

SRS 303 sequential X-ray spectrometer

Application



Fig. 1/1 SRS 303 sequential X-ray spectrometer with sample changer for 10 samples

Application

The SRS 303 is a microprocessor-controlled high-performance sequential X-ray spectrometer for universal application.

It can be used for fast qualitative and quantitative analyses of solids, powders and liquids for elements with atomic numbers from 5 (B) upwards. Easy operation.

The main applications are in industrial and research laboratories in which a wide variety of samples have to be analyzed for a large number of different elements.

Advantages

- High sensitivity for light elements by use of a 3-kW end window tube with a beryllium window only 125 μm thick (window diameter 19.5 mm) and with a small distance between the anode and the sample
- Simple dialog operation using keyboard and monitor as result of integrated microprocessor
- Additional simplification of operation by storing and calling complete parameter sets
- Rapid and easy changing of X-ray tube
- Primary radiation filters can be inserted between the X-ray tube and the sample to adapt the excitation spectrum to special analytical problems (filter changer with 4 positions, 3 fitted with filters and 1 empty)
- Beam path can be adapted to the sample size using aperture slits which are inserted in front of the collimator (slit changer with 3 positions, 2 fitted with slits and 1 empty)
- Sample changer for 10 samples
- Analytical results independent of the sample position and the sample cup since all sample cups are fixed in the measuring position against the same edge. The sample cup bearing area is the same as the sample bearing area and constitutes the reference area
- Non-homogeneities of the sample surface are eliminated by sample rotation

- 2 collimators with opening angles of 0.15° and 0.4°
- Coupled double worm gear for highly accurate setting of angle
- Electronic crank for adjustment of $\theta/2\theta$ (optional accessory)
- Crystal changer for 6 analyzer crystals
- Pre-adjusted analyzer crystals on magnetic holders
- Tandem arrangement of scintillation counter and flow counter results in high sensitivity for wavelengths in the center of the spectral range
- Temperature-stabilized and pressure-stabilized counter tube gas
- Fully protected instrument according to the X-ray regulations
- Modern, compact design, good serviceability.

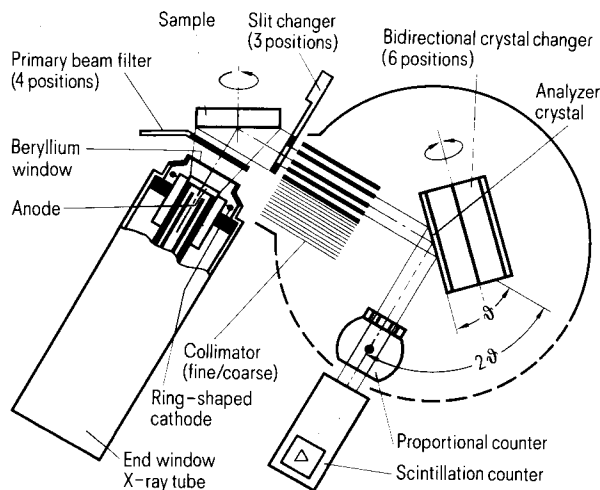


Fig. 1/2 Beam path in the SRS 303 sequential X-ray spectrometer, functional diagram

Design and mode of operation

Design

The SRS 303 sequential X-ray spectrometer consists of:

- Spectrometer with built-in KRISTALLOFLEX K730 X-ray generator, measuring electronics and microprocessor
- Control keyboard and monitor.

The spectrometer is designed for vacuum, helium or air/nitrogen operation. It operates with high-power end window tubes up to 3 kW. The sample is irradiated from below.

6 analyzer crystals, 2 Soller slits and 2 detectors are available in a tandem arrangement for the analysis. To optimize the beam path, unrequired radiation is suppressed using primary beam filters and Soller slit diaphragms. The spectrometer is equipped with a sample changer for 10 samples.

The spectrometer is temperature-stabilized; the counter tube gas for the flow counter is density-stabilized.

The measuring and control electronics (microprocessor) of the SRS 303 is fitted as an independent module in the spectrometer housing. Separate amplifiers and high-voltage supplies are present for the scintillation counter and the flow counter. The dependence of pulse height on the energy of the radiation is compensated by a microprocessor-controlled sine-wave amplifier. A digital ratemeter calculates the pulse rate digitally from dead-time corrected counted values. The pulse-height distribution and the position of the pulse-height thresholds are output on the pulse spectroscopy screen during the measurement.

The microprocessor controls the spectrometer and monitors the instrument functions. It stores the test conditions and controls the test parameters. It corrects the dead time and line shifts and controls data transfer with the peripheral computer. It can test all SRS 303 functions and indicates faults on the screen.

The KRISTALLOFLEX K730 X-ray generator supplies the power to operate the end window X-ray tubes. The tube current and voltage can be triggered by the computer. The generator is highly stabilized; power supply fluctuations of 1% only change the tube current and voltage by 0.001% (typical value).

A complete analytical system with the SRS 303 sequential X-ray spectrometer includes the following accessories:

- Counter tube gas cylinder with reducing valve
- Helium cylinder with reducing valve (for helium operation)
- Evaluation computer with monitor and printer
- Evaluation program(s)

Mode of operation

The sample in the measuring position is irradiated from below and is excited to emit fluorescence radiation. To optimize the analysis, test parameters such as the mode of operation, type of excitation, analyzer crystal, primary beam filter, Soller slit, Soller slit diaphragm, detectors, pulse-height window, measuring time etc. are defined for each element of a family of samples (matrix) already during calibration of the equipment and stored in the evaluation computer. Following calibration, the calibration data (slope, background and correction factors) are also stored separately for each matrix.

In order to analyze unknown samples, the test parameters stored under a particular matrix designation are called and set. Following measurement of the unknown samples, the concentrations of the elements are calculated using the calibration data stored in the evaluation computer and then output on the printer together with the measured intensities.

The pulse-height thresholds are set separately for the two detectors. The line shift which occurs with the flow counter with high pulse rates is corrected. The dead time correction is carried out separately for the scintillation counter and the flow counter. The gain which is dependent on the angle is corrected by the sine-wave amplifier.

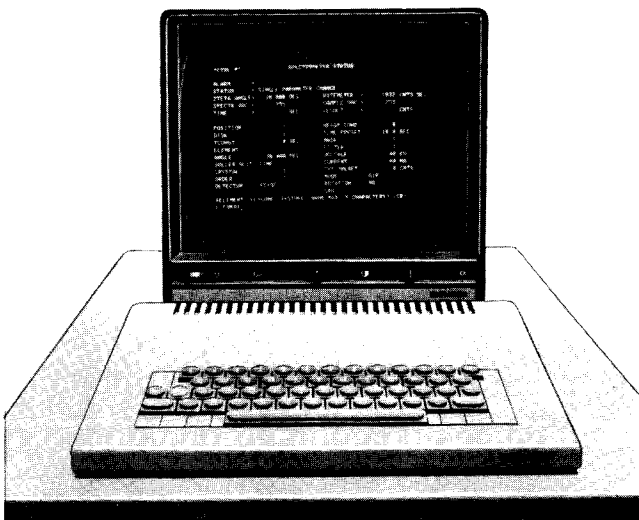


Fig. 1/3 Monitor and control keyboard

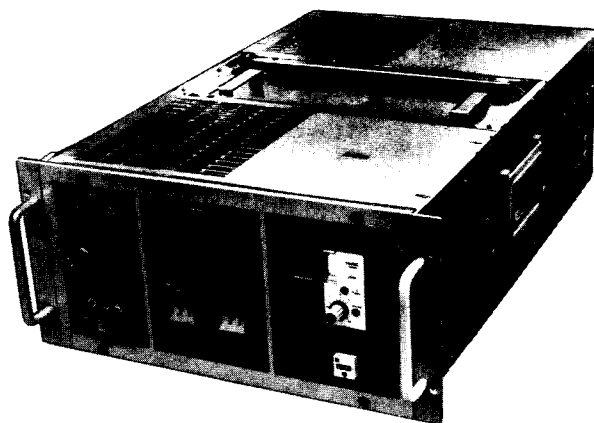


Fig. 1/4 KRISTALLOFLEX K730 X-ray generator

SRS 303 sequential X-ray spectrometer

Technical data

Spectrometer

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|--|---|
| Angular range (2 ϕ) | 4 to 93° |
| For scintillation counter | 4 to 148° |
| For flow counter | 0.001° |
| Reproducibility (2 ϕ) | 0.1; 0.5; 1; 2; 5; 10; 20; |
| Angular speed (2 ϕ) | 50; 100 or 500°/min |
| Max. sample size | 51 mm diam. or 36 mm \times 36 mm, 40 mm high |
| Irradiated sample area | 8, 23, 30 or 34 mm diam. |
| Material of the sample holder mask | Spectrally pure sintered carbon or gold-plated brass |
| Sample rotation | 30 rpm |
| Collimators (Soller slit) | Opening angles 0.4° and 0.15° |
| Crystal changer | For up to 6 analyzer crystals as selected |
| Analyzer crystals available | LiF(100), LiF(110), LiF(210), PET, KAP, AdP, PbSt, InSb, TIAP, Ge, OVO (multilayer) |
| Detectors | Scintillation counter and flow counter, counter tube gas pressure-stabilized and temperature-stabilized |
| Dose rate outside the spectrometer housing | $\leq 2 \mu\text{Sv/h}$, fully protected instrument |
| X-ray tube | End window tube type AG 66 G |
| Anode material | Rh (other materials on request) |
| Power rating | Max. 3 kW |

• High-voltage generator for the detectors

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|--|---|
| Voltage range | 700 to 2300 V, continuously adjustable |
| Repeatability of setting | 0.1% of actual value |
| Permissible current consumption | Max. 1 mA |
| Ripple U_{PP} | $\leq 100 \text{ mV}$ |
| Influence of temperature | $\leq 0.02 \text{ V/K}$ |
| Drift over 24 h at constant conditions | $\leq 0.02\%$ of actual value |
| Short-circuit resistance | Unlimited |

General data

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|--------------------------------------|--|
| Radiation protection certificate | BW/145/83/Rö of 15.3.1983 |
| Dimensions (h \times w \times d) | |
| Spectrometer | 1010 mm \times 1576 mm \times 860 mm |
| Desk | 720 mm \times 1200 mm \times 780 mm |
| Weight | |
| Spectrometer | Approx. 670 kg |
| Desk | Approx. 50 kg |

KRISTALLOFLEX K730 X-ray generator

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|--|--|
| High-voltage, can be triggered by computer | 20 to 60 kV, adjustable in 1-kV steps |
| Tube current, can be triggered by computer | 5 to 100 mA, adjustable in 1-mA steps |
| Power output | Max. 3 kW |
| Stability of high-voltage and tube current | Max. 0.001% of setpoint with power supply fluctuations of $\pm 1\%$ |
| Regulating time constant | 100 ms |
| Limit monitoring of | Power, max. tube current, max. tube voltage, heating current, cooling water data (conductivity, temperature, flow rate) |
| Power supply | AC 50 or 60 Hz 208, 220 or 240 V |
| Power consumption | Max. 5.5 kVA |

Measuring electronics

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|---|--|
| • Pulse amplifier | |
| Gain factor | 3 to 20, adjustable using potentiometer; can be halved for measurements of 2nd order lines |
| Influence | |
| Of angle | $< 0.5\%$ of setpoint |
| Of temperature | $< 0.2\%/K$ |
| Max. pulse amplitude | 2.5 V |
| Pulse width | 200 ns |
| Pulse-height thresholds | |
| Lower threshold | (0.2 to 0.9) \cdot reference value |
| Upper threshold | (1.1 to 4.2) \cdot reference value |
| Pulse-height reference value | 0.5 V |
| Influence of temperature of the pulse-height thresholds | 0.1 mV/K |
| Counting capacity | 2 ²⁴ pulses (\approx approx. $16 \cdot 10^6$ pulses) |
| Measuring time | 0 to 1000 s, adjustable in steps of 0.1 s; |
| Time constant for mean value determination | 0.1 to 1 s, adjustable in steps of 0.1 s 1 to 25 s, adjustable in steps of 1 s |
| Dead time error | $< 1\%$ with $5 \cdot 10^5$ pulses/s |

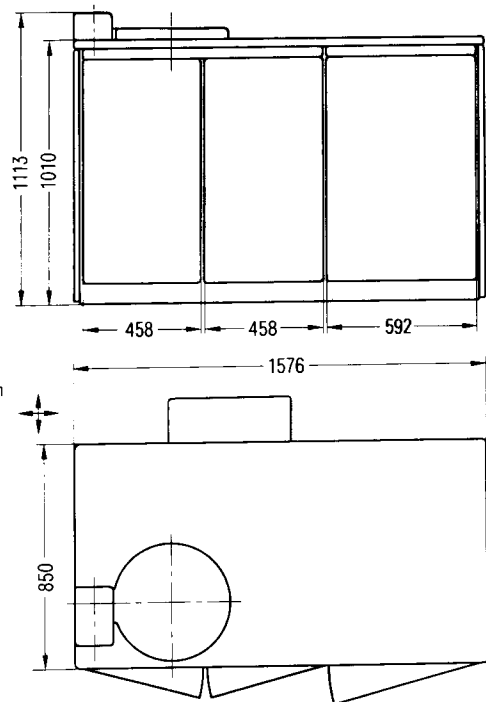


Fig. 1/5 SRS 303 sequential X-ray spectrometer, dimensions