.0612	
2	0.0600	3.25	91.59	0.30	0.0900	4.55	-	-
	0.0563		91.27		0.0823			
	0.0589		91.82		0.0850			

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