Electronic Supplementary Material (ESI) for CrystEngComm

Inclusion of Chiral Guest in a Centrosymmetric Organic Host Lattice

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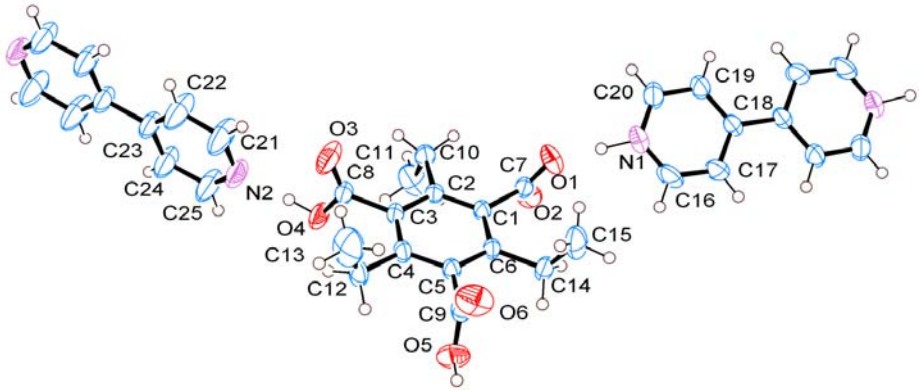
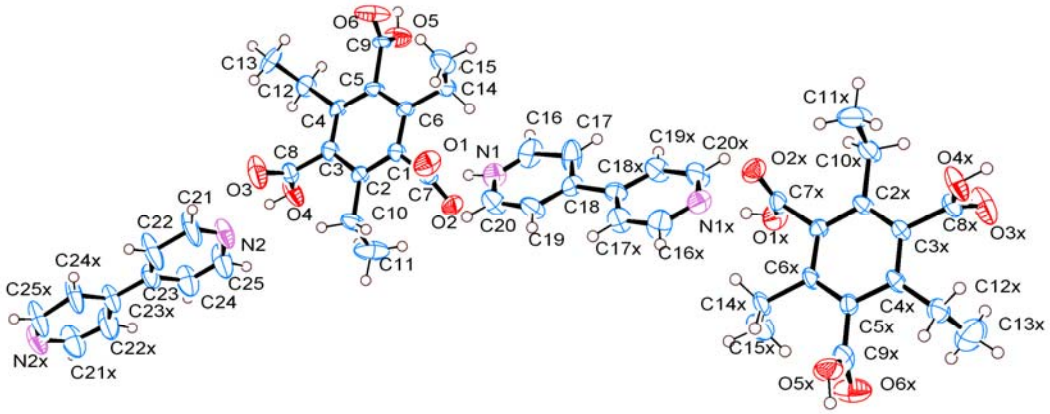
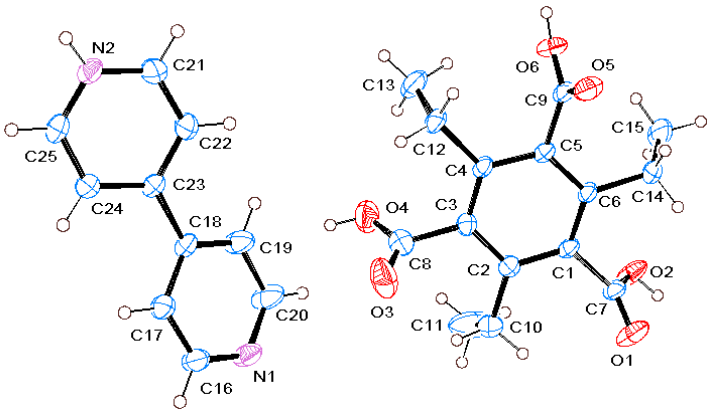
Supplementary Information



(a)



(b)



(c)

Fig. S1: ORTEP of **(a)** racemic solvate, **1**, **(b)** chiral solvate, **2**, **(c)** guest free complex, **3**, drawn at 50 % probability level.

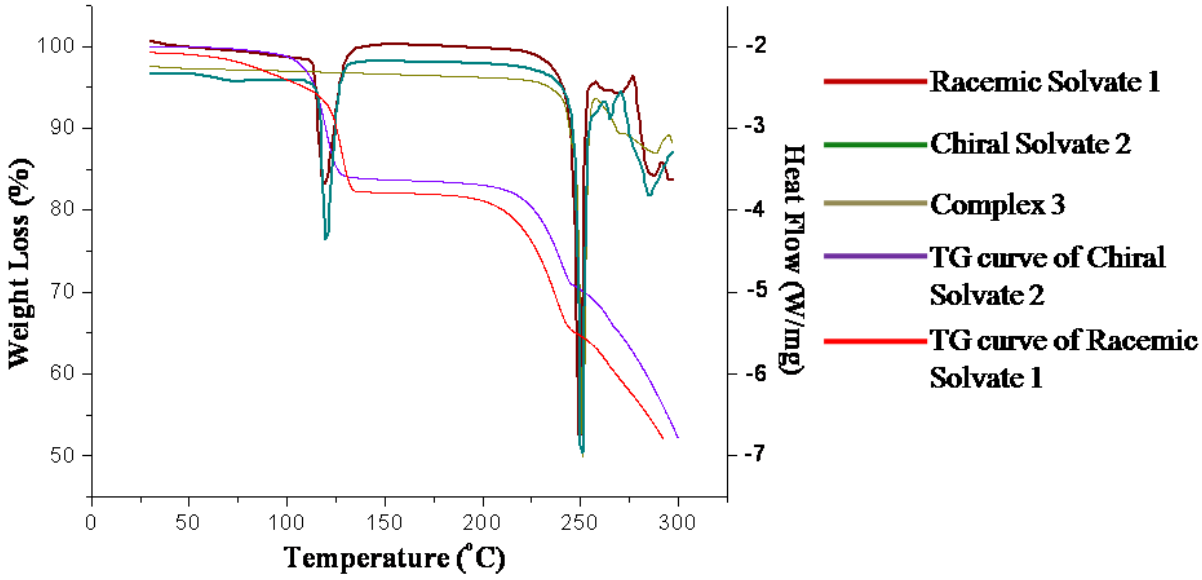
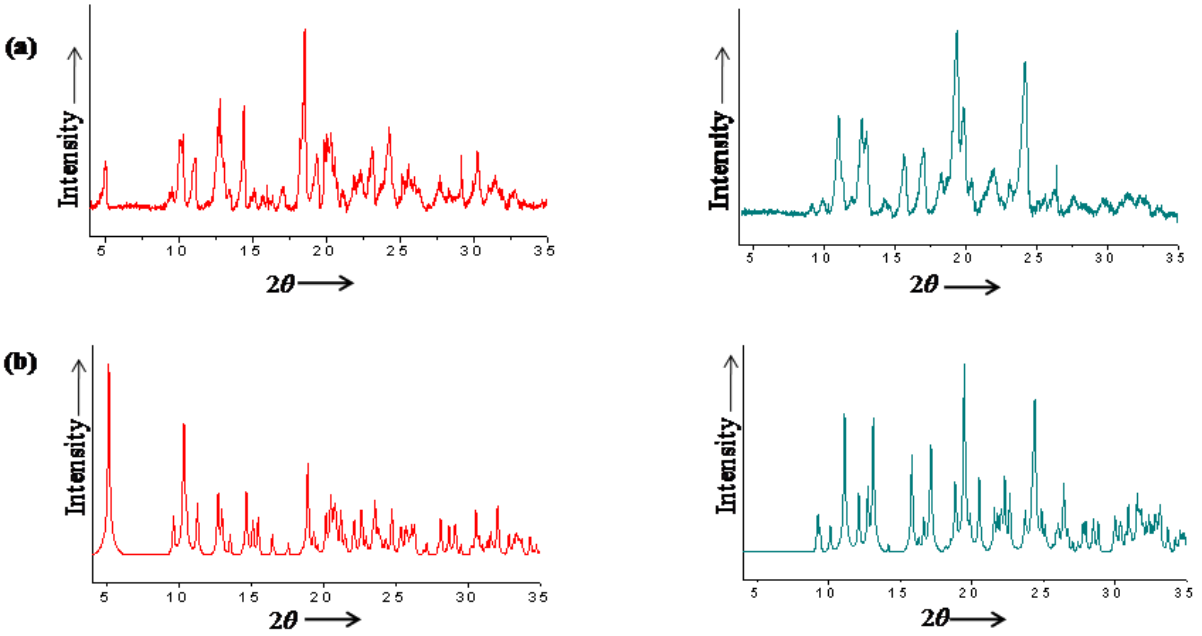


Fig. S2: TG and DSC plots of the solvates and the guest free complex. The % weight loss due to guest release from the systems **1** and **2** are 15.83 and 15.95 respectively. These values are close to the calculated value 16.36 % for the 1:1:1 ratio.



(b)

Fig. S3: (a) Experimental PXRD patterns of the chiral solvate, **2** (red) and after removing the guest by heating (green), (b) simulated PXRD patterns of the chiral solvate **2** (red) and guest free complex **3**.

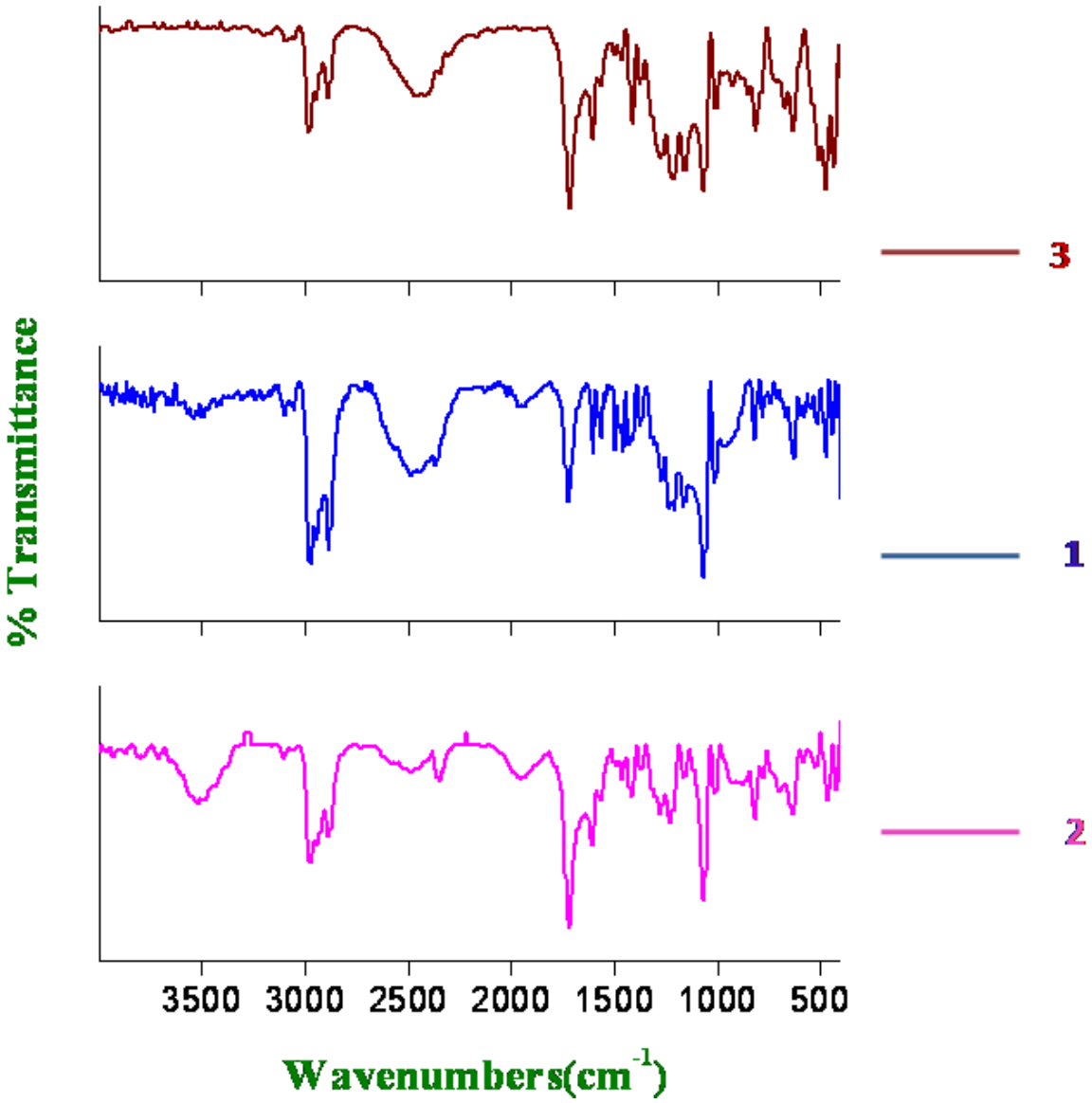


Fig. S4: IR spectra of the three systems, **1**, **2** and **3** reported in the article. Spectrum for **3**, ν (CO, carboxylic acid) 1717.5 cm^{-1}, ν_{anti} (COO^{-}) 1604.4 cm^{-1}, ν_{sym} (COO^{-}) 1370.1 cm^{-1}. Spectrum for **1**, ν (CO, carboxylic acid) 1724.2 cm^{-1}, ν_{anti} (COO^{-}) 1604.9 cm^{-1}, ν_{sym} (COO^{-}) 1368.6 cm^{-1}. Spectrum for **2**, ν (CO, carboxylic acid) 1719.9 cm^{-1}, ν_{anti} (COO^{-}) 1607.8 cm^{-1}, ν_{sym} (COO^{-}) 1368.3 cm^{-1}.

Table S1: Crystal data for the solvates, **1** and **2**, and the guest free complex **3**.

Complex	**Racemic solvate 1 (Squeezed)**	**Chiral solvate 2 (Squeezed)[a]**	**Guest free complex 3**
Formula	$C_{15}H_{18}O_6$, $C_{10}H_8N_2$, $C_5H_{12}O$	$2C_{15}H_{18}O_6$, $2C_{10}H_8N_2$, $2\ C_5H_{12}O$	$C_{15}H_{18}O_6$, $C_{10}H_8N_2$
*M*r	538.62	1077.25	450.48
Crystal system	Triclinic	Triclinic	Monoclinic
Space group	*P*-1	*P*1	$P2_1/n$

a (Å)	9.0116(7)	8.9857(2)	11.4001(11)
b (Å)	9.4080(6)	9.4136(2)	17.4597(16)
c (Å)	18.0095(14)	17.9866(4)	11.6727(9)
α (°)	83.760(6)	83.674(2)	
β (°)	76.517(7)	76.620(2)	101.406(9)
γ (°)	79.610(6)	79.597(2)	
V (Å^3)	1456.89(19)	1452.09(6)	2277.5(4)
T (K)	298(2)	298(2)	298(2)
Z	2	1	4
F(000)	576	576	952
μ (mm^{-1})	0.087	0.088	0.094
Ref. collected/unique	11664/ 6586	51666 / 14074	15057/5311
R_{int}	0.0237	0.0377	0.0355
Parameters	310	615	314
Final R indices [I>2σ(I)]	R_1 = 0.0566, wR_2 = 0.1785	R_1 = 0.0483 wR_2 = 0.1363	R_1 = 0.0637 wR_2 = 0.1799
R indices (all data)	R_1 = 0.0884, wR_2= 0.1877	R_1 = 0.0727 wR_2 = 0.1451	R_1 = 0.1122 wR_2 = 0.1967
Goodness of fit on F^2	0.986	0.903	1.048

[a] The E*E-1 value for the P1 structure is 1.027 which is closer to the value of a centrosymmetric structure. This could be due to the presence of ordered centrosymmetric host network and highly disordered chiral guest molecules. As a result the diffraction statistics is dominated by the host network rather than the guest molecules. The Flack parameter of this structure is 0.2(11).

Table S2: Normalized hydrogen bond parameters in the solvates, **1** and **2**, and the guest free complex **3**.

D—H···A	**D—H(Å)**	**H···A(Å)**	**D···A(Å)**	**D—H···A(°)**
Racemic solvate **1**, in *P*-1				
N(1)—H(1)···O(1)^{i}	1.01	1.58	2.569(2)	167
O(4)—H(4)···N(2)^{ii}	0.98	1.62	2.599(2)	176
O(5)—H(5)···O(2)^{iii}	0.98	1.77	2.727(2)	162
Chiral solvate **2**, in *P*1				
N(1)—H(1)···O(1)^{iv}	1.01	1.58	2.568(4)	166
O(4)—H(4)···N(2)^{v}	0.98	1.64	2.613(5)	173
O(4X)—H(4X)···N(2X)^{vi}	0.98	1.62	2.595(4)	172
O(5X)—H(5X)···O(2X) ^{vii}	0.98	1.75	2.725(4)	170
O(1X)—H(1X)···N(1X)^{viii}	0.98	1.61	2.565(4)	162
O(5)—H(5)···O(2)^{ix}	0.98	1.73	2.708(4)	175
Guest free complex **3**, in $P2_1/n$				
O(2)—H(2)···N(1)^{x}	0.98	1.63	2.610(3)	174

O(4)—H(4)···O(5)^{xi}	0.98	1.76	2.702(3)	160
O(6)—H(6)···N(2)^{xii}	0.98	1.57	2.545(3)	173

Symmetry codes: (i) x, y, z; (ii) x, y, z; (iii) −1+x, y, z; (iv) x, y, z; (v) x, y, z; (vi) −2+x, 3+y, 1+z; (vii) −1+x, y, z; (viii) x, y, z; (ix) 1+x, y, z; (x) 0.5−x, −0.5+y,1.5−z; (xi) −0.5+x, 0.5−y, −0.5 +z; (xii) 1.5−x, -0.5+y, 0.5−z.

Materials and Methods:

Racemic 2-methyl-1-butanol, (*S*)-(-)-2-methyl-1-butanol (99%) and 4,4′-bipyridine **(BPY)** were purchased from Sigma Aldrich. 2,4,6-triethyl-1,3,5-benzenetricarboxylic acid **(TEB)** was prepared in laboratory by combining different procedures reported in literature cited in the reference section. Crystals were prepared by slow solvent evaporation method at room temperature. The PXRD spectrum of solvate **2** and its apohost were recorded at room temperature using Cu *Kα* radiation (λ =1.54056 Å) from X-ray diffractometer (model: X-pert Panalytical). The spectrum was recorded with 2θ ranging from 2° to 45° and a step size of 0.02°. The apohost was prepared by grinding and subsequent heating of solvate **2** crystals at 135-140 °C under vacuum. TG-DSC experiments were performed on TA Instruments at a heating rate of 10 °C/min. Optical rotation for the solvates were measured at room temperature, in Autopol IV—Rudolph Research Analytical Polarimeter at two different concentrations (0.01 g/ml, 0.02 g/ml) in ethanol medium. IR spectra were recorded on a Fourier transform infrared spectrometer (model: Thermo Nicolet 6700) in the range of 400—4000 cm^{-1} using KBr.