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Synthesis and crystal structure of $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4$

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Abstract

A tetrameric complex $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4$ (**1**) is formed in high yield when the reaction product of $Re(CO)_5Cl$ with acetic acid in refluxing 1,2-dichlorobenzene is dissolved in THF. The use of $Re_2(CO)_{10}$ instead of rhenium pentacarbonyl chloride also affords **1**. The characterization of $\mathbf{1}\cdot 3THF\cdot 0.5C_6H_{14}$ has been accomplished by X-ray crystallography. The structure of the tetrameric molecule **1** consists of four rhenium(I) atoms bridged by four acetate ligands. The coordination of each metal center is fulfilled by three CO groups and one THF molecule in a way that all carbonyls are *trans* to oxygen atoms. The geometry of each acetate bridge is *anti–syn* providing a Re to Re separation of 5.40 Å. This bonding mode is seen here for the first time in rhenium carboxylates. Different coordination types for $Re–RCO_2$ interaction are also discussed in the paper. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Crystal structures; Rhenium complexes; Tetrameric complexes; Carbonyl complexes; Rhenium carboxylates

1. Introduction

The reaction of $Mo(CO)_6$ with RCO_2H in a suitable high-boiling solvent or even in a mixture of the acid and its anhydride to obtain $Mo_2(RCO_2)_4$ compounds is well-known [1–3]. There are other reactions of this type for example, all three of the hexacarbonyls $M(CO)_6$ (M = Cr, Mo, W) react with 2-hydroxypyridines in refluxing diglime to produce $M_2(6\text{-}X,2\text{-}O\text{-}C_5NH_3)_4$ (X = H, Me, F, Cl, Br) products [4–6], including heteronuclear Mo–W species [7].

In an effort to see if this sort of approach might be extended to the carbonyls of other metals we made an attempt to carry out the following reaction:

$$2Re(CO)_5Cl + 4CH_3CO_2H \rightarrow Re_2(CH_3CO_2)_4Cl_2 + 10CO + 2H_2$$

At least under the conditions used, the reaction that occurred was a different one:

$$Re(CO)_5Cl + CH_3CO_2H + THF \rightarrow 1/4[Re(CO)_3(CH_3CO_2)(THF)]_4 + 2CO + HCl$$

It affords a tetranuclear rhenium(I) complex formulated as $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4$ (**1**) in very high yield.

Herein we report the synthesis and crystal structure of the novel mixed carbonyl–acetate rhenium(I) complex.

2. Experimental

2.1. General procedures

All manipulations were performed under an atmosphere of dinitrogen using standard Schlenk techniques. $Re(CO)_5Cl$ and $Re_2(CO)_{10}$ were purchased from Aldrich, $Mo(CO)_6$ was received from Strem Chemicals. Acetic acid and acetic anhydride (Aldrich) were of a reagent grade and used as received. Other organic solvents were freshly distilled under N_2 from appropriate drying agents prior to use. Elemental analyses were performed by Canadian Microanalytical Services. IR spectra were obtained as KBr pellets on a Perkin-Elmer 16-PC spectrophotometer in the range 4000–400 cm^{-1}.

2.2. Synthesis of $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4\cdot 3THF\cdot 0.5C_6H_{14}$

Conditions similar to those used for the preparation of molybdenum(II) acetate from $Mo(CO)_6$ were employed [1–3]. A mixture of 0.613 g (1.7 mmol) $Re(CO)_5Cl$, 6 ml 1,2-dichlorobenzene, 8 ml acetic acid, 0.8 ml acetic anhydride and 1.2 ml hexanes was stirred at room temperature for 24 h. The temperature was then increased and the solution was refluxed for 2 days. At the beginning of the reflux the colorless solution turned to a pale pink, but did not change any more. After cooling to room temperature, the solvent

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was removed under reduced pressure. The resulting crystalline solid was washed with hexanes (2 × 15 ml), dried under vacuum and then dissolved in 10 ml THF; about 15 ml isomeric hexanes were carefully layered on top. After a few days large colorless crystals of $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4 \cdot 3THF \cdot 0.5C_6H_{14}$ ($\mathbf{1} \cdot 3THF \cdot 0.5C_6H_{14}$) were found on the bottom of the Schlenk tube. Yield: 0.656 g (83%). These crystals were stable in solution, but upon exposure to air they slowly lost the solvent and decomposed. *Anal.* Calc. for **1**, $Re_4C_{36}O_{24}H_{44}$: C, 26.93; H, 2.77. Found: C, 26.48; H, 2.66%. IR data (KBr pellet, ν (cm^{-1})): 3554 (w, br), 2964 (w), 2025 (s), 1923 (s), 1917 (s), 1895 (s), 1878 (s), 1562 (s), 1445 (m), 1416 (m), 1330 (w), 1263 (m), 1094 (s), 1028 (s), 919 (w), 879 (s), 804 (s), 697 (m), 668 (m), 648 (w), 506 (m), 479 (m).

Reaction of $Re_2(CO)_{10}$ (0.500 g, 0.77 mmol) with CH_3CO_2H under exactly the same experimental conditions also afforded complex **1**. Yield: 0.565 g (79%).

When $Re(CO)_5Cl$ and $Mo(CO)_6$ (molar ratio 1:1) were treated together in this reaction, all molybdenum was converted to $Mo_2(CH_3CO_2)_4$, which precipitated. The product containing rhenium remained in solution was not identified.

2.3. X-ray crystallographic procedures

Single crystals of $\mathbf{1} \cdot 3THF \cdot 0.5C_6H_{14}$ were obtained as described above. A colorless acicular crystal with dimensions of 0.45 × 0.15 × 0.10 mm^3 was selected and fixed on the tip of a quartz fiber with grease at −100°C on a CAD-4S diffractometer equipped with monochromated Mo Kα radiation. Detailed procedures have previously been described [8,9]. Indexing based on 25 reflections resulted in a primitive monoclinic cell and the cell parameters were further refined with data in the range $32 < 2\theta < 38°$. The Laue class and lattice dimensions were verified by axial oscillation photographs. The intensity data were gathered by the ω–θ scan technique for $4 < 2\theta < 50°$. No significant decay was found during data collection. An empirical absorption correction based on azimuthal scans of nine reflections with their ψ angles near 90° was applied.

All calculations were performed on a DEC Alpha running VMS. The coordinates of Re atoms were determined by direct methods as programmed in SHELXTL [10]. The positions of the remaining non-hydrogen atoms were located using subsequent sets of least-squares refinement cycles followed by difference Fourier synthesis using the SHELXL-93 structure refinement program [11]. Anisotropic displacement parameters were assigned to all non-hydrogen atoms of the tetrameric molecule **1**. All solvent molecules appeared to be disordered and were refined isotropically. Hydrogen atoms were included in the structure factor calculations at idealized positions in each model and were allowed to ride on the neighboring carbon atoms. Full refinement of 371 parameters and 22 restraints resulted in $R = 0.032$ (for 4839 reflections with $I > 2\sigma(I)$) and $R = 0.040$ (for all 5404 data). The final difference Fourier map showed no significant peaks other than those located in the vicinity of the Re atoms. Crystallographic data and refinement results are summarized in Table 1.

Table 1
Crystallographic data for $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4 \cdot 3THF \cdot 0.5C_6H_{14}$ ($\mathbf{1} \cdot 3THF \cdot 0.5C_6H_{14}$)

Formula	$Re_4O_{27}C_{51}H_{75}$
Formula weight	1864.91
Crystal system	monoclinic
Space group	*P2/n* (No. 13)
a (Å)	15.276(2)
b (Å)	8.608(2)
c (Å)	23.446(3)
β (°)	90.942(9)
V ($Å^3$)	3082.6(9)
Z	2
ρ_{calc} ($g\ cm^{-3}$)	2.009
μ (mm^{-1})	7.910
Radiation (λ, Å)	Mo Kα (0.71073)
Temperature (K)	173
Transmission factors	0.9322–0.9998
$R1^a$, $wR2^b$ [$I > 2\sigma(I)$]	0.0324, 0.0857
$R1^a$, $wR2^b$ (all data)	0.0398, 0.0914
Quality-of-fit^c	1.071

[a] $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$.
[b] $wR2 = [\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]]^{1/2}$.
[c] Quality-of-fit $= [\Sigma[w(F_o^2 - F_c^2)^2]/(N_{obs} - N_{params})]^{1/2}$, based on all data.

3. Results and discussion

The reactions of both $Re(CO)_5Cl$ and $Re_2(CO)_{10}$ with CH_3CO_2H under reflux conditions in dichlorobenzene result in a tetranuclear rhenium(I) complex $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4$ (**1**) in ca. 75–90% yield. While the first reaction goes without change of formal oxidation state of rhenium atom, the second process requires a one-electron oxidation of Re. On the basis of this synthetic work, we are unable to provide a simple explanation of the striking difference between the behavior of rhenium and molybdenum (or tungsten). Probably the reaction temperature was not high enough to complete the substitution of carbonyl groups in $Re(CO)_5Cl$ by acetates. Nevertheless, the results obtained appear to be of interest and we discuss the remarkable structure of complex **1** in detail.

Crystals of $\mathbf{1} \cdot 3THF \cdot 0.5C_6H_{14}$ conform to the monoclinic space group *P2/n* with two molecules of the tetramer $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4$ in the unit cell related by an inversion center. The unit cell also contains six molecules of THF and one of hexane which fill what would otherwise be large interstices in the crystal structure between tetrameric species (Fig. 1). Interestingly, those holes can be occupied by different solvent molecules without change of the major structure. We have also got crystals from a THF/CH_2Cl_2 mixture with cell dimensions pretty close to the ones for crystals obtained from THF/hexanes.

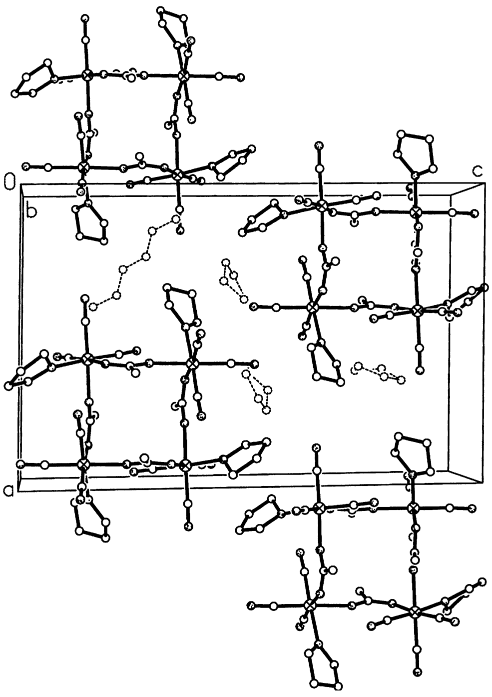


Fig. 1. Packing diagram of $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4\cdot3THF\cdot0.5C_6H_{14}$ (**1**$\cdot3THF\cdot0.5C_6H_{14}$). Hydrogen atoms are not shown. Only the main orientation of disordered groups is displayed.

The central part of the structure is a tetrameric molecule **1** formed by four rhenium(I) centers (two of them are crystallographically independent) connected by bridging acetate groups as shown in Fig. 2. The molecule of **1** itself has $\bar{4}$ (S_4) symmetry but crystallographically has only a two-fold axis (C_2 symmetry). Each Re atom in the tetramer possesses the same coordination: it is surrounded by three oxygen atoms, one provided by a coordinated THF molecule, while two of them belong to the $[CH_3CO_2]^-$ bridges. In addition, three terminal carbonyl groups complete the 'octahedral' coordination sphere of each rhenium center in such a way that all CO groups are *trans* to the O-bonded ligands (Figs. 2 and 3). The ReO_3C_3 core is a very distorted octahedron with interbond L–Re–L′ angles from 78.4(2) to 100.0(2)°.

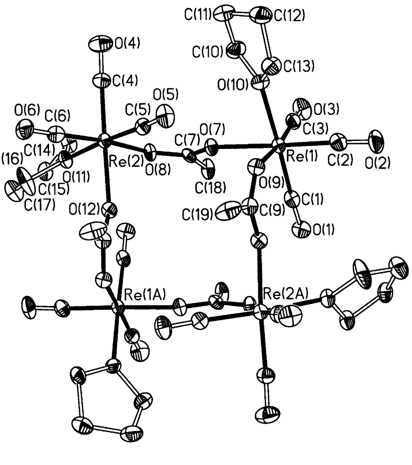


Fig. 2. Perspective drawing of $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4$ (**1**). Atoms are represented by thermal ellipsoids at the 40% probability level. Hydrogen atoms are omitted for clarity.

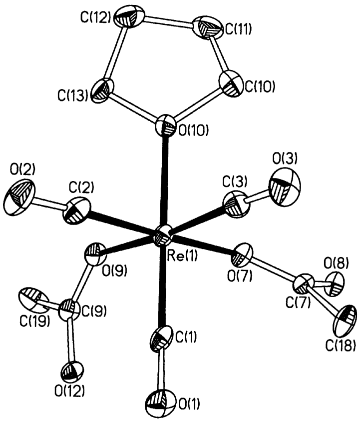


Fig. 3. Coordination polyhedron of Re(1) atom in $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4$ (**1**). Atoms are represented by thermal ellipsoids at the 40% probability level. Hydrogen atoms are omitted for clarity.

The Re–C distances in **1** (Table 2) fall within a narrow range 1.889(7)–1.900(7) Å and are typical for numerous Re(I) carbonyls. However, they are significantly shorter than those in the parent rhenium pentacarbonyl chloride [12] ($Re\text{–}CO_{av.} = 1.997$ Å) where all CO groups, except one, are *trans* to each other. Bonded THF molecules have an average Re–O distance 2.193(4) Å in **1**. Only four Re(I) compounds that have THF ligands together with carbonyl groups have been structurally defined, and the reported Re–L distances for these complexes (Table 3) are close to those found in **1**.

The essential feature of the structure is the presence of acetato-bridges, connecting pairs of rhenium atoms, with metal–metal separations of 5.409 and 5.402 Å for Re(1)–Re(2) and Re(1)–Re(2A), respectively (Fig. 2). That is achieved by an *anti–syn* configuration of acetate bridging groups (Scheme 1(e)). The *anti–syn* configuration in metal carboxylates is known to lead frequently to polymeric structures [17]. In our case the presence of two acetate groups in *anti–syn* geometry coordinated to each rhenium center with an O–Re–O angle of 79.0(2)° between them makes possible the formation of a cyclic tetrameric molecule. In the bridging acetate groups the average Re–O distance is 2.128(4) Å and average angles are: ∠Re–O–C 130.9(4)° (*syn*) and 135.7(4)° (*anti*), ∠O–C–O 122.9(5)°, $\angle O\text{–}C\text{–}CH_3$ 116.1(5)° (*syn*) and 121.1(6)° (*anti*).

The present example of acetate bridges coordinated in the *anti–syn* mode is the first one in the chemistry of rhenium carboxylates. A survey of all the known types of $Re\text{–}RCO_2$ interactions (Scheme 1) shows only a few examples of carboxylate anions functioning as unidentate ligands (Scheme 1(a)), and most of them contain trifluoroacetates [18–23]. A dangling acetate group was found in a quadruply bonded $Re_2Cl_4(OPPh_3)_2(CH_3CO_2)_2$ [24], but structural data are not available in full. The Re–O bond lengths for these

Table 2
Selected bond distances (Å) and angles (°) for [Re(CO)$_3$(μ-CH$_3$CO$_2$)(THF)]$_4$ (**1**)

Re–CO	Re(1)–C(1)	1.893(6)	Re(2)–C(5)	1.899(7)
	Re(1)–C(2)	1.895(7)	Re(2)–C(4)	1.889(7)
	Re(1)–C(3)	1.900(7)	Re(2)–C(6)	1.889(7)
Re–Ac	Re(1)–O(7)	2.134(4)	Re(2)–O(12)	2.130(4)
	Re(1)–O(9)	2.125(4)	Re(2)–O(8)	2.124(4)
Re–THF	Re(1)–O(10)	2.198(4)	Re(2)–O(11)	2.188(4)
OC–Re–CO	C(1)–Re(1)–C(2)	86.1(3)	C(5)–Re(2)–C(4)	86.9(3)
	C(1)–Re(1)–C(3)	87.1(3)	C(5)–Re(2)–C(6)	87.7(3)
	C(2)–Re(1)–C(3)	88.0(3)	C(4)–Re(2)–C(6)	88.3(3)
OC–Re–Ac	C(1)–Re(1)–O(7)	98.5(2)	C(5)–Re(2)–O(12)	98.3(2)
	C(1)–Re(1)–O(9)	100.0(2)	C(5)–Re(2)–O(8)	99.5(2)
	C(2)–Re(1)–O(7)	173.0(2)	C(4)–Re(2)–O(12)	172.5(2)
	C(2)–Re(1)–O(9)	95.1(2)	C(4)–Re(2)–O(8)	94.7(2)
	C(3)–Re(1)–O(7)	97.6(2)	C(6)–Re(2)–O(12)	97.2(2)
	C(3)–Re(1)–O(9)	172.4(2)	C(6)–Re(2)–O(8)	172.3(2)
OC–Re–THF	C(1)–Re(1)–O(10)	177.7(2)	C(5)–Re(2)–O(11)	176.6(2)
	C(2)–Re(1)–O(10)	95.7(2)	C(4)–Re(2)–O(11)	95.9(2)
	C(3)–Re(1)–O(10)	94.3(2)	C(6)–Re(2)–O(11)	94.2(2)
Ac–Re–Ac	O(7)–Re(1)–O(9)	78.9(2)	O(12) –Re(2)–O(8)	79.1(2)
Ac–Re–THF	O(7)–Re(1)–O(10)	79.6(2)	O(12)–Re(2)–O(11)	78.7(2)
	O(9)–Re(1)–O(10)	78.5(2)	O(8)–Re(2)–O(11)	78.4(2)
Re–O–C	Re(1)–O(7)–C(7)	135.2(4)	Re(2)–O(12)–C(9A)	136.1(4)
	Re(1)–O(9)–C(9)	131.0(4)	Re(2)–O(8)–C(7)	130.7(4)
O–C–O	O(7)–C(7)–O(8)	122.5(5)	O(9)–C(9)–O(12A)	123.2(6)
O–C–C	O(7)–C(7)–C(18)	121.5(6)	O(12A)–C(9)–C(19)	120.7(6)
	O(9)–C(9)–C(19)	116.1(6)	O(8)–C(7)–C(18)	116.1(5)

Table 3
Averaged Re–C and Re–O distances (Å) for carbonyl and tetrahydrofuran ligands in Re(I) complexes

Complex	Re–C$_{CO}$	Re–O$_{THF}$	Ref.
[Re(CO)$_3$(μ-CH$_3$CO$_2$)(THF)]$_4$	1.894	2.193	This work
Re$_2$(μ-Cl)$_2$(CO)$_6$(THF)$_2$	1.875	2.212	[13]
Re$_2$(μ-Br)$_2$(CO)$_6$(THF)$_2$	1.808	2.133	[14]
[Re(CO)$_3$(PMe$_2$O$_2$)(THF)]$_2$	1.880	2.154	[15]
Re[HB(pz)$_3$](CO)$_2$(THF)[a]	1.875	2.172	[16]

[a] [HB(pz)$_3$] = hydrotris(1-pyrazolyl)borate.

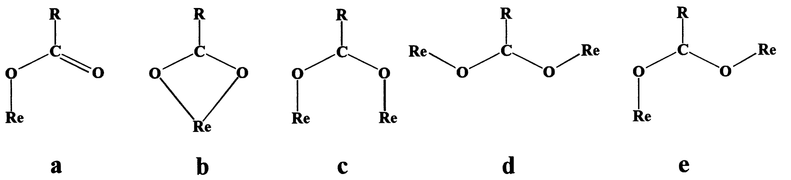
complexes range from 2.141 to 2.188 Å and the angles Re–O–C and O–C–O are 125.2–132.1°.

Examples of carboxylates chelating to the rhenium atom (Scheme 1(b)) are very rare. Four structures have been done with bidentate chelating acetates including unsymmetrical (Re–O distances are 2.144 and 2.462 Å) [25] and symmetrical [20,26,27] RCO$_2^-$-groups. These compounds show very small Re–O–C and O–Re–O angles (around 92.3 and 58.5°, respectively) owing to limitations on the four-membered rings.

Among the bidentate bridging rhenium carboxylates (Scheme 1(c)–(e)) the most common is *syn–syn* geometry (Scheme 1(c)) which brings the metal atoms close enough to form structures with Re–Re bonds. Data (about 40 structures) are available for the Re$_2^{6+}$, Re$_2^{5+}$ and Re$_2^{4+}$ core species with bond orders 4, 3.5 and 3, respectively. The geometrical data for acetates [28–30] are summarized in Table 4.

The only example of an *anti–anti* geometry (Scheme 1(d)) is reported [31] for [Re$_2$Cl$_4$(μ-HCO$_2$)$_2$(HCO$_2$)]^-_n where formate ions connect quadruply bonded dirhenium units in infinite chains. The Re–O distances are about 2.35 Å which provide Re–Re separation across the bridge of 6.598 Å, the longest known for bridging rhenium carboxylates.

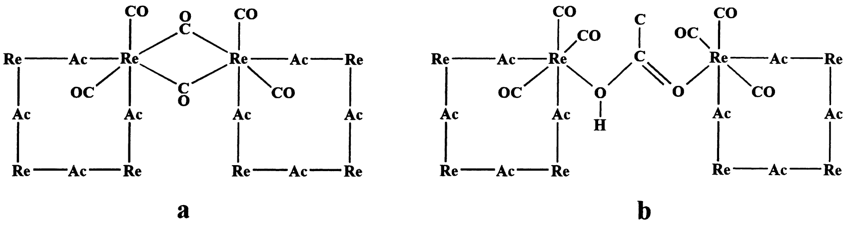
Finally, as we have mentioned before, the first *anti–syn* geometry (Scheme 1(e)) for an Re–RCO$_2$ interaction has



Scheme 1.

Table 4
The range of geometrical characteristics (Å and °) for dirhenium complexes with bridging acetate groups and with multiple bonds between metal atoms

$Re_2{}^{n+}$ core	Bond order	Re–Re	Re–O	∠Re–Re–O	∠Re–O–C	∠O–C–O
$Re_2{}^{6+}$	4.0	2.177–2.244	1.931–2.096	87.4–91.2	118.5–129.2	107.6–121.9
$Re_2{}^{5+}$	3.5	2.216–2.300	2.049–2.099	85.6–93.3	114.3–124.0	121.8–123.3
$Re_2{}^{4+}$	3.0	2.276–2.315	2.072–2.166	86.8–90.7	117.6–121.6	120.3–123.4



Scheme 2.

been found in this work. Some rhenium complexes with that type of coordination mode are known for R other than alkyl or H, but those compounds have carbon atoms connected with another Re [32] or O [33,34] atoms. The rhenium–rhenium separations in the crystal structures are in the range 5.015–5.361 Å and the dimensions of bridging groups are close to those in compound **1**.

A few comments should be made about the probable structure of the initial reaction product before the addition of THF. Since it has proven impossible to get single crystals of this compound we can only speculate on its composition. Two models seem to be reasonable. Both are based on the assumption that in the absence of coordinated THF molecules all rhenium atoms still have a coordination number of 6 and oxidation state +1, so that all tetramers must be connected to each other. That could be achieved either by μ-carbonyl groups (Scheme 2(a)) or by μ-CH_3CO_2H molecules (Scheme 2(b)). In the first case the addition of tetrahydrofuran may be supposed to break carbonyl bridges which become terminal in compound **1**. In the latter case a bridging molecule of acetic acid is simply replaced by two THF groups. From analysis of IR data of the initial product this second model looks more favorable. Also, the connection by acetic acid is much weaker than that by carbonyl bridges. One example of such a carboxylate bridge was reported [35,36] for a rhenium complex.

4. Concluding remarks

The striking difference between the Re and Mo (or W) carbonyls in their reactions with CH_3CO_2H has been revealed in this study. In the rhenium case, the reaction does not result in total loss of CO groups but instead yields the remarkable tetranuclear Re(I) complex in which acetate ions act as bridges between each pair of metal centers. Interestingly, both $Re(CO)_5Cl$ and $Re_2(CO)_{10}$ afford the same product **1** under the experimental conditions employed in this work. The observation that rhenium carbonyls are only partly decarbonylated by acetate, even under severe reflux conditions, is quite surprising. While fully substituted acetates cannot be prepared by this route, perhaps with higher boiling carboxylic acids $Re_2(O_2CR)_4$ species may be obtainable. Nevertheless, the synthesis of the mixed carbonyl–acetate tetranuclear rhenium complex **1** is very interesting since it provides a convenient entry into the chemistry of such rare species. For instance, complex **1** could be useful in strategic ligand substitution transformations when the tetranuclear rhenium core to be retained.

5. Supplementary material

A listing of observed and calculated structure factors (20 pages) and complete tables of crystal data, positional and isotropic thermal parameters and anisotropic displacement parameters, as well as complete tables of bond distances and angles and coordinates of hydrogen atoms for $[Re(CO)_3(\mu\text{-}CH_3CO_2)(THF)]_4 \cdot 3THF \cdot 0.5C_6H_{14}$ are available from the authors upon request.

Acknowledgements

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