Electronic Supplementary Material (ESI) for Chemical Science.

Supporting Information

Multi-step and multi-component organometallic synthesis in one pot using orthogonal mechanochemical reactions

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1. Experimental Section

The experiments were carried out in a Retsch MM400 mill at a frequency of 30 Hz using a 10 mL tungsten carbide milling jar (or 20 mL stainless steel milling jar) and a single ball made of the same material (10 mm). ^{1}H spectra were recorded on a Varian MERCURY plus-300 or plus-400 spectrometer (300 or 400 MHz). Chemical shift (δ) are given in parts per million. The molecular weight was determined using high-resolution mass spectra (HRMS). FT-IR spectra were collected using a Fourier Diagform-Infrared Attenuated Total Reflection PerkinElmer UATR Two spectrometer in the range 400 cm^{-1} to 4000 cm^{-1}. Far-IR spectra in the range 100 cm^{-1} to 500 cm^{-1} were obtained on a Bruker Vertex 70 FT-IR spectrometer. Room temperature powder X-ray diffraction (PXRD) patterns were collected in the 2θ range from 3° to 40° on a Bruker D2 phaser X-ray diffractometer using a Cu-K_α(λ=1.54 Å) source equipped with a LinxEye detector and a nickel filter. The X-ray tube was operating at the power setting of 30 kV and 10 mA power. Data analysis was carried out using the Panalytical X'pert Highscore Plus program. X-ray diffraction data was obtained on Bruker D8 single-crystal X-ray diffractometer equipped with a MoK_α X-ray source and a graphite monochromator. Multi-scan absorption correction (SADABS) was applied. Structures were solved by direct methods and refined using SHELX-97 software.

2. Synthesis of compounds $Re(CO)_5X$ and $Mn(CO)_5X$ 3-4

A mixture of **1** or **2** (100 mg, 0.153 mmol or 0.256 mmol respectively), Oxone® (1.5 equiv) and halide source (2 equiv) was milled using a tungsten carbide ball (10 mm) in a tungsten carbide jar (10 mL) for 5-30 min at a frequency of 30 Hz in Retsch MM400 mixer mill. After that, the reaction mixture was purified by filtration using acetone to remove the insoluble material or by sublimation. In the case of **3c** and **4b**, X-ray quality crystals were obtained from slow evaporation of an acetone solution of the complexes.

3. Synthesis of compound $[Re(CO)_3Br_3](TEA)_2$ 5

Method a:
A mixture of $Re(CO)_5Br$ **3b** (100 mg, 0.246 mmol) and tetraethylammonium bromide (TEAB) (2.5 equiv) was milled using a tungsten carbide ball (10 mm) in a tungsten carbide jar (10 mL) for 3h at a frequency of 30 Hz in a Retsch MM400 mixer mill. After that, the reaction mixture was washed with a solution of hexane-ethyl acetate (50:1 v/v) to remove the unreacted TEAB. Filtration and drying yielded the product (82%).

Method b (one-pot two-step reaction)
A mixture of $Re_2(CO)_{10}$ **1** (50 mg, 0.0766 mmol), Oxone® (1.5 equiv), and tetraethylammonium bromide (TEAB) (7.0 equiv) was milled using a tungsten carbide ball (10 mm) in a tungsten carbide jar (10 mL) for 3h at a frequency of 30 Hz in a Retsch MM400 mixer mill. After that, the reaction mixture was washed with a solution of hexane-ethyl acetate (50:1 v/v) and filtered to remove the unreacted TEAB. The residue was washed and filtered with ethanol. Evaporation of the ethanol yielded the product (84%).

Method c (two-step sequential reaction)
A mixture of $Re_2(CO)_{10}$ **1** (100 mg, 0.153 mmol), Oxone® (1.5 equiv), NaBr (2.0 equiv) was milled using a tungsten carbide ball (10 mm) in a tungsten carbide jar (10 mL) for 5 min at a frequency of 30 Hz in Retsch MM400 mixer mill. After that, TEAB (4.0 equiv) was added and the mixture was milled for 3h. After that, the reaction mixture was washed with a solution of hexane-ethyl acetate (50:1 v/v) and filtered to remove the unreacted TEAB. The residue was washed and filtered with ethanol. Evaporation of the ethanol yielded the product (78%).

4. Synthesis of compound *fac*-PhenRe(CO)$_3$Cl 6

Method a:
A mixture of Re(CO)$_5$Cl **3a** (80 mg, 0.221 mmol) and 1,10-phenanthroline (39.82 mg, 0.221 mmol) was milled using a tungsten carbide ball (10 mm) in a tungsten carbide jar (10 mL) for 3 h at a frequency of 30 Hz in Retsch MM400 mixer mill. After that, the reaction mixture was purified by column chromatography (CH$_2$Cl$_2$:MeOH 30:1) to obtain a yellow solid (68%).

Method b (one-pot two-step reaction)
A mixture of Re$_2$(CO)$_{10}$ **1** (100 mg, 0.153 mmol), oxone (1.0 equiv), NaCl (2.0 equiv) and 1,10-phenanthroline (2.0 equiv) was milled using a tungsten carbide ball (10 mm) in a tungsten carbide jar (10 mL) for 3 h at a frequency of 30 Hz in Retsch MM400 mixer mill. After that, the reaction mixture was purified by column chromatography (CH$_2$Cl$_2$:MeOH 30:1) to obtain the *fac*-PhenRe(CO)$_3$Cl **6** as a yellow solid (56%) and (CH$_2$Cl$_2$:MeOH 10:1) to get the *cis*-[(Phen)$_2$Re(CO)$_2$][ReO$_4$] **7** as a red solid (7%). X-ray quality crystals were obtained from slow evaporation of an acetonitrile solution of the complex.

Method c (two-step sequential reaction)
A mixture of Re$_2$(CO)$_{10}$ **1** (100 mg, 0.153 mmol), oxone (1.0 equiv), NaCl (2.0 equiv) was milled using a tungsten carbide ball (10 mm) in a tungsten carbide jar (10 mL) for 5 min at a frequency of 30 Hz in Retsch MM400 mixer mill. After that, 1,10-phenanthroline (2.0 equiv) was added and the mixture was milled for 3h. The reaction mixture was purified by column chromatography (CH$_2$Cl$_2$:MeOH 30:1) to obtain the *fac*-PhenRe(CO)$_3$Cl **6** as a yellow solid (61%) and (CH$_2$Cl$_2$:MeOH 10:1) to get the *cis*-[(Phen)$_2$Re(CO)$_2$][ReO$_4$] **7** as a red solid (4%).

5. Synthesis of compound *fac*-BipyMn(CO)$_3$Cl 8

A mixture of Mn(CO)$_5$Cl **4a** (111 mg, 0.482 mmol) and 2,2'-bipyridine (1.0 equiv) was milled using a tungsten carbide ball (10 mm) in a tungsten carbide jar (10 mL) for 3 h at a frequency of 30 Hz in Retsch MM400 mixer mill. After that, the reaction mixture was purified by column chromatography (CH$_2$Cl$_2$:MeOH 20:1) to obtain a yellow solid (65%).

6. Synthesis of compound *fac*-(TMEDA)Re(CO)$_3$F 10

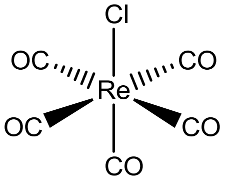
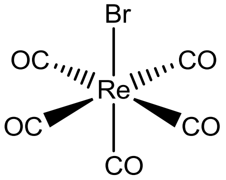
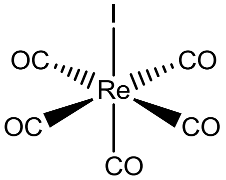
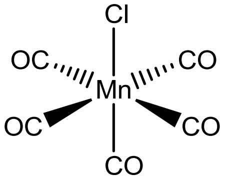
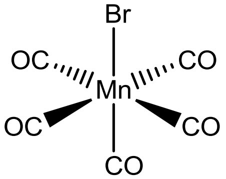
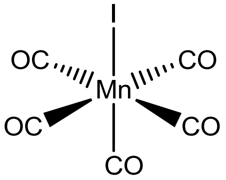
Method a:
A mixture of Re(CO)$_5$I **3c** (100 mg, 0.220 mmol) and AgF (1.1 equiv) was milled using a tungsten carbide ball (10 mm) in a tungsten carbide jar (10 mL) for 30 min at a frequency of 30 Hz. Immediately after that, the N,N,N′,N′-tetramethylethylenediamine (TMEDA) (1.1 equiv) was added and the milling was continued for 1.5 h. The reaction mixture was purified by column chromatography (CH$_2$Cl$_2$:MeOH 50:1) to obtain an off-white solid (70%).

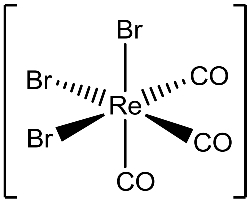
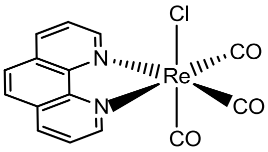
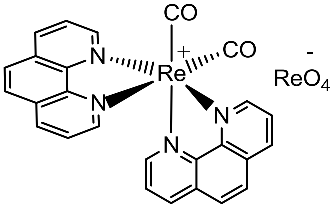
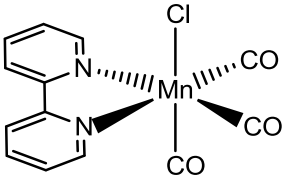
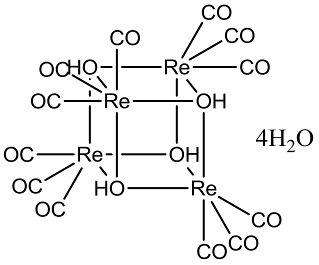
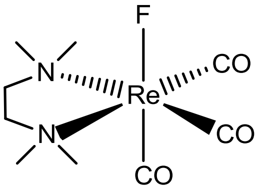
Method b (one-pot reaction starting from Re(CO)$_5$I)**:**
A mixture of Re(CO)$_5$I **3c** (100 mg, 0.220 mmol), AgF (1.1 equiv) and N,N,N′,N′-tetramethylethylenediamine (TMEDA) (1.1 equiv) was milled using a tungsten carbide ball (10 mm) in a tungsten carbide jar (10 mL) for 2h at a frequency of 30 Hz. The reaction mixture was purified by filtration using acetone to obtain an off-white solid (79%).

Method c (one-pot reaction starting from Re$_2$(CO)$_{10}$)**:**
A mixture of Re$_2$(CO)$_{10}$ **1** (100 mg, 0.153 mmol), oxone (1.0 equiv), NaI (2.0 equiv), AgF (4.4 equiv) and N,N,N′,N′-tetramethylethylenediamine (TMEDA) (2.2 equiv) was milled using a tungsten

carbide ball (10 mm) in a tungsten carbide jar (10 mL) for 2 h at a frequency of 30 Hz in Retsch MM400 mixer mill. The reaction mixture was purified by filtration using acetone to obtain an off-white solid (45%).

7. Selected FTIR-ATR and MS data

$Re(CO)_5Cl$ 3a 	**FTIR-ATR** v_{max} 2156 ,2033, 1976, 1958, 283 cm^{-1}. **MS**: m/z $[M^+]$ 359.85, 361.82, 363.83
$Re(CO)_5Br$ 3b 	**FTIR-ATR** v_{max} 2153, 2022, 1975, 1959, 202 cm^{-1}. **MS**: m/z $[M^+]$ 403.64, 405.60, 407.60
$Re(CO)_5I$ 3c 	**FTIR-ATR** v_{max} 2150, 2020, 1983, 1946, 161 cm^{-1}. **MS**: m/z $[M^+]$ 451.54, 453.56, 454.58
$Mn(CO)_5Cl$ 4a 	**FTIR-ATR** v_{max} 2045, 1984, 1951 cm^{-1}. **MS**: m/z $[2M\text{-}Cl_2]$ =389.8
$Mn(CO)_5Br$ 4b 	**FTIR-ATR** v_{max} 2033, 1985, 1952 cm^{-1}. **MS**: m/z $[2M\text{-}Br_2]$ =389.7
$Mn(CO)_5I$ 4c 	**FTIR-ATR** v_{max} 2130, 1977 (br) cm^{-1}. **MS**: m/z $[2M\text{-}I_2]$ =389.8

$[Re(CO)_3Br_3]TEA_2$ 5  	**FTIR-ATR** v_{max} 2987, 1997, 1848 $cm^{-1.}$ HRMS: (positive mode) Calculated for TEA^+ $(C_8H_{20}N)^{1+}$ [M]: 130.1596; found: 130.1594; (negative mode). Calculated for $(C_3O_3Br_3Re)^{-2}$ [M/2]: 254.9747; found 255.2328.
***fac*-PhenRe(CO)$_3$Cl 6**  	**FTIR-ATR** v_{max} 2956, 2922, 2852, 2012, 1922, 1885, 1861cm^{-1}. **^{1}H-NMR** CD_3CN (δ) 9.39 (dd, 2H, *J*=5.13, 1.32 Hz), 8.76 (dd, 2H, *J*=8.31, 1.41Hz), 8.18 (s, 2H), 7.97 (dd, 2H, *J*=8.31, 5.1 Hz) ppm. HRMS: Calculated for $C_{15}H_8ClN_2O_3Re$ [M+Na]: 508.9669; found: 508.9664.
***cis*- [(Phen)$_2$Re(CO)$_2$][ReO$_4$] 7**  	**FTIR-ATR** v_{max} 2958, 2922, 2852 1897, 1821 cm^{-1}. **^{1}H-NMR** CD_3CN (δ) 9.92 (dd, 1H, *J*=5.5, 1.2 Hz), 8.76 (dd, 1H, *J*=8.25, 1.2 Hz), 8.49 (dd, 1H, *J*=8.21, 1.31 Hz), 8.22 (d, 1H, *J*=8.87 Hz), 8.10 (d, 1H, *J*=8.84 Hz), 8.03 (dd, 1H, *J*=8.20, 5.15 Hz), 7.58 (dd, 1H, *J*=5.10, 1.34 Hz), 7.46 (dd, 1H, *J*=8.26, 5.17 Hz) ppm. HRMS (positive mode): Calculated for $C_{26}H_{16}N_4O_2Re$ [M^+]: 603.0831; found: 603.0819. HRMS (negative mode): Calculated for ReO_4 [M^-]: 250.9360; found: 250.9349.
***fac*-BipyMn(CO)$_3$Cl 8**  	**FTIR-ATR** v_{max} 3060, 2924, 2856, 2020, 1908, 1888 cm^{-1}. **^{1}H-NMR** CD_3CN (δ) 9.19 (d, 2H, *J*=5.2), 8.33 (d, 2H, *J*=8.0, 1.2 Hz), 8.11 (t, 2H, *J*=6.80 Hz), 7.61 (d, 2H, *J*=6.40 Hz) ppm. HRMS (positive mode): Calculated for $C_{13}H_8ClN_2O_3MnNa$ [M+Na]: 352.9496; found: 352.9493.
[Re(CO)$_3$OH]$_4$·4H$_2$O 9  	**FTIR-ATR** v_{max} 3575, 3215, 2028, 1919, 1889 cm^{-1}. **^{1}H-NMR** $(CD_3)_2CO$ (δ) 5.00-3.50 (br-s) ppm, **^{13}C-NMR** $(CD_3)_2CO$ (δ) 197.23 ppm. HRMS (negative mode): Calculated for $C_{12}H_3O_{16}Re_4$ [M-1H]: 1148.7488; found: 1148.7463.
***fac*-(TMEDA)Re(CO)$_3$F 10**  	**FTIR-ATR** v_{max} 2921, 2850, 2005, 1855 cm^{-1}. **^{1}H-NMR** $CDCl_3$ (δ) 3.07 (2, 6H), 2.94-2.81 (m, 2H), 2.79 (d, 6H, J=1.8 Hz), 2.76-2.56 (2H) ppm. **^{19}F-NMR** $CDCl_3$ (δ) -263.28 (s) ppm (referenced to the internal standard C_6F_6 signal -164.9 ppm); acetone-d_6 (δ) -256.01 (s) ppm. HRMS: Calculated for $C_9H_{16}FN_2O_3Re$ [M+Na]: 429.0600; found: 429.0589.

8. Selected FTIR-ATR spectra

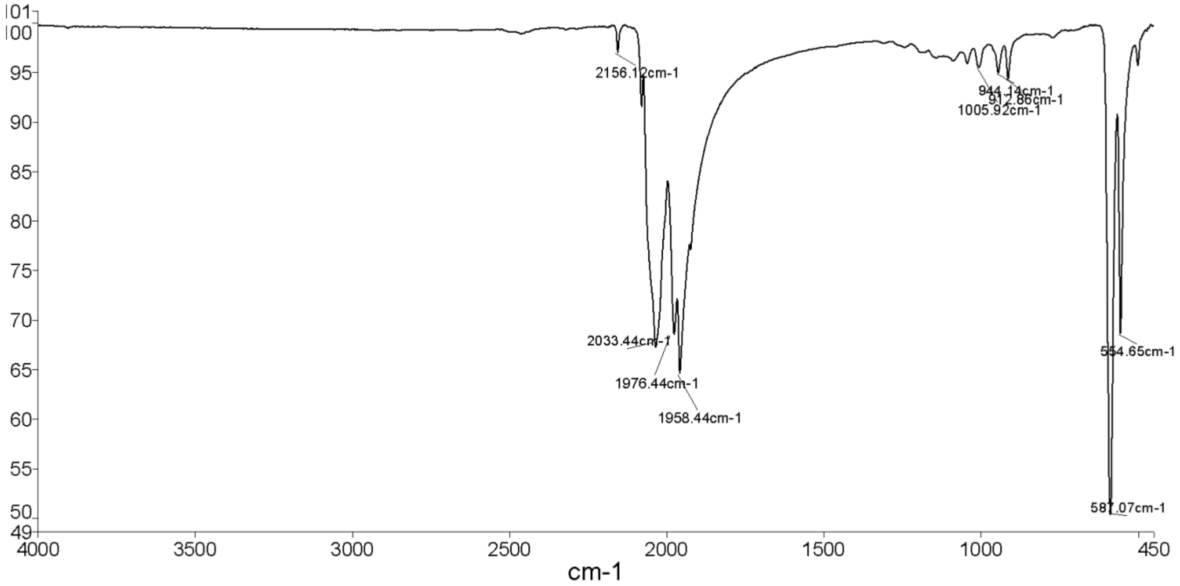


Figure S1. FTIR-ATR spectrum of Re(CO)$_5$Cl **3a**

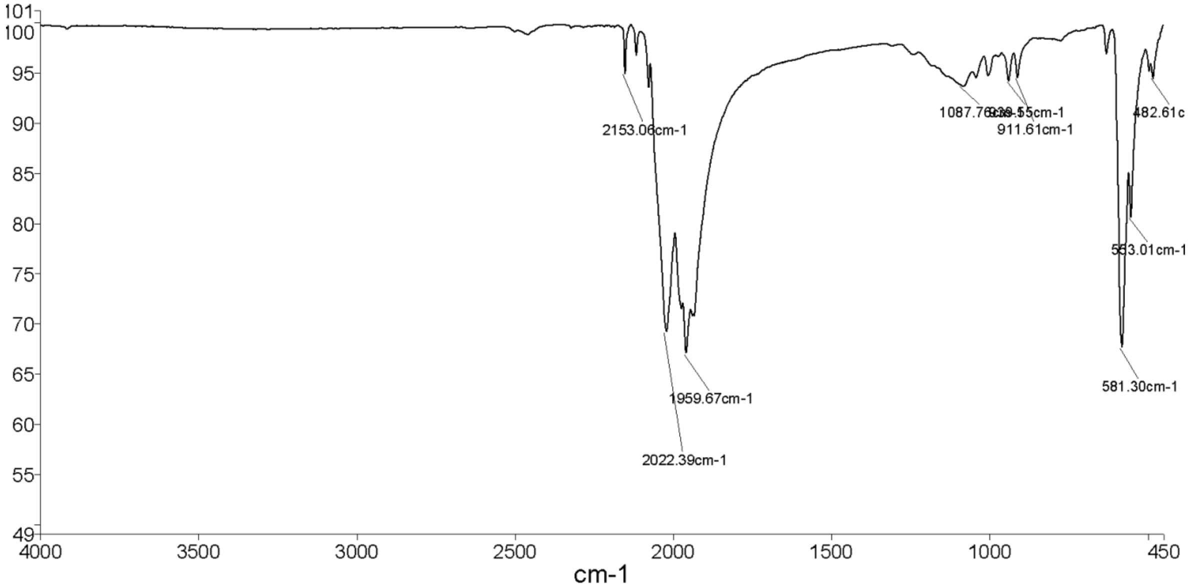


Figure S2. FTIR-ATR spectrum of Re(CO)$_5$Br **3b**

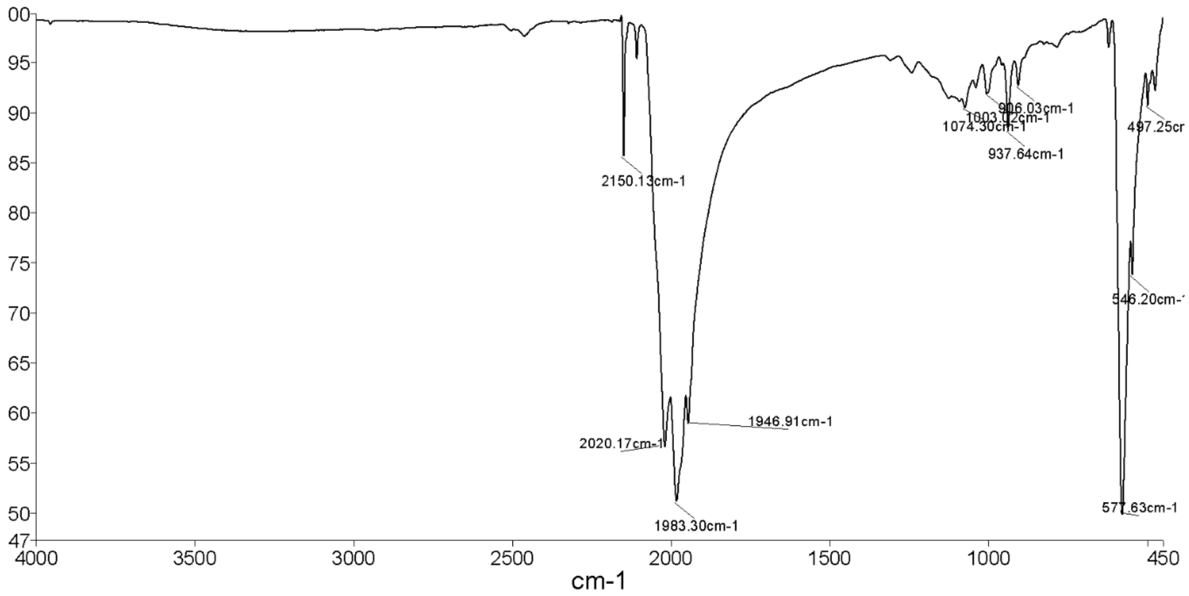


Figure S3. FTIR-ATR spectrum of $Re(CO)_5I$ **3c**

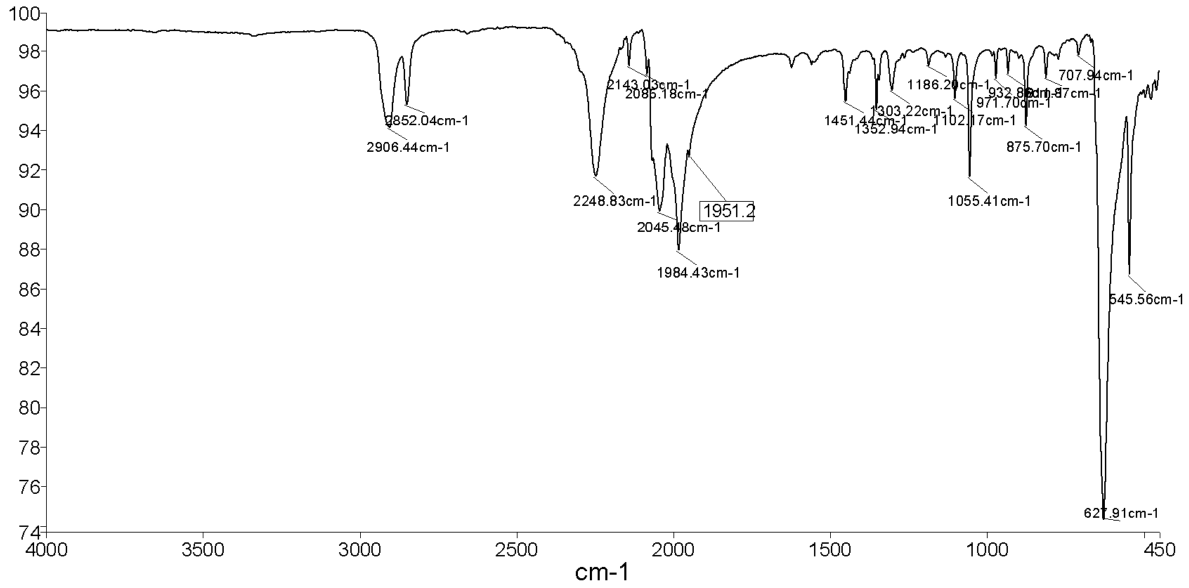


Figure S4. FTIR-ATR spectrum of $Mn(CO)_5Cl$ **4a**

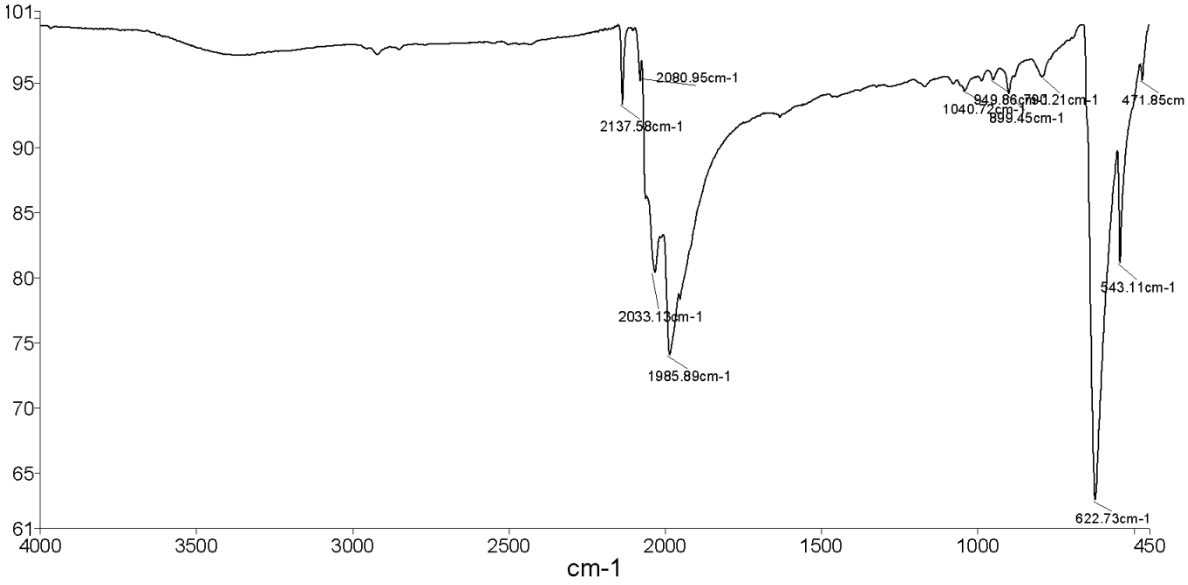


Figure S5. FTIR-ATR spectrum of $Mn(CO)_5Br$ **4b**

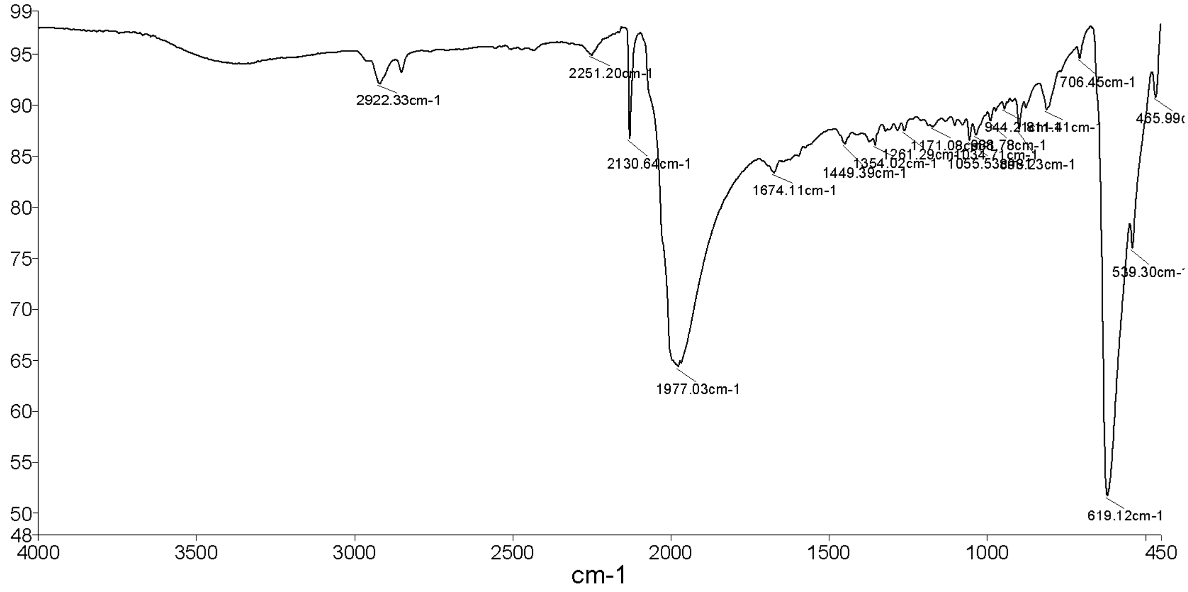


Figure S6. FTIR-ATR spectrum of $Mn(CO)_5I$ **4c**

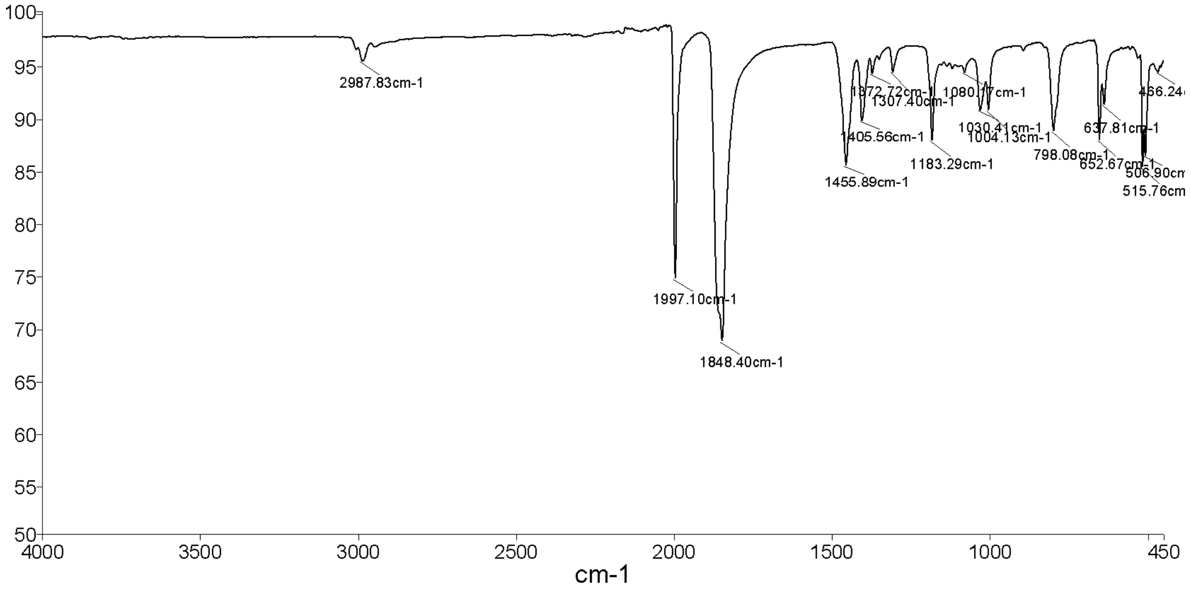


Figure S7. FTIR-ATR spectrum of [Re(CO)$_3$Br$_3$]TEA$_2$ **5**

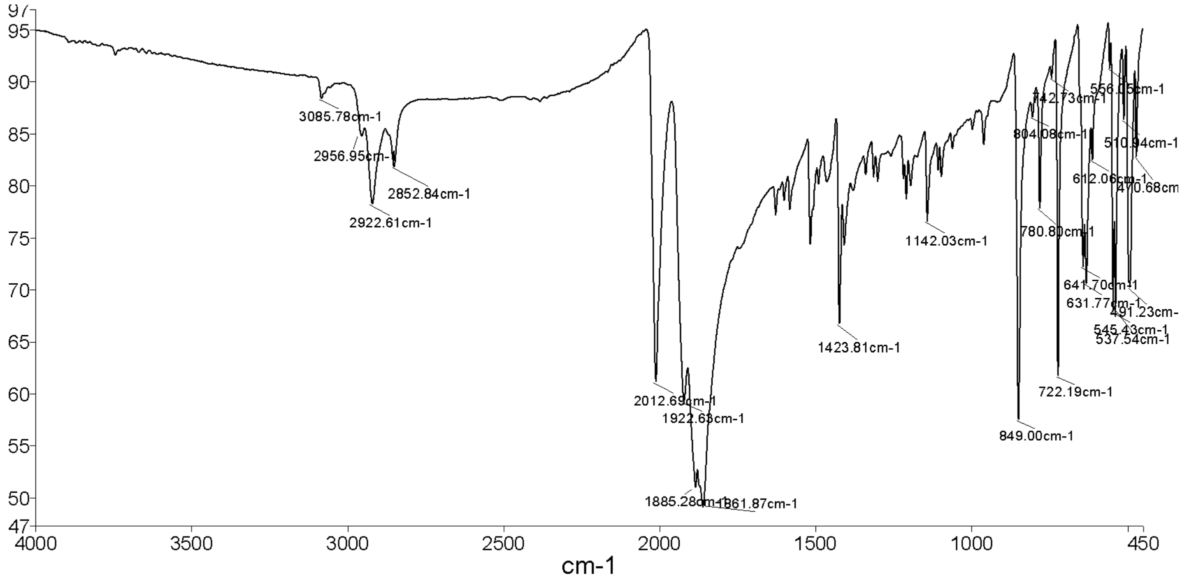


Figure S8. FTIR-ATR spectrum of *fac*-PhenRe(CO)$_3$Cl **6**

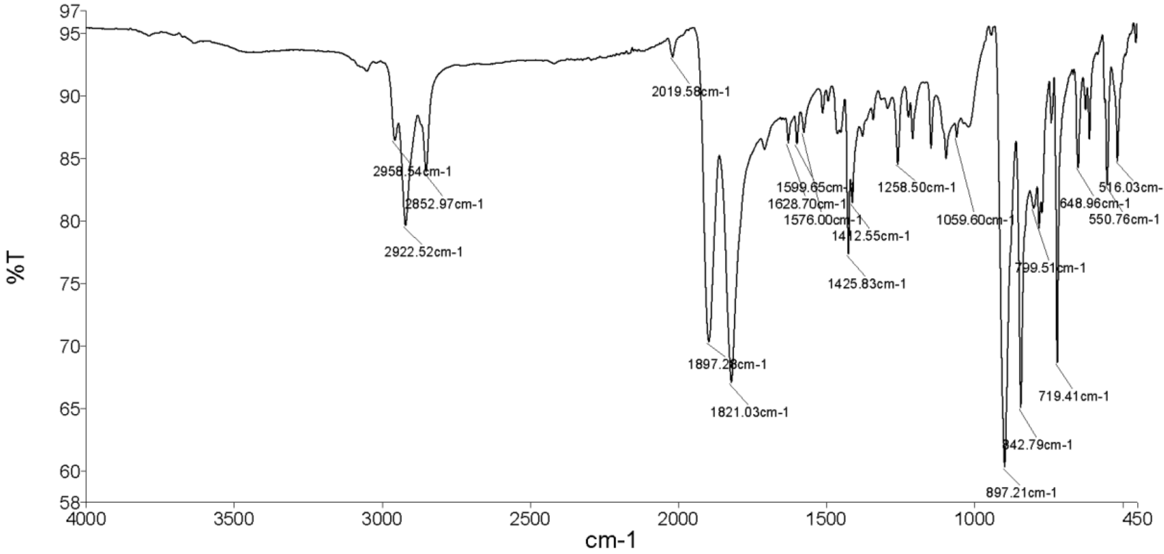


Figure S9. FTIR-ATR spectrum of *cis*-[(Phen)$_2$Re(CO)$_2$][ReO$_4$] **7**

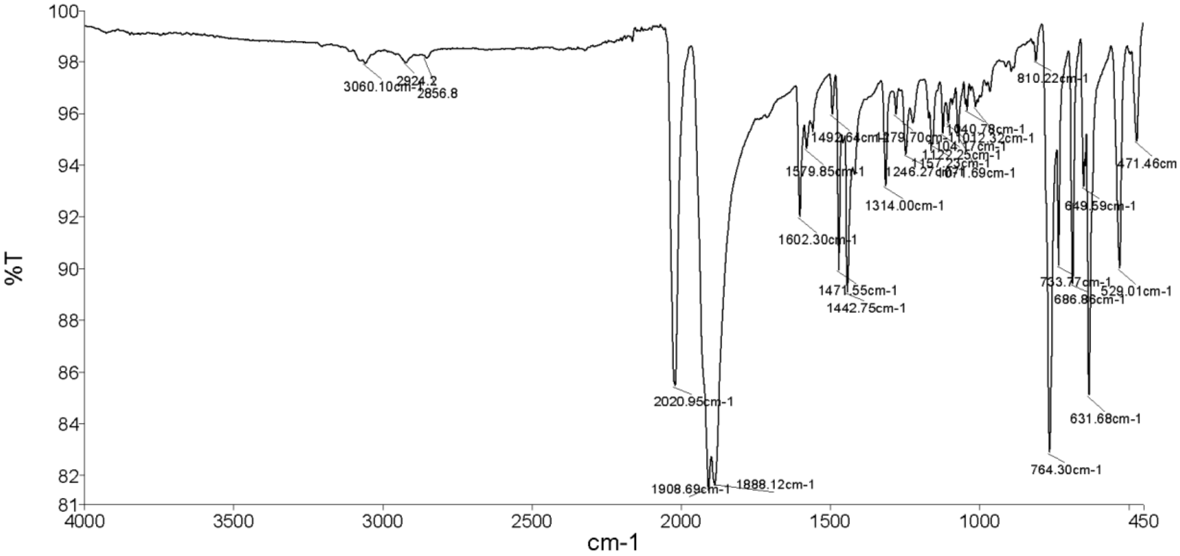


Figure S10. FTIR-ATR spectrum of *fac*-(Bipy)Mn(CO)$_3$Cl **8**

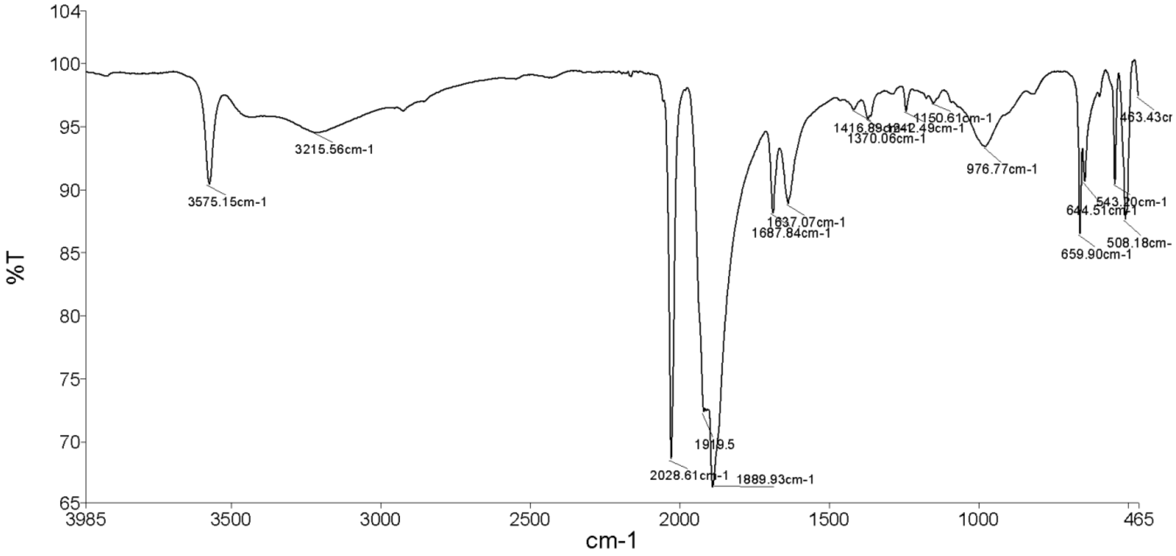


Figure S11. FTIR-ATR spectrum of $[Re(CO)_3OH]_4 \cdot 4H_2O$ **9**

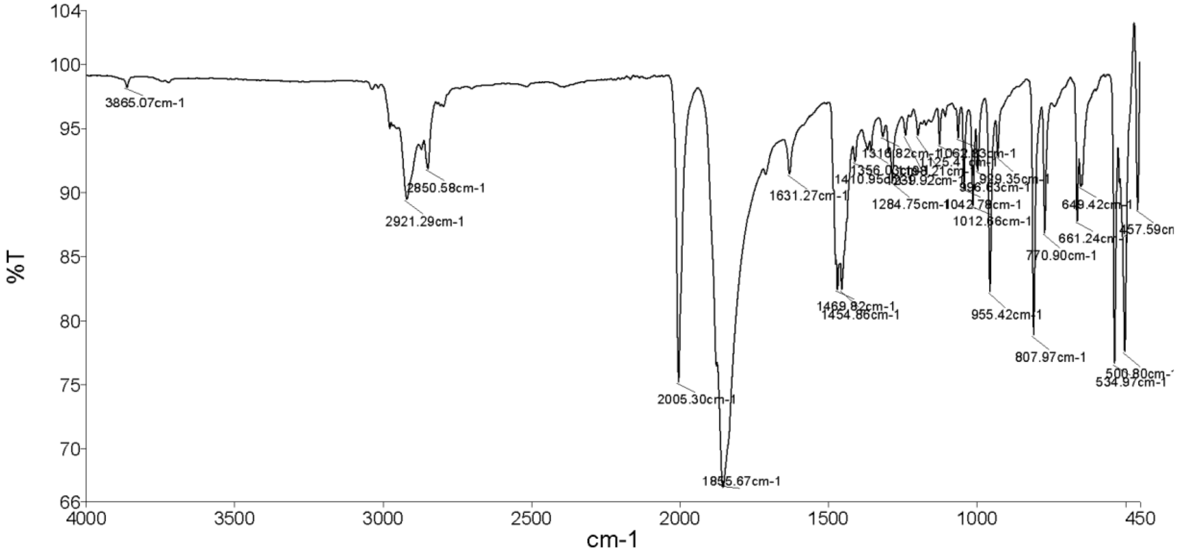


Figure S12. FTIR-ATR spectrum of $(TMEDA)Re(CO)_3F$ **10**

9. Selected ^{1}H, ^{19}F and ^{13}C NMR spectra

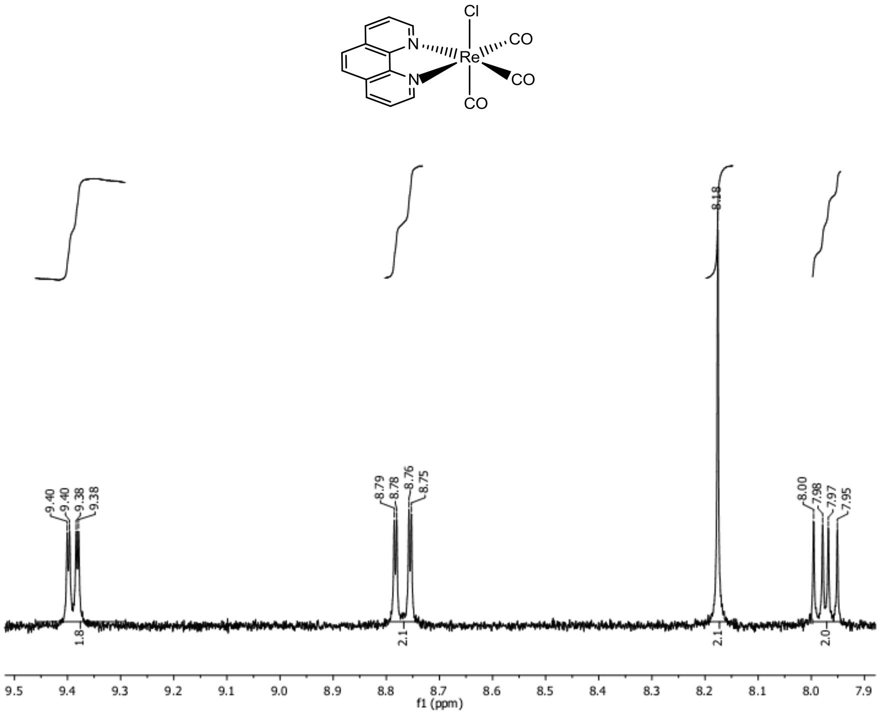


Figure S13. ^{1}H NMR spectrum of *fac*-PhenRe(CO)$_3$Cl **6**

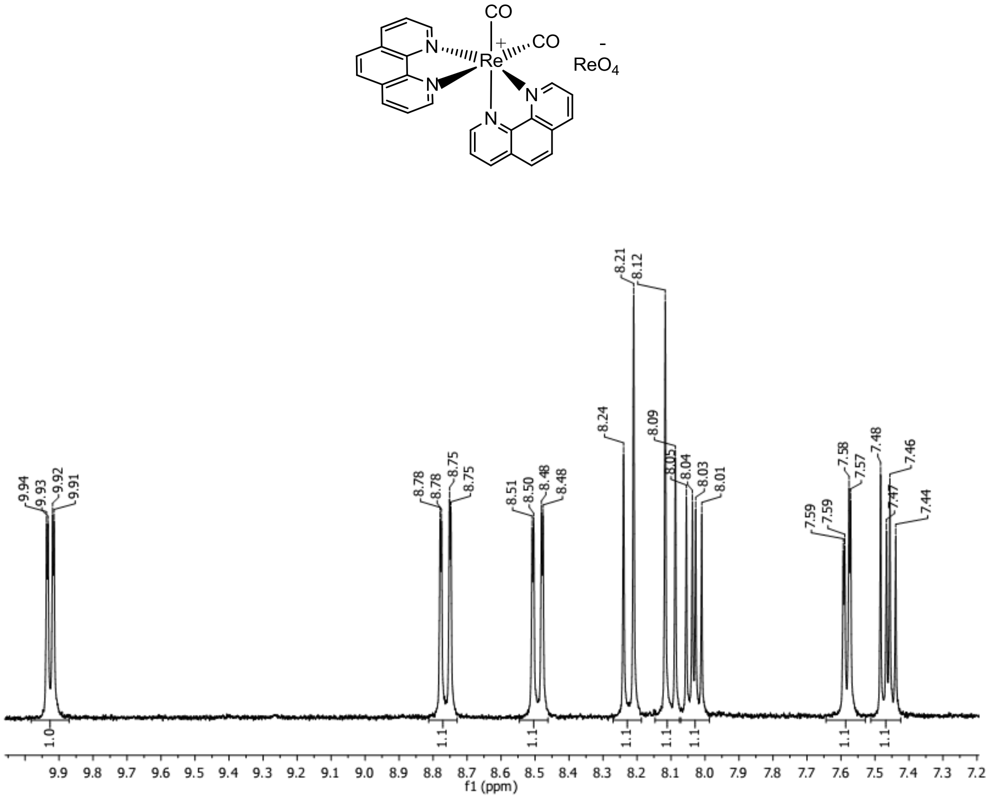


Figure S14. ^{1}H NMR spectrum of *cis*-[(Phen)$_2$Re(CO)$_2$][ReO$_4$] **7**

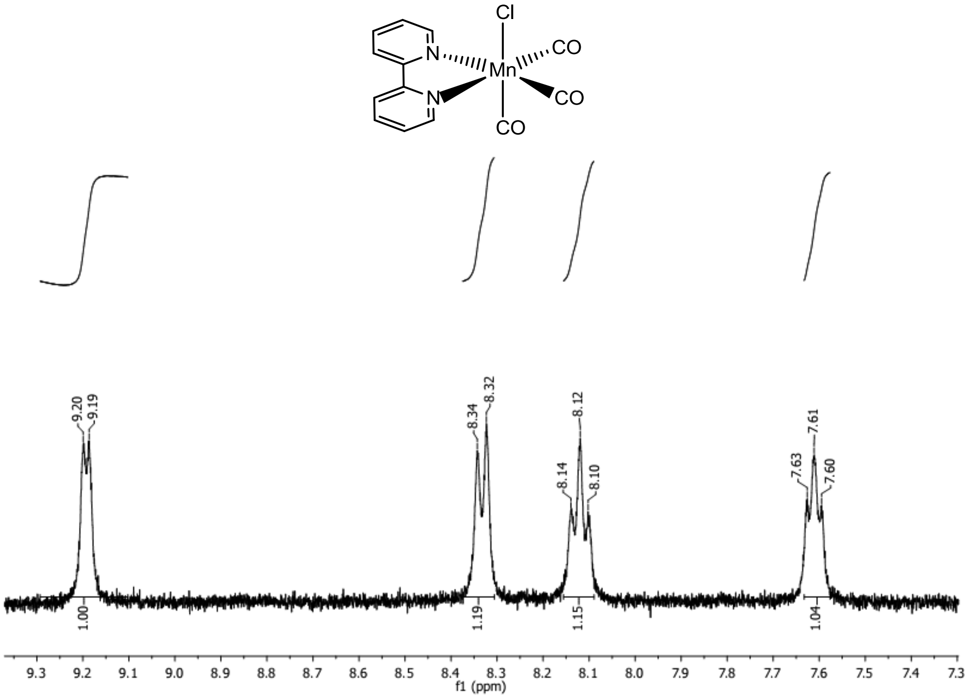


Figure S15. ^{1}H NMR spectrum of *fac*-(2,2'Bipy)Mn(CO)$_3$Cl **8**

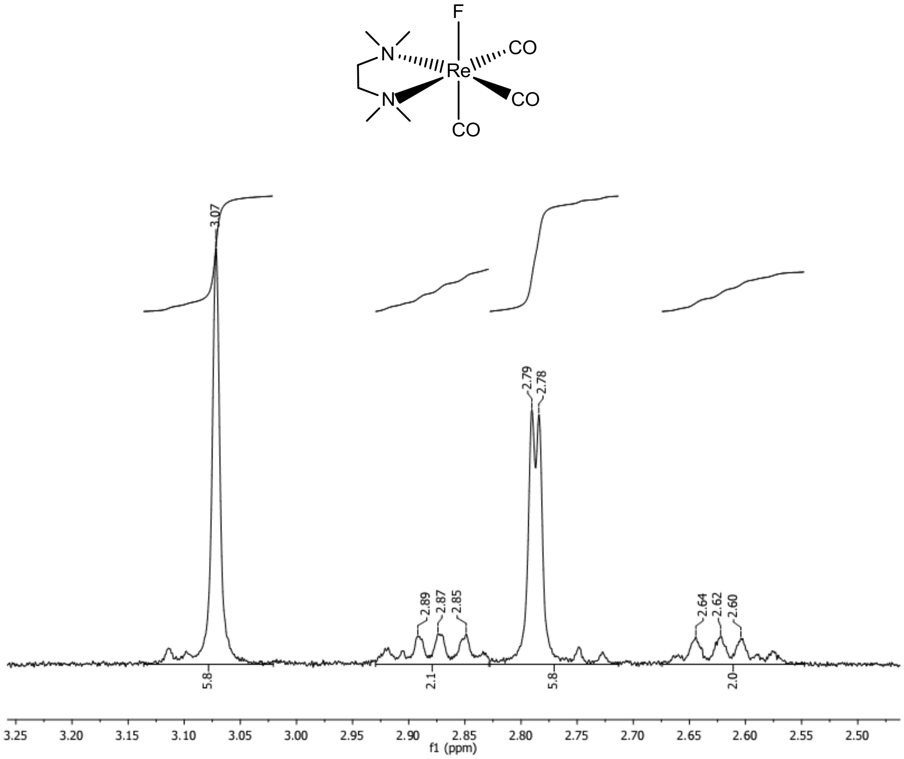


Figure S16. ^{1}H NMR spectrum of (TMEDA)Re(CO)$_3$F **10**

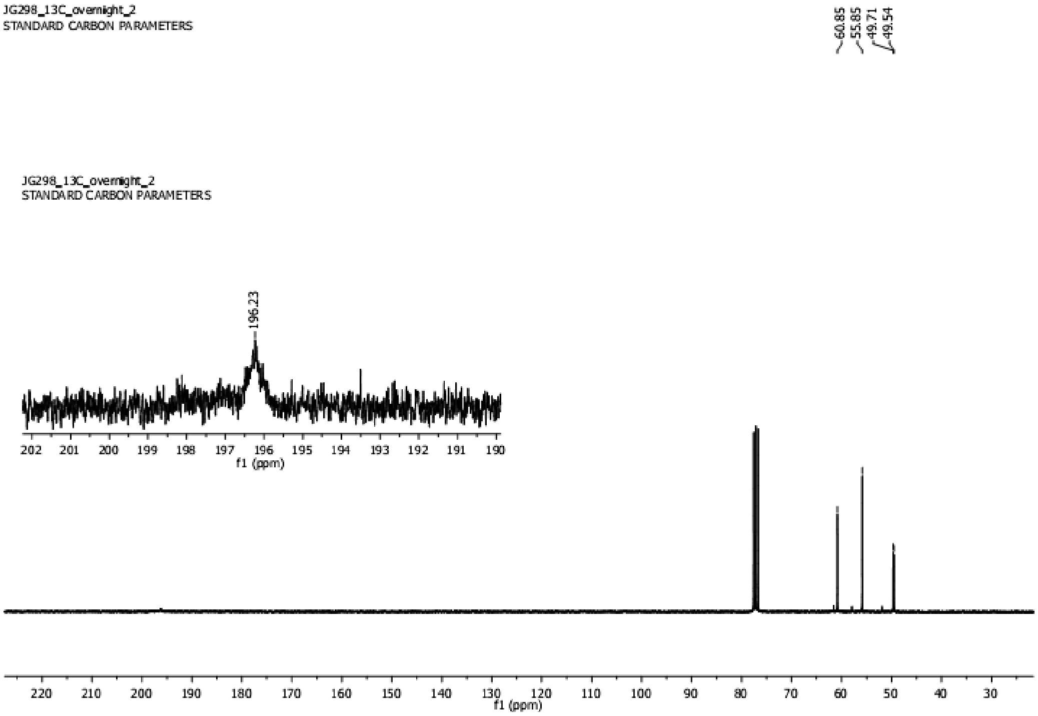


Figure S17. ^{13}C NMR spectrum of (TMEDA)Re(CO)$_3$F **10**

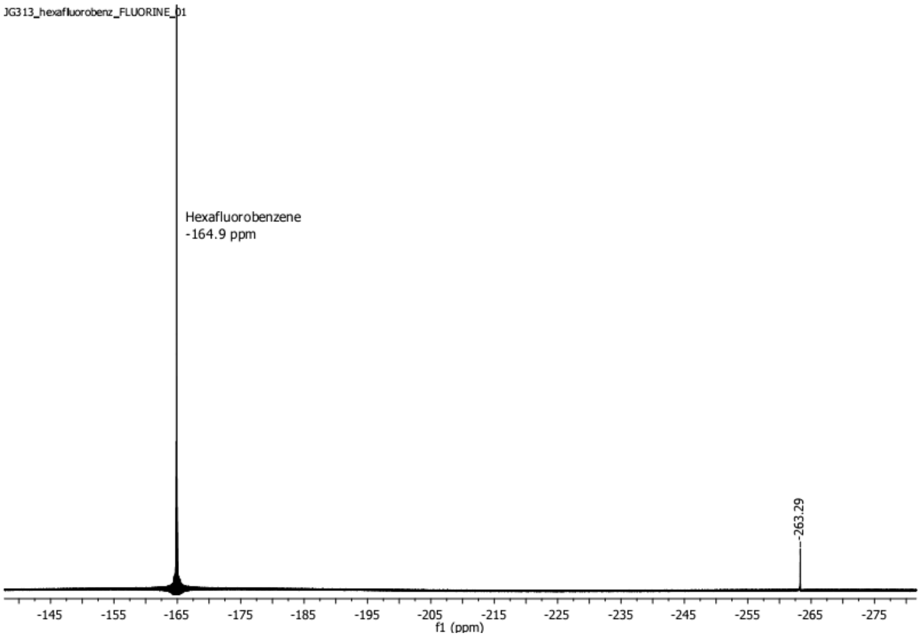
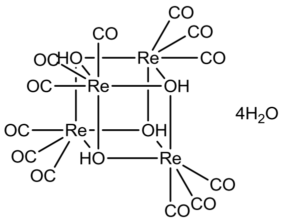


Figure S18. ^{19}F NMR spectrum of (TMEDA)Re(CO)$_3$F **10** in CDCl$_3$.



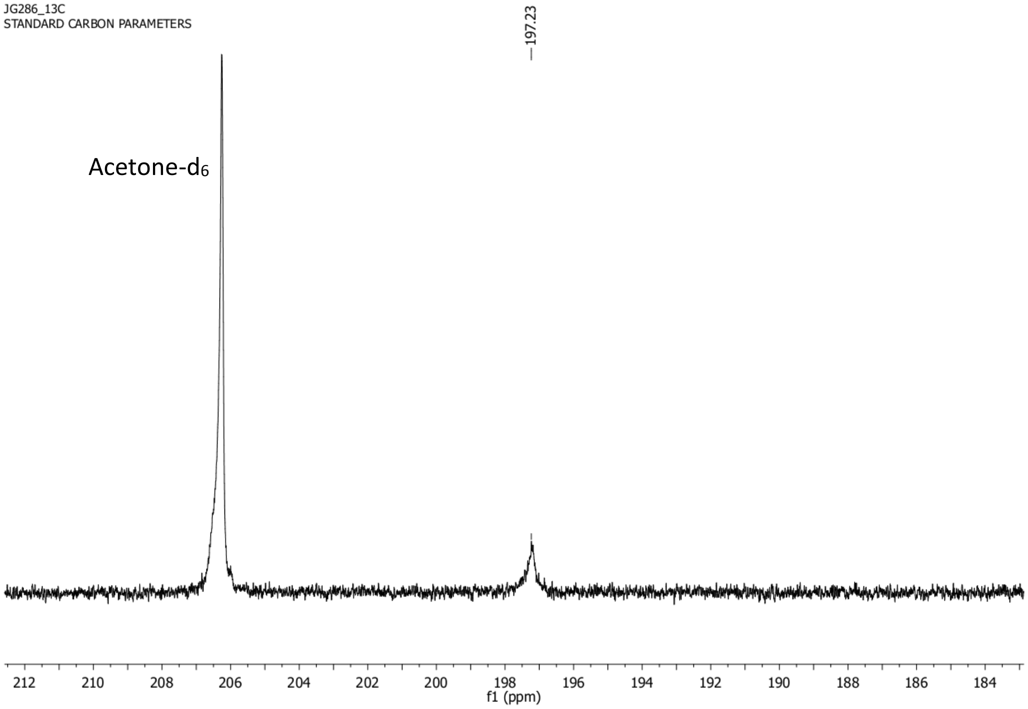
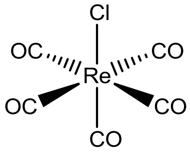


Figure S19. ^{13}C NMR spectrum of $[Re(CO)_3OH]_4\cdot 4H_2O$ **9** in acetone-*d*$_6$

10. Comparison of experimental and simulated PXRD patterns

$Re(CO)_5Cl$ 3a



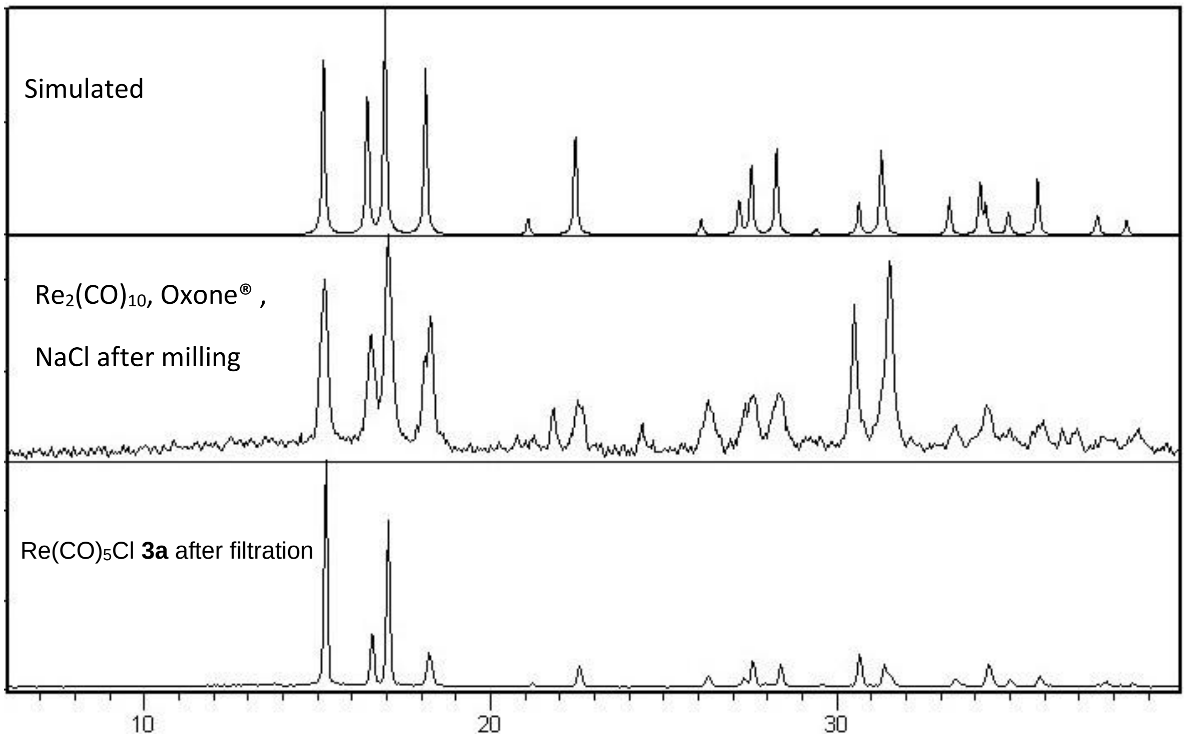
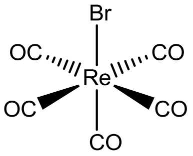


Figure S20. (Top) Simulated PXRD pattern for $Re(CO)_5Cl$ **3a** (*Acta Cryst.* (**1983**). *C39*, 1495-1496); (middle) measured PXRD pattern for the mixture of $Re_2(CO)_{10}$, Oxone® and NaCl after milling; (bottom) measured PXRDpattern for $Re(CO)_5Cl$ **3a** after filtration

Re(CO)$_5$Br 3b



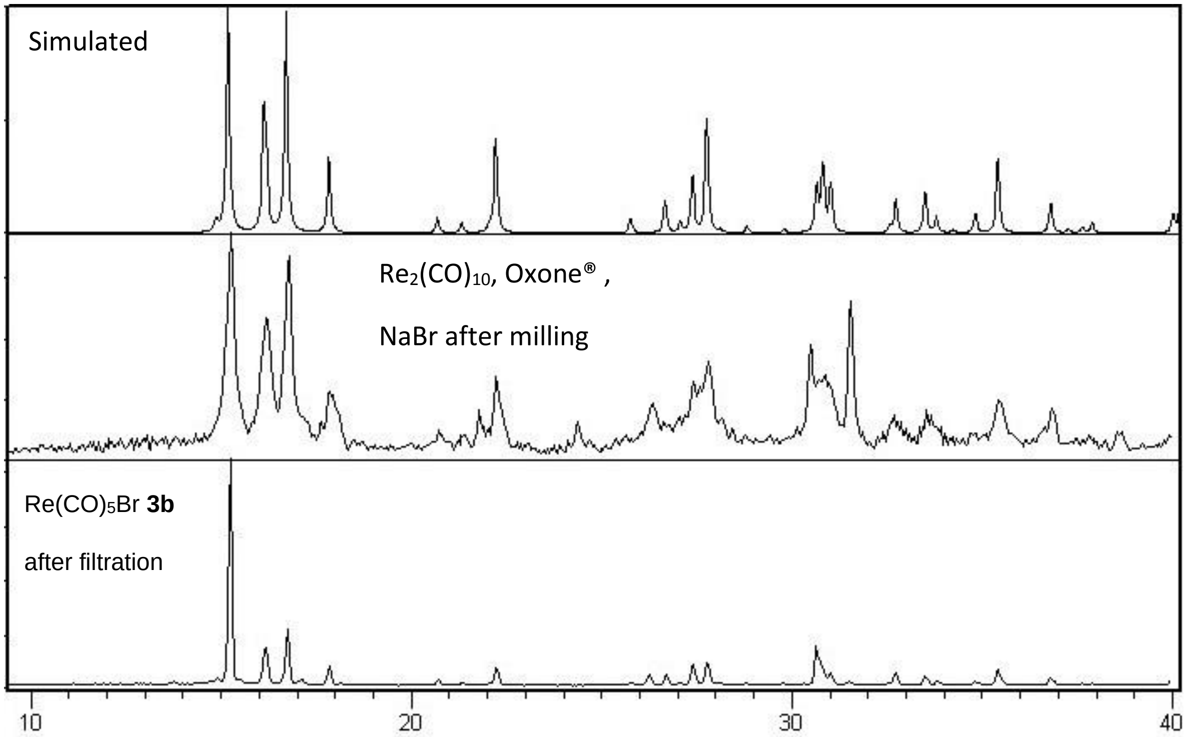
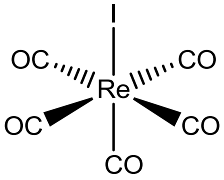


Figure S21. (Top) Simulated PXRD pattern of Re(CO)$_5$Br **3b** (*Cryst.Struct.Commun*. **1977**, *6,* 1); (middle) measured PXRD pattern for the mixture of Re$_2$(CO)$_{10}$, Oxone® and NaBr after milling; (bottom) measured PXRD patterns for Re(CO)$_5$Br **3b** after filtration

Re(CO)$_5$I 3c



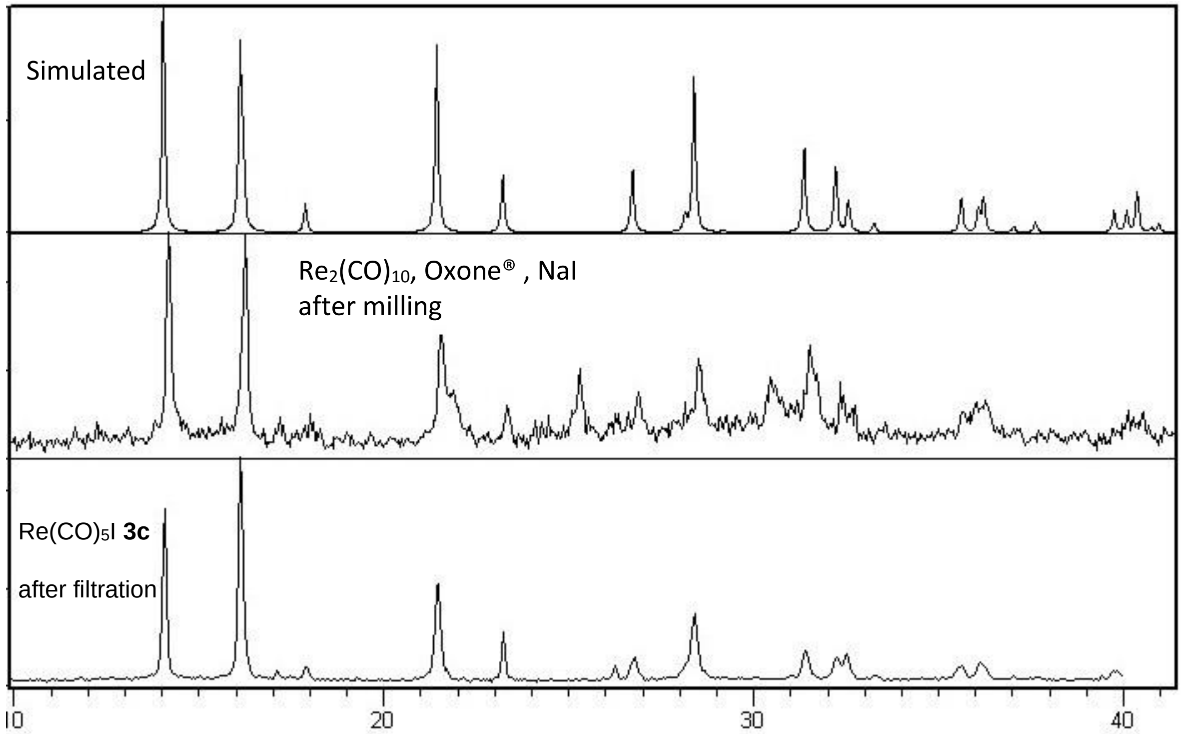
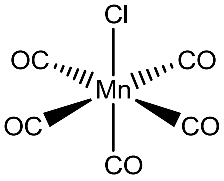


Figure S22. (Top) Simulated PXRD pattern for Re(CO)$_5$I **3c** (from this work, see also *Faraday Trans.*, **1991**, *87,* 1831-1836); (middle) measured PXRD pattern for a mixture of Re$_2$(CO)$_{10}$, Oxone® and NaI after milling; (bottom) measured for Re(CO)$_5$I **3c** after filtration

Mn(CO)$_5$Cl **4a**



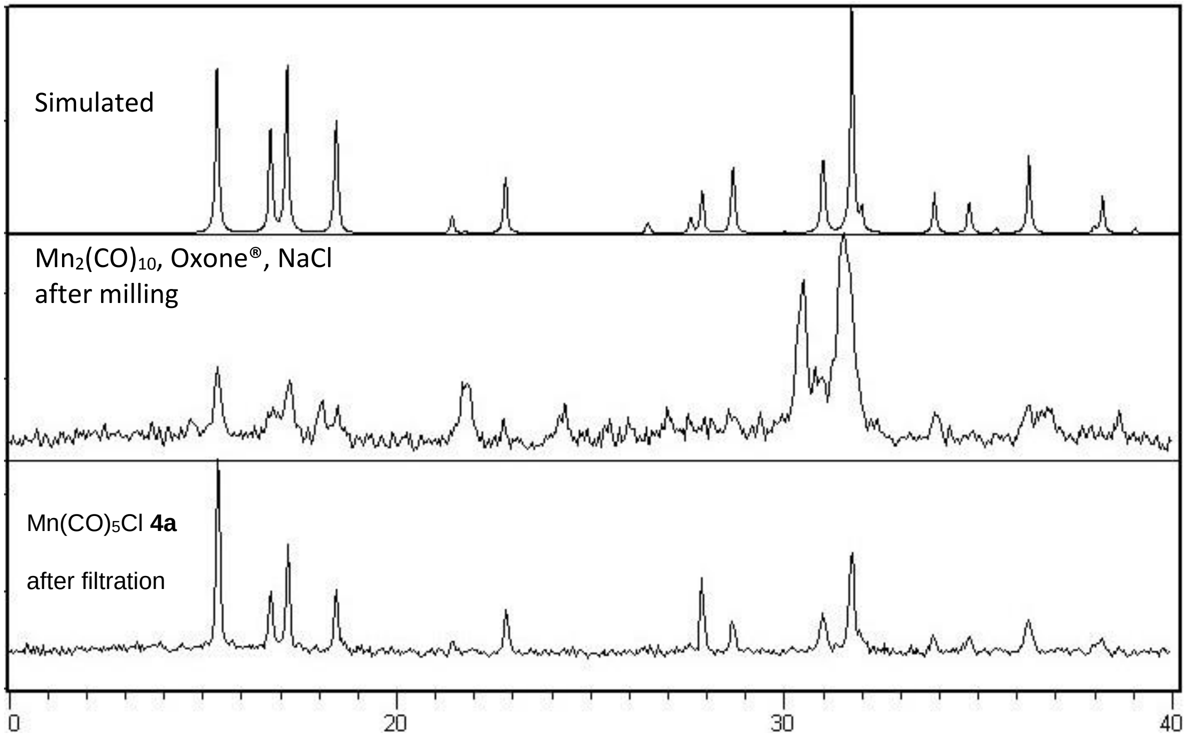
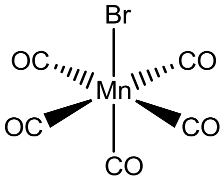


Figure S23. (Top) Simulated PXRD pattern for Mn(CO)$_5$Cl **4a** (*J. Chem. Soc. A*, **1971**, 1559-1562); (middle) measured PXRD pattern for a mixture of Mn$_2$(CO)$_{10}$, Oxone® and NaCl after milling; (bottom) measured for Mn(CO)$_5$Cl **4a** after filtration.

$Mn(CO)_5Br$ **4b**



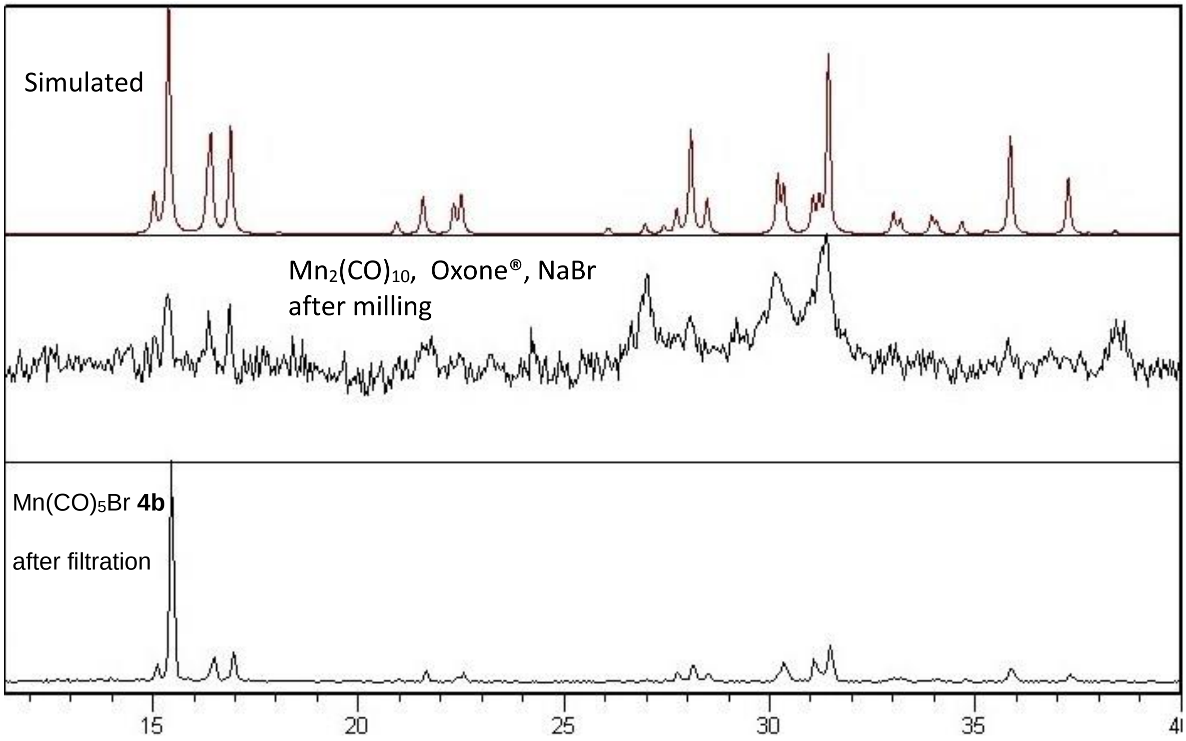
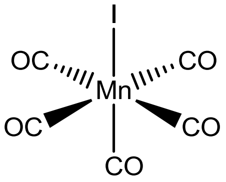


Figure S24. (Top) Simulated PXRD pattern for $Mn(CO)_5Br$ **4b** (*this work*), (middle) measured PXRD pattern for a mixture of $Mn_2(CO)_{10}$, Oxone® and NaBr after milling; (bottom) measured for $Mn(CO)_5Br$ **4b** after filtration

$Mn(CO)_5I$ **4c**



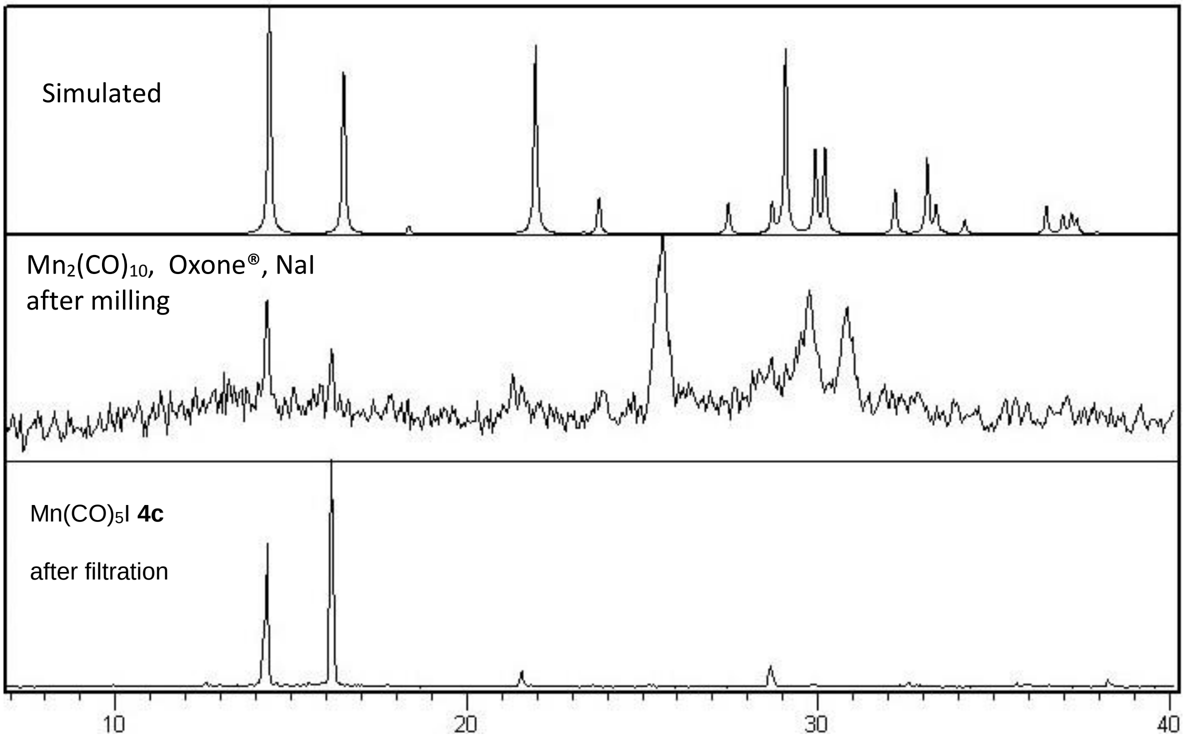
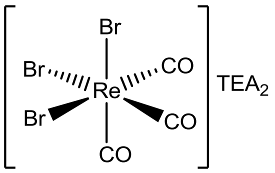


Figure S25. (Top) Simulated PXRD pattern for $Mn(CO)_5I$ **4c** (*Acta Cryst.* **1992,** *C48,* 1708-1709); (middle) measured PXRD pattern for $Mn_2(CO)_{10}$, Oxone® and NaI after milling; (bottom) measured for $Mn(CO)_5I$ **4c** after filtration

[Re(CO)$_3$Br$_3$]TEA$_2$ 5



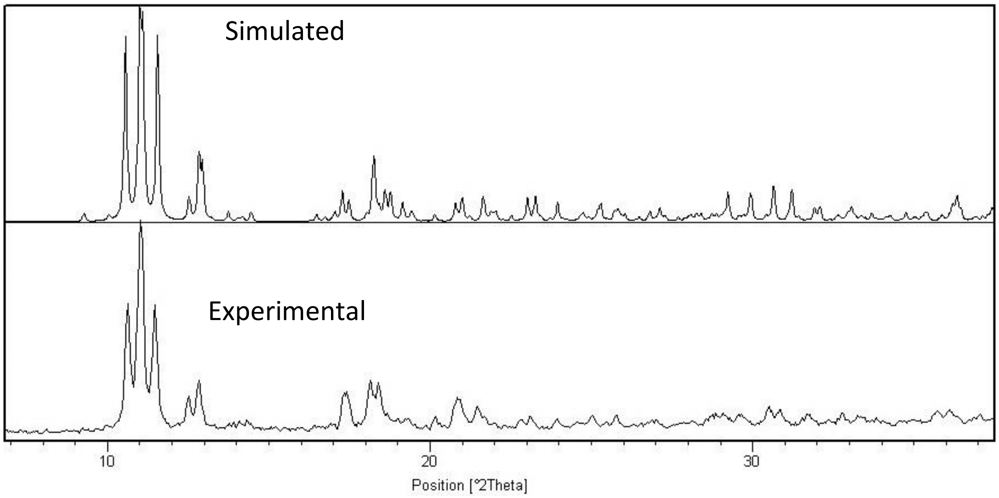
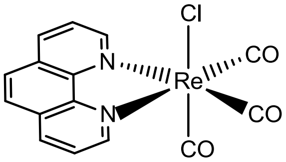


Figure S26. (Top) Simulated PXRD pattern for [Re(CO)$_3$Br$_3$]TEA$_2$ **5** (*J. Organomet. Chem.* **1995**, *493, 119-127*); (bottom) measured PXRD for the product of mechanochemical synthesis

PhenRe(CO)$_3$Cl 6



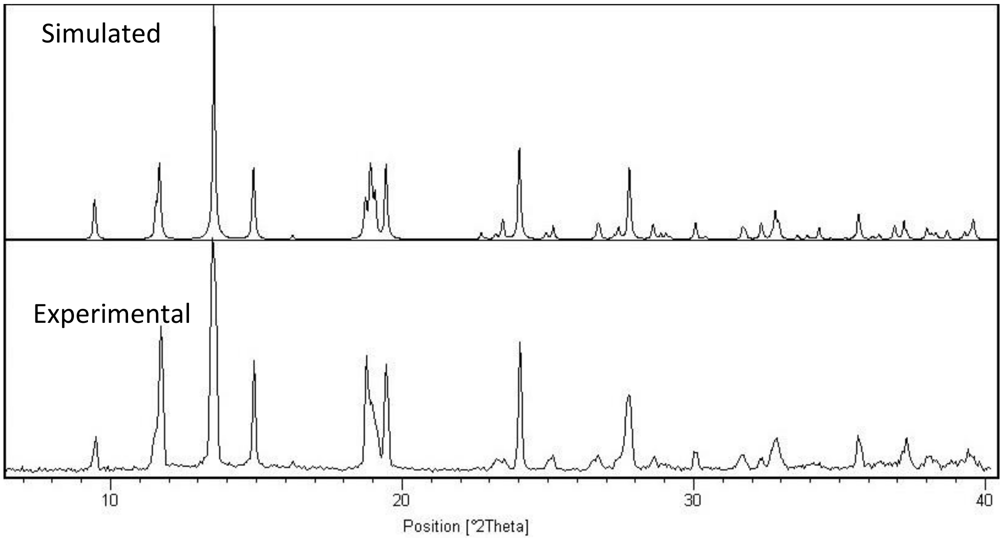
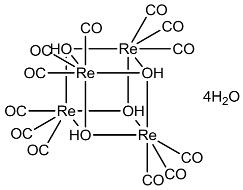


Figure S27. (Top) Simulated PXRD pattern for (Phen)Re(CO)$_3$Cl **6** (*Eur. J. Inorg. Chem.* ***2005****. 118-124*); (bottom) measured PXRD pattern for the mechanochemical product

$[Re(CO)_3OH]_4{\cdot}4H_2O$ 9



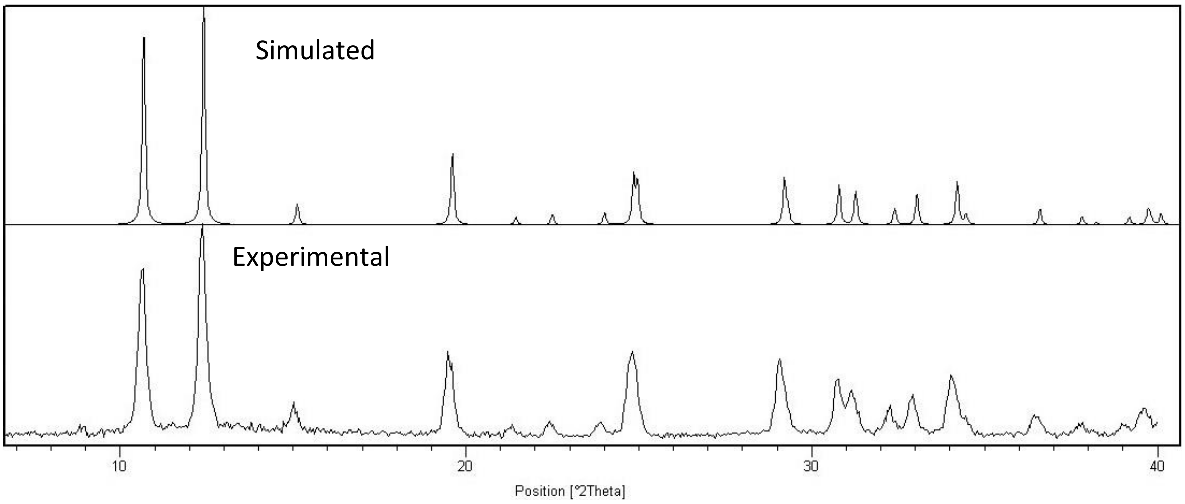
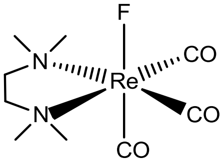


Figure S28. (Top) Simulated PXRD pattern for $[Re(CO)_3OH]_4{\cdot}4H_2O$ **9** (*Inorg. Chem. Commun. 7,* **2004** 1023); (bottom) measured PXRD pattern for the product of mechanochemical reaction of $Re(CO)_5I$ and AgF, after washing with acetone and further grinding in air

$(TMEDA)Re(CO)_3F$ 10



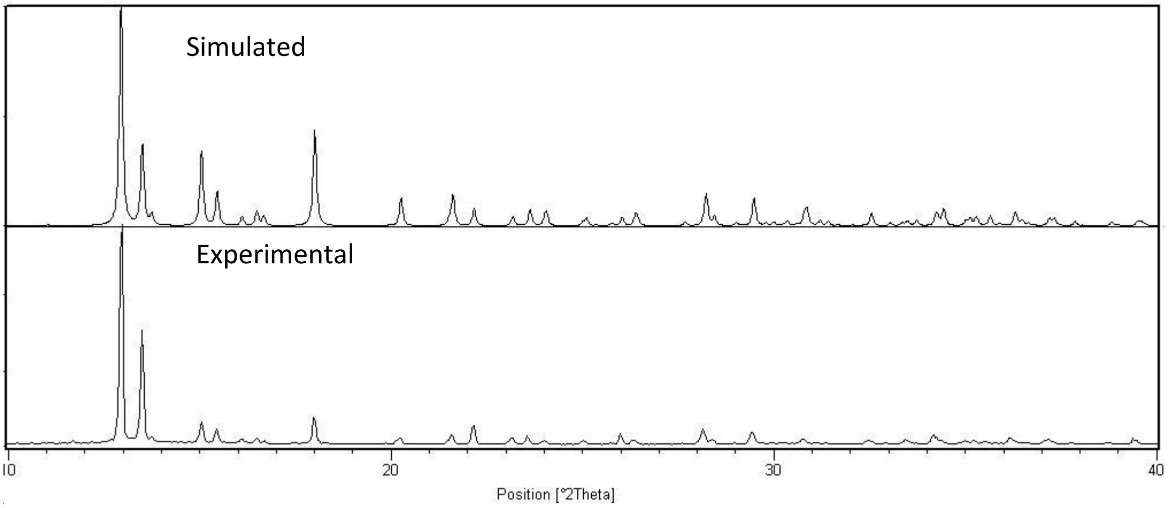
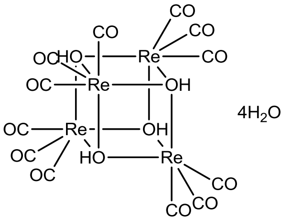
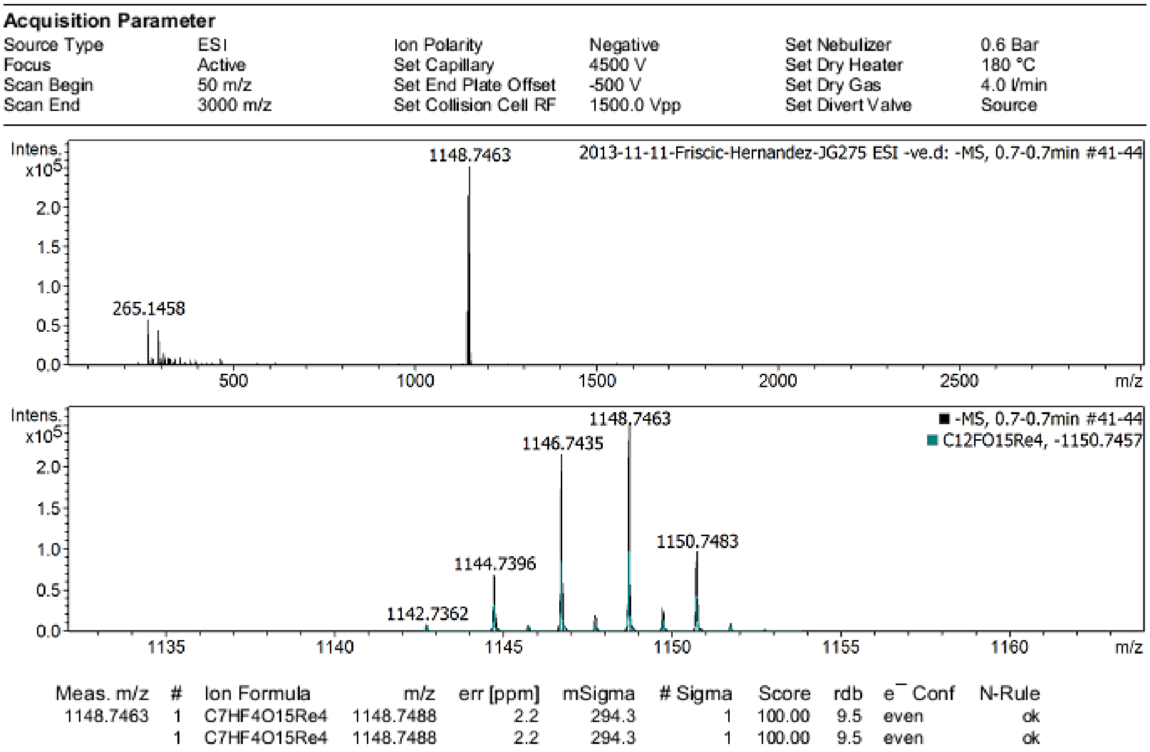


Figure S29. (Top) Simulated PXRD pattern for *fac*-(N,N-TMEDA)$Re(CO)_3F$ **10** (*Aus. J. Chem.* **1984**. *37,* 35); (bottom) measured PXRD pattern for the mechanochemically synthesized product

11. Selected HRMS spectra

$[Re(CO)_3OH]_4\cdot 4H_2O$ 9





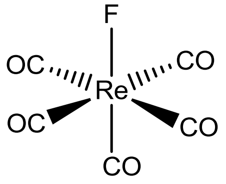
Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.6 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	1500.0 Vpp	Set Divert Valve	Source

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e⁻ Conf	N-Rule
1148.7463	1	C7HF4O15Re4	1148.7488	2.2	294.3	1	100.00	9.5	even	ok
	1	C7HF4O15Re4	1148.7488	2.2	294.3	1	100.00	9.5	even	ok

Figure S30. HRMS (negative mode): Calculated for $C_{12}H_3O_{16}Re_3$ $[M-1H]^-$: 1148.7488; found: 1148.7463**.**

Re(CO)$_5$F 3d



Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
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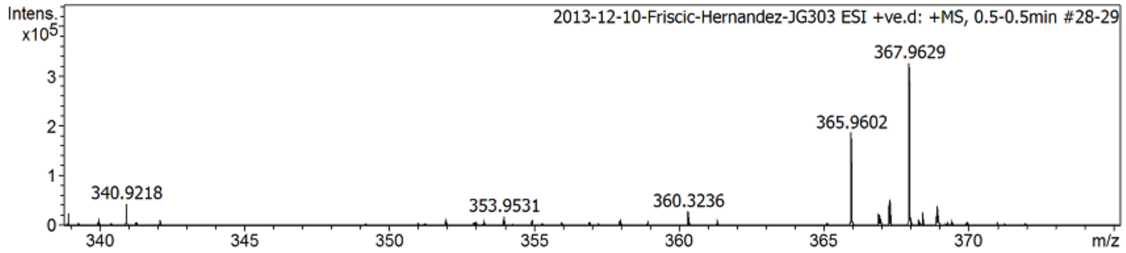
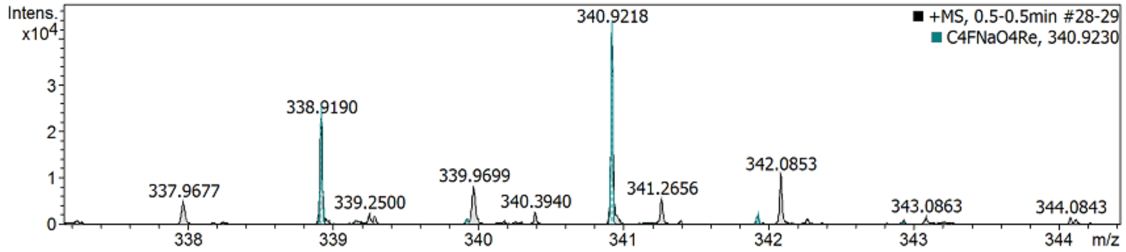
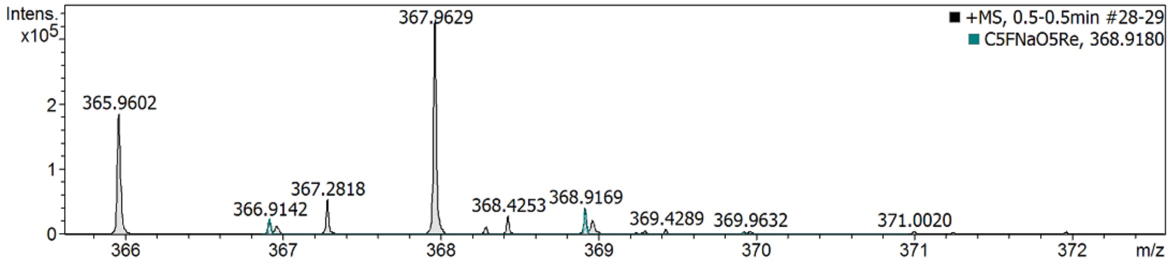
Scan End	1000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Source





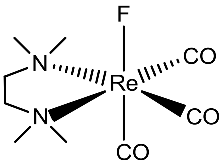
Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e⁻ Conf	N-Rule
340.9218	1	C4FNaO4Re	340.9230	3.7	32.4	1	100.00	5.0	odd	-
368.9169	1	C5FNaO5Re	368.9180	2.9	30.0	1	100.00	6.0	odd	-

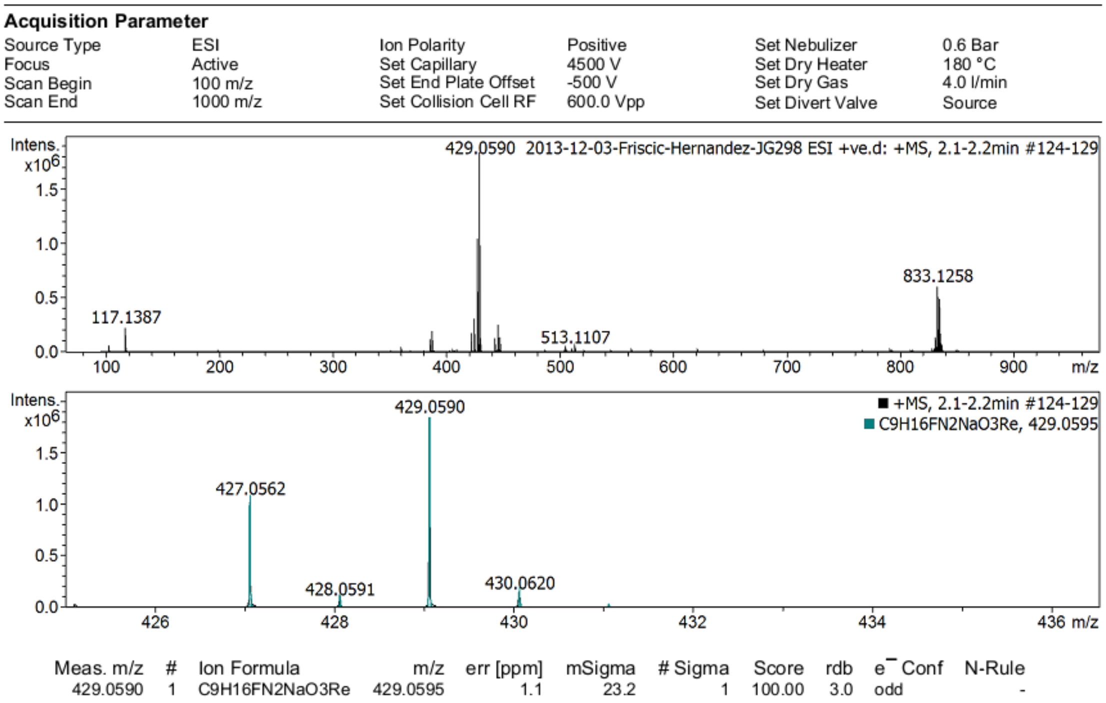


Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e⁻ Conf	N-Rule
340.9218	1	C4FNaO4Re	340.9230	3.7	32.4	1	100.00	5.0	odd	-
368.9169	1	C5FNaO5Re	368.9180	2.9	30.0	1	100.00	6.0	odd	-

Figure S31. HRMS (Positive mode): Calculated for C_4O_4ReFNa [(**3d**+Na)-CO]$^+$: 340.9230; found: 340.9218; Calculated for C_5O_5ReFNa [**3d**+Na]$^+$: 368.9180; found: 368.9169

(TMEDA)Re(CO)$_3$F 10





Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	100 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Source

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e⁻ Conf	N-Rule
429.0590	1	C9H16FN2NaO3Re	429.0595	1.1	23.2	1	100.00	3.0	odd	-

Figure S32. HRMS (Positive mode): Calculated for $C_9H_{16}O_3N_2ReFNa$ [(**10**+Na)]$^+$: 429.0595; found: 429.0590.