out for various bridged dinuclear bis(cyclopentadienyl)-lanthanide compounds. Molecular structures are known for the dilanthanide compounds of the type $R_2Ln(\mu\text{-}X)_2LnR_2$ with halogen,[20–22] carbon,[6,23] and hydrogen bridges.[24,25] Structures of dinuclear complexes containing a lanthanide metal and a group 3A element with different bridging groups are also known.[26,27] We report herein an example of a dinuclear lanthanide–lithium complex with two methyl bridges between the metal centers.

The crystal structure of $(C_5H_5)_2Er(\mu\text{-}CH_3)_2Li(tmed)$ consists of discrete molecules which show no unusually short intermolecular distances. There are four molecules per unit cell. The $(C_5H_5)_2Er$ moiety is bonded through two methyl bridges to a lithium cation, which in turn is bonded to a molecule of tmed as shown in Figure 1.

The Er–Cp distance (Cp denotes the centroid of the cyclopentadienyl group) of 2.348 (14) Å falls within the expected range based on earlier observations for erbium compounds (2.325 (20) and 2.345 (20) Å for $[(C_5H_5)_2ErCCC(CH_3)_3]_2$;[23] 2.389 (3) Å for $(C_5H_5)ErCl_2(THF)_3$[28]). However, the Er–C bond length for the methyl carbon (Er–C6 = 2.458 (19) Å) is significantly shorter than in the permethylated complex $[Li(tmed)]_3[Er(CH_3)_6]$ (2.57 (2) Å).[10,11] This is not surprising, since the permethylated complex contains six electron-deficient methyl bridges.

Previous studies for nonbridged f complexes showed that alkynide–metal bond lengths are shorter than alkyl–metal bond lengths (2.33 (2) Å in $(C_5H_5)_3UCCC_6H_5$[29] vs. 2.43 (3) Å in $(C_5H_5)_3U(n\text{-}Bu)$[30]). It was predicted that this trend should persist for bridged f-element systems.[23] We found, however, that for alkyl and alkynide bridges in dinuclear erbium complexes there exists no difference in the erbium–carbon (br) bond length ($[(C_5H_5)_2ErCCC(CH_3)_3]_2$, Er–$C_{br}$ = 2.42 (2) and 2.47 (2) Å;[23] $(C_5H_5)_2Er(\mu\text{-}CH_3)_2Li(tmed)$, Er–$C_{br}$ = 2.458 (19) Å). The metal–carbon (br) distance in the two complexes above is significantly longer than the Ln–C_{br} distance in ylide complexes (2.344 (15) and 2.378 (15) for $((CH_3)_5C_5)_2Lu(CH_2)_2P(CH_3)_2$[1]). The Li–C and Li–N distances show no abnormality and are within the range observed for the permethylated erbium complex stabilized with tmed.[11]

In 1, both metals are surrounded in a distorted tetrahedral fashion. The angle involving the metal and the two carbon bridges is the smallest for erbium (C6–Er–C6′ = 95.0 (6)°), whereas the angle Cp–Er–Cp′ (131.4 (5)°) is the largest and shows the greatest deviation from the ideal tetrahedral geometry. This behavior is not unusual and has been observed for other bridged cyclopentadienyl-lanthanide compounds ($(C_5H_5)_2Ln(\mu\text{-}CH_3)_2Ln(C_5H_5)_2$; Ln = Y, C–Ln–C = 92.3 (3)°, Cp–Ln–Cp = 128.9 (5)°; Ln = Yb, C–Ln–C = 93.4 (5)°, Cp–Ln–Cp = 128.2 (5)°[6]). The tetrahedral geometry at lithium is less distorted with the angle N–Li–N′ (88 (1)°) being the smallest. The angle at the bridging carbon (Er–C6–Li = 79.2 (9)°) is much smaller than the Ln–C–Ln angle for the dilanthanide compounds (Ln = Y, 87.7 (3)°; Ln = Yb, 86.8 (5)°[6]; Ln = Er, 96.6 (7)°[23]), but in the range of this angle for the permethylated complex (77 (2)°[11]).

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Registry No. 1, 93895-51-3; 2, 93895-53-5; 3, 93895-54-6; 4, 93895-55-7; $(C_5H_5)_2ErCl\cdot(THF)$, 93895-56-8; $(C_5H_5)_2LuCl\cdot(THF)$, 82293-69-4; $ErCl_3$, 10138-41-7; $LuCl_3$, 10099-66-8.

Supplementary Material Available: Tables of thermal parameters (Table VI), and hydrogen parameters (Table VII), and the least-squares planes (Table VIII) and a listing of observed and calculated structure factors for 1 (8 pages). Ordering information is given on any current masthead page.

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Yttrium-89 NMR Spectra of Organoyttrium Complexes

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The ^{89}Y NMR spectra of the following organoyttrium complexes are reported: $(CH_3C_5H_4)_3Y(THF)$, $[(CH_3C_5H_4)_2Y(\mu\text{-}Cl)]_2$, $(CH_3C_5H_4)_2YCl(THF)$, $[(CH_3C_5H_4)_2Y(\mu\text{-}CH_3)]_2$, $(CH_3C_5H_4)_2YCH_3(THF)$, $\{(CH_3C_5H_4)_2Y[\mu\text{-}C{\equiv}CC(CH_3)_3]\}_2$, $[(CH_3C_5H_4)_2Y(\mu\text{-}H)(THF)]_2$, $\{[(C_5H_5)_2Y(\mu\text{-}H)]_3(\mu_3\text{-}H)\}\{Li(THF)_4\}$, and $(C_5Me_5)_2Y(\mu\text{-}Cl)_2K(THF)_2$. Chemical shifts relative to YCl_3 vary over a 400 ppm range. A 27-Hz ^{89}Y–^1H coupling was resolved in the spectrum of $[(CH_3C_5H_4)_2Y(\mu\text{-}H)(THF)]_2$.

As part of a general investigation of the organometallic chemistry of the lanthanide elements,[2–4] we have studied the chemistry of organometallic yttrium complexes.[4–7] Although yttrium is not formally a lanthanide element, it

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Table I. ^{89}Y NMR Shifts of Organoyttrium Complexes

complex	shift[a]	solv	molar concn of Y^{3+}
$(CH_3C_5H_4)_3Y(THF)$	−371	THF	0.90
$[C_5(CH_3)_5]_2Y(\mu\text{-}Cl)_2K(THF)_2$	−324	THF	0.11
$(CH_3C_5H_4)_2YCl\ (THF)$	−103	THF	1.5
	−101	THF	1.7
$[(CH_3C_5H_4)_2YCl]_2$	−97	toluene	1.5
$[(CH_3C_5H_4)_2Y(\mu\text{-}H)\ (THF)]_2$	−92[b]	THF	0.28
$\{(CH_3C_5H_4)_2Y[\mu\text{-}C{\equiv}CC(CH_3)_3]\}_2$	−74	THF	0.32
$\{[(C_5H_5)_2Y(\mu\text{-}H)]_3(\mu_3\text{-}H)\}\ \{Li(THF)_4\}$	−67[c]	THF	0.41
$[(CH_3C_5H_4)_2Y(\mu\text{-}CH_3)]_2$	−15	toluene	0.26
$YCl_3(aq)$	0	H_2O	3.0
$(CH_3C_5H_4)_2YCH_3(THF)$	+40	THF	0.72

[a] Relative to YCl_3 in H_2O. [b] Triplet due to Y-$(\mu\text{-}H)_2$-Y coupling; J_{YH} = 27 Hz. [c] Multiplet due to Y-H coupling.

is congeneric with lanthanum and in the +3 oxidation state it has been found to behave chemically and structurally like the lanthanides at the end of the series which have a similar radial size[4-10] (the ionic radii of Er^{3+} and Y^{3+} are 0.881 and 0.88 Å, respectively[11]).

One advantage of investigating yttrium along with the lanthanides is that Y^{3+} is diamagnetic and hence allows straightforward product characterization of organometallic products by ^{1}H and ^{13}C NMR spectroscopy. In comparison the room-temperature magnetic moments of the late lanthanides Tb^{3+}, Dy^{3+}, Ho^{3+}, Er^{3+}, Tm^{3+}, and Yb^{3+} are 9.4–9.6, 10.4–10.5, 10.3–10.5, 9.4–9.6, 7.1–7.4, and 4.4–4.9 μ_B, respectively. Although lutetium is diamagnetic in the trivalent state, yttrium is often a more desirable metal with which to work since it is considerably less expensive and, as a 100% natural abundance $I = {}^1/_2$ element, it can provide valuable structural information via Y–H and Y–C coupling.[4-7]

An additional advantage of the $I = {}^1/_2$ yttrium nucleus is that it can be studied by NMR spectroscopy. In comparison, the only two diamagnetic trivalent lanthanide ions La^{3+} and Lu^{3+} have $I = {}^7/_2$. All of the rest of the naturally occuring trivalent lanthanides are paramagnetic, and, except for Tm^{3+} and Yb^{3+}, all have nuclear spin $I \geq 1$. ^{169}Tm is a 100% natural abundance $I = {}^1/_2$ nucleus, but Tm^{3+} has a room-temperature moment of 7.1–7.4 μ_B. ^{171}Yb has $I = {}^1/_2$, but it has a natural abundance of only 14.31% (^{173}Yb, $I = {}^5/_2$, is more abundant, 16.13%) and a room-temperature magnetic moment of 4.4–4.9 μ_B in the trivalent state.

Hence, ^{89}Y is one of only a few isotopes of the lanthanide and lanthanide-like metals that allows NMR study. Since a wide range of organolanthanide analogues can be made with yttrium, we have investigated ^{89}Y NMR spectroscopy as a means of more fully characterizing these organometallic complexes. Only four previous studies of ^{89}Y NMR spectroscopy have been published to our knowledge.[12-15] All of these involved aqueous, nonorganometallic systems.

Experimental Section

Sample Preparation. The organoyttrium complexes used in this study are all extremely air and moisture sensitive. Therefore, both the syntheses and subsequent manipulations of these compounds were conducted with rigorous exclusion of air and water using Schlenk, vacuum line, and glovebox (Vacuum/Atmospheres HE-553 Dri Lab) techniques. All solvents were thoroughly dried before use as previously described.[4] Each sample was dissolved in a proteo solvent in the glovebox in a 12-mm NMR tube to which a 24/40 joint and a stopcock adapter were attached. The assembly was attached to a vacuum line, and the tube was sealed with a torch. $[(CH_3C_5H_4)_2Y(\mu\text{-}Cl)]_2$,[5] $[(CH_3C_5H_4)_2Y(\mu\text{-}CH_3)]_2$,[4] $[(CH_3C_5H_4)_2Y(\mu\text{-}H)(THF)]_2$,[4] $\{(CH_3C_5H_4)_2Y[\mu\text{-}C{\equiv}CC(CH_3)_3]\}_2$,[4] and $\{[(C_5H_5)_2Y(\mu\text{-}H)]_3(\mu_3\text{-}H)\}\{Li(THF)_4\}$[7] were prepared according to the literature (THF = tetrahydrofuran). $(CH_3C_5H_4)_2YCl(THF)$ and $(CH_3C_5H_4)_2YCH_3(THF)$ were prepared by dissolving the unsolvated dimers in THF. $(C_5Me_5)_2Y(\mu\text{-}Cl)_2K(THF)_2$ was made from YCl_3 and KC_5Me_5.[16] $(CH_3C_5H_4)_3Y(THF)$ was prepared in the following way by variation of the procedure for the synthesis of $(CH_3C_5H_4)_3Ln$ complexes in the literature.[17] In a glovebox, anhydrous YCl_3 (0.611 g, 3.13 mmol) was combined with $NaCH_3C_5H_4$ (0.964 g, 9.45 mmol) in a flask to which 20 mL of THF was added. After being stirred for 4 h, the suspension was filtered and the filtrate was evaporated to give $(CH_3C_5H_4)_3Y(THF)$ (1.11 g, 89%): ^{1}H NMR (500 MHz, THF-d_8) δ 5.78 (s, $C_5H_4CH_3$), 5.63 (s, $C_5H_4CH_3$), 3.61 (m, THF), 2.16 (s, $C_5H_4CH_3$), 1.77 (m, THF); $^{13}C\{^1H\}$ NMR (22.631 MHz, THF-d_8) δ 115.1 (s, $C_5H_4CH_3$), 108.4 (s, $C_5H_4CH_3$), 67.4 (THF), 25.32 (THF).

NMR Spectra. The spectra were obtained in the pulsed-Fourier transform mode on a Nicolet-200 wide-bore instrument at a transmitter frequency of 9.803600 MHz using 12-mm diameter sample tubes containing 3-4 mL of solvent. Because of interference with the normal deuterium lock channel the spectra were acquired unlocked. The stability of the superconducting magnet was sufficient to allow 48-h signal averaging without undue broadening. Because of the very long relaxation times common for ^{89}Y[12-15] delays between $\pi/2$ pulses were kept to 300 s or longer. Typical spectral acquisitions required overnight runs. Shifts are reported with respect to a 3 M sample of YCl_3 in D_2O. Negative shifts mean the nucleus is found in a more shielded environment than the reference and therefore comes into resonance at a higher field.

Results and Discussion

The ^{89}Y NMR shifts of nine organoyttrium complexes are presented in Table I. The samples chosen for this

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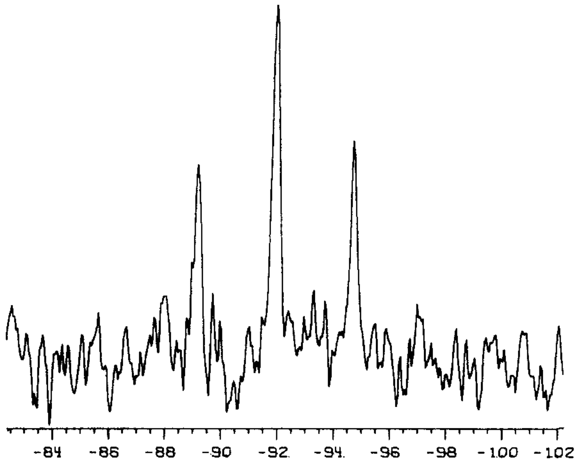

Figure 1. ^{89}Y NMR spectrum of $[(CH_3C_5H_4)_2Y(\mu\text{-}H)(THF)]_2$ in THF (shifts are in ppm vs. aqueous YCl_3; J_{YH} = 27 Hz).

study are representative of well-characterized classes of organoyttrium and organolanthanide species. Methyl-substituted cyclopentadienyl complexes were used in preference to C_5H_5 derivatives because of their enhanced solubility. Saturated solutions were used whenever possible.

The variation in chemical shift for these samples, ca. 400 ppm, is substantial and indicates that the magnetic environment of the metal is significantly affected by the ligands surrounding it. For example, comparison of the shifts of $(CH_3C_5H_4)_3Y(THF)$, $(CH_3C_5H_4)_2YCl(THF)$, and YCl_3 at −371, −101 to −103, and 0 ppm, respectively, demonstrates the effect of a cyclopentadienyl ring on shielding the nucleus. With an increasing number of cyclopentadienyl rings, the resonance is found at higher field (a more negative shift).

The spectrum of $[(CH_3C_5H_4)_2Y(\mu\text{-}H)(THF)]_2$ (Figure 1) deserves special comment in that a 27-Hz ^{89}Y–^1H coupling was resolved. In the solid state, this complex exists as a dimer that possesses a crystallographic center of inversion. Two equivalent bridging hydrides hold the monomeric units together. A triplet is observed in the ^{89}Y NMR spectrum of this complex indicating that it is dimeric in solution as well. Consistent with this, the ^1H NMR spectrum of $[(CH_3C_5H_4)_2Y(\mu\text{-}H)(THF)]_2$ contained a triplet for the bridge hydrogens.[5]

^{89}Y–^1H coupling is also observed in the spectrum of $\{[(C_5H_5)_2Y(\mu\text{-}H)]_3(\mu_3\text{-}H)\}\{Li(THF)_4\}$, but the low solubility of this sample and the fact that the signal is split into six lines by the coupling gave a signal-to-noise ratio which precluded definitive coupling assignments. A triplet of doublets pattern similar to that expected was observed, but the exact peak positions could not be specified well enough to compare with the simulated spectrum based on the coupling constants ($^1J_{Y\text{-}H}$ = 29.7 Hz (3 H), 17 Hz (1 H)) derived from the ^1H NMR study.[7] The 3.6-Hz ^{89}Y–^1H (bridge methyl) coupling observed in the ^1H NMR spectrum of $[(CH_3C_5H_4)_2Y(\mu\text{-}CH_3)]_2$[4] was not resolved in the ^{89}Y spectrum of that sample.

Conclusion

The present study provides the first ^{89}Y NMR data on organoyttrium complexes and demonstrates the viability of ^{89}Y NMR spectroscopy as a general characterization technique for organoyttrium species. The shift range is clearly broad enough to provide information on the local environment of the metal. Hence, as more ^{89}Y NMR data are collected, it will be possible to determine more fully how ^{89}Y NMR shifts correlate with ligand environments in organoyttrium complexes. We believe it is premature to make detailed correlations at this time.[18]

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Registry No. $(CH_3C_5H_4)_3Y(THF)$, 93895-57-9; $[C_5(CH_3)_5]_2Y(\mu\text{-}Cl)_2K(THF)_2$, 93895-59-1; $(CH_3C_5H_4)_2YCl(THF)$, 93895-60-4; $[(CH_3C_5H_4)_2YCl]_2$, 80642-81-5; $[(CH_3C_5H_4)_2Y(\mu\text{-}H)(THF)]_2$, 80658-44-2; $\{(CH_3C_5H_4)_2Y[\mu\text{-}C{\equiv}CC(CH_3)_3]\}_2$, 88181-91-3; $\{[(C_5H_5)_2Y(\mu\text{-}H)]_3(\mu_3\text{-}H)\}\{Li(THF)_4\}$, 90762-81-5; $[(CH_3C_5H_4)_2Y(\mu\text{-}CH_3)]_2$, 72556-67-3; $(CH_3C_5H_4)_2YCH_3(THF)$, 93895-61-5; YCl_3, 10361-92-9; $NaCH_3C_5H_4$, 55562-83-9.

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Diarsenic, As_2, as a Four-, Six-, or Eight-Electron Donor Ligand

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Diarsenic, As_2, is a versatile ligand. While it is side-on coordinated as a 4π ligand in $[Cp(CO)_2M]_2As_2$ (M = Mo (**4a**), W (**4b**)), it acts as a six-electron donor in $[Cp(CO)_2M]_2As_2Cr(CO)_5$ (M = Mo (**3a**) or W (**3b**)) or an eight-electron donor in $[Cp(CO)_2M]_2As_2[Cr(CO)_5]_2$ (M = Mo (**2a**) or W (**2b**)), respectively, by additional end-on coordination via its lone pairs. Another form of an eight-electron donor, As_2, is found for $[Cp(CO)_2Mn]_2As\text{–}As[Mn(CO)_2Cp]_2$ (**6a**), prepared by reductive coupling of two arsinidene complexes $[Cp(CO)_2Mn]AsCl$. In **6a** As_2 is bonded in a diarsinidene, $\overline{As}\text{–}\overline{As}$, valence state. As_2, side-on coordinated as a six-electron donor is present in the star-type compound $[(CO)_5Mo]_3As_2$ (**7a**) obtained from $Na_2Mo_2(CO)_{10}$ and $AsCl_3$. Its tungsten analogue $[(CO)_5W]_3As_2$ reacts with iodine to give $[(CO)_7W_2I](\mu\text{-}I)(\mu\text{-}\eta^2\text{-}As_2)$ (**8**) with a side-on coordinated four-electron donor, As_2. Syntheses and properties including structures as determined by X-ray methods are discussed.

As a higher homologue of dinitrogen, diarsenic, As_2, is a major constituent of arsenic vapor at high temperatures.[1] Even though the binding energy of this binuclear ten-electron species is quite respectable (3.93 eV),[2] it is

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