MS01.06.02 PRELIMINARY ANALYSIS OF CCD DATA FOR OXALIC ACID DIHYDRATE AT 100 K. A. A. Pinkerton, Department of Chemistry, University of Toledo, Toledo, Ohio 43606, C. F. Campana and M. R. Pressprich, Siemens Energy and Automation, Inc., Analytical Instrumentation, 6300 Enterprise Lane, Madison, Wisconsin 53719-1173, U.S.A.

We have used a standard Siemens CCD-based SMART diffractometer system equipped with a Mo-target sealed-tube X-ray source (operated at 2000 watts) to collected an 86.3 hour data set on a single-crystal specimen of oxalic acid dihydrate, $C_2H_2O_4{\cdot}2H_2O$, at 100 K. The detector was placed at a distance of 4.984 cm. from the crystal. This data set consisted of three overlapping shells of data to maximum 2θ angle of 142.76° (0.376 Å resolution). A total of 8400 frames of data were collected with 30 second exposure times and 0.3° frame widths in ω.

The frames were integrated with the Siemens SAINT© software package using a narrow-frame integration algorithm. The integration of the data using a primitive monoclinic unit cell yielded a total of 15657 reflections, of which 4675 were independent (redundancy 3.35) and 3858 (82.5%) were greater than $4\sigma(F)$. The final cell constants for a $P2_1/n$ unit cell of dimensions $a = 6.1039(3)$ Å, $b = 3.4988(2)$ Å, $c = 11.9576(5)$ Å, $\beta = 105.785(5)°$, volume = 245.74(6) Å^3, were obtained from the refinement of the XYZ-centroids of 8192 reflections above 20 $\sigma(I)$.

These preliminary data have been analyzed and the structure refined using several commonly used refinement packages, in order to evaluate the suitability of CCD data for charge density studies. These results will be compared with the previously reported analyses[1]. The evaluation of this preliminary data will help us to optimize experimental parameters for more careful measurements to be carried out in the near future.

[1]Coppens et al., *Acta Cryst.*, (1984), A40, 184-195.

MS01.06.03 TWO-DIMENSIONAL HIGH RESOLUTION X-RAY DETECTORS FOR IMAGING AND INTENSITY MEASUREMENTS F. Fandrich and R. Köhler Max-Planck-Arbeitsgruppe „Röntgenbeugung" an der Humboldt-Universität zu Berlin, Hausvogteiplatz 5-7, D-10117 Berlin, Germany

Two CCD slow-scan camera based detector systems with different concepts for x-ray sensitivity provide a high lateral resolution and the capability of single x-ray photon counting. The performance of both systems was studied and compared at a photon energy of 8 keV (copper-K-radiation). While the concept of direct exposure has the better resolution of about 7 µm, the camera using a phosphor and an image intensifier provides a much better quantum efficiency of 80 % compared with about 10 % of the direct system. The response of both systems is high enough in order to detect single x-ray photon 'events' reliably. The image accumulation process is a numerical one, i.e. camera frames with single x-ray events are analyzed immediately after exposure. Accumulation means then counting the number of x-ray photons at those positions where they hit the sensitive area. These positions are calculated from the centres of the x-ray 'events'. The image is build by accumulation of many frames. Topographic imaging or the detection of scattering intensities under different exit angles are possible fields of application. The well-defined intensity measurement based on the counting process during accumulation allows quatitative data evaluation. The performance of the systems is demonstrated by means of x-ray double crystal topographs.

MS01.06.04 COMPARISON OF SINGLE CRYSTAL STRUCTURE DETERMINATIONS WITH AREA AND SINGLE POINT DETECTOR DIFFRACTOMETERS Matthew S. Legge and A. Guy Orpen, School of Chemistry, University of Bristol, Bristol BS8 1TS, U.K.

The time required for data collection for single crystals may be cut drastically by use of an area detector diffractometer. Typically 6-15 hours are needed as opposed to several days with a conventional four-circle diffractometer. This study details the quality of results obtained using each method of intensity data acquisition.

A comparison is presented of X-ray structures whose data collections have been made on both three-circle area detector (Siemens SMART) and four-circle (Siemens P4 & R3m/V) diffractometers. Samples for analysis were chosen so as to represent a range of small molecule crystal types. Thus, a strongly diffracting organometallic compound $[Ru_2Cp_2(\mu\text{-}CO)(CO)\{\mu\text{-}(MeO_2C)CC(CO_2Me)CCH(CO_2Me)\}]$[1], a strongly absorbing crystal $[W_2Cp_2(C_8Me_7CH_2)][BPh_4]$[2] and a typical organic molecule $[C_{27}H_{30}O_2]$[3] were used for the comparisons. In common with other studies involving comparisons of data sets[4,5], the quality of each structure analyis was evaluated by examining the crystal and intensity data, refinement parameters and also the data collection conditions. Data will also be analysed using a variety of statistical techniques (including normal and half-normal probability plots[6]) on intensity data, coordinate and displacement parameters and uncertainty estimates.

[1]P. King, S.A.R. Knox, A. Martin, A.G. Orpen, unpublished work.
[2]N.G. Connelly, M.S. Legge, B. Metz, unpublished work.
[3]R.W. Alder, A. Martin, A.G. Orpen, unpublished work.
[4]F. Iwasaki *et al. Acta Crystallogr., Sect.B*, 1995, **51**, 1028.
[5]Pohl, E.; Herbst-Irmer R. *et al, Helv. Chim. Acta,* 1995, **78**, 355.
[6]S.C. Abrahams *et al* Acta Crystallor., Sect. B, **34**, 2981.

MS01.06.05 COMPARISON OF 5 HIGH RESOLUTION X-RAY DATA SETS OF DL-ASPARTIC ACID MEASURED AT 20 K. Dieter Zobel[1], Ralf Flaig[1], Peter Luger[1], Hans-Georg Krane[2], [1]Inst. f. Krist., FU Berlin, Takustr. 6, D- 14195 Berlin, [2]Mineral. Inst. d. Uni. Würzburg, Am Hubland, D-97074 Wurzburg

Based on Bader's formalism /1/ topological properties of selected amino acids are being investigated. To obtain charge density information very accurate high resolution data sets at $\sin\theta/\lambda \geq 1.0$ Å^{-1} have to be collected at the lowest possible temperature. For DL-aspartic acid (*C2/c*, V=1057.4 Å^3), our first example in that course, we measured a total of five data sets, each one at 20 K, to get an impression on optimum experimental conditions:

1) Mo-X-ray tube ($\sin\theta/\lambda \leq 1.07$Å^{-1}), Nb-ß-filter, szintil. counter.
2) Mo-X-ray tube ($\sin\theta/\lambda \leq 1.07$Å^{-1}), Si(Li)-solid state detector.
3) Ag-X-ray tube ($\sin\theta/\lambda \leq 1.37$Å^{-1}), Pd-ß-filter, szintil. counter.
4) Ag-X-ray tube ($\sin\theta/\lambda \leq 1.37$Å^{-1}), Si(Li)-solid state detector.
5) Synchrotron radiation ($\sin\theta/\lambda \leq 1.01$Å^{-1}), monochr. szintil. counter.

The use of the solid state detector (measurement 2 and 4) with an energy resolution of better than 200 eV avoids the attenuation of α-radiation by conventional monochromators or filters and results in a peak-to-background improvement. Critical comparison of the measurements represented by $I/\sigma(I)$ will be discussed. In all cases our 20 K He closed-cycle refrigerator /2/ was used for cooling the sample.

Full order refinements resulted in R=0.028 for 7047 unique reflexions of measurement 4. Further refinements on the basis of multipole expansion (program XD /3/) reached R=0.020 giving the experimental basis for charge density calculations and related properties.

[1] Bader, R. C. W., "Atoms in Molecules"Clarendon Press,Oxford,1990
[2] Zobel,D., Luger,P. & Dreißig,W. 1992. Acta Cryst. B 48.835-848.
[3] XD: "A Program for refinement and analysis of charge density", T. Koritsanszky et al.(1995), FU Berlin.