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1-[1-(4-Chlorophenyl)ethylidene]-carbonohydrazide

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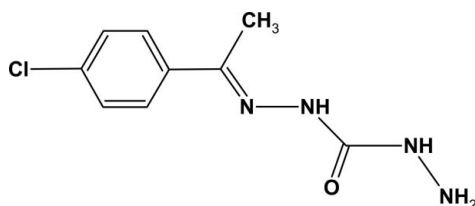
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.045; wR factor = 0.126; data-to-parameter ratio = 13.4.

The molecular skeleton of the title molecule, $\text{C}_9\text{H}_{11}\text{ClN}_4\text{O}$, is essentially planar, the dihedral angle between the ring and the and N/N/C plane being $6.7(3)^\circ$. In the crystal, intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds link the molecules into ribbons propagated along [010].

Related literature

For the biological activity of carbonohydrazide derivatives, see: Loncle *et al.* (2004). For related structures, see Meyers *et al.* (1995).



Experimental

Crystal data

$\text{C}_9\text{H}_{11}\text{ClN}_4\text{O}$
 $M_r = 226.67$
Monoclinic, $P2_1/c$
 $a = 14.6429(14)$ Å

$b = 9.6041(12)$ Å
 $c = 7.4327(9)$ Å
 $\beta = 90.102(1)^\circ$
 $V = 1045.3(2)$ Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.34$ mm⁻¹

$T = 298$ K
 $0.40 \times 0.30 \times 0.12$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.875$, $T_{\max} = 0.960$

4419 measured reflections
1837 independent reflections
1085 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.126$
 $S = 1.01$
1837 reflections

137 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{N4}^{\text{i}}$	0.86	2.24	3.024 (3)	152
$\text{N3}-\text{H3}\cdots\text{O1}^{\text{ii}}$	0.86	2.09	2.850 (3)	147
$\text{N4}-\text{H4A}\cdots\text{O1}^{\text{iii}}$	0.89	2.34	3.206 (3)	164

Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2587).

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supplementary materials

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1-[1-(4-Chlorophenyl)ethylidene]carbonohydrazide

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Comment

A number of carbonohydrazide derivatives have been claimed to possess a bioactivity such as antibacterial, antifungal, anticonvulsant and anticancer activities (Loncle *et al.*, 2004). We describe in this paper a user-friendly, solvent-free protocol for the synthesis of substituted carbonohydrazide starting from the fragrant ketones and carbohydrazide under solvent-free conditions in this paper. Using this method, which can be considered as a general method for the synthesis of substituted carbonohydrazides, we obtained the title compound, (I). We present here its crystal structure.

In (I) (Fig. 1), the bond lengths and angles are normal and correspond to those observed in bis(3-fluorophenylmethine)carbonohydrazide (Meyers *et al.*, 1995). The N4/N3/C1 and N2/N1/C1 planes form a dihedral angle of 4.09 (4)°, while ring C4-C9 and N2/N1/C1 plane form a dihedral angle of 2.64 (29)°.

In the crystal, intermolecular N—H···O and N—H···N hydrogen bonds (Table 1) link the molecules into ribbons propagated in direction [010].

Experimental

p-Chloroacetophenone (5.0 mmol) and carbohydrazide (5.0 mmol) were mixed in 50 ml flask under solvent-free conditions. After stirring 3 h at 373 K, the resulting mixture was cooled to room temperature, and recrystallized from ethanol, and afforded the title compound as a crystalline solid. Elemental analysis: calculated for C₉H₁₁ClN₄O: C 47.69, H 4.89, N 24.72%; found: C 47.63, H 4.75, N 24.64%.

Refinement

All H atoms were placed in geometrically idealized positions (N—H 0.86 and C—H = 0.93–0.96 Å) and treated as riding on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}(\text{C})$ (C,N).

Figures

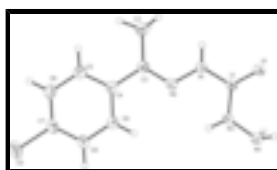


Fig. 1. The molecular structure of (I) showing the atomic numbering scheme and 30% probability displacement ellipsoids.

1-[1-(4-Chlorophenyl)ethylidene]carbonohydrazide

Crystal data

$C_9H_{11}ClN_4O$	$F_{000} = 472$
$M_r = 226.67$	$D_x = 1.440 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 14.6429 (14) \text{ \AA}$	Cell parameters from 963 reflections
$b = 9.6041 (12) \text{ \AA}$	$\theta = 2.5\text{--}22.7^\circ$
$c = 7.4327 (9) \text{ \AA}$	$\mu = 0.34 \text{ mm}^{-1}$
$\beta = 90.1020 (10)^\circ$	$T = 298 \text{ K}$
$V = 1045.3 (2) \text{ \AA}^3$	Needle, colourless
$Z = 4$	$0.40 \times 0.30 \times 0.12 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1837 independent reflections
Radiation source: fine-focus sealed tube	1085 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.037$
$T = 298 \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.4^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -11 \rightarrow 17$
$T_{\text{min}} = 0.875$, $T_{\text{max}} = 0.960$	$k = -11 \rightarrow 11$
4419 measured reflections	$l = -7 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H-atom parameters constrained
$wR(F^2) = 0.126$	$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.3562P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
1837 reflections	$(\Delta/\sigma)_{\text{max}} = 0.002$
137 parameters	$\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
	Extinction correction: none

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.

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.94869 (7)	1.35122 (11)	0.60169 (13)	0.0725 (4)
N1	0.57886 (16)	0.7625 (2)	0.3769 (3)	0.0405 (7)
H1	0.5945	0.6791	0.4057	0.049*
N2	0.63579 (17)	0.8715 (2)	0.4127 (3)	0.0375 (6)
N3	0.47946 (16)	0.9177 (2)	0.2451 (3)	0.0429 (7)
H3	0.5175	0.9828	0.2719	0.051*
N4	0.39947 (16)	0.9500 (2)	0.1494 (3)	0.0434 (7)
H4A	0.4001	0.9058	0.0443	0.065*
H4B	0.3513	0.9224	0.2129	0.065*
O1	0.44329 (14)	0.6900 (2)	0.2677 (3)	0.0460 (6)
C1	0.4970 (2)	0.7867 (3)	0.2949 (4)	0.0358 (7)
C2	0.7519 (2)	0.7025 (3)	0.5085 (4)	0.0518 (9)
H2A	0.7062	0.6479	0.5682	0.078*
H2B	0.8056	0.7075	0.5825	0.078*
H2C	0.7669	0.6601	0.3955	0.078*
C3	0.7158 (2)	0.8469 (3)	0.4762 (4)	0.0357 (7)
C4	0.77306 (19)	0.9705 (3)	0.5105 (4)	0.0353 (7)
C5	0.8596 (2)	0.9605 (3)	0.5861 (4)	0.0458 (8)
H5	0.8821	0.8733	0.6173	0.055*
C6	0.9131 (2)	1.0768 (4)	0.6161 (4)	0.0511 (9)
H6	0.9710	1.0676	0.6663	0.061*
C7	0.8802 (2)	1.2056 (3)	0.5714 (4)	0.0443 (8)
C8	0.7950 (2)	1.2196 (4)	0.4976 (4)	0.0513 (9)
H8	0.7729	1.3074	0.4678	0.062*
C9	0.7423 (2)	1.1031 (3)	0.4676 (4)	0.0475 (9)
H9	0.6845	1.1136	0.4173	0.057*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0749 (7)	0.0666 (7)	0.0758 (7)	-0.0359 (5)	-0.0187 (5)	0.0017 (5)
N1	0.0364 (16)	0.0234 (14)	0.0618 (18)	0.0002 (12)	-0.0087 (13)	0.0031 (12)
N2	0.0367 (15)	0.0287 (15)	0.0471 (15)	-0.0058 (12)	-0.0022 (12)	-0.0013 (11)
N3	0.0377 (16)	0.0238 (15)	0.0671 (18)	-0.0020 (12)	-0.0125 (13)	0.0000 (12)
N4	0.0361 (15)	0.0335 (15)	0.0605 (17)	0.0027 (12)	-0.0060 (12)	0.0004 (12)
O1	0.0397 (13)	0.0231 (12)	0.0753 (16)	-0.0055 (10)	-0.0085 (11)	0.0006 (10)
C1	0.0372 (19)	0.0216 (18)	0.0488 (19)	0.0004 (14)	0.0016 (15)	-0.0018 (14)

supplementary materials

C2	0.055 (2)	0.043 (2)	0.058 (2)	0.0051 (18)	-0.0117 (17)	-0.0020 (16)
C3	0.0360 (19)	0.0370 (18)	0.0342 (16)	0.0020 (15)	-0.0013 (14)	-0.0022 (14)
C4	0.0332 (18)	0.0379 (19)	0.0349 (17)	-0.0019 (14)	-0.0016 (14)	-0.0011 (14)
C5	0.042 (2)	0.044 (2)	0.051 (2)	-0.0002 (17)	-0.0057 (16)	0.0064 (16)
C6	0.041 (2)	0.061 (3)	0.052 (2)	-0.0070 (19)	-0.0125 (16)	0.0029 (18)
C7	0.045 (2)	0.045 (2)	0.0431 (19)	-0.0125 (17)	-0.0041 (16)	-0.0033 (15)
C8	0.052 (2)	0.037 (2)	0.065 (2)	-0.0075 (17)	-0.0142 (18)	0.0026 (16)
C9	0.0366 (19)	0.042 (2)	0.064 (2)	-0.0034 (16)	-0.0142 (16)	0.0021 (17)

Geometric parameters (Å, °)

C1—C7	1.735 (3)	C2—H2B	0.9600
N1—C1	1.364 (4)	C2—H2C	0.9600
N1—N2	1.364 (3)	C3—C4	1.476 (4)
N1—H1	0.8600	C4—C5	1.388 (4)
N2—C3	1.285 (4)	C4—C9	1.387 (4)
N3—C1	1.336 (3)	C5—C6	1.382 (4)
N3—N4	1.404 (3)	C5—H5	0.9300
N3—H3	0.8600	C6—C7	1.368 (4)
N4—H4A	0.8900	C6—H6	0.9300
N4—H4B	0.8900	C7—C8	1.369 (4)
O1—C1	1.234 (3)	C8—C9	1.378 (4)
C2—C3	1.502 (4)	C8—H8	0.9300
C2—H2A	0.9600	C9—H9	0.9300
C1—N1—N2	119.5 (2)	N2—C3—C2	123.3 (3)
C1—N1—H1	120.2	C4—C3—C2	121.1 (3)
N2—N1—H1	120.3	C5—C4—C9	116.9 (3)
C3—N2—N1	119.1 (2)	C5—C4—C3	122.1 (3)
C1—N3—N4	120.5 (2)	C9—C4—C3	121.0 (3)
C1—N3—H3	119.7	C6—C5—C4	121.7 (3)
N4—N3—H3	119.7	C6—C5—H5	119.1
N3—N4—H4A	109.2	C4—C5—H5	119.1
N3—N4—H4B	109.1	C7—C6—C5	119.5 (3)
H4A—N4—H4B	109.5	C7—C6—H6	120.2
O1—C1—N3	122.8 (3)	C5—C6—H6	120.2
O1—C1—N1	120.3 (3)	C6—C7—C8	120.4 (3)
N3—C1—N1	116.9 (3)	C6—C7—C11	119.6 (2)
C3—C2—H2A	109.5	C8—C7—C11	119.9 (3)
C3—C2—H2B	109.5	C7—C8—C9	119.7 (3)
H2A—C2—H2B	109.5	C7—C8—H8	120.2
C3—C2—H2C	109.5	C9—C8—H8	120.2
H2A—C2—H2C	109.5	C8—C9—C4	121.8 (3)
H2B—C2—H2C	109.5	C8—C9—H9	119.1
N2—C3—C4	115.6 (3)	C4—C9—H9	119.1

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots N4 ⁱ	0.86	2.24	3.024 (3)	152

N3—H3···O1 ⁱⁱ	0.86	2.09	2.850 (3)	147
N4—H4A···O1 ⁱⁱⁱ	0.89	2.34	3.206 (3)	164

Symmetry codes: (i) $-x+1, y-1/2, -z+1/2$; (ii) $-x+1, y+1/2, -z+1/2$; (iii) $x, -y+3/2, z-1/2$.

Fig. 1

