Volume 29
Number 15
July 25, 1990

Inorganic Chemistry

Communications

Synthesis and Characterizations of Two Novel Heterometallic Trinuclear Incomplete Cubane-Like Clusters $[(CH_3CH_2)_4N]\{M_2CuS_4\}(S_2C_2H_4)_2(PPh_3)$ (M = Mo, W)

There is extensive interest in trinuclear triangular metal clusters.[1] The trimolybdenum clusters with the incomplete cubane-like cluster core $[Mo_3S_4]^{4+}$ [2] have attracted many chemists, not only because the $[Mo_3S_4]^{4+}$ aquo ion has special stability[3] but also because it may be employed as a starting material to react with many metals, e.g., Fe,[4] Cu,[5,6] Ni,[7] etc., to form cubane-like clusters or to dimerize to form an Mo_6S_8 cluster.[8] Several tritungsten clusters with the incomplete cubane-like cluster core $W_3S_4^{4+}$ [9] and the cubane-like cluster core $W_3CuS_4^{3+}$ [10] have also been synthesized. However, a heterometallic trinuclear incomplete cubane-like cluster has not been previously reported. Herein, the synthesis and characterization of the novel heterometallic trinuclear clusters with the incomplete cubane-like cores $[M_2CuS_4]^{3+}$ (M = Mo, W) are described.

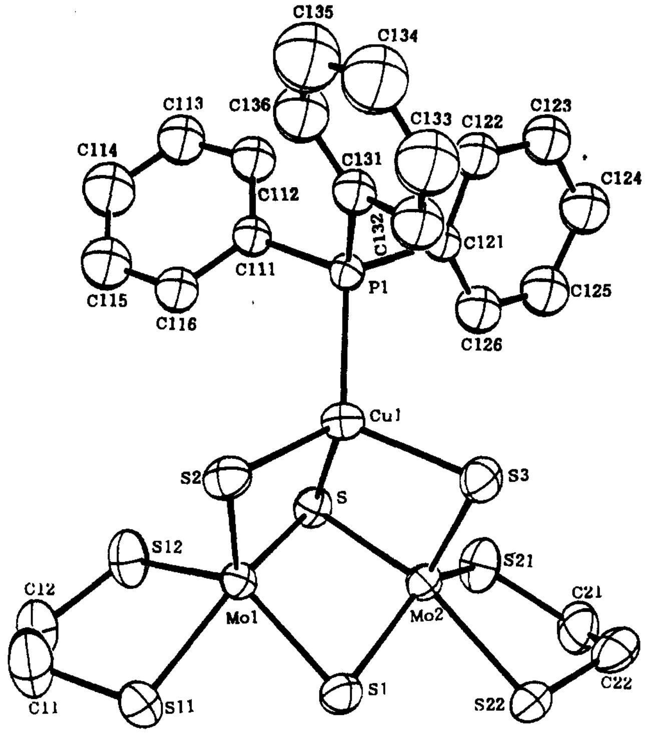

Figure 1. Configuration of $Mo_2CuS_4(edt)_2(PPh_3)^-$ anion and selected bond lengths (Å). Mo1–Mo2 = 2.852 (2), Mo1–Cu1 = 2.802 (2), Mo2–Cu1 = 2.760 (2), Mo1–S = 2.353 (4), Mo2–S = 2.358 (4), Mo1–S1 = 2.307 (4), Mo2–S1 = 2.316 (4), Mo1–S2 = 2.169 (4), Mo2–S3 = 2.161 (4), Cu1–S = 2.265 (4), Cu1–S2 = 2.409 (4), Cu1–S3 = 2.313 (4), Cu1–P = 2.228 (4).

The title compounds $[(CH_3CH_2)_4N]M_2CuS_4(PPh_3)(edt)_2$ (M = Mo (I) and M = W (II), where edt is the ligand 1,2-ethanedithiolato) were synthesized by the unit-construction method,[11] a convenient method that uses reactive fragments as building blocks to obtain clusters.

The dinuclear dimolybdenum compound $[(CH_3CH_2)_4N]_2Mo_2S_4(edt)_2$ (III) was prepared under dinitrogen atmosphere according to the procedure of Stiefel et al.[12] and reacted with the copper complex $Cu(PPh_3)_2(dtp)$[13] (where dtp is the ligand $S_2P(OCH_2CH_3)_2^-$). Dark red crystals of I were ob-

---

(1) Müller, A.; Jostes, R.; Cotton, F. A. *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 875.

(2) (a) Vergamini, P. J.; Vahrenkamp, H.; Dahl, L. F. *J. Am. Chem. Soc.* **1971**, *93*, 6327. (b) Müller, A.; Reinsch, U. *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 72. (c) Halbert, T. R.; McGauley, K.; Pan, W. H.; Czernuszewicz, R. S.; Stiefel, E. I. *J. Am. Chem. Soc.* **1984**, *106*, 1849. (d) Cotton, F. A.; Dori, D.; Llusar, R.; Schowotzer, W. *Inorg. Chem.* **1986**, *25*, 3654. (e) Kathirgamanathan, P.; Martinez, M.; Sykes, A. G. *J. Chem. Soc., Chem. Commun.* **1985**, 1437. (f) Lin, X.-T.; Lin, Y.-H.; Huang, J.-L.; Huang, J.-Q. *Kexue Tongbao* **1987**, *32*, 810. (g) Shibahara, T.; Kuroya, H. *Polydedron* **1986**, *5*, 357.

(3) Cotton, F. A.; Dori, Z.; Llusar, R.; Schwotzer, W. *J. Am. Chem. Soc.* **1985**, *107*, 6734.

(4) Shibahara, T.; Akashi, H.; Kuroya, H. *J. Am. Chem. Soc.* **1986**, *108*, 1342.

(5) (a) Wu, X.-T.; Lu, S.-F.; Zhu, N.-Y.; Wu, Q.-J.; Lu, J.-X. *Inorg. Chim. Acta* **1987**, *133*, 39. (b) Lu, S.-F.; Zhu, N.-Y.; Wu, X.-T.; Wu, Q.-J.; Lu, J.-X. *J. Mol. Struct.* **1989**, *197*, 15.

(6) Shibahara, T.; Akashi, H.; Kuroya, H. *J. Am. Chem. Soc.* **1988**, *110*, 3313.

(7) Shibahara, T.; Kuroya, H. *Abstracts of Papers*, 1987 International Miniconference for Young Chemists in Tokyo Macrocyclic and Coordination Chemistry, Tokyo, July 1987.

(8) (a) Shibahara, T.; Yamamoto, T.; Kanadani, H.; Kuroya, H. *J. Am. Chem. Soc.* **1987**, *109*, 3495. (b) Saito, T.; Yamamoto, N.; Yamagata, T.; Imoto, H. *J. Am. Chem. Soc.* **1988**, *110*, 1646.

(9) (a) Shibahara, T.; Kohda, K.; Ohtsuji, A.; Yasuda, K.; Kuroya, H. *J. Am. Chem. Soc.* **1986**, *108*, 2757. (b) Shibahara, T.; Takeuchi, A.; Ohtsuji, A.; Kohda, K.; Kuroya, H. *Inorg. Chim. Acta* **1987**, *127*, L45. (c) Zhang, H.-Q.; Zheng, Y.-F.; Wu, X.-T.; Lu, J.-X. *J. Mol. Struct.* **1989**, *196*, 241. (d) Zheng, Y.-F.; Zhang, H.-Q.; Wu, X.-T. *Acta Crystallogr.* **1989**, *C45*, 1424.

(10) (a) Zhang, H.-Q.; Zheng, Y.-F.; Wu, X.-T.; Lu, J.-X. *Inorg. Chim. Acta* **1989**, *156*, 277. (b) Zheng, Y.-F.; Zhang, H.-Q.; Wu, X.-T.; Lu, J.-X. *Transition Met. Chem. (London)* **1989**, *14*, 161.

(11) Wu, X.-T.; Lu, J.-X. *Jiegou Huaxue (J. Struct. Chem.)* **1989**, *8*, 399.

(12) Pan, W.-H.; Chandler, T.; Enemark, J. H.; Stiefel, E. I. *Inorg. Chem.* **1984**, *23*, 4265.

(13) Anal. Found (calcd): Cu, 8.68 (8.22); S, 7.82 (8.28); C, 61.84 (62.10); P, 12.54 (12.03); H, 5.08 (5.17). IR (KBr pellet, cm^{-1}): 3020 (w), 1580 (w), 740 (s), 690 (s) for PPh_3; 2960 (w), 2880 (w), 1470 (s), 1430 (s), 1380 (s), 1090 (s), 1070–1000 (wide, vs), 960–910 (wide, vs), 780 (s), 660 (s), 510 (s) for $S_2P(OCH_2CH_3)_2^-$.

0020-1669/90/1329-2705$02.50/0 © 1990 American Chemical Society

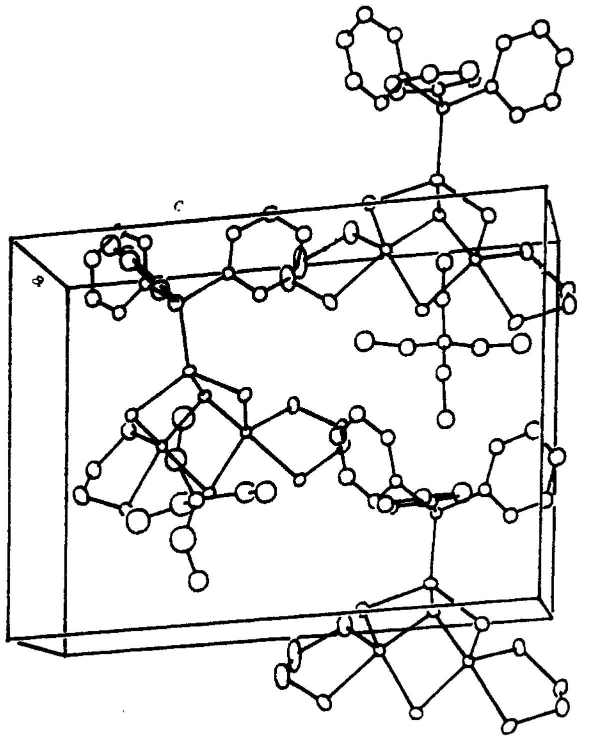

Figure 2. Packing drawing of the unit cell. For clarity only one conformation of the disordered $[(CH_3CH_2)_4N]^+$ is shown.

tained from the CH_2Cl_2/CH_3CN solutions at room temperature. Satisfactory analytical results were obtained for all elements. The infrared spectrum is given in ref 14a.

The isomorphous compound II was synthesized as above by the reaction of the dinuclear ditungsten complex $[(CH_3CH_2)_4N]_2W_2S_4(edt)_2$ (IV)[12] with $Cu(PPh_3)_2(dtp)$. Orange crystals[15] were obtained from CH_2Cl_2/CH_3CN by gaseous diffusion of CH_3CH_2OH solvent. Satisfactory analytical results were also obtained for all elements, and the infrared spectrum is given in ref 14b.

Diffraction data were collected on a CAD-4 κ-geometry diffractometer using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). Cell constants were obtained by a least-squares fit to 25 diffraction maxima ($24° \leqslant 2\theta \leqslant 28°$). Crystal data for $[Mo_2CuS_8PNC_{30}H_{43}]_2$ are as follows: space group triclinic $P1$, a = 9.671 (3) Å, b = 12.341 (2) Å, c = 16.857 (3) Å, α = 83.91 (1)°, β = 86.40 (4)°, γ = 79.30 (3)°, V = 1963.8 Å^3, Z = 1; fw = 1834.5, d_{calcd} = 1.55 g·cm^{-3}; μ = 16.2 cm^{-1}, T = 297 K, $\omega/2\theta$ scan, scan speed varied from 1 to 7° min^{-1} (in ω); the scan width was 0.47° + 0.35° tan θ. The intensities were corrected for Lorentz and polarization factors and for abaorption by using empirical scan data and DIFABS[16] (transmission factors ranged from 0.893 to 1.114 with an averaged value of 1.000) to give a total of 7154 reflections, up to a maximum 2θ of 50° ($-11 \leqslant h \leqslant +11$, $-14 \leqslant k \leqslant +14$, $0 \leqslant l \leqslant +20$), and 6122 reflections with $I > 3\sigma(I)$ were used in the refinement. The structure was solved by direct methods using MULTAN 11/82[17] and the positions of six heavy atoms were obtained from the E map. The remaining non-hydrogen atoms were located in the succeeding difference Fourier synthesis. The structure was refined by full-matrix least-squares techniques with anisotropic thermal parameters for the 32 atoms of the cores and isotropic thermal parameters for the remaining non-H atoms (527 variables). All calculations were performed on a VAX 785 computer using the SDP program package, the scattering factors were taken from Cromer and Waber.[18] Final refinement converged to R = 0.056, R_w = 0.065, and S = 1.27.[19]

The ORTEP drawing of the one independent anion of $\{Mo_2CuS_4(edt)_2(PPh_3)\}^-$ is shown in Figure 1, and the packing drawing of the unit cell, in Figure 2. The unit cell contains two discrete anions and two cations. The two discrete anions have the same orientation for the $CuPPh_3$ groups along the b axis, so that the space group is undoubtedly noncentrosymmetric. The PPh_3 ligands of the two independent anions have some difference in configuration. One of the two cations $[(CH_3CH_2)_4N]^+$ in the unit cell is disordered. Except for the difference of the PPh_3 ligands of the two independent anions, the two cores are very similar, so only one core (Figure 1) is discussed below.

Each Mo atom displays tetragonal-pyramidal coordination by five sulfurs: one μ_3-S atom, two μ_2-S atoms, and two terminal sulfurs of the edt ligand. The Cu atom is tetrahedrally coordinated by one μ_3-S atom, two μ_2-S atoms, and one PPh_3 ligand. The bond lengths and bond angles of the $Mo_2S_4(edt)_2$ moiety in the trinuclear cluster are almost the same as in the isolated dinuclear dimolybdenum complex.[20] Only the Mo-S double bond lengths elongate from 2.10 Å (average) to 2.165 Å (average), when the terminal S atom becomes a μ_2-S atom.

The $Mo_3S_4^{4+}$ core has approximately C_{3v} symmetry. But, the heterometallic trinuclear cluster core, $Mo_2CuS_4^{3+}$, has lower symmetry. In the core (Figure 1), the Mo-Cu bonds are 2.771 Å (average), which is shorter than Mo–Mo bond. The Cu–μ_3-S has a bond length of 2.265 Å, but the two Cu–μ_2-S bond lengths are significantly different; one Cu–μ_2-S is 2.313 Å and the other is 2.409 Å. Therefore, the cluster core $Mo_2CuS_4^{3+}$ is distorted. Although the $Mo_2CuS_4^{3+}$ and $Mo_3S_4^{4+}$ cores do not have the same symmetry, they are expected to have the similar reactivity, e.g. to combine with a metal atom to form a cubane-like cluster. On the basis of the stability, substitution, addition and oxidation, etc, the quasi-aromaticity hypothesis in the $Mo_3(\mu_2\text{-}S)_3$ structural unit of the $Mo_3S_4^{4+}$ core has been proposed by Huang et al.[21] Whether the $Mo_2Cu(\mu_2\text{-}S)_3$ structural unit of the $Mo_2CuS_4^{3+}$ core also has such quasi-aromaticity will be advanced in the future.

The UV-vis spectrum of the four compounds are cited in ref 22 for comparison. The cyclic voltammography has been done in the range of 0.0 to −1.75 V. I displays a pair of redox waves at −1.28 and −1.07 V while compound II displays no electron-transfer process in this range.

Acknowledgment. This research was supported by grants from the Structural Chemistry Research Laboratory of the Fujian Institute of Research on Structure of Matter, Chinese Academy of Sciences, and the National Science Foundation of China.

Supplementary Material Available: Tables of atomic coordinates and equivalent isotropic thermal parameters, bond distances, bond angles, general displacement parameters, and crystallographic data (5 pages);

---

(14) (a) IR of Mo_2CuS_4 core cluster (KBr pellet, cm^{-1}): 780 (m), 750 (s), 710 (m), 695 (s) for C–H vibrations of benzyl; 520 (s), 510 (m), 495 (s), 465 (w), 430 (w), 350 (m), 325 (w), 310 (w). (b) IR of W_2CuS_4 core cluster (KBr pellet, cm^{-1}): 780 (m), 750 (s), 710 (m), 695 (s) for C–H vibrations of benzyl; 520 (s), 508 (m), 500 (m), 480 (s), 425 (w), 350 (m), 310 (m).

(15) Primary cell constants: a = 9.675 (8) Å, b = 12.389 (11) Å, c = 16.865 (14) Å, α = 83.91 (7); β = 86.19 (7)°, γ = 79.17 (8)°; V = 1972.1 Å^3.

(16) Walker, N.; Stuart, D. *Acta Crystallogr.* **1983**, *A39*, 159.

(17) Main, P.; J. Fiske, S.; Hull, S. E.; Lessinger, L.; Germain, G.; Declercq, J. P.; Woolfson, M. M. MULTAN 11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data. Universites of York, England, and Louvain, Belgium, 1982.

(18) Cromer, D. T.; Waber, J. T. *International Tables for X-ray Crystallogrphy*; Kynoch Press: Birmingham, England, 1974; Vol. IV, Tables 2.2a, 2.3.1 (present distributor Kulwer Academic Publishers, Dordrecht, The Netherlands).

(19) $R = \sum||F_o| - |F_c||/\sum|F_o|$, $R_w = (\sum w(|F_o| - |F_c|)^2/\sum wF_o^2)^{1/2}$, $S = \sum w(|F_o| - |F_c|)^2/(N_{obs} - N_{var}))^{1/2}$, and $w = 1/(\sigma(F_o)^2 + [PWT(F_o)]^2 + QWT) - F_o$ (for PWT = 0.020, QWT = 1.0).

(20) Bunzey, G.; Enemark, J. H. *Inorg. Chem.* **1978**, *17*, 682.

(21) Huang, J.-Q.; Lu, S.-F.; Shang, M.-Y.; Lin, X.-T.; Huang, M.-D.; Lin, Y.-H.; Wu, D.-M.; Zhuang, H.-H.; Huang, J.-L.; Lu, J.-X. *Jiegou Huaxue* (*J. Struct. Chem.*) **1987**, *6*, 219 (Chinese).

(22) UV-vis spectrum (λ, nm (ϵ, 10^3 M^{-1}·cm^{-1})): compound I, 228 (49), 305 (23), 482 (8.6); compound II, 228 (44), 280 (30.7), 405 (11); compound III, 228 (37), 306 (26), 445 (24); compound IV, 235 (24), 284 (20), 362 (6.6).

tables of structure factors (18 pages). Ordering information is given on any current masthead page.

Fuzhou Laboratory of Structural Chemistry
Fujian Institute of Research on Structure of Matter
Chinese Academy of Sciences
Fuzhou, Fujian 350002, P. R. China

Zhu Nianyong
Zheng Yifan
Wu Xintao*

Received January 24, 1990

Facile Synthesis and Structural Principles of the Strontium Phenoxide $Sr_4(OPh)_8(PhOH)_2(THF)_6$

The current knowledge of composition and structure of aryloxides and alkoxides of divalent metals throughout the periodic table remains underdeveloped:[1] only compounds of germanium,[2] tin,[2] chromium,[3] cobalt,[4] and zinc[5] have been structurally characterized. The propensity of binary alkoxides of the heavier metals derived from low molecular weight alcohols to be polymeric,[6] involatile, and insoluble has led to recent emphasis on bulky R groups, including ortho-disubstituted phenoxides.[7] To avoid several characteristics of such elaborate aryloxides, we have examined unsubstituted phenoxide as a ligand for strontium in the presence of Lewis bases.[8,9] Our goal is molecular phenoxides that are volatile and/or hydrolyzable.[10] Our choice of strontium[11] is dictated by its importance as a constituent of materials valued for their structural[12] ($SrTiO_3$ or $SrNb_2O_6$) and superconducting ($La_{2-x}Sr_xCuO_4$[13] or $Pb_2Sr_2LnCu_3O_{8+x}$[14]) properties.

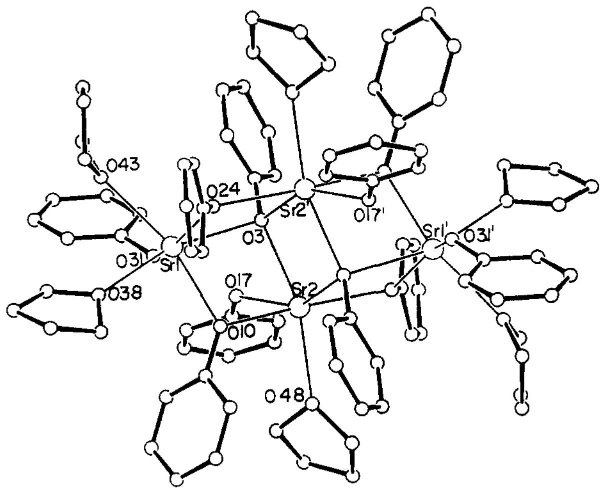

Figure 1. ORTEP drawing of the non-hydrogen atoms of $Sr_4(OPh)_8(PhOH)_2(THF)_6$. Unlabeled atoms are carbon, and primed atoms are related to those not primed by a center of symmetry. Selected structural parameters: Sr1–O31, 2.450 (7) Å; Sr2–O17, 2.548 (7) Å; Sr–(μ_2-O), 2.404 (7)–2.495 (7) Å; Sr–(μ_3-O), 2.512 (7)–2.558 (6) Å; Sr–OC_4H_8, 2.548 (8)–2.584 (8) Å.

Reaction of strontium metal with phenol in THF at reflux, followed by crystallization by the addition of hexane, gives a 90% yield of a material[15] of empirical formula $Sr(OPh)_2(PhOH)_{0.5}(THF)_{1.5}$. Although this gives a ligand:metal ratio of 4:1, the curious stoichiometry and the incorporation of two *different* neutral ligands require explanation.

The solid state structure[16] of this compound (Figure 1) shows it to contain four metals in a centrosymmetric structure of formula $Sr_4(\mu_3\text{-}OPh)_2(\mu_2\text{-}OPh)_4(OPh)_2(THF)_6(PhOH)_2$. This structure results in a coordination number of 6 for each metal (yet the metals Sr1 and Sr2 have different environments) and approximate octahedral geometry. It is rare to find phenoxide as a μ_3-ligand.[17] The compound possesses the generic $M_2M'_2(\mu_3\text{-}X)_2(\mu_2\text{-}X)_4L_n$ stoichiometry and structure also adopted by $Li_2Sn_2(O^tBu)_6$[18] and $Mo_4O_{10}(OMe)_6^{2-}$.[19] When n (the number of terminal ligands) equals 10, this structure contains both edge- and face-shared octahedra and is thus highly suited to a metal like Ti(IV) in $Ti_4(OMe)_{16}$.[1] The generality of the structure,[20] however, rests in its ability to accommodate a variety of different terminal ligands (n values) and thus metals in oxidation states other than +4. For example, the Grignard-derived material of empirical formula $MgEt_{0.5}Cl_{1.5}(THF)_{1.5}$ is in fact[21] $Mg_4Et_2Cl_6(THF)_6$ with the same $Mg_4(\mu_3\text{-}Cl)_2(\mu_2\text{-}Cl)_4$ core structure and terminal ethyl and THF ligands that give two 6- and two 5-coordinate Mg^{2+} ions.

The incorporation of only two phenol molecules and the site they occupy in the structure are controlled by hydrogen bonding. The two phenol ligands occupy coordination sites that place their Sr–O bonds approximately parallel to the Sr–O bonds of the only two *terminal* phenoxides in the structure. Such a parallel alignment of M–O(H)R and M–OR bonds is clearly optimal for intramolecular hydrogen bonding. The hydrogen bonding in $Sr_4(OPh)_8(PhOH)_2(THF)_6$ is unusually strong.[22] This is evident

---

(1) Bradley, D. C.; Mehrotra, R. C.; Gaur, D. P. *Metal Alkoxides*; Academic Press: London, 1978.

(2) Cetinkaya, B.; Gumrukcu, I.; Lappert, M. F.; Atwood, J. L.; Rogers, R. D.; Zaworotko, M. J. *J. Am. Chem. Soc.* **1980**, *102*, 2088.

(3) Murray, B. D.; Hope, H.; Power, P. P. *J. Am. Chem. Soc.* **1985**, *107*, 169.

(4) Sigel, G. A.; Bartlett, R. A.; Decker, D.; Olmstead, M. M.; Power, P. P. *Inorg. Chem.* **1987**, *26*, 1773.

(5) Geerts, R. L.; Huffman, J. C.; Caulton, K. G. *Inorg. Chem.* **1986**, *25*, 1803.

(6) Solid $Sr(OMe)_2$ has been shown by powder patterns to be polymeric and lacking in any molecular subunits: Staeglich, H.; Weiss, E. *Chem. Ber.* **1978**, *111*, 901.

(7) Duff, A. W.; Hitchcock, P. B.; Lappert, M. F.; Taylor, R. G.; Segal, J. A. *J. Organomet. Chem.* **1985**, *293*, 271.

(8) The available ether is essential since strontium metal is not consumed by phenol at reflux for 5 h in toluene (i.e., no THF present).

(9) The occurrence of alcohol solvates has been reported: Lutz, H. D. *Z. Anorg. Allg. Chem.* **1968**, *356*, 132.

(10) The very weakly basic di- and trinitrophenoxide groups will form compounds with several alkaline-earth metals *from Aqueous Solution*. In these, the aryloxide may not be coordinated or may be bidentate (through phenoxo and nitro oxygen). See: Kanters, J. A.; Postma, R.; Duisenberg, A. J. M.; Venkata Subramanian, K.; Poonia, N. S. *Acta Crystallogr.* **1983**, *C39*, 1221, 1519. Sarma, R.; Ramirez, F.; Narayanan, P.; McKeever, B.; Maracek, J. F. *J. Am. Chem. Soc.* **1979**, *101*, 5015. Cole, L. B.; Holt, E. M. *J. Chem. Soc., Perkin Trans. 2*, **1986**, 1997.

(11) Smith, J. S.; Dolloff, R. T.; Mazdiyasni, K. S. *J. Am. Ceram. Soc.* **1970**, *53*, 91.

(12) Riman, R. E.; Haaland, D. M.; Northrup, C. J. M.; Bowen, H. K.; Bleier, A. In *Ultrastructure Processing of Advanced Ceramics*; Mackenzie, J. D., Ulrich, D. R., Eds.; Wiley: New York, 1988; p 233.

(13) Bednorz, J. G.; Müller, K. A. *Z. Phys. B: Condens. Matter* **1986**, *64*, 189.

(14) Cava, R. J.; Ballogg, B.; Krajewski, J. J.; Rupp, L. W.; Schneemeyer, L. F.; Siegrist, T.; Van Dover, R. B.; Marsh, P.; Peck, W. F.; Gallagher, P. K.; Glarum, S. H.; Marshall, J. H.; Farrow, R. C.; Waszczak, J. V.; Hall, R.; Trevor, P. *Nature (London)* **1988**, *336*, 211.

(15) Anal. Calcd for $C_{84}H_{100}O_{16}Sr_4$ (found): C, 58.72 (58.49); H, 5.83 (5.82). DSC (below 150 °C): endotherms with maxima at 99, 109, and 118 °C. Selected spectroscopic data are as follows. IR (Nujol): 3480 cm^{-1} (ν(OH)). ^1H NMR (500 MHz, 25 °C, THF-d_8): δ 8.32 (br s, OH), 7.15 (m, Ph), 6.98 (m, Ph), 6.84 (br s, Ph), 6.45 (t, Ph), 3.6 (m, THF), 1.8 (m, THF). ^{13}C NMR (126 MHz, 25 °C, THF-d_8): δ 25.3 (m, THF), 67.4 (m, THF), 116.0 (s, Ph), 119.7 (s, Ph), 130.1 (s, Ph), 165.2 (s, Ph).

(16) Crystallographic data for $C_{84}H_{100}O_{16}Sr_4$ (grown from toluene/pentane) at −100 °C: a = 25.296 (4) Å, b = 14.477 (2) Å, c = 22.649 (4) Å, β = 100.36 (1)° with Z = 4 in space group $C2/c$. $R(F)$ = 0.0891 and $R_w(F)$ = 0.0836 for 4417 observed ($F \geq 3\sigma(F)$) reflections.

(17) Malhotra, K. C.; Martin, R. T. *J. Organomet. Chem.* **1982**, *239*, 159. Edema, J. J. H.; Gambarotta, S.; Bolhuis, F.; Spek, A. L. *J. Am. Chem. Soc.* **1989**, *111*, 2142.

(18) Veith, M.; Rösler, R. *Z. Naturforsch.* **1986**, *B41*, 1071.

(19) Liu, S.; Shaikh, S. N.; Zubieta, J. *Inorg. Chem.* **1987**, *26*, 4303.

(20) Caulton, K. G.; Hubert-Pfalzgraf, L. G. *Chem. Rev.*, submitted for publication.

(21) Toney, J.; Stucky, G. D. *J. Organomet. Chem.* **1971**, *28*, 5.

0020-1669/90/1329-2707\$02.50/0 © 1990 American Chemical Society