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2-Hydroxymethylbenzimidazole

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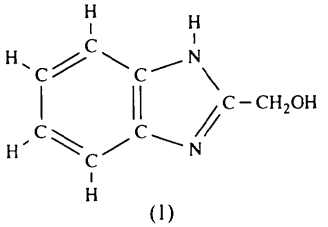
Abstract

Interest in the title compound, 2-hydroxymethylbenzimidazole, $C_8H_8N_2O$, arises from its two chemical functional groups, the imidazole ring and the hydroxymethyl group, which are often encountered in the active sites of some enzymes. This compound crystallizes with two independent molecules per asymmetric unit which are connected through intermolecular hydrogen bonds (N—H···N and O—H···O).

Comment

2-Hydroxymethylbenzimidazole (1), which is described as a model for the active site of some metalloenzymes, can be complexed in aqueous or micellar media by bivalent metallic cations. The resulting complex, the stoichiometry of which depends upon the nature of the medium, exhibits interesting catalytic properties in the hydrolysis reaction of co-complexing activated esters (Brembilla & Lochon, 1988). In order to establish a relationship between the chemical structure of this ligand, its ability to form complexes and the efficiency of the these complexes, we have undertaken the determination of the crystal structure by X-ray crystallography.

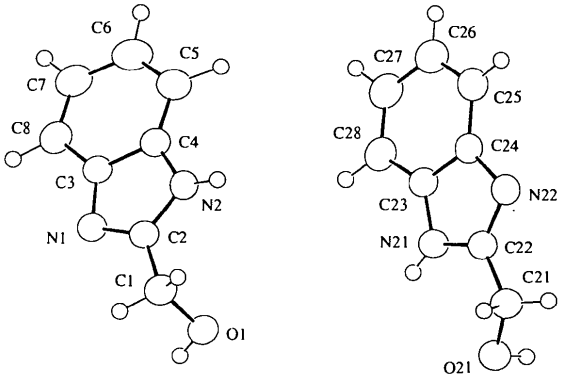


(1)

The experimental intramolecular distances of the heterocyclic ring are in good agreement with those determined for benzimidazole (Escande & Galigné, 1974). Moreover, the presence of the hydroxymethyl group gives, in comparison to the unsubstituted benzimidazole molecule, additional possible associations through a particular hydrogen-bonding network.

Examination of the intermolecular and interatomic distances shows that the two molecules (I) and (II) are associated by means of two types of hydrogen bonds [N2—H···N22^{i} = 2.922 (2) and O21—H···O1^{ii} = 2.735 (2) Å].

Each (I) [or (II)] molecule is also bonded to another identical neighbouring molecule of (I) [or (II)] [O1—H···N1^{iii} = 2.723 (2) and N21—H···O21^{ii} = 2.846 (2) Å; symmetry codes: (i) $1-x, 1-y, -z$; (ii) $1-x, -y, -z$; (iii) $2-x, -y, -z$].



(I) (II)

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the 2-hydroxymethylbenzimidazole molecule with the atom-numbering scheme.

Experimental

Crystal data

$C_8H_8N_2O$
M_r = 148.16
Monoclinic
$P2_1/c$
a = 11.410 (1) Å
b = 7.663 (1) Å
c = 17.404 (2) Å
β = 103.31 (1)°
V = 1481 Å^3
Z = 8
D_x = 1.33 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 20–30°
μ = 0.656 mm^{-1}
T = 293 K
Prismatic
0.30 × 0.2 × 0.15 mm
Colourless
Crystal source: recrystallization from ethanol–nitromethane (25/75 *v/v*)

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2538 measured reflections
2533 independent reflections
2330 observed reflections [$I \geq 3\sigma(I)$]
θ_{max} = 70°
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 16$
2 standard reflections
frequency: 120 min
intensity variation: none

Refinement

Refinement on F	$w = 1/[\sigma^2(F) + 0.00395F^2]$
$R = 0.0425$	$(\Delta/\sigma)_{max} = 0.3$
$wR = 0.057$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
$S = 0.97$	$\Delta\rho_{min} = -0.29$ e Å^{-3}
2330 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
248 parameters	
Only coordinates of H atoms refined	

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}
Molecule (I)				
O1	0.80587 (9)	0.0451 (1)	−0.05992 (7)	3.68 (3)
C1	0.8811 (2)	0.1869 (2)	−0.0707 (1)	3.64 (4)
C2	0.9389 (1)	0.2719 (2)	0.00570 (9)	2.99 (3)
N1	1.0495 (1)	0.2413 (2)	0.04599 (7)	3.26 (3)
C3	1.0634 (1)	0.3424 (2)	0.11368 (9)	3.06 (3)
C4	0.9568 (1)	0.4329 (2)	0.11275 (9)	3.16 (3)
N2	0.8789 (1)	0.3850 (2)	0.04297 (1)	3.15 (3)
C5	0.9444 (2)	0.5403 (2)	0.1747 (1)	4.14 (4)
C6	1.0436 (2)	0.5567 (3)	0.2371 (1)	4.63 (4)
C7	1.1522 (2)	0.4701 (2)	0.2378 (1)	4.31 (4)
C8	1.1640 (1)	0.3637 (2)	0.1763 (1)	3.87 (4)
Molecule (II)				
O21	0.3368 (1)	0.0119 (2)	−0.04423 (8)	4.12 (3)
C21	0.3126 (2)	0.1926 (2)	−0.0541 (1)	3.65 (4)
C22	0.3815 (1)	0.2983 (2)	0.01385 (9)	3.13 (3)
N21	0.4887 (1)	0.2441 (2)	0.05828 (8)	3.40 (3)
C23	0.5277 (1)	0.3689 (2)	0.11477 (9)	3.19 (3)
C24	0.4382 (1)	0.4976 (2)	0.10114 (9)	3.29 (3)
N22	0.3468 (1)	0.4486 (2)	0.03662 (8)	3.41 (3)
C25	0.4484 (2)	0.6415 (3)	0.1508 (1)	4.37 (4)
C26	0.5476 (2)	0.6501 (3)	0.2136 (1)	4.73 (5)
C27	0.6369 (2)	0.5202 (3)	0.2262 (1)	4.35 (4)
C28	0.6291 (1)	0.3787 (2)	0.1767 (1)	4.05 (4)

Table 2. *Selected geometric parameters* (Å, °)

Molecule (I)		Molecule (II)	
O1—C1	1.423 (2)	O21—C21	1.414 (2)
C1—C2	1.491 (2)	C21—C22	1.498 (2)
C2—N1	1.316 (2)	C22—N22	1.309 (2)
C2—N2	1.359 (2)	C22—N21	1.353 (2)
N1—C3	1.388 (2)	N21—C23	1.370 (2)
C3—C4	1.398 (2)	C23—C24	1.400 (2)
C3—C8	1.398 (2)	C23—C28	1.389 (2)
C4—N2	1.379 (2)	C24—N22	1.396 (2)
C4—C5	1.389 (2)	C24—C25	1.389 (2)
C5—C6	1.383 (2)	C25—C26	1.382 (2)
C6—C7	1.404 (3)	C26—C27	1.406 (3)
C7—C8	1.377 (3)	C27—C28	1.376 (3)
O1—C1—C2	111.9 (1)	O21—C21—C22	112.4 (1)
C1—C2—N1	124.8 (1)	C21—C22—N21	121.8 (1)
C1—C2—N2	122.4 (1)	C21—C22—N22	125.1 (1)
N1—C2—N2	112.7 (1)	N21—C22—N22	113.2 (1)
N1—C3—C4	109.8 (1)	N21—C23—C24	105.4 (1)
N1—C3—C8	129.7 (1)	N21—C23—C28	132.0 (1)
C2—N1—C3	105.1 (1)	C22—N21—C23	107.3 (1)
C2—N2—C4	107.3 (1)	C22—N22—C24	105.0 (1)
C3—C4—N2	105.1 (1)	C23—C24—N22	109.1 (1)
C3—C4—C5	121.9 (1)	C23—C24—C25	120.0 (1)
C3—C8—C7	117.8 (2)	C23—C28—C27	116.8 (1)
N2—C4—C5	132.9 (1)	N22—C24—C25	130.8 (1)
C4—C3—C8	120.5 (1)	C24—C23—C28	122.5 (1)
C4—C5—C6	116.9 (2)	C24—C25—C26	117.8 (2)
C5—C6—C7	121.7 (2)	C25—C26—C27	121.4 (2)
C6—C7—C8	121.1 (2)	C26—C27—C28	121.4 (2)
O1—C1—C2—N1	98.63	O21—C21—C22—N21	27.78
O1—C1—C2—N2	−78.14	O21—C21—C22—N22	−152.28

Program used to solve structure: *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Molecular drawing: *ORTEP* (Johnson, 1965). Full-matrix least-squares refinement: *SHELX* (Sheldrick, 1976). Following recommendations by Taylor & Kennard (1983), the H(N) atoms were placed at 1.03 Å from the parent N atom in the direction obtained from the refinement.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: PA1100). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Isomeric Sulfites of 10β-Pinane-2,3α-diol

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Abstract

In the title compounds, *cis*- and *trans*-3a,8,8-trimethyl-3a,4,5,6,7,7a-hexahydro-4,6-methano-1,3,2-dioxathiolane 2-oxide, $C_{10}H_{16}O_3S$, the five-membered ring adopts a half-chair (envelope) conformation. The orientation of the methyl group C7 substituted on C1, being either *cis* or *trans* to the exocyclic S=O bond, is the main structural difference between the two isomers.

Comment

The structure determination of isomer (1) and isomer (2) of the cyclic sulfites of (±)-10β-pinane-