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2-Bromo-1-(4-methoxyphenyl)ethanone

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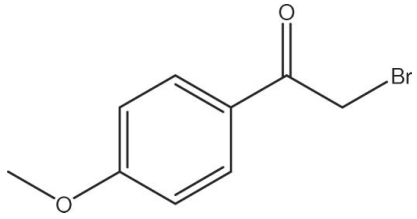
Key indicators: single-crystal X-ray study; T = 305 K; mean σ(C–C) = 0.009 Å; R factor = 0.054; wR factor = 0.116; data-to-parameter ratio = 15.0.

The title compound, $C_9H_9BrO_2$, prepared by the reaction of 4-methoxyacetophenone and cupric bromide, , is approximately planar (r.m.s. deviation 0.0008 Å). In the crystal, weak intermolecular aromatic C—H···$O_{carbonyl}$ hydrogen-bonding interactions result in a one-dimensional chain structure.

Related literature

For background to hydrazone compounds, see: Domiano *et al.* (1984); Li *et al.* (1988); Sadik *et al.* (2004). For background to thiazole compounds, see: Shinagawa *et al.* (1997); Shivarama *et al.*(2003); Dinçer *et al.* (2005); Zhang *et al.* (2009). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_9H_9BrO_2$
M_r = 229.06
Monoclinic, $P2_1/c$
a = 7.7360 (15) Å
b = 12.441 (3) Å
c = 10.048 (2) Å
β = 111.42 (3)°
V = 900.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.52 mm^{-1}
T = 305 K
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.465, T_{max} = 0.661
1634 measured reflections
1634 independent reflections
924 reflections with $I > 2\sigma(I)$
3 standard reflections every 200 reflections intensity decay: 9%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.116
S = 1.01
1634 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7A···O1^{i}	0.93	2.58	3.505 (7)	171

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: ZS2006).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Dinçer, M., Özdemir, N., Çukurovalı, A. & Yılmaz, İ. (2005). *Acta Cryst.* E**61**, o1712-o1714.
Domiano, P., Pelizzi, C. & Predieri, G. (1984). *Polyhedron*, **3**, 281–286.
Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Li, X. R., Sun, Z. M. & Chang, J. C. (1988). *Synth. React. Inorg. Met. Org. Chem.* **18**, 657–665.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sadık, G., Necmi, D., Ibrahim, Y., Alaaddin, Ç. & Dinçer, M. (2004). *Acta Cryst.* E**60**, o889-o891.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Shinagawa, H., Yamaga, H., Houchigai, H., Sumita, Y. & Sunagawa, M. (1997). *Bioorg. Med. Chem.* **5**, 601–621.
Shivarama, H. B., Malini, K. V., Sooryanarayana, R. B., Sarojini, B. K. & Suchetha, K. N. (2003). *Eur. J. Med. Chem.* **38**, 313–318.
Zhang, J., Wu, L., Zhuang, L. & Wang, G. (2009). *Acta Cryst.* E**65**, o884.

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S1. Comment

The chemistry of hydrazones, owing to their coordinating capability, pharmacological activity, antibacterial and antifungal properties, and their use in analytical chemistry as highly selective extractants, has been intensively investigated (Domiano *et al.*, 1984; Li *et al.*, 1988; Sadık *et al.*, 2004). In addition, many thiazole compounds are of considerable importance because of their antibacterial and anti-inflammatory activity (Shinagawa *et al.*, 1997; Shivarama *et al.*, 2003; Dinçer *et al.*, 2005). We have focused our synthetic and structural studies on new derivatives of thiazole-substituted hydrazones (Zhang *et al.*, 2009). We report here the crystal structure of a bromo-substituted methoxyacetophenone, the title compound $C_9H_9BrO_2$ (I), which is a very important intermediate for the synthesis of thiazole-substituted hydrazones.

In (I), all bond lengths are within normal ranges (Allen *et al.*, 1987). The presence of a strong intramolecular aromatic C8–H···$O1_{carbonyl}$ hydrogen bond (Table 1) forms a pseudo five-membered ring [O1/C2/C3/C8/H8A with an r.m.s. deviation 0.0069 Å], maintaining essential coplanarity of the ketone side chain with the benzene ring (Fig. 1) [torsion angle: C1–C2–C3–C8, -178.0 (5)°]. The methoxy group is similarly essentially coplanar [torsion angle: C5–C6–O2–C9, 172.0 (6)°].

The molecules of (I) associate through weak intermolecular aromatic C—H···$O_{carbonyl}$ hydrogen bonds forming one-dimensional chains which extend along the *b* axial direction in the unit cell (Fig.2).

S2. Experimental

4-Methoxyacetophenone (1.50 g, 0.01 mol) was dissolved in 50 ml ethyl acetate, cupric bromide (3.36 g, 0.015 mol) was added and the mixture was refluxed for *ca.* 3 h. On cooling, the solid which separated was filtered and recrystallized from ethyl acetate. Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of ethyl acetate. ^{1}H NMR ($CDCl_3$, δ, p.p.m.) 8.17 (d, 2 H), 7.49 (d, 2 H), 4.5 (s, 2 H), 3.81 (s,3 H).

S3. Refinement

All H atoms were positioned geometrically, with C—H = 0.93 Å, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where x= 1.5 for methyl H and x = 1.2 for methylene and aromatic H atoms.

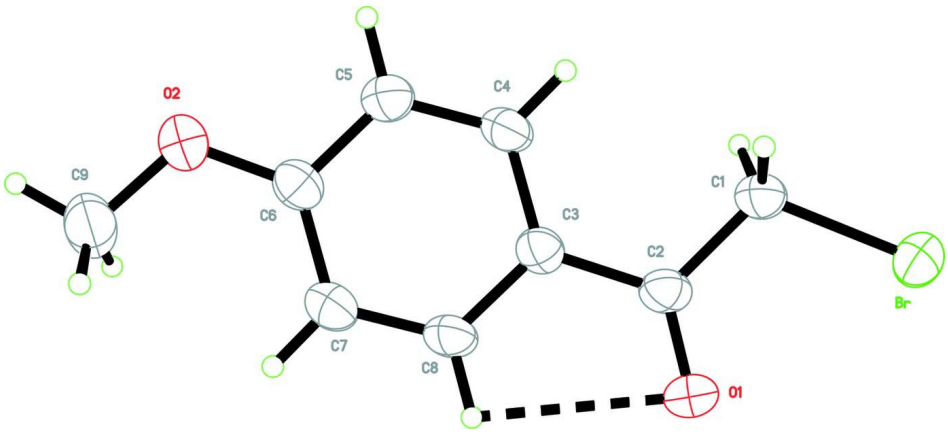

Figure 1

A view of the molecular structure of (I) showing the atom-numbering scheme with non-H atoms drawn as 30% displacement ellipsoids. The intramolecular hydrogen bond is shown as a dashed line.

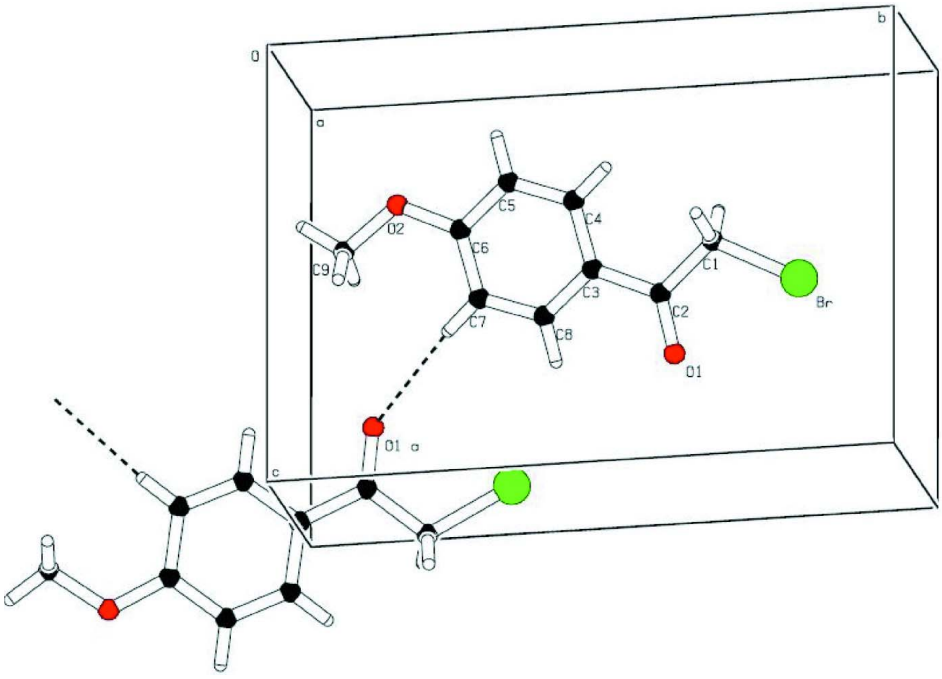

Figure 2

The crystal packing of (I) showing hydrogen bonds as dashed lines.

2-Bromo-1-(4-methoxyphenyl)ethanone

Crystal data

$C_9H_9BrO_2$
M_r = 229.06
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 7.7360 (15) Å
b = 12.441 (3) Å
c = 10.048 (2) Å
β = 111.42 (3)°
V = 900.3 (4) Å^3
Z = 4
F(000) = 456
D_x = 1.690 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 27 reflections
θ = 1–25°
μ = 4.52 mm^{-1}

T = 305 K
Block, colorless
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.465, T_{max} = 0.661
1634 measured reflections
1634 independent reflections
924 reflections with $I > 2\sigma(I)$
R_{int} = 0.000
θ_{max} = 25.3°, θ_{min} = 2.7°
$h = -9 \rightarrow 8$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 12$
3 standard reflections every 200 reflections
intensity decay: 9%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.116
S = 1.01
1634 reflections
109 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.048P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Br	0.20597 (10)	0.66539 (6)	0.07859 (7)	0.0717 (3)
O1	0.3621 (7)	0.8754 (4)	0.2083 (5)	0.0848 (15)
C1	0.1809 (8)	0.8016 (5)	−0.0164 (6)	0.0576 (17)
H1A	0.2308	0.7957	−0.0918	0.069*
H1B	0.0500	0.8190	−0.0608	0.069*
O2	0.2187 (6)	1.3075 (4)	−0.1487 (5)	0.0689 (13)
C2	0.2781 (8)	0.8917 (5)	0.0820 (6)	0.0534 (16)
C3	0.2657 (7)	0.9994 (5)	0.0181 (6)	0.0478 (14)
C4	0.1597 (8)	1.0221 (5)	−0.1242 (6)	0.0593 (17)
H4A	0.0957	0.9671	−0.1849	0.071*
C5	0.1493 (9)	1.1250 (5)	−0.1751 (7)	0.0633 (18)
H5A	0.0780	1.1390	−0.2703	0.076*
C6	0.2432 (8)	1.2086 (5)	−0.0870 (7)	0.0538 (16)

C7	0.3497 (8)	1.1859 (5)	0.0560 (7)	0.0600 (17)
H7A	0.4138	1.2406	0.1172	0.072*
C8	0.3586 (8)	1.0831 (5)	0.1051 (6)	0.0564 (16)
H8A	0.4297	1.0688	0.2003	0.068*
C9	0.2906 (10)	1.3980 (6)	−0.0610 (8)	0.083 (2)
H9A	0.2625	1.4618	−0.1186	0.124*
H9B	0.4227	1.3908	−0.0151	0.124*
H9C	0.2353	1.4028	0.0102	0.124*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0721 (5)	0.0588 (5)	0.0722 (5)	0.0064 (4)	0.0121 (3)	0.0076 (4)
O1	0.104 (4)	0.067 (3)	0.050 (3)	0.002 (3)	−0.011 (3)	0.003 (2)
C1	0.054 (4)	0.060 (4)	0.050 (3)	0.002 (3)	0.008 (3)	−0.003 (3)
O2	0.079 (3)	0.054 (3)	0.070 (3)	−0.007 (2)	0.021 (2)	−0.002 (2)
C2	0.042 (3)	0.060 (4)	0.049 (4)	0.007 (3)	0.006 (3)	−0.005 (3)
C3	0.036 (3)	0.050 (4)	0.053 (4)	−0.001 (3)	0.011 (3)	−0.003 (3)
C4	0.060 (4)	0.056 (4)	0.049 (3)	−0.002 (3)	0.005 (3)	−0.012 (3)
C5	0.067 (4)	0.059 (4)	0.052 (4)	0.005 (4)	0.007 (3)	0.000 (3)
C6	0.047 (4)	0.056 (4)	0.058 (4)	−0.004 (3)	0.018 (3)	−0.007 (3)
C7	0.054 (4)	0.062 (5)	0.058 (4)	−0.011 (3)	0.013 (3)	−0.010 (3)
C8	0.044 (4)	0.064 (4)	0.049 (3)	−0.004 (3)	0.001 (3)	−0.005 (3)
C9	0.087 (5)	0.063 (5)	0.096 (5)	−0.012 (4)	0.031 (4)	−0.004 (4)

Geometric parameters (Å, º)

Br—C1	1.920 (6)	C4—H4A	0.9300
O1—C2	1.213 (6)	C5—C6	1.387 (8)
C1—C2	1.502 (8)	C5—H5A	0.9300
C1—H1A	0.9700	C6—C7	1.400 (8)
C1—H1B	0.9700	C7—C8	1.363 (8)
O2—C6	1.359 (7)	C7—H7A	0.9300
O2—C9	1.412 (8)	C8—H8A	0.9300
C2—C3	1.474 (8)	C9—H9A	0.9600
C3—C8	1.380 (7)	C9—H9B	0.9600
C3—C4	1.393 (8)	C9—H9C	0.9600
C4—C5	1.370 (8)		
C2—C1—Br	113.3 (4)	C4—C5—H5A	119.4
C2—C1—H1A	108.9	C6—C5—H5A	119.4
Br—C1—H1A	108.9	O2—C6—C5	115.8 (5)
C2—C1—H1B	108.9	O2—C6—C7	125.6 (6)
Br—C1—H1B	108.9	C5—C6—C7	118.6 (6)
H1A—C1—H1B	107.7	C8—C7—C6	119.6 (6)
C6—O2—C9	118.7 (5)	C8—C7—H7A	120.2
O1—C2—C3	122.1 (6)	C6—C7—H7A	120.2
O1—C2—C1	120.8 (6)	C7—C8—C3	122.2 (6)

C3—C2—C1	117.0 (5)	C7—C8—H8A	118.9
C8—C3—C4	118.2 (6)	C3—C8—H8A	118.9
C8—C3—C2	118.3 (5)	O2—C9—H9A	109.5
C4—C3—C2	123.4 (6)	O2—C9—H9B	109.5
C5—C4—C3	120.3 (6)	H9A—C9—H9B	109.5
C5—C4—H4A	119.9	O2—C9—H9C	109.5
C3—C4—H4A	119.9	H9A—C9—H9C	109.5
C4—C5—C6	121.2 (6)	H9B—C9—H9C	109.5

Br—C1—C2—O1	0.0 (8)	C9—O2—C6—C5	172.0 (6)
Br—C1—C2—C3	−179.8 (4)	C9—O2—C6—C7	−6.3 (9)
O1—C2—C3—C8	2.2 (9)	C4—C5—C6—O2	−178.5 (6)
C1—C2—C3—C8	−178.0 (5)	C4—C5—C6—C7	0.0 (9)
O1—C2—C3—C4	−175.3 (6)	O2—C6—C7—C8	178.3 (6)
C1—C2—C3—C4	4.5 (9)	C5—C6—C7—C8	0.0 (9)
C8—C3—C4—C5	0.1 (9)	C6—C7—C8—C3	0.0 (9)
C2—C3—C4—C5	177.6 (6)	C4—C3—C8—C7	−0.1 (9)
C3—C4—C5—C6	0.0 (10)	C2—C3—C8—C7	−177.8 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O1	0.93	2.47	2.780 (8)	100
C7—H7*A*···O1^{i}	0.93	2.58	3.505 (7)	171

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1/2.