# LABNOTE IPDS 2T

## CHEMICAL CRYSTALLOGRAPHY APPLICATIONS WITH THE STOE IPDS 2T DIFFRACTOMETER EQUIPPED WITH A XENOCS GENIX CU $K_\alpha$ MICROBEAM DELIVERY SYSTEM



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## CHARACTERISTICS

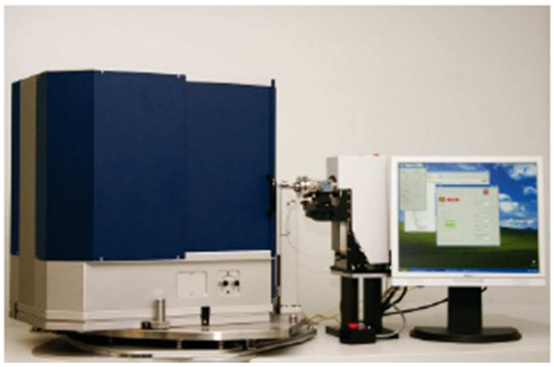

***Fig.1:*** *IPDS2T-GeniX setup at the EPFL*

Although Mo $K_\alpha$ radiation is widely used in routine small molecule structure determination, a number of applications in chemical crystallography are best treated with Cu $K_\alpha$ radiation. These include studies on weakly diffracting and/or very small crystals, measurements on partially disordered or fibrous samples, absolute structure determination and others.

To evaluate the suitability of the STOE image-plate system IPDS2T equipped with a GeniX Cu $K_\alpha$ microbeam system manufactured by XENOCS for applications in chemical crystallography, we have carried out trial measurements on two samples that are commonly used for testing purposes.

### 1. Data collection on YLID ($C_{11}H_{10}O_2S$)

YLID is a small-molecule standard widely used in the single-crystal diffraction. It belongs to the orthorhombic system so that the refinement of the cell angles provides a good check of the alignment, geometric accuracy and general performances of a laboratory diffractometer setup.

The GeniX microbeam system was operated at 50 kV, 1 mA. The circular image plate (scanned surface: 170 mm radius) was positioned at a distance of 40 mm from the sample with a 60° 2θ offset, thus allowing data to be collected to minimum Bragg spacings of 0.83 Å. A highly redundant data set, consisting of three 0–180° omega-scans recorded at three different phi angles (0°, 45° and 90°) was collected. The data comprise a total of 540 frames, each corresponding to a 1° omega rotation and an exposure of 60 seconds. The frames were indexed and integrated with the X-Area software from STOE. Data reduction (scaling and merging) was carried out with the program XRed from STOE. For comparison, reference data on the same sample were also collected on a sealed tube Mo $K_\alpha$ source in our laboratory, using an IPDS 2 diffractometer from STOE. Structure refinement was carried out with SHELXL.

Refined cell parameters are reported in Table 1 for both measurements, along with available reference data (G.L.Bryant[1]). Data reduction and structure refinement statistics are reported in Table 2. The data collected on the STOE IPDS 2T / Xenocs–GeniX system are slightly better than the literature values that were obtained from one of the best reputed Cu $K_\alpha$ instruments on the market (Bryant[1]). Most certainly, they would be even better had we been given a better mounted and shaped test-crystal. The comparison of the two measurements ascertains the superior intensity of the Xenocs/GeniX source. For roughly the same $R\sigma$ value the exposure time with Mo radiation (53kV, 43mA) had to be five times bigger than that with the microbeam system.

[1] *http://www.avtechlabs.com/ylid2/ylid.htm*

## 2. Data Collection on Zeolite LTA $\{Na_{12}[Al_{12}Si_{12}O_{48}] \cdot 27\ H_2O\}_8$

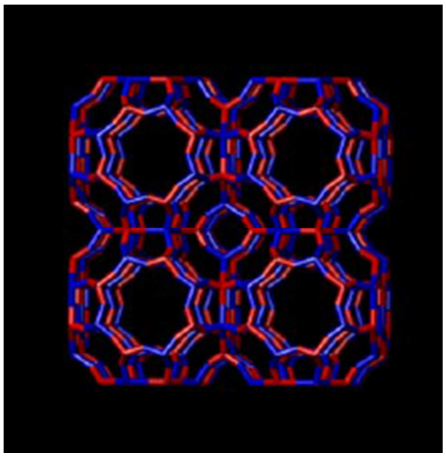

***Fig.2:*** *The framework structure of Zeolite A with Si-O and Al-O bonds colored respectively in blue and red*

Zeolites are alumino-silicate minerals. Their porous molecular structures have made them very attractive materials for a wide range of industrial applications including molecular sieves, catalysts and detergents.

Zeolite A exhibits the LTA (Linde Type A) structure. The *topological* unit cell of symmetry is doubled by the chemical partition between Al and Si, giving rise to a *supercell* with unit-cell parameters of 24.572 Å. The doubling of the unit-cell generates very weak superlattice reflections.

The STOE image plate system *IPDS 2T*, equipped with a *GeniX* microbeam system from *XENOCS* was used to collect diffraction data on a small crystal of zeolite A.

The GeniX microbeam system was operated at 50kV, 1mA and an exposure time of 90 seconds per 1° omega-rotation allowed to record data with good statistics *(Fig. 3 )*. The image plate was positioned at a distance of 70 mm from the sample and was offset in 2θ by 45°. Three omega scans were collected at different phi angles producing highly redundant data. Data collection parameters and data reduction statistics are reported in Tables 3 and 4. In a reciprocal lattice reconstruction from the experimental data *(Fig. 4)*, the superlattice reflections are clearly visible, thus substantiating the quality of the data. The framework structure *(Fig. 2)* could be solved using SHELXS with default parameters.

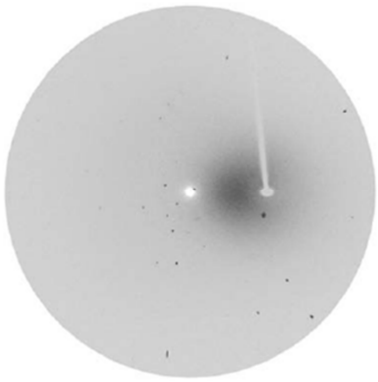

***Fig.3:*** *Diffraction frame recorded on a crystal of Zeolite A. Scan width 1° (omega), exposure time 90 sec. Crystal-detector distance 70 mm, 2θ offset 45°*

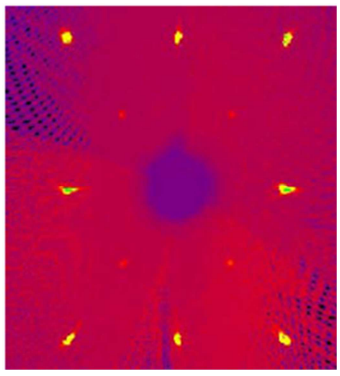

***Fig.4:*** *Reciprocal lattice reconstruction of the (hk0) layer from experimental data. The weak superlattice reflections are clearly visible.*

**CONCLUSION:**

**Accurate data were collected on samples from organic molecules and complicated inorganic structures with the combined GeniX – IPDS 2T setup installed at EPFL-Lausanne.**

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| Data | | a [Å] | b [Å] | c [Å] | α [Å] | β [Å] | γ [Å] |
|---|---|---|---|---|---|---|---|
| IPDS 2T + GeniX Cu $K_\alpha$ | nc | 5.9603(5) | 9.0354(5) | 18.3859(12) | 90.036(5) | 90.033(6) | 90.036(6) |
| | c | 5.9677(4) | 9.0336(5) | 18.3883(11) | | | |
| IPDS 2 + sealed tube Mo $K_\alpha$ | nc | 5.9606(5) | 9.0337(7) | 18.3818(15) | 90.014(7) | 90.010(6) | 90.027(6) |
| | c | 5.9606(3) | 9.0338(5) | 18.3796(15) | | | |
| Reference (Bryant) Cu $K_\alpha$ | c | 5.96260(10) | 9.03940(10) | 18.3890(2) | | | |

**Table 1:** Cell parameter refinement of YLID. nc: non constrained refinement, c: constrained refinement (i.e. cell angles are fixed at 90°)

| Data | $d_{min}$ / Å | $R_{int}$ | $R_\sigma$ | SAV | IEq | $R_1$ | Flack par. |
|---|---|---|---|---|---|---|---|
| IPDS2T + GeniX Cu $K_\alpha$ | 0.8354 | 0.0629 | 0.0362 | 0 | 291 | 0.0292 | -0.019(21) |
| | 0.9020 | 0.0627 | 0.0353 | 0 | 285 | 0.0240 | -0.011(18) |
| IPDS2 + sealed tube Mo $K_\alpha$ | 0.5717 | 0.0255 | 0.0242 | 0 | 3 | 0.0363 | -0.005(61) |
| Reference (Bryant et al.) Cu $K_\alpha$ | 0.9020 | 0.0644 | 0.0343 | 13 | 218 | 0.0277 | -0.022(19) |

**Table 2:** Data reduction and structure refinement statistics for Ylid. SAV: Systematic absence violations, IEq: Inconsistent Equivalents

| | |
|---|---|
| Refined unit cell parameter: a / Å | 24.5719 (0.0009) |
| Space group | F *m* -3 *c* |
| Number of measured reflections | 16249 |
| Number of unique reflections | 881 |
| Rint | 0.0575 |
| Absorption correction transmission factors | Face-indexed 0.41 0.51 |
| Rint after absorption correction | 0.0545 |

**Table 3:** Data collection and reduction statistics for Zeolite A

| *dmin / Å* | 14.19 | 2.60 | 1.90 | 1.62 | 1.42 | 1.31 | 1.21 | 1.13 | 1.06 | 0.99 | 0.93 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| *Mean I/sd(I)* | 28.12 | 12.48 | 16.43 | 18.12 | 15.40 | 15.01 | 7.11 | 5.34 | 5.76 | 3.25 | |
| $R_{int}$ | 28.12 | 0.076 | 0.068 | 0.054 | 0.043 | 0.040 | 0.050 | 0.054 | 0.072 | 0.152 | |
| *Completeness / %* | 98 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | |

**Table 4:** Data quality indicators for Zeolite A.