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2-Hydroxymethyl-1-pentylbenzimidazole Hemi(1,4-dioxane) Solvate

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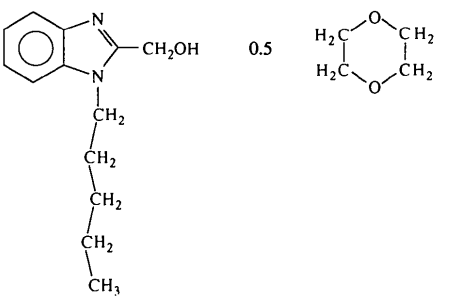
Abstract

2-Hydroxymethyl-1-pentylbenzimidazole serves as a simple model for the active site of hydrolytic metalloenzymes. The crystal structure shows that this compound crystallizes with 1,4-dioxane molecules: two benzimidazole units for one solvent molecule, $C_{13}H_{18}N_2O.0.5C_4H_8O_2$. The benzimidazole rings are hydrogen bonded (O—H···N) into dimers.

Comment

2-Hydroxymethyl-1-pentylbenzimidazole was found to be active under its complexed form in the trans-esterification reaction of co-complexing activated esters (*e.g.* 4-nitrophenyl-5-alkoxypicolinic esters; Faivre, 1993) in cetyltrimethylammonium bromide (CTAB) micellar solutions. Indeed, this compound is capable, through its chelating sites, of forming active complexes with bivalent metal ions (*e.g.* Zn^{2+}) in the presence of non-ionic or cationic surfactants. Unlike the 5(6)-alkyl-chain substituted homologues which give, whatever the surfactant concentration, an active complex with only a 2/1 stoichiometry, 2-hydroxymethyl-1-pentylbenzimidazole forms two active bidentate complexes simultaneously with 1/1 and 2/1 stoichiometry types at low surfactant concentrations (slightly above the cmc). At higher surfactant concentrations, these complexes evolve to only an active 2/1 complex type (Faivre, Brembilla, Roizard & Lochon, 1991). Therefore, the interpretation of the complexation mode as a function of the surfactant concentration initially prompted us to determine the crystal structure of this ligand (I) by X-ray crystallography.



(I)

The experimental intramolecular parameters (bond lengths and angles) of the heterocyclic ring are in good agreement with those determined for the non-substituted 2-hydroxymethylbenzimidazole (Aubry, Brembilla, Faivre & Lochon, 1995).

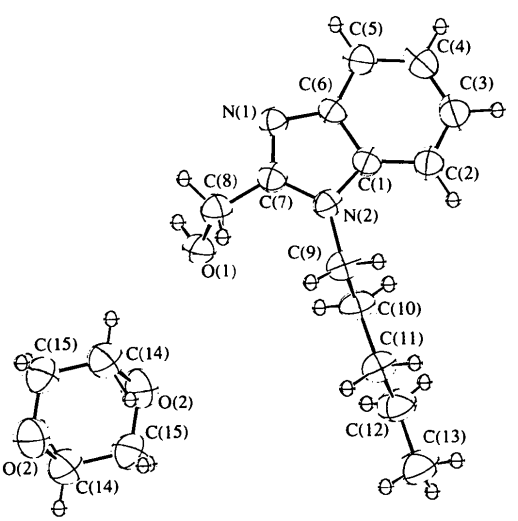


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title molecule with the atom-numbering scheme. Ellipsoids are plotted at the 50% probability level.

The examination of the intermolecular distances shows that the molecules are associated as dimers by means of hydrogen bonds with an O(1)···N(1) distance of 2.813 (2) Å [symmetry code: $-x$, $-y$, $-z$] and O(1)—H···N(1) = 164 (2)°. The aliphatic chain on the N atom is fully extended with an average torsion angle of 177 (1)°.

Experimental

Crystals of the title compound were obtained by recrystallization from dioxane.

Crystal data

$C_{13}H_{18}N_2O.0.5C_4H_8O_2$
$M_r = 262.35$
Monoclinic
$P2_1/c$
$a = 12.801\ (1)$ Å
$b = 7.819\ (1)$ Å
$c = 15.169\ (2)$ Å
$\beta = 101.08\ (1)°$
$V = 1490$ Å^3
$Z = 4$
$D_x = 1.17$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 20$–$30°$
$\mu = 0.547$ mm^{-1}
$T = 293$ K
Needle
0.6 × 0.1 × 0.1 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2834 measured reflections
2664 independent reflections
2355 observed reflections [$I \geq 3\sigma(I)$]
$\theta_{max} = 70°$
$h = -15 \rightarrow 15$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 18$
2 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F
$R = 0.044$
$wR = 0.046$
$S = 4.34$
2355 reflections
261 parameters
All H-atom parameters refined
$w = 25.18/[\sigma^2(F) + 0.00004F^2]$
$(\Delta/\sigma)_{max} = 0.4$ (on heavy atoms)
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = 0.22$ e Å^{-3}
Extinction correction: *SHELX*90 (Sheldrick, 1990)
Extinction coefficient: 0.032
Atomic scattering factors from *SHELX*90

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
N(1)	−0.0565 (1)	0.2249 (2)	−0.0425 (9)	3.87 (3)
N(2)	0.0790 (1)	0.3866 (2)	−0.0682 (9)	3.67 (3)
O(1)	0.1805 (1)	0.0730 (2)	0.0465 (9)	5.06 (3)
C(1)	−0.0036 (1)	0.4119 (2)	−0.1405 (1)	3.67 (4)
C(2)	−0.0116 (2)	0.5099 (2)	−0.2181 (1)	4.67 (4)
C(3)	−0.1069 (2)	0.5027 (3)	−0.2777 (1)	5.28 (5)
C(4)	−0.1919 (2)	0.4054 (3)	−0.2609 (1)	5.25 (5)
C(5)	−0.1839 (1)	0.3089 (3)	−0.1838 (1)	4.64 (5)
C(6)	−0.0875 (1)	0.3113 (2)	−0.1234 (1)	3.67 (4)
C(7)	0.0428 (1)	0.2722 (2)	−0.0125 (1)	3.69 (4)
C(8)	0.1113 (2)	0.1994 (3)	0.0699 (1)	4.60 (5)
C(9)	0.1848 (1)	0.4643 (3)	−0.0582 (1)	4.27 (4)
C(10)	0.2504 (1)	0.3853 (3)	−0.1210 (1)	4.44 (4)
C(11)	0.3559 (2)	0.4747 (3)	−0.1172 (1)	4.52 (5)
C(12)	0.4182 (2)	0.4041 (4)	−0.1841 (2)	5.69 (6)
C(13)	0.5244 (2)	0.4886 (5)	−0.1804 (2)	7.54 (9)
O(2)	0.5539 (1)	0.4411 (2)	−0.4166 (9)	7.42 (5)
C(14)	0.4033 (2)	0.5830 (4)	−0.5045 (2)	6.73 (7)
C(15)	0.4411 (2)	0.4448 (5)	−0.4396 (2)	7.45 (8)

Table 2. *Geometric parameters* (Å, °)

N(1)—C(7)	1.318 (2)	C(6)—N(1)	1.391 (2)
N(2)—C(9)	1.466 (2)	C(7)—C(8)	1.495 (2)
C(1)—C(2)	1.392 (2)	C(7)—N(2)	1.371 (2)
C(1)—C(6)	1.395 (2)	C(8)—O(1)	1.418 (3)
C(1)—N(2)	1.383 (2)	C(9)—C(10)	1.516 (3)
C(2)—C(3)	1.373 (3)	C(10)—C(11)	1.511 (3)
C(3)—C(4)	1.391 (3)	C(11)—C(12)	1.512 (3)
C(4)—C(5)	1.388 (2)	C(12)—C(13)	1.503 (4)
C(5)—C(6)	1.391 (2)		

C(6)—N(1)—C(7)	105.2 (1)	C(1)—C(6)—C(5)	120.3 (2)
C(1)—N(2)—C(7)	106.5 (1)	C(1)—C(6)—N(1)	109.8 (1)
C(1)—N(2)—C(9)	125.0 (1)	C(5)—C(6)—N(1)	130.0 (2)
C(7)—N(2)—C(9)	128.4 (1)	C(8)—C(7)—N(2)	123.4 (1)
C(2)—C(1)—C(6)	122.2 (1)	C(7)—C(8)—O(1)	110.5 (1)
C(2)—C(1)—N(2)	132.1 (2)	C(9)—C(10)—C(11)	112.7 (2)
C(6)—C(1)—N(2)	105.7 (1)	C(10)—C(11)—C(12)	112.7 (2)
C(1)—C(2)—C(3)	116.5 (2)	C(11)—C(12)—C(13)	113.9 (2)
C(2)—C(3)—C(4)	122.0 (1)	N(1)—C(7)—C(8)	123.6 (2)
C(3)—C(4)—C(5)	121.4 (2)	N(1)—C(7)—N(2)	112.9 (2)
C(4)—C(5)—C(6)	117.6 (2)	N(2)—C(9)—C(10)	112.3 (2)

N(2)—C(7)—C(8)—O(1)	−75.7 (2)
C(7)—N(2)—C(9)—C(10)	104.3 (2)
N(2)—C(9)—C(10)—C(11)	175.0 (2)
C(9)—C(10)—C(11)—C(12)	−176.1 (2)
C(10)—C(11)—C(12)—C(13)	−178.9 (3)

Following recommendations by Taylor & Kennard (1983), the H(N) atoms were placed at 1.03 Å from the parent N atoms in the direction obtained by refinement.

Programs used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Molecular drawing: *ORTEP* (Johnson, 1965). Full-matrix least-squares refinement: *SHELX*90 (Sheldrick, 1990).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: PA1179). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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