

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 collect 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.** Alexei Teplyakov<sup>1</sup> and Michael N. Isupov<sup>2</sup>, <sup>1</sup>EMBL, c/o DESY, Notkestr. 85, Hamburg 22603, Germany, <sup>2</sup>Department of Chemistry, University of Exeter, Exeter EX4 4QD, UK.

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,  $P2_12_12_1$  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.