s10.m1.p7 **Crystal Structures of HFCs and HCFC's by High Resolution Powder X-ray Diffraction.** M. Brunelli, A.N. Fitch, *ESRF, BP220, F38043 Grenoble Cedex, France.*
Keywords: powder diffraction, low-temperature, structure solution.

The crystal structures of a number of the replacement refrigerants, the hydrofluorocarbons or hydrochloro-fluorocarbons, (HFC's and HCFC's), have been studied. The gas is condensed *in situ* into a cooled, 1-mm-diameter, silica-glass capillary mounted on the high-resolution powder diffractometer BM16 at the ESRF. The gas-handling apparatus is disconnected, the liquified gas solidified by further cooling, and the sample spun about the capillary axis. High-resolution powder diffraction patterns have been acquired from which the crystal structures have been solved, using autoindexing, intensity extraction, then direct methods. A number of solid-state phase changes have also been discovered in some compounds by monitoring the diffraction patterns as a function of temperature. For routine work we have used the Cryostream for cooling down to 80K. To access temperatures down to 28 K, the Oxford Cryosystems Helix cold-helium-gas blower has been employed.

s10.m1.p8 **Crystal structure of complex organic compound by powder di ffraction methods.** W. Lasocha[I], P. Milart[I], A. Rafalska-Lasocha[I], H. Schenk[II]. *[I] Faculty of Chemistry, Jagiellonian University, Ingardena 3, 30-060 Kraków, Poland. [II] Laboratory for Crystallography, University of Amsterdam. Nieuwe Achtergracht 166, 1018 WV Amsterdam, The Netherlands.*
Key words: powder diffraction, structure determination, proton sponge

Crystal structure solution of organic compounds without heavy atoms by powder diffraction method is still a challenging task, particularly when there is no a rigid group in the compound, or when there are more than one molecule in an asymmetric unit. In this paper crystal structure solution of the complex of proton sponge 1,8-bis (dimethylamino) naphthalene (DMAN) and *p*-nitrosophenol: $C_{14}H_{18}N_2 \cdot 2(C_6H_5NO_2)$ is presented. The crystal structure with 25 atoms and three independent molecules in the asymmetric unit was 'ab initio' solved from powder data by 'pseudo-atom' method[1]. In this method rigid organic groups are replaced by 'pseudo-atom' containing the same number of electrons. After Rietveld refinement of pseudo-atom model (R_F and R_{wp} were 41 and 42%, respectively), 'real' atoms can be found by difference Fourier method. Due to small number of unique extinctions (the ratio of parameters c^2/b^2=3.00015) the space group determination was the most difficult and ambiguous part of this work.

Crystal data: space group Pnma (62), a=12.2125(5), b=10.7524(7), c=18.6199(14) Å. Measurement at ESRF Grenoble, BM 16, λ=0.65296 Å.

Methods and programs: pattern decomposition - LSQPROF, structure solution - SHELXS, Rietveld refinement - XRS-82.

[1] Dinnebier, R.E.: Supramolecular structures from high resolution powder diffraction. IUCr XVII Congress, Seattle, (1996) MS02.05.01
***A*cknowledgement: The support of Polish Committee of Science.** *Research (KBN, 0312/T09/98/15) is kindly acknowledged.*