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Crystallographic report

Crystal structure of tris(2,6-di-*tert*-butyl-4-methylphenolato-*O*) (tetrahydrofuran-*O*) erbium toluene solvate

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A low-coordinate aryloxo erbium complex, $[(ArO)_3Er(THF)](MePh)$, has been synthesized by the reaction of anhydrous $ErCl_3$ with three equivalents of NaOAr in tetrahydrofuran. The central erbium atom is coordinated by three oxygen atoms of the aryloxo ligands and one oxygen atom of the tetrahydrofuran molecule, resulting in a distorted tetrahedron. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: erbium; aryloxo ligand; crystal structure; X-ray

COMMENT

In recent years, lanthanide alkoxides and aryloxides have found a variety of applications as homogeneous catalysts for organic reactions[1] and high purity oxide materials.[2,3] The syntheses and structural studies of aryloxo lanthanide complexes have revealed that the low-coordinate lanthanide complexes can be easily obtained using bulky aryloxo ligands.[4] Here, we report the synthesis and crystal structure of a low-coordinate aryloxo lanthanide complex, $[(ArO)_3Er(THF)](MePh)$ (ArO = 2,6-di-*tert*-butyl-4-methylphenolate; THF = tetrahydrofuran). X-ray structure analyses reveal that the title complex is isostructural with $[Sm(OAr)_3(THF)](THF)$[5] and $[Nd(OAr)_3(THF)](MePh)$.[6] The complex is a four-coordinate monomer with three aryloxo oxygen atoms and one THF oxygen atom around the erbium atom, forming a distorted tetrahedron; Fig. 1. One *tert*-butyl group on the arene ring is disordered due to strong thermal motion. The three aryloxo oxygen atoms around the erbium atom show a nearly trigonal planar array with $\sum O-Er-O = 351.6°$. The Er–O(Ar)

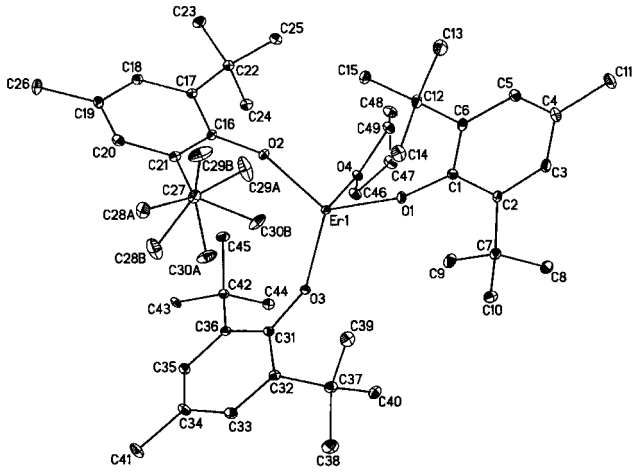

Figure 1. Molecular structure of the title complex. The disordered tert-butyl group is shown in two possible orientations. Key geometric parameters: Er(1)–O(1) 2.090(3), Er(1)–O(2) 2.068(3), Er(1)–O(3) 2.091(3), Er(1)–O(4) 2.300(3), O(1)–C(1) 1.363(5), O(2)–C(16) 1.371(5), O(3)–C(31) 1.361(5) Å; O(1)–Er(1)–O(2) 117.4(1), O(1)–Er(1)–O(3) 115.8(1), O(1)–Er(1)–O(4) 91.4(1), O(2)–Er(1)–O(3) 118.5(1), O(2)–Er(1)–O(4) 99.7(1), O(3)–Er(1)–O(4) 107.6(1), Er(1)–O(1)–C(1) 155.5(3), Er(1)–O(2)–C(16) 158.4(3), Er(1)–O(3)–C(31) 147.6(3)°.

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distances range from 2.068(3) to 2.091(3) Å, giving an average Er–O(Ar) distance of 2.083(3) Å. There is a toluene molecule in the unit cell.

EXPERIMENTAL

Synthesis of title complex

To a suspension of $ErCl_3$ (1.07 g, 3.91 mmol) in 20 ml THF was slowly added a solution of NaOAr (12.4 ml, 11.7 mmol) in THF at room temperature. After stirring for 72 h, the NaCl was separated from the reaction mixture by centrifugation. The solvent was removed in vacuum and toluene was added to extract the product. The dissolved portion was removed by centrifugation. The filtrate was concentrated and cooled at −20 °C for crystallization. Pink single crystals were obtained over a few days (yield 2.74 g, 78%); m.p. 113–115 °C (dec.). Anal. Found: C, 65.86; H, 8.42; Er, 18.43; Calc. for $Er(ArO)_3(THF)$: C, 65.59; H, 8.65; Er, 18.64%; IR absorptions (cm^{-1}): 2952(s), 2918(w), 2873(w), 1637(s), 1435(s), 1392(m), 1365(m), 1230(m), 1152(s), 1118(m), 1023(w), 864(s), 771(s). The crystals suitable for X-ray crystal structure studies were obtained by recrystallization from THF–toluene solution at −10 °C over a few days.

Crystallography

Intensity data for the title compound were collected at 193.1 K on a Rigaku Mercury CCD area detector for a pink crystal with dimensions of $0.60 \times 0.50 \times 0.11$ mm^3. Crystallographic data: $C_{56}H_{85}O_4Er$, $M = 989.50$, monoclinic, Cc (#9), $a = 25.544(3)$, $b = 10.5717(9)$, $c = 19.976(2)$ Å, $\beta = 103.226(5)°$, $V = 5251.2(9)$ $Å^3$, $D_c = 1.252$ g cm^{-3}, $Z = 4$; 25 669 data collected, 11 770 unique data ($3.09 \leq \theta \leq 27.48°$), 11 346 observed data with ($I > 2.00\sigma(I)$). $R_{obs} = 0.0361$, $wR = 0.776$ (all data), $\rho_{max} = 1.103 e^- Å^{-3}$, $\rho_{min} = -0.493$ e^- $Å^{-3}$. Programs used: SHELXS-97, SHELXL-97. CCDC number: 230124.

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