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Note

Reactions of $Os_3(CO)_{12}$ with carboxylic acids in a microwave reactor; synthesis of $Os_2(benzoate)_2(CO)_6$, a dinuclear osmium(I) compound with aromatic carboxylate ligands

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ABSTRACT

Microwave heating is used to synthesize compounds of the type $Os_2(\mu\text{-}O_2CR)_2(CO)_6$, including the first example with aromatic carboxylate ligands. The one-step preparation of these complexes is straightforward and does not require an air-free reaction environment. The compounds for which R = Me (**1**) or Et (**2**) are produced in higher yield than previously reported by the microwave irradiation of $Os_3(CO)_{12}$ for less than 15 min in acetic or propionic acid, respectively. This method may also be used to prepare the new compound $Os_2(\mu\text{-}O_2CH)_2(CO)_6$ (**3**) by reaction with formic acid, but a higher yield is obtained when 1,2-dichlorobenzene is used as a co-solvent. Irradiating a mixture of $Os_3(CO)_{12}$ and excess benzoic acid in 1,2-dichlorobenzene gives another new complex $Os_2(\mu\text{-}O_2CC_6H_5)_2(CO)_6$ (**4**). X-ray crystallographic analyses of **3** and **4** reveal molecular structures similar to that of **1** with approximate C_{2v} symmetry. When the molar ratio of benzoic acid to $Os_3(CO)_{12}$ is 1:1, then the major product is the trinuclear cluster $Os_3(\mu\text{-}H)(\mu\text{-}O_2CC_6H_5)(CO)_{10}$ (**5**) instead of a dinuclear compound. This represents the first instance in which a cluster of this type has been produced directly from $Os_3(CO)_{12}$ instead of through a multi-step procedure.

1. Introduction

Dinuclear osmium(I) carbonyl complexes with carboxylate ligands are relatively rare. Lewis, Johnson and coworkers discovered in 1969 that the diosmium(I) compounds $Os_2(\mu\text{-}O_2CR)_2(CO)_6$, R = Me (**1**) or Et (**2**) could be synthesized by heating a mixture of $Os_3(CO)_{12}$ and neat acetic acid or neat propionic acid in a sealed Carius tube at 185 °C [1]. Compound **1** was produced in 76% yield after 5 h of heating, and **2** was prepared in 70% yield after 14 h of heating. In 1987, Deeming et al. employed the same method to synthesize $Os_2(\mu\text{-}O_2CCHMe_2)_2(CO)_6$ in 71% yield after 6 h of heating in isobutyric acid [2]. In 1988, Deeming's group showed that heating a mixture of $Os_3(CO)_{12}$ and trifluoroacetic acid at 120 °C for only 2 h produced $Os_2(\mu\text{-}O_2CCF_3)_2(CO)_6$ in 50% yield, while heating at higher temperatures led to the formation of mononuclear products [3]. To date, only these four aliphatic carboxylic acids have been used to prepare Os_2^{2+} compounds. An X-ray crystallographic analysis of $Os_2(\mu\text{-}O_2CMe)_2(CO)_6$ was carried out by Bullitt and Cotton in 1971 [4]. The molecular geometry was shown to have approximate C_{2v} symmetry with cis bridging carboxylate ligands as illustrated by structure **A** in Chart 1. Spectroscopic evidence suggests that the other $Os_2(\mu\text{-}O_2CR)_2(CO)_6$ compounds have a similar structural arrangement.

To our knowledge, there have been no reports of direct reactions of aromatic carboxylic acids with $Os_3(CO)_{12}$. Because of the difficulty of removing the first two CO ligands from $Os_3(CO)_{12}$, the standard practice is to initially convert this cluster into derivatives with more labile ligands. Such activated clusters react with either aliphatic or aromatic carboxylic acids to generate triosmium products of the type $Os_3(\mu\text{-}H)(\mu\text{-}O_2CR)(CO)_{10}$, which have the molecular structure represented by **B** in Chart 1. Thus the starting materials $Os_3(CO)_{10}(MeCN)_2$, $Os_3(CO)_{10}(C_6H_8)$, $Os_3(CO)_{10}(C_8H_{14})_2$, $Os_3(\mu\text{-}H)(\mu\text{-}OH)(CO)_{10}$, and the silica-anchored species $Os_3(\mu\text{-}H)(\mu\text{-}O{-}Si{-}O{-})(CO)_{10}$ have been used to synthesize dozens of such compounds [5–19].

Microwave heating is becoming more widespread in the preparation of coordination and organometallic complexes due to reaction rate enhancements, energy savings, higher product yields, automated control of reaction parameters, and expanded ranges of reaction temperature and pressure [20]. Several recent publications

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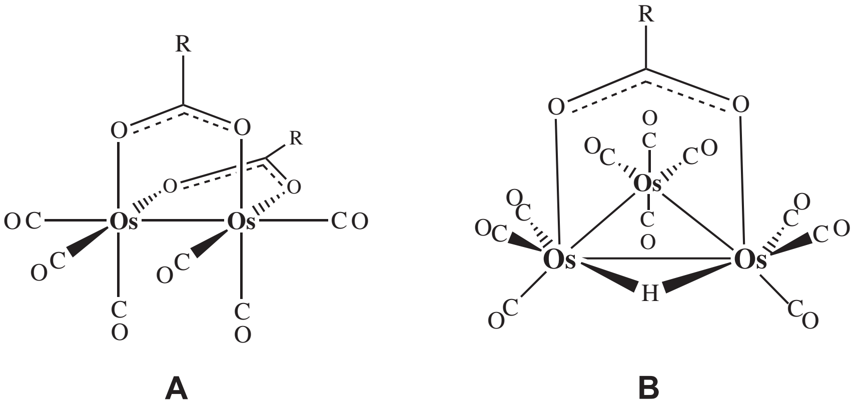

Chart 1. Principal products of the reactions of Os_3 clusters with carboxylic acids.

have focused on microwave-promoted reactions of $Os_3(CO)_{12}$, including its conversion to $Os_3(\mu\text{-}H)_2(CO)_{10}$, $[Os_{10}C(CO)_{24}]^{2-}$, $Os_3(CO)_{11}(NCMe)$, $Os_3(\mu\text{-}H)(\mu\text{-}OH)(CO)_{10}$, and $Os_6(CO)_{18}$ [21–24]. We chose to use a microwave reactor to carry out reactions between $Os_3(CO)_{12}$ and various carboxylic acids with the intent of improving the yield of known $Os_2(\mu\text{-}O_2CR)_2(CO)_6$ compounds as well as synthesizing new complexes including one with bridging aromatic carboxylate ligands. This note presents the results of our investigations with formic, acetic, propionic and benzoic acids.

2. Experimental

2.1. Methods and materials

All syntheses were performed using a Discover-SP microwave reactor (2455 MHz, CEM Corp., Matthews, NC) with thick-walled 35-mL glass vessels equipped with a magnetic stir bar and a PFTE-lined cap. In each case, the reactor was set to a maximum pressure of 300 psi, and the highest stir rate was used. Caution was exercised due to the toxic nature of CO and metal carbonyl compounds. All manipulations were carried out in a highly efficient fume hood. Extra precautions were taken when the reactions were under pressure; the microwave reactor was placed in a fume hood and the hood sash was left down until a few minutes after the pressure had been released. The $Os_3(CO)_{12}$ was purchased from Strem; all other reagents and solvents were obtained from Aldrich. All chemicals were used as received. Infrared spectra were obtained using a Nicolet Avatar 320 FTIR with a CaF_2 solution cell. ^1H NMR spectra were recorded on an EM360 NMR spectrometer. Preparative thin-layer chromatography (TLC) was carried out on Analtech silica gel 60 (0.50 mm) plates. Columbia Analytical Services carried out the elemental analyses.

2.2. Synthesis of $Os_2(\mu\text{-acetate})_2(CO)_6$ (**1**)

A 154.6 mg (0.1705 mmol) sample of $Os_3(CO)_{12}$ was added to 7 mL of glacial acetic acid, and the mixture was irradiated at an initial power of 300 W. After 2 min, the temperature reached 180 °C and this temperature was maintained as the sample was irradiated for an additional 10 min. Distilled water (70 mL) was added to the resulting colorless solution, and the product precipitated as a white solid. A portion of CH_2Cl_2 (55 mL) was then added to re-dissolve the crude product, and the mixture was stirred for 10 min before being placed in a separatory funnel. The CH_2Cl_2 layer was collected, dried with anhydrous $MgSO_4$, and then allowed to evaporate. The crude product was recrystallized from a 1:3 dichloromethane/hexanes mixture. Yield: 161.2 mg (94.5%) of $Os_2(\mu\text{-}O_2CCH_3)_2(CO)_6$ (**1**). IR (ν_{CO}, $CHCl_3$): 2100 (m), 2066 (vs), 2014 (s) and 1998 (vs) cm^{-1}. Anal. Calc. for $C_{10}H_6O_{10}Os_2$: C, 18.02; H, 0.91%. Found: C, 18.06; H, 0.97%. ^1H NMR ($CDCl_3$): δ 2.07 (s, CH_3) ppm.

2.3. Synthesis of $Os_2(\mu\text{-propionate})_2(CO)_6$ (**2**)

The same method used to prepare **1** was employed with 92.0 mg (0.101 mmol) of $Os_3(CO)_{12}$ and 8 mL of propionic acid. The yield of **2** was 85.3 mg (80.7%). IR (ν_{CO}, $CHCl_3$): 2099 (m), 2066 (vs), 2014 (s) and 1997 (vs). Anal. Calc. for $C_{12}H_{10}O_{10}Os_2$: C, 20.75; H, 1.45%. Found: C, 21.39; H, 1.35%. ^1H NMR ($CDCl_3$): δ 2.34 (q, 2H, CH_2), 1.10 (t, 3H, CH_3) ppm.

2.4. Synthesis of $Os_2(\mu\text{-formate})_2(CO)_6$ (**3**)

A 91.3 mg (0.101 mmol) sample of $Os_3(CO)_{12}$ was added to 8 mL of formic acid, and the mixture was irradiated at an initial power of 300 W. After 3 min of heating, the temperature reached 177 °C and the pressure reached 300 psi. At this point the microwave source was turned off, the vessel was cooled to 60 °C, and the pressure was released. Microwave heating was then resumed, and the temperature reached 177 °C after 2.2 min. This temperature was maintained for 9 additional minutes, and then the reaction mixture was evaporated under a stream of nitrogen. The residue was dissolved in dichloromethane and filtered through silica gel. The crude product formed upon slow evaporation. It was recrystallized from a 1:3 dichloromethane/hexanes mixture. The yield of **3** was 60.6 mg (62.8%).

In order to investigate the source of the pressure buildup mentioned above, we conducted a control experiment in which 8 mL of formic acid was subjected to microwave heating in the absence of $Os_3(CO)_{12}$. A pressure of 300 psi was reached after 4.4 min of irradiation at 180 °C. After cooling to 25 °C, the pressure was still 190 psi. We assume that these high pressures are due to the well-known thermal decomposition of formic acid to produce hydrogen and carbon dioxide [25].

In an effort to minimize the decomposition of formic acid and the resulting pressure buildup, we decided to attempt the preparation of **3** with 1,2-dichlorobenzene as a solvent. A 96.8 mg (0.107 mmol) sample of $Os_3(CO)_{12}$ was added to 7 mL of 1,2-dichlorobenzene and 2.5 mL of formic acid. After 4 min of microwave heating, the temperature reached 177 °C and the pressure reached 155 psi. This temperature was maintained for 10 additional minutes. The pressure reached a maximum of 162 psi. The reaction mixture was evaporated under a stream of nitrogen, and the product was isolated as described above. The yield of **3** was 81.1 mg (79.4%). IR (ν_{CO}, $CHCl_3$): 2104 (m), 2071 (vs), 2021 (s) and 2004 (vs). Anal. Calc. for $C_8H_2O_{10}Os_2$: C, 15.05; H, 0.32%. Found: C, 15.46; H, 0.41%. ^1H NMR ($CDCl_3$): δ 8.44 (s, CH). Single crystals for X-ray analysis were grown by slow evaporation of a hexane/CH_2Cl_2 solution.

2.5. *Synthesis of Os_2(μ-benzoate)$_2$(CO)$_6$ (**4**)*

Benzoic acid (66.4 mg, 0.544 mmol) and $Os_3(CO)_{12}$ (70.4 mg, 0.0776 mmol) were added to 6 mL of 1,2-dichlorobenzene and the mixture was irradiated at an initial power of 300 W. After 3.5 min the temperature reached 188 °C, and this temperature was maintained as the sample was irradiated for an additional 15 min. A light yellow solution was produced. The solvent was removed by evaporation under a stream of N_2 gas. The residue was dissolved in CH_2Cl_2 and subjected to TLC with an eluent of 3.5:1 *n*-hexane/CH_2Cl_2. Two bands were collected. The top band ($R_f = 0.69$) contained 2.9 mg of an unidentified light yellow product. IR (ν_{CO}, $CHCl_3$): 2075 (m), 2065 (s), 2048 (m), 2027 (vs), 2017 (s, sh), 2003 (w), 1982 (w). The second band ($R_f = 0.54$) contained 61.4 mg (66.7% yield) of colorless $Os_2(\mu\text{-}O_2CC_6H_5)_2(CO)_6$ (**4**). IR (ν_{CO}, $CHCl_3$): 2099 (m), 2067 (vs), 2015 (s), 2002 (vs), 1994 (w). Anal. Calc. for $C_{20}H_{10}O_{10}Os_2$: C, 30.38; H, 1.27%. Found: C, 30.42; H, 1.30%. ^1H NMR ($CDCl_3$): δ 8.07 (m, 2H, C_6H_5), 7.95 (m, 1H, C_6H_5), 7.44 (m, 2H, C_6H_5) ppm. Crystals were grown by slow evaporation of a hexane/CH_2Cl_2 solution.

2.6. *Synthesis of Os_3(μ-H)(μ-benzoate)(CO)$_{10}$ (**5**)*

Benzoic acid (9.9 mg, 0.081 mmol) and $Os_3(CO)_{12}$ (71.2 mg, 0.0785 mmol) were added to 8 mL of 1,2-dichlorobenzene in a 35-mL reaction vessel equipped with a magnetic stir bar. The vessel was sealed and irradiated at an initial power of 300 W. After 3 min, the temperature reached 189 °C. A temperature range of 188–190 °C was maintained as the sample was irradiated for an additional 12 min. A yellow-brown solution was produced. The solvent was removed by evaporation under a stream of N_2 gas. The residue was dissolved in CH_2Cl_2 and subjected to TLC with an eluent of 3:1 *n*-hexane/CH_2Cl_2. Two bands were collected. The top band ($R_f = 0.81$) contained 38.4 mg (50.3% yield) of $Os_3(\mu\text{-}H)(\mu\text{-}O_2CC_6H_5)(CO)_{10}$ (**5**). IR (ν_{CO}, $CHCl_3$): 2113 (m), 2099 (w), 2074 (vs), 2064 (vs), 2024 (vs), 2017 (vs), 1986 (m, sh). Anal. Calc. for $C_{17}H_6O_{12}Os_3$: C, 20.99; H, 0.62%. Found: C, 21.03; H, 0.65%. ^1H NMR ($CDCl_3$): δ −12.19 (s, 1H, OsH), 7.27 (m, 2H, C_6H_5), 7.07 (m, 1H, C_6H_5), 6.52 (m, 2H, C_6H_5) ppm. The second band ($R_f = 0.59$) contained 2.6 mg of an unidentified yellow product. IR (ν_{CO}, $CHCl_3$): 2125 (m), 2082 (m), 2036 (s), 2024 (vs), 2004 (m), 1980 (s).

2.7. *X-ray crystallography*

The crystal structure determination of compounds **3** and **4** were carried out using a Bruker APEX2 CCD-based diffractometer equipped with a low-temperature device and an Mo-target X-ray tube. The X-ray data were collected at 100(2) K. Data collection, indexing, and initial cell refinements were carried out using APEX2 [26], with the frame integrations and final cell refinements carried out using SAINT [27]. An absorption correction was applied using SADABS [28], and all non-hydrogen atoms were refined anisotropically. All hydrogen atoms were placed in idealized positions and were refined using a riding model. The structures were examined using the Addsym subroutine of PLATON to ensure that no additional symmetry could be applied to the models [29]. The structure was solved and refined using SHELXTL [30]. Refinement details and structural parameters for the compounds are summarized in Table 1.

3. Results and discussion

3.1. *Improved preparation of previously known diosmium(I) compounds*

Compounds **1** and **2** were prepared much more efficiently with microwave heating than with conventional heating. The acetate compound was produced in 95% yield in only 12 min of microwave irradiation, while the conventional method produced a 76% yield with 5 h of heating. For the propionate compound, the new method gave an 81% yield in 14 min compared to a 70% yield in 14 h for the old method. Not only are the yields higher and the rates faster for the microwave method, it is also simpler and more convenient to perform. There is no need to carefully place the reactants in a Carius tube, seal it with a torch under nitrogen or vacuum, and then cut it open to retrieve the products. Instead reactants are easily loaded into a wide-mouthed glass vessel exposed to room atmosphere and a cap is snapped onto the top.

3.2. *Synthesis of new diosmium(I) compounds*

The use of a microwave reactor allowed for the rapid, single-step preparation of the new compounds **3** and **4** directly from $Os_3(CO)_{12}$. The formate compound **3** may be synthesized by the same method used to prepare **1** and **2** (using neat carboxylic acid), but the use of 1,2-dichorobenzene as a solvent eliminated several problems and produced a higher yield. Our attempts to prepare **4** without a solvent were unsuccessful, while the use of 1,2-dichorobenzene as a solvent worked well. This benzoate complex is the first example of a $Os_2(\mu\text{-}O_2CR)_2(CO)_6$ compound with aromatic carboxylate ligands.

3.3. *Crystallographic characterization of **3** and **4***

X-ray crystallographic analyses of **3** and **4** were carried out, and the data collection and refinement parameters are given in Table 1. The molecular structure of **3** is illustrated in Fig. 1. For **4**, there are two independent molecules in the unit cell, and the structure of

Table 1
X-ray crystallographic data and processing parameters for clusters **3** and **4**.

Compound	**3**	**4**
CCDC entry no.	897819	896037
Cryst syst	Triclinic	Monoclinic
Space group	*P* −1	*P*2(1)/*n*
a, Å	7.3402(6)	10.1543(6)
b, Å	7.6918(6)	11.6656(6)
c, Å	11.665(1)	35.796(2)
α, deg	75.890(1)	90
β, deg	84.795(1)	92.845(1)
γ, deg	79.924(1)	90
V, Å^3	628.08(9)	4235.0(4)
Mol formula	$C_8H_2O_{10}Os_2$	$C_{20}H_{10}O_{10}Os_2$
FW	638.50	790.68
Formula units per cell (Z)	2	8
D_{calcd} (Mg/m^3)	3.376	2.480
λ (Mo Kα), Å	0.71073	0.71073
Absorption coeff (mm^{-1})	20.258	12.045
Abs corr factor	0.6209/0.1159	0.5842/0.1725
Total reflections	7607	49,875
Independent reflections	2756	9392
Data/res/parameters	2756/0/182	9392/0/577
*R*1[a] [$I \geq 2\sigma(I)$]	0.0180	0.0258
*wR*2[b]	0.0478	0.0664
GOF on F^2	1.033	1.017
Δρ(max), Δρ(min) (e/Å^3)	1.252/−1.862	1.122/−1.380

[a] $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$.
[b] $R2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$.

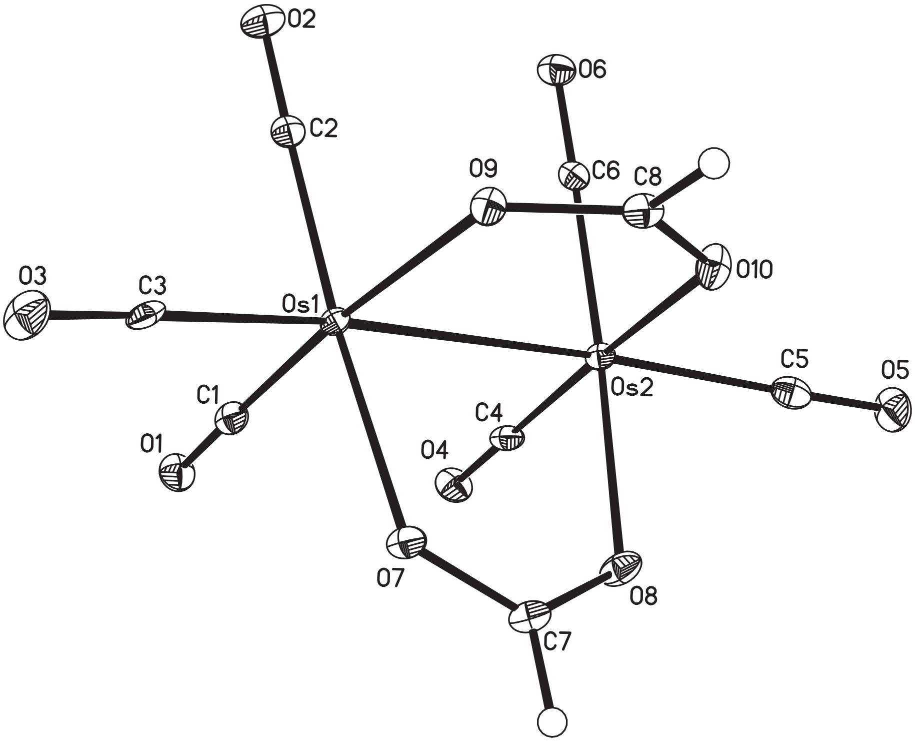

Fig. 1. Thermal ellipsoid plot of the molecular structure of **3** at the 50% probability level.

one of these is illustrated in Fig. 2. Both of these new complexes have a molecular geometry similar to that of **1** [4]. The carboxylate ligands adopt a cis arrangement and two of the CO ligands are axially coordinated while the other four are bound in equatorial sites. The axial carbonyl ligands are bent toward the carboxylate ligands and are more weakly bound to the metal atoms than the equatorial CO ligands. Selected bond distances and angles for **1**, **3**, and **4** are compared in Table 2. The Os–Os bond distances get

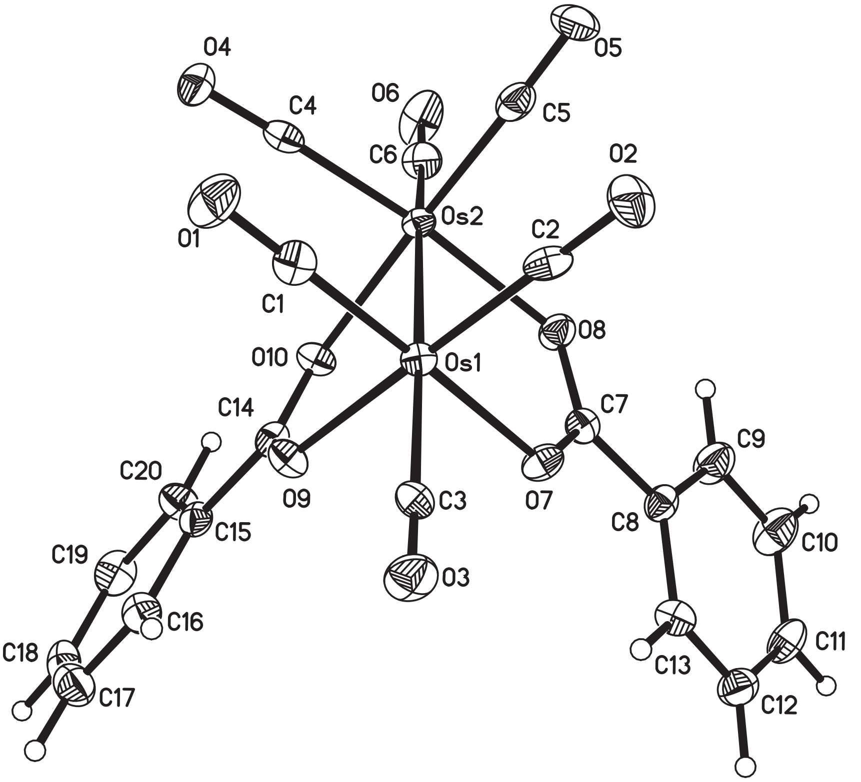

Fig. 2. Thermal ellipsoid plot at the 50% probability level of the molecular structure of one of the two independent molecules of **4**.

Table 2
Selected average bond distances (Å) and angles (°) for $Os_2(\mu\text{-}O_2CR)_2(CO)_6$ complexes.

	R = H (**3**)	R = C_6H_5 (**4**)	R = CH_3 (**1**)
Bond distances			
Os–Os	2.7545(2)	2.748(1)	2.731(2)
Os–O	2.124(6)	2.119(3)	2.076(6)
Os–C_{ax}	1.983(4)	1.989(8)	1.96(2)
Os–C_{eq}	1.877(4)	1.873(4)	1.79(1)
C–$O_{carboxylate}$	1.257(5)	1.265(5)	1.29(1)
C_{ax}–O_{ax}	1.124(5)	1.124(5)	1.10(2)
C_{eq}–O_{eq}	1.147(5)	1.150(5)	1.16(1)
Bond angles			
Os–Os–C_{ax}	168.0(6)	169.8(4)	170.5(6)
Os–Os–C_{eq}	93(1)	94.5(5)	93.4(3)
Os–Os–O	83.0(3)	83.0(3)	83.5(2)
C_{eq}–Os–C_{eq}	90.1(1)	90.1(5)	89.7(7)
O–Os–O	83.4(7)	84.1(3)	83.5(4)
O–C–O	126.8(3)	125.2(2)	124(1)

slightly longer as the strength of the parent carboxylic acid increases (acetate < benzoate < formate).

3.4. *Preparation of $Os_3(\mu\text{-}H)(\mu\text{-benzoate})(CO)_{10}$ (**5**)*

Using the same reaction conditions that were successful for the synthesis of **4**, but changing the molar ratio of benzoic acid to $Os_3(CO)_{12}$ from 7:1 to 1:1 produces the trinuclear cluster $Os_3(\mu\text{-}H)(\mu\text{-}O_2CC_6H_5)(CO)_{10}$ (**5**) in 50% yield with no trace of the dinuclear compound. As noted above, it has previously been necessary to utilize multi-step procedures involving the initial conversion of $Os_3(CO)_{12}$ to a more reactive species in order to prepare compounds of the type $Os_3(\mu\text{-}H)(\mu\text{-}O_2CR)(CO)_{10}$. A three-step method was used to prepare **5** with an overall yield of 39% based on $Os_3(CO)_{12}$ [1]. Thus a microwave reactor allows for a higher yield preparation in only one step.

4. Conclusion

Microwave heating is shown to be effective for the high-yield, single-step synthesis of dinuclear osmium(I) compounds of the type $Os_2(\mu\text{-}O_2CR)_2(CO)_6$, including the first examples for which R is H or Ph. Using a one-step microwave method, the trinuclear cluster $Os_3(\mu\text{-}H)(\mu\text{-benzoate})(CO)_{10}$ is also easily prepared in higher yield than previously reported. This is accomplished without the need to replace two CO groups with more labile ligands, and is the first instance in which a cluster of this type has been synthesized directly from $Os_3(CO)_{12}$. The use of a microwave reactor has many advantages over traditional preparative methods including very rapid reaction rates, automated control of temperature and pressure, and simpler procedures with no need for inert gases. We intend to continue exploring the microwave-promoted reactions of $Os_3(CO)_{12}$ with other reagents such as di-, tri-, and tetracarboxylic acids.

Acknowledgments

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Appendix A. Supplementary material

CCDC 897819 and 896037 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via http://www.ccdc.cam.ac.uk/data_request/cif.

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