Correction of Tangential Couette SANS data

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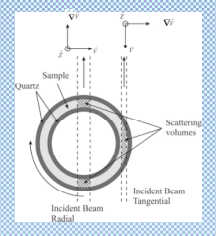
ABSTRACT:

In SANS Couette shear cell measurements conversion to an absolute scale for the radial scattering configuration can be done in the standard manner using the direct beam as a primary standard and then correcting for sample and cell transmissions and the sample thickness (simply twice the Couette gap). Absolute calibration in the tangential geometry is more problematic – the geometrical limitations of asymmetries of sample path and absorption lead to clear asymmetries in the observed scattering patterns and difficulties in calibration.

We recently reported a simple and effective way to normalize tangential scattering: from the tangential transmission we obtain an effective thickness consistent with the radial transmission and its known thickness[1]. However, this does not address the asymmetries often observed in our simply corrected tangential configuration scattering patterns. This geometry dependent asymmetry is due to neutrons scattered toward the cell axis traversing a significantly longer path through the sample fluid than neutrons scattered away from it. As would be expected this effect is more pronounced for high sample absorption, scattering angle and sample annulus width.

To correct for this effect we propose to use a "sensitivity file" obtained from a mixture of heavy and light water matching the sample absorption in the same tangential geometry scattering configuration and thus subjecting evenly scattered incoherent neutrons to a similar absorption (re-scattering) geometry to that experienced in the sample. Furthermore, it is also possible to create a "calculated sensitivity" based on a sample's absorption and geometrical considerations which allow correction of the scattering asymmetry observed in the tangential configuration to a very good approximation. We will discuss the limits of validity of such approaches.

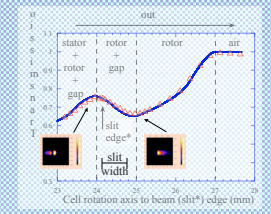
Couette Shear Cell measurements

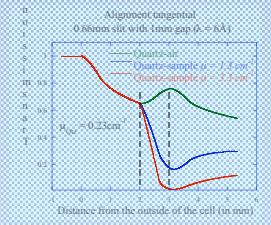


For Couette cell scattering there are two main scattering configurations. In the radial geometry, the neutron beam passes through the sample along the velocity gradient direction, across the diameter of the cylinders, while in the tangential geometry, the beam is along the flow direction, tangent to the sample annulus. These configurations allow us to measure orientation relative to (Z,V) and (Z,VV) planes respectively, where Z denotes the rotation axis of the cylinders.

Tangential Slit alignment

Tangential measurements require a rectangular slit of width smaller than that of the gap in order to minimize reflections from the curved interfaces. By translating the slit across the tangential position an accurate alignment may be achieved by noting the sharp transmission variation. These measurements can be performed with the cell either empty or containing any solution. Below, we show the transmission variation as the beam defining slit is translated across the tangential position with the cell empty and loaded with different samples of absorption _ where the transmission T is written as T=exp-_t.
In practice we usually opt for an empty cell as the edges are more clearly defined.

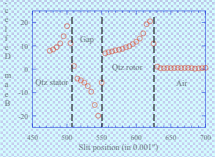




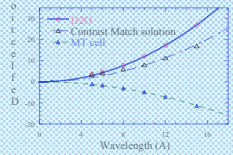
Measuring the transmission variation as the beam defining slit is translated across the tangential position with the cell empty for the 1.00 mm gap, the sharp changes in the curve correspond to interception of material boundaries by the neutron beam. The continuous line represents the calculated average transmission across the beam. The final slit edge position used for the Couette cell SANS measurements in this case is indicated by the arrow. Also represented above are the direct beams reflecting from the quartz stator and rotor surfaces respectively.

Direct beam deflection

Another important issue in tangential configuration is the direct beam deflection which alters the position of the beam center. This effect is especially strong for long sample to detector distances and long wavelengths.



Here the strong effect of the exact slit position relative to the shear cell is shown for 12_ neutrons with the detector at 15m. As the tangential slit is translated across the cell. The beam center deflection changes sign as the beam hits the quartz, gap, and quartz again.

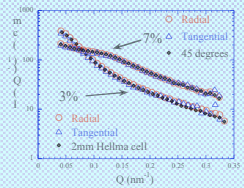


Fixing the slit position relative to the gap reveals the effect of wavelength on the beam center deflection for an empty cell, a D2O, and a D2O/H2O mixture contrast matched to the quartz. The lines represent fits from a simple prism formula and clearly show the expected λ^2 dependence.

Absolute scale for tangential configuration [1]

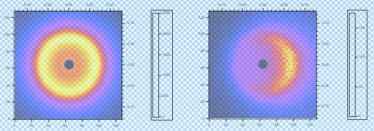
Placing tangential data on an absolute scale, where the sample thickness is extremely sensitive to alignment, is problematic, and indeed is usually ignored. In the rare instances where it is attempted, it has been done by initially determining a scale factor between absolutely scaled radial scattering data from isotropic scattering from the sample and a corresponding tangential measurement. We have suggested an alternative approach, valid for any off-radial position using the radial transmission to determine the absorption coefficient μ of the solution from $T_{rad}=\exp-\mu t$ where the known sample thickness $t=2g$ is twice the Couette gap, so $\mu=-\ln(T_{rad})/2g$. The experimentally determined tangential transmission can then be used to determine the effective thickness of the sample in this configuration: $t_{eff} = -\ln(T_{tg})/\mu = 2g \cdot \ln(T_{tg})/\ln(T_{rad})$.

Here, the scattered intensity I(Q) in cm⁻¹ for a 3% and 7% sweetened sponge phase (isotropic membrane phase) determined for different configurations at zero shear is reproduced from ref 1. For the 3% solution, Couette cell measurements in radial and tangential configuration are compared to conventional 2 mm Hellma cell SANS measurements, while for the 7% solution Couette cell measurements in radial, tangential, and 45° configurations are compared. The non-radial Couette configurations were absolutely normalized by the effective thickness method and all data are in superb agreement.



Absorption problems

Asymmetry can be visible in the tangential configuration scattering patterns because neutrons scattered toward the cell axis will traverse a longer path through the sample fluid than those scattered away. This effect is more pronounced for high sample absorption, scattering angle and sample annulus width. This is shown below for an isotropic solution measured in the radial (left) and tangential (right) configurations.



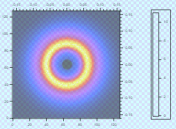
Absorption correction

The simplest approach to the problem is to measure a solvent in the same tangential geometry with a similar absorption coefficient, _, as the sample solution and to then use this data as a "sensitivity" correction (an "absorption sensitivity file") to the sample data. Unfortunately this can often lead to prohibitive accumulation times as the solvent will have to be measured to quite good statistics in a geometry which throws away most of the neutrons. Furthermore, samples differing significantly in tra nsmission will require different "absorption sensitivity" runs. An alternative method is to mathematically create the absorption sensitivity file from geometric considerations and the sample absorption.

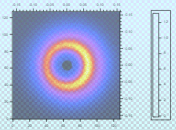
As we show below, these first order corrections work remarkably well, particularly in the case of low absorption samples though they are somewhat less satisfactory for high absorption, high incoherent scattering samples. As these latter make clear, while the further work would be useful to offer even more generally applicable and easily accessible correction methods.

1- Simple case: *Isotropic solution with no incoherent scattering and high transmission*

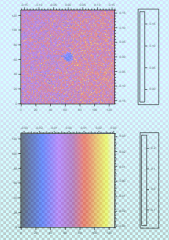
Radial



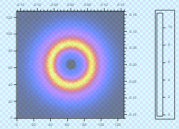
Tangential



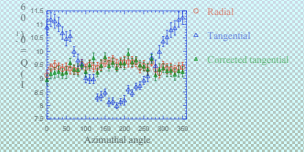
Scattering from a 5% CPCl/D2O micellar solution in radial and tangential geometry



Tangential scattering from a D2O solution and an "absorption sensitivity file" calculated from geometrical considerations

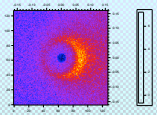


Absorption corrected tangential data

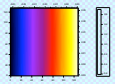


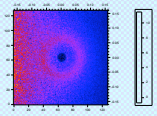
2- More complex cases: *Isotropic solution with incoherent scattering and low transmission*

Tangential

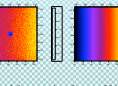


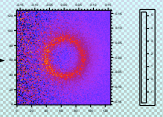
Scattering from a 5% CPCl in 50/50 H2O/D2O in tangential geometry





I_{tan} / Absorp





$I_{tan} - I_{incoh}$ / Absorp

Tangential scattering from a 50/50 H2O/D2O solution and an "absorption sensitivity file" calculated from geometrical considerations

Conclusions:

Two dimensional Couette cell data taken in the tangential configuration can be placed on an absolute scale after some extra corrections allowing for more quantitative interpretation of such data. However, the long path length involved can make these corrections somewhat difficult and time consuming, particularly for samples with high incoherent scattering contributions.

Future directions:
1) Monte Carlo method to estimate the effect of multiple incoherent scattering in the tangential geometry.
2) Numerical method to account for multiple scattering effects.

REFERENCE

1. L. Porcar, W. A. Hamilton, P.D. Butler, and G.G. Warr, *A new vapor barrier couette shear cell, for small angle neutron scattering measurements*, Review of Scientific Instruments **73**, 2345 (2002)