

Miniflex

Rigaku/”Miniflex” X-ray Diffractometer System

Rigaku Corporation

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1. Introduction

Rigaku’s general purpose X-ray diffractometer systems are broadly classified into two series; the D/MAX series and the Miniflex series.

The D/MAX series is a research grade tool which meets a wide range of X-ray analysis needs and can be configured with a variety of diffraction attachments and cameras. According to diversified measurement purposes, attachments allow multiple axis rotation, mapping, stress loading, heating and cooling, and so on.

The Miniflex series is designed to be functionally simple while maintaining precision measurement capability comparable to the D/MAX series. It is ideally suited for teaching in the college /university environment and quality control and inspection in production. As examples, the Miniflex is widely used to control composition and mixture ratios as well as for quantitative measurement of environmentally hazardous materials. In these applications, the operational procedures are routine, the sample materials and the measuring conditions remain similar.

The Miniflex is the smallest, lightest weight diffractometer in the world. It fits easily on a desktop and simply plugs into the wall. It is rugged enough to load into a vehicle and be transported and used in the field. This capability is ideal for using at geological sites, quarries, or for sorting concrete skeleton materials.

2. Features

(1) Radiation Enclosure & Safety

When the radiation enclosure door is opened, the X-ray shutter will automatically close. This function completely protects the user from exposure.

(2) Display of Instrument Status

Instrument status is available on demand to monitor the functions of the

components.

(3) Continuously Variable Slit

The continuously variable slit (patent pending) is linked to the 2θ becomes larger. This will improve the peak-to background ratio at low 2θ angles as well as increase the X-ray intensity at high 2θ angles. These improvement are important for the measurement of clay minerals, concrete skeleton materials, etc.

(4) Automatic Sample Changer with 8 Positions

Up to 6 samples can be mounted and measurement with the automatic sample changer. Each sample can be made to spin to enhance data precision for qualitative and quantitative analysis. The changer option is helpful for routine sample analysis of geological samples, asbestos dust, row material consistency, etc.

(5) Sample Holders

Two(2) types of holders, for powder and bulk samples, are available for use with the Miniflex's vertical goniometer.

(6) Standard Measurement Software

The software is written in Windows for the PC, has multiple functions, and is easy to use.

Measurement condition parameters include the following:

Sample Name (ID)

Scan rang in degrees 2θ

Measurement mode (continuous scan, step scan, or skip scan)

Sampling width in degrees per step

Scanning speed

(7) Qualitative Analysis Software

Identification of phases is achieved through accessing the ICDD databases. This is accomplished through the primary search/march to ICDD card information, a secondary search/march which compares the d-spacings and intensities with the measured data, and a residual search/march which is performed after excluding the major components.

3. Configuration

3.1 Standard Configuration

- (1) Goniometer
- (2) Tube shield
- (3) Detector
- (4) X-ray generator
- (5) Electronic circuit panel
- (6) X-ray proof console
- (7) Flow rate detection system
- (8) X-ray tube
- (9) Personal computer
- (10) Standard measurement software

3.2 Options

- (1) Air cooling system heat exchanger
- (2) Specimen rotation attachment
- (3) Auto sample changer
- (4) Autotransformer
- (5) Optional software
 - 1) Multiple recording software
 - 2) Qualitative analysis software
 - 3) Simplified qualitative analysis (concrete skeleton material, qualitative analysis, compositive ratio, etc.)
 - 4) ICDD database control
 - 5) Database software for ICDD qualitative analysis
 - 6) Environmental particle dust quantitation (asbestos particle dust) software

4. Specifications

4.1 Goniometer

An automatic variable divergence slit (patent pending) is standard on the system. The slit width increases as angles increases, keeping the irradiated area on the sample constant. The goniometer is a high precision design which operates by maintaining a 2 to 1 ratio of the 2θ and the ω axes.

- (1) Type : Vertical type
- (2) Goniometer radius : 150mm
- (3) Scanning axis : $\omega/2$ coupled
- (4) 2θ angle measurement range : $-3^\circ \sim +150^\circ$
- (5) Slew speed : $1000^\circ/\text{min}(2\theta)$
- (6) Scanning speed : $0.01 \sim 100^\circ/\text{min}$ (keyboard input)
- (7) Datum angle : $2\theta/10^\circ$
- (8) Slit
DS : Variable slit interlocked with ω axis, max. 2.8°
SS : 2.8° fixed
RS : 0.3mm fixed
Soller slit : 2.5°
- (9) Setting precision : $2\theta \pm 0.05^\circ$
- (10) Setting angle reproducibility : $2\theta \pm 0.01^\circ$
- (11) X-ray take-off angle : 6° fixed

4.2 Tube Shield

Contains shutter mechanism, X-ray tube, and high voltage connection.

- (1) Applicable X-ray tube : Toshiba A-41, Philips PW22XX, etc.
- (2) Focus position adjuster : ± 1 plate spring system, continuously variable(patent pending)
- (3) X-ray shutter : Mechanical rotary shutter interlocked with open/close door of X-ray proof console
- (4) Filter for K removal : Ni filter (for Cu-target X-ray tube. Other

are optional.)

4.3 Detector SC-M

Incident X-ray are diffracted by the sample. The resultant photons are illuminated on a scintillation crystal and are converted to electrical signals by a photomultiplier tube specifically developed for the Miniflex. The SC-M is an exceedingly small-sized scintillation counter.

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| (1) Scintillator | : NaI (Ta) |
| (2) Window material | : Be |
| (3) Outer dimensions | : 24mm dia. x 45mm long |

4.4 X-ray Generator

The 450W high frequency generator is a highly stable source.

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| (1) Tube voltage output | : 30kV (fixed) |
| (2) Tube current output | : 15mA (fixed) |
| (3) high voltage generation system | : High-frequency Cockcroft-Walton system |
| (4) 7-hour continuous stability | : Within $\pm 0.05\%$ for both tube voltage and tube current as against $\pm 10\%$ input power variations |
| (5) Safety circuit | |
| 1) Error lights and audio alarms are tripped when there is an overload of voltage or current and/or insufficient water flow. At the time of the failure, the high voltage will shut down. | |
| 2) X-ray generation, shutter open/close display lamp | |
| 3) Emergency push-button switch | |

4.5 Electronic Circuit Panel

The electronic circuit panel (ECP) controls the 2 / stepping motor, the DC high voltage power supply for the detector, the PHA discriminator, and scaler. The ECP also displays the operating status of each component.

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| (1) / 2 axis minimum step angle | : 0.005 ° /pulse (2) |
| (2) H.V. power supply for counter | : 0 ~ 1000V \pm 0.02% |
| (3) Pulse height discrimination circuit | : Fixed pulse height system |
| (4) Scaler | : 24-bit counter |
| (5) Maintenance display function | : |
| 1) SCAN | : Stays on while the detector keeps on scanning in order during goniometer motor run. |
| 2) HV | : Stays on while high voltage is applied to the detector. |
| 3) PHA | : Stays on while counting is made as X-ray are being detected by the detector. |
| 4) HVL | : Lights up instantly when the tube voltage exceeds 31.5KV to turn off X-ray along with buzzer alarm. |
| 5) LVL | : Lights up instantly when the tube voltage falls below 28.5KV to turn |

off X-ray along with buzzer alarm

6) HTC : Lights up instantly when the tube current exceeds 16mA to turn off X-ray along with buzzer alarm

7) LTC : Lights up instantly when the tube voltage falls below 14mA to turn off X-ray along with buzzer alarm

8) HFC : Lights up instantly when the filament current for the X-ray tube flows in excess of 150 percent to turn off X-ray along with buzzer alarm

9) LFC : Lights up instantly when the filament current for the X-ray tube falls below 50 percent to turn off X-ray along with buzzer alarm

10) CW : Lights up when the cooling water flow rate becomes less than 1.8lit/min to turn off X-ray along with buzzer alarm

11) X-RAY : Stays on during X-ray generation.

12) SHUTTER : Stays on while the X-ray shutter is open for irradiation.

13) READY : Lights when all is set for X-ray generation, with power on, a sufficient cooling water flow rate, and fully closed console door.

4.6 Water cooling unit

Cooling water to dissipate the heat form the X-ray tube can be provided from an in'house supply, or where unavailable, from a dedicated portable air cooled, recirculating heat exchanger.

(1) Flow meter : For detection of a 1.8 lit/min or less flow rate on the drain side.

(2) Hose : 10mm dia. I.D., 16mm dia. O.D., 5 meters each (TETRON braided), with two hose bands

4.7 X-ray Tube

The system requires a vacuum sealed X-ray tube of various target materials. Precision alignment of the optical system is performed at the factory prior to shipment.

(1) X-ray tube : Toshiba Cu (standard) 1 set

(2)Max. output : 1KW

(3) Focus size : 1 x 10mm

4.8 Computer

This computer controls the goniometer / 2 axis via the electronic circuit panel. It also performs data storage, various corrections, computing analysis, display and output.

4.9 Standard Measurement Software

(1) System environment parameters

(2) Manual measurement

(3) Standard measurement

(4) Integrated intensity calculation

(5) Peak search

4.10 Options

- (1) air cooling system recirculation water feed pump (heat exchanger)
- (2) Specimen rotation attachment
- (3) Auto sample changer
- (4) Optional software
 - 1) Multiple recording software
 - 2) Qualitative analysis software
 - 3) Civil engineering material analysis (concrete skeleton material analysis)
 - 4) ICDD database management
 - 5) ICDD database
 - 6) Environmental particles dust quantitative analysis (asbestos quantitative analysis)

5. Installation Requirements

5.1 Dimensions and Weight

- (1) Basic console unit : 560mmW x 315mmD x 582mmH., 59kg
- (2) Air cooling system : 333mmW x 540mmD x 425mmH., 21kg
Heat exchanger

5.2 Installation Requirements

- (1) Floor strength : No vibrations sensible to the human body.
- (2) Temperature : 10 ~ 30
- (3) Relative Humidity : 40% ~ 70% (no condensation)
- (4) Magnetic field : Nothing must be nearby which will generate a magnetic field that affects the computer's CRT image.

5.3 Power Capacity

The power supply of the following capacity and rating should be used for the Miniflex. The total capacity may vary depending on combined units.

Single phase AC: 115V, 15A

Earth-grounded receptacle (3P) 3 pieces

5.4 Power Supply Quality

- (1) Frequency stability : $59\text{Hz} \pm 1\%$
- (2) Voltage stability : $100\text{V} \pm 10\%$
- (3) For the single phase, one wire must be ground.
- (4) No such device as will cause surge and noise must be connected to the same wiring system.

5.5 Grounding

- (1) Grounding resistance : 100 or less