Supporting information for:

**Guest-induced Crystal-to-crystal Expansion and Contraction of a 3-D Porous Coordination Polymer**

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1. General Methods

Elemental analyses were performed by the Campbell Microanalytical Laboratory at the University of Otago. Infrared spectra were recorded on a PerkinElmer Spectrum BX FT-IR in Nujol using NaCl windows. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) was performed on a PerkinElmer STA 6000 Simultaneous Thermal Analyzer under ambient conditions using an aluminium pan inside a platinum crucible. Unless otherwise stated, NMR spectra were recorded on a Varian Gemini 300 MHz spectrometer at 23°C using a 5 mm probe. \(^1\)H NMR spectra of digested samples were performed in CD\(_3\)CN.

Unless otherwise stated, all chemicals were obtained from commercial sources and used as received. Tetrahydrofuran was dried by standard literature procedures\(^1\) and distilled fresh from sodium/benzophenone. Diisopropylamine was freshly distilled from potassium hydroxide. Di-2-pyrazinyl methane (dpzm) was prepared according to the methods described in literature.\(^2\)

Sample Preparation for Analysis

Due to the volatile nature of the solvent in the pores of 1, preparation of the material for analysis was carried by drying the crystals on a paper towel (1\(^{as}\) – crystals up to 2-3 mm) or by N\(_2\) flow (1\(^{dem}\) and 1\(^{ace}\)) which ensured minimal solvent loss from the pores. This method of sample preparation was consistent for all of the analyses performed.

2. Synthesis

[Ag(dpzm)]ClO\(_4\)·1.1(DMSO)·0.9(EtOAc), 1\(^{as}\)

In a typical experiment, dpzm (61.3 mg, 0.36 mmol) was combined with the silver perchlorate (74.0 g, 0.36 mmol) and dissolved in DMSO (1 mL). This mixture was divided evenly between five separate vials. Slow vapour diffusion of EtOAc into the DMSO solution gave large colourless to pale yellow block-shaped crystals after 3 weeks. The crystals were isolated and dried at room temperature (~23 mg, 66% per vial – calculated from the formula C\(_9\)H\(_8\)N\(_4\)AgClO\(_4\)·1.2DMSO). \(\nu_{\text{max}}\) (nujol, cm\(^{-1}\)): 1048 (s, S=O), 1094 (s, Cl-O), 1407, (s, CH\(_2\)), 1520 (w, C=C), 1594 (w, C=C), 1747 (s, C=O). Found: C 28.7, H 3.2, N 11.6; C\(_9\)H\(_8\)N\(_4\)AgClO\(_4\)·1.2C\(_2\)H\(_6\)SO requires: C 28.9, H 3.2 N 11.8%. \(^1\)H NMR (300 MHz/CD\(_3\)CN):
δ 1.20 (t, 3H, CH₃EtOAc), 1.97 (s, 3H, CH₃EtOAc), 2.50 (s, 3H, CH₃DMSO) 4.06 (q, 2H, CH₂EtOAc), 4.36 (s, 2H, H1a/H1b), 8.49 (dd, 2H, H5/H5’), 8.53 (d, 2H, H6/H6’), 8.73 (d, 2H, H3/H3’).

formula, based on ¹H NMR spectroscopy: [Ag(dpzm)]ClO₄·1.1(DMSO)·0.9(EtOAc).

[Ag(dpzm)]ClO₄, 2

Silver perchlorate (52.7 mg, 0.254 mmol) in methanol (2 mL) was added dropwise to dpzm (65.6 mg, 0.381 mmol) in methanol (2 mL). Immediately a white precipitate formed, and after standing for 1 h, was isolated under reduced pressure. The pale white solid was washed with ether and dried under vacuum (94.0 mg). A portion of the precipitate (32.4 mg) was dissolved in acetonitrile/water (1:8) and the mixture was allowed to slowly evaporate until large rod-like crystals formed in the bottom of the vial. The crystals were washed with ether and dried under vacuum (15.0 mg). νₓₓ max (nujol, cm⁻¹): 1074 (s, Cl-O), 1406, (s, CH₂), 1519 (w, C=C), 1594 (w, C=C). Found C 28.7, H 2.1, N 14.8, C₉H₈N₄ClO₄Ag requires: C 28.5, H 2.1, N 14.8%.

Caution! Whilst no problems were encountered in the course of this work, perchlorate salts are strong oxidising agents and are potentially explosive and should be handled on a small scale with appropriate care.

3. Guest-induced breathing

General procedure for crystal-to-crystal expansion/contraction

Crystals of 1ˢˢ (up to several millimetres in size) were manually selected from the ethyl acetate/DMSO mixture using tweezers and transferred to a clean vial containing the desired solvent in a swift manner. Upon exposure to dichloromethane, audible “popping” sounds were heard, accompanied by splintering of the original crystal into many smaller crystals. Thereafter, exposure to different solvents did not result in further crystal breakdown. The crystals were left in the desired solvent for 2 days with 3 washes performed during this period. For the procedure of crystal-to-crystal expansion and contraction (Scheme SI 1), the ¹H NMR spectra of the digested samples (Figure SI 1), and the IR spectra of the crystalline samples (Figure SI 2) are given below.
Scheme SI 1. solvent induced crystal-to-crystal expansion/contraction of 1. After every solvent exchange experiment, unit cell determinations were performed and a portion of the material was digested in CD$_3$CN for $^1$H NMR spectroscopy.

Figure SI 1. $^1$H NMR spectra (300 MHz/CD$_3$CN) of crystals of 1 subjected to solvent cycles as described in scheme SI 1; (a) 1$^{as}$; (b) 1$^{dcm(1)}$; (c) 1$^{ace(1)}$; (d) 1$^{dcm(2)}$; (e) 1$^{ace(2)}$. Solvent exchange was performed in the order indicated (a) to (e) (see, scheme SI 1).
Figure SI 2. IR spectra (nujol) of (a) $1^a$, (b) $1^{dcm}$, (c) $1^{ace}$. 
Spectral data of 1_{dcm(1)} and 1_{ace(1)}

1_{dcm(1)}: $^1$H NMR (300 MHz/CD$_3$CN): δ 4.35 (s, 2H, H1a/H1b), 5.45 (s, 2H, CH$_2^{DCM}$), 8.48 (dd, 2H, H5/H5'), 8.51 (d, 2H, H6/H6'), 8.71 (d, 2H, H3/H3'), formula, based on $^1$H NMR spectroscopy: [Ag(dpzm)]ClO$_4$·1.2(CH$_2$Cl$_2$). ν$_{max}$ (nujol, cm$^{-1}$): 733(s, C-Cl), 1091 (s, Cl-O), 1269 (w, C-H), 1408 (s, CH$_2$), 1521 (w, C=C), 1596 (w, C=C)

1_{ace(1)}: $^1$H NMR (300 MHz/CD$_3$CN): δ 2.09 (s, 3H, CH$_3$ Acetone), δ 4.36 (s, 2H, H1a/H1b), 8.48 (dd, 2H, H5/H5'), 8.52 (d, 2H, H6/H6'), 8.71 (d, 2H, H3/H3'), formula, based on $^1$H NMR spectroscopy: [Ag(dpzm)]ClO$_4$·1.4(CH$_3$)$_2$CO. ν$_{max}$ (nujol, cm$^{-1}$): 1092 (s, Cl-O), 1522 (w, C=C), 1408 (s, CH$_2$), 1596 (w, C=C), 1707 (C=O).

The $^1$H NMR spectra of 1_{dcm(2)} and 1_{ace(2)} were essentially unchanged, giving the formulae [Ag(dpzm)]ClO$_4$·1.3(CH$_2$Cl$_2$) and [Ag(dpzm)]ClO$_4$·1.5(CH$_3$)$_2$CO respectively. See Figure SI 1 showing the $^1$H NMR spectra of 1_{dcm(2)} and 1_{ace(2)}.

4. Crystal to crystal transformation

Large crystals of 1_{as} were selected and deposited on a glass slide containing paratone oil such that the crystals were completely immersed (Figure SI 3). Some large crystals were crushed into smaller ones. The slide was stored in the dark for 1-2 weeks. After this period, most of the crystals had split into small crystalline rods consistent with the structure of 2 (Figure SI 4). This was confirmed by selecting several crystals for unit cell determinations and the digestion of the converted samples were analysed by $^1$H NMR spectroscopy (Figure SI 5).
**Figure SI 3.** Typical examples of crystals of 1\( ^{\text{st}} \) in paratone-N, some of which have been broken into smaller pieces.

**Figure SI 4.** Typical examples of crystals of 1\( ^{\text{st}} \) that have been transformed into the 2D structure 2.
Figure SI 5. $^1$H NMR spectra (300 MHz/CD$_3$CN): a) sample of 1$^{\text{as}}$ after immersion in paratone-N for 6 weeks, showing complete solvent loss consistent with compound 2. b) 1$^{\text{as}}$.

5. X-ray crystallography

*General methods*

Crystals were mounted in paratone-N oil on a plastic loop. X-ray diffraction data were collected with (i) Mo-K$\alpha$ radiation ($\lambda = 0.7107$ Å) using an Oxford Diffraction X-calibur single crystal X-ray diffractometer at 150(2) K. Data sets were corrected for absorption using a multi-scan method, and structures were solved by direct methods using SHELXS-97$^3$ and refined by full-matrix least squares on $F^2$ by SHELXL-97,$^4$ interfaced through the program X-Seed.$^5$ In general, all non-hydrogen atoms were refined anisotropically and hydrogen atoms were included as invariants at geometrically estimated positions, unless specified otherwise in additional details below. Figures were produced using the program CrystalMaker.$^6$ Publication materials were prepared using CIFTAB.$^7$ CCDC 855398 - 855402 contain the supplementary crystallographic data for these structures. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. A summary of the crystallographic data and structure refinements are given in Table 1.
The crystal structure of \(1^{ \text{dcm}}\) was obtained from the second solvent cycle, i.e. \(1^{ \text{dcm(2)}}\). The crystal structure of \(1^{ \text{ace}}\) was obtained by soaking \(1^{ \text{as}}\) directly in acetone. The crystal structures of 2 were obtained by directly synthesising this material and by transformation from \(1^{ \text{as}}\).

*Specific details of each refinement*

\(1^{ \text{as}}\): The structure has large solvent accessible voids. These contained a number of diffuse electron density peaks that could not be adequately identified and refined as solvent. The SQUEEZE routine of PLATON\(^8\) was applied to the collected data, which resulted in significant reductions in \(R_1\) and \(wR_2\) and an improvement in the GOF. \(R_1\), \(wR_2\) and GOF before SQUEEZE routine: 13.03%, 43.03% and 1.036; after SQUEEZE routine: 10.58%, 33.73% and 1.124.

\(1^{ \text{dcm}}\): In the crystal structure of \(1^{ \text{dcm}}\) the dichloromethane solvate molecule is 50% occupied and the carbon atom of this molecule was refined isotropically. The structure also has large solvent accessible voids. These contained a number of diffuse electron density peaks that could not be adequately identified and refined as solvent. The SQUEEZE routine of PLATON\(^8\) was applied to the collected data, which resulted in significant reductions in \(R_1\) and \(wR_2\) and an improvement in the GOF. \(R_1\), \(wR_2\) and GOF before SQUEEZE routine: 10.99%, 39.99% and 1.295; after SQUEEZE routine: 7.96%, 29.50% and 1.084.

\(1^{ \text{ace}}\): The acetone molecule has large thermal ellipsoids consistent with some disorder, particularly of the methyl groups. The structure also has large solvent accessible voids. These contained a number of diffuse electron density peaks that could not be adequately identified and refined as solvent. The SQUEEZE routine of PLATON\(^8\) was applied to the collected data, which resulted in a reduction in \(R_1\) and \(wR_2\) and an improvement in the GOF. \(R_1\), \(wR_2\) and GOF before SQUEEZE routine: 15.30%, 48.07% and 1.409; after SQUEEZE routine: 13.06%, 41.74% and 1.200.
Table 1. Crystal data and X-ray experimental data for 1<sup>as</sup> – 2.

<table>
<thead>
<tr>
<th>Compound</th>
<th>1&lt;sup&gt;as&lt;/sup&gt;</th>
<th>1&lt;sup&gt;dem&lt;/sup&gt;</th>
<th>1&lt;sup&gt;ace&lt;/sup&gt;</th>
<th>2 (direct synthesis)</th>
<th>2 (from 1&lt;sup&gt;as&lt;/sup&gt;)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Empirical formula</td>
<td>C&lt;sub&gt;11&lt;/sub&gt;H&lt;sub&gt;14&lt;/sub&gt;AgClN&lt;sub&gt;4&lt;/sub&gt;O&lt;sub&gt;5&lt;/sub&gt;S</td>
<td>C&lt;sub&gt;9.5&lt;/sub&gt;H&lt;sub&gt;9&lt;/sub&gt;AgCl&lt;sub&gt;2&lt;/sub&gt;N&lt;sub&gt;4&lt;/sub&gt;O&lt;sub&gt;4&lt;/sub&gt;</td>
<td>C&lt;sub&gt;12&lt;/sub&gt;H&lt;sub&gt;14&lt;/sub&gt;AgClN&lt;sub&gt;4&lt;/sub&gt;O&lt;sub&gt;5&lt;/sub&gt;</td>
<td>C&lt;sub&gt;9.10&lt;/sub&gt;AgClN&lt;sub&gt;4&lt;/sub&gt;O&lt;sub&gt;5&lt;/sub&gt;</td>
<td>C&lt;sub&gt;9.10&lt;/sub&gt;AgClN&lt;sub&gt;4&lt;/sub&gt;O&lt;sub&gt;5&lt;/sub&gt;</td>
</tr>
<tr>
<td>Formula weight</td>
<td>457.64</td>
<td>421.98</td>
<td>437.59</td>
<td>397.53</td>
<td>397.53</td>
</tr>
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<td>Crystal system</td>
<td>trigonal</td>
<td>trigonal</td>
<td>trigonal</td>
<td>monoclinic</td>
<td>monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>R&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>R&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>R&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>P&lt;sub&gt;2&lt;/sub&gt;&lt;sup&gt;1&lt;/sup&gt;/c</td>
<td>P&lt;sub&gt;2&lt;/sub&gt;&lt;sup&gt;1&lt;/sup&gt;/c</td>
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<tr>
<td>a (Å)</td>
<td>27.978(2)</td>
<td>28.3911(13)</td>
<td>28.3534(15)</td>
<td>7.1340(6)</td>
<td>7.1312(2)</td>
</tr>
<tr>
<td>b (Å)</td>
<td>27.978(2)</td>
<td>28.3911(13)</td>
<td>28.3534(15)</td>
<td>18.3483(17)</td>
<td>18.2962(4)</td>
</tr>
<tr>
<td>c (Å)</td>
<td>14.9798(10)</td>
<td>11.7766(17)</td>
<td>13.9544(19)</td>
<td>10.5892(12)</td>
<td>10.5785(3)</td>
</tr>
<tr>
<td>α (°)</td>
<td>90</td>
<td>90</td>
<td>90</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>β (°)</td>
<td>90</td>
<td>90</td>
<td>90</td>
<td>108.596(10)</td>
<td>108.561(3)</td>
</tr>
<tr>
<td>γ (°)</td>
<td>120</td>
<td>120</td>
<td>120</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>Volume (Å&lt;sup&gt;3&lt;/sup&gt;)</td>
<td>10155.0(13)</td>
<td>8220.8(13)</td>
<td>9715.2(15)</td>
<td>1313.7(2)</td>
<td>1308.43(6)</td>
</tr>
<tr>
<td>Z</td>
<td>18</td>
<td>18</td>
<td>18</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Density (calc.) (Mg/m&lt;sup&gt;3&lt;/sup&gt;)</td>
<td>1.347</td>
<td>1.534</td>
<td>1.346</td>
<td>2.010</td>
<td>2.018</td>
</tr>
<tr>
<td>Absorption coefficient (mm&lt;sup&gt;-1&lt;/sup&gt;)</td>
<td>1.125</td>
<td>1.410</td>
<td>1.079</td>
<td>1.763</td>
<td>1.770</td>
</tr>
<tr>
<td>F(000)</td>
<td>4104</td>
<td>3726</td>
<td>3924</td>
<td>784</td>
<td>784</td>
</tr>
<tr>
<td>Crystal size (mm&lt;sup&gt;3&lt;/sup&gt;)</td>
<td>0.24 x 0.12 x 0.09</td>
<td>0.16 x 0.13 x 0.08</td>
<td>0.38 x 0.24 x 0.19</td>
<td>0.29 x 0.10 x 0.07</td>
<td>0.19 x 0.12 x 0.09</td>
</tr>
<tr>
<td>θ range for data collection (°)</td>
<td>2.52 to 29.24</td>
<td>2.49 to 29.13</td>
<td>2.49 to 29.22</td>
<td>3.01 to 29.31</td>
<td>3.01 to 29.21</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>24591</td>
<td>15970</td>
<td>21843</td>
<td>14822</td>
<td>14744</td>
</tr>
<tr>
<td>Observed reflections [R(int)]</td>
<td>5459 [0.0714]</td>
<td>4358 [0.0585]</td>
<td>5230 [0.0746]</td>
<td>3234 [0.0573]</td>
<td>3219 [0.0444]</td>
</tr>
<tr>
<td>Goodness-of-fit on F&lt;sup&gt;2&lt;/sup&gt;</td>
<td>1.124</td>
<td>1.084</td>
<td>1.200</td>
<td>1.054</td>
<td>1.057</td>
</tr>
<tr>
<td>R&lt;sub&gt;1&lt;/sub&gt; [I&gt;2σ(I)]</td>
<td>0.1058</td>
<td>0.0796</td>
<td>0.1306</td>
<td>0.0400</td>
<td>0.0348</td>
</tr>
<tr>
<td>wR&lt;sub&gt;2&lt;/sub&gt; (all data)</td>
<td>0.3373</td>
<td>0.2950</td>
<td>0.4174</td>
<td>0.0888</td>
<td>0.0736</td>
</tr>
<tr>
<td>Largest diff. peak and hole (e.Å&lt;sup&gt;-3&lt;/sup&gt;)</td>
<td>1.375 and -0.770</td>
<td>1.510 and -0.867</td>
<td>3.332 and -1.114</td>
<td>1.131 and -0.773</td>
<td>0.755 and -0.703</td>
</tr>
</tbody>
</table>
Figure SI 6. Perspective views of a sodalite unit in the crystal structures of (a) $1^{\text{dcm}}$ and (b) $1^{\text{ace}}$, viewed along the $c$ axis.

Figure SI 7. The asymmetric units of: (a) $1^{\text{as}}$; (b) $1^{\text{dcm}}$; (c) $1^{\text{ace}}$; (d) 2.
**Crystal-to-crystal breathing**

**Table S1 2.** Summary of the unit cell parameters for 1.

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Volume (Å³)</th>
<th>$a$ &amp; $b$ (Å)</th>
<th>$c$ (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DMSO/EtOAc ($1_{as}$)</td>
<td>10155.0(13)</td>
<td>27.978(2)</td>
<td>14.970(1)</td>
</tr>
<tr>
<td>Dichloromethane ($1_{dcm(1)}$)</td>
<td>8296(1)</td>
<td>28.380(14)</td>
<td>11.8939(16)</td>
</tr>
<tr>
<td>Acetone ($1_{ace(1)}$)</td>
<td>9815(20)</td>
<td>28.33(4)</td>
<td>14.12(2)</td>
</tr>
<tr>
<td>Dichloromethane ($1_{dcm(2)}$)</td>
<td>8220.8(13)</td>
<td>28.3911(13)</td>
<td>11.7766(17)</td>
</tr>
<tr>
<td>Acetone ($1_{ace(2)}$)</td>
<td>9748(15)</td>
<td>28.35(4)</td>
<td>14.01(5)</td>
</tr>
<tr>
<td>Acetone (directly from $1_{as}$)</td>
<td>9715.2(15)</td>
<td>28.3534(15)</td>
<td>13.9544(19)</td>
</tr>
</tbody>
</table>

* Note: the crystallographic parameters in this table represent individual single crystals.
6. Thermogravimetric Analysis and Differential Scanning Calorimetry (TGA/DSC) traces

![Figure SI 8](image)

**Figure SI 8.** The TGA/DSC traces for 1a. The initial weight loss corresponds to loss of ethyl acetate solvate molecules located in the channels. Further weight losses correspond to ~1.125 DMSO molecules (as calculated) over 2 steps. The exothermic peak in the DSC trace at ~185 °C is attributed to structural transformation of 1a to 2.

![Figure SI 9](image)

**Figure SI 9.** The TGA/DSC traces of 1dcm. The initial weight loss corresponds to loss of the volatile dichloromethane solvate molecules located in the channels. Subsequent weight loss corresponds to ~0.5 dichloromethane molecules (as calculated) over two steps that are lost from the walls of the structure. The exothermic peak in the DSC trace at ~142 °C is attributed to structural transformation of 1dcm to 2.
Figure S1 10. The TGA/DSC traces of 1\textsuperscript{ace}. The initial weight loss corresponds to loss of acetone solvate molecules in the channels. Further weight loss corresponds to approximately one acetone molecule over 3 steps (as calculated) lost from the walls of the structure. The exothermic peak in the DSC trace at \(~194\) °C is attributed to structural transformation of 1\textsuperscript{ace} to 2.

Figure S1 11: The TGA/DSC traces of 1\textsuperscript{as} (grey) and the TGA/DSC traces (blue) of the same sample after the first heating cycle. The second experiment corresponds to the transformed sample (i.e. 2) showing no further solvent loss and no heat flow in the DSC trace.
7. Miscellaneous data

$^1$H NMR digestions

Figure SI 12. The full $^1$H NMR spectrum (300MHz/CD$_3$CN) of 1''n.

Figure SI 13. The full $^1$H NMR spectrum (300MHz/CD$_3$CN) of 1'dcm(1).
Figure SI 14. The full $^1$H NMR spectrum (300MHz/CD$_3$CN) of 1$^{ace(1)}$.

Figure SI 15. The full $^1$H NMR spectrum (300MHz/CD$_3$CN) of 1$^{dcm(2)}$. 
Figure SI 16. The full $^1$H NMR spectrum (300MHz/CD$_3$CN) of $1^{\text{ace}(2)}$.

8. References