Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1112). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L. & Orpen, A. G. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

Bar, I. & Bernstein, J. (1982). *Acta Cryst.* **B38**, 121–125.

Baumeister, U., Hartung, H. & Gdaniec, M. (1987). *Acta Cryst.* **C43**, 1117–1119.

Bürgi, H.-B. & Dunitz, J. D. (1970). *Helv. Chim. Acta*, **53**, 1747–1764.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–389.

Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Nakai, H., Ezumi, K. & Shiro, M. (1981). *Acta Cryst.* **B37**, 193–197.

Nakai, H., Shiro, M., Ezumi, K., Sakata, S. & Kubota, T. (1976). *Acta Cryst.* **B32**, 1827–1833.

Ondráček, J., Kovářová, Z., Maixner, J. & Jursik, F., (1993). *Acta Cryst.* **C49**, 1948–1949.

Spek, A. L. (1992). *PLUTON92. Molecular Graphics Program*. Univ. of Utrecht, The Netherlands.

Steiner, T. & Saenger, W. (1993). *J. Am. Chem. Soc.* **115**, 4540–4547.

Stoe & Cie (1992*a*). *DIF4. Diffractometer Control Program*. Version 7.0. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (1992*b*). *REDU4. Data Reduction Program*. Version 7.0. Stoe & Cie, Darmstadt, Germany.

Acta Cryst. (1995). **C51**, 1878–1880

N,*N*′-[Dithiobis(*o*-phenylene)]bis(2-hydroxy-1-naphthaldimine)

Ayhan Elmali

Department of Physics Engineering, Faculty of Sciences, University of Ankara, 06100 Besevler, Ankara, Turkey

Süheyla Özbey and Engin Kendi

Department of Physics Engineering, Hacettepe University, 06532 Beytepe, Ankara, Turkey

Mehmet Kabak and Yalcin Elerman

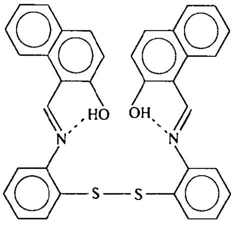
Department of Physics Engineering, Faculty of Sciences, University of Ankara, 06100 Besevler, Ankara, Turkey

Abstract

The crystal structure of the title compound, 1,1′-[2,2′-dithiobis(phenyliminomethyl)]di-2-naphthol, $C_{34}H_{24}N_2O_2S_2$, has been determined. Two 2-hydroxy-2-mercaptophenyl-1-naphthaldimine moieties, which can act as tridentate ligands, are joined by the S atoms. The molecule is not planar and contains intramolecular O···N hydrogen bonds.

Comment

Schiff bases have been used extensively as ligands in the field of coordination chemistry. Some of the reasons are that the intramolecular hydrogen bond between the O and N atoms plays an important role in the formation of metal complexes, and that Schiff base compounds show photochromism and thermochromism in the solid state by proton transfer from the hydroxyl O atom to the imine N atom. Proton transfer may also be a basis for the development of molecular switches. The charge transport occurs through overlapping intermolecular π orbitals with proton transfer. Both mononuclear and binuclear complexes of Mn^{II} with the Schiff base disulfide ligand *N*,*N*′-[dithiobis(*o*-phenylene)]bis(salicylideneaminato) have been synthesized and characterized by electrochemical, magnetic, spectral and diffraction methods (Kessissoglou, Butler & Pecoraro, 1987). This paper reports the structure of the Schiff base disulfide ligand (I).



(I)

Two 2-hydroxy-2-mercaptophenyl-1-naphthaldimine moieties, *A* and *B* (Fig. 1), which can act as tridentate ligands, are bonded by the S atoms, forming a disulfide bridge. The S(1)—S(2) distance is 2.041 (2) Å; this is in good agreement with disulfide bonds reported for analogous structures (Bonds & Ibers, 1972; Riley & Seff, 1972; Kessissoglou, Butler & Pecoraro, 1987; Sellmann, Prechtel, Knoch & Moll, 1993). The most interesting feature of this structure is the significant twist of *A* relative to *B*. The orientation of *A* with respect to *B* is defined by the torsion angle between them [85.0 (2)°]. The planarity of *A* and *B* differs; in *A* the angle between the naphthalene and benzene moieties is 3.63°, whereas in *B* it is 28.16°.

Intramolecular hydrogen bonds occur between O(1) and N(1) [2.603 (7) Å] and O(2) and N(2) [2.512 (7) Å], the H atoms essentially being bonded to the O atoms. The O(1)—C(1) and O(2)—C(34) bond lengths [1.352 (7) and 1.373 (7) Å, respectively] are in the range observed for similar structures (Inabe, Hoshino, Mitani

& Maruyama, 1989; Bregman, Leiserowitz & Schmidt, 1964). The N(1)—C(11) and N(2)—C(23) bond lengths are typical of C=N double bonds.

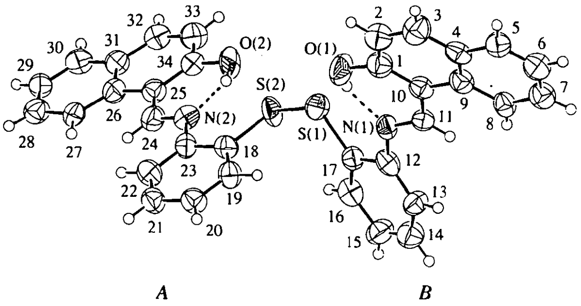


Fig. 1. The molecular structure of the title compound. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

The title compound was synthesized from acetonitrile by well-established methods (Vogel, 1966). The crystal density D_m was measured by pycnometric methods.

Crystal data

$C_{34}H_{24}N_2O_2S_2$
$M_r = 556.67$
Triclinic
$P\bar{1}$
$a = 8.656\ (1)$ Å
$b = 11.810\ (1)$ Å
$c = 13.521\ (1)$ Å
$\alpha = 78.93\ (2)°$
$\beta = 82.28\ (1)°$
$\gamma = 86.13\ (2)°$
$V = 1343.0\ (2)$ Å^3
$Z = 2$
$D_x = 1.377$ Mg m^{-3}
$D_m = 1.38$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 2.55–12.80°$
$\mu = 0.234$ mm^{-1}
$T = 293\ (2)$ K
Needle
0.15 × 0.10 × 0.05 mm
Black

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (Kopfmann & Huber, 1968)
$T_{\min} = 0.849$, $T_{\max} = 0.987$
3267 measured reflections
2679 independent reflections

2291 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.036$
$\theta_{\max} = 20.56°$
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: 1.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0459$
$wR(F^2) = 0.1141$
$S = 3.051$
2679 reflections
361 parameters
H atoms located geometrically with fixed $U_{\rm iso}$
$w = 1/\sigma^2(F_o^2)$

$(\Delta/\sigma)_{\max} = -0.099$
$\Delta\rho_{\max} = 0.350$ e Å^{-3}
$\Delta\rho_{\min} = -0.346$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
S(1)	0.3430 (2)	0.3660 (2)	0.6314 (1)	0.0610 (6)
S(2)	0.2982 (2)	0.2746 (1)	0.5256 (1)	0.0611 (6)
O(1)	0.2506 (5)	0.4345 (4)	0.8802 (4)	0.075 (2)
O(2)	0.1009 (5)	0.2637 (4)	0.3310 (3)	0.072 (2)
N(1)	0.4985 (6)	0.4795 (4)	0.7497 (4)	0.047 (1)
N(2)	0.1967 (6)	0.0902 (4)	0.4493 (4)	0.047 (1)
C(1)	0.2891 (8)	0.5189 (6)	0.9260 (5)	0.059 (2)
C(2)	0.1975 (8)	0.5387 (6)	1.0163 (5)	0.062 (2)
C(3)	0.2303 (8)	0.6240 (7)	1.0658 (5)	0.071 (2)
C(4)	0.3626 (8)	0.6921 (5)	1.0276 (5)	0.051 (2)
C(5)	0.3933 (8)	0.7825 (6)	1.0787 (5)	0.055 (2)
C(6)	0.5190 (8)	0.8457 (6)	1.0406 (5)	0.066 (2)
C(7)	0.6196 (8)	0.8317 (6)	0.9532 (5)	0.064 (2)
C(8)	0.5939 (7)	0.7460 (5)	0.9008 (5)	0.050 (2)
C(9)	0.4586 (7)	0.6757 (5)	0.9366 (5)	0.053 (2)
C(10)	0.4226 (7)	0.5857 (5)	0.8831 (4)	0.047 (2)
C(11)	0.5286 (7)	0.5608 (5)	0.7955 (4)	0.041 (2)
C(12)	0.6130 (8)	0.4480 (5)	0.6722 (5)	0.051 (2)
C(13)	0.7750 (7)	0.4617 (6)	0.6596 (5)	0.056 (2)
C(14)	0.8668 (8)	0.4207 (6)	0.5862 (6)	0.066 (2)
C(15)	0.8161 (8)	0.3659 (6)	0.5179 (5)	0.067 (2)
C(16)	0.6560 (8)	0.3479 (5)	0.5297 (5)	0.056 (2)
C(17)	0.5519 (7)	0.3843 (5)	0.6067 (5)	0.049 (2)
C(18)	0.3357 (7)	0.1268 (5)	0.5812 (4)	0.047 (2)
C(19)	0.4187 (7)	0.0923 (6)	0.6638 (5)	0.053 (2)
C(20)	0.4425 (7)	−0.0208 (6)	0.7016 (5)	0.055 (2)
C(21)	0.3853 (8)	−0.1035 (6)	0.6588 (5)	0.056 (2)
C(22)	0.3025 (7)	−0.0705 (6)	0.5774 (5)	0.055 (2)
C(23)	0.2760 (7)	0.0457 (5)	0.5327 (5)	0.049 (2)
C(24)	0.1545 (7)	0.0229 (6)	0.3935 (5)	0.057 (2)
C(25)	0.0781 (7)	0.0682 (6)	0.3060 (5)	0.050 (2)
C(26)	0.0229 (7)	−0.0126 (6)	0.2532 (5)	0.054 (2)
C(27)	0.0344 (7)	−0.1310 (6)	0.2777 (5)	0.050 (2)
C(28)	−0.0265 (7)	−0.2023 (6)	0.2249 (5)	0.057 (2)
C(29)	−0.0934 (7)	−0.1534 (5)	0.1404 (5)	0.049 (2)
C(30)	−0.1076 (7)	−0.0378 (6)	0.1134 (5)	0.056 (2)
C(31)	−0.0528 (7)	0.0374 (6)	0.1681 (5)	0.052 (2)
C(32)	−0.0713 (7)	0.1585 (6)	0.1385 (5)	0.062 (2)
C(33)	−0.0244 (8)	0.2320 (6)	0.1919 (5)	0.067 (2)
C(34)	0.0535 (7)	0.1870 (6)	0.2778 (5)	0.051 (2)

Table 2. *Selected geometric parameters* (Å, °)

S(1)—S(2)	2.041 (2)	C(13)—C(14)	1.331 (9)
S(1)—C(17)	1.814 (6)	C(14)—C(15)	1.356 (9)
S(2)—C(18)	1.790 (6)	C(15)—C(16)	1.400 (8)
O(1)—C(1)	1.352 (7)	C(16)—C(17)	1.397 (8)
O(2)—C(34)	1.373 (7)	C(18)—C(19)	1.390 (8)
N(1)—C(11)	1.295 (7)	C(18)—C(23)	1.419 (8)
N(1)—C(12)	1.428 (7)	C(19)—C(20)	1.347 (8)
N(2)—C(24)	1.296 (7)	C(20)—C(21)	1.377 (8)
N(2)—C(23)	1.396 (7)	C(21)—C(22)	1.373 (8)
C(1)—C(10)	1.436 (8)	C(22)—C(23)	1.406 (8)
C(1)—C(2)	1.413 (9)	C(24)—C(25)	1.430 (8)
C(2)—C(3)	1.375 (9)	C(25)—C(34)	1.391 (8)
C(3)—C(4)	1.426 (9)	C(25)—C(26)	1.436 (8)
C(4)—C(9)	1.426 (8)	C(26)—C(27)	1.375 (8)
C(4)—C(5)	1.435 (8)	C(26)—C(31)	1.411 (8)
C(5)—C(6)	1.348 (8)	C(27)—C(28)	1.374 (8)

C(6)—C(7)	1.401 (9)	C(28)—C(29)	1.362 (8)
C(7)—C(8)	1.387 (8)	C(29)—C(30)	1.345 (8)
C(8)—C(9)	1.457 (8)	C(30)—C(31)	1.402 (8)
C(9)—C(10)	1.466 (8)	C(31)—C(32)	1.413 (9)
C(10)—C(11)	1.462 (8)	C(32)—C(33)	1.343 (9)
C(12)—C(13)	1.406 (8)	C(33)—C(34)	1.419 (8)
C(12)—C(17)	1.436 (8)		
C(17)—S(1)—S(2)	104.3 (2)	C(16)—C(17)—S(1)	127.3 (5)
C(18)—S(2)—S(1)	104.7 (2)	C(12)—C(17)—S(1)	115.0 (5)
C(11)—N(1)—C(12)	118.5 (5)	C(19)—C(18)—S(2)	123.7 (5)
C(24)—N(2)—C(23)	120.8 (6)	C(23)—C(18)—S(2)	114.4 (5)
O(1)—C(1)—C(10)	119.6 (6)	N(2)—C(23)—C(22)	128.4 (6)
O(1)—C(1)—C(2)	119.1 (6)	N(2)—C(23)—C(18)	116.9 (6)
N(1)—C(11)—C(10)	119.8 (5)	N(2)—C(24)—C(25)	121.2 (6)
C(13)—C(12)—N(1)	128.2 (6)	O(2)—C(34)—C(25)	122.3 (6)
N(1)—C(12)—C(17)	113.5 (6)	O(2)—C(34)—C(33)	118.1 (6)

The structure was solved by direct phase determination. The parameters of the complete structure were refined by full-matrix least squares. All phenyl rings were refined without constraint. Values of distances and angles in the rings show no significant differences from those of an ideal benzene ring. All H-atom positions were calculated using a riding model and were included with fixed isotropic $U_{\rm iso}$ in all refinements.

Data collection: *Enraf–Nonius SDP-Plus Structure Determination Package* (Frenz, 1985). Cell refinement: *Enraf–Nonius SDP-Plus Structure Determination Package.* Data reduction: *Enraf–Nonius SDP-Plus Structure Determination Package.* Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1992). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1033). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bonds, W. D. & Ibers, J. A. (1972). *J. Am. Chem. Soc.* **94**, 3413–3419.

Bregman, J., Leiserowitz, L. & Schmidt, G. M. J. (1964). *J. Chem. Soc.* pp. 2068–2085.

Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package.* Version 3.0. Enraf–Nonius, Delft, The Netherlands.

Inabe, T., Hoshino, N., Mitani, T. & Maruyama, Y. (1989). *Bull. Chem. Soc. Jpn,* **62**, 2245–2251.

Kessissoglou, D. P., Butler, W. M. & Pecoraro, V. L. (1987). *Inorg. Chem.* **26**, 495–503.

Kopfmann, G. & Huber, R. (1968). *Acta Cryst.* A**24**, 348–351.

Riley, P. E. & Seff, K. (1972). *Inorg. Chem.* **11**, 2999.

Sellmann, G., Prechtel, W., Knoch, F. & Moll, M. (1993). *Inorg. Chem.* **32**, 538–546.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.

Spek, A. L. (1992). *PLUTON92. Molecular Graphics Program.* Univ. of Utrecht, The Netherlands.

Vogel, A. I. (1966). *Practical Organic Chemistry,* p. 653. London: Longmans.

Acta Cryst. (1995). **C51**, 1880–1883

7-Hydroxyflavanone (I) and 7-Ethoxycarbonylmethoxyflavanone (II)

ENGİN KENDİ AND SÜHEYLA ÖZBEY

Department of Physics Engineering, Hacettepe University, 06532 Beytepe, Ankara, Turkey

RAHMİYE ERTAN

Department of Pharmaceutical Chemistry, Faculty of Pharmacy, University of Ankara, 06100 Tandoğan, Ankara, Turkey

HOONG-KUN FUN AND BOON-CHUAN YIP

School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

(*Received* 19 *July* 1994; *accepted* 28 *February* 1995)

Abstract

The γ-pyrone rings in both of the title molecules, 2,3-dihydro-7-hydroxy-2-phenyl-4*H*-1-benzopyran-4-one, $C_{15}H_{12}O_3$, (I), and ethyl (2,3-dihydro-2-phenyl-4-oxo-4*H*-1-benzopyran-7-yl)oxyacetate, $C_{19}H_{18}O_5$, (II), adopt a C(2)-sofa conformation. In (I), the phenyl ring is twisted 103.0 (1)° from the plane of the fused rings and the hydroxy group at C(7) forms an intermolecular hydrogen bond to O(4) of the adjacent molecule. In (II), the phenyl ring is twisted 121.5 (2)° from the plane of the fused rings and the acetyl group is oriented slightly out of the plane of the benzene ring.

Comment

As a result of pharmacological interest in the flavanoids, a number of crystal structures have been resolved. The most studied are the flavones. In contrast, only a few single-crystal X-ray studies of flavanones have been reported so far (Cantrell, Stalzer & Becker, 1974; Mariezcurrena, 1978; Tomlin & Cantrell, 1990). The spasmolytic activity of a series of flavanone ether derivatives, including (I) and (II), was investigated using efloxate (Ertan, 1981*a*) as the reference compound. According to the results obtained, most of the compounds have an antagonistic activity comparable with that of the reference compound against the agonistic acetylcholine, histamine and $BaCl_2$ contractions, but no advantage over efloxate was found. Only compound (I) showed more potent activity than efloxate against histamine contractions (Ertan, 1981*b*). The determination of the structures of (I) and (II) was undertaken in order to study the conformations of the molecules and to establish any structure–activity relationship.

Acta Crystallographica Section C
ISSN 0108-2701 ©1995