

C25	0.7422 (7)	0.1352 (7)	0.2620 (4)	0.072 (2)
C26	0.6015 (6)	0.1274 (6)	0.2750 (3)	0.055 (2)
C31	0.3038 (6)	-0.0400 (5)	0.3950 (2)	0.0406 (12)
C32	0.3903 (7)	-0.1238 (5)	0.4260 (3)	0.0500 (14)
C33	0.3343 (9)	-0.2210 (6)	0.4605 (3)	0.067 (2)
C34	0.1965 (10)	-0.2363 (6)	0.4640 (3)	0.078 (2)
C35	0.1099 (8)	-0.1549 (7)	0.4320 (4)	0.082 (2)
C36	0.1636 (6)	-0.0580 (6)	0.3980 (3)	0.062 (2)
C41	0.4172 (6)	-0.1721 (5)	0.2504 (2)	0.0403 (12)
C42	0.5572 (6)	-0.1767 (5)	0.2591 (3)	0.0490 (14)
C43	0.6207 (7)	-0.2735 (6)	0.2910 (3)	0.063 (2)
C44	0.5442 (8)	-0.3674 (6)	0.3151 (3)	0.070 (2)
C45	0.4046 (8)	-0.3648 (6)	0.3084 (3)	0.065 (2)
C46	0.3423 (6)	-0.2680 (5)	0.2754 (3)	0.0520 (14)
C51	0.1986 (6)	-0.1242 (5)	0.1722 (3)	0.0476 (14)
C52	0.0679 (7)	-0.1161 (7)	0.2054 (4)	0.072 (2)
C53	-0.0398 (8)	-0.1739 (8)	0.1817 (5)	0.093 (3)
C54	-0.0161 (10)	-0.2387 (7)	0.1253 (5)	0.092 (3)
C55	0.1113 (10)	-0.2486 (7)	0.0926 (4)	0.087 (3)
C56	0.2194 (7)	-0.1912 (5)	0.1163 (3)	0.060 (2)
C61	0.4665 (6)	-0.0231 (5)	0.1385 (2)	0.0439 (13)
C62	0.4890 (7)	0.0944 (6)	0.1159 (3)	0.057 (2)
C63	0.5922 (8)	0.1127 (7)	0.0664 (3)	0.079 (2)
C64	0.6729 (8)	0.0146 (8)	0.0384 (4)	0.083 (2)
C65	0.6527 (7)	-0.1017 (8)	0.0600 (3)	0.075 (2)
C66	0.5496 (7)	-0.1215 (6)	0.1095 (3)	0.059 (2)

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

Pt—C1	2.053 (5)	P1—C11	1.846 (5)
Pt—C1'	2.095 (5)	P2—C61	1.831 (5)
Pt—P1	2.311 (1)	P2—C51	1.836 (6)
Pt—P2	2.348 (1)	P2—C41	1.845 (5)
P1—C21	1.829 (5)	C1—C6	1.334 (7)
P1—C31	1.828 (5)	C1'—C2'	1.358 (8)
C1—Pt—C1'	77.7 (2)	C61—P2—C51	105.6 (3)
C1—Pt—P1	92.99 (15)	C61—P2—C41	101.1 (2)
C1'—Pt—P1	168.4 (2)	C51—P2—C41	99.7 (2)
C1—Pt—P2	171.33 (15)	C61—P2—Pt	115.0 (2)
C1'—Pt—P2	95.1 (2)	C51—P2—Pt	111.9 (2)
P1—Pt—P2	94.77 (5)	C41—P2—Pt	121.4 (2)
C21—P1—C31	110.2 (2)	C6—C1—C2	120.2 (5)
C21—P1—C11	97.4 (2)	C6—C1—Pt	126.4 (4)
C31—P1—C11	101.5 (2)	C2—C1—Pt	112.5 (4)
C21—P1—Pt	113.8 (2)	C2'—C1'—C6'	118.2 (5)
C31—P1—Pt	113.0 (2)	C2'—C1'—Pt	113.4 (4)
C11—P1—Pt	119.3 (2)	C6'—C1'—Pt	127.8 (4)

The intensity contribution of the disordered tetrahydrofuran molecule was calculated with *PLATON/SQUEEZE* (Spek, 1994) and subtracted from the full data set; ratio of complex to tetrahydrofuran was found to be 1:0.85. The new data set, with the solvent contribution removed, was used in the final refinement.

Data collection: *SHELXTL-Plus* (Sheldrick, 1991). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: FG1046). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 1911–1913

(Diethyl ether-*O*)[*N*-(2,6-diisopropylphenyl)-*N*-(trimethylsilyl)amido-*N*-(pyridine-*N*)lithium

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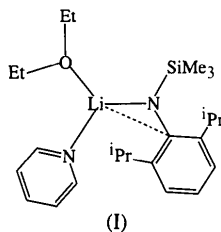
(Received 28 February 1996; accepted 25 March 1996)

Abstract

The title compound, $[\text{Li}(\text{C}_{15}\text{H}_{26}\text{NSi})(\text{C}_4\text{H}_{10}\text{O})(\text{C}_5\text{H}_5\text{N})]$, contains an Li atom with approximately trigonal-planar coordination, whose principal coordination sphere consists of one *N*-(2,6-diisopropylphenyl)-*N*-(trimethylsilyl)amido, one pyridine and one diethyl ether ligand. There is a further possible bonding contact between the Li atom and the *N*-*ipso* C atom of the 2,6-diisopropylphenyl group.

Comment

Lithium amides are of considerable importance in organic and inorganic synthesis, and their structures have been the focus of much attention (Gregory, Schleyer & Snaith, 1991). The structures of two monomeric lithium arylamides have been described previously; [Li{N(H)(2,4,6-C₆H₂'Bu₃)}(tmeda)] (tmeda is *N,N,N',N'*-tetramethyldiaminoethane) (Fjeldberg, Hitchcock, Lappert & Thorne, 1984) and [Li{N(Si^{*i*}Pr₂Cl)(2,4,6-C₆H₂'Bu₃)}(thf)₂] (thf is tetrahydrofuran) (Boese & Klingebiel, 1986). The compound [Li{N(C₆H₅)(C₁₀H₇)}(tmeda)] (C₁₀H₇ is naphthyl) (Barr, Clegg, Mulvey, Snaith & Wright, 1987) exists as a 'loose dimer' in the solid state through weak intermolecular interactions. In all of these complexes, the coordination number of Li exceeds three through either intermolecular or intramolecular ligation by atoms other than the 'primary' O and/or N Lewis-base donor atoms. In contrast, the monomeric lithium bis(triarylsilyl or diaryl monoalkylsilyl)amides [Li{N(SiPh₂R)₂}(thf)₂] (R = Ph or Me) have approximately trigonal-planar Li centres with apparently no close bonding contacts between Li and non-N or non-O atoms (Chen, Bartlett, Dias, Olmstead & Power, 1989). We describe here the structure of [Li{N(SiMe₃)(2,6-C₆H₃'Pr₂)}(py)(OEt₂)] (py is pyridine), (I).



The coordination geometry around the Li atom in (I) is slightly distorted trigonal planar (Fig. 1). The angles around Li1 sum to 359.9(4)° and the deviation of Li1 from the plane containing atoms N1, N2 and O1 is 0.006(3) Å. The variations in the angles subtended at Li1 appear to reflect the relative steric demands of the ligating groups. The Li1...C1 distance of 2.714(9) Å may represent a loose bonding contact and the next closest contact made by Li1 is 2.940(10) Å to C11. An Li—C_{ipso} distance of 2.466(10) Å is observed in [Li{N(Si^{*i*}Pr₂Cl)(2,4,6-C₆H₂'Bu₃)}(thf)₂] (Boese & Klingebiel, 1986). In the dimeric lithium arylamido complex [(μ-Li)₂{N(SiMe₃)(2,6-C₆H₃'Pr₂)}₂] (Kenepohl, Brooker, Sheldrick & Roesky, 1991), the unsolvated analogue of the title compound, Li—C_{ipso} distances of 2.424(5), 2.585(5), 2.668(5) and 2.771(4) Å are found. The Li1—N1—Si1 and Li1—N1—C1 angles [123.2(3) and 109.0(4)°, respectively] are possible consequences of a loose bonding contact between the Li1 and C1 atoms. The N1 atom is trigonal planar and the angle between the least-squares plane containing atoms

Li1, N1, O1 and N2, and that containing N1, Li1, Si1 and C1 is 0.2(9)°. The plane of the six C atoms of the 2,6-diisopropylphenyl ring (C1—C6) is almost normal to these planes [angles between planes 85.2(9) and 85.3(9)°, respectively]. This is possibly a consequence of the sterically demanding diisopropyl groups, but also allows for interaction of the out-of-plane *p*-π orbital of C1 with Li1.

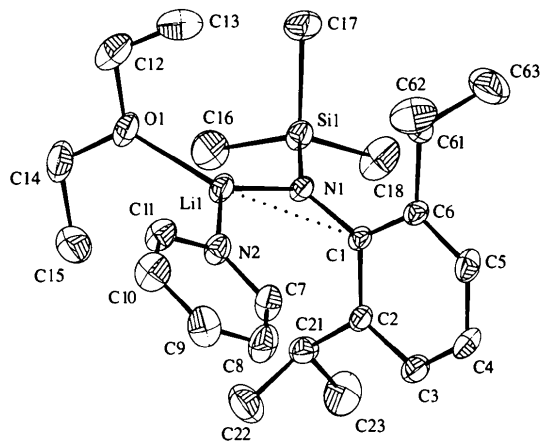


Fig. 1. View of the title compound with the atom-numbering scheme. The displacement ellipsoids are drawn at the 20% probability level and H atoms have been omitted for clarity.

Experimental

2,6-Diisopropylaniline in hexane was treated with *n*-butyllithium in hexane. Crystallization of the resultant solid from diethyl ether in the presence of pyridine afforded the title compound as pale yellow air- and moisture-sensitive crystals.

Crystal data

[Li(C₁₅H₂₆NSi)(C₄H₁₀O)-
(C₅H₅N)]
*M*_r = 408.63
Monoclinic
*P*2₁/*n*
a = 12.556(6) Å
b = 14.408(7) Å
c = 14.996(8) Å
β = 91.56(2)°
V = 2712(2) Å³
Z = 4
*D*_x = 1.001 Mg m⁻³
*D*_m not measured

Mo *K*α radiation
λ = 0.71073 Å
Cell parameters from 13
reflections
θ = 14–17°
μ = 0.101 mm⁻¹
T = 298 K
Column
0.96 × 0.42 × 0.40 mm
Pale yellow

Data collection

Stoe Stadi-4 four-circle
diffractometer
ω-*θ* scans
Absorption correction:
none

*R*_{int} = 0.045
*θ*_{max} = 25°
h = -14 → 14
k = -1 → 17
l = 0 → 17

6695 measured reflections
3840 independent reflections
1698 observed reflections
[$I > 2\sigma(I)$]

Refinement

Refinement on F
 $R = 0.0749$
 $wR = 0.0655$
 $S = 1.0673$
1698 reflections
262 parameters
H atoms placed in calculated positions 1.00 Å from the supporting C atom and with $U_{\text{iso}}(\text{H}) = 1.3U_{\text{eq}}(\text{C})$

3 standard reflections
frequency: 60 min
intensity decay: 2.5%

Weighting scheme from Chebychev polynomial (Carruthers & Watkin, 1979)
 $(\Delta/\sigma)_{\text{max}} = 0.25$
 $\Delta\rho_{\text{max}} = 0.23 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.22 \text{ e } \text{Å}^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

ray beam in all orientations, it might have been preferable to transfer them to an inert oil and cut them there. However, given the low value of the absorption coefficient and the absence of extinction effects in this particular case, this does not appear to have given rise to significant problems. We attribute the somewhat high residuals to the poor diffracting power of the crystal, resulting in a data set with a mean $I/\sigma(I)$ of only 5.69, and to the fact that the structure contains a large number of methyl groups whose librational motion may be difficult to model accurately. There are no indications, however, that a static disorder model is appropriate. The reversed order of the weighted and unweighted residuals does not have an obvious explanation; only one reflection appears to have been down-weighted and the agreement analysis is satisfactory.

Data collection: *STADIA* (Stoe & Cie, 1995a). Cell refinement: *STADIA*. Data reduction: *X-RED* (Stoe & Cie, 1995b). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994). Program(s) used to refine structure: *CRYSTALS* (Watkin, Prout, Carruthers & Betteridge, 1996). Molecular graphics: *CAMERON* (Watkin, Prout & Pearce, 1996). Software used to prepare material for publication: *CRYSTALS*.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j$$

	x	y	z	U_{eq}
Li1	0.8527 (7)	0.2335 (7)	0.1426 (5)	0.060 (3)
N1	0.9937 (3)	0.2351 (3)	0.1946 (2)	0.048 (2)
N2	0.7135 (3)	0.2006 (3)	0.2049 (3)	0.064 (2)
O1	0.8083 (3)	0.2610 (3)	0.0198 (2)	0.072 (2)
Si1	1.1015 (1)	0.2634 (1)	0.1378 (1)	0.0620 (5)
C1	0.9887 (4)	0.2105 (4)	0.2846 (3)	0.044 (3)
C2	1.0013 (4)	0.1162 (4)	0.3117 (3)	0.051 (3)
C3	0.9856 (5)	0.0924 (4)	0.4002 (4)	0.067 (3)
C4	0.9581 (5)	0.1562 (5)	0.4618 (4)	0.067 (3)
C5	0.9472 (4)	0.2480 (5)	0.4376 (4)	0.069 (3)
C6	0.9626 (4)	0.2764 (4)	0.3507 (3)	0.051 (3)
C7	0.7093 (5)	0.1678 (5)	0.2876 (4)	0.077 (3)
C8	0.6147 (7)	0.1460 (5)	0.3269 (4)	0.088 (3)
C9	0.5216 (6)	0.1600 (6)	0.2791 (5)	0.089 (3)
C10	0.5256 (5)	0.1935 (5)	0.1953 (6)	0.092 (3)
C11	0.6214 (5)	0.2130 (4)	0.1606 (4)	0.071 (3)
C12	0.7808 (6)	0.3492 (6)	-0.0150 (5)	0.103 (4)
C13	0.7716 (7)	0.4174 (6)	0.0579 (6)	0.121 (4)
C14	0.8055 (7)	0.1916 (6)	-0.0469 (5)	0.102 (4)
C15	0.8244 (8)	0.0985 (6)	-0.0055 (6)	0.125 (4)
C16	1.1117 (6)	0.1965 (6)	0.0320 (5)	0.108 (4)
C17	1.1037 (6)	0.3871 (5)	0.1004 (5)	0.094 (3)
C18	1.2280 (4)	0.2441 (7)	0.2026 (5)	0.104 (4)
C21	1.0304 (5)	0.0422 (4)	0.2452 (4)	0.067 (2)
C22	0.9388 (7)	-0.0227 (5)	0.2248 (5)	0.115 (4)
C23	1.1279 (6)	-0.0139 (6)	0.2768 (5)	0.109 (4)
C61	0.9513 (5)	0.3774 (4)	0.3277 (4)	0.070 (2)
C62	0.8458 (6)	0.4179 (5)	0.3495 (6)	0.116 (4)
C63	1.0402 (7)	0.4352 (5)	0.3675 (7)	0.130 (5)

Table 2. Selected geometric parameters (Å , °)

Li1—N1	1.916 (9)	Li1...C1	2.714 (9)
Li1—N2	2.06 (1)	N1—Si1	1.669 (4)
Li1—O1	1.951 (8)	N1—C1	1.398 (6)
N1—Li1—N2	127.1 (4)	Li1—N1—Si1	123.2 (3)
N1—Li1—O1	128.3 (5)	Li1—N1—C1	109.0 (4)
N2—Li1—O1	104.5 (4)	Si1—N1—C1	127.8 (3)

Owing to their sensitivity to atmosphere, crystals could not be cut and were supplied mounted in glass capillary tubes. As such long crystals may not be uniformly irradiated by the X-

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1370). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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