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Tris(2,6-di-t-butylphenoxo)yttrium: a Three-coordinate Hydrocarbon-soluble Aryloxide of Yttrium*

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The chemistry of the group 3 metal (Sc, Y, La, and the $4f^n$ metals; collectively here abbreviated as Ln) aryloxides is reasonably well documented [1]. Most group 3 metal aryloxides are involatile solids, insoluble in hydrocarbon solvents, and considered to be polymeric [2]. In 1983 it was reported that using the sterically hindered 2,6-di-t-butylphenoxo ligands $\bar{O}Ar^R$ ($Ar^R = C_6H_2Bu^t{}_2$-2,6-R-4; R = H, Me, or Bu^t), monomeric, volatile, hydrocarbon-soluble aryloxides, such as $[Ln(OAr^{Me})_3]$ (Ln = Sc, Y, La, Pr, Nd, Dy, Ho, Er, or Yb) could be prepared [3]; further synthetic details have recently been published [4]. X-ray data were previously available only on the scandium complex $[Sc(OAr^{Me})_3]$ [3]. We now report the synthesis and structural characterization of $[Y(OAr^H)_3]$, which appears to be the first X-ray-authenticated yttrium aryloxide [1].

Experimental

All operations were performed under argon using standard Schlenk techniques. Tetrahydrofuran (THF) was predried over sodium wire, freshly distilled from sodium/benzophenone, and degassed before use.

$YCl_3(THF)_3$ (2.00 g, 4.86 mmol) was added, with stirring, to $[Li(\mu\text{-}OAr^H)(OEt_2)]_2$ [5] (4.17 g, 7.3 mmol) in THF (30 ml) at room temperature. The reaction mixture was refluxed for 8 h. The volatiles were removed *in vacuo*, producing an off-white solid, which was sublimed at 250–255 °C and 10^{-3} Torr to produce colourless crystals of the sublimate $[Y(OAr^H)_3]$ (3.42 g, 71%). ^1H NMR chemical shifts (δ in ppm, relative to external $SiMe_4$, 80 MHz, C_6D_6, 305 K): δ 1.47(s) (Bu^t); 6.78(t) (*p*-H); 7.20(d) (*m*-H). *Anal.* Calc. for $C_{42}H_{63}O_3Y$ (M = 704.9): (C, 71.6; H, 8.95. Found: C, 71.8; H, 9.17%. X-ray quality single crystals were sealed under argon in a 0.3 mm diameter Lindemann tube.

Crystal Data

$C_{42}H_{63}O_3Y$, $M = 704.9$, monoclinic, space group $P2_1/c$, $a = 11.278(8)$, $b = 32.094(23)$, $c = 11.732(7)$ Å, $\beta = 104.89(5)°$, $U = 4103.9$ Å^3, $Z = 4$, $D_c = 1.14$ g cm^{-3}. Monochromated Mo Kα radiation, $\lambda = 0.71069$ Å, $\mu = 14.6$ cm^{-1}.

Data Collection and Processing

A crystal of dimensions 0.5 × 0.3 × 0.15 mm was mounted on an Enraf-Nonius CAD-4 diffractometer. Data collection was in the $\theta-2\theta$ mode, $\Delta\theta = (0.8 + 0.35 \tan\theta)°$. 3085 reflections were measured for $2 < \theta < 18°$, $+h + k \pm l$, and 1276 unique reflections with $|F^2| > \sigma(F^2)$ were used in the refinement, $\sigma(F^2) = [\sigma^2(I) + (0.04I)^2]^{1/2}/Lp$. No crystal decay was observed. No absorption correction was applied. The structure was solved by routine heavy atom methods, and was refined by full-matrix least-squares with the Y atom anisotropic. H atoms were omitted. The weighting scheme was $w = 1/\sigma^2(F)$, yielding final residuals $R = 0.104$, $R' = 0.113$. Programs used were from the Enraf-Nonius SDP-Plus package run on a PDP11/34 computer.

Results and Discussion

The yttrium complex $[Y(OAr^H)_3]$ (1) was prepared by refluxing anhydrous yttrium chloride with three equivalents of the lithium aryloxide [4]. This produced the THF adduct $Y(OAr^H)_3(THF)_n$. Subsequent sublimation afforded 1 in good yield.

The X-ray structure of the aryloxide (1) was determined using single crystal X-ray diffraction techniques. The results are summarized in Fig. 1 and Table I.

The Y atom is in a trigonal planar environment, coordinated by the three oxygen atoms. The deviation of the Y atom from the YO_3 plane is only 0.09 Å and the average of the three O–Y–O angles

TABLE I. Some Important Bond Lengths (A) and Angles (°) in $[Y(OC_6H_3Bu^t{}_2\text{-}2,6)_3]$ (1)

Bond lengths	
Y–O(1)	2.00(1)
Y–O(2)	2.00(2)
Y–O(3)	2.00(1)
Bond angles	
O(1)–Y–O(2)	119.3(5)
O(2)–Y–O(3)	117.5(6)
O(1)–Y–O(3)	122.5(6)
Y–O(1)–C(1)	175(1)
Y–O(1)–C(15)	173(1)
Y–O(3)–C(29)	171(1)

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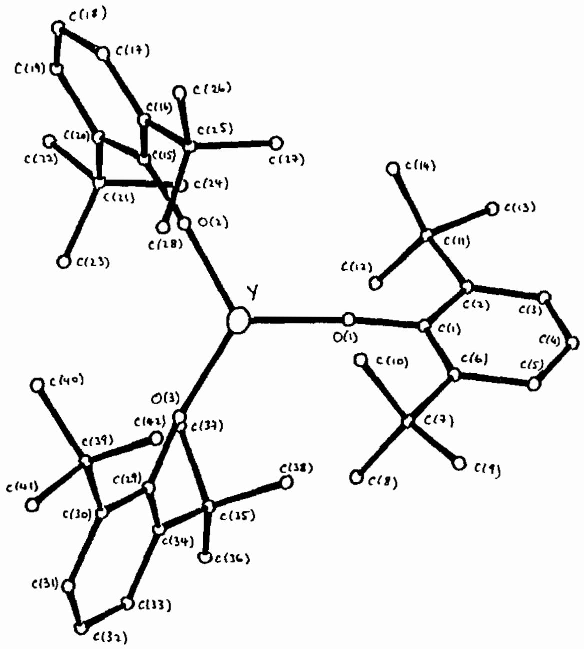

Fig. 1. The molecular structure and atom numbering scheme for $[Y(OC_6H_3Bu^t{}_2\text{-}2,6)_3]$ (1).

is 119.8(6)°. The Y–O–C angles are large, average 173(1)°, reflecting the steric bulk of the chosen aryloxide ligand. The Y–O bond length, average 2.00(1) Å, is the shortest in the literature [6]. It is, however, comparable to the Sc–O distance in $[Sc(OAr^{Me})_3]$ [3] (taking into account the differences in ionic radii for the appropriate central Ln^{3+} ion), and is identical to the Y–O distance found in another yttrium complex recently prepared at Sussex, $[Y\{OC(Bu^t)_2CH_2PMe_2\}_3]$ [7].

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