

Porous salts based on the pamoate ion

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Electronic Supplementary Information

All chemicals were obtained from Sigma-Aldrich and used without further purification.

Compounds are labelled as follows:

| | |
|------------------|---|
| 1•THF | 3,4-lutidinium pamoate hydrate with THF in channels |
| 1•Ether | 3,4-lutidinium pamoate hydrate with diethyl ether in channels |
| 1•Acetone | 3,4-lutidinium pamoate hydrate with acetone in channels |
| 1•DCM | 3,4-lutidinium pamoate hydrate with dichloromethane in channels |
| 2 | 2,5-lutidinium pamoate NMP solvate |
| 2b | 2,5-lutidinium pamoate NMP solvate after immersion in supercritical CO ₂ |
| 3 | 3,4-lutidinium pamoate NMP solvate |
| 3b | 3,4-lutidinium pamoate NMP solvate after immersion in supercritical CO ₂ |
| 4 | 3-picolinium pamoate NMP solvate |
| 4b | 3-picolinium pamoate NMP solvate after immersion in supercritical CO ₂ |

Single-crystal X-ray diffraction data were collected on a Bruker-Nonius SMART Apex diffractometer equipped with a Mo fine-focus sealed tube and a 0.5 mm MonoCap collimator, as well as an Oxford Cryosystems Cryostat (700 Series Cryostream Plus). Crystals were mounted on MiTeGen mounts in paratone oil, and data were collected at 100 K. Data reduction, absorption corrections and unit cell determination were carried out using the diffractometer software (APEXII, Bruker).¹

Structures were solved and refined using the SHELX-97² package implemented through XSeed³. Carboxylic acid and pyridinium hydrogen atoms were located in the difference map in most cases. The disordered electron density in the channels was analysed with Platon/SQUEEZE⁴ implemented in WinGX.⁵ Diagrams were generated using POV-Ray⁶ implemented in X-Seed.³ Difference maps were generated in Olex^{2,7}.

Powder X-ray diffraction patterns were collected on a PANalytical X'Pert PRO diffractometer with an X'Celerator detector and Cu radiation source with a Ni filter. Samples were spun during the data collection. Variable temperature PXRD on **1•THF** was collected in a capillary with an Oxford Cryosystems Cryostat as temperature controller. Variable temperature PXRD on **2-4** was collected using an Anton Paar TTK450 hot stage. In all powder diffraction patterns, the y -axis gives intensity counts in arbitrary units.

TGA data were collected on a TA Instruments TGA Q500 with a heating rate of 10 °C per minute.

Immersion of samples in supercritical CO₂ was carried out using a SAMDRI®-PVT-3D critical point dryer. Crystals were placed in glass vials inside the dryer.

Experimental data for 1·THF, 1·Ether, 1·Acetone and 1·DCM:

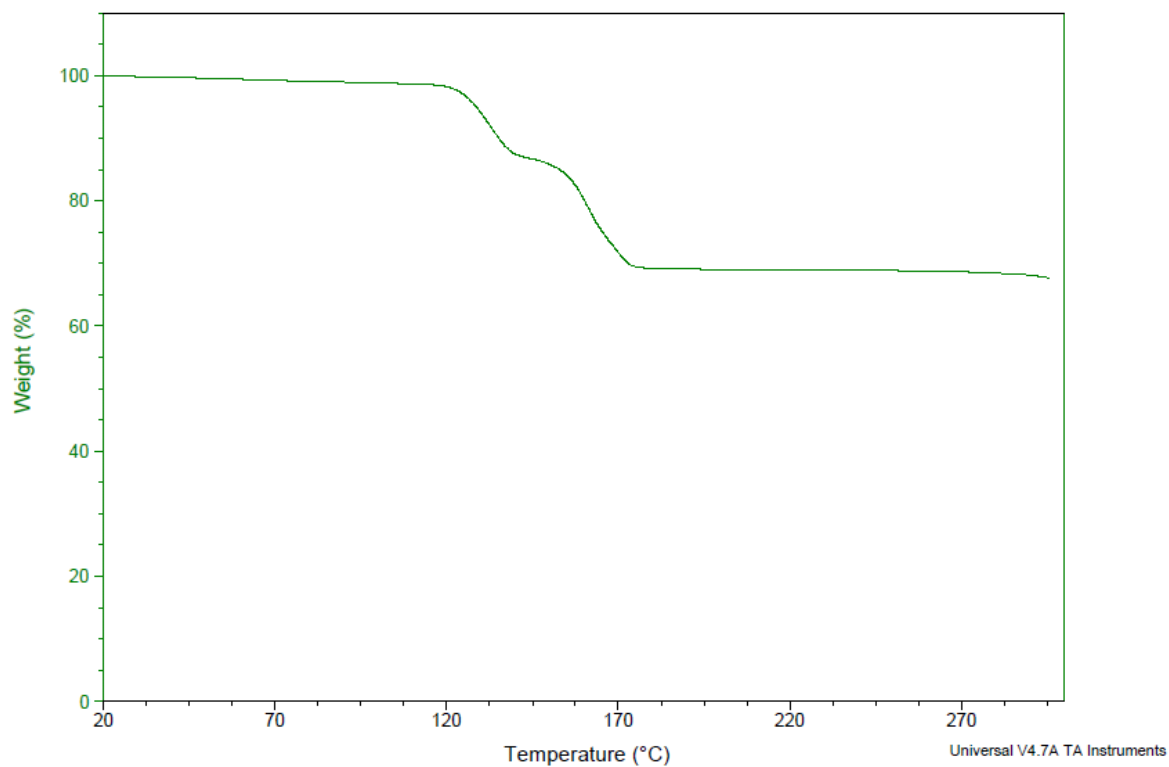


Fig. S1 TGA trace of 1·THF

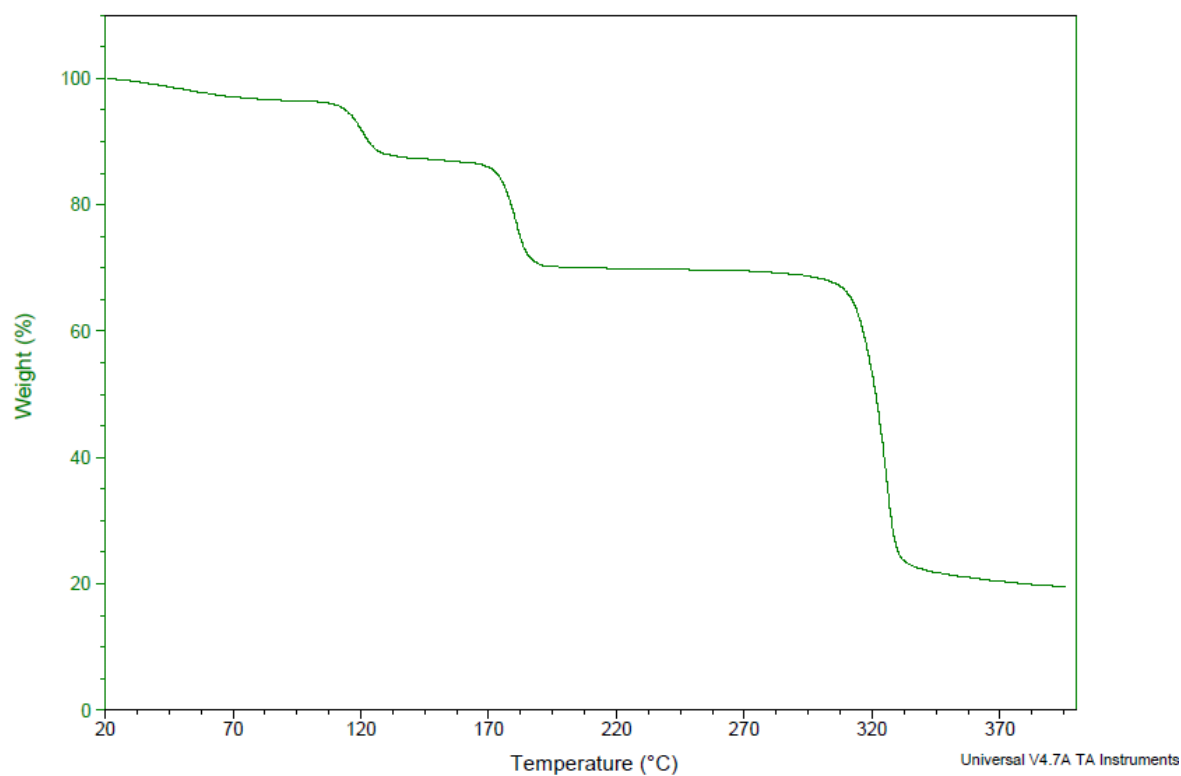


Fig. S2 TGA trace of 1·Ether

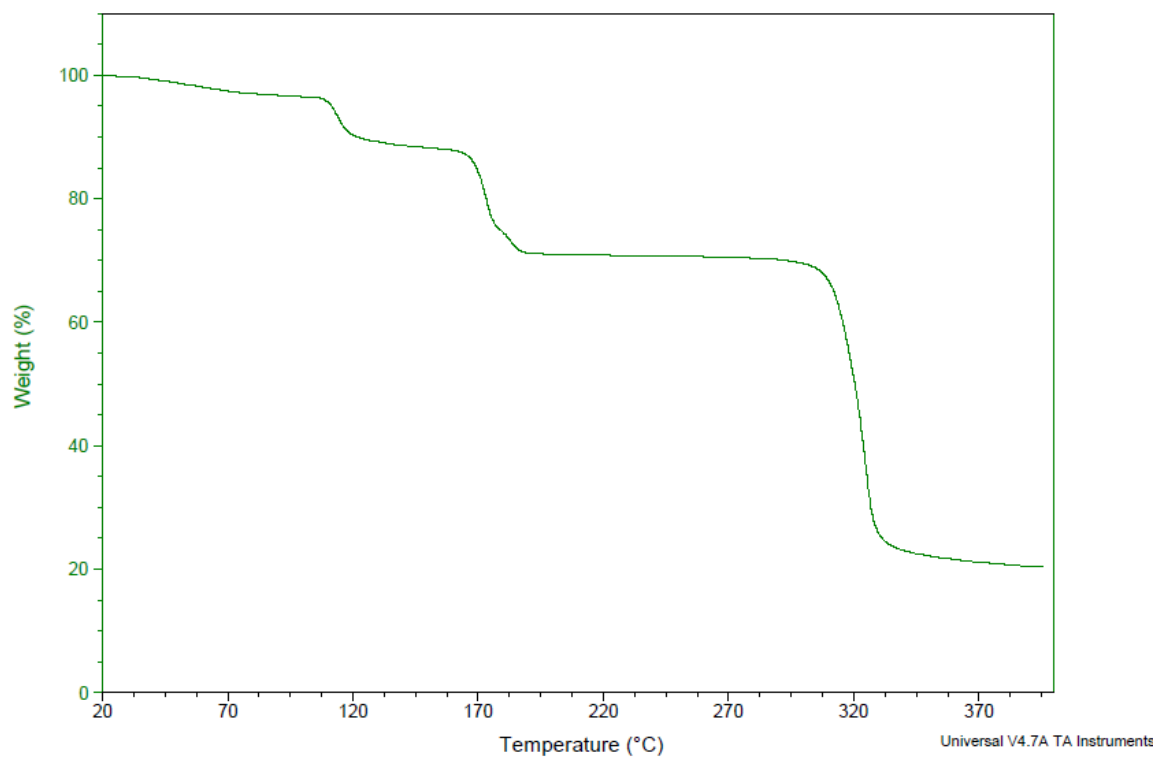


Fig. S3 TGA trace of 1·Acetone

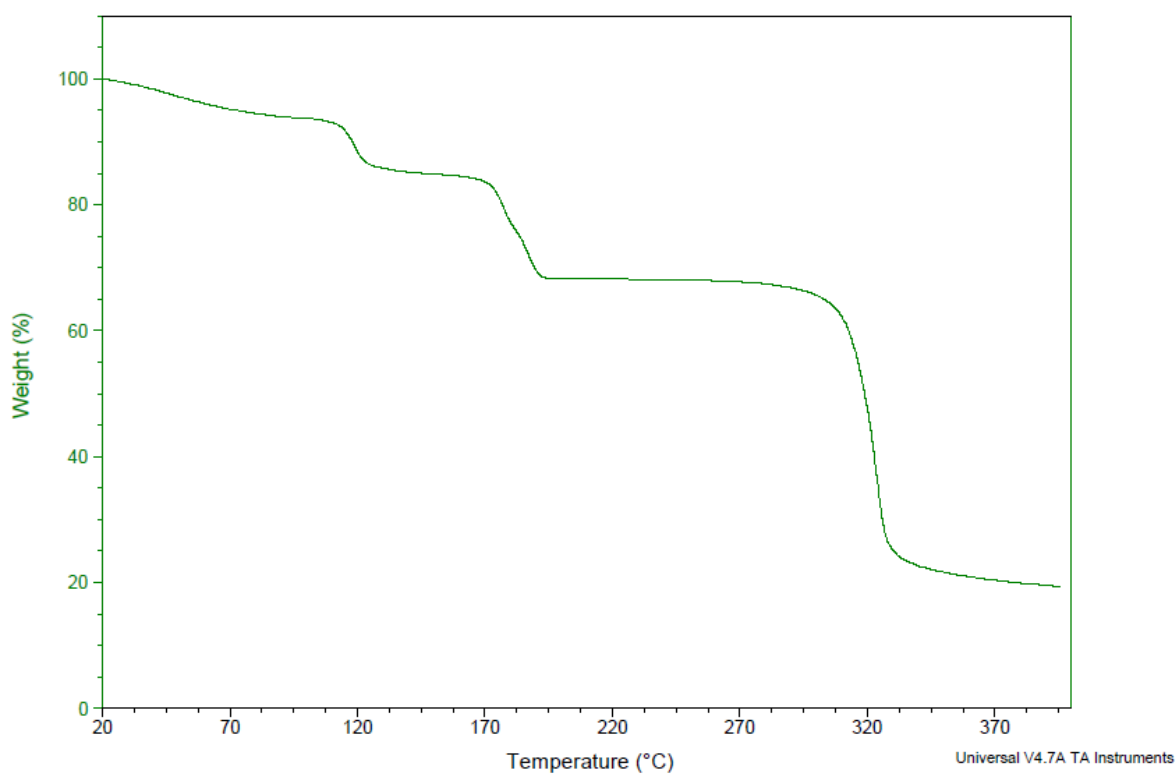


Fig. S4 TGA trace of 1·DCM

Table S1 Summary of TGA results for **1·THF**, **1·Ether**, **1·Acetone** and **1·DCM**

| | Experimental % mass loss | Calculated % mass loss | pamoate:lutidinium:solvent:water |
|------------------|--------------------------|------------------------|----------------------------------|
| 1·THF | 31.0 | 31.1 | 1:1:1:0.5 |
| 1·Ether | 30.0 | 31.3 | 1:1:1:0.5 |
| 1·Acetone | 29.0 | 29.4 | 1:1:1:0.5 |
| 1·DCM | 31.7 | 32.6 | 1:1:1:0.5 |

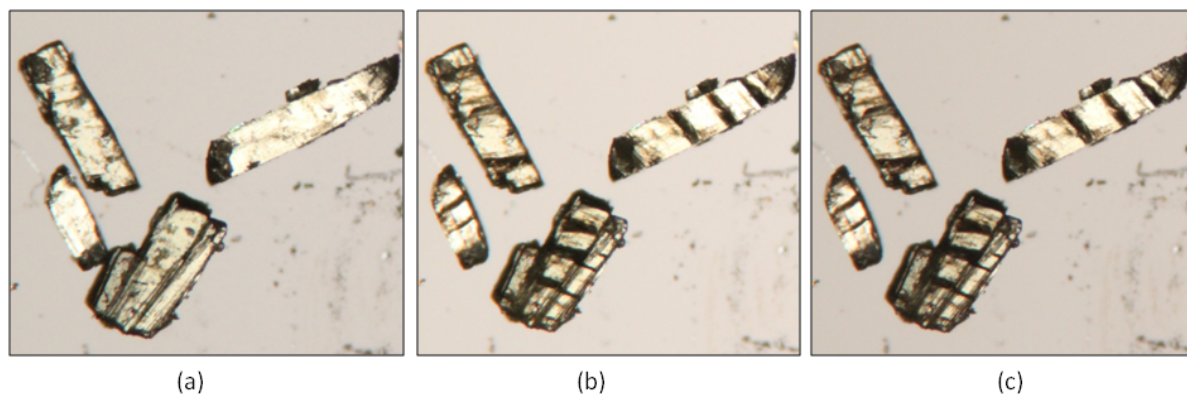


Fig. S5 (a) crystals of **1·THF** freshly removed from mother liquor; (b) crystals of **1·THF** after being exposed to DCM vapour for 12 hours and (c) crystals of **1·THF** after being exposed to DCM vapour for 24 hours.

Notes on the structure refinement for **1·THF**, **1·Ether**, **1·Acetone** and **1·DCM**

1·THF has been modelled with one THF molecule per asymmetric unit. The total of one molecule is made up of two molecules on special positions. Both molecules are disordered. The NMR (see page 17 of ESI) indicates that there is, in fact, a total of one THF per asymmetric unit.

We were unable to model the solvent in **1·Ether** satisfactorily. We have therefore applied SQUEEZE⁴ to this structure. NMR data (pg. 17) indicate that the channel is occupied by diethyl ether.

In the case of **1·Acetone**, we were able to satisfactorily model one molecule of acetone (again on a special position). No good model could be obtained for the second acetone molecule, and SQUEEZE⁴ was used. The NMR data confirm that this material contains one molecule of acetone per asymmetric unit.

1·DCM has been modelled with slightly less than one molecule of DCM per asymmetric unit. Again there are two molecules of DCM, both on special positions. The second molecule of DCM has been modelled as partially occupied and disordered with a partially occupied water molecule. This gave the best model, and also corresponds to the NMR data (pg. 17), which suggest that there are only 0.75 molecules of DCM per asymmetric unit.

Crystal data for **1·THFb**: $C_{32}H_{30}NO_7$, $M = 540.57$, yellow plate, $0.22 \times 0.11 \times 0.10 \text{ mm}^3$, monoclinic, space group $C2/c$ (No. 15), $a = 22.110(6)$, $b = 19.808(5)$, $c = 14.613(4) \text{ \AA}$, $\beta = 114.992(4)^\circ$, $V = 5801(3) \text{ \AA}^3$, $Z = 8$, $D_c = 1.238 \text{ g/cm}^3$, $F_{000} = 2280$, Bruker SMART APEX CCD area-detector, MoK α radiation, $\lambda = 0.71073 \text{ \AA}$, $T = 100(2) \text{ K}$, $2\theta_{\text{max}} = 50.0^\circ$, 15212 reflections collected, 5120 unique ($R_{\text{int}} = 0.0526$). Final $Goof = 1.013$, $RI = 0.0485$, $wR2 = 0.1096$, R indices based on 3386 reflections with $I > 2\sigma(I)$ (refinement on F^2), 451 parameters, 81 restraints. Lp and absorption corrections applied, $\mu = 0.087 \text{ mm}^{-1}$.

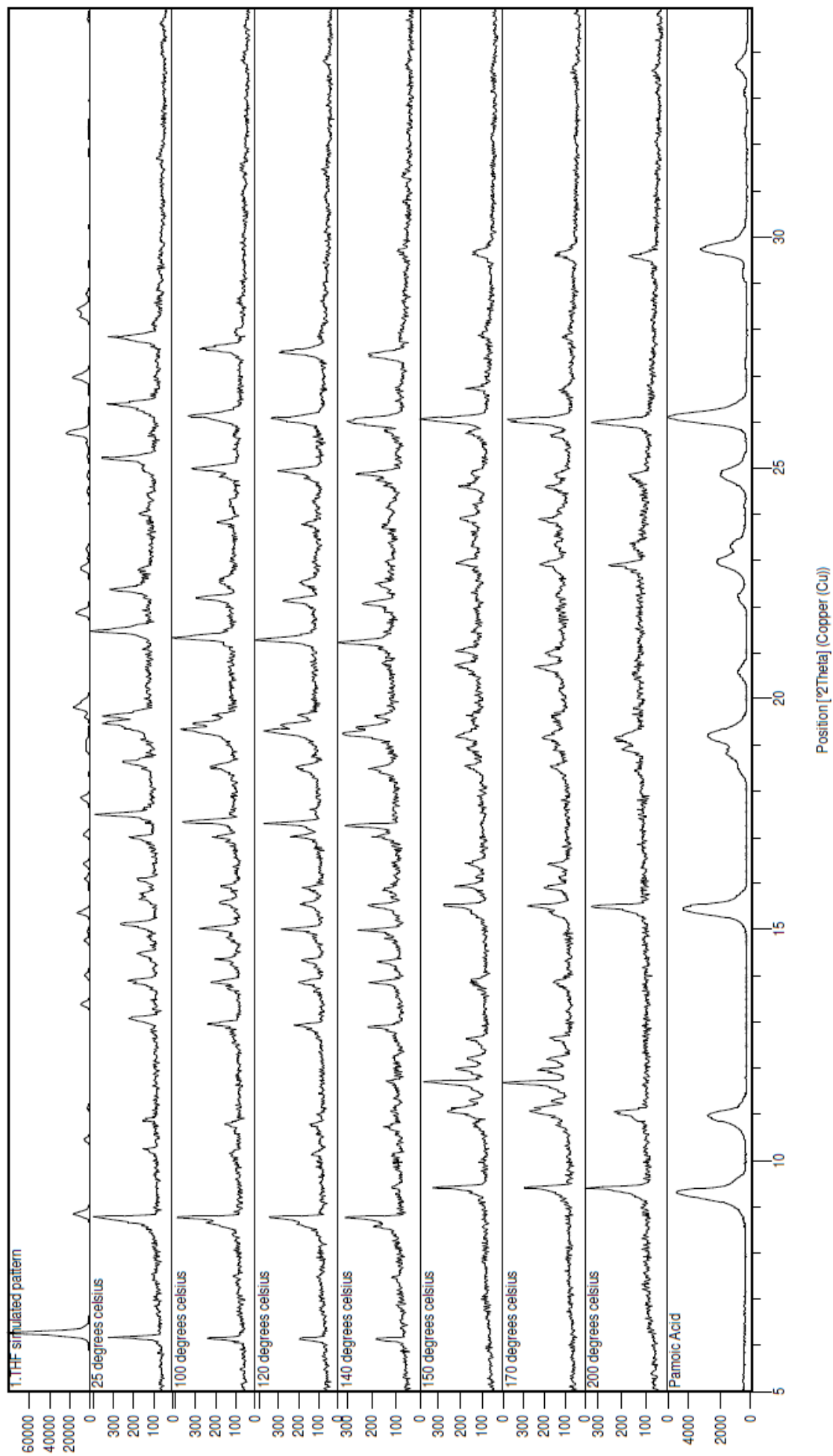
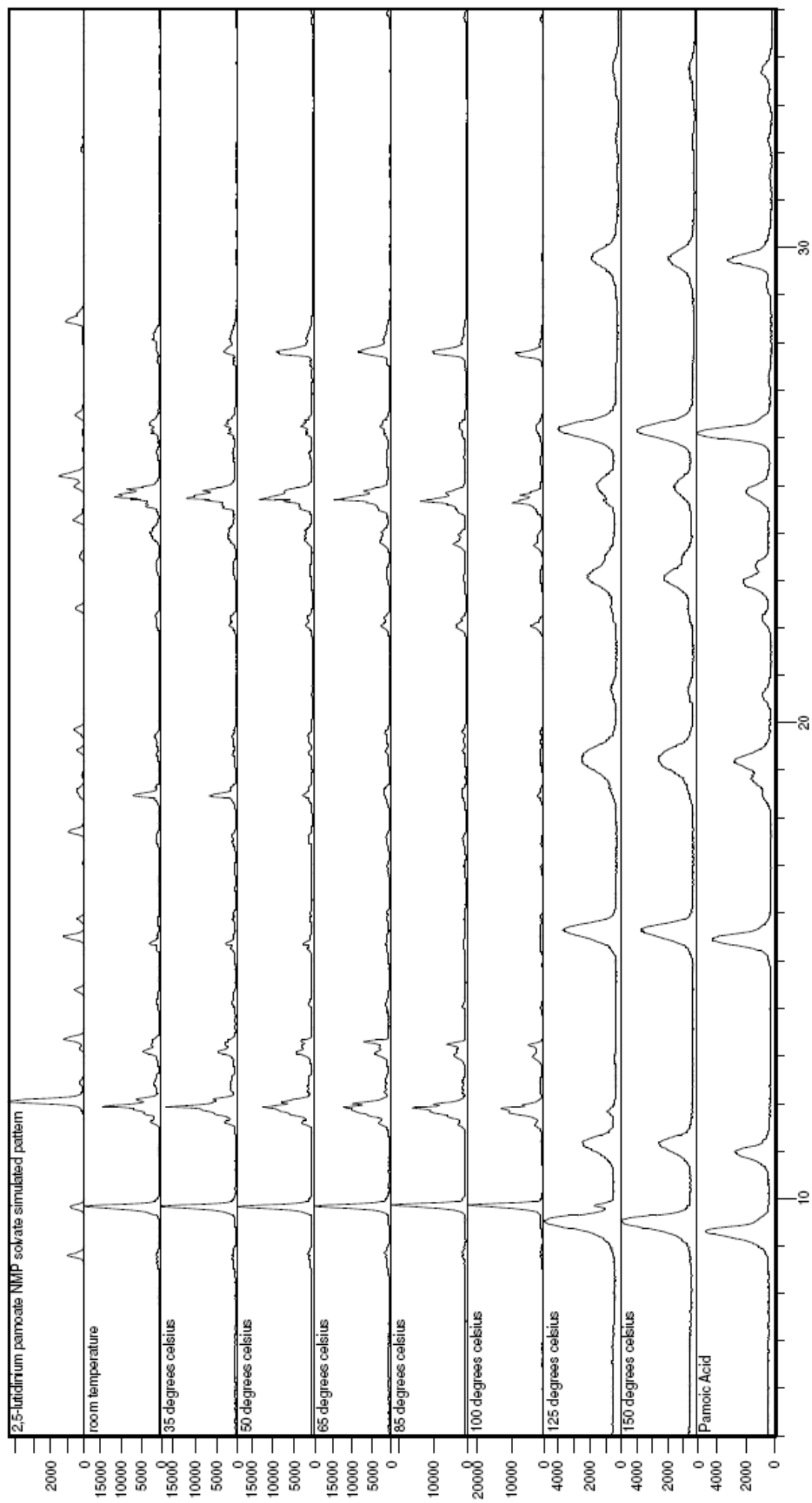


Fig. S6 Variable temperature powder diffraction for **1-THF**. The additional peaks (aside from those due to pamoic acid) visible in the patterns at 150 and 170 °C are from an unidentified phase.

Experimental data for 2



Position [2θ] (Copper (Cu))

Fig. S7 Variable temperature powder diffraction for 2

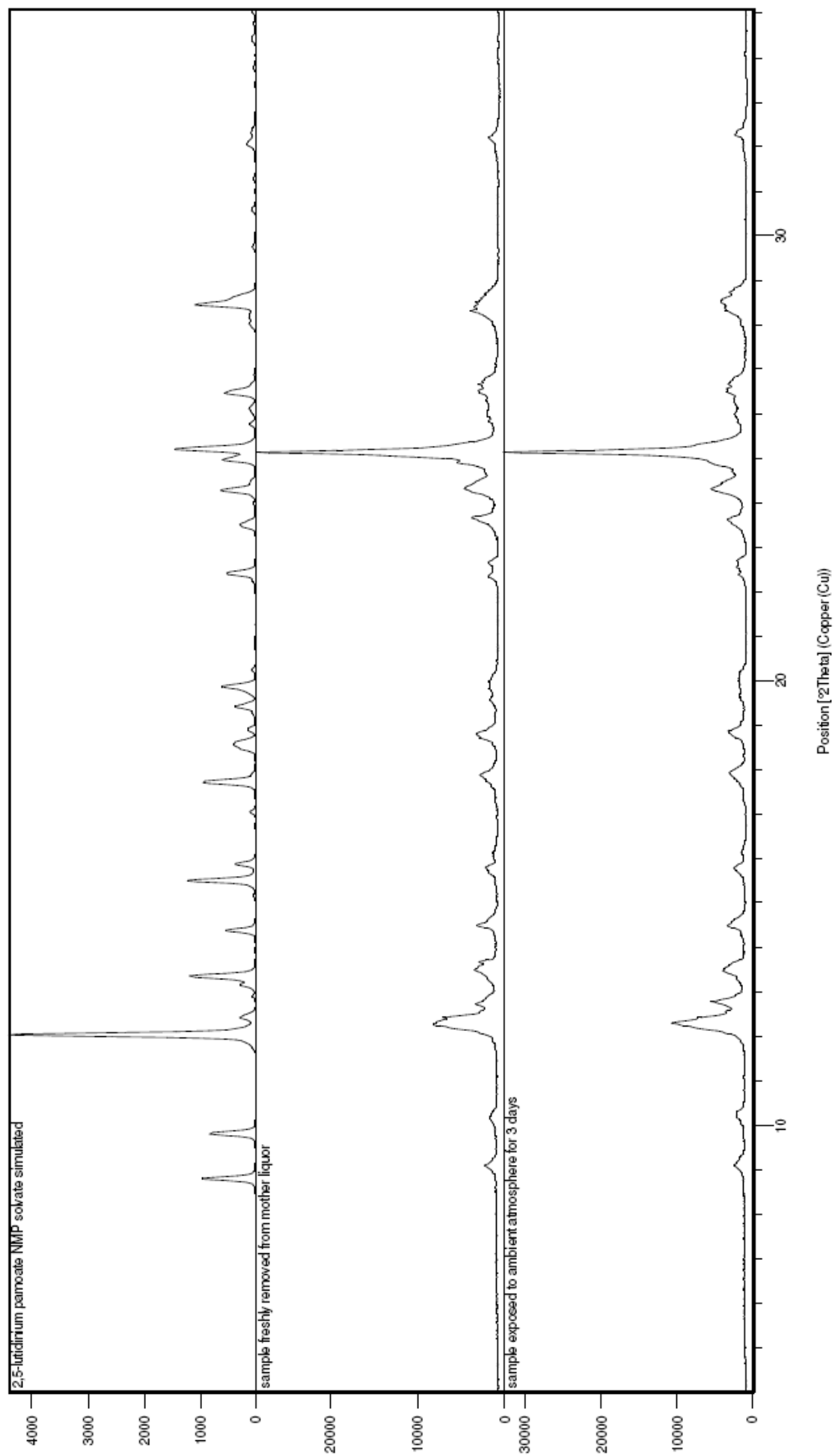


Fig. S8 Powder diffraction of fresh **2** as well as **2** exposed to ambient conditions for three days

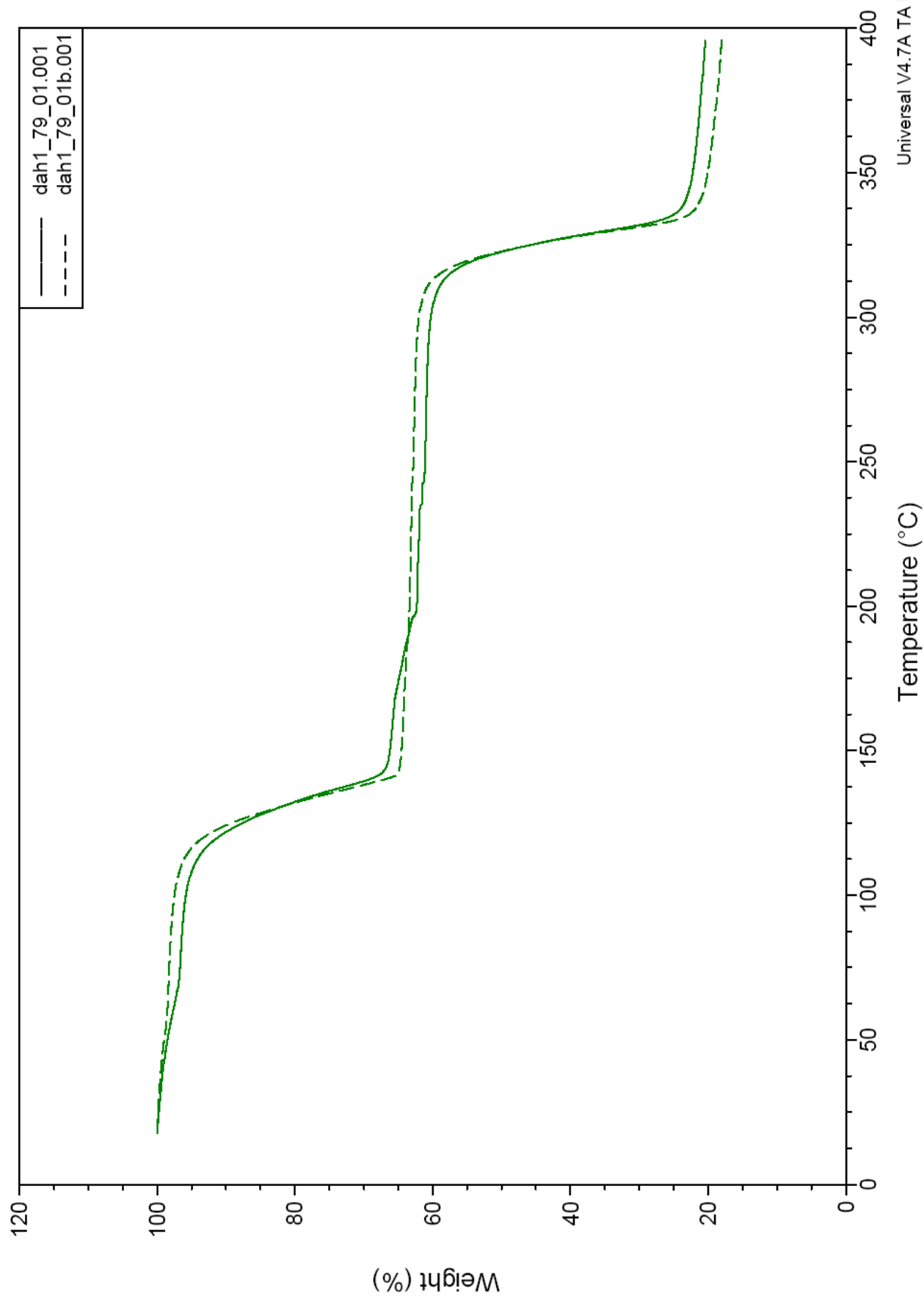


Fig. S9 TGA traces of 2 (dah1_79_01: sample fresh from mother liquor; dah1_79_01b: sample exposed to ambient conditions for 3 days)

Experimental data for 3

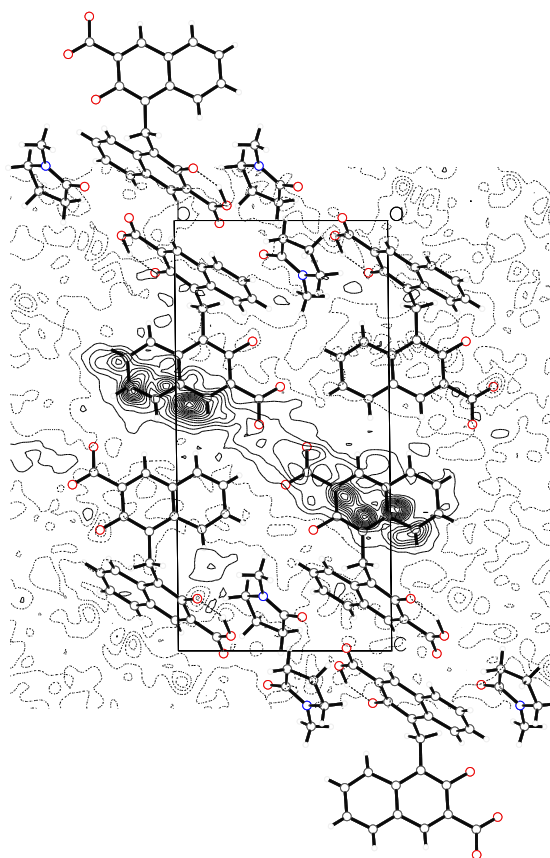
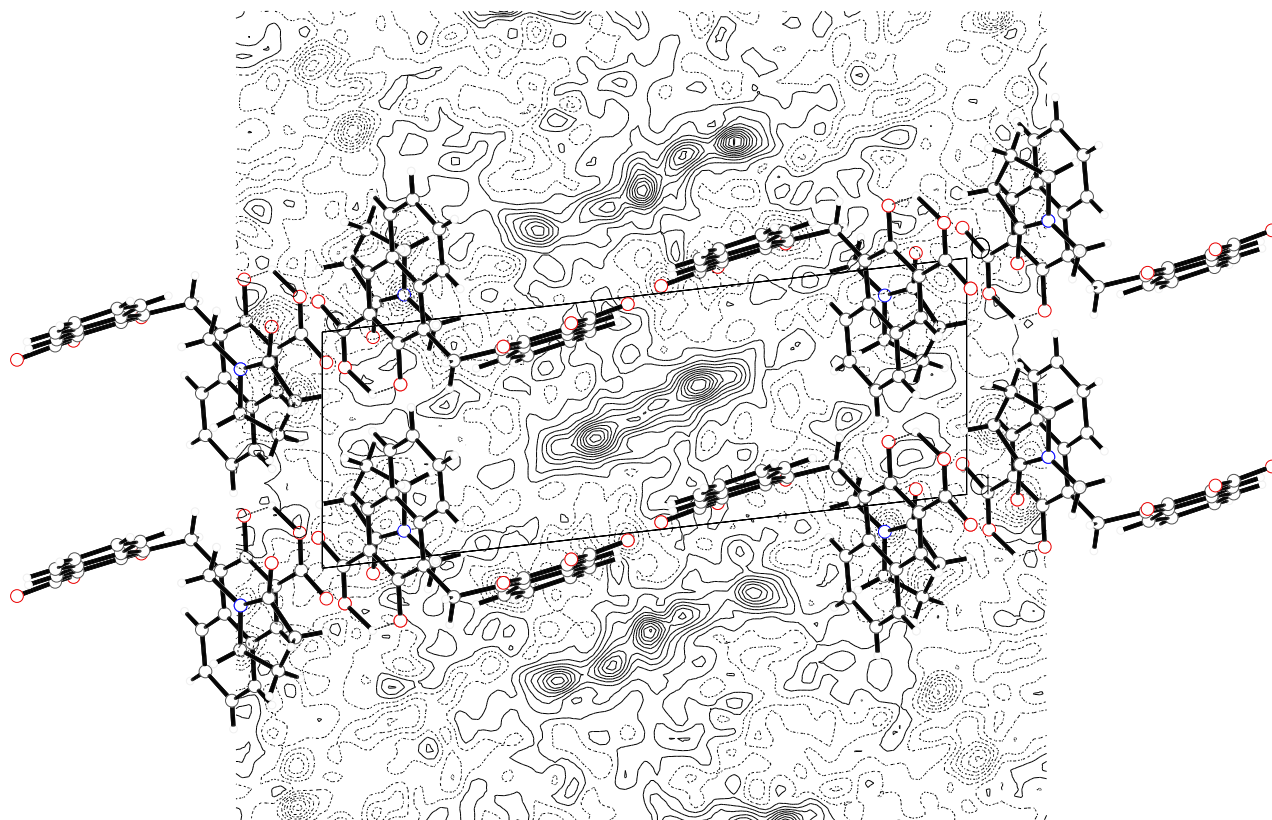
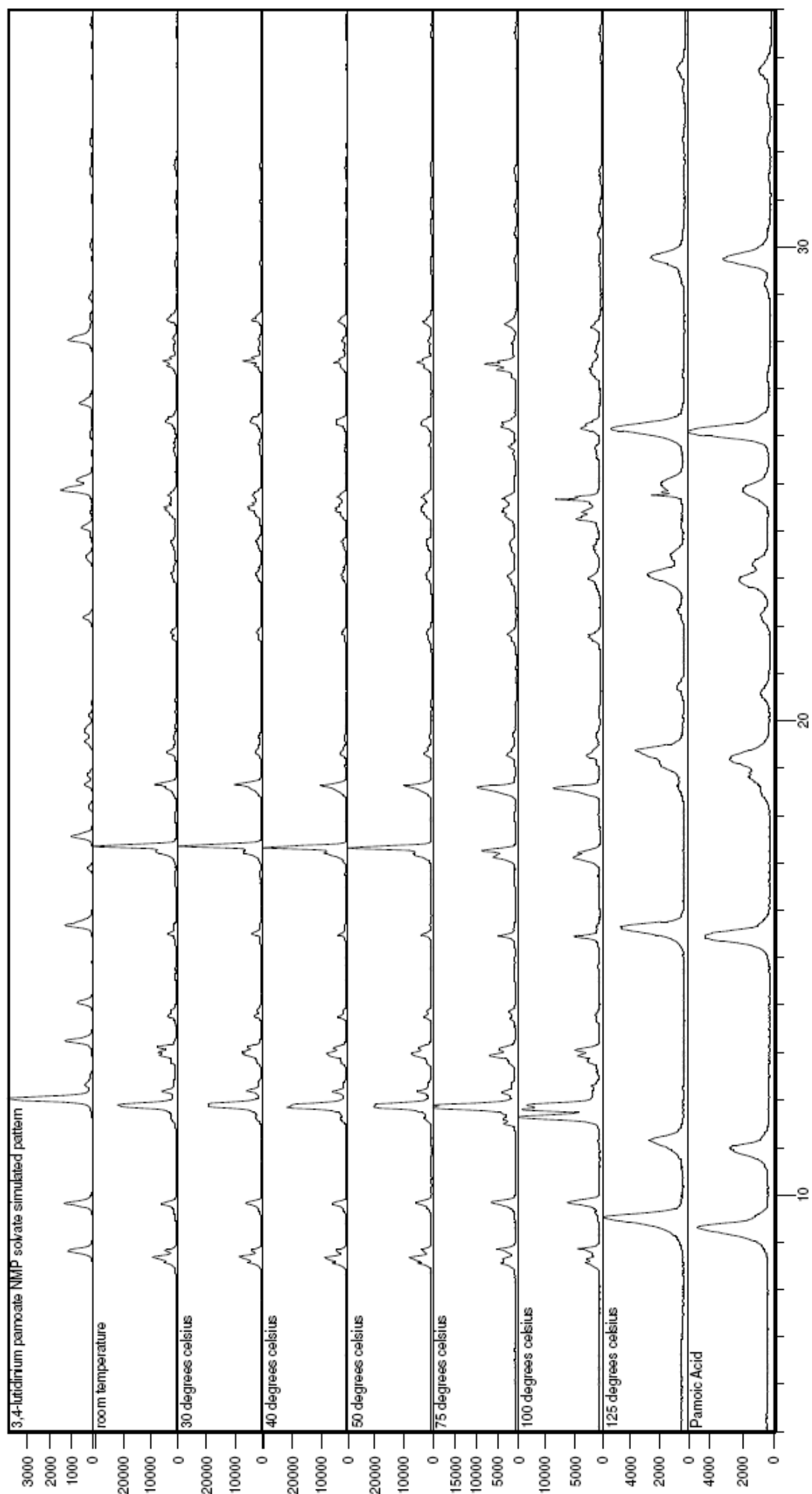
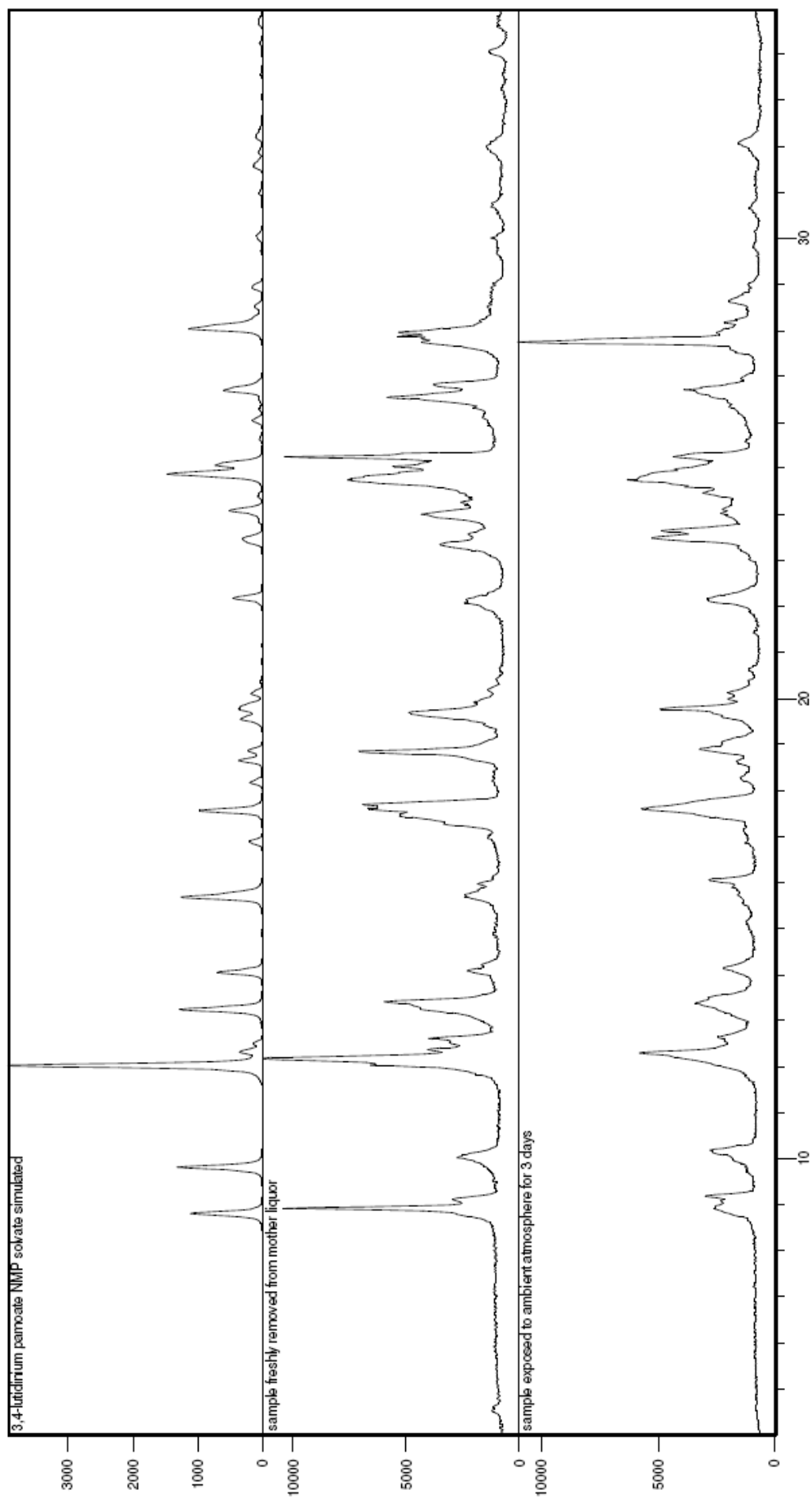


Fig. S10 Electron density maps of the channels in **2** prior to application of SQUEEZE. View is down the channel above and perpendicular to the channel walls below.



Position [2θ] (Copper (Cu))
Fig. S11 Variable temperature powder diffraction for **3**



Position [2θ] (Copper (Cu))
Fig. S12 Powder diffraction of fresh **3** as well as **3** exposed to ambient conditions for three days

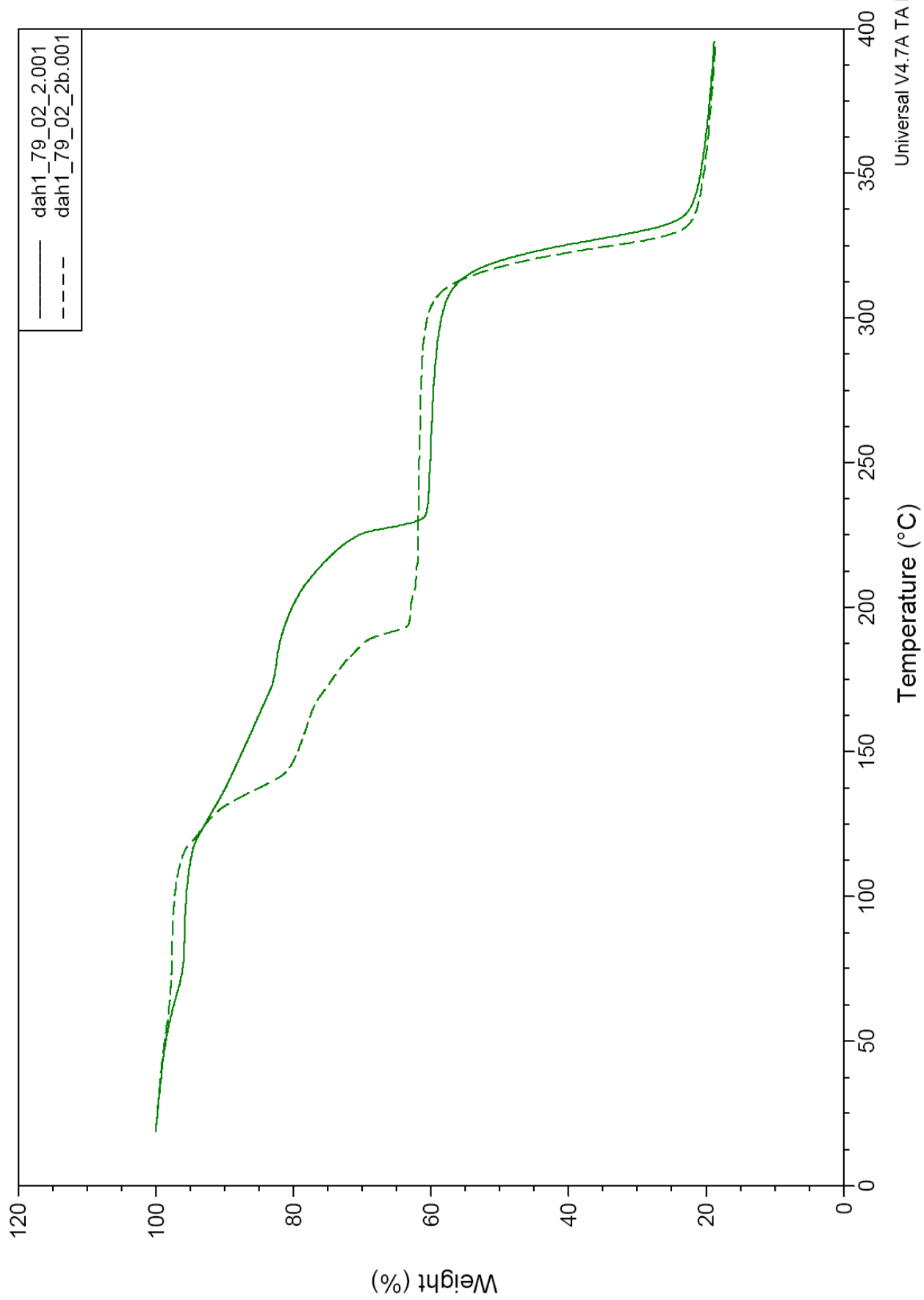


Fig. S13 TGA traces of **3** (dah1_79_02_2: sample fresh from mother liquor; dah1_79_02_2b: sample exposed to ambient conditions for 3 days)

Experimental data for **4**

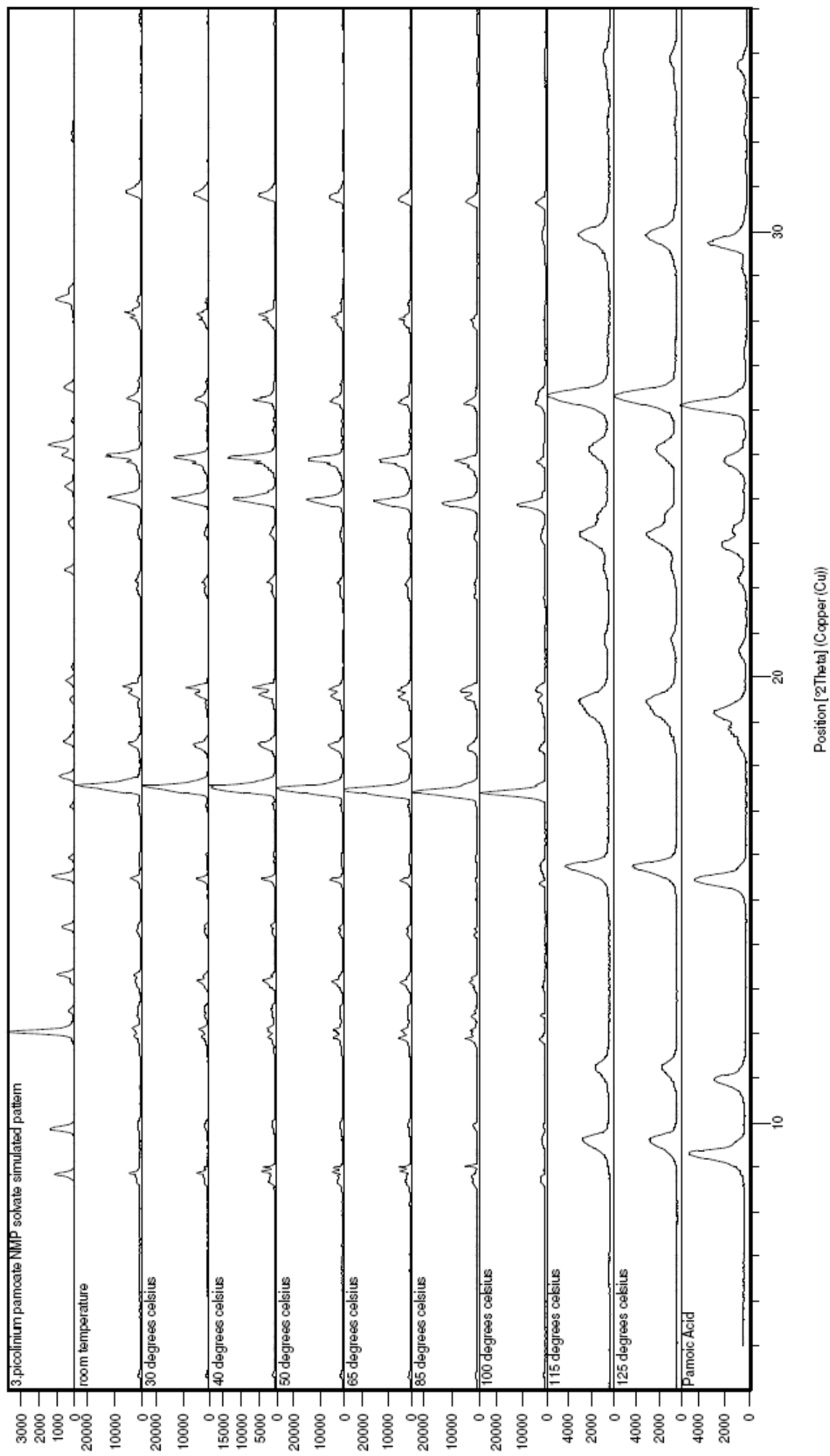


Fig. S14 Variable temperature powder diffraction for **4**

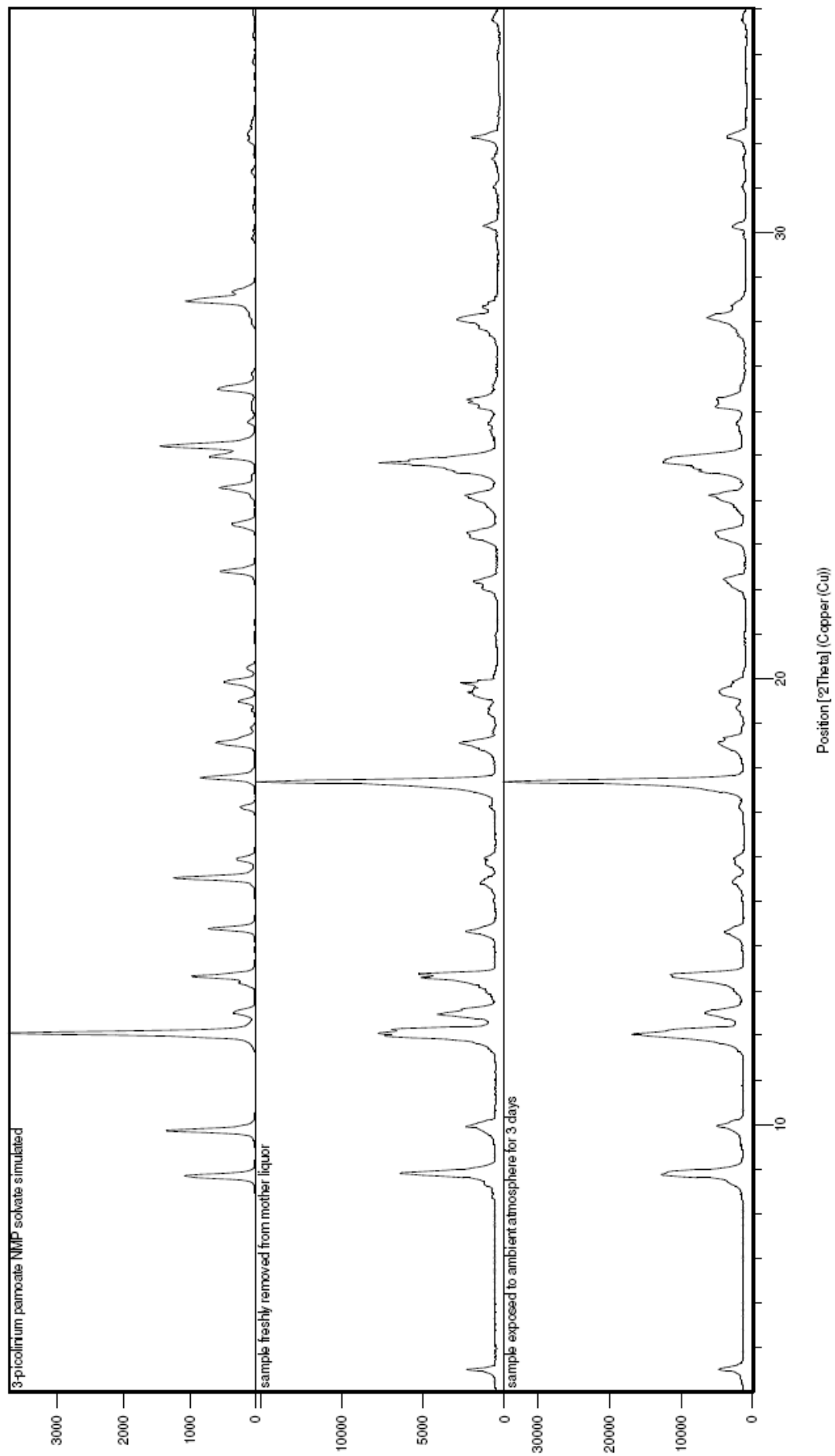


Fig. S15 Powder diffraction of fresh **4** as well as **4** exposed to ambient conditions for three days

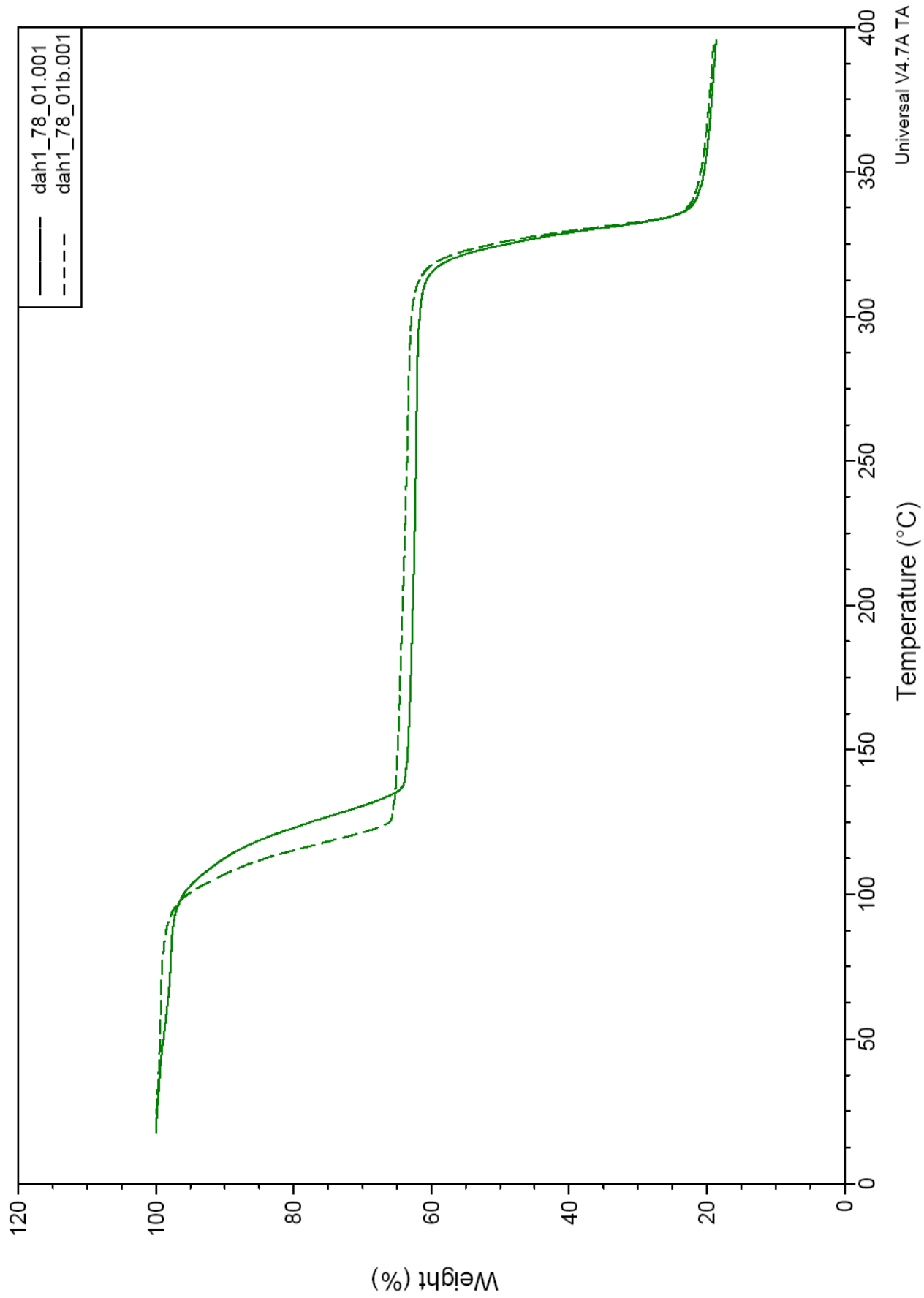


Fig. S16 TGA traces of **3** (dah1_78_01: sample fresh from mother liquor; dah1_78_01b: sample exposed to ambient conditions for 3 days)

Mechanochemical synthesis of 2, 3 and 4

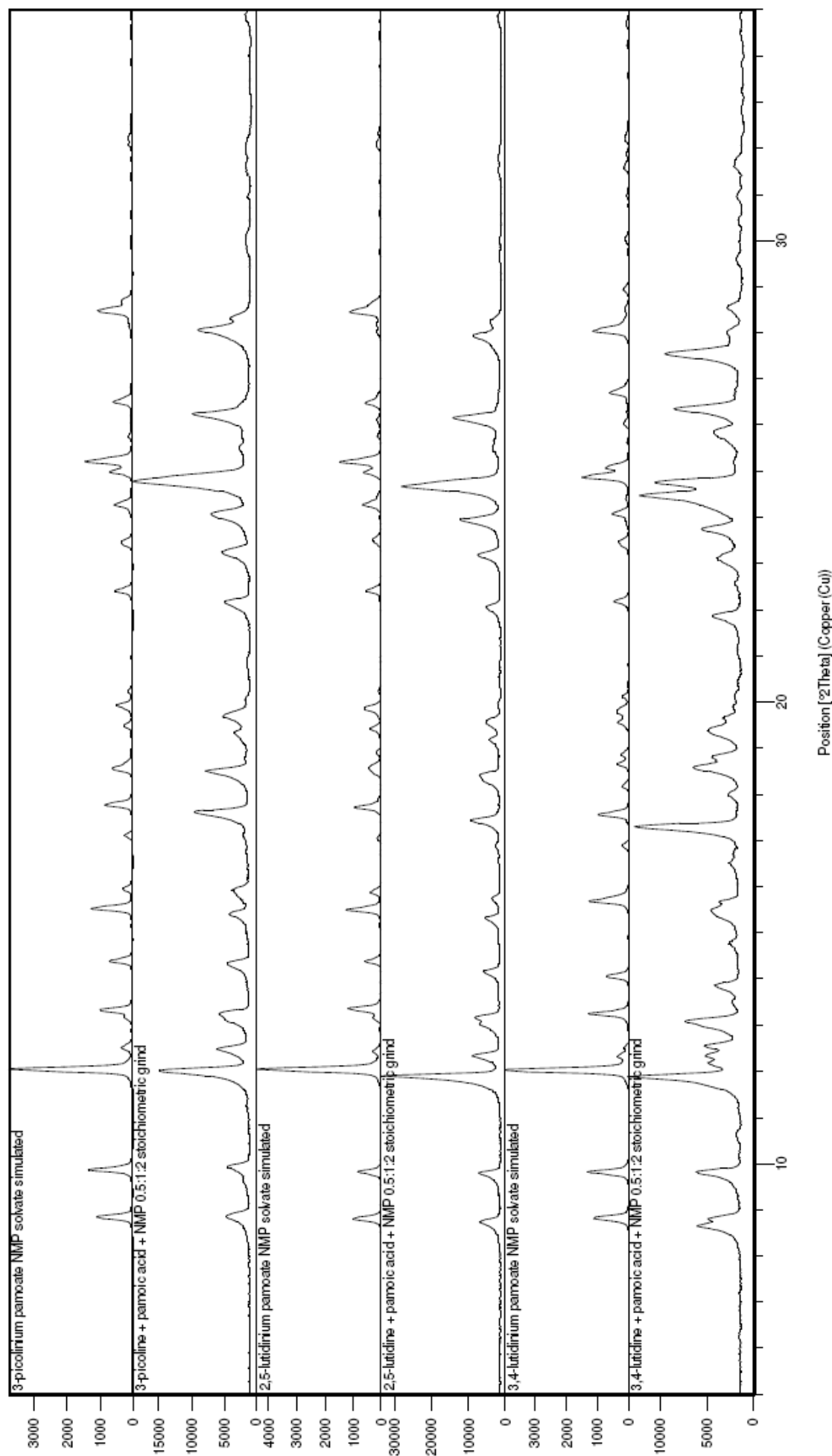


Fig. S17 Powder diffraction patterns showing products of mechanochemical synthesis of 2-4

Summary of NMR and SQUEEZE data showing partial emptying of channels in 2, 3 and 4

| | 2 | 3 | 4 |
|---|--------------------|--------------------|--------------------|
| channel contents per unit cell | 1 lutidinium 2 NMP | 1 lutidinium 2 NMP | 1 picolinium 2 NMP |
| NMP molecules in channel per cell based on NMR | 2.2 | 2 | 1.87 |
| expected electron count in channel per unit cell | 167 | 167 | 159 |
| SQUEEZE electron count per unit cell | 156 | 152 | 145 |
| void volume (full) (Å ³) | 458 | 484 | 466.5 |
| NMP molecules in channel per unit cell inferred from NMR after immersion in supercritical CO ₂ | 1.64 | 1.56 | 1.38 |
| SQUEEZE electron count after immersion in supercritical CO ₂ * | 96 | 99 | 90 |
| void volume after immersion in supercritical CO ₂ (Å ³) | 468.5 | 488.2 | 468 |

* 1 cation and 1 NMP corresponds to 113/105 electrons for 2,3/4

Summary of TGA analysis for 2-4

| | Experimental % mass loss at 250 °C (before the decomposition of pamoic acid) | Calculated % mass loss for 0.5 cation and 2 NMP molecules |
|--------------------------------------|--|---|
| 2 | 38.90 | 39.42 |
| 2 after 3 days exposed to air | 37.18 | 39.42 |
| 3 | 39.89 | 39.42 |
| 3 after 3 days exposed to air | 38.31 | 39.42 |
| 4 | 37.77 | 38.67 |
| 4 after 3 days exposed to air | 36.44 | 38.67 |

NMR spectral data

¹H spectra were recorded on a Varian 300 MHz VNMRs NMR Instrument and are referenced to DMSO-*d*₆. Data for ¹H spectra are reported as follows: chemical shift (δ ppm) (integration, multiplicity, coupling constant (Hz)). Number of solvent molecules calculated by integration are given in brackets after the compound name.

1·THF (1 THF)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.71 - 1.79 (3 H, m) 2.26 (3 H, s) 2.31 (3 H, s) 3.54 - 3.65 (3 H, m) 4.79 (2 H, s) 7.19 (2 H, t, *J*=7.48 Hz) 7.29 - 7.43 (3 H, m) 7.86 (2 H, d, *J*=7.48 Hz) 8.14 (2 H, d, *J*=8.51 Hz) 8.39 (1 H, d, *J*=5.28 Hz) 8.43 (1 H, s) 8.45 (2 H, s)

1·DCM (0.75 DCM)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 2.25 (3 H, s) 2.30 (3 H, s) 4.79 (2 H, s) 5.75 (2 H, s) 7.20 (2 H, t, *J*=7.41 Hz) 7.29 - 7.40 (3 H, m) 7.86 (2 H, d, *J*=7.78 Hz) 8.13 (2 H, d, *J*=8.07 Hz) 8.36 (1 H, d, *J*=5.28 Hz) 8.40 (1 H, s) 8.44 (2 H, s)

1·Ether (0.57 diethyl ether)

¹H NMR (400 MHz, DMSO-*d*₆) δ ppm 1.09 (3 H, t, *J*=6.93 Hz) 2.25 (3 H, s) 2.31 (3 H, s) 3.38 (2 H, q, *J*=7.03 Hz) 4.79 (2 H, s) 7.20 (2 H, t, *J*=7.52 Hz) 7.30 - 7.39 (3 H, m) 7.86 (2 H, d, *J*=8.01 Hz) 8.14 (2 H, d, *J*=8.79 Hz) 8.38 (1 H, d, *J*=5.08 Hz) 8.41 (1 H, s) 8.44 (2 H, s)

1·Actone (1 acetone)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 2.08 (4 H, s) 2.25 (3 H, s) 2.31 (3 H, s) 4.79 (2 H, s) 7.20 (2 H, t, *J*=7.48 Hz) 7.30 - 7.40 (3 H, m) 7.86 (2 H, d, *J*=8.22 Hz) 8.13 (2 H, d, *J*=8.66 Hz) 8.37 (1 H, d, *J*=5.14 Hz) 8.41 (1 H, s) 8.44 (2 H, s)

1 from supercritical CO₂ (trace amounts of THF)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 2.25 (3 H, s) 2.30 (3 H, s) 4.79 (2 H, s) 7.20 (2 H, t, *J*=7.92 Hz) 7.30 - 7.40 (3 H, m) 7.86 (2 H, d, *J*=7.78 Hz) 8.13 (2 H, d, *J*=8.07 Hz) 8.35 (1 H, d, *J*=5.28 Hz) 8.39 (1 H, s) 8.44 (2 H, s)

2 (2.2 NMP)

¹H NMR (400 MHz, DMSO-*d*₆) δ ppm 1.88 (4.4 H, quin, *J*=7.57 Hz) 2.17 (4.4 H, t, *J*=8.01 Hz) 2.31 (1.5 H, s) 2.52 (1.5 H, s) 2.69 (6.5 H, s) 3.29 (4.4 H, t) 4.79 (2 H, s) 7.21 (2 H, t, *J*=7.03 Hz) 7.36 (2 H, t, *J*=7.71 Hz) 7.41 (0.5 H, d, *J*=8.01 Hz) 7.81 (0.5 H, d, *J*=7.23 Hz) 7.87 (2 H, d, *J*=7.62 Hz) 8.13 (2 H, d, *J*=8.59 Hz) 8.43 (0.5 H, s) 8.46 (2 H, s)

3 (2 NMP)

¹H NMR (400 MHz, DMSO-*d*₆) δ ppm 1.84 - 1.93 (3.8 H, m) 2.17 (3.9 H, t, *J*=8.10 Hz) 2.28 (1.5 H, s) 2.35 (1.5 H, s) 2.68 (5.4 H, s) 3.25 - 3.32 (5 H, m) 4.79 (2 H, s) 7.17 - 7.24 (2 H, m) 7.36 (2 H, t, *J*=8.40 Hz) 7.47 (0.5 H, d, *J*=5.47 Hz) 7.86 (2 H, d, *J*=7.81 Hz) 8.13 (2 H, d, *J*=8.79 Hz) 8.44 (0.5 H, d, *J*=5.47 Hz) 8.46 (2 H, s) 8.48 (0.5 H, s)

4 (1.87 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.82 - 1.95 (3.8 H, m) 2.17 (3.8 H, t, *J*=8.07 Hz) 2.33 (1 H, s) 2.69 (5.4 H, s) 3.25 - 3.33 (3.8 H, m) 4.80 (2 H, s) 7.22 (2 H, t, *J*=6.97 Hz) 7.37 (2 H, t, *J*=7.78 Hz) 7.43 (1 H, d, *J*=4.99 Hz) 7.76 (1 H, d, *J*=7.19 Hz) 7.88 (2 H, d, *J*=8.22 Hz) 8.13 (2 H, d, *J*=8.66 Hz) 8.44 (1 H, br. s.) 8.48 (2 H, s) 8.50 (1 H, br. s.)

2 exposure to air for 3 days (2.2 NMP)

¹H NMR (400 MHz, DMSO-*d*₆) δ ppm 1.83 - 1.93 (4.1 H, m) 2.17 (4.2 H, t, *J*=8.10 Hz) 2.31 (1.5 H, s) 2.52 (1.5 H, s) 2.68 (5.9 H, s) 3.26 - 3.32 (5.7 H, m) 4.78 (2 H, s) 7.20 (2 H, t, *J*=7.91 Hz) 7.35 (2 H, t, *J*=7.71 Hz) 7.43 (0.5 H, d, *J*=8.01 Hz) 7.85 (2.5 H, d, *J*=8.40 Hz) 8.12 (2 H, d, *J*=8.59 Hz) 8.43 (0.5 H, br. s.) 8.44 (2 H, s)

3 (1.82 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.83 - 1.96 (3.7 H, m) 2.17 (3.7 H, t, *J*=8.07 Hz) 2.27 (1.5 H, s) 2.34 (1.5 H, s) 2.69 (5.3 H, s) 3.25 - 3.33 (3.7 H, m) 4.80 (2 H, s) 7.21 (2 H, t, *J*=7.48 Hz) 7.36 (2 H, t, *J*=7.04 Hz) 7.43 (0.5 H, d, *J*=5.14 Hz) 7.87 (2 H, d, *J*=8.22 Hz) 8.13 (2 H, d, *J*=8.66 Hz) 8.42 (0.5 H, d) 8.46 (2.5 H, s)

4 (1.79 NMP)

¹H NMR (400 MHz, DMSO-*d*₆) δ ppm 1.84 - 1.94 (3.6 H, m) 2.17 (3.6 H, t, *J*=8.01 Hz) 2.33 (1.5 H, s) 2.69 (5.2 H, s) 3.26 - 3.32 (3.7 H, m) 4.80 (2 H, s) 7.22 (2 H, t, *J*=7.42 Hz) 7.38 (2 H, t, *J*=7.13 Hz) 7.42 (0.5 H, d, *J*=5.08 Hz) 7.76 (0.5 H, d, *J*=8.40 Hz) 7.88 (2 H, d, *J*=8.01 Hz) 8.13 (2 H, d, *J*=9.18 Hz) 8.45 (0.5 H, d, *J*=4.49 Hz) 8.48 (2 H, s) 8.50 (0.5 H, s)

2 under vacuum and heat (2 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.82 - 1.96 (4 H, m) 2.17 (4 H, t, *J*=8.14 Hz) 2.27 (1.5 H, s) 2.46 (1.5 H, s) 2.69 (5.8 H, s) 3.24 - 3.34 (4 H, m) 4.78 (2 H, s) 7.18 (2 H, t, *J*=7.41 Hz) 7.26 (0.5 H, d, *J*=7.63 Hz) 7.33 (2 H, t, *J*=7.85 Hz) 7.64 (0.5 H, d, *J*=8.66 Hz) 7.84 (2 H, d, *J*=8.07 Hz) 8.14 (2 H, d, *J*=8.66 Hz) 8.35 (0.5 H, s) 8.42 (2 H, s)

3 under vacuum and heat (1.86 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.83 - 1.96 (3.8 H, m) 2.17 (3.7 H, t, *J*=8.00 Hz) 2.27 (1.5 H, s) 2.34 (1.5 H, s) 2.69 (5.4 H, s) 3.29 (3.8 H, t, *J*=7.04 Hz) 4.79 (2 H, s) 7.21 (2 H, t, *J*=7.48 Hz) 7.36 (2 H, t, *J*=7.70 Hz) 7.42 (0.5 H, d, *J*=5.14 Hz) 7.87 (2 H, d, *J*=8.07 Hz) 8.13 (2 H, d, *J*=8.66 Hz) 8.41 (0.5 H, d, *J*=5.14 Hz) 8.45 (0.5 H, br. s.) 8.46 (2 H, s)

4 under vacuum and heat (1.78 NMP)

¹H NMR (400 MHz, DMSO-*d*₆) δ ppm 1.84 - 1.94 (3.6 H, m) 2.17 (3.5 H, t, *J*=8.01 Hz) 2.33 (1.5 H, s) 2.69 (5.2 H, s) 3.29 (3.7 H, t, *J*=7.13 Hz) 4.80 (2 H, s) 7.22 (2 H, t, *J*=8.01 Hz) 7.38 (2 H, t, *J*=7.81 Hz) 7.42 (0.5 H, d, *J*=6.84 Hz) 7.76 (0.5 H, d, *J*=7.81 Hz) 7.88 (2 H, d, *J*=8.20 Hz) 8.13 (2 H, d, *J*=8.79 Hz) 8.45 (0.5 H, d, *J*=4.88 Hz) 8.48 (2 H, s) 8.50 (0.5 H, br. s.)

2b from supercritical CO₂ (1.82 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.82 - 1.96 (3.7 H, m) 2.17 (3.6 H, t, *J*=8.14 Hz) 2.30 (1.5 H, s) 2.49 (1.5 H, s) 2.69 (5.4 H, s) 3.29 (3.7 H, t, *J*=7.04 Hz) 4.79 (2 H, s) 7.21 (2 H, t, *J*=7.41 Hz) 7.36 (2.5 H, t, *J*=7.70 Hz) 7.75 (0.5 H, d, *J*=8.51 Hz) 7.87 (2 H, d, *J*=8.07 Hz) 8.13 (2 H, d, *J*=8.66 Hz) 8.40 (0.5 H, s) 8.46 (2 H, s)

3b from supercritical CO₂ (1.78 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.83 - 1.96 (3.6 H, m) 2.17 (3.7 H, t, *J*=8.00 Hz) 2.27 (1.5 H, s) 2.33 (1.5 H, s) 2.69 (5.1 H, s) 3.30 (3.6 H, t, *J*=7.04 Hz) 4.79 (2 H, s) 7.21 (2 H, t, *J*=7.48 Hz) 7.32 - 7.44 (2.5 H, m) 7.87 (2 H, d, *J*=8.22 Hz) 8.13 (2 H, d, *J*=8.80 Hz) 8.40 (0.5 H, d, *J*=4.70 Hz) 8.44 (0.5 H, br. s.) 8.46 (2 H, s)

4b from supercritical CO₂ (1.69 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.83 - 1.95 (3.5 H, m) 2.17 (3.4 H, t, *J*=8.07 Hz) 2.33 (1.5 H, s) 2.69 (4.8 H, s) 3.29 (3.5 H, t, *J*=7.04 Hz) 4.80 (2 H, s) 7.22 (2 H, t, *J*=7.48 Hz) 7.33 - 7.41 (2 H, m) 7.43 (0.5 H, d, *J*=4.99 Hz) 7.77 (0.5 H, d, *J*=8.36 Hz) 7.89 (2 H, d, *J*=8.22 Hz) 8.13 (2 H, d, *J*=8.66 Hz) 8.45 (0.5 H, br. s.) 8.48 (2 H, s) 8.50 (0.5 H, br. s.)

2 exposed to ether vapour (1.86 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.89 (3.8 H, quin) 2.17 (3.7 H, t, *J*=8.14 Hz) 2.30 (1.5 H, s) 2.69 (5.4 H, s) 3.30 (3.8 H, t, *J*=7.04 Hz) 4.80 (2 H, s) 7.21 (2 H, t, *J*=7.48 Hz) 7.37 (2.5 H, t, *J*=7.63 Hz) 7.77 (0.5 H, d, *J*=7.78 Hz) 7.88 (2 H, d, *J*=8.22 Hz) 8.13 (2 H, d, *J*=8.66 Hz) 8.41 (0.5 H, s) 8.47 (2.5 H, s)

3 exposed to ether vapour (1.83 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.83 - 1.97 (3.7 H, m) 2.17 (3.7 H, t, *J*=8.07 Hz) 2.27 (1.5 H, s) 2.33 (1.5 H, s) 2.69 (5.3 H, s) 3.30 (3.8 H, t, *J*=7.04 Hz) 4.79 (2 H, s) 7.21 (2 H, t, *J*=7.41 Hz) 7.32 - 7.40 (2 H, m) 7.42 (0.5 H, d, *J*=5.28 Hz) 7.87 (2 H, d, *J*=7.92 Hz) 8.13 (2 H, d, *J*=8.66 Hz) 8.40 (0.5 H, d, *J*=5.28 Hz) 8.44 (0.5 H, s) 8.46 (2 H, s)

4 exposed to ether vapour (1.72 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.89 (3.5 H, quin) 2.17 (3.5 H, t, *J*=8.07 Hz) 2.33 (1.5 H, s) 2.49 (1.5 H, br. s.) 2.69 (5 H, s) 3.30 (3.5 H, t, *J*=7.04 Hz) 4.80 (2 H, s) 7.22 (2 H, t, *J*=7.48 Hz) 7.33 - 7.43 (2.5 H, m) 7.76 (0.5 H, d, *J*=8.66 Hz) 7.89 (2 H, d, *J*=8.22 Hz) 8.12 (2 H, d, *J*=8.80 Hz) 8.44 (0.5 H, d, *J*=4.40 Hz) 8.48 (2.5 H, s)

2 exposed to DCM (1.76 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.83 - 1.96 (3.6 H, m) 2.18 (3.6 H, t, *J*=8.07 Hz) 2.31 (1.5 H, s) 2.51 (1.5 H, s) 2.70 (5 H, s) 3.25 - 3.34 (3.6 H, m) 4.80 (2 H, s) 7.17 - 7.26 (2 H, m) 7.33 - 7.42 (2.5 H, m) 7.79 (0.5 H, d, *J*=6.16 Hz) 7.88 (2 H, d, *J*=8.07 Hz) 8.14 (2 H, d, *J*=8.80 Hz) 8.43 (0.5 H, s) 8.47 (2 H, s)

3 exposed to DCM (1.82 NMP)

¹H NMR (300 MHz, DMSO-*d*₆) δ ppm 1.83 - 1.96 (3.8 H, m) 2.17 (3.8 H, t, *J*=8.07 Hz) 2.26 (1.5 H, s) 2.32 (1.5 H, s) 2.66 - 2.71 (5.3 H, m) 3.25 - 3.34 (3.8 H, m) 4.79 (2 H, s) 7.20 (2 H, t, *J*=7.85 Hz) 7.31 - 7.41 (2.5 H, m) 7.87 (2 H, d, *J*=7.63 Hz) 8.13 (2 H, d, *J*=8.66 Hz) 8.39 (0.5 H, d, *J*=5.28 Hz) 8.43 (0.5 H, s) 8.45 (2 H, s)

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