
Product Information

Fully Automated RIX3001 X-ray Spectrometer



1. Introduction

Rigaku designed the RIX Series to give engineers and scientists freedom in configuring the optimum system for their needs, from the simplest routine to the most complex analytical problem, at all budgetary levels. Rigaku also designed this series to meet the increasing demands of high performance and flexibility for X-ray instrumentation, including sensitivity, speed of measurement, sample throughput, and simplicity.

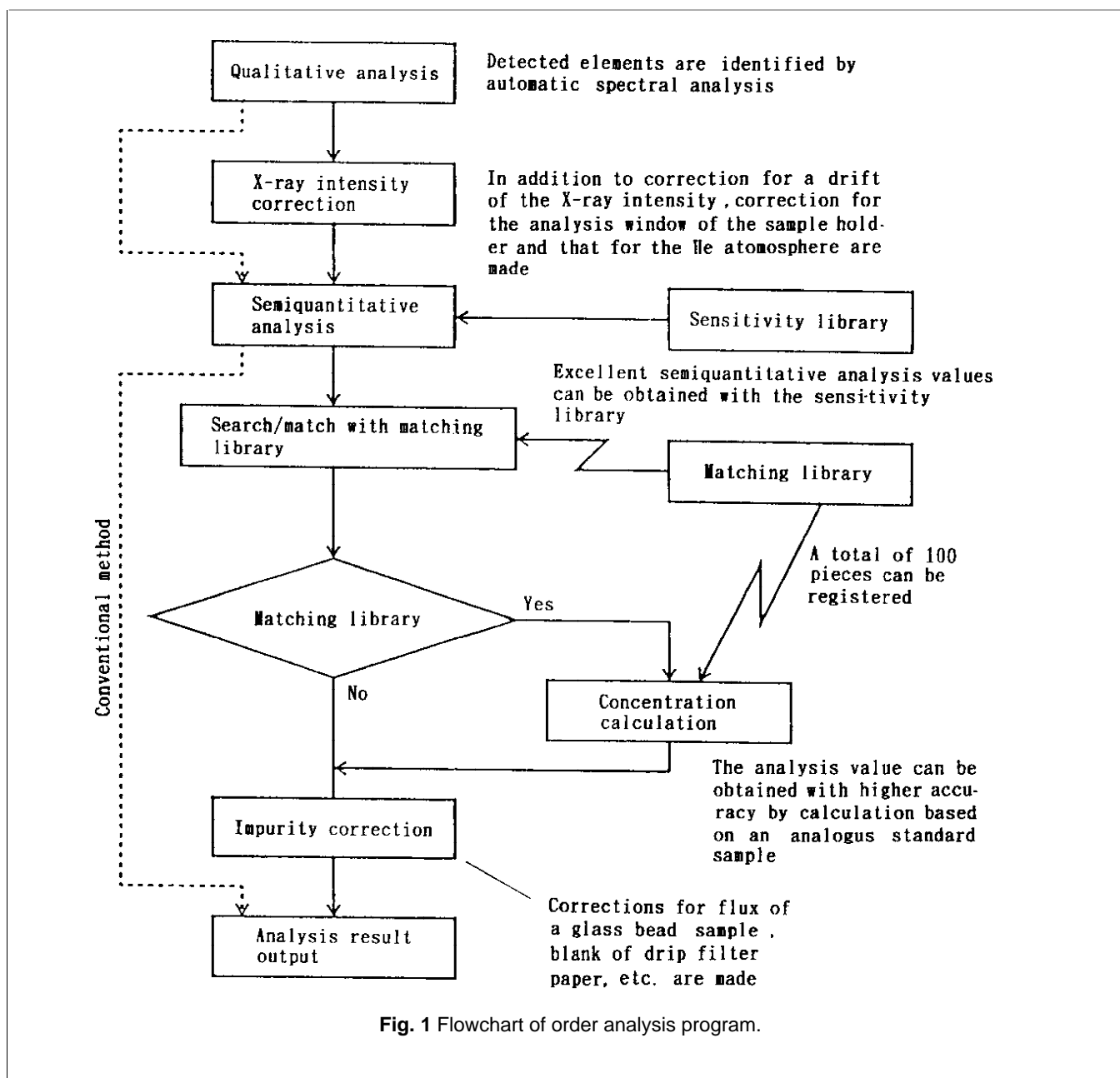
The RIX Series of sequential WDXRF spectrometers is the most comprehensive in the industry; it is completely supported by Rigaku, with its solid foundation and commitment to expand product offerings. Sophisticated hardware and multitasking software combine to make the RIX Series the most advanced, fully automated X-ray spectrometer system available today.

The RIX3001 is based on the RIX3000, the most powerful and sophisticated of the RIX Series. The RIX3001 was specifically designed with inverted optics for customers who routinely analyze liquids or

loose powders and require minimal sample preparation time.

The RIX3001 is a multi-purpose tool for performing routine analysis of elements. Be through U, as well as for fundamental research and development at the frontiers of science. The RIX3001 can analyze light to heavy elements at ultra-high speeds and has numerous automated functions such as automated aging of the X-ray tube and an automated PC center wire cleaner. The RIX3001 is equipped for three sollar slits, ten-crystals, four primary beam filters, and five area limiting diaphragms. This system can even be configured with dual target X-ray tubes. The sophisticated hardware of the RIX3001 results in high sensitivity and outstanding analytical precision.

The RIX3001 utilizes fourth generation RIX software based on IBM's Warp Operating System. Consequently, it is a true 32 bit multi-tasking, multi-window software package. This sophisticated, yet easy-to-use software easily integrates into other commercially available host computers, network or LIMS systems.



The many benefits of the RIX software include time and cost saving operational features as well as a variety of analytical techniques for the diverse requirements of today's analysts. The analytical techniques include both qualitative and quantitative programs for bulk, medium-, thin-, and multilayer-film analysis. One of the unique features of the RIX Software is the Order Analysis Method, a simple and accurate method for standardless analysis. High-speed scanning, a matching library, and corrections for the atmosphere and common impurities allow for accurate analysis in as little as three minutes.

This powerful combination of sophisticated RIX3001 hardware and multitasking software results in ever faster analysis combined with higher accuracy and precision.

2. Features

1) Inverted optics

The RIX3001 is configured with the X-ray tube below the sample. This optics arrangement allows for simple and quick sample preparation of loose powders and liquids. Liquids or powders can easily be poured into disposable sample cups and covered with disposable films for analysis.

The inverted optics configuration is known to have the potential of liquids or powders falling onto the X-ray tube and into the analytical chamber. However, great care was taken to protect the X-ray tube and analytical chamber from potential accidents with Beryllium windows that automatically cover each section when a helium path is chosen for the analysis of a liquid or loose powder.

2) Order analysis method (Fig. 1)

a) High-speed Continuous Scan in Three Minutes

A complete qualitative scan with semi-quantitative fundamental parameters analysis results can be completed in three minutes with the Order Analysis Method. In addition, Rigaku's unique double vacuum system permits pumping for the next sample while measurement of another sample is underway. This contributes to overall high-speed qualitative analysis.

b) *Matching (Individual) Library* The Order Analysis Method provides a Matching (Individual) Library that can be added to the current Common (Standard) Library. The Common Library contains pure elements and compounds, while the Matching Library is one that contains the users own standard samples. When an unknown sample is analyzed, semi-quantitative analysis is first performed with the Common Library, followed by an automatic search of the Matching Library of any reference materials which is closer in composition to the unknown sample. The results are then recalculated using that library. Consequently, even samples containing ultralight elements can be analyzed with a higher degree of accuracy.

c) *Atmosphere & Impurity Corrections* Once the standards are measured under vacuum and put into the libraries, the Order Analysis Method allows the operator to specify if a measurement is being made under vacuum or Helium at the time of analysis. The software will then correct for X-ray absorption automatically according to the atmosphere specified.

Trace contaminants are found in polymer films used to cover liquid or powder sample cups, in fusing agents used in glass bead manufacture, and in filter paper used when concentrating liquid samples. The Order Analysis Method software contains a function that will correct for these types of impurities.

3) Selection of optimum X-ray tube

A choice of X-ray tubes is available to satisfy today's diverse needs. X-ray tubes available include a standard 3 kW Rh target tube, a high-power 4 kW tube (combined with a high-frequency X-ray generator), and a dual-target tube featuring better sensitivity for the intermediate elements, Ti through Cl. Analysis can be made over a wider range of elements without the time-consuming need of installing different X-ray tubes when the switchable dual target X-ray tube, which allows the exchange of two kinds of target materials, is used. (Table 1, Fig. 2, Fig. 3)

Table 1 Features of different X-ray tubes

Type of X-ray tube	Features
Standard Rh X-ray tube	The development of this vertical X-ray tube in early 1970 superseded the common horizontal X-ray tube, leading to a substantial improvement of sensitivity for light elements. Over the last 25 years, this vertical type tube has been used as the optimum X-ray tube for analysis over a wide elemental range, from light to heavy elements.
Dual target X-ray tube	A single X-ray tube is equipped with two kinds of targets, e.g., Rh/Cr or Rh/W, for analyses that require measurement of an element region where the sensitivity of the Rh target is insufficient.
Ultratube UT X-ray tube	An Rh X-ray tube designed to improve further the sensitivity for light elements.
4 kW Rh X-ray tube	This tube enables up to 4 kW of power. This tube improves sensitivity over the entire elemental region.

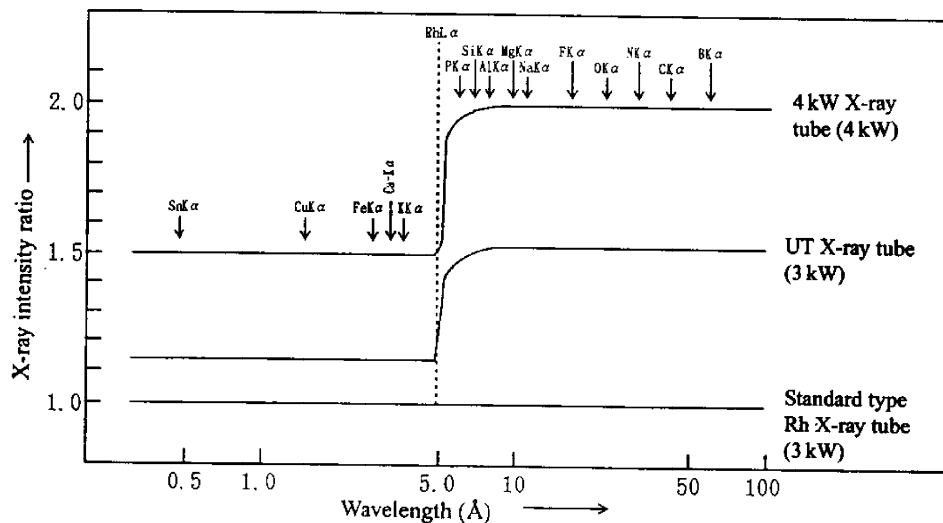


Fig. 2 Sensitivity comparison of X-ray tubes.

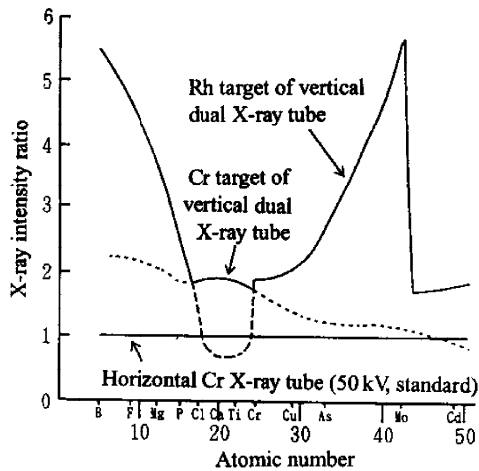
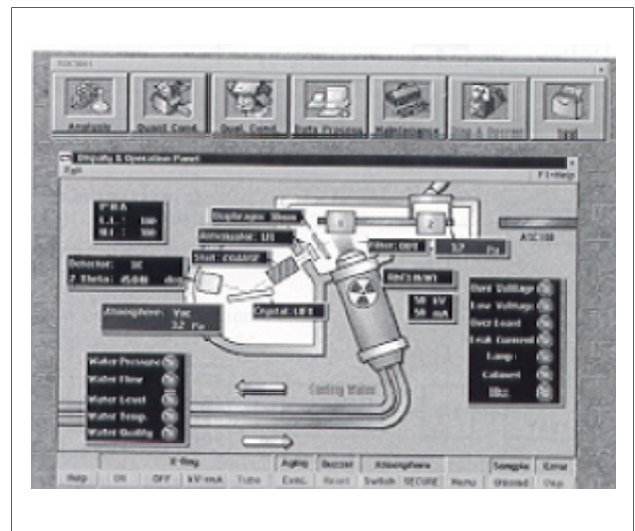


Fig. 3 Sensitivity of Rh/Cr dual X-ray tube.

4) Easy-to-understand operational screen

An illustrative screen enables the operator to evaluate the status of the entire spectrometer at a glance. Even the novice can easily evaluate the status.



3. Applications

Applications and typical examples are listed.

Application	Example
Electronics, Magnetic material	LSI, memory disk, CRT parts, magnetic material, hydrogen storage alloy
Chemical industry	Catalyst, polymer, pharmaceuticals, fertilizers, pigments, paint, oil and fat, detergent, cosmetics
Ceramics	Silicon nitride, alumina, glass, firebrick, enamel, cement
Steel	Special steel, surface treated steel plate, ferro alloy, cast iron, cast steel, iron ore, plating fluid
Non-ferrous metal	Aluminum can material, shape memory alloy, copper alloy, precious metal, Ni alloy, solder, plating fluid
Mining	Ore, rock, volcanic ash
Petroleum, Coal	Grease, lubricant, cutting oil, kerosene, heavy oil
Environment	Waste water, seawater, river water, air-borne dust, industrial wastes
Other	Soil, rock, plant, organism, food

4. System Configuration

4.1 Hardware

Figure 4 shows the RIX3001 system configuration. When a sample is irradiated with a beam of

primary X-rays, secondary X-rays, or fluorescent X-rays, are induced. The wavelengths of these fluorescent X-rays are characteristic of the elements in the sample. The fluorescent X-rays are then dispersed by

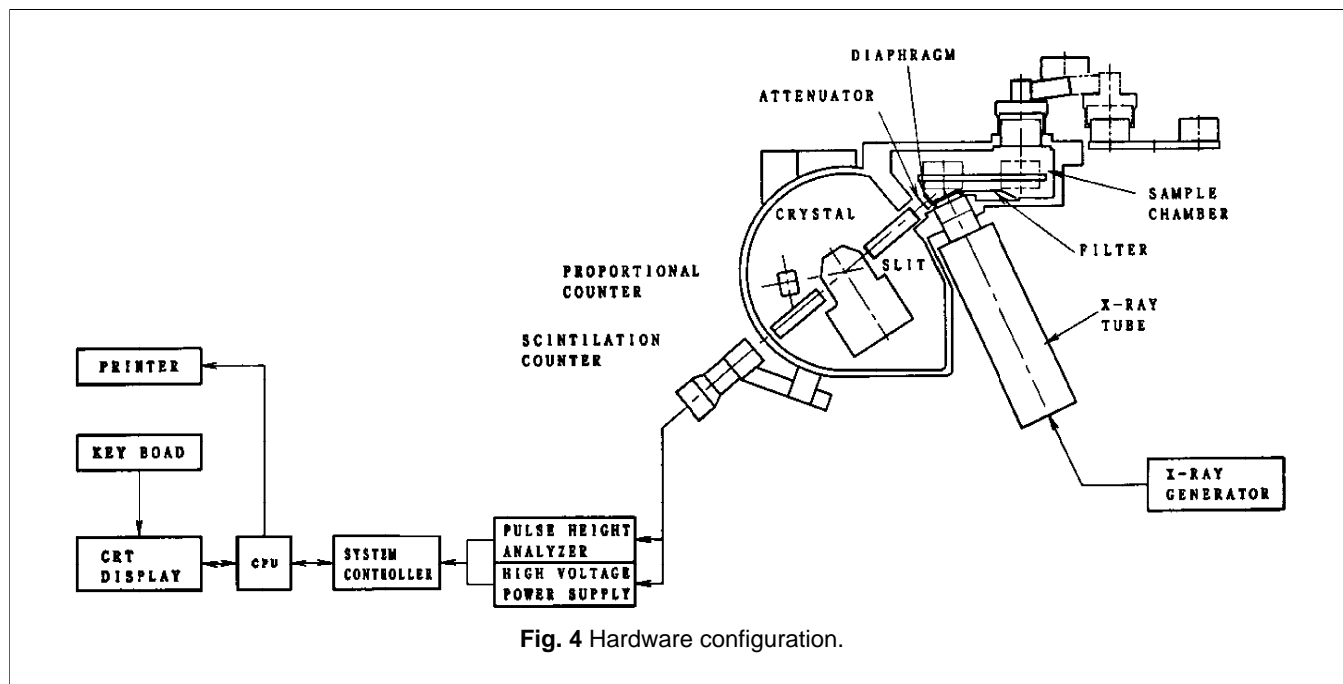


Fig. 4 Hardware configuration.

an analyzing crystal and detected with a Scintillation Counter or a Flow-Proportional Counter.

The fluorescent X-rays have an intensity that is proportional to the number of excited atoms of the given element. For quantitative analysis, the intensity of a given element is measured at its fixed spectral angle.

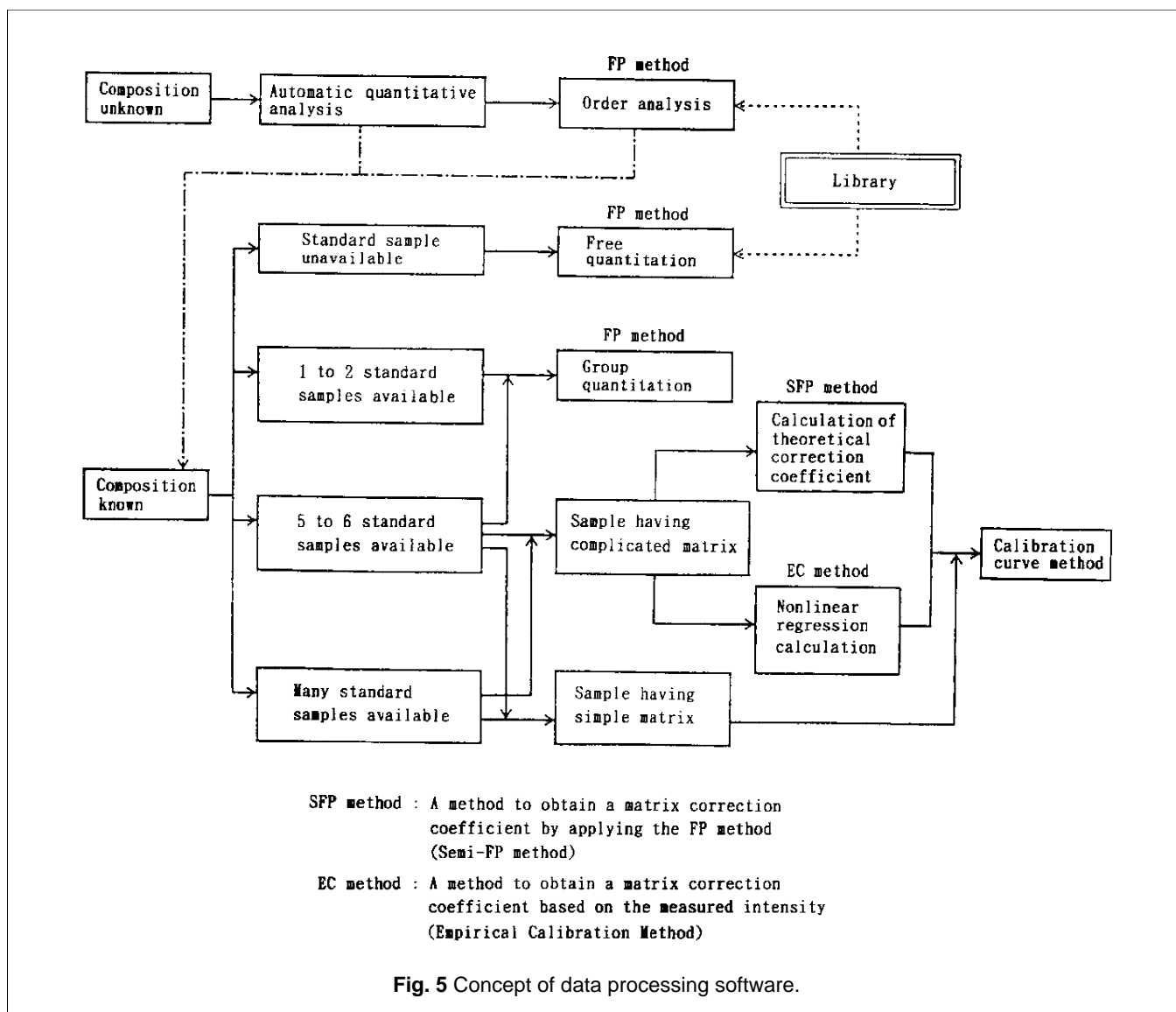
A microcomputer controls sample placement, goniometer movements, and the operation of the counting circuits, while a personal computer handles the data analysis and presentation.

4.2 Data Processing Software

Many functions are incorporated in the RIX3001 data processing software, as shown in Figure 5. These functions meet the diverse analytical needs for routine

analysis as well as for research and development analysis.

Operational features include PC control of kV and mA, automated power on and power down of kV and mA, automated PHA adjustments, and peak angle updates. Repetitive analysis procedures can be programmed into Batch files. Unique preparation and adjustment software allows 20 and PHA scans of each element to be measured in a group for easy setup of measuring conditions. The Programmed Analysis can run PHA, drift corrections, and check standards before one arrives at work or at the beginning of each shift. The Statistical Process Control Software includes reporting, plotting and histograms, X-R control chart, accuracy test evaluation, and variance analysis.



Qualitative software includes peak search and identification, background fitting and subtraction, peak deconvolution, high-speed scanning, and matching libraries. The Qualitative program contains comprehensive spectral information for all elements, including optimum instrument settings and the determination of the presence of overlap and higher order peaks.

Quantitative software includes Semiquantitative Fundamental Parameters for situations where no standards are available. Quantitative Fundamental Parameters to automatically correct for Matrix effects, theoretical intensity calculations, and matrix correction coefficient calculations. Calibration curve methods include regressions of up to second order polynomials and up to three segments, five empirical methods for interelement corrections, and background fitting for multiple point fittings.

5. Specifications

Measurable element range (${}^4\text{Be}$, ${}^5\text{B}$, ${}^6\text{C}$, ${}^7\text{N}$, ${}^8\text{O}$, ${}^9\text{F}$ ~ ${}_{92}\text{U}$)

X-ray Spectrometer Assembly	
X-ray tube:	Rh, Rh (UT), Rh/Cr, Rh/W, Cr, W, Mo, Pt
Primary beam filter:	Automatic exchange of 4 kinds
Area limiting:	Automatic exchange of 5 kinds
Diaphragm:	(10, 20, 25, 30, 35mm ϕ)
Attenuator:	Automatic exchange (attenuation rate: 1/10)
Slit system:	Automatic exchange of 3 kinds (Coarse/fine/ultralight elements)
Crystal exchanger:	Automatic exchanger of 10 crystals
Analyzing crystal:	(Standard) 7 kinds LiF (200), LiF (200)-PC, PET, Ge, TAP, RX40, RX60 (Optional) RX4, RX35, RX70, RX80, LiF (220)
Detector:	For heavy elements: Scintillation counter (SC) For light elements : Gas flow proportional counter (F-PC) Automatic center wire cleaner Gas density stabilizer 0.6 μm polyester window
PR gas system:	Standard
Sample chamber:	Sample turret: 6-sample changer (standard) Automatic sample changer: ASC50 (optional) ASC100 (optional) Sample size: 51 mm dia. X 30 mm high, max. Sample spin: 30 rpm Atmosphere: Vacuum/air (standard) He or N ₂ (optional)
Goniometer:	Scan range: 5° to 118°(2 θ) with SC 8° to 148° (2 θ) with F-PC Scan speed: Max. 1000°/min (Step scan): 5/1000°, 1/100°, 2/100°, 5/100°, 1/10° (Continuous scan): 0.1°~240°/min Angular reproducibility: Better than $\pm 0.0005^\circ$
X-ray Generator	
Load:	3kW max. 4kW(optional)
Tube voltage:	60 kV max.
Tube current:	100 mA max. (L50 mA at 4kW)
Stability:	$\pm 0.01\%$ for $\pm 10\%$ line variation
Control:	Microcomputer control Automatic aging (standard) Automatic analysis programming (optional)

HeatExchanger

Deionized water recirculating system (built in)

X-ray Counting System and System Controller

Pulse height analyzer: 2 θ pulse height linking method with peak shift correction circuit and counting loss correction circuit
Max. counting: SC system: 1 X 10⁶ cps
rate F-PC system: 2 x 10⁶ cps

Timer: 0.1 to 999 sec.

Scaler: Max. counting capacity: 2³²-1

High voltage power: One each for SC and F-PC
supply for detector

Microcomputer for: 16 bits
control

Data Processing System

HardwareComputer: 32-bit personal computer, self-containing hard disk
Floppy disk drive 3.5" 1.2 MB x 1
17" color CRT, 15" color printer, mouse
(optional): X-Y plotter, page printer

Operating system: OS/2

On-line:
data processing
(major functions) *(Quantitative analysis)*
Fundamental parameter method
Group analysis: 40 components for bulk samples, 10 layers/40 components for thin films
Free analysis: 40 components for bulk samples, 10 layers/40 components for thin films
Order analysis: 40 components for bulk samples, 1 layer/40 components for thin films
Calibration curve method
Quadratic equation, 3 divisions (automatic selection)
Matrix correction: 5 methods including JIS Multipoint background fitting (16 points max.)

(Qualitative analysis)
Counting loss correction, smoothing, peak search, background subtraction, identification
analysis, concentration calculation (order analysis)

Off-line:
data processing
(major functions) *(Quantitative analysis)*
Fundamental parameter method (same as on-line)
Theoretical intensity calculation, matrix correction constant calculation
Calibration curve method (same as on-line)

(Qualitative analysis)
Smoothing, peak search, background subtraction, identification analysis, background fitting,
peak separation, concentration calculation (order analysis), quantitative calculation
Display function: Overlap display,
Horizontal axis: 2 θ angle/wavelength/energy by switchover
Vertical axis: X-ray intensity: Linear/log by switchover/etc.

Report and Data: Report, plot & histogram, X-R control chart, accuracy test evaluation,
transmission variance analysis, (unitary arrangement), external CPU transmission, ASCII file output

Preparation &:
adjustment Storing of quantitative measuring conditions, automatic PHA adjustment,
2 θ correction

6. Application Examples

1) Order analysis of tool steel and high-speed steel

Table 2 shows a comparison of the Order Analysis results using the common library and the matching (individual) library for tool and high-speed steel. The use of the matching library gives better results in general. However, the analysis accuracy of carbon, an ultralight element, improved dramatically.

2) Order analysis of S, Fe, and Ni in fuel oil

Table 3 shows the Order Analysis results of fuel oil samples. The improvements in analytical results by using corrections for atmosphere, analysis windows and impurities are clearly illustrated for S, Fe, and Ni.

3) Quantitative analysis of Ni and V in fuel oil

This is an example of the analysis of metallic components in fuel oil. The results show that the primary beam filter can be used to reduce background and improve the detection limit. Figures 6 and 7 show examples of the analysis of Ni and V, respectively, in fuel oil.

4) Analysis using the oil caking method

When light elements in an oily sample are analyzed, the use of a liquid sample cell creates poor detection sensitivity due to the attenuation of the X-ray intensity from the analysis window (polymer) and the atmosphere. In such cases, if the oil caking method is employed, the liquid sample cell is not necessary. The Oil Caking Method literally makes a cake of the sample by mixing it with special caking agents. Figure 8 shows an example of a quantitative analysis of Na in oil using the Oil Caking Method.

Table 2 Order analysis of tool steel and high-speed steel

Unit: % (m/m)

	Sample (tool steel): JSS602-1					Sample (high-speed steel): JSS608-5				
	St'd value	With common library		With matching library		St'd value	With common library		With matching library	
		Order analysis value	Difference	Order analysis value	Difference		Order analysis value	Difference	Order analysis value	Difference
C	1.24	7.3	+6.06	1.22	-0.02	0.77	4.9	+4.13	0.78	+0.01
Si	0.26	0.24	-0.02	0.26	0	0.28	0.24	-0.04	0.274	-0.006
P	0.013	0.013	0	0.013	0	0.013	0.013	0	0.011	-0.002
S	0.017	0.026	+0.009	0.026	+0.009					
V	0.20	0.22	+0.02	0.215	+0.015	1.20	1.3	+0.1	1.18	-0.02
Cr	0.44	0.51	+0.07	0.463	+0.023	4.06	4.5	+0.4	3.86	-0.20
Mn	0.33	0.38	+0.05	0.330	0	0.31	0.36	+0.05	0.315	+0.005
Co						9.19	9.40	+0.21	9.10	-0.09
Ni						0.054	0.059	+0.005	0.055	+0.001
Cu	0.019	0.026	+0.007	0.022	+0.003	0.032	0.044	+0.012	0.04	+0.015
Mo	0.082	0.09	+0.008	0.071	-0.011	0.058	0.61	+0.05	0.559	-0.001
W	3.28	3.55	+0.27	3.17	-0.11	16.94	18	+1.06	16.8	-0.14

Table 3 Order analysis of fuel oil using liquid sample holder
(corrections for atmosphere, analysis window, impurities)

		S (%(m/m))	Fe (ppm)	Ni (ppm)
	Standard value	0.19	1	<1
No correction	Order analysis value	0.151	9	15
	Difference	-0.039	+8	+14
Corrections for atmosphere, analysis window	Order analysis value	0.199	10	15
	Difference	+0.009	+9	+14
Corrections for atmosphere, analysis window, impurities	Order analysis value	0.197	1	<1
	Difference	+0.007	0	

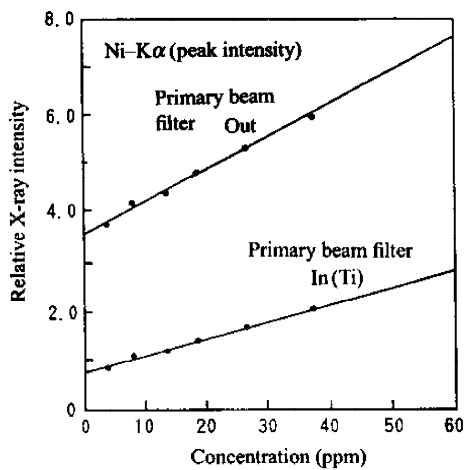


Fig. 6 Calibration curve of Ni in fuel oil (effect of Ti filter)

Analysis of Ni in fuel oil

	Primary beam filter Out	Primary beam filter In(Ti)
S/N	1.20	2.62
Detection limit	0.19 ~ 0.24	0.17 ~ 0.22

Measured sample: S265 (Ni: 37ppm)

Standard Rh X-ray tube, 50kV-60mA
Holder analysis window: t = 6 μmpp film
Analyzing crystal : LiF - SC

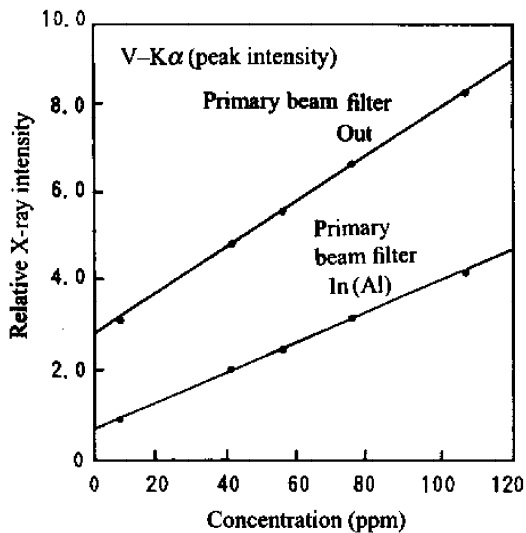


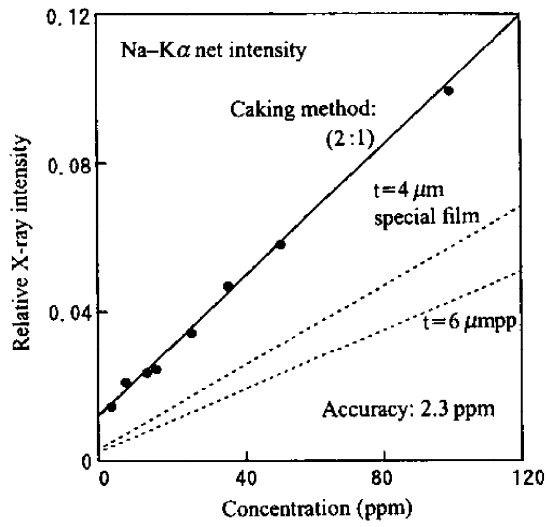
Fig. 7 Calibration curve of V in fuel oil (effect of Al filter)

Analysis of V in fuel oil

	Primary beam filter Out	Primary beam filter In(Ti)
S/N	2.31	5.77
Detection limit	0.21 ~ 0.28	0.18 ~ 0.22

Measured sample: S265 (V: 107ppm)

Standard Rh X-ray tube, 50kV-60mA
Holder analysis window: t = 6 μmpp film
Analyzing crystal : LiF - PC



Standard Rh X-ray tube, 30 kV-100 mA
 Analyzing crystal: RX-35, He substitution
 Sample : Caking agent=2:1

Fig. 8 Calibration curve of Na in oil (caking method)