Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

trans-*N*,*N*′-Bis(3-methoxysalicylidene)cyclohexane-1,2-diamine

Mehmet Aslantaş,[a]* Mehmet Tümer,[b] Ertan Şahin[c] and Ferhan Tümer[c]

[a]Department of Physics, Faculty of Sciences and Arts, University of Kahramanmaras Sutcu Imam, Avsar Campus 46100, Kahramanmaras, Turkey, [b]Department of Chemistry, Faculty of Sciences and Arts, University of Kahramanmaras Sutcu Imam, Avsar Campus 46100, Kahramanmaras, Turkey, and [c]Department of Chemistry, Faculty of Sciences and Arts, University of Atatürk, 25240 Erzurum, Turkey

Correspondence e-mail: aslantas@hacettepe.edu.tr

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.061
wR factor = 0.178
Data-to-parameter ratio = 24.0

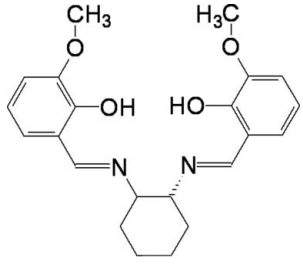
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 19 December 2006
Accepted 5 January 2007

The title compound, $C_{22}H_{26}N_2O_4$, was prepared by reaction of *trans*-1,2-cyclohexanediamine and 2-hydroxy-3-methoxybenzaldehyde. The molecular structure is stabilized by intramolecular O—H···N hydrogen bonds.

Comment

Schiff base ligands derived from salicylaldehyde and chiral amines have been widely applied in enantioselective cyclopropanation of styrenes (Zhang *et al.*, 1997; Holland *et al.*, 1981), asymmetric aziridination of olefins (Kenneth *et al.*, 1992), enantioselective epoxidation (Kureshy *et al.*, 1999; Hosoya *et al.*, 1999), enantioselective ring opening of epoxides (Kim *et al.*, 1999), borohydride reduction of aromatic ketones, asymmetric oxidation of methyl phenyl sulfide (Sasaki *et al.*, 1991), enantioselective oxidation of silyl enol (Waldemar *et al.*, 1998) and trimethylsilylcyanation of benzaldehydes. In particular, the Merck company has successfully developed a process for the industrial manufacture of the antibacterial drug Cilastatin using chiral copper(II) Schiff base complexes derived from salicylaldehyde and chiral amine (Aratani, 1985). We present here the crystal structure of the title compound, (I), which contains a *trans*-1,2-cyclohexanediamine and two 2-hydroxy-3-methoxybenzaldehyde groups (Fig. 1). The crystal structure of the compound containing a chiral amine, (1*R*,2*R*)-(−)-1,2-cyclohexanediamine, has been reported by Mohamed *et al.* (2003).



(I)

All bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987) and agree with those reported for structures containing the 2-hydroxy-3-methoxybenzaldehyde group (Jing *et al.*, 2005; Chen *et al.*, 2006). The cyclohexane ring adopts a chair conformation. Each 2-hydroxy-3-methoxybenzaldehyde group in the molecule is nearly planar; the maximum deviations from the mean plane are 0.064 (3) and 0.034 (4)Å for atoms C1 and C22, respectively. The dihedral angle between the planes of these groups is 62.65 (4)°. The molecular structure is stabilized by two intramolecular O—H···N hydrogen bonds (Table 1).

Experimental

The title compound was prepared by a known method (Tümer, 2000). *o*-Vanillin (2 mmol, 0.304 g) in ethanol (20 ml) and *trans*-1,2-cyclohexanediamine (1 mmol, 0.114 g) in ethanol (20 ml) were mixed and refluxed for about 4 h at 358 K. The color of the solution changed to pale yellow. After cooling the solution, the resulting precipitate was filtered and washed with cold ethanol. Compound (I) was obtained by crystallization from an ethanol solution after a few days (yield 85%; m.p. 418 K). Elemental analysis calculated: C 69.09, H 6.85, N 7.32%; found: C 69.05, H 6.88, N 7.35%. ^{1}H NMR: (CD_3OD): δ 8.33 (*s*, CH═N, 2H), 6.62–6.95 (*m*, Ar–H, 6H), 3.81 (*s*, OCH_3, 6H), 3.35–1.25 (*m*, CH/CH_2, 10H); ^{13}C NMR: (CD_3OD): δ 168.68 (CH═N), 117.62–157.33 (Ar–C), 58.39 (OCH_3), 52.07–27.00 (CH/CH_2). MS (LC/MS APCI): *m*/*z* 391 $[M + 1]^+$.

Crystal data

$C_{22}H_{26}N_2O_4$
M_r = 382.45
Triclinic, $P\overline{1}$
a = 10.090 (5) Å
b = 10.970 (5) Å
c = 11.678 (5) Å
α = 115.254 (5)°
β = 112.401 (5)°
γ = 95.258 (5)°
V = 1029.7 (8) Å^3
Z = 2
D_x = 1.234 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
Block, pale yellow
0.2 × 0.2 × 0.2 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer
ω scans
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.973, T_{max} = 0.983
30731 measured reflections
6285 independent reflections
4115 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 30.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.179
S = 1.05
6285 reflections
262 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0753P)^2 + 0.1247P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.039 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H7···N1	0.82	1.86	2.590 (3)	147
O3—H21···N2	0.82	1.92	2.644 (2)	147

The H atoms attached to atoms C8 and C15 were located in a difference map and refined freely. The other H atoms were placed in geometrically idealized positions (C—H = 0.93–0.98 and O—H = 0.82 Å) and treated as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(O, methyl C).

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

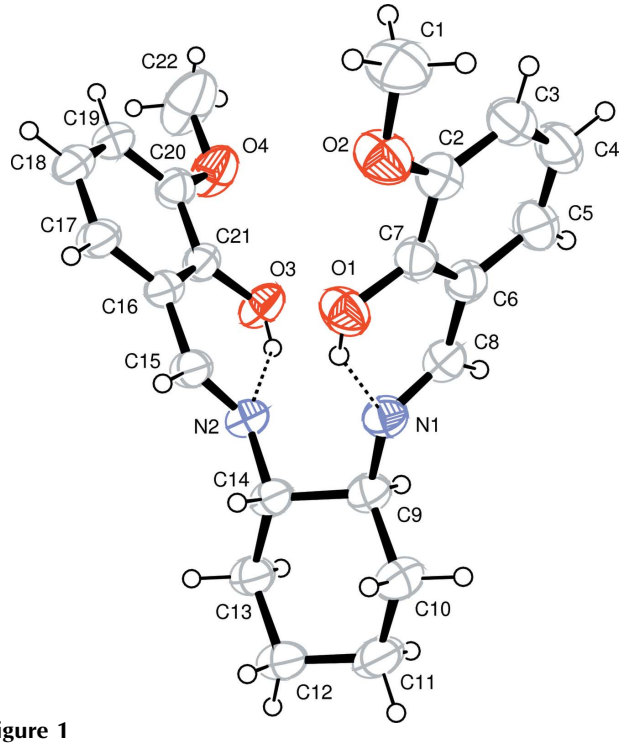


Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 40% probability level. Dashed lines indicate hydrogen bonds.

The authors are indebted to the Department of Chemistry, Atatürk University, Turkey, for the use of the X-ray diffractometer purchased under grant number 2003/219 of the University Research Fund.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* **2**, S1–19.
Aratani, T. (1985). *Pure Appl. Chem.* **57**, 1839–1844.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Chen, S.-W., Yin, H.-D., Wang, D.-Q., Kong, X. & Chen, X.-F. (2006). *Acta Cryst.* E**62**, o2043–o2044.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Holland, D., Laidler, D. A. & Milner, D. J. (1981). *J. Mol. Catal.* **11**, 119–127.
Hosoya, N., Kim, G. J. & Shin, J. H. (1999). *Catal. Lett.* **63**, 83–89.
Jing, Z.-L., Yu, M., Chen, X., Diao, C.-H., Deng, Q.-L. & Fan, Z. (2005). *Acta Cryst.* E**61**, o145–o146.
Kenneth, J. O., Shiow, J. W. & Cynthia, J. B. (1992). *Tetrahedron Lett.* **33**, 1001–1004.
Kim, G. J. & Shin, J. H. (1999). *Catal. Lett.* **63**, 83–89.
Kureshy, R. I., Khan, N. H. & Abdi, S. T. (1999). *J. Mol. Catal.* **150**, 163–173.
Mohamed, E. M., Muralidharan, S., Panchanatheswaran, K., Ramesh, R., Low, J. N. & Glidewell, C. (2003). *Acta Cryst.* C**59**, o367–o369.
Rigaku/MSC (2005). *CrystalClear*. Version 1.3.6. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sasaki, C., Nakajima, K. & Kojima, M. (1991). *Bull. Chem. Soc. Jpn*, **64**, 1318–1324.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tümer, M. (2000). *Synth. React. Inorg. Met.-Org. Chem.* **30**, 1139–1158.
Waldemar, A., Rainer, T. & Veit, R. S. (1998). *J. Am. Chem. Soc.* **120**, 708–714.
Zhang, J. X., Zhou, Y. & Cai, G. (1997). *J. Mol. Catal. (China)*, **11**, 41–44.