

Second and third row transition metal complexes of 1,1'-bi-2-naphthol and the X-ray crystal structures of (Buⁿ₄N)[MoO₂(acac)(BINO)] (1) and (Buⁿ₄N)₂(H₂BINO)₂[Mo₂O₇] (2)

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Abstract—The new complexes $[MoO_2(BINO)(acac)]^-(H_2BINO = 1,1'-bi-2-naphthol and acac = acetyl$ $acetonate), <math>[Mo_2O_5(BINO)_2]^{2-}$, $[MO_2(BINO)_2]^{2-}$ (M = Mo, W, Os) and $[M(PPh_3)_2(BINO)]$ (M = Pd, Pt) have been prepared. The X-ray crystal structures of the title compounds 1 and 2 are described and the vibrational spectra of the complexes reported. Copyright © 1997 Elsevier Science Ltd

Keywords: molybdenum; 1,1'-bi-2-napthol; X-ray crystal structure; Raman spectra.

The ligand 1,1'-bi-2-naphthol (H₂BINO) has potential uses as a chiral auxiliary in oxidation catalysis [1]. In the course of our work on peroxometalates as epoxidation catalysts [2] we have prepared a number of new complexes containing BINO for possible use as chiral epoxidation catalysts. In this paper we report the X-ray crystal structures of $(Bu_4^nN)[MoO_2(acac)$ (BINO)] (1) and $(Bu_4^nN)_2(H_2BINO)_2[Mo_2O_7]$ (2), and also the syntheses and vibrational spectra of other new complexes of BINO with tungsten, osmium, palladium and platinum.

Four X-ray crystal structures of H_2BINO complexes with titanium and lanthanides have been reported, for [Ti{ethylene-bis(4,5,6,7-tetrahydro-(S)-1-indenyl)}((S)-BINO)] [3] and Na₃[Ln((S)-BINO)₃] · 6THF · H₂O [4] (Ln = Pr, Nd or Eu). A number of BINO complexes of tungsten [5,6,7] and titanium [3,8,9] have been isolated, including [W₂ (BINO)₂(OBu¹₂)₂] [7], [W₂(BINO)₃] [7] and [W (BINO)₂Cl₂] · H₂O [5], the latter having been used as a catalyst for the ring opening polymerization of alkenes. The complex [W(BINO)₃] was used as a precatalyst in polymerization catalysis [6].

RESULTS AND DISCUSSION

Preparations and X-ray crystallography

Synthesis of the complexes. The molybdenum complexes were made from the tetrabutylammonium or tetramethylammonium salts of 1,1'-bi-2-naphthol and cis-[MoO₂(acac)₂] (acac = acetylacetonate); the tungsten complex from 1,1'-bi-2-naphthol and Na₂[WO₄] with tetrabutylammonium chloride; the osmium complex from trans-K₂[OsO₂(OCH₃)₄] and 1,1'-bi-2-naphthol; and the palladium and platinum complexes from [M(PPh)₃)₂Cl₂] and 1,1'-bi-2-naphthol with triethylamine.

X-ray crystal structure of 1. Orange crystals of the compound, suitable for X-ray analysis, were obtained after recrystallization in acetonitrile.

The X-ray analysis of 1 reveals a MoO₂ cis-dioxo coordination, the remaining coordination sites being occupied by cis-coordinated binaphthol and acetylacetonate ligands, as shown in Fig. 1. The octahedral coordination at molybdenum is severely distorted with angles at Mo ranging between 78.3(1) to 104.5(2)°, and 162.4(1) to 169.2(1)°. The bite angles associated with the chelating ligands are markedly reduced from orthogonal to 82.8(1) and 81.1(1)° for the binaphthol and acetylacetonate groups, respec-

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Fig. 1. The molecular structure of the anion in 1.

tively. There are characteristic displacements of the molybdenum atom towards the oxo substituents out of the plane of the atoms normal to these directions of 0.25 and 0.17 Å for O(1) and O(2), respectively; the two associated Mo—O distances, 1.698(3) [O(1)] and 1.701(3) Å [O(2)], are normal with the O—Mo—O angle being $104.5(2)^{\circ}$.

For both chelating ligands there is a noticeable asymmetry in their Mo—O coordination distances reflecting the powerful *trans* influence of the oxo ligands. In the case of the binaphthol unit [which exhibits a mean torsional twist of *ca* 58° about the C(13)—C(14) linkage] the two Mo—O distances differ by 0.13 Å and in the acetylacetonate moiety the difference is 0.16 Å. Furthermore, these asymmetries extend to include the adjacent C—O bonds in both ligands (Table 1); indeed in the acetylacetonate ligand a pattern of alternation of bond lengths extends throughout the whole ligand (with the exception of the terminal methyl groups), which maintains a coplanar geometry (to within 0.04 Å) with the molybdenum atom.

The only significant non van der Waals packing interaction is a $C-H\cdots O$ hydrogen bond between

one of the binaphthol C—H groups in one molecule and the O(1) oxo atom of a *c*-glide-related molecule, thereby forming a loosely-linked chain that extends in the crystallographic *c* direction [C···O, H···O distances 3.26, 2.34 Å; C—H···O angle 176°], Fig. 2.

X-ray crystal structure of 2. Recrystallization of the orange crystals of 1 from acetonitrile gave beige crystals which were found by X-ray analysis to be $(Bu_4^nN)_2(H_2BINO)_2[Mo_2O_7]$ (2). The structure comprises an Mo₂O₇ core hydrogen-bonded to two binaphthol units (vide infra), Fig. 3. Thus, unlike 1 where the binaphthol acts as a bidentate ligand, here no coordination has occurred. Initial analysis of the structure indicated a linear geometry for the Mo-O-Mo linkage (with somewhat short Mo-O distances), the bridging oxygen atom being positioned on a crystallographic inversion centre. However, the bridging atom exhibited significant thermal anisotropy normal to the Mo...Mo direction, indicating that a more realistic model is one in which there is a superimposition of two half occupancy non-linear geometries related by the centre of symmetry. Splitting of the position of this bridging oxygen atom resulted in more realistic thermal parameters, and bridging

MoO(1)	1.698(3)	Mo-O(2)	1.701(3)
Mo-O(24)	1.955(3)	Mo-O(25)	2.060(3)
Mo—O(3)	2.087(3)	Mo-O(31)	2.222(3)
O(3)—C(4)	1.335(5)	C(4)—C(13)	1.392(5)
C(13)—C(14)	1.488(5)	C(14)C(23)	1.384(5)
C(23)—O(24)	1.364(4)	O(25)C(27)	1.284(6)
C(27)—C(28)	1.369(8)	C(28)C(29)	1.394(7)
C(29)O(31)	1.254(5)		
$O(1) - M_0 - O(2)$	104 5(2)	$O(1) - M_2 - O(24)$	00 51(13)
O(1) = MO = O(2)	04.3(2)	$O(1) = M_0 = O(24)$	01.70(14)
$O(2) = M_0 = O(24)$	94.43(13)	O(1) = MO = O(23)	91.79(14)
O(2) - Mo - O(25)	94.56(14)	O(24)-Mo-O(25)	163.32(12)
O(1)—Mo—O(3)	93.10(12)	O(2)MoO(3)	162.41(13)
O(24)—Mo—O(3)	82.79(10)	O(25)—Mo—O(3)	84.39(11)
O(1)—Mo—O(31)	169.22(13)	O(2)-Mo-O(31)	84.21(14)
O(24)MoO(31)	85.94(11)	O(25)—Mo—O(31)	81.03(12)
O(3)—Mo—O(31)	78.28(11)	C(4)O(3)Mo	126.2(2)
O(3) - C(4) - C(13)	121.3(3)	C(4) - C(13) - C(14)	119.0(3)
O(24)—C(23)—C(14)	121.0(3)	C(23)—O(24)—Mo	120.3(2)
C(27)—O(25)—Mo	133.5(3)	O(25)C(27)C(28)	125.3(5)
C(27)—C(28)—C(29)	125.5(5)	O(31)C(29)C(28)	123.9(5)
C(29)-O(31)-Mo	130.4(3)		

Table 1. Selected bond lengths (Å) and angles (°) for 1



Fig. 2. Part of one of the loosely hydrogen-bonded chains present in the crystal structure of 1.

Mo—O distances and a Mo—O—Mo angle $[155(2)^{\circ}]$ comparable to those found in other related systems such as $(Bu_4^nN)_2[Mo_2O_7]$ [10] and $[(\eta-C_5H_4CH_3)$ Mo(CO)₂(μ -dppm)Pt(dppm)]₂[Mo₂O₇] [11]. The remaining Mo—O distances are unexceptional, Table 2. A detailed analysis of the angular geometry at molybdenum is not justified in view of the disorder described above. The binaphthol unit exhibits a characteristic skewed geometry with a mean torsional twist about the bond linking the two naphthalene rings of *ca* 85°, a value substantially larger than that observed for the coordinated counterpart of 1.

An inspection of the packing shows that the pattern of hydrogen-bonding between the binaphthol units and the Mo₂O₇ core alluded to above extends to form chains of molecules, which propagate in the crystallographic *a* direction (O···O distances 2.68 and 2.70 Å), Fig. 4. Adjacent chains are interleaved and stabilized by T-type edge to face aromatic–aromatic interactions between naphthyl rings (centroid··· centroid distance between C₆H₄ rings 5.02 Å).

Vibrational and ¹H NMR spectra

IR and Raman spectroscopic data for the free ligand and the complexes are listed in Table 3. General assignments of the IR spectrum of 1,1'-bi-2-naphthol have been reported [12,13]. The existence of two bands of the O---H stretch, v(O--H), at 3485 and 3401 cm⁻¹, was ascribed [12] to the asymmetry of 1,1'-bi-2-naphthol in the solid state, since the two naphthyl rings lie in two different planes. In the IR spectra of our BINO complexes the band corresponding to v(O-H) was not observed due to loss of protons on coordination and the v(C-O) band is shifted from 1382 cm⁻¹ in H_2BINO to approximately 1366 cm⁻¹ in the complexes, suggesting coordination through the oxygen atoms of the hydroxyl groups (similar effects are noted for the IR spectra of catecholato complexes [14,15]). The C—C stretch bands of H₂BINO are only slightly shifted in the complexes. A new band at approximately 1557 cm⁻¹, assigned to v(C--C), is also seen in the Raman spectrum of 1, together with



Fig. 3. The Mo₂O₇ core and adjacent hydrogen-bonded binaphthol units in the structure of 2; the broken bonds correspond to the alternative orientation of the Mo—O—Mo bridge.

Table 2. Selected bond lengths (Å) for 2

Mo-O(1)	1.693(9)	Mo-O(2)	1.718(7)
Mo-O(3)	1.647(14)	Mo-O(4)	1.90(2)
Mo'—O(4)	1.88(2)	Mo· · ·Mo′	3.701(2)

acetylacetonate bands in both IR and Raman spectra. In 2 the O—H stretch of 1,1'-bi-2-naphthol is shifted from 3485 and 3401 cm⁻¹ to 3124 cm⁻¹. In this compound the v(C-O) band is shifted to 1370 cm⁻¹, an intermediate position between the values for the complexes (shifted to lower wave-numbers) and for H₂BINO.

¹H NMR data for H_2BINO were measured and the data agree well with those of Puga *et al.* [16], there was, however, relatively little change in the spectra on complexation.

EXPERIMENTAL

All starting materials were supplied by Aldrich and used without further purification. The complexes $[MoO_2(acac)_2]$ [17], trans-K₂[OsO₂(OCH₃)₄] [18],

 $[Pd(PPh_3)_2Cl_2 [19]$ and $[Pt(PPh_3)_2Cl_2]$ [20] were made by the cited literature procedures. The salts $(Bu_4^nN)(HBINO)$ and $(Me_4N)(HBINO)$ were prepared by refluxing ethanolic solutions of H_2BINO and Bu_4^nNCl or Me_4NCl in equimolar proportions, filtered and cooled.

Preparation of (Bu₄ⁿN)[MoO₂(acac)(BINO)]

To a solution of $[MoO_2(acac)_2]$ (0.16 g, 0.5 mmol) in acetone (5 cm³) was added a solution of (Bu^a₄N)(HBINO) (0.26 g, 0.5 mmol) in acetone (10 cm³). The yellow precipitate formed was filtered, washed with acetone and dried in a desiccator over silica gel. Yield = 0.32 g, 0.24 mmol, 49%. Orange crystals of the compound were obtained by recrystallization in acetonitrile.

Preparation of $(Bu_4^nN)_2(H_2BINO)_2[Mo_2O_7]$

The orange crystals obtained by recrystallization of $(Bu_4^nN)[MoO_2(acac)(BINO)]$ were left in acetonitrile for a week. The colour faded and beige crystals of $(Bu_4^nN)_2(H_2BINO)_2[Mo_2O_7]$ were obtained.



Fig. 4. Part of a pair of interleaved hydrogen-bond chains of Mo_2O_7 and binaphthol molecules in the structure of 2.

Preparation of (Me₄N)₂[MoO₂(BINO)₂] · 2MeOH

To a solution of $[MoO_2(acac)_2]$ (0.16 g, 0.5 mmol) in methanol (20 cm³) was added 1,1'-bi-2-naphthol (0.29 g, 1.0 mmol) and tetramethylammonium chloride (0.11 g, 1.0 mmol). The resulting yellow solution was heated to reflux for 2 h, filtered hot and reduced to approximately half of the volume by rotary evaporation. Beige crystals were obtained after refrigeration for 4 days, filtered and dried in a desiccator with silica gel. Yield = 0.050 g, 0.055 mmol, 11%.

Preparation of (Me₄N)₂[Mo₂O₅(BINO)₂]

To a solution of $[MoO_2(acac)_2]$ (0.082 g, 0.25 mmol) in acetone (3 cm³) was added a solution of (Me₄N)(HBINO) (0.18 g, 0.5 mmol) in acetone (10 cm³). The yellow suspension obtained was stirred for 1 h. The yellow precipitate was filtered and dried in a desiccator over silica gel. Yield = 0.13 g, 0.24 mmol, 49%.

Preparation of (Bu₄ⁿN)₂[WO₂(BINO)₂]·2H₂O

To an aqueous solution of sodium tungstate $(0.17 \text{ g}, 0.5 \text{ mmol}, 10 \text{ cm}^3 \text{ of water})$ was added an ethanolic suspension of 1,1'-bi-2-naphthol (0.29 g, 1.0 mmol, 20 cm³ of ethanol) and an aqueous solution of tetrabutylammonium chloride (0.28 g, 1.0 mmol, 2 cm³ of water). The resulting light yellow solution (with some 1,1'-bi-2-naphthol suspended) was heated at reflux for 3 h and filtered. Beige crystals were obtained after refrigeration for 3 days, filtered, washed with ethanol and dried in a desiccator with silica gel. Yield = 0.279 g, 0.21 mmol, 43%.

Preparation of K₂[OsO₂(BINO)₂] ·6H₂O

To a stirred solution of trans-K₂[OsO₂(OCH₃)₄] (0.20 g, 0.47 mmol) in methanol (15 cm³) was added a methanolic solution of 1,1'-bi-2-naphthol (0.29 g, 1.0 mmol, 5 cm³ of methanol). The resulting dark green solution was left stirring for *ca* 10 min, a dark-

					Vibrational		
	Analysis" (%)				spectra ^{<i>b,c</i>} (cm ⁻¹)		
Compound	С	Н	Ν	v(CC)	v(C—O)	$v^{s}(MO_{2})^{d}$	$v^{as}(MO_2)$
1,1'-Bi-2-naphthol				1618 vs/1596 vs	1381 vs		
				1620 (2)/1592 (3)	1381 (10)		
(Bu ₄ "N)[MoO ₂ (acac)(BINO)]	64.6	7.3	2.1	1612 vs/1590 vs	1365 m	920 vs	892 vs
	(65.3)	(7.3)	(2.1)	1616 (2)/1591 (2)	1366 (10)	921 (4)	896 (2)
$(Bu_4^n N)_2 (H_2 BINO)_2 [Mo_2 O_7]$	63.2	7.3	2.0	1624 s/1584 m	1379 m	933 m	872 vs
	(63.5)	(7.4)	(2.1)	1622 (3)/1584 (5)	1370 (9)	937 (10)	866 (4)
$(Me_4N)_2[MoO_2(BINO)_2] \cdot 2MeOH$	66.3	6.6	3.2	1620 s/1583 m	1370 s	950 s	871 m
	(66.1)	(6.2)	(3.1)	1623 (3)/1584 (8)	1367 (10)	957 (2)	
$(Me_4N)_2[Mo_2O_5(BINO)_2]$	57.9	5.8	3.2	1624 vs/1584 s	1360 sh	896 s	808 vs
	(58.3)	(4.9)	(2.8)	1624 (2)/1584 (6)	1366 (10)	900 (6)	804 (2)
$(Bu_4^{\prime\prime}N)_2[WO_2(BINO)_2] \cdot 2H_2O$	66.0	6.5	1.4	1618 vs/1593 vs	1365 sh	945 s	878 m
	(66.3)	(7.7)	(2.2)	1618 (2)/1590 (3)	1370 (10)	951 (1)	
$K_2[OsO_2(BINO)_2] \cdot 6H_2O$	49.3	2.0		1615 s/1586 s	1360 m		844 vs
	(49.2)	(3.7)					
[Pd(PPh ₃) ₂ (BINO)] · EtOH	72.4	5.3		1619 s/1594 s	1370 sh	_	
	(72.5)	(5.0)		1615 (1)/1585 (10)	1368 (8)		
[Pt(PPh ₃) ₂ (BINO)] · EtOH	65.6	4.0		1615 m/1587 m	1368 m		
	(66.3)	(4.6)		1613 (2)/1586 (7)	1368 (4)		

Table 3. Analytical and IR and Raman spectroscopic data for complexes of 1,1'-bi-2-naphthol

"Calculated values in parentheses.

^b Raman data in italics.

v indicates a stretching mode; intensity of bands: vs = very strong, s = strong, m = medium, sh = shoulder.

 d v(Mo-O) for Mo₂O₇.

brown precipitate started to form and then diethyl ether (20 cm^3) was added. The solution turned darker and upon filtration a brown solid was isolated. The solid was washed with diethyl ether and dried in a desiccator with silica gel. Yield = 0.23 g, 0.23 mmol, 50%.

Preparation of [Pd(PPh₃)₂(BINO)] · EtOH

To a stirred suspension of $[Pd(PPh_3)_2Cl_2]$ (0.35 g, 0.5 mmol) in ethanol (10 cm³) was added a methanolic solution of 1,1'-bi-2-naphthol (0.29 g, 1.0 mmol, 4 cm³ of methanol) and triethylamine (0.6 cm³, 4.0 mmol). The resulting yellow suspension was stirred for 7 days and a suspension of a yellow solid in a red solution was obtained. It was centrifuged and the red solution isolated. To this solution was added the same volume of water and an orange solid was formed. It was centrifuged and dried in a desiccator over silica gel. Yield = 0.13 g, 0.14 mmol, 27%.

Preparation of [Pt(PPh₃)₂(BINO)] · EtOH

To a stirred suspension of $[Pt(PPh_3)_2Cl_2]$ (0.40 g, 0.5 mmol) in ethanol (10 cm³) was added a methanolic solution of 1,1'-bi-2-naphthol (0.29 g, 1.0 mmol, 4 cm³ of methanol) and triethylamine (0.6 cm³, 4.0 mmol). The resulting white suspension was stirred for 7 days and a yellow suspension was obtained. It was centrifuged and the yellow solid isolated and dried in a desiccator over silica gel. Yield = 0.39 g, 0.37 mmol, 74%. Yellow crystals of the compound were obtained by recrystallization in acetonitrile.

Crystal structure determinations

Crystal data for 1. $[C_{25}H_{19}O_6Mo][(C_4H_9)_4N]$, M = 753.8, monoclinic, space group $P2_1/c$, a = 10.708(2), b = 18.451(2), c = 19.439(3) Å, $\beta = 91.92(1)^\circ$, V = 3838(1) Å³, Z = 4, $D_c = 1.30$ g cm⁻³, μ (Cu- K_{α}) = 31.6 cm⁻¹, F(000) = 1592. An orange/yellow plate of dimensions $0.33 \times 0.20 \times 0.05$ mm was used.

Data collection and processing. Data were measured on a Siemens P4/PC diffractometer with Cu- K_{α} radiation (graphite monochromator) using ω -scans. 5351 independent reflections were measured ($2\theta \leq 116^{\circ}$) of which 4443 had $|F_{\circ}| > 4\sigma(|F_{\circ}|)$ and were considered to be observed. The data were corrected for Lorentz and polarization factors, and a Gaussian absorption correction (face-indexed numerical) was applied; the maximum and minimum transmission factors were 0.851 and 0.539, respectively.

Structure analysis and refinement. The structure was solved by the heavy atom method and the non-hydrogen atoms were refined anisotropically. The hydrogen atoms of the methyl groups attached to sp^2 centres were located from ΔF maps and subsequently optimized. The positions of the remaining hydrogens were

idealized, assigned isotropic thermal parameters, $[U(H) = 1.2U_{eq}(C); U(H) = 1.5U_{eq}(C-Me)]$, and allowed to ride on their parent carbon atoms. Refinement was by full-matrix least-squares based on F^2 to give $R_1 = 0.042$, $wR_2 = 0.105$ for the observed data and 443 parameters. The maximum and minimum residual electron densities in the final ΔF map were 0.58 and -0.64 e Å⁻³, respectively. The mean and maximum shift/error ratios in the final refinement cycle were 0.003 and -0.023, respectively.

Crystal data for 2. $[Mo_2O_7][(C_4H_9)_4N]_2 \cdot [C_{20}H_{14}O_2]_2$, M = 1361.4, triclinic, space group $P\overline{1}$, a = 10.371(3), b = 10.759(3), c = 16.759(5) Å, $\alpha = 103.84(2)$, $\beta = 102.89(2)$, $\gamma = 92.73(2)^\circ$, V = 1759.9(8) Å³, Z = 1 (the molecule has crystallographic C_i symmetry), $D_c = 1.29$ g cm⁻³, μ (Cu- K_{α}) = 33.8 cm⁻¹, F(000) = 718. A clear prismatic plate of dimensions $0.21 \times 0.12 \times 0.01$ mm was used.

Data collection and processing. Data were measured on a Siemens P4/PC diffractometer with Cu- K_{α} radiation (graphite-monochromator) using ω -scans. 2851 independent reflections were measured ($2\theta \le 120^{\circ}$), of which 2177 had $|F_o| > 4\sigma(F_o|)$ and were considered to be observed. The data were corrected for Lorentz and polarization factors and a Gaussian absorption correction (face-indexed numerical) was applied; the maximum and minimum transmission factors were 0.961 and 0.729, respectively.

Structure analysis and refinement. The structure was solved by the heavy-atom method and the non-hydrogen atoms were refined anisotropically. The hydroxyl hydrogen atoms were located from a ΔF map and were refined isotropically subject to an O-H distance constraint. The positions of the remaining hydrogens were idealized, assigned isotropic thermal parameters, $[U(H) = 1.2U_{eq}(C), U(H) = 1.5U_{eq}(C-Me)]$ and allowed to ride on their parent carbon atoms. Refinement was by full-matrix least-squares based on F^2 to give $R_1 = 0.061$, $wR_2 = 0.149$ for the observed data and 405 parameters. The maximum and minimum residual electron densities in the final ΔF map were 0.53 and -0.66 e Å⁻³, respectively. The mean and maximum shift/error ratios in the final refinement cycle were 0.000 and 0.000, respectively.

For both structures, computations were carried out on 50 MHz 486 PC computers using the SHELXTL-PC program system [21]. Additional material available from the Cambridge Crystallographic Data Centre comprises fractional atomic coordinates, Hatom coordinates, thermal parameters and remaining bond lengths and angles.

Characterization

IR spectra were measured as KBr discs on a Perkin– Elmer 1720 FT instrument. Raman spectra were recorded on as powders on a Perkin–Elmer 1760 X FT-IR instrument fitted with a 1700 X NIR FT-Raman accessory (Spectron Nd: YAG laser, 1064 nm excitation). ¹H NMR spectra were measured on a Jeol EX-270 spectrometer (¹H, 270.05 MHz). Microanalyses were measured by the Microanalytical Department, Imperial College.

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