

X-ray Diffractometer

# XRD-7000 S/L XRD-7000 S/L OneSight



# New Concept in Multifunction X-Ray Diffractometry for the 21st Century

Shimadzu X-ray Diffractometer

## XRD-7000 S/L

## XRD-7000 S/L OneSight™



# Handles Extra-Large Samples and Liquid Samples Features a High-Precision Vertical $\theta$ - $\theta$ Goniometer

The XRD-7000 Series X-ray diffractometers feature a high-precision vertical  $\theta$ - $\theta$  goniometer and are able to handle large samples up to W400 × D550 × H400mm.

In addition to basic qualitative and quantitative analysis, the XRD-7000 Series handles residual austenite quantitation, environmental quantitative analysis, precise lattice constant determination, degree of crystallinity calculations, crystallite size and crystal strain calculations, crystal system determination, as well as other software-based crystal structure analysis. The addition of attachments permits measurement of stress, heated samples and thin films. The range of applications becomes even wider by using the powerful parallel beam optical system equipped with wide-range high-speed detector and polycapillary unit.

## Features

### High-Precision Vertical $\theta$ - $\theta$ Goniometer

The high-precision vertical  $\theta$ - $\theta$  goniometers used in the XRD-7000 Series boast a minimum step size of 0.0001°. Select from the L model to analyze large samples or the general-purpose S model. Both models feature a variable goniometer radius to handle the analysis of any sample.

### Comprehensive Range of Options Expands the System

To match the aim of the analysis, options for the measurement of thin films, stress, or heated samples can be combined with the new strong parallel beam optical system with built-in polycapillary unit.

### Windows® 10-Compatible

The system uses the stable Windows® 10 software platform, resulting in excellent multitasking and networkability. The unit control and data processing software is based on the highly regarded XRD-6100 software with enhancements to improve ease of operation.

### Safety-First Design

The casing incorporates the same door lock mechanism as the XRD-6100 and was designed with safety from X-ray exposure in mind.

### OneSight Wide-Range High-Speed Detector Available

The OneSight is a wide-range high-speed detector that consists of a number of semiconductor devices. It is able to achieve an intensity more than 100 times higher than a scintillation detector.

# Applications of X-Ray Diffractometer



## Ferrous Metals

### Steel

Qualitative analysis of steel sheet, measurement of residual austenite and residual stress, analysis of friction and wear test samples, measurement of iron oxide films and nitride layers, evaluation of plating and texture.

### Cast Iron

Qualitative analysis of precipitates and additives in cast iron, etc.

### Surface-Treated Steel

Evaluation of characteristics of surface-treated areas, quality control, residual stress measurement.



## Non-Ferrous Metals

### Copper and Zinc

Qualitative analysis of alloys, orientation measurements of foil samples, evaluation of texture, qualitative analysis of plated areas, etc.

### Aluminum

Qualitative analysis (aluminum, aluminum alloys, oxides and nitrides), evaluation of texture in rolled material.

### Other Metals

Qualitative analysis (metallic compounds, oxides and nitrides), characteristic evaluation of surface-treated areas, residual stress measurement.



## Machinery, Automobiles and Shipbuilding

### Machinery

Qualitative analysis of tool steels, surface condition analysis of machined parts, analysis of austenite layers, qualitative analysis of surface plating, residual stress measurement.

### Automobiles and Shipbuilding

Qualitative analysis of structural parts surface, quantitative analysis of austenite, residual stress measurement, qualitative analysis of exhaust gas catalysis, etc.



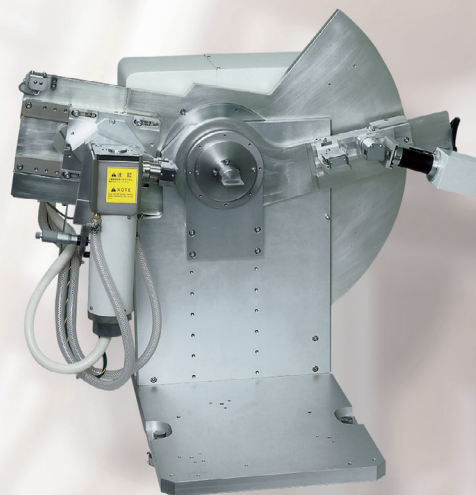
## Chemicals and Catalysts

### Chemicals

Qualitative analysis of organic and inorganic chemicals, impurity analysis.

### Catalysts

Qualitative analysis and measurement of crystallinity, measurement of crystallite size to check final product.



Model S Vertical  $\theta$ - $\theta$  Goniometer



## Ceramics

### Porcelain and Ceramics

Qualitative analysis of raw materials, final product evaluation, analysis of crystal structures during heating (crystal system, crystallite size, lattice constant).

### Cement and Glass

Qualitative and quantitative analysis of clinker and cement (free lime, etc.), qualitative analysis of raw materials. Qualitative analysis and orientation measurements of thin film layers formed at the glass surface.



## Pharmaceuticals and Medical Treatment

### Pharmaceuticals

Qualitative analysis of pharmaceutical raw materials and identification of impurities. Crystal polymorphism analysis and crystallinity measurements, quality control during pharmaceutical manufacture using crystallite size measurement, final product quality check, crystal polymorphism analysis related to patents.

### Dental Materials

Qualitative analysis of dental materials such as apatite, etc.



OneSight Wide-Range High-Speed Detector  
for XRD-6100/7000



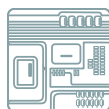
## Resources and Energy

### Coal, Oil and Natural Gas

Plant-scale qualitative analysis, evaluation of carbon materials, evaluation of catalysts during petroleum refining.

### Rocks and Minerals

Qualitative analysis of raw materials and identification of impurities. Qualitative/quantitative analysis of asbestos minerals (compatible with PRTR method).



## Electrical and Electronic Materials

### Electrical Components

Qualitative analysis of corrosion and adhering matter on electrical contacts. Stress measurements in structural parts, qualitative analysis of plated parts, etc.

### Electronic Components

Qualitative analysis and orientation measurements of thin-film electronic materials. Measurement of substrate crystal orientation for magnetic heads and electronic elements.

### Battery Materials

Crystal structure analysis of battery materials.



## Construction and Civil Engineering

Qualitative/quantitative analysis of asbestos and free silicic acids in construction materials, qualitative analysis of construction materials such as tiles and bricks.



## Environment and Industrial Wastes

### Environment

Qualitative/quantitative analysis of asbestos and free silicic acids in the work environment. Qualitative analysis of dust.

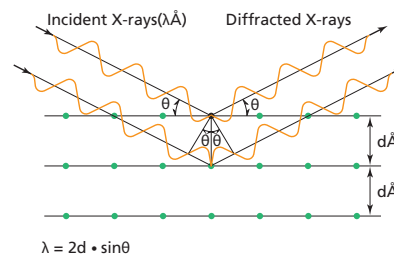
### Industrial Waste

Qualitative analysis of residual matter in plating liquids, combustion ash, coal ash, and furnace slag.

# Basic Construction is Corresponded with a Wide Range of Applications

## Principle of Operation

The XRD-7000 is an X-ray diffractometer that analyzes crystalline states under normal atmospheric conditions. Furthermore, this method is non-destructive. X-rays focused on a sample fixed on the axis of the spectrometer (goniometer) are diffracted by the sample. The changes in the diffracted X-ray intensities are measured, recorded and plotted against the rotation angles of the sample. The result is referred to as the X-ray diffraction pattern of the sample. Computer analysis of the peak positions and intensities associated with this pattern enables qualitative analysis, lattice constant determination and/or stress determination of the sample. Qualitative analysis may be conducted on the basis of peak height or peak area. The peak angles and profiles may be used to determine crystalline size and degree of crystallization, and are useful in conducting precise X-ray structural analysis.



## Fields of Application

Ferrous metals, non-ferrous metals, machinery, shipbuilding, welding, automobiles, ceramics, cement, glass, catalysts, electric components, electronic materials, magnetic materials, battery materials, textiles, paper and pulp, foods, chemicals, pesticides, dyes, pigments, paints and ink, pharmaceuticals, dental materials, biological materials, oil and coal, electrical power, gas, ores, soil and rocks, clays, minerals, construction and civil engineering, environment, industrial waste products.

## Construction

### X-ray protective housing

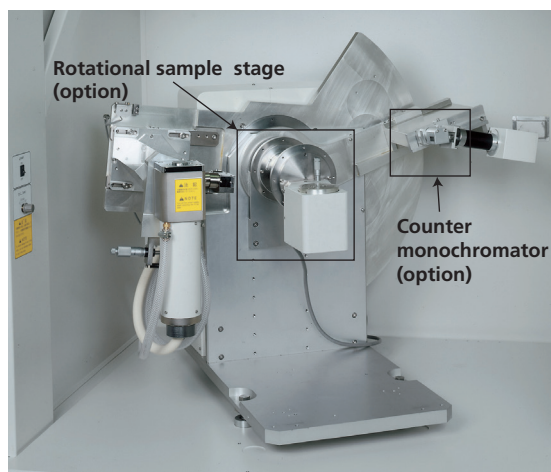
The front door is mounted on guide rollers to enable extremely light-touch and smooth door opening to facilitate installation/exchange of samples and attachments. A magnet latch assures certain door closing, and to further ensure safety, a door interlock mechanism is automatically activated whenever X-rays are generated.

### High-precision, vertical $\theta$ - $\theta$ goniometer

High-speed rate (1000°/min) and high-precision angle reproducibility (0.0002°) provide fast measurement and highly reliable data. The  $\theta$ - $\theta$  vertical goniometer unit allows analysis of samples in various states, substantially widening the application range. The drive mechanism features an independent dual axis  $\theta$ - $2\theta$  linkage drive, and independent  $2\theta$  and  $\theta$  axis drives, freely selectable for efficient thin film and various other types of analysis.

### High-voltage transformer for high output X-ray tube

The high-voltage transformer supports either the 2.2kW high output fine focus X-ray tube or 2.7kW high output broad focus X-ray tube.



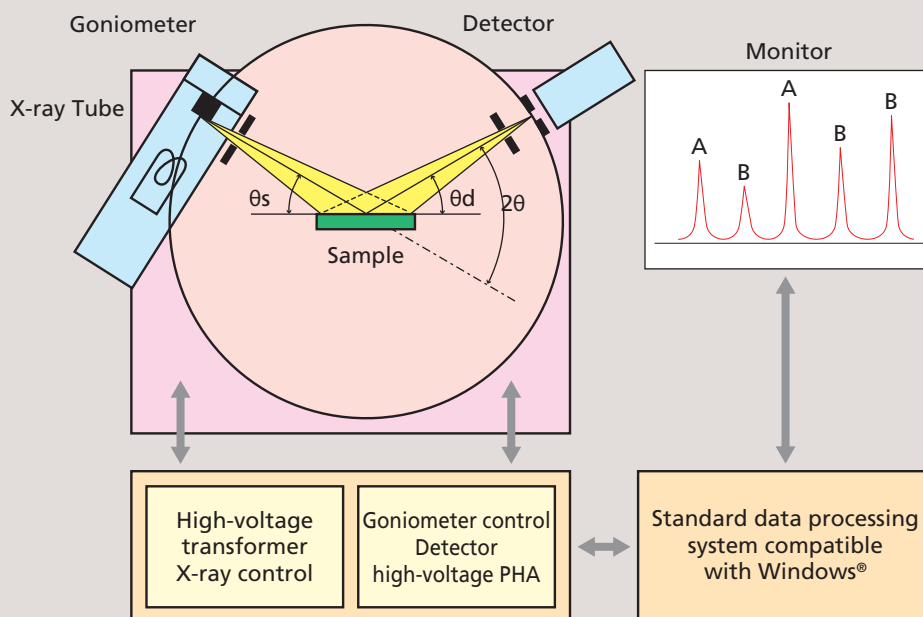
High-precision  $\theta$ - $\theta$  Goniometer

## X-ray tubes

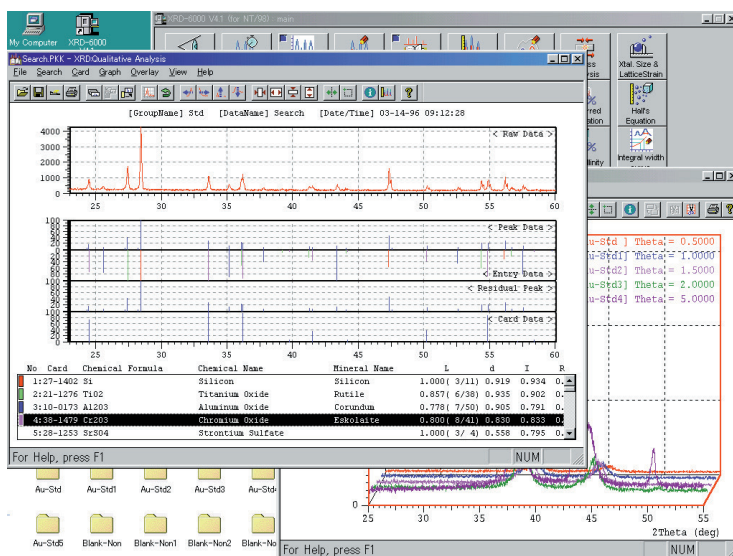
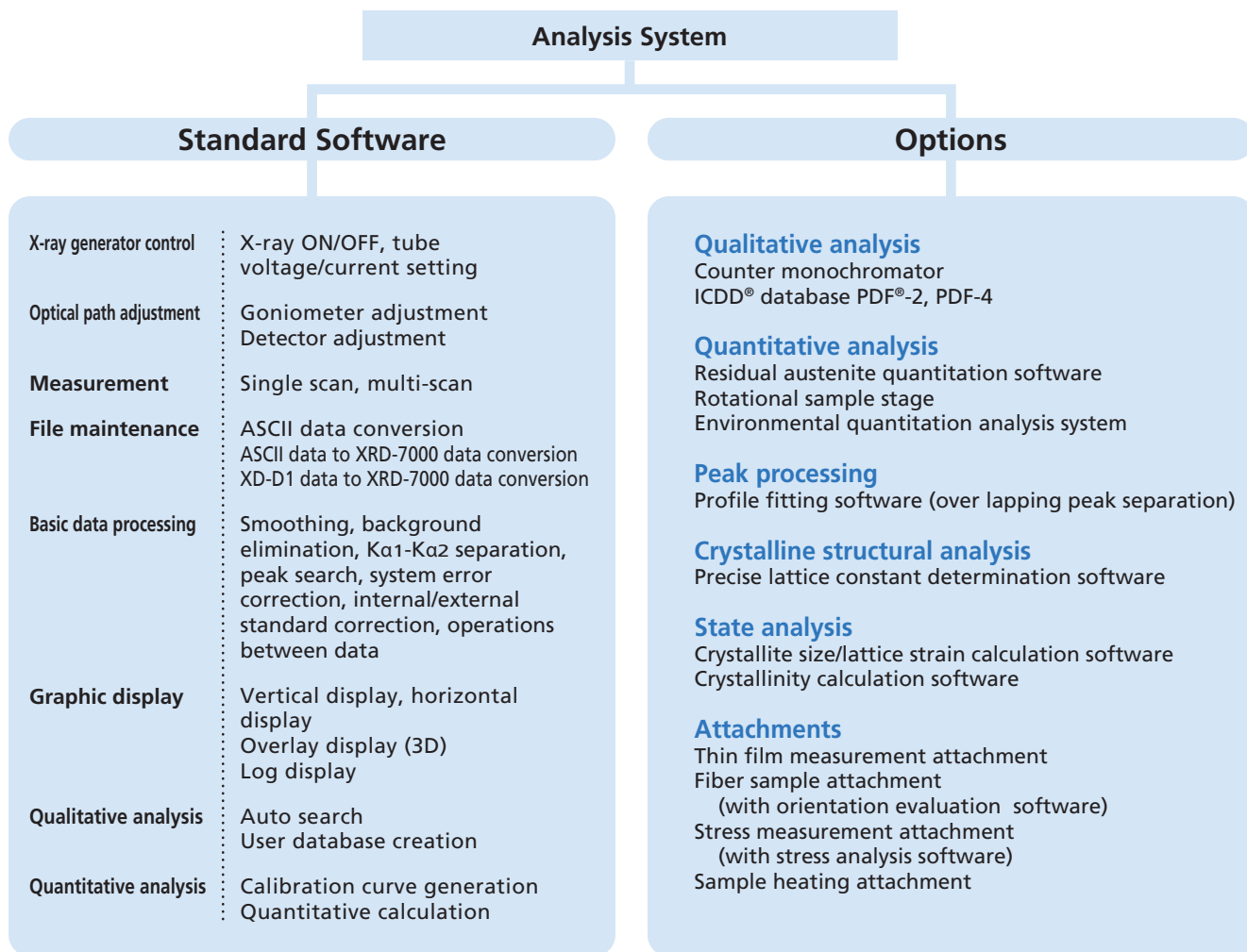
The XRD-7000 will accept various types of X-ray tubes, including the normal focus (NF) 2kW type and broad focus (BF) 2.7kW type, which are standard accessories, as well as the optional long fine focus (LFF) 2.2kW type. By attaching the optional counter monochromator, all types of samples, including Fe samples, can be analyzed using the standard Cu X-ray tube.

## Highly stable X-ray generator

Shimadzu's extensive experience in producing high-performance X-ray generators has provided an X-ray generator of high stability, with tube voltage and tube current both stable to within  $\pm 0.01\%$  with respect to 10% voltage fluctuation. This stability is unaffected during fluctuation of source voltage or ambient temperature, ensuring high reliability of data even during prolonged periods of data acquisition.



# Providing a Complete Analysis System



Auto Search Results and Thin Film Sample Overlay Display

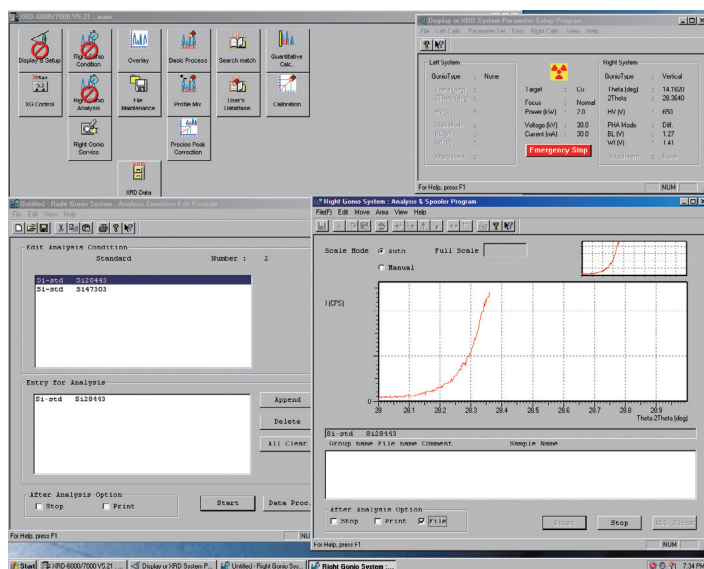


# Automatic Measurement, Easy Operation

## Measurement Display

Sample measurement conditions can be set easily.

The scheduling and the progress condition of the measurement can be confirmed at one view by the analysis & spooler.

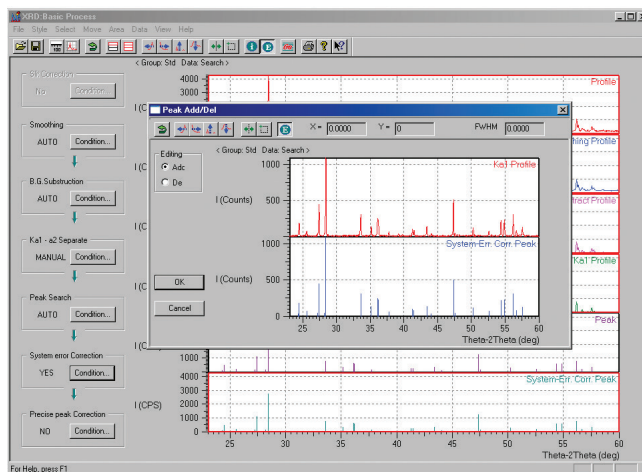


Measurement Screen

# Multitasking Enhances Analysis Efficiency

## Basic Data Processing

The multitasking capability provided with the Windows® operating environment allows measurement and data processing to be conducted simultaneously, enhancing the efficiency of analysis operations.

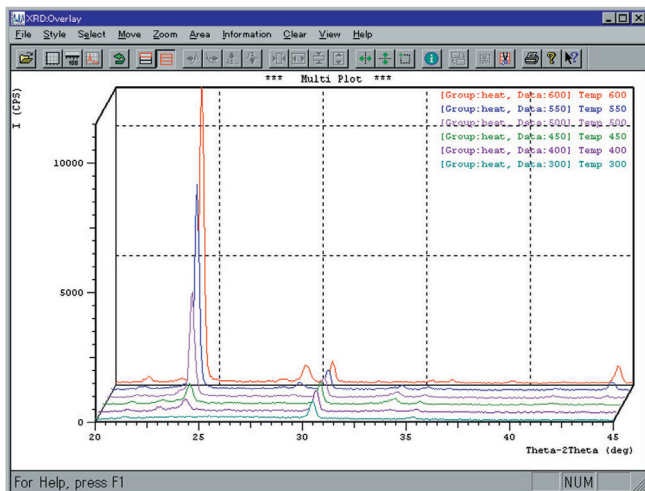


Basic Data Processing Screen

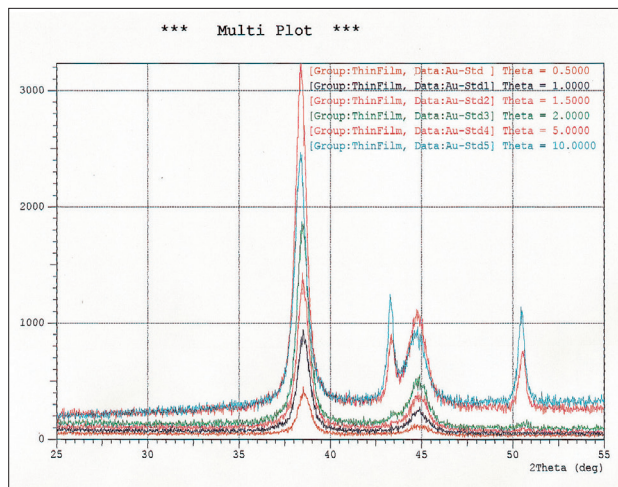
# Comfortable Data Processing Environment

## Graphic Display

Data can be freely zoomed with a click of the mouse, so profile comparison of thin film data or heating measurement data can be easily accomplished using a combined 2-dimensional or 3-dimensional display. The software also features a variety of other useful graphic functions, such as intensity Log conversion display and hidden-line processing on the 3-dimensional display. Each type of data can be output to the color printer, so differences between samples can be recognized at a glance.



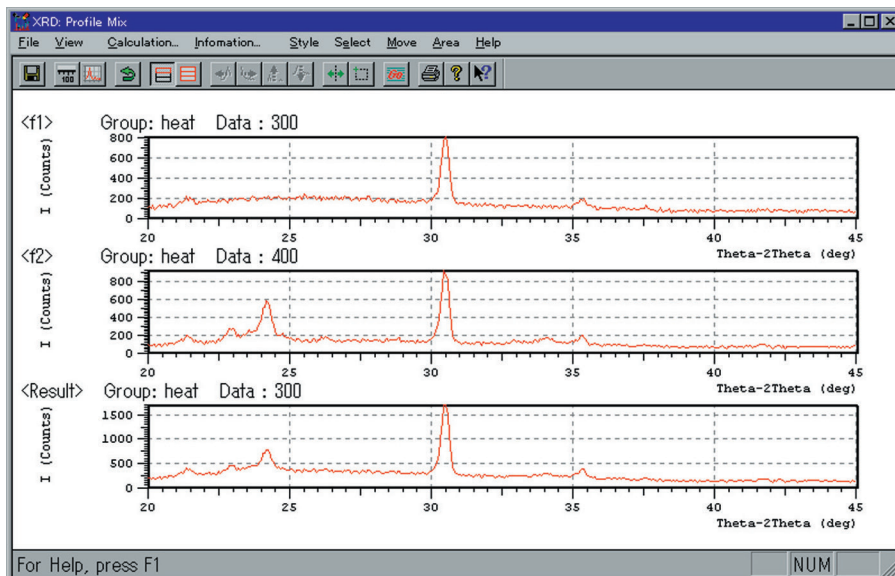
3-Dimensional Screen of Thin Film Sample



2-Dimensional Output of Thin Film Sample

## Addition/Subtraction Operations

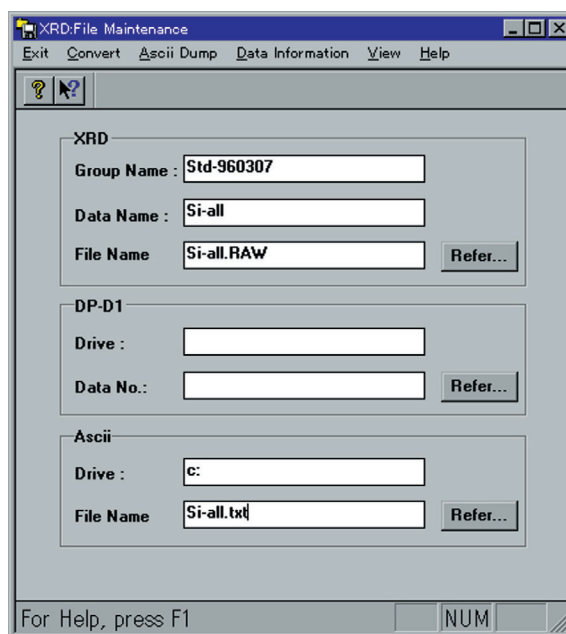
Data manipulation functions such as deletion of unnecessary peak profiles and addition of duplicate measurement data to obtain a summed profile are some of the invaluable tools available for conducting efficient data analysis. Profile calculations are conducted as shown in the window below.



Profile Calculation Window

## File Maintenance –Data Format Conversion–

With the XRD-7000, data measured by other X-ray diffractometers can be converted into files to enable analysis using this data processing software. The data from Shimadzu's X-ray diffractometers XRD-6000/6100 can be used without alteration and a dedicated file conversion window has been made available for the XD-D1 model. In the case of other X-ray diffraction data, 2 $\theta$  angle and X-ray intensity text files (ASCII data) can be converted into XRD-7000 data format. In reverse, raw data measured by the XRD-7000 also can be converted into text files. Furthermore, processed data including peak data, as well as raw data, can be converted to text format, facilitating data processing in customized formats.

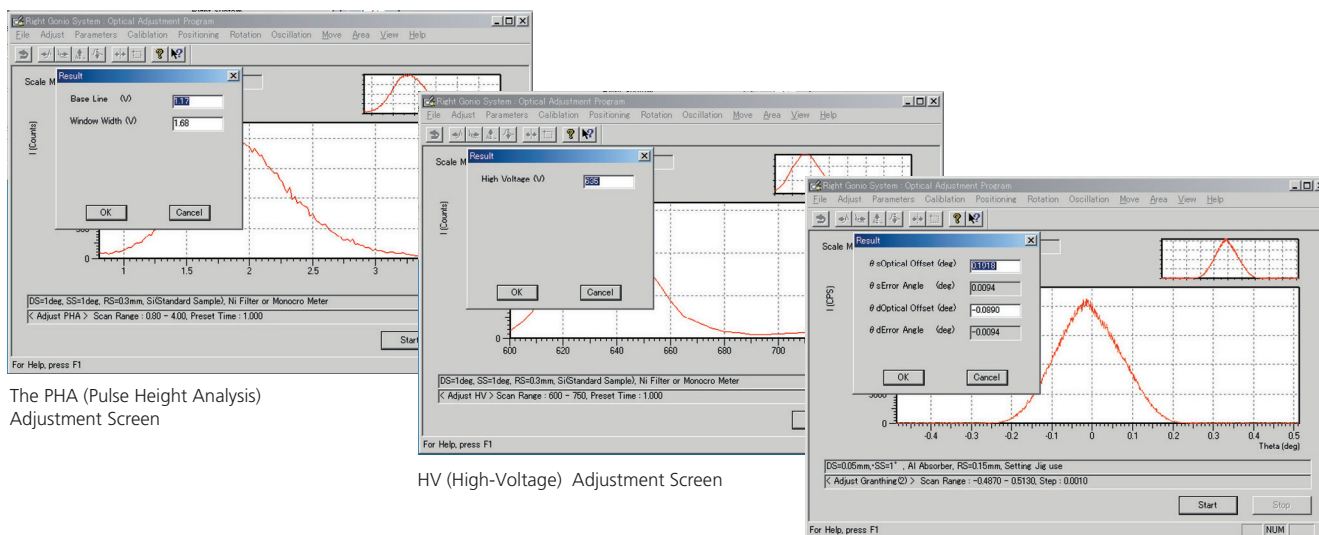


File Conversion Window

## Fully automatic goniometer optical system adjustment with automatic saving of adjustment data.

### Optical System Adjustment

The XRD-7000 system makes fully automatic optical adjustments to the goniometer from the computer screen, even for optional attachments. In addition to completely automatically adjusting all settings, such as the zero angle for the  $\theta$ s and  $\theta$ d axes, the X-ray detector high-voltage settings, the PHA baseline and window width settings, it automatically saves the settings information. This feature can be utilized for routine maintenance.



The PHA (Pulse Height Analysis) Adjustment Screen

HV (High-Voltage) Adjustment Screen

$\theta$ s/ $\theta$ d-axis Adjustment Screen

# Enhanced Auto Search System

[ Auto Search, General Quantitation Software Provided as Standard ]

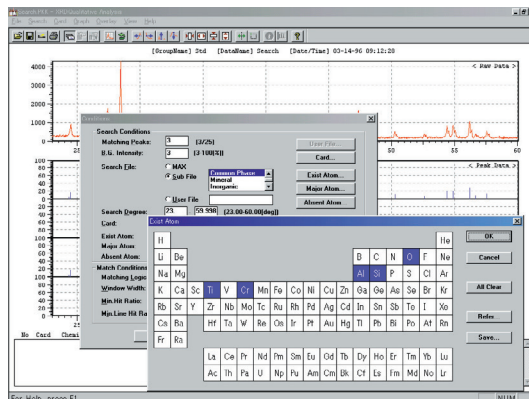
Identification work can be performed efficiently on screen.

## Detailed search parameters can be set.

To obtain correct results with automatic search/match, search parameters that conform to each sample must be set. The XRD-7000 enables the setting of detailed search parameters such as selection of files to be used in the search and three levels of element data input.

## Replete with second search function for authoritative identification of small amount of components.

Identifying a small amount of components with a primary search is difficult; a second search is often needed after the major components have been identified. The XRD-7000 comes replete with a second search function to provide easy identification of a small amount of components.

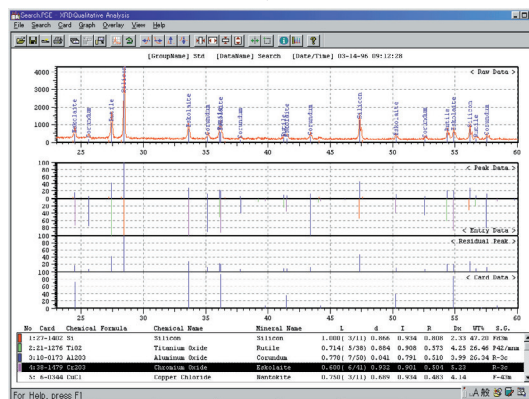


Search Parameter Setting Screen

## Various search result data can be displayed.

Search results can be stack-displayed with each standard data display over raw data. Also, for easy comparison, standard substance names, chemical equations, ore names, Miller indices, and numbers can be displayed on each peak. Furthermore, an easy quantitative calculation function using a corundum ratio for candidate substances (intensity ratio for the  $\alpha$ - $\text{Al}_2\text{O}_3$  strongest peak) is included in the equipment.\*

\* When using this function, please obtain the ICDD database separately.



Search Result Screen

## Dedicated user database can be created.

The user's very own database file - separate from the sub-file supplied by ICDD (International Center for Diffraction Data) - can be created. Data obtained through measurements by the XRD-7000 and manually entered data can be registered as they are in the database file, which means that the user's basic samples can be registered, and comparisons made with those substances to provide an extra dimension to quality control.

No.	JCPDS #	Chemical Formula	Mineral Name	3-Strongest lines...d(1)	q.m.
1	10-0173	$\text{Al}_2\text{O}_3$	Corundum, syn	2.02(100) 2.55(90) 1.50(14)	*
2	33-0820	$\text{Al}_2\text{O}_3$	Corundum, syn	2.05(100) 1.50(25) 1.45(14)	*
3	04-0787	Al	Aluminum, syn	2.34(100) 2.02(47) 1.22(24)	*
4	04-0788	Au	Gold, syn	2.36(100) 2.04(52) 1.23(36)	*
5	04-0789	Ag	Silver- $\beta$ -ITCNO	2.36(100) 2.04(48) 1.23(36)	*
6	38-1420	TiN	Sassonite, syn	2.12(100) 2.45(72) 1.50(45)	*
7	06-0696	Fe	Iron, syn	2.03(100) 1.17(38) 1.43(28)	*
8	31-0619	(Fe,C)		2.08(100) 1.80(88) 1.08(100)	o
9	26-1079	C	Graphite- $\beta$ ITR	3.35(100) 2.08(11) 1.96(9)	o
10	11-0865	CrH	Carlsbergite, s	2.07(100) 2.39(88) 1.46(88)	h
11	06-0694	Cr	Chromium, syn	2.04(100) 1.16(39) 0.93(20)	*
12	03-0727	Co	Cobalt, syn	1.91(100) 1.25(98) 1.15(98)	h
13	04-0836	Cu	Copper, syn	2.09(100) 1.81(46) 1.28(20)	*
14	35-0821	Hg		2.45(100) 2.61(36) 2.78(25)	*
15	30-0794	HgO		3.05(100) 2.04(100) 1.17(100)	o

User Database Creation Screen

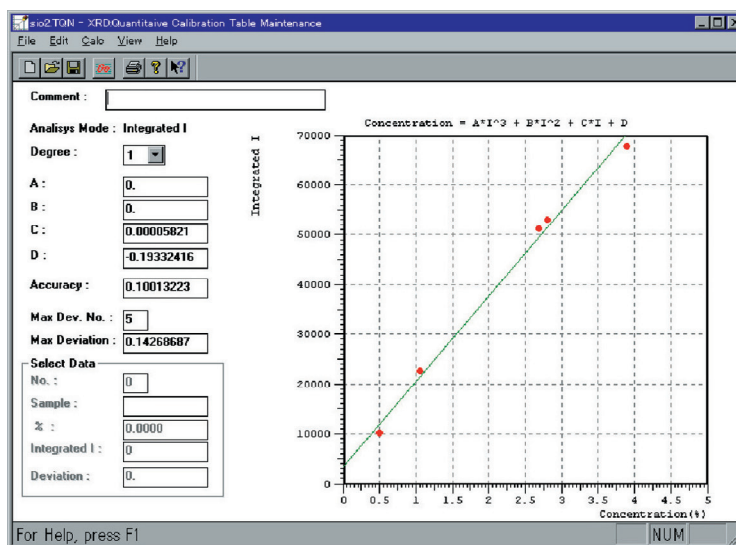
# Sophisticated Quantitation Software

[ Satisfies your analysis objectives ]

## Creating Calibration Curves

Calibration curves can be generated for intensity, integrated intensity or intensity ratio.

Intensity and integrated intensity calculations are used for the internal standard and standard addition methods. Intensity ratio calculation is used for 2 components peak.

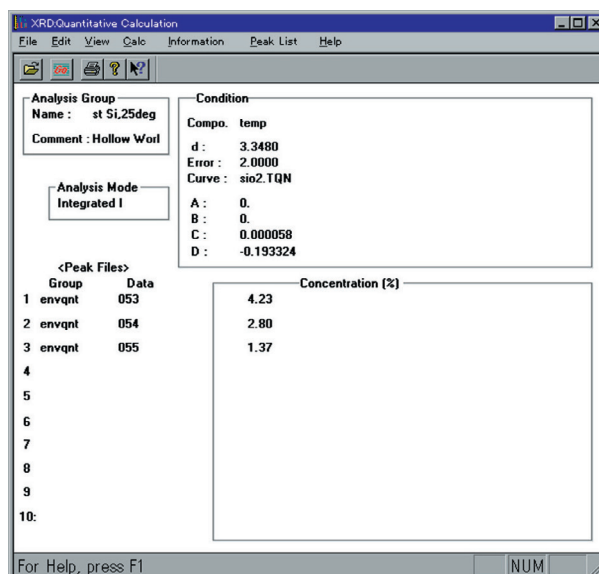


Calibration Curve Screen for Integrated Intensity

## Quantitative Analysis

The internal standard method and 2 intensity methods are available to satisfy most of the application needs.

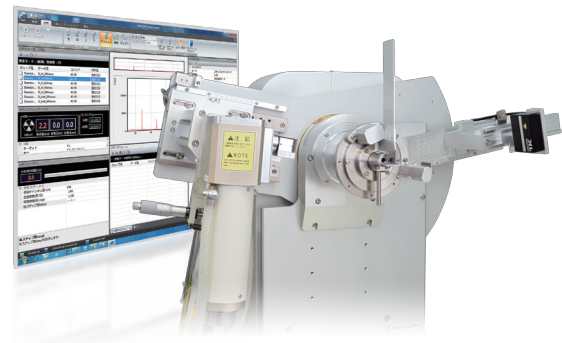
Further, up to 5 peaks may be specified for quantitation and up to 10 sets of data may be calculated simultaneously.



Integrated Intensity Quantitation Results Screen

# OneSight Wide-Range High-Speed Detector for High-Speed and High-Sensitivity Measurement

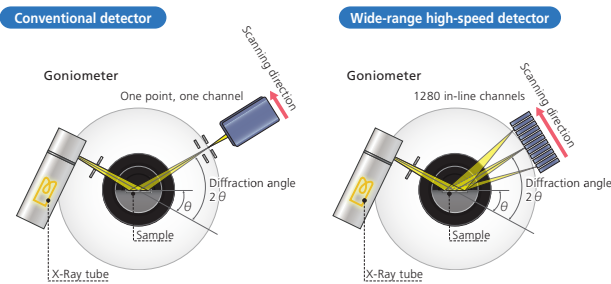
The OneSight is a wide-range high-speed detector consisting of a number of semiconductor devices. The wider angle of acquiring diffraction lines allows measurement to be performed with the goniometer fixed. By offering high-speed, high-sensitivity measurements, the time required for qualitative and quantitative analysis can be significantly reduced. The OneSight can be mounted on existing XRD-6100/7000 units installed at customers' sites.



## Wide-range array detector with 1280 channels

A conventional scintillation detector has only one channel at one point whereas the OneSight has 1280 channels on a wide-range array.

Thus, compared with scintillation detectors, this detector can acquire diffraction lines over a wide angle at one time.



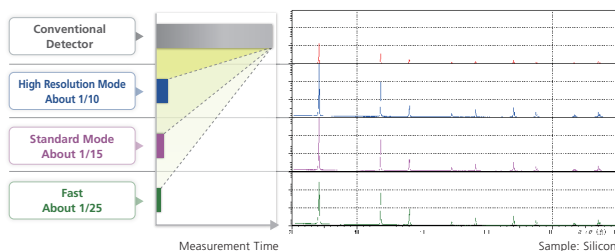
## Advanced user interface enhances operational efficiency.

The measurement software for the OneSight has been redesigned. The analytical profile is located in the center, the analytical conditions list and instrument status display are indicated on the left, the analysis schedule is displayed on the bottom center, and the detailed analysis conditions display is shown on the right. The window layout can also be changed. By displaying necessary information in one window, the new design makes it easy for a user to understand the measurement status at a glance.

- Analysis Profile Display Window**: Displays the profile window. Allows both zoom in and out.
- Analytical Conditions Detailed Display and Setting Window**: Allows users to edit the detailed conditions of the analytical conditions file. Displayed when editing conditions.
- Analytical Conditions File Display and Editing Window**: Allows analytical conditions to be read, edited and new conditions to be created.
- Instrument Status Display Window**: Displays the status of the X-ray generator, goniometer, and other units.
- Analysis Progress Status Window**: Displays the OneSight status and analysis progress. An analysis progress bar is displayed so that users can check the status at a glance.
- Analytical Conditions Registration Window**: Displays the registered analytical conditions file. Users can check or change the analysis schedule based on this file.

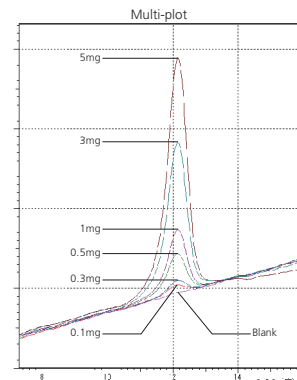
## High-speed quantitative analysis using three measurement modes

The OneSight features three kinds of measurement modes: High resolution, Standard, and Fast. It enables measurement speeds that are 10 times faster (High resolution), 15 times faster (Standard), and 25 times faster (Fast) than those attained with a conventional scintillation detector.



## ONE SHOT mode achieves simultaneous measurement of diffraction profiles at a specific angle range.

The OneSight can perform a simultaneous diffraction profile measurement over a range of more than 10 deg. with a fixed-position goniometer. This is useful in quantitative analysis using a specific diffraction peak.



Standard Sample Data of Asbestos (Chrysotile)  
(30 sec. measurement time per sample)

## Qualitative Analysis

### Counter Monochromator

Qualitative analysis (identification) is the most common analysis conducted with X-ray diffractometers. The counter monochromator is an attachment that enhances the accuracy of qualitative analysis. The counter monochromator obtains data with a good S/N ratio. A combination of Cu X-ray tube and Cu tube monochromator can cut the fluorescent X-rays from Mn, Fe, Co, and Ni samples and is applicable to a wide range of sample types.

Note: This attachment cannot be used in combination with OneSight wide-range high-speed detector.

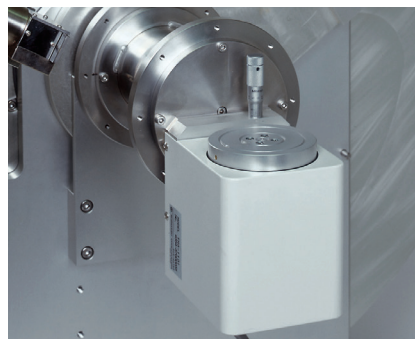


## Thin Film Measurements

### Thin Film Measurement Attachment

This attachment is effective for the measurement of samples with sub-micron thickness. Conducting parallel beam diffractometry at low angles of incidence restricts the penetration of incident X-rays into the underlying substrate to achieve high-sensitivity X-ray analysis of the surface layer only. In addition, by conducting measurements while changing the angle of X-ray incidence, the thin film thickness can be approximately determined by detecting the angle at which diffracted X-rays from the substrate are detected. The sample stage incorporates a rotation mechanism that permits orientation measurement of the thin film layer.

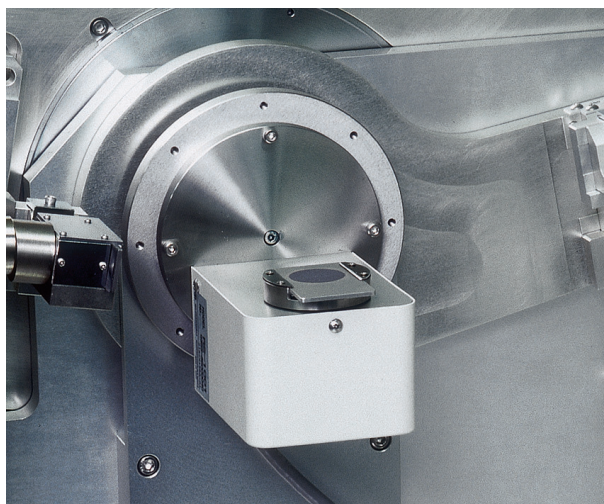
Note: This attachment cannot be used in combination with OneSight wide-range high-speed detector.



## Quantitative Analysis

### Rotational Sample Stage/Environmental Measurement Stage

Rotational sample stage is effective for quantitative measurement precisely because the X-Ray diffraction from the sample can be detected more efficiently and the influence of orientation when loading the sample can be reduced by rotation of stage. Environment measurement stage can measure the quantitative analysis for compliance with the "work environment quantification method" for measurement of asbestos and free silicic acid in a work environment.



Rotational Sample Stage

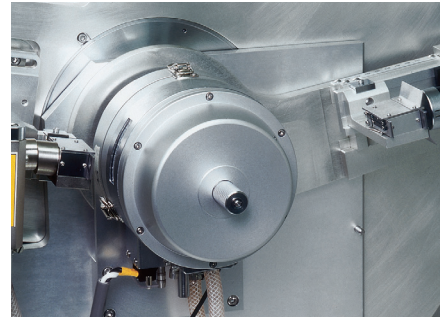


Environmental Measurement Stage

## Heated Sample Measurement

### ■ Sample Heating Attachment

The sample heating attachment allows analysis of samples at various temperatures. This system is used to heat sample during X-ray diffractometry to study the influence of heat on the crystalline structure. (e.g. Lattice constant)



## High Sensitivity Analysis

### ■ Polycapillary Unit

The polycapillary unit is a new optical X-ray element that splits a single X-ray beam emitted from a point light source into multiple X-ray beams using three-dimensionally arranged capillary optics to create a powerful parallel beam output that covers a large area.

- 1) Compared to conventional methods, this unit uses the X-ray more effectively and increases the intensity of the diffracted X-ray, allowing more sensitive analysis.
- 2) With conventional methods, variations in sample surface height are directly translated into variations in X-ray diffraction angles. This polycapillary unit uses parallel beams, so it is not affected by variations in sample surfaces.

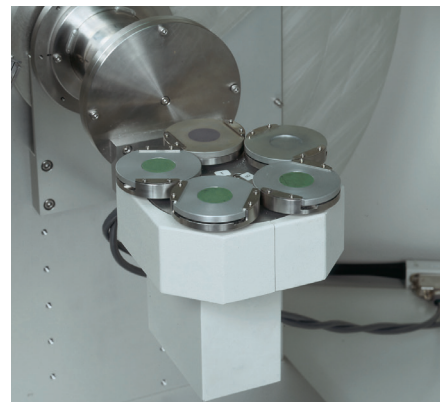


Note: This attachment cannot be used in combination with OneSight wide-range high-speed detector.

## Automatic Analysis

### ■ Auto Sample Changer for 5 Samples

This stage automatically changes samples for measurement. Up to five samples can be loaded for fully automatic qualitative analysis or other measurements.



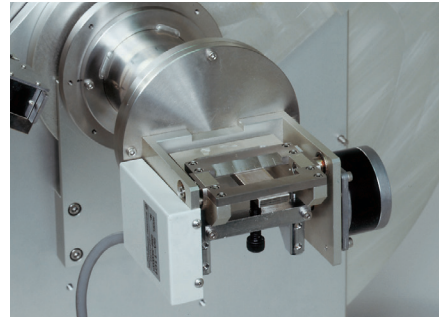


## Stress Measurement

### ■ Stress Measurement Attachment

Stress measurement using X-ray diffraction is non-destructive and permits the measurement of residual stresses, making it a widely used method for the performance evaluation and quality control of mechanical parts.

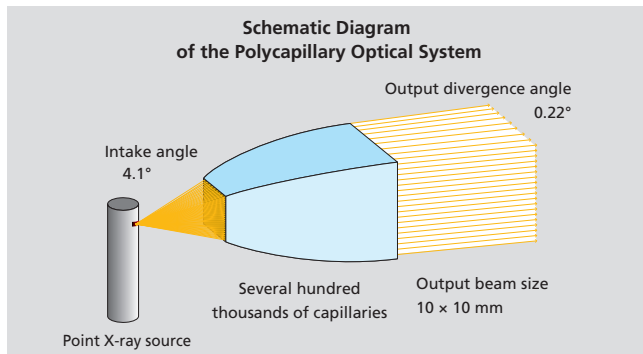
The stress measurement attachment permits measurement by both the side-inclination and iso-inclination methods to achieve stress measurements in mutually perpendicular directions at the same position. The side-inclination method permits measurements in depressions in a sample, such as at the root of gear teeth.



# Polycapillary Optical System

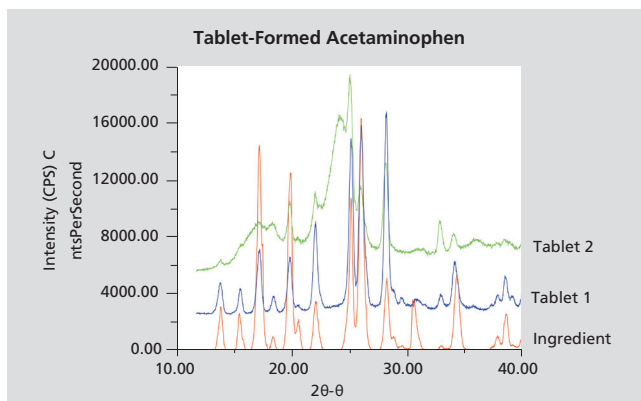
## Principle of the Polycapillary Optical System

The fine glass capillaries in the order of several microns are arranged in a solid as guides to multiple X-rays. The X-rays pass along each capillary while repeating total internal reflection and exit from the opposite end of the polycapillary system. The capillaries are curved so that repeated total internal reflection and the X-rays from the point X-ray source exit the unit as a parallel beam with a large solid angle.



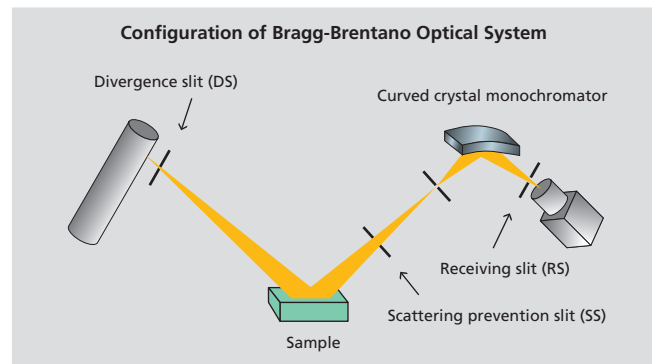
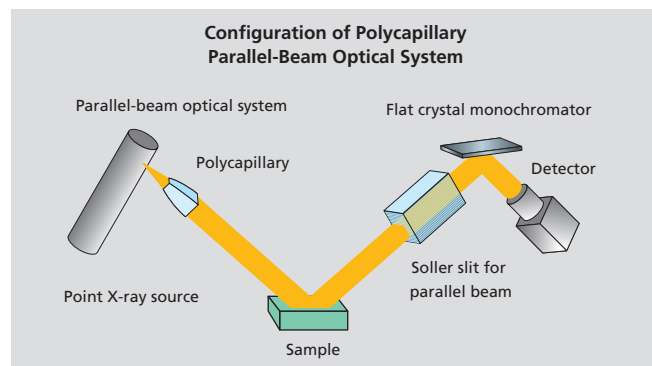
## Sample Measurement Using the Polycapillary Optical System

This example shows measurements of the raw drug acetaminophen and its tablets during the process of manufacturing. Tablets can be directly analyzed to evaluate the degree of crystallinity and crystal polymorphism. The XRD-7000 is able to perform accurate, highly sensitive measurements on irregular surfaces or curved surfaces like this.



## Features of the Polycapillary Optical System

Compared with the conventional focused-beam system and the normal parallel-beam system, the polycapillary optical system more efficiently exploits the beam from the X-ray tube, resulting in higher diffraction X-ray intensity. A displacement of the sample in a Bragg-Brentano optical system can move it outside the focus, causing a significant displacement in height in diffraction angle and a dramatic drop-off in diffraction X-ray intensity. Conversely, a displacement of a few millimeters in a parallel-beam system has no effect on the diffraction angle and a minimal decrease on the diffraction X-ray intensity. Consequently, incorrect loading of the upper and lower sample faces or an irregular sample surface causes no angular displacement and accurate measurement is possible. The parallel-beam system also allows analysis of curved surfaces, something not possible with conventional optical systems.

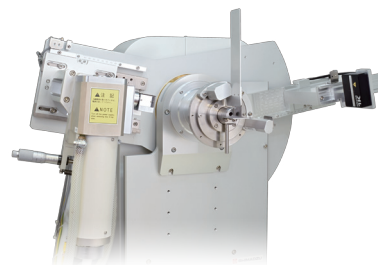


# Options

## High Speed Analysis

### OneSight Wide-Range High-Speed Detector (FD-1001 1D high-speed detector P/N S215-24320-93)

It is an optional detector that can be mounted on existing XRD-6100/7000 units. The wide-range detector consists of 1280 semiconductor devices, and achieves qualitative analysis more than 10 times faster than conventional scintillation detectors. It also features a ONE SHOT mode that takes advantage of the wide measurement angle to perform analysis with a fixed goniometer. Operability is improved by using software that supports measurements made using the OneSight.



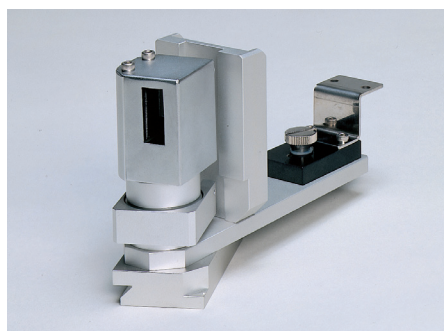
Number of Channels	1280
Strip Width	50 μm
Sensor Area	W64 × L8 mm
Dimensions	W70 × D22 × H62 mm

## Qualitative Analysis

### Counter Monochromator

Installed in the X-ray detector unit, the counter monochromator transforms X-rays which have passed through the entrance slit into monochromatic X-rays, allowing only the characteristic X-rays (K $\alpha$  X-rays) to be detected. Exclusion of all other X-rays from the sample, including continuous X-rays and K $\beta$  X-rays as well as fluorescent X-rays, ensures diffraction patterns with a high signal-to-noise ratio.

Part Description	Application	P/N
Counter monochromator CM-3121	Cu X-ray tube	215-22360-02
Counter monochromator CM-3131	Co X-ray tube	215-22360-03
Counter monochromator CM-3141	Fe X-ray tube	215-22360-04
Counter monochromator CM-3151	Cr X-ray tube	215-22360-05



Note: It is not possible to be used in combination with OneSight wide-range high-speed detector.

### ICDD PDF-2

This is the powder X-ray diffraction database provided by ICDD. PDF-2 contains substance name, chemical formula, d-I data. Furthermore, it also contains miller indices, lattice constants, space groups and other crystallographic information. Using additional PDF-2 Automatic Search Software, unknown substances may be easily identified via the registered crystallographic information.

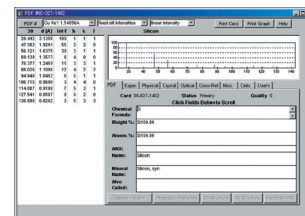
ICDD PDF-2	P/N for Educational institutions	P/N for Other uses
Single license	239-50002-12	239-50002-11

Note: The license (before 2017) is valid for five years. It can be extended for five more years for free at the time the license period ends. The license since 2017 is not available for extending for five more years.

### PDF-2 Data Mining Software

Searches can be performed from the card No., as well as based on multiple elements using "AND" or "OR" conditions, with analyte identification and crystalline structure obtained simultaneously.

Note: Included in PDF-2 Database.



Search Result Screen by Searching Card Number

### ICDD PDF-4

In addition to the functions of PDF-2, database PDF-4 has the display of 3D structural chart, various lattice parameters, simulation wave form by the calculation, and the import of the measurement data.

There are two kinds of databases: PDF-4+ (for general) and PDF-4/Organics (for organics).

ICDD PDF-4+	P/N for Educational institutions	P/N for Other uses
Single license (New, 1-year license)	239-50015-02	239-50015-01
Single license (Renewal, 1-year license)	239-50015-04	239-50015-03
Single license (Renewal, 3-year license)	239-50015-06	239-50015-05
Single license (Renewal, 5-year license)	239-50015-08	239-50015-07

ICDD PDF-4 / Organics	P/N for Educational institutions	P/N for Other uses
Single license (New, 1-year license)	239-50015-22	239-50015-21
Single license (Renewal, 1-year license)	239-50015-24	239-50015-23
Single license (Renewal, 3-year license)	239-50015-26	239-50015-25
Single license (Renewal, 5-year license)	239-50015-28	239-50015-27

# Options

## Quantitative Analysis

### Rotational Sample Stage RS-1001

The RS-1001 performs in-plane rotation of the sample in combination with oscillation around the goniometer sample axis ( $\theta$ ). It is available to minimize the variation in diffraction pattern intensities attributable to the sample crystalline orientation, and thereby enhance the precision in most types of quantitative analysis.

#### Main specifications

- Rotation  $\beta$  axis (sample in-plane)
- Rotation speed 1 to 60rpm
- Minimum step width 0.1°
- Operation modes Constant speed rotation, oscillation sample in-plane rotation scan (continuous, step)
- Measuring angle range  $2\theta$  5° to 163°

Part Description	P/N
Rotational sample stage (without option driver)	215-21766-01

Note: Please arrange optional additional ASSY with optional driver ASSY at the same time. Please refer to the paragraph of a special accessory on page 29.



### Environmental Measurement Stage RS-2001

A complete environmental analysis system, this comprises a special environmental quantitative analysis stage, filter holder and quantitation software. A special filter holder is provided which allows measurement using an asbestos and free silicic acids imbedded filter just as it is. The main specifications of the environmental stage are the same as those of the general-purpose rotational sample stage. The calibration curve correction is based on Zn, however, when the diffraction line of the sample overlaps with that of Zn; an Al filter holder (optional) is also available.

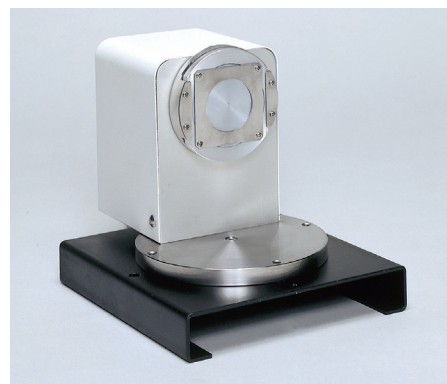
The sample stage option driver can also be used with the rotational sample stage. Zn filter folder ( $\phi 25$ ) is one of the standard accessories of RS-2001.

#### Main specifications

- Measuring angle range  $2\theta$  5° to 163°

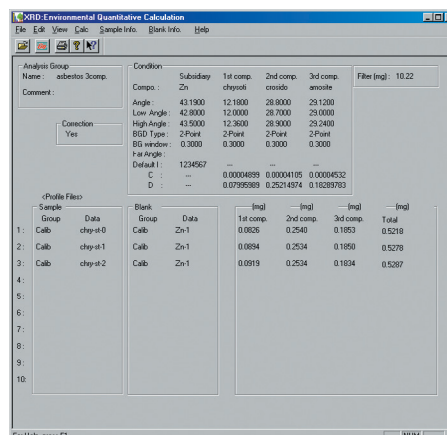
Part Description	P/N
Environmental Analysis Stage (with S/W)	215-21767-03
Al filter holder ( $\phi 25$ )	215-23765-92
Aluminum sample holder (5pc)	215-22507-06
Aluminum sample holder with penetration-hole (5pc, with plated through hole)	215-22507-10

Note: Please arrange optional additional ASSY with optional driver ASSY at the same time. Please refer to the paragraph of a special accessory on page 29.



## Environmental Quantitation Software (P/N 215-00421-92)

Even if a sample such as airborne dust in the work environment is collected by a filter, the amount is small, and even if a general-purpose quantitative analysis method is applied, a reliable calibration curve cannot be obtained due to the effect of matrix substances, and thus, good results cannot be obtained. The environmental quantitation software employs the substrate standard mass absorption correction method to eliminate the effect of X-ray absorption of matrix substances, and provides a calibration curve analysis method with good linearity and high accuracy. Used in combination with the filter holder shown in the table above.



Quantitation Results Screen

## Automatic Analysis

### Auto 5 Position Sample Changer ASC-1001

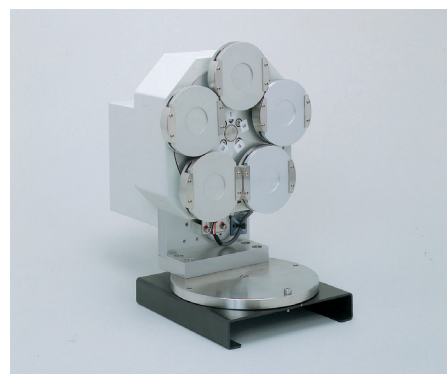
This stage is used in order to automatically measure a maximum of 5 samples. The ASC-1001 performs in-plane rotation of the sample in combination with oscillation around the goniometer sample axis ( $\theta$ ) to minimize the variation in diffraction pattern intensities attributable to the sample crystalline orientation. Also it is possible to avail filter holder (option) for Environmental Measurement Stage RS-2001.

#### Main specifications

- Sample position Max. 5
- Powder Sample Holder 25mm  $\phi$ , 5pc (standard)
- Sample Size Powder: 25mm  $\phi$   
Filter: 25mm  $\phi$  (option)
- Rotation speed 1 to 60rpm
- Measuring angle range 2 $\theta$  5° to 163°

Part Description	P/N
Auto 5 position sample changer (with a option driver unit)	215-23175-01
Zn filter holder (25mm $\phi$ ) 5pc/set	215-23760-91
Al filter holder (25mm $\phi$ ) 5pc/set	215-23760-92

Note: Please arrange optional additional ASSY with optional driver ASSY at the same time. Please refer to the paragraph of a special accessory on page 28.



### Sample Plates for RS-2001 and ASC-1001

Part Description	P/N
Aluminum sample holder (5pc)	215-22507-06
Glass sample holder (5pc)	215-22507-07
Glass Micro sample holder (5pc)	215-22507-08
Non-reflective sample holder (2pc)	215-22507-09

# Options

## Attachments

### ■ Thin Film Analysis Using Attachment THA-1101

This specialized system includes a thin film sample stage, monochromator and suction pump.

Employing the fixed incidence angle, parallel X-ray diffractometry method, penetration of incident X-rays into the substrate sample is limited as much as possible, providing low background, thin film X-ray diffraction patterns.

Samples are easily set in place using the suction pump.

The sample stage option driver can also be used with the rotational sample stage.

#### Main specifications

- Rotation  $\beta$  axis (sample in-plane)
- Rotation speed 1 to 60rpm
- Minimum incidence angle  $0.1^\circ$
- Sample suction pump AC100V, 10W (1 pump)
- Operation modes Constant speed rotation, oscillation, sample in-plane rotation scan, (continuous, step)

Part Description	P/N
Thin film analysis attachment (without option driver)	215-21765-01

Note1: Please arrange optional additional ASSY with optional driver ASSY at the same time. Refer to the paragraph of a special accessory on page 29.

Note2: It is not possible to be used in combination with OneSight wide-range high-speed detector.



## Fiber Sample Attachment

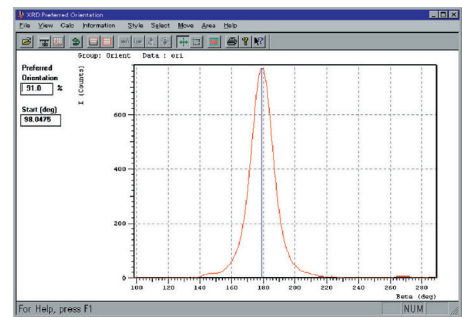
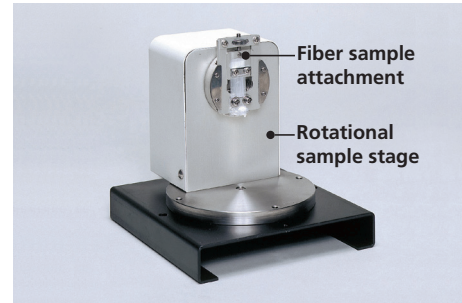
Used in combination with the Rotational Sample Stage (RS-1001), this system measures the degree of orientation for fibers. The acquired data is then processed using the provided fiber sample attachment software to calculate degree of orientation.

Part Description	P/N
Fiber sample attachment (with S/W)	215-22624

Note: It is not possible to be used in combination with OneSight wide-range high-speed detector.

### Orientation evaluation software (P/N215-00428-92)

- This software evaluates the degree of orientation for fiber samples, using the data of peak width at half height acquired from orientation measurement (sample in-plane  $\beta$  axis measurement).



Degree of Orientation Evaluation Screen





## Polycapillary Attachment PCL-1002

The polycapillary unit is a new optical X-ray element that splits a single X-ray beam emitted from a point light source into multiple X-ray beams using three-dimensionally arranged capillary optics to create a powerful parallel beam output that covers a large area.

- 1) Compared to conventional methods, this unit uses the X-ray more effectively and increases the intensity of the diffracted X-ray, allowing more sensitive analysis.
- 2) With conventional methods, variations in sample surface height are directly translated into variations in X-ray diffraction angles. This polycapillary unit uses parallel beams, so it is not affected by variations in sample surfaces.

Part Description	P/N
PCL-1002 Polycapillary Unit	215-24375-91
CM-4121 Counter Monochromator Assembly (for parallel beams)	215-22360-06
X-Ray Tube (Long fine focus, with Cu target)	210-24100-11

Note: It is not possible to be used in combination with OneSight wide-range high-speed detector.



## Sample Heating Attachment HA-1001

The system is used to heat the sample during X-ray diffractometry to study the influence of heat on the crystalline structure. It consists of a special sample heating furnace and temperature controller. The atmosphere in the furnace, consisting of air, an inert gas or a vacuum, may be heated to 1500°C during measurement. The measurement results are output in multiple data formats to enable comparison of X-ray diffraction patterns obtained at various temperatures.

### Main specifications

- Thermocouple Pt-Pt/Rh
- Measurement 1500°C max. in vacuum, air  
1200°C max. using inert gas (N<sub>2</sub>)
- temperature PID value setting, fixed temperature control  
Control functions (temperature increase, decrease, hold, stop)
- Power supply Single phase 200/220V±10% 5A

Part Description	P/N
Sample heating attachment for XRD-7000 (with temperature controller)	215-23000-01

Note: It is not possible to be used in combination with OneSight wide-range high-speed detector.

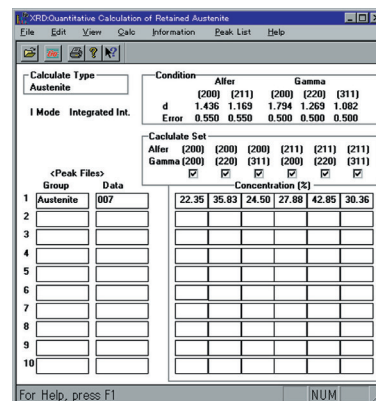


# Various Optional Software

## Quantitative Analysis

### Residual Austenite Quantitation Software (P/N 215-00430-92)

A common method to quantify the residual austenite is to apply the method for samples consisting of 2 components such as tempered copper,  $\alpha$ -iron and  $\gamma$ -iron. The special software allows the determination without the need of a standard sample. The software directly uses the intensity ratio of the measured X-ray peaks of the  $\alpha$ -iron and  $\gamma$ -iron components to theoretically perform the calculation. The five-peak average method is used to make the determination, so scattering due to the matrix effect is reduced to enhance the reliability of the results. Using the rotational sample stage (P/N 215-21766-01) for measurement further helps to overcome data scattering.

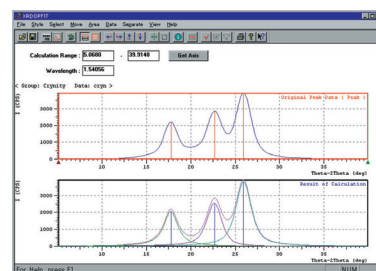


Quantitation Results Screen

## Peak Processing

### Overlapping Profile Fitting Software (P/N 215-00423-92)

Using the Gauss and Lorentz models, overlapping peaks are separated one by one, with information including position, intensity, width at half height and integrated intensity calculated for each diffraction peak. These are then utilized to conduct quantitative analysis and crystalline structure analysis.

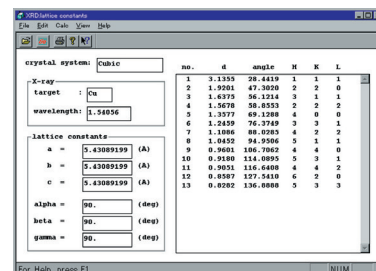


Peak Separation Screen

## Crystalline Structure Analysis

### Precise Lattice Constant Determination Software (P/N 215-00424-92)

In X-ray diffractometry, a higher accuracy is often required to determine the lattice constant, which is a fundamental parameter for determining a substance's crystalline structure. This is most often used for quantitating solid solution metal. This software corrects the raw diffraction angle data calculated via basic data processing to determine enhanced precision lattice constants for up to 7 crystals concurrently, employing the least squares method to further minimize error in diffraction angles. In addition, the miller index is applied to each peak.

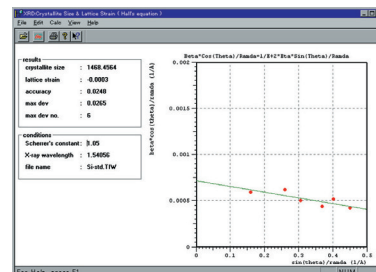


Precise Lattice Constant Determination Calculation Result Screen

## State Analysis

### Crystallite Size & Lattice Strain Software (P/N 215-00426-92)

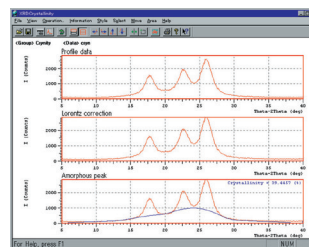
Samples normally consist of crystallites ranging in size from several  $\mu\text{m}$  to tens of  $\mu\text{m}$ . However, in the case of catalyst crystallites, which may measure several hundred  $\text{\AA}$ , X-ray diffraction is insufficient, resulting in diffraction peak spreading. This software quantitatively determines that spread, and applies Scherrer's equation to calculate the crystallite size. When there is involvement of lattice strain, the diffraction spread is determined for a number of diffraction peaks, and from the resultant line slope and intercepts, the size of each of the crystallites and the lattice strain are calculated. (Hall's Method)



Hall's Equation Calculation Result Screen

## Crystallinity Calculation Software (P/N 215-00427-92)

The degree of crystallization of a mixture of crystalline and amorphous substance, such as found in high polymer samples, is an important parameter of substance characterization. This software automatically or manually separates the measured diffraction patterns into those of crystalline components and those of amorphous components. Then, it calculates the integrated intensity of the two types of substances, called degree of crystallization using the peak area ratio of the two classes of components.

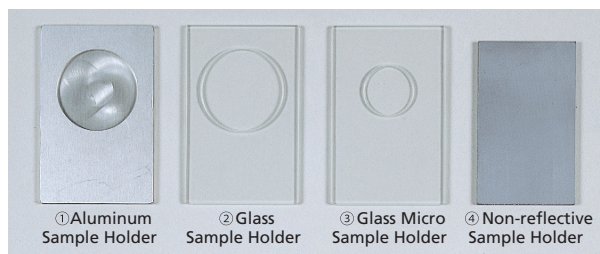


Crystallinity Calculation Result Screen

## Other Accessories

### Sample Holders

The following sample holders are available to allow different applications, including the aluminum sample holder, which is supplied as standard with the diffractometer.



Part Description	Sample area	Application	Remarks	P/N
Aluminum Sample Holder	ø25 (dia.) x 1mm (d)	General purpose	Made of aluminum, 5pc	215-22507-01
Glass Sample Holder	ø25 (dia.) x 1mm (d)	Lattice constant	Made of glass, 5pc	215-22507-02
Glass Micro Sample Holder	ø15 (dia.) x 0.5mm (d)	Micro samples	Made of glass, 5pc	215-22507-03
Non-reflective Sample Holder		Ultramicro samples	Made of silicon, 2pc	215-22507-05

## Cooling Water Circulator

### RKE1500B-V-G2-SP

With its built-in cooler, the Cooling Water Circulator cools the X-ray tube and X-ray generator by circulating cooled, clean water. The unit is recommended when no tap water is available or the available water is of poor quality.

#### Main specifications

- Power supply Three phase 200V ± 10% 10A (RKE1500B-V-G2-SP)
- Ambient temperature 5 to 40°C
- Cooling capacity 5.3kW (50/60Hz) (RKE1500B-V-G2-SP)

Part Description	P/N
RKE1500B-V-G2-SP	239-15049-02



RKE1500B-V-G2-SP

# Specifications

## XRD-7000

Item	XRD-7000L		XRD-7000S
X-ray generator	Max. output	3kW	
	Tube voltage/tube current stability	±0.01% (for 10% power fluctuations)	
	Max. tube voltage	60kV (50kV)*1	
	Max. tube current	80mA (50mA)*1	
	Tube voltage step width	1kV	
	Tube current step width	1mA	
	Overload limit setting	Change setting to suit tube type.	
	Tube protection	Overload, overvoltage, overcurrent, cooling water abnormalities	
Goniometer	Safety mechanisms	Door interlock mechanism (X rays generated after confirming door closed.)	
	Type	Vertical $\theta$ - $\theta$	
	Goniometer radius	275mm standard (variable from 200 to 275 mm)	200mm standard (variable from 200 to 275 mm)
	X-ray beam to attachment base distance	220mm	85mm
	Min. step angle	0.0001°( $\theta$ s), 0.0001°( $\theta$ d)	
	Angular reproducibility	0.0002°	
	Operation angle range	-6 to 82° ( $\theta$ s), -6 to 132° ( $\theta$ d)	
	Operation system	$\theta$ s- $\theta$ d linked; $\theta$ s, $\theta$ d individual	
	Operation mode	Continuous scan measurement, step scan measurement, calibration, positioning	
	Slewing speed	500°/min ( $\theta$ s, $\theta$ d)	
	Operating speed	0.05° to 50°/min ( $\theta$ s, $\theta$ d), 0.1 to 100°/min	
	Divergence slit (DS)	0.5°, 1°, 2°, 0.05mm	
	Scattering prevention slit (SS)	0.5°, 1°, 2°	
	Receiving slit (RS)	0.15mm, 0.3mm	

\*1 When using OneSight wide-range high-speed detector.

## X-Ray Tubes and X-Ray Filters

Focus Type	Type NF	Type BF	Type LFF*3
Focus Size	1 x 10mm	2 x 12mm	0.4 x 12mm
Tube voltage, current	60kV, 50mA	60kV, 60mA	60kV, 55mA
Target	X-ray Tube Maximum Load & P/N		
Cu	2.0kW (239-24014-01)*2	2.7kW (210-24016-21)	2.2kW (210-24100-11)
Co	1.8kW (062-40003-04)	2.7kW (210-24016-24)	1.8kW (210-24100-14)
Fe	1.5kW (062-40003-05)	2.2kW (210-24016-25)	1.0kW (210-24100-15)
Cr	2.0kW (062-40003-06)	2.7kW (210-24016-26)	1.9kW (210-24100-16)

X-Ray Filter	
Part Description	P/N
Ni filter (for Cu)	(215-22500-02)
Fe filter (for Co)	(215-22500-03)
Mn filter (for Fe)*4	
V filter (for Cr)	(215-22500-05)

\*2 When using it as a point focus, combine an X-ray tube and a point focus head of following P/N.

- 1) X-ray tube (Cu target, 2.0kW, NF) P/N 210-24016-11
- 2) Point focus head P/N 239-16047

\*3 When using the polycapillary system, use type LFF.

\*4 Custom support.

## Detectors

### OneSight Wide-Range High-Speed Detector

(FD-1002 1D High-Speed Detector P/N 215-24320-93)

Scan range	0-159° : Radius of Goniometer 275 mm 0-150° : Radius of Goniometer 200 mm
Operation mode	Step-scan mode, One-shot mode
Sensor	Reverse biased pn-junction array
Detection principle	Single photon counting
Weight	280 g
Active area	64 x 8 mm
Number of channels	1280
Width of one channel	50 $\mu$ m

Data processing unit	Casing	Dimensions	W1120 x D1049 x H1790
		OS	Windows® 10
		Controlled elements	Goniometer, X-ray generation, tube voltage, tube current, detector high voltage*5, PHA*5, scaler
		Basic data processing	Smoothing, BG elimination, Ka1-Ka2 separation, peak searching, peak width at half height, integrated intensity, systematic error correction, internal/external standard correction, operations between data, graphic display
		Qualitative analysis	Database generation, automatic searching (ICDD PDF-2/PDF-4 optional)
	Quantitative analysis	Calibration curve generation, quantitative analysis	

\*5 When using Scintillation detector.

### Scintillation Detector

SC-1003 P/N 215-24385-92

Scintillator	Nal
Scaler	Preset time: 0.1 to 1000s; digits: 7
HV/PHA	500 to 1200 V high-voltage power supply, baseline and window auto-controlled

# Accessories

## Special Accessories

◎: Absolutely required   ○: Required   ●: Required depending on objective

Analysis Objective	Part Description	P/N															
			Iron and steel related	Non-ferrous metals, precious metals	Machinery, automotive, shipbuilding, welding	Brickmaking, ceramics	Cement and glass	Electrical, electronic materials	Foodstuffs, textiles, paper, pulp	Chemicals, catalysts, dyes, paints	Medical, dental materials, biological organisms	Natural resources, energy	Construction, engineering	Environment, industrial waste	Environment (Asbestos)		
1 BG reduction, especially iron samples	Counter Monochromator CM-3121	215-22360-02	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
2 Qualitative analysis PDF-2 Search	ICDD PDF-2 file	239-50002-11,12	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
3 Qualitative analysis PDF-4 Search	ICDD PDF-4 + file	239-50015-01,02	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎
4 General purpose quantitative analysis	*Rotational Sample Stage RS-1001 <sup>Note1</sup>	215-21766-01	○	○	○	○	○	○	○	○	○	○	○	○	○	○	○
	*Auto 5 position sample changer ASC-1001 <sup>Note1, 2</sup>	215-23175-02															
5 Residual austenite quantitation	Residual austenite quantitation S/W	215-00430-92	○		○												
	Rotational Sample Stage RS-1001 <sup>Note1</sup>	215-21766-01	○		○												
6 Environmental quantitative analysis system	*Environmental Quantitative Analysis Stage RS-2001 <sup>Note1</sup> (Filter holders Zn, with S/W)	215-21767-03													◎	◎	
	Filter holder Al (ø 25mm)	215-23765-92													◎	◎	
7 Multiple peak separation	Profile fitting S/W	215-00423-92	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●
8 Precise lattice constant determination	Precise lattice constant determination S/W		●	●	●	●	●	●	●		●						
9 Crystallite size / lattice stress	Crystallite Size / Lattice Stress S/W	215-00426-92								○	○	○					
10 Degree of crystallization	Degree of Crystallization S/W	215-00427-92	●							○	○	○					
11 Heating analysis	Sample Heating Attachment HA-1001	215-23000-01	●	●	●	●	●	●	●		●	●					
12 Thin film analysis	*Thin Film Analysis Attachment THA-1101 <sup>Note1</sup> (stage, monochromator, suction pump)	215-21765-01	●	●	●	●	●	●	●								
13 Fiber degree of orientation analysis	Rotational Sample Stage RS-1001 1)	215-21766-01	●	●	●	●	●	●	●	○	●						
	Fiber Sample Attachment (with S/W)	215-22624	●							○	●						
14 Residual stress analysis	*Stress analysis attachment SA-1101 compatible with scintillation detector (with Cr tube) <sup>Note1</sup>	215-21769-01	○	○	○	●	●	●									
	*Stress analysis attachment SA-1111 compatible with scintillation detector (with Co tube) <sup>Note1</sup>	215-21769-03	○	○	○	●	●	●									
	*Stress analysis attachment SA-2101 compatible with OneSight detector (with Cr tube) <sup>Note1</sup>	215-21769-95	○	○	○	●	●	●									
	*Stress analysis attachment SA-2111 compatible with OneSight detector (with Co tube) <sup>Note1</sup>	215-21769-96	○	○	○	●	●	●									
	Stress analysis software (compatible with scintillation detector/OneSight detector)	215-00429-91	○	○	○	●	●	●									
15 Strong parallel beam X-ray source	Poly-capillary unit PCL-1002	215-24375-91	●	●	●	●	●	●	●	●	●	●	●	●	●	●	●

Note1: Please arrange optional additional ASSY(P/N215-23705) with optional driver ASSY(P/N215-21764) at the same time when you arrange the accessories of the asterisk. Moreover, even when two or more accessories are arranged, the option driver ASSY and optional additional ASSY can be combined with one unit.  
 Note2: When arranging an auto 5 position sample changer, please arrange two optional additional ASSY(P/N 215-23705).

The following attachment software cannot be used in combination with OneSight wide-range high-speed detector.

- Counter Monochromator CM-3121
- Sample Heating Attachment HA-1001
- Thin Film Analysis Attachment THA-1101 (can be used as rotational sample stage)

The following analysis objectives are unmeasurable.

11 Heating analysis, 12 Thin film analysis, 13 Fiber degree of orientation analysis, 15 Strong parallel beam X-ray source.

Please contact us for further information.

# Installation Requirements

## Installation Site

This instrument uses X-rays for measurement and analysis. Accordingly, before installing the instrument, be sure to consult local regulations regarding measures associated with X-ray generation, and comply with all necessary regulatory procedures.

## Power Requirements

For main unit	Single phase 200/220V $\pm 10\%$ 2kW type: 30A 3kW type: 50A
Data processing unit	Single phase 100V $\pm 10\%$ 10A
Ground	Independent, at least 100 $\Omega$ resistance

Power supply voltage fluctuation must not exceed 10%.

If the sample heating attachment, cooling water pump or cooling water circulator is used, a separate power supply is required.

## Installation Site Environment

The following ambient temperature and humidity are required.

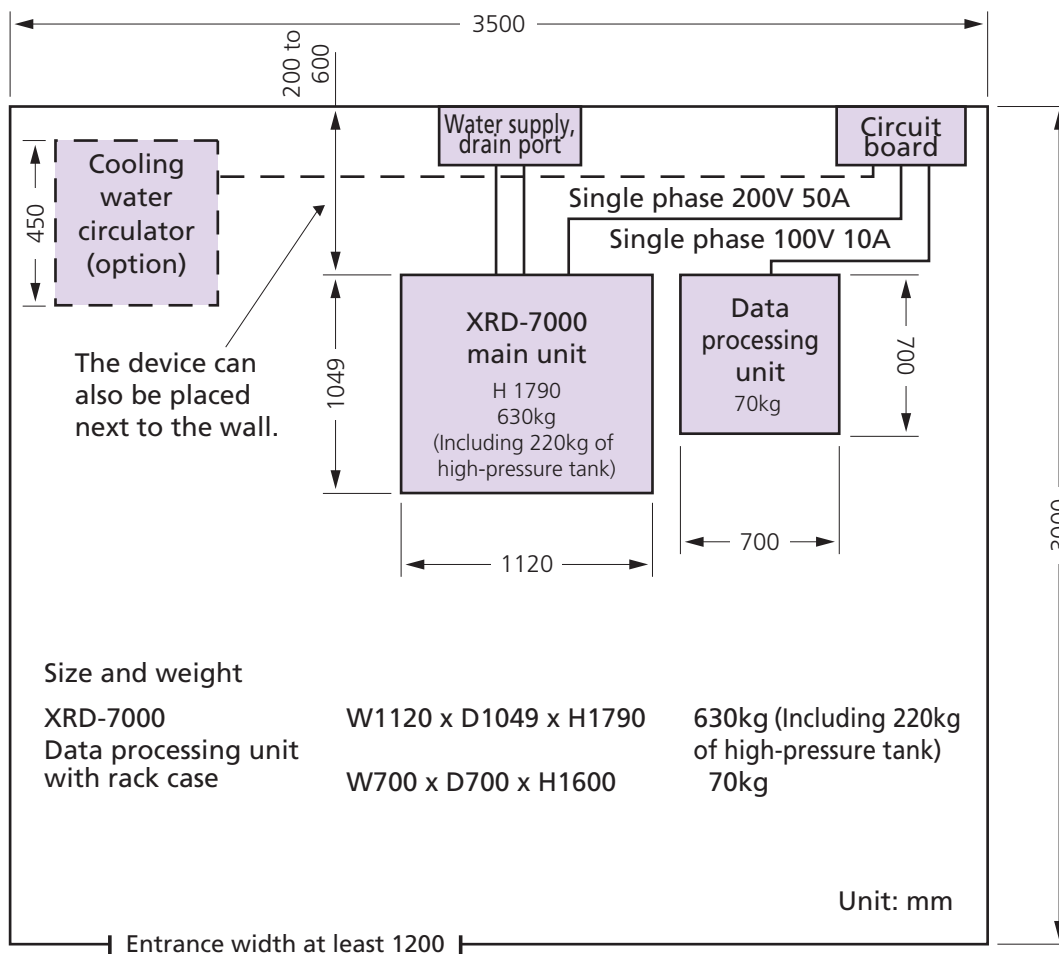
Temperature	23°C $\pm$ 5°C
Humidity	Less than 75%

Avoid any sudden changes in temperature, which might cause condensation to form on the surfaces of internal parts.

Heat generated from the instrument is approximately 1kW. When the cooling water circulator is installed in the same room, this is increased by 2.6kW for the NF type X-ray tube, 2.9kW for the LFF type X-ray tube and 3.9kW for the BF type X-ray tube.

Flow rate	at least 4.0L/min
Water pressure	0.3 to 0.5 MPa
Water quality	pH6~8, hardness less than 80ppm
Particulates	less than 0.1mm
Supply water port diameter	12.7mm $\phi$
Drain water port	Natural drainage

If the flow rate is lower than 4.0L/min, the safety circuit for protection of the X-ray tube is active, disabling the X-ray generation circuit. When minimum flow rate conditions cannot be fulfilled, use the cooling water circulator, available as an option.



**XRD-7000 Floor Plan Example**

OneSight is a trademark of Shimadzu Corporation.  
Windows is either registered trademark or trademark of Microsoft Corporation in the United States and/or other countries.  
ICDD and PDF are registered trademarks of JCPDS-International Centre for Diffraction Data Corporation in the United States.



Shimadzu Corporation  
[www.shimadzu.com/an/](http://www.shimadzu.com/an/)

**For Research Use Only. Not for use in diagnostic procedures.**

This publication may contain references to products that are not available in your country. Please contact us to check the availability of these products in your country.

Company names, products/service names and logos used in this publication are trademarks and trade names of Shimadzu Corporation, its subsidiaries or its affiliates, whether or not they are used with trademark symbol "TM" or "®".

Third-party trademarks and trade names may be used in this publication to refer to either the entities or their products/services, whether or not they are used with trademark symbol "TM" or "®".

Shimadzu disclaims any proprietary interest in trademarks and trade names other than its own.

The contents of this publication are provided to you "as is" without warranty of any kind, and are subject to change without notice. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to the use of this publication.