Electronic Supplementary Information

for

Low-overpotential CO₂ reduction by phosphine-substituted Ru(II) polypyridyl complex

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Experimental details

General procedures

All the solvents were purchased from Wako Pure Chemical Industries, while the chemicals were purchased from Sigma-Aldrich Co. All the reagents were of highest quality available and were used as received. $^1$H-NMR, and $^{31}$P-NMR spectra were collected at room temperature on a JEOL JNM-ECS400 spectrometer. UV-vis absorption spectra were measured on a Shimadzu UV-2450SIM spectrophotometer at room temperature. Elemental analyses were performed on a J-Science Lab Micro Corder JM10 elemental analyzer. ESI-TOF MS spectra were collected on a JEOL JMS-T100LC mass spectrometer.

Syntheses

8-(diphenylphosphanyl)quinoline (pqn) was synthesized according to literature procedures.$^{51}$

$^1$H NMR (CDCl$_3$): $\delta$ 7.12 (m, 1 H), 7.30 (m, 10 H), 7.43 (m, 2 H), 7.81 (d, 1 H), 8.16 (d, 1 H), 8.87 (dd, 1 H). $^{31}$P{$^1$H} NMR (CDCl$_3$): $\delta$ −14.32 (s). Anal. Found: C, 78.52; H, 5.36; N, 4.16. Calculated for C$_{21}$H$_{16}$NP∙0.5H$_2$O (pqn∙0.5 H$_2$O): C, 78.25; H, 5.32; N, 4.35.

trans($P$,MeCN)‐[Ru$^{II}$(tpy)(pqn)(MeCN)](PF$_6$)$_2$ (RuP) was synthesized according to literature procedures.$^{52}$ ESI-TOF MS (positive ion, acetonitrile): m/z 324.1 ([Ru(tpy)(pqn)]$^{2+}$), 344.6 ([Ru(tpy)(pqn)(MeCN)]$^{2+}$). $^1$H NMR (CD$_3$CN): $\delta$ 6.56 (t, 4 H), 6.96 (t, 4 H), 7.12 (d, 2 H), 7.21 (t, 2 H), 7.55 (t, 2 H), 7.81 (t, 2 H), 7.93 (m, 3 H), 8.04 (d, 2 H), 8.21 (t, 1 H), 8.29 (d, 2 H), 8.50 (d, 1 H), 8.81 (d, 1 H), 9.83 (d, 1 H). $^{31}$P{$^1$H} NMR (CD$_3$CN): $\delta$ 58.74 (s). Anal. Found: C, 46.02; H, 3.35; N, 7.18. Calculated for C$_{38}$H$_{30}$N$_5$P$_3$F$_{12}$Ru∙0.5H$_2$O (RuP∙0.5 H$_2$O): C, 46.21; H, 3.16; N, 7.09.

[Ru$^{II}$(tpy)(bpy)(MeCN)](PF$_6$)$_2$ (RuN) was synthesized according to literature procedures.$^{53}$ ESI-TOF MS (positive ion, acetonitrile): m/z 266.0 ([Ru(tpy)(bpy)(MeCN)]$^{2+}$), 676.9 ([Ru(tpy)(bpy)(MeCN)]$^{2+}$PF$_6$). $^1$H NMR (CD$_3$CN): $\delta$ 7.05 (m, 1 H), 7.26 (d, 1 H), 7.32 (m, 2 H), 7.66 (d, 2 H), 7.78 (t, 1 H), 7.98 (m, 3 H), 8.30 (m, 3 H), 8.40 (d, 2 H), 8.54 (d, 2 H), 8.60 (d, 1 H), 9.58 (d, 1 H). Anal. Found: C, 39.42; H, 2.75; N, 10.24. Calculated for C$_{37}$H$_{22}$N$_6$P$_2$F$_{12}$Ru (RuN): C, 39.48; H, 2.70; N, 10.23.
**Electrochemistry**

Electrochemical experiments were performed at room temperature on a BAS ALS Model 650DKMP electrochemical analyzer in acetonitrile or γ-butyrolactone ([cat.] = 0.5 mM; 0.1 M tetraethylammonium perchlorate (TEAP)). Cyclic voltammetry was performed by using a one-compartment cell with a three-electrode configuration, which consisted of a glassy carbon disk, platinum wire, and Ag/Ag⁺ electrode (Ag/0.01 M AgNO₃) as the working, auxiliary, and reference electrodes, respectively. The glassy carbon disc working electrode was polished using alumina prior to each measurement. The concentration of CO₂ during the measurements was controlled using KOFLOC RK1200M and 8500MC-0-1-1 flowmeters.

**UV-vis spectro-electrochemistry**

A thin-layer quartz glass cell (light path length 1 mm) was used. A piece of 80 mesh platinum gauze, a platinum wire, and a Ag/Ag⁺ electrode (Ag/0.01 M AgNO₃) were used as the working, auxiliary, and reference electrodes, respectively. All solutions were purged with Ar or saturated with CO₂ (0.28 M) before the measurements. Spectra were obtained after electrolysis at appropriate potentials for 8 mins. UV-vis spectra in the range from 250–800 nm were recorded. The temperature was controlled at 20 °C during the measurements, and a weak Ar/CO₂ flow was supplied throughout the experiments. The redox potentials of samples were calibrated against the redox signal for the ferrocene/ferrocnium (Fc/Fc⁺) couple.

**Controlled-potential electrolysis**

Controlled-potential electrolysis was performed in a gas-tight two-compartment electrochemical cell. In the first compartment, the carbon rod working electrode (1.2 cm² surface area) and a leakless Ag/AgCl reference electrode (Innovative Instruments, Inc.) were immersed in 0.1 M TEAP/MeCN (5 ml) containing the catalyst (0.5 mM) and H₂O (2.65 M). In the second compartment, the Pt auxiliary electrode was immersed in 0.1 M TEAP/MeCN (5 ml) containing ferrocene (40 mM) as a sacrificial reductant. The two compartments were separated by an anion exchange membrane (Selemion DSV). The solution was purged vigorously with CO₂ for 30 min prior to electrolysis. The electrolysis was performed for 1 h with constant stirring. The amount of CO and H₂ produced at the headspace of the cell was
quantified by a Shimadzu GC-8A with a TCD detector equipped with a packed column with Molecular Sieve 13X-S 60/80. Additionally, liquid product was quantified by using a Shimadzu LC-20AD with SPD-20A and RID-10A detectors equipped with a Shim-pack SCR102H column. Calibration curves were obtained by sampling known amounts of H₂, CO, and HCOOH.

**DFT calculations**

Geometric optimization and electronic structures were obtained at the B3LYP or UB3LYP functional and LanL2DZ basis set with the Gaussian 09 program package.
Figure S1. A CV of RuP in acetonitrile (black line, [complex] = 0.5 mM; 0.1 M TEAP; WE: GC, CE: Pt wire, RE: Ag/Ag+, scan rate, 0.10 V/s) under Ar, and the simulated CV (red circle). Elchsoft DigiElch 7.FD software was used for simulation of CV to obtain redox potentials of RuP as reported previously.52
**Table S1.** Simulation parameters for the CV. Elchsoft DigiElch 7.0. software was used for simulation.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>RuP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sweep rate ([v]) (V/s)</td>
<td>0.10</td>
</tr>
<tr>
<td>Resistance ([R]) (Ω)</td>
<td>200</td>
</tr>
<tr>
<td>Capacitance ([C_{dl}]) (F)</td>
<td>(7.0 \times 10^{-6})</td>
</tr>
<tr>
<td>Temperature ([T]) (K)</td>
<td>293</td>
</tr>
<tr>
<td>Surface area ([A]) (cm(^2))</td>
<td>0.07</td>
</tr>
<tr>
<td>Diffusion constant ([D]) (cm(^2)/s)</td>
<td>(1.0 \times 10^{-5})</td>
</tr>
<tr>
<td>Concentration ([c]) (mol/dm(^3))</td>
<td>(5.0 \times 10^{-4})</td>
</tr>
<tr>
<td>(E^{\circ'}_1) (V)</td>
<td>−1.69</td>
</tr>
<tr>
<td>(k_{s1}) (cm/s)</td>
<td>0.05</td>
</tr>
<tr>
<td>(\alpha_1)</td>
<td>0.50</td>
</tr>
<tr>
<td>(E^{\circ'}_2) (V)</td>
<td>−1.78</td>
</tr>
<tr>
<td>(k_{s2}) (cm/s)</td>
<td>0.05</td>
</tr>
<tr>
<td>(\alpha_2)</td>
<td>0.50</td>
</tr>
</tbody>
</table>

\(E^{\circ'}_1\) and \(E^{\circ'}_2\) are referred to Fc/Fc\(^+\).
Figure S2. Isodensity surface plots of selected frontier molecular orbitals of RuP and RuP$^-$ based on the optimized ground-state geometry. The geometric optimization and electronic structures for RuP and RuP$^-$ were calculated at the B3LYP/LanL2DZ level and UB3LYP/LanL2DZ level, respectively with the Gaussian 09 program package.
Figure S3. Experimental (black lines) and simulated CVs (red circles) of RuP (0.5 mM) in 0.1 M TEAP/MeCN under Ar at various scan rates, (a) = 0.25 V/s, (b) = 0.50 V/s, (c) = 0.75 V/s, (d) = 1.00 V/s. Working electrode, glassy carbon; counter electrode, Pt wire; reference electrode, Ag/Ag⁺.
Figure S4. Variation of peak current ($i_p$) of RuP (0.5mM) at the (a) first redox wave and (b) second redox wave versus square root of scan rate. The $i_p$ values were obtained from simulated CVs.
Table S2. Summary of the data used for the $i_p$ vs. $\nu^{1/2}$ plot.

<table>
<thead>
<tr>
<th>$\nu$ (V/s)</th>
<th>$\nu^{1/2}$</th>
<th>$i_p$ (µA)</th>
<th>Second redox wave</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.10</td>
<td>0.316</td>
<td>7.00</td>
<td>7.41</td>
</tr>
<tr>
<td>0.25</td>
<td>0.500</td>
<td>11.13</td>
<td>10.95</td>
</tr>
<tr>
<td>0.50</td>
<td>0.707</td>
<td>15.06</td>
<td>14.60</td>
</tr>
<tr>
<td>0.75</td>
<td>0.866</td>
<td>17.89</td>
<td>18.05</td>
</tr>
<tr>
<td>1.00</td>
<td>1.00</td>
<td>20.13</td>
<td>20.90</td>
</tr>
</tbody>
</table>
Figure S5. CVs of RuP (0.5 mM) in anhydrous 0.1 M TEAP/MeCN under various concentrations of CO₂ (CO₂/Ar, v/v%). Working electrode, glassy carbon; counter electrode, Pt wire; reference electrode, Ag/Ag⁺; scan rate, 0.1 V/s.
Figure S6. (a) CVs of RuP (0.5 mM) in 0.1 M TEAP/MeCN under various concentrations of CO₂ (CO₂/Ar, v/v%) in the presence of H₂O (2.65 M). (b) CVs of RuP (0.5 mM) in 0.1 M TEAP/MeCN at various concentrations of H₂O under CO₂ (0.28 M). Working electrode, glassy carbon; counter electrode, Pt wire; reference electrode, Ag/Ag⁺; scan rate, 0.1 V/s.
Figure S7. The result of controlled-potential electrolysis of RuP (0.5 mM) in 0.1 M TEAP/MeCN under CO$_2$ (0.28 M) at −1.7 V (vs. Fc/Fc$^+$) in the presence of H$_2$O (2.65 M) for 1 h. Working electrode, glassy carbon (1.2 cm$^2$); counter electrode, Pt wire; reference electrode, Ag/AgCl. Approximately 1.75 C has been transferred during 1 h of electrolysis.

Table S3. Summary of CPE experiments$^a$

<table>
<thead>
<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>[H$_2$O], M</th>
<th>Charge, C</th>
<th>Faradaic Efficiency, %$^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>CO</td>
</tr>
<tr>
<td>1</td>
<td>RuP</td>
<td>2.65</td>
<td>1.75</td>
<td>55.8</td>
</tr>
<tr>
<td>2</td>
<td>-</td>
<td>2.65</td>
<td>0.15</td>
<td>-</td>
</tr>
</tbody>
</table>

$^a$ Conditions: 0.50 mM catalyst, applied voltage: −1.70 V (vs. Fc/Fc$^+$), duration: 1 h.
$^b$ Further reduced species of CO$_2$ such as formaldehyde and methanol have not been detected, and the fate of the rest of the charge is not clear at this stage.
**Table S4.** Overpotentials ($\eta$) and operating conditions of selected homogeneous CO$_2$ reduction electrocatalysts

<table>
<thead>
<tr>
<th>Entry</th>
<th>Molecule</th>
<th>Solvent</th>
<th>Overpotentials, $\eta$ (V)</th>
<th>TOF (s$^{-1}$)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>[Ru(tpy)(pqn)(MeCN)]$^{2+}$ (RuP)</td>
<td>MeCN + 2.65 M H$_2$O</td>
<td>0.40$^{a,b}$</td>
<td>4.7$^{f}$</td>
<td>This work</td>
</tr>
<tr>
<td>2</td>
<td>[Ru(tpy)(bpy)(MeCN)]$^{2+}$ (RuN)</td>
<td>MeCN + 2.65 M H$_2$O</td>
<td>0.60$^{a,b}$</td>
<td>5.1$^{e}$</td>
<td>S9</td>
</tr>
<tr>
<td></td>
<td>MeCN</td>
<td></td>
<td>0.87</td>
<td>5.5$^{e}$, 38.4$^{f}$</td>
<td>S10, S11</td>
</tr>
<tr>
<td>3</td>
<td>[Ru(tpy)(Mebim-py)(MeCN)]$^{2+}$</td>
<td>MeCN</td>
<td>0.81</td>
<td>19$^{e}$, 31$^{f}$</td>
<td>S10, S11</td>
</tr>
<tr>
<td>4</td>
<td>[Ru(tpy)(bpy)(CO)]$^{2+}$</td>
<td>MeCN</td>
<td>0.50$^{a,c}$</td>
<td>-</td>
<td>S12</td>
</tr>
<tr>
<td>5</td>
<td>[Ru(tBu$_3$tpy)(6-mbpy)(MeCN)]$^{2+}$</td>
<td>MeCN</td>
<td>0.47</td>
<td>1.1$^{e}$</td>
<td>S13</td>
</tr>
<tr>
<td>6</td>
<td>trans(Cl)‐Ru(mesbpy)(CO)$_2$Cl$_2$</td>
<td>MeCN + 0.5 M phenol</td>
<td>0.75$^{a,c}$</td>
<td>1300$^{g}$</td>
<td>S14</td>
</tr>
<tr>
<td>7</td>
<td><a href="OTf">Mn(mesbpy)(CO)$_3$(MeCN)</a></td>
<td>MeCN + 3.2 M MeOH</td>
<td>0.70</td>
<td>2000$^{g}$</td>
<td>S15</td>
</tr>
<tr>
<td></td>
<td>MeCN + 120 mM Mg$^{2+}$</td>
<td></td>
<td>0.30–0.45$^{d}$</td>
<td>20$^{g}$</td>
<td>S16</td>
</tr>
<tr>
<td>8</td>
<td>FeTDHPP</td>
<td>DMF + 2 M H$_2$O</td>
<td>0.41–0.56</td>
<td>3200$^{f}$</td>
<td>S11</td>
</tr>
<tr>
<td>9</td>
<td>Fe-o-TMA</td>
<td>DMF + 3 M phenol</td>
<td>0.22</td>
<td>100000$^{f}$</td>
<td>S17</td>
</tr>
</tbody>
</table>

$^{a}$The overpotentials, $\eta$, were calculated using previously reported methods,$^{518}$ which is the difference between the standard potential of CO$_2$/CO couple in a specific solvent system with potential at half the catalytic current ($E_{cat/2}$) of catalyst (Eq. S1).

$$\eta = |E_{CO_2/CO} - E_{cat/2}|$$  (Eq. S1)

$^{b}$Calculated based on the data shown in Figure S11.

$^{c}$The values of $E_{cat/2}$ were estimated from the CVs reported in the references.

For the standard potential of CO$_2$/CO couple, $E^{\circ}_{CO_2/CO, MeCN} = -1.25$ V vs. Fc/Fc$^+$ or $E^{\circ}_{CO_2/CO, DMF} = -1.30$ V vs. Fc/Fc$^+$ was used.$^{511}$

$^{d}$$E^{\circ}_{CO_2}$ for 2CO$_2$ + Mg$^{2+}$ → CO + MgCO$_3$ was estimated between −1.15 V to −1.30 V vs. Fc/Fc$^+$. $^{516}$

$^{e}$The value is calculated based on results of CV as reported in S10.

$^{f}$The value is calculated based on results of controlled potential electrolysis as reported in S11.

$^{g}$The value is calculated based on results of CV as reported in S15.
Estimation of TOF and TON for RuP from controlled-potential electrolysis

\[
\frac{I}{FA} = \frac{\sqrt{k_{\text{cat}}D[\text{cat}]} }{1 + \exp\left(\frac{E_{\text{applied}} - E^*_\text{cat}}{RT}\right)} \tag{Eq. S2}
\]

\[
\text{TOF} = \frac{k_{\text{cat}}}{(1 + \exp\left(\frac{E_{\text{applied}} - E^*_\text{cat}}{RT}\right))} \tag{Eq. S3}
\]

\[
\text{TON} = \frac{k_{\text{cat}}t}{(1 + \exp\left(\frac{E_{\text{applied}} - E^*_\text{cat}}{RT}\right))} \tag{Eq. S4}
\]

The equations were previously adapted by Savéant et al. in electrocatalytic CO2 reduction reaction.\textsuperscript{S11} By using these formula, the amount of active catalyst is the number of moles contained within the thin reaction-diffusion layer that develops adjacent to the electrode surface.\textsuperscript{S11} In these equations, \(i\) represents stable current transferred during controlled-potential electrolysis, \(F\) is Faraday constant (96485 C/mol), \(A\) is the surface area of working electrode (1.2 cm\(^2\)), \(k_{\text{cat}}\) is the overall rate constant of the catalytic CO\(_2\) reduction reaction, \(D\) is the diffusion coefficient (~5 x 10\(^{-6}\) cm\(^2\)/s), [cat] is the concentration of catalyst used (5 x 10\(^{-7}\) mol/cm\(^3\)), \(R\) is the universal gas constant (8.31 J K\(^{-1}\) mol\(^{-1}\)), \(T\) is temperature (298 K), \(E_{\text{applied}}\) is the applied potential during electrolysis, \(E^*_\text{cat}\) is the standard potential of the catalyst, \(t\) is the electrolysis duration, TOF is the turnover frequency, and TON is the turnover number.

The average current density of 0.42 mA/cm\(^2\) (the faradaic efficiency for CO formation is 56%), corresponds to \(i/A = 0.24\) mA/cm\(^2\) was obtained for 1 h electrolysis at \(-1.70\) V vs. Fc/Fc\(^+\). Since the electrolysis is performed on the plateau of the catalytic wave, \(i/FA = (k_{\text{cat}}D)^{1/2}[\text{cat}]\) (Eq. S2) leading to the TOF = 4.7 s\(^{-1}\) and TON = 1.7 \(\times\) 10\(^4\). We also calculated the TON value based on the total cell volume and the value was estimated to be 2 for 1h. The result indicate the CO\(_2\) reduction reaction mediated by RuP is catalytic.
Figure S8. CVs of RuN (0.5 mM, open circuit = −0.27 V) in 0.1 M TEAP/MeCN under various concentrations of CO₂ (CO₂/Ar, v/v%) in the absence of H₂O. Working electrode, glassy carbon; counter electrode, Pt wire; reference electrode, Ag/Ag⁺; scan rate, 0.1 V/s.
Figure S9. Experimental UV-Vis absorption spectra of RuP (0.5 mM) at various applied potentials in 0.1 M TEAP/MeCN under Ar using BASi Spectro-electrochemical Cell (open-circuit potential = −0.27 V). Working electrode, Pt mesh; counter electrode, Pt wire; reference electrode, Ag/Ag⁺. Solutions were purged with Ar for 10 mins prior to measurements. Weak Ar flow was maintained throughout the measurement. Spectra were acquired after electrolysis at respective potentials for 8 mins.
Figure S10. Experimental UV-Vis absorption spectra of RuP (0.5 mM) at various applied potentials in 0.1 M TEAP/MeCN under CO₂. (a) Resting potential to −1.60 V and (b) −1.60 V to −1.70 V. Working electrode, Pt mesh; counter electrode, Pt wire; reference electrode, Ag/Ag⁺. Spectra were acquired after electrolysis at respective potentials for 8 mins.
Figure S11. CVs of 0.5 mM of RuN (top) and RuP (bottom) in 0.1 M TEAP/MeCN under Ar (black line), CO₂ (0.28 M, red line), and CO₂ in the presence of 2.65 M H₂O (blue line). Working electrode, glassy carbon; counter electrode, Pt wire; reference electrode, Ag/Ag⁺; scan rate, 0.1 V/s. Potential sweeps were started from the open circuit potential (−0.26 V for RuN, −0.27 V for RuP). Arrows represent the direction of potential sweeping. In the presence of H₂O, the current enhancement was observed at $E_{pc} = −1.95$ V for RuN and $E_{pc} = −1.73$ V for RuP, which attributed to the electrocatalytic CO₂ reduction.
Figure S12. HOMO of the one-electron reduced species, \( \text{RuP}^- \) (left) and HOMO–LUMO of one-electron reduced \( \text{RuP}^- \) with a \( \text{CO}_2 \) molecule bound to the Ru center, \( \text{RuP}_{\text{CO}_2}^- \) (middle & right) based on the optimized ground-state geometry. DFT calculations were performed using the UB3LYP functional and LanL2DZ basis set.
**Figure S13.** (a) CV of RuP (0.5 mM) in 0.1 M TEAP/γ-butyrolactone under Ar at different potential scan range. (b) CV of RuP (0.5 mM) in 0.1 M TEAP/γ-butyrolactone under Ar added with various amount of MeCN. Working electrode, glassy carbon; counter electrode, Pt wire; reference electrode, Ag/Ag⁺; scan rate, 0.1 V/s.
Figure S14. (a) CV of RuN (0.5 mM) in 0.1 M TEAP/γ-butyrolactone under Ar at different potential scan range. (b) CV of RuN (0.5 mM) in 0.1 M TEAP/γ-butyrolactone under Ar added with various amount of MeCN. Working electrode, glassy carbon; counter electrode, Pt wire; reference electrode, Ag/Ag+; scan rate, 0.1 V/s.
Figure S15. (a) CVs of RuN (0.5 mM) in 0.1 M TEAP/γ-butyrolactone under CO₂. (b) CVs of RuP (0.5 mM) in 0.1 M TEAP/γ-butyrolactone under CO₂. Working electrode, glassy carbon; counter electrode, Pt wire; reference electrode, Ag/Ag⁺; scan rate, 0.1 V/s.
References


