Electronic Supplementary Information (ESI)

Direct Conversion of Esters, Lactones, and Carboxylic Acids to Oxazolines Catalyzed by Tetranuclear Zinc Cluster

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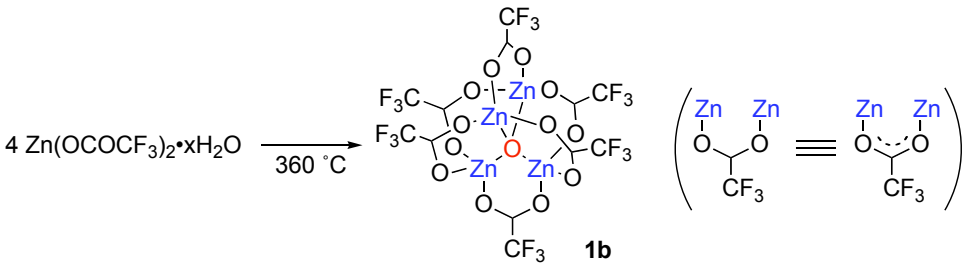
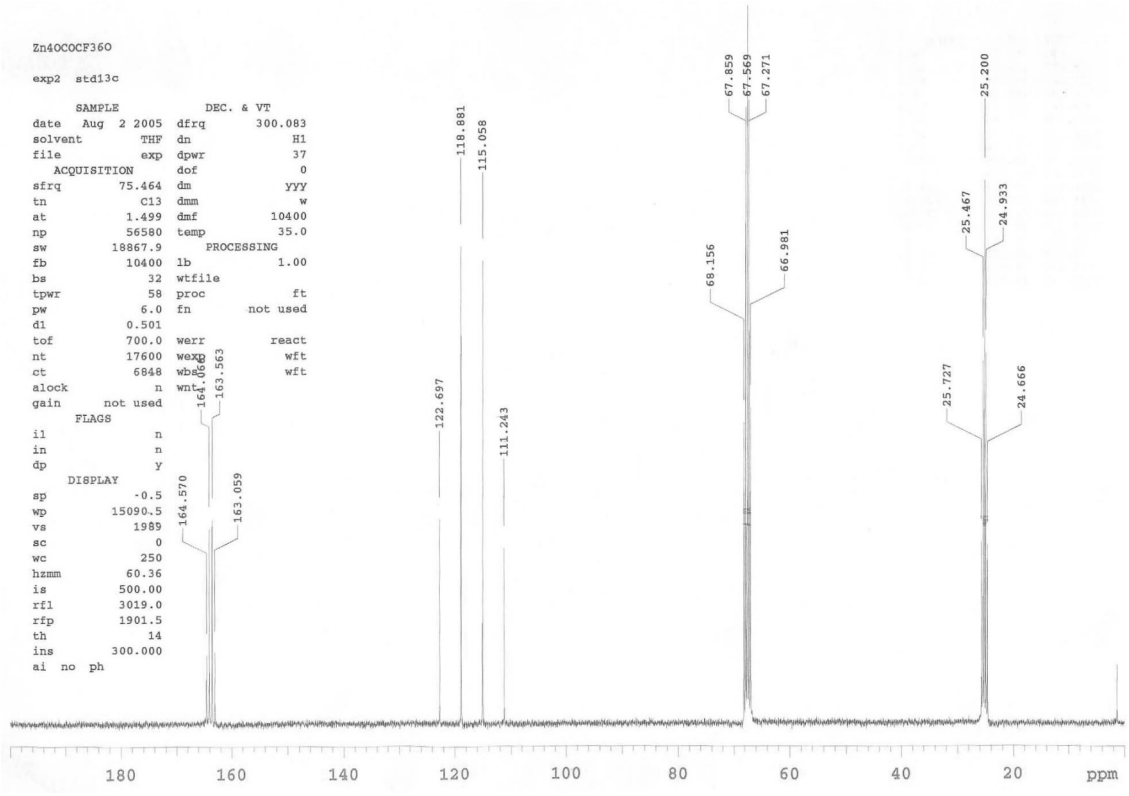
Preparation of Tetranuclear Zinc Cluster 1b:



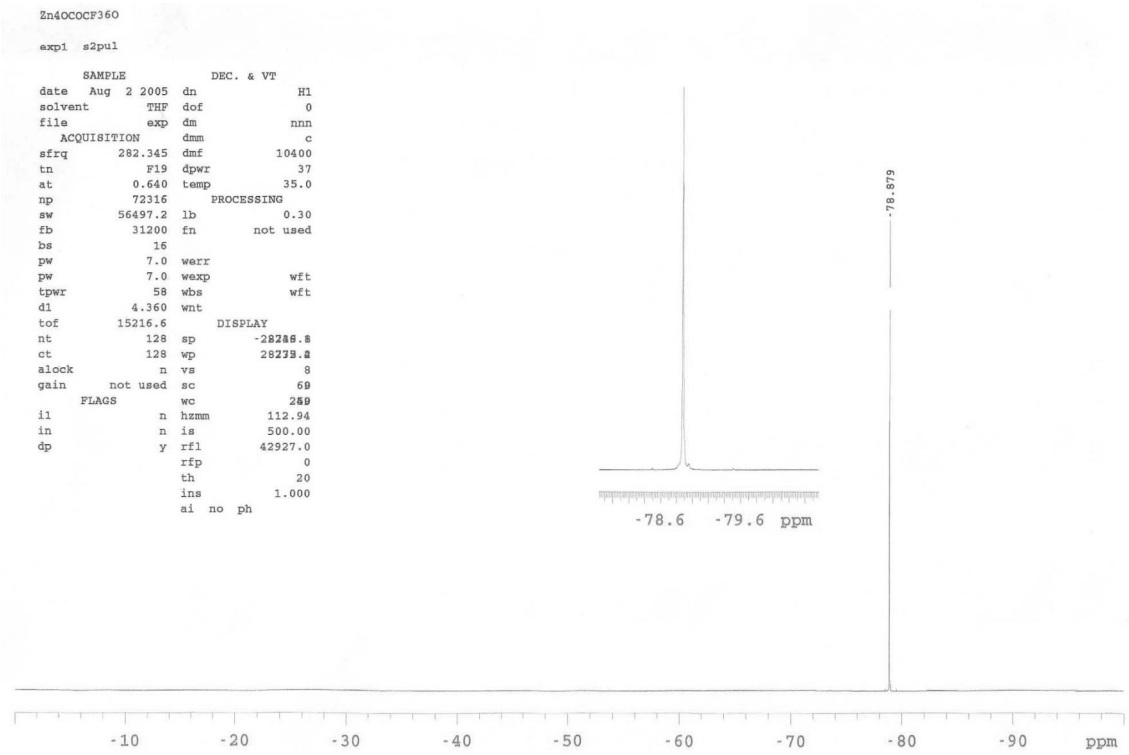
Under an inert atmosphere of argon, zinc trifluoroacetate hydrate (845 mg, 2.73 mmol as a monohydrate) was heated at 120 ˚C for 4 h under reduced pressure (≤0.02 mmHg) to remove water. The resulting dry powder was then heated at 360 ˚C on a sand bath, and μ-oxo-tetranuclear zinc cluster **1b** was sublimed from the reaction mixture. The zinc cluster was collected and stored under an inert atmosphere of argon (545 mg, 84%).

White solid; IR (nujol NaCl, ν) 2923, 1701, 1202, 856, 796, 730 cm^{-1}; ^{13}C NMR (75 MHz, THF-d_8, 35 ˚C) δ 163.81 (q, J_{C-F} = 38 Hz, CF_3COO) 116.97 (q, J_{C-F} = 288 Hz, CF_3COO); ^{19}F NMR (282 MHz, THF-d_8, 35 ˚C) δ –78.88 (s); Anal. calcd for $C_{12}F_{18}O_{13}Zn_4$: C 15.08%; found C 15.52%.

^{13}C NMR of μ-oxo-tetranuclear zinc cluster 1b in THF-d_8:

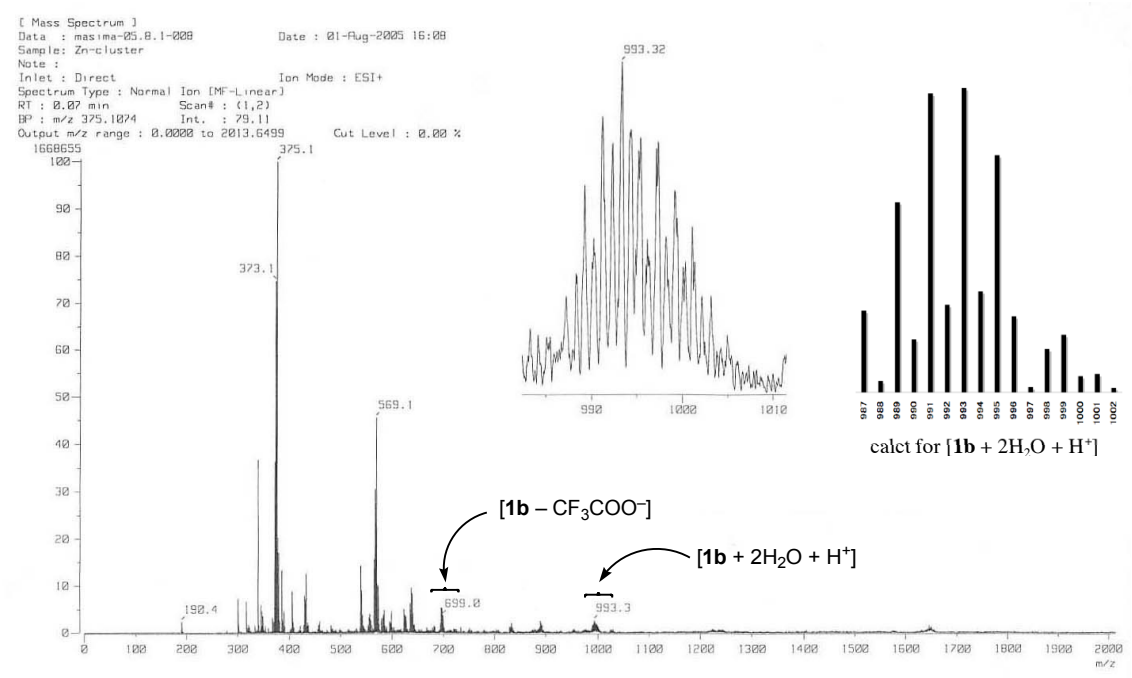


^{19}F NMR of μ-oxo-tetranuclear zinc cluster 1b in THF-d_8:

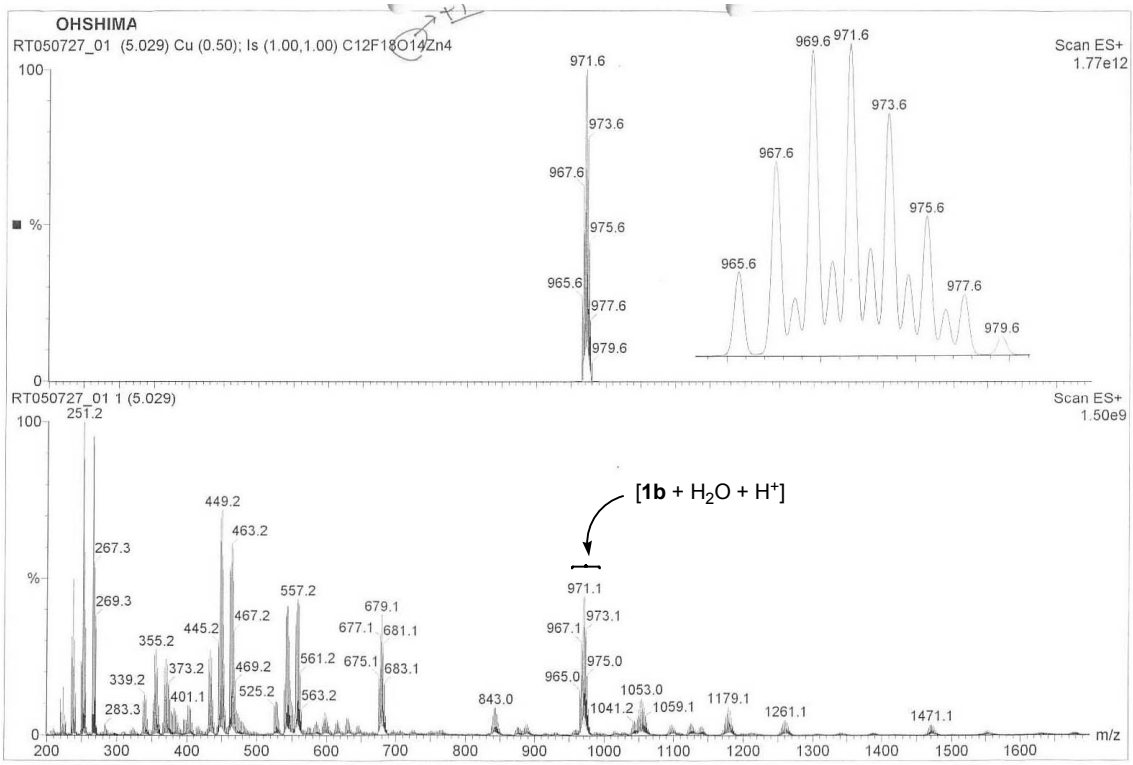


ESI-MS analysis of μ-oxo-tetranuclear zinc cluster 1b in MeOH:

ESI-MS analysis on JEOL JMS-700

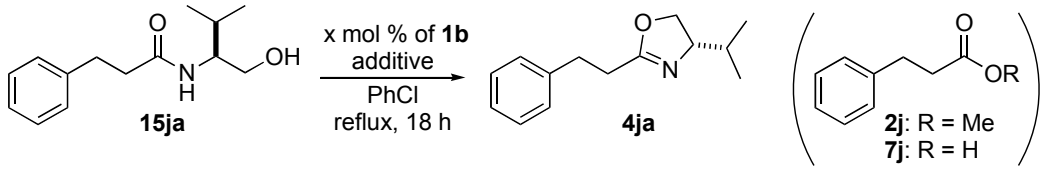


ESI-MS analysis on Waters micromass ZQ with Waters 2695 Separation Module



Control Reaction Using the Hydroxyamide Intermediate 15ja:

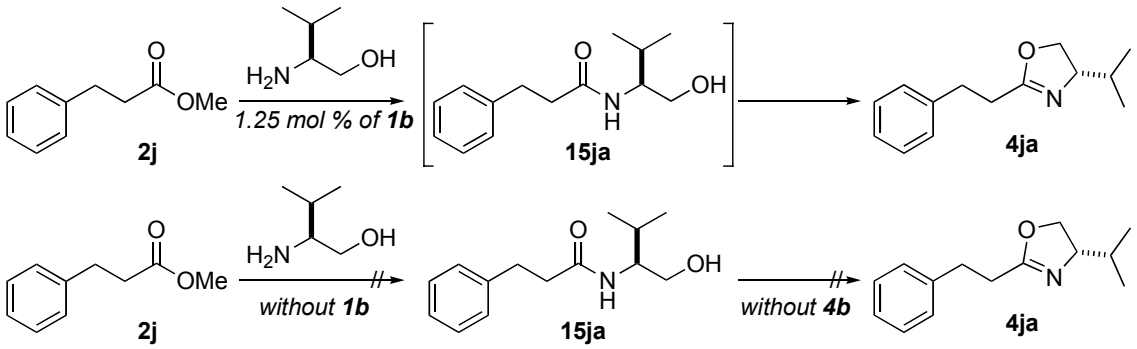
Table S-1



Entry	**1b** (x mol %)	Additive	Yield of **4ja** (%)[a]	Comments
1	none	none	3	
2	1.25	none	97	
3	1.25	MeOH (10 equiv to **15ja**) (400 equiv to **1b**)	96	
4	1.25	H_2O (10 equiv to **15ja**) (400 equiv to **1b**)	85	
5	1.25	MeOH (100 equiv to **15ja**) (4000 equiv to **1b**)	36	**2j** was obtained in 16% yield.[a]
6	1.25	H_2O (100 equiv to **15ja**) (4000 equiv to **1b**)	20	**7j** was obtained in 4% yield.[a]

[a] Determined by GC analysis of the crude sample.

(i) Based on the results of Entry 1 in Table 1 and Entry 1 in Table S-1, Zn cluster **1b** is essential for not only the condensation reaction but also cyclodehydration reaction.



(ii) Based on the results of Entries 2 to 4 in Table S-1, Zn cluster **1b** showed comparable catalyst activity even in the presence of 400 equivalents of water or methanol.

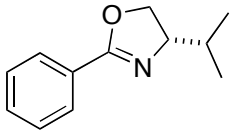
(iii) Based on the results of entries 5 and 6 in Table S-1, in the presence of excess water or methanol (100 equiv to **15ja** = 4000 equiv to **1b**), Zn cluster **1b** promoted hydrolysis or amide esterification.

Experimental Procedures and Characterization of the Products

General: Nuclear magnetic resonance (^{1}H NMR, ^{13}C NMR, and ^{19}F NMR) spectra were measured on a Varian MERCURY300-C/H spectrometer operating at 300 MHz (^{1}H NMR), 75 MHz (^{13}C NMR), and 282 MHz (^{19}F NMR), in 5 mm NMR tubes. All ^{1}H NMR chemical shifts were reported in ppm relative to internal references of TMS at δ 0.00. All ^{13}C NMR chemical shifts were reported in ppm relative to carbon resonance in chloroform-d_1 at δ 77.00, THF-d_8 at δ 25.20. The ^{19}F NMR chemical shifts were reported in ppm relative to external references of α,α,α-trifluorotoluene at δ –63.9. Low and high resolution mass spectra (EI, FAB) were measured on JEOL JMS-700. ESI mass spectra were measured on JEOL JMS-700 coupled with an electrospray ionization probe (MS-ESIP15) and Waters micromass ZQ with Waters 2695 Separation Module. IR spectra were recorded on Jasco FT/IR-230 spectrometer and Jasco FT/IR-410 spectrometer. Elemental analyses were conducted by Perkin-Elmer 2400II. Melting points of air- and moisture-sensitive compounds were measured using Yanaco micro melting point apparatus. All manipulations involving air- and moisture-sensitive compounds were carried out using the standard Schlenk techniques under an argon atmosphere. Chlorobenzene was distilled from calcium hydride. (*S*)-Valinol, (*R*)-phenylglycinol, and (*S*)-phenylalaninol were prepared according to the literature procedures.[SI-1] α,α-Disubstituted aminoalcohol **3g** was prepated from (*S*)-phenylalanine methyl ester hydrochloride with methyl magunesium iodide.[SI-2] Ester substrates were synthesized from the corresponding carboxylic acids by standard esterification reaction with catalytic amounts of an acid ($SOCl_2$, H_2SO_4, or *p*-toluenesurfonic acid).

General procedure for the catalytic tandem condensation–cyclodehydration reaction of esters, lactones, and carboxylic acids. Catalyst **1b** (0.0375 mmol = 0.15 mmol on metal), substrate (3.0 mmol), and aminoalcohol (3.6 mmol) in chlorobenzene (5 mL) were refluxed for periodic time under an argon atmosphere. The resulting mixture was concentrated *in vacuo* and purified by silica gel column chromatography.

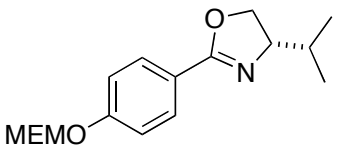
(*S*)-4-Isopropyl-2-phenyloxazoline (4aa)[SI-3]



Purified by flush column chromatography (silica gel, Hexane/EtOAc = 8/1); colorless oil; IR (neat NaCl, ν) 1652, 1450, 1354, 1081, 1065, 1026, 968, 694 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.91 (d, J = 6.9 Hz, 3H, CH(CH_3)$_2$), 1.01 (d, J = 6.9 Hz, 3H, CH(CH_3)$_2$), 1.8-1.9 (m, 1H, CH(CH$_3$)$_2$), 4.08 (m, 2H, CH_2O), 4.35 (m, 1H, CHCH$_2$), 7.39 (m, 3H, Ar), 7.73 (dd, J = 8.4, 1.8, 2H, Ar); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 18.06, 18.83, 32.77, 69.93, 72.52, 127.97, 127.98, 130.80, 162.94; MS

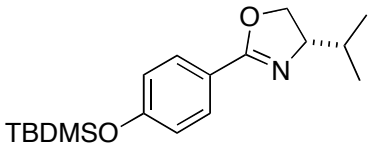
(EI) *m/z* (relative intensity) 190 ([M^++H], 17), 189 (100), 174 (12), 162 (28); HRMS (EI) *m/z* calcd. for $C_{12}H_{15}NO$ 189.1154, found 189.1147; $[\alpha]^{25}_{589}$ –61.1 (*c* 0.39, CH_2Cl_2).

(*S*)-4-Isopropyl-2-[4-(methoxyethoxymethoxy)phenyl]oxazoline (4ba)



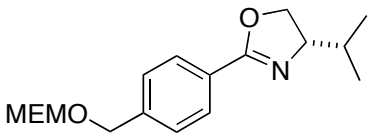
Purified by flush column chromatography (silica gel, Hexane/EtOAc = 8 to 4/1); colorless oil; IR (neat NaCl, *ν*) 1667, 1604, 1495, 1454, 1078, 753, 699 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 ˚C) δ 0.92 (d, *J* = 6.9 Hz, 3H, $CH(CH_3)_2$), 1.02 (d, *J* = 6.9 Hz, 3H, $CH(CH_3)_2$), 1.8-1.9 (m, 1H, $CH(CH_3)_2$), 3.35 (s, 1H, $CH_3O(CH_2)_2OCH_2O$), 3.5 (m, 2H, $CH_3O(CH_2)_2OCH_2O$), 3.8 (m, 2H, $CH_3O(CH_2)_2OCH_2O$), 4.0-4.1 (m, 2H, $NCHCH_2$), 4.3-4.4 (m, 1H, $NCHCH_2$), 5.28 (s, 2H, $CH_3O(CH_2)_2OCH_2O$), 7.05 (d, *J* = 9.0 Hz, 2H, *Ar*), 7.88 (d, *J* = 9.0 Hz, 2H, *Ar*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 ˚C) δ 18.41, 19.26, 33.17, 59.21, 68.09, 70.24, 71.83, 72.86, 93.47, 115.96, 121.82, 130.02, 159.70, 163.05; MS (EI) *m/z* (relative intensity) 293 ([M^+], 24), 276 (40), 251 (52), 250 (100), 220 (17), 218 (17); HRMS (EI) *m/z* calcd. for $C_{16}H_{23}NO_4$ 293.1627, found 293.1554; $[\alpha]^{25}_{589}$ –44.7 (*c* 1.1, CH_2Cl_2).

(*S*)-4-Isopropyl-2-[4-(*tert*-butyldimethylsiloxy)phenyl]oxazoline (4ca)



Purified by flush column chromatography (silica gel, Hexane/EtOAc = 20 to 4/1); colorless oil; IR (neat NaCl, *ν*) 2957, 2859, 1651, 1606, 1512, 1471, 1354, 1268, 1164, 1074, 913, 839, 806, 782, 673 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 ˚C) δ 0.20 (s, 6H, ^{t}BuMe_2Si), 1.03 (d, *J* = 6.6 Hz, 3H, $CH(CH_3)_2$), 1.05 (s, 9H, ^{t}*Bu*Me_2Si), 1.30 (d, *J* = 6.6 Hz, 3H, $CH(CH_3)_2$), 1.9-2.0 (m, 1H, $CH(CH_3)_2$), 4.1-4.2 (m, 2H, $NCHCH_2$), 4.4-4.5 (m, 1H, $NCHCH_2$), 4.87 (s, $TBDMSCH_2$), 7.45 (d, *J* = 8.1 Hz, 2H, *Ar*), 8.02 (d, *J* = 8.1 Hz, 2H, *Ar*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 ˚C) δ -5.14, 18.17, 18.47, 19.01, 25.99, 32.92, 64.69, 70.02, 72.64, 125.64, 126.54, 128.09, 144.64, 163.10; MS (EI) *m/z* (relative intensity) 319 ([M^+], 27), 304 (15), 278 (29), 277 (97), 276 (100), 262 (48), 248 (48), 221 (30), 217 (13), 212 (11); HRMS (EI) *m/z* calcd. for $C_{18}H_{29}NO_2Si$ 319.1968, found 319.1953; $[\alpha]^{25}_{589}$ –39.1 (*c* 1.0, CH_2Cl_2).

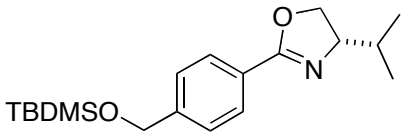
(*S*)-4-Isopropyl-2-[4-(methoxyethoxymethoxymethyl)phenyl]oxazoline (4da)



Purified by flush column chromatography (silica gel, Hexane/EtOAc = 6/1); colorless oil; IR (KBr disk, *ν*) 2963, 2890, 1721, 1668, 1651, 1456, 1363, 1049 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 ˚C) δ 0.91 (d, *J* = 6.9 Hz, 3H, $CH(CH_3)_2$), 1.02 (d, *J* = 7.2 Hz, 3H, $CH(CH_3)_2$), 1.8-1.9 (m, 1H, $CH(CH_3)_2$), 3.36 (s, 3H, $CH_3O(CH_2)_2OCH_2O$), 3.5-3.7 (m, 4H, $CH_3O(CH_2)_2OCH_2O$), 4.0-4.1 (m, 2H, $NCHCH_2O$), 4.3-4.4 (m, 1H, $NCHCH_2O$), 4.63 (s, 2H,

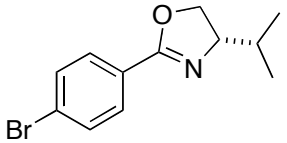
$CH_3O(CH_2)_2OCH_2O$), 4.78, (s, 2H, MEMOCH_2Ar), 7.37 (d, J = 8.1 Hz, 2H, *Ar*), 7.93 (d, J = 8.1 Hz, 2H, *Ar*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 17.93, 18.70, 32.65, 58.65, 66.77, 68.59, 69.80, 71.53, 72.37, 94.65, 126.92, 127.02, 127.96, 140.95, 162.69; MS (FAB) *m*/*z* (relative intensity) 309 ([M^++H], 28), 308 (100), 225 (15), 210 (15); HRMS (FAB) *m*/*z* calcd. for $C_{17}H_{27}NO_4$ 309.1940, found 309.1862; $[\alpha]^{25}_{589}$ –23.4 (*c* 1.1, CH_2Cl_2).

(*S*)-4-Isopropyl-2-[4-(*tert*-butyldimethylsiloxymethyl)phenyl]oxazoline (4ea)



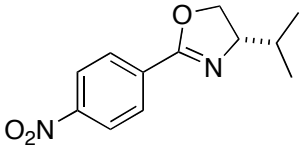
Purified by flush column chromatography (silica gel, Hexane/EtOAc = 20 to 10/1); colorless oil; IR (neat NaCl, *ν*) 2956, 2857, 1651, 1470, 1360, 1256, 1116, 1074, 838, 777, 726, 671 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.20 (s, 6H, ^{t}BuMe_2Si), 1.03 (d, J = 6.6 Hz, 3H, CH(CH_3)$_2$), 1.05 (s, 9H, ^{t}*Bu*Me$_2$Si), 1.30 (d, J = 6.6 Hz, 3H, CH(CH_3)$_2$), 1.9-2.0 (m, 1H, C*H*(CH$_3$)$_2$), 4.1-4.2 (m, 2H, NCHCH_2), 4.4-4.5 (m, 1H, NC*H*CH$_2$), 4.87 (s, TBDMSCH_2), 7.45 (d, J = 8.1 Hz, 2H, *Ar*), 8.02 (d, J = 8.1 Hz, 2H, *Ar*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ –5.14, 18.17, 18.47, 19.01, 25.99, 32.92, 64.69, 70.02, 72.64, 125.64, 126.54, 128.09, 144.64, 163.10; MS (EI) *m*/*z* (relative intensity) 333 ([M^+], 26), 278 (100), 277 (100), 276 (100), 274 (17); HRMS (EI) *m*/*z* calcd. for $C_{19}H_{31}NO_2Si$ 333.2124, found 333.2105; $[\alpha]^{25}_{589}$ –37.7 (*c* 1.1, CH_2Cl_2).

(*S*)-2-(4-Bromophenyl)-4-isopropyloxazoline (4fa)[SI-4]



Purified by flush column chromatography (silica gel, Hexane/EtOAc = 8/1); white solid; mp 69-70 °C; IR (film NaCl, *ν*) 2956, 1649, 1396, 1351, 1076, 1008, 964, 831, 665 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.92 (d, J = 6.6 Hz, 3H, CH(CH_3)$_2$), 1.02 (d, J = 6.6 Hz, 3H, CH(CH_3)$_2$), 1.8–1.9 (m, 1H, C*H*(CH$_3$)$_2$), 4.1–4.2 (m, 2H, NCHCH_2), 4.3–4.4 (m, 1H, NC*H*CH$_2$), 7.52 (d, J = 8.4 Hz, 2H, *Ar*), 7.81 (d, J = 8.4 Hz, 2H, *Ar*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 18.23, 18.98, 32.91, 70.33, 72.80, 125.67, 126.89, 129.69, 131.40, 162.36; MS (EI) *m*/*z* (relative intensity) 269 ([M(^{81}Br)$^+$], 5), 267 ([M(^{79}Br)$^+$], 5), 226 (78), 224 (79); HRMS (EI) *m*/*z* calcd. for $C_{12}H_{14}{}^{79}BrNO$ 267.0259 found 267.0234, calcd. for $C_{12}H_{14}{}^{81}BrNO$ 269.0239 found 269.0236; $[\alpha]^{25}_{589}$ –50.6 (*c* 1.0, CH_2Cl_2).

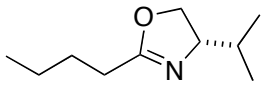
(*S*)-4-Isopropyl-2-(4-nitrophenyl)oxazoline (4ga)[SI-3]



Purified by flush column chromatography (silica gel, Hexane/EtOAc = 8/1); white–yellow solid; mp 54-55 °C; IR (film NaCl, *ν*/ cm^{-1}) 2968, 1647, 1597, 1523, 1346, 1079, 966, 865, 850, 699; ^{1}H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.95 (d, J = 6.9 Hz, 3H, CH(CH_3)$_2$), 1.05 (d, J = 6.9

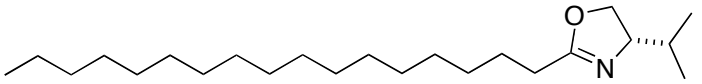
Hz, 3H, CH(CH_3)$_2$), 1.8-1.9 (m, 1H, CH(CH$_3$)$_2$), 4.1-4.2 (m, 2H, NCHCH_2), 4.4-4.5 (m, 1H, NCHCH$_2$), 8.11 (d, J = 8.7 Hz, 2H, *Ar*), 8.20 (d, J = 8.7 Hz, 2H, *Ar*); ^{13}C NMR (75 MHz, CDCl$_3$, 35 ˚C) δ 18.27, 18.86, 32.88, 70.73, 73.03, 123.25, 129.06, 133.65, 149.26, 161.29; MS (EI) *m*/*z* (relative intensity) 234 ([M$^+$], 5), 191 (100), 163 (19), 117 (18); HRMS (EI) *m*/*z* calcd. for $C_{12}H_{14}N_2O_3$ 234.1004 found 234.1032; [α] $^{25}_{589}$ –60.5 (*c* 1.0, CH_2Cl_2).

(*S*)-2-Butyl-4-isopropyloxazoline (4ha).



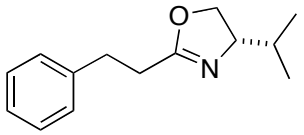
Purified by flush column chromatography (silica gel, Hexane/EtOAc = 8/1); colorless oil; IR (neat NaCl, *ν*) 2959, 1672, 1468, 1384, 1365, 1236, 1173, 984 cm^{-1}; ^{1}H NMR (300 MHz, CDCl$_3$, 35 ˚C) δ 0.87 (d, J = 6.9 Hz, 3H, CH(CH_3)$_2$), 0.92 (t, J = 7.2 Hz, 3H, CH_3CH$_2$), 0.95 (d, J = 6.9 Hz, 3H, CH(CH_3)$_2$), 1.37 (m, 2H, CH$_3$CH$_2$CH_2), 1.59 (m, 2H, CH$_3$CH_2), 1.64 (sept like, 1H, CH(CH$_3$)$_2$), 2.27 (t, J = 7.2, 2H, CH_2C), 3.91 (m, 2H, CH_2O), 4.18 (m, 1H, CHCH$_2$); ^{13}C NMR (75 MHz, CDCl$_3$, 35 ˚C) δ 13.62, 17.95, 18.64, 22.29, 28.26, 32.48, 69.54, 71.93, 167.14; MS (EI) *m*/*z* (relative intensity) 169 ([M$^+$], 13), 168 (66), 126 (100); HRMS (EI) *m*/*z* calcd. for $C_{10}H_{19}NO$ 169.1467, found 169.1440; [α] $^{25}_{589}$ –64.9 (*c* 1.0, CH_2Cl_2).

(*S*)-2-Heptadecanyl-4-isopropyloxazoline (4ia)



Purified by flush column chromatography (silica gel, Hexane/EtOAc = 8/1); white solid; mp 33-34 ˚C; IR (neat NaCl, *ν*) 2919, 2848, 1672, 1469, 1378, 1216, 978, 923, 903, 719 cm^{-1}; ^{1}H NMR (300 MHz, CDCl$_3$, 35 ˚C) δ 0.8-0.9 (m, 2H, CH_2), 0.87 (d, J = 6.6 Hz, 3H, CH(CH_3)$_2$), 0.95 (d, J = 6.6 Hz, 3H, CH(CH_3)$_2$), 1.26 (br, 29H, CH_3, CH_2), 1.5-1.7 (m, 2H, CH_2), 1.7-1.8 (m, 1H, CH(CH$_3$)$_2$), 2.26 (t, J = 7.8 Hz, 2H, CH_2–oxazoline), 3.8-4.0 (m, 2H, NCHCH_2), 4.1-4.2 (m, 1H, NCHCH$_2$); ^{13}C NMR (75 MHz, CDCl$_3$, 35 ˚C) δ 14.15, 18.10, 18.81, 22.75, 26.31, 28.20, 29.31, 29.41, 29.54, 29.65, 29.71, 29.75, 31.98, 32.62, 69.69, 72.58, 167.32; MS (EI) *m*/*z* (relative intensity) 351 ([M$^+$], 16), 322 (11), 309 (61), 308 (100), 293 (11), 280 (14), 167 (11); HRMS (EI) *m*/*z* calcd. for $C_{23}H_{45}NO$ 351.3518, found 351.3501; [α] $^{25}_{589}$ –33.3 (*c* 1.0, CH_2Cl_2).

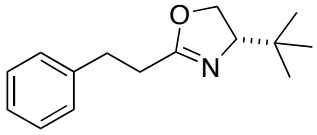
(*S*)-4-Isopropyl-2-phenethyloxazoline (4ja)[SI-3]



Purified by flush column chromatography (silica gel, Hexane/EtOAc = 4/1); colorless oil; IR (neat NaCl, *ν*) 2958, 1671, 1455, 1365, 1234, 1164, 985, 750, 698 cm^{-1}; ^{1}H NMR (300 MHz, CDCl$_3$, 35 ˚C) δ 0.84 (d, J = 6.6 Hz, 3H, CH(CH_3)$_2$), 0.93 (d, J = 6.6 Hz, 3H, CH(CH_3)$_2$), 1.69 (sept like, 1H, CH(CH$_3$)$_2$), 2.58 (t, J = 7.8 Hz, 2H, CH$_2$CH$_2$), 2.95 (t, J = 7.8 Hz, 2H, CH$_2$CH$_2$), 3.89 (m, 2H, CH_2O), 4.18 (m, 1H, CHCH$_2$), 7.1-7.3 (m, 5H, *Ph*); ^{13}C NMR (75 MHz, CDCl$_3$, 35 ˚C) δ 18.05, 18.70, 29.80, 32.30, 32.52,

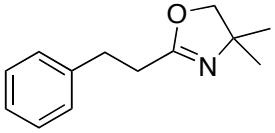
69.77, 72.06, 125.96, 128.11, 128.20, 140.51, 166.23; MS (EI) *m/z* (relative intensity) 217 ([M^+], 84), 174 (92), 91 (82), 18 (100); HRMS (EI) *m/z* calcd. for $C_{14}H_{19}NO$ 217.1467, found 217.1460; $[\alpha]^{25}_{589}$ –47.3 (*c* 1.0, CH_2Cl_2).

(*S*)-2-Phenethyl-4-*tert*-butyloxazoline (4jb)[SI-3]



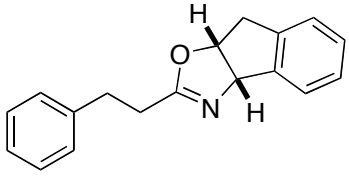
Purified by flush column chromatography (silica gel, Hexane/EtOAc = 6/1); colorless oil; IR (neat NaCl, *ν*) 2954, 1673, 1362, 1230, 1209, 1171, 983, 698 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.84 (s, 9H, $C(CH_3)_3$), 2.5-2.6 (m, 2H, CH_2CH_2), 2.94 (t, J = 8.1, 2H, CH_2CH_2), 3.7-3.8 (m, 1H, $NCHCH_2$), 4.00 (dd, J = 8.4, 8.4 Hz, 1H, $NCHCH_2$), 4.11 (dd, *J* = 8.4, 10.2 Hz, 1H, $NCHCH_2$), 7.1-7.3 (m, 5H, *Ph*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 25.72, 29.73, 32.30, 33.41, 68.30, 75.70, 125.91, 128.11, 128.15, 140.49, 166.09; MS (EI) *m/z* (relative intensity) 231 ([M^+], 64), 174 (100), 91 (55); HRMS (EI) *m/z* calcd. for $C_{15}H_{21}NO$ 231.1623 found 231.1612; $[\alpha]^{25}_{589}$ –47.1 (*c* 1.1, CH_2Cl_2).

4,4-Dimethyl-2-phenethyloxazoline (4jc)[SI-5]



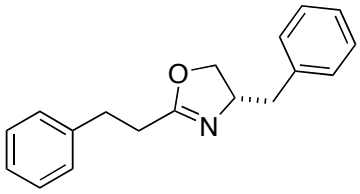
Purified by flush column chromatography (silica gel, Hexane/EtOAc = 4/1); colorless oil; IR (neat NaCl, *ν*) 1668, 1604, 1496, 1455, 1363, 1146, 1078, 990, 750, 699 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 °C) δ 1.23 (s, 6H, $NC(CH_3)_2$), 2.55 (t, *J* = 7.5 Hz, 2H, $PhCH_2CH_2$), 3.03 (t, *J* = 7.5 Hz, 2H, $PhCH_2CH_2$), 3.88 (s, 2H, $NCCH_2$), 7.2-7.3 (m, 5H, *Ph*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 28.43, 29.93, 32.29, 66.93, 78.95, 126.04, 128.20, 128.26, 140.50, 164.92; MS (EI) *m/z* (relative intensity) 203 ([M^+], 100), 202 (65), 188 (22), 162 (16); HRMS (EI) *m/z* calcd. for $C_{13}H_{17}NO$ 203.1310, found 203.1283.

(3a*S*,8a*R*)-2-Phenethyl-3a,8a-dihydro-8*H*-indeno[1,2-*d*]oxazole (4jd)



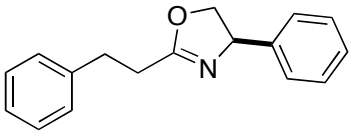
Purified by flush column chromatography (silica gel, Hexane/EtOAc = 4/1); white solid; mp 78-81 °C; IR (neat NaCl, *ν*) 1667, 1604, 1495, 1454, 1078, 753, 699 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 °C) δ 2.53 (t, *J* = 7.5 Hz, 2H, $PhCH_2CH_2$), 2.90 (t, *J* = 7.5 Hz, 2H, $PhCH_2CH_2$), 3.17 (dd, *J* = 1.5, 17.7 Hz, 1H, NCHCHO), 3.38 (dd, *J* = 7.2 17.7 Hz, 1H, NCHCHO), 5.26 (ddd, *J* = 3.0, 7.2, 7.5 Hz, 1H, $OCHCH_2Ar$), 5.49 (d, *J* = 7.5 Hz, 1H, $OCHCH_2Ar$), 7.1-7.5 (m, 9H, *Ar*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 30.01, 32.30, 39.83, 76.53, 82.85, 125.12, 125.37, 126.02, 127.32, 128.14, 128.27, 139.55, 140.40, 142.04, 167.12; MS (EI) *m/z* (relative intensity) 263 ([M^+], 100), 262 (43), 236 (20), 228 (10), 227 (74), 224 (10), 212 (20), 203 (42), 202 (25), 186 (38), 182 (18), 170 (13), 162 (55); HRMS (EI) *m/z* calcd. for $C_{18}H_{17}NO$ 263.1310, found 263.1297; $[\alpha]^{25}_{589}$ –157.8 (*c* 1.0, CH_2Cl_2).

(*S*)-2-Phenethyl-4-phenylmethyloxazoline (4je)[SI-6]



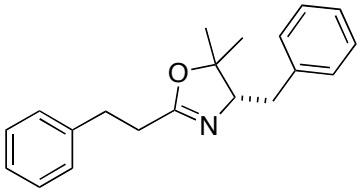
Purified by flush column chromatography (silica gel, Hexane/EtOAc = 4/1); colorless oil; IR (neat NaCl, ν) 1667, 1496, 1454, 983, 751, 699 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 ˚C) δ 2.5-2.6 (m, 3H, PhCH_2, PhCH_2CH_2), 2.93 (t, J = 8.0 Hz, 2H, PhCH_2CH_2), 3.00 (dd, J = 5.1, 13.8 Hz, 1H, PhCH_2), 3.89 (dd, J = 7.5, 8.4 Hz, 1H, NCHCH_2), 4.09 (dd, J = 8.4, 8.4 Hz, 1H, NCHCH_2), 4.2-4.4 (m, 1H, NCHCH$_2$), 7.1-7.3 (m, 10H, *Ph*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 ˚C) δ 29.63, 32.03, 41.67, 67.08, 71.37, 125.92, 126.15, 128.02, 128.15, 128.16, 128.94, 137.67, 140.36, 166.75; MS (EI) *m/z* (relative intensity) 266 ([M^++H], 19), 265 (100), 244 (13), 236 (23), 224 (10), 217 (26), 212 (20); HRMS (EI) *m/z* calcd. for $C_{12}H_{21}NO$ 265.1467, found 265.1483; $[\alpha]^{25}_{589}$ –40.9 (*c* 1.1, CH_2Cl_2).

(*R*)-4-Phenyl-2-(3-phenylethyl)oxazoline (4jf)[SI-3,6]



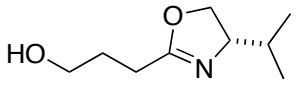
Purified by flush column chromatography (silica gel, Hexane/EtOAc = 4/1); colorless oil; IR (neat NaCl, ν) 1667, 1604, 1495, 1454, 1078, 753, 699 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 ˚C) δ 2.71 (t, J = 7.5 Hz, 2H, PhCH_2CH_2), 3.03 (t, J = 7.5 Hz, 2H, PhCH_2CH$_2$), 4.02 (dd, J = 8.4, 8.4 Hz, 1H, NCHCH_2), 4.56 (dd, J = 8.4 10.2 Hz, 1H, NCHCH_2), 5.13 (m, 1H, NCHCH$_2$), 7.1-7.3 (m, 10H, *Ph*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 ˚C) δ 29.72, 32.15, 69.58, 74.54, 126.10, 126.40, 127.27, 128.24, 128.34, 128.47, 140.36, 142.22, 167.71; MS (EI) *m/z* (relative intensity) 251 ([M^+], 88), 250 (41), 220 (10), 204 (12), 203 (100), 202 (56), 188 (25), 174 (11), 212 (20); HRMS (EI) *m/z* calcd. for $C_{17}H_{17}NO$ 251.1310, found 251.1305; $[\alpha]^{25}_{589}$ +47.8 (*c* 1.0, CH_2Cl_2).

(*S*)-5,5-Dimethyl-2-(3-phenylethyl)-4-phenylmethyloxazoline (4jg)



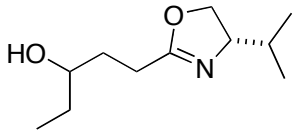
Purified by flush column chromatography (silica gel, Hexane/EtOAc = 8/1); colorless oil; IR (neat NaCl, ν) 3027, 2974, 1663, 1496, 1454, 1386, 1370, 1146, 742, 698 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 ˚C) δ 1.22 (s, 3H, CH_3), 1.25 (s, 3H, CH_3), 2.54 (t, J = 8.1 Hz, 2H, PhCH_2CH_2), 2.66 (dd, 1H, J = 7.2, 14.1 Hz, CHCH_2Ph), 2.86 (dd, J = 7.8, 14.1 Hz, 1H, CHCH_2Ph), 2.93 (t, J = 8.1 Hz, 1H, PhCH_2CH$_2$), 3.92 (dd, J = 7.2, 7.8 Hz, 1H, CHCH$_2$Ph), 7.2-7.3 (m, 10H, *Ph*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 ˚C) δ 21.89, 28.50, 30.20, 32.14, 37.64, 74.72, 85.68, 125.97, 128.16, 128.18, 128.22, 128.81, 139.07, 140.52, 165.16; MS (EI) *m/z* (relative intensity) 293 ([M^+], 35), 202 (100); HRMS (EI) *m/z* calcd. for $C_{20}H_{23}NO$ 293.1780, found 293.1791; $[\alpha]^{25}_{589}$ –99.1 (*c* 1.1, CH_2Cl_2).

(*S*)-2-(3-Hydroxypropyl)-4-isopropyloxazoline (6aa)



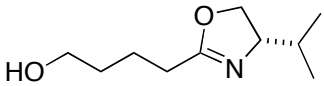
Purified by flush column chromatography (silica gel, Hexane/EtOAc/Et_3N = 20/10/1); colorless oil; IR (neat NaCl, ν) 3298, 2960, 1664, 1468, 1368, 984 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.88 (d, J = 6.9 Hz, 3H, $CH(CH_3)_2$), 0.95 (d, J = 6.9 Hz, 3H, $CH(CH_3)_2$), 1.6-1.8 (m, 1H, $CH(CH_3)_2$), 1.89 (tt, J = 6.0, 6.6 Hz, 2H, $HOCH_2CH_2CH_2$), 2.42 (t, J = 6.6 Hz, 2H, $HOCH_2CH_2CH_2$), 3.69 (t, J = 6.0 Hz, 2H, $HOCH_2CH_2CH_2$), 3.8-4.0 (m, 2H, $NCHCH_2$), 4.2-4.3 (m, 1H, $NCHCH_2$), 4.4 (br, 1H, O*H*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 18.23, 18.67, 26.02, 28.51, 32.62, 62.25, 70.38, 71.71, 168.29; MS (EI) *m*/*z* (relative intensity) 128 ([M^+–*i*-Pr], 95), 110 (100); $[\alpha]^{25}_{589}$ –49.5 (*c* 1.0, CH_2Cl_2).

(*S*)-2-[(*rac*)-3-Hydroxypentyl]-4-isopropyloxazoline (6ba)



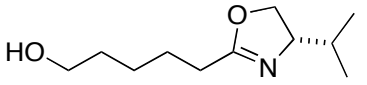
Purified by flush column chromatography (silica gel, Hexane/EtOAc/Et_3N = 20/20/1); colorless oil; IR (neat NaCl, ν) 3316, 2961, 1668, 1465, 1364, 987 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.87 (d, J = 6.9 Hz, 3H, $CH(CH_3)_2$), 0.95 (t, J = 6.9 Hz, 3H, CH_2CH_3), 1.4-1.6 (m, 2H, CH_2CH_3), 4.7-4.9 (m, 3H, $HOCH(Et)CH_2CH_2$, $CH(CH_3)_2$), 2.3-2.5 (m, 2H, $HOCH(Et)CH_2CH_2$), 3.5-3.6 (m, 1H, $HOCH(Et)CH_2CH_2$), 3.8-4.0 (m, 2H, $NCHCH_2$), 4.2-4.3 (m, 1H, $NCHCH_2$), 4.4 (br, 1H, O*H*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 10.03, 10.10, 18.29, 18.31, 18.73, 18.79, 25.34, 25.39, 30.44, 32.35, 32.50, 32.67, 70.32, 70.38, 71.77, 71.85, 72.78, 72.86, 168.36, 168.47; MS (EI) *m*/*z* (relative intensity) 198 ([M^+–H], 33), 182 (14), 179 (20), 167 (29), 165 (58), 153 (94); HRMS (EI) *m*/*z* calcd. for $C_{11}H_{20}NO_2$([M^+–H]) 198.1494 found 198.1515.

(*S*)-2-(4-Hydroxybutyl)-4-isopropyloxazoline (6ca)



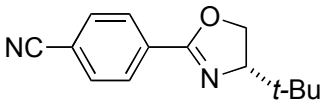
Purified by flush column chromatography (silica gel, Hexane/EtOAc/Et_3N = 10/20/1); colorless oil; IR (neat NaCl, ν) 3299, 2959, 2873, 1662, 1542, 1464, 1368, 1066, 984 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.87 (d, J = 6.9 Hz, 3H, $CH(CH_3)_2$), 0.95 (d, J = 6.9 Hz, 3H, $CH(CH_3)_2$), 1.5-1.8 (m, 5H, $CH_2CH_2CH_2OH$ and $CH(CH_3)_2$), 2.31 (t, J = 7.2 Hz, 2H, $CH_2(CH_2)_3OH$), 3.60 (t, J = 6.0 Hz, 2H, CH_2OH), 3.8-3.9 (m, 2H, $NCHCH_2$), 4.0 (br, 1H, O*H*), 4.1-4.3 (m, 1H, $NCHCH_2$); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 17.85, 18.51, 21.96, 27.38, 32.01, 32.30, 61.07, 69.65, 71.51, 167.68; MS (EI) *m*/*z* (relative intensity) 185 ([M^+], 100), 170 (11), 168 (19), 156 (63); HRMS (EI) *m*/*z* calcd. for $C_{10}H_{19}NO_2$ 185.1416, found 185.1411; $[\alpha]^{25}_{589}$ –43.3 (*c* 1.2, CH_2Cl_2).

(*S*)-2-(5-Hydroxypentyl)-4-isopropyloxazoline (6da)



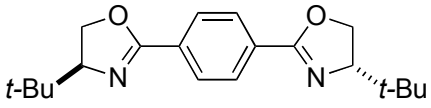
Purified by flush column chromatography (silica gel, Hexane/EtOAc/Et_3N = 10/20/1); colorless oil; IR (neat NaCl, ν) 3309, 2934, 2871, 1664, 1465, 1368, 984 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.87 (d, J = 6.9 Hz, 3H, $CH(CH_3)_2$), 0.94 (d, J = 6.9 Hz, 3H, $CH(CH_3)_2$), 1.3-1.8 (m, 7H, $(CH_2)_3CH_2OH$ and $CH(CH_3)_2$), 2.28 (t, J = 7.5 Hz, 2H, $CH_2(CH_2)_3OH$), 3.5 (br, 1H, O*H*), 3.60 (t, J = 6.3 Hz, 2H, CH_2OH), 3.8-3.9 (m, 2H, $NCHCH_2$), 4.1-4.3 (m, 1H, $NCHCH_2$); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 17.79, 18.54, 25.26, 25.53, 27.80, 32.08, 32.29, 61.77, 69.54, 71.54, 167.48; MS (EI) *m/z* (relative intensity) 199 ([M^+], 28), 184 (10), 182 (36), 170 (20), 169 (27), 158 (36), 157 (100); HRMS (EI) *m/z* calcd. for $C_{11}H_{21}NO_2$ 199.1572, found 199.1543; $[\alpha]^{25}_{589}$ –46.0 (*c* 1.2, CH_2Cl_2).

(*S*)-4-*tert*-Butyl-2-(4-cyanophenethyl)oxazoline (9a)



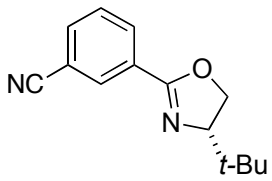
Purified by column chromatography (silica gel, Hexane/EtOAc = 8/1); white solid; mp 105-107 °C; IR (film NaCl, ν) 2965, 2229, 1651, 1361, 1260, 1082, 955 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.95 (s, 9H, $C(CH_3)_3$), 4.08 (dd, J = 8.1, 10.2 Hz, 1H, $CHCH_2$), 4.26 (dd, J = 8.1, 10.2 Hz, 1H, $CHCH_2$), 4.38 (dd, J = 8.1, 8.1 Hz, 10.2 Hz, 1H, $CHCH_2$), 7.68 (dd, J = 1.8, 6.6 Hz, 2H, *Ar*), 8.05 (dd, J = 1.8, 6.6 Hz, 2H, *Ar*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 25.92, 34.09, 69.16, 76.50, 114.50, 118.18, 128.69, 131.88, 132.05, 161.48; MS (EI) *m/z* (relative intensity) 228 ([M^+], 3), 213 (4), 183 (4), 172 (100); HRMS (EI) *m/z* calcd. for $C_{14}H_{16}N_2O$ 228.1263 found 228.1251; $[\alpha]^{25}_{589}$ –66.3 (*c* 1.0, CH_2Cl_2).

1,4-Bis[(*S*)-4-*tert*-butyloxazolin-2-yl]benzene (10)



Purified by column chromatography (silica gel, Hexane/EtOAc = 8/1); white solid; mp 190-195 °C; IR (film NaCl, ν) 2959, 1650, 1356, 1263, 1075, 1054, 968, 904 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.96 (s, 18H, $C(CH_3)_3$), 4.05 (dd, J = 8.1, 10.2 Hz, 2H, $CHCH_2$), 4.24 (dd, J = 8.1, 8.1 Hz, 2H, $CHCH_2$), 4.35 (dd, J = 8.1, 10.2 Hz, 2H, $CHCH_2$), 7.98 (s, 4H, *Ar*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 25.99, 34.12, 68.83, 76.41, 128.02, 130.31, 162.52; MS (EI) *m/z* (relative intensity) 328 ([M^+], 1), 313 (4), 271 (100); HRMS (EI) *m/z* calcd. for $C_{20}H_{28}N_2O_2$ 328.2151 found 328.2144; $[\alpha]^{25}_{589}$ –98.3 (*c* 0.27, CH_2Cl_2).

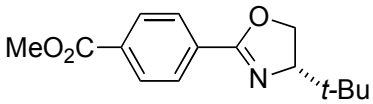
(*S*)-4-*tert*-Butyl-2-(3-cyanophenethyl)oxazoline (9b)



Purified by flush column chromatography (silica gel, Hexane/EtOAc = 8/1); white solid; mp 65–67 °C; IR (film NaCl, ν) 2234, 1653, 1361, 1180, 1079, 962, 815, 698 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.96 (s, 9H, $C(CH_3)_3$), 4.07 (dd, J = 8.1, 10.2 Hz, 1H, $NCHCH_2$), 4.26 (dd, J = 8.1, 8.7 Hz, 1H, $NCHCH_2$), 4.38 (dd, J = 8.7, 10.2 Hz, 1H, $NCHCH_2$), 7.51 (dd, J = 6.3, 7.5 Hz, 1H, *Ar-5*), 7.73

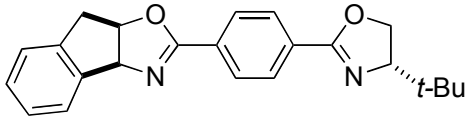
(dd, *J* = 1.5, 7.5 Hz, 1H, *Ar-4* or *6*), 8.18 (dd, *J* = 1.5, 6.3 Hz, 1H, *Ar-4* or *6*), 8.25 (dd, *J* = 1.5, 1.5 Hz, *Ar-2*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 ˚C) δ 25.89, 34.02, 69.16, 76.39, 112.65, 117.97, 129.01, 129.31, 131.75, 132.13, 134, 01, 161.07; MS (EI) *m/z* (relative intensity) 228 ([M^++H], 5), 172 (100), 143 (20), 130 (35); HRMS (EI) *m/z* calcd. for $C_{14}H_{16}N_2O$ 228.1263, found 228.1241; $[\alpha]^{25}_{589}$ –59.5 (*c* 1.0, CH_2Cl_2).

(*S*)-4-*tert*-Butyl-2-(4-methoxycarbonylphenethyl)oxazoline (13)



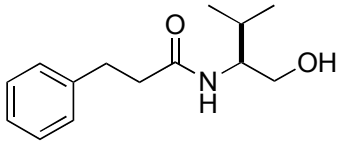
Purified by flush column chromatography (silica gel, Hexane/EtOAc = 8/1); white solid; mp 117–118 ˚C; IR (film NaCl, *ν*) 1720, 1651, 1434, 1407, 1279, 1105, 1079, 1058, 1014, 972, 866, 708 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, 35 ˚C) δ 0.96 (s, 9H, $C(CH_3)_3$), 3.93 (s, 3H, $COOCH_3$), 4.07 (dd, *J* = 9.0, 10.2 Hz, 1H, $NCHCH_2$), 4.25 (dd, *J* = 9.0, 9.0 Hz, 1H, $NCHCH_2$), 4.37 (dd, *J* = 9.0, 10.2 Hz, 1H, $NCHCH_2$), 8.01 (d, *J* = 8.4 Hz, 2H, *Ar*), 8.06 (d, *J* = 8.4 Hz, 1H, *Ar*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 ˚C) δ 25.98, 34.12, 52.26, 68.96, 76.47, 128.13, 129.35, 132.00, 132.24, 162.30, 166.35; MS (EI) *m/z* (relative intensity) 266 ([M^++H], 19), 265 (100), 244 (13), 236 (23), 224 (10), 217 (26), 212 (20); HRMS (EI) *m/z* calcd. for $C_{15}H_{19}NO_3$ 261.1365, found 261.1350; $[\alpha]^{25}_{589}$ –48.9 (*c* 1.0, CH_2Cl_2).

1-[(3a*S*,8a*R*)-8,8a-dihydro-3a*H*-indeno[1,2-*d*]oxazol-2-yl]-4-[(*S*)-4-*tert*-butyloxazolin-2-yl]benzene (14)



Purified by column chromatography (silica gel, Hexane/EtOAc = 8/1); white solid; mp 178-179 ˚C (dec.); IR (film NaCl, *ν*) 2956, 1644, 1412, 1355, 1086, 1071, 860, 751, 688 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, 35 ˚C) δ 0.95 (s, 9H, $C(CH_3)_3$), 3.36 (dd, *J* = 1.8, 18.0 Hz, 1H, CH_2Ar), 3.51 (dd, *J* = 6.6, 18.0 Hz, 1H, CH_2Ar), 4.04 (dd, *J* = 8.1, 10.2 Hz, 1H, $CHCH_2$), 4.22 (dd, *J* = 8.1, 1H, $CHCH_2$), 4.34 (dd, *J* = 8.1, 10.2 Hz, 1H, $CHCH_2$), 5.49 (ddd, *J* = 1.8, 6.6, 7.8 Hz, 1H, NCHCHO), 5.75 (d, *J* = 7.8 Hz, 1H, NCHCHO), 7.25–7.27 (m, 3H, Ar), 7.55–7.58 (m, 1H, *Ar*), 7.95 (s, 4H, *Ar*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 ˚C) δ 25.99, 34.11, 39.90, 68.84, 76.42, 77.18, 125.21, 125.56, 127.42, 128.01, 128.15, 128.44, 130.21, 130.42, 139.60, 141.74, 162.47, 163.28; MS (EI) *m/z* (relative intensity) 360 ([M^+], 1), 345 (1), 303 (45), 227 (16); HRMS (EI) *m/z* calcd. for $C_{23}H_{24}N_2O_2$ 360.1838 found 360.1843; $[\alpha]^{25}_{589}$ –140.6 (*c* 1.0, CH_2Cl_2).

(*S*)-*N*-(1-hydroxy-3-methylbutan-2-yl)-3-phenylpropanamide (15ja)[SI-7]



purified by flush column chromatography (silica gel, Hexane/EtOAc = 1/4 to 8); white solid (2.42 g, 50% yield); mp

76-79 °C; IR (neat NaCl, ν) 3309, 2934, 2871, 1664, 1465, 1368, 984 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 35 °C) δ 0.82 (d, J = 6.9 Hz, 3H, $CH(CH_3)_2$), 0.87 (d, J = 6.9 Hz, 3H, $CH(CH_3)_2$), 1.7–1.9 (m, 1H, $CH(CH_3)_2$), 2.52 (t, J = 7.5 Hz, 2H, $PhCH_2CH_2$), 2.82 (bs, 1H, OH), 2.96 (t, J = 7.5 Hz, 2H, $PhCH_2CH_2$), 3.5–3.7 (m, 3H, $NCHCH_2$), 5.69 (d, J = 7.5 Hz, 1H, NH), 7.1–7.3 (m, 5H, *Ph*); ^{13}C NMR (75 MHz, $CDCl_3$, 35 °C) δ 18.75, 19.42, 28.95, 31.85, 38.58, 57.08, 63.65, 126.18, 128.21, 128.43, 140.52, 172.82; MS (EI) *m/z* (relative intensity) 199 ($[M^+]$, 28), 184 (10), 182 (36), 170 (20), 169 (27), 158 (36), 157 (100); HRMS (EI) *m/z* calcd. for $C_{11}H_{21}NO_2$ 199.1572, found 199.1543; $[\alpha]^{25}_{589}$ –29.2 (*c* 1.0, CH_2Cl_2).

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