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Ethyl (*Z*)-2-chloro-2-[2-(4-methoxyphenyl)hydrazin-1-ylidene]acetate

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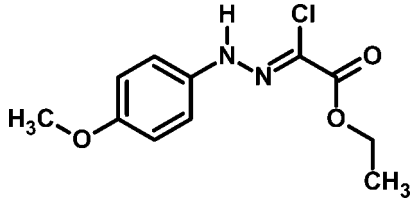
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.109; data-to-parameter ratio = 11.5.

The molecule of the title compound, $C_{11}H_{13}ClN_2O_3$, is planar (r.m.s. deviation = 0.0587 Å for non-H atoms) and adopts a Z conformation about the C=N double bond. In the crystal, molecules are linked *via* an N—H···O hydrogen bond, forming zigzag chains propagating along [010]. These chains are consolidated by C—H···O hydrogen bonds.

Related literature

For closely related structures, see: Asiri *et al.* (2011*a*,*b*). For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{11}H_{13}ClN_2O_3$
M_r = 256.68
Monoclinic, $P2_1$
a = 4.7480 (2) Å
b = 9.9256 (4) Å
c = 13.3084 (4) Å
β = 91.468 (3)°
V = 626.98 (4) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 2.71 mm^{-1}
T = 296 K
0.23 × 0.11 × 0.06 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) CCD diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.860, T_{max} = 1.000
3153 measured reflections
1824 independent reflections
1685 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.109
S = 1.07
1824 reflections
159 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), 466 Friedel pairs
Flack parameter: 0.01 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O2^{i}	0.97 (4)	2.11 (4)	3.053 (3)	164 (3)
C6—H6···O2^{i}	0.93	2.59	3.368 (3)	141

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

Data collection: *CrysAlis PRO* (Agilent, 2012); 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: *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

We would like to thank the Deanship of Scientific Research at King Abdulaziz University for the support of this research *via* a Research Group Track Grant (No. 3-102/428).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2515).

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supplementary materials

Acta Cryst. (2012). E**68**, o3274 [doi:10.1107/S1600536812044285]

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Abdullah M. Asiri, Muhammad Nadeem Arshad, Mohie E. M. Zayed, Khalid A. Alamry and Muhammad Shafiq

Comment

The present structure analysis is a continuation of our interest in related compounds already reported by our group, that is, 1-Chloro-1-[(4-methoxyphenyl)hydrazinylidene]propan-2-one (Asiri *et al.*, 2011*a*) and 1-Chloro-1-[(4-methylphenyl)-hydrazinylidene]propan-2-one (Asiri *et al.*, 2011*b*).

In the title compound, Fig. 1, the methoxy aromatic ring (C1—C6) is oriented at a dihedral angle of 3.05 (2) ° with respect to the mean plane of the ester moiety (N1/N2/C7-Cl1; planar to within 0.0 \%A). The molecule adopts a Z conformation around the C7═N2 double bond.

In the crystal, N-H···O and C-H···O hydrogen bonds connect the molecules to form zigzag chains along the *b* axis, enclosing six membered $R^1_2(6)$ ring motifs (Bernstein *et al.*, 1995) - see Table 1 and Fig. 2.

Experimental

The molecule was synthesised according to the literature procedure (Asiri *et al.*, 2011*a*) and recrystallized from ethanol giving yellow needle-like crystals.

Refinement

The NH H atom was located in a difference Fourier map and refined with U_{iso}(H) = 1.2U_{eq}(N). The C-bound H-atoms were included in calculated positions and treated as riding atoms: C-H = 0.93, 0.96 and 0.97 Å for CH(aromatic), CH_3, and CH_2 H atoms, respectively, with U_{iso}(H) = k × U_{eq}(parent C-atom), where k = 1.5 for CH_3 H atoms and = 1.2 for other H atoms.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

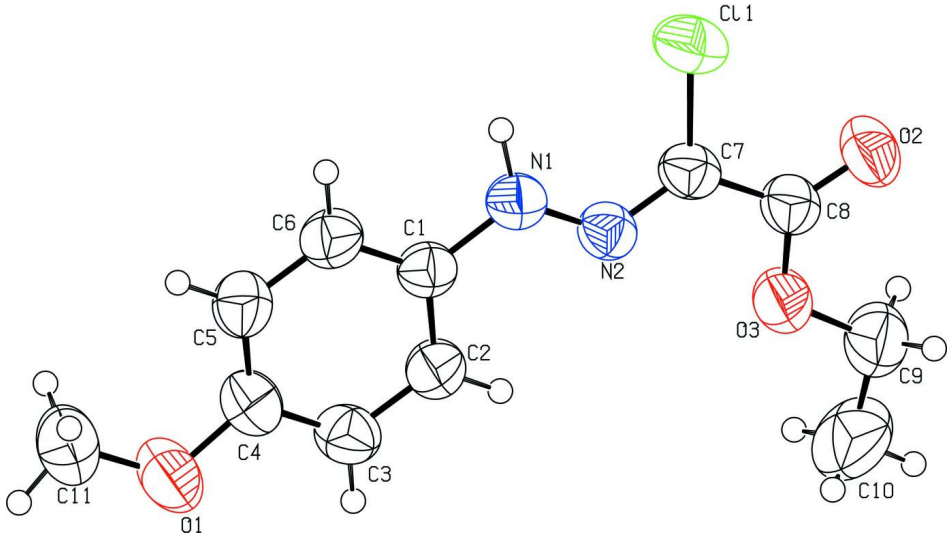

Figure 1

A view of the molecular structure of the title molecule with the atom numbering. The displacement ellipsoids are drawn at the 50% probability level.

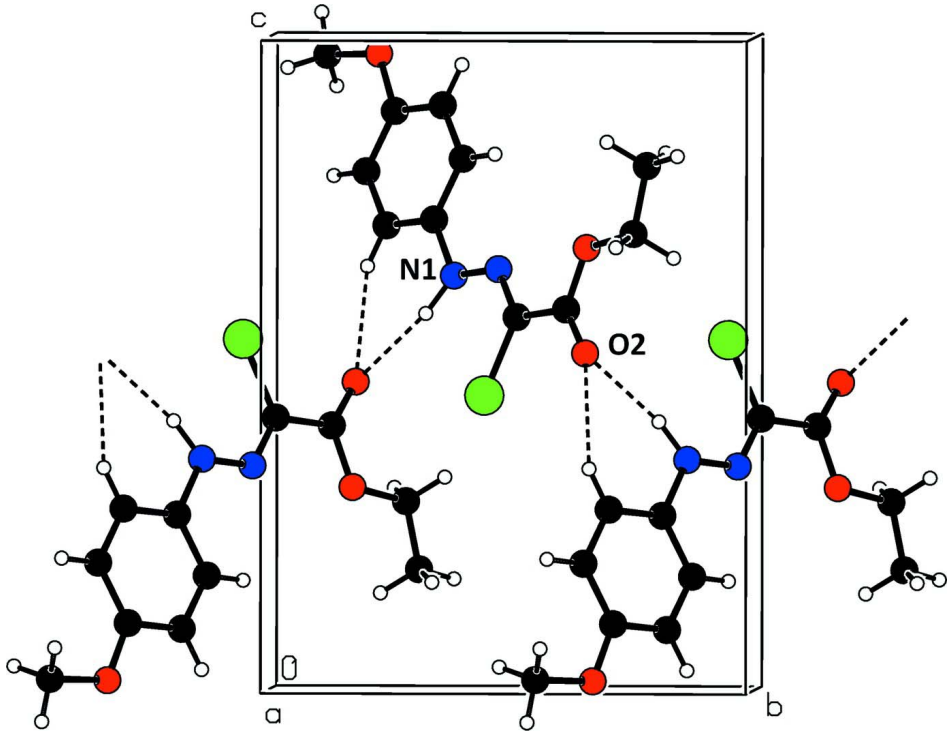

Figure 2

A partial view along the a axis of the crystal packing of the title compound. The N-H···O and C-H···O hydrogen bonds are shown as dashed lines - see Table 1 for details.

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Crystal data

$C_{11}H_{13}ClN_2O_3$
$M_r = 256.68$
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$b = 9.9256$ (4) Å
$c = 13.3084$ (4) Å
$\beta = 91.468$ (3)°
$V = 626.98$ (4) Å^3
$Z = 2$
$F(000) = 268$
$D_x = 1.360$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 2064 reflections
$\theta = 4.5$–$75.6°$
$\mu = 2.71$ mm^{-1}
$T = 296$ K
Needle, yellow
0.23 × 0.11 × 0.06 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) CCD diffractometer
Radiation source: SuperNova (Cu) X-ray Source
Mirror monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.860$, $T_{max} = 1.000$
3153 measured reflections
1824 independent reflections
1685 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 75.8°$, $\theta_{min} = 5.6°$
$h = -5 \rightarrow 5$
$k = -12 \rightarrow 9$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.109$
$S = 1.07$
1824 reflections
159 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.0434P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Absolute structure: Flack (1983), 466 Friedel pairs
Flack parameter: 0.01 (2)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
Cl1	1.03853 (17)	0.46169 (11)	0.45757 (5)	0.0873 (3)
O1	0.1393 (5)	0.2136 (3)	0.96956 (16)	0.0895 (7)

O2	1.4090 (4)	0.6817 (2)	0.52714 (16)	0.0785 (6)
O3	1.2764 (5)	0.6800 (2)	0.68691 (15)	0.0735 (6)
N1	0.7239 (5)	0.3882 (3)	0.63734 (15)	0.0607 (5)
N2	0.9085 (4)	0.4860 (2)	0.64964 (14)	0.0566 (5)
C1	0.5738 (5)	0.3420 (3)	0.72120 (18)	0.0542 (5)
C2	0.6135 (6)	0.4022 (3)	0.81488 (19)	0.0616 (6)
H2	0.7405	0.4728	0.8235	0.074*
C3	0.4620 (6)	0.3557 (3)	0.89470 (19)	0.0675 (7)
H3	0.4869	0.3960	0.9574	0.081*
C4	0.2750 (6)	0.2509 (3)	0.8836 (2)	0.0656 (7)
C5	0.2349 (6)	0.1908 (3)	0.7907 (2)	0.0666 (7)
H5	0.1083	0.1199	0.7824	0.080*
C6	0.3865 (6)	0.2378 (3)	0.7097 (2)	0.0637 (7)
H6	0.3604	0.1979	0.6469	0.076*
C7	1.0590 (6)	0.5288 (3)	0.57810 (18)	0.0584 (6)
C8	1.2652 (6)	0.6375 (3)	0.59250 (18)	0.0605 (6)
C9	1.4822 (8)	0.7834 (4)	0.7110 (2)	0.0894 (10)
H9A	1.6681	0.7533	0.6923	0.107*
H9B	1.4376	0.8647	0.6734	0.107*
C10	1.4804 (13)	0.8107 (5)	0.8152 (3)	0.1310 (18)
H10A	1.2976	0.8430	0.8329	0.197*
H10B	1.6195	0.8780	0.8315	0.197*
H10C	1.5228	0.7297	0.8520	0.197*
C11	−0.0364 (8)	0.1011 (4)	0.9655 (3)	0.0903 (11)
H11A	−0.1846	0.1157	0.9162	0.135*
H11B	−0.1166	0.0869	1.0301	0.135*
H11C	0.0709	0.0233	0.9473	0.135*
H1N	0.712 (7)	0.330 (5)	0.579 (3)	0.108*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1022 (6)	0.1004 (6)	0.0602 (4)	−0.0065 (5)	0.0182 (3)	−0.0155 (4)
O1	0.0895 (14)	0.112 (2)	0.0679 (12)	−0.0237 (15)	0.0142 (10)	0.0084 (13)
O2	0.0887 (14)	0.0810 (15)	0.0666 (11)	−0.0067 (12)	0.0164 (9)	0.0181 (10)
O3	0.0847 (13)	0.0763 (13)	0.0597 (10)	−0.0197 (11)	0.0073 (9)	0.0004 (9)
N1	0.0667 (12)	0.0627 (13)	0.0528 (11)	−0.0039 (11)	0.0032 (9)	−0.0050 (9)
N2	0.0595 (11)	0.0586 (14)	0.0518 (9)	0.0038 (10)	0.0020 (8)	0.0034 (9)
C1	0.0548 (13)	0.0542 (13)	0.0537 (12)	0.0056 (11)	0.0003 (9)	−0.0011 (10)
C2	0.0642 (14)	0.0615 (15)	0.0591 (14)	−0.0054 (13)	0.0007 (10)	−0.0026 (11)
C3	0.0701 (16)	0.0773 (19)	0.0551 (13)	−0.0018 (15)	0.0021 (11)	−0.0052 (13)
C4	0.0605 (14)	0.0745 (18)	0.0619 (15)	0.0016 (14)	0.0031 (11)	0.0094 (13)
C5	0.0634 (15)	0.0655 (17)	0.0708 (15)	−0.0077 (14)	−0.0009 (11)	0.0018 (13)
C6	0.0685 (16)	0.0640 (16)	0.0583 (14)	−0.0046 (14)	−0.0035 (11)	−0.0065 (12)
C7	0.0618 (14)	0.0601 (15)	0.0533 (12)	0.0101 (12)	0.0053 (10)	0.0017 (10)
C8	0.0687 (15)	0.0577 (15)	0.0554 (12)	0.0069 (13)	0.0042 (10)	0.0079 (11)
C9	0.106 (3)	0.083 (2)	0.0788 (19)	−0.026 (2)	0.0017 (17)	0.0007 (17)
C10	0.191 (5)	0.103 (3)	0.098 (3)	−0.042 (4)	−0.014 (3)	−0.020 (3)
C11	0.081 (2)	0.088 (3)	0.103 (2)	−0.006 (2)	0.0184 (18)	0.020 (2)

Geometric parameters (Å, º)

Cl1—C7	1.737 (3)	C3—H3	0.9300
O1—C4	1.378 (3)	C4—C5	1.381 (4)
O1—C11	1.394 (5)	C5—C6	1.393 (4)
O2—C8	1.202 (3)	C5—H5	0.9300
O3—C8	1.325 (3)	C6—H6	0.9300
O3—C9	1.447 (4)	C7—C8	1.466 (4)
N1—N2	1.315 (3)	C9—C10	1.414 (5)
N1—C1	1.416 (3)	C9—H9A	0.9700
N1—H1N	0.97 (4)	C9—H9B	0.9700
N2—C7	1.277 (3)	C10—H10A	0.9600
C1—C6	1.370 (4)	C10—H10B	0.9600
C1—C2	1.391 (3)	C10—H10C	0.9600
C2—C3	1.378 (4)	C11—H11A	0.9600
C2—H2	0.9300	C11—H11B	0.9600
C3—C4	1.373 (4)	C11—H11C	0.9600
C4—O1—C11	118.3 (3)	N2—C7—C8	122.1 (2)
C8—O3—C9	116.5 (2)	N2—C7—Cl1	122.8 (2)
N2—N1—C1	119.3 (2)	C8—C7—Cl1	115.10 (19)
N2—N1—H1N	124 (2)	O2—C8—O3	124.1 (3)
C1—N1—H1N	115 (2)	O2—C8—C7	124.3 (3)
C7—N2—N1	122.4 (2)	O3—C8—C7	111.6 (2)
C6—C1—C2	119.8 (2)	C10—C9—O3	109.4 (3)
C6—C1—N1	119.7 (2)	C10—C9—H9A	109.8
C2—C1—N1	120.5 (3)	O3—C9—H9A	109.8
C3—C2—C1	119.1 (3)	C10—C9—H9B	109.8
C3—C2—H2	120.5	O3—C9—H9B	109.8
C1—C2—H2	120.5	H9A—C9—H9B	108.2
C4—C3—C2	121.3 (2)	C9—C10—H10A	109.5
C4—C3—H3	119.4	C9—C10—H10B	109.5
C2—C3—H3	119.4	H10A—C10—H10B	109.5
C3—C4—O1	115.4 (3)	C9—C10—H10C	109.5
C3—C4—C5	119.9 (2)	H10A—C10—H10C	109.5
O1—C4—C5	124.8 (3)	H10B—C10—H10C	109.5
C4—C5—C6	119.1 (3)	O1—C11—H11A	109.5
C4—C5—H5	120.5	O1—C11—H11B	109.5
C6—C5—H5	120.5	H11A—C11—H11B	109.5
C1—C6—C5	120.9 (2)	O1—C11—H11C	109.5
C1—C6—H6	119.6	H11A—C11—H11C	109.5
C5—C6—H6	119.6	H11B—C11—H11C	109.5
C1—N1—N2—C7	177.1 (2)	C2—C1—C6—C5	−0.1 (4)
N2—N1—C1—C6	−178.3 (2)	N1—C1—C6—C5	−179.6 (3)
N2—N1—C1—C2	2.2 (4)	C4—C5—C6—C1	0.1 (4)
C6—C1—C2—C3	−0.2 (4)	N1—N2—C7—C8	−179.7 (2)
N1—C1—C2—C3	179.4 (3)	N1—N2—C7—Cl1	−0.6 (4)
C1—C2—C3—C4	0.5 (5)	C9—O3—C8—O2	−1.8 (4)
C2—C3—C4—O1	179.4 (3)	C9—O3—C8—C7	177.3 (3)

C2—C3—C4—C5	−0.4 (5)	N2—C7—C8—O2	−179.3 (3)
C11—O1—C4—C3	−175.0 (3)	Cl1—C7—C8—O2	1.5 (4)
C11—O1—C4—C5	4.9 (5)	N2—C7—C8—O3	1.6 (4)
C3—C4—C5—C6	0.1 (5)	Cl1—C7—C8—O3	−177.6 (2)
O1—C4—C5—C6	−179.7 (3)	C8—O3—C9—C10	−176.1 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2^{i}	0.97 (4)	2.11 (4)	3.053 (3)	164 (3)
C6—H6···O2^{i}	0.93	2.59	3.368 (3)	141

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