

2.51 Å, and much shorter than the sum of van der Waals radii, 4.15 Å (Pauling, 1960).

The structure of this entire group of atoms, namely  $\text{Se}_2\text{Te}^{\text{II}}\text{Br}_2\text{Te}^{\text{IV}}\text{Br}_4$ , is very similar to that found for the unit  $\text{Br}_2\text{Se}^{\text{II}}\text{Br}_2\text{Se}^{\text{IV}}\text{Br}_4$  in  $[(\text{C}_2\text{H}_5)_2\text{N}]_2\text{Se}_2\text{Br}_8$  (Hauge, Maroy & Odegard, 1988). In that structure, the terminal  $\text{Se}^{\text{II}}-\text{Br}$  bond lengths are 2.461 (2) and 2.427 (2) Å, while the bridging  $\text{Se}^{\text{II}}-\text{Br}$  bond lengths are 2.993 (2) and 3.010 Å; the bridging  $\text{Se}^{\text{IV}}-\text{Br}$  bond distances are 2.748 (2) and 2.720 (2) Å, the bonds *trans* to them are 2.459 (2) and 2.442 (2) Å and the third pair of  $\text{Se}^{\text{IV}}-\text{Br}$  bond distances is 2.522 (2) and 2.600 (2) Å. For Se and Br, the sum of the covalent radii is 2.31 Å and the sum of the van der Waals radii is 3.95 Å (Pauling, 1960). The dihedral angle between the  $\text{Se}^{\text{II}}\text{Br}_4$  plane and the equatorial  $\text{Se}^{\text{IV}}\text{Br}_4$  plane is 57°. The corresponding angles in the present structure are 58° for the planes that share Br(5) and Br(6), and 37° for the planes that share Br(11) and Br(12). The various DMSeU planes make dihedral angles with their  $\text{Se}_2\text{Te}^{\text{II}}\text{Br}_2$  planes that range from 68.0 to 71.4°.

(c)  $\text{CH}_3\text{CN}$  and  $\text{CH}_3\text{OH}$ . Four molecules of solvation were found in the structure, three  $\text{CH}_3\text{CN}$  and one  $\text{CH}_3\text{OH}$ . For  $\text{CH}_3\text{CN}$ , the average distances and angles are  $\text{C}-\text{C} = 1.10$  (3) Å,  $\text{C}-\text{N} = 1.47$  (2) Å and  $\text{C}-\text{C}-\text{N} = 173$  (2)° and for  $\text{CH}_3\text{OH}$ ,  $\text{C}-\text{O} = 1.40$  (5) Å. All of these values appear to be normal. Each of the four solvent molecules has one close contact with one of the four available H atoms of the  $\text{NH}_2$  groups of DMSeU. The  $\text{N}\cdots\text{H}$  contacts from  $\text{CH}_3\text{CN}$  vary from 2.12 to 2.26 Å, while the angles

$\text{C}-\text{N}\cdots\text{H}$  vary from 99 to 139°. The contact  $\text{O1}\cdots\text{H8Ea} = 1.99$  Å and  $\text{C19}-\text{O1}\cdots\text{H8Ea} = 128^\circ$ . All of these distances are considerably shorter than van der Waals contacts, and may be hydrogen bonds. The angles  $\text{N}\cdots\text{H}-\text{N}$  vary from 151 to 167° and  $\text{N8}-\text{H8Ea}\cdots\text{O1} = 150^\circ$ , all values that deviate considerably from 180°.

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## Structure of Bis(2,2'-bipyridine)dichlororhodium(III) Chloride Dihydrate

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**Abstract.**  $[\text{RhCl}_2(\text{C}_{10}\text{H}_8\text{N}_2)_2]\text{Cl}\cdot 2\text{H}_2\text{O}$ ,  $M_r = 557.7$ , triclinic,  $P\bar{1}$ ,  $a = 12.9712$  (5),  $b = 12.402$  (1),  $c = 6.9998$  (4) Å,  $\alpha = 100.123$  (5),  $\beta = 93.607$  (3),  $\gamma =$

$90.668$  (4)°,  $V = 1106.0$  (1) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.67$  Mg m<sup>-3</sup>, Mo  $K\alpha$  radiation (graphite crystal monochromator,  $\lambda = 0.71069$  Å),  $\mu = 11.49$  cm<sup>-1</sup>,  $F(000) = 560$ ,  $T = 293$  K. Final  $R = 0.052$  for 3633 independent observed reflections and 325 variables.

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The structure consists of cationic units with rhodium as the central atom in a pseudo-octahedral coordination. The crystal packing is completed by one chloride anion and two water molecules. The angles around the Rh atom range from 176.0 (2) to 177.2 (2)° for *trans* angles, and from 79.7 (2) to 98.3 (2)° for *cis* angles.

**Introduction.** Rh<sup>III</sup> polyamine and polypyridine complexes can be used as potential precursors of Rh<sup>II</sup> and Rh<sup>I</sup> species (Kew, DeArmond & Hank, 1974; Mulazzani, Emmi, Hoffman & Venturi, 1981; Lilie, Simic & Endicott, 1975; Schwarz & Creutz, 1983; Anderson & Gregory, 1989; *Comprehensive Coordination Chemistry*, 1987). We have recently prepared the cationic complex [RhCl(η<sup>2</sup>-PC)(η<sup>2</sup>-PCCI)-(N-N)]SbF<sub>6</sub>, where PC = (C<sub>6</sub>H<sub>4</sub>)PPh<sub>2</sub>, PCCI = (*o*-ClC<sub>6</sub>H<sub>4</sub>)PPh<sub>2</sub> and N-N = 2,2'-bipyridine, 1,10-phenanthroline, from the neutral complex RhCl<sub>2</sub>(η<sup>2</sup>-PC)(η<sup>2</sup>-PCCI) (Lahuerta, Latorre, Martínez-Máñez, Paya & Tiripicchio, 1991). Cyclic voltammetry measurements indicate that [RhCl(η<sup>2</sup>-PC)(η<sup>2</sup>-PCCI)-(N-N)](SbF<sub>6</sub>) reduces to an Rh<sup>I</sup> species in two one-electron partially reversible processes. The intermediate Rh<sup>II</sup> species can be detected by ESR spectroscopy; however, a disproportionation reaction prevents its isolation.

Following our investigation of Rh<sup>III</sup> complexes containing polypyridine ligands, we have investigated the reaction between RhCl<sub>2</sub>(η<sup>2</sup>-PC)(η<sup>2</sup>-PCCI) and 2,2'-bipyridine. Surprisingly, we have observed that under mild reaction conditions (CH<sub>2</sub>Cl<sub>2</sub> refluxing and a tenfold excess of bipyridine), cleavage of the metal-carbon bond occurs giving the well known compound [RhCl<sub>2</sub>(bpy)<sub>2</sub>]Cl·2H<sub>2</sub>O (bpy = 2,2'-bipyridine). We report here the crystal and molecular structure of the [RhCl<sub>2</sub>(bpy)<sub>2</sub>]Cl·2H<sub>2</sub>O complex.

**Experimental.** Chemical reaction was carried out under dry argon by Schlenk-line procedures. Over a solution of RhCl<sub>2</sub>(η<sup>2</sup>-PC)(η<sup>2</sup>-PCCI) (Lahuerta, Martínez-Máñez, Sanz, Cantarero & Torrens, 1988) in CH<sub>2</sub>Cl<sub>2</sub> (100 mg, 0.137 mmol), was added 2,2'-bipyridine (214 mg, 1.37 mmol). The mixture was refluxed for 1 h 45 min. The resulting yellow solution was evaporated to dryness under vacuum and the residue dissolved in CH<sub>2</sub>Cl<sub>2</sub> and crystallized by slow diffusion of *n*-hexane into the CH<sub>2</sub>Cl<sub>2</sub> solution. After four days yellow crystals of [RhCl<sub>2</sub>(bpy)<sub>2</sub>]Cl·2H<sub>2</sub>O suitable for X-ray analysis were obtained. The reaction seems to be complicated. The only crystalline compound isolated was [RhCl<sub>2</sub>(bpy)<sub>2</sub>]Cl·2H<sub>2</sub>O (50% yield). A crystal having approximate dimensions 0.1 × 0.1 × 0.3 mm was used for the structure determination.

Diffraction data were collected on an Enraf-Nonius CAD-4 single-crystal diffractometer using

graphite-monochromated Mo *K*α radiation (λ = 0.71069 Å). Unit-cell parameters were determined from the angular settings of 25 reflections with 20 < θ < 25°. A total of 6950 reflections were measured, *hkl* range (-18, -17, 0) to (18, 17, 9) 0 < θ < 30°, using the ω-2θ scan technique and a variable scan rate with a maximum scan time of 60 s per reflection. Intensity checked by monitoring three standard reflections every 60 min. Final drift corrections were between 0.99 and 1.02. Profile analysis was performed on all reflections (Lehmann & Larsen, 1974; Grant & Gabe, 1978). Empirical absorption correction applied using ψ scans (North, Phillips & Mathews, 1968), correction factors ranged from 0.74 to 0.99. Some double-measured reflections averaged,  $R_{\text{int}} = \sum(I - \langle I \rangle) / \sum(I) = 0.027$ , resulting in 5430 unique reflections, 3633 of which were 'observed' with  $I > 3\sigma(I)$ . Lorentz and polarization corrections were applied and the data reduced to  $|F_o|$  values.

The structure was solved by Patterson interpretation using SHELXS86 (Sheldrick, 1985) which allowed us to locate the Rh atom. The remaining non-H atoms were located from successive Fourier syntheses. The structure was refined by least squares using SHELX76 (Sheldrick, 1976). Isotropic and anisotropic refinements followed by difference Fourier synthesis gave the location of some H atoms, the remainder of which were included in geometrically calculated positions. During the final stages of refinement, the positional and anisotropic thermal parameters for all non-H atoms were refined. The H atoms were refined riding on their parent atoms. For all H atoms, the isotropic parameters were fixed ( $U = 0.09 \text{ \AA}^2$ ). Final conventional agreement factors were  $R = 0.052$  and  $wR = 0.057$  for 3633 'observed' reflections and 325 variables. The function minimized was  $\sum w(F_o - F_c)^2$ .  $w = 1 / [\sigma^2(F_o) + 0.00669(F_o)^2]$ , with  $\sigma^2(F_o)$  from counting statistics. The maximum shift/e.s.d. ratio in the last full-matrix least-squares cycle was less than 0.2. The final difference Fourier map showed no peaks higher than 1.2 e Å<sup>-3</sup> or deeper than -1.4 e Å<sup>-3</sup>. The maximum residual electron density was located near the Rh atom. Atomic scattering factors and corrections for anomalous dispersion for the Rh atom were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99-101, 149-150). Geometrical calculations were performed with PARST (Nardelli, 1983).

**Discussion.** By reaction of RhCl<sub>2</sub>(η<sup>2</sup>-PC)(η<sup>2</sup>-PCCI) and bidentate phosphines, the PCCI ligand is readily released to give the corresponding complex RhCl<sub>2</sub>(η<sup>2</sup>-PC)(η<sup>2</sup>-P-P), P-P = bidentate phosphine (Lahuerta, Latorre, Martínez-Máñez & Sanz, 1988; Lahuerta, Latorre, Martínez-Máñez, Paya & Tiripicchio, 1991). In contrast, the same reaction

with a bidentate nitrogen donor ligand such as bipyridine was more complicated and the PCl and orthometallated phosphines were released from the  $\text{RhCl}_2(\eta^2\text{-PC})(\eta^2\text{-PCCl})$  complex to give the title compound  $[\text{RhCl}_2(\text{bpy})_2]\text{Cl}\cdot 2\text{H}_2\text{O}$ .

The complex  $[\text{RhCl}_2(\text{bpy})_2]\text{Cl}\cdot 2\text{H}_2\text{O}$  has been studied extensively; electronic spectra (Gillard, Osborn & Wilkinson, 1965), acid adduct formation (Gillard & Wilkinson, 1964), IR and Raman spectra (Kulasingam, McWhinnie & Miller, 1969; Martin, McWhinnie & Waind, 1961; McWhinnie, 1964),  $^1\text{H}$  NMR spectra (Kulasingam, McWhinnie & Miller, 1969), a proposed mechanism of formation (Rund, Basolo & Pearson, 1964; Rund, 1968) and X-ray powder patterns (Berka, Gagne, Philippon & Wheeler, 1970) have been given. However, assignment of the geometric configuration for the  $[\text{RhX}_2(\text{bpy})_2]^+$  unit, where  $X$  is a halogen atom, as well as the phenanthroline analogue, has been unclear for some years.

Fig. 1 (ORTEP, Johnson, 1965) shows a perspective view of the molecule with the atomic numbering scheme. A list of atomic coordinates for the non-H atoms is given in Table 1.\* Selected bond distances and angles are given in Table 2.

The structure consists of a mononuclear cationic unit with the Rh atom coordinated by two bipyridine ligands and two *cis* Cl atoms. The crystal packing is completed by one chloride anion and two water molecules. The water molecules contribute to the molecular packing forming a network of hydrogen bonds, which includes the chloride anion. The main

\* Lists of structure amplitudes, anisotropic thermal parameters, torsion angles, full tables of distances and angles, and least-squares-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53515 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

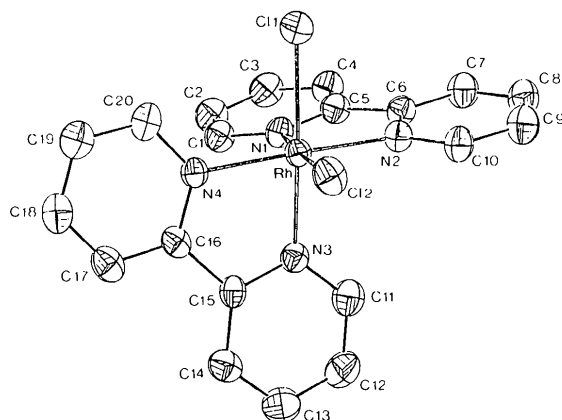


Fig. 1. Perspective view and atomic numbering of the  $[\text{RhCl}_2(\text{bpy})_2]^+$  unit.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters, for the non-H atoms, with *e.s.d.*'s in parentheses

$$B_{\text{eq}} = (1/3)\sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}$ ( $\text{\AA}^2$ )
Rh	0.3705 (1)	0.2160 (1)	0.1006 (1)	2.07 (1)
Cl(1)	0.4467 (1)	0.0816 (1)	0.2561 (2)	3.10 (4)
Cl(2)	0.4373 (1)	0.3536 (1)	0.3491 (2)	3.24 (4)
N(1)	0.3044 (4)	0.0950 (4)	-0.1034 (7)	2.4 (1)
N(2)	0.2417 (4)	0.1834 (4)	0.2320 (7)	2.6 (1)
C(1)	0.3378 (5)	0.0596 (5)	-0.2788 (8)	2.9 (2)
C(2)	0.2952 (6)	-0.0314 (5)	-0.4017 (9)	3.4 (2)
C(3)	0.2139 (6)	-0.0879 (5)	-0.340 (1)	3.5 (2)
C(4)	0.1774 (5)	-0.0490 (5)	-0.1605 (9)	3.3 (2)
C(5)	0.2235 (5)	0.0431 (5)	-0.0426 (8)	2.7 (2)
C(6)	0.1881 (5)	0.0937 (5)	0.1461 (9)	2.6 (1)
C(7)	0.1050 (5)	0.0548 (6)	0.232 (1)	3.4 (2)
C(8)	0.0775 (6)	0.1134 (6)	0.405 (1)	3.6 (2)
C(9)	0.1318 (6)	0.2063 (6)	0.492 (1)	3.6 (2)
C(10)	0.2141 (5)	0.2400 (5)	0.4028 (9)	2.1 (2)
N(3)	0.3130 (4)	0.3349 (4)	-0.0341 (7)	2.4 (1)
N(4)	0.4956 (4)	0.2484 (4)	-0.0438 (6)	2.3 (1)
C(11)	0.2153 (5)	0.3732 (6)	-0.026 (1)	3.4 (2)
C(12)	0.1851 (6)	0.4577 (6)	-0.113 (1)	3.8 (2)
C(13)	0.2544 (6)	0.5088 (6)	-0.212 (1)	3.8 (2)
C(14)	0.3539 (5)	0.4703 (5)	-0.2230 (8)	3.0 (1)
C(15)	0.3812 (5)	0.3837 (5)	-0.1340 (8)	2.4 (1)
C(16)	0.4857 (5)	0.3342 (5)	-0.1368 (7)	2.5 (1)
C(17)	0.5692 (6)	0.3725 (5)	-0.2232 (8)	3.1 (2)
C(18)	0.6610 (5)	0.3200 (6)	-0.2185 (9)	3.3 (2)
C(19)	0.6713 (6)	0.2315 (6)	-0.123 (1)	3.5 (2)
C(20)	0.5869 (5)	0.1976 (5)	-0.038 (1)	3.2 (2)
Cl(3)	-0.0370 (2)	0.2287 (2)	-0.1148 (3)	4.31 (5)
O(1)	-0.0146 (6)	0.6018 (6)	0.733 (1)	6.7 (2)
O(2)	0.8965 (6)	0.3890 (7)	-0.403 (1)	6.8 (2)

Table 2. Selected distances ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

Rh—Cl(1)	2.341 (2)	Rh—Cl(2)	2.328 (2)
Rh—N(1)	2.022 (5)	Rh—N(2)	2.027 (6)
Rh—N(3)	2.011 (5)	Rh—N(4)	2.037 (5)
N(1)—C(1)	1.330 (8)	N(1)—C(5)	1.354 (8)
N(2)—C(6)	1.333 (8)	N(2)—C(10)	1.347 (8)
N(3)—C(11)	1.360 (8)	N(3)—C(15)	1.361 (8)
N(4)—C(16)	1.345 (8)	N(4)—C(20)	1.350 (8)
N(3)—Rh—N(4)	80.3 (2)	N(2)—Rh—N(4)	177.2 (2)
N(2)—Rh—N(3)	97.8 (2)	N(1)—Rh—N(4)	98.3 (2)
N(1)—Rh—N(3)	93.1 (2)	N(1)—Rh—N(2)	79.7 (2)
Cl(2)—Rh—N(4)	85.7 (2)	Cl(2)—Rh—N(3)	87.6 (2)
Cl(2)—Rh—N(2)	96.3 (2)	Cl(2)—Rh—N(1)	176.0 (2)
Cl(1)—Rh—N(4)	96.8 (2)	Cl(1)—Rh—N(3)	176.8 (2)
Cl(1)—Rh—N(2)	85.2 (2)	Cl(1)—Rh—N(1)	88.6 (2)
Cl(1)—Rh—Cl(2)	90.86 (6)	Rh—N(1)—C(5)	114.0 (4)
Rh—N(1)—C(1)	126.0 (4)	C(1)—N(1)—C(5)	119.8 (5)
Rh—N(2)—C(10)	124.4 (4)	Rh—N(2)—C(6)	115.4 (4)
C(6)—N(2)—C(10)	120.2 (5)	Rh—N(3)—C(15)	115.6 (4)
Rh—N(3)—C(11)	126.0 (4)	C(11)—N(3)—C(15)	118.3 (5)
Rh—N(4)—C(20)	125.3 (4)	Rh—N(4)—C(16)	114.8 (4)
C(16)—N(4)—C(20)	119.6 (5)		
H110...Cl(3) <sup>i</sup>	2.469 (7)	H120...Cl(3) <sup>iii</sup>	2.188 (9)
H210...O(2) <sup>ii</sup>	1.89 (1)	H220...O(1) <sup>iv</sup>	2.32 (1)

Symmetry code: (i)  $-x, -y+1, -z+1$ ; (ii)  $-x+1, -y+1, -z$ ; (iii)  $x+1, y, z$ ; (iv)  $x+1, y, z-1$ .

features of this hydrogen bonding are shown in Table 2.

Obviating the angular distortions owing to the rigidity of the bipyridine ligands, the geometry around the central atom may be regarded as octahedral. The *trans* angles around the Rh atom range from 176.0 (2) to 177.2 (2) $^\circ$ , and the *cis* angles from 79.7 (2) to 98.3 (2) $^\circ$ .

The atoms N(1), N(2) and C(1)–C(10), defining the bipyridine ligand, are planar within  $\pm 0.08$  Å, and the Rh atom lies 0.2 Å from the mean plane. The other bipyridine ligand, defined by the N(3), N(4) and C(11)–C(20) atoms, is planar within  $\pm 0.02$  Å, the Rh atom being 0.2 Å from this plane. The mean planes make a dihedral angle of 79.0 (1)°.

Both the bipyridine-ligand rigidity and the Rh—N equilibrium distance result in the angles Rh—N(1)—C(5), Rh—N(2)—C(6), Rh—N(3)—C(15) and Rh—N(4)—C(16) being smaller than Rh—N(1)—C(1), Rh—N(2)—C(10), Rh—N(3)—C(11) and Rh—N(4)—C(20), as well as in the N—Rh—N angles differing from the theoretical value of 90°.

For the bipyridine ligands, the C—C bond distances range from 1.352 (11) to 1.494 (9) Å, averaging 1.389 (9) Å. The N—C bond distances range from 1.330 (8) to 1.361 (8) Å, averaging 1.347 (8) Å.

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## Structures of [1,3-Bis(diphenylphosphino)propane]tetracarbonylmolybdenum(0) (1) and [1,4-Bis(diphenylphosphino)butane]tetracarbonylmolybdenum(0) (2)

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**Abstract.** (1) C<sub>31</sub>H<sub>26</sub>MoO<sub>4</sub>P<sub>2</sub>, [Mo{(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>PC<sub>3</sub>-H<sub>6</sub>P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>}(CO)<sub>4</sub>], *M<sub>r</sub>* = 620.4, orthorhombic, *Pnma*, *a* = 16.854 (4), *b* = 21.970 (4), *c* = 7.723 (2) Å, *V* = 2860 (1) Å<sup>3</sup>, *Z* = 4, *D<sub>m</sub>* = 1.47 (3), *D<sub>x</sub>* = 1.48 Mg m<sup>-3</sup>, λ(Mo *Kα*) = 0.7093 Å, μ = 0.59 mm<sup>-1</sup>, *F*(000) = 1264, *T* = 298 K, final *R* = 0.033 for 1871 observed reflections. (2) C<sub>32</sub>H<sub>28</sub>MoO<sub>4</sub>P<sub>2</sub>, [Mo{(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>PC<sub>4</sub>H<sub>8</sub>P(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>}(CO)<sub>4</sub>], *M<sub>r</sub>* = 634.3, monoclinic, *P2<sub>1</sub>/n*, *a* =

12.072 (2), *b* = 15.379 (5), *c* = 16.607 (5) Å, β = 104.65 (2)°, *V* = 2983 (1) Å<sup>3</sup>, *Z* = 4, *D<sub>m</sub>* = 1.36 (3), *D<sub>x</sub>* = 1.35 Mg m<sup>-3</sup>, λ(Mo *Kα*) = 0.7093 Å, μ = 0.57 mm<sup>-1</sup>, *F*(000) = 1296, *T* = 298 K, final *R* = 0.032 for 3888 observed reflections. The molecular structures of both compounds are similar, but the numbers of the ring members are six in (1) and seven in (2). In the order of compound (1), (2), the following changes occur: P—Mo—P = 89.74 (4), 91.65 (4)°; *cis* C—Mo—C = 88.7 (2), 86.1 (2)°; *trans* C—Mo—C = 174.8 (3), 176.8 (2)°. The increase in

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