

and each structure may be described in terms of two c glide related sub-structures of $C-1$ symmetry. The coexistence of the substructures destroys the inversion as a symmetry element. Only half the c -glides of $P2_1/c$ are retained, the choice between halves selects between equivalent structures that are inversion related. Within the substructures only half of the inversion centres of $P2_1/c$ are retained and the choice between halves gives geometrically distinct options. Only the intensities of the weak extra reflections are sensitive to this choice, the wrong choice being unrefinable. The correct choice gave a sensible H-bonding network between O atoms while the other did not. Unconstrained refinement in spacegroup Cc creates difficulties for atoms that overlap in the average structure. This was overcome using features available in the Constrained Least Squares Refinement Program *RAELS92*. The 4 molecules in the asymmetric unit were constrained to have identical geometry that included a local mirror plane. This geometry was refined using local coordinates defined relative to refinable local orthonormal axial systems. Refinable *TLX* rigid body thermal parameterisation were used for each molecule, molecules inversion related in the substructure maintaining this relationship. The inversion symmetry of the substructures need not be maintained but departure from this was found to be minimal.

PS02.01.17 STRUCTURE DETERMINATION OF $PbZrO_3$ USING X-RAY ANOMALOUS DISPERSION. K. Yamasaki, Y. Soejima, Department of Physics, Kyushu University, Fukuoka 812-81, Japan, K. F. Fischer, Fachrichtung Kristallographie, der Universität des Saarlandes, D-66041 Saarbrücken, Germany (FRG)

On the basis of the method for superlattice structure analysis using X-ray anomalous dispersion, the intensity of several superlattice reflections from a $PbZrO_3$ single crystal have been re-examined: the intensity measurements were made as a function of incident X-ray energy around the Pb L_{III} and Zr K absorption edges. Since the structure factors of superlattice reflection are directly concerned with the displacement of anomalous scatterers from the higher symmetry position, they are expected to vary around the absorption edge when the anomalous scatterers have displacement. On the other hand, the structure factor is invariant when anomalous scatterers have no displacement. The experimental results show: Pb atoms have displacements only on the ab plane in the orthorhombic unit cell, and the pattern of the displacement is in anti-phase along the c direction. Zr atoms have an anti-phase type of displacements both on the ab plane and along c . These results support the structure determination by Glazer, Roleder and Dec (1993), except for the Zr displacement along c .

For refining our model, data were collected using 0.35 \AA radiation up to $\sin\theta/\lambda=1.0 \text{ \AA}^{-1}$. To avoid a serious problem of domains, and to obtain information on the atomic displacement as precisely as possible, only superlattice reflections were used. The structure has been determined with R-factor 5 %: the results show anti-phase displacements of Zr atoms along c ; this is consistent with the results of the structure factor examination mentioned above. The present investigation requires that the antiphase type of displacement of Zr atoms must be considered.

PS02.01.18 INTERESTING INFORMATIONS FROM MODELING AND REFINEMENT OF DISORDERED MOLECULAR STRUCTURES. Hoong-Kun Fun and K.Sivakumar, X-Ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 Penang, Malaysia

Nowadays crystal structure determination of small molecules is nearly an automated process with little or no human intervention due to the availability of high speed computers and sophisticated software packages. However it is non-trivial when problems are faced in individual cases where the structure has some form of disorder. This then needs the role of a crystallographer for the careful analysis of the refinement results and to carry out the next step with reasoning. Analysis of the mode of disorder and suitably carrying out the refinement with constraints and restraints come with experience. In our laboratory, we have come across many such occasions with different types of disorder existing in the molecular structures. Some examples of disorders are those due to the conformational flexibility of certain rings and mixture of two isomorphous structures. A detailed survey of the disordered cases encountered in our laboratory and how the structures were refined will be discussed. The presentation will also include how to identify the minor conformational components present in the structure and model them to get better structural information.

PS02.01.19 SMALL CRYSTALS ARE OFTEN POOR CRYSTALS - WHY? Marjorie M Harding, Robert J Rule, Department of Chemistry, University of Liverpool PO Box 147, Liverpool L69 3BX, UK

Diffraction patterns recorded with synchrotron radiation have given us some clues. We have recorded synchrotron radiation diffraction data for structure determination for a substantial number of very small single crystals ($< 0.02 \text{ mm}$ for example). In most cases the use of synchrotron radiation was a 'last resort' because it had not proved possible to grow crystals of good size and quality suitable for use with conventional X-ray generators and diffractometers.

A large proportion of small crystals are either very thin plates or very fine needles; this is probably associated with poor growth in one or more directions in the structure. Three kinds of observation support this view:

a) For many of the small crystals studied because they would not grow bigger, the mosaic spread is unusually large (it can be quite accurately estimated in synchrotron radiation experiments because of the low beam divergence).

b) Following structure determination it is often possible to identify structural features which could explain the potential for faulted growth. Examples will be given.

c) The geometry of the Laue method for recording single crystal diffraction patterns is particularly sensitive to mosaic spread. By this method we have shown, for two compounds which form very fine needle crystals, that the mosaic spread is anisotropic, and is larger in the direction perpendicular to the needle axis than parallel to it. One structure is an aluminophosphate, with pores parallel to the needle axis; the other is organic, but also has large channels parallel to the needle axis.

So although synchrotron radiation has allowed structural information to be derived for a number of very small crystals, many of them were faulted. This prevented us from obtaining geometrical data of the highest quality, and probably also prevented them from growing larger.