Crystal structure of (1-cyano-indenyl-*N*)-((–)spateine-*N*,*N*')-(tetrafuran-*O*)-lithium, $C_{29}H_{40}LiN_3O$

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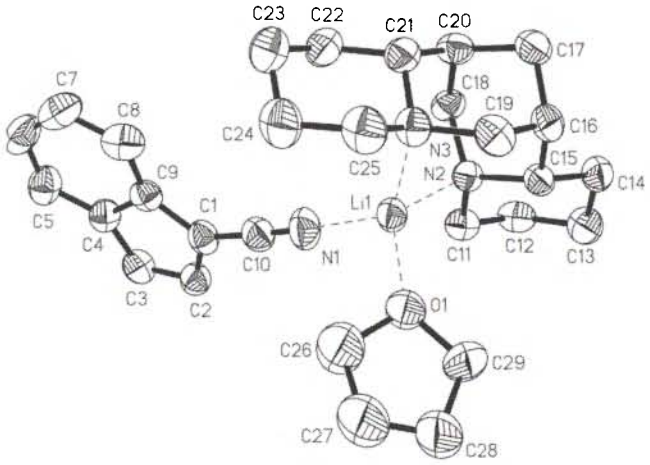

Abstract

$C_{29}H_{40}LiN_3O$, monoclinic, $P12_11$(No. 4), a = 8.403(1) Å, b = 17.024(2) Å, c = 9.406(2) Å, β = 100.805(9)°, V = 1321.7 Å^3, Z = 2, $R_{gt}(F)$ = 0.043, $wR(F^2)$ = 0.113, T = 193 K.

Discussion

The absolute configuration could be determined through the known configuration of (–)sparteine. Besides earlier published structures of dimers of lithiated cyanides [1, 2], the structure of (1-cyano-indenyl-*N*)-((–)spateine-*N*,*N*')-(tetrafuran-*O*)-lithium together with the structure (benzylcyano-*N*)-12-crown-4-*O*,*O*',*O*'',*O*''')-lithium [3] reveals for the first time a structure of a lithiated cyanide which is a monomer.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.25 × 0.3 × 0.35 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	5.24 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω
$2\theta_{max}$:	110.26°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3973, 2744
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2545
$N(param)_{refined}$:	318
Programs:	SHELXL-93 [4], SHELXS-86 [5], XCAD4 [6]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*a*	0.625(5)	0.523(2)	-0.271(4)	0.07(1)
H(2)	2*a*	0.534(4)	0.623(2)	-0.475(3)	0.040(8)
H(5A)	2*a*	0.2323(5)	0.6881(3)	-0.6183(4)	0.071(5)
H(6A)	2*a*	-0.0462(5)	0.6752(3)	-0.6301(4)	0.071
H(7A)	2*a*	-0.1480(5)	0.5885(3)	-0.4763(5)	0.071
H(8A)	2*a*	0.0248(4)	0.5084(3)	-0.3148(4)	0.071
H(11A)	2*a*	0.5623(4)	0.4501(2)	0.1743(3)	0.058(2)
H(11B)	2*a*	0.6313(4)	0.3655(2)	0.1932(3)	0.058
H(12A)	2*a*	0.6380(4)	0.4748(3)	0.4166(3)	0.058
H(12B)	2*a*	0.7884(4)	0.4458(3)	0.3578(3)	0.058
H(13A)	2*a*	0.7665(4)	0.3822(3)	0.5745(4)	0.058
H(13B)	2*a*	0.7633(4)	0.3211(3)	0.4510(4)	0.058
H(14A)	2*a*	0.4885(4)	0.3769(3)	0.5564(3)	0.058
H(14B)	2*a*	0.5527(4)	0.2913(3)	0.5722(3)	0.058
H(15A)	2*a*	0.4784(4)	0.2699(2)	0.3291(3)	0.058
H(16A)	2*a*	0.2746(4)	0.2495(2)	0.4638(3)	0.058
H(17A)	2*a*	0.2291(4)	0.3813(2)	0.5169(3)	0.058
H(17B)	2*a*	0.0652(4)	0.3398(2)	0.4552(3)	0.058
H(18A)	2*a*	0.2928(4)	0.4779(2)	0.2036(3)	0.058
H(18B)	2*a*	0.3591(4)	0.4703(2)	0.3686(3)	0.058
H(19A)	2*a*	0.2210(4)	0.2075(3)	0.2228(3)	0.058
H(19B)	2*a*	0.0635(4)	0.2275(3)	0.2785(3)	0.058
H(20A)	2*a*	0.0847(4)	0.4583(3)	0.3351(3)	0.058
H(21A)	2*a*	-0.0624(4)	0.3590(3)	0.2084(3)	0.058
H(22A)	2*a*	0.0771(4)	0.4483(3)	0.0145(4)	0.058
H(22B)	2*a*	-0.0702(4)	0.4756(3)	0.0800(4)	0.058
H(23A)	2*a*	-0.1544(5)	0.4246(3)	-0.1557(4)	0.058
H(23B)	2*a*	-0.2305(5)	0.3777(3)	-0.0431(4)	0.058
H(24A)	2*a*	0.0389(5)	0.3279(3)	-0.1562(4)	0.058
H(24B)	2*a*	-0.1294(5)	0.2870(3)	-0.1872(4)	0.058
H(25A)	2*a*	-0.0978(5)	0.2484(3)	0.0524(4)	0.058
H(25B)	2*a*	0.0473(5)	0.2162(3)	-0.0115(4)	0.058
H(33)	2*a*	0.447(1)	0.2580(5)	-0.1986(6)	0.28(2)
H(34)	2*a*	0.279(1)	0.2197(5)	-0.1973(6)	0.28
H(35)	2*a*	0.5109(9)	0.1409(4)	-0.2592(6)	0.28
H(36)	2*a*	0.3774(9)	0.1020(4)	-0.1877(6)	0.28
H(37)	2*a*	0.6888(8)	0.1370(3)	-0.0632(5)	0.28
H(38)	2*a*	0.5746(8)	0.0745(3)	-0.0146(5)	0.28
H(39)	2*a*	0.5124(6)	0.1554(3)	0.1420(5)	0.28
H(40)	2*a*	0.6397(6)	0.2145(3)	0.1039(5)	0.28

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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}
N(1)	2*a*	0.3480(4)	0.405	–0.0976(3)	0.077(2)	0.058(2)	0.046(2)	0.006(2)	0.011(1)	0.015(2)
C(1)	2*a*	0.3729(4)	0.5113(2)	–0.2849(3)	0.059(2)	0.037(2)	0.037(2)	–0.001(2)	0.011(2)	0.003(1)
C(2)	2*a*	0.5130(4)	0.5417(3)	–0.3253(3)	0.052(2)	0.045(2)	0.042(2)	0.002(2)	0.004(2)	–0.004(2)
C(3)	2*a*	0.4720(4)	0.5982(3)	–0.4297(3)	0.054(2)	0.043(2)	0.045(2)	–0.009(2)	0.012(2)	0.001(2)
C(4)	2*a*	0.2988(4)	0.6054(2)	–0.4590(3)	0.054(2)	0.033(2)	0.044(2)	0.001(2)	0.005(2)	–0.002(1)
C(5)	2*a*	0.1915(5)	0.6513(3)	–0.5566(4)	0.070(3)	0.043(2)	0.057(2)	0.002(2)	0.001(2)	0.004(2)
C(6)	2*a*	0.0273(5)	0.6441(3)	–0.5623(4)	0.064(3)	0.051(2)	0.072(2)	0.016(2)	–0.008(2)	–0.002(2)
C(7)	2*a*	–0.0331(5)	0.5918(3)	–0.4714(5)	0.047(2)	0.055(2)	0.087(3)	0.005(2)	–0.001(2)	–0.020(2)
C(8)	2*a*	0.0684(4)	0.5446(3)	–0.3757(4)	0.054(2)	0.046(2)	0.066(2)	–0.010(2)	0.015(2)	–0.011(2)
C(9)	2*a*	0.2369(4)	0.5509(2)	–0.3676(3)	0.049(2)	0.032(2)	0.041(2)	–0.001(1)	0.009(2)	–0.006(1)
C(10)	2*a*	0.3627(4)	0.4527(2)	–0.1827(4)	0.058(2)	0.049(2)	0.041(2)	0.003(2)	0.009(2)	–0.003(2)
N(2)	2*a*	0.4137(3)	0.3764(2)	0.2558(2)	0.038(1)	0.032(1)	0.038(1)	–0.002(1)	0.009(1)	0.002(1)
N(3)	2*a*	0.1117(3)	0.3043(2)	0.1295(3)	0.048(2)	0.041(1)	0.041(1)	–0.006(1)	0.002(1)	–0.006(1)
C(11)	2*a*	0.5775(4)	0.4056(2)	0.2382(3)	0.044(2)	0.037(2)	0.049(2)	–0.003(1)	0.012(1)	–0.002(1)
C(12)	2*a*	0.6847(4)	0.4303(3)	0.3771(3)	0.042(2)	0.051(2)	0.054(2)	–0.005(2)	0.012(2)	–0.013(2)
C(13)	2*a*	0.7050(4)	0.3636(3)	0.4844(4)	0.042(2)	0.072(2)	0.048(2)	–0.003(2)	–0.002(2)	–0.004(2)
C(14)	2*a*	0.5392(4)	0.3363(3)	0.5097(3)	0.061(2)	0.051(2)	0.039(2)	0.009(2)	0.007(2)	0.001(2)
C(15)	2*a*	0.4293(4)	0.3142(2)	0.3669(3)	0.047(2)	0.038(2)	0.036(2)	0.003(1)	0.012(1)	0.000(1)
C(16)	2*a*	0.2603(4)	0.2874(2)	0.3870(3)	0.050(2)	0.043(2)	0.041(2)	–0.005(2)	0.012(2)	0.006(1)
C(17)	2*a*	0.1668(4)	0.3566(2)	0.4328(3)	0.044(2)	0.058(2)	0.042(2)	–0.001(2)	0.011(1)	–0.003(2)
C(18)	2*a*	0.3071(4)	0.4422(2)	0.2841(3)	0.044(2)	0.033(2)	0.047(2)	0.005(1)	0.006(1)	–0.004(1)
C(19)	2*a*	0.1595(4)	0.2497(3)	0.2532(3)	0.054(2)	0.046(2)	0.052(2)	–0.012(2)	0.015(2)	–0.001(2)
C(20)	2*a*	0.1420(4)	0.4137(3)	0.3072(3)	0.042(2)	0.045(2)	0.046(2)	0.003(2)	0.010(1)	–0.009(2)
C(21)	2*a*	0.0346(4)	0.3757(3)	0.1771(3)	0.039(2)	0.057(2)	0.044(2)	0.003(2)	0.005(1)	–0.003(2)
C(22)	2*a*	–0.0171(4)	0.4305(3)	0.0491(4)	0.054(2)	0.063(2)	0.053(2)	0.013(2)	0.002(2)	0.000(2)
C(23)	2*a*	–0.1306(5)	0.3902(3)	–0.0735(4)	0.056(2)	0.097(3)	0.056(2)	0.010(2)	–0.003(2)	0.001(2)
C(24)	2*a*	–0.0541(5)	0.3148(3)	–0.1153(4)	0.063(2)	0.087(3)	0.051(2)	–0.008(2)	–0.007(2)	–0.016(2)
C(25)	2*a*	–0.0029(5)	0.2632(3)	0.0157(4)	0.062(2)	0.062(2)	0.057(2)	–0.012(2)	0.001(2)	–0.018(2)
Li(1)	2*a*	0.3255(6)	0.3292(3)	0.0578(5)	0.057(3)	0.038(3)	0.034(3)	0.009(2)	0.007(2)	0.002(2)
O(1)	2*a*	0.4176(3)	0.2327(2)	–0.0039(2)	0.094(2)	0.055(1)	0.047(1)	0.021(1)	0.008(1)	–0.013(1)
C(26)	2*a*	0.392(1)	0.2183(5)	–0.1544(6)	0.215(7)	0.143(5)	0.064(3)	0.101(5)	–0.013(4)	–0.037(3)
C(27)	2*a*	0.4604(9)	0.1413(4)	–0.1758(6)	0.201(7)	0.113(4)	0.069(3)	0.060(5)	0.003(4)	–0.037(3)
C(28)	2*a*	0.5815(8)	0.1278(3)	–0.0457(5)	0.178(5)	0.080(3)	0.063(3)	0.050(3)	0.021(3)	–0.010(2)
C(29)	2*a*	0.5461(6)	0.1838(3)	0.0648(5)	0.115(4)	0.069(3)	0.071(3)	0.046(3)	0.018(3)	0.000(2)

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