

Time Dependent SANS Sample Environment Capabilities at NIST

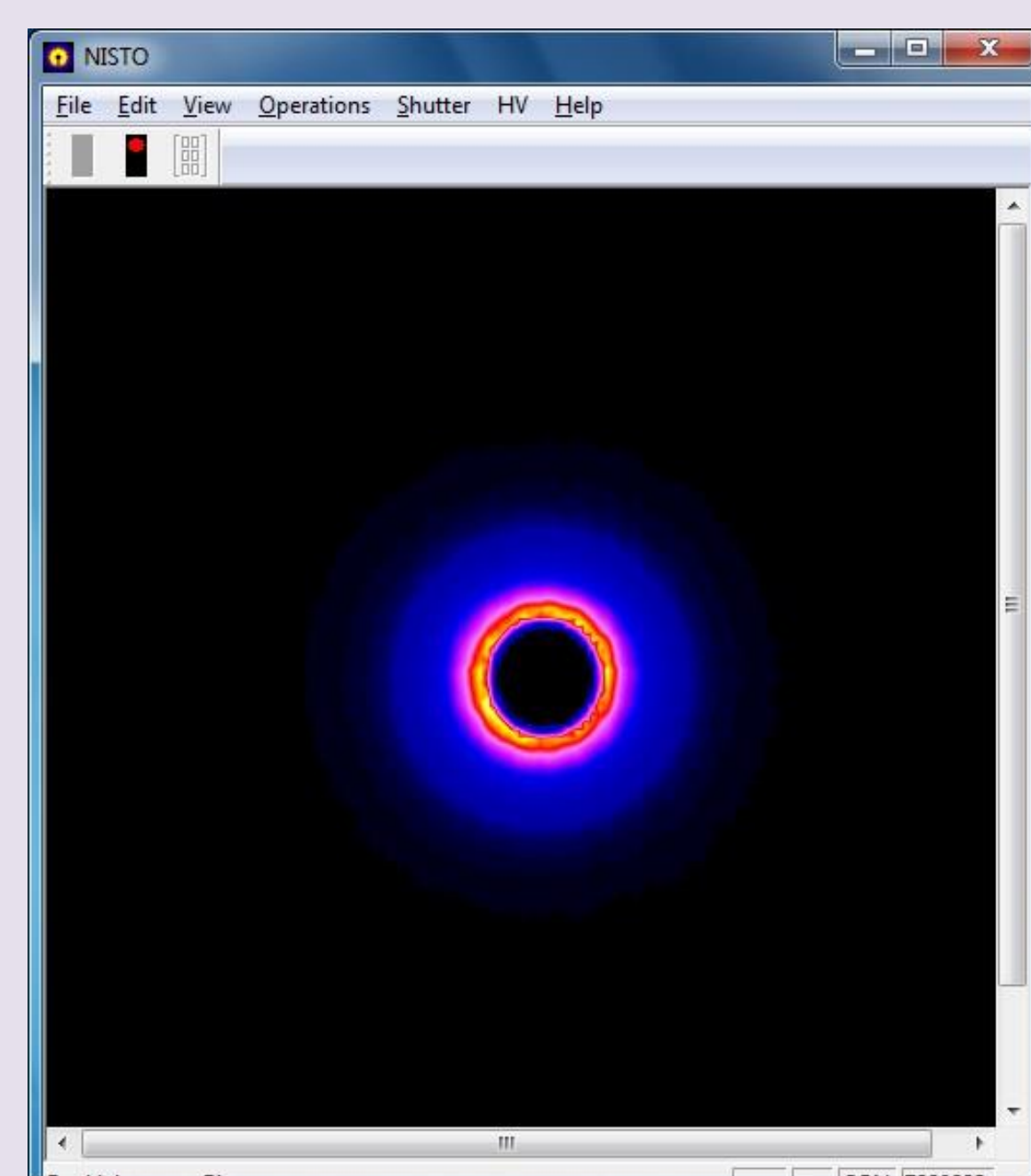
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Generating Time Dependent Data

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.



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b11f1c42 41740000 820a4d2a 41940000 98623e41 41960000 85e84e48
419b0000 92c8323f 41d10000 85833c50 42e10000 88483f46 42e40000
96ac3e41 60210000 99282b40 40520000 9c904b2c 40560000 89743134
40560000 9a423929 40730000 9e173c3b 40780000 8e123b3b 40490000
98c73951 40fd0000 94353d3b 41040000 8781413a 41170000 80380e79
41190000 98ac302a 411b0000 89f80b51 412f0000 983e4435 41460000
96eb5758 419e0000 9256404f 41d70000 96e64a50 42340000 9a222541
42590000 9227432a 42850000 991d6629 42aa0000 8a354b67 42b60000
9a432a40 60440000 8be13f43 405e0000 96ed382b 40980000 82512945
409a0000 86324e4d 40a60000 9b6b412c 40e00000 87244229 40e60000
89f93d32 41000000 819a5244 413c0000 8982823d 41460000 88424f4e
416d0000 83ae2e2d 416e0000 94f73d46 41760000 843472d 41d80000
9b76414a 412b0000 9394242f 421f0000 8261483b 42510000 8462393e
42750000 0955433b 9d72363e 42c30000 96403042 42d50000 876b4e49
601e0000 94654141 404b0000 9dfc3f43 40500000 93e3342e 405b0000
98232d25 404e0000 9a8d4e34 41360000 91f92a51 41490000 95e84141
41840000 87855046 41b10000 97da684d 41c50000 91323f4b 41c60000
8c8a411c 41ea0000 8a023523 41fc0000 91dc4d36 422e0000 9ea22e3c
423d0000 81cd3931 42420000 92fa2062 42460000 8c13293d 42470000
    
```

Data Handling

The SANS Igor Reduction software package¹ 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.

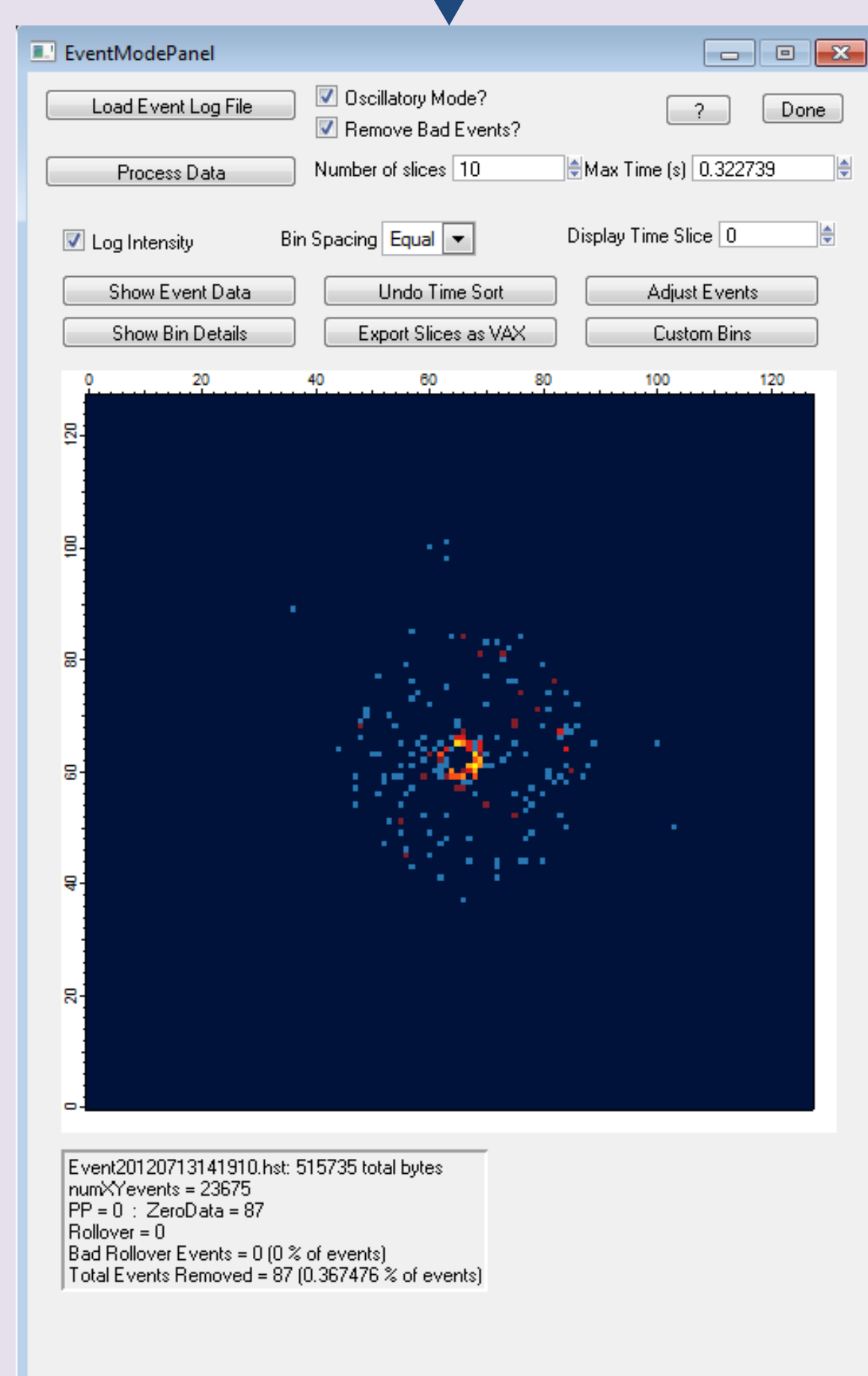
For large data sets, loading the data can be time consuming.

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

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b11f1c42 41740000 820a4d2a 41940000 98623e41 41960000 85e84e48
419b0000 92c8323f 41d10000 85833c50 42e10000 88483f46 42e40000
96ac3e41 60210000 99282b40 40520000 9c904b2c 40560000 89743134
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42590000 9227432a 42850000 991d6629 42aa0000 8a354b67 42b60000
9a432a40 60440000 8be13f43 405e0000 96ed382b 40980000 82512945
409a0000 86324e4d 40a60000 9b6b412c 40e00000 87244229 40e60000
89f93d32 41000000 819a5244 413c0000 8982823d 41460000 88424f4e
416d0000 83ae2e2d 416e0000 94f73d46 41760000 843472d 41d80000
9b76414a 412b0000 9394242f 421f0000 8261483b 42510000 8462393e
42750000 0955433b 9d72363e 42c30000 96403042 42d50000 876b4e49
601e0000 94654141 404b0000 9dfc3f43 40500000 93e3342e 405b0000
98232d25 404e0000 9a8d4e34 41360000 91f92a51 41490000 95e84141
41840000 87855046 41b10000 97da684d 41c50000 91323f4b 41c60000
8c8a411c 41ea0000 8a023523 41fc0000 91dc4d36 422e0000 9ea22e3c
423d0000 81cd3931 42420000 92fa2062 42460000 8c13293d 42470000
    
```



```

Event: 2071 3141 910 In: 515735 total bytes
Number of Events = 23075
PP = 0 : ZeroData = 87
Rollover = 0
Bad Rollover Events = 0 (0.0% of events)
Total Events Removed = 87 (0.367476% of events)
    
```

Sample Environment and Equipment

Anton Paar MCRXX1 Series Rheometer¹

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 μ Nm 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

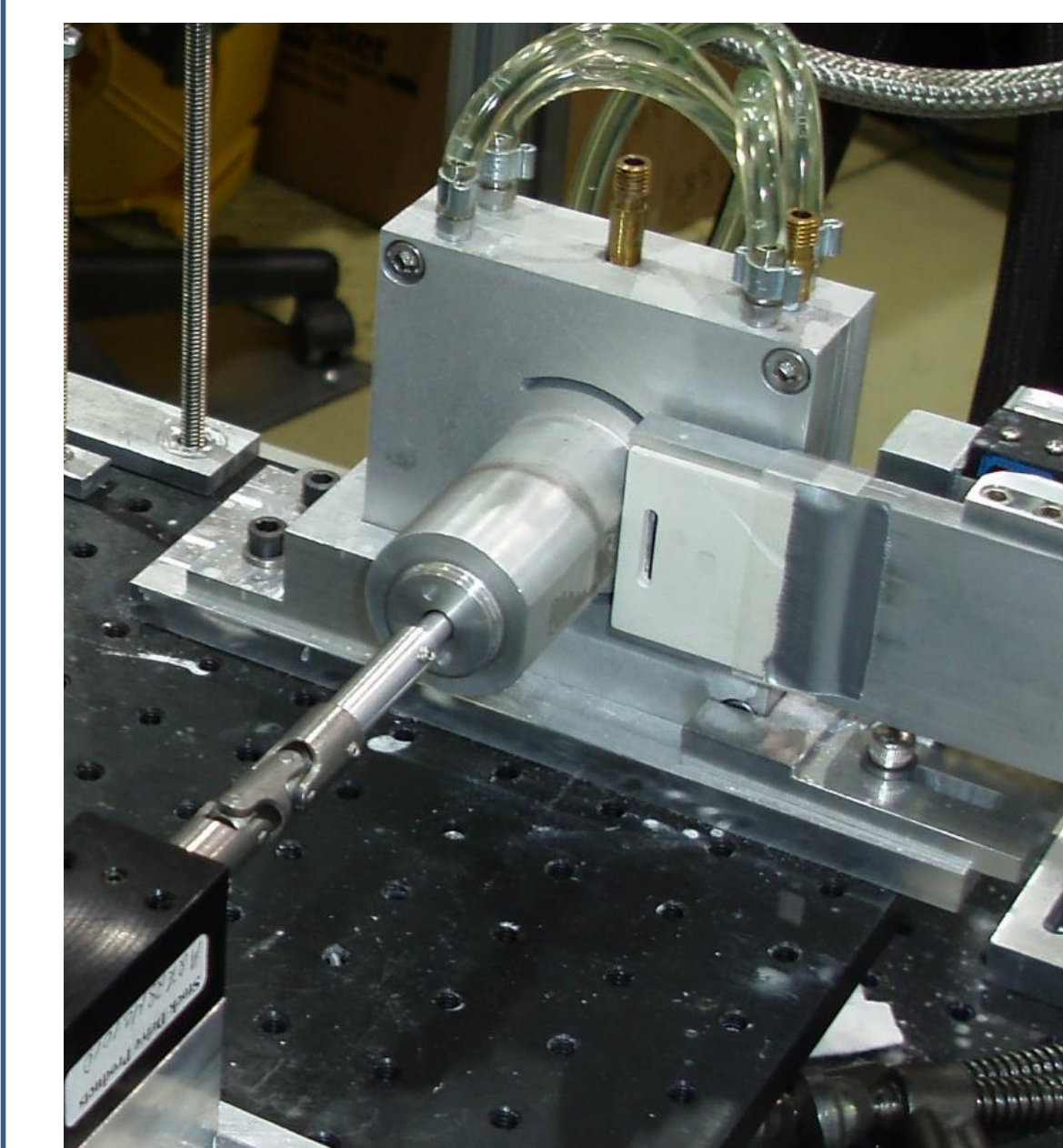
- Primary Use: SANS of samples under shear in the 1,2 plane
- Modes of Operation: Continuous, Oscillatory
- No torque feedback – shearing cell only

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 Holder⁴

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) Chopper⁵

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



- 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
- 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
- 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
- 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
- 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