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Isolation and X-ray crystal structures of the organolithium etherate complexes, $[Li(Et_2O)_2(CPh_3)]$ and $[\{Li(Et_2O)(2,4,6\text{-}(CHMe_2)_3C_6H_2)\}_2]$ *

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Abstract

The organolithium etherate complexes $[Li(Et_2O)_2(CPh_3)]$ (**1**) and $[\{Li(Et_2O)\text{-}(2,4,6\text{-}(CHMe_2)_3C_6H_2)\}_2]$ (**2**) have been crystallized from their ether solutions and structurally characterized by X-ray crystallography. The complex **1** has the rare mononuclear structure, with lithium coordinated to two ethers and at least three carbon atoms of the CPh_3 group; it thus offers an interesting comparison to the structure of $[Li(12\text{-crown-}4)_2][CPh_3]$, which contains a free $[CPh_3]^-$ ion. The structure of complex **2** is dimeric, and features two lithium atoms bridging between two $2,4,6\text{-}(CHMe_2)_3C_6H_2$ (Trip) groups. The lithium atoms are three-coordinate, being bonded to diethyl ether in addition to two carbon atoms. A significant feature of this structure is the asymmetric nature of the lithium bridging, with significant distortion in the angles and distances surrounding lithium being apparent. This is suggestive of the weak association due to the large size of the Trip group. These structures represent rare additions to the small number of known structures involving organolithium compounds solvated by commonly used monodentate ether solvents.

Introduction

The renewed interest in the reactivity, structure, and bonding of organolithium compounds is reflected in a number of recent reviews [1–3], which have detailed the large amount of structural and theoretical data now available. Much of the structural data [1] deal with either solvent-free compounds, adducts with chelating bases like tmeda ($Me_2NCH_2CH_2NMe_2$), such as $[\{PhLi(tmeda)\}_2]$ [4] or intramolecularly

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chelated complexes such as $[Li_4(C_6H_4\text{-}2\text{-}CH_2NMe_2)_4]$, $[\{Li(Et_2O)(Naphthyl\text{-}8\text{-}NMe_2)\}_2]$ [6] or $[\{\overline{LiCH(CH_3)(C}H_2)_2(OMe)\}_4]$ [7] and these have provided much valuable information. However, organolithium reagents are, with few important exceptions, often used in ether solvents. To date, the known structures of organolithium complexes in which a monodentate ether is the only solvate are limited to 1,1,3,3-bis(2,2′-biphenylenepropene) lithium diethyl etherate [8], $[\{PhLi(Et_2O)\}_4]$ [9], $[\{PhLi(Et_2O)\}_3LiBr]$ [9], $[\{Li(thf)_2(2,4,6\text{-}Me_3C_6H_2)\}_2]$ [10], $[\{Li(Et_2O)(CH_2Ph)\}_\infty]$ [10] and $[\{\overline{LiCH(C}H_2)_2\}_2\ LiBr_2 \cdot 4Et_2O]$ [11]. In this laboratory, we have attempted to crystallize and determine the structures of organolithium etherate complexes, in order to provide accurate structural data on species which are closely related to the structure in the solution phase [12,13]. Here, we report the structures of two further examples which differ significantly from those previously reported.

Experimental

All operations were performed under an N_2 atomosphere. All solvents were distilled from conventional drying agents and degassed twice prior to use. $HCPh_3$ and $2,4,6\text{-}(CHMe_2)_3C_6H_2Br$ (TripBr) were purchased from Lancaster Synthesis and used as received. **2** was synthesized in a manner similar to that reported by Whitesides [14] and more recently by Bickelhaupt [15].

Synthesis of the etherate complexes, 1 and 2

$[Li(Et_2O)_2(CPh_3)]$ (1). **1** was synthesized by treating $HCPh_3$ (2.44 g, 10 mmol) in Et_2O (35 ml), cooled in an ice bath, with BuLi in hexane (1.6 *M*; 6.25 ml). The solution was allowed to warm to ambient temperature and stirred for a further 4 h. Removal of Et_2O under reduced pressure to incipient crystallization and slow (20 h) cooling in a freezer (−20 °C) gave the product, orange-red crystals. Yield 80%, m.p. 95–99 °C.

$[\{Li(Et_2O)(2,4,6\text{-}(CHMe_2)_3C_6H_2)\}_2]$ (2). In manner similar to $2,4,6\text{-}Me_3C_6H_2Li$ [10,16] and $2,4,6\text{-}(CMe_3)_3C_6H_2Li$, $2,4,6\text{-}(CHMe_2)_3C_6H_2Br$ (TripBr) was converted to **2** via reaction with BuLi in Et_2O. TripBr (1.4 g, 5 mmol) in Et_2O (20 ml) was cooled to ca. −30 °C, treated with the equivalent of BuLi in hexane (1.6 *M*) and stirred for 2 h. The reaction mixture was concentrated under reduced pressure (to 10 ml) and cooled in a −20 °C freezer for 3 days, to give the product **2** as colorless crystals. Yield 73%. The crystals soften at 162 °C and melt to a reddish oil at 176–177 °C.

X-Ray crystallographic studies

All data were collected with a Syntex $P2_1$ diffractometer equipped with a locally modified low temperature device using Mo-K_α radiation. Calculations were carried out on a Data General Eclipse computer using SHELXTL programs version 4 and 5. The atomic form factors including anomalous dispersion were from ref. 17. Orange-red needles of **1** and colorless parallelepipeds of **2** were obtained as described above. To prevent possible reaction with the air or loss of solvent, the crystals were left in the mother liquor until ready for use. A sample was removed under N_2 flow and covered with a hydrocarbon oil from which a single crystal was selected. The crystal was mounted on a glass fiber in a mounting pin with silicon grease and immediately placed on the diffractometer in a low temperature N_2

Table 1

Crystal data and summary of data collection and refinement [a]

	$[Li(Et_2O)_2(CPh_3)]$ (**1**)	$[\{Li(Et_2O)(2,4,6\text{-}(CHMe_2)_3C_6H_2)\}_2]$ (**2**)
formula	$C_{27}H_{35}LiO_2$	$C_{38}H_{66}O_2Li_2$
fw	398.5	568.9
crystal system	monoclinic	triclinic
space group	$P2_1/a$	$P\bar{1}$
conditions	$h0l, h = 2n, 0k0, k = 2n$	none
crystal dims. (mm)	0.05 × 0.15 × 0.50	0.25 × 0.53 × 0.80
crystal color and habit	orange needles	colorless, rectangular plates
a (Å)	15.381(4)	9.479(1)
b (Å)	10.087(2)	9.532(2)
c (Å)	16.525(3)	10.641(1)
α (°)		90.56(1)
β (°)	110.73(2)	94.17(1)
γ (°)		102.68(1)
V (Å^3)	2398(1)	935.1(2)
Z	4	1
d_{calc} (g cm^{-3})	1.10	1.01
linear abs. coeff. (cm^{-1})	0.62	0.60
range of abs. corr. fctrs.	1.00–1.03	1.01–1.05
scan speed (° min^{-1})	15	20
scan width (°)	1.2	1.0
w offset for bkgd. (°)	1.2	1.0
2θ range (°)	0–55	0–55
octants collected	$+h+k\pm l$	$+h\pm k\pm l$
no. of refl. collected	6005	4365
no. of unique data	5492	4317
R(merge)	0.03	0.005
no. of check refl.	2;(0,1,3)(2,0,0) no decay	2;(4, − 1,2)(0,3,1) no decay
no. of data used in rfmt.	1484 $I > 3\sigma(I)$	2942 $I > 3\sigma(I)$
no. of variables	133	214
data/variable ratio	11.2	13.7
$R(F)$	0.074	0.050
$R_w(F)$	0.071	0.057
weighting scheme	$[\sigma^2(F_o)+0.0005F_o^2]^{-1}$	$[\sigma^2(F_o)]^{-1}$
largest Δ/σ	0.005	0.013
largest feature on final diff. map (eÅ^{-3})	0.48 near C(20)	0.28 near C(4) and C(10)

[a] All data were collected on a $P2_1$ diffractometer with a graphite monochromator using Mo-K_α (λ 0.71069 Å) radiation at 130 K by a ω scan method. Computer programs are from SHELXTL, version 5, installed on a Data General Eclipse computer. $R = \Sigma \| F_o | - | F_c \| / | F_o |$ and $R_\omega = \Sigma \| F_o | - | F_c \| w^{1/2} / \Sigma | F_o w^{1/2} |$ with w defined in the table. Neutral atom scattering factors and corrections for anomalous dispersions are from the International Tables for X-Ray Crystallography, Kynoch Press, Birmingham, England, 1974, Vol. IV.

stream (130 K). A summary of experimental details is provided in Table 1 with additional details given below. Atom coordinates are listed in Table 2 and selected bond distances and angles in Tables 3 and 4. Complete bond distances, angles,

Table 2. Atomic coordinates ($\times 10^4$) and isotropic thermal parameters ($Å^2 \times 10^3$) for $[Li(Et_2O)_2(CPh_3)]$ (**1**) and $[\{Li(OEt_2)(2,4,6\text{-}(CHMe_2)_3C_6H_2)\}_2]$ (**2**)

Atom	*x*	*y*	*z*	*U*
Compound 1				
Li(1)	3849(8)	1873(13)	2322(7)	28(3)
C(1)	5340(4)	1095(6)	2622(4)	18(2)
C(2)	5173(4)	1838(7)	1830(4)	20(2)
C(3)	4904(4)	3184(7)	1733(4)	21(2)
C(4)	4700(4)	3879(7)	970(4)	24(2)
C(5)	4739(4)	3272(8)	230(4)	30(2)
C(6)	5031(4)	1943(7)	300(4)	26(2)
C(7)	5236(4)	1263(7)	1057(4)	23(2)
C(8)	5749(4)	1771(7)	3470(4)	22(2)
C(9)	6330(4)	2893(7)	3601(4)	25(2)
C(10)	6707(5)	3501(7)	4398(4)	31(2)
C(11)	6522(5)	3007(7)	5105(5)	32(2)
C(12)	5971(5)	1919(8)	5011(5)	35(2)
C(13)	5578(4)	1310(7)	4204(4)	25(2)
C(14)	5360(5)	−368(6)	2622(4)	21(2)
C(15)	4723(4)	−1129(7)	1961(4)	24(2)
C(16)	4748(5)	−2505(8)	1967(5)	29(2)
C(17)	5395(5)	−3196(8)	2643(5)	34(2)
C(18)	6022(5)	−2484(7)	3306(4)	30(2)
C(19)	6018(4)	−1102(7)	3285(4)	22(2)
O(1)	3495(3)	3355(5)	2901(3)	29(1)
O(2)	2780(3)	910(5)	1615(3)	28(1)
C(20)	2614(5)	4000(7)	2486(4)	33(2)
C(21)	2677(6)	5234(8)	1995(5)	53(3)
C(22)	4048(5)	3981(8)	3709(4)	34(2)
C(23)	3778(5)	3506(8)	4461(5)	45(2)
C(24)	2407(5)	−153(8)	1973(5)	45(2)
C(25)	2974(5)	−270(4)	2919(4)	41(2)
C(26)	2274(6)	949(9)	694(5)	59(3)
C(27)	2538(6)	2084(8)	312(5)	61(3)
Compound 2				
Li	10778(3)	4441(3)	5688(3)	28(1) [a]
O(1)	11723(1)	3531(1)	7014(1)	32(1)
C(1)	8418(2)	4312(2)	5945(1)	22(1)
C(2)	7744(2)	2823(2)	5866(1)	24(1)
C(3)	6975(2)	2115(2)	6833(2)	24(1)
C(4)	6828(2)	2838(2)	7950(1)	21(1)
C(5)	7428(2)	4307(2)	8035(1)	23(1)
C(6)	8184(2)	5030(2)	7064(1)	22(1)
C(7)	7803(2)	1970(2)	4655(2)	31(1)
C(8)	8207(4)	548(3)	4871(2)	85(1)
C(9)	6406(3)	1769(3)	3837(2)	60(1)
C(10)	6039(2)	1993(2)	9000(2)	25(1)
C(11)	5722(2)	2927(2)	10073(2)	34(1)
C(12)	6894(2)	918(2)	9534(2)	32(1)
C(13)	8795(2)	6640(2)	7239(2)	28(1)
C(14)	7683(2)	7468(2)	7652(2)	41(1)
C(15)	10146(2)	6960(2)	8155(2)	36(1)
C(16)	11038(2)	3111(2)	8159(2)	32(1)
C(17)	10888(2)	1539(2)	8414(2)	42(1)
C(18)	13265(2)	3639(3)	7142(2)	45(1)
C(19)	13859(2)	3721(3)	5911(2)	54(1)

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[a] Equivalent isotropic *U* defined as one third of the trace of the orthogonalised U_{ij} tensor.

Table 3

Selected bond distances (Å) and angles (°) for **2**

Li–O(1)	1.930(3)
Li–C(1)	2.249(3)
Li–C(1′)	2.203(3)
O(1)–Li–C(1)	113.0(1)
O(1)–Li–C(1′)	132.8(2)
C(1)–Li–C(1′)	114.1(1)
Li–C(1)–Li′	65.9(1)

anisotropic thermal parameters and hydrogen atom coordinates and structure factor tables are provided in the supplementary material.

[Li(Et$_2$O)$_2$(CPh$_3$)] (1). The orientation matrix and cell dimensions were obtained by a least-squares fit of 24 well-centred reflections with $25° < 2\theta < 35°$. The structure was solved by direct methods. Hydrogen atoms were included using a riding model with r(C–H) of 0.96 Å and U_{iso}(H)-1.2 U_{iso}(C). The structure was not refined anisotropically due to the low number of observed reflections (1484, $I > 3\sigma(I)$) compared to the number of refining parameters (133).

[{Li(Et$_2$O)(2,4,6-(CHMe$_2$)$_3$C$_6$H$_2$)}$_2$] (2). Cell determination was made using 10 well-centered reflections with $19° < 2\theta < 24°$. The structure was solved by direct

Table 4

Comparison of important bond distances (Å) and angles (°) in [Li(Et$_2$O)$_2$(CPh$_3$)] (**1**), [Li(12-crown-4)$_2$][CPh$_3$] (**3**) and [Li(tmeda)(CPh$_3$)] (**4**)

	1	**3**	**4**
C(1)–C(2)	1.450(10)	1.451(5)	1.462(13)
C(1)–C(8)	1.484(9)	1.459(5)	1.488(10)
C(1)–C(14)	1.476(9)	1.450(4)	1.448(9)
C(2)C(1)C(8)	119.7(6)	119.4(3)	117.0(6)
C(2)C(1)C(14)	121.0(6)	122.3(3)	122.8(7)
C(8)C(1)C(14)	117.2(5)	118.4(3)	118.3(6)
Σ C(1) angle	357.9	360.1	358.1
phenyl ring twist with respect to C(1) C(2) C(8) C(14) plane			
C(2) twist	24.8	21.3	30.6
C(8) twist	35.5	30.3	44.8
C(14) twist	31.9	42.0	19.7
distance out of plane C(1)	0.12	0.0	0.12
Li–C interactions			
Li–C(1)	2.306(14)	–	2.227(8)
Li–C(2)	2.446(16)	–	2.511(3)
Li–C(14)	–	–	2.488(1)
Li–C(15)	–	–	2.541(9)
Li–O(1)	1.954(14)		
Li–O(2)	1.911(11)		

methods. An absorption correction was made using the program XABS [18]. Hydrogens were added by the riding model with r(C–H) of 0.96 Å and U_{iso}(H) = 1.2 U_{iso}(C) where U_{iso} is the equivalent isotropic thermal parameter. All non-hydrogen atoms are refined anisotropically. A large thermal ellipsoid was observed for C(8), possibly due to motion or disorder in the U_{11} direction. No large features were observed on the final difference map.

Description of structures

[Li(Et$_2$O)$_2$(CPh$_3$)] (1). The asymmetric unit contains a mononuclear structure of lithium coordinated to two ethers and three carbon atoms in the CPh$_3$ group, as illustrated in Fig. 1. The Li–C interactions range from 2.306(14) for C(1) to 2.540(16) Å for C(3). The next closest Li–C interaction is 2.868 Å for C(8), and this is probably too long to be considered a bonding interaction. The C(1) atom sits slightly above the plane (0.12 Å) of C(2), C(8) and C(14). Twist angles of the phenyl rings with respect to the C(1), C(2), C(8), C(14) plane, which give the CPh$_3$ group its propeller geometry, are given in Table 4. The C(2) ring with two carbon–lithium interactions has the smallest twist angle (24.8°). Phenyl carbon–carbon distances are normal, and average 1.395 Å.

[{Li(Et$_2$O)(2,4,6-(CHMe$_2$)$_3$C$_6$H$_2$)}$_2$] (2). The structure consists of an asymmetric dimer, as illustrated in Fig. 2. The lithium atoms are coordinated to one ether and two Trip groups, with asymmetric Li–C distances of 2.249(3) and 2.203(3) Å. The Trip groups are parallel to each other, and approximately perpendicular to the Li–C(1)–Li′–C(1′) plane (84.7°). A line connecting C(1) and C(4) forms a 11.4°

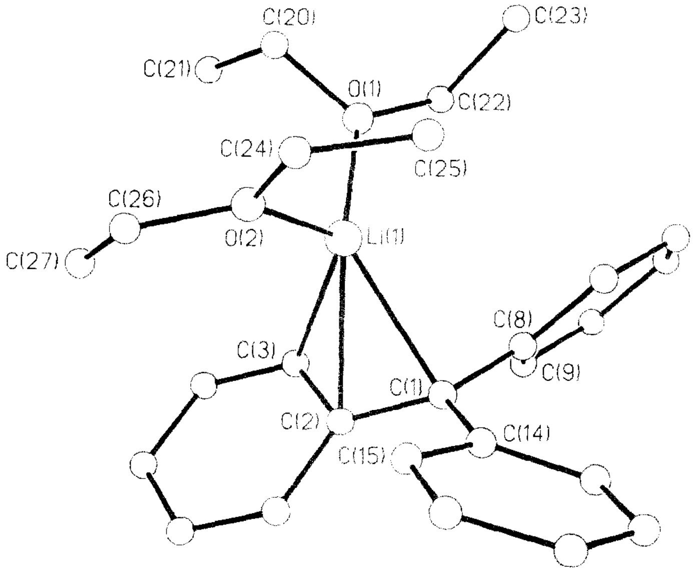

Fig. 1. Computer generated plot of **1**. Arbitrary radius circles used to represent isotropic atoms. Hydrogens omitted for clarity.

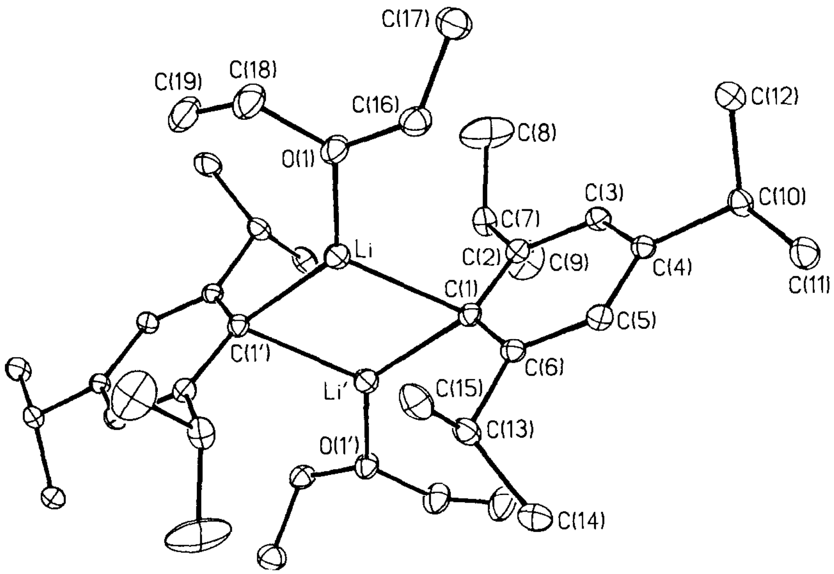

Fig. 2. Computer generated plot of **2**. Thermal ellipsoids at 30% probability level for anisotropic atoms.

angle with the C(1)–Li′ vector. The carbon–carbon distances within the Trip and ether moieties are all normal.

Discussion

The relationship between the solid state and solution phase structures of organolithium reagents is of considerable interest. Since these reagents are normally used in ether solvents, their solvate structures are of obvious relevance to the solution species. There are few structures of compounds of this type reported. The reasons of the scarcity of data are mainly because of the perceived, and sometimes real, difficulties in crystal handling due to ready desolvation. There is no doubt that this is a difficulty in many of these compounds. However, this problem can usually be overcome, as described in the X-ray section and previous reports [9,10]. In the case of both compounds **1** and **2**, there is little problem with desolvation when handled as described, and the melting points of both compounds are quite high.

The structure of **1** is a rare example of a mononuclear organolithium complex. Only a few such complexes are known, and these are confined to compounds with either very bulky organic groups or organic anions stabilized by delocalization. The closest related complexes to **1** are [Li(12-crown-4)$_2$][CPh$_3$] (**3**) [19] and [Li(tmeda)(CPh$_3$)] (**4**) [20]. The molecular structure of **1** is a contact ion-pair between [Li(Et$_2$O)$_2$]$^+$ and [CPh$_3$]$^-$, similar to that found in **4**. A comparison of important distances and angles in the [CPh$_3$]$^-$ moiety in **1**, **3** and **4** is given in Table 4. The major conclusion from these data is that the structures of the [CPh$_3$]$^-$ moieties in **1** and **4** are almost identical, with similar degrees of pyramidicity and distortion as a result of the interaction with the [Li(Et$_2$O)$_2$]$^+$ or [Li(tmeda)]$^+$ moieties. A fuller discussion of these factors is given in ref. 20. However, the interaction of the lithium ion with the [CPh$_3$]$^-$ anion in **1** differs from **4**. In **4**, the closest contacts involve C(1), C(2), C(14) and C(15), as listed in Table 4. In **1**, however, the lithium atom interacts with only three atoms, C(1), C(2) and C(3), at similar distances. This is similar to the case of [{Li(1,8-diamino-3,6-diazaoctane)(CH_2Ph)}$_\infty$] [21] and [{Li(Et_2O)(CH_2Ph)}$_\infty$] [10], where Li^+ interacts with just two carbon atoms in the latter, rather than with three carbons in the former. It is not known at present why this is so, since weaker interactions with the carbanion centre would be expected when more strongly coordinating amine bases are used. The Li–O bond distances are quite normal, and very similar to those for Li · OEt_2 complexes with similar coordination numbers at lithium [10].

The structure of the [{Li(Et_2O)(2,4,6-($CHMe_2$)$_3C_6H_2$)}$_2$] complex (**2**) may be compared with those of the less sterically-crowded [{Li(Et_2O)Ph}$_4$] (**5**) [9], and [{Li(thf)$_2$(2,4,6-$Me_3C_6H_2$)}$_2$] (**6**) [10]. The phenyllithium etherate, **5**, is tetrameric with the familiar distorted cubane structure. Replacement of Ph by 2,4,6-$Me_3C_6H_2$ affords the dimer **6**, but the degree of solvation has now been doubled. In this case, thf rather than Et_2O was used for practical reasons involving the lack of solubility of 2,4,6-$Me_3C_6H_2Li$ in Et_2O. Nevertheless, the trend is clear raising the steric requirements reduces the degree of aggregation from four to two. A further increase in size in the case of **2** also yields a dimer, but now the solvation is reduced to one molecule of Et_2O per lithium atom, yielding three coordinate lithium centres. It may be anticipated that the use of the even bulkier 2,4,6-(CMe_3)$_3C_6H_2$ group should, for steric reasons, result in either a mononuclear complex or a desolvated dimer. Unfortunately, suitable crystals of such a complex have so far proved elusive.

The structure of **2** displays a number of other features which point to a considerable degree of distortion, which suggests that it might easily dissociate to monomers either in solution or with a slightly more strongly coordinating solvent. For example, the lithium bridging is not symmetric. This is illustrated by the significant difference in the Li–C(1) and Li–C(1′) bonds of 0.046 Å and the irregularity of the O(1)LiC(1) and C(1′) angles, which are 113.0(1) and 132.8(2)°. These distortions, which are presumably due to steric crowding, suggest that dissociation is quite likely if crowding is increased further. It may also be argued that the Li–C distances in **2** are relatively short, since they are only slightly longer than those seen for **6**. This is despite the fact that lithium is only three-coordinate in **2**, whereas in **6** it is four-coordinate. Attempts to crystallize the next highest member of the series, a 2,4,6-(CMe_3)$_3C_6H_2Li$ ether solvate, are continuing.

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