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X-RAY CRYSTAL STRUCTURE OF TRIMETHYLSILYLMETHYLLITHIUM

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Summary

The solid state structure of trimethylsilylmethyllithium has been determined by single crystal X-ray diffraction techniques. The compound crystallizes in the monoclinic system, space group $P2_1/n$. Cell dimensions were determined as follows: a 10.931(3), b 18.397(6), c 21.490(8) Å, β 96.0(2)°, V 4298(2) Å^3, $Z = 4$, and a final R_f 5.1% based on 2203 data with $\sigma(I) \geq 2.5\sigma(I)$. The compound is formed by hexameric units, $\{LiCH_2Si(CH_3)_3\}_6$, with two distinct classes of Li–Li distances of 2.46 and 3.18 Å. There also are two Li–C distances av. 2.20 and 2.27 Å. The Li–H distances to the methylene H atoms have been determined and are short varying between 2.0 and 2.3 Å to the closest lithium atom. The structure, including possible Li–H interactions, is discussed and compared with the other known hexameric aggregates.

Introduction

The investigation of the solid state and solution structure of organolithium aggregates has received increasing attention in the past several years [1]. The known structures have been summarized recently [1f]. From these data it can be seen that the majority of structures determined are of solvent adducts or more complex derivatives. The structures of the simple solvated complexes take on a variety of forms ranging form tetrameric adducts such as $\{(LiMe)_4 \cdot 2TMEDA\}$ [2] and $\{LiPh \cdot OEt_2\}_4$ [3], dimers such as {lithiobicyclo[1.1.0]butane · 2TMEDA} [4] and $\{LiPh \cdot TMEDA\}_2$ [5] while monomeric LiR units have been found in a variety of derivatives [6–9]. With the very bulky moiety, $C(SiMe_3)_3$, even a lithium "ate" complex, $\{Li(THF)_3\}^+\{Li[C(SiMe_3)_3]_2\}^-$, has been observed [10]. For the uncomplexed species only two stable aggregate forms, tetramer [11,12] and hexamer have been established for alkyl- [13,14] and silyllithium [15,16] derivatives. This also holds in the mixed bromocyclopropyllithium complex where a tetrameric species is observed [17].

In solution studies, the information is more difficult to interpret and, it would appear that the systems are more complex with a number of equilibria involved.

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Early colligative property measurements [1a], established that the average degree of aggregation for a number of simple alkyllithium compounds LiR (R = Et, n-Pr, n-Bu) is 6 in hydrocarbon solvents such as benzene and cyclohexane. When bulkier groups such as i-Pr, s-Bu, or CH_2SiMe_3 are present, the degree of aggregation is dependent both on the bulkiness of the group and on the solvent system with benzene favoring tetramer and saturated solvents favoring hexamers. Finally, with R = t-Bu, the only aggregate present was tetramer and with the extremely bulky menthyl group dimeric menthyllithium was observed in hydrocarbon solvents [18]. In coordinating solvents the strong interaction between the solvent and the lithium atom leads to formation of smaller aggregates with tetramers, dimers, and even solvated monomers observed [1].

Recent reports based on ^6Li and ^{13}C NMR studies have suggested that in Li-n-Pr, as well as in other systems, higher aggregates exist [19–21]. With this information in mind and the limited number of structures reported for uncomplexed organolithium derivatives, we now wish to report the structure of $LiCH_2SiMe_3$. This is the third organolithium derivative determined to be hexameric in the solid state by X-ray diffraction and the fifth lithium derivative which shows this conformation.

Experimental

An initial sample of $(LiCH_2SiMe_3)_6$ was kindly provided by Dr. O.T. Beachley. Subsequent material was prepared by the reaction of Li metal with $ClCH_2SiMe_3$ in pentane following the procedure of Beachley and Tessier–Youngs [22].

X-ray data collection

Initially, crystals of $(LiCH_2SiMe_3)_6$ were prepared by sublimation in an evacuated tube from a 55 °C zone to room temperature, but these crystals which were thin plates and showed some cracking. Examination of peak scans on these crystals showed doubling and they, therefore, were rejected in favor of crystals grown from pentane by slow evaporation in an argon filled drybox. Suitable crystals were cut to size and placed in Lindemann Glass capillaries, plugged with silicon grease, removed from the drybox and immediately flame sealed.

A crystal determined to be suitable from examination by microscopy was mounted on the Syntex $P2_1$ diffractometer. Fifteen reflections were centered, orientation matrix and cell constants determined. Data were collected using θ–2θ scan. Other details of the data collection, cell parameters and the remaining relevant information are collected in Table 1. At the end of data collection 64 reflections with 2θ values in the range of 11.45 to 26.5° were centered and a least-squares fit to the setting angles gave the final cell constants, which were used for the solution and refinement of the structure.

Solution and refinement of the structure

Absence of the $0k0$, $k = 2n + 1$ and $h0l$, $h + l = 2n + 1$ reflections made it possible to unambiguously assign the crystal to the space group $P2_1/n$. Further, based on the assumption that $Z = 4$ and the known cell volume, the only reasonable density for the compound D_{calc} 0.893 g cm^{-3}, was found for a hexameric lithium species.

TABLE 1

PHYSICAL CONSTANTS AND EXPERIMENTAL CRYSTAL DATA FOR THE STRUCTURE DETERMINATION OF $Li(CH_2SiMe_3)_6$

Molecular formula	$Li_6Si_6C_{24}H_{66}$
Molecular weight	564.95 (hexamer)
Crystal system	monoclinic
Space group	$P2_1/n$
Z	4
Cell dimensions	a 10.931(3) Å b 18.397(6) Å c 21.490(8) Å β 96.00(2)° V 4298(2) Å^3
$F(000)$	1248
ρ_{calcd} (g cm^{-3})	0.873
Radiation	Mo-$K_{\bar{\alpha}}$ (λ 0.71069 Å)
Monochromator	graphite
2θ scan range	2–45°
Scan width	$[2\theta(\text{Mo-}K_{\alpha_1})-1.0]° \rightarrow [2\theta(\text{Mo-}K_{\alpha_2})+1.0]°$
Scan rate	2° min^{-1} in 2θ
Reflections measured	$\pm h, +k, +l$
Standards	3 observed after every 97 reflections $1,3,\bar{3}$; $\bar{1},\bar{3},3$; $1,\bar{3},\bar{3}$
Max. dev. of standards during data collection	6%, random, no decay correction applied
No. of unique data collected	6247
No. of data used, $\sigma(I) > 2.5\sigma(I)$	2203
linear absorption coefficient, m	1.99 cm^{-1}
$R_F = \Sigma \lVert F_o \rvert - \lvert F_c \rVert / \Sigma \lvert F_o \rvert =$	0.051
$R_{wF} = \{\Sigma w(\lvert F_o \rvert - \lvert F_c \rvert)^2/\Sigma w(\lvert F_o \rvert^2\}^{1/2} =$	0.058
max residual electron density	0.17 e/Å^3; at 0.584; 0.043; 0.654
GOF	1.52
NV	373

With this information in hand the structure was solved by the light atom technique through the use of MULTAN [23,24] which generated the positions of six silicon atoms and all twenty-four carbon atoms. One complete cycle of least-squares refinement varying x, y, and z, the scale factor, and the isotropic temperature factor for all atoms produced an R value of 19.2%, confirming the gross structure. The remaining six independent lithium atoms and twelve methylene hydrogen atoms were located from a subsequent Fourier difference map. The fifty-four methyl hydrogen atoms were placed 0.95 Å from the carbon atoms in idealized positions calculated with the program HFINDR, and were adjusted after every second least-squares cycle and their temperature factors assigned values 1.1 times those of the carbon atoms to which they are bonded. Temperature factors for Si, C, and Li atoms were refined anisotropically and that for methylene H atoms isotropically along with their positional parameters and scale factors. All parameters associated with methyl hydrogen atoms were held fixed throughout the refinement. Full matrix least-squares refinement yielded residual indices of R_F 5.1% and R_{wF} 5.8%.

Neutral atom scattering factors [25] were used for all the atoms. Final positional parameters of Si, C, Li and methylene H atoms are presented in Table 2. Selected

TABLE 2

ATOMIC COORDINATES FOR ALL OF THE NON-HYDROGEN ATOMS AND THE METHYLENE HYDROGEN ATOMS IN $(LiCH_2SiMe_3)_6$

Atom	*x*	*y*	*z*
Li(1)	0.5217(11)	0.2985(7)	0.8251(5)
Li(2)	0.4597(11)	0.4236(7)	0.8413(6)
Li(3)	0.3147(10)	0.3982(7)	0.7508(5)
Li(4)	0.4783(11)	0.4614(7)	0.7013(6)
Li(5)	0.5384(12)	0.3344(7)	0.6833(6)
Li(6)	0.6868(10)	0.3606(7)	0.7745(5)
Si(1)	0.8089(2)	0.3622(1)	0.8998(1)
Si(2)	0.3258(2)	0.2664(1)	0.8984(1)
Si(3)	0.3206(2)	0.5606(1)	0.8395(1)
Si(4)	0.1890(2)	0.4017(1)	0.6280(1)
Si(5)	0.6732(2)	0.4898(1)	0.6236(1)
Si(6)	0.6774(2)	0.2012(1)	0.6818(1)
C(11)	0.6494(8)	0.3854(6)	0.8706(4)
C(12)	0.9057(7)	0.3607(5)	0.8335(4)
C(13)	0.8845(8)	0.4268(6)	0.9576(4)
C(14)	0.8149(8)	0.2710(5)	0.9370(4)
C(21)	0.3332(9)	0.3275(6)	0.8322(5)
C(22)	0.4546(7)	0.1996(5)	0.9010(4)
C(23)	0.1836(8)	0.2137(6)	0.8999(5)
C(24)	0.3488(12)	0.3183(6)	0.9732(5)
C(31)	0.3918(9)	0.5107(5)	0.7793(5)
C(32)	0.1655(8)	0.5270(6)	0.8492(5)
C(33)	0.4117(10)	0.5471(7)	0.9154(5)
C(34)	0.3056(12)	0.6596(6)	0.8255(7)
C(41)	0.3483(7)	0.3761(6)	0.6558(4)
C(42)	0.1124(8)	0.3452(6)	0.5647(4)
C(43)	0.1822(7)	0.4975(5)	0.5998(4)
C(44)	0.0927(6)	0.3962(5)	0.6942(3)
C(51)	0.6681(8)	0.4318(5)	0.6921(4)
C(52)	0.5506(8)	0.5587(5)	0.6222(4)
C(53)	0.8187(8)	0.5410(6)	0.6190(5)
C(54)	0.6485(10)	0.4358(6)	0.5511(4)
C(61)	0.6089(9)	0.2489(5)	0.7456(4)
C(62)	0.5838(9)	0.2207(5)	0.6064(4)
C(63)	0.8364(8)	0.2322(6)	0.6744(5)
C(64)	0.6876(10)	0.1004(5)	0.6896(5)
H(111)	0.6009(55)	0.3757(34)	0.9016(26)
H(112)	0.6473(57)	0.4320(32)	0.8681(28)
H(211)	0.2852(70)	0.3556(43)	0.8418(37)
H(212)	0.2968(56)	0.3006(33)	0.7963(27)
H(311)	0.4628(51)	0.5391(32)	0.7796(25)
H(312)	0.3460(77)	0.5184(50)	0.7449(37)
H(411)	0.3845(46)	0.3828(29)	0.6196(24)
H(412)	0.3441(54)	0.3281(31)	0.6618(27)
H(511)	0.7262(51)	0.3961(31)	0.6879(25)
H(512)	0.6968(59)	0.4554(35)	0.7272(28)
H(611)	0.5310(55)	0.2243(35)	0.7440(27)
H(612)	0.6457(51)	0.2296(33)	0.7808(25)

TABLE 3

SELECTED INTERATOMIC DISTANCES (Å) AND ANGLES (°) IN $Li(CH_2SiMe_3)_6$

Li(1)–Li(2)	2.43(2)	Li(1)–Li(6)	2.48(2)	Li(1)–Li(3)	3.20(2)	Li(1)–Li(5)	3.14(2)
Li(2)–Li(3)	2.42(2)	Li(3)–Li(4)	2.47(2)	Li(2)–Li(4)	3.12(2)	Li(2)–Li(6)	3.21(2)
Li(4)–Li(5)	2.47(2)	Li(5)–Li(6)	2.46(2)	Li(3)–Li(5)	3.19(2)	Li(4)–Li(6)	3.22(2)
Li(1)–C(11)	2.28(2)	Li(1)–C(21)	2.15(2)	Li(2)–C(31)	2.17(2)	Li(3)–C(41)	2.15(1)
Li(2)–C(11)	2.22(2)	Li(2)–C(21)	2.24(2)	Li(3)–C(31)	2.29(2)	Li(4)–C(41)	2.27(2)
Li(6)–C(11)	2.20(1)	Li(3)–C(21)	2.17(2)	Li(4)–C(31)	2.20(1)	Li(5)–C(41)	2.24(1)
Li(4)–C(51)	2.17(4)	Li(1)–C(61)	2.24(1)				
Li(5)–C(51)	2.28(2)	Li(5)–C(61)	2.15(2)				
Li(6)–C(51)	2.20(1)	Li(6)–C(61)	2.29(1)				
Li(1)–H(611)	2.22(6)	Li(2)–H(111)	2.10(6)	Li(3)–H(211)	2.16(8)	Li(4)–H(311)	2.23(6)
Li(1)–H(612)	2.15(6)	Li(2)–H(112)	2.08(6)	Li(3)–H(212)	2.06(6)	Li(4)–H(312)	2.09(9)
Li(1)–H(111)	2.27(6)	Li(2)–H(211)	2.28(8)	Li(3)–H(311)	3.08(6)	Li(4)–H(411)	2.42(5)
Li(1)–H(612)	2.92(6)	Li(2)–H(212)	2.98(6)	Li(3)–H(312)	2.24(9)	Li(4)–H(412)	2.94(6)
Li(1)–H(211)	2.85(7)	Li(2)–H(311)	2.51(6)	Li(3)–H(411)	3.01(5)	Li(4)–H(511)	3.00(6)
Li(1)–H(212)	2.47(6)	Li(2)–H(312)	2.89(8)	Li(3)–H(412)	2.36(6)	Li(4)–H(512)	2.40(6)
Li(5)–H(411)	2.24(5)	Li(6)–H(511)	2.06(5)				
Li(5)–H(412)	2.23(6)	Li(6)–H(512)	2.03(6)				
Li(5)–H(511)	2.34(6)	Li(6)–H(611)	3.06(6)				
Li(5)–H(512)	2.92(6)	Li(6)–H(612)	2.46(6)				
Li(5)–H(611)	2.41(6)	Li(6)–H(111)	2.99(6)				
Li(5)–H(612)	3.00(6)	Li(6)–H(112)	2.48(6)				

Li(2)–Li(1)–Li(6)	81.6(5)	Li(3)–Li(2)–Li(1)	82.6(6)	Li(4)–Li(3)–Li(2)	79.2(5)
Li(5)–Li(4)–Li(3)	80.7(6)	Li(6)–Li(5)–Li(4)	81.6(5)	Li(1)–Li(6)–Li(5)	79.0(5)
C(11)–Li(1)–Li(2)	56.0(5)	C(21)–Li(2)–Li(1)	54.6(5)	C(31)–Li(3)–Li(2)	54.6(5)
C(11)–Li(1)–Li(6)	54.8(4)	C(21)–Li(2)–Li(3)	55.4(5)	C(31)–Li(3)–Li(4)	55.0(5)
C(11)–Li(2)–Li(1)	58.4(5)	C(21)–Li(1)–Li(2)	58.1(5)	C(31)–Li(2)–Li(3)	59.6(5)
C(11)–Li(6)–Li(1)	57.8(5)	C(21)–Li(3)–Li(2)	58.1(5)	C(31)–Li(4)–Li(3)	58.5(5)
C(41)–Li(4)–Li(3)	53.9(5)	C(51)–Li(5)–Li(4)	54.3(5)	C(61)–Li(6)–Li(1)	55.8(4)
C(41)–Li(4)–Li(5)	56.1(5)	C(51)–Li(5)–Li(6)	55.1(5)	C(61)–Li(6)–Li(5)	53.9(5)
C(41)–Li(3)–Li(4)	58.4(5)	C(51)–Li(4)–Li(5)	58.4(5)	C(61)–Li(1)–Li(6)	57.7(4)
C(41)–Li(5)–Li(4)	57.4(5)	C(51)–Li(6)–Li(5)	58.3(5)	C(61)–Li(5)–Li(6)	59.0(5)
Li(1)–C(11)–Li(2)	65.6(5)	Li(2)–C(21)–Li(1)	67.3(5)	Li(3)–C(31)–Li(2)	65.7(5)
Li(1)–C(11)–Li(6)	67.4(5)	Li(2)–C(21)–Li(3)	66.6(5)	Li(3)–C(31)–Li(4)	66.5(6)
Li(4)–C(41)–Li(3)	67.8(5)	Li(5)–C(51)–Li(4)	67.3(5)	Li(6)–C(61)–Li(1)	66.5(5)
Li(4)–C(41)–Li(5)	66.5(5)	Li(5)–C(51)–Li(6)	66.6(5)	Li(6)–C(61)–Li(5)	67.2(5)

bond distances and bond angles are given in Table 3. Methyl hydrogen atom coordinates, thermal parameters and the calculated and observed structure factors as well as a complete listing of all bond distances and bond angles are available [22].

Results and discussion

The structures of organolithium derivatives and of the related silyl- and amino-lithium compounds are of major interest as cited in the introduction because of the similar properties exhibited both in the solid state and as shown in solution by colligative property measurements. Here we may compare the structures of the three known hexameric carbon bridged species, $(Li\text{-}c\text{-}C_6H_{11})_6$ [13]; lithiomethyl-1,1,2,2-tetramethylcyclopropane [14] and the $(LiCH_2SiMe_3)_6$ reported in this work, with the silicon derivative $(LiSiMe_3)_6$ [15,16] and with the nitrogen bridged complex

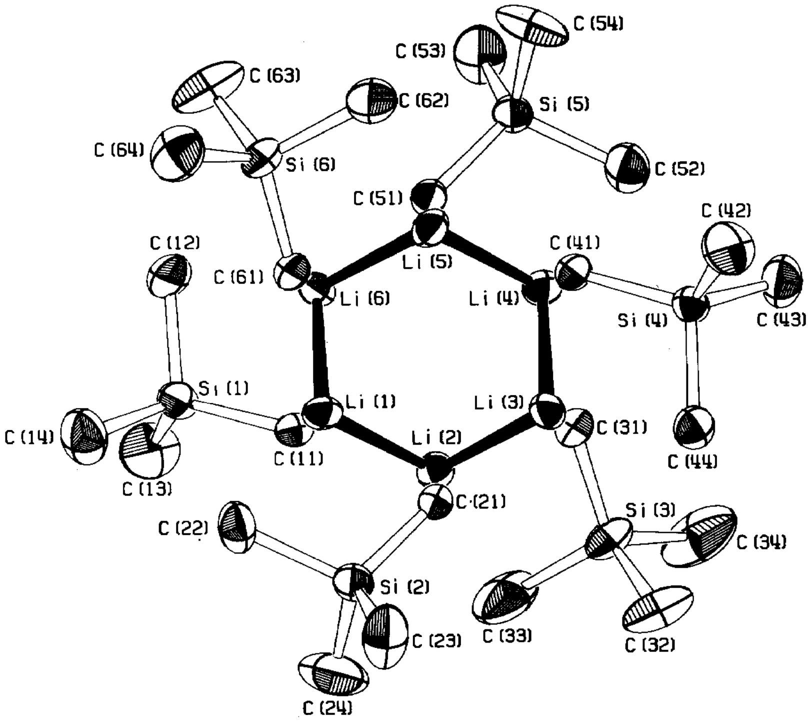

Fig. 1. An ORTEP drawing of the $(LiCH_2Si(CH_3)_3)_6$ molecule with the atoms labeled. All hydrogen atoms have been omitted for the sake of clarity.

di-t-butylmethyleneaminolithium, $(LiN{=}C(t\text{-}Bu)_2)_6$ [27]. The five structures are all quite similar. The general shapes are the same and may be described as a distorted octahedron, a trigonal antiprism, or as a "folded chair" arrangement as shown in Fig. 1 for the $LiCH_2SiMe_3$ hexamer. A summary of several of the pertinent

TABLE 4

STRUCTURAL PARAMETERS OF HEXAMERIC LITHIUM DERIVATIVES

Compound	Li–Li (Å)	Li–X (Å) (X = C, Si, N)	Back to seat angle (°)
$(Li\text{-}c\text{-}C_6H_{11})_6$ [a]	2.397 2.968	2.184 2.300	73.0
$[LiSi(CH_3)_3]_6$ [b]	2.73 3.26	2.65 2.77	70.5
$[LiN{=}C(t\text{-}Bu)_2]_6$ [c]	2.35 3.21	2.06	85.0
[Lithiomethyl-1,1,2,2-tetramethylcyclopropane]$_6$ [d]	2.462 2.976	2.123 2.159	70.3
$[LiCH_2Si(CH_3)_3]_6$	2.45 3.18	2.20 2.28	79.5

[a] Ref. 13. [b] Ref. 15, 16. [c] Ref. 27. [d] Ref. 14.

parameters is given in Table 4. From these data it can be seen that the structures differ in the Li–X distances and to some extent in the Li–Li distances as well as in the "openness" of the chair. The primary bonding in all cases is between the X atoms and the Li atoms with little or no direct Li–Li interaction.

The major point of discussion concerning these solid state structures is associated with the extent of Li–H interaction in the various compounds. The proposal for this type of interaction was first made by Craubner [28] and subsequently supported by Stucky et al. [13]. This stabilization also has been proposed by Shearer et al. [27] in the LiN=C(t-Bu)$_2$ bridged compounds. Further, it should be noted that in the tetrameric species such as (LiEt)$_4$ [29] and in the lithium tetramethylaluminate [30] the Li–H distances are relatively short, thus one may argue that the Li–H interactions are of significance. In opposition to this, it was suggested that this form of interaction was of limited significance in the (LiSiMe$_3$)$_6$ species where the structure is significantly expanded because of the increase in the Li–Si bond distance and the fact that no α-H atoms are present [16].

In the present system the calculated Li–α-H distances are quite short as indicated in Table 3 and clearly could contribute to stabilization of the structure. The problem associated with this argument is that in most of the instances cited, the Li–H separation also is dictated by the geometry associated with the other atoms of the species involved. Thus, even if the interaction contributes little to the stability of the complex, the hydrogen atoms will be located in the observed positions and might be considered to represent a classic case of the "chicken and the egg", i.e. is the location of the hydrogen dictated by bonding interaction or does the location result in some interaction.

We would like to propose that the dominant interaction determining the features of these structures is the steric effect of the substituent groups. A visual indication of this is given in Fig. 2 which shows a PLUTO drawing of the present system

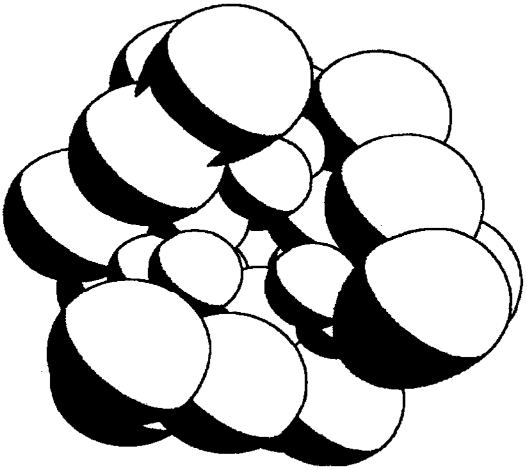

Fig. 2. A space filling PLUTO diagram drawn perpendicular to the plane described by Li(1)–Li(2)–Li(3) plane which shows the intra-molecular interactions and the shape of the (LiCH$_2$Si(CH$_3$)$_3$)$_6$ molecule. The covalent radii used are Li (1.34 Å), C (0.77 Å), and Si (1.18 Å), and Van der Waals radii of 2.0 Å for the methyl groups and 1.2 Å for the methylene H atoms.

utilizing the covalent radii for the Li (1.34 Å), C (0.77 Å), and, Si (1.18 Å) atoms. Van der Waals radii were used for the methyl groups (2.0 Å) and the methylene H atoms (1.2 Å) [31]. The congestion shown in Fig. 2 implies that significant repulsion terms occur between the various substituents and must influence the resulting structure.

The structures now available, provide solid information concerning the approximate location of the H atoms and their proximity to the lithium framework. It does not answer the question regarding Li–H bonding. NMR studies have shown that some form of Li–H interaction of some type exists, as shown by examination of the coupled and decoupled ^{6}Li spectra which show line narrowing with decoupling under all circumstances [21]. The coupling interaction shows magnetic effects are transmitted but these are normally associated with through bond interactions involving σ systems, not through space interactions of the type suggested.

There remain two areas of study which may shed light on this problem, (a) spectroscopic studies such as IR which may show direct Li–H interaction when a comparison is made between systems likely to have Li–H interactions and systems in which this is precluded by the proximity of the atoms and (b) theoretical calculations. We must await results from these areas before a final conclusion concerning the Li–H interaction can be drawn.

Acknowledgments

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