Crystal structure of bis-μ_3(*N,N'*-bis-(trimethylsilyl)-pyridine-2-amine-6-amido)-μ_4(*N,N'*-bis-(trimethylsilyl)-pyridine-2,6-diamido)-di-tetrahydrofuran-tetralithium, $[(C_{11}H_{21}N_3Si_2)(C_{11}H_{22}N_3Si_2)_2(C_4H_8O)_2Li_4]$

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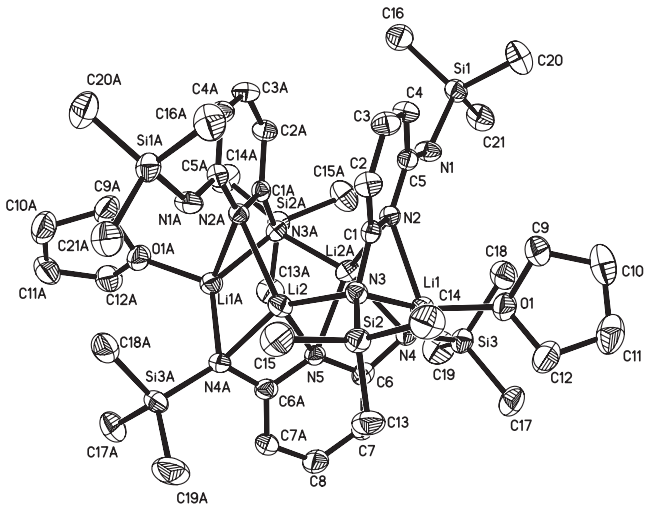

Abstract

$C_{41}H_{81}Li_4N_9O_2Si_6$, monoclinic, *C*12/*c*1 (no. 15), *a* = 24.285(1) Å, *b* = 11.8023(7) Å, *c* = 20.221(1) Å, β = 104.798(5)°, *V* = 5603.5 Å^3, *Z* = 4, $R_{gt}(F)$ = 0.043, $wR_{ref}(F^2)$ = 0.098, *T* = 133 K.

Source of material

A solution of two equivalents of *tert*-butyl-(4-methyl- pyridin-2-yl)-amine in THF was treated at –50 °C with two equivalents of n-butyllithium followed by a reaction with one equivalent of $CuBr_2$ suspended in THF. After removal of the solvent the residue was extracted twice with hexane and filtered. Colourless crystals suitable for X-ray analysis were obtained after slow evaporation of the solvent.

Experimental details

The H atoms bonded to the N atoms were located and refined. The remaining H atoms were accounted at calculated positions.

Discussion

To our best knowledge there are no metal complexes known containing the *N,N'*-bis-(trimethylsilyl)-pyridine-2,6-amine ligand in its neutral, deprotonated or double deprotonated form [1,2]. Only a few examples of complexes of an cyclic ligand showing a similarity to the ligand presented here are existing but were not characterized *via* X-ray single crystal structure analysis [3].

The title complex contains one double deprotonated ligand and two single deprotonated ligands coordinating four lithium atoms. The coordination of each lithium atom is a best described as distorted tetrahedron. The Li2 atom is coordinated by two $N_{pyridine}$ atoms and two N_{amido} atoms with d(Li2—N_{amido}) = 2.014 Å or 2.053 Å and d(Li2—$N_{pyridine}$) = 2.109 Å or 2.287 Å. The Li1 atom is coordinated by two N_{amido} atoms, one $N_{pyridine}$ atom and one O atom with d(Li1—N_{amido}) = 2.054 Å or 2.086 Å; d(Li1—$N_{pyridine}$) = 2.179 Å) and d(Li1—O) = 1.931 Å].

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.31 × 0.40 × 0.75 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.88 cm^{-1}
Diffractometer, scan mode:	STOE-IPDSII, ω
$2\theta_{max}$:	51.52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	35770, 5312
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3954
$N(param)_{refined}$:	294
Programs:	SIR-97 [4], SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	8*f*	0.4279	0.5037	0.8506	0.035
H(3)	8*f*	0.4842	0.6630	0.8544	0.038
H(4)	8*f*	0.5779	0.6467	0.8459	0.035
H(7)	8*f*	0.575	–0.1244	0.8221	0.047
H(8)	4*e*	½	–0.2216	¾	0.052
H(9A)	8*f*	0.5546	0.3982	1.0158	0.056
H(9B)	8*f*	0.6017	0.3944	0.9724	0.056
H(10A)	8*f*	0.6323	0.3761	1.1048	0.066
H(10B)	8*f*	0.6676	0.3114	1.0585	0.066
H(11A)	8*f*	0.6400	0.1478	1.0944	0.069
H(11B)	8*f*	0.5987	0.2126	1.1328	0.069
H(12A)	8*f*	0.5605	0.0938	1.0193	0.057
H(12B)	8*f*	0.5231	0.1888	1.0450	0.057
H(13A)	8*f*	0.4067	0.0627	0.8380	0.062
H(13B)	8*f*	0.3790	0.0745	0.9014	0.062
H(13C)	8*f*	0.4467	0.0800	0.9135	0.062
H(14A)	8*f*	0.4497	0.3241	0.9821	0.071
H(14B)	8*f*	0.3857	0.2859	0.9780	0.071
H(14C)	8*f*	0.3977	0.4051	0.9466	0.071
H(15A)	8*f*	0.3311	0.3721	0.8045	0.073
H(15B)	8*f*	0.3044	0.2662	0.8348	0.073
H(15C)	8*f*	0.3292	0.2503	0.7692	0.073
H(16A)	8*f*	0.6743	0.6067	0.7256	0.067
H(16B)	8*f*	0.7008	0.7106	0.7743	0.067
H(16C)	8*f*	0.6342	0.6830	0.7592	0.067
H(17A)	8*f*	0.6366	0.0087	0.9871	0.073
H(17B)	8*f*	0.6857	–0.0682	0.9706	0.073
H(17C)	8*f*	0.6203	–0.0966	0.9359	0.073
H(18A)	8*f*	0.7037	0.2279	0.8776	0.070

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Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(18B)	8*f*	0.7262	0.1635	0.9490	0.070
H(18C)	8*f*	0.6690	0.2369	0.9349	0.070
H(19A)	8*f*	0.6685	0.0805	0.8010	0.086
H(19B)	8*f*	0.7271	0.0407	0.8519	0.086
H(19C)	8*f*	0.6968	0.0373	0.7883	0.086
H(20A)	8*f*	0.6651	0.6888	0.9183	0.076

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(20B)	8*f*	0.7326	0.6825	0.9282	0.076
H(20C)	8*f*	0.7009	0.5863	0.9603	0.076
H(21A)	8*f*	0.7588	0.4206	0.8875	0.064
H(21B)	8*f*	0.7831	0.5156	0.8461	0.064
H(21C)	8*f*	0.7436	0.4163	0.8058	0.064
H(1N)	8*f*	0.6378(8)	0.398(2)	0.829(1)	0.019(6)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*f*	0.48790(7)	0.3891(2)	0.83702(9)	0.0239(9)	0.0255(9)	0.0184(9)	0.0006(7)	0.0053(7)	0.0007(7)
C(2)	8*f*	0.46576(8)	0.4965(2)	0.8463(1)	0.027(1)	0.028(1)	0.034(1)	0.0039(8)	0.0122(8)	0.0003(8)
C(3)	8*f*	0.49928(8)	0.5904(2)	0.8489(1)	0.036(1)	0.023(1)	0.038(1)	0.0054(8)	0.0142(9)	0.0008(8)
C(4)	8*f*	0.55455(8)	0.5815(2)	0.8437(1)	0.031(1)	0.023(1)	0.036(1)	0.0039(8)	0.0092(9)	0.0012(8)
C(5)	8*f*	0.57503(7)	0.4744(2)	0.83523(9)	0.0222(9)	0.0261(9)	0.023(1)	0.0010(7)	0.0053(7)	0.0020(7)
C(6)	8*f*	0.54404(8)	0.0348(2)	0.7931(1)	0.037(1)	0.025(1)	0.024(1)	0.0044(8)	0.0108(8)	0.0002(8)
C(7)	8*f*	0.5445(1)	0.0839(2)	0.7930(1)	0.051(1)	0.024(1)	0.036(1)	0.0071(9)	0.001(1)	0.0016(9)
C(8)	4*e*	½	0.1411(2)	¾	0.065(2)	0.021(2)	0.039(2)	0	0.003(2)	0
C(9)	8*f*	0.5847(1)	0.3515(2)	1.0043(1)	0.062(2)	0.041(1)	0.032(1)	0.004(1)	0.004(1)	0.007(1)
C(10)	8*f*	0.6298(1)	0.3189(2)	1.0683(1)	0.047(1)	0.073(2)	0.040(1)	0.005(1)	0.003(1)	0.019(1)
C(11)	8*f*	0.6097(1)	0.2057(2)	1.0891(1)	0.062(2)	0.060(2)	0.042(1)	0.025(1)	0.001(1)	0.003(1)
C(12)	8*f*	0.5590(1)	0.1748(2)	1.0316(1)	0.066(2)	0.043(1)	0.033(1)	0.009(1)	0.010(1)	0.006(1)
C(13)	8*f*	0.41006(9)	0.0998(2)	0.8822(1)	0.041(1)	0.036(1)	0.050(1)	0.0082(9)	0.017(1)	0.007(1)
C(14)	8*f*	0.4103(1)	0.3262(2)	0.9542(1)	0.056(1)	0.053(2)	0.041(1)	0.006(1)	0.029(1)	0.004(1)
C(15)	8*f*	0.33410(9)	0.2903(2)	0.8128(1)	0.027(1)	0.060(2)	0.060(2)	0.000(1)	0.011(1)	0.014(1)
C(16)	8*f*	0.67227(9)	0.6499(2)	0.7663(1)	0.038(1)	0.047(1)	0.048(1)	0.012(1)	0.008(1)	0.008(1)
C(17)	8*f*	0.6479(1)	0.0355(2)	0.9516(1)	0.051(1)	0.049(1)	0.041(1)	0.010(1)	0.001(1)	0.013(1)
C(18)	8*f*	0.69223(9)	0.1871(2)	0.9141(1)	0.034(1)	0.045(1)	0.056(2)	0.004(1)	0.001(1)	0.000(1)
C(19)	8*f*	0.6904(1)	0.0150(2)	0.8232(2)	0.054(2)	0.058(2)	0.066(2)	0.023(1)	0.025(1)	0.001(1)
C(20)	8*f*	0.6976(1)	0.6378(2)	0.9215(1)	0.042(1)	0.061(2)	0.045(1)	0.009(1)	0.005(1)	0.015(1)
C(21)	8*f*	0.75055(8)	0.4663(2)	0.8457(1)	0.022(1)	0.049(1)	0.056(2)	0.0061(9)	0.010(1)	0.003(1)
N(1)	8*f*	0.63088(7)	0.4582(2)	0.83302(9)	0.0243(8)	0.022(1)	0.044(1)	0.0005(7)	0.0099(7)	0.0002(8)
N(2)	8*f*	0.54279(6)	0.3803(1)	0.83028(7)	0.0210(7)	0.0220(8)	0.0231(8)	0.0003(6)	0.0055(6)	0.0008(6)
N(3)	8*f*	0.46048(6)	0.2887(1)	0.83457(8)	0.0249(8)	0.0245(8)	0.0238(8)	0.0011(6)	0.0069(6)	0.0017(6)
N(4)	8*f*	0.58414(7)	0.1058(1)	0.83227(8)	0.0317(8)	0.0223(8)	0.0258(8)	0.0052(7)	0.0048(7)	0.0004(6)
N(5)	4*e*	½	0.0910(2)	¾	0.029(1)	0.021(1)	0.022(1)	0	0.0068(9)	0
Si(1)	8*f*	0.68647(2)	0.55440(5)	0.84142(3)	0.0237(3)	0.0335(3)	0.0323(3)	0.0060(2)	0.0050(2)	0.0007(2)
Si(2)	8*f*	0.40584(2)	0.25630(5)	0.86997(3)	0.0244(3)	0.0310(3)	0.0300(3)	0.0022(2)	0.0101(2)	0.0020(2)
Si(3)	8*f*	0.64979(2)	0.05923(5)	0.87760(3)	0.0308(3)	0.0301(3)	0.0331(3)	0.0091(2)	0.0058(2)	0.0024(2)
O(1)	8*f*	0.56229(6)	0.2458(1)	0.97516(7)	0.0533(9)	0.0356(8)	0.0256(7)	0.0003(7)	0.0051(6)	0.0008(6)
Li(1)	8*f*	0.5397(1)	0.2158(3)	0.8781(2)	0.033(2)	0.028(2)	0.027(2)	0.002(1)	0.008(1)	0.000(1)
Li(2)	8*f*	0.4451(1)	0.2322(3)	0.7376(2)	0.031(2)	0.026(2)	0.030(2)	0.003(1)	0.008(1)	0.004(1)

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