

upon convolution with the undistorted lattice could give rise to Moiré fringes as were observed in the images. High resolution EM substantiated by image computation confirms the existence of these defects on an atomic scale.

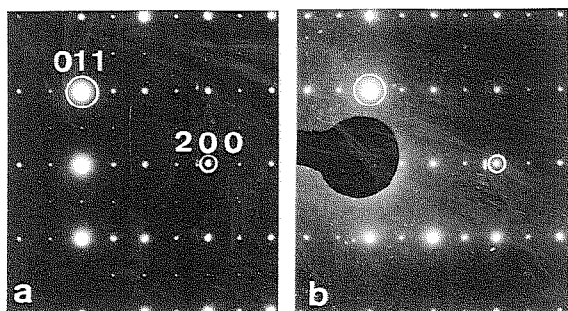


Fig. 1 : Phase transition in  $\text{RbFeF}_4$  as observed with electron diffraction in  $a[011]$  zone pattern. (a) RT (b) LNT

05.1-33 AN X-RAY DIFFRACTION STUDY OF THE PHASE TRANSITION IN  $\text{PbHPO}_4$ . By A. Katrusiak\* and R.J. Nelmes, Department of Physics, University of Edinburgh, Mayfield Road, Edinburgh EH9 3JZ, U.K.

$\text{PbHPO}_4$  (hereafter LHP) has a second-order phase transition at  $T_C = 310$  K. Phenomenologically this transition is very similar to that in  $\text{KH}_2\text{PO}_4$  (hereafter KDP). LHP has a particularly simple crystal structure in which the  $\text{PO}_4$  groups are linked into one-dimensional chains by hydrogen bonds. Above  $T_C$  the protons are disordered over two sites in these bonds, and below  $T_C$  become fully ordered onto one of the sites — as in KDP. It is generally assumed that such transitions, of the KDP type, are "driven" by this proton ordering. One respect in which LHP differs from KDP is that its spontaneous polarisation,  $P^S$ , rises only slowly with falling temperature below  $T_C$ , and does not reach saturation until approximately  $T_C - 100$  K. A neutron-diffraction study (Nelmes, Ferroelectrics (1980) 24, 237) has shown that the degree of proton ordering (from 50/50 above  $T_C$ , through 75/25, to 100/0 at  $-T_C - 100$  K) has the same temperature dependence as  $P^S$ . But, by contrast, the heavy-atom displacements (e.g. the distortion of the  $\text{PO}_4$  groups) were found to reach their saturated values only  $\sim 10$  K below  $T_C$ . This difference is an unexpected and puzzling result that challenges the generally accepted understanding of transitions of this kind as being "driven" by the proton ordering, with strong coupling between the proton and heavy-atom fluctuations. To check the neutron-diffraction result for the (rather small) heavy-atom displacements, and obtain information about these displacements closer to  $T_C$ , we have now carried out a careful, high-resolution X-ray diffraction study of LHP. Data have been collected at  $T_C + 10$  K, in the range  $T_C - 2$  K to  $T_C - 20$  K, and at  $\sim T_C - 100$  K. The combined results of the neutron-diffraction and X-ray diffraction studies will be presented. \*On leave from A. Mickiewicz Univ., Poland.

05.1-34 THE INCOMMENSURATE-COMMENSURATE PHASE TRANSITION IN  $\text{Rb}_2\text{ZnCl}_4$ . By K. H. Ehses, U. Schürmann, Fachrichtung Kristallographie, Universität des Saarlandes, D-6600 Saarbrücken Federal Republic of Germany.

For the investigation of the modulation vector in incommensurate (IC) phases, a high resolution in reciprocal space is necessary. Such a resolution can be obtained by means of our double-axis-diffractometer 'AMADEUS' in the dispersion free arrangement, where the resolution is only determined by the sample quality.

In the space group  $\text{Pcmn}$  for  $\text{Rb}_2\text{ZnCl}_4$  the incommensurate wave number is  $q_z = (1/3 - \delta)c^*$ . The half width of the IC-reflections is clearly greater than that of the main reflections. The temperature dependence of the parameter  $\delta$  coincides over a large temperature range with that of previous measurements (H. Mashiyama, J. Phys. Soc. Jap. (1982) 51, 2538). But at  $T_C$  the transition temperature to the ferroelectric phase, we find a discontinuous behaviour of  $\delta$ . There is a region of about two degrees, where the IC- and the commensurate (C) peaks coexist. In this range the half width of the IC-reflection increases to  $T_C$ , the commensurate one remains constant. The integrated intensity of the IC-satellite decreases to  $T_C$ , whereas that of the corresponding C-reflection increases, the sum remaining nearly constant.

The influence of an electric field on the behaviour of  $\delta$  has been investigated.

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05.1-35 LOW TEMPERATURE PHASE TRANSITIONS IN  $\text{LiKSO}_4$ : A NEUTRON DIFFRACTION STUDY. By Sandhya Bhakay-Tamhane, A. Sequeira and R. Chidambaram, Neutron Physics Division, Bhabha Atomic Research Centre, Trombay, Bombay 400 085, India.

A single crystal neutron diffraction study of the low-temperature structural phase transitions in  $\text{LiKSO}_4$  has been carried out using the closed-cycle cryo-tip on the 4-circle diffractometer at the CIRUS reactor, Trombay. Various other techniques (laser Raman, EPR, NMR, IR, etc.) employed earlier for studying these phase transitions have given somewhat different descriptions of the phases and the transition temperatures in this system.

The room temperature neutron diffraction structure of  $\text{LiKSO}_4$  (Bhakay-Tamhane, Sequeira & Chidambaram, Acta Cryst. C, (1984)) has the space group  $\text{P6}_3$  with  $a = 5.140(1)$ ,  $c = 8.636(2)$  Å and  $Z = 2$  (though the alternate space group  $\text{P2}_1$  could not be ruled out) and all the samples studied show merohedral twinning about the  $[110]$  axis. The intensities and profiles (in  $\theta$ - $2\theta$  scan) of a group of 20 Bragg reflections were studied from room temperature down to 150K. Cooling the sample down to about 200K resulted in a small general increase in the intensities of the reflections which could be accounted for by the Debye-Waller factor. Below 200K however, the Bragg intensities undergo pronounced changes. This transition to a new phase takes over a day to equilibrate at 190K. From about 189K, many of the Bragg reflection profiles are characterised by split peaks (which start as shoulders to the main profile) and this continues to the lowest temperatures reached in this study. The relative strengths and positions of the various peaks within a profile, however, change with temperature and time. These changes are very sluggish and the system takes several days to