

PTSA-Catalyzed Reaction of Alkyl/Aryl Methyl Ketones with Aliphatic Alcohols in the Presence of Selenium Dioxide: A Protocol for the Generation of an α -Ketoacetals Library

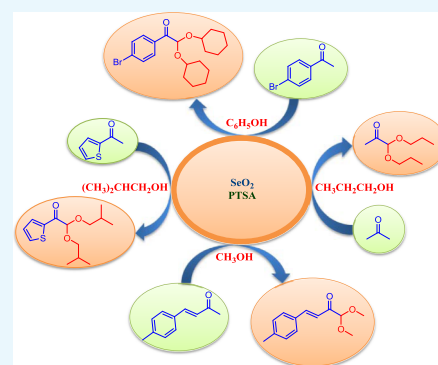
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Supporting Information

ABSTRACT: A novel approach has been developed for the synthesis of a wide range of α -ketoacetals by the reaction of alkyl/aryl methyl ketones and aliphatic alcohols in the presence of selenium dioxide catalyzed by *p*-toluenesulfonic acid. This method represents a general route to obtain a wide variety of α -ketoacetals in a simple, rapid, and practical manner. This approach is particularly attractive because of the easy availability of the starting materials, mild reaction temperature, and good yields of the products. The resulting α -ketoacetals are of much synthetic value as organic intermediates.

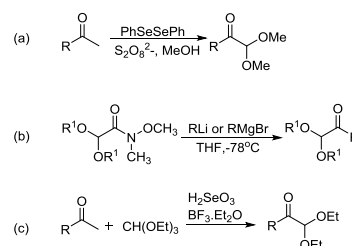


INTRODUCTION

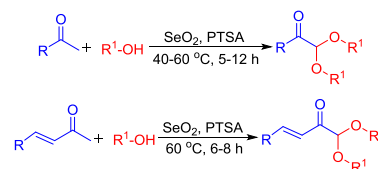
The α -ketoacetals are important functional moieties and are useful building blocks in organic synthesis. They are useful intermediates in that they provide an array of functional groups that are extremely valuable in organic syntheses. For instance the α -ketoacetals are a key intermediate in the synthesis of various biological active compounds such as chiral α -hydroxy acetals,¹ chiral α -amino acetals,² chiral auxiliaries,³ and cyanosilylation⁴ and also for the construction of important heterocycles.⁵ Several methods have been described for the preparation of α -ketoacetals.^{5–9} Goswami et al., reported the synthesis of aliphatic α -ketoacetals starting from ketones via a two step procedure using SeO_2 .^{5a,b} Tiecco and co-workers reported the synthesis of α -ketoacetals catalyzed by diphenyldiselenide and an excess of ammonium peroxydisulfate under reflux conditions (Scheme 1a).⁷ Ayala-Mata and group employed Weinreb amides as a starting material for the synthesis of α -ketoacetals (Scheme 1b).⁸ More recently, we have reported the synthesis of phenylglyoxal diethylacetals via the reaction of aromatic ketones with triethylorthoformate in the presence of H_2SeO_3 catalyzed by $\text{BF}_3 \cdot \text{Et}_2\text{O}$ (Scheme 1c).⁹ This method, though simple is limited by the use of triethylorthoformate as the sole source of alkoxide nucleophile. Generally, most of the other reported methods involved multistep reactions. Besides, the high cost of the reagents coupled with sensitive reaction conditions limit their scope of applications. Development of an alternative method for the synthesis of α -ketoacetals with wide substrate scope starting

Scheme 1. Synthesis of α -Ketoacetals

Previous Work



Our Work



from simple and easily available starting materials would therefore be a welcome addition to synthetic organic chemists.

The reactive behavior of SeO_2 toward organic substrates in the presence of a Lewis acid or a strong organic acid, however, has found few or no mention at all in the literature. Earlier we

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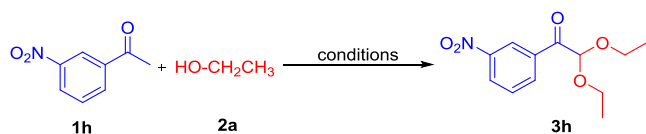
reported an efficient regio-selective protocol for the C–C bond formation by an unexpected α , α -diarylation of aromatic ketones in the presence of selenium dioxide, catalyzed by boron trifluoride etherate.¹⁰ This unusual reactivity of SeO₂ toward aromatic ketones in the presence of a Lewis acid prompted us to explore its reactions with organic substrates by varying the nature of the acid and the solvent used. Since then, we have further demonstrated this unique reactivity of SeO₂ with aromatic ketones by changing the acid catalyst and/or the solvent used, leading to the formation of important organic intermediates.¹¹

As part of our on-going investigation on the synthetic utility of selenium dioxide,^{9–11} we wish to report here an efficient general method for the synthesis of α -ketoacetals from a wide range of ketones and alcohols in presence of SeO₂ catalyzed by *p*-toluenesulfonic acid (PTSA) (Scheme 1).

RESULTS AND DISCUSSION

Initially, when a mixture of 1-(3-nitrophenyl)ethanone (**1h**) (1.0 mmol), ethanol (**2a**) (1 mL), SeO₂ (0.5 equiv), and PTSA (0.5 equiv) was stirred at room temperature for 12 h, only a trace amount of the product was formed as observed by thin-layer chromatography (TLC) (Table 1, entry 1). When the

Table 1. Optimization of the Reaction Conditions^a



entry	oxidant (equiv)	catalyst (equiv)	temperature °C	t (h)	yield (%)
1	SeO ₂ (0.5)	PTSA (0.5)	rt	12	trace
2	SeO ₂ (0.5)	PTSA (0.5)	60	8	52
3	SeO ₂ (0.5)	PTSA (1.0)	60	12	61
4	SeO ₂ (0.7)	PTSA (1.0)	60	8	85
5	SeO ₂ (0.7)	PTSA (1.0)	80	8	83
6	SeO ₂ (1.0)	PTSA (1.0)	60	8	86

^aReaction conditions: ketones (**1**) (1.0 mmol), ethanol (**2**) (1 mL).

reaction temperature was raised to 60 °C for 8 h, the yield increased to 52% (Table 1, entry 2). Any further increase in the temperature did not increase the yield any further. Further optimization reactions by varying the number of equivalents of selenium dioxide and PTSA (Table 1, entry 2–6) were carried out. It was found that (0.7 equiv) of SeO₂ and (1.0 equiv) of PTSA gave optimum yield of the product (85%) (Table 1, entry 4). To establish whether PTSA is unique to this reaction, other organic acids such as TFA and CH₃COOH were employed and in both cases the desired product was not formed. It may be noted that the use of either SeO₂ or PTSA alone failed to give the desired product.

With the optimized conditions in hand, the scope and generality of the reaction of aryl methyl ketones with alcohols were investigated. First, the reaction demonstrated a wide substrate scope in terms of the aromatic ketone (**1**) with ethanol (**2a**) (Scheme 2). Aryl methyl ketones bearing electron-neutral (4-H), electron-donating [e.g., 4-Me, 4-OH, 4-OMe, and 3,4-(OMe)₂] or electron-withdrawing (e.g., 3-NO₂ and 4-NO₂) substituent in the ring were successfully converted to the corresponding products **3a–b**, **3e–f**, and **3g–i** in moderate-to-good yields (59–90%). The procedure was

also found to be compatible with halogenated aryl methyl ketone (4-Br, 4-Cl), which gave the desired products in good yields (**3c**, 71%; **3d**, 74%). The scope of the reaction was also extended to sterically hindered 2-acetyl naphthalene (**1j**), which readily yielded product **3j** in 78% yield.

Second, homologs of alcohol from the aliphatic series (Scheme 2) were randomly selected. Both primary and secondary aliphatic alcohols easily undergo double nucleophilic attack on the α -carbon atom of the ketone to give the desired products **3k–w** in moderate-to-good yields (60–89%). Methanol (**2b**), 1-propanol (**2c**), 1-butanol (**2d**), 1-hexanol (**2e**), benzyl alcohol (**2f**), and iso-butanol (**2g**) readily reacted with aryl methyl ketones bearing electron-withdrawing group (3-NO₂), electron-donating groups [3,4-(OMe)₂], and halogenated aryl methyl ketones (4-Br, 4-Cl) to give the corresponding products **3k–p** and **3s–t** in moderate-to-good yields (60–88%). Ordinarily one would have expected that long-chain aliphatic alcohol would not react because of steric consideration. Surprisingly however, the reaction with cetyl alcohol (**2h**) proceeded cleanly in 8h to give excellent yields of the desired product (**3v**, 85%; **3w**, 89%). Similarly, secondary alcohols such as iso-propanol (**2i**) and cyclohexanol (**2j**) also reacted smoothly to give the corresponding products **3q–r** and **3u** in satisfactory yields (62–79%). However, tertiary aliphatic alcohol (**2k**) failed to react evidently because of the bulky nature of the substituent adjacent to the reacting nucleophile **3x**.

The methodology was further extended to the reaction of hetero-aryl methyl ketones (**4**) with aliphatic alcohol (**2**) which effortlessly gave the desired products **5a–d** in moderate-to-good yields (72–93%) (Scheme 3).

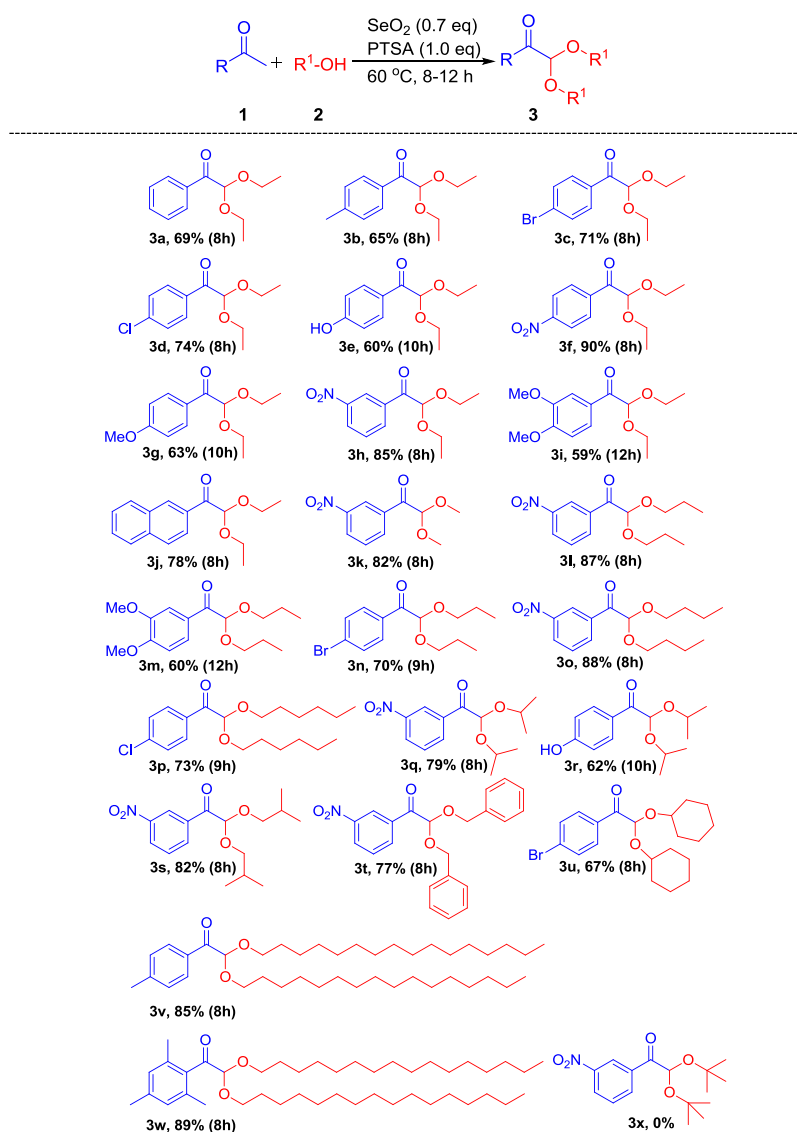
To further explore the efficacy of the method, reactions of substituted benzylidene acetones (**6**) with alcohols (**2**) were performed (Scheme 4). Benzylidene acetone bearing electron neutral (4-H), electron donating (e.g., 4-Me and 4-OMe), electron withdrawing (4-NO₂), or halogenated group (4-Br) all gave the expected products **7a–e** in good yield (82–92%).

Having met with unprecedented success in the above reactions, we finally turned to the aliphatic ketones **8**. We were delighted to note that the method continues to hold good with representative examples of cyclic and acyclic aliphatic ketones (Scheme 5). Reactions with aliphatic ketones such as acetone (**8a**), ethyl methyl ketone (**8b**), and pentanone (**8c**) proceeded as expected to give the corresponding products **9a–d** in moderate yield (62–70%). Reactions with branched aliphatic ketones such as iso-butyl methyl ketone (**8d**) and isopentyl methyl ketone (**8e**) also gave the desired products (**9e**, 69%; **9f**, 67%) in good yields. Similarly, secondary aliphatic ketones 3-methyl-2-butanone (**8f**) and the strained cyclopropyl ketone (**8g**) afforded the desired products **9g** and **9h** in 65 and 73% yields respectively.

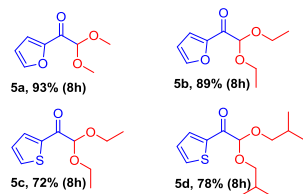
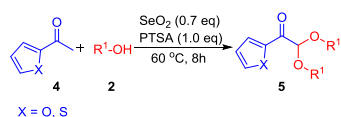
Previously, we had proposed the reactions to proceed via the intermediate **10** (Scheme 6) where precipitation of elemental selenium occurred. In the present work, particularly, the precipitation of elemental selenium was clean with no formation of colloidal Se. Although we have not succeeded in isolating the intermediate **10** so far, evidently the mechanism follows the same route as reported in our previous work (Scheme 6).^{9,10,11c}

CONCLUSION

We have developed a simple and efficient approach for the synthesis of α -ketoacetals from aryl/alkyl methyl ketones with

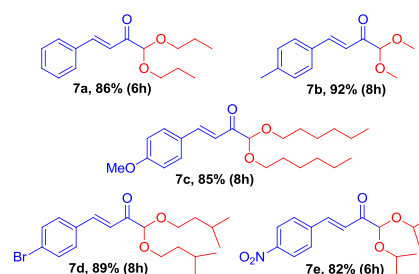
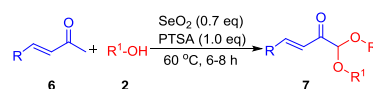
Scheme 2. Scope of Aryl Methyl Ketones That Couple with Alcohols^a

^aReaction conditions: ketones (1) (1.0 mmol), alcohols (2) (1 mL), SeO_2 (0.7 equiv), PTSA (1.0 equiv) at 60 °C, 8–12 h.

Scheme 3. Scope of Heteroaryl Methyl Ketones That Couple with Alcohols^a

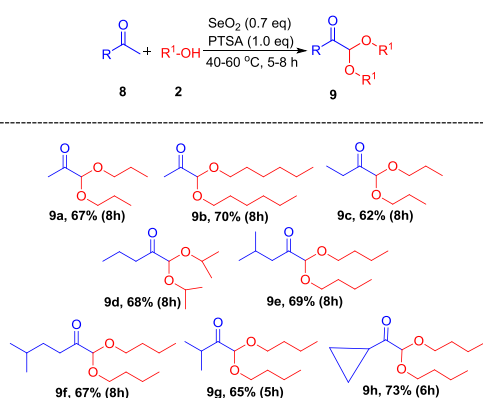
^aReaction conditions: ketones (4) (1.0 mmol), alcohol (2) (1 mL), SeO_2 (0.7 equiv), PTSA (1.0 equiv) at 60 °C, 8 h.

aliphatic alcohols in the presence of selenium dioxide and PTSA. The methodology further demonstrates its generality for a diversity-oriented synthesis of novel α -ketoacetals library.

Scheme 4. Scope of α , β -Unsaturated Ketones That Couple with Alcohols^a

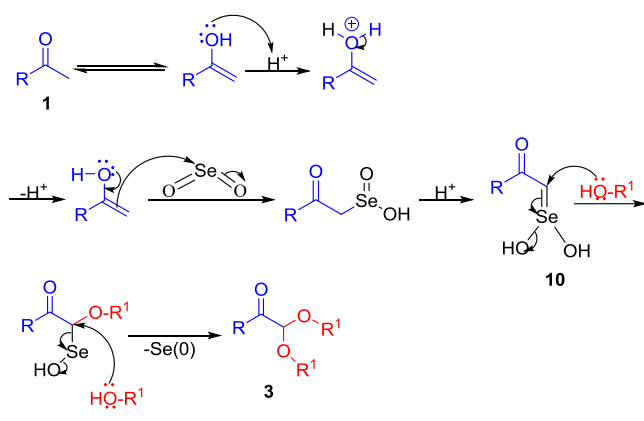
^aReaction conditions: ketones (6) (1.0 mmol), alcohols (2) (1 mL), SeO_2 (0.7 equiv), PTSA (1.0 equiv) at 60 °C, 6–8 h.

Scheme 5. Scope of Aliphatic Ketones That Couple with Alcohols^a



^aReaction conditions: ketones (8) (1.0 mmol), alcohol (2) (1 mL), SeO_2 (0.7 equiv), PTSA (1.0 equiv), 40–60 °C, 5–8 h.

Scheme 6. Plausible Mechanism



The reactions exhibited wide substrate tolerance in both the reactants. The easy availability of the reactants used coupled with the simplicity of the reaction procedure involved will certainly make this methodology a more attractive and viable alternative.

EXPERIMENTAL SECTION

General Methods. All reagents were purchased from Sigma-Aldrich, TCI Chemicals, and Alfa Aesar and were used without further purification unless noted. Melting points were recorded by the open capillary tube method and are uncorrected. IR spectra were recorded on a PerkinElmer Spectrum 400 FTIR instrument. ^1H NMR and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra were recorded on a Bruker Avance II-400 spectrometer in CDCl_3 with TMS as the internal standard. Mass spectral data were obtained with a Waters UPLC-TQD mass spectrometer (ESI-MS). Elemental analyses were carried out on PerkinElmer 2400 Series II. High-resolution mass spectra (ESI-HRMS) were recorded on a MaXis (Bruker Daltonics, Bremen, Germany) time of flight mass spectrometer. All reactions were monitored by TLC using precoated aluminium sheets (silica gel 60 F254 0.2 mm thickness). Column chromatography was carried out on silica gel (100–200 mesh), and flash chromatography was carried out on silica gel (230–400 mesh).

General Procedure for the Synthesis of Compounds 3, 5, 7, and 9. A mixture of the aromatic ketone 1 or 4 or α ,

β -unsaturated ketones 6 or aliphatic ketones 8 (1.0 mmol), selenium dioxide (77 mg, 0.7 mmol, 0.7 equiv), PTSA (190 mg, 1.0 mmol, 1.0 equiv), and alcohol 2 (1 mL) was allowed to stir at 40–60 °C for 5–12 h. Then, a thick precipitate of elemental selenium settled at the bottom of the flask which was then filtered off, washed with ethyl acetate (2×10 mL), and the combined filtrate was transferred to a separating funnel, washed with conc. sodium bicarbonate solution (2×10 mL) followed by brine (2×10 mL). The organic layer was separated, dried over anhydrous Na_2SO_4 , and concentrated in vacuo. The crude mass was purified by column chromatography using silica gel (100–200 mesh) or flash chromatography using silica gel (230–400 mesh) and ethyl acetate–hexane as the eluent.

2,2-Diethoxy-1-phenylethanone (3a).⁹ The title compound was prepared via the general procedure from acetophenone 1a (120 mg, 1.0 mmol) and ethanol 2a (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (143 mg, 69% yield); IR (KBr film): 3064, 2979, 2883, 1686, 1599, 1449, 1360, 1266, 1119, 1062 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.17–7.43 (m, 5H), 5.29 (s, 1H), 3.80–3.64 (m, 4H), 1.25 (t, $J = 7.2$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 194.0, 133.7, 133.4, 129.7, 128.3, 102.3, 63.1, 15.2 ppm. MS (ES^+): for $\text{C}_{12}\text{H}_{16}\text{O}_3$, 208.1; found m/z , 231.2 [$M + \text{Na}$]⁺.

2,2-Diethoxy-1-(*p*-tolyl)ethanone (3b).⁹ The title compound was prepared via the general procedure from 4-methylacetophenone 1b (134 mg, 1.0 mmol) and ethanol 2a (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (144 mg, 65% yield); IR (KBr film): 3030, 3004, 2922, 2870, 1682, 1606, 1574, 1428, 1405, 1358, 1268, 1181, 1122, 1018 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.99 (d, $J = 7.2$ Hz, 2H), 7.18 (d, $J = 8.8$ Hz, 2H), 5.21 (s, 1H), 3.71–3.56 (m, 4H), 2.34 (s, 3H), 1.17 (t, $J = 7.2$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 192.6, 143.3, 130.2, 128.8, 128.0, 101.2, 62.0, 20.7, 14.1 ppm. MS (ES^+): for $\text{C}_{13}\text{H}_{18}\text{O}_3$, 222.1; found m/z , 245.1 [$M + \text{Na}$]⁺.

1-(4-Bromophenyl)-2,2-diethoxyethanone (3c).⁹ The title compound was prepared via the general procedure from 4-bromoacetophenone 1c (199 mg, 1.0 mmol) and ethanol 2a (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (203 mg, 71% yield); IR (KBr film): 3094, 2979, 2931, 2883, 1693, 1585, 1484, 1400, 1287, 1118, 1070, 1011 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.97 (d, $J = 8.8$ Hz, 2H), 7.51 (d, $J = 8.4$ Hz, 2H), 5.10 (s, 1H), 3.73–3.53 (m, 4H), 1.16 (t, $J = 7.2$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 193.2, 132.2, 131.6, 131.4, 128.7, 103.0, 63.5, 15.2 ppm. MS (ES^+): for $\text{C}_{12}\text{H}_{15}\text{BrO}_3$, 286.0; found m/z , 309.1 [$M + \text{Na}$]⁺.

1-(4-Chlorophenyl)-2,2-diethoxyethanone (3d).⁹ The title compound was prepared via the general procedure from 4-chloroacetophenone 1d (154 mg, 1.0 mmol) and ethanol 2a (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (178 mg, 74% yield); IR (KBr film): 3077, 2980, 2931, 2883, 1693, 1589, 1570, 1488, 1403, 1286, 1200, 1092, 1015 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.05 (d, $J = 9.6$ Hz, 2H), 7.35 (d, $J = 8.4$ Hz, 2H), 5.10 (s, 1H), 3.73–3.54 (m, 4H), 1.17 (t, $J = 7.6$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 193.0, 139.9, 131.8, 131.3, 128.6, 103.0, 63.5, 15.1 ppm. MS (ES^+): for $\text{C}_{12}\text{H}_{15}\text{ClO}_3$, 242.0; found m/z , 265.2 [$M + \text{Na}$]⁺.

2,2-Diethoxy-1-(4-hydroxyphenyl)ethanone (3e).⁹ The title compound was prepared via the general procedure from

4-hydroxy-acetophenone **1e** (136 mg, 1.0 mmol) and ethanol **2a** (1 mL). The product was isolated by flash chromatography using 2% ethyl acetate/hexane as an eluent; oil (134 mg, 60% yield); IR (KBr film): 3330, 2982, 2935, 2896, 1675, 1602, 1515, 1443, 1372, 1290, 1164, 1056 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.00 (d, $J = 8.4$ Hz, 2H), 7.65 (s br, 1H), 6.83 (d, $J = 8.4$ Hz, 2H), 5.28 (s, 1H), 3.68–3.53 (m, 4H), 1.15 (t, $J = 7.2$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 193.4, 161.8, 132.4, 126.0, 115.5, 101.2, 62.8, 15.1 ppm. MS (ES^+): for $\text{C}_{12}\text{H}_{16}\text{O}_4$, 224.1; found m/z , 225.0 $[\text{M} + \text{H}]^+$.

2,2-Diethoxy-1-(4-nitrophenyl)ethanone (3f).⁹ The title compound was prepared via the general procedure from 4-nitro-acetophenone **1f** (165 mg, 1.0 mmol) and ethanol **2a** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (227 mg, 90% yield); IR (KBr film): 3107, 3051, 2978, 2939, 2903, 1702, 1603, 1528, 1481, 1370, 1345, 1329, 1278, 1109, 1053, 1017 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.28 (d, $J = 9.6$ Hz, 2H), 8.21 (d, $J = 8.4$ Hz, 2H), 5.07 (s, 1H), 3.77–3.54 (m, 4H), 1.18 (t, $J = 7.2$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 192.7, 150.3, 138.0, 131.0, 123.3, 103.7, 64.1, 15.1 ppm. MS (ES^+): for $\text{C}_{12}\text{H}_{15}\text{NO}_5$, 253.1; found m/z , 276.1 $[\text{M} + \text{Na}]^+$.

2,2-Diethoxy-1-(4-methoxyphenyl)ethanone (3g).⁹ The title compound was prepared via the general procedure from 4-methoxy-acetophenone **1g** (150 mg, 1.0 mmol) and ethanol **2a** (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (149 mg, 63% yield); IR (KBr film): 2978, 2935, 2896, 2844, 1679, 1601, 1575, 1511, 1460, 1422, 1308, 1260, 1173, 1114, 1062, 1029 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.08 (d, $J = 8.8$ Hz, 2H), 6.85 (d, $J = 8.4$ Hz, 2H), 5.17 (s, 1H), 3.80 (s, 3H), 3.71–3.55 (m, 4H), 1.17 (t, $J = 7.6$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 192.7, 163.7, 132.1, 130.6, 113.5, 102.5, 63.0, 55.4, 15.2 ppm. MS (ES^+): for $\text{C}_{13}\text{H}_{18}\text{O}_4$, 238.1; found m/z , 261.1 $[\text{M} + \text{Na}]^+$.

2,2-Diethoxy-1-(3-nitrophenyl)ethanone (3h).⁹ The title compound was prepared via the general procedure from 3-nitro-acetophenone **1h** (165 mg, 1.0 mmol) and ethanol **2a** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (215 mg, 85% yield); IR (KBr film): 3088, 2980, 2935, 2885, 1703, 1614, 1580, 1534, 1478, 1441, 1351, 1271, 1228, 1062 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.96 (s, 1H), 8.43 (d, $J = 8.8$ Hz, 1H), 8.34 (d, $J = 6.0$ Hz, 1H), 7.58 (t, $J = 8.8$ Hz, 1H), 5.09 (s, 1H), 3.80–3.57 (m, 4H), 1.19 (t, $J = 7.2$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 192.0, 148.1, 135.6, 134.6, 129.4, 127.5, 125.0, 103.5, 64.1, 15.1 ppm. MS (ES^+): for $\text{C}_{12}\text{H}_{13}\text{NO}_5$, 253.1; found m/z , 276.1 $[\text{M} + \text{Na}]^+$.

2,2-Diethoxy-1-(3,4-dimethoxyphenyl)ethanone (3i). The title compound was prepared via the general procedure from 3,4-dimethoxy-acetophenone **1i** (180 mg, 1.0 mmol) and ethanol **2a** (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (158 mg, 59% yield); IR (KBr film): 2977, 2935, 2844, 1681, 1595, 1515, 1464, 1421, 1343, 1273, 1229, 1174, 1121, 1060, 1023 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.84 (d, $J = 8.8$ Hz, 1H), 7.59 (s, 1H), 6.82 (d, $J = 8.8$ Hz, 1H), 5.19 (s, 1H), 3.88 (s, 3H), 3.86 (s, 3H), 3.72–3.54 (m, 4H), 1.18 (t, $J = 6.0$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 192.6, 153.5, 148.7, 126.7, 124.8, 111.4, 109.9, 102.4, 63.0, 56.0, 55.9, 15.2 ppm. MS (ES^+): for $\text{C}_{14}\text{H}_{20}\text{O}_5$, 268.1; found m/z , 269.7 $[\text{M} + \text{H}]^+$. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{14}\text{H}_{20}\text{O}_5\text{Na}$, 291.1208; found, 291.1209.

2,2-Diethoxy-1-(naphthalen-2-yl)ethanone (3j).⁹ The title compound was prepared via the general procedure from 2-acetylnaphthalene **1j** (170 mg, 1.0 mmol) and ethanol **2a** (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (201 mg, 78% yield); IR (KBr film): 3059, 2977, 2882, 1681, 1627, 1597, 1468, 1438, 1361, 1281, 1115, 1061 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.71 (s, 1H), 8.08–7.46 (m, 6H), 5.34 (s, 1H), 3.76–3.60 (m, 4H), 1.19 (t, $J = 7.2$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 194.0, 135.8, 132.0, 129.9, 129.5, 128.6, 128.4, 128.1, 127.7, 126.6, 124.9, 102.4, 63.1, 15.2 ppm. MS (ES^+): for $\text{C}_{16}\text{H}_{18}\text{O}_3$, 258.1; found m/z , 281.0 $[\text{M} + \text{Na}]^+$.

2,2-Dimethoxy-1-(3-nitrophenyl)ethanone (3k). The title compound was prepared via the general procedure from 3-nitro-acetophenone **1h** (165 mg, 1.0 mmol) and methanol **2b** (1 mL). The product was isolated by flash chromatography using 2% ethyl acetate/hexane as an eluent; oil (184 mg, 82% yield); IR (KBr film): 3088, 2942, 2837, 1697, 1615, 1532, 1351, 1192, 1093 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.90 (s, 1H), 8.40–8.35 (m, 2H), 7.60 (t, $J = 7.6$ Hz, 1H), 5.03 (s, 1H), 3.46 (s, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 191.5, 148.2, 135.4, 134.6, 129.6, 127.7, 124.8, 104.8, 55.4 ppm. MS (ES^+): for $\text{C}_{10}\text{H}_{11}\text{NO}_5$, 225.0; found m/z , 248.0 $[\text{M} + \text{Na}]^+$.

2,2-Dipropoxy-1-(3-nitrophenyl)ethanone (3l). The title compound was prepared via the general procedure from 3-nitro-acetophenone **1h** (165 mg, 1.0 mmol) and 1-propanol **2c** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (244 mg, 87% yield); IR (KBr film): 3120, 2967, 2937, 2878, 1702, 1615, 1534, 1477, 1438, 1350, 1300, 1267, 1191, 1122, 1100, 1071 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.97 (s, 1H), 8.43 (d, $J = 7.2$ Hz, 1H), 8.34 (d, $J = 6.0$ Hz, 1H), 7.58 (t, $J = 7.6$ Hz, 1H), 5.05 (s, 1H), 3.69–3.45 (m, 4H), 1.62–1.53 (m, 4H), 0.84 (t, $J = 7.2$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 192.1, 148.1, 135.6, 134.6, 129.4, 127.5, 125.1, 104.2, 70.4, 22.9, 10.5 ppm. MS (ES^+): for $\text{C}_{14}\text{H}_{19}\text{NO}_5$, 281.1; found m/z , 299.0 $[\text{M} + \text{NH}_4]^+$. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{14}\text{H}_{19}\text{NO}_5\text{Na}$, 304.1161; found, 304.1158.

1-(3,4-Dimethoxyphenyl)-2,2-dipropoxyethanone (3m). The title compound was prepared via the general procedure from 3,4-dimethoxy-acetophenone **1h** (180 mg, 1.0 mmol) and 1-propanol **2c** (1 mL). The product was isolated by flash chromatography using 3% ethyl acetate/hexane as an eluent; oil (177 mg, 60% yield); IR (KBr film): 2964, 2939, 2877, 1680, 1595, 1515, 1464, 1421, 1344, 1273, 1228, 1119, 1069, 1024 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.84 (d, $J = 8.4$ Hz, 1H), 7.61 (s, 1H), 6.82 (d, $J = 8.4$ Hz, 1H), 5.14 (s, 1H), 3.88 (s, 3H), 3.86 (s, 3H), 3.61–3.43 (m, 4H), 1.61–1.52 (m, 4H), 0.84 (t, $J = 7.2$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 191.7, 152.5, 147.6, 125.7, 123.9, 110.6, 108.9, 102.2, 68.4, 55.0, 54.9, 21.9, 9.5 ppm. MS (ES^+): for $\text{C}_{16}\text{H}_{24}\text{O}_5$, 296.1; found m/z , 297.2 $[\text{M} + \text{H}]^+$. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{16}\text{H}_{24}\text{O}_5\text{Na}$, 319.1521; found, 319.1523.

1-(4-Bromophenyl)-2,2-dipropoxyethanone (3n). The title compound was prepared via the general procedure from 4-bromo-acetophenone **1c** (199 mg, 1.0 mmol) and 1-propanol **2c** (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (220 mg, 70% yield); IR (KBr film): 2965, 2935, 2877, 1690, 1586, 1483, 1396, 1265, 1116, 1071, 1010 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.04 (d, $J = 8.8$ Hz, 2H), 7.59 (d, $J = 9.2$ Hz, 2H), 5.13 (s, 1H), 3.69–3.48 (m, 4H), 1.67–1.58 (m, 4H), 0.90 (t,

$J = 7.6$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 192.2, 131.2, 130.5, 130.4, 127.6, 102.7, 68.8, 21.8, 9.5 ppm. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{14}\text{H}_{19}\text{BrO}_3\text{Na}$, 337.0415; found, 337.0411.

2,2-Dibutoxy-1-(3-nitrophenyl)ethanone (3o). The title compound was prepared via the general procedure from 3-nitro-acetophenone **1h** (165 mg, 1.0 mmol) and 1-butanol **2d** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (271 mg, 88% yield); IR (KBr film): 3087, 2960, 2935, 2874, 1702, 1614, 1580, 1534, 1466, 1350, 1299, 1269, 1228, 1125, 1073 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.97 (s, 1H), 8.42 (d, $J = 7.2$ Hz, 1H), 8.34 (d, $J = 6.0$ Hz, 1H), 7.58 (t, $J = 7.2$ Hz, 1H), 5.03 (s, 1H), 3.73–3.48 (m, 4H), 1.56–1.49 (m, 4H), 1.33–1.24 (m, 4H), 0.82 (t, $J = 7.2$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 192.1, 148.1, 135.6, 134.6, 129.4, 127.5, 125.1, 104.3, 68.5, 31.6, 19.1, 13.7 ppm. MS (ES^+): for $\text{C}_{16}\text{H}_{23}\text{NO}_5$, 309.1; found m/z , 309.0 $[\text{M}]^+$. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{16}\text{H}_{23}\text{NO}_5\text{Na}$, 332.1474; found, 332.1736.

1-(4-Chlorophenyl)-2,2-bis(hexyloxy)ethanone (3p). The title compound was prepared via the general procedure from 4-chloro-acetophenone **1d** (154 mg, 1.0 mmol) and 1-hexanol **2e** (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (258 mg, 73% yield); IR (KBr film): 3136, 2956, 2932, 2861, 1694, 1589, 1401, 1385, 1284, 1192, 1121, 1092 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.04 (d, $J = 8.4$ Hz, 2H), 7.34 (d, $J = 8.4$ Hz, 2H), 5.04 (s, 1H), 3.64–3.44 (m, 4H), 1.56–1.49 (m, 4H), 1.27–1.15 (m, 12H), 0.78 (t, $J = 6.8$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ -NMR (100 MHz, CDCl_3): δ 193.1, 139.8, 131.8, 131.3, 128.5, 103.6, 68.1, 31.4, 29.5, 25, 6, 22.5, 13.9 ppm; MS (ES^+): for $\text{C}_{20}\text{H}_{31}\text{ClO}_3$, 354.2; found m/z , 377.3 $[\text{M} + \text{Na}]^+$. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{20}\text{H}_{31}\text{ClO}_3\text{Na}$, 377.1859; found, 377.1856.

2,2-Diisopropoxy-1-(3-nitrophenyl)ethanone (3q). The title compound was prepared via the general procedure from 3-nitro-acetophenone **1h** (165 mg, 1.0 mmol) and isopropanol **2i** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (222 mg, 79% yield); IR (KBr film): 3088, 2977, 2934, 2892, 1703, 1615, 1580, 1535, 1467, 1438, 1378, 1351, 1320, 1269, 1180, 1122, 1099, 1034 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 9.01 (s, 1H), 8.48–8.31 (m, 2H), 7.56 (t, $J = 7.2$ Hz, 1H), 5.07 (s, 1H), 3.94–3.85 (m, 2H), 1.22 (d, $J = 6.0$ Hz, 6H) 1.07 (d, $J = 6.0$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 192.7, 148.0, 136.1, 134.4, 129.2, 127.3, 125.5, 102.2, 71.0, 22.9, 22.2 ppm; MS (ES^+): for $\text{C}_{14}\text{H}_{19}\text{NO}_5$, 281.1; found m/z , 299 $[\text{M} + \text{NH}_4]^+$. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{14}\text{H}_{19}\text{NO}_5\text{Na}$, 304.1161; found, 304.1162.

1-(4-Hydroxyphenyl)-2,2-diisopropoxyethanone (3r). The title compound was prepared via the general procedure from 4-hydroxy-acetophenone **1e** (136 mg, 1.0 mmol) and isopropanol **2i** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (156 mg, 62% yield); IR (KBr film): 3339, 2975, 2933, 1674, 1602, 1582, 1515, 1443, 1438, 1383, 1288, 1241, 1171, 1119, 1038 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.15 (d, $J = 8.8$ Hz, 2H), 6.87 (d, $J = 8.8$ Hz, 2H), 5.20 (s, 1H), 3.97–3.90 (m, 12H), 1.24 (d, $J = 6.0$ Hz, 6H) 1.14 (d, $J = 6.0$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 194.6, 161.3, 133.2, 126.2, 115.4, 101.6, 70.5, 23.1, 22.5 ppm. MS (ES^+): for $\text{C}_{14}\text{H}_{20}\text{O}_4$, 252.1; found m/z , 275.0 $[\text{M} + \text{Na}]^+$. HRMS (ESI)

m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{14}\text{H}_{20}\text{O}_4\text{Na}$, 275.1259; found, 275.1257.

2,2-Diisobutoxy-1-(3-nitrophenyl)ethanone (3s). The title compound was prepared via the general procedure from 3-nitro-acetophenone **1h** (165 mg, 1.0 mmol) and iso-butanol **2g** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (253 mg, 82% yield); IR (KBr film): 3090, 2960, 2875, 1703, 1615, 1580, 1536, 1472, 1438, 1349, 1301, 1266, 1055 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 9.00 (s, 1H), 8.43 (d, $J = 7.2$ Hz, 1H), 8.35–8.33 (m, 1H), 7.58 (t, $J = 7.2$ Hz, 1H), 4.99 (s, 1H), 3.50–3.25 (m, 4H), 1.88–1.78 (m, 2H), 0.83 (t, $J = 6$ Hz, 12H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 192.0, 148.1, 135.6, 134.6, 129.3, 127.5, 125.2, 104.8, 75.5, 28.5, 19.2, 19.1 ppm. MS (ES^+): for $\text{C}_{16}\text{H}_{23}\text{NO}_5$, 309.1, found m/z , 327.1 $[\text{M} + \text{NH}_4]^+$. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{16}\text{H}_{23}\text{NO}_5\text{Na}$, 332.3518; found, 332.3514.

2,2-Bis(benzyloxy)-1-(3-nitrophenyl)ethanone (3t).^{4c} The title compound was prepared via the general procedure from 3-nitro-acetophenone **1h** (165 mg, 1.0 mmol) and benzyl alcohol **2f** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (290 mg, 77% yield); IR (KBr film): 3088, 3034, 2931, 2874, 1698, 1614, 1580, 1532, 1497, 1454, 1350, 1257, 1124, 1101 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.93 (s, 1H), 8.38–8.31 (m, 2H), 7.54 (t, $J = 7.6$ Hz, 1H), 7.25 (s, 5H), 7.24 (s, 5H), 5.26 (s, 1H), 4.72–4.58 (m, 4H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 191.6, 148.1, 136.3, 135.5, 134.6, 129.5, 128.6, 128.3, 128.2, 127.6, 125.0, 101.6, 70.0 ppm. MS (ES^+): for $\text{C}_{22}\text{H}_{19}\text{NO}_5$, 377.13; found m/z , 395.42 $[\text{M} + \text{NH}_4]^+$.

1-(4-Bromophenyl)-2,2-bis(cyclohexyloxy)ethanone (3u). The title compound was prepared via the general procedure from 4-bromo-acetophenone **1c** (199 mg, 1.0 mmol) and cyclohexanol **2j** (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; off-white solid (264 mg, 67% yield); mp 47–49 °C; IR (KBr film): 2933, 2856, 1689, 1585, 1449, 1281, 1117, 1038 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 8.03 (d, $J = 8.8$ Hz, 2H), 7.50 (d, $J = 8.8$ Hz, 2H), 5.10 (s, 1H), 3.57–3.50 (m, 2H), 1.85–1.11 (m, 20H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 194.3, 132.0, 131.9, 131.4, 128.4, 102.2, 76.4, 32.9, 32.2, 25.4 ppm. HRMS (ESI) m/z : $[\text{M} + \text{Na}]^+$ calcd for $\text{C}_{20}\text{H}_{27}\text{BrO}_3\text{Na}$, 417.1041; found, 417.1034.

2,2-Bis(hexadecyloxy)-1-(p-tolyl)ethanone (3v). The title compound was prepared via the general procedure from 4-methyl-acetophenone **1b** (134 mg, 1.0 mmol) and cetyl alcohol **2h** (0.5 g). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; white solid (522 mg, 85% yield); mp 43–45 °C; IR (KBr film): 3054, 2955, 2917, 2850, 1689, 1605, 1471, 1242, 1132, 1041 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3): δ 7.98 (d, $J = 8.4$ Hz, 2H), 7.17 (d, $J = 8$ Hz, 2H), 5.15 (s, 1H), 3.60–3.47 (m, 4H), 2.33 (s, 3H), 1.56–1.49 (m, 4H), 1.22–1.16 (m, 52H), 0.80 (t, $J = 6.8$ Hz, 6H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 193.8, 144.2, 131.2, 129.9, 129.0, 102.6, 67.6, 31.9, 29.72, 29.70, 29.68, 29.64, 29.61, 29.5, 29.38, 29.33, 26.0, 22.7, 21.7, 14.1 ppm. MS (ES^+): for $\text{C}_{41}\text{H}_{74}\text{O}_3$, 614.5; found m/z , 615.8 $[\text{M} + \text{H}]^+$. Anal. Calcd for $\text{C}_{41}\text{H}_{74}\text{O}_3$: C, 80.07; H, 12.13; O, 7.80. Found: C, 80.21; H, 12.35; O, 7.75.

2,2-Bis(hexadecyloxy)-1-mesitylethanone (3w). The title compound was prepared via the general procedure from 2,4,6-trimethyl-acetophenone **1c** (162 mg, 1.0 mmol) and cetyl alcohol **2h** (0.5 g). The product was isolated by flash

chromatography using 1% ethyl acetate/hexane as an eluent; off-white solid (571 mg, 89% yield); mp 29–31 °C; IR (KBr film): 2954, 2918, 2851, 1721, 1611, 1469, 1127, 1052 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 6.73 (s, 2H), 4.87 (s, 1H), 3.66–3.43 (m, 4H), 2.19 (s, 3H), 2.14 (s, 6H), 1.54–1.47 (m, 4H), 1.24–1.18 (m, 52H), 0.80 (t, *J* = 6.8 Hz, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 204.7, 138.5, 136.7, 133.9, 128.2, 102.2, 68.1, 31.9, 29.72, 29.70, 29.69, 29.67, 29.64, 29.61, 29.59, 29.3, 25.9, 22.7, 21.0, 19.5, 14.1 ppm. Anal. Calcd for C₄₃H₇₈O₃: C, 80.31; H, 12.23; O, 7.46. Found: C, 80.53; H, 12.25; O, 7.49.

1-(Furan-2-yl)-2,2-dimethoxyethanone (5a).⁷ The title compound was prepared via the general procedure from 2-acetylfuran **4a** (110 mg, 1.0 mmol) and methanol **2b** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (158 mg, 93% yield); IR (KBr film): 3138, 2942, 2837, 1688, 1565, 1466, 1396, 1273, 1131, 1066 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.60 (dd, *J* = 0.8 Hz, 1H), 7.38 (dd, *J* = 0.8 Hz, 1H), 6.49 (dd, *J* = 1.6 Hz, 1H), 5.03 (s, 1H), 3.40 (s, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 181.2, 148.9, 146.6, 120.1, 111.2, 101.1, 53.3 ppm. MS (ES⁺): for C₈H₁₀O₄, 170.0, found *m/z*, 193.1 [M + Na]⁺.

2,2-Diethoxy-1-(furan-2-yl)ethanone (5b).⁹ The title compound was prepared via the general procedure from 2-acetylfuran **4a** (110 mg, 1.0 mmol) and ethanol **2a** (1 mL). The product was isolated by flash chromatography using 2% ethyl acetate/hexane as an eluent; oil (176 mg, 89% yield); IR (KBr film): 3139, 2980, 2935, 2887, 1681, 1565, 1465, 1394, 1306, 1268, 1228, 1125, 1061 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.59 (s, 1H), 7.43 (d, *J* = 3.6 Hz, 1H), 6.49–6.46 (m, 1H), 5.08 (s, 1H), 3.73–3.57 (m, 4H), 1.19 (t, *J* = 7.2 Hz, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 183.0, 147.4, 121.2, 112.2, 101.2, 63.0, 15.1 ppm. MS (ES⁺): for C₁₀H₁₄O₄, 198.1; found *m/z*, 199.0 [M + H]⁺.

2,2-Diethoxy-1-(thiophen-2-yl)ethanone (5c).⁹ The title compound was prepared via the general procedure from 2-acetylthiophene **4b** (126 mg, 1.0 mmol) and ethanol **2a** (1 mL). The product was isolated by flash chromatography using 2% ethyl acetate/hexane as an eluent; oil (154 mg, 72% yield); IR (KBr film): 3102, 2979, 2931, 2883, 1670, 1509, 1413, 1363, 1323, 1288, 1240, 1115, 1059 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.00 (d, *J* = 3.6 Hz, 1H), 7.60 (d, *J* = 5.2 Hz, 1H), 7.07 (t, *J* = 4.8 Hz, 1H), 5.06 (s, 1H), 3.72–3.57 (m, 4H), 1.20 (t, *J* = 7.2 Hz, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 187.7, 139.6, 134.8, 134.6, 128.0, 102.2, 63.1, 15.1 ppm. MS (ES⁺): for C₁₀H₁₄O₃S, 214.0; found *m/z*, 236.7 [M + Na]⁺.

2,2-Diisobutoxy-1-(thiophen-2-yl)ethanone (5d). The title compound was prepared via the general procedure from 2-acetylthiophene **4b** (126 mg, 1.0 mmol) and iso-butanol **2g** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (210 mg, 78% yield); IR (KBr film): 3104, 2960, 2874, 1670, 1517, 1414, 1366, 1288, 1124, 1055 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.00 (dd, *J* = 1.2, 0.8 Hz, 1H), 7.60 (dd, *J* = 0.8, 1.2 Hz, 1H), 7.07–7.05 (m, 1H), 4.96 (s, 1H), 3.43–3.26 (m, 4H), 1.90–1.80 (m, 2H), 0.86–0.83 (m, 12H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 186.8, 138.6, 133.8, 133.4, 126.9, 102.4, 73.4, 27.5, 18.3, 18.2 ppm. MS (ES⁺): for C₁₄H₂₂O₃S, 270.1; found *m/z*, 293.0 [M + Na]⁺. HRMS (ESI) *m/z*: [M + Na]⁺ calcd for C₁₄H₂₂O₃Na, 293.1187; found, 293.1186.

(E)-4-Phenyl-1,1-dipropoxybut-3-en-2-one (7a).^{4b} The title compound was prepared via the general procedure from benzylidene acetone **6a** (146 mg, 1.0 mmol) and 1-propanol **2c** (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (225 mg, 86% yield); IR (KBr film): 3061, 3028, 2966, 2935, 2876, 1697, 1610, 1453, 1317, 1070 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.72 (d, *J* = 16.4 Hz, 1H), 7.53–7.31 (m, 5H), 7.03 (d, *J* = 16 Hz, 1H), 4.73 (s, 1H), 3.58–3.43 (m, 4H), 1.64–1.55 (m, 4H), 0.88 (t, *J* = 7.6 Hz, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 194.3, 144.7, 134.6, 130.6, 128.8, 128.5, 120.8, 102.8, 69.2, 22.9, 10.5 ppm. HRMS (ESI) *m/z*: [M + Na]⁺ calcd for C₁₆H₂₂O₃Na, 285.1467; found, 285.1463.

(E)-1,1-Dimethoxy-4-(p-tolyl)but-3-en-2-one (7b). The title compound was prepared via the general procedure from 4-methyl-benzylidene acetone **6b** (160 mg, 1.0 mmol) and methanol **2b** (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (202 mg, 92% yield); IR (KBr film): 2996, 2935, 2834, 1696, 1602, 1512, 1321, 1183, 1068 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.71 (d, *J* = 16.4 Hz, 1H), 7.43 (d, *J* = 8 Hz, 2H), 7.13 (d, *J* = 8 Hz, 2H), 6.96 (d, *J* = 16 Hz, 1H), 4.68 (s, 1H), 3.38 (s, 6H), 2.30 (s, 3H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 192.8, 144.2, 140.4, 130.7, 128.6, 127.7, 118.6, 102.6, 53.3, 20.5 ppm. HRMS (ESI) *m/z*: [M + Na]⁺ calcd for C₁₃H₁₆O₃Na, 243.0997; found, 243.0995.

(E)-1,1-Bis(hexyloxy)-4-(4-methoxyphenyl)but-3-en-2-one (7c). The title compound was prepared via the general procedure from 4-methoxy-benzylidene acetone **6c** (176 mg, 1.0 mmol) and 1-hexanol **2e** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (319 mg, 85% yield); IR (KBr film): 2956, 2931, 2863, 1694, 1596, 1512, 1463, 1256, 1174, 1066 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.69 (d, *J* = 16 Hz, 1H), 7.48 (d, *J* = 8.4 Hz, 2H), 6.90 (d, *J* = 16.4 Hz, 1H), 6.83 (d, *J* = 8.8 Hz, 2H), 4.71 (s, 1H), 3.77 (s, 3H), 3.61–3.44 (m, 4H), 1.59–1.52 (m, 4H), 1.33–1.19 (m, 12H), 0.80 (t, *J* = 6.8 Hz, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 194.3, 161.7, 144.5, 130.3, 127.4, 118.5, 114.3, 102.8, 67.5, 55.3, 31.5, 29.6, 25.5, 22.5, 14.0 ppm. HRMS (ESI) *m/z*: [M + Na]⁺ calcd for C₂₃H₃₆O₄Na, 399.2511; found, 399.2515.

(E)-4-(4-Bromophenyl)-1,1-bis(isopentyloxy)but-3-en-2-one (7d). The title compound was prepared via the general procedure from 4-bromo-benzylidene acetone **6d** (225 mg, 1.0 mmol) and iso-amyl alcohol **2l** (1 mL). The product was isolated by flash chromatography using 1% ethyl acetate/hexane as an eluent; oil (353 mg, 89% yield); IR (KBr film): 2958, 2872, 1700, 1610, 1486, 1313, 1165, 1068 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.63 (d, *J* = 16 Hz, 1H), 7.45 (d, *J* = 8.8 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 6.99 (d, *J* = 16.4 Hz, 1H), 4.68 (s, 1H), 3.67–3.46 (m, 4H), 1.70–1.59 (m, 2H), 1.48–1.43 (m, 4H), 0.84 (d, *J* = 2 Hz, 6H), 0.82 (d, *J* = 2.4 Hz, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 193.0, 142.2, 132.5, 131.1, 128.8, 123.9, 120.2, 101.9, 65.1, 37.3, 23.9, 21.5 ppm. HRMS (ESI) *m/z*: [M + H]⁺ calcd for C₂₀H₃₀BrO₃, 397.1378; found, 397.1343.

(E)-1,1-Diisopropoxy-4-(4-nitrophenyl)but-3-en-2-one (7e). The title compound was prepared via the general procedure from 4-nitro-benzylidene acetone **6e** (191 mg, 1.0 mmol) and iso-propanol **2i** (1 mL). The product was isolated by flash chromatography using 5% ethyl acetate/hexane as an eluent; oil (251 mg, 82% yield); IR (KBr film): 3110, 2974, 2932, 2897, 1700, 1616, 1519, 1345, 1107, 1041 cm⁻¹; ¹H

NMR (400 MHz, CDCl₃): δ 8.18 (d, J = 8.8 Hz, 2H), 7.72–7.66 (m, 3H), 7.14 (d, J = 16 Hz, 1H), 4.74 (s, 1H), 3.89–3.82 (m, 2H), 1.21 (d, J = 6 Hz, 6H), 1.11 (d, J = 6.4 Hz, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 193.0, 147.5, 140.1, 139.9, 128.0, 123.4, 123.0, 99.6, 69.5, 21.9, 21.3 ppm. HRMS (ESI) m/z : [M + Na]⁺ calcd for C₁₆H₂₁NO₃Na, 330.1317; found, 330.1316.

1,1-Dipropoxypropan-2-one (9a). The title compound was prepared via the general procedure from acetone **8a** (58 mg, 1.0 mmol) and 1-propanol **2c** (1 mL). The product was isolated by column chromatography using 1% ethyl acetate/hexane as an eluent; oil (172 mg, 67% yield); IR (KBr film): 2967, 2937, 2879, 1734, 1462, 1381, 1354, 1255, 1111, 1072 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 4.44 (s, 1H), 3.56–3.35 (m, 4H), 2.13 (s, 3H), 1.61–1.52 (m, 4H), 0.87 (t, J = 7.2 Hz, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 203.4, 102.0, 68.4, 23.5, 21.9, 9.5 ppm. HRMS (ESI) m/z : [M + Na]⁺ calcd for C₉H₁₈O₃Na, 197.1154; found, 197.1146.

1,1-Bis(hexyloxy)propan-2-one (9b). The title compound was prepared via the general procedure from acetone **8a** (58 mg, 1.0 mmol) and 1-hexanol **2e** (1 mL). The product was isolated by column chromatography using 1% ethyl acetate/hexane as an eluent; oil (180 mg, 70% yield); IR (KBr film): 2957, 2932, 2865, 1733, 1464, 1353, 1114, 1071 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 4.42 (s, 1H), 3.58–3.39 (m, 4H), 2.12 (s, 3H), 1.57–1.50 (m, 4H), 1.30–1.21 (m, 12H), 0.81 (t, J = 6.4 Hz, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 204.3, 103.0, 67.8, 31.5, 29.5, 25.6, 24.4, 22.5, 13.9 ppm. HRMS (ESI) m/z : [M + Na]⁺ calcd for C₁₅H₃₀O₃Na, 281.2093; found, 281.2084.

1,1-Dipropoxybutan-2-one (9c). The title compound was prepared via the general procedure from ethyl methyl ketone **8b** (72 mg, 1.0 mmol) and 1-propanol **2c** (1 mL). The product was isolated by column chromatography using 1% ethyl acetate/hexane as an eluent; oil (116 mg, 62% yield); IR (KBr film): 2968, 2938, 2878, 1730, 1601, 1461, 1381, 1258, 1103, 1074 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 4.48 (s, 1H), 3.55–3.36 (m, 4H), 2.54 (q, J = 7.6 Hz, 2H), 1.60–1.51 (m, 4H), 0.98 (t, J = 7.2 Hz, 3H), 0.87 (t, J = 7.6 Hz, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 207.0, 102.9, 69.4, 30.1, 22.8, 10.5, 6.9 ppm. HRMS (ESI) m/z : [M + Na]⁺ calcd for C₁₀H₂₀O₃Na, 211.1310; found, 211.1309.

1,1-Diisopropoxypentan-2-one (9d). The title compound was prepared via the general procedure from 2-pentanone **8c** (86 mg, 1.0 mmol) and iso-propanol **2i** (1 mL). The product was isolated by column chromatography using 1% ethyl acetate/hexane as an eluent; oil (137 mg, 68% yield); IR (KBr film): 2971, 2932, 2877, 1726, 1463, 1378, 1104, 1029 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 4.50 (s, 1H), 3.82–3.73 (m, 2H), 2.51 (t, J = 7.2 Hz, 2H), 1.57–1.48 (m, 2H), 1.16 (d, J = 6.4 Hz, 6H), 1.08 (d, J = 6 Hz, 6H), 0.85 (t, J = 7.2 Hz, 3H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 206.0, 99.7, 69.0, 36.6, 21.8, 21.2, 15.4, 12.7 ppm. HRMS (ESI) m/z : [M + Na]⁺ calcd for C₁₁H₂₂O₃Na, 225.1467; found, 225.1459.

1,1-Dibutoxy-4-methylpentan-2-one (9e). The title compound was prepared via the general procedure from isobutylmethyl ketone **8d** (100 mg, 1.0 mmol) and 1-butanol **2d** (1 mL). The product was isolated by column chromatography using 1% ethyl acetate/hexane as an eluent; oil (168 mg, 69% yield); IR (KBr film): 2960, 2935, 2873, 1729, 1465, 1073 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 4.41 (s, 1H), 3.58–3.37 (m, 4H), 2.38 (d, J = 6.8 Hz, 2H), 2.14–2.04 (m, 1H), 1.55–

1.48 (m, 4H), 1.36–1.27 (m, 4H), 0.87–0.83 (m, 12H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 204.8, 102.2, 66.5, 44.6, 30.7, 22.6, 21.5, 18.2, 12.8 ppm. HRMS (ESI) m/z : [M + Na]⁺ calcd for C₁₄H₂₈O₃Na, 267.1936; found, 267.1929.

1,1-Dibutoxy-5-methylhexan-2-one (9f). The title compound was prepared via the general procedure from 5-methyl-2-hexanone **8e** (114 mg, 1.0 mmol) and 1-butanol **2d** (1 mL). The product was isolated by column chromatography using 1% ethyl acetate/hexane as an eluent; oil (172 mg, 67% yield); IR (KBr film): 2960, 2935, 2873, 1729, 1467, 1074 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 4.45 (s, 1H), 3.59–3.37 (m, 4H), 2.50 (t, J = 7.6 Hz, 2H), 1.55–1.47 (m, 4H), 1.36–1.27 (m, 4H), 0.87–0.81 (m, 12H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 205.6, 102.1, 66.5, 44.6, 33.7, 30.6, 26.6, 21.3, 18.2, 12.8 ppm. HRMS (ESI) m/z : [M + Na]⁺ calcd for C₁₅H₃₀O₃Na, 281.2093; found, 281.2087.

1,1-Dibutoxy-3-methylbutan-2-one (9g). The title compound was prepared via the general procedure from 3-methyl-2-butanone **8f** (86 mg, 1.0 mmol) and 1-butanol **2d** (1 mL). The product was isolated by column chromatography using 1% ethyl acetate/hexane as an eluent; oil (149 mg, 65% yield); IR (KBr film): 2963, 2935, 2873, 1727, 1465, 1157, 1074 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 4.57 (s, 1H), 3.58–3.48 (m, 4H), 3.04–2.93 (m, 1H), 1.56–1.49 (m, 4H), 1.37–1.27 (m, 4H), 1.02 (d, J = 6.4 Hz, 6H), 0.85 (t, J = 7.2 Hz, 6H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 209.6, 102.3, 67.3, 35.3, 31.7, 19.2, 18.3, 13.7 ppm. HRMS (ESI) m/z : [M + Na]⁺ calcd for C₁₃H₂₆O₃Na, 253.1780; found, 253.1774.

2,2-Dibutoxy-1-cyclopropylethanone (9h). The title compound was prepared via the general procedure from cyclopropyl methyl ketone **8g** (84 mg, 1.0 mmol) and 1-butanol **2d** (1 mL). The product was isolated by column chromatography using 1% ethyl acetate/hexane as an eluent; oil (166 mg, 73% yield); IR (KBr film): 3009, 2960, 2935, 2873, 1713, 1462, 1385, 1161, 1070 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 4.61 (s, 1H), 3.59–3.44 (m, 4H), 2.32–2.26 (m, 1H), 1.58–1.51 (m, 4H), 1.38–1.29 (m, 4H), 1.02–0.98 (m, 2H), 0.91–0.84 (m, 8H) ppm; ¹³C{¹H}NMR (100 MHz, CDCl₃): δ 205.3, 101.6, 66.1, 30.7, 18.2, 15.2, 12.8, 10.8 ppm. HRMS (ESI) m/z : [M + Na]⁺ calcd for C₁₃H₂₄O₃Na, 251.1623; found, 251.1614.

■ ASSOCIATED CONTENT

📄 Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsomega.9b00361.

Copies of ¹H NMR and ¹³C NMR spectra (PDF)

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Notes

The authors declare no competing financial interest.

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