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3,6-Bis(4-hydroxybenzyl)piperazine-2,5-dione

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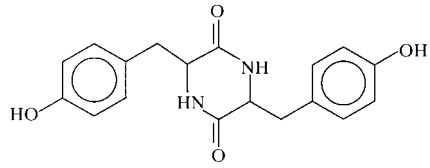
In the title compound, $C_{18}H_{16}N_2O_4$, the piperidine ring adopts a chair conformation, lying on an inversion centre. The 4-hydroxybenzyl groups are in quasi-axial positions. A two-dimensional network is formed through N—H···O and O—H···O intermolecular hydrogen bonds and C—H···O interactions.

Comment

The optical properties of the title compound were investigated as a function of molecular conformation (Snow *et al.*, 1977). The synthesis and antimicrobial activity of the title compound were studied by Gadaginamath *et al.* (1996). Michael & John (1985) studied the organic chemisty of L-tyrosine and concluded several general synthetic methods of chiral piperazines from amino acids. Liebscher & Jin (1999) reviewed the synthetic methods of the piperazine-2,5-diones from 3-ylidenepiperazine-2,5-diones.

In the title compound, (I), the bond lengths and angles of the diketopiperazine ring are comparable with related reported values (Szkaradzinska *et al.*, 1994; Sterns *et al.*, 1989). The piperidine ring adopts a chair conformation inclining towards planarity. The total puckering amplitude Q_T = 0.087 (3) Å (Cremer & Pople, 1975). The O1 atom deviates by 0.125 (2) Å from the mean plane through the piperazine ring. The 4-hydroxybenzyl groups at C8 and C8*A* are in quasi-axial positions [C7—C8—N1—C9 −113.4 (2)°]. This conformation is also found in the derivative of lichen diketopiperazine metabolite methylanhydropicroroccellin reported by Sterns *et al.* (1989). The phenyl rings are planar, making a dihedral angle of 54.6 (1)° with the mean plane of the piperidine ring.



(I)

In the crystal, N1—H1*B*···O2(x, $1-y$, $-\frac{1}{2}+z$) and O2—H2*B*···O1($\frac{1}{2}-x$, $-\frac{1}{2}+y$, $\frac{3}{2}-z$) intermolecular hydrogen bonds link the molecules along the *c* axis. Adjacent chains are interlinked by C4—H4*A*···O1($-\frac{1}{2}+x$, $-\frac{1}{2}+y$, z) interactions. These N—H···O and O—H···O intermolecular hydrogen bonds and C—H···O interactions form a two-dimensional network.

Experimental

The title compound was synthesized under solvothermal conditions. A heavy walled Pyrex tube containing a mixture of NaMe (0.0108 g, 0.2 mmol), L-tyrosine (0.0181 g, 0.1 mmol) and $LaCl_3$ (0.0246 g, 0.1 mmol) in methanol (1 ml) was frozen and sealed under vacuum and placed inside an oven at 393 K. The colourless prismatic crystals obtained were harvested after two weeks of heating. The IR spectrum confirmed the pure phase.

Crystal data

$C_{18}H_{18}N_2O_4$	D_x = 1.320 Mg m^{-3}
M_r = 326.34	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 2073 reflections
a = 16.2590 (6) Å	θ = 2.93–28.31°
b = 8.0575 (2) Å	μ = 0.094 mm^{-1}
c = 14.7794 (6) Å	T = 293 (2) K
β = 121.967 (2)°	Plate, colourless
V = 1642.6 (1) Å^3	0.22 × 0.16 × 0.06 mm
Z = 4	

Data collection

Siemens SMART CCD area-detector diffractometer	R_{int} = 0.082
ω scans	θ_{max} = 27.49°
5346 measured reflections	$h = -11 \rightarrow 21$
1876 independent reflections	$k = -10 \rightarrow 10$
1028 reflections with $I > 2\sigma(I)$	$l = -19 \rightarrow 18$
	Intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0650P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.055	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.146	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
S = 0.90	$\Delta\rho_{min}$ = −0.28 e Å^{-3}
1876 reflections	Extinction correction: *SHELXTL*
110 parameters	Extinction coefficient: 0.0078 (16)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å).

O1—C9	1.236 (2)	N1—C8	1.463 (3)
O2—C3	1.381 (3)	C7—C8	1.552 (3)
N1—C9	1.332 (3)		

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve

structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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