TABLE VII

WEIGHTED LEAST-SQUARES PLANES AND DISTANCES OF THE ATOMS FROM THEIR RESPECTIVE PLANES IN $Cu_4[(i\text{-}C_3H_7O)_2PS_2]_4$[a]

Plane no.	Equation of best plane: $Ax + By + Cz - D = 0$ A	B	C	D	Defining atoms and distances (Å) from plane
1	7.504	4.050	22.708	20.159	Cu(1), −0.026 (3); Cu(2), 0.031 (3); Cu(3), 0.022 (3); S(8), −0.145 (7)
2	3.010	2.820	−31.676	−15.930	Cu(1), 0.039 (3); Cu(2), 0.039 (3); Cu(4), −0.031 (3); S(6), −0.193 (6)
3	9.491	−5.641	−9.853	−4.100	Cu(1), −0.029 (3); Cu(3), 0.026 (3); Cu(4), −0.036 (3); S(4), 0.187 (7)
4	0.842	12.336	1.290	6.177	Cu(2), 0.031 (4); Cu(3), −0.030 (3); Cu(4), −0.032 (4); S(2), 0.144 (7)
5	4.201	10.487	−12.826	0.691	Cu(1), −0.076 (3); S(1), 0.119 (7); S(4), 0.094 (7); S(6), 0.124 (7)
6	10.358	−4.041	7.616	9.903	Cu(2), −0.073 (3); S(3), 0.106 (7); S(6), 0.139 (8); S(8), 0.142 (7)
7	4.474	7.668	−22.920	−9.877	Cu(3), 0.046 (3); S(2), −0.075 (7); S(5), −0.096 (7); S(8), −0.080 (7)
8	9.678	−0.992	17.176	13.456	Cu(4), 0.046 (3); S(2), −0.059 (6); S(4), −0.088 (7); S(7), −0.076 (7)
9	4.998	−1.457	30.062	20.016	Cu(1), 0.009 (3); S(1), −0.080 (8); S(2), −0.038 (7); P(1), 0.058 (7)
10	6.660	9.842	4.738	11.139	Cu(2), 0.003 (4); S(3), −0.035 (8); S(4), −0.021 (7); P(2), 0.033 (7)
11	6.446	−9.987	5.111	2.522	Cu(3), 0.024 (3); S(5), −0.242 (8); S(6), −0.182 (8); P(3), 0.240 (7)
12	8.702	2.060	−20.783	−7.614	Cu(4), −0.021 (3); S(7), 0.228 (8); S(8), 0.126 (7); P(4), −0.263 (8)

[a] Equations of the best planes are expressed in terms of the orthorhombic unit cell axes.

2.90, 2.47, and 2.35 Å, respectively,[23] compared with the respective van der Waals radius sums of 3.0, 2.6, and 2.4 Å, indicating that there are no unusual intermolecular interactions. The closest Cu···H approach occurs at 3.9 Å, well beyond the distance for any significant interaction.

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(23) Based on the final hydrogen coordinates adjusted to give C–H bond lengths of 1.11 Å.

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The Crystal Structure of Triethylammonium Phenylthioarsenate

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The crystal and molecular structure of triethylammonium phenylthioarsenate, $(C_2H_5)_3NH^+(C_6H_5)AsSO(OH)^-$, was determined from three-dimensional X-ray data collected with a manual diffractometer using Mo Kα (λ 0.71069 Å) radiation. The compound crystallizes in the triclinic space group $P\bar{1}$, with a = 11.807 (1) Å, b = 10.558 (1) Å, c = 8.788 (1) Å, α = 116.21 (2)°, β = 98.95 (1)°, γ = 117.91 (1)°, Z = 2, d_{measd} = 1.40 (1) g cm^{-3}, and d_{calcd} = 1.373 (3) g cm^{-3}, at 23°. The structure was determined by Patterson and Fourier methods and refined with 362 observed reflections by full-matrix least squares to an R of 0.051. In the final refinement the phenyl ring was constrained as a rigid group, and the As and S atoms were refined with anisotropic thermal parameters. The anion of the structure consists of an arsenic atom tetrahedrally bonded to a phenyl group, two oxygen atoms, and a sulfur atom at distances of 1.90 (2), 1.65 (2), 1.75 (3), and 2.08 (2) Å, respectively. Hydrogen bonding holds pairs of anions together and one cation to each anion. This is the first monothioarsenate structure to be reported, and the As–S bond length is one of the shortest values reported for that bond.

Introduction

Several structures of cyclic arsenic–sulfur molecules are under investigation in our laboratory, with an interest in the structural evidence for possible p → d or d → d π bonding in the As–S linkages. There is a paucity, however, of As–S bond distances in the structural literature: less than a dozen molecular structures with As–S bonds have been reported. The As–S distances reported range from 2.075 (7) in $(Me_2AsS)_2$[1] to 2.21 to 2.25 Å for several arsenic sulfides (As_4S_3,[2] As_4S_4,[3] and As_4S_6[4]) and the tetrathioarsenate ion,[5] to 2.32–2.35 Å for As(III)–S bonds.[6,7]

We wish to report here the structure of the phenylthioarsenate ion, $(C_6H_5)AsSO(OH)^-$, which has an As–S distance (2.08 (2) Å) that is equal in length to the shortest As–S bond distance previously reported.

Data Collection and Reduction

Triethylammonium phenylthioarsenate, $(C_2H_5)_3NH^+(C_6H_5)AsSO(OH)^-$, was obtained from the reaction of $As_2S_3(C_6H_5)_2$ with an excess of moist triethylamine. The reaction was carried out over a period of 48 hr in benzene under reflux. *Anal.* Calcd for $(C_2H_5)_3NH^+(C_6H_5)AsSO(OH)^-$: As, 23.46; S, 10.04; N, 4.39; C, 45.13; H, 6.96. Found: As, 24.55; S, 10.52; N, 4.42; C, 45.34; H, 7.27; mp 114–115°. Crystals were obtained from dioxane solutions by slow cooling. Since the compound

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decomposes slowly in air, the crystal was sealed in a thin-walled Lindemann glass capillary. The needle-shaped crystal used for data collection was 0.10 × 0.14 × 0.60 mm (all ±0.01 mm) in the (100), (010), and (001) directions. The crystal was mounted with c approximately parallel to the ϕ axis.

The diffractometer used was a GE XRD-5 manually operated quarter-circle system, with Mo Kα (λ 0.71069 Å) radiation filtered with 1-mil zirconium foil. A scintillation counter was used with a pulse height analyzer set for a 90% window. Least-squares refinement[8] of the setting angles of 16 carefully centered reflections gave, at 23°, a = 11.807 (1) Å, b = 10.558 (1) Å, c = 8.788 (1) Å, α = 116.21 (2)°, β = 98.95 (1°), and γ = 117.91 (1)°. The crystal density, measured by suspension in a mixture of 1-bromobutane and 1-iodopropane, is 1.40 (1) g cm^{-3}, which compares to 1.373 (3) g cm^{-3} calculated for two formula units per unit cell. Space group $P\bar{1}$ imposes no crystallographic symmetry on the ions of the structure.

Absorption corrections were neglected. Approximation of the crystal shape as cylindrical makes $\mu r_{\max}$ = 0.17 and absorption correction factors range from 1.32 to 1.30. No corrections were made for extinction.

For intensity data collection a θ-2θ scan of 1.5° (2θ < 30°) or 2.0° (2θ > 30°) in 2θ was made at a speed of 2°/min with a take-off angle of 3°. Stationary-crystal, stationary-counter background readings were taken for 10 sec at each end of the scans. All reflections with $0 < 2\theta < 45°$ were examined (2203). Only 362 reflections had $I \geqslant 2\sigma(I)$; these were used for the solution and refinement of the structure. Four reflections were measured periodically during the 7-day data collection as a monitor of crystal and electronic stability. A linear decrease in the intensity of all four reflections (maximum of 38% decrease or about 6σ) led to a linear correction based on total elapsed time.

The standard deviations for I were calculated by $\sigma(I) = (S + BT^2 + 0.0016I^2)^{1/2}$ (where S = total scan count, B = sum of the background counts, T = scan time/total background time, and $I = S - BT$), except for a small number of reflections for which the two background readings differed by more than twice the calculated σ due to tailing of the reflection peak. For the latter reflections the standard deviations were set equal to the deviation of the background readings from the mean background value.

Scattering factors for neutral atoms were calculated from the analytical functions of Cromer and Weber,[9a] and real and imaginary anomalous dispersion corrections were made for As and S using the values given by Cromer.[9b]

Solution and Refinement of the Structure[10]

The arsenic atom was located in the unit cell from analysis of a sharpened Patterson map, and the other atoms were located on subsequent Fourier maps. Because of the modest parameter: reflection ratio, it was decided that both unconstrained refinement of the atoms of the phenyl ring and refinement with anisotropic thermal parameters for the two heavy atoms should not be attempted. The drop in R upon changing from a refinement with a constrained phenyl ring and all isotropic thermal factors to a refinement with a constrained phenyl group and anisotropic thermal factors for As and S was highly significant.[11] The change in R upon changing from constrained phenyl—all isotropic to independent phenyl atoms—all isotropic was much less significant, and when the latter change was made, the carbon atoms shifted by less than 2σ from their rigid-group positions. Consequently, in the final refinements the carbons and hydrogens of the phenyl group were constrained to the parameters given in Table I; the isotropic thermal parameters for the phenyl carbons were refined, while those of the phenyl hydrogen atoms were held at B = 5.0 Å.

In the least-squares refinement, the function minimized was $\Sigma w(|F_o| - |F_c|)^2$ where $w = 1/\sigma(F) \approx ((I_o + \sigma(I_o))^{1/2} - F_o)^{-2}$, and the R factors are defined as $R_1 = \Sigma||F_o| - |F_c||\Sigma/|F_o|$ and $R_2 = (\Sigma w||F_o| - |F_c||^2/\Sigma w F_o^2)^{1/2}$. The final value of R_1 was 0.051 and R_2 was 0.047. In the final cycle of refinement no positional parameter was shifted more than 0.10 times its estimated standard deviation. The standard deviation of an observation of unit weight is 1.02, and no $||F_o| - |F_c||$ values were more than $4\sigma(F_o)$. A final difference map did not have any peaks which were chemically interpretable; the highest peaks were 10% of the value of a carbon peak on an electron density map. The ethyl group hydrogen atoms were neither located nor assigned.

TABLE I

SELECTED DISTANCES AND ANGLES IN $(C_2H_5)_3NH^+(C_6H_5)AsSO(OH)^-$

Atoms	Distance, Å	Atoms	Distance, Å
As–S	2.08 (2)	N–C(7)	1.57 (5)
As–O(1)	1.65 (2)	N–C(9)	1.59 (5)
As–O(2)	1.75 (3)	N–C(11)	1.62 (5)
As–C(1)	1.90 (2)	C(7)–C(8)	1.54 (6)
C–C (phenyl)[a]	1.394	C(9)–C(10)	1.55 (6)
C–H (phenyl)[a]	1.084	C(11)–C(12)	1.51 (7)
		O(1)···N	2.64 (4)

Atoms	Angle, deg	Atoms	Angle, deg
S–As–O(1)	116.9 (9)	C(7)–N–C(9)	113 (2)
S–As–O(2)	108.6 (9)	C(7)–N–C(11)	107 (2)
S–As–C(1)	113.6 (6)	C(9)–N–C(11)	115 (3)
O(1)–As–C(1)	106 (1)	N–C(7)–C(8)	114 (3)
O(1)–As–O(2)	108 (1)	N–C(9)–C(10)	109 (3)
O(2)–As–C(1)	103 (1)	N–C(11)–C(12)	109 (4)
C–C–C (phenyl)[a]	120	C–C–H (phenyl)[a]	120

[a] Constrained as a rigid group in the refinement (see text).

Selected intramolecular distances and angles are listed in Table I. Figure 1 is an ORTEP drawing of the anion, and Figure 2 shows the crystal packing.

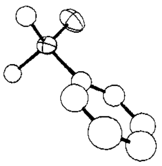

Figure 1.—ORTEP drawing of the $(C_6H_5)AsSO(OH)^-$ anion.

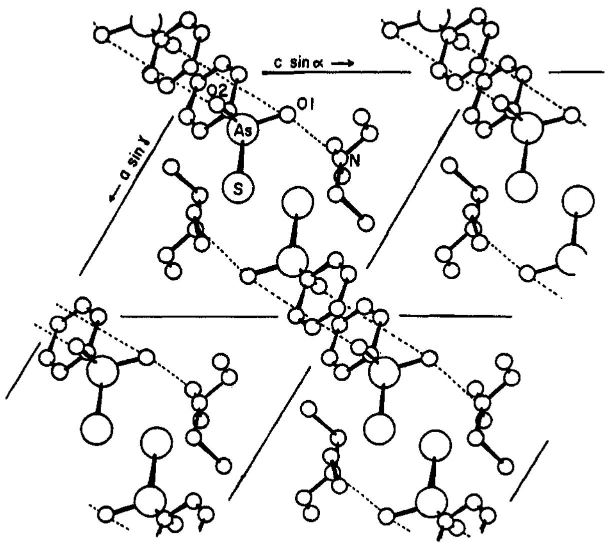

Figure 2.—Packing diagram of the crystal structure of $(C_2H_5)_3NH^+(C_6H_5)AsSO(OH)^-$, as viewed down the b axis.

The final positional and thermal parameters are given in Tables II and III.

Discussion

The crystal structure consists of $(C_6H_5)AsSO(OH)^-$ anions forming "dimers" across a crystallographic

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(10) A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Single copies may be obtained from the Business Operations Office, Books and Journals Division, American Chemical Society, 1155 Sixteenth St., N.W., Washington, D. C. 20036, by referring to author, title of article, volume, and page number. Remit check or money order for $3.00 for photocopy or $2.00 for microfiche.

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TABLE II[a]

FINAL ATOMIC POSITIONAL AND ISOTROPIC THERMAL PARAMETERS FOR $(C_2H_5)_3NH^+(C_6H_5)AsSO(OH)^-$

Atom	*x*	*y*	*z*	*B*, Å^2
As	0.2293 (5)	0.0615 (5)	0.0743 (5)	. . .
S	0.470 (1)	0.227 (1)	0.199 (1)	. . .
O(1)	0.173 (2)	0.062 (2)	0.216 (3)	3.7 (5)
O(2)	0.123 (2)	0.094 (2)	−0.108 (3)	3.8 (5)
C(1)[b]	0.147 (4)	−0.155 (4)	−0.056 (4)	3.6 (8)
C(2)[b]	0.227 (4)	−0.184 (4)	−0.116 (4)	4.6 (9)
C(3)[b]	0.167 (4)	−0.342 (4)	−0.216 (4)	5.5 (10)
C(4)[b]	0.027 (4)	−0.471 (4)	−0.257 (4)	6.6 (10)
C(5)[b]	−0.054 (4)	−0.441 (4)	−0.197 (4)	8.6 (13)
C(6)[b]	0.006 (4)	−0.283 (4)	−0.097 (4)	6.4 (10)
H(1)[b]	0.337	−0.084	−0.084	5
H(2)[b]	0.230	−0.365	−0.262	5
H(3)[b]	−0.020	−0.593	−0.335	5
H(4)[b]	−0.164	−0.541	−0.229	5
H(5)[b]	−0.057	−0.261	−0.051	5
N	0.358 (3)	0.201 (3)	0.592 (3)	3.7 (6)
C(7)	0.500 (4)	0.379 (3)	0.666 (4)	4.0 (8)
C(8)	0.623 (4)	0.474 (4)	0.885 (5)	6.0 (9)
C(9)	0.252 (4)	0.190 (4)	0.661 (5)	5.8 (10)
C(10)	0.185 (4)	0.282 (4)	0.591 (4)	6.0 (10)
C(11)	0.433 (4)	0.106 (4)	0.648 (5)	7.2 (11)
C(12)	0.302 (4)	−0.073 (4)	0.531 (5)	8.7 (12)

[a] In all tables the numbers in parentheses are the estimated standard deviations in the least significant digits unless otherwise noted. [b] Constrained as a rigid group in the refinement (see text).

TABLE III

FINAL ANISOTROPIC THERMAL PARAMETERS[a] AND ROOT-MEAN-SQUARE VIBRATION AMPLITUDES OF As AND S IN $(C_2H_5)_3NH^+(C_6H_5)AsSO(OH)^-$

							Rms Amplitudes, Å		
Atom	$10^4\beta(1,1)$	$10^4\beta(2,2)$	$10^4\beta(3,3)$	$10^4\beta(1,2)$	$10^4\beta(1,3)$	$10^4\beta(2,3)$	Min	Intermed	Max
As	126 (9)	169 (9)	192 (12)	79 (8)	111 (8)	81 (8)	0.18	0.22	0.25
S	159 (26)	294 (29)	309 (40)	129 (25)	124 (25)	160 (29)	0.23	0.27	0.34

[a] The form of the anisotropic parameter is $\exp[-(h^2\beta(1,1) + k^2\beta(2,2) + l^2\beta(3,3) + 2hk\beta(1,2) + 2hl\beta(1,3) + 2kl\beta(2,3)$.

center of symmetry by means of two strong (O–O distance of 2.65 (3) Å) O(1)H···O(2) H bonds. One triethylammonium cation is H bonded to each anion *via* an N–H···O(1) linkage with an N–O distance of 2.64 (3) Å. The H-bond system thus associates two anions and two cations around the unit cell origins rather than forming infinite chains or layers of ions.

The weighted-average values and root-mean-square deviations for the N–C distances, C–C distances, C–N–C angles, and N–C–C angles of the triethylammonium ion are 1.59 (3) Å, 1.53 (2) Å, 112 (4)°, and 111 (3)° respectively. The thermal parameters of the methyl carbon atoms indicate a not-uncommon amount of motion in these groups. No special significance is given to any structural feature of the cation.

In the anion the As–C distance of 1.90 (2) Å is equal, within experimental error, to the values of 1.91 (3), 1.895, and 1.91 Å, found for the $As(C_6H_5)_4^+$ cation,[12] the $As(C_6H_5)_3OH^+$ cation,[13] and $As(C_6H_5)O(OH)_2$,[14] respectively. The As–O distances of 1.65 (2) and 1.75 (3) Å in $(C_6H_5)AsSO(OH)^-$ suggest O(2) is the protonated oxygen. These distances compare to the 1.70 (1) and 1.71 (1) Å As–O distances found for the $As(C_6H_5)_3OH^+$ cation[13] and the range of 1.59–1.71 (2) Å As–O distances found in H_3AsO_4[15] and arsenate structures.[16] The two As–O bond distances of $(C_6H_5)AsSO(OH)^-$ correspond to As–O bond orders of 1.35 and 1.0 using the correlation suggested by Worzala.[15]

Table IV compares the As–S bond distances of this structure to other As–S bond distances which have been reported. Since the As–S bond distance of $(C_6H_5)AsSO(OH)^-$ is considerably shorter than any of the other values on Table IV except for $(Me_2AsS)_2$ and no bond lengths for species such as AsS or AsS^+ have been reported, the significance of the short As–S distance with respect to the As–S ring bonds is best analyzed by comparison with analogous P–S distances for which more data are available. The phosphorus sulfides (P_4S_3,[17] P_3S_5,[18] P_4S_7[19], P_4S_9,[20] and P_4S_{10}[21]) possess two kinds of P–S bonds; those to bridging S atoms (2.09 (2) Å) and those to terminal S atoms (1.95 (1) Å). There is a further shortening of about 0.10 Å for the P to terminal S bond when the phosphorus is bonded to more electronegative elements: 1.85 (2) Å in $PSCl_3$,[22] 1.87 (3) Å in PSF_3,[22] and 1.85 (2) Å in $P_4O_6S_4$.[23] Thus there is a 11% reduction in bond length on going from a P to bridging S bond in the phosphorus sulfides to a P to terminal S bond in which the phosphorus is bonded to three electronegative elements. This compares to the 8% reduction in bond length for the As–S bonds of the arsenic ring compounds in Table IV, all of which involve bridging S atoms, to the As–S bond of $(C_6H_5)AsSO(OH)^-$ in which only two of the three non-S bonds are to electronegative elements. It therefore appears that the 2.08 (2) Å As–S bond distance of the title compound is, insofar as it parallels P–S values, consistent with the other reported As–S bond distances.

TABLE IV

As–S BOND DISTANCES (Å)

Compound	As–S distance
$(C_6H_5)AsSO(OH)^-$	2.08 (2)
$(Me_2AsS)_2$[a]	2.075 (7), 2.214 (7), 2.279 (7)
As_4S_3,[b] As_4S_4,[c] As_4S_6[d]	2.21–2.25 (2)
$As_4S_4(C_6H_5)_4$[e]	2.25 (1)
$As_2S_3(C_6H_5)_2$[e]	2.243 (5)
$As(S_2CNEt_2)_3$[f]	2.35, 2.9
$C_6H_5As(S_2CNEt_2)_2$[g]	2.33 (1), 2.87 (4)
Ag_3AsS_3[h]	2.25
$(NH_4)_3AsS_4$[i]	2.20–2.26

[a] Reference 1. [b] Reference 2. [c] Reference 3. [d] Reference 4. [e] A. W. Cordes, R. Shores, and P. Gwinup, unpublished results. [f] Reference 6. [g] Reference 7. [h] D. Harker, *J. Chem. Phys.*, **4**, 381 (1936). [i] Reference 5.

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Reactions of Eight-Coordinate Metal Cyanide Complexes. III.[1,2] Synthesis and Structure of Iodohexakis(*tert*-butyl isocyanide)molybdenum(II) Iodide

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The compound of empirical formula $Mo(CNR)_6I_2$, R = *tert*-butyl, has been isolated from the reaction products of silver octacyanomolybdate(IV) and *tert*-butyl iodide. A single-crystal X-ray diffraction study of the red-orange, crystalline material revealed the correct structural formula to be $[Mo(CNR)_6I]I$. In the cation, the coordination geometry of the molybdenum atom is that of a monocapped trigonal prism, with the carbon atoms at the vertices and an iodine atom above one of the square faces. The average Mo–C distances are 2.06 (2) Å trans to the iodine atom and 2.12 (2) Å cis to the iodine atom. The Mo–I distance is 2.862 (3) Å. Steric crowding by the iodine atom may be judged by the average interligand distances and from the distortions of the trigonal prism defined by the isocyanide ligands. The *tert*-butyl groups are disordered in the lattice. The compound crystallizes in the orthorhombic system, with $a = 22.437$ (9) Å, $b = 22.336$ (9) Å, $c = 16.377$ (7) Å, ρ_{calcd} (for $Z = 8$) = 1.374 g/cm³, ρ_{obsd} = 1.38 g/cm³, and space group *Acam*. The molecule has crystallographically imposed mirror symmetry. Full-matrix, anisotropic refinement of all atoms except the *tert*-butyl carbon atoms, which were assigned isotropic thermal parameters, converged at $R_1 = 0.068$ for 1408 independent, observed reflections.

Introduction

Eight-coordinate complexes of general formula MX_4Y_4, where the π-donor and π-acceptor properties of the ligands X and Y are substantially different, are of interest for the reasons discussed previously.[1,3,4] Compounds originally formulated as $K_4[M(CN)_4(OH)_4]\cdot 4H_2O$ (M = Mo, W) have been shown by chemical, spectroscopic, and X-ray diffraction studies to be $K_4[MO_2(CN)_4]\cdot 6H_2O$[1,5] or $K_3Na[MoO_2(CN)_4]\cdot 6H_2O$[6] and therefore do not belong to the MX_4Y_4 class. Subsequently, we have turned our attention to the molecules $M(CN)_4(CNR)_4$ (M = Mo,[7] W[7,8]) which reportedly may be obtained from the reaction between alkyl halides and the silver salt of the appropriate octacyanometalate(IV), eq 1. The tetracyanotetrakis(alkyl isocyanide)molybdenum(IV) complexes are not particularly well characterized.[9] Recently, however, we have been able to prepare crystalline $Mo(CN)_4(CNR)_4$ (R = CH_3, n-C_3H_7, *tert*-C_4H_9) through a modification of the published synthetic procedure,[7] and the crystal structure of $Mo(CN)_4(CNCH_3)_4$ has been determined.[10] In the course of this study, a new material of empirical formula $Mo(CNR)_6I_2$ (R = *tert*-C_4H_9) was isolated in a redox reaction which *tentatively* may be represented by eq 2. The molybdenum(II) isocyanide complex was obtained as beautiful red-orange prisms, and its solid-state structure has been elucidated in a single-crystal X-ray diffraction investigation. While this structure determination was in progress, the synthesis and characterization of the closely related molybdenum(II) isocyanide complexes $Mo(CNR)_5X_2$ (X = Cl, Br) was reported, and the presence of seven-coordinate molybdenum(II) was suggested.[11] Our study reinforces the conclusions drawn by these authors and provides much needed structural information about seven-coordinate complexes of MX_6Y type, where X and Y are both monodentate ligands.[12]

$$Ag_4[M(CN)_8] + 4RX \longrightarrow M(CN)_4(CNR)_4 + 4AgX \quad (1)$$

$$R = CH_3,\ C_2H_5,\ n\text{-}C_3H_7,\ i\text{-}C_3H_7,\ tert\text{-}C_4H_9,\ \text{etc.}$$

$$M = Mo, W$$

$$Ag_4Mo(CN)_8 + 6RI \longrightarrow [Mo(CNR)_6I]I + 2AgCN + 2AgI + I_2 \quad (2)$$

Experimental Procedure and Results

Collection and Reduction of X-Ray Data.—The compound was prepared by shaking a suspension of silver octacyanomolybdate(IV) in *tert*-butyl iodide for 6 days, extracting the residue with methanol, and crystallizing by slow evaporation from methanol after chromatography on alumina. This synthesis has been reproduced only with great difficulty, and futher studies are in progress. *Anal.* Calcd for $C_{30}H_{54}N_6I_2Mo$: C, 42.46; H, 6.41; N, 9.90. Found (by Galbraith Laboratories, Knoxville, Tenn.): C, 43.0; H, 6.44; N, 9.39.

A rectangular prism of approximate dimensions 0.1 × 0.1 × 0.3 mm was selected from the small crop of red-orange crystals and mounted with clear nail polish on the end of a glass fiber. The mounting axis was the longest of the three and parallel to *c*. The crystal, which has been kept in air for over 2 years without any visible signs of decomposition, was used in all subsequent work reported here. The space group and approximate unit cell parameters were determined using Ni-filtered Cu Kα radiation

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