# Application Package for X-ray fluorescence analysis —Simple, fast calibration for quantitative analysis—



# 1. Introduction

X-ray fluorescence (XRF) analysis is widely used in a number of fields, including quality control and research & development, due to its high degree of accuracy and extremely simple sample preparation. Quantitative analysis with XRF spectrometry requires reference materials in order to generate calibration curves.

The rapidly improving performance of personal computers has opened the door to sophisticated data processing techniques like SQX Scatter FP (Fundamental Parameter) method, improving the accuracy of semi-quantitative analysis, where reference material is not used for quantitative calculation by the FP method. The even more accurate quantitative analysis method, however, requires the user to generate calibration curves using calibration standards made of the same material as the samples to be analyzed.

While quantitative analysis provides an unparalleled level of accuracy, correctly setting up the sample measurement conditions and creating the calibration curves requires expertise in XRF spectrometry and can be a time-consuming process. Rigaku has seen a growing need among XRF spectrometry users to be able to start up quantitative analyses quickly and easily.

The Application Package for XRF analysis introduced here is a product which is designed to enable even novices in XRF to start up quantitative analyses easily. The package provides the user with reference materials for calibration, drift correction samples for daily analysis and analysis parameters such as measurement conditions and correction coefficients for calibration.

# 2. The Application Package

The Application Package includes the following items:

- Calibration standards (reference material)
- Drift correction samples
- Installation CD, instruction manual

The user prepares the calibration samples as the same preparation method needs to be applied to both the calibration samples and analysis samples. Since the standard values for the calibration standards are stored in the software, the user does not need to type in the standard values.

By measuring the included drift correction samples prior to sample analyses, the user can correct for the sensitivity drift that can occur with long-term use of a spectrometer. Using the drift correction samples prior to sample analysis enables the user to employ a given set of calibration curves for an extended period of time without re-calibrating.

Parameters such as measurement conditions and correction coefficients for calibration are installed automatically from the installation CD. After installation, the instruction manual provides simple, step-by-step instructions on how to set up quantitative analyses. The inclusion of matrix correction coefficients and/or spectrum overlap correction coefficients for calibration makes it possible for less-experienced users to generate accurate calibration curves.

#### 3. Product line-up

The series of the Application Package currently

Series (material)	Analyte	Quant. method	Sample prep.
Low alloy steel, stainless steel	Si Mn P S Ni Cr Mo Cu Co V Nb	*Emp. cal.	Polishing
Special steel, Nickel alloy	Mn Si Cr Ni Co Mo W Nb Ti Al Fe P Cu Ta	FP	Polishing
Brass, Lead brass	Cu Fe Pb Sn Zn	Emp. cal.	Polishing
Refractory: Clay	SiO2 Al2O3 Fe2O3 TiO2 MnO CaO MgO etc.	Emp. cal.	Fusion bead
Refractory: Silica	SiO2 Al2O3 Fe2O3 TiO2 MnO CaO MgO etc.	Emp. cal.	Fusion bead
Refractory: High alumina	Al2O3 SiO2 Fe2O3 TiO2 MnO CaO MgO etc.	Emp. cal.	Fusion bead
Refractory: Magnesia	MgO SiO2 Al2O3 Fe2O3 CaO TiO2 etc.	Emp. cal.	Fusion bead
Refractory: Chrome-Magnesia	Cr2O3 MgO SiO2 Al2O3 Fe2O3 TiO2 etc.	Emp. cal.	Fusion bead
Refractory: Zircon-Zirconia	ZrO2 HfO2 SiO2 Al2O3 Fe2O3 TiO2 etc.	Emp. cal.	Fusion bead
Polymer (Polyethylene)	Na Mg Al Si P S Ca Ti Zn Fe Cr	Emp. cal.	Sand paper polishing
Petro-Pak (Fuel oil)	S	Emp. cal.	-

 Table 1.
 List of Application Package series

\* Empirical calibration

available are listed in Table 1. The packages are available for the ZSX Primus series (ZSX Primus and ZSX Primus II) and ZSX 100e (N/A for Polymer).

## (1) Alloy series

The alloy package series use commercially available alloy standards for calibration.

The "Special steel, Nickel alloy" package makes use of an FP method—sensitivity calibration curves created using this FP method are linear throughout a wide range of concentrations. In addition, a secondary calibration method is applied, where calibration curves have been established with plenty of the reference materials Rigaku owns and several reference materials commercially available are used for re-establishing the calibration curves. Thus, this package is suitable for use with a wide concentration range of Fe- and Ni-based alloys.

## (2) Refractories series

These series are designed to conform to JIS R 2216, "Methods for X-ray fluorescence spectrometric analysis of refractory products." The certified reference material series by The Technical Association of Refractories, Japan are used as calibration standards.

These package series employ a fusion bead sample preparation method, in which a powder sample is mixed with flux and the mixture is fused into a glass disk. This eliminates the impact of differences in particle size or mineral composition, improving the accuracy of the quantitative analysis.

### (3) Polymer (Polyethylene)

This series is designed for application with polymer plates. The samples are polished with sand paper prior to measurement.

With this package series, pellet samples are reshaped into plates before being analyzed.

#### (4) Petro-Pak (Application Package for fuel oil)

This package series is used for the analysis of sulfur in fuel oil (ASTM D2622, ISO 20884 and JIS K 2541-7).

Petro-Pak consists of a "Low Sulfur" series and

"High Sulfur" series. All required parameters for the materials contained in each series are provided. The user does not have to purchase calibration standards for all of the materials contained in each series. The user can buy the requisite standards for the material that the user intends to analyze. Table 2 shows a list of the materials in Petro-Pak.

Liquid samples such as fuel oil are poured into a liquid cell with analysis film and then measured. As an option, the Petro-Pak includes a starter kit containing liquid cells and analysis film, along with a number of disposable pipettes. The process is simple. After assembling the liquid cell and film, the user dispenses a fuel oil sample into the cell with a disposable pipette. Then, the sample is measured in a helium atmosphere. The catalog numbers of the liquid cell and the film from the starter kit are shown in the instruction manual, so the user can order the replacement cells and films as needed.

Currently, Petro-Pak is only available for the ZSX Primus (not available for ZSX PrimusII). Petro-Pak for Supermini and Mini-Z Sulfur Analyzer will be released soon.

#### 4. Summary

The Application Package makes it easier for users of all experience levels to add quantitative analysis to their process. Rigaku will continue to expand the Application Package library in order to meet the analytical needs of our users.

Table 2. List of the materials in Petro-Pak.

Low Sulfur series			
Sulfur concentration range: 0 - 1000 ppm			
Material:	Polysulfide (mineral oil matrix)		
	Diesel fuel		
	Gasoline		
	Kerosene		
High Sulfur series			
Sulfur concentration range: 0 - 5 mass%			
Material:	Polysulfide (mineral oil matrix)		
	Diesel fuel		
	Crude oil		
	Residual oil		