Electronic Supplementary Material (ESI) for CrystEngComm.
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2-D and 3-D porous structures from tetrakis(4-thyminylmethylphenyl)methane

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SUPPLEMENTARY INFORMATION

Table S1 Hydrogen-bond distances (Å) and angles (°) for (**I**)[a]

D–H···A	D–H	H···A	D···A	∠(D–H···A)
N2–H2···O8^{i}	0.88	1.99	2.853(5)	168.3
N4–H4···O1^{ii}	0.88	2.11	2.973(5)	165.1
N6–H6···O6^{iii}	0.88	1.95	2.795(5)	161.8
N8–H8···O2^{iv}	0.88	1.96	2.818(5)	165.8
C24–H241···O5^{v}	0.95	2.33	3.267(6)	170.5
C36–H361···O7^{v}	0.95	2.14	3.082(6)	170.4
C48–H481···O3^{v}	0.95	2.30	3.228(6)	167.0

[a] Symmetry codes i) x, $0.5 - y$, $0.5 + z$; ii) x, $1.5 - y$, $-0.5 + z$; iii) $2 - x$, $1 - y$, $1 - z$; iv) x, $0.5 - y$, $0.5 + z$; v) $1 - x$, $1 - y$, $1 - z$.

Table S2 Hydrogen-bond distances (Å) and angles (°) for (**II**)[a]

D–H···A	D–H	H···A	D···A	∠(D–H···A)
N4A–H4A···O1C^{i}	0.88	1.99	2.868(4)	175.6
N6A–H6A···O5A^{i}	0.88	1.95	2.817(4)	167.9
N8A–H8A···O2B^{ii}	0.88	1.98	2.843(5)	165.2
C24A–H24A···O5A^{iii}	0.95	2.45	3.391(5)	170.2
C36A–H36A···O7A^{iii}	0.95	2.18	3.107(5)	166.4
C48A–H48A···O3A^{iii}	0.95	2.40	3.305(6)	159.5
N2B–H2B···O1B^{iv}	0.88	1.97	2.843(4)	174.2
N4B–H4B···O6A^{i}	0.88	1.99	2.864(4)	170.2
N6B–H6B···O6B^{v}	0.88	1.95	2.828(4)	171.4
N8B–H8B···O2A^{vi}	0.88	1.92	2.783(6)	166.8
C24B–H24B···O5B^{vii}	0.95	2.26	3.197(5)	171.4
C32B–H32B···O2A^{viii}	0.95	2.22	3.124(6)	151.4
C36B–H36B···O7B^{vii}	0.95	2.22	3.074(6)	149.3
C37B–H37B···O4A^{i}	0.95	2.41	3.346(5)	159.7
C48B–H48B···O3B^{vii}	0.95	2.24	3.087(6)	148.2
O1C–H1C···O6B	0.883(19)	2.00(2)	2.874(4)	168(5)
C1C–H13C···O5B^{v}	0.98	2.35	3.261(6)	153.5

[a] Symmetry codes i) $1 - x$, $1 - y$, $2 - z$; ii) $-1 + x$, y, $1 + z$; iii) $-x$, $1 - y$, $2 - z$; iv) $1 - x$, $2 - y$, $-z$; v) $2 - x$, $2 - y$, $1 - z$; vi) x, $1 + y$, z; vii) $1 - x$, $2 - y$, $1 - z$; viii) $1 - x$, $1 - y$, $1 - z$.

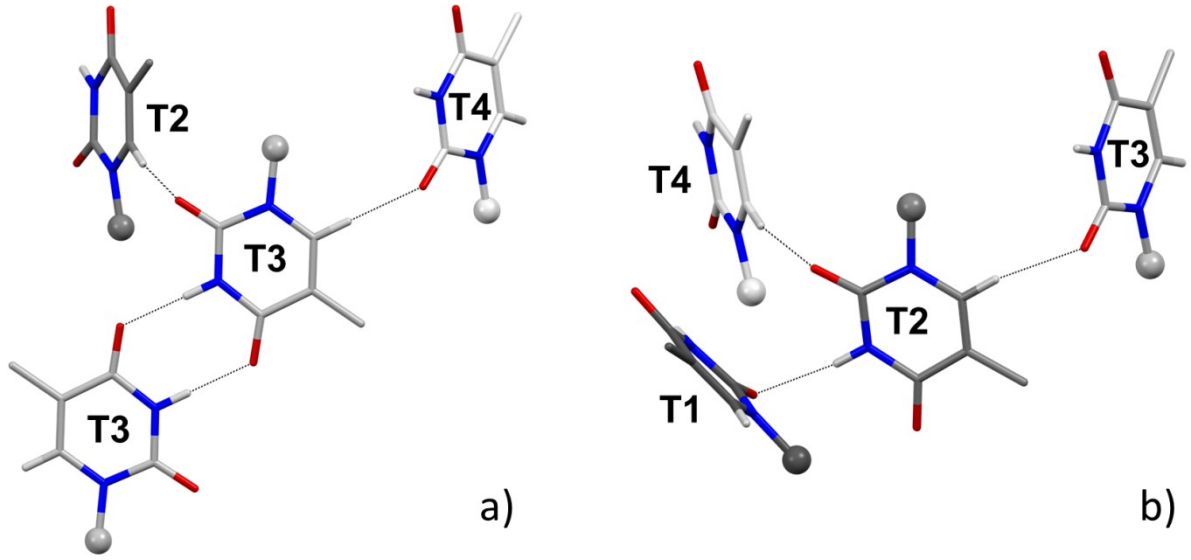

Fig. S1 Detailed view of intermolecular interactions in structure (**I**). The **T3**:::**T3** contact in a) is essentially identical to the **T1**:::**T4** ring system in Fig. 2b, while b) shows that **T2** is involved in only one hydrogen bond (to **T1**) in addition to the two C-H···O interactions connecting the dimer.

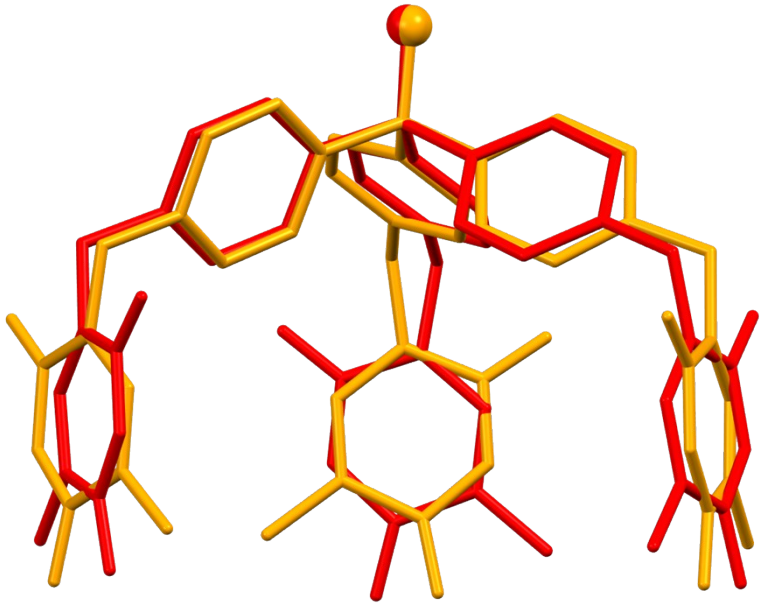

Fig. S2 Least squares fit between a 4-tmpm molecule of (**I**) (orange) and the inverted 4-tmpm molecule **A** of (**II**) (red), arm **1** being curtailed beyond C2A/C2 (small spheres). The RMSD-value is 1.945 Å compared to 0.691 Å for the fit shown in Fig. 6b.

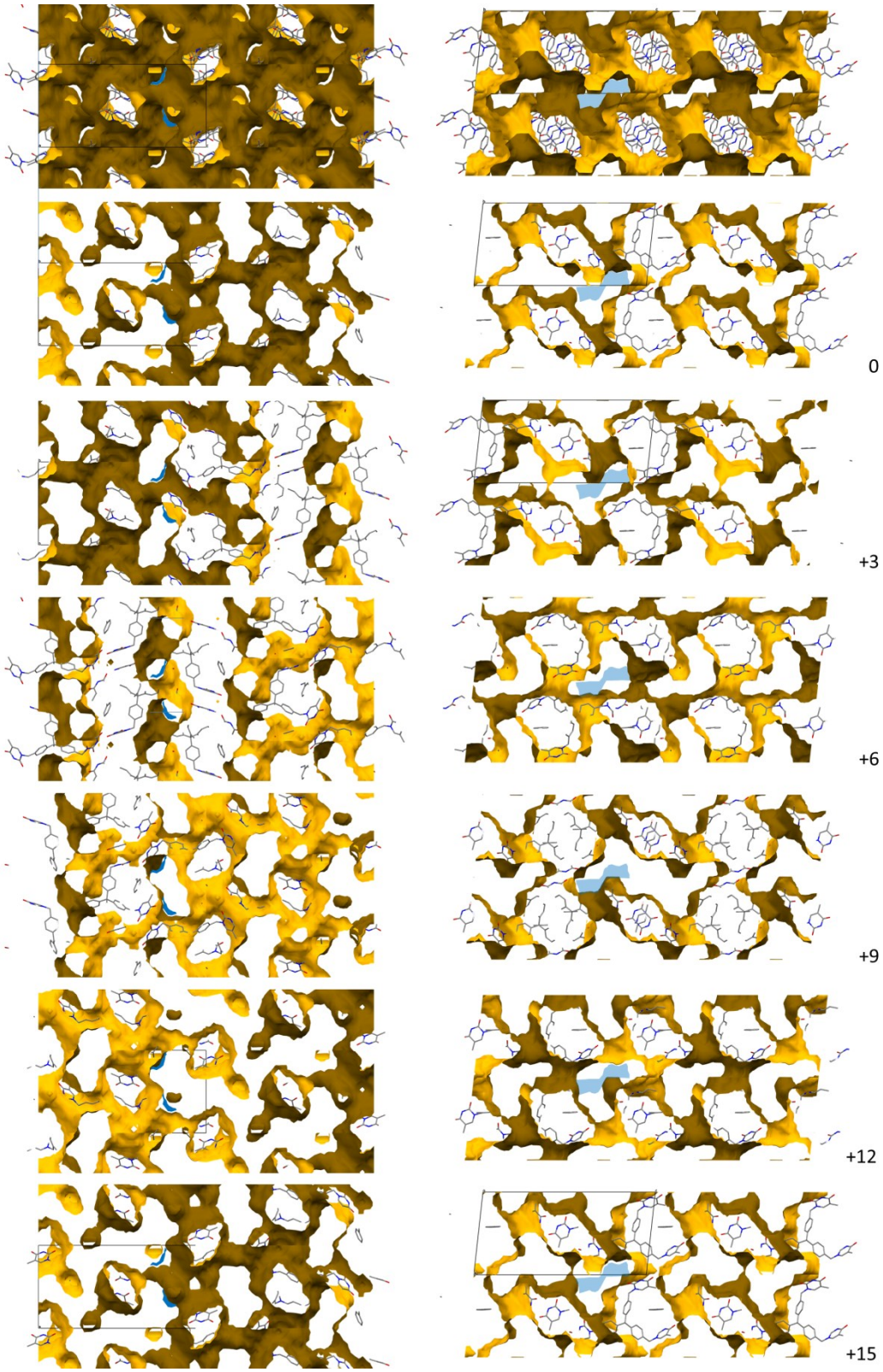

Fig. S3 Voids in the crystal structure of (**I**) calculated as in Fig. 4,[1] but viewed along the *a*-axis (top, left) and *b*-axis (top, right) (inner surface dark orange, outer surface light orange). Images below show 4 Å thick slices with vertical shifts of 3 Å between each as indicated to the right. As both axes are approximately 15 Å in length, 0 Å and +15 Å images are almost identical. Blue shades highlight the see-through holes in the top illustrations.

Experimental

^{1}H NMR spectra were recorded at 600 MHz with a Bruker AV 600 instrument. The decoupled ^{13}C NMR spectra were recorded at 150 MHz using instrument mentioned above. Electrospray mass (ESI) spectra were obtained on a Micromass QTOF II spectrometer. Melting points were determined with Büchi melting point B-545 apparatus and are uncorrected. Dry DMF was obtained from a MB SPS-800 solvent purification system. TMSCl was distilled from CaH_2. Triethylamine was dried over KOH and toluene over sodium.

1,1',1'',1'''-[4,4',4'',4'''-Methanetetrayltetrakis(benzene-4,1-diyl)tetrakis(methylene)]tetrakis[5-methylpyrimidine-2,4(1*H*,3*H*)-dione] (4-tmpm).

To a magnetically stirred suspension of thymine (416 mg, 3.30 mmol) in dry toluene (10 mL) was added chlorotrimethylsilane (0.881 mL, 6.94 mmol). Triethylamine (1.15 mL, 8.25 mmol) in toluene (10 mL) was added and the resulting mixture was stirred for 48 h under N_2. The mixture was filtered and the solid washed with toluene (3 x 5 mL) and the combined filtrates were evaporated. The residue was dissolved in dry DMF (15 mL) and tetrakis(4-(bromomethyl)phenyl)methane[2] (415 mg, 0.600 mmol) was added. The reaction mixture was stirred under N_2 and heated at 160 °C for 24 hours. The resulting mixture was cooled to ambient temperature and evaporated in vacuo. The crude product was purified by flash chromatography on silica eluting with 5% MeOH saturated with NH_3 in CH_2Cl_2; yield 255 mg (49 %) colorless powdery solid, m.p. > 380 °C. ^{1}H NMR (DMSO-d_6, 600 MHz) δ 11.28 (s, 4H, NH), 7.61 (s, 4H, H-6), 7.16 (m, 16H, Ph), 4.78 (s, 8H, CH_2), 1.73 (s, 12H, CH_3); ^{13}C NMR (DMSO-d_6, 600 MHz) δ 164.26 (C-4), 150.98 (C-2), 145.65 (C in Ph), 141.44 (C-4), 134.57 (C in Ph), 130.40 (CH in Ph), 126.64 (CH in Ph), 109.00 (C-5), 63.73 (C), 49.60 (CH_2), 11.95 (CH_3); MS (ESI): 895.3 (100, *M*+Na).

Reference

1. C. F. Macrae, I. J. Bruno, J. A. Chisholm, P. R. Edgington, P. McCabe, E. Pidcock, L. Rodriguez-Monge, R. Taylor, J. van de Streek and P. A. Wood, *J. Appl. Crystallogr.*, 2008, **41**, 466-470.