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N′-[(1E)-1-(4-Chlorophenyl)ethylidene]-formohydrazide

Zahid Shafiq,[a] Muhammad Yaqub,[a] M. Nawaz Tahir,[b] Mian Hasnain Nawaz[a] and M. Saeed Iqbal[c]*

[a]Department of Chemistry, Bahauddin Zakariya University, Multan-60800, Pakistan, [b]Department of Physics, University of Sargodha, Sargodha, Pakistan, and [c]Department of Chemistry, Government College University, Lahore, Pakistan
Correspondence e-mail: dmntahir_uos@yahoo.com

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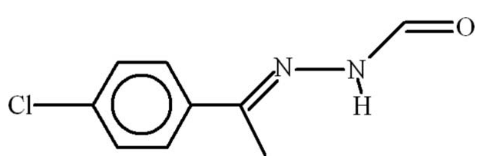
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.050; wR factor = 0.150; data-to-parameter ratio = 19.4.

The structure of the title compound, $C_9H_9ClN_2O$, consists of centrosymmetric dimers due to intermolecular N—H···O hydrogen bonding, forming $R_2^2(8)$ ring motifs. The dihedral angle between the *p*-chlorophenyl unit and the remaining heavy-atom group is 6.77 (17)°.

Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For a related structure, see: Guo (2007).



Experimental

Crystal data

$C_9H_9ClN_2O$
M_r = 196.63
Monoclinic, $P2_1/c$
a = 5.9373 (5) Å
b = 6.2178 (4) Å
c = 25.3495 (18) Å
β = 93.900 (4)°
V = 933.66 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 296 K
0.25 × 0.22 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.914, T_{max} = 0.940
9690 measured reflections
2311 independent reflections
1426 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.150
S = 1.05
2311 reflections
119 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.8600	2.0800	2.920 (3)	164.00

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2157).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Guo, H.-M. (2007). *Acta Cryst.* E**63**, o3870.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2009). E**65**, o2494 [**doi:10.1107/S1600536809037143**]

N'-[(1*E*)-1-(4-Chlorophenyl)ethylidene]formohydrazide

Z. Shafiq, M. Yaqub, M. N. Tahir, M. H. Nawaz and M. S. Iqbal

Comment

Schiff bases are important intermediates in a number of enzymatic reactions involving interaction of an enzyme with an amino or a carbonyl group of the substrate. The title compound (I, Fig. 1), has been prepared as a derivative.

The crystal structures of *N*'-(1-(4-Chlorophenyl)ethylidene)propionohydrazide (Guo, 2007) has been published which differs from the title compound (I) due to the attachment of ethyl moiety instead of H-atom with the carbonyl group.

In the title compound, due to intermolecular H-bonding the molecules are dimerized forming 8-membered $R_2^2(8)$ ring motifs (Table 1, Fig. 2) (Bernstein *et al.*, 1995). The *p*-Clorophenyl moiety A (C1—C6, Cl1) and the remaining heavy atoms group B (C8, C7, N1, N2 C9, O1) are almost planar with r.m.s. deviations of 0.007 and 0.009 Å, respectively, with a 6.77 (17)° dihedral angle between them.

Experimental

formohydrazide (1 g, 0.017 mol) was dissolved in ethanol (10 ml) and stirred. To this solution 4-Chlororoacetophenone (2.067 ml, 0.017 mol) was added dropwise and refluxed for 30 min. During refluxing precipitates were formed and the reaction mixture was further heated for 2 h. The completion of reaction was monitored by TLC. The solution was cooled to room temperature and the crued solid was collected by suction filtration. The precipitates were washed with hot ethanol, filtered and dried. The colorless prisms of title compound (I) were obtained by crystallization of the crude material in 1,4-dioxan.

Refinement

The H-atoms were positioned geometrically with N—H = 0.86, C—H = 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively and constrained to ride on their parent atoms, with U_{iso}(H) = xUeq(C,N), where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

Figures

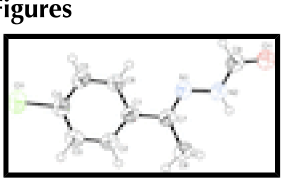

Fig. 1. View of the title compound with the atom numbering scheme. The thermal ellipsoids are drawn at the 50% probability level.

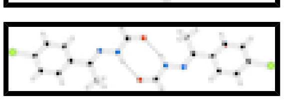

Fig. 2. The partial packing (*PLATON*; Spek, 2009) which shows that molecules are dimerized and form ring motifs.

N'-[(1*E*)-1-(4-Chlorophenyl)ethylidene]formohydrazide

Crystal data

$C_9H_9ClN_2O$	F_{000} = 408
M_r = 196.63	D_x = 1.399 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1864 reflections
a = 5.9373 (5) Å	θ = 2.3–28.0°
b = 6.2178 (4) Å	μ = 0.37 mm^{-1}
c = 25.3495 (18) Å	T = 296 K
β = 93.900 (4)°	Prismatic, colorless
V = 933.66 (12) Å^3	0.25 × 0.22 × 0.18 mm
Z = 4	

Data collection

Bruker Kappa APEXII CCD diffractometer	2311 independent reflections
Radiation source: fine-focus sealed tube	1426 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.025
Detector resolution: 7.40 pixels mm^{-1}	θ_{max} = 28.3°
T = 296 K	θ_{min} = 3.2°
ω scans	$h = -7 \rightarrow 7$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$k = -8 \rightarrow 8$
T_{min} = 0.914, T_{max} = 0.940	$l = -33 \rightarrow 32$
9690 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.050	H-atom parameters constrained
$wR(F^2)$ = 0.150	$w = 1/[\sigma^2(F_o^2) + (0.0631P)^2 + 0.2896P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ < 0.001
2311 reflections	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
119 parameters	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction coefficient: ?

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 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.38204 (13)	1.27773 (12)	0.21878 (3)	0.0806 (3)
O1	0.2494 (3)	0.0349 (3)	−0.03180 (7)	0.0740 (7)
N1	0.1941 (3)	0.4376 (3)	0.06100 (7)	0.0500 (6)
N2	0.1350 (3)	0.2578 (3)	0.03140 (7)	0.0536 (7)
C1	0.1329 (3)	0.6969 (3)	0.12510 (8)	0.0447 (7)
C2	0.0192 (4)	0.7732 (4)	0.16716 (9)	0.0611 (9)
C3	0.0928 (4)	0.9511 (4)	0.19580 (10)	0.0654 (9)
C4	0.2836 (4)	1.0560 (4)	0.18273 (9)	0.0523 (8)
C5	0.3992 (4)	0.9858 (4)	0.14095 (10)	0.0622 (9)
C6	0.3239 (4)	0.8092 (4)	0.11281 (9)	0.0593 (8)
C7	0.0599 (4)	0.5018 (3)	0.09485 (8)	0.0481 (7)
C8	−0.1579 (4)	0.3947 (4)	0.10580 (11)	0.0743 (10)
C9	0.2786 (4)	0.1908 (4)	−0.00268 (10)	0.0623 (9)
H2	−0.11039	0.70239	0.17636	0.0733*
H2A	0.00968	0.19185	0.03497	0.0644*
H3	0.01322	0.99959	0.22383	0.0785*
H5	0.52814	1.05794	0.13180	0.0746*
H6	0.40342	0.76308	0.08454	0.0712*
H9	0.41191	0.26791	−0.00468	0.0747*
H81	−0.13024	0.24613	0.11431	0.1115*
H82	−0.26116	0.40444	0.07507	0.1115*
H83	−0.22185	0.46470	0.13504	0.1115*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0897 (6)	0.0672 (5)	0.0858 (5)	−0.0185 (3)	0.0123 (4)	−0.0250 (3)
O1	0.0641 (12)	0.0777 (12)	0.0818 (12)	−0.0196 (9)	0.0177 (9)	−0.0322 (10)
N1	0.0503 (11)	0.0477 (10)	0.0521 (10)	−0.0062 (8)	0.0042 (8)	−0.0047 (8)
N2	0.0498 (11)	0.0522 (12)	0.0592 (12)	−0.0094 (9)	0.0059 (9)	−0.0090 (9)
C1	0.0426 (12)	0.0427 (11)	0.0491 (12)	−0.0028 (9)	0.0055 (9)	0.0032 (9)
C2	0.0555 (15)	0.0653 (15)	0.0647 (15)	−0.0166 (12)	0.0207 (11)	−0.0094 (11)
C3	0.0656 (17)	0.0681 (16)	0.0649 (15)	−0.0096 (13)	0.0224 (12)	−0.0138 (12)

C4	0.0548 (14)	0.0467 (12)	0.0553 (13)	−0.0031 (10)	0.0032 (10)	−0.0035 (9)
C5	0.0566 (15)	0.0567 (14)	0.0756 (16)	−0.0178 (11)	0.0215 (12)	−0.0066 (12)
C6	0.0558 (14)	0.0593 (14)	0.0658 (14)	−0.0143 (11)	0.0252 (11)	−0.0120 (11)
C7	0.0454 (12)	0.0467 (12)	0.0524 (12)	−0.0047 (10)	0.0048 (10)	0.0036 (9)
C8	0.0589 (16)	0.0718 (18)	0.0944 (19)	−0.0237 (13)	0.0209 (14)	−0.0215 (14)
C9	0.0531 (15)	0.0669 (16)	0.0677 (16)	−0.0143 (12)	0.0107 (12)	−0.0152 (12)

Geometric parameters (Å, °)

Cl1—C4	1.733 (3)	C4—C5	1.372 (3)
O1—C9	1.224 (3)	C5—C6	1.368 (3)
N1—N2	1.379 (3)	C7—C8	1.497 (3)
N1—C7	1.274 (3)	C2—H2	0.9300
N2—C9	1.322 (3)	C3—H3	0.9300
N2—H2A	0.8600	C5—H5	0.9300
C1—C2	1.384 (3)	C6—H6	0.9300
C1—C7	1.484 (3)	C8—H81	0.9600
C1—C6	1.385 (3)	C8—H82	0.9600
C2—C3	1.378 (3)	C8—H83	0.9600
C3—C4	1.367 (3)	C9—H9	0.9300
Cl1···C4^{i}	3.535 (2)	H2···C8	2.6200
O1···N2^{ii}	2.920 (3)	H2···H83	1.9000
O1···C8^{ii}	3.288 (3)	H2A···C8	2.4600
O1···C9^{iii}	3.202 (3)	H2A···H81	2.2500
O1···H2A^{ii}	2.0800	H2A···H82	2.3600
O1···H6^{iv}	2.8300	H2A···O1^{ii}	2.0800
O1···H9^{iii}	2.8600	H2A···C9^{ii}	3.0100
O1···H81^{ii}	2.7800	H5···C8^{ix}	2.9100
N2···O1^{ii}	2.920 (3)	H5···H81^{ix}	2.4100
N1···H6	2.4300	H6···N1	2.4300
N2···H81	2.7100	H6···O1^{iv}	2.8300
N2···H82	2.8200	H6···C9^{iv}	2.9100
C4···Cl1^{v}	3.535 (2)	H6···H9^{iv}	2.3700
C7···C9^{vi}	3.537 (3)	H9···O1^{iii}	2.8600
C8···O1^{ii}	3.288 (3)	H9···H6^{iv}	2.3700
C9···O1^{iii}	3.202 (3)	H81···N2	2.7100
C9···C9^{iii}	3.537 (3)	H81···C3^{x}	3.0000
C9···C7^{vi}	3.537 (3)	H81···H2A	2.2500
C2···H83	2.5000	H81···H5^{viii}	2.4100
C3···H81^{vii}	3.0000	H81···O1^{ii}	2.7800
C8···H2A	2.4600	H82···N2	2.8200
C8···H5^{viii}	2.9100	H82···H2A	2.3600
C8···H2	2.6200	H83···C2	2.5000
C9···H2A^{ii}	3.0100	H83···H2	1.9000

C9···H6^{iv}	2.9100		
N2—N1—C7	118.23 (18)	O1—C9—N2	124.9 (2)
N1—N2—C9	117.37 (18)	C1—C2—H2	119.00
C9—N2—H2A	121.00	C3—C2—H2	119.00
N1—N2—H2A	121.00	C2—C3—H3	120.00
C6—C1—C7	120.71 (19)	C4—C3—H3	120.00
C2—C1—C7	122.53 (18)	C4—C5—H5	120.00
C2—C1—C6	116.75 (19)	C6—C5—H5	120.00
C1—C2—C3	121.8 (2)	C1—C6—H6	119.00
C2—C3—C4	119.6 (2)	C5—C6—H6	119.00
Cl1—C4—C5	119.44 (19)	C7—C8—H81	109.00
Cl1—C4—C3	120.45 (19)	C7—C8—H82	109.00
C3—C4—C5	120.1 (2)	C7—C8—H83	109.00
C4—C5—C6	119.7 (2)	H81—C8—H82	109.00
C1—C6—C5	122.1 (2)	H81—C8—H83	109.00
N1—C7—C1	115.47 (19)	H82—C8—H83	109.00
N1—C7—C8	124.97 (19)	O1—C9—H9	118.00
C1—C7—C8	119.56 (19)	N2—C9—H9	118.00
C7—N1—N2—C9	−178.5 (2)	C6—C1—C2—C3	−0.5 (3)
N2—N1—C7—C8	1.0 (3)	C7—C1—C2—C3	178.1 (2)
N2—N1—C7—C1	−179.61 (17)	C6—C1—C7—C8	−174.7 (2)
N1—N2—C9—O1	−179.6 (2)	C1—C2—C3—C4	−0.2 (4)
C2—C1—C6—C5	0.6 (3)	C2—C3—C4—C5	0.8 (4)
C7—C1—C6—C5	−178.0 (2)	C2—C3—C4—Cl1	−179.02 (19)
C2—C1—C7—C8	6.7 (3)	Cl1—C4—C5—C6	179.14 (19)
C6—C1—C7—N1	5.9 (3)	C3—C4—C5—C6	−0.7 (4)
C2—C1—C7—N1	−172.7 (2)	C4—C5—C6—C1	−0.1 (4)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) −*x*, −*y*, −*z*; (iii) −*x*+1, −*y*, −*z*; (iv) −*x*+1, −*y*+1, −*z*; (v) −*x*+1, *y*−1/2, −*z*+1/2; (vi) −*x*, −*y*+1, −*z*; (vii) *x*, *y*+1, *z*; (viii) *x*−1, *y*−1, *z*; (ix) *x*+1, *y*+1, *z*; (x) *x*, *y*−1, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1^{ii}	0.8600	2.0800	2.920 (3)	164.00

Symmetry codes: (ii) −*x*, −*y*, −*z*.

Fig. 1

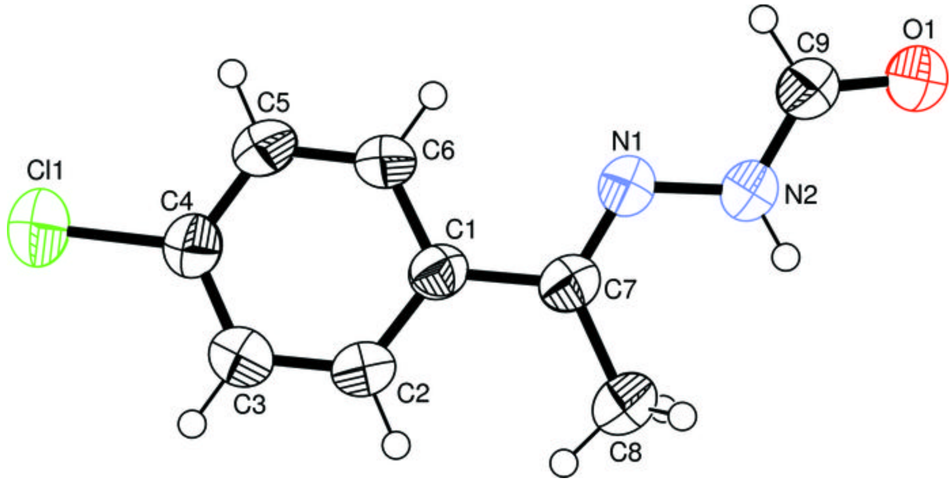

Fig. 2