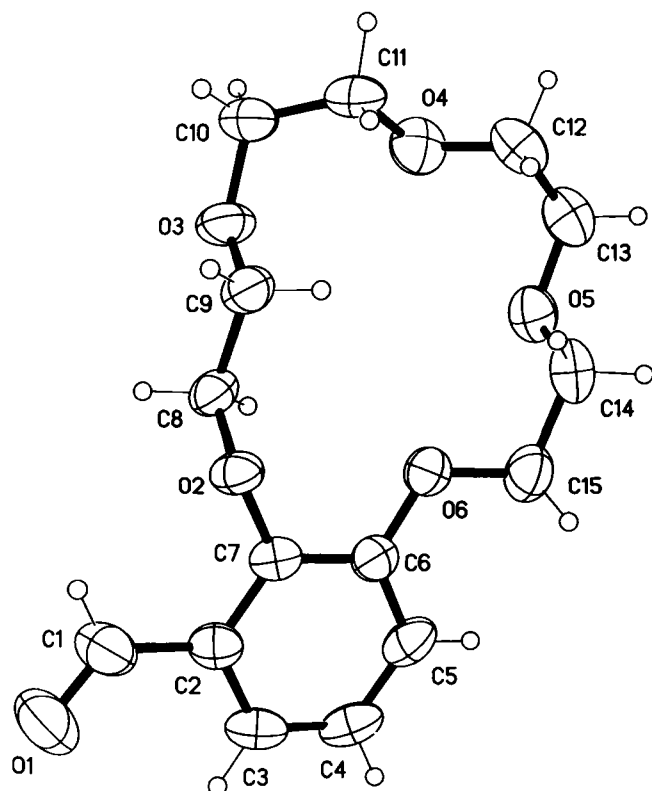


Crystal structure of 3'-formyl-benzo-15-crown-5, C₁₅H₂₀O₆

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their novel coordination modes, crown ethers have been widely used in catalyst, solvent extraction, isotope separation, bionics, material chemistry, host-guest chemistry and supramolecular chemistry [2–3].

In the crystal structure of the title compound the average C_{sp2}—O bond length is 1.374(5) Å, shorter than a normal C—O distance of 1.44 Å [4]. Meanwhile, the average bond length of C_{sp3}—O is 1.424 Å, slightly longer than that of 3,3'-dimethyl-dibenzo-18-crown-6 (1.411 Å) [5]. Both lengths and angles within the benzo-15-crown-5 system agree with the values reported in the literature [6]. The distances of non-bonded O2...O3, O3...O4 and O4...O5 are 2.700(3) Å, 2.845(2) Å and 2.864(1) Å, respectively, indicating that all the oxygen atoms lose their original symmetry because of the substituted benzene.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.08 × 0.25 × 0.28 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.00 cm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX CCD, φ/ω
$2\theta_{\max}$:	50.02°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	7148, 1513
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 1286
$N(\text{param})_{\text{refined}}$:	190
Program:	SHELX-97 [7]

Abstract

C₁₅H₂₀O₆, orthorhombic, $P2_12_12_1$ (no. 19), $a = 7.5235(9)$ Å, $b = 9.459(1)$ Å, $c = 21.3518(2)$ Å, $V = 1519.5$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.076$, $wR_{\text{ref}}(F^2) = 0.142$, $T = 298$ K.

Source of material

The title compound was prepared from the reaction of 1,11-dichloro-3,6,9-trioxaundecane (18.0 mmol) with 2,3-dihydroxybenzaldehyde (18.1 mmol) and NaOH (37.75 mmol) in water/n-butanol. After recrystallization from hot n-heptane and activated charcoal, the separated solid was obtained. Single crystals suitable for X-ray data collection were obtained by slow evaporation of an ethanol solution (yield 21.6 %).

Elemental analysis – found: C, 60.06 %; H, 6.68 %; calc. for C₁₅H₂₀O₆: C, 60.80 %; H, 6.80 %. ¹H NMR data are available in the CIF.

Discussion

The crown ether compounds have attracted much attention since the report about unusual coordination numbers and arrangements to metal ions of crown ether compounds by Pedersen [1]. Due to

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U _{iso}
H(1)	4a	−0.2286	0.8312	1.0830	0.072
H(3)	4a	0.0023	0.6901	1.2134	0.063
H(4)	4a	0.2046	0.5071	1.2134	0.068
H(5)	4a	0.2845	0.3975	1.1209	0.062
H(8A)	4a	−0.3181	0.6165	0.9898	0.053
H(8B)	4a	−0.1901	0.4871	0.9999	0.053
H(9A)	4a	−0.1855	0.6626	0.8921	0.059
H(9B)	4a	−0.0519	0.5362	0.9025	0.059
H(10A)	4a	−0.3427	0.5381	0.8033	0.074
H(10B)	4a	−0.3861	0.3781	0.8139	0.074
H(11A)	4a	−0.1401	0.3906	0.7512	0.066
H(11B)	4a	−0.0430	0.4911	0.7988	0.066
H(12A)	4a	0.1921	0.3525	0.8142	0.077
H(12B)	4a	0.1334	0.2266	0.7709	0.077
H(13A)	4a	0.1312	0.0744	0.8569	0.081
H(13B)	4a	0.3206	0.1407	0.8484	0.081
H(14A)	4a	0.3234	0.3902	0.9089	0.069
H(14B)	4a	0.4441	0.2642	0.9313	0.069
H(15A)	4a	0.2849	0.2778	1.0306	0.070
H(15B)	4a	0.4195	0.4018	1.0178	0.070

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
O(1)	4a	−0.2308(5)	0.8620(5)	1.1678(2)	0.111(3)	0.088(3)	0.064(2)	0.032(3)	0.010(2)	−0.021(2)
O(2)	4a	−0.0684(4)	0.6733(3)	1.0081(1)	0.055(2)	0.037(2)	0.036(2)	0.002(2)	−0.011(1)	0.002(1)
O(3)	4a	−0.3006(4)	0.4745(4)	0.8896(1)	0.047(2)	0.088(3)	0.041(2)	−0.012(2)	−0.007(1)	−0.009(2)
O(4)	4a	−0.0563(4)	0.2934(4)	0.8271(1)	0.057(2)	0.060(2)	0.059(2)	−0.007(2)	0.010(2)	0.008(2)
O(5)	4a	0.1926(4)	0.2132(4)	0.9222(1)	0.050(2)	0.061(2)	0.063(2)	0.000(2)	0.008(2)	−0.001(2)
O(6)	4a	0.1670(4)	0.4578(3)	1.0067(1)	0.054(2)	0.053(2)	0.048(2)	0.018(2)	−0.009(2)	−0.003(2)
C(1)	4a	−0.1800(7)	0.8031(6)	1.1210(2)	0.080(4)	0.053(3)	0.047(3)	0.006(3)	0.001(3)	−0.008(3)
C(2)	4a	−0.0458(6)	0.6897(5)	1.1202(2)	0.048(3)	0.035(3)	0.044(2)	−0.010(2)	−0.003(2)	−0.001(2)
C(3)	4a	0.0323(7)	0.6459(6)	1.1760(2)	0.069(3)	0.057(3)	0.032(2)	−0.011(3)	−0.006(2)	−0.003(2)
C(4)	4a	0.1543(7)	0.5368(6)	1.1759(2)	0.065(3)	0.062(4)	0.044(3)	−0.010(3)	−0.024(2)	0.010(3)
C(5)	4a	0.2025(6)	0.4710(5)	1.1204(2)	0.051(3)	0.048(3)	0.056(3)	0.002(3)	−0.020(2)	0.008(3)
C(6)	4a	0.1280(6)	0.5151(5)	1.0639(2)	0.041(2)	0.043(3)	0.042(2)	0.001(2)	−0.004(2)	−0.001(2)
C(7)	4a	0.0016(5)	0.6250(4)	1.0639(2)	0.043(2)	0.033(2)	0.035(2)	−0.010(2)	−0.004(2)	0.005(2)
C(8)	4a	−0.1998(6)	0.5803(5)	0.9813(2)	0.042(3)	0.052(3)	0.040(2)	0.003(2)	−0.007(2)	0.006(2)
C(9)	4a	−0.1705(6)	0.5707(6)	0.9116(2)	0.044(3)	0.063(3)	0.042(2)	0.000(3)	−0.006(2)	0.001(2)
C(10)	4a	−0.3012(7)	0.4522(7)	0.8234(2)	0.062(3)	0.086(5)	0.039(3)	0.002(3)	−0.008(2)	−0.010(3)
C(11)	4a	−0.1252(7)	0.4125(6)	0.7953(2)	0.069(4)	0.066(4)	0.031(2)	0.000(3)	−0.007(2)	0.005(2)
C(12)	4a	0.1243(7)	0.2653(6)	0.8128(2)	0.071(3)	0.071(4)	0.051(3)	0.001(3)	0.020(3)	−0.004(3)
C(13)	4a	0.1984(7)	0.1618(7)	0.8593(2)	0.069(4)	0.065(4)	0.068(3)	0.011(3)	0.011(3)	−0.011(3)
C(14)	4a	0.3295(6)	0.3094(6)	0.9369(2)	0.046(3)	0.051(3)	0.074(3)	0.009(3)	0.013(3)	0.001(3)
C(15)	4a	0.3101(7)	0.3580(6)	1.0037(2)	0.050(3)	0.055(3)	0.070(3)	0.011(3)	−0.007(3)	0.000(3)

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References

- Pedersen, C. J.: Cyclic polyethers and their complexes with metal salts. *J. Am. Chem. Soc.* **89** (1967) 7017–7022.
- Komia, N.; Naota, T.; Murahashi, S. I.: Aerobic oxidation of alkanes in the presence of acetaldehyde catalysed by copper-crown ether. *Tetrahedron Lett.* **37** (1996) 1633–1639.
- Clark, D. L.; Keogh, D. W.; Palmer, P. D.; Scott, B. L.: Synthesis and Structure of the First Transuranium Crown Ether Inclusion Complex: [NpO₂([18]Crown-6)]Cl. *Angew. Chem., Int. Ed.* **37** (1998) 164–169.
- Ma, J.; Zhu, H. J.; Wei, C. M.; Song, G. L.; Wang, J. T.: Synthesis and Crystal Structure of 5-(3,4-Dimethoxyphenyl)-1-phenyl-3-hydroxy-pyrazole. *Chin. J. Struct. Chem.* **23** (2004) 1312–1315.
- Niu, M. J.; Dou, J. M.; Wang, D. Q.; Li, D. C.: Synthesis and Crystal Structure of 3,3'-Dimethyl-dibenzo-18-crown-6. *Chin. J. Struct. Chem.* **25** (2006) 757–760.
- Wei, T. B.; Zhou, Y. Q.; Zhang, Y. M.; Zong, G. Q.: 4'-Acetylbenzo-15-crown-5 2-naphthyloxyacetylhydrazone. *Acta Crystallogr. E* **60** (2004) o678–o680.
- Sheldrick, G. M.: SHELX-97. Programs for Crystal Structure Analysis (Release 97-2). University of Göttingen, Germany 1997.