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An Effective Diels–Alder Reaction of Vinyl Allenols with Dienophiles

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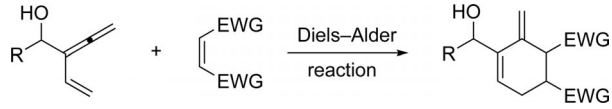
Vinyl allenols obtained from reaction of aldehydes with vinyl propargyl bromide and indium underwent Diels–Alder reactions with a variety of symmetric and unsymmetric dienophiles in dichloromethane or toluene regioselectively, producing cyclohexenylmethyl alcohols possessing an *exo* methylene moiety in good to excellent yield.

Introduction

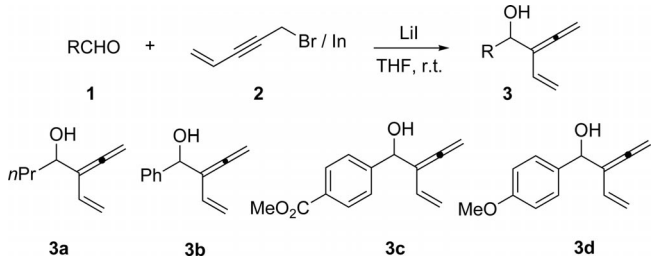
Allene having two adjacent double bonds is a very interesting functional group due to its hybrid character of having both carbon–carbon double and triple bonds.[1] Especially, vinyl allene compounds (allene having a vinyl group: 1,2,4-pentatriene) have been recognized as versatile building blocks in organic synthesis.[1] These compounds have been used in Diels–Alder reactions[2] as well as in transition-metal-catalyzed organic reactions.[3] However, because vinyl allenes are generally less stable than their isomeric enynes and because synthetic methods to selectively prepare a variety of vinyl allenes are difficult, their applications to organic synthesis have been limited. Therefore, the development of a synthetic method for preparing compounds having a functionalized vinyl allene moiety such as vinyl allenol and vinyl allenamine and its application to Diels–Alder reactions and transition-metal-catalyzed organic reactions is required. Although vinyl allenes have been used as the diene moiety in Diels–Alder reactions,[2] as far as we are aware, the use of vinyl allenols as the diene moiety in Diels–Alder reactions has not been reported. Because so much is now known about the Diels–Alder reaction,[4] successful use of vinyl allenols as diene moieties in Diels–Alder reactions would prove useful in the synthesis of cyclohexenylmethyl alcohols having an *exo* methylene moiety. We have recently demonstrated that a variety of organoindium reagents generated in situ from indium and allyl and propargyl halides are effective nucleophiles in Pd-catalyzed cross-coupling and addition reactions.[5] In continuation of our studies on preparative methods of vinyl allenols with organoindium reagents and their applications,[6] we herein describe an efficient Diels–Alder reaction of vinyl allenols with a range of dienophiles to give cyclohexenylmethyl alcohols having an *exo* methylene moiety (Scheme 1).



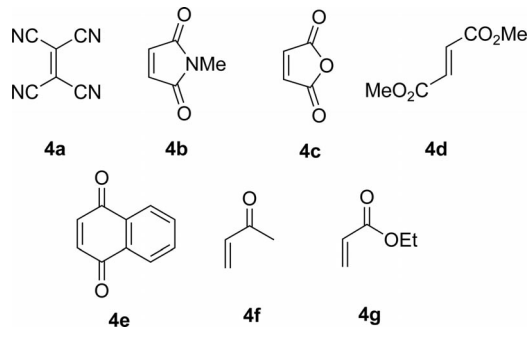
Scheme 1. Diels–Alder reaction of vinyl allenol with dienophile.

Results and Discussion

First, vinyl allenols **3** were produced in good to excellent yields from reaction of aldehydes with organoindium reagents generated in situ from vinyl propargyl bromide (**2**) and indium (Scheme 2).[6] For example, compound **3a** was regioselectively prepared in 81% yield from reaction of butanal (1 equiv.) with **2** (1 equiv.) and indium (1 equiv.) in the presence of lithium iodide (1 equiv.) in THF at 25 °C for



Scheme 2. Synthesis of vinyl allenol via selective 1,2,4-pentatriene-3-ylation to aldehydes.



Scheme 3. Dienophiles.

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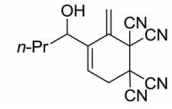
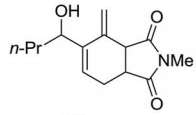
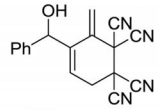
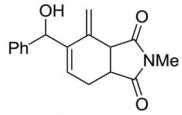
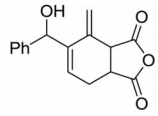
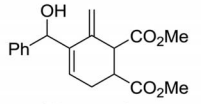
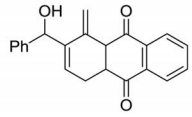
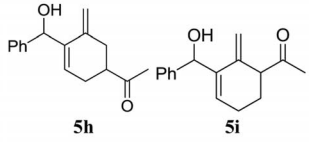
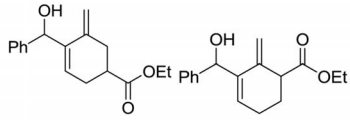
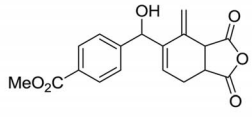
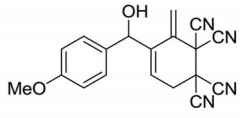
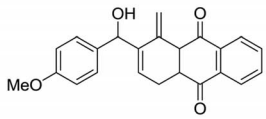
Table 1. Diels–Alder reaction of vinyl allenol with dienophile.

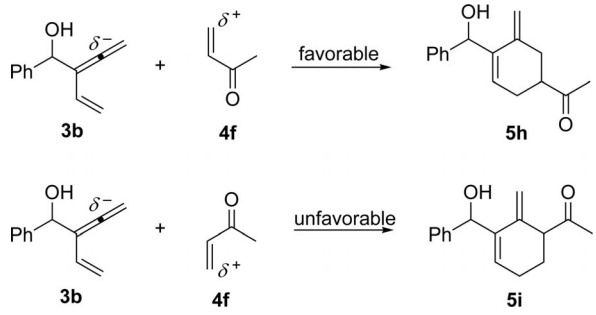
3 4 5

Entry	Vinyl allenol/ Dienophile	Solvent[a]	Time [h]	Product		Yield[b] [%]
1	**3a** / **4a**	CH_2Cl_2	1.5		**5a**	90
2	**3a** / **4b**	CH_2Cl_2	1		**5b**	92 (1:1.5)
3	**3b** / **4a**	CH_2Cl_2	2		**5c**	96
4	**3b** / **4b**	CH_2Cl_2	2		**5d**	97 (1:1.1)
5	**3b** / **4c**	CH_2Cl_2 toluene	4 3		**5e**	50 (1:4) 92 (1:4.4)
6	**3b** / **4d**	CH_2Cl_2 toluene	6 3		**5f**	40 (1:2) 62 (1:2)
7	**3b** / **4e**	CH_2Cl_2 toluene	5 3		**5g**	35 (1:1.3) 78 (1:1.8)
8	**3b** / **4f**[c]	CH_2Cl_2	3		**5h** **5i**	74 (1:2) 6 (1:5)
				5h **5i**		
9	**3b** / **4g**[d]	CH_2Cl_2	5		**5j**	34 (1:1)
		toluene	3		**5j** **5k**	62 (1:1.3) 7 (1:1.4)
				5j **5k**		
10	**3c** / **4c**	CH_2Cl_2	1.5		**5l**	93 (1:2.5)
11	**3d** / **4a**	CH_2Cl_2	3		**5m**	90
12	**3d** / **4e**	toluene	3.5		**5n**	86 (1:1.8)

[a] Reaction temperature: CH_2Cl_2, 25 °C; toluene, 80 °C. [b] Isolated yield. Diastereomeric ratio is given in parentheses, as determined by ^{1}H NMR spectroscopy. Dienophile **4** (2 equiv.) was used. [c] Dienophile **4** (4 equiv.) was used. [d] Dienophile **4** (6 equiv.) was used.

2 h under a nitrogen atmosphere. Vinyl allenols **3b**, **3c**, and **3d** were selectively obtained by treatment of the corresponding aldehyde with **2** and indium.

Next, Diels–Alder reactions of vinyl allenols **3** with dienophiles **4** (Scheme 3) were examined to obtain cyclohexenylmethyl alcohols **5** possessing an *exo* methylene moiety. The results are summarized in Table 1. Reaction of vinyl allenol **3a** with tetra(cyano)ethylene (**4a**) produced Diels–Alder adduct **5a** in 90% yield in dichloromethane at 25 °C after 1.5 h (Table 1, Entry 1). In addition, exposure of **3a** to *N*-methylmaleimide (**4b**) produced **5b** in 92% yield in dichloromethane at 25 °C after 1 h (Table 1, Entry 2). A coupling constant of the allylic proton at the bridgehead carbon is 8.76 Hz, indicating that the stereoisomer of the bicyclic ring is *cis*. The diastereomeric ratio of **5b** (1:1.5 *dr*) was determined from the integration ratio of the *exo* methylene protons (four singlets: δ = 5.57, 5.41, 5.34, and 5.27 ppm). Because Diels–Alder reactions of vinyl allenes with dienophiles proceed generally under harsh conditions (high temperature),[2g] it is noteworthy that the present Diels–Alder reactions proceed smoothly under mild reaction conditions (room temperature). Treatment of **3b** with **4a** afforded the corresponding adduct **5c** in 96% yield in dichloromethane (Table 1, Entry 3). Subjecting **3b** to **4b** gave adduct **5d** in 97% (1:1.1 *dr*) yield in dichloromethane (Table 1, Entry 4). The diastereomeric ratio of **5d** was determined from the integration ratio of the benzylic and *N*-methyl protons. Although reaction of **3b** with **4c** produced Diels–Alder adduct **5e** in 50% (1:4 *dr*) yield (dichloromethane, 25 °C, 4 h), use of toluene as solvent provided **5e** in 92% (1:4.4 *dr*) yield (80 °C, 3 h; Table 1, Entry 5). The diastereomeric ratio of **5e** was determined from the integration ratio of the vinylic protons (δ = 6.23 and 6.15 ppm) on the cyclohexenyl moiety. We were pleased to obtain **5f** in 62% (1:2 *dr*) yield by subjecting **3b** to dimethyl fumarate (**4d**) in toluene (Table 1, Entry 6). The diastereomeric ratio of **5f** was determined from the integration ratio of the *exo* methylene protons (δ = 5.16 and 5.11 ppm). In the case of naphthoquinone (**4e**), toluene gave a better result (78%, 1:1.8 *dr*) than dichloromethane (35%, 1:1.3 *dr*; Table 1, Entry 7). The diastereomeric ratio of **5g** was determined from the integration ratio of the vinylic protons (δ = 6.13 and 6.04 ppm) on the cyclohexenyl moiety. Reaction of **3b** with methyl vinyl ketone (**4f**, 4 equiv.) produced cyclohexenylmethyl alcohol **5h** (74%, 1:2 *dr*) and **5i** (6%, 1:5 *dr*) in dichloromethane at 25 °C after 3 h (Table 1, Entry 8). These results indicate that the electron-rich central carbon of vinyl allene preferentially adds to the more electron-deficient carbon of the dienophile (Scheme 4). The diastereomeric ratios of **5h** and **5i** were determined from the integration ratio of the benzylic protons (δ = 5.53 and 5.50 ppm) and the *exo* methylene protons (δ = 5.18 and 5.12 ppm), respectively. With ethyl acrylate (**4g**, 6 equiv.), adducts **5j** and **5k** were obtained in 62% (1:1.3 *dr*) and 7% (1:1.4 *dr*) yield, respectively, in toluene at 80 °C for 3 h (Table 1, Entry 9). The diastereomeric ratios of **5j** and **5k** were determined from the integration ratio of the vinylic protons (δ = 6.07 and 6.02 ppm) on the cyclohexenyl moiety and the benzylic protons (δ = 5.16 and 5.07 ppm), respectively. Encouraged by these results, we next examined the influence of the electron density of the aromatic rings on the reactivity of vinyl allenol. Vinyl allenol **3c** possessing a 4-methoxycarbonyl group as an electron-withdrawing group on its aromatic ring was treated with **4c** in dichloromethane at 25 °C for 1.5 h, providing corresponding adduct **5l** (93%, 1:2.5 *dr*; Table 1, Entry 10). The diastereomeric ratio of **5l** was determined from the integration ratio of the vinylic protons (δ = 6.21 and 6.09 ppm) on the cyclohexenyl moiety. Corresponding adduct **5m** was obtained in 90% yields from the Diels–Alder reaction of **4a** with vinyl allenol **3d** possessing a 4-methoxy group as an electron-donating group on its aromatic ring in dichloromethane at 25 °C after 3 h (Table 1, Entry 11). Reaction of **3d** with **4e** proceeded smoothly to produce adduct **5n** in 86% yield (1:1.8 *dr*) in toluene at 80 °C for 3.5 h (Table 1, Entry 12). These results imply that the reactivity of **3c** obtained from 4-methoxycarbonylbenzaldehyde is higher than that of **3d** obtained from 4-methoxybenzaldehyde. The diastereomeric ratio of **5n** was determined from the integration ratio of the *exo* methylene protons (δ = 4.82 and 4.75 ppm) on the cyclohexenyl moiety.



Scheme 4. Diels–Alder reaction of **3b** with **4f**.

Conclusions

In summary, cyclohexenylmethyl alcohols possessing an *exo* methylene moiety were regioselectively produced from Diels–Alder reactions of vinyl allenols, derived from selective addition reactions of organoindium reagents generated in situ from vinyl propargyl bromide and indium to aldehydes, with a variety of dienophiles in good to excellent yields in dichloromethane or toluene.

Experimental Section

General: Reactions were carried out in oven-dried glassware under a nitrogen atmosphere. All commercial reagents were used without purification, and all solvents were reaction grade. CH_2Cl_2 was freshly distilled from CaH_2. Toluene was freshly distilled from sodium under a nitrogen atmosphere. All reaction mixtures were magnetically stirred and were monitored by thin-layer chromatography by using Merck silica gel 60 F_{254} precoated glass plates, which were visualized with UV light and developed by using either iodine or a solution of anisaldehyde. Flash column chromatography was carried out by using Merck silica gel 60 (0.040–0.063 mm, 230–

400 mesh). ^{1}H and ^{13}C NMR spectra were recorded with a Bruker DPX FT (300 or 400 MHz) spectrometer. Deuterated chloroform was used as the solvent, and chemical shift values (δ) are reported in parts per million relative to the residual signals of this solvent (δ = 7.24 for ^{1}H and δ = 77.0 for ^{13}C). Infrared spectra were recorded with a JASCO FT/IR-460 plus FTIR spectrometer as either a thin film pressed between two sodium chloride plates or as a solid suspended in a potassium bromide disk. High-resolution mass spectra were recorded with a Jeol JMS 700 high-resolution mass spectrometer.

4-(1-Hydroxybutyl)-3-methylenecyclohex-4-ene-1,1,2,2-tetracarbonitrile (5a): Yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 6.14 (t, J = 3.3 Hz, 2 H, CH_2=), 6.07 (d, J = 2.0 Hz, 1 H, CH), 4.54 (s, 1 H, CH), 3.29 (d, J = 4.7 Hz, 2 H, CH_2), 2.11 (d, J = 3.6 Hz, 1 H, OH), 1.70–1.33 (m, 4 H, CH_2), 0.96 (t, J = 7.3 Hz, 3 H, CH_3) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 138.2, 128.4, 123.2, 119.4, 110.8, 110.7, 109.9, 109.8, 71.4, 45.8, 40.0, 38.5, 33.2, 14.6, 14.2 ppm. IR (film): $\tilde{\nu}$ = 3329, 3067, 2247, 1642, 1024, 899 cm^{-1}. HRMS (EI): calcd. for $C_{15}H_{14}N_4O$ 266.1168; found 266.1169.

5-(1-Hydroxybutyl)-2-methyl-4-methylene-3a,4,7,7a-tetrahydro-1*H*-isoindole-1,3(2*H*)-dione (5b): A mixture of 3-vinyl-1,2-heptadien-4-ol (**3a**; 36.1 mg, 0.5 mmol) and *N*-methylmaleimide (**4b**; 111.1 mg, 1.0 mmol) in CH_2Cl_2 (2.0 mL) at room temperature using a test tube. After being stirred for 1 h, the solvent was removed under reduced pressure. The product was purified by silica gel column chromatography (EtOAc/hexane, 1:2) to give **5b** (115 mg, 92%) as a yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 5.90 (d, J = 3.5 Hz, 1 H, CH_2), 5.57 (s, 1 H, CH_2=), 5.41(s, 1 H, CH_2=), 4.27 (t, J = 6.5 Hz, 1 H, CH), 3.69 (d, J = 8.8 Hz, 1 H, CH), 3.23–3.15 (m, 1 H, CH), 2.95 (s, 3 H, NCH_3), 2.83–2.68 (m, 1 H, CH_2), 2.39–2.23 (m, 1 H, CH_2), 1.86 (s, 1 H, OH), 1.65–1.48 (m, 2 H, CH_2), 1.41–1.05 (m, 2 H, CH_2), 0.90 (t, J = 7.4 Hz, 3 H, CH_3) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 179.9, 177.7, 141.8, 134.2, 125.2, 116.2, 73.8, 47.9, 39.3, 37.7, 25.7, 23.5, 19.6, 14.3 ppm. Data for the minor isomer: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 5.95 (d, J = 6.7 Hz, 1 H, CH_2), 5.34 (s, 1 H, CH_2=), 5.27 (s, 1 H, CH_2=), 4.48 (s, 1 H, CH), 3.69 (d, J = 8.8 Hz, 1 H, CH), 3.23–3.15 (m, 1 H, CH), 2.95 (s, 3 H, NCH_3), 2.83–2.68 (m, 1 H, CH_2), 2.39–2.23 (m, 1 H, CH_2), 1.77 (s, 1 H, OH), 1.65–1.48 (m, 2 H, CH_2), 1.41–1.05 (m, 2 H, CH_2), 0.90 (t, J = 7.4 Hz, 3 H, CH_3) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 180.0, 177.6, 142.8, 135.4, 121.8, 114.9, 71.2, 48.3, 39.4, 38.4, 25.6, 23.4, 18.7, 14.2 ppm. IR (film): $\tilde{\nu}$ = 3397, 2957, 2874, 1679, 1067, 911, 816 cm^{-1}. HRMS (EI): calcd. for $C_{14}H_{19}NO_3$ 249.1365; found 249.1361.

4-[Hydroxy(phenyl)methyl]-3-methylenecyclohex-4-ene-1,1-2,2-tetracarbonitrile (5c): Yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.43–7.31 (m, 5 H, ArH), 6.40 (t, J = 4.0 Hz, 1 H, CH), 6.00 (s, 1 H, CH_2=), 5.78 (d, J = 2.4 Hz, 1 H, CH_2=), 5.53 (s, 1 H, CH), 3.39 (d, J = 4.1 Hz, 2 H, CH_2), 1.28 (s, 1 H, OH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 140.2, 136.1, 129.8, 1219.6, 127.7, 127.2, 124.7, 120.9, 110.9, 110.7, 109.7, 109.6, 73.8, 45.7, 40.0, 33.5 ppm. IR (film): $\tilde{\nu}$ = 3068, 2281, 2248, 1602, 1489, 1376, 760, 709 cm^{-1}. M.p. 117–119 °C. HRMS (EI): calcd. for $C_{18}H_{12}N_4O$ 300.1011; found 300.1013.

5-[Hydroxy(phenyl)methyl]-2-methyl-4-methylene-3a,4,7,7a-tetrahydro-1*H*-isoindole-1,3(2*H*)-dione (5d): Pale yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.36–7.17 (m, 5 H, ArH), 5.96 (d, J = 5.4 Hz, 1 H, CH), 5.39 (s, 1 H, CH), 5.31 (s, 1 H, CH_2=), 5.27 (s, 1 H, CH_2=), 3.61 (t, J = 9.1 Hz, 1 H, CH), 3.14 (q, J = 7.6 Hz, 1 H, CH), 2.92 (s, 3 H, NCH_3). 2.88–2.72 (m, 1 H, CH_2), 2.44–2.27 (m, 1 H, CH_2), 2.20 (s, 1 H, OH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 179.8, 177.5, 141.5, 141.0, 134.1, 128.9, 128.3, 126.7, 126.0, 116.5, 75.0, 47.4, 39.1, 25.8, 23.1 ppm. Data for the minor isomer: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.36–7.17 (m, 5 H, ArH), 6.19 (d, J = 6.4 Hz, 1 H, CH), 5.44 (s, 1 H, CH), 5.35 (s, 1 H, CH), 5.32 (s, 1 H, CH), 3.61 (t, J = 9.1 Hz, 1 H, s, 1 H, CH), 3.14 (q, J = 7.6 Hz, 1 H, s, 1 H, CH), 2.74 (s, 3 H, NCH_3). 2.88–2.72 (m, 1 H, CH_2), 2.44–2.27 (m, 1 H, CH_2), 2.20 (s, 1 H, OH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 179.8, 176.8, 142.4, 142.0, 134.5, 128.8, 128.3, 127.2, 123.3, 116.0, 74.3, 48.0, 39.6, 25.6, 23.5 ppm. IR (film): $\tilde{\nu}$ = 3036, 2934, 1747, 1067, 884, 708 cm^{-1}. M.p. 97–99 °*C*. HRMS (EI): calcd. for $C_{17}H_{17}N_4O_3$ 283.1208; found 283.1206.

5-[Hydroxy(phenyl)methyl]-4-methylene-3a,4,7,7a-tetrahydroisobenzofuran-1,3-dione (5e): Yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.38–7.22 (m, 5 H, ArH), 6.16 (s, 1 H, CH), 5.43 (s, 1 H, CH), 5.36 (d, J = 10.1 Hz, 2 H, CH_2=), 3.90 (d, J = 9.4 Hz, 1 H, CH), 3.45 (td, J = 8.5, 3.5 Hz, 1 H, CH), 2.85–2.73 (m, 1 H, CH_2), 2.58–2.43 (m, 1 H, CH_2), 2.30 (s, 1 H, OH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 173.6, 171.4, 141.8, 140.2, 131.2, 129.1, 128.5, 127.3, 125.6, 117.6, 74.5, 46.7, 39.6, 23.0 ppm. Data for the minor isomer: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.38–7.22 (m, 5 H, ArH), 6.23 (s, 1 H, CH), 5.48 (s, 1 H, CH), 5.36 (d, J = 10.1 Hz, 2 H, CH_2=), 3.90 (d, J = 9.4 Hz, 1 H, CH), 3.45 (td, J = 8.5, 3.5 Hz 1 H, CH), 2.85–2.73 (m, 1 H, CH_2), 2.58–2.43 (m, 1 H, CH_2), 2.30 (s, 1 H, OH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 173.8, 171.7, 141.8, 140.3, 131.5, 129.2, 128.6, 127.0, 124.5, 117.4, 74.2, 47.1, 39.9, 23.0 ppm. IR (film): $\tilde{\nu}$ = 3066, 1778, 1718, 1046, 898, 711 cm^{-1}. M.p. 83–85 °C. HRMS (EI): calcd. for $C_{16}H_{14}O_4$ 270.0892; found 270.0889.

Dimethyl 4-[Hydroxy(phenyl)methyl]-3-methylenecyclohex-4-ene-1,2-dicarboxylate (5f): Yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.34–7.23 (m, 5 H, ArH), 6.02 (s, 1 H, CH), 5.53 (s, 1 H, CH), 5.15 (s, 1 H, CH_2=), 4.90 (s, 1 H, CH_2=), 3.70 (s, 3 H, OCH_3), 3.68 (s, 1 H, CH), 3.67 (s, 3 H, OCH_3), 3.22 (q, J = 6.7 Hz, 1 H, CH), 2.68–2.49 (m, 2 H, CH_2), 2.20 (s, 1 H, OH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 174.3, 173.3, 142.6, 137.9, 136.8, 128.9, 128.1, 127.3, 126.8, 114.2, 74.0, 52.6, 49.6, 41.8, 26.8 ppm. Data for the minor isomer: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.34–7.23 (m, 5 H, ArH), 6.06 (s, 1 H, CH), 5.53 (s, 1 H, CH), 5.11 (s, 1 H, CH_2=), 4.90 (s, 1 H, CH_2=), 3.70 (s, 3 H, OCH_3), 3.67 (s, 3 H, OCH_3), 3.64 (s, 1 H, CH), 3.22 (q, J = 6.7 Hz, 1 H, CH), 2.68–2.49 (m, 2 H, CH_2), 2.20 (s, 1 H, OH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 174.3, 173.3, 142.7, 137.9, 136.7, 128.8, 128.1, 127.2, 126.6, 114.3, 74.1, 52.5, 49.6, 41.7, 36.7 ppm. IR (film): $\tilde{\nu}$ = 3074, 1772, 1741, 1603, 1498, 1377, 1222, 910, 812 cm^{-1}. HRMS (EI): calcd. for $C_{18}H_{20}O_5$ 316.1311; found 316.1313.

2-[Hydroxy(phenyl)methyl]-1-methylene-1,4,4a,9a-tetrahydroanthracene-9,10-dione (5g): A mixture of 1-phenyl-2-vinyl-2,3-butadien-1-ol (**3b**; 86.1 mg, 0.5 mmol) and 1,4-naphthoquinone (**4e**; 158.2 mg, 1.0 mmol) in toluene (2.0 mL) was heated at 80 °C for 3 h. The solvent was removed under reduced pressure. The product was purified by silica gel column chromatography (EtOAc/hexane, 1:2) to give **5g** (128.8 mg, 78%) as a brown solid. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 8.04–7.95 (m, 2 H, ArH), 7.76–7.67 (m, 2 H, ArH), 7.36–7.22 (m, 5 H, ArH), 6.03 (t, J = 4.0 Hz, 1 H, CH), 5.55 (s, 1 H, CH), 5.24 (s, 1 H, CH_2=), 4.8 (s, 1 H, CH_2=), 4.0 (t, J = 6.2 Hz 1 H, CH), 3.49 (quint., J = 5.4 Hz, 1 H, CH), 2.83–2.65 (m, 1 H, CH_2), 2.56–2.39 (m, 1 H, CH_2), 2.56–2.39 (m, 1 H, OH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 196.9, 142.4, 138.5, 135.7, 135.3, 134.8, 134.7, 129.2, 128.8, 128.1, 127.8, 127.4, 127.3, 73.7, 48.0, 26.3 ppm. Data for the minor isomer: ^{1}H

NMR (300 MHz, $CDCl_3$, 25 °C): δ = 8.04–7.95 (m, 2 H, ArH), 7.76–7.67 (m, 2 H, ArH), 7.36–7.22 (m, 5 H, ArH), 6.03 (t, J = 4.0 Hz, 1 H, CH), 5.51 (s, 1 H, CH), 5.13 (s, 1 H, CH_2=), 4.75 (s, 1 H, CH_2=), 4.0 (t, J = 6.2 Hz 1 H, CH), 3.49 (quint., J = 5.4 Hz, 1 H, CH), 2.83–2.65 (m, 1 H, CH_2), 2.56–2.39 (m, 1 H, CH_2), 2.56–2.39 (m, 1 H, OH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 197.5, 142.6, 137.8, 135.6, 135.3, 134.9, 134.5, 128.9, 128.6, 128.2, 127.7, 127.4, 127.3, 74.2, 55.4, 26.2 ppm. IR (film): $\tilde{\nu}$ = 3396, 3351, 3082, 2903, 1936, 1688, 1272, 897, 707 cm^{-1}. M.p. 82–83 °C. HRMS (EI): calcd. for $C_{22}H_{18}O_3$ 330.1256; found 330.1256.

1-{4-[Hydroxy(phenyl)methyl]-5-methylenecyclohex-3-enyl}ethanone (5h): Pale yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.38–7.23 (m, 5 H, ArH), 6.03 (t, J = 4.2 Hz, 1 H, CH), 5.50 (s, 1 H, CH), 4.94 (s, 1 H, CH_2=), 4.86 (s, 1 H, CH_2=), 2.82–2.70 (m, 1 H, CH), 2.57 (d, J = 3.2 Hz, 1 H, OH), 2.47–2.32 (m, 4 H, CH_2), 2.17 (s, 3 H, CH_3) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 210.8, 143.1, 139.8, 138.8, 128.8, 128.0, 127.8, 127.3, 127.2, 111.7, 73.9, 47.9, 35.2, 28.4, 28.0 ppm. Data for the minor isomer: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.38–7.23 (m, 5 H, ArH), 6.07 (t, J = 4.2 Hz, 1 H, CH), 5.53 (s, 1 H, CH), 4.95 (s, 1 H, CH_2=), 4.86 (s, 1 H, CH_2=), 2.82–2.70 (m, 1 H, CH), 2.61 (d, J = 3.1 Hz, 1 H, OH), 2.47–2.32 (m, 4 H, CH_2), 2.17 (s, 3 H, CH_3) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 210.8, 143.1, 139.8, 138.6, 128.8, 127.4, 127.3, 111.6, 73.9, 47.9, 35.2, 38.4, 28.0 ppm. IR (film): $\tilde{\nu}$ = 3351, 2995, 2918, 1688, 1213, 1020, 899, 710 cm^{-1}. HRMS (EI): calcd. for $C_{16}H_{18}O_2$ 242.1307; found 242.1306.

4-(1-Hydroxybutyl)-3-methylenecyclohex-4-ene-1,1,2,2-tetracarbonitrile (5i): Pale yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.34–7.20 (m, 5 H, ArH), 5.89 (s, 1 H, CH), 5.49 (s, 1 H, CH), 5.18 (s, 1 H, CH_2=), 4.85 (s, 1 H, CH_2=), 3.18 (t, J = 4.5 Hz, 1 H, CH), 2.29–2.10 (m, 3 H, CH_2), 2.05 (s, 1 H, OH), 2.02 (s, 3 H, CH_3), 1.77–1.71 (m, 1 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 208.6, 206.0, 138.4, 136.6, 129.0, 127.3, 126.5, 125.7, 112.7, 73.4, 29.9, 27.0, 23.1, 22.2 ppm. Data for the minor isomer: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.34–7.20 (m, 5 H, ArH), 6.06 (s, 1 H, CH), 5.49 (s, 1 H, CH), 5.12 (s, 1 H, CH_2=), 4.85 (s, 1 H, CH_2=), 3.14 (t, J = 5.0 Hz, 1 H, CH), 2.29–2.10 (m, 3 H, CH_2), 1.92 (s, 1 H, OH), 1.81 (s, 3 H, CH_3), 1.77–1.71 (m, 1 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 208.6, 206.0, 141.3, 136.1, 127.6, 127.4, 126.7, 125.8, 72.7, 54.4, 26.9, 22.8, 22.0 ppm. IR (film): $\tilde{\nu}$ = 3352, 2995, 2919, 1689, 1213, 1020, 899, 709 cm^{-1}. HRMS (EI): calcd. for $C_{16}H_{18}O_2$ 242.1307; found 242.1306.

Ethyl 3-[Hydroxy(phenyl)methyl]-2-methylenecyclohex-3-enecarboxylate (5j): Pale yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.39–7.26 (m, 5 H, ArH), 6.02 (s, 1 H, CH), 5.52 (s, 1 H, CH), 4.93 (s, 1 H, CH_2=), 4.86 (s, 1 H, CH_2=), 4.14 (q, J = 7.3 Hz, 2 H, CO_2CH_2), 2.74–2.42 (m, 5 H, CH_2), 2.15 (d, J = 3.5 Hz, 1 H, OH), 1.25 (td, J = 7.1, 2.1 Hz, 3 H, CH_3) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 169.8, 144.0, 143.5, 142.4, 128.8, 127.7, 127.2, 127.1, 122.3, 72.0, 62.7, 48.2, 36.6, 26.1, 14.6 ppm. Data for the minor isomer: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.39–7.26 (m, 5 H, ArH), 6.07 (s, 1 H, CH), 5.54 (s, 1 H, CH), 4.93 (s, 1 H, CH_2=), 4.86 (s, 1 H, CH_2=), 4.14 (q, J = 7.3 Hz, 2 H, $CO_2CH_2CH_3$), 2.74–2.42 (m, 5 H, CH_2), 2.11 (d, J = 4.0 Hz, 1 H, OH), 1.25 (td, J = 7.1, 2.1 Hz, 3 H, CH_3) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 169.9, 144.2, 143.2, 142.2, 128.7, 127.6, 127.2, 126.8, 121.1, 71.9, 62.5, 48.2, 36.6, 26.3, 14.4 ppm. IR (film): $\tilde{\nu}$ = 3349, 3074, 2998, 1212, 899.712 cm^{-1}. HRMS (EI): calcd. for $C_{17}H_{20}O_3$ 272.1412; found 272.1412.

Ethyl 3-[Hydroxy(phenyl)methyl]-2-methylenecyclohex-3-enecarboxylate (5k): Pale yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.39–7.26 (m, 5 H, ArH), 5.92 (s, 1 H, CH), 5.52 (s, 1 H, CH), 5.16 (s, 1 H, CH_2=), 4.91 (s, 1 H, CH_2=), 4.14 (q, J = 7.3 Hz, 2 H, CO_2CH_2), 3.30 (t, J = 4.3 Hz, 1 H, CH), 2.74–2.42 (m, 4 H, CH_2), 2.15 (d, J = 3.5 Hz, 1 H, OH), 1.25 (td, J = 7.1, 2.1 Hz, 3 H, CH_3) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 169.8, 144.0, 143.5, 142.4, 128.8, 127.7, 127.2, 127.1, 122.3, 72.0, 62.7, 48.2, 36.6, 26.1, 14.6 ppm. Data for the minor isomer: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.39–7.26 (m, 5 H, ArH), 6.07 (s, 1 H, CH), 5.54 (s, 1 H, CH), 5.07 (s, 1 H, CH_2=), 4.91 (s, 1 H, CH_2=), 4.14 (q, J = 7.3 Hz, 2 H, $CO_2CH_2CH_3$), 3.30 (t, J = 4.3 Hz, 1 H, CH), 2.74–2.42 (m, 4 H, CH_2), 2.11 (d, J = 4.0 Hz, 1 H, OH), 1.25 (td, J = 7.1, 2.1 Hz, 3 H, CH_3) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 169.9, 144.2, 143.2, 142.2, 128.7, 127.6, 127.2, 126.8, 121.1, 71.9, 62.5, 48.2, 36.6, 26.3, 14.4 ppm. IR (film): $\tilde{\nu}$ = 3349, 3074, 2998, 1212, 899.712 cm^{-1}. HRMS (EI): calcd. for $C_{17}H_{20}O_3$ 272.1412; found 272.1412.

Methyl 4-[Hydroxy(4-methylene-1,3-dioxo-1,3,3a,4,7,7a-hexahydroisobenzofuran-5-yl)methyl]benzoate (5l): Yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.97 (d, J = 8.1 Hz, 2 H, ArH), 7.38 (d, J = 8.3 Hz, 2 H, ArH), 6.09 (s, 1 H, CH), 5.50 (s, 1 H, CH), 5.37 (d, J = 10.7 Hz, 2 H, CH_2=), 3.94 (d, J = 9.8 Hz, 2 H, CH), 3.89 (s, 3 H, OCH_3), 3.49 (q, J = 7.0 Hz, 1 H, OH), 2.83–2.71 (m, 1 H, CH_2), 2.57–2.44 (m, 1 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 173.6, 171.3, 167.3, 164.6, 146.8, 137.0, 131.1, 130.3, 127.1, 126.7, 117.8, 74.3, 52.6, 46.7, 39.6, 23.0 ppm. Data for the minor isomer: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.61 (d, J = 7.9 Hz, 2 H, ArH), 7.33 (d, J = 8.3 Hz, 2 H, ArH), 6.20 (s, 1 H, CH), 5.53 (s, 1 H, CH), 5.37 (d, J = 5.2 Hz, 2 H, CH_2=), 3.94 (d, J = 9.8 Hz, 2 H, CH), 3.88 (s, 3 H, OCH_3), 3.49 (q, J = 7.0 Hz, 1 H, OH), 2.83–2.71 (m, 1 H, CH_2), 2.57–2.44 (m, 1 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 173.7, 171.1, 167.3, 164.6, 146.9, 140.0, 131.3, 130.3, 126.9, 125.5, 117.7, 73.9, 52.6, 47.0, 39.8, 23.0 ppm. IR (film): $\tilde{\nu}$ = 3459, 3055, 2953, 1610, 1778, 1046, 897, 710 cm^{-1}. M.p. 132–134 °C. HRMS (EI): calcd. for $C_{18}H_{16}O_6$ 328.0947; found 328.0949.

4-[Hydroxy(4-methoxyphenyl)methyl]-3-methylenecyclohex-4-ene-1,1,2,2-tetracarbonitrile (5m): Beige solid. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 7.25 (d, J = 7.4 Hz, 2 H, ArH), 6.91 (d, J = 8.8 Hz, 2 H, ArH), 6.45 (t, J = 4.0 Hz, 1 H, CH), 6.00 (s, 1 H, CH), 5.73 (d, J = 2.5 Hz, 1 H, CH_2=), 5.50 (s, 1 H, CH_2=), 3.81 (s, 3 H, OCH_3), 3.41 (d, J = 4.0 Hz, 2 H, CH_2) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 159.9, 152.6, 142.6, 136.1, 128.8, 128.5, 123.0, 122.2, 117.4, 115.4, 111.2, 109.9, 71.0, 56.6, 33.4, 22.7, 21.8 ppm. IR (film): $\tilde{\nu}$ = 3419, 3002, 2837, 2248, 1940, 1610, 907 cm^{-1} M.p. 105–107 °C. HRMS (EI): calcd. for $C_{18}H_{12}N_4O$ 330.1117; found 330.1115.

2-[Hydroxy(4-methoxyphenyl)methyl]-1-methylene-1,4,4a,9a-tetrahydroanthracene-9,10-dione (5n): Brown solid. ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 8.08–7.97 (m, 2 H, ArH), 7.80–7.69 (m, 2 H, ArH), 7.29–7.20 (m, 2 H, ArH), 6.89–6.83 (m, 2 H, ArH), 6.07 (t, J = 4.0 Hz, 1 H, CH), 5.54 (s, 1 H, CH), 5.23 (s, 1 H, CH_2=), 4.82 (s, 1 H, CH_2=), 4.01 (d, J = 5.2 Hz, 1 H, CH), 3.80 (s, 3 H, OCH_3), 3.56 (m, 1 H, CH), 2.89–2.68 (m, 1 H, CH_2), 2.57–2.43 (m, 1 H, CH_2), 2.03 (s, 1 H, OH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 199.8, 189.9, 161.1, 144.3, 143.2, 138.2, 137.7, 133.6, 133.3, 133.2, 129.7, 125.9, 124.7, 114.6, 114.5, 110.1, 74.1, 63.3, 54.8, 46.9, 23.8 ppm. Data for the minor isomer: ^{1}H NMR (300 MHz, $CDCl_3$, 25 °C): δ = 8.08–7.97 (m, 2 H, ArH), 7.80–7.69 (m, 2 H, ArH), 7.29–7.20 (m, 2 H, ArH), 6.89–6.83 (m, 2 H, ArH), 6.22 (t, J = 4.0 Hz, 1 H, CH), 5.48 (s, 1 H, CH), 5.07 (s, 1 H, CH_2=), 4.75 (s, 1 H, CH_2=), 4.04 (d, J = 5.0 Hz, 1 H, CH), 3.79 (s, 3 H, OCH_3), 3.56 (m, 1 H, CH), 2.89–2.68 (m, 1 H, CH_2), 2.57–2.43 (m, 1 H,

CH_2), 1.86 (s, 1 H, OH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 199.7, 189.8, 161.1, 144.1, 143.2, 138.0, 137.9, 133.7, 133.4, 133.1, 129.4, 125.8, 124.7, 114.5, 114.2, 110.1, 74.2, 63.1, 54.6, 46.6, 23.9 ppm. IR (film): $\tilde{\nu}$ = 3418, 3004, 2837, 1941, 1688, 1611, 1271, 905, 708 cm^{-1}. M.p. 99–100 °C. HRMS (EI): calcd. for $C_{23}H_{20}O_4$ 360.1362; found 360.1365.

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