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Carbonyl Oxygen Coordinated to Potassium in a Monomeric and a Dimeric Crown Ether Complex: The Crystal Structure of [18]⟨O_5-(2,2')Benzophenono·2_4-coronand-5⟩*·KNCS

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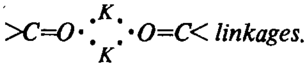
Colourless crystals of the title compound, M_r = *469.61, are monoclinic,* $P2_1/c$, a = *21.631(9),* b = *9.170(4),* c = *23.746(9)* Å, β = *93.48(5)°,* U = *4701.5* Å^3, D_c = *1.327 Mg* m^{-3} *for* Z = *8. The structure was solved by direct methods and refined to* R = *0.058 for 4850 diffractometer data.*

There are two independent ion pairs per asymmetric unit. In complex A *the cation is additionally coordinated to the five ether O atoms (mean distance 2.92* Å*) of the macrocycle, to the carbonyl O at 3.049(2)* Å *and to a symmetry related second* >C=O *at 2.846(2)* Å, *thus giving rise to a dimer* via



$>C{=}O\cdot\overset{K}{\underset{K}{:\,:}}\cdot O{=}C<$ *linkages.*

The monomeric complex B *is characterised by a relatively short* $>C{=}O\cdot\cdot K^+$ *contact of 2.587(2)* Å *and three strong (mean distance 2.73* Å*) and two weak (mean distance 3.14* Å*)* $K^+\cdot\cdot O$*(ether) interactions.*

Introduction

One reason for the interest in crown ethers and their complexes is their model character for compounds of biological relevance, *e.g.* for ionophore antibiotics. However, most of the latter also contain >C=O groups which participate in the coordination of K^+ [*e.g.* 2]. Syntheses and cation-binding properties of a number of crowns with >O partially or fully substituted by >C=O have been described [3–5], but the X-ray structure of a polyether/potassium complex involving $>C{=}O\cdot\cdot K^+$ interactions is known only for an open-chain ligand with terminal -COOH groups [6]. In 2,6-diketo-18-crown-6·KNCS [7], contacts between the cation and the carbonyl oxygens are not observed, possibly because of steric requirements.

The present macrocycle contains a benzophenone entity; it therefore appeared worthwhile to investigate the structure of the KNCS complex and, in particular, the coordination behaviour of the keto group.

Experimental

The compound was prepared by F.-A. von Itter and F. Vögtle, University of D-5300 Bonn, from whom a sample is gratefully acknowledged.

Colourless platelets were grown from ethyl acetate/methanol 9:1. Preliminary Weissenberg photographs revealed monoclinic symmetry with systematic absences OkO: k = 2n + 1, and hOl: l = 2n + 1. Cell dimensions were obtained from least-squares of the settings for 44 strong reflections with $20 < 2\theta < 25°$.

Crystal Data

$C_{21}H_{24}O_6$·KNCS, M_r = 469.61, space group $P2_1/c$, a = 21.631(9), b = 9.170(4), c = 23.746(9) Å, β = 93.48(5)°, U = 4701.5 Å^3, D_c = 1.327 Mg m^{-3} for Z = 8, $F(000)$ = 1968; μ(MoKα) = 0.343 mm^{-1}.

8248 unique profile-fitted [8] data were collected from a crystal *ca.* 0.35 X 0.25 X 0.05 mm, up to 2θ = 50° with monochromated MoKα radiation (λ = 0.71069 Å); three standards monitored every 100 reflections showed no significant change in intensity. Data were corrected for Lorentz and polarisation effects.

The structure was solved by multisolution direct methods and subsequent Fourier syntheses [9] and refined anisotropically on F by blocked-cascade least-squares using 4850 intensities > $2\sigma(I)$. H atoms could then be located in a difference map; they were included in idealised positions (C–H = 0.96 Å) and

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*Nomenclature according to [1].

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TABLE I. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Thermal Parameters ($Å^2 \times 10^3$) Defined as one Third of the Trace of the Orthogonalised U_{ij} Matrix.

A					B				
Atom	x	y	z	U	Atom	x	y	z	U
K	5301(1)	9797(1)	1043(1)	55(1)	K(1)	−736(1)	355(1)	3601(1)	62(1)
S	3022(1)	8538(1)	3239(1)	107(1)	S(1)	−2179(1)	6530(1)	2790(1)	80(1)
C	3656(2)	7846(4)	3503(2)	65(1)	C(1)	−1741(2)	7322(4)	3280(2)	67(1)
N	4122(2)	7376(4)	3677(2)	112(2)	N(1)	−1421(2)	7879(4)	3622(2)	99(2)
O(1)	6360(1)	8130(3)	1018(1)	64(1)	O(51)	−1787(1)	1914(2)	3629(1)	71(1)
C(2)	6327(1)	6995(4)	618(2)	67(2)	C(52)	−1813(2)	3027(4)	4041(2)	73(2)
C(3)	5812(1)	5956(4)	710(2)	58(1)	C(53)	−1321(1)	4143(4)	4013(2)	60(1)
O(4)	5239(1)	6736(2)	600(1)	53(1)	O(54)	−738(1)	3425(2)	4117(1)	56(1)
C(5)	4696(1)	6017(3)	653(1)	44(1)	C(55)	−220(1)	4242(3)	4226(1)	47(1)
C(6)	4146(1)	6800(3)	527(1)	43(1)	C(56)	334(1)	3484(3)	4364(1)	44(1)
C(7)	4136(1)	8254(3)	252(1)	46(1)	C(57)	354(1)	1876(4)	4452(1)	49(1)
C(8)	3568(1)	9193(3)	281(1)	51(1)	C(58)	963(1)	1102(4)	4477(1)	54(1)
C(9)	3463(1)	9945(3)	772(2)	56(1)	C(59)	1140(2)	365(4)	3998(2)	68(1)
O(10)	3897(1)	9722(3)	1212(1)	67(1)	O(60)	741(1)	495(3)	3534(1)	80(1)
C(11)	3749(2)	10182(5)	1764(2)	91(2)	C(61)	900(2)	−269(5)	3035(2)	105(2)
C(12)	4165(2)	9610(6)	2192(2)	110(2)	C(62)	486(2)	197(6)	2558(2)	113(2)
O(13)	4780(1)	9986(3)	2122(1)	96(1)	O(63)	−138(1)	−184(3)	2628(1)	87(1)
C(14)	5168(2)	9628(6)	2607(2)	122(2)	C(64)	−510(2)	109(6)	2124(2)	112(2)
C(15)	5819(2)	9844(5)	2493(2)	112(2)	C(65)	−1177(2)	−28(5)	2222(2)	98(2)
O(16)	5977(1)	8820(3)	2073(1)	85(1)	O(66)	−1338(1)	1096(3)	2598(1)	82(1)
C(17)	6604(2)	8907(4)	1952(2)	83(2)	C(67)	−1983(2)	1082(4)	2694(2)	89(2)
C(18)	6724(2)	7773(4)	1522(2)	78(2)	C(68)	−2103(2)	2268(4)	3106(2)	89(2)
C(19)	4668(2)	4590(3)	837(1)	56(1)	C(69)	880(2)	4296(4)	4438(1)	59(1)
C(20)	4103(2)	3966(4)	913(2)	68(1)	C(70)	882(2)	5783(4)	4400(2)	65(1)
C(21)	3561(2)	4723(4)	820(2)	72(2)	C(71)	334(2)	6509(4)	4276(1)	65(1)
C(22)	3587(2)	6143(4)	628(1)	58(1)	C(72)	−213(2)	5753(4)	4181(1)	57(1)
C(23)	3165(1)	9379(4)	−188(2)	66(1)	C(73)	1332(2)	1014(4)	4968(2)	71(1)
C(24)	2662(2)	10308(4)	−163(2)	80(2)	C(74)	1874(2)	219(4)	4994(2)	95(2)
C(25)	2569(2)	11048(4)	324(2)	79(2)	C(75)	2043(2)	−485(4)	4523(2)	103(2)
C(26)	2966(1)	10880(4)	796(2)	68(1)	C(76)	1685(2)	−433(4)	4028(2)	92(2)
O(27)	4560(1)	8713(3)	−7(1)	71(1)	O(77)	−109(1)	1147(2)	4512(1)	64(1)

TABLE II. Bond Lengths (Å).

A		B	
Atoms	Distance	Atoms	Distance
O(1)–C(2)	1.409(5)	O(51)–C(52)	1.417(4)
O(1)–C(18)	1.429(4)	O(51)–C(68)	1.420(5)
C(2)–C(3)	1.493(5)	C(52)–C(53)	1.480(5)
C(3)–O(4)	1.440(4)	C(53)–O(54)	1.430(4)
O(4)–C(5)	1.360(3)	O(54)–C(55)	1.360(4)
C(5)–C(6)	1.406(4)	C(55)–C(56)	1.406(4)
C(5)–C(19)	1.382(4)	C(55)–C(72)	1.389(5)
C(6)–C(7)	1.485(4)	C(56)–C(57)	1.490(5)
C(6)–C(22)	1.386(4)	C(56)–C(69)	1.398(4)
C(7)–C(8)	1.505(4)	C(57)–C(58)	1.495(4)
C(7)–O(27)	1.212(4)	C(57)–O(77)	1.219(4)
C(8)–C(9)	1.385(5)	C(58)–C(59)	1.396(5)
C(8)–C(23)	1.383(5)	C(58)–C(73)	1.375(5)
C(9)–O(10)	1.378(4)	C(59)–O(60)	1.363(4)
C(9)–C(26)	1.378(4)	C(59)–C(76)	1.386(5)
O(10)–C(11)	1.432(5)	O(60)–C(61)	1.435(5)
C(11)–C(12)	1.418(6)	C(61)–C(62)	1.465(6)
C(12)–O(13)	1.394(5)	C(62)–O(63)	1.413(6)
O(13)–C(14)	1.421(5)	O(63)–C(64)	1.427(5)
C(14)–C(15)	1.463(6)	C(64)–C(65)	1.481(7)
C(15)–O(16)	1.427(5)	C(65)–O(66)	1.421(5)
O(16)–C(17)	1.406(5)	O(66)–C(67)	1.428(5)
C(17)–C(18)	1.492(6)	C(67)–C(68)	1.495(6)
C(19)–C(20)	1.372(5)	C(69)–C(70)	1.367(5)
C(20)–C(21)	1.370(5)	C(70)–C(71)	1.376(5)
C(21)–C(22)	1.382(5)	C(71)–C(72)	1.378(5)
C(23)–C(24)	1.386(5)	C(73)–C(74)	1.378(5)
C(24)–C(25)	1.365(6)	C(74)–C(75)	1.361(7)
C(25)–C(26)	1.379(5)	C(75)–C(76)	1.367(7)
S–C	1.603(4)	S(1)–C(1)	1.626(4)
C–N	1.151(5)	C(1)–N(1)	1.153(5)

TABLE III. Bond Angles (°).

A		B	
Atoms	Angle	Atoms	Angle
C(2)–O(1)–C(18)	113.5(3)	C(52)–O(51)–C(68)	113.7(3)
O(1)–C(2)–C(3)	112.3(3)	O(51)–C(52)–C(53)	114.1(3)
C(2)–C(3)–O(4)	107.3(3)	C(52)–C(53)–O(54)	107.6(3)
C(3)–O(4)–C(5)	118.7(2)	C(53)–O(54)–C(55)	119.2(2)
O(4)–C(5)–C(6)	117.2(3)	O(54)–C(55)–C(56)	116.9(3)
O(4)–C(5)–C(19)	122.9(3)	O(54)–C(55)–C(72)	123.2(3)
C(6)–C(5)–C(19)	119.9(3)	C(56)–C(55)–C(72)	119.8(3)
C(5)–C(6)–C(7)	122.9(3)	C(55)–C(56)–C(57)	122.6(3)
C(5)–C(6)–C(22)	118.5(3)	C(55)–C(56)–C(69)	117.9(3)
C(7)–C(6)–C(22)	118.5(3)	C(57)–C(56)–C(69)	119.5(3)
C(6)–C(7)–C(8)	119.0(3)	C(56)–C(57)–C(58)	119.6(3)
C(6)–C(7)–O(27)	123.0(3)	C(56)–C(57)–O(77)	122.8(3)
C(8)–C(7)–O(27)	118.1(3)	C(58)–C(57)–O(77)	117.6(3)
C(7)–C(8)–C(9)	120.0(3)	C(57)–C(58)–C(59)	118.8(3)
C(7)–C(8)–C(23)	120.7(3)	C(57)–C(58)–C(73)	121.8(3)
C(9)–C(8)–C(23)	119.2(3)	C(59)–C(58)–C(73)	119.3(3)
C(8)–C(9)–O(10)	115.1(3)	C(58)–C(59)–O(60)	115.2(3)
C(8)–C(9)–C(26)	121.0(3)	C(58)–C(59)–C(76)	119.2(3)
O(10)–C(9)–C(26)	123.9(3)	O(60)–C(59)–C(76)	125.6(4)
C(9)–O(10)–C(11)	118.1(3)	C(59)–O(60)–C(61)	116.9(3)
O(10)–C(11)–C(12)	112.4(4)	O(60)–C(61)–C(62)	109.3(4)
C(11)–C(12)–O(13)	113.0(4)	C(61)–C(62)–O(63)	112.3(4)
C(12)–O(13)–C(14)	111.5(3)	C(62)–O(63)–C(64)	110.4(3)
O(13)–C(14)–C(15)	110.3(4)	O(63)–C(64)–C(65)	110.9(4)
C(14)–C(15)–O(16)	108.3(4)	C(64)–C(65)–O(66)	108.5(4)
C(15)–O(16)–C(17)	112.4(3)	C(65)–O(66)–C(67)	112.0(3)
O(16)–C(17)–C(18)	108.1(3)	O(66)–C(67)–C(68)	107.9(3)
O(1)–C(18)–C(17)	107.6(3)	O(51)–C(68)–C(67)	108.2(3)
C(5)–C(19)–C(20)	119.6(3)	C(56)–C(69)–C(70)	122.0(3)
C(19)–C(20)–C(21)	121.9(3)	C(69)–C(70)–C(71)	119.4(3)
C(20)–C(21)–C(22)	118.7(3)	C(70)–C(71)–C(72)	120.7(3)
C(6)–C(22)–C(21)	121.3(3)	C(55)–C(72)–C(71)	120.2(3)
C(8)–C(23)–C(24)	120.0(3)	C(58)–C(73)–C(74)	121.1(4)
C(23)–C(24)–C(25)	119.9(3)	C(73)–C(74)–C(75)	119.0(4)
C(24)–C(25)–C(26)	121.2(3)	C(74)–C(75)–C(76)	121.6(4)
C(9)–C(26)–C(25)	118.8(4)	C(59)–C(76)–C(75)	119.9(4)
S–C–N	177.5(4)	S(1)–C(1)–N(1)	178.7(4)

treated as 'riding atoms', with $U(H_i)$ kept fixed at $1.2\ U_{eq}(C_i)$ during further refinement. The highest residual electron density in the final difference map was 0.33 e^- per $Å^3$, close to S. Convergence was achieved at $R = \Sigma(|F_o - |F_c||)/\Sigma F_o = 0.058$ and $R_w = \Sigma(|F_o - |F_c||)\sqrt{w}/\Sigma F_o\sqrt{w} = 0.053$; the weighting scheme $w^{-1} = \sigma^2(F) + 3.5 \times 10^{-3} F^2$ gave a flat analysis of variances.

A cell reduction program [10] revealed neither a higher symmetry cell nor unusual extinctions indicative of a possible twinning; the presence of two independent molecules in $P2_1/c$ is thus confirmed.

Atom coordinates are given in Table I; chemically equivalent distances and angles in both complexes may be directly compared from Tables II and III (for numbering scheme see Fig. 1). Lists of anisotropic temperature factors, H atom parameters and structure factors are available from GW on request.

Results and Discussion

There are two crystallographically independent complex ion pairs, *A* and *B*, in the asymmetric unit. Corresponding bond lengths and angles show no unusual features and are (with few exceptions) identical within 1 e.s.d. (Table II). The somewhat shortened $>C=O$ distance (as compared to that in free benzophenone [11]) might be attributed to the alkoxy substituents, since in *p,p'*-dimethoxybenzophenone [12] a similarly short contact is found.

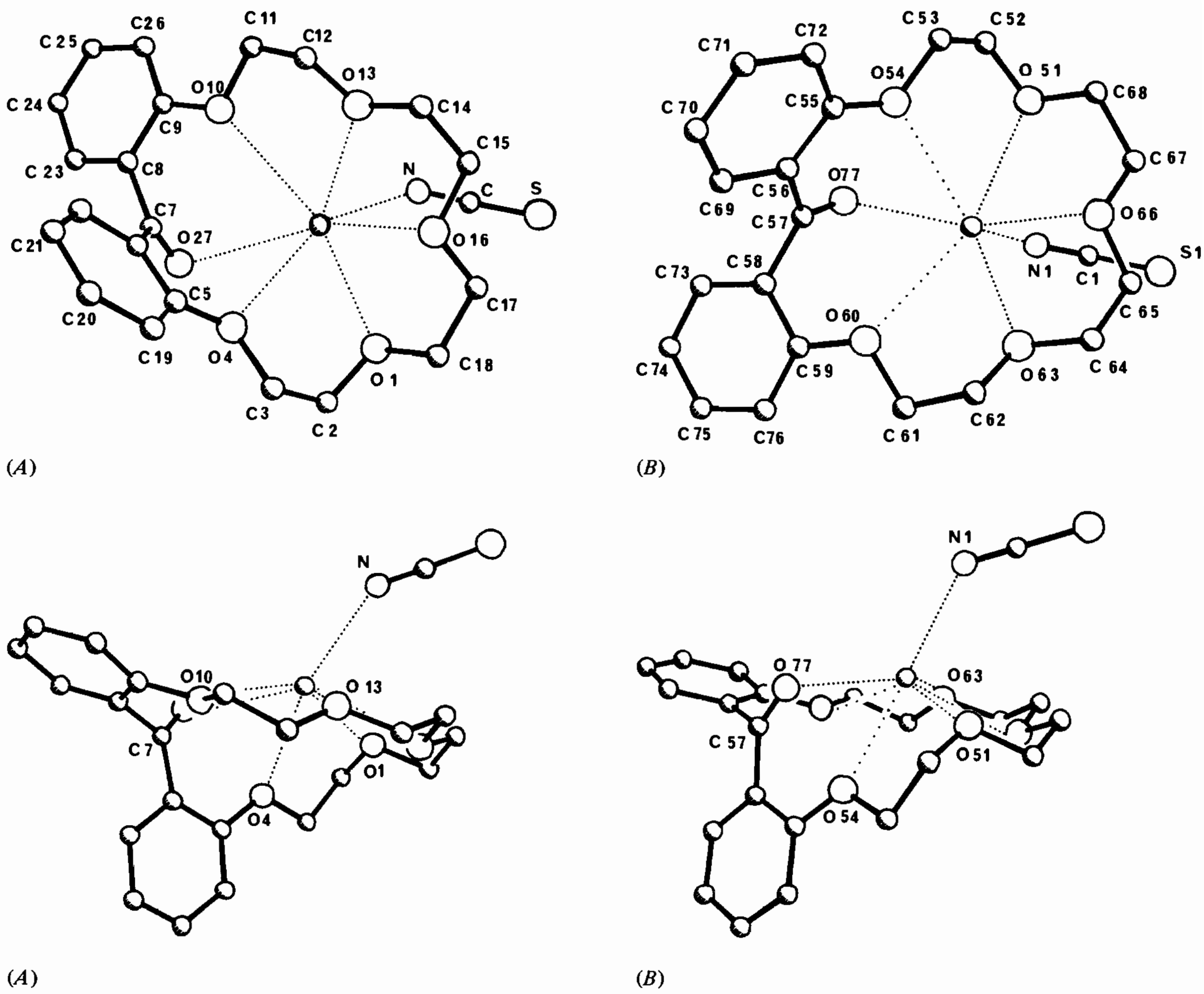

Fig. 1. Perspective views of the two independent complexes *A* and *B* including the numbering scheme. Radii are arbitrary, H atoms omitted for clarity.

TABLE IV. Endocyclic Torsion Angles (°).

A		B	
Atoms	Angle	Atoms	Angle
C(18)–O(1)–C(2)–C(3)	87.2(3)	C(68)–O(51)–C(52)–C(53)	–86.4(3)
C(2)–O(1)–C(18)–C(17)	–167.4(3)	C(52)–O(51)–C(68)–C(67)	174.0(3)
O(1)–C(2)–C(3)–O(4)	66.7(3)	O(51)–C(52)–C(53)–O(54)	–62.1(4)
C(2)–C(3)–O(4)–C(5)	179.7(3)	C(52)–C(53)–O(54)–C(55)	–167.0(3)
C(3)–O(4)–C(5)–C(6)	–178.3(3)	C(53)–O(54)–C(55)–C(56)	175.7(3)
O(4)–C(5)–C(6)–C(7)	10.6(4)	O(54)–C(55)–C(56)–C(57)	–6.6(4)
C(5)–C(6)–C(7)–C(8)	–162.7(3)	C(55)–C(56)–C(57)–C(58)	167.2(3)
C(5)–C(6)–C(7)–O(27)	18.8(5)	C(55)–C(56)–C(57)–O(77)	–13.7(5)
C(6)–C(7)–C(8)–C(9)	77.3(4)	C(56)–C(57)–C(58)–C(59)	–100.2(4)
O(27)–C(7)–C(8)–C(9)	–104.2(4)	O(77)–C(57)–C(58)–C(59)	80.6(4)
C(7)–C(8)–C(9)–O(10)	–1.8(4)	C(57)–C(58)–C(59)–O(60)	3.4(5)
C(8)–C(9)–O(10)–C(11)	–165.9(3)	C(58)–C(59)–O(60)–C(61)	–178.9(3)
C(9)–O(10)–C(11)–C(12)	166.8(3)	C(59)–O(60)–C(61)–C(62)	–170.0(3)

(continued on facing page)

TABLE IV. *(continued)*

A		B	
Atoms	Angle	Atoms	Angle
O(10)–C(11)–C(12)–O(13)	57.8(5)	O(60)–C(61)–C(62)–O(63)	–64.3(5)
C(11)–C(12)–O(13)–C(14)	169.4(4)	C(61)–C(62)–O(63)–C(64)	–172.1(4)
C(12)–O(13)–C(14)–C(15)	172.1(4)	C(62)–O(63)–C(64)–C(65)	–169.6(4)
O(13)–C(14)–C(15)–O(16)	–65.6(5)	O(63)–C(64)–C(65)–O(66)	66.3(5)
C(14)–C(15)–O(16)–C(17)	–177.6(3)	C(64)–C(65)–O(66)–C(67)	177.2(3)
C(15)–O(16)–C(17)–C(18)	178.5(3)	C(65)–O(66)–C(67)–C(68)	178.3(3)
O(16)–C(17)–C(18)–O(1)	63.8(4)	O(66)–C(67)–C(68)–O(51)	–65.3(4)

TABLE V. Coordination Distances (Å).

A		B	
Atoms	Distance	Atoms	Distance
K···O(1)	2.758(3)	K(1)···O(51)	2.690(3)
K···O(4)	2.999(2)	K(1)···O(54)	3.070(3)
K···O(10)	3.087(3)	K(1)···O(60)	3.211(3)
K···O(13)	2.865(3)	K(1)···O(63)	2.760(4)
K···O(16)	2.913(3)	K(1)···O(66)	2.730(3)
K···O(27)	3.049(2)	K(1)···O(77)	2.587(2)
K···O(27)[1]	2.846(2)		
K···N[2]	2.736(3)	K(1)···N(1)[3]	2.714(3)

[1] Generated by inverstion at 0.5, 1,5, 0. [2] Coordinates as given in Table I are to be transformed by 1 – x, 0.5 + y, 0.5 – z. [3] y-Coordinate as given in Table I to be shifted by –1.

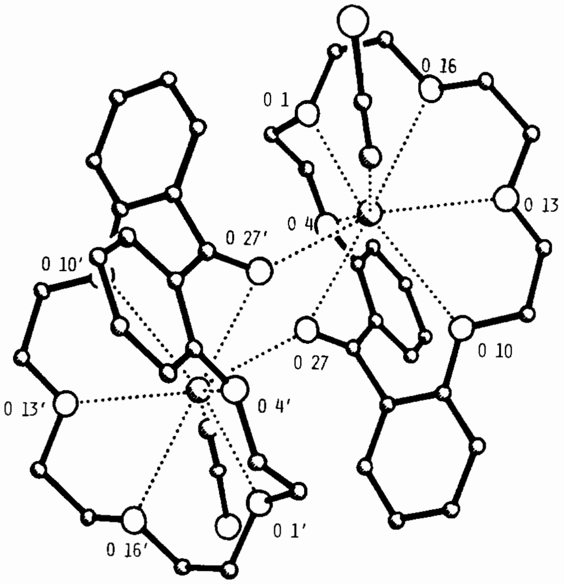

Fig. 2. The dimer *A*/*A*'. The second half (primed atoms) is related by a centre of inversion at 0.5, 1.5, 0.

The present macrocycle is the oxo-analogue of a previously reported [13] diphenyl-methane crown which forms crystalline complexes with Na^+ but not with K^+, possibly because of the sterically hindering effect of the CH_2 group on macrocycle coordination to the bigger cation. Substitution of this methylene group by a >C=O group, however, seems to provide a cavity for potassium uptake, with the carbonyl oxygens in positions suitable for dipole··cation interactions (see Fig. 1). Endocyclic torsion angles (Table IV) indicate the nearly identical, strain-free conformations adopted by the ligand in both complexes: Mean C–C–O–C = $|173(6)|^\circ$ in *A* and $|174(5)|^\circ$ in *B*, and mean O–C–C–O = $|163(3)|^\circ$ in *A* and $|164(2)|^\circ$ in *B* correspond to almost ideal values, *i.e.* anti and gauche, respectively [14]; torsions around O(1)–C(2), O(51)–C(52) of about 90° (neglected in the above estimation) seem to be a common feature with benzo-crowns [*e.g.* 15–17] and probably compensate for the syn-arrangement required at the aromatic nuclei. Mutually exchanged torsions about C(7)–C(8), C(57)–C(58), accompanied by differing dihedral angles between aromatic planes (87.7(5) in *A*, 71.5(5)° in *B*; *cf.* 56° in benzophenone [11]) are the most distinct differences between the two conformations.

However, these slight differences seem to affect the K^+ coordination appreciably. In complex *A*, the cation environment is probably best described as a distorted bipyramid, the base of which is an incomplete hexagon formed by O(1), O(27), O(10), O(13) and O(16) (co-planar to within ±0.15 Å); one apex is occupied by O(4), the other by $^-$NCS (and a symmetry-related O(27)'). The mean K^+··O distance of 2.93 Å (see Table V) exceeds the sum of respective radii by *ca.* 0.2 Å and is notably longer than normally found with higher-coordinated potassium (*cf.* 2.80 Å in 18-crown-6·KNCS [18] and 2.86 Å in (benzo-15-crown-5)$_2$·KI [17]). This lack of strong interactions might facilitate dimer formation (Fig. 2) resulting in a centrosymmetric K–(carbonyl)O–K–(carbonyl)O bridge, similar to that in [6] and comparable to some other known K–X–K–X geometries [*e.g.* 16, 19, 20].

In complex *B*, two groups of K$^+$··O(ether) contacts (Table V) can easily be distinguished: distances (mean 2.73 Å) of O(51), O(63) and O(66) (co-planar with O(77) to within ±0.12 Å) reflect the stronger binding forces of 'equatorially' coordinating atoms [21, 22]; weak interactions of O(54) (*ca.* 3.1 Å) and O(60) (*ca.* 3.2 Å) may be attributed to the 'catechol' properties of these oxygens [21, 23] and/or to their 'apical' positions [21, 22].

The extremely close contact of K(1)$^+$··O(77) = 2.587(2) Å is nearly 0.5 Å shorter than K$^+$··O(27) in *A* (thus possibly precluding dimerisation in *B*) and about 0.14 Å shorter than the sum of the respective radii. A greater difference (0.21 Å) was found only in a benzophenonedilithium compound [24] but this may not be strictly comparable because of the partially co-valent character of Li-bonds. Even with anionic ethyl acetoacetate, K$^+$ is linked at a distance 0.09 Å longer [25] than the present one. A correlation between the strength of the K$^+$··O interaction and the >C=O bond length as reported for N-methylacetamide complexes of alkali metals [26] is not obvious in *A* and *B*.

Most structures with K$^+$··O=C< units investigated so far involve amides [*e.g.* 26], esters [*e.g.* 2], carboxylic acids [*e.g.* 6, 27] or mesomeric anions [25, 27–29], and only a few ketones are known to ligate potassium. In K$^+$ complexes of isonitrosoacetophenone [30, 31], both, K$^+$··O=C< linkages of 2.8 to 3.0 Å and, in particular, the bifurcated coordination of >C=O groups [30], bear strong resemblance to interactions in the current complex *A*. However, the title compound seems the first example of a cyclic ketone (or better oxo-crown) coordinating to potassium.

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