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The crystal structure of bis{(μ_2 -3,3-dimethyl-1-phenylbut-1-en-2-yl)((dimethylamino)dimethylsilyl)amido- $\kappa^3 N, N': N'$ }dilithium, $C_{32}H_{54}Li_2N_4Si_2$

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Abstract

$C_{32}H_{54}Li_2N_4Si_2$, monoclinic, $P2_1/n$ (no. 14), $a = 11.611(4)$ Å, $b = 11.157(6)$ Å, $c = 13.307(5)$ Å, $\beta = 100.12(3)^\circ$, $V = 1697.0(12)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0622$, $wR_{ref}(F^2) = 0.1561$, $T = 213$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.30 \times 0.20 \times 0.20$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.13 mm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX II, φ and ω
$\theta_{\max}$, completeness:	25.0° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6768, 2982, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2878
$N(\text{param})_{\text{refined}}$:	199
Programs:	Siemens [1], ORTEP-3 [2], SHELX [3–5]

Source of material

All manipulations were carried out using standard Schlenk and glovebox techniques under an argon or nitrogen atmosphere. The title compound was synthesized according to literature methods [6–8]. To a solution of ((dimethylamino)dimethylsilyl)toluollithium (6 mmol) in diethyl ether (20 ml), *tert*-butyl nitrile (6 mmol) was added at *ca.* 273 K and the solution was stirred for 15 min and then kept stirring for 5 h at room temperature. The solvent was slowly concentrated under vacuum, crystals of the title compound were obtained within two days.

Experimental details

Coordinates of hydrogen atoms were refined without any constraints or restraints. Their U_{iso} values were set to $1.2U_{\text{eq}}$ of the parent atoms.

Comment

Amidinate compounds are well known and have played important role in searching for novel spectator ligands [6–8, 13]. Lappert and co-workers have prepared a variety of complexes including some main group elements and transition metals [9–12]. Quite a long time ago we have got similar complexes [14, 15]. As part of an subsequent

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	U_{iso}^*/U_{eq}
Li1	0.5090 (4)	0.0789 (4)	0.0653 (3)	0.0317 (10)
N1	0.36147 (18)	−0.02269 (18)	0.00870 (16)	0.0262 (5)
N2	0.45798 (19)	0.0373 (2)	0.20620 (16)	0.0312 (5)
Si1	0.34215 (6)	−0.04527 (6)	0.13059 (6)	0.0272 (2)
C1	0.2707 (3)	0.2271 (2)	−0.0360 (2)	0.0340 (6)
C2	0.1946 (3)	0.3073 (3)	−0.0014 (2)	0.0441 (7)
H2	0.114229	0.289556	−0.011757	0.053*
C3	0.2343 (3)	0.4125 (3)	0.0479 (3)	0.0512 (9)
H3	0.180700	0.465128	0.070118	0.061*
C4	0.3511 (3)	0.4407 (3)	0.0647 (3)	0.0498 (8)
H4	0.377999	0.511409	0.099518	0.060*
C5	0.4287 (3)	0.3641 (3)	0.0299 (2)	0.0430 (7)
H5	0.508812	0.383099	0.040122	0.052*
C6	0.3886 (3)	0.2592 (2)	−0.0200 (2)	0.0360 (7)
H6	0.442412	0.208271	−0.043872	0.043*
C7	0.2258 (2)	0.1167 (3)	−0.0896 (2)	0.0355 (6)
H7	0.162078	0.125252	−0.143484	0.043*
C8	0.2666 (2)	0.0048 (2)	−0.0693 (2)	0.0296 (6)
C9	0.2019 (2)	−0.1010 (3)	−0.1301 (2)	0.0376 (7)
C10	0.1335 (3)	−0.0627 (4)	−0.2349 (3)	0.0578 (10)
H10A	0.185061	−0.019013	−0.271899	0.087*
H10B	0.068690	−0.011671	−0.225411	0.087*
H10C	0.103798	−0.133289	−0.273532	0.087*
C11	0.1145 (3)	−0.1557 (3)	−0.0684 (3)	0.0508 (9)
H11A	0.072321	−0.220685	−0.106995	0.076*
H11B	0.059627	−0.094604	−0.055308	0.076*
H11C	0.156609	−0.186287	−0.004093	0.076*
C12	0.2888 (3)	−0.1986 (3)	−0.1472 (3)	0.0509 (9)
C13	0.1986 (2)	0.0090 (3)	0.1588 (2)	0.0395 (7)
H13A	0.177577	0.083482	0.122537	0.059*
H13B	0.205212	0.022378	0.231567	0.059*
H13C	0.138785	−0.050680	0.136753	0.059*
C14	0.3595 (3)	−0.2044 (3)	0.1753 (3)	0.0436 (7)
H14A	0.297399	−0.252848	0.137145	0.065*
H14B	0.355473	−0.208114	0.247413	0.065*
H14C	0.434654	−0.234776	0.164459	0.065*
C15	0.4246 (3)	0.1518 (3)	0.2479 (2)	0.0418 (7)
H15A	0.388402	0.136531	0.307042	0.063*
H15B	0.369704	0.193570	0.196358	0.063*
H15C	0.493833	0.200914	0.267952	0.063*
C16	0.5414 (3)	−0.0256 (3)	0.2841 (2)	0.0482 (8)
H16A	0.604663	0.028226	0.311720	0.072*
H16B	0.572831	−0.094484	0.253573	0.072*
H16C	0.501790	−0.052198	0.338534	0.072*
H12A	0.347 (3)	−0.168 (3)	−0.188 (2)	0.046 (9)*
H12B	0.246 (3)	−0.267 (3)	−0.182 (3)	0.062 (10)*
H12C	0.332 (3)	−0.230 (3)	−0.079 (3)	0.057 (10)*

investigation of the chemical and physical properties of amidinate complexes, we have prepared the title complex, and present its structure here (the Figure).

The title compound is a centrosymmetric dimer which contains two NSiN fragments bonded to two central Li atoms. Thus it is a good spectator ligand to study its conjugation and chemical properties. The two amidinate ligands bound to two Li atoms, with Li–N bond lengths 2.081(5), 2.114(5) and 2.036(5) $\text{\AA}$. The central Li atoms are part of three planes: two crystallographically identical four-membered planes (N1/Si1/N2/Li1 with RMS deviation = 0.0111 $\text{\AA}$) and one central four-membered ring (N/Li with symmetry). The former plane encloses an angle of 45.7(3)° with the latter plane. And these three planes form a cascade-step configuration. Each lithium atom is surrounded by three nitrogen atoms. Some slightly different Li complexes have been found with similar configuration which have similar manner bonded to central Li atoms [8, 12, 14, 16].

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