Electronic Supplementary Information(ESI)

Molecular tiltation and supramolecular interactions induced uniaxial NTE and biaxial PTE in bis-imidazole based co-crystals

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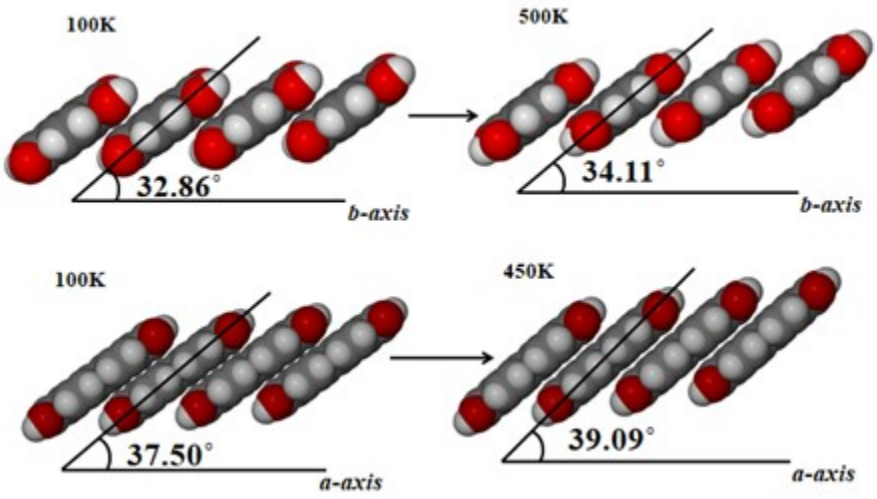


1. Experimental Section: Synthesis of the ligand **BIMB** and co-crystals **BIMB-TA** and **BIMB-BPDCA**

All chemicals were of reagent grade and were used without further purification.The ligand [1,4-bis[(2-methylimidazol-1-yl)methyl]benzene] **(BIMB)**was synthesized by slightly modified procedure reported in the literature.[1]In a two neck round bottom flask 1g (14.70mmol) of imidazole was dissolved in 15ml of dry THF solvent. The resulting solution was stirred at room temperature for 1h. Under inert atmosphere 500mg (20 mmol) of NaH was added to the imidazole solution of THF.The resulting suspension was stirred at room temperature for another 6h. THF solution of α, α'-dichloro-*p*-xylene(700mg, 4 mmol)was added dropwise to the reaction mixture, the resulting solution is further stirred at room temperature for another 5h. The solvent was removed under vacuum and the product was extracted with dichloromethane solvent. Colorless solid was obtained (approximately 850mg) and resulting solid was dissolved in hot water to get the colorless crystals of **BIMB**. Molecular structure of **BIMB** ligand has been characterized by ^{1}H NMRspectroscopy and IR. This compound was used to synthesize new Organic co-crystals with terephthalic acid **(TA)** and 4,4'-biphenyl dicarboxylic acid **(BPDCA).** Both the co-crystals have been prepared by solvent assisted grinding method. Few drops of methanol solvent were added in the mixture of components taken in 1:1 stoichiometric ratio and grinded in a mortar and pestle.

BIMB-TA co-crystal has been prepared by mixing 25mg (104.91mmol) of **BIMB** and 17.42mg(104.91mmol) of **TA**, while **BIMB-BPDCA** co-crystal has been prepared by taking 30mg (125.89mmol) of **BIMB** and 30.49mg (125.89mmol) of **BPDCA**. In both the cases the mixture was grinded by adding few drops of methanol in mortar and pestle. After grinding, the powdered material was dissolved in 10ml of methanol (for **BIMB-TA**) and 15ml of DMF (for **BIMB-BPDCA**) the resulting mixture was mechanically stirred for 15 minutes. The resulting solution is filtered and kept at room temperature for slow evaporation of the solvent. Within a week colorless

block shaped crystals were obtained. The co-crystals were characterized by IR, PXRD and the structures were confirmed by SCXRD.

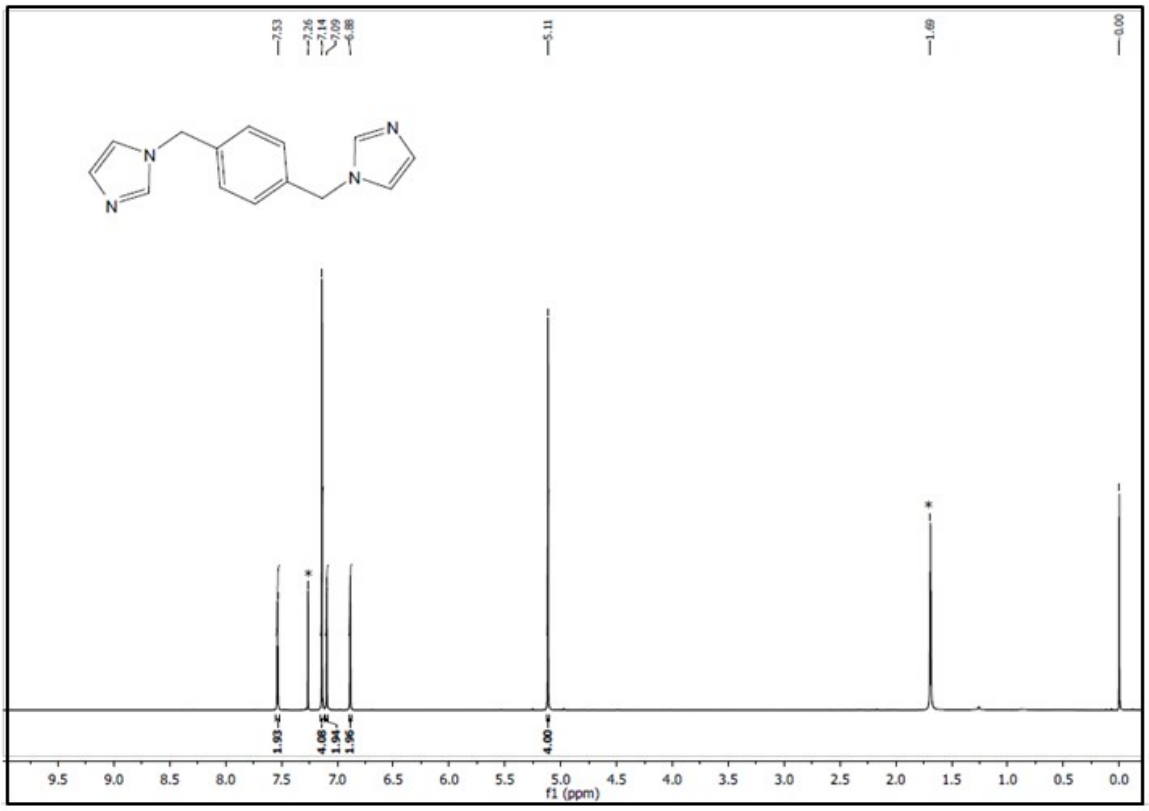


Figure S1. ^{1}H NMR spectrum of **BIMB** in $CDCl_3$ at RT.

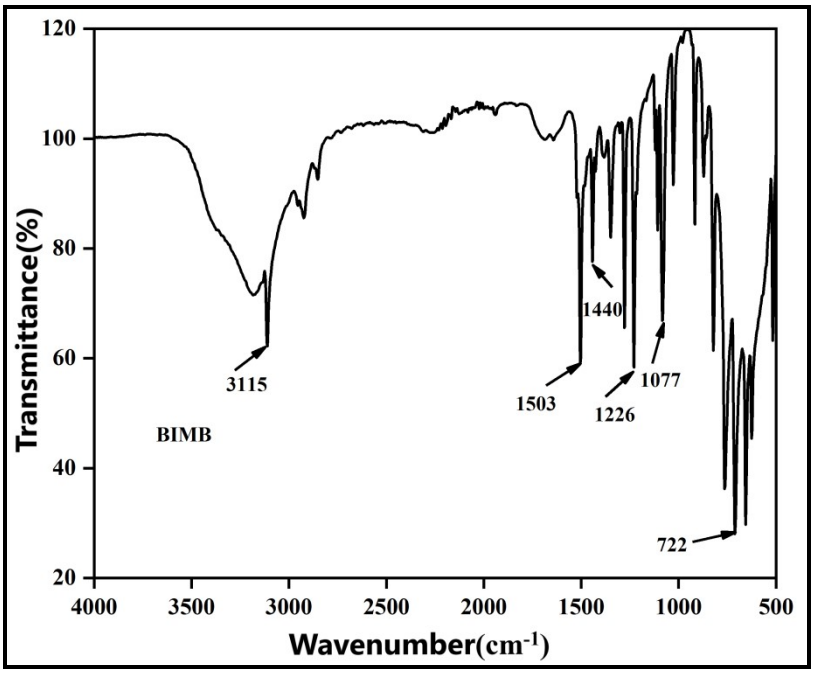


Figure S2.FT-IR spectra of **BIMB**

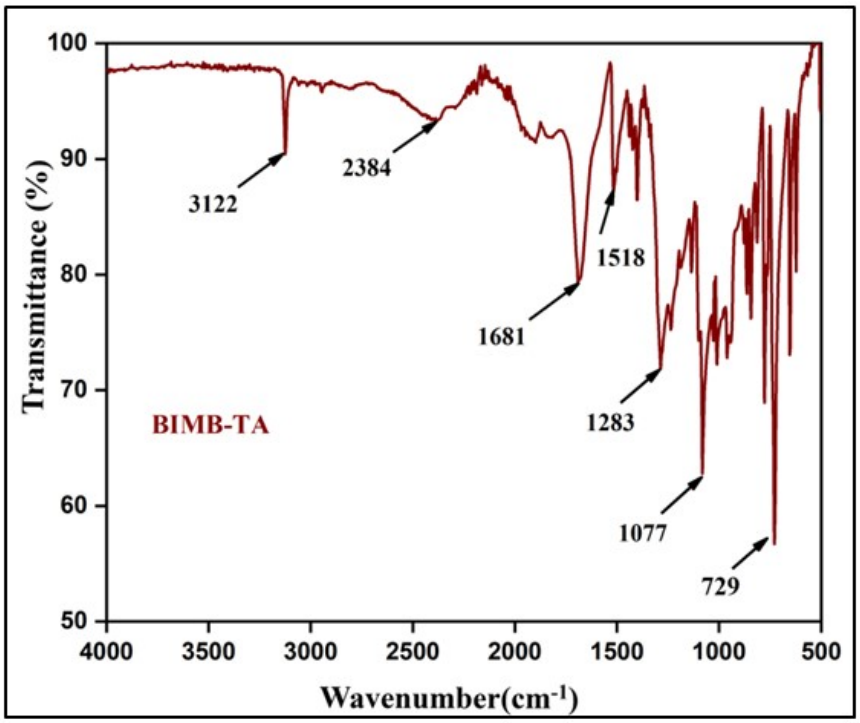


Figure S3.FT-IR spectra of **BIMB-TA** co-crystal

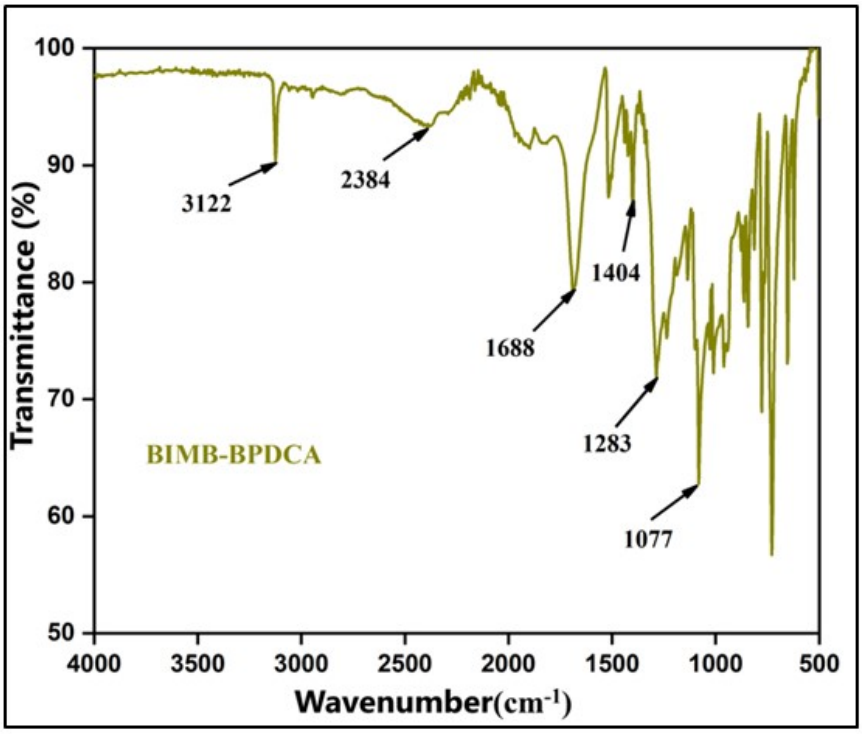


Figure S4.FT-IR spectra of **BIMB-BPDCA** co-crystal

2.Differential Scanning Calorimetry

Differential scanning calorimetric measurements of the co-crystals **BIMB-TA** and **BIMB-BPDCA** has been carried using Mettler-Toledo DSC1 instrument.Approximately 3.5 mg of pure crystalline powder was sealed in aluminium pan and covered with a pierced lid. Samples were heated at the rate of 5°C/min under the flow of N_2 gas with the rate of 20 ml/min from 25 °C to 280°C in case of **BIMB-TA** and 25°C to 300°C for **BIMB-BPDCA**.

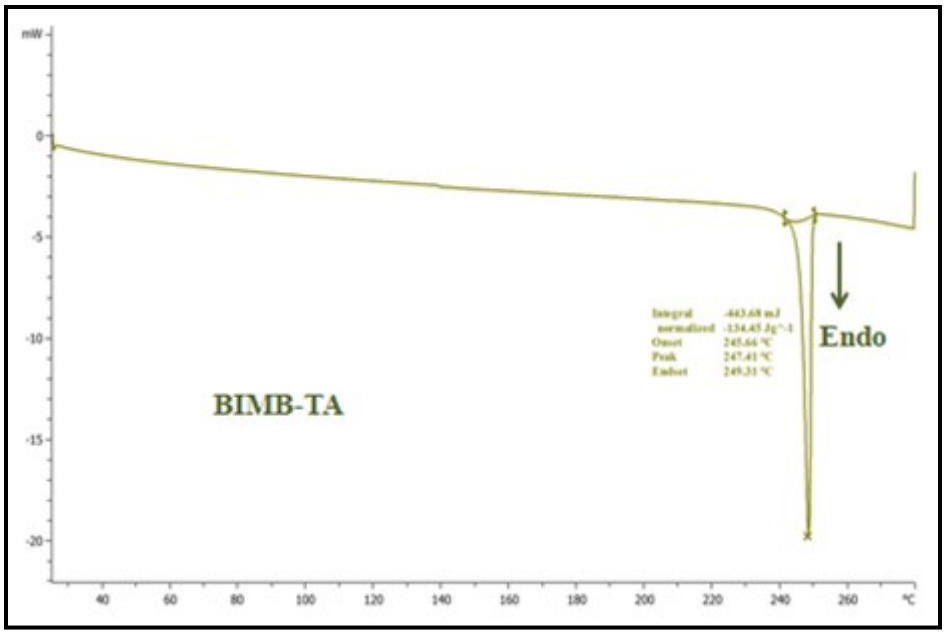


Figure S5. DSC thermogram shows melting of **BIMB-TA** at 247.41°C with an onset temperature of 245.66°C.

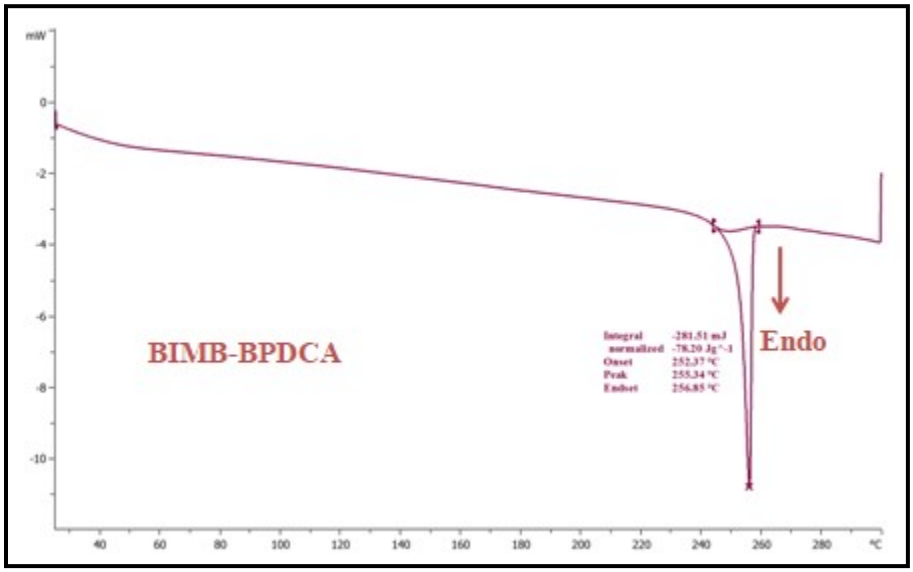
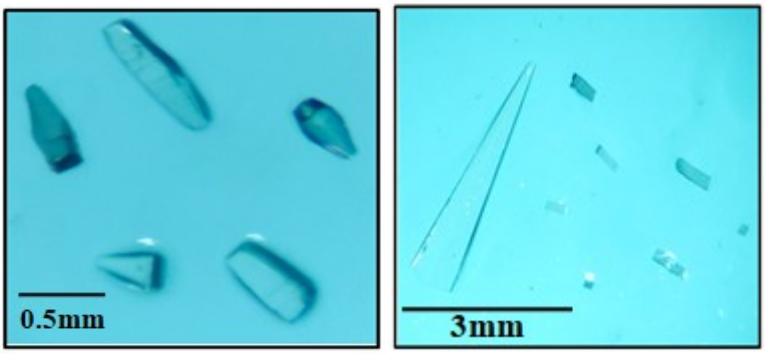


Figure S6. DSC thermogram shows melting of **BIMB-BPDCA** at 255.34°C with an onset temperature of 252.37°C.

3. Photographs of Single crystals



(a) **(b)**

Figure S7. Photograph of single crystals of (a)**BIMB-TA**, (b) **BIMB-BPDCA**

4.Variable temperature Single Crystal X-ray Diffraction.

Single-crystal X-ray data of **BIMB-TA** and **BIMB-BPDCA** were collected on Bruker D8 Quest single crystal X-ray diffractometer equipped with a microfocus anode (MoKα) and PHOTON-II detector. A suitable single crystals was mounted on a glass fibre attached with epoxy glue and mounted on a goniometer head for data collection. Variable temperature on the crystal was maintained using an oxford cryostream 800 plus cryostat. Single crystal data was recorded at every 50 K interval in the temperature range of 100K to 500K for **BIMB-TA** and 100K to 450K for **BIMB-BPDCA** co-crystal. Data integration and scaling was done using Bruker suite program.[2] Structures were solved by direct methods using SHELXT-2014/5[3] and refined by full-matrix least-squares on F^2 using SHELXL-2018/3.[4] Acidic protons (COOH) in the molecules of each co-crystals were assigned using difference Fourier map and rest of the hydrogen atoms were fixed in the riding model. All the non-hydrogen atoms were refined anisotropically. Crystallographic data and structure refinement parameters for each co-crystal is given in table below:

Table S1. Crystallographic details of **BIMB-TA**

BIMB-TA	**100K**	**150K**	**200K**	**250K**	**300K**
Moiety formula	$C_{22}H_{20}N_4O_4$	$C_{22}H_{20}N_4O_4$	$C_{22}H_{20}N_4O_4$	$C_{22}H_{20}N_4O_4$	$C_{22}H_{20}N_4O_4$
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	*C2/c*	*C2/c*	*C2/c*	*C2/c*	*C2/c*
a/Å	28.293(3)	28.341(3)	28.394(3)	28.446(3)	28.507(4)
b/Å	6.7462(8)	6.7394(8)	6.7333(8)	6.7253(8)	6.7177(8)
c/Å	11.0709(12)	11.0937(12)	11.1211(12)	11.1474(12)	11.1775(13)
α/(°)	90	90	90	90	90
β/(°)	106.737(4)	106.681(4)	106.624(4)	106.568(4)	106.525(4)
γ/(°)	90	90	90	90	90
V/Å^3	2023.6(4)	2029.8(4)	2037.3(4)	2044.1(4)	2052.1(4)
Z	4	4	4	4	4
D_{cal}/g cm^{-3}	1.327	1.323	1.319	1.314	1.309
T/K	100(2)	150(2)	200(2)	250(2)	300(2)
μ/mm^{-1}	0.094	0.093	0.093	0.093	0.092
F_{000}	848	848	848	848	848
*Crystal size/mm^3	0.480×0.190×0.180	0.480×0.190×0.180	0.480×0.190×0.180	0.480×0.190×0.180	0.480×0.190×0.180
Reflections measured	22052	22230	22382	22460	22584
Unique reflections	2513	2515	2530	2536	2546
Observed reflections	2298	2255	2231	2195	2150
Parameters	152	140	140	140	140
R_{int}	0.0336	0.0295	0.0269	0.0261	0.0263
final *R* (I >2σ(I))	0.0387	0.0393	0.0398	0.0415	0.0424
final *R* (all data)	0.0419	0.0436	0.0454	0.0479	0.0506

GOF on F^2	1.025	1.038	1.033	1.043	1.068
CCDC no	2190757	2190759	2190758	2190763	2190760

BIMB-TA	**350K**	**400K**	**450K**	**500K**
Moiety formula	$C_{22}H_{20}N_4O_4$	$C_{22}H_{20}N_4O_4$	$C_{22}H_{20}N_4O_4$	$C_{22}H_{20}N_4O_4$
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	*C2/c*	*C2/c*	*C2/c*	*C2/c*
a/Å	28.556(3)	28.607(4)	28.670(5)	28.740(5)
b/Å	6.7097(8)	6.7015(8)	6.6867(11)	6.6810(12)
c/Å	11.2036(13)	11.2320(13)	11.2601(18)	11.2925(19)
α/(°)	90	90	90	90
β/(°)	106.450(4)	106.380(4)	106.366(6)	106.308(6)
γ/(°)	90	90	90	90
V/Å^3	2058.8(4)	2065.9(4)	2071.2(6)	2081.0(6)
Z	4	4	4	4
D_{cal}/g cm^{-3}	1.305	1.300	1.297	1.291
T/K	350(2)	400(2)	450(2)	500(2)
μ/mm^{-1}	0.092	0.092	0.092	0.091
F_{000}	848	848	848	848
*Crystal size/mm^3	0.480×0.190×0.180	0.480×0.190×0.180	0.480×0.190×0.180	0.480×0.190×0.180
Reflections measured	22672	45366	22881	22992
Unique reflections	2546	2558	2579	2595
Observed reflections	2082	2124	1914	1799
Parameters	152	152	152	152
R_{int}	0.0285	0.0319	0.0356	0.0409
final *R* (I >2σ(I))	0.0432	0.0475	0.0453	0.0470
final *R* (all data)	0.0539	0.0584	0.0637	0.0704
GOF on F^2	1.035	1.077	1.037	1.034
CCDC no	2190765	2190764	2190762	2190761

*__Note:__ Crystal size has been measured at the 100K data.

Table S2. Crystallographic details of **BIMB-BPDCA**

BIMB-BPDCA	**100K**	**150K**	**200K**	**250K**	**300K**
Moiety formula	$C_{28}H_{24}N_4O_4$	$C_{28}H_{24}N_4O_4$	$C_{28}H_{24}N_4O_4$	$C_{28}H_{24}N_4O_4$	$C_{28}H_{24}N_4O_4$
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/n$	$P2_1/n$	$P2_1/n$	$P2_1/n$
a/Å	6.6714(7)	6.6622(7)	6.6480(7)	6.6319(8)	6.6119(17)
b/Å	18.245(2)	18.301(2)	18.373(2)	18.452(2)	18.529(5)
c/Å	10.1498(13)	10.1785(12)	10.2097(13)	10.2411(14)	10.282(3)
α/(°)	90	90	90	90	90
β/(°)	108.052(4)	107.716(4)	107.337(4)	106.930(5)	106.490(10)
γ/(°)	90	90	90	90	90
V/Å^3	1174.6(2)	1182.1(2)	1190.4(2)	1198.9(3)	1207.9(6)
Z	2	2	2	2	2
D_{cal}/g cm^{-3}	1.359	1.350	1.341	1.331	1.321
T/K	100(2)	150(2)	200(2)	250(2)	300(2)
μ/mm^{-1}	0.093	0.092	0.092	0.091	0.090
F_{000}	504	504	504	504	504
*Crystal size/mm^3	0.370×0.210×0.050	0.370×0.210×0.050	0.370×0.210×0.050	0.370×0.210×0.050	0.370×0.210×0.050
Reflections measured	17897	18342	18547	18674	18840
Unique reflections	2919	2941	2956	2972	3015
Observed reflections	2592	2562	2492	2400	2276
Parameters	167	179	175	179	179
R_{int}	0.0399	0.0274	0.0271	0.0439	0.0454
final R (I >2σ(I))	0.0560	0.0433	0.0447	0.0475	0.0504
final R (all data)	0.0626	0.0505	0.0540	0.0596	0.0688

GOF on F^2	1.040	1.031	1.065	1.029	1.047
CCDC no	2190766	2190771	2190767	2190768	2190770

BIMB-BPDCA	**350K**	**400K**	**450K**
Moiety formula	$C_{28}H_{24}N_4O_4$	$C_{28}H_{24}N_4O_4$	$C_{28}H_{24}N_4O_4$
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/n$	$P2_1/n$
a/Å	6.5953(9)	6.5718(9)	6.5537(9)
b/Å	18.631(3)	18.708(3)	18.802(3)
c/Å	10.3065(17)	10.3308(17)	10.3681(18)
α/(°)	90	90	90
β/(°)	105.967(5)	105.470(5)	104.937(6)
γ/(°)	90	90	90
V/Å^3	1217.6(3)	1224.1(3)	1234.4(3)
Z	2	2	2
D_{cal}/g cm^{-3}	1.311	1.304	1.293
T/K	350(2)	400(2)	450(2)
μ/mm^{-1}	0.090	0.089	0.088
F_{000}	504	504	504
*Crystal size/mm^3	0.370×0.210×0.050	0.370×0.210×0.050	0.370×0.210×0.050
Reflections measured	16960	19382	19694
Unique reflections	2529	3039	3067
Observed reflections	1977	2000	1825
Parameters	167	179	179
R_{int}	0.0435	0.0487	0.0519
final R (I >2σ(I))	0.0503	0.0567	0.0606
final R (all data)	0.0651	0.0890	0.1036
GOF on F^2	1.048	1.026	1.033
CCDC no	2190772	2190769	2190773

*__Note:__ Crystal size has been measured at the 100K data.

Table S3. Change of Unit cell parameters of **BIMB-TA** with change of temperature

Temperature (K)	*a* (Å)	*b* (Å)	*c* (Å)	*α*(°)	*β*(°)	*γ*(°)	Vol.(Å^3)
100	28.293(3)	6.7462(8)	11.0709(12)	90	106.737(4)	90	2023.6(4)
150	28.341(3)	6.7394(8)	11.0937(12)	90	106.681(4)	90	2029.8(4)
200	28.394(3)	6.7333(8)	11.1211(12)	90	106.624(4)	90	2037.3(4)
250	28.446(3)	6.7253(8)	11.1474(12)	90	106.568(4)	90	2044.1(4)
300	28.507(4)	6.7177(8)	11.1775(13)	90	106.525(4)	90	2052.1(4)
350	28.556(3)	6.7097(8)	11.2036(13)	90	106.450(4)	90	2058.8(4)
400	28.607(3)	6.7015(8)	11.2320(13)	90	106.380(4)	90	2065.9(4)
450	28.670(5)	6.6867(11)	11.2601(18)	90	106.366(6)	90	2071.1(6)
500	28.740(5)	6.6810(12)	11.2925(19)	90	106.308(6)	90	2081.0(6)

Note: NTE of *b* axis from 100K to 500K highlighted by light orange color

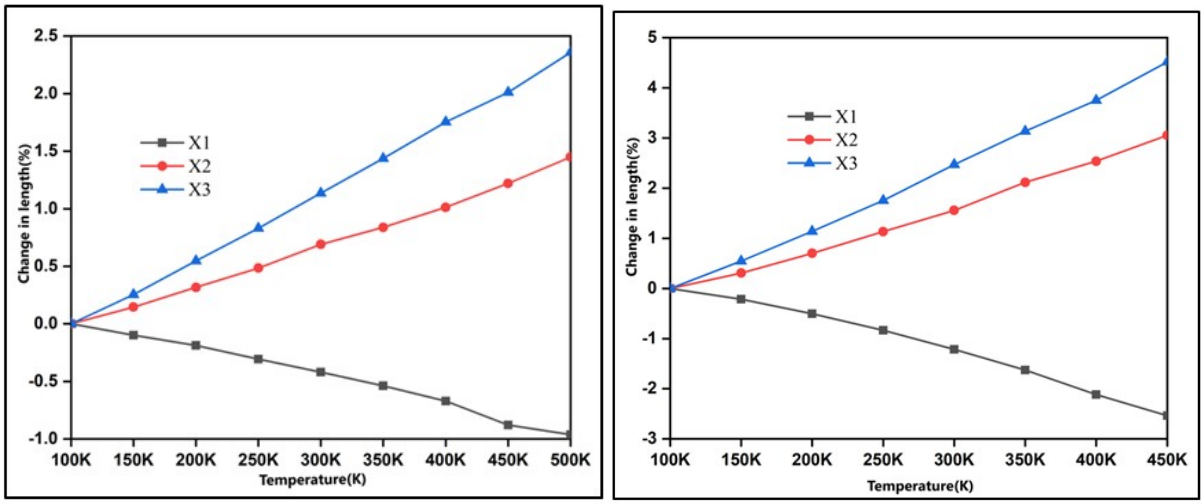
Table S4. Change of Unit cell parameters of **BIMB-BPDCA** with change of temperature

Temperature (K)	*a* (Å)	*b* (Å)	*c* (Å)	*α*(°)	*β*(°)	*γ*(°)	Vol.(Å^3)
100	6.6714(7)	18.245(2)	10.1498(13)	90	108.052(4)	90	1174.6(2)
150	6.6622(7)	18.301(2)	10.1785(12)	90	107.716(4)	90	1182.1(2)
200	6.6480(7)	18.373(2)	10.2097(13)	90	107.337(4)	90	1190.4(2)
250	6.6319(8)	18.452(2)	10.2411(14)	90	106.930(5)	90	1198.9(3)
300	6.6119(17)	18.529(5)	10.282(3)	90	106.490(10)	90	1207.9(6)
350	6.5953(9)	18.631(3)	10.3065(17)	90	105.967(5)	90	1217.6(3)
400	6.5718(9)	18.708(3)	10.3308(17)	90	105.470(5)	90	1224.1(3)
450	6.5537(9)	18.802(3)	10.3681(18)	90	104.937(6)	90	1234.4(3)

Note: NTE of *a* axis from 100K to 450K highlighted by light green color

Table S5. Calculation of thermal expansion coefficients of **BIMB-TA** and **BIMB-BPDCA** and by PASCal program.[5]

Cocrystals				Directions		
	Axes	α(MK^{-1})	σα (MK^{-1})	a	b	c
BIMB-TA	X_1	-24.6114	0.9115	-0.0000	1.0000	-0.0000
	X_2	35.7840	0.8289	-0.6268	0.0000	0.7791
	X_3	59.0444	0.6220	0.2569	-0.0000	0.9664
	V	70.2946	1.1427			
BIMB-BPDCA	X_1	-73.8290	3.4588	0.9730	-0.0000	-0.2309
	X_2	88.5435	2.4994	0.0000	1.0000	0.0000
	X_3	129.3825	2.2908	0.6767	-0.0000	0.7363
	V	145.8850	1.9874			



(a) **(b)**

Figure S8(a) Percent change in length of the principal axes with temperature in (a) **BIMB-TA** and (b) **BIMB-BPDCA**

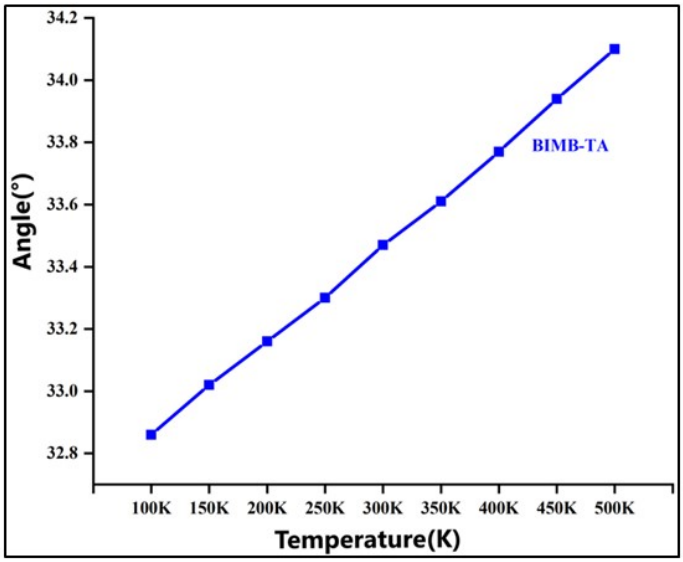
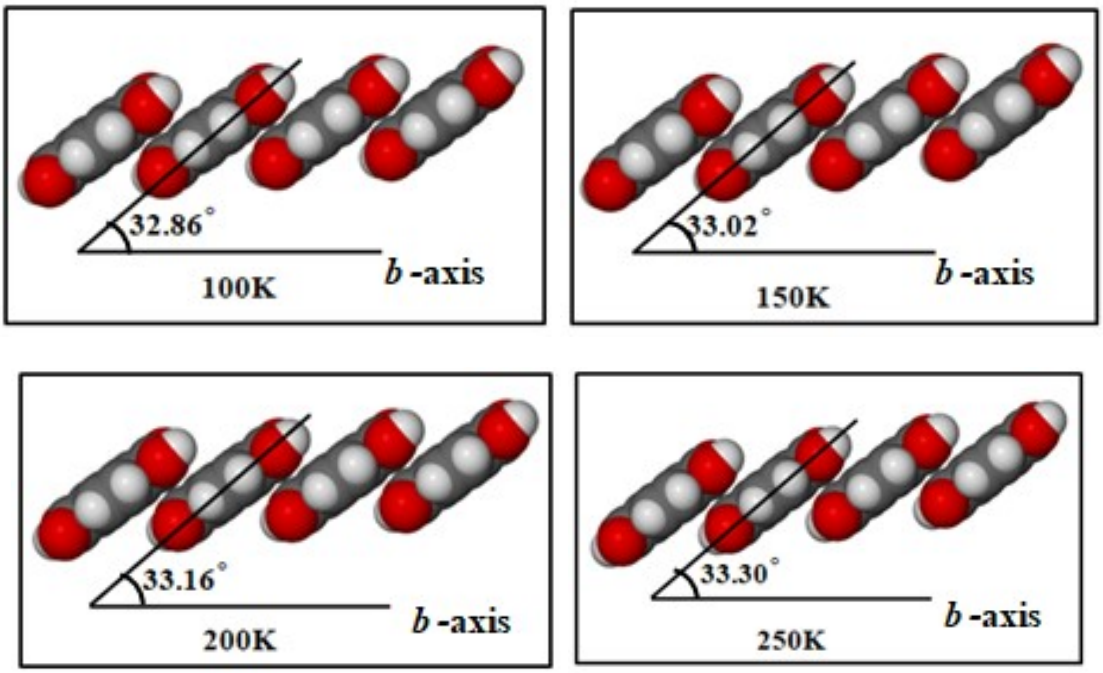


Figure S9. Change of tilt angle of **TA** molecules along *b* axis in the crystal structure of **BIMB-TA** with increasing temperature.

Tilt Angle of TA molecules in the crystal structure of BIMB-TA at different temperature



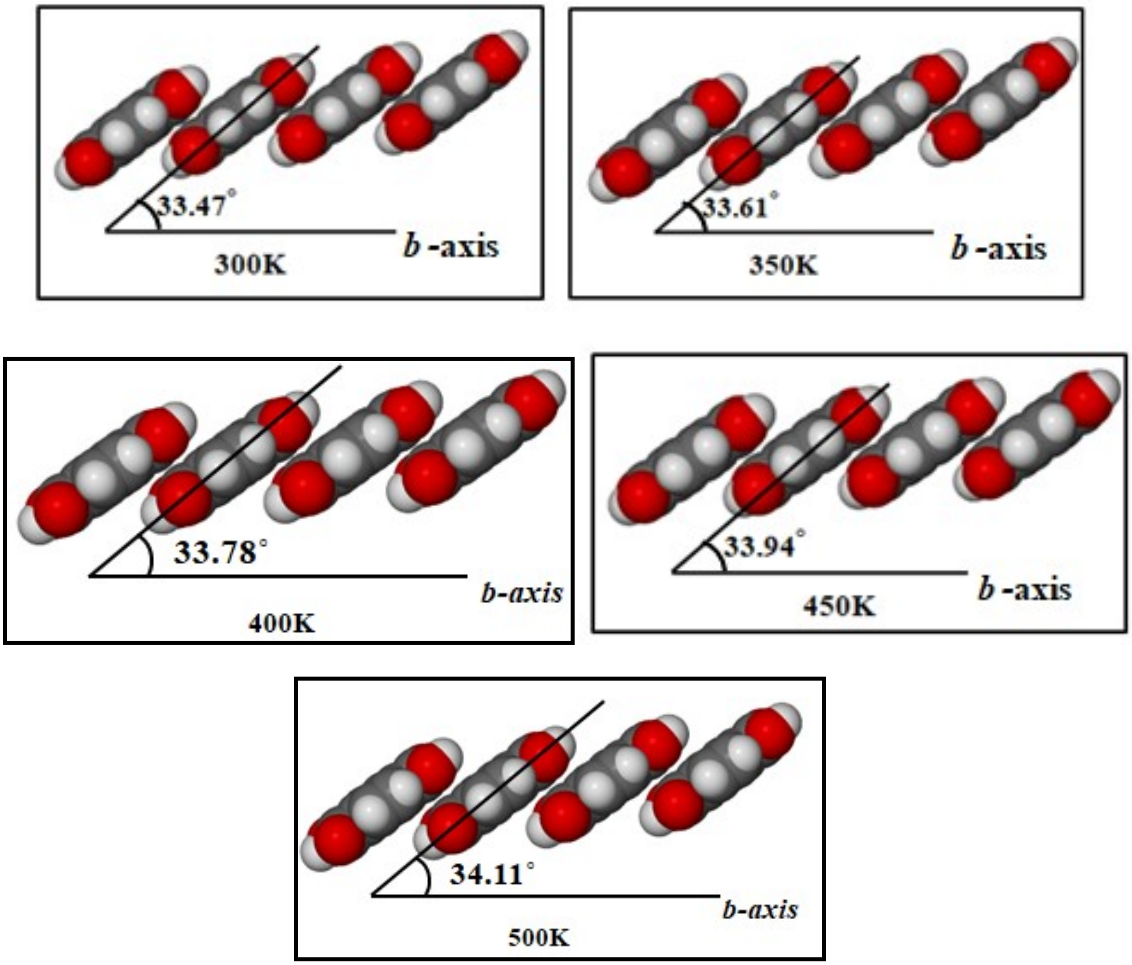


Figure S10. Tiltation of **TA** molecules along *b* axis in the crystal structure of **BIMB-TA** at different temperature.

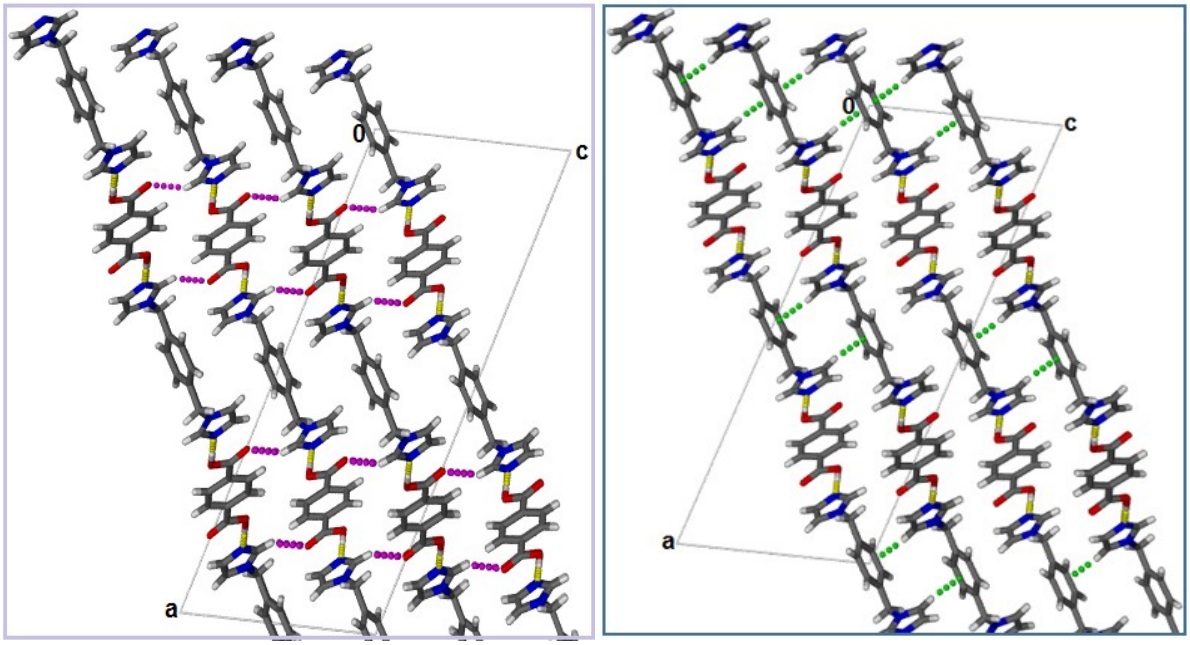


Figure S11. Packing of molecules in the crystal structure of **BIMB-TA**.(a) C−H···O(magenta)(b) C−H···π interactions are shown in green dotted lines.

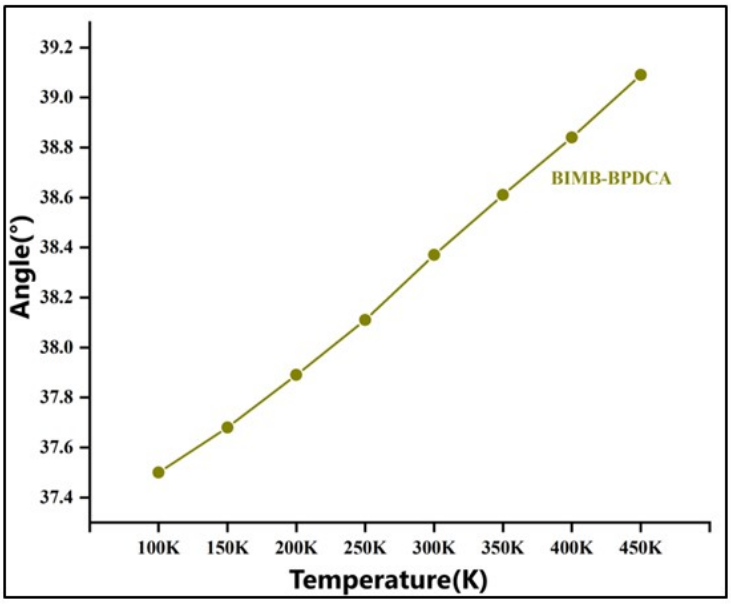


Figure S12. Change of tilt angle of **BPDCA** molecules in the crystal structure of **BIMB-BPDCA** determined at different temperature

Tilt angle of BPDCA molecules in the crystal structure of BIMB-BPDCA determined at different temperature BIMB-BPDCA

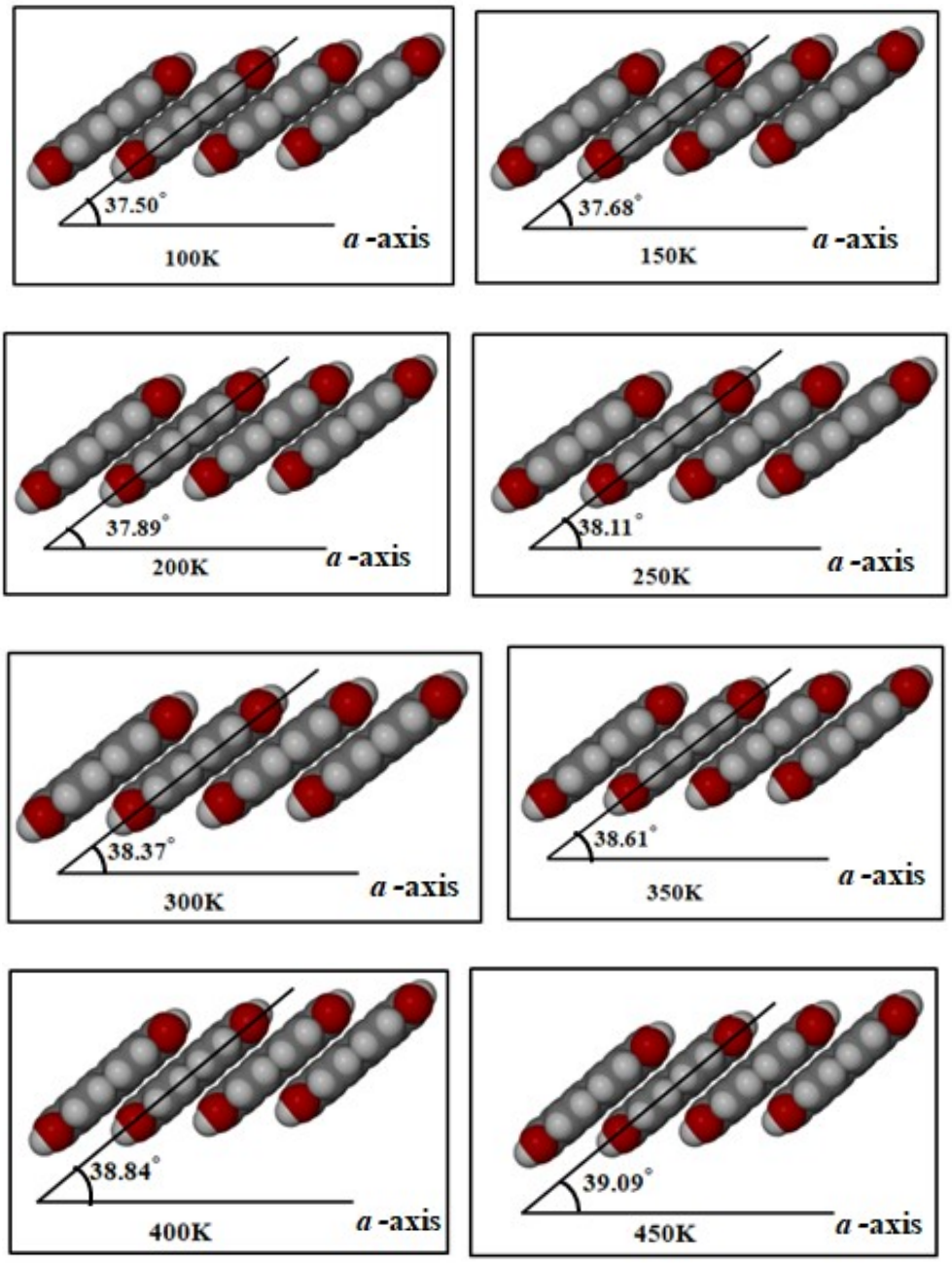


Figure S13. Tiltation of **BPDCA** molecule along *a-axis* in the crystal structure of **BIMB-BPDCA** at different temperature.

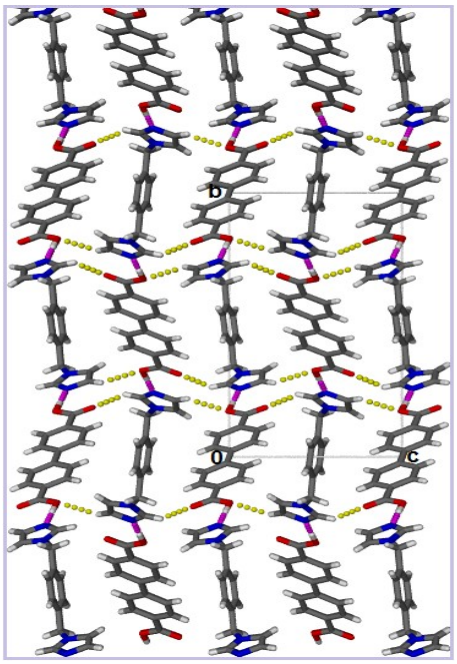


Figure S14. Intermolecular interactions in the crystal structure of **BIMB-BPDCA** viewed down *a* axis: C−H···O hydrogen bonding interactions shown in yellow colour.

Table S6. Hydrogen bonding parameters in the crystal structures of **BIMB-TA** determined at different temperature[6].

Donor– H···Acceptor	T(K)	D – H (Å)	H···A (Å)	D···A (Å)	∠D – H···A (°)
O(1) – H(1A) ···N(1)	**100K**	0.98(2)	1.61(2)	2.5914(14)	179(2)
	150K	0.96(2)	1.63(2)	2.5942(14)	179(2)
	200K	0.98(2)	1.62(2)	2.5971(15)	178(2)
	250K	0.98(2)	1.62(2)	2.6013(16)	179(3)
	300K	0.97(2)	1.64(2)	2.6053(16)	178(2)
	350K	0.98(3)	1.63(3)	2.6088(18)	178(2)
	400K	0.98(3)	1.63(3)	2.6120(18)	177(2)
	450K	0.96(3)	1.65(3)	2.6142(19)	178(3)

	500K	0.96(3)	1.66(3)	2.620(2)	179(3)
C(5) – H(5) ···O(2)		**C(5) – H(5)**	**H(5) ···O(2)**	**C(5) ···O(2)**	**C(5) -H(5)···O(2)**
	100K	0.95	2.32	3.1293(14)	143
	150K	0.95	2.33	3.1368(14)	143
	200K	0.95	2.33	3.1452(16)	143
	250K	0.94	2.35	3.1537(16)	143
	300K	0.93	2.37	3.1633(18)	143
	350K	0.93	2.37	3.1714(18)	144
	400K	0.93	2.38	3.1810(19)	144
	450K	0.93	2.39	3.191(2)	144
	500K	0.93	2.40	3.200(2)	144
C(7)−H(7)···π		**C(7)−H(7)**	**H(7)···π**	**C(7)···π**	**∠C(7)−H(7)···π**
	100K	0.97(2)	2.809(18)	3.7577(15)	166.7(14)
	150K	0.950	2.83	3.7677(16)	171
	200K	0.950	2.84	3.7789(16)	170
	250K	0.940	2.86	3.7923(19)	170
	300K	0.930	2.89	3.807(2)	170
	350K	0.95(2)	2.91(2)	3.818(2)	162.2(16)
	400K	0.94(3)	2.93(2)	3.831(2)	161.1(18)
	450K	0.92(3)	2.97(2)	3.846(2)	160.1(18)
	500K	0.92(3)	2.99(2)	3.861(3)	159.3(18)

Table S7. Hydrogen bonding parameters in the crystal structures of **BIMB-BPDCA** determined at different temperature.

Donor – H···Acceptor	**T(K)**	**D – H (Å)**	**H···A (Å)**	**D···A (Å)**	**∠D – H···A (°)**
O(1)– H(1A)···N(1)	**100K**	0.95(2)	1.60(2)	2.545(2)	176(2)
	150K	0.950(16)	1.600(17)	2.5496(15)	177.9(18)
	200K	0.949(16)	1.609(17)	2.5566(17)	178(2)
	250K	0.950(16)	1.613(17)	2.5625(18)	179(2)
	300K	0.951(19)	1.618(19)	2.569(2)	178(3)
	350K	0.952(19)	1.630(19)	2.579(2)	174(2)
	400K	0.95(2)	1.63(2)	2.582(2)	176(3)
	450K	0.95(2)	1.64(2)	2.587(3)	176(3)
C(1)– H(1)···O(2)		**C(1)– H(1)**	**H(1)···O2**	**C(1)···O(2)**	**∠C(1)– H(1)···O(2)**
	100K	0.95	2.32	3.069(2)	135
	150K	0.965(16)	2.281(16)	3.0732(17)	138.7(12)
	200K	0.95	2.32	3.0817(18)	137
	250K	0.953(18)	2.304(18)	3.0895(19)	139.3(14)
	300K	0.959(19)	2.303(19)	3.100(2)	140.1(14)
	350K	0.93	2.34	3.105(2)	140
	400K	0.99(2)	2.28(2)	3.112(3)	141.7(16)
	450K	1.00(3)	2.27(3)	3.121(3)	142.5(18)
C(2)– H(2)···O(1)		**C(2)– H(2)**	**H(2)···O(1)**	**C(2)···O(1)**	**∠C(2)– H(2)···O(1)**
	100K	0.95	2.44	3.061(2)	123
	150K	0.948(19)	2.525(17)	3.0767(16)	117.3(14)
	200K	0.942(19)	2.531(18)	3.0982(17)	119.0(14)
	250K	0.95(2)	2.551(18)	3.1165(19)	118.5(14)
	300K	0.95(2)	2.55(2)	3.141(2)	120.1(15)
	350K	0.93	2.55	3.166(2)	124
	400K	0.96(2)	2.59(2)	3.185(2)	120.4(15)
	450K	0.97(3)	2.61(3)	3.214(3)	121(2)

5. Powder X-ray Diffraction

Powder X-ray diffractogram was measured on Rigaku powder X-ray diffractometer (Miniflex600 with Cu Kα radiation, λ = 1.54059 Å) operating in Bragg−Brentano geometry. Crystals of the compound was crushed gently and layered on a sample holder. Data was recorded at room temperature at a scan rate of 2°/min from 5° to 40° (2θ value).

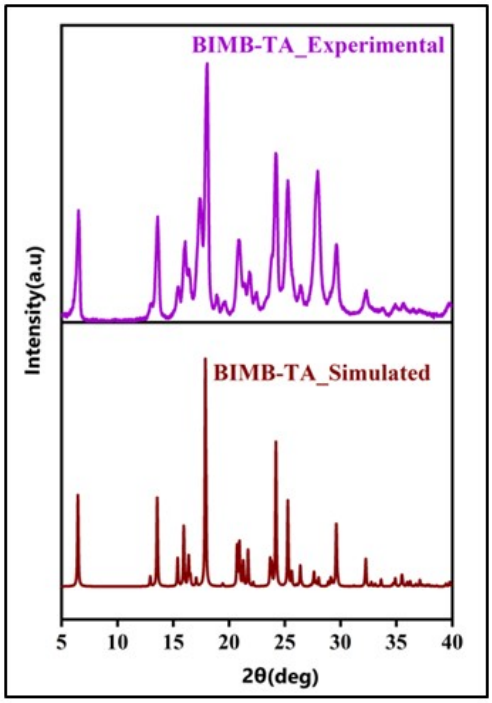


Figure S15. Powder X-ray Diffractogram of bulk sample of BIMB-TA (purple) and simulated pattern obtained from SCXRD data (red)

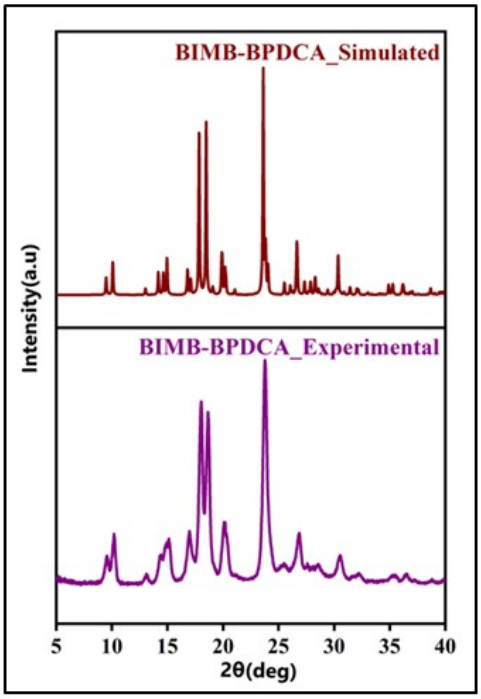
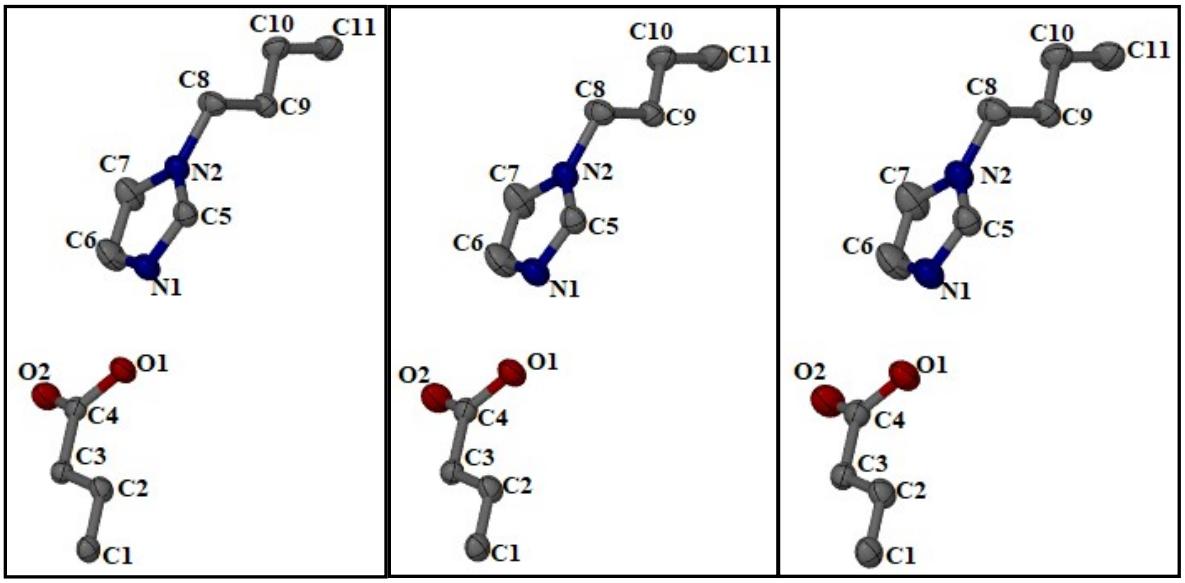
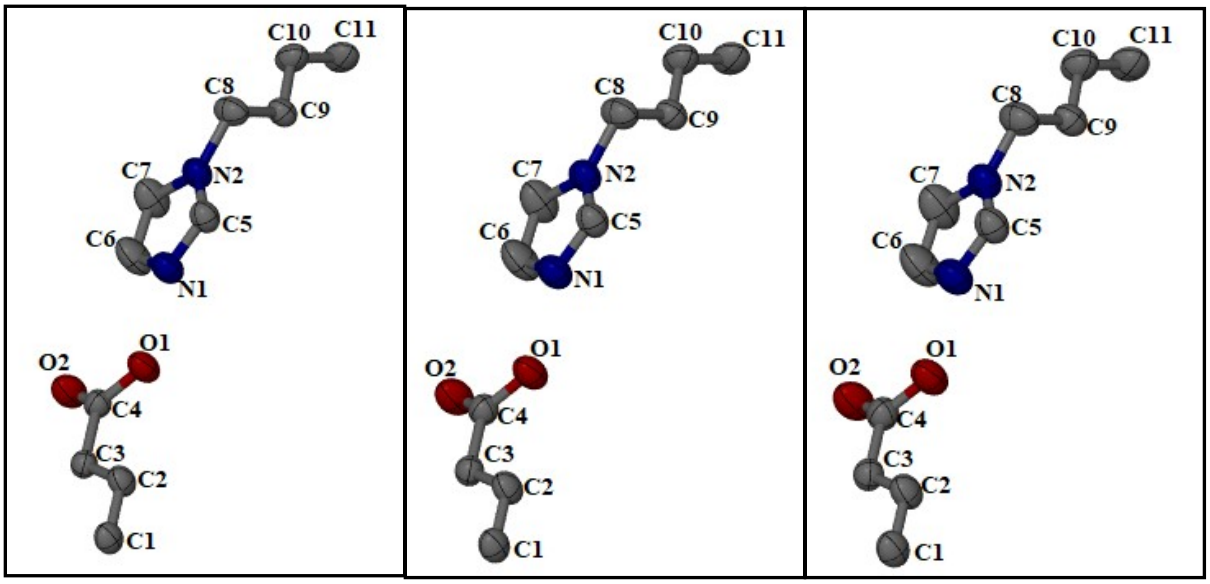


Figure S16. Powder X-ray Diffractogram of bulk sample of BIMB-BPDCA (purple) and simulated pattern obtained from SCXRD data (red)

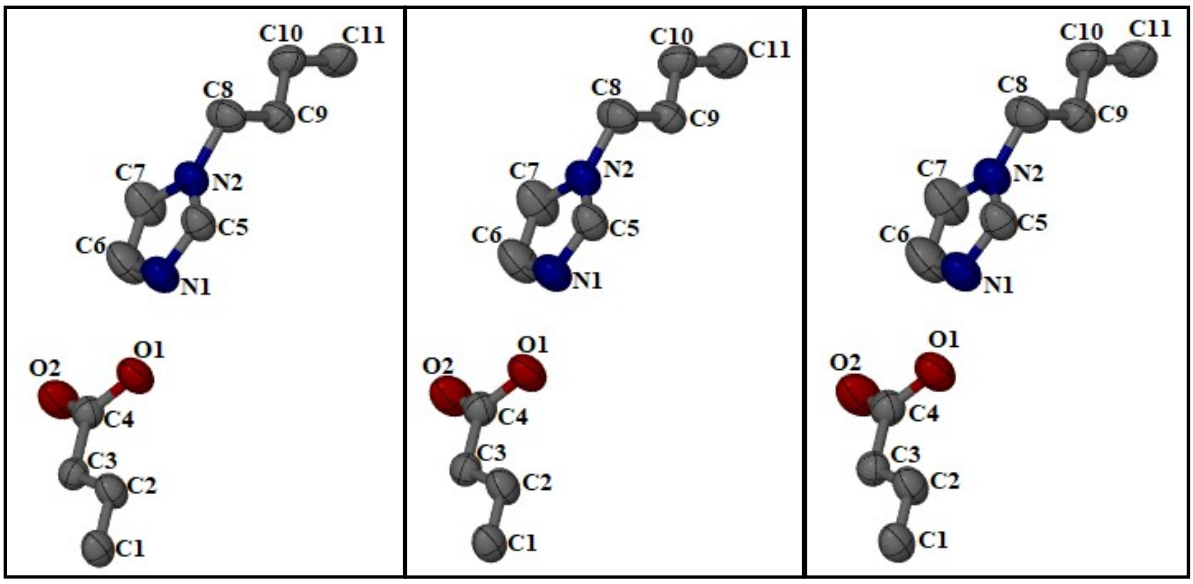
6. Thermal Ellipsoid plot of the asymmetric unit at different temperatures



100K 150K 200K

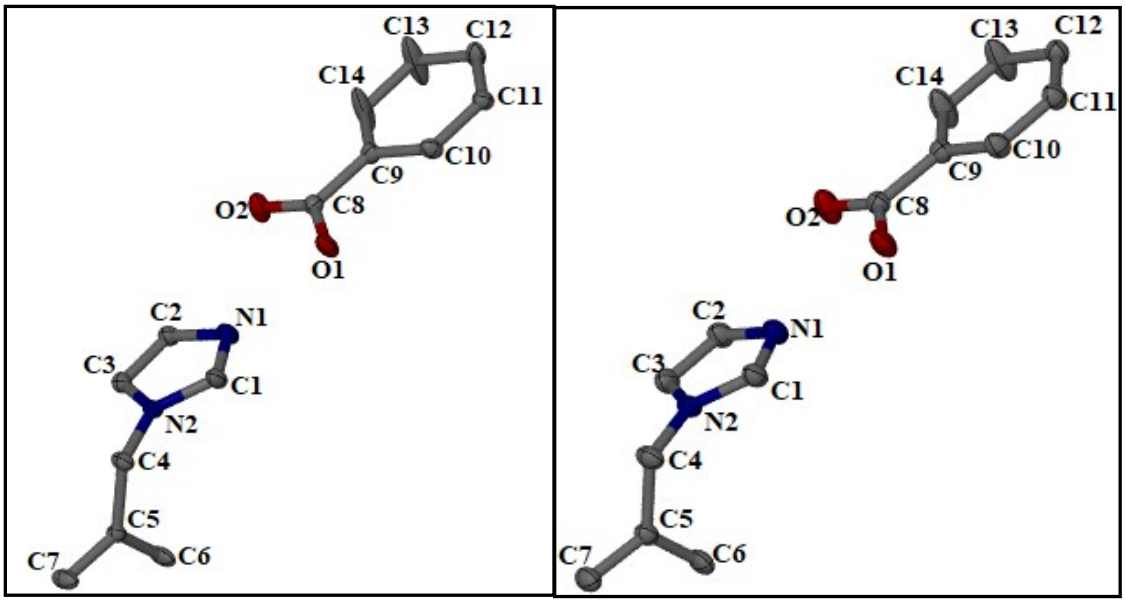


250K 300K 350K

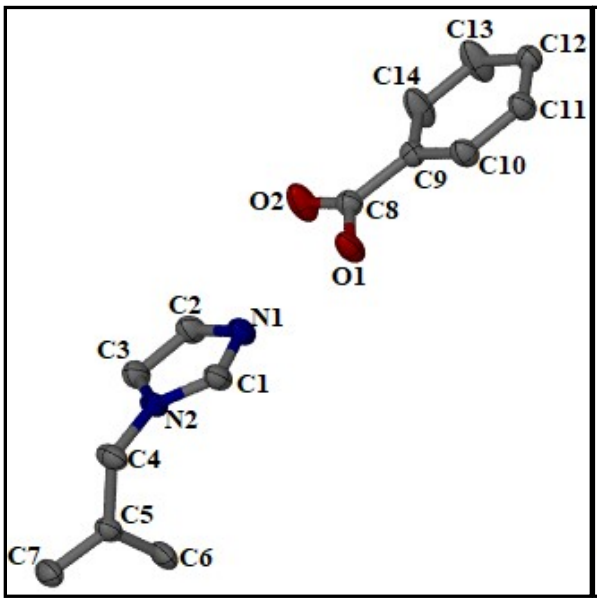


400K **450K** **500K**

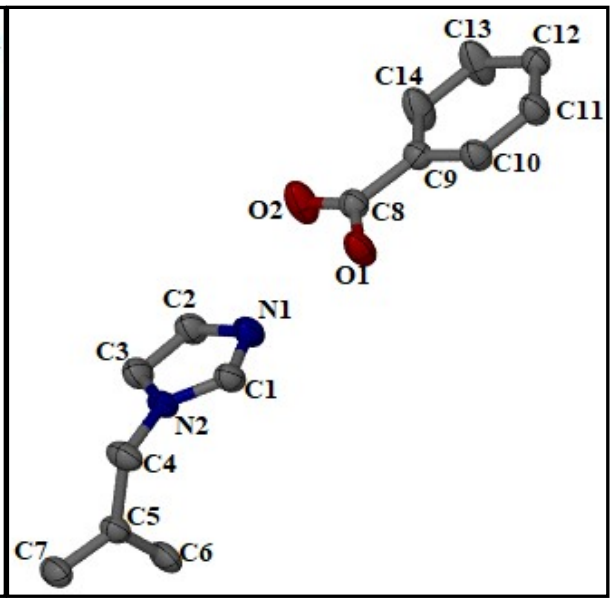
Figure S17. Thermal ellipsoid plot of the molecule in the asymmetric unit of the crystal structure of **BIMB-TA** at different temperature. Thermal ellipsoid plots are shown in 50 % probability.



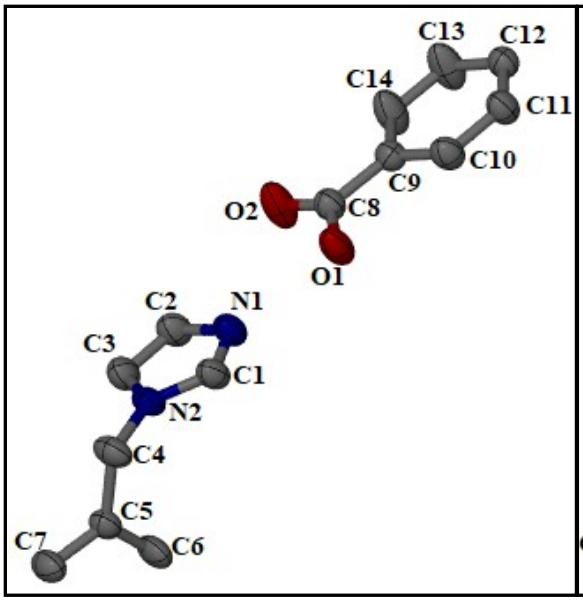
100K **150K**



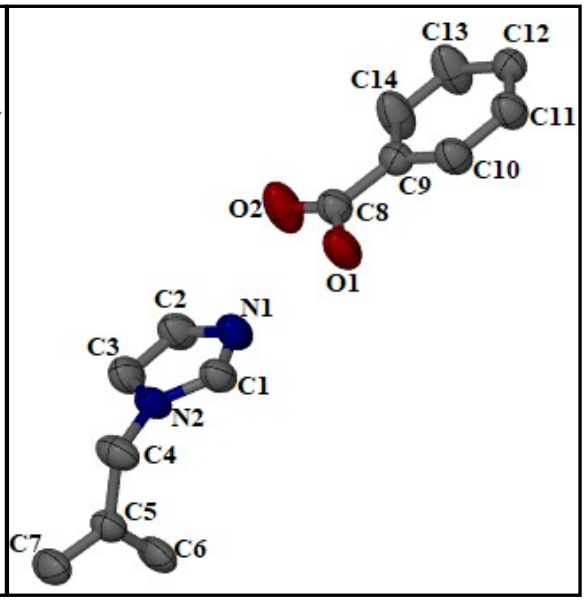
200K



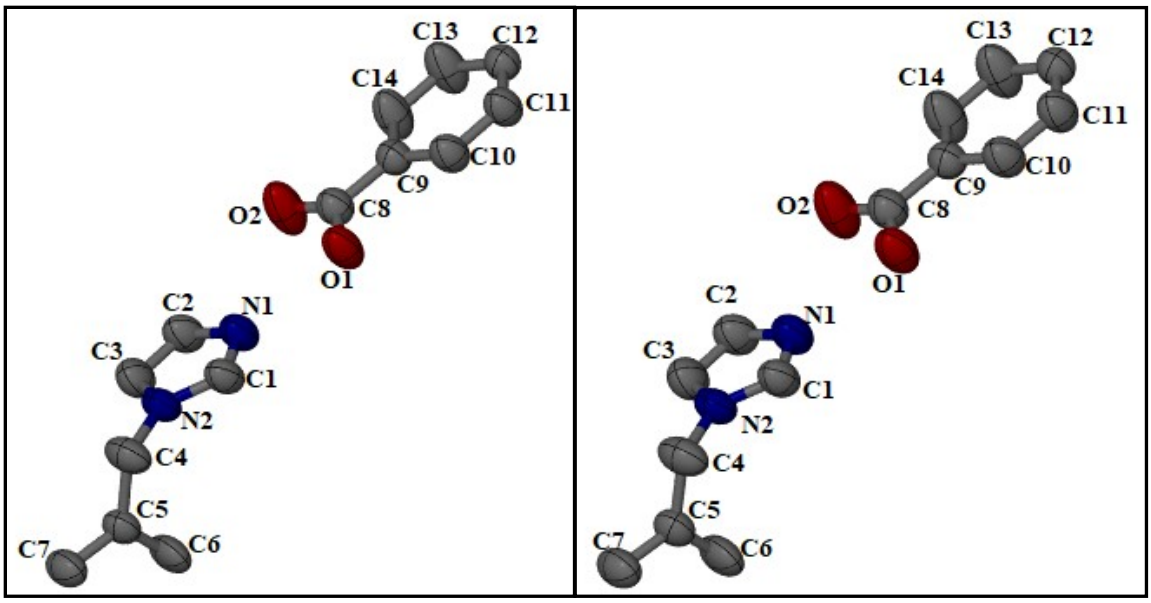
250K



300K



350K



400K **450K**

Figure S18. Thermal ellipsoid plot of the molecule in the asymmetric unit of the crystal structure of **BIMB-BPDCA** at different temperature. Thermal ellipsoid plots are shown in 50% probability.

- **References**

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