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2′-Chloroacetophenone azine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.050
wR factor = 0.171
Data-to-parameter ratio = 18.3

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

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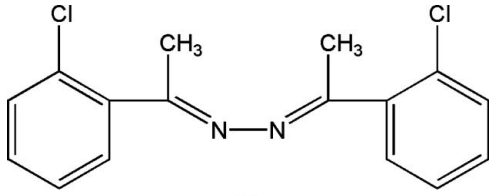
The title compound, $C_{16}H_{14}Cl_2N_2$, was prepared by the reaction of hydrazine with 2-chloroacetophenone at room temperature. The molecule has C_2 symmetry with the mid-point of the N—N bond lying on the twofold axis.

Comment

Schiff bases have received considerable attention because of their possible applications in catalysis (Cimerman *et al.*, 2000) and pharmacology (Parashar *et al.*, 1988). As part of our ongoing investigation of Schiff bases and their metal complexes, we report here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The molecule has C_2 symmetry, the twofold axis passing through the mid-point of the N1—N1A bond [symmetry code: (A) $1-x, y, -z+\frac{1}{2}$]. The N1—N1A bond distance is comparable with that reported by Lewis *et al.* (1998). The C7═N1 bond distance (Table 1) shows double-bond character. The dihedral angle between the N1/C7/C8 plane and benzene ring is 67.30 (2)°.

A weak intermolecular C—H$\cdots\pi$ interaction is observed in the crystal structure; C3—H3A = 0.93 Å, H3$A\cdots Cg^{ii}$ = 2.97 Å, C3$\cdots Cg^{ii}$ = 3.854 (3) Å and C3—H3$A\cdots Cg^{ii}$ = 160° [Cg is the centroid of the benzene ring; symmetry code: (ii) $\frac{3}{2}-x, y, \frac{1}{2}+z$].

Experimental

An aqueous solution (40 ml) of hydrazine (0.02 mol) and 2-chloroacetophenone (0.04 mol) was stirred for 5 h at room temperature. The solution was then filtered and concentrated to afford crystals of (I). Recrystallization from an ethanol solution gave single crystals of (I).

Crystal data

$C_{16}H_{14}Cl_2N_2$
M_r = 305.21
Orthorhombic, *Pcca*
a = 17.149 (3) Å
b = 11.636 (2) Å
c = 8.0120 (16) Å
V = 1598.8 (5) Å^3
Z = 4
D_x = 1.270 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 293 (2) K
Block, colourless
0.25 × 0.20 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3325 measured reflections
1706 independent reflections
991 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 27.0^\circ$
3 standard reflections
every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.171$
$S = 0.93$
1706 reflections
93 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0968P)^2 + 0.4695P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.057 (6)

Table 1
Selected geometric parameters (Å, °).

Cl1—C5	1.748 (3)	C6—C7	1.491 (4)
N1—C7	1.273 (3)	C7—C8	1.497 (4)
N1—N1^{i}	1.399 (4)		
C7—N1—N1^{i}	117.6 (2)	N1—C7—C8	125.0 (2)

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

Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$. Aromatic H atoms were placed in calculation positions, with C—H = 0.93 Å, and refined in riding mode, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*-PC (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

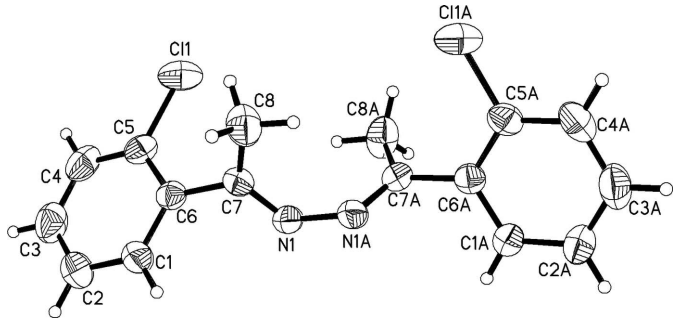


Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms) [symmetry code: (A) $1-x, y, -z+\frac{1}{2}$].

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