in flame-dried glassware under an atmosphere of dry nitrogen.

(*S*)-(−)-Bis(trimethylsilyl) 2-[(Trimethylsilyl)oxy]butanedioate (2a).[11] To (*S*)-(−)-malic acid (20.0 g, 149 mmol) was added HMDSH (34.6 mL, 164 mmol). To the resulting stirred slurry was added dropwise Me_3SiCl (20.8 mL, 164 mmol) at such a rate so as to avoid boiling. The slurry was stirred for 12 h and filtered through sintered glass. The solid was washed with CH_2Cl_2, and the filtrate was distilled (bp 137–140 °C, 11 mmHg) to provide **2a** (44.6 g, 127 mmol, 85.2%) as a colorless liquid: α^{28}_D −43.20° (neat): ^1H NMR ($CDCl_3$) δ 4.54 (dd, J = 8.3 and 4.2 Hz, H(2), H_X of ABX), 2.82 (dd, J = 16.1 and 4.2 Hz, H_A of ABX), 2.65 (dd, J = 16.0 and 8.3, H_B of ABX), 0.30 (s, 9 H), 0.29 (s, 9 H), and 0.14 (s, 9 H); IR (neat) 1723, 1254 cm^{-1}. Anal. Calcd for $C_{13}H_{30}O_5Si_3$: C, 44.51; H, 8.86. Found C, 44.76; H, 8.86.

(±)-Trimethylsilyl 2-[(Trimethylsilyl)oxy]-2-phenylethanoate (2b).[12] To a solution of (±)-mandelic acid (10.0 g, 65.7 mmol) in 30 mL of dry CH_2Cl_2 was added HMDSH (15.3 mL, 72.3 mmol) dropwise with formation of a white preciptate. The slurry was stirred overnight and the CH_2Cl_2 was removed by distillation under aspirator pressure with concomitant disappearance of the white precipitate. The residual liquid was distilled (bp 97–98 °C, 0.25 mmHg) to provide **2b** (19.24 g, 99%) as a colorless liquid: ^1H NMR ($CDCl_3$) δ 7.38 (m, Ar H), 5.13 (s, H(2)), 0.20 (s, 9 H), and 0.13 (s, 9 H); IR (neat) 1739 (s), 1715 (m) (presumably split by Fermi resonance), 1254, 849 cm^{-1}. Anal. Calcd for $C_{14}H_{24}O_3Si_2$: C, 56.69; H, 8.16. Found: C, 56.84; H, 8.16.

(*S*)-(−)-Trimethylsilyl 2-[(Trimethylsilyl)oxy]propanoate (2c).[13] Following the procedure for the preparation of **2b**, (*S*)-lactic acid (2.09 g, 23.2 mmol) was converted to **2c** (2.63 g, 10.0 mmol, 43.2%) as a colorless liquid (bp 43–48 °C, 3.3 mmHg): α^{28}_D −32.6° (neat); ^1H NMR ($CDCl_3$) δ 4.24 (q, J = 6.8 Hz, H(2)), 1.40 (d, J = 6.8 Hz, CH_3), 0.30 (s, 9 H), and 0.14 (s, 9 H); IR (neat) 1738, 1254 cm^{-1}.

(±)-Trimethylsilyl 2-[(Trimethylsilyl)thio]propanoate (2d).[14] Following the procedure for the preparation of **2b**, (±)-thiolactic acid (5.98 g, 56.3 mmol) was converted to **2d** (6.54 g, 46%) as a colorless liquid (bp 68 °C, 7 mmHg): ^1H NMR ($CDCl_3$) δ 3.38 (q, J = 7 Hz, H(2)), 1.47 (d, J = 7 Hz, CH_3), 0.35 (s, 9 H), and 0.30 (s, 9 H); IR (neat) 1718, 1253 cm^{-1}. Anal. Calcd for $C_9H_{22}O_2SSi_2$: C, 43.13; H, 8.85. Found: C, 43.04; H, 8.89.

(2*S*,4*S*)-(−)-2-*tert*-Butyl-5-oxo-1,3-dioxolane-4-acetic Acid (*cis*-1a).[1c] The diester **2a** (20.0 mL, 19.4 g, 55.4 mmol) was dissolved in 200 mL of CH_2Cl_2 and cooled to −25 °C. Pivaldehyde (6.9 mL, 63.9 mmol) was added, and the stirred mixture was treated with Me_3SiOTf (1.1 mL, 5.8 mmol). The mixture was stirred at −25 °C for 6 h and then quenched with 1 N HCl. Extraction with CH_2Cl_2, drying ($MgSO_4$), and concentration left crude **1a** as a white solid. Treatment of a portion of this material with ethereal diazomethane and analysis by capillary GC under conditions known to resolve the cis and trans diastereomers of **1a** [obtained after separation (MPLC on silica gel in 6:1 hexane/EtOAc containing 0.5% AcOH) and CH_2N_2 treatment of mixtures of 1 resulting from reactions at higher temperatures] revealed a single peak. The crude **1a** was purified by recrystallization to leave *cis*-**1a** (78% yield, mp 104–105 °C).

General Procedure for Preparation of 1b, 1c, and 1d. The bis(trimethylsilyl) derivatives **2b–d** (0.3 M in CH_2Cl_2) were treated sequentially with 1.1 equiv of *t*-BuCHO and 0.10–0.15 equiv of Me_3SiOTf at −25 or 0 °C. After 10 min to several hours (depending upon substrate and temperature), water was added. Extraction (CH_2Cl_2), drying ($MgSO_4$), concentration, and separation by MPLC on silica gel provided the individual diastereomers of **1b–d** in the yields and ratios indicated in Table I. Spectral data were consistent with those previously descirbed.[1c] In addition: (±)-*trans*-**1b**: colorless oil; IR (neat) 1800, 1200 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.47–7.36 (m, 5 H), 5.45 (s, 1 H), 5.39 (s, 1 H), and 1.04 (s, 9 H); (±)-*cis*-**1b**: mp 111–114 °C.

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Synthesis of 1,3-Dioxolan-4-ones. An Improved Procedure

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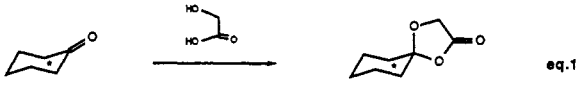
In conjunction with our efforts to develop a new method for the asymmetric synthesis of α-hydroxy acids,[2] we required chiral 1,3-dioxolan-4-ones such as **1**, which are formally derived from condensation of glycolic acid with chiral cycloalkanones (eq 1). Although 1,3-dioxolan-4-ones



1

may generally be prepared from carbonyl compounds and branched α-hydroxy acids such as lactic and mandelic acids,[3] those derived from glycolic acid are rare.[3a,4] For example, acid-catalyzed condensation of glycolic acid with cyclohexanone is reported to afford 1,4-dioxaspiro[4.5]-decane-2-one **3** in low yield as an oil that is unstable at room temperature.[3a] In this paper we report a new route to such materials that proceeds in good to excellent yield to provide stable dioxolanones.[11] Further, the stereo-

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(9) ^1H NMR spectra were recorded on a Bruker WM-300 or WM-360 spectrometer, using tetramethylsilane as an internal standard. ^{13}C NMR spectra were obtained on a Bruker WM-300 (75 MHz) or WM-360 (90 MHz) spectrometer and are reported relative to deuteriated chloroform as an internal standard. Infrared spectra were recorded on a Nicolet 5-DX (FTIR) or 60-SX (FTIR) spectrometer. Mass spectra were obtained on a Finnigan 4500 GC/MS-EICI system at 70 eV. Elemental analyses were performed by Spang Microanalytical Laboratory at Eagle Harbor, MI, or Galbraith Laboratories, Inc. at Knoxville, TN. Gas chromatographic analyses were performed on a Hewlett-Packard 5890 chromatograph equipped with a fused silica capillary column (Carbowax) and a flame ionization detector. HPLC analyses were performed with Rainin Microsorb SiO_2 or C18 HPLC columns (analytical) or a Rainin Dynamax Macro HPLC SiO_2 column (preparative).

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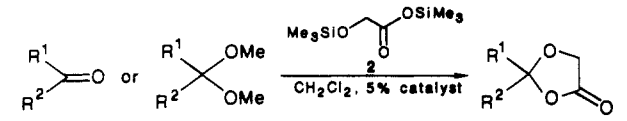
Table I. Preparation of 1,3-Dioxolan-4-ones



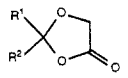
substrate	scale[a] (mmol)	catalyst	temperature (°C)/time (h)	product	yield[b] (%)	ratio[c] A:B
cyclohexanone	20.0	Me_3SiOTf	−78/5	3[d]	99	
	10.0	Me_3SiI	−78/3	3	99	
1,1-dimethoxycyclohexane	5.0	Me_3SiOTf	−78/4	3	83	
	5.0	Me_3SiI	−78/4	3	82	
3-methylcyclohexanone	19.0	Me_3SiOTf	−78/5	4	99	1.1:1.0
menthone[e]	10.0	Me_3SiOTf	−78/6 −10/2	5	99	1.1:1.0
8-phenylmenthone[f]	29.0	Me_3SiOTf	−78/2 rt[j]/22	6	74 (25)[g]	1.3:1.0
	0.7	Me_3SiOTf[h]	−78/2 rt/36	6	85 (13)[g]	1.1:1.0
c-$C_6H_{11}CHO$	1.0	Me_3SiOTf	−78/3	7	91	
	1.0	Me_3SiI	−78/3	7	89	
c-$C_6H_{11}CH(OMe)_2$	1.0	Me_3SiOTf	−78/3	7	89	
	1.0	Me_3SiI	−78/3	7	84	
PhCHO	1.0	Me_3SiOTf	−78/3	8[i]	86	
	1.0	Me_3SiI	−78/3	8	86	
$PhCH(OMe)_2$	1.0	Me_3SiOTf	−78/3	8	82	
	1.0	Me_3SiI	−78/3	8	80	

[a] Refers to mmol substrate. Approximaately 1.3 equiv of **2** used in all cases. All reactions were run ca. 1 M. [b] Isolated, purified yield. [c] Determined by GC and HPLC. [d] Known compound; see ref 3a. [e] Reference 10a. [f] Reference 10. [g] Parentheses refers to percent recovered starting ketone. [h] Di-*tert*-butylpyridine (2%) present during reaction. [i] Known compound; see ref 4a. [j] rt = room temperature.

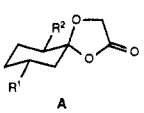
chemistry of diastereomeric spiro-fused dioxolanones has been assigned.

Results and Discussion

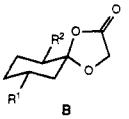
Under the conditions of Farines[3a] (catalytic boron trifluoride or *p*-toluenesulfonic acid), reaction of several ketones and aldehydes with glycolic acid either failed to afford the desired dioxolanones or proceeded in poor yield. However, treatment of a mixture of the desired carbonyl compound and trimethylsilyl (trimethylsilyloxy)acetate (**2**)[5] with a catalytic amount of trimethylsilyl trifluoromethanesulfonate (Me_3SiOTf),[6,11] or trimethylsilyl iodide (Me_3SiI) provided dioxolanones **3**–**8** in good to excellent yield (Table I).[7] The reaction was also successful with ketals and acetals. In contrast to the literature report,[3a] these dioxolanones were quite stable and could be distilled or chromatographed.



3 $R^1, R^2 = -(CH_2)_5-$
7 $R^1 = H$; $R^2 = c\text{-}C_6H_{11}$
8 $R^1 = H$; $R^2 = Ph$



A



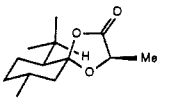
B

4 $R^1 = Me$; $R^2 = H$
5 $R^1 = Me$; $R^2 = i\text{-}Pr$
6 $R^1 = Me$; $R^2 = CMe_2Ph$

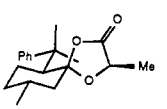
Two diastereomers **A** and **B** were formed in almost equal amounts where possible (compounds **4**–**6**). Easy chromatographic separation of the diastereomeric dioxolanones was crucial to our endeavors into asymmetric synthesis.[2] Although the diastereomeric dioxolanones **4** and **5** were separated satisfactorily only by MPLC or HPLC, examination of several solvent systems led to the separation of the diastereomeric dioxolanones **6A** and **6B** by flash chromatography.[8]

The low stereoselectivity seen for **4**–**6** and the incomplete conversion to **6** suggested that these reactions were taking place under thermodynamically controlled conditions. Indeed, resubjection of pure dioxolanone **6A** to the action of catalytic Me_3SiOTf led to the same distribution of **6A**, **6B**, and 8-phenylmenthone as that reported in Table I. Increased reaction time and temperature, or the use of an excess amount of **2**, failed to increase the conversion to **6**. The reaction was unsuccessful with more crowded ketones (e.g., benzophenone, fenchone, and camphor) and proceeded in low yield with α,β-unsaturated ketones (e.g., cyclohexenone).

The stereochemistry of the dioxolanones was next investigated. Since spectroscopic techniques did not allow direct assignment of stereochemistry, chemical transformation and X-ray crystallography were utilized. Methylation of the enolates derived from **5B** and **6B** (LDA, THF, −78 °C; then MeI) gave compounds **9** and **10** as the



9



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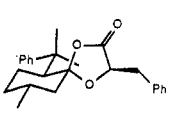

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major diastereomers, respectively. Ethanolysis (EtOH, HCl, reflux) of each provided (*R*)-ethyl lactate, thus indicating that the configurations of **5B** and **6B** are consistent with the structures shown. Benzylation ($PhCH_2Br$) of the enolate of **6B** provided the crystalline dioxolanone **11**, whose stereochemistry was confirmed by X-ray crystallography.[2]

Hence, a practical method has been developed for the preparation of dioxolanones formally derived from glycolic acid. Further, the stereochemistry of the diastereomeric spiro-fused dioxolanones has been assigned. Studies involving the synthesis of related heterocycles and their use

(10) Prepared by chromic oxidation[10a] of Corey's 8-phenylmenthol.[10b,c] (a) Brown, H. C.; Carg, C. P. *J. Am. Chem. Soc.* **1961**, *83*, 2952. (b) Corey, E. J.; Ensley, H. E. *J. Am. Chem. Soc.* **1975**, *97*, 6908. See also: (c) Whitesell, J. K.; Liu, C. L.; Buchanan, C. M.; Chen, H.-H.; Minton, M. A. *J. Org. Chem.* **1986**, *51*, 551.

(11) In the accompanying note, Professor T. R. Hoye and co-workers demonstrate a similar method for the synthesis of 1,3-dioxolan-4-ones and 1,3-oxathiolan-4-ones. We thank Professor Hoye for sharing his results with us prior to publication.

in asymmetric synthesis are currently underway.

Experimental Section[9]

General Procedure for 1,3-Dioxolan-4-one Preparation. A solution of the carbonyl compound, acetal or ketal (1 equiv), in anhydrous dichloromethane (3 M) was added in a dropwise fashion to a solution of Me_3SiOTf or Me_3SiI (5%) and **2** (1.2–1.3 equiv) in dichloromethane at −78 °C (ca. 1 M final concentration). In some cases, di-*tert*-butylpyridine (2%) was also present. The solution was stirred at the temperature/time indicated in Table I. On occasion, additional Me_3SiOTf catalyst was necessary for complete reaction (TLC or GC analysis). Pyridine (1.3 equiv) was added, and the solution was poured into saturated aqueous sodium bicarbonate and extracted with ether. The organic phase was dried ($MgSO_4$), concentrated, and flash chromatographed[8] to provide the desired dioxolanone. Table I provides specific reaction scales and conditions.

Cyclohexanespiro-2′-(1′,3′-dioxolan-4′-one) (3):[3a] R_f 0.25 (SiO_2, 10% EtOAc/hexane); IR (film) 1807 (s), 1796 (s), 1674 (w), 1450 (w), 1300 (m), 1296 (m), 1222 (m), 1105 (m) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 4.33 (2 H, s), 1.90–1.60 (8 H, m), 1.54–1.40 (2 H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 112.7, 62.7, 34.8, 24.0, 22.6; MS (70 eV), *m/e* (relative intensity) 157 (3), 156 (M^+, 17), 113 (47), 98 (58), 70 (5), 69 (7), 56 (25), 55 (100), 54 (22), 43 (42).

(*R*)-3-Methylcyclohexanespiro-2′-(1′,3′-dioxolan-4′-one) (4A,B): R_f 0.24 (SiO_2, 10% EtOAc/hexane). Preparative HPLC or MPLC with 5% EtOAc/hexane provided **4A** and **4B** in a 1.1:1.0 ratio. The retention time of **4A** is longer than that of **4B**.

4A: $[\alpha]^{25}_D$ −18.86° (*c* 5.77, CH_2Cl_2); IR (film) 1804 (s), 1679 (m), 1457 (m), 1372 (m), 1228 (m), 1210 (m), 1165 (m) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 4.35 (2 H, s), 1.96–1.90 (2 H, m), 1.88–1.70 (3 H, m), 1.70–1.54 (2 H, m), 1.30 (1 H, t, *J* = 12.6 Hz), 0.95 (3 H, d, *J* = 6.5 Hz), 0.92–0.85 (1 H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.0, 113.5, 63.0, 43.6, 35.0, 33.1, 29.8, 22.4, 21.7; MS (70 eV), *m/e* (relative intensity) 171 (3), 170 (M^+, 8), 127 (69), 113 (19), 94 (25), 69 (100), 56 (61), 55 (64), 43 (26), 42 (61). See **4B** for combustion analysis.

4B: $[\alpha]^{25}_D$ −12.39° (*c* 5.73, CH_2Cl_2); IR (film) 1808 (m), 1448 (m), 1358 (m), 1290 (s), 1238 (s), 1118 (m) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 4.31 (2 H, s), 1.92–1.88 (2 H, m), 1.80–1.55 (5 H, m), 1.40 (1 H, t, *J* = 12.5 Hz), 0.96 (3 H, d, *J* = 6.5 Hz), 0.93–0.86 (1 H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.2, 113.4, 63.1, 43.0, 34.3, 33.0, 29.5, 22.1, 21.8; MS (70 eV), *m/e* (relative intensity) 171 (4), 170 (M^+, 8), 127 (70), 113 (19), 94 (25), 69 (100), 56 (62), 55 (64), 43 (26), 42 (63). Anal. Calcd for $C_9H_{14}O_3$: C, 63.51; H, 8.29. Found (for the mixture of isomers **4A** and **4B**): C, 63.42; H, 8.21.

(2*S*,5*R*)-2-(1-Methylethyl)-5-methylcyclohexanespiro-2′-(1′,3′-dioxolan-4′-one) (5A,B): R_f 0.18 (SiO_2, 5% EtOAc/hexane); bp 105–106 °C/9 mm Hg. Preparative HPLC or MPLC with 5% EtOAc/hexane provided **5A** and **5B** in a 1.1:1.0 ratio. The retention time of **5A** is longer than that of **5B**.

5A: $[\alpha]^{25}_D$ −36.26° (*c* 4.70, $CHCl_3$); IR (film) 1790 (s), 1445 (w), 1300 (m), 1275 (m), 1210 (m), 1105 (m) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 4.32 (2 H, s), 2.00 (1 H, dt, *J* = 6.9, 2.2 Hz), 1.89 (1 H, ddd, *J* = 13.1, 3.5, 2.2 Hz), 1.80–1.60 (3 H, m), 1.58 (1 H, ddd, *J* = 12.9, 3.5, 2.3 Hz), 1.45 (1 H, dt, *J* = 12.9, 3.2 Hz), 1.34 (2 H, t, *J* = 12.8 Hz), 0.94 (3 H, d, *J* = 6.4 Hz), 0.93 (3 H, d, *J* = 7.0 Hz), 0.88 (3 H, d, *J* = 6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.4, 115.8, 63.5, 48.8, 44.8, 34.0, 30.0, 24.8, 23.1, 22.8, 21.6, 18.5, MS (70 eV), *m/e* (relative intensity) 212 (M^+, 7), 197 (3), 153 (8), 136 (11), 127 (100), 112 (11), 99 (15), 69 (48), 55 (17), 41 (19). See **5B** for combustion analysis.

5B: $[\alpha]^{25}_D$ −0.77° (*c* 5.18, $CHCl_3$); IR (film) 1790 (s), 1450 (m), 1225 (s), 1155 (m) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 4.37 (2 H, s), 2.09 (1 H, dt, *J* = 7.0, 1.5 Hz), 1.91 (1 H, ddd, *J* = 13.3, 3.5, 2.1 Hz), 1.86–1.60 (4 H, m), 1.50–1.40 (2 H, m), 1.27 (1 H, t, *J* = 12.6 Hz), 0.95 (3 H, d, *J* = 7.0 Hz), 0.92 (3 H, d, *J* = 6.5 Hz), 0.85 (3 H, d, *J* = 7.0 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.0, 115.6, 63.5, 49.4, 45.8, 34.1, 29.9, 24.8, 23.2 (2 C's), 21.6, 18.2; MS (70 eV), *m/e* (relative intensity) 212 (M^+, 6), 197 (3), 153 (8), 136 (11), 127 (100), 112 (11), 99 (16), 69 (55), 55 (20), 41 (22). Anal. Calcd for $C_{12}H_{20}O_3$: C, 67.89; H, 9.50. Found (for the mixture of isomers **5A** and **5B**): C, 68.12; H, 9.44.

(2*S*,5*R*)-2-(1-Methyl-1-phenylethyl)-5-methylcyclohexanespiro-2′-(1′,3′-dioxolan-4′-one) (6A,B). 6A: R_f 0.25 (SiO_2, 5:15:80 MeCN/PhH/hexane); $[\alpha]^{25}_D$ −1.14° (*c* 3.95, $CHCl_3$); IR (film) 1807 (s), 1795 (s), 1455 (m), 1445 (m), 1318 (m), 1281 (s), 1217 (s), 1112 (s) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.35–7.14 (5 H, m), 4.01, 4.16 (2 H, AB, *J* = 15.4 Hz), 2.16–2.08 (1 H, m), 1.77–1.56 (5 H, m), 1.55–1.38 (1 H, m), 1.41 (3 H, s), 1.39 (3 H, s), 1.31 (1 H, t, *J* = 13.6 Hz), 0.88 (3 H, d, *J* = 6.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.4, 150.5, 127.9, 125.8, 125.5, 116.0, 62.5, 52.5, 46.1, 40.3, 34.4, 30.0, 29.1, 26.8, 25.1, 21.3; MS (70 eV), *m/e* (relative intensity) 288 (M^+, 2), 230 (0.2), 197 (0.4), 170 (12), 120 (10), 119 (100), 111 (5), 91 (20), 41 (12). Anal. Calcd for $C_{18}H_{24}O_3$: C, 74.97; H, 8.39. Found: C, 74.83; H, 8.64.

6B: R_f 0.29 (SiO_2, 5:15:80) MeCN/PhH/hexane); $[\alpha]^{25}_D$ + 38.68° (*c* 3.33, $CHCl_3$); IR (film) 1800 (s), 1455 (m), 1446 (m), 1314 (m), 1236 (s), 1225 (s), 1166 (m) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.34–7.13 (5 H, m), 3.69, 4.07 (2 H, AB, *J* = 15.4 Hz), 2.07 (1 H, dd, *J* = 12.8, 3.6 Hz), 1.80–1.70 (4 H, m), 1.70–1.50 (2 H, m), 1.40 (3 H, s), 1.35 (3 H, s), 1.26 (1 H, t, *J* = 13.4 Hz), 0.87 (3 H, d, *J* = 6.4 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.9, 150.6, 127.8, 125.7, 125.4, 116.2, 62.4, 53.0, 47.5, 40.1, 34.6, 29.9, 28.6, 27.1, 25.9, 21.4; MS (70 eV), *m/e* (relative intensity) 288 (M^+, 2), 230 (0.2), 197 (0.4), 170 (12), 120 (10), 119 (100), 111 (5), 91 (21), 41 (12). Anal. Calcd for $C_{18}H_{24}O_3$: C, 74.97; H, 8.39. Found: C, 74.88; H, 8.25.

2-Cyclohexyl-1,3-dioxolan-4-one (7): R_f 0.29 (SiO_2, 10% EtOAc/hexane); IR (film) 1808 (s), 1451 (m), 1396 (w), 1351 (w), 1322 (m), 1218 (s), 1198 (s), 1080 (w) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.36 (1 H, d, *J* = 4.72 Hz), 4.21, 4.31 (2 H, AB, *J* = 15.1 Hz), 1.90–1.60 (6 H, m), 1.35–1.05 (5 H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.4, 109.2, 63.8, 41.7, 26.0, 25.9, 25.2; MS (70 eV) *m/e* (relative intensity) 171 (0.2), 170 (M^+, 0.2), 111 (55), 95 (11), 87 (92), 83 (87), 67 (30), 59 (61), 55 (100), 41 (82), 39 (47). Anal. Calcd for $C_9H_{14}O_3$: C, 63.51; H, 8.29. Found: C, 63.34; H, 8.31.

2-Phenyl-1,3-dioxolan-4-one (8):[4a] R_f 0.17 (SiO_2, 10% EtOAc/hexane); IR (film) 1811 (s), 1479 (m), 1395 (w), 1319 (w), 1225 (s), 1207 (m), 1188 (m), 1071 (w) cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.55–7.25 (5 H, m), 6.45 (1 H, s), 4.34, 4.44 (2 H, AB, *J* = 15.0 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.0, 148.8, 130.4, 128.6, 126.3, 105.1, 64.0; MS (70 eV), *m/e* (relative intensity) 165 (5), 164 (M^+, 41), 163 (15), 119 (13), 106 (77), 105 (100), 90 (42), 78 (82), 77 (87), 63 (25), 51 (85), 39 (37). Anal. Calcd for $C_9H_8O_3$: C, 65.85; H, 4.91. Found: C, 65.76; H, 4.90.

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A Convenient Synthesis of Cyclopenta[*cd*]pyrene[1]

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Cyclopenta[*cd*]pyrene (CPP, **6**), a non bay region polycyclic aromatic hydrocarbon of widespread environmental distribution,[2-4] has been shown to be carcinogenic to mice[5] and a very potent bacterial mutagen.[6] These findings have

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