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1,4-Bis(4-methoxyphenyl)piperazine-2,5-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.119
Data-to-parameter ratio = 10.7

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

In the title compound, $C_{18}H_{18}N_2O_4$, the molecule has a crystallographically imposed center of symmetry. The dihedral angles between the piperazinedione ring and the two outer aromatic rings are 57.25 (7)°. Molecules of (I) interact *via* weak intermolecular C—H···O interactions, forming ribbons along the *b* axis.

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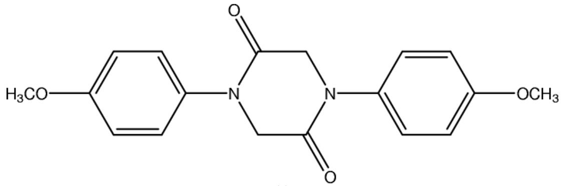
Comment

Piperazinedione and its derivatives are the smallest structural cyclodipeptides and are regarded as a by-product in the synthesis, especially in the solid-phase synthesis, of peptides (Gisin & Merrifield, 1972; Khosla *et al.*, 1972; Giralt *et al.*, 1981). Two hydrogen-bond donors and two acceptors are present in the molecule of piperazinedione, so its derivatives have been widely used in pharmaceutical chemistry, *e.g.* for the inhibition of cell growth cycles of mammals (Cui *et al.*, 1996; Edmondson *et al.*, 1999), the inhibition of glutathione-*S*-transferase (Sanatamaria *et al.*, 1999) and the inhibition of the activated factor of thrombocytes (Chu *et al.*, 1993). In the present paper, we describe the synthesis of the title compound, (I) (Fig. 1). An X-ray crystal analysis of (I) was undertaken to establish its molecular structure.



(I)

The title molecule has a crystallographically imposed center of symmetry. All bond lengths and angles in (I) have normal values (Allen *et al.*, 1987). The three rings are each coplanar with their attached groups, excluding methyl H atoms and the H atoms attached to the piperazinedione ring, while the whole molecule is not planar, with dihedral angles of 57.25 (7)° between the piperazinedione and the two aromatic rings. The

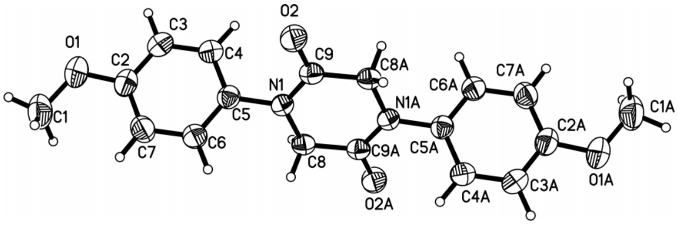


Figure 1
A view of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

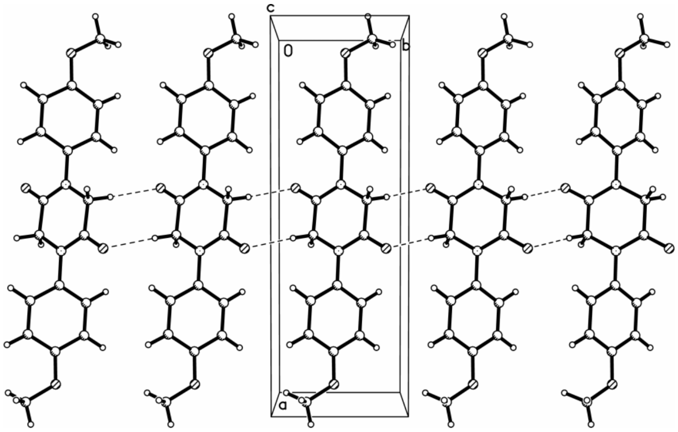


Figure 2
A packing diagram of (I), showing the formation of ribbons along the *b* axis *via* intermolecular C—H···O interactions (dashed lines).

sum of the bond angles around atom N1 [359.85°] indicates a planar configuration, different from the normal pyramidal configuration of an N atom. This difference is mainly due to the π conjugation effects arising from the presence of the two C=O double bonds.

Molecules of (I) interact *via* weak intermolecular C—H···O interactions (Table 2) to form ribbons along the *b* axis (Fig. 2). The packing is further stabilized by van der Waals forces.

Experimental

N-*p*-Methoxyphenyl chloroacetamide was prepared by the reaction of *p*-methoxyaniline and chloroacetyl chloride in the presence of triethylamine according to the method of Li (2001). To a solution of *N*-*p*-methoxyphenyl chloroacetamide (3.9 g, 20 mmol) in acetone (30 ml) were added K_2CO_3 (3.04 g, 22 mmol) and NaI (0.5 g), and the mixture was stirred at 329 K for 5 h. The mixture was washed three times with water and then filtered. The filter cake was washed with a small amount of acetone and water. The title compound was obtained after the resulting white powder had been dried at room temperature for 48 h. Colorless single crystals suitable for X-ray diffraction analysis were obtained, after 7 d, by slow evaporation of a $CHCl_3$–CH_3CH_2OH (1:1, *v*/*v*) solution.

Crystal data

$C_{18}H_{18}N_2O_4$
$M_r = 326.24$
Monoclinic, $P2_1/c$
$a = 16.9231\ (17)$ Å
$b = 5.8166\ (6)$ Å
$c = 8.0580\ (8)$ Å
$\beta = 92.687\ (2)°$
$V = 792.32\ (14)$ Å^3
$Z = 2$

$D_x = 1.368$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1695 reflections
$\theta = 2.4$–$26.1°$
$\mu = 0.10$ mm^{-1}
$T = 293\ (2)$ K
Column, colorless
0.42 × 0.21 × 0.19 mm

Data collection

Simens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.960$, $T_{\max} = 0.982$
4225 measured reflections

1557 independent reflections
1318 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\max} = 26.1°$
$h = -18 \rightarrow 20$
$k = -7 \rightarrow 6$
$l = -8 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.119$
$S = 1.04$
1557 reflections
146 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.1534P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.15$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.028 (5)

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.3736 (18)	N1—C9	1.3436 (17)
O1—C1	1.419 (2)	N1—C5	1.4399 (17)
O2—C9	1.2278 (16)	N1—C8	1.4569 (16)
C2—O1—C1	117.45 (14)	C9—N1—C8	122.62 (11)
C9—N1—C5	120.56 (11)	C5—N1—C8	116.67 (10)

Table 2
Intermolecular C—H···O contacts (A, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O2$^{\rm i}$	0.98 (1)	2.42 (1)	3.389 (1)	174 (1)

Symmetry code: (i) *x*, 1 + *y*, *z*.

All H atoms were located in difference Fourier maps and were refined isotropically [C—H = 0.946 (18)–1.04 (2) Å].

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*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Chu, M., Mierzwa, R., Fruumees, I., Gentile, F., Patel, M., Gullo, V., Chan, T.-M. & Puar, M. S. (1993). *Tetrahedron Lett.* **34**, 7537–7540.
Cui, C. B., Kakeya, H. & Osada, H. (1996). *Tetrahedron*, **52**, 12651–12666.
Edmondson, S., Danishefsky, S. J., Sepp-Lorenzino, L. & Rosen, N. (1999). *J. Am. Chem. Soc.* **121**, 2147–2155.
Giralt, E., Eritja, R. & Pedroso, E. (1981). *Tetrahedron Lett.* **22**, 3779–3782.
Gisin, B. F. & Merrifield, R. B. (1972). *J. Am. Chem. Soc.* **94**, 3102–3106.
Khosla, M. C., Smeby, R. R. & Bumpus, F. (1972). *J. Am. Chem. Soc.* **94**, 4721–4724.
Li, Z. G. (2001). *Preparation of Organic Intermediates*, pp. 103–107. Beijing: Chemical Industry Press.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sanatamaria, A., Cabezas, N. & Avendano, C. (1999). *Tetrahedron*, **55**, 1173–1186.

Sheldrick, G. M. (1996). *SADABS.* University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXTL.* Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Siemens (1996). *SMART* and *SAINT.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.