ZSX Primus IV

Sequential wavelength dispersive X-ray fluorescence

AXT

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**Elemental analysis by WDXRF spectroscopy
employing advanced Guidance expert system**

Rigaku

Leading With Innovation

CES200A/151215E

Elemental analysis by WDXRF spectroscopy

ZSX Primus IV

Sequential wavelength dispersive
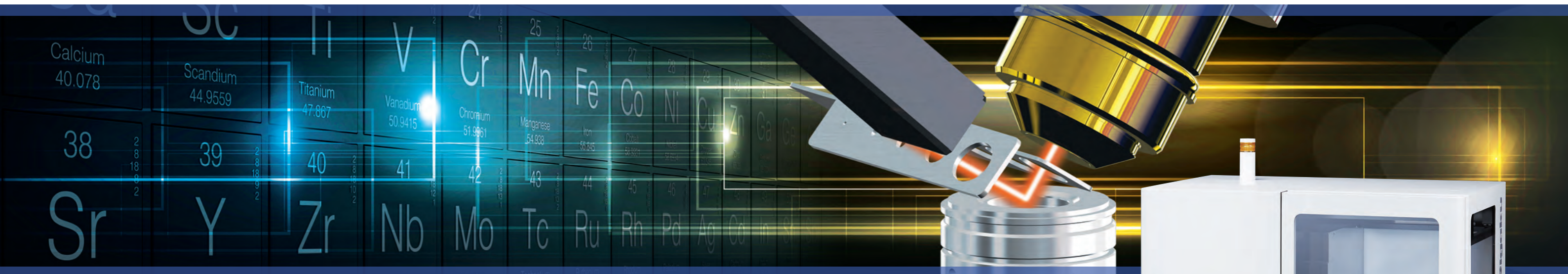X-ray fluorescence

ZSX Primus IV

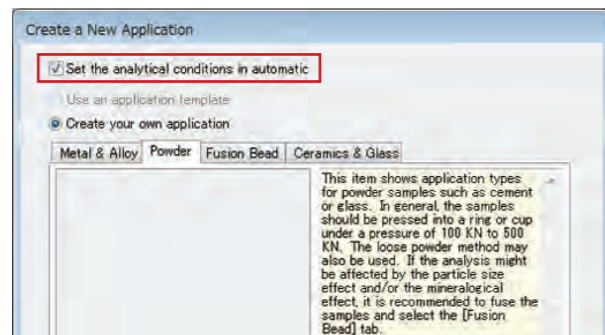# Operation software "ZSX Guidance" supports you in measurement and data analysis

Can accurate analysis only be performed by experts ? No—that is in the past. ZSX Guidance software with the built-in XRF expertise and know-how of skilled experts takes care of sophisticated settings. Optional application packages help you get started with new analyses.
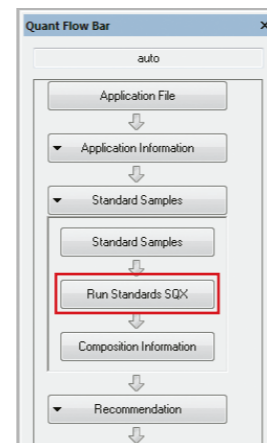
Automatic quant application setup

> **XRF beginners can easily conduct quant applications thanks to automatic setting of measurement conditions and corrections.**

Operators simply input basic information about samples, analysis components and standard composition. Measuring lines with less overlapping, optimum backgrounds and correction parameters —including line overlaps— are automatically set with aid of qualitative spectra.

Dedicated flowbar button for automatic application setup

(Applied to bulk samples of metal & alloy, powder, glass bead and ceramics & glass.)

Searching optimum lines and analysis evaluation for multilayer thin films

> **The best combination of measuring lines giving greatest precision can be suggested, and analyzing depth and absorption by upper layers and line overlap can be simulated.**

Evaluation functions for thin film analysis

There is one searching function for optimum measuring lines and two simulation functions that can be used to display analytical advice for X-ray interference and analyzing depth.

Mode selection and analytical advice

Analyzing depth evaluation

Improved analysis data search and re-quantification

The desired results can be retrieved with a simple operation. Re-quantification can easily be done with revised calibration parameters for quant analysis or with revised calculation model for SQX analysis.

Result view window

Pull-down list
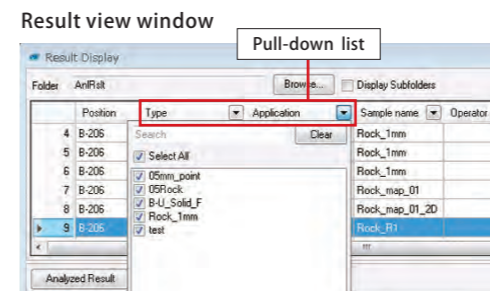
Superior operability in "EZ Analysis"

Functions of "EZ Analysis" for routine operation are enhanced. Recalling a series of sample ID settings can be done by a single-click operation and frequently used applications can be bookmarked.
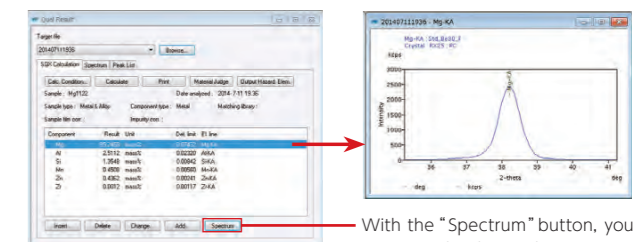
EZ Analysis window

Single click recalling function of sample IDs

"Bookmark" applications
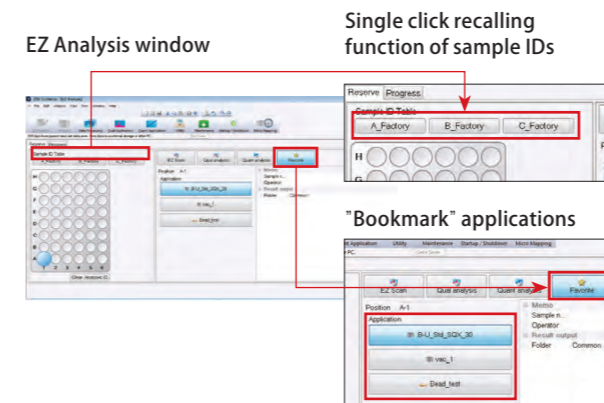
Standardless (semi-quant) analysis "Scan Quant X (SQX)" with built-in sensitivity libraries

> **Accurate results can be easily obtained with simple operation and convenient functions.**

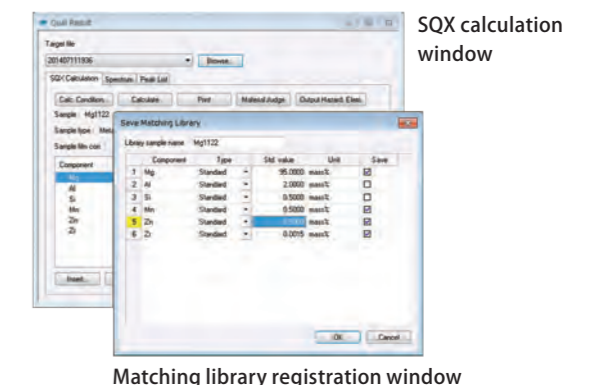### Combined display of SQX analysis and spectrum window

Data handling of SQX analysis can easily be done by referring to both the analysis results and spectra.

With the "Spectrum" button, you can immediately see the spectrum for the element you have selected.

Simplified registration of matching library

The SQX results can be easily added to the matching library for improvement of accuracy.
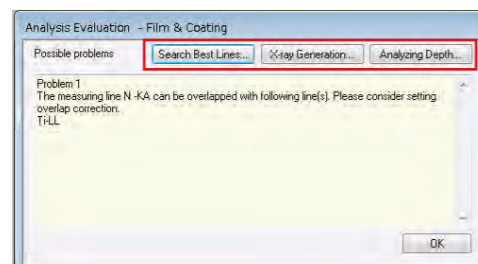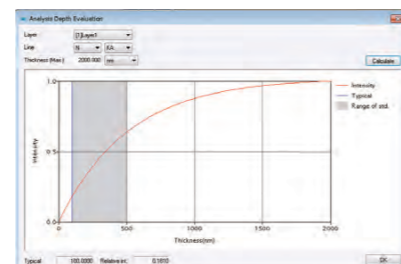
SQX calculation window

Matching library registration window

Analysis packages* support your analysis startup

- **Pre-calibration package** : pre-installed calibration curves and drift correction samples.
- **Application package** : the CRM standard samples and analysis conditions supplied on CD.
- **Master matching library** : the sensitivities optimized for the specific application can provide better semi-quantitative analysis with SQX software.

＊ optional

More accurate even for trace— and high throughput

ZSX Primus IV introduces many cutting-edge technologies that enable unprecedented levels of high-sensitivity and high-precision analysis with high throughput. ZSX Primus IV is a trustworthy partner for high-precision research applications and for quality control applications with large volumes of samples.

High-speed analysis

Throughput has greatly been improved by high-speed sample transportation, high-speed goniometer drive, high-speed data processing and effective driving control. Measurement time has been reduced by 40% for qualitative analysis and 20% for quantitative analysis (in-house comparison) resulting in the highest-possible throughput.

Throughput comparison
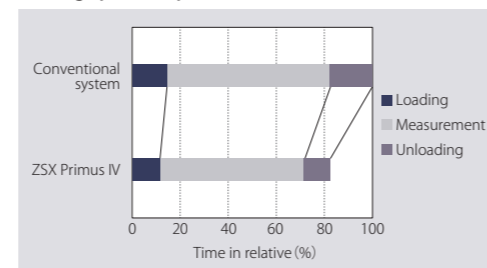
## Improved precision with higher counting linearity by D-MCA System

Counting linearity has been improved by introducing a fast Digital Multi-channel Analyzer (D-MCA), resulting in better precision. Maximum count rate (linearity 1%) : **SC**：1800 kcps, **F-PC**：3000 kcps
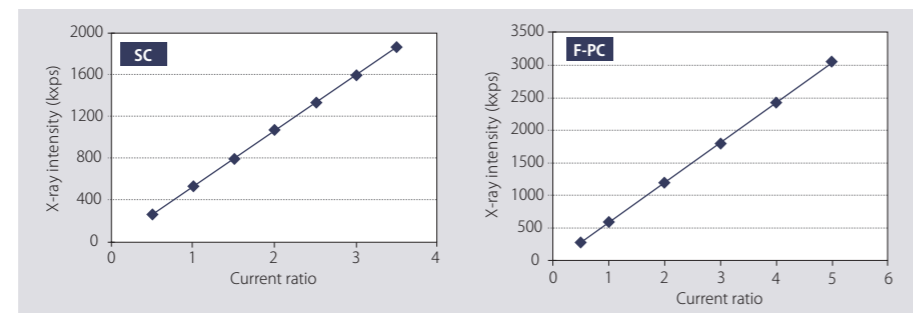
Unique optics reduce sample surface curvature effects

Generally, X-ray intensity is affected by the distance between the sample surface and the X-ray tube. Rigaku's unique optics can minimize the change in intensity caused by such distance variation. This is particularly important when measuring fused beads are measured.

Distance between sample and X-ray tube
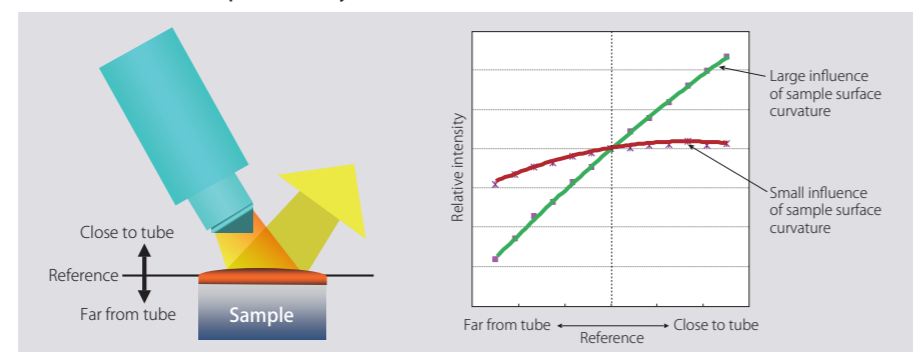
4 kW SuperTrace 30 high-intensity X-ray tube

- **30 μm Be window tube with the best performance for light elements**
  Ultra thin (30 μm) Be window maximizes Rh L line transmission from the X-ray tube, as well as light element fluorescent X-ray intensities.
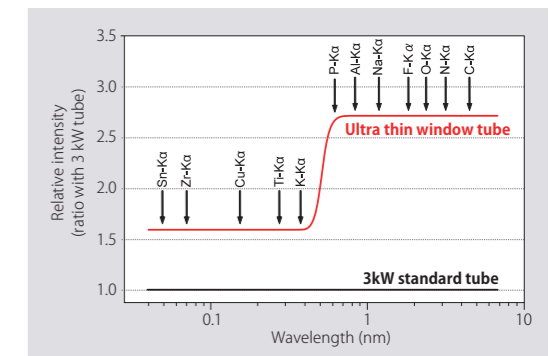
- **Low heat damage to sample**
  The improved materials and structure in the nose of the X-ray tube reduce the influence of heat from tube on the sample, and ensure sample stability during measurement.

- **Stable X-ray emission**
  Significant improvements in the anode cooling and cathode structure minimize X-ray intensity reduction over time and ensure long term stability.
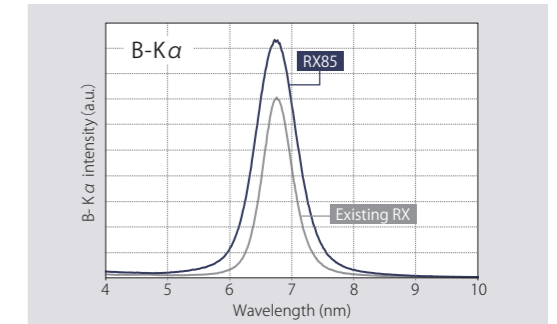

Analyzing crystals

Synthetic multi-layers, RX-SERIES*

The new synthetic multi-layer crystal "RX85" produces about 30% greater intensity than existing multi-layers for B-Kα.

＊Rigaku has been developing proprietary synthetic multi-layers with innovated technology.

Synthetic multi-layer for boron, RX85 (Sample: Boron metal)

Highly sensitive curved crystals

Curved PET and Ge crystals are incorporated in the standard configurations. The intensity for P and S by curved Ge increases by 30% compared with flat Ge. The intensity for Al and Si by curved PET increases by 30% compared with flat PET. Sensitivities in semi-quantitative analysis by SQX analysis could be enhanced with the curved crystals.

Primary beam filters

Primary beam filters inserted between the X-ray tube and the sample eliminate tube target lines or reduce background.

Filter	Description
Al125	Analysis of Cr, Co, Fe and Zn
Al25	Remove Rh- Lα for Cd-Lα measurement
Ni40	Measurements of Pb-Lα and As-Kα
Ni400	Remove Rh-Kα, Kβ for Cd-Kα measurement
Be30	X-ray tube protection (option)

＊1 PET, Ge, RX4: intensity improved by 30% by curved crystal
＊2 RX85 high intensity, RX75 high resolution

ZSX Primus IV

Safety and security features of hardware and software
Minimal maintenance

With high-precision analytical instruments, there is always the worry that the spectrometer could be damaged by an inexperienced operator. This is not the case with ZSX Primus IV—thanks to its tube-above optics, the instrument is safe even if a powder sample spills inside the spectrometer. Also, the access level to software controls can be set for each operator to avoid potential operational errors.

No trouble for powder spills — Tube-above optics

Thanks to the tube-above optics in the ZSX Primus IV, powder sample spills do not cause any problems for the optical system. Since no protective film is required, intensity reduction due to film is avoided.

X-ray tube

Sample

Sample

X-ray tube
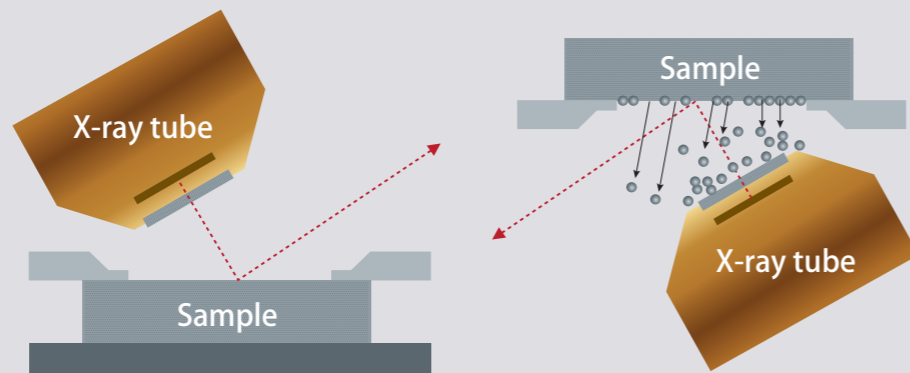
Powder sample attachment installed as standard suppresses entry of dust into vacuum pump.

Prevention of human errors

User access level setting to software

Access level to software can be set for each operator, and modifying or deleting a database by operational errors can be avoided.
The program menu can be configured according to each user's level so that unnecessary items are not displayed, thereby preventing operator errors.
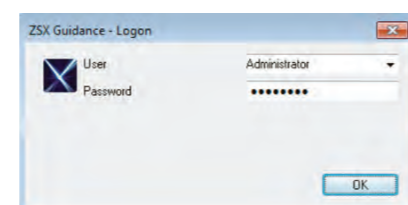
Full menu display
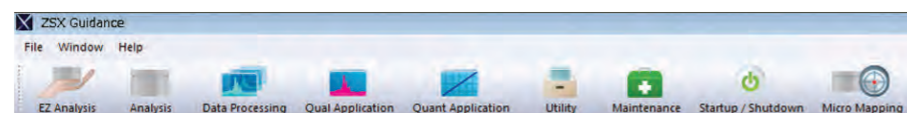
Menu specialized for routine analysis
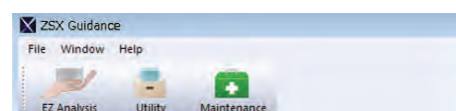
ZSX Guidance logon window

Liquid sample recognition function*

Liquid samples are analyzed under a helium atmosphere. The optional liquid sample recognition function detects when a liquid sample is inserted into the vacuum chamber by mistake, allowing for safe operation.

∗ optional

S-PC LE* : P-10 gas not required

A gas-sealed proportional counter for light elements (S-PC LE) is available as an option instead of a gas flow proportional counter (F-PC) for sites where P-10 gas is not available.

∗ optional

Improved visibility of sample holders

The operator can visually check sample holder locations inside the ASC (Auto Sample Changer) cabinet through the wide transparent window located on the right side of the main body.
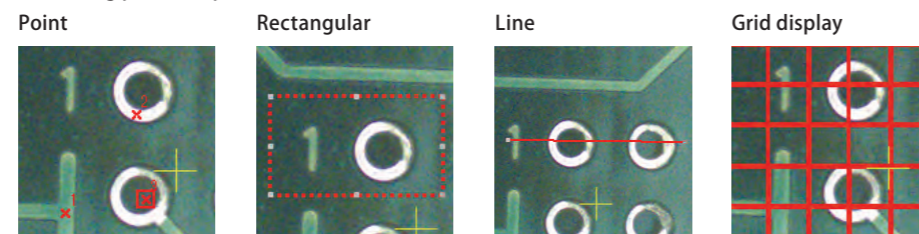
ZSX Primus IV

Rigaku's unique convenience functions

Elemental mapping of sample surface; analysis without the input of unmeasured and liquid samples — ZSX Primus IV can respond to these kinds of analysis elements, including organic samples (C, H, O, N main elements); rapid analysis of powder requirements.

Point and mapping analysis by WDX system

Regions of interest can be located in enlarged images of the sample taken with a high-resolution camera. The r-θ sample stage can then be positioned accurately to make measurements with uniform sensitivity on these small areas.
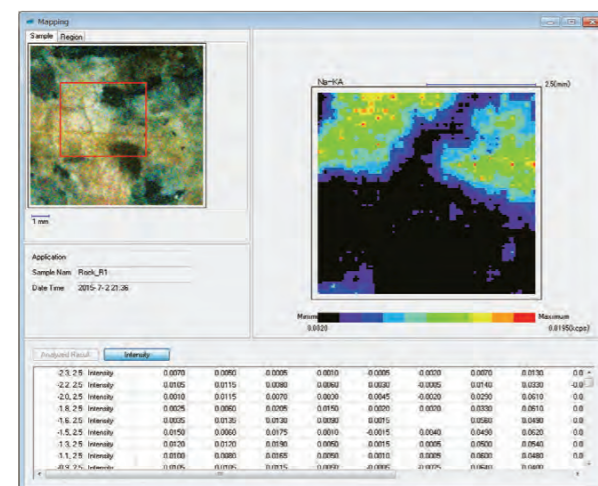

Whole area image
Tool box
Magnified area
Magnified image
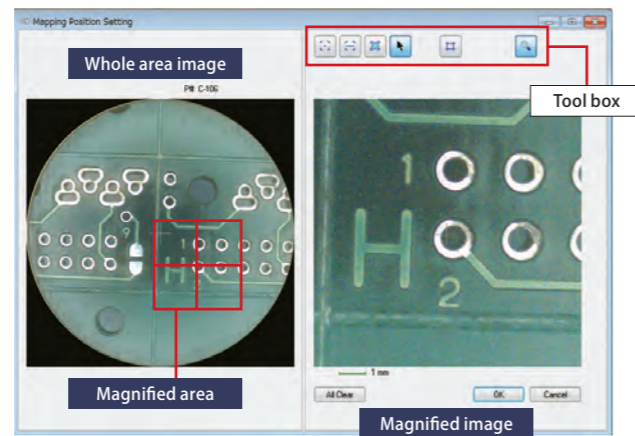
Measuring position specification
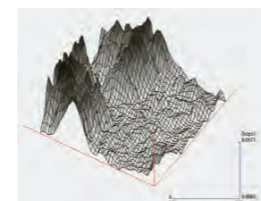
Point Rectangular Line Grid display
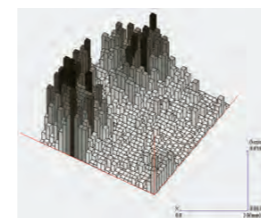

Micro mapping

Sample image and data display (mapping graph, measured data) are integrated in the map view window. A variety of graphical modes are available for mapping visualization.
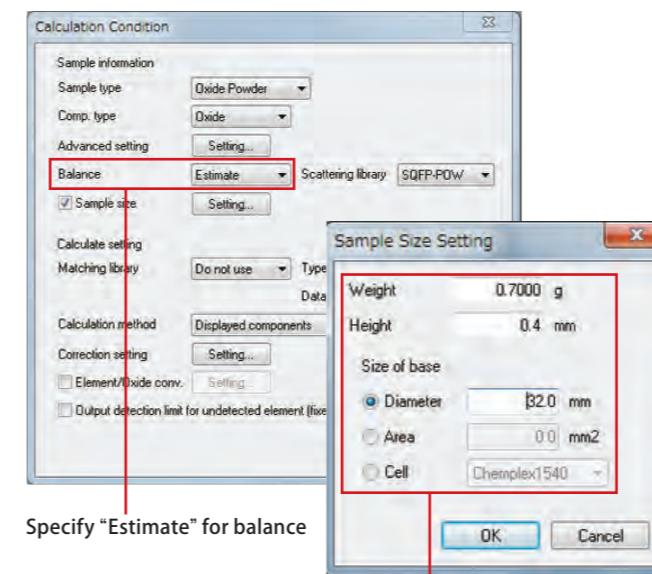

Sample image

3 D display – bird's eye view

3 D display – bar graph
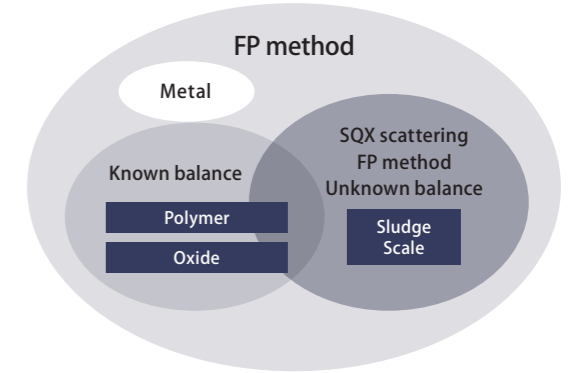
SQX scattering FP method

The presence of major ultra-light elements C, H, O and N affects the analysis results in samples such as sludge and scale. The SQX scattering FP method estimates the influence of these unmeasured ultra-light elements using scattering X-ray intensity and gives accurate semi-quantitative analysis results.

Specify "Estimate" for balance

Input sample weight, height and size

Vacuum partition system for liquid analysis

An automatic helium purge system with a vacuum partition between the sample chamber and the main chamber reduces the time required to convert the sample chamber from vacuum to helium atmosphere while keeping the main chamber under vacuum.

Main chamber (Vacuum)
Vacuum partition (Computer controlled drive)
Sample chamber (Helium)

Applications of SQX scattering FP method

FP method
Metal
Known balance
Polymer
Oxide
SQX scattering FP method
Unknown balance
Sludge
Scale

SQX analysis results of bottom ash municipal solid waste
— Comparison with existing method — (Unit: mass%)

Element	Standard value	SQX scattering FP method Balance estimated	Conventional SQX Balance: oxygen
Ti	0.851	0.883	0.807
Cr	0.086	0.089	0.082
Mn	0.14	0.13	0.12
Fe	2.13	1.96	1.79
Ni	0.012	0.013	0.012
Cu	0.13	0.13	0.12
Zn	2.6	2.5	2.3
Se	0.0041	0.0041	0.0038
Cd	0.047	0.044	0.042
Sb	0.04	0.05	0.05
Pb	1.09	1.02	0.94

Point/mapping analysis of rock

Point and mapping analysis of a rock sample was carried out for the area in the magnified image taken by the built-in camera. The red box in the sample image is magnified and displayed in the right-hand of window, allowing the measurement position to be specified precisely.

Mapping result

- Plagioclase
- Amphibolite
- Alkali feldspar
- Black mica

Fe-KA 2.5(mm) | Mg-KA 2.5(mm) | Ti-KA 2.5(mm)
Ca-KA 2.5(mm) | K-KA 2.5(mm) | Si-KA 2.5(mm)
Sr-KA 2.5(mm) | Rb-KA 2.5(mm) | Al-KA 2.5(mm)
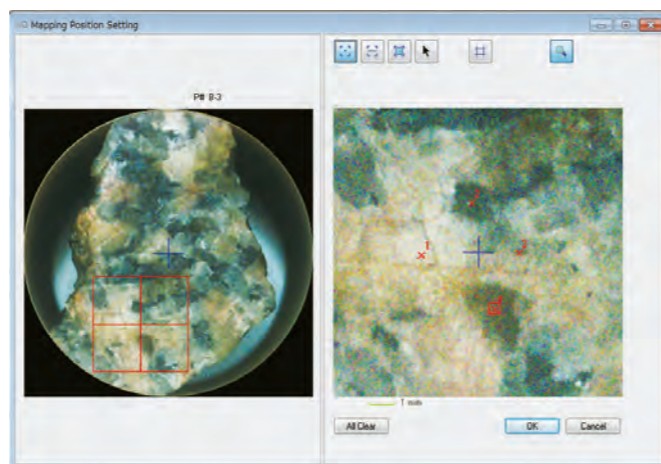
Mg-Kα spectra at each measurement position

2θ angle (deg)

Point analysis results by SQX

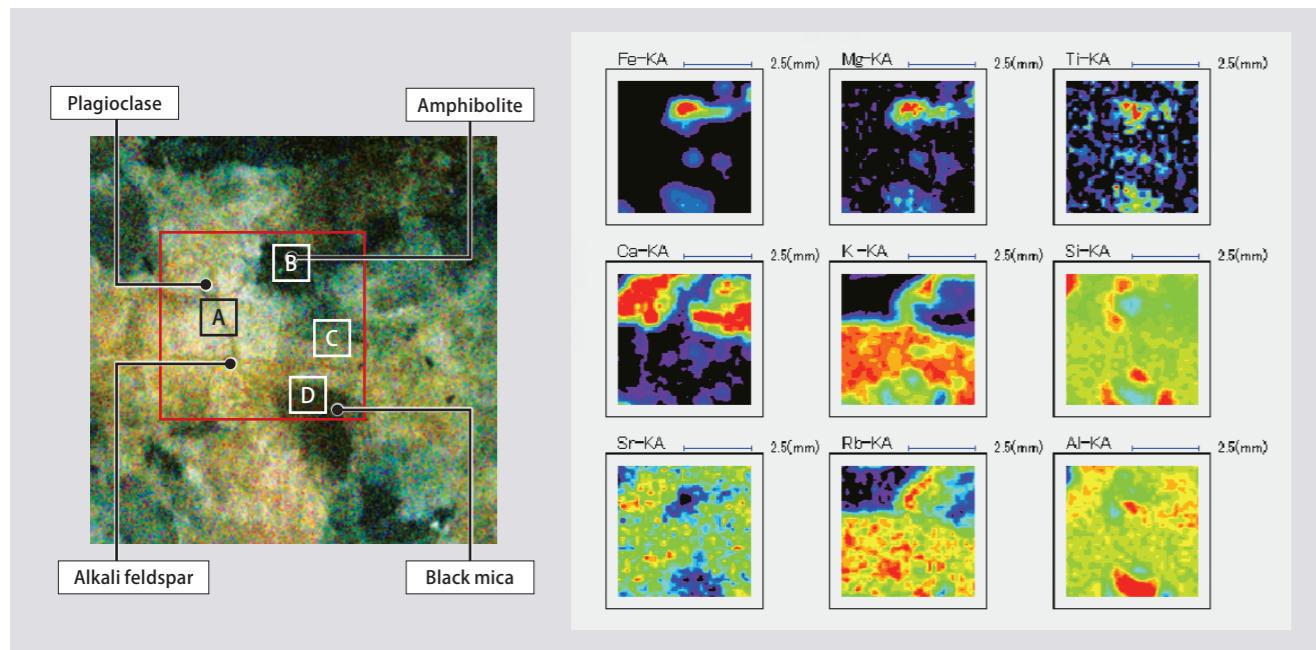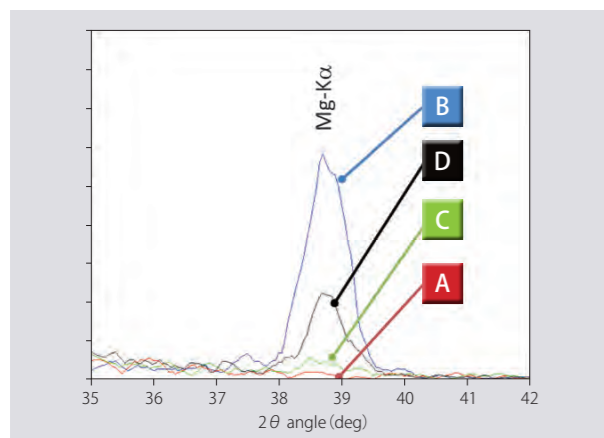(Unit: mass%)

Component	Measurement position			
	A	B	C	D
Na₂O	1.4	2.7	2.3	0.5
MgO	-	2.7	-	0.8
Al₂O₃	20	21	20	30
SiO₂	68	50	66	56
P₂O₅	0.22	-	0.31	-
K₂O	10.3	5.3	8.8	6.8
CaO	0.4	2.5	1.1	0.5
TiO₂	-	1.2	-	0.9
MnO	-	0.1	-	-
Fe₂O₃	-	15	1	5
Rb₂O	0.07	0.1	0.06	0.06
SrO	0.02	0.02	0.03	-
Y₂O₃	-	-	0	-
ZrO₂	-	0.02	0.02	0.02
BaO	0.25	-	0.2	0.23

High-precision analysis of Rh, Pd, Pt in automotive catalyst recovery

Many additive elements with high concentration are contained in honeycomb and pellet catalysts. Therefore, it is crucial to make calibration curves with inter-element corrections for accurate analysis. Excellent calibration curves were obtained for Rh, Pd, Pt. The relative precision for each element obtained is within 1%, which demonstrates that the ZSX Primus IV is suitable for QC analysis for automotive catalyst recovery.

Calibration curves of Rh, Pd, Pt
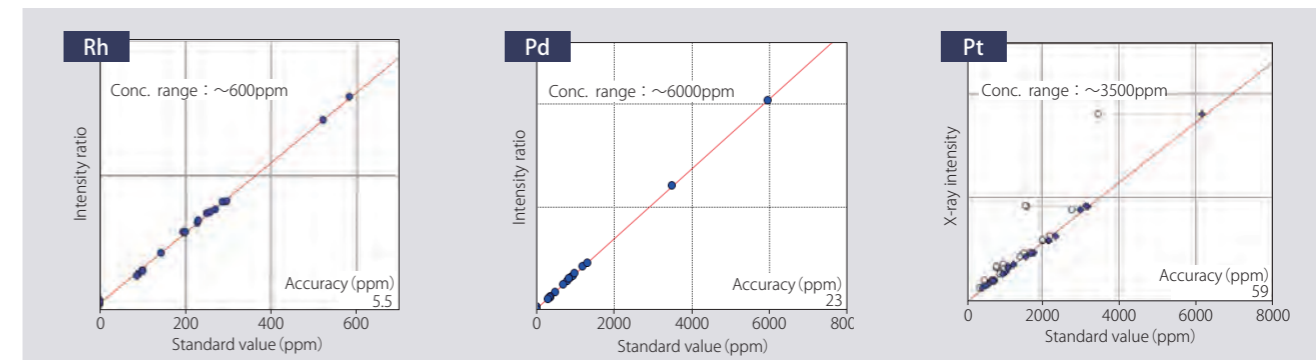

Repeatability test result

(Unit: ppm)

| Element	Rh	Pd	Pt
N=1	230	798	908
2	233	795	911
3	233	790	910
4	230	795	913
5	233	792	902
Average	232	794	909
Range	3.0	8.0	11.0
Std dev.	1.6	3.1	4.2
RSD (%)	0.71	0.39	0.46

Spectra for trace elements in rocks using crystals LiF(200), LiF(220), LiF(420)

High-resolution measurement is required for the region where many trace element peaks are located close to each other in rock analysis. Three types of LiF crystals with different Miller indices are available for heavy element analysis.

LiF(200)

2θ angle (deg)

LiF(220)

2θ angle (deg)

LiF(420)

2θ angle (deg)
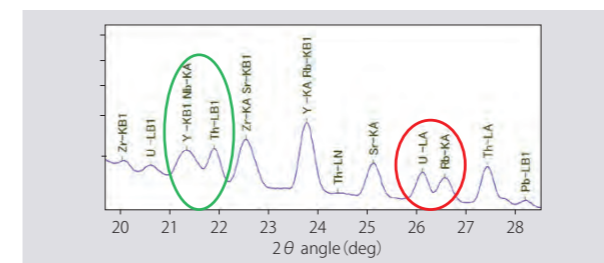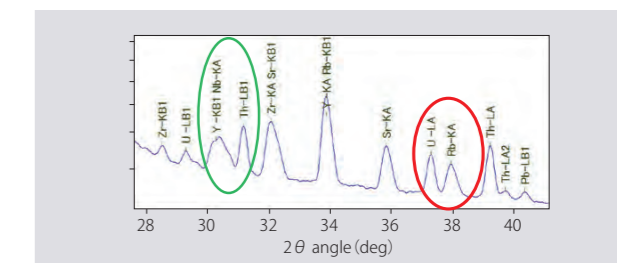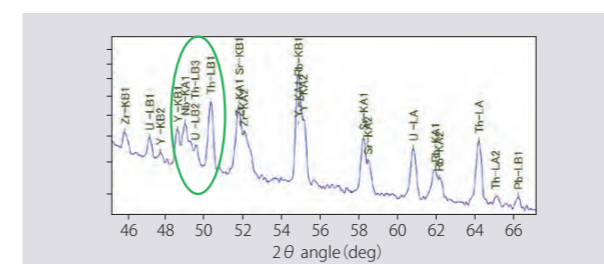
Red circles show that peaks from U-Lα and Rb-Kα overlap at their tails with LiF (200), but they are separated in LiF (220).
Green circles show that peaks of Y-Kβ₁, Nb-Kα, U-Lβ₂ and Th-Lβ₃ are identified in LiF (420) more clearly than with other crystals. With high-resolution crystals, overlapping of peaks can be minimized and background measurement settings can be made easier.
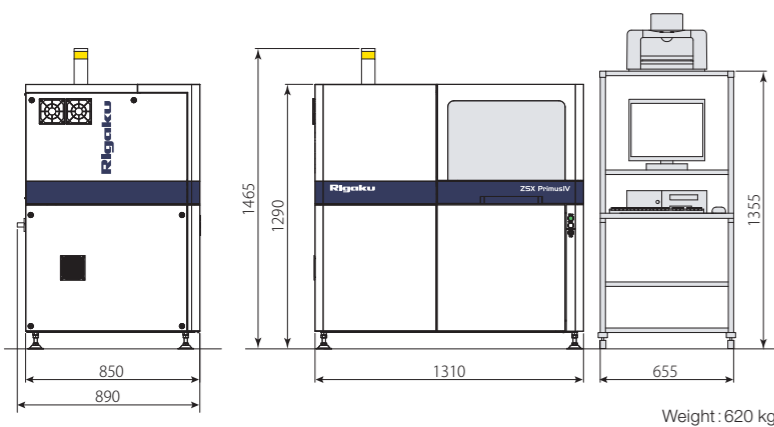
System and installation specifications

Specifications

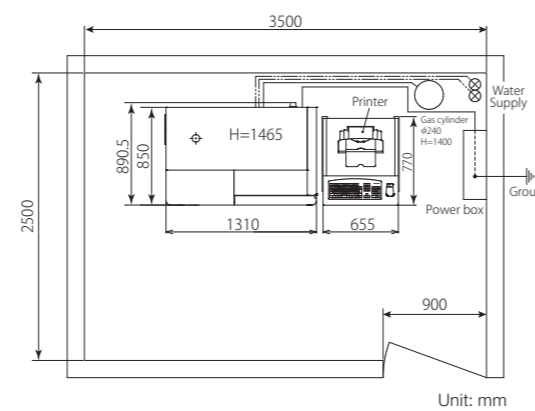|		
X-ray generator	X-ray tube	End window type, Rh target 4 kW or 3 kW
	High voltage generator	High frequency inverter system Max rating: 4 kW
Spectrometer	Maximum sample size	ϕ52 mm × 30 mm (H)
	Primary X-ray filter	Ni400, Ni40, Al125, Al25
	Analysis area diaphragm	6 sizes automatic exchange mechanism (ϕ35, 30, 20, 10, 1, 0.5 mm)
	Crystal exchanger	10 crystal automatic exchange mechanism
Counting system	Detector	SC for heavy elements F-PC for light elements Heating-type center-wire automatic cleaning or Optional S-PC LE (Sealed proportional counter for light element)

Installation specifications

Power	3 Phases 200 V 40 A, Single phase 100 V 15 A (PC)
Earth grounding	Independent grounding with resistance less than 30Ω
Cooling water	Temperature: Lower than 30 ℃ Pressure: 0.29 - 0.49 MPa Flow: More than 5 L/min Quality: Equivalent to drinking water
Drain	Gravity drain
Room temperature	15 – 30 ℃ (with daily variation within ±2 ℃)
Humidity	Less than 75 %RH
Vibration	Less than 2 m/s^2 (Below human sensitivity level)
P-10 gas for F-PC	Ar 90 % Methane 10 % Mixture gas pressure 0.15 MPa Gas flow 7 ml/min

External shape and dimensions

850
890
1465
1290
1310
655
1355

Weight : 620 kg

Typical layout

3500
2500
890.5
850
H=1465
770
Printer
Gas cylinder
φ240
H=1400
Water Supply
Ground
Power box
1310
655
900

Unit: mm

Backed by Rigaku

Since its inception in 1951, Rigaku has been at the forefront of analytical and industrial instrumentation technology. Today, with hundreds of major innovations to our credit, the Rigaku Group of Companies are world leaders in the field of analytical X-ray instrumentation. Rigaku employs over 1,100 people worldwide in operations based in Japan, the U.S., Europe, South America and China.

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