

Wavelength dispersive X-ray fluorescence spectrometer

ZSX Primus III NEXT

1. Introduction

X-ray fluorescence analysis is an elemental analysis method that can quickly and nondestructively analyze elements contained in a sample with simple sample preparation. Furthermore, it is widely used for process control and quality control analysis of steel, cement, refractories, and other materials owing to its excellent measurement reproducibility.

To meet the requirements of our customers, Rigaku's ZSX Primus series of sequential wavelength dispersive X-ray fluorescence spectrometers include the high-end tube-above ZSX Primus IV, high-end tube-below ZSX Primus IVi and ZSX Primus400 for XRF analysis of large samples.

Rigaku has developed a new spectrometer, ZSX Primus III NEXT equipped with tube above optics, as a successor to the ZSX Primus III+. The spectrometer has improved hardware and software performance and functions and an ability for enhanced support for daily analysis compared to the previous model.

The features of ZSX Primus III NEXT are as follows:

- High speed and high accuracy
 - High throughput achieved by optimizing the measurement sequence
 - Newly adopted digital multi-channel analyzer (D-MCA) enables measurement of high-intensity X-rays with high accuracy
- Enhanced support for configuration, measurement, and analysis
 - Enhanced routine analysis performed by improved "ZSX Guidance" software
 - Improved accuracy by fully utilizing D-MCA data
 - Enhanced features to support setting quantitative measurement conditions
 - Display function of standard deviation calculated from each quantitative analysis result
 - Scheduler function for more efficient routine analysis management
- Safety and security
 - Safe measurement of powder samples by tube-above system
 - Adopts a dust-proof housing for dusty environment
 - Automatic sample unloading if communication between spectrometer and PC is interrupted
 - Enhanced linkage between equipment condition

monitoring function and analysis results

- Improved Application Support Functions
 - Easy migration of application settings between user equipment
 - Faster service and application support with the data pack function

Here we describe the automatic background (BG) angle search function and the application support tool.

2. Automatic BG (Background) Angle Search Considering Interference Lines

In the "Optimize Measurement Conditions (MCs)" menu of ZSX Primus III NEXT, a new function, the Automatic BG angle search, has been added. The function works as follows: 1) extract samples from standard samples that contain elements that interfere (overlap and high-order) with the analysis line, 2) add extracted samples to measurement list for optimized MCs, 3) search BG angles that have no spectral interferences from other elements from superimposed profile results. Then, when an operator executes "BG Angle Search" function, candidates for BGs that minimize spectral interferences from other coexisting elements are displayed. As an example of this new functionality, the analysis of selenium (Se) in high alloys will be presented.

When creating a calibration curve, it is necessary to optimize MCs such as analyzing crystal, slit and PH window, as well as the peak angle and BG angle of the measurement line. In a conventional system, this operation is performed using only the measurement results of the sample with the maximum and minimum content of the analyzed element automatically extracted from the standard sample list (Fig. 1).

The standard sample set for high-alloy analysis includes samples containing tungsten (W), tantalum (Ta), and lead (Pb) as coexisting elements. When a sample containing many of these elements is measured and superimposed on the data in Fig. 1, spectra derived from the coexisting elements in the standard sample are found to overlap at positions that were thought to be appropriate BG positions (Fig. 2). With this new function, more appropriate BG positions can be automatically set by monitoring not only the conventional content component information but also interference lines.

The "BG angle search" function displays candidate BG angle positions that minimize the influence of

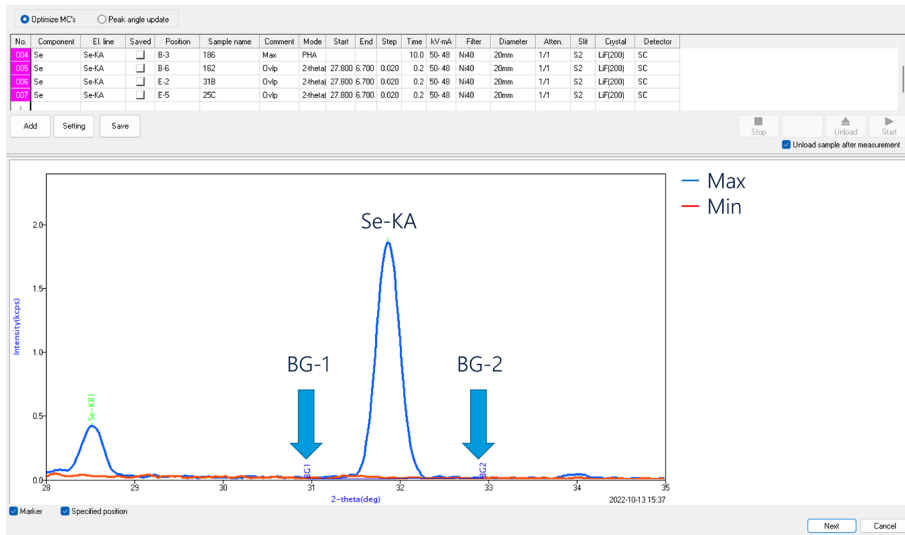


Fig. 1. BG position in a conventional system.

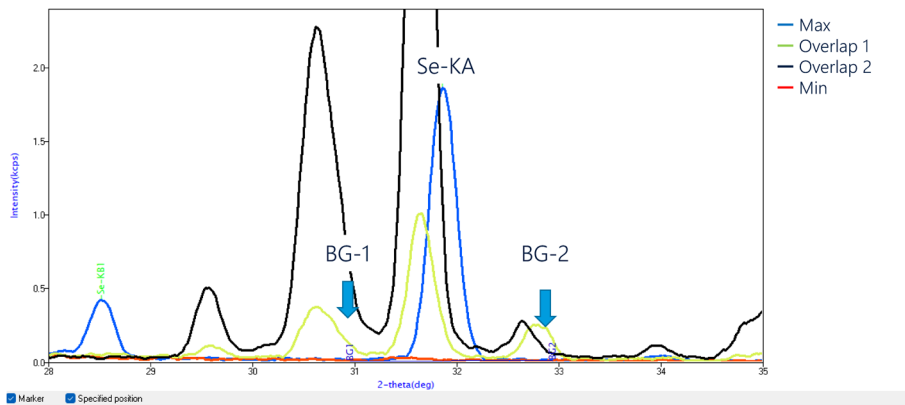


Fig. 2. Overlapped spectra of standard samples with and without interference lines.

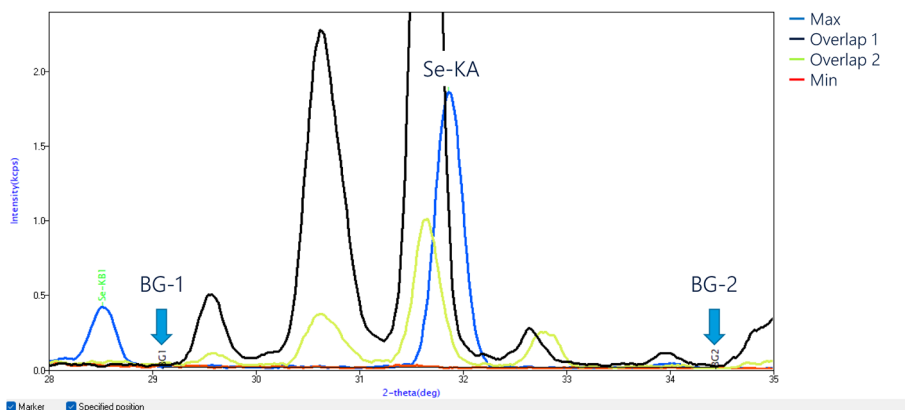


Fig. 3. BG position set by BG angle search.

interference lines after checking the reference spectrum of analysis lines, which is automatically executed after the measurement is completed (Fig. 3). Figure 4 shows the results of a comparison of a calibration curve created using this function with a calibration curve created using a conventional method. Figure 4 shows that this function facilitates appropriate BG settings, improves the efficiency of calibration curve creation, and supports

the creation of accurate calibrations.

3. Application Sharing Support 3.1 Application import/export function

The application configuration (quantitative/quantitative) import/export function first released for ZSX Primus IVi is also available for ZSX Primus III NEXT. This allows users to easily copy configurations of established

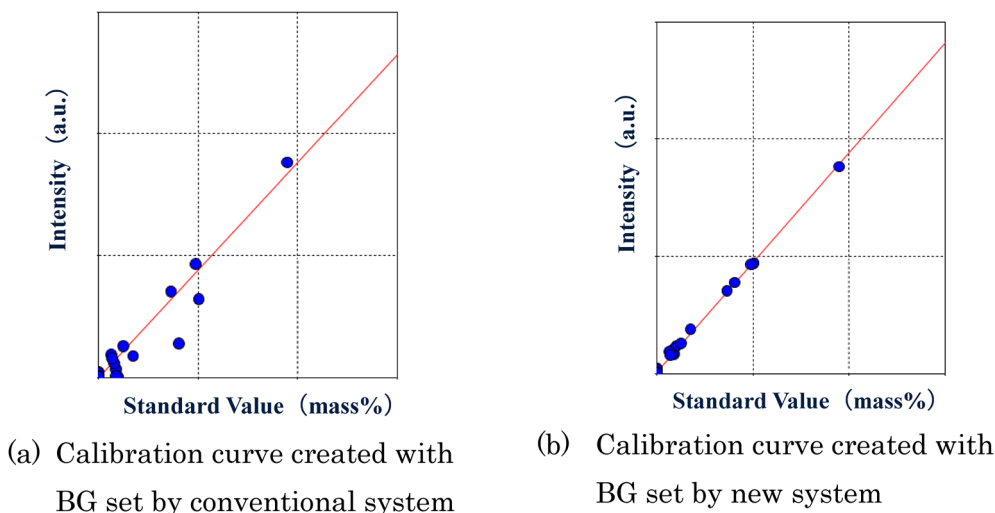


Fig. 4. Comparison of calibration curves from new and conventional systems.

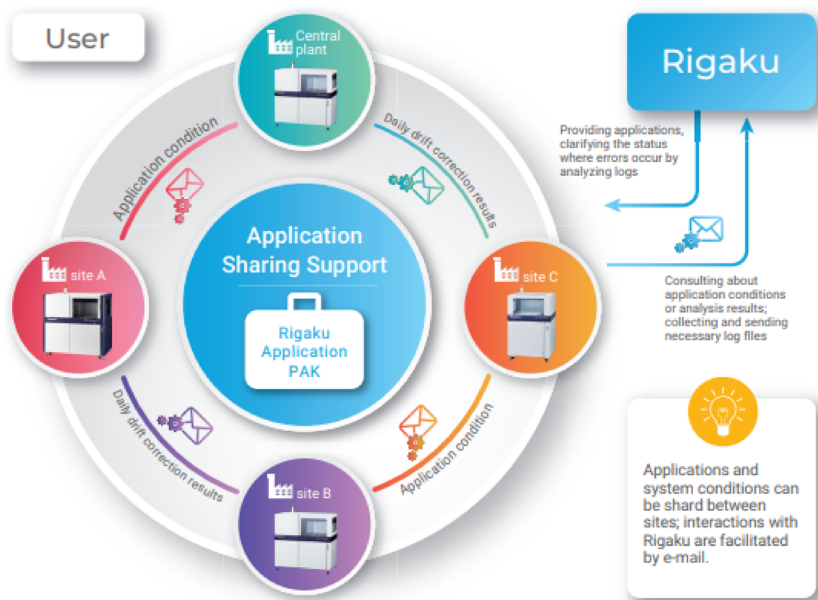


Fig. 5. Rigaku Application Support Overview Chart.

applications between ZSX Primus III NEXT, ZSX Primus IV, and ZSX Primus IVi,

This makes it easy for users to start up analysis on existing equipment. It also allows for smooth migration of existing applications in the event of future equipment upgrades. In addition, it is easy to start up applications remotely and to monitor the status of the equipment such as the results of daily drift correction, between instruments (Fig. 5).

3.2 Application packages

Rigaku also offers original application packages for a wide variety of industries. Pre-calibration packages contain calibration curve coefficients and various parameters. Application packages with certified standard samples will help to establish applications. Two representative application packages are shown below (Fig. 6).



Fig. 6. Examples of application packages offered by Rigaku.

○ Pre-calibration package OXIDE-FB-PAK

This is a quantitative application for fused bead samples, created from 44 standard samples, allowing quantitative analysis of 23 components in samples. OXIDE-FB-PAK is available for oxide materials such as

rocks, soils, oxide ores, slag, cement, etc. The package is available for mixed fluxes of lithium tetraborate (LT) and lithium metaborate (LM) (LT66%/LM34%) and lithium tetraborate (LT100%), and includes a quantitative application for each flux. Glass samples for drift correction for maintenance of the calibration curve, bead samples for confirmation of analytical values, and fluxes are included for each application.

○Pre-calibration package GEO-TRACE-PAK

GEO-TRACE-PAK is capable of analyzing 26 trace elements and 9 major components as references in geological samples by the pressed powder method. It is created from about 100 standard samples of mainly

oxide samples such as rocks, soils, ores, and minerals. In addition, a drift-corrected glass sample for maintenance of the calibration curve and cellulose as a binder for difficult to form samples are included.

4. Conclusion

The newly developed ZSX Primus III NEXT has improved performance and functions in terms of both hardware and software, allowing safer and more reliable use of the instrument. In addition, the analytical support functions that are useful for application management have been enhanced, allowing the instrument to be used for quality and process control analysis.