

N-(2-Bromophenyl)acetamide

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Key indicators

Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.032
 wR factor = 0.074
Data-to-parameter ratio = 18.0

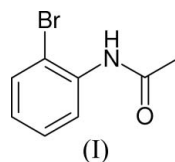
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_8\text{H}_8\text{BrNO}$, possesses normal geometrical parameters. The crystal packing is influenced by an intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond.

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Comment

The title compound, $\text{C}_8\text{H}_8\text{BrNO}$, (I), (Fig. 1) was prepared as an intermediate in a natural product synthesis.



The dihedral angle between the mean planes of the benzene ring (atoms C1–C6) and the N1/O1/C7/C8 side-chain grouping in (I) is 42.75 (14)°. This is intermediate between the situation in acetanilide [*i.e.* (I) without the Br atom], $\text{C}_8\text{H}_9\text{NO}$ (Brown, 1966; Wasserman *et al.*, 1985), where the aromatic ring and side chain are twisted by 17.6° , and *N*-methylacetanilide, $\text{C}_9\text{H}_{11}\text{NO}$ (Pederson, 1967), where the two corresponding groups of atoms are constrained by symmetry to be perpendicular. The $\text{C}_{\text{ar}}-\text{N}$ (ar = aromatic) bond distances are almost identical in (I) and acetanilide (Brown, 1966), being 1.418 (4) and 1.417 (2) Å respectively, as are the $\text{C}_{\text{c}}-\text{N}$ (c = carbonyl) distances, at 1.358 (4) and 1.355 (2) Å, respectively. The equivalent distances in *N*-methylacetanilide (Pederson, 1967), where any electronic conjugation between the benzene ring and amide group is presumably impossible because of their perpendicular orientation, are distinctly different, with $\text{C}_{\text{ar}}-\text{N}$ much longer at 1.474 Å and $\text{C}_{\text{c}}-\text{N}$ significantly shorter at 1.325 Å.

The bond angle sum about N1 in (I) is 360.0° , suggesting that this atom is essentially sp^2 -hybridized. 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 $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond (Table 1 and Fig. 2) that links the molecules into chains propagating along [100]. There are no $\pi-\pi$ stacking interactions in (I). The packing is shown in Fig. 3.

Experimental

2-Bromoaniline (3.44 g, 20.0 mmol) was added to a solution of acetyl chloride (1.88 g, 24.0 mmol) and DIPEA (*N,N*-diisopropylethylamine) (3.12 g, 24.0 mmol) in dry tetrahydrofuran (20 ml) at 273 K. On completion (as monitored by thin-layer chromatography), the reaction mixture was diluted with water (20 ml) and the product was

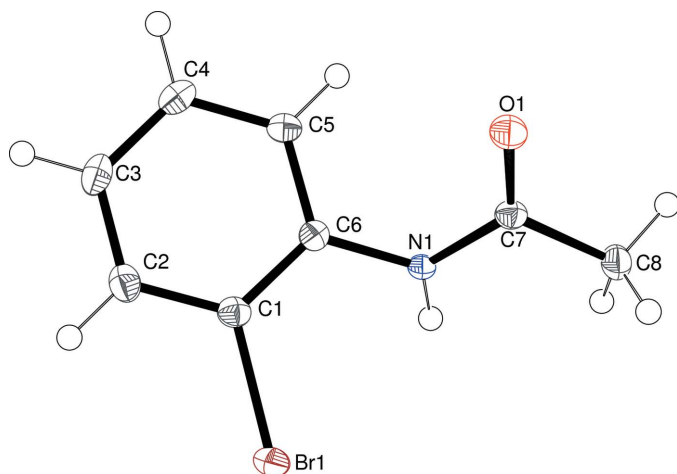


Figure 1
View of (I) (50% probability displacement ellipsoids; H atoms are drawn as small spheres of arbitrary radii).

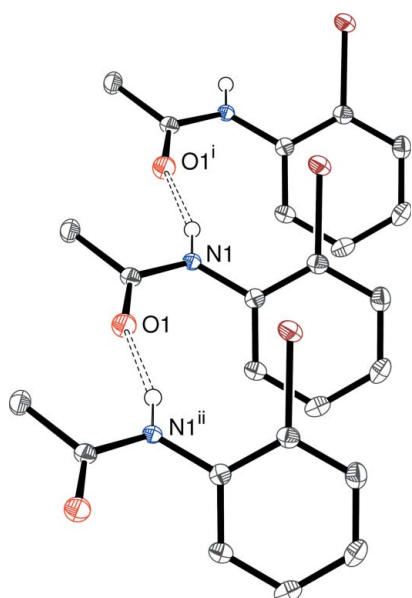


Figure 2
Detail of (I) showing how the N–H...O hydrogen bond (dashed lines) links molecules into a chain. The view direction is perpendicular to the mean plane of the benzene ring of the central molecule, showing that no π – π stacking occurs. All H atoms except atom H1 and its symmetry equivalents have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

extracted with EtOAc (3 \times 20 ml). The organic phase was then washed with water (2 \times 20 ml) and brine (20 ml), then dried (MgSO₄) and evaporated under reduced pressure to yield the crude product, which was recrystallized from CH₂Cl₂ to give (I) (yield 97%, 4.13 g) as clear needles; one of these was cut to a block for data collection; m.p. 363–364 K; $R_F = 0.12$ [hexane/EtOAc (5:1)]. IR (KBr disc, cm⁻¹): ν_{\max} 3272 (NH), 3159 (Ar–H), 1647 (C=O), 1518 (Ar C=C); ¹H NMR (250 MHz; CDCl₃): δ_H 2.21 (3H, s, CH₃), 6.97 (1H, t, $J = 7.5$ Hz, Ar–H), 7.29 (1H, t, $J = 7.5$ Hz, Ar–H), 7.51 (1H, d, $J = 8.0$ Hz, Ar–H), 7.61 (1H, bs, NH), 8.31 (1H, d, $J = 7.5$ Hz, Ar–H); ¹³C NMR (CDCl₃): δ_C 24.9 (–COCH₃), 113.2 (Ar C–Br), 122.0, 125.2, 128.4, 132.2 (4 \times Ar C), 135.7 (–CO–NH–C–), 168.3 (–C=O). Mass spectrum: $[M+H]^+$ 212.979, C₈H₈BrNO requires 212.979.

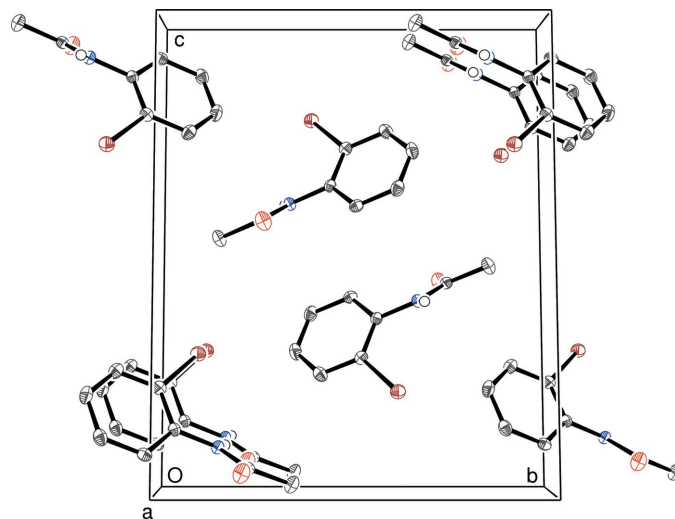


Figure 3
The packing in (I), viewed down [100], with all H atoms except H1 omitted for clarity.

Crystal data

C₈H₈BrNO
 $M_r = 214.06$
Monoclinic, $P2_1/n$
 $a = 4.7790$ (1) Å
 $b = 11.9257$ (4) Å
 $c = 14.6703$ (3) Å
 $\beta = 96.8173$ (16)°
 $V = 830.19$ (4) Å³
 $Z = 4$

$D_x = 1.713$ Mg m⁻³
Mo K α radiation
Cell parameters from 1977 reflections
 $\theta = 2.9$ – 27.5°
 $\mu = 4.89$ mm⁻¹
 $T = 120$ (2) K
Block, colourless
0.24 \times 0.10 \times 0.07 mm

Data collection

Nonius KappaCCD diffractometer
 ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2003)
 $T_{\min} = 0.387$, $T_{\max} = 0.726$
9212 measured reflections
1905 independent reflections

1750 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$
 $\theta_{\max} = 27.5^\circ$
 $h = -6 \rightarrow 5$
 $k = -15 \rightarrow 15$
 $l = -18 \rightarrow 19$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.074$
 $S = 1.26$
1905 reflections
106 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + 1.9011P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.76$ e Å⁻³
 $\Delta\rho_{\min} = -0.38$ e Å⁻³
Extinction correction: SHELXL97
Extinction coefficient: 0.0163 (11)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1 ⁱ ...O1 ⁱ	0.83 (4)	2.10 (4)	2.896 (3)	161 (3)

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

All the C-bound H atoms were placed in idealized positions (C–H = 0.95–0.98 Å) and refined as riding on their carriers with the constraint $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl carrier})$ applied. The methyl group was allowed to rotate about the C7–C8 bond as a rigid group. The N-bound H atom was located in a

difference map and its position was freely refined with the constraint $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK*, *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: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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