ESRF	**Experiment title: Crystal structure solution from powder diffraction data via a global optimisation approach**	**Experiment number**: CH-500
Beamline: BM16	**Date of experiment**: from: 12th to: 15th Sept 1998	**Date of report**: 20th Aug 99
Shifts: 9	**Local contact(s)**: A.N. Fitch	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Prof. WIF David, ISIS Facility, Rutherford Appleton Lab, Didcot, Oxon UK.*

Dr. Kenneth Shankland, ISIS Facility, Rutherford Appleton Lab, Didcot, Oxon UK. *

Dr. Norman Shankland, Dept. of Pharmaceutical Sciences, Strathclyde Univ., Glasgow.

Miss Lorraine McBride, Dept. of Pharmaceutical Sciences, Strathclyde Univ., Glasgow. *

Dr. Alan Kennedy, Dept. of Chemistry, Strathclyde Univ., Glasgow. *

Report:

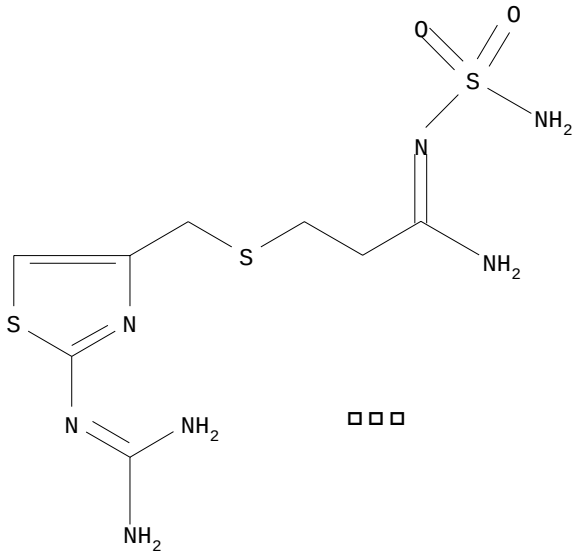
This proposal set out to determine a variety of crystal structures using a global optimisation approach to structure determination from powder diffraction data. We were successful in solving the crystal structures of ;

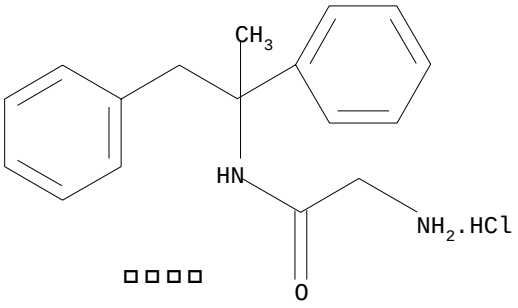
Famotidine forms A and B (diagram I)
Remacemide free base (see diagram II for hydrochloride structure)
Remacemide nitrate (see diagram II for hydrochloride structure)
Remacemide acetate (see diagram II for hydrochloride structure)
Pd(CN)2(Cl)2(Ph)2 (see diagram IV)
Uridine (not shown)

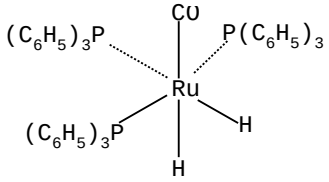
Seven crystal structures solved in 3 days represents an excellent use of beamtime. In addition, we collected other data sets that we were not able to solve ;

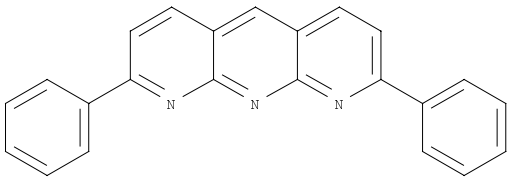
- **2,8-diphenyl-1,9,10-anthridine (V), as the sample had turned amorphous with time**
- **Remacemide hydrochloride, which with four independent fragments in the asymmetric unit represents a major challenge for our methodology**
- **A Ruthenium organometallic (IV), where a space group ambiguity exists**

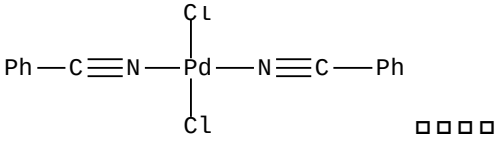
These latter two compounds will be solved very shortly with improvements in our methodology.











Conclusion

This experiment has demonstrated that it is now possible to solve many compounds in a very short time frame, thus making optimal use of valuable synchrotron beamtime. It has also demonstrated the power of the global optimisation approach to structure solution and highlighted some of its shortcomings. These shortcomings are being addressed and we confidently expect to be able to solve the remacemide hydrochloride structure and the Ruthenium organometallic structure in due course.

The high resolution and ease of use of BM16 (including the routine nature of low temperature work) were essential in allowing this experiment to be performed successfully. The famotidine structures and the palladium organometallic have been submitted for publication. The remacemide structures will be submitted shortly.