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Journal of Organometallic Chemistry 663 (2002) 263–268

Journal of Organo metallic Chemistry

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Synthesis and structures of crystalline bis(trimethylsilyl)methanido complexes of potassium, calcium and ytterbium

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Received 20 July 2002; received in revised form 24 July 2002; accepted 26 July 2002

Dedicated to Professor Pascual Royo, as a mark of respect and for MFL of friendship

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Abstract

Crystalline $[K(\mu\text{-}R)(thf)]_\infty$ (**1**) was obtained from equivalent portions of *n*-butyllithium in hexane, bis(trimethylsilyl)methane (= RH) and potassium *t*-butoxide in thf, removal of volatiles and extraction with hexane. Desolvation of **1** in a vacuum led to KR. The first three-coordinate metallate(II) alkyls $[K(MR_3)]_\infty$ [M = Ca (**2**), M = Yb (**3**)] of calcium and ytterbium(II) were prepared from the appropriate metal(II) iodide and three equivalents of KR in benzene. Mixing LiR, YbI_2 and two equivalents of KR in a mixture of diethyl ether and small amount of thf yielded the red (like **3**) $[Li(thf)_4][YbR_3]$ (**4**). Each of **1**–**4** was obtained in good yield and was characterised by multinuclear NMR spectra in C_6D_6 and single crystal X-ray diffraction. The central metal is in a trigonal planar **1** or pyramidal **2**–**4** environment and the average M–C bond lengths are 2.98 (**1**), 2.50 (**2**), 2.52 (**3** and **4**) Å. Crystalline **2** and **3** are isomorphous and consist of double chains of $[MR_3]^-$ anions linked by K^+ cations along the *a* axis, whereas complex **4** has an ionic structure with isolated $[Li(thf)_4]^+$ cation and $[YbR_3]^-$ anion.

Keywords: Potassium; Calcium; Ytterbium(II); Alkyls; Metallate(II) anions; X-ray structures

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1. Introduction

The trimethylsilyl-substituted methyls $[CH_{3-n}(SiMe_3)_n]^-$ ($n = 1$–3) were recognised as valuable ligands in 1969 [1]. They often confer useful properties on their metal complexes, such as thermal stability, lipophilicity and convenient NMR spectroscopic sensors by virtue of their bulk, absence of β-hydrogen or -alkyl substituents and multiplicity of methyl groups [2]. Our group has concentrated on metal bis(trimethylsilyl)methyls, i.e. complexes of the ligand $[CH(SiMe_3)_2]^-$ ($\equiv R^-$), a topic which we reviewed about ten years ago [3]. Organometallic complexes containing tris(trimethylsilyl)methyl ($\equiv R'^-$) or related ligands having a $^-CSi_3$ or $^-C(Si)_2X(Si_2)C^-$ core have received much attention, as evident from a recent comprehensive review by Eaborn and Smith [4]. Their most recent paper on this topic is in ref. [5], while our latest, on metal bis(trimethylsilyl)methyls, is in ref. [6].

2. Results and discussion

2.1. Synthesis of $[K(\mu\text{-}R)(thf)]_\infty$ (1), $[K(MR_3)]_\infty$ [M = Ca (2), M = Yb (3)] and $[Li(thf)_4][YbR_3]$ (4) ($R = CH(SiMe_3)_2$)

The hexane-insoluble KR was previously obtained by addition of solid $KOBu^t$ to LiR in hexane [7]; addition of either methyl *t*-butyl ether or $MeN(CH_2CH_2NMe_2)_2$ (≡ pmdeta) in hexane afforded the crystalline adducts $[K(\mu\text{-}R)\{O(Me)Bu^t\}]_\infty$ (**A**) [6] or $[(pmdeta)K(\mu\text{-}R)K(\mu\text{-}R)]_2$ (**B**) [7], respectively. It was also previously observed that KR was highly reactive, not only towards toluene, but also tetrahydrofuran [6]. We now show that crystalline $[K(\mu\text{-}R)(thf)]_\infty$ (**1**) was obtained (i in Scheme 1) by mixing bis(trimethylsilyl)methane, $KOBu^t$ and $LiBu^n$ in

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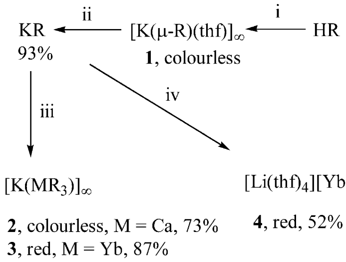
PII: S0022-328X(02)01766-7



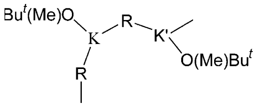
Scheme 1. Reagents and conditions: i, LiBu^n, KOBu^t, thf, −70 to ca. 25 °C; ii, vacuum, ca. 25 °C; iii, MI_2, C_6H_6, ca. 25 °C; iv, LiR, YbI_2, Et_2O–THF, −30 to ca. 25 °C.

thf at low temperature. Extraction with hexane then removed the more soluble coproduct, LiOBu^t, from **1**. Complex **1** was labile in benzene; thus, NMR spectra of a freshly prepared sample of **1** in C_6D_6 revealed that even at ambient temperature **1** was slowly decomposed with formation of RH and cleavage of thf. However, crystalline **1** was stable when stored at −70 °C for several days, and this allowed us to record not only its X-ray structure but also its ambient temperature ^{1}H-NMR spectrum in C_6D_6. It readily lost thf in a vacuum to yield neutral, ligand-free, thermally robust solid KR (ii in Scheme 1), which was used in the syntheses of **2**–**4**.

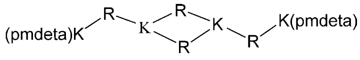
Addition of calcium or ytterbium(II) iodide to a suspension of KR in benzene afforded (iii in Scheme 1) in high yield the appropriate crystalline potassium metallate(II) $[K(MR_3)]_\infty$ [M = Ca (**2**), M = Yb (**3**)], irrespective of stoichiometry. By contrast, a similar procedure using the tris(trimethylsilyl)methyl ligand R'^- had led to the neutral homoleptic complexes $[CaR'_2]$ (**C**) [8] and $[YbR'_2]$ (**D**) [9]. This difference in outcome is attributed to the greater steric hindrance of R'^- (compared with R^-), which is adequate to shield the metal in MR'_2 from further attack by R'^-. In support, we note that while MR'_3 compounds are unknown [4], several $[MR_3]$ complexes (e.g. M = Cr or In) and even $[MR_3Cl]$ (e.g. M = Sn) have been characterised [3].

Although the compounds $KMR_3(thf)_3$ (M = Ca, Sr or Ba) are in the literature, having been obtained from KR and $M[OS(O)_2C_6H_4Me\text{-}4]_2$ and were NMR spectroscopically characterised [10], there appears to be a sole crystal structure for an alkylcalcate, the phosphine-stabilised octahedral complex **E** [11]. While some alkyllanthanate(III)s are known, e.g. [Li(tmeda)][LuBu$^t_4$] [12] and $[Li(tmeda)]_3[HoMe_6]$ [13], complex **3** is the first alkyllanthanate(II) complex.

The lithium trialkylytterbate(II) complex **4** was obtained (iv in Scheme 1) from YbI_2, LiR and KR in ether which was admixed by a small amount of tetrahydrofuran at low temperature. The first calcium alkyl $[CaR_2(1,4\text{-dioxane})_2]$ (**F**) was obtained, using a metal vapour synthesis apparatus, by co-condensation of calcium with $BrCH(SiMe_3)_2$ in thf at 77 K [14]. Other calcium alkyls to have been crystallographically characterised are $[Ca(CPh(SiMe_3)_2)_2(thf)_2]$ [15], $[Ca(C(H)Ph(SiMe_3))_2(thf)_2]$ [16], $[Ca(C(H)SiMe_3(C_6H_4\text{-}NMe_2\text{-}4))_2(thf)_2]$ [17], and $[Ca(\mu\text{-}CH_2(C_6H_4NMe_2\text{-}4))(SiMe_3\text{-fluorenyl})]_2$ [18]; $[K(\mu\text{-}CPh(SiMe_3)_2)]_\infty$ has also been described [15]. The first structurally characterised barate complex was $[PPh_4]^+[BaCp_3]^-$, containing a linear $[BaCp_3]_\infty$ chain, in which one of the three Cp^- ligands bridges adjacent tetrahedrally co-ordinated barium ions [19].



A [6]



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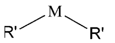
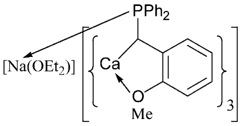
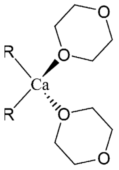


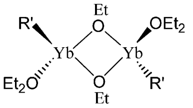
C M = Ca [8]
D M = Yb [9]



E [11]



F [14]



H [21]

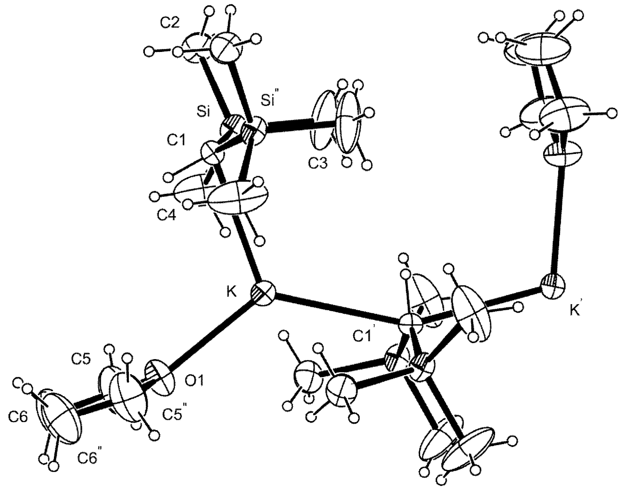


Fig. 1. Molecular structure of **1** with atom labelling. Selected bond lengths (Å) and angles (°): K–O(1) 2.645(3), K–C(1) 2.956(4), K–C(1)′ 3.012(4); O(1)–K–C(1) 108.93(12), C(1)–K–C(1)′ 122.18(5).

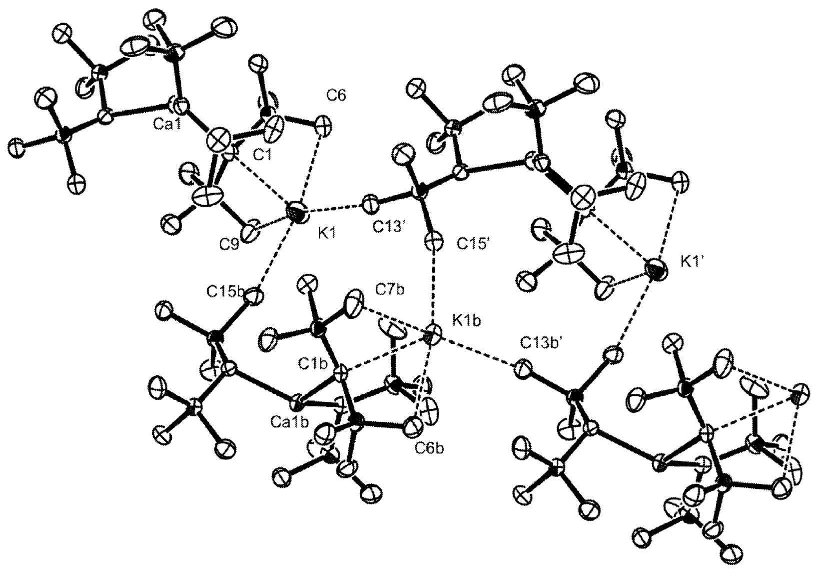


Fig. 2. Double chain structure of **2** with atom labelling (H atoms omitted). Selected bond lengths (Å) and angles (°); corresponding data for **3** in parentheses: K(1)–C(1) 3.196(5) [3.207(10)], K(1)–C(6) 3.191(5) [3.199(10)], K(1)–C(9) 3.314(5) [3.337(10)], K(1)–C(13)′ 3.181(5) [3.172(9)], K(1)–C(15b) 3.262(5) [3.256(9)], K(1b)–C(1b) 3.194(5) [3.197(10)], K(1b)–C(6b) 3.205(5) [3.217(10)], K(1b)–C(7b) 3.340(5) [3.362(10)], K(1b)–C(13b)′ 3.176(5) [3.161(8)], K(1b)–C(15)′ 3.280(5) [3.277(9)]; Ca(1)–C(1)–K(1) 134.9 [134.6], Ca(1b)–C(1b)–K(1b) 134.1 [134.4].

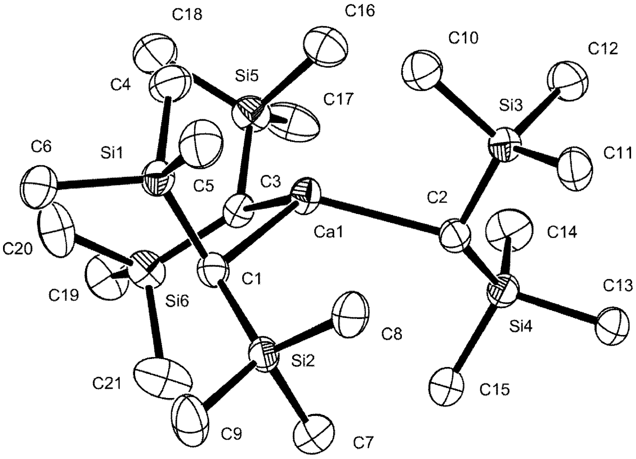


Fig. 3. Molecular structure of [CaR$_3$]$^-$ anion of **2** with atom labelling (H atoms omitted). Selected bond lengths (Å) and angles (°); corresponding data for **3** in parentheses: Ca(1)–C(1) 2.553(4) [2.551(7)], Ca(1)–C(2) 2.484(4) [2.505(8)], Ca(1)–C(3) 2.474(4) [2.503(8)], Ca(1b)–C(1b) 2.556(4) [2.555(8)], Ca(1b)–C(2b) 2.484(4) [2.521(7)], Ca(1b)–C(3b) 2.480(4) [2.508(8)], Ca(1)···C(10) 3.117(5) [3.057(9)], Ca(1)···C(4) 3.061(5) [3.033(10)], Ca(1b)···C(10b) 3.052(5) [3.010(9)], Ca(1b)···C(4b) 3.057(5) [3.025(8)]; C(3)–Ca(1)–C(2) 115.61(14) [113.1(3)], C(3)–Ca(1)–C(1) 107.86(14) [106.1(2)], C(2)–Ca(1)–C(1) 114.38(13) [112.6(3)], C(3b)–Ca(1b)–C(2b) 116.14(14) [113.9(3)], C(3b)–Ca(1b)–C(1b) 108.45(14) [106.9(3)], C(2b)–Ca(1b)–C(1b) 113.43(13) [111.3(3)].

2.2. The X-ray structures of the crystalline complexes 1–4

The molecular structures of the new K, Ca and Yb alkyls **1**–**4** are shown in Figs. 1–4, respectively. Their M–C bond lengths and corresponding C–M–C' bond angles are listed in Table 1, together with data on the related compounds **A**–**F**, [K(μ-R′)]$_\infty$ (**G**) [20] and **H** [21].

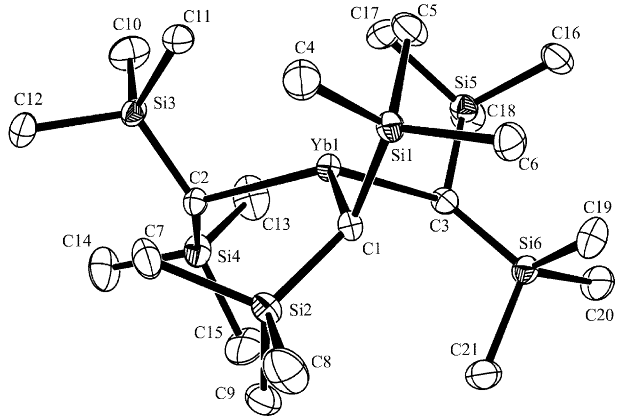


Fig. 4. Molecular structure of the [YbR$_3$]$^-$ anion in the salt **4** with atom labelling (H atoms omitted). Selected bond lengths (Å) and angles (°): Yb–C(1) 2.520(5), Yb–C(2) 2.527(5), Yb–C(3) 2.515(5), Yb···C(5) 3.217(6), Yb···C(11) 3.168(6), Yb···C(17) 3.168(6); C(1)–Yb–C(2) 115.24(16), C(2)–Yb–C(3) 112.90(17), C(1)–Yb–C(3) 111.55(16).

Crystalline **1** consists of a polymeric chain, in which potassium cations are bridged by R$^-$ anions, as shown in Fig. 1 with selected geometric parameters. A similar structure was found in **A** [6]. The small differences in K–C and K–O [2.676(9) Å] bond lengths, somewhat shorter in **1** than **A**, and the wider C–K–C′ angle in **A**, are attributed to the lower steric requirements of thf than O(Me)Bu^t. The neutral ligand-free complex **G** has slightly longer K–C bond lengths than in **1** or **A** and differs from them in that the (KC)$_\infty$ core atoms in **G** are arranged in a linear fashion [20].

Table 1
M–C bond lengths and C–M–C′ bond angles for selected complexes

Compound	**1**	**A** [6]	**B** [6]	**G** [20]	**2**	**C** [8]
M–C (Å)	2.956(4), 3.012(4)	2.988(8), 3.012(8)	3.040(4) [a] 3.095(5) [a]	3.090(11), 3.104(11)	2.474(4), 2.484(4), 2.553(4)	2.459(9)
C–M–C′ (°)	122.18(5)	137.02(9)	–	178.5(3)	107.86(14), 114.38(13), 115.61(14)	149.7(6)
Compound	**E** [11]	**F** [14]	**3**	**4**	**D** [9]	**H** [21]
M–C (Å)	2.532(4), 2.554(4), 2.573(4)	2.483(5)	2.508(8), 2.521(7), 2.555(8)	2.515(5), 2.520(5), 2.527(5)	2.490(8), 2.501(8)	2.573(13)
C–M–C′ (°)	100.2(2), 100.5(2), 105.6(2)	133.7(2)	106.9(3), 111.3(3), 113.9(3)	111.55(6), 112.90(17), 115.24(6)	137.0(4)	-

[a] These refer to terminal K–C bond lengths.

Complexes **2** and **3** are isomorphous, containing two independent ‘molecules’ in the unit cell (data for just one are here cited; for further details, see the Cambridge Crystallographic Data Base). Each consists of double chains of $[MR_3]^-$ anions linked by potassium cations along the *a* axis, Fig. 2. Each potassium cation has four additional close contacts to methyl carbon atoms: e.g. K1 has two intramolecular (C6 and C9) and two intermolecular (C13 and C15b of adjacent chains) such contacts.

Fig. 3 illustrates the structure about the Ca (**2**) and Yb (**3**) (shown in parentheses in the legend to Fig. 3) atoms in the $[MR_3]^-$ anion. The central metal M is in a trigonal pyramidal environment, the sum of the three C–M–C′ angles being 337.85° in **2** and 332.1° in **3**. This is consistent with the notion that the bonding is largely ionic, the geometry being dictated by polarisability considerations. By contrast in $[Mg(cryptand)(CH_2Bu^t)][Mg(CH_2Bu^t)_3]$ [22], the anion has a distorted trigonal planar Mg atom, consistent with the Mg–C bond being largely covalent. The M–C bond lengths and the C–M–C′ bond angles are compared for the calcium alkyls **2**, **C**, **E** (bond length only) and **F** and the ytterbium alkyls **3**, **4**, **D** and **H** in Table 1. The longest of the three M–C bonds in **2** or **3** is that which also has a close K···C contact, e.g. M–C1···K1.

The complex **4** is an ionic crystal. The ytterbium atom is in a trigonal pyramidal environment in the anion $[YbR_3]^-$, Fig. 4, the sum of the angles subtended at the ytterbium atom being 339.7°, which is significantly greater than in **3**. While the average Yb–C distance in the two compounds is similar, 2.528 in **3** and 2.521 Å in **4**, the difference between the shortest and the longest Yb–C bond lengths is greater in **3** (0.047) than in **4** (0.012 Å). Each of the R groups has one additional reasonably close $Yb\cdots CH_3$ contact of 3.22 Å (to C5) or 3.17 Å (to C11 or C17).

Table 2
Crystal data and structure refinement for **1**–**4**

Compound	**1**	**2**	**3**	**4**
Empirical formula	$C_{11}H_{27}KOSi_2$	$C_{21}H_{57}KSi_6Yb$	$C_{21}H_{57}CaKSi_6$	$C_{37}H_{89}LiOSi_6Yb$
Formula weight	270.61	690.35	557.39	946.60
Crystal system	Orthorhombic	Triclinic	Triclinic	Orthorhombic
Space group	*Pnma*	$P\bar{1}$	$P\bar{1}$	*Pbca*
a (Å)	10.1763(6)	12.1158(7)	12.1184(9)	21.5613(2)
b (Å)	14.5836(5)	15.9341(10)	15.9156(11)	22.2495(3)
c (Å)	11.7286(7)	19.3424(12)	19.3865(10)	22.7613(3)
α (°)	90	76.695(3)	76.599(4)	90
β (°)	90	89.902(3)	89.976(4)	90
γ (°)	90	81.897(3)	81.973(3)	90
V (Å^3)	1740.6(2)	3595.8(4)	3599.7(4)	10919.2(2)
Z	4	4	4	8
d_{calc} (Mg m^{-3})	1.03	1.28	1.03	1.15
λ (Å)	0.71073	0.71073	0.71073	0.71073
μ (mm^{-1})	0.42	2.92	0.50	1.87
Reflections collected	7332	20396	18437	61612
Independent reflections	1580 ($R_{int} = 0.043$)	9924 ($R_{int} = 0.067$)	8700 ($R_{int} = 0.062$)	9589 ($R_{int} = 0.072$)
Reflections with $I > 2\sigma(I)$	1323	7262	6286	6350
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.057$	$R_1 = 0.049$	$R_1 = 0.049$	$R_1 = 0.045$
R indices (all data)	$wR_2 = 0.153$	$wR_2 = 0.117$	$wR_2 = 0.100$	$wR_2 = 0.092$

3. Experimental

3.1. Materials and procedures

All manipulations were carried out under vacuum or argon by Schlenk techniques. Solvents were dried and distilled over sodium–potassium alloy (benzene, pentane, hexane) or sodium–benzophenone (Et_2O, THF) and stored over a K or Na mirror under argon. Microanalyses were carried out by Medac Ltd. (Brunel University) but due to desolvation or instability, satisfactory elemental analysis data were not obtained. The NMR spectra were recorded using the following Bruker instruments: DPX 300 (^{1}H, 300.1; ^{13}C, 75.5 MHz) and AMX 500 (^{29}Si, 49.7; ^{171}Yb, 87.5 MHz) and calibrated internally to residual solvent resonance in the case of ^{1}H and ^{13}C spectra; external $SiMe_4$ and $[Yb(C_5Me_5)_2(THF)]$ were used for ^{29}Si and ^{171}Yb spectra, respectively. All NMR spectra were recorded at ambient temperature in benzene-d_6.

3.2. Synthesis of KR

HR (2.6 ml, 1.94 g, 12.09 mmol) was added to a stirred solution of KO^{t}Bu (1.36 g, 12.12 mmol) in thf (100 ml). The solution was cooled in a −70 °C bath and 7.6 ml of a 1.6 M solution of LiBu^n (12.16 mmol) in hexane was added. The solution was allowed to warm to room temperature (r.t.) (evolution of ^{n}BuH!). Volatiles were evaporated in a vacuum and the residue was washed with hexane (70 ml). The residual solid was dried thoroughly in a vacuum at r.t. giving KR (2.23 g, 93%) as a white powder. From the extract, a small additional amount of product in the form of colourless crystals of the thf-solvate **1** was obtained upon cooling. ^{1}H: $\delta = 3.56$ (br.s., 4 H, OCH_2CH_2, thf), 1.40 (br.s., 4 H, OCH_2CH_2, thf), 0.26 (s, 18 H, $Si(CH_3)_3$), −2.25 (s, 1 H, C*H*).

3.3. Synthesis of 3

YbI_2 (0.92 g, 2.16 mmol) was added to a stirred suspension of KR (1.27 g, 6.41 mmol) in benzene (150 ml). The mixture was stirred for two days at r.t. The dark red mixture was filtered. The solvent was removed from the filtrate in a vacuum. The resulting dark red oil was washed with hexane producing red crystals of **3** (1.28 g, 87%). ^{1}H: $\delta = 0.48$ (s, 18 H, $Si(CH_3)_3$), −1.46 (s, 1 H, C*H*). ^{13}C^{1}H: $\delta = 27.27$ (*C*H), 6.66 ($Si(CH_3)_3$). ^{29}Si^{1}H: $\delta = -9.20$ (*Si*Me$_3$). ^{171}Yb: $\delta = 1284.68$ ($w_{\frac{1}{2}} \sim 80$ Hz).

3.4. Synthesis of 2

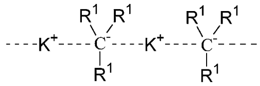
Colourless complex **2** (0.85 g, 73%) was synthesised similarly to **3**. ^{1}H: $\delta = 0.46$ (s, 18 H, $Si(CH_3)_3$), −1.58 (s, 1 H, C*H*). ^{13}C^{1}H: $\delta = 18.00$ (*C*H), 6.81 ($Si(CH_3)_3$). ^{29}Si^{1}H: $\delta = -8.59$ (*Si*Me$_3$).

3.5. Synthesis of 4

YbI_2 (0.63 g, 1.48 mmol) was added to a stirred solution of KR (0.59 g, 2.96 mmol) and LiR (0.24 g, 1.47 mmol) in a mixture of ether (150 ml) and thf (1 ml) at −30 °C. The mixture was stirred for 4 h and filtered. The solvent was removed from the filtrate in a vacuum. The resulting solid was extracted with pentane. The extract was kept at −27 °C producing red crystals of **4** (0.72 g, 52%). ^{1}H: $\delta = 3.45$ (br.s., 16 H, OCH_2CH_2, thf), 1.23 (br.s., 16 H, OCH_2CH_2, thf), 0.39 (s, 54 H, $Si(CH_3)_3$), −1.39 (s, 3 H, C*H*). ^{13}C^{1}H: $\delta = 68.83$ (OCH_2CH_2, thf), 26.54 (*C*H), 24.98 (OCH_2CH_2, thf), 6.13 ($Si(CH_3)_3$). ^{29}Si^{1}H(318 K): $\delta = -8.00$ (*Si*Me$_3$). ^{7}Li^{1}H: $\delta = -0.63$.

3.6. X-ray structure determinations for the complexes 1–4

Data for the crystal structure determinations were collected on a KappaCCD area detector at 173(2) K. Crystal data and refinement details are listed in Table 2. The structures were solved by direct methods and refined using SHELXL-97 [23]. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms on C(1) and C(1B) of **2** were freely refined. All other H atoms were in riding mode.



G [20] $R^1 = SiMe_3$

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 188046 for compound **1**, CCDC no. 188047 for compound **2**, CCDC no. 188048 for compound **3** and CCDC no. 188049 for compound **4**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

Acknowledgements

We thank the Royal Society for the award of an R.S./NATO fellowship for A.V.K. and EPSRC for financial support, Dr. A.V. Protchenko for providing the synthetic procedure for KR and Dr. A.G. Avent for recording and interpreting ^{29}Si- and ^{171}Yb-NMR spectra.

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