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A trigonal-bipyramidal coordinated ytterbium(III) alkyl: tris(trimethylsilylmethyl)bis(tetrahydrofuran-*O*)-ytterbium(III)

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Key indicators

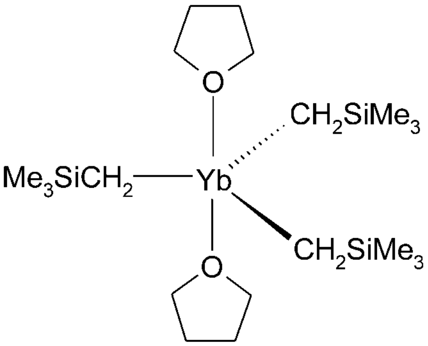
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.015 Å
Disorder in main residue
R factor = 0.055
wR factor = 0.127
Data-to-parameter ratio = 29.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, [Yb(CH$_2$SiMe$_3$)$_3$(thf)$_2$] (thf is tetrahydrofuran, C$_4$H$_8$O), was obtained by direct synthesis from ICH$_2$SiMe$_3$ and Yb chips. The Yb atom shows a slightly distorted trigonal-bipyramidal environment formed by the C atoms of three alkyl ligands in equatorial positions (average Yb—C = 2.374 Å) and the O atoms of two tetrahydrofuran molecules (average Yb—O = 2.330 Å).

Comment

σ-Bonded europium or ytterbium organyls may be conveniently prepared by a direct synthesis route from organyl iodides and lanthanide metals (Evans *et al.*, 1971; Heckmann & Niemeyer, 2000; Niemeyer, 2000). For the Grignard-analogous ytterbium^{II} aryl Yb(Dpp)I(thf)$_3$ (Dpp = 2,6–Ph$_2$C$_6$H$_3$), it was possible to detect a Schlenk-like equilibrium between Yb(Dpp)I(thf)$_3$, Yb(Dpp)$_2$(thf)$_2$ and YbI$_2$(thf)$_4$ species in thf solution, using ^{171}Yb NMR spectroscopy as a probe (Heckmann & Niemeyer, 2000). Depending on the nature of the aryl substituent and the solvent used, either the diaryl compound or the mixed aryl ytterbium iodide species may be crystallized from solution. Surprisingly, the reaction of Yb with 2,2-dimethylpropyl iodide (NeoI) did not stop at the divalent state. Instead, the trivalent ytterbium alkyl Yb(Neo)$_3$(thf)$_2$ was obtained (Niemeyer, 2000). As a continuation of this work, the reaction between Yb metal and trimethylsilylmethyl iodide has now been studied. It has been shown that this reaction produces orange crystals of the title compound, Yb(CH$_2$SiMe$_3$)$_3$(thf)$_2$, (I), in moderate yield.



(I)

It should be noted that the preparation of (I) and other tris(trimethylsilylmethyl) compounds of the late lanthanide elements by conventional metathesis between LiCH$_2$SiMe$_3$ and LnCl$_3$ (Ln = rare earth metal) was reported more than 20 years ago (Atwood *et al.*, 1978; Schumann & Müller, 1978).

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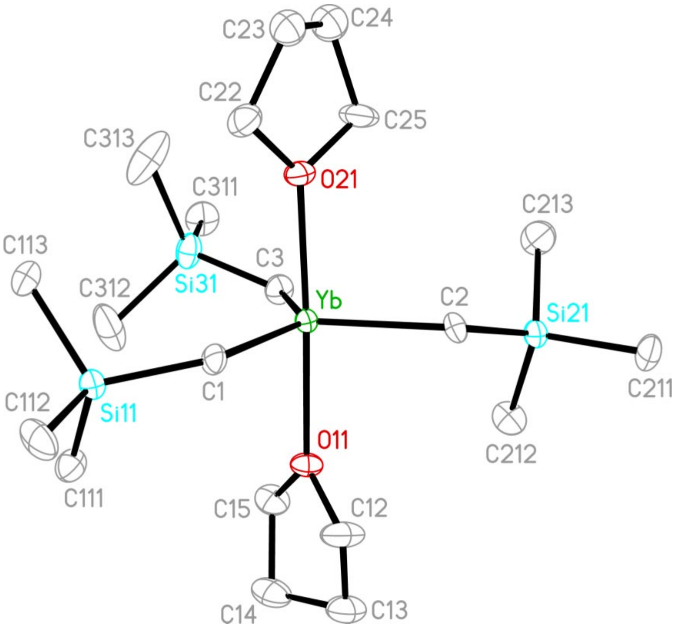

Figure 1

Structure of (I) with displacement ellipsoids at the 30% probability level. H atoms and minor sites of the disordered thf molecule have been omitted for clarity.

However, no X-ray structural determinations have been published, presumably due to the limited stability of these highly reactive species and the lack of low-temperature devices at that time.

The molecular structure of (I) shows monomeric units (Fig. 1) with a five-coordinate Yb atom. The three CH_2SiMe_3 alkyl groups and two thf ligands occupy the equatorial and axial positions of a distorted trigonal bipyramid. While the angles between the axial and equatorial ligands ($90\pm3^\circ$) and the O11—Yb—O21 angle of 177.49 (19)° are close to the ideal values, there are larger deviations for the inter-equatorial angles. This is due to the fact that the highest possible local symmetry (C_{3h}) for the $Yb(CH_2SiMe_3)_3$ fragment, *i.e.* a propeller-like arrangement of the alkyl groups, is not realised. Instead, each pair of ligands shows a *syn*/*syn, syn*/*anti* or *anti*/*anti* conformation (Fig. 2) in which the corresponding C—Yb—C angles of 134.0 (3) (C1—Yb—C3), 116.1 (3) (C2—Yb—C3) and 109.8 (2)° (C1—Yb—C2) reflect the different degrees of interligand repulsion. For the same reason, there are significant variations of the Yb—C distances {the longest being Yb—C3 [2.389 (7) Å] to the most sterically crowded alkyl group} and Yb—C—Si angles {the most acute being Yb—C2—Si21 [122.9 (3)°] to the least sterically crowded group}. The average Yb—C (2.374 Å) and Yb—O (2.330 Å) bond distances may be compared with the corresponding values in the isostructural compound $Yb(Neo)_3(thf)_2$ (Niemeyer, 2000) which are 2.379 and 2.354 Å, respectively.

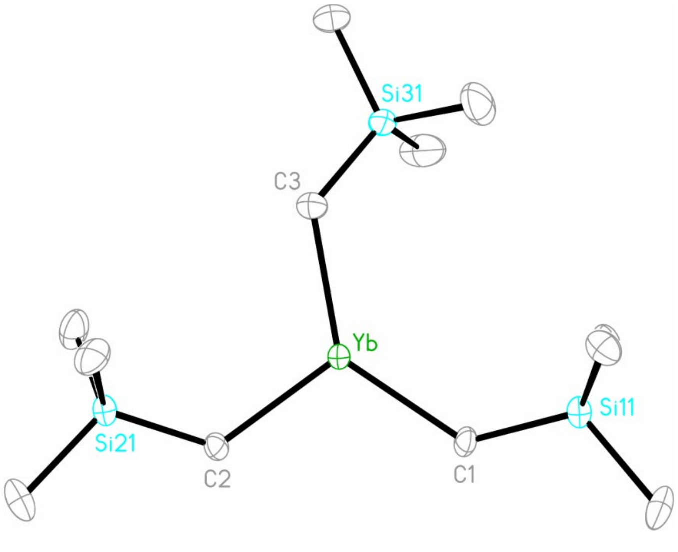

Figure 2

View perpendicular to the central $Yb(CH_2SiMe_3)_3$ fragment of (I), showing the conformation of the alkyl groups. H atoms and thf ligands have been omitted for clarity.

Experimental

All manipulations were carried out under strictly anaerobic and anhydrous conditions using purified argon as inert atmosphere. ICH_2SiMe_3 (0.56 ml, 3.8 mmol) was added at 273 K to a stirred suspension of Yb chips (0.65 g, 3.8 mmol) in 20 ml of tetrahydrofuran. Stirring of the resulting red–brown mixture was continued for 1 h, whereupon all volatile materials were removed under reduced pressure. The remaining solid was extracted with *ca.* 15 ml of *n*-hexane and the solution was separated by centrifugation. The volume of the resulting red solution was reduced in vacuum to *ca* 3 ml. Cooling in a 213 K freezer afforded orange–red crystals of (I) in 48% yield; m.p. below 293 K. ^{1}H NMR (C_6D_6, c = 0.08 mol l^{-1}, 250 MHz): δ −231.0 (s [$w_{1/2}$ = 780 Hz], 6 H, $CH_2{}^t$Bu), −33.3 (s [$w_{1/2}$ = 110 Hz], 27 H, ^{t}Bu), 83.3 (s [$w_{1/2}$ = 1050 Hz], 8 H, OCH_2CH_2), 170.5 p.p.m. (s [$w_{1/2}$ = 3000 Hz], 8 H, OCH_2). IR (CsBr, Nujol): 1249 *s*, 1237 *s*, 1071 *w*, 1038 *sh*, 1025 *ms*, 920 *sh*, 883 *sh*, 857 *vs*, 820 *m*, 737 *m*, 712 *ms*, 696 *sh*, 671 *m*, 433 *m*. UV/VIS (*n*-pentane, c = 4.0 mol l^{-1}): λ_{max} (ε) = 350 (210), 474 (180), 914 (33), 937 (21), 982 (45). A crystal suitable for X-ray crystallographic studies was selected under a protecting layer of precooled perfluorinated polyether, attached to a glass fiber and immediately transferred on a block of dry ice to the low-temperature device of the diffractometer.

Crystal data

$[Yb(CH_2SiC_3H_9)_3(C_4H_8O)_2]$
M_r = 578.90
Monoclinic, $P2_1/n$
a = 10.6627 (12) Å
b = 18.549 (2) Å
c = 14.742 (2) Å
β = 95.293 (10)°
V = 2903.3 (6) Å^3
Z = 4

D_x = 1.324 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 36 reflections
θ = 6.0–12.1°
μ = 3.36 mm^{-1}
T = 173 (2) K
Block, orange
0.40 × 0.35 × 0.20 mm

Data collection

Rebuilt Syntex $P2_1$/Siemens $P3$ four-circle diffractometer
Wyckoff scans
Absorption correction: ψ scan North *et al.* (1968)
$T_{\min} = 0.467$, $T_{\max} = 0.818$
7465 measured reflections
7113 independent reflections
5064 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.058$
$\theta_{\max} = 29.0°$
$h = -9 \rightarrow 14$
$k = -21 \rightarrow 25$
$l = -20 \rightarrow 20$
2 standard reflections every 198 reflections intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.127$
$S = 1.15$
7113 reflections
245 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 1.23$ e Å^{-3}
$\Delta\rho_{\min} = -1.07$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Yb—C1	2.369 (6)	Yb—O11	2.341 (5)
Yb—C2	2.364 (7)	Yb—O21	2.319 (5)
Yb—C3	2.389 (7)		
O11—Yb—O21	177.49 (19)	C1—Yb—C2	109.8 (2)
C1—Yb—O11	87.1 (2)	C1—Yb—C3	134.0 (3)
C1—Yb—O21	92.1 (2)	C2—Yb—C3	116.1 (3)
C2—Yb—O11	90.9 (2)	Yb—C1—Si11	130.1 (4)
C2—Yb—O21	91.6 (2)	Yb—C2—Si21	122.9 (3)
C3—Yb—O11	89.5 (2)	Yb—C3—Si31	128.0 (4)
C3—Yb—O21	89.3 (3)		

The disordered ethylene bridge in one thf ligand was modelled with split positions (0.65/0.35), isotropic displacement parameters for C23, C24, C23*a* and C24*a*, and *SADI* equal-distance restraints. All H atoms were positioned with idealized geometry and refined in a riding model approximation, including free rotation for the methyl groups. A common isotropic displacement parameter was refined for groups of CH_3, CH_2 (bonded to Yb) and CH_2 (thf) H atoms, respectively.

Data collection: *P*3 (Siemens, 1989); cell refinement: *P*3; data reduction: *XDISK* (Siemens, 1989); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL*.

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