Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Benzyl-5-(2,5-dioxo-2,5-dihydro-1*H*-pyrrol-1-yl)-2,3-dihydro-1*H*-isoindole-1,3-dione

Da-Xin Shi, Ya-Qing Feng,* Xiao-Fang Li and Li-Shan Zhou

School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: daxinmail@yahoo.com.cn

Received 18 June 2003
Accepted 25 June 2003
Online 30 June 2003

Key indicators

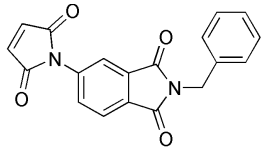
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.115
Data-to-parameter ratio = 11.9

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

The title compound, $C_{19}H_{12}N_2O_4$, was synthesized by dehydrative condensation of maleic anhydride and *N*-benzyl-4-aminophthalimide. In the structure, the maleimide ring is rotated by 43.8 (3)° with respect to the phthalimide plane. The dihedral angle between the mean planes of the phthalimide and benzyl groups is 91.0 (3)°.

Comment

N-Substituted maleimides are a class of organic compounds with numerous applications in synthetic and polymer chemistry. A search of the literature revealed that some *N*-substituted maleimides have important biological properties, such as antimicrobial activity (Zentz *et al.*, 2002), antibacterial activity (Filho *et al.*, 1994) and antitumor activity (Kratz *et al.*, 1997). Some phthalimide derivatives have cytotoxicity (Hall *et al.*, 1995) and anti-HIV activity (Van Derpoorten *et al.*, 1997). It was assumed that compounds having both phthalimide and maleimide residues in the same molecule may possess some interesting biological activities. With this in mind, the synthesis and structure determination of the title compound, (I), were undertaken.



(I)

The phthalimide group is planar, the mean deviation from the least-squares plane being 0.010 (3) Å. This observation is in good agreement with our previous report on ethyl *N*-(2-butyl-1,3-dioxo-2,3-dihydro-1*H*-isoindol-5-yl) carbamate (Shi *et al.*, 2003). The maleimide moiety is also planar, the mean deviation of the atoms from this plane being 0.008 (3) Å. The dihedral angle between the mean planes of the phthalimide plane and maleimide ring is 43.8 (3)°, which is somewhat different from the corresponding angles found in bis(4-maleimidophenyl)methane, *viz*. 52.1 (1)° (Usman *et al.*, 2003), and *N*-(4-hydroxyphenyl)maleimide, *viz*. 52.8 (5)° (Rodringuez *et al.*, 2002). The benzyl group and phthalimide group are folded towards each other, making an angle of 91.0 (2)°.

Experimental

To a stirred solution of maleic anhydride (22 mmol) in 20 ml of acetone was added dropwise a solution of *N*-benzyl-4-aminophthalimide (20 mmol) in 30 ml of acetone. After an addition time of 1 h, the maleamic acid separated almost immediately at room tempera-

ture. After stirring for 3 h to ensure complete reaction, 0.3 ml of triethylamine and 4 ml of acetic anhydride were added. The mixture was heated to 323 K and kept at this temperature for 1 h. Then the mixture was diluted with four times its volume of cold water, thereby precipitating (I) as fine white feathery needles. M.p. 426–428 K; IR (KBr): 3080 (C—H), 1730 (C═O) cm^{-1}; ^1H NMR ($CDCl_3$, p.p.m.): 4.86 (2H, *s*), 6.93 (2H, *s*), 7.26–7.43 (5H, *m*), 7.78 (1H, *d*), 7.92–7.95 (2H, *m*). Compound (I) (50 mg) was dissolved in chloroform (15 ml) and the solution was kept at room temperature for 3 d, yielding colorless single crystals.

Crystal data

$C_{19}H_{12}N_2O_4$
M_r = 332.31
Triclinic, $P\overline{1}$
a = 5.061 (2) Å
b = 11.803 (4) Å
c = 14.186 (5) Å
α = 112.607 (6)°
β = 94.050 (6)°
γ = 95.755 (6)°
V = 772.8 (5) Å^3
Z = 2
D_x = 1.428 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 997 reflections
θ = 3.1–26.3°
μ = 0.10 mm^{-1}
T = 293 (2) K
Plate, colorless
0.25 × 0.20 × 0.15 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.976, T_{max} = 0.985
3218 measured reflections
2700 independent reflections
2082 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 25.0°
$h = -5 \rightarrow 6$
$k = -10 \rightarrow 14$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.115
S = 1.06
2700 reflections
226 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0604P)^2 + 0.1101P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.207 (2)	N1—C5	1.427 (2)
O2—C4	1.201 (2)	N2—C13	1.465 (2)
O3—C10	1.216 (2)	C2—C3	1.310 (3)
O4—C9	1.203 (2)		
C1—N1—C4	109.52 (16)	C2—C3—C4	109.33 (19)
C10—N2—C9	112.12 (15)	C6—C5—N1	118.66 (16)
C10—N2—C13	123.58 (16)	N2—C13—C14	112.12 (15)
C4—N1—C5—C12	−42.0 (2)	N2—C13—C14—C19	−43.1 (3)
C1—N1—C5—C6	−43.3 (2)	N2—C13—C14—C15	137.13 (18)

All H atoms were placed in calculated positions, with C—H distances of 0.93 (sp^2) and 0.97 Å (sp^3), and included in the refinement in the riding-motion approximation, with $U_{iso} = 1.2U_{eq}$ of the carrier atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

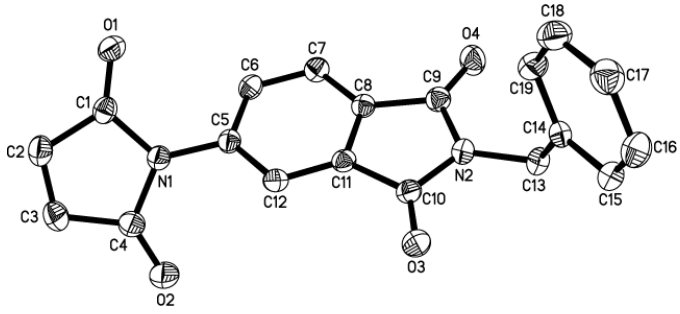


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted.

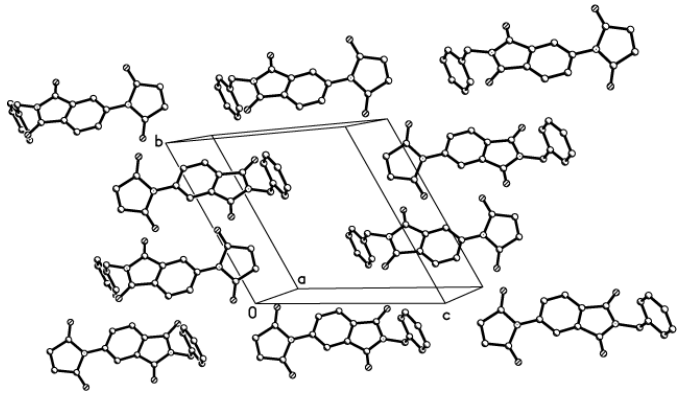


Figure 2
The crystal structure of (I), viewed approximately along the *a* axis.

References

Bruker (1998). *SMART, SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Filho, V. C., Pinheiro, T., Nunes, R. J., Yunes, R. A. & Cruz, A. B. (1994). *Farmaco*, **49**, 675–578.
Hall, I. H., Wong, O. T. & Scovill, J. P. (1995). *Biomed. Pharmcother*. **49**, 251–258.
Kratz, F., Beyer, U., Schumacher, P., Kruger, M., Zahn, H., Roth, T., Fiebig, H. H. & Unger, H. (1997). *Bioorg. Med. Chem. Lett.* **7**, 617–622.
Rodringuez, M. A., Aubert, J. H., McElhanon, J. R. & Eatough, M. O. (2002). *Acta Cryst.* E**58**, o742–o744.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Shi, D.-X., Feng, Y.-Q. & Li, X.-F. (2003). *Acta Cryst.* E**59**, o739–o741.
Usman, A., Fun, H.-K., Zhu, H.-L. & Ke, G. (2003). *Acta Cryst.* E**59**, o652–o653.
Van Derpoorten, K., Balzarini, J., De Clercq, E. & Poupaert, J. H. (1997). *Biomed. Pharmcother*. **51**, 464–468.
Zentz, F., Valla, A., Le Guillou, R., Labia, R., Mathot, A.-G. & Sirot, D. (2002). *Farmaco*, **57**, 421–426.