

Table 2. Selected geometric parameters (\AA , $^\circ$)

Sn1—S1	2.592 (2)	C2—C7	1.401 (5)
S1—C1	1.755 (4)	C3—C4	1.400 (7)
O1—C1	1.224 (5)	C4—C5	1.377 (8)
C1—C2	1.504 (5)	C5—C6	1.393 (8)
C2—C3	1.394 (6)	C6—C7	1.387 (6)
S1—Sn1—S1'	89.76 (6)	C7—C2—C1	122.7 (4)
C1—S1—Sn1	92.70 (13)	C2—C3—C4	120.2 (4)
O1—C1—C2	120.3 (4)	C5—C4—C3	120.1 (4)
O1—C1—S1	121.8 (3)	C4—C5—C6	120.1 (5)
C2—C1—S1	117.8 (3)	C7—C6—C5	120.2 (5)
C3—C2—C7	119.1 (4)	C6—C7—C2	120.2 (4)
C3—C2—C1	118.2 (3)		

Symmetry code: (i) $1 - x + y, 2 - x, z$.

All H atoms were placed in ideal calculated positions for the purpose of structure-factor calculations only. A common isotropic displacement parameter was refined.

Data collection: XSCANS (Siemens, 1990). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990a). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC (Sheldrick, 1990b). Software used to prepare material for publication: SHELXL93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1045). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Benzyllithium *tert*-Butyl Methyl Etherate†

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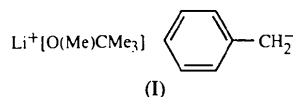
(Received 17 November 1995; accepted 19 January 1996)

Abstract

The title compound $[\text{Li}(\text{MeO}^i\text{Bu})\text{benzyl}]$, $[\text{Li}(\text{C}_7\text{H}_7)(\text{C}_5\text{H}_{12}\text{O})]$, forms infinite chains consisting of alternating lithium cations and benzyl anions, each of the latter being η^2 -coordinated through their C_α and C_{ipso} atoms to two lithium counterions.

Comment

The structure of $[\text{Li}(\text{MeO}^i\text{Bu})\text{benzyl}]$, (I) (Fig. 1), closely resembles that of $[\text{Li}(\text{OEt}_2)\text{benzyl}]$, determined previously (Beno, Hope, Olmstead & Power, 1985). Slight differences exist, mainly between the Li—C and Li—O bonds, clearly induced by the different ether coordinated to lithium. In contrast to the structure determination of $[\text{Li}(\text{OEt}_2)\text{benzyl}]$, in $[\text{Li}(\text{MeO}^i\text{Bu})\text{benzyl}]$ all H atoms could be located and refined. This enables the complete 'coordination' geometry of the benzyl-ic C_α atoms to be determined unambiguously. As a



result of the unsymmetrical η^2 -complexation of the C_α ($C7$) and C_{ipso} ($C1$) atoms of each benzyl anion by two Li atoms, the terminal C_α atoms adopt distorted trigonal bipyramidal 'pentacoordination' consisting of two H atoms and C_{ipso} in the equatorial plane (sum of the angles at C_α excluding the Li atoms: 360°) and two Li atoms in axial positions [$\text{Li}-C_\alpha-\text{Li}' = 151.6(1)^\circ$; see Fig. 1 and Table 2]. This coordination geometry of carbon was first suggested by calculations for $[\text{M}-\text{CH}_3-\text{M}]^+$ cations ($M = \text{Li}, \text{Na}$ and other metals) and could be verified in a small number of organometallic compounds (Schade, Ragué Schleyer, Dietrich & Mahdi, 1986, and references therein; Hoffmann *et al.*, 1994). The preferred coordination of benzyl anions to lithium via their C_α and C_{ipso} atoms was also demonstrated both by theory (Zarges, Marsch, Harms & Boche, 1989; Sygula & Rabideau, 1992, and references therein) and by structure determinations of the (pseudo)monomeric benzyllithium donor adducts $\{\text{Li}[\text{N}(\text{C}_2\text{H}_4)_3\text{N}]\text{benzyl}\}$ (Patterman, Karle & Stucky, 1970) and benzyllithium.thf.tmeda (thf = tetrahydro-

† (*tert*-Butyl methyl ether-*O*)lithium phenylmethanide.

furan; *tmeda* = tetramethylethylenediamine (Zarges *et al.*, 1989). In the latter structure, incompletely pyramidalized $C_{ipso}-C_{\alpha}H_2$ geometry was found, as a consequence of the coordination of the benzylic carbon to only one lithium cation.

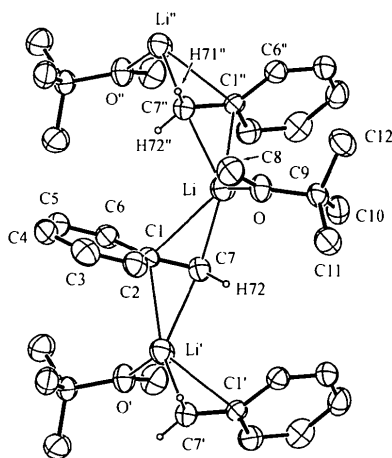


Fig. 1. Portion of the infinite chain structure of $[Li(MeO'Bu)benzyl]$ in the crystal and crystallographic numbering scheme adopted (ORTEP II; Johnson, 1976). Displacement ellipsoids are shown at the 50% level. For clarity, only the benzylic H atoms were drawn (spheres of arbitrary size). Primed atoms are related to those without a prime by $1-x, y+\frac{1}{2}, \frac{1}{2}-z$; symmetry code of doubly primed atoms: $1-x, y-\frac{1}{2}, \frac{1}{2}-z$.

Experimental

$[Li(MeO'Bu)benzyl]$ was obtained in lithiation experiments of benzyl phosphanes by P—C bond cleavage and recrystallized from *tert*-butyl methyl ether (Waldkircher, 1994). This ether was chosen because of its advantageous boiling point and its low tendency to form peroxides.

Crystal data

$[Li(C_7H_7)(C_5H_{12}O)]$

$M_r = 186.21$

Monoclinic

$P2_1/c$

$a = 13.103(4) \text{ \AA}$

$b = 7.734(1) \text{ \AA}$

$c = 13.392(4) \text{ \AA}$

$\beta = 119.09(1)^\circ$

$V = 1185.8(5) \text{ \AA}^3$

$Z = 4$

$D_r = 1.043 \text{ Mg m}^{-3}$

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 25

reflections

$\theta = 11.98\text{--}12.79^\circ$

$\mu = 0.062 \text{ mm}^{-1}$

$T = 198(2) \text{ K}$

Irregular

$0.3 \times 0.2 \times 0.2 \text{ mm}$

Colourless

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer

ω scans

Absorption correction: none

2786 measured reflections

2673 independent reflections

1353 observed reflections

$[I > 2\sigma(I)]$

$R_{int} = 0.0453$

$\theta_{max} = 27.36^\circ$

$h = 0 \rightarrow 16$

$k = 0 \rightarrow 9$

$l = -17 \rightarrow 15$

3 standard reflections

frequency: 60 min

intensity decay: 2.4%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.0526$

$wR(F^2) = 0.1379$

$S = 1.006$

2672 reflections

203 parameters

All H-atom parameters

refined

$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.022$

$\Delta\rho_{max} = 0.142 \text{ e \AA}^{-3}$

$\Delta\rho_{min} = -0.162 \text{ e \AA}^{-3}$

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

U_{iso} for H atoms; $U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$ for all others.

	x	y	z	U_{eq}/U_{iso}
Li	0.5829 (3)	0.1320 (5)	0.2627 (3)	0.0425 (9)
O	0.70846 (12)	0.1755 (2)	0.23019 (12)	0.0388 (4)
C1	0.4108 (2)	0.3401 (2)	0.1773 (2)	0.0316 (5)
C2	0.4188 (2)	0.3961 (3)	0.0805 (2)	0.0390 (6)
C3	0.3306 (2)	0.3686 (3)	-0.0296 (2)	0.0477 (6)
C4	0.2289 (2)	0.2834 (3)	-0.0519 (2)	0.0461 (6)
C5	0.2180 (2)	0.2278 (3)	0.0407 (2)	0.0432 (6)
C6	0.3044 (2)	0.2539 (3)	0.1506 (2)	0.0367 (5)
C7	0.5004 (2)	0.3660 (3)	0.2905 (2)	0.0379 (6)
C8	0.6792 (3)	0.1498 (4)	0.1136 (2)	0.0501 (7)
C9	0.8313 (2)	0.2039 (3)	0.3126 (2)	0.0350 (5)
C10	0.8328 (2)	0.2149 (4)	0.4253 (2)	0.0455 (6)
C11	0.8726 (2)	0.3718 (4)	0.2859 (3)	0.0500 (7)
C12	0.9044 (2)	0.0518 (4)	0.3103 (3)	0.0521 (7)
H71	0.488 (2)	0.335 (3)	0.354 (2)	0.043 (6)
H72	0.569 (2)	0.426 (3)	0.306 (2)	0.051 (7)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Li—O	1.925 (4)	C2—C3	1.377 (3)
Li—C7	2.230 (4)	C3—C4	1.383 (4)
Li—C7'	2.275 (4)	C4—C5	1.384 (4)
Li—C1'	2.384 (4)	C5—C6	1.367 (3)
Li—C1	2.544 (4)	C7—H71	0.97 (2)
O—C8	1.429 (3)	C7—H72	0.94 (3)
O—C9	1.459 (2)	C9—C10	1.502 (3)
C1—C7	1.409 (3)	C9—C11	1.515 (3)
C1—C2	1.419 (3)	C9—C12	1.526 (3)
C1—C6	1.425 (3)		
C7—Li—C1'	130.7 (2)	C5—C6—C1	122.5 (2)
C7'—Li—C1	104.2 (2)	C1—C7—Li	85.6 (2)
C8—O—C9	117.3 (2)	C1—C7—Li''	76.7 (2)
C8—O—Li	114.7 (2)	Li—C7—Li''	151.6 (1)
C9—O—Li	127.3 (2)	C1—C7—H71	120 (1)
C7—C1—C2	123.2 (2)	C1—C7—H72	121 (1)
C7—C1—C6	122.6 (2)	H71—C7—H72	119 (2)
C2—C1—C6	114.3 (2)	O—C9—C10	103.8 (2)
Li''—C1—Li	124.7 (1)	O—C9—C11	109.9 (2)
C3—C2—C1	122.3 (2)	C10—C9—C11	110.9 (2)
C2—C3—C4	121.6 (2)	O—C9—C12	110.0 (2)
C5—C4—C3	117.6 (2)	C10—C9—C12	111.2 (2)
C6—C5—C4	121.8 (2)	C11—C9—C12	110.9 (2)
C2—C1—C7—Li	-81.7 (2)	C2—C1—C7—Li''	75.9 (2)

Symmetry codes: (i) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MOLEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP II* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1111). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Acridine)trimethylgallium–Acridine (2/1)

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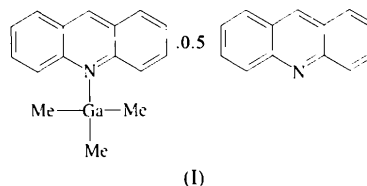
(Received 10 January 1995; accepted 23 October 1995)

Abstract

The reaction of excess trimethylgallium with acridine in diethyl ether gave the title layer inclusion compound, [Ga(CH₃)₃(C₁₃H₉N)]_{0.5}C₁₃H₉N. The Ga—N bond length is 2.203 (3) Å.

Comment

Metal-organic chemical vapour deposition (MOCVD) has become a powerful method for the preparation of thin films of electronic materials. A major effort has been concentrated on exploring the toxicity, stability and volatility of precursors for MOCVD (Bradley, Dawes, Hursthouse, Smith & Thornton-Pett, 1990; Bradley, Frigo, Harding, Hursthouse & Motevalli, 1992). The title compound, (I), was obtained and studied by X-ray analysis, revealing that the (acridine)trimethylgallium and free acridine molecules lie in layers.



Analysis of (I) (Fig. 1) shows that the asymmetric unit has one molecule of (acridine)trimethylgallium in a general position and an uncoordinated acridine molecule lying about an inversion centre (which requires that it be disordered). The Ga atom has distorted tetrahedral coordination, with N—Ga—C angles in the range 98.9(1)–108.3(1)° and C—Ga—C angles in the range 107.5(1)–117.3(1)°. The Ga—N distance of 2.203 (3) Å is slightly longer than previously reported

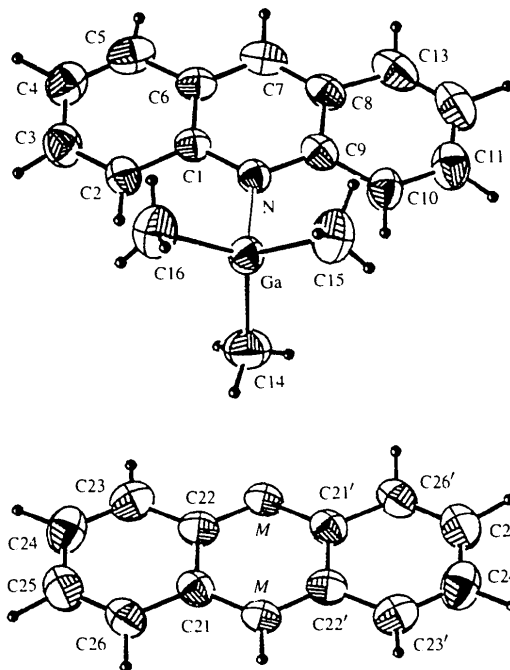


Fig. 1. The molecular structure of the title compound showing 50% probability displacement ellipsoids.