Refinement of the crystal structure of (9,10-triptyceno)-25-crown-7, $C_{34}H_{40}O_7$, *measured at 173 K*

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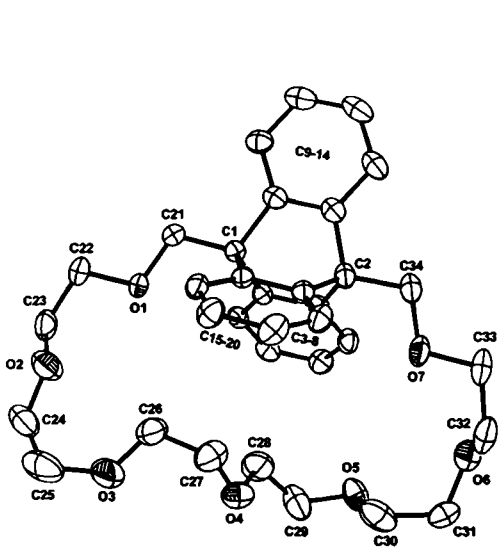
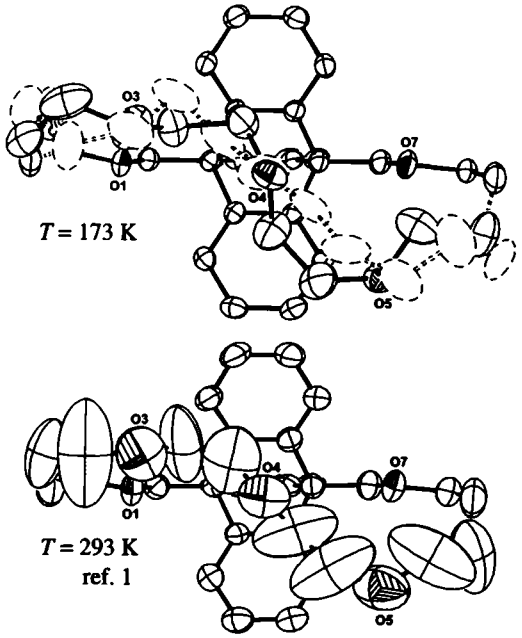


T = 173 K, major disorder component



T = 173 K

T = 293 K
ref. 1

Abstract

$C_{34}H_{40}O_7$, monoclinic, $P12_1/n1$(No. 14), a = 12.115(2) Å, b = 15.747(1) Å, c = 15.225(2) Å, β = 97.642(7)°, V = 2878.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.046, $wR_{ref}(F^2)$ = 0.126, T = 173 K.

Source of material

(9,10-Triptyceno)-25-crown-7 was prepared following established procedures [1]. Suitable crystals for X-ray analysis were obtained by slow evaporation from methanol.

Experimental details

Atoms between C23 and O6 on the crown ether ring are disordered, and are modeled over two sites (56:44). Chemically equivalent bond distances, angles between the two disorder components are restrained to be approximately equal. Additionally, the thermal parameters for all disordered atoms are restrained to be similar to atoms bound to it, or any atom within 0.7 Å.

Discussion

As part of our investigation of triptycene-substituted crown ether rings, we reported the room-temperature structure of (9,10-triptyceno)-25-crown-7 [1]. We noted that the thermal ellipsoids of some of the atoms in the polyether chain were unusually large, and some of the torsion angles in this part of the molecule were near zero, suggesting disorder. Attempts to model the putative disorder using the room-temperature data proved fruitless. More recently, we have been able to obtain low-temperature (173 K) data on another crystal of this compound, with the results reported here.

As can be seen in the figure, expansion and combination of the disordered atoms at 173 K would appear to approximate those observed at 293 K. Examination of the two disorder components shows that < 1 Å separates each disordered pair. Torsion angles within the disordered segment fall within ranges normally observed for crown ether rings. This structure provides an excellent example of the possible dangers interpreting room-temperature structural results without considering the thermal displacement parameters.

Table 1. Data collection and handling.

Crystal:	colourless thick plate, size 0.27 × 0.37 × 0.47 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.89 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10890, 5053
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3715
$N(param)_{refined}$:	478
Programs:	SHELXS-86 [2], SHELXL-97 [3], SHELXTL [4], PLATON [5]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(17)	4e		0.3287	0.7300	1.2829	0.038
H(5)	4e		-0.0447	0.7561	1.1995	0.039
H(6)	4e		-0.1704	0.8294	1.0960	0.043
H(7)	4e		-0.1384	0.8389	0.9491	0.039
H(8)	4e		0.0170	0.7728	0.9027	0.033
H(11)	4e		0.1119	0.4981	1.2242	0.045
H(12)	4e		0.1067	0.3742	1.1394	0.051
H(13)	4e		0.1306	0.3801	0.9910	0.048
H(14)	4e		0.1662	0.5101	0.9258	0.040
H(26B)	4e	0.56	0.2194	0.9220	0.9679	0.062
H(27A)	4e	0.56	0.2760	0.8997	1.1192	0.060
H(27B)	4e	0.56	0.3535	0.9821	1.1225	0.060
H(28A)	4e	0.56	0.0928	0.9100	1.1104	0.071
H(28B)	4e	0.56	0.0659	0.9731	1.0279	0.071
H(29A)	4e	0.56	0.0234	1.0734	1.1490	0.072
H(29B)	4e	0.56	-0.0697	1.0133	1.0975	0.072
H(30A)	4e	0.56	0.0885	1.0475	1.2932	0.073
H(30B)	4e	0.56	0.1305	0.9528	1.2783	0.073
H(31A)	4e	0.56	0.0897	0.9736	1.4245	0.073
H(31B)	4e	0.56	-0.0367	0.9944	1.3862	0.073
H(32A)	4e		0.0972	0.8317	1.4880	0.058
H(32B)	4e		0.1650	0.8603	1.4094	0.058
H(33A)	4e		0.1562	0.7074	1.4322	0.048
H(33B)	4e		0.0266	0.7133	1.3930	0.048
H(34A)	4e		0.0520	0.6325	1.2701	0.042
H(34B)	4e		0.1834	0.6225	1.2999	0.042
H(18)	4e		0.4986	0.7776	1.2446	0.044
H(19)	4e		0.5300	0.7789	1.0972	0.043
H(20)	4e		0.391	0.7336	0.9859	0.035

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(21A)	4e		0.2664	0.6392	0.9003	0.034
H(21B)	4e		0.1348	0.6454	0.8697	0.034
H(22A)	4e		0.2040	0.7246	0.7502	0.041
H(22B)	4e		0.3287	0.7351	0.7998	0.041
H(23A)	4e	0.56	0.2777	0.8506	0.7037	0.054
H(23B)	4e	0.56	0.1776	0.8725	0.7587	0.054
H(24A)	4e	0.56	0.2290	1.0015	0.7888	0.068
H(24B)	4e	0.56	0.3431	1.0018	0.7452	0.068
H(25A)	4e	0.56	0.4438	1.0115	0.8936	0.087
H(25B)	4e	0.56	0.3713	1.0947	0.8656	0.087
H(26A)	4e	0.56	0.3507	0.9076	0.9889	0.062
H(23C)	4e	0.44	0.2643	0.8518	0.7001	0.054
H(23D)	4e	0.44	0.1835	0.8781	0.7715	0.054
H(24C)	4e	0.44	0.3473	0.9938	0.7497	0.072
H(24D)	4e	0.44	0.4417	0.9903	0.8335	0.072
H(25C)	4e	0.44	0.2957	1.0864	0.8401	0.077
H(25D)	4e	0.44	0.2158	1.0077	0.8458	0.077
H(26C)	4e	0.44	0.3974	1.0085	1.0634	0.090
H(26D)	4e	0.44	0.3906	0.9348	0.9904	0.090
H(27C)	4e	0.44	0.2015	0.9095	1.0118	0.081
H(27D)	4e	0.44	0.2860	0.8920	1.1001	0.081
H(28C)	4e	0.44	0.1588	0.9279	1.2032	0.065
H(28D)	4e	0.44	0.0703	0.9225	1.1149	0.065
H(29C)	4e	0.44	0.1062	1.0740	1.2171	0.073
H(29D)	4e	0.44	0.0097	1.0598	1.1361	0.073
H(30C)	4e	0.44	0.0394	1.0700	1.3537	0.094
H(30D)	4e	0.44	0.1153	0.9901	1.3349	0.094
H(31C)	4e	0.44	-0.1007	0.9695	1.3740	0.082
H(31D)	4e	0.44	0.0024	0.9765	1.4511	0.082

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4e		0.2118(1)	0.75679(7)	0.87724(7)	0.0404(7)	0.0316(7)	0.0240(6)	-0.0004(5)	0.0123(5)	0.0000(5)
O(2)	4e		0.325(1)	0.8996(5)	0.8207(9)	0.069(4)	0.040(3)	0.058(5)	-0.008(3)	0.009(4)	0.010(3)
O(3)	4e		0.308(1)	1.0217(7)	0.9529(6)	0.079(5)	0.037(3)	0.089(5)	0.005(3)	0.036(4)	0.004(3)
O(4)	4e		0.1925(7)	1.0080(6)	1.1137(6)	0.040(3)	0.037(4)	0.046(3)	0.002(2)	-0.006(2)	-0.005(2)
O(5)	4e		-0.0189(4)	0.9768(3)	1.2206(3)	0.034(2)	0.054(3)	0.051(2)	-0.006(2)	0.008(2)	0.015(2)
O(6)	4e		0.0017(1)	0.87095(9)	1.38025(9)	0.058(1)	0.0496(9)	0.0446(8)	0.0025(7)	0.0180(7)	0.0017(7)
O(7)	4e		0.1290(1)	0.74083(9)	1.30491(8)	0.0439(8)	0.0490(8)	0.0263(7)	0.0024(6)	0.0152(6)	0.0035(6)
C(1)	4e		0.1785(1)	0.6723(1)	1.0031(1)	0.0259(9)	0.0239(8)	0.0260(9)	-0.0003(7)	0.0055(7)	0.0007(7)
C(2)	4e		0.1454(1)	0.6656(1)	1.1707(1)	0.0286(9)	0.032(1)	0.0262(9)	0.0027(7)	0.0073(7)	0.0055(7)
C(3)	4e		0.0767(1)	0.7233(1)	1.0230(1)	0.0260(9)	0.0225(8)	0.0278(9)	-0.0033(7)	0.0064(7)	0.0006(7)
C(4)	4e		0.0587(1)	0.7190(1)	1.1120(1)	0.0243(8)	0.0268(9)	0.0286(9)	-0.0020(7)	0.0047(7)	0.0029(7)
C(5)	4e		-0.0326(1)	0.7588(1)	1.1391(1)	0.030(1)	0.038(1)	0.0321(9)	0.0015(8)	0.0116(8)	0.0031(8)
C(6)	4e		-0.1068(2)	0.8029(1)	1.0778(1)	0.0264(9)	0.038(1)	0.044(1)	0.0052(8)	0.0097(8)	0.0005(8)
C(7)	4e		-0.0880(1)	0.8081(1)	0.9905(1)	0.0264(9)	0.030(1)	0.039(1)	0.0012(7)	-0.0004(8)	0.0064(8)
C(8)	4e		0.0041(1)	0.7686(1)	0.9628(1)	0.0295(9)	0.0260(9)	0.0269(9)	-0.0038(7)	0.0027(7)	0.0026(7)
C(9)	4e		0.1582(1)	0.5819(1)	1.0360(1)	0.0207(8)	0.0261(9)	0.037(1)	0.0017(7)	0.0034(7)	0.0032(7)
C(10)	4e		0.1416(1)	0.5786(1)	1.1251(1)	0.0220(8)	0.0303(9)	0.037(1)	0.0022(7)	0.0049(7)	0.0064(7)
C(11)	4e		0.1226(2)	0.5009(1)	1.1636(1)	0.031(1)	0.035(1)	0.047(1)	0.0014(8)	0.0083(8)	0.0149(9)
C(12)	4e		0.1193(2)	0.4274(1)	1.1130(2)	0.033(1)	0.028(1)	0.067(1)	0.0006(8)	0.007(1)	0.015(1)
C(13)	4e		0.1343(2)	0.4308(1)	1.0252(1)	0.031(1)	0.026(1)	0.061(1)	0.0007(8)	0.0031(9)	-0.0011(9)
C(14)	4e		0.1547(1)	0.5079(1)	0.9863(1)	0.0274(9)	0.030(1)	0.042(1)	0.0022(8)	0.0029(8)	-0.0016(8)
C(15)	4e		0.2772(1)	0.7051(1)	1.0679(1)	0.0262(9)	0.0230(8)	0.0252(8)	0.0031(7)	0.0049(7)	0.0005(7)
C(16)	4e		0.2589(1)	0.7029(1)	1.1568(1)	0.0260(9)	0.0254(9)	0.0268(9)	0.0055(7)	0.0057(7)	0.0035(7)
C(17)	4e		0.3412(2)	0.7305(1)	1.2227(1)	0.034(1)	0.039(1)	0.0233(9)	0.0056(8)	0.0034(8)	-0.0006(7)
C(18)	4e		0.4419(2)	0.7588(1)	1.1997(1)	0.0283(9)	0.046(1)	0.033(1)	0.0010(8)	-0.0012(8)	-0.0074(8)
C(19)	4e		0.4605(2)	0.7598(1)	1.1121(1)	0.0266(9)	0.043(1)	0.038(1)	-0.0032(8)	0.0081(8)	-0.0062(8)
C(20)	4e		0.3781(1)	0.7329(1)	1.0461(1)	0.0301(9)	0.0316(9)	0.0267(9)	-0.0008(7)	0.0091(7)	-0.0025(7)
C(21)	4e		0.1986(2)	0.6725(1)	0.9067(1)	0.0311(9)	0.0269(9)	0.0276(9)	0.0008(7)	0.0055(7)	-0.0029(7)
C(22)	4e		0.2525(2)	0.7591(1)	0.7940(1)	0.035(1)	0.045(1)	0.0231(9)	-0.0003(9)	0.0094(8)	-0.0006(8)
C(23)	4e	0.56	0.2539(2)	0.8490(1)	0.7634(1)	0.055(1)	0.051(1)	0.031(1)	0.007(1)	0.0154(9)	0.0090(9)
C(24)	4e	0.56	0.3092(5)	0.9872(4)	0.7989(5)	0.054(4)	0.041(3)	0.080(4)	0.000(3)	0.021(3)	0.019(3)
C(25)	4e	0.56	0.3669(5)	1.0334(3)	0.8790(4)	0.046(3)	0.046(3)	0.126(5)	-0.014(3)	0.018(3)	-0.003(3)
C(26)	4e	0.56	0.2897(5)	0.9477(3)	0.9965(3)	0.073(4)	0.039(2)	0.042(3)	0.010(2)	0.010(2)	-0.002(2)
C(27)	4e	0.56	0.2830(5)	0.9569(4)	1.0935(3)	0.055(3)	0.050(3)	0.044(3)	0.011(3)	0.001(3)	-0.005(2)

Table 3. Continued

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(28)	4*e*	0.56	0.0881(4)	0.9706(3)	1.0928(4)	0.056(3)	0.055(3)	0.068(3)	–0.021(2)	0.017(2)	–0.015(2)
C(29)	4*e*	0.56	0.0012(4)	1.0134(3)	1.1382(3)	0.057(3)	0.058(3)	0.068(3)	0.001(2)	0.020(2)	0.023(2)
C(30)	4*e*	0.56	0.0654(4)	0.9871(3)	1.2896(4)	0.057(3)	0.047(3)	0.073(4)	–0.017(2)	–0.010(3)	0.010(3)
C(31)	4*e*	0.56	0.0292(6)	0.9605(4)	1.3761(4)	0.088(5)	0.041(3)	0.059(3)	0.004(3)	0.031(3)	–0.016(2)
C(32)	4*e*		0.0977(2)	0.8302(2)	1.4230(1)	0.044(1)	0.075(2)	0.028(1)	–0.014(1)	0.0115(9)	–0.006(1)
C(33)	4*e*		0.1004(2)	0.7401(1)	1.3925(1)	0.031(1)	0.067(1)	0.0229(9)	0.0063(9)	0.0079(8)	0.0079(9)
C(34)	4*e*		0.1255(2)	0.6588(1)	1.2668(1)	0.035(1)	0.043(1)	0.030(1)	0.0033(8)	0.0098(8)	0.0095(8)
C(24A)	4*e*	0.44	0.3627(7)	0.9769(5)	0.8127(6)	0.077(6)	0.041(4)	0.068(5)	0.002(4)	0.032(5)	0.009(3)
C(25A)	4*e*	0.44	0.2929(7)	1.0279(4)	0.8634(4)	0.102(6)	0.040(3)	0.055(4)	0.016(4)	0.032(4)	0.014(3)
C(26A)	4*e*	0.44	0.3468(6)	0.9787(5)	1.0170(5)	0.065(4)	0.079(5)	0.070(4)	0.034(4)	–0.026(3)	–0.023(4)
C(27A)	4*e*	0.44	0.2544(7)	0.9367(5)	1.0585(6)	0.085(5)	0.043(4)	0.065(6)	0.030(3)	–0.030(4)	–0.014(3)
C(28A)	4*e*	0.44	0.1201(5)	0.9611(3)	1.1531(4)	0.065(3)	0.031(3)	0.055(3)	–0.004(2)	–0.032(3)	–0.005(2)
C(29A)	4*e*	0.44	0.0541(5)	1.0314(3)	1.1870(5)	0.036(3)	0.043(3)	0.099(5)	–0.006(2)	–0.012(3)	0.004(3)
C(30A)	4*e*	0.44	0.0377(8)	1.0102(5)	1.3335(5)	0.121(7)	0.046(4)	0.065(4)	–0.015(4)	–0.002(4)	–0.018(4)
C(31A)	4*e*	0.44	–0.0197(7)	0.9593(5)	1.3887(6)	0.062(5)	0.082(5)	0.068(4)	–0.006(4)	0.034(4)	–0.045(3)
O(4A)	4*e*	0.44	0.197(1)	0.9977(9)	1.105(1)	0.054(5)	0.027(3)	0.115(8)	0.002(3)	0.001(4)	–0.012(4)
O(2A)	4*e*	0.44	0.348(1)	0.8874(6)	0.819(1)	0.086(7)	0.029(3)	0.045(4)	–0.010(3)	0.033(4)	–0.004(3)
O(3A)	4*e*	0.44	0.305(1)	1.0357(8)	0.9528(6)	0.068(5)	0.040(4)	0.070(4)	0.017(3)	–0.014(4)	–0.018(3)
C(23A)	4*e*	0.44	0.2539(2)	0.8490(1)	0.7634(1)	0.055(1)	0.051(1)	0.031(1)	0.007(1)	0.0154(9)	0.0090(9)
O(5A)	4*e*	0.44	–0.0194(7)	1.0038(4)	1.2475(5)	0.089(4)	0.052(4)	0.068(4)	0.010(3)	–0.015(3)	0.003(3)
O(6A)	4*e*	0.44	0.0017(1)	0.87095(9)	1.38025(9)	0.058(1)	0.0496(9)	0.0446(8)	0.0025(7)	0.0180(7)	0.0017(7)

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