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An Efficient Synthesis of Polysubstituted Naphthalene Derivatives by Gold-Catalyzed Cyclization of 1-Arylalka-2,3-dienyl Acetates

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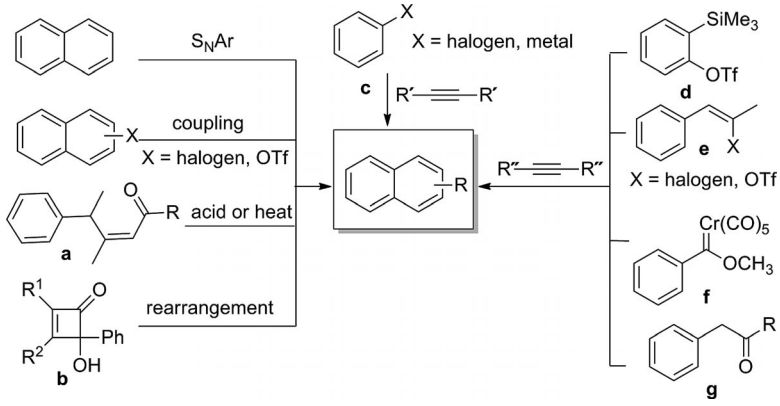
Dedicated to Professor Barluenga on the occasion of his 70th birthday

Keywords: Allenes / Naphthalene / Gold / Cyclization / Reaction mechanisms

An efficient synthetic strategy to generate differently polysubstituted naphthalenes and iodonaphthalenes through a gold-catalyzed cyclization reaction of 1-arylalka-2,3-dienyl acetates was described. Due to the substituent loading capability of both the aromatic ring and the allene moiety, different substituents may be introduced to the different locations of the naphthalenes. A possible mechanism of the reaction involving the formation of alkenyl and naphthyl Au species was proposed on the basis of the mechanistic study. Iodination of the gold species afforded iodonaphthalenes, which are useful building blocks to introduce molecular complexity and diversity by coupling reactions.

Introduction

Differently substituted naphthalene derivatives have played an important role in the chemical and pharmaceutical industries[1] as well as in the fields of optical and electronic materials.[2] The development of new and efficient methodologies for the synthesis of polysubstituted naphthalene derivatives has recently attracted much attention.[3] Traditionally, the regioselective construction of polysubstituted aromatic compounds has been carried out by the stepwise introduction of substituents through electrophilic substitutions[4] or coupling reactions.[5,6] Other important methods include cyclic alkylation of γ-aryl-α,β-unsaturated carbonyl compounds **a** (Scheme 1),[7] rearrangement of 4-hydroxycyclobut-2-enones **b**,[8] reactions of aryl halides or aryl metal compounds **c** with two molecules of alkynes,[9] reactions of highly reactive benzynes **d** with two molecules of alkynes,[10] palladium-catalyzed annulation of alkynes with 1-phenylalken-2-yl iodides/triflates **e**,[11] annulations via Fischer carbenes **f** with alkynes,[12] annulation of α-aryl-substituted carbonyl compounds **g** with alkynes,[13] and so on[14] (Scheme 1). Although there are many useful synthetic routes to naphthalenes, the mild, efficient, regiocontrolled, and diversified preparation of these compounds with specific substitution patterns remains a significant challenge to synthetic organic chemists.



Scheme 1. Known methodologies for the synthesis of polysubstituted naphthalene.

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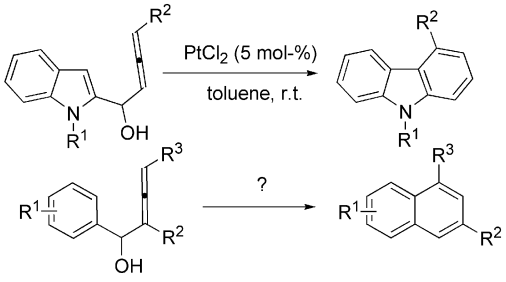
On the other hand, recently, we reported a $PtCl_2$-catalyzed intramolecular cyclization reaction of 1-(indol-2-yl)-2,3-allenols to afford carbazole derivatives (Scheme 2).[15] On the basis of these results, we wondered whether naphthalene derivatives could be afforded by using arenes instead of indoles as the starting point (Scheme 2).[16] Due to the substituent loading capability of both the aromatic ring and the allene moiety, this type of method will be of high diversity. In this area, Lee et al. reported a Au-catalyzed cyclization of 1-aryl-(2-ethoxycarbonyl)butadienols for the synthesis of naphthalenes.[17] Ohno[18] and Gagné[19] et al. also reported the Au-catalyzed cyclization of *N*-alkoxycarbonylallenylaniline and 6-arylhexadienes to afford dihydroquinolines and benzocyclohexanes, respectively. In this paper, we wish to report our recent observation on the efficient synthesis of naphthalene and iodonaphthalene derivatives from the readily available 1-aryl-alka-2,3-dienyl acetates through a mechanism different from our previous study.[15]

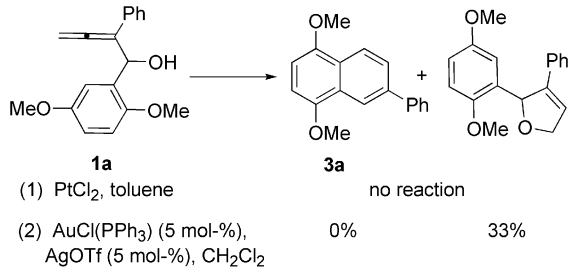


Scheme 2. Cyclization of 2,3-allenols for the formation of the benzene ring.

Results and Discussion

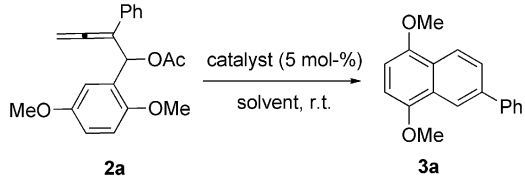
Initially, we tried the cyclization of 1-(2,5-dimethoxyphenyl)-2-phenylbuta-2,3-dienol (**1a**) with $PtCl_2$ as the catalyst;[15] however, **1a** was recovered (Scheme 3). With $AuCl(PPh_3)$ and AgOTf as the catalyst used by Lee et al.,[16] the reaction did occur; however, the cycloisomerization product, that is, 2,5-dihydrofuran, instead of the corresponding naphthalene derivatives 1,4-dimethoxy-6-phenylnaphthalene (**3a**) was formed obviously due to the higher nucleophilicity of the hydroxy oxygen atom.[20] Thus, in order to prevent the cycloisomerization involving the hydroxy group, this functionality was protected in the form of acetate to test this type of transformation.

Luckily, the reaction of acetate **2a** in 1,4-dioxane with $AuCl(PPh_3)$ and AgOTf as the catalyst gave desired product **3a**, albeit in only 17% yield (Table 1, Entry 3). It should be noted that the starting material was recovered with $PtCl_2$[15] or $AuCl_3$ as the catalyst (Table 1, Entries 1 and 2). Then, several combinations of $AuCl(PPh_3)$ with different silver salts were tested, among which $AgBF_4$ turned out to be the best, affording product **3a** in 82% NMR yield (Table 1, Entries 3–5). Notably, the solvent effect is obvious, as the reaction in CH_3CN led to recovery of substrate **2a** in 79% NMR yield, and the reaction in toluene became complicated (Table 1, Entries 6 and 7). In addition, it should be noted that with the catalyst used by Lee et al.,[17] the reaction of acetate **2a** is complicated (Table 1, Entry 8).



Scheme 3. Pt^{2+} or Au^+/Ag^+-catalyzed cyclization of allenol **1a**.

Table 1. Optimization of the reaction conditions for the naphthalene-formation reaction of 1-(2,5-dimethoxyphenyl)-2-phenylbuta-2,3-dienyl acetate (**2a**).[a]

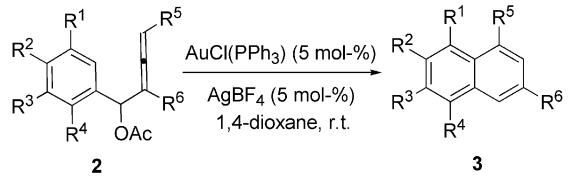


Entry	Catalyst	Solvent	Time [h]	NMR yield of **3a** [%][b]
1	$AuCl_3$	dioxane	19.6	[c]
2	$PtCl_2$	dioxane	[d]	[e]
3	$AuCl(PPh_3)$/AgOTf	dioxane	11.5	17
4	$AuCl(PPh_3)/AgSbF_6$	dioxane	11.5	56
5	$AuCl(PPh_3)/AgBF_4$	dioxane	4	82 (77)[f]
6	$AuCl(PPh_3)/AgBF_4$	CH_3CN	10	[g]
7	$AuCl(PPh_3)/AgBF_4$	toluene	12	[h]
8	$AuCl(PPh_3)/AgBF_4$	CH_2Cl_2	12	[h]

[a] The reaction was conducted with **2a** (0.2 mmol) and the catalyst (5 mol-%) at room temperature. [b] ^1H NMR yield obtained by using CH_2Br_2 as the internal standard. [c] The recovery of **2a** was 86%. [d] Room temperature, 1.3 h; 80 °C, 17 h. [e] The recovery of **2a** was 32%. [f] Isolated yield. [g] The recovery of **2a** was 79%. [h] A complicated reaction mixture was formed.

With the optimized reaction conditions in hand, the scope of this Au-catalyzed cyclization reaction of acetates **2** was studied. Substituents on the phenyl ring could be 2,5-dimethoxy, 3,4-methylenedioxy, 3,4-dimethoxy, and 3,4,5-trimethoxy, affording the corresponding naphthalenes in good yields. The R^6 group could be phenyl and alkyl. It is noteworthy that this reaction shows an interesting exclusive elimination of the OAc group to form the naphthalene ring even when R^6 is CH_2OEt or CH_2OAc (Table 2, Entries 6 and 7).

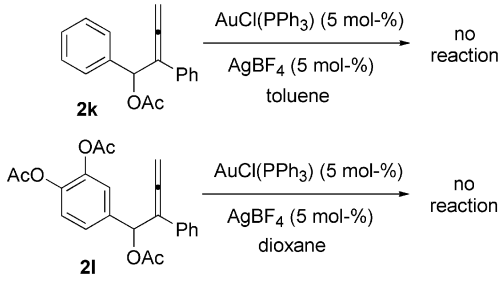
Table 2. Synthesis of naphthalene derivatives through gold-catalyzed cyclization of 1-aryl-alka-2,3-dienyl acetates.[a]



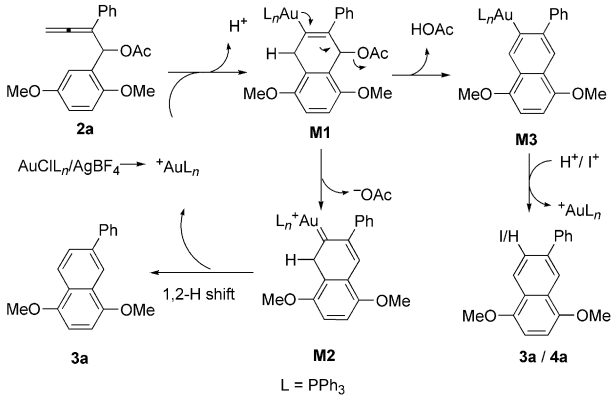
Entry	2				Time	Yield
	R^1	R^2	R^3; R^4; R^5	R^6	[h]	of **3** [%]
1	MeO	H	H; MeO; H	Ph (**2a**)	4	77 (**3a**)
2	MeO	H	H; MeO; H	*n*Bu (**2b**)	19	87 (**3b**)
3	–OCH$_2$O–		H; H; H	Ph (**2c**)	11	85 (**3c**)
4	–OCH$_2$O–		H; H; H	*n*Bu (**2d**)	9	89 (**3d**)
5	–OCH$_2$O–		H; H; H	*p*-MeC$_6$H$_5$ (**2e**)	6	86 (**3e**)
6	–OCH$_2$O–		H; H; H	CH$_2$OEt (**3f**)	12	74 (**3f**)
7	–OCH$_2$O–		H; H; H	CH$_2$OAc (**3g**)	17	84 (**3g**)
8	MeO	MeO	H; H; H	Ph (**2h**)	19	84 (**3h**)
9	MeO	MeO	MeO; H; H	*n*Bu (**2i**)	12	80 (**3i**)
10	–OCH$_2$O–		H; H; *n*-hexyl	Me (**2j**)	17	61 (**3j**)

[a] A solution of **2**, AuCl(PPh$_3$) (5 mol-%), and AgBF$_4$ (5 mol-%) was stirred in 1,4-dioxane at room temperature.

Relatively electron-rich aryl groups are required, as the reaction of **2k** or **2l** failed to proceed (Scheme 4). A mechanism involving metal carbene intermediate **M2**[21] was proposed (Scheme 5).

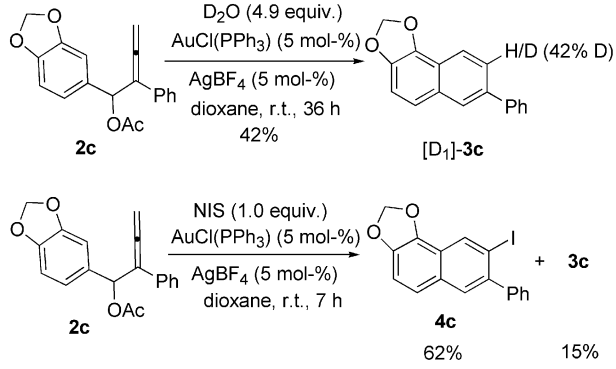


Scheme 4. The electronic effect of the substituents on the aromatic ring.



Scheme 5. Two proposed mechanisms.

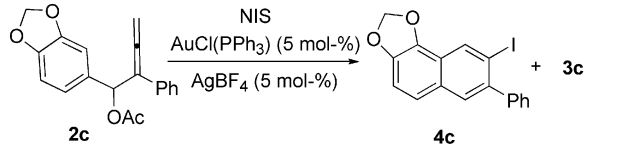
However, when D_2O was added to the reaction mixture, the reaction proceeded smoothly to afford [D_1]-**3c** in 42% yield with 42% D-incorporation at the central carbon of the allene moiety, which excludes the mechanism involving **M2** (Scheme 5). This observation prompted us to quench the reaction with I^+. In fact, β-iodonaphthalene derivative **4c** was afforded in 62% NMR yield with 15% NMR yield of **3c** when the reaction was conducted in the presence of NIS (Scheme 6).[22] It should be noted that the reaction of **2a** with 1.0 equiv. of NIS in dioxane did not proceed.



Scheme 6. The reaction in the presence of D_2O and NIS.

We were not satisfied with the results of the β-iodonaphthalene formation. Therefore, **2c** was used as the model substrate to optimize the conditions with some typical results listed in Table 3. Initially, the yield of **4c** was improved to 72% with 1.5 equiv. of NIS (Table 3, Entry 2); however, the yield of **4c** was lower when 2.0 equiv. of NIS was applied (Table 3, Entry 3); similar results were observed in acetone (Table 3, Entry 7), better than those in other solvents such as THF, DMF, Et_2O, and so on (Table 3, Entries 4–6); to improve the selectivity of iodonolysis versus protonolysis, the reaction was conducted at 0 °C in acetone, and the best yield (77%) and selectivity (94:6) were achieved (Table 3, Entry 8); no better results was obtained at –20 °C or with more NIS (Table 3, Entries 9 and 10). Thus, we de-

Table 3. Optimization of the reaction conditions for the β-iodonaphthalene formation reaction from **2c**.

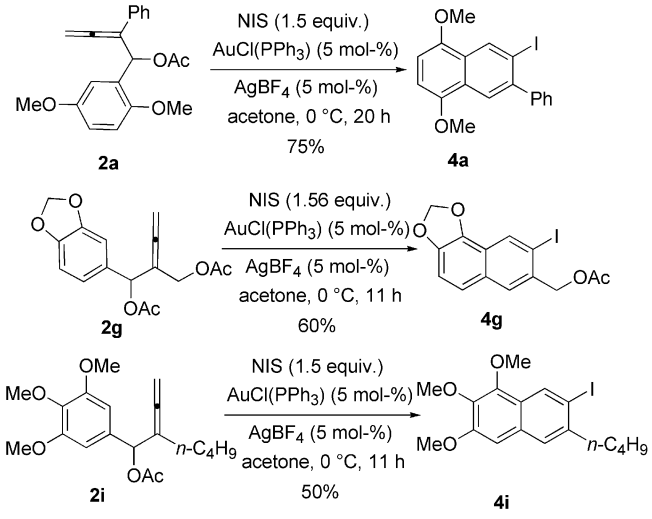


Entry	NIS [equiv.]	Solvent	*T* [°C]	Time [h]	Yield of **4c** [%][a]	Ratio of **4c**/**3c**
1	1.0	dioxane	r.t.	7	62	81:19
2	1.5	dioxane	r.t.	11	72	86:14
3	2.0	dioxane	r.t.	17	66	81:19
4	1.5	THF	r.t.	19.5	56	84:16
5	1.5	Et_2O	r.t.	19.5	20	83:17
6	1.5	DMF	r.t.	19.5	[b]	
7	1.5	acetone	r.t.	10.5	64	91:9
8	1.5	acetone	0	21	77	94:6
9	1.5	acetone	–20	22	71	93:7
10	2.0	acetone	0	15	64	94:6

[a] NMR yield determined by ^{1}H NMR spectroscopic analysis by using CH_2Br_2 as the internal standard. [b] Compound **2c** was recovered in 99% yield.

fined the experimental protocol for the cyclization of 1-arylalka-2,3-dienyl acetates under the catalysis of 5 mol-% of $AuCl(PPh_3)/AgBF_4$ and 1.5 equiv. of NIS in acetone at 0 °C as the standard reaction conditions to afford β-iodonaphthalenes (Table 3, Entry 8).

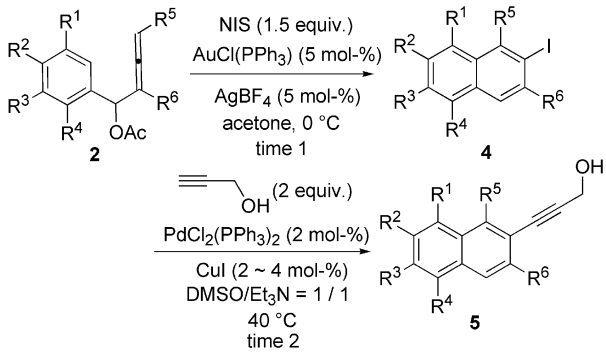
β-Iodonaphthalenes **4a**, **4g**, and **4i** were then easily synthesized under the standard conditions (Scheme 7).



Scheme 7. The reaction in the presence of NIS.

On the basis of these results, we proposed a possible mechanism for the reaction, which is different from that proposed for the similar cyclization of 1-(indol-2-yl)-2,3-allenols.[15] β-Naphthyl gold intermediate **M3** was formed by elimination of acetic acid from six-membered cyclohexenyl gold species **M1**. Finally, protonolysis or iodonolysis released the gold catalyst into the catalytic cycle and afforded target naphthalene **3a** or iodonaphthalene **4a**, respectively (Scheme 5). The reaction of some substrates afforded **4** together with a very minor amount of **3**, which could not be separated by chromatography on silica gel. Thus, all the iodonaphthalenes were further converted into 3-(naphthalen-2-yl)prop-2-yn-1-ol derivatives by Sonogashira coupling with propargyl alcohol (Table 4).

Table 4. Synthesis of β-iodonaphthalene derivatives by gold-catalyzed cyclization of 1-arylalka-2,3-dienyl acetates and subsequent Sonogashira coupling reaction with propargyl alcohol.



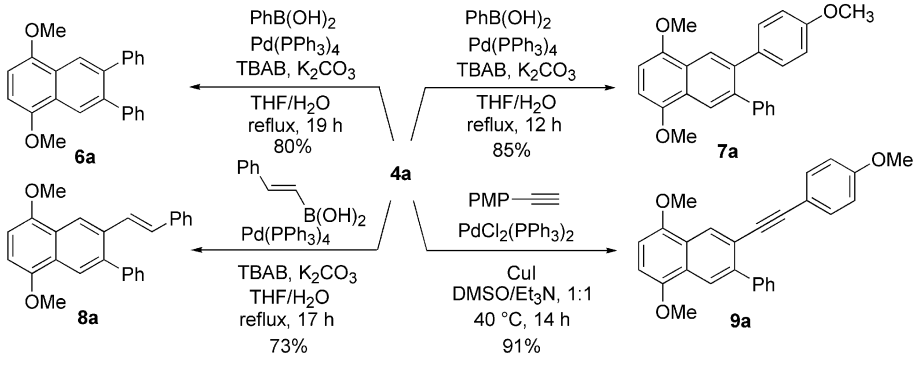
Entry	Substrate	Time 1 [h]	Yield of **4** [%][a]	Ratio of **4**/**3**	Time 2 [h]	Yield of **5** [%][b]
1	**2a**	11	84 (**4a**)	100:0	31	58 (**5a**)
2	**2b**	12	79 (**4b**)	94:6	14	53 (**5b**)
3	**2c**	21	77 (**4c**)	94:6	24	61 (**5c**)
4	**2d**	12	89 (**4d**)	95:5	18	67 (**5d**)
5	**2e**	16	89 (**4e**)	95:5	14	52 (**5e**)
6	**2f**	14	71 (**4f**)	96:4	23	47 (**5f**)
7	**2g**	11	62 (**4g**)	100:0	11	48 (**5g**)
8	**2h**	12	71 (**4h**)	91:9	37.5	43 (**5h**)
9	**2i**	11	62 (**4i**)	100:0	32	48 (**5i**)

[a] NMR yield determined by ^1H NMR spectroscopic analysis by using CH_2Br_2 as the internal standard. [b] Combined isolated yields of two steps from **2**.

Transformations of **4a** by Suzuki coupling with different boronic acids and Sonogashira cross-coupling with other terminal alkynes, leading to the formation **6a**, **7a**, **8a**, and **9a** in yields of 73–91%, were further demonstrated to show the synthetic potential of these cyclization products (Scheme 8).[23]

Conclusions

In summary, we have developed an efficient method to generate naphthalene and β-iodonaphthalene derivatives through the simple intramolecular *C*-alkylation of 1-aryl-



Scheme 8. Pd-catalyzed cross-coupling strategy of **4a**.

buta-2,3-dienyl acetate catalyzed by 5 mol-% each of $AuCl(PPh_3)$ and $AgBF_4$. A possible mechanism of the reaction was proposed on the basis of the mechanistic studies. Due to the easy availability of the starting materials,[24] mild reaction conditions (room temperature), and potential of the products, this method may be useful in organic synthesis, material science, and medicinal chemistry.

Experimental Section

Synthesis of 1-(2,5-Dimethoxyphenyl)-2-phenylbuta-2,3-dienyl Acetate (2a) as a Representative General Procedure for the Preparation of 1-Aryl-alka-2,3-dienyl Acetates 2a–j: To a dried one-neck round-bottomed flask equipped with a magnetic stir bar were added **1a** (0.5645 g, 2.0 mmol), Et_3N (0.5 mL), DMAP (49.5 mg, 0.41 mmol), and dry Et_2O (8 mL). To this stirred solution was added Ac_2O (0.7524 g, 7.4 mmol), and the resulting mixture was stirred at room temperature until the reaction was complete as monitored by TLC. The reaction was quenched with saturated aqueous $NaHCO_3$. The aqueous layer was extracted with diethyl ether (3 × 15 mL), and the combined organic layers were washed with water and dried with anhydrous Na_2SO_4. Following filtration and evaporation, the residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate, 10:1) to give **2a** (0.5421 g, 84%) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.43–7.34 (m, 2 H, Ar*H*), 7.32–7.23 (m, 2 H, Ar*H*), 7.22–7.13 (m, 2 H, Ar*H*), 6.99 (d, *J* = 2.1 Hz, 1 H, C*H*OAc), 6.84–6.74 (m, 2 H, Ar*H*), 5.19–5.08 (m, 2 H, CH_2=), 3.79 (s, 3 H, OCH_3), 3.73 (s, 3 H, OCH_3), 2.08 (s, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 208.9, 169.9, 153.4, 151.0, 133.9, 128.3, 127.9, 127.0, 126.4, 114.5, 113.4, 111.6, 106.8, 80.6, 67.4, 56.2, 55.6, 21.1 ppm. IR (neat): $\tilde{\nu}$ = 2940, 2835, 1943, 1743, 1597, 1499, 1464, 1431, 1371, 1280, 1227, 1179, 1158, 1047, 1026 cm^{-1}. MS (70 eV, EI): *m*/*z* (%) = 324 (5.41) [M]$^+$, 251 (100). HRMS: calcd. for $C_{20}H_{20}O_4$ [M]$^+$ 324.1362; found 324.1359.

1-(2,5-Dimethoxyphenyl)-2-vinylidenehexyl Acetate (2b): According to the general procedure, the reaction of **1b** (1.5410 g, 5.9 mmol), Et_3N (3.4 mL), DMAP (143.2 mg, 11.7 mmol), and Ac_2O (2.2 mL, *d* = 1.082 g/mL, 2.3804 g, 23 mmol) in Et_2O (40 mL) afforded **2b** (1.4970 g, 84%) after chromatography (petroleum ether/ethyl acetate = 10:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.95 (t, *J* = 1.5 Hz, 1 H, Ar*H*), 6.85–6.75 (m, 2 H, Ar*H*), 6.55 (t, *J* = 2.7 Hz, 1 H, C*H*OAc), 4.78 (q, *J* = 2.7 Hz, 2 H, CH_2=), 3.78 (s, 3 H, OCH_3), 3.77 (s, 3 H, OCH_3), 2.09 (s, 3 H, COCH_3), 1.98–1.83 (m, 2 H), 1.48–1.20 (m, 4 H), 0.86 (t, *J* = 7.2 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 206.0, 169.9, 153.4, 151.1, 128.2, 114.1, 113.2, 111.7, 104.3, 78.3, 69.3, 56.2, 55.7, 29.5, 28.2, 22.2, 21.2, 13.9 ppm. IR (neat): $\tilde{\nu}$ = 2956, 2931, 2836, 1959, 1743, 1591, 1501, 1465, 1430, 1370, 1280, 1230, 1179, 1159, 1049, 1027 cm^{-1}. MS (70 eV, EI): *m*/*z* (%) = 304 (11.27) [M]$^+$, 231 (100). HRMS: calcd. for $C_{18}H_{24}O_4$ [M]$^+$ 304.1675; found 304.1677.

1-(Benzo[*d*][1,3]dioxol-5-yl)-2-phenylbuta-2,3-dienyl Acetate (2c): According to the general procedure, the reaction of **1c** (2.6615 g, 10 mmol), Et_3N (5 mL), DMAP (244.1 mg, 2 mmol), and Ac_2O (4 mL, *d* = 1.082 g/mL, 4.3280 g, 42.4 mmol) in Et_2O (40 mL) afforded **2c** (2.0649 g, 67%) after chromatography (petroleum ether/ethyl acetate, 10:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.38–7.30 (m, 2 H, Ar*H*), 7.29–7.22 (m, 2 H, Ar*H*), 7.21–7.13 (m, 1 H, Ar*H*), 6.96–6.88 (m, 2 H, Ar*H*), 6.78–6.71 (m, 1 H, Ar*H*), 6.68 (t, *J* = 2.6 Hz, 1 H, C*H*OAc), 5.92 (s, 2 H, OCH_2O), 5.22 (d, *J* = 2.4 Hz, 2 H, CH_2=), 2.08 (s, 3 H, COCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 208.7, 170.0, 147.7, 147.6, 133.6, 132.1, 128.4, 127.1, 126.6, 121.5, 108.0, 106.8, 101.1, 80.7, 73.1, 21.2 ppm. IR (neat): $\tilde{\nu}$ = 2901, 1942, 1742, 1598, 1504, 1489, 1445, 1369, 1227, 1099, 1038 cm^{-1}. MS (70 eV, EI): *m*/*z* (%) = 308 (16.20) [M]$^+$, 266 (100). HRMS: calcd. for $C_{19}H_{16}O_4$ [M]$^+$ 308.1049; found 308.1056.

1-(Benzo[*d*][1,3]dioxol-5-yl)-2-vinylidenehexyl Acetate (2d): According to the general procedure, the reaction of **1d** (0.4901 g, 2 mmol), Et_3N (1 mL), DMAP (49.6 mg, 0.41 mmol), and Ac_2O (0.7201 g, 7.1 mmol) in Et_2O (10 mL) afforded **2d** (0.3400 g, 59%) after chromatography (petroleum ether/ethyl acetate, 20:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.88–6.80 (m, 2 H, Ar*H*), 6.76 (d, *J* = 8.1 Hz, 1 H, Ar*H*), 6.04 (t, *J* = 2.7 Hz, 1 H, C*H*OAc), 6.00–5.90 (m, 2 H, OCH_2O), 4.86 (q, *J* = 3.1 Hz, 2 H, CH_2=), 2.08 (s, 3 H, COCH_3), 1.90–1.78 (m, 2 H), 1.45–1.20 (m, 4 H), 0.85 (t, *J* = 7.2 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 205.5, 169.9, 147.6, 147.4, 132.4, 121.1, 107.9, 107.7, 104.5, 101.1, 78.6, 75.3, 29.4, 27.9, 22.3, 21.2, 13.9 ppm. IR (neat): $\tilde{\nu}$ = 2957, 2930, 2877, 1959, 1743, 1504 1489, 1444, 1369, 1231, 1099, 1040 cm^{-1}. MS (70 eV, EI): *m*/*z* (%) = 288 (13.27) [M]$^+$, 189 (100). HRMS: calcd. for $C_{17}H_{20}O_4$ [M]$^+$ 288.1362; found 288.1360.

1-(Benzo[*d*][1,3]dioxol-5-yl)-2-*p*-tolylbuta-2,3-dienyl Acetate (2e): According to the general procedure, the reaction of **1e** (1.4257 g, 5.1 mmol), Et_3N (2.5 mL), DMAP (125.1 mg, 1.0 mmol), and Ac_2O (1.9102 g, 18.7 mmol) in Et_2O (20 mL) afforded **2e** (1.1896 g, 73%) after chromatography (petroleum ether/ethyl acetate, 80:1–40:1) as a solid; m.p. 66–68 °C (ethyl acetate/*n*-hexane). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.23 (d, *J* = 8.7 Hz, 2 H, Ar*H*), 7.07 (d, *J* = 8.4 Hz, 2 H, Ar*H*), 6.92 (d, *J* = 8.4 Hz, 2 H, Ar*H*), 6.74 (d, *J* = 8.7 Hz, 1 H, Ar*H*), 6.70–6.63 (m, C*H*OAc), 5.92 (s, 2 H, OCH_2O), 5.19 (d, *J* = 2.1 Hz, 2 H, CH_2=), 2.28 (s, 3 H, COCH_3), 2.08 (s, 3 H, ArCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 208.4, 169.9, 147.6, 147.5, 136.8, 132.1, 130.5, 129.1, 126.5, 121.5, 108.01, 107.99, 106.6, 101.1, 80.5, 73.1, 21.2, 21.0 ppm. IR (neat): $\tilde{\nu}$ = 2897, 1942, 1742, 1606, 1504, 1489, 1445, 1369, 1229, 1099, 1038 cm^{-1}. MS (70 eV, EI): *m*/*z* (%) = 322 (7.86) [M]$^+$, 280 (100). $C_{20}H_{18}O_4$ (322.36): calcd. C 74.52, H 5.63; found C 74.45, H 5.50.

1-(Benzo[*d*][1,3]dioxol-5-yl)-2-(ethoxymethyl)buta-2,3-dienyl Acetate (2f): According to the general procedure, the reaction of **1f** (0.6011 g, 2.4 mmol), Et_3N (0.5 mL), DMAP (25.0 mg, 0.2 mmol), and Ac_2O (0.9125 g, 8.9 mmol) in Et_2O (10 mL) afforded **2f** (0.5887 g, 84%) after chromatography (petroleum ether/ethyl acetate, 20:1–10:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.92–6.83 (m, 2 H, Ar*H*), 6.76 (d, *J* = 4.5 Hz, 1 H, Ar*H*), 6.19 (t, *J* = 2.7 Hz, 1 H, C*H*OAc), 5.97–5.92 (m, 2 H, OCH_2O), 4.91 (d, *J* = 2.7 Hz, 2 H, CH_2=), 3.98 (dt, *J* = 12.0, 2.7 Hz, 1 H, CH_2O), 3.86 (dt, *J* = 12.0, 2.7 Hz, 1 H, CH_2O), 3.57–3.30 (m, 2 H, OCH_2CH$_3$), 2.07 (s, 3 H, COCH_3), 1.16 (t, *J* = 6.9 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 206.2, 169.8, 147.6, 147.5, 132.0, 121.2, 107.9, 107.8, 102.1, 101.1, 78.4, 73.0, 68.8, 65.2, 21.1, 15.0 ppm. IR (neat): $\tilde{\nu}$ = 2976, 2894, 1959, 1744, 1610, 1504, 1489, 1445, 1370, 1232, 1096, 1039 cm^{-1}. MS (70 eV, EI): *m*/*z* (%) = 290 (6.95) [M]$^+$, 151 (100). HRMS: calcd. for $C_{16}H_{18}O_5$ [M]$^+$ 290.1154; found 290.1154.

1-(Benzo[*d*][1,3]dioxol-5-yl)-2-vinylidenepropane-1,3-diyl Diacetate (2g): According to the general procedure, the reaction of **1g** (0.3764 g, 1.7 mmol), Et_3N (0.5 mL), DMAP (42.5 mg, 0.35 mmol), and Ac_2O (0.6752 g, 6.6 mmol) in Et_2O (8 mL) afforded **2g** (0.4041 g, 78%) after chromatography (petroleum ether/ethyl acetate, 10:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.87–6.82 (m, 2 H, Ar*H*), 6.76 (d, *J* = 8.4 Hz, 1 H, Ar*H*), 6.20 (t, *J* = 2.7 Hz, 1 H, C*H*OAc), 5.96 (s, 2 H, OCH_2O), 4.98 (q, *J* = 2.4 Hz, 2 H, CH_2=), 4.61 (dt, *J* = 12.3, 2.1 Hz, 1 H, CH_2O), 4.45

(dt, J = 12.3, 2.1 Hz, 1 H, CH_2OAc), 2.09 (s, 3 H, COCH_3), 2.02 (s, 3 H, COCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 207.0, 170.5, 169.7, 147.63, 147.56, 131.5, 121.0, 107.9, 107.6, 101.1, 100.7, 79.3, 72.9, 62.0, 21.0, 20.7 ppm. IR (neat): $\tilde{\nu}$ = 1961, 1743, 1504, 1489, 1445, 1374, 1232, 1098, 1037 cm^{-1}. MS (70 eV, EI): m/z (%) = 305 (0.73) [M + 1]$^+$, 304 (4.06) [M]$^+$, 43 (100). HRMS: calcd. for $C_{16}H_{16}O_6$ [M]$^+$ 304.0947; found 304.0946.

1-(3,4-Dimethoxyphenyl)-2-phenylbuta-2,3-dienyl Acetate (2h): According to the general procedure, the reaction of **1h** (1.0012 g, 3.6 mmol), Et_3N (1 mL), DMAP (86.4 mg, 0.71 mmol), and Ac_2O (1.2912 g, 12.7 mmol) in Et_2O (15 mL) afforded **2h** (0.9508 g, 83%) after chromatography (petroleum ether/ethyl acetate, 10:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.39–7.32 (m, 2 H, ArH), 7.31–7.23 (m, 2 H, ArH), 7.22–7.14 (m, 1 H, ArH), 7.01 (dd, J = 8.4, 1.8 Hz, 1 H, ArH), 6.95 (d, J = 1.8 Hz, 1 H, ArH), 6.81 (d, J = 8.4 Hz, 1 H, ArH), 6.73 (t, J = 2.4 Hz, 1 H, CHOAc), 5.20 (d, J = 2.7 Hz, 2 H, CH_2=), 3.86 (s, 6 H, OCH_3), 2.10 (s, 3 H, COCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 208.8, 170.1, 149.0, 148.8, 133.8, 130.6, 128.4, 127.1, 126.6, 120.3, 110.8, 110.7, 106.9, 80.6, 73.2, 55.83, 55.79, 21.2 ppm. IR (neat): $\tilde{\nu}$ = 2936, 2836, 1942, 1740, 1595, 1517, 1496, 1464, 1452, 1420, 1370, 1231, 1155, 1140, 1027 cm^{-1}. MS (70 eV, EI): m/z (%) = 324 (2.68) [M]$^+$, 264 (100). HRMS: calcd. for $C_{20}H_{20}O_4$ [M]$^+$ 324.1362; found 324.1362.

1-(3,4,5-Trimethoxyphenyl)-2-vinylidenehexyl Acetate (2i): According to the general procedure, the reaction of **1i** (0.8474 g, 2.9 mmol), Et_3N (1 mL), DMAP (70.0 mg, 0.57 mmol), and Ac_2O (1.1012 g, 10.8 mmol) in Et_2O (10 mL) afforded **2i** (0.8564 g, 88%) after chromatography (petroleum ether/ethyl acetate, 10:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.58 (s, 2 H, ArH), 6.07 (t, J = 2.4 Hz, 1 H, CHOAc), 4.87 (q, J = 3.0 Hz, 2 H, CH_2=), 3.859 (s, 3 H, OCH_3), 3.857 (s, 3 H, OCH_3), 3.834 (s, 3 H, OCH_3), 2.11 (s, 3 H, COCH_3), 1.92–1.79 (m, 2 H), 1.45–1.20 (m, 4 H), 0.85 (t, J = 7.1 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 205.6, 170.0, 153.0, 137.8, 133.9, 104.5, 104.3, 78.6, 75.5, 60.7, 56.1, 29.4, 27.9, 22.2, 21.2, 13.8 ppm. IR (neat): $\tilde{\nu}$ = 2967, 2935, 2873, 2839, 1958, 1744, 1592, 1506, 1463, 1421, 1371, 1332, 1232, 1128, 1012 cm^{-1}. MS (70 eV, EI): m/z (%) = 334 (2.28) [M]$^+$, 274 (100). HRMS: calcd. for $C_{19}H_{26}O_5$ [M]$^+$ 334.1780; found 334.1782.

1-(Benzo[*d*][1,3]dioxol-5-yl)-2-methyldeca-2,3-dienyl Acetate (2j): According to the general procedure, the reaction of **1j** (0.8654 g, 3.0 mmol), Et_3N (1 mL), DMAP (74.9 mg, 0.61 mmol), and Ac_2O (1.0210 g, 10.0 mmol) in Et_2O (10 mL) afforded **2j** (0.7566 g, 77%) after chromatography (petroleum ether/ethyl acetate, 40:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.90–6.70 (m, 3 H, ArH), 6.02 (dd, J = 6.9, 2.1 Hz, 1 H, CHOAc), 5.96–5.89 (m, 2 H, OCH_2O), 5.30–5.10 (m, 1 H, CH=), 2.08 (s, 3 H, COCH_3), 2.10–1.90 (m, 2 H), 1.59 (t, J = 2.6 Hz, 3 H, CH_3), 1.45–1.20 (m, 8 H), 0.88 (t, J = 6.8 Hz, 3 H, CH$_2$CH_3) ppm. IR (neat): $\tilde{\nu}$ = 2961, 2927, 2856, 1961, 1744, 1606, 1504, 1489, 1444, 1368, 1232, 1097, 1040 cm^{-1}. MS (70 eV, EI): m/z (%) = 330 (11.51) [M]$^+$, 203 (100). $C_{20}H_{26}O_4$ (330.42): calcd. C 72.70, H 7.93; found C 72.89, H 8.10.

Synthesis of 1,4-Dimethoxy-6-phenylnaphthalene (3a) as a Representative General Procedure for the Preparation of Naphthalenes 3a–j: A dried Schlenk tube was charged with AuCl(PPh$_3$) (4.9 mg, 0.01 mmol), $AgBF_4$ (2.5 mg, 0.013 mmol), **2a** (64.0 mg, 0.20 mmol), and dioxane (1 mL) sequentially under an atmosphere of N_2. After continuous stirring for 4 h at room temperature, the reaction was complete as monitored by TLC. Evaporation and column chromatography on silica gel (petroleum ether/ethyl acetate, 80:l) afforded **3a** (40.0 mg, 77%) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.48 (d, J = 1.8 Hz, 1 H, ArH), 8.31 (d, J = 8.7 Hz, 1 H, ArH), 7.86–7.75 (m, 3 H, ArH), 7.51 (t, J = 7.2 Hz, 2 H, ArH), 7.45–7.35 (m, 1 H, ArH), 6.75 (d, J = 8.4 Hz, 1 H, ArH), 6.71 (d, J = 8.4 Hz, 1 H, ArH), 4.00 (s, 3 H), 3.99 (s, 3 H, OCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 149.7, 149.4, 141.3, 138.4, 128.7, 127.4, 127.2, 126.5, 125.4, 125.3, 122.4, 119.8, 103.6, 103.2, 55.7 ppm. IR (neat): $\tilde{\nu}$ = 2991, 2936, 2832, 1629, 1599, 1494, 1463, 1422, 1394, 1363, 1272, 1236, 1165, 1104, 1042, 1001 cm^{-1}. MS (70 eV, EI): m/z (%) = 265 (14.88) [M + 1]$^+$, 264 (75.57) [M]$^+$, 249 (100) [M]$^+$. HRMS: calcd. for $C_{18}H_{16}O_2$ [M]$^+$ 264.1150; found 264.1151.

6-Butyl-1,4-dimethoxynaphthalene (3b): According to the general procedure, the reaction of AuCl(PPh$_3$) (7.5 mg, 0.015 mmol), $AgBF_4$ (3.2 mg, 0.016 mmol), and **2b** (90.7 mg, 0.3 mmol) in dioxane (1.5 mL) at room temperature for 19 h afforded **3b** (63.1 mg, 87%) after chromatography (petroleum ether/ethyl acetate, 60:l) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.17 (d, J = 8.7 Hz, 1 H, ArH), 8.04 (d, J = 0.9 Hz, 1 H, ArH), 7.40 (dd, J = 8.6, 1.7 Hz, 1 H, ArH), 6.70 (d, J = 8.4 Hz, 1 H, ArH), 6.66 (d, J = 8.4 Hz, 1 H, ArH), 3.99 (s, 3 H, OCH_3), 3.97 (s, 3 H, OCH_3), 2.83 (t, J = 7.8 Hz, 2 H), 1.82–1.66 (m, 2 H), 1.54–1.32 (m, 2 H), 0.99 (t, J = 7.4 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 149.5, 149.1, 140.6, 127.3, 126.4, 124.7, 121.7, 120.2, 103.1, 102.1, 55.6, 36.0, 33.7, 22.4, 14.0 ppm. IR (neat): $\tilde{\nu}$ = 2997, 2954, 2931, 2857, 2833, 1633, 1603, 1510, 1463, 1426, 1369, 1271, 1243, 1210, 1194, 1162, 1098, 1006 cm^{-1}. MS (70 eV, EI): m/z (%) = 245 (11.70) [M + 1]$^+$, 244 (65.88) [M]$^+$, 229 (100) [M]$^+$. HRMS: calcd. for $C_{16}H_{20}O_2$ [M]$^+$ 244.1463; found 244.1462.

7-Phenylnaphtho[2,1-*d*][1,3]dioxole (3c): According to the general procedure, the reaction of AuCl(PPh$_3$) (7.5 mg, 0.015 mmol), $AgBF_4$ (3.5 mg, 0.018 mmol), and **2c** (90.5 mg, 0.29 mmol) in dioxane (2 mL) at room temperature for 11 h afforded **3c** (61.7 mg, 85%) after chromatography (petroleum ether/ethyl acetate, 100:l) as a solid; m.p. 138–139 °C (diethyl ether/*n*-hexane). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.90 (d, J = 1.2 Hz, 1 H, ArH), 7.80–7.69 (m, 3 H, ArH), 7.63 (dd, J = 8.4, 1.8 Hz, 1 H, ArH), 7.58–7.46 (m, 2 H, ArH), 7.45–7.35 (m, 1 H, ArH), 7.18 (d, J = 8.4 Hz, 2 H, ArH), 6.05 (s, 2 H, OCH_2O) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 147.8, 147.6, 141.1, 137.1, 130.7, 129.6, 128.7, 127.4, 127.2, 127.1, 125.0, 123.9, 104.1, 103.6, 101.0 ppm. IR (KBr): $\tilde{\nu}$ = 2934, 2831, 1625, 1579, 1498, 1459, 1413, 1322, 1274, 1237, 1107, 1091 cm^{-1}. MS (70 eV, EI): m/z (%) = 249 (18.73) [M + 1]$^+$. 248 (100) [M]$^+$. $C_{17}H_{12}O_2$ (248.28): calcd. C 82.26, H 4.84; found C 82.24, H 4.99.

7-Butylnaphtho[2,1-*d*][1,3]dioxole (3d): According to the general procedure, the reaction of AuCl(PPh$_3$) (7.4 mg, 0.015 mmol), $AgBF_4$ (3.1 mg, 0.015 mmol), and **2d** (87.1 mg, 0.3 mmol) in dioxane (2 mL) at room temperature for 9 h afforded **3d** (61.2 mg, 89%) after chromatography (petroleum ether/ethyl acetate, 100:l) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.61 (d, J = 8.1 Hz, 1 H, ArH), 7.47 (s, 1 H, ArH), 7.22 (d, J = 8.1 Hz, 1 H, ArH), 7.11 (d, J = 7.2 Hz, 2 H, ArH), 6.02 (s, 2 H, OCH_2O), 2.75 (t, J = 7.8 Hz, 2 H), 1.78–1.62 (m, 2 H), 1.52–1.35 (m, 2 H), 1.00 (t, J = 7.4 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 147.5, 146.9, 138.9, 130.6, 128.6, 126.8, 125.7, 103.6, 103.5, 100.8, 35.6, 33.6, 22.4, 14.0 ppm. IR (neat): $\tilde{\nu}$ = 2958, 2925, 2856, 1618, 1495, 1466, 1445, 1257, 1177, 1150, 1044. MS (70 eV, EI): m/z (%) = 229 (6.35) [M + 1]$^+$, 228 (39.25) [M]$^+$, 185 (100). HRMS: calcd. for $C_{15}H_{16}O_2$ [M]$^+$ 228.1150; found 228.1152.

7-*p*-Tolylnaphtho[2,1-*d*][1,3]dioxole (3e): According to the general procedure, the reaction of AuCl(PPh$_3$) (7.2 mg, 0.015 mmol), $AgBF_4$ (3.1 mg, 0.015 mmol), and **2e** (95.4 mg, 0.3 mmol) in dioxane (2 mL) at room temperature for 6 h afforded **3e** (66.8 mg, 86%) after chromatography (petroleum ether/ethyl acetate, 80:l) as a solid; m.p. 152–153 °C (ethyl acetate/*n*-hexane). ^{1}H NMR (300 MHz,

$CDCl_3$): δ = 7.83 (s, 1 H, Ar*H*), 7.71 (d, *J* = 8.4 Hz, 1 H, Ar*H*), 7.53–7.51 (m, 3 H, Ar*H*), 7.27 (d, *J* = 8.4 Hz, 2 H, Ar*H*), 7.15 (s, 1 H, Ar*H*), 7.12 (s, 1 H, Ar*H*), 6.03 (s, 2 H, OCH_2O), 2.41 (s, 3 H, ArCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 147.8, 147.5, 138.2, 137.0, 136.8, 130.7, 129.5, 129.4, 127.3, 127.0, 124.7, 123.8, 104.0, 103.6, 101.0, 21.1 ppm. IR (KBr): $\tilde{\nu}$ = 3024, 2913, 1618, 1507, 1494, 1474, 1443, 1385, 1273, 1245, 1202, 1159, 1078, 1047. MS (70 eV, EI): *m*/*z* (%) = 263 (19.40) $[M + 1]^+$, 262 (100) $[M]^+$. $C_{18}H_{14}O_2$ (262.31): calcd. C 82.42, H 5.38; found C 82.51, H 5.46.

7-(Ethoxymethyl)naphtho[2,1-*d*][1,3]dioxole (3f): According to the general procedure, the reaction of $AuCl(PPh_3)$ (7.3 mg, 0.015 mmol), $AgBF_4$ (3.2 mg, 0.016 mmol), and **2f** (69.1 mg, 0.3 mmol) in dioxane (1.5 mL) at room temperature for 12 h afforded **3f** (40.3 mg, 74%) after chromatography (petroleum ether/ethyl acetate, 40:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.68–7.58 (m, 2 H, Ar*H*), 7.33 (dd, *J* = 8.4, 1.8 Hz, 1 H, Ar*H*), 7.11 (s, 2 H, Ar*H*), 6.02 (s, 2 H, OCH_2O), 4.62 (s, 2 H, ArCH_2O), 3.58 (q, *J* = 7.0 Hz, 2 H, OCH_2CH$_3$), 1.28 (t, *J* = 7.0 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 147.7, 147.5, 134.5, 130.3, 129.9, 127.1, 125.8, 124.4, 103.9, 103.7, 101.0, 72.8, 65.7, 15.2 ppm. IR (neat): $\tilde{\nu}$ = 2974, 2864, 1618, 1498, 1467, 1444, 1371, 1341, 1323, 1239, 1177, 1145, 1113, 1071, 1041. MS (70 eV, EI): *m*/*z* (%) = 231 (8.68) $[M + 1]^+$, 230 (55.75) $[M]^+$, 185 (100). HRMS: calcd. for $C_{14}H_{14}O_3$ $[M]^+$ 230.0943; found 230.0945.

Naphtho[2,1-*d*][1,3]dioxol-7-ylmethyl Acetate (3g): According to the general procedure, the reaction of $AuCl(PPh_3)$ (5.6 mg, 0.011 mmol), $AgBF_4$ (2.5 mg, 0.013 mmol), and **2g** (61.2 mg, 0.2 mmol) in dioxane (1 mL) at room temperature for 17 h afforded **3g** (41.5 mg, 84%) after chromatography (petroleum ether/ethyl acetate, 20:1) as a white solid; m.p. 97–98 °C (ethyl acetate/*n*-hexane). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.68–7.60 (m, 2 H, Ar*H*), 7.33 (dd, *J* = 8.4, 1.8 Hz, 1 H, Ar*H*), 7.11 (s, 2 H, Ar*H*), 6.05 (s, 2 H, OCH_2O), 5.21 (s, 2 H, ArCH_2O), 2.12 (s, 3 H, COCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 170.9, 147.9, 131.8, 130.2, 127.3, 126.7, 124.5, 103.9, 103.7, 101.1, 66.5, 21.1 ppm. IR (KBr): $\tilde{\nu}$ = 2955, 1737, 1467, 1380, 1365, 1253, 1173, 1149, 1076, 1034. MS (70 eV, EI): *m*/*z* (%) = 245 (9.26) $[M + 1]^+$, 244 (61.22) $[M]^+$, 185 (100). $C_{14}H_{12}O_4$ (244.25): calcd. C 68.85, H 4.95; found C 68.55, H 4.98.

1,2-Dimethoxy-6-phenylnaphthalene (3h): According to the general procedure, the reaction of $AuCl(PPh_3)$ (7.5 mg, 0.015 mmol), $AgBF_4$ (3.5 mg, 0.018 mmol), and **2h** (98.5 mg, 0.3 mmol) in dioxane (1.5 mL) at room temperature for 19 h afforded **3h** (67.5 mg, 84%) after chromatography (petroleum ether/ethyl acetate, 10:1) as a solid; m.p. 126–127 °C (ethyl acetate/*n*-hexane). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.91 (s, 1 H, Ar*H*), 7.81–7.67 (m, 3 H, Ar*H*), 7.66–7.56 (m, 1 H, Ar*H*), 7.48 (t, *J* = 7.5 Hz, 2 H, Ar*H*), 7.36 (t, *J* = 7.5 Hz, 1 H, Ar*H*), 7.19 (s, 1 H, Ar*H*), 7.15 (s, 1 H, Ar*H*), 4.03 (s, 6 H, OCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 149.7, 149.5, 141.3, 136.9, 129.4, 128.8, 128.3, 127.2, 127.0, 126.8, 124.3, 123.8, 106.5, 106.0, 55.9 ppm. IR (KBr): $\tilde{\nu}$ = 2967, 2931, 2871, 1627, 1606, 1497, 1463, 1414, 1255, 1196, 1165, 1134, 1007. MS (70 eV, EI): *m*/*z* (%) = 265 (18.89) $[M + 1]^+$, 264 (100) $[M]^+$. $C_{18}H_{16}O_2$ (264.32): calcd. C 81.79, H 6.10; found C 81.99, H 6.34.

6-Butyl-1,2,3-trimethoxynaphthalene (3i): According to the general procedure, the reaction of $AuCl(PPh_3)$ (7.8 mg, 0.015 mmol), $AgBF_4$ (3.5 mg, 0.018 mmol), and **2i** (101.5 mg, 0.3 mmol) in dioxane (1.5 mL) at room temperature for 12 h afforded **3i** (67.0 mg, 80%) after chromatography (petroleum ether/ethyl acetate, 60:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.98 (d, *J* = 8.4 Hz, 1 H, Ar*H*), 7.49 (s, 1 H, Ar*H*), 7.23 (dd, *J* = 8.6, 1.7 Hz, 1 H, Ar*H*), 6.91 (s, 1 H, Ar*H*), 4.06 (s, 3 H, OCH_3), 3.981 (s, 3 H, OCH_3), 3.977 (s, 3 H, OCH_3), 2.75 (t, *J* = 7.7 Hz, 2 H), 1.78–1.60 (m, 2 H), 1.49–1.30 (m, 2 H), 0.97 (t, *J* = 7.4 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 152.9, 147.9, 140.3, 140.1, 131.0, 125.2, 125.0, 122.6, 121.5, 101.9, 61.3, 61.1, 55.7, 35.7, 33.5, 22.3, 14.0 ppm. IR (neat): $\tilde{\nu}$ = 2956, 2932, 2857, 1629, 1605, 1576, 1502, 1480, 1465, 1411, 1393, 1336, 1252, 1203, 1130, 1105, 1040, 1003. MS (70 eV, EI): *m*/*z* (%) = 275 (19.24) $[M + 1]^+$, 274 (100) $[M]^+$. HRMS: calcd. for $C_{17}H_{22}O_3$ $[M]^+$ 274.1569; found 274.1571.

9-Hexyl-7-methylnaphtho[2,1-*d*][1,3]dioxole (3j): According to the general procedure, the reaction of $AuCl(PPh_3)$ (7.1 mg, 0.014 mmol), $AgBF_4$ (3.1 mg, 0.015 mmol), and **2j** (99.1 mg, 0.3 mmol) in dioxane (2 mL) at room temperature for 17 h afforded **3j** (49.2 mg, 61%) after chromatography (petroleum ether/ethyl acetate, 100:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.37–7.28 (m, 2 H, Ar*H*), 7.10–7.03 (m, 2 H, Ar*H*), 6.03 (s, 2 H, OCH_2O), 2.92 (t, *J* = 7.8 Hz, 2 H, ArCH_2), 2.45 (s, 3 H, ArCH_3), 1.79–1.65 (m, 2 H), 1.53–1.28 (m, 6 H), 0.93 (t, *J* = 7.1 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 147.0, 146.9, 137.9, 133.5, 133.1, 126.77, 126.71, 124.8, 104.0, 100.8, 100.4, 33.6, 31.8, 30.7, 29.6, 22.7, 21.4, 14.1 ppm. IR (neat): $\tilde{\nu}$ = 2928, 1621, 1501, 1469, 1232, 1042. MS (70 eV, EI): *m*/*z* (%) = 271 (6.08) $[M + 1]^+$, 270 (27.47) $[M]^+$, 199 (100). HRMS: calcd. for $C_{18}H_{22}O_2$ $[M]^+$ 270.1620; found 270.1629.

Synthesis of Iodonaphthalenes 4

6-Iodo-1,4-dimethoxy-7-phenylnaphthalene (4a): A dried Schlenk tube was charged with $AuCl(PPh_3)$ (8.0 mg, 0.016 mmol), $AgBF_4$ (3.2 mg, 0.016 mmol), **2a** (96.1 mg, 0.30 mmol), acetone (2 mL), and NIS (100.1 mg, 0.44 mmol) sequentially at 0 °C under an atmosphere of N_2. After stirring for 20 h, the reaction was complete as monitored by TLC. The reaction mixture was diluted with diethyl ether (10 mL) and quenched with saturated aqueous solution of $Na_2S_2O_3$. The mixture was extracted with diethyl ether (3 × 15 mL), and the combined organic layers were washed with water and dried with anhydrous Na_2SO_4. Following filtration and evaporation, the residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate, 40:1) to afford **4a** (86.9 mg, 75%; ^{1}H NMR yield by using CH_2Br_2 as the internal standard, **4a**/**3a** = 100:0) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.89 (s, 1 H, Ar*H*), 8.17 (s, 1 H, Ar*H*), 7.52–7.40 (m, 5 H, Ar*H*), 6.74–6.68 (m, 2 H, Ar*H*), 3.97 (s, 3 H, OCH_3), 3.93 (s, 3 H, OCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 149.2, 148.1, 144.2, 143.1, 133.1, 129.6, 127.7, 127.5, 126.5, 125.6, 122.7, 104.0, 103.9, 96.8, 55.7, 55.6 ppm. IR (neat): $\tilde{\nu}$ = 3009, 2967, 2934, 2823, 1625, 1579, 1498, 1459, 1413, 1395, 1322, 1274, 1237, 1212, 1155, 1107, 1089, 1020. MS (70 eV, EI): *m*/*z* (%) = 391 (21.10) $[M + 1]^+$, 390 (100) $[M]^+$. HRMS: calcd. for $C_{18}H_{15}IO_2$ $[M]^+$ 390.0117; found 390.0117.

(8-Iodonaphtho[2,1-*d*][1,3]dioxol-7-yl)methyl Acetate (4g): According to the procedure outlined for **4a**, the reaction of $AuCl(PPh_3)$ (7.4 mg, 0.015 mmol), $AgBF_4$ (3.1 mg, 0.015 mmol), **2g** (90.1 mg, 0.3 mmol), and NIS (105.1 mg, 0.47 mmol) in acetone (2 mL) at 0 °C for 11 h afforded **4g** (65.4 mg, 60%; 62% ^{1}H NMR yield by using CH_2Br_2 as the internal standard, **4g**/**3g** = 100:0) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.14 (s, 1 H, Ar*H*), 7.62 (s, 1 H, Ar*H*), 7.04 (s, 1 H, Ar*H*), 6.95 (s, 1 H, Ar*H*), 6.05 (s, 2 H, OCH_2O), 5.19 (s, 2 H, ArCH_2O), 2.16 (s, 3 H, COCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 170.6, 148.4, 148.3, 137.5, 132.9, 131.8, 129.8, 127.8, 103.7, 102.4, 101.3, 93.1, 70.0, 21.0 ppm. IR (neat): $\tilde{\nu}$ = 2913, 1737, 1615, 1486, 1459, 1377, 1236, 1039. MS (70 eV, EI): *m*/*z* (%) = 370 (31.79) $[M]^+$, 201 (100). HRMS: calcd. for $C_{14}H_{11}O_4I$ $[M]^+$ 369.9702; found 369.9708.

6-Butyl-7-iodo-1,2,3-trimethoxynaphthalene (4i): According to the procedure outlined for **4a**, the reaction of $AuCl(PPh_3)$ (7.4 mg, 0.015 mmol), $AgBF_4$ (3.5 mg, 0.015 mmol), **2i** (101.5 mg, 0.3 mmol), and NIS (102.5 mg, 0.45 mmol) in acetone (2 mL) at 0 °C for afforded **4i** (60.9 mg, 50%; 60% ^1H NMR yield by using CH_2Br_2 as the internal standard, **4i/3i** = 100:0) as a liquid. ^1H NMR (300 MHz, $CDCl_3$): δ = 8.52 (s, 1 H, Ar*H*), 7.49 (s, 1 H, Ar*H*), 6.83 (s, 1 H, Ar*H*), 4.04 (s, 3 H, OCH_3), 3.96 (s, 3 H, OCH_3), 3.95 (s, 3 H, OCH_3), 2.81 (t, *J* = 7.8 Hz, 2 H), 1.71–1.58 (m, 2 H), 1.54–1.38 (m, 2 H), 0.98 (t, *J* = 7.4 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 153.5, 146.6, 141.5, 140.4, 132.5, 130.5, 125.7, 124.3, 101.5, 96.2, 61.4, 61.1, 55.8, 40.3, 32.6, 22.4, 14.0 ppm. IR (neat): $\tilde{\nu}$ = 2955, 2933, 2871, 1622, 1589, 1570, 1483, 1459, 1424, 1393, 1368, 1241, 1205, 1151, 1110, 1042, 1002. MS (70 eV, EI): *m*/*z* (%) = 400 (100) $[M]^+$. HRMS: calcd. for $C_{17}H_{21}O_3I$ $[M]^+$ 400.0535; found 400.0530.

Synthesis of 3-(5,8-Dimethoxy-3-phenylnaphthalen-2-yl)prop-2-yn-1-ol (5a) as a Representative General Procedure for the Preparation of 3-(Naphthalen-2-yl)prop-2-yn-1-ols by Gold-Catalyzed Cyclization of 1-Arylalka-2,3-dienyl Acetates and Subsequent Sonogashira Coupling Reaction with Propargyl Alcohol 2: The reaction of $AuCl(PPh_3)$ (5.1 mg, 0.01 mmol), $AgBF_4$ (2.5 mg, 0.01 mmol), **2a** (64.4 mg, 0.2 mmol), and NIS (67.9 mg, 0.3 mmol) in acetone (1.5 mL) at 0 °C for 11 h afforded **4a** in 84% ^1H NMR yield by using CH_2Br_2 as the internal standard (**4a/3a** = 100:0). Product **4a** was then used without further purification. A Schlenk tube was charged with $Pd(PPh_3)_2Cl_2$ (2.9 mg, 0.004 mmol), CuI (1.2 mg, 0.006 mmol), **4a** (prepared in the previous step), Et_3N (1 mL), prop-2-yn-1-ol (25.1 mg, 0.45 mmol), and DMSO (1 mL) sequentially. The mixture was heated at 40 °C under an atmosphere of nitrogen. After the reaction was complete as monitored by TLC, the reaction was quenched with water (5 mL). The aqueous layer was extracted with diethyl ether (3 × 15 mL), and the combined organic layers were washed with water and dried with anhydrous Na_2SO_4. Following filtration and evaporation, the residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate, 5:1) to afford **5a** (36.9 mg, combined yield from **2a** to **5a** is 58%) as a liquid. ^1H NMR (300 MHz, $CDCl_3$): δ = 8.47 (s, 1 H, Ar*H*), 8.20 (s, 1 H, Ar*H*), 7.75–7.65 (m, 2 H, Ar*H*), 7.51–7.34 (m, 3 H, Ar*H*), 6.72 (d, *J* = 8.4 Hz, 1 H, Ar*H*), 6.69 (d, *J* = 8.4 Hz, 1 H, Ar*H*), 4.38 (s, 2 H, CH_2OH), 3.96 (s, 3 H, OCH_3), 3.94 (s, 3 H, OCH_3), 1.76 (br. s, 1 H, O*H*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 149.3, 148.9, 140.6, 140.3, 129.5, 127.8, 127.6, 127.3, 125.7, 124.8, 122.5, 118.9, 104.6, 103.8, 90.1, 85.9, 55.71, 55.66, 51.7 ppm. IR (neat): $\tilde{\nu}$ = 3385, 3000, 2936, 2833, 2252, 2216, 1626, 1592, 1499, 1460, 1431, 1393, 1333, 1277, 1266, 1213, 1157, 1101, 1038, 1024. MS (70 eV, EI): *m*/*z* (%) = 319 (23.48) $[M + 1]^+$, 318 (100) $[M]^+$. HRMS: calcd. for $C_{21}H_{18}O_3$ $[M]^+$ 318.1256; found 318.1257.

3-(3-Butyl-5,8-dimethoxynaphthalen-2-yl)prop-2-yn-1-ol (5b): According to the general procedure, the reaction of $AuCl(PPh_3)$ (7.3 mg, 0.015 mmol), $AgBF_4$ (3.1 mg, 0.015 mmol), **2b** (91.2 mg, 0.3 mmol), and NIS (100.9 mg, 0.45 mmol) in acetone (2 mL) at 0 °C for 12 h afforded **4b** in 79% ^1H NMR yield by using CH_2Br_2 as the internal standard (**4b/3b** = 94:6). After column chromatography on silica gel (petroleum ether/ethyl acetate, 40:1), the mixture was used in next step. The reaction of $Pd(PPh_3)_2Cl_2$ (4.4 mg, 0.006 mmol), CuI (2.1 mg, 0.011 mmol), **4b** (prepared in the previous step), DMSO (1 mL), prop-2-yn-1-ol (35.6 mg, 0.64 mmol), and Et_3N (1 mL) afforded **5b** (47.7 mg, combined yield from **2b** to **5b** is 53%) after chromatography (petroleum ether/ethyl acetate, 7:1) as a liquid. ^1H NMR (300 MHz, $CDCl_3$): δ = 8.33 (s, 1 H, Ar*H*), 7.99 (d, *J* = 0.3 Hz, 1 H, Ar*H*), 6.65 (d, *J* = 8.4 Hz, 1 H, Ar*H*), 6.61 (d, *J* = 8.4 Hz, 1 H, Ar*H*), 4.57 (s, 2 H, HOCH_2), 3.94 (s, 3 H, OCH_3), 3.92 (s, 3 H, OCH_3), 2.92 (t, *J* = 7.8 Hz, 2 H), 2.04 (br. s, 1 H, O*H*), 1.80–1.65 (m, 2 H), 1.50–1.35 (m, 2 H), 0.97 (t, *J* = 7.4 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 149.1, 148.8, 141.3, 126.8, 125.8, 124.3, 120.7, 120.4, 104.2, 102.8, 90.2, 85.0, 55.6, 51.7, 34.4, 32.8, 22.5, 14.0 ppm. IR (neat): $\tilde{\nu}$ = 3384, 2955, 2932, 2859, 1633, 1597, 1463, 1433, 1344, 1326, 1268, 1240, 1199, 1169, 1090, 1022. MS (70 eV, EI): *m*/*z* (%) = 298 (100) $[M]^+$. HRMS: calcd. for $C_{19}H_{22}O_3$ $[M]^+$ 298.1569; found 298.1569.

3-(7-Phenylnaphtho[2,1-*d*][1,3]dioxol-8-yl)prop-2-yn-1-ol (5c): According to the general procedure, the reaction of $AuCl(PPh_3)$ (5.1 mg, 0.01 mmol), $AgBF_4$ (2.4 mg, 0.01 mmol), **2c** (64.4 mg, 0.2 mmol), and NIS (66.8 mg, 0.3 mmol) in acetone (1 mL) at 0 °C for 21 h afforded **4c** in 77% ^1H NMR yield by using CH_2Br_2 as the internal standard (**4c/3c** = 94:6). Product **4c** was then used without further purification. The reaction of $Pd(PPh_3)_2Cl_2$ (2.9 mg, 0.004 mmol), CuI (1.1 mg, 0.006 mmol), **4c** (prepared in the previous step), DMSO (1 mL), prop-2-yn-1-ol (23.0 mg, 0.4 mmol), and Et_3N (1 mL) afforded **5c** (38.7 mg, combined yield from **2c** to **5c** is 61%) after chromatography (petroleum ether/ethyl acetate, 5:1 to 2:1) as a solid; m.p. 143–144 °C (ethyl acetate/*n*-hexane). ^1H NMR (300 MHz, $CDCl_3$): δ = 7.87 (s, 1 H, Ar*H*), 7.65–7.60 (m, 3 H, Ar*H*), 7.47–7.37 (m, 3 H, Ar*H*), 7.07 (d, *J* = 8.4 Hz, 2 H, Ar*H*), 5.99–6.11 (m, 2 H, OCH_2O), 4.37 (d, *J* = 5.4 Hz, 2 H, HOCH_2), 1.68 (br. s, 1 H, O*H*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 148.6, 148.1, 140.4, 139.2, 132.1, 130.4, 129.4, 129.1, 127.9, 127.5, 127.3, 117.4, 103.8, 103.2, 101.2, 89.7, 85.7, 51.7 ppm. IR (KBr): $\tilde{\nu}$ = 3327, 2204, 1483, 1460, 1444, 1275, 1227, 1158, 1038, 1023. MS (70 eV, EI): *m*/*z* (%) = 302 (100) $[M]^+$. $C_{20}H_{14}O_3$ (302.33): calcd. C 79.46, H 4.67; found C 79.42, H 4.74.

3-(7-Butylnaphtho[2,1-*d*][1,3]dioxol-8-yl)prop-2-yn-1-ol (5d): According to the general procedure, the reaction of $AuCl(PPh_3)$ (7.4 mg, 0.015 mmol), $AgBF_4$ (3.5 mg, 0.015 mmol), **2d** (87.0 mg, 0.3 mmol), and NIS (101.2 mg, 0.45 mmol) in acetone (2 mL) at 0 °C for 12 h afforded **4d** in 89% ^1H NMR yield by using CH_2Br_2 as the internal standard (**4d/3d** = 95:5). Product **4d** was then used without further purification. The reaction of $Pd(PPh_3)_2Cl_2$ (4.3 mg, 0.006 mmol), CuI (1.4 mg, 0.007 mmol), **4d** (prepared in the previous step), DMSO (1 mL), prop-2-yn-1-ol (34.1 mg, 0.6 mmol), and Et_3N (1 mL) afforded **5d** (56.8 mg, combined yield from **2d** to **5d** is 67%) after chromatography (petroleum ether/ethyl acetate, 10:1 to 5:1) as a solid; m.p. 98–99 °C (ethyl acetate/*n*-hexane). ^1H NMR (300 MHz, $CDCl_3$): δ = 7.73 (s, 1 H, Ar*H*), 7.40 (s, 1 H, Ar*H*), 7.00 (s, 1 H, Ar*H*), 6.98 (s, 1 H, Ar*H*), 6.00 (s, 2 H, OCH_2O), 4.57 (s, 2 H, HOCH_2), 2.83 (t, *J* = 7.8 Hz, 2 H), 2.10 (br. s, 1 H, O*H*), 1.75–1.59 (m, 2 H), 1.50–1.31 (m, 2 H), 0.96 (t, *J* = 7.4 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 148.2, 147.3, 139.8, 131.3, 130.5, 128.2, 126.1, 118.8, 103.3, 103.2, 101.0, 89.9, 84.8, 51.7, 34.0, 32.7, 22.5, 14.0 ppm. IR (KBr): $\tilde{\nu}$ = 3331, 2961, 2928, 2847, 1633, 1613, 1491, 1461, 1441, 1353, 1257, 1230, 1211, 1155, 1040. MS (70 eV, EI): *m*/*z* (%) = 282 (100) $[M]^+$. $C_{18}H_{18}O_3$ (282.34): calcd. C 76.57, H 6.43; found C 76.55, H 6.32.

3-(7-*p*-Tolylnaphtho[2,1-*d*][1,3]dioxol-8-yl)prop-2-yn-1-ol (5e): According to the general procedure, the reaction of $AuCl(PPh_3)$ (7.3 mg, 0.015 mmol), $AgBF_4$ (3.1 mg, 0.015 mmol), **2e** (96.6 mg, 0.3 mmol), and NIS (102.4 mg, 0.45 mmol) in acetone (2 mL) at 0 °C for 16 h afforded **4e** in 89% ^1H NMR yield by using CH_2Br_2 as the internal standard (**4e/3e** = 95:5). After column chromatography on silica gel (petroleum ether/ethyl acetate, 40:1), the mixture was used in next step. The reaction of $Pd(PPh_3)_2Cl_2$ (4.5 mg, 0.006 mmol), CuI (2.0 mg, 0.011 mmol), **4e** (prepared in the previous step), DMSO (1 mL), prop-2-yn-1-ol (40.0 mg, 0.71 mmol), and Et_3N (1 mL) afforded **5e** (49.5 mg, combined yield from **2e** to

5e is 52%) as a solid; m.p. 166–167 °C (ethyl acetate/*n*-hexane). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.86 (s, 1 H, Ar*H*), 7.60 (s, 1 H, Ar*H*), 7.53 (d, *J* = 7.8 Hz, 2 H, Ar*H*), 7.24 (d, *J* = 7.2 Hz, 2 H, Ar*H*), 7.06 (d, *J* = 6.9 Hz, 2 H, Ar*H*), 6.04 (s, 2 H, OCH_2O), 4.38 (s, 2 H, HOCH_2), 2.41 (s, 3 H, ArCH_3), 1.65–1.45 (m, 1 H, O*H*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 148.6, 148.1, 139.2, 137.5, 137.1, 132.1, 130.5, 129.2, 128.9, 128.6, 127.4, 117.4, 103.8, 103.2, 101.2, 89.7, 85.9, 51.8, 21.2. IR (KBr): $\tilde{\nu}$ = 3382, 3009, 2905, 2233, 1612, 1485, 1458, 1438, 1384, 1342, 1278, 1226, 1183, 1158, 1115, 1088, 1035, 1000. MS (70 eV, EI): *m*/*z* (%) = 316 (100) $[M]^+$. $C_{21}H_{16}O_3$ (316.36): calcd. C 79.73, H 5.10; found C 79.74, H 5.37.

3-{7-(Ethoxymethyl)naphtho[2,1-*d*][1,3]dioxol-8-yl}prop-2-yn-1-ol (5f): According to the general procedure, the reaction of AuCl(PPh_3) (7.9 mg, 0.016 mmol), $AgBF_4$ (4.2 mg, 0.022 mmol), **2f** (87.1 mg, 0.3 mmol), and NIS (102.5 mg, 0.45 mmol) in acetone (2 mL) at 0 °C for 14 h afforded **4f** in 71% ^{1}H NMR yield by using CH_2Br_2 as the internal standard (**4f**/**3f** = 96:4). After column chromatography on silica gel (petroleum ether/ethyl acetate, 40:1), the mixture was used in next step. The reaction of $Pd(PPh_3)_2Cl_2$ (4.3 mg, 0.006 mmol), CuI (1.9 mg, 0.010 mmol), **4f** (prepared in the previous step), DMSO (1.5 mL), prop-2-yn-1-ol (34.1 mg, 0.6 mmol), and Et_3N (1.5 mL) afforded **5f** (47 mg, combined yield from **2f** to **5f** is 47%) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.72 (s, 1 H, Ar*H*), 7.66 (s, 1 H, Ar*H*), 7.06 (s, 1 H, Ar*H*), 6.99 (s, 1 H, Ar*H*), 6.02 (s, 2 H, OCH_2O), 4.73 (d, *J* = 0.6 Hz, 2 H, HOCH_2), 4.53 (s, 2 H, CH_2OEt), 3.63 (q, *J* = 7.0 Hz, 2 H), 2.85–2.72 (br. s, 1 H, O*H*), 1.30 (t, *J* = 6.9 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 148.3, 147.9, 135.0, 131.3, 130.2, 129.2, 125.7, 117.5, 103.8, 103.3, 101.1, 91.0, 83.5, 70.8, 66.0, 51.5, 15.2 ppm. IR (neat): $\tilde{\nu}$ = 3386, 2991, 2903, 2865, 2222, 1609, 1495, 1462, 1371, 1359, 1256, 1223, 1101, 1038. MS (70 eV, EI): *m*/*z* (%) = 284 (100) $[M]^+$. HRMS: calcd. for $C_{17}H_{16}O_4$ $[M]^+$ 284.1049; found 284.1057.

{8-(3-Hydroxyprop-1-ynyl)naphtho[2,1-*d*][1,3]dioxol-7-yl}methyl Acetate (5g): According to the general procedure, the reaction of AuCl(PPh_3) (5.1 mg, 0.01 mmol), $AgBF_4$ (2.2 mg, 0.01 mmol), **2g** (60.5 mg, 0.2 mmol), and NIS (68.2 mg, 0.3 mmol) in acetone (1.5 mL) at 0 °C for 11 h afforded **4g** in 62% ^{1}H NMR yield by using CH_2Br_2 as the internal standard (**4g**/**3g** = 100:0). Product **4g** was then used without further purification. The reaction of $Pd(PPh_3)_2Cl_2$ (2.9 mg, 0.004 mmol), CuI (1.2 mg, 0.006 mmol), **4g** (prepared in the previous step), DMSO (1 mL), prop-2-yn-1-ol (23.9 mg, 0.4 mmol), and Et_3N (1 mL) afforded **5g** (28.6 mg, combined yield from **2g** to **5g** is 48%) after chromatography (petroleum ether/ethyl acetate, 5:1 to 1:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.75 (s, 1 H, Ar*H*), 7.61 (s, 1 H, Ar*H*), 7.05 (s, 1 H, Ar*H*), 7.01 (s, 1 H, Ar*H*), 6.05 (s, 2 H, OCH_2O), 5.35 (s, 2 H, HOCH_2), 4.52 (d, *J* = 5.4 Hz, 2 H, CH_2OAc), 2.45 (br. s, 1 H, O*H*), 2.14 (s, 3 H, COCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 171.3, 148.6, 148.4, 132.5, 131.3, 130.0, 129.8, 127.3, 118.0, 103.9, 103.4, 101.3, 91.4, 83.2, 64.9, 51.5, 21.1 ppm. IR (neat): $\tilde{\nu}$ = 3445, 2933, 2859, 2225, 1626, 1602, 1567, 1489, 1467, 1427, 1398, 1300, 1267, 1226, 1203, 1154, 1102, 1036, 1001. MS (70 eV, EI): *m*/*z* (%) = 298 (100) $[M]^+$, 238 (85.62) $[M - HOAc]^+$. HRMS: calcd. for $C_{17}H_{14}O_5$ $[M]^+$ 298.0841; found 298.0847.

3-(7,8-Dimethoxy-3-phenylnaphthalen-2-yl)prop-2-yn-1-ol (5h): According to the general procedure, the reaction of AuCl(PPh_3) (7.5 mg, 0.015 mmol), $AgBF_4$ (3.5 mg, 0.015 mmol), **2h** (97.1 mg, 0.3 mmol), and NIS (101.1 mg, 0.45 mmol) in acetone (2 mL) at 0 °C for 12 h afforded **4h** in 71% ^{1}H NMR yield by using CH_2Br_2 as the internal standard (**4h**/**3h** = 91:9). Product **4h** was then used without further purification. The reaction of $Pd(PPh_3)_2Cl_2$ (4.3 mg, 0.006 mmol), CuI (2.0 mg, 0.010 mmol), **4h** (prepared in the previous step), DMSO (1.5 mL), prop-2-yn-1-ol (35.2 mg, 0.6 mmol), and Et_3N (1.5 mL) afforded **5h** (41.2 mg, combined yield from **2h** to **5h** is 43%) after chromatography (petroleum ether/ethyl acetate, 3:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.92 (s, 1 H, Ar*H*), 7.80–7.58 (m, 3 H, Ar*H*), 7.57–7.28 (m, 3 H, Ar*H*), 7.08 (d, *J* = 8.7 Hz, 2 H, Ar*H*), 4.39 (d, *J* = 5.7 Hz, 2 H, HOCH_2), 4.01 (s, 6 H, OCH_3), 1.62–1.44 (m, 1 H, O*H*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 150.5, 150.0, 140.6, 139.1, 131.5, 129.5, 129.1, 127.9, 127.8, 127.3, 126.9, 117.2, 106.2, 105.6, 89.6, 85.9, 55.9, 51.8 ppm. IR (neat): $\tilde{\nu}$ = 3429, 2937, 2859, 2240, 1621, 1600, 1497, 1420, 1356, 1275, 1241, 1149, 1007. MS (70 eV, EI): *m*/*z* (%) = 300 (19.20) $[M - H_2O]^+$, 284 (100). HRMS: calcd. for $C_{21}H_{18}O_3$ $[M]^+$ 318.1256; found 318.1251.

3-(3-Butyl-6,7,8-trimethoxynaphthalen-2-yl)prop-2-yn-1-ol (5i): According to the general procedure, the reaction of AuCl(PPh_3) (7.5 mg, 0.015 mmol), $AgBF_4$ (3.2 mg, 0.015 mmol), **2i** (101.5 mg, 0.3 mmol), and NIS (101.5 mg, 0.45 mmol) in acetone (2 mL) at 0 °C for 11 h afforded **4i** in 62% ^{1}H NMR yield by using CH_2Br_2 as the internal standard (**4i**/**3i** = 100:0). Product **4i** was then used without further purification. The reaction of $Pd(PPh_3)_2Cl_2$ (4.4 mg, 0.006 mmol), CuI (1.2 mg, 0.006 mmol), **4i** (prepared in the previous step), DMSO (1.5 mL), prop-2-yn-1-ol (35.1 mg, 0.6 mmol), and Et_3N (1.5 mL) afforded **5i** (47.5 mg, combined yield from **2i** to **5i** is 48%) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.14 (s, 1 H, Ar*H*), 7.44 (s, 1 H, Ar*H*), 6.84 (s, 1 H, Ar*H*), 4.57 (d, *J* = 2.1 Hz, 2 H, HOCH_2), 4.03 (s, 3 H, OCH_3), 3.95 (s, 6 H, OCH_3), 2.85 (t, *J* = 7.8 Hz, 2 H), 2.13 (br. s, 1 H, O*H*), 1.74–1.60 (m, 2 H), 1.48–1.32 (m, 2 H), 0.95 (t, *J* = 7.2 Hz, 3 H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 153.7, 147.4, 141.1, 140.4, 130.7, 126.5, 125.4, 122.3, 118.2, 101.7, 89.7, 85.0, 61.4, 61.1, 55.8, 51.7, 34.0, 32.7, 22.4, 13.9 ppm. IR (neat): $\tilde{\nu}$ = 3391, 2924, 1728, 1699, 1492, 1453, 1434, 1380, 1261, 1230, 1086, 1026. MS (70 eV, EI): *m*/*z* (%) = 329 (22.18) $[M + 1]^+$, 328 (100). HRMS: calcd. for $C_{20}H_{24}O_4$ $[M]^+$ 328.1675; found 328.1682.

Synthesis of 1,4-Dimethoxy-6,7-diphenylnaphthalene (6a) as a Representative General Procedure for the Suzuki Coupling Reactions:[23] A Schlenk tube was charged with $Pd(PPh_3)_4$ (8.9 mg, 0.0077 mmol), TBAB (2.5 mg, 0.0075 mmol), K_2CO_3 (42.0 mg, 0.30 mmol), H_2O (0.15 mL), $PhB(OH)_2$ (25.1 mg, 0.21 mmol), **4a** (57.0 mg, 0.15 mmol), and THF (1.5 mL) sequentially. The mixture was heated at reflux under an atmosphere of nitrogen. After the reaction was complete as monitored by TLC, evaporation of the solvents and purification by column chromatography on silica gel (petroleum ether/ethyl acetate, 20:l) afforded **6a** (39.8 mg, 80%) as a white solid; m.p. 171–172 °C (ethyl acetate/*n*-hexane). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.26 (s, 2 H, Ar*H*), 7.30–7.15 (m, 10 H, Ar*H*), 6.72 (s, 2 H, Ar*H*), 3.97 (s, 6 H, OCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 149.6, 141.8, 138.9, 130.1, 127.7, 126.4, 125.6, 123.7, 103.6, 55.8 ppm. IR (KBr): $\tilde{\nu}$ = 2991, 2934, 2831, 1625, 1579, 1498, 1459, 1413, 1322, 1274, 1237, 1107, 1091. MS (70 eV, EI): *m*/*z* (%) = 341 (26.01) $[M + 1]^+$, 340 (100) $[M]^+$. $C_{24}H_{20}O_2$ (340.42): calcd. C 84.68, H 5.92; found C 84.41, H 6.02.

1,4-Dimethoxy-6-(4-methoxyphenyl)-7-phenylnaphthalene (7a): According to the general procedure, the reaction of $Pd(PPh_3)_4$ (12.1 mg, 0.01 mmol), TBAB (3.3 mg, 0.01 mmol), K_2CO_3 (56.1 mg, 0.40 mmol), H_2O (0.20 mL), 4-methoxyphenylboronic acid (34.0 mg, 0.22 mmol), **4a** (78.5 mg, 0.20 mmol), and THF (2 mL) afforded **7a** (63.3 mg, 85%) after chromatography (petroleum ether/ethyl acetate, 80:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.23 (d, *J* = 1.5 Hz, 2 H, Ar*H*), 7.30–7.10 (m, 5 H, Ar*H*), 7.16 (d, *J* = 8.7 Hz, 2 H, Ar*H*), 6.77 (d, *J* = 8.7 Hz, 2 H, Ar*H*), 6.69 (s, 2 H, Ar*H*), 3.944 (s, 3 H, OCH_3), 3.939 (s, 3 H,

OCH_3), 3.76 (s, 3 H, OCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 158.3, 149.5, 149.4, 141.9, 138.9, 138.5, 134.1, 131.1, 130.0, 127.8, 126.3, 125.6, 125.3, 123.7, 123.4, 113.2, 103.5, 103.4, 55.7, 55.1 ppm. IR (neat): $\tilde{\nu}$ = 2999, 2934, 2834, 1609, 1594, 1515, 1460, 1432, 1333, 1271, 1247, 1224, 1178, 1105, 1048, 1034. MS (70 eV, EI): *m*/*z* (%) = 371 (26.90) $[M + 1]^+$, 370 (100) $[M]^+$. HRMS: calcd. for $C_{25}H_{22}O_3$ $[M]^+$ 370.1569; found 370.1566.

(*E*)-1,4-Dimethoxy-6-phenyl-7-styrylnaphthalene (8a): According to the general procedure, the reaction of $Pd(PPh_3)_4$ (12.5 mg, 0.01 mmol), TBAB (3.5 mg, 0.01 mmol), K_2CO_3 (59.2 mg, 0.40 mmol), H_2O (0.20 mL), (*E*)-styrylboronic acid (35.1 mg, 0.22 mmol), **4a** (74.1 mg, 0.19 mmol), and THF (2 mL) afforded **8a** (50.5 mg, 73%) after chromatography (petroleum ether/ethyl acetate, 200:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.57 (s, 1 H, CH=C*H*), 8.16 (s, 1 H, CH=C*H*), 7.54–7.38 (m, 7 H, Ar*H*), 7.31 (t, *J* = 7.5 Hz, 2 H, Ar*H*), 7.26–7.18 (m, 3 H, Ar*H*), 6.72 (d, *J* = 8.4 Hz, 1 H, Ar*H*), 6.69 (d, *J* = 8.4 Hz, 1 H, Ar*H*), 4.02 (s, 3 H, OCH_3), 3.95 (s, 3 H, OCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 149.53, 149.49, 141.1, 139.4, 137.7, 134.0, 130.1, 129.9, 128.6, 128.2, 128.0, 127.4, 127.0, 126.6, 125.7, 125.5, 123.2, 118.9, 103.5, 103.4, 55.8, 55.7 ppm. IR (neat): $\tilde{\nu}$ = 2943, 1593, 1492, 1461, 1435, 1326, 1269, 1241, 1143, 1094. MS (70 eV, EI): *m*/*z* (%) = 367 (31.96) $[M + 1]^+$, 366 (100) $[M]^+$. HRMS: calcd. for $C_{26}H_{22}O_2$ $[M]^+$ 366.1620; found 366.1619.

Synthesis of 1,4-Dimethoxy-6-[(4-methoxyphenyl)ethynyl]-7-phenylnaphthalene (9a) Following the Procedure for the Sonogashira Coupling:[23] The reaction of $Pd(PPh_3)_2Cl_2$ (3.5 mg, 0.005 mmol), CuI (1.9 mg, 0.01 mmol), **4a** (78.0 mg, 0.20 mmol), Et_3N (1 mL), 1-ethynyl-4-methoxybenzene (35.1 mg, 0.27 mmol), and DMSO (1 mL) afforded **9a** (72.1 mg, 91%) after chromatography (petroleum ether/ethyl acetate, 30:1) as a liquid. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.55 (d, *J* = 0.3 Hz, 1 H, Ar*H*), 8.27 (d, *J* = 0.6 Hz, 1 H, Ar*H*), 7.88–7.78 (m, 2 H, Ar*H*), 7.58–7.40 (m, 3 H, Ar*H*), 7.38–7.28 (m, 2 H, Ar*H*), 6.90–6.80 (m, 2 H, Ar*H*), 6.71 (s, 2 H, Ar*H*), 3.99 (s, 3 H, OCH_3), 3.95 (s, 3 H, OCH_3), 3.80 (s, 3 H, OCH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ = 159.4, 149.5, 149.0, 140.9, 140.4, 132.8, 129.7, 127.7, 127.3, 126.7, 125.5, 125.0, 122.4, 120.1, 115.7, 113.9, 104.3, 103.8, 92.5, 88.8, 55.8, 55.7, 55.3 ppm. IR (neat): $\tilde{\nu}$ = 3000, 2934, 2835, 2205, 1625, 1605, 1591, 1512, 1462, 1432, 1393, 1335, 1273, 1248, 1235, 1173, 1135, 1091, 1033. MS (70 eV, EI): *m*/*z* (%) = 395 (29.55) $[M + 1]^+$, 394 (100) $[M]^+$. HRMS: calcd. for $C_{27}H_{22}O_3$ $[M]^+$ 394.1569; found 394.1564.

Supporting Information (see footnote on the first page of this article): Detailed experimental procedures, analytical data, and copies of the ^{1}H and ^{13}C NMR spectra of all compounds.

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