3 Experimental

3.1 Sample Preparation

Distilled H_2O and D_2O 99.9 % pure (Aldrich) were used for measurements and for the preparation of solutions. The following substances: β-CD (Aldrich), TRIMEG >97% and DIMEB >98% (CycloLab), γ-CD >98% (ROTH), were used without further purification. All solutions were filtered (Millipore, 0.22 μm) after preparation. In the calculations the densities of the solutions were assumed to be equal to the density of pure water (1.0 g/mL and 1.1 g/mL for H_2O and D_2O, respectively) because the used concentrations were low.

3.2 QENS experiments

Spectra recorded with the following neutron scattering spectrometers were analyzed:

- NEAT
  The time-of-flight (TOF) spectrometer NEAT (Berlin Neutron Scattering Center (BENSC), Hahn-Meitner-Institut (HMI), Berlin). For description of the instrument, see [66,67,105]. Spectra were corrected using the program FITMO-M [32,34].
- IN16
  The backscattering spectrometer IN16 (Institut-Laue-Langevin (ILL), Grenoble, France). Description of the instrument can be found in [38,49]. Spectra were corrected using the program SQW-F [125].
- IN13
  The backscattering spectrometer IN13 (Institut-Laue-Langevin (ILL), Grenoble, France), for description of the instrument see e.g. [49,88]. Spectra were treated by the program CAPRI [19] followed by corrections by SQW-F [125].
- IN5
  Description of the TOF spectrometer IN5 (Institut-Laue-Langevin (ILL), Grenoble, France) can be found in [95]. Spectra were corrected using the program INX-IN5 [49, 52].
- HFBS (High-Flux-Backscattering spectrometer)
  The instrument is located at NIST (National Institute of Standards and Technology) Center for Neutron Research, Gaithersburg, Maryland, USA. For description of the instrument, see [81]. Initial treatment of the spectra was done using the DAVE software package [23], further correction was done using the program SQW-F.

Spectra of vanadium were either recorded in the course of every particular experiment or were available from the previous measurements (performed with *the same* configuration of the spectrometer, as the configuration employed for recording of the samples' spectra). Spectra of the empty sample container were recorded in every experiment, except for the experiment with IN16. (In the experiment with IN16 the spectra of water could be sucessfully used to correct for the container scattering). In the experiments performed with NEAT, IN5, IN13, IN16 the sample container was a plain slab, in the experiment with HFBS it was a hollow cylinder.

Information on the measured samples is given in Tab. 3.1. Tab. 3.2 contains a short account on the details of QENS experiments.

3.3 SAXS experiment

The synchrotron radiation X-ray scattering data were collected on the X33 camera of EMBL at the storage ring DORIS III (DESY, Hamburg, Germany) [12]. The data were recorded using a MAR345 two-dimensional image plate detector at a sample-detector distance of 2.7 m and a wavelength of λ_0 = 1.5 Å, covering the range of momentum transfer $0.012 < Q < 0.50$ Å^{-1} ($Q = (4\pi/\lambda_0) \times \sin(\varphi/2)$, where φ is the scattering angle). Solutions of β-CD (7.1 mg/mL {6.2 mM}), γ-CD (11.3, 25.6, 53.5 mg/mL {8.7, 19.7, 41.3 mM}), DIMEB (6.6, 15.0, 26.3, 45.5, 51.5, 79.5 mg/mL {5.0, 11.3, 19.7, 34.2, 38.7, 59.8 mM}) and TRIMEG (10.0, 17.0, 35.9, 66.7 and 95.4 mg/mL {6.1, 10.4, 22.0, 40.9, 58.4 mM}) in D_2O were measured in the temperature range 19 °C – 80 °C. The data were circularly (i.e. over the polar angle ψ, see Fig. 3.1) averaged after normalization to the intensity of the incident beam using the program package PRIMUS [58].

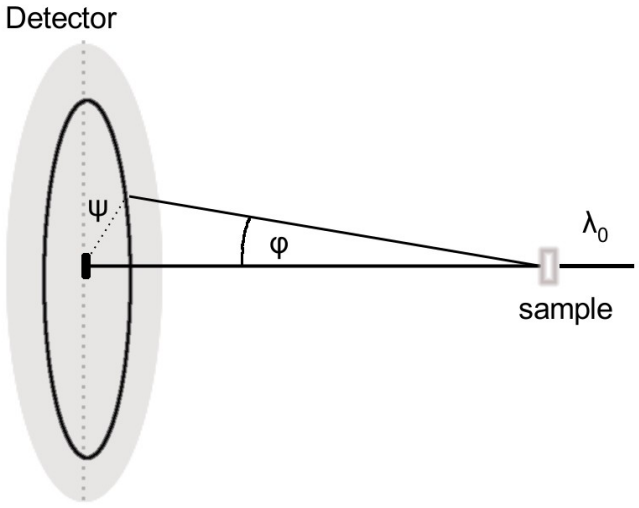

Figure 3.1 **The geometry of the small-angle scattering experiment.**

3.4 SANS experiment

SANS spectra of a DIMEB solution in D_2O (45.2 mg/mL {34 mM}) in the temperature range 20 °C – 60 °C and 6.6, 32.8, 45.5 mg/mL {5.0, 24.6, 34.2 mM} at 25 °C were recorded with the small-angle scattering spectrometer V4 (BENSC, HMI, Berlin) by M. Kammel. The data were reduced and normalized to the scattering by 1 mm H_2O (for details see e.g. [119]) with the program BerSANS [56], rendering SANS patterns $I_{S/SANS}(Q, c)$.

Table 3.1 **Samples measured in QENS experiments.** The columns from left to right: the instrument, temperature range studied, solvent, the number of water molecules per one solute molecule (F_w), solutions which were measured. The name of the instrument (together with the number in the parenthesis, if the same instrument was used to measure more than one sample set) serves for the reference to a particular dataset throughout the whole work. The value of F_w was found assuming the densities of the solutions to be equal to the density of pure water (1.0 g/mL and 1.1 g/mL for H_2O and D_2O, respectively).

Instrument	T °C	Solvent	F_w	Solutions measured
NEAT(1)[a]	12 ≤ 45	D_2O	1463	γ-CD 48.7 mg/mL {37.6 mM}, DIMEB 50.0 mg/mL {37.6 mM}, TRIMEG 61.4 mg/mL {37.6 mM}
NEAT(2)[a]	12 ≤ 45	H_2O	1478	γ-CD 48.7 mg/mL {37.6 mM}, DIMEB 50.0 mg/mL {37.6 mM}, TRIMEG 61.4 mg/mL {37.6 mM}
NEAT(3)[b]	12 ≤ 45	D_2O	1463	γ-CD 48.7 mg/mL {37.6 mM}, DIMEB 50.0 mg/mL {37.6 mM}, TRIMEG 61.4 mg/mL {37.6 mM}
NEAT(4)	7 ≤ 43	D_2O	1382-6962	β-CD 9.0 mg/mL {7.9 mM}, DIMEB 53 mg/mL {39.8 mM}
IN16	7 ≤ 43	D_2O	1382-5914	β-CD 10.6 mg/mL[c] {9.3 mM}[c], DIMEB 27.0 and 53.0 mg/mL {20.3 and 39.8 mM}
IN13	6 ≤ 30	D_2O	1382	DIMEB 53 mg/mL {39.8 mM}
IN5(1)	7 ≤ 43	D_2O	1382-6962	β-CD 9.0 mg/mL {7.9 mM}, DIMEB 53 mg/mL {39.8 mM}
IN5(2)	7 ≤ 50	H_2O	1432-2081	DIMEB 49.4 and 35.5 mg/mL {37.1 and 26.7 mM} TRIMEG 63.4 mg/mL {38.8 mM}
HFBS	7 ≤ 44	H_2O	1474-1535	γ-CD 47.0 mg/mL {36.2 mM}, DIMEB 49.4 mg/mL {37.1 mM}, TRIMEG 61.6 mg/mL {37.7 mM}

[a] recorded by R. E. Lechner in 1999
[b] recorded by R. E. Lechner in 2001
[c] the actual concentration was somewhat smaller, because not all material was dissolved; the solution was filtered before filling the sample container.

Table 3.2 **The details of the QENS experiments.** Incident neutron wavelength λ_0 [Å], elastic energy resolution ΔE [μeV], sample angle α, range of the scattering angles φ, range of the elastic momentum transfers Q_{EL} [$Å^{-1}$], sample thickness d_S [mm]. The column "SC geometry" lists types of the sample containers employed in the experiments. For rectangular slabs the column "Size" provides width and height (width×height) [mm], for circular slabs the column "Size" provides diameter [mm].

	λ_0	ΔE	α	φ	Q_{EL}	d_S	SC geometry	Size
NEAT(1)	5.1	90	45	13.3° ≤ 136.7°	0.29 ≤ 2.29	1.6	slab	N/A[a]
NEAT(2)	5.1	90	45	13.3° ≤ 136.7°	0.29 ≤ 2.29	0.2	slab	N/A[a]
NEAT(3)	15.3	10	60	13.3° ≤ 136.7°	0.095 ≤ 0.76	1.6	Rectangular slab	N/A[a]
NEAT(4)	5.1	90	45	13.3° ≤ 136.7°	0.29 ≤ 2.29	1.6	Circular slab [b]	50
IN16	6.27	1	135	11° ≤ 148.5°	0.19 ≤ 1.93	1.6 - 2.0	Rectangular slab [b]	30 × 40
IN13	2.23	8	120	4° ≤ 336°	0.2 ≤ 4.9	1.6	Rectangular slab [b]	30 × 40
IN5(1)	7.6	30	135[c]	14.5° ≤ 132.5°	0.21 ≤ 1.51	1.6	Circular slab [b]	50
IN5(2)	7.6	30	135[c]	14.5° ≤ 132.5°	0.21 ≤ 1.51	0.2 - 0.4	Circular slab [b]	50
HFBS	6.27	1	-	14.5° ≤ 121.2°	0.25 ≤ 1.75	0.2	Hollow cylinder	[d]

[a] No information is available.
[b] Containers were covered by thin nickel (≈ 5-7 μm) and then gold (< 2 μm) layers.
[c] In the IN5 notation the sample angle is 45°, for consistency NEAT notation is used here.
[d] Inner diameter of the outer cylinder 28.9 mm; outer diameter of the inner cylinder 28.5 mm.