Cromer, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.2. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academics Publishers, Dordrecht.)
Cruickshank, D. W. T. (1949). *Acta Cryst.* **2**, 154–157.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
Ibers, J. A. & Hamilton, W. C. (1964). *Acta Cryst.* **17**, 781–782.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Johnson, J. F. & Scheidt, W. R. (1978). *Inorg. Chem.* **17**, 1280–1287.
Kee, T. P. & Gibson, V. C. (1992). *Polyhedron*, **11**, 587–589.
Killean, R. C. G. & Lawrence, J. L. (1969). *Acta Cryst.* B**25**, 1750–1752.
Nugent, W. A. & Mayer, J. M. (1987). *Metal–Ligand Multiple Bonds*, Ch. 5, Table 5.2. New York: Wiley.
Steffey, B. D., Fanwick, P. E. & Rothwell, I. P. (1990). *Polyhedron*, **9**, 963–968.
Visciglio, V. M. & Rothwell, I. P. (1993). In preparation.
Yu, J. S., Fanwick, P. E. & Rothwell, I. P. (1990). *J. Am. Chem. Soc.* **112**, 8171–8712.
Yu, J. S., Fanwick, P. E. & Rothwell, I. P. (1992). *Acta Cryst.* C**48**, 1759–1761.

Acta Cryst. (1994). C**50**, 902–904

$[Nb(CH_2SiMe_3)(OC_6H_3Ph_2\text{-}2,6)(\mu\text{-}CSiMe_3)]_2$

EDNA TORREZ, ROBERT D. PROFILET, PHILLIP E. FANWICK AND IAN P. ROTHWELL*

Department of Chemistry, 1393 Brown Building, Purdue University, West Lafayette, IN 47907-1393, USA

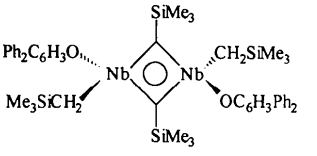
(*Received* 24 *February* 1993; *accepted* 22 *October* 1993)

Abstract

The compound bis(2,6-diphenylphenoxy)bis(trimethylsilylmethane)bis(μ-trimethylsilylmethylidyne)diniobium has a crystallographic center of inversion with tetrahedral geometry adopted around each Nb metal center. Nb—Nb = 2.9082 (8), Nb—O = 1.909 (3), Nb—C(terminal alkyl) = 2.146 (5), Nb—C(bridging) = 1.979 (4) Å (average).

Comment

Recently, it has been shown that the Group 5 metal–alkylidyne bridged compounds $[(Me_3SiCH_2)_2M(\mu\text{-}CSiMe_3)]_2$ (M = Nb, Ta) (Mowat & Wilkinson, 1973) supported on silica will catalyze the exhaustive hydrogenation of a variety of aromatic substances (Profilet, Rothwell & Rothwell, 1993). Previously, our group had also shown that treatment of the tantalum compound with phenolic reagents led to a range of substitution products in which the central alkylidyne core was maintained (Fanwick, Ogilvy & Rothwell, 1987). We report here the isolation and structural characterization of a related dinuclear compound of niobium, $[Nb(CH_2SiMe_3)(OC_6H_3Ph_2\text{-}2,6)(\mu\text{-}CSiMe_3)]_2$, (I).



(I)

A view of the molecule is shown in Fig. 1. Earlier work has shown that all three of the possible substitutional isomers are formed when $[Me_3SiCH_2)_2Ta(\mu\text{-}CSiMe_3)]_2$ is reacted with two equivalents of 2,6-diphenylphenol (Fanwick, Ogilvy & Rothwell, 1987). The solid-state structure of the title niobium compound (Fig. 1) shows it to contain the *anti*-1,2-substituted isomer with a crystallographically imposed center of inversion. The Nb—Nb distance of 2.9082 (8) Å is only slightly longer than the distance of 2.897 (2) Å in $[(Me_3SiCH_2)_2Nb(\mu\text{-}CSiMe_3)]_2$. The Nb—O distance of 1.909 (3) Å and large Nb—O—C angle of 167.4 (3)° are typical for aryloxide ligands bound to niobium(V) metal centers (Steffey, Fanwick & Rothwell, 1990).

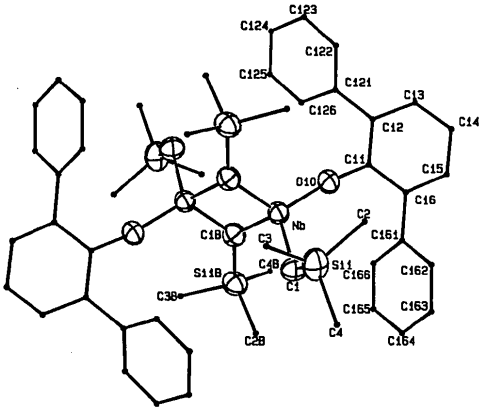


Fig. 1. A view of the molecule emphasizing the central coordination sphere. Displacement ellipsoids are drawn at the 50% probability level and H atoms are omitted for clarity.

Experimental

The title compound was prepared, in moderate yield, by addition of two equivalents of 2,6-diphenylphenol to a toluene solution of $[Nb(CH_2SiMe_3)_2(\mu\text{-}CSiMe_3)]_2$ and heating at 373 K

©1994 International Union of Crystallography
Printed in Great Britain – all rights reserved

for 36 h. Concentration and subsequent cooling of the red solution to 263 K led to the formation of large red plates of the pure product.

Crystal data

$[Nb_2(C_{18}H_{13}O)_2(C_4H_9Si)_2$-$(C_4H_{11}Si)_2]$	D_x = 1.288 Mg m^{-3}
M_r = 1021.26	Mo $K\alpha$ radiation
Monoclinic	λ = 0.71073 Å
$P2_1/c$	Cell parameters from 25 reflections
a = 11.785 (4) Å	θ = 17–21°
b = 19.408 (2) Å	μ = 0.54 mm^{-1}
c = 12.476 (6) Å	T = 293 K
β = 112.70 (2)°	Plate
V = 2632 (3) Å^3	0.60 × 0.52 × 0.06 mm
Z = 2	Red

Data collection

Enraf-Nonius CAD-4 diffractometer	2382 observed reflections [$F > 3\sigma(F)$]
ω–2θ scans	θ_{max} = 22.5°
Absorption correction: empirical	$h = -12 \rightarrow 11$
T_{min} = 0.49, T_{max} = 1.00	$k = 0 \rightarrow 20$
3603 measured reflections	$l = 0 \rightarrow 13$
3603 independent reflections	3 standard reflections
	frequency: 83.33 min
	intensity variation: <3%

Refinement

Refinement on F	$w = 1/[\sigma^2 F + (0.02F)^2 + 1 - F]$
R = 0.035	$(\Delta/\sigma)_{max}$ = 0.01
wR = 0.041	$\Delta\rho_{max}$ = 0.38 e Å^{-3}
S = 1.225	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
2382 reflections	Atomic scattering factors from Cromer & Waber (1974)
271 parameters	
H-atom parameters not refined	

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

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Nb	0.08398 (3)	0.03525 (2)	−0.04165 (4)	3.293 (8)
Si(1)	0.1839 (1)	−0.04916 (8)	−0.2414 (1)	5.16 (4)
Si(1*B*)	−0.2188 (1)	0.09808 (8)	−0.1545 (1)	4.43 (4)
O(10)	0.2064 (3)	0.1040 (2)	0.0268 (3)	4.29 (8)
C(1)	0.0690 (4)	0.0040 (3)	−0.2116 (4)	4.3 (1)
C(2)	0.3412 (6)	−0.0143 (4)	−0.1639 (7)	8.5 (2)
C(3)	0.1748 (6)	−0.1389 (3)	−0.1970 (6)	7.7 (2)
C(4)	0.1497 (7)	−0.0493 (5)	−0.4006 (6)	9.4 (2)
C(11)	0.3009 (4)	0.1497 (2)	0.0559 (4)	3.8 (1)
C(12)	0.3792 (4)	0.1576 (2)	0.1728 (4)	4.2 (1)
C(13)	0.4810 (5)	0.2001 (3)	0.1973 (5)	5.5 (2)
C(14)	0.5034 (5)	0.2345 (3)	0.1119 (6)	6.4 (2)
C(15)	0.4216 (4)	0.2291 (3)	−0.0025 (5)	5.4 (1)
C(16)	0.3196 (4)	0.1874 (2)	−0.0323 (4)	4.1 (1)
C(1*B*)	−0.0872 (4)	0.0475 (2)	−0.0503 (4)	3.8 (1)
C(2*B*)	−0.2290 (5)	0.0888 (3)	−0.3063 (5)	6.1 (2)
C(3*B*)	−0.3679 (5)	0.0680 (4)	−0.1518 (7)	7.8 (2)
C(4*B*)	−0.1942 (6)	0.1919 (3)	−0.1126 (5)	6.3 (2)
C(121)	0.3550 (4)	0.1267 (2)	0.2699 (4)	4.2 (1)
C(122)	0.4515 (5)	0.0994 (3)	0.3665 (5)	5.3 (1)
C(123)	0.4331 (6)	0.0778 (3)	0.4632 (5)	6.6 (2)
C(124)	0.3190 (6)	0.0833 (3)	0.4675 (5)	6.7 (2)
C(125)	0.2212 (5)	0.1088 (3)	0.3725 (5)	5.8 (2)
C(126)	0.2400 (5)	0.1306 (3)	0.2757 (4)	4.7 (1)
C(161)	0.2311 (4)	0.1863 (2)	−0.1541 (4)	4.3 (1)
C(162)	0.2739 (5)	0.1798 (3)	−0.2429 (5)	5.6 (2)
C(163)	0.1934 (6)	0.1855 (3)	−0.3580 (5)	7.0 (2)
C(164)	0.0706 (7)	0.1981 (3)	−0.3853 (5)	7.3 (2)
C(165)	0.0265 (5)	0.2050 (3)	−0.2984 (6)	6.4 (2)
C(166)	0.1071 (5)	0.1991 (3)	−0.1829 (5)	5.2 (1)

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

Nb—Nb	2.9082 (8)	Nb—C(1*B*)	1.993 (4)
Nb—O(10)	1.909 (3)	Nb—C(1*B*)	1.965 (4)
Nb—C(1)	2.146 (5)	O(10)—C(11)	1.359 (5)
C(1)—Nb—C(1*B*)	106.5 (2)	O(10)—Nb—C(1*B*)	118.0 (2)
C(1)—Nb—C(1*B*)	108.7 (2)	Nb—O(10)—C(11)	167.4 (3)
C(1*B*)—Nb—C(1*B*)	85.4 (2)	Nb—C(1*B*)—Nb	94.6 (2)
O(10)—Nb—C(1)	113.6 (2)	Nb—C(1*B*)—Si(1*B*)	130.6 (3)
O(10)—Nb—C(1*B*)	121.0 (2)	Nb—C(1*B*)—Si(1*B*)	131.2 (2)

The structure was solved using the Patterson heavy-atom method which revealed the position of one Nb atom. The remaining atoms were located using *DIRDIF* (Beurskens *et al.*, 1984) and in succeeding difference Fourier syntheses. H atoms were located and included in the structure-factor calculations, but their positions were not refined. The structure was refined by full-matrix least squares where the function minimized was $\Sigma w(|F_o|-|F_c|)^2$ and the weight, w, was as defined by the Killean & Lawrence (1969) method with terms of 0.020 and 1.0. Anomalous-dispersion effects were included in F_c (Ibers & Hamilton, 1964); the values for f' and f'' were those of Cromer (1974). There were 19 correlation coefficients greater than 0.50. The highest correlation coefficient was 0.62 between parameters 70 and 72. The highest peak in the final difference Fourier map had an estimated error based on ΔF (Cruickshank, 1949) of 0.06. Plots of $\Sigma w(|F_o|-|F_c|)^2$ *versus* $|F_o|$, reflection order in data collection, $\sin\theta/\lambda$ and various classes of indices showed no unusual trends. All calculations were performed on a VAX computer using *SDP* (Frenz, 1978).

We thank the National Science Foundation (grant No. CHE-8915573) for support of this research.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71770 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1065]

References

Beurskens, P. T., Bosman, W. P., Doesburg, H. M., Gould, R. O., van den Hark, Th. E. M., Prick, P. A., Noordik, J. H., Beurskens, G., Parthasarathi, V., Bruins Slot, H. J., Haltiwanger, R. C., Strumpel, M. & Smits, J. M. M. (1984). Technical Report 1984/1. Crystallography Laboratory, Toernooiveld, 6525 ED Nijmegen, The Netherlands.

Cromer, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.2. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Cruickshank, D. W. T. (1949). *Acta Cryst.* **2**, 154–157.

Fanwick, P. E., Ogilvy, A. E. & Rothwell, I. P. (1987). *Organometallics*, **6**, 73–80.

Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – a Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution. Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft. Univ. Press.

Ibers, J. A. & Hamilton, W. C. (1964). *Acta Cryst.* **17**, 781–782.

Killean, R. C. G. & Lawrence, J. L. (1969). *Acta Cryst.* **B25**, 1750–1752.

Mowat, W. & Wilkinson, G. (1973). *J. Chem. Soc. Dalton Trans.* pp. 770–778.

Profilet, R. D., Rothwell, A. P. & Rothwell, I. P. (1993). *J. Chem. Soc. Chem. Commun.* pp. 42–44.

Steffey, B. D., Fanwick, P. E. & Rothwell, I. P. (1990). *Polyhedron*, **9**, 963–968.