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2,2′-Dichloro-1,1′-[(butane-1,4-diyldioxy)bis(nitrilomethylidyne)]dibenzene

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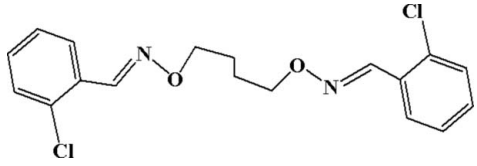
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.075; wR factor = 0.164; data-to-parameter ratio = 14.0.

The molecule of the title compound, $C_{18}H_{18}Cl_2N_2O_2$, lies across a crystallographic inversion centre and adopts an E configuration with respect to the azomethine C=N bond. The imino group is coplanar with the aromatic ring. Within the molecule, the planar units are parallel, but extend in opposite directions from the dimethylene bridge. In the crystal structure, the title compound exhibits a layer packing structure *via* weak π–π stacking interactions [intermolecular plane-to-plane distances between adjacent aromatic rings are 3.461 (3) Å]. Molecules in each layer are linked by intermolecular C—H···O hydrogen-bonding interactions.

Related literature

For related literature, see: Collison & Fenton (1996); Dong, He *et al.* (2007); Dong, Duan *et al.* (2007); Dong *et al.* (2008); Liu *et al.* (2008); Lu *et al.* (2006); Mandal *et al.* (1996); Shi *et al.* (2007); Yu *et al.* (2007, 2008).



Experimental

Crystal data

$C_{18}H_{18}Cl_2N_2O_2$
M_r = 365.24
Monoclinic, $P2_1/n$
a = 4.5296 (5) Å
b = 6.6231 (8) Å
c = 29.963 (2) Å
β = 92.526 (2)°
V = 898.02 (16) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 298 (2) K
0.48 × 0.28 × 0.13 mm

Data collection

Bruker SMART 1000 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.841, T_{max} = 0.953
4304 measured reflections
1531 independent reflections
1310 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.164
S = 1.10
1531 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O1^{i}	0.93	2.66	3.581 (5)	171

Symmetry code: (i) $x+1, y-1, z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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: OM2252).

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2,2′-Dichloro-1,1′-[(butane-1,4-diyldioxy)bis(nitrilomethylidyne)]dibenzene

Zong-Li Ren, Wen-Kui Dong, Wen-Juan Bai, Xue-Ni He and Li Wang

S1. Comment

Schiff bases are an important class of compounds which can be used in a variety of studies such as organic synthesis, catalyst, drug design, material science and life science and so on (Collison, *et al.*, 1996; Mandal, *et al.*, 1996). In the past decades, a continuing attention has been drawn to the Schiff bases derived from benzaldehyde or salicylaldehyde and their metal complexes for the investigation of luminescent properties which could be finely tuned by different substituent groups bonded to the phenolic ring (Lu *et al.*, 2006; Yu *et al.*, 2007; Yu *et al.*, 2008). Here, in continuation of our previous studies (Dong, Duan *et al.*, 2007; Shi, *et al.*, 2007), we report the synthesis and X-ray structure of a new Schiff base bis-oxime compound 2,2′-dichloro-1,1′-[butane-1,4-diyldioxybis(nitrilomethylidyne)]dibenzene.

The crystal structure of the title compound is built up by only the $C_{18}H_{18}Cl_2N_2O_2$ molecules, in which all bond lengths are in normal ranges. The molecule, as shown in Fig. 1, lies across a crystallographic inversion centre (symmetry code: -*x*, -*y*, -*z*) and adopts an E configuration with respect to the azomethine C=N bond. The imino group is coplanar with the aromatic ring. Within the molecule, the planar units are parallel, with the distance 1.480 (4) Å [intra-molecular plane-to-plane distance], but extend in opposite directions from the dimethylene bridge. In the crystal structure, (Fig. 2) the title compound exhibits a layer packing structure *via* weak *π*-*π* stacking interactions [inter-molecular plane-to-plane distances between adjacent aromatic rings is 3.461 (3) Å]. Molecules in each layer are linked by intermolecular C8—H8···O1 hydrogen bonding interactions [C8···O1, 3.581 (5) Å].

S2. Experimental

2,2′-Dichloro-1,1′-[butane-1,4-diyldioxybis(nitrilomethylidyne)]dibenzene was synthesized according to an analogous method reported earlier (Dong, He *et al.*, 2007; Dong, *et al.*, 2008; Liu, *et al.*, 2008). To an ethanol solution (3 ml) of 2-chloro-benzaldehyde (281.1 mg, 2.00 mmol) was added an ethanol solution (2 ml) of 1, 4-bis(aminooxy)butane (120.2 mg, 1.00 mmol). The mixture solution was stirred at 328 K for 4 h. When cooled to room temperature, the precipitate was filtered, and washed successively with ethanol and hexane, respectively. The product was dried under vacuum and purified with recrystallization from ethanol to yield 219.8 mg of the title compound. Yield, 60.1%. mp. 334–335 K. Anal. Calc. for $C_{18}H_{18}Cl_2N_2O_2$: C, 59.19; H, 4.97; N, 7.67. Found: C, 59.22; H, 5.03; N, 7.58.

Colorless needle-shaped single crystals suitable for X-ray diffraction studies were obtained after several weeks by slow evaporation from an ethyl-acetate/acetone mixed solution of the title compound.

S3. Refinement

Non-H atoms were refined anisotropically. H atoms were treated as riding atoms with distances C—H = 0.97 (CH_2), 0.93 Å (CH), and U_{iso}(H) = 1.2 U_{eq}(C) and 1.5 U_{eq}(O).

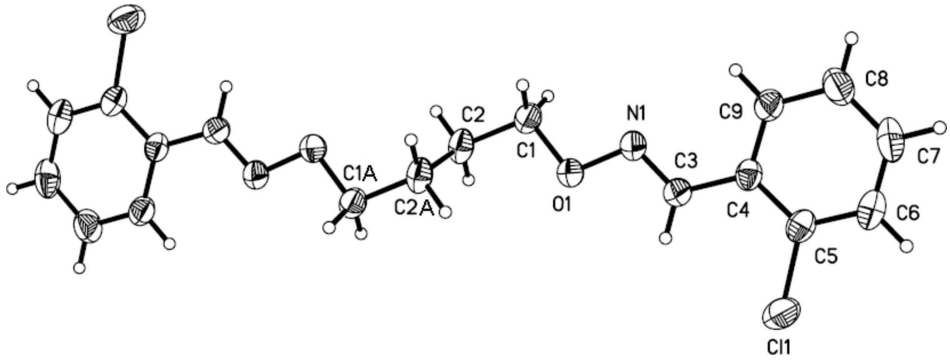

Figure 1

The molecular structure of the title compound with atom numbering scheme [Symmetry codes: -*x*, -*y* + 2, -*z* + 1]. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30% probability level.

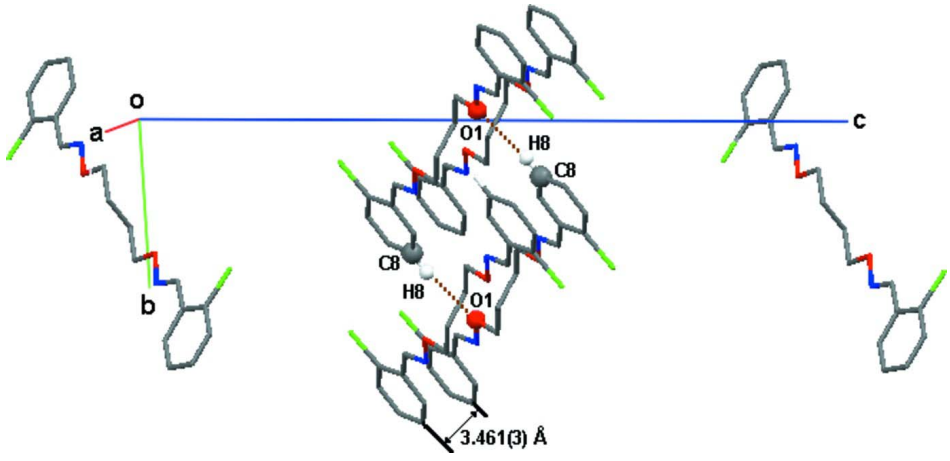

Figure 2

The packing diagram of the title compound showing intermolecular hydrogen bonds and π-π stacking interactions.

2,2′-Dichloro-1,1′-[(butane-1,4-diyldioxy)bis(nitrilomethylidyne)]dibenzene

Crystal data

$C_{18}H_{18}Cl_2N_2O_2$
$M_r = 365.24$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.5296\ (5)$ Å
$b = 6.6231\ (8)$ Å
$c = 29.963\ (2)$ Å
$\beta = 92.526\ (2)°$
$V = 898.02\ (16)$ Å^3
$Z = 2$
$F(000) = 380$
$D_x = 1.351$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2364 reflections
$\theta = 3.2–28.2°$
$\mu = 0.37$ mm^{-1}
$T = 298$ K
Needle, colorless
0.48 × 0.28 × 0.13 mm

Data collection

Bruker SMART 1000 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.841$, $T_{max} = 0.953$
4304 measured reflections
1531 independent reflections

1310 reflections with $I > 2\sigma(I)$
$R_{int} = 0.057$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.4°$
$h = -5 \rightarrow 5$
$k = -7 \rightarrow 5$
$l = -35 \rightarrow 34$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.074$
$wR(F^2) = 0.164$
$S = 1.10$
1531 reflections
109 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
$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 1.0643P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ 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	0.8257 (3)	0.7688 (2)	0.71001 (4)	0.0939 (5)
N1	0.4408 (7)	0.6370 (5)	0.57897 (10)	0.0570 (8)
O1	0.2543 (6)	0.7961 (4)	0.56512 (8)	0.0607 (7)
C1	0.0979 (8)	0.7356 (6)	0.52466 (11)	0.0598 (9)
H1A	−0.0273	0.6204	0.5304	0.072*
H1B	0.2370	0.6968	0.5025	0.072*
C2	−0.0885 (8)	0.9111 (6)	0.50759 (13)	0.0643 (10)
H2A	−0.2160	0.8650	0.4828	0.077*
H2B	−0.2141	0.9551	0.5311	0.077*
C3	0.5650 (8)	0.6708 (6)	0.61688 (12)	0.0570 (9)
H3	0.5233	0.7892	0.6321	0.068*
C4	0.7733 (7)	0.5252 (5)	0.63692 (11)	0.0521 (8)
C5	0.9101 (8)	0.5566 (6)	0.67866 (12)	0.0591 (9)
C6	1.1129 (9)	0.4202 (7)	0.69728 (13)	0.0697 (11)
H6	1.2038	0.4454	0.7252	0.084*
C7	1.1776 (10)	0.2505 (8)	0.67465 (16)	0.0820 (13)
H7	1.3133	0.1587	0.6871	0.098*
C8	1.0431 (11)	0.2122 (7)	0.63302 (16)	0.0826 (13)
H8	1.0867	0.0946	0.6177	0.099*
C9	0.8444 (9)	0.3496 (6)	0.61452 (12)	0.0636 (10)
H9	0.7562	0.3241	0.5865	0.076*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1045 (9)	0.1019 (10)	0.0731 (7)	0.0144 (8)	−0.0213 (6)	−0.0275 (7)
N1	0.0621 (18)	0.0562 (17)	0.0522 (17)	0.0094 (15)	−0.0026 (14)	0.0042 (14)
O1	0.0694 (16)	0.0559 (15)	0.0555 (14)	0.0125 (12)	−0.0105 (12)	−0.0013 (12)
C1	0.063 (2)	0.064 (2)	0.0514 (19)	−0.0040 (19)	−0.0059 (15)	0.0049 (18)
C2	0.056 (2)	0.075 (3)	0.061 (2)	−0.0034 (19)	−0.0106 (17)	0.018 (2)
C3	0.064 (2)	0.057 (2)	0.0498 (19)	0.0071 (18)	−0.0003 (16)	−0.0012 (16)
C4	0.0524 (19)	0.060 (2)	0.0436 (17)	0.0034 (17)	0.0046 (14)	0.0085 (16)
C5	0.059 (2)	0.073 (2)	0.0451 (18)	−0.0021 (19)	0.0045 (15)	0.0073 (18)
C6	0.061 (2)	0.092 (3)	0.055 (2)	0.003 (2)	−0.0034 (17)	0.020 (2)
C7	0.074 (3)	0.089 (3)	0.083 (3)	0.022 (3)	0.000 (2)	0.033 (3)
C8	0.097 (3)	0.071 (3)	0.080 (3)	0.030 (3)	0.011 (2)	0.013 (2)
C9	0.074 (2)	0.069 (2)	0.0483 (19)	0.010 (2)	0.0036 (17)	0.0047 (18)

Geometric parameters (Å, º)

Cl1—C5	1.742 (4)	C3—H3	0.9300
N1—C3	1.265 (4)	C4—C5	1.387 (5)
N1—O1	1.402 (4)	C4—C9	1.387 (5)
O1—C1	1.434 (4)	C5—C6	1.388 (5)
C1—C2	1.513 (5)	C6—C7	1.351 (6)
C1—H1A	0.9700	C6—H6	0.9300
C1—H1B	0.9700	C7—C8	1.387 (7)
C2—C2^{i}	1.506 (8)	C7—H7	0.9300
C2—H2A	0.9700	C8—C9	1.379 (5)
C2—H2B	0.9700	C8—H8	0.9300
C3—C4	1.460 (5)	C9—H9	0.9300
C3—N1—O1	111.8 (3)	C5—C4—C3	121.8 (3)
N1—O1—C1	108.0 (3)	C9—C4—C3	121.0 (3)
O1—C1—C2	108.6 (3)	C4—C5—C6	121.7 (4)
O1—C1—H1A	110.0	C4—C5—Cl1	120.5 (3)
C2—C1—H1A	110.0	C6—C5—Cl1	117.8 (3)
O1—C1—H1B	110.0	C7—C6—C5	119.6 (4)
C2—C1—H1B	110.0	C7—C6—H6	120.2
H1A—C1—H1B	108.4	C5—C6—H6	120.2
C2^{i}—C2—C1	114.0 (4)	C6—C7—C8	120.4 (4)
C2^{i}—C2—H2A	108.8	C6—C7—H7	119.8
C1—C2—H2A	108.8	C8—C7—H7	119.8
C2^{i}—C2—H2B	108.8	C9—C8—C7	119.6 (4)
C1—C2—H2B	108.8	C9—C8—H8	120.2
H2A—C2—H2B	107.7	C7—C8—H8	120.2
N1—C3—C4	120.4 (3)	C8—C9—C4	121.3 (4)
N1—C3—H3	119.8	C8—C9—H9	119.3
C4—C3—H3	119.8	C4—C9—H9	119.3
C5—C4—C9	117.3 (3)		

C3—N1—O1—C1	−173.4 (3)	C3—C4—C5—Cl1	2.6 (5)
N1—O1—C1—C2	−176.2 (3)	C4—C5—C6—C7	−0.9 (6)
O1—C1—C2—C2^{i}	66.7 (5)	Cl1—C5—C6—C7	178.0 (3)
O1—N1—C3—C4	−178.7 (3)	C5—C6—C7—C8	0.1 (7)
N1—C3—C4—C5	−179.1 (4)	C6—C7—C8—C9	0.7 (7)
N1—C3—C4—C9	1.5 (6)	C7—C8—C9—C4	−0.7 (7)
C9—C4—C5—C6	0.8 (5)	C5—C4—C9—C8	−0.1 (6)
C3—C4—C5—C6	−178.6 (3)	C3—C4—C9—C8	179.4 (4)
C9—C4—C5—Cl1	−178.0 (3)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O1^{ii}	0.93	2.66	3.581 (5)	171

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