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Supporting Information

Grignard Reactions of 4-Substituted-2-keto-1,3-dioxanes: Highly Diastereoselective Additions Controlled by a Remote Alkyl Group

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Experimental Section

General. Compound numbers, Tables, and references refer to those contained in the main body of the paper. The concentration of commercial solutions of Grignard reagents was determined immediately prior to use.[13] Gas chromatograpic (GC) analyses were performed on a 15 m x 0.2 mm x 0.33 μm OV-17 capillary column; optical rotations were determined in a 10 cm cell on a JASCO DIP-1000 digital polarimeter.

2-Phenylpropenal diethyl acetal. A suspension of 5.01 g (14.1 mmol) of methyltriphenylphosphonium bromide and 1.85 g (14.5 mmol) of potassium *t*-butoxide in 100 mL of THF was stirred at room temperature for 30 min, 2.7 mL (13.5 mmol) of 2,2-diethoxyacetophenone was added, and the reaction mixture was stirred at room temperature

for 7 h. The reaction mixture was then filtered through a pad of Celite and concentrated by rotary evaporation. Kugelrohr distillation of the residue gave 1.84 g (65 %) of a colorless liquid: bp (bath temp) 200 °C (1 mm) [lit.[14] bp 70 °C (0.5 mm)], ^{1}H NMR δ 1.24 (t, *J* = 7.1 Hz, 6H), 3.54-3.71 (m, 4H), 5.29 (s, 1H), 5.59 (s, 2H), 7.30-7.38 (m, 3H), 7.56 (d, *J* = 3.9 Hz, 2H); ^{13}C NMR δ 15.56, 61.78, 102.26, 116.06, 127.21, 127.98, 128.34, 138.96, 145.41.

***cis*-2-Acetyl-4-methyl-1,3-dioxane (1).** A round-bottomed flask, equipped with a short path distillation head, was charged with 12 mL (100 mmol) of pyruvaldehyde dimethyl acetal, 9.0 mL (100 mmol) of 1,3-butanediol and 5 drops of concentrated sulfuric acid. The reaction mixture was gradually warmed and ethanol was removed by distillation. Heating was discontinued when the still-head temperature dropped below the boiling point of ethanol, the reaction mixture was then allowed to cool to room temperature, ~1 g of sodium carbonate was added, and the mixture was stirred for 1 h to neutralize the acid catalyst. The mixture was then diluted with 100 mL of methylene chloride and washed successively with three 100-mL portions of aqueous sodium carbonate, water and brine, dried over Na_2SO_4, and concentrated to give an oil. Kugelrohr distillation of this material afforded 7.12 g (49%) of an approximately 4:1 mixture of cis and trans isomers of the title compound as a colorless oil: bp (bath temp) 60-80 °C (2.5 mm). The cis-isomer was isolated by column chromatography on silica gel (5 % EtOAc–hexanes, R_f = 0.18): ^{1}H NMR δ 1.30 (d, *J* = 6.2 Hz, 3H), 1.53 (dq, *J* = 13.5, J = 2.3 Hz, 1H), 1.77 (qd, *J* = 11.2, J = 5.0 Hz, 1H), 2.25 (s, 3H), 3.79-3.88 (m, 2H), 4.21 (dd, *J* = 11.5, J = 4.5 Hz, 1H), 4.77 (s, 1H); ^{13}C NMR δ 21.56, 24.90, 32.82, 66.73, 73.43, 100.35, 202.11.

***cis*-2-Benzoyl-4-methyl-1,3-dioxane (2). Method A** A round-bottomed flask, equipped with a short path distillation head, was charged with 20.25 g (97 mmol) of 2,2-diethoxyacetophenone, 10.0 mL (0.11 mol) of 1,3-butanediol and 5 drops of concentrated sulfuric acid. The reaction mixture was gradually warmed and ethanol was removed by distillation. Heating was discontinued when the still-head temperature dropped below the boiling point of ethanol, the dark reaction mixture was then allowed to cool to room temperature, and 250 mL of diethyl ether was added in one portion. The resulting solution was washed successively with three 100-mL portions of aqueous sodium carbonate, water and brine, dried over Na_2SO_4, and concentrated to give an oil. Kugelrohr distillation of this material afforded 18.25 g (91 %) of a mixture of products consisting mainly of an approximately 4:1 mixture of cis and trans isomers of the title compound: bp (bath temp) 135 °C (2 mm). The cis-isomer was isolated by column chromatography on silica gel (5 % EtOAc – hexanes, R_f = 0.10): ^{1}H NMR δ 1.33 (d, J = 6.1 Hz, 3H), 1.58 (dq, *J* = 13.5, J = 2.3 Hz, 1H), 1.91 (qd, *J* = 12.6, J = 5.0 Hz, 1H), 3.90-4.00 (m, 2H), 4.30 (dd, *J* = 11.5, J = 4.9 Hz, 1H), 5.48 (s, 1H), 7.42-7.54 (m, 2H), 7.55-7.56 (m, 1H), 8.11-8.14 (m, 2H); ^{13}C NMR δ 21.91, 33.30, 67.32, 74.19, 100.77, 128.51, 130.15, 133.72, 1344.07, 191.49; HRMS (EI) calcd for $C_{12}H_{13}O_3$ (M–1) 205.0865, found 205.0870.

Method B: A round-bottomed flask, equipped with a short path distillation head, was charged with 1.98 g (9.60 mmol) of 2-phenylpropenal diethyl acetal, 820.4 mg (9.11 mmol) of 1,3-butanediol, 40 mL of cyclohexane and approximately 100 mg of pyridinium *p*-toluenesulfonate. The reaction mixture was heated until the temperature of the distillate reached 80 °C and heating was then discontinued. The reaction mixture was allowed to cool to room temperature, 1 g of sodium carbonate was added, the mixture was stirred for 1 h to

neutralize the acid catalyst, and the mixture was then concentrated by rotary evaporation. The residue was suspended in 100 mL of diethyl ether and washed successively with two 50-mL portions of aqueous sodium carbonate and water, dried over Na_2SO_4, and concentrated to give an oil. The oil was dissolved in 100 mL of methylene chloride, the solution was cooled to –78 °C, and ozone was bubbled through the solution until a blue color persisted. The reaction mixture was flushed with nitrogen for 15 min to discharge excess ozone, 5 mL of dimethyl sulfide was added, and the mixture was allowed to warm and stir at room temperature for 12h. The resulting solution was washed successively with two 50-mL portions of aqueous sodium carbonate and water, dried over Na_2SO_4 and concentrated to give an oil. Column chromatography of this oil on silica gel (5% EtOAc – hexanes) afforded 1.09 g (58% for two-step conversion) of the title compound identical in all respects to that prepared by Method A.

2-Acetyl-4,4-dimethyl-1,3-dioxane (3). A round-bottomed flask, equipped with a Vigreux column and a short path distillation head, was charged with 6.00 g (50.8 mmol) of pyruvaldehyde dimethyl acetal, 5.29 g (50.8 mmol) of 3-methyl-1,3-butanediol,[15] approximately 100 mg of *p*-toluenesulfonic acid, and 150 mL of toluene. The reaction mixture was gradually warmed and the ethanol – toluene azeotrope was removed by distillation. Heating was discontinued when the still-head temperature dropped below the boiling point of the azeotrope, the reaction mixture was then allowed to cool to room temperature, ~1 g of sodium carbonate was added, and the mixture was stirred for 1 h to neutralize the acid catalyst. The reaction mixture was then washed with two 50-mL portions of aqueous sodium carbonate, two 50-mL portions of brine, dried over Na_2SO_4, and

concentrated to give an oil. Kugelrohr distillation of this material afforded 2.70 g (34%) of the title compound: bp (bath temp) 80 °C (2 mm); ^{1}H NMR δ 1.27 (s, 3H), 1.30-1.36 (m, 1H), 1.31 (s, 3H), 1.91 (td, *J* = 13.2, J = 5.5 Hz, 1 H), 2.17 (s, 3H), 3.89 (td, *J* = 11.8, J = 2.5 Hz, 1 H), 4.00 (m, 1H), 4.89 (s, 1H).

2-Benzoyl-4,4-dimethyl-1,3-dioxane (4). A round-bottomed flask, fitted with a Vigreux column and a short path distillation head, was charged with 7.98 g (38.3 mmol) of 2,2-diethoxyacetophenone, 3.55 g (34.1 mmol) of 3-methyl-1,3-butanediol,[15] approximately 100 mg of *p*-toluenesulfonic acid, and 150 mL of toluene. The reaction mixture was gradually warmed and the ethanol – toluene azeotrope was removed by distillation. Heating was discontinued when the still-head temperature dropped below the boiling point of the azeotrope, the reaction mixture was then allowed to cool to room temperature, ~1 g of sodium carbonate was added, and the mixture was stirred for 1 h to neutralize the acid catalyst. The reaction mixture was then washed with two 50-mL portions of aqueous sodium carbonate, two 50-mL portions of brine, dried over Na_2SO_4, and concentrated to give an oil. Kugelrohr distillation of this material followed by column chromatography on silica gel (5 % EtOAc – hexanes, R_f = 0.14) afforded 2.06 g (27%) of the title compound: bp (bath temp) 135 °C (2 mm); ^{1}H NMR δ 1.35 (s, 3H), 1.44-1.48 (m, 1H), 1.48 (3, 3H), 1.21 (td, *J* = 13.2, J = 5.6 Hz, 1 H), 4.03-4.09 (m, 1H), 4.14-4.19 (m, 1H), 5.68 (s, 1H), 7.42-7.46 (m, 2H), 7.54-7.58 (m, 1H), 8.13 (d, *J* = 7.2 Hz ,2H); ^{13}C NMR δ 21.35, 31.65, 36.18, 63.54, 73.41, 95.00, 128.52, 130.06, 133.55, 134.15, 192.01; HRMS (EI) calcd for $C_{13}H_{15}O_3$ (M–1) 219.1021, found 219.1020.

***cis*-2-Acetyl-4-*t*-butyl-1,3-dioxane (5).** A round-bottomed flask equipped with a short path distillation head was charged with 1.97 mL (14.1 mmol) of α-methyl-*trans*-cinnamaldehyde, 1.86 g (14.1 mmol) of 4,4-dimethyl-1,3-pentanediol,[16] 60 mL of cyclohexane and approximately 100 mg of pyridinium *p*-toluenesulfonate. The reaction mixture was heated until the temperature of the distillate reached 80 °C and heating was then discontinued. The reaction mixture was allowed to cool to room temperature, 1 g of sodium carbonate was added, the mixture was stirred for 1 h to neutralize the acid catalyst, and the mixture was then concentrated by rotary evaporation. The residue was taken up in 100 mL of diethyl ether and washed successively with two 50-mL portions of aqueous sodium carbonate and water, dried over Na_2SO_4, and concentrated to give an oil. The oil was dissolved in 150 mL of methylene chloride, the solution was cooled to –78 °C, and ozone was bubbled through the solution until a blue color persisted. The reaction mixture was flushed with nitrogen for 15 min to discharge excess ozone, 5 mL of dimethyl sulfide was added, and the mixture was allowed to warm and stir at room temperature for 12h. The resulting solution was washed successively with two 50-mL portions of aqueous sodium carbonate and water, dried over Na_2SO_4 and concentrated to give an oil. Chromatography of this oil on silica gel (2.5% EtOAc – hexanes, R_f = 0.13) afforded 1.04 g (40 % for two-step conversion) of the title compound: ^{1}H NMR δ 0.93 (s, 9H), 1.2-1.3 (m, 1H), 1.80 (qd, *J* = 12.1, J = 5.0 Hz, 1H), 2.23 (s, 3H), 3.35 (dd, *J* = 9.2, J = 2.3 Hz, 1H), 3.78 (td, *J* = 11.9, J = 2.5 Hz, 1H), 4.24 (m, 1H), 4.75 (s, 1H); ^{13}C NMR δ 24.86, 25.47, 25.49, 34.05, 62.58, 84.89, 100.78, 202.29.

***cis*-2-Benzoyl-4-*t*-butyl-1,3-dioxane (6) .** A round-bottomed flask equipped with a short path distillation head was charged with 1.18 g (5.70 mmol) of 2-phenylpropenal diethyl acetal, 0.751 g (5.68 mmol) of 4,4-dimethyl-1,3-pentanediol,[16] 30 mL of cyclohexane and approximately 100 mg of pyridinium *p*-toluenesulfonate. The reaction mixture was heated until the temperature of the distillate reached 80 °C and heating was then discontinued. The reaction mixture was allowed to cool to room temperature, 1 g of sodium carbonate was added, the mixture was stirred for 1 h to neutralize the acid catalyst, and the mixture was then concentrated by rotary evaporation. The residue was suspended in 50 mL of diethyl ether and washed successively with two 50-mL portions of aqueous sodium carbonate and water, dried over Na_2SO_4, and concentrated to give an oil. The oil was dissolved in 100 mL of methylene chloride, the solution was cooled to –78 °C, and ozone was bubbled through the solution until a blue color persisted. The reaction mixture was flushed with nitrogen for 15 min to discharge excess ozone, 5 mL of dimethyl sulfide was added, and the mixture was allowed to warm and stir at room temperature for 12h. The reaction mixture was then concentrated by rotary evaporation, the residue was taken up in 100 mL of diethyl ether and washed successively with two 50-mL portions of aqueous sodium carbonate and water, dried over Na_2SO_4 and concentrated to give an oil. Chromatography of this oil on silica gel (5% EtOAc – hexanes, R_f = 0.15) afforded 0.656 g (45% for the two-step conversion) of the title compound: ^{1}H NMR δ 0.94 (s, 9H), 1.50-1.53 (m, 1H), 1.98 (qd, *J* = 13.0, J = 4.9 Hz, 1H), 3.50 (dd, *J* = 11.5, J = 2.2 Hz, 1H), 3.92 (td, *J* = 12.1, J = 2.5 Hz, 1H), 4.35 (dt, *J* = 4.9, 1.2 Hz, 1H), 5.43 (s, 1H), 7.42-7.46 (m, 2H), 7.54-7.56 (m, 1H), 8.13 (dd, *J* = 7.2, J = 5.4 Hz, 2H); ^{13}C NMR δ 25.51 , 25.58, 33.99, 67.21, 85.66, 101.02,128.23, 130.05, 133.76, 191.49. Anal. Calcd for $C_{15}H_{20}O_3$: C, 72.55; H, 8.12. Found: C, 72.21; H, 8.45.

Grignard Reactions of 4-Substituted-2-keto-1,3-dioxanes. The following procedures are representative; yields and diastereoisomeric product composition are given in Table 1. Product compositions (d.r.) were determined by capillary GC analysis of the corresponding TMS derivatives on a 15 m x 0.2 mm x 0.33 μm OV-17 column. The following analysis procedure is representative: approximately 5 mg of the product mixture was dissolved in 1 mL of dry pyridine in a 2-mL conical vial under an atmosphere of dry nitrogen, ~0.5 mL of bis(trimethylsilyl)trifluoroacetamide containing 10% trimethylsilyl chloride was added, and the solution was stirred for 1 h at room temperature prior to GC analysis.

Reaction of *cis*-2-Acetyl-4-methyl-1,3-dioxane (1) with PhMgBr. A solution of 54.8 mg (0.380 mmol) of **1** in 2.3 mL of dry diethyl ether was cooled to –78 °C under an atmosphere of dry nitrogen and 0.25 mL of a 3.1 M solution of phenylmagnesium bromide (0.78 mmol) in diethyl ether was added by syringe. The reaction mixture was stirred at –78 °C for 1 h, 0.5 mL of MeOH was then added, the cooling bath was removed, and the reaction mixture was allowed to warm to room temperature. Saturated aqueous potassium carbonate (0.25 mL) was added and the mixture was stirred for 10 min. Solid potassium carbonate was then added until all solids were granular, the solids were removed by filtration, and the filtrate was concentrated to give 71.2 mg (84 %) of product as a 93:7 mixture of diastereoisomers. The major diastereoisomer, ***cis*-2-[(1R*)-1-hydroxy-1-phenylethyl]-(4S*)-4-methyl-1,3-dioxane**, displayed the following spectroscopic properties: ^{1}H NMR δ 1.25 (d, J = 6.2 Hz, 3H), 1.40-1.46 (m, 1H), 1.56 (s, 3H), 1.63-1.74 (m, 1H), 2.90 (broad s,

1H), 3.73-3.81 (m, 2H), 4.13 (dd, J = 11.4, J = 3.7 Hz, 1H), 4.61 (s, 1H), 7.25-7.28 (m, 1H), 7.34-7.37 (m, 2H), 7.54-7.56 (m, 2H); ^{13}C NMR δ 21.51, 24.81, 32.87, 66.75, 74.85, 76.49, 104.20, 125.64, 126.75, 127.75, 144.27; HRMS (EI) calcd for $C_{13}H_{17}O_3$ (M–1) 221.1178, found 221.1180.

Reaction of *cis*-2-Benzoyl-4-methyl-1,3-dioxane (2) with MeMgBr. A solution of 2.05 g (10.0 mmol) of **2** in 60 mL of dry diethyl ether was cooled to –78 °C under an atmosphere of dry nitrogen and 6.9 mL of a 2.9 M solution of methylmagnesium bromide (20 mmol) in diethyl ether was added by syringe. The reaction mixture was stirred at –78 °C for 1 h, 2 mL of methanol was then added, the cooling bath was removed, and the reaction mixture was allowed to warm to room temperature Saturated aqueous potassium carbonate (4 mL) was added and the mixture was stirred for 10 min. Solid potassium carbonate was then added until all solids were granular, the solids were removed by filtration, and the filtrate was concentrated to give 1.97 g (86%) of the title compound as an 85:15 mixture of diastereoisomers. The major diastereoisomer, ***cis*-2-[(1S*)-1-hydroxy-1-phenylethyl]-(4S*)-4-methyl-1,3-dioxane**, displayed the following spectroscopic properties: ^{1}H NMR δ 1.24 (d, *J* = 6.0 Hz, 3 H), 1.44 (m, 1 H), 1.57 (s, 3 H), 1.60–1.70 (m, 1 H), 2.9 (broad s, 1H), 3.71-3.78 (m, 2H), 4.12 (dd, *J* = 11.4, J = 5.0, 1 H), 4.58 (s, 1 H), 7.27–7.38 (m, 3 H), 7.55–7.59 (m, 2 H); ^{13}C NMR δ 21.89, 24.22, 33.20, 66.96, 73.35, 75.02, 104.71, 126.13, 127.05, 127.95, 144.59; HRMS (EI) calcd for $C_{13}H_{17}O_3$ (M–1) 221.1178, found 221.1179.

2R*-[(1R*)-1-Hydroxy-1-phenylethyl]-4,4-methyl-1,3-dioxane was prepared as the major diastereoisomer by reaction of 2-acetyl-4,4-dimethyl-1,3-dioxane (**3**) with PhMgBr

(Table 1, entry 2): ^{1}H NMR δ 1.19 (two s, 6 H), 1.43 (s, 3 H), 1.49 (m, 1 H), 1.80 (m, 1 H), 2.83 (s, 1 H), 3.72 - 3.76 (m, 2 H), 4.72 (s, 1 H), 7.17 - 7.26 (m, 3 H), 7.45 - 7.48 (m, 2 H); HRMS (EI) calcd for $C_{14}H_{19}O_3$ (M–1) 235.1334, found 235.1337.

2R*-[(1S*)-1-Hydroxy-1-phenylethyl]-4,4-methyl-1,3-dioxane was prepared as the major diastereoisomer by reaction of 2-benzoyl-4,4-dimethyl-1,3-dioxane (**4**) with MeMgBr (Table 1, entry 5): ^{1}H NMR δ 1.18 (s, 3 H), 1.20 (s, 3 H), 1.48 (s, 3 H), 1.49 (m, 1 H), 1.79 - 1.80 (m, 1 H), 2.85 (s, 1 H), 3.73 - 3.79 (m, 2 H), 4.70 (s, 1 H), 7.18 - 7.28 (m, 3 H), 7.46 - 7.48 (m, 2 H).

***cis*-2-[(1R*)-1-Hydroxy-1-phenylethyl]-(4S*)-4-*t*-butyl-1,3-dioxane** was prepared as the major diastereoisomer by reaction of *cis*-2-acetyl-4-*t*-butyl-1,3-dioxane (**5**) with PhMgBr (Table 1, entry 3): ^{1}H NMR δ 0.91 (s, 9 H), 1.33 - 1.36 (m, 1 H), 1.55 (s, 3H), 1.71 – 1.74 (m, 1 H), 2.80 (broad s, 1 H), 3.21 – 3.25 (m, 1 H), 3.69 – 3.72 (m, 1 H), 4.15 – 4.17 (m, 1 H), 4.53 (s, 1 H), 7.24 – 7.61 (m, 5 H).

***cis*-2-[(1S*)-1-Hydroxy-1-phenylethyl]-(4S*)-4-*t*-butyl-1,3-dioxane** was prepared as the major diastereoisomer by reaction of *cis*-2-benzoyl-4-*t*-butyl-1,3-dioxane (**6**) with MeMgBr (Table 1, entry 6): ^{1}H NMR δ 0.89 (s, 9H), 1.33 – 1.36 (m, 1H), 1.56 (s, 3H), 1.71 (m, 1H), 2.85 (br s, 1H), 3.25 – 3.28 (m, 1H), 3.64 - 3.71 (m, 1H), 4.14 – 4.19 (m, 1H), 4.55 (s, 1H), 7.24-7.35 (m, 3H), 7.54- 7.56 (m, 2H); ^{13}C NMR δ 24.16, 24.34, 25.56, 34.04, 66.95, 75.08, 84.56, 104.74, 125.99, 126.80, 127.63, 144.24; HRMS (EI) calcd for $C_{16}H_{23}O_3$ (M–1) 263.1647, found 263.1646.

(4R)-(+)-*cis*-2-Benzoyl-4-methyl-1,3-dioxane [(+)-2]. A round-bottomed flask equipped with a short path distillation head was charged with 1.35 g (6.54 mmol) of 2-phenylpropenal diethyl acetal, 0.567 g (6.29 mmol) of (R)-(–)-1,3-butanediol (Aldrich, 98% ee), 40 mL of cyclohexane and approximately 100 mg of pyridinium *p*-toluenesulfonate. The reaction mixture was heated until the temperature of the distillate reached 80 °C and heating was then discontinued. The reaction mixture was allowed to cool to room temperature, 1 g of sodium carbonate was added, the mixture was stirred for 1 h to neutralize the acid catalyst, and the mixture was then concentrated by rotary evaporation. The residue was suspended in 100 mL of diethyl ether and washed successively with two 50-mL portions of aqueous sodium carbonate and water, dried over Na_2SO_4, and concentrated to give an oil. The oil was dissolved in 100 mL of methylene chloride, the solution was cooled to –78 °C, and ozone was bubbled through the solution until a blue color persisted. The reaction mixture was flushed with nitrogen for 15 min to discharge excess ozone, 5 mL of dimethyl sulfide was added, and the mixture was allowed to warm and stir at room temperature for 12h. The resulting solution was washed successively with two 50-mL portions of aqueous sodium carbonate and water, dried over Na_2SO_4 and concentrated to give an oil. Column chromatography of this oil on silica gel (5% EtOAc – hexanes, R_f = 0.10) afforded 656 mg (45% for the two-step conversion) of the title compound: $[\alpha]^{24}_D$ = +17.21 [*c* 2.42, EtOH]; NMR spectra were identical with those recorded for the racemic compound.

R-(–)-Atrolactic Acid. Addition of MeMgBr to a solution of 452.2 mg (2.19 mmol) of (4R)-(+)-*cis*-2-benzoyl-4-methyl-1,3-dioxane [**(+)-2**] in diethyl ether at –78 °C, as

described above for racemic **2**, afforded 428.0 mg (86%) of an 85:15 mixture of diastereoisomeric alcohols as determined by GC analysis of the TMS derivatives. A solution of 690 mg (3.1 mmol) of this mixture of alcohols in 100 mL of methylene chloride was treated with ozone at room temperature for 1 h, and the reaction mixture was then purged with nitrogen. After cautious removal of solvent by rotary evaporation at room temperature, the residue was taken up in 20 mL of methanol and 20 mL of 1M aqueous sodium hydroxide solution was added. The solution was stirred at room temperature for 15 min, heated at 40 °C for 30 min, and then stirred for a further 30 min at room temperature. The methanolic solution was washed with three 100-mL portions of diethyl ether, acidified with concentrated hydrochloric acid to a pH of ~1, and extracted with four 50-mL portions of diethyl ether. The combined ethereal extracts were dried (Na_2SO_4) and concentrated to afford 310.8 mg (60% for the two-step conversion) of an off-white solid. Recrystallization of this material from benzene/cyclohexane gave 120.6 mg of pure (R)-(–)-atrolactic acid: mp 112-114 °C (lit.[12] mp 114-116 °C); $[\alpha]^{24}_D$ = –36.3 [c 3.43, EtOH] (lit.[12] $[\alpha]^{13.8}_D$ = –37.7 [c 3.4, EtOH], lit.[17] $[\alpha]^{25}_D$ = –36.7 [c 2.93, EtOH].

References and Notes

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