

no systematic variation with ligand and are in normal ranges found in other phenyl isocyanide complexes (Bohling & Mann, 1984).

The C—N≡C—Cr—C≡N—C moieties show significant angular distortion. The *trans* ligands deviate in opposite directions producing an S-type distortion. The normal 90° C—Cr—C angles range from a high value of 92.0 (2) to a low value of 88.0 (2)°. The Cr—C—N angles, normally 180°, ranged from 176.8 (5) to 177.5 (5)°. The C≡N—C angles, normally 180°, ranged from 163.7 (5) to 168.0 (5)°. In the Cr(CNC₆H₅)₆ compound the *trans* C≡N—C bonds were distorted in the same direction, producing a bowed geometry, with Cr—C 1.938 (3) Å, C≡N 1.176 (4) Å, C—Cr—C 85.9 (2)°, C≡N—C 172.9 (3)° and Cr—C≡N 173.7 (2)° (Ljungström, 1978). This same bowed geometry was observed for the Cr^I, Cr^{II} and Cr^{III} Cr(CNC₆H₅)₆ complexes and the extent of the bowing was found to increase with the oxidation state (Bohling & Mann 1984).

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Complexes of Lithium and Sodium with *sym*-(Dibenzo-14-crown-4)methylphenyl Phosphinic Acid

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Abstract. (1) (6-Methylphenylphosphinato-2,3,9,10-dibenzo-1,4,8,11-tetraoxacyclotetradeca-2,9-diene)-lithium-ethanol (1/1), [Li(C₂₅H₂₆PO₆)]·C₂H₅OH, *M_r* = 506.46, triclinic, *P* $\bar{1}$, *a* = 10.158 (1), *b* = 10.663 (1), *c* = 12.766 (2) Å, α = 100.93 (1), β = 109.55 (1), γ = 99.10 (1)°, *V* = 1242.2 (6) Å³, *Z* = 2, *D_x* = 1.35 g cm⁻³, λ (Mo *K*α) = 0.71069 Å, μ = 1.49 cm⁻¹, *F*(000) = 536, *T* = 295 K, *R*(*F*) = 0.048 for 1712 reflections [*I* > σ (*I*)]. (2) (6-Methylphenylphosphinato-2,3,9,10-dibenzo-1,4,8,11-tetraoxacyclotetradeca-2,9-diene)diaquasodium trihydrate, [Na(C₂₅H₂₆PO₆)(H₂O)₂]·3H₂O, *M_r* = 566.52, triclinic, *P* $\bar{1}$, *a* = 10.493 (6), *b* = 11.770 (5), *c* = 13.162 (5) Å, α = 98.16 (3), β = 108.73 (4), γ = 108.81 (4)°, *V* = 1402 (3) Å³, *Z* = 2, *D_x* = 1.35, *D_m* = 1.34 g cm⁻³, λ (Mo *K*α) = 0.71069 Å, μ = 1.63 cm⁻¹, *F*(000) = 600, *T* = 296 K, *R*(*F*) = 0.058 for 2849 reflections [*I* > σ (*I*)]. In neither compound is there intramolecular bonding of the phosphinate anion to the cation enclosed in the crown. Compound (1) crystallizes as dimers in which the phosphinate group of one ligand is bonded to the Li⁺ ion enclosed in the ether cavity of its partner and *vice versa*. Molecules of compound

(2) are isolated from each other by channels of water molecules.

Introduction. The crown ethers with pendant sidearms containing functional groups (lariats) are primarily of two types: the oxa crowns in which the sidearm is attached to a C atom (C pivot) and the aza or oxa-aza crowns that are of the N-pivot type. In the latter the functionality is in a position axial (*A*) to the crown by virtue of the N-bonding geometry and usually coordinates any cation enclosed in the crown (Gandour, Fronczek, Gatto, Minganti, Schultz, White, Arnold, Mazzocchi, Miller & Gokel, 1986; Arnold, Echevoyen, Fronczek, Gandour, Gatto, White & Gokel, 1987). For C-pivot crowns the sidearm may be *A* or *E* (equatorial) (Echevoyen, Kaifer, Durst, Schultz, Dishong, Goli & Gokel, 1984). In one case, a substituted 18-crown-6 derived from tartaric acid, two carboxylate groups occupy *A* positions but they are induced to be there by bulky vicinal substituents (Behr, Lehn, Moras & Thierry, 1981).

Studies of lariat ethers based on dibenzo-14-crown-4 have found sidearms in the *A* position in two cases, but the bonding to the cation in the crown was through an oxy link in the sidearm; the functional groups at the ends of the arms were not bonded (Shoham, Christianson, Bartsch, Heo, Olsher & Lipscomb, 1984; Sachleben, Burns & Brown, 1988). Owing to the advantage in solvent extraction of having an extractable complex containing both cation and anion (Bartsch, Heo, Kang, Liu & Strzelbicki, 1982), we have synthesized a series of lariat ethers for testing and studied the structures of their metal complexes. Among these are several lariats without the oxy link in the sidearm, which better enable us to assess the role of the pivot atom as it affects intramolecular anion-cation bonding. Carboxylates were reported previously (Burns & Sachleben, 1990) and two phosphinates are described here.

Experimental. The ligand was prepared, as shown below, from the previously reported crown ether, *sym*-methyleno-dibenzo-14-crown-4 (3) (Burns & Sachleben, 1990). Hydroboration/oxidation of (3) gave the alcohol (4). It was converted to the bromide (5), which, when heated with diisopropoxy(phenyl)phosphine (prepared from dichlorophenylphosphine and 2-propanol, Hoffman & Moore, 1958), gave the crown ether phosphinic ester (6). Hydrolysis in refluxing HBr gave a viscous orange oil from which a white solid containing (7) as a major constituent was obtained by precipitation from isopropyl alcohol. (1) and (2) were prepared by addition of the appropriate aqueous hydroxide solutions to (7) in ethanol. Crystals were obtained by removal of the solvent from ethanol solutions of (1) and (2) through slow diffusion in a closed container into a beaker of glycerol.

Most of the crystal data and experimental details are summarized in the *Abstract* and in Table 1. Colorless thin plates of (1) and (2) were attached to glass fibers and studied by precession-camera photography before mounting on the diffractometer, where unit-cell dimensions and intensities were measured. The intensity data were corrected with Lorentz and polarization factors, calculated absorption factors based on measured crystal shape, and for variation of three reference reflections in each case. All calculations employed the Enraf-Nonius *Structure Determination Package* (Frenz, 1983). *MULTAN82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) was used to locate most of the atoms of the ligand molecule and the remainder were found by use of difference Fourier syntheses.

H atoms attached to C atoms were included in calculated positions with bond lengths of 0.95 Å. For H₂O and C₂H₅OH molecules the H atoms were positioned by inference from O...O distances, and the H₂O molecules were symmetrically aligned about the O...O...O angles. Only the H atom involved in hydrogen bonding was included for the ethanol. All H atoms were given temperature factors equal to the *B*_{eq} values of the atoms to which they are attached.

Full-matrix least-squares refinements of the structures, based on *F*, included anisotropic thermal parameters on all non-H atoms. Observations, *F*_o, were given weights, *w*, as $w = 4F_o^2 / \{[\sigma(I)]^2 + (0.05F_o^2)^2\}$, where *I* = scaled, observed intensity. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and anomalous-dispersion factors were from Cromer & Liberman (1970). Final Δ/σ values were ≤ 1.0 and final difference Fourier syntheses had $\rho_{\max} = 0.19$, $\rho_{\min} = -0.25 \text{ e } \text{Å}^{-3}$ for (1) and $\rho_{\max} = 0.43$, $\rho_{\min} = -0.41 \text{ e } \text{Å}^{-3}$ for (2). Atomic coordinates and

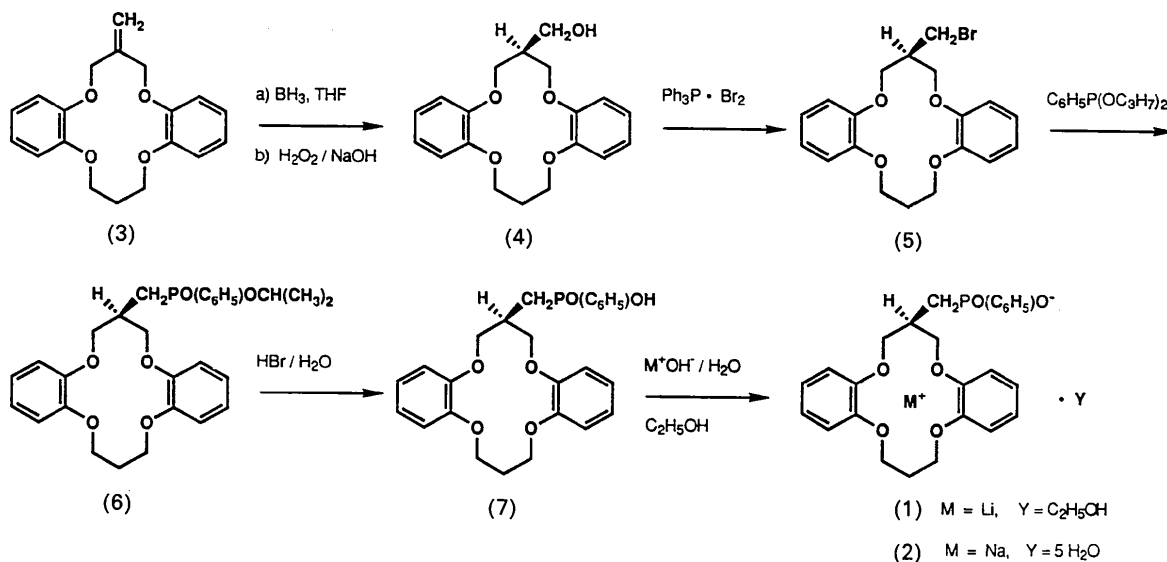


Table 1. Crystal data and experimental details

	(1)	(2)
Crystal dimensions (mm)	0.06 × 0.22 × 0.22	0.05 × 0.29 × 0.28
Density by flotation (g cm ⁻³)	—	1.34
Diffractionmeter	Enraf-Nonius CAD-4	
Radiation, λ(Å)	Mo Kα, 0.71069	
Scan type	ω/2θ	
Reflections for cell parameters	17	22
Transmission factors	0.970–0.992	0.962–0.992
Maximum sin θ/λ (Å ⁻¹)	0.481	0.538
Range of h, k, l	0, 9; -10, 10; -12, 11	0, 11; -12, 11; -14, 12
Reference reflections	031, 231, 425	792, 037, 711
with variation (%)	-3.6	-9.6
Number of reflections measured	2918	3854
R(F) internal	0.014	0.030
Number of unobserved reflections	1044	832
I < σ(I)		
Number of reflections used in refinement	1712	2848
Number of parameters refined	325	343
R	0.048	0.058
wR	0.057	0.062
S	1.638	1.606

Table 2. Coordinates and equivalent isotropic thermal parameters for (1)

$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	B _{eq} (Å ²)
Li	0.5505 (7)	0.3431 (7)	-0.2197 (5)	3.6 (2)
P	0.5673 (1)	0.4045 (1)	0.24139 (9)	3.32 (3)
O(1)	0.3956 (3)	0.1938 (3)	-0.3547 (2)	4.32 (8)
O(4)	0.4667 (3)	0.2178 (3)	-0.1407 (2)	3.35 (7)
O(5)	0.6500 (3)	0.4449 (3)	0.3686 (2)	4.58 (8)
O(6)	0.4921 (3)	0.4990 (3)	0.1859 (2)	4.14 (7)
O(7)	0.1122 (5)	0.3777 (6)	0.4958 (4)	14.6 (2)
O(8)	0.7493 (3)	0.3403 (3)	-0.1026 (2)	3.72 (7)
O(11)	0.6765 (3)	0.3161 (3)	-0.3178 (2)	4.53 (8)
C(2)	0.3083 (4)	0.1037 (4)	-0.3269 (3)	3.6 (1)
C(3)	0.3472 (4)	0.1170 (4)	-0.2091 (3)	3.3 (1)
C(5)	0.5182 (4)	0.2290 (4)	-0.0195 (3)	3.6 (1)
C(6)	0.6465 (4)	0.3457 (4)	0.0436 (3)	2.9 (1)
C(7)	0.7767 (4)	0.3315 (4)	0.0138 (3)	3.8 (1)
C(9)	0.8580 (4)	0.3266 (4)	-0.1436 (3)	3.5 (1)
C(10)	0.8166 (4)	0.3120 (4)	-0.2627 (3)	3.9 (1)
C(12)	0.6223 (5)	0.2895 (5)	-0.4426 (3)	5.5 (1)
C(13)	0.4685 (5)	0.2984 (5)	-0.4823 (2)	5.1 (1)
C(14)	0.3634 (5)	0.1858 (5)	-0.4757 (3)	5.5 (1)
C(15)	0.1931 (5)	0.0045 (4)	-0.4051 (4)	4.7 (1)
C(16)	0.1151 (5)	-0.0789 (5)	-0.3636 (4)	5.7 (2)
C(17)	0.1503 (5)	-0.0631 (5)	-0.2486 (4)	5.2 (1)
C(18)	0.2679 (4)	0.0350 (4)	-0.1683 (3)	4.0 (1)
C(19)	0.9976 (4)	0.3264 (4)	-0.0778 (4)	4.3 (1)
C(20)	1.0957 (4)	0.3111 (5)	-0.1299 (4)	5.3 (1)
C(21)	1.0545 (5)	0.2956 (5)	-0.2473 (4)	6.1 (1)
C(22)	0.9152 (5)	0.2975 (5)	-0.3137 (3)	5.0 (1)
C(23)	0.6903 (4)	0.3625 (4)	0.1740 (3)	3.4 (1)
C(24)	0.4311 (4)	0.2550 (4)	0.2107 (3)	3.1 (1)
C(25)	0.2871 (5)	0.2536 (5)	0.1620 (4)	5.0 (1)
C(26)	0.1808 (5)	0.1431 (6)	0.1447 (5)	7.1 (2)
C(27)	0.2187 (5)	0.0342 (5)	0.1757 (4)	6.4 (2)
C(28)	0.3601 (5)	0.0325 (4)	0.2221 (4)	5.4 (1)
C(29)	0.4664 (4)	0.1419 (4)	0.2395 (3)	4.2 (1)
C(30)	0.086 (2)	0.400 (1)	0.411 (1)	46.7 (7)
C(31)	0.079 (2)	0.379 (2)	0.317 (2)	35 (1)

equivalent isotropic thermal parameters are given in Tables 2 and 3. The principal bond lengths are listed in Table 4.*

* Tables of structure factors, anisotropic thermal parameters, H-atom parameters, and selected bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54260 (40 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Coordinates and equivalent isotropic thermal parameters for (2)

$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	B _{eq} (Å ²)
Na	0.5311 (1)	-0.2873 (1)	0.7934 (1)	3.12 (3)
P	0.93952 (9)	0.25727 (8)	0.84082 (7)	2.57 (2)
O(1)	0.4038 (2)	-0.3916 (2)	0.5920 (2)	2.88 (6)
O(2)	0.5368 (3)	0.3477 (3)	0.0638 (2)	4.59 (7)
O(3)	0.7476 (3)	-0.3136 (3)	0.8765 (2)	4.74 (7)
O(4)	0.6240 (2)	-0.1788 (2)	0.6712 (2)	3.13 (6)
O(5)	0.8281 (2)	0.3099 (2)	0.8470 (2)	3.36 (6)
O(6)	1.1002 (2)	0.3382 (2)	0.9090 (2)	3.42 (6)
O(8)	0.5133 (2)	-0.0835 (2)	0.8200 (2)	3.16 (6)
O(9)	1.1028 (3)	0.4511 (2)	1.1896 (2)	4.23 (7)
O(10)	0.8046 (3)	0.4016 (2)	0.0378 (2)	3.90 (7)
O(11)	0.2855 (2)	-0.2921 (2)	0.7368 (2)	2.95 (6)
O(12)	0.3547 (3)	0.3758 (3)	-0.1248 (2)	4.68 (8)
C(2)	0.4629 (3)	-0.3519 (3)	0.5170 (3)	2.52 (8)
C(3)	0.5810 (3)	-0.2353 (3)	0.5595 (3)	2.70 (8)
C(5)	0.7285 (4)	-0.0513 (3)	0.7110 (3)	3.21 (9)
C(6)	0.7598 (3)	0.0020 (3)	0.8323 (3)	2.73 (9)
C(7)	0.6375 (3)	0.0339 (3)	0.8504 (3)	3.08 (9)
C(9)	0.3872 (3)	-0.0777 (3)	0.8273 (3)	2.70 (8)
C(10)	0.2644 (3)	-0.1914 (3)	0.7841 (3)	2.59 (8)
C(12)	0.1578 (4)	-0.4058 (3)	0.6777 (3)	3.5 (1)
C(13)	0.2045 (4)	-0.5066 (3)	0.6367 (3)	3.5 (1)
C(14)	0.2607 (4)	-0.4917 (3)	0.5454 (3)	3.4 (1)
C(15)	0.4121 (4)	-0.4164 (3)	0.4074 (3)	3.28 (9)
C(16)	0.4764 (4)	-0.3668 (4)	0.3384 (3)	3.9 (1)
C(17)	0.5906 (4)	-0.2519 (4)	0.3792 (3)	4.3 (1)
C(18)	0.6436 (4)	-0.1857 (4)	0.4908 (3)	4.0 (1)
C(19)	0.3763 (4)	0.0310 (3)	0.8720 (3)	3.5 (1)
C(20)	0.2429 (4)	0.0257 (4)	0.8761 (3)	3.9 (1)
C(21)	0.1235 (4)	-0.0855 (4)	0.8347 (3)	3.8 (1)
C(22)	0.1333 (3)	-0.1947 (3)	0.7890 (3)	3.14 (9)
C(23)	0.9078 (3)	0.1171 (3)	0.8881 (3)	2.81 (9)
C(24)	0.9149 (3)	0.2141 (3)	0.6963 (3)	2.66 (8)
C(25)	0.7938 (4)	0.2131 (3)	0.6105 (3)	3.6 (1)
C(26)	0.7732 (4)	0.1735 (4)	0.5003 (3)	4.5 (1)
C(27)	0.8737 (4)	0.1373 (4)	0.4740 (3)	4.4 (1)
C(28)	0.9960 (4)	0.1408 (4)	0.5576 (3)	3.8 (1)
C(29)	1.0169 (4)	0.1780 (3)	0.6680 (3)	3.26 (9)

Table 4. Selected bond lengths (Å)

	M = Li	M = Na		M = Li	M = Na
M—O(1)	2.081 (7)	2.462 (2)	C(17)—C(18)	1.396 (5)	1.394 (5)
M—O(4)	2.063 (6)	2.441 (2)	C(18)—C(3)	1.385 (5)	1.369 (5)
M—O(8)	2.081 (7)	2.450 (2)	C(9)—C(19)	1.386 (5)	1.386 (4)
M—O(11)	2.084 (7)	2.421 (2)	C(19)—C(20)	1.384 (6)	1.399 (5)
M—O(2)		2.336 (3)	C(20)—C(21)	1.383 (6)	1.372 (5)
M—O(3)		2.325 (3)	C(21)—C(22)	1.391 (6)	1.388 (5)
M—O(6)	1.813 (7)		C(22)—C(10)	1.377 (5)	1.386 (4)
P—O(5)	1.495 (2)	1.507 (2)	C(6)—C(23)	1.538 (5)	1.554 (4)
P—O(6)	1.501 (3)	1.518 (2)	C(24)—C(25)	1.382 (5)	1.398 (5)
P—C(23)	1.803 (4)	1.814 (3)	C(25)—C(26)	1.393 (6)	1.382 (5)
P—C(24)	1.820 (4)	1.809 (3)	C(26)—C(27)	1.366 (7)	1.377 (5)
O(1)—C(2)	1.381 (4)	1.384 (4)	C(27)—C(28)	1.363 (6)	1.380 (5)
C(2)—C(3)	1.394 (5)	1.404 (4)	C(28)—C(29)	1.385 (6)	1.379 (5)
C(3)—O(4)	1.383 (4)	1.382 (3)	C(29)—C(24)	1.391 (5)	1.404 (4)
O(4)—C(5)	1.432 (4)	1.441 (4)	C(30)—C(31)	1.16 (4)	
C(5)—C(6)	1.515 (5)	1.515 (4)	O(7)—C(30)	1.10 (2)	
C(6)—C(7)	1.515 (5)	1.526 (3)	O(5)—O(7)	2.631 (5)	
C(7)—O(8)	1.441 (4)	1.455 (3)	O(2)—O(10)		2.819 (3)
O(8)—C(9)	1.387 (4)	1.379 (4)	O(2)—O(12)		2.741 (3)
C(9)—C(10)	1.405 (5)	1.406 (4)	O(3)—O(6)		2.871 (3)
C(10)—O(11)	1.372 (5)	1.375 (4)	O(3)—O(9)		2.824 (3)
O(11)—C(12)	1.455 (5)	1.439 (4)	O(9)—O(5 ⁱⁱ)		2.822 (3)
C(12)—C(13)	1.497 (6)	1.517 (5)	O(9)—O(10 ⁱⁱⁱ)		2.932 (3)
C(13)—C(14)	1.510 (6)	1.507 (5)	O(10)—O(6 ^{iv})		2.800 (3)
C(14)—O(1)	1.450 (4)	1.442 (4)	O(10)—O(5 ^v)		2.708 (3)
C(2)—C(15)	1.381 (5)	1.370 (4)	O(12)—O(6 ^{iv})		2.754 (3)
C(15)—C(16)	1.392 (6)	1.382 (5)	O(12)—O(2 ^{vi})		2.969 (4)
C(16)—C(17)	1.360 (6)	1.374 (5)			

Symmetry code: (i) 2 - x, -y, 2 - z; (ii) 2 - x, 1 - y, 2 - z; (iii) x, y, 1 + z; (iv) 2 - x, 1 - y, 1 - z; (v) x, y, z - 1; (vi) x - 1, y, z - 1; (vii) 1 - x, 1 - y, -z.

Discussion. The structure of (1) is shown in Fig. 1. It consists of centrosymmetric dimers which are formed by the bonding of the phosphinate anion of one

molecule to the Li⁺ cation of another; the anion of the second molecule is attached to the cation of the first in a reciprocal manner. Analogous dimers have been found to exist in the structure of Li-dibenzo-14-crown-4-acetate-ethanol (2/1) (Sachleben & Burns, 1991) and in Na-dibenzo-14-crown-4-acetate-methanol (1/1) hydrate (Burns & Sachleben, 1990). In these latter compounds the coordinating O atom is provided by the acetate anion.

The pendant arm attached at C(6) is in the *E* site; which, of course, makes it inaccessible for intramolecular bonding. The four ether O atoms are planar (± 0.002 Å) and the Li⁺ ion lies 0.87 Å from this plane. Two halves of the crown ether, including benzo groups, are planar (± 0.1 Å) and intersect along the C(6)—C(13) line at a dihedral angle of 127.4°. The single molecule of ethanol is hydrogen bonded to O(5) of the phosphinate group. The very large thermal parameters and unrealistic geometry found suggest disorder in this molecule, but maxima in the Fourier maps were not resolvable into two sites.

An asymmetric unit of the structure of (2) is shown in Fig. 2. The ligand configuration is similar to that in (1); the planar (± 0.2 Å) halves of the crown ether intersect at an angle of 126.5°. The Na⁺ ion is bonded to the four ether O atoms and is 1.49 Å away from their plane (± 0.025 Å), but makes neither intra- nor intermolecular bonds to an anion. As in (1), the anion is extended away from the center of the crown, because the pendant arm is attached at the *E* site of the ring. The six coordination of the Na⁺ ion is completed by two water molecules which, with three other water molecules and the phosphinate O atoms, engage in an extensive network of hydrogen bonding (Fig. 3) to produce hydrophilic channels along the *a* axis. Each complex molecule has its two inner-sphere H₂O molecules in

one channel and its phosphinate O atoms in another. The crown ether molecules are packed together between these channels and form hydrophobic regions.

Molecular-mechanics calculations on isolated Na complexes of dibenzo-14-crown-4 with various sidearms bearing carboxylate groups indicate that intramolecular cation-anion bonding should be favored, but such has not been found in the crystals studied so far (Burns & Sachleben, 1990). Nor does it occur in the present two phosphinate complexes. Thus, under the influence of neighbors, either solvent molecules or other complexes, the sidearm adopts the *E* site in all these crystals.

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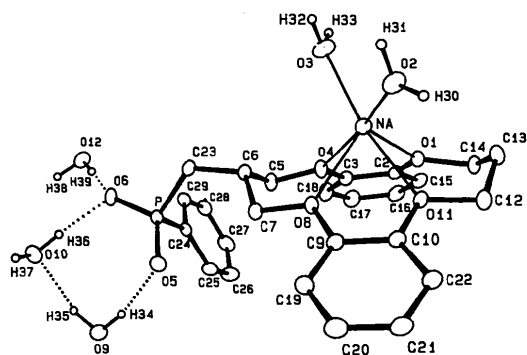


Fig. 2. One asymmetric unit of (2). Atoms are labeled as in Table 2 and are represented by 20% probability ellipsoids. H atoms are included only for H₂O molecules.

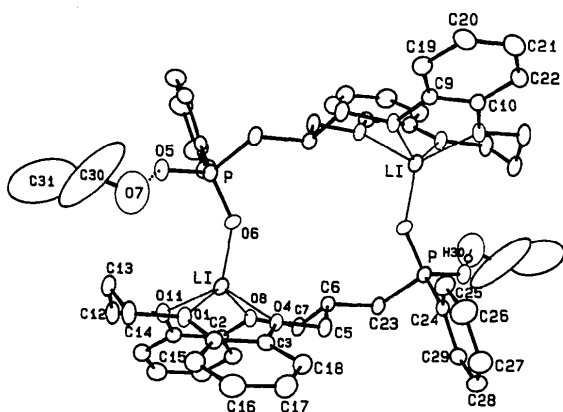


Fig. 1. The dimeric structure of (1). Atoms are labeled as in Table 1 and are represented by thermal ellipsoids of 20% probability. H atoms are omitted except for ethanol.

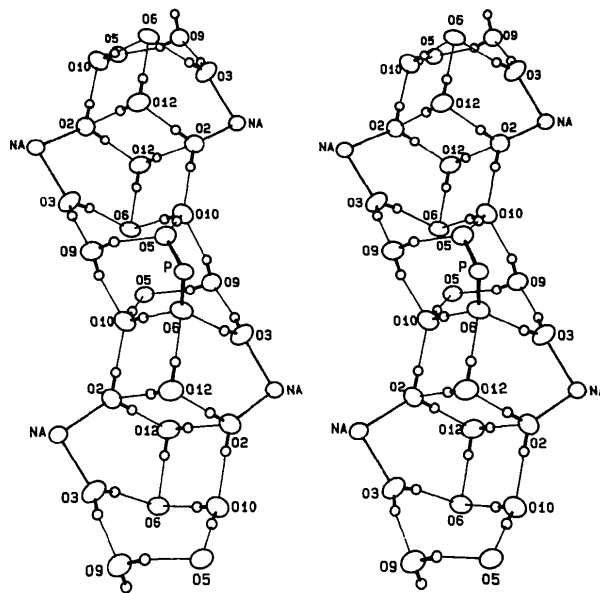


Fig. 3. A stereoscopic drawing of a water channel which separates and links the crown ethers in (2).

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Structure of $[(\mu_3\text{-CH})\text{CoMo}_2(\text{CO})_7(\eta^5\text{-C}_5\text{H}_5)]$; a Cobaltodimolybdenum Triangular Carbonyl Cluster Capped by an Alkylidyne Unit

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Abstract. Heptacarbonylbis(η^5 -cyclopentadienyl)- μ_3 -methylidyne-cobaltodimolybdenum (1) is one of the products isolated from the reaction of the cyclopentadienyltricarbonylmolybdenum anion, $(\eta^5\text{-C}_5\text{H}_5)\text{Mo}(\text{CO})_3^-$, and nonacarbonyl- μ_3 -chloromethylidyne-tricobalt, $(\mu_3\text{-CCl})\text{Co}_3(\text{CO})_9$. The crystal and molecular structure of (1) has been determined. The compound crystallizes in the monoclinic space group $P2_1/c$ with $M_r = 590.09$, $a = 10.007$ (2), $b = 12.886$ (2), $c = 16.536$ (4) Å, $\beta = 116.74$ (1)°, $V = 1908$ (1) Å³, $Z = 4$, $D_x = 2.05$ g cm⁻³. $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu(\text{Mo } K\alpha) = 21.58$ cm⁻¹, $F(000) = 1144$, $T = 295$ K, $R = 0.020$ for 2818 independent reflections with $I > 3\sigma(I)$. The structure consists of a cobaltodimolybdenum triangle capped by an alkylidyne unit and with the two cyclopentadienyl groups in a *transoid* arrangement.

Introduction. $(\mu_3\text{-CH})\text{CoMo}_2(\text{CO})_7(\eta^5\text{-C}_5\text{H}_5)_2$, (1), is one of a number of related alkylidyne clusters that can be obtained by exchange of the cobalt atoms in the metal triangle of $(\mu_3\text{-CR})\text{Co}_3(\text{CO})_9$ ($R = \text{H, Cl, Me, Ph etc.}$) by reaction with transition-metal car-

bonyl anions, or with transition-metal carbonyl dimers (Blumhofer, Fischer & Vahrenkamp, 1986; Duffy, Kassis & Rae, 1990; Duffy, Kassis & Rae, unpublished; Beurich & Vahrenkamp, 1982). This paper reports the structure of (1), which was synthesized *via* the reaction of $(\mu_3\text{-CCl})\text{Co}_3(\text{CO})_9$ with the metal-transfer reagent $(\eta^5\text{-C}_5\text{H}_5)\text{Mo}(\text{CO})_3^-$.

Experimental. $(\mu_3\text{-CH})\text{CoMo}_2(\text{CO})_7(\eta^5\text{-C}_5\text{H}_5)_2$ crystallizes as green/black crystals in the monoclinic space group $P2_1/c$. An irregularly shaped crystal of maximum dimensions $0.16 \times 0.14 \times 0.08$ mm was mounted on a quartz fibre and the lattice parameters ($T = 295$ K, 24 reflections, θ range $20\text{--}23^\circ$) were determined and refined with the least-squares routine on an Enraf-Nonius CAD-4 four-circle diffractometer equipped with a graphite monochromator.

A total of 3341 unique reflections (of 3728 collected) were collected in the range $1.5 < \theta < 25^\circ$ using an ω - 2θ scan mode ($R_{\text{int}} = 0.014$). The intensity of one standard reflection was measured after every 2000 s of X-ray exposure. No decomposition occurred during data collection. Lorentz, polarization and absorption corrections (Gaussian integration method, maximum and minimum transmission

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