Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Bis[3-(chloroacetyl)phenyl]diazene

De-Mei Tian,[a,b] Yong-Sheng Niu,[c] Chuan-Xi Xiong[a]* and Bo Hu[b]

[a]School of Materials and Technology, Wuhan University of Technology, Wuhan Hubei 430070, People's Republic of China, [b]College of Chemistry, Central China Normal University, Wuhan Hubei 430079, People's Republic of China, and [c] Department of Chemical Engineering, Anyang Institue of Technology, Anyang Henan 455000, People's Republic of China
Correspondence e-mail: tiandm@mail.ccnu.edu.cn

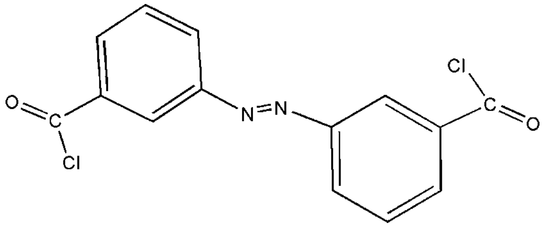
Received 8 June 2007; accepted 22 June 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.050; wR factor = 0.137; data-to-parameter ratio = 16.2.

The title molecule, $C_{14}H_8Cl_2N_2O_2$, is a *trans*-azobenzene which has a crystallographic inversion center at the mid-point of the N═N bond [N═N = 1.228 (4) Å]. There are neither hydrogen bonds nor π–π interactions between the aromatic rings, and the crystal structure is stabilized by van der Waals interactions.

Related literature

For background information, see: Dürr & Bouas-Laurent (1990); Martin *et al.* (1995); Zhao *et al.* (1999). For the synthetic procedure, see: Xiong *et al.* (2006). For bond length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{14}H_8Cl_2N_2O_2$
M_r = 307.12
Monoclinic, $P2_1/c$
a = 12.7512 (15) Å
b = 3.9116 (4) Å
c = 16.6550 (14) Å
β = 126.189 (6)°
V = 670.44 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 294 (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.909, T_{max} = 0.953
3784 measured reflections
1475 independent reflections
1109 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.137
S = 1.06
1475 reflections
91 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 2001).

The authors acknowledge financial support from the State '863' Science Foundation (2002AA333110).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2430).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2000). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dürr, H. & Bouas-Laurent, H. (1990). *Photochromism: Molecules and Systems*. Amsterdam: Elsevier.
Martin, P. J., Petty, M. C., Bryce, M. R. & Bloor, D. (1995). *An Introduction to Molecular Electronics*, ch. 6. New York: Oxford University Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Xiong, C. X., Niu, Y. S., Zhou, W. & Dong, L. J. (2006). *J. Chem. Res.* pp. 139–140.
Zhao, W., Wu, C. X. & Iwamoto, M. (1999). *Chem. Phys. Lett.* **312**, 572–577.

doi:10.1107/S1600536807030577

supplementary materials

Acta Cryst. (2007). E**63**, o3373 [**doi:10.1107/S1600536807030577**]

Bis[3-(chloroacetyl)phenyl]diazene

D.-M. Tian, Y.-S. Niu, C.-X. Xiong and **B. Hu**

Comment

In azobenzene compounds, a conversion from the *trans* to *cis* form can lead to photochromism. Photochromic compounds are of great interest for the control and measurement of radiation intensity, optical computers and display systems (Dürr & Bouas-Laurent, 1990) and for potential applications in molecular electronic devices (Martin *et al.*, 1995). In addition, azo polymers have interest in various fields (Zhao *et al.*, 1999). As a part of our investigation of preparing azo polymers, we report the crystal structure of the title compound.

There is an inversion center at the mid-point of the N═N bond. The central N═N bond length of 1.228 (4) Å is slightly shorter than the average value of 1.257 Å for azobenzene N═N bonds (Allen *et al.*, 1987). But the C1—N1 bond length of 1.437 (3) Å is almost the same as the average value (1.43 Å). The molecular structure is shown in Fig. 1.

Experimental

The title compound was prepared by literature method (Xiong *et al.*, 2006). Single crystals were obtained by slow evaporation of a heptane solution.

Refinement

All H-atoms were discernible in a difference Fourier map. H atoms bound to C atoms were included in calculated positions and allowed to ride during refinement, with C—H = 0.93 Å, and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

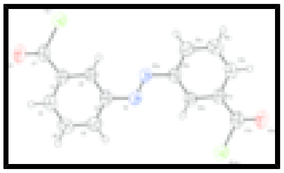


Fig. 1. The molecular structure with displacement ellipsoids drawn at the 50% probability level [symmetry code: (*a*) $2 - x, 1 - y, 1 - z$]

Bis[3-(chloroacetyl)phenyl]diazene

Crystal data

$C_{14}H_8Cl_2N_2O_2$	$F_{000} = 312$
$M_r = 307.12$	$D_x = 1.521$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 374 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å

a = 12.7512 (15) Å	Cell parameters from 1015 reflections
b = 3.9116 (4) Å	θ = 2.5–25.8°
c = 16.6550 (14) Å	μ = 0.49 mm^{-1}
β = 126.189 (6)°	T = 294 (2) K
V = 670.44 (12) Å^3	Block, red
Z = 2	0.20 × 0.20 × 0.10 mm

Data collection

Bruker APEX CCD area-detector diffractometer	1475 independent reflections
Radiation source: fine-focus sealed tube	1109 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.030
T = 294(2) K	θ_{max} = 27.2°
φ and ω scans	θ_{min} = 2.0°
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	$h = -15 \rightarrow 16$
T_{min} = 0.909, T_{max} = 0.953	$k = -4 \rightarrow 5$
3784 measured reflections	$l = -20 \rightarrow 21$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.050	H-atom parameters constrained
$wR(F^2)$ = 0.137	$w = 1/[\sigma^2(F_o^2) + (0.0695P)^2 + 0.1223P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{\text{max}} < 0.001$
1475 reflections	$\Delta\rho_{\text{max}}$ = 0.34 e Å^{-3}
91 parameters	$\Delta\rho_{\text{min}}$ = −0.21 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 > 2sigma(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	$U_{\rm iso}$*/$U_{\rm eq}$
Cl1	0.85788 (6)	0.37064 (18)	0.72874 (5)	0.0627 (3)
O1	0.63441 (18)	0.6568 (6)	0.62472 (14)	0.0739 (6)
N1	0.95418 (18)	0.5708 (5)	0.46251 (13)	0.0484 (5)
C1	0.7350 (2)	0.6528 (5)	0.54165 (16)	0.0405 (5)
C2	0.84462 (19)	0.5682 (6)	0.54617 (15)	0.0405 (5)
H2	0.9155	0.4622	0.6023	0.049*
C3	0.8461 (2)	0.6448 (5)	0.46547 (16)	0.0427 (5)
C4	0.6292 (2)	0.8121 (6)	0.45726 (18)	0.0494 (6)
H4	0.5566	0.8705	0.4547	0.059*
C5	0.7410 (2)	0.7994 (6)	0.38163 (18)	0.0530 (6)
H5	0.7428	0.8478	0.3278	0.064*
C6	0.6323 (2)	0.8824 (6)	0.37792 (18)	0.0551 (6)
H6	0.5612	0.9863	0.3214	0.066*
C7	0.7233 (2)	0.5837 (6)	0.62321 (17)	0.0488 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0672 (5)	0.0749 (5)	0.0573 (4)	0.0146 (3)	0.0431 (4)	0.0124 (3)
O1	0.0552 (11)	0.1128 (18)	0.0736 (13)	0.0100 (11)	0.0490 (11)	0.0056 (11)
N1	0.0470 (11)	0.0591 (12)	0.0472 (10)	0.0016 (9)	0.0324 (9)	0.0006 (9)
C1	0.0392 (11)	0.0402 (12)	0.0454 (11)	−0.0048 (9)	0.0268 (10)	−0.0066 (9)
C2	0.0361 (11)	0.0427 (12)	0.0404 (11)	−0.0020 (9)	0.0214 (9)	−0.0034 (9)
C3	0.0417 (12)	0.0445 (12)	0.0475 (12)	−0.0053 (9)	0.0294 (10)	−0.0065 (9)
C4	0.0395 (12)	0.0500 (14)	0.0567 (13)	0.0010 (10)	0.0274 (11)	−0.0061 (10)
C5	0.0551 (14)	0.0593 (16)	0.0464 (13)	−0.0021 (11)	0.0309 (12)	0.0019 (11)
C6	0.0459 (14)	0.0575 (15)	0.0497 (13)	0.0044 (11)	0.0215 (11)	0.0025 (11)
C7	0.0431 (13)	0.0547 (14)	0.0529 (13)	−0.0052 (10)	0.0306 (11)	−0.0076 (11)

Geometric parameters (Å, °)

Cl1—C7	1.780 (2)	C2—H2	0.9300
O1—C7	1.183 (3)	C3—C5	1.379 (3)
N1—N1^{i}	1.228 (4)	C4—C6	1.373 (4)
N1—C3	1.437 (3)	C4—H4	0.9300
C1—C2	1.395 (3)	C5—C6	1.389 (3)
C1—C4	1.395 (3)	C5—H5	0.9300
C1—C7	1.477 (3)	C6—H6	0.9300
C2—C3	1.388 (3)		

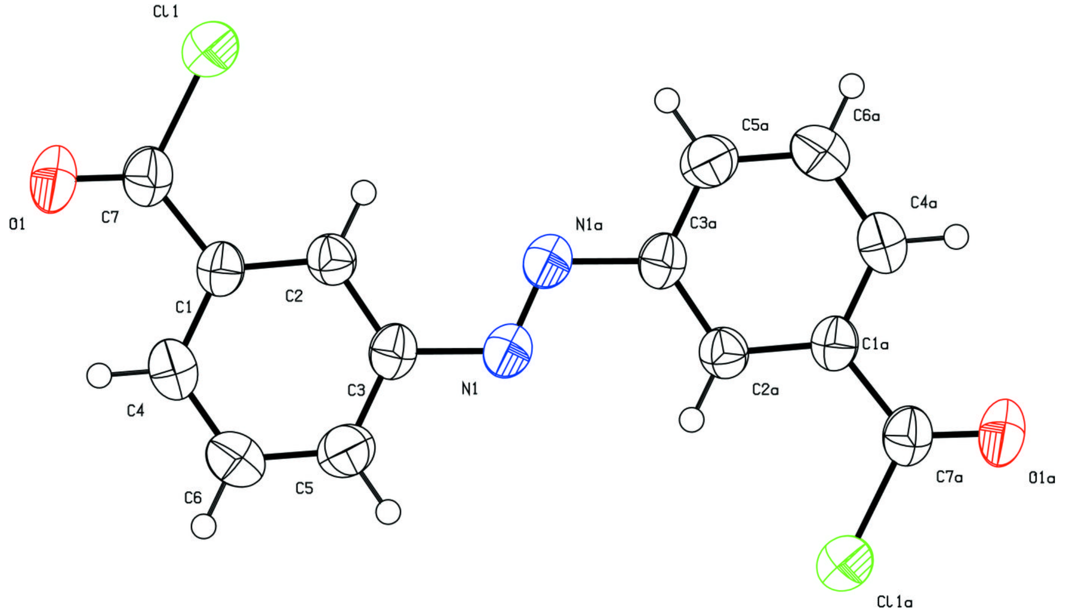
N1^{i}—N1—C3	114.3 (2)	C6—C4—H4	120.0
C2—C1—C4	120.2 (2)	C1—C4—H4	120.0
C2—C1—C7	123.3 (2)	C3—C5—C6	119.7 (2)
C4—C1—C7	116.5 (2)	C3—C5—H5	120.1
C3—C2—C1	118.9 (2)	C6—C5—H5	120.1

C3—C2—H2	120.6	C4—C6—C5	120.4 (2)
C1—C2—H2	120.6	C4—C6—H6	119.8
C5—C3—C2	120.9 (2)	C5—C6—H6	119.8
C5—C3—N1	115.61 (19)	O1—C7—C1	126.4 (2)
C2—C3—N1	123.46 (19)	O1—C7—Cl1	118.00 (19)
C6—C4—C1	119.9 (2)	C1—C7—Cl1	115.57 (16)

C4—C1—C2—C3	−0.1 (3)	C2—C3—C5—C6	0.5 (4)
C7—C1—C2—C3	−179.9 (2)	N1—C3—C5—C6	−179.6 (2)
C1—C2—C3—C5	−0.6 (3)	C1—C4—C6—C5	−0.7 (4)
C1—C2—C3—N1	179.61 (19)	C3—C5—C6—C4	0.1 (4)
N1^{i}—N1—C3—C5	−179.1 (3)	C2—C1—C7—O1	178.0 (2)
N1^{i}—N1—C3—C2	0.8 (4)	C4—C1—C7—O1	−1.8 (4)
C2—C1—C4—C6	0.7 (3)	C2—C1—C7—Cl1	−1.7 (3)
C7—C1—C4—C6	−179.5 (2)	C4—C1—C7—Cl1	178.46 (16)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1.

Fig. 1


Cl1
O1
C7
C1
C2
C4
C6
C5
C3
N1
N1a
C3a
C5a
C6a
C4a
C1a
C2a
C7a
O1a
Cl1a