Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,2-Bis(2-chlorobenzylidene)hydrazine

Chun-Niu Zhang and Yun-Fa Zheng*

Department of Chemistry, Lishui College, 323000 Lishui, ZheJiang, People's Republic of China
Correspondence e-mail: zjlsxyhx@126.com

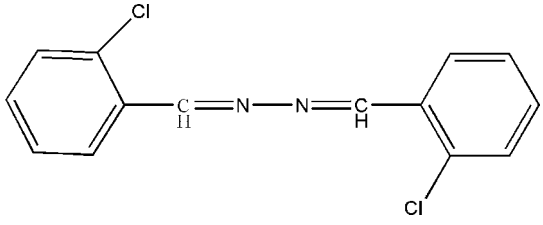
Received 13 November 2007; accepted 22 November 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.066; data-to-parameter ratio = 13.6.

The title Schiff base compound, $C_{14}H_{10}Cl_2N_2$, crystallizes with one half-molecule in the asymmetric unit. The mid-point of the N—N bond [1.418 (3) Å] lies on an inversion centre. The molecular skeleton is approximately planar, the largest deviation from the mean plane being 0.143 (4) Å for the N-bonded C atom. The crystal packing exhibits no classical intermolecular hydrogen bonds.

Related literature

For related literature, see: Alemi & Shaabani (2000); Alizadeh *et al.* (1999); Allen (2002).



Experimental

Crystal data

$C_{14}H_{10}Cl_2N_2$
M_r = 277.14
Monoclinic, $P2_1/c$
a = 3.9449 (17) Å
b = 13.548 (6) Å
c = 11.993 (5) Å
β = 93.931 (6)°
V = 639.5 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 298 (2) K
0.29 × 0.25 × 0.17 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.871, T_{max} = 0.922
3767 measured reflections
1119 independent reflections
738 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.066
S = 0.97
1119 reflections
82 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to the Natural Science Foundation of Zhejiang Province for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2367).

References

Alemi, A. A. & Shaabani, B. (2000). *Acta Chim. Slov.* **47**, 363–369.
Alizadeh, N., Ershad, S., Naeimi, H., Sharghi, H. & Shamsipur, M. (1999). *Pol. J. Chem.* **73**, 915–925.
Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Winconsin, USA.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.

  doi:10.1107/S1600536807062150

supporting information

Acta Cryst. (2008). E**64**, o36 [https://doi.org/10.1107/S1600536807062150]

1,2-Bis(2-chlorobenzylidene)hydrazine

Chun-Niu Zhang and Yun-Fa Zheng

S1. Comment

Schiff base ligands have significant importance in chemistry, especially in the development of Schiff base complexes, (Alizadeh *et al.*, 1999). Schiff bases exhibiting olvent-dependent UV/vis spectra (solvatochromicity) can be suitable NLO (nonlinear optically active) materials (Alemi & Shaabani, 2000). In this paper, we report the synthesis and crystal structure of the title compound, (I).

The molecular structure of the title compound has crystallographically imposed inversion symmetry located in the middle of the N—N bond (Fig. 1). The molecule is approximately planar with the largest deviation from the plane being 0.143 (4) for C7. The C7—N1 of 1.272 (2)Å is indicative of a C?N double bond. The other C—N, C—Cl, and C—C distances show no remarkable features (Allen, 2002).

S2. Experimental

Under nitrogen, a mixture of 2-chlorobenzaldehyde (2.8 g, 20 mmol), Na_2SO_4 (3.0 g) and hydrazine (30% in water, 10 mmol) in absolute ethanol (70 ml) was refluxed for about 3 h to yield a yellow precipitate. The product was collected by vacuum filtration and washed with ethanol. The crude solid was redissolved in CH_2Cl_2 (100 ml) and washed with water (2*10 ml)and brine(10 ml). After dried over Na_2SO_4, the solvent was removed under vacuum, and yellow solid was isolated in yield 90% (2.5 g). Colourless single crystals of the compound suitable for X-ray analysis were grown from CH_2Cl_2 and absolute ethanol(3:1) by slow evaporation of the solvent at room temperature over a period of about two weeks.

S3. Refinement

All H atoms were placed in calculated positions (C—H = 0.93 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

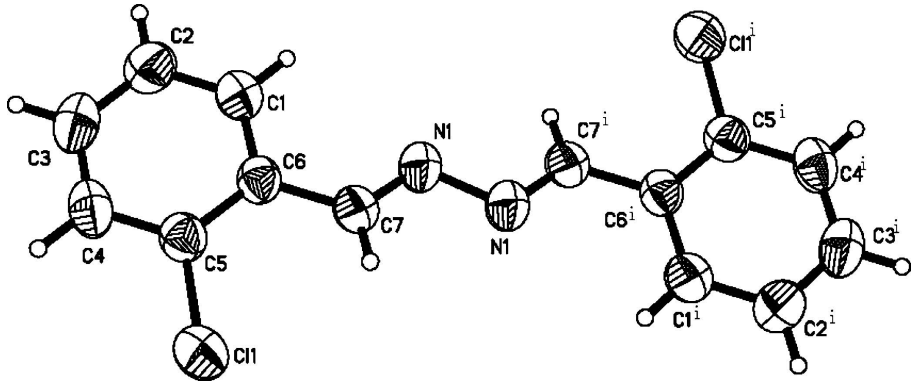


Figure 1

The molecular structure of (I) showing the atomic numbering scheme and 30% probability displacement ellipsoids [symmetry code (i):-*x*, 1 - *y*, 1 - *z*].

1,2-Bis(2-chlorobenzylidene)hydrazine

Crystal data

$C_{14}H_{10}Cl_2N_2$
$M_r = 277.14$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 3.9449\ (17)$ Å
$b = 13.548\ (6)$ Å
$c = 11.993\ (5)$ Å
$\beta = 93.931\ (6)°$
$V = 639.5\ (5)$ Å^3
$Z = 2$
$F(000) = 284$
$D_x = 1.439$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1119 reflections
$\theta = 2.3–25.2°$
$\mu = 0.49$ mm^{-1}
$T = 298$ K
Block, colourless
0.29 × 0.25 × 0.17 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scan
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.871$, $T_{max} = 0.922$
3767 measured reflections
1119 independent reflections
738 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.2°$, $\theta_{min} = 2.3°$
$h = -4\rightarrow4$
$k = -16\rightarrow16$
$l = -13\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.066$
$S = 0.97$
1119 reflections
82 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
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.47589 (13)	0.61367 (3)	0.83542 (4)	0.0708 (2)
C1	0.0082 (4)	0.75417 (13)	0.57454 (14)	0.0566 (5)
H1	−0.0887	0.7395	0.5036	0.068*
C7	0.1598 (4)	0.57711 (12)	0.59883 (13)	0.0500 (4)
H7	0.3006	0.5310	0.6361	0.060*
C6	0.1555 (4)	0.67868 (12)	0.64024 (13)	0.0458 (4)
C4	0.2921 (4)	0.80006 (13)	0.78504 (14)	0.0595 (5)
H4	0.3884	0.8156	0.8558	0.071*
C5	0.2958 (4)	0.70373 (12)	0.74636 (13)	0.0499 (4)
C3	0.1449 (5)	0.87235 (13)	0.71780 (17)	0.0653 (5)
H3	0.1408	0.9370	0.7436	0.078*
C2	0.0033 (5)	0.85042 (13)	0.61264 (16)	0.0644 (5)
H2	−0.0950	0.9000	0.5676	0.077*
N1	−0.0248 (4)	0.55045 (9)	0.51295 (11)	0.0567 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0842 (4)	0.0692 (3)	0.0563 (3)	0.0015 (3)	−0.0145 (2)	−0.0021 (2)
C1	0.0631 (12)	0.0559 (11)	0.0499 (11)	−0.0027 (9)	−0.0024 (9)	−0.0033 (8)
C7	0.0549 (12)	0.0494 (10)	0.0449 (10)	−0.0040 (8)	−0.0021 (8)	−0.0021 (8)
C6	0.0446 (11)	0.0467 (10)	0.0464 (9)	−0.0065 (8)	0.0050 (8)	−0.0043 (8)
C4	0.0617 (13)	0.0614 (12)	0.0552 (11)	−0.0110 (10)	0.0037 (9)	−0.0167 (9)
C5	0.0483 (11)	0.0529 (10)	0.0482 (10)	−0.0053 (8)	0.0015 (8)	−0.0020 (8)
C3	0.0707 (14)	0.0491 (11)	0.0767 (13)	−0.0049 (10)	0.0103 (11)	−0.0142 (10)
C2	0.0714 (15)	0.0538 (12)	0.0681 (12)	0.0018 (9)	0.0058 (10)	0.0023 (10)
N1	0.0687 (10)	0.0462 (8)	0.0539 (9)	−0.0061 (8)	−0.0050 (8)	−0.0069 (7)

Geometric parameters (Å, º)

Cl1—C5	1.7406 (16)	C4—C3	1.372 (2)
C1—C2	1.382 (2)	C4—C5	1.385 (2)
C1—C6	1.393 (2)	C4—H4	0.9300
C1—H1	0.9300	C3—C2	1.376 (3)
C7—N1	1.272 (2)	C3—H3	0.9300
C7—C6	1.463 (2)	C2—H2	0.9300

C7—H7	0.9300	N1—N1^{i}	1.418 (2)
C6—C5	1.394 (2)		
C2—C1—C6	121.35 (16)	C5—C4—H4	120.4
C2—C1—H1	119.3	C4—C5—C6	121.48 (15)
C6—C1—H1	119.3	C4—C5—Cl1	117.90 (13)
N1—C7—C6	121.49 (15)	C6—C5—Cl1	120.60 (13)
N1—C7—H7	119.3	C4—C3—C2	120.89 (17)
C6—C7—H7	119.3	C4—C3—H3	119.6
C1—C6—C5	117.48 (15)	C2—C3—H3	119.6
C1—C6—C7	120.82 (14)	C3—C2—C1	119.50 (17)
C5—C6—C7	121.70 (15)	C3—C2—H2	120.3
C3—C4—C5	119.29 (16)	C1—C2—H2	120.3
C3—C4—H4	120.4	C7—N1—N1^{i}	111.78 (16)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···Cl1	0.93	2.69	3.060 (2)	105