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N′-Benzylidene-2-chloro-*N*-(4-chlorophenyl)acetohydrazide

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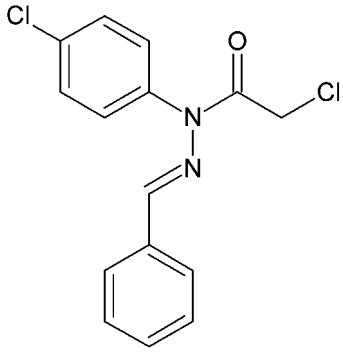
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.053; wR factor = 0.145; data-to-parameter ratio = 13.3.

In the title compound, $C_{15}H_{12}Cl_2N_2O$, the atoms not making up the chlorobenzene ring are approximately coplanar (r.m.s. deviation = 0.073 Å). The dihedral angle between these 13 atoms and the chlorobenzene ring is 67.37 (10)°. The C=O and Csp^2—Cl groups are almost eclipsed [Cl—C—C=O = −6.5 (3)°]. In the crystal, $C(6)$ chains linked by C—H···O hydrogen bonds result in [100] chains.

Related literature

For background to Schiff bases, see: Nithinchandra *et al.* (2013); Shyma *et al.* (2013).



Experimental

Crystal data

$C_{15}H_{12}Cl_2N_2O$ M_r = 307.17
Monoclinic, $P2_1/c$
a = 5.8548 (5) Å
b = 8.8892 (7) Å
c = 28.273 (2) Å
β = 93.574 (4)°
V = 1468.6 (2) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 3.95 mm^{-1}
T = 296 K
0.24 × 0.23 × 0.23 mm

Data collection

Bruker X8 Proteum diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.451, T_{max} = 0.464
10066 measured reflections
2424 independent reflections
2059 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.145
S = 1.04
2424 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.93	2.40	3.256 (3)	154

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

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7168).

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supplementary materials

Acta Cryst. (2014). E**70**, o31 [doi:10.1107/S1600536813032790]

N′-Benzylidene-2-chloro-*N*-(4-chlorophenyl)acetohydrazide

N. Vinutha, S. Madan Kumar, P. C. Shyma, B. Kalluraya, N. K. Lokanath and D. Revannasiddaiah

1. Comment

Schiff bases are important class of compounds in medicinal chemistry, showing e.g.: antimicrobal (Shyma *et al.*, 2013), and anticonvulsant (Nithinchandra *et al.*, 2013) activities. As part of our studies in this area, we now describe the structure of the title compound, (I), (Fig. 1).

The dihedral angle between the benzene ring is 63.18° (13) and the molecules are linked to one another with hydrogen bonds C3—H3···O1 (Table 1, Fig. 2) along *a*-axis.

2. Experimental

4-Chlorophenylhydrazine (0.01 mol) was stirred with benzaldehyde (0.01 mol) in methanol (10 mL) in presence of few drops of acetic acid. The resulting precipitate was filtered and washed with chilled methanol and dried. The resulting Schiff base, 1-benzylidene-2-(4-chlorophenyl)hydrazine (0.01 mol) and triethylamine (0.01 mol) was taken in dioxane solvent. Chloroacetylchloride (0.01 mol) was added to the above mixture at 0–5°C. After completion of the reaction, the mixture was poured on to ice cold water. The precipitated solid was filtered, washed with water and recrystallized from ethanol. Brown blocks were obtained from a 1:2 mixture of DMF and ethanol solution by slow evaporation.

3. Refinement

All the H atoms were fixed geometrically (C—H= 0.93–0.96 Å) and allowed to ride on their parent atoms with U_{iso}(H) =1.5U_{eq}(*C*-methyl) and = 1.2U_{eq}(C) for other H atoms.

Computing details

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

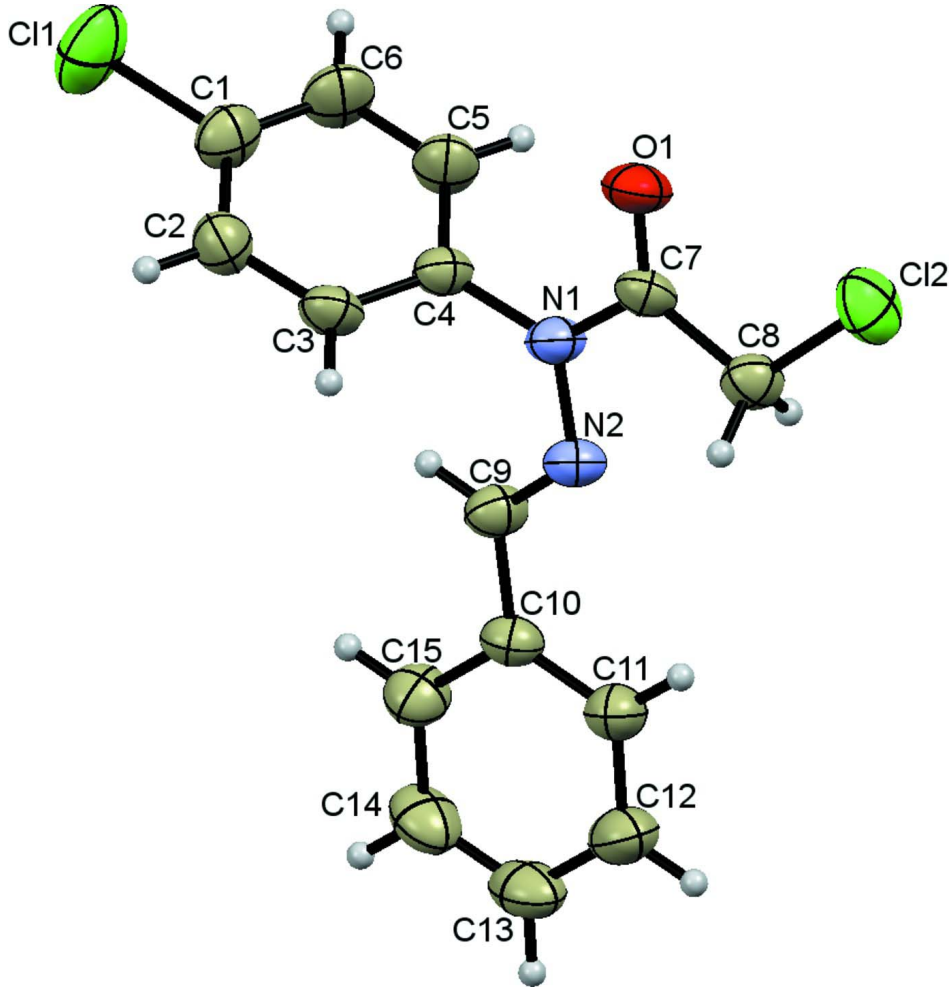

Figure 1

ORTEP diagram of the title compound with 50% probability ellipsoids.

N′-Benzylidene-2-chloro-*N*-(4-chlorophenyl)acetohydrazide

Crystal data

$C_{15}H_{12}Cl_2N_2O$
$M_r = 307.17$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.8548$ (5) Å
$b = 8.8892$ (7) Å
$c = 28.273$ (2) Å
$\beta = 93.574$ (4)°
$V = 1468.6$ (2) Å^3
$Z = 4$

$F(000) = 632$
$D_x = 1.389$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 2424 reflections
$\theta = 3.1$–$64.9°$
$\mu = 3.95$ mm^{-1}
$T = 296$ K
Block, brown
0.24 × 0.23 × 0.23 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 10.7 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.451, T_{max} = 0.464
10066 measured reflections
2424 independent reflections
2059 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 64.9°, θ_{min} = 3.1°
h = −3→6
k = −10→10
l = −32→32

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.145
S = 1.04
2424 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.0867P)^2 + 0.4858P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), FC*=KFC[1+0.001XFC$^2\Lambda^3$/SIN(2Θ)]$^{-1/4}$
Extinction coefficient: 0.0091 (10)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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}
Cl1	0.88967 (18)	1.37204 (11)	0.24673 (3)	0.0826 (4)
Cl2	0.20082 (12)	0.83970 (9)	−0.00451 (2)	0.0605 (3)
O1	0.3525 (3)	1.0325 (2)	0.07342 (7)	0.0507 (6)
N1	0.6637 (3)	0.9050 (2)	0.10193 (7)	0.0379 (6)
N2	0.8010 (3)	0.7816 (2)	0.09360 (7)	0.0374 (6)
C1	0.8254 (5)	1.2311 (3)	0.20500 (9)	0.0492 (9)
C2	0.9644 (5)	1.2137 (3)	0.16799 (9)	0.0474 (8)
C3	0.9104 (4)	1.1056 (3)	0.13393 (9)	0.0428 (8)
C4	0.7207 (4)	1.0156 (3)	0.13769 (8)	0.0365 (7)
C5	0.5846 (5)	1.0340 (3)	0.17556 (9)	0.0483 (8)
C6	0.6354 (5)	1.1429 (3)	0.20918 (10)	0.0563 (10)
C7	0.4802 (4)	0.9266 (3)	0.06984 (8)	0.0372 (7)
C8	0.4559 (4)	0.8111 (3)	0.03078 (9)	0.0433 (8)
C9	0.9704 (4)	0.7535 (3)	0.12257 (8)	0.0419 (8)
C10	1.1217 (4)	0.6259 (3)	0.11428 (9)	0.0394 (7)

C11	1.0788 (5)	0.5252 (3)	0.07751 (9)	0.0472 (8)
C12	1.2319 (5)	0.4110 (3)	0.07002 (12)	0.0588 (10)
C13	1.4303 (5)	0.3975 (3)	0.09870 (13)	0.0617 (10)
C14	1.4735 (5)	0.4958 (3)	0.13506 (12)	0.0611 (10)
C15	1.3196 (5)	0.6093 (3)	0.14347 (10)	0.0501 (9)
H2	1.09330	1.27390	0.16590	0.0570*
H3	1.00200	1.09370	0.10850	0.0510*
H5	0.45790	0.97230	0.17830	0.0580*
H6	0.54240	1.15640	0.23430	0.0680*
H8A	0.45510	0.71110	0.04450	0.0520*
H8B	0.58570	0.81810	0.01120	0.0520*
H9	0.99860	0.81410	0.14910	0.0500*
H11	0.94640	0.53450	0.05780	0.0570*
H12	1.20130	0.34280	0.04550	0.0710*
H13	1.53430	0.32140	0.09320	0.0740*
H14	1.60730	0.48650	0.15440	0.0730*
H15	1.34880	0.67480	0.16880	0.0600*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1113 (8)	0.0867 (6)	0.0492 (5)	−0.0170 (5)	0.0013 (4)	−0.0269 (4)
Cl2	0.0502 (5)	0.0824 (6)	0.0476 (4)	−0.0035 (3)	−0.0079 (3)	0.0018 (3)
O1	0.0393 (10)	0.0575 (11)	0.0555 (11)	0.0166 (9)	0.0053 (8)	−0.0058 (8)
N1	0.0360 (11)	0.0419 (11)	0.0361 (10)	0.0096 (9)	0.0041 (8)	−0.0049 (8)
N2	0.0371 (11)	0.0385 (10)	0.0374 (10)	0.0085 (9)	0.0078 (8)	−0.0007 (8)
C1	0.0585 (17)	0.0553 (15)	0.0333 (12)	0.0032 (13)	−0.0010 (11)	−0.0035 (11)
C2	0.0422 (15)	0.0547 (15)	0.0451 (14)	−0.0037 (12)	0.0022 (11)	−0.0007 (11)
C3	0.0374 (14)	0.0517 (14)	0.0406 (13)	0.0050 (11)	0.0124 (10)	−0.0020 (10)
C4	0.0366 (13)	0.0412 (12)	0.0320 (11)	0.0071 (10)	0.0049 (9)	−0.0017 (9)
C5	0.0489 (15)	0.0581 (16)	0.0395 (13)	−0.0045 (12)	0.0150 (11)	−0.0031 (11)
C6	0.0636 (19)	0.0707 (18)	0.0363 (13)	−0.0025 (15)	0.0164 (12)	−0.0100 (12)
C7	0.0316 (13)	0.0444 (13)	0.0366 (12)	0.0043 (11)	0.0109 (9)	0.0023 (10)
C8	0.0421 (14)	0.0516 (14)	0.0366 (12)	0.0030 (11)	0.0061 (10)	−0.0022 (10)
C9	0.0455 (14)	0.0452 (13)	0.0352 (12)	0.0088 (11)	0.0033 (10)	−0.0027 (10)
C10	0.0369 (13)	0.0397 (12)	0.0420 (13)	0.0076 (10)	0.0060 (10)	0.0035 (10)
C11	0.0434 (15)	0.0474 (14)	0.0510 (15)	0.0059 (12)	0.0035 (11)	−0.0056 (11)
C12	0.0563 (18)	0.0488 (15)	0.0724 (19)	0.0063 (13)	0.0139 (15)	−0.0121 (13)
C13	0.0497 (18)	0.0458 (15)	0.091 (2)	0.0124 (13)	0.0147 (16)	0.0043 (15)
C14	0.0450 (16)	0.0559 (16)	0.081 (2)	0.0102 (14)	−0.0062 (14)	0.0127 (15)
C15	0.0516 (17)	0.0460 (14)	0.0519 (15)	0.0089 (12)	−0.0042 (12)	0.0031 (11)

Geometric parameters (Å, º)

Cl1—C1	1.746 (3)	C11—C12	1.379 (4)
Cl2—C8	1.762 (3)	C12—C13	1.380 (4)
O1—C7	1.210 (3)	C13—C14	1.361 (5)
N1—N2	1.389 (3)	C14—C15	1.383 (4)
N1—C4	1.435 (3)	C2—H2	0.9300
N1—C7	1.376 (3)	C3—H3	0.9300

N2—C9	1.271 (3)	C5—H5	0.9300
C1—C2	1.374 (4)	C6—H6	0.9300
C1—C6	1.372 (4)	C8—H8A	0.9700
C2—C3	1.383 (4)	C8—H8B	0.9700
C3—C4	1.378 (3)	C9—H9	0.9300
C4—C5	1.384 (4)	C11—H11	0.9300
C5—C6	1.376 (4)	C12—H12	0.9300
C7—C8	1.508 (4)	C13—H13	0.9300
C9—C10	1.467 (4)	C14—H14	0.9300
C10—C11	1.383 (4)	C15—H15	0.9300
C10—C15	1.388 (4)		

N2—N1—C4	123.31 (18)	C10—C15—C14	120.3 (3)
N2—N1—C7	115.78 (19)	C1—C2—H2	120.00
C4—N1—C7	120.46 (19)	C3—C2—H2	120.00
N1—N2—C9	118.9 (2)	C2—C3—H3	120.00
Cl1—C1—C2	118.9 (2)	C4—C3—H3	120.00
Cl1—C1—C6	119.6 (2)	C4—C5—H5	120.00
C2—C1—C6	121.6 (3)	C6—C5—H5	120.00
C1—C2—C3	119.2 (3)	C1—C6—H6	121.00
C2—C3—C4	120.0 (2)	C5—C6—H6	120.00
N1—C4—C3	119.8 (2)	Cl2—C8—H8A	109.00
N1—C4—C5	120.4 (2)	Cl2—C8—H8B	110.00
C3—C4—C5	119.8 (2)	C7—C8—H8A	109.00
C4—C5—C6	120.5 (3)	C7—C8—H8B	109.00
C1—C6—C5	118.9 (3)	H8A—C8—H8B	108.00
O1—C7—N1	121.0 (2)	N2—C9—H9	120.00
O1—C7—C8	124.1 (2)	C10—C9—H9	120.00
N1—C7—C8	115.0 (2)	C10—C11—H11	120.00
Cl2—C8—C7	110.76 (17)	C12—C11—H11	120.00
N2—C9—C10	120.2 (2)	C11—C12—H12	120.00
C9—C10—C11	122.5 (2)	C13—C12—H12	120.00
C9—C10—C15	118.5 (2)	C12—C13—H13	120.00
C11—C10—C15	118.9 (2)	C14—C13—H13	120.00
C10—C11—C12	120.2 (3)	C13—C14—H14	120.00
C11—C12—C13	120.3 (3)	C15—C14—H14	120.00
C12—C13—C14	119.9 (3)	C10—C15—H15	120.00
C13—C14—C15	120.4 (3)	C14—C15—H15	120.00

C4—N1—N2—C9	9.3 (3)	C2—C3—C4—C5	0.1 (4)
C7—N1—N2—C9	−178.4 (2)	N1—C4—C5—C6	−178.2 (2)
N2—N1—C4—C3	65.1 (3)	C3—C4—C5—C6	0.9 (4)
N2—N1—C4—C5	−115.8 (3)	C4—C5—C6—C1	−1.2 (4)
C7—N1—C4—C3	−106.8 (3)	O1—C7—C8—Cl2	−6.5 (3)
C7—N1—C4—C5	72.3 (3)	N1—C7—C8—Cl2	174.25 (17)
N2—N1—C7—O1	−178.6 (2)	N2—C9—C10—C11	−5.1 (4)
N2—N1—C7—C8	0.6 (3)	N2—C9—C10—C15	172.5 (2)
C4—N1—C7—O1	−6.1 (3)	C9—C10—C11—C12	177.1 (3)
C4—N1—C7—C8	173.1 (2)	C15—C10—C11—C12	−0.4 (4)

N1—N2—C9—C10	−178.7 (2)	C9—C10—C15—C14	−176.1 (3)
Cl1—C1—C2—C3	−177.9 (2)	C11—C10—C15—C14	1.5 (4)
C6—C1—C2—C3	0.6 (4)	C10—C11—C12—C13	−0.9 (4)
Cl1—C1—C6—C5	178.9 (2)	C11—C12—C13—C14	1.1 (5)
C2—C1—C6—C5	0.4 (4)	C12—C13—C14—C15	0.0 (5)
C1—C2—C3—C4	−0.8 (4)	C13—C14—C15—C10	−1.3 (4)
C2—C3—C4—N1	179.2 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.93	2.40	3.256 (3)	154

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