PS01.06.11 THERMAL-DIFFUSE-SCATTERING CORRECTIONS FOR SINGLE-CRYSTAL DIFFRACTION DATA FROM AREA DETECTORS. G.J. McIntyre, Institut Laue-Langevin, B.P. 156, 38042 Grenoble Cedex 9, France.

X-ray and neutron single-crystal diffraction studies on the same compound consistently give comparable coordinates, but frequently quite different displacement parameters. Attributed mainly to uncorrected thermal-diffuse-scattering (TDS) contributions, these differences can be considerably reduced by using an area detector in the neutron experiment to give an integration envelope comparable in size in reciprocal space to the integration envelope of the X-ray experiment.

But there is a more important benefit afforded by area detectors! If the resolution of the diffractometer is assumed to be infinitely small the amount of one-phonon TDS included in the scan through a Bragg reflection is directly proportional to the radius of the peak integration volume, the amount of two-phonon TDS to the square of the radius, and the amount of incoherent (flat) background to the cube of the radius. These differences in the dependence on the size of the integration volume can be exploited to correct for TDS and to estimate the elastic constants *empirically*, provided each reflection is sampled in three dimensions, as in scans made with an area detector.

The TDS corrections for X-ray or neutron intensities derived by summation of counts in three dimensions will be discussed in detail. The precision in the empirical method as described above is poor for weak reflections, but, because of the slow variation of TDS with the scattering vector, the corrections for these reflections can be estimated from those of nearby strong reflections. Another advantage offered by area detectors is optimal delineation of peak and background to minimise the estimated error in the background-corrected integrated intensity. For weak reflections this might imply integration within an envelope smaller than the instrumental resolution volume. Exact correction can even be made in this circumstance, within the reasonable assumption that the integration envelope and the resolution volume have the same shape. In a similar fashion allowance can also be made for the TDS contributions from neighbouring reflections.

PS01.06.12 FOUR-CIRCLE DIFFRACTOMETER AND SMALL AREA POSITION SENSITIVE DETECTOR. Meyer, M.; Paciorek, W. A., Chapuis, G. Universit de Lausanne, Institut de cristallographie, BSP, CH-1015 Lausanne (Switzerland).

Single crystal X-ray reflections are intrinsically three-dimensional in nature. Conventional scanning (1-dimensional or w-scanning) with a fairly large, finite aperture obscures the contributions of mosaic spread, spectral dispersion and source divergence by convolution of those entities. High-resolution 1-dimensional position sensitive detectors (PSD) allow an efficient implementation of the so-called $\omega/2\theta$slice scans as standard scanning technique. The fine 2θ resolution of the PSD along with the fine scanning in ω allows partly an experimental deconvolution of the aforementioned contributions. This idea was introduced by Mathieson (1982, *Acta Cryst.* A38, 378-387).

Based on these ideas we implemented a high resolution fibre-optically coupled 2-dimensional CCD-detector on a 4-circle Kappa-geometry diffractometer with an active area of 6.5 x 27mm^2. A 1-dimensional PSD can be simulated with the CCD detector by choosing an appropriate electronic and/or software binning ratio. An arbitrary subwindow of the active area may be defined. The detector resolution and data flow can thus be adapted to the particular problem of interest. The implementation allows thus an arbitrary combination of continuous, step or step-continuous scans with 0-, 1- or 2-dimensional detector windows. The short readout time for 1-dimensional images (60ms/image) allows the execution of $\omega/2\theta$ slice scans as standard integration technique.

Experimental examples will include normal, incommensurate and twinned crystals and the sampling of diffuse scattering. The problem of extraction of integrated intensity from 2- and 3-dimensional slice scans will be addressed in this presentation.

PS01.06.13 bioteX: A SUITE OF PROGRAMS FOR THE COLLECTION, REDUCTION AND INTERPRETATION OF AREA DETECTOR DATA. J. W. Pflugrath, C. L. Day, D. Chen, J. D. Ferrara, P. N. Swepston, J. M. Troup, B. R. Vincent, L. Xiong, Molecular Structure Corporation, The Woodlands TX, R. A. Jacobson, Iowa State University, Ames, Iowa, T. Higashi, Rigaku Corporation, Tokyo, Japan

We have created an OSF/Motif™ based GUI (graphical user interface) for the purpose of collecting, processing and interpreting area detector data. The design goal is to give the novice user the ability to collect and process data effectively while giving the experienced user all the control necessary to complete difficult tasks.

The interface has been designed to be as intuitive as possible. Many steps of data processing have been automated, relieving the user of such tasks. Visualization of all the steps of data processing has been implemented. Where possible, tables of results have been replaced with interactive graphical displays. Online help is provided through NCSA Mosaic and includes hyperlinked text and images.

A new direct-space Fourier transform indexing method has been introduced. Support for narrow-angle oscillations has been provided, while the traditional wide-angle oscillation methods have been retained. Algorithms for recursive, Fourier and spherical harmonic absorption corrections have also been added.

PS01.06.14 STRUCTURE ANALYSIS OF TWINNED CRYSTALS OF $[Ti_{18}O_{28}H](OBu^t)_{17}$. M.R. Pressprich, R.A. Sparks and C. F. Campana, Siemens Energy and Automation, Inc., 6300 Enterprise Lane, Madison WI 53719-1173, USA; Y.W. Chen, Department of Chemistry, University of Illinois, Urbana, Illinois 61801; and V.W. Day, Department of Chemistry, University of Nebraska, Lincoln, Nebraska 68588 USA

A single crystal X-ray diffraction study of the title complex reveals a $Ti_{18}O_{45}$ metal-oxygen framework having a pentacapped Keggin structure[1]. The analysis required a deconvolution of the twin components by a version of the Siemens Autoindexing program[2], which was modified to obtain reflection indices, orientation matrices and lattice parameters for twinned crystals. Once the orientation matrices were obtained, which indicated the twinning relation of a 2-fold rotation about **c***, reflections were grouped into three categories: (1) almost complete overlap of reflections from the two components, (2) partial overlap and (3) no overlap. Reflections from the first and third categories were analyzed with SHELXTL 5.0[3] to yield the full structure.

1. C.F. Campana, Y.W. Chen, W.G. Klemperer and R.A. Sparks J. Chem. Soc. Dalton Trans, 1996, Issue 1, pp xxv-xxx.
2. Siemens Autoindexing Program; Sparks, R. A. in *Crystallographic Computing Techniques*; Ahmed, F. R., ed.; Munksgaard; Copenhage, 1976; pp 452-467.
3. SHELXTL PC Operations Manual, Release 5.0; Siemens Industrial Automation, Inc.; Madison, WI, 1994.