Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-[({2-[(2-Hydroxy-5-methoxybenzylidene)amino]ethyl}imino)methyl]-4-methoxyphenol

Ali Ourari,[a] Lotfi Baameur,[a] Sofiane Bouacida[b]* and Kamel Ouari[a]

[a]Laboratoire d'Electrochimie, d'Ingénierie Moléculaire et de Catalyse Redox (LEIMCR), Faculté des Sciences de l'Ingénieur, Université Farhat Abbas, Sétif 19000, Algeria, and [b]Unité de Recherche de Chimie de l'Environnement et Moléculaire Structurale (CHEMS), Université Mentouri-Constantine, 25000 Algeria
Correspondence e-mail: bouacida_sofiane@yahoo.fr

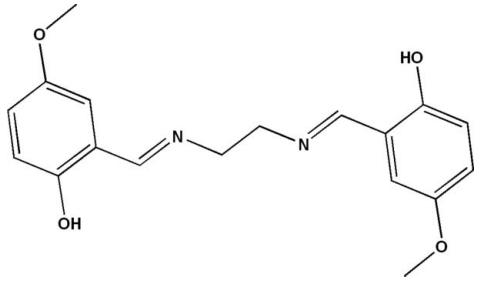
Received 23 April 2012; accepted 24 April 2012

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.054; wR factor = 0.167; data-to-parameter ratio = 15.0.

The asymmetric unit of the title compound, $C_{18}H_{20}N_2O_4$, contains one-half molecule with an inversion center located at the centroid of the molecule. In the crystal, molecules are linked by C—H$\cdots\pi$ interactions, forming layers parallel to (101). An intramolecular O—H$\cdots$N hydrogen bond also occurs.

Related literature

For the synthesis of similar compounds see: Srinivasan *et al.* (1986); Moutet & Ourari (1997); Ourari *et al.* (2008).



Experimental

Crystal data

$C_{18}H_{20}N_2O_4$
M_r = 328.36
Monoclinic, $P2_1/c$
a = 15.0040 (12) Å
b = 5.9722 (3) Å
c = 9.3128 (8) Å
β = 92.001 (3)°
V = 833.98 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 K
0.50 × 0.23 × 0.19 mm

Data collection

Nonius KappaCCD diffractometer
3001 measured reflections
1664 independent reflections
1097 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.167
S = 1.05
1664 reflections
111 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

Cg is the centroid of the C4–C9 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O5—H5$\cdots$N2	0.82	1.85	2.5844 (18)	148
C10—H10$C\cdots Cg^{i}$	0.96	2.64	3.521 (2)	152

Symmetry code: (i) $x, -y+\frac{1}{2}, z-\frac{3}{2}$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg & Berndt, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank the Algerian Ministère de l'Enseignement Supérieur et de la Recherche Scientifique for financial support and Professor L. Ouahab (Laboratoire des Sciences Chimiques, Rennes1, France) for his valuable contribution and insightful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2353).

References

Brandenburg, K. & Berndt, M. (2001). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Moutet, J. C. & Ourari, A. (1997). *Electrochim. Acta*, **42**, 2525–2531.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Ourari, A., Baameur, L., Bouet, G. & Khan, A. M. (2008). *J. Electrochem. Commun.* **10**, 1736–1739.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Srinivasan, K., Michaud, P. & Kochi, J. K. (1986). *J. Am. Chem. Soc.* **108**, 2309–2320.

supporting information

Acta Cryst. (2012). E**68**, o1760 [doi:10.1107/S1600536812018405]

2-[({2-[(2-Hydroxy-5-methoxybenzylidene)amino]ethyl}imino)methyl]-4-methoxyphenol

Ali Ourari, Lotfi Baameur, Sofiane Bouacida and Kamel Ouari

S1. Comment

The tetradentate Schiff base ligands derived from salicylaldehyde derivatives and diamino compounds have been found to be excellent chelating agents for most applications in coordination chemistry such as in catalysis (Srinivasan *et al.*, 1986) and electrocatalysis (Moutet & Ourari, 1997; Ourari *et al.*, 2008). Here, we report the synthesis of the title compound and its crystal structure.

The molecular structure of (I), and the atomic numbering used, is illustrated in Fig. 1. The asymmetric unit of the title compound, consists of one-half of the molecule, with the other half generated by a crystallographic inversion center. The crystal packing in the title structure can be described by a zigzag layers parallel to (101) plane (Fig. 2). There is one intramolecular O—H···N hydrogen bonding in this packing (Table 1, Fig. 2), which it is stabilized C—H···π and Van der Walls interactions (table 1) All these interactions link the molecules within the layers and also link the layers together and reinforcing the cohesion of the structure.

S2. Experimental

60 mg of 1,2-diaminoethane (1 mmol) were dissolved in 10 ml of absolute ethanol. This solution was drop wise added, under stirring, to an ethanolic solution (10 ml) containing 304 mg of 5-methoxysalicylaldehyde (2 mmol). This mixture was refluxed for 1 h after which a yellow precipitate is formed, recovered by filtration, washed several times with diethyl oxide and dried to yield 282 mg (86%) of the title compound. The suitable crystals for X-ray analysis were obtained by slow evaporation from a mixture of solvents ethanol/dichloromethane (8/2, *v*/*v*).

S3. Refinement

The H atoms were localized on Fourier maps but introduced in calculated positions and treated as riding on their parent atoms (C and O) with C—H = 0.93 Å (methine, aromatic), 0.96 Å (methyl), 0.97 Å (methylene) and O—H = 0.82 Å (hydroxyl) with U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(O).

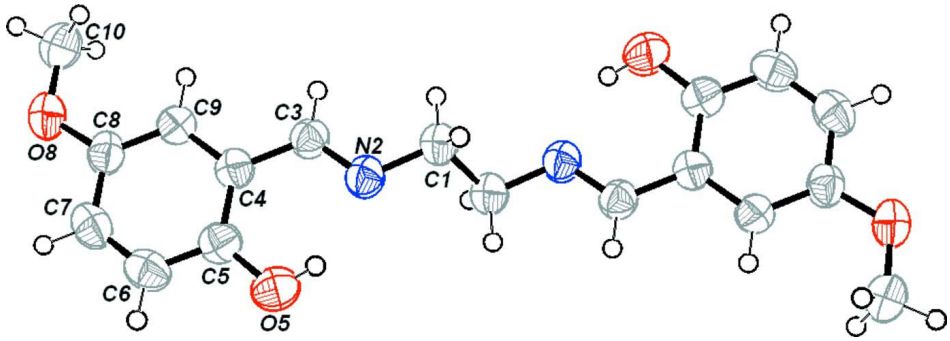


Figure 1

The molecular geometry of (I) with the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. Only the contents of the asymmetric unit are numbered.

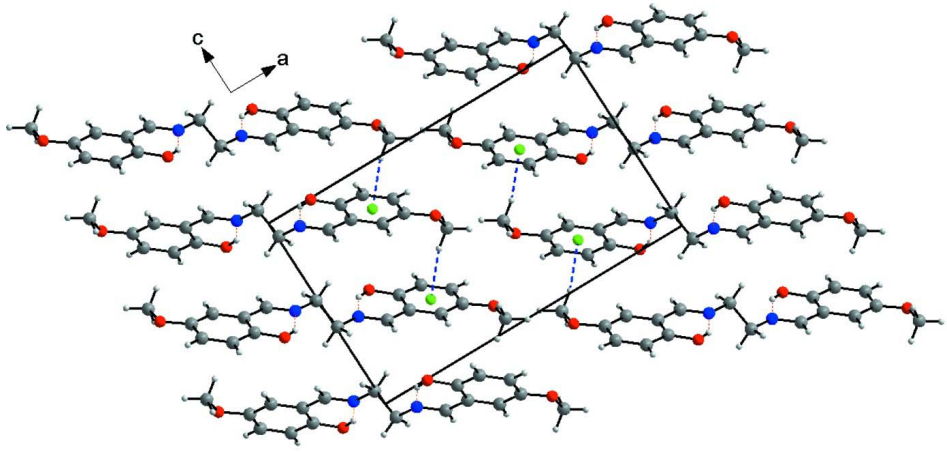


Figure 2

Diagram of layered packing parallel to (101) plane and showing O—H···N and C—H···π interactions.

2-[({2-[(2-Hydroxy-5-methoxybenzylidene)amino]ethyl}imino)methyl]- 4-methoxyphenol

Crystal data

$C_{18}H_{20}N_2O_4$
$M_r = 328.36$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.0040\ (12)$ Å
$b = 5.9722\ (3)$ Å
$c = 9.3128\ (8)$ Å
$\beta = 92.001\ (3)°$
$V = 833.98\ (11)$ Å^3
$Z = 2$
$F(000) = 348$
$D_x = 1.308$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1761 reflections
$\theta = 1.0–26.4°$
$\mu = 0.09$ mm^{-1}
$T = 295$ K
Prism, colorless
0.50 × 0.23 × 0.19 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Enraf–Nonius FR590
Graphite monochromator
Detector resolution: 9 pixels mm^{-1}
CCD rotation images, thick slices scans
3001 measured reflections
1664 independent reflections
1097 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$

θ_{max} = 26.4°, θ_{min} = 2.7°
h = −18→18
k = −6→7
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.167
S = 1.05
1664 reflections
111 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
$w = 1/[\sigma^2(F_o^2) + (0.101P)^2 + 0.0006P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.08 (2)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.00544 (12)	0.5332 (3)	0.42227 (18)	0.0554 (5)
H1A	0.0237	0.404	0.3674	0.066*
H1B	−0.0511	0.586	0.3816	0.066*
C3	0.13689 (11)	0.6835 (3)	0.32973 (18)	0.0500 (5)
H3	0.1399	0.5537	0.2749	0.06*
C4	0.20642 (11)	0.8520 (3)	0.31789 (17)	0.0488 (5)
C5	0.20521 (13)	1.0483 (3)	0.40154 (19)	0.0545 (5)
C6	0.27360 (14)	1.2037 (3)	0.3894 (2)	0.0620 (5)
H6	0.2731	1.3341	0.4439	0.074*
C7	0.34154 (14)	1.1672 (3)	0.2983 (2)	0.0623 (5)
H7	0.3863	1.2739	0.2909	0.075*
C8	0.34469 (12)	0.9719 (3)	0.21605 (18)	0.0542 (5)
C9	0.27715 (11)	0.8166 (3)	0.22553 (18)	0.0515 (5)
H9	0.2784	0.687	0.1702	0.062*
C10	0.42592 (14)	0.7497 (3)	0.0532 (3)	0.0764 (7)
H10A	0.4313	0.6263	0.119	0.115*
H10B	0.4781	0.7572	−0.0033	0.115*
H10C	0.3742	0.7287	−0.0089	0.115*
N2	0.07208 (9)	0.7091 (2)	0.41315 (16)	0.0544 (5)
O5	0.13958 (10)	1.0889 (2)	0.49361 (15)	0.0712 (5)
H5	0.1037	0.9853	0.4903	0.107*

O8	0.41724 (10)	0.9514 (2)	0.13122 (15)	0.0727 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0480 (10)	0.0591 (10)	0.0594 (11)	−0.0045 (7)	0.0065 (8)	−0.0027 (8)
C3	0.0467 (10)	0.0539 (9)	0.0494 (10)	0.0010 (7)	0.0029 (8)	−0.0004 (7)
C4	0.0494 (10)	0.0483 (9)	0.0486 (10)	0.0007 (7)	0.0001 (8)	0.0034 (6)
C5	0.0598 (12)	0.0495 (10)	0.0541 (10)	0.0057 (8)	0.0025 (9)	0.0020 (7)
C6	0.0718 (13)	0.0466 (9)	0.0672 (12)	−0.0003 (8)	−0.0038 (10)	−0.0033 (8)
C7	0.0652 (12)	0.0518 (10)	0.0696 (12)	−0.0104 (8)	−0.0025 (10)	0.0063 (9)
C8	0.0517 (11)	0.0589 (10)	0.0521 (10)	−0.0062 (7)	0.0040 (8)	0.0086 (7)
C9	0.0546 (11)	0.0514 (9)	0.0488 (10)	−0.0030 (7)	0.0044 (9)	−0.0013 (7)
C10	0.0628 (13)	0.0883 (14)	0.0793 (15)	−0.0105 (10)	0.0209 (11)	−0.0118 (11)
N2	0.0486 (9)	0.0571 (9)	0.0578 (9)	−0.0012 (6)	0.0068 (7)	−0.0004 (6)
O5	0.0726 (10)	0.0631 (8)	0.0789 (10)	0.0062 (6)	0.0182 (8)	−0.0140 (6)
O8	0.0643 (9)	0.0764 (9)	0.0786 (10)	−0.0194 (7)	0.0215 (7)	−0.0033 (7)

Geometric parameters (Å, º)

C1—N2	1.455 (2)	C6—H6	0.93
C1—C1^{i}	1.515 (3)	C7—C8	1.397 (2)
C1—H1A	0.97	C7—H7	0.93
C1—H1B	0.97	C8—O8	1.373 (2)
C3—N2	1.275 (2)	C8—C9	1.379 (2)
C3—C4	1.456 (2)	C9—H9	0.93
C3—H3	0.93	C10—O8	1.415 (2)
C4—C9	1.405 (2)	C10—H10A	0.96
C4—C5	1.408 (2)	C10—H10B	0.96
C5—O5	1.350 (2)	C10—H10C	0.96
C5—C6	1.391 (3)	O5—H5	0.82
C6—C7	1.366 (3)		
N2—C1—C1^{i}	109.99 (18)	C6—C7—C8	120.91 (16)
N2—C1—H1A	109.7	C6—C7—H7	119.5
C1^{i}—C1—H1A	109.7	C8—C7—H7	119.5
N2—C1—H1B	109.7	O8—C8—C9	125.19 (16)
C1^{i}—C1—H1B	109.7	O8—C8—C7	115.64 (15)
H1A—C1—H1B	108.2	C9—C8—C7	119.18 (17)
N2—C3—C4	121.80 (15)	C8—C9—C4	120.69 (16)
N2—C3—H3	119.1	C8—C9—H9	119.7
C4—C3—H3	119.1	C4—C9—H9	119.7
C9—C4—C5	119.26 (16)	O8—C10—H10A	109.5
C9—C4—C3	120.03 (15)	O8—C10—H10B	109.5
C5—C4—C3	120.68 (16)	H10A—C10—H10B	109.5
O5—C5—C6	119.26 (16)	O8—C10—H10C	109.5
O5—C5—C4	121.64 (16)	H10A—C10—H10C	109.5
C6—C5—C4	119.10 (17)	H10B—C10—H10C	109.5

C7—C6—C5	120.86 (16)	C3—N2—C1	119.31 (15)
C7—C6—H6	119.6	C5—O5—H5	109.5
C5—C6—H6	119.6	C8—O8—C10	117.41 (14)
N2—C3—C4—C9	179.47 (16)	C6—C7—C8—C9	1.2 (3)
N2—C3—C4—C5	1.4 (3)	O8—C8—C9—C4	178.87 (17)
C9—C4—C5—O5	−178.94 (15)	C7—C8—C9—C4	−0.8 (3)
C3—C4—C5—O5	−0.9 (3)	C5—C4—C9—C8	−0.2 (2)
C9—C4—C5—C6	0.8 (2)	C3—C4—C9—C8	−178.27 (15)
C3—C4—C5—C6	178.82 (15)	C4—C3—N2—C1	−178.95 (15)
O5—C5—C6—C7	179.38 (17)	C1^{i}—C1—N2—C3	127.0 (2)
C4—C5—C6—C7	−0.3 (3)	C9—C8—O8—C10	−3.6 (3)
C5—C6—C7—C8	−0.7 (3)	C7—C8—O8—C10	176.06 (17)
C6—C7—C8—O8	−178.45 (17)		

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

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C4–C9 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···N2	0.82	1.85	2.5844 (18)	148
C10—H10*C*···*Cg*^{ii}	0.96	2.64	3.521 (2)	152

Symmetry code: (ii) *x*, −*y*+1/2, *z*−3/2.