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Methyl (2*E*)-2-(4-chlorobenzylidene)-hydrazinecarboxylate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.126
Data-to-parameter ratio = 16.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_9H_9ClN_2O_2$, crystallizes with two independent molecules in the asymmetric unit, the conformations of which are slightly different. Intermolecular N—H···O hydrogen bonds link the molecules into ladder-type ribbons extending along the *c* axis.

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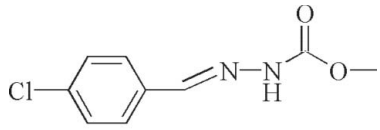
Comment

Chlorobenzaldehydehydrazone derivatives are important intermediates in the synthesis of 1,4-dihydrotetrazines, which demonstrate antitumour activity (Hu *et al.*, 2005). In our work on the preparation of chlorobenzaldehydehydrazone derivatives, the title compound, (I), has been obtained and we present its crystal structure here.



(I)

Compound (I) crystallizes with two independent molecules in the asymmetric unit (Fig. 1). In the molecules, the benzene rings C1–C6 and C11–C16 are twisted out of the central C7═N1—N2 (*A*) and C17═N3—N4 (*B*) planes by 13.8 (2) and 8.4 (2)°, respectively. The planes C8/O1/O2 and C18/O3/O4 are twisted out of the planes *A* and *B* by 12.4 (3) and 5.3 (2)°, respectively.

In the crystal structure of (I), intermolecular N—H···O hydrogen bonds (Table 1, Fig. 1) link the molecules into ladder-type ribbons extending along the *c* axis.

Experimental

4-Chlorobenzaldehyde (14.0 g, 0.1 mol) and methylhydrazinocarboxylate (9.0 g, 0.1 mol) were dissolved in stirred ethanol (100 ml) and left for 3 h at room temperature. The resulting solid was filtered off and recrystallized from ethyl acetate to give the title compound in 86% yield. The solid product was dissolved in acetone and then evaporated gradually at room temperature to afford single crystals of (I) (m.p. 437–439 K).

Crystal data

$C_9H_9ClN_2O_2$
M_r = 212.63
Monoclinic, $P2_1/c$
a = 13.492 (2) Å
b = 9.5342 (14) Å
c = 15.527 (2) Å
β = 94.800 (2)°
V = 1990.4 (5) Å^3
Z = 8
Mo *Kα* radiation
μ = 0.36 mm^{-1}
T = 296 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.900, T_{max} = 0.955
12107 measured reflections
4322 independent reflections
3235 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.126
S = 1.09
4322 reflections
264 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2X···O4	0.862 (14)	1.999 (15)	2.8434 (19)	166.4 (18)
N4—H4X···O2^{i}	0.843 (14)	2.067 (15)	2.9016 (18)	170.5 (18)

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

Atoms H2X and H4X were located in a difference map and refined isotropically with an N—H bond restraint of 0.85 (2) Å. Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, and torsion angles were refined to fit the electron density; U_{iso}(H) = 1.5U_{eq}(C). The remaining H atoms were placed in calculated positions, with C—H = 0.93, and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2005); software used to prepare material for publication: *SHELXTL*.

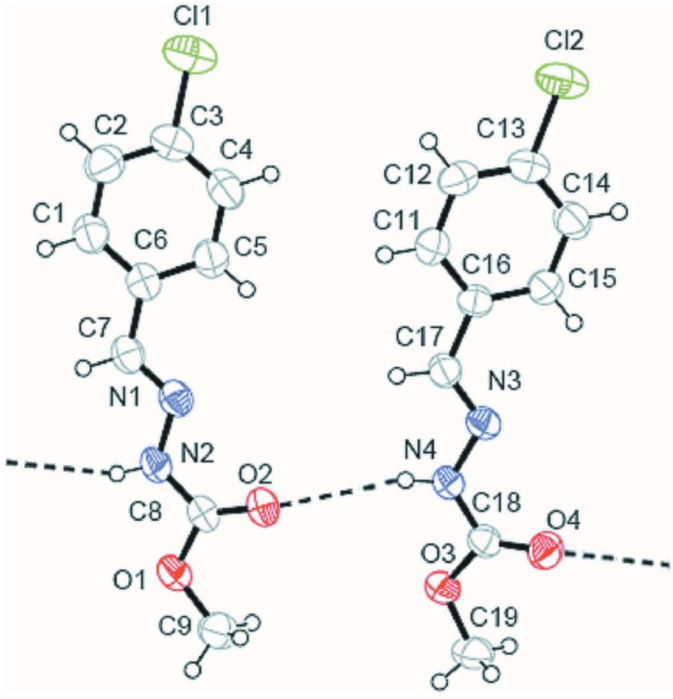


Figure 1
The asymmetric unit of (I), showing the atomic labelling and 40% probability displacement ellipsoids. Dashed lines denote hydrogen bonds.

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