Using a Range of Techniques to Provide Comprehensive Data on Battery Materials

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Introduction

Understanding battery materials from a chemical, thermal and mechanical standpoint is of incredible importance for ensuring quality and understanding every part of the battery manufacturing chain, from mining to failure and recyclability. There is a plethora of techniques available to analyze and investigate battery materials and components.

Detection of H_2O and CO_2 on Electrodes

TG-MS

One of the key parameters in a finished electrode is the water content. The presence of moisture can seriously hinder the performance of the finished battery due to side reactions with the electrolyte and other processes.[1]

One of the most common methods for determination of water is a Karl-Fisher titration. However, there are two disadvantages to this:

- Requires grinding of the sample which could introduce more moisture during sample preparation
- Only quantifies water

Thermogravimetric analysis hyphenated to mass spectrometry provides a method whereby adsorbed water can be quantified, alongside other small molecules.

Table 1 shows the instrumental parameters used for electrode analysis.

Table 1. TG-GC/MS Instrument Parameters

Parameter	Value
TGA Temperature Range	30 – 450 ºC
TGA Ramp Rate	20 ºC/min
Sample Purge Rate (Purge Gas)	60 mL/min (He)
Pump Rate	30 mL/min
Interface Set Temperature	270 ºC
Selected Ions	18 (H_2O) & 44 (CO_2) amu

The TG-MS data from an anode sample is shown in Figure 1.

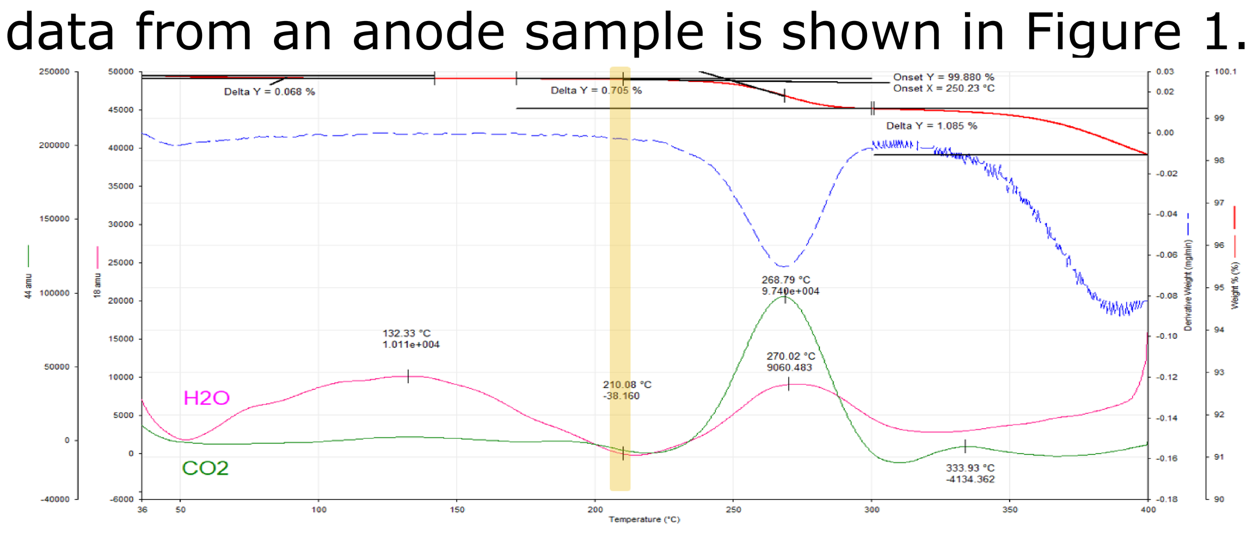

Figure 1. TG-MS data showing weight loss events and abundance of H_2O and CO_2

This data demonstrates that there is adsorption of water to the electrode at around 210 ºC, whereas there is strong co-desorption of H_2O and CO_2 at 270 ºC. This data could be used to inform processing parameters used in the manufacturing process.

Verification of Electrolyte Raw Materials

FT-IR

Verification of incoming raw materials is an important step in any end market, not least those in which the standard of the product can fall dramatically as a result of incorrect raw materials.

FT-IR spectroscopy provides a rapid method whereby materials can be identified by searching sample spectra against spectral libraries.

Electrolyte raw materials were measured using attenuated total reflectance (ATR) FT-IR spectroscopy at a resolution of 4 cm^{-1}, 4 scans and a range of 4000 – 450 cm^{-1}. The spectra, with the library results overlaid, are shown in Figure 2.

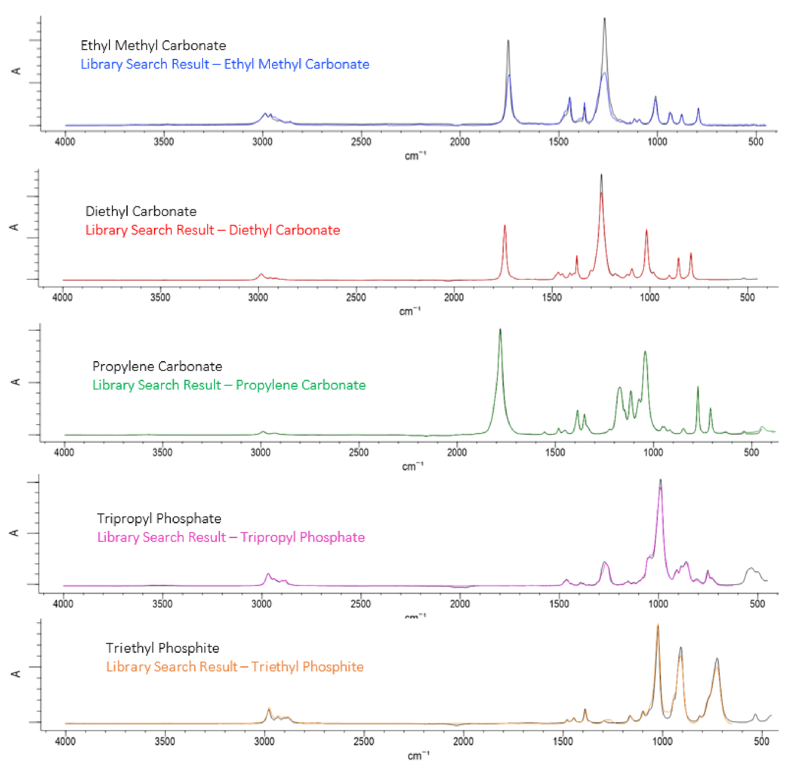

Figure 2. FT-IR Spectra of Electrolyte Raw Materials

This method demonstrates the ease with which electrolyte raw materials may be verified with infrared spectroscopy.

High-Precision Determination of Element Ratios in Battery Materials

ICP-OES

An important aspect in obtaining optimum battery performance is accurately knowing the ratios of major components as deviations can have a major impact. Furthermore, understanding the concentration of both major and minor constituents in recycled active material will be incredibly important in the near future.[2]

HP-ICP-OES provides the ability to measure impurities down to the µg/L level, while achieving an RSDs of 0.1% or less for major components.

A mimic digestion of battery materials, containing Mn, P, Li, Ni and Zn was measured with the following instrumental parameters.

Table 2. ICP-OES Instrumental Conditions

Parameter	Value
Nebulizer	SeaSpray™
Spray Chamber	Baffled Glass Cyclonic
Sample Uptake Rate	1.0 mL/min
RF Power	1500 W
Injector	2.0 mm id Alumina
Nebulizer Gas Flow	0.65 L/min
Auxiliary Gas Flow	0.2 L/min
Plasma Gas Flow	10 L/min
Torch Position	-3
Replicates	3

The % Recovery from spiked samples for all five elements is shown in Figure 3. This data shows an excellent spike recovery range (99.2-100.4%)

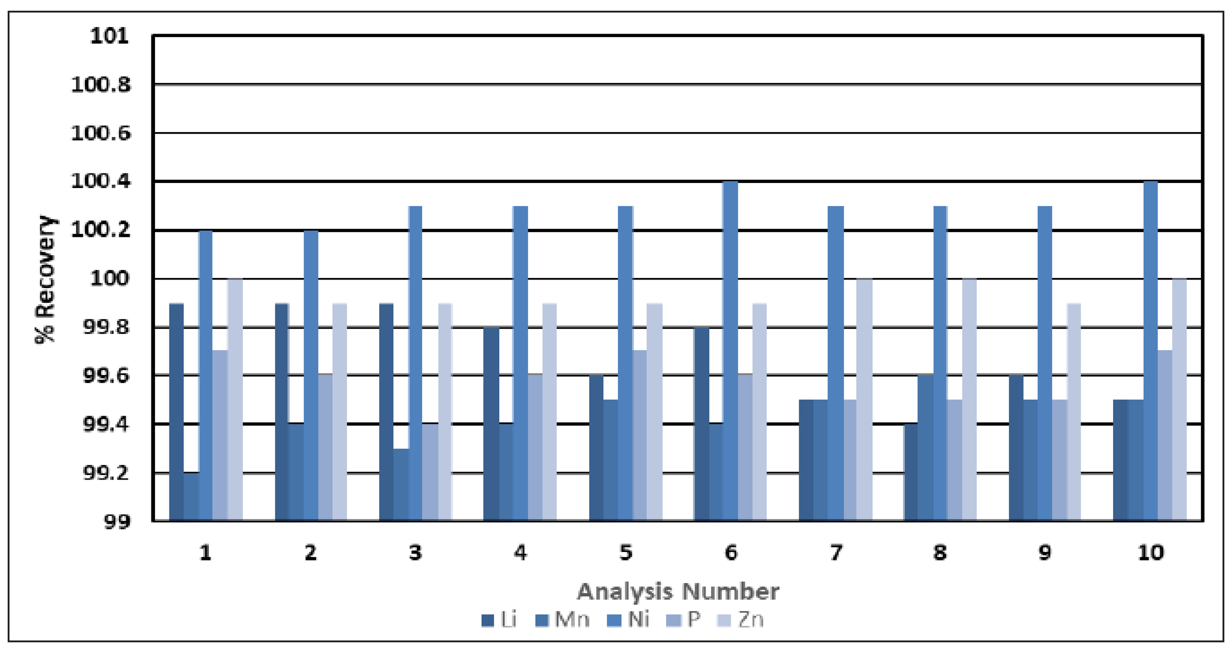

Figure 3. % Recovery for the major elements

The RSD values for both the major and minor components are shown in Table 3.

Table 3. RSDs for Major Elements and Impurities.

Major Components	
Element	**RSD**
Mn	0.12%
P	0.10%
Li	0.19%
Ni	0.07%
Zn	0.05%
Impurities	
Element	**RSD**
Cu	1.3%
Cd	1.2%
Hg	1.8%
Mo	1.3%
Pb	0.84%

Summary

This work shows the significance of accurate analysis throughout the battery value chain. Accurate and precise analytical techniques provide insights into batteries and raw materials from mining to recycling, giving manufacturers and R&D facilities the ability to investigate key areas in improving a facilitating the energy storage space.

References

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2. Nasser, O. A. and Petranikova, M. (2021), Review of Achieved Purities after Li-ion Batteries Hydrometallurgical Treatment and Impurities Effects on the Cathode Performance, *Batteries,* **7**, 60