01-Instrumentation and Experimental Techniques (X-rays, Neutrons, Electrons)

For this newly synthesized material only a rather small crystal was available, of dimensions 270x60x40 μm. Laue diffraction photographs were recorded on the wigglers beamline at SERC Daresbury Laboratory, with exposure times <15 s.

For one set of photographs the incident beam was attenuated by a palladium foil (9.1 μm); from these photographs, in which the minimum wavelength is sharply defined by the palladium absorption edge (0.509 Å), the unit cell was determined on an absolute scale.3

Intensity data were measured from five film packs using the Daresbury Laue software suite.4 For these film packs the incident beam was attenuated by 0.2 mm Al and 0.114 mm Cu; these shifted the spectral distribution to shorter wavelengths, effectively 0.24-0.7 Å, reducing absorption, radiation damage, and background due to air scattering. 1241 individual intensity measurements were averaged to give 166 unique reflections from which the structure was solved and refined to R=0.12.

Subsequently an absorption correction was applied5 and the unmerged data was refined using SHELXL-92 (for which we are most grateful to Profs George Sheldrick). In the Laue method, different reflections are measured at different wavelengths, the anomalous scattering factors, f′ and f″, and therefore the structure factors, vary with wavelength, and this variation can be substantial for heavy atoms. SHELXL-92 can account for this variation, and refinement has given R1=0.075.

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01.02 - Synchrotron Data Collection for Macromolecules

MS-01.02.01. MACROMOLECULAR CRYSTALLOGRAPHY AT LURE: INSTRUMENTATION FOR X-RAY DIFFRACTION DATA COLLECTION AND RESULTS. by R. Fourm—, R. N. Kahn, W. Shepard and A. Benley. LURE, BP, 85903. Université Paris-Sud, 91405 Orsay, France

The x-ray sources available at LURE using the positron storage ring D23 are generated from bending magnets (critical wavelength λ~0.4 Å) and from a 5-pole wiggler (λ~0.4 Å). Synchrotron radiation is provided 90 hours per week for 31 weeks of the year. The ring is refilled only every 2 days since the decay time of the beam is 4-6 days. Such characteristics are favorable for uninterrupted data collection.

A total of 11 scans (including D1 and D22 on the wiggler line) will soon be available for macromolecular crystallography. Two of these are full-time machines, while the other stations are 20% shared with other disciplines. The station D11, in the process of being assembled, is a setup for unfocused Laue data collection. The station W11, in the process of being assembled, is a setup for unfocused Laue data collection.

For macromolecular data collection, a large imaging plate scanner (Two elliptically curved reflectors, a crystal and a multilayer) is equipped with a high-speed image plate scanner with an off-axis translation, Exposure times with X-rays from a Ge(111) crystal (λ=0.6 Å) are 1-30s/deg and 8-20s/deg from a Si(111) crystal (λ=0.4 Å). The station will soon be upgraded by increasing the diameter of the imaging plate from 25 cm to 32 cm. The smaller imaging plate will then be transferred to the part-time D43 station, which is equipped with a curved crystal monochromator.

For high precision data collection, large spherical drift MAPPs are equipped with a curved crystal monochromator. For high precision data collection, large spherical drift MAPPs are equipped with a curved crystal monochromator. The detector on D41 is an improved model with a higher spatial resolution, allowing data collection on crystals with larger unit cells. The station is equipped with a single bent crystal monochromator and is close to completion.