Crystal structure of (benzylcyano-*N*)-(1,4,7,10-tetraoxocyclodecane-*O*,*O'*,*O''*,*O'''*)-lithium, $C_{16}H_{22}LiNO_4$

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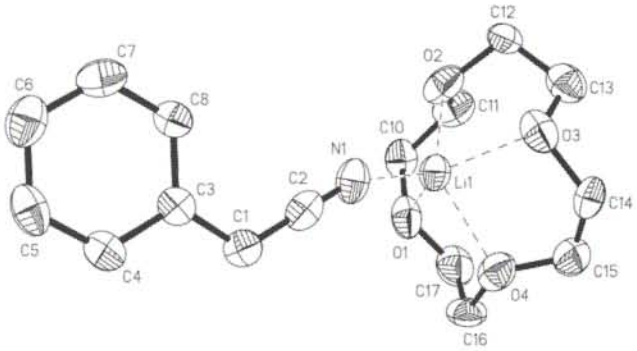
Received March 25, 1999, CCDC-No. 1267/151



Abstract

$C_{16}H_{22}LiNO_4$, orthorhombic, *Pbca* (No. 61), $a = 13.725(1)$ Å, $b = 13.261(1)$ Å, $c = 18.083(2)$ Å, $V = 3291.2$ Å^3, $Z = 8$, $R_{gt}(F) = 0.079$, $wR(F^2) = 0.212$, $T = 193$ K.

Source of material

The synthesis was done according to [1, 2].

Discussion

Besides earlier published structures of dimers of lithiated cyanides [1, 2], the structure of (benzylcyano-*N*)-12-crown-4-*O*,*O'*,*O''*,*O'''*)-lithium together with the structure (1-cyano-indenyl-*N*)-((–)spateine-*N*,*N'*)-(tetrafuran-*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:	light yellow prism, size 0.2 × 0.3 × 0.6 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	6.92 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω
$2\theta_{max}$:	110.22°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2055, 2055
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1779
$N(param)_{refined}$:	222
Programs:	SHELXL-93 [4], SHELXS-86 [5], XCAD4 [6], DIFABS [7]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*c*	0.606(3)	0.252(4)	0.293(2)	0.04(1)
H(2)	8*c*	0.471(3)	0.213(4)	0.217(3)	0.057(6)
H(3)	8*c*	0.366(4)	0.084(4)	0.184(3)	0.057
H(4)	8*c*	0.351(4)	–0.073(4)	0.227(3)	0.057
H(5)	8*c*	0.469(4)	–0.112(4)	0.315(3)	0.057
H(6)	8*c*	0.587(4)	–0.002(4)	0.355(3)	0.057
H(10A)	8*c*	0.6795(3)	0.2280(4)	0.6510(2)	0.070(4)
H(10B)	8*c*	0.7414(3)	0.3105(4)	0.6896(2)	0.070
H(11A)	8*c*	0.8810(4)	0.2182(4)	0.6689(3)	0.070
H(11B)	8*c*	0.8072(4)	0.1522(4)	0.7130(3)	0.070
H(12A)	8*c*	0.8855(3)	–0.0110(3)	0.5781(3)	0.070
H(12B)	8*c*	0.9171(3)	0.0274(3)	0.6558(3)	0.070
H(13A)	8*c*	1.0048(4)	0.1547(4)	0.6026(3)	0.070
H(13B)	8*c*	1.0398(4)	0.0537(4)	0.5678(3)	0.070
H(14A)	8*c*	1.0123(3)	0.1995(3)	0.4150(2)	0.070
H(14B)	8*c*	1.0854(3)	0.1911(3)	0.4805(2)	0.070
H(15A)	8*c*	1.0036(3)	0.3173(4)	0.5406(3)	0.070
H(15B)	8*c*	1.0352(3)	0.3570(4)	0.4630(3)	0.070
H(16A)	8*c*	0.7919(4)	0.4288(3)	0.4778(3)	0.070
H(16B)	8*c*	0.8936(4)	0.4688(3)	0.5018(3)	0.070
H(17A)	8*c*	0.8818(4)	0.3737(4)	0.6086(3)	0.070
H(17B)	8*c*	0.7955(4)	0.4499(4)	0.6043(3)	0.070

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)	8*c*	0.7223(3)	0.1600(3)	0.4280(2)	0.040(2)	0.059(2)	0.045(2)	–0.007(2)	–0.005(2)	–0.013(2)
C(1)	8*c*	0.6075(3)	0.1864(3)	0.3187(2)	0.045(2)	0.040(3)	0.032(2)	–0.002(2)	0.000(2)	–0.002(2)
C(2)	8*c*	0.6706(3)	0.1710(3)	0.3773(2)	0.042(2)	0.040(2)	0.042(3)	–0.007(2)	0.011(2)	–0.012(2)
C(3)	8*c*	0.5412(3)	0.1119(3)	0.2921(2)	0.037(2)	0.037(2)	0.027(2)	0.005(2)	0.005(2)	–0.007(2)
C(4)	8*c*	0.4712(3)	0.1369(3)	0.2379(2)	0.047(2)	0.038(2)	0.034(2)	0.011(2)	0.002(2)	–0.005(2)
C(5)	8*c*	0.4024(3)	0.0686(4)	0.2144(3)	0.043(3)	0.062(3)	0.039(3)	0.013(2)	–0.009(2)	–0.012(2)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6)	8*c*	0.3990(3)	–0.0280(4)	0.2424(3)	0.048(3)	0.056(3)	0.055(3)	–0.008(2)	0.000(2)	–0.016(3)
C(7)	8*c*	0.4684(4)	–0.0546(3)	0.2943(3)	0.069(3)	0.034(2)	0.051(3)	–0.002(2)	0.007(2)	–0.002(2)
C(8)	8*c*	0.5382(3)	0.0118(3)	0.3187(2)	0.043(2)	0.036(2)	0.036(2)	0.004(2)	–0.001(2)	0.000(2)
Li(1)	8*c*	0.8147(5)	0.1949(5)	0.5059(4)	0.034(3)	0.044(4)	0.039(4)	–0.004(3)	–0.004(3)	–0.006(3)
O(1)	8*c*	0.7596(2)	0.3107(2)	0.5778(2)	0.038(2)	0.056(2)	0.067(2)	0.005(1)	–0.009(1)	–0.022(2)
O(2)	8*c*	0.8150(2)	0.1182(2)	0.6085(2)	0.065(2)	0.059(2)	0.049(2)	–0.026(2)	–0.005(2)	0.001(2)
O(3)	8*c*	0.9533(2)	0.1311(2)	0.5033(2)	0.050(2)	0.051(2)	0.053(2)	0.011(2)	–0.011(1)	–0.014(2)
O(4)	8*c*	0.8974(2)	0.3210(2)	0.4698(2)	0.053(2)	0.061(2)	0.044(2)	–0.017(2)	–0.010(1)	0.006(2)
C(10)	8*c*	0.7418(3)	0.2607(4)	0.6513(2)	0.042(2)	0.068(3)	0.032(2)	0.002(2)	0.007(2)	–0.006(2)
C(11)	8*c*	0.8184(4)	0.1862(4)	0.6670(3)	0.074(3)	0.062(3)	0.050(3)	–0.009(3)	0.004(2)	0.002(2)
C(12)	8*c*	0.9013(3)	0.0478(3)	0.6063(3)	0.046(3)	0.035(2)	0.065(3)	0.003(2)	–0.013(2)	0.008(2)
C(13)	8*c*	0.9854(4)	0.0983(4)	0.5730(3)	0.059(3)	0.048(3)	0.063(3)	0.011(2)	–0.004(2)	–0.006(2)
C(14)	8*c*	1.0191(3)	0.2060(3)	0.4676(2)	0.034(2)	0.059(3)	0.048(3)	–0.007(2)	0.009(2)	–0.001(2)
C(15)	8*c*	0.9951(3)	0.3091(4)	0.4883(3)	0.050(3)	0.060(3)	0.056(3)	–0.005(2)	0.002(2)	0.011(2)
C(16)	8*c*	0.8502(4)	0.4125(3)	0.5046(3)	0.066(3)	0.028(2)	0.062(3)	–0.002(2)	–0.011(2)	0.004(2)
C(17)	8*c*	0.8246(4)	0.3924(4)	0.5812(3)	0.057(3)	0.047(3)	0.084(4)	0.009(2)	–0.006(3)	–0.015(3)

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