Electronic Supplementary Material (ESI) for Chemical Communications

The First Asymmetric Ring-Expansion Carbonylation of *meso*-Epoxides

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Supporting Information

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General remarks

NMR spectra were recorded on a Varian 400 MHz FT spectrometer at room temperature with $CDCl_3$ as the internal standard. The reference values used for $CDCl_3$ were 7.26 and 77.0 ppm for ^1H and ^{13}C NMR spectra, respectively. High resolution mass spectra were measured on a Waters/Micromass GCT and Waters 2996 Photodiode Array Detector instruments. Infrared spectra were recorded on a Varian 3100 FT-IR spectrometer at room temperature. Melting points were recorded in open capillaries on a digital Barnsted Electro Thermal 9300 melting point apparatus and are uncorrected. Chiral HPLC analysis was performed on an Agilent technologies 1200 series instrument. Optical rotation values were measured at room temperature on a Perkin-Elmer 343 polarimeter. All carbonylation reactions were carried out in a 100 mL custom built Parr reactor equipped with magnetic stirrer, which has an inlet, outlet, syringe port and pressure burst valve. Heating was performed in an oil bath.

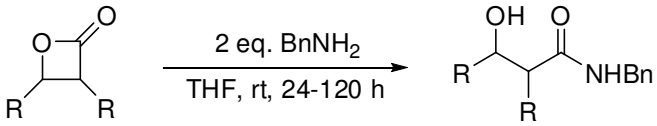
Materials

All reagents were obtained from commercial suppliers and used without further purification. Carbon monoxide gas (99.97% purity) was purchased from CK Gas Ltd. $Co_2(CO)_8$ was purchased from Strem Chemicals and used as received. Jacobsen's catalysts (*R,R*)-(salen)CrCl **1** was purchased from Sigma Aldrich and used as received. Anhydrous DME was purchased from Sigma Aldrich and degassed prior to use. Dry THF was obtained from a solvent purification system and degassed prior to use. Thin layer chromatography was performed on Merck Aluminium sheets (silica gel 60 F_{254}). Detection was carried out by UV and by coloration with ceric ammonium molybdate (CAM) or vanillin. Flash column chromatography was performed using Merck silica gel 60 (230-400 mesh). The enantiomeric excess was determined by chiral HPLC analysis on a CHIRALPAK® IB column using HPLC

grade *n*-heptane and ethanol as the eluent. β-Lactones without a UV active chromophore were measured after conversion into the β-hydroxy benzylamide derivatives.

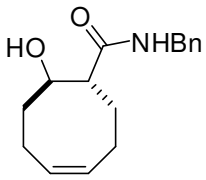
The following compounds were synthesized according to literature procedures: [(*R,R*)-(salen)Cr]BF_4 **2**,[1] D_4-symmetric porphyrin Cr(III) chloride **3**,[2] 1,2-epoxy-5-cyclooctene,[3] *cis*-cyclooctane oxide,[4] *cis*-cyloheptene oxide,[5] *cis*-cyclopentene oxide,[6] *cis*-1,2-epoxycyclododecane,[7] *cis*-1,4-dibenzyloxy-2,3-epoxybutane,[8] 3,4-epoxytetrahydro-furan,[9] dimethyl 6-oxabicyclo[3.1.0]hexane-3,3-dicarboxylate[10] and 1-tosyl-2,3,6,7-tetrahydro-1*H*-azepine.[11] All epoxides were purified/dried by either column chromatography or by distillation over CaH_2 prior to use. Racemic β-lactones were prepared according to our previously published procedure.[10]

General procedure for the preparation of β-hydroxy benzylamides (GP I)



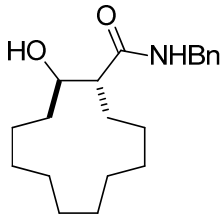
In an oven-dried Schlenk tube, a β-lactone (0.6-1.0 mmol) and anhydrous THF (3 mL) were charged under N_2 atmosphere. To this stirred solution was added $BnNH_2$ (2 eq.) *via* a syringe and the resulting mixture was stirred at room temperature until TLC analysis showed complete consumption of the β-lactone (24-120 h). The reaction mixture was evaporated under reduced pressure and the residue was purified by flash column chromatography to give the β-hydroxy benzylamide derivatives.

(±)-*trans*-*N*-Benzyl-8-hydroxycyclooct-4-enecarboxamide



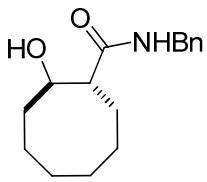
According to GP I, the racemic β-lactone (100 mg, 0.65 mmol) and benzylamine (137 mg, 1.3 mmol) in THF (3 mL) were stirred at room temperature for 24 h. The title compound was isolated by flash column chromatography (SiO_2; pentane/EtOAc, 90:10) as a white solid (164 mg, 96%). Mp: 172-176 ^{o}C; IR (KBr, cm^{-1}): 3397, 2948, 1654, 1283; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.48-1.72 (m, 2 H, CH_2), 1.68-1.82 (m, 1 H, CH*H*), 1.91-2.01 (m, 1 H, C*H*H), 2.02-2.35 (m, 4 H, CH_2), 2.36-2.48 (m, 1 H, C*H*H), 2.52 (ddd, *J* = 11.3, 8.1, 3.3 Hz, 1 H, C*H*CO), 3.12 (brs, 1 H, CHO*H*), 4.01 (ddd, *J* = 16.0, 8.1, 3.4 Hz, 1 H, C*H*OH), 4.40 (dd, *J* = 14.8, 5.7 Hz, 1 H, C*H*HPh), 4.47 (dd, *J* = 14.8, 5.7 Hz, 1 H, CH*H*Ph), 5.52-5.56 (m, 1 H, CH=CH), 5.63-5.77 (m, 1 H, CH=CH), 5.86 (brs, 1 H, CONH), 7.16-7.43 (m, 5 H, Ar-H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.2, 24.0, 30.2, 35.3, 43.6, 52.3, 72.0, 127.6, 127.7, 128.0, 128.8, 131.0, 138.0, 175.9; HRMS (EI): $C_{16}H_{22}NO_2$ $[M + H]^+$ calculated: 260.1651, found: 260.1656.

(±)-*trans*-*N*-Benzyl-2-hydroxycyclododecanecarboxamide



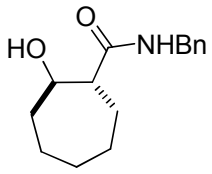
According to GP I, the racemic β-lactone (100 mg, 0.47 mmol), benzylamine (137 mg, 1.3 mmol) in THF (3 mL) were stirred at room temperature for 5 days. The title compound was isolated by flash column chromatography (SiO_2; pentane/EtOAc, 90:10) as a white solid (131 mg, 87%). Mp: 181-185 °C; IR (KBr, cm^{-1}): 3312, 1661, 1610, 1220; ^1H NMR (400 MHz, $CDCl_3$): δ 1.13-1.53 (m, 16 H, CH_2), 1.52-1.69 (m, 6 H, CH_2), 2.40 (dd, *J* = 13.1, 6.0 Hz, 1 H, C*H*CO), 3.07 (brs, 1 H, CHO*H*), 4.04-4.08 (m, 1 H, C*H*OH), 4.48 (dd, *J* = 15.6, 5.7 Hz, 1 H, C*H*HPh), 4.48 (dd, *J* = 15.6, 5.7 Hz, 1 H, CH*H*Ph), 6.01 (brs, 1 H, CONH), 7.14-7.46 (m, 5 H, Ar-H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 20.6, 22.2, 23.3, 23.35, 23.6, 23.7, 23.9, 26.7, 31.0, 43.5, 48.0, 69.0, 76.7, 127.5, 127.7, 128.7, 138.2, 175.2; HRMS (EI): $C_{20}H_{31}NO_2$ [M + H]$^+$ calculated: 318.2433, found: 318.2444.

(±)-*trans*-*N*-Benzyl-2-hydroxycyclooctanecarboxamide



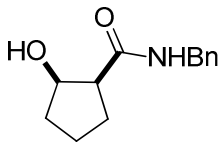
According to GP I, the racemic β-lactone (100 mg, 0.65 mmol) and benzylamine (101 mg, 0.95 mmol) in THF (3 mL) were stirred at room temperature for 24 h. The title compound was isolated by flash column chromatography (SiO_2; pentane/EtOAc, 90:10) as a white solid (159 mg, 94%). Mp: 186-189 °C; IR (KBr, cm^{-1}): 3403, 2929, 1654, 1236; ^1H NMR (400 MHz, $CDCl_3$): δ 1.40-1.80 (m, 10 H, CH_2), 1.81-1.98 (m, 2 H, CH_2), 2.35 (ddd, *J* = 10.1, 7.7, 2.7 Hz, 1 H, CHCO), 2.61 (brs, 1 H, CHO*H*), 4.01 (virtual t, *J* = 8.5 Hz, 1 H, C*H*OH), 4.44 (dd, *J* = 14.6, 5.6 Hz, 1 H, C*H*HPh), 4.46 (dd, *J* = 14.8, 5.9 Hz, 1 H, CH*H*Ph), 6.14 (brs, 1 H, CONH), 7.16-7.43 (m, 5 H, Ar-H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.1, 25.3, 26.7, 27.5, 33.3, 43.5, 52.1, 72.4, 127.4, 127.7, 128.5, 128.7, 138.3, 176.2; HRMS (ESI): $C_{16}H_{23}NO_2$ [M + Na]$^+$ calculated: 284.1626, found: 284.1638.

(±)-*trans*-*N*-Benzyl-2-hydroxycycloheptanecarboxamide



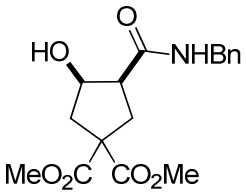
According to GP I, the racemic β-lactone (100 mg, 0.71 mmol) and benzylamine (151 mg, 1.42 mmol) in THF (3 mL) were stirred at room temperature for 24 h. The title compound was isolated by flash column chromatography (SiO_2; pentane/EtOAc, 90:10) as a white solid (158 mg, 90%). Mp: 148-153 ^oC; IR (KBr, cm^{-1}): 3403, 2929, 1654, 1236; ^1H NMR (400 MHz, $CDCl_3$): δ 1.39-2.02 (m, 10 H, CH_2), 2.24 (ddd, *J* = 12.1, 9.4, 2.5 Hz, 1 H, CHCO), 2.61 (brs, 1 H, CHO*H*), 4.01 (virtual t, *J* = 8.5 Hz, 1 H, C*H*OH), 4.42 (dd, *J* = 14.8, 5.7 Hz, 1 H, C*H*HPh), 4.46 (dd, *J* = 14.8, 5.7 Hz, 1 H, CH*H*Ph), 6.14 (brs, 1 H, CONH), 7.16-7.43 (m, 5 H, Ar-H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.8, 27.3, 28.0, 28.3, 36.7, 43.9, 55.3, 74.4, 127.9, 128.1, 129.2, 138.8, 176.3; HRMS (EI): $C_{15}H_{21}NO_2$ $[M]^+$ calculated: 247.1572, found: 247.1564.

(±)-*cis*-*N*-Benzyl-2-hydroxycyclopentanecarboxamide[12]



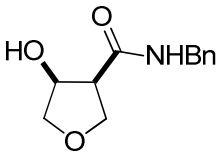
According to GP I, the racemic β-lactone (100 mg, 0.9 mmol) and benzylamine (189 mg, 1.8 mmol) in THF (3 mL) were stirred at room temperature for 24 h. The title compound was isolated by flash column chromatography (SiO_2; pentane/EtOAc, 90:10) as a white solid (195 mg, 98%). Mp: 85-89 ^oC; IR (KBr, cm^{-1}): 3400, 3020, 1655, 1222; ^1H NMR (400 MHz, $CDCl_3$): δ 1.55-1.87 (m, 6 H, CH_2), 2.44 (ddd, *J* = 10.8, 8.2, 3.9 Hz, 1 H, CHCO), 4.22 (brs, 1 H, CHO*H*), 4.40-4.45 (m, 3 H, CH_2Ph, C*H*OH), 6.26 (brs, 1 H, CONH), 7.14-7.50 (m, 5 H, Ar-H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.1, 27.6, 34.3, 43.4, 49.9, 74.1, 127.6, 127.7, 128.8, 137.9, 175.0; HRMS (EI): $C_{13}H_{18}NO_2$ $[M + H]^+$ calculated: 220.1338, found: 220.1330.

(±)-*cis*-Dimethyl-3-(benzylcarbamoyl)-4-hydroxycyclopentane-1,1-dicarboxylate



According to GP I, the racemic β-lactone (100 mg, 0.43 mmol) and benzylamine (93 mg, 0.86 mmol) in THF (3 mL) were stirred at room temperature for 72 h. The title compound was isolated by flash column chromatography (SiO_2; pentane/EtOAc, 70:30) as pale yellow coloured oil (129 mg, 88%). IR (neat, cm^{-1}): 3403, 2954, 1734, 1638, 1182; ^1H NMR (400 MHz, $CDCl_3$): δ 2.29 (dd, *J* = 14.5, 4.3 Hz, 1 H, C*H*CO), 2.64-2.81 (m, 2 H, C*H*H), 2.49-2.63 (m, 2 H, CH*H*), 3.70 (s, 3 H, CH_3), 3.74 (s, 3 H, CH_3), 4.35 (brs , 1 H, CHO*H*), 4.42 (d, *J* = 3.2 Hz, 1 H, C*H*HPh), 4.44 (d, *J* = 3.2 Hz, 1 H, CH*H*Ph), 4.45-4.47 (m, 1 H, C*H*OH), 6.60 (virtual t, *J* = 5.9 Hz, 1 H, CONH), 7.16-7.44 (m, 5 H, Ar-H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 35.6, 42.7, 43.5, 49.4, 53.1, 58.4, 73.5, 127.7, 127.8, 128.8, 137.8, 172.3, 172.9, 173.0; HRMS (ESI): $C_{17}H_{21}NO_6$ $[M + Na]^+$ calculated: 358.1267, found: 358.1278.

(±)-*cis*-*N*-Benzyl-4-hydroxytetrahydrofuran-3-carboxamide



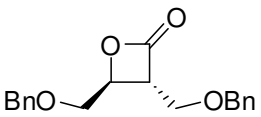
According to GP I, the racemic β-lactone (100 mg, 0.87 mmol) and benzylamine (185 mg, 1.75 mmol) in THF (3 mL) were stirred at room temperature for 72 h. The title compound was isolated by flash column chromatography (SiO_2; pentane/EtOAc, 20:80) as a cream coloured solid (169 mg, 88%). Mp: 114-118 ^oC; IR (KBr, cm^{-1}): 3406, 1642, 1473, 1226, 1122; ^1H NMR (400 MHz, $CHCl_3$): δ 2.90 (ddd, *J* = 14.0, 9.0, 5.0 Hz, 1 H, CHCO), 3.87-3.91 (m, 2 H, CH_2), 4.09 (d, *J* = 9.0 Hz, 2 H, CH_2), 4.29 (d, *J* = 3.9 Hz, 1 H, CHO*H*), 4.50 (dd, *J* = 14.6, 5.5 Hz, 1 H, C*H*HPh), 4.53 (dd, *J* = 14.6, 5.7 Hz, 1 H, CH*H*Ph), 4.58-4.61 (m, 1 H, C*H*OH), 6.08 (brs, 1 H, CONH), 7.24-7.40 (m, 5 H, Ar-H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 43.7, 49.3, 69.0, 72.6, 75.9, 127.78, 127.8, 128.8, 128.9, 137.4, 170.96; HRMS (ESI): $C_{17}H_{21}NO_6$ $[M + Na]^+$ calculated: 244.0950, found: 244.0943.

General procedure for the asymmetric carbonylation of *meso*-epoxides (GP II)

In an oven-dried Schlenk tube, chiral chromium chloride catalyst **1** or **3** (0.005-0.05 mmol) and $Co_2(CO)_8$ (0.0075-0.05 mmol) were charged under N_2 atmosphere. Dry DME (1.5 mL) and the *meso*-epoxide (1.0 mmol) were added and the reaction mixture was degassed by three vacuum/nitrogen cycles. The reaction mixture was injected *via* a syringe into an oven-dried reactor under N_2. The reactor was pressurised with CO gas to about 20 psi and vented. Subsequently it was pressurised to 500 psi, placed in an oil bath and heated at 70 ^{o}C for 16 h. The reactor was allowed to cool to room temperature and the CO gas was vented off carefully. The reaction mixture was evaporated under reduced pressure and the residue was purified by flash column chromatography to afford the pure β-lactone.

(Conversions in Table 1 and 2 were determined by ^{1}H NMR spectroscopy of the aforementioned residue before purification by flash column chromatography. The β-lactone product was the only observable compound for conversions of ≥98%. In all other cases (Table 1 and Table 2, entries 1, 15 and 16), the starting *meso*-epoxide and the β-lactone product were the only detectable components.)

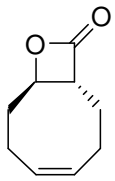
***trans*-3,4-Bis(benzyloxymethyl)oxetan-2-one[10]**



According to the GP II using catalyst **1** (32 mg, 0.05 mmol), $Co_2(CO)_8$ (17 mg, 0.05 mmol), *cis*-1,4-dibenzyloxy-2,3-epoxybutane (284 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as a white solid (152 mg, 49%). Enantiomeric excess was determined by HPLC analysis (CHIRALPAK IB, *n*-heptane/EtOH, 90:10, 1.0 mL/min, 210 nm): t_r (minor) = 9.8 min; t_r (major) = 14.4 min: 19% *ee*; $[\alpha]_D^{20}$ = +9.1 (*c* 1.0, $CHCl_3$).

According to the GP II using catalyst **3** (6.2 mg, 0.005 mmol), $Co_2(CO)_8$ (2.6 mg, 0.0075 mmol), *cis*-1,4-dibenzyloxy-2,3-epoxybutane (284 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as a white solid (174 mg, 56%). Enantiomeric excess was determined by HPLC analysis (CHIRALPAK IB, *n*-heptane/EtOH, 90:10, 1.0 mL/min, 210 nm): t_r (minor) = 9.7 min; t_r (major) = 13.4 min: 13% *ee*; $[\alpha]_D^{20}$ = +3.9 (*c* 1.0, $CHCl_3$).

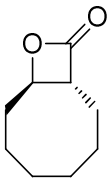
***trans*-9-Oxabicyclo[6.2.0]dec-4-en-10-one**[10]



According to GP II using catalyst **1** (32 mg, 0.05 mmol), $Co_2(CO)_8$ (17 mg, 0.05 mmol), epoxide (124 mg, 1.0 mmol), and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as colourless oil (126 mg, 82%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 97:3, 1.0 mL/min, 210 nm): t_r (minor) = 14.2 min; t_r (major) = 15.5 min: 6% ee; $[\alpha]_D^{20}$ = +4.1 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = +4.6 (*c* 1.0, $CHCl_3$).

According to GP II using catalyst **3** (6.1 mg, 0.05 mmol), $Co_2(CO)_8$ (2.5 mg, 0.075 mmol), epoxide (124 mg, 1.0 mmol) and DME (1.5 mL) were used in the reaction. Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as colourless oil (130 mg, 85%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 90:10, 1.0 mL/min, 210 nm); t_r (minor) = 13.7 min; t_r (major) = 15.3 min: 2% ee. $[\alpha]_D^{20}$ = +2.4 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = +2.8 (*c* 1.0, $CHCl_3$).

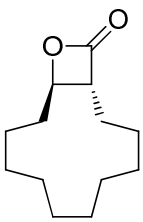
***trans*-9-oxabicyclo[6.2.0]decan-10-one**[10]



According to GP II using catalyst **1** (32 mg, 0.05 mmol), $Co_2(CO)_8$ (17 mg, 0.05 mmol), epoxide (126 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as colourless oil (127 mg, 83%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 90:10, 1.0 mL/min, 210 nm): t_r (minor) = 14.8 min; t_r (major) = 15.8 min: 11% *ee*; $[\alpha]_D^{20}$ = +5.4 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = +5.7 (*c* 1.0, $CHCl_3$).

According to GP II using catalyst **3** (6.1 mg, 0.005 mmol), $Co_2(CO)_8$ (2.5 mg, 0.0075 mmol), epoxide (126 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as colourless oil (145 mg, 94%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 90:10, 1.0 mL/min, 210 nm): t_r (major) = 14.6 min; t_r (minor) = 15.7 min: 6% *ee*; $[\alpha]_D^{20}$ = −3.1 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = −3.4 (*c* 1.0, $CHCl_3$).

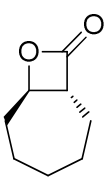
***trans*-13-Oxabicyclo[10.2.0]tetradecan-14-one**[10]



According to the GP II using catalyst **1** (32 mg, 0.05 mmol), $Co_2(CO)_8$ (17 mg, 0.05 mmol), epoxide (182 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as colourless oil (180 mg, 86%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 97:3, 1.0 mL/min, 210 nm): t_r (major) = 10.4 min; t_r (minor) = 11.2 min: 13% *ee*; $[\alpha]_D^{20}$ = +3.3 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = +3.8 (*c* 1.0, $CHCl_3$).

According to the GP II using catalyst **3** (6.1 mg, 0.005 mmol), $Co_2(CO)_8$ (2.5 mg, 0.0075 mmol), epoxide (182 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as colourless oil (149 mg, 71%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 90:10, 1.0 mL/min, 210 nm): t_r (major) = 10.5 min; t_r (minor) = 11.2 min: 11% *ee*; $[\alpha]_D^{20}$ = +2.8 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = +3.1 (*c* 1.0, $CHCl_3$).

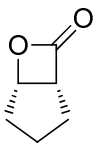
***trans*-8-oxabicyclo[5.2.0]nonan-9-one**[10]



According to GP II using catalyst **1** (32 mg, 0.05 mmol), $Co_2(CO)_8$ (17 mg, 0.05 mmol), epoxide (112 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as colourless oil (127 mg, 91%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 95:5, 1.0 mL/min, 210 nm): t_r (major) = 10.1 min; t_r (minor) = 10.9 min: 4% *ee*; $[\alpha]_D^{20}$ = −3.8 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = −3.1 (*c* 1.0, $CHCl_3$).

According to the GP II using catalyst **3** (6.1 mg, 0.005 mmol), $Co_2(CO)_8$ (2.5 mg, 0.0075 mmol), epoxide (112 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as colourless oil (114 mg, 81%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 95:5, 1.0 mL/min, 210 nm): t_r (major) = 10.0 min; t_r (minor) = 10.7 min: 16% *ee*; $[\alpha]_D^{20}$ = −5.7 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = −6.1 (*c* 1.0, $CHCl_3$).

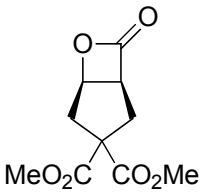
(-)-(1*S*,2*R*)-6-Oxabicyclo[3.2.0]heptan-7-one[10, 13]



According to the GP II using catalyst **1** (32 mg, 0.05 mmol), $Co_2(CO)_8$ (17 mg, 0.05 mmol) epoxide (84 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as colourless oil (100 mg, 89%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 98:2, 1.0 mL/min, 210 nm): t_r (major) = 48.2 min; t_r (minor) = 51.9 min: 40% *ee*; $[\alpha]_D^{20}$ = −6.7 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = −7.2 (*c* 1.0, $CHCl_3$).

According to the GP II using catalyst **3** (6.0 mg, 0.005 mmol), $Co_2(CO)_8$ (2.5 mg, 0.0075 mmol), epoxide (84 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 90:10) gave the title compound as colourless oil (93 mg, 83%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 98:2, 1.0 mL/min, 210 nm): t_r (major) = 48.3 min; t_r (minor) = 51.8: 33% *ee*; $[\alpha]_D^{20}$ = −4.3 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = −4.8 (*c* 1.0, $CHCl_3$).

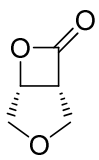
(+)-(1*R*,2*S*)-Dimethyl-7-oxo-6-oxabicyclo[3.2.0]heptane-3,3-dicarboxylate[10, 14]



According to the GP II using catalyst **1** (32 mg, 0.05 mmol), $Co_2(CO)_8$ (17 mg, 0.05 mmol), epoxide (200 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/EtOAc, 50:50) gave the title compound as colourless oil (177 mg, 78%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 93:7, 0.5 mL/min, 210 nm): t_r (major) = 48.2 min; t_r (minor) = 50.4 min: 45% *ee*; $[\alpha]_D^{20}$ = +14.7 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = +15.1 (*c* 1.0, $CHCl_3$).

According to the GP II using catalyst **3** (24 mg, 0.02 mmol), $Co_2(CO)_8$ (11 mg, 0.03 mmol), epoxide (200 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/EtOAc, 50:50) gave the title compound as colourless oil (189 mg, 83%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 93:7, 0.5 mL/min, 210 nm): t_r (major) = 47.0 min; t_r (minor) = 49.5 min: 41% *ee*; $[\alpha]_D^{20}$ = +12.9 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = +13.3 (*c* 1.0, $CHCl_3$).

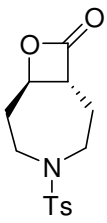
(-)-(1*R*,2*S*)-3,6-dioxabicyclo[3.2.0]heptan-7-one[10,13]



According to the GP II using catalyst **1** (32 mg, 0.05 mmol), $Co_2(CO)_8$ (17 mg, 0.05 mmol), epoxide (86 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 50:50) gave the title compound as colourless oil (106 mg, 93%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 90:10, 1 mL/min, 210 nm): t_r (minor) = 15.1; t_r (major) = 16.4 min: 56% *ee*; $[\alpha]_D^{20}$ = −5.8 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = −6.2 (*c* 1.0, $CHCl_3$).

According to the GP II using catalyst **3** (6.1 mg, 0.005 mmol), $Co_2(CO)_8$ (2.5 mg, 0.0075 mmol), epoxide (86 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/Et_2O, 50:50) gave the title compound as colourless oil (98 mg, 86%). Enantiomeric excess was determined by HPLC analysis after derivatisation into the β-hydroxy benzylamide (CHIRALPAK IB, *n*-heptane/EtOH, 90:10, 1 mL/min, 210 nm): t_r (minor) = 15.3; t_r (major) = 16.4 min: 33% *ee*; $[\alpha]_D^{20}$ = −4.1 (*c* 1.0, $CHCl_3$); $[\alpha]_D^{20}$ (benzylamide) = −4.5 (*c* 1.0, $CHCl_3$).

***trans*-4-Tosyl-8-oxa-4-azabicyclo[5.2.0]nonan-9-one**[10]



According to the GP II using catalyst **1** (32 mg, 0.05 mmol), $Co_2(CO)_8$ (17 mg, 0.05 mmol), epoxide (267 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/EtOAc, 90:10) gave the title compound as a white solid (138 mg, 47%). Enantiomeric excess was determined by HPLC analysis (CHIRALPAK IB, *n*-heptane/EtOH, 90:10, 0.5 mL/min, 230 nm): t_r (major) = 50.9 min; t_r (minor) = 53.2 min: 31% *ee*; $[\alpha]_D^{20}$ = +8.1 (*c* 1.0, $CHCl_3$).

According to the GP II using catalyst **3** (24 mg, 0.02 mmol), $Co_2(CO)_8$ (10 mg, 0.03 mmol), epoxide (267 mg, 1.0 mmol) and DME (1.5 mL). Purification by flash column chromatography (SiO_2; pentane/EtOAc, 90:10) gave the title compound as a white solid (165 mg, 56%). Enantiomeric excess was determined by HPLC analysis (CHIRALPAK IB, *n*-heptane/EtOH, 90:10, 0.5 mL/min, 230 nm): t_r (minor) = 52.0 min; t_r (major) = 54.5 min: 31% *ee*; $[\alpha]_D^{20}$ = −6.7 (*c* 1.0, $CHCl_3$).

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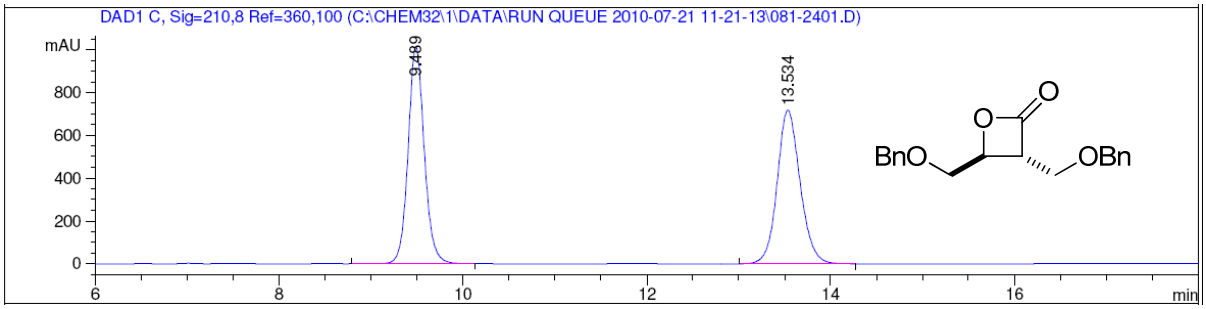
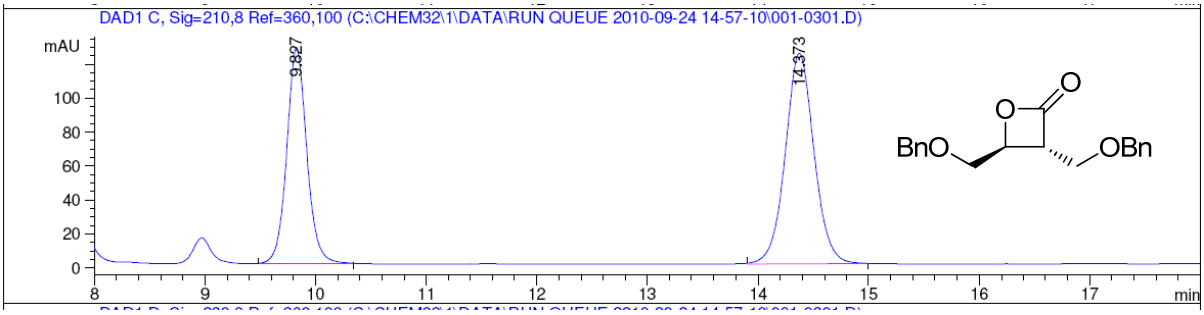
(±)-*trans*-3,4-Bis((benzyloxy)methyl)oxetan-2-one (racemic)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

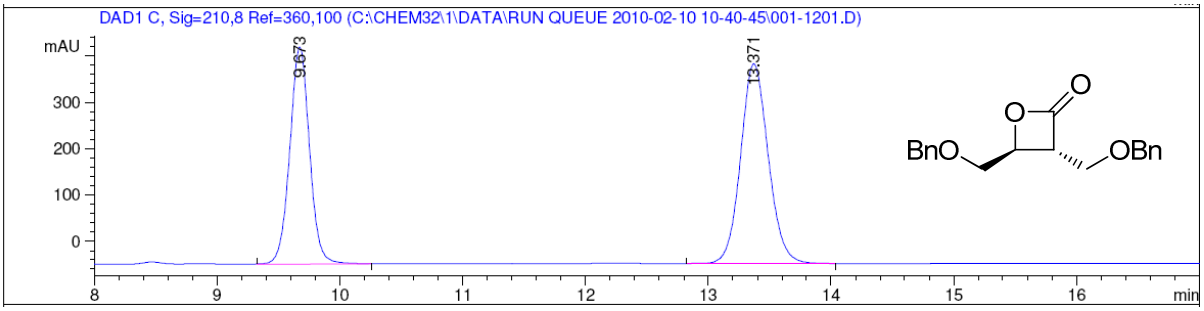
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.489	BB	0.1870	1.24438e4	1015.25397	49.9142
2	13.534	BB	0.2646	1.24865e4	718.91705	50.0858
Totals :				2.49303e4	1734.17102	

(+)-*trans*-3,4-Bis((benzyloxy)methyl)oxetan-2-one with catalyst 1 (19% *ee*)



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.827	BB	0.1838	28.32489	2.36381	40.7463
2	14.373	BB	0.2732	41.19036	2.34132	59.2537
Totals :				69.51525	4.70512	

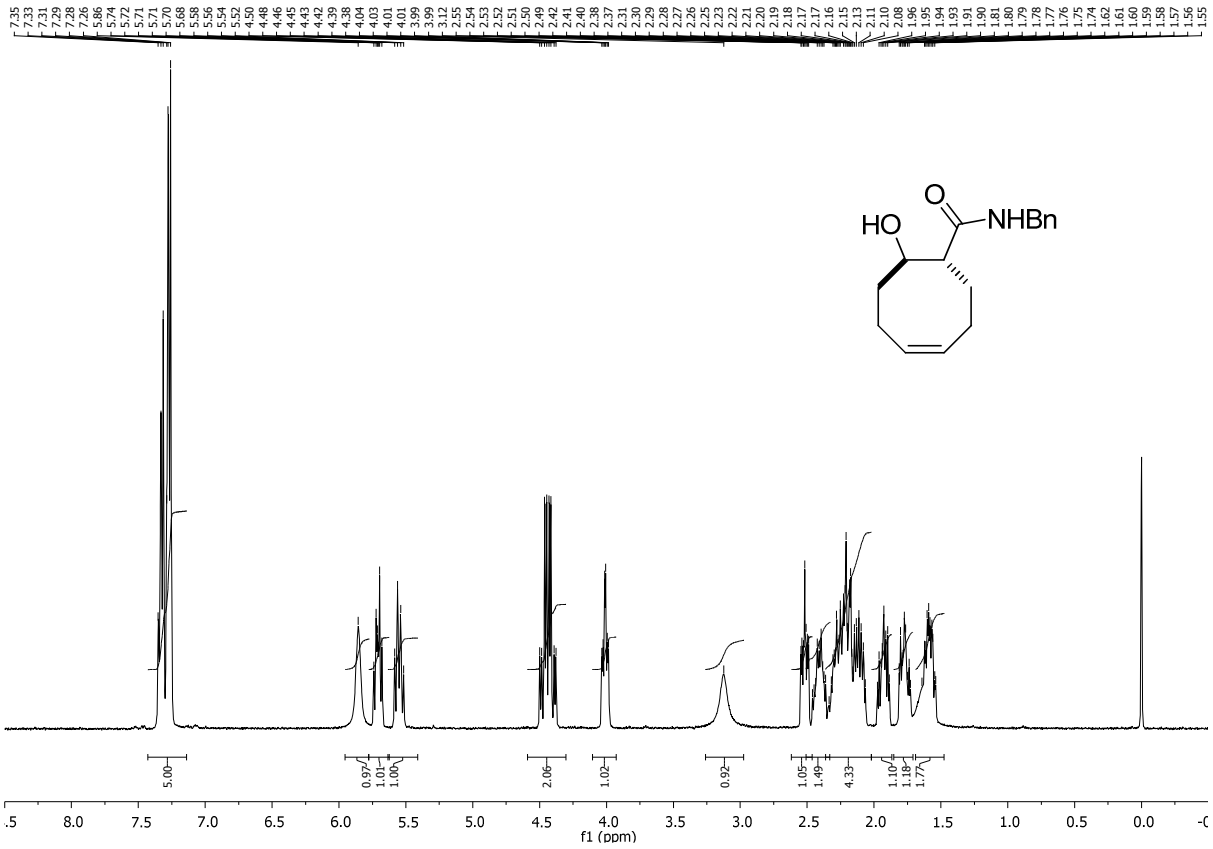
(+)-*trans*-3,4-Bis((benzyloxy)methyl)oxetan-2-one with catalyst 3 (13% *ee*)

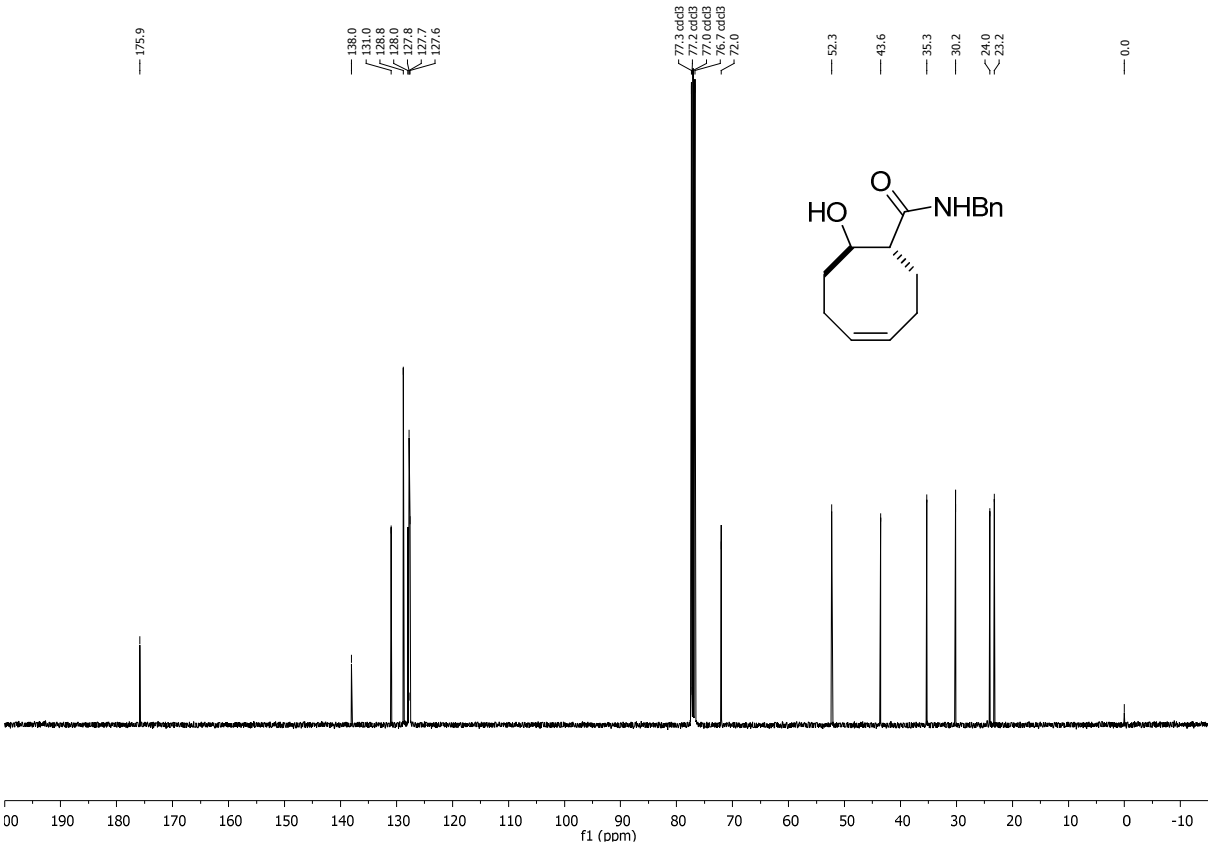


Signal 2: DAD1 C, Sig=210,8 Ref=360,100

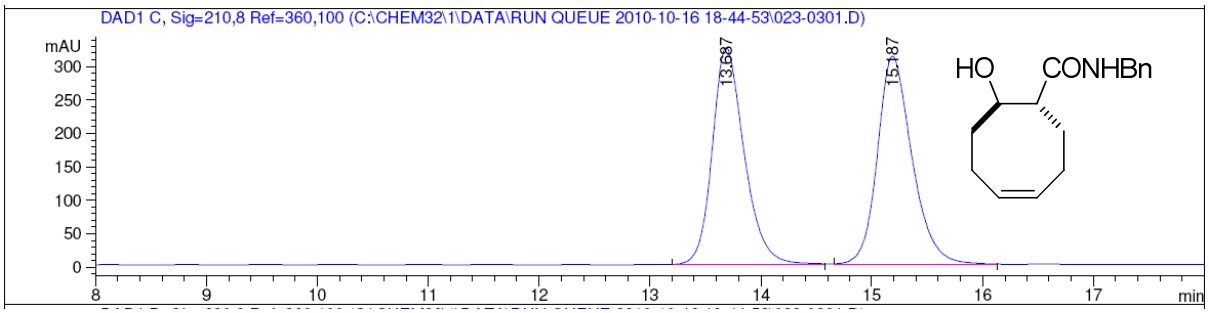
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.673	BB	0.1724	5236.38818	468.58530	43.5183
2	13.371	BB	0.2416	6796.21875	431.76999	56.4817
Totals :				1.20326e4	900.35529	

(±)-*trans*-*N*-Benzyl-8-hydroxycyclooct-4-enecarboxamide





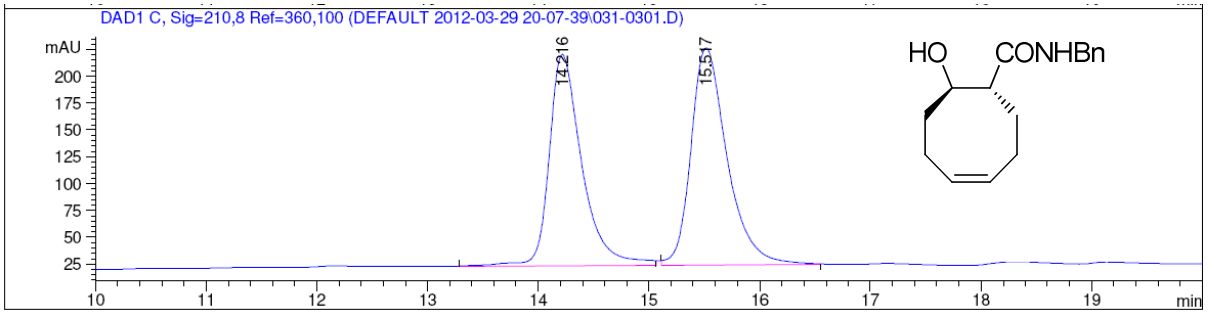
(±)-*trans*-*N*-Benzyl-8-hydroxycyclooct-4-enecarboxamide (racemic)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.687	BB	0.2976	86.84924	4.49182	49.2388
2	15.187	BB	0.3132	89.53459	4.29510	50.7612
Totals :				176.38383	8.78692	

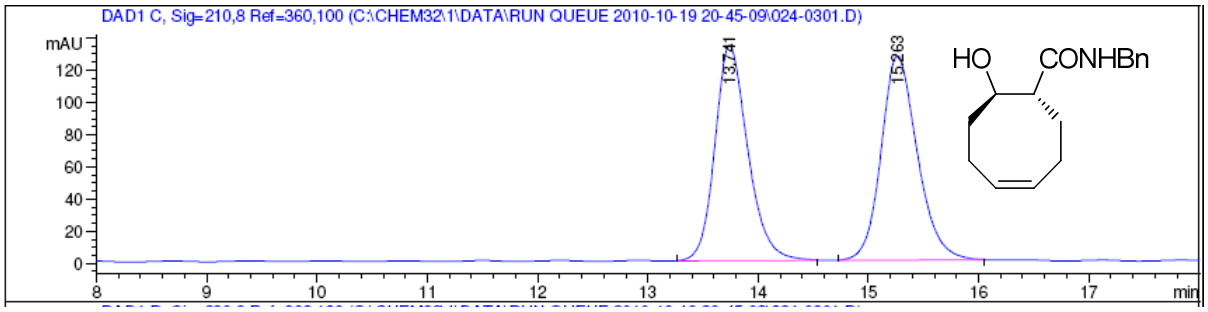
(+)-*trans*-*N*-Benzyl-8-hydroxycyclooct-4-enecarboxamide with catalyst 1 (6% *ee*)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.216	BB	0.3086	4065.42773	197.12209	46.9809
2	15.517	BB	0.3384	4587.92822	202.43925	53.0191
Totals :				8653.35596	399.56134	

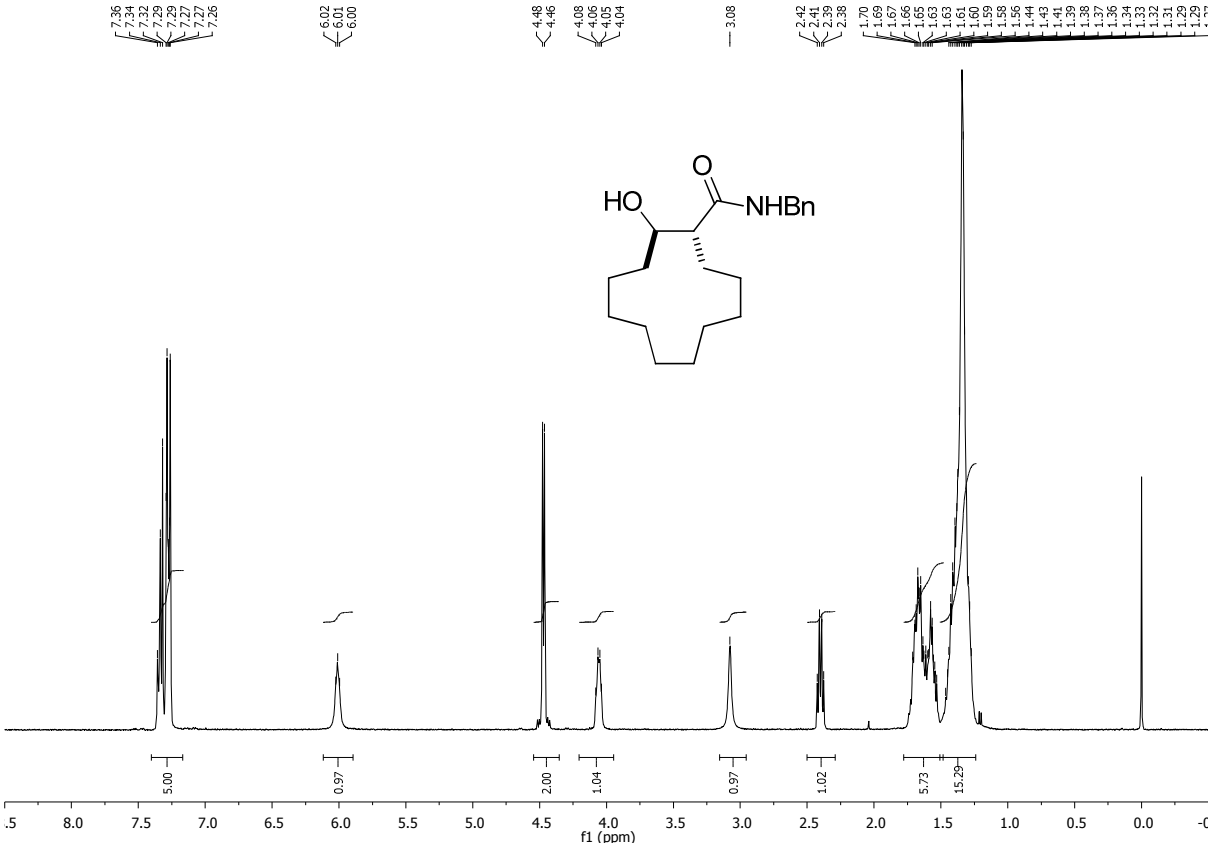
***(+)-trans-N*-Benzyl-8-hydroxycyclooct-4-enecarboxamide with catalyst 3 (2% *ee*)**

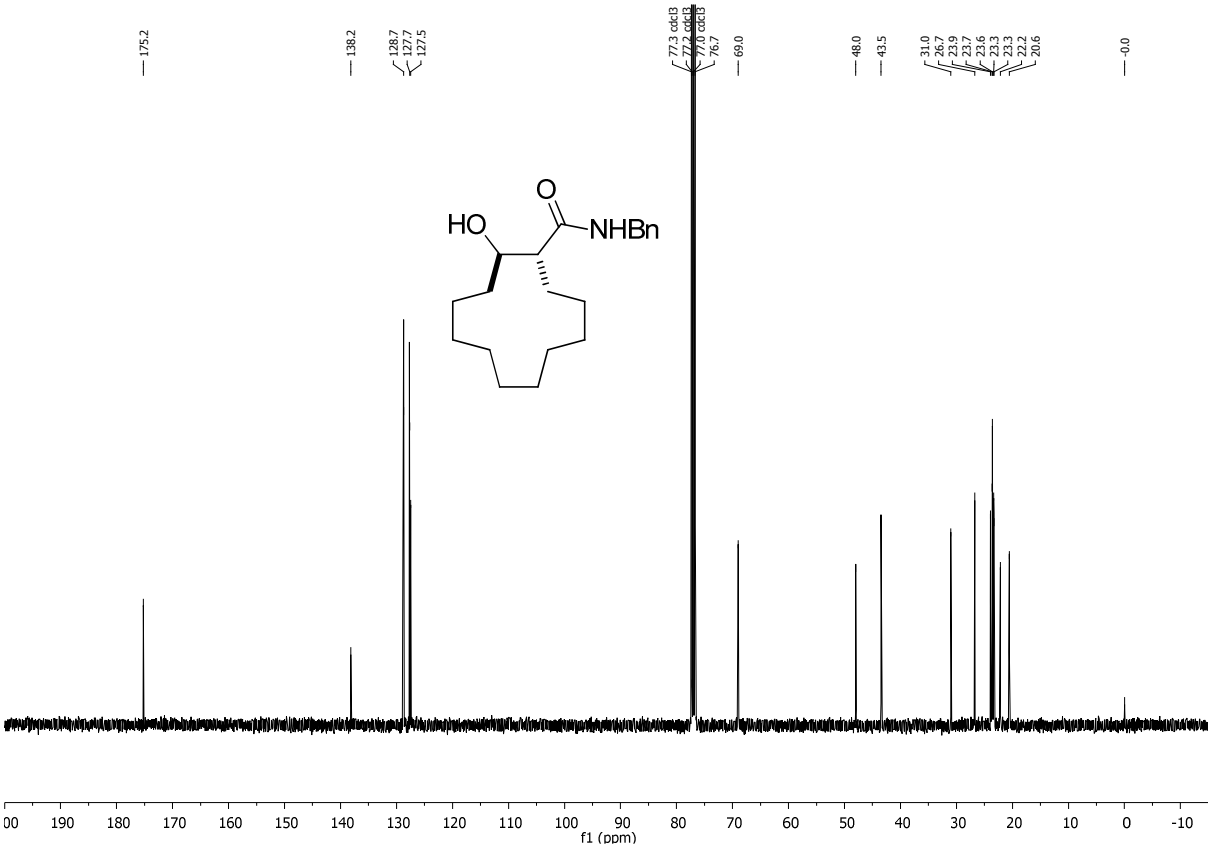


Signal 2: DAD1 C, Sig=210,8 Ref=360,100

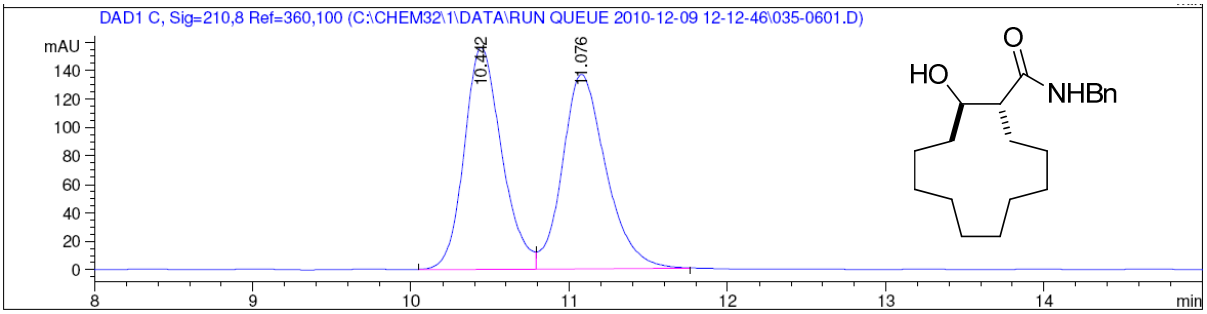
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.741	BB	0.3159	2770.30151	133.59807	48.8746
2	15.263	BB	0.3465	2897.87769	126.84022	51.1254
Totals :				5668.17920	260.43829	

trans-*N*-Benzyl-2-hydroxycyclododecanecarboxamide





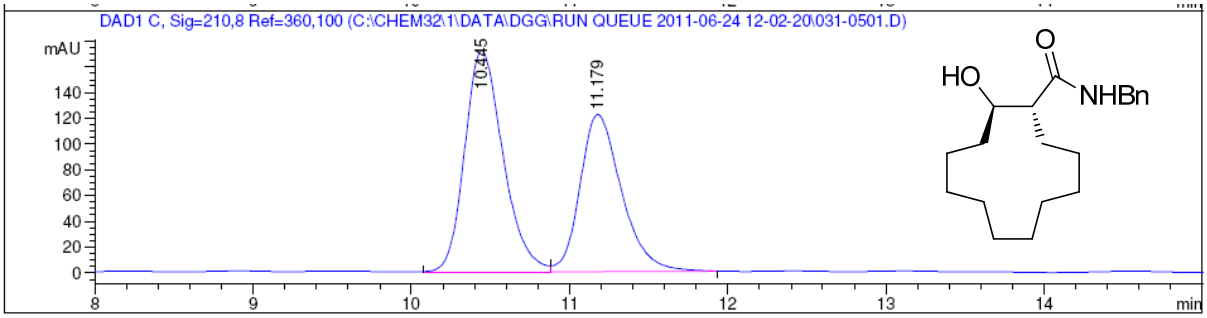
(±)-*trans*-*N*-Benzyl-2-hydroxycyclododecanecarboxamide (racemic)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.442	BV	0.2524	2569.92065	155.86620	49.5552
2	11.076	VB	0.2893	2616.05762	136.68903	50.4448
Totals :				5185.97827	292.55522	

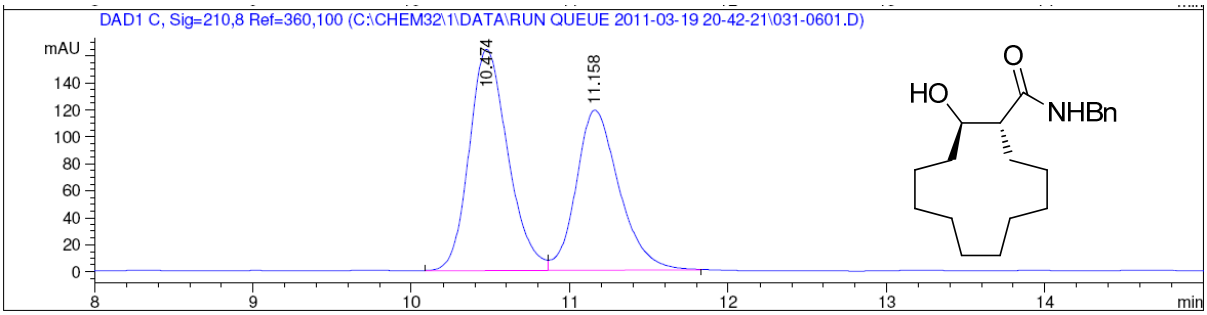
(+)-*trans*-*N*-Benzyl-2-hydroxycyclododecanecarboxamide with catalyst 1 (13% *ee*)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.445	BV	0.2581	2891.17065	172.00726	56.4455
2	11.179	VB	0.2772	2230.88550	122.07956	43.5545
Totals :				5122.05615	294.08682	

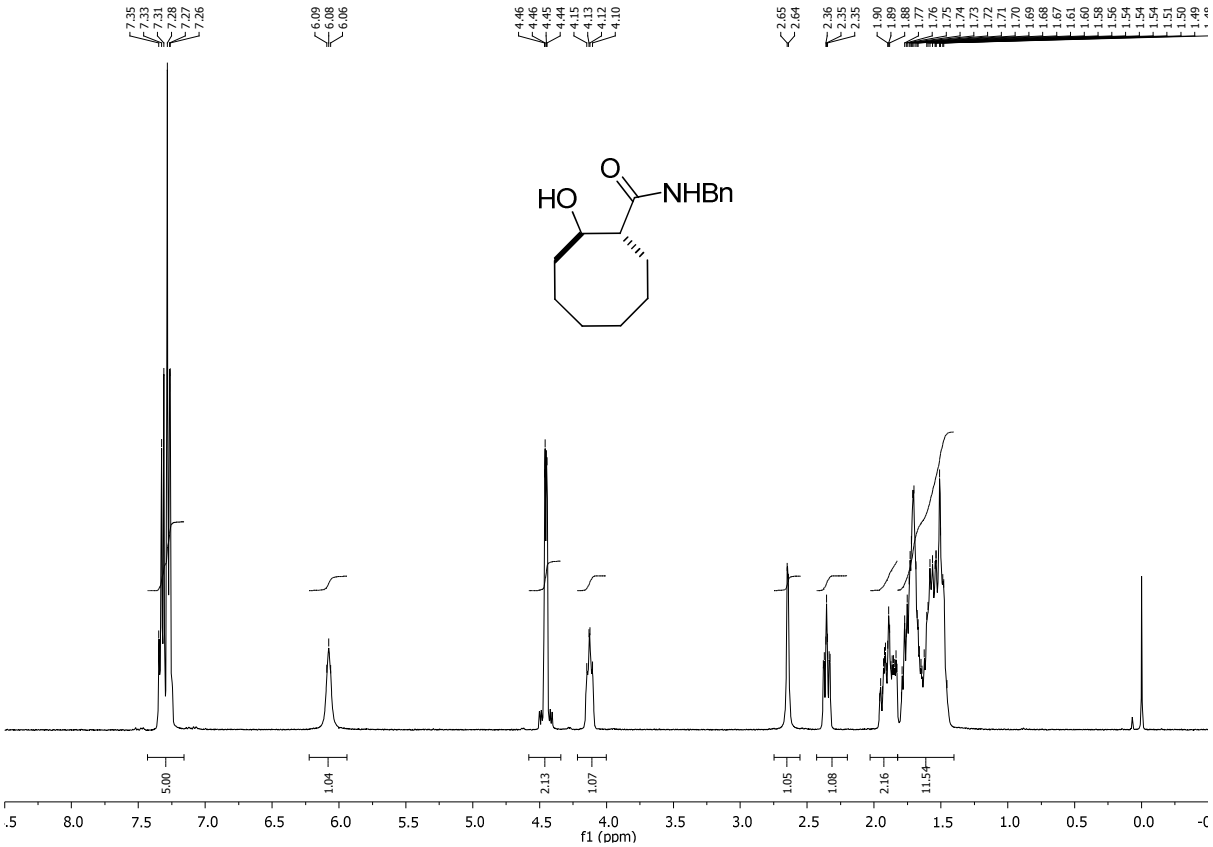
(+)-*trans*-*N*-Benzyl-2-hydroxycyclododecanecarboxamide with catalyst 3 (11% *ee*)

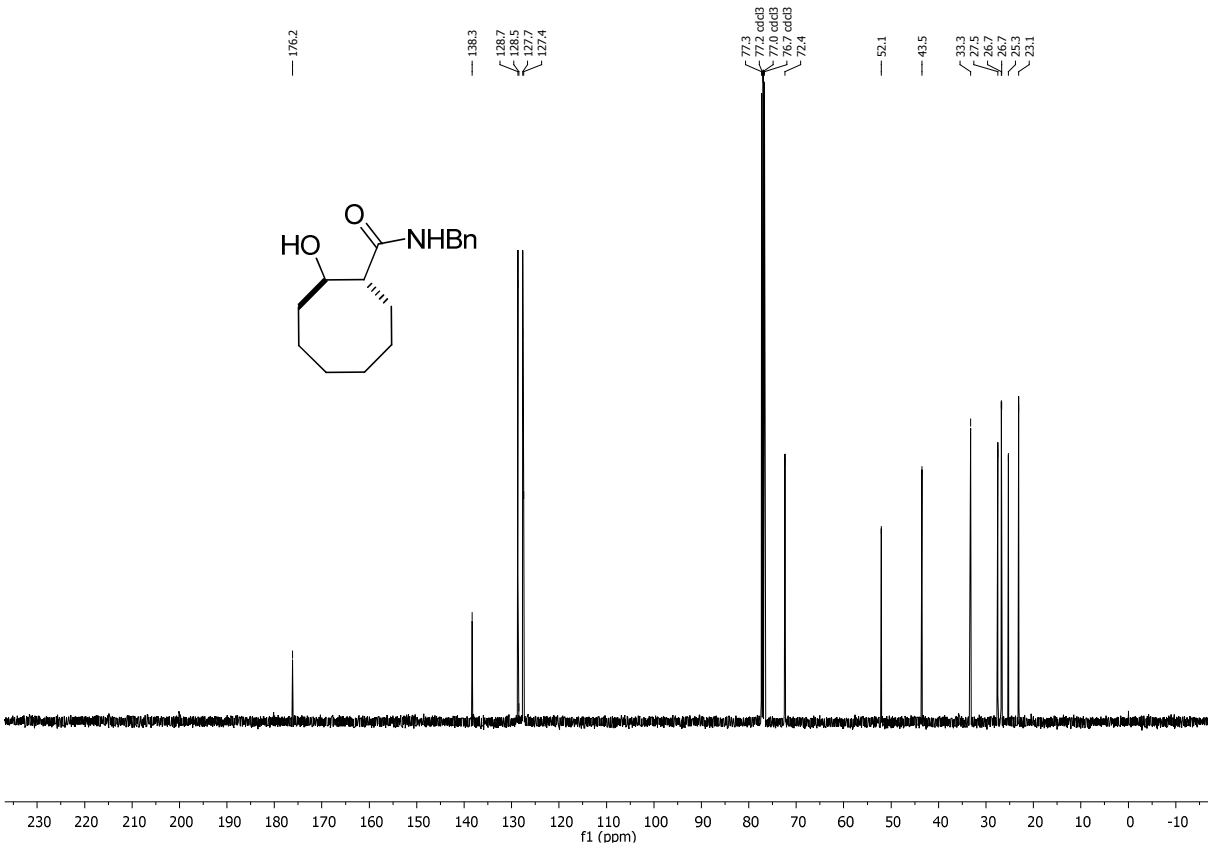


Signal 2: DAD1 C, Sig=210,8 Ref=360,100

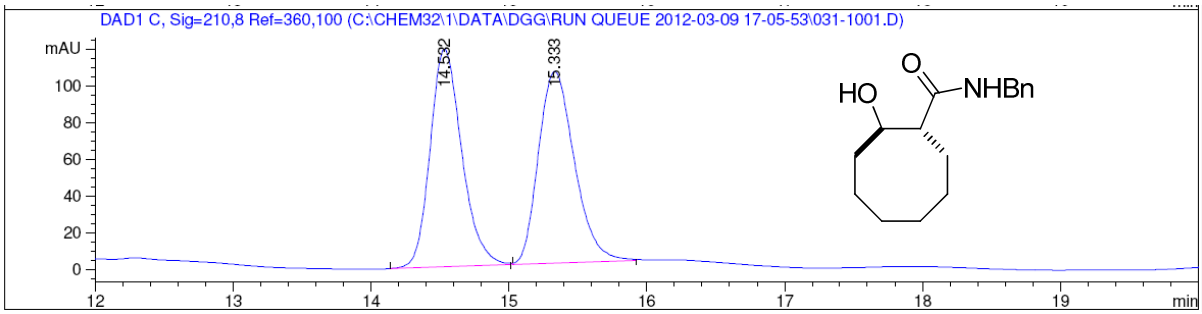
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.474	BV	0.2602	2786.83691	164.07961	55.4109
2	11.158	VB	0.2862	2242.56543	118.80946	44.5891
Totals :				5029.40234	282.88906	

trans-N-Benzyl-2-hydroxycyclooctanecarboxamide





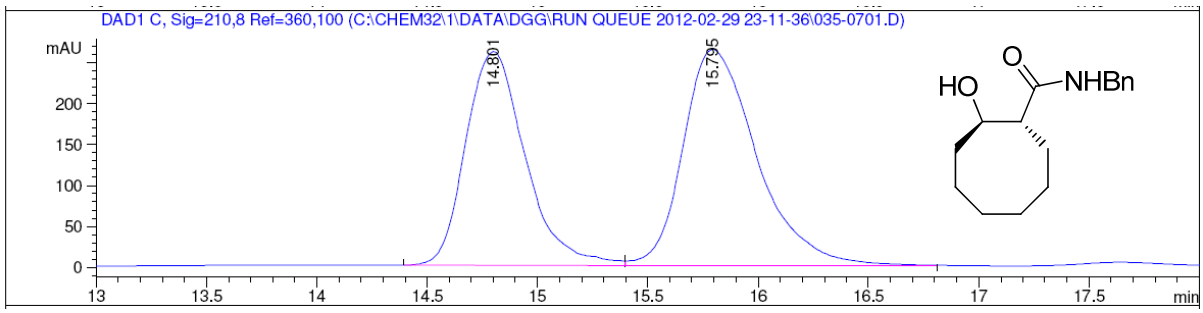
(±)-*trans*-*N*-Benzyl-2-hydroxycyclooctanecarboxamide (racemic)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.532	BB	0.2461	1893.20935	118.67139	49.9813
2	15.333	BB	0.2793	1894.62891	104.61658	50.0187
Totals :				3787.83826	223.28796	

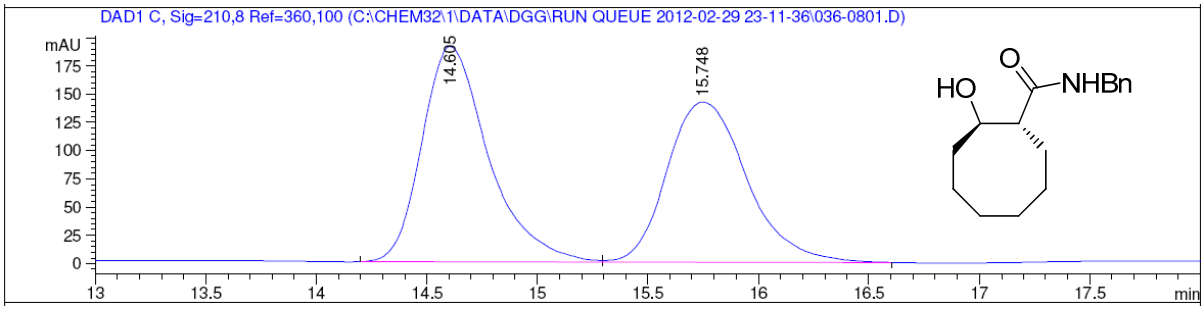
(+)-*trans*-*N*-Benzyl-2-hydroxycyclooctanecarboxamide with catalyst 1 (11% *ee*)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.801	BV	0.2871	4892.23633	260.49808	44.4637
2	15.795	VB	0.3607	6110.52588	263.30746	55.5363
Totals :				1.10028e4	523.80554	

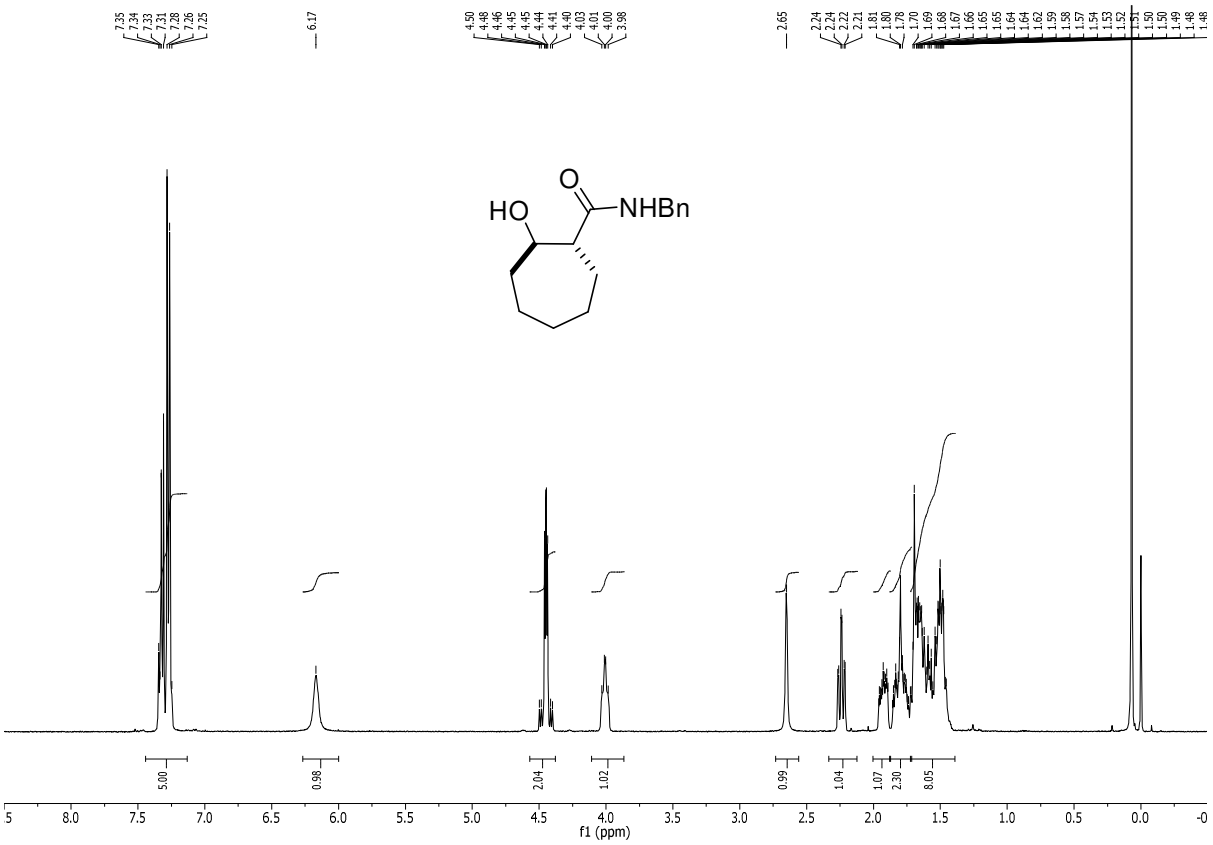
(-)-*trans*-*N*-Benzyl-2-hydroxycyclooctanecarboxamide with catalyst 3 (6% *ee*)

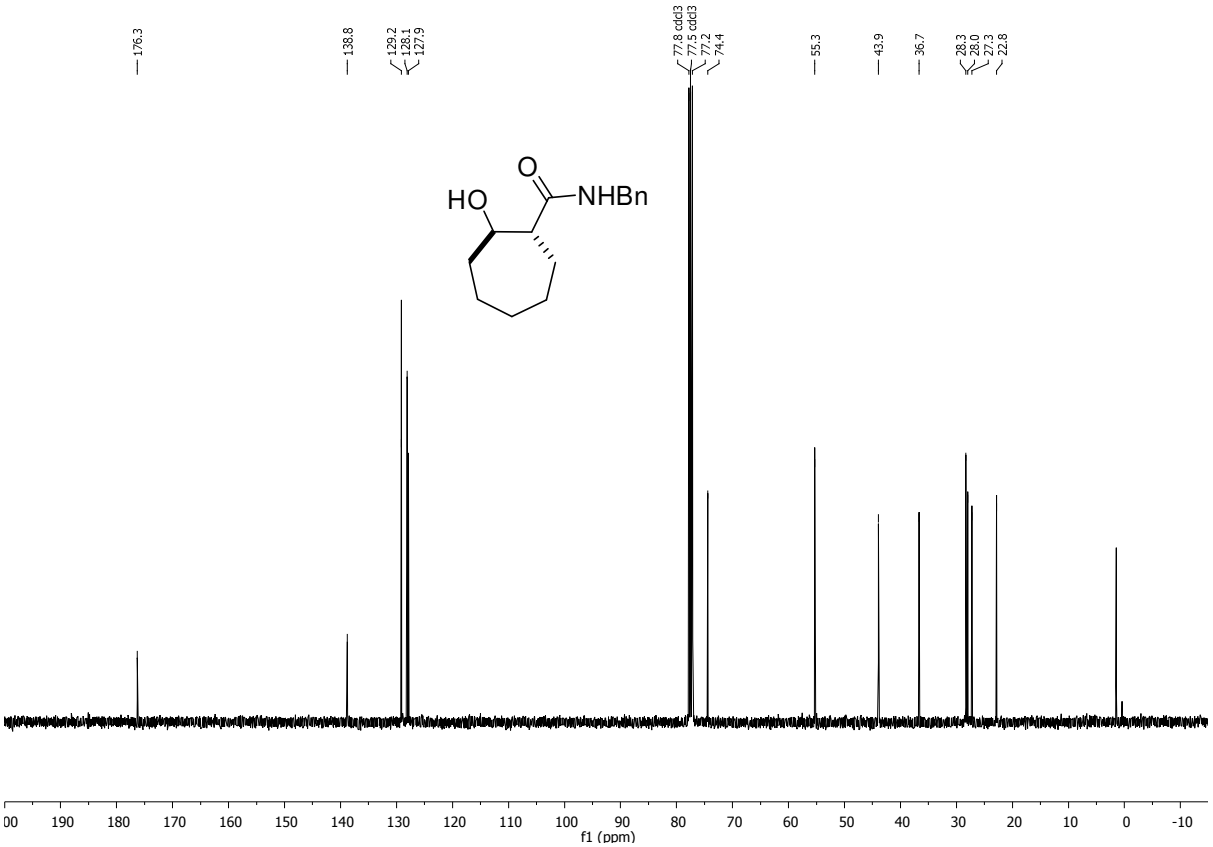


Signal 2: DAD1 C, Sig=210,8 Ref=360,100

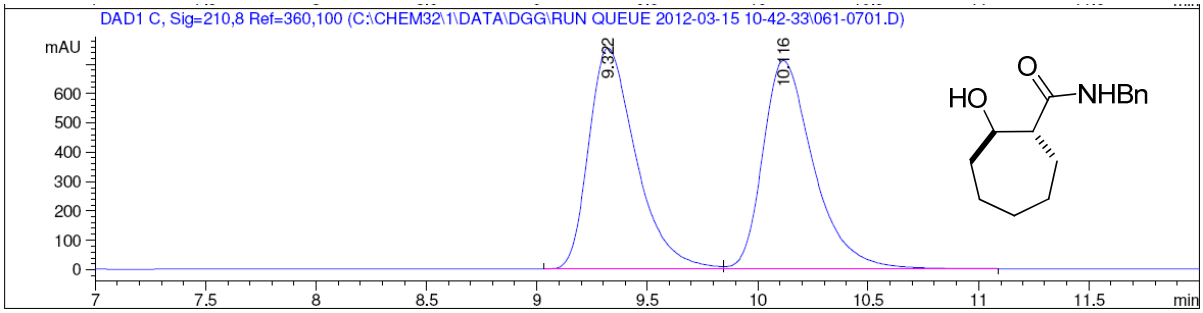
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.605	BV	0.3181	3992.18311	190.82686	52.7507
2	15.748	VB	0.3976	3575.83325	142.08011	47.2493
Totals :				7568.01636	332.90697	

trans-_N_-Benzyl-2-hydroxycycloheptanecarboxamide





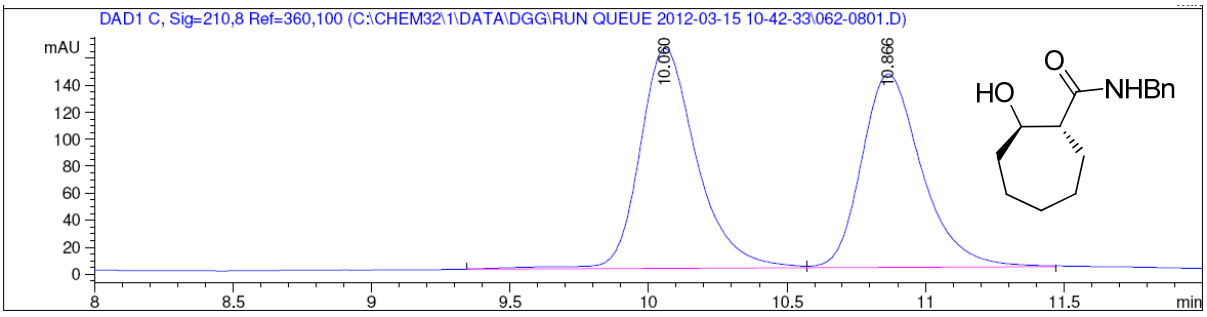
(±)-*N*-Benzyl-2-hydroxycycloheptanecarboxamide (racemic)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.322	BV	0.2296	1.12177e4	753.76172	49.6929
2	10.116	VB	0.2423	1.13563e4	711.02148	50.3071
Totals :				2.25740e4	1464.78320	

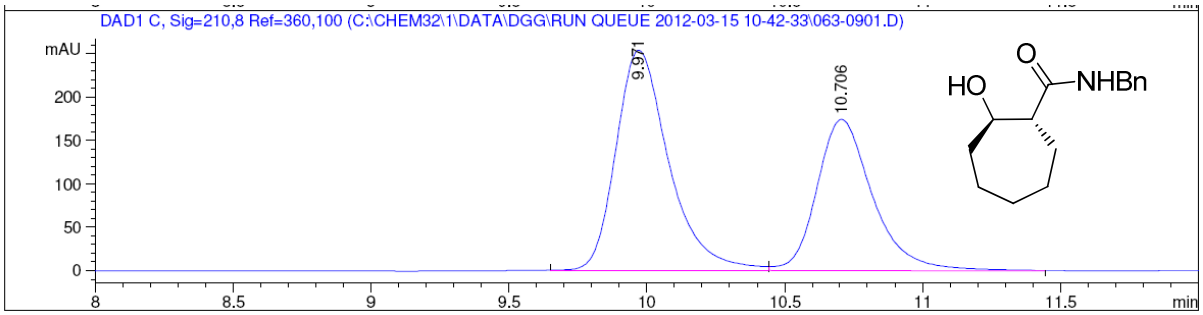
(-)-*N*-Benzyl-2-hydroxycycloheptanecarboxamide with catalyst 1 (4% *ee*)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.060	BV	0.2157	2342.89160	163.00114	52.0488
2	10.866	VB	0.2319	2158.44702	143.17899	47.9512
Totals :				4501.33862	306.18013	

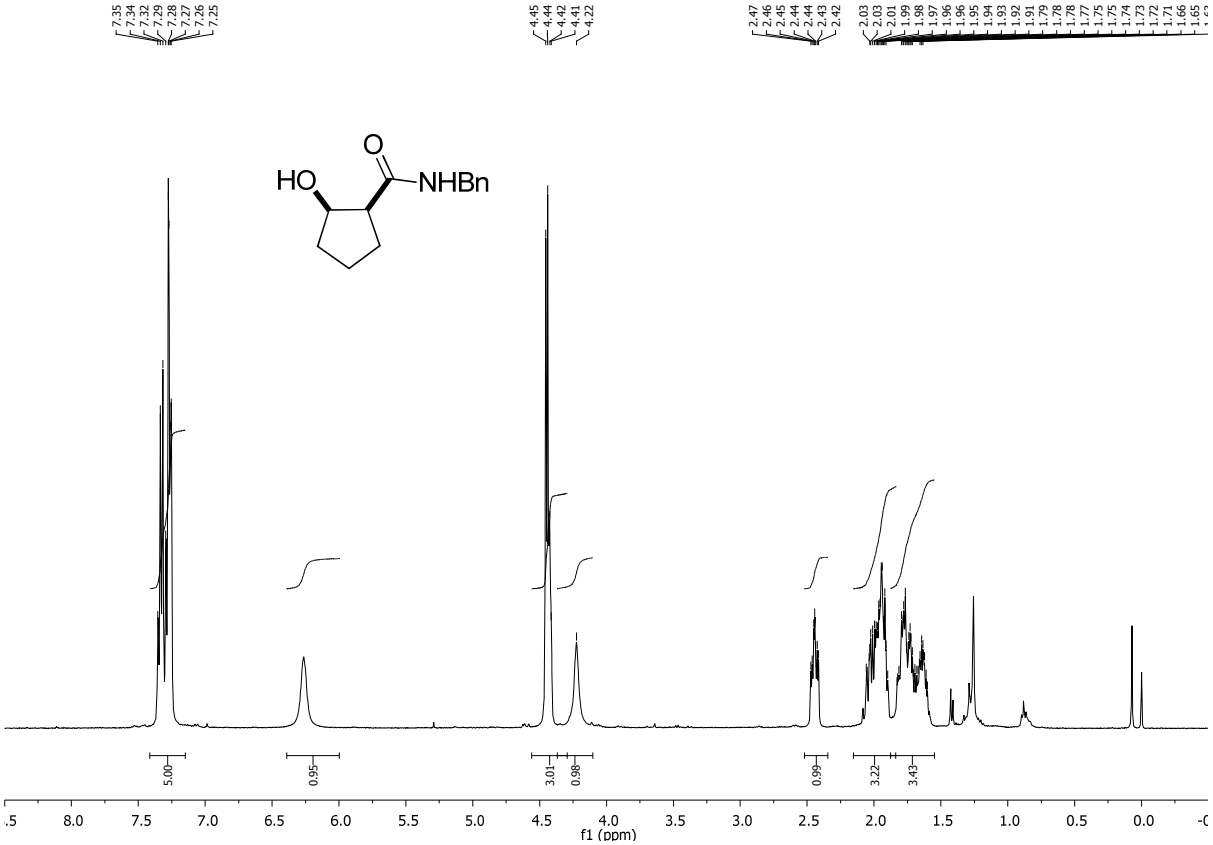
(-)-*N*-Benzyl-2-hydroxycycloheptanecarboxamide with catalyst 3 (16% *ee*)

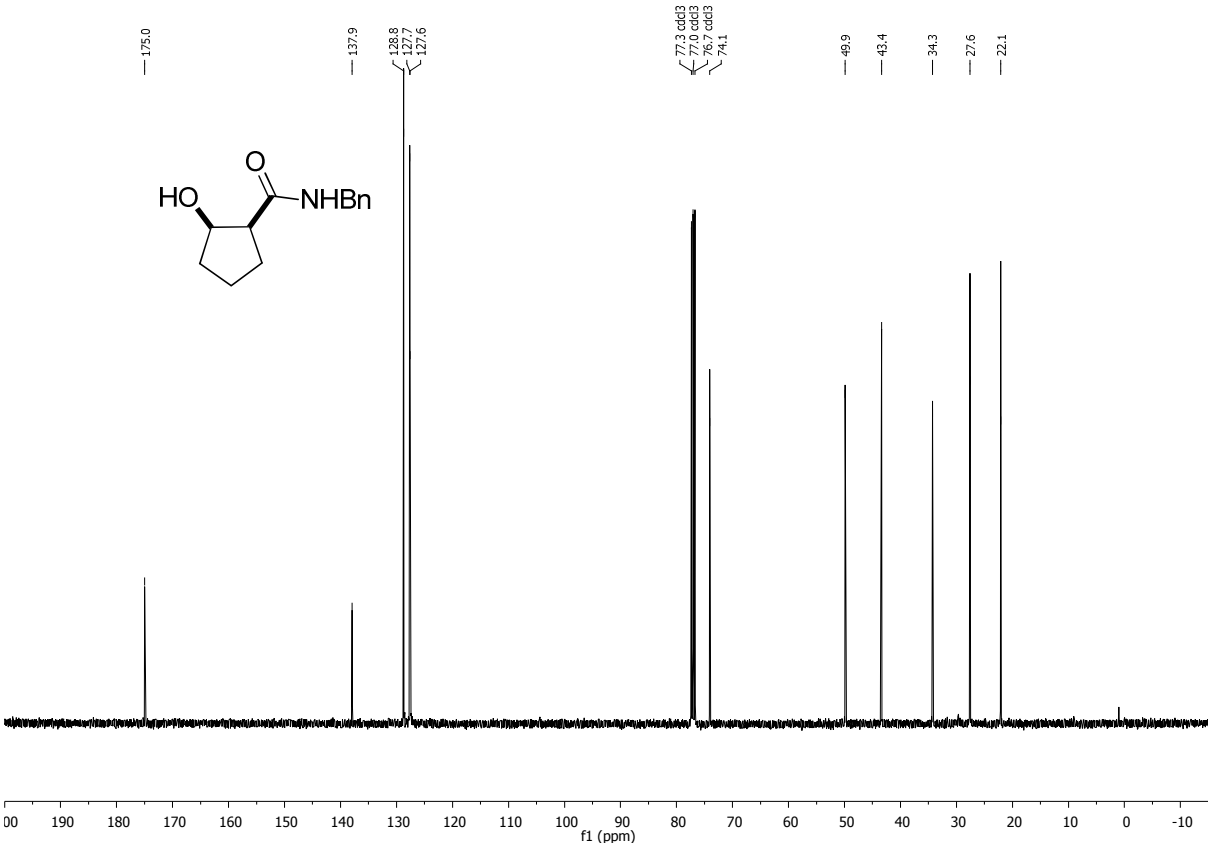


Signal 2: DAD1 C, Sig=210,8 Ref=360,100

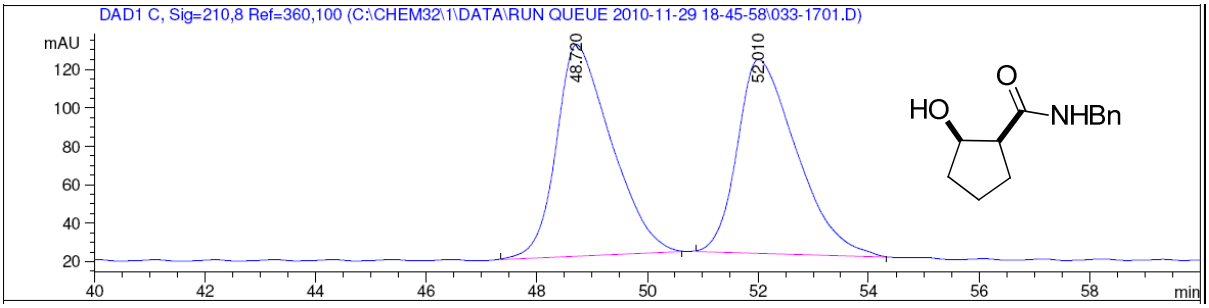
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.971	BV	0.2021	3400.28442	254.16425	57.9633
2	10.706	VB	0.2127	2465.98218	174.63393	42.0367
Totals :				5866.26660	428.79817	

(±)-*cis*-*N*-Benzyl-2-hydroxycyclopentanecarboxamide[12]





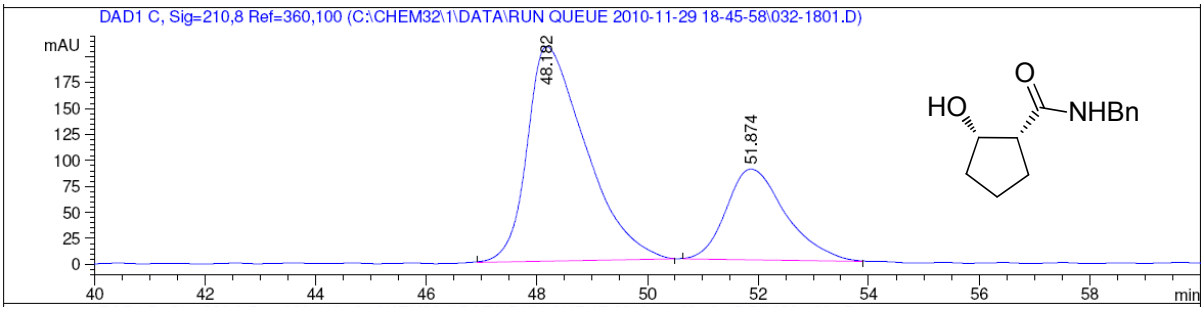
(±)-*cis*-*N*-Benzyl-2-hydroxycyclopentanecarboxamide (racemic)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	48.720	BB	1.0221	7587.07471	110.08331	50.2412
2	52.010	BB	1.1006	7514.23584	100.69859	49.7588
Totals :				1.51013e4	210.78190	

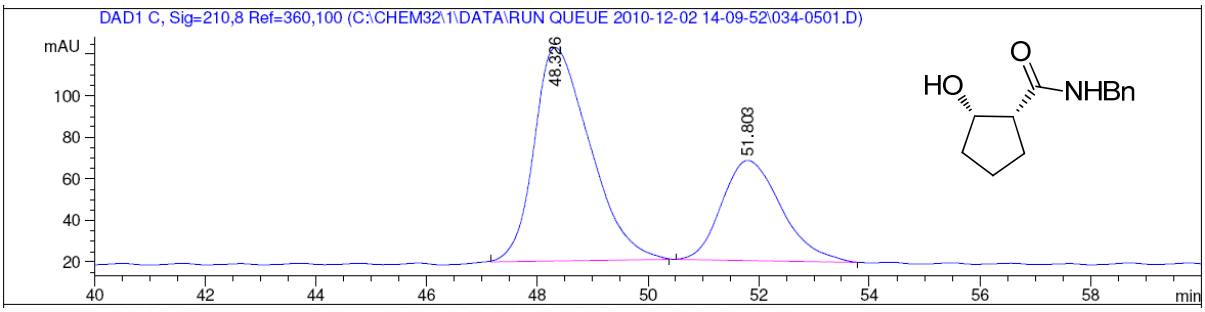
(-)-(1*S*,2*R*)- *N*-Benzyl-2-hydroxycyclopentanecarboxamide with catalyst 1 (40% *ee*)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	48.182	BB	1.0698	1.52087e4	206.36102	70.1076
2	51.874	BB	1.1245	6484.66016	87.10454	29.8924
Totals :				2.16934e4	293.46556	

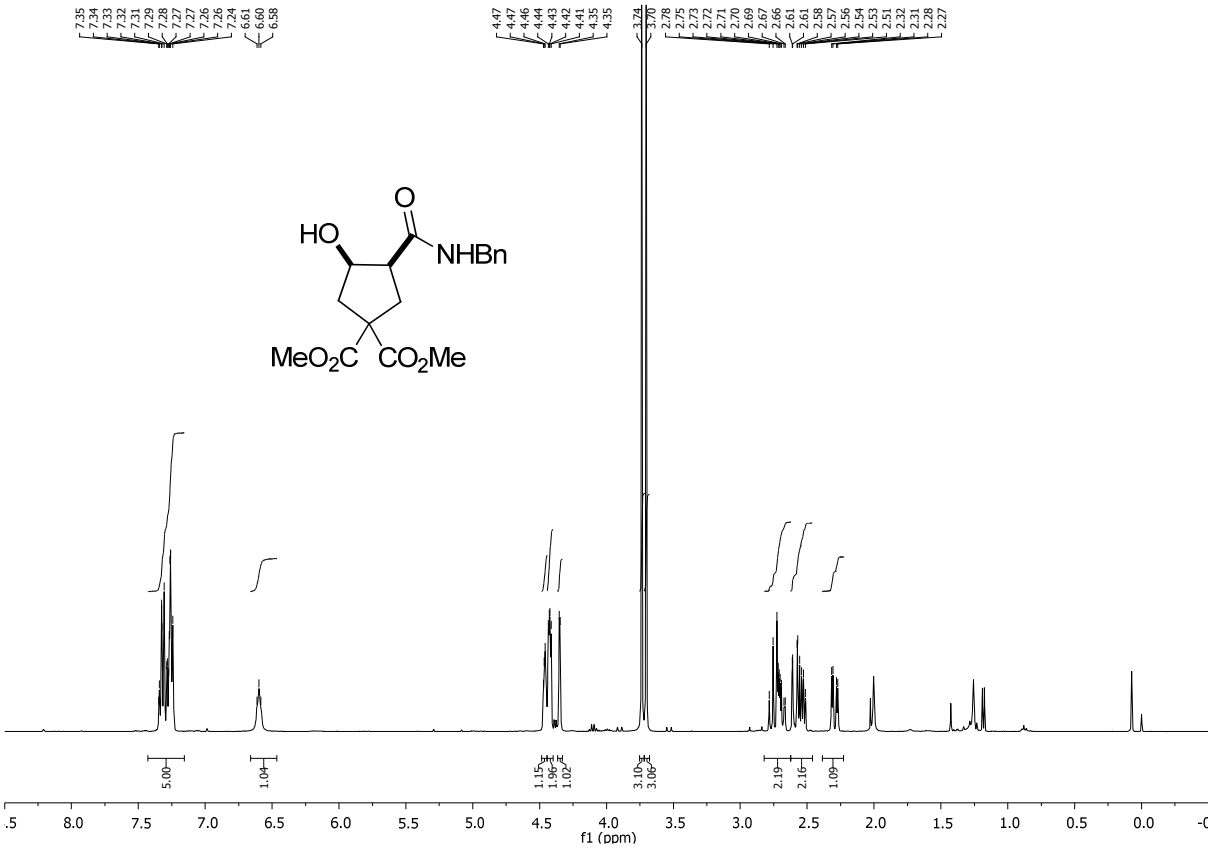
(-)-(1*S*,2*R*)-*N*-Benzyl-2-hydroxycyclopentanecarboxamide with catalyst 3 (33% *ee*)

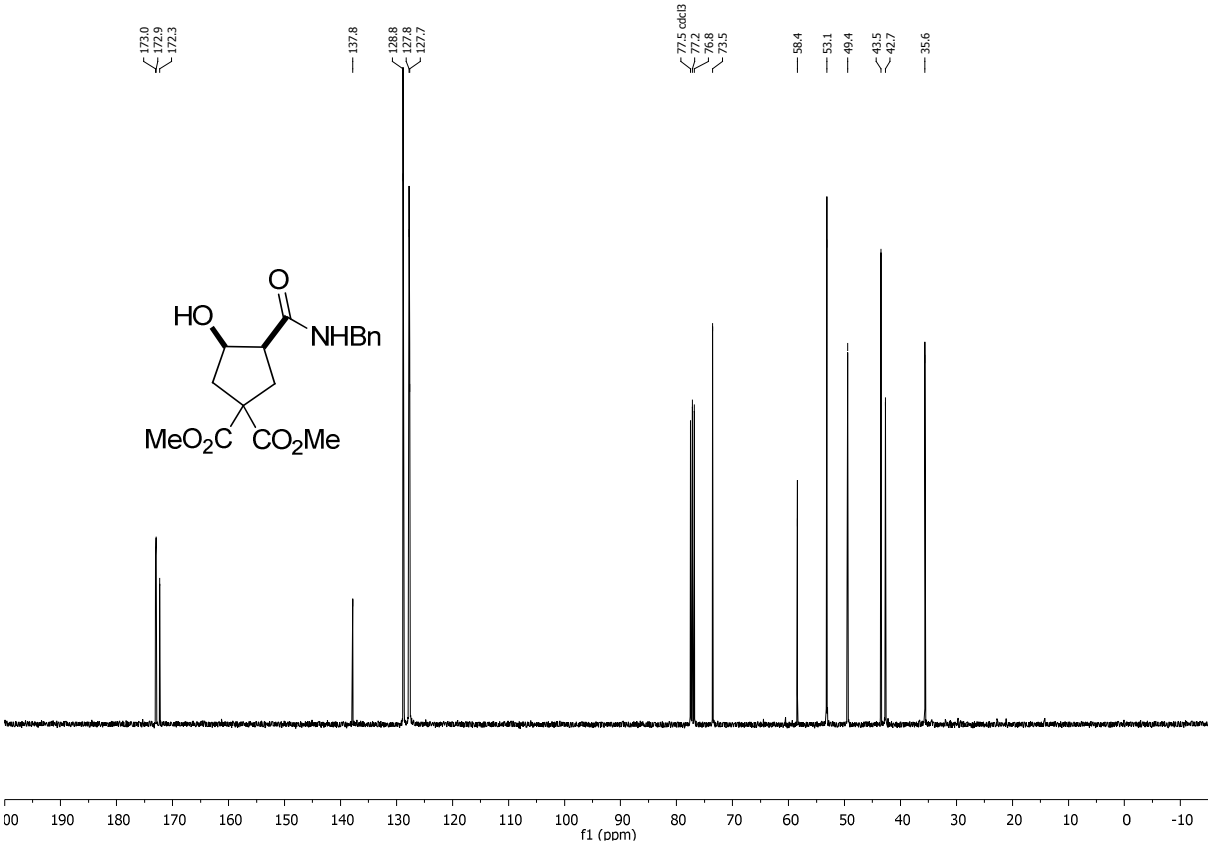


Signal 2: DAD1 C, Sig=210,8 Ref=360,100

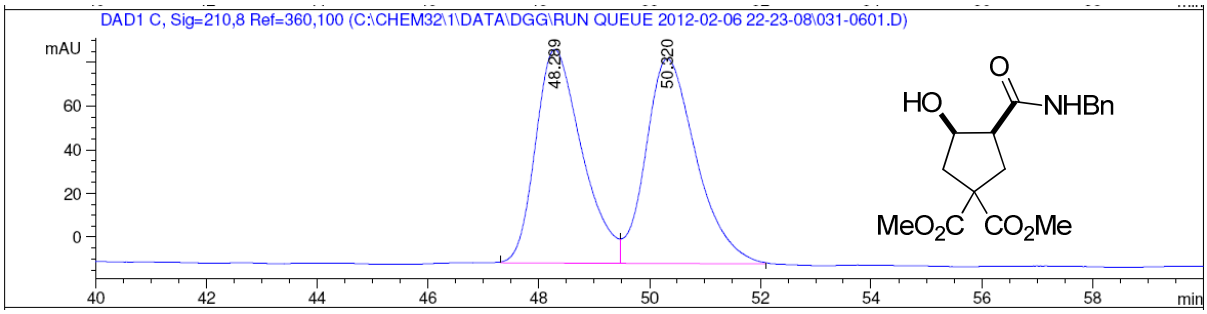
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	48.326	BB	1.0418	7197.42773	102.68043	66.6250
2	51.803	BB	1.1403	3605.46387	48.22161	33.3750
Totals :				1.08029e4	150.90203	

cis-Dimethyl-3-(benzylcarbamoyl)-4-hydroxycyclopentane-1,1-dicarboxylate





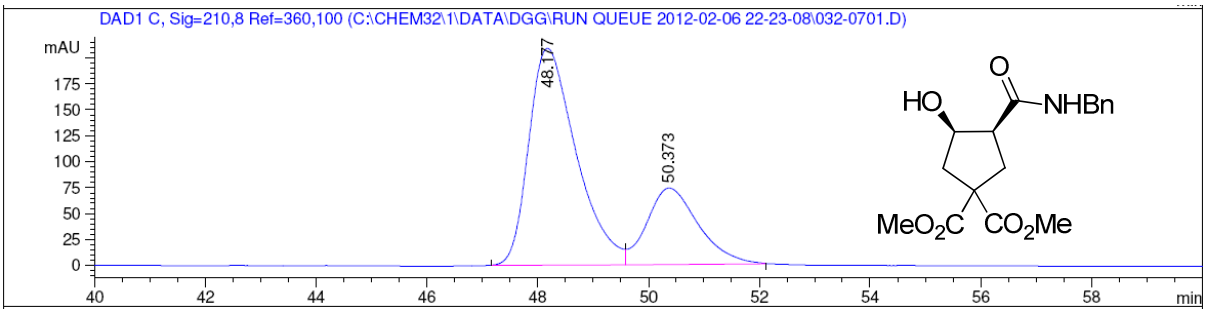
(±)-*cis*-Dimethyl-3-(benzylcarbamoyl)-4-hydroxycyclopentane-1,1-dicarboxylate (racemic)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	48.289	BV	0.8577	5654.68604	97.63580	48.8129
2	50.320	VB	0.9053	5929.73193	93.57759	51.1871
Totals :				1.15844e4	191.21339	

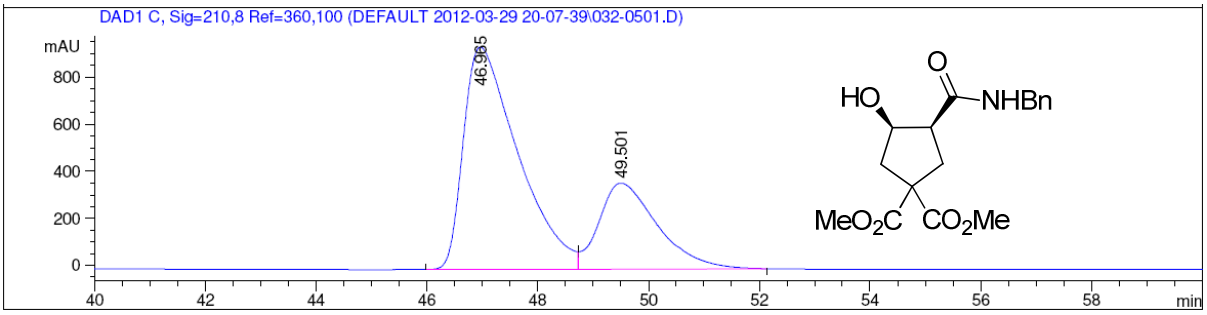
(+)-(1*R*,2*S*)-Dimethyl-3-(benzylcarbamoyl)-4-hydroxycyclopentane-1,1-dicarboxylate with catalyst 1 (45% *ee*)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	48.177	BV	0.8603	1.24249e4	208.77896	72.2503
2	50.373	VB	0.9356	4772.11084	73.67954	27.7497
Totals :				1.71970e4	282.45850	

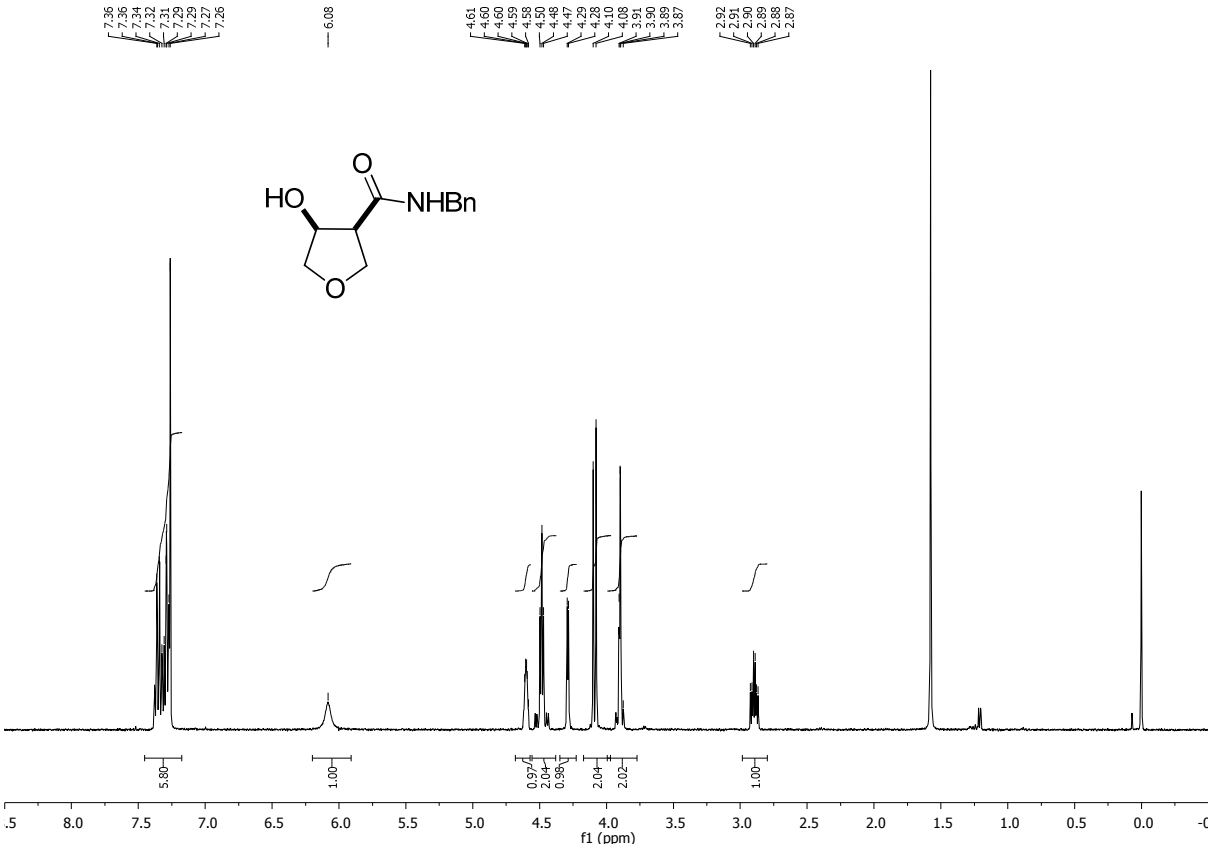
(+)-(1*R*,2*S*)-Dimethyl-3-(benzylcarbamoyl)-4-hydroxycyclopentane-1,1-dicarboxylate with catalyst 3 (41% *ee*)

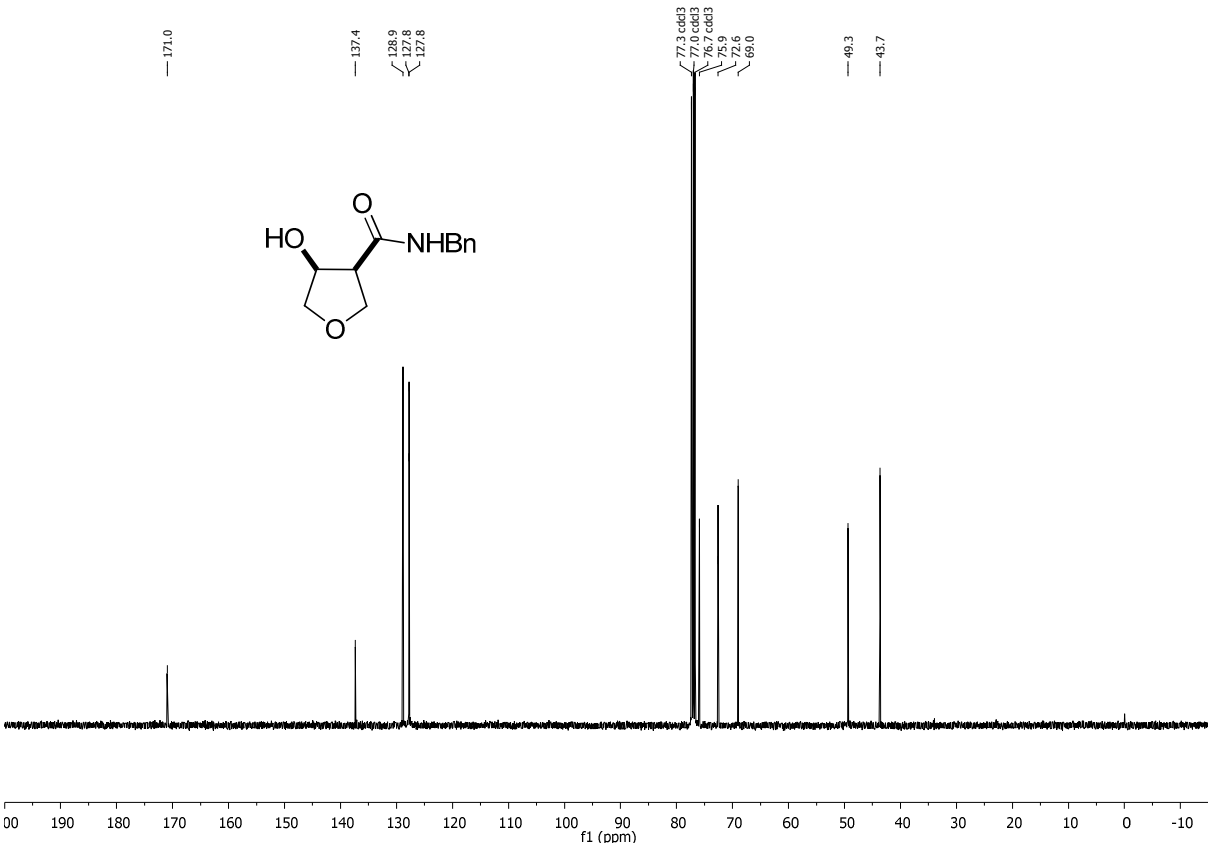


Signal 2: DAD1 C, Sig=210,8 Ref=360,100

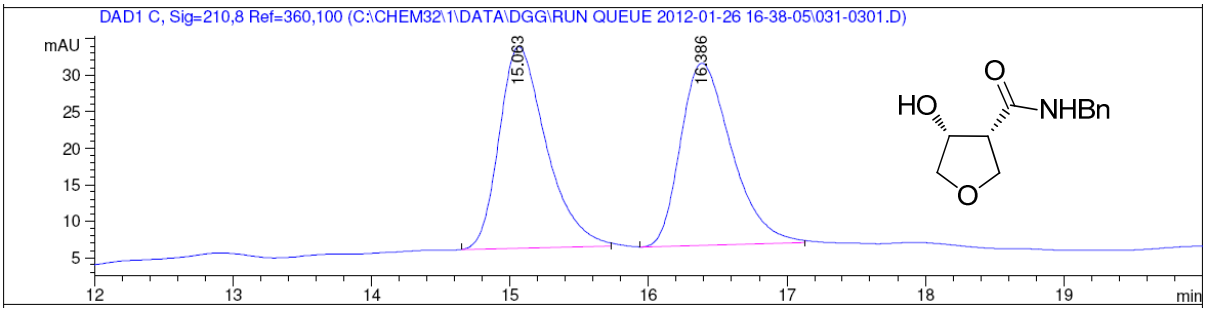
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	46.965	BV	0.9614	6.46423e4	945.72095	70.6761
2	49.501	VB	1.0194	2.68205e4	365.81467	29.3239
Totals :				9.14628e4	1311.53561	

cis-*N*-Benzyl-4-hydroxytetrahydrofuran-3-carboxamide





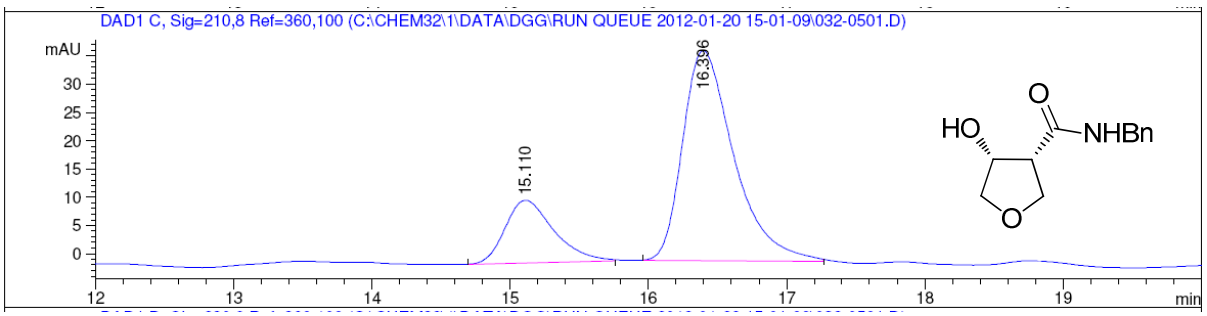
***cis*-*N*-Benzyl-4-hydroxytetrahydrofuran-3-carboxamide (racemic)**



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.063	BB	0.3616	652.18219	27.61157	50.5362
2	16.386	BB	0.3906	638.34375	24.93894	49.4638
Totals :				1290.52594	52.55051	

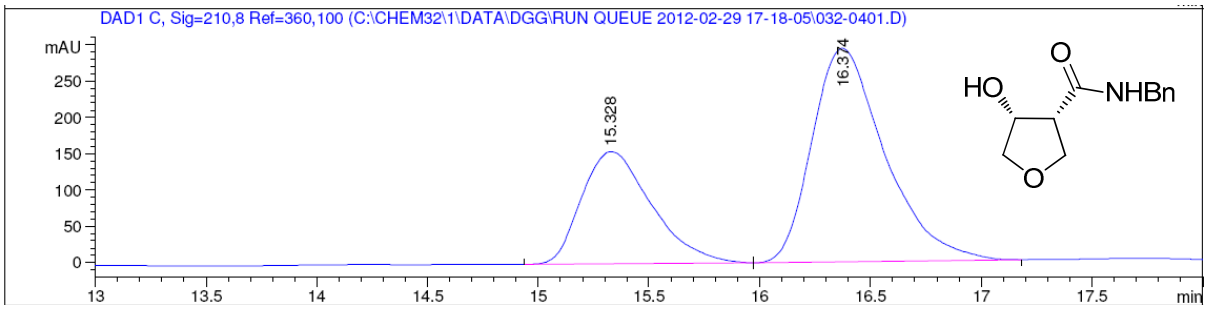
(-)-(1*R*,2*S*)-*N*-Benzyl-4-hydroxytetrahydrofuran-3-carboxamide with catalyst 1 (56% *ee*)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.110	BB	0.3725	269.93988	11.06958	21.9482
2	16.396	BB	0.3953	959.95636	37.17350	78.0518
Totals :				1229.89624	48.24307	

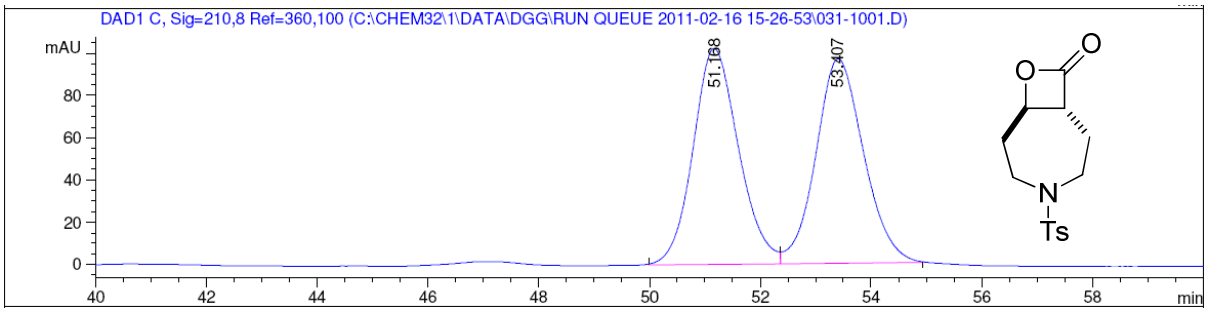
(-)-(1*R*,2*S*)-*N*-Benzyl-4-hydroxytetrahydrofuran-3-carboxamide with catalyst 3 (33% *ee*)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.328	BV	0.3337	3389.81177	154.68469	33.4747
2	16.374	VB	0.3449	6736.67578	294.48859	66.5253
Totals :				1.01265e4	449.17328	

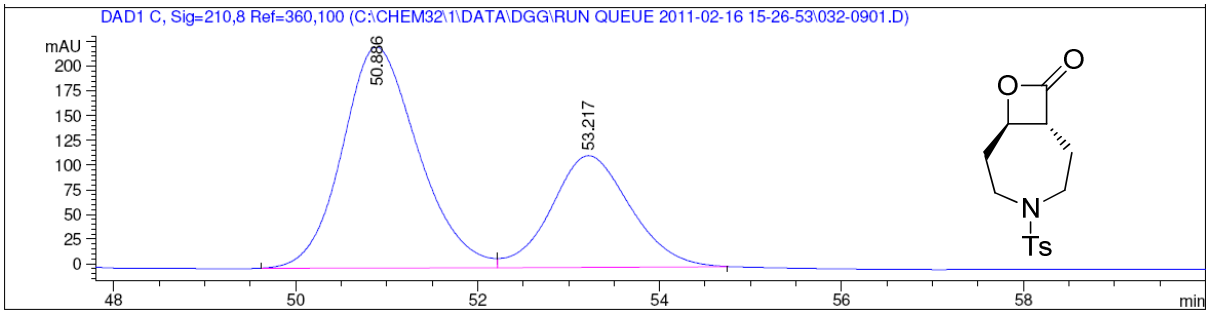
(±)-*trans*-4-Tosyl-8-oxa-4-azabicyclo[5.2.0]nonan-9-one (racemic)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	51.168	BV	0.8676	5833.23682	102.24943	50.0870
2	53.407	VB	0.9025	5812.96826	96.81593	49.9130
Totals :				1.16462e4	199.06535	

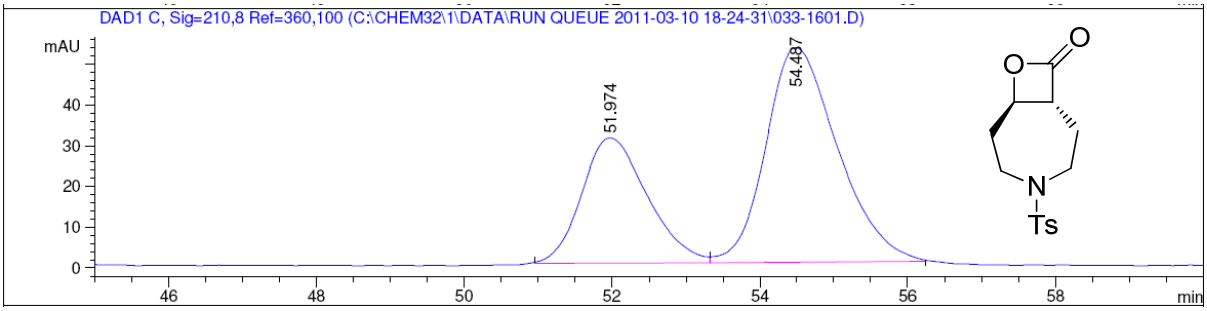
(+)-*trans*-4-Tosyl-8-oxa-4-azabicyclo[5.2.0]nonan-9-one with catalyst 1 (31% *ee*)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	50.886	BV	0.8812	1.30254e4	223.72398	65.5532
2	53.217	VB	0.9109	6844.55469	112.96080	34.4468
Totals :				1.98699e4	336.68478	

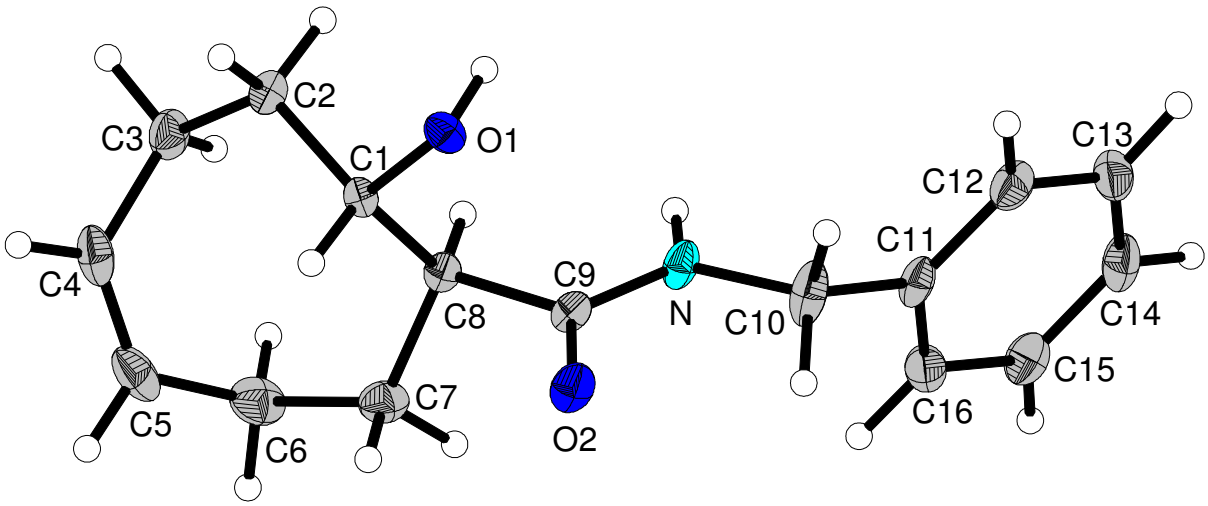
(-)-*trans*-4-Tosyl-8-oxa-4-azabicyclo[5.2.0]nonan-9-one with catalyst 3 (31% *ee*)



Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	51.974	BV	0.9068	1892.13208	30.71216	34.7161
2	54.487	VB	1.0054	3558.17212	52.58985	65.2839
Totals :				5450.30420	83.30202	

Crystal structure of (±)-*trans*-*N*-Benzyl-8-hydroxycyclooct-4-enecarboxamide



β-hydroxy benzylamide derivative of cyclooctene drawn at the 50% probability level