Preparation of (*S*, *S*)-2: To a stirred solution of (*S*, *S*)-(-)-hydrobenzoin (2.0 g, 9.33 mmol) in dry benzene (125 mL) was added di-*n*-butyltin oxide (2.79 g, 11.20 mmol). The mixture was refluxed azeotropically and the water formed was removed by a Dean-Stark apparatus. After 16 h of reflux, reaction mixture was cooled to rt. Tetra-*n*-butylammonium iodide (3.50 g, 9.47 mmol) and *t*-butyl bromoacetate (2.24 g, 1.7 mL, 11.45 mmol) were added and refluxing continued for another 12 h. The reaction mixture turned initially to pale yellow color which turns to a creamy white color. The reaction mixture was cooled and the solid was allowed to settle for 30 min. The supernatant liquid was filtered through a pad of silica gel. The solid was rinsed with anhydrous ether and filtered. The silica gel pad was washed thoroughly with ether. The ethereal layer was concentrated and chromatographed to give a mixture of desired lactone and the uncyclized ester. Complete lactonization was achieved by refluxing (13 h) the mixture with TFA (0.933 mmol) in benzene. The reaction mixture was cooled and washed with aq. $NaHCO_3$ (20 mL). The organic layer was separated, dried over anhydrous Na_2SO_4 and concentrated. Silica gel chromatographic purification (15% EtOAc / hexanes) of the concentrate gave **2** (1.51 g, 67 %) as a fine white solid. R_f 0.53 (30% EtOAc / hexanes). mp 98-100 ^{o}C. $[\alpha]_D$ -63.4 (c = 1.0, $CHCl_3$). ^{1}H (300 MHz, $CDCl_3$): δ 7.29-7.18 (m, 6 H), 7.04-6.97 (m, 4 H), 5.45 (d, 1 H, *J* = 9.3 Hz), 4.75 & 4.56 (AB q, 2 H, *J* = 17.8 Hz), 4.58 (d, 1 H, *J* = 9.3 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 167.06, 135.03, 134.62, 129.16, 129.06, 128.49,128.46, 127.49, 127.42, 86.47, 80.84, 66.28. MS (EI) *m/z* (relative Intensity): 254 (M^+, 43), 241 (33), 211 (95), 197 (35), 183 (100), 165 (20), 107 (35). HRMS (CI) calcd for $C_{16}H_{15}O_3$ (M^+ + H) 255.1021, found 255.1009. Anal. Calcd for $C_{16}H_{14}O_3$: C, 75.57; H, 5.55. Found: C, 75.43; H, 5.52.

General Aldol Reaction Procedure: To a stirred solution of **2** (100 mg, 0.39 mmol) in dry CH_2Cl_2 (18 mL), cooled to -78 ^{o}C, was added dry NEt_3 (94 mg, 0.13 mL, 0.93 mmol) dropwise over 5 min. The reaction mixture was stirred for 5 min after the addition. To this a solution of *c*-hexyl$_2$BOTf (1.0 M in hexane, 1.2 mL, 1.2 mmol) was added dropwise by syringe over 10 min and the reaction mixture was allowed to stir for 2-3 h at -78 ^{o}C. An aldehyde (0.43 mmol) was added neat or in dry CH_2Cl_2 (2 mL) over 5 min. The reaction mixture was stirred at -78 ^{o}C for 2 h. The reaction was quenched by the addition of pH 7 buffer (2 mL), followed by MeOH (0.4 mL) and 30% aq. H_2O_2 (0.2 mL). The mixture was stirred while allowing it to warm to rt. The reaction mixture was diluted with anhydrous ether (75 mL) and washed with 5 mL of aq. $NaHCO_3$. The organic layer was separated and the aqueous layer was back extracted with ether (4 X 30 mL). The combined organic extracts were successively treated with HCl (10%, 10 mL) and brine (20 mL). The ether layer was dried over anhydrous Na_2SO_4, concentrated and dried under vacuum. Silica gel radial chromatographic purification of the crude residue provided the desired aldol products as a mixture and whose ^{1}H NMR characterization gave the diastereomeric ratio. Further purification with a less polar eluent furnished the individual diastereomers:

3 (R=*i*-Pr): (*S, S, S, S*) major isomer. White crystalline solid. R_f 0.57 (30% EtOAc / hexanes). mp 120-121 °C. $[\alpha]_D$ -99.8 (c = 0.7, $CHCl_3$). ^1H (300 MHz, $CDCl_3$): δ 7.31-7.19 (m, 6 H), 7.06-7.01 (m, 4 H), 5.48 (d, 1 H, *J* = 9.0 Hz), 4.96 (d, 1 H, *J* = 9.0 Hz), 4.63 (d, 1 H, *J* = 6.6 Hz), 3.97-3.92 (m, 1 H), 3.10 (d, 1 H, D_2O exchangeable, *J* = 3.9 Hz), 2.22-2.07 (m, 1 H), 1.06 (d, 3 H, *J* = 6.6 Hz), 0.99 (d, 3 H, *J* = 6.9 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 171.02, 135.77, 134.51, 129.31, 129.03, 128.57, 127.58, 127.52, 85.55, 78.36, 77.02, 74.34, 29.71, 19.59, 16.31. MS (CI) *m/z* (relative Intensity): 327 (M^+ + H, 82), 309 (20), 281 (10), 255 (50), 197 (100), 180 (25), 131 (47). HRMS (CI) calcd for $C_{20}H_{23}O_4$ (M^+ + H) 327.1596, found 327.1600. Anal. Calcd for $C_{20}H_{22}O_4$: C, 73.60; H, 6.79. Found: C, 73.69; H, 6.81.

4 (R=*i*-Pr): (*S, S, R, R*) minor isomer. White solid. R_f 0.47 (30% EtOAc / hexanes). mp 153-154 °C. $[\alpha]_D$ -57.6 (c = 0.81, $CHCl_3$). ^1H (500 MHz, $CDCl_3$): δ 7.30-7.21 (m, 6 H), 7.03-6.98 (m, 4 H), 5.44 (d, 1 H, *J* = 9.2 Hz), 4.67 (d, 1 H, *J* = 9.3 Hz), 4.53 (d, 1 H, *J* = 5.8 Hz), 3.98-3.95 (m, 1 H), 3.17 (d, 1 H, D_2O exchangeable, *J* = 4.4 Hz), 2.27-2.18 (m, 1 H), 1.08 (d, 3 H, *J* = 6.8 Hz), 1.01 (d, 3 H, *J* = 6.8 Hz). ^{13}C (125 MHz, $CDCl_3$): δ 169.72, 135.22, 134.61, 129.34, 129.13, 128.59, 128.55, 127.58, 127.54, 86.97, 86.17, 78.06, 76.87, 29.62, 19.68, 16.79. MS (CI) *m/z* (relative Intensity): 327 (M^+ + H, 100), 309 (25), 281 (5), 255 (80), 197 (85), 131 (25). HRMS (CI) calcd for $C_{20}H_{23}O_4$ (M^+ + H) 327.1596, found 327.1581. Anal. Calcd for $C_{20}H_{22}O_4$: C, 73.60; H, 6.79. Found: C, 73.30; H, 6.61.

3 (R = Et): (*S, S, S, S*) major isomer. White solid. R_f 0.46 (30% EtOAc / hexanes). mp 108-110 °C. $[\alpha]_D$ = -98.6 (c = 0.73, $CHCl_3$). ^1H (300 MHz, $CDCl_3$): δ 7.31-7.19 (m, 6 H), 7.06-7.01 (m, 4 H), 5.46 (d, 1 H, *J* = 9.0 Hz), 4.98 (d, 1 H, *J* = 9.0 Hz), 4.56 (d, 1 H, *J* = 5.6 Hz), 4.13-4.05 (m, 1 H), 3.03 (d, 1 H, D_2O exchangeable, *J* = 4.6 Hz), 1.95-1.82 (m, 1 H), 1.76-1.62 (m, 1 H), 1.04 (t, 3 H, *J* = 7.3 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 170.23, 135.80, 134.47, 129.28, 128.99, 128.54, 127.58, 127.47, 85.49, 78.53, 75.93, 74.52, 26.53, 9.84. MS (CI) *m/z* (relative Intensity): 313 (M^+ + H, 95), 295 (100), 255(20), 197 (85), 100 (35), 167 (15). HRMS (CI) calcd for $C_{19}H_{21}O_4$ (M^+ + H) 313.1440, found 313.1437. Anal. Calcd for $C_{19}H_{20}O_4$: C, 73.06; H, 6.45. Found: C, 72.89; H, 6.62.

4 (R = Et): (*S, S, R, R*) minor isomer. White solid. R_f 0.43 (30% EtOAc / hexanes). ^1H (300 MHz, $CDCl_3$): δ 7.32-7.21 (m, 6 H), 7.12-6.97 (m, 4 H), 5.44 (d, 1 H, *J* = 9.3 Hz), 4.67 (d, 1 H, *J* = 9.5 Hz), 4.52 (d, 1 H, *J* = 5.3 Hz), 4.16-4.12 (m, 1 H), 2.94 (d, 1 H, D_2O exchangeable, *J* = 4.6 Hz), 1.95-1.69 (m, 2 H), 1.07 (t, 3 H, *J* = 7.3 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 169.02, 135.07, 134.45, 129.35, 129.19, 128.59, 128.57, 127.56, 127.48, 86.94, 80.96, 79.21, 73.92, 25.79, 10.05. HRMS (CI) calcd for $C_{19}H_{21}O_4$ (M^+ + H) 313.1440, found 313.1426.

3 (R = *n*-C_5H_{11}): (*S, S, S, S*) major isomer. White solid. R_f 0.46 (25% EtOAc / hexanes). mp 115-116 °C. $[\alpha]_D$ -92.3 (c = 0.97, $CHCl_3$). ^1H (500 MHz, $CDCl_3$): δ 7.31-7.19 (m, 6 H), 7.07-7.01 (m, 4 H), 5.46 (d, 1 H, *J* = 9.0 Hz), 4.99 (d, 1 H, *J* = 9.0 Hz), 4.55 (d, 1 H, *J* = 5.3 Hz), 4.19-4.10 (m, 1 H), 2.92 (d, 1 H, D_2O exchangeable, *J* = 4.9 Hz), 1.86-1.23 (m, 8 H), 0.99 (distorted t, 3 H, *J* = 7.0 Hz). ^{13}C (125 MHz, $CDCl_3$): δ 170.14, 135.86, 134.52, 129.31, 129.02, 128.57, 127.61, 127.50, 85.47, 78.58, 76.32, 73.44, 33.54, 31.82, 25.16, 22.79, 14.22. MS (CI) *m/z* (relative Intensity): 355 (M^+ + H, 40), 337 (30), 255 (50), 197 (100), 180 (20), 159 (22), 107 (10). HRMS (CI) calcd for $C_{22}H_{27}O_4$ (M^+ + H) 355.1909, found. 355.1906. Anal. Calcd for $C_{22}H_{26}O_4$: C, 74.55; H, 7.39. Found: C, 74.42; H, 7.65.

4 (R = *n*-C_5H_{11}): (*S, S, R, R*) minor isomer. White solid. R_f 0.36 (25% EtOAc / hexanes). mp 135 °C. ^1H (500 MHz, $CDCl_3$): δ 7.32-7.21 (m, 6 H), 7.04-6.98 (m, 4 H), 5.45(d, 1 H, *J* = 9.3 Hz), 4.67 (d, 1 H, *J* = 9.5 Hz), 4.53 (d, 1 H, *J* = 5.1 Hz), 4.25-4.17 (m, 1 H), 2.86 (d, 1 H, D_2O exchangeable, *J* = 5.8 Hz), 1.82-1.23 (m, 8 H), 0.91 (distorted t, 3 H, *J* = 7.0 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 168.82, 135.09, 134.47, 129.33, 129.18, 128.56, 128.58, 127.57, 127.49, 86.92, 80.96, 79.61, 72.78, 32.76, 31.93, 25.40, 22.84, 14.26. MS (CI) *m/z* (relative Intensity): 355 (M^+ + H, 55), 337 (40), 255 (55), 197 (100), 107 (22). HRMS (CI) calcd for $C_{22}H_{27}O_4$ (M^+ + H) 355.1909, found 355.1899.

3 (R = *i*-$PrCH_2$): (*S, S, S, S*) major isomer. White solid R_f 0.53 (30% EtOAc / hexanes). mp 120-122 °C. $[\alpha]_D$ -99.5 (c = 0.62, $CHCl_3$). ^1H (300 MHz, $CDCl_3$): δ 7.32-7.20 (m, 6 H), 7.06-7.02 (m, 4 H), 5.46 (d, 1 H, *J* = 9.0 Hz), 5.01 (d, 1 H, *J* = 9.0 Hz), 4.54 (d, 1 H, *J* = 5.1 Hz), 4.27-4.19 (m, 1 H), 2.79 (d, 1 H, D_2O exchangeable, *J* = 5.6 Hz), 1.94-1.83 (m, 1 H), 1.73-1.53 (m, 2 H), 0.95 (apparent t, 6 H, *J* = 7.0 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 169.96, 135.92, 134.52, 129.33, 129.05, 128.61, 128.59, 127.64, 127.52, 85.46, 78.63, 76.88, 71.95, 42.61, 24.48, 23.86, 21.67. MS (CI) *m/z* (relative Intensity): 341 (M^+ + H, 87), 323 (100), 255 (38), 197 (68), 180 (30), 107 (9). HRMS (CI) calcd for $C_{21}H_{25}O_4$ (M^++ H) 341.1753, found. 341.1741. Anal. Calcd for $C_{21}H_{24}O_4$: C, 74.09; H, 7.11. Found: C, 73.98; H, 7.19.

4 (R = *i*-$PrCH_2$): (*S, S, R, R*) minor isomer (9 / 1 mixture of *anti / syn*). White solid. R_f 0.47 (30% EtOAc / hexanes). ^1H (300 MHz, $CDCl_3$): δ 7.31-7.21 (m, 6 H), 7.04-7.97 (m, 4 H), 5.46 (d, 1 H, *J* = 9.5 Hz), 4.66 [d, 1 H, *J* = 9.2 Hz (4.69 (d, *syn*, *J* = 9.0 Hz))], 4.53 [d, 1 H, *J* = 4.8 Hz (4.56 (d, *syn*, 2.4 Hz))], 4.33-4.25 (m, 1 H), 2.73 [d, 1 H, D_2O exchangeable, *J* = 6.3 Hz (2.35 (d, *syn*, 9.5 Hz))], 1.99-1.87 (m, 1 H), 1.80-1.67 (m, 1 H), 1.57-1.48 (m, 1 H), 1.00 (d, 3 H, *J* = 6.3 Hz), 0.98 (d, 3 H, *J* = 6.3 Hz). ^{13}C (75 MHz, CDCl3): δ 168.57, 135.09, 134.49, 129.33, 129.20, 128.58, 127.58, 127.48, 86.87 (86.53, *syn*), 80.98 (80.72, *syn*), 80.10 (79.58, *syn*), 71.10 (70.91, *syn*), 41.73 (42.19, *syn*), 24.63 (24.83, *syn*), 23.95 (23.65, *syn*), 21.72 (22.14, *syn*). MS (CI) m/z (relative Intensity): 341 (M^+ + H, 90), 323 (25), 255 (90), 197 (100),145 (20), 107 (15). HRMS (CI) calcd for $C_{21}H_{25}O_4$ (M^+ + H) 341.1753, found. 341.1745.

3 (R = Ph): (S, S, S, S) major anti isomer. White crystalline solid. R_f 0.36 (30% EtOAc / hexanes). mp 151-152 OC. $[\alpha]_D$ = -159.2 (c = 1.0, $CHCl_3$). ^1H (300 MHz, CDCl3): δ 7.50-7.36 (m, 5 H), 7.26-7.12 (m, 6 H), 6.81-6.77 (m, 4 H), 5.39 (apparent dd, 1 H, *J* = 4.6 and 5.6 Hz), 5.37 (d, 1 H, *J* = 9.0 Hz), 4.96 (d, 1 H, *J* = 4.6 Hz), 4.27 (d, 1 H, *J* = 9.0 Hz), 3.25 (d, 1 H, D_2O exchangeable, *J* = 5.6 Hz). ^{13}C (75 MHz, CDCl3): δ 168.85, 139.57, 135.19, 134.24, 129.25, 128.98, 128.79, 128.70, 128.50. 127.52, 127.44. 126.96, 85.67, 78.53, 77.91, 74.64. MS (CI) m/z (relative Intensity): 361 (M^+ + H, 5), 343 (27), 211 (80), 197 (40), 183 (100), 167 (15), 107 (72). HRMS (CI) calcd for $C_{23}H_{21}O_4$ (M^+ + H) 361.1440, found 361.1429. Anal. Calcd for $C_{23}H_{20}O_4$: C, 76.65; H, 5.59. Found: C, 76.72; H, 5.53.

3 (R=Ph): syn isomer. White solid. mp 210 ^OC. R_f 0.41 (30% EtOAc / hexanes). ^1H (500 MHz, $CDCl_3$): δ 7.44-7.15 (m, 11 H), 6.95-6.92 (m, 4 H), 5.44 (d, 1 H, *J* = 9.0 Hz), 5.39 (dd, 1 H, *J* = 3.4 and 6.3 Hz), 4.98 (d, 1 H, *J* = 9.0 Hz), 4 90 (d, 1 H, *J* = 3.4 Hz), 3.21 (d, 1 H, *J* = 5.3 Hz). ^{13}C (125 MHz, $CDCl_3$): δ 169.36, 140.02, 135.48, 134.69, 129.19, 128.93, 128.77, 128.66, 128.50. 127.69, 127.55, 127.16, 86.48, 78.56, 77.36, 76.39. MS (CI) m/z (relative Intensity): 361 (M^+ + H, 5), 343 (25), 318 (30), 255 (90), 197 (100), 180 (35), 165 (32), 148 (17), 107 (97). HRMS (CI) calcd for $C_{23}H_{21}O_4$ (M^+ + H) 361.1440, found. 361.1431.

3 (R = PhCH=CH): (*S, S, S, S*) major isomer. Foam. R_f 0.40 (30% EtOAc / hexanes). $[\alpha]_D$ -58.7 (c = 1.56, $CHCl_3$). ^1H (300 MHz, $CDCl_3$): δ 7.42-7.16 (m, 11 H), 7.03-7.02 (m, 4 H), 6.76 (d, 1 H, *J* = 16.1 Hz), 6.53-6.46 (dd, 1 H, *J* = 6.3 and 16.1 Hz), 5.47 (d, 1 H, *J* = 8.7 Hz), 5.10 (d, 1 H, *J* = 8.7 Hz), 4.93-4.88 (m, 1 H), 4. 77 (d, 1 H, *J* = 4.4 Hz), 2.99 (d, 1 H, D_2O exchangeable, *J* = 5.6 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 168.64, 136.45, 135.57, 134.56, 132.72, 129.25, 129.06, 128.88, 128.59, 128.55, 128.23, 127.60, 127.56, 127.22, 126.90, 85.62, 78.78, 77.07, 74.62. MS (FAB^+) *m/z* (relative Intensity): 387 (M^+ + H, 15), 369 (90), 341 (45), 323 (15), 255 (75), 133 (100). HRMS calcd for $C_{25}H_{23}O_4$ (M^+ + H) 387.1596, found 387.1583. Anal. Calcd for $C_{25}H_{22}O_4$: C, 77.70; H, 5.74. Found: C, 77.65; H, 5.72.

4 (R = PhCH=CH): (*S, S, R, R*) minor. Foam. R_f 0.36 (30% EtOAc / hexanes). ^1H (300 MHz, $CDCl_3$): δ 7.46-7.19 (m, 11 H), 7.04-6.96 (m, 4 H), 6.83 (d, 1 H, *J* = 16.1 Hz), 6.54-6.46 (dd, 1 H, *J* = 6.3 and 15.8 Hz), 5.43 (d, 1 H, *J* = 9.3 Hz), 4.97-4.91 (m, 1 H), 4.72 (d, 1 H, *J* = 4.6 Hz), 4.71 (d, 1 H, *J* = 9.0 Hz), 3.00 (d, 1 H, D_2O exchangeable, *J* = 5.8 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 168.87, 136.58, 134.99, 134.38, 133.53, 129.35, 129.22, 128.84, 128.59, 128.58, 128.26, 127.61, 127.51, 127.00, 126.37, 86.77, 80.93, 79.76, 73.66. MS (CI) *m/z* (relative Intensity): 387 (M^+ + H, 2), 255 (22), 133 (100), 107 (14). HRMS calcd for $C_{25}H_{23}O_4$ (M^++ H) 387.1596, found 387.1580.

3 (R = PhCCH): (*S, S, S, S*) major isomer. Foam. R_f 0.34 (30% EtOAc / hexanes). $[\alpha]_D$ -12.6 (c = 1.0, $CHCl_3$). ^1H (300 MHz, $CDCl_3$): δ 7.50-7.47 (m, 2 H), 7.39-7.28 (m, 3 H), 7.28-7.19 (m, 4 H), 7.15-7.12 (m, 2 H), 7.03-7.02 (m, 2 H), 6.98-6.96 (m, 2 H), 5.56 (d, 1 H, J = 9.0 Hz), 5.49 (d, 1 H, J = 9.0 Hz), 5.22-5.17 (m, 1 H), 4. 98 (d, 1 H, J = 3.9 Hz), 2.99 (d, 1 H, D_2O exchangeable, J = 9.7 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 166.77, 135.19, 134.34, 131.93, 129.29, 129.27, 129.21, 128.68, 128.60, 128.51, 127.74, 127.56, 121.96, 87.55, 86.86, 86.26, 78.88, 76.91, 64.30. MS (FAB^+) *m/z* (relative Intensity): 385 (M^+ + H, 100), 367 (20), 255 (40), 197 (55), 131 (55), 107 (20). HRMS (FAB^+) calcd for $C_{25}H_{21}O_4$ (M^+ + H), 385.1440, found. 385.1441.

3 Aldol reaction with (*R, R*)-2 and matched 2,3-*O*-(Isopropylidene)-D-glyceraldehyde: (*R, R, R, R*) major isomer. White crystalline solid. R_f = 0.27 (30% EtOAc / hexanes). mp 175-177 °C. $[\alpha]_D$ +70.5 (c = 0.44, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): 7.28-7.20 (m, 6 H), 7.06-7.02 (m, 4 H), 5.45 (d, 1 H, J = 9.0 Hz), 5.18 (d, 1 H, J = 9.0 Hz), 4.92 (d, J = 2.1 Hz, 1 H), 4.33-4.13 (m, 3 H), 4.06 (dd, 1 H, J = 5.0 and 8.9 Hz), 2.62 (d, 1 H, J = 8.4 Hz), 1.48 (s, 3 H), 1.41 (s, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): 167.7, 135.7, 134.8, 129.2, 129.1, 128.6, 128.5, 127.7, 127.6, 110.3, 86.0, 78.2, 75.9, 75.6, 75.4, 67.4, 26.6, 25.6; HRMS (CI) calcd for (M^+ + H) $C_{22}H_{25}O_6$ 385.1573, found 385.1653.

Preparation of 5: (This compound was prepared in four steps in the following order as described below.) **Preparation of (2-Methylbenzyl) phosphonic acid diisopropyl ester**: 2-Methylbenzyl bromide (6.38 g, 4.62 mL, 34.47 mmol) was heated with excess triisopropyl phosphite (21.1 g, 25 mL, 101.14 mmol) at 130 °C for 3 h. Excess phosphite was removed in vacuo and the remaining crude residue was distilled (94-96 °C / 4 mmHg) under vacuum to give the phosophonate (8.83 g, 95%) as a clear oil. ^{31}P (121 MHz, $CDCl_3$): δ 25.71. ^1H (300 MHz, $CDCl_3$): δ 7.30-7.25 (m, 1H), 7.16-7.10 (m, 3 H), 4.66-4.52 (m, 2 H), 3.11 (d, 2 H, $^2J_{PH}$ = 21.9 Hz), 2.38 (d, 3 H, $^5J_{PH}$ = 1.6 Hz), 1.27 (d, 3 H, J = 6.1 Hz), 1.14 (d, 3 H, J = 6.3 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 137.05 (d, J_{PC} = 7.0 Hz), 130.74 (d, J_{PC} = 5.5 Hz), 130.52 (d, J_{PC} = 9.0 Hz), 130.35 (d, J_{PC} = 3.0 Hz), 126.90 (d, J_{PC} = 4.0 Hz), 125.87 (d, J_{PC} = 3.5 Hz), 70.49 (d, $^2J_{POC}$ = 7.0 Hz), 32.06 (d, $^1J_{PC}$ = 140.0 Hz), 24.15 (d, $^3J_{PC}$ = 4.0 Hz), 23.77 (d, $^3J_{PC}$ = 5.0Hz), 20.09 (d, $^4J_{PC}$ = 1.5 Hz). Anal Calcd for $C_{14}H_{24}0_3P$: C, 62.21; H, 8.58. Found: C, 62.14; H, 8.72.

Preparation of 2,2'-dimethyl-*E*-stilbene: The above phosphonate was added (6.55 g, 24.23 mmol) to dry DMF (60 mL) containing NaOMe (1.32 g, 24.44 mmol) and stirred for 15 min. The white coagulated material was cooled to 0 °C and *o*-tolualdehyde (2.28g, 2.20 mL, 19.02 mmol) was added. The reaction mixture was stirred at rt for 2 h and heated at 100 °C for 2 h followed by stirring at rt overnight. To this water/methanol (2:1, 30 mL) was added and stirred for 30 min to form the fine white powder. The precipitated solid was collected by filtration. The solid was dried and recrystallized from ethanol to

provide the required stilbene (3.46 g, 87%). mp. 81-82 ^{O}C (lit^{R}. mp 83-84 ^{O}C). ^{1}H (300 MHz, $CDCl_3$): δ 7.59 (d, 2 H, J = 6.3 Hz), 7.25-7.17 (m, 8 H), 2.42 (s, 6 H). ^{13}C (75 MHz, $CDCl_3$): δ 137.03, 136.60, 130.60, 128.23, 127.75, 126.41, 125.77, 20.17. MS (EI) *m/z* (relative intensity): 208 (M^+, 100), 193 (33), 178 (25), 165 (5), 115 (15). HRMS (EI) calcd for $C_{16}H_{16}$ 208.1252, found. 208.1247. Anal. Calcd for $C_{16}H_{16}$: C, 92.66; H, 7.74. Found: C, 92.75; H, 7.75.

(*S*, *S*)-(-)-*o*-1,2-bis(2-methylphenyl)ethane diol: To AD-mix-α (4.2 g) were added 30 mL of *t*-BuOH:H_2O with vigorous stirring at rt for 15 min. Methanesulfonamide (0.285 g, 3.0 mmol) was added and the reaction mixture was cooled to 0 ^{O}C. The reaction mixture turned to orange color where upon 2,2'-dimethyl-*E*-stilbene (0.624 g, 3.0 mmol) was added at 0 ^{O}C. The reaction was stopped after 11 h of vigorous stirring. While stirring at 0 ^{O}C, solid sodium sulfite (4.5 g) was added and the mixture was allowed to warm to rt. Stirring was continued for 1 h and CH_2Cl_2 (30 mL) was added and the organic layer was separated. The aqueous layer was further extracted with CH_2Cl_2 (3 X 30 mL) and the combined organic layers were washed with 2 N KOH (50 mL), dried over anhydrous Na_2SO_4, concentrated and purified by silica gel column chromatography to provide the diol as a crystalline solid (0.57 g, 78%). R_f = 0.31 (30% EtOAc / hexanes) mp. 108-109 ^{O}C. $[\alpha]_D$ -64.8 (c = 0.83, EtOH) {lit. mp 109-110 ^{O}C. $[\alpha]_D$ -64.8 (c = 1.07, EtOH)}. ^{1}H (300 MHz, $CDCl_3$): δ 7.60 (dd, 2 H, J = 1.4 and 7.3 Hz), 7.19 (dt, 2 H, J = 1.4 Hz and 7.3 Hz), 7.11 (dt, 2 H, J = 1.4 and 7.3 Hz), 6.91 (dd, 2 H, J = 1.4 and 7.3 Hz), 4.94 (s, 2 H), 3. 03 (bs, 2 H, D_2O exchangeable), 1.64 (s, 6 H).). ^{13}C (75 MHz, $CDCl_3$): δ 138.15, 136.09, 130.36, 127.90, 127.38, 126.13, 74.79, 18.93. MS (CI^+) *m/z* (relative Intensity): 241 (5), 121 (100). HRMS (EI) calcd for $C_{16}H_{18}O_2$ 242.1307, found 242.1320. Anal. Calcd for $C_{16}H_{18}O_2$: C, 79.31; H, 7.49. Found: C, 79.12; H, 7.31.

Preparation of 5 : Lactone (**5**) was prepared according to the aforementioned procedure for **2** by using (S,S)-*o*-1,2-bis(2-methylphenyl)ethane diol (2.18 g, 9.02 mmol), di-*n*-butyltin oxide (2.48 g, 10.22 mmol), tetrabutylammonium iodide (3.66 g, 9.91 mmol) and *t*-butyl bromoacetate (2.38 g, 1.8 mL, 12.20 mmol). Yield (1.92 g, 75%). White crystalline solid. R_f = 0.66 (30% EtOAc / hexanes). mp 153-154 ^{O}C. $[\alpha]_D$ -25.0 (c = 1.0, $CHCl_3$). ^{1}H (300 MHz, $CDCl_3$): δ 7.49 (d, 1 H, J = 7.3 Hz), 7.35 (d, 1 H, J = 6.6 Hz), 7.15-7.04 (m, 4 H), 6.83 (d, 2 H, J = 7.3 Hz), 5.72 (d, 1 H, J = 9.3 Hz), 4.80 (d, 1 H, J = 9.3 Hz), 4.66 & 4.47 (AB q, 2 H, J = 17.5 Hz), 1.49 (s, 3 H), 1.47 (s, 3 H). ^{13}C (75 MHz, $CDCl_3$): δ 167.19, 136.53, 136.17, 133.46, 132.91, 130.57, 130.50, 128.90, 128.80, 127.84, 127.40, 126.26, 126.21, 82.82, 76.75, 66.26, 18.72, 18.58. MS (EI) *m/z* (relative Intensity): 354 (M^+, 5), 208 (8), 195 (15), 178 (15), 162 (90), 134 (20), 119 (100), 91 (60). HRMS (CI) calcd for $C_{18}H_{19}O_3$ (M^+ + H) 283.1334, found. 283.1328. Anal. Calcd for $C_{18}H_{18}O_3$: C, 76.57; H, 6.43. Found: C, 76.68; H, 6.50.

6 (R = *i*-Pr): (*S, S, S, S*) major isomer. White crystalline solid. R_f = 0.64 (30% EtOAc / hexanes). mp 150-151 °C. $[\alpha]_D$ -84 6 (c = 0.7, $CHCl_3$). ^1H (300 MHz, $CDCl_3$): δ 7.57 (dd, 1 H, *J* = 7.5 and 1.3 Hz), 7.51 (dd, 1 H, *J* = 7.5 and 1.3 Hz), 7.25-7.13 (m, 4 H), 6.96-6.92 (apparent d, 2 H, *J* = 7.1 Hz), 5.85 (d, 1 H, *J* = 9.0 Hz), 5.34 (d, 1 H, *J* = 9.0 Hz), 4.62 (d, 1 H, *J* = 6.1 Hz), 3.97-3.92 (m, 1 H), 3.09 (d, 1 H, D_2O exchangeable, *J* = 4.1 Hz), 2.22-2.07 (m, 1 H), 1.64 (s, 3 H), 1.63 (s, 3 H), 1.05 (d, 3 H, *J* = 6.8 Hz), 0.98 (d, 3 H, *J* = 6.8 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 170.85, 136.72, 136.04, 134.15, 132.95, 130.68, 130.55, 128.99, 128.85, 128.11, 127.52, 126.33, 126.31, 81.80, 77.71, 74.34, 73.66, 29.94, 19.57, 18.89, 18.73, 16.36. MS (CI) *m/z* (relative Intensity): 355 (M^+ + H, 8), 337 (30), 283 (70), 225 (100), 207 (75), 195 (15), 121 (33), 105 (20). HRMS (CI) calcd for $C_{22}H_{27}O_4$ (M^+ + H) 355.1909, found 355.1903. Anal. Calcd for $C_{22}H_{26}O_4$ C, 74.36; H, 7.19. Found: C, 74.55; H, 7.39.

6 (R = Et): (*S, S, S, S*) major isomer. White crystalline solid R_f = 0.51 (30% EtOAc / hexanes). mp 143-144 °C. $[\alpha]_D$ -89.6 (c = 0.7, $CHCl_3$). ^1H (300 MHz, $CDCl_3$): δ 7.57 (dd, 1 H, *J* = 7.5 and 1.4 Hz), 7.52 (dd, 1 H, *J* = 7.5 and 1.3 Hz), 7.25-7.14 (m, 4 H), 6.99-6.92 (apparent d, 2 H, *J* = 7.3 Hz), 5.85 (d, 1 H, *J* = 9.0 Hz), 5.36 (d, 1 H, *J* = 9.0 Hz), 4.55 (d, 1 H, *J* = 5.3 Hz), 4.14-4.06 (m, 1 H), 2.98 (distorted d, 1 H, D_2O exchangeable, *J* = 3.9 Hz), 1.94-1.83 (m, 1 H), 1.78-1.68 (m, 1 H), 1.64 (s, 3H), 1.63 (s, 3 H), 1.04 (t, 3 H, *J* = 7.3 Hz). ^{13}C (75 MHz, $CDCl_3$): δ 170.19, 136.73, 136.05, 134.20, 132.91, 130.70, 130.59, 129.01, 128.86, 128.13, 127.50, 126.33, 81.72, 75.95, 75.04, 73.96, 26.80, 18.92, 18.75, 9.91. MS (CI) *m/z* (relative Intensity): 341 (M^+ + H, 100), 323 (5), 283 (10), 225 (45), 207 (5), 121 (10). HRMS (CI) calcd for $C_{21}H_{25}O_4$ (M^+ + H) 341.1753, found 355.1753. Anal. Calcd for $C_{21}H_{24}O_4$: C, 74.09; H, 7.11. Found: C, 73.86; H, 6.99.

8: (*5S*)-5-Phenyl-4-oxa-pyran-2-one (prepared according to footnote 19) Viscous oil. R_f = 0.40 (30% EtOAc / hexanes). $[\alpha]_D$ +117.1 (c = 0.22, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ 7.40-7.36 (m, 5 H), 4.84 (dd, 1 H, *J* = 3.3 and 9.9 Hz), 4.65 & 4.48 (AB q, 2 H, *J* = 18.0 Hz), 4.50 (dd, 1 H, *J* = 3.3 Hz and 11.4 Hz), 4.43 (dd, 1 H, *J* = 9.9 Hz and 11.4 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.5, 135.2, 129.2, 126.4, 74.2, 73.2, 66.2. HRMS (CI) calcd for (M^+ + H) $C_{10}H_{11}O_3$ 179.0630, found 179.0730.

9 (R=*i* -Pr): Major product of aldol reaction with 8. Colorless viscous oil. R_f = 0.35 (30% EtOAc / hexanes). ^1H NMR (300MHz, $CDCl_3$): δ 7.44-7.35 (m, 5 H), 5.09 (dd, 1 H, *J* = 3.9 Hz and 8.4 Hz), 4.60-4.47 (m, 2 H), 4.42 (d, 1 H, *J* = 7.5 Hz), 3.92-3.87 (m, 1 H), 3.12 (d, 1 H, *J* = 4.2 Hz), 2.19-2.08 (m, 1 H), 1.04 (d, 3 H, *J* = 6.7 Hz), 0.98 (d, *J* = 6.7 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.0, 135.6, 129.2, 129.1, 126.4, 76.0, 73.8, 72.2, 72.0, 29.2, 19.6, 15.7.

General procedure for hydrogenation: **3** (R = *i*-Pr, 100 mg) was dissolved in MeOH (50 mL) and to it 10% Pd/C (100 mg) was added under a nitrogen atmosphere. This solution was hydrogenated (100-200

psi) in a metal reactor for 8-16 h. After completion, the reaction mixture was filtered and the filtrate was dried and concentrated under vacuum. The diphenylethane by-product was removed by rinsing the crude residue with hexanes (10 mL). The desired compound was dried under vacuum to give **11** (R = *i*-Pr): (*S*, *S*), (33.4 mg 73%) viscous liquid. $[\alpha]_D$ +11.1 (c = 1.0, MeOH). ^{1}H (500 MHz, CD_3OD): δ 4.09 (d, 1 H, *J* = 4.4 Hz), 3.48 (br s, 1 H), 4.96 (d, 1 H, *J* = 9.0 Hz), 1.97 (br s, 1 H), 0.97 (d, 3 H, *J* = 6.8 Hz), 0.95 (d, 3 H, *J* = 6.8 Hz). ^{13}C (125 MHz, CD_3OD): δ 177.12, 79.24, 74.02, 30.84, 20.13, 17.76. MS (CI) *m/z* (relative Intensity): 149 (M^+ + H, 65), 131 (60), 113 (70).

11 (R = Et): (*S*, *S*). 86%, viscous liquid. $[\alpha]_D$ +4.0 (c = 1.0, MeOH) ^{1}H (500 MHz, CD_3OD): δ 4.06 (d, 1 H, *J* = 4.4 Hz), 3.72-3.68 (m, 1 H),1.57-1.50 (m, 1 H), 0.97 (t, 3 H, *J* = 7.3 Hz). ^{13}C (125 MHz, CD_3OD): δ 176.16, 75.67, 75.46, 25.79, 10.69. MS (CI) *m/z* (relative Intensity): 135 (M^+ + H, 100), 117 (25), 107 (55).

12 (*S*, *S*). 74%, viscous oil. $[\alpha]_D$ +12.2 (c = 0.5, H_2O). ^{1}H (500 MHz, CD_3OD): δ 4.26 (d, 1 H, *J* = 4.4 Hz), 3.44 (s, 3 H), 3.16 (dd, 1 H, *J* = 4.4 and 6.1 Hz), 2.05-1.95 (m, 1 H), 0.97 (d, 6 H, *J* = 6.8 Hz). ^{13}C (125 MHz, CD_3OD): δ 176.57, 89.90, 72.56, 60.33, 30.86, 20.12, 18.54.

Table 1. Crystal data and structure refinement for 3.

Identification code	ma9
Empirical formula	$C_{20}H_{22}O_4$
Formula weight	326.38
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell dimensions	a = 12.167(3) Å alpha = 90° b = 5.8264(13) Å beta = 112.752(14)° c = 13.674(4) Å gamma = 90°
Volume, Z	893.9(4) $Å^3$, 2
Density (calculated)	1.213 Mg/m^3
Absorption coefficient	0.084 mm^{-1}
F(000)	348
Crystal size	0.5 x 0.4 x 0.15 mm
Θ range for data collection	2.86 to 25.01°
Limiting indices	$-14 \le h \le 0$, $0 \le k \le 6$, $-14 \le l \le 16$
Reflections collected	1809
Independent reflections	1726 (R_{int} = 0.0388)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1726 / 1 / 218
Goodness-of-fit on F^2	1.037
Final R indices [I>2σ(I)]	R1 = 0.0500, wR2 = 0.1075
R indices (all data)	R1 = 0.0914, wR2 = 0.1278
Absolute structure parameter	0(3)
Extinction coefficient	0.018(5)
Largest diff. peak and hole	0.122 and -0.131 $eÅ^{-3}$

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 3. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(1)	4494(2)	972(6)	70(2)	69(1)
C(1)	4878(4)	1269(8)	1022(3)	55(1)
O(2)	6054(2)	1186(6)	1555(2)	64(1)
C(3)	6600(3)	1473(9)	2702(3)	59(1)
C(4)	5789(3)	2801(9)	3106(3)	56(1)
O(5)	4643(2)	1711(6)	2711(2)	63(1)
C(6)	4058(4)	1811(8)	1579(3)	55(1)
C(7)	7784(4)	2585(10)	2956(3)	62(1)
C(8)	7865(4)	4642(11)	2484(3)	74(2)
C(9)	8962(5)	5676(15)	2744(4)	105(2)
C(10)	9969(6)	4705(19)	3478(6)	123(3)
C(11)	9885(6)	2721(18)	3937(6)	120(3)
C(12)	8794(4)	1656(12)	3681(4)	88(2)
C(13)	6252(4)	2758(8)	4306(3)	53(1)
C(14)	6028(4)	931(10)	4840(3)	65(1)
C(15)	6461(4)	918(11)	5937(4)	76(2)
C(16)	7111(4)	2721(12)	6497(4)	79(2)
C(17)	7349(5)	4579(10)	5989(4)	79(1)
C(18)	6912(4)	4576(9)	4887(3)	69(1)
C(19)	3409(4)	4070(9)	1210(3)	62(1)
O(19)	4216(3)	5872(6)	1257(2)	66(1)
C(20)	2342(4)	3972(11)	132(4)	77(2)
C(21)	1866(5)	6351(14)	-236(4)	102(2)
C(22)	1356(5)	2467(14)	237(6)	139(3)

Table 3. Bond lengths [Å] and angles [°] for 3.

O(1)-C(1)	1.213(4)	C(1)-O(2)	1.332(4)
C(1)-C(6)	1.505(6)	O(2)-C(3)	1.457(4)
C(3)-C(7)	1.492(6)	C(3)-C(4)	1.517(5)
C(4)-O(5)	1.434(5)	C(4)-C(13)	1.515(5)
O(5)-C(6)	1.433(4)	C(6)-C(19)	1.518(7)
C(7)-C(12)	1.358(6)	C(7)-C(8)	1.383(7)
C(8)-C(9)	1.380(7)	C(9)-C(10)	1.370(10)
C(10)-C(11)	1.338(11)	C(11)-C(12)	1.381(9)
C(13)-C(14)	1.376(6)	C(13)-C(18)	1.380(6)
C(14)-C(15)	1.384(6)	C(15)-C(16)	1.360(8)
C(16)-C(17)	1.376(8)	C(17)-C(18)	1.389(6)
C(19)-O(19)	1.422(5)	C(19)-C(20)	1.543(6)
C(20)-C(21)	1.511(9)	C(20)-C(22)	1.537(8)

O(1)-C(1)-O(2)	118.0(3)	O(1)-C(1)-C(6)	121.2(4)
O(2)-C(1)-C(6)	120.8(3)	C(1)-O(2)-C(3)	122.1(3)
O(2)-C(3)-C(7)	107.4(3)	O(2)-C(3)-C(4)	111.1(3)
C(7)-C(3)-C(4)	113.5(4)	O(5)-C(4)-C(13)	107.5(3)
O(5)-C(4)-C(3)	108.2(4)	C(13)-C(4)-C(3)	111.4(3)
C(6)-O(5)-C(4)	113.1(3)	O(5)-C(6)-C(1)	112.9(3)
O(5)-C(6)-C(19)	111.8(3)	C(1)-C(6)-C(19)	112.0(4)
C(12)-C(7)-C(8)	118.5(5)	C(12)-C(7)-C(3)	121.1(5)
C(8)-C(7)-C(3)	120.3(4)	C(9)-C(8)-C(7)	119.7(5)
C(10)-C(9)-C(8)	120.7(7)	C(11)-C(10)-C(9)	119.4(7)
C(10)-C(11)-C(12)	120.7(7)	C(7)-C(12)-C(11)	121.0(7)
C(14)-C(13)-C(18)	118.6(4)	C(14)-C(13)-C(4)	121.4(4)
C(18)-C(13)-C(4)	120.0(4)	C(13)-C(14)-C(15)	120.6(5)
C(16)-C(15)-C(14)	120.1(5)	C(15)-C(16)-C(17)	120.8(4)
C(16)-C(17)-C(18)	118.7(5)	C(13)-C(18)-C(17)	121.2(5)
O(19)-C(19)-C(6)	111.3(3)	O(19)-C(19)-C(20)	112.4(4)
C(6)-C(19)-C(20)	115.0(4)	C(21)-C(20)-C(22)	110.0(5)
C(21)-C(20)-C(19)	110.9(5)	C(22)-C(20)-C(19)	109.3(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for 3.

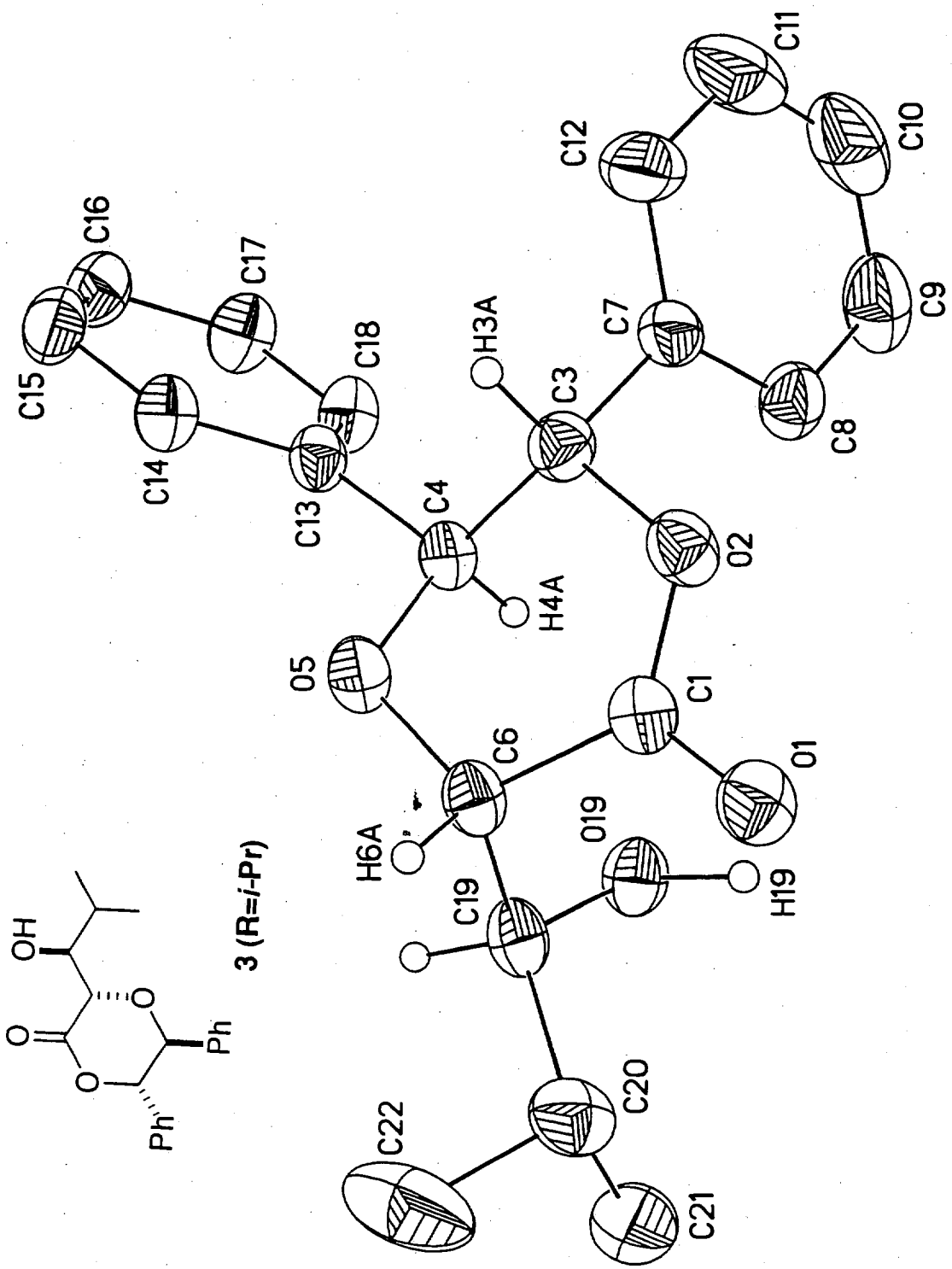
The anisotropic displacement factor exponent takes the form:

$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
O(1)	74(2)	75(2)	62(2)	-16(2)	32(2)	1(2)
C(1)	66(3)	45(3)	61(3)	-6(2)	31(2)	-8(3)
O(2)	62(2)	77(2)	61(2)	-14(2)	31(1)	-3(2)
C(3)	65(2)	60(3)	55(2)	0(2)	28(2)	5(3)
C(4)	59(3)	56(3)	58(2)	1(2)	30(2)	-7(2)
O(5)	66(2)	71(2)	61(2)	-3(2)	34(1)	-12(2)
C(6)	59(2)	54(3)	58(2)	-9(2)	30(2)	-11(2)
C(7)	56(3)	79(4)	52(2)	-2(3)	24(2)	5(3)
C(8)	72(3)	88(4)	62(3)	2(3)	25(2)	-19(3)
C(9)	105(4)	133(6)	89(4)	-14(4)	52(3)	-43(5)
C(10)	79(4)	192(10)	108(5)	-46(6)	46(4)	-53(6)
C(11)	61(4)	182(9)	106(5)	-35(6)	18(3)	9(5)
C(12)	77(3)	106(5)	75(3)	-4(3)	25(3)	21(4)
C(13)	64(2)	52(3)	54(2)	-1(3)	34(2)	2(3)
C(14)	75(3)	60(3)	66(3)	2(3)	35(2)	-10(3)
C(15)	86(3)	83(4)	65(3)	16(3)	35(2)	-6(4)
C(16)	86(3)	102(5)	51(3)	7(3)	29(2)	-1(4)
C(17)	101(4)	71(4)	65(3)	-9(3)	33(3)	-13(3)
C(18)	92(3)	59(3)	68(3)	0(3)	43(3)	-7(3)
C(19)	71(3)	67(4)	63(3)	-8(3)	42(2)	-4(3)
O(19)	81(2)	59(2)	65(2)	-4(2)	35(2)	-10(2)
C(20)	73(3)	84(4)	79(3)	-15(3)	34(3)	11(3)
C(21)	90(4)	120(6)	99(4)	13(5)	40(3)	20(5)
C(22)	70(3)	105(6)	217(7)	1(6)	28(4)	-12(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 3.

	x	y	z	U(eq)
H(3A)	6734(3)	-52(9)	3029(3)	70
H(4A)	5713(3)	4391(9)	2854(3)	67
H(6A)	3447(4)	609(8)	1375(3)	66
H(8A)	7184(4)	5326(11)	1994(3)	88
H(9A)	9019(5)	7045(15)	2417(4)	126
H(10A)	10705(6)	5420(19)	3657(6)	148
H(11A)	10567(6)	2051(18)	4432(6)	145
H(12A)	8752(4)	282(12)	4011(4)	105
H(14A)	5582(4)	-305(10)	4461(3)	78
H(15A)	6307(4)	-326(11)	6291(4)	91
H(16A)	7399(4)	2699(12)	7234(4)	95
H(17A)	7793(5)	5811(10)	6374(4)	94
H(18A)	7068(4)	5822(9)	4536(3)	83
H(19A)	3072(4)	4464(9)	1734(3)	75
H(19)	4398(3)	5239(6)	599(2)	99
H(20A)	2605(4)	3282(11)	-394(4)	93
H(21A)	2487(5)	7279(14)	-299(4)	153
H(21B)	1596(5)	7032(14)	271(4)	153
H(21C)	1213(5)	6252(14)	-912(4)	153
H(22A)	1665(5)	960(14)	471(6)	209
H(22B)	702(5)	2361(14)	-439(6)	209
H(22C)	1084(5)	3141(14)	744(6)	209


C16
C17
C15
C18
C14
C13
C4
H3A
C3
C7
C12
C11
C10
C9
C8
O2
H4A
O5
C1
C6
O1
H6A
O19
H19
C19
C20
C22
C21
OH
O
O
Ph
Ph

3 (R=*i*-Pr)

Table 1. Crystal data and structure refinement for 3 (R=Ph).

Identification code	ma3
Empirical formula	$C_{23}H_{20}O_4$
Formula weight	360.39
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell dimensions	a = 9.7140(10) Å alpha = 90° b = 9.316(2) Å beta = 106.193(8)° c = 10.9660(10) Å gamma = 90°
Volume, Z	953.0(2) $Å^3$, 2
Density (calculated)	1.256 Mg/m^3
Absorption coefficient	0.085 mm^{-1}
F(000)	380
Crystal size	0.5 x 0.4 x 0.4 mm
Θ range for data collection	2.48 to 27.43°
Limiting indices	$-12 \le h \le 0$, $-12 \le k \le 0$, $-13 \le l \le 14$
Reflections collected	2426
Independent reflections	2297 (R_{int} = 0.0146)
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2296 / 1 / 245
Goodness-of-fit on F^2	1.062
Final R indices [I>2σ(I)]	R1 = 0.0424, wR2 = 0.0995
R indices (all data)	R1 = 0.0635, wR2 = 0.1147
Absolute structure parameter	1(2)
Extinction coefficient	0.004(4)
Largest diff. peak and hole	0.117 and -0.123 $eÅ^{-3}$

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for R=Ph U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(1)	4469(3)	1660(3)	-606(2)	83(1)
C(1)	3761(3)	1744(3)	137(3)	60(1)
O(2)	2804(2)	721(2)	124(2)	61(1)
C(3)	1860(3)	794(3)	956(2)	50(1)
C(4)	2615(3)	1598(3)	2161(2)	46(1)
O(5)	3005(2)	3003(2)	1823(2)	53(1)
C(6)	3953(3)	2988(3)	1051(2)	54(1)
C(7)	1484(3)	-719(3)	1220(2)	52(1)
C(8)	2435(4)	-1827(3)	1332(3)	69(1)
C(9)	2091(6)	-3185(4)	1697(3)	88(1)
C(10)	817(6)	-3396(4)	1968(4)	99(2)
C(11)	-138(5)	-2299(5)	1859(4)	90(1)
C(12)	175(4)	-953(4)	1463(3)	69(1)
C(13)	1671(3)	1806(3)	3020(2)	49(1)
C(14)	460(3)	2673(3)	2652(3)	58(1)
C(15)	-407(4)	2872(4)	3443(4)	78(1)
C(16)	-89(5)	2214(5)	4596(4)	88(1)
C(17)	1086(5)	1351(4)	4971(3)	84(1)
C(18)	1979(4)	1152(3)	4188(3)	63(1)
C(19)	5544(3)	3152(3)	1836(3)	60(1)
O(19)	5752(3)	4510(2)	2443(2)	77(1)
C(20)	5986(3)	2001(4)	2820(3)	62(1)
C(21)	6400(4)	665(4)	2498(4)	86(1)
C(22)	6697(5)	-426(5)	3368(7)	116(2)
C(23)	6601(5)	-214(7)	4559(6)	117(2)
C(24)	6200(4)	1099(7)	4917(4)	101(2)
C(25)	5895(3)	2231(5)	4039(3)	73(1)

Table 3. Bond lengths [Å] and angles [°] for 3 (R=Ph).

O(1)-C(1)	1.207(3)	C(1)-O(2)	1.328(4)
C(1)-C(6)	1.509(4)	O(2)-C(3)	1.464(3)
C(3)-C(7)	1.504(4)	C(3)-C(4)	1.519(3)
C(4)-O(5)	1.440(3)	C(4)-C(13)	1.499(3)
O(5)-C(6)	1.414(3)	C(6)-C(19)	1.553(4)
C(7)-C(8)	1.367(4)	C(7)-C(12)	1.388(4)
C(8)-C(9)	1.395(5)	C(9)-C(10)	1.365(6)
C(10)-C(11)	1.363(6)	C(11)-C(12)	1.388(5)
C(13)-C(18)	1.374(4)	C(13)-C(14)	1.391(4)
C(14)-C(15)	1.380(4)	C(15)-C(16)	1.360(5)
C(16)-C(17)	1.363(6)	C(17)-C(18)	1.392(4)
C(19)-O(19)	1.418(4)	C(19)-C(20)	1.496(4)
C(20)-C(25)	1.381(5)	C(20)-C(21)	1.384(5)
C(21)-C(22)	1.369(6)	C(22)-C(23)	1.349(7)
C(23)-C(24)	1.374(8)	C(24)-C(25)	1.402(6)

O(1)-C(1)-O(2)	118.4(3)	O(1)-C(1)-C(6)	120.7(3)
O(2)-C(1)-C(6)	120.9(2)	C(1)-O(2)-C(3)	121.2(2)
O(2)-C(3)-C(7)	107.7(2)	O(2)-C(3)-C(4)	109.3(2)
C(7)-C(3)-C(4)	112.3(2)	O(5)-C(4)-C(13)	107.2(2)
O(5)-C(4)-C(3)	108.8(2)	C(13)-C(4)-C(3)	112.3(2)
C(6)-O(5)-C(4)	114.1(2)	O(5)-C(6)-C(1)	115.1(2)
O(5)-C(6)-C(19)	112.4(2)	C(1)-C(6)-C(19)	111.3(2)
C(8)-C(7)-C(12)	119.8(3)	C(8)-C(7)-C(3)	122.0(3)
C(12)-C(7)-C(3)	118.0(3)	C(7)-C(8)-C(9)	120.0(4)
C(10)-C(9)-C(8)	119.8(4)	C(11)-C(10)-C(9)	120.6(4)
C(10)-C(11)-C(12)	120.1(4)	C(7)-C(12)-C(11)	119.6(4)
C(18)-C(13)-C(14)	118.4(3)	C(18)-C(13)-C(4)	121.1(3)
C(14)-C(13)-C(4)	120.5(2)	C(15)-C(14)-C(13)	120.7(3)
C(16)-C(15)-C(14)	120.3(4)	C(15)-C(16)-C(17)	120.0(3)
C(16)-C(17)-C(18)	120.4(3)	C(13)-C(18)-C(17)	120.3(3)
O(19)-C(19)-C(20)	109.1(2)	O(19)-C(19)-C(6)	109.7(3)
C(20)-C(19)-C(6)	111.7(2)	C(25)-C(20)-C(21)	119.2(3)
C(25)-C(20)-C(19)	120.3(3)	C(21)-C(20)-C(19)	120.4(3)
C(22)-C(21)-C(20)	120.6(4)	C(23)-C(22)-C(21)	120.6(5)
C(22)-C(23)-C(24)	120.5(4)	C(23)-C(24)-C(25)	119.7(4)
C(20)-C(25)-C(24)	119.3(4)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for 3 (R=Ph).

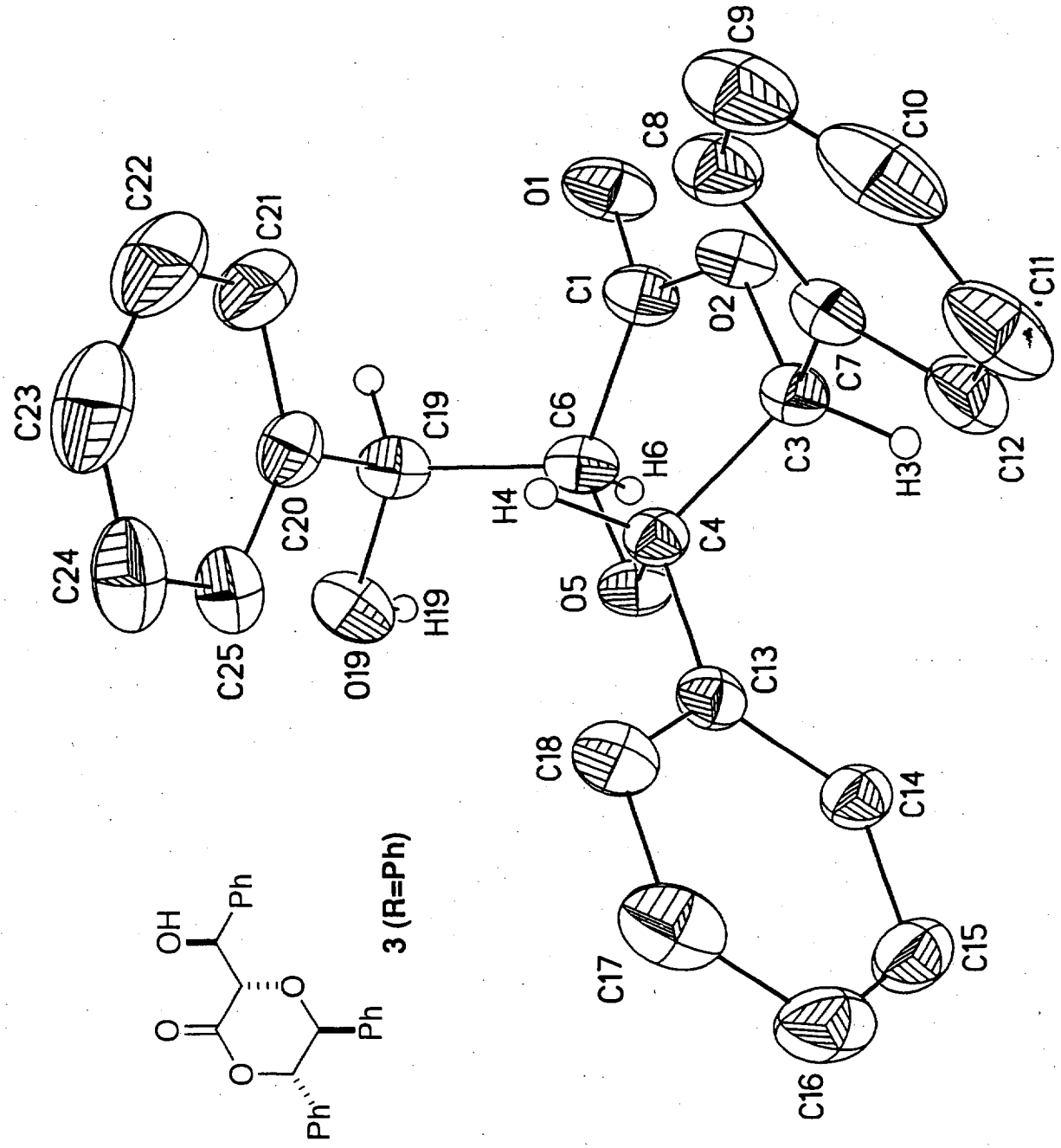
The anisotropic displacement factor exponent takes the form:

$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
O(1)	115(2)	80(2)	73(1)	-16(1)	56(1)	-20(2)
C(1)	79(2)	54(2)	52(1)	-1(1)	26(1)	-5(2)
O(2)	83(1)	52(1)	54(1)	-10(1)	27(1)	-7(1)
C(3)	58(2)	46(1)	48(1)	-1(1)	16(1)	1(1)
C(4)	53(1)	34(1)	50(1)	-1(1)	14(1)	1(1)
O(5)	68(1)	35(1)	61(1)	-2(1)	28(1)	-1(1)
C(6)	76(2)	37(1)	54(2)	5(1)	26(1)	0(1)
C(7)	67(2)	45(2)	42(1)	-6(1)	10(1)	-6(1)
C(8)	91(2)	49(2)	64(2)	-2(2)	18(2)	7(2)
C(9)	140(4)	45(2)	73(2)	-1(2)	20(2)	4(2)
C(10)	172(5)	52(2)	67(2)	-8(2)	23(3)	-35(3)
C(11)	120(3)	80(3)	73(2)	-22(2)	31(2)	-51(3)
C(12)	74(2)	61(2)	69(2)	-16(2)	15(2)	-17(2)
C(13)	55(1)	39(1)	52(1)	-7(1)	16(1)	-9(1)
C(14)	58(2)	47(2)	72(2)	-9(1)	22(1)	-3(1)
C(15)	60(2)	67(2)	113(3)	-24(2)	36(2)	-7(2)
C(16)	91(2)	92(3)	100(3)	-21(2)	60(2)	-23(2)
C(17)	114(3)	78(3)	72(2)	-3(2)	47(2)	-27(2)
C(18)	78(2)	51(2)	62(2)	-2(1)	24(2)	-8(2)
C(19)	68(2)	55(2)	65(2)	-2(2)	31(1)	-10(2)
O(19)	105(2)	54(1)	72(1)	-3(1)	26(1)	-23(1)
C(20)	49(2)	60(2)	77(2)	10(2)	19(1)	-3(1)
C(21)	75(2)	71(2)	113(3)	4(2)	28(2)	13(2)
C(22)	101(3)	78(3)	162(5)	30(3)	27(3)	27(3)
C(23)	83(3)	104(4)	146(5)	64(4)	1(3)	9(3)
C(24)	72(2)	138(4)	82(2)	38(3)	3(2)	-12(3)
C(25)	61(2)	89(2)	64(2)	13(2)	8(2)	-10(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 3 (R=Ph).

	x	y	z	U(eq)
H(3)	982(3)	1308(3)	518(2)	60
H(4)	3480(3)	1074(3)	2615(2)	55
H(6)	3728(3)	3851(3)	522(2)	65
H(8)	3311(4)	-1675(3)	1164(3)	83
H(9)	2728(6)	-3944(4)	1755(3)	106
H(10)	598(6)	-4297(4)	2229(4)	119
H(11)	-1001(5)	-2453(5)	2050(4)	108
H(12)	-490(4)	-213(4)	1361(3)	83
H(14)	233(3)	3124(3)	1864(3)	70
H(15)	-1211(4)	3457(4)	3188(4)	93
H(16)	-673(5)	2354(5)	5128(4)	105
H(17)	1293(5)	893(4)	5754(3)	100
H(18)	2788(4)	574(3)	4457(3)	75
H(19A)	6152(3)	3092(3)	1259(3)	72
H(19)	5598(3)	5246(2)	1820(2)	115
H(21)	6477(4)	506(4)	1682(4)	103
H(22)	6967(5)	-1321(5)	3137(7)	139
H(23)	6807(5)	-964(7)	5142(6)	141
H(24)	6132(4)	1237(7)	5738(4)	121
H(25)	5635(3)	3127(5)	4276(3)	88


C9
C8
C10
O1
C22
C21
C1
O2
C11
C23
C7
C6
C19
C3
H3
C12
H6
H4
C20
C4
C24
O5
H19
C25
O19
C13
C18
C14
C17
C15
C16
OH
Ph
O
O
Ph
Ph
3 (R=Ph)