Anisotropic displacement parameters were used for all non-H atoms. All C-bound H atoms were placed in calculated positions, refined using a riding model, and given isotropic displacement parameters equal to 1.2 times the equivalent isotropic displacement parameter of the C atom to which they are attached. The C—H distances used depend on the temperature and type of C atom: for (1) and (2) $C_{aromatic}$—H = 0.95 Å at 173 K; for (3) $C_{tertiary}$—H = 0.98, $C_{secondary}$—H = 0.97, $C_{aromatic}$—H = 0.93 Å at 296 K. Positional parameters for all heteroatom-bound H atoms were refined. They were given isotropic displacement parameters equal to 1.5 times the equivalent isotropic displacement parameter of the atom to which they are attached. Two full octants of data ($-h$, $-k$, $+l$ and $+h$, $+k$, $-l$) and one partial octant ($\theta \le 15°$ $+h$, $+k$, $+l$) were collected for (1). The refinement of (1) was performed without averaging Friedel pairs. The absolute structure of (1) was not reliably determined as the Flack parameter (Flack, 1983) has a high s.u. for both structures, while the residuals were unchanged.

For all compounds, data collection: CAD-4-PC (Enraf-Nonius, 1993); cell refinement: CAD-4-PC; data reduction: XCAD4 (Harms, 1995); program(s) used to solve structures: SHELXS86 (Sheldrick, 1990); program(s) used to refine structures: SHELXL93 (Sheldrick, 1993); molecular graphics: SHELXTL (Siemens, 1990); software used to prepare material for publication: PLATON (Spek, 1990).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1128). Services for accessing these data are described at the back of the journal.

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1,4-Bis(imidazol-1-ylmethyl)benzene Dihydrate

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Abstract

An analysis of the structure of 1,4-bis(imidazol-1-ylmethyl)benzene dihydrate, $C_{14}H_{14}N_4.2H_2O$, shows that the imidazole moieties hydrogen bond to water molecules to form an extended two-dimensional sheet.

Comment

Metal complexes of 1,4-bis(imidazol-1-ylmethyl)benzene, hereafter bix, have been shown recently to form coordination polymers incorporating unusual polyrotaxane interactions (Hoskins *et al.*, 1997*a,b*). Within each coordination polymer two distinct types of conformation are observed, *i.e.* a centrosymmetric conformation and a 'half-loop' conformation in which bix forms part of an $M_2(bix)_2$ macrocycle. As part of a more general investigation into the ligand and its conformational preferences, bix was isolated as its dihydrate, (I).

(I)
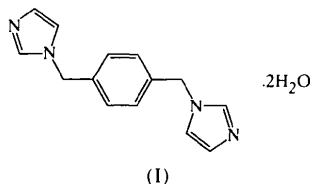
The midpoint of the phenylene ring lies on a centre of symmetry and the terminal N atoms of the imidazole rings form hydrogen bonds [N···O 2.822 (5) Å] to the O atoms of two water molecules, as shown in Fig. 1. Further hydrogen bonding between water molecules [O···O 2.727 (9) and 2.781 (9) Å] results in a

two-dimensional sheet with the same connectivity as a hexagonal grid (Fig. 2). The conformation adopted by the uncoordinated bix molecule is very similar to the centrosymmetric type of conformation adopted by bix in the reported coordination polymers.
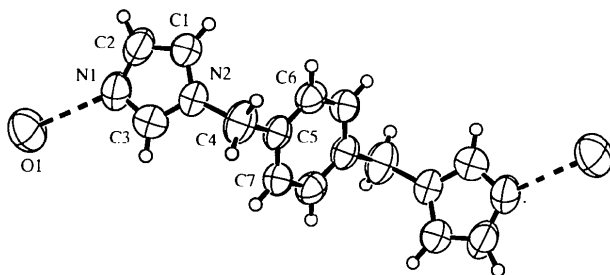

Fig. 1. *ORTEP*II (Johnson, 1976) diagram of (I). Displacement ellipsoids are at the 50% probability level and H atoms are of arbitrary size. Hydrogen bonds are indicated by dashed lines.
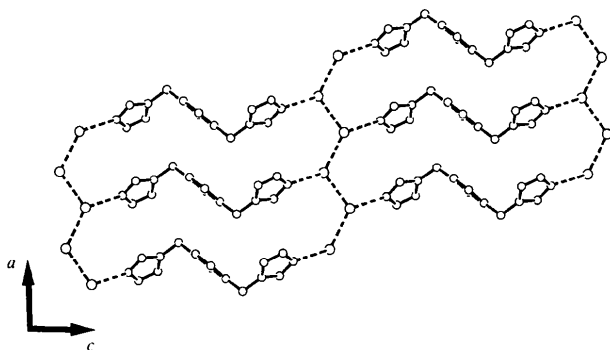

Fig. 2. *ORTEP*II (Johnson, 1976) diagram showing the hydrogen bonding between bix and the water molecules. Atoms are of arbitrary size. Hydrogen bonds are indicated by dashed lines.

Experimental

The title compound, bix dihydrate, was prepared according to the literature method of Hoskins *et al.* (1997a). Small needles of the dihydrate were obtained by recrystallization from water.

Crystal data

$C_{14}H_{14}N_4.2H_2O$
$M_r = 274.32$
Monoclinic
$P2_1/n$
$a = 4.669 (2)$ Å
$b = 9.622 (3)$ Å
$c = 16.377 (6)$ Å
$\beta = 94.57 (5)°$
$V = 733.3 (5)$ Å^3
$Z = 2$
$D_x = 1.242$ Mg m^{-3}
$D_m = 1.25 (1)$ Mg m^{-3}
D_m measured by flotation in chlorobenzene and dichloromethane

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 4.92–10.54°$
$\mu = 0.086$ mm^{-1}
$T = 293 (2)$ K
Needle
$0.200 \times 0.050 \times 0.038$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega–2\theta$ scans
Absorption correction: analytical, indexed crystal faces (Sheldrick, 1976)
$T_{min} = 0.990$, $T_{max} = 0.993$
1803 measured reflections
1295 independent reflections

618 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 24.97°$
$h = -1 \rightarrow 5$
$k = 0 \rightarrow 11$
$l = -19 \rightarrow 19$
3 standard reflections frequency: 120 min intensity decay: 0 (1)%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.255$
$S = 1.011$
1294 reflections
96 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.1326P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.259$ e Å^{-3}
$\Delta\rho_{min} = -0.193$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
N1	-0.0766 (10)	0.8351 (5)	0.1863 (2)	0.0916 (14)
N2	0.1318 (7)	0.8096 (4)	0.3108 (2)	0.0664 (10)
C1	-0.0637 (11)	0.7069 (5)	0.2982 (3)	0.0775 (13)
C2	-0.1837 (11)	0.7242 (6)	0.2227 (3)	0.089 (2)
C3	0.1127 (12)	0.8835 (5)	0.2416 (3)	0.088 (2)
C4	0.3135 (11)	0.8404 (6)	0.3862 (3)	0.087 (2)
C5	0.1515 (9)	0.9226 (5)	0.4456 (2)	0.0640 (11)
C6	0.1673 (10)	1.0658 (5)	0.4465 (2)	0.0721 (13)
C7	-0.0148 (10)	0.8585 (5)	0.4995 (3)	0.0729 (12)
O1	-0.2341 (10)	0.9929 (4)	0.0446 (2)	0.1210 (15)

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

N1—C3	1.301 (6)	C1—C2	1.328 (6)
N1—C2	1.338 (6)	C4—C5	1.503 (6)
N2—C3	1.335 (5)	C5—C7	1.368 (6)
N2—C1	1.350 (5)	C5—C6	1.381 (6)
N2—C4	1.471 (5)	C6—C7^i	1.386 (6)
C3—N1—C2	103.4 (4)	N2—C4—C5	111.3 (4)
C3—N2—C1	105.2 (4)	C7—C5—C6	118.4 (4)
C3—N2—C4	126.9 (4)	C7—C5—C4	121.4 (4)
C1—N2—C4	127.9 (4)	C6—C5—C4	120.2 (5)
C2—C1—N2	106.3 (4)	C5—C6—C7^i	120.1 (4)
C1—C2—N1	111.7 (5)	C5—C7—C6^i	121.5 (4)
N1—C3—N2	113.4 (4)		

Symmetry code: (i) $-x, 2 - y, 1 - z$.

Crystals of the title compound were small and weakly diffracting. The structure was solved using the *TREF* routine of *SHELXS86* (Sheldrick, 1990). Refinement was performed using *SHELXL93* (Sheldrick, 1993). All non-H atoms were refined with anisotropic displacement parameters. The H atoms of the bix molecule were refined isotropically and constrained to their geometrically calculated positions. The H atoms of the water molecule were not found, assigned or refined.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *PRO-CESS DATA* (Gable *et al.*, 1993). Molecular graphics: *OR-*

*TEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

We are grateful to the Australian Research Council for its support.

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Acta Cryst. (1998). C**54**, 1668–1670

Bis(4-chlorophenyl)(5-pyrimidinyl)methanol

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Abstract

The title compound, $C_{17}H_{12}Cl_2N_2O$, has a twisted conformation with the following angles between the three planes in the molecule: $86.0\,(4)°$ between the two *p*-chlorophenyl groups [planes 1 and 2], $85.0\,(4)°$ between plane 1 and the pyrimidine ring (plane 3), and $90.5\,(4)°$ between planes 2 and 3. There is an intermolecular hydrogen bond [O···N $2.847\,(4)$ Å and O—H···N $161\,(5)°$].

Comment

The title compound (LY43578), (1), is very similar in structure to both parinol, (2) (Kennard *et al.*, 1981),

and fenarimol, (3) (Albinati *et al.*, 1988), which show fungicidal activity as a result of inhibition of a P450 enzyme. Parinol, (2), has a pyridine group instead of the pyrimidine heterocycle in the title compound. Fenarimol, (3), is a structural isomer of the title compound, where one of the two Cl atoms is in an *ortho* position rather than the *para* position of the title compound. Following reports that certain nitrogen heterocyclic antifungal agents were effective inhibitors of another P450 enzyme, aromatase (Taylor *et al.*, 1987; Mason *et al.*, 1985), the title compound was synthesized as part of a structure–activity study in which inhibitors for the P450–aromatase system were developed (Jones *et al.*, 1990). It shows moderate activity as a P450–aromatase inhibitor (Jones *et al.*, 1990) and is related to LY113174 (Caruso & Rossi, 1998) and LY56110 (Lindstrom & Whitaker, 1987; Hirsch *et al.*, 1987).

(1)

(2)

(3)
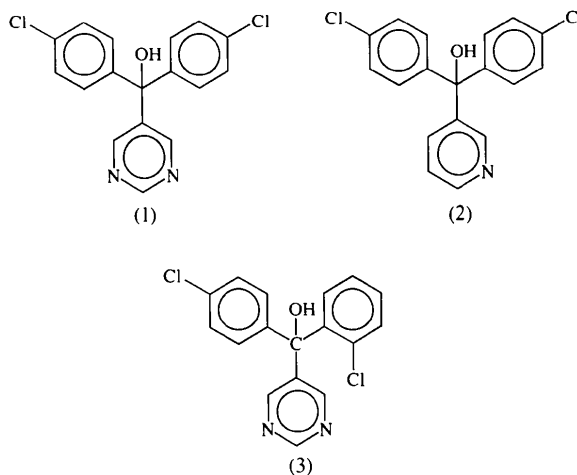
The bond distances and angles in the molecule, including the single bonds involving C(17) and the aromatic bonds, show no unusual features. The expected phenyl-ring bond-angle distortion pattern caused by Cl-atom substitution is seen (Brisse & Sygusch, 1974; Domenicano & Murray-Rust, 1979). There is no significant deviation of any atom from the three principal planes of the molecule, deviations which include Cl(1) at $0.036\,(4)$ Å and Cl(2) at $0.059\,(4)$ Å from their respective planes. The O—C_{tet}—C_{ipso}—C_{ortho} torsion angles, which show the relationship between the four substituents on the C(17) atom, are listed in Table 1.

The analogous O—C_{tet}—C_{ipso}—C_{ortho} torsion angles for parinol, (2), are: $155.1\,(3)$ and $-23.3\,(3)°$ for the first *p*-chlorophenyl ring, $155.9\,(3)$ and $-20.4\,(3)°$ for the second *p*-chlorophenyl ring, and $-78.2\,(3)$ and $91.5\,(3)°$ for the pyridine ring. Whereas the title compound has angles between the planes of the rings close to 90°, in parinol, these angles are 77° between the two *p*-chlorophenyl rings, 116° between one phenyl ring and the