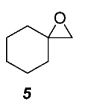
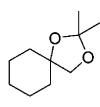
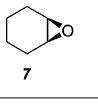
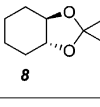
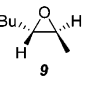
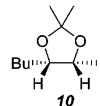
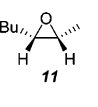
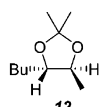
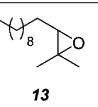
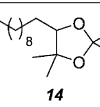


TABLE 2. Conversion of Di- and Trisubstituted Epoxides to Acetonides^a

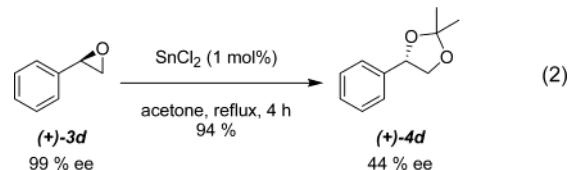
Entry	Epoxide	Time	Product	% Yield ^b
1		1		57 ^c
2		7		64 ^d
3		3		50
4		1		45
5		1		81

^a Epoxide was treated with anhydrous SnCl₂ (1 mol %) in refluxing acetone for the time period indicated. ^b Isolated yield of purified material. ^c 1-Cyclohexenylmethanol also isolated (16%). ^d Reaction conducted at room temperature.

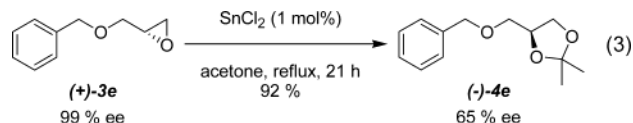
epoxides **3** were then evaluated using the optimized reaction protocol (Table 1). The yields of dioxolanes **4** range from good to excellent for all substrates; the yield of dioxolane **4g** is lower due to product volatility. Common alcohol protecting groups such as methoxymethyl (MOM) ether (**3i**), benzyl ether (**3j**), *tert*-butyldimethylsilyl (TBS) ether (**3k**), and *p*-methoxybenzyl (PMB) ether (**3l**) are all compatible with the reaction conditions. Attempts to prepare 1,3-dioxolanes derived from other ketones (3-pentanone, cyclohexanone) were unsuccessful, however. Apparently the reaction is sensitive to steric hindrance with respect to the ketone reaction partner.

Di- and trisubstituted epoxides also participate in the reaction to yield the corresponding dioxolanes (Table 2). Spiroepoxide **5** provided the corresponding dioxolane **6** in moderate yield along with 1-cyclohexenylmethanol from elimination, although no cyclohexanecarboxaldehyde, another likely rearrangement product, was detected in this reaction. Cyclohexene oxide (**7**) provided only *trans*-1,2-cyclohexanediol acetonide (**8**) in moderate yield, but the reaction had to be done at room temperature to prevent the formation of an unidentified side product. Reaction of *trans*-2,3-epoxyheptane (**9**) gave only *cis*-acetonide **10**, and *cis*-2,3-epoxyheptane (**11**) provided only the *trans*-acetonide **12**, respectively.¹⁸ The reaction of epoxide **9** took 3 h to reach completion, presumably due to the increased steric congestion that occurs after the epoxide opening. Epoxide **11** was completely consumed in less than 1 h, however, since steric congestion is released upon epoxide opening in this case. Trisubstituted epoxide **13** also reacted smoothly under the standard conditions to yield acetonide **14** in good yield.

Reaction of optically pure epoxides under the standard conditions (acetone, 1 mol % anhydrous SnCl₂, reflux) resulted in significant loss of optical activity in the products. (*R*)-(+)-Styrene oxide [(+)-**3d**, 99% ee] produced acetonide (+)-**4d** in 94% yield, but the optical purity had eroded to 44% ee (eq 2).¹⁹ Conducting this reaction at



lower temperature (−78 °C to room temperature) produced (+)-**4d** in a slightly improved 55% ee. The enantiomeric excess of the product did not change during the course of the reaction, however, nor did the enantiomeric excess change after extended treatment of the product with SnCl₂ in refluxing acetone. To determine to what extent an S_N1-type epoxide opening was occurring, we next examined epoxides that were not electronically biased toward formation of a resonance-stabilized cation intermediate. (*S*)-(+)-Benzylglycidyl ether [(+)-**3e**, 99% ee] led to acetonide (−)-**4e** in 92% yield, but the optical purity had eroded to 65% ee (eq 3).^{19,20} The major



enantiomer produced in the reaction of both (+)-**3d** and (+)-**3e** results from attack of acetone at the more substituted epoxide position with inversion of stereochemistry. Treatment of (*R*)-(+)-1,2-epoxynonane under the standard conditions formed the corresponding acetonide with only a 6% enantiomeric excess (not shown). This result shows that acetone attacks both epoxide positions with nearly equal frequency when the oxirane is not electronically biased. Thus the high *diastereoselectivity* of this reaction (Table 2, entries 2–4) coupled with the erosion of optical purity from single enantiomer epoxides (eqs 2 and 3) supports an S_N2-like opening of the epoxide by acetone that occurs with low regioselectivity.^{9,21}

In summary, we have found that anhydrous SnCl₂ is an efficient catalyst for the conversion of mono-, di-, and trisubstituted epoxides directly to their 2,2-dimethyl-1,3-dioxolane derivatives. The reaction is compatible with a

(18) The relative stereochemistry of the dioxolane products was confirmed by analysis of the ¹H–¹H coupling constants and their comparison to literature values: Pihlaja, K.; Nummelin, H.; Klika, K.; Czombos, J. *Magn. Reson. Chem.* **2001**, *39*, 657–671.

(19) Enantiomeric excesses were determined by GC analysis on a Cyclosil-B chiral column. The absolute configuration of the major enantiomer was determined by measuring the optical rotation of the product and correlating it to literature values. For (+)-**4d**: Schaus, S. E.; Brandes, B. D.; Larrow, J. F.; Tokunaga, M.; Hansen, K. B.; Gould, A. E.; Furrow, M. E.; Jacobsen, E. N. *J. Am. Chem. Soc.* **2002**, *124*, 1307–1315. For (−)-**4e**: Fondy, T. P.; Pero, R. W.; Karker, K. L.; Ghangas, G. S.; Batzold, F. H. *J. Med. Chem.* **1974**, *17*, 697–702.

(20) A similar decrease in enantiomeric excess with this substrate has been observed with other Lewis acids. See ref 6.

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variety of common protecting groups but leads to varying degrees of racemization with optically pure epoxides.

Experimental Section²²

General Procedure. Preparation of 4-Hexyl-2,2-dimethyl-1,3-dioxolane (4a).¹² Anhydrous tin(II) chloride (98%) (9.9 mg, 0.05 mmol) was placed in a two-necked flask (50 or 100 mL) equipped with a magnetic stir bar and a reflux condenser. The system was sequentially evacuated and filled with argon six times. Dry acetone (25 mL) was added through the septum and stirring was started. 1,2-Epoxyoctane (**3a**) (0.76 mL, 5.0 mmol) was added dropwise and the solution was heated to reflux and maintained at that temperature until GC analysis indicated complete consumption of the epoxide (typically 1 h). The acetone was removed by rotary evaporation and the residue was taken up in CH₂Cl₂ (20 mL), and 10% NaOH solution (10 mL) and water (5 mL) were added. The layers were separated, and the aqueous layer was extracted CH₂Cl₂ (2 × 5 mL). The combined organic layers were dried over Na₂SO₄. After solvent removal via rotary evaporation, the crude product was purified by MPLC (75:1 hexanes/ethyl acetate) to give **4a** (0.919 g, 97%) as a colorless oil. ¹H NMR (CDCl₃, 300 MHz): δ 4.05 (m, 2H), 3.49 (m, 1H), 1.7–1.2 (m, 10H), 1.40 (s, 3H), 1.35 (s, 3H), and 0.88 (t, *J* = 7.0, 3H). ¹³C NMR (CDCl₃, 75 MHz): δ 108.4, 76.1, 69.5, 33.6, 31.7, 29.4, 27.0, 25.8 (2C), 22.6, and 14.1.

4-Decyl-2,2-dimethyl-1,3-dioxolane (4b). Anhydrous SnCl₂ (9.8 mg, 0.05 mmol), acetone (25 mL), and epoxide **3b** (1.10 mL, 5 mmol) yielded **4b** (1.117 g, 92%) after MPLC (30:1 hexanes/ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 4.05 (m, 2H), 3.49 (m, 1H), 1.7–1.2 (m, 14H), 1.40 (s, 3H), 1.36 (s, 3H), and 0.88 (t, *J* = 7.0, 3H). ¹³C NMR (CDCl₃, 75 MHz): δ 108.2, 75.9, 69.3, 33.5, 31.8, 29.6, 29.5, 29.5, 29.4, 29.3, 26.8, 25.7, 25.6, 22.6, and 14.0. Anal. Calcd for C₁₅H₃₀O₂: C, 74.32; H, 12.47. Found: C, 73.97; H, 12.20.

4-Allyloxymethyl-2,2-dimethyl-1,3-dioxolane (4c).⁸ Anhydrous SnCl₂ (9.8 mg, 0.05 mmol), acetone (25 mL), and epoxide **3c** (0.59 mL, 5 mmol) yielded **4c** (0.818 g, 95%) after MPLC (15:1 hexanes/ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 5.89 (ddt, *J* = 17.3, 10.3, 5.7, 1H), 5.26 (ddt, *J* = 17.3, 1.8, 1.8, 1H), 5.18 (ddt, *J* = 10.3, 1.2, 1.2, 1H), 4.28 [tt (app pentet), *J* = 6.0, 6.0, 1H], 4.04 (m, 3H), 3.73 (dd, *J* = 8.2, 6.4, 1H), 3.48 (m, 2H), 1.43 (s, 3H), and 1.37 (s, 3H). ¹³C NMR (CDCl₃, 75 MHz): δ 134.2, 117.0, 109.1, 74.5, 72.3, 70.9, 66.6, 26.6, and 25.3.

2,2-Dimethyl-4-phenyl-1,3-dioxolane (4d).⁵ Anhydrous SnCl₂ (9.6 mg, 0.05 mmol), acetone (25 mL), and epoxide **3d** (0.57 mL, 5 mmol) yielded **4d** (0.803 g, 90%) after MPLC (50:1 hexanes/ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 7.33 (m, 5H), 5.06 (dd, *J* = 8.0, 6.2, 1H), 4.29 (dd, *J* = 8.0, 6.2, 1H), 3.70 (t, *J* = 8.0, 1H), 1.55 (s, 3H), and 1.49 (s, 3H). ¹³C NMR (CDCl₃, 75 MHz): δ 138.8, 128.2, 127.7, 125.9, 109.4, 77.8, 71.5, 26.5, and 25.9.

2,2-Dimethyl-4-[(phenylmethoxy)methyl]-1,3-dioxolane (4e).²³ Anhydrous SnCl₂ (9.5 mg, 0.05 mmol), acetone (25 mL), and epoxide **3e** (0.76 mL, 5 mmol) yielded **4e** (0.886 g, 80%) after MPLC (12:1 hexanes/ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 7.32 (m, 5H), 4.57 (AB, *J* = 12.3, 2H), 4.29 [dddd (app tt), *J* = 6.2, 5.6, 1H], 4.05 (dd, *J* = 8.2, 6.2), 3.73 (dd, *J* = 8.2, 6.5, 1H), 3.55 (dd, *J* = 10.0, 5.6, 1H), 3.46 (dd, *J* = 10, 5.6, 1H), 1.42 (s, 3H), and 1.36 (s, 3H). ¹³C NMR (CDCl₃, 75 MHz): δ 137.6, 128.1, 127.4 (2C), 109.1, 74.5, 73.3, 70.8, 66.6, 26.6, and 25.3.

4-(3-Butenyl)-2,2-dimethyl-1,3-dioxolane (4f).²⁴ Anhydrous SnCl₂ (9.7 mg, 0.05 mmol), acetone (25 mL), and epoxide **3f** (0.56 mL, 5 mmol) yielded **4f** (0.556 g, 71%) after MPLC (30:1 hexanes/ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 5.81 (ddt, *J* = 17.0, 10.3, 6.5, 1H), 5.03 (ddt, *J* = 17.0, 1.8, 1.8, 1H), 4.97

(ddt, *J* = 10.0, 1.8, 1.2, 1H), 4.06 (m, 2H), 3.51 (m, 1H), 2.13 (m, 2H), 1.75 (m, 1H), 1.59 (m, 1H), 1.40 (s, 3H), and 1.35 (s, 3H). ¹³C NMR (CDCl₃, 75 MHz): δ 137.5, 114.7, 108.4, 75.3, 69.2, 32.7, 29.8, 26.8, and 25.6.

4-Vinyl-2,2-dimethyl-1,3-dioxolane (4g).⁹ Anhydrous SnCl₂ (73 mg, 0.39 mmol), acetone (80 mL), and butadiene monoxide (**3g**) (3.14 mL, 39 mmol) yielded **4g** as a colorless oil (2.401 g, 48%) after distillation (bp 125 °C). ¹H NMR (CDCl₃, 300 MHz): δ 5.82 (ddd, *J* = 17.3, 10.3, 7.3, 1H), 5.34 (ddd, *J* = 17.3, 1.5, 1.5, 1H), 5.21 (ddd, *J* = 10.3, 1.5, 0.9, 1H), 4.49 (dddd, *J* = 8.2, 7.3, 6.2, 1.5, 0.9, 1H), 4.10 (dd, *J* = 6.2, 2.0, 1H), 3.60 (dd, *J* = 8.2, 6.2, 1H), 1.43 (s, 3H), and 1.40 (s, 3H). ¹³C NMR (CDCl₃, 75 MHz): δ 135.6, 117.6, 109.0, 77.2, 69.1, 26.5, and 25.7.

4-Cyclohexyl-2,2-dimethyl-1,3-dioxolane (4h). Anhydrous SnCl₂ (9.6 mg, 0.05 mmol), acetone (25 mL), and epoxide **3h** (0.623 g, 5 mmol) yielded **4h** as a colorless oil (0.575 g, 63%) after MPLC (19:1 hexanes: ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 3.97 (dd, *J* = 7.6, 5.8, 1H), 3.78 (dd, *J* = 7.6, 5.8, 1H), 3.60 (t, *J* = 7.6, 1H), 1.90–0.98 (m, 11H), 1.39 (s, 3H), and 1.35 (s, 3H). ¹³C NMR (CDCl₃, 75 MHz): δ 108.2, 80.2, 67.6, 41.3, 29.4, 28.6, 26.6, 26.3, 25.8, 25.6, and 25.5. Anal. Calcd for C₁₁H₂₀O₂: C, 71.70; H, 10.94. Found: C, 71.54; H, 10.86.

4-(9-Methoxymethoxynonyl)-2,2-dimethyl-1,3-dioxolane (4i). Anhydrous SnCl₂ (14 mg, 0.07 mmol), acetone (25 mL), and epoxide **3i** (1.159 g, 5 mmol) yielded **4i** (1.101 g, 77%) after MPLC (15:1 hexanes/ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 4.60 (s, 2H), 4.05 (m, 2H), 3.50 (m, 3H), 3.38 (s, 3H), 1.7–1.5 (m, 2H), 1.40 (s, 3H), 1.35 (s, 3H), and 1.5–1.3 (m, 14H). ¹³C NMR (CDCl₃, 75 MHz): δ 108.5, 96.3, 76.1, 69.5, 67.8, 55.1, 33.6, 29.8, 29.7, 29.55, 29.51, 29.50, 27.0, 26.3, and 25.8 (2C). Anal. Calcd for C₁₆H₃₂O₄: C, 66.63; H, 11.18. Found: C, 66.39; H, 11.47.

4-(9-Benzyloxynonyl)-2,2-dimethyl-1,3-dioxolane (4j). Anhydrous SnCl₂ (9.5 mg, 0.05 mmol), acetone (25 mL), and epoxide **3j** (1.380 g, 5 mmol) yielded **4j** (1.120 g, 67%) after MPLC (12:1 hexanes/ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 7.3 (m, 5H), 4.5 (s, 2H), 4.0 (m, 2H), 3.45 (m, 3H), 1.7–1.5 (m, 2H), 1.40 (s, 3H), 1.35 (s, 3H), and 1.5–1.2 (m, 14H). ¹³C NMR (CDCl₃, 75 MHz): δ 138.5, 128.1, 127.4, 127.3, 108.4, 76.1, 72.8, 70.4, 69.5, 33.6, 29.8, 29.7, 29.5, 29.46(2C), 27.0, 26.2, and 25.8 (2C). Anal. Calcd for C₂₁H₃₄O₃: C, 75.41; H, 10.25. Found: C, 75.44; H, 10.37.

tert-Butyl-[9-(2,2-dimethyl-1,3-dioxolan-4-yl)nonyloxy]-dimethylsilane (4k). Anhydrous SnCl₂ (9.6 mg, 0.05 mmol), acetone (25 mL) and epoxide **3k** (1.50 g, 5 mmol) yielded **4k** (1.32 g, 73%) after MPLC (19:1 hexanes/ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 4.05 (m, 2H), 3.58 (t, *J* = 6.6, 2H), 3.50 (m, 1H), 1.7–1.4 (m, 2H), 1.40 (s, 3H), 1.35 (s, 3H), 1.4–1.2 (m, 14H), 0.85 (s, 9H), and 0.05 (s, 6H). ¹³C NMR (CDCl₃, 75 MHz): δ 108.4, 76.1, 69.5, 63.3, 33.6, 32.9, 29.7, 29.6, 29.5, 29.4, 27.0, 26.0 (2C), 25.8 (2C), 18.4, and –5.2. Anal. Calcd for C₂₀H₄₂O₃Si: C, 66.98; H, 11.80. Found: C, 66.66; H, 12.14.

4-[9-(4-Methoxybenzyloxy)nonyl]-2,2-dimethyl-1,3-dioxolane (4l). Anhydrous SnCl₂ (9.6 mg, 0.05 mmol), acetone (25 mL), and epoxide **3l** (1.82 g, 5.9 mmol) yielded **4l** (1.38 g, 64%) after MPLC (9:1 hexanes/ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 7.24 (m, 2H), 6.85 (m, 2H), 4.42 (s, 2H), 4.05 (m, 2H), 3.79 (s, 3H), 3.6–3.4 (m, 3H), 1.40 (s, 3H), 1.35 (s, 3H), and 1.7–1.2 (m, 16H). ¹³C NMR (CDCl₃, 75 MHz): δ 158.8, 130.6, 129.1, 113.6, 108.4, 76.1, 72.4, 70.1, 69.5, 55.2, 33.6, 29.8, 29.7, 29.5, 29.47 (2C), 27.0, 26.2, and 25.8 (2C). Anal. Calcd for C₂₂H₃₆O₄: C, 72.49; H, 9.95. Found: C, 72.36; H, 10.20.

2,2-Dimethyl-1,3-dioxaspiro[4.5]decane (6).²⁵ Anhydrous SnCl₂ (9.5 mg, 0.05 mmol), acetone (25 mL), and epoxide **5** (0.56 g, 5 mmol) yielded **6** as a colorless oil (0.488 g, 57%) along with (1-cyclohexenyl)methanol²⁶ (0.137 g, 16%) after MPLC (19:1 hexanes/ethyl acetate). Characterization data for **6**: ¹H NMR (CDCl₃, 300 MHz): δ 3.76 (s, 2H), 1.8–1.5 (m, 5H), 1.39 (s, 6H), and 1.4–1.2 (m, 5H). ¹³C NMR (CDCl₃, 75 MHz): δ 108.3, 80.9, 73.1, 36.5, 27.3, 25.2, and 23.7.

(22) For general experimental considerations, see Supporting Information.

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Hexahydro-2,2-dimethyl-trans-1,3-benzodioxole (8).²⁷ Anhydrous SnCl₂ (8.7 mg, 0.05 mmol), acetone (25 mL), and epoxide **7** (0.51 mL, 5 mmol) yielded **8** (0.498 g, 64%) after MPLC (19:1 hexanes/ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 3.27 (m, 2H), 2.10 (m, 2H), 1.80 (m, 2H), 1.5–1.3 (m, 8H), and 1.3–1.2 (m, 2H). ¹³C NMR (CDCl₃, 75 MHz): δ 107.8, 79.8, 28.6, 26.7, and 23.5.

cis-4-Butyl-2,2,5-trimethyl-1,3-dioxolane (10). Anhydrous SnCl₂ (9.5 mg, 0.05 mmol), acetone (25 mL), and epoxide **9** (0.564 g, 4.9 mmol) yielded **10** (0.426 g, 50%) after MPLC (50:1 hexanes/ethyl acetate). An analytical sample was prepared by distillation (bp 72–73 °C, 15 mmHg). ¹H NMR (CDCl₃, 300 MHz): δ 4.22 (dq, *J* = 6.2, 6.2, 1H), 4.02 (m, 1H), 1.6–1.2 (m, 6H), 1.44 (s, 3H), 1.33 (s, 3H), 1.14 (d, *J* = 6.4, 3H), and 0.91 (t, *J* = 7.0, 3H); ¹³C NMR (CDCl₃, 75 MHz): δ 106.9, 77.9, 73.6, 29.4, 28.5, 28.4, 25.7, 22.6, 15.5, and 13.9. Anal. Calcd for C₁₀H₂₀O₂: C, 69.72; H, 11.70. Found: C, 69.64; H, 11.92.

trans-4-Butyl-2,2,5-trimethyl-1,3-dioxolane (12). Anhydrous SnCl₂ (9.5 mg, 0.05 mmol), acetone (25 mL), and epoxide **11** (0.570 g, 5.0 mmol) yielded **12** (0.390 g, 45%) after MPLC (50:1 hexanes/ethyl acetate). An analytical sample was prepared by distillation (bp 62 °C, 16 mmHg). ¹H NMR (CDCl₃, 300 MHz): δ 3.70 (dq, *J* = 8.2, 6.1, 1H), 3.51 (m, 1H), 1.6–1.2 (m, 6H), 1.39 (s, 3H), 1.38 (s, 3H), 1.24 (d, *J* = 6.1, 3H), and 0.91 (t,

J = 7.0 Hz, 3H). ¹³C NMR (CDCl₃, 75 MHz): δ 107.4, 82.4, 76.7, 32.0, 28.3, 27.2 (2C), 22.9, 17.6, and 13.9. Anal. Calcd for C₁₀H₂₀O₂: C, 69.72; H, 11.70. Found: C, 69.65; H, 11.38.

2,2,4,4-Tetramethyl-5-nonyl-1,3-dioxolane (14). Anhydrous SnCl₂ (6.0 mg, 0.03 mmol), acetone (10 mL), and epoxide **13** (0.479 g, 2.42 mmol) yielded **14** (0.500 g, 81%) after MPLC (30:1 hexanes/ethyl acetate). ¹H NMR (CDCl₃, 300 MHz): δ 3.67 (dd, *J* = 8.8, 3.5, 1H), 1.6–1.2 (m, 16H), 1.42 (s, 3H), 1.33 (s, 3H), 1.27 (s, 3H), 1.09 (s, 3H), and 0.88 (t, *J* = 7.0, 3H). ¹³C NMR (CDCl₃, 75 MHz): δ 106.2, 83.3, 80.0, 31.9, 29.8, 29.6, 29.5, 29.4, 29.3, 28.5, 27.0, 26.8, 26.0, 22.8, 22.7, and 14.1. Anal. Calcd for C₁₆H₃₂O₂: C, 74.94; H, 12.58. Found: C, 74.86; H, 12.80.

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Supporting Information Available: Experimental procedures and characterization data for epoxides **3h–l** and **13**. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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