

SCOPE

This Application Note shows the analysis three rock Reference Materials from Rocklabs, Ltd., using Rigaku RPF-SQX fundamental parameters. Performance is shown for both hydraulically pressed pellets and loose powders.

BACKGROUND

Elemental analysis is important in the analysis of rocks and ores, from screening at the quarry site or mine, throughout the processing areas to final analysis. Majors and minors are important during processing, to ensure proper extraction and process control. Trace elemental analysis, especially of precious metals, is also extremely important to ensure the value of the processing is optimized. Along the entire processing line, a fast and simple technique is required *without the need for large suites of assayed calibration standards to match each matrix type*. To meet these industry challenges, Rigaku offers the NEX CG EDXRF elemental analyzer. The NEX CG, using indirect excitation and polarization, offers the non-technical operator a powerful and simple tool for the measurement of the elements sodium through uranium in rocks and ores, while retaining the power and versatility for the XRF expert involved in rock and ore analysis.



INSTRUMENTATION

Model:	Rigaku NEX CG
X-ray tube:	50 W Pd-anode
Detector:	High performance SDD
Sample Type:	Pressed Pellets & Loose Powders
Environment:	Helium Purge
Optional:	Vacuum System
Standard:	15-position Sample Tray (32mm)
Optional:	10-position Sample Tray (40mm)
Optional:	9-position Sample Spinner Tray
Analysis Time:	500 sec



SAMPLES

The samples used for this report were obtained from Rocklabs, Ltd., and are certified for gold content with uncertified reference values for the other elements. The samples used were Ox P61, Ox L63 and Ox i67.

SAMPLE PREPARATION

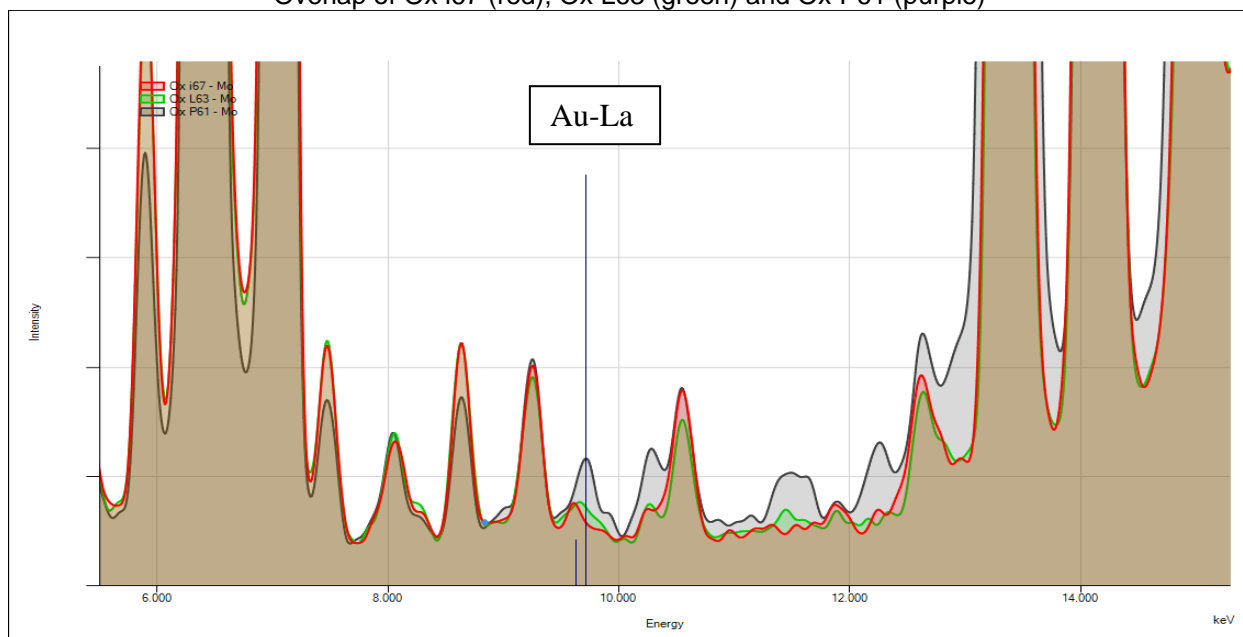
Samples were supplied as finely ground and dried powder. A fine powder (<200 mesh) minimizes mineralogical effects and reduces extraneous scattering of X-rays. For the analysis of powdered samples, simply place 5g of sample in an XRF sample and tap-pack the loose powder. For optimum analysis, hydraulically pressed pellets were also prepared by pressing 5g of powder at 20 tons pressure for 20-30 seconds.

CALIBRATION and MEASUREMENT

Two calibration methods were built using the Rigaku RPF-SQX fundamental parameters (FP) template for powders and pressed pellets. RPF-SQX is an advanced FP program that automatically deconvolutes spectral peaks and models the sample matrix using fundamental XRF equations. Each basic template was optimized by enabling the Rigaku scattering FP selection. Rigaku scattering FP optimizes the matrix modeling by using an advanced approach to estimating the non-measured elements (H through F) and using the natural scatter peaks generated by the X-ray source and the sample matrix. This allows for a semi-quantitative measurement of elemental concentrations without the need for known assayed calibration standards. Each method was then further optimized by making a Matching Library based on two of the Rocklabs samples (Ox P61 and Ox i67). Matching Library samples are chosen to model the specific matrix being measured and bracket key elements high and low. The given concentration values were registered with their associated X-ray intensities. The Matching Library is used in conjunction with the standard FP library in the modeling of the rock matrix and calculation of concentration results.

QUALITATIVE SPECTRA - GOLD

Overlap of Ox i67 (red), Ox L63 (green) and Ox P61 (purple)



To demonstrate the matrix modeling improvements using the Matching Library, sample Ox L63 was measured against the RPF-SQX with Matching Library method.

RPF-SQX RESULTS – PRESSED PELLETS

Ox L63

Component	Units	Standard Value	RPF-SQX Result	Statistical Std Dev	% Relative Difference
Au	ug/g	5.865	16.31	0.91	178%
Na ₂ O	%	6.85	7.50	0.04	9%
MgO	%	2.46	1.48	0.01	40%
Al ₂ O ₃	%	17.57	16.01	0.02	9%
SiO ₂	%	62.28	63.55	0.03	2%
P ₂ O ₅	%	0.23	0.26	<0.01	13%
K ₂ O	%	3.36	3.29	0.01	2%
CaO	%	2.40	2.32	0.01	3%
TiO ₂	%	0.70	0.62	<0.01	11%
MnO	%	0.05	0.05	<0.01	0%
Fe ₂ O ₃	%	3.74	3.26	<0.01	13%
Rb ₂ O ₃	%	---	0.02	<0.01	---
SrO	%	---	0.03	<0.01	---

RPF-SQX *with* MATCHING LIBRARY RESULTS – PRESSED PELLETS

Ox L63

Component	Units	Standard Value	RPF-SQX Result	Statistical Std Dev	% Relative Difference
Au	ug/g	5.865	5.38	0.86	8%
Na ₂ O	%	6.85	7.43	0.05	8%
MgO	%	2.46	2.74	0.02	11%
Al ₂ O ₃	%	17.57	16.82	0.03	4%
SiO ₂	%	62.28	59.64	0.03	4%
P ₂ O ₅	%	0.23	0.23	<0.01	0%
K ₂ O	%	3.36	3.08	0.01	8%
CaO	%	2.40	2.43	0.01	1%
TiO ₂	%	0.70	0.72	<0.01	3%
MnO	%	0.05	0.06	<0.01	20%
Fe ₂ O ₃	%	3.74	3.77	<0.01	1%
Rb ₂ O ₃	%	---	0.02	<0.01	---
SrO	%	---	0.03	<0.01	---

RPF-SQX RESULTS – LOOSE POWDER

Ox L63

Component	Units	Standard Value	RPF-SQX Result	Statistical Std Dev	% Relative Difference
Au	ug/g	5.865	16.05	0.95	274%
Na ₂ O	%	6.85	7.56	0.12	10%
MgO	%	2.46	1.48	0.02	40%
Al ₂ O ₃	%	17.57	15.63	0.03	11%
SiO ₂	%	62.28	59.64	0.04	4%
P ₂ O ₅	%	0.23	0.26	<0.01	13%
K ₂ O	%	3.36	3.27	0.01	3%
CaO	%	2.40	2.26	0.01	6%
TiO ₂	%	0.70	0.62	<0.01	11%
MnO	%	0.05	0.05	<0.01	0%
Fe ₂ O ₃	%	3.74	3.23	0.01	14%
Rb ₂ O ₃	%	---	0.02	<0.01	---
SrO	%	---	0.03	<0.01	---

RPF-SQX *with* MATCHING LIBRARY RESULTS – LOOSE POWDER

Ox L63

Component	Units	Standard Value	RPF-SQX Result	Statistical Std Dev	% Relative Difference
Au	ug/g	5.865	7.51	0.82	28%
Na ₂ O	%	6.85	6.76	0.11	1%
MgO	%	2.46	2.87	0.04	17%
Al ₂ O ₃	%	17.57	16.64	0.04	5%
SiO ₂	%	62.28	58.72	0.05	6%
P ₂ O ₅	%	0.23	0.23	<0.01	0%
K ₂ O	%	3.36	2.97	0.01	10%
CaO	%	2.40	2.37	0.01	1%
TiO ₂	%	0.70	0.70	<0.01	0%
MnO	%	0.05	0.05	<0.01	0%
Fe ₂ O ₃	%	3.74	3.64	0.01	3%
Rb ₂ O ₃	%	---	0.02	<0.01	---
SrO	%	---	0.03	<0.01	---

ESTIMATED LLD for Au – LOWER LIMIT OF DETECTION

The LLD is estimated using calculations of the peak intensities, background intensities, scatter peaks and counting statistics. LLDs are dependent on many factors, including matrix type, concentrations of other elements present, use of helium, air or vacuum, sample preparation and analysis counting times. The LLD for gold based on calculation is approximately 2 ppm. In field use the factors listed above may result in a higher LLD for gold of perhaps 3-5 ppm, depending on rock composition.

DISCUSSION

The results indicate optimum measurement performance using FP is achieved analyzing samples in pressed pellet form. Grinding samples into a powder and pressing pellets minimizes mineralogical effects due to grain size inconsistencies and inhomogeneity of oxide distributions. The pressed pellet method also compacts the sample into a smaller volume. This allows for improved measurement of the light elements Na – P while minimizing statistical errors and improving detection limits. This sample preparation method is ideal for process quality control and quality assurance. Analyzing samples as loose powders introduces error due to X-ray scatter and inconsistencies in path lengths X-rays must travel to escape the matrix and reach the detector. While measurement variability is larger using the loose powder method, such measurements can be ideal for screening and initial characterization of rocks and ores.

RPF-SQX is an excellent tool for semi-quantitative analysis, and characterizing the exact sample matrix with a Matching Library greatly improves accuracy even further. A Matching Library is easily registered by the operator by analyzing one or more assayed samples of the particular matrix of interest.

Analysis is carried out under helium flush in order to purge air from the X-ray path. This technique allows for improved sensitivity of the measurement of the light elements Na – P, which have relatively weak X-rays. If helium is not available or due avoid the cost of helium, the same performance can be achieved using the optional vacuum system available with the NEX CG. The vacuum system also ensures no down time if helium is temporarily unavailable.

CONCLUSION

The NEX CG offers the analyst a simple yet powerful and versatile system for indentifying and quantifying elemental composition. The results of this study indicate the Rigaku NEX CG EDXRF analyzer with indirect excitation and polarization is an excellent tool for the screening and quantification of major, minor and trace elements in rocks. Use of the Rigaku RPF-SQX fundamental parameters program in conjunction with the Matching Library yields excellent performance without the need for large suites of calibration to characterize each matrix, and is ideally suited for uses throughout quarrying and processing of rocks, from initial screening and all throughout the processing line.

