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The reaction of tetrahydridododecacarbonyltetrarhenium(0) with pyridine and some nitrogen-containing bases

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Abstract

The reactions of $H_4Re_4(CO)_{12}$ with some nitrogen-containing bases L in chloroform at room temperature were investigated. For L = pyridine, γ-picoline and piperidine, two types of products, namely $[H_4Re_3(CO)_9L]^-[LH]^+$ and $H_3Re_3(CO)_9L_3$, were isolated. When L = α-picoline, 4-CN-pyridine, imidazole or 2,4-dimethylimidazole, $[H_4Re_3(CO)_9L]^-[LH]^+$ is the only product with a yield close to 75%. The structure of $H_3Re_3(CO)_9L_3$ (L = pyridine) (**1**) and $[H_4Re_3(CO)_9L]^-[LH]^+$ (L = α-picoline) (**3**) were determined by the X-ray diffraction method. For **1**, the three rhenium atoms form an equilateral triangle with edge-bridging hydrides. Each rhenium is coordinated by three carbonyl groups and an axial pyridine. The three pyridines are not all on the same side of the triangular plane to reduce the steric repulsion which is obvious from bond angles and distances data. In **3**, the three rhenium atoms form an isosceles with two hydride-bridged Re–Re single bonds and an Re–Re double bond bridged by two hydrides. Each rhenium is coordinated by three CO groups, and α-picoline is coordinated to rhenium in the axial position.

Keywords: Rhenium; Pyridine; Cluster; Imidazole

1. Introduction

$H_4Re_4(CO)_{12}$ [1–5] is a 56-electron unsaturated rhenium cluster with delocalized double bonds and face-bridging hydrides. The other known unsaturated cluster [6,7], $H_4Re_4(CO)_{12}$ is very reactive. It can react with common solvents, such as acetone, dimethylformamide (DMF), ethylacetate and alcohol. Recently, we showed that it can react with tripod ligand $HC(PPh_2)_3$ to form four tripod adducts [5]: $HRe(CO)_3(HC(PPh_2)_3)$, $H_2Re_2(CO)_6(HC(PPh_2)_3)$, $H_3Re_3(CO)_9(HC(PPh_2)_3)$ and $H_4Re_4(CO)_{12}(HC(PPh_2)_3)$. It seems that $H_4Re_4(CO)_{12}$ disintegrates in units of $HRe(CO)_3$. When $H_4Re_4(CO)_{12}$ reacts with methanol, we demonstrated that the reaction follows a clean fifth-order rate law to form an reactive species [8]. This species can further be transformed quantitatively into $H_3Re_3(CO)_9(MeOH)$ with a μ_3-OMe group.

In our continuing effort to understand the chemistry of $H_4Re_4(CO)_{12}$, the reactions with pyridine and its derivatives are investigated. There are only two reported rhenium cluster compounds containing pyridine and its derivatives. From the reaction of $H_3Re_3(CO)_{10}^{2-}$ with I_2–pyridine, $H_3Re_3(CO)_{10}(Py)_2$ can be obtained [9]. The reaction between $H_4Re_3(CO)_{10}^-$ and acetonitrile, either thermally or with the assistance of trimethylamine oxide, followed by addition of pyridine yielded $H_4Re_3(CO)_9Py^-$ [10]. Here we report that both $H_3Re_3(CO)_9(Py)_3$ and $H_4Re_3(CO)_9Py^-$ can be obtained in the reaction between $H_4Re_4(CO)_{12}$ and pyridine. When some other nitrogen-containing bases were used, the relative yields of the two products depend on the nature of base.

2. Experimental details

2.1. General method

$Re_2(CO)_{10}$ was purchased from Strem Chemical Co. Pyridine; α-picoline, γ-picoline, 4-cyanopyridine, 4-pyridinecarboxylic acid, piperidine, imidazole and 2,4-dimethylimidazole were purchased from Aldrich Chem.

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Table 1
Spectroscopic data of products **1** ($H_3Re_3(CO)_9(Py)_3$), **2** ($[H_4Re_3(CO)_9Py]^-[PyH]^+$) and **3** ($[H_4Re_3(CO)_9Pic]^-[PicH]^+$)

	1	**2**	**3**
^{1}H NMR ($CDCl_3$) [a], δ (ppm)	−9.13 (2H, s), −8.77 (1H, s), 7.06 (4H, t, 6.4 Hz), 7.30 (2H, t, 6.4 Hz), 7.55 (2H, t, 7.6 Hz), 7.72 (1H, t, 7.8 Hz), 8.83 (4H, d, 5.2 Hz), 8.94 (2H, d, 5.2 Hz)	−9.21 (2H, s), −8.73 (1H, s) −7.77 (1H, s), 7.11 (2H, tt, 7.52; 0.92 Hz), 7.60 (4H, m), 8.07 (1H, tt, 7.6; 1.4 Hz), 8.38 (2H, dt, 4.88; 1.52 Hz), 8.76 (2H, dt, 5.08; 1.32 Hz),	−9.43 (2H, s), −8.77 (1H, s), −8.28 (1H, s), 2.91 (3H, s), 3.03 (3H, s), 7.03 (1H, t, 6.0 Hz), 7.38 (1H, d, 8.4 Hz), 7.72 (1H, td, 7.2; 1.6 Hz), 7.96 (1H, t, 4.2 Hz), 7.99 (1H, s), 8.03 (1H, d, 8.0 Hz), 8.86 (1H, td, 8.0; 1.6 Hz), 8.93 (1H, d, 5.6 Hz), 9.23 (1H, d, 6.0 Hz)
^{13}C NMR (d_6-acetone); δ (ppm)	125.0, 125.7, 137.0, 137.4, 157.0, 157.1, 191.1, 192.0, 192.1, 192.6, 193.4	125.8, 125.9, 138.1, 138.2, 157.5, 157.6, 189.7, 193.2, 194.4, 196.8, 198.3	
IR ($CHCl_3$), ν(CO) (cm^{-1})	2036.5(m), 2014.1(s), 2009.6(s, sh), 1933.8(s, br), 1919.8(s, br)	2033.0(m), 2003.8(s), 1996.5(s, sh), 1914.0(s, br)	
Mass spectroscopy, (m/e)	1053(M^+, ^{187}Re)	974(M^+-2H, ^{187}Re)	

[a] For **3**, d_6-acetone was used because of solubility limitation.

Table 2
Summary of crystal data and intensity collection conditions for single crystals of **1** and **3**

Formula	$Re_3C_{24}H_{18}N_3O_9$ (**1**)	$Re_3C_{21}H_{19}N_2O_9$ (**3**)
Crystal size (mm)	0.73 × 0.27 × 0.20	0.60 × 0.52 × 0.30
Crystal system	Monoclinic	Orthorhombic
Space group	$P2_1/c$	*Ibam*
a (Å)	12.966(3)	16.006(7)
b (Å)	13.517(3)	23.357(7)
c (Å)	16.640(2)	14.969(6)
β (°)	95.71(2)	
V (Å^3)	2902(5)	5596(4)
Z	4	8
d_{calc} (g cm^{-3})	2.406	2.378
Scan speed (° min^{-1})	2.93–14.65	2.93–14.65
Absorption coefficient (cm^{-1})	127.06	131.7
Scan range (°)	$2.5 < 2\theta < 50$	$2.0 < 2\theta < 50$
Number of reflections	5789 (3970 $I > 3\sigma(I)$)	3289 (1755 $I > 3\sigma(I)$)
Number of independent reflections	5001 (3542 $I > 3\sigma(I)$)	2581 (1678 $I > 3\sigma(I)$)
R (%)	4.54	5.62
R_w (%)	4.78	6.14
Goodness of fit	0.941	1.81
Data-to-parameter ratio	10.0 to 1	18.6 to 1

Co. These chemicals were used directly without further purification. Chloroform was purchased from Merck Chemical Co., dried over P_2O_5 and distilled just before use. $H_4Re_4(CO)_{12}$ was prepared by the reaction between $Re_2(CO)_{10}$ and H_2 in refluxing decane according to the method in the literature [5].

^1H and ^{13}C NMR spectra were obtained on a Bruker AM-400 or Varian Gemini-300 spectrometers. All chemical shifts were reported vs. tetramethylsilane for both ^1H and ^{13}C spectra. IR spectra were obtained on a Bomen Michelson series 100 Fourier transform spectrometer. Mass spectra were recorded on a JEOL JMX-HX-110 mass spectrometer.

2.2. Reaction between $H_4Re_4(CO)_{12}$ and pyridine

A 10 ml $CHCl_3$ solution of pyridine (24 μl, 0.30 mmol) was added dropwise to a 10 ml $CHCl_3$ solution of $H_4Re_4(CO)_{12}$ (80 mg, 0.07 mmol) at room temperature. The color of the solution turned from red to yellow within a few minutes. On separation of the resulting mixture by silica gel preparative thin layer chromatography (TLC) using chloroform as eluent, two yellow products **1** (52 mg) and **2** (37 mg) were obtained at $R_f = 0.7$ and 0.05 respectively. The spectroscopic data of the two products are listed in Table 1. Single crystals of **1** were obtained from chloroform solution by the slow evaporation method. However, we were unable to obtain a single crystal of **2** by the same method.

2.3. Reaction of $H_4Re_4(CO)_{12}$ with other nitrogen bases

When the reaction was carried out under identical reaction conditions using α-picoline instead of pyridine, a single product **3** (70 mg, 72 mmol) was obtained. The ^1H NMR data of **3** are included in Table 1.

Table 3
Selected bond distances (Å) and bond angles (°) of $H_3Re_3(CO)_9(Py)_3$ (**1**)

Bond distances					
Re(1)–Re(2)	3.257(1)	Re(1)–Re(3)	3.311(1)	Re(2)–Re(3)	3.298(1)
Re(1)–N(1)	2.216(11)	Re(2)–N(2)	2.205(11)	Re(3)–N(3)	2.235(11)
Re(1)–C(1)	1.909(15)	Re(1)–C(2)	1.907(18)	Re(1)–C(3)	1.893(17)
Re(2)–C(4)	1.896(15)	Re(3)–C(7)	1.913(15)	C(1)–O(1)	1.148(17)
C(2)–O(2)	1.161(19)	C(3)–O(3)	1.153(18)		
Bond angles					
Re(3)–Re(1)–Re(2)	60.3(1)	Re(1)–Re(2)–Re(3)	60.7(1)	Re(2)–Re(3)–Re(1)	59.0(1)
Re(2)–Re(1)–N(1)	87.2(3)	Re(3)–Re(1)–N(1)	100.6(3)	Re(1)–Re(2)–N(2)	91.5(3)
Re(3)–Re(2)–N(2)	91.3(3)	Re(1)–Re(3)–N(3)	104.1(3)	Re(2)–Re(3)–N(3)	84.8(3)
N(1)–Re(1)–C(1)	175.4(5)	N(1)–Re(1)–C(2)	92.5(5)	N(1)–Re(1)–C(3)	87.8(6)
Re(2)–Re(1)–C(1)	94.6(5)	Re(2)–Re(1)–C(2)	160.7(4)	Re(2)–Re(1)–C(3)	106.4(4)
Re(3)–Re(1)–C(1)	83.9(5)	Re(3)–Re(1)–C(2)	100.9(5)	Re(3)–Re(1)–C(3)	163.4(5)
C(1)–Re(1)–C(2)	87.2(7)	C(1)–Re(1)–C(3)	87.6(7)	C(2)–Re(1)–C(3)	92.8(6)
Re(1)–C(1)–O(1)	173.7(14)	Re(1)–C(2)–O(2)	177.2(14)	Re(1)–C(3)–O(3)	176.7(14)
Re(2)–C(4)–O(4)	178.5(12)	Re(3)–C(7)–O(7)	169.9(17)		

Single crystals of **3** can be grown from a chloroform solution by the slow evaporation method. The reaction between $H_4Re_4(CO)_{12}$ and other nitrogen bases, including γ-picoline, 4-cyanopyridine, piperidine, imidazole, 2,4-dimethylimidazole and 4-pyridinecarboxylic acid, were carried out in the same fashion.

2.4. X-ray crystallography

Single crystals of **1** and **3** were mounted in glass capillaries. Intensity data were collected on a Siemens R3m/V diffractometer using Mo Kα radiation (0.7107 Å) monochromated from a highly oriented graphite crystal. Three standard reflections were measured every 50 reflections. The general data collection conditions and results are summarized in Table 2 for compound **1** and **3**. The numbers of independent data are also reported in Table 2. The heavy-atom positions were determined by the direct method. The non-hydrogen atoms were subsequently located from the Fourier map and then anisotropically refined by the full-matrix least-squares method. The positions of all hydrogen atoms were calculated and refined isotropically. R and R_w values after least-squares refinement are also reported in Table 2. All calculations were performed on a Micro Vax II computer based on the Siemens SHELXTL PLUS program [11].

3. Results and discussion

The reaction between $H_4Re_4(CO)_{12}$ and pyridine yielded two yellow solid products **1** and **2**. The ^1H NMR data, listed in Table 1 indicate that **1** contains three hydrides and three pyridines. The M^+ peak pattern is consistent with the presence of three rhenium atoms in **1**. From the M^+ and the ^1H NMR data, **1** can be unambiguously assigned to be $H_3Re_3(CO)_9(Py)_3$. The ^1H NMR data of **2** indicates that it contains four hydrides and two pyridines. The pattern of highest mass peak ($M^+ - 2H$) indicates that **2** also contains three rhenium atoms. The chemical shifts of hydrides are identical to those in $[H_4Re_3(CO)_9Py]^-[NEt_4]^+$ [10]. Therefore **2** contains a $[H_4Re_3(CO)_9Py]^-$ anion. The cation can be reasonably assigned to be a pyridinium ion. Thus **2** is assigned to be $[H_4Re_3(CO)_9Py]^-[PyH]^+$. In the reaction between $H_4Re_4(CO)_{12}$ and α-picoline, only one product **3** was obtained. ^1H NMR data indicate that there are two α-picoline in **3**. The hydride chemical shifts and their pattern are remarkably similar to those in **2**. Therefore **3** is assigned to be $[H_4Re_3(CO)_9Pic]^-[PicH]^+$(Pic = α-picoline). The presence of a proton signal at 7.99 ppm is indicative of the presence of a proton in the picolinium cation. The assignment of **1** and **3** is further supported by the structures determined from X-ray diffraction method described below.

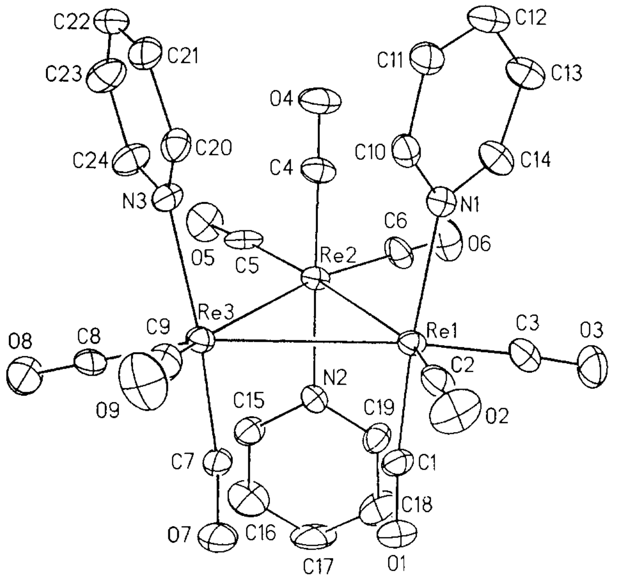

Fig. 1. ORTEP diagram of $H_3Re_3(CO)_9L_3$ (L = pyridine) showing the atomic numbering scheme.

The structure of $H_3Re_3(CO)_9(Py)_3$ is shown in Fig. 1. Selected bond distances and bond angles are collected in Table 3. The three rhenium atoms form a nearly equilateral triangle. Although the Re–Re bond distances (3.257, 3.298 and 3.311Å) are not exactly the same, the angles in the triangle are all close to 60°. Each rhenium has an axially coordinated pyridine. One pyridine is located on the rhenium triangular plane opposite to that for the other two pyridines. This configuration minimizes the steric repulsion between the axial pyridines. The presence of steric interaction between pyridines on the same side of a triangular plane is obvious from considering the bond angles. The Re(3)–Re(1)–N(1)(101°) and Re(1)–Re(3)–N(3)(104°) bond angles, which should reflect the steric interaction, are about 10° larger than the Re(1)–Re(2)–N(2)(91.5°) and Re(3)–Re(2)–N(2)(91.3°) bond angles, which should be the normal bond angles for non-steric interaction. The Re–N distances also show the effect of steric interaction between the axial pyridines. The Re(2)–N(2) distance (2.205 Å) is slightly smaller than those for the sterically crowded pyridines (Re(1)–N(1), 2.216 R; Re(3)–N(3), 2.235 Å). Despite the presence of the steric repulsion, the pyridines still prefer the axial positions. This is in contrast with $H_3Re_3(CO)_9(PPh_3)_3$ [12] which contains equatorial PPh_3. The preference for the axial position is related to the better donating ability of the imino nitrogen in pyridine. The bonding of *trans* carbonyl to rhenium is consequently strengthened expectedly. However, no significant Re–C bond shortening of the *trans* carbonyl groups is detected.

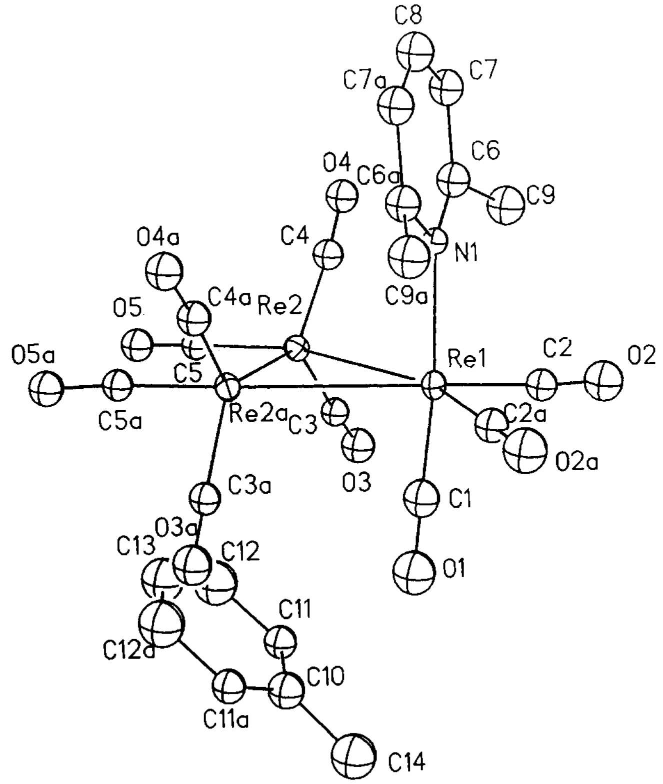

Fig. 2. ORTEP diagram of $[H_4Re_3(CO)_9L]^-[LH]^+$ (L = α-picoline) showing the atomic numbering scheme. Coordinated α-picoline is disordered; the occupancy numbers of C(9) and C(9a) are 0.5. Either C(11) or (C11a) should be a nitrogen atom.

The Re–Re bond distances are much longer than the Re–Re single bond distances in $Re_2(CO)_{10}$ (3.01 Å) [13]. This indicates the presence of edge-bridging hydrides, which were not located directly from X-ray structural determination.

The Re–C bond distances are in the narrow range 1.89–1.92 Å (average, 1.915 Å); C–O bond distances are also in a narrow range 1.13–1.16 Å (average, 1.15 Å). These values are normal for rhenium carbonyl compound. All carbonyl groups are linearly bonded to rhenium atoms. However, C(1)O(1) and C(7)O(7), the carbonyl groups *trans* to the sterically crowded pyridines deviate from linearity significantly (the Re(1)–C(1)–O(1) and Re(3)–C(7)–O(7) bond angles are 173.7° and 169.9° respectively).

The structure of $[H_4Re_3(CO)_9Pic]^-[PicH]^+$ is shown in Fig. 2. Selected bond distances and angles are shown in Table 4. The three rhenium atoms form an isosceles triangle. Disordered α-picoline, where the occupancies of the methyl carbon atoms C(9) and C(9a) are only 0.5, is coordinated to Re(1) at the axial position with the plane of the α-picoline ring normal to the rhenium triangle and parallel to the short Re(2)–Re(2a) edge. This aromatic ring configuration minimizes the possible interaction with the axial carbonyl groups. From the Re–Re bond distances, it is obvious that Re(1)–Re(2) and Re(1)–Re(2a) are single bonds with edge-bridging hydrides. Re(2)–Re(2a) is a double bond with two edge-bridging hydrides. The counter-cation is obviously a picolinium ion. However, we were unable to distinguish N–H from C–H in the picolinium cation by the X-ray structural determination. The structure of the anion is remarkably similar to that of $[H_4Re_3(CO)_9Py]^-$ [10].

In the presence of excess pyridine, **2** converts to **1** only slowly. For example, in a 36 h period, only 3% conversion can be detected by ^1H NMR in chloroform solution at room temperature. This indicates that, under our experimental conditions, **1** and **2** are formed by two different pathways. Besides pyridine and α-picoline, the reaction of some other nitrogen bases L, including γ-picoline, 4-cyanopyridine, 4-pyridinecarboxylic acid, piperidine, imidazole and 2,4-dimethylimidazole, with $H_4Re_4(CO)_{12}$ were also investigated to understand the reactivity pattern. The results are summarized in Table 5. Only 4-pyridinecarboxylic acid does not react with $H_4Re_4(CO)_{12}$. As a probable result of the internal zwitterion formation, the imino nitrogen lost its ability to coordinate to rhenium. Pyridine, γ-picoline and piperidine are the three nitrogen bases, yielding two products of the formula: $H_3Re_3(CO)_9L_3$ and $[H_4Re_3(CO)_9L]^-[LH]^+$. The other four nitrogen bases yield only a single isolable product: $[H_4Re_3(CO)_9L]^-[LH]^+$. These product distributions can be attributed to both the electronic and the steric factors of the nitrogen bases. When the nitrogen bases are good π acceptors, then only $[H_4Re_3(CO)_9L]^-$-

Table 4
Selected bond distances (Å) and bond angles (°) of $[H_4Re_3(CO)_9Pic]^-[PicH]^+$ (**3**)

Bond distances					
Re(1)–Re(2)	3.205(2)	Re(1)–N(1)	2.235(25)	Re(1)–C(1)	1.805(5)
Re(1)–C(2)	1.899(28)	Re(2)–Re(2a)	2.806(2)	Re(2)–C(3)	1.879(25)
Re(2)–C(4)	1.861(26)	Re(2)–C(5)	1.865(28)	C(1)–O(1)	1.221(65)
C(2)–O(2)	1.185(37)	C(4)–O(4)	1.191(33)		
Bond angles					
Re(2)–Re(1)–Re(2a)	51.9(1)	Re(1)–Re(2)–Re(2a)	64.0(1)	Re(2)–Re(1)–N(1)	89.1(6)
Re(2)–Re(1)–C(1)	85.1(12)	Re(2)–Re(1)–C(2)	110.0(8)	C(1)–Re(1)–N(1)	173.6(15)
C(2)–Re(1)–N(1)	95.5(9)	C(2)–Re(1)–C(1)	89.1(12)	C(2)–Re(1)–C(2a)	95.5(9)
Re(1)–Re(2)–C(3)	101.9(8)	Re(1)–Re(2)–C(4)	106.9(8)	Re(1)–Re(2)–C(5)	155.9(8)
Re(2a)–Re(2)–C(3)	133.3(8)	Re(2a)–Re(2)–C(4)	134.3(8)	Re(2a)–Re(2)–C(5)	92.2(8)

Table 5
Yields and ^{1}H NMR chemical shifts of hydrides of the products $H_3Re_3(CO)_9L_3$ and $[H_4Re_3(CO)_9L]^-[LH]^+$, from the reaction between $H_4Re_4(CO)_{12}$ and nitrogen-containing bases L

Base	$H_3Re_3(CO)_9L_3$		$[H_4Re_3(CO)_9L]^-$	
	Yield [a] (%)	δ [b] (ppm)	Yield [a] (%)	δ [b] (ppm)
Pyridine	50	−8.77(1), −9.13(2)	37	−7.77(1), −8.73(1), −9.21(2)
α-picoline	0		73	−8.28(1), −8.77(1), −9.43(2)
γ-picoline	43	−8.74(1), −9.14(2)	38	−8.13(1), −8.89(1), −9.24(2)
4-CN-pyridine	0		74	−7.82(1), −8.89(1), −9.24(2)
4-COOH-pyridine	0		0	
Piperidine	64	−10.72(1), −10.79(2)	30	−8.40(1), 8.93(1), −9.57(2)
Imidazole	0		73	−7.98(1), −8.66(1), −9.85(2)
2,4-Me_2-imidazole	0		73	−7.59(1), −8.43(1), −9.81(2)

[a] Yields are the isolated yields based on rhenium.

[b] The numbers in parentheses are the relative numbers of protons.

$[LH]^+$ is formed. This is probably caused by the ability of the nitrogen base to stabilize the anionic charge on the product. When the nitrogen base is sterically crowded, then it is impossible to form $H_3Re_3(CO)_9L_3$; consequently only a single product is found. Otherwise two products can be formed.

When $[H_4Re_3(CO)_9L]^-[LH]^+$ (L = α-picoline, 4-CN-pyridine, imidazole or 2,4-dimethylimidazole) is the only product, the isolated yields (based on rhenium) are invariably close to 75%. It is possible that, when the starting four-rhenium cluster reacts with the nitrogen base, one rhenium fragment separates from the cluster and its product was not isolated from our TLC separation. Under this circumstance, 75% is the maximum yield for the formation of $[H_4Re_3(CO)_9L]^-[LH]^+$. This deduction is consistent with our data. Taking this into account, then the yields for $H_3Re_3(CO)_9L_3$ (L = pyridine, γ-picoline or piperidine) from $H_4Re_4(CO)_{12}$ are close to 100%. One then reaches the conclusion that Re–Re bond breaking and re-formation must be involved in the formation of trinuclear $H_3Re_3(CO)_9L_3$ from tetronuclear $H_4Re_4(CO)_{12}$. This reasoning is consistent with our proposal that $H_3Re_3(CO)_9L_3$ and $[H_4Re_3(CO)_9L]^-[LH]^+$ are formed by two different pathways.

4. Supplementary material available

Tables of fractional atomic coordinates of all atoms, thermal parameters, complete sets of bond distances and bond angles of **1** and **3** are available from the authors.

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