Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

N-(2-Bromophenyl)propionamide

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The title compound, $C_9H_{10}BrNO$, posseses normal geometrical parameters. The crystal packing is influenced by an N—H···O hydrogen bond.

Received 1 September 2005
Accepted 6 September 2005
Online 14 September 2005

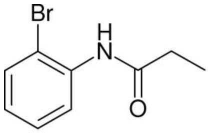
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.027
wR factor = 0.065
Data-to-parameter ratio = 18.4

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

Comment

The title compound, (I) (Fig. 1), was prepared as part of our ongoing studies of acetanilide derivatives (Ronaldson *et al.*, 2005).



(I)

The dihedral angle between the mean planes of the benzene ring (atoms C1–C6) and the N1/O1/C7/C8 side chain is 40.92 (10)°. This is very similar to the value [42.75 (14)°] in *N*-(2-bromophenyl)acetamide, (II) (Ronaldson *et al.*, 2005). The bond angle sum about N1 in (I) is 360.0°, indicating that this atom is sp^2 hybridized. The C6—N1 distance of 1.416 (3) Å in (I) is almost identical to the equivalent distance [1.418 (4) Å] in (II). All the other geometrical parameters for (I) lie within their expected ranges (Allen *et al.*, 1995).

The crystal packing in (I) is influenced by an N—H···O hydrogen bond (Table 1 and Fig. 2) that links the molecules into chains propagating in the [100] direction. There are no π–π interactions in (I). The unit-cell packing is shown in Fig. 3; (I) is essentially isostructural (same space group, similar unit-cell parameters) with (II), except that the unit cell for (I) is slightly expanded in the c-axis direction to accommodate the more bulky terminal ethyl moiety.

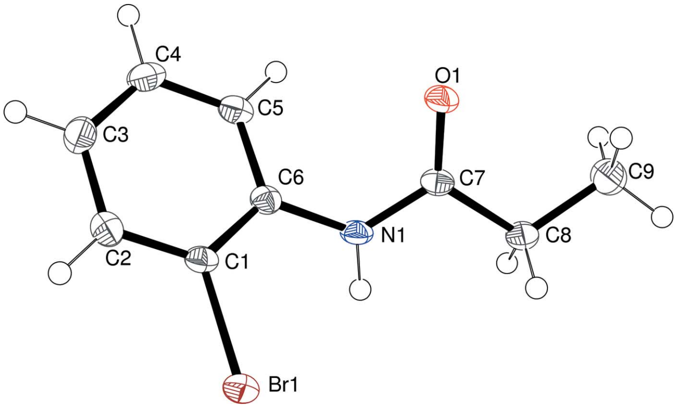


Figure 1
View of (I), shown with 50% displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radius.

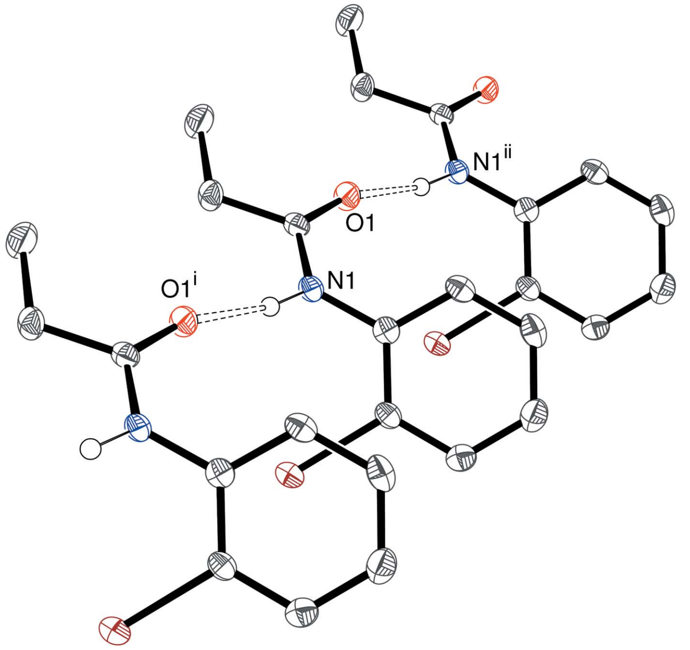


Figure 2
Detail of (I), showing how N—H···O hydrogen bonds (dashed lines) link molecules into a chain. The view direction is perpendicular to the mean plane of the benzene ring of the central molecule, showing that no π–π stacking occurs. All H atoms except H1 have been omitted for clarity. [Symmetry codes: (i) $x - 1, y, z$; (ii) $x + 1, y, z$.]

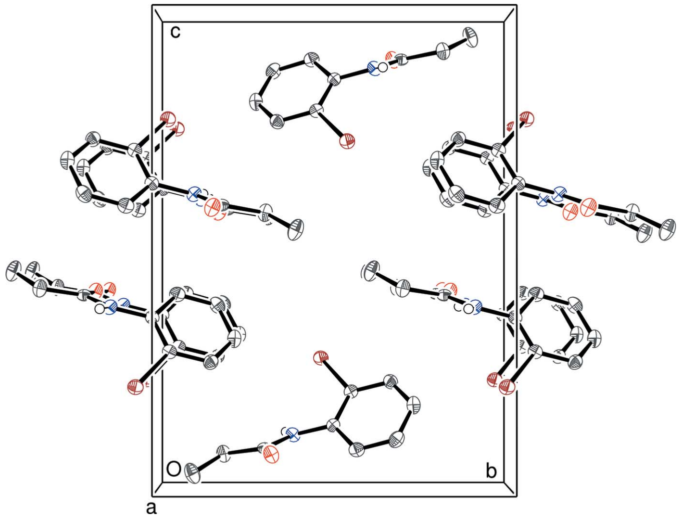


Figure 3
The packing in (I), viewed down [010], with all H atoms except H1 omitted for clarity.

Experimental

2-Bromoaniline (3.44 g, 20.0 mmol) was added to a solution of propionyl chloride (2.22 g, 24.0 mmol) and DIPEA (*N*,*N*-diisopropylethylamine) (3.12 g, 24.0 mmol) in dry tetrahydrofuran (20 ml) at 273 K. The reaction was monitored by thin-layer chromatography and, when complete, the mixture was diluted with water (20 ml) and the product was extracted with EtOAc (3 × 20 ml). The organic phase was washed with water (2 × 20 ml) and brine (20 ml) then dried over $MgSO_4$ and evaporated under reduced pressure to yield the crude product which was recrystallized from CH_2Cl_2 to give 4.31 g (95% yield) of (I) as colourless needles. M.p. 360–362 K; R_F = 0.19 [hexane/EtOAc (10:1)]; ν_{max} (KBr disc)/cm^{-1}: 3277 (NH), 2971–2932 (C—H), 1655 (C═O), 1573 (Ar C═C); δ_H (250 MHz, $CDCl_3$) 1.27 (3H, *t*, *J* = 7.5 Hz, CH_3), 2.46 (2H, *q*, *J* = 7.5 Hz, CH_2), 6.95 (1H, *t*, *J* = 7.5 Hz, Ar—H), 7.29 (1H, *t*, *J* = 8.0 Hz, Ar—H), 7.51 (1H, *d*, *J* = 8.0 Hz, Ar—H), 8.35 (1H, *d*, *J* = 7.5 Hz, Ar—H); δ_C ($CDCl_3$) 9.6 (CH_3), 31.1 (—CO–CH_2—), 113.2, 121.9, 125.0, 128.4, 132.2 and 135.7 (Ar–C), 172.0 (C═O). $[M+H]^+$ 226.994, $C_9H_{10}{}^{79}BrNO$ requires 226.995.

Crystal data

$C_9H_{10}BrNO$
M_r = 228.09
Monoclinic, $P2_1/n$
a = 4.8701 (1) Å
b = 11.8048 (4) Å
c = 15.9296 (5) Å
β = 93.964 (2)°
V = 913.61 (5) Å^3
Z = 4
D_x = 1.658 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2165 reflections
θ = 2.9–27.5°
μ = 4.45 mm^{-1}
T = 120 (2) K
Needle, colourless
0.36 × 0.05 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.297, T_{max} = 0.916
13742 measured reflections
2092 independent reflections
1822 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 27.5°
$h = -6 \rightarrow 6$
$k = -15 \rightarrow 15$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.065
S = 1.07
2092 reflections
114 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0226P)^2 + 0.9463P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0039 (9)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.85 (3)	2.06 (3)	2.889 (2)	164 (2)

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

All C-bound H atoms were placed in idealized positions (C—H = 0.95–0.99 Å) and refined as riding on their carriers with the constraint U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(methyl C) applied. The methyl group was allowed to rotate about the C9—C8 bond as a rigid group. The N-bound H atom was located in a difference map and its position was freely refned with the constraint U_{iso}(H) = 1.2U_{eq}(N).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997), and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Acta Cryst. (2005). E**61**, o3200–o3202 Ronaldson *et al.* • $C_9H_{10}BrNO$ **o3201**

The authors thank the EPSRC National Mass Spectrometry Service (University of Swansea) and the EPSRC National Crystallography Service (University of Southampton) for data collections.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1995). *International Tables for Crystallography*, Vol. C, Section 9.5, pp. 685–706. Dordrecht: Kluwer Academic Publishers.

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.

Bruker (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

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

Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Ronaldson, V., Storey, J. M. D. & Harrison, W. T. A. (2005). *Acta Cryst.* E**61**, o3156–o3158.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

o3202 Ronaldson *et al.* • $C_9H_{10}BrNO$ *Acta Cryst.* (2005). E**61**, o3200–o3202

supporting information

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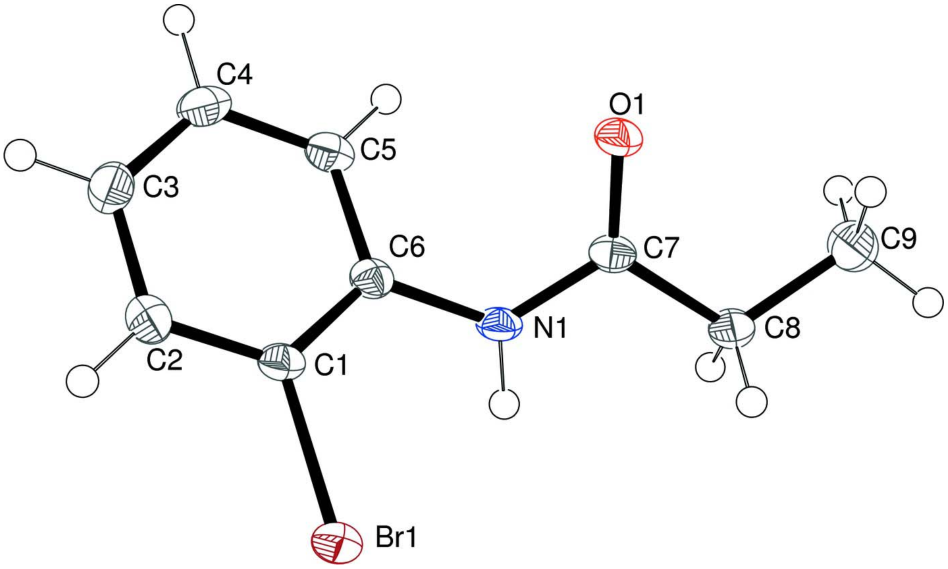
All C-bound H atoms were placed in idealized positions (C—H = 0.95–0.99 Å) and refined as riding on their carriers with the constraint $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}$(methyl C) applied. The methyl group was allowed to rotate about the C9—C8 bond as a rigid group. The N-bound H atom was located in a difference map and its position was freely refned with the constraint $U_{iso}(H) = 1.2U_{eq}(N)$.



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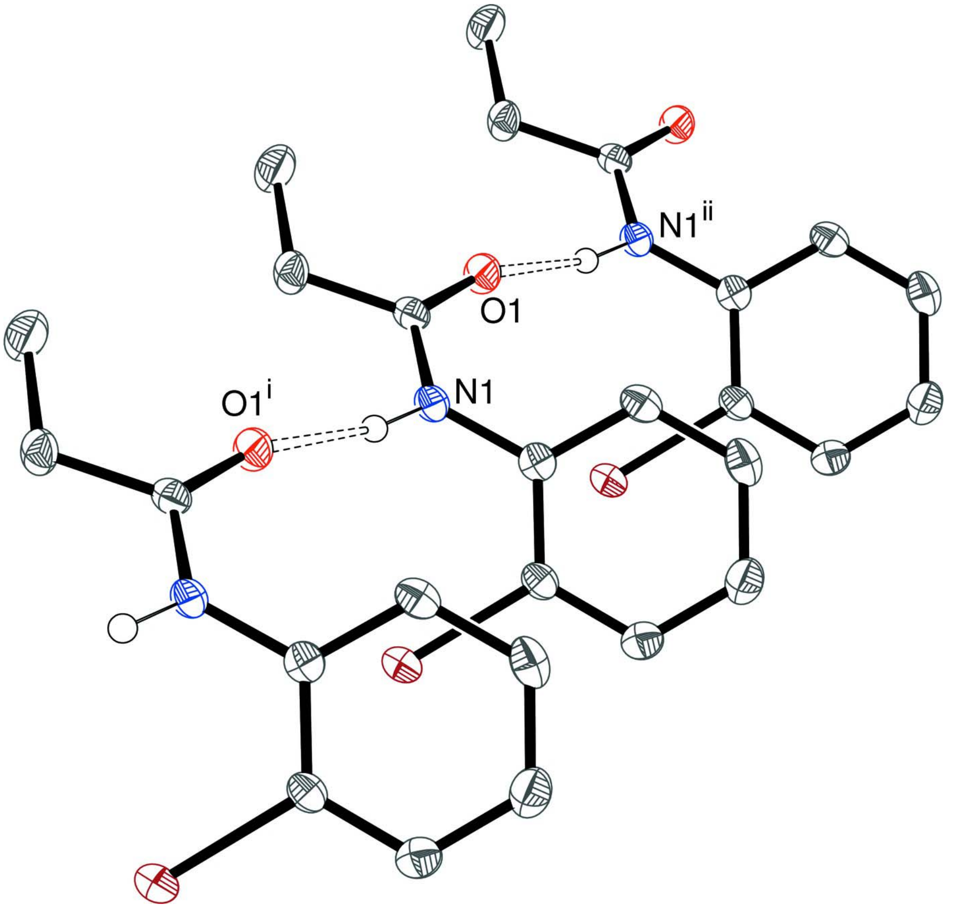

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Detail of (I), showing how N—H···O hydrogen bonds (dashed lines) link molecules into a chain. The view direction is perpendicular to the best plane of the benzene ring of the central molecule, showing that no *π*–*π* stacking occurs. All H atoms except H1 have been omitted for clarity. [Symmetry codes: (i) *x* − 1, *y*, *z*; (ii) *x* + 1, *y*, *z*.]

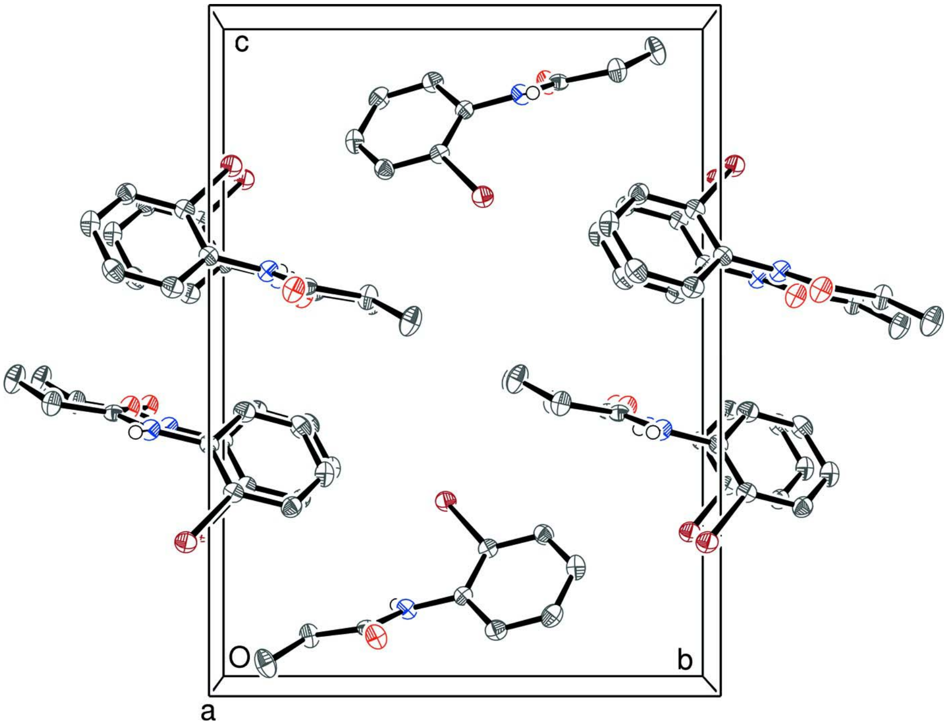

Figure 3

Unit-cell packing in (I), viewed down [010], with all H atoms except H1 omitted for clarity.

N-(2-Bromophenyl)propionamide

Crystal data

$C_9H_{10}BrNO$	$F(000) = 456$
$M_r = 228.09$	$D_x = 1.658$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 2165 reflections
$a = 4.8701$ (1) Å	$\theta = 2.9$–$27.5°$
$b = 11.8048$ (4) Å	$\mu = 4.45$ mm^{-1}
$c = 15.9296$ (5) Å	$T = 120$ K
$\beta = 93.964$ (2)°	Needle, colourless
$V = 913.61$ (5) Å^3	0.36 × 0.05 × 0.02 mm
$Z = 4$	

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Nonius KappaCCD diffractometer	13742 measured reflections
Radiation source: fine-focus sealed tube	2092 independent reflections
Graphite monochromator	1822 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.043$
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)	$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
	$h = -6\rightarrow6$
	$k = -15\rightarrow15$
$T_{min} = 0.297$, $T_{max} = 0.916$	$l = -20\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.065$
$S = 1.07$
2092 reflections
114 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0226P)^2 + 0.9463P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.38$ e Å^{-3}
$\Delta\rho_{\min} = -0.37$ e Å^{-3}
Extinction correction: *SHELXL97*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0039 (9)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.4616 (5)	0.54914 (19)	0.20494 (14)	0.0194 (5)
C2	0.5180 (5)	0.6609 (2)	0.22655 (15)	0.0237 (5)
H2	0.4342	0.6949	0.2724	0.028*
C3	0.6975 (5)	0.7226 (2)	0.18071 (16)	0.0273 (5)
H3	0.7417	0.7985	0.1960	0.033*
C4	0.8129 (5)	0.6731 (2)	0.11218 (16)	0.0260 (5)
H4	0.9370	0.7153	0.0809	0.031*
C5	0.7481 (5)	0.5631 (2)	0.08939 (15)	0.0236 (5)
H5	0.8228	0.5311	0.0412	0.028*
C6	0.5740 (4)	0.4983 (2)	0.13620 (14)	0.0186 (5)
C7	0.6917 (4)	0.3057 (2)	0.09094 (13)	0.0186 (5)
C8	0.5718 (5)	0.1890 (2)	0.07664 (16)	0.0238 (5)
H8A	0.4053	0.1950	0.0376	0.029*
H8B	0.5139	0.1594	0.1308	0.029*
C9	0.7679 (5)	0.1053 (2)	0.04102 (19)	0.0334 (6)
H9A	0.6908	0.0288	0.0435	0.050*
H9B	0.9451	0.1080	0.0741	0.050*
H9C	0.7949	0.1249	−0.0176	0.050*
N1	0.5104 (4)	0.38443 (17)	0.11491 (12)	0.0199 (4)
H1	0.342 (6)	0.367 (2)	0.1167 (16)	0.024*
O1	0.9361 (3)	0.32624 (14)	0.08417 (11)	0.0241 (4)
Br1	0.23270 (5)	0.46366 (2)	0.273034 (14)	0.02474 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0135 (10)	0.0232 (12)	0.0217 (11)	−0.0002 (9)	0.0026 (8)	0.0011 (9)
C2	0.0237 (12)	0.0234 (12)	0.0243 (12)	0.0024 (9)	0.0030 (9)	−0.0022 (10)
C3	0.0258 (13)	0.0202 (12)	0.0357 (14)	−0.0008 (10)	0.0011 (10)	−0.0007 (11)
C4	0.0195 (12)	0.0277 (13)	0.0311 (13)	−0.0039 (10)	0.0034 (10)	0.0064 (10)
C5	0.0195 (12)	0.0279 (13)	0.0242 (12)	0.0007 (9)	0.0064 (9)	0.0003 (10)
C6	0.0138 (10)	0.0211 (11)	0.0205 (11)	0.0014 (9)	−0.0008 (9)	0.0013 (9)
C7	0.0162 (11)	0.0244 (12)	0.0152 (10)	−0.0014 (9)	0.0017 (8)	−0.0003 (9)
C8	0.0149 (11)	0.0229 (12)	0.0336 (13)	−0.0034 (9)	0.0019 (9)	−0.0025 (10)
C9	0.0265 (13)	0.0269 (13)	0.0472 (16)	−0.0046 (11)	0.0046 (12)	−0.0115 (12)
N1	0.0119 (9)	0.0239 (10)	0.0242 (10)	−0.0033 (8)	0.0038 (7)	−0.0028 (8)
O1	0.0142 (8)	0.0249 (9)	0.0334 (9)	−0.0022 (6)	0.0043 (7)	−0.0052 (7)
Br1	0.02460 (16)	0.02600 (15)	0.02478 (16)	−0.00150 (9)	0.01004 (10)	−0.00078 (10)

Geometric parameters (Å, º)

C1—C2	1.386 (3)	C6—N1	1.416 (3)
C1—C6	1.393 (3)	C7—O1	1.227 (3)
C1—Br1	1.899 (2)	C7—N1	1.355 (3)
C2—C3	1.384 (3)	C7—C8	1.507 (3)
C2—H2	0.9500	C8—C9	1.512 (3)
C3—C4	1.390 (4)	C8—H8A	0.9900
C3—H3	0.9500	C8—H8B	0.9900
C4—C5	1.379 (3)	C9—H9A	0.9800
C4—H4	0.9500	C9—H9B	0.9800
C5—C6	1.395 (3)	C9—H9C	0.9800
C5—H5	0.9500	N1—H1	0.85 (3)

C2—C1—C6	121.5 (2)	O1—C7—N1	122.9 (2)
C2—C1—Br1	118.57 (17)	O1—C7—C8	122.4 (2)
C6—C1—Br1	119.90 (17)	N1—C7—C8	114.60 (19)
C3—C2—C1	119.4 (2)	C7—C8—C9	113.92 (19)
C3—C2—H2	120.3	C7—C8—H8A	108.8
C1—C2—H2	120.3	C9—C8—H8A	108.8
C2—C3—C4	119.9 (2)	C7—C8—H8B	108.8
C2—C3—H3	120.1	C9—C8—H8B	108.8
C4—C3—H3	120.1	H8A—C8—H8B	107.7
C5—C4—C3	120.3 (2)	C8—C9—H9A	109.5
C5—C4—H4	119.9	C8—C9—H9B	109.5
C3—C4—H4	119.9	H9A—C9—H9B	109.5
C4—C5—C6	120.8 (2)	C8—C9—H9C	109.5
C4—C5—H5	119.6	H9A—C9—H9C	109.5
C6—C5—H5	119.6	H9B—C9—H9C	109.5
C1—C6—C5	118.1 (2)	C7—N1—C6	125.68 (19)
C1—C6—N1	120.4 (2)	C7—N1—H1	119.4 (18)
C5—C6—N1	121.6 (2)	C6—N1—H1	114.9 (18)

C6—C1—C2—C3	−2.3 (4)	C4—C5—C6—C1	1.9 (4)
Br1—C1—C2—C3	176.17 (18)	C4—C5—C6—N1	−178.5 (2)
C1—C2—C3—C4	1.9 (4)	O1—C7—C8—C9	−8.7 (3)
C2—C3—C4—C5	0.4 (4)	N1—C7—C8—C9	173.8 (2)
C3—C4—C5—C6	−2.3 (4)	O1—C7—N1—C6	−0.9 (4)
C2—C1—C6—C5	0.4 (4)	C8—C7—N1—C6	176.7 (2)
Br1—C1—C6—C5	−178.01 (17)	C1—C6—N1—C7	−138.0 (2)
C2—C1—C6—N1	−179.3 (2)	C5—C6—N1—C7	42.4 (3)
Br1—C1—C6—N1	2.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1[i]	0.85 (3)	2.06 (3)	2.889 (2)	164 (2)

Symmetry code: (i) *x*−1, *y*, *z*.