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

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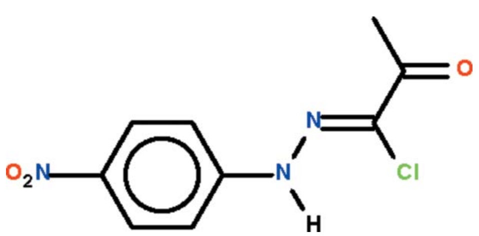
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.152; data-to-parameter ratio = 14.0.

The non-H atoms of the title compound, $C_9H_8ClN_3O_3$, lie approximately on a plane (r.m.s. deviation = 0.111 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 [101].

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

$C_9H_8ClN_3O_3$
M_r = 241.63
Monoclinic, $P2_1/n$
a = 7.0628 (3) Å
b = 13.4182 (5) Å
c = 11.2884 (5) Å
β = 95.589 (4)°
V = 1064.72 (8) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 3.19 mm^{-1}
T = 100 K
0.20 × 0.10 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010) T_{min} = 0.568, T_{max} = 0.857
4113 measured reflections
2105 independent reflections
1839 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.152
S = 1.09
2105 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 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 (4)	2.26 (4)	3.000 (3)	145 (3)

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

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: XU5261).

References

Agilent (2010). *CrysAlis PRO*. Agilent Technologies, Yarnton, Oxfordshire, England.
Asiri, A. M., Zayed, M. E. M. & Ng, S. W. (2010). *Acta Cryst.* E**66**, o2374.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Benincori, T., Fusco, R. & Sannicolo, F. (1990). *Gazz. Chim. Ital.* **120**, 635–659.
Sayed, S. M., Khalil, M. A., Ahmed, M. A. & Raslan, M. A. (2002). *Synth. Commun.* **32**, 481–495.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2011). E**67**, o1963 [**doi:10.1107/S1600536811026407**]

1-Chloro-1-[(4-nitrophenyl)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-nitro substituted compound (Scheme I, Fig. 1), the non-hydrogen atoms lie on a plane [r.m.s. deviation 0.111 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 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*-nitrobenzene-diazonium chloride, prepared by diazotizing *p*-nitroaniline (1.38 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.

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 H6.

Figures

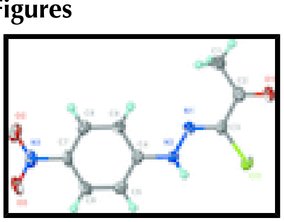

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_9H_8ClN_3O_3$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_9H_8ClN_3O_3$	$F(000) = 496$
$M_r = 241.63$	$D_x = 1.507$ Mg m^{-3}
Monoclinic, $P2_1/n$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Hall symbol: -P 2yn	Cell parameters from 1647 reflections
$a = 7.0628$ (3) Å	$\theta = 3.3$–$74.3°$
$b = 13.4182$ (5) Å	$\mu = 3.19$ mm^{-1}
$c = 11.2884$ (5) Å	$T = 100$ K
$\beta = 95.589$ (4)°	Prism, yellow
$V = 1064.72$ (8) Å^3	0.20 × 0.10 × 0.05 mm
$Z = 4$	

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector	2105 independent reflections
Radiation source: SuperNova (Cu) X-ray Source	1839 reflections with $I > 2\sigma(I)$
Mirror	$R_{int} = 0.020$
Detector resolution: 10.4041 pixels mm^{-1}	$\theta_{max} = 74.4°$, $\theta_{min} = 5.1°$
ω scans	$h = -8 \rightarrow 5$
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)	$k = -16 \rightarrow 16$
$T_{min} = 0.568$, $T_{max} = 0.857$	$l = -14 \rightarrow 14$
4113 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
$R[F^2 > 2\sigma(F^2)] = 0.054$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.152$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.09$	$w = 1/[\sigma^2(F_o^2) + (0.0849P)^2 + 0.8946P]$ where $P = (F_o^2 + 2F_c^2)/3$
2105 reflections	$(\Delta/\sigma)_{max} = 0.001$
150 parameters	$\Delta\rho_{max} = 1.21$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.48$ e Å^{-3}

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

	x	y	z	$U_{iso}*/U_{eq}$

Cl1	0.63780 (10)	0.29860 (4)	0.39614 (6)	0.0299 (2)
O1	0.4446 (3)	0.25504 (13)	0.15943 (16)	0.0275 (4)
O2	0.8827 (3)	−0.34157 (14)	0.6658 (2)	0.0369 (5)
O3	1.0132 (3)	−0.25969 (16)	0.81885 (18)	0.0383 (5)
N1	0.6623 (3)	0.10048 (16)	0.37953 (18)	0.0213 (5)
N2	0.7385 (3)	0.09908 (17)	0.49160 (19)	0.0226 (5)
H2	0.753 (5)	0.154 (3)	0.528 (3)	0.042 (10)*
N3	0.9267 (3)	−0.26356 (18)	0.7182 (2)	0.0300 (5)
C1	0.5294 (4)	0.0846 (2)	0.1360 (2)	0.0304 (6)
H1A	0.4572	0.0912	0.0578	0.046*
H1B	0.4743	0.0311	0.1808	0.046*
H1C	0.6622	0.0686	0.1258	0.046*
C2	0.5211 (4)	0.1804 (2)	0.2026 (2)	0.0251 (6)
C3	0.6129 (4)	0.18280 (19)	0.3275 (2)	0.0224 (5)
C4	0.7818 (3)	0.00828 (19)	0.5472 (2)	0.0209 (5)
C5	0.8644 (4)	0.0084 (2)	0.6644 (2)	0.0246 (5)
H5	0.8885	0.0696	0.7054	0.030*
C6	0.9115 (4)	−0.0816 (2)	0.7210 (2)	0.0250 (5)
H6	0.9677	−0.0825	0.8009	0.030*
C7	0.8754 (4)	−0.16926 (19)	0.6594 (2)	0.0237 (5)
C8	0.7916 (4)	−0.17086 (19)	0.5427 (2)	0.0241 (5)
H8	0.7668	−0.2324	0.5026	0.029*
C9	0.7449 (4)	−0.08185 (19)	0.4860 (2)	0.0229 (5)
H9	0.6882	−0.0815	0.4061	0.027*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0424 (4)	0.0166 (4)	0.0299 (4)	0.0005 (2)	0.0002 (3)	0.0006 (2)
O1	0.0353 (10)	0.0225 (9)	0.0246 (9)	0.0050 (8)	0.0026 (8)	0.0069 (7)
O2	0.0469 (12)	0.0195 (10)	0.0448 (12)	0.0028 (9)	0.0070 (10)	0.0060 (9)
O3	0.0513 (13)	0.0354 (11)	0.0272 (10)	0.0140 (10)	−0.0015 (9)	0.0142 (9)
N1	0.0244 (10)	0.0193 (10)	0.0202 (10)	0.0008 (8)	0.0019 (8)	0.0023 (8)
N2	0.0300 (11)	0.0183 (10)	0.0195 (10)	0.0001 (9)	0.0014 (8)	−0.0007 (9)
N3	0.0329 (12)	0.0262 (12)	0.0322 (12)	0.0067 (10)	0.0099 (10)	0.0094 (10)
C1	0.0410 (15)	0.0266 (14)	0.0233 (12)	0.0013 (12)	0.0021 (11)	−0.0004 (11)
C2	0.0279 (12)	0.0229 (12)	0.0253 (13)	0.0009 (10)	0.0066 (10)	0.0058 (10)
C3	0.0265 (12)	0.0169 (11)	0.0244 (12)	0.0022 (10)	0.0062 (10)	0.0026 (10)
C4	0.0214 (11)	0.0179 (12)	0.0242 (12)	0.0006 (9)	0.0060 (9)	0.0034 (10)
C5	0.0291 (12)	0.0206 (13)	0.0245 (12)	−0.0018 (10)	0.0046 (10)	−0.0028 (10)
C6	0.0259 (12)	0.0297 (14)	0.0193 (11)	0.0032 (10)	0.0016 (9)	0.0044 (10)
C7	0.0266 (12)	0.0197 (13)	0.0254 (13)	0.0052 (10)	0.0059 (10)	0.0097 (10)
C8	0.0287 (12)	0.0186 (12)	0.0259 (13)	−0.0007 (10)	0.0071 (10)	−0.0007 (10)
C9	0.0275 (12)	0.0198 (12)	0.0214 (12)	−0.0009 (10)	0.0027 (9)	0.0002 (10)

Geometric parameters (Å, °)

Cl1—C3	1.737 (3)	C1—H1C	0.9800
O1—C2	1.217 (3)	C2—C3	1.494 (4)

O2—N3	1.227 (3)	C4—C5	1.393 (4)
O3—N3	1.238 (3)	C4—C9	1.405 (4)
N1—C3	1.283 (3)	C5—C6	1.391 (4)
N1—N2	1.326 (3)	C5—H5	0.9500
N2—C4	1.391 (3)	C6—C7	1.377 (4)
N2—H2	0.85 (4)	C6—H6	0.9500
N3—C7	1.458 (3)	C7—C8	1.391 (4)
C1—C2	1.493 (4)	C8—C9	1.380 (4)
C1—H1A	0.9800	C8—H8	0.9500
C1—H1B	0.9800	C9—H9	0.9500

C3—N1—N2	121.0 (2)	N2—C4—C5	118.7 (2)
N1—N2—C4	119.6 (2)	N2—C4—C9	120.7 (2)
N1—N2—H2	118 (3)	C5—C4—C9	120.6 (2)
C4—N2—H2	122 (3)	C6—C5—C4	119.6 (2)
O2—N3—O3	123.9 (2)	C6—C5—H5	120.2
O2—N3—C7	118.7 (2)	C4—C5—H5	120.2
O3—N3—C7	117.4 (2)	C7—C6—C5	119.1 (2)
C2—C1—H1A	109.5	C7—C6—H6	120.5
C2—C1—H1B	109.5	C5—C6—H6	120.5
H1A—C1—H1B	109.5	C6—C7—C8	122.1 (2)
C2—C1—H1C	109.5	C6—C7—N3	119.1 (2)
H1A—C1—H1C	109.5	C8—C7—N3	118.8 (2)
H1B—C1—H1C	109.5	C9—C8—C7	119.1 (2)
O1—C2—C1	123.0 (2)	C9—C8—H8	120.4
O1—C2—C3	119.7 (2)	C7—C8—H8	120.4
C1—C2—C3	117.3 (2)	C8—C9—C4	119.5 (2)
N1—C3—C2	119.1 (2)	C8—C9—H9	120.2
N1—C3—Cl1	123.7 (2)	C4—C9—H9	120.2
C2—C3—Cl1	117.12 (18)		

C3—N1—N2—C4	176.8 (2)	C5—C6—C7—C8	−0.6 (4)
N2—N1—C3—C2	−177.8 (2)	C5—C6—C7—N3	179.3 (2)
N2—N1—C3—Cl1	−0.4 (3)	O2—N3—C7—C6	175.2 (2)
O1—C2—C3—N1	167.5 (2)	O3—N3—C7—C6	−4.9 (4)
C1—C2—C3—N1	−12.9 (4)	O2—N3—C7—C8	−4.9 (4)
O1—C2—C3—Cl1	−10.0 (3)	O3—N3—C7—C8	175.0 (2)
C1—C2—C3—Cl1	169.63 (19)	C6—C7—C8—C9	0.8 (4)
N1—N2—C4—C5	179.0 (2)	N3—C7—C8—C9	−179.1 (2)
N1—N2—C4—C9	−0.3 (3)	C7—C8—C9—C4	−0.3 (4)
N2—C4—C5—C6	−179.0 (2)	N2—C4—C9—C8	179.2 (2)
C9—C4—C5—C6	0.3 (4)	C5—C4—C9—C8	−0.2 (4)
C4—C5—C6—C7	0.1 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1[i]	0.85 (4)	2.26 (4)	3.000 (3)	145 (3)

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

Fig. 1