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Bis[4-(2-hydroxy-3-methoxybenzylideneamino)phenyl] ether

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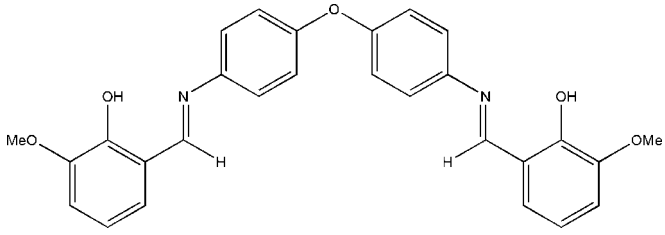
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.062; wR factor = 0.166; data-to-parameter ratio = 16.3.

The title compound, $C_{28}H_{24}N_2O_5$, a flexible Schiff base ligand, was prepared in high yield by a Schiff base condensation of 3-methoxysalicylaldehyde and bis(4-aminophenyl) ether in methanol. The molecule lies on a twofold rotation axis, and each half exhibits an imine E configuration and an O—H···N hydrogen bond. The dihedral angle between the two benzene rings attached to the central O atom is 69.22 (6)°, and that between each of these rings and the other benzene ring in the same half of the molecule is 24.29 (11)°, illustrating the degree of twisting of the flexible molecule.

Related literature

For related literature, see: Chu *et al.* (2007); Guo *et al.* (2002); He *et al.* (2000); Tesouro Vallina *et al.* (2001); Yoshida *et al.* (1999).



Experimental

Crystal data

$C_{28}H_{24}N_2O_5$
M_r = 468.49
Monoclinic, $C2/c$
a = 15.585 (7) Å
b = 7.578 (4) Å
c = 19.859 (9) Å
β = 92.760 (8)°
V = 2342.7 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
8736 measured reflections
2689 independent reflections
2016 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.166
S = 1.11
2689 reflections
165 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H3···N1	0.84	1.87	2.611 (2)	147

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2200).

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supporting information

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S1. Comment

Within the field of supramolecular inorganic chemistry, self-assembly is one of the most efficient methods for complex architectures comprising spatially and geometrically well defined arrays of metal ions. Because of easy syntheses by simple one-pot condensation reactions between aldehydes (or ketones) and amines and their coordinating ability with metal ions, multidentate Schiff base ligands such as pyridylimines (He *et al.*, 2000; Guo *et al.*, 2002; Tesouro Vallina *et al.*, 2001) and salicyladimines (Yoshida *et al.*, 1999; Chu *et al.*, 2007) were designed and used to prepare complexes in recent years. Here we report the synthesis and structure of a new flexible Schiff base ligand, bis(*N*-(3-methoxysalicylidene)-4-aminophenyl) ether. The molecule lies on a twofold rotation axis, and each half exhibits an imine E configuration and an O—H···N hydrogen bond. The dihedral angle between the two benzene rings attached to the central O atom is 69.22 (6)°, and that between each of these rings and the other benzene ring in the same half of the molecule is 24.29 (11)°, illustrating the degree of twisting of the flexible molecule. The bond lengths and angles are in agreement with those reported for other salicyladimines ligands (Chu *et al.*, 2007).

S2. Experimental

The title compound was prepared by a Schiff-base condensation of 3-methoxysalicylaldehyde (3.04 g, 20 mmol) and bis-(4-aminophenyl) ether (2.02 g, 10 mmol) in methanol (40 ml). The solution was stirred and refluxed for 1 day. The orange precipitate was filtered off, washed with a small amount of methanol and dried *in vacuo*. Yield: 91%. Well shaped orange crystals suitable for X-ray diffraction analysis were grown by slow evaporation of a chloroform solution of the title compound at room temperature.

S3. Refinement

H atoms were positioned geometrically (C—H = 0.96 Å; O—H = 0.84 Å), assigned isotropic displacement parameters equal to $1.2U_{eq}$ of the parent atoms, and allowed to ride on these parent atoms.

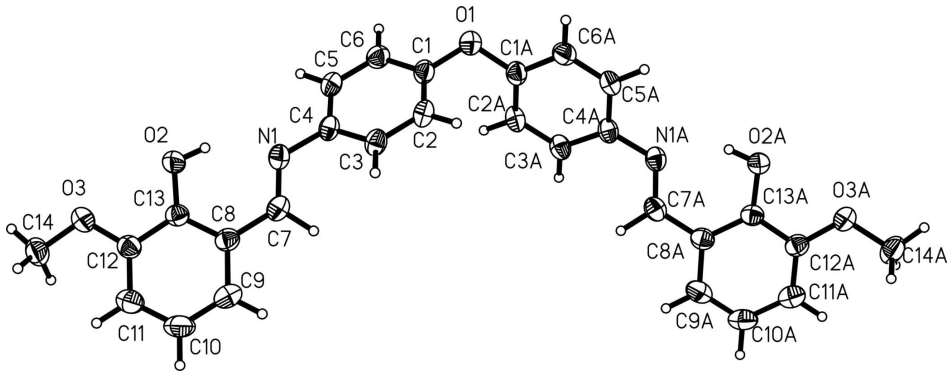

Figure 1

The molecular structure of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code A: 2-x, y, 3/2-z.]

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Crystal data

$C_{28}H_{24}N_2O_5$
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$b = 7.578\ (4)$ Å
$c = 19.859\ (9)$ Å
$\beta = 92.760\ (8)°$
$V = 2342.7\ (19)$ Å^3
$Z = 4$
$F(000) = 984$
$D_x = 1.328$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2635 reflections
$\theta = 1.7–25.1°$
$\mu = 0.09$ mm^{-1}
$T = 173$ K
Prism, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
8736 measured reflections
2689 independent reflections
2016 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.6°$
$h = -20\rightarrow19$
$k = -9\rightarrow9$
$l = -22\rightarrow25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.166$
$S = 1.11$
2689 reflections
165 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0739P)^2 + 0.5764P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.15$ 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}
N1	0.88064 (10)	0.0091 (2)	0.52377 (7)	0.0561 (4)
O1	1.0000	−0.3717 (3)	0.7500	0.0694 (6)
O2	0.78593 (9)	0.02484 (16)	0.41186 (7)	0.0660 (4)
H3	0.8101	−0.0219	0.4461	0.079*
C4	0.91503 (11)	−0.0853 (3)	0.58068 (8)	0.0527 (5)
C13	0.80476 (12)	0.1980 (2)	0.41092 (9)	0.0521 (4)
O3	0.71769 (10)	0.21180 (19)	0.31172 (8)	0.0739 (5)
C12	0.77074 (13)	0.3000 (2)	0.35765 (10)	0.0572 (5)
C3	0.98968 (12)	−0.0344 (3)	0.61733 (9)	0.0574 (5)
H8	1.0210	0.0659	0.6036	0.069*
C6	0.90099 (13)	−0.3309 (3)	0.65622 (10)	0.0610 (5)
H9	0.8709	−0.4333	0.6694	0.073*
C5	0.87355 (12)	−0.2368 (3)	0.59967 (10)	0.0591 (5)
H10	0.8250	−0.2772	0.5733	0.071*
C8	0.85726 (12)	0.2774 (2)	0.46152 (10)	0.0564 (5)
C2	1.01805 (12)	−0.1291 (3)	0.67334 (9)	0.0581 (5)
H12	1.0689	−0.0941	0.6981	0.070*
C7	0.89139 (13)	0.1765 (3)	0.51873 (10)	0.0602 (5)
C1	0.97295 (13)	−0.2738 (3)	0.69341 (9)	0.0562 (5)
C11	0.79102 (15)	0.4744 (3)	0.35347 (12)	0.0718 (6)
H15	0.7685	0.5426	0.3165	0.086*
C14	0.66750 (17)	0.3156 (3)	0.26521 (12)	0.0825 (7)
H16A	0.6388	0.4096	0.2895	0.124*
H16B	0.6243	0.2408	0.2418	0.124*
H16C	0.7048	0.3679	0.2322	0.124*
C9	0.87598 (16)	0.4578 (3)	0.45639 (12)	0.0744 (6)
H17	0.9110	0.5138	0.4905	0.089*
C10	0.84410 (17)	0.5530 (3)	0.40260 (14)	0.0825 (7)
H18	0.8584	0.6743	0.3988	0.099*
H4	0.9250 (14)	0.249 (3)	0.5555 (12)	0.082 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0574 (9)	0.0669 (10)	0.0438 (8)	−0.0043 (8)	0.0010 (7)	−0.0009 (7)
O1	0.0892 (14)	0.0622 (12)	0.0554 (11)	0.000	−0.0097 (10)	0.000

O2	0.0793 (10)	0.0517 (7)	0.0653 (9)	−0.0103 (7)	−0.0143 (7)	0.0074 (6)
C4	0.0519 (10)	0.0664 (11)	0.0400 (9)	−0.0021 (8)	0.0041 (7)	−0.0068 (8)
C13	0.0562 (10)	0.0481 (10)	0.0528 (10)	−0.0019 (8)	0.0097 (8)	0.0000 (8)
O3	0.0880 (11)	0.0637 (9)	0.0679 (9)	0.0048 (7)	−0.0170 (8)	0.0087 (7)
C12	0.0614 (11)	0.0546 (11)	0.0561 (11)	0.0051 (9)	0.0081 (9)	0.0020 (9)
C3	0.0554 (10)	0.0723 (12)	0.0448 (10)	−0.0124 (9)	0.0057 (8)	−0.0027 (9)
C6	0.0654 (12)	0.0565 (11)	0.0610 (12)	−0.0098 (9)	0.0007 (10)	−0.0014 (9)
C5	0.0557 (11)	0.0639 (11)	0.0566 (11)	−0.0071 (9)	−0.0074 (9)	−0.0077 (9)
C8	0.0591 (11)	0.0536 (10)	0.0571 (11)	0.0004 (8)	0.0091 (9)	−0.0054 (8)
C2	0.0528 (10)	0.0767 (13)	0.0446 (10)	−0.0101 (9)	−0.0009 (8)	−0.0068 (9)
C7	0.0628 (12)	0.0658 (12)	0.0521 (11)	−0.0022 (10)	0.0030 (9)	−0.0116 (9)
C1	0.0614 (11)	0.0625 (11)	0.0443 (10)	0.0019 (9)	−0.0013 (8)	−0.0048 (8)
C11	0.0819 (15)	0.0571 (12)	0.0768 (14)	0.0050 (11)	0.0087 (12)	0.0074 (11)
C14	0.1016 (18)	0.0836 (15)	0.0610 (13)	0.0289 (13)	−0.0081 (12)	0.0036 (11)
C9	0.0833 (15)	0.0583 (12)	0.0814 (15)	−0.0083 (11)	0.0010 (12)	−0.0145 (11)
C10	0.1018 (18)	0.0466 (11)	0.0993 (19)	−0.0044 (12)	0.0076 (15)	0.0029 (12)

Geometric parameters (Å, º)

N1—C7	1.284 (3)	C6—C1	1.382 (3)
N1—C4	1.421 (2)	C6—H9	0.950
O1—C1^{i}	1.394 (2)	C5—H10	0.950
O1—C1	1.394 (2)	C8—C9	1.402 (3)
O2—C13	1.345 (2)	C8—C7	1.449 (3)
O2—H3	0.840	C2—C1	1.372 (3)
C4—C5	1.379 (3)	C2—H12	0.950
C4—C3	1.397 (3)	C7—H4	1.04 (2)
C13—C12	1.394 (3)	C11—C10	1.383 (3)
C13—C8	1.401 (3)	C11—H15	0.950
O3—C12	1.375 (2)	C14—H16A	0.980
O3—C14	1.419 (2)	C14—H16B	0.980
C12—C11	1.363 (3)	C14—H16C	0.980
C3—C2	1.379 (3)	C9—C10	1.363 (3)
C3—H8	0.950	C9—H17	0.950
C6—C5	1.381 (3)	C10—H18	0.950
C7—N1—C4	120.94 (16)	C1—C2—C3	120.11 (17)
C1^{i}—O1—C1	115.8 (2)	C1—C2—H12	119.9
C13—O2—H3	109.5	C3—C2—H12	119.9
C5—C4—C3	118.51 (17)	N1—C7—C8	122.55 (17)
C5—C4—N1	118.23 (16)	N1—C7—H4	122.3 (12)
C3—C4—N1	123.24 (18)	C8—C7—H4	115.2 (12)
O2—C13—C12	118.43 (17)	C2—C1—C6	120.57 (17)
O2—C13—C8	121.98 (16)	C2—C1—O1	121.33 (16)
C12—C13—C8	119.59 (17)	C6—C1—O1	118.06 (18)
C12—O3—C14	117.23 (17)	C12—C11—C10	120.5 (2)
C11—C12—O3	124.41 (18)	C12—C11—H15	119.8
C11—C12—C13	120.21 (19)	C10—C11—H15	119.8

O3—C12—C13	115.39 (17)	O3—C14—H16A	109.5
C2—C3—C4	120.22 (18)	O3—C14—H16B	109.5
C2—C3—H8	119.9	H16A—C14—H16B	109.5
C4—C3—H8	119.9	O3—C14—H16C	109.5
C5—C6—C1	118.99 (19)	H16A—C14—H16C	109.5
C5—C6—H9	120.5	H16B—C14—H16C	109.5
C1—C6—H9	120.5	C10—C9—C8	120.2 (2)
C4—C5—C6	121.44 (17)	C10—C9—H17	119.9
C4—C5—H10	119.3	C8—C9—H17	119.9
C6—C5—H10	119.3	C9—C10—C11	120.5 (2)
C13—C8—C9	118.89 (19)	C9—C10—H18	119.7
C13—C8—C7	121.02 (17)	C11—C10—H18	119.7
C9—C8—C7	120.09 (18)		

Symmetry code: (i) $-x+2, y, -z+3/2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H3···N1	0.84	1.87	2.611 (2)	147