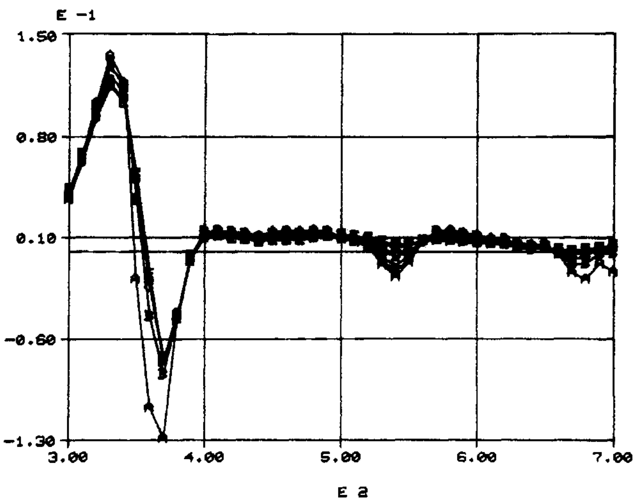

Figure 1. Transient difference spectra (absorbance vs. wavelength) for the laser pulse photolysis (Nd-YAG laser operating at 355 nm) of $Pt_2(\mu\text{-}P_2O_5H_2)_4^{4-}$ and C_6F_5Br in aqueous methanol. Time after pulse (μs): A = 0.1, B = 0.4, C = 1, D = 2.2, E = 6.5, F = 13.5.

or $1,2\text{-}C_2H_4Br_2$ gives, in each case, conversion to $Pt_2(\mu\text{-}P_2O_5H_2)_4Br^{4-}$ within 1 μs after the pulse (eq 2). The complex

$$Pt_2(\mu\text{-}P_2O_5H_2)_4^{4-*} + RBr \rightarrow Pt_2(\mu\text{-}P_2O_5H_2)_4Br^{4-} + R^{\bullet} \quad (2)$$

$Pt_2(\mu\text{-}P_2O_5H_2)_4Br^{4-}$ then disproportionates to $Pt_2(\mu\text{-}P_2O_5H_2)_4^{4-}$ and $Pt_2(\mu\text{-}P_2O_5H_2)_4Br_2^{4-}$ with a second-order rate constant $k_2 = 9.5 \times 10^8$ M^{-1} s^{-1} (eq 3).[8] In each case we observe a rapid rise

$$2Pt_2(\mu\text{-}P_2O_5H_2)_4Br^{4-} \xrightarrow{k_2} Pt_2(\mu\text{-}P_2O_5H_2)_4^{4-} + Pt_2(\mu\text{-}P_2O_5H_2)_4Br_2^{4-} \quad (3)$$

in the 340-nm absorption band of $Pt_2(\mu\text{-}P_2O_5H_2)_4Br^{4-}$ to maximum concentration within 1 μs, and we detect no growth in this absorption band subsequent to the laser pulse.

It is particularly significant that $Pt_2(\mu\text{-}P_2O_5H_2)_4Br^{4-}$ is formed rapidly in the reaction with C_6F_5Br. Saveant has shown that there is an approximate linear correlation between the rate constant for halide ion cleavage from the haloaromatic radical anion and the standard potential of the aromatic halide.[9] For bromobenzene ($E = -2.3$ V vs. SCE) we anticipate k_1 in reaction 4 to be approximately 10^8 s^{-1}, but for bromopentafluorobenzene ($E = -0.93$ V vs. SCE), we expect k_1 to have the much smaller value of 10^{-2} s^{-1}.[10,11] Since our laser photolyses are carried out in solutions which have $[C_6F_5Br] \approx 10^{-2}$ M, this rate constant k_1 of 10^{-2} s^{-1} is some 8-9 orders of magnitude too slow for the $S_{RN}1$ pathway to be operative. The reaction with C_6F_5Br, therefore, follows a pathway analogous to that proposed for the reaction of $Pt_2(\mu\text{-}P_2O_5H_2)_4^{4-*}$ with isopropyl alcohol.[3] Our results support a mechanism where the diradical triplet excited state abstracts a bromine atom directly from bromopentafluorobenzene.

$$ArBr^{-} \rightarrow Ar^{\bullet} + Br^{-} \quad (4)$$

For the case of C_6H_5Br and $1,2\text{-}C_2H_4Br_2$ the case is less clear. If, however, the $S_{RN}1$ pathway is followed, it is unlikely that diffusion outside the solvent cage occurs, especially since the final recombination step between $Pt_2(\mu\text{-}P_2O_5H_2)_4^{3-}$ and Br^- involves two negatively charged ions.

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Received June 25, 1986

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[8] As required for a second order decay, the half life of the reaction is dependent on $[Pt_2(\mu\text{-}P_2O_5H_2)_4Br]_0$.

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Discrete Metal-Sulfide-Thiolate Complexes of an Early 3d Transition Metal

Sir:

We have recently initiated a program directed toward the development of discrete vanadium-sulfide-thiolate chemistry. Such work is designed to provide soluble mechanistic and structural models for polymeric vanadium sulfides, which, together with the sulfides of the other group 5 metals Nb and Ta, display interesting magnetic and electrical properties.[1] Of additional and particular stimulus to our work is the involvement of vanadium sulfides in crude oil refining. These sulfides act both as poisons of the Mo/Co hydrodesulfurization (HDS) fixed-bed catalysts[2] and also, because of their own intrinsic activity,[3] as in situ generated HDS catalysts in a moving-bed system (the Aurabon process).[4] Both of the above are particularly topical as the requirement to process the heavier crudes, which contain large amounts of vanadium, becomes necessary with increasing depletion of lighter crudes. In addition, of course, such studies should provide interesting comparisons with both 4d Mo/S and 5d W/S chemistry, which have been extensively explored.[5,6] We herein report the preparation of some unusual and sulfur-rich products, which represent initial progress toward a better understanding of soluble V/S chemistry and which display features not seen in Mo or W chemistry.

Oxidation of V(III) by elemental sulfur in a reaction mixture comprising VCl_3:NaSPh:S:NEt_4Br in a molar ratio of 1:7:3:2 in MeCN[7] yielded an intensely dark green solution and some purple powder. After being stirred for 18 h, the reaction mixture was filtered and $Me_3NBzlCl$ (2 equiv) added to the filtrate. The solution was refiltered and ether added to initiate the crystallization of $(Me_3NBzl)_2[VS_2(S_2)(SPh)]$ (**1**). After overnight storage, black well-formed plates were collected by filtration and washed with MeCN/Et_2O (1:3).[8] Yields are typically 30-40%. (With product identity established, we have employed a V:S ratio of 1:4, attempting to improve the yield, but instead obtained a completely different product currently under investigation. Consequently, we continue to employ the 1:3 ratio, which gives adequate yields reproducibly.) The structure[9,10] of the anion of **1** is depicted in

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(6) Muller, A. *Polyhedron* **1986**, *5*, 323.

(7) All reactions and manipulations were performed under an inert atmosphere employing freshly distilled solvents.

(8) Complex **1** is soluble to any significant extent only in DMF in which it has limited stability. Anal. Calcd for $C_{26}H_{37}N_2S_5V$: C, 53.03; H, 6.33; N, 4.76; V, 8.65. Found: C, 53.60; H, 6.40; N, 4.85; V, 9.50.

(9) Structures were solved by a combination of direct methods (MULTAN) and Fourier techniques and refined by full-matrix least squares.

(10) Monoclinic space group $P2_1/n$, $a = 18.178$ (8) Å, $b = 8.843$ (2) Å, $c = 18.852$ (7) Å, $\beta = 107.06$ (2)°, and $Z = 4$, for 2807 unique data with $F > 3\sigma(F)$; non-hydrogen atoms were refined anisotropically, and hydrogen atoms were readily located and refined isotropically; final R (R_w) = 5.65 (5.63).

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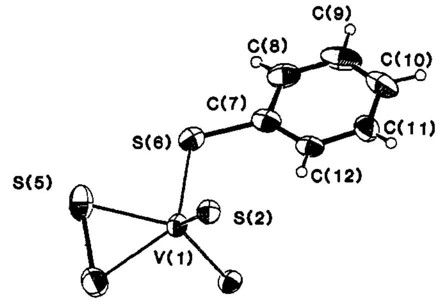

Figure 1. ORTEP projection of the anion of **1** showing the atom-labeling scheme. Non-hydrogen atoms are depicted as 50% probability ellipsoids; hydrogen atoms are depicted as spheres of arbitrary size. V–S(2) = 2.092 (2), V–S(3) = 2.355 (2), V–S(4) = 2.099 (2), V–S(5) = 2.323 (2), and V–S(6) = 2.367 (2) Å; S(2)–V–S(4) = 111.52 (8), S(2)–V–S(6) = 106.14 (8), S(4)–V–S(6) = 101.08 (8), S(3)–V–S(5) = 50.97 (8), and V–S(6)–C(7) = 111.67 (3)°.

Figure 1. The mononuclear vanadium(V) center is ligated by three types of sulfur, viz. one PhS^-, one persulfido (S_2^{2-}), and two sulfido (S^{2-}) groups, to yield a VS_5 core of idealized C_s symmetry. The coordination geometry is best described as tetrahedral with the S_2^{2-} considered as occupying one site. The S–S bond length in the latter (2.013 (3) Å) is similar to that in the V(IV) monomer $Cp_2V(S_2)$ (2.028 (4) Å);[11] such values are typical of this unit as a ligand and are consistent with π-donation from S_2^{2-} π^* orbitals into empty vanadium d orbitals (free S_2^{2-}; S–S = 2.13 Å).[5] The V=S(2) and V=S(4) distances are 2.092 (2) and 2.099 (2) Å, respectively, somewhat shorter than those in VS_4^{3-} (2.135–2.175 (1) Å),[12] the only other known vanadium(V) monomer with sulfide ligation. The vanadium(V)–thiolate bond in **1** is the first occurrence of such a linkage at this oxidation level, having been observed up to now only with the lower oxidation states in compounds such as $[VO(edt)_2]^{2-}$,[12] $Cp_2V(SPh)_2$,[13] $Cp_2V(SPh)$,[13] and $[V_2(edt)_4]^{2-}$ (edt = ethane-1,2-dithiolate).[14] The overall structure of **1** has no counterpart in isoelectronic Mo(VI) or W(VI) chemistry.

The variety of sulfur ligation around the vanadium(V) center of **1** suggested that it might display extensive reactivity chemistry. Reactions with elemental sulfur or selenium, CS_2, activated acetylenes, edt^{2-}, or monomeric vanadium complexes such as $[VS(SPh)_4]^{2-}$ and VS_4^{3-} [15] support this belief and yield soluble products currently being investigated. There is no doubt in our minds that **1** will prove to be a convenient and versatile synthetic reagent in higher oxidation state V/S chemistry.

Reaction of VCl_3:Na_2edt:S:NEt_4Br in a molar ratio of 1:2.5:2:2 in MeCN[7] followed by stirring for 24 h yielded an intense red-brown solution and some brown solid. The mixture was filtered and an equal volume of ether added to the filtrate to yield a black oil, which solidified on storage in a freezer. The product was collected by filtration, dissolved in MeCN, and refiltered, and the filtrate layered with Et_2O. This procedure yielded black microcrystalline $(NEt_4)_2Na[V_2S_5(edt)]$ (**2**) and very large purple crystals of $(NEt_4)_2Na[V_2OS_4(edt)]$ (**3**); the two materials are readily separable manually and are obtained in 30% and 20% yields, respectively, based on available vanadium. The structure[9,16] of the anion of **3** is shown in Figure 2. The two vanadium atoms are 2.977 (1) Å apart and bridged by two μ-S^{2-} atoms to yield a nonplanar $V_2S_2^{5+}$ core. A terminal edt^{2-} and multiply bonded O(3), S(10), and S(11) complete coordination to the metals to yield idealized C_s symmetry. The trianionic nature of the anion necessitates a mixed-valence V(IV)V(V) description. V(1) and V(2) are formally assigned as the V(IV) and V(V) centers, respectively, based on (i) the square-pyramidal geometry at V(1) characteristic of VO^{2+} species; (ii) the V=O bond length (1.626 (3) Å) identical with that in $[VO(edt)_2]^{2-}$ (1.625 (3) Å)[12], and (iii) the tetrahedral geometry at V(2) as seen in VS_4^{3-}.[17] With the identity of **3** established, we believe **2** to be isostructural with V(1)=S instead of V(1)=O. The separated complexes have identical IR spectra except for the presence of a band at 915 cm^{-1} in **3** assignable to a V=O stretch. Further evidence for the identity of **2** is available from electronic spectroscopy in MeCN solution; the spectra of **2** and **3** are very similar[18] except for a blue shift in the bands of the latter on conversion of V=S to V=O, behavior paralleling that seen in the $[VE(edt)_2]^{2-}$ (E = O, S) pair.[12] In an attempt to optimize the formation of **2**, an increased S:V ratio of 5:2 was employed in a reaction mixture comprising V:Na_2edt:S:NEt_4Br in a molar ratio of 2:4.5:5:3 in MeCN, followed by workup similar to that described above. This procedure yielded analytically pure black microcrystalline **2** as the sole product; IR and electronic spectra were identical with those of the manually separated material from the original reaction. We have yet to develop a cleaner synthesis of **3** but its extreme crystallinity makes its present means of isolation adequate and reproducible.

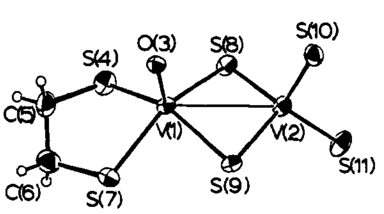

Figure 2. ORTEP projection of the anion of **3** showing the atom-labeling scheme. The comments in the caption to Figure 1 apply to this figure also. V(1)–O = 1.626 (3), V(1)–S_t(av) = 2.370, V(1)–S_b(av) = 2.375, V(2)–S_b(av) = 2.207, and V(2)–S_t(av) = 2.116 Å; S(4)–V(1)–S(7) = 84.81 (7), S(8)–V(1)–S(9) = 92.04 (7), S(8)–V(2)–S(9) = 101.47 (7), and S(10)–V(2)–S(11) = 111.63 (7)°.

Complex **3** displays a reversible one-electron reduction at −1.23 V vs. NHE;[19] complex **2** yields an identical cyclic voltammogram and we believe this extremely sensitive material is scavenging adventitious O_2 or H_2O in the electrochemical cell to convert to **3**.[20] As for **1**, the anion of **3** has no exact counterpart in known Mo or W chemistry with the edt^{2-} ligand, the closest related complexes being $[M_2S_4(edt)_2]^{2-}$ (M = Mo, W).[21,22] However, the anion of **3** is similar to some non-thiolate-containing Mo and W complexes in its overall structure, such as $[(MS_4)MO(MS_4)]^{2-}$ (M = Mo, W)[23] and $[(MoS_4)MoO(S_2MoO(S_2))]^{2-}$.[23]

The complexes described above undoubtedly presage an extensive new area of discrete early-transition-metal sulfur chemistry,

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(16) Monoclinic space group $P2_1/c$, a = 12.955 (6) Å, b = 15.963 (8) Å, c = 15.338 (8) Å, β = 109.83 (2)°, and Z = 4 for 3112 unique data with $F > 3\sigma(F)$; non-hydrogen atoms were refined anisotropically; hydrogen atoms were readily located and refined isotropically; final R (R_w) = 4.34 (4.37).

(17) Do, Y.; Simhon, E. D.; Holm, R. H., *J. Am. Chem. Soc.* **1983**, *105*, 6731.

(18) Complex 2: λ_{max} (ϵ_m) = 756 (1125), 558 (6380), 440 (sh, 4720), 328 (sh, 11 730) nm. Anal. Calcd. for $C_{18}H_{44}N_2S_7NaV_2$: C, 33.86; H, 6.90; N, 4.39. Found: C, 33.68; H, 6.79; N, 4.70. Complex **3**: λ_{max} (ϵ_M) = 728 (808), 528 (6425), 380 (10 155), 312 (sh, 14 510) nm. Anal. Calcd for $C_{18}H_{44}N_2OS_6NaV_2$: C, 34.77; H, 7.13; N, 4.15. Found: C, 34.55; H, 7.15; N, 4.48.

(19) Measurements were made in the cyclic voltammetry mode in MeCN solution with 0.1 M TBAP employed as supporting electrolyte and a carbon working electrode, referenced to the NHE by using ferrocene (+0.400 V vs. NHE) as a standard.

(20) Similarly, in the absence of enough S to yield only **2** when the V:S reaction ratio is 1:2, we believe adventitious O_2 or H_2O most likely supplies the O atom to form significant amounts of **3**.

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(23) For these and other complexes with some structural relation to **3**, see ref 5 and additional references therein.

being developed here for the first time for an early 3d metal. Differences between the behaviors of V and the heavier metals Mo and W are already apparent, but further work is necessary before any trends can be established. Variation of reaction stoichiometries is in progress and other products are being characterized. Further results in this rich new area will be reported in due course.

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Supplementary Material Available: Tables of atomic coordinates and isotropic and anisotropic thermal parameters (4 pages). Ordering information is given on any current masthead page.

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Received August 5, 1986

Articles

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Synthetic, Spectroscopic, and Solution NMR Studies of Dimethyltin(IV) Fluorosulfate Derivatives

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The ability of $(CH_3)_2Sn(SO_3F)_2$ to act as a SO_3F^- ion acceptor is illustrated by the synthesis and subsequent characterization of the new complexes $Cs[(CH_3)_2Sn(SO_3F)_3]$, $Li_2[(CH_3)_2Sn(SO_3F)_4]$, $Ba[(CH_3)_2Sn(SO_3F)_4]$, and $Sn[(CH_3)_2Sn(SO_3F)_4]$. In addition a new structural form of $(CH_3)_2Sn(SO_3F)_2$ is produced with distinctly different thermal and spectroscopic properties. The ^{119}Sn Mössbauer spectra with the isomer shift $\delta = 1.8$–1.9 mm s^{-1} and the quadrupole splitting $\Delta E_Q = 5.4$–5.5 mm s^{-1} suggest octahedral coordination in all instances, with the electronic structure of the dimethyltin group invariant in all complexes. A similar common electronic structure is found for these species in HSO_3F solution as probed by ^{1}H, ^{13}C, and ^{119}Sn NMR.

Introduction

Strong protonic acids like fluorosulfuric acid, HSO_3F, and related monosubstituted sulfuric acids have been used in the past to synthesize a variety of alkyltin(IV) chlorosulfonates by the solvolysis of alkyltin(IV) chlorides.[1-6] In these studies ^{119}Sn Mössbauer spectroscopy has played a pivotal role in the structural characterization of the produced materials with support from the reported crystal structure of $(CH_3)_2Sn(SO_3F)_2$,[7] revealing a sheetlike polymer, similar to the one found for $(CH_3)_2SnF_2$,[8] with a linear C–Sn–C grouping in a trans-octahedral coordination environment for tin due to bidentate-bridging fluorosulfate groups.

The observed bond parameters as well as the unusually large quadrupole splitting, ΔE_Q, of 5.54 mm s^{-1} [1,4-6] for $(CH_3)_2Sn(SO_3F)_2$ has suggested the presence of a cationic $(CH_3)_2Sn$ moiety in this compound.

A very recent study by us[9] has focused attention more narrowly on the possible existence of the $(CH_3)_2Sn^{2+}$ cation. If HSO_3F and the related superacid systems $HSO_3F/Sn(SO_3F)_4$[10] and $HSO_3F/Pt(SO_3F)_4$[11] are employed, materials containing not only this cation but also $[(CH_3)_2Sn(SO_3F)]^+$, both stabilized by $[M(SO_3F)_6]^{2-}$ with M = Sn or Pt, are obtained and stepwise, acidity dependent dissociation of $(CH_3)_2Sn(SO_3F)_2$ is postulated,[9] according to

$$(CH_3)_2Sn(SO_3F)_2(solv) \xrightleftharpoons{-SO_3F^-} [(CH_3)_2Sn(SO_3F)]^+(solv) \xrightleftharpoons{-SO_3F^-} [(CH_3)_2Sn]^{2+}(solv) \quad (1)$$

to explain the synthetic results.

In the basic region of HSO_3F, generated by dissolution of $CsSO_3F$, dimethyltin(IV) bis(fluorosulfate) acts as an acid, or SO_3F^- acceptor, and in the presence of 2 mol of $CsSO_3F$, the compound $Cs_2[(CH_3)_2Sn(SO_3F)_4]$ forms, while with $Sn(SO_3F)_2$ as donor a material tentatively identified as $Sn[(CH_3)_2Sn(SO_3F)_4]$ forms.[9]

The present study is intended to complement our earlier work[9] in several ways. Synthetic efforts are aimed primarily at the anionic dimethyltin(IV) fluorosulfate derivatives. The product of the reaction of equimolar amounts of $Sn(SO_3F)_2$ and $(CH_3)_2Sn(SO_3F)_2$ produces a complex ^{119}Sn Mössbauer spectrum with partly overlapping lines due to two different tin environments. Support for the suggested formulation as $Sn^{II}[(CH_3)_2Sn(SO_3F)_4]$ should come from the synthesis and characterization of the corresponding barium salt $Ba[(CH_3)_2Sn(SO_3F)_4]$.

The thermal stability of the anion $[(CH_3)_2Sn(SO_3F)_4]^{2-}$ is probed by the synthesis of $Li_2[(CH_3)_2Sn(SO_3F)_4]$ and a spectroscopic comparison to the previously reported $Cs_2[(CH_3)_2Sn(SO_3F)_4]$. The attempted metathesis of this cesium salt with $(CH_3)_2Sn(SO_3F)_2$ follows a recent precedent[9] involving $Cs_2[Sn(SO_3F)_6]$

$$2(CH_3)_2Sn(SO_3F)_2 + Cs_2[Sn(SO_3F)_6] \xrightarrow{HSO_3F} 2CsSO_3F + [(CH_3)_2Sn(SO_3F)]_2[Sn(SO_3F)_6] \quad (2)$$

A corresponding dimethyltetrakis(fluorosulfato)tin(IV) derivative with either $[(CH_3)_2Sn(SO_3F)]^+$ or $(CH_3)_2Sn^{2+}$ as counterion would constitute a structural or ionization isomer of the polymeric $(CH_3)_2Sn(SO_3F)_2$.[1,7] Its identification is expected to present a challenge.

Finally, the addition of SO_3F^- to $(CH_3)_2Sn(SO_3F)_2$, just like its dissociation in acidic medium, reaction sequence 1, is expected

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