

## Standardless XRF analysis for LOI-rich rock samples by scatter fundamental parameter method

AL MARTIN, HISASHI INOUE<sup>1</sup>, YAUJIRO YAMADA,  
HISAYUKI KOHNO<sup>2</sup>

<sup>1</sup>Rigaku/MSC, Inc.

<sup>2</sup>Rigaku Industrial Corporation

X-ray fluorescence (XRF) spectrometry has been used for bulk rock analysis instead of AA and ICP due to easy sample preparation and the high precision capabilities. In addition, XRF is used as a screening tool prior to other analysis methods such as ICP-MS. In some cases it is difficult to prepare calibration samples because of availability. In this case a standardless analysis would be performed using a Fundamental Parameter (FP) routine. However, since the analysis results are usually calculated as a total concentration of 100% (normalized), components that are not analyzed, such as loss on ignition (LOI), would increase the analysis error. Therefore, it is necessary to obtain the values of these missing components for an accurate analysis.

In order to accurately analyze LOI-rich samples without the pre-determination of an LOI content, scatter lines derived from the characteristic lines of the x-ray tube have been used. This estimation of the LOI content in standardless analysis has been combined with a fundamental parameter (FP) method.

Using the Scatter FP Method in standardless analysis by XRF, it is possible to carry out accurate screening even for LOI-rich samples such as dolomite, limestone, clay and soil.

Table

NIST 88b Dolomitic Limestone		
Analysis result by non-standard FP method for dolomite (mass%)		
Element	Std. Value	Scatter FP
Mg	12.7	12.0
Al	0.18	0.34
Si	0.53	0.64
K	0.09	0.10
Ca	21.4	22
Mn	0.012	0.0139
Fe	0.19	0.21

## Total sample evaporation of ng-sized Li samples using the Finnigan TRITON thermal ionization mass spectrometer in static mode

DIETMAR TUTTAS, CLAUDIA BOUMAN AND  
JOHANNES B. SCHWIETERS

Thermo Electron Bremen GmbH, Germany  
(dietmar.tuttas@thermo.com)

Lithium isotope analysis using TIMS is very challenging and time-consuming. Although the TIMS technique is extremely sensitive, sample preparation and sample heat up are critical to the stability of the mass bias during the run and even more with respect to the attainable external reproducibility.

A completely different approach involves the total evaporation technique, in which the sample is completely evaporated during the run and the isotopic ratio is taken from the integral of both intensities accumulated during the whole run. In theory this should avoid problems with time-dependent mass fractionation. Previous TIMS instruments had a limited parallel mass range so that the measurement of Li isotopes had to be done by single collector peak jumping measurements. This was one of the reasons why the total evaporation method gave limited reproducibility. However, the multicollector of the Finnigan TRITON instrument has up to 17% mass range, enabling the parallel detection of both Li isotopes. In this study we will show the potential of the total evaporation method for Li isotope ratio measurements with respect to sample throughput and achievable external precision.