

## Electronic Supporting Information

### Effects of Interfacial Specific Cation and Water Molarities on AOT Micelle-to-Vesicle Transitions by Chemical Trapping: The Specific Ion-Pair/Hydration Model

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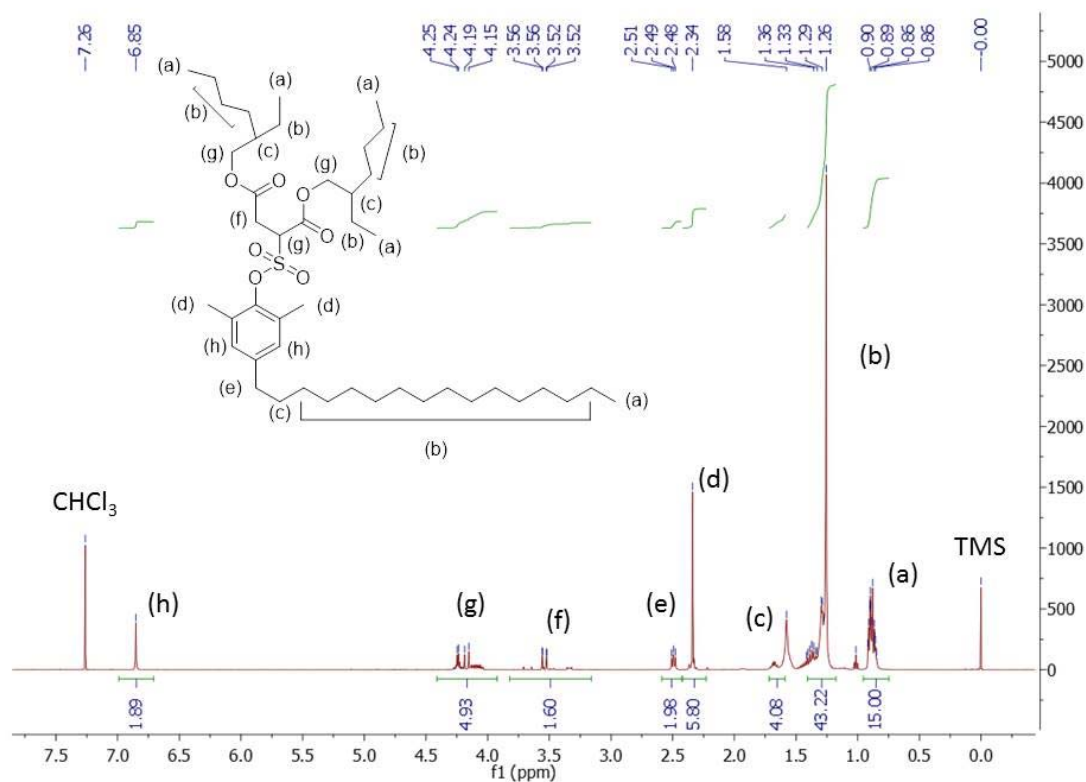
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**S1. Synthesis of bis(2-ethylhexyl) 2-((4-hexadecyl-2,6-dimethylphenoxy)sulfonyl)succinate, 16-ArS.** 16-ArS was prepared by trapping of 16-ArN<sub>2</sub><sup>+</sup> by AOT, modification of the method of Chaudhuri and Srilakshmi.<sup>1</sup> The dediazonation reaction was carried out in a mixture of 15 mM AOT, 20 mM TBAB, and 1 mM HCl. 150 mL 25 mM AOT solution, 5 mL 1 M TBAB, and 2.5 mL 0.1 M HCl were mixed in a 250 mL flask. 67 mg long-chain probe 16-ArN<sub>2</sub>BF<sub>4</sub> was dissolved in 5 mL ice-cold MeCN, and added to the mixture. The reaction mixture was stirred for 2 days at room temperature in dark. The mixture was extracted with EtOAc (3 x 250 mL). The organic layer was isolated, and EtOAc was removed by rotatory evaporation. The crude product was purified by HPLC with a 65% MeOH/35% *i*-PrOH (v/v) mobile phase. <sup>1</sup>H NMR (CDCl<sub>3</sub>) (PPM): 0.88 (15H, t), 1.26 (42H, m), 1.58-1.69 (4H, m), 2.34 (6H, s), 2.48 (2H, t), 3.33-3.56 (2H, 2dd), 4.08-4.25 (5H, d), 6.86 (2H, s), **Figure S1**.



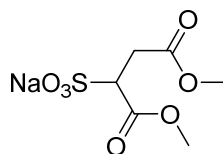
**Figure S1.**  $^1\text{H}$  NMR spectrum of 16-ArS at 500 MHz in  $\text{CDCl}_3$ . Note: f1 =  $\delta$ .

## S2. Standard curves used for the calculation of $\text{AOT}_m$ and $\text{H}_2\text{O}_m$

The chemical trapping reactions with short-chain probe  $1\text{-ArN}_2^+$  in aqueous sodium dimethylsulfosuccinate, SDSS (**Scheme S1**), solutions were carried out by Chaudhuri and Srilakshmi and used for the preparation of standard curves used for the calculation of  $\text{AOT}_m$  and  $\text{H}_2\text{O}_m$ , Equation **S1** and **S2**.<sup>1</sup>

$$\%16\text{-ArS} = 9.036[\text{AOT}_m] (\text{M}) - 0.103 \quad (\text{S1})$$

$$\%16\text{-ArOH} = 0.914[\text{H}_2\text{O}_m] (\text{M}) + 49.72 \quad (\text{S2})$$



**Scheme S1.** Chemical structure of sodium dimethylsulfosuccinate (SDSS)

### S3. Calibration curves for the calculation of dediazonation product yields

**Table S1.** Linear calibration equations for long-chain dediazonation products.<sup>a</sup>

Reaction Products	Calibration Equations <sup>b</sup>	R <sup>2</sup>
16-ArOH	$y = 10.00 \times 10^{10} x - 28660$	0.9998
16-ArS	$y = 1.380 \times 10^{11} x$	1.0000

- a. HPLC Eluting solvent: 35%/65% v/v, *i*-PrOH/MeOH. Flow rate: 0.4 mL/min. Detector wavelength: 220 nm. Injection volume: 100  $\mu$ L.
- b. Units: y-peak area (in  $\mu$ v's), x-concentration (in molarity), and R<sup>2</sup>.

### S4. Chemical trapping data

**Tables S2-S20** list the average peak areas, observed product yields, and normalized product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of various salts, estimated values of AOT<sub>m</sub>, H<sub>2</sub>O<sub>m</sub>, and H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>.

**Table S2.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 1-50 mM **TEABr**, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HBr] = 1 mM.<sup>a</sup>

[TEABr] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
1	Yes	4.98	0.71	86.4	8.9	95.3	90.6	9.4	1.04	44.85	43.2
1	No	5.04	0.71	87.4	8.9	96.3	90.8	9.2			
5	Yes	5.02	0.78	87.0	9.7	96.7	90.0	10.0	1.11	44.10	39.6
5	No	4.95	0.76	85.8	9.4	95.2	90.1	9.9			
10	Yes	4.68	0.78	81.1	9.7	90.8	89.3	10.7	1.20	43.30	36.2
10	No	4.80	0.80	83.2	10.0	93.2	89.3	10.7			
13	Yes	4.64	0.80	80.6	10.0	90.6	88.9	11.1	1.23	42.93	34.8
13	No	4.78	0.82	82.8	10.2	93.1	89.0	11.0			
15	Yes	4.64	0.83	80.4	10.4	90.8	88.6	11.4	1.27	42.60	33.6
15	No	4.74	0.84	82.2	10.4	92.6	88.7	11.3			
20	Yes	4.55	0.86	78.9	10.7	89.6	88.0	12.0	1.32	42.07	31.8
20	No	4.69	0.86	81.3	10.7	92.0	88.3	11.7			
30	Yes	4.51	0.96	78.3	12.0	90.3	86.7	13.3	1.48	40.50	27.4
30	No	4.53	0.96	78.6	12.0	90.6	86.8	13.2			
50	Yes	4.26	1.30	73.9	16.3	90.2	81.9	18.1	2.02	35.11	17.3
50	No	4.25	1.33	73.8	16.6	90.3	81.7	18.3			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.8 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S3.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazotiation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 1-50 mM **TPABr**, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HBr] = 1 mM.<sup>a</sup>

[TPABr] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
1	Yes	4.84	0.70	83.9	8.7	92.6	90.6	9.4	1.06	44.62	42.0
1	No	4.92	0.73	85.3	9.1	94.4	90.4	9.6			
3	Yes	4.84	0.74	83.9	9.2	93.1	90.1	9.9	1.11	44.15	39.8
3	No	4.90	0.75	85.0	9.4	94.3	90.1	9.9			
5	Yes	4.68	0.77	81.1	9.6	90.7	89.4	10.6	1.19	43.34	36.4
5	No	4.77	0.80	82.7	10.0	92.7	89.2	10.8			
10	Yes	4.54	1.03	78.8	12.9	91.7	85.9	14.1	1.56	39.68	25.4
10	No	4.60	1.04	79.8	12.9	92.7	86.1	13.9			
15	Yes	4.41	1.25	76.6	15.7	92.2	83.0	17.0	1.88	36.51	19.4
15	No	4.43	1.25	76.8	15.6	92.3	83.2	16.8			
20	Yes	4.38	1.25	76.0	15.6	91.5	83.0	17.0	1.88	36.51	19.4
20	No	4.37	1.23	75.8	15.3	91.1	83.2	16.8			
30	Yes	4.40	1.24	76.4	15.5	91.9	83.1	16.9	1.87	36.62	19.6
30	No	4.40	1.23	76.4	15.4	91.7	83.2	16.8			
50	Yes	4.38	1.21	76.0	15.2	91.1	83.4	16.6	1.84	36.90	20.0
50	No	4.35	1.19	75.4	14.9	90.3	83.5	16.5			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.8 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S4.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 1-30 mM **TBABr**, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HBr] = 1 mM.<sup>a</sup>

[TBABr] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
1	Yes	5.16	0.75	82.3	8.7	91.0	90.5	9.5	1.06	44.61	42.0
1	No	5.28	0.77	84.2	8.8	93.0	90.5	9.5			
3	Yes	5.12	0.91	81.8	10.5	92.3	88.6	11.4	1.26	42.70	34.0
3	No	5.13	0.89	81.9	10.2	92.1	88.9	11.1			
5	Yes	4.90	1.13	78.2	13.0	91.2	85.7	14.3	1.58	39.55	25.1
5	No	5.04	1.14	80.4	13.1	93.4	86.0	14.0			
10	Yes	4.96	1.26	79.2	14.5	93.7	84.5	15.5	1.69	38.37	22.6
10	No	5.04	1.23	80.4	14.1	94.5	85.1	14.9			
15	Yes	3.45	2.50	55.3	28.8	84.1	65.7	34.3	3.83	17.25	4.5
15	No	3.52	2.61	56.3	30.0	86.3	65.2	34.8			
20	Yes	3.50	2.79	56.0	32.1	88.0	63.6	36.4	4.03	15.28	3.8
20	No	2.79	2.21	44.7	25.4	70.1	63.8	36.2			
30	Yes	3.35	2.67	53.6	30.7	84.3	63.6	36.4	4.07	14.89	3.7
30	No	2.01	1.65	32.4	18.9	51.3	63.1	36.9			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 6.3 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S5.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 1-50 mM **TriEABr**, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HBr] = 1 mM.<sup>a</sup>

[TriEABr] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
1	Yes	4.88	0.73	83.1	8.9	92.1	90.3	9.7	1.08	44.49	41.4
1	No	4.82	0.71	82.1	8.7	90.8	90.5	9.5			
5	Yes	4.80	0.78	81.9	9.5	91.4	89.6	10.4	1.17	43.60	37.4
5	No	4.83	0.78	82.3	9.6	91.9	89.6	10.4			
10	Yes	4.58	0.86	78.1	10.5	88.6	88.1	11.9	1.29	42.34	32.7
10	No	4.76	0.84	81.1	10.3	91.4	88.7	11.3			
13	Yes	4.40	0.93	75.1	11.4	86.4	86.8	13.2	1.43	41.03	28.8
13	No	4.56	0.90	77.7	11.0	88.7	87.6	12.4			
15	Yes	4.28	1.11	73.0	13.7	86.6	84.2	15.8	1.72	38.15	22.2
15	No	4.44	1.09	75.7	13.4	89.0	85.0	15.0			
20	Yes	4.24	1.29	72.4	15.8	88.2	82.1	17.9	1.99	35.43	17.8
20	No	4.25	1.29	72.5	15.8	88.3	82.1	17.9			
30	Yes	4.24	1.32	72.4	16.2	88.6	81.7	18.3	2.03	35.08	17.3
30	No	4.26	1.31	72.6	16.1	88.7	81.9	18.1			
50	Yes	4.13	1.30	70.5	16.0	86.5	81.5	18.5	2.06	34.75	16.9
50	No	4.21	1.34	71.8	16.4	88.2	81.4	18.6			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.9 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S6.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 5-50 mM NH<sub>4</sub>Br, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HBr] = 1 mM.<sup>a</sup>

[NH <sub>4</sub> Br] (mM)	Shaker <sup>b</sup>	Peak Areas <sup>c</sup> (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
5	Yes	4.83	0.75	81.7	9.1	90.8	89.9	10.1	1.11	44.19	40.0
5	No	4.99	0.75	84.3	9.1	93.3	90.3	9.7			
7	Yes	4.88	0.78	82.5	9.5	92.0	89.7	10.3	1.13	43.98	39.0
7	No	5.06	0.77	85.5	9.4	94.9	90.1	9.9			
9	Yes	4.85	0.80	82.0	9.8	91.8	89.3	10.7	1.17	43.54	37.2
9	No	4.95	0.79	83.7	9.6	93.3	89.7	10.3			
13	Yes	4.63	0.99	78.2	12.1	90.3	86.6	13.4	1.45	40.82	28.2
13	No	4.75	0.95	80.3	11.6	91.9	87.4	12.6			
15	Yes	4.46	1.10	75.4	13.4	88.9	84.9	15.1	1.65	38.77	23.4
15	No	4.61	1.09	77.9	13.3	91.2	85.4	14.6			
20	Yes	4.37	1.24	74.0	15.1	89.1	83.0	17.0	1.87	36.67	19.7
20	No	4.41	1.22	74.5	14.8	89.3	83.4	16.6			
30	Yes	4.35	1.26	73.5	15.4	88.9	82.7	17.3	1.89	36.46	19.3
30	No	4.36	1.21	73.7	14.7	88.3	83.4	16.6			
50	Yes	4.37	1.25	74.0	15.3	89.2	82.9	17.1	1.88	36.52	19.4
50	No	4.37	1.22	73.8	14.8	88.6	83.3	16.7			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.9 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.



**Table S7.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 5-50 mM LiCl, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HCl] = 1 mM.<sup>a</sup>

[LiCl] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
5	Yes	4.68	0.74	84.0	9.5	93.5	89.8	10.2	1.14	43.88	38.6
5	No	4.81	0.76	86.2	9.8	95.9	89.8	10.2			
7	Yes	4.67	0.74	83.8	9.6	93.4	89.7	10.3	1.15	43.71	37.8
7	No	4.82	0.78	86.4	10.0	96.4	89.6	10.4			
9	Yes	4.65	0.75	83.5	9.7	93.1	89.6	10.4	1.15	43.72	37.9
9	No	4.77	0.76	85.5	9.8	95.3	89.8	10.2			
13	Yes	4.67	0.77	83.8	9.9	93.7	89.4	10.6	1.17	43.51	37.1
13	No	4.72	0.76	84.6	9.8	94.4	89.6	10.4			
15	Yes	4.68	0.79	84.0	10.2	94.2	89.2	10.8	1.21	43.12	35.5
15	No	4.71	0.80	84.4	10.3	94.7	89.1	10.9			
20	Yes	4.64	0.82	83.2	10.5	93.7	88.7	11.3	1.26	42.70	34.0
20	No	4.66	0.82	83.6	10.6	94.2	88.8	11.2			
30	Yes	4.59	0.87	82.3	11.2	93.5	88.0	12.0	1.32	42.06	31.8
30	No	4.63	0.85	83.0	11.0	93.9	88.3	11.7			
50	Yes	4.14	1.22	74.2	15.8	90.0	82.5	17.5	1.94	35.95	18.5
50	No	4.22	1.23	75.6	15.8	91.5	82.7	17.3			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.6 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S8.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 5-50 mM NaCl, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HCl] = 1 mM.<sup>a</sup>

[NaCl] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
5	Yes	5.09	0.74	84.0	8.8	92.7	90.6	9.4	1.04	44.88	43.3
5	No	5.17	0.72	85.2	8.5	93.7	90.9	9.1			
7	Yes	5.08	0.78	83.7	9.3	93.0	90.0	10.0	1.12	44.07	39.4
7	No	5.21	0.80	85.9	9.5	95.5	90.0	10.0			
9	Yes	5.00	0.80	82.4	9.5	91.8	89.7	10.3	1.14	43.82	38.3
9	No	5.10	0.80	84.0	9.5	93.5	89.8	10.2			
15	Yes	4.72	1.02	77.8	12.2	89.9	86.5	13.5	1.49	40.42	27.2
15	No	4.78	1.01	78.9	12.0	90.8	86.8	13.2			
20	Yes	4.55	1.23	75.0	14.6	89.6	83.7	16.3	1.80	37.35	20.8
20	No	4.72	1.25	77.8	14.8	92.7	84.0	16.0			
30	Yes	4.45	1.27	73.4	15.0	88.5	83.0	17.0	1.89	36.45	19.3
30	No	4.41	1.25	72.7	14.8	87.5	83.1	16.9			
50	Yes	4.55	1.26	75.1	15.0	90.1	83.3	16.7	1.84	36.98	20.2
50	No	4.55	1.23	75.1	14.6	89.7	83.7	16.3			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 6.1 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S9.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 5-50 mM KCl, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HCl] = 1 mM.<sup>a</sup>

[KCl] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
5	Yes	4.49	0.71	83.0	9.4	92.4	89.8	10.2	1.14	43.90	38.7
5	No	4.57	0.72	84.5	9.5	94.1	89.9	10.1			
7	Yes	4.44	0.75	82.2	9.9	92.2	89.2	10.8	1.22	43.09	35.4
7	No	4.49	0.77	83.0	10.3	93.2	89.0	11.0			
9	Yes	4.28	0.97	79.2	12.9	92.1	86.0	14.0	1.57	39.63	25.3
9	No	4.31	0.99	79.8	13.1	92.9	85.9	14.1			
13	Yes	4.12	1.11	76.2	14.8	91.0	83.7	16.3	1.81	37.27	20.6
13	No	4.16	1.12	76.9	14.9	91.8	83.8	16.2			
15	Yes	4.08	1.12	75.6	14.9	90.5	83.6	16.4	1.82	37.11	20.4
15	No	4.09	1.11	75.7	14.7	90.4	83.7	16.3			
20	Yes	4.13	1.12	76.4	14.9	91.3	83.7	16.3	1.82	37.13	20.4
20	No	4.13	1.12	76.4	14.9	91.3	83.7	16.3			
30	Yes	4.12	1.11	76.3	14.8	91.2	83.7	16.3	1.81	37.23	20.6
30	No	4.16	1.12	77.0	14.9	91.9	83.8	16.2			
50	Yes	3.99	1.10	73.9	14.7	88.6	83.5	16.5	1.85	36.79	19.8
50	No	3.97	1.11	73.4	14.8	88.2	83.2	16.8			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.4 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S10.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 5-50 mM **RbCl**, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HCl] = 1 mM.<sup>a</sup>

[RbCl] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
5	Yes	4.64	0.76	85.9	10.1	96.0	89.5	10.5	1.18	43.44	36.8
5	No	4.57	0.76	84.4	10.1	94.5	89.4	10.6			
7	Yes	4.45	0.95	82.3	12.6	94.9	86.7	13.3	1.49	40.36	27.0
7	No	4.34	0.94	80.2	12.5	92.7	86.5	13.5			
9	Yes	4.23	1.06	78.2	14.1	92.3	84.7	15.3	1.71	38.18	22.3
9	No	4.19	1.07	77.5	14.3	91.8	84.5	15.5			
13	Yes	4.07	1.13	75.3	15.1	90.4	83.3	16.7	1.85	36.81	19.9
13	No	4.17	1.15	77.1	15.3	92.4	83.4	16.6			
15	Yes	4.08	1.12	75.6	15.0	90.6	83.5	16.5	1.82	37.12	20.4
15	No	4.16	1.12	76.9	14.9	91.8	83.8	16.2			
20	Yes	4.12	1.12	76.2	15.0	91.2	83.6	16.4	1.82	37.11	20.4
20	No	4.06	1.10	75.2	14.7	89.8	83.7	16.3			
30	Yes	4.11	1.13	76.0	15.0	91.0	83.5	16.5	1.82	37.11	20.4
30	No	4.07	1.10	75.3	14.6	89.8	83.8	16.2			
50	Yes	4.09	1.11	75.6	14.7	90.4	83.7	16.3	1.82	37.16	20.4
50	No	4.21	1.14	77.8	15.2	93.0	83.7	16.3			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.4 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S11.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 5-50 mM CsCl, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HCl] = 1 mM.<sup>a</sup>

[CsCl] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
5	Yes	4.80	0.79	86.1	10.2	96.3	89.4	10.6	1.18	43.42	36.7
5	No	4.79	0.79	85.8	10.2	96.0	89.4	10.6			
7	Yes	4.53	0.98	81.2	12.6	93.8	86.6	13.4	1.48	40.47	27.3
7	No	4.58	0.96	82.1	12.4	94.5	86.9	13.1			
9	Yes	4.48	1.12	80.4	14.5	94.9	84.7	15.3	1.70	38.32	22.6
9	No	4.48	1.12	80.3	14.4	94.7	84.8	15.2			
13	Yes	4.37	1.19	78.4	15.4	93.8	83.6	16.4	1.82	37.15	20.4
13	No	4.40	1.19	78.9	15.3	94.2	83.7	16.3			
15	Yes	4.28	1.18	76.8	15.2	92.0	83.4	16.6	1.83	37.05	20.3
15	No	4.37	1.18	78.4	15.2	93.6	83.7	16.3			
20	Yes	4.34	1.18	77.9	15.2	93.1	83.6	16.4	1.82	37.14	20.4
20	No	4.42	1.20	79.3	15.4	94.7	83.7	16.3			
30	Yes	4.36	1.16	78.2	14.9	93.2	84.0	16.0	1.78	37.49	21.0
30	No	4.33	1.15	77.7	14.8	92.5	84.0	16.0			
50	Yes	4.35	1.14	78.0	14.7	92.7	84.2	15.8	1.77	37.61	21.2
50	No	4.25	1.12	76.2	14.5	90.6	84.0	16.0			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.6 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S12.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 0.5-7.5 mM CaCl<sub>2</sub>, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HCl] = 1 mM.<sup>a</sup>

[CaCl <sub>2</sub> ] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
0.5	Yes	4.95	0.79	83.8	9.7	93.5	89.6	10.4	1.13	43.92	38.8
0.5	No	5.14	0.79	86.9	9.6	96.5	90.1	9.9			
1.5	Yes	4.70	1.10	79.7	13.5	93.1	85.5	14.5	1.60	39.32	24.6
1.5	No	4.82	1.11	81.6	13.5	95.2	85.8	14.2			
2.5	Yes	4.40	1.23	74.5	15.0	89.5	83.2	16.8	1.85	36.83	19.9
2.5	No	4.49	1.23	76.1	15.0	91.1	83.5	16.5			
3.5	Yes	4.52	1.31	76.6	15.9	92.5	82.8	17.2	1.91	36.24	19.0
3.5	No	4.49	1.29	76.1	15.7	91.7	82.9	17.1			
3.0	Yes	4.42	1.29	74.9	15.7	90.6	82.7	17.3	1.91	36.20	18.9
3.0	No	4.50	1.29	76.2	15.7	91.8	82.9	17.1			
5.5	Yes	4.31	1.26	73.0	15.3	88.3	82.6	17.4	1.93	36.01	18.6
5.5	No	4.66	1.36	78.9	16.6	95.5	82.6	17.4			
6.5	Yes	4.48	1.29	75.8	15.7	91.5	82.9	17.1	1.90	36.33	19.1
6.5	No	4.43	1.26	75.1	15.4	90.4	83.0	17.0			
7.5	Yes	4.35	1.23	73.7	15.0	88.7	83.0	17.0	1.89	36.40	19.2
7.5	No	4.24	1.21	71.8	14.8	86.5	82.9	17.1			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.9 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S13.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 0.5-7.5 mM MgCl<sub>2</sub>, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HCl] = 1 mM.<sup>a</sup>

[MgCl <sub>2</sub> ] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
0.5	Yes	5.49	0.86	79.9	9.1	89.0	89.8	10.2	1.12	44.05	39.3
0.5	No	5.58	0.85	81.2	8.9	90.1	90.1	9.9			
1.5	Yes	5.17	1.13	75.4	11.9	87.3	86.4	13.6	1.52	40.13	26.5
1.5	No	5.30	1.16	77.2	12.1	89.4	86.4	13.6			
2.5	Yes	4.97	1.37	72.4	14.4	86.8	83.4	16.6	1.83	37.02	20.2
2.5	No	5.05	1.37	73.5	14.3	87.9	83.7	16.3			
3.5	Yes	5.01	1.50	73.0	15.7	88.7	82.3	17.7	1.96	35.70	18.2
3.5	No	5.00	1.48	72.9	15.5	88.4	82.4	17.6			
3.0	Yes	4.95	1.51	72.1	15.9	88.0	82.0	18.0	2.00	35.35	17.7
3.0	No	5.10	1.55	74.3	16.2	90.6	82.1	17.9			
5.5	Yes	4.93	1.53	71.9	16.1	88.0	81.7	18.3	2.02	35.18	17.4
5.5	No	5.71	1.74	83.1	18.2	101	82.0	18.0			
6.5	Yes	4.92	1.53	71.7	16.0	87.7	81.7	18.3	2.02	35.14	17.4
6.5	No	5.49	1.68	79.9	17.6	97.5	82.0	18.0			
7.5	Yes	4.94	1.52	72.0	15.9	87.9	81.9	18.1	2.01	35.21	17.5
7.5	No	5.41	1.66	78.7	17.4	96.1	81.9	18.1			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 6.9 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S14.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the presence of 2.5-7.5 mM ZnCl<sub>2</sub>, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HCl] = 1 mM.<sup>a</sup>

[ZnCl <sub>2</sub> ] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
2.5	Yes	4.64	1.31	71.9	14.6	86.5	83.1	16.9	1.86	36.73	19.7
2.5	No	4.68	1.29	72.4	14.3	86.7	83.5	16.5			
3.0	Yes	4.56	1.41	70.6	15.7	86.3	81.8	18.2	2.01	35.23	17.5
3.0	No	4.66	1.42	72.1	15.8	88.0	82.0	18.0			
7.5	Yes	4.52	1.41	69.9	15.7	85.6	81.6	18.4	2.02	35.12	17.4
7.5	No	4.53	1.38	70.1	15.4	85.4	82.0	18.0			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 6.5 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.



**Table S15.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the presence of 0.3-3 mM AlCl<sub>3</sub>, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HCl] = 1 mM.<sup>a</sup>

[AlCl <sub>3</sub> ] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
0.3	Yes	4.87	0.85	82.5	10.4	92.9	88.8	11.2	1.25	42.78	34.3
0.3	No	5.03	0.88	85.1	10.7	95.8	88.9	11.1			
1.0	Yes	5.54	1.13	85.7	12.6	98.3	87.2	12.8	1.39	41.41	29.9
1.0	No	5.96	1.14	92.1	12.7	104.8	87.9	12.1			
1.7	Yes	4.71	1.24	79.7	15.1	94.8	84.1	15.9	1.69	38.45	22.8
1.7	No	5.37	1.25	90.9	15.2	106.1	85.7	14.3			
2.3	Yes	4.41	1.31	74.8	15.9	90.7	82.4	17.6	1.95	35.89	18.4
2.3	No	4.38	1.28	74.2	15.6	89.8	82.6	17.4			
3.0	Yes	4.04	1.29	68.6	15.7	84.3	81.3	18.7	2.02	35.16	17.4
3.0	No	4.36	1.30	73.9	15.8	89.7	82.4	17.6			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.9 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S16.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 5-50 mM **NaBr**, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HBr] = 1 mM.<sup>a</sup>

[NaBr] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
5	Yes	5.04	0.76	86.0	9.4	95.3	90.2	9.8	1.10	44.21	40.0
5	No	5.04	0.77	85.9	9.5	95.4	90.1	9.9			
7	Yes	5.00	0.78	85.3	9.6	94.9	89.9	10.1	1.12	44.08	39.5
7	No	5.03	0.77	85.7	9.4	95.0	90.1	9.9			
9	Yes	4.94	0.78	84.3	9.6	93.9	89.8	10.2	1.14	43.82	38.3
9	No	5.01	0.79	85.4	9.7	95.1	89.8	10.2			
13	Yes	4.79	0.83	81.7	10.2	91.9	88.9	11.1	1.24	42.83	34.4
13	No	4.90	0.86	83.5	10.5	94.0	88.8	11.2			
15	Yes	4.65	0.93	79.2	11.4	90.6	87.4	12.6	1.41	41.22	29.3
15	No	4.73	0.95	80.6	11.7	92.2	87.4	12.6			
20	Yes	4.47	1.17	76.2	14.4	90.6	84.1	15.9	1.75	37.82	21.6
20	No	4.53	1.16	77.3	14.2	91.5	84.4	15.6			
30	Yes	4.29	1.22	73.2	15.0	88.2	83.0	17.0	1.90	36.30	19.1
30	No	4.32	1.25	73.7	15.3	89.0	82.8	17.2			
50	Yes	4.42	1.20	75.5	14.7	90.2	83.7	16.3	1.84	36.95	20.1
50	No	4.47	1.25	76.2	15.3	91.5	83.3	16.7			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.9 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S17.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 5-50 mM NaSCN, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HBr] = 1 mM.<sup>a</sup>

[NaSCN] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
5	Yes	5.35	0.82	85.6	9.5	95.1	90.1	9.9	1.10	44.28	40.4
5	No	5.44	0.81	87.0	9.3	96.3	90.3	9.7			
7	Yes	5.30	0.82	84.8	9.5	94.3	89.9	10.1	1.11	44.11	39.6
7	No	5.40	0.82	86.5	9.5	96.0	90.1	9.9			
9	Yes	5.26	0.83	84.3	9.6	93.8	89.8	10.2	1.14	43.87	38.5
9	No	5.31	0.83	85.1	9.6	94.7	89.8	10.2			
13	Yes	5.05	0.93	80.8	10.7	91.5	88.3	11.7	1.30	42.27	32.5
13	No	5.19	0.95	83.2	10.9	94.1	88.4	11.6			
15	Yes	4.96	1.10	79.4	12.7	92.1	86.3	13.7	1.52	40.12	26.4
15	No	5.10	1.10	81.7	12.7	94.4	86.5	13.5			
20	Yes	4.74	1.29	75.9	14.8	90.7	83.6	16.4	1.82	37.17	20.5
20	No	4.81	1.30	77.1	15.0	92.1	83.7	16.3			
30	Yes	4.63	1.32	74.2	15.2	89.5	83.0	17.0	1.88	36.53	19.4
30	No	4.76	1.33	76.2	15.3	91.5	83.2	16.8			
50	Yes	4.75	1.32	76.0	15.2	91.2	83.3	16.7	1.86	36.78	19.8
50	No	4.75	1.32	76.1	15.2	91.3	83.3	16.7			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 6.3 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S18.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 5-50 mM sodium benzoate **NaBenz**, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HBr] = 1 mM.<sup>a</sup>

[NaBenz] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
5	Yes	5.06	0.76	82.3	9.0	91.3	90.2	9.8	1.09	44.32	40.6
5	No	5.25	0.79	85.3	9.2	94.5	90.3	9.7			
7	Yes	4.99	0.77	81.2	9.0	90.2	90.0	10.0	1.12	44.09	39.5
7	No	5.19	0.80	84.4	9.3	93.7	90.0	10.0			
9	Yes	4.94	0.78	80.5	9.1	89.6	89.8	10.2	1.13	43.94	38.8
9	No	5.15	0.80	83.7	9.4	93.1	89.9	10.1			
13	Yes	4.90	0.82	79.8	9.6	89.3	89.3	10.7	1.18	43.45	36.8
13	No	5.12	0.83	83.2	9.7	92.9	89.6	10.4			
15	Yes	4.71	0.92	76.6	10.7	87.4	87.7	12.3	1.38	41.48	30.1
15	No	4.90	0.97	79.7	11.3	91.0	87.6	12.4			
20	Yes	4.47	1.14	72.8	13.3	86.2	84.5	15.5	1.73	38.00	21.9
20	No	4.59	1.18	74.7	13.8	88.5	84.4	15.6			
30	Yes	4.46	1.26	72.6	14.7	87.4	83.1	16.9	1.87	36.60	19.5
30	No	4.52	1.27	73.5	14.8	88.4	83.2	16.8			
50	Yes	4.52	1.22	73.7	14.3	88.0	83.7	16.3	1.82	37.16	20.5
50	No	4.58	1.24	74.5	14.5	89.0	83.7	16.3			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 6.2 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

**Table S19.** HPLC average peak areas, observed and normalized (subscript N) product yields for dediazonation reaction of 16-ArN<sub>2</sub><sup>+</sup> in solutions of 15 mM AOT in the present of 5-50 mM sodium salicylate **NaSal**, estimated interfacial molarities of headgroup, AOT<sub>m</sub>, water, H<sub>2</sub>O<sub>m</sub>, and their molar ratio, H<sub>2</sub>O<sub>m</sub>/AOT<sub>m</sub>, at 28°C. [HBr] = 1 mM.<sup>a</sup>

[NaSal] (mM)	Shaker <sup>b</sup>	Peak Areas (10 <sup>6</sup> μv•s) <sup>c</sup>		Observed Yields (%)			Normalized Yields (%) <sup>d</sup>		AOT <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> <sup>e</sup> (M)	H <sub>2</sub> O <sub>m</sub> /AOT <sub>m</sub> <sup>e</sup>
		16-ArOH	16-ArS	16-ArOH	16-ArS	Total	16-ArOH <sub>N</sub>	16-ArS <sub>N</sub>			
5	Yes	4.36	0.67	79.9	8.8	88.7	90.0	10.0	1.09	44.31	40.5
5	No	4.51	0.67	82.6	8.8	91.4	90.4	9.6			
7	Yes	4.02	0.62	73.7	8.2	81.9	90.0	10.0	1.11	44.18	39.9
7	No	4.47	0.67	81.9	8.9	90.7	90.2	9.8			
9	Yes	4.22	0.67	77.2	8.9	86.1	89.7	10.3	1.13	44.00	39.1
9	No	4.42	0.67	80.9	8.8	89.8	90.2	9.8			
13	Yes	4.20	0.70	76.9	9.3	86.2	89.2	10.8	1.19	43.33	36.3
13	No	4.35	0.72	79.6	9.4	89.1	89.4	10.6			
15	Yes	4.12	0.82	75.5	10.8	86.2	87.5	12.5	1.40	41.31	29.6
15	No	4.21	0.84	77.0	11.1	88.1	87.4	12.6			
20	Yes	3.88	1.02	71.1	13.5	84.6	84.1	15.9	1.77	37.67	21.3
20	No	4.01	1.04	73.4	13.7	87.2	84.2	15.8			
30	Yes	3.76	1.08	68.9	14.2	83.1	82.9	17.1	1.89	36.42	19.2
30	No	3.80	1.08	69.6	14.2	83.8	83.1	16.9			
50	Yes	3.86	1.08	70.6	14.2	84.9	83.2	16.8	1.86	36.71	19.7
50	No	3.81	1.06	69.7	14.0	83.7	83.3	16.7			

a. Reaction time ca. 48 hours. Prior to HPLC analysis, the product mixture was mixed with an equal volume of water. The final concentrations of 16-ArN<sub>2</sub><sup>+</sup> were ca. 5.5 x 10<sup>-5</sup> M.

b. The reaction tubes were either placed in a thermostated shaker or water bath.

c. 100 μL sample injections. Peak areas are average of triplicate or duplicate injections. Eluting solvents: 65%MeOH/35%*i*-PrOH; Flow rate: 0.4 mL/min; Detector wavelength: 220 nm.

d. % 16-ArS<sub>N</sub> = 100 (%16-ArS)/(%16-ArOH + %16-ArS); % 16-ArOH<sub>N</sub> = 100 (%16-ArOH)/(%16-ArOH + %16-ArS).

e. For each solution composition, the estimated AOT<sub>m</sub> and H<sub>2</sub>O<sub>m</sub> values were average results calculated from both experiments carried out with or without shaker.

## References:

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