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1-Methylethyl 2-[(1-bromo-2-naphthyl)oxy]ethanoate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.007 Å
R factor = 0.042
wR factor = 0.112
Data-to-parameter ratio = 16.0

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

Molecules of the title compound, $C_{15}H_{15}BrO_3$, form C—H$\cdots\pi$ interactions leading to an infinite chain parallel to the [010] direction.

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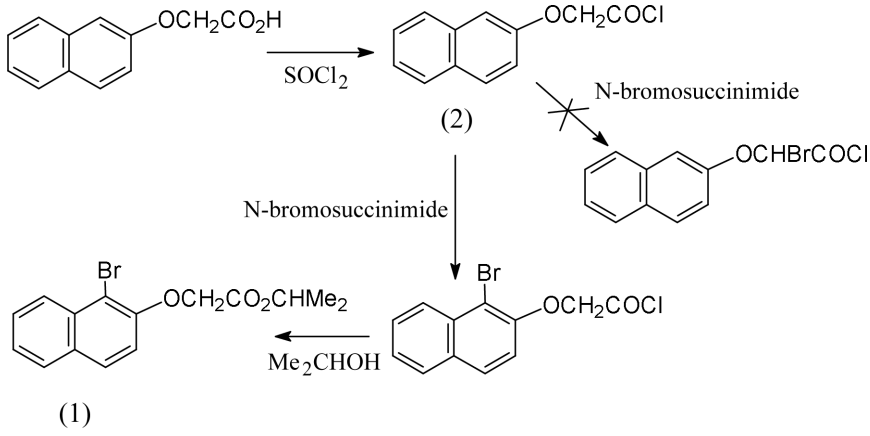
Comment

The title compound, (1), was obtained by the reaction sequence shown in the scheme below. Of particular interest in this scheme were the products of reaction of (2) with N-bromosuccinimide and the competition between bromination of the aromatic ring and the methylene group, α to the carbonyl group. NMR spectroscopy of the final reaction mixture clearly showed that reaction occurred completely in the aromatic ring: no indications for 2-bromo-2-(2-naphthyloxy)ethanoic acid were found.



(1)

The asymmetric unit of (1) is shown in Fig. 1 with the numbering scheme; intramolecular C—H$\cdots$Br interactions are present (C5—H5$\cdots$Br1, Table 1) although not shown in this diagram.

Molecules of (1) are linked by C—H$\cdots\pi$ interactions, *viz*. C4—H4$\cdots\pi$. The C1–C6 ring is the acceptor at ($2-x$, $-\frac{1}{2}+y$, $-z$), with a C4–centroid distance of 4.146 (7) Å. These are represented in Fig. 2, which shows the zigzag of the resultant chain parallel to the [010] direction (Spek, 2001).

Experimental

A solution of 2-naphthyloxyethanoic acid (5.06 g, 0.025 mol), prepared according to a published procedure (Howie *et al.*, 2001), and thionyl chloride (7.5 ml, 0.1 mol) in $CHCl_3$ (10 ml) was refluxed for 1.5 h and cooled. A mixture of *N*-bromosuccinimide (4.45 g, 0.025 mol) and aqueous HBr (48%, 2 drops) in CH_2Cl_2 (10 ml) was added, the mixture refluxed for 2 h, cooled, poured on to cold 2-

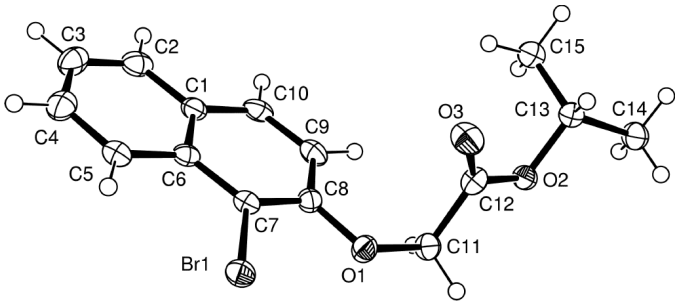


Figure 1
Asymmetric unit of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

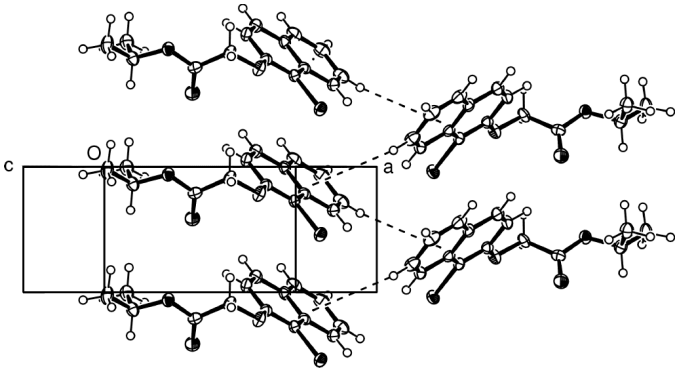


Figure 2
Molecules of the title compound related by $(2 - x, y - \frac{1}{2}, -z)$, illustrating C—H$\cdots\pi$ intermolecular interactions. The unit cell is shown, normal to (001).

propanol (50 ml) with stirring, and left for 2 h. The solution was rotary evaporated to leave a colourless residue, which was separated by chromatography on silica. 1-Methylethyl 2-[(1-bromo-2-naphthyl)oxy]ethanoate was recrystallized from 2-propanol; 2.2 g, m.p. 331–332 K. Crystals for the X-ray study were grown by slow evaporation of a 2-propanol solution. 1H NMR (400 MHz, $CDCl_3$, p.p.m.): δ 1.23 (*d*, 6H, *J* = 6.1 Hz, Me), 4.76 (*s*, 2H, CH_2), 5.11 (*sept*, *J* = 6.1 Hz, CH), 7.14 (*d*, 1H, *J* = 9.2hz), 7.39 (*ddd*, 1H, *J* = 1.0, 6.8, *ca* 8 Hz), 7.54 (*ddd*, 1H, *J* = 1.4, 6.8, 8.6 Hz), 7.65 (*d*, 2H, *J* = 8.6 Hz), 8.11 (*dd*, 1H, *J* = 1.0, 8.6 Hz). IR (KBr, cm^{-1}): 2980, 1736, 1624, 1597, 1504, 1464, 1445, 1374, 1383, 1287, 1209, 1178, 1096, 936, 906, 804, 765, 744,709, 641, 596, 518, 414.

Crystal data

$C_{15}H_{15}BrO_3$
M_r = 323.18
Monoclinic, $P2_1$
a = 10.3369 (4) Å
b = 4.7557 (2) Å
c = 14.2208 (7) Å
β = 102.4951 (18)°
V = 682.52 (5) Å^3
Z = 2
D_x = 1.573 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 4552 reflections
θ = 1.0–27.5°
μ = 3.01 mm^{-1}
T = 120 (2) K
Plate, colourless
0.35 × 0.08 × 0.02 mm

Data collection

Enraf–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: empirical (*SORTAV*; Blessing, 1995, 1997)
T_{min} = 0.633, T_{max} = 0.633
4741 measured reflections
2792 independent reflections
2507 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 27.5°
h = −13 → 13
k = −6 → 5
l = −16 → 18

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.112
S = 1.08
2792 reflections
174 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.5450P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.79 e Å^{-3}
$\Delta\rho_{min}$ = −0.96 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.015 (16), 1058 Friedel pairs

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C5—H5$\cdots$Br1	0.95	2.76	3.182 (5)	108

H atoms were placed in geometrical positions and refined using a riding model.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEX* in *OSCAIL* (McArdle, 1994, 2000) and *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *CIFTAB* (Sheldrick, 1997).

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References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–37.
Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565–565.
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.
Howie, R. A., Skakle, J. M. S. & Wardell, S. M. S. V. (2001). *Acta Cryst.* E**57**, o72–o74.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
McArdle, P. (1994). *J. Appl. Cryst.* **27**, 438–439.
McArdle, P. (2000). *OSCAIL for Windows*. National University of Ireland, Galway, Ireland.
Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.
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. (2001). *PLATON*. January 2001 version. University of Utrecht, The Netherlands.