The Bells and Whistles: a workflow to get excellent experiments out of (lab) X-ray scattering instruments

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1. User discussions

- match user expectations with capabilities,
- adapt samples for scattering (if possible)

2.1 Project description

- Summarizes project & expectations
- Contains all relevant sample phase information (density, composition)
- Machine readable (Excel)-document

2.2 Sample design

- [custom] samples mounted in appropriate holders:
- continually pumped flow-through cell with external reservoir for liquid samples and their backgrounds

3. Experiment design / e-logbookkeeping

- Set up instrument configurations, sample locations, measurement conditions in electronic logbook (another machine-readable excel sheet)

4.logbook to measurement scripting

- Automatic conversion of logbook to script, makes exact logbookkeeping an integral part of the workflow
- All required supplementary measurements are automatically included: e.g. beam profile, -center & -flux, sample transmission,
- Automatic repetitions of every measurement are carried out to help identify temporal changes in the sample or instrument.
- Full automation configuration changes means no "convenience"-tradeoffs in measurement procedure required

5. Measure & Collect datafiles

This is the easy part of a scattering experiment: sit back and wait! Multiple data and metadata are collected from various sources

6. Translate datafiles to NeXus

Tree-like, hierarchical structure structures every possible bit of information from the measurement

7. Correct for everything

Using a complete data correction procedure, turn the collected raw data into a most accurate scattering cross-section complete with (multiple) uncertainty estimates. See [2].

8. Data quality check

- Evaluation on whether the data is of sufficient quality for analysis
- Check for excessive uncertainty estimates, negative average intensities, low transmission or high probability of multiple scattering

9. Interpret data

- Cross-check with users to ensure only the minimum of necessary assumptions are made.
- Exploit absolute units as cross- / sanity-check
- Use uncertainty estimates to weigh by the true value of the collected data and avoid overfitting

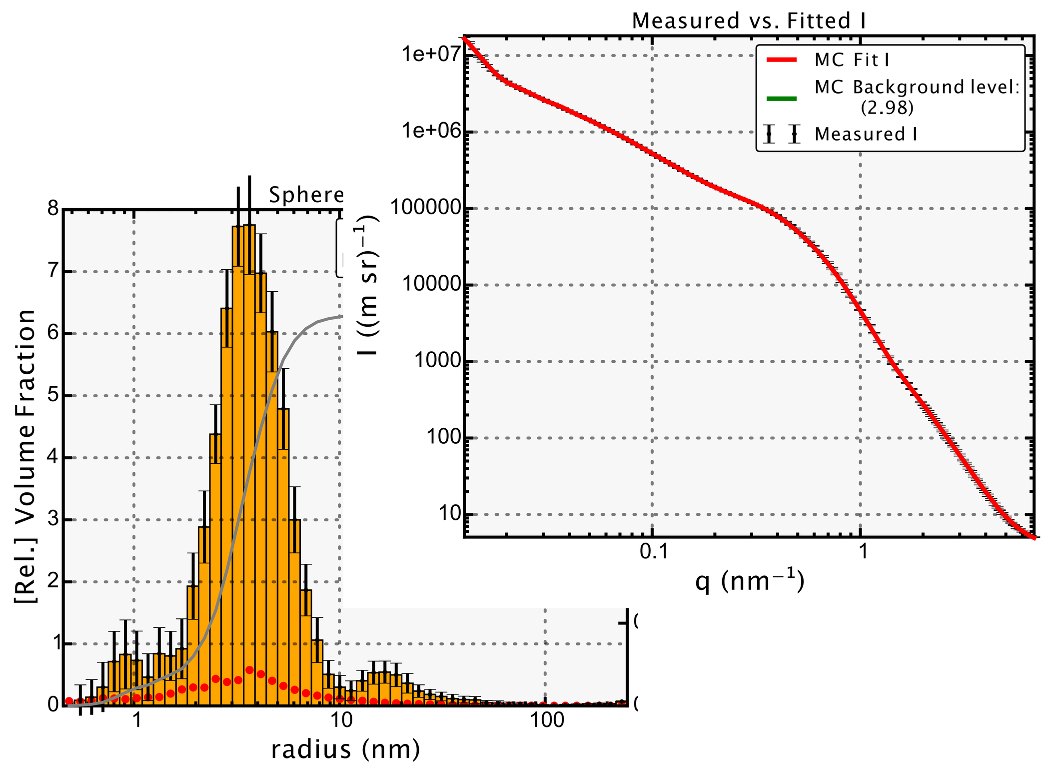
10. Interpretation quality check

- Evaluation on whether the interpretation is of sufficient quality, and whether all possible information has been extracted. Check observability limits and cross-check with information from symbiotic techniques (e.g. TEM, BET, APT, physics)

"What is an excellent experiment?"

In an "excellent scattering experiment", you have arrived at all possible conclusions determinable from the experiment, with as much confidence as warranted by the quality of your workflow steps..

for scattering experiments, this requires an extra level of rigor and care, due to the low information content and lack of obvious data quality indicators.

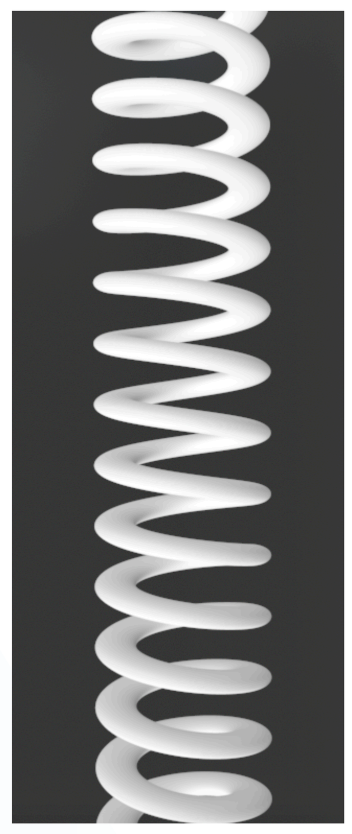


Analyze using appropriate models

- Models should match reality

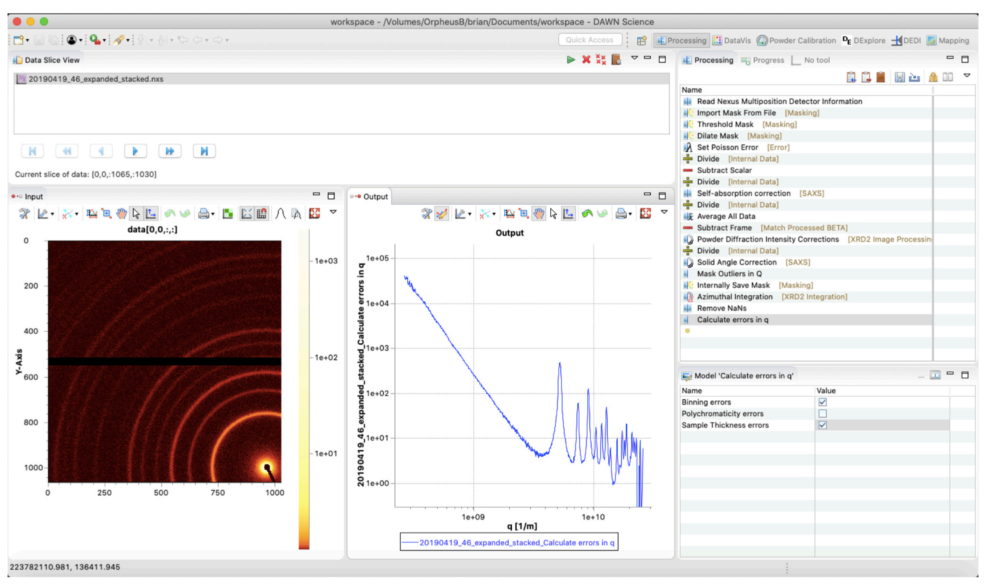
Simulate scattering

- Check your fitting model by having it analyze a scattering pattern from a known structure similar to that of your sample



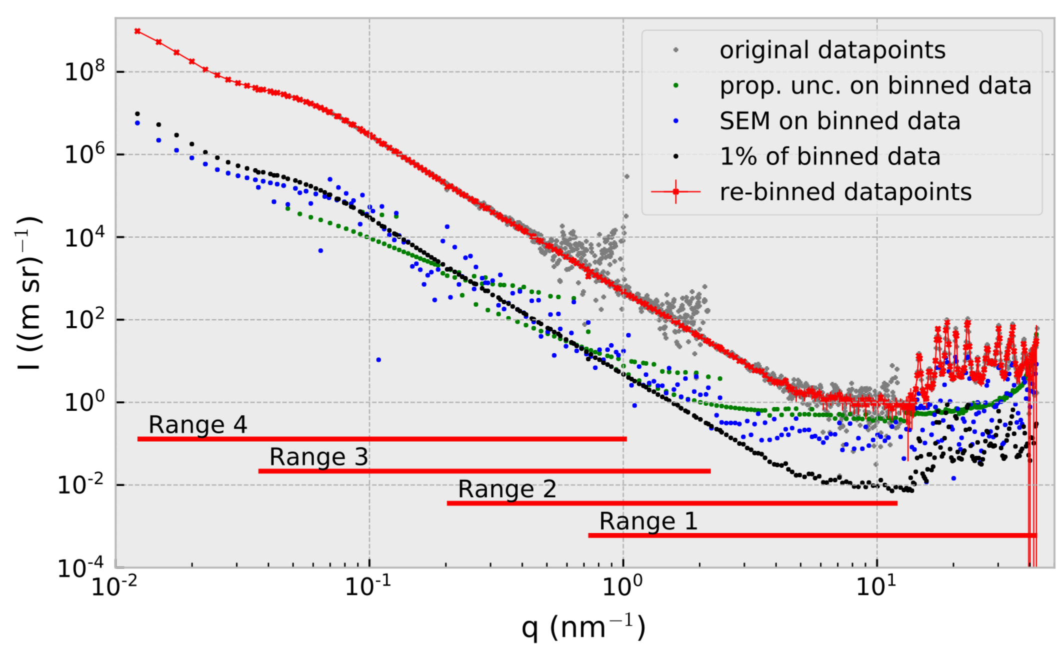
Store in SciCat [3]

MongoDB-based database stores raw and derived measurements alongside cross-linked project and sample descriptions



Process in DAWN [5]

The open-source Data Analysis Workbench contains a comprehensive pipeline for accurate correction of data



Merge data if appropriate

- Merging datasets from different ranges weighted by their uncertainty, can only be done if not significantly affected by beam smearing

Further reading

[1] B.R. Pauw. (2013) Everything SAXS: small-angle scattering pattern collection and correction. J. Phys.: Condens. Matter 25: 383201. DOI: 10.1088/0953-8984/25/38/383201

[2] B. R. Pauw, A. J. Smith, T. Snow, N. J. Terrill, A. F. Thünemann, (2017) The modular SAXS data correction sequence for solids and dispersions, Journal of Applied Crystallography, 50: 1800–1811, DOI: 10.1107/S1600576717015096

[3] https://scicatproject.github.io/

[4] G.J. Smales, B. R. Pauw, (2020) The science behind *the Instrument*: a comprehensive methodology for obtaining traceable, wide-range scattering data from a (lab) X-ray machine, *submitted*

[5] Filik J. et al. (2017) Processing two-dimensional X-ray diffraction and small-angle scattering data in DAWN, Journal of Applied Crystallography, 50, DOI: 10.1107/S1600576717004708