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Crystal structure of bis((1-methylbenzimidazol-2-yl)methyl)amine, $C_{18}H_{19}N_5$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.12 × 0.10 × 0.06 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker Apex II, φ and ω
$\theta_{\max}$, completeness:	26.9°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8564, 3284, 0.028
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2882
$N(\text{param})_{\text{refined}}$:	213
Programs:	Bruker [1], SHELX [2–4], Diamond [5], Olex2 [6]

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Abstract

$C_{18}H_{19}N_5$, triclinic, $P\bar{1}$ (no. 2), $a = 6.612(4)$ Å, $b = 8.800(5)$ Å, $c = 13.777(8)$ Å, $\alpha = 83.219(7)^\circ$, $\beta = 79.329(6)^\circ$, $\gamma = 78.744(6)^\circ$, $V = 769.8(8)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0394$, $wR_{\text{ref}}(F^2) = 0.0996$, $T = 100(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All the reagents and solvents were used as obtained without further purification. Bis((1-methylbenzimidazol-2-yl)methyl)

amine (MeIDB) was prepared according to a slightly modified method described by Adams *et al.* [7] in which only the raw material *o*-phenylenediamine was changed to be *N,N'*-dimethyl-1,2-phenylenediamine. Crystals of MeIDB were obtained three days later by slow evaporation in methanol (95%) at room temperature.

Experimental details

Hydrogen atoms bonded to carbon atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms with C–H = 0.95 Å (aromatic), 0.99 Å (methylene), 0.98 Å (methyl) $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ (aromatic and methylene) and $1.5U_{\text{eq}}$ (methyl). The H atom at N1 was found from the difference maps, which was refined freely and the U_{iso} value set to 1.2 times of $U_{\text{eq}}(\text{N1})$.

Comment

Multi-benzimidazole ligands have been often used in the synthesis of various metal-organic complexes or modelling the biomimetic coordination environment around the enzymatic active site [7]. For instance, by employing a tripodal bis(benzimidazole) ligand bis((1-methylbenzimidazol-2-yl)methyl)amine (MeIDB) and its derivative as the main ligand, a series of nickel and copper complexes were synthesized which allow a systematic investigation of a biomimetic

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6990 (2)	0.87744 (15)	0.38431 (9)	0.0231 (3)
H1A	0.750452	0.809345	0.329364	0.028*
H1B	0.545026	0.905601	0.390128	0.028*
C2	0.7522 (2)	0.78962 (15)	0.47868 (9)	0.0205 (3)
C3	0.9439 (2)	0.65481 (14)	0.58565 (9)	0.0209 (3)
C4	1.0994 (2)	0.57547 (15)	0.63811 (10)	0.0254 (3)
H4	1.243623	0.572320	0.612476	0.030*
C5	1.0325 (2)	0.50132 (16)	0.72976 (10)	0.0283 (3)
H5	1.133474	0.445801	0.767920	0.034*
C6	0.8193 (2)	0.50651 (15)	0.76730 (10)	0.0282 (3)
H6	0.778991	0.454756	0.830416	0.034*
C7	0.6660 (2)	0.58549 (15)	0.71432 (10)	0.0265 (3)
H7	0.521838	0.588559	0.740195	0.032*
C8	0.7301 (2)	0.66057 (14)	0.62154 (9)	0.0215 (3)
C9	1.1454 (2)	0.76232 (17)	0.42700 (10)	0.0272 (3)
H9A	1.131995	0.748151	0.359115	0.041*
H9B	1.263540	0.686814	0.446856	0.041*
H9C	1.169421	0.867948	0.430006	0.041*
C10	0.76723 (19)	1.08687 (14)	0.25882 (9)	0.0199 (3)
H10A	0.810416	1.189947	0.247988	0.024*
H10B	0.618511	1.102050	0.251113	0.024*
C11	0.89728 (19)	0.98427 (14)	0.18280 (9)	0.0179 (3)
C12	0.99944 (19)	0.82134 (14)	0.07210 (9)	0.0184 (3)
C13	1.0174 (2)	0.71265 (15)	0.00300 (9)	0.0230 (3)
H13	0.897240	0.680915	-0.011022	0.028*
C14	1.2158 (2)	0.65314 (15)	-0.04409 (10)	0.0253 (3)
H14	1.231487	0.579007	-0.091015	0.030*
C15	1.3946 (2)	0.69955 (15)	-0.02430 (10)	0.0250 (3)
H15	1.528120	0.656249	-0.058367	0.030*
C16	1.3814 (2)	0.80672 (15)	0.04355 (9)	0.0227 (3)
H16	1.501975	0.838268	0.057207	0.027*
C17	1.18051 (19)	0.86563 (14)	0.09066 (9)	0.0183 (3)
C18	1.2411 (2)	1.05296 (17)	0.20302 (11)	0.0275 (3)
H18A	1.243686	1.154931	0.166004	0.041*
H18B	1.383646	0.993132	0.197540	0.041*
H18C	1.183862	1.066871	0.272921	0.041*
N1	0.78974 (17)	1.01903 (13)	0.36043 (8)	0.0210 (2)
H1	0.713 (2)	1.0893 (19)	0.4025 (12)	0.025*
N2	0.95392 (16)	0.73860 (12)	0.49388 (8)	0.0208 (2)
N3	0.61208 (17)	0.74629 (13)	0.55304 (8)	0.0231 (2)
N4	1.11084 (16)	0.96983 (12)	0.16239 (8)	0.0190 (2)
N5	0.82334 (16)	0.89714 (12)	0.13125 (8)	0.0196 (2)

chemistry [8, 9]. However, up to now the detailed crystallization properties of MeIDB has been not reported which can be helpful for the design of various multifunctional compounds due to its central acyclic $-\text{CH}_2-\text{N}-\text{CH}_2-$ linkage moiety, making the ligand possess various steric arrangements. We have also synthesized some complexes involving tetrazole ligands, to get fluorescent materials [10, 11].

The titled compound was crystallized as one anhydrous form in the triclinic $P\bar{1}$ space group with only one MeIDB molecule in its asymmetric unit. The MeIDB molecule adopts a nearly V-shaped conformation which is reflected by the torsion angle of $-\text{C2}-\text{C1}-\text{N1}-\text{C10}-$ ($169.6(1)^\circ$), $-\text{C1}-\text{N1}-\text{C10}-\text{C11}$ ($-67.3(1)^\circ$) and the dihedral angle between two benzimidazole groups ($74.4(1)^\circ$) which is apparently distinct from most of its analogs [7]. The single bonds $\text{C2}-\text{N2}$ and $\text{C11}-\text{N4}$ are respectively about $0.05 \text{ \AA}$ longer than the neighboring $\text{C2}-\text{N3}$ and $\text{C11}-\text{N5}$ double bonds, indicating their potentially donating electron ability.

In the crystal packing, the MeIDB molecules are linked mainly by a: one $\text{N1}-\text{H1}\cdots\text{N3}$ hydrogen bond ($d_{\text{N1}\cdots\text{N3}} = 3.164(1) \text{ \AA}$, $146.4(1)^\circ$, symmetry code: $1-x, 2-y, 1-z$); b: one $\pi\cdots\pi$ stacking between imidazole group ($\text{N4}/\text{N5}/\text{C11}/\text{C12}/\text{C17}$) and the symmetry related benzene rings ($\text{C12}-\text{C17}$, symmetry code: $2-x, 2-y, -z$) with a centroid-to-centroid distance of $3.569(2) \text{ \AA}$; c: one $\text{C}-\text{H}\cdots\pi$ interaction ($\text{C6}-\text{H6}\cdots\text{Cg}$ (Cg is the centroid of phenyl $\text{C12}-\text{C17}$, symmetry code: $2-x, 1-y, 1-z$) connecting molecules into a two-dimensional network parallel to the $[111]$ plane. There are no specific directional intermolecular interactions between these adjacent two dimensional layers PLATON [12]. Finally it should be mentioned that the title molecule has been used as a ligand [13].

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