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EDXRF1241 - Analysis of gemstones

Scope

The analysis of gemstones is demonstrated, using low power 50W EDXRF with indirect excitation.

Background



Elemental measurement is important in gemology for identification, classification and characterization of both natural and synthetic gemstones. Minor and trace levels of metals, especially the transition metals, as well as alkali elements and alkaline earth elements (such as Mg, K, Ca, Sr and Rb) help to establish the geographic region and environmental conditions during the formation of a gemstone. Other elements like Au and Pb can be used to indicate processing requirements of synthetics.

EDXRF (Energy dispersive X-ray Fluorescence) is a simple, non-contact, non-destructive analysis technique that is ideal for use in precious materials testing. The technique can be used for qualitative screening or gemstone characterization by elemental quantification. EDXRF is useful in quantifying the elements that occur in many gem materials, as well as other elements that are evidence of certain treatment processes. The benefit of using low power 50W EDXRF is that the X-ray source will not damage the gems. Secondary targets and polarization also make the analysis simple by eliminating most if not all of the extraneous background scatter X-rays.



Model: NEX CG

Rigaku RPF-SQX Fundamental Parameters (FP)

A Fundamental Parameters (FP) method was developed from Rigaku's RPF-SQX Metals Template. The RPF-SQX method uses an advanced FP program that automatically deconvolutes spectral peaks and models the sample matrix using fundamental XRF equations. This provides an estimate of the percentage of the sample that cannot be measured and yields more accurate analytical results for the remaining elements that can be measured, affording a semi-quantitative measurement of elemental concentrations without the need for a large suite of known assayed calibration standards.

In addition, a matrix-specific Matching Library can be created using one assayed sample of each gemstone to further optimize analytical results. The Matching Library is easy to create and is employed in conjunction with the standard FP library to optimize model of each matrix so as to improve the calculation of concentration results.

The results shown here use standardless analysis and indicate excellent performance without the use of a Matching Library.

RPF-SQX Results

SW Pearl		
Units: Mass%		
Component	NEX CG value	Stat. error
Mg	0.218	0.0048
Al	ND**	--
Si	0.116	0.0013
P	0.0152	0.0003
S	0.153	0.0004
Cl	0.126	0.0003
K	0.0739	0.0020
CaCO ₃	99.06	--

Ti	ND	--
V	ND	--
Cr	ND	--
Mn	0.0092	0.0006
Fe	0.0081	0.0004
Co	ND	--
Ni	ND	--
Cu	0.0231	0.0004
Zn	0.0081	0.0002
Ga	ND	--
Br	0.0012	0.0001
Rb	ND	--
Sr	0.1817	0.0004
Mo	ND	--
Yb	ND	--
Ir	ND	--
Au	0.0016	0.0002
Hg	0.0012	0.0001
Tl	ND	--
Pb	ND	--

Spinel		
Units: Mass%		
Component	NEX CG value	Stat. error
Mg	5.69	0.0303
Al ₂ O ₄ †	94.21	--
Si	ND	--
P	ND	--
S	ND	--
Cl	0.0195	0.0003
K	0.0079	0.0011
Ca	0.0040	0.0007
Ti	0.0282	0.0007

V	0.0159	0.0005
Cr	ND	--
Mn	ND	--
Fe	0.0066	0.0004
Co	0.0135	0.0004
Ni	ND	--
Cu	0.0051	0.0002
Zn	0.0015	0.0001
Ga	ND	--
Br	ND	--
Rb	ND	--
Sr	ND	--
Mo	ND	--
Yb	ND	--
Ir	ND	--
Au	ND	--
Hg	0.0002	0.0001
Tl	ND	--
Pb	ND	--

Sapphire		
Units: Mass%		
Component	NEX CG value	Stat. error
Mg	ND	--
Al ₂ O ₃	98.90	--
Si	ND	--
P	ND	--
S	0.0152	0.0003
Cl	0.117	0.0004
K	0.0269	0.0012
Ca	0.0147	0.0007
Ti	0.0090	0.0003
V	0.0017	0.0002

Cr	ND	--
Mn	ND	--
Fe	0.823	0.0016
Co	0.0090	0.0004
Ni	ND	--
Cu	0.0278	0.0003
Zn	0.0070	0.0001
Ga	0.0157	0.0002
Br	ND	--
Rb	ND	--
Sr	ND	--
Mo	ND	--
Yb	ND	--
Ir	0.0034	0.0002
Au	0.0022	0.0001
Hg	ND	--
Tl	0.0019	0.0001
Pb	ND	--

Emerald		
Units: Mass%		
Component	NEX CG value	Stat. error
Be ₃ Al ₂	14.75	0.0191
Mg	ND	--
SiO ₃	84.16	--
P	0.0337	0.0005
S	0.0060	0.0003
Cl	0.0949	0.0004
K	0.0328	0.0015
Ca	0.0158	0.0011
Ti	0.0007	0.0003
V	0.0558	0.0007
Cr	0.707	0.0018

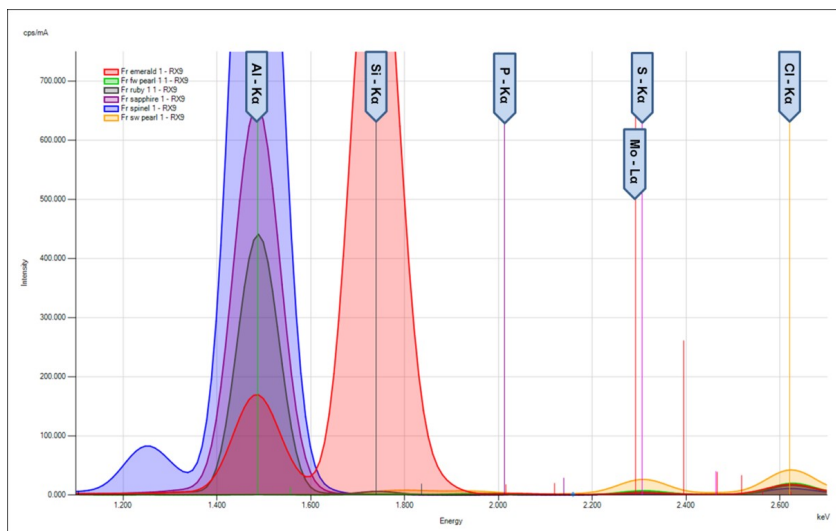
Mn	0.0134	0.0010
Fe	0.0185	0.0004
Co	ND	--
Ni	0.0018	0.0001
Cu	0.0182	0.0002
Zn	0.0046	0.0001
Ga	0.0015	0.0001
Br	ND	--
Rb	0.0012	<0.0001
Sr	ND	--
Mo	0.0617	0.0017
Yb	0.0041	0.0004
Ir	ND	--
Au	0.0148	0.0002
Hg	ND	--
Tl	ND	--
Pb	0.0052	0.0001

** ND means the component was not detected.

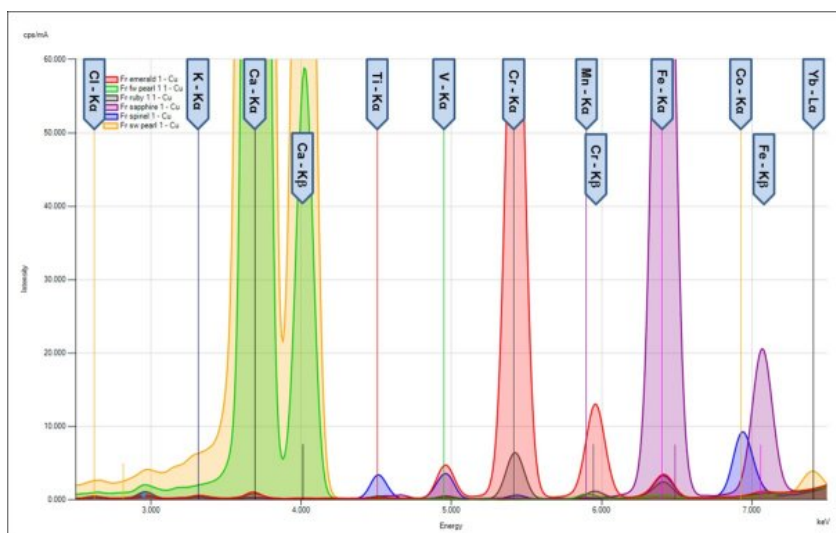
† The main component of Spinel is MgAl_2O_4 ; alternately, MgO and Al_2O_3 could be entered as components

Qualitative analysis

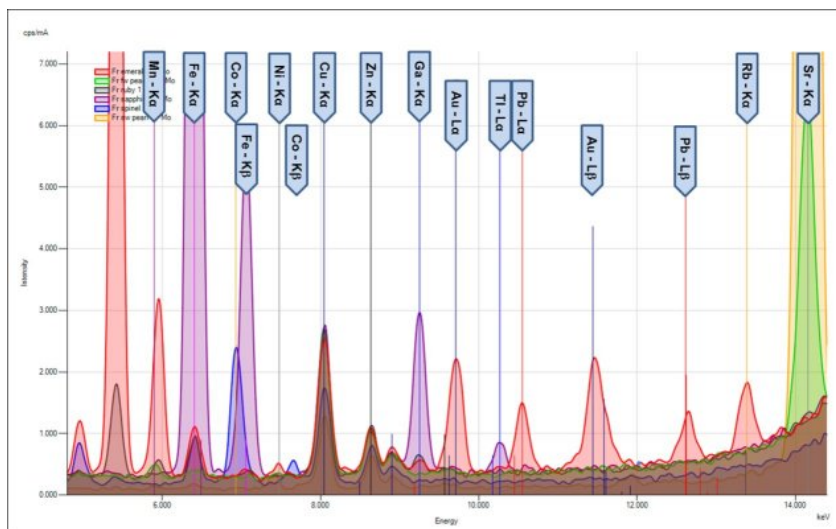
As an example of qualitative analysis, the gemstone spectra are overlaid and provided below.



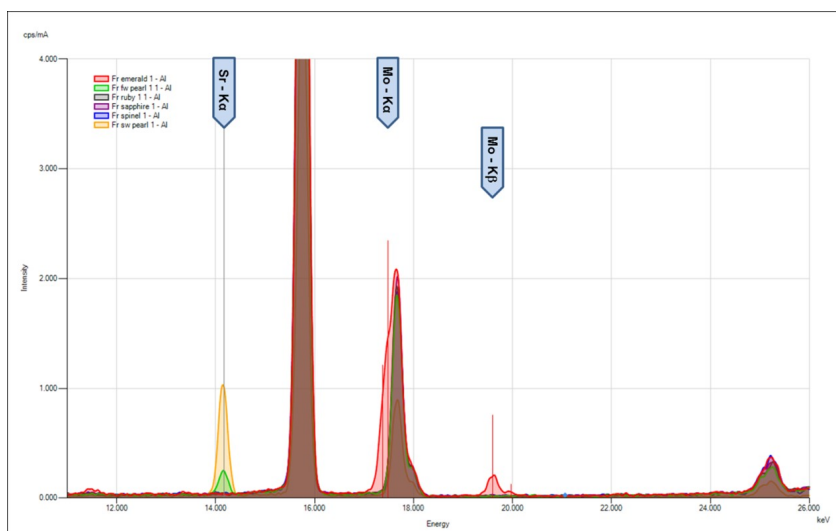
RX9 (HOPG polarizer) secondary target



Cu secondary target



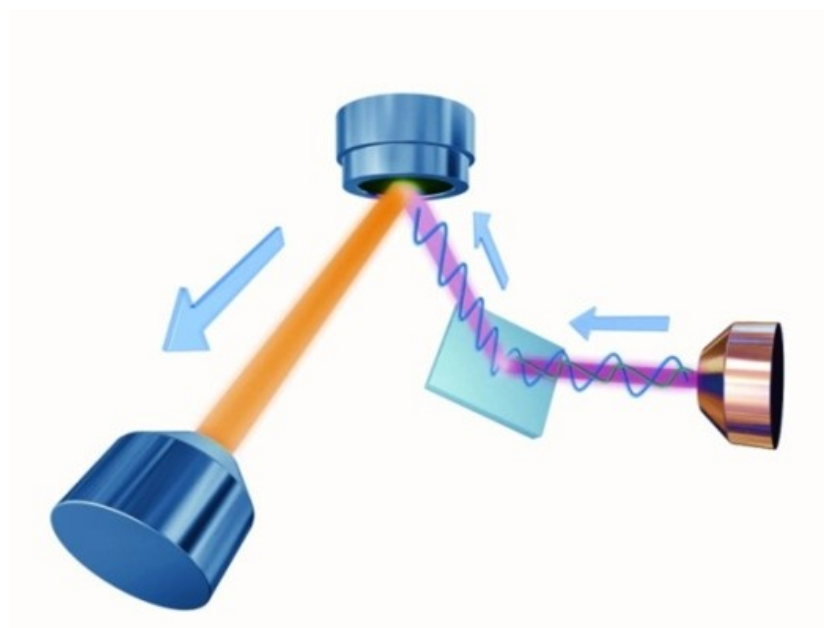
Mo secondary target



Al secondary target

Discussion

The Rigaku NEX CG energy dispersive X-ray fluorescence (EDXRF) spectrometer combines indirect excitation with secondary targets, polarization targets and a high performance SDD to give the operator a powerful and versatile analysis tool with a very simple to operate software interface. In contrast to conventional EDXRF systems, indirect excitation removes virtually all the background and thus affords spectra with a very high characteristic signal-to-noise ratio. This allows for much lower detection limits and a higher degree of accuracy.



Secondary target schematic

The spectra and quantitative results shown above indicate NEX CG can be used as an important tool in the characterization of gemstones. When a gem is known, the analysis method can be optimized by defining the gem's major component as the balance of the matrix. Example of this is Al_2O_3 as the major component of sapphire, the CaCO_3 of pearl and the Be_3Al_2 of emerald. Information about synthetics and processing can be determined in part by the presence of trace Au and/or Pb, indications of the processing crucible used. Other identifiers may lie in certain elemental ratios, such as the Sr/Mn ratio in fresh water and sea water pearls.

Pearl Type	Sr/Mn Ratio
Fresh Water Pearl 1	0.658
Fresh Water Pearl 2	0.404
Sea Water Pearl	19.75

Conclusion

The Rigaku NEX CG using the RPF-SQX Fundamental Parameters method yields excellent performance for the elemental analysis of gemstones. Indirect excitation and polarization give the gemologist an excellent low background tool, and low 50W power ensures no damage to the gemstone during analysis. The use of RPF-SQX eliminates the need for calibration standards, and if desired quantification can be optimized with Matching Libraries based on one assayed sample of each gemstone. These features and more make the NEX CG an ideal EDXRF tool for the elemental identification and characterization of gemstones and similar materials.