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Palladium-Catalyzed Cycloaromatization/Alkylation of *o*-(Alkynyl)styrenes

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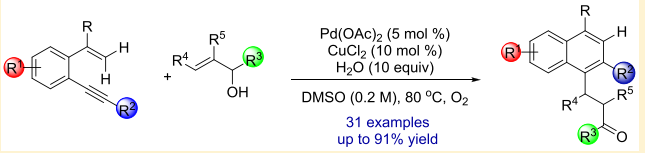
Supporting Information

ABSTRACT: A Pd(II)-catalyzed mild and highly regioselective 6-*endo* cyclization/alkylation reaction of *o*-(alkynyl)-styrenes with simple allylic alcohols has been developed. Under mild reaction conditions, the vinyl palladium species generated in situ after cyclization could insert a C−C double bond of allylic alcohol through a cross-coupling reaction and led to the formation of (alkyl)naphthalenes. This cascade cross-coupling reaction represents a direct and atom economic method for the construction of functionalized naphthalene derivatives in moderate to good yields.



INTRODUCTION

1,3-Dien-5-ynes as the versatile building blocks in organic synthesis have been widely applied for the synthesis of various carbo- and heterocycles.[1] Depending on the location and nature of the substituents, the 1,3-dien-5-ynes can undergo 1,6-cycloaromatization, 5-*exo*-dig or 5-*endo*-dig cyclization process to access the benzene, 1-methyleneindane, and indene derivatives in the presence of various metal catalysts such as Au,[2] Pt,[3] Ru,[4] Rh,[5] Pd,[6] In,[7] and W.[8] It is worth noting that a few examples on base[9] or acid[10] promoted, radical initiated[11] cyclization of 1,3-dien-5-ynes also have been elegantly established to prepare the functionalized carbocycles. Among them, the synthesis of functionalized naphthalene derivatives has attracted continuous attention due to their broad utilities in organic materials and wide distribution as core skeleton in many pharmaceutical compounds.[12] In 1998, Iwasawa and co-workers had developed a cycloaromatization reaction of *o*-(ethynyl)styrenes to form the naphthalenes using $W(CO)_5$·THF as a catalyst.[8a] After that, a ruthenium-catalyzed 6-*endo*-dig cyclization reaction of *o*-(ethynyl)styrenes was reported by Liu et al.[4b] Except for the above examples on the 1,3-dien-5-ynes possessing a terminal alkyne, different metal-catalyzed cycloaromatization of *o*-(alkynyl)styrenes having an internal triple bond was also developed to prepare the functionalized naphthalenes by Dankwardt,[13] Shibata,[14] Miura,[15] and Cheng,[16] et al. Recently, a few examples on base-promoted cycloaromatization,[9] NIS triggered iodocycloaromatization,[17] and radical mediated[11] process have been described for the synthesis of multisubstituted naphthalenes. Noticeably, the cycloaromatization process mentioned above is still limited by the protodemetalation side-reaction and the nature of initiators (Scheme 1a). To improve the synthetic efficiency of metal-catalyzed cycloaromatization to access the highly functionalized aromatic products, our group reported the palladium-catalyzed bisolefination[18] and bisalkylation[19] reactions of *o*-(alkynyl)styrenes (Scheme 1b and 1c). The vinylpalladium species generated in situ could be trapped by the electron-

Scheme 1. Cyclization Reactions of *o*-(Alkynyl)styrenes

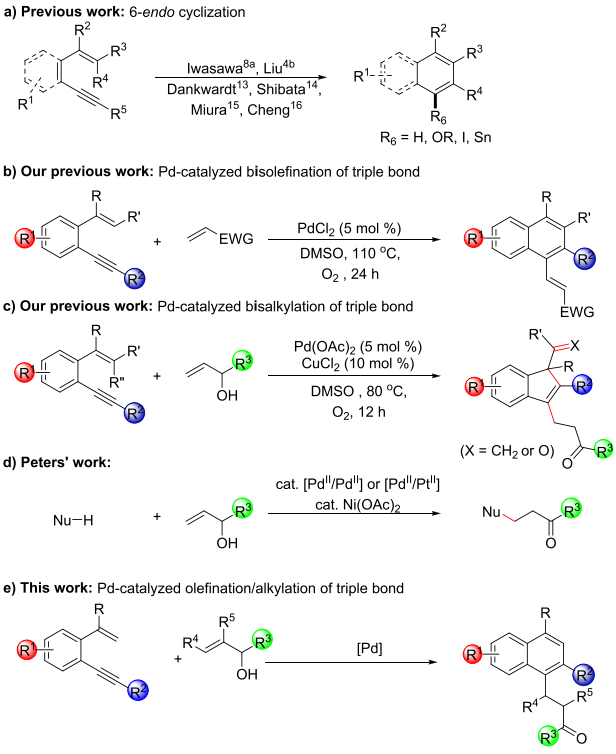


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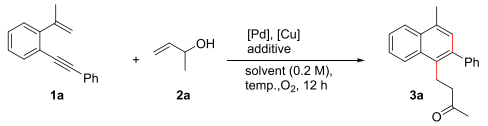
Table 1. Optimization of the Reaction Conditions[a]



entry	[Pd] (mol %)	[Cu] (mol %)	additive (equiv)	solvent	temp. (°C)	yield (%)[b]
1	$Pd(OAc)_2$	$CuCl_2$ (10)	–	DMSO	30	9
2	$Pd(OAc)_2$	$CuCl_2$ (10)	–	DMSO	80	70
3	$Pd(OAc)_2$	$CuCl_2$ (10)	–	DMSO	100	60
4	$PdCl_2$ (10)	$CuCl_2$ (10)	–	DMSO	80	56
5	$Pd(CH_3CN)_2Cl_2$ (10)	$CuCl_2$ (10)	–	DMSO	80	57
6	$Pd(OAc)_2$ (10)	$Cu(OAc)_2$ (10)	–	DMSO	80	trace
7	$Pd(OAc)_2$ (10)	$CuBr_2$ (10)	–	DMSO	80	59
8	$Pd(OAc)_2$ (10)	$CuSO_4$ (10)	–	DMSO	80	59
9	$Pd(OAc)_2$ (10)	$CuCl_2$ (10)	–	toluene	80	trace
10	$Pd(OAc)_2$ (10)	$CuCl_2$ (10)	–	dioxane	80	trace
11	$Pd(OAc)_2$ (10)	$CuCl_2$ (10)	–	DMF	80	53
12	$Pd(OAc)_2$ (10)	$CuCl_2$ (10)	CF_3COOH (1.0)	DMSO	80	65
13	$Pd(OAc)_2$ (10)	$CuCl_2$ (10)	Et_3N (1.0)	DMSO	80	7
14	$Pd(OAc)_2$ (10)	$CuCl_2$ (10)	H_2O (10)	DMSO	80	76
15	**$Pd(OAc)_2$ (5)**	**$CuCl_2$ (10)**	**H_2O (10)**	**DMSO**	**80**	73[c]
16	–	$CuCl_2$ (10)	H_2O (10)	DMSO	80	N.R.
17	$Pd(OAc)_2$ (10)	–	H_2O (10)	DMSO	80	7
18[d]	$Pd(OAc)_2$ (10)	$CuCl_2$ (10)	H_2O (10)	DMSO	80	63

[a]Unless noted otherwise, reactions were carried out on a 0.2 mmol scale of **1a** with 3 equiv of **2a** in 1.0 mL of solvent under oxygen atmosphere. [b]NMR yield with dibromomethane as internal standard. [c]The yields of isolated product. [d]Air atmosphere.

deficient alkenes as well as the environment-friendly allylic alcohols[20] to form the (alkenyl)naphthalenes, indenes, or β-arylketone products (Scheme 1c, 1d). Another work on platinum-catalyzed cycloaromatization and [3 + 2] cycloaddition of *o*-(alkynyl)styrenes with silyl enol ethers was successfully developed by Iwasawa and co-workers to make the five-membered-ring-fused naphthalenes.[21] Comparing with the extensive studies on the cycloaromatization of *o*-(alkynyl)-biaryls and *o*-(alkynyl)heterobiaryls for construction of fused ring compounds,[22] so far only rare examples have been explored to prepare the highly functionalized naphthalenes via transition-metal-catalyzed cycloaromatization of *o*-(alkynyl)-styrenes.[18,21] Herein, we would like to describe a palladium-catalyzed tandem cycloaromatization and alkylation reactions of *o*-(alkynyl)styrenes for the synthesis of functionalized naphthalenes (Scheme 1e).

RESULTS AND DISCUSSION

We began our study by investigating the *o*-(alkynyl)styrene **1a** to couple with 3-buten-2-ol **2a** (Table 1). As illustrated in the previous work, the substituent locations at alkenyl double bonds are crucial for the regioselective cyclization of 1,3-dien-5-ynes.[23] It was found that the desired product **3a** could be formed in 9% yield and along with mainly recovering the starting material with using $Pd(OAc)_2$ as catalyst, $CuCl_2$ as oxidant in DMSO at 30 °C under oxygen atmosphere (Table 1, entry 1). To improve the conversion of this reaction, the temperature was elevated to 80 °C, and the yield of the desired product was then increased up to 70% (Table 1, entry 2). With this promising result, different catalysts, oxidants, solvents, and additives were further screened in sequence. It was found that the combination of $Pd(OAc)_2$ with $CuCl_2$ more favored the formation of product in a high yield in DMSO solution (Table 1, entries 4−11). Next, to further improve the product yield, different additives were examined. The product **3a** was obtained in 76% when 10 equiv of H_2O were used as additive (Table 1, entries 12−14). Decreasing the $Pd(OAc)_2$ catalyst loading to 5 mol %, the desired product **3a** still could be obtained in 73% isolated yield (Table 1, entry 15). Finally, the control experiments show that palladium catalyst and copper oxidant are necessary to afford the desired product **3a** in a good yield (Table 1, entries 16−18).

With the optimized reaction conditions at hand, the substrate scope of *o*-(alkynyl)styrenes was tested (Table 2). It was noticed that all the substrates with an electron-donating or electron-withdrawing group on the phenyl ring (R^4) could be smoothly transformed to the corresponding desired products in good to high yields (**3b−3h**). Comparing with electron-donating group, it was observed that the electron-withdrawing group at *ortho*-position on the phenyl ring (R^4) was less effective to furnish the desired product (**3i** and **3j**). In addition, the substrate having an alkyl substituent on the triple bond also furnished the desired products in moderate to good yields under the standard reaction conditions (**3k−3m**). The vinyl substituted substrate also worked for this transformation (**3n**), albeit affording the desired product in a low yield. On the other hand, changing the substituent R^1 on the phenyl ring from electron-donating to electron-withdrawing groups both could afford the desired product (**3o−3r**). Next, when the R^2 substituent was replaced by the ethyl or phenyl group, the desired products **3s** and **3t** were obtained in high yields, respectively, while when changing the R^2 and R^3 to methyl group, the desired product **3u** was formed only in 27% isolated yield along with formation of another 5-*endo*-*dig* cyclization/alkylation product in 69% yield. This result is consistent with our previous observation.[19] Finally, the product **3v** was isolated in a very low yield accompanying with cyclization/protonation product as the major product (70% yield). This possibly was a

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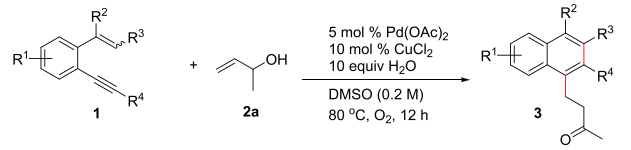
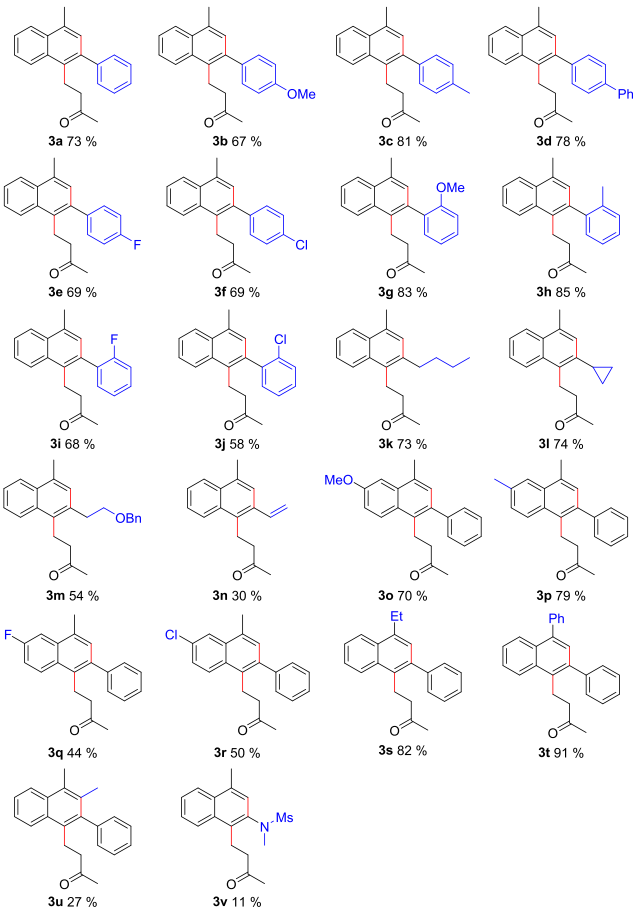
Table 2. Substrate Scope of 1,3-Dien-5-yne[a,b]



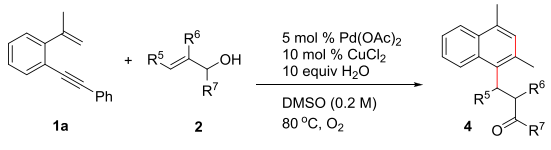


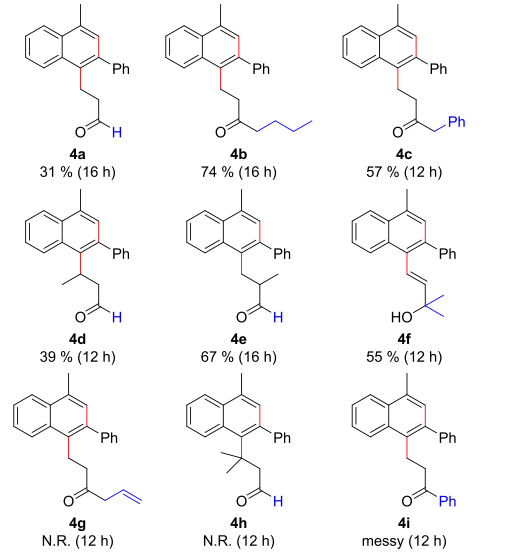
[a]Reaction conditions: The reactions were carried out under the standard conditions: **1** (0.3 mmol), **2a** (0.9 mmol, 3 equiv), $Pd(OAc)_2$ (0.015 mmol, 0.05 equiv), $CuCl_2$ (0.03 mmol, 0.1 equiv), and H_2O (3 mmol, 10 equiv) in 1.5 mL DMSO were stirred at 80 °C for 12 h under oxygen atmosphere. [b]The yields of isolated product.

result of the easy protodemetalation of electron-rich carbopalladium intermediate generated in situ.

Following, we probed the generality of different allylic alcohols as the coupling partners. The results are summarized in Table 3. When the prop-2-en-1-ol was subjected to this reaction, a naphthalenyl propanal product **4a** was isolated only in 31% yield. A similar result was observed when the (*E*)-but-2-en-1-ol was used as the coupling partner (**4d**). Installing a methyl group at the 2-position of allylic alcohol will significantly increase the yield of product (**4e**). Unfortunately, no desired product was detected with using the 3-methylbut-2-en-1-ol as coupling partner possibly due to its steric hindrance (**4h**). Interestingly, the 1,1-dimethyl substituted allylic alcohol could afford the (alkenyl)naphthalene product **4f** in moderate yield. When the side chain of the allylic alcohol was prolonged, the corresponding desired products could be obtained in moderate to good yields (**4b**, **4c**). Unfortunately, the hexa-1,5-

Table 3. Substrate Scope of Different Allylic Alcohols[a,b]



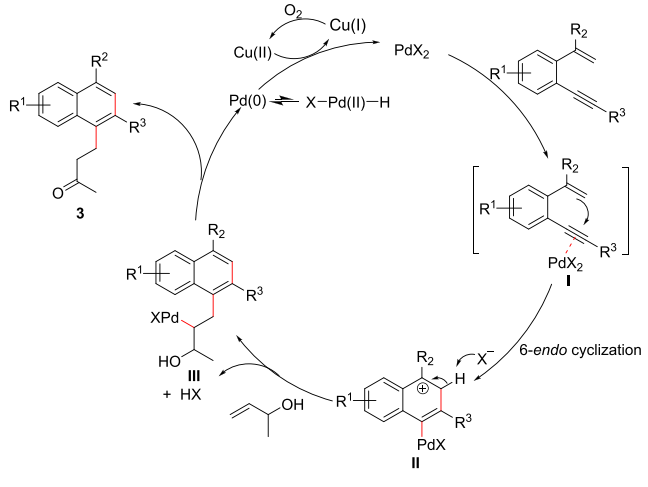


[a]Reaction conditions: The reactions were carried out under the standard conditions: **1a** (0.3 mmol), **2** (0.9 mmol, 3 equiv), palladium acetate (0.015 mmol, 0.05 equiv), copper chloride (0.06 mmol, 0.2 equiv), and H_2O (3 mmol, 10 equiv) in 1.5 mL DMSO were stirred at 80 °C under oxygen atmosphere for corresponding hours shown in parentheses. [b]The yields of isolated product.

dien-3-ol and 1-phenylprop-2-en-1-ol failed to give the desired product.

On the basis of the above results and previous reports,[24] we proposed a possible mechanistic pathway of current reaction as shown in Scheme 2. First, Pd(II) coordinates with the internal alkyne to form a π-complex **I**. Then the alkenyl double bond as initiator triggers the intramolecular carbopalladation to afford an intermediate **II**. Following, an external olefin insertion process affords the carbopalladium species **III**. After elimination,[25] the desired product **3** was produced with

Scheme 2. Proposed Mechanism Pathway



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releasing the Pd(0), which could be activated again by using $CuCl_2$ and molecular oxygen as oxidants.

CONCLUSION

In conclusion, a palladium-catalyzed tandem cycloaromatization/alkylation reaction of *o*-(ethynyl)styrenes has been developed. Under mild reaction conditions, the multifunctionalized naphthalenes were obtained in moderate to high yields with using the abundant and environmentally friendly allylic alcohols as alkylation reagent. This tandem reaction provides a simple, cost-effective route for the regioselective synthesis of alkyl naphthalenes.

EXPERIMENTAL SECTION

General Information. $PdCl_2$, $CuCl_2$, and the allylic alcohols were purchased from commercial suppliers and used as received unless otherwise noted. All reactions were performed under oxygen environment unless otherwise specified. All commercial solvents and reagents were employed without further purification. Reactions were monitored through analytical thin layer chromatography (SiO_2 60 F-254 plates). The spots visualization was performed under UV radiation (254 nm), further visualization was possible using basic solution of potassium permanganate. Flash chromatography was carried out using 200−300 mesh silica gel (SiO_2 60) with distilled solvents. Proton nuclear magnetic resonance (^{1}H NMR) and carbon nuclear magnetic resonance ($^{13}C\{^1H\}$ NMR) spectra were recorded on Bruker Advance 400 M NMR spectrometers. Chloroform-*d* was used as the solvent and $SiMe_4$ (TMS) as internal standard. Chemical shifts for ^{1}H NMR spectra are reported as δ in units of parts per million (ppm) downfield from TMS (δ 0.00 ppm) and relative to the signal of chloroform-*d* (δ 7.260 ppm, singlet). Multiplicities are recorded as *s* (singlet); *d* (doublet); *t* (triplet); *q* (quartet); *dd* (doublets of doublet); *m* (multiplets). Coupling constants are expressed as a *J* value in Hz. $^{13}C\{^1H\}$ NMR are reported as δ in units of parts per million (ppm) downfield from TMS (δ 0.00 ppm) and relative to the signal of chloroform-*d* (δ 77.03 ppm, triplet). HRMS spectra were recorded on XEVO-G2 Q-TOF (Waters Corporation). The starting materials of **1a−1v** were prepared according to the reported procedures, and the NMR spectroscopic data of these compounds are identified with the literatures.[26]

Procedure for the Synthesis of Products (3a−3v), (4a−4f). A 35 mL sealed tube equipped with a stirring bar was charged with $Pd(OAc)_2$ (5 mol %, 3.4 mg) and $CuCl_2$ (10 mol %, 4.0 mg), and a septum cap was affixed. The sealed tube was evacuated and refilled with oxygen two times, and then a needle connected to an oxygen balloon was inserted through the septum cap. DMSO (1.5 mL) was added into the sealed tube by a syringe. To the resulting mixture, substrate **1** (0.3 mmol), **2** (0.9 mmol), and water (3.0 mmol, 54 μL) were added. The reaction mixture was allowed to stir at 80 °C (oil bath) for 12 h. Then the mixture was cooled to room temperature, diluted with diethyl ether (5 mL), and filtered through a Celite pad. The filtrate was washed with water three times (3 × 30 mL), dried over Na_2SO_4, and concentrated in vacuo. The residue was purified by thin layer chromatography on silica gel (300−400 mesh) using (petroleum ether/ethyl acetate = 20/1) as eluting solvent to afford the desired product.

Procedure for the Synthesis of Product 3a on a 5 mmol Scale. A 100 mL Schlenk tube equipped with a stirring bar was charged with $Pd(OAc)_2$ (5 mol %, 56.1 mg) and $CuCl_2$ (10 mol %, 67.2 mg), and a septum cap was affixed. The sealed tube was evacuated and refilled with oxygen two times, and then a needle connected to an oxygen balloon was inserted through the septum cap. DMSO (25.0 mL) was added into the sealed tube by a syringe. To the resulting mixture, substrate **1a** (5.0 mmol), **2** (15.0 mmol), and water (50.0 mmol, 900.0 μL) were added. The reaction mixture was allowed to stir at 80 °C (oil bath) for 15 h. Then the mixture was cooled to room temperature, diluted with diethyl ether (80 mL), and filtered through a Celite pad. The filtrate was washed with water three times (3 × 100 mL), dried over Na_2SO_4, and concentrated in vacuo. The residue was purified via column chromatography on silica gel (eluents: petroleum ether/ethyl acetate = 50:1−20:1) to afford the desired product **3a** (1056.8 mg, 3.664 mmol, 73%) as yellow solid.

*4-(4-Methyl-2-phenylnaphthalen-1-yl)butan-2-one (**3a**).* Yellow solid; 63.2 mg, 0.219 mmol, yield 73%; mp 87−89 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (t, *J* = 8.3 Hz, 2H), 7.60−7.50 (m, 2H), 7.46−7.29 (m, 5H), 7.20 (s, 1H), 3.26 (t, *J* = 8.1 Hz, 2H), 2.70−2.63 (m, 5H), 2.02 (s, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 208.0, 142.6, 139.3, 132.5, 132.4, 132.1, 131.8, 129.2, 129.2, 128.3, 127.1, 126.4, 125.5, 125.1, 124.6, 45.1, 29.8, 23.2, 19.4; HRMS (ESI, *m*/*z*) Calcd for $C_{21}H_{20}NaO$ $[M + Na]^+$: 311.1412, found 311.1407.

*4-(2-(4-Methoxyphenyl)-4-methylnaphthalen-1-yl)butan-2-one (**3b**).* Yellow solid; 64.0 mg, 0.201 mmol, yield 67%; mp 115−117 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (td, *J* = 7.5, 1.8 Hz, 2H), 7.61−7.53 (m, 2H), 7.31−7.20 (m, 3H), 7.00 (d, *J* = 8.5 Hz, 2H), 3.89 (s, 3H), 3.31 (t, *J* = 8.2 Hz, 2H), 2.73−2.67 (m, 5H), 2.07 (s, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 208.1, 158.7, 138.9, 134.9, 132.4, 132.4, 132.2, 131.8, 130.3, 129.6, 126.3, 125.4, 125.0, 124.6, 113.8, 55.4, 45.1, 29.8, 23.2, 19.4. HRMS (ESI, *m*/*z*) Calcd for $C_{22}H_{22}NaO_2$ $[M + Na]^+$: 341.1517, found 341.1510.

*4-(4-Methyl-2-(p-tolyl)naphthalen-1-yl)butan-2-one (**3c**).* Yellow solid; 73.5 mg, 0.243 mmol, yield 81%; mp 89−91 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07−8.01 (m, 2H), 7.55 (tt, *J* = 6.9, 5.1 Hz, 2H), 7.25−7.19 (m, 5H), 3.28 (t, *J* = 8.2 Hz, 2H), 2.71−2.65 (m, 5H), 2.43 (s, 3H), 2.05 (s, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ208.1, 139.6, 139.3, 136.7, 132.5, 132.4, 132.1, 131.8, 129.5, 129.1, 129.0, 126.3, 125.4, 125.1, 124.6, 45.2, 29.8, 23.2, 21.3, 19.5; HRMS (ESI, *m*/*z*) Calcd for $C_{22}H_{22}NaO$ $[M + Na]^+$: 325.1568, found 325.1567.

*4-(2-([1,1′-Biphenyl]-4-yl)-4-methylnaphthalen-1-yl)butan-2-one (**3d**).* Yellow solid; 85.3 mg, 0.234 mmol, yield 78%; mp 115−117 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (t, *J* = 9.3 Hz, 2H), 7.67 (d, *J* = 7.8 Hz, 4H), 7.61−7.53 (m, 2H), 7.48 (t, *J* = 7.5 Hz, 2H), 7.39 (t, *J* = 7.3 Hz, 3H), 7.25 (s, 1H), 3.32 (t, *J* = 8.1 Hz, 2H), 2.74−2.68 (m, 5H), 2.06 (s, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 208.1, 141.6, 140.9, 140.0, 138.9, 132.6, 132.2, 131.8, 129.7, 129.3, 129.0, 127.5, 127.2, 127.1, 126.4, 125.6, 125.1, 124.7, 45.2, 29.9, 23.3, 19.5; HRMS (ESI, *m*/*z*) Calcd for $C_{27}H_{24}NaO$ $[M + Na]^+$: 387.1725, found 387.1725.

*4-(2-(4-Fluorophenyl)-4-methylnaphthalen-1-yl)butan-2-one (**3e**).* Yellow solid; 63.4 mg, 0.207 mmol, yield 69%; mp 92−94 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09−8.00 (m, 2H), 7.62−7.53 (m, 2H), 7.31−7.25 (m, 2H), 7.14 (dd, *J* = 16.0, 7.6 Hz, 3H), 3.25 (t, *J* = 8.1 Hz, 2H), 2.71−2.63 (m, 5H), 2.06 (s, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 207.9, 162.1 (d, *J* = 245.9 Hz), 138.5 (d, *J* = 3.4 Hz), 138.2, 132.6, 132.6, 132.3, 131.8, 130.8 (d, *J* = 7.9 Hz), 129.2, 126.5, 125.7, 125.1, 124.6, 115.3 (d, *J* = 21.3 Hz), 45.0, 29.9, 23.2, 19.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ −115.62 (ddd, *J* = 14.3, 8.9, 5.4 Hz); HRMS (ESI, *m*/*z*) Calcd for $C_{21}H_{19}FNaO$ $[M + Na]^+$: 329.1318, found 329.1315.

*4-(2-(4-Chlorophenyl)-4-methylnaphthalen-1-yl)butan-2-one (**3f**).* Yellow solid; 66.8 mg, 0.207 mmol, yield 69%; mp 110−112 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08−8.00 (m, 2H), 7.61−7.53 (m, 2H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.26 (d, *J* = 8.1 Hz, 2H), 7.15 (s, 1H), 3.25 (t, *J* = 8.2 Hz, 2H), 2.70−2.63 (m, 5H), 2.06 (s, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 207.8, 141.0, 138.0, 133.2, 132.8, 132.6, 132.2, 131.8, 130.6, 129.0, 128.6, 126.5, 125.8, 125.1, 124.6, 45.0, 29.9, 23.1, 19.5; HRMS (ESI, *m*/*z*) Calcd for $C_{21}H_{19}ClNaO$ $[M + Na]^+$: 345.1022, found 345.1018.

*4-(2-(2-Methoxyphenyl)-4-methylnaphthalen-1-yl)butan-2-one (**3g**).* Yellow solid; 79.3 mg, 0.249 mmol, yield 83%; mp 34−35 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (dd, *J* = 9.7, 6.9 Hz, 2H), 7.57 (qd, *J* = 6.8, 3.5 Hz, 2H), 7.40 (td, *J* = 7.9, 1.7 Hz, 1H), 7.19 (q, *J* = 2.3, 1.6 Hz, 2H), 7.06 (t, *J* = 7.5 Hz, 1H), 7.02 (d, *J* = 8.3 Hz, 1H), 3.77 (s, 3H), 3.26 (ddd, *J* = 14.3, 11.1, 5.2 Hz, 1H), 3.10 (ddd, *J* = 14.1, 10.9, 5.8 Hz, 1H), 2.80−2.60 (m, 5H), 2.04 (s, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 208.5, 156.6, 135.6, 133.2, 132.6, 132.3, 131.7, 131.1, 131.1, 129.5, 128.9, 126.1, 125.3, 125.1, 124.6, 120.6,

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110.9, 55.5, 44.6, 29.7, 23.7, 19.5; HRMS (ESI, *m*/*z*) Calcd for $C_{22}H_{22}NaO_2$ [M + Na]$^+$: 341.1517, found 341.1513.

4-(4-Methyl-2-(o-tolyl)naphthalen-1-yl)butan-2-one (**3h**). Yellow solid; 77.1 mg, 0.255 mmol, yield 85%; mp 34−35 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13−8.06 (m, 2H), 7.61 (qd, *J* = 7.2, 3.5 Hz, 2H), 7.36−7.27 (m, 3H), 7.20 (d, *J* = 7.3 Hz, 1H), 7.15 (s, 1H), 3.36 (ddd, *J* = 15.2, 11.0, 5.1 Hz, 1H), 2.93 (ddd, *J* = 14.0, 10.7, 6.0 Hz, 1H), 2.78−2.56 (m, 5H), 2.12 (s, 3H), 2.04 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.1, 141.7, 138.6, 135.8, 132.6, 132.5, 132.2, 131.8, 130.2, 129.4, 128.7, 127.5, 126.2, 125.7, 125.4, 125.1, 124.5, 44.6, 29.7, 23.3, 20.3, 19.5; HRMS (ESI, *m*/*z*) Calcd for $C_{22}H_{22}NaO$ [M + Na]$^+$: 325.1568, found 325.1569.

4-(2-(2-Fluorophenyl)-4-methylnaphthalen-1-yl)butan-2-one (**3i**). Yellow solid; 62.5 mg, 0.204 mmol, yield 68%; mp 78−80 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10−8.02 (m, 2H), 7.61−7.55 (m, 2H), 7.42−7.34 (m, 1H), 7.30−7.14 (m, 4H), 3.21 (t, J = 8.2 Hz, 2H), 2.80−2.62 (m, 5H), 2.05 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.1, 159.8 (d, *J* = 244.6 Hz), 133.5, 132.9, 132.7, 132.6, 131.8, 131.7, 129.6 (d, *J* = 17.3 Hz), 129.4 (d, *J* = 8.0 Hz), 129.0, 126.4, 125.8, 125.1, 124.7, 124.2 (d, *J* = 3.7 Hz), 115.8 (d, *J* = 22.4 Hz), 44.5 (d, *J* = 1.5 Hz), 29.8, 23.7, 19.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ −114.34 (d, *J* = 4.3 Hz); HRMS (ESI, *m*/*z*) Calcd for $C_{21}H_{19}FNaO$ [M + Na]$^+$: 329.1318, found 329.1323.

4-(2-(2-Chlorophenyl)-4-methylnaphthalen-1-yl)butan-2-one (**3j**). Yellow solid; 56.2 mg, 0.174 mmol, yield 58%; mp 34−35 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11−8.02 (m, 2H), 7.61−7.56 (m, 2H), 7.54−7.49 (m, 1H), 7.34 (dt, *J* = 7.7, 3.9 Hz, 2H), 7.30−7.25 (m, 1H), 7.12 (s, 1H), 3.25−3.15 (m, 1H), 3.14−3.04 (m, 1H), 2.79−2.57 (m, 5H), 2.03 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.1, 140.9, 136.4, 133.5, 132.9, 132.8, 132.7, 131.7, 131.3, 129.7, 129.0, 128.5, 126.8, 126.4, 125.8, 125.2, 124.6, 44.4, 29.8, 23.5, 19.5; HRMS (ESI, *m*/*z*) Calcd for $C_{21}H_{19}ClNaO$ [M + Na]$^+$: 345.1022, found 345.1011.

4-(2-Butyl-4-methylnaphthalen-1-yl)butan-2-one (**3k**). Yellow solid; 58.8 mg, 0.219 mmol, yield 73%; mp 36−38 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00−7.94 (m, 2H), 7.54−7.44 (m, 2H), 7.16 (s, 1H), 3.35 (t, *J* = 8.2 Hz, 2H), 2.80−2.71 (m, 4H), 2.66 (s, 3H), 2.20 (s, 3H), 1.67−1.57 (m, 2H), 1.45 (h, *J* = 7.4 Hz, 2H), 0.97 (t, *J* = 7.3 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.4, 137.8, 132.6, 132.2, 131.8, 131.5, 129.4, 125.9, 125.0, 124.7, 124.0, 44.9, 34.2, 33.6, 30.1, 23.1, 22.0, 19.5, 14.2; HRMS (ESI, *m*/*z*) Calcd for $C_{19}H_{24}NaO$ [M + Na]$^+$: 291.1725, found 291.1727.

4-(2-Cyclopropyl-4-methylnaphthalen-1-yl)butan-2-one (**3l**). Yellow solid; 56.0 mg, 0.222 mmol, yield 74%; mp 40−41 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03−7.95 (m, 2H), 7.51 (dtd, *J* = 15.9, 7.4, 6.0 Hz, 2H), 7.00 (s, 1H), 3.58 (d, *J* = 8.2 Hz, 2H), 2.82 (d, *J* = 8.2 Hz, 2H), 2.66 (s, 3H), 2.22 (s, 3H), 2.11 (td, *J* = 8.6, 4.3 Hz, 1H), 1.09−1.00 (m, 2H), 0.78 (h, *J* = 4.5 Hz, 2H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.6, 137.3, 133.7, 132.7, 131.9, 131.6, 126.0, 125.4, 124.9, 124.8, 123.8, 44.3, 30.1, 22.2, 19.6, 13.6, 7.6; HRMS (ESI, *m*/*z*) Calcd for $C_{18}H_{20}NaO$ [M + Na]$^+$: 275.1412, found 275.1411.

4-(2-(2-(Benzyloxy)ethyl)-4-methylnaphthalen-1-yl)butan-2-one (**3m**). Yellow solid; 56.1 mg, 0.162 mmol, yield 54%; mp 48−49 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.6 Hz, 1H), 7.95 (d, *J* = 7.8 Hz, 1H), 7.53−7.46 (m, 2H), 7.35−7.27 (m, 5H), 7.18 (s, 1H), 4.53 (s, 2H), 3.72 (t, *J* = 7.3 Hz, 2H), 3.34 (t, *J* = 8.2 Hz, 2H), 3.08 (t, *J* = 7.3 Hz, 2H), 2.74 (t, *J* = 8.1 Hz, 2H), 2.65 (s, 3H), 2.11 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.3, 138.4, 133.6, 132.7, 132.1, 132.1, 129.5, 128.5, 127.8, 127.7, 126.0, 125.0, 124.1, 73.2, 71.2, 44.7, 34.2, 30.1, 22.0, 19.5; HRMS (ESI, *m*/*z*) Calcd for $C_{24}H_{26}NaO_2$ [M + Na]$^+$: 369.1830, found 369.1828.

4-(4-Methyl-2-vinylnaphthalen-1-yl)butan-2-one (**3n**). Yellow solid; 21.4 mg, 0.09 mmol, yield 30%; mp 40−41 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (t, *J* = 9.1 Hz, 2H), 7.56−7.47 (m, 3H), 7.19 (dd, *J* = 17.3, 11.1 Hz, 1H), 5.77 (d, *J* = 17.3 Hz, 1H), 5.42 (d, *J* = 11.0 Hz, 1H), 3.42 (t, *J* = 8.1 Hz, 2H), 2.75 (t, *J* = 8.1 Hz, 2H), 2.68 (s, 3H), 2.18 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.2, 134.8, 133.0, 132.9, 132.9, 132.2, 132.0, 126.3, 125.6, 125.0, 124.8, 124.4, 116.6, 44.4, 30.1, 21.8, 19.7; HRMS (ESI, *m*/*z*) Calcd for $C_{17}H_{18}NaO$ [M + Na]$^+$: 261.1255, found 261.1259.

4-(6-Methoxy-4-methyl-2-phenylnaphthalen-1-yl)butan-2-one (**3o**). Yellow solid; 66.9 mg, 0.210 mmol, yield 70%; mp 140−142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 9.2 Hz, 1H), 7.43 (t, *J* = 7.4 Hz, 2H), 7.39−7.28 (m, 4H), 7.23 (d, *J* = 9.1 Hz, 1H), 7.18 (s, 1H), 3.97 (s, 3H), 3.23 (t, *J* = 8.0 Hz, 2H), 2.68−2.61 (m, 5H), 2.02 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.1, 157.4, 142.6, 137.2, 133.8, 132.1, 131.2, 129.9, 129.4, 128.3, 127.0, 126.3, 118.3, 103.9, 55.5, 45.3, 29.8, 23.3, 19.7; HRMS (ESI, *m*/*z*) Calcd for $C_{22}H_{22}NaO_2$ [M + Na]$^+$: 341.1517, found 341.1514.

4-(4,6-Dimethyl-2-phenylnaphthalen-1-yl)butan-2-one (**3p**). Yellow solid; 71.7 mg, 0.237 mmol, yield 79%; mp 127−128 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.6 Hz, 1H), 7.83 (s, 1H), 7.47−7.31 (m, 6H), 7.18 (s, 1H), 3.26 (t, *J* = 8.2 Hz, 2H), 2.70−2.64 (m, 5H), 2.58 (s, 3H), 2.03 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.1, 142.6, 138.4, 135.1, 132.7, 131.9, 131.8, 129.9, 129.3, 129.3, 128.5, 128.3, 127.0, 124.5, 124.2, 45.2, 29.8, 23.2, 21.9, 19.5; HRMS (ESI, *m*/*z*) Calcd for $C_{22}H_{22}NaO$ [M + Na]$^+$: 325.1568, found 325.1559.

4-(6-Fluoro-4-methyl-2-phenylnaphthalen-1-yl)butan-2-one (**3q**). Yellow solid; 40.4 mg, 0.132 mmol, yield 44%; mp 113−115 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 9.3, 5.8 Hz, 1H), 7.65 (dd, *J* = 10.9, 2.6 Hz, 1H), 7.48−7.30 (m, 6H), 7.24 (s, 1H), 3.26 (t, *J* = 8.0 Hz, 2H), 2.69−2.60 (m, 5H), 2.05 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 207.8, 160.5 (d, *J* = 246.0 Hz), 142.2, 138.6 (d, *J* = 2.5 Hz), 133.8 (d, *J* = 8.1 Hz), 132.3, 131.9 (d, *J* = 5.2 Hz), 130.3, 129.2, 128.8, 128.4, 127.2 (d, *J* = 8.8 Hz), 127.2, 116.2 (d, *J* = 24.6 Hz), 108.7 (d, *J* = 20.6 Hz), 45.1, 29.8, 23.3, 19.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ −114.86. HRMS (ESI, *m*/*z*) Calcd for $C_{21}H_{19}FNaO$ [M + Na]$^+$: 329.1318, found 329.1311.

4-(6-Chloro-4-methyl-2-phenylnaphthalen-1-yl)butan-2-one (**3r**). Yellow solid; 48.4 mg, 0.150 mmol, yield 50%; mp 150−151 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 2.1 Hz, 1H), 7.96 (d, *J* = 9.0 Hz, 1H), 7.50 (dd, *J* = 9.1, 2.1 Hz, 1H), 7.45 (t, *J* = 7.3 Hz, 2H), 7.39 (d, *J* = 7.0 Hz, 1H), 7.31 (d, *J* = 7.8 Hz, 2H), 7.23 (s, 1H), 3.24 (t, *J* = 8.1 Hz, 2H), 2.66−2.61 (m, 5H), 2.03 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 207.8, 142.1, 139.6, 133.5, 132.3, 131.8, 131.6, 130.4, 130.2, 129.2, 128.4, 127.3, 127.0, 126.4, 124.2, 45.0, 29.8, 23.2, 19.4; HRMS (ESI, *m*/*z*) Calcd for $C_{21}H_{19}ClNaO$ [M + Na]$^+$: 345.1022, found 345.1010.

4-(4-Ethyl-2-phenylnaphthalen-1-yl)butan-2-one (**3s**). Yellow solid; 74.4 mg, 0.246 mmol, yield 82%; mp 91−92 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16−8.12 (m, 1H), 8.08−8.04 (m, 1H), 7.60−7.53 (m, 2H), 7.46 (t, *J* = 7.3 Hz, 2H), 7.40 (d, *J* = 6.5 Hz, 1H), 7.35 (d, *J* = 7.5 Hz, 2H), 7.24 (s, 1H), 3.29 (t, *J* = 8.2 Hz, 2H), 3.13 (q, *J* = 7.5 Hz, 2H), 2.70 (t, *J* = 8.2 Hz, 2H), 2.05 (s, 3H), 1.40 (t, *J* = 7.5 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.1, 142.7, 139.4, 138.4, 132.0, 131.7, 129.3, 128.4, 127.5, 127.1, 126.3, 125.5, 124.8, 124.7, 45.1, 29.8, 25.9, 23.3, 15.1; HRMS (ESI, *m*/*z*) Calcd for $C_{22}H_{22}NaO$ [M + Na]$^+$: 325.1568, found 325.1567.

4-(2,4-Diphenylnaphthalen-1-yl)butan-2-one (**3t**). Yellow solid; 95.7 mg, 0.273 mmol, yield 91%; mp 67−69 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 8.6 Hz, 1H), 8.02 (d, *J* = 8.5 Hz, 1H), 7.60 (t, *J* = 7.7 Hz, 1H), 7.56−7.38 (m, 11H), 7.36 (s, 1H), 3.39 (t, *J* = 8.1 Hz, 2H), 2.78 (t, *J* = 8.1 Hz, 2H), 2.09 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 207.9, 142.3, 140.6, 139.2, 138.6, 133.5, 132.0, 131.5, 130.3, 129.5, 129.3, 128.4, 128.4, 127.4, 127.2, 127.1, 126.6, 125.7, 124.4, 45.0, 29.8, 23.4; HRMS (ESI, *m*/*z*) Calcd for $C_{26}H_{22}NaO$ [M + Na]$^+$: 373.1568, found 373.1565.

4-(3,4-Dimethyl-2-phenylnaphthalen-1-yl)butan-2-one (**3u**). Yellow solid; 24.5 mg, 0.081 mmol, yield 27%; mp 84−86 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.2 Hz, 1H), 7.99 (d, *J* = 8.1 Hz, 1H), 7.57−7.44 (m, 4H), 7.42−7.37 (m, 1H), 7.19 (d, *J* = 8.0 Hz, 2H), 3.06 (t, *J* = 8.1 Hz, 2H), 2.67 (s, 3H), 2.61 (t, *J* = 8.1 Hz, 2H), 2.11 (s, 3H), 2.00 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.2, 142.2, 140.7, 132.6, 132.4, 132.2, 130.2, 123.0, 129.2, 128.6, 127.0, 125.6, 125.4, 124.8, 124.4, 45.0, 29.7, 24.3, 18.9, 15.4; HRMS (ESI, *m*/*z*) Calcd for $C_{22}H_{22}NaO$ [M + Na]$^+$: 325.1568, found 325.1572.

N-Methyl-N-(4-methyl-1-(3-oxobutyl)naphthalen-2-yl)-methanesulfonamide (**3v**). Yellow solid; 10.5 mg, 0.033 mmol, yield

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11%; mp 129−130 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (dt, J = 8.0, 4.2 Hz, 2H), 7.57 (dd, J = 6.7, 3.2 Hz, 2H), 7.17 (s, 1H), 3.56−3.37 (m, 2H), 3.28 (s, 3H), 3.13−2.97 (m, 4H), 2.78 (ddd, J = 17.6, 10.9, 5.8 Hz, 1H), 2.68 (s, 3H), 2.20 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 208.5, 136.2, 136.0, 135.1, 132.8, 132.6, 126.8, 126.6, 125.4, 125.1, 124.7, 44.2, 39.4, 37.4, 30.1, 21.6, 19.6; HRMS (ESI, m/z) Calcd for $C_{17}H_{21}NNaO_3S$ [M + Na]$^+$: 342.1140, found 342.1140.

*3-(4-Methyl-2-phenylnaphthalen-1-yl)propanal (**4a**).* Yellow solid; 25.5 mg, 0.093 mmol, yield 31%; mp 35−36 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.69 (s, 1H), 8.10−8.07 (m, 1H), 8.05−8.02 (m, 1H), 7.62−7.54 (m, 2H), 7.48−7.43 (m, 2H), 7.40 (d, J = 7.0 Hz, 1H), 7.34 (d, J = 7.3 Hz, 2H), 7.23 (s, 1H), 3.35 (t, J = 8.0 Hz, 2H), 2.75−2.69 (m, 5H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 201.6, 142.5, 139.4, 132.7, 132.6, 131.7, 131.4, 129.3, 129.2, 128.4, 127.2, 126.5, 125.6, 125.2, 124.5, 45.4, 21.5, 19.5; HRMS (ESI, m/z) Calcd for $C_{20}H_{18}NaO$ [M + Na]$^+$: 297.1255, found 297.1248.

*1-(4-Methyl-2-phenylnaphthalen-1-yl)heptan-3-one (**4b**).* Yellow solid; 73.4 mg, 0.222 mmol, yield 74%. mp 37−38 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, J = 9.4 Hz, 2H), 7.63−7.54 (m, 2H), 7.47 (t, J = 7.3 Hz, 2H), 7.41 (d, J = 7.1 Hz, 1H), 7.36 (d, J = 6.8 Hz, 2H), 7.25 (s, 1H), 3.30 (t, J = 8.2 Hz, 2H), 2.72 (s, 3H), 2.68 (t, J = 8.2 Hz, 2H), 2.29 (t, J = 7.5 Hz, 2H), 1.51 (p, J = 7.6 Hz, 2H), 1.27 (h, J = 7.5 Hz, 2H), 0.90 (t, J = 7.3 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 210.5, 142.6, 139.2, 132.5, 132.4, 132.3, 131.8, 129.2, 128.3, 127.1, 126.3, 125.5, 125.0, 124.6, 44.1, 42.5, 26.1, 23.3, 22.4, 19.4, 13.9; HRMS (ESI, m/z) Calcd for $C_{24}H_{26}NaO$ [M + Na]$^+$: 353.1881, found 353.1879.

*4-(4-Methyl-2-phenylnaphthalen-1-yl)-1-phenylbutan-2-one (**4c**).* Yellow solid; 62.3 mg, 0.171 mmol, yield 57%; mp 88−89 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10−8.06 (m, 1H), 7.98−7.93 (m, 1H), 7.60−7.52 (m, 2H), 7.47−7.39 (m, 3H), 7.36−7.27 (m, 5H), 7.23 (s, 1H), 7.14 (d, J = 6.5 Hz, 2H), 3.60 (s, 2H), 3.28 (t, J = 8.2 Hz, 2H), 2.78−2.68 (m, 5H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 207.6, 142.5, 139.2, 134.2, 132.4, 132.0, 131.8, 129.4, 129.2, 129.2, 128.8, 128.3, 127.1, 127.1, 126.3, 125.5, 125.0, 124.6, 50.1, 43.2, 23.4, 19.4; HRMS (ESI, m/z) Calcd for $C_{27}H_{24}NaO$ [M + Na]$^+$: 387.1725, found 387.1729.

*3-(4-Methyl-2-phenylnaphthalen-1-yl)butanal (**4d**).* Yellow solid; 33.7 mg, 0.117 mmol, yield 39%; mp 53−54 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.50 (s, 1H), 8.29−8.23 (m, 1H), 8.12−8.06 (m, 1H), 7.60−7.53 (m, 2H), 7.50−7.38 (m, 3H), 7.33 (d, J = 7.3 Hz, 2H), 7.20 (s, 1H), 4.11 (s, 1H), 3.08−2.87 (m, 2H), 2.69 (s, 3H), 1.59 (d, J = 7.3 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 201.9, 143.3, 139.6, 135.8, 133.4, 132.8, 131.6, 129.7, 129.2, 128.4, 127.2, 126.0, 125.6, 125.6, 125.3, 50.8, 30.4, 21.7, 19.5; HRMS (ESI, m/z) Calcd for $C_{21}H_{20}NaO$ [M + Na]$^+$: 311.1412, found 311.1403.

*2-Methyl-3-(4-methyl-2-phenylnaphthalen-1-yl)propanal (**4e**).* Yellow solid; 58.0 mg, 0.201 mmol, yield 67%; mp 30−32 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.45 (s, 1H), 8.09 (d, J = 8.3 Hz, 2H), 7.63−7.55 (m, 2H), 7.46 (t, J = 7.3 Hz, 2H), 7.42−7.33 (m, 3H), 7.25 (s, 1H), 3.60 (dd, J = 14.3, 6.2 Hz, 2H), 3.15 (dd, J = 14.3, 8.5 Hz, 2H), 2.75−2.62 (m, 4H), 0.87 (d, J = 7.1 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 204.6, 142.6, 140.2, 132.9, 132.6, 132.1, 130.0, 129.9, 129.4, 128.4, 127.1, 126.3, 125.6, 125.2, 124.8, 47.9, 29.4, 19.5, 13.6; HRMS (ESI, m/z) Calcd for $C_{21}H_{20}NaO$ [M + Na]$^+$: 311.1412, found 311.1411.

*(E)-2-Methyl-4-(4-methyl-2-phenylnaphthalen-1-yl)but-3-en-2-ol (**4f**).* Yellow solid; 49.9 mg, 0.165 mmol, yield 55%; mp 30−32 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26−8.21 (m, 1H), 8.09−8.05 (m, 1H), 7.61−7.53 (m, 2H), 7.46−7.30 (m, 6H), 6.86 (d, J = 16.2 Hz, 1H), 5.72 (d, J = 16.3 Hz, 1H), 2.75 (s, 3H), 1.29 (s, 6H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 145.0, 142.6, 137.9, 133.3, 132.2, 132.0, 131.6, 130.4, 129.2, 127.9, 126.6, 126.3, 126.0, 125.7, 124.4, 123.1, 71.3, 29.5, 19.6; HRMS (ESI, m/z) Calcd for $C_{22}H_{22}NaO$ [M + Na]$^+$: 325.1568, found 325.1568.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.joc.9b01630.

Spectral data for all novel compounds (^{1}H NMR, ^{13}C NMR) (PDF)

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Notes

The authors declare no competing financial interest.

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