

## Research Article

# Synthesis and Spectroscopic Studies of Some New Molybdenum, Tungsten, and Ruthenium Carbonyl Derivatives of 2-Hydroxymethylpyridine

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Thermal reactions of 2-hydroxymethylpyridine (HMP) with  $[M(CO)_6]$  in air resulted in formation of the oxo-complexes  $[M_2O_6(HMP)_2]$ , where  $M = Mo, 1,$  or  $W, 2$ . The tricarbonyl complex  $[Ru(CO)_3(HMP)]$ , **3**, was obtained from the reaction of  $[Ru_3(CO)_{12}]$  with HMP. In presence of triphenyl phosphine ( $PPh_3$ ), the reaction of HMP with  $Ru_3(CO)_{12}$  gave  $[Ru(CO)_2(HMP)(PPh_3)]$ , **4**. All the complexes were characterized by elemental analysis, mass spectrometry, IR, and NMR spectroscopy. The thermal properties of the complexes were also investigated by thermogravimetry technique.

## 1. Introduction

Hydroxymethylpyridines are valuable intermediates and have promising application, for example, in the synthesis of pharmaceuticals and agrochemicals, such as mefloquine hydrochloride and pinoprofen [1]. Carbonyl derivatives of transition metals, on the other hand, are useful intermediates in the synthesis of important coordination compounds [2–5] and have applications in catalysis of important reactions such as epoxidation, carbonylation, hydrogenation, and hydroformylation reactions [6–9]. For example, the complex  $[Rh(CO)_2Cl(HMP)]$  was synthesized and characterized. It showed higher efficiency as catalyst for carbonylation of methanol to yield acetic acid and methyl acetate. It proved to be much better than the industrially used species  $[Rh(CO)_2I_2]^-$  [10].

Molybdenum complexes also catalyze nitrogen fixation in plants by some microorganisms [11]. Many metal carbonyl derivatives are used in photochemical, photochromic, and thermochromic processes [12]. Pyridine derivatives have biological and pharmaceutical importance [13, 14]. Oxidation-reduction of pyridine derivatives plays important roles in biological activity [15]. These derivatives are char-

acterized by their antiviral, antifungal, antioxidant, antithyroid, and diuretic action [16]. Some pyridine complexes act as active catalysts for olefin polymerization [17].

Recently we have reported the reactions of  $[Mo(CO)_6]$  and  $[Ru_3(CO)_{12}]$  with 1-(2-pyridylazo)-2-naphthol (PAN) [18]. Several interesting structural features were indicated in those derivatives. Our interest in investigating the reactions of transition metal carbonyls with various pyridine derivatives has prompted us to investigate the reactions of 2-hydroxymethylpyridine, Scheme 1, with some metal carbonyls. The reactions were carried out in air with either HMP alone or in presence of triphenyl phosphine ( $PPh_3$ ).

## 2. Experimental

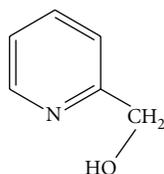
**2.1. Reagents.**  $[Mo(CO)_6]$  and  $[Ru_3(CO)_{12}]$ , HMP, and  $PPh_3$  were supplied by Fluka. All chemicals were of analytical reagent grade and were used without purification. All solvents were purified by distillation prior to their use.

**2.2. Instrumentation.** Infrared measurements (KBr pellets) were carried out on a Unicam-Mattson 1000 FT-IR spectrometer. Electronic absorption spectra were measured on a

TABLE 1: Elemental analysis and mass spectrometry data for the complexes.

Complex		C% Found (Calcd.)	H% Found (Calcd.)	N% Found (Calcd.)	Mass spectrometry	
Molecular formula	Chemical formula				Molecular weight	$m/z^a$
$\text{Mo}_2\text{O}_6(\text{HMP})_2$	$[\text{Mo}_2(\text{C}_{12}\text{H}_{14}\text{O}_8\text{N}_2)]$	28.2 (28.5)	2.6 (2.8)	5.7 (5.5)	506.14	507 $[\text{P}^+]$
$\text{W}_2\text{O}_6(\text{HMP})_2$	$[\text{W}_2\text{O}_6(\text{HMP})_2]$	21.5 (21.2)	1.9 (2.1)	4.3 (4.1)	681.96	682 $[\text{P}^+]$
$\text{Ru}(\text{CO})_3(\text{HMP})$	$[\text{Ru}(\text{C}_9\text{H}_7\text{O}_4\text{N})]$	36.5 (36.8)	2.2 (2.4)	4.6 (4.8)	294.23	239 $[\text{P}-2\text{CO}]^+$
$\text{Ru}(\text{CO})_2(\text{HMP})(\text{PPh}_3)$	$[\text{Ru}(\text{C}_{26}\text{H}_{22}\text{O}_3\text{NP})]$	58.8 (59.1)	4.0 (4.2)	2.9 (2.7)	528.47	501 $[\text{P}-\text{CO}]^+$

<sup>a</sup> $m/z$ : mass over charge ratio in the mass spectrum.



SCHEME 1: 2-hydroxymethylpyridine (HMP).

Unicam UV2-300 spectrometer. Nuclear magnetic resonance measurements ( $^1\text{H}$  NMR) were performed on a Spectrospin-Bruker AC 200 MHz spectrometer. Samples were dissolved in deuterated dimethylsulphoxide (DMSO) using tetramethylsilane (TMS) as internal reference. Magnetic measurements of the complexes in the solid state (Gouy method) were recorded on a Sherwood magnetic susceptibility balance. Elemental analyses for carbon, hydrogen, and nitrogen (CHN) were performed on a Perkin-Elmer 2400 CHN elemental analyzer. Mass spectrometry measurements of the solid complexes (70 eV, EI) were carried out on a Finnigan MAT SSQ 7000 spectrometer. Thermogravimetric (TG) analyses were carried out under nitrogen atmosphere with a heating rate of  $10^\circ\text{C min}^{-1}$  using a Shimadzu DT-50 thermal analyzer. Table 1 gives the elemental analyses and mass spectrometry data of the complexes.

**2.3. Synthesis of  $[\text{Mo}_2\text{O}_6(\text{HMP})_2]$  Complex (1).**  $[\text{Mo}(\text{CO})_6]$  (0.10 g, 0.38 mmol) and HMP (0.04 g, 0.38 mmol) were mixed in tetrahydrofuran (THF) (ca.  $30\text{ cm}^3$ ) and then heated to reflux in air for 4 hours; a brown solution was formed. The reaction mixture was cooled and the solvent was evaporated under vacuum line. The solid residue was washed several times with boiling petroleum ether (60–80), recrystallized from hot THF to yield fine brown crystals and then dried under vacuum for several hours (yield 75%).

**2.4. Synthesis of  $[\text{W}_2\text{O}_6(\text{HMP})_2]$  Complex (2).** Similar procedure was employed as used for the preparation of  $[\text{Mo}_2\text{O}_6(\text{HMP})_2]$  complex (reaction period: 10 hours; fine brown crystals; yield 80%).

**2.5. Synthesis of  $[\text{Ru}(\text{CO})_3(\text{HMP})]$  Complex (3).**  $[\text{Ru}_3(\text{CO})_{12}]$  (0.032 g, 0.05 mmol), and HMP (0.016 g, 0.15 mmol) were mixed in THF (ca.  $30\text{ cm}^3$ ) and heated to reflux in air for 5

hours. After complete reaction, a brown solid was collected by filtration. The solid was washed several times with hot petroleum ether (60–80) and then recrystallized from hot ethanol giving fine brown crystals (yield 80%).

**2.6. Synthesis of  $[\text{Ru}(\text{CO})_2(\text{HMP})(\text{PPh}_3)]$  Complex (4).** A mixture of  $[\text{Ru}_3(\text{CO})_{12}]$  (0.032 g, 0.05 mmol), HMP (0.016 g, 0.15 mmol) and  $\text{PPh}_3$  (0.04 g, 0.15 mmol) in THF (ca.  $30\text{ cm}^3$ ) was heated to reflux in air for 4 hours. A reddish-brown solid was obtained, separated by filtration and then recrystallized from hot ethanol to give brownish crystals (yield 60%).

### 3. Results and Discussion

**3.1. IR and NMR Studies.** Interaction of  $[\text{M}(\text{CO})_6]$ ,  $\text{M} = \text{Mo}$  and  $\text{W}$ , with 2-hydroxymethylpyridine (HMP) in THF in air gave the oxo complexes  $[\text{Mo}_2\text{O}_6(\text{HMP})_2]$ , **1**, and  $[\text{W}_2\text{O}_6(\text{HMP})_2]$ , **2**, respectively. The IR spectrum of HMP displayed characteristic stretching ( $\nu$ ) and bending ( $\delta$ ) bands due to its functional groups;  $\nu(\text{OH})$ ,  $\delta(\text{OH})$ ,  $\nu(\text{C}-\text{O})$ ,  $\nu(\text{C}=\text{N})$ , and  $\delta(\text{Py})$ , Table 2, [19–23]. The IR spectrum of the molybdenum complex, **1**, exhibited the ligand characteristic bands with the appropriate shifts due to complex formation, Table 2. The IR spectrum showed shifts in the  $\nu(\text{OH})$ ,  $\delta(\text{OH})$ , and  $\nu(\text{C}-\text{O})$  frequencies of the free ligand indicating the coordination of HMP to molybdenum through its oxygen atom of hydroxyl group. The magnetic studies showed that the oxo complex  $[\text{Mo}_2\text{O}_6(\text{HMP})_2]$ , **1**, is diamagnetic. So, the  $^1\text{H}$  NMR spectrum of **1** was performed and exhibited signals due to the ligand moieties, Table 3. The presence of the OH group of ligand in **1** was also confirmed by  $^1\text{H}$  NMR spectroscopy, Table 3. The higher shifts in the  $\nu(\text{OH})$  and  $\delta(\text{OH})$  on complexation may indicate the presence of intra- and intermolecular hydrogen bonding [19]. Complex **1** also showed that the in-plane ring deformation bands,  $\delta(\text{Py})$ , shifted to lower frequency. The shift of stretching frequency of the  $\text{C}=\text{N}$  and  $\delta(\text{Py})$  of the pyridyl moiety indicated that pyridyl nitrogen is involved in coordination as well, Table 2 [20]. Furthermore, the IR spectrum of the complex displayed nonligand bands at  $954\text{ cm}^{-1}$  and  $880\text{ cm}^{-1}$  which were assigned owing to asymmetric and symmetric stretching frequencies of  $\text{Mo}=\text{O}$  bonds in a *cis*  $\text{MoO}_2$  fragment of a dimeric structure having the core  $\text{Mo}_2\text{O}_6$  [19, 21]. In addition, the IR spectrum of the complex exhibited two

TABLE 2: Important IR data for HMP and its molybdenum, tungsten, and ruthenium complexes.

Compound	IR data (cm <sup>-1</sup> ) <sup>a</sup>							
	$\nu(\text{OH})$	$\nu(\text{C}=\text{O})$	$\nu(\text{C}=\text{N})$ (Py)	$\nu(\text{N}=\text{N})$	$\delta(\text{OH})$	$\nu(\text{C}-\text{O})$	$\delta(\text{Py})$	$\nu(\text{C}-\text{H})$
HMP	3249 (s)	—	1597 (s)	1438 (s)	1364 (m)	1222 (m)	633 (m)	—
	—	—	—	—	—	—	—	1430 (m)
PPh <sub>3</sub>	—	—	—	—	—	—	—	745 (s)
	—	—	—	—	—	—	—	694 (s)
	—	—	—	—	—	—	—	493 (s)
[Mo <sub>2</sub> O <sub>6</sub> (HMP) <sub>2</sub> ] <sup>b</sup>	3445 (s)	—	1609 (s)	1478 (m)	1401 (m)	1288 (m)	651 (m)	—
[W <sub>2</sub> O <sub>6</sub> (HMP) <sub>2</sub> ] <sup>c</sup>	3424 (s)	—	1613 (s)	1441 (m)	1400 (m)	1286 (m)	655 (m)	—
[Ru(CO) <sub>3</sub> (HMP)] <sup>d</sup>	3423 (s)	2040 (m) 1969 (s) 1923 (s)	1650 (m)	1458 (m)	1432 (m)	1282 (m)	672 (m)	—
[Ru(CO) <sub>2</sub> (HMP)(PPh <sub>3</sub> )] <sup>e</sup>	3422 (s)	1972 (s) 1896 (s)	1651 (s)	1433 (m)	1478 (m)	1186 (m)	695 (s)	748 (m) 722 (m)
	—	—	—	—	—	—	—	517 (s)

<sup>a</sup>s: strong; m: medium.

<sup>b</sup>The  $\nu(\text{Mo}-\text{N})$ : 468 cm<sup>-1</sup> (m);  $\nu(\text{Mo}-\text{O})$ : 540 cm<sup>-1</sup> (m);  $\nu(\text{Mo}=\text{O})$ : 954 cm<sup>-1</sup> (s) and 880 cm<sup>-1</sup> (m);  $\nu(\text{Mo}-\text{O}-\text{Mo})$ : 800 (m) and 651 cm<sup>-1</sup> (m).

<sup>c</sup>The  $\nu(\text{W}-\text{N})$ : 534 cm<sup>-1</sup> (m);  $\nu(\text{W}-\text{O})$ : 565 cm<sup>-1</sup> (m);  $\nu(\text{W}=\text{O})$ : 944 cm<sup>-1</sup> (s) and 865 cm<sup>-1</sup> (s);  $\nu(\text{W}-\text{O}-\text{W})$ : 803 (s) and 655 cm<sup>-1</sup> (m).

<sup>d</sup>The  $\nu(\text{Ru}-\text{N})$ : 422 cm<sup>-1</sup> (m);  $\nu(\text{Ru}-\text{O})$ : 558 cm<sup>-1</sup> (m).

<sup>e</sup>The  $\nu(\text{Ru}-\text{N})$ : 459 cm<sup>-1</sup> (m);  $\nu(\text{Ru}-\text{O})$ : 572 cm<sup>-1</sup> (m).

TABLE 3: <sup>1</sup>H NMR data of HMP and its molybdenum, tungsten, and ruthenium complexes.

Compound	<sup>1</sup> H NMR data (ppm)
HMP	8.47 (s, 1H, OH), 7.79–7.73, 7.50–7.47, 7.22–7.20 (m, 4H, py), 5.44 (s, 2H, CH <sub>2</sub> )
[Mo <sub>2</sub> O <sub>6</sub> (HMP) <sub>2</sub> ]	9.99 (s, 2H, 2OH), 8.41–8.39, 8.05–7.89, 7.60–7.36 (m, 8H, 2py), 5.59 (s, 4H, 2CH <sub>2</sub> )
[W <sub>2</sub> O <sub>6</sub> (HMP) <sub>2</sub> ]	9.99 (s, 2H, 2OH), 8.71–8.44, 8.10–7.68, 7.67–6.11 (m, 8H, 2py), 4.68 (s, 4H, 2CH <sub>2</sub> )
[Ru(CO) <sub>3</sub> (HMP)]	8.75 (s, 1H, OH), 8.13–8.04, 7.89–7.76, 7.56–7.28 (m, 4H, py), 5.18 (s, CH <sub>2</sub> )
[Ru(CO) <sub>2</sub> (HMP)(PPh <sub>3</sub> )]	8.79 (s, 1H, OH), 7.78–7.22 (m, 19H, py & Ph), 4.53(s, 2H, CH <sub>2</sub> )

stretching vibrational bands at 800 and 651 cm<sup>-1</sup> due to two  $\nu(\text{Mo}-\text{O}-\text{Mo})$  frequencies [19].

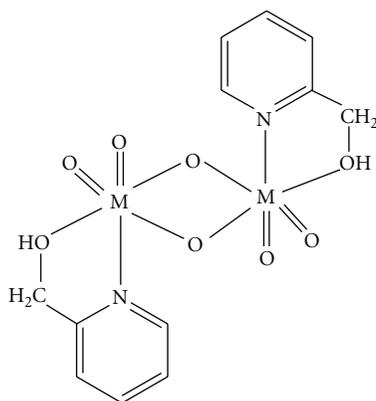
In the far IR spectrum of **1**, nonligand bands at 540 cm<sup>-1</sup> and 468 cm<sup>-1</sup> due to Mo–O and Mo–N bonds provided further evidence for bonding of pyridyl nitrogen and hydroxyl oxygen to molybdenum [20, 24, 25]. Scheme 2 gives the proposed structure of the complex. According to the proposed structure, molybdenum may have +6 formal oxidation state with *d*<sup>0</sup> electronic configuration. Similarly, the reaction of [W(CO)<sub>6</sub>] with HMP in THF in air resulted in the formation of the brown oxo complex [W<sub>2</sub>O<sub>6</sub>(HMP)<sub>2</sub>], **2**. Spectroscopic studies of the complex revealed identical trend as observed for [Mo<sub>2</sub>O<sub>6</sub>(HMP)<sub>2</sub>] complex, Tables 2 and 3. Therefore, identical structure was proposed for both Mo and W complexes (Scheme 2).

Thermal reaction of a mixture of [Ru<sub>3</sub>(CO)<sub>12</sub>] and HMP produced a brown complex with a molecular formula of [Ru(CO)<sub>3</sub>(HMP)], **3**. The IR spectrum of **3** displayed

the ligand bands with the corresponding shifts due to complex formation, Table 2. In addition, three stretching bands (one medium and two strong) in the terminal metal carbonyl range were observed at 2040, 1969, and 1923 cm<sup>-1</sup> due to three terminal M–C≡O bonds. The number and pattern of these bands suggested the presence of two axial and one equatorial CO groups in a trigonal bipyramidal structure as shown in Scheme 3 [18, 21]. The IR spectrum of the ruthenium complex also displayed nonligand bands at 422 cm<sup>-1</sup> and 558 cm<sup>-1</sup> due to Ru–N and Ru–O bonds, respectively. In addition, the IR spectrum showed a strong and broad band at 3423 cm<sup>-1</sup> due to stretching frequency of the OH group, and a band at 1432 cm<sup>-1</sup> due to bending OH frequency. Magnetic measurements of the complex indicated its diamagnetic characteristics. The <sup>1</sup>H NMR spectrum of **3** displayed a signal at 8.75 ppm indicating the presence of the OH group. The signal showed lower field shift and indicated the coordination of HMP ligand to the ruthenium atom

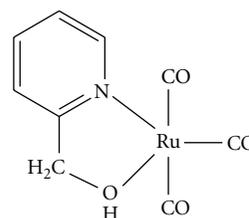
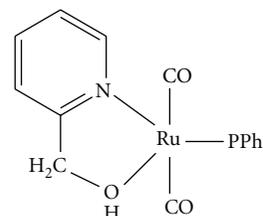
TABLE 4: Thermal analysis data for HMP and its molybdenum, tungsten, and ruthenium complexes.

Complex	Decomposition step, K	% Weight loss	Mol. wt.	Species eliminated	% Solid residue
[Mo <sub>2</sub> (C <sub>12</sub> H <sub>14</sub> O <sub>8</sub> N <sub>2</sub> )]	490–620	21.76	110.14	C <sub>6</sub> H <sub>6</sub> + O <sub>2</sub>	2MoO 44.26%
	620–760	22.15	112.11	C <sub>6</sub> H <sub>8</sub> + O <sub>2</sub>	
	760–900	11.86	60.03	2NO	
[W <sub>2</sub> (C <sub>12</sub> H <sub>14</sub> O <sub>8</sub> N <sub>2</sub> )]	480–650	12.48	85.11	C <sub>4</sub> H <sub>5</sub> + O <sub>2</sub>	2WO 58.61%
	650–800	12.62	86.06	C <sub>4</sub> H <sub>6</sub> + O <sub>2</sub>	
	800–1000	16.29	111.09	C <sub>4</sub> H <sub>3</sub> + 2NO	
[Ru(C <sub>9</sub> H <sub>7</sub> O <sub>4</sub> N)]	341–500	19.04	56.02	2CO	RuO 39.76%
	500–734	41.20	121.22	C <sub>7</sub> H <sub>7</sub> + NO	
[Ru(C <sub>26</sub> H <sub>22</sub> O <sub>3</sub> NP)]	341–467	10.60	56.02	2CO	RuO 22.14%
	467–730	27.67	146.23	C <sub>11</sub> H <sub>14</sub>	
	730–1200	39.58	209.17	C <sub>13</sub> H <sub>8</sub> NP	

SCHEME 2: The proposed structure of [M<sub>2</sub>O<sub>6</sub>(HMP)<sub>2</sub>] [M = Mo, 1 or W, 2].

through the OH group. Accordingly, the complex may have zero formal oxidation state with  $d^8$  electronic configuration as shown in Scheme 3.

Reaction of [Ru<sub>3</sub>(CO)<sub>12</sub>] with HMP in presence of PPh<sub>3</sub> resulted in the formation of [Ru(CO)<sub>2</sub>(HMP)(PPh<sub>3</sub>)], **4**. The mass spectrum of **4** gave a molecular ion peak at  $m/z = 501$  corresponding to [P–CO]<sup>+</sup>, Table 1. The IR spectrum of the complex displayed the HMP and PPh<sub>3</sub> characteristic bands with the appropriate shifts indicating the formation of a mixed ligands complex, Table 2. Also, the IR spectrum of **4** displayed two bands at 1972 and 1896 cm<sup>-1</sup> in the terminal metal carbonyl region due to the presence of two terminal CO groups. In addition, the IR spectrum of the complex showed one stretching and one bending frequency due to the OH group at 3422 and 1478 cm<sup>-1</sup>, respectively. The shifts in OH bands with respect to that of ligand suggested that the OH group was involved in coordination. The magnetic studies showed that the complex is diamagnetic. The presence of the coordinated OH moiety was confirmed by <sup>1</sup>H NMR studies, Table 3. The shifts observed for  $\delta(\text{Py})$  and  $\nu(\text{C}=\text{N})$  in the IR spectrum of **4** on complexation indicated the binding of HMP to ruthenium via the pyridyl

SCHEME 3: The proposed structure of [Ru(CO)<sub>3</sub>(HMP)], **3**.SCHEME 4: The proposed structure of [Ru(CO)<sub>2</sub>(HMP)(PPh<sub>3</sub>)], **4**.

nitrogen, Table 2. In the far IR spectrum of **4**, nonligand bands were observed at 459 and 572 cm<sup>-1</sup> due to (Ru–N) and (Ru–O) bonds [25]. According to the spectroscopic studies, the proposed structure given in Scheme 4 suggested that ruthenium metal may have zero formal oxidation state ( $d^8$  electronic configuration).

**3.2. TG Analysis.** In order to give more insight into the structure of the complexes, thermal studies on the solid complexes using TG and differential thermogravimetric (DTG) techniques were performed [26]. The TG and DTG plots of Mo<sub>2</sub>O<sub>6</sub>(HMP)<sub>2</sub>[Mo<sub>2</sub>(C<sub>12</sub>H<sub>14</sub>O<sub>8</sub>N<sub>2</sub>); M. wt. = 506.14] exhibited three decomposition steps. The first step occurred from 490–620 K, with a net weight loss of 21.76%, is probably due to elimination of (C<sub>6</sub>H<sub>6</sub> + O<sub>2</sub>) moieties, Table 4. The second step occurred from 620–760 K with a net weight loss of 22.15%, is probably due to elimination of (C<sub>6</sub>H<sub>8</sub> + O<sub>2</sub>). The third step occurred in the temperature range 760–900 K

with a net weight loss of 11.86%, consistent with elimination of 2NO, to give finally 2MoO, Table 4.

The TG and DTG plots of  $W_2O_6(HMP)_2$  [ $W_2(C_{12}H_{14}O_8N_2)$ ]; M. wt. = 681.96] exhibited also three decomposition steps in the temperature range 480–1000 K. The first, second, and third steps occurred in the temperature ranges (480–650), (650–800), and (800–1000) K with a net weight loss of 12.48%, 12.62% and 16.29% corresponding to the elimination of  $(C_4H_5 + O_2)$ ,  $(C_4H_6 + O_2)$ , and  $(C_4H_3 + 2NO)$ , respectively, to give finally 2WO as residual, Table 4.

Thermal studies of the ruthenium complex,  $Ru(CO)_3(HMP)$ , were carried out using thermogravimetry. The TG plot of  $Ru(CO)_3(HMP)$  [ $Ru(C_9H_7O_4N)$ ]; M. wt. = 294.23] showed two well-defined and non-overlapping steps in the 341–734 K range to give finally a solid residual of RuO, Table 4.

A TG plot of  $Ru(CO)_2(HMP)(PPh_3)$  [ $Ru(C_{26}H_{22}O_3NP)$ ]; M. wt. = 528.47] showed three-step decomposition. The first at 341–467 K, with a net weight loss of 10.60% corresponds to elimination of 2CO [26]. The second decomposition peak (476–730 K) showed a net weight loss of 27.67%, Table 4, and the third at 730–1200 K, with a net weight loss of 39.58% to give finally a solid residual of RuO, Table 4.

#### 4. Conclusion

2-Hydroxymethylpyridine is an important substance in biological and pharmaceutical fields. Reactions of molybdenum, tungsten, and ruthenium carbonyls with HMP yielded the two oxo complexes  $[Mo_2O_6(HMP)_2]$  and  $[W_2O_6(HMP)_2]$  and the tricarbonyl complex  $[Ru(CO)_3(HMP)]$ . In presence of  $PPh_3$  the reaction gave the dicarbonyl complex  $[Ru(CO)_2(HMP)(PPh_3)]$ .

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