that a small amount of the vinylic thioaldehyde complex[1] had also formed. The mixture was filtered through SiO_2, the solvent removed, and the residue chromatographed (hexane/CH_2Cl_2, 3:1). The reddish purple carbene complex (0.14 g) was obtained in 8.6% yield (0.14 g).

Preparation of [$W(CO)_5${CC(Ph)=C(Ph)SSe}] (4) and [$W(CO)_5${Se=CC(Ph)=C(Ph)SS}] (5). A solution of [$W(CO)_5${C(Ph)OEt}] (1.8 g, 4.0 mmol) in THF was treated at room temperature with 1 equiv of PhC≡CSeLi, and 2 mol equiv of S (as S_8) were added immediately. After 1 h the mixture was acidified with HCl in diethyl ether and the solvent removed. The residue was chromatographed, and the purple carbene complex 4 was isolated as the main product in a yield of 16.3% (0.41 g). The more polar light blue selone complex 5 appeared as a side product but was obtained as the main product in a yield of 10% by doing the reaction at −30 °C.

Preparation of [$W(CO)_5${CC(Ph)=C(Ph)SeSe}] (6) and [$W(CO)_5${Se=CC(Ph)=C(Ph)SeSe}] (7). A solution of [$W(CO)_5${C(OEt)Ph}] (1.2 g, 2.6 mmol) in THF was added to 1 mol equiv of PhC≡CSeLi at −30 °C. After 30 min 2 mol equiv of Se (as Se_8) were added and the mixture was stirred at 0 °C for 40 h. The reaction mixture was cooled to −78 °C and acidified with HCl in diethyl ether. The solvent was removed and the residue chromatographed (hexane/CH_2Cl_2, 1:2) to give the purple carbene complex 6 (0.15 g, 9% yield) and the more polar turquoise selone complex 7 (0.50 g, 26% yield).

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Registry No. 1, 92141-55-4; 2, 114957-55-0; 3, 114957-56-1; 4, 114957-57-2; 5, 114957-58-3; 6, 92180-83-1; 7, 92141-56-5; PhC≡CSLi, 20568-13-2; PhC≡CSeLi, 78531-03-0; PhC≡CPh, 536-74-3; $W(CO)_5C(OEt)Ph$, 36834-98-7; $Cr(CO)_5C(OEt)Ph$, 26160-57-6.

Supplementary Material Available: Tables of bond lengths and angles, temperature factors, and calculated hydrogen positions for 3 and 6 (Tables A–F) (8 pages); listing of calculated and observed structure factors for 3 and 6 (Tables G and H) (28 pages). Ordering information is given on any current masthead page.

Electron-Transfer Chemistry of $(Me_5C_5)_2Yb$: Cleavage of Diorganoperoxide and Related Chalcogenides To Give $(Me_5C_5)_2Yb(ER)(L)$ (E = O, S, Se, or Te; L = a Lewis Base). Crystal Structure of $(Me_5C_5)_2Yb(TePh)(NH_3)$

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The divalent metallocenes of ytterbium $(Me_5C_5)_2Yb(OEt_2)$ or $(Me_5C_5)_2Yb(NH_3)_2$ react with molecules of the type REER to give the trivalent ytterbium complexes $(Me_5C_5)_2Yb(ER)(L)$, where L is OEt_2 or NH_3, E is S, Se, or Te, and R is a phenyl or substituted phenyl group. The ammonia complexes are easier to characterize than the diethyl ether complexes since the latter complexes lose ether in the solid state and give unsatisfactory microanalytical data whereas the ammonia complexes give satisfactory elemental analyses. In addition, the line width of the Me_5C_5 protons in the ^1H NMR spectra of the diethyl ether complexes is ca. 500 Hz whereas the line width at half-height is ca. 50 Hz for the ammonia complexes, consistent with the notion that the barrier to chemical exchange is higher for the ammonia complexes. The peroxides ROOR, where R is Me_3C or Me_3Si, give the alkoxides $(Me_5C_5)_2Yb(OR)(NH_3)$, and $Et_2NC(S)SS(S)CNEt_2$ gives the known $(Me_5C_5)_2Yb(S_2CNEt_2)$. In contrast, dialkyl dithiophosphinates give $(Me_5C_5)_2Yb(S_2PR_2)$ and R_2PPR_2, where R is Me or Et. The synthetic routes developed in this work are the best methods currently available for synthesis of these trivalent species. The crystal structure of $(Me_5C_5)_2Yb(TePh)(NH_3)$ has been done. The crystals are orthorhombic, $P2_12_12_1$, with $a = 11.823$ (3) Å, $b = 25.917$ (6) Å, $c = 8.539$ (2) Å, and $V = 2616.5$ Å^3. For $Z = 4$, the calculated density is 1.69 g cm^{-3}. The structure was refined by full-matrix least squares to a conventional R factor of 0.046 [4991 data, $F^2 > \sigma(F^2)$]. The Yb–Te distance is 3.039 (1) Å, and the Yb–Te–C(Ph) angle is 113.0 (3)°.

The divalent ytterbium metallocene $(Me_5C_5)_2Yb(OEt_2)$ reacts with a wide variety of transition-metal carbonyls and substituted metal carbonyls with single metal–metal bonds as shown in eq 1, where X is $Co(CO)_4$,[1a] $Mn(CO)_5$,[1b] $CpFe(CO)_2$,[1c] and $CpMo(CO)_3$.[1c] The reaction in eq 1 involves the transfer of an electron from $(Me_5C_5)_2Yb(OEt_2)$ to the lowest unoccupied molecular orbital of the dimeric transition-metal carbonyl which is primarily metal–metal antibonding. The net reaction is formation of a $(Me_5C_5)_2Yb^{III}$ fragment which is bonded to the transition metal by way of M–CO–Yb bonds. The electron-transfer process that results in oxidation of $(Me_5C_5)_2Yb$ to $(Me_5C_5)_2Yb^{III}$ is an exothermic one by ca. 1.35 V (ca. 30

$$2(Me_5C_5)_2Yb(L) + XX \rightarrow 2Me_5C_5Yb(X)(L) \quad (1)$$

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kcal mol^{-1}).[2] The use of $(Me_5C_5)_2Yb$ as a hydrocarbon-soluble source of a single electron has been extended to other transition-metal cluster carbonyl systems in which metal–metal bonds are broken or formed.[3] We wanted to extend the reaction shown in eq 1 to other classes of X–X molecules, i.e., those with single X–X bonds. In this paper we show that molecules of the type REER, where R is an organic group and E is O, S, Se, or Te, can be cleaved to give compounds of the type $(Me_5C_5)_2Yb(ER)(L)$, where L is a Lewis base.

Synthetic Studies

Two molar equivalents of the diethyl ether complex $(Me_5C_5)_2Yb(OEt_2)$ react on mixing with PhEEPh, E is S, Se, or Te, in diethyl ether giving the purple (E = S, Se) or green (E = Te), trivalent compounds $(Me_5C_5)_2Yb(EPh)(OEt_2)$ (eq 1, X = EPh). The substituted alkane- and arenethiolates, $PhCH_2S$, *m*-MeC_6H_4S, and *p*-MeC_6H_4S, were prepared similarly as was the mesitylenethiolate, $(Me_5C_5)_2Yb(2,4,6\text{-}Me_3C_6H_2S)$. The latter compound was the only compound prepared in this study that crystallized free of diethyl ether, a result consistent with the large size of the mesityl group relative to the others used in this study.

The diethyl ether complexes are rather difficult to characterize since elemental analyses of all of them give low values (C, H) in combustion microanalysis. Hydrolysis of the compounds with D_2O in benzene solution shows that 1 equiv of diethyl ether is present, except in the case of the mesitylenethiolate, since the integrated intensity of the resonances in the ^1H NMR spectrum of the hydrolyzed material shows that the ratio of $Et_2O:Me_5C_5D$ is 1:2. Diethyl ether is lost in the mass spectrum, since the highest mass peak of all of the diethyl ether complexes is $[M - OEt_2]^+$. The ^1H NMR spectra of the paramagnetic complexes do not show resonances due to diethyl ether, from +100 to −85 °C, though the resonances for the Yb–ER and Yb–C_5Me_5 portions are observed. The line width, in contrast to the usual line width of the $(Me_5C_5)_2Yb^{III}$ fragment of ca. 30–50 Hz at half-height, is an order of magnitude broader in the diethyl ether complexes, presumably due to chemical exchange processes on the NMR time scale. All these observations suggest that diethyl ether is not coordinated particularly strongly in these complexes.

In order to overcome these difficulties we have prepared the ammonia complexes, since ammonia is a better base to $(Me_5C_5)_2Yb$ than is diethyl ether.[4] The ammonia complexes are readily prepared in high yield by the reaction shown in eq 2, where R is Ph and E is S, Se, or Te

$$2(Me_5C_5)_2Yb(NH_3)_2 + REER \rightarrow 2(Me_5C_5)_2Yb(ER)(NH_3) + NH_3 \quad (2)$$

and R is Me and E is S. This synthetic route is more convenient than displacing the coordinated diethyl ether in $(Me_5C_5)_2Yb(ER)(OEt_2)$ by addition of excess ammonia. In all cases the ammonia complexes give satisfactory combustion microanalytical data though, in those cases that were examined, $(M - NH_3)^+$ ions rather than molecular ions are observed in the mass spectra. The line widths in the ^1H NMR spectra are normal, i.e., ca. 30–50 Hz, though resonances due to ammonia are not observed. Further, the ammonia complexes are nicely crystalline, and an X-ray structure of $(Me_5C_5)_2Yb(SPh)(NH_3)$ has been described[5] and that of the tellurium analogue is described below.

Cleavage of sulfur–sulfur bonds is RSSR compounds by metals is not new since sodium amalgam gives NaSR and $Fe_3(CO)_{12}$ gives $Fe_2(CO)_6(\mu\text{-}SR)_2$, to quote two classic examples.[6] The reduction potential of PhSeSePh ($E_{1/2}$ = −1.20 V vs SCE)[7a] and PhTeTePh (−1.06 V)[7b] is less than that of PhSSPh (−1.70 V),[7a] and it is not surprising that $(Me_5C_5)_2Yb$ cleaves the E–E bonds in these compounds. This is the first time that lanthanide compounds have been used to cleave E–E bonds in REER compounds. Only two lanthanide thiolate complexes have been prepared $[(Me_3Si)_2N]_4Gd_2(\mu\text{-}SCMe_3)_2$ from reaction of $[(Me_3Si)_2N]_2GdCl$ and $LiSCMe_3$ or $[(Me_3Si)_2N]_3Gd$ and Me_3CSH[8a] and $(Me_5C_5)_2Lu(SCMe_3)_2Li(THF)_2$ from reaction of $(Me_5C_5)_2LuMe_2Li(THF)_2$ and Me_3CSH.[8b]

The synthetic route, eq 1 or 2, is the best one that we have been able to develop for complexes of the type $(Me_5C_5)_2Yb(SR)(L)$. We decided to extend the synthetic method to cleavage of peroxide bonds. The alkoxides $(Me_5C_5)_2Yb(OR)(NH_3)$ where R is CMe_3 or $SiMe_3$ are readily prepared from $Me_3COOCMe_3$ or $Me_3SiOOSiMe_3$. Diphenyl peroxide is not stable so the phenoxide cannot be obtained from this synthetic route.[9] Few cyclopentadienyl lanthanide alkoxides are known, and the known ones have been prepared by traditional synthetic routes of salt[10a] or alkane elimination.[8b,10b]

The scope of the non-metal to non-metal bond cleavage reaction has been examined in the following set of reactions. The diethyl ether complex $(Me_5C_5)_2Yb(OEt_2)$ reacts with tetraethyldithiuram disulfide to give the known $(Me_5C_5)_2YbS_2CNEt_2$[11] (eq 3). The phosphorus–phosphorus bond in Ph_2PPPh_2 cannot be cleaved by $(Me_5C_5)_2Yb(OEt_2)$ since the latter is recovered unchanged after the reactants are refluxed in toluene for several hours. This lack of reaction is not due to the thermodynamic instability of M–PR_2 bonds as this class of compound has been prepared by alternative synthetic methods[12] but is due either to steric effects or the high reduction potential of Ph_2PPPh_2 ($E_{1/2}$ = −2.05 V)[7c] which is more negative than that of PhSSPh by ca. 0.3 V. The metallocene

$$2(Me_5C_5)_2Yb(OEt_2) + Et_2NC(S)SS(S)CNEt_2 \rightarrow 2(Me_5C_5)_2YbS_2CNEt_2 + Et_2O \quad (3)$$

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Table I. ^{1}H NMR Spectra of $(Me_2C_5)_2Yb(ER)(L)$[a]

				R		
E	R	L	Me_5C_5	ortho	meta	para
S	Ph	OEt_2	9.7 (540)	5.5 (50)	73.2 (300)	42.6 (100)
S	*p*-tolyl	OEt_2	8.5 (500)	5.5 (35)	76.4 (300)	30.3 (50, Me)
S	*m*-tolyl	OEt_2	9.2 (500)	9.2 (*b*) 5.9 (100)	78.4 (250) 56.0 (100, Me)	44.7 (60)
S	mesityl	OEt_2	9.2 (460)	3.7 (25)	*c*	18.6 (80)
Se	Ph	OEt_2	9.7 (300)	9.7 (*d*)	62.7 (170)	37.9 (65)
Te	Ph	OEt_2	8.5 (145)	11.9 (80)	46.2 (300)	16.4 (280)
S	CH_2Ph	OEt_2	4.2 (100)[e]	−14.0 (100)	−16.0 (120)	−25.9 (70)
S	Ph	NH_3	3.8 (55)	40.0 (95)	12.8 (25)	8.7 (20)
S	*p*-tolyl	NH_3	3.8 (65)	38.8 (110)	12.7 (22)	3.8 (*b*, Me)
S	*m*-tolyl	NH_3	3.8 (57)	51.6 (120) 41.7 (100)	14.0 (20) 9.8 (*b*, Me)	9.8 (*b*)
Se	Ph	NH_3	4.3 (45)	25.1 (60)	9.6 (30)	7.4 (35)
Te	Ph	NH_3	4.2 (45)	16.5 (280)	6.7 (34)	6.0 (44)
S	CH_2Ph	NH_3	4.0 (70)[e]	−2.4 (49)	9.1 (30)	11.2 (46)
S	Me	NH_3	4.1 (65)	−5.9 (67, Me)		
O	CMe_3	NH_3	6.1 (260)	−57.5 (124, Me)		
O	$SiMe_3$	NH_3	4.8 (210)	−37.5 (67, Me)		

[a] 32 °C in either C_6D_6 or C_7D_8. The chemical shifts are expressed in δ units, positive values to high frequency, and the width at half-height is expressed in hertz which appear in parenthesis after δ. [b] The methyl resonance is under the Me_5C_5 resonance at 32 °C, but it moves out at high and low temperature. [c] Not observed. [d] Assigned on the basis of the integrated intensity. [e] The chemical shift of the methylene group is assigned at δ −6.8 (100) in OEt_2 and δ −16.0 (78) in NH_3 complexes.

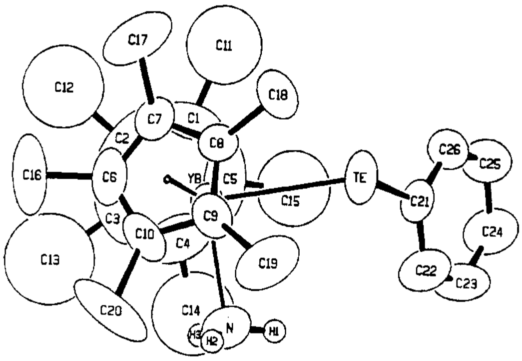

Figure 1. ORTEP drawing of $(Me_5C_5)_2Yb(TePh)(NH_3)$. All atoms are shown as 50% probability surfaces except those of the hydrogen atoms which are arbitrary. The carbon atoms C1–C5 and C11–C15 are refined isotropically.

$(Me_5C_5)_2Yb(OEt_2)$ reacts with $Me_2P(S)P(S)Me_2$ whose reduction potential is −2.48 V,[7c] but the rearrangement product $(Me_5C_5)_2Yb(S_2PMe_2)$ is the only metal-containing species isolated. Examination of the reaction solution by $^{31}P\{^1H\}$ NMR spectroscopy shows that Me_2PPMe_2 and $Me_2P(S)PMe_2$ are the only other phosphorus-containing species formed, and the net reaction can be written as in eq 4. The diethyl derivative $Et_2P(S)P(S)Et_2$ behaves similarly.

$$4(Me_5C_5)_2Yb(OEt_2) + 5Me_2P(S)P(S)Me_2 \rightarrow 4(Me_5C_5)_2Yb(S_2PMe_2) + 4OEt_2 + Me_2PPMe_2 + 2Me_2P(S)PMe_2 \quad (4)$$

^{1}H NMR Spectra

The ^{1}H NMR spectra of the paramagnetic diethyl ether complexes are complicated by chemical exchange processes that render the line widths of the Me_5C_5 and aryl-ring resonances broader than usually observed.[1] In addition the resonances due to diethyl ether are not observed. Nonetheless, information is available since the chemical shift range is large, and the resonances may be assigned as shown in Table I. These assignments are made by examination of the substituted derivatives. The methyl protons of the Me_5C_5-ring resonate at ca. δ 9, and the aryl-ring hydrogens lie in the order of decreasing chemical shift, meta > para > ortho. In the benzeneselenolates, benzenetellurolates, and phenylmethanethiolates, we have assigned the most deshielded resonance to the meta proton by analogy to that of the thiolates.

Table II. Positional Parameters with Estimated Standard Deviations for $(Me_5C_5)_2Yb(TePh)(NH_3)$

atom	*x*	*y*	*z*
Yb	0.05903 (3)	0.12049 (1)	0.12454 (5)
Te	0.12370 (6)	0.03861 (3)	−0.11399 (11)
N	0.1678 (9)	0.0734 (5)	0.3224 (13)
C1	−0.1607 (8)	0.1149 (6)	0.0854 (11)
C2	−0.1418 (9)	0.1561 (4)	0.1838 (16)
C3	−0.1034 (9)	0.1358 (5)	0.3251 (12)
C4	−0.0967 (10)	0.0832 (5)	0.3113 (14)
C5	−0.1309 (9)	0.0706 (4)	0.1621 (16)
C6	0.1259 (11)	0.2169 (4)	0.1316 (17)
C7	0.1091 (11)	0.2064 (4)	−0.0234 (13)
C8	0.1872 (8)	0.1722 (4)	−0.0785 (10)
C9	0.2615 (8)	0.1594 (4)	0.0461 (12)
C10	0.2225 (11)	0.1881 (5)	0.1795 (12)
C11	−0.202 (4)	0.1159 (13)	−0.0876 (25)
C12	−0.196 (5)	0.2098 (10)	0.165 (5)
C13	−0.079 (5)	0.1653 (12)	0.4773 (29)
C14	−0.075 (4)	0.0414 (10)	0.4370 (29)
C15	−0.160 (3)	0.0144 (7)	0.114 (4)
C16	0.0852 (19)	0.2600 (6)	0.2373 (24)
C17	0.0238 (12)	0.2345 (6)	−0.1398 (29)
C18	0.2065 (13)	0.1574 (5)	−0.2498 (12)
C19	0.3653 (11)	0.1247 (8)	0.0340 (22)
C20	0.2916 (24)	0.1902 (10)	0.3366 (17)
C21	0.0414 (8)	−0.0331 (4)	−0.0749 (132)
C22	0.0683 (12)	−0.0637 (5)	0.0457 (18)
C23	0.0114 (16)	−0.1096 (6)	0.0712 (20)
C24	−0.0712 (13)	−0.1263 (5)	−0.0288 (19)
C25	−0.0954 (10)	−0.0954 (5)	−0.1583 (16)
C26	−0.0381 (9)	−0.0505 (4)	−0.1777 (12)

In the ammonia complexes, the Me_5C_5-ring protons are shielded ca. 5 ppm relative to the values in the diethyl ether complexes. In these complexes the chemical shift of the aryl-ring hydrogens lie in the order of decreasing chemical shift, ortho > meta > para. In two cases, $(Me_5C_5)_2Yb(S\text{-}p\text{-}C_6H_4Me)(NH_3)$ and $(Me_5C_5)_2Yb(S\text{-}m\text{-}C_6H_4Me)(OEt_2)$, we have done a variable-temperature study to show that the resonances follow the Curie law[13]

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Table III. Selected Interatomic Distances and Angles in $(Me_5C_5)_2Yb(TePh)(NH_3)$

	Distances (Å)		
N–Yb	2.50 (1)	C8–Yb	2.66 (1)
Te–Yb	3.039 (1)	C9–Yb	2.68 (1)
C1–Yb	2.62 (1)	C10–Yb	2.65 (1)
C2–Yb	2.60 (1)	H1–Yb	2.74 (7)
C3–Yb	2.60 (1)	H2–Yb	2.72 (6)
C4–Yb	2.62 (1)	H3–Yb	2.76 (6)
C5–Yb	2.61 (1)	Cp1–Yb[a]	2.33
C6–Yb	2.62 (1)	Cp2–Yb[a]	2.37
C7–Yb	2.63 (1)	C21–Te	2.12 (1)
	Angles (deg)		
N–Yb–Te	89.0 (3)	Cp1–Yb–Te	113.0
Cp1–Yb–Cp2	136.9	Cp2–Yb–Te	100.9
Cp1–Yb–N	102.9	C21–Te–Yb	113.0 (3)
Cp2–Yb–N	103.5		

[a] Cp1 is the centroid of carbon atoms C1–C5, and Cp2 is the centroid of carbon atoms C6–C10.

and to show that the aryl methyls are located under the Me_5C_5-ring methyls.

Crystal Structure of $(Me_5C_5)_2Yb(TePh)(NH_3)$

An ORTEP diagram is shown in Figure 1. Positional parameters are in Table II, some bond lengths and bond angles are in Table III, and crystal data are in Table IV. The geometry of $(Me_5C_5)_2Yb(TePh)(NH_3)$ is similar to that of $(Me_5C_5)_2Yb(SPh)(NH_3)$; both compounds are pseudotetrahedral, though the crystals are not isomorphous since the latter crystallizes in the triclinic crystal system in space group $P\bar{1}$. One of the Me_5C_5 rings in the tellurium compound is severely disordered, and the carbon atoms C(1–5) and C(11–15) were refined with positional restraints as described in the Experimental Section. The disorder does not appear to affect the other groups in the molecule; in particular the hydrogen atoms in ammonia were located and refined as an anisotropic rigid group.

The $(Me_5C_5)_2Yb$ and $Yb(NH_3)$ fragments in $(Me_5C_5)_2Yb(SPh)(NH_3)$ and $(Me_5C_5)_2Yb(TePh)(NH_3)$ are nearly identical. The averaged Yb–C(C_5Me_5) distances are 2.64 (2) Å (S) and 2.63 (2) Å (Te), the Yb–C_5Me_5 centroid distances are 2.35 Å in each case, the Me_5C_5 centroid–Yb–Me_5C_5 centroid angle is 136° (S) and 137° (Te), and the Yb–N distance is 2.428 (3) Å (S) and 2.45 (1) Å (Te). Further, the Yb–S–C(Ph) and Yb–Te–C(Ph) bond parameters change in a predictable manner. The data in Table V show that in simple organic and inorganic compounds the Te–X distance is longer than the equivalent S–X distance by 0.32–0.37 Å and the angle at telluirum is smaller by ca. 7°. In the ytterbium compounds, the Yb–Te distance is 0.31 Å longer than the Yb–S distance and the Yb–Te–C(Ph) angle is closed by 4.5°. Thus, the $(Me_5C_5)_2Yb^{III}$ fragment behaves in a consistent and predictable manner.

Experimental Section

All operations were done under nitrogen. Microanalyses were performed by the microanalytical laboratory of this department. The ^1H NMR spectra were measured at 89.56 MHz, and the $^{31}P\{^1H\}$ NMR spectra were measured at 36.25 MHz on a JEOL-

Table IV. Crystallographic Summary and Data Processing for $(Me_5C_5)_2Yb(TePh)(NH_3)$

a, Å	11.823 (3)
b, A	25.917 (6)
c, A	8.539 (2)
cryst syst	orthorhombic
space group	$P2_12_12_1$
V, Å^3	2616.5
d(calcd), g/cm^{-3}	1.689
Z	4
temp, °C	23
empirical formula	$C_{26}H_{38}NTeYb$
f(000)	1292
fw	665.24
color	red
systematic absences	$h00$, $h = 2n + 1$; $0k0$, $k = 2n + 1$; $00l$, $l = 2n + 1$
X-ray	Mo Kα (graphite monochromated)
wavelength (Kα_1, Kα_2), Å	0.709 30, 0.713 59
cryst size, mm	0.16 × 0.17 × 0.69
μ, cm^{-1}	46.72
abs corr range	1.39–2.05
cryst decay corr range	0.83–1.23
diffractometer	modified Picker FACS-1
2θ limits, deg	14.7–55.1
$(\sin\theta)/\lambda$: min, max	0.180, 0.651
hkl limits	h, 0, 15; k, 0, 33; l, –11, 11
scan type	θ–2Θ
scan width, deg	1.00 + 0.693 tan θ
no. of stds	3
no. of reflctns between stds	250
variatn of stds, %	15.1, 15.6, 15.4
no. of scan data	6435
no. of unique reflctns	5996 (includes Friedel pairs)
R_{int}[b]	0.031
no. of nonzero weighted data	4991 ($F^2 > 1\sigma$)
p[c]	0.070
extinctn k[d]	0.473 × 10^{-7}
max % extinctn corr	11.9
no. of parameters	249
R (nonzero wtd dat)[e]	0.046
R_w[f]	0.059
R (all data)	0.067
goodness of fit[g]	1.26
max shift/esd in lst sqrs	0.304
max/min in diff map, e/A^3	0.94, –1.49

[a] Unit cell parameters were derived by a least-squares fit to the setting angles of the unresolved Mo Kα components of 25 reflections ($20 < 2\theta < 33$). [b] R_{int} = agreement factor between equivalent or multiply measured reflections = $\sum[I(hkl) - I(hkl)_{av}]/\sum[I(hkl)_{av}]$. [c] In the least squares, the assigned weights to the data are $[\sigma(F)]^{-2}$ were derived from $\sigma(F^2) = S^2 + (pF^2)^2$, where S^2 is the variance due to counting statistics and p is assigned a value that adjusts the weighted residuals of the strong reflections to be comparable to the weak ones. [d] Simple extinction correction has the form $(F_o)_{corr} = (1 + kI)F_o$ where I is the uncorrected intensity and F_o is the observed scattering amplitude. [e] $R = \sum[|F_o| - |F_c|]/\sum[|F_o|]$. [f] $R_w = \sqrt{\{\sum[w(|F_o| - |F_c|]^2/\sum(wF_o^2)\}}$. [g] σ_1 = error in observation of unit weight = $\sqrt{\{\sum(w[|F_o| - |F_c|]^2)/(NO - NV)\}}$, where NO is the number of observations and NV is the number of variables.

Table V. Comparison of Bond Length and Bond Angle Data for Some Sulfur and Tellurium Compounds

compd	E–X, Å	EEX or XEX, deg	E–M, Å	ref
Me_2S	1.807 (1)	99.5 (2)		14a
Me_2Te	2.142 (5)	94 (2)		14a
Cl_2S	2.005 (4)	103.0 (4)		14b
Cl_2Te	2.329 (3)	97.0 (6)		14c
PhSSPh	1.80 (1)	106.2 (2)		14d
PhTeTePh	2.12 (2)	98.9 (7)		14e
$Re_2(CO)_6(\mu\text{-}Br)_2(\mu\text{-}PhSSPh)$	1.81 (2)	109.1 (1)	2.487 (6)	14f
$Re_2(CO)_6(\mu\text{-}Br)_2(\mu\text{-}PhTeTePh)$	2.18 (2)	101.4 (1)	2.794 (5)	14g
$(Me_5C_5)_2Yb(SPh)(NH_3)$	1.771 (8)	118.5 (2.3)	2.675 (5)	5
$(Me_5C_5)_2Yb(TePh)(NH_3)$	2.12 (1)	113.0 (3)	3.039 (1)	this work

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FX90Q instrument. The ^1H chemical shifts are reported in Table I. Infrared spectra were recorded as Nujol mulls between CsI plates on a Nicolet 5DX-FTIR instrument. Mass spectra were obtained on an AEI-MS-12 machine equipped with a direct inlet using electron ionization.

$(Me_5C_5)_2Yb(SPh)(NH_3)$. An intimate mixture of $(Me_5C_5)_2Yb(OEt_2)$ (0.84 g, 1.6 mmol) and Ph_2S_2 (0.18 g, 0.81 mmol) was placed in a Schlenk flask, and diethyl ether (60 mL) was added. The purple solution was stirred for 1 h and filtered, and the filtrate was concentrated to ca. 20 mL. Cooling (−10 °C) afforded purple needles in 40% (0.37g) yield, mp 62–65 °C. Anal. Calcd for $C_{30}H_{45}OSYb$: C, 57.5; H, 7.24, S, 5.12. Found: C, 56.3; H, 6.74; S, 5.28. M^+: 553 (M − OEt_2). Hydrolysis of a small amount of the complex with D_2O in C_6D_6 and examination by ^1H NMR spectroscopy of the C_6D_6 soluble portion showed the presence of OEt_2 and Me_5C_5D in a ratio of 1:2. IR: 2720 w, 1576 w, 1142 w, 1082 s, 1020 s, 732 vs, 690 s, 472 w, 420 w, 375 br, w, 300 br, s, 225 br, m cm^{-1}.

$(Me_5C_5)_2Yb(S\text{-}p\text{-}C_6H_4Me)(OEt_2)$. This complex was prepared and isolated in a manner similar to that of the benzenethiolate above, as purple crystals in 70% yield, mp 127–129 °C. Anal. Calcd for $C_{31}H_{47}OSYb$: C, 58.1; H, 7.40. Found: C, 57.7; H, 7.21. M^+: 567 $(M - OEt_2)^+$. IR: 2730 w, 1595 w, 1480 m, 1220 w, 1085 m, 1072 m, 1012 m, 800 s, 788 w, sh, 720 w, 625 w, 490 m, 390 w, 380 w, 310 s cm^{-1}.

$(Me_5C_5)_2Yb(S\text{-}m\text{-}C_6H_4Me)(OEt_2)$. This complex was prepared in a manner similar to that of the benzenethiolate, above, and isolated as purple prisms in 86% yield, mp 130–132 °C; M^+: 567 $(M - OEt_2)^+$. IR: 3080 w, 2720 m, 1585 s, 1569 s, 1260 w, 1212 w, 1160 m, 1098 m, 1076 s, 1019 m, 862 w, 849 s, 800 w, 760 vs, 684 s, 679 s, sh, 591 m, 518 w, 490 w, 441 w, 420 w, 375 vs, br cm^{-1}.

$(Me_5C_5)_2Yb(SC_6H_2\text{-}2,4,6\text{-}Me_3)$. This compound was prepared from $(Me_5C_5)_2Yb(OEt_2)$ (0.63 g, 1.2 mmol) and dimesityl disulfide[15] (0.18 g, 0.61 mmol) in a manner similar to that of the benzenethiolate, above, and crystallized as purple blocks from diethyl ether in 89% (0.65 g) yield, mp 159–163 °C. Anal. Calcd for $C_{29}H_{41}SYb$: C, 58.6; H, 6.95; S, 5.39. Found: C, 58.3; H, 6.95; S, 5.25. M^+: 595 (M^+). IR: 3060 w, 3020 w, 2725 w, 1717 w, 1600 w, 1560 wbr, 1345 w, 1295 w, 1171 m, 1150 m, 1090 s, 1051 s, 1018 s, 962 w, 948 w, 923 w, 843 vs, 799 m, 710 m, 703 w, 669 w, 619 s, 560 w, 549 m, 395 s, 323 vs, 312 w, sh, 300 w, sh cm^{-1}.

$(Me_5C_5)_2Yb(SCH_2Ph)(OEt_2)$. This complex was prepared in a manner similar to that of the benzenethiolate, above, and crystallized as purple crystals from diethyl ether in 61% yield, mp 152–156 °C. M^+: 595 $(M - OEt_2)^+$. IR: 3063 w, 2720 w, 1600 w, 1492 m, 1322 w, 1289 w, 1262 w, 1221 w, 1187 w, 1143 w, 1121 w, 1090 wbr, 1067 w, 1039 m, 1033 m, 998 w, 915 w, 890 w, 850 w, 803 sbr, 768 sbr, 709 vs, 697 vs, 680 w, 623 w, 595 w, 563 m, 523 w, 468 w, 377 mbr, 350 w, 310 vs, 301 vs, 290 ssh cm^{-1}.

$(Me_5C_5)_2Yb(SePh)(OEt_2)$. An intimate mixture of $(Me_5C_5)_2Yb(OEt_2)$ (0.68 g, 1.3 mmol) and Ph_2Se_2 (0.20 g, 0.64 mmol) was placed in a Schlenk flask, and hexane (100 mL) was added. The purple solution was stirred for 1 h and filtered, and the filtrate was concentrated to ca. 25 mL. Cooling (−25 °C) produced purple crystals in 76% (0.67 g) yield, mp 156–160 °C. M^+: 601 $(M - OEt_2)^+$. IR: 3066 w, 2723 w, 1576 s, 1469 vs, 1460 wsh, 1151 w, 1093 w, 1072 s, 1021 s, 802 mbr, 728 vs, 690 s, 668 m, 580 wbr, 462 mbr, 384 mbr, 304 vs, 281 s cm^{-1}.

$(Me_5C_5)_2Yb(TePh)(OEt_2)$. An intimate mixture of $(Me_5C_5)_2Yb(OEt_2)$ (0.62 g, 1.2 mmol) and diphenyl ditelluride[16] (0.24 g, 0.59 mmol) was placed in a Schlenk flask, and hexane (100 mL) was added. The blue-green solution was stirred for 3 h and filtered, and the filtrate was concentrated to ca. 20 mL and then cooled (−78 °C) to produce olive green crystals in 29% (0.25 g) yield, mp 142–145 °C. IR: 3064 w, 2725 w, 1570 s, 1434 s, 1325 w, 1298 w, 1261 w, 1155 w, 1093 wbr, 1059 w, 1016 s, 1000 w, 904 w, 802 mbr, 727 vs, 696 s, 653 m, 460 w, 386 m, 306 vs cm^{-1}.

$(Me_5C_5)_2Yb(SPh)(NH_3)$. A mixture of $(Me_5C_5)_2Yb(NH_3)_2$[4] (0.57 g, 1.2 mmol) and Ph_2S_2 (0.13 g, 0.60 mmol) was placed in a Schlenk flask, and toluene (60 mL) was added. The mixture was stirred for 2 h, the red solution was filtered, and the filtrate was concentrated to ca. 25 mL. Cooling (−20 °C) afforded red crystals that were isolated and dried under reduced pressure. The color of the crystals turned to magenta upon exposure to vacuum. Yield was 0.53 g (78%); mp 206–208 °C. Anal. Calcd for $C_{26}H_{38}NSYb$: C, 54.8; H, 6.72; N, 2.46. Found: C, 54.7; H, 6.84; N, 2.34. IR: 3350 w, 3266 w, 3058 w, 2723 w, 1596 wsh, 1577 s, 1560 w, 1220 vs, 1188 w, 1130 w, 1085 s, 1062 w, 1023 m, 893 w, 800 w, 740 vs, 735 wsh, 700 s, 693 s, 596 w, 507 mbr, 484 m, 429 w, 382 w, 302 vs, 280 wsh cm^{-1}.

$(Me_5C_5)_2Yb(S\text{-}p\text{-}C_6H_4Me)(NH_3)$. This complex was prepared and isolated in a manner similar to that of the benzenethiolate, above, in 88% yield, mp 208–211 °C. Anal. Calcd for $C_{27}H_{40}NSYb$: C, 55.6; H, 6.91; H, 2.40. Found: C, 55.4; H, 6.72; N, 2.24. IR: 3360 m, 3275 m, 3090 w, 3060 w, 2730 m, 1890 w, 1790 w, 1642 w, 1595 sbr, 1481 s, 1212 vs, 1100 w, 1087 s, 1012 sbr, 808 vs, 792 wsh, 720 w, 630 m, 595 w, 510 wsh, 495 s, 395 mbr, 370 mbr, 295 s, 275 wsh cm^{-1}.

$(Me_5C_5)Yb(S\text{-}m\text{-}C_6H_4Me)(NH_3)$. This complex was prepared and isolated in a manner similar to that of the benzenethiolate, above, in 78% yield, mp 206–207 °C. Anal. Calcd for $C_{27}H_{40}NSYb$: C, 55.6, H, 6.91; N, 2.40. Found: C, 55.3; H, 6.90; N, 2.28. IR: 3356 w, 3267 w, 2724 w, 1599 wsh, 1585 s, 1566 m, 1217 vs, 1161 w, 1097 w, 1075 w, 1021 wbr, 893 w, 854 mbr, 809 wbr, 774 vs, 729 wbr, 696 s, 679 m, 510 sbr, 438 m, 381 w, 300 s, 279 w, 255 w cm^{-1}.

$(Me_5C_5)_2Yb(SCH_2Ph)(NH_3)$. This complex was prepared in a manner analogous to that of the benzenethiolate, above, and crystallized from a mixture of toluene/hexane at −10 °C in 66% yield, mp 221–223 °C. Anal. Calcd for $C_{27}H_{40}NSYb$: C, 55.6; H, 6.91; N, 2.40; S, 5.49. Found: C, 55.7; H, 7.09; N, 2.36; S, 5.46. IR: 3362 w, 3318 w, 3201 w, 3119 m, 2725 m, 1597 m, 1492 m, 1260 vs, 1221 w, 1156 w, 1065 m, 1023 m, 1000 w, 920 w, 803 m, 775 m, 725 w, 704 vs, 691 w, 562 w, 516 mbr, 375 wbr, 313 m, 303 vs, 282 wsh cm^{-1}.

$(Me_5C_5)_2Yb(SMe)(NH_3)$. This complex was prepared and isolated in a manner analogous to that of the benzenethiolate described above, in 49% yield, mp 174–176 °C. Anal. Calcd for $C_{21}H_{36}NSYb$: C, 49.7 H, 7.13; N, 2.75; S, 6.32. Found: C, 49.8; H, 7.28; N, 2.95; S, 6.20. M^+: 491 $(M - NH_3)^+$. IR: 3374 m, 3320 m, 3203 w, 3115 m, 2722 m, 1773 wbr, 1595 s, 1309 m, 1259 wsh, 1227 vs, 1166 w, 1152 w, 1090 w, 1060 w, 1022 m, 944 wbr, 803 mbr, 726 m, 702 m, 656 w, 542 sbr, 386 mbr, 375 m, 305 vs, 282 wsh cm^{-1}.

$(Me_5C_5)_2Yb(SePh)(NH_3)$. This complex was prepared and isolated in a manner similar to that used to prepare the benzenethiolate, above, in 87% yield, mp 195–198 °C. Anal. Calcd for $C_{26}H_{38}NSeYb$: C, 50.6; H, 6.21; N, 2.27. Found: C, 50.2; H, 6.21; N, 2.07. IR: 3355 w, 3314 w, 3264 w, 3056 m, 2726 w, 1594 wbr, 1574 s, 1221 vs, 1067 s, 1021 s, 734 vs, 694 s, 675 m, 525 mbr, 470 m, 384 mbr, 301 vs cm^{-1}.

$(Me_5C_5)_2Yb(TePh)(NH_3)$. This complex was prepared in a manner similar to that used to prepare and isolate the thiolate, above, in 54% yield, mp 190–191 °C. Anal. Calcd for $C_{26}H_{38}NTeYb$: C, 46.9; H, 5.76; N, 2.10. Found: C, 46.9; H, 5.75; N, 2.08. M^+: 649 $(M - NH_3)^+$. IR: 3353 w, 3254 w, 3063 w, 3047 w, 2724 w, 1955 w, 1941 w, 1882 w, 1867 w, 1811 w, 1753 w, 1570 m, 1487 m, 1430 m, 1298 w, 1226 vs, 1155 w, 1059 w, 1018 m, 999 w, 803 wbr, 730 vs, 696 s, 650 w, 597 w, 517 mbr, 491 mbr, 458 m, 388 m, 308 vs cm^{-1}.

$(Me_5C_5)_2Yb(OCMe_3)(NH_3)$. Di-*tert*-butyl peroxide (0.30 mL, 1.6 mmol) was added to $(Me_5C_5)_2Yb(NH_3)_2$ (0.49 g, 1.0 mmol) suspended in toluene (50 mL). The color changed to orange after the solution was stirred for 30 min. The solution was stirred for a further 2 h, and then the toluene was removed under reduced pressure. The residue was extracted with hexane (60 mL), the extract was filtered, and the filtrate was concentrated to ca. 40 mL and cooled (−10 °C). The orange-red crystals were collected, and several additional crops were collected from the mother liquors on concentration and cooling. The combined yield was 0.35 g (64%), mp 184–186 °C. Anal. Calcd for $C_{24}H_{42}NOYb$: C, 54.0; H, 7.93; N, 2.62. Found: C, 53.0; H, 7.91; N, 2.49. M^+: 517 $(M - NH_3)^+$. IR: 3368 m, 3264 w, 2720 w, 1592 w, 1349 m, 1310 w, 1196 vs, 1096 w, 1060 w, 1000 vs, 893 w, 803 w, 789 w, 727 m, 501 m, 483 m, 378 w, 350 w, 285 sbr cm^{-1}.

$(Me_5C_5)_2Yb(OSiMe_3)(NH_3)$. The siloxide was prepared from $(Me_5C_5)_2Yb(NH_3)_2$ and $(Me_3Si)_2O_2$[17] in a manner similar to the

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tert-butoxide and crystallized as orange plates from hexane in 48% yield, mp 221–222 °C. Anal. Calcd for $C_{23}H_{42}NOSiYb$: C, 50.3; H, 7.70; N, 2.55. Found: C, 47.1; H, 7.85; N, 2.03. M^+: 533 $(M - NH_3)^+$. IR: 3374 w, 3289 w, 2724 w, 1597 w, 1253 s, 1240 vs, 1200 vs, 980 vsbr, 865 wsh, 827 vsbr, 803 wsh, 746 s, 669 s, 495 sbr, 385 wbr, 309 s, 295 s, 279 vs cm^{-1}.

$(Me_5C_5)_2Yb(S_2CNEt_2)$. A mixture of $(Me_5C_5)_2Yb(OEt_2)$ (0.62 g, 1.2 mmol) and tetraethylthiuram disulfide (0.18 g, 0.61 mmol) were stirred in pentane (50 mL) for 2 h. The purple solution was filtered and the filtrate was concentrated to ca. 20 mL and cooled (−10 °C). The large purple crystals were collected and identified by melting point, 227–228 °C (lit. 226–227 °C),[11] and IR.

$(Me_5C_5)_2Yb(S_2PMe_2)$. A mixture of $(Me_5C_5)_2Yb(OEt_2)$ (0.54 g, 1.0 mmol) and $Me_2P(S)P(S)Me_2$ (0.20 g, 1.1 mmol) was stirred in toluene (60 mL) for 2 h. The blue-violet solution was concentrated to ca. 30 mL and cooled (−10 °C) affording dark violet crystals in 81% yield (0.48 g), mp 78–82 °C. Anal. Calcd for $C_{22}H_{36}PSYb$: C, 46.5; H, 6.38; S, 11.3. Found: C, 46.3; H, 6.37; S, 10.9. M^+: 569. IR: 2920 s, 2720 w, 1290 w, 1280 m, 1260 w, 1015 w, 945 s, 910 m, 845 w, 795 w, 730 wsh, 720 s, 622 w, 590 s, 495 s, 375 wbr, 298 s, cm^{-1}. ^1H NMR (21 °C, C_6D_6): δ 7.5 (30 H $\nu_{1/2} = 70$ Hz), −23.1 (6 H, $\nu_{1/2} = 30$ Hz). $^{31}P\{^1H\}$ NMR (21 °C, C_6D_6): δ −28.2. In a separate reaction, $(Me_5C_5)_2Yb(OEt_2)$ (40 mg) and $Me_2P(S)P(S)Me_2$ (15 mg) were mixed in C_6D_6 (2 mL) for 15 min and the mixture was then placed in a 5-mm NMR tube, the contents of which were examined by $^{31}P\{^1H\}$ NMR spectroscopy. In addition to a resonance due to $(Me_5C_5)_2Yb(S_2PMe_2)$ there were resonances due to Me_2PPMe_2 at δ −58.4 (lit. δ −58.7)[18a] and a doublet of doublets at δ 36.2 and −59.1 with $J_{PP} = 220$ Hz due to $Me_2P(S)PMe_2$ (lit. δ 35.6 and −58.7 with $J_{PP} = 224$ Hz).[18b] The total integrated intensity of the resonances due to trivalent phosphorus was approximately equal to that of $(Me_5C_5)_2Yb(S_2PMe_2)$.

$(Me_5C_5)_2Yb(S_2PEt_2)$. This complex was prepared and isolated in a manner similar to that of its dimethyl dithiophosphinate homologue in 96% yield, mp 75–77 °C. Anal. Calcd for $C_{24}H_{40}PS_2Yb$: C, 48.3; H, 6.76; S, 10.7. Found: C, 48.5; H, 6.92; S, 10.6. M^+: 597. IR: 2724 w, 1407 w, 1260 w, 1227 w, 1157 w, 1097 w, 1039 s, 1028 s, 977 w, 802 m, 767 s, 744 m, 738 m, 712 m, 671 s, 623 w, 616 vs, 507 s, 386 mbr, 307 vsbr cm^{-1}. ^1H NMR (32 °C, C_6D_6): δ 7.4 (30 H, $\nu_{1/2} = 71$ Hz), −7.5 (6 H, $\nu_{1/2} = 31$ Hz), −27.9 (4 H, $\nu_1/_2 = 32$ Hz). $^{31}P\{^1H\}$ NMR (32 °C, C_6D_6): δ 5.1. In a separate experiment 50 mg of $(Me_5C_5)_2Yb(OEt_2)$ and $Et_2P(S)P(S)Et_2$ (30 mg) were mixed in C_6D_6 (1.5 mL), the mixture was transferred into a 5-mm NMR tube, and the contents were monitored by $^{31}P\{^1H\}$ NMR spectroscopy. In addition to the resonance due to the Yb complex, resonances were found at δ −32.0 due to Et_2PPEt_2 (lit. δ −32.7)[18a] and at δ 50.7 due to $Et_2P(S)P(S)Et_2$ (lit. δ 51.3)[18a] and a doublet of doublets at δ 55.3 and −37.1 with J_{PP} of 244 Hz due to $Et_2P(S)PEt_2$ (lit. δ 55.3 and −37.1 with $J_{PP} = 243$ Hz).[18c] The total integrated intensity of the trivalent phosphorus resonances was approximately equal to that of $(Me_5C_5)_2Yb(S_2PEt_2)$.

X-ray Crystallography of $(Me_5C_5)_2Yb(TePh)(NH_3)$. The air-sensitive crystals were sealed inside thin-walled quartz capillaries under argon and then mounted on a modified Picker FACS-1 automatic diffractometer equipped with a Mo X-ray tube and a graphite monochromator. A set of θ–2θ scan data was collected and corrected for absorption (analytical method)[19] and Lorentz and polarization effects. The Yb and Te atoms were located with the use of three-dimensional Patterson maps, and subsequent least-squares refinements and electron-density maps revealed the locations of the other atoms. The structure was refined by full-matrix least squares. Anisotropic thermal parameters were assigned to all of the non-hydrogen atoms with the exception of the methyl carbon atoms on the C1–C5 cyclopentadienyl ring. The pentamethylcyclopentadienyl carbon atoms C1–C5 and C11–C15 exhibited severe anisotropic thermal motion, and it was expedient to refine these positional parameters with distance restraints[20] [C–C(ring) 1.38 ± 0.01 Å; C–C(methyl) 1.54 ± 0.01 Å]. The three ammonia hydrogen atoms were indicated in the electron difference maps and were included in the least-squares refinements with imposed N–H and H–H distance restraints[21] of 0.92 ± 0.02 and 1.59 ± 0.03 Å, respectively, and a single isotropic thermal parameter was used for all three atoms. The calculated positional parameters of the five phenyl hydrogen atoms were included (C–H = 0.95 Å) but not refined; a single isotropic thermal parameter for the five atoms was refined. Details of the data collection and the least-squares refinements are given in Table IV. A Rogers[22] parameter of 1.07 (3), ideally 1.0, confirms the absolute configuration of the crystal as described by the coordinates in Table II. Atomic scattering factors and anomalous dispersion terms were taken from ref 21. With the exception of the ORTEP program all of the computer programs used are our own.

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Registry No. $(Me_5C_5)_2Yb(SPh)(OEt_2)$, 115017-87-3; $(Me_5C_5)_2Yb(S\text{-}p\text{-}C_6H_4Me)(OEt_2)$, 115017-88-4; $(Me_5C_5)_2Yb(S\text{-}m\text{-}C_6H_4Me)(OEt_2)$, 115017-89-5; $(Me_5C_5)_2Yb(SC_6H_2\text{-}2,4,6\text{-}Me_3)(OEt_2)$, 115017-90-8; $(Me_5C_5)_2Yb(SCH_2Ph)(OEt_2)$, 115031-69-1; $(Me_5C_5)_2Yb(SePh)(OEt_2)$, 115017-91-9; $(Me_5C_5)_2Yb(TePh)(OEt_2)$, 115017-92-0; $(Me_5C_5)_2Yb(SPh)(NH_3)$, 107444-26-8; $(Me_5C_5)_2Yb(S\text{-}p\text{-}C_6H_4Me)(NH_3)$, 115017-93-1; $(Me_5C_5)_2Yb(S\text{-}m\text{-}C_6H_4Me)(NH_3)$, 115017-94-2; $(Me_5C_5)_2Yb(SCH_2Ph)(NH_3)$, 115017-95-3; $(Me_5C_5)_2Yb(SMe)(NH_3)$, 115017-96-4; $(Me_5C_5)_2Yb(SePh)(NH_3)$, 115017-97-5; $(Me_5C_5)_2Yb(TePh)(NH_3)$, 115017-98-6; $(Me_5C_5)_2Yb(OCMe_3)(NH_3)$, 115017-99-7; $(Me_2C_5)_2Yb(OSiMe_3)(NH_3)$, 115018-00-3; $(Me_5C_5)_2Yb(S_2CNEt_2)$, 81276-69-9; $(Me_2C_5)_2Yb(S_2CNEt_2)$, 81276-69-9; $(Me_5C_5)_2Yb(S_2PMe_2)$, 115018-01-4; $(Me_5C_5)_2Yb(S_2PEt_2)$, 115018-02-5; $(Me_5C_5)_2Yb(OEt_2)$, 74282-47-6; Ph_2S_2, 882-33-7; $(p\text{-}MeC_6H_4)_2S_2$, 103-19-5; $(m\text{-}MeC_6H_4)_2S_2$, 20333-41-9; $(PhCH_2)_2S_2$, 150-60-7; Ph_2Se_2, 1666-13-3; Ph_2Te_2, 32294-60-3; $(Me_5C_5)_2Yb(NH_3)_2$, 115018-03-6; Me_2S_2, 624-92-0; $Me_3COOCMe_3$, 110-05-4; $(Me_3Si)_2O_2$, 5796-98-5; $Me_2P(S)P(S)Me_2$, 3676-97-9; Me_2PPMe_2, 3676-91-3; $Et_2P(S)P(S)Et_2$, 3790-23-6; Et_2PPEt_2, 3040-63-9; dimesityl disulfide, 1483-92-7; tetraethylthiuram disulfide, 97-77-8.

Supplementary Material Available: Tables of thermal parameters, least-squares planes, and additional bond lengths and distances (5 pages); a structure factor table (11 pages). Ordering information is given on any current masthead page.

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