Supporting Information for

Synthesis of cyclic organic carbonates via catalytic oxidative carboxylation of olefins in flow reactors

Ajay A Sathe†, Anirudh M K Nambiar§, Robert M Rioux*§

†Department of Chemistry and §Department of Chemical Engineering
The Pennsylvania State University
*email: rioux@engr.psu.edu.

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I. General Information

All reagents were obtained from commercial vendors (TCI, Sigma-Aldrich, Alfa-Aesar) and used without purification unless noted. NMR data was collected on Bruker Avance CDPX 300 or DRX 400 MHz instruments. Spectra were referenced internally to tetramethylsilane (TMS) (CDCl₃: 0.00 ppm (1H), 0.0 (13C). Gas chromatography was performed on Agilent 7890A equipped with a HP-5 column and flame ionization detector (GC-FID). The following method was used for analysis. Inlet temperature: 230 °C, carrier gas (Argon) flow rate: 5 mL/min (column), split ratio 38:1, oven program: 50 °C for 1 minute, 10 °C/min to 100 °C, hold at 100 °C for 2.5 minutes, FID temperature: 250 °C, H₂ flow rate: 30 mL/min, air flow rate: 300 mL/min, make-up flow rate: 30 mL/min. Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) measurements were performed on Agilent 700 series instrument. The Re standard (100 ppm Re in 2% HNO₃) for the ICP analysis were purchased from High Purity Standards Ltd. Mass spectrometric measurements were performed at University of Illinois (Champaign, IL) and the Pennsylvania State University Proteomics and Mass Spectrometry Core Facility (University Park, PA). Standard tube fittings were purchased from Swagelok and IDEX health and science.

II. Effect of stirring speed on yield in batch reactor

In a 25 ml round bottom flask equipped with a 1/4 inch stir bar were added methyltrioxorhenium (7.5 mg, 0.03 mmol), dodecane (68 μL, 0.3 mmol), styrene (0.345 mL, 3 mmol), 3-methylpyrazole (58 μL, 0.72 mmol), and dichloromethane (to adjust final volume to 1.5 mL). 30% H₂O₂ (aq) solution (0.64 mL, 6 mmol) was then added to the mixture and the flask was sealed with a rubber septum. The flask was placed on a stir plate and the reaction mixture was stirred at the desired RPM for 1.5 hours at room temperature.

After 1.5 hours the stirring was stopped and approximately 0.5 mL of the organic layer was pipetted into a 2 mL vial. The crude organic layer was analyzed using GC. The styrene conversion was determined by gas chromatography. Figure S1 shows the plot for conversion as a function of stirring rate.
Figure S1. Effect of stirring speed on epoxidation of styrene using MTO and hydrogen peroxide.
III. Optimization of epoxidation flow reactor

a. Preparation of reactant solutions

In a 20 mL scintillation vial, methyltrioxorhenium, dodecane (internal standard) (10 mol%), styrene, 3-methylpyrazole and dichloromethane were added together. The quantity of reactants added depended on the variable being optimized. The mixture was stirred until the catalyst dissolved and subsequently loaded into a 5 mL gas-tight glass syringe after passing through a 0.2 μm PTFE syringe filter. A small aliquot of the solution was analyzed using GC before the reaction. Another 5 mL glass gas tight syringe was filled with 30% H₂O₂ (aq) solution.

b. Reaction procedure and product quantification

The temperature controller was turned on and the epoxidation reactor was set to the desired temperature. After the temperature stabilized the gas tight syringes containing the styrene and H₂O₂ (aq) solutions were loaded onto syringe pumps (New Era NE-510). A 40 psi back pressure regulator from IDEX was attached to the reactor outlet. The pumps were started with their respective flow rates. After constant segmented flow was seen, the system was run for 1.5 residence times. This ensured that the system reached steady state when the reaction mixture was analyzed. For analysis, the reaction mixture was collected in a vial containing MnO₂ to quench the excess peroxide and terminate the reaction. After effervescence ceased, approximately 0.5 mL of DCM was added to the reaction mixture. The organic layer was separated from the aqueous layer using a Pasteur pipette. The crude organic layer was analyzed using GC-FID. The conversion of styrene was determined by comparing the initial and final area ratios of styrene to the internal standard (dodecane).

The conversion of styrene as a function of catalyst loading, co-catalyst loading, oxidant equivalents, residence time, concentration and temperature was studied and the data collected is presented in Figures S2-S7.
Figure S2. Effect of catalyst loading on styrene epoxidation. Temperature = 40 °C, oxidant = 5 equiv, co-catalyst = 24 mol%, residence time = 30 mins, concentration = 4M, organic flow rate = 0.32 mL/h, aqueous flow rate = 0.68 mL/h.

Figure S3. Effect of co-catalyst loading on styrene epoxidation. Temperature = 40 °C, oxidant = 5 equiv, catalyst = 1 mol%, residence time = 30 mins, concentration = 4M, organic flow rate = 0.32 mL/h, aqueous flow rate = 0.68 mL/h.
Figure S4. Effect of temperature on styrene epoxidation. Oxidant = 4.7 equiv, co-catalyst = 24 mol%, catalyst = 1 mol%, residence time = 30 mins, concentration = 2M, organic flow rate = 0.5 mL/h, aqueous flow rate = 0.5 mL/h.

Figure S5. Effect of H₂O₂ equivalents on styrene epoxidation. Temperature = 40 °C, co-catalyst = 24 mol%, catalyst = 1 mol%, residence time = 30 mins, concentration = 4M, total flow rate = 1 mL/h. Note: Oxidant equivalents were varied by changing the ratio of organic to aqueous flow rate.
Figure S6. Effect of residence time on styrene epoxidation. Temperature = 40 °C, oxidant = 5 equiv., co-catalyst = 24 mol%, catalyst = 1 mol%, concentration = 4M.

Figure S7. Effect of concentration on styrene epoxidation. Temperature = 40 °C, oxidant = 5 equiv, co-catalyst = 24 mol%, catalyst = 1 mol%, residence time = 30 mins, total flow rate = 1 mL/h. Note: Oxidant equivalents were kept constant by changing the ratio of organic to aqueous flow rate.
IV. Optimization of carboxylation flow reactor

a. Preparation of reactant solutions

In a 20 mL scintillation vial, aluminum catalyst, styrene oxide, tetrabutylammonium iodide, DCM and THF were combined. The mixture was stirred until all the solids dissolved and subsequently transferred into a 5 mL gas-tight glass syringe after passing through a 0.2 μm PTFE syringe filter.

b. Initialization of flow reactor

A syringe containing dichloromethane was loaded onto the syringe pump and the CO₂ cylinder was pressurized to 140 psi, and the Argon cylinder for the back pressure regulator (BPR) was pressurized to 100 psi. The pressure was allowed to equilibrate and the CO₂ mass flow controller was adjusted to the desired setting. The micro metering valve on the BPR was opened to maintain a slow bleed. After the pressure at the CO₂ inlet to the reactor stabilized, the temperature controllers were turned on and adjusted to the desired set point.

c. Reaction procedure and product quantification

After the temperature stabilized the syringes were replaced with those containing the reagent solution. The liquid flow rates were set to 10 mL/h until the liquid pressure just exceeded the system pressure, i.e. when flow starts. The liquid and gas flow rates were adjusted to obtain the desired residence times. After steady state was reached (2 residence times), the reaction mixture was withdrawn using the 6-way valve. Approximately 200 μL of the sample was withdrawn and the solvent removed in-vacuo. The crude mixture was analyzed using ¹H NMR (in CDCl₃) and the conversion was determined by comparing the ratio of the protons of the styrene oxide to the protons of the cyclic carbonate.

The styrene oxide conversion was studied as a function of residence time (Figure S8), temperature (Figure S9) and co-catalyst loading (Figure S10).
Figure S8. Effect of residence time on conversion of styrene oxide to styrene carbonate. Temperature = 100 °C, Al catalyst loading = 2 mol%, co-catalyst (Bu₄NI) = 10 mol%, concentration = 2M, CO₂ pressure = 110 psi.

Figure S9. Effect of temperature on conversion of styrene oxide to styrene carbonate. Al catalyst loading = 2 mol%, co-catalyst (Bu₄NI) = 10 mol%, concentration = 2M, CO₂ pressure = 110 psi, residence time = 40 mins.
**Figure S10.** Effect of co-catalyst loading on conversion of styrene oxide to styrene carbonate. Al catalyst loading = 2 mol%, concentration = 2M, CO₂ pressure = 110 psi, residence time = 40 mins.
V. Procedure for sequential batch epoxidation and carboxylation of styrene

To a 20 mL three necked flask equipped with a reflux condensor, methyltrioxorhenium (30 mg), 1,4-dinitrobenzene (internal standard) (50.4 mg), olefin (12 mmol), 3-methylpyrazole (0.232 mL), and dichloromethane (to adjust final volume to 3 mL) were added together. This flask was placed in an ice bath and 6.4 mL of 30% H$_2$O$_2$ solution was added dropwise to the vial. Once the addition was complete the flask was removed from the ice bath and transferred to an oil bath set at 40 °C. The reaction was carried out for 30 minutes, after which the contents of the flask were transferred to a separating funnel. The aqueous and organic layer were separated and the organic layer was collected in a 20 mL vial.

To the vial was added the aluminum catalyst (0.124 g, 0.24 mmol), Bu$_4$NI (0.443g, 1.2mmol), DCM (2.4mL) and THF (0.6 mL). This was then transferred to an autoclave which was subsequently pressurized to 110 psi. The autoclave was placed in an oil bath maintained at 110 °C. After 45 minutes the autoclave was placed in an ice bath and once it cooled down to room temperature was vented.

An aliquot of the reaction mixture was withdrawn for NMR analysis in CDCl$_3$, which indicated a yield of 88% styrene carbonate.
VI. General procedure for the direct oxidative carboxylation of olefins

a. Preparation of reactant solutions

In a 20 mL scintillation vial, methyltriioxorhenium (30 mg), 1,4-dinitrobenzene (internal standard) (50.4 mg), olefin (12 mmol), 3-methylpyrazole (0.232 mL), and dichloromethane (to adjust final volume to 3 mL) were added together. The mixture was stirred until all the solids dissolved and subsequently transferred into a 5 mL gas tight glass syringe. Another 5 mL gas-tight glass syringe was filled with 30% H₂O₂ (aq) solution. In another 20 mL scintillation vial, the aluminum catalyst (0.124 g, 0.24 mmol), Bu₄NI (0.443g, 1.2mmol), DCM (2.4mL) and THF (0.6 mL) were combined. The mixture was stirred until all the solids were dissolved, and the solution loaded into a 5 mL gas-tight glass syringe after passing through a 0.2 μm PTFE syringe filter.

b. Initialization of flow reactor

The syringe pumps were initially loaded with syringes containing pure solvents, dichloromethane for the organic reaction streams and distilled water for the aqueous stream. All three pumps were started with an initial flow rate of 6 mL/h. The CO₂ cylinder was pressurized to 140 psi, and the Argon cylinder for the back pressure regulator (BPR) was pressurized to 100 psi. The pressure was allowed to equilibrate and the CO₂ mass flow controller was adjusted to the desired setting (1 sccm). The micro metering valve on the BPR was opened to maintain a slow bleed. After the pressure at the CO₂ inlet to the reactor stabilized, the temperature controllers were turned on and the epoxidation reactor was set to 40 °C and the carboxylation reactor was set to 100 °C.

c. Reaction procedure and product quantification

After the temperature stabilized the syringes were swapped with those containing the reagent solutions. The liquid flow rates were set to 10 mL/h until the liquid pressure reached the system pressure i.e. when flow starts. The liquid flow rates were then adjusted as follows: olefin – 0.32 mL/h; peroxide – 0.68 mL/h; and the carboxylation catalyst – 0.32 mL/h. The CO₂ flow rate was set at 1 sccm.

After steady segmented flow was seen, the system was run for 7 h. This ensures that the system is at steady-state when analyzing the reaction mixture. The reaction mixture was then withdrawn using a 6 way valve. Approximately 200 μL of the sample was withdrawn and the solvent removed in-vacuo. The crude mixture was analyzed using ¹H NMR (in CDCl₃) and the yield of the reaction is determined by comparing the ratio of the protons of the internal standard (1,4-dinitrobenzene) to the protons of the cyclic carbonate.

Once the reaction was completed, the flow reactor was depressurized by turning off the gas inlets and opening the micro metering valve to vent the BPR. The product collected in the BPR was chromatographed on silica gel using ethyl acetate/hexanes on a Combiflash Rf+.
VI. Procedure for ICP analysis of Re in reactor effluent.

50 μL of reactor effluent (aqueous or organic) was taken in a 20 mL scintillation vial. It was evaporated to dryness under vacuum. To the residue was added 2 mL of conc. HNO₃ which was subsequently boiled off to digest all the Re. To this solution was added 10 mL of 2% HNO₃ solution and the resulting solution was filtered through a 0.2 μm PTFE syringe filter. The solution was analyzed by ICP-OES to determine the concentration of the rhenium in each phase.

It was found that the Re partitioned to 56% in the aqueous and 44% in the organic phase.
VII. Reactor Setup

The oxidative carboxylation reactor was setup as shown in Figure S11. The organic solution, containing the olefin, catalyst and co-catalyst, and the aqueous peroxide solution were loaded into gastight syringes and introduced into the system using syringe pumps. The fluidic connections were made using either flat bottom or compression fittings. The streams were mixed using a T-mixer before entering the epoxidation reactor, which was made from 1/8" PTFE tubing and packed with sand. A PTFE membrane based separator was utilized to partition the aqueous and organic phases coming out of the epoxidation reactor. The aqueous phase was sent to a bulk collection vessel and the separator outlet containing the organic stream was mixed with the carboxylation catalyst solution in another T-mixer. This solution was then mixed with CO₂ gas which was introduced from a mass flow controller. The gas-liquid segmented flow then entered the carboxylation reactor, made from 1/8" stainless steel tubing and packed with sand. The back pressure of the system was set by pressurizing the bulk collection vessels with Argon. The temperature of both reactors was maintained using heat tape and a PID temperature controller. Effluent sampling from the carboxylation reactor was done using a 6-way valve.

Figure S11. Reactor setup for oxidative carboxylation of olefins.
Figure S12. Reactor set-up: (1) syringe pumps, (2) check valves, (3) epoxidation reactor, (4) BPR and aqueous waste, (5) temperature controller, (6) pressure gauge, (7) BPR and product collection, (8) sampling valve, (9) carboxylation reactor, (10) membrane separator.

Figure S13: The membrane separator splitting an inlet stream of food dye (aqueous) and dichloromethane (organic) into respective components.
VIII. Reactor parts list

Figure S14. Components used for construction of the reactor set-up in Figure S12. For details on vendors and part numbers, see Table S1.
## Table S1: Oxidative carboxylation reactor parts list

<table>
<thead>
<tr>
<th>Item</th>
<th>Vendor</th>
<th>Part #</th>
<th>Part description</th>
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</thead>
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<td>New Era Pump Systems Inc.</td>
<td>NE-510</td>
<td>Syringe pump</td>
</tr>
<tr>
<td>B</td>
<td>Hamilton</td>
<td>81520</td>
<td>5 mL glass syringe</td>
</tr>
<tr>
<td>C</td>
<td>IDEX H&amp;S</td>
<td>P-628/P-678</td>
<td>Luer adapter</td>
</tr>
<tr>
<td>D</td>
<td>IDEX H&amp;S</td>
<td>P-287</td>
<td>Super flangeless nut for 1/16” tubing</td>
</tr>
<tr>
<td>E</td>
<td>IDEX H&amp;S</td>
<td>P-250</td>
<td>Super flangeless ferrule for 1/16” tubing</td>
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<td>Zeus tubing</td>
<td>IWTT-063-C</td>
<td>1/16” PTFE tubing (1/32” ID)</td>
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<tr>
<td>G</td>
<td>IDEX H&amp;S</td>
<td>CV-3330</td>
<td>Check valve</td>
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<tr>
<td>H</td>
<td>IDEX H&amp;S</td>
<td>P-632</td>
<td>Mixing tee</td>
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<td>Swagelok</td>
<td>SS-400-6-1</td>
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<td>J</td>
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<td>18421</td>
<td>Glass wool</td>
</tr>
<tr>
<td>K</td>
<td>Saint Gobain</td>
<td>TSPF35-0250-062-25</td>
<td>1/4” PTFE tubing (1/8” ID) (epoxidation reactor)</td>
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<td>L</td>
<td>Fischer Scientific</td>
<td>S23-3</td>
<td>Sand for packed bed reactor</td>
</tr>
<tr>
<td>M</td>
<td>Fischer Scientific</td>
<td>S25-500</td>
<td>Sand for heating reactor</td>
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<tr>
<td>N</td>
<td>McMaster Carr</td>
<td>Aluminum tube for heating reactor</td>
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<td>N/A</td>
<td>Heat tape</td>
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<td>P</td>
<td>Auber Instruments</td>
<td>WS-1510DPM</td>
<td>PID temperature controller &amp; thermocouple</td>
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<tr>
<td>Q</td>
<td>Zaiput Flow Technologies</td>
<td>SEP10</td>
<td>Membrane Separator with 0.2 μm membrane from sterlitech</td>
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<tr>
<td>R</td>
<td>Swagelok</td>
<td>SS-200-6-1</td>
<td>Steel reducing union, 1/8” x 1/16”</td>
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<td>Autoclave</td>
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<td>Hoke</td>
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<td>McMaster Carr</td>
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<td>Tylan</td>
<td>FC-260V/AA9304091</td>
<td>MFC (0-10 sccm CO₂)</td>
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<tr>
<td>Z</td>
<td>Air Gas</td>
<td>Gas regulators</td>
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<tr>
<td>AA</td>
<td>Swagelok</td>
<td>89785K824</td>
<td>1/4” steel tubing (0.18” ID) (carboxylation reactor)</td>
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<tr>
<td>BB</td>
<td>IDEX H&amp;S</td>
<td>V-540</td>
<td>6-way valve</td>
</tr>
</tbody>
</table>
IX. Characterization Data

(a) 4-phenyl-1,3-dioxolan-2-one

$^1$H (CDCl$_3$, 300 MHz) $\delta$ = 7.47-7.26 (m, 5H), 5.70 (t, 1H, $J$ = 8.4 Hz), 4.35 (t, 1H, $J$ = 8 Hz); $^{13}$C (CDCl$_3$, 75 MHz) $\delta$ = 154.9, 135.8, 129.7, 129.2, 125.9, 78.0, 71.2. HRMS calculated for C$_9$H$_{12}$NO$_3$ [M+NH$_4$]$^+$: 182.0817. Found: 182.0831. R$_f$ (silica gel with 30% EtOAc in Hexanes as eluent): 0.35

(b) 4-(4-fluorophenyl)-1,3-dioxolan-2-one

$^1$H (CDCl$_3$, 300 MHz) $\delta$ = 7.40-7.35 (m, 2H), 7.15-7.09 (m, 2H), 5.72 (t, 1H, $J$ = 8 Hz), 4.84 (t, 1H, $J$ = 8.5 Hz), 4.36 (t, 1H, $J$ = 8.3 Hz); $^{13}$C (CDCl$_3$, 75 MHz) $\delta$ = 165.0, 161.7, 154.8, 131.7, 131.7, 128.2, 128.1, 116.4, 116.1, 77.6, 71.2. HRMS: calculated for C$_9$H$_7$O$_3$F: 182.0379. Found: 182.0370. R$_f$ (silica gel with 30% EtOAc in Hexanes as eluent): 0.28

(c) 4-(naphthalen-2-yl)-1,3-dioxolan-2-one

$^1$H (CDCl$_3$, 300 MHz) $\delta$ = 7.87-7.77 (m, 4H), 7.52-7.49 (m, 2H), 7.37 (dd, 1H, $J$ = 8.5, 1.6 Hz), 5.78 (t, 1H, $J$ = 8 Hz), 4.81 (t, 1H, $J$ = 9 Hz), 4.38 (t, 1H, $J$ = 9 Hz); $^{13}$C (CDCl$_3$, 75 MHz) $\delta$ = 155.0, 133.6, 132.9, 132.8, 129.4, 128.1, 127.8, 127.1, 126.9, 125.9, 122.5, 78.2, 71.0. HRMS calculated for C$_{13}$H$_{14}$NO$_3$ [M+NH$_4$]$^+$: 232.0974. Found: 232.0955. R$_f$ (silica gel with 30% EtOAc in Hexanes as eluent): 0.28

(d) 4-(4-chlorophenyl)-1,3-dioxolan-2-one

$^1$H (CDCl$_3$, 300 MHz) $\delta$ = 7.40 (d, 2H, $J$ = 9 Hz), 7.32 (d, 2H, $J$ = 9 Hz), 5.71 (t, 1H, $J$ = 9 Hz), 4.85 (t, 1H, $J$ = 9 Hz); $^{13}$C (CDCl$_3$, 75 MHz) $\delta$ = 154.8, 135.5, 134.4, 129.4, 127.5, 77.3, 71.0. HRMS: calculated for C$_9$H$_7$O$_3$Cl: 198.0084. Found: 198.0074. R$_f$ (silica gel with 30% EtOAc in Hexanes as eluent): 0.36

(e) 4-(3-chlorophenyl)-1,3-dioxolan-2-one

$^1$H (CDCl$_3$, 300 MHz) $\delta$ = 7.39-7.37 (m, 3H), 7.27-7.24 (m, 1H), 5.70 (t, 1H, $J$ = 7.9 Hz), 4.86 (t, 1H, $J$ = 8.5 Hz), 4.34 (t, 1H, $J$ = 8.2 Hz); $^{13}$C (CDCl$_3$, 75 MHz) $\delta$ = 154.6, 137.9, 135.1, 130.6, 129.8, 126.0, 123.9, 77.0, 71.0. HRMS: calculated for C$_9$H$_7$O$_3$Cl: 198.0084. Found: 198.0074. R$_f$ (silica gel with 30% EtOAc in Hexanes as eluent): 0.42
(f) 4-(2-chlorophenyl)-1,3-dioxolan-2-one

$^1$H (CDCl$_3$, 300 MHz) $\delta = 7.50$-$7.47$ (m, 1H), 7.41-$7.33$ (m, 3H), 6.00 (t, 1H, $J = 9$ Hz), 4.99 (t, 1H, $J = 8.5$ Hz), 4.27 (t, 1H, $J = 8$ Hz); $^{13}$C (CDCl$_3$, 75 MHz) $\delta = 154.7, 134.5, 131.1, 130.4, 129.9, 127.6, 126.2, 75.0, 70.4$. HRMS calculated for C$_9$H$_{11}$ClNO$_3$ [M+NH$_4^+$]: 216.0427. Found: 216.0436. $R_f$ (silica gel with 30% EtOAc in Hexanes as eluent): 0.38

(g) 4-(4-(tert-butyl)phenyl)-1,3-dioxolan-2-one

$^1$H (CDCl$_3$, 400 MHz) $\delta = 7.45$ (d, 2H, $J = 8.4$ Hz), 7.31 (d, 2H, $J = 8$ Hz), 5.66 (t, 1H, $J = 8$ Hz), 4.77 (t, 1H, $J = 8.4$ Hz), 4.33 (t, 1H, $J = 8$ Hz), 1.31 (s, 9H); $^{13}$C (CDCl$_3$, 75 MHz) $\delta = 155.0, 152.9, 132.8, 126.1, 125.9, 78.0, 71.1, 34.7, 31.2$. HRMS calculated for C$_{13}$H$_{20}$NO$_3$ [M+NH$_4^+$]: 238.1443. Found: 238.1438. $R_f$ (silica gel with 30% EtOAc in Hexanes as eluent): 0.45

(h) 4-phenethyl-1,3-dioxolan-2-one

$^1$H (CDCl$_3$, 300 MHz) $\delta = 7.29$-$7.13$ (m, 5H), 4.61-$4.57$ (m, 1H), 4.39 (t, 1H, $J = 9$ Hz), 3.97 (t, 1H, $J = 9$ Hz), 2.76-$2.63$ (m, 2H), 2.03-$1.88$ (m, 2H); $^{13}$C (CDCl$_3$, 75 MHz) $\delta = 155.1, 140.0, 128.6, 128.4, 126.4, 76.2, 69.3, 35.3, 30.7$. HRMS calculated for C$_{11}$H$_{16}$NO$_3$ [M+NH$_4^+$]: 210.1130. Found: 210.1132. $R_f$ (silica gel with 30% EtOAc in Hexanes as eluent): 0.30

(i) 4-pentyl-1,3-dioxolan-2-one

$^1$H (CDCl$_3$, 300 MHz) $\delta = 4.74$-$4.70$ (m, 1H), 4.57 (t, 1H, $J = 9$ Hz), 4.10 (t, 1H, $J = 9$ Hz), 1.82-$1.65$ (m, 2H), 1.48-$1.30$ (m, 8H), 0.89 (t, 3H); $^{13}$C (CDCl$_3$, 75 MHz) $\delta = 155.2, 69.5, 33.9, 31.5, 28.8, 24.4, 22.5, 14.0$. HRMS calculated for C$_{8}$H$_{18}$NO$_3$ [M+NH$_4^+$]: 176.1287. Found: 176.1269. $R_f$ (silica gel with 30% EtOAc in Hexanes as eluent): 0.31

(j) 4-(2-oxo-1,3-dioxolan-4-yl)butanenitrile

$^1$H (CDCl$_3$, 300 MHz) $\delta = 4.80$ (t, 1H, $J = 6$ Hz), 4.61 (t, 1H, $J = 8.2$ Hz), 4.14 (t, 1H, $J = 8$ Hz), 2.49-$2.45$ (m, 2H), 1.91-$1.75$ (m, 4H); $^{13}$C (CDCl$_3$, 75 MHz) $\delta = 155.0, 119.6, 76.4, 69.3, 32.5, 20.9, 16.6$. HRMS calculated for C$_{7}$H$_{13}$N$_2$O$_3$ [M+NH$_4^+$]: 173.0926. Found: 173.0919. $R_f$ (silica gel with 50% EtOAc in Hexanes as eluent): 0.23
Current Data Parameters
NAME acetone_carb
EXPND 2
PROWD 1

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Time_ 14:53
INSTRM spect
PROBHD 5 mm GNP 5H/1
PULPROG zgq20
TD 90036
SOLVENT CDCl3
NS 150
DO 4
SN 18756.992 Hz
FIGRES 0.266810 Hz
AD 1.743076 sec
RG 4000
SW 26.600 usec
DE 6.00 usec
TE 300 D H
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00000000 sec

********** CHANNEL 11 **********
NUC1 13C
P1 5.25 usec
PL1 -6.00 dB
SP01 75.4106337 MHz

********** CHANNEL 12 **********
CPDPO2 wait10
NUC2 1H
POD2 115.00 usec
PL2 0.00 dB
PL12 19.70 dB
PL13 19.70 dB
SP02 299.871595 MHz

F2 - Processing parameters
SI 30768
SF 75.4002600 MHz
MDW 1M
SSB 0
LB 1.00 Hz
GS 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
FMP 230.000 ppm
F1 17342.54 Hz
FDP -10.000 ppm
F2 -2.0202 Hz
PPMCM 12.00000 ppm/cm
H2CM 904.82837 Hz/cm