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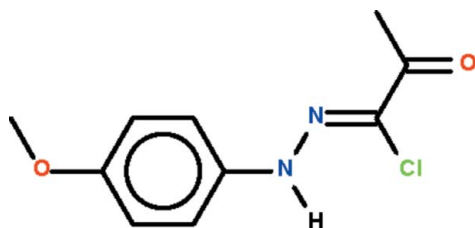
1-Chloro-1-[(4-methoxyphenyl)-hydrazinylidene]propan-2-one

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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.038; wR factor = 0.102; data-to-parameter ratio = 14.7.The non-H atoms of the title compound, $\text{C}_{10}\text{H}_{11}\text{ClN}_2\text{O}_2$, lie nearly on a plane (r.m.s. deviation = 0.150 Å), and the $\text{C}=\text{N}$ double bond has a Z configuration. In the crystal, adjacent molecules are linked by an $\text{N}-\text{H}\cdots\text{O}_{\text{carbonyl}}$ hydrogen bond, forming a chain running along [201].

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

 $\text{C}_{10}\text{H}_{11}\text{ClN}_2\text{O}_2$
 $M_r = 226.66$ Monoclinic, $P2_1/c$
 $a = 5.8873$ (3) Å $b = 25.0467$ (10) Å
 $c = 7.3041$ (3) Å
 $\beta = 99.016$ (4)°
 $V = 1063.74$ (8) Å³
 $Z = 4$ Cu $K\alpha$ radiation
 $\mu = 3.05$ mm⁻¹
 $T = 100$ K
 $0.35 \times 0.10 \times 0.05$ mm

Data collection

Agilent SuperNova Dual
diffractometer with an Atlas
detector
Absorption correction: multi-scan
(*CrysAlis PRO*; Agilent, 2010)
 $T_{\text{min}} = 0.415$, $T_{\text{max}} = 0.863$ 3802 measured reflections
2090 independent reflections
1776 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.102$
 $S = 1.05$
2090 reflections
142 parametersH atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.30$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{O1}^i$	0.87 (3)	2.22 (3)	3.021 (2)	153 (2)

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: XU5259).

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supplementary materials

Acta Cryst. (2011). E67, o1961 [doi:10.1107/S1600536811026389]

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

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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-methoxy substituted compound (Scheme I, Fig. 1), the non-hydrogen atoms lie on a plane [r.m.s. deviation 0.150 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 [2 0 1].

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*-nitrobenzenediazonium chloride, prepared by diazotizing *p*-methoxyaniline (1.23 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 hydrazoneyl chloride.

Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.98 Å, $U_{\text{iso}}(\text{H})$ 1.2 to 1.5 $U_{\text{eq}}(\text{C})$] 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.

Figures

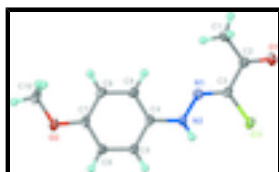


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of C₁₀H₁₁N₂O₂ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_{10}H_{11}ClN_2O_2$	$F(000) = 472$
$M_r = 226.66$	$D_x = 1.415 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 1678 reflections
$a = 5.8873 (3) \text{ \AA}$	$\theta = 3.5\text{--}74.0^\circ$
$b = 25.0467 (10) \text{ \AA}$	$\mu = 3.05 \text{ mm}^{-1}$
$c = 7.3041 (3) \text{ \AA}$	$T = 100 \text{ K}$
$\beta = 99.016 (4)^\circ$	Prism, yellow
$V = 1063.74 (8) \text{ \AA}^3$	$0.35 \times 0.10 \times 0.05 \text{ mm}$
$Z = 4$	

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector	2090 independent reflections
Radiation source: SuperNova (Cu) X-ray Source	1776 reflections with $I > 2\sigma(I)$
Mirror	$R_{\text{int}} = 0.026$
Detector resolution: $10.4041 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 74.2^\circ$, $\theta_{\text{min}} = 3.5^\circ$
ω scan	$h = -5 \rightarrow 7$
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2010)	$k = -20 \rightarrow 31$
$T_{\text{min}} = 0.415$, $T_{\text{max}} = 0.863$	$l = -8 \rightarrow 7$
3802 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.038$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.102$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.2595P]$
2090 reflections	where $P = (F_o^2 + 2F_c^2)/3$
142 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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Cl1	0.79949 (8)	0.222023 (16)	0.49001 (6)	0.02053 (15)
O1	0.3432 (2)	0.23571 (5)	0.2668 (2)	0.0248 (3)
O2	1.2583 (2)	0.53251 (5)	0.8487 (2)	0.0247 (3)
N1	0.7372 (3)	0.32750 (6)	0.4928 (2)	0.0162 (3)
N2	0.9408 (3)	0.33231 (6)	0.5974 (2)	0.0165 (3)
H2	1.023 (4)	0.3046 (10)	0.634 (4)	0.038 (7)*
C1	0.3119 (3)	0.33074 (8)	0.2551 (3)	0.0240 (4)
H1A	0.3295	0.3380	0.1263	0.036*
H1B	0.3820	0.3597	0.3348	0.036*
H1C	0.1481	0.3282	0.2646	0.036*
C2	0.4288 (3)	0.27892 (7)	0.3161 (3)	0.0184 (4)
C3	0.6546 (3)	0.28185 (7)	0.4355 (3)	0.0168 (4)
C4	1.0117 (3)	0.38372 (7)	0.6642 (2)	0.0155 (4)
C5	1.2413 (3)	0.39119 (7)	0.7442 (3)	0.0182 (4)
H5	1.3455	0.3620	0.7566	0.022*
C6	1.3153 (3)	0.44152 (7)	0.8052 (3)	0.0202 (4)
H6	1.4710	0.4467	0.8599	0.024*
C7	1.1642 (3)	0.48447 (7)	0.7872 (3)	0.0179 (4)
C8	0.9350 (3)	0.47698 (7)	0.7091 (3)	0.0185 (4)
H8	0.8307	0.5062	0.6972	0.022*
C9	0.8601 (3)	0.42636 (7)	0.6485 (3)	0.0186 (4)
H9	0.7037	0.4210	0.5960	0.022*
C10	1.1081 (4)	0.57745 (7)	0.8382 (3)	0.0242 (4)
H10A	1.1951	0.6090	0.8875	0.036*
H10B	0.9851	0.5704	0.9112	0.036*
H10C	1.0408	0.5838	0.7086	0.036*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0247 (3)	0.0108 (2)	0.0250 (3)	0.00228 (17)	0.00048 (18)	0.00008 (17)
O1	0.0238 (7)	0.0152 (7)	0.0340 (8)	-0.0046 (6)	-0.0002 (6)	-0.0032 (6)
O2	0.0255 (7)	0.0124 (6)	0.0348 (8)	-0.0011 (6)	0.0000 (6)	-0.0050 (6)
N1	0.0174 (7)	0.0134 (7)	0.0174 (8)	-0.0007 (6)	0.0016 (6)	0.0004 (6)
N2	0.0169 (8)	0.0101 (7)	0.0211 (8)	-0.0006 (6)	-0.0010 (6)	-0.0004 (6)
C1	0.0216 (10)	0.0160 (9)	0.0321 (11)	0.0011 (8)	-0.0032 (8)	-0.0006 (8)
C2	0.0185 (9)	0.0147 (9)	0.0214 (9)	-0.0011 (7)	0.0018 (7)	-0.0002 (7)
C3	0.0179 (9)	0.0107 (9)	0.0220 (9)	0.0013 (7)	0.0035 (7)	0.0007 (7)
C4	0.0190 (9)	0.0113 (8)	0.0162 (9)	-0.0007 (7)	0.0033 (7)	-0.0003 (7)
C5	0.0181 (9)	0.0120 (9)	0.0241 (10)	0.0018 (7)	0.0020 (7)	-0.0006 (7)
C6	0.0167 (9)	0.0172 (9)	0.0260 (10)	0.0009 (7)	0.0014 (7)	-0.0022 (8)
C7	0.0241 (10)	0.0116 (9)	0.0180 (9)	-0.0014 (7)	0.0035 (7)	-0.0014 (7)
C8	0.0207 (9)	0.0127 (9)	0.0221 (9)	0.0044 (7)	0.0029 (7)	0.0014 (7)
C9	0.0172 (9)	0.0169 (9)	0.0213 (9)	0.0018 (7)	0.0019 (7)	-0.0006 (8)
C10	0.0355 (11)	0.0102 (9)	0.0268 (11)	0.0010 (8)	0.0047 (9)	-0.0005 (8)

Geometric parameters (\AA , $^\circ$)

Cl1—C3	1.7395 (18)	C4—C9	1.385 (2)
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supplementary materials

O1—C2	1.224 (2)	C4—C5	1.398 (3)
O2—C7	1.371 (2)	C5—C6	1.384 (3)
O2—C10	1.426 (2)	C5—H5	0.9500
N1—C3	1.287 (2)	C6—C7	1.389 (3)
N1—N2	1.322 (2)	C6—H6	0.9500
N2—C4	1.417 (2)	C7—C8	1.393 (3)
N2—H2	0.87 (3)	C8—C9	1.392 (3)
C1—C2	1.503 (2)	C8—H8	0.9500
C1—H1A	0.9800	C9—H9	0.9500
C1—H1B	0.9800	C10—H10A	0.9800
C1—H1C	0.9800	C10—H10B	0.9800
C2—C3	1.473 (3)	C10—H10C	0.9800
C7—O2—C10	117.36 (15)	C6—C5—H5	120.3
C3—N1—N2	122.09 (16)	C4—C5—H5	120.3
N1—N2—C4	118.27 (15)	C5—C6—C7	120.67 (18)
N1—N2—H2	121.2 (18)	C5—C6—H6	119.7
C4—N2—H2	120.3 (18)	C7—C6—H6	119.7
C2—C1—H1A	109.5	O2—C7—C6	115.38 (17)
C2—C1—H1B	109.5	O2—C7—C8	124.65 (17)
H1A—C1—H1B	109.5	C6—C7—C8	119.96 (17)
C2—C1—H1C	109.5	C9—C8—C7	119.43 (17)
H1A—C1—H1C	109.5	C9—C8—H8	120.3
H1B—C1—H1C	109.5	C7—C8—H8	120.3
O1—C2—C3	120.66 (17)	C4—C9—C8	120.53 (18)
O1—C2—C1	121.88 (18)	C4—C9—H9	119.7
C3—C2—C1	117.45 (16)	C8—C9—H9	119.7
N1—C3—C2	119.81 (16)	O2—C10—H10A	109.5
N1—C3—C11	122.97 (15)	O2—C10—H10B	109.5
C2—C3—C11	117.21 (13)	H10A—C10—H10B	109.5
C9—C4—C5	119.98 (17)	O2—C10—H10C	109.5
C9—C4—N2	121.38 (17)	H10A—C10—H10C	109.5
C5—C4—N2	118.63 (16)	H10B—C10—H10C	109.5
C6—C5—C4	119.42 (17)		
C3—N1—N2—C4	-175.92 (16)	C4—C5—C6—C7	-0.2 (3)
N2—N1—C3—C2	-178.79 (16)	C10—O2—C7—C6	-178.14 (16)
N2—N1—C3—C11	0.3 (3)	C10—O2—C7—C8	2.5 (3)
O1—C2—C3—N1	-176.92 (18)	C5—C6—C7—O2	-178.62 (17)
C1—C2—C3—N1	4.3 (3)	C5—C6—C7—C8	0.8 (3)
O1—C2—C3—C11	3.9 (3)	O2—C7—C8—C9	178.87 (17)
C1—C2—C3—C11	-174.89 (14)	C6—C7—C8—C9	-0.5 (3)
N1—N2—C4—C9	10.9 (3)	C5—C4—C9—C8	1.1 (3)
N1—N2—C4—C5	-167.94 (16)	N2—C4—C9—C8	-177.73 (17)
C9—C4—C5—C6	-0.7 (3)	C7—C8—C9—C4	-0.5 (3)
N2—C4—C5—C6	178.08 (17)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
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N2—H2···O1ⁱ 0.87 (3) 2.22 (3) 3.021 (2) 153 (2)
 Symmetry codes: (i) $x+1, -y+1/2, z+1/2$.

Fig. 1

