ESRF	**Experiment title: Crystal structures and supramolecular bonding motifs of azo pigments determined using powder diffraction data**	**Experiment number**: CH-849
Beamline:	**Date of experiment**: from: 12/4/00 to: 15/4/00	**Date of report**: 31st Aug 2000
Shifts:	**Local contact(s)**:Olivier Masson	*Received at ESRF:*

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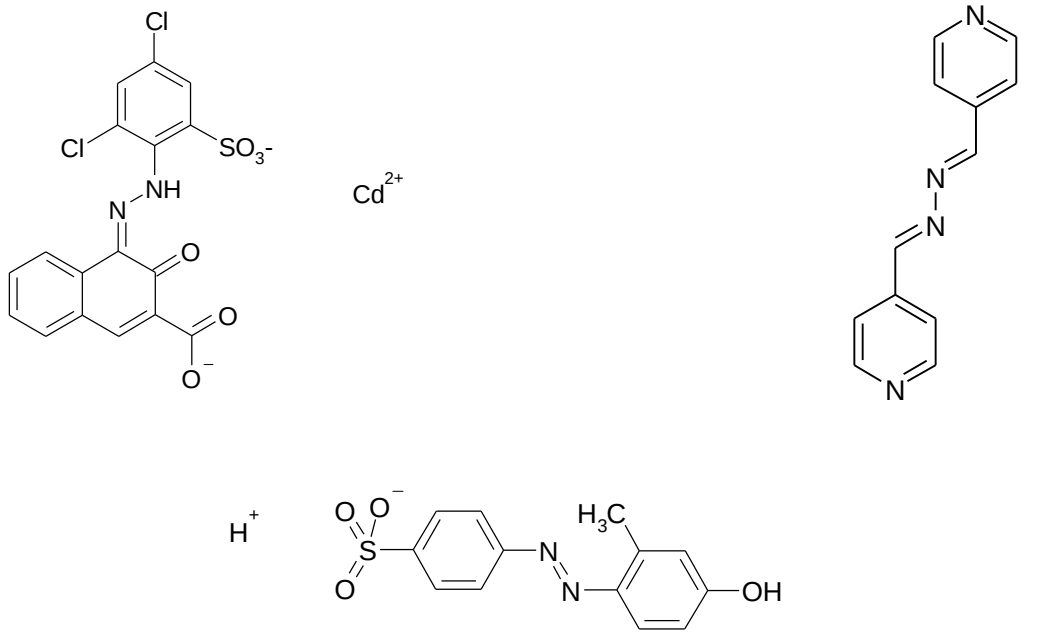
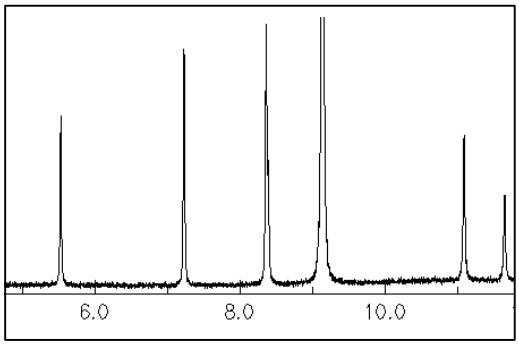
Report:

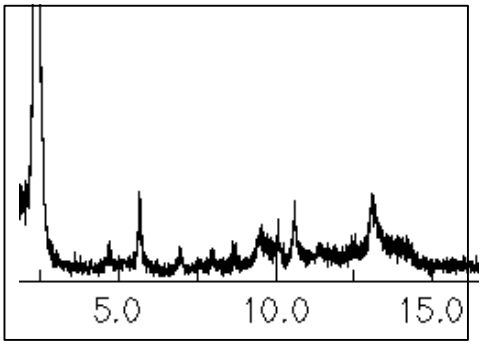
This proposal set out to study a number of azo pigment crystal structures of which a representative sample is shown below:



None of the crystal structures had been previously determined and none could be obtained as single crystals. The quality of the powder diffraction data was variable. Of the fourteen samples examined,

- **four failed to exhibit diffraction data of sufficient quality over sufficient range in order to merit complete data collection**
- **three have been indexed and solved**
- **another two have been indexed but not yet solved**
- **five have resisted indexing**

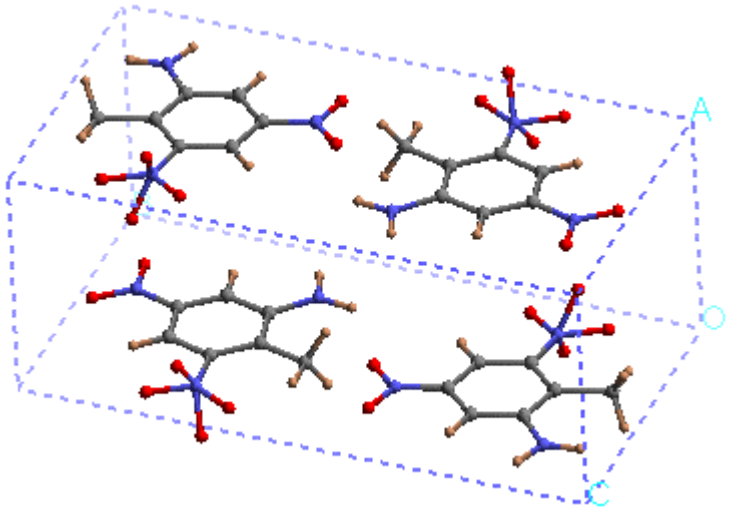




high quality data, indexes and solves ***insufficient diffraction to proceed***

It is clear from some subsquent microcrystal work that at least one of the samples fails to index due to the presence of a monoclinic and an orthorhombic phase. Interestingly, the existence of two phases for the compound in question was previously unknown.

One of the structures solved (reduced TNT) exhibited a rotational disorder of a nitro group, the presence of which was detected via the structure solution process and incorporated into it.



From our previous experience, it seems likely that most of the compounds that fail to index (despite exhibiting good diffraction) either possess large unit cells (with Z'>1) or consist of phase mixtures. We are currently working on computational strategies to overcome both problems.