

Supporting Information (Part 2):

Determination of the Exact Particle Radius Distribution for Silica Nanoparticles via Capillary Electrophoresis and Modelling the Electrophoretic Mobility with a Modified Analytic Approximation

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Section S4. DLS: Theoretical Background, Measurement Conditions and Data Evaluation

Autocorrelation functions were recorded for a large set of data gained under variation of the mass fraction of nanoparticles in the sample, under variation of the borax concentration in the sample (30-40 mmol L⁻¹), and under variation of the measurement temperature (15-25 °C). Preliminary experiments performed with a mass fraction of nanoparticles in the sample of 1.0 to 6.2% (refer to Table S3) clearly reveal an increase in the estimated hydrodynamic radius with reduced mass fraction of nanoparticles in the sample. The results also show that the polydispersity index (PDI) with SNP7 is much higher (about 0.220) than that obtained for SNP12 and SNP22 (about 0.080) indicating the presence of aggregates in those samples prepared from SNP7.

With the aim to reach a concentration region allowing to measure the hydrodynamic diameter d_H without a significant influence of the mass fraction of nanoparticles in the sample, we further extended the range of mass fractions investigated reaching mass fractions as low as 0.12-0.22% (refer to Tables S4e-e). Experiments with lower mass fraction (e.g. 0.06% with SNP22 or 0.09% with SNP12) show an unexpected increase in the PDI, which is indicating measurement artefacts due to a too low sample concentration (too low signal-to-noise-ratio).

Results (mean hydrodynamic diameters for different types of distribution) obtained with varied ionic strength, varied mass fraction of nanoparticles in the sample and varied temperature are listed in Tables S4e-e. While the unexpectedly high result for SNP7 (in combination with the high PDI) indicates the presence of aggregates, the results for SNP12 and SNP22 (in combination with a lower PDI) underestimate the mean hydrodynamic diameter of the number-based size distribution (taking the size distribution determined by TEM as a reference, *cf.*, Table 1).

In Figure S3 the experimental diffusion coefficient D_e (measured for SNP12 and SNP22 at constant scattering angle 173° [I], determined for $c(\text{borax}) = 30$ or 40 mmol L^{-1} and for $T = 25 \text{ }^\circ\text{C}$ or $15 \text{ }^\circ\text{C}$) is plotted against the mass fraction of nanoparticles in the sample. As expected from theory [II], we observe a linear decrease in D_e with increasing mass concentration β (or increasing mass fraction w) of nanoparticles in the sample (refer to Figure S4), which can be described with following equation [II]:

$$D_e = D_0 (1 + \alpha_D \beta) \quad (\text{S2.1})$$

with D_0 = diffusion coefficient at infinitely low concentration of nanoparticles, α_D = constant. From regression data α_D can be calculated, being a measure for particle-particle interactions. Extrapolation to infinitely low concentration gives access to D_0 . The resulting data are listed in Table S2 (for regression lines refer to Figure S4). Comparison of the data reveals that the ionic strength of the dispersant has a measurable impact on α_D (temperature-invariant), while extrapolation to infinite concentration results in a constant value of D_0 independent of the ionic strength of the dispersant. The results are in agreement with those values reported by Finsy *et al.* [III] (see Table S2 and Figure S5). We therefore conclude, that plotting $1/r_{H,e}$ ($r_{H,e}$ = result of CONTIN algorithm [IV] for mean of number-based size distribution) against w should result in a straight line. The y-intercept obtained by this method corresponds to the reciprocal mean of the corrected number-based size distribution and allows to calculate the hydrodynamic diameter d_H (mean of number-based size

^I Perez Holmberg, J.; Abbas, Z.; Ahlberg, E.; Hassellöv, M.; Bergenholtz, J. Nonlinear Concentration Dependence of the Collective Diffusion Coefficient of TiO₂ Nanoparticle Dispersions. *J. Phys. Chem. C* **2011**, *115*, 13609-13616.

^{II} Kops-Werkhoven, M. M.; Fijnaut, H. M. Dynamic light scattering and sedimentation experiments on silica dispersions at finite concentrations. *Chem. Phys.* **1981**, *74*, 1618-1624.

^{III} Finsy, R.; Moreels, E.; Bottger, A.; Lekkerkerker, H. Study of the relation between diffusion and sedimentation of charged silica sols by dynamic light scattering, ultracentrifugation, and turbidimetry. *J. Chem. Phys.* **1985**, *82*, 3812-3816.

^{IV} Pecora, R. Dynamic light scattering measurement of nanometer particles in liquids. *J. Nanopart. Res.* **2000**, *2*, 123-131.

distribution) without bias due to the difference between D_e and D_0 . The resulting regression lines are depicted in Figure S6.

The CONTIN algorithm, which is based on a reverse Laplace transformation [IV], allows to calculate size distributions directly from recorded autocorrelation functions. However, the number-based size distribution functions generated (refer to Figures S2a and b) indicate that the observed difference Δd_H is due to the limitations of the CONTIN algorithm employed. The number-based size distribution functions generated show (as expected) a dependence of the mean value on the mass fraction of nanoparticles in the sample. In contrast to our expectations, these curves have (invariant of the measurement conditions) in first approximation the shape of a logarithmic Gaussian function, which is in sharp contrast to the narrower left-skewed distributions obtained via TEM (refer to Figure 1). Pecora [IV] has emphasized that the CONTIN algorithm allows only in favourable circumstances to calculate correctly the weight- and number-based size distributions of colloidal nanoparticle populations. In our case, DLS has failed to provide an alternative to TEM for obtaining accurate number-based size distribution functions. Linearity of the function $D_e = f(w)$ (w = mass fraction of nanoparticles in the sample) suggests that the observed deviations in the characteristic parameters of the generated number-based size distribution functions are not due to the presence of aggregates in the sample [I], which is in full agreement with the TDA results.

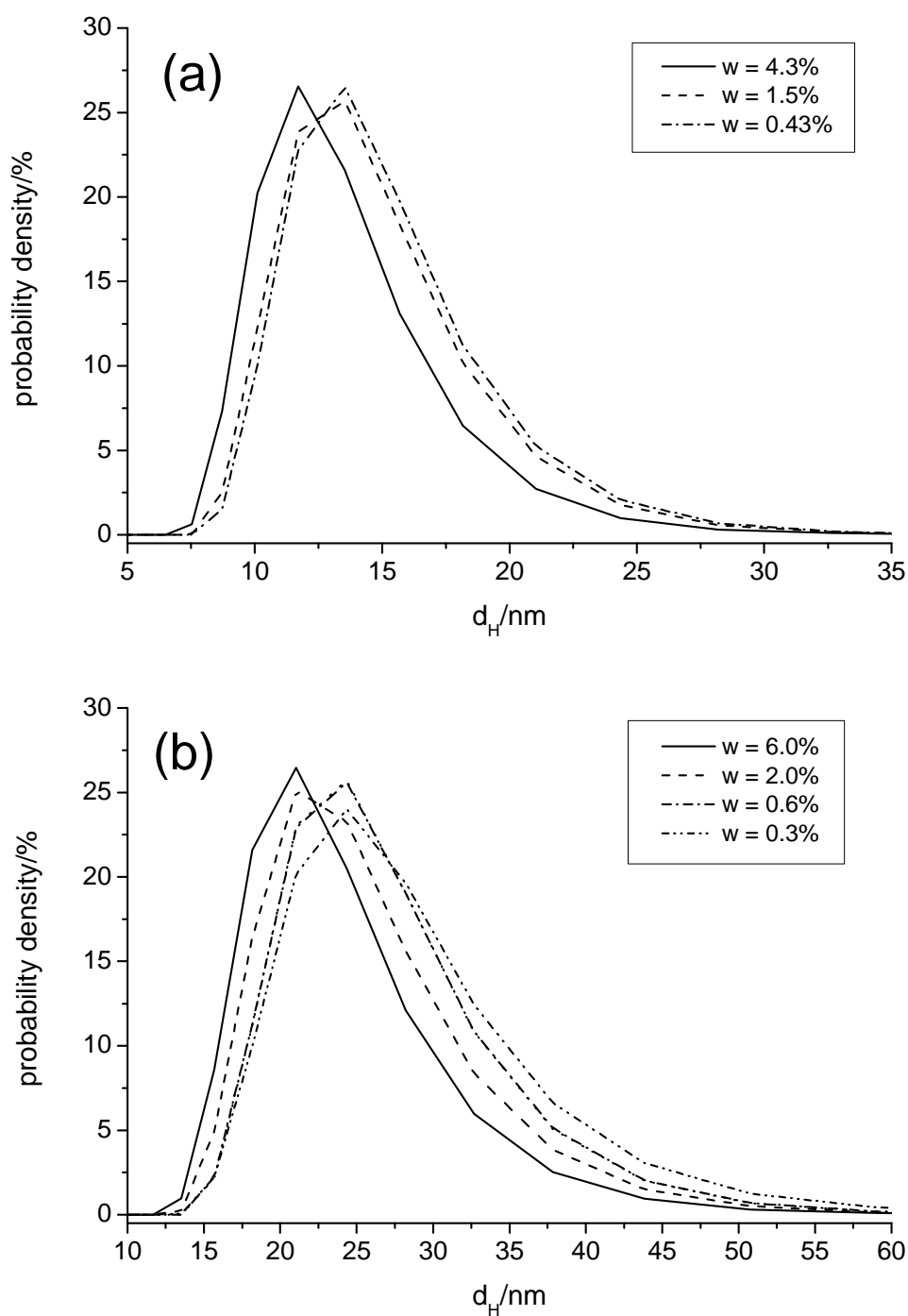


Figure S2. Number-based probability density plot for (a) SNP12 and (b) SNP22 from DLS data dependent on mass fraction w of nanoparticles in the sample, $c(\text{borax}) = 40 \text{ mmol L}^{-1}$, $T = 25 \text{ }^\circ\text{C}$ (each data point = mean of five consecutive measurements, *cf.*, Tables S4a-e).

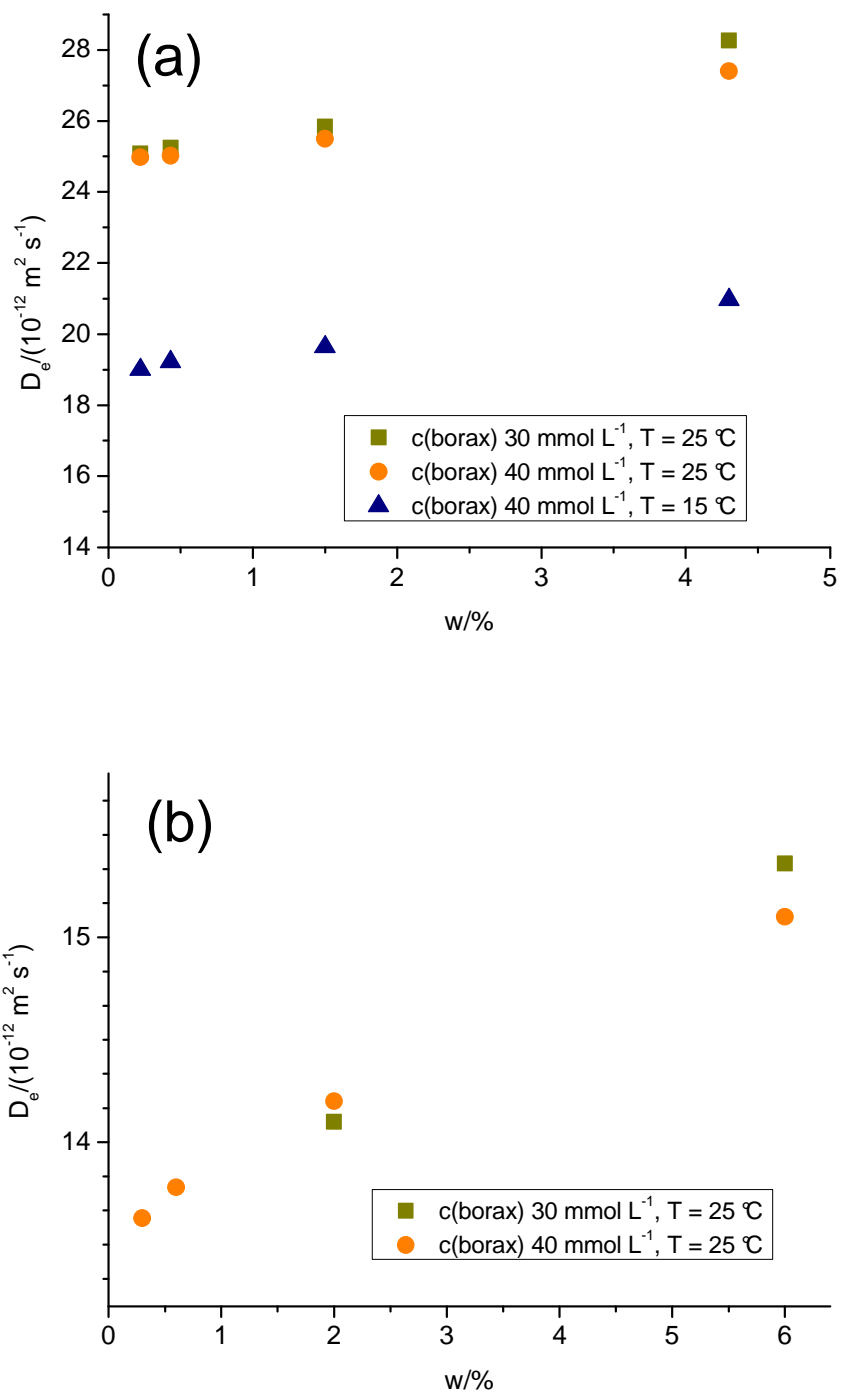
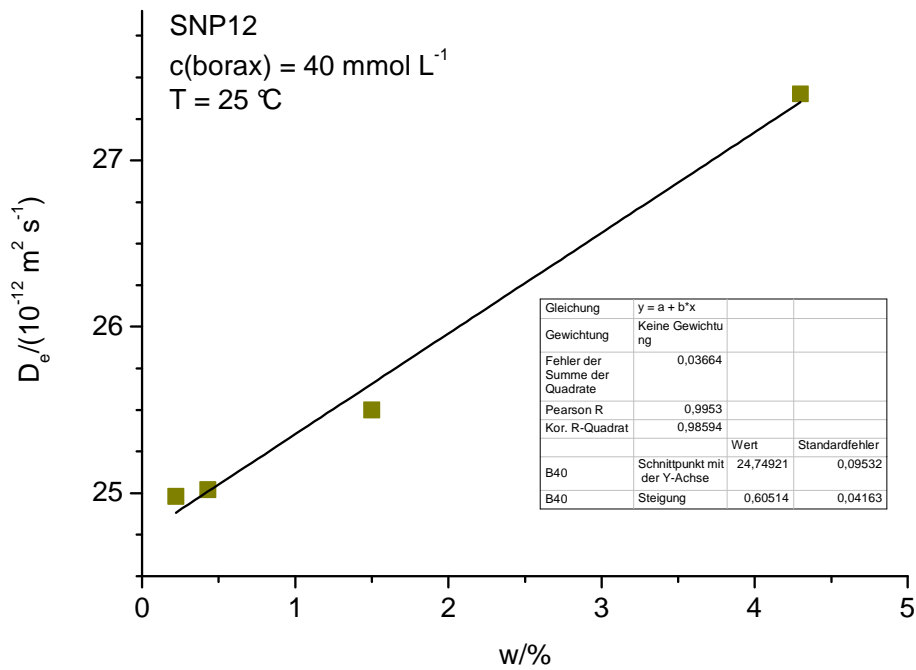
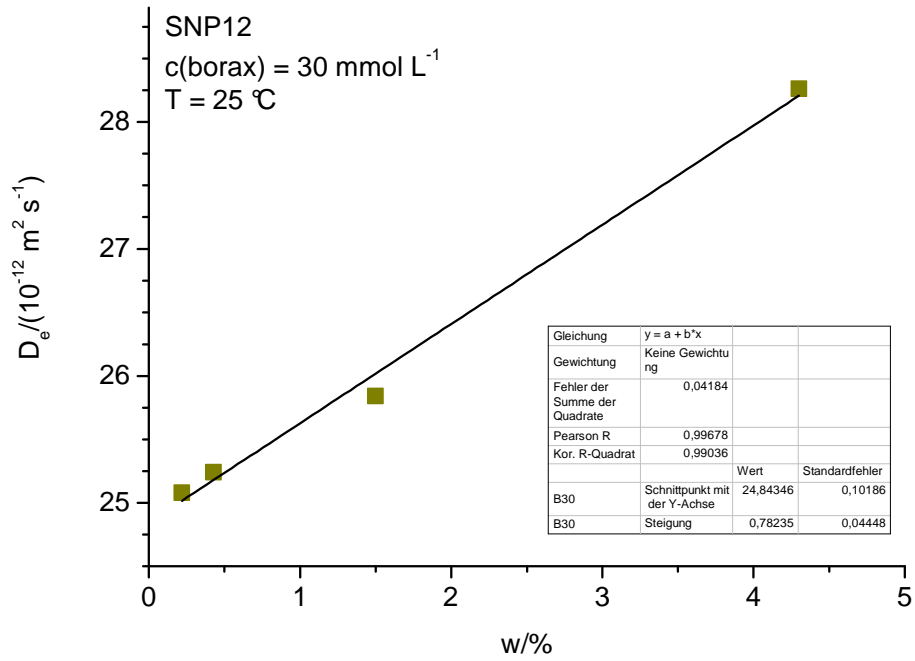
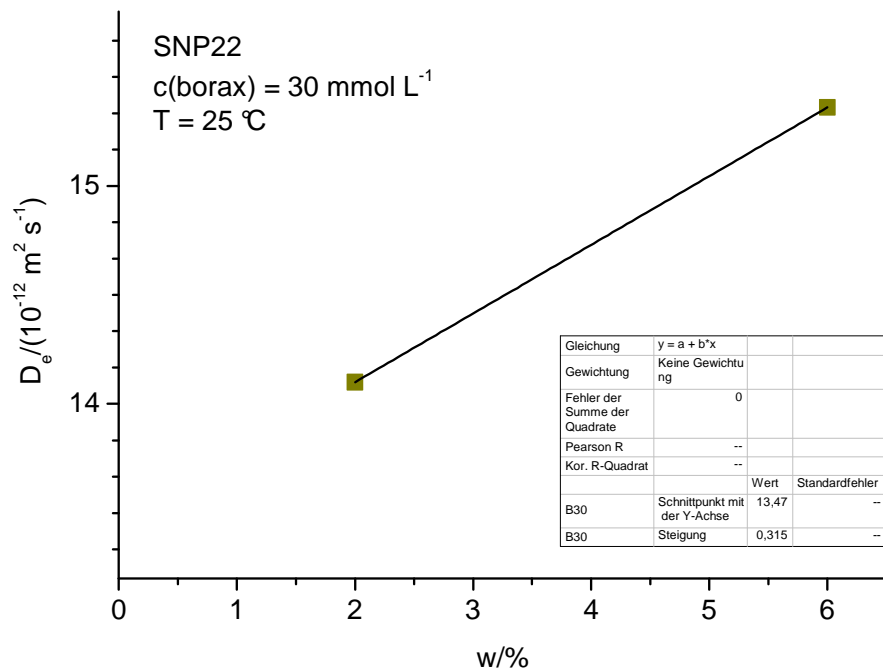
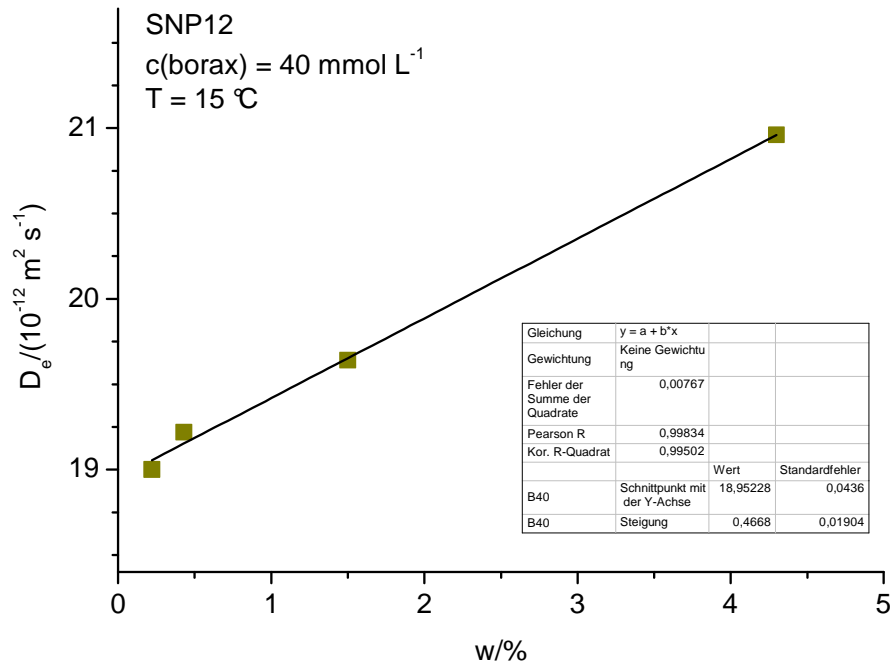


Figure S3. Plotting of experimental diffusion coefficient D_e against mass fraction w of nanoparticles in the sample (a) SNP12, (b) SNP22 (for experimental values refer to Tables S4a-e).





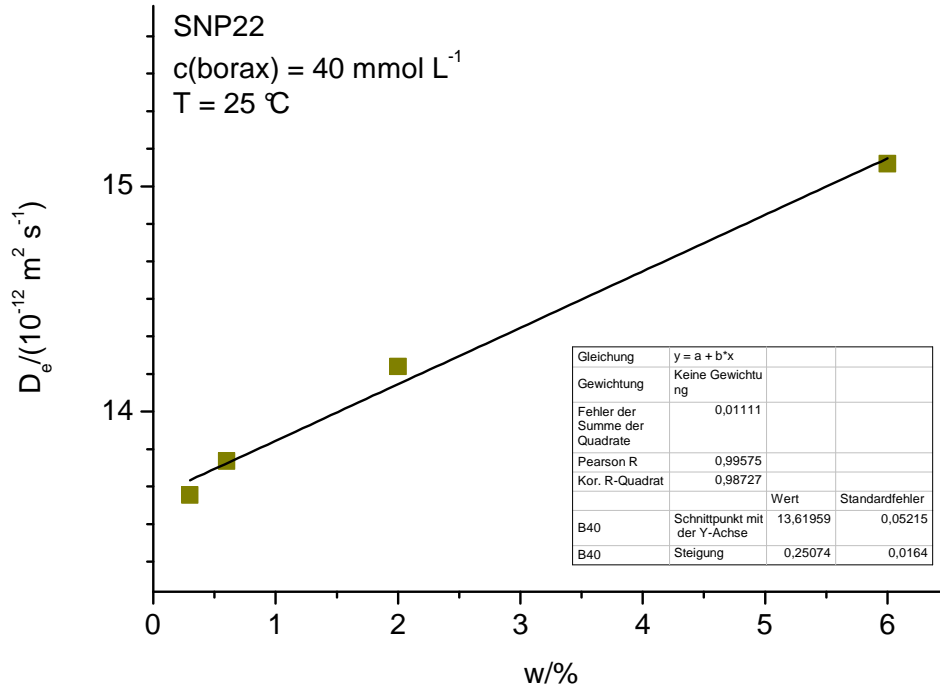


Figure S4. Linear regression analysis, plotting experimental diffusion coefficient D_e against mass fraction w of nanoparticles in the sample (for experimental values refer to Tables S4a-e).

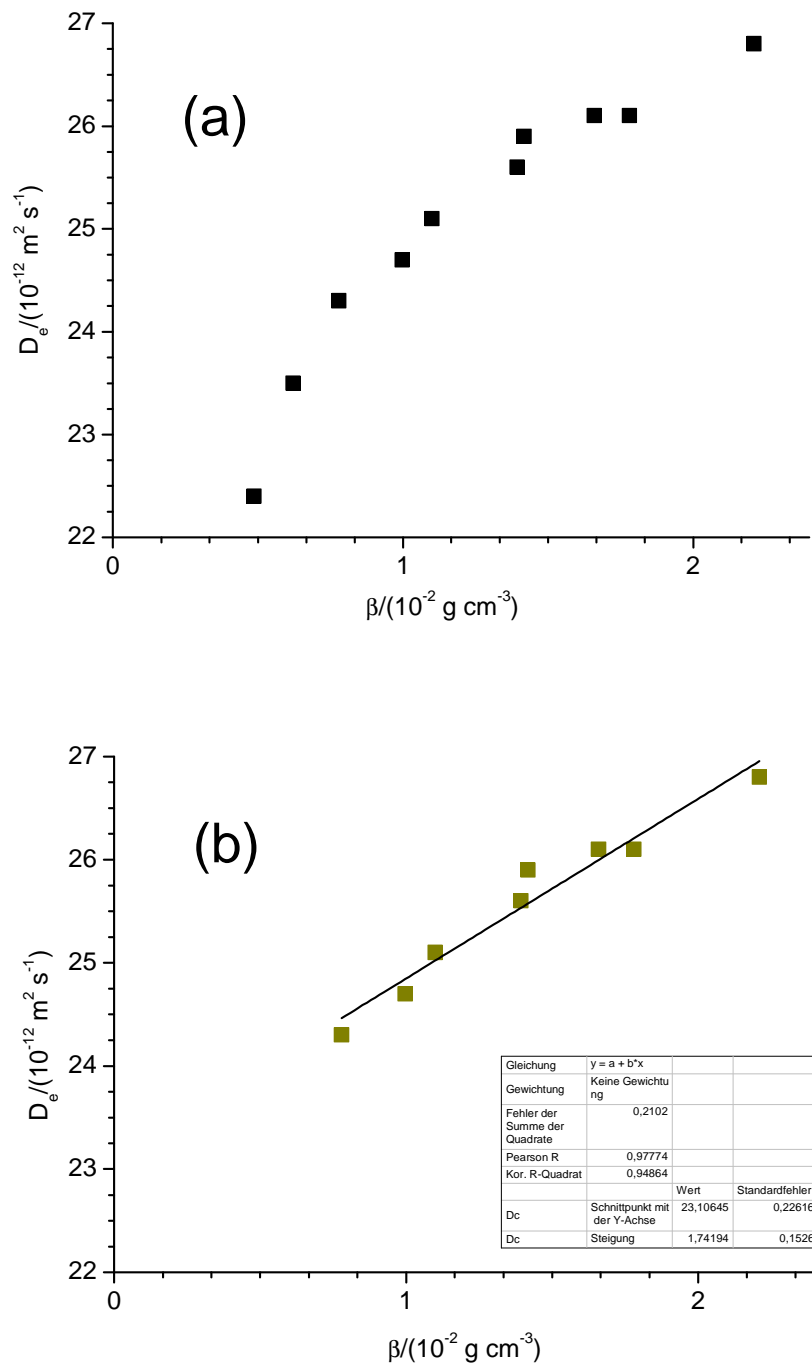
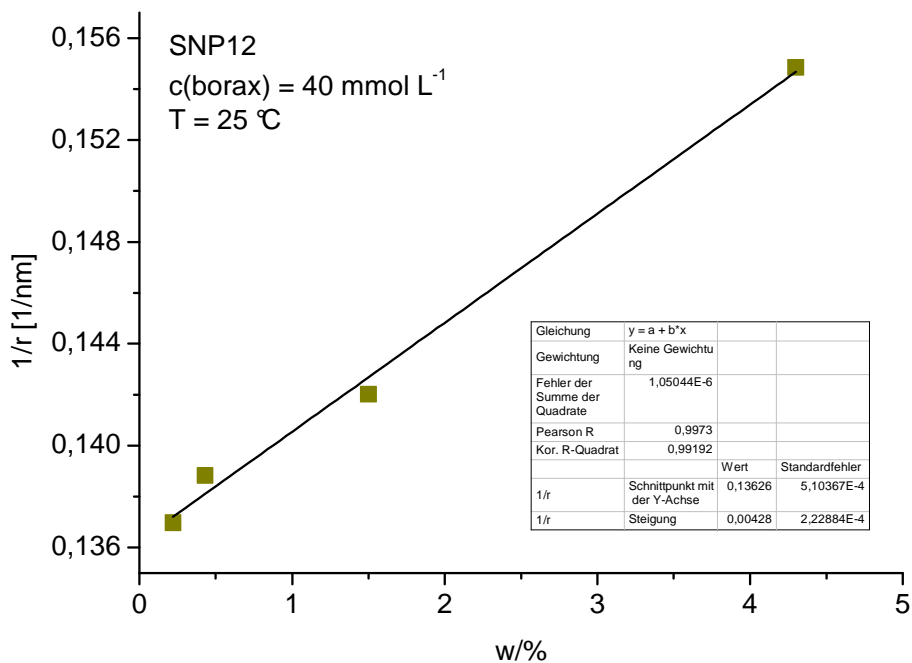
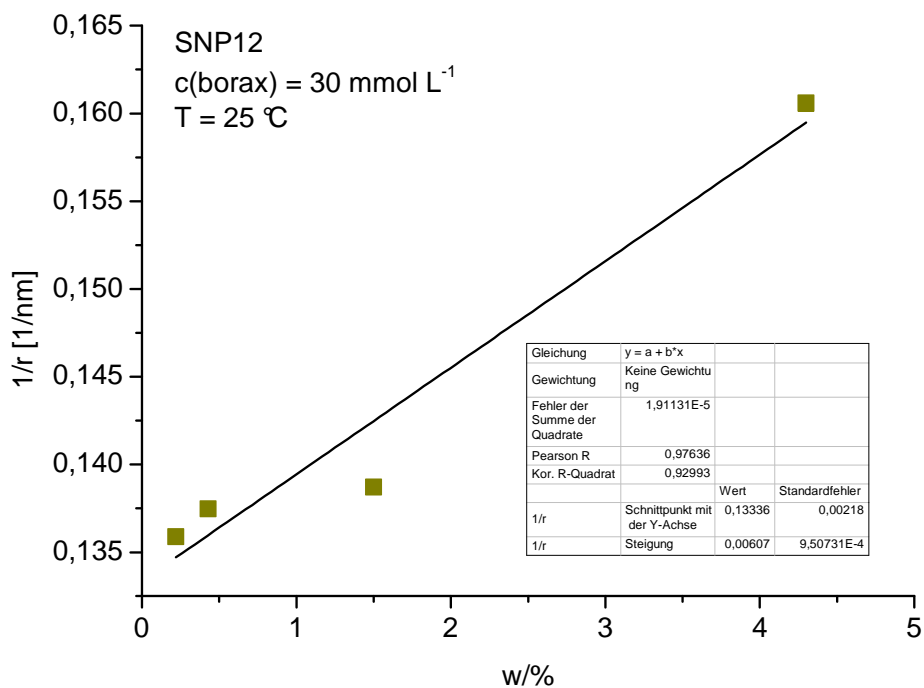
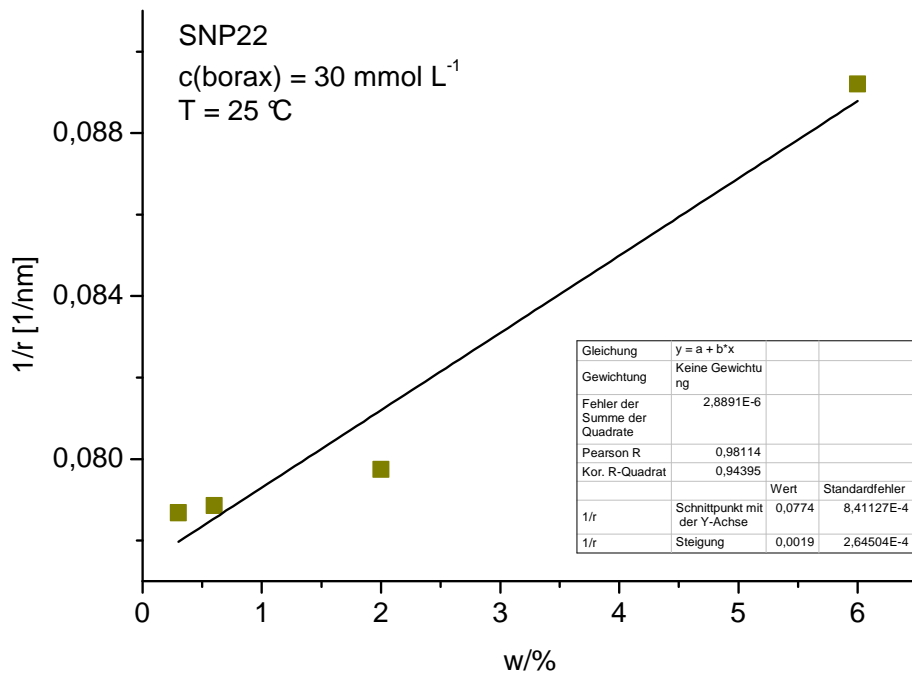
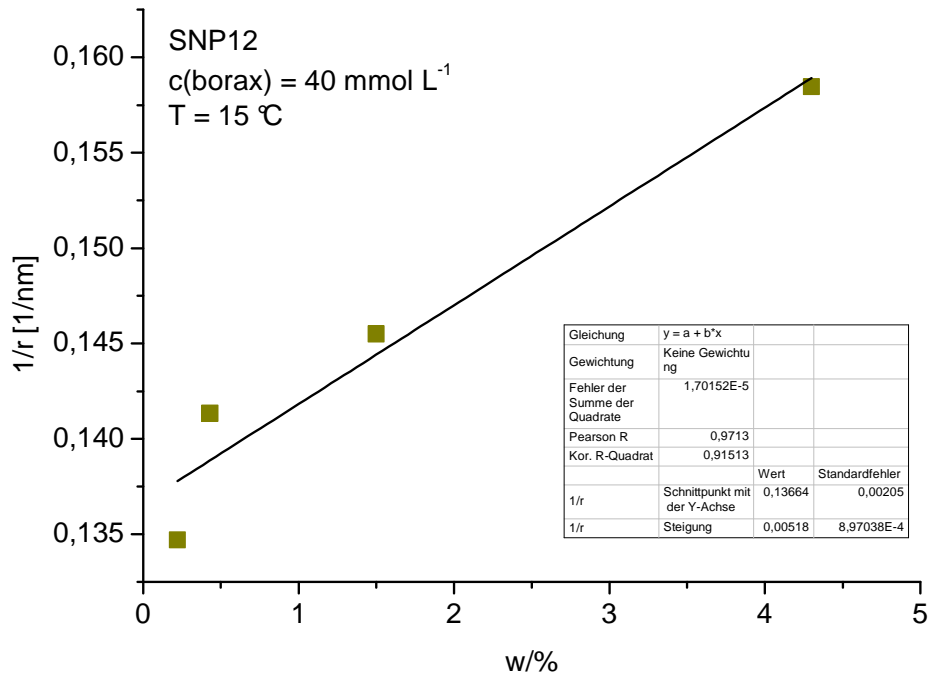


Figure S5. (a) Plotting experimental diffusion coefficient D_e against mass concentration β of nanoparticles in the sample, (b) linear regression analysis within selected range (data taken from [III]).





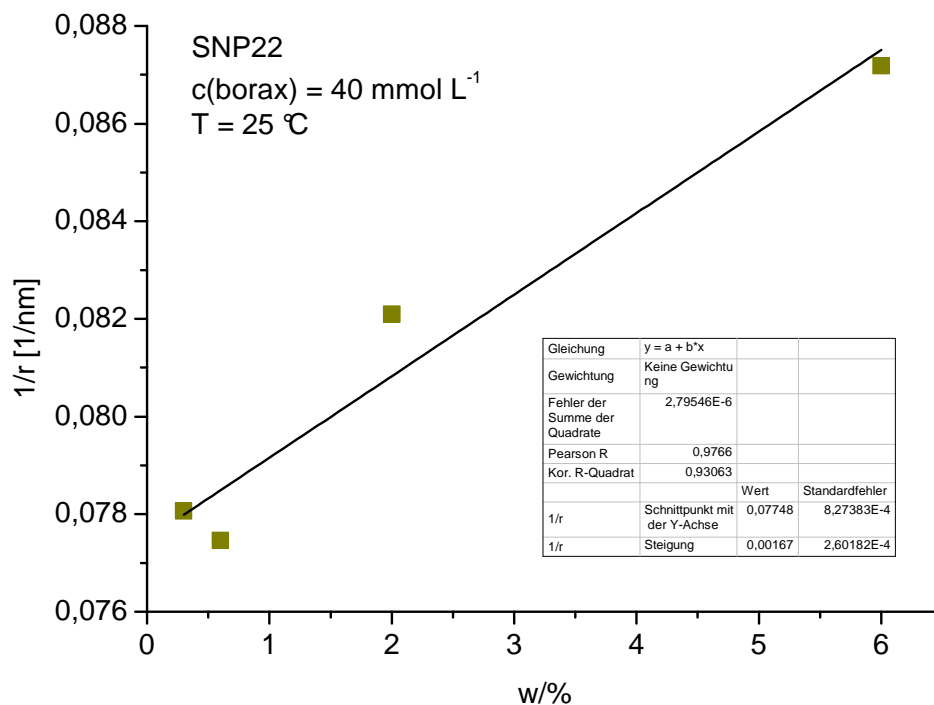


Figure S6. Linear regression analysis, plotting the reciprocal number-based mean hydrodynamic radius $1/r_H$ against mass fraction w of nanoparticles in the sample (for experimental parameters refer to Tables S4a-e).

Table S2. Diffusion constant D_0 at infinite dilution and constant α_D obtained by DLS at a scattering angle of 173° under variation of experimental parameters (mean values from five subsequent measurements, standard errors (from regression analysis) in brackets, for experimental conditions refer to Tables S4a-e). The corresponding regression lines are given in Figures S4 and S5.

	$D_0/(10^{-12} \text{ m}^2 \text{ s}^{-1})$	$\alpha_D/(\text{cm}^3 \text{ g}^{-1})$	R^2
SNP12, T = 25 °C, c(borax) = 30 mmol L ⁻¹	24.8 (± 0.10)	3.15 (± 0.18)	0.99036
SNP12, T = 25 °C, c(borax) = 40 mmol L ⁻¹	24.7 (± 0.10)	2.45 (± 0.17)	0.98594
SNP12, T = 15 °C, c(borax) = 40 mmol L ⁻¹	19.0 (± 0.04)	2.46 (± 0.10)	0.99502
SNP22, T = 25 °C, c(borax) = 30 mmol L ⁻¹	13.5 ^a	2.34 ^a	----- ^a
SNP22, T = 25 °C, c(borax) = 40 mmol L ⁻¹	13.6 (± 0.05)	1.84 (± 0.12)	0.98727
SNP12 ^b , T = 25 °C, c(NaCl) = 0.01 mmol L ⁻¹	23.1 (± 0.23)	7.54 (± 0.66)	0.94864

^a values calculated from only two data points, ^b data taken from [III].

Table S3. Results of dynamic light scattering experiments (preliminary results under variation of the mass fraction of nanoparticles in the sample), N = 3; T = 25 °C.

Sample		<z> /nm	PDI	Mean I /nm	Mean V /nm	Mean N /nm	Width N /nm
SNP7 (5.8%)	MW	9.74	0.228	12.39	6.173	4.543	1.368
	SD	0.0165	0.0038	0.3479	0.0836	0.0615	0.0163
	RSD	0.17%	1.66%	2.81%	1.35%	1.35%	1.19%
SNP7 (3.0%)	MW	10.71	0.220	14.04	6.992	5.110	1.548
	SD	0.0416	0.0070	0.7580	0.1348	0.2324	0.0284
	RSD	0.39%	3.20%	5.40%	1.93%	4.55%	1.84%
SNP7 (1.0%)	MW	11.16	0.202	14.47	7.109	5.178	1.570
	SD	0.0000	0.0055	0.2836	0.0821	0.0233	0.0099
	RSD	0.00%	2.72%	1.96%	1.16%	0.45%	0.63%
SNP12 (5.9%)	MW	8.300	0.092	9.156	6.989	5.779	1.479
	SD	0.0059	0.0045	0.0411	0.0382	0.0471	0.0085
	RSD	0.07%	4.88%	0.45%	0.55%	0.81%	0.58%
SNP12 (3.0%)	MW	8.786	0.078	9.559	7.556	6.349	1.576
	SD	0.0433	0.0084	0.0593	0.2057	0.3069	0.0047
	RSD	0.49%	10.71%	0.62%	2.72%	4.83%	0.30%
SNP12 (1.0%)	MW	9.403	0.076	10.20	8.138	6.849	1.705
	SD	0.0500	0.0129	0.0289	0.0489	0.0631	0.0074
	RSD	0.53%	16.99%	0.28%	0.60%	0.92%	0.43%
SNP22 (6.2%)	MW	15.90	0.090	17.55	13.40	11.02	2.856
	SD	0.0586	0.0123	0.2294	0.2363	0.3900	0.0121
	RSD	0.37%	13.76%	1.31%	1.76%	3.54%	0.42%
SNP22 (3.0%)	MW	16.81	0.077	18.30	14.48	12.10	3.048
	SD	0.0656	0.0095	0.0723	0.1646	0.1744	0.0361
	RSD	0.39%	12.39%	0.40%	1.14%	1.44%	1.18%
SNP22 (1.0%)	MW	17.48	0.075	19.03	15.00	12.47	3.183
	SD	0.0557	0.0010	0.0404	0.1358	0.1686	0.0204
	RSD	0.32%	1.33%	0.21%	0.91%	1.35%	0.64%

<z> = z-average value; PDI = polydispersity index; Mean I = mean of intensity-based size distribution; Mean V = mean of volume-based size distribution; Mean N = mean of number-based size distribution; Width N = width of number-based size distribution; MW = arithmetic mean; SD = standard deviation; RSD = relative standard deviation

Table. S4a. Results of dynamic light scattering experiments, SNP7, $c(\text{borax}) = 40 \text{ mmol L}^{-1}$, $N = 5$; $T = 15$ or $25 \text{ }^\circ\text{C}$.

Sample		$\langle z \rangle$ /nm	PDI	Mean I /nm	Mean V /nm	Mean N /nm	Width N /nm
SNP7 (4.3%) 25 °C	MW	11.09	0.214	14.230	7.118	5.120	1.581
	SD	0.04658	0.0058	0.1691	0.1221	0.1783	0.0181
	RSD	0.42%	2.72%	1.19%	1.72%	3.48%	1.14%
SNP7 (1.5%) 25 °C	MW	12.68	0.209	16.120	8.217	5.833	1.838
	SD	0.07887	0.0072	0.2889	0.2922	0.2849	0.0704
	RSD	0.62%	3.44%	1.79%	3.56%	4.88%	3.83%
SNP7 (0.43%) 25 °C	MW	13.00	0.181	16.260	8.664	6.139	1.950
	SD	0.0356	0.0058	0.1173	0.0952	0.2039	0.0236
	RSD	0.27%	3.20%	0.72%	1.10%	3.32%	1.21%
SNP7 (0.22%) 25 °C	MW	14.58	0.256	20.676	9.107	6.714	1.994
	SD	0.2021	0.0177	4.0609	0.2195	0.3892	0.0203
	RSD	1.39%	6.89%	19.64%	2.41%	5.80%	1.02%
SNP7 (4.3%) 15 °C	MW	10.95	0.213	13.62	7.272	5.328	1.613
	SD	0.0744	0.0078	0.0740	0.1435	0.2172	0.0259
	RSD	0.68%	3.67%	0.54%	1.97%	4.08%	1.61%
SNP7 (1.5%) 15 °C	MW	12.42	0.206	15.15	8.287	6.009	1.852
	SD	0.1791	0.0056	0.7093	0.5549	0.8641	0.0634
	RSD	1.44%	2.73%	4.68%	6.70%	14.38%	3.42%

$\langle z \rangle$ = z-average value; PDI = polydispersity index; Mean I = mean of intensity-based size distribution; Mean V = mean of volume-based size distribution; Mean N = mean of number-based size distribution; Width N = width of number-based size distribution; MW = arithmetic mean; SD = standard deviation; RSD = relative standard deviation

Table S4b. Results of dynamic light scattering experiments, SNP12, $c(\text{borax}) = 30 \text{ mmol L}^{-1}$, $N = 5$; $T = 25 \text{ }^{\circ}\text{C}$.

Sample		$\langle z \rangle$ /nm	PDI	Mean I /nm	Mean V /nm	Mean N /nm	Width N /nm
SNP12 (4.3%) 25 °C	MW	8.707	0.084	9.523	7.445	6.227	1.560
	SD	0.03885	0.0089	0.0544	0.1396	0.2084	0.0072
	RSD	0.45%	10.65%	0.57%	1.87%	3.35%	0.46%
SNP12 (1.5%) 25 °C	MW	9.520	0.062	10.212	8.388	7.210	1.702
	SD	0.0299	0.0103	0.0487	0.0537	0.1100	0.0195
	RSD	0.31%	16.52%	0.48%	0.64%	1.53%	1.15%
SNP12 (0.43%) 25 °C	MW	9.755	0.059	10.474	8.521	7.275	1.74
	SD	0.0223	0.0116	0.0555	0.1016	0.1803	0.0194
	RSD	0.23%	19.62%	0.53%	1.19%	2.48%	1.11%
SNP12 (0.22%) 25 °C	MW	9.814	0.063	10.542	8.597	7.359	1.752
	SD	0.0458	0.0033	0.0396	0.0902	0.1298	0.0066
	RSD	0.47%	5.33%	0.38%	1.05%	1.76%	0.38%

$\langle z \rangle$ = z-average value; PDI = polydispersity index; Mean I = mean of intensity-based size distribution; Mean V = mean of volume-based size distribution; Mean N = mean of number-based size distribution; Width N = width of number-based size distribution; MW = arithmetic mean; SD = standard deviation; RSD = relative standard deviation

Table S4c. Results of dynamic light scattering experiments, SNP12, $c(\text{borax}) = 40 \text{ mmol L}^{-1}$, $N = 5$; $T = 15$ or $25 \text{ }^\circ\text{C}$.

Sample		$\langle z \rangle$ /nm	PDI	Mean I /nm	Mean V /nm	Mean N /nm	Width N /nm
SNP12 (4.3%) 25 °C	MW	8.978	0.087	9.826	7.705	6.458	1.610
	SD	0.02172	0.0039	0.0620	0.1310	0.1856	0.0144
	RSD	0.24%	4.47%	0.63%	1.70%	2.87%	0.89%
SNP12 (1.5%) 25 °C	MW	9.657	0.073	10.466	8.338	7.041	1.726
	SD	0.0727	0.0080	0.1318	0.1998	0.3088	0.0313
	RSD	0.75%	11.09%	1.26%	2.40%	4.39%	1.81%
SNP12 (0.43%) 25 °C	MW	9.837	0.069	10.636	8.523	7.203	1.771
	SD	0.0327	0.0051	0.0619	0.0759	0.1255	0.0096
	RSD	0.33%	7.39%	0.58%	0.89%	1.74%	0.54%
SNP12 (0.22%) 25 °C	MW	9.849	0.065	10.606	8.583	7.301	1.767
	SD	0.0475	0.0084	0.0336	0.1021	0.1622	0.0125
	RSD	0.48%	13.07%	0.32%	1.19%	2.22%	0.71%
SNP12 (4.3%) 25 °C	MW	8.800	0.090	9.649	7.537	6.311	1.576
	SD	0.0821	0.0068	0.1400	0.0532	0.1069	0.0155
	RSD	0.93%	7.61%	1.45%	0.71%	1.69%	0.98%
SNP12 (1.5%) 15 °C	MW	9.394	0.073	10.173	8.146	6.873	1.700
	SD	0.1200	0.0073	0.1131	0.1373	0.1587	0.0213
	RSD	1.28%	9.95%	1.11%	1.69%	2.31%	1.25%
SNP12 (0.43%) 15 °C	MW	9.594	0.071	10.368	8.344	7.075	1.726
	SD	0.1031	0.0067	0.1117	0.1554	0.2211	0.0141
	RSD	1.07%	9.44%	1.08%	1.86%	3.12%	0.82%
SNP12 (0.22%) 15 °C	MW	9.705	0.059	10.388	8.550	7.424	1.739
	SD	0.0349	0.0109	0.1076	0.1304	0.3320	0.0145
	RSD	0.36%	18.35%	1.04%	1.53%	4.47%	0.84%

$\langle z \rangle$ = z-average value; PDI = polydispersity index; Mean I = mean of intensity-based size distribution; Mean V = mean of volume-based size distribution; Mean N = mean of number-based size distribution; Width N = width of number-based size distribution; MW = arithmetic mean; SD = standard deviation; RSD = relative standard deviation

Table S4d. Results of dynamic light scattering experiments, SNP22, $c(\text{borax}) = 30 \text{ mmol L}^{-1}$, $N = 5$; $T = 15$ or $25 \text{ }^\circ\text{C}$.

Sample		$\langle z \rangle$ /nm	PDI	Mean I /nm	Mean V /nm	Mean N /nm	Width N /nm
SNP22 (6.0%) 25 °C	MW	16.04	0.094	17.72	13.56	11.21	2.868
	SD	0.0625	0.0116	0.1842	0.3117	0.4559	0.0231
	RSD	0.39%	12.38%	1.04%	2.30%	4.07%	0.80%
SNP22 (2.0%) 25 °C	MW	17.46	0.079	19.05	15.00	12.54	3.142
	SD	0.0205	0.0172	0.2579	0.3125	0.5072	0.0273
	RSD	0.12%	21.68%	1.35%	2.08%	4.05%	0.87%
SNP22 (0.60%) 25 °C	MW	17.54	0.072	19.03	15.15	12.68	3.19
	SD	0.0561	0.0097	0.0669	0.1374	0.1977	0.0111
	RSD	0.32%	13.48%	0.35%	0.91%	1.56%	0.35%
SNP22 (0.30%) 25 °C	MW	17.57	0.077	19.09	15.17	12.71	3.178
	SD	0.0600	0.0104	0.2277	0.2206	0.3807	0.0259
	RSD	0.34%	13.48%	1.19%	1.45%	3.00%	0.81%
SNP22 (0.12%) 25 °C	MW	17.55	0.066	18.89	15.45	13.23	3.122
	SD	0.0611	0.0078	0.0522	0.0400	0.0773	0.0365
	RSD	0.35%	11.79%	0.28%	0.26%	0.58%	1.17%
SNP22 (0.60%) 15 °C	MW	17.12	0.074	18.60	14.76	12.34	3.105
	SD	0.2253	0.0092	0.2928	0.2977	0.3386	0.0525
	RSD	1.32%	12.40%	1.57%	2.02%	2.74%	1.69%
SNP22 (0.30%) 15 °C	MW	17.10	0.073	18.51	14.86	12.51	3.111
	SD	0.2422	0.0120	0.3891	0.1446	0.1920	0.0555
	RSD	1.42%	16.41%	2.10%	0.97%	1.54%	1.78%
SNP22 (0.12%) 15 °C	MW	17.33	0.065	18.64	15.21	12.96	3.122
	SD	0.0929	0.0083	0.1069	0.2671	0.4452	0.0365
	RSD	0.54%	12.76%	0.57%	1.76%	3.43%	1.17%

$\langle z \rangle$ = z-average value; PDI = polydispersity index; Mean I = mean of intensity-based size distribution; Mean V = mean of volume-based size distribution; Mean N = mean of number-based size distribution; Width N = width of number-based size distribution; MW = arithmetic mean; SD = standard deviation; RSD = relative standard deviation

Table S4e. Results of dynamic light scattering experiments, SNP22, $c(\text{borax}) = 40 \text{ mmol L}^{-1}$, $N = 5$; $T = 15$ or $25 \text{ }^{\circ}\text{C}$.

Sample		$\langle z \rangle$ /nm	PDI	Mean I /nm	Mean V /nm	Mean N /nm	Width N /nm
SNP22 (6.0%) 25 °C	MW	16.31	0.093	17.98	13.83	11.47	2.921
	SD	0.0543	0.0081	0.1144	0.0549	0.0999	0.0234
	RSD	0.33%	8.68%	0.64%	0.40%	0.87%	0.80%
SNP22 (2.0%) 25 °C	MW	17.36	0.090	19.12	14.72	12.18	3.107
	SD	0.0731	0.0152	0.2495	0.4473	0.6273	0.0459
	RSD	0.42%	16.89%	1.30%	3.04%	5.15%	1.48%
SNP22 (0.60%) 25 °C	MW	17.86	0.084	19.50	15.38	12.91	3.22
	SD	0.0669	0.0128	0.1716	0.3563	0.6379	0.0139
	RSD	0.37%	15.29%	0.88%	2.32%	4.94%	0.43%
SNP22 (0.30%) 25 °C	MW	17.64	0.078	19.18	15.26	12.81	3.196
	SD	0.0896	0.0055	0.1300	0.2012	0.3258	0.0278
	RSD	0.51%	7.02%	0.68%	1.32%	2.54%	0.87%
SNP22 (0.12%) 25 °C	MW	17.79	0.083	19.41	15.35	12.85	3.219
	SD	0.0581	0.0125	0.2311	0.2515	0.4262	0.0123
	RSD	0.33%	15.12%	1.19%	1.64%	3.32%	0.38%
SNP22 (0.60%) 15 °C	MW	17.31	0.071	18.70	15.11	12.80	3.131
	SD	0.2736	0.0057	0.3419	0.3038	0.3776	0.0445
	RSD	1.58%	7.97%	1.83%	2.01%	2.95%	1.42%
SNP22 (0.12%) 15 °C	MW	17.45	0.077	18.92	15.21	12.87	3.148
	SD	0.0537	0.0097	0.2354	0.2536	0.4669	0.0440
	RSD	0.31%	12.54%	1.24%	1.67%	3.63%	1.40%

$\langle z \rangle$ = z-average value; PDI = polydispersity index; Mean I = mean of intensity-based size distribution; Mean V = mean of volume-based size distribution; Mean N = mean of number-based size distribution; Width N = width of number-based size distribution; MW = arithmetic mean; SD = standard deviation; RSD = relative standard deviation