## Supporting Information

# Visible Light Photoredox Cross-Coupling of Acyl Chlorides with Potassium Alkoxymethyltrifluoroborates: Synthesis of $\alpha$ Alkoxyketones 

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## EXPERIMENTAL

General Considerations: All reactions were carried out under an inert atmosphere of nitrogen or argon unless otherwise noted. THF was dried over activated alumina. $\operatorname{IrCl}_{3} \cdot \mathrm{xH}_{2} \mathrm{O}$, and $\mathrm{NiCl}_{2} \cdot d$ dme were purchased from commercial sources. $\mathrm{Cs}_{2} \mathrm{CO}_{3}$ was used as received. All other reagents were purchased commercially and used as received. Photoredox reactions were irradiated with two or three standard 26 W compact fluorescent light bulbs. Stereoconvergent cross-couplings were irradiated with blue LED light strips ( $\sim 425 \mathrm{~nm}$ ). Melting points $\left({ }^{\circ} \mathrm{C}\right.$ ) are uncorrected. NMR spectra were recorded on a 400 or 500 MHz spectrometer. Data are presented as follows: chemical shift $(\mathrm{ppm})$, multiplicity $(\mathrm{s}=$ singlet, $\mathrm{d}=$ doublet, $\mathrm{t}=$ triplet, $\mathrm{m}=$ multiplet, $\mathrm{br}=$ broad), coupling constant $J(\mathrm{~Hz})$ and integration. Analytical thin-layer chromatography (TLC) was performed on TLC silica gel plates $(0.25 \mathrm{~mm})$ precoated with a fluorescent indicator. Visualization of the TLC plates was effected with ultraviolet light. Standard flash chromatography procedures were followed using 100-200 mesh silica gel. HRMS data were obtained by either ESI or CI using a TOF mass spectrometer.

## Synthesis of $\boldsymbol{\alpha}$-alkoxymethyltrifluoroborates:

Most potassium $\alpha$-alkoxymethyltrifluoroborates were purchased commercially. The unavailable potassium $\alpha$-alkoxymethyltrifluoroborates were synthesized from the corresponding alcohols according to the literature procedure. ${ }^{1}$

## Synthesis of $\operatorname{Ir}\left[\mathrm{dF}\left(\mathrm{CF}_{3}\right) \mathrm{ppy}\right]_{2}(\mathrm{bpy}) \mathrm{PF}_{6}$ as the photocatalyst I:

Photocatalyst I was synthesized according to the literature procedure. ${ }^{2}$

High-Throughput Experiments in the design and optimization of the photoredox crosscoupling reaction of hydrocinnamoyl chloride with potassium benzyloxymethyltrifluoroborate as model coupling partners:

High Throughput Experimentation (HTE) was performed at the Penn/Merck Center for High Throughput Experimentation at the University of Pennsylvania. The screens were performed on a $10 \mu \mathrm{~mol}$ scale. To reaction vials equipped with a Teflon coated magnetic stir bar in a glovebox was added a solution of Ni source and ligand [1:1] dissolved in THF. The solvent was removed in vacuo under an inert atmosphere. Then a solution of a desired additive, potassium benzyloxymethyltrifluoroborate, hydrocinnamoyl chloride, and photocatalyst I in a desired solvent, was added to each vial. The vials were sealed and stirred over blue LED lights. After 24 h the reactions were opened to air, $1 \mu \mathrm{~mol}$ of 4,4'-di-tert-butylbiphenyl ( $500 \mu \mathrm{~L}$ of a $0.002 \mu \mathrm{M}$ solution in MeCN ) was added to each vial as an internal standard, and the reaction mixtures were diluted with MeCN. The reaction mixtures were then analyzed by UPLC. The product-to-internal standard ( $\mathrm{P} / \mathrm{IS}$ ) ratios from the UPLC are shown in Figures S1-S3.


## First Screen Variables:




Figure S1. Product to internal standard ratio of the cross coupling reaction of hydrocinnamoyl chloride with potassium benzyloxymethyltrifluoroborate using the first screen variables.

According to this screen the best result was obtained for the reaction in the presence of $\mathrm{NiCl}_{2} \cdot \mathrm{dme} / \mathbf{L} 1$ and lutidine as a base.

Second Screen Variables:


| solvents | ligands | bases | Ni sourcrs |
| :---: | :---: | :---: | :---: |
| THF | L3 | lutidine | $\mathrm{NiCl}_{2} \cdot$ dme |
|  | L4 | 2-Me-pyridine | $\mathrm{Ni}\left(\mathrm{PPh}_{3}\right)_{2} \mathrm{Cl}_{2}$ |
|  | L10 |  | $\mathrm{Ni}\left(\mathrm{NO}_{3}\right)_{2}$ |
|  | L1 |  | [(TMEDA) Ni (o-tolyl) Cl ] |
|  | L11 |  |  |
|  | L5 |  |  |



Figure S2. Product to internal standard ratio of the cross coupling reaction of hydrocinnamoyl chloride with potassium benzyloxymethyltrifluoroborate using the second screen variables.

According to the first and second screens, the highest product to internal standard ratio was obtained for the reaction in the presence of $\mathrm{NiCl}_{2} \cdot \mathrm{dme} / \mathbf{L} 1$ and lutidine as a base. To improve the yield of the reaction further, screens using a variety of inorganic bases as well as solvents were carried out (Figure S3).

Third Screen Variables:

| Solvents | ligands | bases | Ni sources |
| :---: | :---: | :---: | :---: |
| THF | L1 | lutidine | $\mathrm{NiCl}_{2} \cdot \mathrm{dme}$ |
| 2-MeTHF |  | $\mathrm{Cs}_{2} \mathrm{CO}_{3}$ |  |
| $\mathrm{CH}_{3} \mathrm{CN}$ |  | $\mathrm{CsHCO}_{3}$ |  |
| EtOAc |  | $\mathrm{K}_{3} \mathrm{PO}_{4}$ |  |
| dioxane |  | $\mathrm{K}_{2} \mathrm{HPO}_{4}$ |  |
| DME |  | $\mathrm{K}_{2} \mathrm{CO}_{3}$ |  |
| acetone |  | $\mathrm{KHCO}_{3}$ |  |
| methyl tert-butyl ether (MTBE) |  | $\mathrm{NH}_{4} \mathrm{CO}_{3}$ |  |
|  |  | $\mathrm{Na}_{2} \mathrm{CO}_{3}$ |  |
|  |  | $\mathrm{NaH}_{2} \mathrm{PO}_{4}$ |  |
|  |  | KF |  |
|  |  | CsF |  |


$\square$ THF $\square$ MeTHF $\square \mathrm{CH} 3 \mathrm{CN} \square$ EtOAc $\square$ dioxane $\square$ DME $\square$ acetone $\square$ MTBE

Figure S3. Product to internal standard ratio of the cross coupling reaction of hydrocinnamoyl chloride with potassium benzyloxymethyltrifluoroborate using the third screen variables.

Accordingly, changing the base of the reaction from lutidine to $\mathrm{Cs}_{2} \mathrm{CO}_{3}$ has a great impact on the product to internal standard ratio.

## Control experiments for the cross-coupling of acyl chlorides with potassium alkoxymethyltrifluoroborates

Table S1. Control Experiments.

| $\approx$ |  |  |  |
| :---: | :---: | :---: | :---: |
| entry | conditions | additive | $1 d^{\mathrm{e}}$ |
| $1^{\text {a }}$ | $6 \mathrm{~mol} \% \mathrm{NiCl}_{2} \cdot \mathrm{dme} / \mathbf{L} \mathbf{1}$ | lutidine | $\begin{aligned} & 74 \\ & \% \end{aligned}$ |
| $2^{\text {a }}$ | $6 \mathrm{~mol} \% \mathrm{NiCl}_{2} \cdot \mathrm{dme} / \mathbf{L} \mathbf{1}$ | $\mathrm{Cs}_{2} \mathrm{CO}_{3}$ | \% ${ }^{83}$ |
| $3^{\text {a }}$ | no Ir photocatalyst | $\mathrm{Cs}_{2} \mathrm{CO}_{3}$ | 0\% |
| $4^{\text {a }}$ | no $\mathrm{NiCl}_{2} \cdot \mathrm{dme}$, no $\mathbf{L} 1$ | $\mathrm{Cs}_{2} \mathrm{CO}_{3}$ | ${ }_{\%}^{<10}$ |
| $5^{\text {a }}$ | no additive | - | ${ }_{\%}^{58}$ |
| $5^{\text {b }}$ | $4 \mathrm{~mol} \% \mathrm{NiCl}_{2} \cdot \mathrm{dme} / \mathbf{L} \mathbf{1}$ | $\mathrm{Cs}_{2} \mathrm{CO}_{3}$ | \% ${ }^{81}$ |
| $6^{\text {c }}$ | $4 \mathrm{~mol} \% \mathrm{NiCl}_{2} \cdot \mathrm{dme} / \mathbf{L} \mathbf{1}$ | $\mathrm{Cs}_{2} \mathrm{CO}_{3}$ | ${ }_{\%}^{86}$ |
| $7{ }^{\text {d }}$ | $4 \mathrm{~mol} \% \mathrm{NiCl}_{2} \cdot \mathrm{dme} / \mathbf{L} \mathbf{1}$ | $\mathrm{Cs}_{2} \mathrm{CO}_{3}$ | ${ }_{0}^{68}$ |

${ }^{a}$ Reactions were carried out using $3 \mathrm{~mol} \% \mathbf{I}$ at 0.1 M . ${ }^{b}$ Using $2 \mathrm{~mol} \% \mathbf{I}$ at 0.1
M. ${ }^{c}$ Using $2 \mathrm{~mol} \% \mathbf{I}$ at $0.05 \mathrm{M} .{ }^{d}$ Using $2 \mathrm{~mol} \% \mathbf{I}$ at $0.2 \mathrm{M} .{ }^{e}$ Isolated yields.

Screen of various chiral ligands under the optimal conditions for the phtoredox stereoconvergent synthesis of enantioenriched $\alpha$-alkoxyketones:

Using hydrocinnamoyl chloride and racemic potassium 3-benzyloxy-3trifluoroboratopropylbenzene as a model reaction, a screen of various chiral ligands $\mathbf{L} 1, \mathbf{L} 2$, L11-39 under the optimal conditions was conducted. The screen was performed on a $10 \mu \mathrm{~mol}$ scale. To reaction vials equipped with a Teflon coated magnetic stir bar in a glovebox was added a solution of $\mathrm{NiCl}_{2} \cdot \mathrm{dme}$ and a desired ligand [1:1] dissolved in THF. The solvent was removed
in vacuo under an inert atmosphere. Then $\mathrm{Cs}_{2} \mathrm{CO}_{3}$ (1.0 equiv) and a solution of potassium 3-benzyloxy-3-trifluoroboratopropylbenzene (1.2 equiv), hydrocinnamoyl chloride, and photocatalyst I ( $2 \mathrm{~mol} \%)$ in THF, was added to each vial. The vials were sealed and stirred over blue LED lights. After 24 h the reactions were opened to air, and diluted with MeCN . The reaction mixtures were then analyzed by SFC. Accordingly, L2 was the best chiral ligand and afforded the desired ketone with enantiomeric ratio of 81:19 in an excellent yield. Figure S 4 shows the SFC chromatograms of the reaction in the presence of ligands $\mathbf{L} 1$ and $\mathbf{L} 2$ [Figure S4, (a) and (b), respectively].




L2


135





Figure S4. SFC chromatograms of the reaction in the presence of ligands L1 (a) and L2 (b).

General procedure for the photoredox cross-coupling reaction of acyl chlorides with potassium alkoxymethyltrifluoroborates:


To a two dram ( 8 mL ) borosilicate glass vial equipped with a Teflon-coated magnetic stir bar was added 4-tert-Butyl-2-(2-pyridyl)oxazoline $\mathbf{L} 1(4.0 \mathrm{mg}, 0.02 \mathrm{mmol})$ and $\mathrm{NiCl}_{2} \cdot \mathrm{dme}(4.4 \mathrm{mg}$, $0.02 \mathrm{mmol})$. The vial was sealed and evacuated under vacuum and purged with Ar three times.

Anhyd and degassed THF ( $\sim 1 \mathrm{~mL}$ ) was added by syringe under Ar, and the resulting mixture was stirred until it appeared as a pale green suspension. Then, the solvent was removed under vacuum. Once dry, alkoxymethyltrifluoroborate ( $0.6 \mathrm{mmol}, 1.2$ equiv), $\operatorname{Ir}\left[\mathrm{dFCF}_{3} \mathrm{ppy}\right]_{2}(\mathrm{bpy}) \mathrm{PF}_{6} \mathbf{I}$ ( $10.1 \mathrm{mg}, 0.01 \mathrm{mmol}$ ) and $\mathrm{Cs}_{2} \mathrm{CO}_{3}(162.9 \mathrm{mg}, 0.5 \mathrm{mmol})$ were added. Next, the vial was sealed and subsequently purged and evacuated with Ar four times. Anhyd and degassed THF ( 4 mL ) was then added by syringe under Ar followed by the corresponding acyl halide ( 0.5 mmol ). The resulting mixture was stirred for 24 h in the presence of two 26 W fluorescent light bulbs while a fan was blown across the reaction setup to maintain an ambient temperature of $24{ }^{\circ} \mathrm{C}$. After completion, the crude reaction mixture was filtered through a plug of Celite and rinsed with EtOAc ( 20 mL ). The resulting solution was concentrated, and the residue was purified by column chromatography on silica gel, with EtOAc/hexanes mixtures as the eluent, to obtain products in pure form.

Gram scale reaction: To $\mathrm{a} \sim 125 \mathrm{~mL}$ long, thin-walled vacuum flask equipped with a Tefloncoated magnetic stir bar was added 4-tert-Butyl-2-(2-pyridyl)oxazoline $\mathbf{L} 1$ ( $24.5 \mathrm{mg}, 0.12$ $\mathrm{mmol}), \mathrm{NiCl}_{2} \cdot \mathrm{dme}(26.4 \mathrm{mg}, 0.12 \mathrm{mmol})$. The vial was sealed and evacuated under vacuum and purged with Ar three times. Anhyd and degassed THF ( $\sim 5 \mathrm{~mL}$ ) was added by syringe under Ar, and the resulting mixture was stirred until it appeared as a pale green suspension. Then, the solvent was removed under vacuum. Next, potassium benzyloxymethyltrifluoroborate (1.62 g, $7.11 \mathrm{mmol}, 1.2$ equiv), $\operatorname{Ir}\left[\mathrm{dFCF}_{3} \mathrm{ppy}\right]_{2}($ bpy $) \mathrm{PF}_{6} \mathbf{I}\left(59.8 \mathrm{mg}, 0.059 \mathrm{mmol}, 0.01\right.$ equiv), and $\mathrm{Cs}_{2} \mathrm{CO}_{3}$ ( $1.93 \mathrm{~g}, 5.93 \mathrm{mmol}, 1.0$ equiv) were added. The vial was sealed and subsequently purged and evacuated with Ar four times. Anhyd and degassed THF ( 59.3 mL ) was then added by syringe under Ar followed by hydrocinnamoyl chloride ( $1.000 \mathrm{~g}, 5.93 \mathrm{mmol}$ ). The resulting mixture was
stirred vigorously for 48 h in the presence of three 26 W fluorescent light bulbs while a fan was blown across the reaction setup to maintain an ambient temperature of $24^{\circ} \mathrm{C}$. After completion, the crude reaction mixture was filtered through a plug of Celite and rinsed with EtOAc ( 50 mL ). The resulting solution was concentrated, and the residue was purified by column chromatography on silica gel, with EtOAc/hexanes mixtures as the eluent, to obtain products in pure form.


1-(Benzyloxy)-4-phenylbutan-2-one (1a). The title compound was obtained as a liquid in $81 \%$ yield ( 0.5 mmol scale, 101.7 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): $\delta 7.38-7.32(\mathrm{~m}, 5 \mathrm{H}), 7.29(\mathrm{t}, J=$ $7.5 \mathrm{~Hz}, 2 \mathrm{H}), 7.22-7.19(\mathrm{~m}, 3 \mathrm{H}), 4.56(\mathrm{~s}, 2 \mathrm{H}), 4.04(\mathrm{~s}, 2 \mathrm{H}), 2.93(\mathrm{t}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}), 2.81(\mathrm{t}, J=$ $7.5 \mathrm{~Hz}, 2 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 208.2,141.1,137.4,128.8,128.6,128.3,128.2$, 126.4, 75.4, 73.6, 40.8, 29.5; FT-IR (neat): 3331, 1725, 1139, 1095, 733, $696 \mathrm{~cm}^{-1}$; HRMS (ES+) $\mathrm{m} / \mathrm{z}$ calcd. for $\mathrm{C}_{17} \mathrm{H}_{18} \mathrm{O}_{2} \mathrm{Na}[\mathrm{M}+\mathrm{Na}]^{+}$277.1204, found 277.1209.


1-(2,6-Dichlorobenzyloxy)-4-phenylbutan-2-one (1b). The title compound was obtained as a solid in $56 \%$ yield ( 0.5 mmol scale, 90.4 mg ). mp $62-64{ }^{\circ} \mathrm{C}$; ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.31 $(\mathrm{d}, J=8 \mathrm{~Hz}, 2 \mathrm{H}), 7.25-7.23(\mathrm{~m}, 3 \mathrm{H}), 7.18-7.15(\mathrm{~m}, 3 \mathrm{H}), 4.83(\mathrm{~s}, 2 \mathrm{H}), 4.07(\mathrm{~s}, 2 \mathrm{H}), 2.91-2.87(\mathrm{~m}$, 2H), 2.83-2.79 (m, 2H); ${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta$ 208.4, 141.2, 137.3, 132.9, 130.6, 128.8, 128.7, 126.4, 75.9, 67.9, 40.8, 29.5; FT-IR (neat): 2923, 2853, 1734, 1565, 1436, 1407,

1104, 1090, 1080, 941, 792, 760, $729 \mathrm{~cm}^{-1}$; HRMS (ES+) m/z calcd. for $\mathrm{C}_{17} \mathrm{H}_{16} \mathrm{O}_{2} \mathrm{NaCl}_{2}$ $[\mathrm{M}+\mathrm{Na}]^{+} 345.0425$, found 345.0419 .


1-(Hex-5-enyloxy)-4-phenylbutan-2-one (1c). The title compound was obtained as a liquid in $82 \%$ yield ( 0.5 mmol scale, 101.0 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.29-7.26 (m, 2H), 7.21$7.18(\mathrm{~m}, 3 \mathrm{H}), 5.82-5.77(\mathrm{~m}, 1 \mathrm{H}), 5.02-4.94(\mathrm{~m}, 2 \mathrm{H}), 3.98(\mathrm{~s}, 2 \mathrm{H}), 3.44(\mathrm{t}, J=6.5 \mathrm{~Hz}, 2 \mathrm{H}), 2.92$ $(\mathrm{t}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}), 2.79(\mathrm{t}, J=8.0 \mathrm{~Hz}, 2 \mathrm{H}), 2.07(\mathrm{q}, J=7.0,2 \mathrm{H}), 1.64-1.58(\mathrm{~m}, 2 \mathrm{H}), 1.48-1.42$ $(\mathrm{m}, 2 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\left.\mathrm{CDCl}_{3}\right): \delta 208.9,141.2,138.9,128.8,128.7,126.5,114.9,76.5$, 72.0, 40.8, 33.8, 29.6, 29.3, 25.6; FT-IR (neat): 2934, 1719, 1147, 1113, 1088, 909, 748, 732, $698 \mathrm{~cm}^{-1} ;$ HRMS $(\mathrm{ES}+) \mathrm{m} / \mathrm{z}$ calcd. for $\mathrm{C}_{16} \mathrm{H}_{22} \mathrm{O}_{2}[\mathrm{M}]^{+}$246.1620, found 246.1622.


4-Phenyl-1-(prop-2-ynyloxy)butan-2-one (1d). The title compound was obtained as a liquid in $60 \%$ yield ( 0.5 mmol scale, 60.6 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): $\delta 7.30-7.26(\mathrm{~m}, 2 \mathrm{H}), 7.21-$ $7.18(\mathrm{~m}, 3 \mathrm{H}), 4.24-4.23(\mathrm{~m}, 2 \mathrm{H}), 4.13(\mathrm{~s}, 2 \mathrm{H}), 2.94-2.91(\mathrm{~m}, 2 \mathrm{H}), 2.82-2.79(\mathrm{~m}, 2 \mathrm{H}) 2.45(\mathrm{t}, J=$ 2.4 Hz, 1H); ${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 207.5,141.0,128.9,128.7,126.5,78.8,75.9$, 74.6, 58.7, 40.9, 29.5; FT-IR (neat): 2936, 1724, 1482, 1452, 1427, 1397, 1332, 1197, 1164, 1105, 1007, $700 \mathrm{~cm}^{-1}$; HRMS (ES + ) m/z calcd. for $\mathrm{C}_{13} \mathrm{H}_{15} \mathrm{O}_{2}[\mathrm{M}+\mathrm{H}]^{+}$203.1072, found 203.1065.


1-(2-Methoxyethoxy)-4-phenylbutan-2-one (1e). The title compound was obtained as a liquid in $86 \%$ yield ( 0.5 mmol scale, 95.5 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.28-7.26 (m, 2H), 7.20$7.17(\mathrm{~m}, 3 \mathrm{H}), 4.07(\mathrm{~s}, 2 \mathrm{H}), 3.63-3.62(\mathrm{~m}, 2 \mathrm{H}), 3.56-3.55(\mathrm{~m}, 2 \mathrm{H}), 3.36(\mathrm{~s}, 3 \mathrm{H}), 2.91(\mathrm{t}, J=7.5$ $\mathrm{Hz}, 2 \mathrm{H}), 2.78(\mathrm{t}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 208.4,141.2,128.8,128.7$, 126.5, 76.9, 72.2, 71.2, 59.3, 40.7, 29.6; FT-IR (neat): 2924, 1720, 1496, 1453, 1199, 1107, 1088, 749, $699 \mathrm{~cm}^{-1}$; HRMS (ES + ) m/z calcd. for $\mathrm{C}_{13} \mathrm{H}_{18} \mathrm{O}_{3} \mathrm{Na}[\mathrm{M}+\mathrm{Na}]^{+}$245.1154, found 245.1122.


4-Phenyl-1-(2-(trimethylsilyl)ethoxy)butan-2-one (1f). The title compound was obtained as a liquid in $82 \%$ yield ( 0.5 mmol scale, 108.4 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): $\delta 7.29-7.26(\mathrm{~m}$, $2 \mathrm{H}), 7.20-7.18(\mathrm{~m}, 3 \mathrm{H}), 3.97(\mathrm{~m}, 2 \mathrm{H}), 3.53(\mathrm{t}, J=8.3 \mathrm{~Hz}, 2 \mathrm{H}), 2.92(\mathrm{t}, J=7.5,2 \mathrm{H}), 2.79(\mathrm{t}, J=$ 7.5, 2H), 0.99-0.95 (m, 2H), $0.02(\mathrm{~s}, 9 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 208.8,141.2,128.7$, 128.6, 126.4, 75.9, 69.3, 40.7, 29.6, 18.4, -1.12; FT-IR (neat): 2952, 1720, 1454, 1248, 1098, 858, 835, 848, $697 \mathrm{~cm}^{-1} ;$ HRMS (ES+) m/z calcd. for $\mathrm{C}_{15} \mathrm{H}_{24} \mathrm{O}_{2} \mathrm{SiNa}[\mathrm{M}+\mathrm{Na}]^{+}$287.1443, found 287.1445.

tert-Butyl-4-((2-oxo-4-phenylbutoxy)methyl)piperidine-1-carboxylate (1g). The title compound was obtained as a liquid in $80 \%$ yield ( 0.5 mmol scale, 144.5 mg ). ${ }^{1} \mathrm{H}$ NMR ( 500 $\left.\mathrm{MHz}, \mathrm{CDCl}_{3}\right): \delta 7.25(\mathrm{t}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}), 7.17(\mathrm{t}, J=7.5 \mathrm{~Hz}, 3 \mathrm{H}), 4.09(\mathrm{~m}, 2 \mathrm{H}), 3.95(\mathrm{~s}, 2 \mathrm{H})$, $3.25(\mathrm{~d}, J=6.5 \mathrm{~Hz}, 2 \mathrm{H}), 2.89(\mathrm{t}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}), 2.75(\mathrm{t}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}), 2.66(\mathrm{~m}, 2 \mathrm{H}), 1.78-$ $1.66(\mathrm{~m}, 3 \mathrm{H}), 1.43(\mathrm{~s}, 9 \mathrm{H}), 1.16-1.08(\mathrm{~m}, 2 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\left.\mathrm{CDCl}_{3}\right): \delta 208.5,155.0$, $141.0,128.7,128.6,126.4,79.5,76.7,76.6,44.0,40.7,36.6,29.5,29.1,28.7$; FT-IR (neat): 2924, 1722, 1686, 1452, 1420, 1365, 1274, 1247, 1233, 1170, 1143, $750 \mathrm{~cm}^{-1} ;$ HRMS (ES+) m/z calcd. for $\mathrm{C}_{21} \mathrm{H}_{32} \mathrm{NO}_{4}[\mathrm{M}+\mathrm{H}]^{+} 362.2331$, found 362.2325.


1-((2-Chloropyridin-3-yl)methoxy)-4-phenylbutan-2-one (1h). The title compound was obtained as a liquid in $66 \%$ yield ( 0.5 mmol scale, 66.6 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): $\delta$ 8.32-8.30 (m, 1H), 7.85-7.83(m, 1H), 7.29-7.24 (m, 3H), 7.20-7.17 (m, 3H), 4.60 (s, 2H), 4.15 (s, 2H), $2.94(\mathrm{t}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}), 2.81(\mathrm{t}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR ( $125.8 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) : $\delta$ $207.3,149.6,148.8,140.8,137.5,132.3,128.8,128.6,126.6,122.9,76.2,69.7,40.8,29.6$; FTIR (neat): $3375,3324,1687,1561,1515,1434,1238,1158,763 \mathrm{~cm}^{-1} ;$ HRMS (ES+) m/z calcd. for $\mathrm{C}_{16} \mathrm{H}_{16} \mathrm{NO}_{2} \mathrm{NaCl}[\mathrm{M}+\mathrm{Na}]^{+} 312.0755$, found 312.0767.


1-((2,6-Dichlorophenyl)(phenyl)methoxy)-4-phenylbutan-2-one (1i) The title compound was obtained as a liquid in $71 \%$ yield ( 0.5 mmol scale, 141.7 mg ). ${ }^{1} \mathrm{H} \mathrm{NMR}\left(500 \mathrm{MHz}, \mathrm{CDCl}_{3}\right.$ ): $\delta$ 7.37-7.26 (m, 9H), 7.23-7.18 (m, 4H), $6.41(\mathrm{~s}, 1 \mathrm{H}), 4.05(\mathrm{dd}, J=46.8,16.4 \mathrm{~Hz}, 2 \mathrm{H}), 3.01-2.99$ $(\mathrm{m}, 2 \mathrm{H}), 2.97-2.94(\mathrm{~m}, 2 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\left.\mathrm{CDCl}_{3}\right): \delta 208.5,141.2,139.4,136.6,135.1$, $130.3,129.8,128.8,128.7,128.4,127.5,126.4,126.1,79.3,74.6,41.1,29.4 ;$ FT-IR (neat): 2933, 2855, 1736, 1565, 1436, 1407, 1104, 1090, 1080, 941, 792, 760, $731 \mathrm{~cm}^{-1} ;$ HRMS (ES+) m/z calcd. for $\mathrm{C}_{23} \mathrm{H}_{20} \mathrm{O}_{2} \mathrm{NaCl}_{2}[\mathrm{M}+\mathrm{Na}]^{+} 421.0738$, found 421.0734 .


1-(tert-Butoxy)-4-phenylbutan-2-one (1j). The title compound was obtained as a liquid in $75 \%$ yield ( 0.5 mmol scale, 82.6 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.30-7.27 (m, 2H), 7.21-7.18 (m, $3 \mathrm{H}), 3.92(\mathrm{~s}, 2 \mathrm{H}), 2.92-2.89(\mathrm{~m}, 2 \mathrm{H}), 2.86-2.82(\mathrm{~m}, 2 \mathrm{H}), 1.19(\mathrm{~s}, 9 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 209.9,141.4,128.8,128.7,126.4,74.3,68.8,41.0,29.7,27.6 ;$ FT-IR (neat): 2974, 2359, 2341, 1720, 1564, 1490, 1392, 1365, 1197, 1082, 877, $727 \mathrm{~cm}^{-1} ;$ HRMS (ES+) m/z calcd. for $\mathrm{C}_{13} \mathrm{H}_{17} \mathrm{O}_{2}\left[\mathrm{M}-\mathrm{CH}_{3}\right]^{+}$205.1229, found 205.1222.


4-(Benzyloxy)-1,6-diphenylhexan-3-one (1k). The title compound was obtained as a solid in $88 \%$ yield ( 0.5 mmol scale, 155.9 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.43-7.32(m,9H), 7.25(t, $J=7.5 \mathrm{~Hz}, 4 \mathrm{H}), 7.16(\mathrm{~d}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}), 4.55(\mathrm{~d}, \mathrm{~J}=11.5 \mathrm{~Hz}, 1 \mathrm{H}), 4.40(\mathrm{~d}, J=11.5 \mathrm{~Hz}, 1 \mathrm{H})$, $3.84(\mathrm{dd}, \mathrm{J}=7.9,4.5 \mathrm{~Hz}, 1 \mathrm{H}), 2.93(\mathrm{dd}, J=14.7,5.8 \mathrm{~Hz}, 4 \mathrm{H}), 2.81-2.75(\mathrm{~m}, 1 \mathrm{H}), 2.71-2.65(\mathrm{~m}$, $1 \mathrm{H}), 2.04-2.00(\mathrm{~m}, 1 \mathrm{H}), 1.98-1.93(\mathrm{~m}, 1 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR ( $125.8 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): $\delta 212.3,141.4$, 141.3, 137.7, 128.8, 128.7, 128.7, 128.7, 128.3, 126.4, 126.3, 84.3, 72.7, 39.7, 33.9, 31.6, 29.6.; FT-IR (neat): 3027, 1713, 1496, 1453, 1092, $1028 \mathrm{~cm}^{-1} ; \operatorname{HRMS}(E S+) \mathrm{m} / \mathrm{z}$ calcd. for $\mathrm{C}_{25} \mathrm{H}_{26} \mathrm{O}_{2} \mathrm{Na}[\mathrm{M}+\mathrm{Na}]^{+}$381.1831, found 381.1819.


2-(Benzyloxy)-1-phenylethanone (2a). The title compound was obtained as a solid in $87 \%$ yield ( 0.5 mmol scale, 99.5 mg ). mp $58-60{ }^{\circ} \mathrm{C} ;{ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): $\delta 7.92(\mathrm{~d}, J=8.0 \mathrm{~Hz}$, $2 \mathrm{H}), 7.57(\mathrm{t}, J=7.5 \mathrm{~Hz}, 1 \mathrm{H}), 7.45(\mathrm{t}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}), 7.40-7.34(\mathrm{~m}, 4 \mathrm{H}), 7.31(\mathrm{t}, J=7.5 \mathrm{~Hz}$, 1H), $4.75(\mathrm{~s}, 2 \mathrm{H}), 4.69(\mathrm{~s}, 2 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR ( $125.8 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): $\delta$ 196.6, 137.6, 135.3, 133.8, $129.0,128.8,128.4,128.3,128.2,73.7,72.9$; FT-IR (neat): 2985, 1692, 1226, 1126, 1078, 975, 905, $742 \mathrm{~cm}^{-1} ;$ HRMS (ES+) m/z calcd. for $\mathrm{C}_{15} \mathrm{H}_{14} \mathrm{O}_{2} \mathrm{Na}[\mathrm{M}+\mathrm{Na}]^{+}$249.0891, found 249.0890.


2-(Cyclopentyloxy)-1-phenylethanone (2b). The title compound was obtained as a liquid in $90 \%$ yield ( 0.5 mmol scale, 93.9 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): $7.93(\mathrm{~d}, J=8.5 \mathrm{~Hz}, 2 \mathrm{H}$ ), 7.57-7.54 (m, 1H), $7.44(\mathrm{t}, J=7.5 \mathrm{~Hz}, 2 \mathrm{H}), 4.68(\mathrm{~s}, 2 \mathrm{H}), 4.04-4.02(\mathrm{~m}, 1 \mathrm{H}), 1.74-1.71(\mathrm{~m}, 6 \mathrm{H})$, 1.53-1.52 (m, 2H); ${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 197.1,135.4,133.6,128.9,128.3,82.8$, 72.4, 32.4, 23.7; FT-IR (neat): 2936, 2851, 1720, 1699, 1499, 1270, 1227, 1206, 1146, 1117, 969, 755, 714, $689 \mathrm{~cm}^{-1}$; HRMS (ES + ) m/z calcd. for $\mathrm{C}_{13} \mathrm{H}_{17} \mathrm{O}_{2}[\mathrm{M}+\mathrm{H}]^{+}$205.1229, found 205.1236.


2-(2-Isopropyl-5-methylcyclohexyloxy)-1-phenylethanone (2c) The title compound was obtained as a liquid in $91 \%$ yield ( 0.5 mmol scale, 127.5 mg ). ${ }^{1} \mathrm{H} \mathrm{NMR}\left(500 \mathrm{MHz}, \mathrm{CDCl}_{3}\right)$ : 7.96-7.94 (m, 2H), 7.58-7.54 (m, 1H), 7.47-7.43(m, 2H), 4.82(d, $J=16.0 \mathrm{~Hz}, 1 \mathrm{H}), 4.67(\mathrm{~d}, J=$ $16.0 \mathrm{~Hz}, 1 \mathrm{H}), 3.24-3.19(\mathrm{~m}, 1 \mathrm{H}), 2.30-2.24(\mathrm{~m}, 1 \mathrm{H}), 2.14-2.11(\mathrm{~m}, 1 \mathrm{H}), 1.66-1.61(\mathrm{~m}, 2 \mathrm{H}), 1.37-$ $1.31(\mathrm{~m}, 2 \mathrm{H}), 0.98-0.87(\mathrm{~m}, 9 \mathrm{H}), 0.73(\mathrm{~d}, J=6.5 \mathrm{~Hz}, 3 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR ( $125.8 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): $\delta$ $197.1,135.5,133.6,128.9,128.5,80.5,71.8,48.5,40.3,34.7,31.9,25.7,23.5,22.6,21.3,16.4 ;$ FT-IR (neat): 2954,2923, 2870, 1721, 1271, 1176, 1151, 1128, 1097, 1146, 1117, 969, 755, 714, $689 \mathrm{~cm}^{-1} ;$ HRMS (ES + ) $\mathrm{m} / \mathrm{z}$ calcd. for $\mathrm{C}_{18} \mathrm{H}_{27} \mathrm{O}_{2}[\mathrm{M}+\mathrm{H}]^{+}$275.2011, found 275.2010.


1-Phenyl-2-((tetrahydro-2H-pyran-3-yl)methoxy)ethanol (2d). The title compound was obtained as a liquid in $78 \%$ yield ( 0.5 mmol scale, 91.3 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.91$7.89(\mathrm{~m}, 2 \mathrm{H}), 7.57-7.53(\mathrm{~m}, 1 \mathrm{H}), 7.45-7.42(\mathrm{~m}, 2 \mathrm{H}), 4.88-4.84(\mathrm{~m}, 1 \mathrm{H}), 4.78-4.64(\mathrm{~m}, 1 \mathrm{H}), 3.98-$ $3.95(\mathrm{~m}, 1 \mathrm{H}), 3.60-3.51(\mathrm{~m}, 3 \mathrm{H}), 3.44-3.39(\mathrm{~m}, 1 \mathrm{H}), 1.84-1.81(\mathrm{~m}, 1 \mathrm{H}), 1.55-1.46(\mathrm{~m}, 4 \mathrm{H}), 1.35-$ $1.27(\mathrm{~m}, 1 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 196.8,135.2,133.7,128.9,128.2,77.32,75.5$, 74.6, 68.6, 28.2, 26.1, 23.3; FT-IR (neat): 2918, 2847, 1728, 1564, 1442, 1407, 1288, 1237, 1148, 1111, 1091, $728 \mathrm{~cm}^{-1}$; HRMS (ES+ $) \mathrm{m} / \mathrm{z}$ calcd. for $\mathrm{C}_{14} \mathrm{H}_{18} \mathrm{O}_{3} \mathrm{Na}[\mathrm{M}+\mathrm{Na}]^{+}$257.1154, found 257.1146.


2-((2-Chloropyridin-3-yl)methoxy)-1-phenylethanone (2e). The title compound was obtained as a liquid in $74 \%$ yield ( 0.5 mmol scale, 100.7 mg ). $\mathrm{mp} 41-43{ }^{\circ} \mathrm{C} ;{ }^{1} \mathrm{H} \mathrm{NMR}\left(500 \mathrm{MHz}, \mathrm{CDCl}_{3}\right)$ : $\delta 8.32-8.31(\mathrm{~m}, 1 \mathrm{H}), 7.97-7.96(\mathrm{~m}, 1 \mathrm{H}), 7.93(\mathrm{~d}, J=8.3 \mathrm{~Hz}, 2 \mathrm{H}), 7.61-7.58(\mathrm{~m}, 1 \mathrm{H}), 7.48(\mathrm{t}, J=$ $7.7 \mathrm{~Hz}, 2 \mathrm{H}), 7.29-7.26(\mathrm{~m}, 1 \mathrm{H}), 4.92(\mathrm{~d}, J=0.4 \mathrm{~Hz}, 2 \mathrm{H}), 4.76(\mathrm{~s}, 2 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\left.\mathrm{CDCl}_{3}\right): \delta 195.9,149.6,148.8,137.8,134.9,134.1,132.6,129.1,128.1,123.0,73.8,69.8$; FT-IR (neat): $2923,1702,1583,1566,1450,1414,1229,1144,975,797,750,684 \mathrm{~cm}^{-1} ;$ HRMS (ES+) $\mathrm{m} / \mathrm{z}$ calcd. for $\mathrm{C}_{14} \mathrm{H}_{13} \mathrm{NO}_{2} \mathrm{Cl}[\mathrm{M}+\mathrm{H}]^{+}$262.0635, found 262.0639.


2-(2-Methoxyethoxy)-1-phenylethanone (2f). The title compound was obtained as a liquid in $83 \%$ yield ( 0.5 mmol scale, 80.6 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.91 (dd, $J=7.5,1.0 \mathrm{~Hz}$, $2 \mathrm{H}), 7.58-7.55(\mathrm{~m}, 1 \mathrm{H}), 7.45(\mathrm{t}, J=7.5,2 \mathrm{H}), 4.83(\mathrm{~s}, 2 \mathrm{H}), 3.77-3.75(\mathrm{~m}, 2 \mathrm{H}), 3.62-3.60(\mathrm{~m}, 2 \mathrm{H})$, 3.37 (s, 3H); ${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta$ 196.6, 135.1, 133.8, 129.0, 128.1, 74.4, 72.4, 71.1, 59.3; FT-IR (neat): 2923, 2876, 1720, 1598, 1449, 1270, 1227, 1199, 1174, 1146, 1117, 969, 755, $689 \mathrm{~cm}^{-1}$; HRMS (ES+) m/z calcd. for $\mathrm{C}_{11} \mathrm{H}_{14} \mathrm{O}_{3} \mathrm{Na}[\mathrm{M}+\mathrm{Na}]^{+}$217.0841, found 217.0842.


2-(Benzyloxy)-1-cyclopropylethanone (3a). The title compound was obtained as a liquid in $93 \%$ yield ( 0.5 mmol scale, 88.4 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.38-7.34 (m, 4H), 7.32-7.29 $(\mathrm{m}, 1 \mathrm{H}), 4.61(\mathrm{~s}, 2 \mathrm{H}), 4.21(\mathrm{~s}, 2 \mathrm{H}), 2.17-2.12(\mathrm{~m}, 1 \mathrm{H}), 1.11-1.08(\mathrm{~m}, 2 \mathrm{H}), 0.94-0.90(\mathrm{~m}, 2 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta$ 208.7, 137.5, 128.7, 128.2, 128.1, 75.6, 73.6, 17.2, 11.6; FT-IR (neat): 2987, 1697, 1454, 1385, 1155, 1103, 1061, 1020, 897, $745 \mathrm{~cm}^{-1} ;$ HRMS (ES+) m/z calcd. for $\mathrm{C}_{12} \mathrm{H}_{15} \mathrm{O}_{2}[\mathrm{M}+\mathrm{H}]^{+}$191.1072, found 191.1063.


2-(Benzyloxy)-1-cyclobutylethanone (3b). The title compound was obtained as a liquid in $80 \%$ yield ( 0.5 mmol scale, 81.7 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.35-7.34 (m, 4H), 7.31-7.28 (m,
$1 \mathrm{H}), 4.56(\mathrm{~s}, 2 \mathrm{H}), 4.06(\mathrm{~s}, 2 \mathrm{H}), 3.43-3.36(\mathrm{~m}, 1 \mathrm{H}), 2.29-2.21(\mathrm{~m}, 2 \mathrm{H}), 2.15-2.09(\mathrm{~m}, 2 \mathrm{H}), 2.02-$ $1.94(\mathrm{~m}, 1 \mathrm{H}), 1.87-1.82(\mathrm{~m}, 1 \mathrm{H}) ;{ }^{13} \mathrm{C} \operatorname{NMR}\left(125.8 \mathrm{MHz}, \mathrm{CDCl}_{3}\right): \delta 209.4,137.6,128.7,128.1$, 128.0, 73.6, 73.5, 42.2, 24.4, 18.3; FT-IR (neat): 2985, 2945, 1721, 1454, 1382, 1160, 1094, 1027, $993 \mathrm{~cm}^{-1}$; HRMS (ES+) m/z calcd. for $\mathrm{C}_{13} \mathrm{H}_{15} \mathrm{O}_{2}[\mathrm{M}-\mathrm{H}]^{+}$203.1072, found 203.1070.


1-(Adamantan-1-yl)-2-(benzyloxy)ethanone (3c). The title compound was obtained as a liquid in $85 \%$ yield ( 0.5 mmol scale, 120.8 mg ). $\mathrm{mp} 56-58{ }^{\circ} \mathrm{C} ;{ }^{1} \mathrm{H} \mathrm{NMR}\left(500 \mathrm{MHz}, \mathrm{CDCl}_{3}\right.$ ): 7.37-7.33 $(\mathrm{m}, 4 \mathrm{H}), 7.31-7.28(\mathrm{~m}, 1 \mathrm{H}), 4.58(\mathrm{~s}, 2 \mathrm{H}), 4.31(\mathrm{~s}, 2 \mathrm{H}), 2.04-2.02(\mathrm{~m}, 3 \mathrm{H}), 1.82-1.81(\mathrm{~m}, 6 \mathrm{H})$, 1.75-1.66 (m, 6H); ${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 211.5,137.8,128.8,128.3,128.2,73.4$, 70.7, 45.6, 38.3, 36.8, 28.1; FT-IR (neat): 2904, 2850, 1703, 1452, 1266, 1118, 1024, 1002, 757, $744,697 \mathrm{~cm}^{-1}$; HRMS (ES + ) m/z calcd. for $\mathrm{C}_{19} \mathrm{H}_{25} \mathrm{O}_{2}[\mathrm{M}+\mathrm{H}]^{+}$285.11855, found 285.1850.


1-(Benzyloxy)-3-phenylpropan-2-one (3d). The title compound was obtained as a liquid in 61\% yield ( 0.5 mmol scale, 73.2 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): $\delta 7.37-7.31(\mathrm{~m}, 7 \mathrm{H}), 7.28-7.20$ $(\mathrm{m}, 3 \mathrm{H}), 4.56(\mathrm{~s}, 2 \mathrm{H}), 4.12(\mathrm{~s}, 2 \mathrm{H}), 3.78(\mathrm{~s}, 2 \mathrm{H}),{ }^{13} \mathrm{C} \mathrm{NMR}\left(125.8 \mathrm{MHz}, \mathrm{CDCl}_{3}\right): \delta 206.3,137.4$, 133.7, 129.8, 129.0, 128.8, 128.4, 128.3, 127.4, 74.7, 73.7, 46.6; FT-IR (neat): 2985, 1720, 1226, 1078, 975, 905, $742 \mathrm{~cm}^{-1}$; HRMS (ES $) \mathrm{m} / \mathrm{z}$ calcd. for $\mathrm{C}_{16} \mathrm{H}_{17} \mathrm{O}_{2}[\mathrm{M}+\mathrm{H}]^{+} 241.1229$, found 241.1234.


1-(Benzo[d][1,3]dioxol-5-yl)-2-(benzyloxy)ethanone (3e). The title compound was obtained as a liquid in $84 \%$ yield ( 0.5 mmol scale, 113.5 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.50 (dd, $J=$ $8.5,1.5 \mathrm{~Hz}, 1 \mathrm{H}), 7.41(\mathrm{~d}, J=1.5 \mathrm{~Hz}, 1 \mathrm{H}), 7.39-7.34(\mathrm{~m}, 4 \mathrm{H}), 7.32-7.28(\mathrm{~m}, 1 \mathrm{H}), 6.82(\mathrm{~d}, J=8.0$ $\mathrm{Hz}, 1 \mathrm{H}), 6.02(\mathrm{~s}, 2 \mathrm{H}), 4.67-4.66(\mathrm{~m}, 4 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 194.5,152.3,148.5$, $137.6,129.9,128.8,128.3,128.2,124.5,108.3,108.0,102.1 .73 .6,72.7$; FT-IR (neat): 3925, 1680, 1603, 1486, 1444, 1426, 1248, 1128, 1100, 1082, 1029, 990, $785 \mathrm{~cm}^{-1} ;$ HRMS (ES + ) m/z calcd. for $\mathrm{C}_{16} \mathrm{H}_{14} \mathrm{O}_{4} \mathrm{Na}[\mathrm{M}+\mathrm{Na}]^{+}$293.0790, found 293.0791.


2-(Benzyloxy)-1-(4-methoxyphenyl)ethanone (3f). The title compound was obtained as a liquid in $88 \%$ yield $(0.5 \mathrm{mmol}$ scale, 112.7 mg$) . \mathrm{mp} 36-38{ }^{\circ} \mathrm{C} ;{ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): $\delta 7.92(\mathrm{dd}$, $J=7.0,1.7 \mathrm{~Hz}, 2 \mathrm{H}), 7.40-7.34(\mathrm{~m}, 4 \mathrm{H}), 7.32-7.29(\mathrm{~m}, 1 \mathrm{H}), 6.93-6.91(\mathrm{~m}, 2 \mathrm{H}), 4.70(\mathrm{~s}, 2 \mathrm{H}), 4.68$ (s, 2H), $3.86(\mathrm{~s}, 3 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 195.1,164.1,147.9,137.7,130.6,128.8$, 128.4, 128.3, 114.2, 73.7, 72.8, 55.8; FT-IR (neat): 2850, 1685, 1597, 1573, 1509, 1455, 1260, 1235, 1168, 1113, 1021, 832, $749 \mathrm{~cm}^{-1}$; HRMS (ES+) m/z calcd. for $\mathrm{C}_{16} \mathrm{H}_{17} \mathrm{O}_{3}[\mathrm{M}+\mathrm{H}]^{+}$ 257.1178, found 257.1176.


4-(Benzyloxy)-2-methyl-3-oxobutan-2-yl acetate (3g). The title compound was obtained as a liquid in $50 \%$ yield ( 0.5 mmol scale, 62.5 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.37-7.33 (m, 4H), 7.31-7.29 (m, 1H), $4.59(\mathrm{~s}, 2 \mathrm{H}), 4.31(\mathrm{~s}, 2 \mathrm{H}), 2.02(\mathrm{~s}, 3 \mathrm{H}), 1.50(\mathrm{~s}, 6 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta$ 205.6, 170.7, 137.7, 128.7, 128.3, 128.2, 82.9, 73.5, 70.9, 24.0, 21.4; FT-IR (neat): 2989, 1730, 1368, 1253, 1146, 1047, 1019, 740, $697 \mathrm{~cm}^{-1}$; HRMS (ES+) m/z calcd. for $\mathrm{C}_{14} \mathrm{H}_{18} \mathrm{O}_{4} \mathrm{Na}[\mathrm{M}+\mathrm{Na}]^{+}$273.1103, found 273.1106.


2-(Benzyloxy)-1-(furan-2-yl)ethanone (3h). The title compound was obtained as a liquid in $36 \%$ yield ( 0.5 mmol scale, 38.9 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.58-7.57 (m, 1H), 7.40-7.35 $(\mathrm{m}, 4 \mathrm{H}), 7.32-7.31(\mathrm{~m}, 2 \mathrm{H}), 6.54-6.53(\mathrm{~m}, 1 \mathrm{H}), 4.68(\mathrm{~s}, 2 \mathrm{H}), 4.59(\mathrm{~s}, 2 \mathrm{H}),{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 185.9,151.3,146.9,137.5,128.8,128.4,128.3,118.5,112.6,73.8,72.5$; FT-IR (neat): 2923, 2856, 1689, 1467, 1132, 1018, 764, 738, $698 \mathrm{~cm}^{-1} ; \mathrm{HRMS}(\mathrm{ES}+) \mathrm{m} / \mathrm{z}$ calcd. for $\mathrm{C}_{13} \mathrm{H}_{13} \mathrm{O}_{3}$ $[\mathrm{M}+\mathrm{H}]^{+}$217.0865, found 217.0861.


2-(Benzyloxy)-1-(3-methylthiophen-2-yl)ethanone (3i). The title compound was obtained as a liquid in $46 \%$ yield ( 0.5 mmol scale, 56.6 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.43-7.40 (m, 3H), 7.38-7.35 (m, 2H), 7.32-7.29 (m, 1H), $6.95(\mathrm{~d}, J=4.9 \mathrm{~Hz}, 1 \mathrm{H}), 4.69(\mathrm{~s}, 2 \mathrm{H}), 4.52(\mathrm{~s}, 2 \mathrm{H}), 2.58$ (s, 3H); ${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 190.6,146.9,137.6,137.5,132.5,130.9,128.8,128.4$,
128.3, 74.4, 73.8, 17.3; FT-IR (neat): 2924, 2850, 1656, 1401, 1371, 1210, 1122, 1028, 972, 733, $696 \mathrm{~cm}^{-1}$; HRMS (ES + ) m/z calcd. for $\mathrm{C}_{14} \mathrm{H}_{14} \mathrm{O}_{2} \mathrm{NaS}[\mathrm{M}+\mathrm{Na}]^{+}$269.0612, found 269.0609.


2-(Benzyloxy)-1-morpholinoethanone (3j). The title compound was obtained as a liquid in 77\% yield ( 0.5 mmol scale, 42.3 mg ). ${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ): 7.37-7.33 (m, 4H), 7.32-7.29 (m, $1 \mathrm{H}), 4.59(\mathrm{~s}, 2 \mathrm{H}), 4.17(\mathrm{~s}, 2 \mathrm{H}), 3.68-3.66(\mathrm{~m}, 2 \mathrm{H}), 3.64-3.59(\mathrm{~m}, 4 \mathrm{H}), 3.50-3.48(\mathrm{~m}, 2 \mathrm{H}) ;{ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ): $\delta 168.1,137.4,128.8,128.4,128.3,73.6,69.7,67.2,67.0,45.9$, 42.4; FT-IR (neat): $2900,2857,1689,1644,1453,1438,1272,1111,1026,844,737 \mathrm{~cm}^{-1}$; HRMS (ES+ $+\mathrm{m} / \mathrm{z}$ calcd. for $\mathrm{C}_{13} \mathrm{H}_{17} \mathrm{NO}_{3} \mathrm{Na}[\mathrm{M}+\mathrm{Na}]^{+}$258.1106, found 258.1115.

## References:

1) Molander, G. A.; Canturk, B. J. Org. Lett. 2008, 10, 2135.
2) Tellis, J. C.; Primer, D. N.; Molander, G. A. Science 2014, 345, 433

${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(Benzyloxy)-4-phenylbutan-2-one (1a)

${ }^{13}$ C NMR (125.8 MHz, CDCl 3 ) Spectrum of 1-(Benzyloxy)-4-phenylbutan-2-one (1a)

${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(2,6-Dichlorobenzyloxy)-4-phenylbutan-2-one (1b)



${ }^{13}$ C NMR ( $\mathbf{1 2 5 . 8} \mathbf{~ M H z}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(2,6-Dichlorobenzyloxy)-4-phenylbutan-2-one (1b)

${ }^{1} \mathrm{H}$ NMR (500 MHz, $\mathrm{CDCl}_{3}$ ) Spectrum of 1-(Hex-5-enyloxy)-4-phenylbutan-2-one (1c)



${ }^{13} \mathrm{C}$ NMR ( $125.8 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(Hex-5-enyloxy)-4-phenylbutan-2-one (1c)

${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 4-Phenyl-1-(prop-2-ynyloxy)butan-2-one (1d)

${ }^{13}$ C NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ) Spectrum of 4-Phenyl-1-(prop-2-ynyloxy)butan-2-one (1d)



${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(2-Methoxyethoxy)-4-phenylbutan-2-one (1e)

${ }^{13} \mathrm{C}$ NMR ( $\mathbf{1 2 5 . 8} \mathbf{~ M H z}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(2-Methoxyethoxy)-4-phenylbutan-2-one (1e)

${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 4-Phenyl-1-(2-(trimethylsilyl)ethoxy)butan-2-one (1f)

${ }^{13}$ C NMR (125.8 MHz, CDCl $_{3}$ ) Spectrum of 4-Phenyl-1-(2-(trimethylsilyl)ethoxy)butan-2-one (1f)



${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of tert-Butyl 4-((2-oxo-4-phenylbutoxy)methyl)piperidine-1-carboxylate (1g)

${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ) Spectrum of tert-Butyl 4-((2-oxo-4-phenylbutoxy)methyl)piperidine-1-carboxylate (1g)



${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-((2-Chloropyridin-3-yl)methoxy)-4-phenylbutan-2-one (1h)




${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ) Spectrum of 1-((2-Chloropyridin-3-yl)methoxy)-4-phenylbutan-2-one (1h)

${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-((2,6-Dichlorophenyl)(phenyl)methoxy)-4-phenylbutan-2-one (1i)

${ }^{13} \mathrm{C}$ NMR ( $\mathbf{1 2 5 . 8} \mathbf{~ M H z , ~} \mathrm{CDCl}_{3}$ ) Spectrum of 1-((2,6-Dichlorophenyl)(phenyl)methoxy)-4-phenylbutan-2-one (1i)

${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(tert-Butoxy)-4-phenylbutan-2-one (1j)

${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ) Spectrum of 1-(tert-Butoxy)-4-phenylbutan-2-one (1j)





${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 4-(Benzyloxy)-1,6-diphenylhexan-3-one (1k)



${ }^{13} \mathrm{C}$ NMR ( $\mathbf{1 2 5 . 8} \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 4-(Benzyloxy)-1,6-diphenylhexan-3-one (1k)

${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-phenylethanone (2a)

${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-phenylethanone (2a)


${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(Cyclopentyloxy)-1-phenylethanone (2b)




${ }^{13}$ C NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ) Spectrum of 2-(Cyclopentyloxy)-1-phenylethanone (2b)



${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(2-Isopropyl-5-methylcyclohexyloxy)-1-phenylethanone (2c)

${ }^{13}$ C NMR ( $\mathbf{1 2 5 . 8} \mathbf{~ M H z}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(2-Isopropyl-5-methylcyclohexyloxy)-1-phenylethanone (2c)



${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-Phenyl-2-((tetrahydro-2H-pyran-3-yl)methoxy)ethanol (2d)

${ }^{13}$ C NMR ( $\mathbf{1 2 5 . 8} \mathbf{~ M H z}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-Phenyl-2-((tetrahydro-2H-pyran-3-yl)methoxy)ethanol (2d)

${ }^{1} \mathrm{H}$ NMR ( $\mathbf{5 0 0} \mathbf{M H z}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-((2-Chloropyridin-3-yl)methoxy)-1-phenylethanone (2e)

${ }^{13}$ C NMR ( $125.8 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-((2-Chloropyridin-3-yl)methoxy)-1-phenylethanone (2e)



${ }^{1} H$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(2-Methoxyethoxy)-1-phenylethanone (2f)




${ }^{13}$ C NMR ( $\mathbf{1 2 5 . 8} \mathbf{~ M H z}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(2-Methoxyethoxy)-1-phenylethanone (2f)



${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-cyclopropylethanone (3a)

${ }^{13}$ C NMR ( $\mathbf{1 2 5 . 8} \mathbf{~ M H z}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-cyclopropylethanone (3a)



${ }^{1} \mathrm{H}$ NMR (500 MHz, $\mathrm{CDCl}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-cyclobutylethanone (3b)



${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-cyclobutylethanone (3b)

${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(Adamantan-1-yl)-2-(benzyloxy)ethanone (3c)

${ }^{13} \mathrm{C}$ NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ) Spectrum of 1-(Adamantan-1-yl)-2-(benzyloxy)ethanone (3c)


${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(Benzyloxy)-3-phenylpropan-2-one (3d)

${ }^{13}$ C NMR ( $\mathbf{1 2 5 . 8} \mathbf{~ M H z}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(Benzyloxy)-3-phenylpropan-2-one (3d)

${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(Benzo[d][1,3]dioxol-5-yl)-2-(benzyloxy)ethanone (3e)




${ }^{13}$ C NMR ( $125.8 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 1-(Benzo[d][1,3]dioxol-5-yl)-2-(benzyloxy)ethanone (3e)




${ }^{1}$ H NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-(4-methoxyphenyl)ethanone (3f)

${ }^{13}$ C NMR (125.8 MHz, $\mathrm{CDCl}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-(4-methoxyphenyl)ethanone (3f)


${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 4-(Benzyloxy)-2-methyl-3-oxobutan-2-yl acetate ( $\mathbf{3 g}$ )



$\qquad$
${ }^{13}$ C NMR ( $\mathbf{1 2 5 . 8} \mathbf{M H z}, \mathrm{CDCl}_{3}$ ) Spectrum of 4-(Benzyloxy)-2-methyl-3-oxobutan-2-yl acetate (3g)

${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-(furan-2-yl)ethanone (3h)

${ }^{13}$ C NMR (125.8 MHz, CDCl $_{3}$ ) Spectrum of 2-(Benzyloxy)-1-(furan-2-yl)ethanone (3h)

${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-(3-methylthiophen-2-yl)ethanone (3i)

${ }^{13}$ C NMR ( $\mathbf{1 2 5 . 8} \mathbf{~ M H z}$, CDCl $_{3}$ ) Spectrum of 2-(Benzyloxy)-1-(3-methylthiophen-2-yl)ethanone (3i)


${ }^{1} \mathrm{H}$ NMR ( $500 \mathrm{MHz}, \mathrm{CDCl}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-morpholinoethanone (3j)




${ }^{13}$ C NMR (125.8 MHz, CDCl $\mathbf{C l}_{3}$ ) Spectrum of 2-(Benzyloxy)-1-morpholinoethanone (3j)
