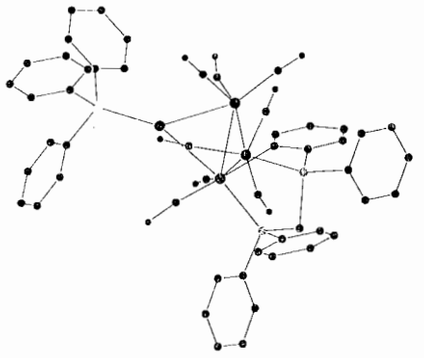
For this newly synthesized material[1] only a rather small crystal was available, of dimensions 270x60x40 μm. Laue diffraction photographs were recorded on the wiggler beamline at SERC Daresbury Laboratory, with exposure times <15 s.
For one set of photographs the incident beam was attenuated by a palladium foil (0.1 mm); from these photographs, in which the minimum wavelength is sharply defined by the palladium absorption edge (0.509 A), the unit cell was determined *on an absolute scale*[2,3].
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 A, reducing absorption, radiation damage, and background due to air scattering. 12183 individual intensity measurements were were merged to give 7163 unique reflections from which the structure was solved and refined to R=0.12.

Subsequently an absorption correction was applied[5] and the unmerged data was refined using SHELXL-92 (for which we are most grateful to Prof 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 allow for this variation, and refinement has given R1=0.075.

1. A K Smith and J Mathews, Liverpool University.
2. P D Carr, D W J Cruickshank, M M Harding (1992) J Appl Cryst 25, 294-308.
3. I M Dodd, P D Carr, M M Harding (1993) J Appl Cryst 26, in the press.
4. J R Helliwell et al, (1989) J Appl Cryst B24, 340-348.
5. S J Maginn, M M Harding and J W Campbell (1993) Acta Cryst B49, in the press.



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. Fourme*, R. Kahn, W. Shepard and A. Beniley, LURE, Bat. 209D, Universite Paris-Sud, 91405 Orsay, France

The X-ray sources available at LURE using the positron storage ring DCI are generated from bending magnets (critical wavelength λ_c=3.4A) and from a 5-pole wiggler (λ_c= 1.1A). 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 4-5 days. Such characteristics are favorable for uninterruped and accurate diffraction data collection. A total of five stations (including W11 and W32 on the wiggler line) will soon be available for macromolecular crystallography. Two of these are full-time stations, while the other stations are 50% shared with other disciplines.
The station W11, in the process of being assembled, is a setup for unfocussed Laue data collection. It will be equipped with a large imaging plate, as well as films, W32, our work horse for monochromatic data collection, has been running successfully since 1991. It features double focussing optics (two elliptically curved reflectors, a crystal and a multilayer) and a Hendrix-Lenfer imaging plate scanner with an off -axis translation. Exposure times with X-rays from a Ge(111) crystal (λ=1.5A) are 1-30s/deg and 8-240 s/deg from a Si(111) crystal (λ= 0.9A). This station will soon be upgraded by increasing the diameter of the imaging plate from 18cm to 32cm. 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 collections, large spherical drift MWPCs (in collaboration with G. Charpak's team, CERN) have been installed on the stations D23 and D41. The detector on D23 has been routinely used since 1988, and is associated with a sagittal focusing two-crystal monochromator for mutiple wavelength experiments. The detector on D41, an improved model with a higher spatial resolution, allows for data collection of crystals with larger unit cells. This station is equipped with a single bent crystal monochromator and is close to completion.
Selected results obtained by various user groups will be discussed, with emphasis on high resolution, large unit cells, MAD data collection and diffuse scattering measurements. The data quality obtained from MWPCs and imaging plate detectors will be compared.

