Single Crystal Diffractometer Development

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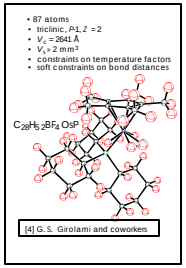
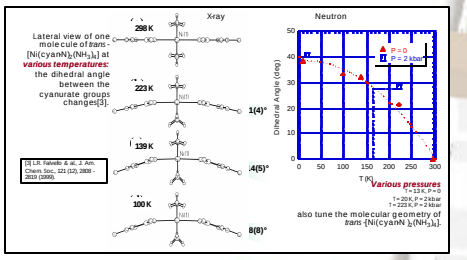
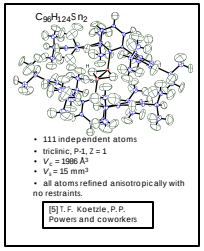
Single Crystal Examples:

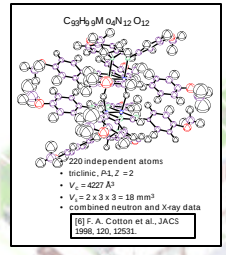
Currently single crystal measurements with various sample enviro nments are routinely collected at the IPNS-SCD. To study temperature dependence of materials high temperature furnaces and low to very low temperature cooling is being used. High pressure can be applied to tune structures accordingly and obtain information on material behavior with pressure changes. As an example, changes in crystal structure with varying conditions for *trans*-$[Ni(cyan\text{-}N)_2(NH_3)_4]$ is shown[3].

Refining and solving large molecular structures from neutron diffraction data has been flux limited. Current examples of relatively large molecular structures refined on data measured at the IPNS-SCD are shown below [4],[5],[6].









Refine ***Large molecular structures*** with V_c=2000-4000Å³ and ~ 200 independent atoms

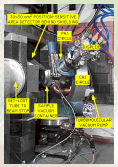
TOF Single Crystal Diffraction:

A single-crystal diffractometer employs area detectors and the time structure of pulsed-neutron sources to measure Laue diffraction patterns. The efficiency of sampling the reciprocal lattice simultaneously in both direction (area detection) and length (time resolution) of the probe wavevector is a powerful advantage of pulsed-source diffractometers [1]. The current generation of instruments can accumulate sufficient data at 10-50 sample orientations over a period of several days to determine structures of moderate complexity. By utilizing the enhanced flux of the SNS, increasing the detector coverage, and employing focusing optics, we can reduce this data collection time to a matter of minutes[2]. More importantly, the vastly increased flux-on-sample will make possible measurements of smaller samples and more complicated structures.

[1] A.J. Shultz, Trans. AM. Cryst. Assoc. 29,29 (1993)
[2] J.F. Ankner & JK. Zhao, ES-1.1.8.4- 6018- RE-A -00 (1999).

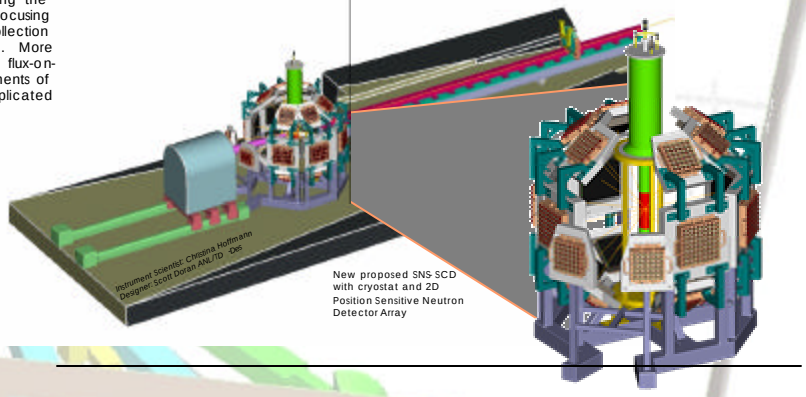
Comparison SNS/IPNS:

Parameter	SNS	IPNS	Ratio
Target neutron production	4.04×10^{15} n/pulse	5.16×10^{13} n/pulse	78
Source frequency	60 Hz	30 Hz	2
Flux Reduction	$1/(12.0\text{m})^2$	$1/(9.5\text{m})^2$	0.63
Sample settings	2	17	8.5

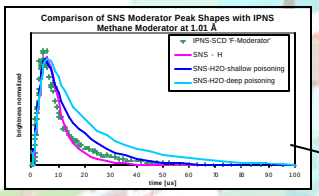


These terms combine to yield about a factor of 100 improvement in flux-on-sample at SNS over IPNS.

To estimate counting times, apply this factor to IPNS SCD data. In a recent measurement, a full data set was collected from an organic crystal (*b*-ARAF), of unit cell volume V_c = 767Å³, using 17 sample orientations over a total counting time of 136 hrs (5.6 days). Using the SNS-SCD the same data would be collected in approximately 15 min.



New proposed SNS SCD with cryostat and 2D Position Sensitive Neutron Detector Array



Design Criteria:

- SNS-SCD is a general-purpose single-crystal diffractometer, with the capability of high speed measurements and resolving larger structures.
- Views a decoupled poisoned hydrogen moderator and, with an overall neutron flight length L_{MD} = 13 m, exhibits a lower limit on d_{min} = 0.63 Å.
- Utilizes a doubly tapered guide to provide a maximum incident-beam divergence $d\phi/d$ fwhm for $\lambda \geq 1.5$ Å. Absorbing apertures will be available to reduce this divergence as needed. The final 1-2 m of this guide can be made removable to allow for the insertion of focusing optics or ^{3}He polarizers for magnetic studies.
- Has area-detector (panels) coverage exceeding 50%, on a sphere of radius 1 m, with an opening at the top for the insertion of magnets, cryostats, etc. All detectors require spatial resolution. The detector radius was chosen to limit the effect of magnetic fields on scintillator PMT tubes. Depending on detector R&D developments, this radius could well change.
- The large sample-detector distance ensures that scintillation-detector photomultiplier tubes are not adversely affected by applied magnetic fields at the sample position.
- The sample goniometer has ϕ and χ motions to enable complete coverage of reciprocal space and access for magnets, furnaces, and cryostats.
- A (Fermi) chopper can be inserted in the incident beam to aid in the elimination of thermal-diffuse scattering.
- Typical data collection time will be less than an hour for a sample of moderate complexity.

SNS Instrument Systems Argonne National Laboratory Oak Ridge National Laboratory

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