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trans-Diiodopentakis(tetrahydrofuran)ytterbium(III) tetraiodo-*trans*-bis(tetrahydrofuran)ytterbium(III)

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Key indicators

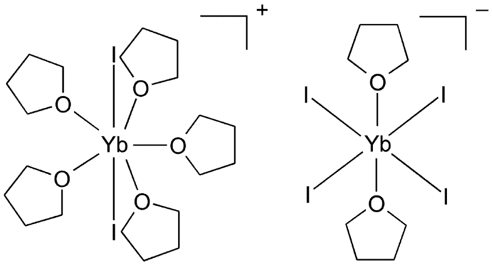
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.015 Å
R factor = 0.047
wR factor = 0.126
Data-to-parameter ratio = 23.9

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

The ionic title complex, $[YbI_2(thf)_5][YbI_4(thf)_2]$, was obtained by oxidation of the arylytterbium(II) iodide $DppYbI(thf)_3$ ($Dpp = 2,6\text{-}Ph_2C_6H_3$) with neopentyl iodide. In the pentagonal-bipyramidal coordinated cation [Yb—I = 2.9366 (9) Å and Yb–O = 2.340 (6), 2.347 (8) and 2.365 (6) Å], a twofold rotation axis passes through the Yb atom, the O atom and the midpoint of the C—C bond of one of the thf ligands. The centrosymmetric anion shows an almost regular coordination octahedron [Yb—I = 2.9641 (8) and 2.9856 (9) Å, and Yb—O 2.279 (6) Å].

Comment

In continuation of our work on σ-bonded rare earth organyls we have recently reported the synthesis and structural characterization of the *m*-terphenyl-substituted compound $DppYbI(thf)_3$ ($Dpp = 2,6\text{-}Ph_2C_6H_3$) (Heckmann & Niemeyer, 2000). The oxidation of this Grignard-analogous complex with neopentyl iodide did not produce the desired organylytterbium(III) diiodide (Niemeyer, 2000). Instead dichroic crystals of the title complex, (I), were isolated as orange/pale-yellow plates. Complex (I) is isomorphous with its samarium analogue $[SmI_2(thf)_5][SmI_4(thf)_2]$ (Xie *et al.*, 1996). In contrast, THF-solvated lanthanum(III) iodide crystallizes as the neutral coordination compound $LaI_3(thf)_4$ (Trifonov *et al.*, 1997).



(I)

The crystal structure of (I) consists of well-separated alternating layers of $[YbI_2(thf)_5]^+$ cations and $[YbI_4(thf)_2]^-$ anions. In the seven-coordinate ytterbium cation a crystallographic twofold rotation axis passes through Yb1, O11, and the midpoint of the C13—C13^i bond. The coordinated iodo ligands and thf molecules occupy respectively the axial and equatorial positions of a rather undistorted (O—Yb1—O 72±0.5°; O—Yb1—I 90±0.8°) pentagonal bipyramid. An almost regular coordination octahedron is observed for the centrosymmetric $[YbI_4(thf)_2]^-$ anion. The Yb1—I1 distance of

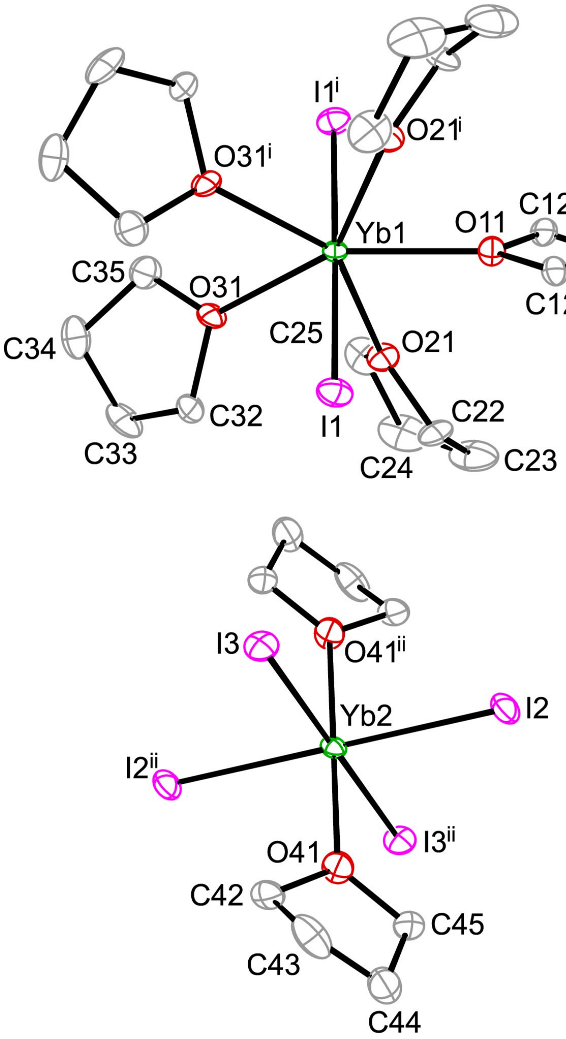


Figure 1
Displacement ellipsoid plot (40% probability) of the cation and anion in the crystal of (I). H atoms have been omitted for clarity.

2.9366 (9) Å in the seven-coordinate cation is significantly shorter than the average Yb2—I bond of 2.9749 Å in the six-coordinate anion. This can be explained by a higher positive partial charge on the ytterbium center in the former (see *Experimental*). In contrast, the average Yb1—O distance of 2.351 Å is considerably longer than the Yb2—O41 bond length of 2.279 (6) Å. The latter is in the normal range for a *trans*-O(thf)—Yb^{III}—O(thf) fragment on a six-coordinate metal center.

Experimental

The title compound was synthesized by the reaction of DppYbI(thf)$_3$ (1.33 g, 1.78 mmol) (Heckmann & Niemeyer, 2000) with neopentyl iodide (0.24 ml, 1.81 mmol) in 30 ml of thf solution. After cooling the solution to 263 K for 4 months, dichroic orange/pale-yellow crystals (0.35 g, 0.22 mmol, 14%) were obtained. A population analysis, based on the X-ray geometry (C—H distances set to 1.09 Å; 6-31G* basis sets for C, H, and O; quasi-relativistic pseudo-potentials of the Stuttgart/Bonn group for Yb and I), was calculated on the HF level of theory for the isolated cation and anion to give the following Mulliken charges: Yb1 +2.014, Yb2 +1.687.

Crystal data

$[YbI_2(C_4H_8O)_5][YbI_4(C_4H_8O)_2]$
$M_r = 1612.21$
Monoclinic, $C2/c$
$a = 12.722\ (2)$ Å
$b = 12.037\ (3)$ Å
$c = 28.551\ (4)$ Å
$\beta = 98.05\ (1)^\circ$
$V = 4329.1\ (13)$ Å^3
$Z = 4$

$D_x = 2.474$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20 reflections
$\theta = 8.5$–12.0°
$\mu = 8.61$ mm^{-1}
$T = 173\ (2)$ K
Plate, orange
0.32 × 0.22 × 0.12 mm

Data collection

Rebuilt Syntex $P2_1$/Siemens $P3$ four-circle diffractometer
Wyckoff scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.404$, $T_{\max} = 1.000$
4945 measured reflections
4733 independent reflections
3643 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.050$
$\theta_{\max} = 27.0^\circ$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 15$
$l = -36 \rightarrow 36$
2 standard reflections every 198 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.126$
$S = 1.14$
4733 reflections
198 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0707P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 3.10$ e Å^{-3}
$\Delta\rho_{\min} = -3.10$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Yb1—I1	2.9366 (9)	Yb2—I2	2.9641 (8)
Yb1—O11	2.347 (8)	Yb2—I3	2.9856 (9)
Yb1—O21	2.365 (6)	Yb2—O41	2.279 (6)
Yb1—O31	2.340 (6)		
I1—Yb1—I1^i	179.44 (3)	O31—Yb1—I1	90.37 (14)
O11—Yb1—O21	71.64 (16)	O21^i—Yb1—I1	90.45 (16)
O21—Yb1—O31	72.1 (2)	O31^i—Yb1—I1	89.17 (14)
O31—Yb1—O31^i	72.5 (3)	I2—Yb2—I3	90.98 (2)
O11—Yb1—I1	90.28 (16)	O41—Yb2—I2	89.19 (17)
O21—Yb1—I1	89.73 (16)	O41—Yb2—I3	92.29 (19)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

The H atoms were positioned with idealized geometry (C—H = 0.99 Å) and refined as riding. The highest residual densities of 3.10, 2.72, and 2.21 e Å^{-3} were located near Yb2, Yb1 and I3, respectively.

Data collection: *P*3 (Siemens, 1989); cell refinement: *P*3; data reduction: *XDISK* (Siemens, 1989); structure solution: *SHELXS*97 (Sheldrick, 1997); structure refinement: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998).

References

Heckmann, G. & Niemeyer, M. (2000). *J. Am. Chem. Soc.* **122**, 4227–4228.
Niemeyer, M. (2000). *Z. Anorg. Allg. Chem.* **626**, 1027–1029.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sheldrick, G. M. (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1989). *P*3 and *XDISK*. Release 4.1. Siemens Analytical Instruments Inc., Madison, Wisconsin, USA.
Trifonov, A. A., Van de Weghe, P., Collin, J., Domingos, A. & Santos, I. (1997). *J. Organomet. Chem.* **527**, 225–237.
Xie, Z., Chiu, K.-Y., Wu, B. & Mak, T. C. W. (1996). *Inorg. Chem.* **35**, 5957–5958.