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# Structure of Hydronium*–1,4,7,10,13,16-Hexaoxacyclooctadecane (18-Crown-6)–Hexamolybdate(VI) (2/2/1)

BY CLARA BRINK SHOEMAKER, LYLE V. MCAFEE, DAVID P. SHOEMAKER AND CARROLL W. DEKOCK

*Department of Chemistry, Oregon State University, Corvallis, Oregon 97331, USA*

**Abstract.** $[Mo_6O_{19}]^{2-}.(C_{12}H_{24}O_6.H_3O^+)_2$, $M_r = 1446.30$, monoclinic, $P2/m$, $a = 11.571$ (13), $b = 11.222$ (11), $c = 9.150$ (9) Å, $\beta = 101.98$ (8)°, $V = 1162$ (2) Å³, $Z = 1$, $D_m = 2.09$ (1), $D_x = 2.07$ g cm⁻³, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 16.28$ cm⁻¹, $F(000) = 714$, $T = 293$ (2) K, final $R = 0.069$ for 1265 observed reflections (47 left out because of twinning). The $[Mo_6O_{19}]^{2-}$ polyanion has the geometry previously reported: it consists of six distorted $MoO_6$ octahedra fused together, with the central O atom, which is at a center of symmetry, shared by all six. The average distances in Å are: Mo—Mo 3.285 (5), Mo—$O_t$ (*terminal*) 1.649 (7), Mo—$O_c$ (*central*) 2.323 (7), Mo—$O_b$ (*bridging*) 1.933 (38). The average angles in degrees are: Mo—$O_c$—Mo 90.0 (1) and 180.0, Mo—$O_b$—Mo 116.4 (1.0), $O_c$—Mo—$O_t$ 178.4 (1.5), $O_c$—Mo—$O_b$ 76.8 (9), $O_b$—

Mo—$O_b$ 87.0 (1.4) and 153.5 (1.7), $O_t$—Mo—$O_b$ 103.2 (1.4). Two O atoms of the 18-crown-6 are on a mirror plane; the symmetry of the crown is approximately $\bar{3}m$ ($D_{3d}$). The hydronium ion is on the mirror plane close to the center of the crown. H atoms are presumed to be between this ion and the three alternating O atoms of the crown that are located, on average, 0.11 Å closer to this ion than the other three O atoms of the crown. The hydronium ion has a large component of thermal motion perpendicular to the ring, which may indicate disorder and does not exclude a pyramidal shape of this ion. The average distances and angles in the crown in Å and degrees are: C—C 1.473 (33), C—O 1.418 (20), O—C—C 108.8 (1.6), C—O—C 112.6 (1.3).

**Introduction.** The title compound was formed in a study of co-condensation products of $MoO_3$ with various

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* IUPAC name: oxonium.

organic ligands (DeKock & McAfee, 1985). $MoO_3$ and tetrahydrofuran (THF) were co-condensed in a 'metal vapor' reactor at 77 K. The resulting gray–green solid was added to peroxide-free THF to form a slurry. A small amount of water was stirred in to form a clear green solution. Addition of 18-crown-6 caused, over a period of 36 h, precipitation of a yellow–green compound. The X-ray diffraction study showed the presence of the polyanion $[Mo_6O_{19}]^{2-}$, and two molecules of the crown ether, each centered by an O atom presumably in the form of a hydronium ion $H_3O^+$, requiring the uptake of three molecules of water. Peroxide-free THF was used, because we have shown previously that in an oxidizing environment a diperoxomolybdenum complex is formed (Shoemaker, Shoemaker, McAfee & DeKock, 1985).

**Experimental.** Crystals, pale yellow, invariably twinned. Parallelepiped, approximate dimensions $0.13 \times 0.09 \times 0.06$ mm, chosen for data collection. Syntex $P\bar{1}$ diffractometer, graphite monochromator, Mo $K\alpha$ radiation, $\theta$–$2\theta$ scan, scan speed $1°$ min$^{-1}$, $2\theta$ range $2.0°$ plus $\alpha_1$, $\alpha_2$ angular separation. 15 reflections used for measuring lattice parameters. Ratio of background time to scan time 1:2, one quarter of reciprocal space measured, $2\theta_{max} = 50°$. Three standard reflections, intensity variation $\pm 5\%$ (maximum). 2181 unique reflections measured, $h$ 0–13, $k$ 0–13, $l$ −10 to +10, 1312 with $F^2 > 2\sigma$ (less 47 with low values of $h$ and $l$ affected by twinning; see *Discussion*) used in refinement. No absorption correction (max. transmission 0.91, min. 0.81). $D_m$ by flotation in $CHBr_3/CCl_4$. Possible space groups: $P2/m$, $Pm$ and $P2$. First space group chosen, leading to satisfactory refinement. Octahedron formed by Mo atoms identified and located with a Patterson map. Refinement and difference Fourier maps revealed the surrounding O atoms, the non-H atoms of the crown ether, and an O atom, assumed to be $H_3O^+$, approximately at the center of the crown. H atoms not located with certainty; put in at calculated positions with isotropic $B$ and not refined. In last cycles of full-matrix least-squares refinement, positional and anisotropic thermal parameters varied for all non-H atoms (159 parameters). Final $R = 0.069$ (1265 reflections), $wR = 0.050$, $S = 1.56$. $(\Delta/\sigma)_{max} = 0.28$ for $B_{33}$ of O(9w), $(\Delta/\sigma)_{mean} = 0.016$. Final $\Delta\rho$ excursions $-0.87$ to $1.15$ e Å$^{-3}$ (near an Mo atom). Computer programs: local versions of those of Zalkin (undated). Quantity minimized $\sum w(|F_o| - |F_c|)^2$ with $w^{-1} = \sigma^2(F)$, derived from $\sigma^2(I) = C + Bt^2 + [0.02(C - Bt)]^2$ [$C$ is integrated count, $B$ total background count and $t$ (=2) ratio of scan time to background time]. Scattering factors from *International Tables for X-ray Crystallography* (1974): for Mo, scattering factor for $Mo^{3+}$ used; for O on the polyanion, $O^-$; for O of crown and hydronium, neutral O.

Table 1. *Positional parameters* $(\times 10^4)$ *and* $B_{eq}$ *for the* non-H *atoms*

$B_{eq}$ is the arithmetic mean of the principal axes of the thermal ellipsoids.

|  | $x$ | $y$ | $z$ | $B_{eq}(Å^2)$ |
|---|---|---|---|---|
| Mo(1) | 5726 (2) | 0 | 7568 (2) | 2·84 (5) |
| Mo(2) | 6926 (2) | 0 | 4611 (2) | 2·74 (5) |
| Mo(3) | 5000 | 2063 (2) | 5000 | 2·59 (5) |
| O(1) | 7151 (11) | 0 | 6693 (13) | 2·5 (4) |
| O(2) | 5934 (13) | 0 | 2607 (14) | 3·2 (4) |
| O(3) | 6314 (14) | 0 | 9379 (15) | 5·0 (6) |
| O(4) | 8274 (14) | 0 | 4279 (17) | 4·7 (5) |
| O(5) | 5617 (8) | 1710 (9) | 7045 (10) | 3·1 (3) |
| O(6) | 5000 | 3525 (13) | 5000 | 4·2 (5) |
| O(7) | 5000 | 0 | 5000 | 2·5 (6) |
| O(8) | 6530 (8) | 1699 (8) | 4628 (9) | 2·5 (3) |
| O(10) | 6560 (13) | 5000 | 1195 (17) | 4·3 (5) |
| C(11) | 6164 (15) | 3957 (18) | 1815 (19) | 4·9 (6) |
| C(12) | 6683 (14) | 2893 (15) | 1209 (18) | 4·2 (5) |
| O(13) | 7931 (10) | 2906 (11) | 1732 (12) | 4·9 (4) |
| C(14) | 8522 (18) | 1877 (19) | 1324 (21) | 6·1 (7) |
| C(15) | 9706 (18) | 1864 (17) | 2357 (23) | 6·3 (7) |
| O(16) | 10337 (10) | 2856 (12) | 2069 (14) | 5·6 (4) |
| C(17) | 11462 (18) | 2902 (17) | 3007 (27) | 8·0 (8) |
| C(18) | 12091 (17) | 3928 (17) | 2667 (26) | 7·4 (7) |
| O(19) | 11477 (15) | 5000 | 2946 (23) | 7·0 (7) |
| O(9w) | 9081 (16) | 5000 | 1912 (29) | 10·2 (9) |

**Discussion.** The crystal apparently is twinned (as shown by doubled spots on oscillation photographs), as are all of the many specimens we examined; one twin seems to be tipped slightly from the other around an axis normal to the $b$ axis. The data set was taken for one twin, with 47 reflections omitted for which there are good reasons (mainly shapes of the reflections) to believe that they are subject to interference from the other twin: these include all $0k0$, $0k1$, and $1k1$, plus five others with low $h$ and $l$. Intensity errors resulting from twin interference elsewhere in the reciprocal lattice are not excluded and may contribute significantly to the $R$ index and goodness of fit. The final parameters are listed in Table 1.* The $[Mo_6O_{19}]^{2-}$ anion (Table 2, Fig. 1a) consists of six Mo atoms, each surrounded by six O atoms in a distorted octahedral arrangement. The central O atom is at a center of symmetry $(2/m)$ and shared by all six octahedra. Four other O atoms of each octahedron are shared by two Mo atoms, and one O of each is terminal. The dimensions of the anion compare well with those observed previously (Clegg, Sheldrick, Garner & Walton, 1982).

The 18-crown-6 ether (Table 2, Fig. 1b) has mirror symmetry with O(10) and O(19) situated on a mirror plane. As in the structure of the 18-crown-6 complex with diperoxomolybdenum mentioned above

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\* Lists of structure factors, H-atom parameters, anisotropic thermal parameters, interatomic distances and angles, and distances to the least-squares plane through the six crown O atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42995 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Shoemaker, Shoemaker, McAfee & DeKock, 1985) the actual symmetry of the ring is close to $D_{3d}$, but the distortion is larger in the present compound. The average C—C distance is 1·473 (33) Å, the average C—O distance 1·418 (20) Å. The average O—C—C angle is 108·8 (1·6)°; the average C—O—C angle, 112·6 (1·3)°. O(10), O(13), O(13)' and O(9w), the O atom of the hydronium ion, are located within ±0·1 Å of the least-squares plane through the six O atoms of the ring. O(16), O(16)' and O(19) are on opposite sides

Table 2. *Dimensions of the* $[Mo_6O_{19}]^{2-}$ *anion and* 18-*crown*-6.H₃O⁺

$t$ = terminal, $c$ = central, $b$ = bridging.

(a) $[Mo_6O_{19}]^{2-}$ anion*

| | Distance (Å) | Range | | Distance (Å) | Range |
|---|---|---|---|---|---|
| Mo—Mo | 3·285 | (3·281–3·294) | Mo—O$_c$ | 2·323 | (2·315–2·328) |
| Mo—O$_t$ | 1·649 | (1·641–1·655) | Mo—O$_b$ | 1·933 | (1·868–1·977) |

| | Angle (°) | Range | | Angle (°) | Range |
|---|---|---|---|---|---|
| Mo—O$_c$—Mo | 90·0 | (89·9–90·1) | O$_c$—Mo—O$_b$ | 76·8 | (75·4–78·0) |
| | 180·0 | (fixed) | O$_b$—Mo—O$_b$ | 87·0 | (85·1–88·6) |
| Mo—O$_b$—Mo | 116·4 | (115·7–117·9) | | 153·5 | (151·9–155·9) |
| O$_c$—Mo—O$_t$ | 178·4 | (177·0–180·0) | O$_t$—Mo—O$_b$ | 103·2 | (101·6–106·5) |

(b) 18-Crown-6.H₃O⁺

| 1  2  3 | Distance (Å) 2–3 | Angle (°) 1–2–3 | Deviation of O from least-squares plane (Å) (e.s.d.'s 0·010 Å) |
|---|---|---|---|
| C(11)'—O(10)—C(11) | 1·418 (18) | 111·3 (1·8) | −0·037 |
| O(10)—C(11)—C(12) | 1·494 (23) | 108·9 (1·2) | |
| C(11)—C(12)—O(13) | 1·424 (18) | 109·0 (1·3) | |
| C(12)—O(13)—C(14) | 1·430 (23) | 114·2 (1·3) | +0·097 |
| O(13)—C(14)—C(15) | 1·494 (26) | 105·6 (1·5) | |
| C(14)—C(15)—O(16) | 1·386 (22) | 108·8 (1·6) | |
| C(15)—O(16)—C(17) | 1·404 (21) | 111·8 (1·5) | −0·221 |
| O(16)—C(17)—C(18) | 1·430 (28) | 110·3 (1·8) | |
| C(17)—C(18)—O(19) | 1·446 (20) | 109·9 (1·8) | |
| C(18)—O(19)—C(18)' | | 112·6 (2·3) | +0·284 |
| O(9w) | | | −0·037 |
| O(13)'—O(9w)—O(13) | 2·689 (15) | 121·8 (8) | |
| O(13)—O(9w)—O(19) | 2·737 (25) | 118·3 (4) | |
| O(16)—O(9w)—O(10) | 2·854 (23) | 120·4 (4) | |
| O(16)'—O(9w)—O(16) | 2·799 (15) | 118·6 (8) | |

* Individual e.s.d.'s in the distances range from 0·003–0·005 for Mo—Mo, 0·014–0·015 for Mo—O$_t$, 0·009–0·014 for Mo—O$_b$, and are 0·003 Å for Mo—O$_c$. The e.s.d.'s in the angles range from 0·1–0·7°.

of this plane at distances of −0·22 and +0·28 Å, respectively. The average distance of O(9w) to O(19), O(13) and O(13)' is 2·705 (28) Å, and that to O(10), O(16) and O(16)' is longer at 2·817 (32) Å. The hydrogen bonds are presumed to be between O(9w) and the first set of O atoms, which makes the hydrogen bonds appear to lie almost in a plane. Little can be said about the H-atom positions or the shape of the H₃O⁺ ion; a pyramidal shape with possible inversion cannot be excluded, particularly in view of the large component of thermal motion perpendicular to the ring (Fig. 2, root-mean-square displacement in that direction about 0·56 Å). Nor can hydrogen bonding to the second set of O atoms be excluded. In a complex of H₃O⁺ with a tetracarboxylic 18-crown-6 described by Behr, Dumas & Moras (1982) the hydrogen atoms were located by the X-ray analysis near the lines connecting the O of the hydronium with one of the two sets of three alternating O atoms of the crown at average distance of 2·714 (37) Å. The other set of O atoms is at average distance 2·791 (31) Å.

The packing diagram is given in Fig. 2. The closest contacts between the Mo moieties are through the terminal O atoms related by a center of symmetry [O(6)—O(6)' = 3·31 (3), O(3)—O(3)' = 3·46 (3), O(4)—O(4)' = 3·94 (3) Å]. O(4) has two short distances to C atoms of the ring: 3·38 (2) and 3·49 (2) Å. Corresponding distances for O(6) are 3·49 (2) and 3·62 (2); for O(3), 3·50 (2) and 3·64 (2) Å. Other distances between atoms of the Mo moieties and the crown ethers are greater than 3·49 Å, except that O(8) has a distance of 3·44 (2) Å to C(12) and 3·13 (2) Å to C(17) on a symmetry-related crown. The thermal motions of both atoms O(8) and C(17) are very anisotropic (Figs. 1a and 2), indicating possible disorder. Some other large anisotropies exist in the Mo moieties. These are not fully understood, but it is doubtful that they are caused by absorption.



Fig. 1. (a) *ORTEP*II (Johnson, 1976) view of the $[Mo_6O_{19}]^{2-}$ ion in $[Mo_6O_{19}]^{2-}.(18$-crown-6.H₃O⁺)₂. The thermal ellipsoids are given at 50% probability. The *b* axis is vertical. Single numbers refer to O atoms. (b) Projection of the 18-crown-6 ether with the water molecule down the *c* axis. Thermal ellipsoids at 25% probability. H atoms are shown by circles of arbitrary size.



Fig. 2. Packing diagram for $[Mo_6O_{19}]^{2-}.(18$-crown-6.H₃O⁺)₂. Thermal ellipsoids at 25% probability. O(7) at center of the Mo polyion is at $\frac{1}{2}0\frac{1}{2}$ and $\frac{1}{2}1\frac{1}{2}$. The crowns are bisected by the mirror plane at $y = \frac{1}{2}$.

Computing costs were provided by the OSU Computer Center.

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# Structures of the Ni^II Complexes of the Imine Ligands derived from 2,2-Diethoxyethylamine with Salicylaldehyde and 1,2- and 2,3-Hydroxynaphthaldehydes

By JUAN M. FERNÁNDEZ-G., MARÍA J. ROSALES,* R. A. TOSCANO AND R. G. TAPIA T.

*Instituto de Química de la Universidad Nacional Autónoma de México, Giudad Universitaria, Circuito Exterior, Coyoacán, 04510 México DF*

**Abstract.** (I): Bis[$N$-(2,2-diethoxyethyl)salicylideneaminato]nickel(II), [Ni($C_{13}H_{18}NO_3$)$_2$], $M_r = 531\cdot3$, monoclinic, $C2/c$, $a = 32\cdot244$ (7), $b = 5\cdot520$ (1), $c = 26\cdot742$ (5) Å, $\beta = 145\cdot798$ (11)°, $V = 2675\cdot50$ Å$^3$, $Z = 4$, $D_x = 1\cdot319$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = $0\cdot71069$ Å, $\mu = 7\cdot66$ cm$^{-1}$, $F(000) = 1128$, $T = 293$ K, $R = 0\cdot069$ for 1504 independent reflections. (II): Bis[$N$-(2,2-diethoxyethyl)-2-oxo-1-naphthylmethylideneaminato]nickel(II), [Ni($C_{17}H_{20}NO_3$)$_2$], $M_r = 631\cdot4$, monoclinic, $P2_1/c$, $a = 13\cdot493$ (5), $b = 6\cdot014$ (2), $c = 19\cdot783$ (5) Å, $\beta = 97\cdot00$ (2)°, $V = 1593\cdot36$ Å$^3$, $Z = 2$, $D_x = 1\cdot316$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = $0\cdot71069$ Å, $\mu = 6\cdot54$ cm$^{-1}$, $F(000) = 668$, $T = 293$ K, $R = 0\cdot053$ for 1674 independent reflections. (III): Bis[$N$-(2,2-diethoxyethyl)-3-oxo-2-naphthylmethylideneaminato]nickel(II), [Ni($C_{17}H_{20}NO_3$)$_2$], $M_r = 631\cdot4$, monoclinic, $C2/c$, $a = 30\cdot921$ (2), $b = 5\cdot658$ (4), $c = 26\cdot018$ (2) Å, $\beta = 134\cdot87$ (4)°, $V = 3225\cdot96$ Å$^3$, $Z = 4$, $D_x = 1\cdot300$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = $0\cdot71069$ Å, $\mu = 6\cdot46$ cm$^{-1}$, $F(000) = 1336$, $T = 293$ K, $R = 0\cdot068$ for 1648 independent reflections. The three compounds exhibit square-planar coordination around the metal atom. The chelate and aromatic ring(s) in each ligand are nearly planar in all three complexes with the $N$-diethoxyethyl groups at angles of 109–113° to the ring system. In compounds (I) to (III) there is a significant step at Ni between the two ligand planes. There are some small but significant differences between the Ni–N bond lengths in the two naphthaldehyde-derived complexes.

**Introduction.** The existence of electronic delocalization in metal complexes containing ligands from salicylaldehyde and naphthaldehyde was first proposed by Calvin & Wilson (1945) as an explanation for the stability order observed for a series of copper complexes. The stability order observed for the ligands acac (acetylacetonate), sal (salicylaldehydate) and 1,2- and 2,3-naphtha (naphtholate ion of hydroxynaphthaldehyde) was acac > 1,2-naphtha > sal > 2,3-naphtha and was related to the relative ease with which the different ligands allowed electronic delocalization to produce resonance structures like those shown in Fig. 1.

Resonance was later extensively used in enolate systems (Mehrotra, Bohra & Gaur, 1978) and electronic delocalization has also been suggested for the chelate ring in some imines derived from acetylacetone (Hatfield & Bunger, 1966).

Metal imine complexes with ligands derived from salicylaldehyde, 2-hydroxy-1-naphthaldehyde and 3-hydroxy-2-naphthaldehyde could also be expected to show delocalization of electron density and the extent of this process might be reflected in some structural features. However, to our knowledge, there have been no reports on structural comparison between compounds of this type.

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* To whom correspondence should be addressed.

Fig. 1. Resonance structures of the 1,2-naphtha ligand.