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N-(4-Bromophenyl)acetamide: a new polymorph

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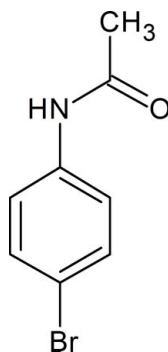
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 Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.033; wR factor = 0.073; data-to-parameter ratio = 26.6.

A new polymorph of the title compound, $\text{C}_8\text{H}_8\text{BrNO}$, has been determined at 173 K in the space group $P2_1/c$. The previous room-temperature structure was reported to crystallize in the orthorhombic space group $Pna2_1$ [Andreotti *et al.* (1968). *Acta Cryst.* **B24**, 1195–1198]. In the crystal, molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds forming chains along [010]. Weak $\text{C}-\text{H}\cdots\pi$ interactions are also present.

Related literature

For 2-arylacetamides, see: Mijin & Marinkovic (2006); Mijin *et al.* (2008) and for amides, see: Wu *et al.* (2008, 2010). For the structure of the orthorhombic polymorph, see: Andreotti *et al.* (1968). For related structures, see: Praveen *et al.* (2011a,b,c). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_8\text{H}_8\text{BrNO}$	$b = 9.3876$ (11) Å
$M_r = 214.06$	$c = 14.4434$ (14) Å
Monoclinic, $P2_1/c$	$\beta = 117.750$ (4)°
$a = 6.7250$ (7) Å	$V = 806.96$ (15) Å ³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 5.03$ mm⁻¹

$T = 173$ K
 $0.32 \times 0.22 \times 0.18$ mm

Data collection

Oxford Diffraction Xcalibur (Eos, Gemini) diffractometer	10902 measured reflections
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Oxford Diffraction 2010)	2689 independent reflections
$T_{\min} = 0.296$, $T_{\max} = 0.465$	2099 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$	101 parameters
$wR(F^2) = 0.073$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.54$ e Å ⁻³
2689 reflections	$\Delta\rho_{\text{min}} = -0.44$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C3–C8 ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^{\text{i}}$	0.89	2.00	2.885 (2)	174
$\text{C1}-\text{H1B}\cdots\text{Cg1}^{\text{ii}}$	0.98	2.84	3.761 (3)	157

 Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x + 1, y, z$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2566).

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supporting information

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***N*-(4-Bromophenyl)acetamide: a new polymorph**

Jerry P. Jasinski, Curtis J. Guild, H. S. Yathirajan, B. Narayana and S. Samshuddin

S1. Comment

Substituted 2-arylacetamides are very interesting compounds because of their structural similarity to the lateral chain of natural benzyl penicillin (Mijin *et al.*, 2006, 2008). Amides are also used as ligands due to their excellent coordination abilities (Wu *et al.*, 2008, 2010). The room temperature crystal structure of the title compound was reported without hydrogen atom coordinates in the orthorhombic space group $Pna2_1$ (Andreotti *et al.*, 1968). We report herein on the crystal structure of the monoclinic polymorph that crystallized in space group $P2_1/c$.

The molecular structure of the title compound is illustrated in Fig. 1. Bond lengths are in normal ranges (Allen *et al.*, 1987) but show slight changes from those reported for some similar acetamide derivatives *viz.*, *N*-(4-chloro-1,3-benzothiazol-2-yl)-2-(3-methylphenyl)acetamide monohydrate (Praveen *et al.*, 2011*a*), *N*-(3-chloro-4-fluorophenyl)-2,2-diphenylacetamide (Praveen *et al.*, 2011*b*) and *N*-(3-chloro-4-fluorophenyl)-2-(naphthalen-1-yl)acetamide (Praveen *et al.*, 2011*c*). The differences observed are primarily in the acetamide and bromophenyl regions [C1–C2 1.501 (3) Å *versus* 1.53 (4) Å; N1–C2 1.347 (2) Å *versus* 1.30 (3) Å; N1–C3 1.401 (2) Å *versus* 1.44 (3) Å and C6–Br 1.8907 (19) Å *versus* 1.91 (1) Å].

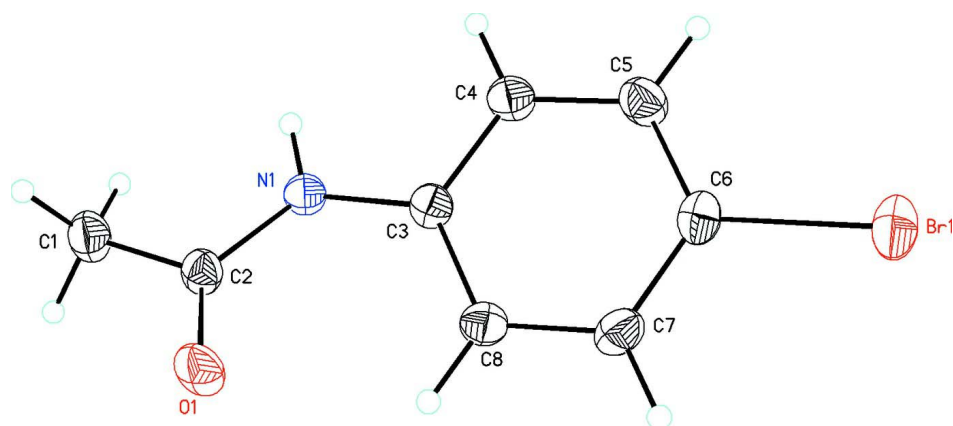
In the crystal, molecules are linked by N—H \cdots O hydrogen bonds forming chains along [010] [Table 1 and Fig. 2]. Weak C—H \cdots π interactions are also present (Table 1) and contribute to the crystal packing stability.

S2. Experimental

4-Bromo aniline (0.172 g, 1 mmol) was dissolved in acetic acid (20 ml) and refluxed for 4 h. The solution was then cooled and poured into 100 ml of ice-cold water with stirring. The precipitate obtained was filtered, washed with water and dried. Block-like yellow crystals were grown from a solution in ethyl acetate by slow evaporation of the solvent (M.p.: 430 K).

S3. Refinement

The NH H atom was located in a difference Fourier map and refined as a riding atom: N–H = 0.89 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. The C-bound H atoms were placed in calculated positions and refined as riding atoms: C—H = 0.95 Å (CH) and 0.98 Å (CH₃) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for CH H atoms and = $1.5U_{\text{eq}}(\text{C})$ for CH₃ H atoms.

**Figure 1**

The molecular structure of the title molecule, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

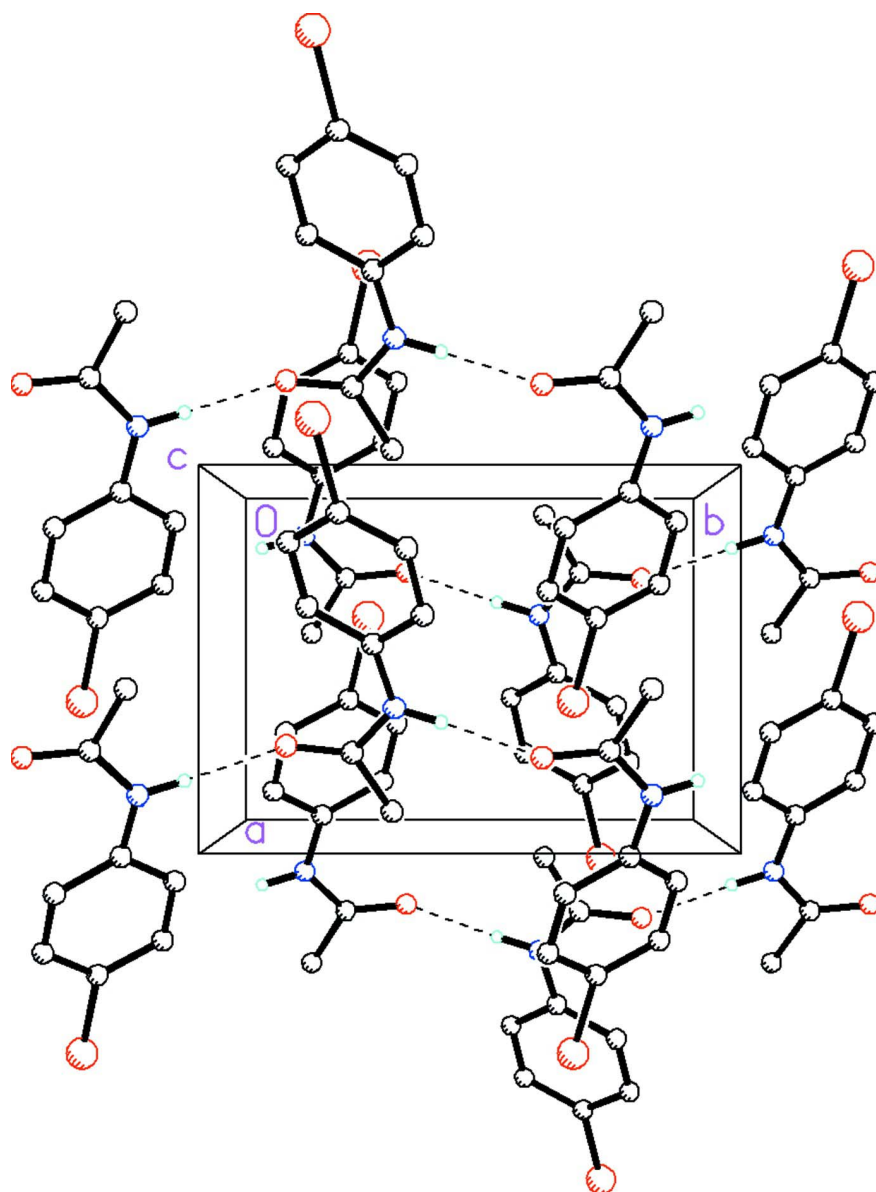


Figure 2

The crystal packing diagram of the title compound viewed along the *c* axis. Dashed lines indicate N—H···O hydrogen bonds linking the molecules into chains along [010]. C-bound H atoms have been omitted for clarity.

***N*-(4-Bromophenyl)acetamide**

Crystal data

C_8H_8BrNO

$M_r = 214.06$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 6.7250$ (7) Å

$b = 9.3876$ (11) Å

$c = 14.4434$ (14) Å

$\beta = 117.750$ (4)°

$V = 806.96$ (15) Å³

$Z = 4$

$F(000) = 424$

$D_x = 1.762$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3269 reflections

$\theta = 3.4$ – 32.3 °

$\mu = 5.03$ mm⁻¹

$T = 173$ K $0.32 \times 0.22 \times 0.18$ mm
 Block, yellow

Data collection

Oxford Diffraction Xcalibur (Eos, Gemini) diffractometer	10902 measured reflections
Radiation source: Enhance (Mo) X-ray Source	2689 independent reflections
Graphite monochromator	2099 reflections with $I > 2\sigma(I)$
Detector resolution: 16.1500 pixels mm ⁻¹	$R_{\text{int}} = 0.035$
ω scans	$\theta_{\text{max}} = 32.3^\circ$, $\theta_{\text{min}} = 3.4^\circ$
Absorption correction: multi-scan	$h = -9 \rightarrow 9$
(<i>CrysAlis PRO</i> ; Oxford Diffraction 2010)	$k = -13 \rightarrow 13$
$T_{\text{min}} = 0.296$, $T_{\text{max}} = 0.465$	$l = -20 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H-atom parameters constrained
$wR(F^2) = 0.073$	$w = 1/[\sigma^2(F_o^2) + (0.0329P)^2 + 0.1779P]$
$S = 1.04$	where $P = (F_o^2 + 2F_c^2)/3$
2689 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
101 parameters	$\Delta\rho_{\text{max}} = 0.54 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.44 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	-0.12616 (4)	0.70900 (2)	0.493749 (17)	0.03329 (8)
O1	0.4333 (2)	0.63844 (14)	0.19611 (11)	0.0286 (3)
N1	0.3872 (3)	0.85329 (15)	0.25584 (12)	0.0212 (3)
H1	0.4358	0.9432	0.2658	0.025*
C1	0.5678 (4)	0.8449 (2)	0.14665 (17)	0.0283 (4)
H1A	0.5162	0.8041	0.0767	0.042*
H1B	0.7312	0.8344	0.1869	0.042*
H1C	0.5281	0.9462	0.1404	0.042*
C2	0.4570 (3)	0.76819 (19)	0.20162 (15)	0.0213 (4)
C3	0.2696 (3)	0.81333 (18)	0.31030 (14)	0.0199 (3)
C4	0.2647 (3)	0.91062 (19)	0.38173 (15)	0.0240 (4)
H4	0.3428	0.9985	0.3931	0.029*
C5	0.1473 (3)	0.8806 (2)	0.43633 (16)	0.0265 (4)
H5	0.1435	0.9475	0.4849	0.032*

C6	0.0351 (3)	0.7520 (2)	0.41948 (15)	0.0233 (4)
C7	0.0369 (3)	0.6541 (2)	0.34821 (15)	0.0247 (4)
H7	-0.0407	0.5662	0.3374	0.030*
C8	0.1522 (3)	0.68544 (19)	0.29313 (15)	0.0235 (4)
H8	0.1518	0.6195	0.2431	0.028*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.03360 (13)	0.03978 (14)	0.03455 (13)	-0.00199 (9)	0.02264 (10)	0.00392 (9)
O1	0.0383 (8)	0.0163 (6)	0.0388 (8)	0.0038 (6)	0.0242 (7)	-0.0003 (5)
N1	0.0257 (8)	0.0132 (7)	0.0290 (8)	-0.0018 (6)	0.0163 (7)	-0.0016 (6)
C1	0.0328 (11)	0.0252 (9)	0.0350 (11)	0.0002 (8)	0.0226 (9)	-0.0001 (8)
C2	0.0214 (9)	0.0189 (9)	0.0260 (9)	0.0025 (7)	0.0129 (7)	0.0009 (7)
C3	0.0207 (9)	0.0161 (8)	0.0237 (9)	0.0028 (6)	0.0109 (7)	0.0020 (6)
C4	0.0296 (10)	0.0159 (8)	0.0288 (10)	-0.0009 (7)	0.0157 (8)	-0.0008 (7)
C5	0.0331 (11)	0.0219 (9)	0.0285 (10)	0.0009 (8)	0.0176 (9)	-0.0028 (7)
C6	0.0213 (9)	0.0263 (9)	0.0239 (9)	0.0019 (7)	0.0119 (8)	0.0050 (7)
C7	0.0239 (9)	0.0204 (8)	0.0301 (10)	-0.0038 (7)	0.0129 (8)	-0.0001 (7)
C8	0.0257 (9)	0.0182 (8)	0.0281 (10)	-0.0014 (7)	0.0138 (8)	-0.0027 (7)

Geometric parameters (Å, °)

Br1—C6	1.8906 (19)	C3—C8	1.394 (2)
O1—C2	1.226 (2)	C4—C5	1.380 (3)
N1—C2	1.347 (2)	C4—H4	0.9500
N1—C3	1.401 (2)	C5—C6	1.384 (3)
N1—H1	0.8922	C5—H5	0.9500
C1—C2	1.501 (3)	C6—C7	1.384 (3)
C1—H1A	0.9800	C7—C8	1.377 (3)
C1—H1B	0.9800	C7—H7	0.9500
C1—H1C	0.9800	C8—H8	0.9500
C3—C4	1.390 (2)		
C2—N1—C3	127.45 (15)	C5—C4—C3	120.52 (17)
C2—N1—H1	117.0	C5—C4—H4	119.7
C3—N1—H1	115.1	C3—C4—H4	119.7
C2—C1—H1A	109.5	C4—C5—C6	119.17 (17)
C2—C1—H1B	109.5	C4—C5—H5	120.4
H1A—C1—H1B	109.5	C6—C5—H5	120.4
C2—C1—H1C	109.5	C5—C6—C7	121.18 (18)
H1A—C1—H1C	109.5	C5—C6—Br1	119.76 (15)
H1B—C1—H1C	109.5	C7—C6—Br1	119.06 (15)
O1—C2—N1	123.83 (17)	C8—C7—C6	119.33 (18)
O1—C2—C1	121.58 (17)	C8—C7—H7	120.3
N1—C2—C1	114.59 (16)	C6—C7—H7	120.3
C4—C3—C8	119.39 (18)	C7—C8—C3	120.39 (18)
C4—C3—N1	117.09 (16)	C7—C8—H8	119.8

C8—C3—N1	123.46 (17)	C3—C8—H8	119.8
C3—N1—C2—O1	-3.1 (3)	C4—C5—C6—C7	-0.7 (3)
C3—N1—C2—C1	176.68 (18)	C4—C5—C6—Br1	179.88 (15)
C2—N1—C3—C4	164.06 (18)	C5—C6—C7—C8	0.0 (3)
C2—N1—C3—C8	-18.6 (3)	Br1—C6—C7—C8	179.37 (15)
C8—C3—C4—C5	0.8 (3)	C6—C7—C8—C3	1.2 (3)
N1—C3—C4—C5	178.23 (17)	C4—C3—C8—C7	-1.6 (3)
C3—C4—C5—C6	0.3 (3)	N1—C3—C8—C7	-178.81 (18)

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C3–C8 ring.

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O1 ⁱ	0.89	2.00	2.885 (2)	174
C1—H1B \cdots Cg1 ⁱⁱ	0.98	2.84	3.761 (3)	157

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