JOURNAL OF APPLIED CRYSTALLOGRAPHY

Volume 50 (2017)

Supporting information for article:

An insight into the synthesis, crystal structure, geometrical modelling of crystal morphology, Hirshfeld surface analysis and characterizations of N-(4-methylbenzyl)benzamide single crystals

Sahil Goel, Harsh Yadav, Nidhi Sinha, Budhendra Singh, Igor Bdikin, Devarapalli Chenna Rao, Kovuru Gopalaiah and Binay Kumar

Supporting information

FTIR, NMR and HRMS analytical techniques were used for the identification of functional groups and confirmation of structure of *N*-(4-methylbenzyl)benzamide compound. Void mapping for titled crystal was performed to visualize and locate the void space present in the crystal lattice and to compute volumes and surface area of void space and porosity.

S1. Functional group analysis using FTIR spectroscopy

FTIR spectroscopy is a powerful technique used for determining the chemical bonding and the functional groups present in the sample. The FTIR transmittance spectra of MBB compound plotted in the wavenumber range 4000-600 cm^{-1} are displayed in Fig. S1. Dichloromethane solution of MBB was used for recording the FTIR spectrum. In the spectrum, the peak at 3309 cm^{-1} is assigned as asymmetric and symmetric stretching modes of N-H of MBB compound. The asymmetric and symmetric stretching vibrations of C-H bond display its characteristic peak at 2920 cm^{-1}. The strong absorption peak at 1638 cm^{-1} is assigned to the stretching frequency of C=O of amide group. Another intensive band appearing at 1546 cm^{-1} corresponds to stretching vibrations of C=C of the aromatic rings. The peaks in the fingerprint region (1500-600 cm^{-1}) can be assigned to all types of vibrating and bending modes within the molecule. All observed vibrational bands confirms the formation of MBB single crystal.

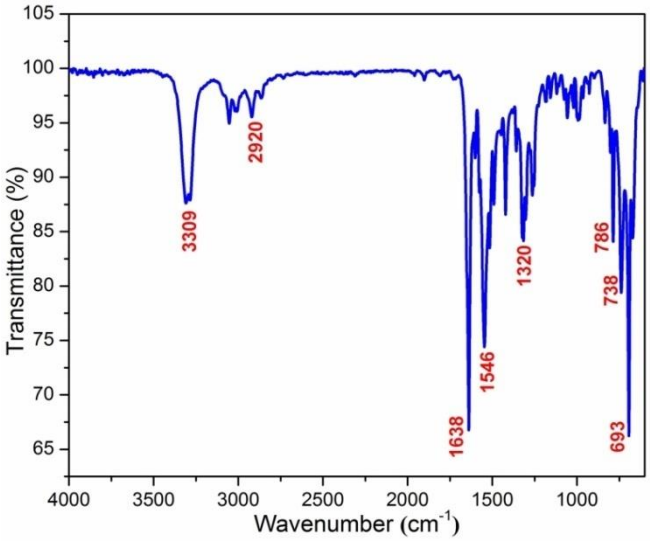

Figure S1 Unit cell packing diagram for MBB compound with 0.002 au void surface. The view is along the '*a*' crystallographic axis.

S2. NMR Spectral Studies

In ^{1}H NMR spectrum of MBB compound (Fig. S2(*a*)), the appearance of a doublet at 7.78 ppm indicates the two CH protons of aromatic ring. The appearance of multiplet at 7.52-7.48 ppm refers to the aromatic C-H. The triplet at 7.43 ppm corresponds to two protons of aromatic ring. Two multiplets appearing at 7.26-7.24 and 7.18-7.16 ppm correspond to two C-H protons of aromatic ring. The signal belonging to N-H proton is observed as a broad singlet at 6.32 ppm. The doublet at 4.61 ppm indicates the CH_2 group which is linked to N-H. The last sharp singlet at 2.35 ppm refers to the CH_3 group. In ^{13}C NMR spectrum of MBB (Fig. S2(*b*)), the peaks at 167.3 ppm and 137.3 ppm denote the C=O group and C6 carbon of aromatic ring (see Fig. 2), respectively. The appearance of signals at 135.1 ppm and 134.5 ppm belong to C9 and C12 of the titled compound. The C3 carbon of the MBB compound displays its characteristic peak at 131.5 ppm. The appearance of a set of peaks at 129.4, 128.5, 127.9, and 126.9 ppm refers to the CH carbons (C11 & C13, C10 & C14, C1 & C5, and C2 & C4) of aromatic rings. The resonance signals at 43.9 ppm and 21.1 ppm correspond to the presence of CH_2 and CH_3 carbons, respectively.

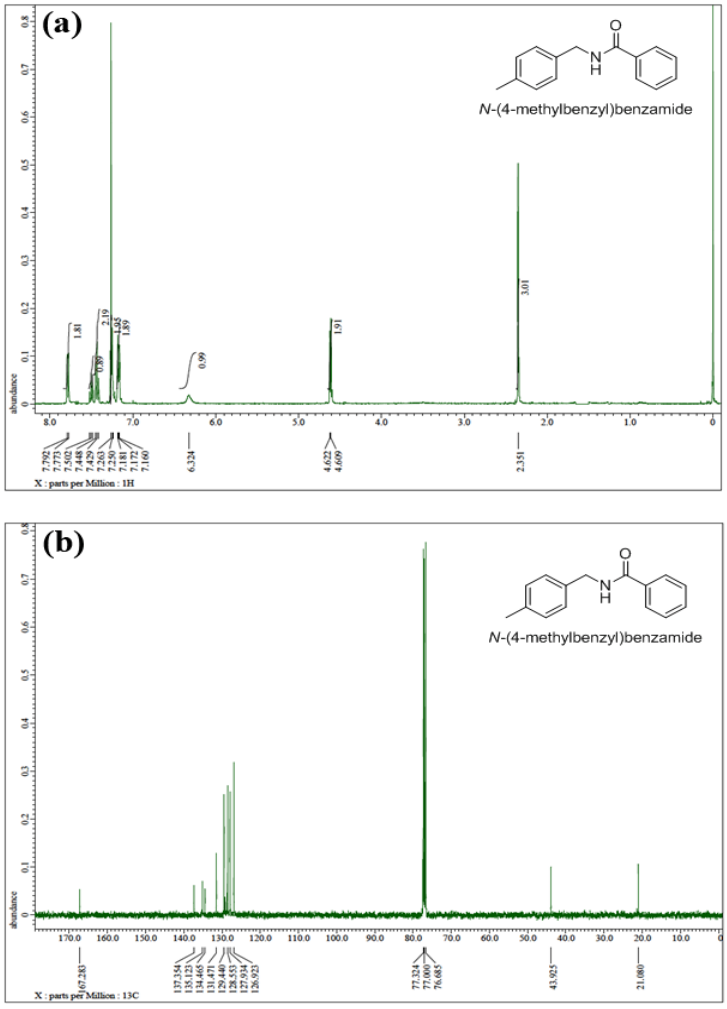

Figure S2 (*a*) ^{1}H NMR Spectrum of MBB (400 MHz, $CDCl_3$). (*b*) ^{13}C NMR of MBB (100 MHz, $CDCl_3$).

S3. HRMS

Fig. S3 displays the HRMS spectrum of the titled compound. The formation of ion at *m/z* 226.1228 of empirical formula $C_{15}H_{16}NO$ confirms the formation of *N*-(4-methylbenzyl)benzamide.

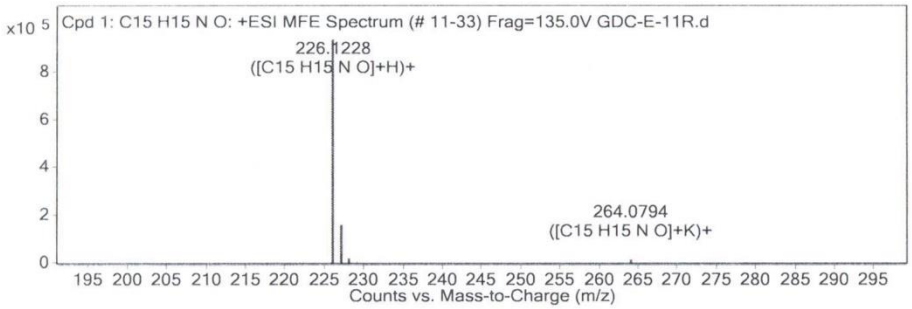

Figure S3 LC-HRMS/MS spectra of *N*-(4-methylbenzyl)benzamide crystal.

S4. Void mapping for *N*-(4-methylbenzyl)benzamide crystal

Fig. S4 displays the unit cell packing diagram of the MBB compound with voids mapped at 0.002 au isosurface along '*a*' crystallographic axis. The void channels were found to occupy 13.5 % of the unit cell volume with surface area of 547.57 $Å^2$. From void analysis, we can conclude that cavity channels are uniformly distributed in the crystal lattice, which shows the scope of binding of titled compound with metal ions to alter its physical properties.

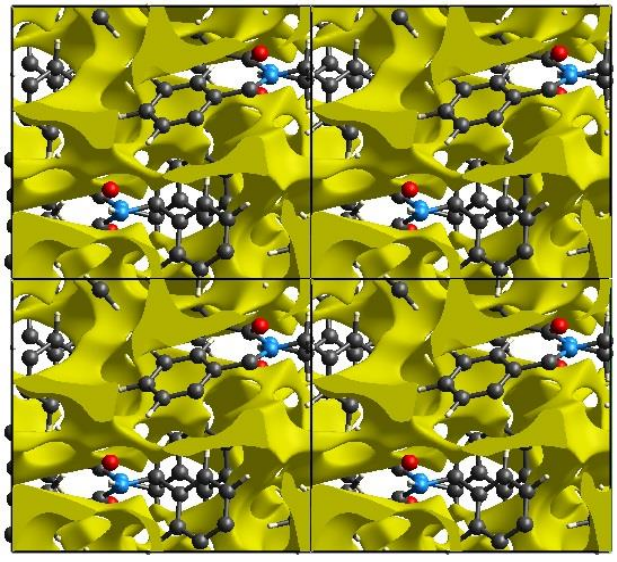

Figure S4 Unit cell packing diagram for MBB compound with 0.002 au void surface. The view is along the '*a*' crystallographic axis.