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2-Bromo-*N*′-[(*E*)-4-chlorobenzylidene]-5-methoxybenzohydrazide

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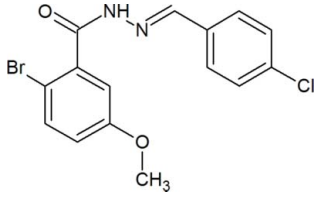
Key indicators: single-crystal X-ray study; T = 203 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.081; data-to-parameter ratio = 26.2.

In the title molecule, $C_{15}H_{12}BrClN_2O_2$, the mean planes of the coplanar 4-chlorophenyl and methylenehydrazide groups are twisted from that of the 2-bromo-5-methoxyphenyl group by 52.3 (2)°. Crystal packing is stabilized by intermolecular N—H···O hydrogen bonds between a hydrazide H atom and the carbonyl O atom, which link the molecules into anti-parallel ribbons along the b axis of the unit cell.

Related literature

For related structures, see: Chen & Yu (2006*a*,*b*); Zhen & Han (2005*a*,*b*); Diao & Yu (2006); Qiu *et al.* (2006*a*,*b*); For related literature, see: Varma *et al.* (1986); Misra *et al.* (1981); Desai *et al.* (2001); Singh & Dash (1988); Hodnett & Dunn (1970); Yathirajan *et al.* (2007).



Experimental

Crystal data

$C_{15}H_{12}BrClN_2O_2$
M_r = 367.63
Monoclinic, $P2_1/c$
a = 7.2921 (3) Å
b = 21.7635 (9) Å
c = 9.5067 (4) Å
β = 95.268 (4)°
V = 1502.36 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.92 mm^{-1}
T = 203 K
0.57 × 0.49 × 0.17 mm

Data collection

Oxford Diffraction Gemini R diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.460, T_{max} = 1.000
(expected range = 0.280–0.609)
17240 measured reflections
4997 independent reflections
2202 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
2 standard reflections
every 50 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.081
S = 0.95
4997 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.85 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.87	2.02	2.870 (2)	165

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

Data collection: *CrysAlisPro* (Oxford Diffraction, 2007); cell refinement: *CrysAlisPro*; data reduction: *CrysAlisPro*; 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: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2351).

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supplementary materials

Acta Cryst. (2007). E**63**, o3652 [**doi:10.1107/S1600536807036458**]

2-Bromo-*N*'-[(*E*)-4-chlorobenzylidene]-5-methoxybenzohydrazide

R. J. Butcher, J. P. Jasinski, B. Narayana, K. Sunil and H. S. Yathirajan

Comment

Schiff bases are used as substrates in the preparation of number of industrial and biologically active compounds *via* ring closure, cycloaddition and replacement reactions. Some Schiff base derivatives were reported to possess antimicrobial, anti-inflammatory and central nervous system activities. Moreover, Schiff bases are also known to have biological activities such as antimicrobial, antifungal, antitumor, and as herbicides. A new Schiff base, $C_{15}H_{12}BrClN_2O_2$ was synthesized and its crystal structure is reported.

The mean planes of 4-chlorophenyl and methylene hydrazide groups are coplanar [N2–C8–C9–C10 dihedral angle = −3.0 (3)°] and twisted from that of the 2-bromo, 5-methoxybenzo group by 52.3 (2)°.

Intermolecular N—H···O hydrogen bonding interactions involving a hydrizide hydrogen (N1–H1A) and the methylene oxygen (O1) link the molecules into inverted parallel ribbons along the *b* axis of the unit cell.

Experimental

A mixture of 2-bromo-5-methoxybenzohydrazide (0.735 g, 0.003 mol) and 4-chlorobenzaldehyde (0.42 g, 0.003 mol) in 15 ml of absolute ethyl alcohol containing 2 drops of 4 *M* sulfuric acid was refluxed for about 3 h. On cooling, the solid separated was filtered and recrystallized from ethyl acetate (m.p.: 445–447 K). Analysis found: C 48.92, H 3.21, N 7.54%; $C_{15}H_{12}BrClN_2O_2$ requires: C 49.01, H 3.29, N 7.62%.

Refinement

All H atoms were refined using a riding model with N—H = 0.87 Å and C—H = 0.94–0.97 Å, and with U_{iso}(H) = 1.18–1.49U_{eq}(C,*N*).

Figures

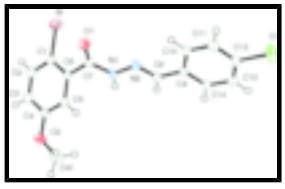

Fig. 1. Molecular structure of the title compound, showing atom labelling and 50% probability displacement ellipsoids.

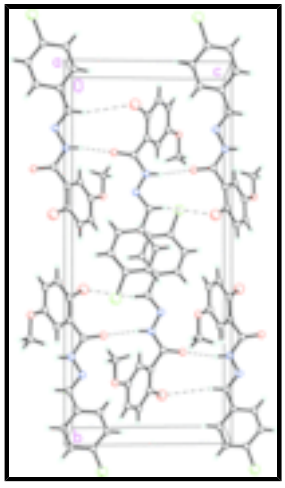

Fig. 2. Packing diagram of the title compound, viewed down the *a* axis. Dashed lines indicate N—H···O hydrogen bonds.

2-Bromo-*N*'-[(*E*)-4-chlorobenzylidene]-5-methoxybenzohydrazide

Crystal data

$C_{15}H_{12}BrClN_2O_2$	F_{000} = 736
M_r = 367.63	D_x = 1.625 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 4592 reflections
a = 7.2921 (3) Å	θ = 4.7–32.5º
b = 21.7635 (9) Å	μ = 2.92 mm^{-1}
c = 9.5067 (4) Å	T = 203 K
β = 95.268 (4)º	Plate, yellow
V = 1502.36 (11) Å^3	0.57 × 0.49 × 0.17 mm
Z = 4	

Data collection

Oxford Diffraction Gemini R diffractometer	R_{int} = 0.056
Radiation source: fine-focus sealed tube	θ_{max} = 32.6º
Monochromator: graphite	θ_{min} = 4.7º
T = 203 K	h = −10→10
φ and ω scans	k = −32→32
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)	l = −14→13
T_{min} = 0.460, T_{max} = 1.000	2 standard reflections
17240 measured reflections	every 50 reflections
4997 independent reflections	intensity decay: none
2202 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.081$	$w = 1/[\sigma^2(F_o^2) + (0.0305P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.95$	$(\Delta/\sigma)_{max} = 0.001$
4997 reflections	$\Delta\rho_{max} = 0.85$ e Å^{-3}
191 parameters	$\Delta\rho_{min} = -0.46$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2$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_{iso}*/U_{eq}
Br	0.69194 (3)	0.101993 (12)	0.41614 (3)	0.03808 (10)
Cl	0.90438 (10)	0.62116 (3)	0.30120 (8)	0.0506 (2)
O1	0.5198 (2)	0.22677 (7)	0.28481 (17)	0.0340 (4)
O2	0.0069 (2)	0.16145 (8)	0.6990 (2)	0.0420 (5)
N1	0.5265 (2)	0.28449 (9)	0.4846 (2)	0.0266 (5)
H1A	0.5063	0.2856	0.5733	0.032*
N2	0.5964 (2)	0.33582 (9)	0.4208 (2)	0.0270 (5)
C1	0.4697 (3)	0.12299 (11)	0.4942 (2)	0.0264 (5)
C2	0.3792 (3)	0.07815 (12)	0.5642 (3)	0.0311 (6)
H2A	0.4255	0.0378	0.5683	0.037*
C3	0.2230 (3)	0.09179 (12)	0.6277 (3)	0.0362 (6)
H3A	0.1595	0.0605	0.6712	0.043*
C4	0.1583 (3)	0.15199 (12)	0.6275 (3)	0.0293 (6)
C41	−0.0660 (4)	0.22188 (13)	0.7003 (3)	0.0549 (9)
H41A	−0.1768	0.2219	0.7495	0.082*
H41B	−0.0954	0.2359	0.6040	0.082*
H41C	0.0244	0.2492	0.7483	0.082*
C5	0.2471 (3)	0.19706 (11)	0.5575 (2)	0.0261 (5)

H5A	0.2019	0.2375	0.5557	0.031*
C6	0.4031 (3)	0.18314 (10)	0.4896 (2)	0.0229 (5)
C7	0.4899 (3)	0.23276 (11)	0.4082 (2)	0.0242 (5)
C8	0.6299 (3)	0.38136 (11)	0.5042 (3)	0.0258 (5)
H8A	0.6084	0.3776	0.5998	0.031*
C9	0.7011 (3)	0.43919 (11)	0.4533 (2)	0.0257 (6)
C10	0.7435 (3)	0.44632 (11)	0.3144 (3)	0.0284 (6)
H10A	0.7305	0.4128	0.2520	0.034*
C11	0.8042 (3)	0.50197 (12)	0.2673 (3)	0.0325 (6)
H11A	0.8315	0.5066	0.1732	0.039*
C12	0.8246 (3)	0.55101 (11)	0.3607 (3)	0.0298 (6)
C13	0.7867 (3)	0.54521 (11)	0.4987 (3)	0.0330 (6)
H13A	0.8026	0.5787	0.5611	0.040*
C14	0.7248 (3)	0.48933 (11)	0.5444 (3)	0.0305 (6)
H14A	0.6982	0.4850	0.6388	0.037*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.04184 (16)	0.03696 (17)	0.03731 (17)	0.00850 (13)	0.01377 (11)	0.00091 (13)
Cl	0.0590 (4)	0.0315 (4)	0.0599 (5)	−0.0118 (3)	−0.0027 (4)	0.0166 (3)
O1	0.0519 (10)	0.0302 (10)	0.0215 (10)	−0.0067 (8)	0.0111 (8)	−0.0010 (8)
O2	0.0403 (10)	0.0351 (11)	0.0545 (13)	−0.0035 (9)	0.0257 (9)	0.0033 (9)
N1	0.0364 (11)	0.0238 (11)	0.0211 (11)	−0.0064 (9)	0.0106 (8)	0.0014 (8)
N2	0.0310 (10)	0.0223 (12)	0.0283 (12)	−0.0035 (10)	0.0068 (8)	0.0015 (9)
C1	0.0317 (12)	0.0258 (14)	0.0223 (14)	0.0005 (11)	0.0062 (10)	−0.0010 (10)
C2	0.0422 (14)	0.0227 (14)	0.0279 (15)	0.0023 (12)	−0.0001 (11)	0.0013 (11)
C3	0.0425 (14)	0.0349 (17)	0.0321 (16)	−0.0101 (13)	0.0075 (12)	0.0051 (12)
C4	0.0282 (12)	0.0303 (15)	0.0306 (15)	−0.0027 (12)	0.0099 (10)	0.0002 (11)
C41	0.0457 (16)	0.046 (2)	0.079 (3)	0.0093 (15)	0.0390 (16)	0.0093 (16)
C5	0.0297 (12)	0.0221 (13)	0.0266 (14)	−0.0019 (11)	0.0038 (10)	0.0008 (10)
C6	0.0269 (12)	0.0214 (13)	0.0204 (13)	−0.0057 (10)	0.0031 (9)	0.0005 (9)
C7	0.0259 (12)	0.0261 (14)	0.0207 (14)	0.0004 (10)	0.0034 (10)	−0.0003 (10)
C8	0.0311 (12)	0.0256 (14)	0.0210 (13)	−0.0003 (11)	0.0041 (10)	0.0017 (10)
C9	0.0229 (12)	0.0264 (14)	0.0278 (15)	−0.0002 (10)	0.0032 (10)	0.0007 (10)
C10	0.0291 (12)	0.0224 (14)	0.0340 (16)	−0.0010 (11)	0.0036 (11)	−0.0024 (11)
C11	0.0332 (14)	0.0343 (16)	0.0301 (15)	−0.0005 (12)	0.0036 (11)	0.0059 (12)
C12	0.0288 (12)	0.0216 (14)	0.0380 (17)	−0.0013 (11)	−0.0026 (11)	0.0077 (11)
C13	0.0350 (13)	0.0254 (15)	0.0378 (17)	−0.0001 (12)	−0.0009 (11)	−0.0053 (12)
C14	0.0364 (13)	0.0254 (14)	0.0306 (15)	−0.0009 (12)	0.0071 (11)	−0.0033 (11)

Geometric parameters (Å, °)

Br—C1	1.899 (2)	C41—H41B	0.9700
Cl—C12	1.746 (2)	C41—H41C	0.9700
O1—C7	1.220 (2)	C5—C6	1.392 (3)
O2—C4	1.365 (3)	C5—H5A	0.9400
O2—C41	1.419 (3)	C6—C7	1.502 (3)
N1—C7	1.353 (3)	C8—C9	1.461 (3)

N1—N2	1.390 (2)	C8—H8A	0.9400
N1—H1A	0.8700	C9—C10	1.392 (3)
N2—C8	1.279 (3)	C9—C14	1.394 (3)
C1—C2	1.383 (3)	C10—C11	1.379 (3)
C1—C6	1.396 (3)	C10—H10A	0.9400
C2—C3	1.369 (3)	C11—C12	1.388 (3)
C2—H2A	0.9400	C11—H11A	0.9400
C3—C4	1.392 (3)	C12—C13	1.370 (3)
C3—H3A	0.9400	C13—C14	1.381 (3)
C4—C5	1.380 (3)	C13—H13A	0.9400
C41—H41A	0.9700	C14—H14A	0.9400
C4—O2—C41	117.70 (19)	C5—C6—C7	118.9 (2)
C7—N1—N2	119.81 (19)	C1—C6—C7	121.92 (19)
C7—N1—H1A	120.1	O1—C7—N1	124.1 (2)
N2—N1—H1A	120.1	O1—C7—C6	122.7 (2)
C8—N2—N1	114.2 (2)	N1—C7—C6	113.18 (19)
C2—C1—C6	119.8 (2)	N2—C8—C9	121.1 (2)
C2—C1—Br	118.72 (19)	N2—C8—H8A	119.5
C6—C1—Br	121.37 (17)	C9—C8—H8A	119.5
C3—C2—C1	120.8 (2)	C10—C9—C14	118.5 (2)
C3—C2—H2A	119.6	C10—C9—C8	121.8 (2)
C1—C2—H2A	119.6	C14—C9—C8	119.7 (2)
C2—C3—C4	119.9 (2)	C11—C10—C9	120.8 (2)
C2—C3—H3A	120.0	C11—C10—H10A	119.6
C4—C3—H3A	120.0	C9—C10—H10A	119.6
O2—C4—C5	124.8 (2)	C10—C11—C12	119.1 (2)
O2—C4—C3	115.5 (2)	C10—C11—H11A	120.4
C5—C4—C3	119.7 (2)	C12—C11—H11A	120.4
O2—C41—H41A	109.5	C13—C12—C11	121.5 (2)
O2—C41—H41B	109.5	C13—C12—Cl	119.54 (19)
H41A—C41—H41B	109.5	C11—C12—Cl	119.0 (2)
O2—C41—H41C	109.5	C12—C13—C14	118.9 (2)
H41A—C41—H41C	109.5	C12—C13—H13A	120.6
H41B—C41—H41C	109.5	C14—C13—H13A	120.6
C4—C5—C6	120.6 (2)	C13—C14—C9	121.3 (2)
C4—C5—H5A	119.7	C13—C14—H14A	119.4
C6—C5—H5A	119.7	C9—C14—H14A	119.4
C5—C6—C1	119.1 (2)		
C7—N1—N2—C8	−178.3 (2)	C5—C6—C7—O1	−126.1 (2)
C6—C1—C2—C3	1.0 (4)	C1—C6—C7—O1	51.1 (3)
Br—C1—C2—C3	177.07 (19)	C5—C6—C7—N1	52.4 (3)
C1—C2—C3—C4	−2.9 (4)	C1—C6—C7—N1	−130.3 (2)
C41—O2—C4—C5	0.7 (4)	N1—N2—C8—C9	−178.99 (18)
C41—O2—C4—C3	−179.1 (2)	N2—C8—C9—C10	−3.0 (3)
C2—C3—C4—O2	−177.1 (2)	N2—C8—C9—C14	175.8 (2)
C2—C3—C4—C5	3.1 (4)	C14—C9—C10—C11	−1.2 (3)
O2—C4—C5—C6	178.8 (2)	C8—C9—C10—C11	177.6 (2)
C3—C4—C5—C6	−1.4 (4)	C9—C10—C11—C12	0.6 (3)

C4—C5—C6—C1	−0.5 (4)	C10—C11—C12—C13	0.4 (4)
C4—C5—C6—C7	176.8 (2)	C10—C11—C12—Cl	179.21 (17)
C2—C1—C6—C5	0.8 (3)	C11—C12—C13—C14	−0.8 (3)
Br—C1—C6—C5	−175.25 (18)	Cl—C12—C13—C14	−179.60 (18)
C2—C1—C6—C7	−176.5 (2)	C12—C13—C14—C9	0.2 (4)
Br—C1—C6—C7	7.5 (3)	C10—C9—C14—C13	0.8 (3)
N2—N1—C7—O1	2.5 (3)	C8—C9—C14—C13	−178.1 (2)
N2—N1—C7—C6	−176.03 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1^{i}	0.87	2.02	2.870 (2)	165

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

Fig. 1

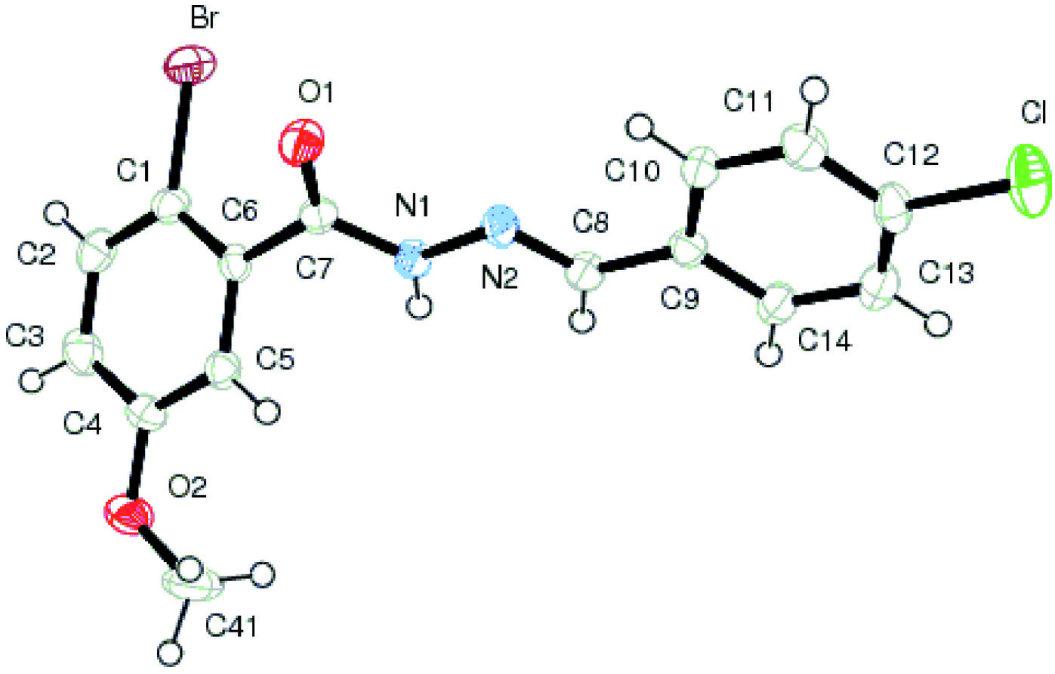

Fig. 2