Supporting Information for:

Anion Pairs Template a Trigonal Prism with Disilver Vertices

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**S1 Experimental Procedures**

Unless otherwise specified, all starting materials, solvents and reagents were used as supplied and without further purification. Flash column chromatography was performed using Silica Gel high purity grade (pore size 60 Å, 230-400 mesh particle size, Sigma-Aldrich). All anhydrous reactions were carried out in oven-dried glassware and under an inert atmosphere of nitrogen provided by a balloon. All reactions were stirred with magnetic followers. Brine refers to a saturated aqueous solution of sodium chloride. Centrifugation of samples was carried out using a Grant-Bio LMC-3000 low speed benchtop centrifuge.
S2 Instrumentation and Characterisation Methods

Mass Spectrometry

Low-resolution electrospray ionisation (ESI) mass spectra were obtained on a Micromass Quattro LC mass spectrometer (cone voltage 20 eV; desolvation temp. 313 K; ionisation temp. 313 K) infused from a Harvard syringe pump at a rate of 4-10 µL min⁻¹. High-resolution ESI mass spectra were obtained by the EPSRC UK National Mass Spectrometry Facility at Swansea University using a Thermo Scientific LTQ Orbitrap XL hybrid ion trap-orbitrap mass spectrometer.

NMR Spectroscopy

NMR spectra were recorded on Bruker 400 Avance III HD Smart Probe, Bruker Avance 500 Cryo, Bruker 500 TCI-ATM Cryo and Bruker 700 TCI-ATM Cryo spectrometers. Chemical shifts (δ) for ¹H NMR spectra are reported in parts per million (ppm) and are reported relative to the solvent residual peak. DOSY experiments were carried out on a Bruker DRX-400 spectrometer. Coupling constants (J) are reported in hertz (Hz). The following abbreviations are used to describe signal multiplicity in ¹H, ¹³C and ¹⁹F NMR spectra: s: singlet, d: doublet, t: triplet, dd: doublet of doublets; dt: doublet of triplets; m: multiplet, br: broad. DOSY NMR experiments were performed on a 400 MHz Avance III HD Smart Probe NMR spectrometer. Maximum gradient strength was 6.57 G/cm A. The standard Bruker pulse program, ledbpgp2s, employing a stimulated echo and longitudinal eddy-current delay (LED) using bipolar gradient pulses for diffusion using 2 spoil gradients was utilised. Rectangular gradients were used with a total duration of 1.5 ms. Gradient recovery delays were 875-1400 µs. Individual rows of the S4 quasi-2D diffusion databases were phased and baseline corrected.
S3 Synthesis of Ag_{12}L_{6} Cage (ClO_{4})_{2} ⊂ 1

Tris(4-aminophenyl)amine (11.6 mg, 40 µmol, 6 equiv.), silver(I) perchlorate (16.6 mg, 80 µmol, 12 equiv.) and 1,8-naphthyridine-2-carbaldehyde (19.0 mg, 121 µmol, 18 equiv.) were stirred in CD_{3}CN (3.00 mL) at room temperature for 18 h in a sealed vessel under a nitrogen atmosphere and in the dark, yielding a dark purple solution. The reaction mixture was then filtered through a glass fibre filter (0.7 µm pore size). The acetonitrile was concentrated under a stream of N_{2} to a volume of 1 mL. Addition of diethyl ether (10 mL) resulted in the precipitation of a dark purple powder. The suspension was then centrifuged (10 min, 3000 RPM), the eluent decanted. Further diethyl ether (10 mL) was added, the powder was resuspended by sonication and then centrifuged. Again, the eluent was decanted. The residue was then dried in vacuo to afford the solid product as a fine dark red powder (44 mg, 94%).

{\textbf{1H NMR}} (500 MHz, 298 K, CD_{3}CN) δ: 9.71 (H_{5}, s, 6H), 9.10 (H_{30}, dd, J = 4.23, 1.88 Hz, 6H), 9.08 (H_{15}, s, 6H), 9.07 (H_{7}, d, J = 8.13 Hz, 6H), 9.02 (H_{6}, d, J = 8.21 Hz, 6H), 8.98 (H_{20}, dd, J = 4.61, 1.74 Hz, 6H), 8.97 (H_{17}, d, J = 8.39 Hz, 6H), 8.80 (H_{27}, d, J = 8.29 Hz, 6H), 8.80 (H_{8}, br, 6H), 8.79 (H_{10}, dd, J = 4.76, 1.76 Hz, 6H), 8.73 (H_{25}, s, 6H), 8.73 (H_{18}, dd, J = 8.20, 1.79 Hz, 6H), 8.64 (H_{28}, dd, J = 8.39, 1.87 Hz, 6H), 8.32 (H_{16}, d, J = 8.31 Hz, 6H), 8.02 (H_{26}, d, J = 8.29 Hz, 6H), 7.95 (H_{4} or H_{2}, d, J = 7.38 Hz, 6H), 7.78 (H_{29}, dd, J = 8.29, 4.25 Hz, 6H), 7.51 (H_{19}, dd, J = 8.2, 4.83 Hz, 6H), 7.49 (H_{9}, dd, J = 8.27, 4.66 Hz, 6H), 7.27 (H_{3} or H_{1}, d, J = 7.8 Hz, 6H), 7.11 (H_{4} or H_{2}, d, J = 8.14 Hz, 6H), 7.07-7.02 (H_{23}, H_{21}, H_{14} and H_{12}, m, 24H), 6.96 (H_{3} or H_{1}, d, J = 7.68 Hz, 6H), 6.81 (H_{13} and H_{11}, d, J = 8.78 Hz, 12H), 6.44 (H_{24} and H_{22}, d, J = 9.14 Hz, 12H)

{\textbf{13C NMR}} (125 MHz, 298 K, CD_{3}CN) δ: 160.5 (imine), 159.6 (naphthyridine), 157.5 (imine), 156.4 (imine), 156.2 (naphthyridine), 155.6 (naphthyridine), 155.5 (naphthyridine), 155.1 (naphthyridine), 154.4 (naphthyridine), 154.1 (naphthyridine), 152.3 (naphthyridine), 151.8 (naphthyridine), 148.8 (naphthyridine), 147.5 (naphthyridine), 147.4 (naphthyridine), 144.1 (naphthyridine), 143.6 (naphthyridine), 143.1 (naphthyridine), 142.8 (naphthyridine), 142.4 (naphthyridine), 142.2 (naphthyridine), 141.2 (naphthyridine), 140.6 (naphthyridine), 139.1 (naphthyridine), 129.7 (aniline), 128.6 (aniline), 128.4 (aniline), 127.7 (aniline), 127.5 (aniline), 126.6 (aniline), 126.5 (aniline), 125.8 (aniline), 125.7 (aniline), 125.2 (aniline), 124.7 (aniline), 122.2 (aniline), 120.0 (aniline), 119.6 (aniline), 117.1 (aniline)

{\textbf{HRMS}} (ESI-LTQ Orbitrap XL, CH_{3}CN) m/z found 1251.2151, calculated 1251.0185 for C_{270}H_{180}Ag_{12}N_{60}(ClO_{4})_{7}^{5+} (Figure S9).
**Figure S1:** Aromatic region of the $^1$H NMR spectrum (500 MHz, 298 K, CD$_3$CN) of (ClO$_4$)$^-$⊂ 1. Minor peaks at 9.2, 8.6 and 8.5 ppm correspond to traces of unreacted 1,8-naphthyridine-2-carbaldehyde.

**Figure S2:** Aromatic region of the $^{13}$C NMR spectrum (125 MHz, 298 K, CD$_3$CN) of (ClO$_4$)$^-$⊂ 1.
Figure S3: Aromatic region of the $^1$H-$^1$H COSY spectrum (500 MHz, 298 K, CD$_3$CN) for (ClO$_4$)$_2 \subset 1$. 
Figure S4: Aromatic region of the $^1$H-$^{13}$C HSQC spectrum (500 MHz, 298 K, CD$_3$CN) of (ClO$_4$)$_2 \subset 1$. 
Figure S5: Aromatic region of the $^1$H-$^1$H NOESY spectrum (500 MHz, 298 K, CD$_3$CN) of (ClO$_4$)$_2 \subset 1$. 
Figure S6: Aromatic region of the $^1$H-$^{13}$C HMBC spectrum (500 MHz, 298 K, CD$_3$CN) of $(\text{ClO}_4^-)_2 \subset 1$.

Figure S7: $^1$H DOSY spectrum (400 MHz, 298 K, CD$_3$CN) of $(\text{ClO}_4^-)_2 \subset 1$. The diffusion coefficient of $(\text{ClO}_4^-)_2 \subset 1$ was measured to be $4.8 \times 10^{-6}$ cm$^2$ s$^{-1}$, giving a solvodynamic radius of 12 Å. This value is comparable to those measured from the crystal structure of $(\text{ClO}_4^-)_2 \subset 1$ (10-16 Å along the various axes of the asymmetric polyhedron).
Figure S8: Low-resolution ESI-mass spectrum of (ClO₄⁻)₂ ⊂ 1 with measured (in blue) and calculated (in orange) m/z values included.
Figure S9: High resolution ESI-mass spectrometry analysis of (ClO₄)₂ ⊂ 1 showing the +5 peak.
S4 Anion Templation of Cage 1

Tris(4-aminophenyl)amine (11.6 mg, 40 µmol, 6 equiv.), silver(I) bis(trifluoromethane)sulfonimide (40.0 mg, 100 µmol, 15 equiv.) and 1,8-naphthyridine-2-carbaldehyde (19.0 mg, 121 µmol, 18 equiv.) were stirred in CD$_3$CN (10 mL) at room temperature for 2 h or until all of the material was in solution. Aliquots of this stock solution (500 µL) were then added to J-Young tubes followed by 2 equivalents of template anion (0.67 µmol) from the salts of TBABF$_4$, TBAPF$_6$, TBAReO$_4$, TBAOTf, TBACIO$_4$, TBAHSO$_4$, Na$_2$SO$_4$, K$_2$S$_2$O$_8$, ethane-1,2-disulfonate disodium salt and propane-1,3-disulfonate disodium salt. The reaction mixture was stirred at room temperature under nitrogen and in the dark for 18 hours. The resulting purple solutions were used directly for characterization.

Figure S10: $^1$H NMR spectrum (500 MHz, 298 K, CD$_3$CN) of (X$^-$)$_2 \subset 1 \cdot (\text{NTf}_2^-)_{10}$ after equilibration at room temperature for 18 h in the dark (X$^-$ = BF$_4^-$, PF$_6^-$, ReO$_4^-$, OTf$^-$, ClO$_4^-$, HSO$_4^-$, SO$_4^{2-}$).
Figure S11: $^1$H NMR spectrum (500 MHz, 298 K, CD$_3$CN) of $(X^-)_2 \subset 1\cdot$(NTf$_2$)$_{10}$ with different templating anions, focusing on the internal imine $^1$H environment (H$_5$ as labelled in Figure S1) which shifts significantly for different anions.
S5 Characterisation of Cage 1 Templated with Different Anions

\[(\text{BF}_4^-)_2 \subset 1 \cdot (\text{NTf}_2^-)_{10}\]

**\(^1H\) NMR** (500 MHz, 298 K, CD\(_3\)CN) \(\delta:\) 9.65 (H\(_5\), s, 6H), 9.10 (H\(_{30}\), dd, \(J = 4.3, 1.9\) Hz, 6H), 9.05 (H\(_{15}\), s, 6H), 9.01 (H\(_{Ar}\), dd, \(J = 4.7, 1.8\) Hz, 6H), 8.97 (H\(_{Ar}\), d, \(J = 8.3\) Hz, 6H), 8.91 (H\(_{Ar}\), d, \(J = 8.3\) Hz, 6H), 8.84 (H\(_{Ar}\), d, \(J = 8.2\) Hz, 6H), 8.81 (H\(_{Ar}\), d, \(J = 8.4\) Hz, 6H), 8.78 (H\(_{Ar}\), dd, \(J = 4.8, 1.8\) Hz, 6H), 8.73 (H\(_{25}\), s, 6H), 8.73 (H\(_{18}\), dd, \(J = 7.8, 1.8\) Hz, 6H), 8.64 (H\(_{Ar}\), dd, \(J = 8.4, 1.7\) Hz, 12H), 8.29 (H\(_{16}\), d, \(J = 8.2\) Hz, 6H), 8.03 (H\(_{26}\), d, \(J = 8.2\) Hz, 6H), 7.79 (H\(_{29}\), dd, \(J = 8.1, 4.3\) Hz, 6H), 7.51 (H\(_{19}\), dd, \(J = 8.6, 4.9\) Hz, 6H), 7.49 (H\(_{9}\), dd, \(J = 8.6, 4.7\) Hz, 6H), 7.01 (H\(_{Ar}\), dd, \(J = 8.4\) Hz, 12H), 6.80 (H\(_{13}\) and H\(_{11}\), d, \(J = 8.7\) Hz, 12H), 6.45 (H\(_{24}\) and H\(_{22}\), d, \(J = 9.1\) Hz, 12H).

**\(^{13}C\) NMR** (125 MHz, 298 K, CD\(_3\)CN) \(\delta:\) 160.7, 159.4, 157.5, 156.6, 155.5, 155.3, 155.1, 154.4, 154.2, 152.4, 151.8, 148.3, 147.5, 147.2, 144.1, 143.5, 143.1, 142.5, 142.3, 142.2, 141.1, 140.8, 139.1, 128.7, 128.3, 127.9, 127.4, 127.4, 126.5, 125.8, 125.6, 125.2, 120.8 (q, \(J = 320\) Hz), 119.5. Due to overlapping peaks, 33 of the expected 39 independent peaks were observed.

**\(^{19}F\) NMR** (376 MHz, 298 K, CD\(_3\)CN) \(\delta:\) 151.19.

Figure S12: Aromatic region of the \(^1H\) NMR spectrum (500 MHz, 298 K, CD\(_3\)CN) of \((\text{BF}_4^-)_2 \subset 1 \cdot (\text{NTf}_2^-)_{10}\). Minor peaks at 9.2, 8.6 and 8.5 ppm correspond to traces of unreacted 1,8-naphthyridine-2-carbaldehyde, and broadening is caused by slow rotation of phenylene rings.
Figure S13: Aromatic region of the $^{13}$C NMR spectrum (125 MHz, 298 K, CD$_3$CN) of (BF$_4^-$)$_2$ ⊂ 1·(NTf$_2^-$)$_{10}$.
Figure S14: Aromatic region of the $^{19}$F NMR spectrum (376 MHz, 298 K, CD$_3$CN) of $(\text{BF}_4^-)_2 \subset _1\text{NTf}_2^-_{10}$.
Figure S15: Aromatic region of the $^1$H-$^1$H COSY spectrum (500 MHz, 298 K, CD$_3$CN) for (BF$_4$)$_2 \subset 1\cdot$(NTf$_2$)$_{10}$. 
Figure S16: Aromatic region of the $^1\text{H}-^{13}\text{C}$ HSQC spectrum (500 MHz, 298 K, CD$_3$CN) for (BF$_4$)$_2 \subset 1\cdot$(NTf$_2$)$_{10}$. 
$\text{(PF}_6^-)_2 \subset 1\cdot\text{(NTf}_2^-)_{10}$

$^1\text{H NMR}$ (500 MHz, 298 K, CD$_3$CN) $\delta$: 9.57 (H$_5$, s, 6H), 9.08 (H$_{30}$, dd, $J = 5.9, 2.7$ Hz, 6H), 9.05 (H$_{15}$, s, 6H), 8.98 (H$_{Ar}$, d, $J = 11.5$ Hz, 6H), 8.88 (H$_{Ar}$, dd, $J = 6.4, 2.2$ Hz, 6H), 8.83 (H$_{Ar}$, dd, $J = 6.7, 2.4$ Hz, 6H), 8.80 (H$_{Ar}$, d, $J = 11.7$ Hz, 6H), 8.77-8.73 (H$_{Ar}$, m, 12H), 8.66 (H$_{Ar}$, s, 6H), 8.64 (H$_{Ar}$, dd, $J = 11.7, 2.4$ Hz, 6H), 8.56 (H$_{Ar}$, dd, $J = 11.4, 2.2$ Hz, 6H), 8.30 (H$_{16}$, d, $J = 11.6$ Hz, 6H), 8.00 (H$_{26}$, d, $J = 11.7$ Hz, 6H), 7.79 (H$_{29}$, dd, $J = 11.7, 5.9$ Hz, 6H), 7.73 (s, 6H), 7.52 (H$_{19}$, dd, $J = 11.3, 6.6$ Hz, 6H), 7.44 (H$_9$, dd, $J = 11.0, 6.2$ Hz, 6H), 6.45 (H$_{24}$ and H$_{22}$, d, $J = 12.9$ Hz, 12H).

$^{13}\text{C NMR}$ (125 MHz, 298 K, CD$_3$CN) $\delta$: 161.0, 159.2, 157.6, 157.4, 156.7, 156.3, 155.5, 155.1, 155.0, 154.8, 154.0, 152.6, 151.7, 148.1, 147.6, 147.5, 144.1, 143.4, 143.2, 142.4, 142.3, 140.9, 140.8, 140.1, 139.1, 128.7, 128.3, 127.4, 127.2, 126.5, 125.8, 125.6, 125.4, 125.3, 125.2, 124.7, 120.8 (q, $J = 320$ Hz), 119.6. Due to overlapping peaks, 37 of the expected 39 independent peaks were observed.

$^{19}\text{F NMR}$ (376 MHz, 298 K, CD$_3$CN) $\delta$: 73.59 (d, $J = 709.3$ Hz).

$^{31}\text{P NMR}$ (162 MHz, 298 K, CD$_3$CN) $\delta$: 144.38 (sept, $J = 707.5$ Hz).

Figure S17: Aromatic region of the $^1\text{H NMR}$ spectrum (500 MHz, 298 K, CD$_3$CN) of $\text{(PF}_6^-)_2 \subset 1\cdot\text{(NTf}_2^-)_{10}$. Minor peaks at 9.2, 8.6 and 8.5 ppm correspond to traces of unreacted 1,8-naphthyridine-2-carbaldehyde, and broadening is caused by slow rotation of phenylene.
rings. Additional broadening may be caused by this anion being a suboptimal template, leading the system to express additional non-templated products.

Figure S18: Aromatic region of the $^{13}$C NMR spectrum (125 MHz, 298 K, CD$_3$CN) of (PF$_6$)$_2 \subset 1 \cdot (\text{NTf}_2^-)_{10}$. 
Figure S19: Aromatic region of the $^{19}$F NMR spectrum (376 MHz, 298 K, CD$_3$CN) of $(\text{PF}_6^-)_2 \subset 1\cdot(\text{NTf}_2^-)_{10}$.
Figure S20: Aromatic region of the $^{31}$P NMR spectrum (162 MHz, 298 K, CD$_3$CN) of (PF$_6^-$)$_2 \subset 1^\cdot$(NTf$_2^-$)$_{10}$. 
Figure S21: Aromatic region of the $^1$H-$^1$H COSY spectrum (500 MHz, 298 K, CD$_3$CN) for (PF$_6^-$)$_2 \subset 1\cdot$(NTf$_2^-$)$_{10}$.

Figure S22: $^1$H DOSY spectrum (400 MHz, 298 K, CD$_3$CN) of (PF$_6^-$)$_2 \subset 1\cdot$(NTf$_2^-$)$_{10}$. We
observed a single species in the aromatic region, along with a species corresponding to the tetrabutylammonium cation in the aliphatic region (present from the TBAX salt used to template cage assembly).

$$(\text{ReO}_4^-)_2 \subset 1\cdot(\text{NTf}_2^-)_{10}$$

$\text{^1H NMR } (500 \text{ MHz}, 298 \text{ K, CD}_3\text{CN}) \delta$: 9.52 (H$_5$, s, 6H), 9.09 (H$_{30}$, dd, $J = 4.31$, 2.00 Hz, 6H), 9.05 (H$_{15}$, s, 6H), 8.98 (H$_{17}$, d, $J = 8.72$ Hz, 6H), 8.91 (H$_{20}$, dd, $J = 4.6$, 1.8 Hz, 1H), 8.82-8.77 (H$_{Ar}$, m, 18H), 8.74 (H$_{17}$, dd, $J = 8.09$, 1.99 Hz, 6H), 8.72 (H$_{Ar}$, d, $J = 8.28$ Hz, 6H), 8.64 (H$_{Ar}$, dd, $J = 8.4$, 1.9 Hz, 6H), 8.59 (H$_{Ar}$, dd, $J = 8.4$, 1.8 Hz, 6H), 8.30 (H$_{16}$, d, $J = 8.3$ Hz, 6H), 8.00 (H$_{26}$, d, $J = 8.3$ Hz, 6H), 7.78 (H$_{29}$, dd, $J = 8.3$, 4.3 Hz, 6H), 7.51 (H$_{19}$, dd, $J = 8.2$, 4.8 Hz, 6H), 7.46 (H$_9$, dd, $J = 8.2$, 4.6 Hz, 6H), 7.21 (s, 12H), 7.06 (H$_{Ar}$, d, $J = 8.8$ Hz, 12H), 6.95 (H$_{Ar}$, s, 6H), 6.89 (H$_{Ar}$, d, $J = 8.8$ Hz, 12H), 6.44 (H$_{24}$ and H$_{22}$, d, $J = 9.06$ Hz, 12H).

$\text{^1^3C NMR } (125 \text{ MHz}, 298 \text{ K, CD}_3\text{CN}) \delta$: 160.9, 159.3, 157.4, 156.5, 155.6, 155.3, 155.0, 155.0, 154.5, 154.1, 152.6, 151.7, 148.3, 147.6, 147.4, 144.1, 143.6, 143.0, 142.4, 142.2, 140.9, 140.6, 139.1, 128.7, 128.3, 127.8, 127.3, 127.0, 126.6, 126.5, 125.9, 125.6, 125.2, 120.8 (q, $J = 320$ Hz), 119.3. Due to overlapping peaks, 35 of the expected 39 independent peaks were observed.

Figure S23: Aromatic region of the $\text{^1H NMR}$ spectrum (500 MHz, 298 K, CD$_3$CN) of $((\text{ReO}_4^-)_2$
\( \subset 1\cdot(\text{NTf}_2^-)_{10} \). Minor peaks at 9.2, 8.6 and 8.5 ppm correspond to traces of unreacted 1,8-naphthyridine-2-carbaldehyde, and broadening is caused by slow rotation of phenylene rings.

**Figure S24:** Aromatic region of the \(^{13}\text{C}\) NMR spectrum (125 MHz, 298 K, CD\(_3\)CN) of \((\text{ReO}_4^-)_2 \subset 1\cdot(\text{NTf}_2^-)_{10}\).
**Figure S25:** Aromatic region of the $^1$H-$^1$H COSY spectrum (500 MHz, 298 K, CD$_3$CN) for (ReO$_4$)$_2$⊂1·(NTf$_2$)$_{10}$.

**Figure S26:** $^1$H DOSY spectrum (400 MHz, 298 K, CD$_3$CN) of (ReO$_4$)$_2$⊂1·(NTf$_2$)$_{10}$. We
observed a single species in the aromatic region, along with a species corresponding to the tetrabutylammonium cation in the aliphatic region (present from the TBAX salt used to template cage assembly).

**Figure S27:** Aromatic region of the $^{1}$H-$^{13}$C HSQC spectrum (500 MHz, 298 K, CD$_3$CN) for (ReO$_4$)$_2 \subset 1$·(NTf$_2$)$_{10}$. 
\[(\text{OTf}^-)_2 \subset 1\text{-}(\text{NTf}_2^-)_{10}\]

\[^1\text{H NMR}\] (500 MHz, 298 K, CD$_3$CN) δ: 9.09 (H$_{30}$, dd, $J = 6.0$, 2.8 Hz, 6H), 9.03 (H$_{15}$, s, 6H), 8.97 (H$_{Ar}$, d, $J = 11.7$ Hz, 12H), 8.93 (H$_{Ar}$, d, $J = 5.0$ Hz, 6H), 8.81 (H$_{Ar}$, d, $J = 11.6$ Hz, 6H), 8.80-8.76 (H$_{Ar}$, m, 12H), 8.74 (H$_{25}$, s, 6H), 8.73 (H$_{18}$, dd, $J = 12.1$, 2.3 Hz, 6H), 8.29 (H$_{16}$, d, $J = 11.7$ Hz, 6H), 8.03 (H$_{26}$, d, $J = 11.7$ Hz, 6H), 7.78 (H$_{29}$, dd, $J = 11.9$, 5.8 Hz, 6H), 7.50 (H$_{19}$, dd, $J = 11.9$, 6.7 Hz, 6H), 7.47 (H$_{9}$, dd, $J = 11.6$, 6.4 Hz, 6H), 7.29 (H$_3$ or H$_1$, br, 6H), 7.09 (H$_{Ar}$, br, 6H), 7.04 (H$_{Ar}$, d, $J = 12.1$ Hz, 12H), 6.95 (H$_{Ar}$, br, 6H), 6.86 (H$_{Ar}$, d, $J = 11.9$ Hz, 12H), 6.48 (H$_{24}$ and H$_{22}$, d, $J = 11.9$ Hz, 12H).

\[^{13}\text{C NMR}\] (125 MHz, 298 K, CD$_3$CN) δ: 160.8, 159.3, 157.4, 156.6, 155.5, 155.3, 155.0, 154.7, 154.1, 152.5, 151.7, 148.3, 147.6, 147.4, 144.1, 143.3, 143.1, 142.9, 142.4, 142.2, 140.9, 140.9, 139.1, 128.7, 128.3, 127.6, 127.3, 126.6, 126.5, 125.8, 125.6, 125.2, 120.8 (q, $J = 320$ Hz), 119.6. Due to overlapping peaks, 33 of the expected 39 independent peaks were observed.

\[^{19}\text{F NMR}\] (376 MHz, 298 K, CD$_3$CN) δ: 78.64.

**Figure S28:** Aromatic region of the $^1$H NMR spectrum (500 MHz, 298 K, CD$_3$CN) of (OTf)$^2_\subset 1\text{-}(\text{NTf}_2^-)_{10}$. Minor peaks at 9.2, 8.6 and 8.5 ppm correspond to traces of unreacted 1,8-naphthyridine-2-carbaldehyde, and broadening is caused by slow rotation of phenylene rings.
Figure S29: Aromatic region of the $^{13}$C NMR spectrum (125 MHz, 298 K, CD$_3$CN) of (OTf$^-$)$_2 \subset 1\cdot$NTf$_2^-$)$_{10}$. 
Figure S30: Aromatic region of the $^{19}$F NMR spectrum (376 MHz, 298 K, CD$_3$CN) of (OTf$^-$)$_2$ ⊂ 1·(NTf$_2^-$)$_{10}$. 
Figure S31: Aromatic region of the $^1$H-$^1$H COSY spectrum (500 MHz, 298 K, CD$_3$CN) for (OTf$^-$)$_2 \subset 1\cdot$(NTf$_2^-$)$_{10}$. 
Figure S32: Aromatic region of the $^1$H-$^{13}$C HSQC spectrum (500 MHz, 298 K, CD$_3$CN) for (OTf$^-$)$_2$ $\subset$ 1•(NTf$_2$)$^{10}$.
\((S_2O_8^{2-}) \subset 1\cdot(NTf_2)^{10}\)

**\(^1H\) NMR** (500 MHz, 298 K, CD\(_3CN\)) \(\delta: 9.82\) (H\(_5\), s, 6H), 9.09 (H\(_{30}\), dd, \(J = 5.7, 2.5\) Hz, 6H), 9.02 (H\(_{15}\), s, 6H), 9.00-8.94 (H\(_{Ar}\), m, 18H), 8.83 (H\(_{Ar}\), d, \(J = 11.6\) Hz, 6H), 8.80 (H\(_{Ar}\), s, 6H), 8.75-8.70 (H\(_{Ar}\), m, 18H), 8.64 (H\(_{Ar}\), dd, \(J = 11.6, 2.5\) Hz, 6H), 8.61 (H\(_{Ar}\), dd, \(J = 11.6, 2.4\) Hz, 6H), 8.29 (H\(_{16}\), d, \(J = 11.5\) Hz, 6H), 8.06 (H\(_{26}\), d, \(J = 11.6\) Hz, 6H), 7.99 (H\(_{Ar}\), br, 12H), 7.78 (H\(_{29}\), dd, \(J = 11.9, 5.6\) Hz, 6H), 7.51 (H\(_{19}\), dd, \(J = 6.3, 5.1\) Hz, 6H), 7.49 (H\(_9\), dd, \(J = 6.7, 5.1\) Hz, 6H), 7.36 (H\(_{Ar}\), br, 6H), 7.01 (H\(_{Ar}\), d, \(J = 11.9\) Hz, 12H), 6.95 (H\(_{Ar}\), br, 6H), 6.74 (H\(_{13}\) and H\(_{11}\), d, \(J = 12.2\) Hz, 12H), 6.48 (H\(_{24}\) and H\(_{22}\), d, \(J = 12.6\) Hz, 12H).

**\(^{13}C\) NMR** (125 MHz, 298 K, CD\(_3CN\)) \(\delta: 160.5, 159.5, 157.4, 156.4, 156.1, 155.6, 155.4, 155.1, 154.3, 154.1, 152.3, 151.7, 148.8, 147.5, 147.4, 144.1, 143.4, 143.0, 142.8, 142.4, 142.2, 141.4, 141.1, 140.5, 139.0, 129.6, 128.6, 128.3, 127.7, 127.5, 127.4, 126.6, 126.5, 125.8, 125.6, 125.2, 120.8 (q, \(J = 320\) Hz), 119.9. Due to overlapping peaks, 37 of the expected 39 independent peaks were observed.

**Figure S33:** Aromatic region of the \(^1H\) NMR spectrum (500 MHz, 298 K, CD\(_3CN\)) of \((S_2O_8^{2-}) \subset 1\cdot(NTf_2)^{10}\). Minor peaks at 9.2, 8.6 and 8.5 ppm correspond to traces of unreacted 1,8-naphthyridine-2-carbaldehyde, and broadening is caused by slow rotation of phenylene rings.
Figure S34: Aromatic region of the $^{13}$C NMR spectrum (125 MHz, 298 K, CD$_3$CN) of ($S_2O_8^{2-}$)⊂$1^\cdot$(NTf$_2^-$)$_{10}$. 
Figure S35: Aromatic region of the $^1$H-$^1$H COSY spectrum (500 MHz, 298 K, CD$_3$CN) for (S$_2$O$_8$)$^-$⊂ 1·(NTf$_2$)$_{10}$. 
Figure S36: Aromatic region of the $^1$H-$^{13}$C HSQC spectrum (500 MHz, 298 K, CD$_3$CN) for $(S_2O_8^{2-}) \subset 1\cdot(NTf_2^-)_{10}$. 
\[(\text{HSO}_4^-)_2 \subset \text{1-(NTf}_2^-)_10\].

\(^1\text{H NMR}\) (500 MHz, 298 K, CD\(_3\)CN) \(\delta: 9.83\) (H\(_5\), s, 6H), 9.09 (H\(_{30}\), dd, \(J = 4.1, 1.9\) Hz, 6H), 9.03 (H\(_{15}\), s, 6H), 8.99 (H\(_{20}\), dd, \(J = 4.6, 1.9\) Hz, 6H), 8.98-8.95 (H\(_{\text{Ar}}\), m, 12H), 8.84-8.79 (H\(_{\text{Ar}}\), m, 18H), 8.72 (H\(_{18}\), dd, \(J = 4.1, 1.7\) Hz, 6H), 8.71 (H\(_{25}\), s, 6H), 8.67 (H\(_{\text{Ar}}\), dd, \(J = 8.3, 1.8\) Hz, 12H), 8.64 (H\(_{\text{Ar}}\), dd, \(J = 8.6, 1.9\) Hz, 12H), 8.29 (H\(_{16}\), d, \(J = 8.5\) Hz, 6H), 8.06 (H\(_{26}\), d, \(J = 8.4\) Hz, 6H), 7.78 (H\(_{29}\), dd, \(J = 8.2, 4.2\) Hz, 6H), 7.51 (H\(_{19}\), dd, \(J = 4.5, 3.4\) Hz, 6H), 7.49 (H\(_9\), dd, \(J = 4.7, 3.5\) Hz, 6H), 7.36 (H\(_{\text{Ar}}\), br, 6H), 7.01 (H\(_{\text{Ar}}\), d, \(J = 8.4\) Hz, 12H), 6.74 (H\(_{13}\) and H\(_{11}\), d, \(J = 8.9\) Hz, 12H), 6.48 (H\(_{24}\) and H\(_{22}\), d, \(J = 9.3\) Hz, 12H).

\(^1\text{C NMR}\) (125 MHz, 298 K, CD\(_3\)CN) \(\delta: 160.5, 159.5, 157.4, 156.3, 156.1, 155.6, 155.4, 155.1, 154.3, 154.1, 152.3, 151.7, 148.8, 147.5, 147.4, 144.1, 143.6, 143.0, 142.8, 142.4, 142.2, 141.3, 140.5, 139.0, 129.7, 129.6, 128.6, 128.3, 127.6, 127.5, 126.6, 126.5, 125.8, 125.6, 125.2, 120.8 (q, \(J = 320\) Hz), 119.9. Due to overlapping peaks, 36 of the expected 39 independent peaks were observed.

Figure S37: Aromatic region of the \(^1\text{H NMR}\) spectrum (500 MHz, 298 K, CD\(_3\)CN) of \((\text{HSO}_4^-)_2 \subset \text{1-(NTf}_2^-)_10\). Minor peaks at 9.2, 8.6 and 8.5 ppm correspond to traces of unreacted 1,8-naphthyridine-2-carbaldehyde, and broadening is caused by slow rotation of phenylene rings.
Figure S38: Aromatic region of the $^{13}$C NMR spectrum (125 MHz, 298 K, CD$_3$CN) of (HSO$_4^-$)$_2$⊂ 1·(NTf$_2^-$)$_{10}$. 
Figure S39: Aromatic region of the $^1$H-$^1$H COSY spectrum (500 MHz, 298 K, CD$_3$CN) for (HSO$_4^-$)$_2 \subset 1 \cdot (N\text{TF}_2^-)_{10}$.

Figure S40: $^1$H DOSY spectrum (400 MHz, 298 K, CD$_3$CN) of (HSO$_4^-$)$_2 \subset 1 \cdot (N\text{TF}_2^-)_{10}$. We
observed a single species in the aromatic region, along with a species corresponding to the tetrabutylammonium cation in the aliphatic region (present from the TBAX salt used to template cage assembly).

Figure S41: Aromatic region of the $^1$H-$^1$C HSQC spectrum (500 MHz, 298 K, CD$_3$CN) for $(\text{HSO}_4^-)_2$⊂1·(NTf$_2^-$)$_{10}$. 
$\text{(SO}_4^{2-})_2 \subset \text{1}\cdot\text{(NTf}_2^-)_8$ \n
$^1\text{H NMR}$ (500 MHz, 298 K, CD$_3$CN) δ: 9.82 (H$_5$, s, 6H), 9.09 (H$_{30}$, dd, $J = 4.3$, 2.0 Hz, 6H), 9.02 (H$_{13}$, s, 6H), 8.99 (H$_{20}$, dd, $J = 4.7$, 1.7 Hz, 6H), 8.96 (H$_{A_r}$, d, $J = 8.2$ Hz, 6H), 8.96 (H$_{A_r}$, d, $J = 8.4$ Hz, 6H), 8.83-7.76 (H$_{A_r}$, m, 18H), 8.72 (H$_{18}$, dd, $J = 3.3$, 1.8 Hz, 6H), 8.71 (H$_{25}$, s, 6H), 8.64 (H$_{A_r}$, dt, $J = 8.4$, 1.9 Hz, 12H), 8.28 (H$_{16}$, d, $J = 8.2$ Hz, 6H), 8.05 (H$_{26}$, d, $J = 8.4$ Hz, 6H), 7.70 (H$_{29}$, dd, $J = 8.2$, 4.2 Hz, 6H), 7.50 (H$_{19}$, dd, $J = 4.2$, 3.5 Hz, 6H), 7.49 (H$_9$, dd, $J = 5.3$, 3.6 Hz, 6H), 7.36 (H$_{A_r}$, br, 6H), 7.00 (H$_{A_r}$, d, $J = 8.6$ Hz, 12H), 6.95 (H$_{A_r}$, br, 6H), 6.73 (H$_{13}$ and H$_{11}$, d, $J = 8.8$ Hz, 12H), 6.47 (H$_{24}$ and H$_{22}$, d, $J = 9.1$ Hz, 12H).

$^{13}\text{C NMR}$ (125 MHz, 298 K, CD$_3$CN) δ: 160.5, 159.5, 157.4, 156.3, 156.1, 155.6, 155.1, 154.3, 154.1, 152.3, 151.7, 148.8, 147.5, 147.4, 144.1, 143.5, 143.0, 142.8, 142.4, 142.2, 141.2, 140.5, 139.0, 129.6, 128.6, 128.3, 127.7, 127.5, 127.4, 126.5, 126.5, 125.8, 125.6, 125.2, 120.8 (q, $J = 320$ Hz), 119.6. Due to overlapping peaks, 36 of the expected 39 independent peaks were observed.

Figure S42: Aromatic region of the $^1\text{H NMR}$ spectrum (500 MHz, 298 K, CD$_3$CN) of (SO$_4^{2-}$)$_2 \subset 1\cdot$(NTf$_2^-$)$_8$. 
Figure S43: Aromatic region of the $^{13}$C NMR spectrum (125 MHz, 298 K, CD$_3$CN) of (SO$_4^{2-}$)$_2$ ⊂ 1·(NTf$_2^{-}$)$_8$. 
Figure S44: Aromatic region of the $^1$H-$^1$H COSY spectrum (500 MHz, 298 K, CD$_3$CN) for $(SO_4^{2-})_2 \subset 1\cdot(NTf_2^-)_8$.

Figure S45: Aromatic region of the $^1$H-$^{13}$C HSQC spectrum (500 MHz, 298 K, CD$_3$CN) for $(SO_4^{2-})_2 \subset 1\cdot(NTf_2^-)_8$. 
(EDS$^2$) $\subset 1\cdot$(NTf$_2$)$_{10}$

$^1$H NMR (500 MHz, 298 K, CD$_3$CN): δ 9.62 (H$_5$, d, $J = 2.60$ Hz, 6H), 9.11 (H$_{30}$, dd, $J = 4.20$, 1.76 Hz, 6H), 9.06 (H$_{20}$, d, $J = 8.26$ Hz, 6H), 9.02 (H$_{15}$, d, $J = 2.52$ Hz, 6H), 9.01 (H$_{20}$, d, $J = 2.52$ Hz, 6H), 8.96 (H$_{17}$, d, $J = 8.21$ Hz, 6H), 8.82 (H$_{27}$, d, $J = 8.34$ Hz, 6H), 8.78 (H$_8$, br, 6H), 8.73-8.69 (H$_{Ar}$, m, 12H), 8.67-8.62 (H$_{Ar}$, m, 18H), 8.59 (H$_{28}$, dd, $J = 8.37$, 1.76 Hz, 6H), 8.28 (H$_{16}$, d, $J = 8.43$ Hz, 6H), 8.05 (H$_{26}$, d, $J = 8.43$ Hz, 6H), 7.78 (H$_{29}$, dd, $J = 8.43$, 4.71 Hz, 6H), 7.51-7.47 (H$_{19}$ and H$_9$, m, 12H), 7.04 (H$_{Ar}$, d, $J = 7.77$ Hz, 12H), 6.76 (H$_{13}$ and H$_{11}$, d, $J = 8.76$ Hz, 12H), 6.49 (H$_{24}$ and H$_{22}$, d, $J = 8.76$ Hz, 12H)

$^{13}$C NMR (125 MHz, 298 K, CD$_3$CN) δ: 160.7, 160.5, 159.5, 157.9, 156.7, 156.4, 155.8, 155.5, 155.1, 154.5, 153.9, 153.6, 152.6, 152.4, 151.8, 151.2, 151.0, 148.9, 147.6, 147.4, 144.2, 143.3, 143.1, 142.4, 142.2, 142.1, 141.5, 141.1, 140.1, 139.9, 139.1, 128.7, 128.5, 128.0, 127.4, 126.5, 125.8, 125.2, 120.8 (q, $J = 320$ Hz), 119.9.

Figure S46: Aromatic region of the $^1$H NMR spectrum (500 MHz, 298 K, CD$_3$CN) of (EDS$^2$) $\subset 1\cdot$(NTf$_2$)$_{10}$.
Figure S47: Aromatic region of the $^{13}$C NMR spectrum (125 MHz, 298 K, CD$_3$CN) of (EDS$^{2-}$) $\subset 1\cdot$(NTf$_2^{-}$)$_{10}$.

Figure S48: Aromatic region of the $^1$H-$^1$H COSY spectrum (500 MHz, 298 K, CD$_3$CN) for (EDS$^{2-}$) $\subset 1\cdot$(NTf$_2^{-}$)$_{10}$.
Figure S49: Aromatic region of the $^1$H-$^{13}$C HSQC spectrum (500 MHz, 298 K, CD$_3$CN) for $(EDS^{2-}) \subset 1\cdot(NTf_2^-)_{10}$. 
Figure S50: $^1$H DOSY spectrum (400 MHz, 298 K, CD$_3$CN) for (EDS$^2^-$) $\subset$ 1·(NTf$_2^-$)$_{10}$.
\( (\text{PDS}^{2-}) \subset 1\cdot(\text{NTf}_2^-)_{10} \) where PDS is 1,3-propanedisulfonate

\(^1\text{H} \text{NMR} \) (500 MHz, 298 K, CD\(_3\)CN): \( \delta \) 9.85 (H\(_5\), s, 6H), 9.40 (H\(_{Ar}\), d, \( J = 8.07 \) Hz, 6H), 9.13 (H\(_{Ar}\), s, 6H), 9.05 (H\(_{Ar}\), s, 6H), 8.99 (H\(_{Ar}\), d, \( J = 6.1 \) Hz, 12H), 8.87-8.72 (H\(_{Ar}\), m, 30H), 8.82 (H\(_{27}\), d, \( J = 8.34 \) Hz, 6H), 8.78 (H\(_8\), br, 6H), 8.73-8.69 (H\(_{Ar}\), m, 12H), 8.67-8.62 (H\(_{Ar}\), m, 18H), 8.69-8.60 (H\(_{Ar}\), m, 18H), 8.30 (H\(_{16}\), d, \( J = 8.07 \) Hz, 6H), 8.08 (H\(_{26}\), d, \( J = 8.20 \) Hz, 6H), 7.81 (H\(_{29}\), dd, \( J = 7.28 \), 4.10 Hz, 6H), 7.55-7.31 (H\(_{Ar}\), m, 36H), 7.11-7.03 (H\(_{Ar}\), m, 24H), 6.86 (H\(_{13}\) and H\(_{11}\), d, \( J = 8.20 \) Hz, 12H), 6.56 (H\(_{14}\) and H\(_{22}\), d, \( J = 8.07 \) Hz, 12H).

\(^{13}\text{C} \text{NMR} \) (125 MHz, 298 K, CD\(_3\)CN): \( \delta \)C 159.7, 158.3, 156.5, 155.9, 155.5, 154.7, 154.5, 154.1, 153.7, 153.3, 151.4, 150.8, 150.8, 147.6, 146.6, 146.4, 143.2, 142.3, 142.2, 142.0, 141.5, 141.3, 140.1, 138.1, 127.7, 127.5, 127.4, 127.2, 127.1, 126.4, 125.7, 125.6, 125.5, 124.4, 124.3, 123.7, 120.8 (q, \( J = 320 \) Hz), 119.6. Due to overlapping peaks, 37 of the expected 39 independent peaks were observed.

Figure S51: Aromatic region of the \(^1\text{H} \text{NMR} \) spectrum (500 MHz, 298 K, CD\(_3\)CN) of \((\text{PDS}^{2-}) \subset 1\cdot(\text{NTf}_2^-)_{10}\).
**Figure S52:** Aromatic region of the $^{13}$C NMR spectrum (125 MHz, 298 K, CD$_3$CN) of (PDS$^{2-}$) $\subset 1^\cdot$(NTf$_2^-$)$_{10}$.
Figure S53: Aromatic region of the $^1$H-$^1$H COSY spectrum (500 MHz, 298 K, CD$_3$CN) for (PDS$^{2-}$) $\subset$ 1·(NTf$_2$)$_{10}$.

Figure S54: Aromatic region of the $^1$H-$^{13}$C HSQC spectrum (500 MHz, 298 K, CD$_3$CN) for (PDS$^{2-}$) $\subset$ 1·(NTf$_2$)$_{10}$. 
Figure S55: Aromatic region of the $^1$H NMR spectra (500 MHz, 298 K, CD$_3$CN) of $(\text{S}_2\text{O}_8^{2-}) \subset 1\cdot(\text{NTf}_2^-)_{10}$ at various time points. The $^1$H NMR spectra of $(\text{SO}_4^{2-})_2 \subset 1\cdot(\text{NTf}_2^-)_8$ and $(\text{HSO}_4^-)_2 \subset 1\cdot(\text{NTf}_2^-)_{10}$ are included for comparison.
Figure S56: Zoom in on two phenyl resonances of the $^1$H NMR spectra (500 MHz, 298 K, CD$_3$CN) of $\left(S_2\text{O}_8^{2-}\right) \subset 1\cdot(\text{NTf}_2^-)_{10}$ at various time points. The $^1$H NMR spectra of $\left(\text{SO}_4^{2-}\right)_{2} \subset 1\cdot(\text{NTf}_2^-)_{8}$ and $\left(\text{HSO}_4^-\right)_{2} \subset 1\cdot(\text{NTf}_2^-)_{10}$ are included for comparison.
Figure S57: Part of the aromatic region of the $^1$H NMR spectra (500 MHz, 298 K, CD$_3$CN) of $(\text{S}_2\text{O}_8^{2-})_2 \subset 1\cdot(\text{NTf}_2^-)_{10}$ at various time points. The $^1$H NMR spectra of $(\text{SO}_4^{2-})_2 \subset 1\cdot(\text{NTf}_2^-)_{8}$ and $(\text{HSO}_4^-)_2 \subset 1\cdot(\text{NTf}_2^-)_{10}$ are included for comparison.
S7 Bisulfate (HSO$_4^-$) Titration into Cage 1

Figure S58: Titration of TBAHSO$_4$ into a solution of untemplated 1 formed from AgNTf$_2$ with 1,3,5-trimethoxybenzene as an internal standard, as monitored by $^1$H NMR spectra (500 MHz, 298 K, CD$_3$CN). Each sample with a different number of equivalents of TBAHSO$_4$ was prepared in a separate NMR tube. Samples were equilibrated over 16 h at room temperature. We were unable to observe the hydrogen bonded hydrogen expected in a bisulfate dimer, and presume that it has broadened into the baseline in our system.
Figure S59: Formation of $(\text{HSO}_4^-)_2 \subset 1\cdot(\text{NTf}_2^-)_{10}$ as a function equivalents of TBAHSO$_4$ added.
S8 X-ray Crystallography

Data were collected at Beamline I19 of Diamond Light Source employing silicon double crystal monochromated synchrotron radiation (0.6889 Å) with ω and ψ scans at 100(2) K. Data integration and reduction were undertaken with Xia2.2-4 Subsequent computations were carried out using the WinGX-32 graphical user interface.5 Multi-scan empirical absorption corrections were applied to the data using the AIMLESS6 tool in the CCP4 suite.7 The structures were solved by direct methods using SHELXT8 then refined and extended with SHELXL.9 In general, non-hydrogen atoms with occupancies greater than 0.5 were refined anisotropically. Carbon-bound hydrogen atoms were included in idealised positions and refined using a riding model. Disorder was modelled using standard crystallographic methods including constraints, restraints and rigid bodies where necessary. Crystallographic data along with specific details pertaining to the refinement follow. Crystallographic data have been deposited with the CCDC (1913631-1913635).

\[
[(PF_6)_{1.65}(OTf)_{0.35}Ag_{12}L_6\cdots9.35PF_6\cdots0.65OTf\cdots2.25MeCN [+ solvent]]
\]

Formula C_{275.50}H_{186.75}Ag_{12}F_{69}N_{62.25}O_{3}P_{11}S, M 7395.28, Triclinic, space group P -1 (#2), a 23.3103(3), b 27.5339(3), c 34.0879(4) Å, α 87.7970(10), β 79.3500(10), γ 70.8740(10)°, V 20308.9(4) Å³, D c 1.209 g cm⁻³, Z 2, crystal size 0.050 by 0.040 by 0.030 mm, color purple, habit block, temperature 100(2) Kelvin, λ(Synchrotron) 0.6889 Å, μ(Synchrotron) 0.628 mm⁻¹, T(Analytical)min,max 0.97689755547, 1.0, 2θmax 48.42, hkl range -27 27, -29 32, -40 38, N 192935, Nind 69422(Rmerge 0.0607), Nobs 38737(I > 2σ(I)), Nvar 3934, residuals* R1(F) 0.0845, wR2(F²) 0.2943, GoF(all) 1.030, Δρmin,max -1.303, 1.274 e⁻ Å⁻³.

*R1 = Σ||Fo| - |Fc||/Σ|Fo| for Fo > 2σ(Fo); wR2 = (Σw(Fo² - Fc²)²/Σ(wFc²)²)¹/2 all reflections w=1/[σ²(Fo²)+(0.1860P)²] where P=(Fo²+2Fc²)/3

Specific refinement details:

The crystals of \([(PF_6)_{1.65}(OTf)_{0.35}Ag_{12}L_6\cdots9.35PF_6\cdots0.65OTf\cdots2.25MeCN [+ solvent]]\) were grown by diffusion of diethyl ether into an acetonitrile solution of \([Ag_{12}L_6\cdots12OTf]\) containing excess Bu₄NPF₆. The crystals employed immediately lost solvent after removal from the mother liquor and rapid handling prior to flash cooling in the cryostream was required to collect data. Data were obtained to 0.84 Å resolution. The asymmetric unit was found to contain one complete Ag₁₂L₆ assembly and associated counterions and solvent molecules.

Bond lengths and angles within pairs of chemically identical organic ligands were restrained to be similar to each other and additional bond length restraints (DFIX) were applied to some sections of the organic ligands. Thermal parameter restraints (SIMU, RIGU) were applied to all atoms except for silver to facilitate anisotropic stable refinement.
One of the encapsulated anions was modelled as a full occupancy PF$_6^-$.

The other one was modelled as a disordered mixture of PF$_6^-$ and OTf$^-$ with occupancies of 0.65 and 0.35 respectively; these occupancies were initially refined and then fixed at the obtained values. The other anions within the structure also show evidence of substantial disorder. A further anion lattice site was modelled as a disordered mixture of PF$_6^-$ and OTf$^-$ and three PF$_6^-$ anions were modelled as disordered over two locations. The occupancies of the all the located anions were allowed to refine freely and then fixed at the obtained values. Some additional minor occupancy positions of the anions could not be located in the electron density map and were not included in the model resulting in a discrepancy of 2.5 anions per Ag$_{12}$L$_6$ assembly; these anions are given as PF$_6^-$ in the formula given above. Some lower occupancy disordered atoms were modelled with isotropic thermal parameters bond length and thermal parameter restraints were applied to facilitate realistic modelling of the disordered anions. Some acetonitrile solvent molecules were also modelled with partial occupancy.

Further reflecting the solvent loss there is a significant amount of void volume in the lattice containing smeared electron density from disordered solvent (and potentially the unresolved anions). Consequently the SQUEEZE$^{10}$ function of PLATON$^{11}$ was employed to remove the contribution of the electron density associated with this highly disordered solvent which gave a potential solvent accessible void of 7729 Å$^3$ per unit cell (a total of approximately 2273 electrons). Since the diffuse solvent molecules could not be assigned conclusively to acetonitrile or diethyl ether they were not included in the formula. Consequently, the molecular weight and density given above are likely to be slightly underestimated. The remaining electron density peaks and holes (up to 1.274 or $-1.303$ eÅ$^3$) are close to the silver centers reflecting absorption effects or a small amount of unresolved disorder.

CheckCIF gives thirteen B level alerts, all resulting from thermal motion and/or unresolved disorder of the anions as described above.

$$\left[\text{ClO}_4\right]_2\text{Ag}_{12}\text{L}_6\cdot10\text{NTf}_2\cdot2.5\text{MeCN} [+\text{ solvent}]$$

Formula C$_{295}$H$_{187.5}$Ag$_{12}$Cl$_2$F$_{60}$N$_{72.5}$O$_{48}$S$_{20}$, $M$ 8662.20, Triclinic, space group P -1 (#2), $a$ 24.5951(5), $b$ 25.8919(4), $c$ 33.0285(4) Å, $\alpha$ 82.2940(10), $\beta$ 80.7880(10), $\gamma$ 67.0800(10)$^\circ$, $V$ 19063.0(6) Å$^3$, $D_c$ 1.509 g cm$^{-3}$, $Z$ 2, crystal size 0.050 by 0.040 by 0.030 mm, colour purple, habit block, temperature 100(2) Kelvin, $\lambda$(Synchrotron) 0.6889 Å, $\mu$(Synchrotron) 0.748 mm$^{-1}$, $T$(Analytical)$\text{min, max}$ 0.976600983573, 1.0, $2\theta_{\text{max}}$ 42.52, $hkl$ range -25 25, -27 27, -33 31, $N_{\text{ind}}$ 44821($R_{\text{merge}}$ 0.0701), $N_{\text{obs}}$ 23407($I\geq2\sigma(I)$), $N_{\text{var}}$ 3556, residuals* $R1(F)$ 0.0919, $wR2(F^2)$ 0.3151, GoF(all) 1.010, $\Delta\rho_{\text{min, max}}$ -0.894, 0.902 eÅ$^{-3}$.

* $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ for $F_o > 2\sigma(F_o)$, $wR2 = (\Sigma w(F_o^2 - F_c^2)^2/\Sigma(wF_c^2)^2)^{1/2}$ all reflections

$w=1/[\sigma^2(F_o^2)+(0.2000P)^2]$ where $P=(F_o^2+2F_c^2)/3$
Specific refinement details:

The crystals of \([(\text{ClO}_4)_2\text{Ag}_{12}\text{L}_6]:10\text{NTf}_2\cdot2.5\text{MeCN}\) were grown by diffusion of diisopropyl ether into an acetonitrile solution of the complex formed via the procedure described above. The crystals employed immediately lost solvent after removal from the mother liquor and rapid handling prior to flash cooling in the cryostream was required to collect data. Despite these measures and the use of synchrotron radiation few reflections at greater than 0.95 Å resolution were observed and the data were trimmed accordingly. Nevertheless, the quality of the data is far more than sufficient to establish the connectivity of the structure. The asymmetric unit was found to contain one complete Ag_{12}L_6 assembly and associated counterions and solvent molecules.

Due to the limited resolution, bond lengths and angles within pairs of chemically identical organic ligands were restrained to be similar to each other and additional restraints (DFIX, FLAT) were applied to some sections of the organic ligands. Thermal parameter restraints (SIMU, RIGU) were applied to all atoms except for silver to facilitate anisotropic stable refinement. Even with these restraints some thermal parameters remain larger than ideal as a consequence of the high level of thermal motion or minor unresolved disorder present throughout the structure.

Both encapsulated ClO_4^- anions were modelled as disordered over two locations. These disordered ClO_4^- anions were restrained to have an idealised tetrahedral geometry and the disordered oxygen atoms were modelled with isotropic thermal parameters. The other resolved anions within the structure were all identified as triflimide and also show evidence of substantial disorder. Two triflimide anions were modelled as disordered over two locations and all triflimide anions were modelled with partial occupancy. The occupancies of the triflimide anions were allowed to refine freely and then fixed at the obtained values. These anions were modelled with isotropic thermal parameters and bond length and thermal parameter restraints were applied to facilitate realistic modelling of the disordered anions. Some acetonitrile solvent molecules were also modelled with partial occupancy.

A further 8.3 anions per Ag_{12}L_6 assembly remain unaccounted for and no satisfactory model for these anions could be obtained despite numerous attempts at modelling, including with rigid bodies. Therefore the SQUEEZE\textsuperscript{10} function of PLATON\textsuperscript{11} was employed to account for the highly disordered anions and further disordered solvent molecules, which gave a potential solvent accessible void of 7120 Å\textsuperscript{3} per unit cell (a total of approximately 2727 electrons). These anions are included as triflimide in the formula given above. Since the diffuse solvent molecules could not be assigned conclusively to acetonitrile or diethyl ether they were not included in the formula. Consequently, the molecular weight and density given above are likely to be underestimated.

CheckCIF gives three A and five B level alerts. These alerts (both A and B level) all result from the limited data resolution, thermal motion and/or unresolved disorder of some anions and solvent molecules and the generally high level of thermal motion present throughout the structure as described above.
[(SO₄H)₂Ag₁₂L₆·10NTf₂·13MeCN·1.5iPr₂O \ [+ solvent]

Formula C₃₂₅H₂₄₂Ag₁₂F₆₀N₈₃O₄₉.₅₀S₂₂,  \( M = 9241.76 \), Monoclinic, space group \( C 2/c \ (\#15) \), \( a = 43.3421(4) \), \( b = 32.8367(2) \), \( c = 27.6735(2) \) \( \text{Å} \), \( \beta = 93.2690(10) \), \( V = 39321.2(5) \) \( \text{Å}^3 \), \( D_c = 1.561 \text{ g cm}^{-3} \), \( Z = 4 \), crystal size 0.030 by 0.020 by 0.010 mm, colour purple, habit block, temperature 100(2) Kelvin, \( \lambda \) (Synchrotron) 0.6889 \( \text{Å} \), \( \mu \) (Synchrotron) 0.729 \( \text{mm}^{-1} \), \( T \) (Analytical)min,max 0.973805583854, 1.0, \( 2\theta_{\text{max}} \) 53.15, \( hkl \) range -52 56, -41 42, -35 33, \( N_{\text{obs}} \) 26507(I > 2\( \sigma(I) \)), \( N_{\text{var}} \) 2742, residuals* \( R1(F) \) 0.0888, \( wR2(F^2) \) 0.3047, GoF(all) 1.117, \( \Delta \rho_{\text{min,max}} \) -1.989, 1.565 e\( \text{-} \)Å\(^{-3}\).

*\( R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o| \) for \( F_o > 2\sigma(F_o) \); \( wR2 = (\Sigma w(F_o^2 - F_c^2)^2/\Sigma(wF_c^2)^2)^{1/2} \) all reflections
\( w=1/[(\sigma^2(F_o^2)+(0.2000P)^2)] \) where \( P=(F_o^2+2F_c^2)/3 \)

Specific refinement details:

The crystals of \([\text{SO}_4\text{H}]_2\text{Ag}_{12}\text{L}_6\cdot10\text{NTf}_2\cdot13\text{MeCN}\cdot1.5\text{iPr}_2\text{O} \) were grown by diffusion of diisopropyl ether into an acetonitrile solution of the complex. The crystals employed immediately lost solvent after removal from the mother liquor and rapid handling prior to flash cooling in the cryostream was required to collect data. Even so data were obtained to 0.77 \( \text{Å} \) resolution by employing synchrotron radiation. The asymmetric unit was found to contain one half of a \( \text{Ag}_{12}\text{L}_6 \) assembly and associated counterions and solvent molecules.

Bond lengths and angles within pairs of chemically identical organic ligands were restrained to be similar to each other. Thermal parameter restraints (SIMU, RIGU) were applied to all atoms except for silver to facilitate anisotropic stable refinement. One naphthyridine arm was modelled as disordered over two locations with bond length (DFIX) and FLAT restraints were applied to the disordered parts to ensure a stable refinement.

The oxygen atoms of the central \( \text{HSO}_4^- \) anion was modelled as disordered over three locations with refined occupancies of 0.35068/0.31665/0.33267. Soft bond length and angle restraints were applied to the disordered atoms to achieve a reasonable refinement. While the distance between the disordered \( \text{HSO}_4^- \) anion and its symmetry equivalent is consistent with the presence of a hydrogen bonded dimer,\(^{12}\) the hydrogen atom of the \( \text{HSO}_4^- \) could not be adequately resolved due to the extensive disorder precluding detailed geometric analysis of the hydrogen bonding. One hydrogen atom was placed on one of the disordered locations, based on a peak in the electron density map with a DFIX restraint applied to the OH bond length and a soft restraint applied to the D-A distance; however the position of this hydrogen is subject to a high degree of uncertainty.

The other anions within the structure, all modelled as triflimide, also show evidence of substantial disorder. All five triflimide anions were modelled as disordered over two or three locations. The occupancies of the disordered anions were allowed to refine freely and then
fixed at the obtained values. Some additional minor occupancy positions of the anions could not be located in the electron density map and were not included in the model resulting in a discrepancy of 1.1 counterions per asymmetric unit. Some lower occupancy disordered atoms were modelled with isotropic thermal parameters and bond length and thermal parameter restraints were applied to facilitate realistic modelling of the disordered anions. Some acetonitrile solvent molecules were also modelled with as disordered over multiple locations and/or with partial occupancy. The hydrogen atoms of some disordered acetonitrile molecules could not be located in the electron density map and were not included in the model.

Further reflecting the solvent loss there is a significant amount of void volume in the lattice containing smeared electron density from disordered solvent (and potentially the unresolved anions). Consequently the SQUEEZE$^{10}$ function of PLATON$^{11}$ was employed to remove the contribution of the electron density associated with this highly disordered solvent which gave a potential solvent accessible void of 1132 Å$^3$ per unit cell (a total of approximately 401 electrons). Since the diffuse solvent molecules could not be assigned conclusively to acetonitrile or diisopropyl ether they were not included in the formula. Consequently, the molecular weight and density given above are likely to be slightly underestimated. The remaining electron density peaks and holes (up to 1.565 or $-$1.990 e Å$^{-3}$) are close to the silver centers reflecting absorption effects or a small amount of unresolved disorder.

CheckCIF gives six B level alerts, all resulting from acetonitrile solvent molecules for which hydrogens were not modelled as described above.

$$[(\text{EDS}^2) \subseteq \text{Ag}_{12}\text{L}_6] \cdot 10\text{NTf}_2\cdot 18\text{MeCN}\cdot 1.5\text{iPr}_2\text{O}\cdot 0.5\text{H}_2\text{O}$$

Formula C$_{337}$H$_{260}$Ag$_{12}$F$_{60}$N$_{88}$O$_{48}$S$_{22}$, $M$ 9450.07, Monoclinic, space group C 2/c (#15), $a$ 43.6194(3), $b$ 32.4028(3), $c$ 27.3509(2) Å, $\beta$ 92.5640(10), $V$ 38618.8(5) Å$^3$, $D_c$ 1.625 g cm$^{-3}$, $Z$ 4, crystal size 0.050 by 0.040 by 0.040 mm, colour purple, habit block, temperature 100(2) Kelvin, $\lambda$(Synchrotron) 0.6889 Å, $\mu$(Synchrotron) 0.744 mm$^{-1}$, $T$(Analytical)$_{\text{min,max}}$ 0.939023710754, 1.0, $2\theta_{\text{max}}$ 58.95, $hkl$ range -51 62, -42 43, -36 39, $N$ 157325, $N_{\text{ind}}$ 53025($R_{\text{merge}}$ 0.0452), $N_{\text{obs}}$ 32723($I > 2\sigma(I)$), $N_{\text{var}}$ 3188, residuals* $R(1(F))$ 0.0666, $wR2(F^2)$ 0.2239, GoF(all) 1.014, $\Delta\rho_{\text{min,max}}$ -1.134, 0.937 e- Å$^{-3}$.

* $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ for $F_o > 2\sigma(F_o)$, $wR2 = (\Sigma w(F_o^2 - F_c^2)^2/\Sigma(wF_c^2)^2)^{1/2}$ all reflections

$w=1/[(\sigma^2(F_o^2)+(0.1443P)^2)]$ where $P=(F_o^2+2F_c^2)/3$

Specific refinement details:

The crystals of $[(\text{EDS}^2) \subseteq \text{Ag}_{12}\text{L}_6] \cdot 10\text{NTf}_2\cdot 18\text{MeCN}\cdot 1.5\text{iPr}_2\text{O}\cdot 0.5\text{H}_2\text{O}$ were grown by diffusion of diisopropyl ether into an acetonitrile solution of the complex formed via the procedure described above. The crystals employed immediately lost solvent after removal from the mother liquor and rapid handling prior to flash cooling in the cryostream was required to
collect data. Even so data were obtained to 0.7 Å resolution by employing synchrotron radiation. The asymmetric unit was found to contain one half of a Ag_{12}L_{6} assembly and associated counterions and solvent molecules.

Bond lengths and angles within pairs of chemically identical organic ligands were restrained to be similar to each other. Thermal parameter restraints (SIMU, RIGU) were applied to all atoms except for silver to facilitate anisotropic stable refinement. One naphthyridine arm was modelled as disordered over two locations with bond length (DFIX) and FLAT restraints were applied to the disordered parts to ensure a stable refinement; one of the silver centers coordinated to the disordered ligand arm was also modelled as disordered over two locations.

The carbon atoms of the encapsulated ethanedisulfonate anion were modelled as disordered over a special position with symmetry enforced half occupancy. The other anions within the structure, all modelled as triflimide, also show evidence of substantial disorder. All triflimide anions were modelled as disordered over two or three locations. Some lower occupancy disordered atoms were modelled with isotropic thermal parameters and bond length and thermal parameter restraints were applied to facilitate realistic modelling of the disordered anions. Some acetonitrile solvent molecules were also modelled with as disordered over multiple locations and/or with partial occupancy. The hydrogen atoms of some disordered acetonitrile molecules and the water molecule could not be located in the electron density map and were not included in the model.

CheckCIF gives sixteen B level alerts, all resulting from thermal motion and/or unresolved disorder of the anions and solvent molecules and solvent molecules for which hydrogens were not modelled as described above.

\[(S_{2}O_{8})\text{Ag}_{12}L_{6}\cdot10NTf_{2}\cdot12MeCN [+ solvent]\]

Formula C_{314}H_{216}Ag_{12}F_{60}N_{82}O_{48}S_{22}, M = 9045.44, Monoclinic, space group C 2/c (#15), a = 42.8029(7), b = 32.7310(4), c = 27.8538(5) Å, β = 93.4120(10), V = 38953.5(11) Å³, Dc = 1.542 g cm⁻³, Z = 4, crystal size 0.100 by 0.100 by 0.030 mm, colour purple, habit block, temperature 100(2) Kelvin, λ(Synchrotron) = 0.6889 Å, μ(Synchrotron) = 0.734 mm⁻¹, T(Analytical)_{min,max} = 0.966892968533, 1.0, 2θ_{max} = 47.81, hkl range -50 50, -38 36, -32 31, N = 128026, N_{ind} = 32151(R_{merge} = 0.1067), N_{obs} = 20558(I > 2σ(I)), N_{var} = 2633, residuals* R1(F) = 0.0872, wR2(F²) = 0.2907, GoF(all) = 1.044, Δρ_{min,max} = -1.064, 1.340 e⁻ Å⁻³.

*R1 = Σ||F_o| - |F_c||/Σ|F_o| for F_o > 2σ(F_o); wR2 = (Σw(F_o² - F_c²)²/Σ(wF_c²)²)⁰.⁵ all reflections

w = 1/[σ²(F_o²) + (0.1970P)²] where P = (F_o² + 2F_c²)/3

Specific refinement details:

The crystals of [(S_{2}O_{8})\text{Ag}_{12}L_{6\cdot10NTf_{2}\cdot12MeCN [+ solvent]} were grown by diffusion of diisopropyl ether into an acetonitrile solution of the complex formed via the procedure
described above. The crystals employed immediately lost solvent after removal from the mother liquor and rapid handling prior to flash cooling in the cryostream was required to collect data. Even so data were obtained to 0.85 Å resolution by employing synchrotron radiation. The asymmetric unit was found to contain one half of a Ag\textsubscript{12}L\textsubscript{6} assembly and associated counterions and solvent molecules.

Bond lengths and angles within pairs of chemically identical organic ligands were restrained to be similar to each other. Thermal parameter restraints (SIMU, RIGU) were applied to all atoms except for silver to facilitate anisotropic stable refinement. One napthyridine arm was modelled as disordered over two locations with bond length (DFIX) and FLAT restraints were applied to the disordered parts to ensure a stable refinement.

The central O-O linkage of the encapsulated peroxydisulfate anion was modelled as disordered over a special position with symmetry enforced half occupancy. The remaining oxygen atoms of this peroxydisulfate anion also show evidence of thermal motion or minor unresolved disorder which could not be adequately modelled with discrete atom positions. The other anions within the structure, all modelled as triflimide, also show evidence of substantial disorder. Four triflimide anions were modelled as disordered over two or three locations and the remaining triflimide was modelled with partial occupancy. The occupancies of the disordered anions were allowed to refine freely and then fixed at the obtained values. Some additional minor occupancy positions of the anions could not be located in the electron density map and were not included in the model resulting in a discrepancy of 1.45 counterions per asymmetric unit. Some lower occupancy disordered atoms were modelled with isotropic thermal parameters and bond length and thermal parameter restraints were applied to facilitate realistic modelling of the disordered anions. Some acetonitrile solvent molecules were also modelled with as disordered over multiple locations and/or with partial occupancy. The hydrogen atoms of some disordered acetonitrile molecules could not be located in the electron density map and were not included in the model.

Further reflecting the solvent loss there is a significant amount of void volume in the lattice containing smeared electron density from disordered solvent (and potentially the unresolved anions). Consequently the SQUEEZE\textsuperscript{10} function of PLATON\textsuperscript{11} was employed to remove the contribution of the electron density associated with this highly disordered solvent which gave a potential solvent accessible void of 4358 Å\textsuperscript{3} per unit cell (a total of approximately 1130 electrons). Since the diffuse solvent molecules could not be assigned conclusively to acetonitrile or diethyl ether they were not included in the formula. Consequently, the molecular weight and density given above are likely to be slightly underestimated.

CheckCIF gives one A level and six B level alerts, all resulting from thermal motion and/or unresolved disorder of the anions and solvent molecules.
Adaption of Cage 1 in the Solid State

The following is an example of the method used to investigate the changes in cage 1 in the solid state for a range of different encapsulated anions. Each vertex of the prism was defined by the centroid between its two silver centers. All measurements were the made from these centroids.

Figure S60: An example of distance measurements for $(\text{ClO}_4^-)_2 \subset 1$, with centroids as red dots.

In order to measure the dihedral angles, centroids were calculated for each of the individual disilver centers. Centroids were then calculated between each of the three disilver complexes which define the triangular aperture of the structure. The dihedral angle was then measured from opposing disilver centers through these two central centroids.

Figure S61: An example of torsion angle measurements for $(\text{ClO}_4^-)_2 \subset 1$ made in Mercury. (a) Top-down view of $(\text{ClO}_4^-)_2 \subset 1$ (b) Side-on view of $(\text{ClO}_4^-)_2 \subset 1$. 
The results of the measurements and their averaged values are detailed in Table S1.

**Table S1**: Measurements and averaged values for \((\text{ClO}_4^-)_2 \subset 1\).

| Measurement                  | Ag···Ag in Disilver Centre | Disilver···Disilver in Triangular Face | Disilver···Disilver Along Length | Torsion Angle |
|------------------------------|-----------------------------|---------------------------------------|---------------------------------|--------------|
| Units                        | (Å)                         | (Å)                                   | (Å)                            | (°)          |
| 2.83                         | 12.799                      | 12.063                                | 29.05                          |
| 2.861                        | 12.664                      | 11.882                                | 30.16                          |
| 2.823                        | 12.57                       | 12.004                                | 30.26                          |
| 2.816                        | 12.919                      |                                       |                                |
| 2.826                        | 12.817                      |                                       |                                |
| 2.835                        | 12.615                      |                                       |                                |
| **Average**                  | **2.83 ± 0.02**             | **12.7 ± 0.1**                        | **11.98 ± 0.06**                | **30 ± 1**   |

In order to calculate the area of the triangular apertures at the end of the triangular antiprism one of the three internal angles of the triangular apertures were measured from the centroids generated for the disilver vertices.

**Figure S62**: Measurement of the three angles which define one of the triangular faces of \((\text{ClO}_4^-)_2 \subset 1\).

The area was calculated according to the formula

\[
\text{Area} = \frac{1}{2} a b \sin(C)
\]

where a and b are the lengths two of the faces of the triangular face and C is the angle defined by these two lines. This calculation was carried out for the two triangular faces of the prism.
**Table S2:** Calculation of the average area of the triangular apertures of \((\text{ClO}_4^-)_2 \subset 1\).

| Cage | Distance \(a\) | Distance \(b\) | Angle \(C\) | Area = \(a \cdot b \cdot \sin(C)\) |
|------|----------------|----------------|-------------|---------------------------------|
| Units | (Å)            | (Å)            | (°)         | (Å²)                            |
|       | 12.799         | 12.57          | 59.88       | 69.58                           |
|       | 12.615         | 12.817         | 61.05       | 70.74                           |
| **Average** |                 |                |             | **70.2 ± 0.5**                  |

Measurements for the four other crystallographic datasets were calculated in a similar manner. The results of these calculates are tabulated in Table S3.

**Table S3:** Calculated average lengths, torsion angles and area of the triangular apertures for cages \((X)_n \subset 1\).

| Measurement | Ag···Ag in disilver centre | Disilver···Disilver in Triangular Face | Disilver···Disilver Along Length | Torsion angle | Triangular Aperture Area |
|-------------|-----------------------------|--------------------------------------|----------------------------------|---------------|-------------------------|
| Units       | (Å)                         | (Å)                                  | (Å)                              | (°)           | (Å²)                    |
| \((\text{ClO}_4^-)_2 \subset 1\) | 2.83 ± 0.02                  | 12.7 ± 0.1                           | 11.98 ± 0.06                    | 30 ± 1        | 70.2 ± 0.5               |
| \((\text{PF}_6^-)_2 \subset 1\) | 2.84 ± 0.01                  | 13.3 ± 0.2                           | 11.86 ± 0.06                    | 31 ± 0.9      | 76.3 ± 0.5               |
| \((\text{HSO}_4^-)_2 \subset 1\) | 2.85 ± 0.02                  | 12.6 ± 0.1                           | 11.96 ± 0.09                    | 28 ± 2        | 68.4 ± 0.4               |
| \((\text{EDS}^{2-})_2 \subset 1\) | 2.85 ± 0.02                  | 12.4 ± 0.1                           | 12.04 ± 0.05                    | 27.9 ± 0.8    | 66.9 ± 0.3               |
| \((\text{S}_2\text{O}_8^-)_2 \subset 1\) | 2.86 ± 0.03                  | 12.6 ± 0.2                           | 11.9 ± 0.07                     | 27.5 ± 1      | 68.5 ± 0.4               |
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