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1,4-Dibenzylpiperazine-2,5-dione

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.067
wR factor = 0.094
Data-to-parameter ratio = 10.2

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

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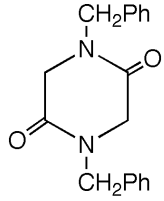
The title compound, $C_{18}H_{18}N_2O_2$, crystallizes in the centrosymmetric monoclinic space group *C*2/*c* with half the molecule as the asymmetric unit (molecules are located on inversion centers). π–π interactions between the benzene rings are found, with a perpendicular distance of 3.657 (11) Å between ring centroids.

Comment

The importance of π–π interactions in many molecules containing aromatic rings (Hunter & Sanders, 1990; Burley & Petsko, 1985; McGaughey *et al.*, 1998) has been well established in the literature. Piperazine-2,5-diones are commonly found in biologically active natural products; their cyclic dipeptide structure and prevalence in nature make them important targets in the search for new drugs (Witiak & Wei, 1990; Dinsmore & Beshore, 2002). This study of 1,4-dibenzylpiperazine-2,5-dione, (I), a piperazinedione with the potential for π–π interactions, is one of a series on these interactions in aromatic compounds, including a novel tris-indole amine (Carpenter *et al.*, 2004).



(I)

The title compound crystallizes in the centrosymmetric monoclinic space group *C*2/*c* (No 15) with half the molecule in the asymmetric unit (Fig. 1). This molecule exhibits π–π interactions between the benzene rings of adjacent molecules stacking down the *b* axis, with a perpendicular distance of 3.657 (11) Å between ring centroids. The benzene rings form an angle of 109.6 (2)° with the piperazine ring, with one angled in the positive direction and one in the negative direction, forming a zigzag pattern (Fig. 2).

The same molecular formula is shared by *cyclo*(-L-phenylalanyl-L-phenylalanyl-) (CPA) (Gdaniec & Liberek, 1986), the difference being that the benzyl groups are on the piperazine ring C atoms in that compound and on the N atoms in the title compound. The title compound clearly exhibits π–π interactions, while CPA does not. The CPA molecule has both benzyl groups *cis* to the piperazine ring, with dihedral angles to it of 55.4 (5) and 35.7 (1)° (our calculations), while in (I) the benzyl groups point in opposite directions. A novel tris-indolyl amine (Carpenter *et al.*, 2004) with three 'arms' extending from the

nitrogen center demonstrates π–π interactions between two of the arms with a perpendicular distance of 3.498 (4) Å, similar to the π–π interaction distance found in (I).

Experimental

The title compound was prepared by a modification of a previously reported procedure (Granacher *et al.*, 1928). Glycine anhydride was added to a stirred solution of NaH in DMF. When gas evolution had ceased (1.5 h), benzyl chloride was added and, after stirring for 18 h, the solution was filtered to give a white solid. Recrystallization of the solid from ethyl acetate gave colorless glassy crystals (yield 93.5%; m.p. 435–437 K). Please give quantities of reagents

Crystal data

$C_{18}H_{18}N_2O_2$
M_r = 294.35
Monoclinic, $C2/c$
a = 11.829 (9) Å
b = 4.482 (3) Å
c = 27.91 (1) Å
β = 91.50 (5)°
V = 1479.0 (16) Å^3
Z = 4
D_x = 1.322 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16 reflections
θ = 5.7–9.4°
μ = 0.09 mm^{-1}
T = 298 (2) K
Block, colorless
0.15 × 0.10 × 0.10 mm

Data collection

Rigaku AFC-7*S* diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.976, T_{max} = 0.991
7672 measured reflections
1705 independent reflections
1042 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.146
θ_{max} = 27.5°
$h = -15 \rightarrow 15$
$k = 0 \rightarrow 5$
$l = -36 \rightarrow 36$
3 standard reflections
every 150 reflections
intensity decay: 1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.094
S = 1.17
1187 reflections
116 parameters
All H-atom parameters refined
Weighting scheme: Chebychev polynomial with 3 parameters (Carruthers & Watkin, 1979)
613.558 846.432 246.609
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.235 (2)	C2—C3	1.369 (4)
N1—C7	1.463 (3)	C3—C4	1.381 (4)
N1—C8	1.341 (2)	C4—C5	1.365 (4)
C1—C2	1.392 (3)	C5—C6	1.381 (4)
C1—C6	1.384 (3)	C8—C9	1.484 (3)
C1—C7	1.507 (3)		
C7—N1—C8	120.6 (2)	C6—C5—C4	120.9 (3)
C2—C1—C6	118.1 (2)	C1—C6—C5	120.3 (2)
C2—C1—C7	121.2 (2)	N1—C7—C1	112.4 (2)
C6—C1—C7	120.7 (2)	C9—C8—O1	117.7 (2)
C3—C2—C1	121.2 (2)	C9—C8—N1	119.4 (2)
C4—C3—C2	120.0 (2)	O1—C8—N1	122.9 (2)
C5—C4—C3	119.4 (3)		

Atoms H2–H8 were positioned geometrically (C—H = 0.95 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C). Atoms H9 and H10 were refined freely [C—H = 0.95 (3) and 0.99 (3) Å]. The large difference in independent reflections and refined reflections is because there was a large number of weak reflections.

Data collection: *WinAFC* (Rigaku/MSC & Rigaku, 2001); cell refinement: *WinAFC*; data reduction: *CrystalStructure* (Rigaku/MSC, 2003); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

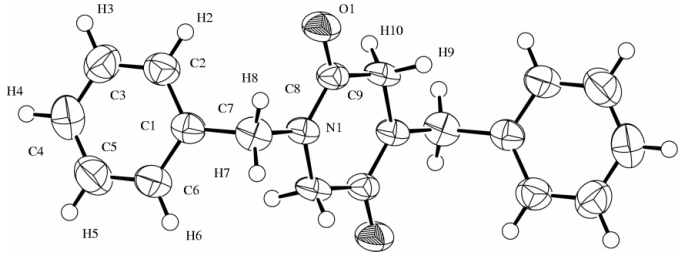


Figure 1
*ORTEP*III (Burnett & Johnson, 1996) drawing of the title molecule, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level for all non-H atoms. Unlabeled atoms are related to labeled atoms by the symmetry code $(-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2})$.

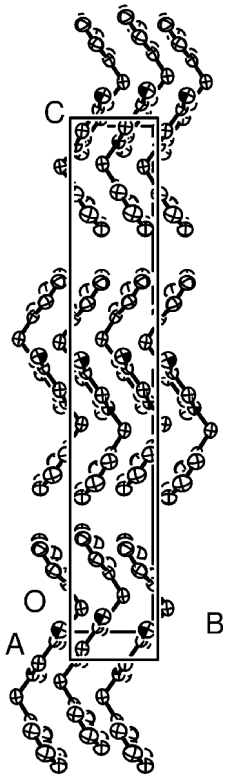


Figure 2
*ORTEP*III (Burnett & Johnson, 1996) packing diagram of the title compound. Displacement ellipsoids are drawn at the 50% probability level for all non-H atoms. H atoms have been omitted.

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