



NEX QC+ QUANTEZ



- **Model**
NEX QC+ QuantEZ®
- **Excitation**
Direct with filters
- **X-ray tube**
4 W Ag-anode
- **Detector**
SDD
- **Autosampler**
6-position
- **Sample type**
Powder
- **Analysis time**
600 sec
- **Atmosphere**
Helium
- **Optional**
Manual compaction press

SCOPE

The analysis of black mass powder is demonstrated using NEX QC+ QuantEZ, which features Rigaku RPF-SQX Fundamental Parameters (FP) for semi-quant standardless screening and user-defined Matching Library to optimize accuracy.

BACKGROUND

Lithium batteries have many uses, from regular batteries to cell phones, tablets, computers, and electric vehicles. At end-of-life, the batteries can be recycled to ensure a sustainable, environmentally friendly use of the raw materials. The devices are collected, disassembled, and the batteries are then shredded and ground. During the process, the plastics are separated and removed leaving a powder called black mass, rich in the cathode metals Co, Mn and Ni, base metals Cu, Fe and Al, as well as the graphite from the anode.

Recovering and repurposing the metals reduces the need to mine fresh raw materials and allows for a sustainable circular economy in the lithium battery industry. Rigaku offers the NEX QC+ QuantEZ EDXRF analyzer for the analysis of black mass during the recycling and recovery of the raw materials to make new, fresh batteries.

SAMPLE PREPARATION

Ensure the black mass powder is homogeneous. Place 5 g of powder in a 32 mm XRF sample cup prepared using 4 μm polypropylene film. If desired, compact slightly using the Rigaku manual compaction press. Do not tap-pack the sample, as the metals are free metals and may settle towards the bottom of the cup. When making splits for ICP analysis for use in a Matching Library, ensure the master sample is homogeneous before taking splits.



RIGAKU RPF-SQX FUNDAMENTAL PARAMETERS (FP)

Rigaku RPF-SQX FP software estimates elemental concentration based on XRF theory called Fundamental Parameters (FP). Rigaku Profile Fitting (RPF) automatically deconvolutes spectral peaks and models the sample matrix using fundamental XRF equations to provide semi-quantitative measurements of elemental concentrations without the need for any known standards.

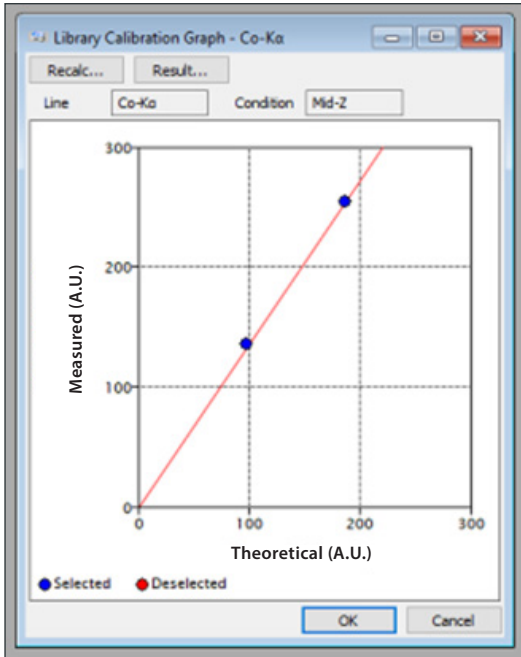
RESULTS: FP STANDARDLESS SEMI-QUANT FOR SCREENING

In this example, the semi-quant analysis of a typical black mass sample is shown. The method set-up is based on the FP Powder template adjusted for black mass with the balance of the material defined as graphite. Standardless FP is excellent for screening to measure the overall elemental composition of the black mass.

Typical Black Mass Sample			
Element	Result (mass%)	Stat. Error	Typical LLD
Co	13.556	0.008	0.0026
Mn	9.963	0.008	0.0014
Ni	16.163	0.008	0.0037
Al	6.156	0.009	0.0075
Cu	6.605	0.009	0.0075
Fe	2.104	0.003	0.0050
Si	0.7577	0.0020	0.0027
P	2.099	0.002	0.0018
S	0.1275	0.0004	0.0008
Cl	0.0317	0.0002	0.0006
K	0.2074	0.0067	0.016
Ca	0.4066	0.0050	0.0077
Ti	0.4774	0.0030	0.0032
V	0.0527	0.0012	0.0029
Cr	0.0382	0.0008	0.0018
Zn	0.3678	0.0013	0.0014
As	0.0028	0.0002	0.0007
Zr	0.1358	0.0003	0.0003
Ag	0.0026	0.0001	0.0003
Cd	0.1214	0.0004	0.0003
Sn	0.0766	0.0003	0.0004
Sb	0.0256	0.0003	0.0006
Ba	0.0248	0.0006	0.0015
Pb	0.0528	0.0005	0.0005

RIGAKU USER-DEFINED MATCHING LIBRARY

The user can easily tune the semi-quant results using a Matching Library by measuring one or more samples of the material with known elemental assay values. In this way, the XRF is tuned specifically to the black mass and referee values to ensure optimum accuracy and reliable, high-quality data without the need for a large set of assayed standards for calibration.



RESULTS: RIGAKU FP WITH MATCHING LIBRARY FOR IMPROVED ACCURACY

In this example the semi-quant accuracy is improved using a 2-point Matching Library for the main metals Co, Mn, Ni, Al, Cu and Fe. Accuracy can be further improved by simply adding a few more samples with ICP numbers to the Matching Library.

Typical Black Mass Sample			
Element	ICP Value (mass%)	NEX QC+ QuantEZ Result Semi-quant Screening (mass%)	NEX QC+ QuantEZ Result Using Matching Library (mass%)
Co	11.94	13.56	12.16
Mn	8.87	9.96	8.47
Ni	14.58	16.16	14.09
Al	5.28	6.16	4.63
Cu	7.30	6.61	7.53
Fe	1.34	2.10	1.49

CONCLUSION

The Rigaku NEX QC+ QuantEZ offers excellent performance for the elemental analysis of black mass. The powerful combination of Rigaku RPF-SQX FP and Matching Library yields accurate and reliable results, making NEX QC+ QuantEZ an excellent tool for the elemental identification, screening, and characterization of black mass during the recycling process.