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 Galdecki, Institute of Low Temperature and Structure Research, Polish Academy of Sciences, ul. Okólna 2, 50-950 Wroclaw, 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

obtained from the irreducible representations associated with **k**. This approach is often more effective than using phase determination methods on structures where heavy atoms dominate reflections **g** but make effectively no contribution to reflections **g** + **k**. The increase in primitive cell volume by a factor N implies that, at most $1/N$ of each set of translationally related symmetry elements {R,t} of the parent structure survive, and a choice is often required. The behaviour of atoms on special positions of the parent structure can be informative, e.g. a heavy atom on an inversion centre in a parent $P2_1/c$ structure may be on a general position in the modulated F-1. In some cases none of a set {R,t} survive or certain elements {R,t} and {R',t'} cannot coexist. This situation is associated with the existence of a degenerate irreducible representation. Disallowed symmetry operations create a different orientation of the true structure but may still describe a localised symmetry or be associated with twinning or disorder. When the equivalent point symmetry of the true structure is less than that of the parent structure, the parent symmetry is lowered and this may be detectable as absence conditions not quite holding, e.g. $Pccn$ modulated to $C112/d$. Problems with refinement often exist in such structures and these are generally associated with atoms overlapped in the parent structure. Constrained refinement and the maintenance of the parent symmetry for thermal parameters is useful. A number of examples of structures solved using this approach will be given and the nature of the refinement problem detailed.

PS02.01.10 EXAMPLES OF NON-ROUTINE APPLICATIONS FOR CCD-BASED DETECTOR SYSTEMS IN THE FIELD OF SMALL MOLECULE CRYSTALLOGRAPHY.

Charles F. Campana, Siemens Energy and Automation, Inc., Analytical Instrumentation, 6300 Enterprise Lane, Madison Wisconsin 53719-1173, U.S.A., and Eric R. Hovestreydt, Siemens AG, Abt. AUT V371, Siemensallee 84, D-76181 Karlsruhe, Germany.

Over the past two years we have used standard Siemens CCD-based SMART diffractometer systems equipped with a Mo-target sealed-tube X-ray source to collected several hundred publication-quality data sets on good quality single-crystal specimens. These data sets have clearly demonstrated that this system is capable of collecting better quality data than conventional scintillation counter diffractometers in a much shorter period of time. For small or weakly diffracting crystals, measurements on the same specimen have shown that the CCD systems running at 2 kW power often yield more observed data and better structures than conventional scintillation-counter diffractometers on rotating anode generators running at 15 kW.

In addition to these studies, however, we have utilized the CCD system to investigate problems which would be very difficult, if not impossible, to pursue with conventional point-detector systems. We will present examples from our applications laboratories which demonstrate the power of CCD-based detector systems for non-routine applications. These examples will illustrate potential applications which include twinning, pseudosymmetry, superlattices, incommensurate structures, phase transitions, long axis and large unit cell problems, and characterization of non single-crystal specimens.

PS02.01.11 X-RAY CRYSTAL STRUCTURE ANALYSIS BY A NEW DIFFRACTOMETER USING IMAGING PLATES.

Hidehiro UEKUSA and Yuji OHASHI, Department of Chemistry, Tokyo Institute of Technology, Ookayama, Tokyo 152, JAPAN.

Recently we have designed and made a new type of diffractometer using two imaging plates for rapid data collection. The new diffractometer is a Weissenberg camera type mechanism with a feature of a k-type goniometer, two imaging plates, and spiral motion reading mechanism. The total accuracy of the new diffractometer is comparable to that of a four circle diffractometer despite a very short measurement time. It is also equipped with a new type of variable temperature system using nitrogen gas stream, which covers a temperature range of 80 K to 420 K.

Following experiments are successfully performed by this diffractometer.

(1) Structure determination of the X-ray sensitive molecule. The initial molecular structure of syn-tricyclooctane derivative has been determined for the first time by 140 minutes measurement, which has not been analyzed due to the rapid bond cleavage reaction under X-ray irradiation.

(2) Data collection without sealing in a glass capillary. Despite of the bad condition that the crystal was easily decomposed by the loss of solvent methanol, the structure of cobalt(II) complex with N-glycoside, which is a model of metal containing enzyme, has been determined by 160 minutes measurement without sealing at room temperature.

(3) Structure after phase transition. The structural change of an organic charge transfer complex due to the phase transition has been clarified by utilizing the low temperature equipment and rapid data collection.

PS02.01.12 STRUCTURE DETERMINATION OF GLUCOSAMINE-6P SYNTHASE: FIGHTING AGAINST PSEUDOSYMMETRY.

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Structure determination of the glutaminase domain of glucosamine-6-phosphate synthase faced difficulties due to pseudosymmetry of the crystals. X-ray diffraction data collected to 1.8 Å resolution indicated strong pseudo C-centering of the primitive orthorhombic lattice which was confirmed by the peak at (0.5, 0.473, 0.0) in the native Patterson map. Molecular replacement with the model of PRPP amidotransferase gave solutions in two possible space groups, $P2_12_12$ and $P2_12_12_1$, indistinguishable due to pseudoabsences. Neither of these solutions could be further refined. Attempts to solve the structure by isomorphous replacement were also unsuccessful. Difference Pattersons for putative heavy atom derivatives were uninterpretable in spite of specific binding in many of them as appeared later. Accidentally, a crystal in the space group $P2_1$ was found which had the same packing of the molecules but without pseudocentering. While using the PRPP amidotransferase model for molecular replacement in this crystal form failed, the preliminary refined "orthorhombic" model gave a unique solution which was successfully refined at 2.4 Å. The phases were improved by solvent flattening and electron density averaging around a non-crystallographic two-fold axis. However, the back transfer to the orthorhombic form did not work until it was realized that the symmetry of the crystal could be lower than orthorhombic. Finally, the structure was refined using the 1.8 Å data reprocessed in the space group $P2_1$ (no difference in R-merge). The R-cryst is 18% for all reflections in the range 10-1.8 Å. Analysis of the structure shows that the pseudo-orthorhombic lattice consists of two sublattices shifted to each other by 4 Å.