# Wavelength dispersive X-ray fluorescence spectrometer

# ZSX Primus IV


## 1. Introduction

X-ray fluorescence spectrometry is one of the common instrumental analysis techniques for routine quality control. This is due to high precision and easy sample preparation compared to other instrumental analytical methods. It is also a powerful analytical tool in the field of research and development for the analysis of advanced materials and products with the recent improvement of data processing of fundamental parameter method.

Rigaku has released a new high power sequential type Wavelength Dispersive X-ray Fluorescence (WDXRF) spectrometer ZSX Primus IV with tube above optics to the ZSX Primus family which meets wide variety of recent application needs.

The advantages of tube above optics have been recognized in the fields more widely due to its safe measurement and easy sample preparation for pressed pellets without using binder and protect film.

The new ZSX Primus IV has been developed as a successor of ZSX Primus II with higher performances and many additional software features.

The following are the major features of the ZSX Primus IV:

(1) Automatic quant application setup function
(2) Improved precision, sensitivity and analysis throughput
(3) Intuitive "ZSX Guidance" software with intelligent functionality

The new "ZSX Guidance" software of ZSX Primus IV is an advanced software package assuring better analytical results and easy operation from novice to skilled operators.

## 2. Automatic quant application setup function

### 2.1. Availability of Automatic quant application setting function

There are cases that application parameter settings for quant application are not simple and not easy for novice operators such as line overlap and matrix correction.

This function provides accurate settings for individual analytical parameters with integration of Rigaku's intelligent expertise of X-ray fluorescence analysis to the software.

The followings are the common mistakes in quant parameter settings.

- Selected analytical line is seriously overlapped with a co-existing element.
- Background measuring angle is interfered with an interfering line.
- Matrix correction is not properly set.
- Overlap correction is not properly set.
- The element of an interfering line is not included in the elements of measuring lines.

The "Automatic quant application setup function" provides both novice and skilled users the capability to set measuring conditions and various correction parameters for quantitative analysis automatically avoiding the mistakes above.

Figure 1 shows setting parameters in the new "ZSX Guidance" for the quant application setup.

In the conventional software, quant parameters are determined by manual measurement of spectrum of each line and study of calibration considering matrix and overlap correction which often requires extensive XRF knowledge. As shown in the Fig. 1, the key parameters

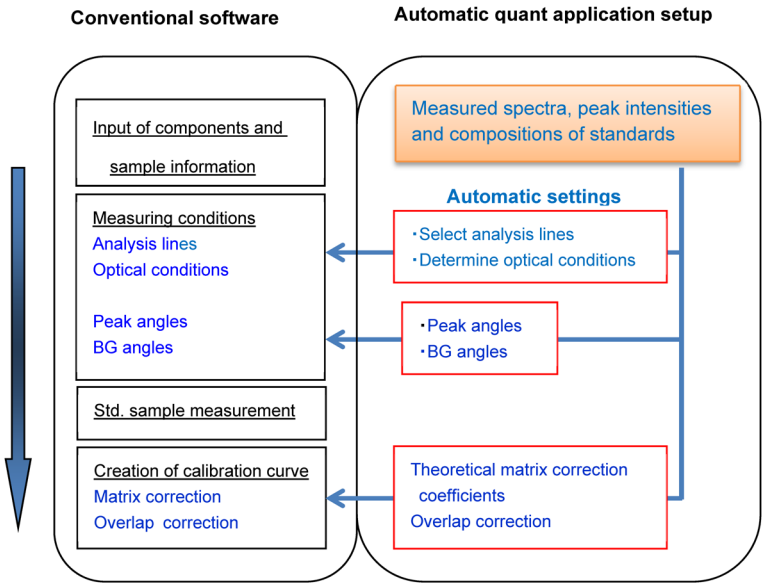


**Fig. 1.** Setting parameters in the new "ZSX Guidance" for quant application setup.

of measuring conditions, necessary corrections are properly determined by using the data of spectra, peak intensities composition of the standards by the software. This function greatly releases novice and skilled operators from complicated procedure of quant application setup.

### 2.2. "Automatic quant application setting function" operation

The operation of the automatic setup is very simple as shown below.

(1) Check 〈Set the analytical conditions in automatic〉 in 〈Create a New Application〉 window
(2) Input sample information, standard sample compositions
(3) Select the menu of "Run Standards SQX" in the quant application flowbar (Fig. 3).

All specified standards are measured with Rigaku semi-quant software of SQX.

After the SQX analysis of the standards, the software setup quant parameters by itself as described below.

(1) Selects optimum analytical lines, determines measuring conditions including peak and background angles using with the qualitative spectra of all standards measured considering line overlap and precision.
(2) When undefined elements are detected in the SQX analysis, the elements are added to measuring elements considering the influence to the original analytes.
(3) Line overlap corrections and matrix corrections are registered with using the composition information and SQX analysis results.
(4) Net intensities for elements of the standards obtained in SQX analysis are registered for tentative evaluation of calibrations.

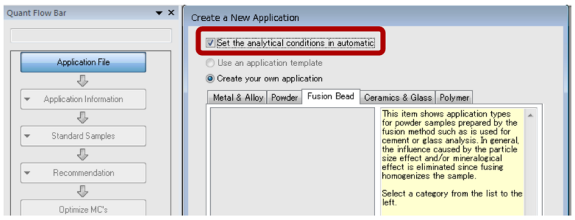


**Fig. 2.** Initiate "Automatic quant setting".

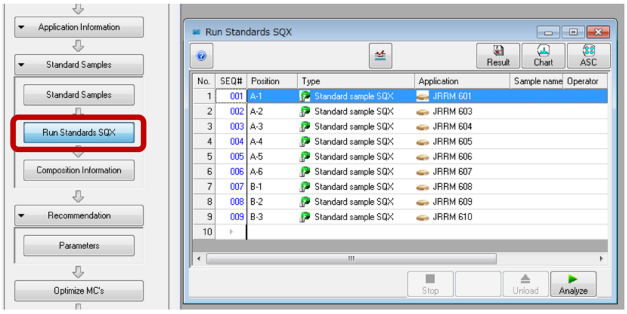


**Fig. 3.** SQX measurements of standards.

## 3. Example of the automatic quant application setting

The application example to zirconia refractory application is explained below.

A qualitative spectrum of the Hf-L$\alpha$ and Hf-L$\beta_1$ lines from hafnium oxide (1.59 mass%) is shown in Fig. 4. Although the L$\alpha$ line or L$\beta_1$ line is generally used for hafnium analysis and the intensity of L$\alpha$ and L$\beta_1$ are nearly equal under normal conditions, this spectrum shows that the L$\alpha$ line intensity is much stronger than the L$\beta_1$ line.

Figure 5 (a) and (b) are the pulse height distribution curves of Hf-L$\alpha$ and Hf-L$\beta_1$ of a zirconia refractory sample. Pulse height value (energy) of around 200 is

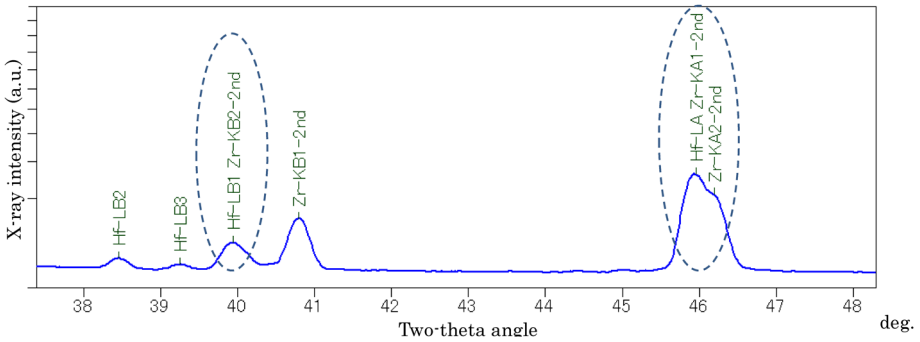


**Fig. 4.** Qualitative chart of Hf-L$\alpha$ and Hf-L$\beta_1$. Analyzing crystal: LiF (200), Detector: SC

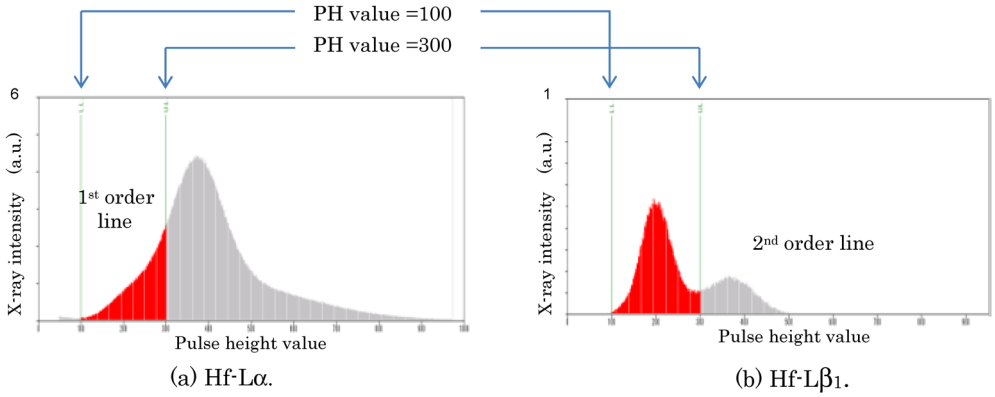


**Fig. 5.** PH distribution curves of Hf-L$\alpha$ and Hf-L$\beta_1$.

the center of the first order line of the objective pulses, and two times higher pulse height value of around 400 is the second order line (which is an interference line). The Hf-L$\alpha$ line should be on the around 200 of the pulse height value in the Fig. 5(a), however, due to the very high intensity of the second order line of the Zr-K$\alpha_1$ (Zr-K$\alpha_1$-2nd from the major component of zirconium), a tail is overlapping into the first order line region. It is obvious that the most of intensity counted under the ordinary pulse height condition 100−300 is Zr-K$\alpha_1$-2nd. On the other hand, Fig. 5(b) clearly shows the separation of the first order line of Hf-L$\beta_1$ from the Zr-K$\beta_1$-2nd (which is the second order line from the major component of zirconium). Therefore, the measurement with an ordinary pulse height condition of 100−300 can be made without the interference of the second order line.

The "ZSX Guidance" software automatically judges the interference situation and selects the proper Hf-L$\beta_1$ line for the hafnium analysis. It is no longer necessary for an analyst to check manually.

The "Run Standards SQX" runs the full range qualitative analyses, even if the major component is not preset to zirconium, for example, and the software checks the interference of all elements detected in SQX and select optimum analysis lines.

Only a portion of the useful features of the "Automatic quant application setup function" is described above. The software includes variety of advanced database based on accumulated XRF expertise and experiences of Rigaku. The burden placed on the analysts is drastically reduced with this software and at the same time the reliability of analysis results can be improved.

## 4. Summary

The new Rigaku wavelength dispersive X-ray spectrometer with tube above optics, ZSX Primus IV, which now incorporates "ZSX Guidance" software can be used with ease by even a XRF beginner. Moreover, the adoption of newly developed hardware also provides advanced performance in sensitivity, precision, and throughput. The ZSX Primus IV is the optimal spectrometer not only for process control but also for R&D.