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Ethyl 3-(2,4-dichlorobenzylidene)-carbazate

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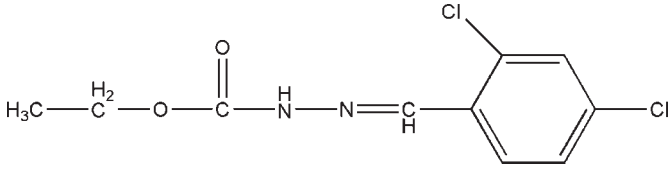
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.121; data-to-parameter ratio = 19.1.

The title compound, $C_{10}H_{10}Cl_2N_2O_2$, was prepared by the reaction of ethyl carbazate and 2,4-dichlorobenzaldehyde. In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds, forming extended chains along [001].

Related literature

For applications of Schiff base compounds, see: Cimerman *et al.* (1997). For the C=N double-bond length in a related structure, see: Girgis (2006).



Experimental

Crystal data

$C_{10}H_{10}Cl_2N_2O_2$ M_r = 261.10
Tetragonal, $I4_1/a$
a = 18.021 (3) Å
c = 14.983 (3) Å
V = 4865.8 (14) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.52 mm^{-1}
T = 293 K
0.25 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.492, T_{max} = 0.729
21376 measured reflections
2789 independent reflections
2409 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.121
S = 1.09
2789 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	2.12	2.927 (2)	156

Symmetry code: (i) $-y+\frac{3}{4}, x+\frac{1}{4}, z+\frac{1}{4}$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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: LH2929).

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supporting information

Acta Cryst. (2009). E**65**, o2919 [https://doi.org/10.1107/S1600536809044420]

Ethyl 3-(2,4-dichlorobenzylidene)carbazate

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S1. Comment

Schiff bases have received considerable attention in the literature and have potential analytical applications (Cimerman *et al.*, 1997). As part of our search for new schiff base compounds we synthesized the title compound (I), and its crystal structure is determined herein.

The molcular structure of (I) is shown in Fig. 1. The C8—N1 bond length of 1.271 (2)Å is comparable with C—N double bond [1.281 (2) Å] reported in a related structure (Girgis, 2006). In the crystal structure, molecules are linked by intermolecular N-H···O hydrogen bonds to form extended chains along [001].

S2. Experimental

A mixture of the 2,4-dichlorobenzaldehyde (0.1 mol), and Ethyl carbazate (0.1 mol) was stirred in refluxing ethanol (20 mL) for 4 h to afford the title compound (0.080 mol, yield 80%). Single crystals suitable for X-ray measurements were obtained by recrystallization of a solution of (I) in ethanol at room temperature.

S3. Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances = 0.93-0.97 Å; N-H = 0.86Å and with U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(C_{methyl}).

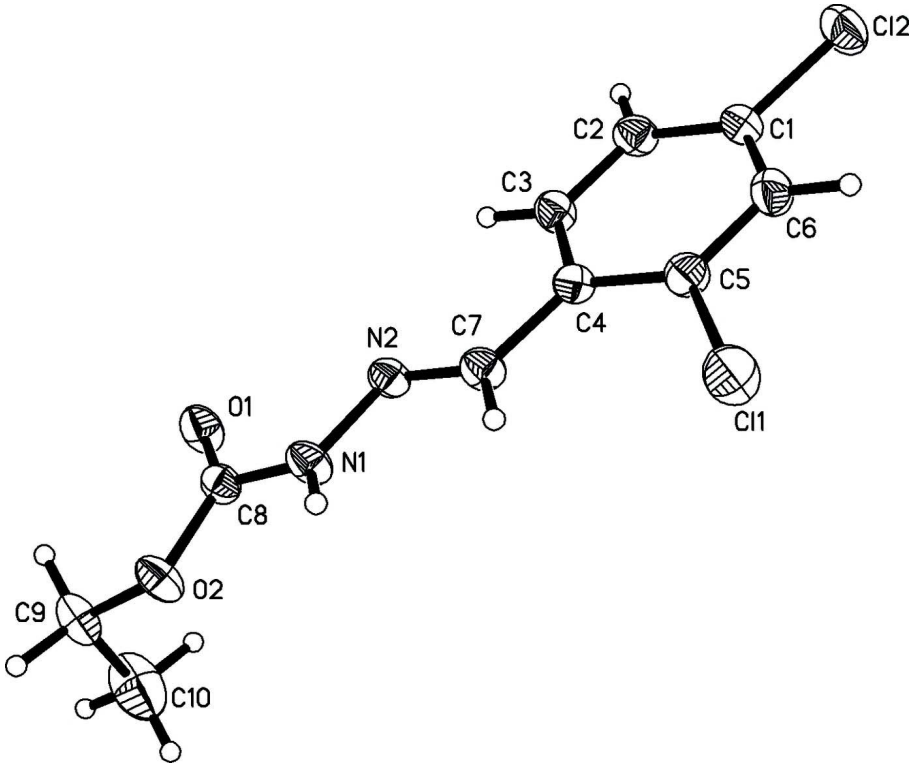

Figure 1

The molecular structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Ethyl 3-(2,4-dichlorobenzylidene)carbazate

Crystal data

$C_{10}H_{10}Cl_2N_2O_2$
$M_r = 261.10$
Tetragonal, $I4_1/a$
Hall symbol: -I 4ad
$a = 18.021$ (3) Å
$c = 14.983$ (3) Å
$V = 4865.8$ (14) Å^3
$Z = 16$
$F(000) = 2144$
$D_x = 1.426$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1977 reflections
$\theta = 3.5$–27.4°
$\mu = 0.52$ mm^{-1}
$T = 293$ K
Block, colorless
0.25 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.492$, $T_{max} = 0.729$
21376 measured reflections
2789 independent reflections
2409 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
$h = -23\rightarrow23$
$k = -23\rightarrow23$
$l = -19\rightarrow18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.121$
$S = 1.09$
2789 reflections
146 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 + 2.5907P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0030 (4)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.22311 (4)	0.76326 (3)	0.01502 (4)	0.0730 (2)
Cl2	0.09897 (3)	0.79872 (3)	−0.30581 (4)	0.0661 (2)
N2	0.28329 (7)	0.54137 (8)	−0.06890 (9)	0.0376 (3)
O2	0.39424 (7)	0.41332 (7)	0.04214 (8)	0.0474 (3)
N1	0.32058 (8)	0.50259 (8)	−0.00379 (9)	0.0416 (3)
H1A	0.3228	0.5189	0.0501	0.050*
O1	0.34815 (8)	0.40718 (7)	−0.09813 (8)	0.0500 (3)
C8	0.35371 (9)	0.43809 (9)	−0.02677 (10)	0.0362 (3)
C7	0.26094 (9)	0.60548 (10)	−0.04588 (11)	0.0411 (4)
H7A	0.2695	0.6224	0.0119	0.049*
C4	0.22188 (8)	0.65273 (9)	−0.10968 (10)	0.0371 (3)
C2	0.16741 (10)	0.67119 (10)	−0.25620 (11)	0.0440 (4)
H2B	0.1568	0.6534	−0.3130	0.053*
C5	0.20078 (10)	0.72492 (10)	−0.08829 (11)	0.0441 (4)
C3	0.20456 (10)	0.62758 (9)	−0.19537 (11)	0.0414 (4)
H3A	0.2186	0.5798	−0.2119	0.050*
C1	0.14623 (10)	0.74200 (10)	−0.23128 (12)	0.0445 (4)
C6	0.16227 (11)	0.76940 (10)	−0.14764 (13)	0.0498 (4)
H6A	0.1475	0.8170	−0.1314	0.060*
C9	0.44238 (11)	0.35079 (11)	0.02348 (14)	0.0538 (5)
H9A	0.4173	0.3167	−0.0166	0.065*
H9B	0.4529	0.3246	0.0786	0.065*
C10	0.51290 (15)	0.37543 (18)	−0.0177 (2)	0.0908 (9)

H10A	0.5436	0.3330	−0.0297	0.136*
H10B	0.5383	0.4083	0.0225	0.136*
H10C	0.5025	0.4010	−0.0725	0.136*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1010 (5)	0.0595 (3)	0.0586 (3)	0.0176 (3)	−0.0303 (3)	−0.0252 (2)
Cl2	0.0726 (4)	0.0565 (3)	0.0693 (4)	0.0135 (2)	−0.0261 (3)	0.0084 (2)
N2	0.0396 (7)	0.0420 (7)	0.0312 (6)	0.0035 (6)	−0.0042 (5)	0.0013 (5)
O2	0.0540 (7)	0.0566 (7)	0.0316 (6)	0.0170 (6)	−0.0047 (5)	0.0045 (5)
N1	0.0505 (8)	0.0476 (8)	0.0266 (6)	0.0112 (6)	−0.0060 (5)	−0.0019 (5)
O1	0.0713 (8)	0.0455 (6)	0.0332 (6)	0.0116 (6)	−0.0084 (6)	−0.0044 (5)
C8	0.0401 (8)	0.0411 (8)	0.0275 (7)	0.0010 (6)	−0.0002 (6)	0.0048 (6)
C7	0.0445 (8)	0.0457 (9)	0.0332 (7)	0.0056 (7)	−0.0035 (6)	−0.0033 (7)
C4	0.0351 (7)	0.0399 (8)	0.0361 (8)	0.0016 (6)	−0.0014 (6)	−0.0019 (6)
C2	0.0458 (9)	0.0494 (9)	0.0367 (8)	0.0031 (7)	−0.0066 (7)	−0.0039 (7)
C5	0.0477 (9)	0.0423 (8)	0.0423 (9)	0.0029 (7)	−0.0076 (7)	−0.0086 (7)
C3	0.0451 (9)	0.0403 (8)	0.0388 (8)	0.0063 (7)	−0.0026 (7)	−0.0048 (7)
C1	0.0418 (9)	0.0437 (9)	0.0481 (9)	0.0038 (7)	−0.0090 (7)	0.0051 (7)
C6	0.0548 (10)	0.0383 (8)	0.0562 (11)	0.0077 (7)	−0.0096 (9)	−0.0067 (8)
C9	0.0595 (11)	0.0533 (10)	0.0484 (10)	0.0185 (9)	0.0017 (9)	0.0126 (8)
C10	0.0688 (15)	0.106 (2)	0.098 (2)	0.0277 (14)	0.0305 (14)	0.0353 (17)

Geometric parameters (Å, º)

Cl1—C5	1.7421 (17)	C2—C3	1.377 (2)
Cl2—C1	1.7370 (17)	C2—C1	1.383 (2)
N2—C7	1.271 (2)	C2—H2B	0.9300
N2—N1	1.3755 (18)	C5—C6	1.384 (2)
O2—C8	1.3412 (19)	C3—H3A	0.9300
O2—C9	1.449 (2)	C1—C6	1.378 (3)
N1—C8	1.351 (2)	C6—H6A	0.9300
N1—H1A	0.8600	C9—C10	1.481 (3)
O1—C8	1.2097 (19)	C9—H9A	0.9700
C7—C4	1.461 (2)	C9—H9B	0.9700
C7—H7A	0.9300	C10—H10A	0.9600
C4—C5	1.393 (2)	C10—H10B	0.9600
C4—C3	1.397 (2)	C10—H10C	0.9600
C7—N2—N1	115.10 (13)	C2—C3—H3A	118.9
C8—O2—C9	115.87 (14)	C4—C3—H3A	118.9
C8—N1—N2	118.18 (13)	C6—C1—C2	121.22 (16)
C8—N1—H1A	120.9	C6—C1—Cl2	118.48 (13)
N2—N1—H1A	120.9	C2—C1—Cl2	120.30 (14)
O1—C8—O2	124.90 (15)	C1—C6—C5	118.83 (16)
O1—C8—N1	125.78 (15)	C1—C6—H6A	120.6
O2—C8—N1	109.31 (13)	C5—C6—H6A	120.6

N2—C7—C4	120.32 (14)	O2—C9—C10	111.15 (19)
N2—C7—H7A	119.8	O2—C9—H9A	109.4
C4—C7—H7A	119.8	C10—C9—H9A	109.4
C5—C4—C3	116.98 (15)	O2—C9—H9B	109.4
C5—C4—C7	121.69 (14)	C10—C9—H9B	109.4
C3—C4—C7	121.33 (14)	H9A—C9—H9B	108.0
C3—C2—C1	118.81 (16)	C9—C10—H10A	109.5
C3—C2—H2B	120.6	C9—C10—H10B	109.5
C1—C2—H2B	120.6	H10A—C10—H10B	109.5
C6—C5—C4	122.01 (15)	C9—C10—H10C	109.5
C6—C5—Cl1	117.20 (13)	H10A—C10—H10C	109.5
C4—C5—Cl1	120.79 (13)	H10B—C10—H10C	109.5
C2—C3—C4	122.12 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.86	2.12	2.927 (2)	156

Symmetry code: (i) −*y*+3/4, *x*+1/4, *z*+1/4.