APPLIED ORGANOMETALLIC CHEMISTRY
Appl. Organometal. Chem. 2004; **18**: 252
Main Group Metal Compounds
Published online in Wiley InterScience (www.interscience.wiley.com). DOI:10.1002/aoc.613

Crystallographic report

Bis(μ_2-chloro)-{bis(diethylether)-lithium}-bis(η^5-pentamethyl-cyclopentadienyl)samarium(III)

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Received 12 December 2003; Revised 14 January 2004; Accepted 14 January 2004

The structure is mononuclear with samarium bound by two η^5-cyclopentadienyl ligands and two chloride ligands, the latter of which bridge to a doubly ether-solvated lithium centre. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; samarium; cyclopentadienyl

COMMENT

$[(Cp^*)_2Sm(\mu\text{-}Cl)_2Li(Et_2O)_2]$ is a mononuclear heterobimetallic compound. It is isomorphous with its Ce(III)[1] and Yb(III)[2] analogues and crystallizes with one-half of the molecule in the asymmetric unit, with the other half being generated by a twofold rotation axis. The samarium centre is eight-coordinate, and the lithium is distorted tetrahedral (see Fig. 1 and caption for geometric parameters). The $Sm-Cp^*_{(centroid)}$ (2.45 Å and Sm–Cl (2.6892(12) Å) distances are intermediate between those of the cerium and ytterbium analogues, in line with the variation in Ln^{3+} size.[3] The lithium environment reflects those in the predecessors of the series.

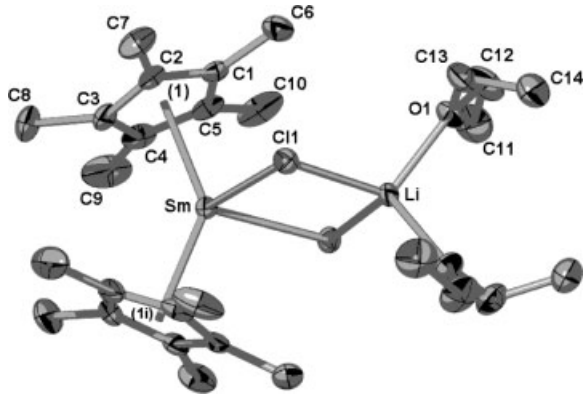

Figure 1. Molecular structure of $[(Cp^*)_2Sm(\mu\text{-}Cl)_2Li(Et_2O)_2]$; hydrogen atoms are omitted for clarity. Important geometric parameters: Sm–Cl1 2.6892(12), Sm–centroid(1) 2.45, Li–Cl1 2.406(6), Li–O1 1.957(6) Å; centroid(1)–Sm–centroid(1^i) 138, centroid(1)–Sm–Cl1 104, centroid(1)–Sm–$Cl1^i$ 107, Cl1–Li1–O1 110.24(11), Cl1–Li1–$Cl1^i$ 97.5(3), Cl1–Li1–$O1^i$ 117.14(11), O1–Li1–$O1^i$ 105.0(5)°. (SUs not given for centroids as these points are not refined.) Symmetry operation i: $1-x, y, 1/2-z$.

EXPERIMENTAL

A slightly modified procedure for the synthesis of the ytterbium analogue was followed.[2] Thus, to a solution of NaCp (38.9 mmol) in diethyl ether was added anhydrous $SmCl_3$ (5.0 g, 19.5 mmol), and the solution stirred overnight. The colourless solution was filtered and concentrated, and colourless crystals deposited after cooling at 0 °C. Yield: 82%. Crystal data for: $C_{28}H_{50}Cl_2LiO_2Sm$, $M = 646.87$, $0.25 \times 0.30 \times 0.30$ mm^3, monoclinic, space group $C2/c$, $a = 16.442(5)$, $b = 13.871(5)$, $c = 14.054(5)$Å, $\beta = 91.389(6)°$, $V = 3204.2(18)$Å^3, $Z = 4$, $D_c = 1.341$ g cm^{-3}. Bruker SMART 1000 CCD diffractometer, $T = 296(2)$K, $2\theta_{max} = 56.0°$, μ(Mo Kα) = 2.019 mm^{-1}, 10 398 reflections collected, 3813 reflections with $I > 2\sigma(I)$. $R = 0.046$ (obs. data), $wR_2 = 0.094$ (all data). Programs used: SAINT, SHELXL97, POVRAY, XSEED. CCDC number: CCDC 218 387.

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