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Supplementary Material

General procedure. All dry solvents were freshly distilled under nitrogen from the appropriate drying agent before use. Diethyl ether was distilled over lithium aluminum hydride and tetrahydrofuran was distilled over sodium-benzophenone. Methylene chloride was distilled over calcium hydride. *N,N*-Dimethyl formamide was distilled over P_2O_5. Benzene was washed with sulfuric acid and water, then dried and distilled. ^1H and ^{13}C NMR spectra were recorded on Bruker AC-200 and Bruker AC-300 spectrometer. The chemical shifts in $CDCl_3$ reported in δ (ppm) relative to Me_4Si or $CDCl_3$ as an internal reference. IR spectra were measured on a BOMEM MB-100 Fourier Transform spectrometer. High resolution mass spectra were obtained on a JEOL JMS-DX 303 GC/MS system using electron impact (EI) method. Liquid chromatography (LC) instruments were Hitachi L-6200, L-4250, 655A-52, D-2500, and used column was 5C18 Waters 4.6x150 mm size. Melting point (mp) was determined on a Thomas-Hoover electrothermal capillary apparatus and was uncorrected. Flash chromatography was carried out on Merck silica 60, 230-400 mesh ASTM; eluents are given in parentheses. Analytical thin-layer chromatography (TLC) was performed on E. Merck precoated silica gel 60 F_{254} plates.

O-Benzyl-formohydroximoyl chloride To a solution of O-benzylformaldoxime (1.35 g, 10 mmol) in DMF (25 mL) was added *N*-chlorosuccinimide (1.50 g, 11 mmol). The reaction mixture was heated for 3 h at 40°C, diluted with diethyl ether (100 ml), and washed with aqueous 10% HCl (2x50 mL) and brine (50 mL). The organic layer was dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The crude product was purified by passing through a short column of silica gel (ethyl acetate : hexane = 1 : 50) to afford O-Benzylformohydroximoyl chloride (1.50 g, 88%) : colorless liquid. ^1H NMR ($CDCl_3$, 200 MHz) δ 5.19 (s, 2H), 6.95 (s, 1H), 7.25~7.40 (m, 5H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 77.1, 124.6, 128.2, 128.4, 136.6. IR (NaCl) 3077, 1583, 1455, 1272, 1027 cm^{-1}. (a) Briddle, H. C. *Ann. Chem.*, **1990**, *30*, 11. (b) Schroeter, G. *Ber.*, **1898**, *31*, 2194.

O-Benzyl-acetohydroximoyl chloride : colorless liquid. ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.23 (s, 3H), 5.16 (s, 2H), 7.35~7.45 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 20.9, 77.9, 124.7, 128.2, 128.4, 136.6. IR (NaCl) 1625, 1454, 1164, 1047 cm^{-1}. Johnson, J. E.; Ghafouripour, A.; Haug, Y. K. *J. Org. Chem.*, **1985**, *50*, 993.

S-Phenyl N-(benzyloxy)-thioformimidate (4a) To a solution of O-benzyl formhydroximoyl chloride (1.19 g, 7 mmol) in THF (20 mL) was added thiophenol sodium salt (1.39 g, 10.5 mmol). The reaction mixture was stirred for 3 h at room temperature, diluted with diethyl ether (60 mL) and washed with aqueous $NaHCO_3$ (2x30 mL) and brine (30 mL). The organic layer was dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was chromatographed on a silica gel chromatography (ethyl acetate : hexane = 1 : 20) to give the product **4a** (1.53 g, 90%) : white solid (mp = 38 ~ 39°C). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 5.23 (s, 2H), 7.25~7.55 (m, 11H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 76.6, 128.0, 128.4, 128.7, 129.5, 131.7, 132.5, 137.5, 146.2. IR (NaCl) 3024, 2933, 1564, 1452, 1266, 1210, 1021 cm^{-1}. HRMS (M^+) calcd for $C_{14}H_{13}NOS$ 243.0718 found 243.0713.

S-Phenyl N-(benzyloxy)-thioacetimidate (4b) : colorless liquid. ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.75 (s, 3H), 5.20 (s, 2H), 7.25~7.55 (m, 10H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 19.5, 76.0, 127.7, 127.8, 128.3, 129.1, 129.2, 129.5, 136.2, 137.7, 152.6. IR (NaCl) 3061, 2923, 1586, 1454, 1041, 1025 cm^{-1}. HRMS (M^+) calcd for $C_{15}H_{15}NOS$ 257.0875 found 257.0879.

O-Benzyl-α-(phenylsulfonyl)-formaldoxime (4c) To a solution of **4a** (486 mg, 2 mmol) in CH_2Cl_2 (10mL) was added $NaHCO_3$ (336 mg, 4 mmol) and MCPBA (1.52 g, 4.4 mmol) at 0°C. After being stirred for 1 h, the reaction mixture was heated for 1 h at 40°C, diluted with CH_2Cl_2 (10 mL) and washed with aqueous $NaHCO_3$ (2x10 mL), aqueous Na_2SO_3 (10 mL) and brine (10 mL). The organic layer was dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was chromatographed on a silica gel chromatography (ethyl acetate : hexane = 1 : 7) to give the product **4c** (484 mg, 88%) : white solid (mp = 51 ~ 52°C). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 5.10 (s, 2H), 6.95~7.95 (m, 11H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 78.8, 128.2, 128.4, 128.5,

128.9, 129.2, 134.3, 135.3, 139.3, 143.8. IR (NaCl) 3045, 1563, 1449, 1317, 1150 cm^{-1}. HRMS (M^+) calcd for $C_{14}H_{13}NO_3S$ 275.0616 found 275.0629.

O-Benzyl-α-(phenylsulfonyl)-acetaldoxime (4d) : white solid (mp = 45 ~ 46°C). ^1H NMR ($CDCl_3$, 200 MHz) δ 2.31 (s, 3H), 4.96 (s, 2H), 6.95~7.85 (m, 10H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 16.7, 77.8, 128.1, 128.2, 128.6, 128.8, 133.8, 135.7, 140.0, 151.6. IR (NaCl) 3055, 2973, 2886, 1583, 1449, 1312, 1150 cm^{-1}. HRMS (M^+) calcd for $C_{15}H_{15}NO_3S$ 289.0772 found 289.0753.

O-Benzyl-5-phenoxy-1-pentanoxime (5a) A benzene solution (2 mL, 0.3M in iodide) of 4-phenoxybutyl iodide **1** (166 mg, 0.6 mmol), **4c** (330 mg, 1.2 mmol) and hexamethylditin (238 mg, 0.72 mmol) was degassed for 30 min. To the reaction mixture was added acetone (0.22 mL, 3.0 mmol) and irradiated with a photoreactor (300 nm) for 4 h. The reaction mixture was concentrated under reduced pressure. Ethyl acetate (10 mL), water (0.5 mL) and potassium fluoride (350 mg, 6 mmol) were then added and the mixture was stirred at room temperature for 1 h. Anhydrous potassium carbonate was added and the mixture was filtered through silica gel. The residue was chromatographed on a silica gel chromatography (ethyl acetate : hexane = 1 : 20) to give the product **5a** (160 mg, 94%) : colorless liquid. *E* : *Z* = 1.4 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 200 MHz) *E* : δ 1.70~1.87 (m, 4H), 2.20~2.31 (m, 2H), 3.94 (t, 2H, J = 6.0 Hz), 5.04 (s, 2H), 6,85~6.96 (m, 3H), 7.20~7.42, (m, 7H), 7.45 (t, 1H, J = 6.1 Hz). *Z* : δ 1.70~1.87 (m, 4H), 2.38~2.49 (m, 2H), 3.94 (t, 2H, J = 6.0 Hz), 5.10 (s, 2H), 6.70 (t, 1H, J = 5.5 Hz), 6.85~6.96 (m, 3H), 7.20~7.42, (m, 7H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 22.8, 23.2, 25.4, 28.6, 28.8, 29.2, 67.1, 75.5, 75.7, 114.4, 120.5, 127.7, 127.8, 127.9, 128.2, 128.3, 129.4, 151.0, 151.9, 158.9. IR (NaCl) 2919, 1596, 1496, 1244 cm^{-1}. HRMS (M^+) calcd for $C_{18}H_{21}NO_2$ 283.1572 found 283.1563

5-phenoxy-1-pentanal (3a) To a solution of **5a** (96 mg, 0.4 mmol) in 35% aqueous formaldehyde (1 mL) and THF (3 mL) was added one drop of 10% HCl at room temperature. After being stirred for 2 h, the reaction mixture was diluted with diethyl ether (10 mL), neutralized with aqueous $NaHCO_3$ and washed with brine (10 mL). The organic layer was dried over anhydrous $MgSO_4$, filtered, and concentrated under

reduced pressure. The residue was chromatographed on a silica gel chromatography (ethyl acetate : hexane = 1 : 15) to give the product (64 mg, 90%) : colorless liquid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.75~1.85 (m, 4H), 2.45~2.51 (m, 2H), 3.96 (t, 2H, J = 5.7 Hz), 6.82~6.95 (m, 3H), 7.21~7.32 (m, 2H), 9.78 (t, 1H, J = 1.5 Hz). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 16.8, 28.7, 43.5, 87.2, 114.4, 120.7, 129.4, 158.9, 202.2. IR (NaCl) 2925, 1724, 1560, 1497, 1245 cm^{-1}. Osei-Twum, E. Y.; McCallion, D.; Nazran, A. S.; Panicucci, R.; Risbood, P. A.; Warkentin, J. *J. Org. Chem.*, **1984**, *49*, 336.

O-Benzyl-5-phenoxy-2-hexanoxime (5b) : colorless liquid. *E* : *Z* = 1.9 : 1 (^{1}H NMR ratio). ^{1}H NMR ($CDCl_3$, 200 MHz) *E* : δ 1.65~1.83 (m, 4H), 1.89 (s, 3H), 2.25 (t, 2H, J = 7.2Hz), 3.94 (t, 2H, J = 5.9 Hz), 5.09 (s, 2H), 6.83~6.98, (m, 3H), 7.25~7.40 (m, 7H). *Z* : δ 1.65~1.83 (m, 4H), 1.88 (s, 3H), 2.44 (t, 2H, J = 7.3 Hz), 3.94 (t, 2H, J = 5.9 Hz), 5.07 (s, 2H), 6.83~6.98, (m, 3H), 7.25~7.40 (m, 7H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 14.1, 19.8, 22.1, 22.8, 28.6, 28.9, 29.0, 35.4, 67.2, 75.2, 114.4, 120.5, 127.5, 127.9, 128.2, 129.4, 138.3, 157.8, 158.4, 158.9. IR (NaCl) 2917, 1596, 1496, 1243 cm^{-1}. HRMS (M^+) calcd for $C_{19}H_{23}NO_2$ 297.1729 found 297.1722.

O-Benzyl-3-(terahydro-furan-2-yloxy)-1-propionaldoxime (7) : colorless liquid. *E* : *Z* = 1.1 : 1 (^{1}H NMR ratio). ^{1}H NMR ($CDCl_3$, 200 MHz) *E* : δ 1.45~1.85 (m, 6H), 2.43~2.52 (m, 2H), 3.40~3.60 (m, 2H), 3.72~3.91 (m, 2H), 4.52~4.62 (m, 1H), 5.05 (s, 2H), 7.25~7.40 (m, 5H), 7.52 (t, 1H, J = 6.0 Hz). *Z* : δ 1.45~1.85 (m, 6H), 2.60~2.70 (m, 2H), 3.40~3.60 (m, 2H), 3.72~3.91 (m, 2H), 4.52~4.62 (m, 1H), 5.11 (s, 2H), 6.82 (t, 1H, J = 5.2 Hz), 7.25~7.40 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 19.3, 25.3, 26.7, 30.2, 30.5, 62.0, 63.8, 64.4, 75.5, 75.7, 98.5, 98.6, 127.7, 127.8, 128.1, 128.3, 137.6, 137.9, 149.0, 149.6. IR (NaCl) 2914, 1453, 1360, 1129 cm^{-1}. HRMS (M^+) calcd for $C_{15}H_{21}NO_3$ 263.1521 found 263.1523.

O-Benzyl-1-cyclohexyl-formaldoxime (9a) : colorless liquid. *E* : *Z* = 3.6 : 1 (^{1}H NMR ratio). ^{1}H NMR ($CDCl_3$, 200 MHz) *E* : δ 1.10~1.45 (m, 5H), 1.65~1.85 (m, 5H), 2.10~2.35 (m, 1H), 5.05 (s, 2H), 7.25~7.40 (m, 6H). *Z* : δ 1.10~1.45 (m, 5H), 1.65~1.85 (m, 5H), 2.91~3.05 (m, 1H), 5.09 (s, 2H), 6.50 (d, 1H, J = 7.3 Hz), 7.25~7.40 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 25.2, 25.3, 25.7, 25.8, 29.5, 30.4, 34.5, 38.4, 75.4, 127.6, 127.7, 128.2, 128.3,

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137.6, 155.4, 156.3. IR (NaCl) 2927, 2853, 1450, 1033 cm^{-1}. HRMS (M^+) calcd for $C_{14}H_{19}NO$ 217.1467 found 217.1465

O-Benzyl-1-cyclohexyl-actaldoxime (9b) : colorless liquid. *E* : Z = 1.3 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 200 MHz) *E* : δ 1.10~1.42 (m, 5H), 1.57~1.72 (m, 5H), 1.87 (s, 3H), 2.08~2.18 (m, 1H), 5.06 (s, 2H), 7.25~7.40 (m, 5H). Z : δ 1.10~1.42 (m, 5H), 1.57~1.72 (m, 5H), 1.81 (s, 3H), 3.08~3.18 (m, 1H), 5.05 (s, 2H), 7.25~7.40 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 12.0, 16.5, 25.9, 26.0, 26.1, 29.0, 30.1, 37.1, 44.4, 75.1, 75.2, 127.4, 127.5, 127.6, 127.9, 128.2, 138.4, 138.6, 162.0, 162.5. IR (NaCl) 2928, 2854, 1450, 1367, 1037 cm^{-1}. HRMS (M^+) calcd for $C_{15}H_{21}NO$ 231.1623 found 231.1620

O-Benzyl-2-methyl-1-octanaldoxime (11a) : colorless liquid. *E* : Z = 3.1 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 200 MHz) *E* : δ 0.87 (t, 3H, J = 6.4 Hz), 1.05 (d, 3H, J = 6.9 Hz), 1.20~1.46 (m, 10H), 2.25~2.43 (m, 1H), 5.05 (s, 2H), 7.25~7.41 (m, 6H). Z : δ 0.87 (t, 3H, J = 6.4 Hz), 1.00 (d, 3H, J = 6.8 Hz), 1.20~1.46 (m, 10H), 3.02~3.22 (m, 1H), 5.08 (s, 2H), 6.46 (d, 1H, J = 7.8 Hz), 7.25~7.41 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 14.1, 17.7, 18.2, 22.6, 26.9, 27.2, 29.2, 30.2, 31.7, 34.4, 34.8, 75.5, 75.6, 127.6, 127.7, 127.8, 128.2, 128.3, 137.7, 138.2, 156.0, 157.3. IR (NaCl) 2926, 2858, 1457, 1036 cm^{-1}. HRMS (M^+) calcd for $C_{16}H_{25}NO$ 247.1936 found 247.1941.

O-Benzyl-3-methyl-2-nonanoxime (11b) : colorless liquid. *E* : Z = 1.1 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 200 MHz) *E* : δ 0.87 (t, 3H, J = 6.4 Hz), 1.03 (d, 3H, J = 6.9 Hz), 1.10~1.42 (m, 10H), 1.77 (s, 3H), 2.25~2.42 (m, 1H), 5.07 (s, 2H), 7.25~7.41 (m, 5H). Z : δ 0.87 (t, 3H, J = 6.4 Hz), 0.97 (d, 3H, J = 7.0 Hz), 1.10~1.42 (m, 10), 1.74 (s, 3H), 3.25~3.46 (m, 1H), 5.04 (s, 2H), 7.25~7.41 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 10.7, 14.1, 15.3, 17.2, 18.1, 22.6, 27.3, 27.4, 29.2, 29.3, 31.7, 33.7, 34.0, 39.3, 75.2, 127.5, 127.8, 128.2, 138.3, 138.5, 162.0, 169.2. IR (NaCl) 2926, 2857, 1457, 1367, 1035 cm^{-1}. HRMS (M^+) calcd for $C_{17}H_{27}NO$ 261.2092 found 261.2070.

(E)-O-Benzyl-2,2-dimethyl-1-propanaldoxime (13a) : colorless liquid. ^1H NMR ($CDCl_3$, 200 MHz) δ 1.10 (s, 9H), 5.05 (s, 2H), 7.25~7.41 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 27.6,

33.6, 75.6, 127.8, 128.3, 128.4, 137.6, 158.7. IR (NaCl) 2963, 2869, 1476, 1366, 1027 cm^{-1}. HRMS (M^+) calcd for $C_{12}H_{17}NO$ 191.1310 found 191.1302.

(E)-O-Benzyl-3,3-dimethyl-2-butanoxime (13b) : colorless liquid. ^1H NMR ($CDCl_3$, 200 MHz) δ 1.09 (s, 9H), 1.81 (s, 3H), 5.05 (s, 2H), 7.25~7.37 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 27.6, 37.1, 75.3, 127.5, 128.1, 128.2, 138.5, 163.7. IR (NaCl) 2949, 1462, 1366, 1036 cm^{-1}. HRMS (M^+) calcd for $C_{13}H_{19}NO$ 205.1466 found 205.1468.

Methyl 11-(O-benzyl-formohydroximoyl)-undecanoate (15a) : colorless liquid. *E* : *Z* = 1.4 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 300 MHz) *E* : δ 1.15~1.38 (m, 12H), 1.38~1.50 (m, 2H), 1.51~1.72 (m, 2H), 2.13~2.45 (m, 2H), 2.28 (t, 2H, J = 7.6 Hz), 3.64 (s, 3H), 5.03 (s, 2H), 7.25~7.36 (m, 5H), 7.42 (t, 1H, J = 6.3 Hz). *Z* : δ 1.15~1.38 (m, 12H), 1.38~1.50 (m, 2H), 1.51~1.72 (m, 2H), 2.28 (t, 2H, J = 7.6 Hz), 2.31~2.39 (m, 2H), 3.64 (s, 3H), 5.08 (s, 2H), 6.65 (t, 1H, J = 5.5 Hz), 7.25~7.36 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 24.9, 25.7, 26.1, 26.6, 29.0, 29.2, 29.3, 29.4, 34.0, 51.4, 75.3, 75.4, 75.6, 127.6, 127.7, 127.8, 128.1, 128.3, 137.7, 138.1, 151.6, 152.5, 174.2. IR (NaCl) 2927, 2855, 1740, 1449, 1199 cm^{-1}. HRMS (M^+) calcd for $C_{20}H_{31}NO_3$ 333.2304 found 333.2302.

Methyl 11-(O-benzyl-acetohydroximoyl)-undecanoate (15b) : colorless liquid. *E* : *Z* = 1.7 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 200 MHz) *E* : δ 1.12~1.38 (m, 12H), 1.40~1.51 (m, 2H), 1.52~1.68 (m, 2H), 1.84 (s, 3H), 2.05~2.19 (m, 2H), 2.28 (t, 2H, J = 7.5 Hz), 3.64 (s, 3H), 5.06 (s, 2H), 7.25~7.39 (m, 5H). *Z* : δ 1.12~1.38 (m, 12H), 1.40~1.52 (m, 2H), 1.51~1.68 (m, 2H), 1.82 (s, 3H), 2.28 (t, 2H, J = 7.5 Hz), 2.28~2.42 (m, 2H), 3.64 (s, 3H), 5.03 (s, 2H), 7.25~7.39 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 14.1, 19.9, 24.9, 25.6, 26.4, 29.1, 29.2, 29.3, 29.4, 29.5, 34.0, 35.8, 51.4, 75.1, 127.5, 127.7, 127.8, 128.2, 138.3, 158.5, 159.0, 174.3. IR (NaCl) 2928, 2855, 1740, 1448, 1366, 1186 cm^{-1}. HRMS (M^+) calcd for $C_{21}H_{33}NO_3$ 347.2460 found 347.2468.

O-Benzyl-4-hydroxy-2-butanoxime (18) : colorless liquid. *E* : *Z* = 3.0 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 200 MHz) *E* : δ 1.88 (s, 3H), 2.32 (s, 1H), 2.38 (t, 2H, J = 5.5 Hz), 3.78 (t, 2H, J =5.6 Hz), 5.06 (s, 2H), 7.25~7.42 (m, 5H). *Z* : δ 1.91 (s, 3H), 2.31 (s, 1H), 2.62 (t, 2H, J = 6.4 Hz), 5.06 (s, 2H), 7.25~7.42 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 15.4, 20.6, 33.2, 38.3,

59.1, 59.4, 75.5, 75.6, 127.8, 128.0, 128.3, 137.9, 156.9. IR (NaCl) 3397, 2914, 1453, 1368, 1047 cm^{-1}. HRMS (M^+) calcd for $C_{11}H_{15}NO_2$ 193.1103 found 193.1116.

Nonyl 2-(O-Benzyl-formohydroximoyl)-acetate (20) : colorless liquid. *E* : *Z* = 0.6 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 200 MHz) *E* : δ 0.87 (t, 3H, J = 6.4 Hz), 1.13~1.41 (m, 12H), 1.52~1.67 (m, 2H), 3.24 (d, 2H, J = 6.3 Hz), 4.09 (t, 2H, J = 6.7 Hz), 5.07 (s, 2H), 7.25~7.45 (m, 5H), 7.56 (t, 1H, J = 6.2 Hz). *Z* : δ 0.87 (t, 3H, J = 6.4 Hz), 1.13~1.41 (m, 12H), 1.52~1.67 (m, 2H), 3.40 (d, 2H, J = 5.0 Hz), 4.09 (t, 2H, J = 6.7 Hz), 5.12 (s, 2H), 7.02 (t, 1H, 4.8Hz), 7.25~7.45 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 14.1, 22.6, 25.8, 28.5, 29.2, 29.4, 31.6, 31.8, 35.2, 65.3, 65.4, 75.9, 76.1, 127.9, 128.0, 128.2, 128.4, 137.3, 137.5, 143.9, 144.4, 169.3. IR (NaCl) 2927, 2856, 1739, 1459, 1177 cm^{-1}. HRMS (M^+) calcd for $C_{19}H_{29}NO_3$ 319.2148 found 319.2144.

O-Benzyl-1-(4-tert-butyl-benzyl)-acetaldoxime (22) : colorless liquid. *E* : *Z* = 1.7 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 200 MHz) *E* : δ 1.30 (s, 9H), 1.79 (s, 3H), 3.43 (s, 2H), 5.14 (s, 2H), 7.11 (d, 2H, J= 8.2 Hz), 7.27~7.40 (m, 7H). *Z* : δ 1.30 (s, 9H), 1.80 (s, 3H), 3.69 (s, 2H), 5.13 (s, 2H), 7.11 (d, 2H, J = 8.2 Hz), 7.27~7.40 (m, 7H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 14.0, 20.0, 31.4, 34.4, 35.2, 41.6, 75.4, 125.4, 127.6, 127.9, 128.3, 128.6, 128.7, 133.5, 133.8, 138.2, 138.3, 149.5, 157.4. IR (NaCl) 2953, 1515, 1455, 1366, 1039 cm^{-1}. HRMS (M^+) calcd for $C_{20}H_{25}NO$ 295.1936 found 295.1931.

(E)-O-Benzyl-1-(2,3,4,6-tetra-O-Benzyl-D-glucopyranosyl)-formaldoxime (24a) : colorless liquid. α : β = 2 : 1 (LC ratio). ^1H NMR ($CDCl_3$, 200 MHz) δ 3.60~3.88 (m, 6H), 4.44~4.94 (m, 9H), 5.14 (s, 2H), 7.10~7.38 (m, 25H), 7.76 (d, 1H, J = 5.9 Hz). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 68.4, 71.4, 72.8, 73.3, 73.4, 74.8, 75.4, 76.1, 77.5, 78.9, 82.6, 127.5, 127.7, 127.8, 128.2, 128.3, 137.1, 137.6, 137.8, 138.0, 138.4, 145.3. IR (NaCl) 3030, 2889, 1496, 1453, 1085, 1028 cm^{-1}. HRMS (M^+) calcd for $C_{42}H_{43}NO_6$ 657.3090 found 657.3108.

O-Benzyl-1-(2,3,4,6-tetra-O-Benzyl-D-glucopyranosyl)-acetaldoxime (24b) : colorless liquid. *E* : *Z* = 7 : 1 (^1H NMR ratio). α : β = 1.2 : 1 (LC ratio). ^1H NMR ($CDCl_3$, 200 MHz) *E* : δ 1.81 (s, 3H), 3.46~3.90 (m, 6H), 4.47~4.95 (m, 9H), 5.13 (s, 2H), 7.14~7.56 (m, 25H). *Z* : δ 1.84 (s, 3H), 3.46~3.90 (m, 6H), 4.47~4.95 (m, 9H), 5.07 (s, 2H), 7.14~7.56 (m, 25H). ^{13}C

NMR ($CDCl_3$, 50 MHz) δ 11.0, 68.9, 73.4, 74.4, 75.1, 75.7, 76.0, 78.0, 78.8, 79.0, 80.4, 86.7, 127.5, 127.6, 127.8, 128.0, 128.1, 128.2, 128.3, 128.4, 137.9, 138.1, 138.3, 138.6, 154.5. IR (NaCl) 3031, 2892, 1497, 1453, 1364, 1086 cm^{-1}. HRMS (M^+) calcd for $C_{43}H_{45}NO_6$ 671.3247 found 671.3284.

O-Benzyl-2-[1-(tert-butoxycarbonyl)-pyrrolidin-2-yl)-formaldoxime (26) : colorless liquid. *E* : *Z* = 1.5 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 200 MHz) *E* : δ 1.43 (s, 9H), 1.60~1.69 (m, 1H), 1.71~1.96 (m, 3H), 2.23~2.76 (m, 2H), 3.29~3.37 (m, 2H), 3.90~3.95 (m, 1H), 5.03 (s, 2H), 7.22~7.33 (m, 5H), 7.40 (t, 1H, J = 6.5 Hz). *Z* : δ 1.43 (s, 9H), 1.60~1.69 (m, 1H), 1.71~1.96 (m, 3H), 2.23~2.76 (m, 2H), 3.29~3.37 (m, 2H), 3.90~3.95 (m, 1H), 5.08 (s, 2H), 6.72 (t, 1H, J = 5.6 Hz), 7.22~7.33 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 23.2, 28.5, 30.9, 34.3, 46.4, 54.7, 54.9, 75.6, 75.7, 79.4, 127.8, 127.9, 128.2, 128.3, 137.6, 138.3, 148.9, 154.4. IR (NaCl) 2959, 1693, 1454, 1396, 1366, 1170 cm^{-1}. HRMS (M^+) calcd for $C_{18}H_{26}N_2O_3$ 318.1943 found 318.1942.

O-Benzyl-1-(2-ethoxy-hexahydro-cyclopenta[b]furan-4-yl)-formaldoxime (28a) : colorless liquid. *E* : *Z* = 4 : 1 (^1H NMR ratio). α : β =1 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 200 MHz) δ 1.10~1.21 (m, 3H), 1.30~2.20 (m, 6H), 2.40~3.12 (m, 2H), 3.30~3.50 (m, 1H), 3.60~3.79 (m, 1H), 4.52~4.67 (m, 1H), 5,03~5.16 (m, 3H), 6.56~6.60 (m, 0.2H, Z-isomer), 7.22~7.40 (m, 5.8H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 15.1, 28.9, 29.3, 29.7, 30.0, 31.2, 31.9, 33.9, 34.2, 39.0, 39.2, 39.6, 42.6, 42.7, 45.4, 45.6, 46.1, 46.5, 47.1, 47.6, 62.3, 62.5, 75.6, 75.7, 83.3, 83.4, 86.1, 104.8, 105.0, 105.2, 105.3, 127.5, 127.6, 127.7, 127.8, 128.2, 128.3, 137.5, 137.9, 138.1, 152.8, 153.6, 154.9, 155.9. IR (NaCl) 2930, 1449, 1367, 1053 cm^{-1}. HRMS (M^+) calcd for $C_{17}H_{23}NO_3$ 289.1678 found 289.1673.

O-Benzyl-1-(2-ethoxy-hexahydro-cyclopenta[b]furan-4-yl)-acetaldoxime (28b) : colorless liquid. *E* : *Z* = 1.5 : 1 (^1H NMR ratio). α : β = 1 : 1 (^1H NMR ratio). ^1H NMR ($CDCl_3$, 200 MHz) δ 1.10~1.20 (m, 3H), 1.48~2.17 (m, 9H), 2.35~2.47 (m, 0.5H), 2.70~3.00 (m, 1.5H), 3.30~3.44 (m, 1H), 3.62~3.78 (m, 1H), 4.53~4.68 (m, 1H), 5,04~5.18 (m, 3H), 7.25~7.42 (m, 5H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 13.4, 13.5, 15.2, 28.7, 29.6, 31.6, 33.8, 38.9, 39.6, 44.4, 44.5, 51.8, 52.3, 62.3, 62.5, 75.4, 83.7, 86.3, 105.0, 105.4, 127.5, 128.0, 128.2, 138.4, 158.3, 159.2. IR

(NaCl) 2933, 1449, 1369, 1105, 1054 cm^{-1}. HRMS (M^+) calcd for $C_{18}H_{25}NO_3$ 303.1835 found 303.1816.

3-[(tert-Butyl-dimethyl-silanyl)-methyl]-4-(2-oxo-ethyl)-cyclopentane-1,1-dicarboxylic acid dimethyl esther (30a) : colorless liquid. *cis* : *trans* = 5 : 1 (isolation ratio). ^1H NMR ($CDCl_3$, 200 MHz) δ -0.03 (s, 9H), 0.30~0.50 (m, 2H), 1.20 (t, 6H, J = 7.0 Hz), 1.72~1.83 (m, 1H), 2.00~2.11 (m, 1H), 2.17~2.52 (m, 6H), 4.07~4.19 (m, 4H), 9.75 (s, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ -0.9, 14.0, 16.9, 38.0, 38.2, 38.6, 40.5, 43.9, 58.8, 61.4, 172.6, 172.7, 202.1. IR (NaCl) 2948, 1731, 1452, 1368, 1259, 1178 cm^{-1}. HRMS (M^+) calcd for $C_{17}H_{30}O_5Si$ 342.1862 found 342.1854.

3-[(tert-Butyl-dimethyl-silanyl)-methyl]-4-(2-oxo-propyl)-cyclopentane-1,1-dicarboxylic acid dimethyl esther (30b) : colorless liquid. *cis* : *trans* = 5 : 1 (isolation ratio). ^1H NMR ($CDCl_3$, 200 MHz) δ -0.02 (s, 9H), 0.29~0.52 (m, 2H), 1.21 (t, 6H, J = 7.0 Hz), 1.69~1.81 (m, 1H), 1.98~2.08 (m, 1H), 2.13 (s, 3H), 2.17~2.32 (m, 2H), 2.35~2.49 (m, 4H), 4.06~4.20 (m, 4H). ^{13}C NMR ($CDCl_3$, 50 MHz) δ -0.9, 14.0, 16.8, 30.6, 38.2, 38.8, 39.2, 40.7, 43.1, 58.7, 61.4, 172.7, 173.0, 208.5. IR (NaCl) 2953, 1730, 1449, 1367, 1258, 1176, 1103 cm^{-1}. HRMS (M^+) calcd for $C_{18}H_{32}O_5Si$ 356.2019 found 356.2013.

Registry No. 3a, 87841-90-5; O-benzyl-formhydroximoyl chloride, 151451-59-1; O-benzyl-acetohydroximoyl chloride, 95017-93-9; formylcyclohexane (hydrolysis of **9a**), 2043-61-0; acetylcyclohexane (hydrolysis of **9b**), 823-76-7; 2-methyloctanal (hydrolysis of **11a**), 7786-29-0; 3-methyl-2-nonanone (hydrolysis of **11b**), 816-78-4; 2,2-dimethyl-propanal (hydrolysis of **13a**), 630-19-3; 3,3-dimethyl-2-butanone (hydrolysis of **13b**), 75-97-8; 4-hydroxy-2-butanone (hydrolysis of **18**), 590-90-9.