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(*Z*)-Ethyl 2-chloro-2-[2-(4-methylphenyl)-hydrazinylidene]acetate

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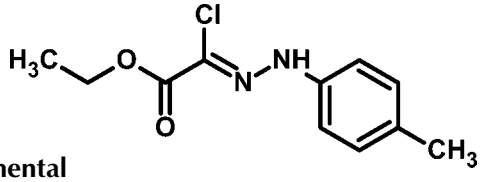
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.113; data-to-parameter ratio = 16.2.

The molecule of the title compound, $C_{11}H_{13}ClN_2O_2$, is approximately planar (r.m.s. deviation = 0.099 Å for non-H atoms) and adopts a Z conformation about the C=N double bond. In the crystal, molecules are linked by N—H···O and C—H···O hydrogen bonds to the same O-atom acceptor, forming zigzag chains propagating along [010]. These interactions give rise to $R_2^1(6)$ loops.

Related literature

For related structures, see: Asiri *et al.* (2011, 2012).



Experimental

Crystal data

$C_{11}H_{13}ClN_2O_2$
M_r = 240.68
Monoclinic, $P2_1/c$
a = 4.6152 (1) Å
b = 9.9444 (1) Å
c = 26.3152 (3) Å
β = 90.692 (1)°
V = 1207.66 (3) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.71 mm^{-1}
T = 296 K
0.41 × 0.14 × 0.13 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) CCD diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.692, T_{max} = 1.000
9526 measured reflections
2436 independent reflections
2224 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.113
S = 1.06
2436 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O1^{i}	0.93	2.52	3.331 (2)	145
N1—H1···O1^{i}	0.85 (2)	2.30 (2)	3.1120 (18)	161 (2)

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

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6984).

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supporting information

Acta Cryst. (2012). E**68**, o3420 [doi:10.1107/S1600536812046521]

(*Z*)-Ethyl 2-chloro-2-[2-(4-methylphenyl)hydrazinylidene]acetate

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S1. Comment

The present structure (I) is related to compounds already reported by our group, that is, (*Z*)-Ethyl 2-chloro-2-(2-(4-methoxyphenyl)hydrazono)acetate (II) (Asiri *et al.*, 2012) and 1-Chloro-1-[(4-methylphenyl)hydrazinylidene]propan-2-one (III) (Asiri *et al.*, 2011).

The title compound, Fig. 1, and II contain methyl and methoxy groups respectively at para positions of aromatic ring, which make them different from each other. The aromatic ring (C1—C6) is oriented at a dihedral angle of 9.49 (8)° with respect to the mean plane of the ester moiety (N1/N2/O1/O2 C7-Cl0; r.m.s. deviation 0.0454 Å), while the same angle in II is 3.05 (2) °. The spatial arrangements of different functional groups around the C7═N2 double bond give rise to trans isomer *i.e. Z* conformation.

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

S2. Experimental

The molecule was synthesised according to the literature procedure (Asiri *et al.*, 2011) and recrystallized from ethanol under slow evaporation giving yellow needles.

S3. Refinement

The N—H 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_{methyl}, and $CH_{methylene}$ H atoms, respectively, with U_{iso}(H) = k × U_{eq}(parent C-atom), where k = 1.5 for CH_{methyl} H atoms and = 1.2 for other H atoms.

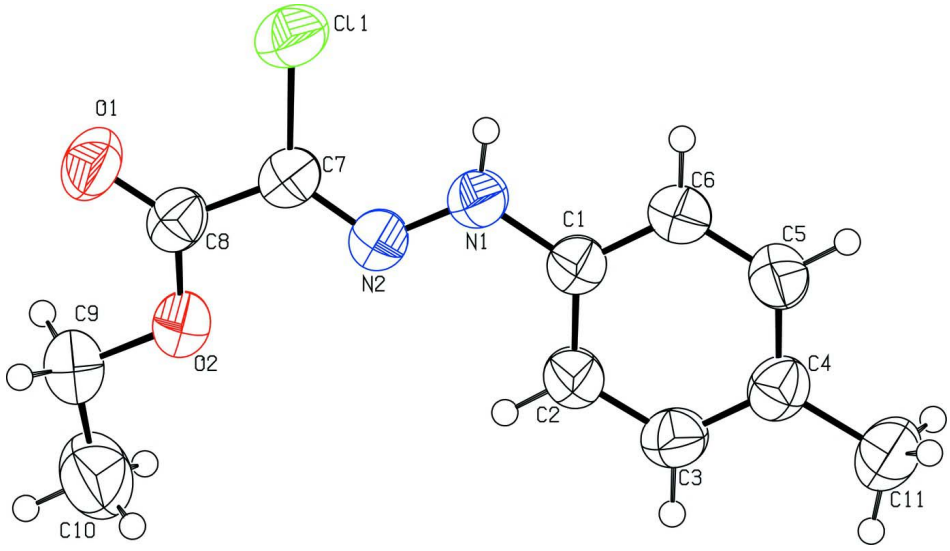

Figure 1

Molecular structure of the title molecule with displacement ellipsoids drawn at the 50% probability level.

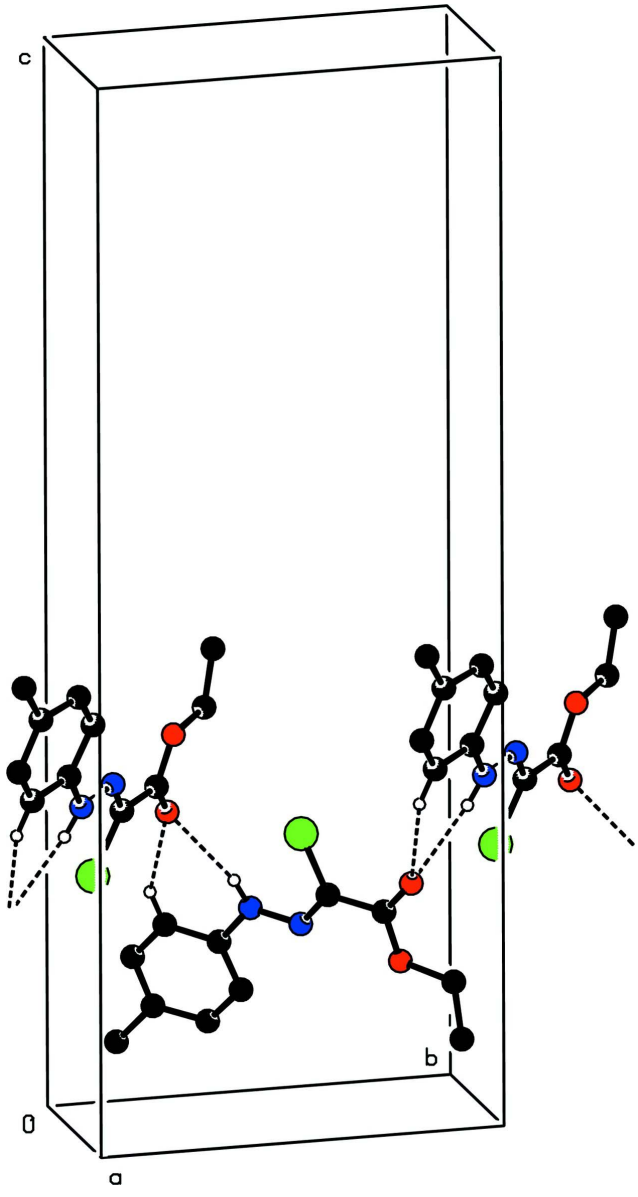

Figure 2

A perspective view of the crystal packing of the title compound showing N-H···O and C-H···O hydrogen bonds as dashed lines - see Table 1 for details.

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Crystal data

$C_{11}H_{13}ClN_2O_2$
$M_r = 240.68$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 4.6152$ (1) Å
$b = 9.9444$ (1) Å
$c = 26.3152$ (3) Å
$\beta = 90.692$ (1)°
$V = 1207.66$ (3) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.324$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 6552 reflections
$\theta = 4.4$–$75.9°$
$\mu = 2.71$ mm^{-1}

T = 296 K
Needle, yellow

0.41 × 0.14 × 0.13 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} = 0.692, T_{max} = 1.000
9526 measured reflections
2436 independent reflections
2224 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 76.1°, θ_{min} = 4.8°
h = −4→5
k = −12→12
l = −32→33

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.113
S = 1.06
2436 reflections
150 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
$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.2618P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.43464 (11)	0.05667 (5)	0.234472 (17)	0.07524 (19)
O1	0.1041 (3)	0.28559 (14)	0.27188 (5)	0.0730 (4)
O2	0.2685 (3)	0.27966 (12)	0.35220 (4)	0.0641 (3)
N1	0.8092 (3)	−0.02052 (14)	0.31951 (5)	0.0541 (3)
N2	0.6346 (3)	0.08135 (12)	0.32924 (5)	0.0508 (3)
C1	0.9873 (3)	−0.07483 (15)	0.35803 (6)	0.0491 (3)
C2	1.0086 (4)	−0.01791 (17)	0.40583 (6)	0.0579 (4)
H2	0.9035	0.0590	0.4136	0.070*
C3	1.1886 (4)	−0.07686 (18)	0.44203 (6)	0.0621 (4)
H3	1.2034	−0.0379	0.4741	0.074*
C4	1.3468 (3)	−0.19164 (17)	0.43198 (6)	0.0574 (4)
C5	1.3267 (4)	−0.24428 (17)	0.38355 (7)	0.0626 (4)

H5	1.4356	−0.3199	0.3755	0.075*
C6	1.1496 (4)	−0.18777 (17)	0.34674 (6)	0.0592 (4)
H6	1.1393	−0.2255	0.3144	0.071*
C7	0.4608 (3)	0.12591 (16)	0.29498 (6)	0.0518 (3)
C8	0.2603 (3)	0.23844 (16)	0.30433 (6)	0.0546 (4)
C9	0.0576 (5)	0.3840 (2)	0.36460 (8)	0.0774 (5)
H9A	0.1042	0.4668	0.3470	0.093*
H9B	−0.1354	0.3560	0.3541	0.093*
C10	0.0685 (7)	0.4052 (3)	0.41932 (10)	0.1069 (9)
H10A	0.0065	0.3250	0.4363	0.160*
H10B	−0.0572	0.4783	0.4281	0.160*
H10C	0.2634	0.4263	0.4297	0.160*
C11	1.5322 (5)	−0.2579 (2)	0.47260 (8)	0.0768 (5)
H11A	1.4576	−0.3460	0.4797	0.115*
H11B	1.5289	−0.2045	0.5030	0.115*
H11C	1.7278	−0.2653	0.4609	0.115*
H1	0.799 (5)	−0.063 (2)	0.2915 (10)	0.092*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0816 (3)	0.0890 (3)	0.0549 (3)	0.0032 (2)	−0.0103 (2)	−0.0051 (2)
O1	0.0795 (8)	0.0712 (8)	0.0680 (7)	0.0077 (6)	−0.0172 (6)	0.0174 (6)
O2	0.0687 (7)	0.0615 (7)	0.0618 (7)	0.0123 (5)	−0.0083 (5)	0.0025 (5)
N1	0.0542 (7)	0.0577 (7)	0.0502 (7)	0.0023 (6)	−0.0021 (6)	−0.0015 (6)
N2	0.0478 (7)	0.0507 (7)	0.0539 (7)	−0.0047 (5)	−0.0002 (5)	0.0055 (5)
C1	0.0459 (7)	0.0495 (7)	0.0520 (8)	−0.0049 (6)	0.0007 (6)	0.0022 (6)
C2	0.0621 (9)	0.0567 (8)	0.0549 (8)	0.0072 (7)	0.0012 (7)	−0.0032 (7)
C3	0.0664 (10)	0.0685 (10)	0.0513 (9)	0.0011 (8)	−0.0026 (7)	−0.0048 (7)
C4	0.0508 (8)	0.0597 (9)	0.0615 (9)	−0.0048 (7)	−0.0038 (7)	0.0064 (7)
C5	0.0611 (10)	0.0557 (9)	0.0709 (10)	0.0073 (7)	−0.0042 (8)	−0.0039 (7)
C6	0.0639 (10)	0.0575 (9)	0.0561 (9)	0.0035 (7)	−0.0023 (7)	−0.0083 (7)
C7	0.0502 (8)	0.0552 (8)	0.0499 (8)	−0.0079 (6)	−0.0021 (6)	0.0064 (6)
C8	0.0549 (8)	0.0529 (8)	0.0557 (8)	−0.0078 (6)	−0.0039 (7)	0.0122 (6)
C9	0.0837 (13)	0.0655 (11)	0.0826 (13)	0.0187 (10)	−0.0084 (10)	0.0009 (9)
C10	0.137 (2)	0.0947 (17)	0.0889 (17)	0.0374 (16)	0.0124 (15)	−0.0014 (13)
C11	0.0738 (12)	0.0788 (12)	0.0773 (12)	0.0022 (9)	−0.0163 (10)	0.0121 (10)

Geometric parameters (Å, º)

Cl1—C7	1.7377 (16)	C4—C11	1.512 (2)
O1—C8	1.2056 (19)	C5—C6	1.380 (2)
O2—C8	1.325 (2)	C5—H5	0.9300
O2—C9	1.462 (2)	C6—H6	0.9300
N1—N2	1.3215 (19)	C7—C8	1.475 (2)
N1—C1	1.405 (2)	C9—C10	1.456 (3)
N1—H1	0.85 (2)	C9—H9A	0.9700
N2—C7	1.2788 (19)	C9—H9B	0.9700

C1—C2	1.382 (2)	C10—H10A	0.9600
C1—C6	1.384 (2)	C10—H10B	0.9600
C2—C3	1.386 (2)	C10—H10C	0.9600
C2—H2	0.9300	C11—H11A	0.9600
C3—C4	1.382 (2)	C11—H11B	0.9600
C3—H3	0.9300	C11—H11C	0.9600
C4—C5	1.380 (2)		

C8—O2—C9	114.85 (13)	N2—C7—Cl1	123.01 (13)
N2—N1—C1	120.49 (13)	C8—C7—Cl1	114.63 (11)
N2—N1—H1	121.5 (16)	O1—C8—O2	124.26 (16)
C1—N1—H1	117.4 (16)	O1—C8—C7	123.24 (16)
C7—N2—N1	120.52 (14)	O2—C8—C7	112.49 (13)
C2—C1—C6	119.67 (15)	C10—C9—O2	107.95 (17)
C2—C1—N1	122.27 (14)	C10—C9—H9A	110.1
C6—C1—N1	118.05 (14)	O2—C9—H9A	110.1
C1—C2—C3	119.20 (15)	C10—C9—H9B	110.1
C1—C2—H2	120.4	O2—C9—H9B	110.1
C3—C2—H2	120.4	H9A—C9—H9B	108.4
C4—C3—C2	122.08 (16)	C9—C10—H10A	109.5
C4—C3—H3	119.0	C9—C10—H10B	109.5
C2—C3—H3	119.0	H10A—C10—H10B	109.5
C5—C4—C3	117.41 (15)	C9—C10—H10C	109.5
C5—C4—C11	121.23 (17)	H10A—C10—H10C	109.5
C3—C4—C11	121.35 (17)	H10B—C10—H10C	109.5
C6—C5—C4	121.77 (16)	C4—C11—H11A	109.5
C6—C5—H5	119.1	C4—C11—H11B	109.5
C4—C5—H5	119.1	H11A—C11—H11B	109.5
C5—C6—C1	119.83 (15)	C4—C11—H11C	109.5
C5—C6—H6	120.1	H11A—C11—H11C	109.5
C1—C6—H6	120.1	H11B—C11—H11C	109.5
N2—C7—C8	122.34 (14)		

C1—N1—N2—C7	−176.05 (13)	C2—C1—C6—C5	1.3 (2)
N2—N1—C1—C2	−6.0 (2)	N1—C1—C6—C5	−179.88 (15)
N2—N1—C1—C6	175.21 (14)	N1—N2—C7—C8	179.40 (13)
C6—C1—C2—C3	−1.3 (2)	N1—N2—C7—Cl1	1.1 (2)
N1—C1—C2—C3	179.99 (15)	C9—O2—C8—O1	3.6 (2)
C1—C2—C3—C4	−0.4 (3)	C9—O2—C8—C7	−175.40 (14)
C2—C3—C4—C5	2.0 (3)	N2—C7—C8—O1	176.63 (15)
C2—C3—C4—C11	−177.38 (17)	Cl1—C7—C8—O1	−4.9 (2)
C3—C4—C5—C6	−1.9 (3)	N2—C7—C8—O2	−4.4 (2)
C11—C4—C5—C6	177.44 (17)	Cl1—C7—C8—O2	174.06 (11)
C4—C5—C6—C1	0.3 (3)	C8—O2—C9—C10	172.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O1^{i}	0.93	2.52	3.331 (2)	145
N1—H1···O1^{i}	0.85 (2)	2.30 (2)	3.1120 (18)	161 (2)

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