Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*,*E*)-*N*,*N*′-Bis(3,4,5-trimethoxybenzylidene)ethylenediamine

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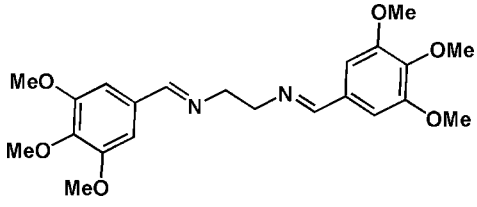
Received 12 October 2007; accepted 15 October 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.121; data-to-parameter ratio = 13.8.

The title compound, $C_{22}H_{28}N_2O_6$, prepared by a condensation reaction between 3,4,5-trimethoxybenzaldehyde and ethylenediamine, acts as an important precursor for the synthesis of Schiff base complexes. The molecule is located on a centre of inversion with one half-molecule in the asymmetric unit. Both C=N double bonds are in a *trans* configuration.

Related literature

For general background, see Khalaji *et al.* (2006, 2007). For related structures, see Li *et al.*(2006), Yang *et al.* (2007), Bomfim *et al.* (2005), Glidewell *et al.* (2006), Sun *et al.* (2004), Xiao *et al.* (2006) and Bahron *et al.* (2007).



Experimental

Crystal data

$C_{22}H_{28}N_2O_6$
M_r = 416.46
Monoclinic, $C2/c$
a = 32.008 (8) Å
b = 4.9175 (12) Å
c = 13.945 (4) Å
β = 95.326 (4)°
V = 2185.4 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
0.24 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.978, T_{max} = 0.984
5347 measured reflections
1933 independent reflections
1321 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.121
S = 1.03
1933 reflections
140 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2540).

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 doi:10.1107/S1600536807050532

supplementary materials

Acta Cryst. (2007). E**63**, o4389 [**doi:10.1107/S1600536807050532**]

(*E*,*E*)-*N*,*N*'-Bis(3,4,5-trimethoxybenzylidene)ethylenediamine

A. D. Khalaji, K. Brad and Y. Zhang

Comment

Schiff bases are used extensively as ligands in the field of coordination chemistry, because they are potentially capable of forming stable complexes with metal ions, such as Ag(I) and Cu(I) (Khalaji *et al.*, 2006, and 2007). The crystal structures of some diamine Schiff bases have been reported (Li *et al.*, 2006.; Yang *et al.*, 2007.; Bomfim *et al.*, 2005.; Glidewell *et al.*, 2006.; Sun *et al.*, 2004.; Xiao *et al.*, 2006.; Bahron *et al.*, 2007). Here we report the crystal structure of the diamine Schiff base, *N*,*N*'-Bis(3,4,5-trimethoxybenzylidene) ethylenediamine.

In *N*,*N*'-Bis(3,4,5-trimethoxybenzylidene)ethylenediamine (Fig. 1), two 3,4,5-trimethoxybenzylidene groups are bridged by the ethylenediamine fragment *via* two C=N double bonds. All the bond lengths and angles are within normal ranges. The N(1)=C(2) bond length of 1.257 (3)Å conforms to the value for a double bond, while the C(1)—N(1) bond length of 1.463 (2)Å conforms to the value for a single bond and are comparable to the corresponding values observed in *N*,*N*'-bis(3,4-dimethoxybenzylidene) ethylenediamine (Li *et al.*, 2006) and *N*,*N*'-Bis(4-nitrobenzylidene) ethane-1,2-diamine (Sun *et al.*, 2004).

Experimental

Ethylenediamine (1 mmol, 60 mg) and 3,4,5-trimethoxybenzaldehyde (2 mmol, 392 mg) were dissolved in methanol (15 ml) at 328 K. The mixture was stirred for 15 min to give a clear and colorless solution. After the solution had been allowed to stand in air for 1 d, colorless crystals formed, in about 91% yield, on slow evaporation of the solvent.

Refinement

All H atoms were positioned geometrically (C—H=0.93–0.97 Å), and refined as riding with U_{iso}(H)=1.2U_{eq}(carrier) or 1.5$_{eq}$(methyl groups). The methyl groups were allowed to rotate but not to tip.

Figures

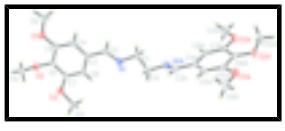


Fig. 1. A view of the molecular of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

(*E*,*E*)—*N*,*N*'-Bis(3,4,5-trimethoxybenzylidene)ethylenediamine

Crystal data

$C_{22}H_{28}N_2O_6$	F_{000} = 888
M_r = 416.46	D_x = 1.266 Mg m^{-3}

Monoclinic, $C2/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 32.008$ (8) Å	Cell parameters from 1598 reflections
$b = 4.9175$ (12) Å	$\theta = 2.9–23.8°$
$c = 13.945$ (4) Å	$\mu = 0.09$ mm^{-1}
$\beta = 95.326$ (4)°	$T = 294$ (2) K
$V = 2185.4$ (9) Å^3	Block, colorless
$Z = 4$	0.24 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1933 independent reflections
Radiation source: fine-focus sealed tube	1321 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
$T = 294(2)$ K	$\theta_{max} = 25.0°$
φ and ω scans	$\theta_{min} = 1.3°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -38\rightarrow37$
$T_{min} = 0.978$, $T_{max} = 0.984$	$k = -5\rightarrow3$
5347 measured reflections	$l = -15\rightarrow16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.038$	$w = 1/[\sigma^2(F_o^2) + (0.0692P)^2 + 0.163P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.121$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.03$	$\Delta\rho_{max} = 0.18$ e Å^{-3}
1933 reflections	$\Delta\rho_{min} = -0.14$ e Å^{-3}
140 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0070 (10)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-

ing *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}
O1	0.13907 (4)	0.4533 (3)	0.20272 (9)	0.0587 (4)
O2	0.20178 (3)	0.1274 (2)	0.15836 (8)	0.0436 (4)
O3	0.19633 (3)	−0.1625 (3)	−0.00262 (8)	0.0464 (4)
N1	0.04056 (5)	−0.0432 (4)	−0.14559 (12)	0.0584 (5)
C1	−0.00111 (6)	−0.0261 (6)	−0.19801 (15)	0.0719 (7)
H1A	−0.0179	−0.1793	−0.1805	0.086*
H1B	−0.0148	0.1391	−0.1795	0.086*
C2	0.04917 (5)	0.1186 (5)	−0.07681 (14)	0.0535 (6)
H2	0.0286	0.2422	−0.0625	0.064*
C3	0.08967 (5)	0.1253 (4)	−0.01738 (12)	0.0418 (5)
C4	0.09424 (5)	0.2955 (4)	0.06202 (12)	0.0447 (5)
H4	0.0722	0.4075	0.0758	0.054*
C5	0.13159 (5)	0.2998 (4)	0.12105 (12)	0.0415 (5)
C6	0.16516 (5)	0.1380 (4)	0.09854 (11)	0.0368 (4)
C7	0.16099 (5)	−0.0248 (3)	0.01564 (12)	0.0362 (4)
C8	0.12310 (5)	−0.0372 (4)	−0.04093 (12)	0.0396 (5)
H8	0.1199	−0.1524	−0.0940	0.047*
C9	0.10634 (6)	0.6251 (5)	0.22895 (15)	0.0632 (6)
H9A	0.0980	0.7463	0.1767	0.095*
H9B	0.1161	0.7290	0.2849	0.095*
H9C	0.0828	0.5163	0.2430	0.095*
C10	0.22852 (6)	0.3588 (4)	0.15304 (15)	0.0549 (6)
H10A	0.2379	0.3699	0.0898	0.082*
H10B	0.2523	0.3411	0.1999	0.082*
H10C	0.2133	0.5208	0.1659	0.082*
C11	0.19456 (6)	−0.3240 (4)	−0.08760 (14)	0.0551 (6)
H11A	0.1744	−0.4673	−0.0834	0.083*
H11B	0.2217	−0.4013	−0.0941	0.083*
H11C	0.1863	−0.2124	−0.1426	0.083*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0538 (8)	0.0740 (10)	0.0457 (8)	0.0172 (7)	−0.0087 (6)	−0.0196 (7)
O2	0.0399 (7)	0.0451 (8)	0.0420 (7)	−0.0001 (6)	−0.0160 (5)	0.0015 (6)
O3	0.0390 (7)	0.0554 (8)	0.0426 (7)	0.0107 (6)	−0.0082 (5)	−0.0079 (6)
N1	0.0357 (9)	0.0874 (13)	0.0490 (10)	−0.0026 (9)	−0.0134 (8)	0.0026 (10)
C1	0.0338 (11)	0.124 (2)	0.0542 (12)	−0.0077 (12)	−0.0137 (10)	−0.0004 (13)
C2	0.0343 (10)	0.0823 (16)	0.0427 (11)	0.0048 (10)	−0.0022 (8)	0.0055 (11)
C3	0.0334 (9)	0.0557 (12)	0.0351 (10)	−0.0013 (9)	−0.0037 (7)	0.0078 (9)
C4	0.0353 (9)	0.0568 (13)	0.0415 (10)	0.0082 (9)	0.0000 (8)	0.0047 (9)
C5	0.0425 (10)	0.0476 (11)	0.0334 (9)	0.0024 (9)	−0.0015 (7)	0.0006 (8)

C6	0.0347 (9)	0.0413 (10)	0.0323 (9)	0.0009 (8)	−0.0072 (7)	0.0060 (8)
C7	0.0336 (9)	0.0382 (10)	0.0354 (9)	0.0004 (8)	−0.0038 (7)	0.0057 (8)
C8	0.0385 (10)	0.0451 (11)	0.0335 (9)	−0.0031 (8)	−0.0051 (8)	0.0016 (8)
C9	0.0611 (13)	0.0706 (15)	0.0577 (13)	0.0142 (11)	0.0050 (10)	−0.0170 (11)
C10	0.0449 (11)	0.0576 (13)	0.0585 (13)	−0.0039 (10)	−0.0143 (9)	−0.0004 (10)
C11	0.0537 (12)	0.0619 (14)	0.0489 (12)	0.0092 (11)	−0.0003 (9)	−0.0131 (10)

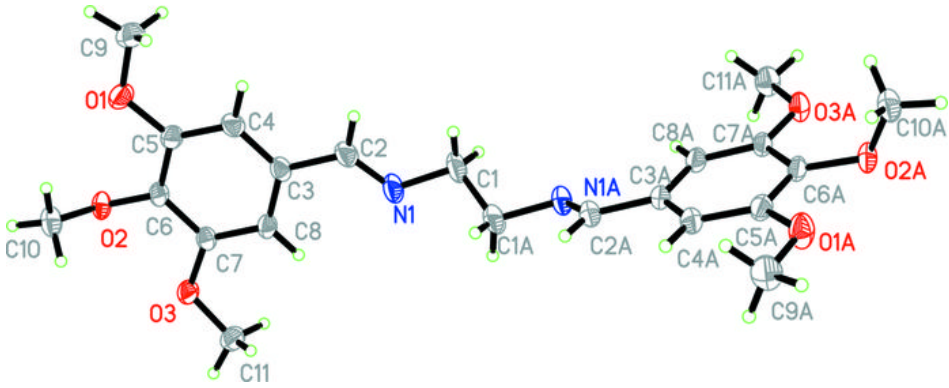
Geometric parameters (Å, °)

O1—C5	1.369 (2)	C4—C5	1.387 (2)
O1—C9	1.420 (2)	C4—H4	0.9300
O2—C6	1.3751 (18)	C5—C6	1.396 (2)
O2—C10	1.430 (2)	C6—C7	1.402 (2)
O3—C7	1.363 (2)	C7—C8	1.385 (2)
O3—C11	1.423 (2)	C8—H8	0.9300
N1—C2	1.257 (3)	C9—H9A	0.9600
N1—C1	1.463 (2)	C9—H9B	0.9600
C1—C1^{i}	1.458 (4)	C9—H9C	0.9600
C1—H1A	0.9700	C10—H10A	0.9600
C1—H1B	0.9700	C10—H10B	0.9600
C2—C3	1.473 (2)	C10—H10C	0.9600
C2—H2	0.9300	C11—H11A	0.9600
C3—C4	1.385 (3)	C11—H11B	0.9600
C3—C8	1.399 (2)	C11—H11C	0.9600
C5—O1—C9	117.79 (14)	O3—C7—C8	124.73 (16)
C6—O2—C10	114.78 (13)	O3—C7—C6	114.87 (13)
C7—O3—C11	117.60 (12)	C8—C7—C6	120.40 (16)
C2—N1—C1	118.03 (19)	C7—C8—C3	119.30 (17)
C1^{i}—C1—N1	111.8 (2)	C7—C8—H8	120.3
C1^{i}—C1—H1A	109.3	C3—C8—H8	120.3
N1—C1—H1A	109.3	O1—C9—H9A	109.5
C1^{i}—C1—H1B	109.3	O1—C9—H9B	109.5
N1—C1—H1B	109.3	H9A—C9—H9B	109.5
H1A—C1—H1B	107.9	O1—C9—H9C	109.5
N1—C2—C3	124.22 (19)	H9A—C9—H9C	109.5
N1—C2—H2	117.9	H9B—C9—H9C	109.5
C3—C2—H2	117.9	O2—C10—H10A	109.5
C4—C3—C8	120.44 (15)	O2—C10—H10B	109.5
C4—C3—C2	119.04 (17)	H10A—C10—H10B	109.5
C8—C3—C2	120.52 (17)	O2—C10—H10C	109.5
C3—C4—C5	120.33 (17)	H10A—C10—H10C	109.5
C3—C4—H4	119.8	H10B—C10—H10C	109.5
C5—C4—H4	119.8	O3—C11—H11A	109.5
O1—C5—C4	125.23 (16)	O3—C11—H11B	109.5
O1—C5—C6	114.99 (15)	H11A—C11—H11B	109.5
C4—C5—C6	119.78 (16)	O3—C11—H11C	109.5
O2—C6—C5	121.03 (15)	H11A—C11—H11C	109.5
O2—C6—C7	119.30 (14)	H11B—C11—H11C	109.5

C5—C6—C7	119.62 (15)		
C2—N1—C1—C1^{i}	132.79 (15)	C4—C5—C6—O2	−176.60 (16)
C1—N1—C2—C3	179.87 (18)	O1—C5—C6—C7	−179.22 (15)
N1—C2—C3—C4	−174.11 (19)	C4—C5—C6—C7	0.9 (3)
N1—C2—C3—C8	6.1 (3)	C11—O3—C7—C8	2.1 (2)
C8—C3—C4—C5	−2.2 (3)	C11—O3—C7—C6	−177.88 (15)
C2—C3—C4—C5	177.99 (17)	O2—C6—C7—O3	−6.1 (2)
C9—O1—C5—C4	−0.9 (3)	C5—C6—C7—O3	176.40 (15)
C9—O1—C5—C6	179.21 (16)	O2—C6—C7—C8	173.94 (15)
C3—C4—C5—O1	−177.88 (16)	C5—C6—C7—C8	−3.6 (2)
C3—C4—C5—C6	2.0 (3)	O3—C7—C8—C3	−176.62 (15)
C10—O2—C6—C5	−76.5 (2)	C6—C7—C8—C3	3.4 (2)
C10—O2—C6—C7	105.98 (18)	C4—C3—C8—C7	−0.5 (3)
O1—C5—C6—O2	3.3 (2)	C2—C3—C8—C7	179.31 (17)

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

Fig. 1


C9
O1
C5
C4
C2
C3
N1
C1
N1A
C1A
C2A
C3A
C8A
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O3A
C10A
O2A
C6A
C5A
O1A
C4A
C9A
C10
O2
C6
C8
C7
O3
C11