Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Chloro-1-[(4-chlorophenyl)hydrazinylidene]propan-2-one

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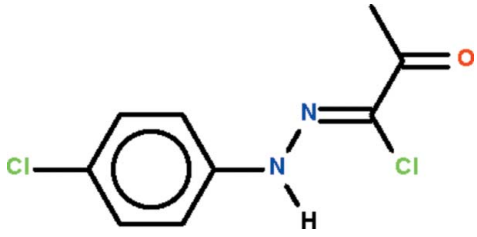
Received 29 June 2011; accepted 3 July 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.007 Å; R factor = 0.070; wR factor = 0.208; data-to-parameter ratio = 14.6.

The non-H atoms of the title compound, $C_9H_8Cl_2N_2O$, lie nearly on a plane (r.m.s. deviation = 0.110 Å), and the C=N double bond has a Z configuration. In the crystal, adjacent molecules are linked by an N—H···$O_{carbonyl}$ hydrogen bond, forming a chain running along [100].

Related literature

For the synthesis, see: Benincori *et al.* (1990); Sayed *et al.* (2002). For background to the title compound, see: Asiri *et al.* (2010).



Experimental

Crystal data

$C_9H_8Cl_2N_2O$
M_r = 231.07
Monoclinic, $P2_1/c$
a = 5.7558 (4) Å
b = 23.3282 (17) Å
c = 7.4107 (6) Å
β = 96.976 (7)°
V = 987.69 (13) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 5.65 mm^{-1}
T = 100 K
0.35 × 0.05 × 0.03 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.243, T_{max} = 0.849
3486 measured reflections
1939 independent reflections
1640 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.208
S = 1.17
1939 reflections
133 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.85 (8)	2.26 (8)	3.029 (6)	150 (7)

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank King Abdulaziz University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5260).

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supporting information

Acta Cryst. (2011). E**67**, o1962 [doi:10.1107/S1600536811026390]

1-Chloro-1-[(4-chlorophenyl)hydrazinylidene]propan-2-one

Abdullah M. Asiri, Abdulrahman O. Al-Youbi, Mohie E. M. Zayed and Seik Weng Ng

S1. Comment

We have previously reported the synthesis of ethyl (*Z*)-2-chloro-2-(2-phenylhydrazin-1-ylidene) acetate by the reaction of benzenediazonium chloride with ethyl 2-chloro-3-oxobutanoate (Asiri *et al.*, 2010). The compound is an ester. In the present study, the use of a substituted benzenediazonium chloride and the methyl ester (instead of the ethyl ester) afforded a 1-chloro-1-(arylhydrazono)-2-propanone. Such ketones are intermediates in the synthesis of pyrazoles (Sayed *et al.*, 2002) and other heterocycles (Benincori *et al.*, 1990). In the 4-chloro substituted compound (Scheme I, Fig. 1), the non-hydrogen atoms lie on a plane [r.m.s. deviation 0.110 Å] (Scheme I, Fig. 1). The C_{aryl}–N(H)–N═ C(S)═O portion adopts an extended zigzag conformation. Adjacent molecules are linked by an *N*–*H*···$O_{carbonyl}$ hydrogen bond to form a chain running [1 0 0].

S2. Experimental

To a stirred solution of methyl 2-chloro-3-oxobutanoate (1.64 g, 10 mmol) in ethanol (100 ml) was added sodium acetate trihydrate (1.30 g, 10 mmol). The mixture was chilled to 273 K and then treated with a cold solution of *p*-nitrobenzene-diazonium chloride, prepared by diazotizing *p*-chloroaniline (1.20 g, 10 mmol) dissolved in 6*M* hydrochloric acid (6 ml) with a solution of sodium nitrite (0.70 g, 10 mmol) in water (10 ml). The addition of the diazonium salt solution was carried out with rapid stirring over a period of 20 min. The reaction mixture was stirred for further 15 min. and left for 3 h in refrigerator. The resulting solid was collected by filtration and washed thoroughly with water. The crude product was crystallized from ethanol to give the corresponding hydrazonoyl chloride.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.98 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

The amino H-atom was located in a difference Fourier map, and was freely refined.

The final difference Fourier map had a peak in the vicinity of H1*b*.

Omitted from the refinement were (-3 5 1), (-3 13 1), (-3 4 2) and (-3 3 3).

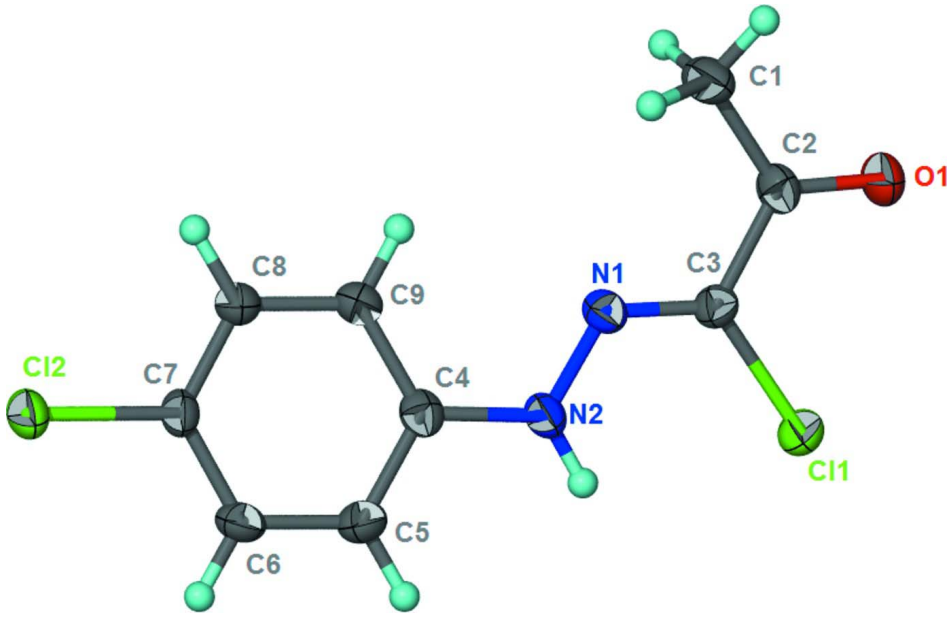

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_9H_8Cl_2N_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.7558$ (4) Å
$b = 23.3282$ (17) Å
$c = 7.4107$ (6) Å
$\beta = 96.976$ (7)°
$V = 987.69$ (13) Å^3
$Z = 4$

$F(000) = 472$
$D_x = 1.554$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 1477 reflections
$\theta = 3.8$–$74.1°$
$\mu = 5.65$ mm^{-1}
$T = 100$ K
Prism, yellow
0.35 × 0.05 × 0.03 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)

$T_{min} = 0.243$, $T_{max} = 0.849$
3486 measured reflections
1939 independent reflections
1640 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 74.3°$, $\theta_{min} = 3.8°$
$h = -6 \rightarrow 7$
$k = -27 \rightarrow 28$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.208$
$S = 1.17$
1939 reflections
133 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0903P)^2 + 4.1934P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 1.28$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0015 (7)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.2378 (2)	0.21916 (5)	0.50652 (16)	0.0233 (4)
Cl2	0.7056 (2)	0.56866 (5)	0.85227 (16)	0.0253 (4)
O1	−0.2165 (6)	0.23416 (15)	0.2828 (5)	0.0272 (8)
N1	0.1754 (7)	0.33248 (18)	0.5054 (5)	0.0194 (9)
N2	0.3787 (7)	0.33782 (18)	0.6093 (5)	0.0204 (9)
H2	0.464 (14)	0.308 (3)	0.632 (10)	0.05 (2)*
C1	−0.2428 (9)	0.3362 (2)	0.2694 (8)	0.0290 (12)
H1A	−0.3553	0.3296	0.1612	0.044*
H1B	−0.1229	0.3634	0.2402	0.044*
H1C	−0.3244	0.3520	0.3669	0.044*
C2	−0.1284 (9)	0.2803 (2)	0.3310 (7)	0.0215 (10)
C3	0.0947 (9)	0.2835 (2)	0.4510 (6)	0.0193 (10)
C4	0.4510 (8)	0.3930 (2)	0.6690 (6)	0.0197 (10)
C5	0.6761 (9)	0.4003 (2)	0.7582 (7)	0.0235 (11)
H5	0.7773	0.3682	0.7794	0.028*
C6	0.7533 (9)	0.4543 (2)	0.8162 (6)	0.0226 (10)
H6	0.9070	0.4595	0.8768	0.027*
C7	0.6036 (9)	0.5004 (2)	0.7845 (6)	0.0195 (10)
C8	0.3792 (9)	0.4940 (2)	0.6972 (7)	0.0221 (10)
H8	0.2789	0.5263	0.6771	0.027*
C9	0.3016 (8)	0.4404 (2)	0.6392 (6)	0.0209 (10)
H9	0.1474	0.4356	0.5793	0.025*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0260 (7)	0.0181 (6)	0.0248 (6)	0.0025 (4)	−0.0012 (5)	0.0005 (4)

Cl2	0.0281 (7)	0.0178 (6)	0.0279 (6)	−0.0023 (5)	−0.0050 (5)	−0.0019 (4)
O1	0.0274 (19)	0.0194 (18)	0.033 (2)	−0.0044 (15)	−0.0023 (15)	−0.0029 (15)
N1	0.019 (2)	0.021 (2)	0.0179 (18)	−0.0010 (16)	−0.0002 (15)	0.0004 (16)
N2	0.021 (2)	0.018 (2)	0.021 (2)	−0.0031 (17)	−0.0028 (16)	−0.0005 (16)
C1	0.024 (3)	0.025 (3)	0.036 (3)	−0.003 (2)	−0.007 (2)	0.002 (2)
C2	0.021 (2)	0.020 (2)	0.024 (2)	−0.0049 (19)	0.0022 (19)	−0.0011 (19)
C3	0.021 (2)	0.018 (2)	0.019 (2)	0.0010 (18)	0.0013 (18)	0.0013 (18)
C4	0.024 (2)	0.020 (2)	0.016 (2)	−0.0012 (19)	0.0014 (18)	0.0006 (18)
C5	0.022 (2)	0.023 (3)	0.024 (2)	0.005 (2)	−0.0008 (19)	0.000 (2)
C6	0.018 (2)	0.027 (3)	0.021 (2)	0.003 (2)	−0.0032 (18)	−0.001 (2)
C7	0.024 (2)	0.015 (2)	0.019 (2)	−0.0017 (18)	−0.0001 (18)	−0.0013 (17)
C8	0.020 (2)	0.020 (2)	0.025 (2)	0.0015 (19)	−0.0023 (19)	−0.0008 (19)
C9	0.018 (2)	0.023 (2)	0.021 (2)	0.0008 (19)	−0.0002 (18)	−0.0001 (19)

Geometric parameters (Å, º)

Cl1—C3	1.737 (5)	C2—C3	1.472 (7)
Cl2—C7	1.749 (5)	C4—C5	1.392 (7)
O1—C2	1.225 (6)	C4—C9	1.401 (7)
N1—C3	1.280 (6)	C5—C6	1.386 (7)
N1—N2	1.326 (6)	C5—H5	0.9500
N2—C4	1.407 (6)	C6—C7	1.382 (7)
N2—H2	0.85 (8)	C6—H6	0.9500
C1—C2	1.506 (7)	C7—C8	1.381 (7)
C1—H1A	0.9800	C8—C9	1.379 (7)
C1—H1B	0.9800	C8—H8	0.9500
C1—H1C	0.9800	C9—H9	0.9500
C3—N1—N2	121.8 (4)	C5—C4—N2	119.0 (4)
N1—N2—C4	118.4 (4)	C9—C4—N2	121.3 (4)
N1—N2—H2	119 (5)	C6—C5—C4	120.2 (5)
C4—N2—H2	123 (5)	C6—C5—H5	119.9
C2—C1—H1A	109.5	C4—C5—H5	119.9
C2—C1—H1B	109.5	C7—C6—C5	119.1 (5)
H1A—C1—H1B	109.5	C7—C6—H6	120.5
C2—C1—H1C	109.5	C5—C6—H6	120.5
H1A—C1—H1C	109.5	C6—C7—C8	121.5 (4)
H1B—C1—H1C	109.5	C6—C7—Cl2	118.7 (4)
O1—C2—C3	121.3 (5)	C8—C7—Cl2	119.8 (4)
O1—C2—C1	121.5 (4)	C9—C8—C7	119.6 (5)
C3—C2—C1	117.2 (4)	C9—C8—H8	120.2
N1—C3—C2	119.4 (4)	C7—C8—H8	120.2
N1—C3—Cl1	123.6 (4)	C8—C9—C4	119.8 (4)
C2—C3—Cl1	117.0 (4)	C8—C9—H9	120.1
C5—C4—C9	119.7 (5)	C4—C9—H9	120.1
C3—N1—N2—C4	−177.6 (4)	N2—C4—C5—C6	179.0 (4)
N2—N1—C3—C2	−178.0 (4)	C4—C5—C6—C7	0.1 (8)

N2—N1—C3—Cl1	0.4 (7)	C5—C6—C7—C8	0.2 (8)
O1—C2—C3—N1	−176.7 (5)	C5—C6—C7—Cl2	−178.2 (4)
C1—C2—C3—N1	3.6 (7)	C6—C7—C8—C9	−0.2 (8)
O1—C2—C3—Cl1	4.8 (7)	Cl2—C7—C8—C9	178.1 (4)
C1—C2—C3—Cl1	−174.9 (4)	C7—C8—C9—C4	−0.1 (7)
N1—N2—C4—C5	−171.9 (4)	C5—C4—C9—C8	0.5 (7)
N1—N2—C4—C9	7.6 (7)	N2—C4—C9—C8	−179.0 (4)
C9—C4—C5—C6	−0.5 (7)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.85 (8)	2.26 (8)	3.029 (6)	150 (7)

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