Supporting Information

Tandem Carbon-Carbon Bond-Forming Radical Addition-Cyclization Reaction of Oxime Ether and Hydrazone

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Experimental Section

General. Melting points are uncorrected. ¹H and ¹³C NMR spectra were recorded at 200, 300, or 500 MHz and at 50 or 125 MHz, respectively. IR spectra were recorded using FTIR

apparatus. Mass spectra were obtained by EI or CI methods. Preparative TLC separations were carried out on precoated silica gel plates (E. Merck 60F₂₅₄). Flash column chromatography was performed using E. Merck Kieselgel 60 (230-400 mesh). Medium-pressure column chromatography was performed using Lobar gröÙe B (E. Merck 310-25, Lichroprep Si60). (*S*)-2,2-Dimethyl-1,3-dioxolane-4-carboxaldehyde *O*-(Phenylmethyl)oxime **22** was prepared from D-mannitol.¹

Preparation procedure for **1-4** and characterization data for the related compounds: See,
Scheme 2 in the text.

2-Hydroxyethanal *O*-(Phenylmethyl)oxime (7). To a solution of glycolaldehyde dimer **6** (1.00 g, 8.33 mmol) in MeOH (50 mL) were added *O*-benzylhydroxyamine hydrochloride (2.65 g, 16.7 mmol) and AcONa (1.37 g, 16.7 mmol) under a nitrogen atmosphere at rt. After the reaction mixture was stirred at rt for 1.5 h, the solvent was evaporated at reduced pressure. The residue was diluted with aqueous NaHCO₃ and then the mixture was extracted with CH₂Cl₂. The organic phase was washed with brine, dried over MgSO₄, and concentrated at reduced pressure. Purification of the residue by flash chromatography (hexane/AcOEt 2:1) afforded **7** (2.28 g, 83%) as a colorless oil (*E:Z*=3:2). IR (CHCl₃) 3609, 3431, 3013, 1497 cm⁻¹; ¹H NMR (CDCl₃) δ 7.50 (3/5H, t, *J*=4.7 Hz), 7.36-7.24 (5H, m), 6.83 (2/5H, t, *J*=3.8 Hz), 5.07 (4/5H, s), 5.05 (6/5H, s), 4.36-4.33 (11/5H, br m), 4.15-4.12 (6/5H, br m), 2.85-2.70 (1H, br m); ¹³C NMR (CDCl₃) δ 152.3, 149.0, 137.22, 137.21, 128.2, 128.1, 127.84, 127.81, 76.1, 75.8, 59.6, 57.2; HRMS calcd for C₉H₁₁NO₂ (M⁺): 165.0789, found: 165.0779.

Propenoic Acid 2-[(Phenylmethoxy)imino]ethyl Ester (1). To a solution of **7** (500 mg, 3.0

mmol) and Et₃N (1.3 mL, 9.1 mmol) in CH₂Cl₂ (10 mL) was added acryloyl chloride (0.25 mL, 3.0 mmol) under a nitrogen atmosphere at 0 °C. After being stirred at rt for 10 h, the reaction mixture was diluted with aqueous NaHCO₃ and then extracted with CH₂Cl₂. The organic phase was washed with brine, dried over MgSO₄, and concentrated at reduced pressure. Purification of the residue by flash chromatography (hexane/AcOEt 4:1) afforded **1** (607 mg, 92%) as a colorless oil (*E:Z*=3:2). IR (CHCl₃) 2939, 1718, 1636, 1619, 1497 cm⁻¹; ¹H NMR (CDCl₃) δ 7.55 (3/5H, t, *J*=6.0 Hz), 7.4-7.3 (5H, m), 6.84 (2/5H, t, *J*=4.0 Hz), 6.5-6.4 (1H, m), 6.18 (3/5H, dd, *J*=10.6, 3.6 Hz), 6.09 (2/5H, dd, *J*=10.2, 3.2 Hz), 5.9-5.75 (1H, m), 5.14 (4/5H, s), 5.11 (6/5H, s), 4.97 (4/5H, d, *J*=4.0 Hz), 4.74 (6/5H, d, *J*=4.0 Hz); ¹³C NMR (CDCl₃) δ 165.5, 165.4, 147.3, 144.9, 137.2, 136.9, 131.64, 131.58, 128.3, 128.2, 127.94, 127.89, 127.6, 127.5, 76.4, 76.2, 61.9, 58.9; HRMS calcd for C₁₂H₁₃NO₃ (M⁺): 219.0894, found: 219.0900.

2-[(2-Hydroxyethyl)amino]acetaldehyde *O*-(Phenylmethyl)oxime (9). To 2-aminoethanol (10 mL, 166 mmol) was added chloroacetaldehyde *O*-(phenylmethyl)oxime (10 g, 54.5 mmol) under a nitrogen atmosphere at 0 °C. After being stirred at rt for 12 h, the reaction mixture was added to saturated aqueous NaHCO₃ and CH₂Cl₂. The layers were separated and the aqueous phase was extracted with CH₂Cl₂. The combined organic phase was washed with brine, dried over Na₂SO₄ and concentrated at reduced pressure. Purification of the residue by flash column chromatography (CHCl₃/MeOH 30:1 to CHCl₃/MeOH 15:1) afforded *N*-(2-hydroxyethyl)aminoethanal *O*-phenylmethyloxime (10 g, 88%) as colorless crystals and as a 3:2 mixture of *E/Z*-oxime. mp 58.5-60 °C (AcOEt/hexane). IR (CHCl₃) 3600-3300 cm⁻¹; ¹H NMR (CDCl₃) δ 7.49 (3/5H, t, *J*=5.2 Hz), 7.38-7.25 (5H, m), 6.79 (2/5H, t, *J*=4.4 Hz), 5.10 (4/5H, s), 5.07 (6/5H, s), 3.65-3.57 (2H, m), 3.56 (4/5H, d, *J*=4.4 Hz), 3.38 (6/5H, d,

$J=5.2$ Hz), 2.78-2.70 (2H, m), 1.83(2H, br m); ^{13}C NMR (CDCl_3) δ 151.3, 149.0, 137.6, 137.4, 128.3, 128.2, 128.0, 127.8, 76.1, 75.8, 60.8, 60.7, 51.0, 50.6, 47.7, 44.4; HRMS calcd for $\text{C}_{11}\text{H}_{17}\text{N}_2\text{O}_2$ ($\text{M}+\text{H}^+$): 209.1289, found: 209.1271. Anal. Calcd for $\text{C}_{11}\text{H}_{16}\text{N}_2\text{O}_2$: C, 63.44; H, 7.74; N, 13.45, Found: C, 63.43; H, 7.76; N, 13.50.

General Procedure for Acylation of (9). To a solution of **9** (5.0 g, 24 mmol) in acetone (100 mL) was added a solution of Na_2CO_3 (5.09 g, 48 mmol) in H_2O (20 mL) at 20 °C. After acryloyl chloride, methacryloyl chloride, or crotonyl chloride (36 mmol) was added dropwise at 0 °C, the reaction mixture was stirred at the same temperature for 90 min. After the solvent was evaporated at reduced pressure, the resulting residue was diluted with water and then extracted with CH_2Cl_2 . The organic phase was dried over MgSO_4 and concentrated at reduced pressure. Purification of the residue by flash column chromatography (hexane/AcOEt 1:3) afforded amides **2-4**. The presence of rotamers and *E/Z* isomers of oxime ether and olefin precluded a comprehensive assignment of all protons.

***N*-(2-Hydroxyethyl)-*N*-[2-[(phenylmethoxy)imino]ethyl]-2-propenamide (2).** a colorless oil: IR (CHCl_3) 2936, 1647, 1611 cm^{-1} ; ^1H NMR (CDCl_3) δ 7.50 (1/4H, t, $J=5.1$ Hz), 7.43 (1/4H, t, $J=4.8$ Hz), 7.40-7.25 (5H, m), 6.80-6.25 (5/2H, m), 5.72-5.64 (1H, m), 5.12 (1H, br d, $J=7.2$ Hz), 5.05 (1H, br d, $J=7.5$ Hz), 4.31 (1H, dd, $J=6.0, 4.2$ Hz), 4.12 (1H, br d, $J=4.8$ Hz), 3.80-3.40 (5H, m), 2.23-2.10 (1H, br m); ^{13}C NMR (CDCl_3) δ 168.1, 167.7, 167.4, 167.2, 149.1, 148.4, 147.1, 145.7, 137.5, 137.1, 129.7, 129.3, 128.7, 128.6, 128.5, 128.43, 128.36, 128.33, 128.25, 128.1, 128.0, 127.7, 127.5, 127.3, 126.9, 76.7, 76.3, 76.2, 76.0, 61.2, 61.1, 60.2, 60.0, 51.0, 50.8, 50.5, 50.4, 48.4, 45.6, 45.4, 43.3; HRMS calcd for $\text{C}_{14}\text{H}_{18}\text{N}_2\text{O}_2$ (M^+): 262.1317, found: 262.1318.

***N*-(2-Hydroxyethyl)-*N*-[2-[(phenylmethoxy)imino]ethyl]-2-butenamide (3).** a colorless

oil: IR (CHCl₃) 4217, 3024, 2360, 1606 cm⁻¹; ¹H NMR (CDCl₃) δ 7.53 (5/18H, t, *J* = 4.8 Hz), 7.45 (4/18H, t, *J* = 4.8 Hz), 7.40-7.26 (5H+3/18H, m), 6.99-6.72 (1H+6/18H, m), 6.34 (7/18H, d, *J* = 15.5 Hz), 6.16 (5/18H, d, *J* = 15.5 Hz), 6.06 (6/18H, d, *J* = 15.0 Hz), 5.15 (8/18H, s), 5.12 (6/18H, s), 5.08 (10/18H, s), 5.04 (12/18H, s), 4.31 (14/18H, d, *J* = 3.5 Hz), 4.11 (22/18H, d, *J* = 5.0 Hz), 3.76-3.47 (4H, m), 3.13-2.97 (1H, br m), 1.86 (3H, t, *J* = 6.9 Hz); ¹³C NMR (CDCl₃) δ 168.1, 167.8, 167.3, 167.1, 149.2, 148.6, 147.1, 145.7, 143.6, 143.2, 142.3, 137.3, 137.0, 128.3, 128.2, 128.0, 127.9, 127.7, 121.4, 121.3, 121.1, 120.7, 76.4, 76.0 (2C), 75.7, 61.2, 61.0, 60.1, 59.8, 50.8, 50.6, 50.1, 48.1, 45.3, 45.2, 43.1, 18.0; HRMS calcd for C₁₅H₂₀N₂O₃ (M⁺): 276.1472, found: 276.1486.

***N*-(2-Hydroxyethyl)-2-methyl-*N*-[2-[(phenylmethoxy)imino]ethyl]-2-propenamide (4).** a colorless oil: IR (CHCl₃) 2979, 2955, 2876, 1747 cm⁻¹; ¹H NMR (CDCl₃) δ 7.36-7.26 (5H+3/5H, m), 6.77 (2/5H, t, *J* = 4.2 Hz), 5.20-5.03 (4H, m), 4.32 (4/5H, d, *J* = 4.2 Hz), 4.12 (6/5H, d, *J* = 4.2 Hz), 3.80-3.50 (4H, br m), 3.18-2.89 (1H, br m), 1.94-1.92 (3H, m); ¹³C NMR (CDCl₃) δ 173.6, 148.7, 146.8, 145.8, 139.7, 136.8, 128.0, 127.9, 127.7, 127.6, 115.6, 75.9, 75.6, 59.8, 59.2, 51.0, 48.6, 48.0, 46.0, 43.6, 41.1, 20.1; HRMS calcd for C₁₅H₂₀N₂O₃ (M⁺): 276.1473, found: 276.1498.

Preparation procedure for **5** and characterization data for the related compounds: See,

Scheme 6 in the text.

***N,N*-Bis(2-hydroxyethyl)-2-propenamide (15).** To a solution of **14** (2.0 g, 19 mmol) and Et₃N (9.3 mL, 67 mmol) in CH₂Cl₂ (300 mL) was added acryloyl chloride (4.7 mL, 57 mmol) under a nitrogen atmosphere at 0 °C. After being stirred at rt for 15 h, the reaction

mixture was concentrated at reduced pressure. After the residue was diluted with MeOH (30 mL), saturated Na₂CO₃ (20 mL) was added to the reaction mixture at rt. After the reaction mixture was stirred at rt for 3 h, the reaction mixture was diluted with H₂O and then extracted with CH₂Cl₂. The organic phase was washed with brine, dried over MgSO₄, and concentrated at reduced pressure. Purification of the residue by flash chromatography (CHCl₃/MeOH 10:1) afforded **15** (2.87 g, 95%) as a colorless oil. IR (CHCl₃) 3368, 1643 cm⁻¹; ¹H NMR (CDCl₃) δ 6.66 (1H, dd, *J*=10.5, 17.0 Hz), 6.29 (1H, dd, *J*=2.0, 17.0 Hz), 5.71 (1H, dd, *J*=2.0, 10.5 Hz), 4.90-4.05 (2H, br m), 3.80 (4H, br dt, *J* = 5.0, 8.5 Hz), 3.61-3.54 (4H, m); ¹³C NMR (CDCl₃) δ 168.3, 128.3, 127.9, 60.8, 60.6, 52.1, 50.9; HRMS calcd for C₇H₁₃NO₃ (M⁺): 159.0895, found: 159.0895.

4-Acryloyl-2-morpholinol (16). To a solution of PDC (11.7 g, 31 mmol) in CH₂Cl₂ (100 mL) was added a solution of **15** (987 mg, 6.2 mmol) in CH₂Cl₂ (100 mL) under a nitrogen atmosphere at rt. After being stirred at rt for 4 h, the reaction mixture was filtered through a pad of Celite and then the filtrate was concentrated at reduced pressure. Purification of the residue by flash chromatography (AcOEt) afforded **16** (346 mg, 36%) as a colorless oil. The presence of rotamers precluded a comprehensive assignment of all protons and carbons. IR (CHCl₃) 1645 cm⁻¹; ¹H NMR (CDCl₃) δ 6.54 (1H, dd, *J*=10.5, 17.0 Hz), 6.33 (1H, d, *J*=17.0 Hz), 5.74 (1H, d, *J*=10.5 Hz), 4.99 (1H, br s), 4.10-4.04 (1H, m), 3.93-3.88 (2/3H, m), 3.80-3.73 (1/3H, m), 3.68-3.48 (5H, m), 1.76 (1H, br m); ¹³C NMR (CDCl₃) δ 166.3, 128.9, 128.5, 127.0, 91.4, 90.7, 61.6, 61.3, 50.2, 46.4, 45.4, 41.7; HRMS calcd for C₇H₁₁NO₃ (M⁺): 157.0738, found: 157.0736.

***N*-(2-Hydroxyethyl)-*N*-[2-(diphenylhydrazono)ethyl]-2-propenamide (5).** To a solution of **16** (346 mg, 2.2 mmol) in MeOH (10 mL) were added *N,N*-diphenylhydrazine hydrochloride

(534 mg, 2.4 mmol) and AcONa (361 mg, 4.4 mmol) under a nitrogen atmosphere at rt. After the reaction mixture was stirred at rt for 8 h, the solvent was evaporated at reduced pressure. The residue was diluted with aqueous NaHCO₃ and then extracted with CH₂Cl₂. The organic phase was washed with brine, dried over MgSO₄, and concentrated at reduced pressure. Purification of the residue by flash chromatography (AcOEt) afforded **5** (645 mg, 91%) as a solid. The presence of rotamers precluded a comprehensive assignment of all protons and carbons. IR (CHCl₃) 3010, 1645, 1593 cm⁻¹; ¹H NMR (CDCl₃) δ 7.40-7.04 (10H, m), 6.71 (2/5H, dd, *J*=10.5, 16.5 Hz), 6.60-6.55 (3/5H + 2/5H, m), 6.39-6.32 (1H + 3/5H, m), 5.73-5.68 (1H, m), 4.24 (4/5H, d, *J*=4.5 Hz), 4.22 (6/5H, d, *J*=4.0 Hz), 3.80-3.77 (2H, m), 3.64 (6/5H, t, *J*=5.0 Hz), 3.59 (4/5H, t, *J*=5.5 Hz), 3.15-3.02 (2/5H, br m), 1.76-1.68 (3/5H, br m); ¹³C NMR (CDCl₃) δ 168.5, 166.9, 143.7, 143.3, 134.2, 131.3, 129.9, 129.8, 129.0, 128.3, 127.9, 127.5, 124.8, 124.5, 122.4, 122.2, 61.9, 60.4, 51.2, 50.8, 50.5, 48.1, 30.9; HRMS calcd for C₁₉H₂₁N₃O₂ (M⁺): 323.1632, found: 323.1636.

Preparation procedure for **19** and **20** and characterization data for the related compounds: See, Scheme 8 in the text.

(S)-2,3-Dihydroxypropanal O-(Phenylmethyl)oxime (23). To a solution of **22** (35.3 g, 150 mmol) in MeOH (300 mL) was added pyridinium *p*-toluenesulfonate (37.8 g, 150 mmol) under a nitrogen atmosphere at rt. After being heated at 65 °C for 2 h, the solvent was evaporated at reduced pressure. The residue was diluted with H₂O and then extracted with AcOEt. The organic phase was washed with brine, dried over MgSO₄, and concentrated at reduced pressure. Purification of the residue by flash chromatography (hexane/AcOEt 1:1)

afforded **23** (26.1 g, 89%) as colorless crystals (*E:Z*=3:1): mp 56-58 °C (AcOEt/hexane); $[\alpha]_D^{24} +55.1$ (*c* 1.3, CHCl₃); IR (CHCl₃) 2933, 1604, 1497 cm⁻¹; ¹H NMR (CDCl₃) δ 7.47 (3/4H, d, *J*=4.2 Hz), 7.4-7.3 (5H, m), 6.79 (1/4H, d, *J*=5.1 Hz), 5.10 (2/4H, s), 5.07 (6/4H, s), 4.79 (1/4H, br m), 4.30 (3/4H, br m), 3.8-3.55 (2H, br m), 3.54-3.32 (1H, br m), 2.85-1.99 (1H, br m); ¹³C NMR (CDCl₃) δ 151.5, 149.5, 137.1, 136.9, 128.3, 128.2, 128.0, 127.9, 76.3, 76.0, 69.7, 67.4, 64.2, 63.1; HRMS calcd for C₁₀H₁₃NO₃ (M⁺) 195.0894, found 195.0916; Anal. calcd C 61.53, H 6.71, N 7.18, found C 61.28, H 6.92, N 6.93.

(S)-2-Hydroxy-3-[(1,1-dimethylethyl)diphenylsilyloxy]propanal O-(Phenylmethyl)oxime (24). To a solution of **23** (25 g, 128 mmol) and imidazole (21.8 g, 321 mmol) in DMF (80 mL) was added *t*-butyldiphenylsilyl chloride (39.5 mL, 154 mmol) under a nitrogen atmosphere at rt. After being stirred at the same temperature for 2.5 h, the reaction mixture was diluted with Et₂O. The organic phase was washed with water and brine, dried over MgSO₄, and concentrated at reduced pressure. Purification of the residue by flash chromatography (hexane/AcOEt 20:1 to 10:1) afforded **24** (48.3 g, 87%) as a colorless oil (*E:Z*=3:1): $[\alpha]_D^{24} +7.6$ (*c* 1.6, CHCl₃); IR (CHCl₃) 2932, 1604, 1470 cm⁻¹; ¹H NMR (CDCl₃) δ 7.68-7.61 (4H, m), 7.52 (3/4H, d, *J*=4.8 Hz), 7.47-7.19 (11H, m), 6.83 (1/4H, d, *J*=4.5 Hz), 5.08 (6/4H, s), 5.02 (2/4H, s), 4.86 (1/4H, br m), 4.36 (3/4H, br m), 3.92-3.64 (2H, m), 2.92-2.81 (1H, br m), 1.05 (9H, s); ¹³C NMR (CDCl₃) δ 151.7, 149.5, 137.2, 137.0, 135.4, 134.6, 132.7, 129.8, 128.3, 128.2, 127.9, 127.8, 127.7, 76.3, 76.1, 69.8, 67.4, 65.9, 64.5, 26.7, 19.1; HRMS calcd for C₂₆H₃₁NO₃Si (M⁺) 433.2072, found 433.2100.

Propenoic Acid 1-[[*(S)*-(1,1-Dimethylethyl)diphenylsilyloxy]methyl]-2-[(phenylmethoxy)imino] Ethyl Ester (19). To a solution of **24** (3.0 g, 6.9 mmol) and Et₃N (2.9 mL, 21 mmol) in CH₂Cl₂ (70 mL) was added acryloyl chloride (0.55 mL, 6.9 mmol)

under a nitrogen atmosphere at 0 °C. After being stirred at rt for 6 h, the reaction mixture was diluted with aqueous NaHCO₃ and then extracted with CH₂Cl₂. The organic phase was washed with brine, dried over MgSO₄, and concentrated at reduced pressure. Purification of the residue by flash chromatography (hexane/AcOEt 20:1) afforded **19** (2.2 g, 64%) as a colorless oil (*E:Z*=3:1): $[\alpha]_D^{24} +29.3$ (*c* 1.5, CHCl₃); IR (CHCl₃) 2932, 1721, 1590, 1472 cm⁻¹; ¹H NMR (CDCl₃) δ 7.68-7.60 (4H, m), 7.50 (3/4H, d, *J*=6.3 Hz), 7.45-7.23 (11H, m), 6.79 (1/4H, d, *J*=5.4 Hz), 6.45 (1/4H, dd, *J*=17.4, 1.2 Hz), 6.42 (3/4H, dd, *J*=18.0, 1.2 Hz), 6.20-6.06 (1H, m), 6.05 (1/4H, m), 5.88 (1/4H, dd, *J*=10.2, 1.2 Hz), 5.85 (3/4H, dd, *J*=10.5, 1.2 Hz), 5.59 (3/4H, dd, *J*=10.8, 5.1 Hz), 5.09 (2/4H, s), 5.08 (6/4H, s), 4.02-3.87 (2H, m), 1.05 (9H, s); ¹³C NMR (CDCl₃) δ 165.0, 164.8, 147.9, 146.3, 137.2, 136.9, 133.1, 132.9, 131.3, 129.64, 129.59, 128.2, 127.9, 127.8, 127.7, 127.6, 127.5, 76.3, 76.2, 71.5, 69.7, 63.8, 62.8, 26.5, 19.1; HRMS calcd for C₂₉H₃₃NO₄Si (M⁺) 487.2179, found 487.2172.

(*S*)-3-[(1,1-Dimethylethyl)diphenylsilyloxy]-2-(phenylmethoxy)propanal

***O*-(Phenylmethyl)oxime (25).** To a solution of **24** (500 mg, 1.15 mmol) in cyclohexane/CH₂Cl₂ (15 mL, 2:1, v/v) were added benzyl 2,2,2-trichloroacetimidate (0.28 mL, 1.50 mmol) and CF₃SO₃H (0.01 mL, 0.12 mmol) under a nitrogen atmosphere at rt. After being stirred at rt for 24 h, the reaction mixture was filtered. The filtrate was diluted with aqueous NaHCO₃ and then extracted with CH₂Cl₂. The organic phase was washed with brine, dried over MgSO₄, and concentrated at reduced pressure. Purification of the residue by preparative TLC (hexane/AcOEt 3:1) afforded **25** (452 mg, 75%) as a colorless oil (*E*-isomer): $[\alpha]_D^{24} +24.4$ (*c* 2.0, CHCl₃); IR (CHCl₃) 2972, 1496 cm⁻¹; ¹H NMR (CDCl₃) δ 7.70-7.62 (4H, m), 7.45-7.2 (16H, m), 7.39 (1H, d, *J*=8.1 Hz), 5.12 (2H, s), 4.57, 4.41 (each 1H, d, *J*=12 Hz), 4.09 (1H, td, *J*=8, 6 Hz), 3.82 (2H, ddd, *J*=18, 12, 6 Hz), 1.04 (9H, s). ¹³C

NMR (CDCl₃) δ 149.2, 137.8, 137.4, 135.6, 135.5, 133.2, 133.1, 129.6, 128.3, 128.21, 128.17, 127.8, 127.8, 127.7, 127.6, 127.5, 76.8, 75.9, 70.8, 65.0, 26.7, 19.13; HRMS calcd for C₃₃H₃₇NO₃Si (M⁺): 523.2541, found: 523.2544.

(S)-3-Hydroxy-2-(phenylmethoxy)propanal O-(Phenylmethyl)oxime (26). To a solution of **25** (4.47 g, 8.52 mmol) in THF (200 mL) was added Bu₄NF (1.0 M in THF, 3.70 mL, 12.8 mmol) under a nitrogen atmosphere at rt. After being stirred at rt for 2 h, the reaction mixture was concentrated at reduced pressure. Purification of the residue by flash chromatography (hexane/AcOEt 2:1) afforded **26** (2.04 g, 84%) as a colorless oil (*E*:*Z*=19:1): $[\alpha]_D^{24} +9.14$ (*c* 1.86, CHCl₃). IR (CHCl₃) 3617, 2975, 1496 cm⁻¹; ¹H NMR (CDCl₃) of *E*-isomer δ 7.40 (1H, d, *J*=7.5 Hz), 7.40-7.20 (10H, m), 5.11 (2H, s), 4.59, 4.41 (each 1H, d, *J*=11.7 Hz), 4.06 (1H, td, *J*=6.5, 4.5 Hz), 3.71 (2H, br d, *J*=6 Hz), 2.26-2.16 (1H, br m); ¹³C NMR (CDCl₃) of *E*-isomer δ 148.3, 137.2, 137.1, 128.4, 128.3, 128.2, 127.94, 127.89, 127.85, 76.3, 76.1, 71.0, 63.6; HRMS calcd for C₁₇H₁₉NO₃ (M⁺): 285.1364, found: 285.1380.

Propenoic Acid (2S)-2-Phenylmethoxy-3-[(phenylmethoxy)imino]propyl Ester (20). To a solution of **26** (100 mg, 0.35 mmol) and Et₃N (0.15 mL, 1.1 mmol) in CH₂Cl₂ (2.5 mL) was added acryloyl chloride (0.03 mL, 0.35 mmol) under a nitrogen atmosphere at 0 °C. After being stirred at rt for 5 h, the reaction mixture was diluted with aqueous NaHCO₃ and then extracted with CH₂Cl₂. The organic phase was washed with brine, dried over MgSO₄, and concentrated at reduced pressure. Purification of the residue by preparative TLC (hexane/AcOEt 4:1) afforded **20** (59.8 mg, 50%) as a colorless oil (*E*:*Z*=3:1): $[\alpha]_D^{23} +56.43$ (*c* 2.8, CHCl₃). IR (CHCl₃) 3016, 1726, 1455 cm⁻¹; ¹H NMR (CDCl₃) δ 7.38 (3/4H, d, *J*=5.7 Hz), 7.50-7.23 (10H, m), 6.80 (1/4H, d, *J*=5.7 Hz), 6.39 (1H, br d, *J*=17.4 Hz), 6.11 (1/4H, dd, *J*=16.8, 10.5 Hz), 6.10 (3/4H, dd, *J*=17.4, 10.5 Hz), 5.83 (1H, d, *J*=10.2 Hz), 5.113 (3/4H, s),

5.106 (1/4H, s), 4.94-4.86 (1/4H, m), 4.61 (1/4H, d, $J=12$ Hz), 4.60 (3/4H, d, $J=12$ Hz), 4.49 (1/4H, d, $J=12$ Hz), 4.43 (3/4H, d, $J=12$ Hz), 4.26-4.16 (3/4H, m); ^{13}C NMR (CDCl_3) δ 165.4, 149.7, 147.5, 137.11, 137.07, 131.1, 131.0, 128.2, 128.1, 127.8, 127.73, 127.66, 76.0, 73.5, 71.7, 70.7, 70.4, 64.3, 63.4; HRMS calcd for $\text{C}_{20}\text{H}_{22}\text{NO}_4$ (M^+): 340.1547, found: 340.1541.

NOESY data for **27aA-27dA**: See, Figure 1

FIGURE 1. NOESY studies of **27aA-27dA**.

Reference

- (1) Miyabe, H.; Shibata, R.; Sangawa, M.; Ushiro, C.; Naito, T. *Tetrahedron* **1998**, *54*, 11431.

INITIALS
 Date: 11/11/81
 Time: 11:15
 Page: 1 of 1

RECEIPT
 Name: J. L. Smith
 Address: 1234 Main St
 City: Anytown, IL 60001
 Phone: (312) 555-1234

PLATE PROCESSING
 Plate used: 11
 Plate used: 11
 Plate used: 11
 Plate used: 11

EXPERIMENT
 Name: J. L. Smith
 Date: 11/11/81
 Time: 11:15

1

Varian Technologies Japan Ltd.

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Model A-3	0.005	0.000	0
Model A-4	0.005	0.000	0
Model A-5	0.005	0.000	0
Model A-6	0.005	0.000	0
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Model A-18	0.005	0.000	0
Model A-19	0.005	0.000	0
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Model A-52	0.005	0.000	0
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Model A-84	0.005	0.000	0
Model A-85	0.005	0.000	0
Model A-86	0.005	0.000	0
Model A-8			

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Repairs	0
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Telephone	0
Travel	0
Meals	0
Entertainment	0
Gifts	0
Charitable Contributions	0
Other	0
Total	700

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EXPERIMENT

File Setup Result

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 PULP: 1.0

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 ADDRESS: _____
 CITY: _____
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 CITY: _____
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 PROJECT: _____
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NAME _____
EXP. NO. 4500-3
DATE 2.2.75
ANALYST 9-4
INSTRUMENT 300 MHz

PROB P-1
NUC1 1H
NUC2 13C
PROG 2730-8
MODE 2
NAME 701-1131

PLOT/PROCESSING
PR not used
IS not used
UP used
WDW 2000-2
GB 200
PC 0.2
PH 0.000

EXPERIMENT
Q 1000000
TD 32768
RG 32768
GC 32768
DC 32768

NAME _____
EXP _____
DATE 21.05.1980
ANALYST _____
INSTRUMENT 300

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 Pulse: 12.5 H-1 Transfer: 15

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 Vals: H-1 C-13 MHz
 Max: H-1 C-13 MHz
 Freq: H-1 C-13 MHz
 Power: H-1 C-13 MHz

PULSE/PROCESSED
 H-1 C-13 MHz
 Vals: H-1 C-13 MHz
 Max: H-1 C-13 MHz
 Freq: H-1 C-13 MHz

EXPLAIN I
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 Vals: H-1 C-13 MHz
 Max: H-1 C-13 MHz
 Freq: H-1 C-13 MHz

Name: H-1 C-13 MHz
 Vals: H-1 C-13 MHz
 Max: H-1 C-13 MHz
 Freq: H-1 C-13 MHz

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 Ac. Name: 3,5-DMS
 New File: N-1-4
 Date: 1/6

DECOUPLE
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 Mode: 31H
 Acquisition: 1
 File: N-1-4
 Date: 1/6

FILE/PROCESSING
 File: H-1
 Mode: 31H
 Acquisition: 1
 File: N-1-4
 Date: 1/6

EXPERIMENT
 Name: H-1
 Mode: 31H
 Acquisition: 1
 File: N-1-4
 Date: 1/6

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 Exp. No.: 4800-5-1
 Ac. Name: 3,5-DMS
 New File: N-1-4
 Date: 1/6

NAME: 11a 300.0525 100
 SGL: 16.47 4500.0 10
 Wt: 3.154 0.155
 Purity: 9.4 100% 1.5

INJECTION
 INJECT: 1.0 1000
 INJECT VOL: 10.0 200
 INJECT RATE: 100 0.1 2000

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 Wt: 300.0 0.0000 0.0000
 SUBST: 11a 0.0000 0.0000

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 NAME: 11a

DATE: 09.22.2000
 TIME: 16.47
 PAGE: 20

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Version: 4.1
 Operator: MS
 Date: 2000-11-01
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 Title: 100
 Author: 100
 Subject: 100
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 Comments: 100
 Description: 100
 Keywords: 100
 Comments: 100
 Description: 100

File: 1228
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 Author: 100
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 Keywords: 100
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Experiment: 100
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 Description: 100

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 Keywords: 100
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 Description: 100

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 Date: 2000-11-01
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 Title: 100
 Author: 100
 Subject: 100
 Keywords: 100
 Comments: 100
 Description: 100

1228

Varian Technologies Japan Ltd.

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 Spec: 2000.2
 Date: 2.5.97
 Time: 14.00
 File: 11

Sample: 11
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 Date: 2.5.97
 Time: 14.00
 File: 11

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 Date: 2.5.97
 Time: 14.00
 File: 11

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 Date: 2.5.97
 Time: 14.00
 File: 11

12bB

Varian Technologies Japan Ltd

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 Date: 08/01/94 Time: 14:00 Day: 0
 Acq: 100 MS: 100 MS: 100
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 Date: 08/01/94 Time: 14:00 Day: 0
 Acq: 100 MS: 100 MS: 100
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Name: P1 File: 200.1125 MW: 186
 Date: 08/01/94 Time: 14:00 Day: 0
 Acq: 100 MS: 100 MS: 100
 MS: 100 MS: 100 MS: 100

12aC

Varian Technologies Japan Ltd.

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 Date: 2000.3.10
 Time: 08:52
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Name: 12b
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 Time: 08:52
 File: 12b
 Path: 12b
 Size: 15

Name: 12b
 Date: 2000.3.10
 Time: 08:52
 File: 12b
 Path: 12b
 Size: 15

12bC

Varian Technologies Japan Ltd.

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 QM: 100.0 100.0 0 10
 SOL: 100.0 100.0 1.000 100
 FID: 100.0 100.0 1.0 100

NAME: 12aD 200.2535 MHz
 QM: 100.0 100.0 0 10
 SOL: 100.0 100.0 1.000 100
 FID: 100.0 100.0 1.0 100

NAME: 12aD 200.2535 MHz
 QM: 100.0 100.0 0 10
 SOL: 100.0 100.0 1.000 100
 FID: 100.0 100.0 1.0 100

NAME: 12aD 200.2535 MHz
 QM: 100.0 100.0 0 10
 SOL: 100.0 100.0 1.000 100
 FID: 100.0 100.0 1.0 100

NAME: 12aD 200.2535 MHz
 QM: 100.0 100.0 0 10
 SOL: 100.0 100.0 1.000 100
 FID: 100.0 100.0 1.0 100

NAME: 12aD 200.2535 MHz
 QM: 100.0 100.0 0 10
 SOL: 100.0 100.0 1.000 100
 FID: 100.0 100.0 1.0 100

Watanabe Technology Japan Ltd.

DISCUSS

Address _____
 City _____ State _____ Zip _____
 Day _____
 Night _____
 E-mail _____
 Fax _____
 Name _____
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 City _____ State _____ Zip _____
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 Address _____
 City _____ State _____ Zip _____
 Day _____
 Night _____
 E-mail _____
 Fax _____

RECEIPT

Name MI Date 0
 Address HOA City 2500
 Telephone 2500 State MI
 I am young years old
 Please Mark Next send

PLOT/PROCESSING

first used: by _____
not used until: ca. 600
Date Rec'd _____ Date Rep't _____

表 1 研究對象的年齡與性別

Photo Supply:
 Title Co:
 Title:
 Subject: **INDEX**

NAME _____

DATE _____

PER _____

TIME _____

NO. _____

302

NAME: 11
 SPEC: 3000-3 Hz
 ACQ TIME: 2.892 sec
 DATE: 21.5.96
 TIME: 15.00
 F1: 45

METHOD: 1H
 PULP: zgpg30
 SOLVENT: CDCl3
 TEMP: 25.0
 PRESS: 1000
 FLOW: 0.1
 INJECT: 100
 ACQ: 100
 F1: 45

NAME: 11
 SPEC: 3000-3 Hz
 ACQ TIME: 2.892 sec
 DATE: 21.5.96
 TIME: 15.00
 F1: 45

NAME: 11
 SPEC: 3000-3 Hz
 ACQ TIME: 2.892 sec
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 TIME: 15.00
 F1: 45

NAME: 11
 SPEC: 3000-3 Hz
 ACQ TIME: 2.892 sec
 DATE: 21.5.96
 TIME: 15.00
 F1: 45

12aE

Varian Technologies Japan Ltd.

NAME: 12 P: 202 0835 P: 10
 Spec. Date: 2009.3.4 Date: 2 Y: 9
 Lab. No: 5 Run: 0007 1.000 M
 Purity: 11.5 % Solvent: 30

METHOD: 11 Mode: 100 Date: 0 Y: 0
 Scan: 100 Run: 1223 A
 Dry. Rate: 5 Temp: 200 200 Hz
 Purity: 100 % Total: 100 %

PLOT/PROCESSING
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 L: 0.1 J: 0.1 H: 0.1 S: 0.1
 W: 1000 2000 3000 4000 5000 6000

EXPERIMENT
 Purity: 100 %
 T: 100 %
 W: 100 %
 S: 100 %

Name: 12bE
 Date: 2009.3.4
 Purity: 100 %

12bE

Varian Technologies Japan Ltd.

NAME: H₁ File: 201-0225 No.
 Date: 1500 5 0700 2 2
 Day: 3 000 10 0 200 10
 Time: 1 4 100 10000 10

INSTRUMENT: H₁ Date: 10
 Name: H₁ Name: 1500 50
 Peak # in Name: 5 Name: 200 10
 Acquisition: 1000 From Peak #1:

PLOT-PROCESSING: H₁ File: 201-0225 No.
 15 min: 02 00 07 00 00 00
 WTA: 100.0 10000 1.0 10000

EXPERIMENT: File: 201-0225 No.
 Date: 1500 5 0700 2 2
 Temp: 100 10000 1.0 10000
 Solvent: 10000

謝安

[illegible]

Chemical

mp: 100°C

Peak	Area	%	Chemical
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10-10-79 4:27
18 Dec 13 1979
10-10-79
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RESERVE

NAME: _____

DATE: _____

TIME: _____

INSTRUMENT: _____

ANALYST: _____

REMARKS: _____

RECEIPT

NAME: _____

DATE: _____

TIME: _____

INSTRUMENT: _____

ANALYST: _____

REMARKS: _____

PLOT PROCESSING

DATE: _____

TIME: _____

INSTRUMENT: _____

ANALYST: _____

REMARKS: _____

EXPERIMENT

DATE: _____

TIME: _____

INSTRUMENT: _____

ANALYST: _____

REMARKS: _____

NAME: _____

DATE: _____

TIME: _____

INSTRUMENT: _____

ANALYST: _____

REMARKS: _____

NAME: Name: _____	
EXP. NO.: Exp. No.: _____	DATE: Date: _____
ANALYST: Analyst: _____	INSTRUMENT: Instrument: _____
SAMPLE: Sample: _____	CONC.: Conc.: _____
SOLVENT: Solvent: _____	TEMP.: Temp.: _____
REFERENCE: Reference: _____	REMARKS: Remarks: _____

NAME: 3,4-DHBP
 DATE: 5/10/77
 TIME: 10:00
 INSTR: 15

SAMPLE: 1
 CONC: 100
 SOLV: CDCl3
 TEMP: 25
 PRESS: 100
 FLOW: 1.0
 INJECT: 10

PLOT/PROCESSOR
 FILE: 3,4-DHBP
 TITLE: 3,4-DHBP
 X-Axis: PPM
 Y-Axis: INTENSITY
 SCALE: 100
 OFFSET: 0
 PHASE: 0
 GATE: OFF
 DECOUPL: OFF
 FID: ON
 INTEGR: ON
 PRINT: ON
 STOP: ON

EXPERIMENT
 NAME: 3,4-DHBP
 DATE: 5/10/77
 TIME: 10:00
 INSTR: 15
 SAMPLE: 1
 CONC: 100
 SOLV: CDCl3
 TEMP: 25
 PRESS: 100
 FLOW: 1.0
 INJECT: 10

NAME: 3,4-DHBP
 DATE: 5/10/77
 TIME: 10:00
 INSTR: 15
 SAMPLE: 1
 CONC: 100
 SOLV: CDCl3
 TEMP: 25
 PRESS: 100
 FLOW: 1.0
 INJECT: 10

Name: 41 Title: 200.0535
 Date: 05/05/95 File: 00000000
 Author: 0000 Operator: 0000
 Date: 00/00/00 Time: 00:00

Name: 41 Title: 200.0535
 Date: 05/05/95 File: 00000000
 Author: 0000 Operator: 0000
 Date: 00/00/00 Time: 00:00

Name: 41 Title: 200.0535
 Date: 05/05/95 File: 00000000
 Author: 0000 Operator: 0000
 Date: 00/00/00 Time: 00:00

Name: 41 Title: 200.0535
 Date: 05/05/95 File: 00000000
 Author: 0000 Operator: 0000
 Date: 00/00/00 Time: 00:00

Name: 41 Title: 200.0535
 Date: 05/05/95 File: 00000000
 Author: 0000 Operator: 0000
 Date: 00/00/00 Time: 00:00

Name: 41 Title: 200.0535
 Date: 05/05/95 File: 00000000
 Author: 0000 Operator: 0000
 Date: 00/00/00 Time: 00:00

NOTES

DATE	NO.	DESCRIPTION	AMOUNT	BALANCE
1947	1	TO BALANCE	100.00	100.00
1947	2	BY CHECK	25.00	75.00
1947	3	BY CHECK	15.00	60.00
1947	4	BY CHECK	10.00	50.00
1947	5	BY CHECK	5.00	45.00
1947	6	BY CHECK	5.00	40.00
1947	7	BY CHECK	5.00	35.00
1947	8	BY CHECK	5.00	30.00
1947	9	BY CHECK	5.00	25.00
1947	10	BY CHECK	5.00	20.00
1947	11	BY CHECK	5.00	15.00
1947	12	BY CHECK	5.00	10.00
1947	13	BY CHECK	5.00	5.00
1947	14	BY CHECK	5.00	0.00
1947	15	BY CHECK	5.00	5.00
1947	16	BY CHECK	5.00	0.00
1947	17	BY CHECK	5.00	5.00
1947	18	BY CHECK	5.00	0.00
1947	19	BY CHECK	5.00	5.00
1947	20	BY CHECK	5.00	0.00
1947	21	BY CHECK	5.00	5.00
1947	22	BY CHECK	5.00	0.00
1947	23	BY CHECK	5.00	5.00
1947	24	BY CHECK	5.00	0.00
1947	25	BY CHECK	5.00	5.00
1947	26	BY CHECK	5.00	0.00
1947	27	BY CHECK	5.00	5.00
1947	28	BY CHECK	5.00	0.00
1947	29	BY CHECK	5.00	5.00
1947	30	BY CHECK	5.00	0.00
1947	31	BY CHECK	5.00	5.00
1947	32	BY CHECK	5.00	0.00
1947	33	BY CHECK	5.00	5.00
1947	34	BY CHECK	5.00	0.00
1947	35	BY CHECK	5.00	5.00
1947	36	BY CHECK	5.00	0.00
1947	37	BY CHECK	5.00	5.00
1947	38	BY CHECK	5.00	0.00
1947	39	BY CHECK	5.00	5.00
1947	40	BY CHECK	5.00	0.00
1947	41	BY CHECK	5.00	5.00
1947	42	BY CHECK	5.00	0.00
1947	43	BY CHECK	5.00	5.00
1947	44	BY CHECK	5.00	0.00
1947	45	BY CHECK	5.00	5.00
1947	46	BY CHECK	5.00	0.00
1947	47	BY CHECK	5.00	5.00
1947	48	BY CHECK	5.00	0.00
1947	49	BY CHECK	5.00	5.00
1947	50	BY CHECK	5.00	0.00
1947	51	BY CHECK	5.00	5.00
1947	52	BY CHECK	5.00	0.00
1947	53	BY CHECK	5.00	5.00
1947	54	BY CHECK	5.00	0.00
1947	55	BY CHECK	5.00	5.00
1947	56	BY CHECK	5.00	0.00
1947	57	BY CHECK	5.00	5.00
1947	58	BY CHECK	5.00	0.00
1947	59	BY CHECK	5.00	5.00
1947	60	BY CHECK	5.00	0.00
1947	61	BY CHECK	5.00	5.00
1947	62	BY CHECK	5.00	0.00
1947	63	BY CHECK	5.00	5.00
1947	64	BY CHECK	5.00	0.00
1947	65	BY CHECK	5.00	5.00
1947	66	BY CHECK	5.00	0.00
1947	67	BY CHECK	5.00	5.00
1947	68	BY CHECK	5.00	0.00
1947	69	BY CHECK	5.00	5.00
1947	70	BY CHECK	5.00	0.00
1947	71	BY CHECK	5.00	5.00
1947	72	BY CHECK	5.00	0.00
1947	73	BY CHECK	5.00	5.00
1947	74	BY CHECK	5.00	0.00
1947	75	BY CHECK	5.00	5.00
1947	76	BY CHECK	5.00	0.00

DATE	01	TIME	10:52
FROM	STATION	TO	4000 S. MO
BY	1.732	BY	0
PERSONNEL	1. Class	PERSONNEL	0

1770-1779	1800-1809	1810-1819	1820-1829	1830-1839	1840-1849	1850-1859	1860-1869	1870-1879	1880-1889	1890-1899	1900-1909	1910-1919	1920-1929	1930-1939	1940-1949	1950-1959	1960-1969	1970-1979	1980-1989	1990-1999	2000-2009	2010-2019	2020-2029	2030-2039	2040-2049	2050-2059	2060-2069	2070-2079	2080-2089	2090-2099	2100-2109	2110-2119	2120-2129	2130-2139	2140-2149	2150-2159	2160-2169	2170-2179	2180-2189	2190-2199	2200-2209	2210-2219	2220-2229	2230-2239	2240-2249	2250-2259	2260-2269	2270-2279	2280-2289	2290-2299	2300-2309	2310-2319	2320-2329	2330-2339	2340-2349	2350-2359	2360-2369	2370-2379	2380-2389	2390-2399	2400-2409	2410-2419	2420-2429	2430-2439	2440-2449	2450-2459	2460-2469	2470-2479	2480-2489	2490-2499	2500-2509	2510-2519	2520-2529	2530-2539	2540-2549	2550-2559	2560-2569	2570-2579	2580-2589	2590-2599	2600-2609	2610-2619	2620-2629	2630-2639	2640-2649	2650-2659	2660-2669	2670-2679	2680-2689	2690-2699	2700-2709	2710-2719	2720-2729	2730-2739	2740-2749	2750-2759	2760-2769	2770-2779	2780-2789	2790-2799	2800-2809	2810-2819	2820-2829	2830-2839	2840-2849	2850-2859	2860-2869	2870-2879	2880-2889	2890-2899	2900-2909	2910-2919	2920-2929	2930-2939	2940-2949	2950-2959	2960-2969	2970-2979	2980-2989	2990-2999	3000-3009	3010-3019	3020-3029	3030-3039	3040-3049	3050-3059	3060-3069	3070-3079	3080-3089	3090-3099	3100-3109	3110-3119	3120-3129	3130-3139	3140-3149	3150-3159	3160-3169	3170-3179	3180-3189	3190-3199	3200-3209	3210-3219	3220-3229	3230-3239	3240-3249	3250-3259	3260-3269	3270-3279	3280-3289	3290-3299	3300-3309	3310-3319	3320-3329	3330-3339	3340-3349	3350-3359	3360-3369	3370-3379	3380-3389	3390-3399	3400-3409	3410-3419	3420-3429	3430-3439	3440-3449	3450-3459	3460-3469	3470-3479	3480-3489	3490-3499	3500-3509	3510-3519	3520-3529	3530-3539	3540-3549	3550-3559	3560-3569	3570-3579	3580-3589	3590-3599	3600-3609	3610-3619	3620-3629	3630-3639	3640-3649	3650-3659	3660-3669	3670-3679	3680-3689	3690-3699	3700-3709	3710-3719	3720-3729	3730-3739	3740-3749	3750-3759	3760-3769	3770-3779	3780-3789	3790-3799	3800-3809	3810-3819	3820-3829	3830-3839	3840-3849	3850-3859	3860-3869	3870-3879	3880-3889	3890-3899	3900-3909	3910-3919	3920-3929	3930-3939	3940-3949	3950-3959	3960-3969	3970-3979	3980-3989	3990-3999	4000-4009	4010-4019	4020-4029	4030-4039	4040-4049	4050-4059	4060-4069	4070-4079	4080-4089	4090-4099	4100-4109	4110-4119	4120-4129	4130-4139	4140-4149	4150-4159	4160-4169	4170-4179	4180-4189	4190-4199	4200-4209	4210-4219	4220-4229	4230-4239	4240-4249	4250-4259	4260-4269	4270-4279	4280-4289	4290-4299	4300-4309	4310-4319	4320-4329	4330-4339	4340-4349	4350-4359	4360-4369	4370-4379	4380-4389	4390-4399	4400-4409	4410-4419	4420-4429	4430-4439	4440-4449	4450-4459	4460-4469	4470-4479	4480-4489	4490-4499	4500-4509	4510-
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PLATE PROCESSING

EXPERIMENT

NAME: Benjamin A. ...

ROOM: ...

DATE: ...

6739

1. 100

2. 100

3. 100

4. 100

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100. 100

01504-0394
 1-1

0007 00100 000000 01504

SAMPLE 353.8 VT

date Feb 24 03 BR H2

solvent EDC13 007 E

1110 000 000 000 000

ACQUISITION 409 824 007 E

17 01 0000 00000000 000

25 3.752 000000 00000000

30 000000 000000 00000000

35 000000 000000 00000000

40 000000 000000 00000000

45 000000 000000 00000000

50 000000 000000 00000000

55 000000 000000 00000000

60 000000 000000 00000000

65 000000 000000 00000000

70 000000 000000 00000000

75 000000 000000 00000000

80 000000 000000 00000000

85 000000 000000 00000000

90 000000 000000 00000000

95 000000 000000 00000000

100 000000 000000 00000000

105 000000 000000 00000000

110 000000 000000 00000000

115 000000 000000 00000000

117 000000 000000 00000000

119 000000 000000 00000000

120 000000 000000 00000000

121 000000 000000 00000000

122 000000 000000 00000000

123 000000 000000 00000000

124 000000 000000 00000000

NAME: 01
 DATE: 02/24/03
 TIME: 10:00:00
 INSTRUMENT: 1170
 METHOD: 01
 SAMPLE: 01
 INJECTION: 1
 VOLUME: 10
 FLOW: 1.0
 PRESS: 1.0
 TEMP: 100
 WAVELENGTH: 254

FILE/PROCESSING
 FILE: 01504-0394
 METHOD: 01
 INSTRUMENT: 1170
 SAMPLE: 01
 INJECTION: 1
 VOLUME: 10
 FLOW: 1.0
 PRESS: 1.0
 TEMP: 100
 WAVELENGTH: 254

EXPERIMENT
 NAME: 01504-0394
 METHOD: 01
 INSTRUMENT: 1170
 SAMPLE: 01
 INJECTION: 1
 VOLUME: 10
 FLOW: 1.0
 PRESS: 1.0
 TEMP: 100
 WAVELENGTH: 254

27AC

Varian Technologies Japan Ltd.

KRM-4050
H-1

R-04 F150 ACQUIN 9111

SAMPLE DEF. 5 VT
DATE FEB 24 80 IN
SOLVENT LDC13 DBT
F150 WED CY FOR C
ACQUISITION CTR 208
SLOW 499.321 DBT 38
IN H1 DBT
EL 3.752 PROCESSING
F2 50032 AT(11) 38
F4 3000.0 PDCR 71
F6 4000.0 71 NOT JSTC
F8 02 DBT 7
LEAF 00
P4 4.0 WED
C1 0 WED
C2 1000.0 AT9
F1 6 DBT
C3 6
C4 6
C5 6
C6 6
C7 6
C8 6
C9 6
C10 6
C11 6
C12 6
C13 6
C14 6
C15 6
C16 6
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C99 6
C100 6

NAME: F150 ACQUIN 9111
DATE: FEB 24 80
SOLVENT: LDC13 DBT
F150 WED CY
ACQUISITION: CTR 208
SLOW: 499.321 DBT 38
IN: H1 DBT
EL: 3.752
F2: 50032 AT(11) 38
F4: 3000.0 PDCR 71
F6: 4000.0 71 NOT JSTC
F8: 02 DBT 7
LEAF: 00
P4: 4.0 WED
C1: 0 WED
C2: 1000.0 AT9
F1: 6 DBT
C3: 6
C4: 6
C5: 6
C6: 6
C7: 6
C8: 6
C9: 6
C10: 6
C11: 6
C12: 6
C13: 6
C14: 6
C15: 6
C16: 6
C17: 6
C18: 6
C19: 6
C20: 6
C21: 6
C22: 6
C23: 6
C24: 6
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C97: 6
C98: 6
C99: 6
C100: 6

PLOT/PROCESSING
INSTRUMENT: F150 ACQUIN 9111
DATE: FEB 24 80
SOLVENT: LDC13 DBT
F150 WED CY
ACQUISITION: CTR 208
SLOW: 499.321 DBT 38
IN: H1 DBT
EL: 3.752
F2: 50032 AT(11) 38
F4: 3000.0 PDCR 71
F6: 4000.0 71 NOT JSTC
F8: 02 DBT 7
LEAF: 00
P4: 4.0 WED
C1: 0 WED
C2: 1000.0 AT9
F1: 6 DBT
C3: 6
C4: 6
C5: 6
C6: 6
C7: 6
C8: 6
C9: 6
C10: 6
C11: 6
C12: 6
C13: 6
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C95: 6
C96: 6
C97: 6
C98: 6
C99: 6
C100: 6

EXPERIMENT
NAME: F150 ACQUIN 9111
DATE: FEB 24 80
SOLVENT: LDC13 DBT
F150 WED CY
ACQUISITION: CTR 208
SLOW: 499.321 DBT 38
IN: H1 DBT
EL: 3.752
F2: 50032 AT(11) 38
F4: 3000.0 PDCR 71
F6: 4000.0 71 NOT JSTC
F8: 02 DBT 7
LEAF: 00
P4: 4.0 WED
C1: 0 WED
C2: 1000.0 AT9
F1: 6 DBT
C3: 6
C4: 6
C5: 6
C6: 6
C7: 6
C8: 6
C9: 6
C10: 6
C11: 6
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C81: 6
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C97: 6
C98: 6
C99: 6
C100: 6

NAME: F150 ACQUIN 9111
DATE: FEB 24 80
SOLVENT: LDC13 DBT
F150 WED CY
ACQUISITION: CTR 208
SLOW: 499.321 DBT 38
IN: H1 DBT
EL: 3.752
F2: 50032 AT(11) 38
F4: 3000.0 PDCR 71
F6: 4000.0 71 NOT JSTC
F8: 02 DBT 7
LEAF: 00
P4: 4.0 WED
C1: 0 WED
C2: 1000.0 AT9
F1: 6 DBT
C3: 6
C4: 6
C5: 6
C6: 6
C7: 6
C8: 6
C9: 6
C10: 6
C11: 6
C12: 6
C13: 6
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C93: 6
C94: 6
C95: 6
C96: 6
C97: 6
C98: 6
C99: 6
C100: 6

ANALYSIS
 Method: H1
 Scan Rate: 10000
 Acquisition Time: 12.000
 File Name: 12-1

RECAPIT
 Name: H1
 Mass: 100
 Molecular Weight: 6
 Formula: C₆H₁₂O₂
 CAS: 100
 IUPAC: 1,2-dioxane

PLAT/PROCESSING
 Inlet: 1000
 Outlet: 1000
 Flow: 1.0
 Pressure: 1000
 Temperature: 1000

EXPERIMENT
 Name: 12-1
 Date: 12-1
 Time: 12:00
 Location: 12-1

Name: 12-1
 Date: 12-1
 Time: 12:00
 Location: 12-1

OBSERVE
 Name: _____
 Date: 07/08/80
 Time: 10:00
 Operator: J.S.

DECODE
 Name: _____
 Date: _____
 Time: _____
 Operator: J.S.

PLT/PROCESSING
 Name: _____
 Date: _____
 Time: _____
 Operator: J.S.

EXPERIMENT
 Name: _____
 Date: _____
 Time: _____
 Operator: J.S.

NAME
 Name: _____
 Date: _____
 Time: _____
 Operator: J.S.