ESRF	**Experiment title:** Structure determination of 3 interesting pharmaceutical compounds from powder data (metergoline and 2 risedronate sodium hydrates)	**Experiment number**: CH-1846
Beamline: BM01B	**Date of experiment**: from: 18/11/2004 to: 19/11/2004	**Date of report**: 23.2.2004
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Report:

Metergolin form II

High resolution powder diffraction record of metergoline form II was recorded at 0.69956 Å wavelength and at room temperature. 1.5 mm capillary was used. Indexation was done with program DICVOL: a monoclinic cell with following lattice parameters: a=20.353(9), b=5.061(3), c=22.29(1), Å β=99.382(7)° was suggested. All first 20 reflections were indexed with M_{20}=155.9 – fig. 1.

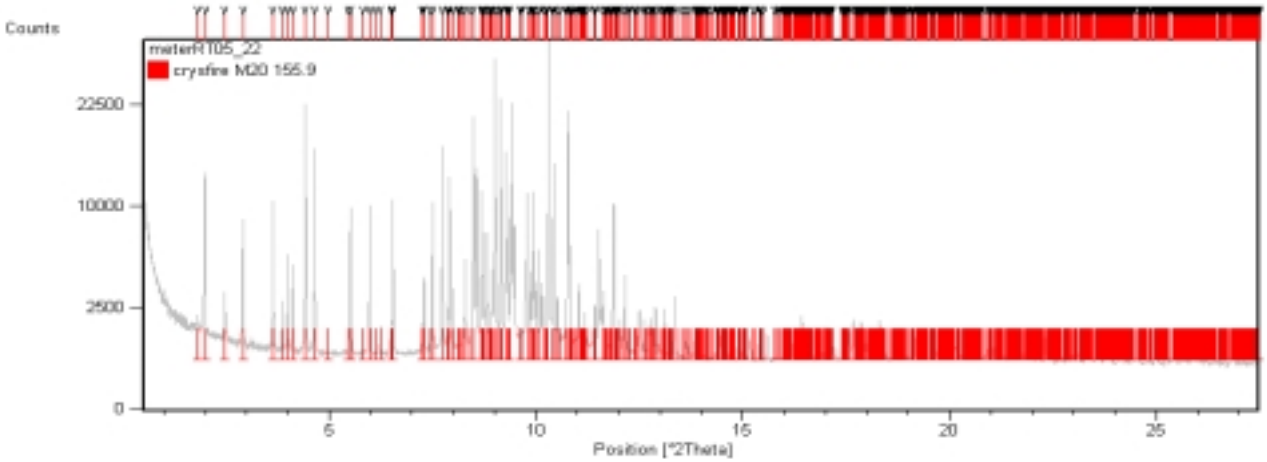

Fig.1 Powder diffraction record of metergoline form II with marked position of reflections predicted from indexation.

The compound is chiral – so the most possible space group is $P2_1$ with two metergoline molecules in asymmetric unit cell. The structure determination performed with program FOX based on search of positions of 2 independent fragments was not fully successful up to now.

Risedronate

High resolution powder diffraction record of risedronate was recorded at 0.69956 Å wavelength and room temperature - fig 2. 1.5 mm capillary was used.

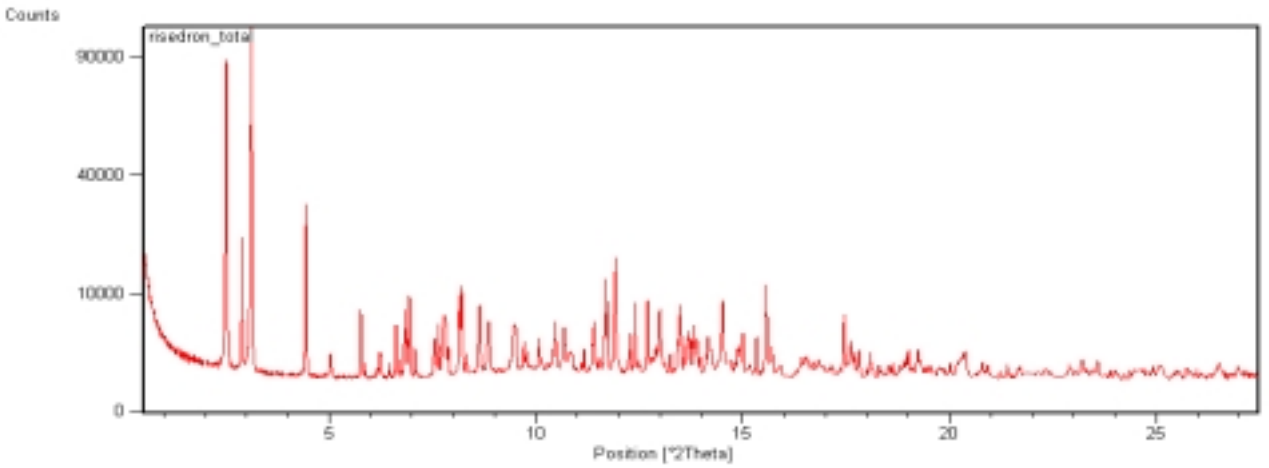

Fig.2 Powder diffraction record of risedronate sodium hydrate

None good indexation of the record was found by any indexation program. The problem is probably connected to unusual variations in peak width for which we do not have an explanation. The powder pattern corresponds to several others samples data obtained with a laboratory source – so we do not expect the problems are coused by presence of another phase.

Methylergometrine maleate

In addition to the compounds originally included in the proposal, we had time to measure an additional substance (ergot alkaloid similar to the metergoline). High resolution powder diffraction record of methylergometrin maleate was recorded at 0.69956 Å wavelength and room temperature. 1.5 mm capillary was used. Indexation was done with program DICVOL: orthorhombic cell with following lattice parameters: a= 5.711 (2), b= 12.771 (6), c= 33.15 (2) Å and space group $P2_12_12_1$ was suggested. The crystal structure was successfully solved by 2 fragment search in FOX. We have used the known structure of ergometrin as the first fragment and relatively rigid structure of the maleate as the second fragment. The missing methyl atom was found from the difference Fourier map fig.3. We continue working on restrained structure refinement in GSAS environment (current Rp=16.5% Rw=19.5 %) fig.4. The structure and packing is similar to already known structure of ergometrine maleate.

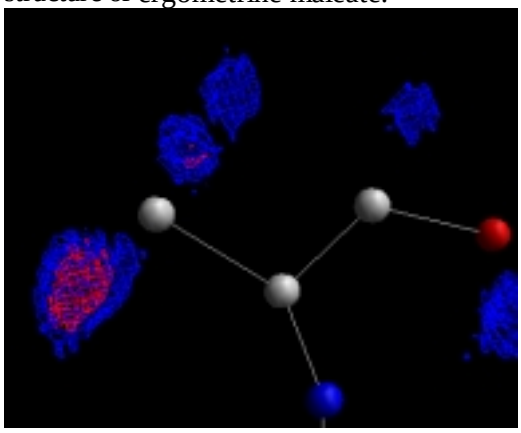

Fig.3 Finding of a missing methyl group form the difference Fourier map

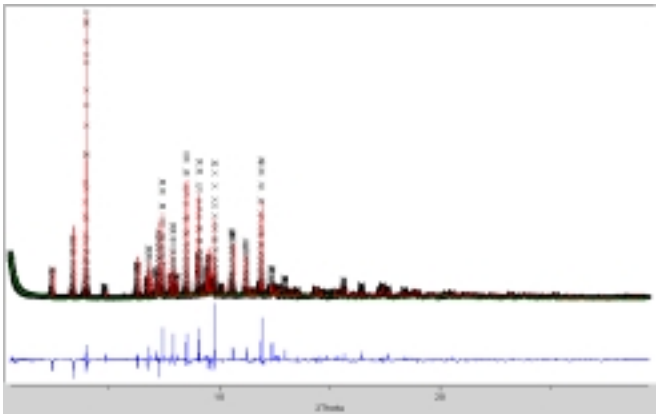

Fig.4 GSAS based refinement of methylergometrine maleate