2.89 (s, 3, CH_3CN), 3.07 (s, 3, CH_3CO), 9.43 (br s, 1, NH). Anal. ($C_8H_7NBO_5Cl_2Re$) C, H, N.

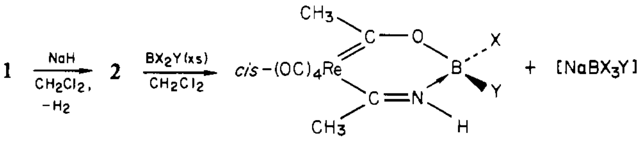
[*cis*-$(OC)_4Re(CH_3CO)(CH_3CNH)$]$BBr_2$ (4): bright yellow crystals (0.175 g, 40%); mp 117–118 °C; IR (CH_2Cl_2) ν(CO) 2100 (m), 2000 (vs), 1995 (vs), 1970 (s), $\nu(C\overset{..}{-}O, C\overset{..}{-}N)$ 1540 (br, m); ^1H NMR δ 2.92 (s, 3, CH_3CN), 3.10 (s, 3, CH_3CO), 9.57 (br s, 1, NH). Anal. ($C_8H_7NO_5BBr_2Re$) C, H, N.

[*cis*-$(OC)_4Re(CH_3CO)(CH_3CNH)$]$BI_2$ (5): dark yellow powder; IR (CH_2Cl_2) ν(CO) 2100 (m), 1995 (br, vs, 1970 (s); ^1H NMR δ 3.01 (s, 3, CH_3CN), 3.25 (s, 3, CH_3CO), 9.91 (br s, 1, NH).

[*cis*-$(OC)_4Re(CH_3CO)(CH_3CNH)$]B(Cl)(Ph) (6): colorless crystals (0.125 g, 31%); mp 155–158 °C; IR (CH_2Cl_2) ν(CO) 2100 (m), 2000 (vs), 1980 (br, s); ^1H NMR δ 2.85 (s, 3, CH_3CN), 3.10 (s, 3, CH_3CO), 7.35 (complex m, 5, C_6H_5), 9.51 (br s, 1, NH). Anal. ($C_{14}H_{12}NO_5BClRe$) C, H, N.

Results and Discussion

When the rhenaacetylacetonimine complex, **1**, is treated with NaH in CH_2Cl_2 solution, the rhenaacetylacetone iminato anion, **2**, is formed. Reaction of **2** with BCl_3, BBr_3, BI_3, or $PhBCl_2$ affords the (rhenaacetylacetone iminato)B (X)(Y) complexes **3–6**. Complexes **3–6** dissolve in benzene, and the mass spectrum of **3** shows a parent ion, principal fragmentation ions, and the correct isotopic pattern consistent with a neutral, monomeric structure, as shown.



3, X = Y = Cl
4, X = Y = Br
5, X = Y = I
6, X = Cl, Y = Ph

Solution-phase IR spectra of **3–6** show terminal carbonyl stretching bands at the frequencies expected for a neutral complex with a relative intensity pattern which is consistent with a *cis*-$(OC)_4Re$ fragment. The ν(CO) frequencies of **3–6** are ca. 20 cm^{-1} lower in energy than those of the corresponding [*cis*-$(OC)_4Re(CH_3CO)_2$]BX_2 complexes.[1] This shift apparently reflects a more electron-rich rhena ligand when an NH group is substituted for an oxygen atom. The $\nu(C\overset{..}{-}O, C\overset{..}{-}N)$ stretching frequencies at ca. 1540 cm^{-1} are 15 cm^{-1} lower in energy than the corresponding band of **1**. This shift may result from a slight decrease in the intra chelate ring C=O and C=N bond orders upon coordination to the boron moieties.

The ^1H NMR spectra show N–H resonances in the range of 9.43–9.91 ppm, which indicates that the N–H hydrogen atom and the iminium–ligand methyl group are syn relative to the $C\overset{..}{-}N$ multiple bond.[4–6] An unusual feature in each of these spectra is that the iminium methyl resonance appears at *higher* field than the acetyl methyl resonance. In complexes **3–6**, the iminium methyl resonances appear in the range 2.85–3.01 ppm, while the acetylmethyl resonances appear in the range of 3.07–3.25 ppm. The average anisochronism[9] for these two methyl resonances is 21 Hz. In complex **1**, the iminium and acetyl methyl resonances appear at 2.86 ppm and 2.68 ppm, respectively. However, in the symmetrical [*cis*-$(OC)_4Re(CH_3CO)_2$]B(X)(Y) complexes, which are analogous to **3–6**, the acetyl methyl resonances appear in the range of 3.09–3.18 ppm. Therefore, in the comlexes **3–6**, the iminium methyl resonances have chemical shifts similar to that of the iminium methyl resonance of **1** (i.e., as if the NH group were protonated), and the acetyl methyl resonances have chemical shifts similar to those of the acetyl groups in (rhenaacetylacetonato)B(X)(Y) complexes [i.e., where the acetyl ligand is coordinated to a B(X)(Y) moiety]. These chemical shift data for complexes **3–6** are entirely consistent with the proposed structure where *both* the oxygen and the nitrogen atoms of the rhenaacetylacetone iminato ligand are coordinated to the boron atom. Unfortunately, single crystalline samples of these complexes have not been isolated yet to permit an X-ray structural determination.

Complexes **3–6** are rhena analogues to the known neutral (2-pentanone-4-iminato)BZ_2 complexes (or related derivatives) where Z can be alkyl, aryl, or F.[10,11] Interestingly, anion **2** does not react with BF_3 to afford a (rhenaacetylacetone iminato)BF_2 complex. Also, ionic boronium salts of the type [(rhenaacetylacetone iminato)$_2$B]$^+X^-$ are not observed. Complexes **3–6** decompose slowly in ether solutions. This effect is not observed with the (rhenaacetylacetonato)BX_2 complexes. However, organic (β-diketonato)BZ_2 complexes are, presumably, slightly more stable than the corresponding Schiff base complexes, also.[10]

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Registry No. **1**, 66808-78-4; **2**, 80719-67-1; **3**, 80720-91-8; **4**, 80720-92-9; **5**, 80720-93-0; **6**, 80720-94-1; BCl_3, 10294-34-5; BBr_3, 10294-33-4; $PhBCl_2$, 873-51-8; BI_3, 13517-10-7.

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Contribution from the Molecular Structure Center and
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Molecular Structure of $Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)_3$, a Compound with a Dangling Dithiophosphate Ligand

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The structure of $Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)_3$, a compound prepared[1] by the oxidation of $Mo(CO)_4Cl_2$ by *p*-tolyl azide in the presence of $(NH_4)S_2P(OC_2H_5)_2$, is important because it is a d^1 complex with a potential coordination number of 7 and a potential 19-electron configuration. No precedents exist. Additional importance arises because this compound is the precursor[1] of an unusual tetranuclear cluster, [$Mo(NC_6H_4CH_3)(\mu_3\text{-}S)(S_2P(OC_2H_5)_2)$]$_4$.[2] Although we have considered several possible structures for the mononuclear compound, accumulated ESR, infrared, and chemical evidence led us to a bias in favor of a cis octahedral structure with a linear arylimido ligand, two bidentate dithiophosphate ligands, and a third monodentate dithiophosphate ligand.[1] Severe twinning of the crystals from the original preparation prevented a meaningful determination of the structure at that time. However, recrystallization from a different mixture of solvents has provided the crystals which were used in the study described herein.

Experimental Section

Orange-brown crystals of $Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)_3$ were obtained by recrystallization from benzene/petroleum ether. Although the compound has a bronze color when it is recrystallized from CH_2Cl_2/CH_3OH,[1] the infrared and ESR spectra of both samples are

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0020-1669/82/1321-2101$01.25/0

Table I. Crystal Data

space group	$P2_1/c$	crystal size, mm	0.12 × 0.28 × 0.24
temp, °C	−161	μ (Mo Kα), cm^{-1}	9.412
a, Å	20.421 (12)	min absorption	0.804
b, Å	8.579 (4)	max absorption	0.905
c, Å	18.449 (11)	no. of unique data	4190
β, deg	93.92 (3)	no. of data with $F > 3\sigma(F)$	3552
V, Å^3	3224.8 (24)		
d_{calcd}, g/cm^3	1.410	no. of variables	473
		$R(F)$	0.038
Z	4	$R_w(F)$	0.039
fw	684.71	largest Δ/σ	0.05

Table II. Fractional Coordinates

atom	x^a	y	z
Mo(1)	7032.4 (2)	4758.7 (5)	1387.4 (2)
S(2)	6143 (1)	5166 (1)	2258 (1)
S(3)	6712 (1)	7503 (1)	1109 (1)
P(4)	6149 (1)	7408 (2)	1958 (1)
O(5)	5442 (2)	8112 (4)	1821 (2)
C(6)	4982 (3)	7400 (7)	1267 (3)
C(7)	4431 (3)	6649 (9)	1634 (4)
O(8)	6404 (2)	8542 (4)	2584 (2)
C(9)	7099 (3)	8487 (7)	2836 (3)
C(10)	7195 (3)	9418 (8)	3512 (3)
S(11)	7691 (1)	4864 (1)	325 (1)
S(12)	9050 (1)	6262 (2)	−255 (1)
P(13)	8587 (1)	5808 (2)	604 (1)
O(14)	8936 (2)	4612 (4)	1158 (2)
C(15)	9604 (3)	4931 (7)	1463 (3)
C(16)	9835 (3)	3546 (8)	1887 (4)
O(17)	8528 (2)	7261 (4)	1134 (2)
C(18)	8383 (3)	8839 (6)	872 (3)
C(19)	8713 (4)	9974 (8)	1389 (4)
S(20)	6108 (1)	4155 (1)	351 (1)
S(21)	6891 (1)	1883 (1)	1483 (1)
P(22)	6225 (1)	1929 (1)	626 (1)
O(23)	5567 (2)	1068 (4)	774 (2)
C(24)	5110 (3)	1765 (7)	1264 (3)
C(25)	4516 (3)	2343 (8)	834 (3)
O(26)	6429 (2)	845 (4)	−11 (2)
C(27)	7054 (3)	1056 (7)	−333 (3)
C(28)	7180 (3)	−377 (8)	−763 (3)
N(29)	7669 (2)	4704 (4)	2057 (2)
C(30)	8126 (2)	4373 (6)	2633 (3)
C(31)	8535 (2)	5534 (6)	2927 (3)
C(32)	8945 (2)	5208 (6)	3536 (3)
C(33)	8963 (2)	3758 (6)	3864 (3)
C(34)	8558 (3)	2590 (7)	3557 (3)
C(35)	8145 (3)	2895 (6)	2943 (3)
C(36)	9390 (4)	3437 (9)	4539 (4)

[a] Fractional coordinates are $\times 10^4$.

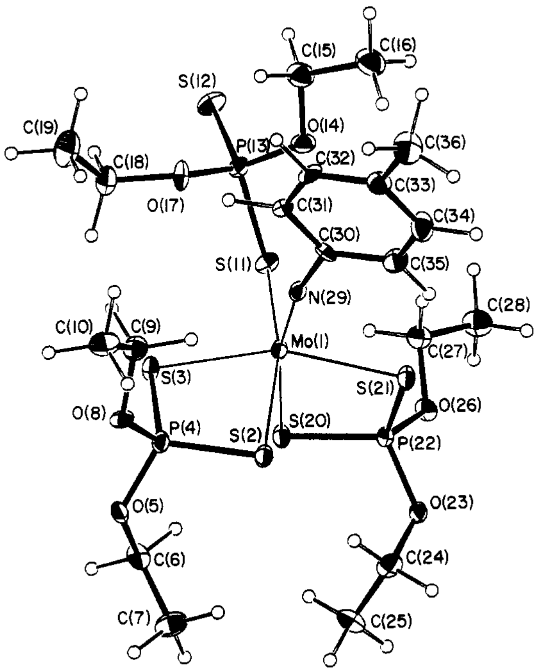

Figure 1. ORTEP drawing of $Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)_3$. Thermal ellipsoids are drawn at 50% probability. Hydrogen atoms are represented by spheres corresponding to $B_{\text{iso}} = 0.5$ Å^2.

Table III. Selected Bond Distances (Å)

Mo(1)–S(2)	2.530 (2)	S(2)–P(4)	2.001 (2)
Mo(1)–S(3)	2.488 (2)	S(3)–P(4)	2.008 (2)
Mo(1)–S(11)	2.453 (2)	S(11)–P(13)	2.035 (2)
Mo(1)–S(20)	2.645 (2)	S(12)–P(13)	1.940 (2)
Mo(1)–S(21)	2.492 (2)	S(20)–P(22)	1.986 (2)
Mo(1)–N(29)	1.732 (4)	S(21)–P(22)	2.014 (2)

Table IV. Selected Bond Angles (Deg)

S(2)–Mo(1)–S(3)	79.1 (0)	S(20)–Mo(1)–S(21)	77.1 (0)
S(2)–Mo(1)–S(11)	163.5 (0)	S(20)–Mo(1)–N(29)	167.0 (1)
S(2)–Mo(1)–S(20)	88.8 (1)	S(21)–Mo(1)–N(29)	90.4 (1)
S(2)–Mo(1)–S(21)	90.0 (0)	Mo(1)–S(2)–P(4)	86.5 (1)
S(2)–Mo(1)–N(29)	95.0 (1)	Mo(1)–S(3)–P(4)	87.5 (1)
S(3)–Mo(1)–S(11)	87.2 (0)	Mo(1)–S(11)–P(13)	110.2 (1)
S(3)–Mo(1)–S(20)	82.6 (0)	Mo(1)–S(20)–P(22)	86.4 (1)
S(3)–Mo(1)–S(21)	157.2 (0)	Mo(1)–S(21)–P(22)	90.0 (1)
S(3)–Mo(1)–N(29)	110.3 (1)	S(2)–P(4)–S(3)	105.6 (1)
S(11)–Mo(1)–S(20)	80.4 (1)	S(11)–P(13)–S(12)	110.7 (1)
S(11)–Mo(1)–S(21)	99.5 (0)	S(20)–P(22)–S(21)	106.4 (1)
S(11)–Mo(1)–N(29)	98.4 (0)	Mo(1)–N(29)–C(30)	168.4 (4)

identical. Moreover, a bronze powder occurs when the orange-brown crystals are crushed.

The Picker goniostat, Furnas monochromator, and low-temperature cooling equipment used in the crystallographic study have been described previously.[3] Data were collected at −161 °C with a standard θ–2θ continuous-scan technique for $\pm h,+k,\pm l$ in the range of $50° \leq 2\theta \leq 6°$ with graphite-monochromatized Mo K-α radiation (λ = 0.71069 Å). The raw data were corrected for absorption, Lorentz, and polarization effects and averaged to yield a unique set of structure amplitudes and estimated standard deviations.[3]

The structure was solved by direct methods and refined by isotropic full-matrix least squares. A difference Fourier synthesis phased on the refined nonhydrogen atoms clearly located all hydrogen atoms. Final refinement included anisotropic thermal parameters for all nonhydrogen atoms, isotropic thermal parameters for hydrogen atoms, an overall scale factor, and positional parameters for all atoms. Crystal and diffractometer data are given in Table I, and final positional parameters for nonhydrogen atoms are listed in Table II. Observed and calculated structure amplitudes, anisotropic thermal parameters, hydrogen positional and thermal parameters, and a complete listing of bond distances and angles are available as supplementary material.

Results and Discussion

The location and refinement of all of the hydrogen atoms in the structure of $Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)_3$ provides ample evidence that the structure is well determined. The molecular structure, shown in Figure 1, is based on a distorted octahedron with the near-linear arylimido ligand immediately adjacent to a monodentate dithiophosphate ligand. The remainder of the coordination sites are occupied by the donor atoms of bidentate dithiophosphate ligands.

Selected bond distances and angles for the immediate vicinity of the molybdenum atom are given in Tables III and IV, respectively. The Mo–N bond distances of 1.732 (4) Å and the nearly linear Mo–N–C(aryl) bond angle of 168.4 (4)° meet the criteria for a Mo≡N linkage which was established for other arylimido compounds of molybdenum.[4] The Mo–S

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distance for the bonded sulfur atom in the monodentate ligand is 2.453 (2) Å, which is somewhat shorter than the range of 2.488 (2)–2.530 (2) Å for the remainder of the sulfur atoms adjacent to the arylimido ligand. The corresponding distance for the sulfur atom trans to that ligand is, however, 2.645 (2) Å, which represents a substantial trans effect and the first observation of the trans influence of any imido ligand in compounds with a potential 19-electron configuration.[5] Finally, the distance between the molybdenum atom and the unbound sulfur atom of the monodentate ligand is 5.434 (2) Å while the distance between this sulfur atom and the molybdenum atom in the adjacent molecule is also greater than 5 Å. In general, there are no unusual nonbonded contacts in the structure.

Distances and angles within the arylimido and bidentate dithiophosphate ligands are in good agreement with those found in $Mo_2O_3(S_2P(OC_2H_5)_2)_4$,[6] $Mo_2O_3(NH)(S_2P(OC_2H_5)_2)_2$,[7] and $[Mo(NC_6H_4CH_3)(\mu_3\text{-}S)(S_2P(OC_2H_5)_2)]_4$.[2] The unique distances and angles associated with the PS_2 group in the monodentate dithiophosphate ligand can be seen, however, by comparing them to the corresponding parameters in the bidentate ligands. While the average value of the P–S distances in the bidentate ligands is 2.002 Å, these distances in the monodentate ligand are 2.035 (2) Å (for the sulfur atom bound to the metal) and 1.940 (2) Å (for the unbound sulfur atom). The latter corresponds well with the average value of 1.94 Å from 16 separately determined crystallographic distances which we have found in the literature[8] for $P{=}S$ bonds. The monodentate nature of this ligand has also allowed the S–P–S bond angle to open slightly to a value of 110.7 (1)° whereas the corresponding values in the bidentate ligands are 105.6 (1) and 106.4 (1)°.

The potential 19-electron configuration of $Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)_3$ and the stereochemical consequences of this configuration have been discussed elsewhere.[1] A brief synopsis with additional comments on similar compounds is appropriate, however. Predictions are based on an effective atomic number rule or, ultimately, the minimization of antibonding energies. A linear arylimido ligand is a 4-electron donor with a $Mo{\equiv}N$ linkage while a bent ligand is a 2-electron donor with a $Mo{=}N$ linkage.[9] The rule can be used, for example, to exclude ionic variants of $Mo(NC_6H_5)Cl_2(S_2CN(C_2H_5)_2)_2$ and to predict one bent and one linear arylimido ligand in $Mo(NC_6H_5)_2(S_2CN(C_2H_5)_2)_2$. These predictions are in accord with the observed structures.[4,9]

Results which are more pertinent to $Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)_3$ are also available. The 18-electron configuration in $Nb(NC_6H_4CH_3)(S_2CN(C_2H_5)_2)_3$ suggests a structure with a coordination number of 7 and a linear arylimido ligand. The actual structure has been shown[10] to be a pentagonal bipyramid with an apical arylimido ligand which is nearly linear. In contrast, $Re(NC_6H_4CH_3)(S_2CN(CH_3)_2)_3$, a compound with a potential 20-electron configuration, can achieve the favored 18-electron configuration either by adopting a coordination of 7 with a bent arylimido ligand or by adopting a coordination number of 6 with a linear arylimido ligand and a monodentate dithiocarbamate ligand. Unambiguous NMR evidence[11] indicates the presence of one monodentate dithiocarbamate ligand and, therefore, a coordination number of 6. Identical structural options are open to $Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)_3$ because of its potential 19-electron configuration, and the 6-coordinate compound is again found as shown herein.

The coordination geometries of $MoO_2(S_2CN(C_3H_7)_2)_2$,[12] $MoOCl_2(S_2CN(C_2H_5)_2)_2$,[13] and $NbO(S_2CN(C_2H_5)_2)_3$[14] mimic those of the arylimido compounds described above because the oxo ligand can also serve as a 2- or a 4-electron donor. Since $MoO(S_2CN(C_2H_5)_2)_3{}^+$ is a pentagonal bipyramid with an apical oxo ligand,[15] it is probably safe to infer the same geometry for $Mo(NC_6H_5)(S_2CN(C_2H_5)_2)_3{}^+$.[16] If so, the additional configurational electron in $Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)_3$ had remarkable structural consequences.

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Registry No. $Mo(NC_6H_4CH_3)(S_2P(OC_2H_5)_2)_3$, 72967-91-0.

Supplementary Material Available: Listings of observed and calculated structure amplitudes, anisotropic thermal parameters, hydrogen positional and thermal parameters, and bond distances and angles (34 pages). Ordering information is given on any current masthead page.

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Synthesis and Characterization of the (4-Hydroxyphenyl phosphato)pentaamminecobalt(III) Ion

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Internal ligand-to-metal electron transfer initiated by the action of an external oxidant on the ligand has received much less attention than the inner- and outer-sphere mechanistic types. Taube's 1970 review of induced electron-transfer reactions[1] cites only 15 references, and little additional work in this area has appeared since then. French and Taube's pioneering study of the action of Ce(IV) on (pyridinemethanol)pentaamminecobalt(III) ions[2] exposed many of the important mechanistic questions concerning the induced electron-transfer effect. More recently, highly efficient induced electron transfer has been reported in the Ce(IV) oxidation of $(NH_3)_5Co^{III}$ complexes of α-hydroxy acids.[3]

We have initiated studies of induced electron transfer linked to the interaction of one-electron oxidants with cobalt(III) complexes of hydroquinone esters. A semiquinone phosphate intermediate is generated in the two-electron oxidation of (4-hydroxyphenyl)phosphoric acid (HQ-*P*) to orthophosphate and 1,4-benzoquinone.[4] Coordinated benzosemiquinone

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