

Synchrotron chemical crystallography – Bill Clegg

Station 9.8 at the SRS is the world's leading synchrotron radiation single-crystal diffraction facility for chemistry and materials science. It is the most popular experimental station of the SRS and is used by a large number of research groups, for work across a wide range of chemistry and materials projects. Apart from a small but significant commercial use, and station development and commissioning, the facility is almost entirely taken up in recent allocation periods by EPSRC-ticketed grants. From October 2001 this includes the new synchrotron component of the EPSRC Chemistry Programme's national X-ray crystallography service, which is already proving to be a highly effective and rapid means of access to this unique and powerful facility for chemists with or without crystallographic experience.

With this pressure of demand, and with the rapid throughput of samples examined on Station 9.8 (typically 3 or 4 diffraction data sets per day, and our recently established record is 8, as part of the national service operation), efficient use of the time is crucial. The availability of the CDS, conveniently located also at Daresbury, is an enormous advantage in this respect. Users of the station are able to make connection to the CDS server for interrogation of the various structural databases, without leaving their experimental work, and so are able to obtain answers to queries rapidly, avoiding the wasted time that can arise from inappropriate experiments, such as the inadvertent redetermination of a known crystal structure.

For this purpose, the database of known crystal unit cells, CDIF, has been widely used. As soon as a unit cell has been determined for a sample (within minutes of mounting a crystal on the diffractometer), it is possible to establish whether this or a similar cell has previously been reported. On a number of occasions, the sample has thus been shown to be a starting material or by-product instead of the desired reaction product, and a fruitless full data collection, which may take a few hours, is prevented. More recently, the CDS staff have developed a new web interface to some of the database contents (CrystalWeb), and this includes a more convenient unit cell matching routine. A major advantage of the new method is that it automatically covers the contents of all the structural databases with a single search; the Cambridge Structural Database and Inorganic Crystal Structure Database are considerably more up to date than CDIF, but the latter contains many unit cells not known by the full structural databases, because the complete structures have not been reported. The web-based search is thus exhaustive, and users can be confident to proceed if they obtain a new unit cell. CrystalWeb also provides a very convenient LitLink connection to the original journal articles for published results, and uses MDL Chime to display molecular structures and unit cell contents if desired. It is a marvellous resource.

On-site access to the CDS databases is also very useful when structures are being determined from the data. Most users carry out at least the initial structure solution during their visit, and often much or all of the refinement can also be completed. Indeed, it is usually possible to obtain at least a preliminary result once a reasonable fraction of the data have been collected, since data processing is carried out in parallel with data collection. Comparison with previously reported structures can be very useful for a number of reasons at this stage; these include the construction of structural models for refinement, the question of how common or novel a particular feature may be, and the identification of solvates and polymorphs of known compounds (which may sometimes be another reason for interrupting and abandoning a data collection, if the structure proves to be relatively uninteresting and there are other, more important, samples available).

Thus the CDS enables users to make best use of a precious and limited experimental resource in pursuing a wide range of research projects, the majority of which are directly funded by EPSRC through standard grants as well as through other support to SRS operations.