Organometallic Compounds of the Lanthanides. 179.[1] Synthesis and Structural Characterization of a Mixed Alkyl (Benzhydryl, Trimethylsilylmethyl) Lutetium Complex

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Summary: The reaction of $LuCl_3(THF)_3$ with 2 equiv of $LiCH_2SiMe_3$ and 1 equiv of $KCHPh_2$ (1) affords $Lu(CH_2SiMe_3)_2(CHPh_2)(THF)_2$ (2). X-ray structural analyses show the benzhydryl unit to be planar in 1, but bent in 2.

Introduction
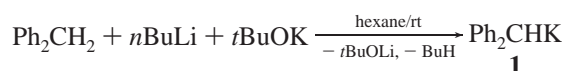
The benzhydryl anion, $CHPh_2^-$, showing a delocalized π-system, has been the subject of intense research activities. Its alkali metal derivatives were studied in detail by UV−vis absorption, NMR and ESR spectrometry, and molar conductivity measurements to determine the effects of the size of the cation and of the respective cation solvating solvent on the distribution of the π-electron density within the anion and on the thermodynamics of the equilibrium between contact ion pairs and solvent-separated ion pairs.[2] The crystal structure of [Li(12-crown-4)$_2$][CHPh$_2$] reveals an almost trigonal-planar benzhydryl anion, which does not interact either with the lithium cation or with atoms of other molecules.[3] Among the cation-solvated sodium derivatives, the monomeric [Na(PMDTA)][CHPh$_2$] exhibits a planar CHPh$_2$ unit, whereas the TMEDA-solvated derivative [Na(TMEDA)][CHPh$_2$] crystallizes as a cyclic tetramer with CHPh$_2^-$ anions showing still an sp^2-hybridized methine carbon atom, but twisted phenyl rings.[4] So far, reports on the molecular structures of comparable potassium compounds are restricted to those of the PMDETA-, DIGLYM-, and THF-adducts of KCH$_2$Ph and KCPh$_3$, which form polymeric zigzag chains or layer structures, respectively.[5,6] Recently, two different monomeric solid-state modifications of crown ether-solvated rubidium benzhydryl derivatives were reported showing η^3- and η^6-bonding modes, respectively.[7]

In the course of our efforts to synthesize alkyl complexes of the lanthanides that are not further stabilized by cyclopentadienyl or heteroatom-substituted chelating ligands, we investigated the suitability of the CHPh$_2^-$ anion for the synthesis of all-σ-bonded alkyl lanthanide complexes. Alkyl lanthanide chemistry is a quite disregarded area of f-element chemistry, which is still dominated by innumerable varieties of cyclopentadienyl complexes. There are only a few structurally characterized organolanthanide compounds containing exclusively σ-bonded alkyl ligands.[8] As expected, such complexes are extremely sensitive and fragile species since three, normally small alkyl ligands are not able to saturate the coordination sphere of the metal center. From this point of view, the CHPh$_2^-$ anion appears to be a promising candidate for the synthesis of stable alkyl lanthanide complexes due to its steric demand and its flexible bonding modes.

In this paper we report on the synthesis and structure of the first neutral heteroleptic lanthanide compound containing only alkyl ligands besides coordinated solvent molecules and on the X-ray structure of a THF solvate of the starting solvent-free KCHPh$_2$.

Results and Discussion

Pyrophoric KCHPh$_2$ (**1**) can be prepared by several methods described in the literature.[2b,d,9−11] We used the method of Schlosser[11] and obtained **1** by metalation of CH$_2$Ph$_2$ with *t*BuOK and *n*BuLi in hexane as solvent-free, bright orange crystals in an almost quantitative yield.

$$\text{Ph}_2\text{CH}_2 + n\text{BuLi} + t\text{BuOK} \xrightarrow[-\,t\text{BuOLi},\,-\,\text{BuH}]{\text{hexane/rt}} \underset{\textbf{1}}{\text{Ph}_2\text{CHK}}$$

The ^{1}H NMR spectrum of **1** recorded in d_5-pyridine corresponds with that recorded in d_8-THF.[2g] The large coupling constant $^1J(^{13}\text{C},^1\text{H}) = 146$ Hz observed for the methine group in the ^{13}C NMR spectrum of **1** reflects the high degree of sp^2-hybridization of this carbon atom.

The potassium salt **1** dissolves in THF or DME, forming dark red solutions. Addition of YbI$_2$(THF)$_2$ (2:1 mol ratio), LuCl$_3$ or YbCl$_3$ (3:1 mol ratio, each) to such solutions did not result

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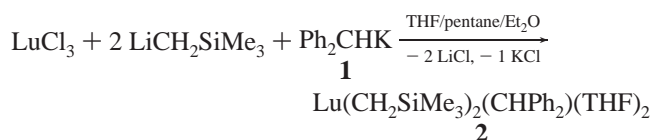
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in the isolation of homoleptic Ln(II) or Ln(III) benzhydryl complexes even under varying reaction conditions. No solid products could be isolated from the dark colored oils remaining after workup of those reaction mixtures either by extensive washing or by drying. Similar problems have been observed with the preparation of benzhydryl complexes of transition metals. Whereas the cyclopentadienyl derivatives $(C_5H_5)_2M\text{-}(CHPh_2)_2$ (M = Zr, Hf) could be structurally characterized, the complex $Ph_2Cr(CHPh_2)$ decomposes very easily with reduction of Cr(III) to Cr(II). In addition, all experiments to obtain stable homoleptic benzhydryl complexes of the transition metals Ti, Zr, Hf, V, Cr, Mo, Mn, Fe, and Y starting from the respective metal halides and benzhydryl alkali metal compounds failed.[12,13]

The problems mentioned above prompted us to investigate the possibility of synthesizing and isolating heteroleptic alkyl lanthanide derivatives containing the benzhydryl ligand. For this purpose, $LuCl_3(THF)_3$ was reacted with 2 equiv of $LiCH_2SiMe_3$ at room temperature followed by addition of 1 equiv of $KCHPh_2$ at 0 °C. Appropriate workup of the reaction mixture afforded light yellow, extremely air- and water-sensitive crystals of Lu-$(CH_2SiMe_3)_2(CHPh_2)(THF)_2$ (**2**) in yields of 32% besides oily yellow to orange side products, which could not be further identified.[14] The complex is stable for some days if kept in evacuated sealed tubes.

$$LuCl_3 + 2\,LiCH_2SiMe_3 + Ph_2CHK \xrightarrow[-\,2\,LiCl,\,-\,1\,KCl]{THF/pentane/Et_2O}$$
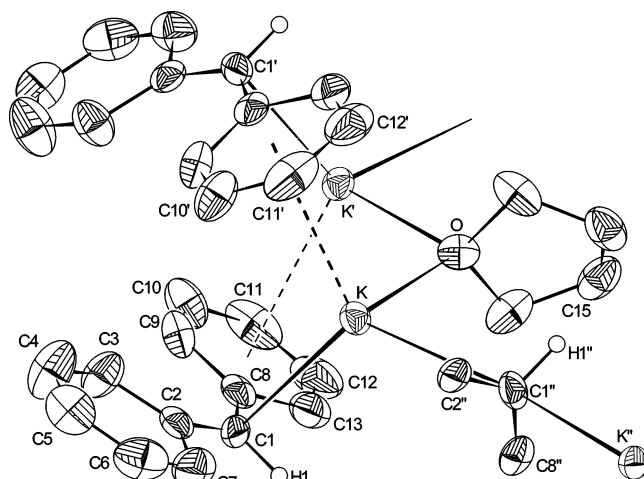$$\mathbf{1}$$
$$Lu(CH_2SiMe_3)_2(CHPh_2)(THF)_2$$
$$\mathbf{2}$$

Crystal Structures

Crystals of **1** and **2** suitable for single-crystal X-ray diffraction analysis were obtained by covering a THF solution of **1** with pentane or by cooling a saturated pentane solution of **2** to −36 °C, respectively.

Compound **1** crystallizes as THF adduct $[KCHPh_2(THF)_{0.5}]_\infty$ in the orthorhombic space group $Fdd2$ with eight molecules in the unit cell. The molecules form a complex three-dimensional structure with each potassium ion being in a distorted tetrahedal environment formed by two benzhydryl methine carbon atoms, one phenyl ring, and the oxygen atom of the THF molecule. The THF-oxygen atom additionally coordinates the potassium ion of a neighboring $KCHPh_2$ molecule. The thus oxygen-bridged potassium ions also show reciprocal interactions to one of the phenyl groups of the respective $CHPh_2$ ligands (Figure 1).

The potassium−carbon distances in $[\mathbf{1}(THF)_{0.5}]_\infty$ (3.00 and 3.10 Å for K−C(1) and K−C(1)″, respectively) show values in the range of K−C σ-bonds (2.91−3.10 Å).[15] The coordination polyhedron around C(1) corresponds to a distorted trigonal bipyramid with potassium atoms in the apical positions (K−C(1)″−K″: 165.4°) and the two phenyl rings and the methine hydrogen atom of the benzhydryl unit forming the equatorial plane. Due to the steric requirement of the phenyl rings, the angles between the equatorial ligands around C(1) slightly deviate from the ideal value of 120° (C(2)−C(1)−C(8): 132.1°, C(8)−C(1)−H(1): 115.9°, H(1)−C(1)−C(2): 111.9°). The

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Figure 1. Molecular structure of $[\mathbf{1}(THF)_{0.5}]_\infty$ (30% probability thermal ellipsoids); hydrogen atoms (except H(1)) have been omitted for clarity. Selected bond lengths [Å] and angles [deg] (Cg is defined by the ring atoms C(8)−C(13)): K−C(1) 3.003(5), K−C(2) 3.248(4), K−C(8) 3.566(5), K−C(1)″ 3.101(5), K−Cg′ 3.067(3), K−C(11)′ 3.076(6), K−C(10)′ 3.140(5), K−C(12)′ 3.255-(5), K−C(9)′ 3.437(5), K−C(13)′ 3.544(5), K−C(8)′ 3.698(5), K−O 2.778(4), C(1)−C(2) 1.441(7), C(1)−C(8) 1.429(8), C(1)−K−Cg′ 111.17(11), K−C(1)″−K″ 165.4(2), C(1)−K−O 101.31-(12), C(2)−C(1)−C(8) 132.1(5). Symmetry operations to generate equivalent atoms: (′) 1.5−*x*, 0.5−*y*, *z*, (″) 1.25−*x*, −0.25+*y*, −0.25+*z*.

torsion angle between the two intersecting planes of the phenyl rings is only 3°. The almost ideal trigonal planar arrangement of the equatorial ligands, the shortness of the C(1)−C(2) (1.44 Å) and C(1)−C(8) (1.43 Å) bonds, and, not least of all, the intense color of the compound indicate a high degree of electron delocalization within the $CHPh_2^-$ unit, which was also established for $[Li(12\text{-}crown\text{-}4)_2][CHPh_2]$.[3] The maintenance of the planarity of the $CHPh_2$ unit in solution is reflected by the large $^1J(^{13}C(1),{}^1H)$ coupling of 146 Hz in the ^{13}C NMR spectrum of the compound.

Small "hard" cations such as Li^+ usually interact with a ligand via the region of its highest electron density, while with increasing radius of the cation ($Li^+ \rightarrow Cs^+$) multihapto ligand interactions play an increasing role. Thus, the "softer" K^+ ion prefers anionic units with a more delocalized electron density such as phenyl rings. In such cases, the direction of the potassium coordination can be shifted away from the real carbanion to the peripheric phenyl groups, leading, at the most, to a η^6-bonding mode.[5,6] The potassium−phenyl coordination in $[\mathbf{1}(THF)_{0.5}]_\infty$ is somewhat asymmetric since the distances of the potassium atom to the *para*- and *meta*-C-atoms (K−C(10)′ 3.14 Å, K−C(11)′ 3.08 Å, K−C(12)′ 3.26 Å) are shorter than those to the *ortho*- and *ipso*-C-atoms (K−C(8)′ 3.70 Å, K−C(9)′ 3.44 Å, K−C(13)′ 3.54 Å). However, the η^3-bond to the center of C(10), C(11), and C(12) (2.99 Å) is only slightly shorter than would be an η^6-bond to the center of the phenyl ring (Cg: 3.07 Å) (Figure 2).

Complex **2** crystallizes in the monoclinic space group $P2_1/n$ with four molecules in the unit cell. The molecular structure shows the lutetium atom in a distorted trigonal-bipyramidal environment with the three alkyl ligands in equatorial positions and the two THF molecules in axial positions (Figure 3).
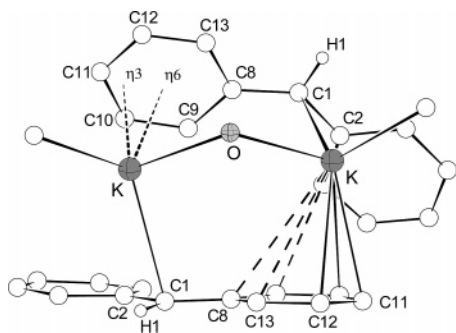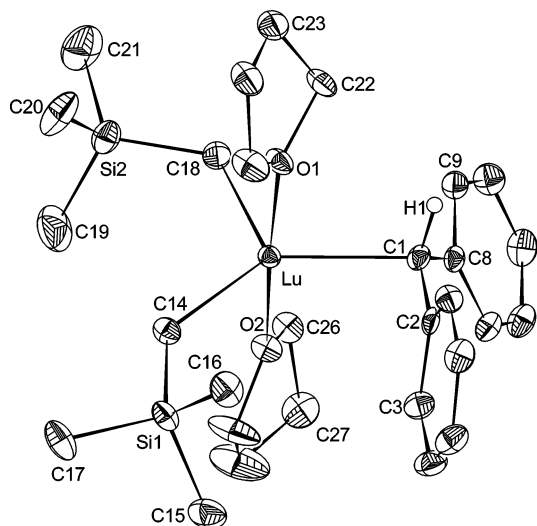
Due to the steric requirement of the benzhydryl ligand, the distance of lutetium to its methine carbon atom (Lu−C(1) (2.45 Å)) is considerably longer than to the methylene carbon atoms of the two trimethylsilylmethyl ligands (Lu−C(14): 2.35 Å,

Figure 2. Interactions between the K atoms and the phenyl rings in [**1**(THF)$_{0.5}$]$_\infty$.

Figure 3. Molecular structure of **2** (30% probability thermal ellipsoids); hydrogen atoms (except H(1)) have been omitted for clarity. Selected bond lengths [Å] and angles [deg]: Lu–C(1) 2.449(4), Lu–C(14) 2.346(4), Lu–C(18) 2.344(4), Lu–O(1) 2.306-(3), Lu–O(2) 2.301(3), C(1)–C(2) 1.460(6), C(1)–C(8) 1.464(5), C(1)–Lu–C(14) 132.81(15), C(1)–Lu–C(18) 114.09(14), C(14)–Lu–C(18) 112.96(16), O(1)–Lu–O(2) 176.50(10), O(1)–Lu–C(1) 83.58(12), O(1)–Lu–C(14) 92.99(12), O(1)–Lu–C(18) 90.04(14), O(2)–Lu–C(1) 93.47(12), O(2)–Lu–C(14) 90.40(12), O(2)–Lu–C(18) 89.44(14), C(2)–C(1)–C(8) 124.6(3), C(2)–C(1)–Lu 100.4-(2), C(8)–C(1)–Lu 110.0(3).

Lu–C(18): 2.34 Å), which in their turn are slightly shorter than the average value of the corresponding bond lengths in Lu-(CH$_2$SiMe$_3$)$_3$(THF)$_2$ (2.36 Å).[14] However, they agree well with the Lu–C distances in [Li(THF)$_3$][(C$_5$Me$_5$)Lu(CH$_2$SiMe$_3$)(CH-(SiMe$_3$)$_2$)Cl] (2.31 and 2.34 Å),[16] [Lu(CH$_2$SiMe$_3$)$_2$(crown)-(THF)$_x$][B(CH$_2$SiMe$_3$)Ph$_3$] (2.34–2.37 Å; [12]-crown-4, x = 1; [15]-crown-5, x = 0; [18]-crown-6, x = 0),[17] Cp$_2$Lu(CH$_2$-SiMe$_3$)(THF) (2.38 Å),[18] and [LuCl(2,6-(Me$_2$NCH$_2$)$_2$C$_6$H$_3$)(CH$_2$-SiMe$_3$)]$_2$) (2.39 Å).[19] The axis O–Lu–O is almost linear (176.5°), and the C–Lu–O angles are all close to 90°. In contrast to **1**, the benzhydryl unit is bent, showing the C(1) atom tetrahedrally coordinated by the lutetium atom, the two phenyl groups, and the hydrogen atom. This tetrahedral ligand arrangement is also maintained in solution, as proved by $^1J(^{13}C(1)^1H)$

= 125 Hz, indicating sp^3-hybridization of the C(1) atom.[20] The phenyl rings of the CHPh$_2$ unit in **2** are twisted, corresponding to a torsion angle of 40.3°, which is smaller than the torsion angles in Cp$_2$Zr(CHPh$_2$)$_2$ and Cp$_2$Hf(CHPh$_2$)$_2$ (Zr: 57.8° and Hf: 56.8°).[12]

Experimental Section

General Remarks. Commercially available reagents were of reagent-grade quality and were used as received. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker ARX 200 or 400 spectrometer. Lu analyses were performed by complexometric titration with EDTA as an indicator.

KCHPh$_2$ (1). The compound was prepared according to the method of Schlosser[11] by adding a solution of *n*BuLi in hexane (11.6 mL, 1.6 mol/L, 18.56 mmol) to a suspension of *t*BuOK (2.10 g, 18.71 mmol) and CH$_2$Ph$_2$ (3.14 g, 18.66 mmol) in hexane. The immediately formed orange precipitate of **1** is separated after 30 min by decantation, washed with toluene and hexane, and dried in a vacuum. Yield: 3.58 g (93%). ^{1}H NMR (200 MHz, d_5-pyridine, 25 °C): δ 7.27–7.08 (m, 8H, $H_{o/m}$), 6.28 (tt, J = 3.4 Hz, J = 1.2 Hz, 2H, H_p), 5.23 (s, 1H, C*H*). ^{13}C NMR (100.64 MHz, d_5-pyridine, 25 °C): δ 146.3 (s, C_i), 128.8 (d, J = 152 Hz, C_m), 117.3 (d, J = 152 Hz, C_o), 107.2 (d, J = 159 Hz, C_p), 81.5 (d, J = 146 Hz, *C*HPh$_2$).

Lu(CH$_2$SiMe$_3$)$_2$(CHPh$_2$)(THF)$_2$ (2). A solution of LiCH$_2$SiMe$_3$ (0.51 g, 5.42 mmol) in pentane (20 mL) was added dropwise to a stirred suspension of LuCl$_3$(THF)$_3$ (1.37 g, 2.75 mmol) in a solvent mixture of THF (1 mL), pentane (10 mL), and diethyl ether (20 mL). After stirring the mixture for 2 h, it was cooled to 0 °C. Then an orange solution of KCHPh$_2$ (**1**) (0.57 g, 2.76 mmol) in THF (4 mL) was added. After stirring the mixture for a further 30 min, it was filtered and the solvents were evaporated in a vacuum. The dark orange, oily residue was extracted with cold pentane. The filtered extract was slowly concentrated in a vacuum at room temperature. On cooling the concentrated solution to −78 °C, light yellow microcrystalline **2** (0.58 g, 32%) precipitated. Mp: 100–105 °C (dec). ^{1}H NMR (400 MHz, C$_6$D$_6$, 25 °C): δ 6.82–7.20 (m, 10H, C$_6$H$_5$), 3.64 (s, 4H, α-C*H$_2$*, THF), 3.10 (s, 1H, C*H*Ph$_2$), 1.22 (quint, 4H, β-C*H$_2$*, THF), 0.28, 0.24 (s's, ratio 1:1.5, 18H, SiC*H$_3$*), −0.86, −0.90 (s, ratio 1.5:1, 4H, C*H$_2$*SiMe$_3$). ^{13}C NMR (100.64 MHz, C$_6$D$_6$, 25 °C): δ 146.6, 129.4, 123.5, 119.8 (C$_6$H$_5$), 75.7 (d, ^1J (^{13}C^{1}H = 126 Hz, *C*HPh$_2$), 70.8 (α-CH$_2$, THF), 41.6, 41.1 (CH$_2$SiMe$_3$), 25.0 (β-CH$_2$, THF), 4.7, 4.5 (SiCH$_3$). Anal. Calcd for C$_{29}$H$_{49}$LuO$_2$Si$_2$ (660.84): Lu, 26.48. Found: Lu 26.65.

X-ray Crystal Structure Determination. Crystal data were collected on a Siemens SMART CCD diffractometer (graphite-monochromated Mo Kα radiation, λ = 0.71073 Å) by use of ω scans at 173 K. The structures were solved by direct methods and refined on F^2 using all reflections with the SHELX-97 software package.[21] The non-hydrogen atoms were refined anisotropically. Hydrogen atoms (except H(1) in **1**) were placed in calculated positions and assigned to an isotropic displacement parameter of 0.08 Å^2. The positional and isotropic thermal parameters of the hydrogen atom H(1) in **1** were refined without constraints. SADABS was used to perform area-detector scaling and absorption corrections.[22] Crystal data for **1**: MF = C$_{30}$H$_{30}$K$_2$O; M_w = 484.74, 0.52 × 0.22 × 0.20 mm^3, orthorhombic, *Fdd*2 (No. 43), a = 22.1371-(5) Å, b = 13.9116(1) Å, c = 16.2626(4) Å, Z = 8, V = 5008.27-

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(17) Å^3, $D_{calcd} = 1.286$ g/cm^3, $F(000) = 2048$, $\mu = 0.399$ mm^{-1}, $T_{max}/T_{min} = 0.9423/0.6929$, *hkl* range -27 to $28/-16$ to $18/-21$ to 16, θ range $2.13-27.50°$, reflections 9198 collected, 2515 independent ($R_{int} = 0.0869$), 1649 observed, 154 parameters, $R_1 = 0.0635$, $wR_2 = 0.0989$ ($I > 2\sigma(I)$), $R_1 = 0.1104$, $wR_2 = 0.1122$ (all data), GOF $= 1.083$, largest difference peak and hole 0.276 and -0.195 e Å^{-3}, respectively. Crystal data for **2**: MF $= C_{29}H_{49}LuO_2Si_2$; $M_w = 660.83$, $0.40 \times 0.32 \times 0.18$ mm^3, monoclinic, $P2_1/n$ (No. 14), $a = 16.7997(3)$ Å, $b = 10.7730(2)$ Å, $c = 19.5836(1)$ Å, $\beta = 112.997(1)°$, $Z = 4$, $V = 3262.62(9)$ Å^3, $D_{calcd} = 1.345$ g/cm^3, $F(000) = 1352$, $\mu = 3.120$ mm^{-1}, $T_{max}/T_{min} = 0.8819/0.7248$, *hkl* range -21 to $15/-13$ to $13/-25$ to 25, θ range $1.36-27.50°$, reflections 24 143 collected, 7444 independent ($R_{int} = 0.0633$), 5679 observed, 313 parameters, $R_1 = 0.0365$, $wR_2 = 0.0709$ ($I >$ $2\sigma(I)$), $R_1 = 0.0585$, $wR_2 = 0.0786$ (all data), GOF $= 1.003$, largest difference peak and hole 1.271 and -1.406 e Å^{-3}, respectively.

Supporting Information Available: Full crystallographic data for **1** and **2** in CIF format. This material is available free of charge via the Internet at http://pubs.acs.org.

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