graphics: *CRYSTAN-GM* for (I); *CRYSTAN* for (II). Software used to prepare material for publication: *CRYSTAN-GM* for (I); *CRYSTAN* for (II).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1034). Services for accessing these data are described at the back of the journal.

References

Edwards, C., Gilmore, C. J., Mackay, S. & Stewart, N. (1995). *CRYSTAN6.3. A Computer Program for the Solution and Refinement of Crystal Structures.* MacScience, Japan.
Gamlin, J. N., Jones, R., Leibovitch, M., Patrick, B., Scheffer, J. R. & Trotter, J. (1996). *Acc. Chem. Res.* **29**, 203–209.
Ito, Y. (1998). In preparation.
Ito, Y., Borecka, B., Scheffer, J. R. & Trotter, J. (1995). *Tetrahedron Lett.* **36**, 6083–6086.
Ito, Y. & Olovsson, G. (1997). *J. Chem. Soc. Perkin Trans.* pp. 127–133.
Ramamurthy, V. & Venkatesan, K. (1987). *Chem. Rev.* **87**, 433–481.
Rigaku Corporation (1993). *AFC/MSC Diffractometer Control System.* Rigaku Corporation, Tokyo, Japan.
Schmidt, G. M. J. (1964). *J. Chem. Soc.* pp. 2014–2021.
Toda, F. (1995). *Acc. Chem. Res.* **28**, 480–486.

Acta Cryst. (1998). C**54**, 145–146

(±)-7,7,9,9-Tetramethyl-2,3:4,5-dinaphtho-1,6,8-trioxa-7,9-disilacyclonona-2,4-diene

Qiancai Liu,[†] Mengxian Ding, Yonghua Lin and Yan Xing

Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, 159 Renmin Street, Changchun 130022, People's Republic of China. E-mail: yangliu@public.cc.jl.cn

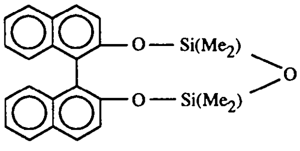
(*Received 21 January 1997; accepted 17 July 1997*)

Abstract

The title compound, $C_{24}H_{24}O_3Si_2$, is a twofold symmetric silicocrown ether with the two dimethylsilyl groups attached to the O atoms of 1,1′-bi-2-naphthol, and bridged by another O atom.

Comment

Optically active 1,1′-bi-2-naphthol and its derivatives are excellent ligands of chiral catalysts and have a C_2 symmetry axis (Narasaka, 1991; Stock & Kellogg, 1996). Their high stereoselectivity stems from their flexible chiral axis which can adapt to the coordination requirements of various substrates and multidentate ligands. Complexes of these molecules with Lewis acids are used as asymmetric catalysts for organic reactions such as Diels–Alder addition (Markó *et al.*, 1996), Michael addition and aldol reactions (Shibasaki & Sasai, 1996). The title compound, (I), is one such molecule (Stock, 1994).



(I)

In this molecule, each Si atom is bonded to two O atoms and two methyl groups with a distorted tetrahedral configuration. The angles around Si1 and Si2 are in the ranges 105.8 (2)–111.7 (2) and 107.2 (2)–112.5 (2)°, respectively. The bond distances Si1—O1 [1.657 (2) Å] and Si2—O3 [1.647 (2) Å] are longer than Si1—O2 [1.626 (2) Å] and Si2—O2 [1.625 (2) Å]. The dihedral angle between the two naphthalene rings is 72.96 (6)°, and that between the least-squares planes through O1, Si1, O2, Si2 and O3 (with a maximum deviation of 0.206 Å), and the two naphthalene rings (C1–C10 and C11–C20) are 74.73 (8) and 78.75 (7)°, respectively.

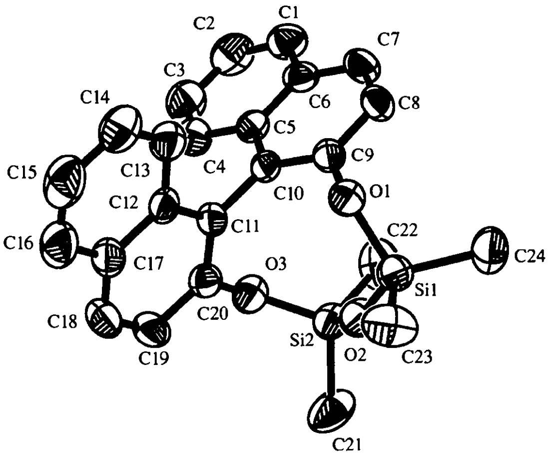


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Experimental

The title compound was synthesized by treatment of racemic 1,1′-bi-2-naphthol with excess dichlorodimethylsilane in the presence of triethylamine, and then subsequent reaction with sodium cyclopentadienide in tetrahydrofuran at room temperature. Compound (I) was obtained as yellow prismatic crystals by slow evaporation from a petroleum ether and tetrahydrofuran mixed solution.

[†] Present address: Institute of Applied Chemistry, East China University of Science and Technology, 130 Meilong Road, Shanghai 200237, People's Republic of China.

ISSN 0108-2701 © 1998

Crystal data

$C_{24}H_{24}O_3Si_2$
$M_r = 416.61$
Monoclinic
$P2_1/n$
$a = 10.694\,(1)$ Å
$b = 13.481\,(1)$ Å
$c = 15.307\,(2)$ Å
$\beta = 94.32\,(1)^\circ$
$V = 2200.5\,(4)$ Å^3
$Z = 4$
$D_x = 1.258$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 30 reflections
$\theta = 4.83$–9.96°
$\mu = 0.183$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.42 × 0.38 × 0.32 mm
Yellow

Data collection

Siemens *P*4 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.243$, $T_{\max} = 0.266$
4615 measured reflections
3451 independent reflections

2170 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\max} = 24^\circ$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 17$
3 standard reflections
frequency: 100 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.164$
$S = 0.489$
3434 reflections
306 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.2186P)^2 + 2.3095P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.179$
$\Delta\rho_{\max} = 0.182$ e Å^{-3}
$\Delta\rho_{\min} = -0.205$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Si1—O2	1.626 (2)	O1—C9	1.381 (3)
Si2—O2	1.625 (2)	O3—C20	1.376 (4)
Si2—O3	1.647 (2)		
O2—Si1—O1	108.91 (11)	Si2—O2—Si1	151.2 (2)
O2—Si2—O3	108.95 (12)	C20—O3—Si2	126.9 (2)
C9—O1—Si1	121.4 (2)		

All H atoms were fixed at calculated positions with common isotropic displacement parameters ($U_{\rm iso} = 0.08$ Å^2).

Data collection: *DIF*4 (Stoe & Cie, 1991*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1991*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *XL* in *SHELXTL-Plus*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1062). Services for accessing these data are described at the back of the journal.

References

Markó, I. E., Evans, G. R., Seres, S., Chellé, I. & Janousek, Z. (1996). *Pure Appl. Chem.* **68**, 113–122.
Narasaka, K. (1991). *Synthesis*, pp. 1–11.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Shibasaki, M. & Sasai, H. (1996). *Pure Appl. Chem.* **68**, 523–530.
Stock, H. T. (1994). PhD dissertation, University of Groningen, The Netherlands. Personal communication with Professor R. M. Kellogg.
Stock, H. T. & Kellogg, R. M. (1996). *J. Org. Chem.* **61**, 3093–3105.
Stoe & Cie (1991*a*). *DIF*4. *Diffractometer Control Program*. Version 7.08. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1991*b*). *REDU*4. *Data Reduction Program*. Version 7.08. Stoe & Cie, Darmstadt, Germany.

Acta Cryst. (1998). C**54**, 146–148

2-(5-Chloro-2-nitrophenylthio)nicotinoyl Chloride

J. Gabriel Garcia,[a]† Simon H. Haydar,[a] A. Paul Krapcho,[a] Yen-Hsiang Liu[b] and Frank R. Fronczek[b]

[a]*Department of Chemistry, The University of Vermont, Burlington, VT 05405, USA, and* [b]*Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA. E-mail: garcia@oberon.cmc.uab.edu*

(*Received 28 April 1997; accepted 1 October 1997*)

Abstract

The two aromatic rings in the title compound, $C_{12}H_6Cl_2N_2O_3S$, form a dihedral angle of $56.7\,(1)^\circ$. The alkanoyl chloride group lies slightly out of the plane of the aromatic ring with the carbonyl O and alkanoyl Cl atoms lying 0.219 (2) and −0.374 (1) Å, respectively, from the ring. The nitro group deviates from coplanarity with its aromatic ring, displaying a torsion angle along N—C of $34.9\,(3)^\circ$. The C—S—C angle is $102.85\,(9)^\circ$. Bond distances C═O and S—C have values of 1.178 (3) Å, and 1.766 (2) and 1.767 (2) Å, respectively.

Comment

The title compound, (I), was prepared as part of a structural study involving heterocyclic fused-ring systems and their key synthetic precursors (Garcia, Haydar & Krapcho, 1997).

The title compound contains two substituted aromatic rings linked by an S atom which form a di-

---

† New address: Centre for Macromolecular Crystallography, The University of Alabama at Birmingham, Birmingham, AL 35294-0005, USA.

Acta Crystallographica Section C
ISSN 0108-2701 © 1998