Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Isopropyl 2-bromo-4-(*N*-phthalimido)butanoate

Janet M. S. Skakle,[a]* Gregory Smyth[b] and Solange M. S. V. Wardell[c]

[a]Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland, [b]School of Physics, University of Aberdeen, Aberdeen AB24 3UE, Scotland, and [c]Instituto de Quimica, Departamento de Quimica Organica, Universidade Federal Fluminense, 24020-150 Niteroi, RJ, Brazil

Correspondence e-mail: j.skakle@abdn.ac.uk

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.013 Å
R factor = 0.073
wR factor = 0.196
Data-to-parameter ratio = 22.4

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

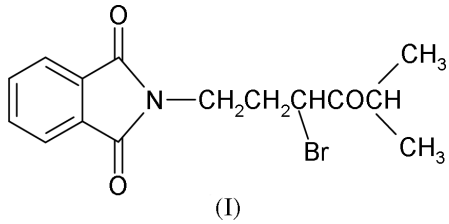
Received 22 February 2001
Accepted 27 February 2001
Online 9 March 2001

Molecules of the title compound, $C_{15}H_{16}BrNO_4$, are hydrogen bonded to form layers perpendicular to [100] and form enantiomeric pairs related by the *c*-glide $(-x+\frac{1}{2}, y, z-\frac{1}{2})$.

Comment

The racemic title compound, (I), was required as an alkylating agent for oxaaza-macrocycles (Kuksa *et al.*, 2000; Wardell & Lin, 1998).

The polar structure of (I) (Fig. 1) consists of alternating pairs of layers made up from one of the two enantiomers. Each layer is formed by one enantiomer linked *via* C11—H11···O3^{i} hydrogen bonds [symmetry code: (i) $x, y-1, z$]; thus, the layers form normal to [100] (Fig. 2). The distance between adjacent carbon rings within each layer is 3.294 (8) Å; hence, there is π–π bonding within the layers. In addition, there is an C9—H9···Br1 intramolecular hydrogen bond.



(I)

The layers form pairs between the two enantiomers where the enantiomers are related by the the symmetry operation $(-x+\frac{1}{2}, y, z-\frac{1}{2})$ (Fig. 3).

Examination of the structure with *PLATON* (Spek, 2000) showed that there were no solvent-accessible voids within the crystal lattice.

Experimental

The title compound was prepared according to a published procedure (Kruper *et al.*, 1993). Purification was achieved *via* column chromatogaphy on silica gel using chloroform as eluent with recrystallization from ethanol (m.p. 344–346 K; literature m.p. 345–347.5 K).

Crystal data

$C_{15}H_{16}BrNO_4$
M_r = 354.20
Orthorhombic, $Pca2_1$
a = 27.307 (2) Å
b = 4.6228 (4) Å
c = 12.5888 (11) Å
V = 1589.2 (2) Å^3
Z = 4
D_x = 1.480 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2380 reflections
θ = 2.2–25.8°
μ = 2.60 mm^{-1}
T = 296 (2) K
Block, colourless
0.50 × 0.50 × 0.40 mm

Data collection

Bruker SMART 1000 area CCD detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*: Bruker, 1999)
T_{min} = 0.356, T_{max} = 0.423
11 746 measured reflections
4189 independent reflections
2201 reflections with $I > 2\sigma(I)$
R_{int} = 0.082
θ_{max} = 31.1°
$h = -31 \rightarrow 39$
$k = -6 \rightarrow 6$
$l = -14 \rightarrow 17$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.196
S = 1.05
4189 reflections
187 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0921P)^2 + 0.1679P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.91 e Å^{-3}
Absolute structure: (Flack, 1983)
Flack parameter = 0.06 (2)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···Br1	0.97	2.84	3.299 (7)	110
C11—H11···O3^{i}	0.98	2.58	3.353 (9)	136

Symmetry code: (i) $x, y-1, z$.

H atoms were placed in geometrical positions and refined using a riding model using the AFIX commands of *SHELX*.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEX* in *OSCAIL* (McArdle, 1994, 2000) and *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *CIFTAB* (Sheldrick, 1997).

We acknowledge the use of the EPSRC's Chemical Database Service at Daresbury (Fletcher *et al.*, 1996).

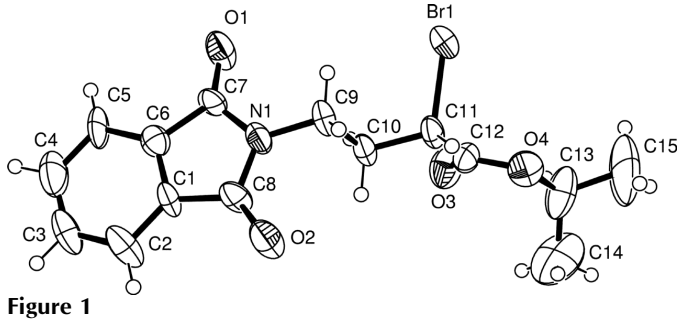


Figure 1
The asymmetric unit of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probablity level.

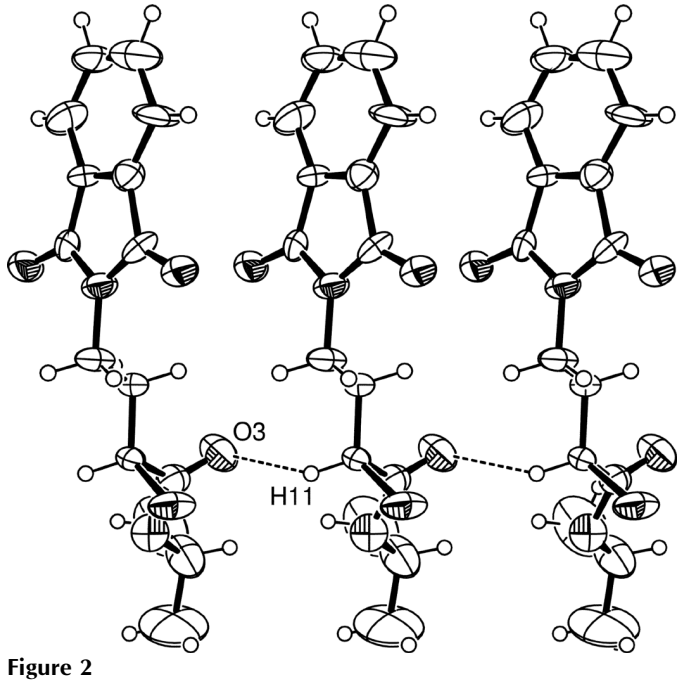


Figure 2
Layers formed by hydrogen bonding of successive layers along *b*.

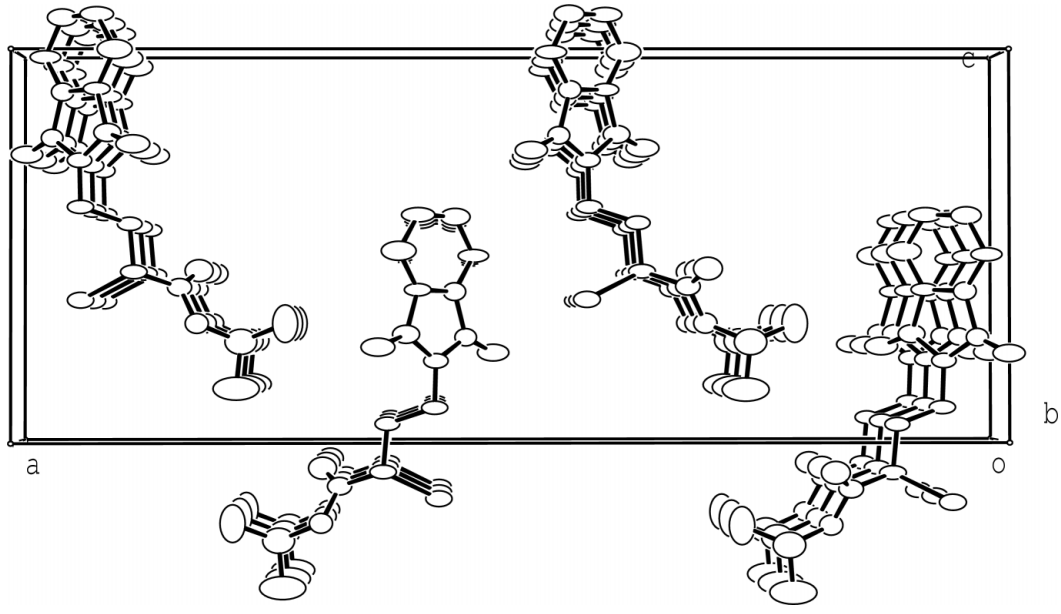


Figure 3
Relative orientation of layers within the unit cell, viewed normal to (010).

References

Bruker (1999). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.

Kruper, E. J. Jr, Rudolf, P. R. & Langhoff, C. A. (1993). *J. Org. Chem.* **58**, 3869–3876.

Kuksa, V. A., Wardell, S. M. S. V. & Lin, P. K. T. (2000). *Inorg. Chem. Commun.* **3**, 267–270.

McArdle, P. (1994). *J. Appl. Cryst.* **27**, 438–439.

McArdle, P. (2000). *OSCAIL for Windows*. National University of Ireland, Galway, Ireland.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97 and *CIFTAB*. University of Göttingen, Germany.

Spek, A. L. (2000). *PLATON*. May 2000 version. University of Utrecht, The Netherlands.

Wardell, S. M. S. V. & Lin, P. K. T. (1998). *J. Chem. Crystallogr.* **28**, 683–687.