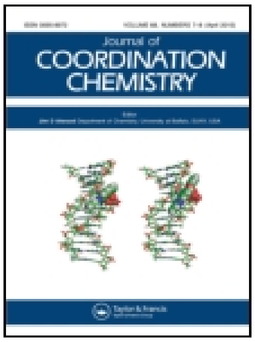

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Microwave synthesis of dirhenium paddlewheel complexes

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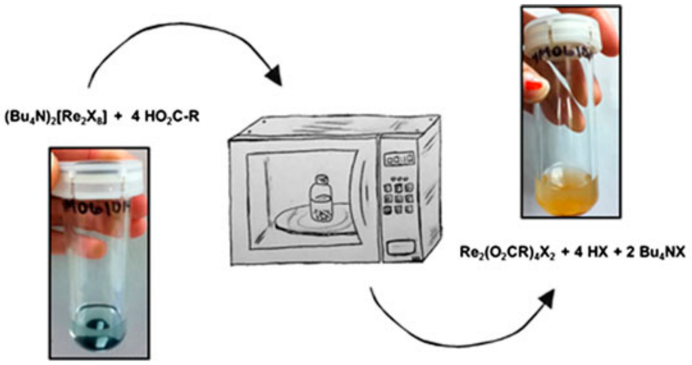

Tetrakis(carboxylato)dihalidodirhenium (III,III) complexes were efficiently synthesized utilizing microwave irradiation. Re_2(acetate)$_4X_2$, Re_2(butyrate)$_4X_2$, and Re_2(pivalate)$_4X_2$, where X = Cl or Br, were produced in under one hour in the microwave with comparable yields to traditional refluxes which take as long as four days. All reaction samples were prepared under inert gas and the septa caps of the microwave reaction vessels were sufficient to maintain this environment during the reaction. Re_2(acetate)$_4X_2$ and Re_2(butyrate)$_4X_2$ were synthesized from the $(Bu_4N)_2[Re_2X_8]$ salt, the acid, the anhydride, and in some cases hexanes. Re_2(pivalate)$_4X_2$, traditionally prepared via melt reactions, were synthesized from the $(Bu_4N)_2[Re_2X_8]$ salt and pivalic acid combined in diglyme or diglyme and hexanes.

Keywords: Microwave synthesis; Tetrakis(carboxylato)dihalidodirhenium (III,III); Dirhenium tetracarboxylate; Quadruple bond; Paddlewheel complex

1. Introduction

Quadruply bonded dimetal complexes are interesting in their reaction chemistry [1], and they have applications in nitrile activation [2], supramolecular assemblies for gas storage and separation [3–5] as well as photon harvesters, charge carriers, and light emitters in photovoltaic and light-emitting devices [6–10]. However, the reaction times are a limitation

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*Corresponding author. Email: creed@brockport.edu to the traditional syntheses of these complexes in high yields. In general, reaction times on the order of hours or days are required for the formation of the quadruple bond or the addition of labile carboxylate ligands [1], with the synthesis of $Mo_2(O_2CC_6H_5)_4$ requiring four hours [11] and $Re_2(O_2CC(CH_3)_3)_4Br_2$ requiring four days [12].

Since the heating effect of microwaves was discovered [13] in 1945, microwave radiation has been used to synthesize a wide range of compounds and materials: organic [14, 15] and organometallic compounds [14–17], metallocarboranes [18–20], inorganic coordination complexes [14, 21], inorganic nanomaterials [13, 22], metal–organic frameworks [23, 24], and carbon nanotubes [13], to name a few. Microwave-assisted synthesis has been applied to dimetal paddlewheel complexes containing rhodium, ruthenium [25–29], and molybdenum with metal–metal bond orders ranging from one to four. The first quadruply bonded dimetal carboxylate complex, where the metal was molybdenum, was synthesized via microwave irradiation by Ardon *et al.* in 2004 in a modified conventional microwave [30]. This synthesis was reproduced in 2007 with improved yield in a laboratory microwave by Johnson and Powell along with two additional dimolybdenum complexes [31]. This new synthetic route is most significant in the field of quadruply bonded dimolybdenum complexes in that it has reduced the reaction times from hours to minutes.

To date, quadruply bonded dirhenium carboxylate complex synthesis using microwaves has not been investigated. Reported here are the syntheses of a series of tetrakis(carboxylato)dihalidodirhenium (III, III) complexes via microwave irradiation.

2. Experimental

2.1. *Materials and methods*

All reactions were carried out in a Discover S microwave reactor with Explorer System (2455 MHz, CEM Corp., Matthews, NC) using dynamic mode, which allowed a reaction temperature to be set while the power fluctuated to maintain the set temperature. Full CEM reaction profile outputs for **1**–**6** can be found in Supporting Information (figures S1–S6). All reactions were performed in thick-walled 35-mL CEM reaction vessels with new Teflon-septa caps. The stir rate of the microwave reactor was always set to high. Samples were prepared in a glove bag or in a Vigor glove box and capped under an Ar or N_2 atmosphere before microwave reactions were carried out. Acetic acid, acetic anhydride, butyric acid, and butyric anhydride were degassed by bubbling argon through the solution in a glove bag for 10 min before use. Anhydrous diglyme was purchased from Acros. Acetic anhydride, butyric acid, butyric anhydride, anhydrous dichlorobenzene, anhydrous acetonitrile, anhydrous dichloromethane, pivalic acid, and $[Bu_4N]_2Re_2Cl_8$ were purchased from Sigma-Aldrich and used without further purification. Glacial acetic acid was purchased from Fisher Scientific. $[Bu_4N]_2Re_2Br_8$ was prepared according to the previously published procedure [32].

Elemental analysis data were obtained from the CENTC Elemental Analysis Facility at the University of Rochester, funded by the NSF CHE-0650456. The samples were weighed with a Perkin Elmer Model AD-6 Autobalance and their compositions were determined with a Perkin Elmer 2400 Series II Analyzer. The samples were handled in a VAC Atmospheres glove box, transferred under argon and combusted in crimp-sealed tin capsules.

Electronic absorption spectra were recorded on a Perkin Elmer Lambda 800 UV–vis spectrometer and carried out on sample solutions in 1 cm × 1 cm quartz cuvettes sealed with Kontes taps. Infrared spectra were recorded with a Thermo Electron Corp. Nicolet 380 FTIR spectrometer in percent reflectance mode using solid state samples. IR bands at 2360 and 2340 cm^{-1} observed in the background scan and in sample spectra are attributed to incomplete subtraction of CO_2 [33]. Full spectra appear in the Supporting Information (figures S7 and S8).

2.2. *Synthesis*

2.2.1. Preparation of tetrakis(acetato)dichloridodirhenium (III,III), $Re_2(O_2CCH_3)_4Cl_2$ (1). In a glove bag, $[Bu_4N]_2[Re_2Cl_8]$ (0.111 g, 0.0973 mmol) was combined with 5 mL of a 4 : 1 mixture by volume of acetic acid/acetic anhydride in a 35-mL reaction vessel equipped with a micro stir bar. The vial was placed in the microwave reactor and the temperature was set to 227 °C. The sample was ramped to 226 °C in 4 min and held at 226 °C for 10 s. The maximum pressure reached was 211 psi. The mixture was cooled to 50 °C, removed from the microwave reactor, and the colorless supernatant was decanted in air. The orange powder was washed in air with ethanol and diethyl ether. Yield: 0.065 g, 98%. Anal. Calcd. for $Re_2O_8C_8H_{12}Cl_2$: C, 14.14; H, 1.78. Found: C, 14.471; H, 1.648. IR: 2985 w, 2931 w, 1446, 1427 sh, 1385, 1346, 1045, 1026, 953, 683, 640, 625 cm^{-1}.

2.2.2. Preparation of tetrakis(acetato)dibromidodirhenium (III,III), $Re_2(O_2CCH_3)_4Br_2$ (2). In a glove bag, $[Bu_4N]_2[Re_2Br_8]$ (0.034 g, 0.023 mmol) was combined with 4 mL of a 4 : 1 mixture by volume of acetic acid/acetic anhydride in a 35-mL reaction vessel equipped with a micro stir bar. The vial was placed in the microwave reactor and the temperature was set to 226 °C. The sample was ramped to 226 °C in 3.5 min and held at 226 °C for 10 s. The maximum pressure reached was 200 psi. The mixture was cooled to 50 °C, removed from the microwave reactor, and the colorless supernatant was decanted in air. The orange powder was washed in air with ethanol and diethyl ether. Yield: 0.016 g, 92%. Anal. Calcd. for $Re_2O_8C_8H_{12}Br_2$: C, 12.50; H, 1.57. Found: C, 12.866; H, 1.369. IR: 2928 w, 1446, 1381, 1346, 1041, 1026 sh, 949, 683, 640, 621 cm^{-1}.

2.2.3. Preparation of tetrakis(butyrato)dichloridodirhenium (III,III), $Re_2(O_2CC_3H_7)_4Cl_2$ (3). In a glove bag, $[Bu_4N]_2[Re_2Cl_8]$ (0.133 g, 0.117 mmol) was combined with 4 mL of a 3 : 1 mixture by volume of butyric acid/butyric anhydride and 1 mL of hexanes in a 35-mL reaction vessel equipped with a micro stir bar. The vial was placed in the microwave reactor and the temperature was set to 220 °C. The sample was ramped to 220 °C in 3 min and held at 220 °C for 25 min. The maximum pressure reached was 108 psi. The mixture was cooled to 50 °C, removed from the microwave reactor, and the blue supernatant was removed under argon using a syringe. The pink product was washed with hexanes and diethyl ether in the glove box. In the glove bag, the pink solid was combined with 3 mL of a 4 : 1 mixture by volume of butyric acid/butyric anhydride and 1 mL of hexanes in a 35-mL reaction vessel equipped with a micro stir bar. The vial was placed in the microwave reactor and the temperature was set to 235 °C. The sample was ramped to 235 °C in 4.5 min and held at 235 °C for 25 min. The maximum pressure reached was 119 psi. The mixture was cooled to 50 °C, removed from the microwave reactor, and the colorless supernatant was removed under argon using a syringe. The orange powder was washed with diethyl ether in the glove box. Yield: 0.055 g, 60%. Anal. Calcd. for $Re_2O_8C_{16}H_{28}Cl_2$: C, 24.27; H, 3.56. Found: C, 24.253; H, 3.382. IR: 2962, 2935, 2877, 1458, 1419, 1400, 1376 sh, 1350, 1327, 1308, 1265, 1203, 1099, 937 w, 891, 864, 802, 752, 725, 656, 640 cm^{-1}. UV–vis (CH_3CN) λ_{max}: 273, 399, 499 nm.

2.2.4. Preparation of tetrakis(butyrato)dibromidodirhenium (III,III), $Re_2(O_2CC_3H_7)_4Br_2$ (4). In a glove bag, $[Bu_4N]_2[Re_2Br_8]$ (0.101 g, 0.0675 mmol) was combined with 3 mL of a 4 : 1 mixture by volume of butyric acid/butyric anhydride and 1 mL hexanes in a 35-mL reaction vessel equipped with a micro stir bar. The vial was placed in the microwave reactor and the temperature was set to 200 °C. The reaction mixture reached 200 °C in 3 min. Over the course of 5 min, the temperature was ramped to 250 °C and held for 25 min. The maximum pressure reached was 149 psi. The mixture was cooled to 50 °C and removed from the microwave reactor. The green supernatant was removed under argon using a syringe and the tan product was washed with hexanes and diethyl ether in the glove box. In a glove bag, the tan solid was combined with 4 mL of a 4 : 1 mixture by volume of butyric acid/butyric anhydride and 1 mL of hexanes in a 35-mL reaction vessel equipped with a micro stir bar. The vial was placed in the microwave reactor and the temperature was set to 240 °C. The sample was ramped to 240 °C in 7.5 min and was held at 240 °C for 25 min. The maximum pressure reached was 124 psi. The mixture was cooled to 50 °C, removed from the microwave reactor, and the colorless supernatant was removed under argon using a syringe. The orange–brown product was washed with hexanes and diethyl ether in the glove box. The product was dissolved in acetonitrile and layered with diethyl ether to precipitate bright orange–brown crystals. Yield: 0.027 g, 45%. Anal. Calcd. for $Re_2O_8C_{16}H_{28}Br_2$: C, 21.82; H, 3.20. Found: C, 21.754; H, 2.982. IR: 2962, 2935, 2877, 1458, 1439, 1419, 1396, 1350, 1327, 1308, 1265, 1200, 1099, 953 w, 937 w, 899, 860 w, 802, 752, 721, 663, 644 cm^{-1}. UV–vis (CH_3CN) λ_{max}: 253 sh, 307, 414, 501 nm.

2.2.5. Preparation of tetrakis(pivalato)dichloridodirhenium (III,III), $Re_2(O_2CC(CH_3)_3)_4Cl_2$ (5). In the glove box, $[Bu_4N]_2[Re_2Cl_8]$ (0.146 g, 0.128 mmol) was combined with pivalic acid (0.171 g, 1.67 mmol) and 4 mL of diglyme in a 35-mL reaction vessel equipped with a micro stir bar. The vial was placed in the microwave reactor and the temperature was set to 175 °C. The sample was ramped to 175 °C in 2 min. Over the course of 16.5 min, the temperature was increased to 275 °C and held for 8 min. The maximum pressure reached was 141 psi. The mixture was cooled to 50 °C, removed from the microwave reactor, and the dark green supernatant was decanted in the glove bag. The dark orange solid was washed with hexanes and acetone in the glove box. Yield: 0.027 g, 25%. Anal. Calcd. for $Re_2O_8C_{20}H_{36}Cl_2$: C, 28.34; H, 4.25. Found: C, 28.130; H, 4.171. IR: 2970 w, 1485, 1450, 1423, 1385, 1365, 1215, 899, 798, 783, 629 cm^{-1}. UV–vis (CH_2Cl_2) λ_{max}: 277, 402, 498 nm.

2.2.6. Preparation of tetrakis(pivalato)dibromidodirhenium (III,III), $Re_2(O_2CC(CH_3)_3)_4Br_2$ (6). In the glove box, $[Bu_4N]_2[Re_2Br_8]$ (0.155 g, 0.104 mmol) was combined with pivalic acid (0.175 g, 1.71 mmol) and 2.5 mL of diglyme and 1 mL hexanes in a 35-mL reaction vessel equipped with a micro stir bar. The vial was placed in the microwave reactor and the temperature was set to 175 °C. The sample was ramped to 175 °C in

2.5 min. Over the course of 16.5 min, the temperature was increased to 250 °C, where it was held for 8.5 min. The maximum pressure reached was 127 psi. The mixture was cooled to 50 °C, removed from the microwave reactor, and the dark green supernatant was decanted in the glove bag. The dark brown solid was washed with hexanes and acetone in the glove box. Yield: 0.058 g, 60%. Anal. Calcd. for $Re_2O_8C_{20}H_{36}Br_2$: C, 25.65; H, 3.87. Found: C, 25.723; H, 3.682. IR: 2962, 1485, 1450, 1423, 1381, 1365, 1215, 903, 798, 779, 625 cm^{-1}. UV–vis (CH_2Cl_2) λ_{max}: 258 sh, 314, 419, 500 nm.

3. Results and discussion

The present work confirms that tetrakis(carboxylato)dihalidodirhenium (III,III) complexes can be readily synthesized via microwave irradiation. In general, the microwave synthesis allowed for reactions to be completed in significantly shorter timeframes compared to the traditional reflux and with slightly higher yields than previously reported in the cases where yields were explicitly stated (table 1). The products precipitated as the reaction vessel cooled allowing for facile isolation.

3.1. *Synthesis of $Re_2(O_2CCH_3)_4X_2$*

It is not entirely surprising that **1** and **2** were able to be synthesized using the microwave method as they are the most forgiving of the three sets of reactions and able to be

Table 1. Synthetic reaction times, temperature, and yields of **1**–**6**.

Compound	Synthesis	Reaction time	Reactants and solvents used	Temperature of reaction (°C)	Percent yield (%)
1	Microwave	10 s	Acetic acid and anhydride (neat)	226	98
	Traditional Ref. [34]	1 h	Acetic acid and anhydride (neat)	[a]	95
2	Microwave	10 s	Acetic acid and anhydride (neat)	226	92
	Traditional Ref. [34]	1 h	Acetic acid and anhydride (neat)	[a]	84[b]
3	Microwave	50 min	Butyric acid and anhydride, hexanes	220–235	60
	Traditional Ref. [34]	1–48 h	Butyric acid and anhydride	[a]	10–40
4	Microwave	55 min	Butyric acid and anhydride, hexanes	200–250	45
	Traditional Ref. [34]	1–48 h	Butyric acid and anhydride (neat)	[a]	10–40
5	Microwave	25 min	Pivalic acid and diglyme	175–275	25
	Traditional Ref. [12]	48 h	Pivalic acid	[a]	10–40
6	Microwave	25 min	Pivalic acid, diglyme, hexanes	175–250	60
	Traditional Ref. [12]	96 h	Pivalic acid	[a]	54[b]

[a] Reaction mixture refluxed at boiling point of reactant mixture or heated at melt temperature of acid.
[b] Yields obtained by current authors when synthesized according to previously published procedures [12, 34].

traditionally synthesized in low yields in open air [34]. These complexes were synthesized with a 10 s hold time at 226 °C, where traditional reflux requires one hour [34]. The yield of **1** under microwave conditions was 98%, comparable to the traditional synthetic yield of 95%. Experiments involving longer hold times (30 s; 6 min.) at similar temperatures (220 °C; 226 °C) produced lower yields for **1** (87%; 88%).

3.2. *Synthesis of* $Re_2(O_2CC_3H_7)_4X_2$

In the synthesis of **3** and **4**, the liquid carboxylic acid and anhydride reagents used allowed them to also act as solvents. Hexanes were included during the synthesis of **3** and **4** to enable complete precipitation of product as these complexes were found to be more soluble than **1** and **2**. However, where the reactions of **1** and **2** led directly to product in one step, complexes **3** and **4** required two successive irradiation steps with replenishment of acid and anhydride to push the reaction to completion. After irradiating the reaction mixture of **3** for an initial 25 min, a pink solid and blue supernatant resulted, and after irradiating the reaction mixture of **4** a tan solid and green supernatant resulted. Comparison of UV–vis spectra to those previously published indicated that complete conversion to product had not occurred [34]. Removal of the supernatant solutions, addition of fresh acid, anhydride and hexanes, and additional heating were necessary to synthesize the orange and brown solids of **3** and **4** in pure form. With a two-step synthesis, the overall hold times in the microwave (50–55 min) were still shorter than traditional reflux (1–48 h). Microwave synthesis of **3** and **4** led to yields (60%, 45%) that exceeded those obtained through traditional reflux (10–40%) [19].

3.3. *Synthesis of* $Re_2(O_2CC(CH_3)_3)_4X_2$

The tetrakis(pivalato)dihalidodirhenium (III,III) complexes were traditionally synthesized via 2–4 day melt reactions with yields of 10–54% [12, 34]. Microwave irradiation reduced the synthesis times to only 25 min. To obtain a liquid mixture for the microwave reactor, diglyme or a mixture of diglyme with hexanes were used in the microwave synthesis of **5** and **6**. Unlike **3** and **4**, only a single irradiation period was necessary to produce **5** and **6** in high yields (25%; 60%).

4. Conclusion

Six dirhenium carboxylate paddlewheel complexes were prepared via microwave synthesis with yields ranging between 25 and 98%. Reaction times were significantly decreased in the microwave (<1 h) compared to traditional reflux syntheses (1–96 h). The septa snap caps of the microwave reaction vessels were capable of maintaining a sufficiently oxygen-free environment during the reaction heating period to protect the oxygen-sensitive products. These results demonstrate that microwave irradiation is an effective time-saving tool for quadruply bonded dirhenium ligand exchange reactions.

Supplementary material

The supplementary material for this paper is available online at http://dx.doi.org/10.1080/00958972.2015.1077954.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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