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Bi(OTf)₃ Catalyzed Disproportionation Reaction of Cinnamyl Alcohols

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Abstract — $Bi(OTf)_3$ catalyzed disproportionation reaction of cinnamyl alcohols provides chalcones and benzyl styrenes. The use of various metal triflates is investigated herein for facile and efficient redox transformation. A plausible mechanism has been proposed.

Introduction

The classical redox disproportionation reaction of two non-enolizable arylaldehydes involving the key step of hydrogen transfer was first reported by Cannizzaro.¹ In 1906, Tishchenko developed a disproportionation route to ester products from two aldehyde molecules via an intramolecular 1,3-hydride shift.² About 20 years later, Al³⁺ catalysis of the hydride shift from the α-carbon of alcohols to carbonyls had been demonstrated by Meerwein,³ Ponndorf^{3b} and Verley^{3c} (MPV) via a six-membered transition state. The reversible nature of this MPV reduction had been realized by Oppenauer.⁴ However, different promoter mediated disproportionation reactions have been regarded, such as metal ions, bases, or Lewis acids, despite over a century of research.⁵ It therefore remained a challenge to develop an alternative catalytic system for the potential redox disproportionation. Among a number of disproportionation routes, the novel redox type of alcohols represents an efficient and versatile conversion for the formation of hydrocarbons and carbonyls.

Scheme 1. Disproportionation Reaction of Alcohols



Therefore, in comparison with the reported works on establishing the disproportionation of allylic alcohols, we found that only two promoters are documented.⁶ One is

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Ir³⁺-Sn⁴⁺ (Roy work),^{6a} and the other is Fe³⁺ (Zhou work).^{6b} Inspired by the above two catalytic systems, we explored the synthetic scope for the disproportionation of cinnamyl alcohols by the use of various metal triflates, as shown in Scheme 1. In our ongoing research interests in the synthetic applications of metal triflates,⁷ we turned the synthetic aim to the chemoselective disproportionation from cinnamyl alcohols to chalcones and benzyl styrenes.

Results and discussion

According to the previous synthetic process,⁸ chalcones 3 were easily prepared in nearly quantitative yields by NaOH-mediated aldol condensation of acetophenones 1 and arylaldehydes 2 (Ar = Ph, 4-MeOC₆H₄, 4-FC₆H₄, 3,4-CH₂O₂C₆H₃, 4-MeC₆H₄, 2-naphthyl, 3,4,5-(MeO)₃C₆H₂, 4-ClC₆H₄, 3-MeOC₆H₄, 4-PhC₆H₄, 2-furyl, 2,4-(MeO)₂C₆H₃, 3,4- $Cl_2C_6H_3$) in refluxing MeOH. With skeleton 3 in hand, we employed 3a (Ar = Ph) as the model substrate to examine the disproportionation. First, NaBH₄ mediated 1,2reduction of 3a provided 4a in an excellent conversion yield (~100%) in the co-solvent of THF and MeOH (v/v =1/1) at 0 °C for 10 min.⁹ For the purification procedure on silica gel, some impurity was isolated. Furthermore, 4a was slowly deteriorated in a CDCl₃ solution (monitored by ¹H NMR and TLC). To avoid the decomposition problems and increase the purity of 4a, we decided to use crude 4a as the starting material to treat with metal triflates, as shown in Table 1. Next, controlling the reaction conditions (rt, 5 mL of CH₂Cl₂, 1 h), Bi(OTf)₃ (2 mol %) was chosen as the optimal catalyst for the disproportionation of 4a among the various screened commercially available 13 metal triflates, which included Bi(OTf)₃, In(OTf)₃, Sn(OTf)₂, Ga(OTf)₃, Sc(OTf)₃, Fe(OTf)₃, AgOTf, Zn(OTf)₂, Yb(OTf)₃, Sm(OTf)₃, Ni(OTf)₂, Ln(OTf)₃, and Cu(OTf)₂; we found that only Bi(OTf)₃ provided better reactivity within 1 h (entry 1). For the disproportionation reaction of 4a, In(OTf)₃ Sn(OTf)₂, Ga(OTf)₃, and Sc(OTf)₃ provided the

Keywords: Disproportionation; Cannizzaro; Tishchenko; MPV reduction; Oppenauer; Metal Triflate.

transformation from **4a** to **5a** and **3a** within 1 h (entries 2-5). For an elongated time $(1 \rightarrow 20 \text{ h})$, the conversion was also achieved and similar yields of **3a** and **5a** with a ratio of 1:1 were observed. For entries 1-5, the disproportionation reaction of **4a** provided the redox products **3a** and **5a** with a ratio of 1 : 1 and no isolation of diallylic ether **6a** was detected. Although entries 1 and 5 gave comparable results for the distributed yields of **3a** and **5a** in initial screening experiments, Bi(OTf)₃ possessed a relatively inexpensive and low-toxic Lewis acid than Sc(OTf)₃.¹⁰ When the reaction was treated with Fe(OTf)₃ at rt for 1 h (entry 6), 25% of **3a** and 22% of **5a** were formed, along with a dimerized product **6a** (15%). For elongated time (20 h), the yields with similar ratios (1 : 1 : 1) were increased slightly.

 Table 1. Reaction Conditions^{a-b}

		O Ph					
Ph 3	Ph NaBH4 THF, MeOH	OH Ph 4a (~100%)	$\frac{M(OTf)_n}{CH_2Cl_2}$	3a +	Ph O Ph Ph Ph 6a		
				5a	ua ua		
entry	catalysts (mol	%) 3a (%) ^c 5a	$(\%)^{c}$	6a (%) ^c		
1	$Bi(OTf)_3(2)$	48 (46	$(5)^d$ 45	i (45) ^a	^d ND		
2	$In(OTf)_3(2)$	40 (42	$(2)^d 37$	' (36) ⁴	^d ND		
3	$Sn(OTf)_2(2)$	34 (35	$(5)^d = 36$	5 (38) ⁴	^d ND		
4	$Ga(OTf)_3(2)$	36 (37	$(1)^d = 36$	5 (40) ⁶	^d ND		
5	$Sc(OTf)_3(2)$	40 (40	$(1)^d 41$	(40)	^d ND		
6	$Fe(OTf)_3(2)$	26 (30	$())^d = 25$	i (31) ^a	d 20 (24) d		
7	AgOTf (2)	ND	N	D	65		
8	$Zn(OTf)_2(2)$	ND	N	D	70		
9	$Yb(OTf)_3(2)$	ND	N	D	72		
10	$Sm(OTf)_3(2)$	ND	N	D	70		
11	$Ni(OTf)_2(2)$	ND	N	D	72		
12	$La(OTf)_3(2)$	ND	N	D	69		
13	$Cu(OTf)_2(2)$	ND	N	D	73		
14	Bi(OTf) ₃ (10)	43	44	ŀ	ND		
15	$\operatorname{Bi}(\operatorname{OTf})_3(2)^e$	45	42	2	ND		
16	$\operatorname{Bi}(\operatorname{OTf})_3(2)^f$	35	32	2	ND		
17	$\operatorname{Bi}(\operatorname{OTf})_3(2)^g$	38	34	ļ	ND		
18	Bi(TFA) ₃ (2)	22	26	5	ND		
19	$Bi(OAc)_3(2)$	10	11		ND		
20	TfOH (2)	ND	N	D	10^{h}		
21	$\operatorname{Bi}(\operatorname{OTf})_3(2)^i$	30	28	3	ND		
22	$Bi(OTf)_3 (2)^j$	ND	N	D	ND		

^{*a*}The reactions were run on a 1.0 mmol scale with **3a**, NaBH₄ (35 mg, 1.0 mmol), THF/MeOH (3 mL/3 mL), 0 °C, 10 min. ^{*b*}The reactions were run on a resulting crude **4a**, CH₂Cl₂ (5 mL), rt, 1 h. ^cIsolated yields. ^{*d*}20 h. ^{*e*}CH₂Cl₂ (20 mL). ^{*f*}MeNO₂ (5 mL). ^{*g*}Reflux. ^{*h*}Major complex mixture was isolated. ^{*i*}TfOH (2 mol %). ^{*j*}TfOH (100 mol %).

However, with the use of AgOTf, Zn(OTf)₂, Yb(OTf)₃ Sm(OTf)₃, Ni(OTf)₂, Ln(OTf)₃, and Cu(OTf)₂, only diallylic ether 6a was isolated in entries 7-13. The plausible mechanism for the formation of **6a** should be generated by the dehydrative dimerization of 2 equivalents of 4a. The phenomenon is similar to Roy^{6a} and Zhou^{6b} reports. Furthermore, after controlling the Bi(OTf)₃ as the catalyst, the factors of equivalency, concentration and solvent were studied next. When using 10 mol % of Bi(OTf)₃, the isolated yields was similar to that of 2 mol % (entry 14). No obvious change occurred after decreasing the reaction concentration (5 mL \rightarrow 20 mL), as shown in entry 15. After changing the solvents (from CH₂Cl₂ to MeNO₂), the yields of 3a and 5a were decreased to 35% and 32%, respectively (entry 16). Compared with room temperature condition (entry 1), **3a** and **5a** provided poorer yields (38% and 34%) by elevating the temperature (rt \rightarrow reflux, entry 17). With the results in hand, we commenced the study with the other Bi(III) salts, Bi(TFA)₃ and Bi(OAc)₃, in CH_2Cl_2 (5 mL) as the medium at rt for 1 h. However, none of them provided higher yields than $Bi(OTf)_3$ (entries 18-19). By the involvement of 2 mol % TfOH (entry 20), only 6a was isolated in low yield (10%). For the combination of Bi(OTf)₃ and TfOH with the catalytic amounts (2 mol %), the result (entry 21) was better than entry 20. But, when the reaction was treated with the combination of catalytic Bi(OTf)₃(2 mol %) and stoichiometric TfOH (100 mol %), complex mixture was detected. According to the results, we envisioned that 2 mol % of Bi(OTf)₃/CH₂Cl₂ (5 mL)/rt/1 h condition would be an optimal combination for the disproportionation of **3a**.¹¹

Table 2. Synthesis of **3** and 5^{a-b}

Ar 3	Ar HF, MeOH	Ar Ar Sion yields)	(OTf) ₃ H ₂ Cl ₂ Ar	Ar 3 + 5
entry	3 , Ar =	4 , % ^{<i>c</i>}	3 , % ^d	5 , % ^{<i>d</i>}
1	3a , Ph	4a , 100	3a , 48	5a , 45
2	3b , 4-MeOC ₆ H ₄	4b , 100	3b , 46	5b , 39
3	3c , 4-FC ₆ H ₄	4c , 100	3c , 41	5c , 39
4	3d , 3,4-CH ₂ O ₂ C ₆ H ₃	4d , 100	3d , 44	5d , 38
5	3e , 4-MeC ₆ H ₄	4e , 100	3e , 42	5e , 37
6	3f, 2-naphthyl	4f , 100	3f , 43	5f , 40
7	3g , 3,4,5-(MeO) ₃ C ₆ H ₂	4g , 100	3g , 41	5g , 39
8	3h , 4-ClC ₆ H ₄	4h , 100	3h , 44	5h , 38
9	3i , 3-MeOC ₆ H ₄	4i , 100	3i , 41	5i , 38
10	3j , 4-PhC ₆ H ₄	4j , 100	3j , 42	5j , 35
11	3k , 2,4-(MeO) ₂ C ₆ H ₃	4k , 100	3k , 21	$5k,-^{e}$
12	31 , 2-furyl	41 , 100	31 , 12	51 , $-^{e}$

^{*a*}The reactions were run on a 1.0 mmol scale with **3a-l**, NaBH₄ (35 mg, 1.0 mmol), THF/MeOH (3 mL/3 mL), 0 $^{\circ}$ C, 10 min. ^{*b*}The reactions were run on a resulting crude **4a-l**,

Bi(OTf)₃ (13 mg, 2 mol%), CH₂Cl₂ (5 mL), rt, 1 h. ^{*c*}Conversion yields (by TLC analysis). ^{*d*}Isolated yields. ^{*e*}Complex mixture.

With the optimal condition (Table 1, entry 1), we further explored the substrate scope of the disproportionation reaction; the results are shown in Table 2 and entries 1-12. For the two-step route (NaBH₄ mediated reduction with 100% conversion yields and Bi(OTf)₃ catalyzed reaction), different diversified and well-tolerated aryl (Ar) groups of chalcones 3a-j (entries 1-10) performed similar distributed ratios (3/5 = 1/1) and isolated yields (3/5 =38%~48%/35%~45%), including electron-donating aryl groups 4-MeOC₆H₄, 3,4-CH₂O₂C₆H₃, 3,4,5-(MeO)₃C₆H₂, 4-ClC₆H₄, 3-MeOC₆H₄), electron-neutral aryl group (Ph, Tol, 2-naphthyl, biphenyl), and an electron-withdrawing aryl group (4-FC₆H₄). In entry 12, however, the optimal condition was inappropriate for the 2-furyl group due to 31 being provided in a 12% yield. Among the complex product mixture, only trace amounts of 5k-l were detected. From the results, we found that 2,4-(MeO)₂C₆H₃ and 2furyl group were unstable in converting into a complex mixture via intramolecular annulation under Bi(OTf)₃ catalyzed conditions. With the above results, we focused next on the two-step route of asymmetric chalcones (Ar \neq Ar^{1}), as shown in Table 3. By NaBH₄ mediated reduction (100% conversion yields) and Bi(OTf)₃ catalyzed reaction protocol, chalcones **3m-r** (Ar or $Ar^1 = Ph$, 4-MeC₆H₄, 4- FC_6H_4 , 4-MeOC₆H₄) obeyed the above-mentioned reaction behaviour to afford sole 3m-r (38~42%) and two pairs of inseparated 5m-o and 5m'-o' (35~40%, entries 1-6). Interestingly, 3' was not detected. On the basis of NMR spectrums, the ratios of 5m-o and 5m'-o' were determined as $1/1 \sim 4/1$. When Ar¹ was changed to bicyclic 2-naphthyl (3s), 41% of 3s was isolated and the ratios of 5s/5s' (33%)

Table 3. Synthesis of 3 and 5-5^{*a-b*}

was maintained as 3/1 (entry 7). For tricyclic 9-anthrancyl group with the bulky steric hindrance, only 22 % of 3t and 20% of 5t/5t' (3/1) were observed (entry 8). For the twostep route of asymmetric oxygenated chalcones (3u-v), the afforded yields of 3u and 5u/5u' (3/1) were similar to 3v and 5v/5v' (1/1) (entries 9-10). According to the above experimental results (Tables 1-3), a possible two-step reaction mechanism is shown in Scheme 2. The initial reduction of 3 yields 4 by NaBH₄. By the $Bi(OTf)_3$ catalyzed complexation of two molecules of 4. A1/A2 and B1/B2 could be generated via paths a and b. On basis of the eight-membered conformation (path a), 3 and 5' are provided via an intramolecular 1,8-hydride shift (pink circle) on A1 or A2 and the removal of Bi(O)OTf and TfOH (2 equiv). In path b, intramolecular 1,6-hydride shift (green circle) could trigger the transformation from **B1** or **B2** to **3** and **5** and release the Bi(O)OTf and TfOH via the six-membered conformation. Subsequently, Bi(OTf)₃ is regenerated by complexation of Bi(O)OTf and TfOH. From the experimental results, we envisioned that the present plausible mechanism provided the reasonable explanation for no generation of 3' via the intramolecular hydride shift of four plausible intermediates A1/A2 and B1/B2. Among the inseparated mixture of 5 and 5', the isolated ratio of 5 was better than 5' due to B1/B2 with a six-membered conformation being orientated and formed more easily than A1/A2 with an eight-membered conformation. Following a successful two-step protocol, we further explored the conversion of other substrate scopes; the results are shown in Scheme 3. By the above protocol, enedione 7 was transformed into 2,5-diphenyltetrahydrofuran 8 in a 71% yield via the 1,4- and 1,2-reduction followed by Bi(OTf)3catalyzed ring-closure. However, when dienone 9 was treated by a two-step strategy, a complex mixture was afforded.

Ar 3	$Ar^{1} \xrightarrow{\text{NaBH}_{4}} \begin{bmatrix} OH \\ Ar & Ar^{1} \end{bmatrix}$ (100% conversion yiel)	Bi(OTf) ₃ CH ₂ Cl ₂ ds) 3'	O Ar 3 Ar 3 Ar (not detected)	$\begin{array}{c c} Ar & Ar^{1} \\ \hline 5 \\ and + \\ Ar^{1} & Ar \\ \hline 5' \end{array}$
entry	3 , $Ar =, Ar^1 =$	4 , % ^{<i>c</i>}	3 , % ^d	5 + 5' , %, ^{<i>d</i>} ratio
1	$3\mathbf{m}$, Ph, 4-MeC ₆ H ₄	4m , 100	3m , 38	5m/5m' , 38, 4:1
2	3n , Ph, 4-FC ₆ H ₄	4n , 100	3n , 40	5n/5n' , 40, 1:1
3	30, Ph, 4-MeOC ₆ H ₄	40 , 100	30 , 42	50/50' , 40, 3:1
4	$3\mathbf{p}$, 4-MeC ₆ H ₄ , Ph	4p , 100	3p , 41	5m/5m' , 38, 5:2
5	3q , 4-FC ₆ H ₄ , Ph	4q , 100	3q , 39	5n/5n' , 36, 1:1
6	$3\mathbf{r}$, 4-MeOC ₆ H ₄ , Ph,	4r , 100	3r , 40	50/50' , 35, 3:1
7	3s , Ph, 2-naphthyl	4s , 100	3s , 41	5s/5s' , 33, 3:1
8	3t , Ph, 9-anthracyl	4t , 100	3t , 22	5t/5t' , 20, 3:1
9	3u , Ph, 3,4-CH ₂ O ₂ C ₆ H ₃	4u , 100	3u , 30	5u/5u' , 32, 3:1
10	3v , Ph, 3,4,5-(MeO) ₃ C ₆ H ₂	4v , 100	3v , 33	5v/5v' , 33, 1:1

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^aThe reactions were run on a 1.0 mmol scale with **3m-v**, NaBH₄ (35 mg, 1.0 mmol), THF/MeOH (3 mL/3 mL), 0 °C, 10 min. ^bThe reactions were run on a resulting crude **4m-v**, Bi(OTf)₃ (13 mg, 2 mol%), CH₂Cl₂ (5 mL), rt, 1 h. ^cConversion yields (by TLC analysis). ^dIsolated yields.

Scheme 2. Proposed Mechanism



Scheme 3. Reactions of 7 and 9



Scheme 4. Synthesis of 11



Scheme 5. Control Deuterated Experiments



To explore the two-step synthetic application, the indene skeleton was examined (Scheme 4). By the involvement of veratrole,¹² **10** with the 1,1,3-triarylpropene skeleton was isolated in a 79% yield via reduction and Friedel-Crafts alkylation. Furthermore, by the oxidation of **10** with

*m*CPBA and intramolecular annulation of the resulting epoxide with Bi(OTf)₃, **11** was generated in a 91% yield. The present (3+2) annulation strategy provided a new synthetic method for the formation of 1,3-diarylindene skeleton. In order to find further insight for the Bi(OTf)₃ mediated disproportionation process, **4a-1** (prepared from NaBD₄ mediated reduction of **3a**) was chosen as the deuterated model substrate (Scheme 5). When the reaction of in-situ generated **4a-1** was treated with Bi(OTf)₃, **3a** and a mixture of di-deuterated **5w** and **5x** (ratio, 4:5) were obtained in 41% and 34% yields, respectively. The expected results of the redox procedure demonstrated that hydride of benzylic position on **I** and **II** triggered the formation of two structural frameworks **3** and **5** via intramolecular S_N2²- or S_N2-type deuterium transfer.

Conclusion

In summary, we have developed a $Bi(OTf)_3$ catalyzed disproportionation from cinnamyl alcohols to chalcones and benzyl styrenes in CH_2Cl_2 at rt. The use of various metal triflates was well-investigated for a facile conversion. A plausible mechanism has been proposed. Further investigations regarding the synthetic application of metal triflates are on-going in our laboratory.

Experimental section

General. All reagents and solvents were obtained from commercial sources and used without further purification. Reactions were routinely carried out under an atmosphere of air with magnetic stirring. Products in organic solvents were dried with anhydrous magnesium sulfate before concentration in vacuo. Melting points were determined with a SMP3 melting apparatus. ¹H and ¹³C NMR spectra were recorded on a Varian INOVA-400 spectrometer operating at 400 and at 100 MHz, respectively. High resolution mass spectra (HRMS) were measured with a mass spectrometer Finnigan/Thermo Quest MAT 95XL.

A representative synthetic procedure of skeletons 3 and 5 is as follows: NaBH₄ (68 mg, 2.0 mmol) was added to a solution of chalcones 3 (1.0 mmol) in the co-solvent of MeOH (3 mL) and THF (3 mL) at rt. The reaction mixture was stirred at rt for 30 min and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Without further purification, Bi(OTf)₃ (13 mg, 0.02 mmol) was added to a solution of resulting crude 4 in CH₂Cl₂ (5 mL) at rt. The reaction mixture was stirred at rt for 1 h and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Purification on silica gel (hexanes/EtOAc = $10/1 \sim 6/1$) afforded skeletons **3** and **5**.

1,3-Diphenylpropenone (*3a*).^{13a} Yield = 48% (100 mg); Colorless solid; mp = 38-40 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for $C_{15}H_{13}O$ 209.0961, found 209.0966; ¹H NMR (400 MHz, CDCl₃): δ 8.05-8.02 (m, 2H), 7.83 (d, *J* = 15.6 Hz, 1H), 7.67-7.61 (m, 2H), 7.60-7.49 (m, 4H), 7.44-7.40 (m, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 190.44, 144.75, 138.11, 134.79, 132.72, 130.47, 128.88 (2x), 128.55 (2x), 128.43 (2x), 128.38 (2x), 121.99.

1,3-Bis-(4-methoxyphenyl)propenone (**3***b*). Yield = 46% (123 mg); Colorless solid; mp = 80-82 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₁₇H₁₇O₃ 269.1178, found 269.1174; ¹H NMR (400 MHz, CDCl₃): δ 8.02 (d, *J* = 8.8 Hz, 2H), 7.76 (d, *J* = 15.2 Hz, 1H), 7.57 (d, *J* = 8.8 Hz, 2H), 7.41 (d, *J* = 15.2 Hz, 1H), 6.95 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 3.85 (s, 3H), 3.82 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 188.59, 163.16, 161.40, 143.66, 131.22, 130.57 (2x), 129.99 (2x), 127.68, 119.41, 114.27 (2x), 113.67 (2x), 55.34, 55.26.

1,3-Bis-(4-fluorophenyl)propenone (*3c*).^{13b} Yield = 41% (100 mg); Colorless solid; mp = 105-107 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for $C_{15}H_{11}F_{2}O$ 245.0778, found 245.0778; ¹H NMR (400 MHz, CDCl₃): δ 8.06-8.02 (m, 2H), 7.77 (d, *J* = 15.6 Hz, 1H), 7.64-7.60 (m, 2H), 7.42 (d, *J* = 15.6 Hz, 1H), 7.19-7.08 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 188.51, 165.59 (d, *J* = 252.5 Hz), 164.07 (d, *J* = 250.9 Hz), 143.64, 134.41 (d, *J* = 3.0 Hz), 131.02 (d, *J* = 9.1 Hz, 2x), 130.75 (d, *J* = 3.1 Hz), 130.33 (d, *J* = 8.4 Hz, 2x), 121.25, 116.11 (d, *J* = 22.0 Hz, 2x), 115.72 (d, *J* = 22.0 Hz, 2x).

1,3-Bis-benzo[*1,3*]*dioxol-5-ylpropenone* (*3d*). Yield = 44% (130 mg); Colorless solid; mp = 174-176 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for $C_{17}H_{13}O_5$ 297.0763, found 297.0766; ¹H NMR (400 MHz, CDCl₃): δ 7.71 (d, *J* = 15.6 Hz, 1H), 7.62 (dd, *J* = 1.6, 8.0 Hz, 1H), 7.51 (d, *J* = 1.6 Hz, 1H), 7.31 (d, *J* = 15.6 Hz, 1H), 7.14 (d, *J* = 1.6 Hz, 1H), 7.10 (dd, *J* = 1.6, 8.0 Hz, 1H), 6.88 (d, *J* = 8.0 Hz, 1H), 6.83 (d, *J* = 8.0 Hz, 1H), 6.05 (s, 2H), 6.01 (s, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 188.07, 151.55, 149.76, 148.36, 148.23, 144.03, 133.13, 129.43, 125.01, 124.46, 119.71, 108.61, 108.37, 107.85, 106.59, 101.80, 101.58.

1,3-Di-p-tolylpropenone (*3e*).^{13c} Yield = 42% (99 mg); Colorless solid; mp = 127-129 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for $C_{17}H_{17}O$ 237.1280, found 237.1285; ¹H NMR (400 MHz, CDCl₃): δ 7.95 (d, J = 8.4 Hz, 2H), 7.80 (d, J = 15.2 Hz, 1H), 7.53 (d, J = 8.4 Hz, 2H), 7.50 (d, J = 15.2 Hz, 1H), 7.29 (d, J = 8.0 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 2.42 (s, 3H), 2.37 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 189.80, 144.25, 143.31, 140.74, 135.60, 132.09, 129.52 (2x), 129.13 (2x), 128.45 (2x), 128.28 (2x), 120.85, 21.48, 21.35. *1,3-Dinaphthalen-2-ylpropenone* (**3***f*).^{13d} Yield = 43% (132 mg); Colorless solid; mp = 197-198 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₂₃H₁₇O 309.1280, found 309.1283; ¹H NMR (400 MHz, CDCl₃): δ 8.59 (s, 1H), 8.15 (dd, *J* = 1.6, 8.8 Hz, 1H), 8.07-7.79 (m, 10 H), 7.65-7.52 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 190.27, 144.87, 135.66, 135.51, 134.41, 133.41, 132.61, 132.47, 130.67, 129.93, 129.54, 128.75, 128.66, 128.59, 128.38, 127.84, 127.81, 127.38, 126.79 (2x), 124.54, 123.74, 122.23.

1,3-Bis-(3,4,5-trimethoxyphenyl)propenone (3g). Yield = 41% (159 mg); Colorless solid; mp = 130-132 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M^++1) calcd for $C_{21}H_{25}O_7$ 389.1600, found 389.1602; ¹H NMR (400 MHz, CDCl₃): δ 7.69 (dd, J = 1.6, 15.6 Hz, 1H), 7.32 (dd, J = 0.8, 15.6 Hz, 1H), 7.24 (d, J = 1.2 Hz, 2H), 6.84 (d, J = 1.6 Hz, 2H), 3.93 (s, 3H), 3.92 (s, 3H), 3.91 (s, 3H), 3.90 (br s, 6H), 3.88 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 189.35, 153.43 (2x), 153.09 (2x), 144.88, 142.52, 140.49, 133.52, 130.30, 121.25, 106.24 (2x), 105.75 (2x), 60.91, 60.88, 56.41 (2x), 56.21 (2x).

1,3-Bis-(4-chlorophenyl)propenone (*3h*).^{13b} Yield = 44% (121 mg); Colorless solid; mp = 156-158 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for $C_{15}H_{11}Cl_2O$ 277.0187, found 277.0186; ¹H NMR (400 MHz, CDCl₃): δ 7.94 (d, *J* = 8.4 Hz, 2H), 7.74 (d, *J* = 15.6 Hz, 1H), 7.55 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.4 Hz, 2H), 7.43 (d, *J* = 15.6 Hz, 1H), 7.37 (d, *J* = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 188.70, 143.68, 139.30, 136.57, 136.24, 133.13, 129.82 (2x), 129.57 (2x), 129.21 (2x), 128.91 (2x), 121.79.

1,3-Bis-(3-methoxyphenyl)propenone (*3i*). Yield = 41% (110 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for $C_{17}H_{17}O_3$ 269.1178, found 269.1180; ¹H NMR (400 MHz, CDCl₃): δ 7.76 (d, *J* = 16.0 Hz, 1H), 7.60-7.58 (m, 1H), 7.54-7.53 (m, 1H), 7.48 (d, *J* = 16.0 Hz, 1H), 7.39 (t, *J* = 8.0 Hz, 1H), 7.32 (t, *J* = 8.0 Hz, 1H), 7.22 (d, *J* = 8.0 Hz, 1H), 7.15-7.10 (m, 2H), 6.96-6.93 (m, 1H), 3.86 (s, 3H), 3.83 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 190.05, 159.85, 159.80, 144.62, 139.45, 136.15, 129.83, 129.46, 122.28, 121.00, 120.94, 119.15, 116.21, 113.36, 112.82, 55.34, 55.21.

1,3-Bis-biphenyl-4-ylpropenone (**3***j*).^{13e} Yield = 42% (151 mg); Colorless solid; mp = 199-201 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₂₇H₂₁O 361.1593, found 361.1590; ¹H NMR (400 MHz, CDCl₃): δ 8.14 (d, *J* = 8.0 Hz, 2H), 7.91 (d, *J* = 15.6 Hz, 1H), 7.75 (d, *J* = 8.0 Hz, 4H), 7.69-7.61 (m, 7H), 7.52-7.38 (m, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 189.83, 145.50, 144.26, 143.28, 140.11, 139.92, 136.95, 133.88, 129.10 (2x), 128.97 (2x), 128.93 (2x), 128.90 (2x), 128.18, 127.88, 127.58 (2x), 127.27 (4x), 127.03 (2x), 121.82.

1,3-Bis-(2,4-dimethoxyphenyl)propenone (**3***k*). Yield = 21% (69 mg); Colorless solid; mp = 126-128 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M^++1) calcd for $C_{19}H_{21}O_5$ 329.1389, found 329.1385; ¹H

5

NMR (400 MHz, CDCl₃): δ 7.93 (d, *J* = 15.6 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 1H), 7.52 (d, *J* = 8.8 Hz, 1H), 7.45 (d, *J* = 16.0 Hz, 1H), 6.55-6.43 (m, 4H), 3.87 (s, 3H), 3.85 (s, 3H), 3.84 (s, 3H), 3.82 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 191.17, 163.63, 162.55, 160.05, 160.01, 137.80, 132.50, 130.11, 125.33, 122.71, 117.46, 105.28, 104.95, 98.60, 98.27, 55.59, 55.39, 55.37, 55.34.

1,3-Difuran-2-ylpropenone (*31*).^{13f} Yield = 12% (23 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₁₁H₉O₃ 189.0552, found 189.0553; ¹H NMR (400 MHz, CDCl₃): δ 7.61 (d, *J* = 1.6 Hz, 1H), 7.59 (d, *J* = 16.0 Hz, 1H), 7.49 (d, *J* = 1.6 Hz, 1H), 7.30-7.27 (m, 2H), 6.68 (d, *J* = 3.2 Hz, 1H), 6.55-6.53 (m, 1H), 6.47-6.46 (m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 177.58, 153.58, 151.43, 146.44, 144.93, 129.71, 118.74, 117.29, 116.23, 112.58, 112.36.

1-Phenyl-3-p-tolylpropenone (**3m**).^{13g} Yield = 38% (84 mg); Colorless solid; mp = 88-90 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₁₆H₁₅O 223.1123, found 223.1123; ¹H NMR (400 MHz, CDCl₃): δ 8.03-8.01 (m, 2H), 7.80 (d, *J* = 15.2 Hz, 1H), 7.60-7.48 (m, 6H), 7.23 (d, *J* = 7.6 Hz, 2H), 2.40 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 190.63, 144.91, 141.06, 138.35, 132.62, 132.14, 129.68 (2x), 128.55 (2x), 128.43 (4x), 121.10, 21.49.

3-(4-Fluorophenyl)-1-phenylpropenone (3n).^{13b} Yield = 40% (90 mg); Colorless solid; mp = 79-81 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₁₅H₁₂FO 227.0872, found 227.0874; ¹H NMR (400 MHz, CDCl₃): δ 8.01 (d, *J* = 8.4 Hz, 2H), 7.77 (d, *J* = 15.6 Hz, 1H), 7.66-7.60 (m, 2H), 7.59-7.57 (m, 1H), 7.51 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 15.6 Hz, 1H), 7.13-7.08 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 190.24, 164.02 (d, *J* = 250.9 Hz), 143.44, 138.10, 132.79, 131.12 (d, *J* = 3.1 Hz), 130.30 (d, *J* = 9.1 Hz, 2x), 128.61 (2x), 128.43 (2x), 121.76 (d, *J* = 2.3 Hz), 116.08 (d, *J* = 22.0 Hz, 2x).

3-(4-Methoxyphenyl)-1-phenylpropenone (30).^{13g} Yield = 42% (100 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₁₆H₁₅O₂ 239.1072, found 239.1076; ¹H NMR (400 MHz, CDCl₃): δ 7.99 (d, J = 8.4 Hz, 2H), 7.76 (d, J = 15.6 Hz, 1H), 7.54 (d, J = 8.4 Hz, 2H), 7.45 (d, J = 7.6 Hz, 2H), 7.39 (d, J = 15.6 Hz, 2H), 6.87 (d, J = 8.8 Hz, 2H), 3.76 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 190.07, 161.42, 144.34, 138.20, 132.29, 129.98 (2x), 128.29 (2x), 128.14 (2x), 127.27, 119.38, 114.15 (2x), 55.06.

3-Phenyl-1-p-tolylpropenone (**3p**).^{13g} Yield = 41% (91 mg); Colorless oil; HRMS (ESI, M^++1) calcd for C₁₆H₁₅O 223.1123, found 223.1122; ¹H NMR (400 MHz, CDCl₃): δ 7.95 (d, *J* = 8.0 Hz, 2H), 7.82 (d, *J* = 15.6 Hz, 1H), 7.63-7.61 (m, 2H), 7.54 (d, *J* = 15.6 Hz, 1H), 7.40-7.38 (m, 3H), 7.28 (d, *J* = 8.0 Hz, 2H), 2.40 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 189.63, 144.09, 143.38, 135.40, 134.76, 130.18, 129.11 (2x), 128.70 (2x), 128.44 (2x), 128.19 (2x), 121.82, 21.41. *l*-(4-*Fluorophenyl*)-3-*phenylpropenone* (3q).^{13g} Yield = 39% (88 mg); Colorless solid; mp = 75-77 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₁₅H₁₂FO 227.0872, found 227.0871; ¹H NMR (400 MHz, CDCl₃): δ 8.07-8.02 (m, 2H), 7.80 (d, *J* = 15.6 Hz, 1H), 7.64-7.60 (m, 2H), 7.49 (d, *J* = 15.6 Hz, 1H), 7.42-7.37 (m, 3H), 7.18-7.12 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 188.58, 165.46 (d, *J* = 252.4 Hz), 144.86, 134.64, 134.40 (d, *J* = 3.0 Hz), 130.97 (d, *J* = 9.1 Hz, 2x), 130.51, 128.85 (2x), 128.35 (2x), 121.43, 115.58 (d, *J* = 21.9 Hz, 2x).

1-(4-Methoxyphenyl)-3-phenylpropenone (**3r**).^{13g} Yield = 40% (95 mg); Colorless solid; mp = 105-107 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₁₆H₁₅O₂ 239.1072, found 239.1074; ¹H NMR (400 MHz, CDCl₃): δ 8.04 (d, *J* = 9.2 Hz, 2H), 7.80 (d, *J* = 16.0 Hz, 1H), 7.65-7.61 (m, 2H), 7.54 (d, *J* = 16.0 Hz, 1H), 7.43-7.38 (m, 3H), 6.97 (d, *J* = 8.8 Hz, 2H), 3.86 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 188.53, 163.33, 143.79, 134.98, 130.98, 130.70 (2x), 130.21, 128.81 (2x), 128.24 (2x), 121.78, 113.74 (2x), 55.35.

3-Naphthalen-2-yl-1-phenylpropenone (3s).^{13h} Yield = 41% (106 mg); Colorless solid; mp = 155-157 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₁₉H₁₅O 259.1123, found 259.1130; ¹H NMR (400 MHz, CDCl₃): δ 8.09-8.06 (m, 2H), 7.98 (d, *J* = 15.6 Hz, 2H), 7.88-7.82 (m, 2H), 7.80 (s, 1H), 7.96 (s, 1H), 7.64 (d, *J* = 15.6 Hz, 1H), 7.61-7.58 (m, 1H), 7.54-7.50 (m, 4H); ¹³C NMR (100 MHz, CDCl₃): δ 190.37, 144.80 (2x), 138.20, 134.29, 133.26, 132.68, 132.28, 130.54, 128.63, 128.55 (2x), 128.44 (2x), 127.71, 127.27, 126.67, 123.59, 122.10.

3-Anthracen-9-yl-1-phenylpropenone (**3t**). Yield = 22% (68 mg); Colorless solid; mp = 132-134 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₂₃H₁₇O 309.1280, found 309.1286; ¹H NMR (400 MHz, CDCl₃): δ 8.30 (s, 1H), 8.11-8.09 (m, 2H), 7.94-7.88 (m, 3H), 7.61-7.58 (m, 2H), 7.47-7.39 (m, 5H), 7.27-7.23 (m, 1H), 7.09-7.05 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 192.56, 139.24 (2x), 137.51, 132.33 (2x), 131.01 (2x), 130.75 (2x), 128.70 (2x), 128.04 (2x), 127.75 (2x), 127.18, 125.80 (2x), 125.41 (2x), 125.04 (2x).

3-Benzo[*1*,*3*]*dioxol-5-yl-1-phenylpropenone* (*3u*).^{13g} Yield = 30% (76 mg); Colorless solid; mp = 106-108 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₁₆H₁₃O₃ 253.0865, found 253.0866; ¹H NMR (400 MHz, CDCl₃): δ 8.02-7.99 (m, 2H), 7.73 (d, *J* = 15.6 Hz, 1H), 7.59-7.55 (m, 1H), 7.52-7.47 (m, 2H), 7.36 (d, *J* = 15.6 Hz, 1H), 7.16 (d, *J* = 1.2 Hz, 1H), 7.12 (dd, *J* = 1.6, 8.0 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 6.02 (s, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 190.36, 149.89, 148.39, 144.63, 138.38, 132.59, 129.34, 128.54 (2x), 128.37 (2x), 125.17, 120.10, 108.63, 106.63, 101.59.

1-Phenyl-3-(3,4,5-trimethoxyphenyl)propenone (3v). Yield = 33% (98 mg); Colorless solid; mp = 141-142 °C (recrystallized from hexanes and EtOAc); HRMS (ESI,

M⁺+1) calcd for C₁₈H₁₉O₄ 299.1283, found 299.1284; ¹H NMR (400 MHz, CDCl₃): δ 7.99-7.97 (m, 2H), 7.68 (d, J = 15.6 Hz, 1H), 7.56-7.51 (m, 1H), 7.48-7.43 (m, 2H), 7.39 (d, J = 15.6 Hz, 1H), 6.82 (s, 2H), 3.87 (s, 6H), 3.86 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 190.24, 153.25, 144.78 (2x), 140.21, 138.05, 132.53, 130.15, 128.41 (2x), 128.27 (2x), 121.19, 105.48 (2x), 60.76, 55.99 (2x).

Cinnamylbenzene (*5a*).^{14a} Yield = 45% (87 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₁₅H₁₅ 195.1174, found 195.1180; ¹H NMR (400 MHz, CDCl₃): δ 7.40-7.20 (m, 10H), 6.49 (d, *J* = 16.0 Hz, 1H), 6.38 (dt, *J* = 3.2, 16.0 Hz, 1H), 3.58 (d, *J* = 6.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 140.14, 137.45, 131.04, 129.20, 128.65 (2x), 128.47 (4x), 127.08, 126.16, 126.10 (2x), 39.33.

(E)-1-Methoxy-4-(3-(4-methoxyphenyl)allyl)benzene

(5b).^{14a} Yield = 39% (99 mg); Colorless oil; HRMS (ESI, M^++1) calcd for $C_{17}H_{19}O_2$ 255.1385, found 255.1386; ¹H NMR (400 MHz, CDCl₃): δ 7.31 (d, J = 8.8 Hz, 2H), 7.17 (d, J = 8.4 Hz, 2H), 6.89-6.83 (m, 4H), 6.40 (d, J = 15.6 Hz, 1H), 6.22 (dt, J = 6.8, 15.6 Hz, 1H), 3.81 (br s, 6H), 3.49 (d, J = 6.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 158.79, 158.00, 132.45, 130.35, 130.08, 129.53 (2x), 127.48, 127.17 (2x), 113.89 (2x), 113.85 (2x), 55.24 (2x), 38.39.

(*E*)-1-Fluoro-4-(3-(4-fluorophenyl)allyl)benzene (5c).^{14c} Yield = 39% (90 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₁₅H₁₃F₂ 231.0985, found 231.0986; ¹H NMR (400 MHz, CDCl₃): δ 7.36-7.31 (m, 2H), 7.23-7.19 (m, 2H), 7.06-6.98 (m, 4H), 6.42 (d, *J* = 15.6 Hz, 1H), 6.26 (dt, *J* = 6.8, 15.6 Hz, 1H), 3.53 (d, *J* = 6.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 162.08 (d, *J* = 244.1 Hz), 161.51 (d, *J* = 243.3 Hz), 135.61 (d, *J* = 3.0 Hz), 133.48 (d, *J* = 3.8 Hz), 130.00, 129.99 (d, *J* = 8.4 Hz, 2x), 128.76, 128.74, 127.76 (d, *J* = 7.5 Hz), 115.36 (d, *J* = 21.2 Hz, 2x), 115.23 (d, *J* = 21.2 Hz, 2x), 38.37.

(E)-1,2-Methylenedioxy-4-(3-(3,4-

methylenedioxyphenyl)allyl)benzene (5d). Yield = 38% (107 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for $C_{17}H_{15}O_4$ 283.0970, found 283.0972; ¹H NMR (400 MHz, CDCl₃): δ 6.90 (d, *J* = 1.6 Hz, 1H), 6.80-6.66 (m, 5H), 6.34 (d, *J* = 16.0 Hz, 1H), 6.14 (dt, *J* = 7.2, 16.0 Hz, 1H), 5.93 (s, 2H), 5.92 (s, 2H), 3.43 (d, *J* = 6.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 147.95, 147.68, 146.82, 145.89, 138.82, 134.07, 131.96, 130.51, 127.62, 121.33, 120.53, 109.13, 108.21, 105.54, 100.94, 100.81, 38.89.

(*E*)-4-*Methyl*-1-(3-(4-*methylphenyl*)*allyl*)*benzene* (5*e*).^{14c} Yield = 37% (82 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₁₇H₁₉ 223.1487, found 223.1489; ¹H NMR (400 MHz, CDCl₃): δ 7.47 (d, *J* = 8.0 Hz, 2H), 7.37-7.33 (m, 4H), 7.31 (d, *J* = 8.0 Hz, 2H), 6.64 (d, *J* = 16.0 Hz, 1H), 6.51 (dt, *J* = 6.8, 16.0 Hz, 1H), 3.71 (d, *J* = 6.4 Hz, 2H), 2.55 (s, 3H), 2.53 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 137.16, 136.60, 135.45, 134.73, 130.67, 129.11 (2x), 129.09 (2x), 128.49 (2x), 128.37, 125.97 (2x), 38.86, 21.06, 20.94. (*E*)-2-(*3*-(2-*Naphthyl*)*allyl*)*naphthalene* (*5f*). Yield = 40% (118 mg); Colorless solid; mp = 134-136 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₂₃H₁₉ 295.1487, found 295.1488; ¹H NMR (400 MHz, CDCl₃): δ 7.86-7.78 (m, 6H), 7.74 (br s, 2H), 7.63 (dd, *J* = 1.6, 8.8 Hz, 1H), 7.52-7.42 (m, 5H), 6.69 (d, *J* = 15.6 Hz, 1H), 6.59 (dt, *J* = 6.4, 15.6 Hz, 1H), 3.79 (d, *J* = 6.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 137.60, 134.91, 133.68, 133.64, 132.78, 132.19, 131.39, 129.55, 128.09, 128.07, 127.86, 127.64, 127.61, 127.50, 127.47, 126.79, 126.16, 125.99, 125.77, 125.60, 125.34, 123.57, 39.56.

(E)-1,2,3-Trimethoxy-5-(3-(3,4,5-

trimethoxyphenyl)allyl)benzene (**5g**).^{14c} Yield = 39% (146 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₂₁H₂₇O₆ 375.1808, found 375.1809; ¹H NMR (400 MHz, CDCl₃): δ 6.59 (br s, 2H), 6.46 (br s, 2H), 6.40-6.35 (m, 1H), 6.28-6.21 (m, 1H), 3.86 (br s, 6H), 3.85 (br s, 6H), 3.83 (br s, 6H), 3.48 (d, *J* = 6.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 153.29 (2x), 153.25 (2x), 135.76, 133.09, 130.96 (2x), 128.50 (2x), 105.67 (2x), 103.27 (2x), 60.85, 60.81, 56.09 (2x), 56.06 (2x), 39.61.

(*E*)-1-Chloro-4-(3-(4-chlorophenyl)allyl)benzene (5h).^{14c} Yield = 38% (100 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₁₅H₁₃Cl₂ 263.0394, found 263.0392; ¹H NMR (400 MHz, CDCl₃): δ 7.30 (d, *J* = 8.4 Hz, 2H), 7.28 (br s, 4H), 7.17 (d, *J* = 8.4 Hz, 2H), 6.42-6.37 (m, 1H), 6.30 (dt, *J* = 6.8, 15.6 Hz, 1H), 3.52 (d, *J* = 6.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 138.24, 135.71, 132.80, 132.03, 130.21, 129.97 (2x), 129.29, 128.64 (2x), 128.59 (2x), 127.30 (2x), 38.52.

(E)-1-Methoxy-3-(3-(3-methoxyphenyl)allyl)benzene

(5i).^{14d} Yield = 38% (97 mg); Colorless oil; HRMS (ESI, M^++1) calcd for $C_{17}H_{19}O_2$ 255.1385, found 255.1380; ¹H NMR (400 MHz, CDCl₃): δ 7.25 (d, J = 8.0 Hz, 1H), 7.21 (d, J = 8.0 Hz, 1H), 6.98-6.77 (m, 6H), 6.47-6.32 (m, 2H), 3.81 (s, 6H), 3.53 (d, J = 6.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 159.77, 141.71, 138.92, 131.04, 129.43 (2x), 129.34, 121.05, 118.83, 114.39, 112.86 (2x), 111.54, 111.36, 55.17, 55.15, 39.31.

(*E*)-1-Phenyl-4-(3-(4-biphenyl)allyl)benzene (5j).^{14c} Yield = 35% (121 mg); Colorless solid; mp = 141-143 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₂₇H₂₃ 347.1800, found 347.1802; ¹H NMR (400 MHz, CDCl₃): δ 7.64-7.57 (m, 8H), 7.49-7.45 (m, 6H), 7.38-7.35 (m, 4H), 6.62-6.43 (m, 2H), 3.65 (d, *J* = 6.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 141.00, 140.77, 139.90, 139.22, 136.50, 131.84, 130.76, 129.21, 129.09 (2x), 128.74 (2x), 128.72 (2x), 127.24 (2x), 127.19 (2x), 127.08, 127.02 (2x), 126.88 (2x), 126.74, 126.55 (2x), 39.02.

(*E*)-4-Cinnamyl-1-methylbenzene (**5m**) and (*E*)-3-(4methylphenyl)allyl)benzene (**5m**').^{14e} Two isomers, ratio = 4:1; Yield = 38% (79 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₁₆H₁₇ 209.1330, found 209.1330; ¹H NMR (400 MHz, CDCl₃): δ 7.62-7.35 (m, 9H), 6.73-6.53 (m, 2H), 3.80-3.76 (m, 2H), 2.59 (s, 3H). (*E*)-4-Cinnamyl-1-fluorobenzene (**5n**) and (*E*)-3-(4-fluorophenyl)allyl)benzene (**5n**').^{14f} Two isomers, ratio = 1:1; Yield = 40% (85 mg); Colorless oil; HRMS (ESI, M^++1) calcd for $C_{15}H_{14}F$ 213.1079, found 213.1082; ¹H NMR (400 MHz, CDCl₃): δ 7.45-7.23 (m, 7H), 7.10-7.02 (m, 2H), 6.54-6.31 (m, 3H), 3.62-3.57 (m, 2H).

(*E*)-4-Cinnamyl-1-methoxybenzene (**5o**) and (*E*)-3-(4methoxyphenyl)allyl)benzene (**5o**').^{14e} Two isomers, ratio = 3:1; Yield = 40% (90 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₁₆H₁₇O 225.1280, found 225.1275; ¹H NMR (400 MHz, CDCl₃): δ 7.39-7.17 (m, 7H), 6.90-6.84 (m, 2H), 6.48-6.21 (m, 2H), 3.82 (s, 3H), 3.58-3.51 (m, 2H).

(*E*)-2-*Cinnamylnaphthalene* (5*s*) and (*E*)-2-(3phenyl)allyl)naphthalene (5*s*').^{14e} Two isomers, ratio = 1:1; Yield = 33% (81 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for $C_{19}H_{17}$ 245.1330, found 245.1332; ¹H NMR (400 MHz, CDCl₃): δ 7.87-7.61 (m, 4H), 7.52-7.23 (m, 8H), 6.68-6.44 (m, 2H), 3.76-3.64 (m, 2H).

(*E*)-9-*Cinnamylanthracene* (5*t*) and (*E*)-9-(3phenyl)allyl)anthracene (5*t*'). Two isomers, ratio = 1:1; Yield = 20% (59 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₂₃H₁₉ 295.1487, found 295.1488; ¹H NMR (400 MHz, CDCl₃): δ 8.31 (s, 1H), 7.94-7.90 (m, 4H), 7.58-7.52 (m, 1H), 7.45-7.28 (m, 7H), 7.15 (d, *J* = 16.0 Hz, 1H), 7.07-7.04 (m, 2H), 6.57-6.51 (m, 1H), 4.42 (d, *J* = 8.0 Hz, 1H).

(*E*)-4-*Cinnamyl*-1,2-*methylenedioxybenzene* (**5***u*) and (*E*)-3-(3,4-*methylenedioxyphenyl*)allyl)benzene (**5***u*').^{14g} Two isomers, ratio = 3:1; Yield = 32% (76 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₁₆H₁₅O₂ 239.1072, found 239.1077; ¹H NMR (400 MHz, CDCl₃): δ 7.39-7.20 (m, 5H), 6.79-6.70 (m, 3H), 6.47-6.29 (m, 2H), 5.94 (s, 2H), 3.48 (d, *J* = 6.4 Hz, 2H).

(*E*)-5-*Cinnamyl*-1,2,3*trimethoxybenzene* (**5***v*) and (*E*)-3-(3,4,5-*trimethyloxyphenyl*)allyl)benzene (**5***v*').^{14h} Two isomers, ratio = 1:1; Yield = 33% (94 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₁₈H₂₁O₃ 285.1490, found 285.1493; ¹H NMR (400 MHz, CDCl₃): δ 7.40 (d, *J* = 8.0 Hz, 2H), 7.34-7.31 (m, 2H), 7.25-7.21 (m, 1H), 6.50 (d, *J* = 15.6 Hz, 1H), 6.49 (br s, 2H), 6.41-6.34 (m, 1H), 3.87 (br s, 9H), 3.52 (d, *J* = 6.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 153.13 (2x), 137.25, 136.32, 135.76, 131.01, 128.83, 128.39 (2x), 127.05 (2x), 126.01 (2x), 105.46, 60.69, 55.93 (2x), 39.56.

((1E,1'E)-oxybis(prop-1-ene-3,1,3-triyl))tetrabenzene

(6a).¹⁵ NaBH₄ (68 mg, 2.0 mmol) was added to a solution of **3a** (208 mg, 1.0 mmol) in the co-solvent of MeOH (3 mL) and THF (3 mL) at rt. The reaction mixture was stirred at rt for 30 min and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Without further purification, AgOTf (5 mg, 0.02 mmol; for Table 1, entry 7) was added to a solution of resulting crude 4a in CH₂Cl₂ (5 mL) at rt. The reaction mixture was stirred at rt for 1 h and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Purification on silica gel (hexanes/EtOAc = $10/1 \sim 6/1$) afforded **6a**. Two isomers, ratio = 1:1; Yield = 65% (131 mg); Colorless oil; HRMS (ESI, M^++1) calcd for C₃₀H₂₇O 403.2062, found 403.2066; ¹H NMR (400 MHz, CDCl₃): δ 7.56-7.28 (m, 20H), 6.71 (dd, J = 3.2, 15.6 Hz, 2H), 6.51-6.40 (m, 2H), 5.21 (t, J =8.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 141.23, 141.15, 136.60 (2x), 131.53, 131.33, 130.46, 130.29, 128.51 (4x), 128.48 (2x), 127.70 (2x), 127.68 (2x), 127.65 (2x), 127.05 (4x), 126.60 (2x), 126.58 (2x), 79.21, 79.09.

2,5-Diphenyltetrahydrofuran (8).¹⁶ NaBH₄ (68 mg, 2.0 mmol) was added to a solution of enedione 7 (236 mg, 1.0 mmol) in the co-solvent of MeOH (3 mL) and THF (3 mL) at rt. The reaction mixture was stirred at rt for 30 min and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Without further purification, Bi(OTf)₃ (13 mg, 0.02 mmol) was added to a solution of resulting crude diol in CH₂Cl₂ (5 mL) at rt. The reaction mixture was stirred at rt for 1 h and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Purification on silica gel (hexanes/EtOAc = $10/1 \sim 6/1$) afforded 8. Yield = 71%(159 mg); Colorless oil; HRMS (ESI, M⁺+1) calcd for C₁₆H₁₇O 225.1280, found 225.1283; ¹H NMR (400 MHz, CDCl₃): δ 7.53 (d, J = 7.6 Hz, 2H), 7.49 (d, J = 7.2 Hz, 2H), 7.45-7.41 (m, 4H), 7.37-7.32 (m, 2H), 5.34 (t, J = 6.0 Hz, 1H), 5.12 (t, J = 5.2 Hz, 1H), 2.58-2.43 (m, 2H), 2.12-2.00 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 143.59, 142.87, 128.28 (2x), 128.26 (2x), 127.21, 127.10, 125.91 (2x), 125.50 (2x), 81.24, 81.14, 35.49, 34.29.

3-(3,4-Dimethoxyphenyl)-1,3-Diphenylprop-1-ene (10).¹⁷ NaBH₄ (68 mg, 2.0 mmol) was added to a solution of 3a (208 mg, 1.0 mmol) in the co-solvent of MeOH (3 mL) and THF (3 mL) at rt. The reaction mixture was stirred at rt for 30 min and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Without further purification, BF3-OEt₂ (285 mg, 2.0 mmol) was added to a solution of resulting crude 4a and veratrole (280 mg, 2.0 mmol) in CH_2Cl_2 (5 mL) at rt. The reaction mixture was stirred at rt for 20 h and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to

8

afford crude product. Purification on silica gel (hexanes/EtOAc = $10/1 \sim 6/1$) afforded **10**. Yield = 79% (261 mg); Colorless solid; mp = 80-81 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M⁺+1) calcd for C₂₃H₂₃O₂ 331.1698, found 331.1697; ¹H NMR (400 MHz, CDCl₃): δ 7.48 (d, J = 7.2 Hz, 2H), 7.45-7.28 (m, 8H), 6.91 (s, 1H), 6.90 (d, J = 9.2 Hz, 2H), 6.80 (dd, J = 7.2 Hz, 1H), 6.48 (d, J = 16.0 Hz, 1H), 4.97 (d, J = 7.2 Hz, 1H), 3.93 (s, 3H), 3.90 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 148.70, 147.42, 143.39, 137.00, 135.79, 132.49, 131.01, 128.34 (2x), 128.27 (2x), 128.21 (2x), 127.47, 126.19, 126.04 (2x), 120.40, 111.82, 110.92, 55.56, 55.53, 55.45.

5,6-Dimethoxy-1,3-diphenyl-1H-indene (11). *m*CPBA (70%, 172 mg, 0.7 mmol) was added to a solution of 10 (165 mg, 0.5 mmol) in the CH₂Cl₂ (3 mL). The reaction mixture was stirred at rt for 4 h and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Without further purification, Bi(OTf)₃ (13 mg, 0.02 mmol) was added to a solution of resulting crude diol in CH₂Cl₂ (5 mL) at rt. The reaction mixture was stirred at rt for 1 h and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Purification on silica gel (hexanes/EtOAc = $10/1 \sim 6/1$) afforded **11**. Yield = 91% (149 mg); Colorless solid; mp = 141-142 °C (recrystallized from hexanes and EtOAc); HRMS (ESI, M^++1) calcd for $C_{23}H_{21}O_2$ 329.1542, found 329.1544; ¹H NMR (400 MHz, CDCl₃): δ 7.47-7.44 (m, 2H), 7.27-7.12 (m, 9H), 6.98 (s, 1H), 6.75 (s, 1H), 4.90 (s, 1H), 3.92 (s, 3H), 3.79 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 149.00, 148.66, 147.77, 142.09, 140.30, 135.83, 135.20, 128.87 (2x), 128.40 (2x), 127.82 (2x), 127.68, 126.87, 126.63, 126.23 (2x), 107.91, 104.58, 56.27, 56.18, 56.12.

Synthesis of deuterated 5w and 5x: NaBD₄ (68 mg, 2.0 mmol) was added to a solution of 3a (208 mg, 1.0 mmol) in the co-solvent of MeOH (3 mL) and THF (3 mL) at rt. The reaction mixture was stirred at rt for 30 min and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Without further purification, Bi(OTf)₃ (13 mg, 0.02 mmol) was added to a solution of resulting crude 4a-1 in CH₂Cl₂ (5 mL) at rt. The reaction mixture was stirred at rt for 1 h and the solvent was concentrated. The residue was diluted with water (10 mL) and the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Purification on silica gel (hexanes/EtOAc = $10/1 \sim 6/1$) afforded **3a** and a mixture of 5w and 5x. For 3a, Yield = 41% (85 mg); For a mixture of 5w and 5x (7:10), Yield = 34% (67 mg); Colorless oil;

HRMS (FAB, M⁺) calcd for $C_{15}H_{12}D_2$ 196.1219, found 196.1228; ¹H NMR (400 MHz, CDCl₃): δ 7.45-7.27 (m, 10H), 6.54 (d, J = 16.0 Hz, 5/9H for **5x**), 6.46-6.40 (m, 1H, for **5w+5x**), 3.58 (br s, 4/9H, for **5w**).

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10

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Supplementary Material

Experimental procedure and scanned photocopies of NMR (CDCl₃) spectral data were supported.