Supporting Information

Consequences of Convex Nanopore Chemistry on Confined Water Dynamics

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Experimental

Materials and Methods. The perdeuterated reagents 1-bromononane-d_{19} (98% D) and tetramethylammonium hydroxide ($N(CD_3)_4OD•5D_2O$, 98% D) were purchased from CDN Isotopes (Quebec, Canada) and Cambridge Isotopes, respectively. High-purity D_2O (99.9% D) was purchased from Sigma-Aldrich. All other materials and reagent grade solvents were purchased from Sigma-Aldrich (Milwaukee, WI) and used as received unless otherwise noted. Type I ultra-pure water was obtained from a Thermo Scientific Barnstead NANOpure^{TM} system (18.2 MΩ resistance) and sparged with $N_2(g)$ prior to use. High purity niobium foil (0.025 ± 0.015 mm thick, 99.8%) was purchased from Alfa Aesar.

A Bruker Avance III HD AM-400 with a BBO SmartProbe was used to record ^2H NMR spectra and ^{13}C NMR spectra were recorded on a Bruker Avance III HD 500 MHz spectrometer with a TCI cryoprobe at the University of Minnesota-Twin Cities housed in the LeClaire-Dow Instrumentation Facility. The ^2H spectra were recorded in the CH_3OH and referenced relative to the residual deuterated solvent peak while the ^{13}C spectra were recorded in CD_3OD and referenced to the solvent peak. Atlantic Microlab, Inc. (Norcross, GA, USA) performed elemental combustion analyses (C, H, N, and S).

d*$_{19}$-1-Nonanesulfonic acid (SO_3Hd).** We adapted a synthetic procedure previously used by Jackson *et al*.[1] to prepare non-deuterated sulfonate amphiphiles. 1-bromononane-d_{19} (6.0 g, 27 mmol) was mixed with ultrapure water (46 mL), anhydrous ethanol (30 mL), and Na_2SO_3 (5.0 g, 40 mmol) and refluxed for 48 h in a 250 mL round bottom flask equipped with a reflux condenser. All volatiles were removed under vacuum and the resulting solid was resuspended in methanol (~50 mL) and stirred for 1 h at 50 °C. The solid byproduct was removed by filtration and all volatiles in the filtrate were removed under vacuum. The resulting white solid was triturated with diethyl ether to remove residual starting material, dried under vacuum, and recrystallized from water. This synthetic intermediate was suspended in diethyl ether and acidified using excess HCl(*g*). ***Caution*: This reaction generates corrosive HCl(g) by adding concentrated HCl (50 mL) to concentrated H_2SO_4 (50 mL), a procedure that should be performed in a fumehood with appropriate personal protective equipment, including nitrile gloves to prevent skin exposure.* The sealed reaction flask was allowed to stir overnight. The mixture was sparged with $N_2(g)$ for 15 min to remove residual HCl(*g*) and all volatiles were removed under vacuum. The product was freeze-dried from benzene three times to yield an off-

white, crystalline powder. Yield: 70% (4.2 g, 18.5 mmol). ^{2}H NMR (61 MHz, CH_3OH) δ 2.75 (CD_2-SO_3H, s, 2H), 1.72 (CD_2, s, 2H), 1.34 (CD_2, s, 2H), 1.24 (CD_2, s, 9H), 0.84 (CD_3, s, 3H). ^{13}C NMR (126 MHz, CD_3OD) δ 51.86 (CD_2-SO_3H, p), 31.63 (CD_2, p), 29.20 (CD_2, m), 28.56 (CD_2, m), 24.88 (CD_2, p), 22.39 (CD_2, p), 13.27 (CD_3, sept). The dried powder was evenly divided to prepare two samples: a QENS sample hydrated using H_2O and a background sample hydrated with D_2O. To minimize the excess incoherent scattering due to residual H_2O, we azeotropically freeze-dried the background sample from D_2O. Elemental analysis, wherein deuterium is analyzed as hydrogen, was used to determine sample purity and estimate the residual water content of surfactant hydrates. *Anal.* Calc.: $C_9D_{19}SO_3H$•$0.4H_2O$: C, 46.07; H, 8.95; S, 13.67; Found: C, 46.08; H, 9.20; S, 14.06. For the background sample: *Anal.* Calc.: $C_9D_{19}SO_3H$•0.72 D_2O: C, 44.96; H, 9.00; S, 13.34; Found: C, 44.96; H, 8.91; S, 13.74.

General Procedure for Deuterated Sulfonate Salt Synthesis. In a 250 mL round bottom flask, 1-nonanesulfonic acid (**SO_3Hd**) was dissolved in methanol (~0.1 M). A stoichiometric equivalent of K_2CO_3 or tetramethylammonium–d_{12} hydroxide ($N(CD_3)_4OH$) aqueous solution (0.03365 M) was added and the mixtures were stirred at ambient temperature for 12 h. All volatiles were removed *in vacuo* and residual water and methanol were azeotropically distilled by freeze-drying from benzene three times under vacuum. All yields were quantitative. Analogous to our treatment of **SO_3Hd**, the dried powders of **SO_3Kd** and **SO_3NMe_4d** were divided in half and used to prepare a QENS sample with H_2O and a background sample hydrated with D_2O.

Potassium 1-nonanesulfonate-d_{19} (SO_3Kd). ^{2}H NMR (61 MHz, CH_3OH) δ 2.74 (CD_2-SO_3H, s, 2H), 1.71 (CD_2, s, 2H), 1.34 (CD_2, s, 2H), 1.24 (CD_2, s, 9H), 0.83 (CD_3, s, 3H). ^{13}C NMR (126 MHz, CD_3OD) δ 51.86 (CD_2-SO_3H, p), 31.64 (CD_2, p), 29.06 (CD_2, m), 28.56 (CD_2, m), 24.88 (CD_2, p), 22.39 (CD_2, p), 13.27 (CD_3, sept). Anal. Calc.: $C_9D_{19}SO_3K$•$0.2H_2O$: C, 40.16; H, 7.26; S, 11.91. Found: C, 40.08; H, 7.11; S, 11.96. For the background sample: Anal. Calc.: $C_9D_{19}SO_3K$•0.2 D_2O: C, 40.20; H, 7.26; S, 11.92; Found: C, 40.20; H, 7.14; S, 12.03.

Tetramethylammonium 1-nonanesulfonate-d_{31} (SO_3NMe_4d). ^{2}H NMR (61 MHz, CH_3OH) δ 3.15 (($CD)_4N^+$, s, 11H), 2.74 (CD_2-SO_3H, s, 2H), 1.71 (CD_2, s, 2H), 1.34 (CD_2, s, 2H), 1.24 (CD_2, s, 9H), 0.83 (CD_3, s, 3H). ^{13}C NMR (126 MHz, CD_3OD) δ 54.76 (($CD)_4N^+$, sept) 51.93 (CD_2-SO_3H, p), 31.62 (CD_2, p), 29.05 (CD_2, m), 28.54 (CD_2, m), 24.91 (CD_2, p), 22.38 (CD_2, p), 13.28 (CD_3, sept). *Anal.* Calc.: $C_{13}D_{31}SO_3N$•$0.38H_2O$: C, 48.87; H, 10.01; S, 10.03; N, 4.38.

Found: C, 48.47; H, 9.69; S, 10.53; N, 3.99. For the background sample: *Anal.* Calc.: $C_{13}D_{31}SO_3N$•0 D_2O: C, 49.94; H, 9.98; S, 10.25; N, 4.48. Found: C, 49.91; H, 9.86; S, 10.49; N, 4.24.

Lab Source Small-angle X-ray Scattering (SAXS). LLC samples were characterized via laboratory and synchrotron X-ray scattering. The Bruker D8 Discover X-ray diffractometer housed in the University of Minnesota-Twin Cities Characterization Facility uses a Cu-K_α point source filtered with a graphite monochromator, which is collimated to a spot size of 0.5 mm. The sample-to-detector distance was 30.00 cm and two-dimensional XRD patterns were collected using a 14 cm diameter Vantec 500 area detector. All patterns were calibrated using a silver behenate standard (d_{100} = 58.38 Å) and samples were typically exposed for 15 min. LLC samples were sealed within a custom-built aluminum sample cell with polyimide (Kapton®) windows. Conditions for synchrotron SAXS analyses are described in the main text.

Table S1. List of the observed and calculated peak positions for a normal ribbon phase (R_I) of **SO_3Kd** at w_0 = 6 at 25 °C using the centered rectangular plane group *C2mm* (space Group #38) and unit cell parameters a = 28.50 Å and b = 86.57 Å. For a centered rectangular lattice, the scattering wavevector modulus $|q|$ is ca

$$q^2 = 4\pi^2\left[\left(\frac{h}{a}\right)^2 + \left(\frac{k}{b}\right)^2\right]$$

where $h + k = 2n$ for the selection rules.

Miller Index (***h k***)	q_{obs} (Å^{-1})	q_{calc} (Å^{-1})	**Residual** $100 \bullet \Delta q/q_{obs}$
(02)	0.1445	0.1452	-0.23
(11)	0.2316	0.2321	0.22
(04)	0.2900	0.2903	0.11
(13)	0.3095	0.3099	0.12
(15)	0.4243	0.4246	0.07
(60)	0.4345	0.4355	0.22
(02)	0.4407	0.4409	0.05
(22)	0.4642	0.4642	0.00
(31)	0.6651	0.6654	0.04
(33)	0.6959	0.6963	0.06

Electron Density Map Reconstructions to Estimate Aqueous Channel Diameter and Counterion Distributions. From the synchrotron 2D-SAXS data, we generated azimuthally-integrated $I(2\theta)$ *v.* 2θ profiles and, in some cases, scaled the unit-cell size by factors of 1/2 or 1/10 to maximize computational efficiency. Using a fifth-order polynomial fit for the X-ray background, we used the *JANA2006*[4] software program to conduct a Le Bail refinement and thus extract the structure factor intensity ($|F_h|$) for each SAXS peak in the scaled 1D SAXS profile. We were able to adequately model all peaks in these scattering traces, as indicated by goodness of fit (GOF) parameters < 5 for all samples (Table S2). The $|F_h|$ value obtained from this refinement procedure were used as inputs for the charge-flipping algorithm *SUPERFLIP*.[5, 6] These input files are given in the following sections. Starting from randomized initial conditions, the *SUPERFLIP* program yielded > 20 independent convergences that were averaged to generate the final electron density maps. The agreement factors for the electron density maps are AF < 13, indicating acceptable fits. The VESTA software package[7] was used to visualize these electron density maps and to produce 1D electron density line profiles. The aqueous channel diameter was estimated using a ratiometric analysis of the number of pixels between adjacent hydrophobic interfaces at the negative 90% isosurface level in relation to the known unit-cell dimensions using Adobe Illustrator.

Table S2. Electron Density Map Figures-of-Merit

System	LLC Phase	*d*-spacing (Å)	Scaling Factor for *JANA2006*	Approximate Scaled 2θ Range (°)	GOF	Converged runs	AF
SO_3Hd w_0 = 6.5	G_I	71.2	10	10-50	0.48	32	12.63
SO_3Hd w_0 = 15	H_I	37.8	1	0.5-5.0	0.31	82	3.59
SO_3Kd w_0 = 6	R_I	a = 28.5 b = 86.6	1	0.5-6.0	0.30	21	0.00
SO_3Kd w_0 = 15	H_I	36.7	1	1.0 - 3.6	4.74	692	0.00
SO_3NMe_4d w_0 = 6	$H_I + L_\alpha$	23.3 (L_α) 33.0 (H_I)	1	1.5-6.0	0.17	8862	0.00
SO_3NMe_4d w_0 = 12	A15	73.0	2	1.5-8.0	0.31	23	2.76
SO_3NMe_4d w_0 = 15	A15	74.2	2	1.5-8.0	0.15	55	1.92

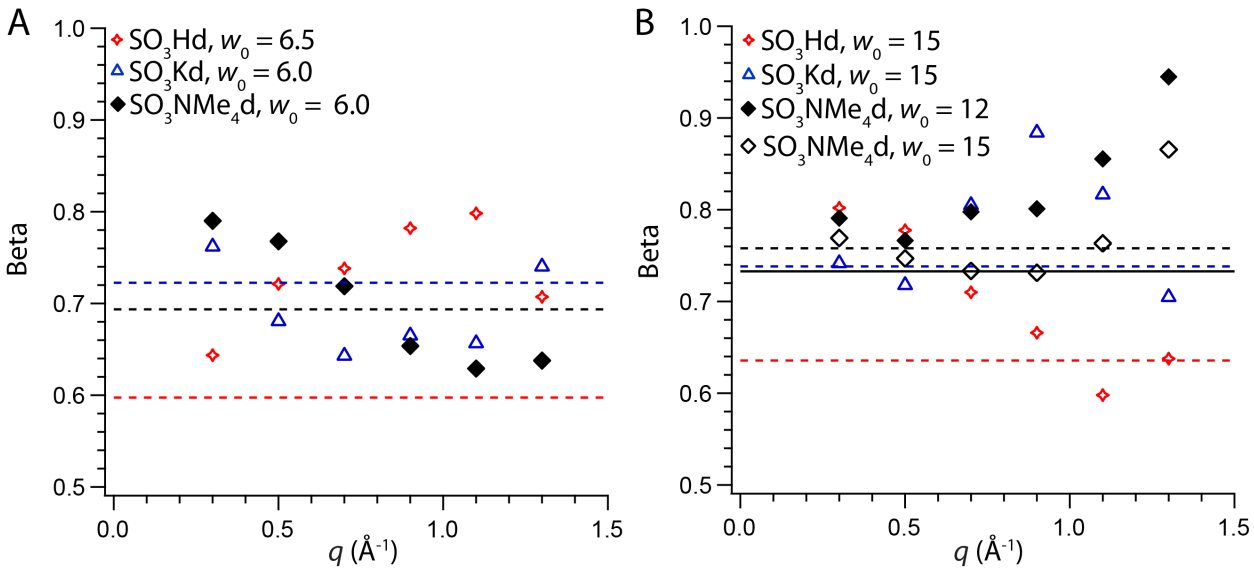

Figure S1. (*A*) Plot of β versus q at $w_0 \approx 6$ comparing a fit to Eq. (1) in which β was either allowed to vary with q (*markers*) or simultaneously optimized over the q-range 0.2-1.4 Å^{-1} (*straight lines*). The estimated error in these values from the Mantid program is smaller than the size of the markers. (*B*) Analogous plot for LLC samples at higher w_0. For **SO_3NMe_4d**, the solid black line is for $w_0 = 12$ and dashed black line for $w_0 = 15$. In both (*A*) and (*B*), the fitted values for β (q) do not show a strong q-dependence as demonstrated by their < 12% relative standard deviations (*i.e.* $100 \cdot \sigma_\beta / \beta_{avg} < 12\%$).

Table S3. Representative QENS fitting parameters from Eq. (1) and R^2 values from Figure 5

System	Hydration	Q (Å^{-1})	b_w	τ (ps)	β	$\langle\tau_\beta\rangle$ (ps)	χ^2	R^2
SO_3Hd	$w_0 = 6.5$	0.3	0.79	784	0.597	1190	4.04	0.97
		0.5	0.79	153	0.597	231		
		0.7	0.79	32.9	0.597	49.8		
		0.9	0.79	19.1	0.597	28.9		
		1.1	0.79	12.5	0.597	18.9		
		1.3	0.79	8.56	0.597	13.0		
SO_3Hd	$w_0 = 15$	0.3	1.07	158	0.636	221	1.00	0.97
		0.5	1.07	39.8	0.636	55.8		
		0.7	1.07	17.8	0.636	24.9		
		0.9	1.07	12.5	0.636	17.5		
		1.1	1.07	6.51	0.636	9.12		
		1.3	1.07	2.16	0.636	3.03		
SO_3NMe_4d	$w_0 = 6$	0.3	0.98	426	0.694	544	0.84	0.99
		0.5	0.98	138	0.694	176		
		0.7	0.98	69.9	0.694	89.3		
		0.9	0.98	50.4	0.694	64.4		
		1.1	0.98	33.6	0.694	42.9		
		1.3	0.98	18.5	0.694	23.6		
SO_3NMe_4d	$w_0 = 12$	0.3	0.98	184	0.733	223	1.42	1.00
		0.5	0.98	56.4	0.733	68.5		
		0.7	0.98	31.0	0.733	37.6		
		0.9	0.98	19.9	0.733	21.2		
		1.1	0.98	13.4	0.733	16.3		
		1.3	0.98	8.01	0.733	9.73		
SO_3NMe_4d	$w_0 = 15$	0.3	0.95	124	0.758	147	1.13	1.00
		0.5	0.95	44.7	0.758	52.8		
		0.7	0.95	23.9	0.758	28.2		
		0.9	0.95	16.1	0.758	19.0		
		1.1	0.95	10.6	0.758	12.5		
		1.3	0.95	6.32	0.758	7.46		
SO_3K	$w_0 = 6$	0.3	1.00	172	0.723	211.7	1.07	0.99
		0.5	1.00	57.8	0.723	71.1		
		0.7	1.00	26.9	0.723	33.1		
		0.9	1.00	17.7	0.723	21.8		
		1.1	1.00	12.5	0.723	15.4		
		1.3	1.00	7.29	0.723	8.96		
SO_3K	$w_0 = 15$	0.3	0.87	81.7	0.738	98.6	0.87	0.99
		0.5	0.87	27.7	0.738	33.4		
		0.7	0.87	13.5	0.738	16.3		
		0.9	0.87	9.06	0.738	10.9		
		1.1	0.87	6.55	0.738	7.90		
		1.3	0.87	4.86	0.738	5.86		

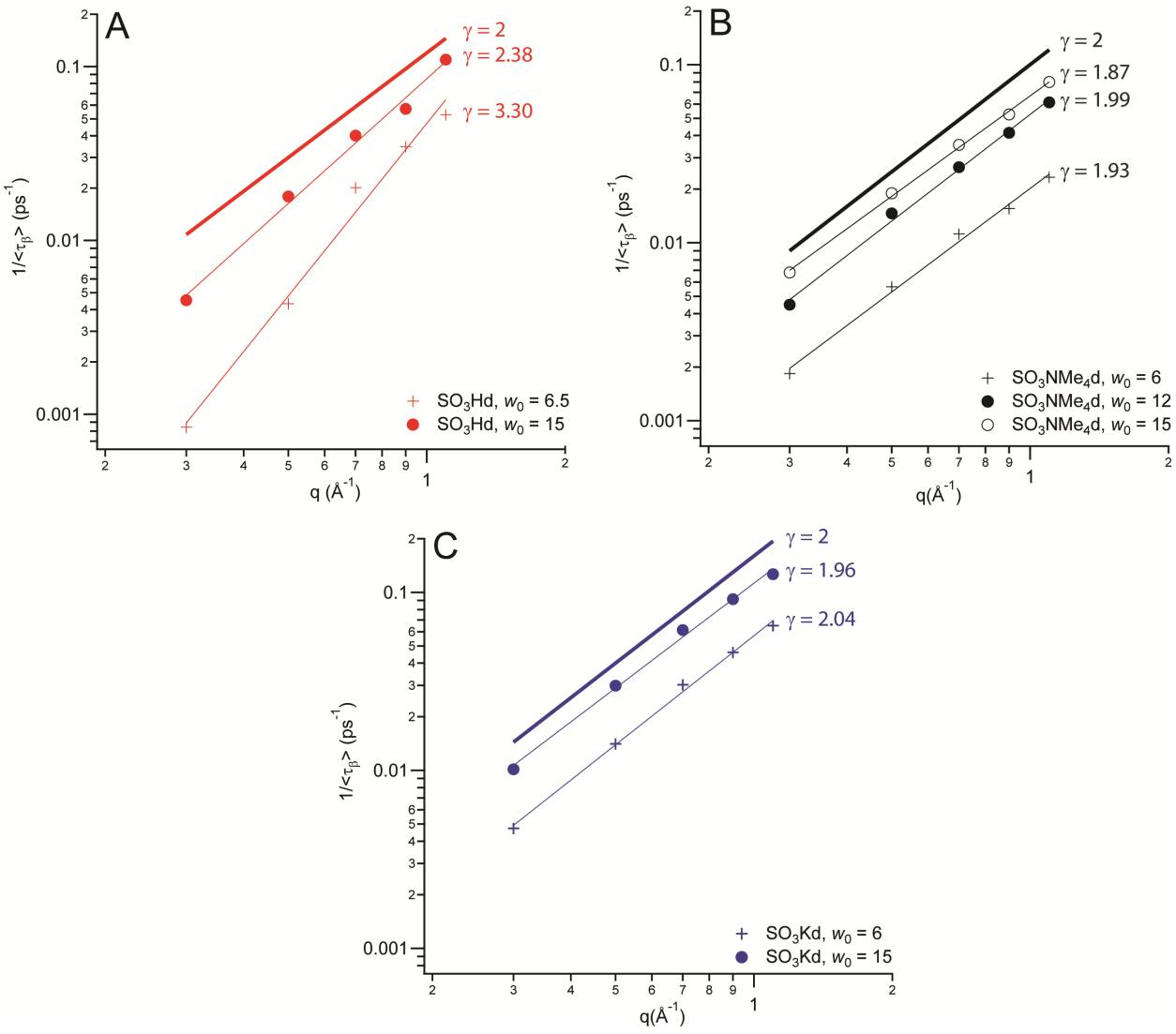

Figure S2. Average relaxation rate ($1/\langle\tau_\beta\rangle$) plotted against q on a log-log plot for (*A*) **SO_3Hd**, (*B*) **SO_3NMe$_4$d**, and (*C*) **SO_3Kd**. The scaling exponents (γ) and power law fits for $1/\langle\tau_\beta\rangle \sim q^\gamma$ are shown for each LLC. For most systems $\gamma \sim 2$ (*bold lines*), indicating diffusive translational water dynamics. We attribute the increased slope for **SO_3Hd** to increased elastic intensity from the LLC microstructure at low q (see Figure 2 in main text for SAXS data) that is not completely captured during our QENS fitting protocol, resulting in artificially lower values for $1/\langle\tau_\beta\rangle$.

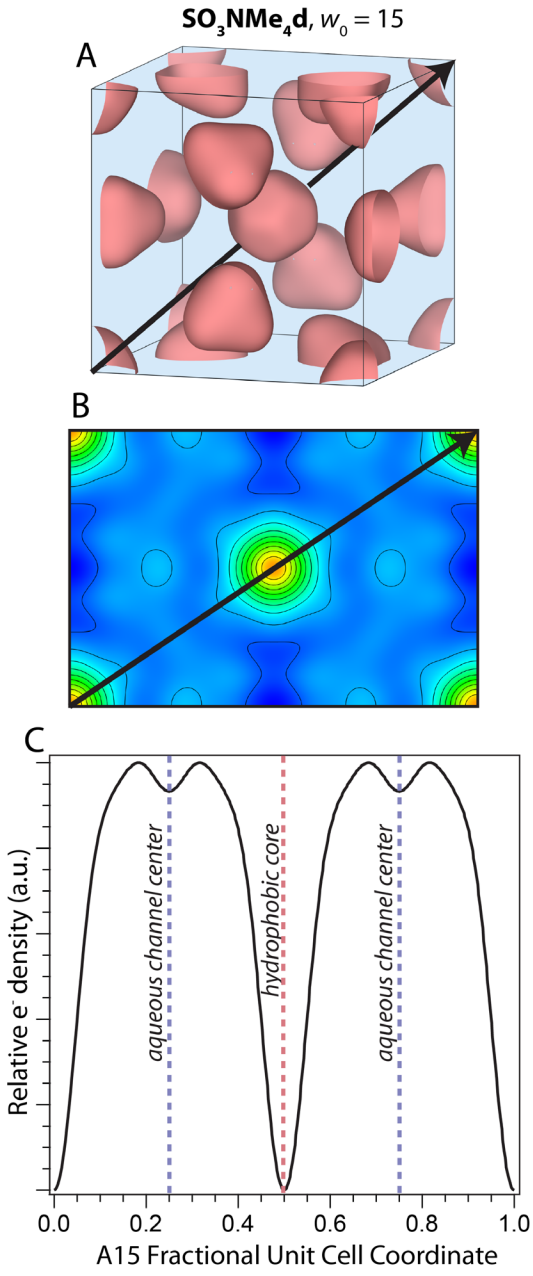

Figure S3. Electron density reconstruction of the **SO_3NMe_4d** A15 phase at w_0 = 15. (*A*) The 90% electron density isosurface reveals polyhedral micelles (*pink*) in a water matrix (*blue*). (*B*) 2D electron density map of the (110) plane from (*A*) with lines demarcating 10% increments in the relative electron density. (*C*) Linear electron density profile shown as relative electron density *versus* fractional unit cell coordinate along the [111]-direction (*black line in B*). Consistent with the A15 phase formed at w_0 = 12, the depleted electron density at the center of the aqueous channel arises from NMe_4^+ ion localization.

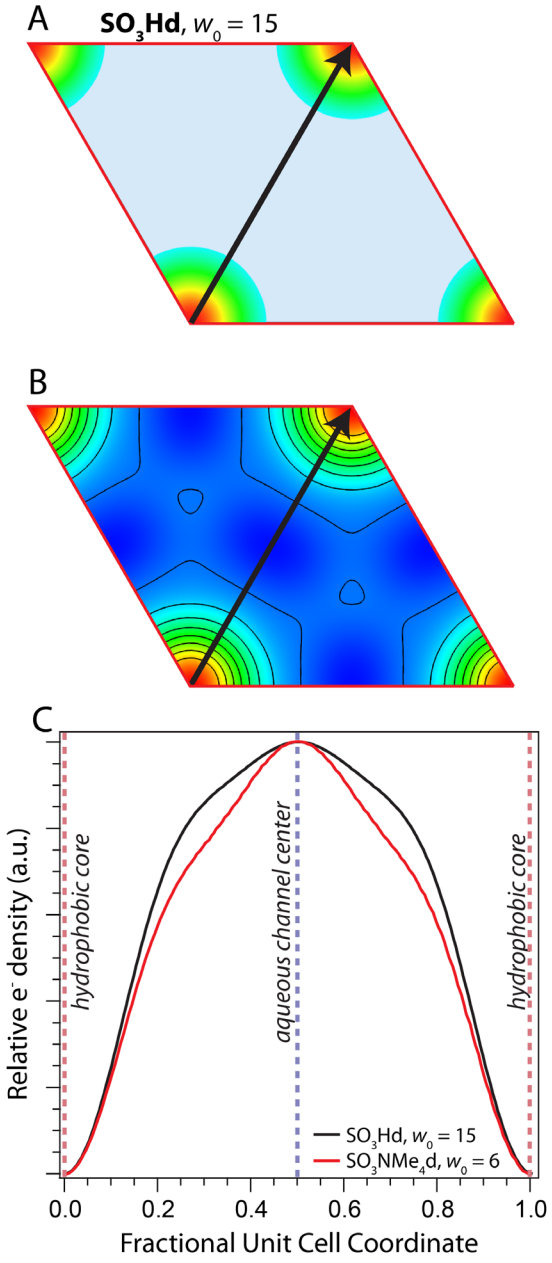

Figure S4. Electron density reconstruction of the **SO_3Hd** H_I phase at w_0 = 15. (*A*) A view of the (01) plane with the micellar interface representing the 90% isosurface. (*B*) 2D electron density map of (*A*) with black lines denoting 10% increments in the relative electron density. (*C*) The relative electron density profile along the [11] direction *versus* fractional H_I unit cell coordinate for **SO_3H** at w_0 = 15 (*black trace, arrow in B*) and **SO_3NMe_4d** at w_0 = 6 (*red trace, main text Figure 8*). The somewhat steeper decline in electron density for **SO_3NMe_4d** upon moving away from the aqueous channel center arises from NMe_4^+ ion localization near the micellar interface.

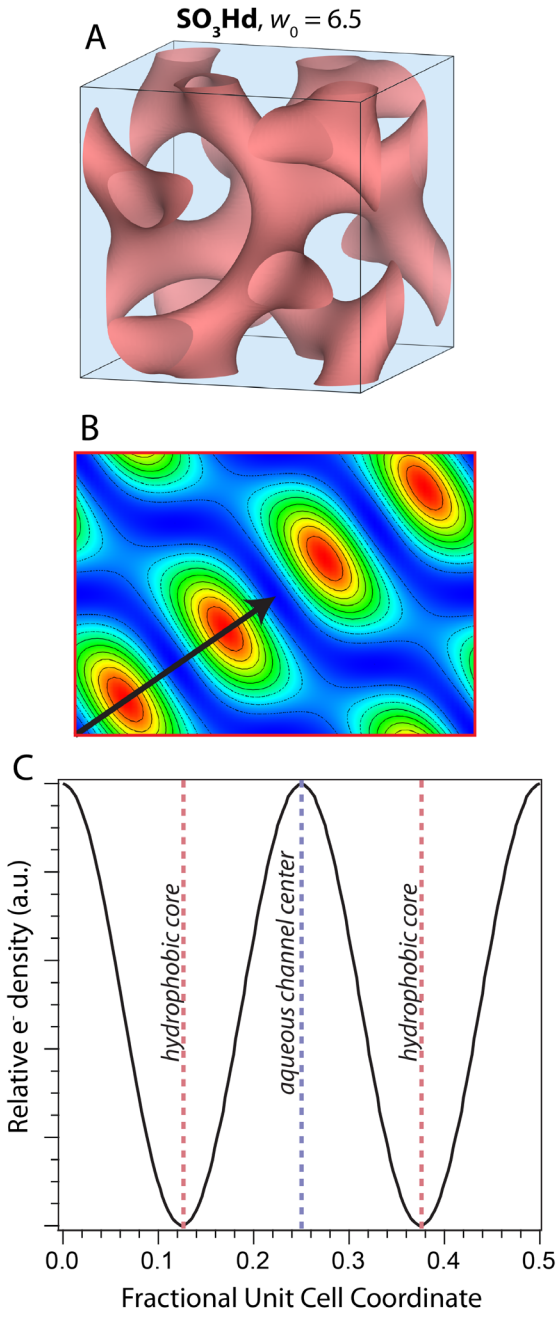

Figure S5. Electron density reconstruction of the **SO_3Hd** G_I phase at w_0 = 6.5. (*A*) The 90% electron density isosurface shows the labyrinthine network of hydrophobic 3-fold connectors (*pink*) in a water matrix (*blue*). (*B*) 2D electron density map of the (110) plane with lines indicating 10% increments in the relative electron density. (*C*) Electron density profile along the [111] direction *versus* fractional unit cell coordinate (*arrow in B*).

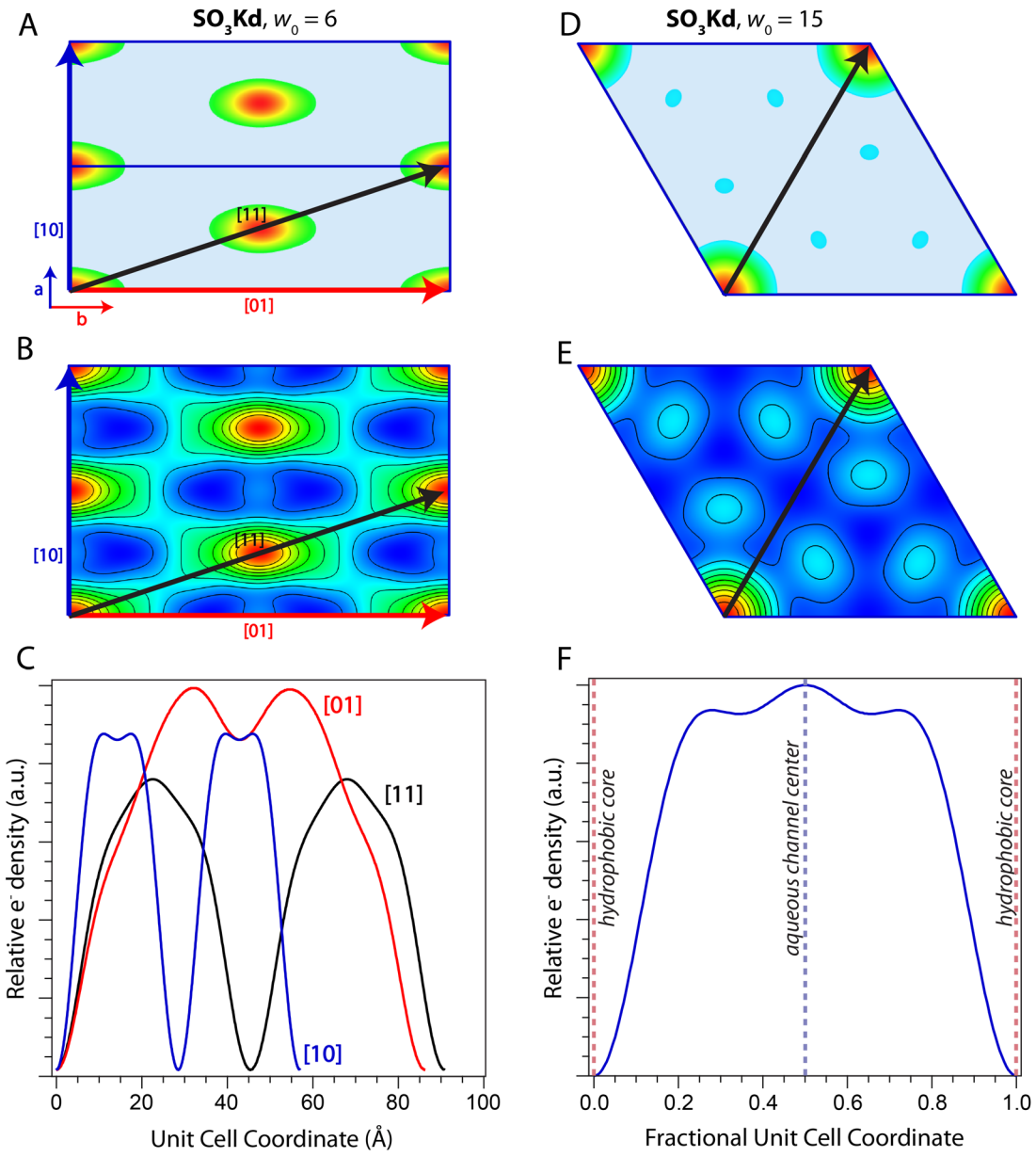

Figure S6. Electron density reconstructions of **SO_3Kd** at w_0 = 6 and 15 indicate spatial localization of K^+ ions. (*A*) 1 x 2 supercell of the **SO_3Kd** R_I phase (01) plane with the micellar interface given by the 90% isosurface. (*B*) 2D electron density map with black lines indicating 10% increments in the relative electron density. (*C*) The linear electron density profiles given as relative electron density *versus* unit cell coordinate reveal significant differences in the electron density between the [10], [01], and [11] directions, indicating that K^+ ions are localized within the R_I morphology. (*D*) Unit cell of the **SO_3Kd** H_I phase and (*E*) 2D electron density map. (*F*) Linear electron density profile along the [11] direction (*arrow in E*) peaks at the center of the aqueous channel due to K^+ ion localization.

***SUPERFLIP* Input File for SO_3Hd G_I phase (w_0 = 6.5) Charge-Flipping Electron Density Reconstruction**

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         x3      x1      x2
     x3+1/2 -x1+1/2     -x2
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endf
```

***SUPERFLIP* Input File for SO_3Hd H_I phase (w_0 = 15) Charge-Flipping Electron Density Reconstruction**

```
*********************************start_file*********************************

   title SO3Hd_Wo15_d37.8_Superflip
   perform CF
   outputfile "SO3Hd_Wo15_d37.8_Superflip.m81"
"SO3Hd_Wo15_d37.8_Superflip.m80"
   outputformat jana
   dimension  3
   cell    37.8078   37.8078   11.8995   90.00   90.00  120.00
   spacegroup P6mm
   centro no
   centers
     0.000000  0.000000  0.000000
   endcenters
   symmetry
        x1     x2     x3
       -x2  x1-x2     x3
    -x1+x2    -x1     x3
       -x1    -x2     x3
        x2 -x1+x2     x3
     x1-x2     x1     x3
       -x2    -x1     x3
    -x1+x2     x2     x3
        x1  x1-x2     x3
        x2     x1     x3
     x1-x2    -x2     x3
       -x1 -x1+x2     x3
   endsymmetry
   composition C378 H840 S42 O126

   # Keywords for charge flipping
   repeatmode 5000 sumgood
   bestdensities 1 symmetry
   polish yes
   maxcycles 5000
   delta AUTO
   weakratio 0.000
   Biso   0.000
   randomseed AUTO
   searchsymmetry average
   derivesymmetry no
   # End of keywords for charge flipping

   # EDMA-specific keywords
   inputfile SO3Hd_Wo15_d37.8_Superflip.m81
   outputbase SO3Hd_Wo15_d37.8_Superflip
   m40forjana yes
   writem40 SO3Hd_Wo15_d37.8_Superflip_tmp.m40
   maxima all
   fullcell no
```

```
scale fractional
plimit    0.3000 sigma
numberofatoms composition
centerofcharge yes
chlimit    0.2500
chlimlist    0.0188 relative
# End of EDMA-specific keywords

electrons       0.0000
dataitemwidths 4 15 15
dataformat intensity fwhm
fbegin
   1   0   0     10000.0000         0.0061
   2  -1   0      2122.7334         0.0058
   2   0   0       286.3814         0.0054
   3  -1   0       107.6284         0.0076
   0   0  -1        57.8162         0.0083
   0   0   1        57.8162         0.0083
   1   0  -1         1.8262         0.0092
   1   0   1         1.8262         0.0092
   3   0   0        42.2099         0.0096
   2   0   1         0.0000         0.0117
   2   0  -1         0.0000         0.0117
   4  -2   0         0.0000         0.0120
   4  -1   0         0.0000         0.0127
   3  -1   1         0.0000         0.0137
   3  -1  -1         0.0000         0.0137
   4   0   0         0.0000         0.0145
   3   0  -1         0.0000         0.0149
   3   0   1         0.0000         0.0149
   5  -2   0         0.0000         0.0162
   4  -2   1         0.0000         0.0165
   4  -2  -1         0.0000         0.0165
   4  -1   1         0.0000         0.0170
   4  -1  -1         0.0000         0.0170
   5  -1   0         0.0000         0.0172
endf
```

***SUPERFLIP* Input File for SO_3Kd R_I phase (w_0 = 6) Charge-Flipping Electron Density Reconstruction**

```
*********************************start_file*********************************

   title SO3K_Wo6_unscaled_Superflip
   perform CF
   outputfile "SO3K_Wo6_unscaled_Superflip.m81"
"SO3K_Wo6_unscaled_Superflip.m80"
   outputformat jana
   dimension  3
   cell    86.2455   28.4929    7.2000   90.00   90.00   90.00
   spacegroup C2mm
   centro no
   centers
     0.000000  0.000000  0.000000
     0.500000  0.500000  0.000000
   endcenters
   symmetry
      x1   x2   x3
      x1  -x2  -x3
      x1   x2  -x3
      x1  -x2   x3
   endsymmetry
   composition C387 S43 O129 K43 H817

   # Keywords for charge flipping
   repeatmode 1500 sumgood
   bestdensities 1 symmetry
   polish yes
   maxcycles 2000
   delta AUTO
   weakratio 0.000
   Biso   0.000
   randomseed AUTO
   searchsymmetry average
   derivesymmetry no
   # End of keywords for charge flipping

   # EDMA-specific keywords
   inputfile SO3K_Wo6_unscaled_Superflip.m81
   outputbase SO3K_Wo6_unscaled_Superflip
   m40forjana yes
   writem40 SO3K_Wo6_unscaled_Superflip_tmp.m40
   maxima all
   fullcell no
   scale fractional
   plimit    0.3000 sigma
   numberofatoms composition
   centerofcharge yes
   chlimit    0.2500
   chlimlist    0.0158 relative
   # End of EDMA-specific keywords
```

```
electrons       0.0000
dataitemwidths 4 15 15
dataformat intensity fwhm
fbegin
   2   0   0      2255.8184         0.0130
   1   1   0     10000.0000         0.0105
   4   0   0       365.0109         0.0079
   3   1   0        20.4727         0.0117
   5   1   0        24.7803         0.0267
   6   0   0        51.7512         0.0281
   0   2   0       295.9213         0.0285
   2   2   0       458.7271         0.0312
   4   2   0        33.4515         0.0383
endf
```

***SUPERFLIP* Input File for SO_3Kd H_I phase (w_0 = 15) Charge-Flipping Electron Density Reconstruction**

```
*********************************start_file*********************************

   title SO3Kd_Wo15_d36.7_Jana_Superflip
   perform CF
   outputfile "SO3Kd_Wo15_d36.7_Jana_Superflip.m81"
"SO3Kd_Wo15_d36.7_Jana_Superflip.m80"
   outputformat jana
   dimension  3
   cell    36.8911   36.8911   10.0000   90.00   90.00  120.00
   spacegroup P6mm
   centro no
   centers
     0.000000  0.000000  0.000000
   endcenters
   symmetry
        x1     x2     x3
       -x2  x1-x2     x3
    -x1+x2    -x1     x3
       -x1    -x2     x3
        x2 -x1+x2     x3
     x1-x2     x1     x3
       -x2    -x1     x3
    -x1+x2     x2     x3
        x1  x1-x2     x3
        x2     x1     x3
     x1-x2    -x2     x3
       -x1 -x1+x2     x3
   endsymmetry
   composition C297 H627 S33 O99 K33

   # Keywords for charge flipping
   repeatmode 5000 sumgood
   bestdensities 1 symmetry
   polish yes
   maxcycles 5000
   delta AUTO
   weakratio 0.000
   Biso   0.000
   randomseed AUTO
   searchsymmetry average
   derivesymmetry no
   # End of keywords for charge flipping

   # EDMA-specific keywords
   inputfile SO3Kd_Wo15_d36.7_Jana_Superflip.m81
   outputbase SO3Kd_Wo15_d36.7_Jana_Superflip
   m40forjana yes
   writem40 SO3Kd_Wo15_d36.7_Jana_Superflip_tmp.m40
   maxima all
   fullcell no
```

```
scale fractional
plimit    0.3000 sigma
numberofatoms composition
centerofcharge yes
chlimit    0.2500
chlimlist    0.0158 relative
# End of EDMA-specific keywords

electrons       0.0000
dataitemwidths 4 15 15
dataformat intensity fwhm
fbegin
   1   0   0     10000.0000         0.0629
   2  -1   0      3363.7949         0.0980
   2   0   0       827.2183         0.1098
   3  -1   0      2419.1187         0.1371
endf
```

***SUPERFLIP* Input File for SO_3NMe_4d H_1 phase (w_0 = 6) Charge-Flipping Electron Density Reconstruction**

```
*********************************start_file*********************************

   title SGJ_68_new_Superflip
   perform CF
   outputfile "SGJ_68_new_Superflip.m81" "SGJ_68_new_Superflip.m80"
   outputformat jana
   dimension  3
   cell    32.9612   32.9612   10.0000   90.00   90.00  120.00
   spacegroup P6mm
   centro no
   centers
     0.000000  0.000000  0.000000
   endcenters
   symmetry
        x1     x2     x3
       -x2  x1-x2     x3
    -x1+x2    -x1     x3
       -x1    -x2     x3
        x2 -x1+x2     x3
     x1-x2     x1     x3
       -x2    -x1     x3
    -x1+x2     x2     x3
        x1  x1-x2     x3
        x2     x1     x3
     x1-x2    -x2     x3
       -x1 -x1+x2     x3
   endsymmetry
   composition C286 H682 O66 N22

   # Keywords for charge flipping
   repeatmode 10000 sumgood
   bestdensities 1 symmetry
   polish yes
   maxcycles 10000
   delta AUTO
   weakratio 0.000
   Biso   0.000
   randomseed AUTO
   searchsymmetry average
   derivesymmetry no
   # End of keywords for charge flipping

   # EDMA-specific keywords
   inputfile SGJ_68_new_Superflip.m81
   outputbase SGJ_68_new_Superflip
   m40forjana yes
   writem40 SGJ_68_new_Superflip_tmp.m40
   maxima all
   fullcell no
   scale fractional
```

```
plimit    0.3000 sigma
numberofatoms composition
centerofcharge yes
chlimit    0.2500
chlimlist    0.0375 relative
# End of EDMA-specific keywords

electrons       0.0000
dataitemwidths 4 15 15
dataformat intensity fwhm
fbegin
   1   0   0     10000.0000         0.0081
   2  -1   0       933.7727         0.0092
   2   0   0         0.5604         0.0094
   3  -1   0       188.6153         0.0138
   0   0   1        63.8442         0.0155
   0   0  -1        63.8442         0.0155
   3   0   0        14.1249         0.0167
   1   0  -1         0.0938         0.0169
   1   0   1         0.0938         0.0169
endf
```

***SUPERFLIP* Input File for SO_3NMe_4d A15 phase (w_0 = 12) Charge-Flipping Electron Density Reconstruction**

```
*********************************start_file*********************************

   title SO3TMA_Wo12_Superflip
   perform CF
   outputfile "SO3TMA_Wo12_Superflip.m81" "SO3TMA_Wo12_Superflip.m80"
   outputformat jana
   dimension  3
   cell    36.4779   36.4779   36.4779   90.00   90.00   90.00
   spacegroup Pm-3n
   centro yes
   centers
     0.000000  0.000000  0.000000
   endcenters
   symmetry
         x1      x2      x3
        -x1     -x2      x3
        -x1      x2     -x3
         x1     -x2     -x3
         x3      x1      x2
         x3     -x1     -x2
        -x3     -x1      x2
        -x3      x1     -x2
         x2      x3      x1
        -x2      x3     -x1
         x2     -x3     -x1
        -x2     -x3      x1
     x2+1/2  x1+1/2 -x3+1/2
    -x2+1/2 -x1+1/2 -x3+1/2
     x2+1/2 -x1+1/2  x3+1/2
    -x2+1/2  x1+1/2  x3+1/2
     x1+1/2  x3+1/2 -x2+1/2
    -x1+1/2  x3+1/2  x2+1/2
    -x1+1/2 -x3+1/2 -x2+1/2
     x1+1/2 -x3+1/2  x2+1/2
     x3+1/2  x2+1/2 -x1+1/2
     x3+1/2 -x2+1/2  x1+1/2
    -x3+1/2  x2+1/2  x1+1/2
    -x3+1/2 -x2+1/2 -x1+1/2
        -x1     -x2     -x3
         x1      x2     -x3
         x1     -x2      x3
        -x1      x2      x3
        -x3     -x1     -x2
        -x3      x1      x2
         x3      x1     -x2
         x3     -x1      x2
        -x2     -x3     -x1
         x2     -x3      x1
        -x2      x3      x1
         x2      x3     -x1
```

```
 -x2+1/2 -x1+1/2  x3+1/2
  x2+1/2  x1+1/2  x3+1/2
 -x2+1/2  x1+1/2 -x3+1/2
  x2+1/2 -x1+1/2 -x3+1/2
 -x1+1/2 -x3+1/2  x2+1/2
  x1+1/2 -x3+1/2 -x2+1/2
  x1+1/2  x3+1/2  x2+1/2
 -x1+1/2  x3+1/2 -x2+1/2
 -x3+1/2 -x2+1/2  x1+1/2
 -x3+1/2  x2+1/2 -x1+1/2
  x3+1/2 -x2+1/2 -x1+1/2
  x3+1/2  x2+1/2  x1+1/2
endsymmetry
composition C1305 H4495 N145 O435

# Keywords for charge flipping
repeatmode 10000 sumgood
bestdensities 1 symmetry
polish yes
maxcycles 2500
delta AUTO
weakratio 0.000
Biso   0.000
randomseed AUTO
searchsymmetry average
derivesymmetry no
# End of keywords for charge flipping

# EDMA-specific keywords
inputfile SO3TMA_Wo12_Superflip.m81
outputbase SO3TMA_Wo12_Superflip
m40forjana yes
writem40 SO3TMA_Wo12_Superflip_tmp.m40
maxima all
fullcell no
scale fractional
plimit    0.3000 sigma
numberofatoms composition
centerofcharge yes
chlimit    0.2500
chlimlist    0.0375 relative
# End of EDMA-specific keywords

electrons       0.0000
dataitemwidths 4 15 15
dataformat intensity fwhm
fbegin
   1   0   1        31.0366         0.0145
   0   0   2      5112.9702         0.0152
   1   0   2     10000.0010         0.0161
   1   1   2      9164.8975         0.0167
   2   0   2       452.9376         0.0176
   1   0   3        44.5720         0.0180
   2   2   2      1247.3428         0.0180
```

```
    2    0    3        432.7714          0.0179
    2    1    3        376.8265          0.0177
    0    0    4        716.5313          0.0168
    1    0    4        452.4106          0.0162
    3    0    3         98.8717          0.0181
    1    1    4         98.8717          0.0181
    2    0    4         24.3707          0.0218
    2    1    4         16.8763          0.0235
    3    2    3         55.6948          0.0250
    2    2    4         78.0731          0.0279
    3    0    4         54.4285          0.0293
    1    0    5         22.5996          0.0306
    3    1    4         22.5996          0.0306
    3    2    4         71.8064          0.0343
    2    0    5         71.8064          0.0343
    2    1    5         42.0632          0.0354
    4    0    4         47.7017          0.0377
    3    0    5          0.0000          0.0398
    3    3    4          0.0000          0.0398
    3    1    5          0.0000          0.0409
    0    0    6          0.0000          0.0419
    4    2    4          0.0000          0.0419
    1    0    6          0.0000          0.0429
    1    1    6          0.0000          0.0439
    3    2    5          0.0000          0.0439
    2    0    6          0.0000          0.0458
    2    1    6          0.0000          0.0468
    4    0    5          0.0000          0.0468
    4    1    5          0.0000          0.0477
    2    2    6          0.0000          0.0495
    3    0    6          0.0000          0.0504
    4    2    5          0.0000          0.0504
    3    1    6          0.0000          0.0513
    4    4    4          0.0000          0.0530
    3    2    6          0.0000          0.0539
    5    0    5          0.0000          0.0547
    4    3    5          0.0000          0.0547
    1    0    7          0.0000          0.0547
    4    0    6          0.0000          0.0564
    4    1    6          0.0000          0.0572
    2    0    7          0.0000          0.0572
    5    2    5          0.0000          0.0580
    2    1    7          0.0000          0.0580
    3    3    6          0.0000          0.0580
endf
```

***SUPERFLIP* Input File for SO_3NMe_4d A15 phase (w_0 = 15) Charge-Flipping Electron Density Reconstruction**

```
*********************************start_file*********************************

   title SO3TMA_Wo15_Superflip
   perform CF
   outputfile "SO3TMA_Wo15_Superflip.m81" "SO3TMA_Wo15_Superflip.m80"
   outputformat jana
   dimension  3
   cell    37.1243   37.1243   37.1243   90.00   90.00   90.00
   spacegroup Pm-3n
   centro yes
   centers
     0.000000  0.000000  0.000000
   endcenters
   symmetry
         x1      x2      x3
        -x1     -x2      x3
        -x1      x2     -x3
         x1     -x2     -x3
         x3      x1      x2
         x3     -x1     -x2
        -x3     -x1      x2
        -x3      x1     -x2
         x2      x3      x1
        -x2      x3     -x1
         x2     -x3     -x1
        -x2     -x3      x1
     x2+1/2  x1+1/2 -x3+1/2
    -x2+1/2 -x1+1/2 -x3+1/2
     x2+1/2 -x1+1/2  x3+1/2
    -x2+1/2  x1+1/2  x3+1/2
     x1+1/2  x3+1/2 -x2+1/2
    -x1+1/2  x3+1/2  x2+1/2
    -x1+1/2 -x3+1/2 -x2+1/2
     x1+1/2 -x3+1/2  x2+1/2
     x3+1/2  x2+1/2 -x1+1/2
     x3+1/2 -x2+1/2  x1+1/2
    -x3+1/2  x2+1/2  x1+1/2
    -x3+1/2 -x2+1/2 -x1+1/2
        -x1     -x2     -x3
         x1      x2     -x3
         x1     -x2      x3
        -x1      x2      x3
        -x3     -x1     -x2
        -x3      x1      x2
         x3      x1     -x2
         x3     -x1      x2
        -x2     -x3     -x1
         x2     -x3      x1
        -x2      x3      x1
         x2      x3     -x1
```

```
 -x2+1/2 -x1+1/2  x3+1/2
  x2+1/2  x1+1/2  x3+1/2
 -x2+1/2  x1+1/2 -x3+1/2
  x2+1/2 -x1+1/2 -x3+1/2
 -x1+1/2 -x3+1/2  x2+1/2
  x1+1/2 -x3+1/2 -x2+1/2
  x1+1/2  x3+1/2  x2+1/2
 -x1+1/2  x3+1/2 -x2+1/2
 -x3+1/2 -x2+1/2  x1+1/2
 -x3+1/2  x2+1/2 -x1+1/2
  x3+1/2 -x2+1/2 -x1+1/2
  x3+1/2  x2+1/2  x1+1/2
endsymmetry
composition C1368 H4712 N152 O456

# Keywords for charge flipping
repeatmode 10000 sumgood
bestdensities 1 symmetry
polish yes
maxcycles 2500
delta AUTO
weakratio 0.000
Biso   0.000
randomseed AUTO
searchsymmetry average
derivesymmetry no
# End of keywords for charge flipping

# EDMA-specific keywords
inputfile SO3TMA_Wo15_Superflip.m81
outputbase SO3TMA_Wo15_Superflip
m40forjana yes
writem40 SO3TMA_Wo15_Superflip_tmp.m40
maxima all
fullcell no
scale fractional
plimit    0.3000 sigma
numberofatoms composition
centerofcharge yes
chlimit    0.2500
chlimlist    0.0375 relative
# End of EDMA-specific keywords

electrons       0.0000
dataitemwidths 4 15 15
dataformat intensity fwhm
fbegin
   1   0   1        28.4806         0.0167
   0   0   2      5342.5327         0.0169
   1   0   2      9852.8613         0.0170
   1   1   2     10000.0020         0.0170
   2   0   2       383.3503         0.0169
   1   0   3         4.4922         0.0168
   2   2   2      2277.3516         0.0166
```

```
    2    0    3        988.8835          0.0165
    2    1    3       1030.8788          0.0164
    0    0    4       1725.5237          0.0161
    1    0    4        487.4051          0.0159
    3    0    3        145.1292          0.0157
    1    1    4        145.1292          0.0157
    2    0    4         44.6244          0.0152
    2    1    4          0.5640          0.0150
    3    2    3         15.3639          0.0146
    2    2    4         46.0763          0.0139
    3    0    4         27.6270          0.0135
    1    0    5         10.3073          0.0131
    3    1    4         10.3073          0.0131
    3    2    4         96.9543          0.0155
    2    0    5         96.9543          0.0155
    2    1    5         67.5026          0.0164
    4    0    4        148.6081          0.0179
endf
```

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