ESRF	**Experiment title: Powder diffraction study of the incommensurate modulated ferroelastic phase of alpha-lead monoxide**	**Experiment number:** CH-423
Beamline: BM16	**Date of experiment:** from: June 19 1998 to: June 22 1998	**Date of report:** Aug 29 1999
Shifts: 9	**Local contact(s):** Dr. Andy Fitch	*Received at ESRF:*

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Report:

5 shifts were necessary to align the beamline, set up the cryo device and get rid of all the parasitic background noises for an optimum peak to background ratio. Two complete almost background free diffraction patterns were recorded in the same experimental conditions: the former at 90 K, the other one at room temperature. It is well known that lead monoxide is very sensitive to carbonates, water adsorption and strain and it is almost impossible to obtain pure samples of pure alpha phase. Therefore, a very careful comparison of the high and low temperature diffraction pattern was necessary to assess the satellite indexing.

The analysis of the main peaks showed that the sample was almost single phased with less than 0.2% of β-PbO.

The analysis of the whole pattern confirms the good crystalline quality of the sample; because of the lamellar structure characterizing this compound, the profile of the 00l diffraction peaks presents an asymmetric broadening. The satellite peaks are slightly larger than the average structure reflections.

The room temperature and the low temperature patterns were refined using the Rietveld program XND. Two models have been tested at present for the incommensurate phase:

- the space group Cmma:s-1 1 proposed by an electron microscopy study [1] (a summary of this refinement is given below)
- the space group C2mb:-1-1 1 proposed by a preliminary X-ray and neutron diffraction study [2,3]. This refinement, almost completed, gives results slightly better than the previous one for the satellites reflections.

Since no second order satellite has been observed unambiguously, the extinctions affecting satellite peaks have been tested only on first order satellites. It should be underlined that the centrosymmetric group allows the refinement of a very limited number of parameters since most of the modulated displacements are symmetry fixed.

Cmma s-1 1	X	Y	Z	B	u(sin)	w(cos)
Pb	0	¼	-0.2373	0.45	0.018	0
O	¼	0	0	0.40	0.2323	0.0286

Rwp=14.1% S=2.08, RI(overall)=6.15%, RF(main)=4.51%, RF(satellites)=6.57%

1 Withers, Schmid, J. Solid State Chem. **113** (1994) 272
2 Moreau, Kiat, Garnier, Calvarin, Phys. Rev. B **39** (1989) 10296
3 Hédoux, Grebille, Garnier, Phys. Rev. B **40** (1989) 10653