Electronic Supplementary Material (ESI) for CrystEngComm

Supporting Information

Co-crystals of *tetrakis*-1,2,3,4-(4'-carboxyphenyl) cyclobutane with dipyridyl spacers: design and serendipity

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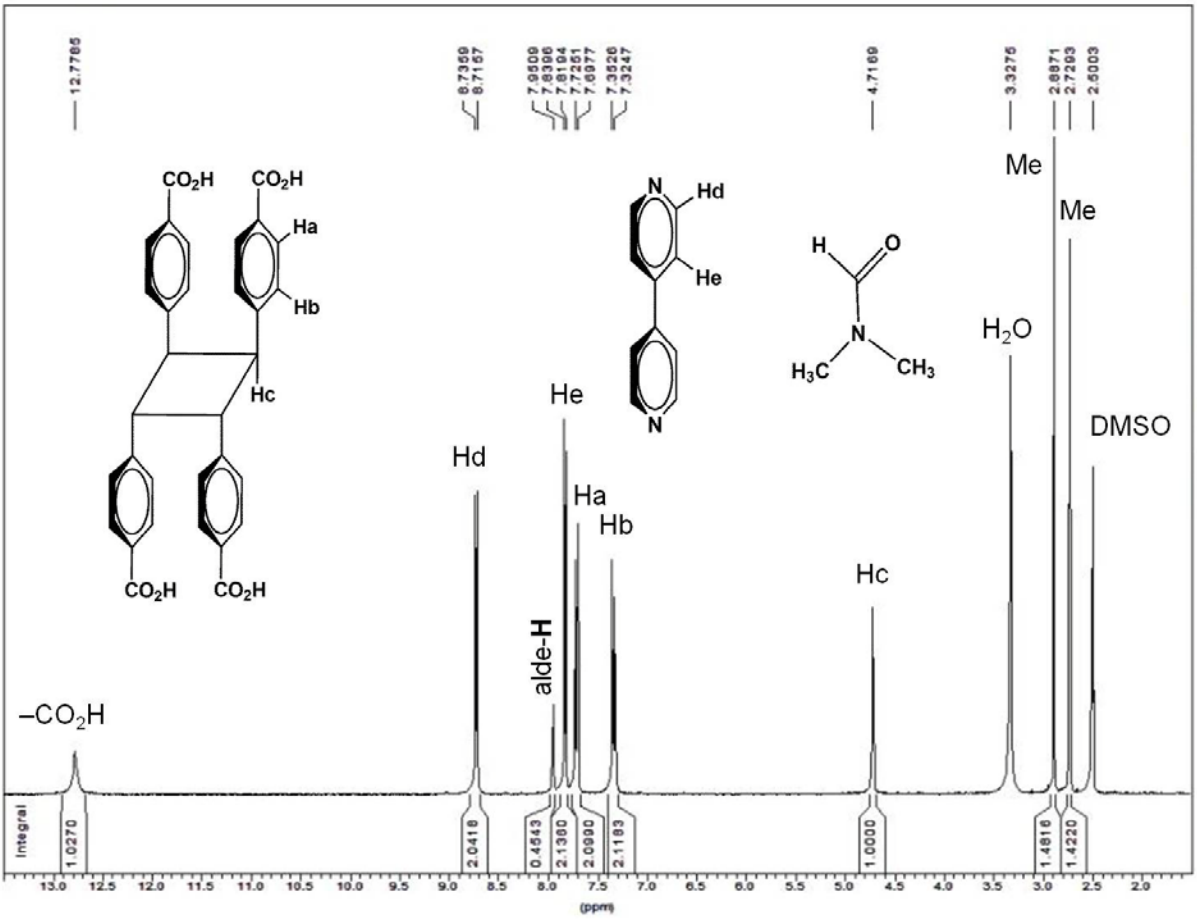

Figure S1: ^{1}H-NMR (300 MHz, d_6-DMSO) spectrum of **1**. The integration values show that the ratio of TCCB and 4,4'-bpy is 1:2.

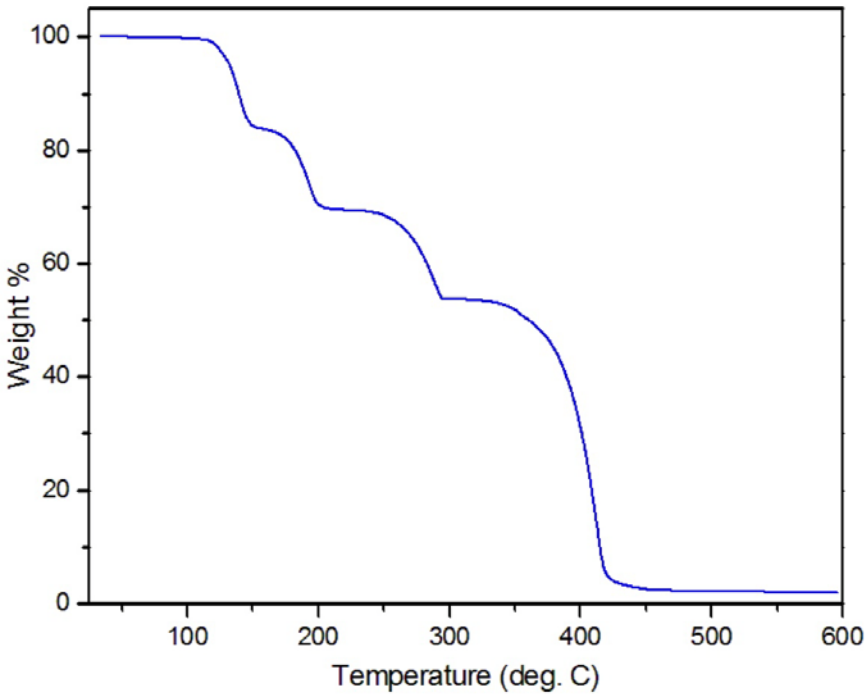

Figure S2: TGA of **1**. The observed solvent (DMF) loss is 15.8% where as the calculated value is 14.7 %.

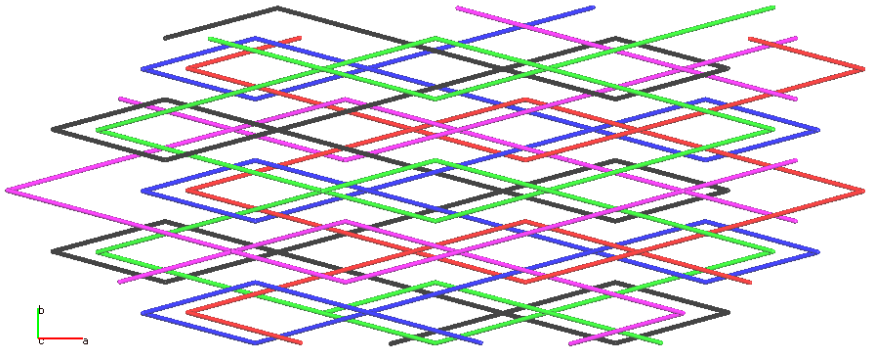

Figure S3: 5 Different interpenetrated ‘dmp’ nets in **1** are shown in 5 different colours.

Table S1 The hydrogen bonding parameters for **1**

D-H···A	D(D-H) (Å)	D(H···A) (Å)	D(D···A) (Å)	<(DHA) (°)	Symmetry Operator
O1-H1···N1	0.84	1.85	2.683(4)	174.4	-x, 1-y, 1-z
O3-H3···N2	0.84	1.84	2.673(4)	175	-x, 1-y, 1-z
C1S-H1S···O2	0.95	2.48	3.195(5)	132.2	-x, 1-y, 1-z
C7S-H7S···O2	0.95	2.46	3.340(5)	154.5	x, y, -1+z
C9S-H9S···O2	0.95	2.46	3.345(5)	156	-x,1-y,1-z
C10S-H10S···O4	0.95	2.53	3.211(5)	128.3	1-x, 1-y, 1-z

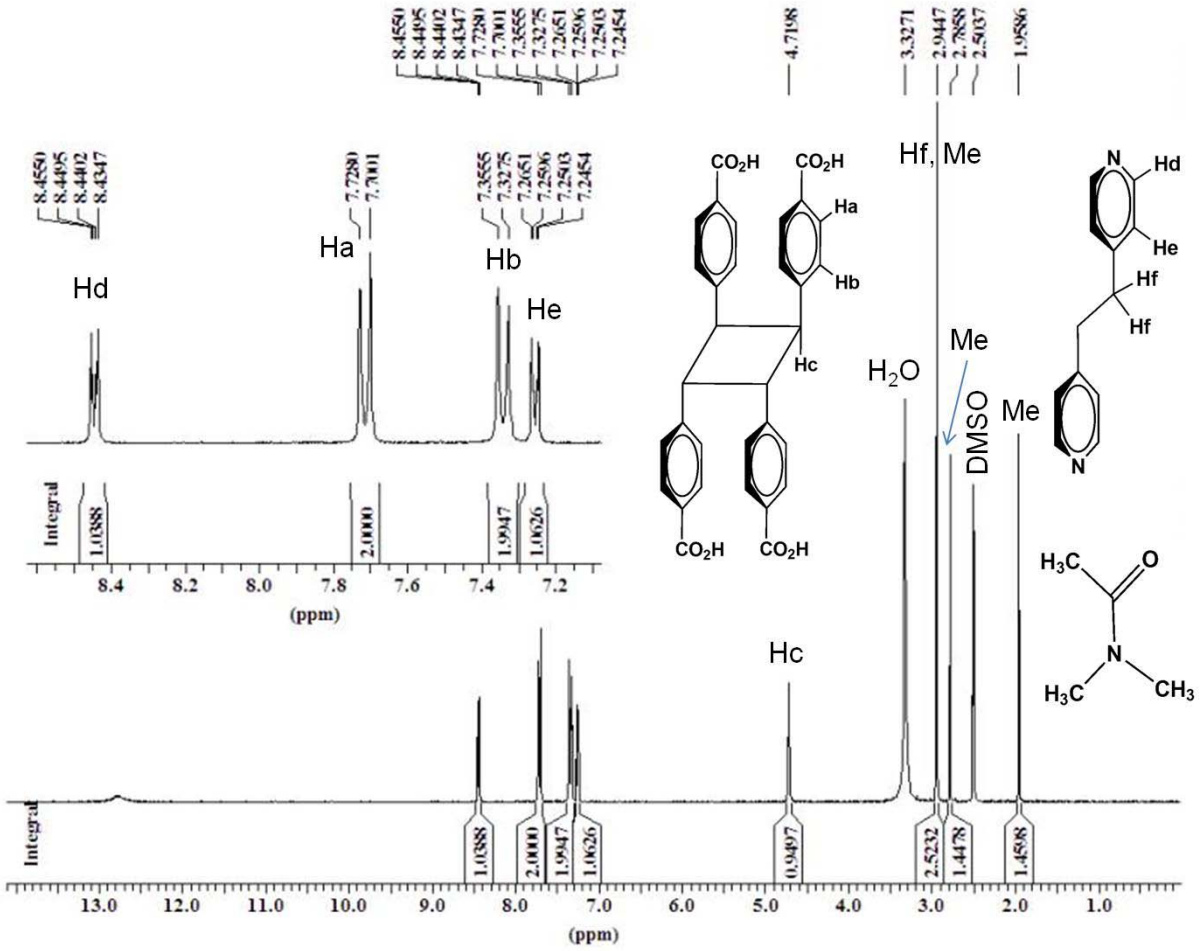

Figure S4: ^{1}H-NMR (300 MHz, d_6-DMSO) spectrum of **2**. The integration values show that the ratio of TCCB and 4,4'-bpethane is 1:1.

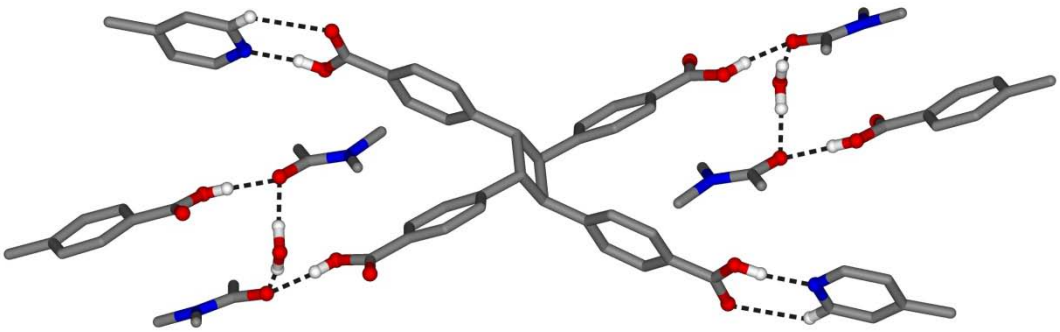

Figure S5: Crystal structure of **2**. Supramolecular interactions around each TCCB are shown.

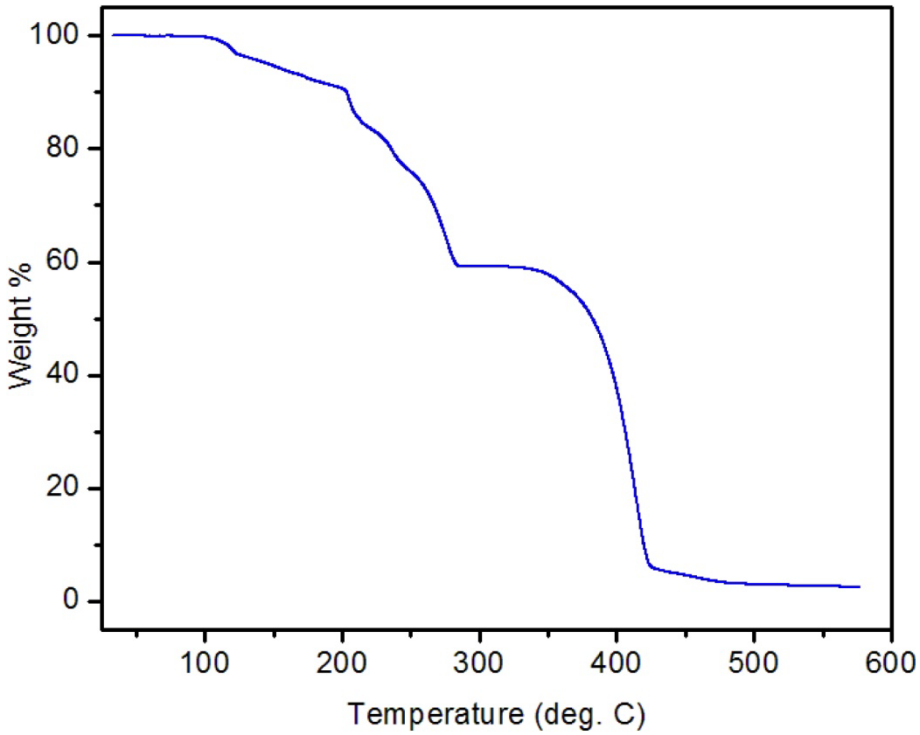

Figure S6: TGA of **2**. The weight loss observed at 285 °C is 40.7% which can be matched with the sum of solvent loss (21.05%) and weight loss due to decarboxylation of TCCB (19.27%).

Table 2 The hydrogen bonding parameters for **2**

D-H···A	D(D-H) (Å)	D(H···A) (Å)	D(D···A) (Å)	<(DHA) (°)	Symmetry Operator
O1-H1···N1	0.84	1.73	2.560(3)	172	x, -1+y, -1+z
O1W-H1WA···O5	0.91	1.96	2.855(5)	168(4)	1-x, y, 1/2-z
O3-H3···O5	0.84	1.76	2.594(3)	172	1-x, y, 1/2-z
C17-H17···O2	0.95	2.57	3.260(4)	130	x, 1+y, 1+z
C20-H20···O4	0.95	2.57	3.340(4)	138	1-x, y, 3/2-z

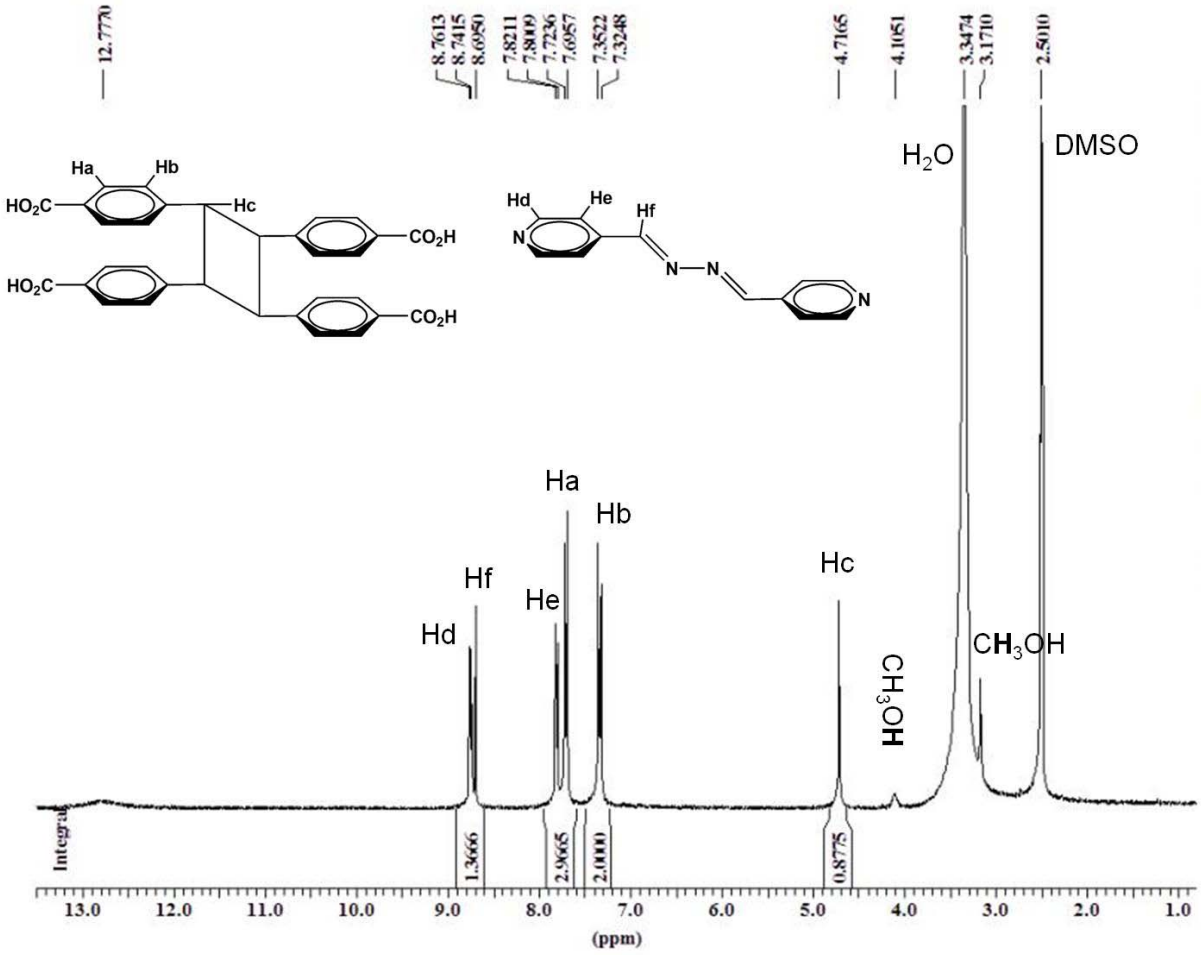

Figure S7: ^{1}H-NMR (300 MHz, d_6-DMSO) spectrum of **4**.

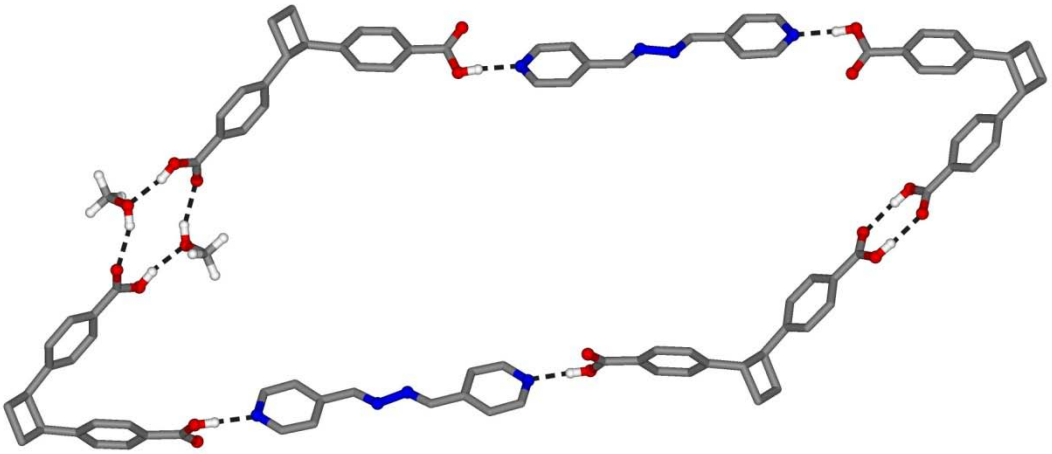

Figure S8: Supramolecular synthons and hydrogen bonded motifs in **4** are shown. One single loop of (4,4) connected net in **4**.

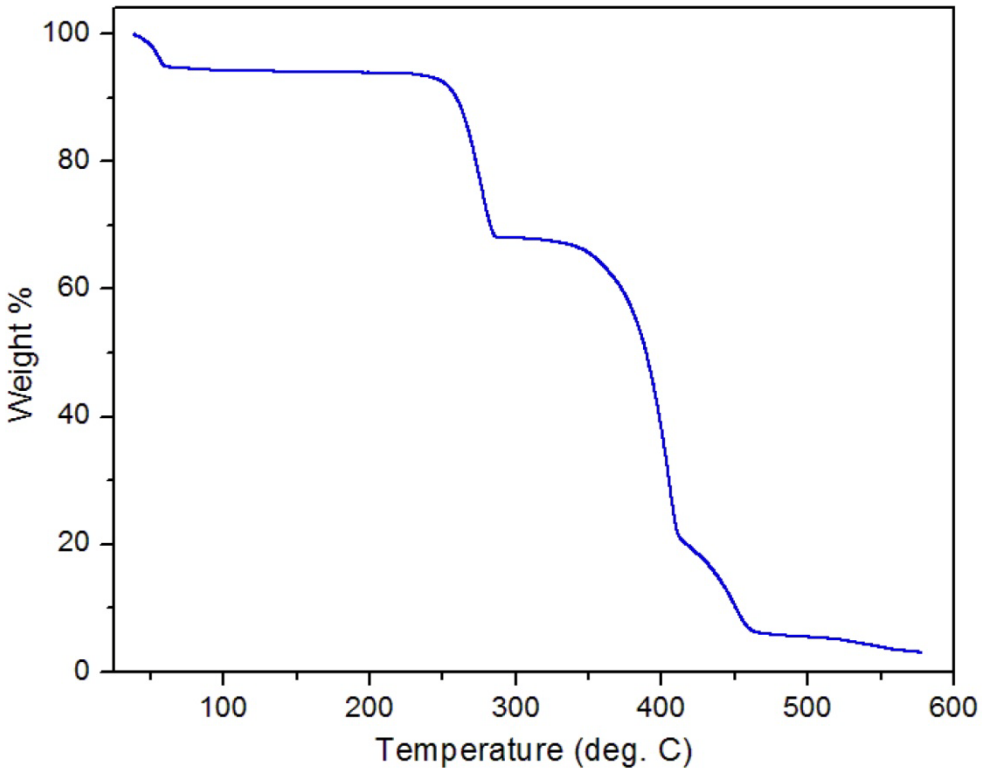

Figure S9: TGA plot for **4**. The observed weight loss in TGA experiment is 5.1%, whereas the calculated value is 4.1%.

Table 7.4 The hydrogen bonding parameters for **4**

D-H···A	D(D-H) (Å)	D(H···A) (Å)	D(D···A) (Å)	<(DHA) (°)	Symmetry Operator
O1S-H1S···O3	0.84	1.95	2.735(2)	156	x+1/2, -y+1/2, z+1/2
O8-H8···O7	0.84	1.84	2.669(2)	172	-x, -y+1, -z+1
O6-H6···N3	0.84	1.80	2.638(3)	175	x-1, y-1, z
O4-H4···O1S	0.84	1.83	2.657(2)	169	-x+1/2, y-1/2, -z+1/2
O2-H2···N1	0.84	1.79	2.628(3)	176	
C27-H27···O2	0.95	2.45	3.313(3)	151	1/2-x, -1/2+y, 1/2-z

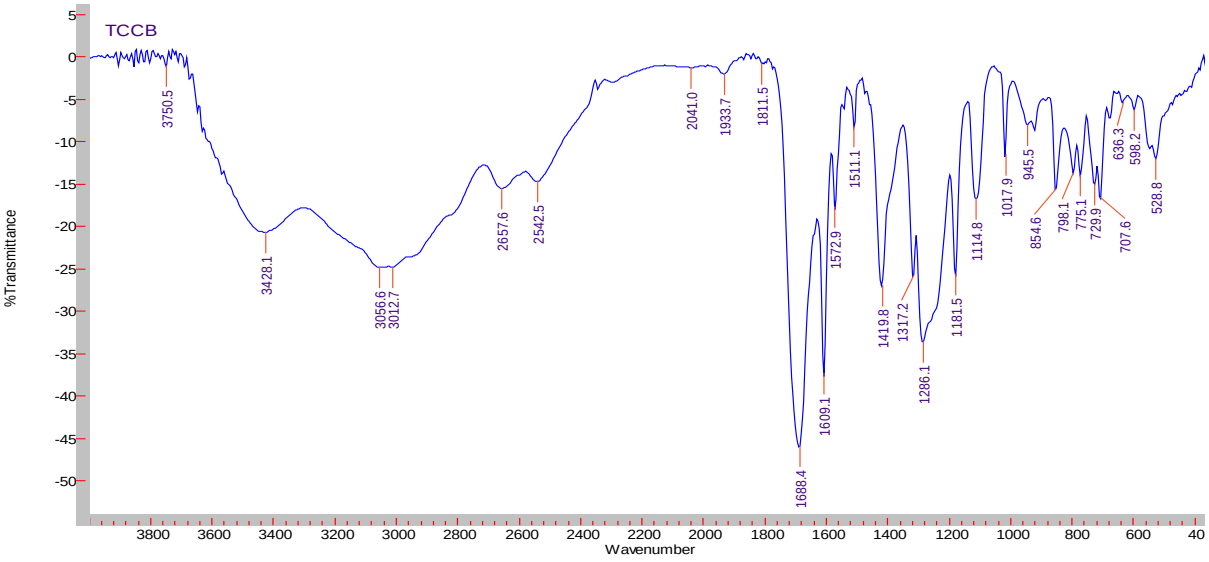

Figure S10: FT-IR spectrum for TCCB alone

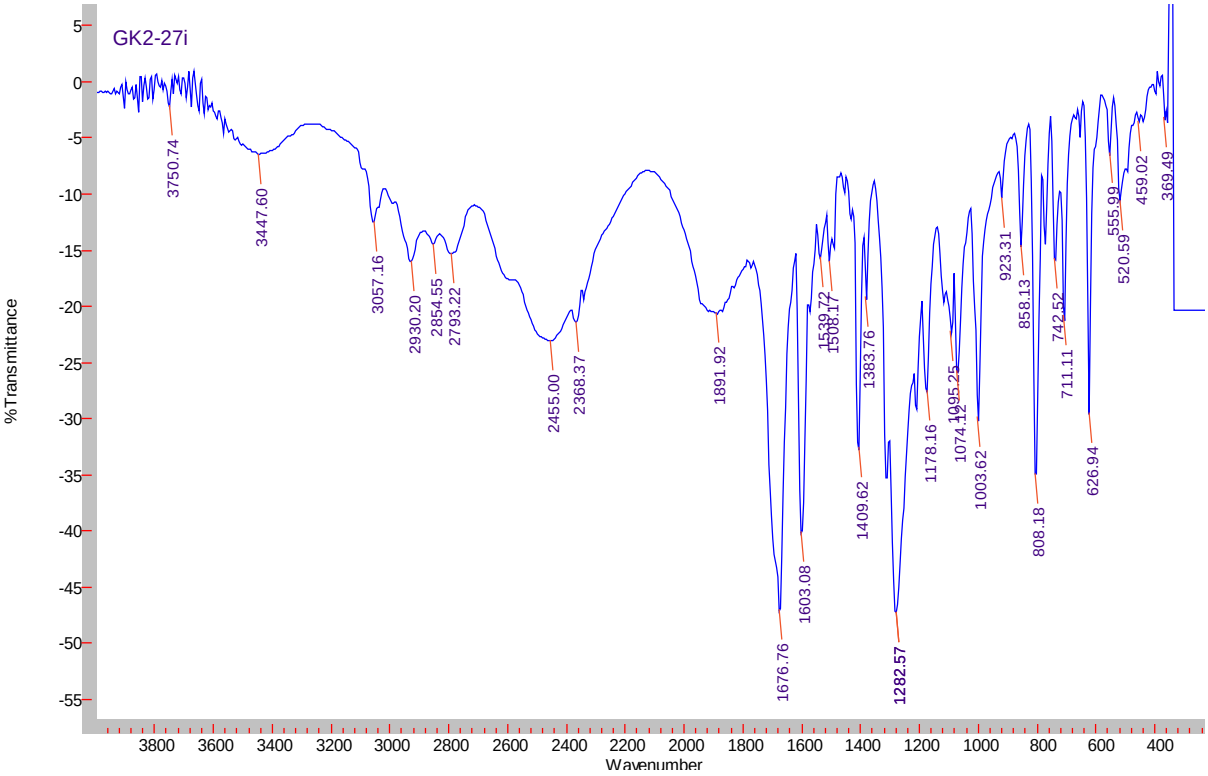

Figure S11: FT-IR spectrum for co-crystal **1**

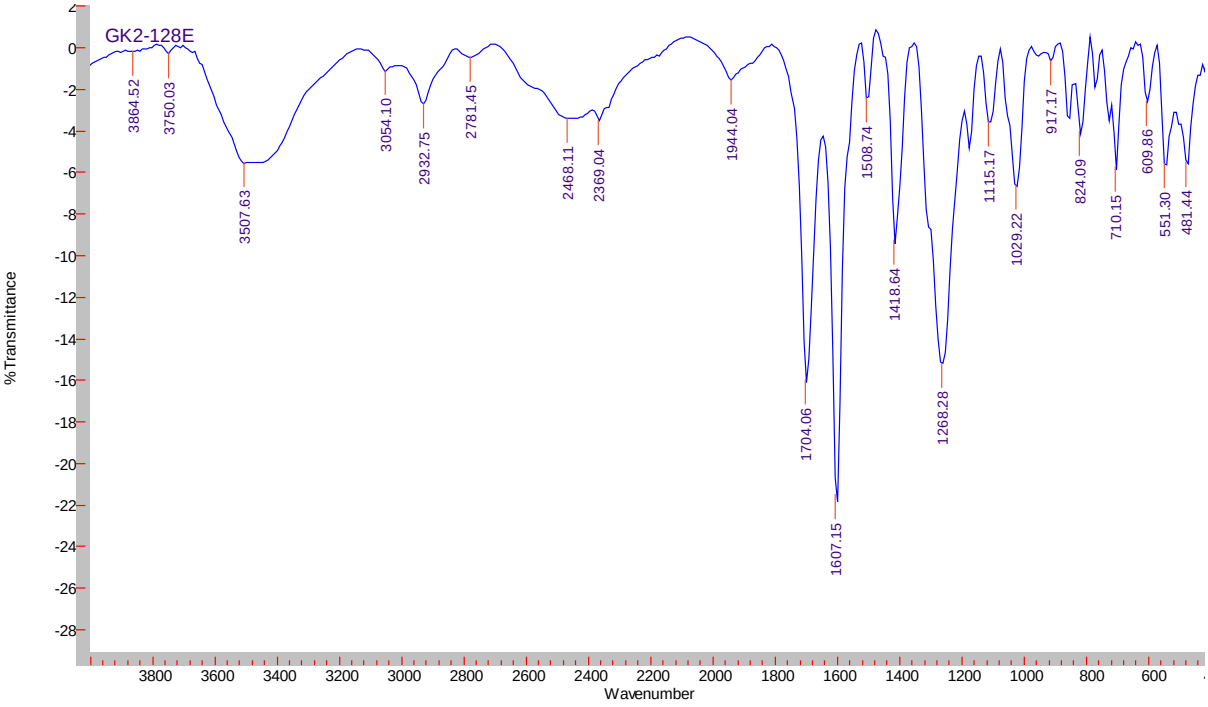

Figure S12: FT-IR spectrum for co-crystal **2**

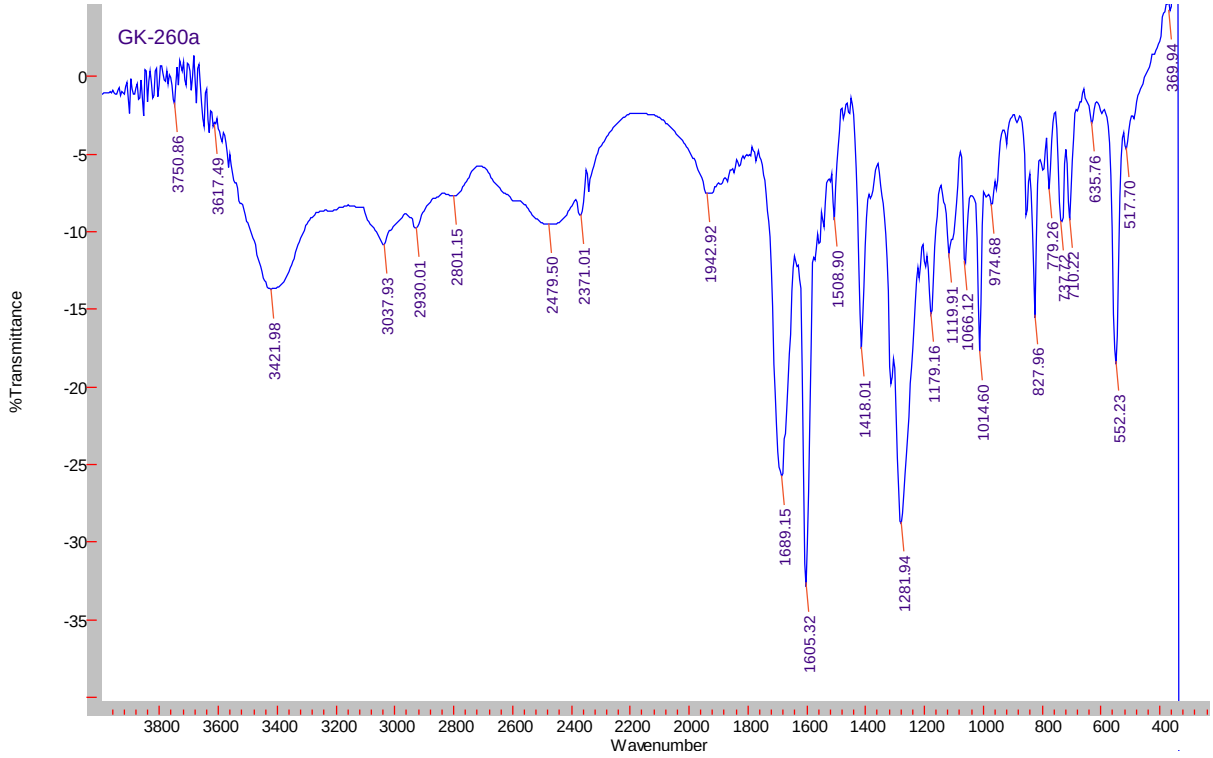

Figure S13: FT-IR spectrum for co-crystal **3**

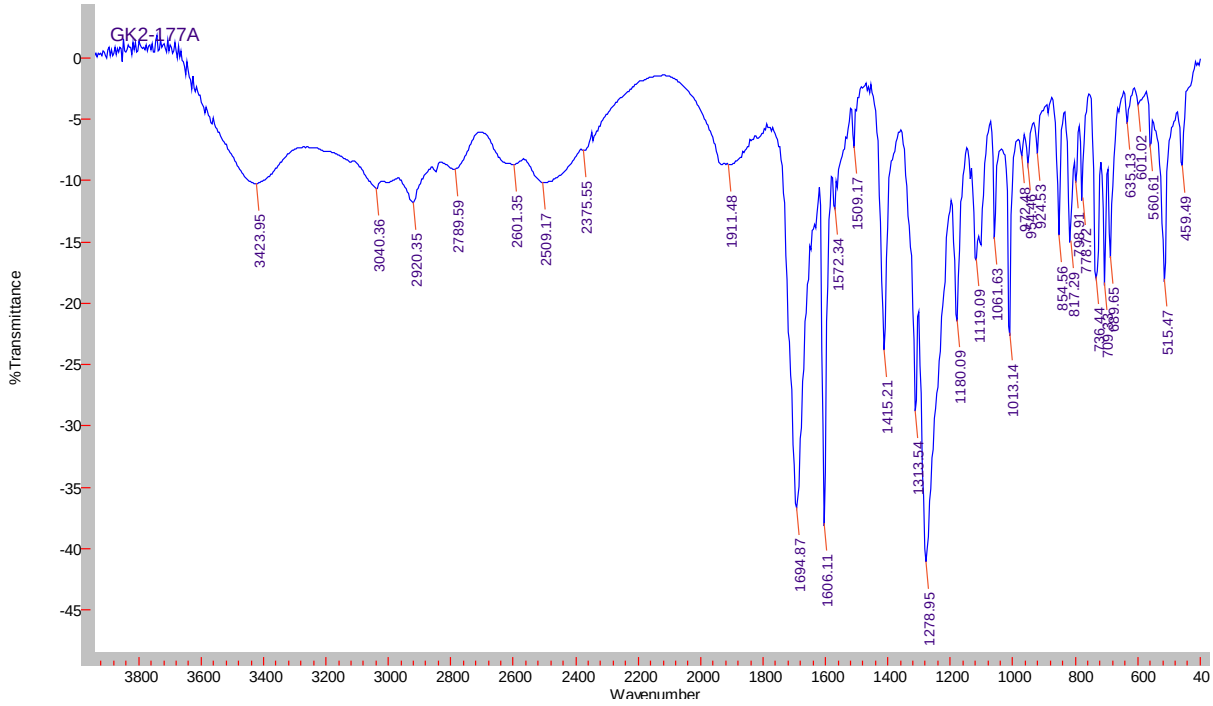

Figure S14: FT-IR spectrum for co-crystal **4**

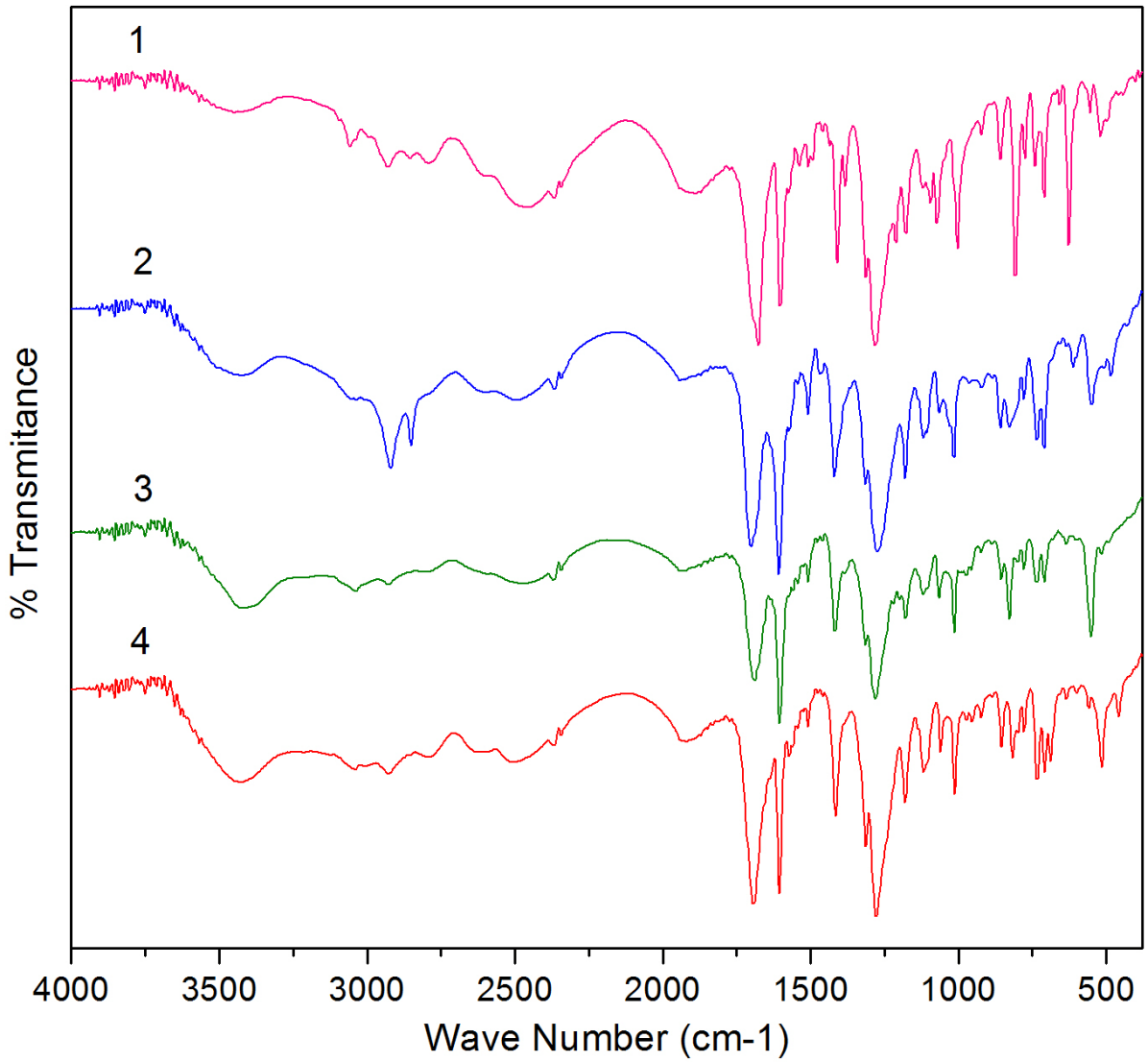

Figure S15: Overlay plot of IR spectra for co-crystals **1** – **4**.