

Trans influence in a *mer*-octahedral triiodidolanthanide: triiodidotris(tetrahydrofuran- κ O)ytterbium(III)

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Received 10 September 2009

Accepted 21 September 2009

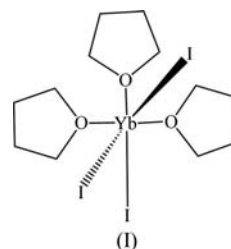
Online 10 October 2009

The structure of the six-coordinate title complex, $[\text{YbI}_3(\text{C}_4\text{H}_8\text{O})_3]$, is the first *mer*-octahedral form of an LnI_3L_3 lanthanide (Ln) compound with neutral *L* ligands, and is closely related to that of several of the seven-coordinate LnX_3L_4 series of compounds, where $X = \text{Cl}, \text{Br}$ or I and $L =$ tetrahydrofuran (THF), isopropanol, pyridine or water. A structural *trans* effect can be assigned to $\text{YbI}_3(\text{THF})_3$, in contrast to the LnX_3L_4 compounds, where steric and crystal packing effects are significant. The $\text{Yb}-\text{I}$ bond lengths are 2.9543 (4) and 2.9151 (6) Å for *I trans* and *cis* to *I*, respectively, and the $\text{Yb}-\text{O}$ bond lengths are 2.299 (5) and 2.251 (3) Å for *O trans* and *cis* to *I*, respectively. The crystal packing allows for six contact distances as weak $\text{C}-\text{H}\cdots\text{I}$ interactions in the range 3.10–3.24 Å. The title molecule has a crystallographic twofold axis passing through a THF O atom, the *trans* I atom and the Yb atom.

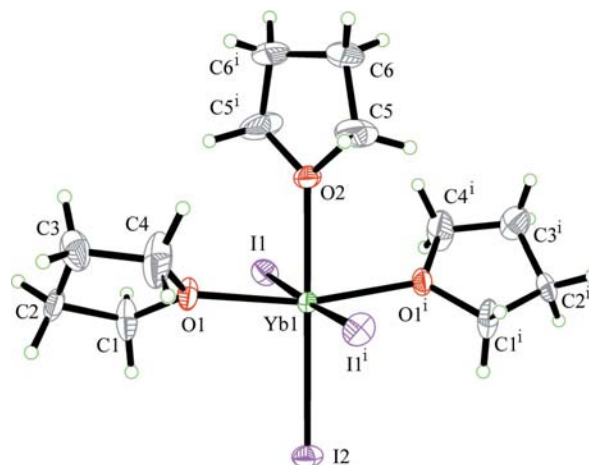
Comment

The crystal structures of many lanthanide (Ln) halide (*X*) compounds with one or more neutral ligands (*L*) are well known. For compounds with $X = \text{Cl}, \text{Br}$ or I and small to moderate size neutral Lewis base ligands like tetrahydrofuran (THF), 1,2-dimethoxyethane (DME), pyridine (py), hexamethylphosphoramide $[\text{O}=\text{P}(\text{NMe}_2)_3]$, isopropyl alcohol (*i*-PrOH) and water (H_2O), the most likely species to crystallize is the seven-coordinate $\text{LnX}_n\text{L}_{7-n}$. This situation is apparently the case whether the lanthanide is di- or trivalent, where the number of neutral organic ligands is usually five or four, respectively. Excluding LnX_6 ions, there are only 18 six-coordinate monomeric LnX_3L_3 species with *L* as described above in the Cambridge Structural Database (CSD) to date (Version 5.30; Allen, 2002). However, the variety of Ln atom is not diminished, and the size of the trivalent lanthanide radii appears to play no role in allowing octahedral coordination, as LnX_3L_3 compounds are observed with Ln = La, Ce, Pr, Nd, Sm, Eu, Gd, Dy, Yb and Lu, and $X = \text{Cl}$, except for one $X = \text{Br}$ compound, $\text{YbBr}_3(\text{THF})_3$ (Deacon *et al.*, 2000). Additionally,

the organic ligands (*L*) may be small (*e.g.* H_2O , py or THF) or large (*e.g.* triphenylarsine oxide; Ryan *et al.*, 1987) and still yield a six-coordinate compound. This group of compounds includes the two $\text{LnX}_3(\text{THF})_3$ analogs most closely related to the title compound, (I) (Fig. 1), where Ln/*X* = Lu/Br (Mago-medov *et al.*, 1992) or Yb/Cl (Deacon *et al.*, 1993).



For the six-coordinate LnX_3L_3 compounds, both *fac* and *mer* isomers are found in the CSD for $X = \text{Cl}$, but only *mer* isomers are found for $X = \text{Br}$, presumably as a result of the increased size of the bromide ion compared with the chloride ion. The title compound is the only six-coordinate triiodido species of this type, and a *trans* influence on bond geometries is evident, as shown by the data in Table 1. Other *mer*-octahedral trihalides of this type with significant *trans* influence are *mer*- $\text{YbBr}_3(\text{THF})_3$ (Deacon *et al.*, 2000) and *mer*- $\text{SmBr}_3[\text{O}=\text{P}(\text{NMe}_2)_3]_2\text{THF}$ (Asakura & Imamoto, 2001). Of the seven *mer* isomers, five are LnCl_3L_3 , *viz.* *mer*- $\text{YbCl}_3(\text{THF})_3$ (Qian *et al.*, 1993), *mer*- $\text{LuCl}_3(\text{THF})_3$ (Mago-medov *et al.*, 1992), *mer*- $\text{DyCl}_3[\text{O}=\text{P}(\text{NMe}_2)_3]_3$ (Zhang *et al.*, 1987), *mer*- $\text{PrCl}_3[\text{O}=\text{P}(\text{NMe}_2)_3]_3$ (Radonovich & Glick, 1973) and *mer*- $\text{YbCl}_3[\text{O}=\text{P}(\text{NMe}_2)_3]_3$ (Hou *et al.*, 1991), and two are LnBr_3L_3 , *viz.* *mer*- $\text{YbBr}_3(\text{THF})_3$ and *mer*- $\text{SmBr}_3[\text{O}=\text{P}(\text{NMe}_2)_3]_2\text{THF}$. The *fac* isomers of six-coordinate LnX_3L_3 usually have larger *L* ligands, as in *fac*-trichloridotris(3-bromo-4-methoxy-lutidine *N*-oxide-*O*)praseodymium (Ban-Oganowska *et al.*, 2002), *fac*-trichloridotris(triphenylarsine oxide)cerium (Ryan *et al.*, 1987), *fac*-trichloridotris(2,6-dimethyl-4-pyrone)gadolinium (Bisi Castellani & Tazzoli, 1984), *fac*-trichloridotris(hexamethylphosphoramide-*O*)samarium (Petricek *et al.*,


Figure 1

The molecular structure of $\text{YbI}_3(\text{THF})_3$, with displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $-x, y, -z + \frac{1}{2}$]

2000), *fac*-trichlorido(caprolactonato-*O*)bis(tetrahydrofuran)-ytterbium (Evans *et al.*, 1995) and *fac*-trichloridotris[bis-(diethylamido)trichloroacetylamidophosphoric acid]praseodymium (Amirkhanov *et al.*, 1995). Instances of *fac*-Ln-Cl₃[O=P(NMe₂)₃]₃ are also known, namely, the isostructural series Ln = La, Pr, Sm, Eu, Nd and Gd (Petricek *et al.* 2000). The only instance of a seven-coordinate Ln₃X₃ compound approximating a *fac* isomer is when the sterically constrained tripodal ligand tris(2-pyridylmethyl)amine is used in a triiodidocerium complex (Natrajan *et al.*, 2005).

When bidentate DME ligand is used, the seven-coordinate compound is most likely to be produced, and there are over 80 instances in the CSD of LnX₃Y₄, where Y is the binding O atom (or N atom for di-, tri- or tetraamine) of a bidentate organic ligand. Unlike the Ce compound mentioned above, about 20 are of the type LnX₃(DME)₂, with X = Cl or Br only. There is one other LnI₃Y₄ compound, where Y are O atoms from a multidentate ligand, namely tetraglyme (Vestergren *et al.*, 2004). In a broad sense, the nine compounds in the CSD of the type LnI₃L₄, where L are not multidentate, approximate the *mer* conformation (namely, having one nearly linear I—Ln—I angle and two ~90° I—Ln—I angles), even though there is seven-coordinate binding. This situation is presumably also due to the large volume occupied by each iodide ligand and to the fact that two I atoms are axial in the pentagonal bipyramidal LnI₃L₄ complexes.

In the search for LnI₃(Y = O, N)_n species, the CSD revealed no six-coordinate, one nine-coordinate, five eight-coordinate and ten seven-coordinate compounds, with five compounds having tripodal ligands. For LnI₃L_n and LnBr₃L_n, neither L = py nor L = H₂O species are found in the CSD. There are four LnI₃(THF)₄ compounds in the CSD; their Ln—I and Ln—O distances are compared with those of the title compound in Table 2, which shows a persistent *trans* influence on the O atom of the two THF ligands furthest from the equatorial I atom (the other two I atoms are *trans* to each other). However, as seven-coordinate pentagonal bipyramidal compounds with five equatorial ligands, these LnI₃(THF)₄ compounds appear to have geometries significantly influenced by steric effects, especially with respect to the equatorial I ligand, such that the *cis* (e.g. equatorial) Ln—I bond is about 0.06 Å longer than either of the *trans* Ln—I bonds.

For LnBr₃(O, N)_n compounds, there are 11 seven-coordinate, one six-coordinate and one eight-coordinate species in the CSD, with five having tripodal ligands. Of these, one is YbBr₃(THF)₃ (Deacon *et al.*, 2000) and four are LnBr₃(THF)₄. Details of their lanthanide bond geometries, as well as those of SmBr₃(*i*-PrOH)₄ (Depero *et al.*, 1991) and NdBr₃(THF)₂(*i*-PrOH)₂ (with THF ligands nearly *trans* to the equatorial Br atom; Hong *et al.*, 1994), are also compared with the Yb—I and Yb—O bonds of the title compound in Table 3. There are many instances of LnCl₃L₃ and LnCl₃L₄, so bond comparisons here will be restricted to only L = THF, py and H₂O, the smallest ligands in the series, and these compounds are included in Table 2.

For lanthanides with coordination saturated with halogens, only the LnX₆ ions are found in the CSD. For LnX₆, the range

of Ln—X bonds for any Ln and X is about ±0.02 Å from the average, which is not very narrow, even though these compounds are simple octahedra, indicating the likelihood of short-range nonsymmetrical crystal packing effects. There is one reported X = Br species, EuBr₆, and the Eu—Br bonds are 2.80 (2) Å (Pellens *et al.*, 2008); there are seven LnI₆ [Ln = La, Pr, Nd (three examples), Sm and Er] compounds, with La—I bonds of 3.16 (2) Å (Babai & Mudring, 2006a), Pr—I bonds of 3.13 (2) Å (Babai & Mudring, 2005b), Nd—I bonds of 3.11 (2) Å (Babai & Mudring, 2005a; Babai & Mudring, 2006b), Sm—I bonds of 3.09 (2) Å (Babai & Mudring, 2005a) and Er—I bonds of 3.03 (1) Å (Babai & Mudring, 2006a); and there are 35 LnCl₆ with an overall average Ln—Cl bond of 2.68 (2) Å and a range of 2.51–2.89 for the entire Ln series, but with a similar 0.01–0.03 Å variation from the mean for any given compound.

In summary, the YbI₃(THF)₃ complex reported here is the first instance of a six-coordinate triiodidolanthanide. In addition, the observed *mer* conformation allows for the possibility of a *trans* influence, which is rarely seen in lanthanide compounds. The bonds affected are the Yb—I and Yb—O bonds, which are 0.03 and 0.05 Å longer when *trans* to an I atom. Finally, six H···I contact distances in the range 3.10 to 3.24 Å are observed (Table 3), which are thus of the order of the van der Waals radii sum (3.18 Å; Bondi, 1964). Although these distances are consistent with weak hydrogen-bond interactions, the THF ligand is traditionally expected to be a poor hydrogen-bond donor.

Experimental

All syntheses were carried out under ultrapure nitrogen (JWS), using conventional dry-box or Schlenk techniques. Solvents (Fisher) were refluxed continuously over molten alkali metals or K/benzophenone and were collected immediately prior to use. Yb was purchased from Strom. PhSeSePh was purchased from Aldrich and recrystallized from hexane. For the synthesis of the title compound, Yb (2.0 mmol), PhSeSePh (2.0 mmol), iodine (1.0 mmol) and Hg (0.25 mmol) were combined in THF (50 ml). The mixture was stirred until all the metal was consumed. To the resulting yellow solution, elemental S (2.0 mmol) was added. After 24 h, the yellow solution was filtered and layered with hexanes (15 ml) to give yellow crystals. This procedure was originally designed to yield the Yb analog of an Er 'double-cubane' cluster compound, *viz.* (THF)₁₀Er₆S₆I₆·6(THF) (Kornienko *et al.*, 2005).

Crystal data

[YbI ₃ (C ₄ H ₈ O) ₃]	V = 1956.9 (3) Å ³
M _r = 770.05	Z = 4
Orthorhombic, <i>Pbcn</i>	Mo Kα radiation
a = 9.3507 (7) Å	μ = 9.51 mm ⁻¹
b = 14.6003 (11) Å	T = 100 K
c = 14.3335 (11) Å	0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer	8941 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2003)	2402 independent reflections
T _{min} = 0.22, T _{max} = 0.41	1808 reflections with I > 2σ(I)
	R _{int} = 0.037

Table 1

 Selected metal atom bond lengths (Å) and angles (°) for YbI₃(THF)₃.

Yb1—I1	2.9543 (4)	Yb1—O1	2.251 (3)
Yb1—I2	2.9151 (6)	Yb1—O2	2.299 (5)
I1—Yb1—I1 ⁱ	177.044 (18)	O2—Yb1—I2	180.0
I1—Yb1—I2	91.478 (9)	O1 ⁱ —Yb1—O1	166.1 (2)

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

Table 2

 Average Ln—X and Ln—Y distances *trans/cis* to atom X or Y for LnX₃L₃ and LnX₃L₄, where X = I, Br and Cl, Y = O and N, and L = THF, py, *i*-PrOH, H₂O and O=P[N(Me₂)₃].

The estimates of uncertainty in the distances are in the range 0.001–0.007 Å.

Average <i>trans/cis</i> Ln—(I, Br or Cl) (Å)	Average <i>trans/cis</i> Ln—(O or N) (Å)	Neutral ligand, L	Reference
Six-coordinate compounds			
Yb—I 2.95/2.92	Yb—O 2.30/2.25	THF	This work
Yb—Br 2.71/2.66	Yb—O 2.33/2.26	THF	Deacon <i>et al.</i> (2000)
Sm—Br 2.85/2.82	Sm—O 2.47/2.28	THF	Asakura & Imamoto (2001)
Yb—Cl 2.53/2.51	Yb—O 2.36/2.27	THF	Qian <i>et al.</i> (1993)
Lu—Cl 2.53/2.50	Lu—O 2.32/2.27	THF	Magomedov <i>et al.</i> (1992)
Dy—Cl 2.73/2.71	Dy—O 2.35/2.35	<i>i</i> -PrOH	Radonovich & Glick (1973)
Yb—Cl 2.59/2.58	Yb—O 2.22/2.23	<i>i</i> -PrOH	Hou <i>et al.</i> (1991)
Seven-coordinate compounds			
La—I 3.14/3.19	La—O 2.56/2.52	THF	Trifonov <i>et al.</i> (1997)
Ce—I 3.12/3.18	Ce—O 2.54/2.50	THF	Liddle & Arnold (2005)
Pr—I 3.10/3.16	Pr—O 2.51/2.47	THF	Izod <i>et al.</i> (2004)
Nd—I 3.08/3.15	Nd—O 2.50/2.46	THF	Balashova <i>et al.</i> (2007)
La—I 3.20/3.21	La—O 2.51/2.51	<i>i</i> -PrOH	Barnhart <i>et al.</i> (1995)
Ce—I 3.17/3.19	Ce—O 2.48/2.49	<i>i</i> -PrOH	Barnhart <i>et al.</i> (1995)
La—Br 2.90/2.90	La—O 2.55/2.51	THF	Deacon <i>et al.</i> (2000)
Ce—Br 2.88/2.91	Ce—O 2.55/2.50	THF	Hitchcock <i>et al.</i> (2004)
Pr—Br 2.87/2.90	Pr—O 2.53/2.49	THF	Petricek (2004)
Sm—Br 2.82/2.86	Sm—O 2.49/2.44	THF	Petricek (2004)
Nd—Br 2.85/2.83	Nd—O 2.52/2.45	THF, <i>i</i> -PrOH	Hong <i>et al.</i> (1994)
Sm—Br 2.85/2.80	Sm—O 2.49/2.43	<i>i</i> -PrOH	Depero <i>et al.</i> (1991)
Eu—Cl 2.62/2.64	Eu—O 2.47/2.45	THF	Kong <i>et al.</i> (1998)
Gd—Cl 2.62/2.66	Gd—O 2.46/2.48	THF	Wiley <i>et al.</i> (1997)
Sm—Cl 2.68/2.68	Sm—O 2.49/2.45	THF	Lin <i>et al.</i> (1992)
Nd—Cl 2.67/2.67	Nd—O 2.52/2.48	THF	Chen <i>et al.</i> (1987)
La—Cl 2.66/2.68	La—N 2.62/2.59	py	Li <i>et al.</i> (2002)
Eu—Cl 2.64/2.67	Eu—N 2.61/2.57	py	Hu <i>et al.</i> (1986)
Er—Cl 2.58/2.62	Er—N 2.54/2.50	py	Li <i>et al.</i> (2002)
Yb—Cl 2.56/2.60	Yb—N 2.51/2.49	py	Deacon <i>et al.</i> (2006)
Er—Cl 2.60/2.66	Er—O 2.37/2.32	H ₂ O	Hines <i>et al.</i> (2008)
Tm—Cl 2.64/2.62	Tm—O 2.35/2.35	H ₂ O	Semenova <i>et al.</i> (2006)
Yb—Cl 2.63/2.62	Yb—O 2.33/2.26	H ₂ O	Semenova <i>et al.</i> (2006)
Lu—Cl 2.59/2.60	Lu—O 2.35/2.28	H ₂ O	Semenova <i>et al.</i> (2006)
Er—Cl 2.64/2.63	Er—O 2.35/2.29	H ₂ O	Semenova <i>et al.</i> (2006)

Table 3

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
C1—H1A...I1 ⁱⁱ	0.99	3.24	3.926 (6)	128
C1—H1B...I1	0.99	3.20	3.785 (6)	120
C3—H3B...I1 ⁱⁱⁱ	0.99	3.10	4.014 (6)	155
C4—H4A...I1 ⁱ	0.99	3.23	3.715 (8)	112
C4—H4B...I2 ^{iv}	0.99	3.22	3.879 (7)	126
C5—H5A...I1 ⁱ	0.99	3.22	3.706 (7)	112

 Symmetry codes: (i) $-x, y, -z + \frac{1}{2}$; (ii) $x + \frac{1}{2}, -y + \frac{1}{2}, -z$; (iii) $x + 1, y, z$; (iv) $x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$.

Refinement

$$R[F^2 > 2\sigma(F^2)] = 0.029$$

$$wR(F^2) = 0.066$$

$$S = 1.00$$

2402 reflections

88 parameters

H-atom parameters constrained

$$\Delta\rho_{\max} = 1.35 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.74 \text{ e } \text{Å}^{-3}$$

All H atoms were located in difference Fourier maps and then treated as riding atoms. H atoms bonded to the THF C atoms were allowed to ride in geometrically idealized positions, with C—H = 0.99 Å (CH₂) and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: SMART (Bruker, 2003); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL and publCIF (Westrip, 2009).

We acknowledge the NSF (grant No. CHE-0747165) and the New Jersey State Commission on Science and Technology for their generous support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG3217). Services for accessing these data are described at the back of the journal.

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