Esoteric Background Correction for SANS Measurements

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Talk Overview

Background signal: Any signal collected by detector that is **not** produced
By Small-angle scattering (SAS) from ***'desired'*** sample structure.

1) Background Sources Review
    - Internal to sample
    - External to sample
2) Annie Brulet's Paper Review: large scattering angle (θ) corrections
    - Sample Transmission
    - Cell / Window Transmission
    - Detector Efficiency
    - Detector Gondola Effect
3) SAS Tests with Vanadium Single Crystal

Sources of Background Internal to Sample

1) Liquid Samples

a) Quasi-elastic incoherent background

[?nearly? Isotropic/flat scattering, strong in hydrogenous materials.]

b) Inelastic Incoherent or Coherent Background

[**Not** truly isotropic, but may appear to be at small θ …]

c) Elastic Coherent Wide-Angle Scattering

[Most important for D_2O and amorphous silica/quartz,

- Diffuse scattering peak produced by nearest atom correlations.
- Compressibility creates I(0) > 0 which may appear nearly isotropic/flat at small angles.]

d) Enhanced Incoherent Bgd from D/H isotopic mixtures

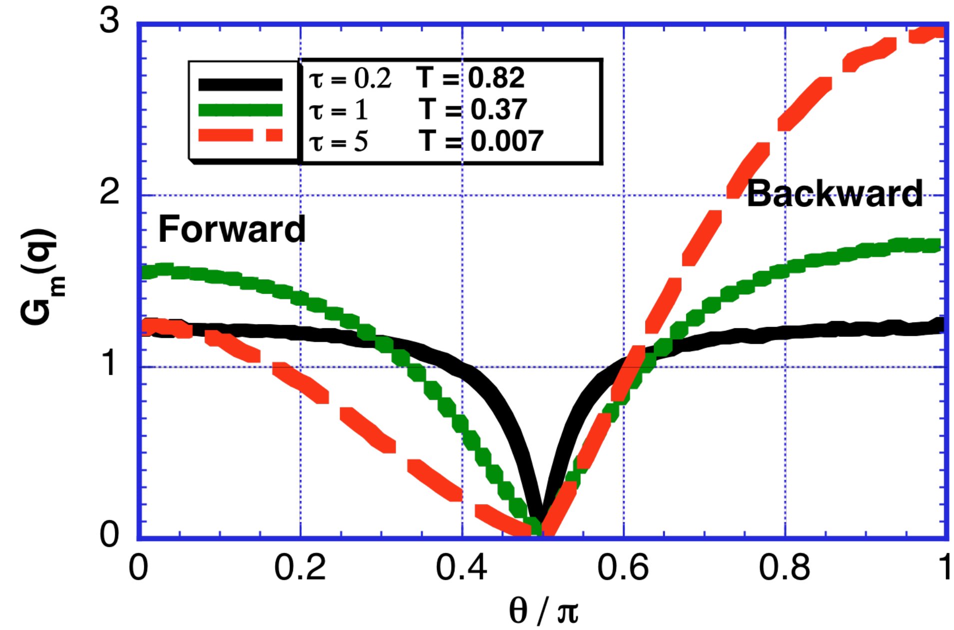
e) Multiple Scattering Distortions

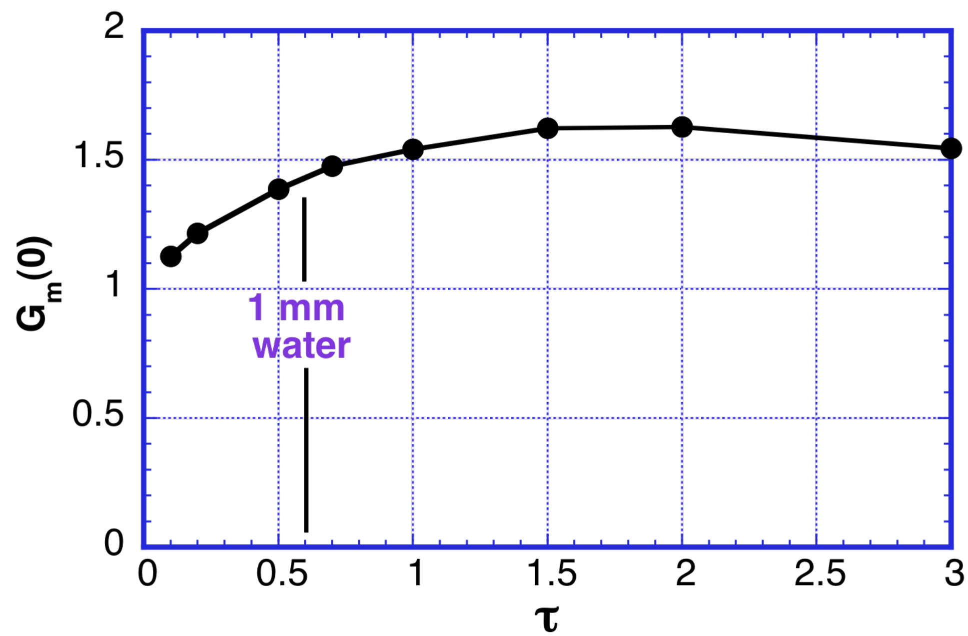
[where $T_{scat} < 0.9$, Shape + amplitude altered → sample geometry]

Simulation of **Multiple Scattering** From Isotropic Scattering Disks

Scattering is pushed away from 90˚

Scattering is thus enhanced at 0˚.





Sources of Background **Internal** to Sample

1) **Crystalline Samples**
   a) Quasi-elastic incoherent background
   [?nearly? Isotropic/flat scattering, strong in hydrogenous materials.]
   b) Inelastic Incoherent or Coherent Background
   [**Not** truly isotropic, but may appear to be at small θ …]
   c) Double Bragg Scattering
      - Polycrystal: $I_{DBS} \sim q^{-m}$ $2 < m < 3$ Taglauer, E. (1968)
      - Single Crystal: can produce streaks or spots.

   [eliminate by using $\lambda > \lambda_{cutoff}$, reduce by improving crystalline perfection.
   d) Grain Boundaries, twins or dislocations
      - Very weak signal from lower density dislocation core.
      - Strong signal from chemical segregation
      - Strong signal in ferromagnets via demagnetization.

   [Pure materials annealed to increase grain size or lower dislocation density.]
   e) Surface Scratches: $I \sim q^{-m}$ $2 < m < 3$ Roth, M. (1977).
   [Very weak for typical polished surface…]
   f) Ferromagnetic Domain Walls - Very Strong multiple Scattering !!!
   [Remove walls in saturating magnetic field…]

Sources of Background External to Sample

1) **Windows and Sample Cells**
   - **Includes All internal source types**
   - **SAS from internal structure (… precipitates …)**

   [Care must be taken in evaluating possible shadowing from shielding.
   … position in multiple sample changer …]

2) **Air scattering**
   - **Appears nearly isotropic at small angles.**
   - **Severely affected by shadowing from shielding.**
   - **Large geometric solid angle gain produced near detector…**

   [Vacuum P < 0.1 Torr, also sensitive to changes in P]

3) **Collimation Scattering (Parasitic Halo)**

   a) Aperture edge scattering (SAS + Refraction) $I \sim q^{-3}$

   [Use Gd foil and polish edge]

   b) Aperture Diffraction $I \sim q^{-3}$

Sources of Background **External** to Sample

4) **Fast Neutron Background**
   - **weak source (< 0.2 s^{-1}) from instrument guide extremely stable**
   - **External sources may change! { NG-6 Physic's shutter…~10 s^{-1} }**

5) **External Thermal neutrons**

[Proper Cd shielding of detector chamber eliminates source.]

6) **2D Detector Dome Scattering : I ~ q^{-2}**

   [0.5 % of neutrons scatter ~ isotropically via phonons]

7) **Gamma-Rays [Current detectors have very low sensitivity…]**

8) **Reactor-off Background: (Stable ~ 1 s^{-1})**

   a) Cosmic Rays

   b) Activity in aluminum of detector

   c) Detector electronics "false" counts

Annie Brulet's Paper Review: large scattering angle (θ) corrections

Annie Brulet et al, *J. Appl. Cryst.* (2007). **40**, 165-177.

Important large angle corrections in Brulet's paper:

1)Flat detector solid angle correction ~ $\cos^3(\theta)$ [C. Glinka, 1980's]

2)Sample transmission correction [Steve Henderson, 1990's]

3)Detector Efficiency Correction [Lindner, 2000 → Kline, 2008]

4)Cell + Window transmission correction

[Brulet, 2007 … not presently incorporated at NIST ….

5)"Gondola Detector Correction

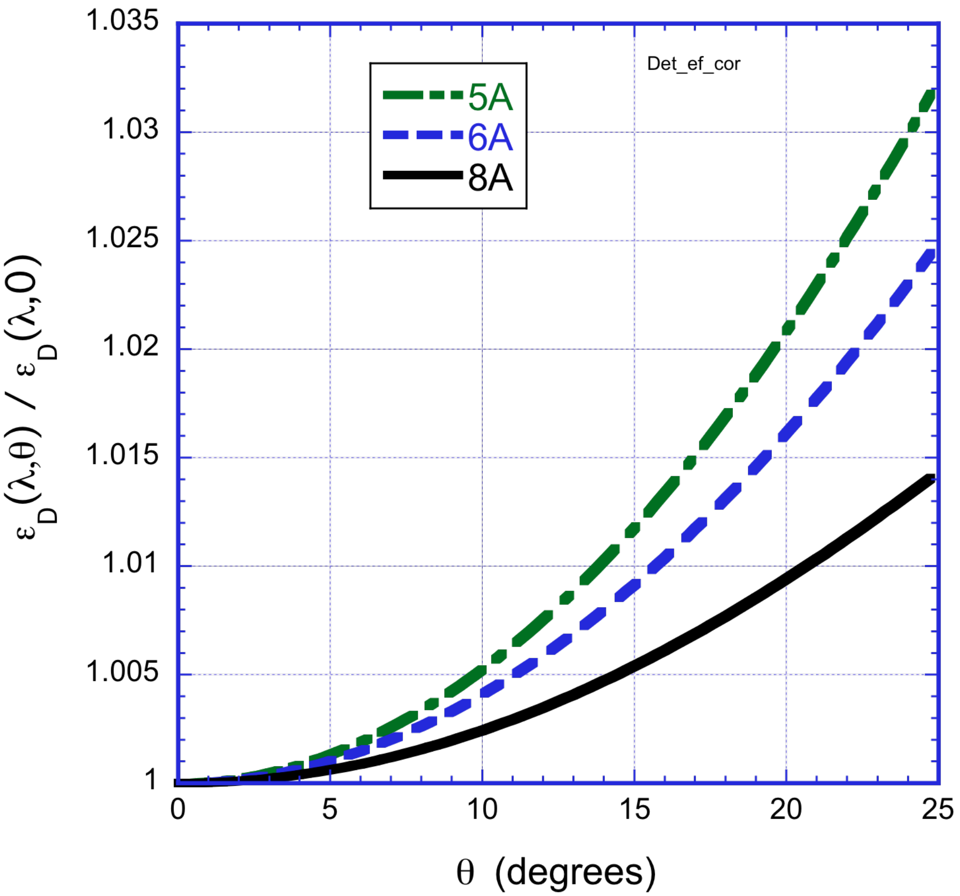
[Deformed counter window causes nonuniformity of ^{3}He gas depth]

- Produces –dependent detector sentivity
- Sensitivity varies radially from detector center

Current Ordela 2660N detectors seem to be **okay**….constant depth

Detector Efficiency Correction:

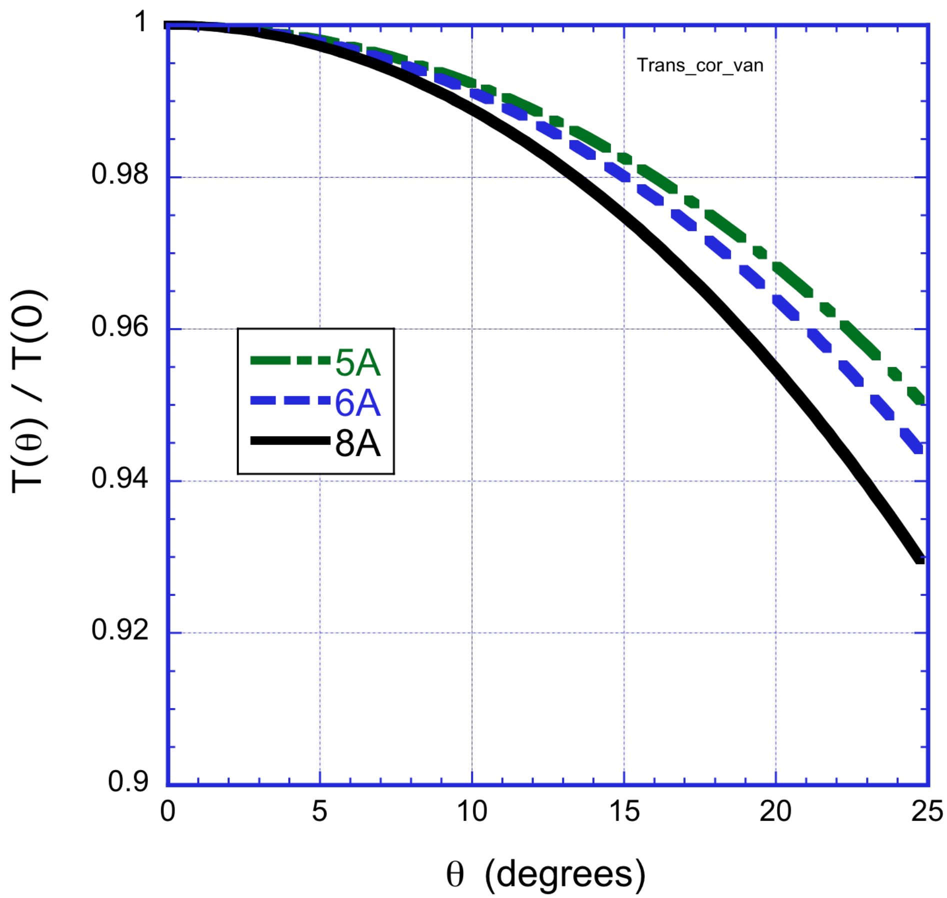
$$\varepsilon_D(\lambda,\theta) = 1 - \exp\left[\frac{-\mu(\lambda)t}{\cos(\theta)}\right]$$



Angle-dependent
Transmission Correction
Brulet's eq. 9

$$T(\theta) = T\,\frac{1 - T^{a(\theta)}}{-a(\theta)\ln(T)}$$

$$a(\theta) = \frac{1}{\cos(\theta)} - 1$$



Cell + Window transmission correction (Brulet)

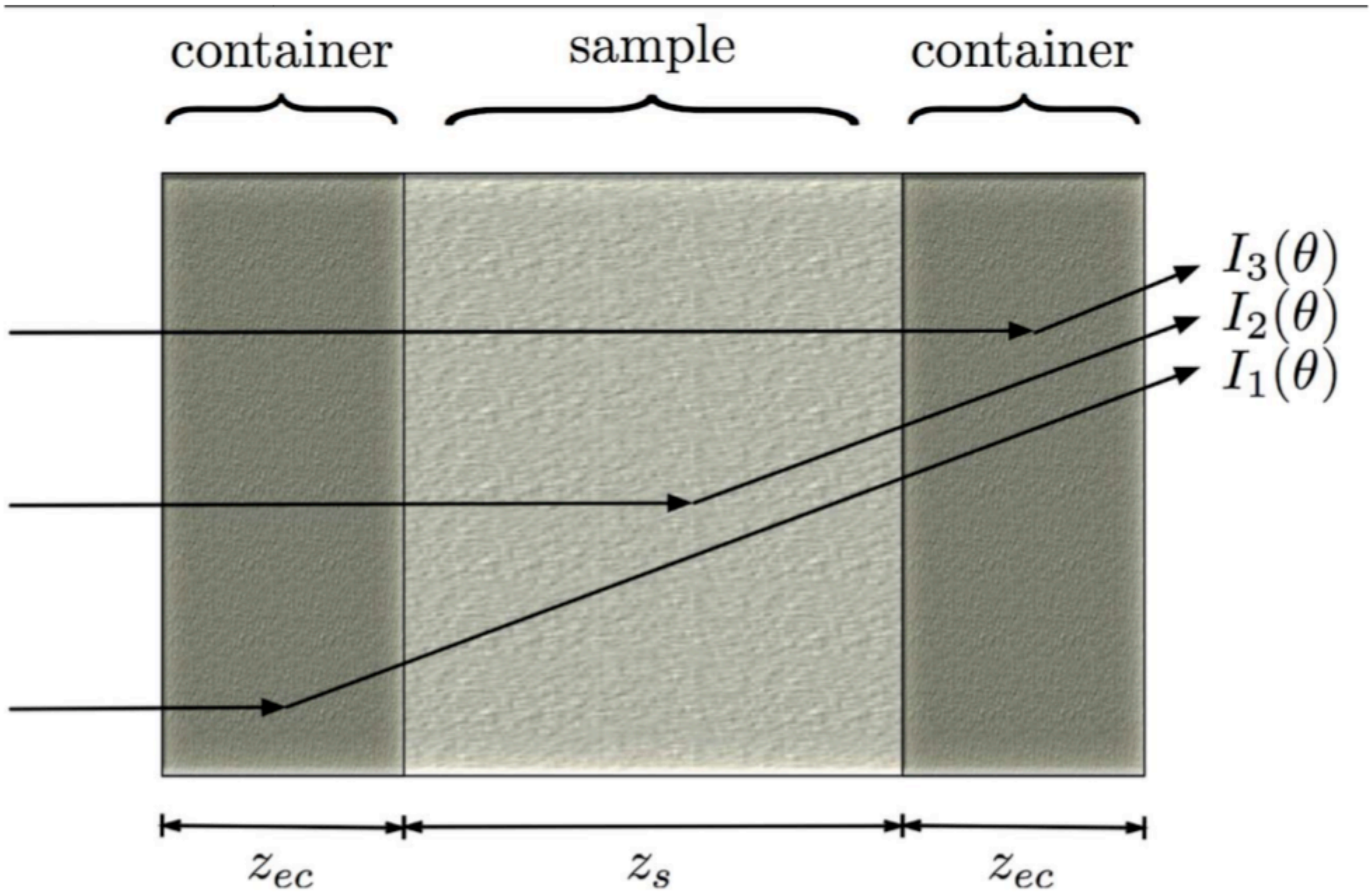


Figure 8

Schematic representation of the scattering by a sample of thickness z_s in a container with front and back windows of thicknesses z_{ec1} and z_{ec2}, respectively.

Brulet's Data Corrections:

- **Detector efficiency**
- **Sample + Cell Transmission**

$$F_s(\theta) = \frac{I_S(\theta) - B}{z_s T_S \alpha_S(\theta)} - \gamma_S(\theta)\left[\frac{I_{EC}(\theta) - B}{\beta_{EC}(\theta) T_{EC}}\right] + \gamma_S(\theta)\left[\frac{T_{EC}^{a(\theta)}}{\beta_{EC}(\theta)} - \frac{T_S^{a(\theta)}}{\beta_S(\theta)}\right] F_b(\theta). \quad (12)$$

The dimensionless quantities $\alpha_S(\theta)$ and $\beta_S(\theta)$ tend to 1 for $\theta \to 0$ and/or $T \to 1$. They are defined by

$$\alpha_S(\theta) = T_{EC}^{a(\theta)/2} \frac{-(T_S/T_{EC})^{a(\theta)}}{-a(\theta)\ln(T_S/T_{EC})} = \mathcal{E}_2[a(\theta)\ln(T_{EC})] \times \mathcal{E}_1[a(\theta)\ln(T_S/T_{EC})] \quad (13)$$

and

$$\beta_S(\theta) = \left[1 + \left(\frac{T_S}{T_{EC}^{1/2}}\right)^{a(\theta)}\right] \times \frac{1 - T_{EC}^{a(\theta)/2}}{-a(\theta)\ln(T_{EC})} = \mathcal{E}_3[a(\theta)\ln(T_S/T_{EC}^{1/2})]\,\mathcal{E}_4[a(\theta)\ln(T_{EC})], \quad (14)$$

with $\mathcal{E}_2(x) = 1 + x/2 + x^2/8 + x^3/48 + x^4/384 + \ldots$, $\mathcal{E}_3(x) = 1 + x/2 + x^2/4 + x^3/12 + x^4/48 + \ldots$ and $\mathcal{E}_4(x) = 1 + x/4 + x^2/24 + x^3/192 + x^4/1920 + \ldots$. The quantity $\gamma_S(\theta)$ has the dimension of a reverse thickness. It is defined as

$$\gamma_S(\theta) = \frac{1}{z_s}\frac{\beta_S(\theta)}{\alpha_S(\theta)}. \quad (15)$$

Vanadium Single Crystal as a Detector Sensitivity Standard

Vanadium versus Hydrogenous Material (Plexiglas or water)
Pro: Very small amount of inelastic scattering
Pro: optically thin sample → limits multiple scattering.
Con: Small and expensive

Sample: 99.99% pure, 3.5 mm thick, 10 mm diameter
(at λ = 6 Å, 24% isotropic incoherent scattering, 76 % absorption)

Vanadium data collection on NG3 using SDD = 1.3 m
In absolute units and corrected for background.
(vacuum in sample chamber…)