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Application and Product Reports

SCXmini™
Benchtop Small Molecule Single Crystal X-ray Diffraction System

1. Overview

SCXmini represents a new paradigm in small molecule crystallography. Specifically engineered to provide colleges, universities, and industry with access to definitive molecular structure determination, this affordable and compact benchtop system was specifically designed to enable single crystal diffraction to become a routine laboratory method and teaching tool in the same way that NMR and FT-IR did more than a decade ago.

This newest member of the small molecule line of Rigaku integrated solutions was designed to offer affordable, reliable, easy-to-use, low cost-of-ownership access for routine solution of crystal structures. The combination of the new advanced Mercury2 CCD detector with a simplified goniometer, a sealed-tube X-ray source, and automated structure solution software makes the SCXmini the perfect routine instrument for use by non-crystallographer researchers as well as a highly accessible teaching tool for all levels of students.

2. Benefits

The elucidation of the structure of "small molecules" is the principal domain of inorganic and organometallic chemistry. Within these disciplines, two techniques are typically employed for structure determination: nuclear magnetic resonance (NMR) spectroscopy and single crystal X-ray diffraction (SXRD).

The former technique requires expensive instrumentation which employs a cryogenically cooled superconducting magnet assembly. Such NMR spectrometers, while now relatively commonplace, have high associated maintenance and overhead costs while producing spectra that can be difficult to unambiguously interpret in many circumstances. While SXRD

does not in any way replace the wealth of different types of chemical information supplied by NMR, it is a critical supplemental tool for NMR spectra assignments, and is the only "mainstream" structural tool available for sparingly soluble or paramagnetic compounds.

Until now, for most chemists, whether they be students or industry professionals, the usual way to obtain an X-ray structure was to submit a crystal to a third party crystallographer and wait for a result. Traditional "main frame" SXRD instrumentation was designed to offer remarkable levels of performance to highly skilled specialized users (crystallographers). But this approach has, for many decades, left a gap in the collective ability to advance structural scientific endeavors at their optimal pace. The goal of Rigaku, relative to the development of the SCXmini, was to offer both researchers and students an affordable instrument that did not require exceptional knowledge or skills to operate.

Thus the SCXmini is remarkable in that it offers levels of performance comparable to traditional "main frame" SXRD instruments at up to a 50% lower price. Taking up minimal space, being a self-contained benchtop instrument, and offering automatic structure solution software as a standard feature, the SCXmini is the first of a new paradigm in X-ray diffraction instrumentation for research and education.

3. System

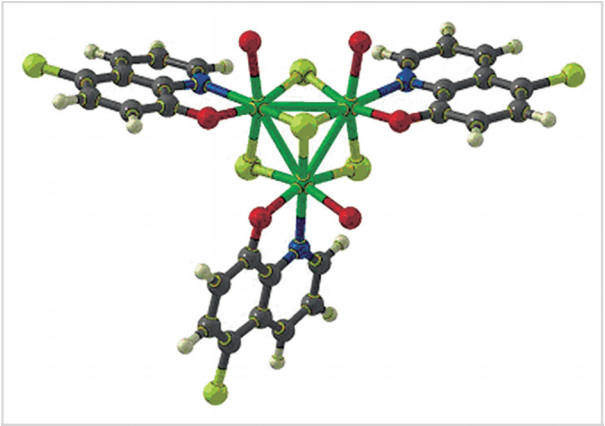
The SCXmini was designed to offer outstanding performance, in a small package and at a fraction of the price and cost-of-ownership of a conventional single crystal XRD system. It features the new advanced Mercury2 CCD detector with a simplified goniometer, a 3 kW sealed-tube X-ray source with an optional high flux optics, and automated software.

Capabilities of the system are demonstrated by results shown in Fig. 1. A complete (>99%) triclinic data set, to 2θ=55°, was obtained from a very tiny platelet crystal of an incomplete cubane-type sulfur-bridged Mo cluster complex. Using only three 180° scans, with an exposure time of 30 s per 0.5 degree, produced a publication quality structure. Even the hydrogen atoms on the interstitial water molecules were located in final difference maps and included in the refinement.

4. Operation

In making X-ray crystal structure determination accessible to a broader audience, it was an imperative that routine operation be as simple as possible. As illustrated in Fig. 2, a researcher can go from mounting a crystal to the final report in a just few hours. Aside from physically attaching and aligning the crystal, all other tasks are fully automated by the SCXmini software.

A typical scenario would involve mounting a crystal onto the goniometer using a convenient magnetic pin system. Then one would use the video microscope, which gives a clear view of the sample from the computer monitor, to quickly center the crystal and measure its dimensions. Unit cell dimensions are then determined by the software, in a couple of minutes, by an auto-indexing and refinement routine. An overlay of predicted spot positions allows verification of the fit of the unit cell to the reflection positions.



Molecular formula: $C_{35}H_{36}Cl_5Mo_3N_3O_{12}S_5$ MW = 1316.04
Crystal size: 0.12 × 0.10 × 0.06 mm Crystal system: triclinic
Space group: $P\bar{1}$ (#2)
Unit Cell: a=12.3016(10) Å, b=13.9575(11) Å, c=15.4437(13) Å
α =75.291(2)°, β =81.926(2)°, γ =61.860(2)°
V=2260.9(3) Å^3, Z=2
X-ray source: MoKα (50kV, 40mA) Meas. temp.: 100K
Data images: 1080 exposures Exposure rate: 30s/0.5°
ω oscillation range (χ=54.0°, φ = 0°, 90°, 180°): -120.0 - 60.0
Structure solution: direct methods
Refinement: full-matrix least squares on F^2
$2\theta_{max}$ cutoff: 55.0° Anomalous dispersion: all non-H atoms
No. observations: 10,313 No. variables: 598
Completeness: >99% Goodness-of-fit: 1.109
Residuals for I>2σ(I): R1 = 0.0593
Residuals (all reflections): R1 = 0.0905, wR2 = 0.1282
Largest diff. peak and hole: 0.97 and -1.52 e$^-$/ Å^3

Fig. 1. Structure solution example: (μ_3-Sulfido)-tris(μ_2-sulfido)-triaqua-tris(5-chloro-8-quinolinato)-trimolybdenum p-toluenesulfonate dichloromethane solvate trihydrate.

* Original structure publication: Shibahara, T. et al., *Inorg. Chem. Comm.*, **8**, 777–781 (2005).

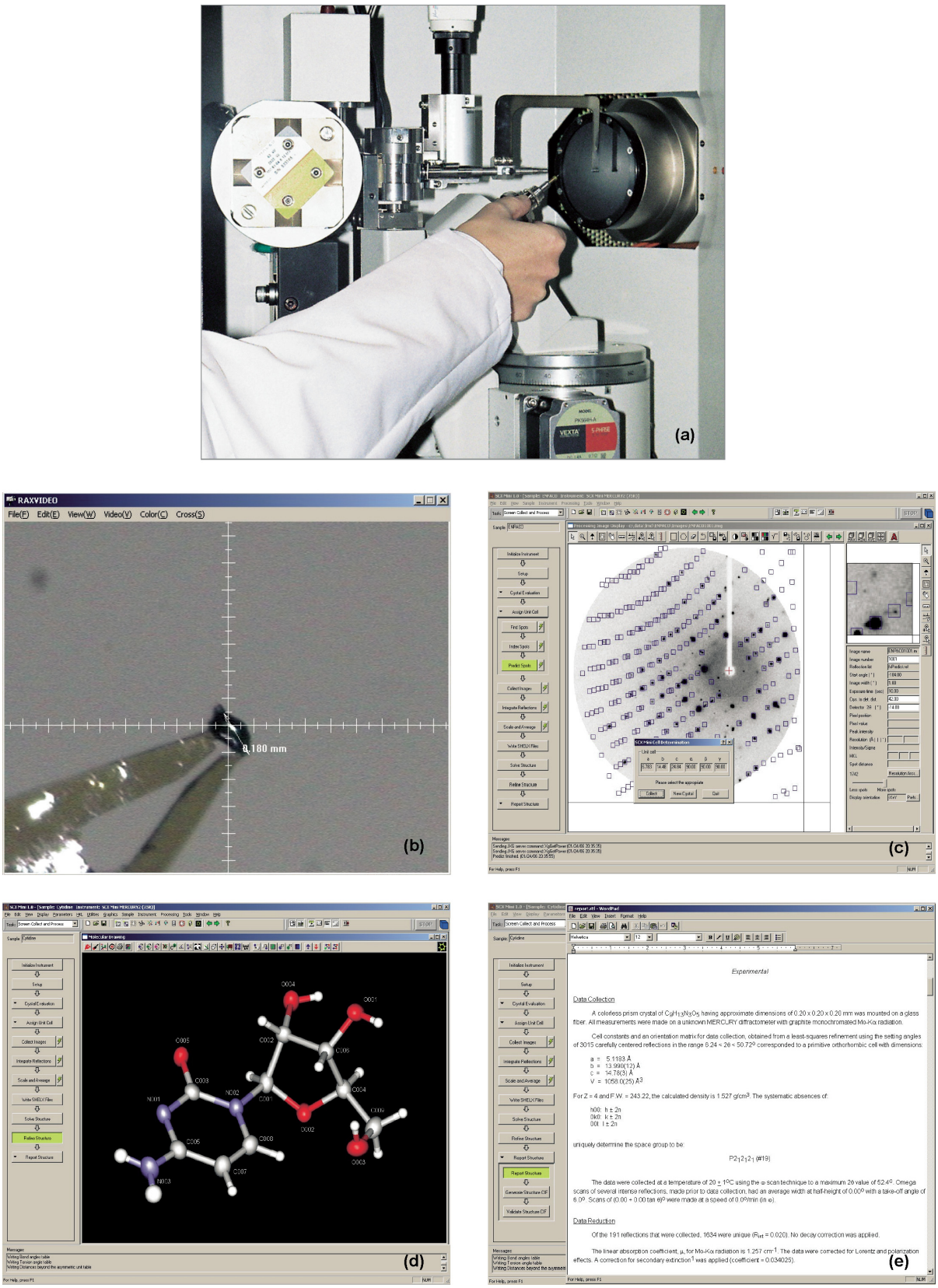


Fig. 2. Steps required to solve a crystal structure: mounting (a) and centering the crystal (b) is followed by automatic indexing and data collection (c). Subsequent steps include automatic structure solution/refinement (d) and report generation (e).

Data collection is followed by automated structure solution and refinement. The software displays the model of the structure in real time as the structure is calculated and refined. One-button report production gives the user a complete description of the experiment from beginning to end, and includes all the text and tables needed for the experimental section of a journal publication.

5. Conclusion

Rigaku's new SCXmini represents a paradigm breakthrough in small molecule crystallography. Specifically engineered to provide colleges, universities, and industry with self-serve access to definitive molecular structure determination, this new "mini" system allows single crystal diffraction to become a routine laboratory method and teaching tool in the same way that NMR, UV-VIS, HPLC and FT-IR did decades ago.

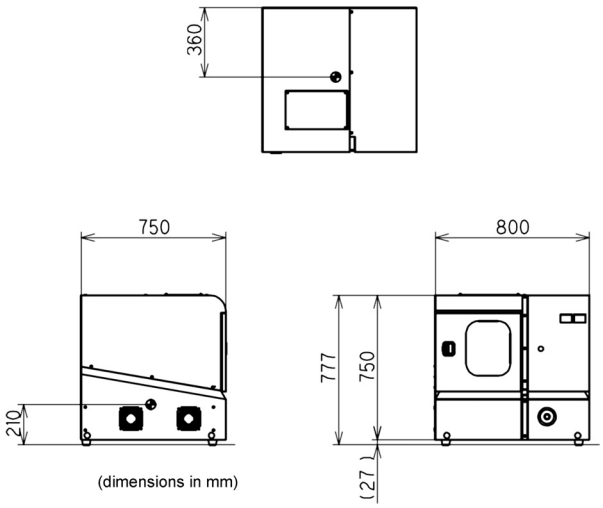


Fig. 3. Wire drawing of the Rigaku SCXmini system.

6. Specifications

Table 1 and Fig. 3 provides basic specifications for the SCXmini.

Table 1. SCXmini specifications.

Source:	
Type:	standard XRD sealed X-ray tube
Anode:	molybdenum
Voltage range:	20 - 60 kV
Emmission current range:	1.0 - 80.0 mA
Maximum power:	3 kW
High voltage stability:	± 0.01%
Optics:	optional high flux optics
Goniometer:	
Type:	AFC-Simplified with fixed 2θ, fixed χ
Angle ranges:	ω-axis: -120° to +60°; 2θ-axis: 20°/25°/30° fixed φ: ± 360°, free rotation for sample centering
Sample to detector distance:	50 mm
Visualization:	70X color CCD video microscope
Detector:	
Model:	Rigaku Mercury2 CCD
Sensor:	Kodak KAF-1001E
Pixel Array:	1024 by 1024 pixels
Aperture:	75 mm round
Computation:	
Software type:	Rigaku SCXmini
Computer platform:	IBM-PC compatible computer
Operating system:	Microsoft Windows®
Monitor:	17" LCD display
Other specifications:	
External components:	water-to-water chiller (requires chilled water)
Optional external components:	air cooled chiller
Operating environment:	15 - 24°C, 40 - 70% RH (non-condensing)
Weight:	250 kg
Power requirement:	3-phase 200/220 VAC, 20 A
Warranty:	1 year parts & labor