

From raw data to the absolute scaling: determination of the water cross sections on D22

The only way to calculate molecular mass, volume fraction or specific surface of particles is to determine the scattering cross section in cm^{-1} . Thanks to the absolute scaling, scattering curves measured on different spectrometers or in different q-range can be compared. It also provides information on possible multiple scattering or unexpected aggregation.

We recall that the scattering intensity in cm^{-1} is:

$$\frac{d\Sigma}{d\Omega} \Big|_{\text{Total}}(q) = \frac{N(q)}{\Phi_o \cdot \Delta\Omega(q) \cdot Tr(q) \cdot e(q) \cdot t} \quad (1)$$

Φ_o : flux at the sample position

$\Delta\Omega(q)$: the solid angle covered by a pixel at the position q ($\Delta\Omega = p(q)^2/D(q)^2$ in steradian).

$Tr(q)$: transmission of the sample in respect to the empty beam

t: acquisition time

$e(q)$: sample thickness

Remark: as shown in the following, the transmission and the thickness depend on q.

The samples are generally filled in a cell (EC) that contributes to the general scattering. The scattering from the EC is subtracted from the total scattering by

$$\begin{aligned} \frac{d\Sigma}{d\Omega} \Big|_{\text{Sample}}(q) &= \frac{1}{e_{\text{Sample}}} \frac{N_{\text{Sample}}(q)}{\Phi_o \cdot \Delta\Omega(q) \cdot Tr_{\text{Sample}} \cdot t_{\text{Sample}}} - \frac{N_{\text{EC}}(q)}{\Phi_o \cdot \Delta\Omega(q) \cdot Tr_{\text{EC}} \cdot t_{\text{EC}}} \quad (2) \\ &= \frac{1}{e_{\text{Sample}} \Phi_o \Delta\Omega(q)} \left| \frac{N_{\text{Sample}}(q)}{Tr_{\text{Sample}} \cdot t_{\text{Sample}}} - \frac{N_{\text{EC}}(q)}{Tr_{\text{EC}} \cdot t_{\text{EC}}} \right| \end{aligned}$$

A possibility to get the absolute scaling is to use standards with known cross section [1,2]. For SANS, samples with predominant incoherent scattering such as light water (H_2O) or vanadium are currently used for the absolute scaling. Water scattering has a flat scattering independent from the scattering angle. It is also used to correct the cells of the detector from efficiency variations.

$$\left(\frac{d\Sigma}{d\Omega} \right)_{\text{sample}} = \frac{I_{\text{sample}}}{I_{\text{standard}}} \left(\frac{d\Sigma}{d\Omega} \right)_{\text{standard}} \quad (3)$$

A water sample of thickness 0.1 cm is generally used as standard for calibration on D22. However, due to the inelastic and multiple scattering effects, the water scattering is not totally isotropic, but stronger in the forward direction. The assumption that the neutrons that are not transmitted are scattered uniformly in 4π steradians is wrong. A wavelength dependent correction factor $g(\lambda)$ has to be introduced to represent the real scattering cross section

$$\left(\frac{d\Sigma}{d\Omega} \right)_{\text{H}_2\text{O}}^{\text{real}} = g(\lambda) \frac{1 - Tr}{4\pi \cdot 0,1 \cdot Tr} \quad (4)$$

$(d\Sigma/d\Omega)_{\text{H}_2\text{O}}^{\text{real}}$ and $g(\lambda)$ are instrument dependent.

For large sample-to-detector distance and collimation, the flux and water count rate are too low to get a good statistics in a reasonable time. Water scattering measured in an other configuration (but same λ) is used to correct the variations of efficiency of the detector. Then, a scaling factor f corrects from the flux and the solid angle difference between the two configurations used. f can be estimated with

$$f = \frac{(\Phi_0 \Delta\Omega)_{\text{sample}}}{(\Phi_0 \Delta\Omega)_{\text{H}_2\text{O}}} = \frac{\text{Coll}_{\text{H}_2\text{O}} D_{\text{H}_2\text{O}}}{\text{Coll}_{\text{sample}} D_{\text{sample}}} \Bigg|^2 = \frac{\Phi_{\text{sample}}}{\Phi_{\text{H}_2\text{O}}} \frac{D_{\text{H}_2\text{O}}}{D_{\text{sample}}} \Bigg|^2 \quad (5)$$

Where $D_{\text{sample}/\text{H}_2\text{O}}$ are the sample-to-detector distances for sample and water respectively; $\text{Coll}_{\text{sample}/\text{H}_2\text{O}}$ the collimations and $\Phi_{\text{sample}/\text{H}_2\text{O}}$ are the fluxes at the sample

Finally, the absolute intensity is obtained after background correction and water normalization according:

$$\frac{d\Sigma}{d\Omega}_{\text{sample}} = \frac{1}{f} \frac{d\Sigma}{d\Omega}_{\text{H}_2\text{O}}^{\text{real}} \frac{\frac{I_{\text{sample}}}{Tr_{\text{sample}}} - \frac{I_{\text{sample-EC}}}{Tr_{\text{sample-EC}}} \Bigg| \frac{1}{e_{\text{sample}}}}{\frac{I_{\text{H}_2\text{O}}}{Tr_{\text{H}_2\text{O}}} - \frac{I_{\text{H}_2\text{O-EC}}}{Tr_{\text{H}_2\text{O-EC}}} \Bigg| \frac{1}{e_{\text{H}_2\text{O}}}} \quad (6)$$

The subscript EC refers to the empty cell. Tr is the transmission calculated in comparison with the empty beam, I is the number of neutron per second measured on the detector.

Determination of the water cross section $(d\Sigma/d\Omega)_{H_2O}^{real}$

A first possibility to determine $(d\Sigma/d\Omega)_{H_2O}^{real}$ is to use standards (polymers) with a known cross section (see Lindner). The author approximate the water cross section by

$$(d\Sigma/d\Omega)_{H_2O}^{real} = 0.66223 + 0.026928\lambda + 0.0021599\lambda^2 - 4.43 \times 10^{-6} \lambda^3 \quad (7)$$

The second possibility is a direct measurement using eq (2) that requires to know Φ_o ; $\Delta\Omega(q)$ and Tr .

Incident intensity Φ_o

The incident intensity is the number of neutrons per second at the sample position. The easiest is to have a calibrated monitor at the sample position. If not one other possibility is to measure the attenuated direct beam. We call Σ_{EB} , the sum of neutron integrated on the surface of the direct beam, t_{EB} the acquisition time and F , the factor of attenuation. Then, taking into account the detector dead time τ

$$\Phi_o (n/s) = F \frac{\Sigma_{EB}/t_{EB}}{1 - \tau \Sigma_{EB}/t_{EB}} \quad (8)$$

Attenuation factor F

The attenuation factor may be determined using a strong forward scattered as teflon or graphite. The 4 following spectra have to be registered in the same instrument configuration.

Teflon without attenuator: c_{teflon} (c/s)

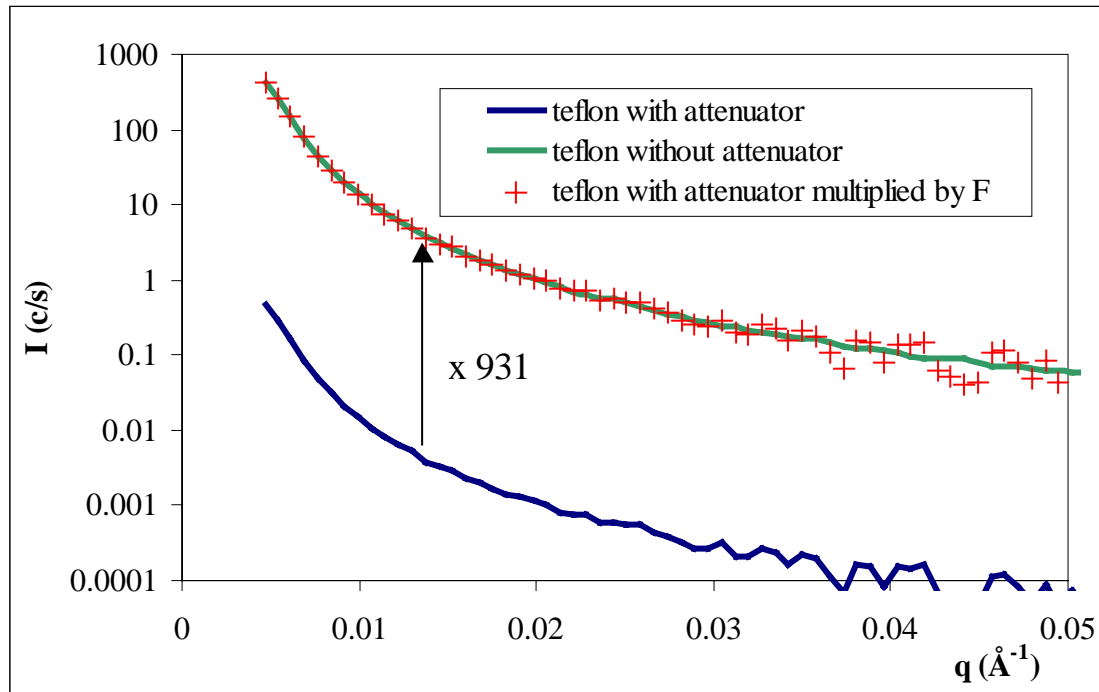
Teflon with attenuator: c_{teflon}^a (c/s)

Electronic background (B4C) without attenuator: c_{B4C} (c/s)

Electronic background (B4C) with attenuator: c_{B4C}^a (c/s)

$$F = \frac{(c_{teflon} - c_{B4C})}{1 - \tau(c_{teflon} - c_{B4C})} \frac{1 - \tau(c_{teflon}^a - c_{B4C}^a)}{(c_{teflon}^a - c_{B4C}^a)} \quad (9)$$

One can also determine the factor necessary to superpose the curves after radial averaging and normalization by time or monitor.



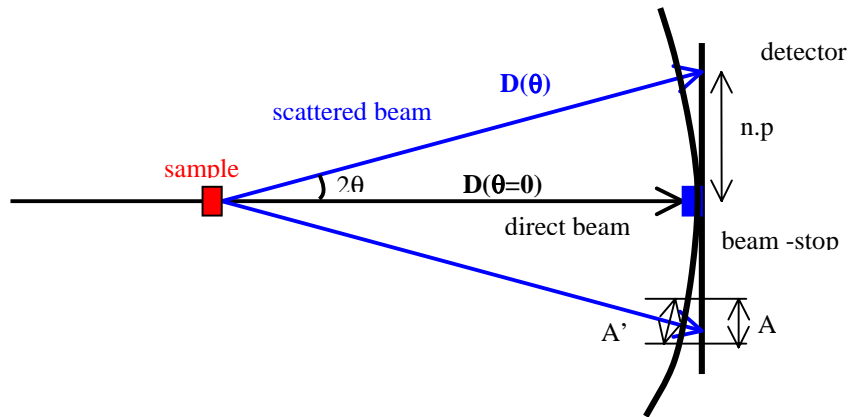
Determination of the attenuation factor with teflon scattering data recorded with and without attenuator

The factors of attenuation for the attenuators n° 2 and 3 and different wavelengths on D22 are summarized below.

Attenuation factors for 5 wavelengths determined with the method described above.

λ (Å)	Attenuator	F
4.48	3	2981
5.83	3	3038
7.75	2	931
9.67	2	927
11.59	2	904
13.52	2	895
15.44	2	887
17.37	2	897

Solid angle $\Delta\Omega(q)$



The solid angle varies with the scattering angle θ and equals

$$\Delta\Omega(2\theta) = A'^2 / (D(2\theta))^2 \text{ with } D(2\theta) = D(2\theta = 0) / \cos(2\theta) \quad (10)$$

$$\text{and } A' = p^2 \cos(2\theta)$$

Finally
$$\Delta\Omega(2\theta) = \frac{p^2 \cos^3(2\theta)}{D(2\theta = 0)}$$

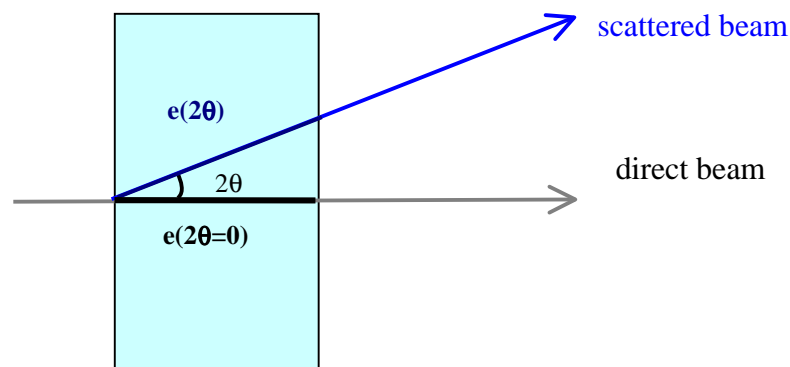
Transmission

The transmission is the ratio of the intensity between the incident beam through the sample and the incident beam.

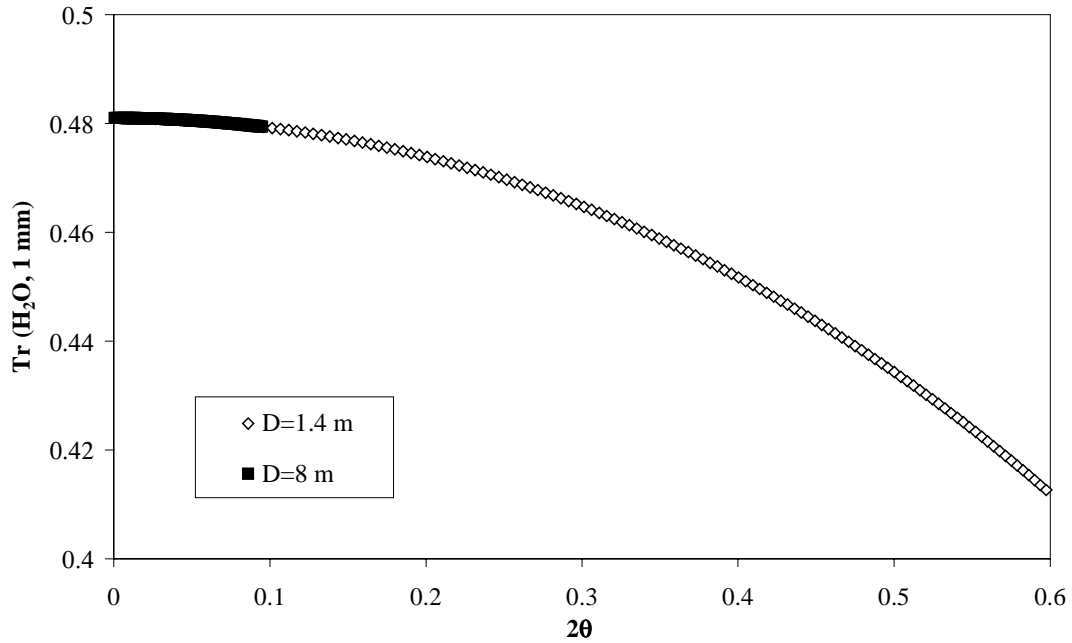
$$Tr(2\theta, \lambda) = \frac{I(2\theta)}{I_0} = \exp(-\mu(\lambda)e(2\theta)) \quad (11)$$

μ the coefficient of adsorption varies with the wavelength. It can be calculated from the transmission at $\theta=0$.

The transmission varies with the scattering angle since the sample thickness increases with θ .

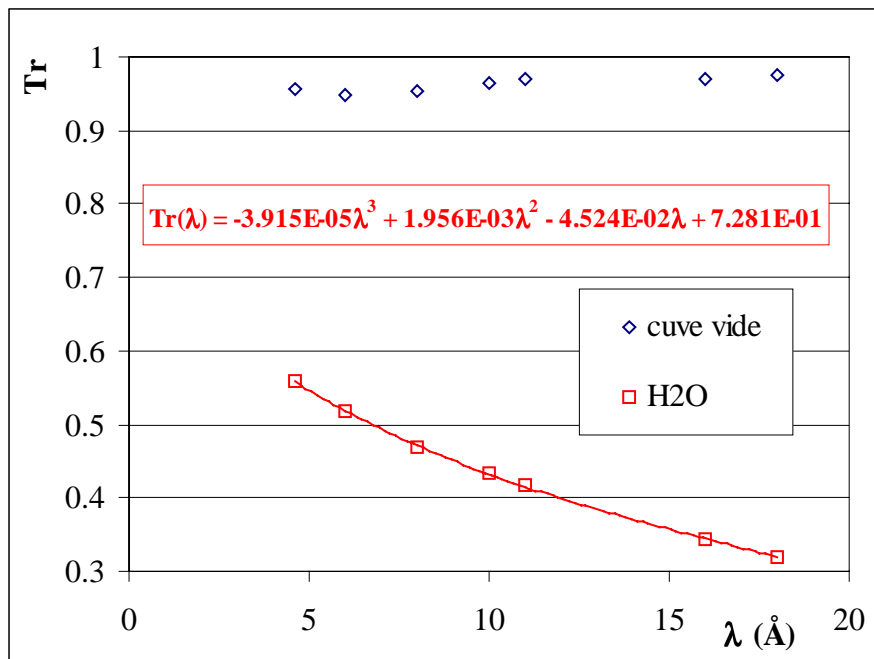


$$e(2\theta) = e(2\theta = 0) / (\cos(2\theta)) \quad (12)$$



Transmission (calculated) of 1 mm water ($\lambda=8 \text{ \AA}$) as a function of the scattering angle 2θ and the sample to detector distance D . For short distances, the transmission decreases of 10% from $\theta=0$ to $\theta=\theta_{\max}$ (edge of the detector).

Transmission of 1mm water as a function of the wavelength



Transmission of 1mm of water as a function of the wavelength

Detector dead time correction

A minimum of time τ is necessary for nearly all the counting devices to separate two events. If a second event comes within a time τ , it will not be detected. This time is called detector dead time. τ depends on the kind of detector and also on the electronics for the data acquisition. τ is of the order of a microsecond, and the dead time losses become important for high-count rate (>50 kHz counts/second).

Two models are generally used to correct the count rate from the detector dead time:

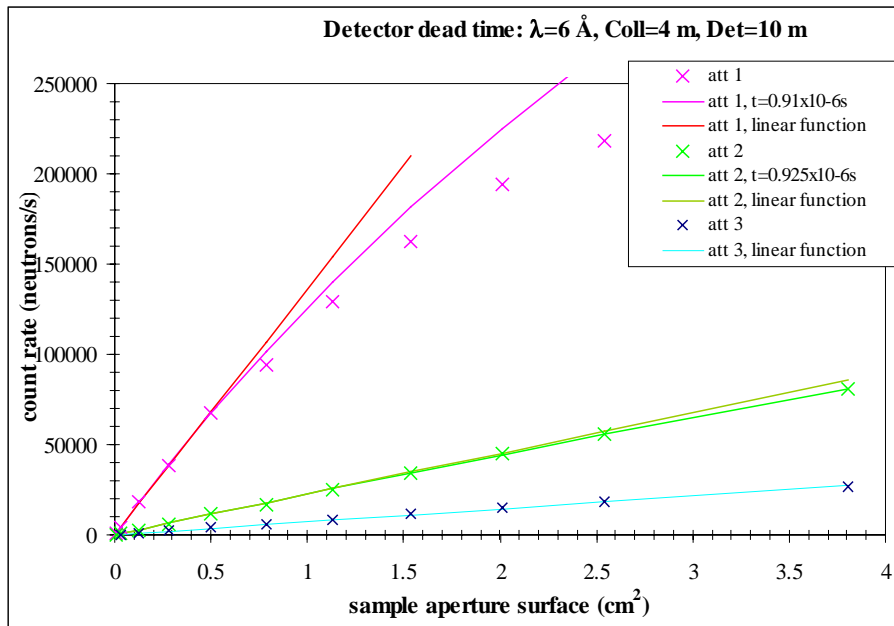
Non-paralysable model

$$(counts / s)_{real} = \frac{(counts / s)_{measured}}{(1 - \tau(counts / s)_{measured})} \quad (13)$$

Paralysable model

$$(counts / s)_{measured} = (counts / s)_{real} \exp(-\tau(counts / s)_{real}) \quad (14)$$

τ measured with the non-paralysable model and used for D22 detector is 0.91×10^{-6} s.



Calibration of the detector dead time.

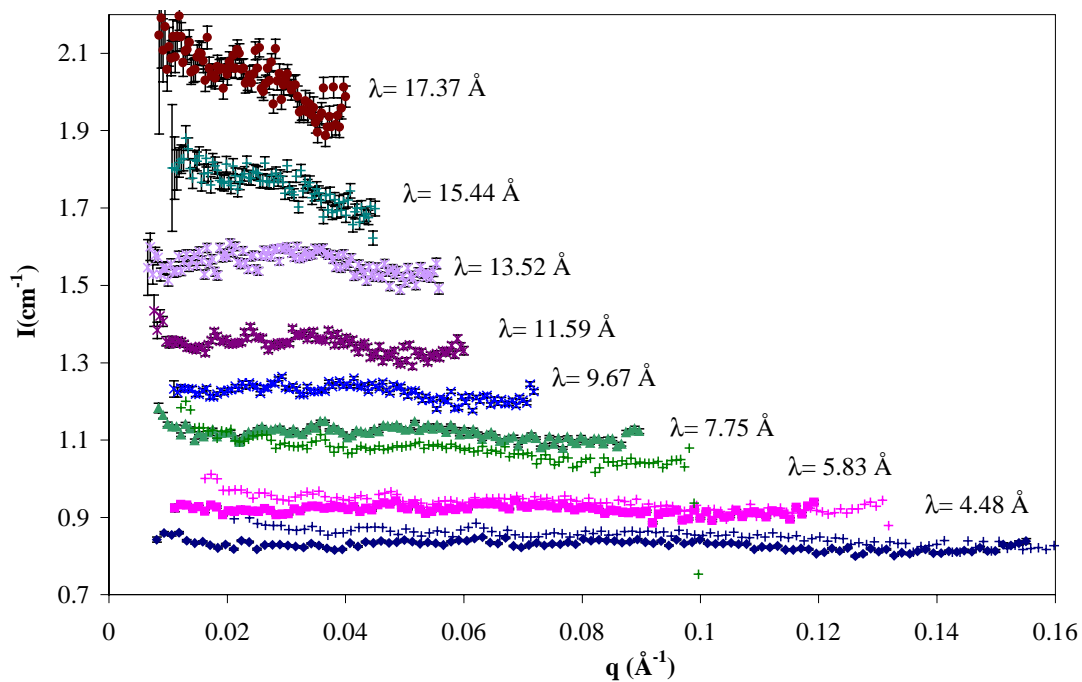
Values of water cross sections for an experiment on D22

In order to minimize the geometric corrections for the detector, we work at D=8m, with a collimation at 8m and a detector offset of 300mm. The wavelengths used are $\lambda=4.48, 5.83, 7.75, 9.67, 11.59$ and 13.51\AA . The scattering curves, after normalization by the incident flux, transmission, water thickness, time and solid angle are shown in the following figure.

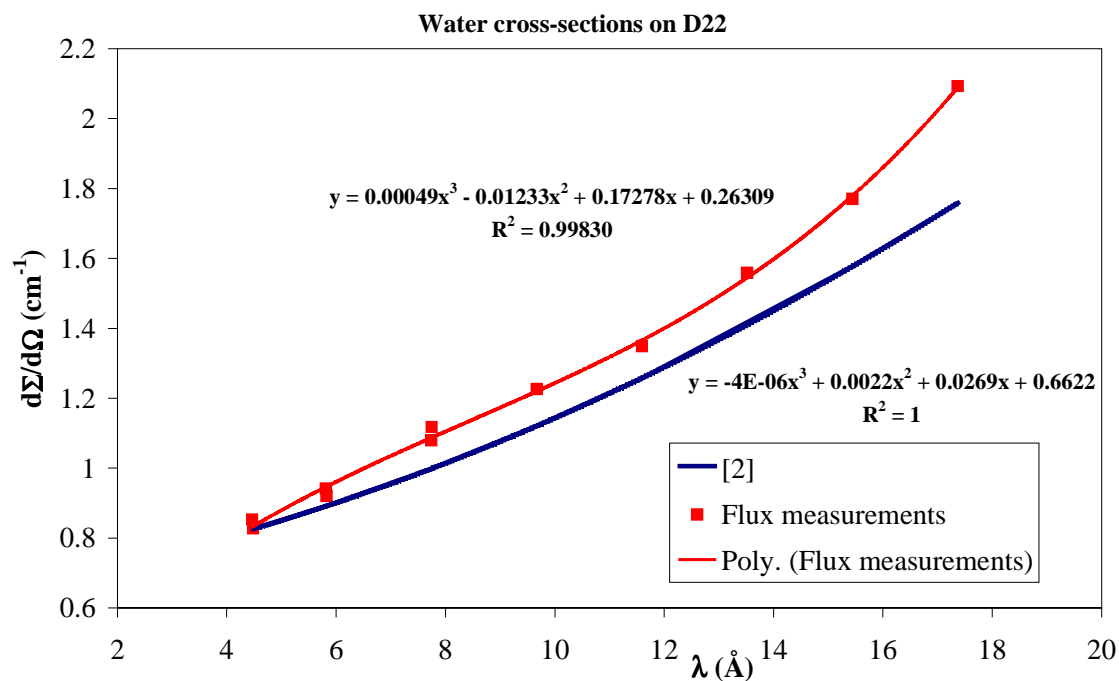
The values of $d\Sigma/d\Omega$ and $g(\lambda)$ obtained through the direct flux measurements (Eq. (2)) are compared to those obtained in [2]. In the wavelength range of 4.5 to 14\AA , the best fit of the $d\Sigma/d\Omega$ experimental values with a polynomial function is:

$$(d\Sigma/d\Omega)_{H_2O}^{real} = 0.26309 + 0.172781\lambda - 0.01233\lambda^2 + 4.9 \cdot 10^{-4} \lambda^3 \quad (15)$$

We note that the values obtained here are higher than those obtained in [2] but of the order of those currently used at ISIS.



Water scattering spectra for 7 wavelengths and after normalization according to Eq. 2.



Comparison of the cross sections of water on D22 obtained by direct flux measurements or using standards polymers [2].

Water cross sections on D22

λ (Å)	$d\Sigma/d\Omega$ (cm ⁻¹) (Eq. 2)	$g(\lambda)$ (Eq. 4)
4.48	0.8273	1.2477
5.83	0.9195	1.1681
7.75	1.1176	1.2184
9.67	1.2259	1.1846
11.59	1.3487	1.1733
15.44	1.770	1.1901
17.37	2.093	1.2636

Do not use these values for data reduction from other small angle instruments!

1 B. Jacrot, G. Zaccai *Biopolymers* (1981) **20**, 2413-2426

2 P. Lindner *J. Appl. Cryst.* (2000) **33**, 807-811