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# XRF1036 - Semiquant analysis by 'SQX Scatter FP' method

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## Introduction

Semi-quantitative analysis by the Fundamental Parameter (FP) method is a unique and powerful method for elemental screening of materials. In the FP calculation, the program requires principally the information of all elements included in a specimen for analysis. This requirement is not convenient when the specimen contains large amount of ultra-light elements from hydrogen to oxygen, which are difficult to determine accurately or non-measurable elements on X-ray spectrometry.

In 2005, Rigaku developed a new method to estimate an average atomic number for non-measured elements from hydrogen to oxygen in a specimen using the intensities of scattered X-rays and applied the estimation to the semi-quantitative FP calculation (SQX) as a balance component.

The successfully advanced SQX method using scattering intensities, the SQX Scatter FP method, becomes available for oxide powders on the newly developed benchtop XRF spectrometer, Supermini200.

This application note introduces some applications by the SQX Scatter FP method.

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## Instrument

The Supermini200 is an improved sequential wavelength dispersive X-ray fluorescence (WDXRF) spectrometer. The spectrometer is a unique benchtop system designed to minimize installation utilities such as cooling water, power supply and installation space etc. It is also equipped with an air-cooled 200W X-ray tube and up to three analyzing crystals, with which elements from oxygen to uranium can be analyzed. The operation software provides users with high flexibility and availability for various applications in easy-to-use operation. Standardless analysis program "SQX" is user friendly and can quickly determine which elements are contained and how much of the element is contained in an unknown sample without any selection and preparation of suitable reference materials.

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## Sample preparation

Two powder samples were prepared to demonstrate by pressed powder method.

The well-dried (2 hours at 105°C) samples were ground with a chromium steel container and then pressed under the pressure of 100 kN using a sample support ring made of PVC.

In order to maximize accuracy, no binder was used, so to prevent contamination in the instrument by powder particles, the pressed pellets were wrapped in 4 µm Prolene® (Chemplex 401) film.

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## Application 1: Dehydrated cake – sludge residue

Dehydrated cake is a residue remaining after dehydration of sludge. High moisture still remains in the residue and the residue also contains a variety of organic matter. The SQX Scatter FP method was applied to the dehydrated cake. Table 1 shows that the results of the SQX Scatter FP method are in good agreement with chemical values. These results indicate that the estimation of average atomic number of balance component is accurately and reliably performed by the FP calculation.

**Table 1:** SQX results of dehydrated cake (unit : mass%)

Method	Na	Mg	Al	Si	P	S	Cl	K	Ca	Ti	Cr
Chemical Value	0.90	1.22	7.26	16.7	0.12	-	-	1.14	3.90	0.40	-
SQX	0.69	1.28	8.20	15.9	0.12	0.19	0.06	1.12	3.69	0.38	0.02
Method	Mn	Fe	Cu	Zn	Br	Rb	Sr	Y	Zr	Pb	Balance
Chemical Value	0.10	4.87	0.012	0.086	0.003	0.006	0.024	0.002	0.008	0.013	-
SQX	0.09	4.90	0.01	0.08	0.003	0.004	0.026	0.003	0.008	0.015	63.1

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## Application 2: Deep sea sediment

In general, soils and marine sediments have complex matrices and the contents of hydrogen to oxygen show very wide variation in each sample. The SQX Scatter FP method is appropriate to determine concentrations of the elements in these materials.

The deep sea sediment, JMS-2, is a certified reference material supplied by the Geological Survey of Japan (GSJ). The analysis results are shown in the Table 2.

**Table 2:** SQX results of deep sea sediment (JMS-2) (unit : mass%)

Method	Na	Mg	Al	Si	P	S	Cl	K	Ca	Ti	V	Cr
Certified Value	4.30	1.95	7.50	19.5	0.55	0.29	4.05	2.24	3.34	0.84	0.018	0.008
SQX	4.29	1.87	7.34	17.3	0.48	0.30	4.32	2.16	3.15	0.78	0.018	0.015
Method	Mn	Fe	Co	Ni	Cu	Zn	As	Rb	Sr	Y	Zr	Balance
Certified Value	1.75	7.67	0.023	0.031	0.045	0.017	0.004	0.007	0.045	0.025	0.022	-
SQX	1.87	7.45	0.021	0.031	0.046	0.020	0.005	0.007	0.050	0.026	0.023	48.0

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## Conclusions

The SQX Scatter FP method was performed for the screening of materials difficult to analyze by conventional SQX. The successful results demonstrate an availability of this advanced method. The method can widely expand the application field of the semi-quantitative analysis.

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## What is SQX?

SQX is a semi-quantitative analysis program to obtain the concentrations by utilizing theoretical intensity calculation of X-ray intensities using fundamental parameters and internal sensitivity library without any standards. The calculation is performed by using the results of sequential scan measurements from fluorine to uranium. The program is integrated with theoretical overlapping correction function. Therefore, SQX analysis is highly optimized for screening analysis for unknown sample with multiple elements.

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## Related products



### Supermini200

Benchtop tube below sequential WDXRF spectrometer analyzes O through U in solids, liquids and powders