product, reaction of the solvent seems to occur with the radical anion or the borane dianion at step 1 or 2.

Para substitution of toluene and the position of substitution on the thiaborane may reflect both steric considerations and a tendency toward electrophilic substitution of the aromatic ring by the borane or, conversely, nucleophilic substitution of the thiaborane by toluene. Substitution on the thiaborane would then be expected at the more positive sites, i.e., B(2,3) or B(8,11).

An alternative proposal is attack on the aromatic system by the radical anion $7\text{-}SB_{10}H_{10}\cdot^-$, a $2n + 3 = 25$ framework electron system. Such a system, one electron short of the 26-electron nido framework requirement,[3] may well seek electrons to achieve the more stable nido configuration. On this basis, electrophilic attack by $7\text{-}SB_{10}H_{10}\cdot^-$ on toluene might be expected to give the exceptionally stable 11-vertex nido thiaborane electron count. Thus, simple radical addition to the aromatic solvent would give $2\text{-}R\text{-}7\text{-}SB_{10}H_{11}\cdot^-$, a $2n + 5 = 27$ electron system. An additional one-electron oxidation by silver ion affords the observed $2\text{-}R\text{-}7\text{-}SB_{10}H_{11}$.

To this time there have been no published reports of an oxidative synthesis technique using silver ion in borane or heteroborane chemistry. However, similar complex reactions involving solvent attack and dimerization have been reported in[19] attempted syntheses of metallocarboranes using other transition metals. In these cases, proposed borane radicals might explain many of the unexpected products observed.

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Registry No. $2\text{-}(p\text{-}CH_3C_6H_4)\text{-}7\text{-}SB_{10}H_{11}$, 60294-95-3; $7\text{-}SB_{10}H_{12}$, 60294-96-4; ^{11}B, 14798-13-1; Ag^+, 14701-21-4.

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(8) The dianion is generated from *nido*-$7\text{-}SB_{10}H_{12}$ by removal of bridge hydrogens with 2 equiv of methyl- or *n*-butyllithium.
(9) It had previously been observed[4,12-14] that reactions of 11-vertex boranes and carboranes with electron-pair-donating solvents result in 26-electron nido frameworks. To prevent complexation nondonating aromatic solvents were used in these reactions.
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Structure of Tetrakis(benzoato)dimolybdenum(II)

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For some time, we have been interested in molecules containing strong metal to metal bonds, of which many are now known.[1] Among them, a series of carboxylato compounds of the type $Mo_2(O_2CR)_4$, where R is H,[2] CH_3,[3] CF_3,[4] or $CH_2NH_3^+$,[5] have been characterized. Their air stability is variable with the arylcarboxylato compounds being distinctly less stable. To see if there were any differences in the basic structures, we undertook the x-ray study of tetrakis(benzoato)dimolybdenum(II).

Experimental Section

Crystal Data and Structure Determination. A yellow crystal, obtained by recrystallization from diglyme, measuring 0.38 × 0.34 × 0.30 mm was sealed in a capillary containing some solution (since crystals lose solvent readily) and examined on a Syntex $P\bar{1}$ four-circle automated diffractometer. Axial photographs and ω scans of several intense reflections showed that the crystal quality was good and that it was suitable for x-ray diffraction studies. At small scattering angles the peak widths at half-height were about 0.2°. For calculation of lattice parameters, 15 of the strongest reflections in the range $20° < 2\theta < 31°$ were selected to give a variety of crystal orientations. Based on angular settings for these reflections, the refined lattice parameters obtained from the Syntex software package are (Mo $K\alpha$, λ 0.710 73 Å) $a = 11.321$ (4) Å, $b = 11.547$ (4) Å, $c = 10.489$ (5) Å, $\alpha = 115.62$ (3)°, $\beta = 98.87$ (3)°, $\gamma = 113.72$ (2)°, and $V = 1038.6$ (7) Å^3. For the triclinic space group $P\bar{1}$ with $Z = 1$ and a molecular weight of 944.71, the calculated density is 1.510 g cm^{-3}.

Intensity data were collected at 22 ± 1 °C using graphite-monochromatized Mo $K\alpha$ radiation and a θ–2θ scan rate varying from 4 to 24°/min, depending on the intensity of the reflection. Background measurements were made at both limits of each scan. Of the 2399 integrated intensities collected in the range $0° < 2\theta(\text{Mo } K\alpha) < 43°$, 2055 unique observations with $I > 3\sigma(I)$ were retained as observed data and corrected for Lorentz and polarization effects. Three standard reflections, measured repeatedly every 50 data points, were stable. Since the linear absorption coefficient of this compound is 6.62 cm^{-1} for Mo $K\alpha$ radiation, for any reflection, the maximum relative error due to absorption is <3% of F_o, and the data were not corrected for absorption.

A three-dimensional Patterson map gave the position of the molybdenum atom (the other molybdenum atom is related by symmetry by an inversion center). A difference Fourier synthesis based on the refined molybdenum position revealed all but one of the nonhydrogen atoms. The missing atom appeared on the next difference map. All atoms were assigned isotropic thermal parameters and least-squares refinement then gave discrepancy indices

$$R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.071$$
$$R_2 = [\Sigma w(|F_o| - |F_c|)^2 / \Sigma w |F_o|^2]^{1/2} = 0.098$$

Refinement was continued with anisotropic thermal parameters for the molybdenum atom and isotropic thermal parameters for the rest of the nonhydrogen atoms to convergence at $R_1 = 0.052$ and $R_2 = 0.076$, the error in an observation of unit weight being 2.0. No attempt was made to locate the hydrogen atoms. The function minimized was $\sum w(|F_o| - |F_c|)^2$ where p = 0.06 was used in the previous defined expression for the weights,[6,7] and the scattering factors were from ref 8. Correction for anomalous scattering by molybdenum were taken from Cromer and Liberman.[9] Programs used have been previously reported.[10] A final difference map was judged to be free of significant features. No unusual trends were observed in an analysis of $\sum w(|F_o| - |F_c|)^2$ as a function of $|F_o|$, reflection number, $\lambda^{-1} \sin \theta$, or various classes of indices. A table of observed and calculated structure factor amplitudes is available.[11]

Results

Figure 1 shows the entire contents of one unit cell and defines the atom numbering scheme. Positional and aniso-

Table I. Positional and Thermal Parameters[a] and Their Estimated Standard Deviations for $Mo_2(O_2CC_6H_5)_4 \cdot 2$(digylme)

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Mo	0.555 96 (6)	0.536 14 (7)	0.440 33 (7)	2.44 (3)	2.58 (3)	2.60 (3)	0.80 (3)	0.83 (2)	1.37 (3)

Atom	x	y	z	B_{iso}, Å^2	Atom	x	y	z	B_{iso}, Å^2
O(1)	0.4203 (5)	0.6025 (5)	0.3787 (6)	3.4 (1)	C(8)	0.3240 (7)	0.2233 (8)	0.2327 (8)	2.9 (1)
O(2)	0.6982 (5)	0.4725 (5)	0.4926 (5)	3.3 (1)	C(9)	0.2241 (7)	0.0775 (8)	0.0867 (8)	3.2 (1)
O(3)	0.6934 (5)	0.7530 (5)	0.6431 (5)	3.1 (1)	C(10)	0.2319 (8)	0.0563 (8)	−0.0536 (9)	3.5 (2)
O(4)	0.4251 (5)	0.3251 (5)	0.2312 (6)	3.3 (1)	C(11)	0.1339 (9)	−0.0793 (9)	−0.1912 (10)	4.2 (2)
O(5)	0.7100 (6)	0.6763 (6)	0.3273 (6)	4.4 (1)	C(12)	0.0258 (9)	−0.1992 (10)	−0.1920 (10)	4.3 (3)
O(6)	0.6513 (7)	0.9071 (7)	0.3162 (7)	5.6 (1)	C(13)	0.0217 (8)	−0.1790 (9)	−0.0522 (9)	3.9 (2)
O(7)	0.4011 (7)	0.7757 (8)	0.0677 (8)	6.7 (2)	C(14)	0.1179 (8)	−0.0424 (8)	0.0886 (9)	3.5 (1)
C(1)	0.3181 (8)	0.5800 (8)	0.4223 (8)	3.3 (1)	C(15)	0.6310 (9)	0.6057 (10)	0.1654 (11)	5.1 (2)
C(2)	0.2124 (7)	0.6107 (8)	0.3661 (9)	3.3 (1)	C(16)	0.8000 (10)	0.8387 (11)	0.4138 (11)	5.5 (2)
C(3)	0.1166 (8)	0.6137 (9)	0.4317 (9)	4.0 (1)	C(17)	0.7209 (10)	0.9170 (11)	0.4505 (12)	5.7 (2)
C(4)	0.0138 (9)	0.6388 (10)	0.3743 (11)	5.0 (2)	C(18)	0.5533 (11)	0.9543 (12)	0.3332 (12)	6.2 (2)
C(5)	0.0106 (10)	0.6601 (11)	0.2539 (11)	5.6 (2)	C(19)	0.4835 (11)	0.9324 (12)	0.1847 (13)	6.5 (2)
C(6)	0.1085 (10)	0.6568 (10)	0.1866 (11)	5.1 (2)	C(20)	0.3362 (12)	0.7446 (13)	−0.0813 (14)	7.2 (3)
C(7)	0.2108 (9)	0.6323 (9)	0.2443 (10)	4.3 (2)					

[a] The B_{ij} (in Å^2) are related to the dimensionless β_{ij} employed during refinement as $B_{ij} = 4\beta_{ij}/a^*_i a^*_j$.

Table II. Interatomic Distances (Å)[a-d]

Mo–Mo′	2.100 (1)	C(1)–O(1)	1.280 (8)
Mo–O(1)	2.106 (5)	C(1)–O(2′)	1.276 (9)
Mo–O(2)	2.112 (5)	C(8)–O(3′)	1.275 (8)
Mo–O(3)	2.110 (5)	C(8)–O(4)	1.277 (8)
Mo–O(4)	2.100 (5)	C(1)–C(2)	1.488 (10)
Mo···O(5)	2.663 (6)	C(8)–C(9)	1.470 (10)

[a] Here and in other tables, numbers in parentheses are the estimated standard deviations in the least significant digits. [b] Atoms are labeled as in Figure 1. [c] Primed atoms are symmetry-related to the corresponding unprimed atoms. [d] The average value of the carbon–carbon distance in the phenyl rings is 1.40 Å in one of them and 1.41 Å in the other. In neither one is the individual deviation more than 2 esd's.

Table III. Bond Angles (deg)[a]

Mo′-Mo-O(1)	91.4 (1)	Mo-O(3)-C(8′)	118.0 (4)
Mo′-Mo-O(2)	92.4 (1)	Mo-O(4)-C(8)	117.1 (5)
Mo′-Mo-O(3)	91.0 (1)	O(1)-C(1)-O(2′)	122.2 (7)
Mo′-Mo-O(4)	92.5 (1)	O(1)-C(1)-C(2)	118.4 (7)
Mo′-Mo-O(5)	169.9 (1)	O(2′)-C(1)-C(2)	119.3 (6)
O(1)-Mo-O(2)	176.2 (2)	O(3′)-C(8)-O(4)	121.4 (6)
O(1)-Mo-O(3)	91.6 (2)	O(3′)-C(8)-C(9)	119.7 (6)
O(1)-Mo-O(4)	88.0 (2)	O(4)-C(8)-C(9)	118.9 (6)
O(2)-Mo-O(3)	88.8 (2)	C(1)-C(2)-C(3)	119.5 (7)
O(2)-Mo-O(4)	91.4 (2)	C(1)-C(2)-C(7)	118.9 (7)
O(3)-Mo-O(4)	176.6 (2)	C(8)-C(9)-C(10)	120.3 (7)
Mo-O(1)-C(1)	117.5 (5)	C(8)-C(9)-C(14)	119.4 (7)
Mo-O(2)-C(1′)	116.4 (5)		

[a] The average value of the C–C–C angle for each phenyl ring is 120.0° with no individual angle deviating by more than 2 esd's from that value.

tropic thermal parameters are listed in Table I. Tables II and III give some pertinent interatomic distances and angles.

Discussion

The arrangement of the benzoato ligands about the molybdenum atoms is not significantly different from that found in other carboxylato compounds;[2-5] thus, there are four bridging ligands with the M_2O_8 skeleton having nearly ideal D_{4h} symmetry. In this particular case, half of the molecule is related by symmetry to the other half by a crystallographic inversion center. The compound crystallizes with two solvent molecules.

The Mo–Mo distance (2.100 (1) Å) is also similar to those found previously.

In the formato, acetato, and trifluoroacetato compounds there are weak interactions of each molybdenum atom with an oxygen atom of a carboxylato in a neighboring molecule, along the metal to metal axis, in such a way that an infinite chain of molecules is formed. In this case, there are discrete molecules but Mo···O interactions still exist because the solvent molecules are present and coordinated loosely to the molybdenum atoms as can be seen in Figure 1. The Mo···O distance is 2.663 (6) Å, which is in the same range as those in the other carboxylato compounds, where the variation is from 2.643 Å in the formate to 2.71 Å in the trifluoroacetate.

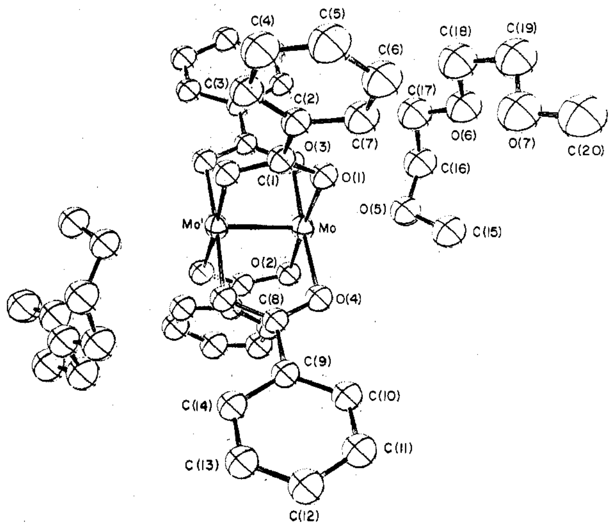

Figure 1. An ORTEP view of $Mo_2(O_2CC_6H_5)_4 \cdot 2$(diglyme). The atom numbering scheme used in all tables is defined. The atoms are represented by 50% probability thermal ellipsoids.

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Registry No. $Mo_2(O_2CC_6H_5)_4 \cdot 2$(diglyme), 60295-02-5.

Supplementary Material Available: Listing of structure factor amplitudes (14 pages). Ordering information is given on any current masthead page.

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