

Appendix: Compositions of Preparations

Table A1.1 Reagent quantities for most synthesis gels studied in this work.

Sample Name	CTAB /g	Water 1 [†] /g	Sodium Silicate Solution /g	Water 2 [†] /g	Conc. H ₂ SO ₄ /g	Water 3 [†] /g	Additive	Total Additives /g
A	3.2279	9.6749	3.7480	7.9351	0.2379	3.9438	-	-
B	2.2441	6.7263	2.6058	5.5166	0.1654	2.7418	-	-
C	3.2279	9.6749	3.7480	7.9351	0.2379	3.9438	-	-
D	2.2441	6.7263	2.6058	5.5166	0.1654	2.7418	-	-
E	3.2279	9.6749	3.7480	7.9351	0.2379	3.9438	-	-
F	2.2441	6.7263	2.6058	5.5166	0.1654	2.7418	-	-
UC Prep (AR1)	16.4031	49.2298	19.0688	40.7028	1.2268	20.1174	-	-
AR2	8.2839	24.6198	9.6000	20.2710	0.6246	10.0243	-	-
GP Prep	16.3866	49.50	19.0634	40.59	1.2015	20.05	-	-
Argonne Prep 1	32.758	98.3399	38.14	81.02	2.407	40.10	-	-
Argonne Prep 2	8.1963	24.5953	9.5378	20.2713	0.6009	10.023	-	-
R Prep heated	20.4772	61.4865	23.8121	50.6878	1.5689	25.3343	CH ₃ CO ₂ H	22.50
R Prep unheated	20.4772	61.4865	23.8121	50.6878	1.5689	25.3343	CH ₃ CO ₂ H	20.10
SAD1	16.7798	50.2503	20.795	40.2360	1.2421	20.4063	CH ₃ CO ₂ H	20.90
SAD2	16.6291	50.1304	19.304	40.1973	1.2799	20.5345	-	-
X2	3.3471	10.0314	3.8720	8.0717	0.2451	4.0229	NaCH ₃ CO ₂	2.9896
X3	3.3471	10.0314	3.8720	8.0717	0.2451	4.0229	H ₂ SO ₄	1.4332
X4	3.3471	10.0314	3.8720	8.0717	0.2451	4.0229	CH ₃ CO ₂ H	2.98
X5	3.3471	10.0314	3.8720	8.0717	0.2451	4.0229	NH ₄ Br	4.1170
LOQ1	0.0010	5.0039	4.6564	1.5273	-	-	-	-
LOQ2	0.0018	5.0004	4.6349	1.5135	-	-	-	-
LOQ3	0.0494	4.9764	4.6500	1.5584	-	-	-	-
LOQ4	0.2519	4.7679	4.6558	1.5353	-	-	-	-
LOQ5	0.5078	4.4964	4.6739	1.5285	-	-	-	-
LOQ6	0.7550	4.2653	4.6397	1.5260	-	-	-	-
LOQ [‡] A-D,I-K, O,P	1.1592	3.3273	1.421	2.6409	0.0806	1.3666	-	-
LOQ [‡] S-W	0.2568	1.6817	0.6985	1.2953	0.0325	0.6841	-	-

LOQ [‡] X- BB	0.1027	1.6703	0.7195	1.3343	0.0335	0.6947	-	-
LOQ shear	8.7726	25.3722	10.8933	20.2642	0.6212	10.3093	-	-
Z1	3.3187	10.0422	4.0722	8.0859	0.2500	4.0328	H ₂ SO ₄	3.1157
Z2	3.3187	10.0422	4.0722	8.0859	0.2500	4.0328	H ₂ SO ₄	1.9577
Z3	3.3187	10.0422	4.0722	8.0859	0.2500	4.0328	H ₂ SO ₄	2.8812
Z4	3.3187	10.0422	4.0722	8.0859	0.2500	4.0328	CH ₃ CO ₂ H	3.70
Z5	3.3187	10.0422	4.0722	8.0859	0.2500	4.0328	HCl	3.8936
IRIS	16.5127	50.0483	20.4422	40.6708	1.2264	20.0620	H ₂ SO ₄	8.15
J1	3.2765	10.1754	3.8082	8.0117	0.2410	4.0003	H ₂ SO ₄	1.70
J2	3.2765	10.1754	3.8082	8.0117	0.2410	4.0003	H ₂ SO ₄	3.15
J3	3.2765	10.1754	3.8082	8.0117	0.2410	4.0003	H ₂ SO ₄	3.15
J4	3.2765	10.1754	3.8082	8.0117	0.2410	4.0003	H ₂ SO ₄	3.47
J5	3.2765	10.1754	3.8082	8.0117	0.2410	4.0003	H ₂ SO ₄	5.00
LOQ [‡] Heated Ordinary 1-5	2.2962	6.6844	2.858	5.316	0.162	2.7232	-	-
LOQ [‡] Heated Acid 1-5	2.3087	6.6500	2.8423	5.3389	0.1688	2.704	H ₂ SO ₄	0.9
QENS	24.4447	75.1470	31.0325	60.1452	1.8367	30.1445	H ₂ SO ₄	19.63
KBr Prep	0.4392	5.2016	1.4680	2.7371	0.0901	-	KBr	0.2425
RP Prep	24.6134	75.1350	31.3480	60.0160	1.8266	30.0140	H ₂ SO ₄	22.62
MARI1	24.3818	75.2116	31.840	60.364	1.8136	30.1656	H ₂ SO ₄	24.11
MARI2	32.8036	100.175	40.997	80.365	2.4038	40.4300	H ₂ SO ₄	23.22
In situ 1	1.1592	3.6997	1.4290	3.7160	0.0869	-	-	-
In situ 2	1.1519	3.3618	1.4249	4.0418	0.1032	-	-	-

Note: † Water 1, 2 and 3 refer to (1) the water added to the solid CTAB to form the surfactant solution, (2) the water added to the acidified sodium silicate solution and (3) the water added after the first two solutions have been mixed during the stirring. Water 3 was also usually used to rinse out the container which formerly contained the surfactant solution.

‡ These solutions were made up with different H₂O/D₂O proportions for the purposes of small angle neutron scattering contrast variation experiments. The quantities given are for the 0% D₂O preparations, but in preparations containing D₂O, the amounts of CTAB, H₂SO₄, and sodium silicate solution would be within ± 0.005 g of the amounts shown. The D₂O was evenly distributed into the various water fractions to create synthesis gels ranging in concentration from 10-90 mol% D₂O.

The preparations are listed in approximately chronological order. Syntheses named with different numbers, but the same letter, correspond to a group of samples which

contain exactly the same amounts of all reagents, except additives. These are cases where a single synthesis gel was made up and then split between the various solutions to observe the effect of the different additives while otherwise maintaining constant reagent proportions.

Table A1.2 Composition of unheated synthesis gels used in shear experiments and a summary of their behaviour under various shear conditions.

CTAB /M	KBr /M	Other Salt	silicate /%	shear rate /s ⁻¹	Temp /K	alignment
0.366	-	0.3042g NaOH 0.1375g H ₂ SO ₄	-	2	298	no
0.366	-	0.3042g NaOH 0.1375g H ₂ SO ₄	-	5	298	no
0.366	-	0.3042g NaOH 0.1375g H ₂ SO ₄	-	500	298	no
0.0336	-	0.0227g NaOH 0.0795g H ₂ SO ₄	-	0	298	no
0.0336	-	0.0227g NaOH 0.0795g H ₂ SO ₄	-	5000	298	no
0.0336	-	1.6227g NaOH 3.4095g H ₂ SO ₄	-	5000	298	no
0.03	0.4	-	-	5000	298	no
0.03	0.4	-	-	5000	313	yes
0.16	0.4	-	-	5000	300-303	no
0.03	0.4	-	1.75	5000	313	yes
0.03	0.4	-	3.86	5000	313	yes
0.06	0.2	-	3.81	5000	313	yes
0.36	-	some NaOH, H ₂ SO ₄	3.86	5000	313	no
0.36	-	some NaOH, H ₂ SO ₄	3.86	5000	323	no
0.06	-	-	3.86	5000	313	no
0.06	-	-	3.86	5000	323	no
0.06	-	0.2M Na ₂ SO ₄	-	5000	313	no
0.03	-	0.4M Na ₂ SO ₄	-	5000	313	no
0.12	0.1	-	-	5000	313	no
0.12	0.2	-	-	5000	313	yes
0.12	0.2	-	-	5000	323	yes
0.12	0.2	-	-	5000	303	yes
0.12	0.2	-	-	5000	293	no
0.12	0.2	-	-	500	303	no
0.12	0.2	-	-	1500	303	no
0.12	0.2	-	-	3000	303	no
0.12	0.2	-	-	5000	303	no
0.12	0.2	-	-	5000	313	yes
0.12	0.2	-	-	5000	303	yes
0.12	0.2	-	-	5000 after stopped 5min	303	yes
0.12	0.2	-	-	1500 after stopped, T inc.	313	yes
0.12	0.2	-	-	3000	313	yes
0.12	0.2	-	-	5000	313	yes
0.12	0.2	-	3.81	5000	313	no

0.12	0.2	-	3.81	5000	323	yes (slight)
0.12	0.2	-	3.81	5000	303	yes
0.12	0.2	-	3.81	5000	293	no
0.12	0.2	-	3.81	5000	313	yes
0.12	0.2	-	3.81	5000 after stopped 5 min	303	yes
0.12	0.2	-	3.81	0	303	no
0.12	0.2	-	3.81	5000	303	no
0.12	0.2	-	3.81	1500 after stopped, T inc.	313	yes (slight)
0.12	0.2	-	3.81	3000	313	yes
0.12	0.2	-	3.81	5000	313	yes
0.12	0.2	-	1.5	5000	313	no
0.12	0.2	-	1.5	5000	323	no
0.12	0.2	-	1.5	5000	303	no
0.12	0.2	-	1.5	5000	313	no
0.12	0.2	-	1.5	5000	323	no
0.12	0.2	-	1.5	5000	333	no
0.12	0.2	-	1.5	5000	323	no
0.12	0.2	-	1.5	5000	313	no
0.12	0.2	-	1.5	5000	303	no
0.36	0.4	-	3.81	0 SANS cell	298	yes
0.36	0.2	-	3.81	0 SANS cell	298	yes
0.36	0.2	-	3.81	0	298	yes (slight)
0.36	0.2	-	3.81	78-3112	298	no
0.36	0.2	-	3.81	39-3112	303	no
0.36	0.2	-	3.81	39-195	313	no
0.36	0.2	-	3.81	389	313	yes
0.36	0.2	-	3.81	778	313	yes
0.36	0.2	-	3.81	1945-3112	313	no
0.19	0.2	-	3.81	0	298	no
0.19	0.2	-	3.81	39	298	yes (slight)
0.19	0.2	-	3.81	79-778	298	no
0.12	0.2	-	3.81	0-389	303	no
0.12	0.2	-	3.81	778	303	yes (slight)
0.12	0.2	-	3.81	1945	303	yes (slight)
0.12	0.2	-	3.81	3112	303	yes
0.12	0.2	-	3.81	0-3112	313	no but IvQ changes noticeably

Note: The shaded region corresponds to the experiments done using the gel which showed the greatest degree of ordering under shear. This is discussed in Chapter 4, section 4.3.3.

The experiment on preparation containing 0.19 M CTAB, despite showing some signs of shear at low temperatures and low shear rates was not continued as the shear cell failed at that point.