

SCOPE

The analysis of carbon black, residual oil and oil ash is demonstrated using semi-quant Fundamental Parameters (FP) .

BACKGROUND

Carbon black is made from coal tar and various petroleum tars from the cracking process. Carbon black is a crystalline form of carbon with very high surface area to volume ratio and is used mainly as a reinforcing filler in tires and used to strengthen engine hoses and gear belts. It is also used as copier toner inks and as pigment in polymers. The elemental characterization of the carbon black is critical to ensure proper quality of the various products. To meet this industry need Applied Rigaku Technologies offers Energy Dispersive X-ray Fluorescence (EDXRF), ideal for the fast and simple analysis of carbon black and related products.



INSTRUMENTATION

Model:	Rigaku NEX DE
X-ray tube:	60kV 12W Ag-anode
Excitation:	Direct with filters
Detector:	High throughput SDD 500,000+ cps
Analysis Time:	300 seconds total
Atmosphere:	Helium
Autosampler:	15-position Sample Tray (32mm)
Option:	Manual Compaction Press

NEX DE



SAMPLE PREPARATION & FP METHODOLOGY

A carbon black sample is powder and prepared by weighing 4 grams of sample in a 32mm XRF sample cup and hand compacting to 250 inch pounds of torque. Ash samples are prepared in the same way using approximately 0.6g of sample. Oil samples are prepared by simply placing 4g of oil in the sample cup.

The default FP templates for Powder and Oil were used to measure the samples by semi-quant FP. These default templates can be further optimized by adding one or more known standards into a Matching Library. A Matching Library can be easily created by the user and is used in conjunction with the standard FP library in the modeling of a sample matrix and calculation of concentration results.

RPF-SQX SEMI-QUANT FP RESULTS

Sample ID: Carbon Black			Units: %
Component	NEX DE VS Value	Stat. Error	Est. LLD**
Na	ND*	0.05	0.16
Mg	ND*	0.006	0.017
Al	0.0539	0.0014	0.0033
Si	(0.0029)*	0.0005	0.0016
P	0.0375	0.0006	0.0018
S	1.70	0.001	0.001
Cl	0.0015	0.0002	0.0005
K	0.0344	0.0030	0.0078
Ca	0.0202	0.0015	0.0038
Ti	ND*	0.0003	0.0010
V	ND*	0.0002	0.0006
Cr	ND*	0.0001	0.0004
Mn	ND*	<0.0001	0.0002
Co	ND*	<0.0001	<0.0001
Fe	0.0042	<0.0001	0.0002
Ni	ND*	<0.0001	<0.0001
Cu	(0.0001)*	<0.0001	<0.0001
Zn	ND*	<0.0001	<0.0001
Ga	ND*	<0.0001	<0.0001
Ge	ND*	<0.0001	<0.0001
As	ND*	<0.0001	<0.0001
Sr	<0.0001	<0.0001	<0.0001
Zr	ND*	<0.0001	<0.0001
Mo	(<0.0001)*	<0.0001	<0.0001
Cd	(<0.0001)*	<0.0001	<0.0001
Sn	ND*	<0.0001	<0.0001
Sb	ND*	<0.0001	<0.0001
Ba	(0.0009)*	0.0001	0.0004
Ta	ND*	<0.0001	0.0001
W	ND*	<0.0001	<0.0001
Pb	ND*	<0.0001	<0.0001
Bi	ND*	<0.0001	<0.0001
C	98.1	Balance	Balance

Sample ID: Residual Oil			Units: %
Component	NEX DE VS Value	Stat. Error	Est. LLD**
Na	ND*	0.088	0.265
Mg	ND*	0.008	0.024
Al	0.2	0.002	0.003
Si	0.131	0.001	0.001
P	0.0452	0.0005	0.0014
S	1.09	0.001	0.001
Cl	0.0084	0.0001	0.0004
K	ND*	0.003	0.008
Ca	ND*	0.001	0.004
Ti	ND*	0.0004	0.0011
V	ND*	0.0002	0.0006
Cr	ND*	0.0001	0.0004
Mn	ND*	<0.0001	0.0002
Fe	0.0015	<0.0001	0.0002
Co	ND*	<0.0001	0.0001
Ni	ND*	<0.0001	<0.0001
Cu	ND*	<0.0001	<0.0001
Zn	ND*	<0.0001	<0.0001
Ga	ND*	<0.0001	<0.0001
Ge	ND*	<0.0001	<0.0001
As	ND*	<0.0001	<0.0001
Sr	ND*	<0.0001	<0.0001
Zr	ND*	<0.0001	<0.0001
Mo	0.0002	<0.0001	<0.0001
Cd	ND*	<0.0001	<0.0001
Sn	ND*	<0.0001	<0.0001
Sb	ND*	<0.0001	<0.0001
Ba	(0.0012)*	0.0002	0.0004
Ta	ND*	<0.0001	0.0001
W	ND*	<0.0001	<0.0001
Pb	ND*	<0.0001	<0.0001
Bi	ND*	<0.0001	<0.0001
Oil	98.5	0.0007	0.0009

* ND means the element was not detected. () * means above detection limit but below quantitation limit.

** Estimated LLD numbers are theoretical and based on current concentration levels and matrix composition

RPF-SQX SEMI-QUANT FP RESULTS

Sample ID: Oil Ash			Units: %
Component	NEX DE VS Value	Stat. Error	Est. LLD**
Na	ND*	0.0752	0.225
Mg	1.93	0.01	0.03
Al	23.9	0.01	0.03
Si	21.3	0.01	0.02
P	0.408	0.001	0.002
S	0.182	0.0005	0.001
Cl	0.0374	0.0004	0.001
K	(0.0438)*	0.0042	0.0117
Ca	0.648	0.005	0.006
Ti	0.250	0.002	0.002
V	0.338	0.002	0.003
Cr	0.0824	0.0008	0.0018
Mn	0.0236	0.0003	0.0007
Fe	2.42	0.002	0.0002
Co	0.0066	0.0004	0.0012
Ni	0.129	0.0003	0.0001
Cu	0.0096	<0.0001	0.0001
Zn	0.0416	0.0001	<0.0001
Ga	0.0075	<0.0001	<0.0001
Ge	ND*	<0.0001	<0.0001
As	0.0011	<0.0001	<0.0001
Rb	0.0004	<0.0001	<0.0001
Sr	0.0146	0.0002	0.0013
Y	0.0028	<0.0001	<0.0001

Sample ID: Oil Ash			Units: %
Component	NEX DE VS Value	Stat. Error	Est. LLD**
Zr	0.0051	<0.0001	0.0001
Mo	0.0026	<0.0001	<0.0001
Sn	ND*	<0.0001	<0.0001
Sb	0.0052	<0.0001	<0.0001
Ba	0.0142	0.0002	0.0004
La	0.155	0.0006	0.0003
Ce	0.155	0.0007	0.0004
Cd	ND*	<0.0001	<0.0001
Ta	ND*	0.0002	0.0007
W	ND*	0.0001	0.0003
Pb	0.0042	<0.0001	0.0001
Bi	(0.0003)*	<0.0001	<0.0001
O	47.9	Balance	Balance

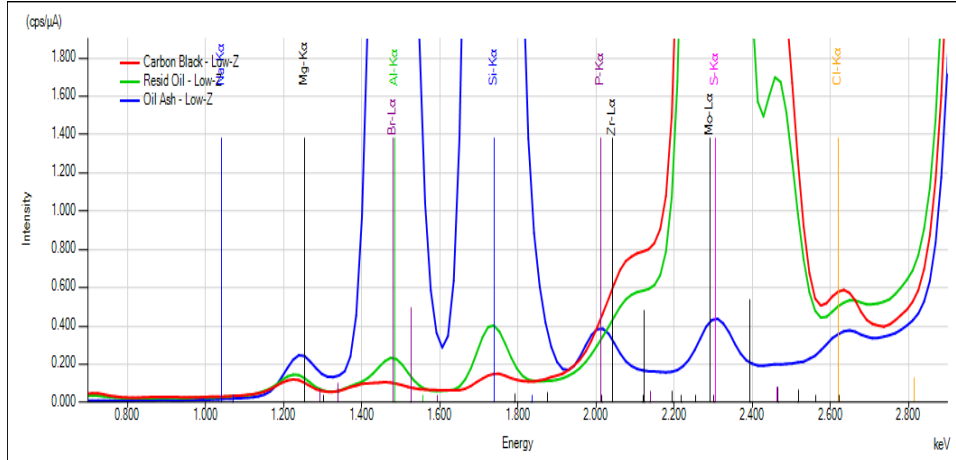
* ND means the element was not detected. ()* means above detection limit but below quantitation limit.

** Estimated LLD numbers are theoretical and based on current concentration levels and matrix composition

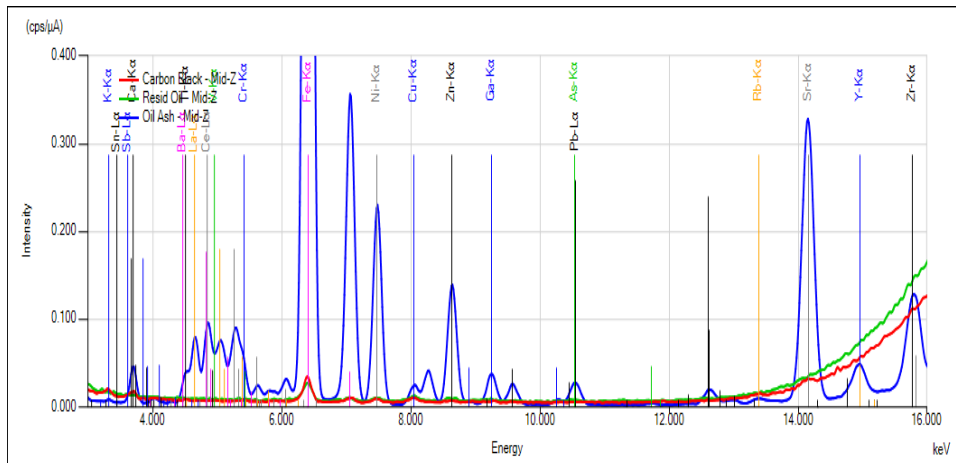
QUALITATIVE ANALYSIS

Spectral analysis of each of the unknown samples revealed clean isolation of the respective peaks of interest.

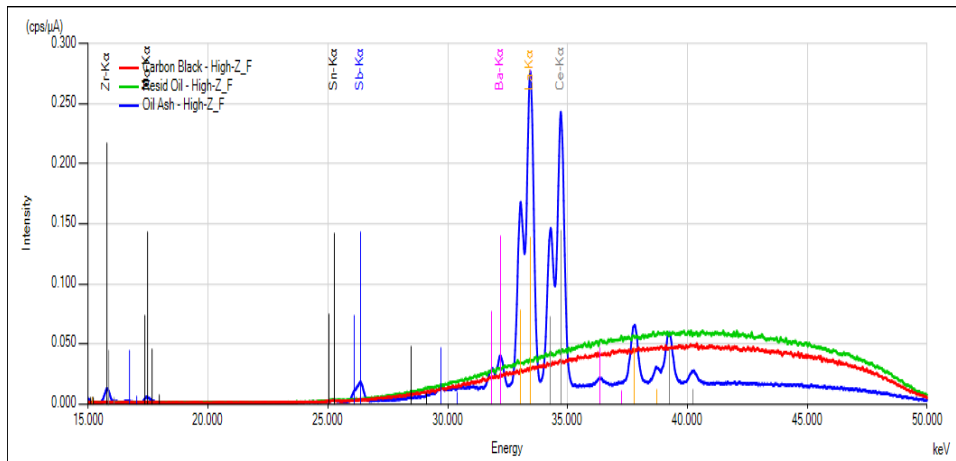
Low-Z Condition (Zoom View)



Mid-Z Condition



High-Z Condition



DISCUSSION

The Rigaku RPF-SQX FP software automatically deconvolutes spectral peaks and models the sample matrix and X-ray absorption/enhancement effects using fundamental XRF equations. The versatile RPF-SQX software is simple to use and allows for semi-quantitative analysis without the need for assayed reference material. Analytical accuracy can be further optimized through the creation of a matrix-matched Matching Library where the users registers at least one assayed standard into the library as a reference.

CONCLUSION

Innovative design using 60kV tube and high resolution detector 500,000+ cps detector throughout provides sensitivity for trace measurements and low detection limits. Simple, intuitive software flow makes the NEX DE an ideal EDXRF tool for the elemental identification (Na-U), screening and characterization of raw materials and final products. Simple to operate, the NEX DE is used by the non-technical operator and researcher alike.