observed upon warming to 100 °C ($CDCl_2CDCl_2$) and 52 °C (acetone-$d_6$), respectively. Since $\eta^1$ and $\eta^2$-$H_2C{=}X$ coordination cannot be rigorously distinguished by NMR, we determined the X-ray crystal structures of **1** and **2**.

X-ray data were collected for **1** and **2** at −158 and 25 °C, respectively, as described in the supplementary material. Crystals of **1** belonged to the monoclinic system, space group $P2_1$ ($Z = 4$), $a = 10.113$ (3) Å, $b = 18.928$ (7) Å, $c = 13.300$ (4) Å, $\beta = 105.03(2)°$. Refinement (supplementary material) yielded the structural data in Figure 1 and Table II. Final $R$ indices were $R = 0.043$ and $R_w = 0.052$, and the goodness of fit was 1.49. Crystals of **2** belonged to the monoclinic system, space group $P2_1/c$ ($Z = 4$), $a = 9.688$ (2) Å, $b = 18.536$ (4) Å, $c = 14.895$ (5) Å, $\beta = 103.53$ (2)°. Refinement yielded the structural data in Figure 1 and Table II. The final $R$ indices were $R = 0.060$ and $R_w = 0.081$, and the goodness of fit was 1.89. In **2**, $H_2C{=}X$ hydrogen atoms were located. Distances and angles (unrefined) are as follows: C1–H1, 1.14 Å; C1–H2, 0.81 Å; H1–C1–H2, 121°; S1–C1–H1, 120°; S1–C1–H2, 108°.

The C–O bond length in **1**, 1.374 (19) Å, is significantly longer than the C=O bond length in free formaldehyde (1.225 Å)[11a] but is slightly shorter than typical C–O single-bond distances (1.41–1.43 Å).[11b] It is close to those found by Berke for $Fe(CO)_2(P(OCH_3)_3)_2(\eta^2\text{-}H_2C{=}O)$ (1.32 (2) Å)[6c,d] and Floriani for $(\eta\text{-}C_5H_5)_2V(\eta^2\text{-}H_2C{=}O)$ (1.353 (10) Å)[6e] but is substantially shorter than that found by Roper for $Os(CO)_2(PPh_3)_2(\eta^2\text{-}H_2C{=}O)$ (1.584 (11) Å).[6a,b]

Complex **2** is the first mononuclear thioformaldehyde complex to be structurally characterized. The C–S bond length, 1.742 (9) Å, is intermediate between that found in $H_2C{=}S$ (1.6108 (9) Å)[12] and typical C–S single bonds (1.80–1.82 Å).[11c] It is significantly shorter than those determined by Adams for a series of triosmium $\mu_2$- and $\mu_3$-thioformaldehyde complexes (1.788 (11)–1.872 (12) Å).[7c–e]

The lengthened $H_2C{=}X$ bonds in **1** and **2** reflect the fact that the $(\eta\text{-}C_5H_5)Re(NO)(PPh_3)^+$ moiety is an extremely good donor. The rhenium HOMO has been shown by Eisenstein[13] to be a d orbital that is bisected by the Re–P bond and perpendicular to the Re–NO bond. Figure 1 shows that **1** and **2** adopt conformations that maximize overlap of the $H_2C{=}X$ $\pi^*$ orbitals with this HOMO.[14]

The chemistry of **1** and **2** is currently under intensive study. After 19 h at 51 °C in $CD_3CN$, **1** was converted to a ca. 50:50 mixture of **1** and the nitrile complex $[(\eta\text{-}C_5H_5)Re(NO)(PPh_3)(CD_3CN)]^+PF_6^-$.[8b] Under identical conditions, **2** showed no sign of reaction. In contrast to other mononuclear $\eta^2$-$H_2C{=}X$ complexes,[6,7b] we have not yet obtained well-defined products from reactions of **1** and **2** with electrophiles. However, both **1** and **2** are attacked by nucleophiles. Treatment of **2** with $NaBH_3CN/CH_3OH$ gave, after workup and $CHCl_3$/heptane recrystallization, the thiomethyl complex $(\eta\text{-}C_5H_5)Re(NO)(PPh_3)(SCH_3)$ (**8**)[9] as bright red crystals in 85% yield. Similar conditions converted **1** to a mixture of products. However, **1** and formyl $(\eta\text{-}C_5H_5)Re(NO)(PPh_3)(CHO)$[8a] rapidly reacted at −25 °C to give $[(\eta\text{-}C_5H_5)Re(NO)(PPh_3)(CO)]^+PF_6^-$ and methoxide $(\eta\text{-}C_5H_5)Re(NO)(PPh_3)(OCH_3)$ (**9**)[9] (99% and 74% yields vs. $Ph_3SiCH_3$ standard). Workup gave spectroscopically pure **9** in 52% yield; deep red crystals were obtained from benzene/hexane. Hydride transfer from a formyl to a formaldehyde ligand is, in our opinion, also a plausible route to catalyst-bound methoxides. Finally, reaction of **1** with $PPh_3$ gave a $\mu_2$-$H_2C{=}O$ adduct which we assign on the basis of NMR data[9] as the ReOCP regioisomer $[(\eta\text{-}C_5H_5)Re(NO)(PPh_3)(OCH_2PPh_3)]^+PF_6^-$ (**10**). This compound, which can be isolated in 89% yield as orange-red needles, equilibrates to a (84 ± 2):(16 ± 2) **10** $\rightleftharpoons$ **1** + $PPh_3$ mixture in acetone. Facile $\mu_1 \rightleftharpoons \mu_2$ $H_2C{=}O$ equilibria may also be important in catalytic CO reduction.

In summary, the methodology described in this communication should, in view of the increasing numbers of electrophilic alkylidene complexes that are available,[15] allow access to a series of new $H_2C{=}O$ and $H_2C{=}S$ (and possibly $RCH{=}O$ and $RCH{=}S$) complexes. These can be expected to have a rich chemistry which will bear upon important mechanistic issues in transition-metal catalysis.

**Acknowledgment.** We thank the Department of Energy for support of this research. The crystal structure determination of **1** and FT NMR measurements made use of equipment obtained via NSF departmental instrumentation grants. W.E.B. thanks the Regents of the University of California for a Fellowship.

**Registry No. 1**, 84369-16-4; **2**, 84369-18-6; **3**, 71763-23-0; **5**, 71763-25-2; **8**, 84369-19-7; **9**, 84369-20-0; **10**, 84369-22-2; $[(\eta\text{-}C_5H_5)Re(NO)(PPh_3)(CO)]^+PF_6^-$, 79919-50-9.

**Supplementary Material Available:** Spectral,[9] microanalytical,[9] and crystallographic (**1**, **2**)[13] data (85 pages). Ordering information is given on any current masthead page.

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(11) Streitwieser, A., Jr.; Heathcock, C. H. "Introduction to Organic Chemistry", 2nd ed.; Macmillan: New York, 1981; (a) p 357, (b) pp 229–230, (c) p 807.

(12) Johnson, D. R.; Powell, F. X.; Kirchhoff, W. H. *J. Mol. Spectrosc.* **1971**, *39*, 136.

(13) Kiel, W. A.; Lin, G.-Y.; Constable, A. G.; McCormick, F. B.; Strouse, C. E.; Eisenstein, O.; Gladysz, J. A. *J. Am. Chem. Soc.* **1982**, *104*, 4865.

(14) The Re—C=X planes make angles 15° (**1**) and 21° (**2**) with the Re–P vector.

(15) Some lead articles are as follows: (a) Brookhart, M.; Tucker, J. R.; Flood, T. C.; Jensen, J. *J. Am. Chem. Soc.* **1980**, *102*, 1203. (b) Kegley, S. E.; Brookhart, M.; Husk, G. R. *Organometallics* **1982**, *1*, 760. (c) Brookhart, M.; Tucker, J. R.; Husk, G. R. *J. Am. Chem. Soc.* **1983**, *105*, 258. (d) Casey, C. P.; Miles, W. H.; Tukada, H.; O'Connor, J. M. *Ibid.* **1982**, *104*, 3761. (e) Marsella, J. A.; Folting, K.; Huffman, J. C.; Caulton, K. G. *Ibid.* **1981**, *103*, 5596.

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## Total Synthesis of (±)-Verrucarol

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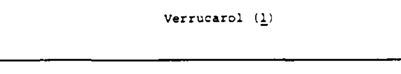
*Received November 5, 1982*

The epoxytrichothecenes are a group of secondary fungal metabolites that possess antibiotic, antifungal, antiviral, and/or cytotoxic properties.[2] The fungi responsible for producing these terpenoids (various *Trichothecium*, *Myrothecium*, and *Fusarium* species, among others) have been implicated in a number of diseases of humans, animals, and plants. Certain members of this group, most notably T-2 toxin, nivalenol, and anguidine, have gained considerable notoriety in recent months as a consequence of the "yellow rain" problem.[3] Our interest in these compounds stems from their activity as potent inhibitors of protein synthesis in eucaryotes. For example, the macrocyclic di- and triester derivatives of verrucarol (**1**) possess promising antitumor activity.[2a]



Verrucarol (1)

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(1) Roger and Georges Firmenich Assistant Professor of Natural Products Chemistry; Fellow of the Alfred P. Sloan Foundation, 1982–1984.

(2) (a) Doyle, T. W.; Bradner, W. T. In "Anticancer Agents Based on Natural Product Models"; Cassidy, J. M., Douros, J., Eds.; Academic Press: New York, 1980; Chapter 2. (b) Ueno, Y. *Adv. Nutr. Res.* **1980**, *3*, 301. (c) Tamm, Ch. *Fortschr. Chem. Org. Naturst.* **1974**, *31*, 63. (d) Bamburg, J. R.; Strong, F. M. In "Microbial Toxins"; Kadis, S., Ciegler, A., Ajl, S. J., Eds.; Academic Press: New York, 1971; Vol. 7, p 207.

(3) For a discussion of this problem, see: Embers, L. *Chem. Eng. News* **1981**, *59* (*48*), 29; **1982** *60* (26), 8. Marshall, E. *Science* (*Washington, D.C.*) **1982**, *217*, 31. Cullen, D.; Caldwell, R. W. *Ibid.* **1982**, *217*, 776. Meisner, H. *Ibid.* **1982**, *217*, 776. Mirocha, C. J. *Ibid.* **1982**, *217*, 776.

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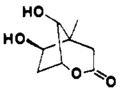
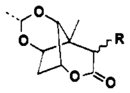
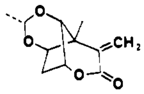
As part of our program on the synthesis of these macrolides[4] we have developed a total synthesis of **1**, a discussion of which is the subject of this communication.[5]

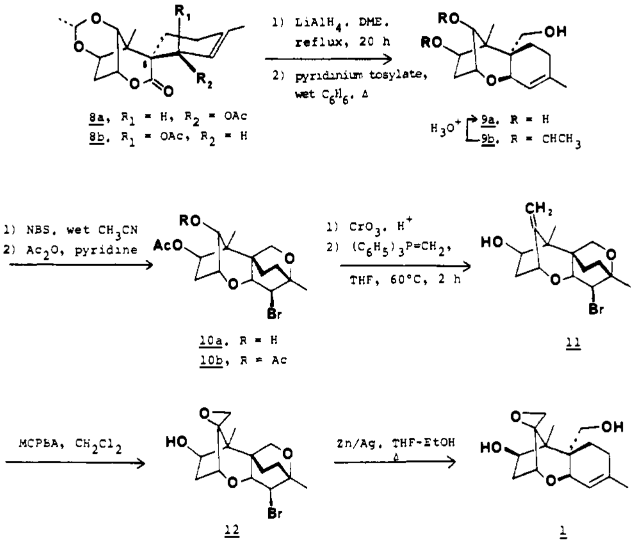

2


3, R = H
4, R = CHO (enolic)
5, R = COOCH$_3$
6, R = CH$_2$OH


7

We established in preliminary studies that a viable route to the epoxytrichothecene ring system involves annelation of the A ring onto a preformed BC bicyclic precursor.[6a] Lactone **2**, prepared by a ten-step sequence from readily available (methylcyclopentadienyl)trimethylsilane,[6b,7] was protected as the ethylidene derivative **3**,[8] mp 123–124.5 °C (10 equiv of CH$_3$CH(OMe)$_2$, C$_6$H$_6$, *p*-TsOH, reflux 12 h; a single diastereomer, 74–79% yield). Attempts to functionalize C-6 of this intermediate, however, proved to be more difficult than anticipated. After considerable experimentation formylation [(i) LDA, THF, −78 °C; (ii) HCO$_2$Et, −78 → 25 °C] and carboxylation [(i) LDA, THF, −78 °C; (ii) CO$_2$, −20 °C; (iii) CH$_2$N$_2$] reactions of **3** to give **4** (19–22%) and **5** (28–33%), respectively, were realized. The efficiencies of these transformations were, however, too poor for these intermediates to be of use in this synthesis.

Scheme I



In an alternative approach, the lithium enolate of **3** was treated with gaseous formaldehyde by using the procedure described by Grieco.[9] The expected α-hydroxymethyl lactones **6** (diastereomeric mixture) were obtained in low yield (ca. 20%) together with 7% of α-methylene lactone **7**.[8] Careful optimization of the reaction conditions [1.2 equiv of LDA, **3**, THF, −78 °C, 30 min; warm to −20 °C and add gaseous H$_2$CO (generated from 6 equiv of dry paraformaldehyde, 160 °C); warm to 23 °C and stir for 24 h] afforded **7**, mp 157.5–159 °C, in 69–71% yield. An analogous α-methylenation has been achieved by Schlessinger.[5a] Treatment of **7** with 3-methyl-1-acetoxybutadiene[10] (4.0 equiv, 140 °C, C$_6$H$_5$CH$_3$, BHT, 48 h) afforded a 3:1 mixture of two Diels–Alder adducts, **8a**[8] (mp 234–236 °C) and **8b**[8] (mp 260.5–262 °C), in 53–57% yield (25–30% of **7** was recovered).[11] Although the stereochemistry at C-11 of **8a,b** has not been established unambiguously, it is clear that both isomers possess identical configurations at C-6, since both adducts cyclize (Scheme I) to **9** in the next stage of the synthesis.[12] Both products, therefore, derive from cycloaddition reactions in which the diene approached the [3.2.1] bicyclic ring system from the least hindered face.[5a,6a] The mixture of cycloadducts **8a** and **8b**, without separation, was reduced with excess LiAlH$_4$ in DME (reflux, 20 h). The resulting triol mixture was treated with catalytic pyridinium *p*-toluenesulfonate in wet benzene (reflux 1 h) to afford a mixture of **9a**[8a] (major product, mp 40–44 °C) and the corresponding ethylidene acetal **9b**,[8a] the latter of which was hydrolyzed quantitatively (1 N HCl, acetone, 5 h, 23 °C) to **9a** in a separate step. The overall yield of **9a** from **8a,b** was 55–65%. This ring-closure sequence resembles the biomimetic pathway examined first by Kamikawa[13] and later applied in our preliminary studies.[6a]

Completion of the synthesis from **9a** proceeded as follows. The C-15 hydroxyl group and the 9,10 olefin were protected simultaneously by treatment of **9a** with NBS in wet CH$_3$CN. The resulting diol bromo ether was then acylated (1.5 equiv of Ac$_2$O, pyridine, 40 h) to give **10a**[8a] in 29–30% overall yield (from **9a**) together with 12–14% of diacetate **10b**[8a] and 25–30% of recovered diol.[14] Acetate **10a** was oxidized with Jones reagent (88%) and the 12,13 olefin was introduced by a Wittig reaction (6 equiv of (C$_6$H$_5$)$_3$P=CH$_2$, THF, 60 °C, 3 h, 52–60% yield) with concomitant cleavage of the acetate. Epoxidation of **11**[8a] (MCPBA, NaHCO$_3$, CH$_2$Cl$_2$, 21 h, 95%) afforded **12**,[8a] mp 227–229 °C, which was identical in all respects with an authentic sample prepared from natural **1** (NBS, wet CH$_3$CN). Finally, treatment of synthetic bromide **12** with zinc–silver couple in 5:1 THF-EtOH (reflux, 12 h)[5b] afforded racemic verrucarol [mp 170–171.5 °C (recrystallized from hexane–benzene), lit.[5a] mp for racemic **1**, 159–161 °C (Et$_2$O)] in 82% yield. The synthetic trichothecene thus obtained was identical in all respects with an authentic sample provided by Professor CH. Tamm.[15-17]

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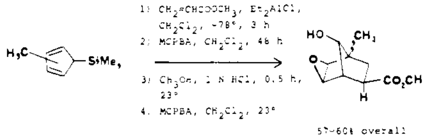
(4) For recent accomplishments in this area, see: (a) Still, W. C.; Ohmizu, H. *J. Org. Chem.* **1981**, *46*, 5242 (verrucarin A). (b) Esmond, R.; Fraser-Reid, B.; Jarvis, B. B. *Ibid.* **1982**, *47*, 3358 (verrucarin J and trichoverrin B). (c) Mohr, P.; Tori, M.; Grossen, P.; Herold, P.; Tamm, Ch. *Helv. Chim. Acta* **1982**, *65*, 1412 (verrucarin A). (d) Roush, W. R.; Blizzard, T. A. *J. Org. Chem.*, in press.

(5) Syntheses of a number of natural trichothecenes have been recorded. See ref 4 and the following: (a) Schlessinger, R. H.; Nugent, R. A. *J. Am. Chem. Soc.* **1982**, *104*, 1116 (verrucarol). (b) Kraus, G. A.; Roth, B.; Frazier, K.; Shimagaki, M. *Ibid.* **1982**, *104*, 1114 (calonectrin). (c) Still, W. C.; Tsai, M. Y. *Ibid.* **1980**, *102*, 3654 (trichodermol). (d) Colvin, E. W.; Malchenko, S.; Raphael, R. A.; Roberts, J. S. *J. Chem. Soc., Perkin Trans. 1* **1973**, 1989 (trichodermin). (e) Citations of other pertinent studies appear in the above ref 5a–d as well as in ref 6.

(6) (a) Roush, W. R.; D'Ambra, T. E. *J. Org. Chem.* **1980**, *45*, 3927. (b) Roush, W. R.; D'Ambra, T. E. *Ibid.* **1981**, *46*, 5045.

(7) Our synthesis of epoxy alcohol i, a precursor of lactone **2**, has been improved as shown below. This sequence is two steps longer than that originally reported[6b] but affords i in 57–60% overall yield together with 4–6% of the alcohol epimer. We thank J. K. Rogowski for performing these experiments.



(i)

(8) (a) All new compounds were fully characterized by 250- or 270-MHz $^1$H NMR, IR, and mass spectroscopy. (b) A satisfactory combustion analysis (±0.3% for C, H) was obtained for this compound.

(9) Grieco, P. A.; Hiroi, K. *J. Chem. Soc., Chem. Commun.* **1972**, 1317.

(10) (a) Snider, B. B.; Amin, S. G. *Synth. Commun.* **1978**, *8*, 117. (b) Cookson, R. C.; Cramp, M. C.; Parsons, P. J. *J. Chem. Soc., Chem. Commun.* **1980**, 197.

(11) Longer reaction times or higher temperatures did not lead to improved yields of **8a,b**. The product ratio was, however, somewhat temperature dependent with greater amounts of **8b** relative to **8a** being obtained at higher cycloaddition temperatures.

(12) Cycloadducts **8a** and **8b** were separated by fractional crystallization from hexane–benzene. Each isomer was transformed independently to a mixture of **9a** and **9b** by using conditions similar to those described in text.

(13) (a) Masuoka, N.; Kamikawa, T. *Tetrahedron Lett.* **1976**, 1691. (b) Masuoka, N.; Kamikawa, T.; Kubota, T. *Chem. Lett.* **1974**, 751.

(14) (a) Diacetate **10b** was recycled to the diol bromo ether by treatment with K$_2$CO$_3$ in methanol (88%). (b) The ratio of **10a**:**10b** was 1:1 when acylation catalysts such as 4-(dimethylamino)pyridine were employed or when greater excesses of Ac$_2$O were used.

**Acknowledgment.** This research was generously supported by a grant from the National Cancer Institute (Grant CA 26830). We thank Professor Ch. Tamm for providing an authentic sample of natural verrucarol.

**Supplementary Material Available:** Spectroscopic data ($^1$H NMR, IR, mass), physical constants, and combustion analytical or high-resolution mass spectral data are reported for all synthetic intermediates (7 pages). Ordering information is given on any current masthead page.

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(15) Natural verrucarol crystallized from hexane–benzene had mp 160–161 °C (lit. mp 158–159 °C (ether–$CH_2Cl_2$); see: Cole, R. J.; Cox, R. H. "Handbook of Toxic Fungal Metabolites"; Academic Press: New York, 1981; p 157).

(16) This synthesis is comparable in length (21 steps from (methylcyclopentadienyl)trimethylsilane)) to that reported by Schlessinger (20 steps from 2-methylcyclopentane-1,3-dione). The synthesis recently reported by Trost[17] also proceeds in 20 steps from 2-methylcyclopentane-1,3-dione.

(17) A synthesis of racemic verrucarol was published by Trost after our paper was submitted for publication (Trost, B. M.; McDougal, P. G. *J. Am. Chem. Soc.* **1982**, *104*, 6110). This group reports a melting point of 165.5–167 °C (ether–$CHCl_3$) for the synthetic trichothecene.

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# Incorporating Metal–Metal Multiple Bonds into Heterometallic Chains: Bis(tris(trimethylstannyl)tin)tetrakis(dimethylamido)-dimolybdenum and -ditungsten and Related Compounds

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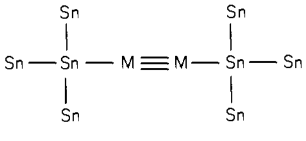
*Received September 23, 1982*

There is now a fairly extensive body of chemistry surrounding compounds containing metal-to-metal multiple bonds.[1] To our knowledge, however, compounds containing the connectivity $M'{-}M{\equiv}M{-}M'$ or $M'{-}M\overset{4}{-}M{-}M'$, wherein a central metal-metal triple or quadruple bond supports metal-metal bonds to different metal atoms, have not been reported. Compounds of this type could play a significant role in the development of polynuclear/cluster chemistry. We here report our preparation and characterization of compounds of formula 1,2-$M_2(Sn(SnMe_3)_3)_2(NMe_2)_4$ which contain the metal atom connectivity shown in I.



I, M = Mo and W

Previous work has shown that $M_2Cl_2(NMe_2)_4$[2] compounds (M = Mo, W) are labile toward metathetic reactions described by the generalized equation (1), where Y = R (alkyl),[3] OR, $NR_2$, and SR.[4]

$$M_2Cl_2(NMe_2)_4 + 2LiY \rightarrow M_2Y_2(NMe_2)_4 + 2LiCl \quad (1)$$

We now find that employing $(THF)_3LiSn(SnMe_3)_3$[5] in eq 1 gives the new compounds $M_2(Sn(SnMe_3)_3)_2(NMe_2)_4$ as orange, hydrocarbon-soluble, air-sensitive crystalline solids.[6]

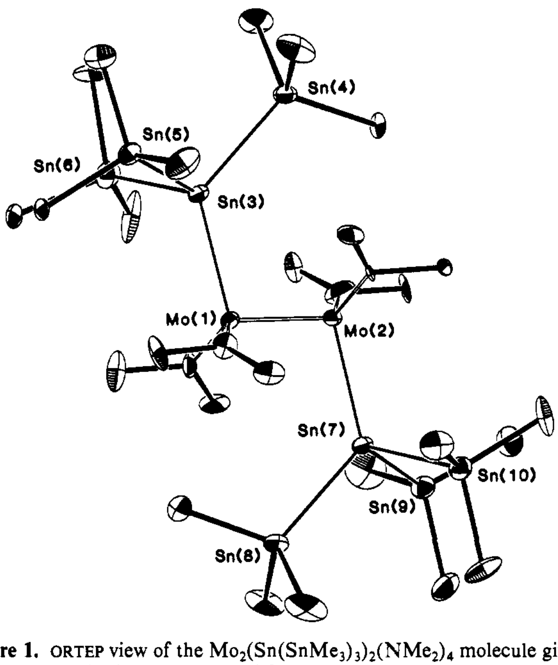


**Figure 1.** ORTEP view of the $Mo_2(Sn(SnMe_3)_3)_2(NMe_2)_4$ molecule giving the atom numbering scheme used for the metal atoms. Atoms are represented by ellipsoids drawn to include 50% probability of thermal displacement. Pertinent bond distances (Å) and angles (deg) are as follows: Mo–Mo = 2.201 (2); Mo(1)–Sn(3) = 2.783 (2); Mo(2)–Sn(7) = 2.774 (2); Sn(3)–Sn(4), –Sn(5), –Sn(6) = 2.775 (2), 2.774 (2), 2.779 (2); Sn(7)–Sn(8), –Sn(9), –Sn(10) = 2.768 (2), 2.777 (2), 2.774 (2); Mo–N = 1.95 (1) (averaged); Sn(3)–Mo(1)–Mo(2) = 100.6 (1); Sn(7)–Mo(2)–Mo(1) = 100.7 (1), Mo–Mo–N = 105 (2) (averaged).

A view of the molecular structure of the molybdenum compound, deduced from a single-crystal X-ray study,[7] is shown in Figure 1. The geometry about each tin atom is close to that expected for its use of essentially tetrahedral hybrid orbitals, while the central $Mo_2Sn_2N_4$ skeleton is akin to that observed in a number of other molecules of formula $M_2X_2(NMe_2)_4$ having the anti conformation, e.g., $Mo_2X_2(NMe_2)_4$, where X = Me[8] and Cl.[2] The distance associated with the Mo$\equiv$Mo bond and the Mo–Sn and Sn–Sn bonds are all well within the range expected on the basis of previous structural studies.[9]

In toluene-$d_8$ and benzene-$d_6$, the anti conformation is apparently maintained to the exclusion of the gauche rotamer.[10]

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(1) Cotton, F. A.; Walton, R. A. "Multiple Bonds Between Metal Atoms"; Wiley: New York, 1982.

(2) Akiyama, M.; Chisholm, M. H.; Cotton, F. A.; Extine, M. W.; Murillo, C. A. *Inorg. Chem.* **1977**, *16*, 2407.

(3) Chisholm, M. H.; Haitko, D. A.; Huffman, J. C. *J. Am. Chem. Soc.* **1981**, *103*, 4046.

(4) Chisholm, M. H.; Corning, J. F.; Huffman, J. C. *Inorg. Chem.* **1982**, *21*, 286.

(5) Brown, T. L.; Wells, W. L. *J. Organomet. Chem.* **1968**, *11*, 271.

(6) Reactions were carried out by using standard procedures for the manipulation of air-sensitive materials. Preparation of $Mo_2(Sn(SnMe_3)_3)_2(NMe_2)_4$: $(THF_3)_3LiSn(SnMe_3)_3$ (1.05 g, 1.26 mmol) dissolved in hexane (20 mL) was added to $Mo_2Cl_2(NMe_2)_4$ (0.277 g, 0.631 mmol) suspended in hexane (20 mL). Upon addition, the mixture turned orange with the formation of an orange precipitate. After the solution stirred for 1 h at room temperature, the solution was filtered, and the solids were washed with hexane (10 mL, five times) until all the orange solids were extracted into the filtrate. The solution was then placed in a refrigerator at −15 °C, and the orange crystals, which formed over a period of 12 h, were collected by filtration and dried in vacuo (0.45 g, 44% based on Mo). $W_2(Sn(SnMe_3)_3)_2(NMe_2)_4$ was prepared similarly. Satisfactory elemental analyses were obtained for both compounds. Crystals of the molybdenum compound suitable for X-ray studies were obtained by recrystallization from toluene.

(7) Crystal data for the $Mo_2(Sn(SnMe_3)_3)_2(NMe_2)_4$ molecule at −162 °C: space group $Pbn2_1$, $a$ = 14.600 (4) Å, $b$ = 31.737 (13) Å, $c$ = 13.545 (4) Å, $Z$ = 4, $d_{calcd}$ = 1.876 g cm$^{-3}$. Of the 4330 unique intensities collected by using Mo K$\alpha$, 6° < 2$\theta$ < 45°, the 4105 reflections having $F > 2.33\sigma(F)$ were used in the full-matrix refinement. The final residuals are $R_F$ = 0.046 and $R_{wF}$ = 0.049. The unit cell contains two toluene molecules. One molecule (C(41)–C(47)) is well ordered while the other (C(49)–C(57)) is disordered.

(8) Chisholm, M. H.; Cotton, F. A.; Extine, M. W.; Murillo, C. A. *Inorg. Chem.* **19788** *17*, 2338.

(9) Mo–Sn = 2.753 (3) Å in $Cl_2MeSnMo(CO)_3(bpy)$: Elder, M.; Graham, W. A. G.; Hall, D.; Kumner, R. *J. Am. Chem. Soc.* **1968**, *90*, 2189. Sn–Sn = 2.77 (1) Å in $(SnPh_2)_6$: Olson, D. H.; Rundle, R. E. *Inorg. Chem.* **1963**, *2*, 1310. Mo–Mo = 2.203 (1) Å in 1,2-$Mo_2Et_2(NMe_2)_4$: ref 3. For an extensive listing of Mo$\equiv$Mo bond distances in $Mo_2X_2(NMe_2)_4$ compounds, see: Chisholm, M. H. *Transition Met. Chem.* **1978**, *3*, 321.

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