so than amplitudes determine the form of electron density maps. Vector (Patterson) maps contain images related by the the twin law. There appear to be no circumstances where least-squares refinements should be made with detwinned data, since the process of detwinning unavoidably introduces considerable noise into what might have been an excellent data set. Various examples will be used to illustrate strategies for the identification of twinning (pre- and post-data collection), derivation of the twin law, and solution and optimal refinement of structures.

MS02.01.05 THE APPLICATION OF SHAKE AND BAKE TO UNSOLVED STRUCTURES. R. Curtis Haltiwanger, Drake S. Eggleston, SmithKline Beecham Pharmaceuticals, UW-2950, P.O. Box 1539, King of Prussia, PA 19406

Shake and Bake(SnB)[1], an ab initio dual-space, multiple-solution direct-methods procedure is a powerful addition to the suite of modern structure solution packages. We have successfully applied it to several previously unsolved structures of varying complexity. These vary from a 220 atom structure in space group $P2_1$ to a 120 atom structure in space group P1. Details of the implementation and successful application of SnB in our laboratory will be presented.

[1] R. Miller, S. M. Gallo, H. G. Khalak, and C. M. Weeks, *J. Appl. Cryst.* **27**, 613-621(1994)

MS02.01.06 STRUCTURE DETERMINATION USING CCD AREA DETECTOR - HOW SMALL A CRYSTAL IS TOO SMALL? Nigam P. Rath, Department of Chemistry, University of Missouri-St. Louis, 8001 Natural Bridge Road, St. Louis, MO 63121-4499, USA.

A Siemens CCD area detector system has been used to collect data on several "very small" crystals and their structure solution and refinement achieved by using SHELXTL 5.0 programs for structure determination. In some cases, structure has been re-determined using a bigger crystal and a comparison of results will be presented.

Diffraction data has been collected on the same crystal for organic and organometallic compounds using point detector diffractometer (Siemens P4) and CCD area detector system for comparison of structural results. Advantages and disadvantages of both the detector types will be discussed. Efforts are underway to develop data collection strategies for small crystals and weak diffractors for use with CCD area detector system. Our experience with the area detector system for small molecule structure determination, both high-points and pitfalls, will be presented.

PS02.01.07 MODELING CRYSTAL STRUCTURES WITH DISORDER USING THE SHELXTL 5.0 SOFTWARE PACKAGE. Izya F. Burshtein and Charles F. Campana, Siemens Energy and Automation, Inc., Analytical Instrumentation, 6300 Enterprise Lane, Madison, Wisconsin 53719-1173, USA.

In the past, the existence of disorder in crystal structures has often prevented investigators from successfully completing the refinement of such structures, even though the X-ray intensity data were of very high quality. As single-crystal X-ray diffraction is utilized to solve increasingly large numbers of important real-world structural analysis problems, the ability to recognize and solve such problems becomes even more essential. The SHELXTL 5.0 Software Package includes a number of useful tools which allow complex disorder problems to be analyzed, modeled and successfully refined.

We intend to show more than a dozen examples in which the SHELXTL package has been used to model a variety of interesting disorder problems. We will present primarily structures where the successful modeling of disorder played the key role in the correct interpretation of the diffraction data. Successful modelling and refinement of a disordered structure not only improves R-values but provides a chemically reasonable description of the structure.

We will describe some of the clues that allow disorder problems to be recognized and graphical methods which may be employed to interpret the disorder mechanism and to construct the model. Specific examples of SHELXTL refinement files illustrating new features will be provided..

PS02.01.08 A NEW EXTENSION OF THE 'LEARNT-PROFILE' METHOD. By Ewa Gałdecki, Institute of Low Temperature and Structure Research, Polish Academy of Sciences, ul. Okólna 2, 50-950 Wrocław, Poland

As proved by the author, the use of 'learnt profiles' as models of measured X-ray single-crystal diffraction peaks rather than selected 'shape functions' leeds to a very effective data processing. The 'learnt profile' can be determined and used in such a way as in appropiate original papers [Clegg, W. (1981), *Acta Cryst.* **A37**, 437, and references quoted therein], but the idea may also be extended. In a system of data-processing computer programs written by the author one may learn the shape(s) of either

(i) **the double peaks** (for various ranges of ϑ values), or

(ii) **the single peak** (one of the components of the $K\alpha$ doublet), or

(iii) **the crystal profile**, that is one of factors of the physical convolution model (the other ones being the spectral distribution for given source of X-rays, and some aberration functions).

In the case (i) of the **'simple learnt-profile'** method, one should collect models of the double-peak shapes within a number (say 25-50) of ϑ ranges (the strongest peaks are selected and averaged), the models are stored in their real angular scale. Thus the widths of the peaks are given. These may be adjusted in the next stage. Using the **'basic learnt-profile'** method, described in the original papers, case (ii), it is necessary - apart from learning the single-peak shape (stored by the present author in a standardized form, as a spline function) - to derive some information on the width of such single peaks, that is treated as a function of ϑ and the direction cosines. In the case (iii) of the **'convolution'** or **'learnt crystal-profile'** method, the spectral distribution of the given $K\alpha_1$ - $K\alpha_2$ line must be known. It should be expressed as a function of angle for respective ϑ values. The function is used for both the crystal-profile determination - *via* a deconvolution of the observed profile in relation to the spectral distribution - and, in the next stage, for syntheses of the profiles. As results from the author's tests, the efficiency of the methods is very good. Their properties and possibilities will be discussed in detail.

PS02.01.09 COMMENSURATE STRUCTURES DESCRIBABLE IN TERMS OF DEGENERATE IRREDUCIBLE REPRESENTATIONS. By A. David Rae, Research School of Chemistry, Australian National University, Canberra, ACT, Australia, 0200

When trying to understand problems in structure solution and refinement, many structures are best described as commensurate modulations of parent structures with a smaller unit cell. The diffraction pattern may then be described in terms of parent reflections **g** and satellite reflections **g** + **k** where **k** has a limited number of values, in the simplest case just one. The Fourier transform of the parent reflections describes a parent structure which is a disordered version of the true structure and is often quite easy to obtain, especially when the inherent disorder is recognised. The satellite reflections then define a modulation of the parent structure and possible resulting symmetries can be