Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(4-phenylpiperazin-1-yl)methane

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.060
wR factor = 0.181
Data-to-parameter ratio = 15.2

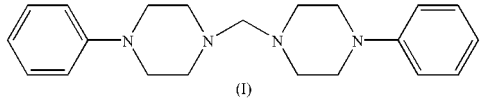
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 29 October 2003
Accepted 11 November 2003
Online 22 November 2003

The title compound, $C_{21}H_{28}N_4$, was synthesized. It is an unusual example of a Z' = 4 structure without obvious pseudosymmetry. The molecules are linked by numerous C—H···N and C—H···π hydrogen bonds; there are many H···H short contacts.

Comment

Crystal structures containing several molecules in the asymmetric unit ($Z' > 1$) include widely distributed species of hydroxy-containing compounds forming strong hydrogen-bonded assemblies in crystals (Kuleshova *et al.*, 2003; Lehmler *et al.*, 2002). Approximate symmetry relationships between independent molecules are apparent in many structures with $Z' > 1$, such as 4-biphenylol (Z' = 2) (Brock & Haller, 1984) and 2-cyclopentyl-2-hydroxy-2-phenylacetic acid (Z' = 2) (Bats *et al.*, 2003). 4-Chloro-2′-biphenyl, a Z' = 4 structure without obvious pseudosymmetry, is an unusual example discovered recently (Lehmler *et al.*, 2002). The title compound, (I), can be regarded as a symmetric molecule; however, the crystal structure has Z' = 4 without obvious pseudosymmetry. The molecules are linked by many C—H···N and C—H···π hydrogen bonds; there are many H···H short contacts (Steiner, 2002).



(I)

Experimental

The title compound was synthesized by reacting diiodomethane and the sodium salt of 1-phenylpiperazine, which was prepared *in situ* by a modification of the literature method (Garrard & Partridge, 1993), in dry DMF at 303 K for 12 h. Work-up gave the desired product (yield 73.2%), which was purified by recrystallization from hot alcohol, yielding white crystals of (I) (m.p. 397–399 K). ^{1}H NMR ($CDCl_3$, 500 MHz): δ 2.69 (*t*, J = 4.93 Hz, 8H), 3.04 (*s*, 2H), 3.20 (*t*, J = 4.93 Hz, 8H), 6.83–7.28 (*m*, 10H); ^{13}C NMR ($CDCl_3$, 500 MHz): δ 49.1, 51.5, 80.9, 116.0, 119.5, 129.0, 151.4; IR (KBr): ν 2957, 2880, 2821, 1600, 1503, 1452, 1371, 1237, 1147, 1014, 924, 756, 689 cm^{-1}. EI–MS m/z (%): 337 (M^+, 1.8), 174 (69.8), 120 (100), 77 (47.5).

Crystal data

$C_{21}H_{28}N_4$
M_r = 336.47
Monoclinic, $P2_1/c$
a = 19.049 (5) Å
b = 9.758 (8) Å
c = 41.950 (10) Å
β = 99.49 (2)°
V = 7691 (7) Å^3
Z = 16
D_x = 1.162 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5814 reflections
θ = 2.2–20.9°
μ = 0.07 mm^{-1}
T = 298 (2) K
Parallelepiped, colourless
0.30 × 0.30 × 0.20 mm

Data collection

Siemens SMART CCD area-detector diffractometer	6119 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.059$
ω scans	$\theta_{max} = 25.1^\circ$
Absorption correction: none	$h = -22 \rightarrow 22$
31805 measured reflections	$k = -11 \rightarrow 11$
13700 independent reflections	$l = -49 \rightarrow 35$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.060$	$w = 1/[\sigma^2(F_o^2) + (0.0808P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.181$	
$S = 0.94$	$(\Delta/\sigma)_{max} = 0.001$
13700 reflections	$\Delta\rho_{max} = 0.14$ e Å^{-3}
902 parameters	$\Delta\rho_{min} = -0.13$ e Å^{-3}

H atoms were refined isotropically. All H atoms were located by geometry calculations riding on their parent atoms.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97/2 (Sheldrick, 1997).

The authors gratefully acknowlege financial support from the Ministry of Education, China, and through project No. 60171008 supported by NSFC.

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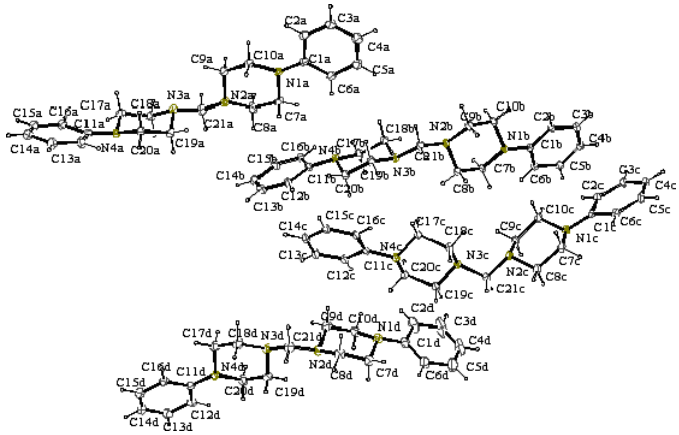

Figure 1
Structure of the asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 10% probability level.

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