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An octahedrally coordinated trialkylyttrium(III): tris(trimethylsilylmethyl)(1,4,7-trimethyl-1,4,7-triazacyclononane)yttrium(III)

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The crystal structure of the title compound, $[Y(C_4H_{11}Si)_3(C_9H_{21}N_3)]$, consists of monomeric species. In each of the three independent molecules in the asymmetric unit, the Y atom is in a slightly distorted octahedral environment, formed by the C atoms of three trimethylsilylmethyl ligands (average Y—C = 2.427 Å) and three facially bound N atoms of the 1,4,7-trimethyl-1,4,7-triazacyclonane ligand (average Y—N = 2.601 Å).

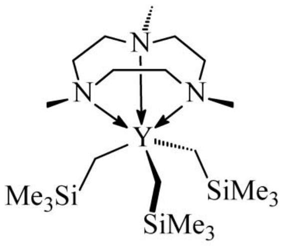
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.011 Å
R factor = 0.068
wR factor = 0.142
Data-to-parameter ratio = 27.4

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

Comment

Homoleptic trialkyl group 3 and lanthanide compounds are valuable starting materials for a broad range of organo–rare-earth-metal complexes. The most prominent example is $[Y(CH_2SiMe_3)_3(THF)_2]$ (THF is tetrahydrofuran; Lappert & Pearce, 1973), which has, in recent years, been used extensively to generate new neutral and cationic derivatives. Such compounds are of interest as catalysts in a range of catalytic transformations such as olefin polymerization (Gromada *et al.*, 2004; Arndt & Okuda, 2005). In the course of our investigations in this field, we have developed a number of amidinate and R_2-TACN-amide-supported yttrium dialkyls (TACN is 1,4,7-triazacyclonane and R_2-TACN is 4,7-disubstituted 1,4,7-triazacyclonane) generated from $[Y(CH_2SiMe_2)_3(THF)_2]$ (Bambirra *et al.*, 2001, 2003). Recently, Lawrence *et al.* (2003) have reported a related yttrium trialkyl, namely the title compound, $[Y(CH_2SiMe_3)_3(Me_3\text{-TACN})]$ (Me_3-TACN is 1,4,7-trimethyl-1,4,7-triazacyclonane), (I).



(I)

We have used this complex for a series of reactivity studies and obtained X-ray quality crystals from pentane solution at 243 K. We describe here the molecular structure of (I). The asymmetric unit consists of three molecules, one of which is shown in Fig. 1. It is seen that the Y atom is six-coordinate in an octahedral arrangement. The Me_3-TACN ligand is facially coordinated to the metal centre. The three alkyl groups, bound to Y, are arranged in a propeller-like motif (Fig. 2). In addition, in order to minimize steric hindrance, the Y-bound alkyls adopt a staggered orientation with respect to the Me_3-TACN

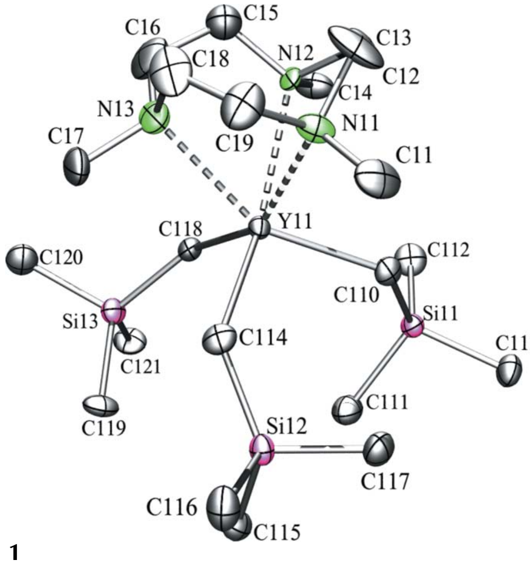


Figure 1
The structure of one of the independent molecules of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

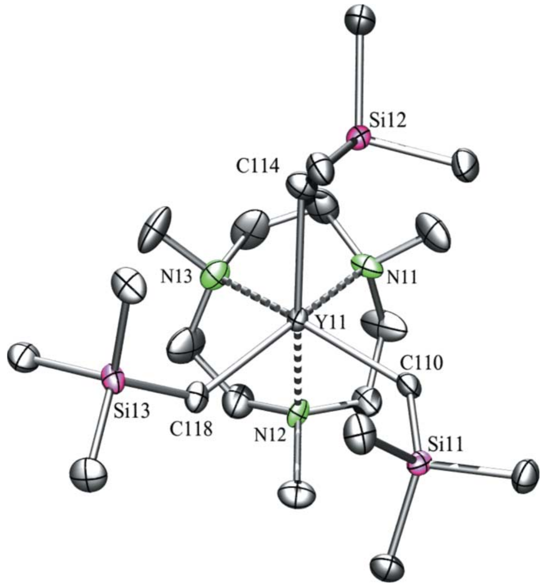


Figure 2
A view down the approximate C_3 axis of one of the independent molecules of (I), showing the staggered conformation of the Me$_3$-TACN ligand and the Y-bound alkyl groups.

methyl substituents. This conformation leaves (I) with approximate C_3 symmetry. The average Y—C bond distance, 2.427 Å, is similar to that in the octahedral trisolvate complex [Y(CH$_2$SiMe$_3$)$_3$(THF)$_3$] (Evans *et al.*, 2001).

Experimental

All preparations were performed under an inert nitrogen atmosphere, using standard Schlenk and glove-box techniques. To a suspension of YCl$_3$(THF)$_{3.5}$ (0.93 g, 2.10 mmol) in tetrahydrofuran (THF, 30 ml, ambient temperature), Me$_3$-TACN (0.36 g, 2.13 mmol) and LiCH$_2$SiMe$_3$ (0.56 g, 6.00 mmol) were added. After 3 h, the solvent was removed under reduced pressure and the residue was stripped of residual THF by stirring with pentane (5 ml), which was subsequently removed in a vacuum. Extraction with pentane (2 × 50 ml) and subsequent concentration and cooling of the extract to 243 K yielded 0.56 g (1.08 mmol, 54%) of the product as colourless crystals. Analysis calculated for C$_{21}$H$_{54}$N$_3$Si$_3$Y: C 48.33, H 10.43, N 8.05%; found: C 47.75, H 10.31, N 7.81%. ^{1}H NMR (300 MHz, 293 K, C$_6$D$_6$, δ, p.p.m.): 2.33 (*s*, 9H, Me$_3$-TACN), 2.29–2.22 (*m*, 6H, NCH$_2$), 1.67–1.62 (*m*, 6H, NCH$_2$), 0.43 (*s*, 27H, CH$_2$SiMe$_3$), −0.63 (*d*, J_{YH} = 2.70 Hz, 6H, YCH$_2$); ^{13}C NMR (125.7 MHz, C$_6$D$_6$, δ, p.p.m.): 54.4 (*t*, J_{CH} = 133.00 Hz, NCH$_2$), 47.8 (*q*, J_{CH} = 135.49 Hz, Me$_3$-TACN), 35.2 (*dt*, J_{YC} = 35.43 Hz, J_{CH} = 96.36 Hz, YCH$_2$), 4.9 (*q*, J_{CH} = 117.17 Hz, YCH$_2$SiMe$_3$).

Crystal data

[Y(C$_4$H$_{11}$Si)$_3$(C$_9$H$_{21}$N$_3$)]
M_r = 521.85
Monoclinic, $P2_1/c$
a = 16.164 (1) Å
b = 30.672 (2) Å
c = 18.290 (1) Å
β = 90.030 (1)°
V = 9067.9 (10) Å^3
Z = 12
D_x = 1.147 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5301 reflections
θ = 2.3–25.3°
μ = 2.06 mm^{-1}
T = 100 (1) K
Block, colourless
0.21 × 0.20 × 0.11 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.672, T_{max} = 0.805
59585 measured reflections
21757 independent reflections
12842 reflections with $I > 2\sigma(I)$
R_{int} = 0.107
θ_{max} = 28.3°
$h = -21 \rightarrow 21$
$k = -40 \rightarrow 40$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.142
S = 0.93
21757 reflections
794 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2 + 9.8014P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Y11—N11	2.606 (5)	Y22—C210	2.450 (7)
Y11—N12	2.612 (5)	Y22—C214	2.383 (7)
Y11—N13	2.587 (5)	Y22—C218	2.451 (7)
Y11—C110	2.422 (6)	Y33—N33	2.603 (5)
Y11—C114	2.449 (6)	Y33—C310	2.461 (6)
Y11—C118	2.412 (6)	Y33—C314	2.382 (7)
Y22—N21	2.572 (5)	Y33—C318	2.419 (7)
Y22—N22	2.628 (5)	Y33—N32	2.624 (5)
Y22—N23	2.594 (5)	Y33—N31	2.576 (5)
N11—Y11—N12	67.76 (16)	C310—Y33—C314	101.6 (2)
N11—Y11—N13	67.00 (17)	C310—Y33—C318	103.1 (2)
N12—Y11—N13	67.84 (16)	N32—Y33—N33	68.66 (15)
C110—Y11—C114	102.7 (2)	N31—Y33—N32	67.66 (16)
C110—Y11—C118	101.4 (2)	N31—Y33—N33	68.51 (17)
C114—Y11—C118	108.7 (2)	C314—Y33—C318	102.2 (2)
C210—Y22—C214	102.3 (2)	Y11—C110—Si11	128.9 (3)
C210—Y22—C218	103.8 (2)	Y11—C114—Si12	133.4 (3)
C214—Y22—C218	101.5 (2)	Y11—C118—Si13	136.4 (3)
N21—Y22—N22	67.43 (17)	Y22—C210—Si21	126.0 (3)
N21—Y22—N23	68.52 (17)	Y22—C214—Si22	154.8 (3)
N22—Y22—N23	68.14 (16)	Y22—C218—Si23	130.6 (3)

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Some atoms showed unrealistic displacement parameters, suggesting some degree of disorder; this is in line with the observation of broad reflections and the relatively high internal consistency. The $wR2$ value did not drop on anisotropic refinement, suggesting the possibility of twinning. The β-angle similarly indicated a possible twin. After introducing a twin matrix $[100, 0\bar{1}0, 00\bar{1}]$, corresponding to 180° rotation about the a axis [suggested by *PLATON* (Spek, 2003)], with scale factors for the fractional contributions of the various twin components, the structure refined smoothly, although a small number of unusual anisotropic displacement parameters remained. The major twin component contribution refined to a value of 0.529 (1). The H atoms were included in the final refinement riding on their carrier atoms, with their positions calculated using sp^2 or sp^3 hybridization at the C atom, as appropriate, and with $U_{\rm iso}(\rm H) = cU_{\rm eq}$ of their parent atom, where $c = 1.2$ for the non-methyl H atoms and $c = 1.5$ for the methyl H atoms. The methyl groups were refined as rigid groups, which were allowed to rotate freely.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* and *XPREP* (Bruker, 2000); program(s) used to solve structure: *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLUTO* (Meetsma, 2001) and *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON* and *SHELXL97*.

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