Time Dependent SANS Sample Environment Capabilities at NIST

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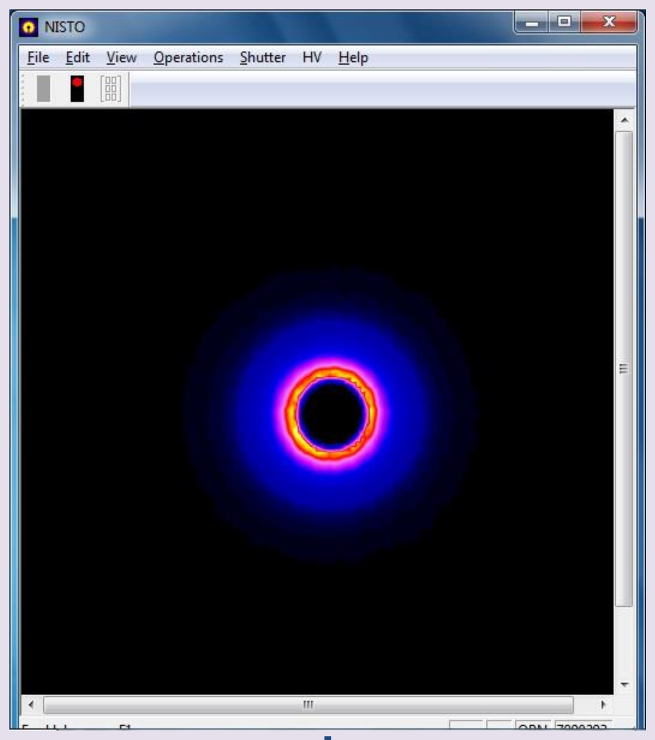
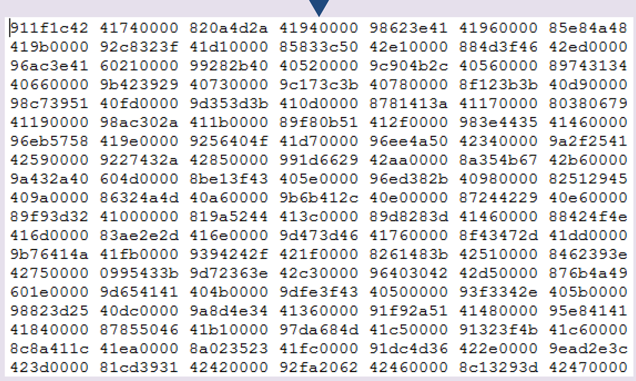
Generating Time Dependent Data

The NISTO software package, used to collect data from Ordela* detectors at NIST, can collect data as a raw data stream from the detector. The raw data are 4-byte (32 bit) neutron event words encoded with the pixel coordinates of the event, a time stamp and event meta data.

Time-dependent neutron events are sent as two consecutive event words, each with a 13 bit time stamp for a total of 26 bits of precision. The time stamp is the amount of time relative to a counter reset, in 10^{-7} seconds, giving a maximum cycle time of 6.71 seconds. To reset the counter to 0, a 5V pulse is sent to the detector, starting a new cycle. If the counter overflows (rollover event) before a reset is given, a specific bit on the next event word is set to 1. Neutron events are processed in a FIFO manner, so all event words are in chronological order.





Data Handling

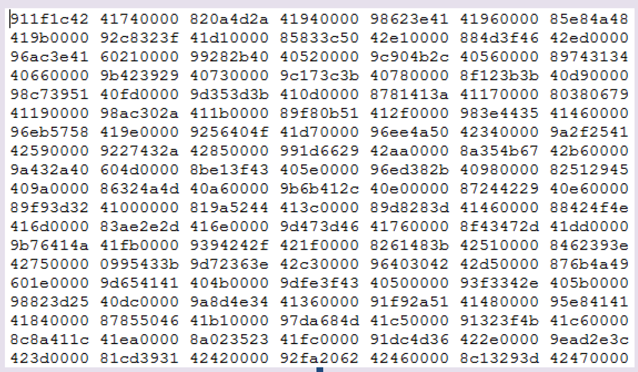
The SANS Igor Reduction software package[1] includes a panel for processing the list of event words, as of v7.2.

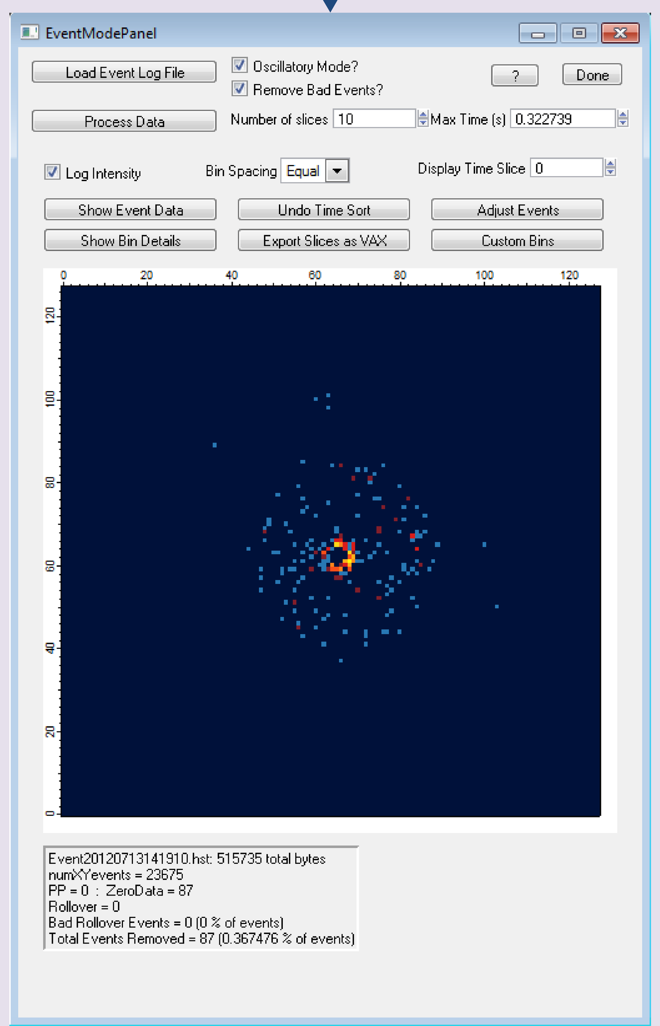
The panel allows the user to take all data between two time points, relative to the cycle start time, and create a separate 2D SANS pattern from it. This 2D pattern is called a slice or bin. The number of bins and the start time and end time for bin are all independently variable. Two bin spacing types are built in, equal time and Fibonacci time scales, but any custom spacing is possible.

The 6.71 seconds cycle time limit is overcome by multiplying the number of rollover events between each consecutive time reset by the maximum cycle time and then adding that time to the event word time.

Occasionally, event words are not in a FIFO manner, or a rollover event is not properly credited. The software can remove these 'bad' events if the user wishes.

For large data sets, loading the data can be time consuming.





Highlights:

- Separate data into any number of bins of any amount of time for each bin
- No effective cycle time limit
- Visualization of event number as a function of time to see any 'bad' events
- Ability to remove these bad events

1. **Reduction and Analysis of SANS and USANS Data Using Igor Pro** S. Kline 2006 *J. Appl. Cryst.* **39**, 895

Sample Environment and Equipment

Anton Paar MCRXX1 Series Rheometer[1]

Overview

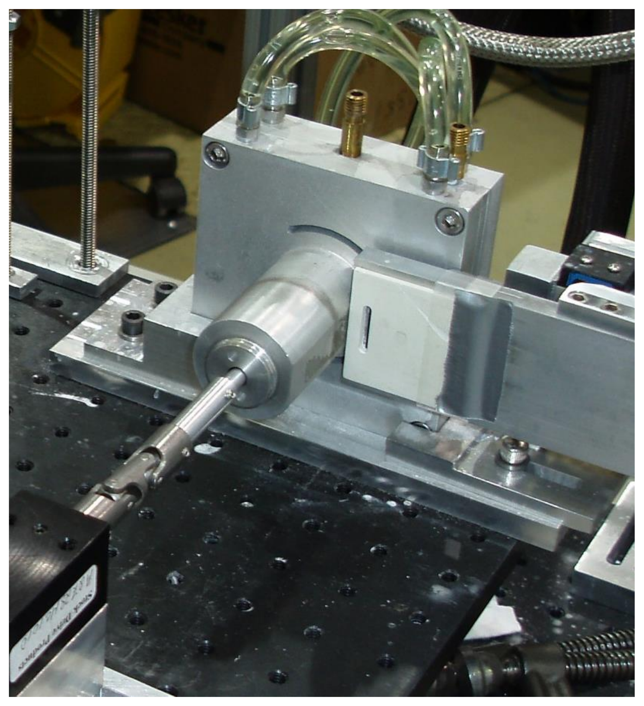
- Primary Use: SANS of samples under stress in the 1,3 and 2,3 planes
- Modes of Operation: Continuous, Oscillatory, Start-Stop/Startup/Stop
- Torque Regime: 0.1 μNm to 230 mNm
- Available Models: MCR 501 (not available for USANS), MCR 301

Time Dependent Capabilities

- RheoSANS as a function of time for any mode of operation

How It's Done

- At the end of each cycle/oscillation, the rheometer sends a voltage pulse to the detector
- SANS instrument and rheometer coordinate via a series of on/off TTL signals (handshaking)



1,2-plane Shear Cell[2,3]

Overview

- Primary Use: SANS of samples under shear in the 1,2 plane
- Modes of Operation: Continuous, Oscillatory
- No torque feedback – shearing cell only

Time Dependent Capabilities

- Shaft position as a function of time for any mode of operation

How It's Done

- Processes are similar to rheometer control

Rotating Cell Holder[4]

Overview

- Primary Use: Prevent sedimentation
- Modes of Operation: Continuous
- Modular Design - Each holder independent
- Center of neutron beam is the center of sample rotation
- Designed/Developed at Uppsala University for use at NIST

Time Dependent Capabilities

- Remove time dependence of sedimentation by tuning rotation speed to settling time

How It's Done

- Direct control of the motor that rotates the holders via SANS control software
- Motor drives a belt attached to the outer part of the holder

TISANE (Stroboscopic) Chopper[5]

Overview

- Dual choppers spinning in opposite direction

Time Dependent Capabilities

- Sub-millisecond time resolution SANS measurements

How It's Done

- Chopper frequencies cause neutrons to hit the detector in pulses with a characteristic time between pulses

1. **Rheo–small-angle neutron scattering at the National Institute of Standards and Technology Center for Neutron Research** L. Porcar *et al* 2011 *Rev. Sci. Instrum.* **82**, 083902
2. **Spatially resolved small-angle neutron scattering in the 1-2 plane: A study of shear-induced phase-separating wormlike micelles** M.W. Liberatore *et al* 2006 *Phys. Rev. E* **73**, 020504
3. **Measuring material microstructure under flow using 1-2 plane flow-small angle neutron scattering** A.K. Gurnon *et al* 2014 *J. Vis. Exp.* **84**, e51068
4. **A holder to rotate sample cells to avoid sedimentation in small-angle neutron scattering and ultra small-angle neutron scattering experiments,** Anders Olsson *et al* 2013 *Meas. Sci. Technol.* **24,** 105901
5. **Stroboscopic Small Angle Neutron Scattering Investigations of Microsecond Dynamics in Magnetic Nanomaterials** A. Wiedenmann *et al* 2010 *Springer Series in Solid-State Sci.* **161,** 241