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A sterically crowded ytterbium(II) thiolate: *trans*-bis(2,4,6,2″,4″,6″-hexaisopropyl-1,1′:3′,1″-terphenyl-2′-thiolato-*S*)tetrakis(tetrahydrofuran-*O*)ytterbium(II)

Mark Niemeyer

Institut für Anorganische Chemie, Universität Stuttgart, Pfaffenwaldring 55, D-70569 Stuttgart, Germany

Correspondence e-mail:
niemeyer@iac.uni-stuttgart.de

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In the title complex [Yb(SAr*)$_2$(thf)$_4$] (Ar* is 2,6-Trip$_2$C$_6$H$_3$, $C_{36}H_{49}S$, where Trip is 2,4,6-^{i}Pr$_3$C$_6$H$_2$; thf is tetrahydrofuran, C_4H_8O), the Yb atom occupies a special position on the inversion center. It shows a slightly distorted *trans*-octahedral environment formed by the O atoms of four tetrahydrofuran ligands (average Yb—O 2.400 Å) and the S atoms of two thiolate ligands [Yb—S 2.8023 (13) Å]. The overcrowding caused by the sterically encumbered Ar* ligands is reflected in the large Yb—S—C angle of 151.16 (16)°.

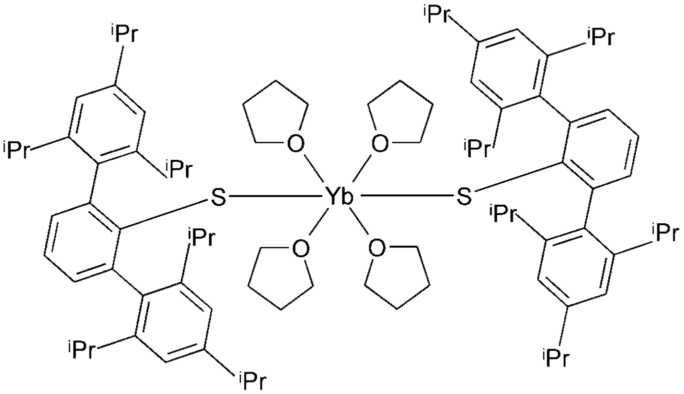
Key indicators

Single-crystal X-ray study
T = 188 K
Mean σ(C–C) = 0.008 Å
R factor = 0.052
wR factor = 0.128
Data-to-parameter ratio = 17.8

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

Comment

As a continuation of our work on low-coordinate σ-bonded rare earth compounds, we have recently reported (Niemeyer, 2001) the synthesis and structural characterization of the σ-donor-free *m*-terphenyl-based metal thiolates M(SAr*)$_2$ (M = Eu, Yb; Ar* = 2,6-Trip$_2$C$_6$H$_3$; Trip = 2,4,6-^{i}Pr$_3$C$_6$H$_2$). The preparation of these compounds by a one-pot direct-synthesis/protolysis route involves the synthesis of the solvated complexes M(SAr*)$_2$(thf)$_n$. Remarkably, the coordinated tetrahydrofuran can be removed completely either by crystallization from weakly coordinating solvents (M = Eu) or by evacuation at ambient temperature (M = Yb). However, in the case of the ytterbium derivative, prolonged storage at low temperature leads to crystallization of the intact solvate.



(I)

The title complex, (I), comprises a six-coordinate Yb atom, occupying a special position on the inversion center. It is bonded to the S atoms of two thiolate ligands in *trans* positions, as well as to the O atoms of four tetrahydrofuran ligands. The symmetry-independent half of the molecule of the complex is shown in Fig. 1. The full molecule (without the isopropyl groups, which were omitted for clarity) is depicted in Fig. 2. The Yb—S distance of 2.8023 (13) Å is considerably

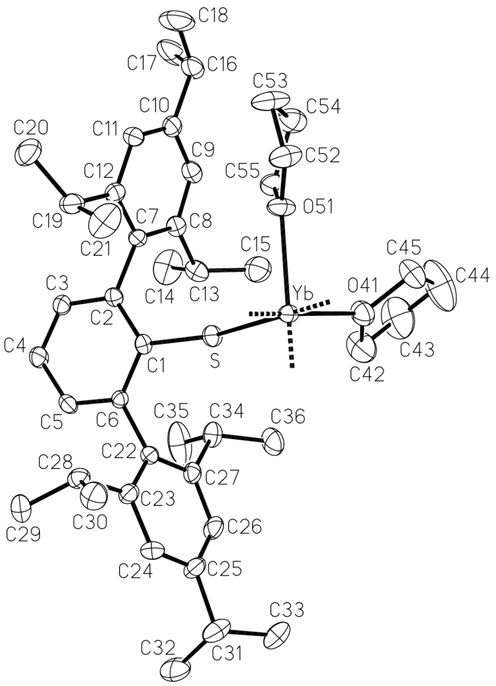


Figure 1
Displacement ellipsoid plot (at the 40% probability level) for the asymmetric unit of (I). H atoms have been omitted for clarity. Bonds from the Yb atom to symmetry-related atoms are indicated by broken lines.

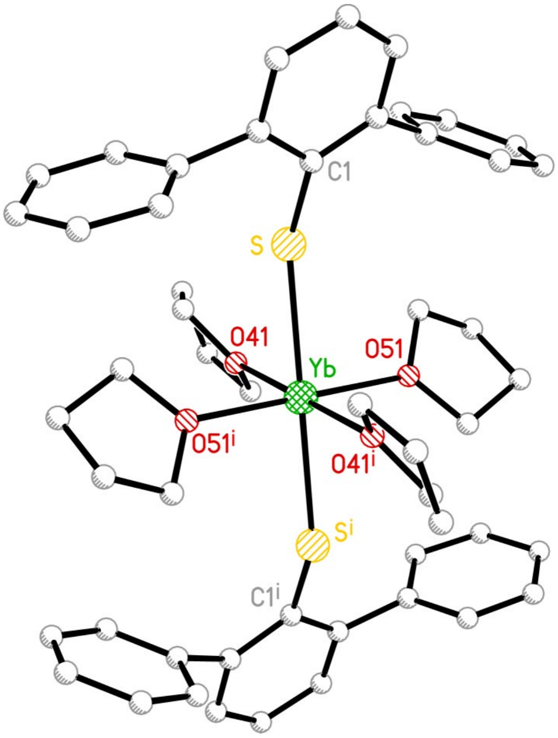


Figure 2
The molecular structure of (I). H atoms and isopropyl C atoms have been omitted for clarity.

longer than the distance of 2.691 Å which was observed in donor-free Yb(SAr*)$_2$ (Niemeyer, 2001). It may be compared with the Yb—S bond lengths in other six-coordinate σ-donor stabilized Yb^{II} thiolates which were reported as 2.739 Å in Yb(SAr*)$_2$(dme)$_2$ (Niemeyer, 2001), 2.756 Å in Yb(SMes*)$_2$(dme)$_2$ (Mes* = 2,4,6-tri-*tert*-butylphenyl; Cetinkaya *et al.*, 1992) and 2.827 Å in Yb(SPh)$_2$(py)$_4$ (Brewer *et al.*, 1994). The Yb—S—C1 angle in (I) is widened to 151.16 (16)°, obviously due to overcrowding caused by the sterically encumbered Ar* ligands.

Experimental

The title compound was synthesized and characterized as described previously (Niemeyer, 2001). Yellow crystals were obtained after storing a heptane/tetrahydrofuran (4:1) solution at 258 K for several months.

Crystal data

[Yb(C$_{36}$H$_{49}$S)$_2$(C$_4$H$_8$O)$_4$]
M_r = 1489.08
Monoclinic, $P2_1/n$
a = 13.666 (3) Å
b = 16.277 (3) Å
c = 18.575 (4) Å
β = 98.99 (3)°
V = 4081.0 (14) Å^3
Z = 2

D_x = 1.212 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 50 reflections
θ = 16.7–25.1°
μ = 1.24 mm^{-1}
T = 188 (2) K
Plate, yellow
0.45 × 0.40 × 0.10 mm

Data collection

Rebuilt Syntex $P2_1$/Siemens $P3$ four-circle diffractometer
Wyckoff scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min}$ = 0.604, $T_{\max}$ = 0.886
9278 measured reflections
8906 independent reflections
6008 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.053
$\theta_{\max}$ = 27.0°
$h = 0 \rightarrow 17$
$k = -20 \rightarrow 0$
$l = -23 \rightarrow 23$
2 standard reflections every 198 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.128
S = 1.08
8906 reflections
499 parameters

Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.76 e Å^{-3}
$\Delta\rho_{\min}$ = −1.04 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Yb—S	2.8023 (13)	S—C1	1.767 (5)
Yb—O41	2.408 (4)	C1—C2	1.413 (6)
Yb—O51	2.392 (3)	C1—C6	1.426 (6)
O51—Yb—O41	86.70 (14)	C2—C1—C6	117.8 (4)
O51—Yb—S	91.77 (9)	C2—C1—S	121.1 (3)
O41—Yb—S	84.26 (10)	C6—C1—S	120.8 (3)
C1—S—Yb	151.16 (16)		

The H atoms were placed in the idealized positions and refined in a riding model approximation, including free rotation for methyl groups. For most H atoms, the assigned $U_{\rm iso}$ was allowed to refine freely. Isotropic displacement parameters larger than 0.15 Å^2 were constrained to 1.2$U_{\rm eq}$ (CH, CH$_2$) or 1.5 $U_{\rm eq}$ (CH$_3$) of the parent atom.

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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