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(FAB) calcd for $C_{13}H_{20}I$ $(MH)^+$: 303.0610, found: 303.0601; Anal. Calcd for $C_{13}H_{19}I$: C, 51.67; H, 6.34; found: C, 51.67, H, 6.46.

(*R*)-2-Methylhexanal (83). Aldehyde **83** was prepared by the reduction of amide **13** with $LiAlH(OEt)_3$, as described for aldehyde **82**. Thus, treatment of amide **13** (990 mg, 3.57 mmol, 1 equiv) with $LiAlH(OEt)_3$ (8.21 mmol, 2.30 equiv) at 0 °C for 1.1 h afforded aldehyde **83** (318 ± 10 mg, 78 ± 3% yield; yield based on capillary GC analysis using the (*R*)-α-methylbenzyl amide of (*R*)-2-methylhexanoic acid as an internal standard). Oxidation of aldehyde **83** to the corresponding carboxylic acid (**47**), as described for aldehyde **82**, and chiral capillary GC analysis of the corresponding (*R*)-α-methylbenzyl amide, as described for acid **46**, established that aldehyde **83** was of 98% ee: ^1H NMR (300 MHz, C_6D_6) δ 9.27 (d, 1H, J = 1.8 Hz), 1.83 (m, 1H), 0.85–1.15 (m, 6H), 0.78 (t, 3H, J = 7.0 Hz), 0.75 (d, 3H, J = 7.0 Hz); FTIR (neat, cm^{-1}) 1729 (s, C=O); HRMS (FAB) calcd for $C_7H_{14}O$ $(M)^+$: 114.1045, found: 114.1047.

(*S*)-α-Methyl Benzenepropanal (84). Aldehyde **84** was prepared by the reduction of amide **17** with $LiAlH(OEt)_3$, as described for aldehyde **82**. Thus, treatment of amide **17** (721 mg, 2.32 mmol, 1 equiv) with $LiAlH(OEt)_3$ (5.33 mmol, 2.30 equiv) at 0 °C for 1.2 h afforded aldehyde **84** as a colorless liquid (263 mg, 77%) after purification by flash column chromatography (10% ethyl acetate–hexanes). Oxidation of aldehyde **84** to the corresponding carboxylic acid (**54**), as described for aldehyde **82**, and chiral capillary GC analysis of the corresponding (*R*)-α-methylbenzyl amide, as described for acid **46**, established that aldehyde **84** was of 94% ee. Spectroscopic data of aldehyde **84** were identical to those of its enantiomer, (*R*)-α-methyl benzenepropanal (**82**).

(*S*)-α-Butyl Benzenepropanal (85). Aldehyde **85** was prepared by the reduction of amide **18** with $LiAlH(OEt)_3$, as described for aldehyde **82**. Thus, treatment of amide **18** (985 mg, 2.79 mmol, 1 equiv) with $LiAlH(OEt)_3$ (6.41 mmol, 2.30 equiv) at 0 °C for 45 min afforded aldehyde **85** as a colorless liquid (427 mg, 80%) after purification by flash column chromatography (gradient elution, 100% hexanes → 9% ethyl acetate–

hexanes). Oxidation of aldehyde **85** to the corresponding carboxylic acid (**55**), as described for aldehyde **82**, and chiral capillary GC analysis of corresponding (*R*)-α-methylbenzyl amide, as described for acid **46**, established that aldehyde **85** was of 97% ee: ^{1}H NMR (300 MHz, C_6D_6) δ 9.34 (d, 1H, J = 2.3 Hz), 6.9–7.3 (m, 5H), 2.71 (dd, 1H, J_1 =13.9 Hz, J_2 = 7.2 Hz), 2.36 (dd, 1H, J_1 =13.9 Hz, J_2 = 7.0 Hz), 2.22 (m, 1H), 1.31 (m, 1H), 0.9–1.2 (m, 5H), 0.74 (t, 3H, J = 6.7 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 138.9, 128.9, 128.5, 126.3, 53.4, 35.0, 29.0, 28.3, 22.7, 13.8; FTIR (neat, cm^{-1}) 1726 (s, C=O); HRMS (FAB) calcd for $C_{13}H_{18}O$ $(M)^+$: 190.1358, found: 190.1346.

(*R*)-α-Butyl Benzenepropanal (86). Aldehyde **86** was prepared by the reduction of amide **20** with $LiAlH(OEt)_3$, as described for aldehyde **82**. Thus, treatment of amide **20** (996 mg, 2.82 mmol, 1 equiv) with $LiAlH(OEt)_3$ (6.48 mmol, 2.30 equiv) at 0 °C for 45 min afforded aldehyde **86** as a colorless liquid (441 mg, 82%) after purification by flash column chromatography (gradient elution, 100% hexanes → 9% ethyl acetate–hexanes). Oxidation of aldehyde **86** to the corresponding carboxylic acid (**48**), as described for aldehyde **82**, and chiral capillary GC analysis of corresponding (*R*)-α-methylbenzyl amide, as described for acid **46**, established that aldehyde **86** was of 97% ee. Spectroscopic data of aldehyde **86** were identical to those of its enantiomer, (*S*)-α-butyl benzenepropanal (**85**).

(*S*)-α-Ethyl Benzeneacetaldehyde (87). Aldehyde **87** was prepared by the reduction of amide **21** with $LiAlH(OEt)_3$, as described for aldehyde **82**. Thus, treatment of amide **21** (1.50 g, 4.80 mmol, 1 equiv) with $LiAlH(OEt)_3$ (11.0 mmol, 2.30 equiv) at 0 °C for 55 min afforded aldehyde **87** as a colorless liquid (569 mg, 80%) after purification by flash column chromatography (10% ethyl acetate–hexanes). Oxidation of aldehyde **87** to the corresponding carboxylic acid (**49**), as described for aldehyde **82**, and chiral capillary GC analysis of the corresponding (*R*)-α-methylbenzyl amide, as described for acid **46**, established that aldehyde **87** was of 90% ee: ^{1}H NMR (300 MHz, C_6D_6) δ 9.34 (d, 1H, J = 1.8 Hz), 6.80–7.15 (m, 5H), 2.87 (m, 1H), 1.87 (m, 1H), 1.48 (m, 1H),

0.66 (t, 3H, J = 7.4 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.9, 136.2, 128.9, 128.7, 127.4, 60.7, 22.8, 11.6; FTIR (neat, cm^{-1}) 1727 (s, C=O); HRMS (FAB) calcd for $C_{10}H_{13}O$ $(MH)^+$: 149.0966, found: 149.0972.

(*R*)-2-Methyl-1-phenyl-1-hexanone (90). Ketone **90** was prepared by the addition of phenyllithium to amide **13** using a procedure analogous to that described for the preparation of ketone **88**. Thus, a solution of phenyllithium in 70% cyclohexane–ether (1.94 M, 1.20 mL, 2.33 mmol, 2.44 equiv) was added to a suspension of amide **13** (264 mg, 0.953 mmol, 1 equiv) in tetrahydrofuran (10 mL) to afford ketone **90** as a clear liquid (168 mg, 93%) after purification by flash column chromatography (gradient elution of ethyl acetate–hexanes, 2 → 10%). High resolution ^{1}H NMR analysis (400 MHz, C_6D_6) of the Mosher ester derivatives[3] of the diastereomeric alcohol mixture obtained by the reduction of ketone **90** with lithium aluminum hydride (as described for ketone **89**) established that ketone **90** was of ≥95% ee: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 (m, 2H), 7.50 (m, 3H), 3.46 (sx, 1H, J = 6.8 Hz), 1.80 (m, 1H), 1.45 (m, 1H), 1.30 (m, 4H), 1.19 (d, 3H, J = 6.9 Hz), 0.87 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.5, 136.7, 132.7, 128.6, 128.2, 40.5, 33.4, 29.6, 22.8, 17.2, 13.9; FTIR (neat, cm^{-1}) 1682 (s, C=O); HRMS (EI) calcd for $C_{13}H_{18}O$ $(M)^+$: 190.1358, found: 190.1363.

***(R)*-2-(Benzyloxymethyl)-1-phenyl-1-propanone (91).** Ketone **91** was prepared by the addition of phenyllithium to amide **14** using a procedure analogous to that described for the preparation of ketone **88.** Thus, a solution of phenyllithium in 70% cyclohexane–ether (1.94 M, 0.910 mL, 1.77 mmol, 2.40 equiv) was added to a suspension of amide **14** (251 mg, 0.736 mmol, 1 equiv) in ether (7 mL) to afford ketone **91** as a clear liquid (119 mg, 64%) after purification by flash column chromatography (gradient elution of ethyl acetate–hexanes, 7.5 → 30%). High resolution ^{1}H NMR analysis (400 MHz, C_6D_6) of the Mosher ester derivatives[3] of the diastereomeric alcohol mixture obtained by the reduction of ketone **91** with lithium aluminum hydride (as described for ketone **89**) established that ketone **91** was of ≥95% ee: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.0

(m, 2H), 7.55 (m, 1H), 7.45 (m, 2H), 7.3 (m, 5H), 4.51 (dd, 2H, J_1 = 17.6 Hz, J_2 = 12.1 Hz), 3.85 (m, 2H), 3.55 (m, 1H), 1.23 (d, 3H, J = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.8, 138.3 136.9, 133.0, 128.6, 128.5, 128.4, 73.4, 72.5, 41.6, 15.0; FTIR (neat, cm^{-1}) 1682 (s, C=O); HRMS (EI) calcd for $C_{17}H_{19}O_2$ $(MH)^+$: 255.1385, found: 255.1383.

***(R)*-3-(Benzyloxymethyl)-2-butanone (92).** Ketone **92** was prepared by the addition of methyllithium to amide **14** using a procedure analogous to that dscribed for the preparation of ketone **88**. Thus, a solution of methyllithium in ether (1.25 M, 1.65 mL, 2.06 mmol, 2.39 equiv) was added to a suspension of amide **14** (294 mg, 0.862 mmol, 1 equiv) in ether (10 mL) to afford ketone **92** as a clear liquid (112 mg, 68%) after purification by flash column chromatography (gradient elution of ethyl acetate–hexanes, 15 → 50%). High resolution ^{1}H NMR analysis (400 MHz, $CDCl_3$) of the Mosher ester derivatives[3] of the diastereomeric alcohol mixture obtained by the reduction of ketone **92** with lithium aluminum hydride (as described for ketone **89**) established that ketone **92** was of 95% ee: ^{1}H NMR (300 MHz, C_6D_6) δ 7.05–7.25 (m, 5H), 4.20 (dd, 2H, J_1 = 16.1 Hz, J_2 = 12.0 Hz), 3.39 (dd, 1H, J_1 = 9.0 Hz, J_2 = 7.3 Hz), 3.18 (dd, 1H, J_1 = 9.0 Hz, J_2 = 5.4 Hz), 2.47 (m, 1H), 1.80 (s, 3H), 0.85 (d, 3H, J = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 211.1, 138.2, 128.4, 127.7, 73.3, 72.2, 47.3, 29.1, 13.4; FTIR (neat, cm^{-1}) 1715 (s, C=O); HRMS (EI) calcd for $C_{12}H_{16}O_2$ $(M)^+$: 192.1150, found: 192.1149.

***(R)*-2-Butyl-1,3-diphenyl-1-propanone (93).** Ketone **93** was prepared by the addition of phenyllithium to amide **20** using a procedure analogous to that described for the preparation of ketone **88.** Thus, a solution of phenyllithium in 70% cyclohexane–ether (1.94 M, 0.91 mL, 1.77 mmol, 2.41 equiv) was added to a suspension of amide **20** (259 mg, 0.733 mmol, 1 equiv) in ether (8 mL) to afford ketone **93** as a clear liquid (186 mg, 96%) after purification by flash column chromatography (gradient elution of ethyl acetate–hexanes, 3 → 5%). High resolution ^{1}H NMR analysis (400 MHz, $CDCl_3$) of the Mosher ester derivatives[3] of the diastereomeric alcohol mixture obtained by the reduction of ketone

93 with lithium aluminum hydride (as described for ketone **89**) established that ketone **93** was of ≥95% ee: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.1–7.9 (m, 10H), 3.74 (m, 1H), 3.10 (dd, 1H, J_1 = 13.6 Hz, J_2 = 7.7 Hz), 2.78 (dd, 1H, J_1 = 13.6 Hz, J_2 = 6.5 Hz), 1.80 (m, 1H), 1.55 (m, 1H), 1.25 (m, 4H), 0.80 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.0, 140.0, 137.5, 132.8, 129.0, 128.5, 128.3, 128.1, 126.1, 48.3, 38.2, 32.1, 29.5, 22.8, 13.9; FTIR (neat, cm^{-1}) 1679 (s, C=O); HRMS (EI) calcd for $C_{19}H_{22}O$ $(M)^+$: 266.1671, found: 266.1673.

***(R)*-2-Butyl-1-phenyl-3-heptanone (94).** Ketone **94** was prepared by the addition of *n*-butyllithium to amide **20** using a procedure analogous to that described for the preparation of ketone **89**. Thus, a solution of *n*-butyllithium in hexanes (1.71 M, 3.50 mL, 5.99 mmol, 2.11 equiv) was added to a suspension of amide **20** (1.00 g, 2.83 mmol, 1 equiv) in ether (30 mL) to afford ketone **94** as a clear liquid (652 mg, 94%) after purification by flash column chromatography (gradient elution of ethyl acetate–hexanes, 2.5 → 10%). High resolution ^{1}H NMR analysis (400 MHz, C_6D_6) of the Mosher ester derivatives[3] of the diastereomeric alcohol mixture obtained by the reduction of ketone **94** with lithium aluminum hydride (as described for ketone **89**) established that ketone **94** was of ≥95% ee: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20 (m, 5H), 2.80 (m, 2H), 2.65 (dd, 1H, J_1 = 12.0 Hz, J_2 = 5.0 Hz), 2.28 (dt, 1H, J_1 = 17.2 Hz, J_2 = 7.3 Hz), 2.11 (dt, 1H, J_1 = 17.2 Hz, J_2 = 7.3 Hz), 1.65 (m, 1H), 1.40 (m, 3H), 1.25 (m, 6H), 0.87 (t, 3H, J = 7.0 Hz), 0.81 (t, 3H, J = 7.3 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 214.6, 139.9, 128.9, 126.1, 54.0, 43.4, 38.2, 31.6, 29.5, 25.2, 22.8, 22.2, 13.9, 13.8; FTIR (neat, cm^{-1}) 1712 (s, C=O); HRMS (EI) calcd for $C_{17}H_{26}O$ $(M)^+$: 246.1984, found: 246.1995.

***(R)*-3-(Phenylmethyl)-2-heptanone (95).** Ketone **95** was prepared by the addition of methyllithium to amide **20** using a procedure analogous to that described for the preparation of ketone **89**. Thus, a solution of methyllithium in ether (1.30 M, 6.20 mL, 8.06 mmol, 2.76 equiv) was added to a suspension of amide **20** (1.03 g, 2.92 mmol, 1 equiv) in ether (30 mL) to afford ketone **95** as a clear liquid (582 mg, 98%) after

purification by flash column chromatography (15% ethyl acetate–hexanes). High resolution ^{1}H NMR analysis (400 MHz, C_6D_6) of the Mosher ester derivatives[3] of the diastereomeric alcohol mixture obtained by the reduction of ketone **95** with lithium aluminum hydride (as described for ketone **89**) established that ketone **95** was of ≥95% ee: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.2 (m, 5H), 2.85 (m, 2H), 2.69 (dd, 1H, J_1 = 12.1 Hz, J_2 = 5.1 Hz), 2.00 (s, 3H), 1.65 (m, 1H), 1.45 (m, 1H), 1.27 (m, 4H), 0.88 (t, 3H, J = 6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 212.5, 139.6, 128.8, 128.4, 126.2, 54.7, 37.9, 31.4, 30.2, 29.4, 22.7, 13.9; FTIR (neat, cm^{-1}) 1713 (s, C=O); HRMS (EI) calcd for $C_{14}H_{20}O$ $(M)^+$: 204.1514, found: 204.1521.

***(S)*-3-Phenyl-2-pentanone (96).** Ketone **96** was prepared by the addition of methyllithium to amide **21** using a procedure analogous to that described for the preparation of ketone **88**. Thus, a solution of methyllithium in ether (1.40 M, 1.40 mL, 1.96 mmol, 2.38 equiv) was added to a solution of amide **21** (256 mg, 0.823 mmol, 1 equiv) in ether (8 mL) to afford ketone **96** as a clear liquid (72 mg, 54%) after purification by flash column chromatography (gradient elution of ethyl acetate–hexanes, 15 → 40%). High resolution ^{1}H NMR analysis (400 MHz, C_6D_6) of the Mosher ester derivatives[3] of the diastereomeric alcohol mixture obtained by the reduction of ketone **96** with lithium aluminum hydride (as described for ketone **89**) established that ketone **96** was of 88% ee: ^{1}H NMR (300 MHz, C_6D_6) δ 6.9–7.1 (m, 5H), 3.08 (t, 1H, J = 7.3 Hz), 2.03 (m, 1H), 1.66 (s, 3H), 1.66 (m, 1H), 0.71 (t, 3H, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.5, 139.0, 128.9, 128.3, 127.2, 61.6, 29.1, 25.0, 12.0; FTIR (neat, cm^{-1}) 1708 (s, C=O); HRMS (EI) calcd for $C_{11}H_{14}O$ $(M)^+$: 162.1045, found: 162.1052.

***(R)*-5-(*tert*-Butyldimethylsilyloxy)-3-methyl-2-pentanone (97).** Ketone **97** was prepared by the addition of methyllithium to amide **29** using a procedure analogous to that described for the preparation of ketone **89**. Thus, a solution of methyllithium in ether (1.00 M, 1.68 mL, 1.68 mmol, 2.50 equiv) was added to a solution of amide **29** (255 mg, 0.673 mmol, 1 equiv) in ether (3 mL) to afford ketone **97** as a clear

liquid (138 mg, 89%) after purification by flash column chromatography (10% ethyl acetate–hexanes). High resolution ^{1}H NMR analysis (500 MHz, C_6D_6) of the Mosher ester derivative[3] of the alcohol obtained from the desilylation of ketone **97** with tetrabutylammonium fluoride established that ketone **97** was of ≥95% ee: ^{1}H NMR (500 MHz, C_6D_6) δ 3.46 (m, 2H), 2.47 (m, 1H), 1.85 (m, 1H), 1.79 (s, 3H), 1.36 (m, 1H), 0.95 (s, 9H), 0.88 (d, 3H, J = 7.1 Hz), 0.02 (s, 6H); ^{13}C NMR (125 MHz, C_6D_6) δ 209.9, 61.0, 43.7, 36.0, 27.7, 26.1, 18.4, 16.3, –5.4; FTIR (neat, cm^{-1}) 1715 (s, C=O); Anal. Calcd for $C_{12}H_{26}O_2Si$: C, 62.56; H, 11.38; found: C, 62.77; H, 11.63.

(*R*)-5-(*tert*-Butyldimethylsilyloxy)-3-phenylmethyl-2-pentanone (98). Ketone **98** was prepared by the addition of methyllithium to amide **30** using a procedure analogous to that described for the preparation of ketone **89**. Thus, a solution of methyllithium in ether (1.00 M, 1.71 mL, 1.71 mmol, 2.50 equiv) was added to a solution of amide **30** (313 mg, 0.685 mmol, 1 equiv) in ether (3 mL) to afford ketone **98** as a colorless liquid (194 mg, 92%) after purification by flash column chromatography (5% ethyl acetate–hexanes). High resolution ^{1}H NMR analysis (500 MHz, C_6D_6) of the Mosher ester derivative[3] of the alcohol obtained from the desilylation of ketone **98** with tetrabutylammonium fluoride established that ketone **98** was of ≥95% ee: ^{1}H NMR (500 MHz, C_6D_6) δ 7.09 (m, 2H), 7.06 (m, 3H), 3.42 (m, 2H), 2.86 (m, 1H), 2.82 (dd, 1H, J_1 = 12.9 Hz, J_2 = 8.2 Hz), 2.49 (dd, 1H, J_1 = 12.7 Hz, J_2 = 6.0 Hz), 1.82 (m, 1H), 1.76 (s, 3H), 1.49 (m, 1H), 0.93 (s, 9H), –0.01 (2 s, 6H); ^{13}C NMR (125 MHz, C_6D_6) δ 209.4, 140.1, 129.2, 128.7, 126.5, 61.2, 53.3, 51.3, 38.3, 34.7, 29.9, 26.1, 18.4, –5.4; FTIR (neat, cm^{-1}) 1715 (s, C=O); HRMS (FAB) calcd for $C_{18}H_{31}O_2Si$ $(MH)^+$: 307.2094, found: 307.2093.

(3*R*,5*R*)-5-(*tert*-Butyldimethylsilyloxy)-3-methyl-2-hexanone (99). Ketone **99** was prepared by the addition of methyllithium to amide **43** using a procedure analogous to that described for the preparation of ketone **89**. Thus, a solution of methyllithium in ether (1.40 M, 0.154 mL, 0.215 mmol, 2.49 equiv) was added to a

solution of amide **43** (33.9 mg, 0.0862 mmol, 1 equiv) in ether (1 mL) to afford ketone **99** as a volatile colorless liquid (19.3 mg, 92%) after purification by flash column chromatography (25% ethyl acetate–hexanes). Chiral capillary GC analysis[6] of the acetate ester derivative of the alcohol obtained by desilylation of ketone **99** with tetrabutylammonium fluoride established that ketone **99** was of 97% ee: ^{1}H NMR (500 MHz, C_6D_6) δ 3.79 (m, 1H), 2.55 (m, 1H), 1.84 (ddd, 1H, J_1 = 13.5 Hz, J_2 = 8.8 Hz, J_3 = 4.1 Hz), 1.82 (s, 3H), 1.27 (ddd, 1H, J_1 = 12.5 Hz, J_2 = 8.0 Hz, J_3 = 4.4 Hz), 1.02 (d, 3H, J = 6.2 Hz), 0.95 (s, 9H), 0.87 (d, 3H, J = 7.2 Hz), 0.02 (s, 6H); ^{13}C NMR (125 MHz, C_6D_6) δ 67.2, 43.5, 43.0, 28.0, 26.1, 24.2, 17.6, –4.2, –4.6; FTIR (neat, cm^{-1}) 1714 (s, C=O); HRMS (FAB) calcd for $C_{13}H_{29}O_2Si$ $(MH)^+$: 245.1938, found: 245.1937.

(3*R*,5*S*)-5-(*tert*-Butyldimethylsilyloxy)-3-methyl-2-hexanone (100). Ketone **100** was prepared by the addition of methyllithium to amide **44** using a procedure analogous to that described for the preparation of ketone **89**. Thus, a solution of methyllithium in ether (1.40 M, 0.196 mL, 0.275 mmol, 2.52 equiv) was added to a solution of amide **44** (43.2 mg, 0.109 mmol, 1 equiv) in ether (1 mL) to afford ketone **100** as a volatile colorless liquid (19.3 mg, 92% yield) after purification by flash column chromatography (25% ethyl acetate–hexanes). Chiral capillary GC analysis[6] of the acetate ester derivative of the alcohol obtained by desilylation of ketone **100** with tetrabutylammonium fluoride established that ketone **100** was of 96% ee: ^{1}H NMR (500 MHz, C_6D_6) δ 3.67 (m, 1H), 2.51 (m, 1H), 1.82 (ddd, 1H, J_1 = 13.9 Hz, J_2 = 8.4 Hz, J_3 = 5.7 Hz), 1.77 (s, 3H), 1.12 (ddd, 1H, J_1 = 12.2 Hz, J_2 = 9.0 Hz, J_3 = 3.9 Hz), 0.99 (d, 3H, J = 6.1 Hz), 0.96 (s, 9H), 0.91 (d, 3H, J = 6.9 Hz), 0.04 (s, 6H); ^{13}C NMR (125 MHz, C_6D_6) δ 66.6, 43.7, 42.9, 27.3, 26.1, 24.2, 16.2,–4.1, –4.7; FTIR (neat, cm^{-1}) 1714 (s, C=O); HRMS (FAB) calcd for $C_{13}H_{29}O_2Si$ $(MH)^+$: 245.1938, found: 245.1937.

References and Notes

(1) In each case, an authentic sample of the minor diastereomeric alkylation product was prepared for comparative analysis (chiral capillary GC analysis of the corresponding trimethylsilyl ether or acetate ester). In the case of amides **16**, **21**, **26**, **27**, and **29–31**, diastereomeric mixtures of α-epimers were obtained by epimerization with LDA (5 equiv) or lithium 2,2,6,6-tetramethylpiperidide (5 equiv) in THF for 5 h at 23 °C followed by quenching with aqueous ammonium chloride solution. Amide **28** was epimerized by stirring with lithium chloride (5 equiv) in *N*,*N*-dimethylformamide at 23 °C for 12 h. Each of the remaining alkylation products in Tables 2 and 3 was epimerized by stirring the substrate with trifluoroacetic acid (10 equiv) in THF at reflux for 1 h (effecting $N \rightarrow O$ acyl transfer as well as α-epimerization), followed by neutralization with aqueous sodium bicarbonate solution at 23 °C for 24 h (causing $O \rightarrow N$ acyl transfer).

(2) Dale, J. A.; Dull, D. L.; Mosher, H. S. *J. Org. Chem.* **1969**, *34*, 2543.

(3) Both (*R*)- and (*S*)-Mosher ester derivatives were prepared for comparative analysis.

(4) Alcohol **61** was used for comparative analysis.

(5) Acetate esters of the diastereomeric alcohol pairs **68** and **69**, **70** and **71**, **72** and **73**, **74** and **75**, and **76** and **77** were separated with baseline resolution when assayed by chiral capillary GC analysis.

(6) Compared against an authentic sample of the corresponding derivative of the minor diastereomer. See: Myers, A. G.; McKinstry, L.; *J. Org. Chem.* **1996**, *61*, 2428.