Electronic Supplementary Material (ESI) for RSC Advances.

Supplementary Information

General Information

All reactions were performed in scintillation vials, round bottom flasks (RBFs), or pressure tubes under an inert atmosphere of dry argon unless otherwise noted. Moisture-sensitive reactions were carried out using standard syringe septum techniques. Reaction solvent tetrahydrofuran (Fisher, HPLC grade) was dried by distillation from sodium-benzophenone radical ketyl. 1,4-Dioxane (Fisher, HPLC grade) was dried by distillation over calcium hydride. DMA (Sigma-Aldrich, ≥ 99.5% GC) was stored over 4Å molecular sieves. Solvents for filtration, transfers, and chromatography were certified ACS grade. Evaporation of solvents was carried out under reduced pressure on the rotary evaporator below 42 °C. Palladium catalysts and phosphine ligands were purchased from Sigma-Aldrich, Strem, and Apollo Scientific. Other reagents were purchased from Sigma-Aldrich, Alfa Aesar, and Acros Organics.

Purification of the reaction mixture was performed by Bruker flash chromatography, column chromatography, or preparative TLC. The stationary phase for chromatography was silica gel 40-63 UM 60A, or 230-400 mesh. Preparative TLC was performed on MERCK precoated silica gel 60-F254 (0.5-mm) aluminium plates. Solvents for purification were purchased from LabServe. TLC visualisation was carried out using ultraviolet light (254 nm) and different staining reagents such as potassium permanganate, 2,4-dinitropyridine, ethanolic phosphomolybdic acid, or ninhydrin when applicable.

Analytical Instruments

Optical rotation was recorded on a Perkin Elmer 241 polarimeter with the sodium lamp emitting at 589 nm (D = 589 nm). All samples were measured in chloroform unless otherwise noted in a 10 cm cell and an average of 3 readings was taken.

Circular Dichroism spectrum was recorded using a Chirascan CD spectrophotometer (150 W Xe arc) from Applied Photophysics. CD spectra (average of at least 2 scans) were recorded between 220 and 350 nm with 1 nm intervals, 120 nm/min scan rate, and 10 mm path length followed by subtraction of a background spectrum (solvent).

Infrared spectroscopy was carried out on ThermoScientific Nicolet iS5 iD7 ATR.

NMR spectroscopy was performed on Bruker 400 MHz, Bruker 500 MHz, or Bruker 700 MHz. Chemical shifts are reported in parts per million (ppm). All spectra were run in $CDCl_3$ unless otherwise stated. Spin multiplicities are described as s (singlet), bs (broad singlet), d (doublet),

dd (doublet of doublets), ddd (doublet of doublet of doublets), dddd (doublet of doublet of doublet of doublets), t (triplet), td (triplet of doublets), q (quartet), quint (quintet), m (multiplet), dt (doublet of triplet), ddt (doublet of doublet of triplets), dtd (doublet of triplet of doublets), dq (doublet of quartets). Coupling constants are reported in Hertz (Hz).

HRMS data were recorded on ThermoScientific Q Exactive Focus Hybrid Quadrupole-orbitrap Mass Spectrometer. MS data were recorded on ThermoScientific MSQ plus. Samples were injected *via* Dionex Ultimate 3000 HPLC system running at 0.1 mL/min, in MeOH.

General procedures for the synthesis of oxazolines

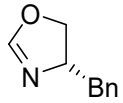
Method I

A solution of amino alcohol (1.0 eq.), triethyl orthoformate (1.5 eq.), and catalytic glacial AcOH (10 mol%) in dry DCE (10 mL) (dried over P_2O_5) was heated to reflux overnight. The solution was cooled down and the volatiles were removed under reduced pressure. The oxazoline was purified by either Kugelrohr distillation or flash chromatography as specified in the respective compound procedure.

Method II

An amino alcohol (1.0 eq.) and DMF-DMA (1.2 eq.) was heated to reflux at 85 °C for 24 hrs. Then *p*-toluenesulfonic acid monohydrate (0.1 eq.) and hexane (10 mL) were added. An addition funnel containing approximately 4 mL of activated 4 Å molecular sieves was placed on top of the flask, along with a condenser. The solution was heated to 90 °C for 24 hrs, allowing the distillate to condense over the sieves. The reaction mixture was washed with saturated aqueous $NaHCO_3$ solution and brine (10 mL). The aqueous layers were combined and back-extracted with Et_2O (3 × 20 mL). The combined organic layers were dried over Na_2SO_4 and filtered. The concentration of the filtrate gave the desired oxazoline.

(4*S*)-4-Benzyl-2-oxazoline 6a



6a

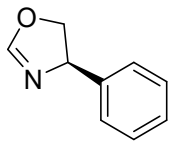
General method **I** was followed with L-phenylalaninol (3.0 g, 29.1 mmol, 1.0 eq.), triethyl orthoformate (6.4 g, 43.6 mmol, 1.5 eq.), and glacial AcOH (0.2 ml, 2.9 mmol, 0.1 eq.).

Purification: Kugelrohr distillation at 55 °C, 5 mTorr to obtain **6a** as a colorless liquid (1.8 g, 11.2 mmol, 56%).

The title compound can also be synthesised by general method **II**: L-phenylalaninol (1.0 g, 6.6 mmol, 1.0 eq.), DMF-DMA (1.0 mL, 7.9 mmol, 1.2 eq.), *p*-toluenesulfonic acid monohydrate (2 mg, 0.01 mmol, 0.001 eq.), to obtain **6a** as a colorless liquid (0.72 g, 4.47 mmol, 68%).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.33-7.30 (2H, m), 7.25-7.21 (3H, m), 6.83 (1H, d, J = 1.8 Hz), 4.43 (1H, ddd, J = 15.5, 7.8, 1.8 Hz), 4.19 (1H, t, J = 9.2 Hz), 3.95 (1H, dd, J = 8.3, 7.5 Hz), 3.12 (1H, dd, J = 13.8, 5.8 Hz), 2.71 (1H, dd, J = 13.9, 8.3 Hz).
Data are comparable to that reported in literature.[1]

(*R*)-4-Phenyl-2-oxazoline 6b



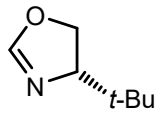
6b

General method **I** was followed with (*R*)-(–)-2-phenylglycinol (2.5 g, 18.2 mmol, 1.0 eq.), triethyl orthoformate (4.5 ml, 27.3 mmol, 1.0 eq.), and glacial AcOH (0.10 ml, 1.8 mmol, 0.1 eq.).

Purification: Kugelrohr distillation at 45 °C, 50 mTorr to obtain **6b** as a colorless liquid (1.4 g, 9.5 mmol, 52%).

IR: ν_{max} 3020, 2973, 2399, 2166, 2009, 1917, 1684, 1517, 1215 cm^{-1}.
^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.38-7.24 (5H, m), 7.05 (1H, d, J = 2.0 Hz), 5.23 (1H, ddd, J = 10.3, 8.5, 1.9 Hz), 4.62 (1H, dd, J = 10.3, 8.6 Hz), 4.09 (1H, dd, J = 8.4 Hz).
^{13}C NMR ($CDCl_3$, 126 MHz): δ 161.68, 138.34, 128.92, 128.02, 126.66, 66.02, 54.75.
HRMS (ESI-TOF) m/z: $[M+H]^+$ Calcd. for $C_9H_{10}NO$ 148.0757; found 148.0755.
Data are comparable to that reported in the literature.[2]

(4*S*)-*tert*-Butyl-2-oxazoline 6c

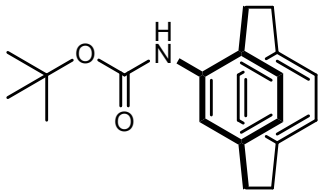


6c

General method **III** was followed by taking DMF-DMA (1.2 mL, 10.2 mmol, 1.2 eq.), L-*tert*-leucinol (1.0 g, 8.5 mmol, 1.0 eq.), *p*-toluenesulfonic acid monohydrate (1.6 mg, 0.1 mmol, 0.01 eq.). The product **6c** was obtained as a clear colorless oil (0.73 g, 5.74 mmol, 67%).

^{1}H NMR ($CDCl_3$, 400 MHz): 6.83 (1H, s), 4.15 (1H, t, J = 9.5 Hz), 4.02 (1H, t, J = 8.6 Hz), 4.02 (1H, t, J = 9.3 Hz), 0.93 (9H, s).
Data are comparable to that reported in the literature.[1]

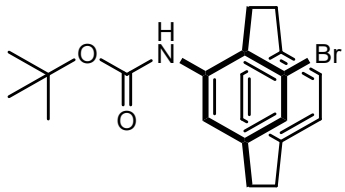
Preparation of (±)-4-bromo-8-amino[2.2]paracyclophane (10 h):
Boc protected-(±)-4-amino[2.2]paracyclophane



(±)-4-Amino[2.2]paracyclophane (1.0 g, 4.5 mmol, 1.0 eq.), $(Boc)_2O$ (1.1 g, 4.9 mmol, 1.1 eq.) and $NaHCO_3$ (1.1 g, 13.45 mmol, 3.0 eq.) were dissolved in H_2O: dioxane (1:1; 10 mL). The reaction mixture was stirred at room temperature overnight and the completion was confirmed by TLC. The crude mixture was purified using isocratic column chromatography [SiO_2, EtOAc:Hexanes (05:95)] to obtain the title compound as an off-white solid (0.34 g, 1.1 mmol, 23%).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 6.80 (1H, dd, J = 7.9, 1.8 Hz), 6.73 (1H, s), 6.57-6.48 (2H, m), 6.43 (1H, d, J = 7.9 Hz), 6.37 (1H, dd, J = 7.4, 1.7 Hz) 6.25 (1H, s), 3.24 (1H, ddd, J = 14.2, 9.6, 1.8 Hz), 3.19-3.10 (1H, m), 3.10-2.97 (5H, m), 2.83-2.73 (1H, ddd, J = 14.1, 8.9, 6.8 Hz), 1.57 (9H, s).

(±)-4-Bromo[2.2]paracyclophane-8-amino *tert*-butyl carbamate



Boc protected-(±)-4-amino[2.2]paracyclophane (200 mg, 0.62 mmol, 1.0 eq.) was dissolved in DMF (1.0 mL) and *N*-bromosuccinimide (122 mg, 0.78 mmol, 1.1 eq.) was added. The reaction was stirred at room temperature for 15 hrs. Complete consumption of the starting material was confirmed by TLC. Two bromo derivatives were separated using isocratic column chromatography [SiO_2, EtOAc: Hexane (05:95)] to obtain the title compound as an off-white solid (114 mg, 0.28 mmol, 45%).

^{1}H NMR ($CDCl_3$, 700 MHz): δ 7.16 (1H, dd, J = 7.9, 1.8 Hz), 6.83 (1H, dd, J = 7.7, 1.8 Hz), 6.57-6.54 (1H, m), 6.83 (1H, dd, J = 7.7, 1.8 Hz), 6.57-6.54 (1H, m), 6.49 (1H, s), 6.47 (1H, dd, J = 7.8, 1.8 Hz), 6.45-6.43 (1H, m), 6.27 (1H, bs, -NH), 3.42 (1H, ddd, J = 12.4, 10.3, 1.8 Hz), 3.22-3.16 (3H, m), 3.10-3.05 (2H, m), 2.82-2.76 (1H, m), 2.71 (1H, ddd, J = 14.3, 10.3, 7.2 Hz), 1.59 (9H, s).

^{13}C NMR ($CDCl_3$, 176 MHz): δ 152.53, 140.28, 139.22, 138.57, 138.36, 136.59, 135.01, 133.17, 132.48, 132.15, 129.72, 129.04, 128.18, 80.78, 35.47, 33.48, 33.21, 32.43, 28.39.

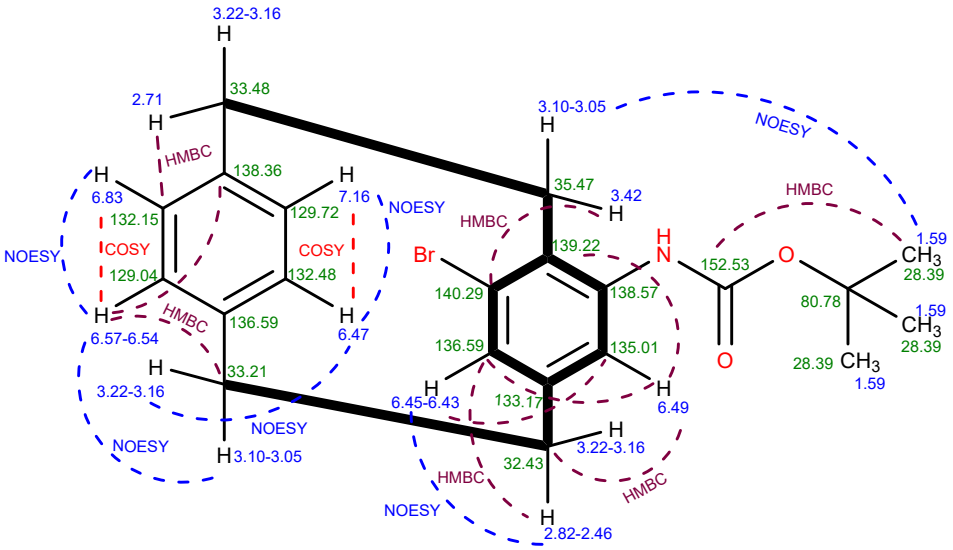
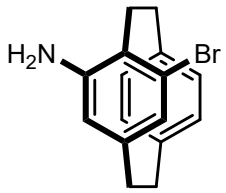


Figure 1. Isomer elucidation by NOESY, COSY, & HMBC (the indicated values are the ppm values obtained in ^{1}H & ^{13}C NMR spectrum).

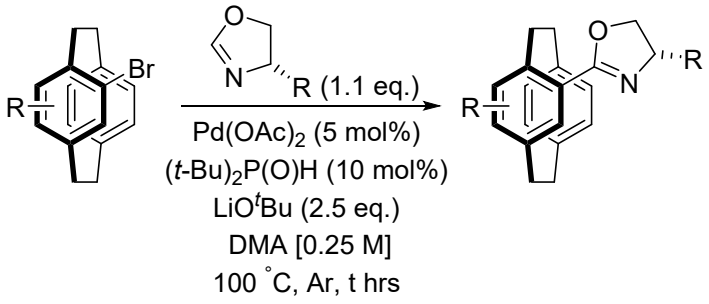
(±)-4-Bromo-8-amino[2.2]paracyclophane



10h

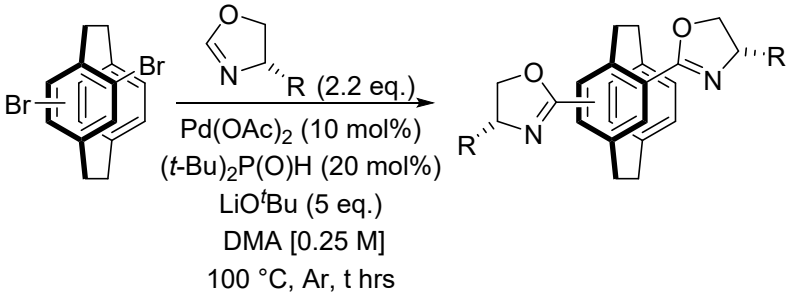
(±)-4-Bromo[2.2]paracyclophane-8-amino *tert*-butyl carbamate (100 mg) was dissolved in DCM (0.5 mL) and was cooled to 0 °C. TFA (0.1 mL) was added slowly to the reaction mixture. The reaction mixture was stirred at room temperature for 3 hrs and completion of the starting material was confirmed by TLC. The crude product was used without further purification.

General conditions A: mono-coupling of oxazoline with 4-bromo[2.2]paracyclophane



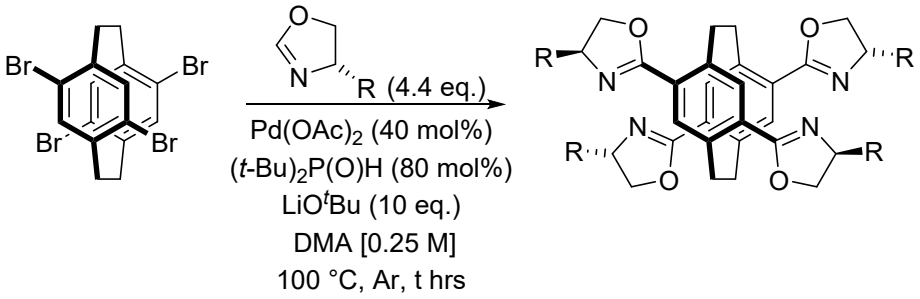
A 4 ml scintillation vial was charged with bromo[2.2]paracyclophane (1.0 eq.), oxazoline (1.1 eq.), LiO^tBu (2.5 eq.), (*t*-Bu)$_2$P(O)H (0.1 eq.), and Pd(OAc)$_2$ (0.05 eq.). The vial was purged and degassed with argon three times. Degassed DMA (0.25 M) was introduced by syringe and the reaction was heated to 100 °C for the time specified. The reaction mixture was cooled and diluted with CH_2Cl_2/MeOH (1:1) before passing through Celite®. The filtrate was concentrated under reduced pressure and the crude material was purified by chromatography as described.

General conditions B: bis-coupling of oxazoline with dibromo[2.2]paracyclophane



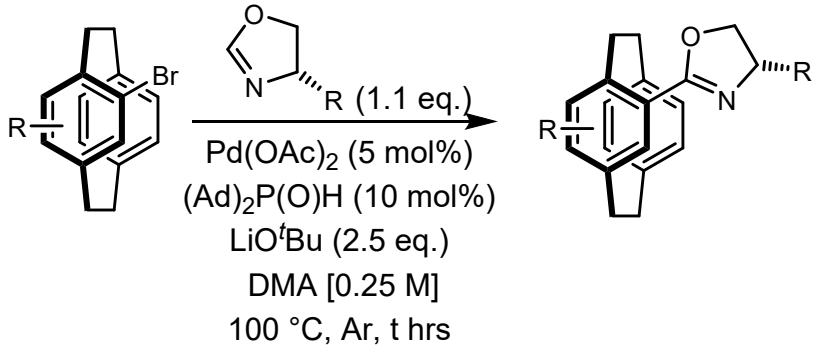
A 4 ml scintillation vial was charged with dibromo[2.2]paracyclophane (1.0 eq.), oxazoline (2.2 eq.), LiO^{t}Bu (5 eq.), (t-Bu)$_2$P(O)H (0.2 eq.), and Pd(OAc)$_2$ (0.1 eq.). The vial was purged and degassed with argon three times. Degassed DMA (0.25 M) was introduced by syringe and the reaction heated to 100 °C for the time specified. The reaction mixture was cooled and diluted with CH_2Cl_2/MeOH (1:1) before filtering through Celite®. The filtrate was concentrated under reduced pressure and the crude material was purified by chromatography as described.

General conditions C: tetra-coupling of oxazoline with tetrabromo[2.2]paracyclophane



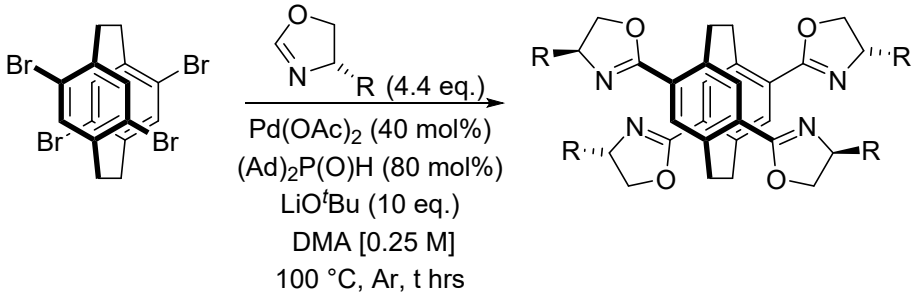
A 4 ml scintillation vial was charged with tetrabromo[2.2]paracyclophane (1.0 eq.), oxazoline (4.4 eq.), LiO^{t}Bu (10.0 eq.), (t-Bu)$_2$P(O)H (0.8 eq.), and Pd(OAc)$_2$ (0.4 eq.). The vial was purged and degassed with argon three times. Degassed DMA (0.25 M) was introduced by syringe and heated to 100 °C for the specified time. The reaction mixture was cooled and diluted with CH_2Cl_2/MeOH (1:1) before passing through a pad of Celite®. The filtrate was concentrated under reduced pressure and the crude material was purified by chromatography as described.

General conditions D: mono-coupling of oxazoline with bromo[2.2]paracyclophane



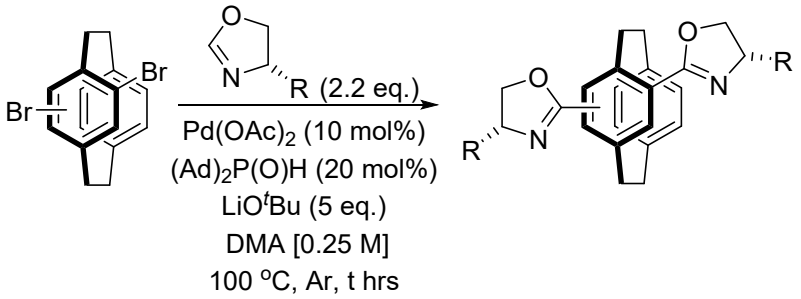
A 4 ml scintillation vial was charged with bromo[2.2]paracyclophane (1.0 eq.), oxazoline (1.1 eq.), LiO^{t}Bu (2.5 eq.), $(Ad)_2P(O)H$ (0.1 eq.), and $Pd(OAc)_2$ (0.05 eq.). The vial was purged and degassed with argon three times. Degassed DMA (0.25 M) was introduced by syringe and the reaction heated to 100 °C. The reaction mixture was cooled and diluted with CH_2Cl_2/MeOH (1:1) filtering through Celite®. The filtrate was concentrated under reduced pressure and the crude material was purified by chromatography as described.

General conditions E: tetra-coupling of oxazoline with tetrabromo[2.2]paracyclophane



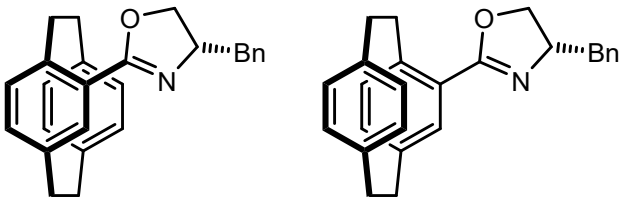
A 4 ml scintillation vial was charged with tetrabromo[2.2]paracyclophane (1.0 eq.), oxazoline (4.4 eq.), LiO^{t}Bu (10.0 eq.), $(Ad)_2P(O)H$ (0.8 eq.), and $Pd(OAc)_2$ (0.4 eq.). The vial was purged and degassed with argon three times. Degassed DMA (0.25 M) was introduced by syringe and heated to 100 °C for the specified time. The reaction mixture was cooled and diluted with CH_2Cl_2/MeOH (1:1) before passing through a pad of Celite®. The filtrate was concentrated under reduced pressure and the crude material was purified by chromatography as described.

General conditions F: bis-coupling of oxazoline with dibromo[2.2]paracyclophane



A 4 ml scintillation vial was charged with dibromo[2.2]paracyclophane (1.0 eq.), oxazoline (2.2 eq.), LiO^{t}Bu (5.0 eq.), $(Ad)_2P(O)H$ (0.2 eq.), and $Pd(OAc)_2$ (0.1 eq.). The vial was purged and degassed with argon three times. Degassed DMA (0.25 M) was introduced by syringe and the reaction heated to 100 °C. The reaction mixture was cooled and diluted with CH_2Cl_2/MeOH (1:1) filtering through Celite®. The filtrate was concentrated under reduced pressure and the crude material was purified by chromatography as described.

(R_p,*S*)-4-(4′-Benzyloxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 8a



(*R*$_p$,*S*)-8a (*S*$_p$,*S*)-8a

General conditions **A** were followed using (±)-4-bromo[2.2]paracyclophane **7** (70 mg, 0.24 mmol, 1.0 eq.), (4*S*)-4-benzyl-2-oxazoline **6a** (43 mg, 0.3 mmol, 1.1 eq.), LiO^{t}Bu (49 mg, 0.6 mmol, 2.5 eq.), (*t*-Bu)$_2$P(O)H (4 mg, 0.03 mmol, 0.1 eq.), $Pd(OAc)_2$ (4 mg, 0.02 mmol, 0.05 eq.), DMA (0.98 ml) for 28 hrs.

Purification: Isocratic column chromatography [SiO_2, EtOAc:Hexanes (05:95)] to obtain the title compound **8a** (72 mg, 0.2 mmol, 82%) as diastereomer 1, **8a1**, off-white solid (16 mg, 0.04 mmol, 18%), mixture of diastereomers (46 mg, 0.13 mmol, 52%), and diastereomer 2, **8a2**, off-white solid (10 mg, 0.03 mmol, 12%).

4-(4′-Benzyloxazolin-2′-yl)[2.2]paracyclophane 8a1

Relative stereochemistry no determined

R_f : 0.3 (EtOAc:Hexanes 05:95)

$[\alpha]_D^{20}$: –22.0 (c = 1.00, CH_2Cl_2)

IR: ν_{max} 2925, 2852, 2121, 1896, 1712, 1634, 1497 cm^{-1}.

^1H NMR ($CDCl_3$, 500 MHz): δ 7.41-7.33 (4H, m), 7.31-7.25 (1H, m), 7.17 (1H, s), 6.64 (1H, d, J = 7.7 Hz), 6.60-6.52 (5H, m), 4.68 (1H, q, J = 5.9 Hz), 4.38 (1H, t, J = 8.5 Hz), 4.30-4.15 (2H, m), 3.35 (1H, dd, J = 13.7, 5.0 Hz), 3.24-3.12 (3H, m), 3.11-3.00 (3H, m), 2.92-2.82 (2H, m).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 141.08, 139.85, 139.73, 139.44, 137.99, 136.31, 136.01, 135.36, 134.59, 133.02, 132.78, 132.42, 131.27, 129.37, 128.83, 128.66, 126.61, 71.91, 41.83, 35.92, 35.31, 35.09, 34.87, 29.71.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{26}H_{26}NO$ 368.2009; found 368.2005.

4-(4′-Benzyloxazolin-2′-yl)[2.2]paracyclophane 8a2

Relative stereochemistry not determined

R_f: 0.2 (EtOAc:Hexanes 05:95)

$[\alpha]_D^{20}$: +12.0 (c = 1.00, CH_2Cl_2)

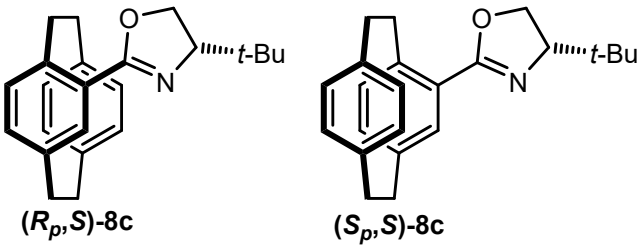
IR: ν_{max} 2925, 1631, 1475, 1328, 1273, 1045 cm^{-1}.

^1H NMR ($CDCl_3$, 500 MHz): δ 7.41-7.33 (4H, m), 7.31-7.25 (1H, m), 7.13 (1H, s), 6.63 (1H, d, J = 7.7 Hz), 6.59-6.49 (5H, m), 4.73-4.66 (1H, m), 4.41 (1H, t, J = 8.7 Hz), 4.18 (1H, t, J = 7.9 Hz), 4.12 (1H, t, J = 11.5 Hz,), 3.33 (1H, dd, J = 13.7, 5.0 Hz), 3.19-3.11 (3H, m), 3.09-2.98 (3H m), 2.92-2.83 (2H, m).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 141.08, 139.80, 139.71, 139.42, 135.91, 135.21, 134.47, 132.93, 132.79, 132.73, 132.40, 131.32, 129.45, 129.36, 128.85, 128.62, 126.60, 71.32, 41.72, 35.93, 35.33, 35.09, 34.98, 29.72.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{26}H_{26}NO$ 368.2009; found 368.2005.

(R_p,*S*)-4-(4′-*tert*-Butyloxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-(4′-*tert*-butyloxazolin-2′-yl)[2.2]paracyclophane 8c



(R_p,*S*)-8c (S_p,*S*)-8c

General conditions **D** were followed using (±)-4-bromo[2.2]paracyclophane **7** (100 mg, 0.35 mmol, 1.0 eq.), (4*S*)-4-*tert*-butyl-2-oxazoline **6c** (49 mg, 0.4 mmol, 1.1 eq.), LiO^{t}Bu (70 mg, 0.9 mmol, 2.5 eq.), $(Ad)_2P(O)H$ (11 mg, 0.04 mmol, 0.1 eq.), $Pd(OAc)_2$ (4 mg, 0.02 mmol, 0.05 eq.), DMA (1.3 ml) for 15 hrs.

Purification: Isocratic column chromatography [SiO_2, EtOAc:Hexanes (05:95)] to obtain the title compound **8c** (70 mg, 0.2 mmol, 60%) as separable diastereomers, 1st diastereomer **(R_p,*S*)-8c**, off-white solid (35 mg, 0.1 mmol, 30%), and 2nd diastereomer **(S_p,*S*)-8c,** off-white solid (35 mg, 0.1 mmol, 30%).

(R_p,*S*)-4-(4′-*tert*-Butyloxazolin-2′-yl)[2.2]paracyclophane 8c

R_f: 0.4 (EtOAc:Hexanes 05:95)

$[\alpha]_D^{20}$: –141.16 (c = 1.00, CH_2Cl_2) [lit., $[\alpha]_D^{20}$: –128 (c = 1.00, CH_2Cl_2)].

IR: ν_{max} 3355, 2933, 1666, 1608, 1506, 1399, 1249, 1032 cm^{-1}.

^1H NMR ($CDCl_3$, 500 MHz): δ 7.05 (1H, s), 6.65 (1H, d, J = 16.4, 7.7 Hz), 6.57-6.52 (3H, m), 6.48 (1H, d, J = 7.7 Hz), 4.37 (1H, t, J = 8.9 Hz), 4.21 (1H, t, J = 8.2 Hz), 4.17-4.09 (2H, m), 3.22-3.09 (4H, m), 3.08-2.99 (2H, m), 2.90-2.83 (1H, m), 1.04 (9H, s).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 163.48, 140.86, 140.05, 139.53, 139.35, 135.73, 134.74, 134.33, 132.91, 132.78, 132.38, 131.24, 128.49, 76.64, 68.00, 35.89, 35.32, 35.07, 34.81, 34.00, 26.09.

MS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{23}H_{28}NO$ 334.22; found 334.21.

Data comparable to that reported in literature.[3]

(S_p,*S*)-4-(4′-*tert*-Butyloxazolin-2′-yl)[2.2]paracyclophane 8c

R_f: 0.45 (EtOAc:Hexanes 05:95)

$[\alpha]_D^{20}$: +40.03 (c = 1.00, CH_2Cl_2) [lit., $[\alpha]_D^{20}$: +56.5 (c = 1.00, CH_2Cl_2)].

IR: ν_{max} 2956, 1641, 1499, 1361, 1332, 1173, 1068 cm^{-1}.

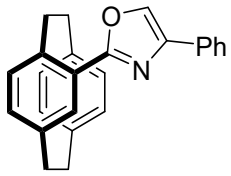
^1H NMR ($CDCl_3$, 500 MHz): δ 7.04 (1H, s), 6.60-6.52 (5H, m), 6.47 (1H, d, J = 7.8 Hz), 4.41(1H, ddd, J = 12.5, 8.5, 3.7 Hz), 4.33-4.29 (1H, m), 4.25 (1H, t, J = 8.3 Hz), 4.12 (1H, t, J = 8.7 Hz), 3.19-3.11 (4H, m), 3.07-3.00 (2H, m), 2.94-2.87 (1H, m), 1.09 (9H, s).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 163.01, 141.03, 140.00, 139.45, 139.37, 135.94, 134.88, 134.00, 132.87, 132.74, 132.52, 131.60, 128.38, 76.83, 67.63, 35.46, 35.37, 35.14, 34.79, 34.34, 26.31.

MS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{23}H_{28}NO$ 334.22; found 334.21.

Data comparable to that reported in literature.[3]

(±)-4-(4′-Phenyloxazole)[2.2]paracyclophane 9



9

General conditions **A** were followed using (±)-4-bromo[2.2]paracyclophane **7** (40 mg, 0.14 mmol, 1.0 eq.), (*R*)-4-phenyl-2-oxazoline **6b** (23 mg, 0.15 mmol, 1.1 eq.), LiO^tBu (28 mg, 0.35 mmol, 2.5 eq.), (t-Bu)$_2$P(O)H (2 mg, 0.014 mmol, 0.1 eq.), $Pd(OAc)_2$ (2 mg, 0.007 mmol, 0.05 eq.), DMA (1.09 ml) for 24 hrs.

Purification: Flash chromatography [SiO_2, EtOAc:Hexanes 05:95] gave **9**, as a white solid (27 mg, 0.076 mmol, 55%).

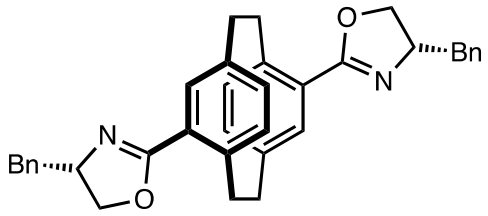
R_f: 0.7 (EtOAc:Hexanes 05:95)

IR: ν_{max} 3019, 2972, 2401, 2254, 2008, 1968, 1394, 1215 cm^{-1}.

^1H NMR ($CDCl_3$, 500 MHz): δ 7.73-7.62 (2H, m), 7.53-7.42 (1H, m), 7.34-7.29 (1H, m), 7.26-7.18 (1H, m), 6.94 (1H, d, J = 7.9 Hz), 6.79 (1H, d, J = 1.3 Hz), 6.76 (1H, d, J = 7.9 Hz), 6.62 (1H, d, J = 7.9 Hz), 6.55 (2H, s), 6.53 (1H, d, J = 7.9 Hz), 3.57 (1H, ddd, J = 13.4, 10.5, 2.3 Hz), 3.35 (1H, ddd, J = 13.2, 10.8, 4.6 Hz), 3.20-3.04 (6H, m).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 144.26, 140.94, 139.54, 139.14, 137.13, 136.80, 134.53, 133.52, 132.90, 132.71, 130.98, 129.93, 129.58, 129.03, 128.96, 128.29, 128.09, 127.30, 118.94, 114.92, 35.33, 35.09, 34.47, 34.24.

(*S*,R_p,S_p,*S*)-4,16-Bis(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane 11a



(*S*,R_p,S_p,*S*)-11a

General conditions **A** were followed using 4,16-dibromo[2.2]paracyclophane **10a** (50 mg, 0.14 mmol, 1.0 eq.), (4*S*)-4-benzyloxazoline **6a** (24 mg, 0.15 mmol, 1.1 eq.), LiO^tBu (27 mg, 0.15 mmol, 2.5 eq.), (t-Bu)$_2$P(O)H (2 mg, 0.014 mmol, 0.1 eq.), Pd(OAc)$_2$ (2 mg, 0.006 mmol, 0.05 eq.), DMA (0.55 ml) for 24 hrs.

Purification: Flash chromatography [SiO_2, EtOAc:Hexanes (30:70)] to obtain the title compound **11a** as a single stereoisomer, pale yellow solid (22 mg, 0.042 mmol, 31%) and by-product **8a** as a mixture of diastereomers, an off-white solid (25 mg, 0.068 mmol, 52%).

General conditions **B** were followed using 4,16-dibromo[2.2]paracyclophane **10a** (50 mg, 0.14 mmol, 1.0 eq.), (4*S*)-4-benzyl-2-oxazoline **6a** (49 mg, 0.30 mmol, 2.2 eq.), LiO^tBu (55 mg, 0.68 mmol, 5 eq.), (t-Bu)$_2$P(O)H (4 mg, 0.027 mmol, 0.2 eq.), Pd(OAc)$_2$ (4 mg, 0.013 mmol, 0.1 eq.), DMA (0.55 ml) for 48 hrs.

Purification: Flash chromatography [SiO_2, EtOAc:Hexanes (30:70)] to obtain the title compound **11a** as a single diastereomer, pale yellowish solid (58 mg, 0.11 mmol, 81% yield) and by-product **8a** as a mixture of diastereomers (9 mg, 0.024 mmol, 18% yield).

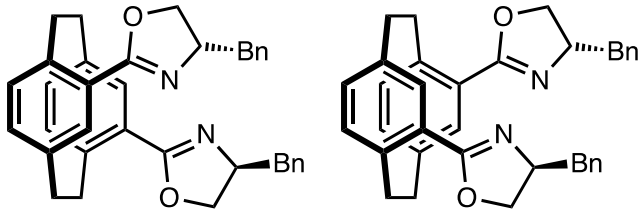
R_f : 0.6 (EtOAc:Hexanes 30:70)

IR: ν_{max} 3018, 2988, 2900, 1393 cm^{-1}.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.40-7.27 (8H, m), 7.30-7.27 (2H, m), 7.11 (2H, dd, J = 7.8, 2.0 Hz), 6.66 (2H, dt, J = 7.6, 2.1 Hz), 6.56 (2H, t, J = 7.6 Hz), 4.72-4.63 (2H, m), 4.44 (2H, dt, J = 22.7, 8.7 Hz), 4.26-4.09 (4H, m), 3.35 (2H, quint, J = 5.5 Hz), 3.13-3.03 (4H, m), 2.96-2.90 (2H, m), 2.87 (2H, m).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 164.40, 164.12, 140.92, 140.66, 139.81, 139.68, 138.18, 138.14, 135.14, 135.06, 134.21, 134.14, 133.37, 133.25, 129.40, 129.36, 128.63, 128.58, 128.50, 128.31, 126.55, 71.85, 70.92, 68.20, 68.16, 42.05, 41.92, 34.94, 34.42, 34.32.
HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{36}H_{35}N_2O_2$ 527.2693; found 527.2689.

(R_p,*S*)-4,12-Bis-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4,12-bis-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11b



(R_p,*S*)-11b (S_p,*S*)-11b

General conditions **A** were followed using (±)-4,12-dibromo[2.2]paracyclophane **10c** (50 mg, 0.13 mmol, 1.0 eq.), (4*S*)-4-benzyloxazoline **6a** (24 mg, 0.15 mmol, 1.1 eq.), LiO^{t}Bu (27 mg, 0.34 mmol, 2.5 eq.), (t-Bu)$_2$P(O)H (2 mg, 0.013 mmol, 0.1 eq.), $Pd(OAc)_2$ (2 mg, 0.006 mmol, 0.05 eq.), DMA (0.55 ml) for 12 hrs.

Purification: Flash chromatography [SiO_2, EtOAc:Hexanes (0-20%, gradient elution)] to obtain the title compound as diastereomer 1, **(S_p,*S*)-11b**, pale yellow solid (10 mg, 0.018 mmol, 15%), and the 2nd diastereomer **(R_p,*S*)-11b,** pale yellow solid (15 mg, 0.028 mmol, 22%), as well as monooxazoline **8a** as a mixture of diastereomers, (12 mg, 0.032 mmol, 25% yield), and monobromo-monooxazoline **11d** as a mixture of diastereomers, **(R_p,*S*)-11d** and **(S_p,*S*)-11d**, white solid (19 mg, 0.042 mmol, 31%).

General conditions **B** were followed using (±)-4,12-dibromo[2.2]paracyclophane **10c** (60 mg, 0.16 mmol, 1.0 eq.), (4*S*)-4-benzyl-2-oxazoline **6a** (58 mg, 0.36 mmol, 2.2 eq.), LiO^{t}Bu (66 mg, 0.82 mmol, 5 eq.), (t-Bu)$_2$P(O)H (5 mg, 0.032 mmol, 0.2 eq.), $Pd(OAc)_2$ (4 mg, 0.016 mmol, 0.1 eq.), DMA (0.66 ml) for 12 hrs.

Purification: Flash chromatography [SiO_2, EtOAc:Hexanes (0-20%, gradient elution)] to obtain the title compound **11b** as separable diastereomers, **(S_p,*S*)-11b**, pale yellow solid (30 mg, 0.057 mmol, 34%), and diastereomer **(R_p,*S*)-11b,** pale yellow solid (31 mg, 0.068 mmol, 36%),

and monobromo-monooxazoline **11d** as a mixture of diastereomers, **(R_p,*S*)-11d** and **(S_p,*S*)-11d**, white solid (5%).

Large Scale: General Conditions B were followed using (±)-4,12-dibromo[2.2]paracyclophane **10c** (6.3 g, 17.2 mmol, 1.0 eq.), (4*S*)-4-benzyl-2-oxazoline **6a** (6.1 g, 37.9 mmol, 2.2 eq.), LiO^{t}Bu (6.89 g, 86.1 mmol, 5 eq.), (t-Bu)$_2$P(O)H (0.56 g, 3.4 mmol, 0.2 eq.), Pd(OAc)$_2$ (0.39 g, 1.72 mmol, 0.1 eq.), DMA (0.66 ml) for 12 hrs.

Purification: Flash chromatography [SiO_2, 8% EtOAc:Hexanes] to obtain the title compound **11b** as separable diastereomers, **(S_p,*S*)-11b**, pale yellow solid (2.1 g, 3.96 mmol, 23%), and diastereomer **(R_p,*S*)-11b,** pale yellow solid (1.7 g, 3.27 mmol, 19%), monobromo-monooxazoline **11d** as a mixture of diastereomers, **(R_p,*S*)-11d** and **(S_p,*S*)-11d** (it is possible to enrich the diastereomeric mixture to favour one diastereomer by recrystallisation), white solid (0.9 g, 2.07 mmol, 12%) and monooxazoline **8a** as a mixture of diastereomers (0.16 g, 0.52 mmol, 3%).

(S_p,*S*)-4,12-Bis-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11b

R_f : 0.6 (EtOAc:Hexanes 20:80)

$[\alpha]_D^{22}$: –61.70 (c = 0.47, $CHCl_3$) [lit.: $[\alpha]_D^{22}$: –68.5 (c = 0.47, $CHCl_3$)]

IR: ν_{max} 3627, 2955, 2919, 1639, 1601, 1590, 1452 cm^{-1}.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.42-7.36 (8H, m), 7.31-7.28 (2H, m), 7.21 (2H, d, J = 1.4 Hz), 6.68 (2H, d, J = 8.3 Hz), 6.60 (2H, d, J = 7.8 Hz), 4.68 (2H, quint, J = 8.5 Hz), 4.36-4.30 (4H, m), 4.12 (2H, t, J = 7.6 Hz), 3.42 (2H, dd, J = 13.6, 6.2 Hz), 3.27-3.21 (2H, m), 3.16-3.12 (2H, m), 2.94-2.90 (2H, dd, J = 13.6, 8.6 Hz), 2.89 (2H, ddd, J = 12.5, 10.3, 7.1 Hz).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 163.49, 141.08, 140.12, 138.62, 135.79, 134.97, 132.08, 129.25, 128.63, 127.98, 126.48, 70.69, 68.73, 42.61, 35.65, 33.69.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{36}H_{35}N_2O_2$ 527.2693; found 527.2690.

Data comparable to that reported in literature.[4]

(R_p,*S*)-4,12-Bis-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11b

R_f: 0.5 (EtOAc:Hexanes 20:80)

$[\alpha]_D^{24}$: +23.61 (c = 0.72, $CHCl_3$) [Lit:$[\alpha]_D^{24}$: +13.9 (c = 0.72, $CHCl_3$)]

IR: ν_{max} 3674, 2925, 1634, 1590, 1495, 1352 cm^{-1}.

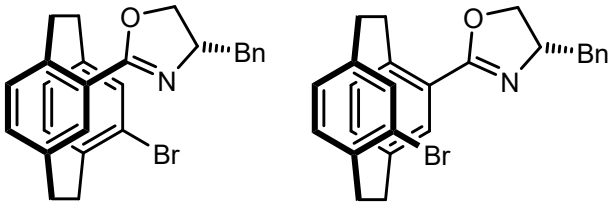
^1H NMR ($CDCl_3$, 400 MHz): δ 7.39-7.29 (8H, m), 7.27 (2H, d, J = 8.1 Hz), 7.15 (2H, d, J = 1.8 Hz), 6.68 (2H, d, J = 7.8 Hz), 6.59 (2H, d, J = 7.8 Hz), 4.67-4.61 (2H, m), 4.35-4.30 (4H, m), 4.07 (2H, t, J = 7.2 Hz), 3.29 (2H, dd, J = 13.7, 5.1 Hz), 3.17 (2H, dd, J = 8.3, 6.4 Hz), 2.93 (2H, m), 2.72 (2H, dd, J = 13.7, 9.1 Hz).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 163.69, 141.02, 140.10, 138.44, 135.76, 134.87, 132.58, 129.25, 128.55, 127.99, 126.43, 70.74, 68.43, 41.97, 36.04, 34.02.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{36}H_{35}N_2O_2$ 527.2693; found 527.2690.

Data comparable to that reported in literature.[4]

(R_p,*S*)-4-Bromo-12-(4′-benzyloxazoline-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-bromo-12-(4′-benzyloxazoline-2′-yl)[2.2]paracyclophane 11d



(R_p,*S*)-11d **(S_p,*S*)-11d**

R_f : 0.5 (EtOAc:Hexanes 30:70)

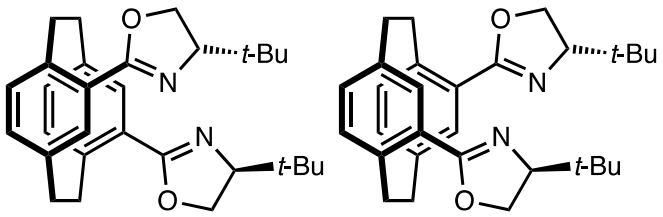
IR: ν_{max} 3675, 2989, 2900, 1405, 1393 cm^{-1}.

^1H NMR ($CDCl_3$, 500 MHz): δ 7.70 (2H, d, J = 8.5 Hz), 7.40-7.35 (8H, m), 7.31-7.24 (2H, m), 6.66-6.58 (8H, m), 6.52 (2H, d, J = 7.7 Hz), 4.70-4.60 (2H, m), 4.40 (1H, t, J = 8.6 Hz), 4.34 (1H, t, J = 8.6 Hz), 4.28 (1H, ddd, J = 12.1, 9.4, 2.3 Hz) 4.22-4.17 (2H, m), 4.15 (1H, t, J = 7.7 Hz), 3.51 (2H, dd, J = 11.7, 3.2 Hz), 3.37 (1H, dd, J = 13.8, 5.6 Hz), 3.30 (1H, dd, J = 13.7, 5.4 Hz), 3.24-3.19 (2H, m), 3.12-2.78 (12H, m).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 163.92, 163.63, 142.16, 142.03, 140.73, 140.57, 139.25, 139.20, 138.86, 138.82, 138.39, 138.32, 135.67, 135.16, 135.11, 134.97, 134.94, 134.90, 131.36, 131.25, 130.44, 130.19, 129.40, 129.30, 128.61, 128.54, 128.06, 127.97, 126.68, 126.63, 126.49, 126.45, 71.05, 70.90, 68.51, 68.29, 42.22, 42.03, 36.06, 35.85, 35.79, 35.69, 33.95, 33.68, 32.66, 32.60.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{26}H_{25}Br^{79}NO$ 446.1114; found 446.1111; $C_{26}H_{25}Br^{81}NO$ 448.1114; found 448.1089.

(R_p,*S*)-4,12-Bis-(4′-*tert*-butyloxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4,12-bis-(4′-*tert*-butyl-oxazolin-2′-yl)[2.2]paracyclophane 11c



(R_p,*S*)-11c (S_p,*S*)-11c

General conditions **F** were followed using (±)-4,12-dibromo[2.2]paracyclophane **10c** (100 mg, 0.27 mmol, 1.0 eq.), (4*S*)-4-*tert*-butyl-2-oxazoline **6c** (77 mg, 0.60 mmol, 2.2 eq.), LiO^{t}Bu (110 mg, 1.37 mmol, 5.0 eq.), $(Ad)_2P(O)H$ (18 mg, 0.027 mmol, 20 mol%), $Pd(OAc)_2$ (6 mg, 0.055 mmol, 10 mol%), DMA (1.1 ml) for 12 hrs.

Purification: Isocratic column chromatography [SiO_2, EtOAc: Hexanes (05:95)] to obtain a mixture of title compound **11c1** (1st diastereomer) and monooxazoline diastereomer **(S_p, *S*)-8c**, (refer to ^{1}H NMR data of **(S_p, *S*)-8c** for data) (1:1 mixture) as an off-white solid (20 mg), a mixture of diastereomers **11c1** & **11c2** (8 mg, 0.016 mmol, 6%), and then 2nd diastereomer **11c2**, as an off-white solid (24 mg, 0.052 mmol, 20%).

4,12-Bis-(4′-*tert*-butyloxazolin-2′-yl)[2.2]paracyclophane 11c1 (mixed with (S_p,*S*)-8c)

Diastereomer 1, relative stereochemistry not determined

R_f : 0.4 (EtOAc: Hexanes 04:96)

Peaks belonging to **11cc1** ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.10 (2H, d, J = 1.6 Hz), 7.02 (1H, d, J = 1.7 Hz), 6.63 (2H, dd, J = 5.96, 1.7 Hz), 6.59-6.50 (7H, m), 6.46 (1H, d, J = 7.9 Hz), 4.49-4.19 (7H, m), 4.02-4.14 (5H, m), 3.27-3.07 (8H, m), 3.07-2.97 (2H, m), 2.95-2.80 (3H, m), 1.11 (18H, s), 1.09 (9H, s).

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd $C_{30}H_{39}N_2O_2$ for 459.6428; Found 459.6434.

4,12-Bis-(4′-*tert*-butyloxazolin-2′-yl)[2.2]paracyclophane 11c2

Diastereomer 2, relative stereochemistry not determined

R_f : 0.3 (EtOAc:Hexanes 04:96)

$[\alpha]_D^{24}$: -50.1 (c = 0.9, $CHCl_3$)

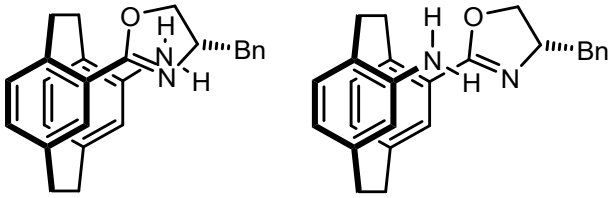
IR: ν_{max} 2954, 1640, 1477, 1259, 1048 cm^{-1}.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.14 (2H, s), 6.66 (2H, d, J = 7.6 Hz), 6.58 (2H, d, J = 7.8 Hz), 4.32 (2H, quint, J = 9.4 Hz), 4.25 (2H, t, J = 10.6 Hz), 4.17 (4H, m), 3.22-3.07 (4H, m), 2.84-2.78 (2H, m), 1.02 (18H, s).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 162.70, 140.87, 140.14, 135.49, 134.53, 132.08, 127.99, 76.90, 67.58, 36.10, 33.96, 33.91, 26.03.

MS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{30}H_{39}N_2O_2$ 459.6428; found 459.6437.

(R_p,*S*)-4-Amino-13-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-amino-13-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11e



(S_p,*S*)-11e **(R_p,*S*)-11e**

General conditions **A** was followed using (±)-4-bromo-13-amino[2.2]paracyclophane **10e** (58 mg, 0.19 mmol, 1.0 eq.), (4*S*)-4-benzyloxazoline **6a** (34 mg, 0.21 mmol, 1.1 eq.), Li*O*^{t}Bu (39 mg, 0.48 mmol, 2.5 eq.), (*t*-Bu)$_2$P(O)H (3 mg, 0.019 mmol, 0.1 eq.), $Pd(OAc)_2$ (2 mg, 0.009 mmol, 0.05 eq.), DMA (0.77 ml) for 24 hrs.

Purification: Flash chromatography [SiO_2, Et_3N:EtOAc:Hexanes (2:12:86)] to give one diastereomer of the title compound **11e1** (13 mg, 0.033 mmol, 18%), a mixture of diastereomers (28 mg, 0.073 mmol, 38%), and the second diastereomer **11e2,** as a brown solid (13 mg, 0.033 mmol, 18%), and in addition, 4-amino[2.2]paracyclophane **12e** was obtained as a solid (10 mg, 0.044 mmol, 24%).

4-Amino-13-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11e1

Diastereomer 1, relative stereochemistry has not been determined.

R_f : 0.4 (Et_3N:EtOAc:Hexanes 2:12:86)

$[\alpha]_D$: No consistent value could be obtained between samples.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.35-7.32 (2H, m), 7.29-7.22 (3H, m), 7.13 (1H, s), 6.61 (1H, d, J = 7.8 Hz), 6.42 (1H, d, J = 8.1 Hz), 6.35 (1H, d, J = 7.6 Hz), 6.17 (1H, d, J = 7.7 Hz), 5.49 (1H, s), 4.60-4.54 (1H, m), 4.31-4.24 (2H m), 4.10 (1H, t, J = 8.2 Hz), 3.31 (1H, dd, J = 13.7,

4.5 Hz), 3.19 (1H, ddd, *J* = 12.0, 9.8, 1.9 Hz), 3.11 (1H, td, *J* = 12.3, 2.5 Hz), 3.04-2.97 (2H, m), 2.93-2.87 (2H, m), 2.76-2.70 (2H, m).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 165.57, 146.48, 140.60, 140.13, 138.49, 138.14, 136.01, 134.87, 134.53, 132.35, 129.34, 128.62, 125.12, 124.17, 126.57, 122.44, 121.32, 71.12, 67.91, 41.56, 34.82, 34.66, 32.14, 31.08.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{26}H_{27}N_2O$ 383.2118; found 383.2114.

4-Amino-13-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11e2

Diastereomer 2, relative stereochemistry not determined

R_f : 0.3 (Et_3N:EtOAc:Hexanes 2:12:86)

$[\alpha]_D^{20}$: –77.78 (*c* = 0.90, $CHCl_3$)

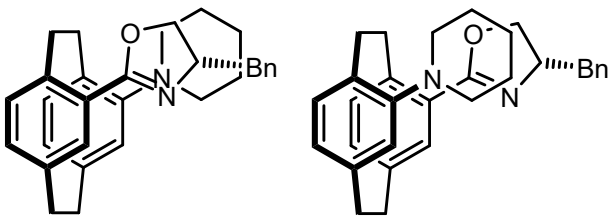
IR: ν_{max} 3745, 3020, 2938, 2400, 1312 cm^{-1}.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.41-7.34 (4H, m), 7.13 (1H, dt, *J* = 8.5, 3.2 Hz), 7.09 (1H, s), 6.62 (1H, dd, *J* = 8.1, 2.2 Hz), 6.41 (1H, d, *J* = 7.8 Hz), 6.34 (1H, d, *J* = 7.7 Hz), 6.17 (1H, dd, *J* = 7.6, 1.6 Hz), 5.41 (1H, s), 4.66 (1H, dtd, *J* = 12.3, 7.7, 4.6 Hz), 4.39 (1H, t, *J* = 8.6 Hz), 4.34-4.29 (1H, m), 4.18 (1H, t, *J* = 7.8 Hz), 3.18 (2H, dd, *J* = 13.7, 4.6 Hz), 3.11-2.87 (8H, m), 2.75 (1H, ddd, *J* = 13.6, 10.2, 5.5 Hz).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 165.22, 146.36, 140.55, 140.13, 138.33, 137.90, 136.00, 134.84, 134.28, 132.17, 129.95, 128.55, 126.66, 125.09, 123.99, 122.16, 121.16, 70.26, 67.65, 40.84, 34.81, 34.64, 32.34, 30.72.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{26}H_{27}N_2O$ 383.2118; found 383.2114.

(R_p,*S*)-4-(Piperidin-1′-yl)-13-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-(piperidin-1′-yl)-13-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11f



(S_p,*S*)-11f (R_p,*S*)-11f

General conditions **A** were followed using (±)-4-bromo-13-(piperidin-1′-yl)-[2.2]paracyclophane **10f** (36 mg, 0.097 mmol, 1.0 eq.), (4*S*)-4-benzyloxazoline **6a** (17 mg, 0.017 mmol, 1.1 eq.), LiO^{t}Bu (20 mg, 0.24 mmol, 2.5 eq.), (*t*-Bu)$_2$P(O)H (2 mg, 0.009 mmol, 0.1 eq.), $Pd(OAc)_2$ (1 mg, 0.004 mmol, 0.05 eq.), DMA (0.39 ml) for 24 hrs.

Purification: Preparative TLC [SiO_2, Et_3N:EtOAc:Hexanes (2:09:89)] to obtain **11f1**, as a brown solid (17 mg, 0.037 mmol, 39%), followed by a mixture of diastereomers (14 mg, 0.031 mmol, 31%), and a diastereomer **11f2,** as a brown solid (11 mg, 0.024 mmol, 24%).

4-(Piperidin-1′-yl)-13-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11f1

Diastereomer 1, relative stereochemistry not determined

R_f : 0.5 (Et_3N:EtOAc:Hexanes 2:09:89)

$[\alpha]_D^{20}$: +50.91 (c = 1.1, $CHCl_3$)

IR: ν_{max} 3681, 3019, 2931, 2400, 2183, 1517 cm^{-1}.

^1H NMR ($CDCl_3$, 500 MHz): δ 7.36-7.31 (2H, m), 7.29-7.25 (3H, m), 7.04 (1H, s), 6.66 (1H, d, J = 7.7 Hz), 6.61 (1H , d, J = 7.7 Hz), 6.56 (1H, d, J = 7.5 Hz), 6.33 (1H, d, J = 7.4 Hz), 5.75 (1H, s), 4.71 (1H, ddd, J = 13.9, 9.2, 4.7 Hz), 4.51 (1H, tt, J = 13.6, 5.3 Hz), 4.32 (1H, t, J = 8.8 Hz), 3.97 (1H, t, J = 8.5 Hz), 3.58 (1H, tt, J = 13.3, 4.9 Hz), 3.42 (1H, dd, J = 13.8, 4.5 Hz), 3.13-3.08 (1H, m), 3.07-3.02 (1H, m), 3.01-2.92 (3H, m), 2.85-2.74 (5H, m), 2.66 (1H, dd, J = 13.6, 9.8 Hz), 1.80-1.71 (2H, m), 1.65-1.56 (2H, m), 1.56-1.48 (2H, m).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 163.31, 152.60, 142.03, 140.05, 138.81, 137.99, 135.81, 135.74, 134.75, 132.29, 131.58, 129.14, 128.59, 126.43, 126.30, 119.01, 70.91, 68.67, 53.40, 42.15, 35.04, 34.80, 34.21, 34.19, 26.60, 24.53.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd $C_{31}H_{35}N_2O$ 451.2744 for; Found 451.2741.

4-(Piperidin-1′-yl)-13-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11f2

Diastereomer 2, relative stereochemistry not determined

R_f : 0.4 (Et_3N:EtOAc:Hexanes 2:09:89)

$[\alpha]_D^{20}$: Too wide range of values between successive measurements.

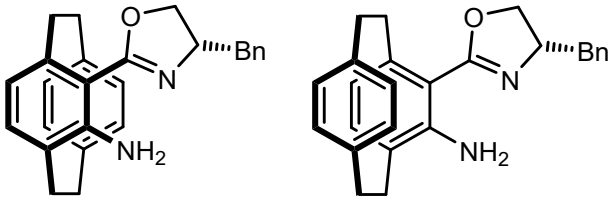
IR: ν_{max} 3680, 3019, 2929, 2165, 2087 cm^{-1}.

^1H NMR ($CDCl_3$, 500 MHz): δ 7.38-7.32 (2H, m), 7.31-7.24 (3H, m), 7.19 (1H, s), 6.69 (1H, d, J = 7.6 Hz), 6.59 (1H, d, J = 7.7 Hz), 6.54 (1H, d, J = 7.5 Hz), 6.31 (1H, d, J = 7.5 Hz,), 5.86 (1H, s), 4.57 (1H, tt, J = 14.8, 5.6 Hz), 4.36 (1H, t, J = 8.7 Hz), 4.25-4.17 (2H, m), 3.53 (1H, tt, J = 12.6, 4.7 Hz), 3.30 (1H, dd, J = 13.6, 4.9 Hz), 3.19-3.10 (1H, m), 3.10-3.01 (1H, m), 2.99-2.80 (8H, m), 2.74 (1H, dd, J = 13.6, 9.4 Hz), 1.78-1.72 (2H, m), 1.63-1.57 (2H, m), 1.54-1.51 (2H, m).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 164.67, 152.76, 141.47, 140.06, 138.45, 138.38, 135.85, 135.38, 134.65, 132.49, 131.87, 129.22, 128.61, 126.63, 126.45, 125.14 118.84, 71.24, 67.74, 53.31, 41.91, 35.28, 35.07, 34.76, 33.40, 26.46, 24.46.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{31}H_{35}N_2O$ 451.2744; found 454.2741.

(R_p,*S*)-4-Amino-5-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-amino-5-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11g



(S_p,*S*)-11g (R_p,*S*)-11g

General conditions **A** were followed using (±)-4-bromo-5-amino[2.2]paracyclophane **10g** (48 mg, 0.16 mmol, 1.0 eq.), (4*S*)-4-benzyloxazoline **6a** (28 mg, 0.17 mmol, 1.1 eq.), Li*O*^{t}Bu (32 mg, 0.39 mmol, 2.5 eq.), (*t*-Bu)$_2$P(O)H (3 mg, 0.015 mmol, 10 mol%), $Pd(OAc)_2$ (2 mg, 0.0079 mmol, 5 mol%), DMA (0.64 ml) for 24 hrs.

Purification: Preparative TLC [SiO_2, EtOAc:Hexanes 15:85] to obtain the separate diastereomer **11g1**, yellow solid (10 mg, 0.026 mmol, 16%) and **11g2**, yellow solid (11 mg, 0.028 mmol, 18%).

The samples decomposed during data collection.

4-Amino-5-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11g1

Diastereomer 1, relative stereochemistry not determined

R_f: 0.6 (EtOAc:Hexanes 15:85)

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.37-7.31 (4H, m), 7.25-7.24 (1H, m), 7.08 (1H, dd, J = 7.7, 1.5 Hz), 6.63 (1H, d, J = 7.8 Hz), 6.43 (1H, d, J = 7.8 Hz), 6.38 (2H, dd, J = 6.7, 1.7 Hz), 6.14 (1H, d, J = 7.6 Hz,), 4.65-4.58 (1H, m), 4.27-4.18 (2H, m), 4.04 (1H, dd, J = 8.2, 6.3 Hz), 3.85-3.76 (1H, m), 3.11-3.03 (4H, m), 2.75-2.63 (4H, m).

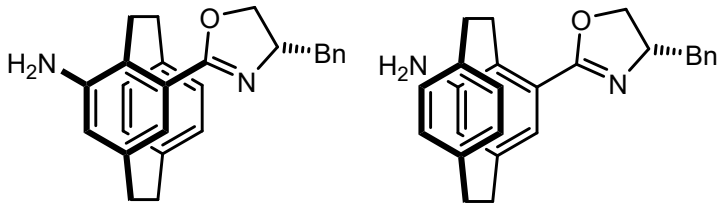
4-Amino-5-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11g2

Diastereomer 2, relative stereochemistry not determined

R_f : 0.5 (EtOAc:Hexanes 15:85)

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.40-7.34 (4H, m), 7.32-7.28 (1H, m), 7.10 (1H, dd, J = 7.7, 1.6 Hz), 6.63 (1H, dd, J = 7.8, 1.4 Hz), 6.43 (1H, dd, J = 7.7, 1.7 Hz), 6.38 (2H, d, J = 7.6 Hz), 6.12 (1H, d, J = 7.6 Hz), 4.62-4.54 (1H, m), 4.27 (1H, t, J = 8.4 Hz), 4.05 (1H, t, J = 8.2 Hz), 3.82-3.75 (1H, m), 3.25 (1H, dd, J = 13.7, 6.0 Hz), 3.13-3.05 (4H, m), 2.94 (1H, dd, J = 13.7, 7.6 Hz), 2.75-2.64 (3H, m).

(R_p,*S*)-4-Amino-8-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-amino-8-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane 11h



(S_p,*S*)-11h **(R_p,*S*)-11h**

General conditions **D** were followed using (±)-4-bromo-8-amino[2.2]paracyclophane **10h** (50 mg, 0.17 mmol, 1.0 eq.), (4*S*)-4-benzyloxazoline **6a** (30 mg, 0.18 mmol, 1.1 eq.), LiO^tBu (33 mg, 0.41 mmol, 2.5 eq.), $(Ad)_2P(O)H$ (6 mg, 0.02 mmol, 0.1 eq.), $Pd(OAc)_2$ (2 mg, 0.08 mmol, 0.05 eq.), DMA (1.0 mL) for 15 hrs.

Purification: Isocratic column chromatography [SiO_2, EtOAc: Hexanes (20:80)] to obtain the first diastereomer, **11h1**, as an off-white solid (10 mg, 0.026 mmol, 15%), a mixture of diastereomers (4 mg, 0.010 mmol, 6%), and the second diastereomer **11h2**, as an off-white solid (12 mg, 0.031 mmol, 18%).

4-Amino-8-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane 11h1

Diastereomer 1, relative stereochemistry not determined

R_f : 0.3 (EtOAc:Hexanes 20:80)

IR: ν_{max} 2920, 2357, 1651, 1495, 1289, 1255, 1017 cm^{-1}.

$[\alpha]_D^{20}$: –63.3 (c = 0.6, $CHCl_3$).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.41-7.32 (4H, m), 7.28-7.23 (1H, m), 7.15 (1H, d, J = 7.6 Hz), 6.93 (1H, s), 6.63 (1H, d, J = 7.6 Hz), 6.52-6.45 (2H, m), 5.45 (1H, s), 4.64-4.53 (1H, m), 4.29

(1H, t, J = 8.7 Hz), 4.23-4.13 (2H, m), 3.77 (2H, bs), 3.33 (1H, dd, J = 13.6 Hz, 4.8 Hz), 3.17-3.00 (5H, m), 2.81 (1H, dd, J = 13.5, 8.9 Hz), 2.75-2.63 (1H, m), 2.62-2.52 (1H, m).

^{13}C NMR ($CDCl_3$, 176 MHz): δ 139.61, 138.84, 132.78, 131.34, 131.22, 129.38, 129.25, 128.99, 128.61, 127.42, 126.51, 124.04, 123.08, 70.87, 35.64, 34.90, 32.55, 31.93, 31.86, 29.71.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{26}H_{27}N_2O$ 383.2118; found 383.2113.

4-Amino-8-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane 11h2

Diastereomer 2, relative stereochemistry not determined

R_f : 0.2 (EtOAc:Hexanes 20:80)

IR: ν_{max} 2921, 1615, 1584, 1347, 1262 cm^{-1}.

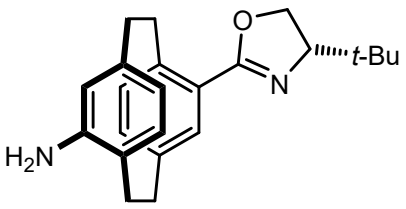
$[\alpha]_D^{20}$: +45 (c = 0.2, $CHCl_3$).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.41-7.34 (4H, m), 7.28-7.22 (1H, m), 7.13 (1H, d, J = 7.6 Hz), 6.91 (1H, s), 6.60 (1H, d, J = 7.5 Hz), 6.52-6.41 (2H, m), 5.44 (1H, s), 4.68-4.57 (1H, m), 4.34 (1H, t, J = 8.6 Hz), 4.13-4.04 (2H, m), 3.76 (2H, bs), 3.33 (1H, dd, J = 13.6 Hz, 4.4 Hz), 3.17-3.04 (4H, m), 3.02-2.93 (1H, m), 2.81 (1H, d, J = 13.4, 9.2 Hz), 2.74-2.64 (1H, m), 2.63-2.54 (1H, m).

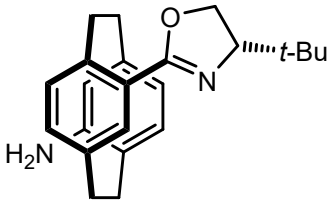
^{13}C NMR ($CDCl_3$, 126 MHz): δ 139.16, 138.84, 137.87, 132.77, 131.41, 131.14, 129.51, 129.37, 128.58, 127.41, 126.56, 123.91, 123.24, 71.24, 35.68, 35.01, 32.55, 31.93, 31.86, 29.70.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{26}H_{27}N_2O$ 383.2118; found 383.2114.

(R_p,*S*)-4-Amino-16-(4′-*tert*-butyl-oxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-amino-16-(4′-*tert*-butyl-oxazolin-2′-yl)[2.2]paracyclophane 11i



(R_p,*S*)-11i



(S_p,*S*)-11i

General conditions **D** were followed using (±)-4-bromo-16-amino[2.2]paracyclophane **10i** (200 mg, 0.66 mmol, 1.0 eq.), (4*S*)-4-*tert*-butyloxazoline **6c** (93 mg, 0.73 mmol, 1.1 eq.),

LiO^{t}Bu (133 mg, 1.66 mmol, 2.5 eq.), $(Ad)_2P(O)H$ (21 mg, 0.066 mmol, 0.1 eq.), $Pd(OAc)_2$ (7 mg, 0.033 mmol, 0.05 eq.), DMA (2.65 ml) for 15 hrs.

Purification: Flash chromatography [SiO_2, EtOAc:Hexanes (20:80)] to obtain first diastereomer, **11i1**, pale yellow solid (30 mg, 0.057 mmol, 13%), a mixture of diastereomers (78 mg, 0.21 mmol, 31%) and separate diastereomer **11i2,** pale yellow solid (31 mg, 0.068 mmol, 14%).

4-Amino-16-(4′-*tert*-butyl-oxazolin-2′-yl)[2.2]paracyclophane 11i1

Diastereomer 1, relative stereochemistry not determined

R_f : 0.3 (EtOAc:Hexanes 10:90)

$[\alpha]_D^{20}$: -32.11 (c = 0.9, $CHCl_3$).

IR: ν_{max} 3433, 2946, 1637, 1475, 1346, 1283, 1021, 800 cm^{-1}.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.28 (1H, d, J = 10.6 Hz), 6.98 (1H, s), 6.43 (1H, d, J = 7.7 Hz), 6.26 (1H, d, J = 7.6 Hz), 6.18 (1H, d, J = 7.6 Hz), 5.43 (1H, s), 4.37 (1H, t, J = 8.8 Hz), 4.22-4.09 (3H, m), 3.49 (2H, bs), 3.17-2.96 (5H, m), 2.94 (1H, t, J = 11.3 Hz), 2.83-2.68 (2H, m), 1.04 (9H, s).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 163.76, 144.85, 141.27, 140.43, 138.60, 134.96, 134.37, 134.15, 129.19, 128.04, 124.37, 122.24, 121.47, 76.50, 67.97, 35.52, 34.13, 33.97, 32.59, 31.40, 26.08.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{23}H_{29}N_2O$ 349.2280; found 349.2274.

4-Amino-16-(4′-tert-butyl-oxazolin-2′-yl)[2.2]paracyclophane 11i2

Diastereomer 2, relative stereochemistry not determined

R_f : 0.2 (EtOAc:Hexanes 10:90)

$[\alpha]_D^{20}$: -76.25 (c = 0.8, $CHCl_3$).

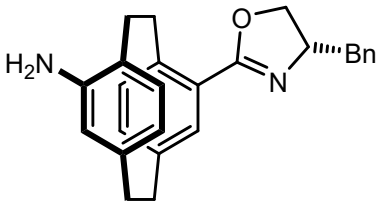
IR: ν_{max} 3338, 2947, 1640, 1500, 1361, 1302, 1066, 986 cm^{-1}.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.27 (1H, dd, J = 7.7, 1.7 Hz), 6.95 (1H, s), 6.45 (1H, d, J = 7.7 Hz), 6.28 (1H, d, J = 7.6 Hz), 6.13 (1H, dd, J = 7.6, 1.4 Hz), 5.44 (1H, d, J = 1.1 Hz), 4.43 (1H, ddd, J = 12.4, 10.1, 1.8 Hz), 4.33 (1H, dd, J = 10.1, 8.5 Hz), 4.24 (1H, t, J = 8.4 Hz), 4.11 (1H, dd, J = 9.9, 1.8 Hz), 3.52 (2H, bs), 3.16-3.08 (2H, m), 3.08-2.97 (2H, m), 2.94 (1H, td, J = 12.8, 2.3 Hz), 2.87 (1H, dd, J = 12.2, 5.3 Hz), 2.77-2.68 (1H, m), 1.10 (9H, s).

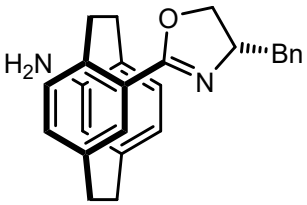
^{13}C NMR ($CDCl_3$, 126 MHz): δ 163.36, 144.88, 141.28, 140.50, 138.59, 134.56, 134.52, 134.39, 129.07, 128.15, 124.38, 122.18, 121.53, 76.50, 67.58, 34.94, 34.33, 34.11, 32.65, 31.49, 26.38.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{23}H_{29}N_2O$ 349.2280; found 349.2274.

(R_p,*S*)-4-Amino-15-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-amino-15-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane 11j



(R_p,*S*)-11j



(S_p,*S*)-11j

General conditions **D** were followed using (±)-4-amino-15-bromo[2.2]paracyclophane (150 mg, 0.50 mmol, 1.0 eq.), (4*S*)-4-benzyl-2-oxazoline **6a** (88 mg, 0.54 mmol, 1.1 eq.), LiO^{t}Bu (99 mg, 1.24 mmol, 2.5 eq.), $(Ad)_2P(O)H$ (16 mg, 0.049 mmol, 10 mol%), $Pd(OAc)_2$ (6 mg, 0.025 mmol, 5 mol%), DMA (2.0 ml) for 13 hrs .

Purification: Flash chromatography [SiO_2, EtOAc:Hexanes (20:80)] to obtain the title compound **11j** as an inseparable mixture of diastereomers, **(R_p,*S*)-11j1** and **(S_p,*S*)-11j2**, yellow-white solid (78 mg, 0.20 mmol, 41%).

R_f : 0.4 (EtOAc:Hexanes 20:80)

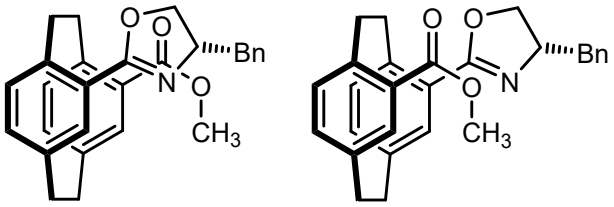
IR: ν_{max} 3414, 3380, 2922, 2854, 2601, 2461, 1633, 1599, 1494, 1452, 1428, 1362, 1179, 1034, 990, 702 cm^{-1}.

^{1}H NMR (MeOD-d_4, 500 MHz): δ 7.34-7.32 (8H, m), 7.25-7.22 (4H, m), 7.10 (1H, d, J = 1.6 Hz), 7.07 (1H, d, J = 1.6 Hz), 6.53 (1H, s), 6.52 (1H, s), 6.24 (1H, d, J = 7.6 Hz), 6.15 (1H, d, J = 7.6 Hz), 6.00 (1H, dd, J = 7.7, 1.4 Hz), 5.95 (1H, dd, J = 7.5, 1.4 Hz), 5.50 (2H, s), 4.62-4.54 (2H, m), 4.40 (2H, q, J = 8.8 Hz), 4.25-4.19 (2H, m), 3.91-3.85 (1H, m), 3.82-3.76 (1H, m), 3.18-2.77 (17 H, m), 2.65-2.51 (2H, m).

^{13}C NMR (MeOD-d_4, 176 MHz): δ 166.01, 165.59, 146.65, 146.59, 140.86, 140.75, 140.47, 140.44, 139.00, 138.97, 137.90, 137.67, 134.36, 133.29, 132.93, 129.40, 129.33, 129.27, 129.11, 128.18, 128.17, 128.01, 127.89, 126.19, 126.16, 123.84, 121.52, 121.49, 121.42, 121.40, 71.07, 70.99, 67.15, 66.83, 41.38, 40.93, 38.39, 38.27, 38.15, 38.03, 37.91, 34.29, 32.57, 32.38, 31.19, 31.17.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{26}H_{27}N_2O$ 383.2118; found 383.2110.

(R_p,*S*)-4-Carbomethoxy-13-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-carbomethoxy-13-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane 11k



(S_p,*S*)-11k (R_p,*S*)-11k

General conditions **A** were followed using (±)-4-bromo[2.2]paracyclophane-13-methyl ester **10k** (75 mg, 0.22 mmol, 1.0 eq.), (4*S*)-4-benzyl-2-oxazoline **6a** (39 mg, 0.24 mmol, 1.1 eq.), LiO^tBu (44 mg, 0.54 mmol, 2.5 eq.), (t-Bu)$_2$P(O)H (4 mg, 0.022 mmol, 0.1 eq.), $Pd(OAc)_2$ (2 mg, 0.010 mmol, 0.05 eq.), DMA (0.87 ml) for 24 hrs.

Purification: Flash chromatography [SiO_2, EtOAc:Hexanes (0-10 to 30:70, gradient elution)] to obtain first diastereomer, **11k1**, white solid (6 mg, 0.037 mmol, 6%), a mixture of diastereomers (11 mg, 0.031 mmol, 12%), and the second diastereomer **11k2**, brown solid (5 mg, 0.011 mmol, 5%). Debromo ester **(±)-12k**, was obtained as a white solid (9 mg, 0.033 mmol, 16%).

4-Carbomethoxy-13-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane 11k1

Diastereomer 1, relative stereochemistry not determined

R_f : 0.7 (EtOAc:Hexanes 30:70)

$[\alpha]_D^{20}$: No consistent reading between successive measurements.

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.31 (2H, m), 7.29-7.28 (1H, m), 7.26-7.22 (2H, m), 7.18 (1H, d, J = 1.8 Hz), 7.02 (1H, d, J = 1.5 Hz), 6.71-6.61 (4H, m), 4.63 (1H, ddd, J = 14.2, 9.2, 4.9 Hz), 4.42 (1H, td, J = 12.6, 3.7 Hz), 4.31 (1H, t, J = 8.4 Hz), 4.20 (1H, td, J = 12.8, 7.2 Hz), 4.02 (1H, t, J = 8.1 Hz), 3.80 (3H s), 3.31 (1H, dd, J = 13.6, 4.9 Hz), 3.18-3.00 (6H, m), 2.70 (1H, dd, J = 13.8, 9.6 Hz).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 167.28, 163.30, 142.76, 141.31, 139.35, 139.08, 138.48, 136.41, 135.99, 135.63, 134.89, 134.01, 132.84, 129.78, 129.18, 128.56, 128.03, 126.43, 70.98, 68.66, 51.47, 41.86, 34.70, 34.57, 34.48.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd $C_{28}H_{28}NO_3$ 426.2064 for; Found 426.2060.

4-Carbomethoxy-13-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane 11k2

Diastereomer 2, relative stereochemistry not determined

R_f : 0.6 (EtOAc:Hexanes 30:70)

$[\alpha]_D^{20}$: Inconsistent values between successive measurements.

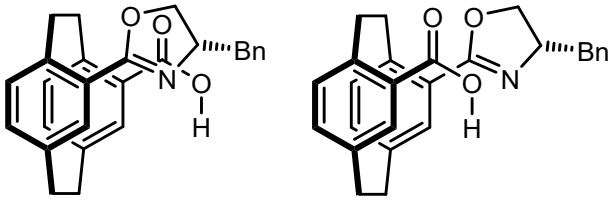
IR: ν_{max} 3680, 2971, 2325, 2167, 1707, 1522 1410 cm^{-1}.

^1H NMR ($CDCl_3$, 400 MHz): δ 7.36-7.28 (4H, m), 7.25-7.21 (1H, m), 7.11 (1H, s), 6.72 (1H, dd, J = 8.2, 2.3 Hz), 6.66-6.56 (4H, m), 4.56-4.47 (1H, m), 4.34 (1H, t, J = 8.5 Hz), 4.25 (2H, td, J = 10.9, 3.5 Hz), 4.13 (1H, t, J = 6.8 Hz), 3.85 (3H, s), 3.33 (1H, dd, J = 13.6, 5.3 Hz), 3.15-3.07 (5H, m), 3.01-2.96 (1H, m) 2.83 (1H, dd, J = 13.6, 9.1 Hz).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 166.94, 164.26, 143.19, 141.14, 139.54, 139.06, 138.45, 136.37, 135.99, 135.68, 135.21, 134.14, 132.91, 129.34, 129.24, 128.59, 128.09, 126.42, 71.47, 67.95, 51.53, 41.39, 34.85, 34.70, 34.16.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{28}H_{28}NO_3$ 426.2064; found 426.2060.

(R_p,*S*)-4-Carboxy-13-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-carboxy-13-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11l



(***S*** $_p$,***S***)-**11l** (***R*** $_p$,***S***)-**11l**

General conditions **A** were followed using (±)-4-bromo[2.2]paracyclophane-13-carboxylic acid **10l** (50 mg, 0.15 mmol, 1.0 eq.), (4*S*)-4-benzyl-2-oxazoline **6a** (27 mg, 0.16 mmol, 1.1 eq.), LiO^{t}Bu (30 mg, 0.38 mmol, 2.5 eq.), (t-Bu)$_2$P(O)H (2 mg, 0.015 mmol, 0.1 eq.), $Pd(OAc)_2$ (2 mg, 0.0075 mmol, 0.05 eq.), DMA (0.6 ml) for 24 hrs.

Purification: Flash chromatography [SiO_2, EtOAc:Hexanes 40:60] to obtain the title compound as an inseparable mixture of diastereomers, **(R_p,*S*)-11l** and **(S_p,*S*)-11l** , as a white semisolid (27 mg, 0.065 mmol, 44%), and 4-carboxylic acid **(±)-12l**, as a white solid (7 mg, 0.027 mmol, 18%).

R_f : 0.3 (EtOAc:Hexanes 40:60)

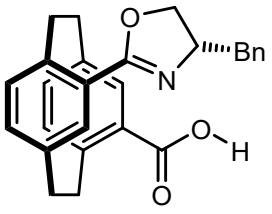
IR: ν_{max} 3650, 2973, 2196, 2007, 1683, 1419 cm^{-1}.

^1H NMR ($CDCl_3$, 500 MHz): δ 12.38 (1H, bs), 7.52 (1H, s), 7.31 (1H, s), 7.23 (2H, d, J = 7.2 Hz), 7.20-7.12 (4H, m), 7.10-7.02 (6H, m), 6.80 (2H, dd, J = 17.1, 7.7 Hz), 6.73-6.66 (6H, m), 4.75-4.66 (1H, m), 4.63 (1H, ddd, J = 13.8, 9.1, 4.6 Hz), 4.49-4.42 (1H, m), 4.41 (2H, t, J = 9.4 Hz), 4.29-4.19 (3H, m), 4.17 (1H, t, J = 7.3 Hz), 3.89 (1H, t, J = 8.2 Hz), 3.35 (2H, ddd, J = 18.4, 13.6, 3.9 Hz), 3.20-3.14 (5H, m), 3.14-3.08 (5H, m), 3.01-2.95 (2H, m), 2.65 (2H, tt, J = 14.2, 9.7 Hz).

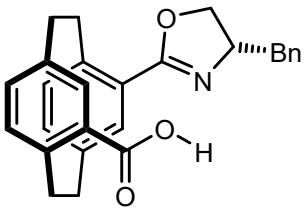
^{13}C NMR ($CDCl_3$, 126 MHz): δ 172.58, 171.02, 165.89, 163.47, 143.92, 143.13, 141.58, 141.34, 139.78, 139.61, 139.35, 139.21, 138.15, 137.26, 136.36, 136.26, 135.93, 135.83, 135.64, 135.01, 134.92, 134.77, 133.12, 133.00, 129.27, 128.88, 128.44, 128.34, 126.31, 126.11, 71.90, 71.12, 68.03, 66.32, 41.49, 41.16, 34.91, 34.91, 34.75, 34.70, 34.48, 34.49, 34.12.

HRMS (ESI-TOF) m/z: $[M - H]^+$ Calcd. for $C_{27}H_{24}NO_3$ 410.1751; found 410.1761.

(R_p,*S*)-4-Carboxy-12-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane and (S_p,*S*)-4-carboxy-12-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11n



(S_p,*S*)-11n



(R_p,*S*)-11n

General conditions **A** were followed using (±)-4-bromo[2.2]paracyclophane-12-carboxylic acid **10n** (50 mg, 0.15 mmol, 1.0 eq.), (4*S*)-4-benzyl-2-oxazoline **6a** (27 mg, 0.16 mmol, 1.1 eq.), LiO^{t}Bu (30 mg, 0.38 mmol, 2.5 eq.), (t-Bu)$_2$P(O)H (2 mg, 0.015 mmol, 10 mol%), $Pd(OAc)_2$ (2 mg, 0.0075 mmol, 5 mol%), DMA (0.6 ml) for 22 hrs.

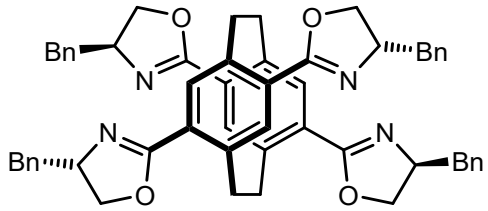
Purification: Flash chromatography [SiO_2, EtOAc:Hexanes 40:60] to obtain the title compound as an inseparable mixture of diastereomers, **(R_p,*S*)-11n** and **(S_p,*S*)-11n**, as a white semisolid (41 mg, 0.093 mmol, 61%).

R_f : 0.3 (EtOAc:Hexanes 40:60)

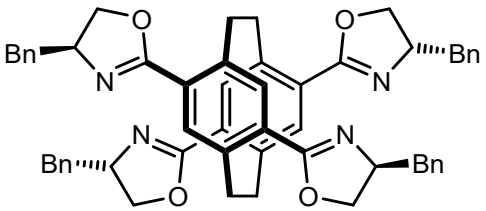
IR: ν_{max} 3647, 2980, 2906, 2240, 2043, 1713, 1523 cm^{-1}.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.42-7.23 (10H, m), 7.16 (2H, d, J = 4.1 Hz), 6.94 (2H, d, J = 9.1 Hz), 6.81 (2H, d, J = 7.9 Hz), 6.66-6.59 (6H, m), 4.83-4.79 (1H, m), 4.76 (1H, ddd, J = 13.6, 8.9, 4.4 Hz), 4.54 (2H, dd, J = 8.8, 6.6 Hz) , 4.37 (2H, t, J = 8.5 Hz), 4.02-3.95 (2H, m), 3.74-3.66 (2H, m), 3.59 (1H, dd, J = 13.9, 4.3 Hz), 3.31 (1H, dd, J = 13.8, 4.5 Hz), 3.22-3.12 (5H, m), 3.11-3.08 (1H, m), 3.04-2.96 (3H, m,), 2.93-2.86 (3H, m,), 2.84-2.73 (2H, m).
^{13}C NMR ($CDCl_3$, 126 MHz): δ 169.48, 169.30, 168.81, 168.58, 140.93, 140.83, 140.02, 139.73, 139.02, 138.93, 136.84, 136.57, 135.97, 135.92, 135.56, 135.51, 134.99, 134.97, 133.04, 132.97, 129.49, 129.26, 129.06, 129.03, 128.94, 128.71, 127.87, 127.85, 127.04, 126.90, 73.91, 72.64, 66.48, 65.67, 41.06, 34.76, 34.74, 34.48, 34.44, 34.29, 33.95, 33.93.
HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{27}H_{26}NO_3$ 412.1907; found 412.1901.

(*S*, *S*,*R*$_p$,*S*,*S*)-4,7,12,15-Tetra(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane and (*S*,*S*,*S*$_p$,*S*,*S*)-4,7,12,15-tetra(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane 11o



(*S*,*S*,*R*$_p$,*S*,*S*)-11o



(*S*,*S*,*S*$_p$,*S*,*S*)-11o

General conditions C was followed using (±)-4,7,12,15-tetrabromo[2.2]paracyclophane **10o** (100 mg, 0.19 mmol, 1.0 eq.), (4*S*)-4-benzyl-2-oxazoline **6a** (136 mg, 0.85 mmol, 4.4 eq.), LiO^{t}Bu (154 mg, 1.92 mmol, 10.0 eq.), (t-Bu)$_2$P(O)H (12.4 mg, 0.077 mmol, 40 mol%), $Pd(OAc)_2$ (9 mg, 0.038 mmol, 20 mol%), DMA (1.1 ml) for 15 hrs.

Purification: Isocratic column chromatography [SiO_2, EtOAc:Hexanes (20:80)] to obtain the title compound **11o** as two diastereomers, **11o1**, as an off-white solid (9 mg, 0.010 mmol, 5% yield), and the second diastereomer **11o2**, as an off-white solid (9 mg, 0.010 mmol, 5% yield), along with two separable diastereomers of *para*-dibromo-*para*-bis(oxazoline) **11p1** (10 mg, 0.015 mmol, 8%) and 2nd diastereomer **11p2** (9 mg, 0.013 mmol, 7%), as well as an inseparable mixture of diastereomers of debromo-tris(oxazoline) **11q** (4 mg, 0.006 mmol, 3%).

General conditions E was followed using (±)-4,7,12,15-tetrabromo[2.2]paracyclophane **10o** (100 mg, 0.19 mmol, 1.0 eq.), (4*S*)-4-benzyl-2-oxazoline **6a** (136 mg, 0.85 mmol, 4.4 eq.),

LiO^{t}Bu (154 mg, 1.92 mmol, 10.0 eq.), $(Ad)_2P(O)H$ (24 mg, 0.077 mmol, 40 mol%), $Pd(OAc)_2$ (9 mg, 0.038 mmol, 20 mol%), DMA (1.1 ml) for 15 hrs.

Purification: Isocratic column chromatography [SiO_2, EtOAc:Hexanes (20:80)] to obtain the title compound **11o** (20 mg, 0.22 mmol, 12% yield) as a separate diastereomer, **11o1**, an off-white solid (10 mg, 0.012 mmol, 6% yield), and a separate diastereomer **11o2**, an off-white solid (10 mg, 0.012 mmol, 6% yield) along with an inseparable mixture of **11q** (13 mg, 0.019 mmol, 10%).

4,7,12,15-Tetra(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane 11o1

Diastereomer 1, relative stereochemistry not determined

R_f : 0.3 (EtOAc:Hexanes 20:80)

IR: ν_{max} 2928, 1628, 1470, 1354, 1275, 1068 cm^{-1}.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.42-7.34 (16H, m), 7.33-7.28 (4H, m), 7.25 (4H, s), 4.69 (4H, quint, J = 7.6 Hz), 4.37 (4H, t, J = 9.5 Hz), 4.22-4.08 (8H, m), 3.41 (4H, dd, J = 13.7, 5.8 Hz), 3.11 (4H, quint, J =7.9 Hz), 2.93 (4H, dd, J = 13.6, 8.5 Hz).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 162.91, 141.45, 138.44, 134.32, 130.23, 129.25, 128.66, 126.54, 70.87, 68.82, 42.18, 33.87.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{56}H_{53}N_4O_4$ 845.4061; found 845.4056.

CD-2 mg/ml sample is prepared in $CDCl_3$.

4,7,12,15-Tetra(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane 11o2

Diastereomer 2, relative stereochemistry not determined

R_f : 0.2 (EtOAc:Hexanes 40:60)

IR: ν_{max} 2923, 1631, 1497, 1349, 1273, 1056 cm^{-1}.

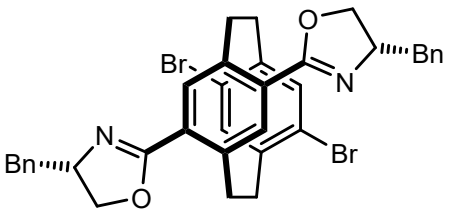
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.37-7.29 (16H, m), 7.28-7.24 (4H, m) 7.21 (4H, s), 4.69 (4H, quint, J = 8.5 Hz), 4.28 (4H, t, J = 8.4 Hz), 4.18 (4H, quint, J = 7.5 Hz), 4.10 (4H, t, J = 7.3 Hz), 3.30 (4H, dd, J = 13.6 Hz, 4.3 Hz), 3.08 (4H, quint, J = 7.1 Hz), 2.73 (4H, dd, J = 12.4, 9.1 Hz).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 163.10, 141.34, 138.27, 134.68, 130.22, 130.22, 128.57, 126.49, 70.89 68.53, 41.87, 34.52.

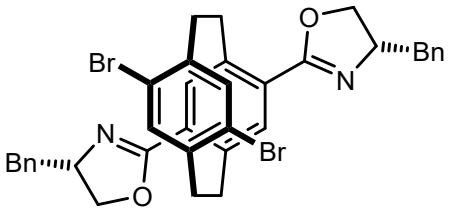
HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{56}H_{53}N_4O_4$ 845.4061; found 845.4052.

CD- 2 mg/ml sample is prepared in $CDCl_3$.

(*S*,R_p,*S*)-4,7-dibromo-12,15-bis(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane and (*S*,S_p,*S*)-4,7-dibromo-12,15-bis(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11p



(*S*,R_p,*S*)-11p



(*S*,S_p,*S*)-11p

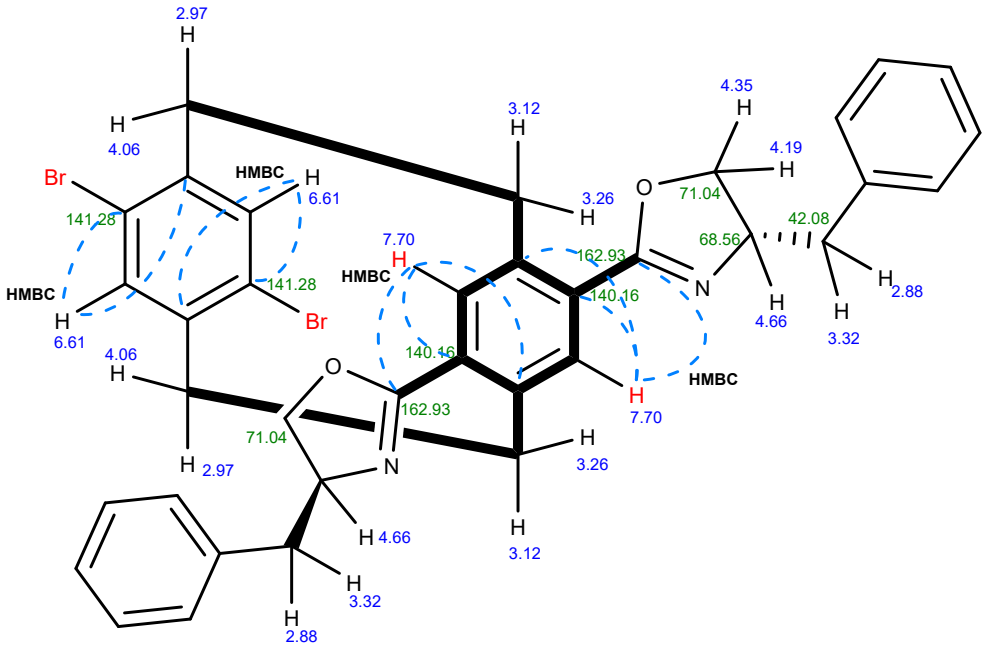


Figure 2. Structure elucidation of 11p (The indicated values are the ppm values obtained in ^{1}H & ^{13}C NMR spectrum and blue dotted lines denote the HMBC correlation).

4,7-Dibromo-12,15-bis(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11p1

The small quantity of this by product made it impossible to get full data.

Diastereomer 1, relative stereochemistry not determined

R_f: 0.5 (EtOAc:Hexanes 20:80)

^{1}H NMR ($CDCl_3$, 700 MHz): δ 7.70 (2H, s), 7.38-7.34 (8H, m), 7.29-7.26 (2H, m), 6.61 (2H, s), 4.66 (2H, quint, J = 8.6 Hz), 4.35 (2H, t, J = 8.5 Hz), 4.19 (2H, t, J = 7.7 Hz), 4.06 (2H, dd, J = 12.4, 10.2 Hz), 3.32 (2H, dd, J = 13.9, 5.5 Hz), 3.26 (2H, dd, J = 13.3, 9.7 Hz), 3.12 (2H, dt, J = 12.7, 7.7 Hz), 2.97 (2H, ddd, J = 12.9, 9.6, 7.6 Hz), 2.88 (2H, dd, J = 13.9, 8.4 Hz).

^{13}C NMR ($CDCl_3$, 176 MHz): δ 162.93, 141.28, 140.16, 138.10, 135.81, 132.80, 130.22, 129.25, 128.88, 128.64, 126.57, 125.31, 71.04, 68.56, 42.08, 33.94, 32.58.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{36}H_{33}Br_2N_2O_2$ 685.0883; found 685.0879.

4,7-Dibromo-12,15-bis(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11p2

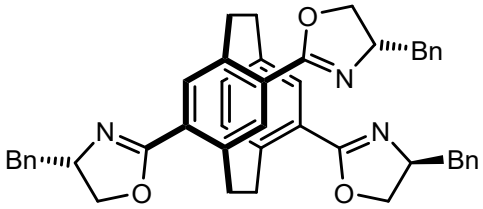
The small quantity of this by product made it impossible to get full data.

Diastereomer 2, relative stereochemistry not determined

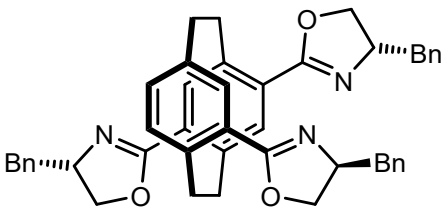
R_f : 0.4 (EtOAc:Hexanes 20:80)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.75 (2H, s), 7.40-7.31 (8H, m), 7.30-7.24 (2H, m), 6.60 (2H, s), 4.70 (2H, q, J = 7.5 Hz), 4.41 (2H, t, J =9.0 Hz), 4.17 (2H, t, J = 8.0 Hz), 4.01 (2H, dd, J = 9.8 Hz, 2.4 Hz), 3.31-3.22 (4H, m), 3.20-3.09 (2H, m), 2.89-2.77 (4H, m).

(*S*,*R*$_p$,*S*,*S*)-4,7,12-tris(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane and (*S*,*S*$_p$,*S*,*S*)-4,7,12-tris(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11q



(*S*,*R*$_p$,*S*,*S*)-11q



(*S*,*S*$_p$,*S*,*S*)-11q

R_f : 0.2 (EtOAc:Hexanes 20:80)

IR: ν_{max} 3059, 2925, 1716, 1634, 1495, 1453, 1261, 1082, 976, 700 cm^{-1}.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.40-7.22 (15H, m), 7.17 (3H, d, J = 10.2 Hz), 6.69-6.55 (2H, m), 4.75-4.62 (3H, m), 4.42 (1H, t, J = 8.8 Hz), 4.30 (3H, dd, J = 17.5, 8.8 Hz), 4.19 (2H, dd, J = 16.2, 7.9 Hz), 4.09 (2H, dd, J = 13.8, 7.0 Hz), 4.03 (1H, dd, J = 12.3, 9.6 Hz), 3.35 (1H, dd, J = 13.7, 5.0 Hz), 3.28 (2H, dd, J = 13.7, 4.0 Hz), 3.20 (2H, m), 3.06 (1H, dt, J = 12.3, 8.9 Hz), 2.92-2.79 (3H, m), 2.73 (2H, dt, J = 13.7, 8.5 Hz).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 163.84, 163.62, 163.07, 141.29, 141.25, 140.86, 139.99, 138.39, 138.28, 138.01, 136.63, 134.89, 134.56, 133.72, 132.25, 130.59, 129.97, 129.41, 129.24, 128.61, 128.55, 128.13, 126.60, 126.60, 126.47, 126.44, 71.36, 70.85, 70.77, 68.54, 68.40, 68.15, 41.94, 41.88, 41.82, 35.62, 35.44, 34.50, 33.81.

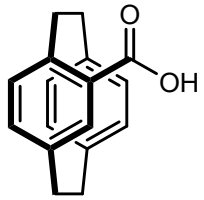
HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd. for $C_{46}H_{44}N_3O_3$ 686.3377; found 686.3357.

Hydrolysis of oxazolines

Conditions G: Aqueous HCl (6M) was added dropwise to a flask containing [2.2]paracyclophane-oxazoline. The resulting mixture was heated to reflux before cooling to room temperature. A solution of aqueous NaOH (40%) was added till the pH of the reaction mixture dropped to 5. The aqueous layer was extracted with CH_2Cl_2 (3 x 10 mL), combined organic layers dried over anhydrous $MgSO_4$, and concentrated under vacuum to afford pure compound.

Conditions H: A pressure tube was charged with [2.2]paracyclophane-oxazoline, H_2SO_4 (3.6 M) and heated at 100 °C for the specified time to control the mono- or bis-hydrolysis. After the completion of the reaction, 10 mL of distilled water was added to the reaction mixture. The diacid by-product was filtered as an off-white product using a Buchner funnel. The filtrate was extracted with CH_2Cl_2 (3 x 30 mL), the combined organic layers were dried over anhydrous $MgSO_4$, and concentrated under vacuum to afford pure compound.

(R_p)-(–)-4-Carboxy[2.2]paracyclophane 13a



(R_p)-13a

General condition H was followed using (R_p,*S*)-4-(4′-*tert*-butyloxazolin-2′-yl)[2.2]paracyclophane **8a** (100 mg, 0.30 mmol, 1.0 eq.), H_2SO_4 (3.6 M, 5 mL) and heated at 100 °C for 12 hrs. After the completion of the reaction, 10 mL of distilled water was added to the reaction mixture. The aqueous layer was extracted with CH_2Cl_2 (3 x 30 mL), combined organic layers dried over anhydrous $MgSO_4$, and concentrated under vacuum before keeping under high vacuum to afford the pure title compound **(R_p)-(–)-13a,** white solid (48 mg, 0.19 mmol, 64%).

R_f : 0.3 (EtOAc:Hexanes 20:80)

IR: ν_{max} 2923, 2852, 1674, 1422, 1300 cm^{-1}.

$[\alpha]_D^{20}$: –146 (c = 0.5, $CHCl_3$) (lit $[\alpha]_D^{23}$ –151.1 (c = 0.5, $CHCl_3$)]

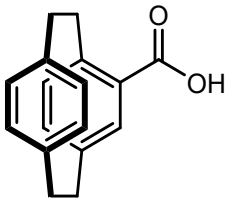
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.32 (1H, s), 6.75 (1H, d, J = 7.7 Hz), 6.66-6.59 (3H, m), 6.55 (2H, d, J = 7.8 Hz), 4.24 (1H, t, J = 10.9 Hz), 3.28-3.17 (4H, m), 3.13-3.03 (2H, m), 2.97-2.89 (1H, m).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 171.99, 143.74, 140.08, 140.04, 139.46, 137.38, 136.39, 136.17, 133.15, 132.79, 132.33, 131.78, 129.61, 36.29, 35.27, 35.11, 34.95.

HRMS (ESI-TOF) m/z: $[M - H]^-$ Calcd $C_{17}H_{15}O_2$ for 251.1067; Found 251.1075.

Data are comparable to that reported in the literature.[5]

(S_p)-(+)-4-Carboxy[2.2]paracyclophane 13a



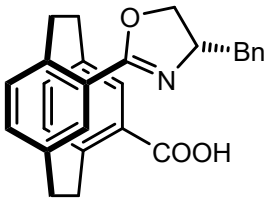
(S_p)-13a

Prepared analogously to (R_p)-(–)-4-carboxy[2.2]paracyclophane **13a** (100 mg, 0.27 mmol, 1.0 eq.) but employing (S_p,*S*)-4-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane **8a**, H_2SO_4 (3.6 M, 5 mL), and heated at 100 °C for 12 hrs to afford the pure title compound **(S_p)-(+)-13a,** white solid (48 mg, 0.19 mmol, 70 %).

$[\alpha]_D^{20}$: +155.1 (c = 0.5, $CHCl_3$) (lit $[\alpha]_D^{23}$ +161 (c = 0.943, $CHCl_3$). The rest of the data were same as the other enantiomer.

Data comparable to that reported in literature.[5]

(S_p,*S*)-(–)-4-Carboxy-12-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11n



(S_p,*S*)-(–)-11n

An ace pressure tube was charged with (R_p,*S*)-(–)-4,12-bis(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane **11b** (100 mg, 0.189 mmol, 1.0 eq.), H_2SO_4 (3.6 M, 5 mL) and heated at 100 °C. The reaction was purposely stopped at 12 hrs. After cooling, 10 mL of distilled water

was added to the reaction mixture. The diacid by-product was filtered as an off-white product using Buchner funnel. The filtrate was extracted with CH_2Cl_2 (3 x 30 mL), combined organic layers dried over anhydrous $MgSO_4$, and concentrated under vacuum.

Purification: Isocratic column chromatography [SiO_2, EtOAc:Hexanes (20:80)] to obtain the title compound **(S_p,*S*)-(–)-11n** as an off-white solid (40 mg, 0.09 mmol, 51%).

R_f : 0.3 (EtOAc:Hexanes 20:80)

$[\alpha]_D^{20}$: –21.83 (c = 1.2, $CHCl_3$)

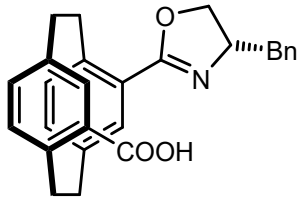
IR: ν_{max} 2920, 2357, 1651, 1495, 1289, 1255, 1017 cm^{-1}.

^1H NMR ($CDCl_3$, 500 MHz): δ 7.37-7.32 (2H, m), 7.31-7.24 (3H, m), 7.12 (1H, s), 6.92 (1H, s), 6.82 (1H, d, J = 7.5 Hz), 6.67-6.56 (3H, m), 4.85 (1H, dtd, J = 14.8, 8.9, 4.5 Hz), 4.55 (1H, t, J = 9.0 Hz), 4.38 (1H, t, J = 8.0 Hz), 4.02 (1H, dd, J = 13.0, 2.5 Hz), 3.70 (1H, t, J = 11.4 Hz), 3.30 (1H, dd, J = 13.7, 4.2 Hz), 3.22-3.06 (3H, m), 3.05-2.97 (1H, m), 2.93-2.83 (2H, m), 2.83-2.75 (1H, m).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 169.29, 168.86, 140.92, 140.69, 139.95, 138.91, 136.46, 135.94, 135.85, 135.59, 135.49, 135.25, 133.04, 129.50, 128.78, 128.72, 127.82, 126.93, 72.75, 65.51, 41.02, 34.71, 34.48, 34.33, 33.82.

HRMS (ESI-TOF) m/z: $[M - H]^-$ Calcd. for $C_{27}H_{24}NO_3$ 410.1751; found 410.1761.

(R_p,*S*)-(+)-4-Carboxy-12-(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane 11n



(R_p,*S*)-(+)-11n

Prepared analogously to (S_p,*S*)-(–)-4-carboxy-12-(4′-benzyloxazolin-2′-yl)-[2.2]paracyclophane **11n** but employing (S_p,*S*)-(+)-4,12-bis(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane **11b** (100 mg, 0.189 mmol, 1.0 eq.) and H_2SO_4 (3.6 M, 5 mL).

Purification: Isocratic column chromatography [SiO_2, EtOAc: Hexanes (20:80)] to obtain the title compound as an off-white solid (43 mg, 0.10 mmol, 56%).

R_f : 0.3 (EtOAc:Hexanes 20:80)

$[\alpha]_D^{20}$: +22 (c = 1, $CHCl_3$).

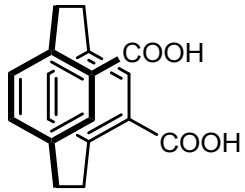
IR: ν_{max} 2927, 1696, 1628, 1452, 1260, 1238, 1189 cm^{-1}.

^1H NMR ($CDCl_3$, 500 MHz): δ 7.42-7.30 (5H, m), 7.15 (1H, s), 6.91 (1H, s), 6.81 (1H, d, J = 7.5 Hz), 6.66-6.59 (3H, m), 4.64 (1H, dtd, J = 13.3, 9.3, 4.2 Hz), 4.53 (1H, t, J = 9.0 Hz), 4.36 (1H, t, J = 8.6 Hz), 3.99 (1H, t, J = 10.9 Hz), 3.68 (1H, t, J = 12.2 Hz), 3.59 (1H, dd, J = 13.8, 3.9 Hz), 3.22-3.11 (2H, m), 3.10-3.05 (1H, m), 3.04-2.95 (2H, m), 2.93-2.86 (1H, m), 2.79-2.69 (1H, m).

^{13}C NMR ($CDCl_3$, 126 MHz): δ 169.16, 168.98, 140.97, 140.76, 139.70, 138.88, 136.79, 135.97, 135.87, 135.59, 135.54, 135.17, 132.97, 129.26, 128.96, 128.88, 127.85, 127.06, 73.26, 66.42, 41.03, 34.71, 34.45, 34.30, 33.90.

HRMS (ESI-TOF) m/z: $[M - H]^+$ Calcd. for $C_{27}H_{24}NO_3$ 410.1751; found 410.1762.

(R_p)-(–)-[2.2]Paracyclophane-4,12-dicarboxylic acid 13c



(R_p)-(–)-13c

General procedure **H** was followed using (R_p,*S*)-(–)-4,12-bis(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane **11ca** (100 mg, 0.189 mmol, 1.0 eq.), H_2SO_4 (3.6 M, 5 mL) for 24 hours to obtain the title compound **(R_p)-(–)-13c** as an off-white product (30 mg, 0.101 mmol, 55%).

R_f : 0.2 (EtOAc:Hexanes 50:50)

$[\alpha]_D^{20}$: –152 (c = 0.25, EtOH) [Lit. $[\alpha]_D^{20}$: –156 (c = 0.25, EtOH)]

IR: ν_{max} 2954, 2658, 1674, 1652, 1422, 1276 cm^{-1}.

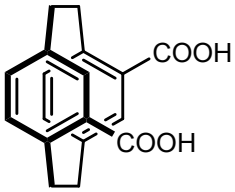
^1H NMR (MeOD-d_4, 500 MHz): δ 7.22 (2H, s), 6.80 (2H, d, J = 7.4 Hz), 6.63 (2H, d, J = 7.8 Hz), 4.14 (2H, t, J = 11.9 Hz), 3.23-3.17 (2H, m), 3.17-3.10 (2H, m), 2.88 (2H, d, J = 7.5 Hz).

^{13}C NMR (MeOD-d_4, 126 MHz): δ 168.94, 142.45, 140.18, 136.20, 135.90, 133.56, 130.52, 35.41, 33.69.

HRMS (ESI-TOF) m/z: $[M - H]^-$ Calcd $C_{18}H_{15}O_4$ for 295.0965; Found 295.0971.

Data comparable to that reported in literature.[6]

(S_p)-(+)-[2.2]Paracyclophane-4,12-dicarboxylic acid 13c



(S_p)-(+)-13c

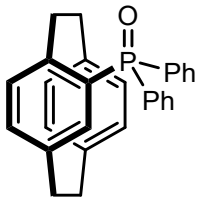
General procedure **H** was followed using (S_p,*S*)-(+)-4,12-bis(4′-benzyloxazolin-2′-yl)[2.2]paracyclophane **11ca** (100 mg, 0.189 mmol, 1.0 eq.), H_2SO_4 (3.6 M, 5 mL) for 24 hours to obtain the title compound **(S_p)-(+)-13c** as an off-white product (30 mg, 0.101 mmol, 55 %).

R_f : 0.2 (EtOAc:Hexanes 50:50)

$[\alpha]_D^{20}$: +155.1 (c = 0.098, EtOH) [Lit. $[\alpha]_D^{20}$: +150 (c = 0.09, EtOH)]. The rest of the data were same as the other enantiomer.

Data comparable to that reported in literature.[6]

(R_p)-(–)-Diphenyl([2.2]paracyclophane-4-yl)phosphine oxide 14



(R_p)-14

A pressure tube was charged with (R_p)-(–)-4-carboxy[2.2]paracyclophane **13a** (40 mg, 0.16 mmol, 1.0 eq.), diphenylphosphine oxide (22 mg, 0.13 mmol, 0.8 eq.), triethylamine (44 µL, 0.32 mmol, 2.0 eq.), $(Boc)_2O$ (48 mg, 0.22 mmol, 1.4 eq.) under argon. In a separate 4 ml scintillation vial, Dppp (13 mg, 0.032 mmol, 0.2 eq.), $Pd(OAc)_2$ (4 mg, 0.016 mmol, 0.1 eq.) were dissolved in 1,4-dioxane (1.0 mL) and transferred to the pressure tube under argon. The reaction mixture was heated at 130 °C for 48 hrs. After cooling, the reaction mixture was diluted with CH_2Cl_2: MeOH (1:1) and filtered. The filtrate was concentrated under reduced pressure.

Purification: Isocratic column chromatography [SiO_2, EtOAc:Hexanes (20:80)] to obtain the title compound **(R_p)-(–)-14** as an off-white solid (4-10 mg, 8-20% in 5 experiments).

R_f : 0.3 (EtOAc:Hexanes 20:80)

$[\alpha]_D^{20}$: –45 (c = 0.2, $CHCl_3$) [Lit. $[\alpha]_D^{32}$: –68.3 (c =1.88, $CHCl_3$)]

IR: ν_{max} 2954, 2658, 1674, 1652, 1422, 1276 cm^{-1}.

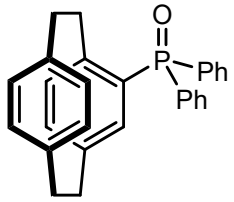
^1H NMR ($CDCl_3$, 500 MHz): δ 7.73 (2H, d, J = 7.1 Hz), 7.62-7.53 (3H, m), 7.53-7.44 (3H, m), 7.43-7.35 (2H, m), 7.20 (1H, d, J = 7.9 Hz), 6.65 (1H, d, J = 7.6 Hz), 6.62-6.50 (3H, m), 6.32-6.24 (2H, m), 3.60-3.48 (2H, m), 3.17-3.07 (2H, m), 3.05 (1H, d, J = 9.7 Hz), 2.99 (1H, ddd, J = 14.2, 11.3, 5.7 Hz), 2.90 (1H, td, J = 12.8, 3.7 Hz), 2.80 (1H, ddd, J = 13.5, 10.9, 7.1Hz).

^{31}P NMR ($CDCl_3$, 200 MHz) δ_P 27.08.

HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd $C_{28}H_{26}OP$ for 409.1716; Found 409.1709.

Data are comparable to that reported in the literature.[7]

(S_p)-(+)-Diphenyl([2.2]paracyclophane-4-yl)phosphine oxide 14



(S_p)-14

Prepared analogously to (R_p)-(–)-diphenyl([2.2]paracyclophane-4-yl)phosphine oxide **14** but employing (S_p)-(+)-4-carboxy[2.2]paracyclophane **13a** (40 mg, 0.16 mmol, 1.0 eq.), diphenylphosphineoxide (32 mg, 0.13 mmol, 0.8 eq.), triethylamine (44 µL, 0.32 mmol, 2.0 eq.), $(Boc)_2O$ (48 mg, 0.22 mmol, 1.4 eq.) Dppp (13 mg, 0.032 mmol, 0.2 eq.), $Pd(OAc)_2$ (4 mg, 0.016 mmol, 0.1 eq.), 1,4-dioxane (1.0 mL) for 48 hrs.

$[\alpha]_D^{20}$: +18.75 (c = 0.16, $CHCl_3$). The rest of the data were same as the other enantiomer.

References

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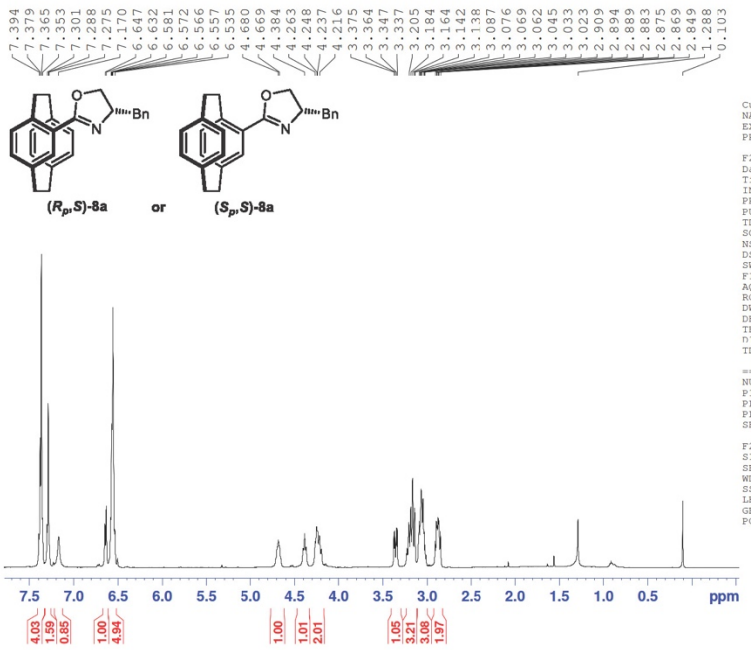

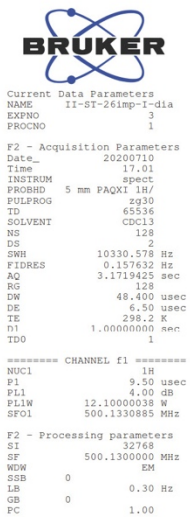


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PROCNO                    1

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DS                      2
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D1             1.00000000 sec
TD0                     1

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PL1                 4.00 dB
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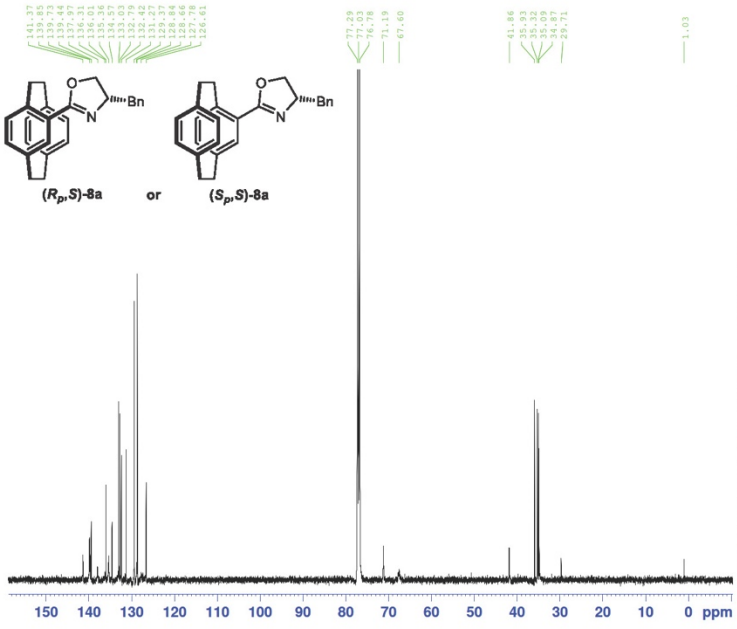


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PL2                 4.00 dB
PL12               22.51 dB
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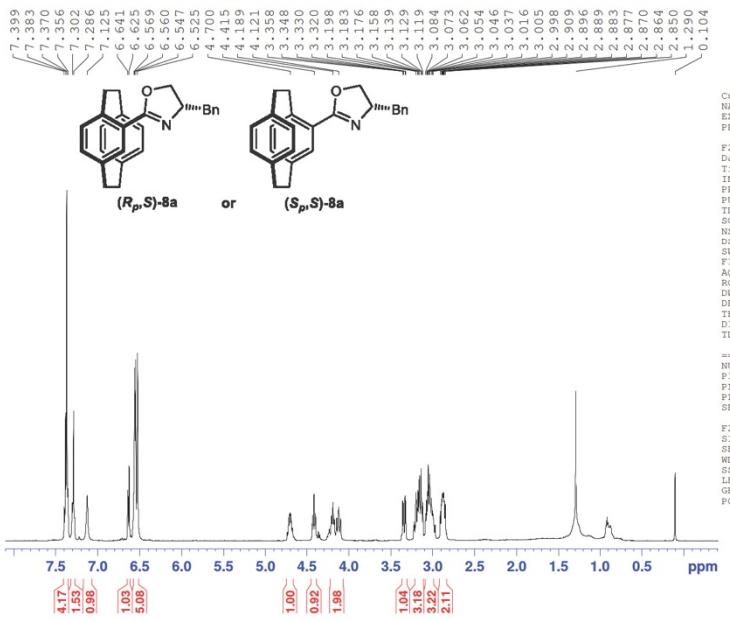


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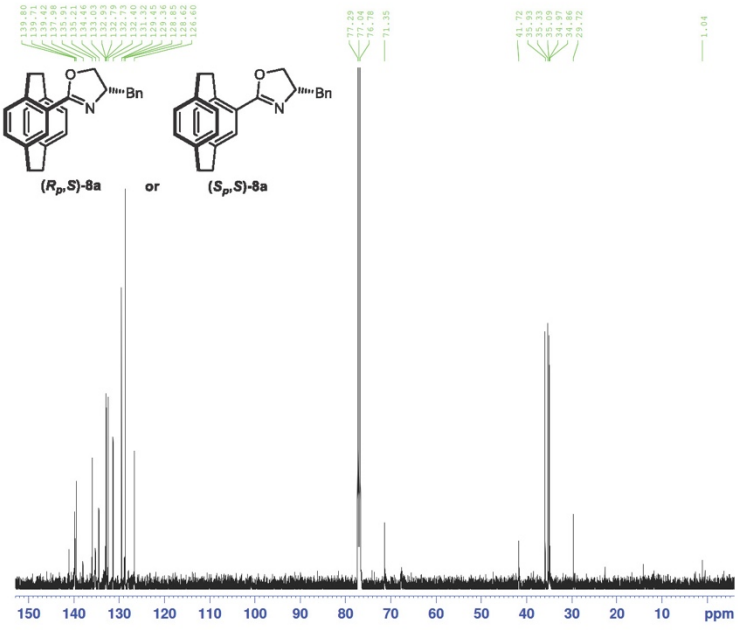


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PROCNO                      1

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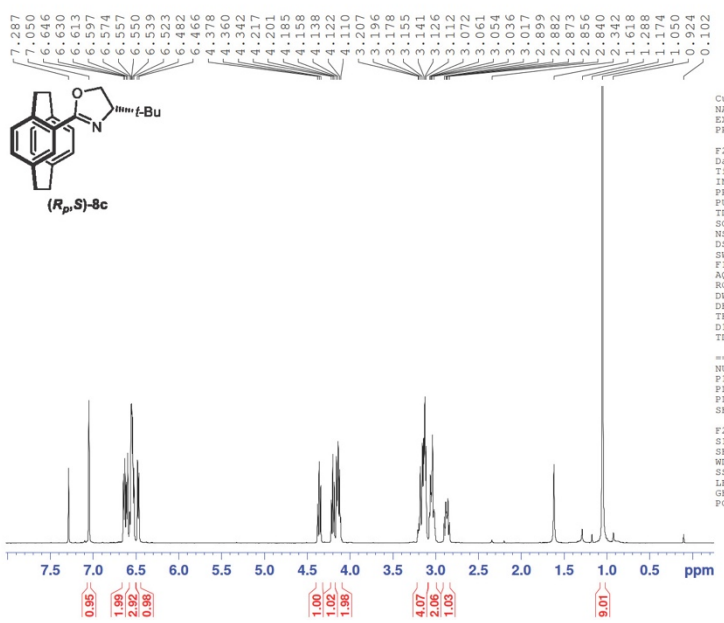
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PL1                -4.00 dB
PL1W       172.88230896 W
SFO1       125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                4.00 dB
PL12              22.51 dB
PL13              25.00 dB
PL2W        12.10000038 W
PL12W        0.17052394 W
PL13W        0.09611372 W
SFO2       500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF            125.7577890 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



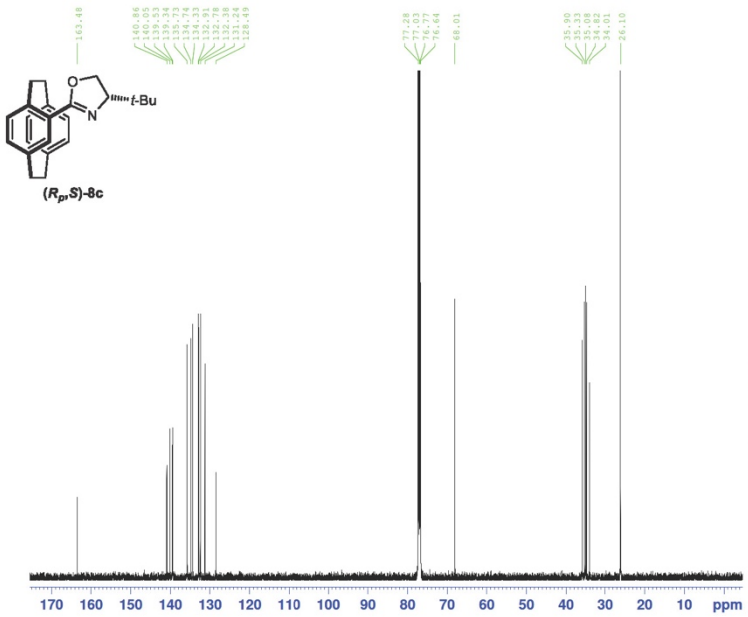
BRUKER

```
Current Data Parameters
NAME                  8ca
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200916
Time                17.29
INSTRUM             spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                     32
DS                      2
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1719425 sec
RG                    114
DW                48.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.50 usec
PL1                 4.00 dB
PL1W          12.10000038 W
SFO1          500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF            500.1300000 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



BRUKER

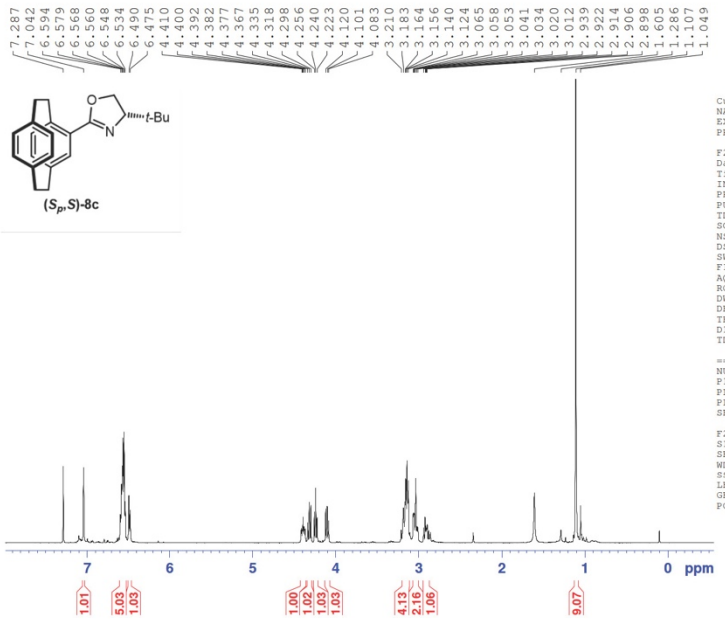
```
Current Data Parameters
NAME                  8ca
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200917
Time                 0.42
INSTRUM             spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT             CDCl3
NS                   8192
DS                      4
SWH            30030.029 Hz
FIDRES          0.458222 Hz
AQ              1.0911744 sec
RG                  32768
DW                16.650 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.00 usec
PL1                -4.00 dB
PL1W        172.88230896 W
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 4.00 dB
PL12               22.51 dB
PL13               25.00 dB
PL2W          12.10000038 W
PL12W         0.17052394 W
PL13W         0.09611372 W
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF            125.7577890 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

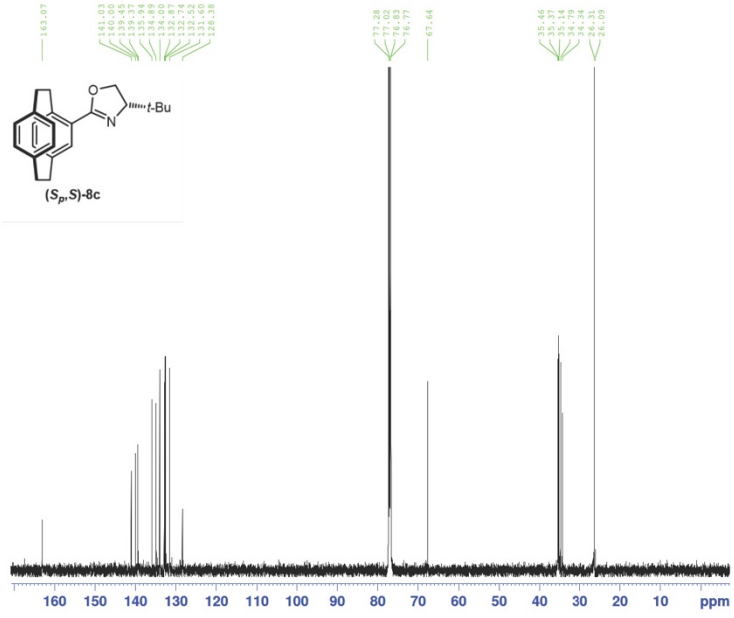



```
Current Data Parameters
NAME                  8cb
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20200917
Time               17.20
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1719425 sec
RG                   128
DW                48.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.50 usec
PL1                4.00 dB
PL1W          12.10000038 W
SFO1         500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF           500.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



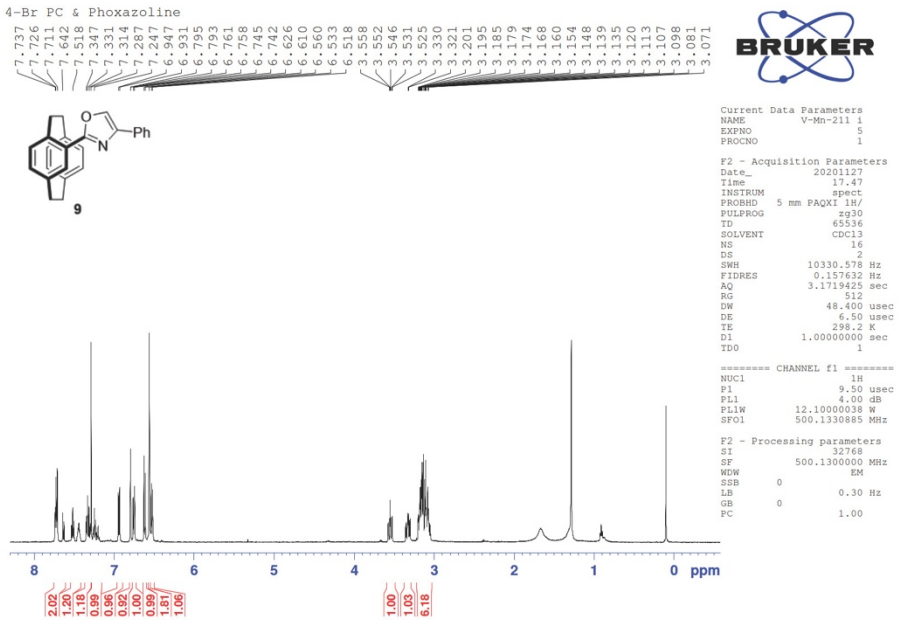

```
Current Data Parameters
NAME                  8cb
EXPNO                   2
PROCNO                  1

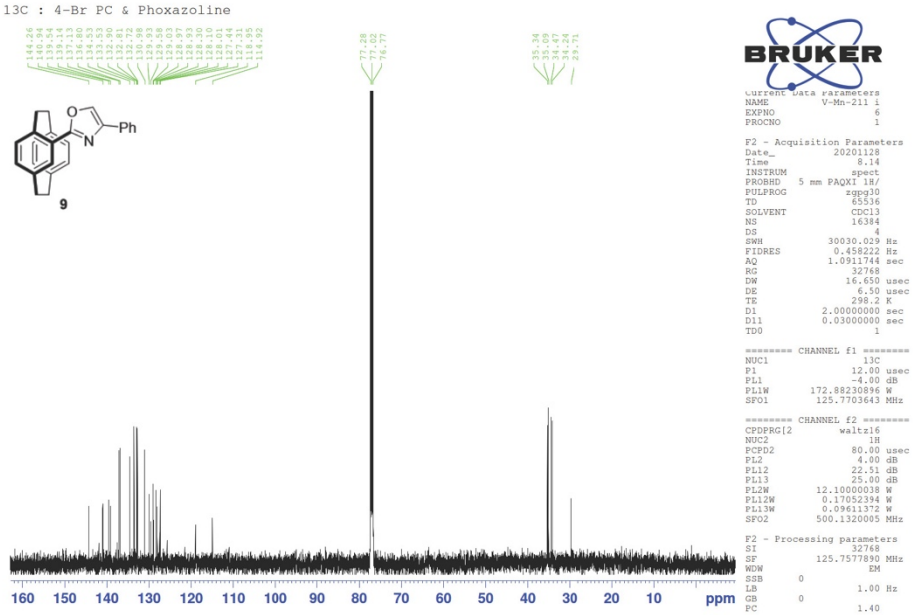
F2 - Acquisition Parameters
Date_           20200918
Time               0.33
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  8192
DS                     4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ             1.0911744 sec
RG                 32768
DW                16.650 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

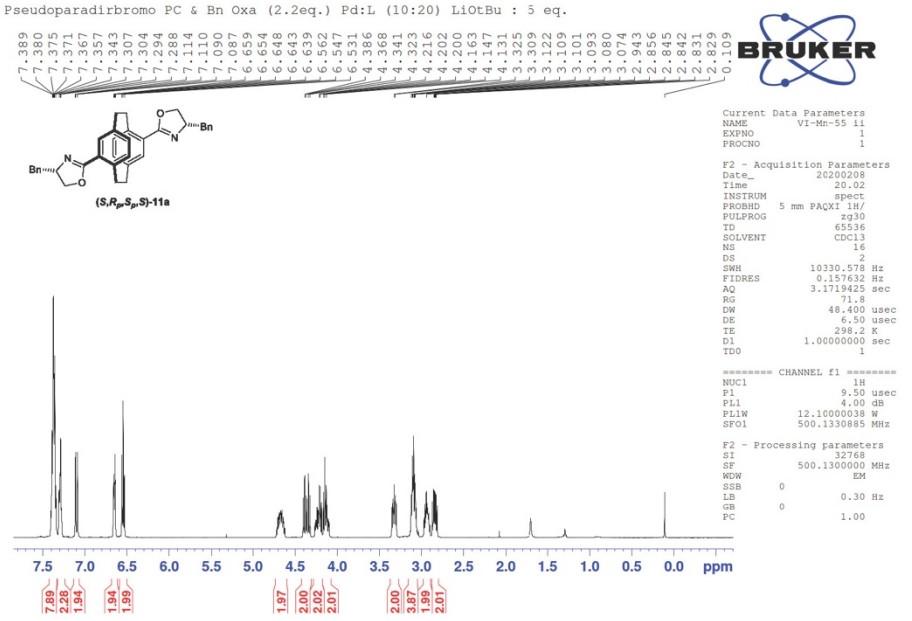
======== CHANNEL f1 ========
NUC1                 13C
P1                12.00 usec
PL1               -4.00 dB
PL1W         172.88230896 W
SFO1         125.7703643 MHz

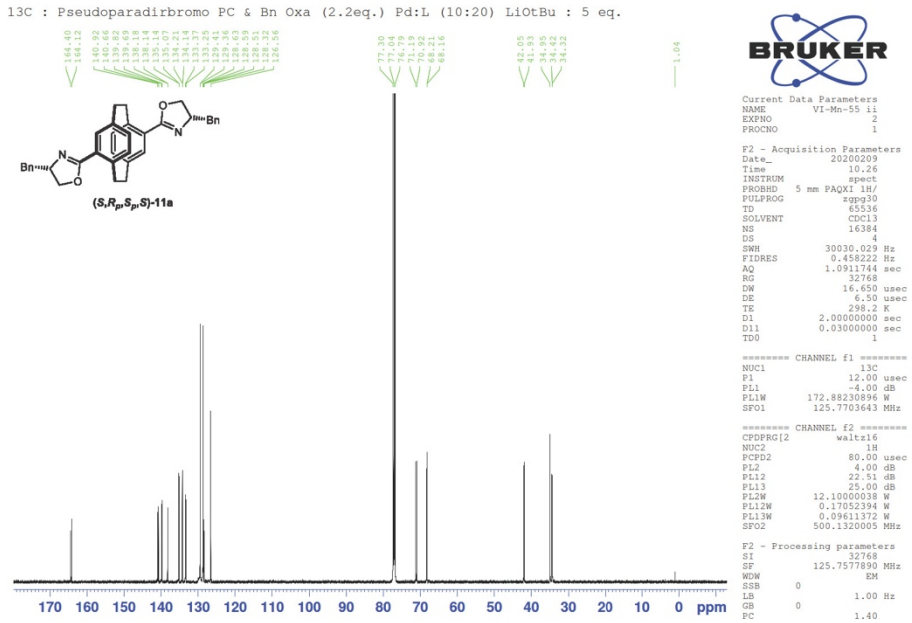
======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                4.00 dB
PL12              22.51 dB
PL13              25.00 dB
PL2W          12.10000038 W
PL12W          0.17052394 W
PL13W          0.09611372 W
SFO2         500.1320005 MHz

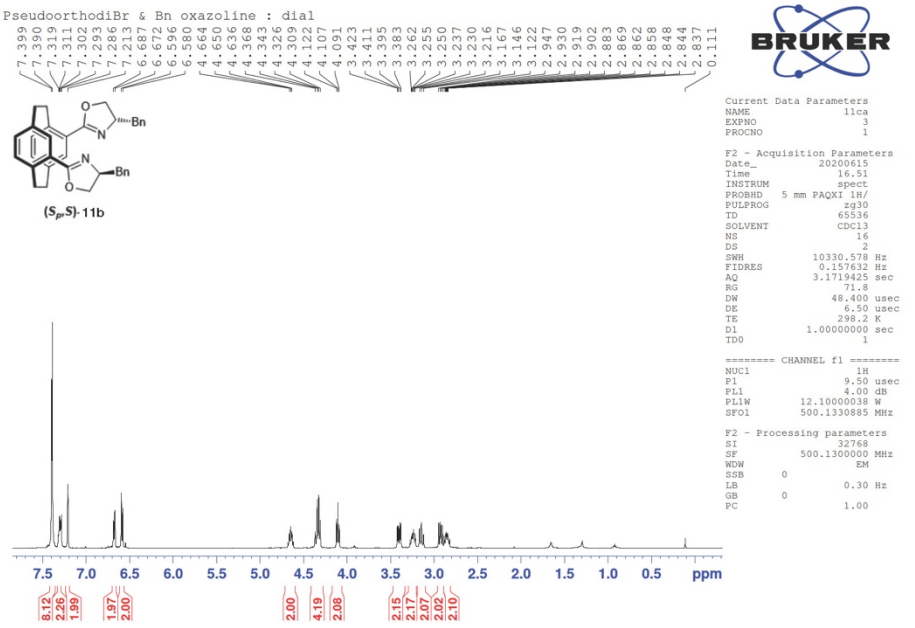
F2 - Processing parameters
SI                 32768
SF           125.7577890 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

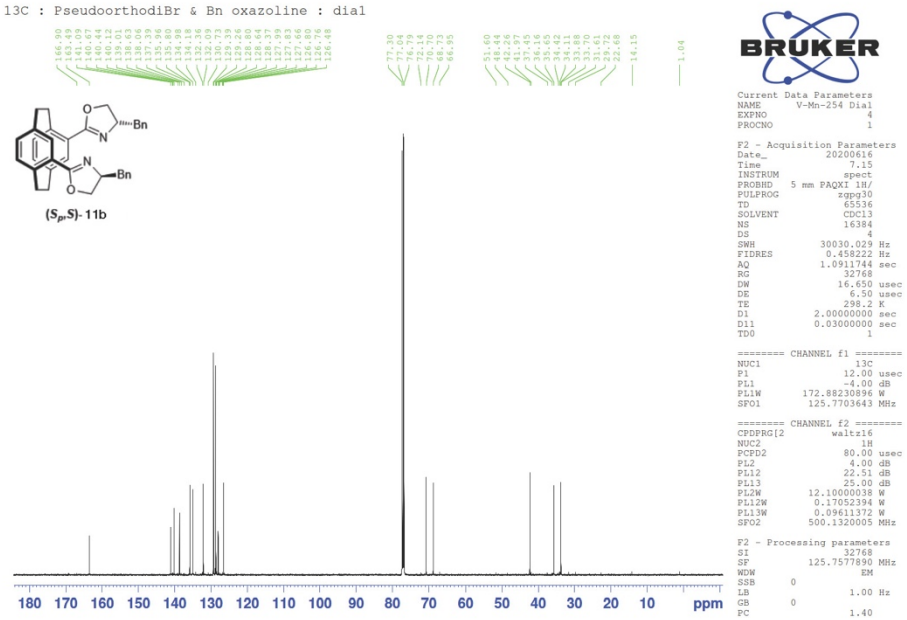

4-Br PC & Phoxazoline
7.737
7.726
7.711
7.642
7.518
7.347
7.331
7.314
7.287
7.247
6.947
6.931
6.795
6.793
6.761
6.758
6.745
6.742
6.626
6.610
6.560
6.533
6.518
3.558
3.552
3.546
3.531
3.525
3.330
3.321
3.201
3.195
3.185
3.179
3.174
3.168
3.160
3.154
3.148
3.139
3.135
3.120
3.113
3.107
3.098
3.081
3.071
Ph
9
BRUKER
Current Data Parameters
NAME V-Mn-211 i
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20201127
Time 17.47
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 512
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0 ppm
2.02
1.20
1.19
0.99
0.96
0.92
1.00
0.99
1.81
1.06
1.00
1.03
6.18

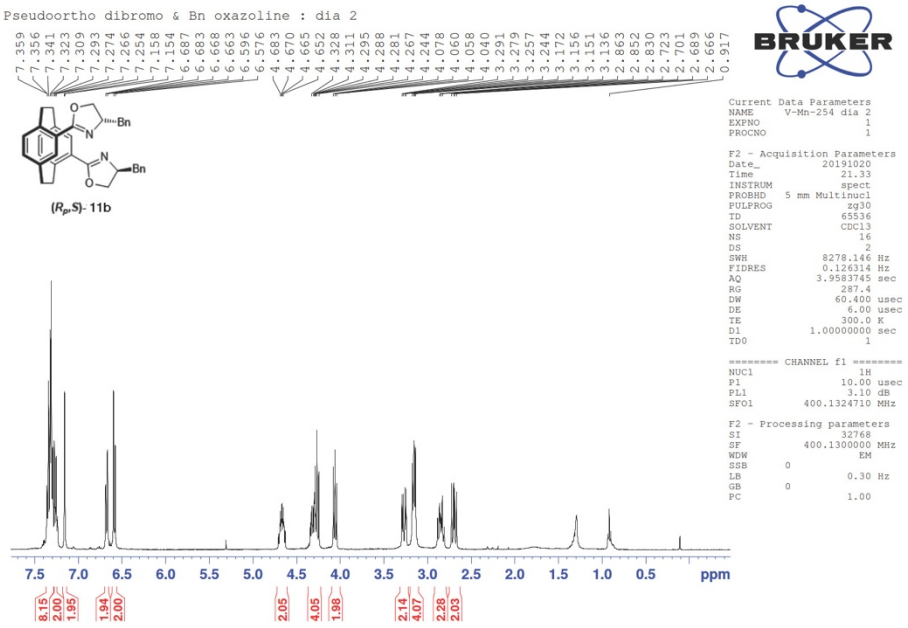

13C : 4-Br PC & Phoxazoline
144.26
140.94
139.14
137.13
136.80
134.57
133.53
132.90
132.72
130.98
129.93
129.58
129.03
128.97
128.30
128.10
128.01
127.44
127.31
118.95
114.92
77.28
77.02
76.77
35.34
35.09
34.47
34.24
29.71
Ph
9
BRUKER
Current Data Parameters
NAME V-Mn-211 i
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20201128
Time 8.14
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

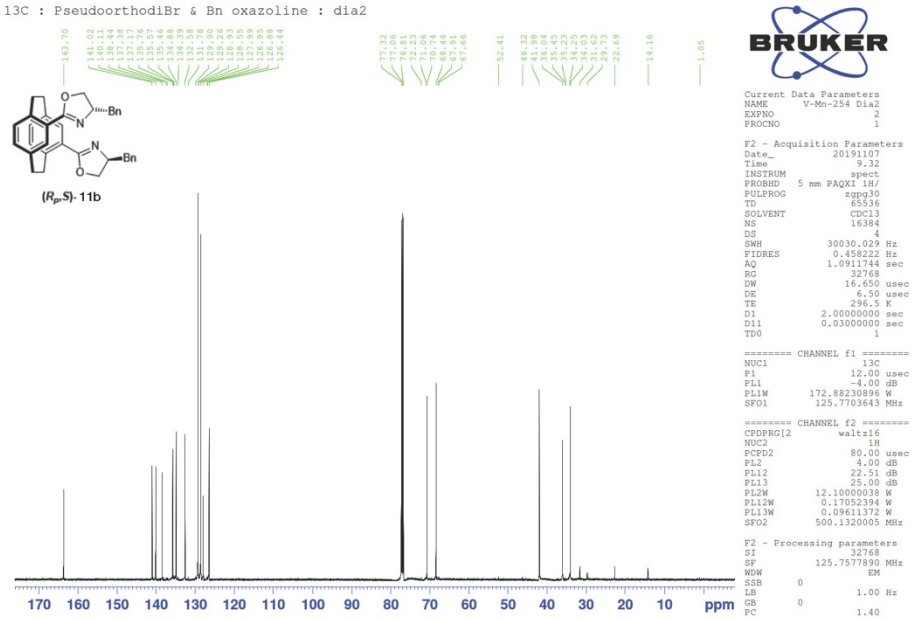

Pseudoparadirbromo PC & Bn Oxa (2.2eq.) Pd:L (10:20) LiOtBu : 5 eq.
BRUKER
(S,Rp,Sp,S)-11a
Current Data Parameters
NAME VI-Mn-55 ii
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200208
Time 20.02
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 71.8
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.13000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

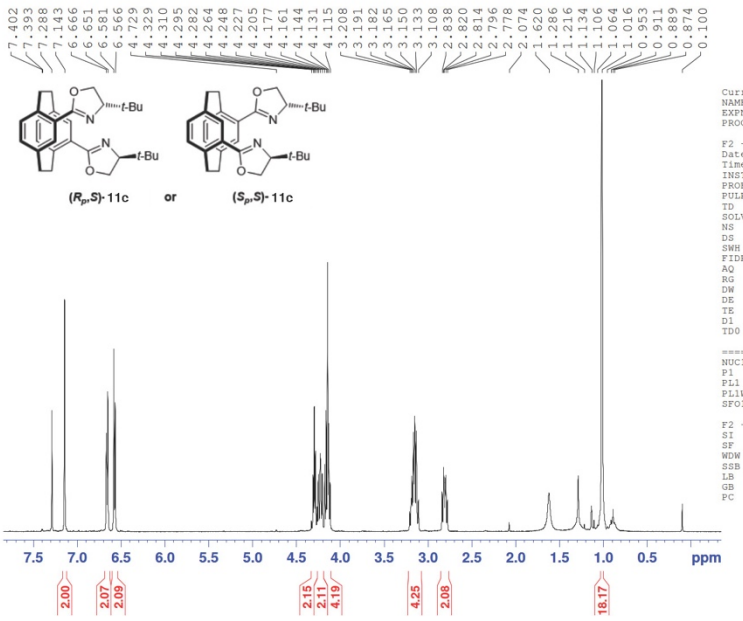

13C : Pseudoparadirbromo PC & Bn Oxa (2.2eq.) Pd:L (10:20) LiOtBu : 5 eq.
BRUKER
(S,Rp,Sp,S)-11a
Current Data Parameters
NAME VI-Mn-55 ii
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200209
Time 10.26
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm


PseudoorthodiBr & Bn oxazoline : dia1
BRUKER
Bn
Bn
(Sp,S)-11b
Current Data Parameters
NAME 11ca
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200615
Time 16.51
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 71.8
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
8.12 2.26 1.99 1.97 2.00 2.00 4.19 2.08 2.15 2.17 2.07 2.02 2.10


13C : PseudoorthodiBr & Bn oxazoline : dia1
BRUKER
Bn
Bn
(Sp,S)-11b
Current Data Parameters
NAME V-Mn-254 Dia1
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200616
Time 7.15
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Pseudoortho dibromo & Bn oxazoline : dia 2
BRUKER
Bn
Bn
(Rpr,S)- 11b
Current Data Parameters
NAME V-Mn-254 dia 2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191020
Time 21.33
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 287.4
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.10 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
8.15 2.00 1.95 1.94 2.00 2.05 4.05 1.98 2.14 4.07 2.28 2.03


13C : PseudoorthodiBr & Bn oxazoline : dia2
BRUKER
Bn
Bn
(Rpr,S)- 11b
Current Data Parameters
NAME V-Mn-254 Dia2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191107
Time 9.32
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



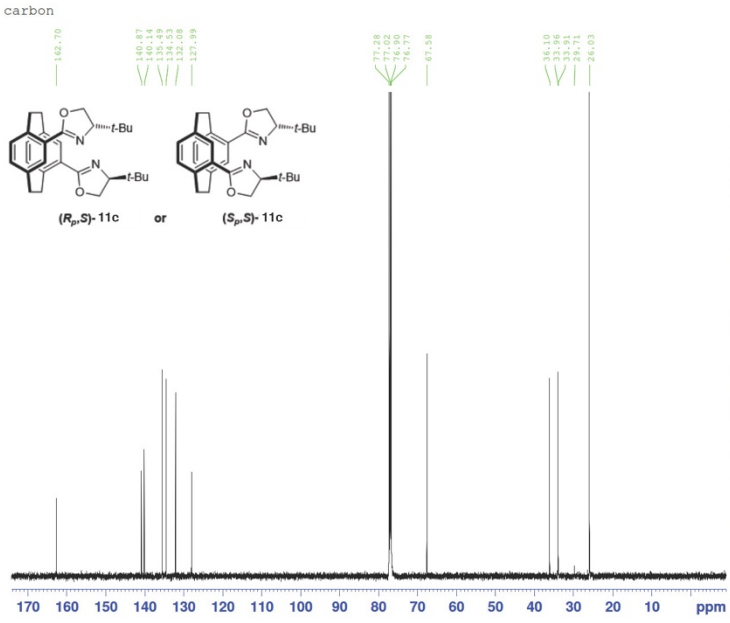
BRUKER

```
Current Data Parameters
NAME                  11cc
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20200919
Time                 15.49
INSTRUM              spect
PROBHD   5 mm PAQXI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                       8
DS                       2
SWH              10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1719425 sec
RG                     128
DW                48.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.50 usec
PL1                 4.00 dB
PL1W          12.10000038 W
SFO1          500.1330885 MHz

F2 - Processing parameters
SI                   32768
SF            500.1300000 MHz
WDW                     EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



BRUKER

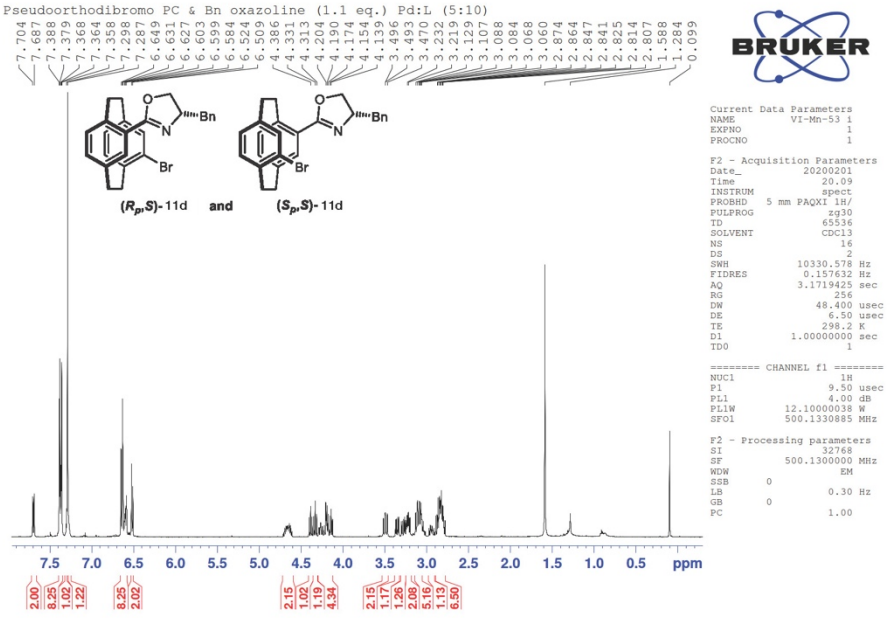
```
Current Data Parameters
NAME                  11cc
EXPNO                    2
PROCNO                   1

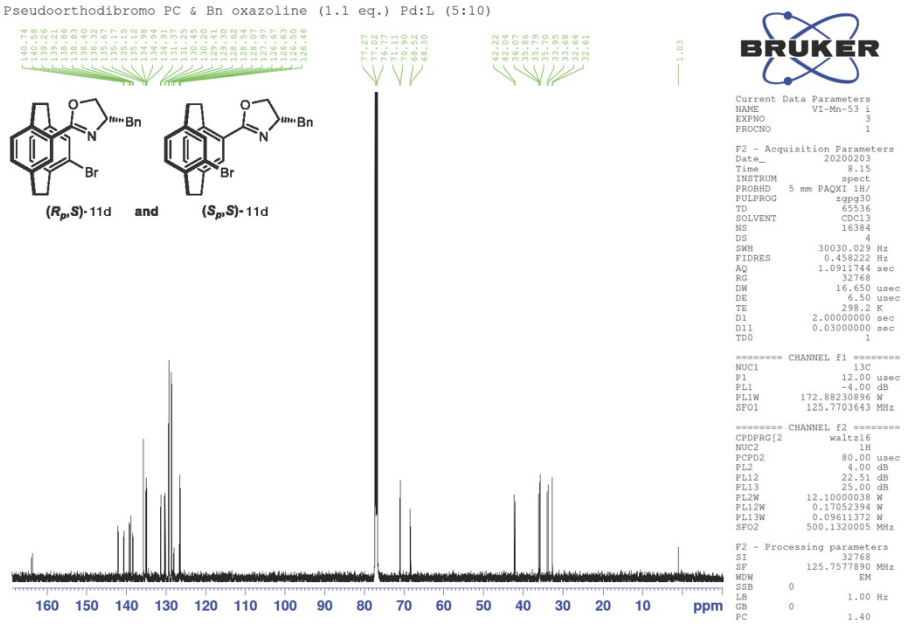
F2 - Acquisition Parameters
Date_              20200919
Time                 23.01
INSTRUM              spect
PROBHD   5 mm PAQXI 1H/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    8192
DS                       4
SWH              30030.029 Hz
FIDRES           0.458222 Hz
AQ               1.0911744 sec
RG                   32768
DW                16.650 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

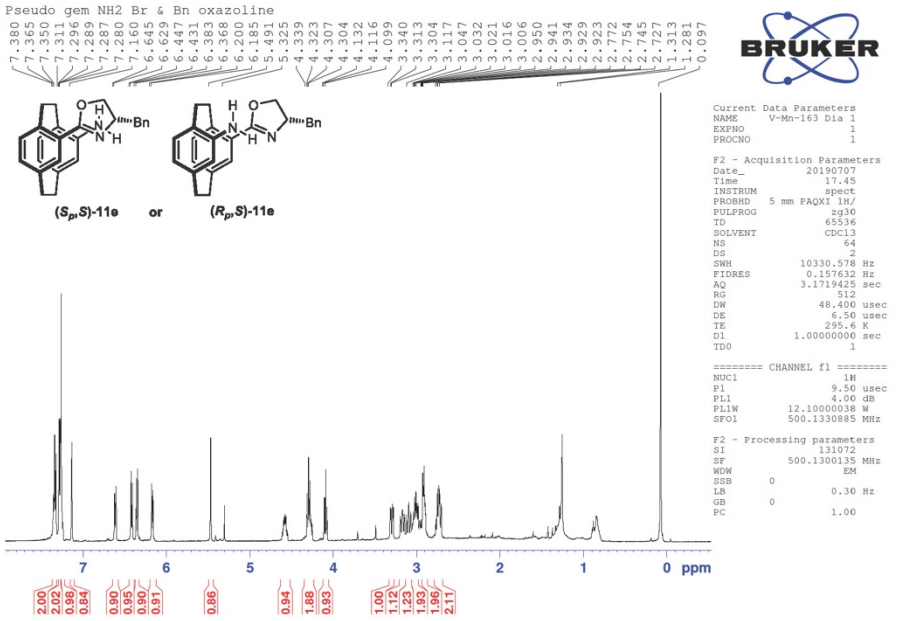
======== CHANNEL f1 ========
NUC1                  13C
P1                 12.00 usec
PL1                -4.00 dB
PL1W         172.88230896 W
SFO1         125.7703643 MHz

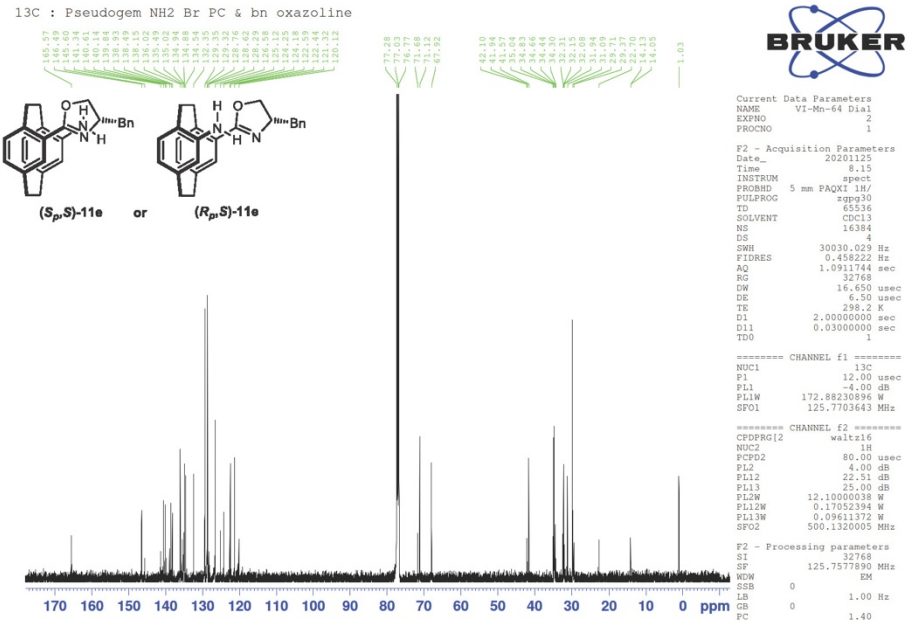
======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 4.00 dB
PL12               22.51 dB
PL13               25.00 dB
PL2W          12.10000038 W
PL12W          0.17052394 W
PL13W          0.09611372 W
SFO2          500.1320005 MHz

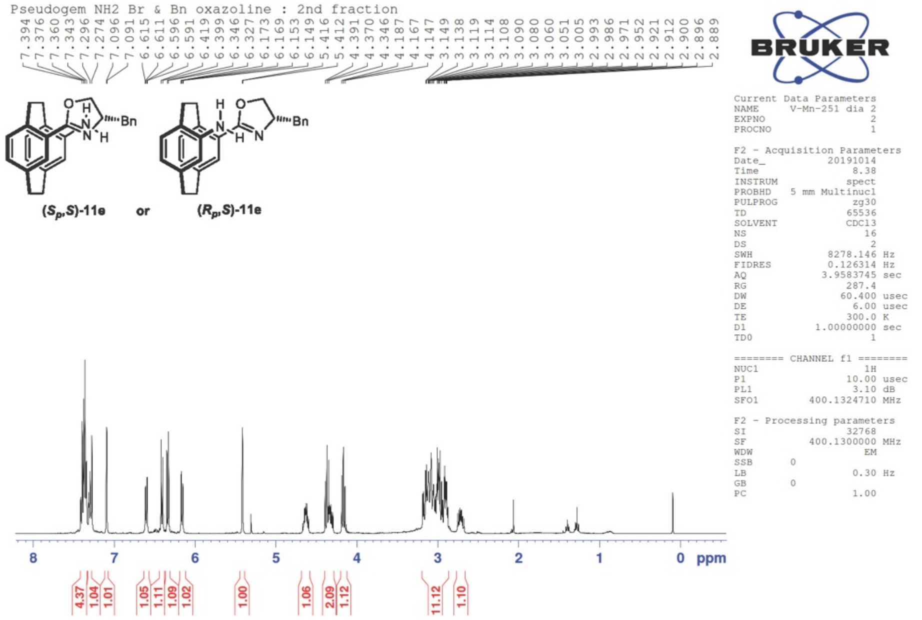
F2 - Processing parameters
SI                   32768
SF            125.7577890 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

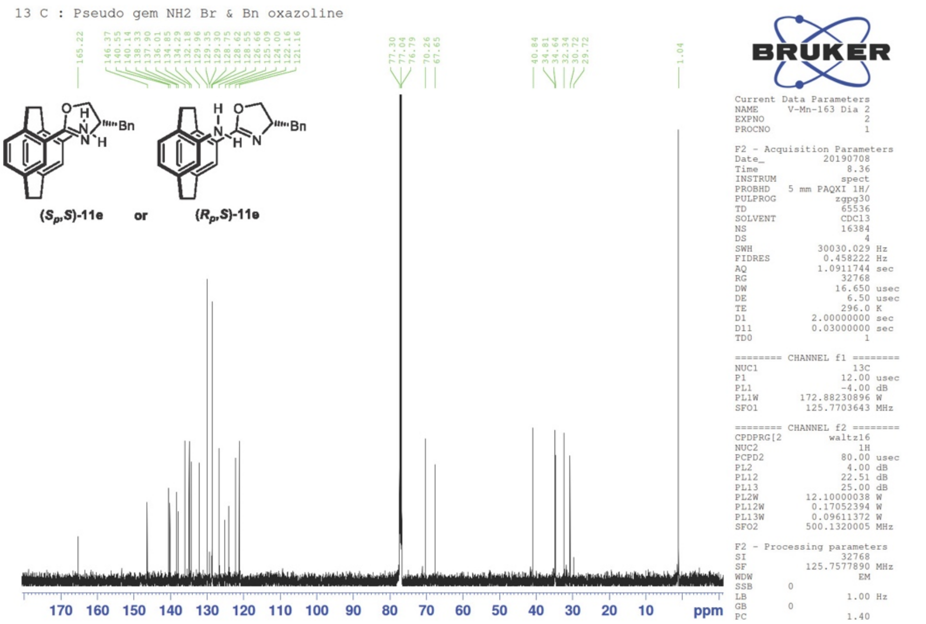

Pseudoorthodibromo PC & Bn oxazoline (1.1 eq.) Pd:L (5:10)
(Rp,S)-11d and (Sp,S)-11d
Current Data Parameters
NAME VI-Mn-53 i
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200201
Time 20.09
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 256
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

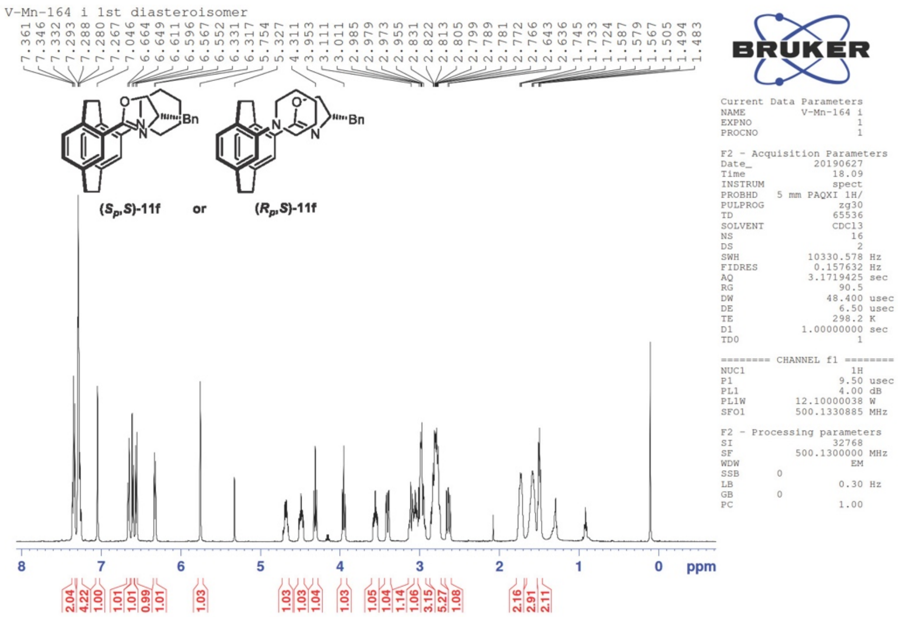

Pseudoorthodibromo PC & Bn oxazoline (1.1 eq.) Pd:L (5:10)
(Rp,S)-11d and (Sp,S)-11d
Current Data Parameters
NAME VI-Mn-53 i
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200203
Time 8.15
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm

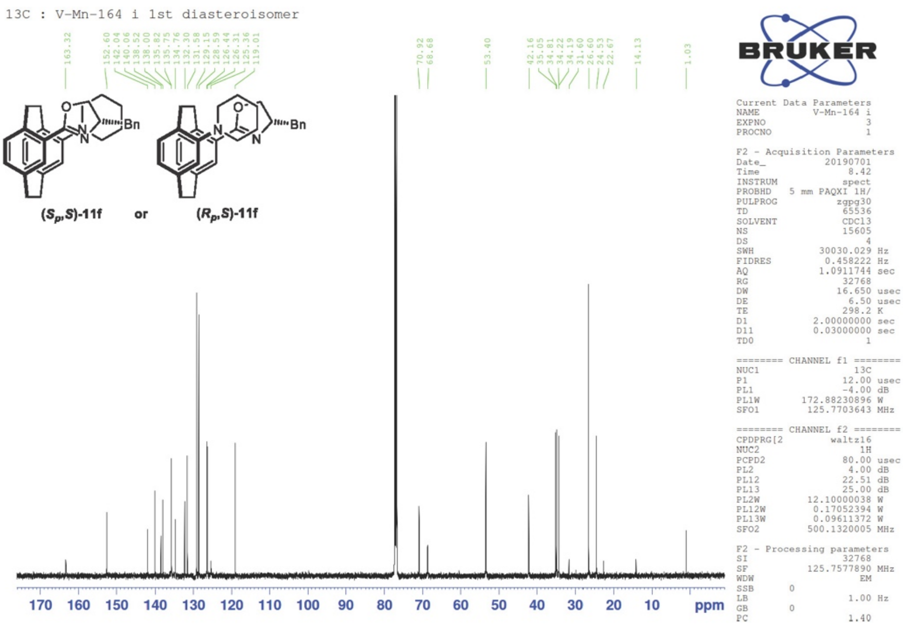

Pseudo gem NH2 Br & Bn oxazoline
(Sp,S)-11e or (Rp,S)-11e
Current Data Parameters
NAME V-Mn-163 Dia 1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190707
Time 17.45
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 512
DW 48.400 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 131072
SF 500.1300135 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7 6 5 4 3 2 1 0 ppm

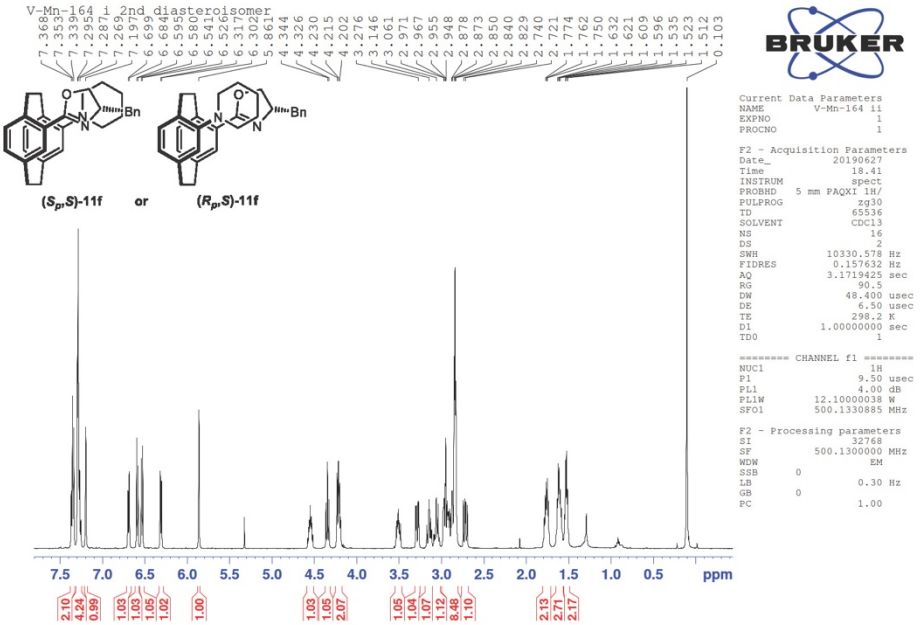

13C : Pseudogem NH2 Br PC & bn oxazoline
(Sp,S)-11e or (Rp,S)-11e
Current Data Parameters
NAME VI-Mn-64 Dia1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20201125
Time 8.15
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

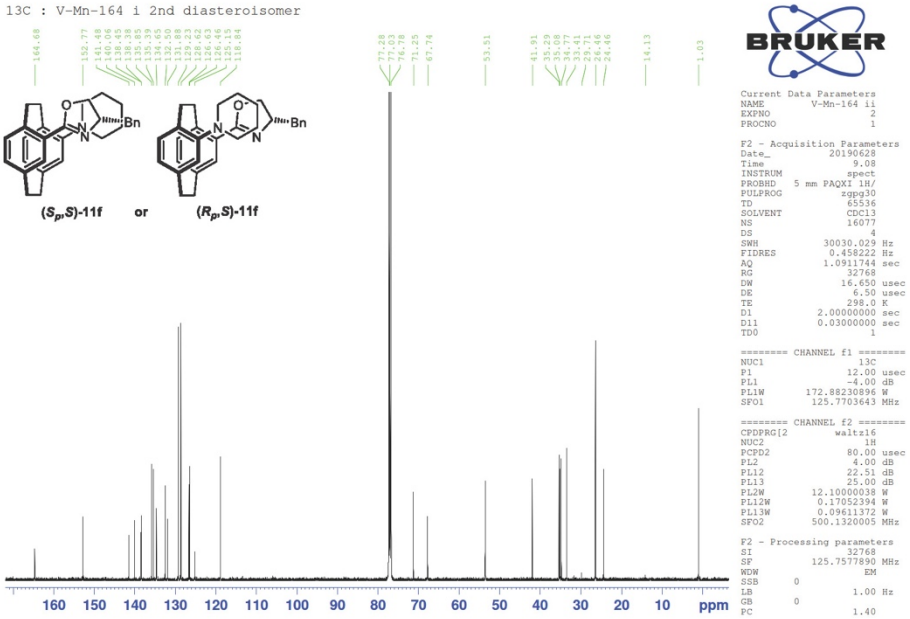

Pseudogem NH2 Br & Bn oxazoline : 2nd fraction
7.394 7.376 7.360 7.343 7.296 7.274 7.096 7.091 6.615 6.611 6.596 6.591 6.419 6.399 6.346 6.327 6.173 6.169 6.153 6.149 5.416 5.412 4.391 4.370 4.346 4.187 4.167 4.147 3.149 3.138 3.119 3.114 3.108 3.090 3.080 3.060 3.051 3.005 2.993 2.986 2.971 2.952 2.921 2.912 2.900 2.896 2.889
(Sp,S)-11e
or
(Rp,S)-11e
Bn
BRUKER
Current Data Parameters
NAME V-Mn-251 dia 2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191014
Time 8.38
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 287.4
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.10 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8 7 6 5 4 3 2 1 0 ppm
4.37 1.04 1.01 1.05 1.11 1.09 1.02 1.00 1.06 2.09 1.12 11.12 1.10

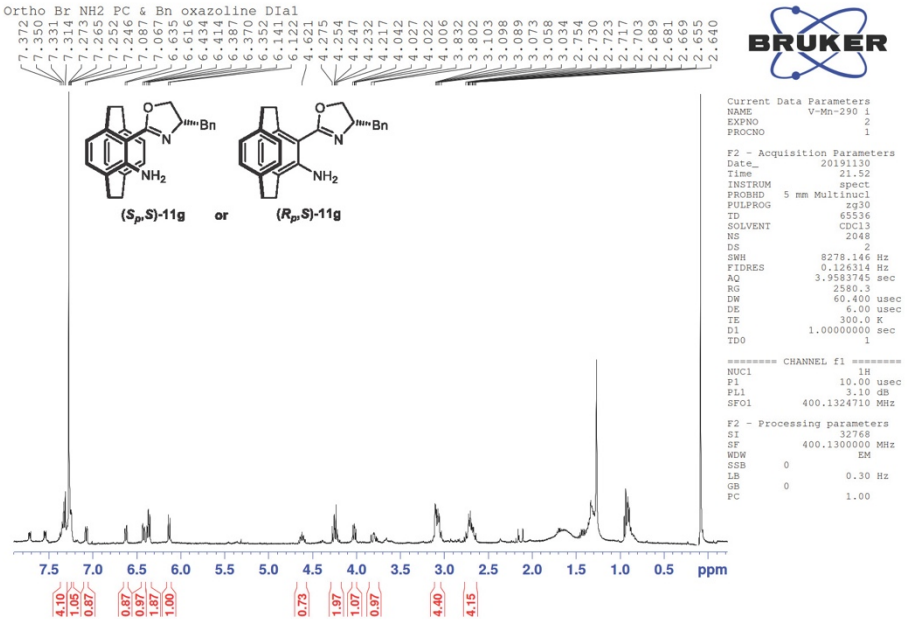

13 C : Pseudo gem NH2 Br & Bn oxazoline
165.22 146.37 140.55 140.14 138.33 137.90 136.01 134.85 134.29 132.18 129.96 129.35 129.30 128.75 128.62 128.55 126.66 125.09 124.00 122.16 121.16 77.30 77.04 76.79 70.26 67.65 40.84 34.81 34.64 32.34 30.72 29.72 1.04
(Sp,S)-11e
or
(Rp,S)-11e
Bn
BRUKER
Current Data Parameters
NAME V-Mn-163 Dia 2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190708
Time 8.36
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

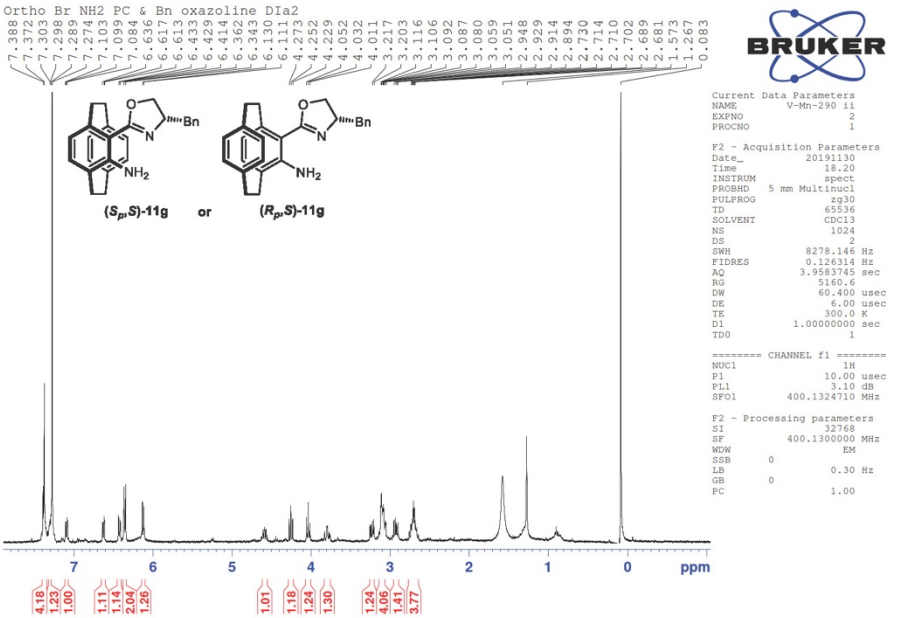

V-Mn-164 i 1st diasteroisomer
(Sp,S)-11f
or
(Rp,S)-11f
Bn
BRUKER
ppm

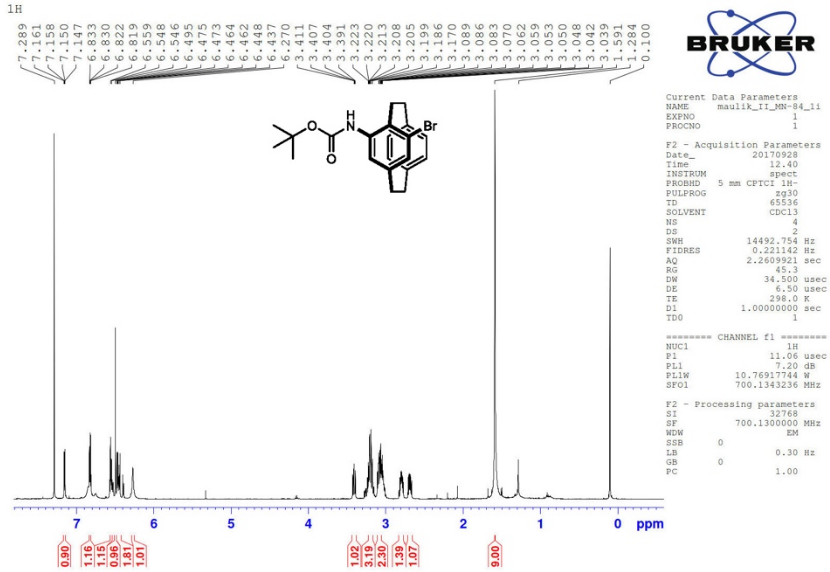

13C : V-Mn-164 i 1st diasteroisomer
(Sp,S)-11f
or
(Rp,S)-11f
Bn
BRUKER
ppm

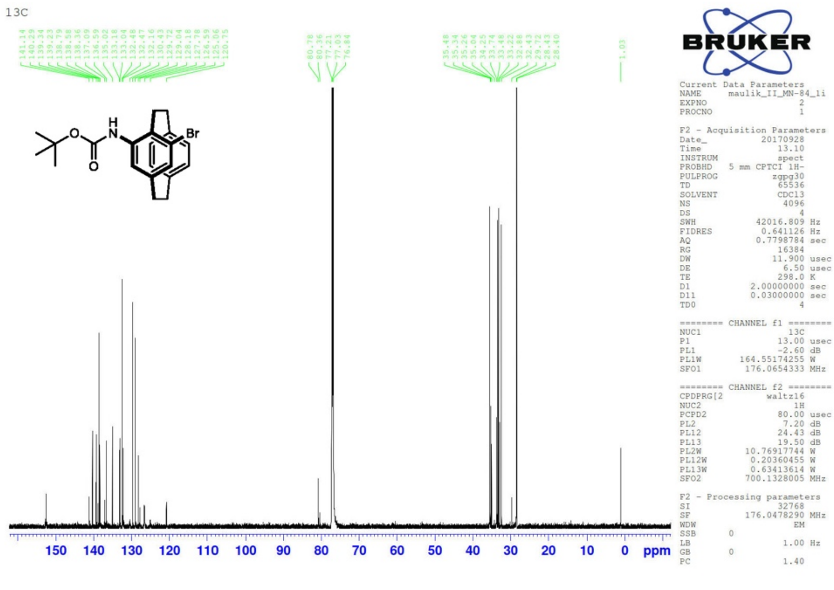

V-Mn-164 i 2nd diasteroisomer
(Sp,S)-11f or (Rp,S)-11f
Current Data Parameters
NAME V-Mn-164 ii
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190627
Time 18.41
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 90.5
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

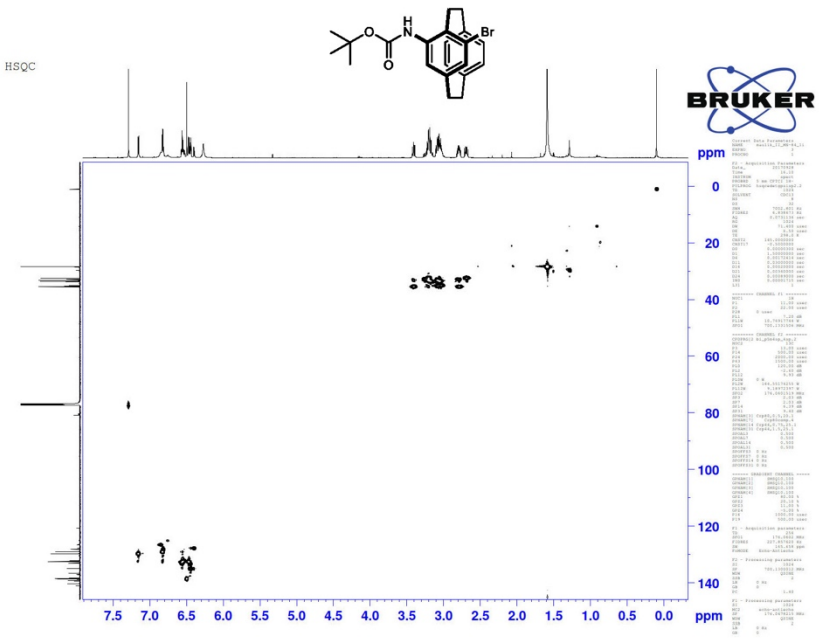

13C : V-Mn-164 i 2nd diasteroisomer
(Sp,S)-11f or (Rp,S)-11f
Current Data Parameters
NAME V-Mn-164 ii
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20190628
Time 9.08
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 16077
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

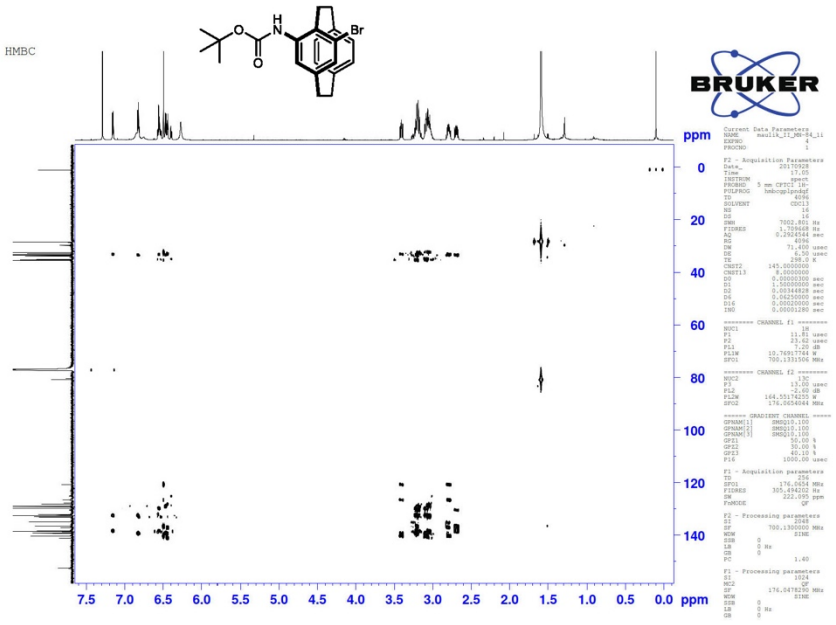

Ortho Br NH2 PC & Bn oxazoline DIa1
BRUKER
(Sp,S)-11g or (Rp,S)-11g
Current Data Parameters
NAME V-Mn-290 i
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191130
Time 21.52
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 2580.3
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.10 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

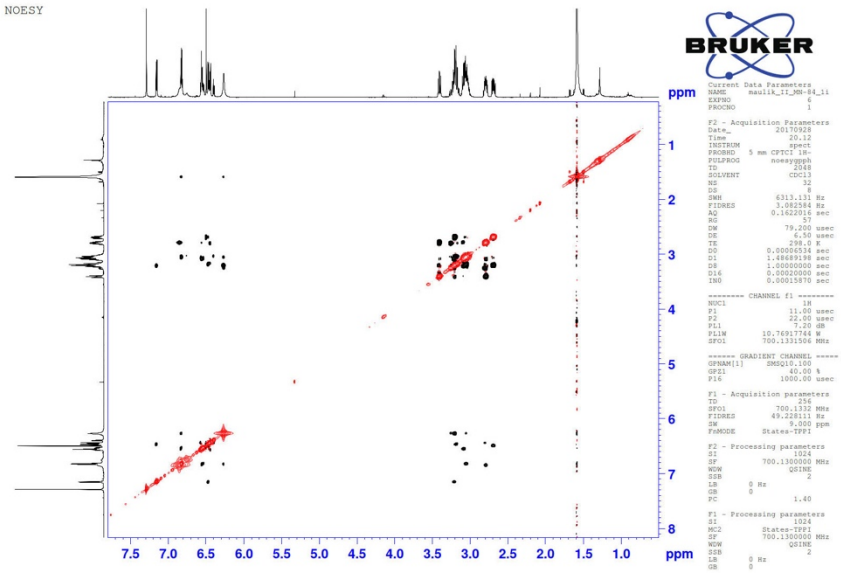

Ortho Br NH2 PC & Bn oxazoline DIa2
BRUKER
(Sp,S)-11g or (Rp,S)-11g
Current Data Parameters
NAME V-Mn-290 ii
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191130
Time 18.20
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 5160.6
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.10 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

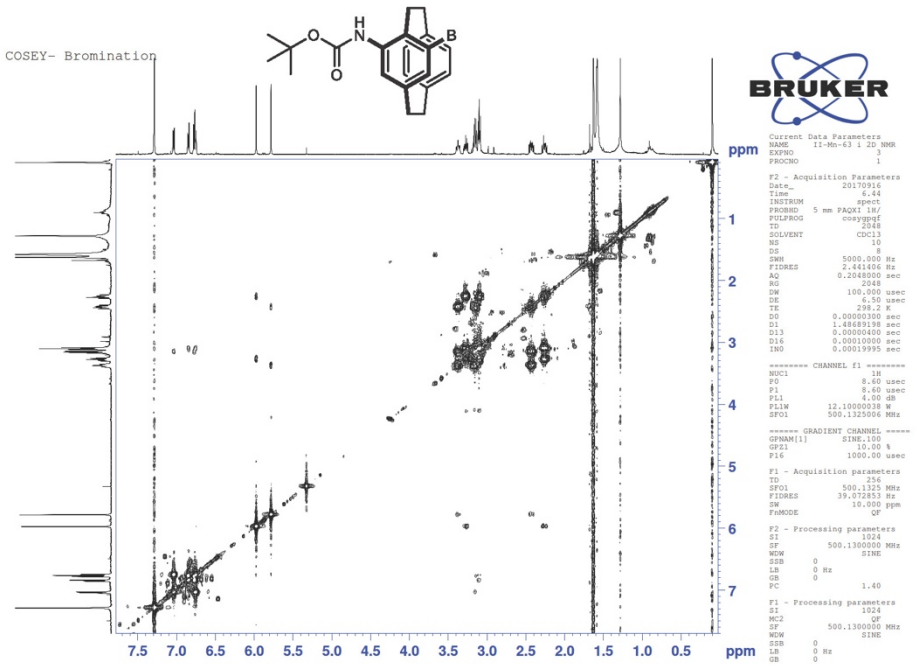


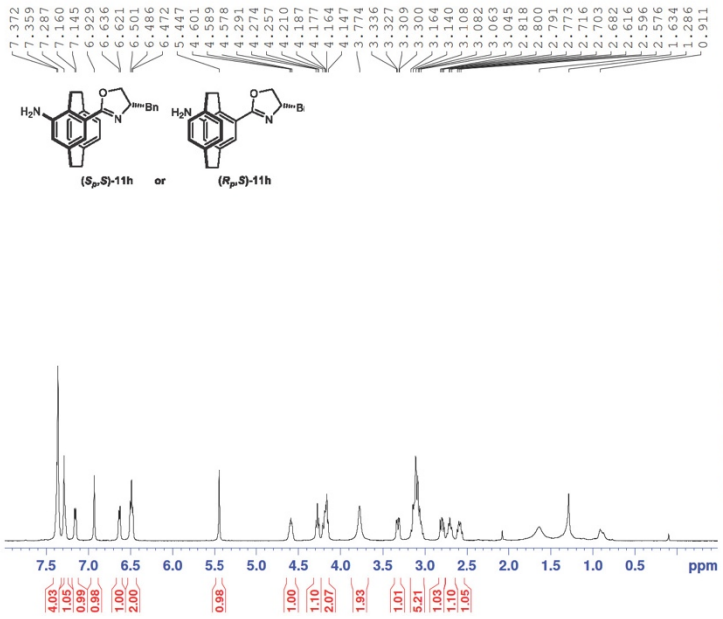



HSQC
BRUKER
ppm
0
20
40
60
80
100
120
140
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm


HMBC
BRUKER
ppm
0
20
40
60
80
100
120
140
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm


NOESY
BRUKER
ppm
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
1 2 3 4 5 6 7 8


COSEY- Bromination
BRUKER
ppm
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
1 2 3 4 5 6 7

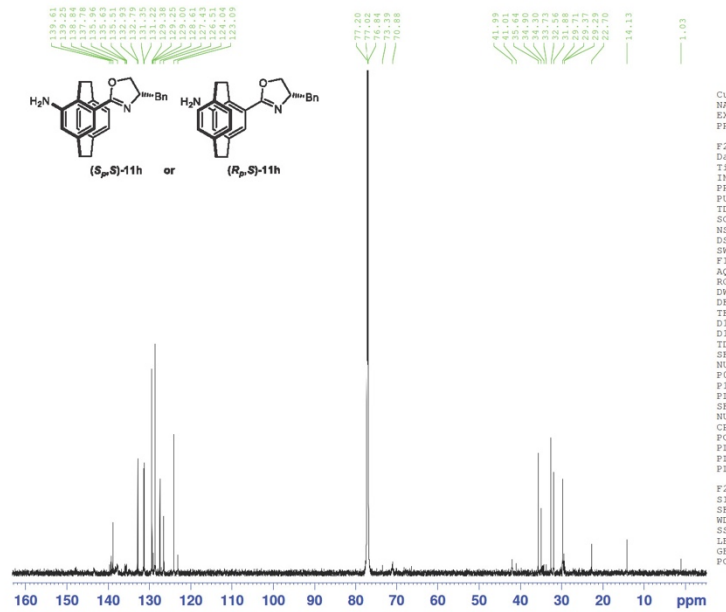



```
Current Data Parameters
NAME                  11ha
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20201207
Time                 10.10
INSTRUM              spect
PROBHD   5 mm PAQXI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      2
SWH              10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1719425 sec
RG                   161.3
DW                  48.400 usec
DE                   6.50 usec
TE                  300.0 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.50 usec
PL1                  4.00 dB
PLW             12.10000038 W
SFO1           500.1330885 MHz

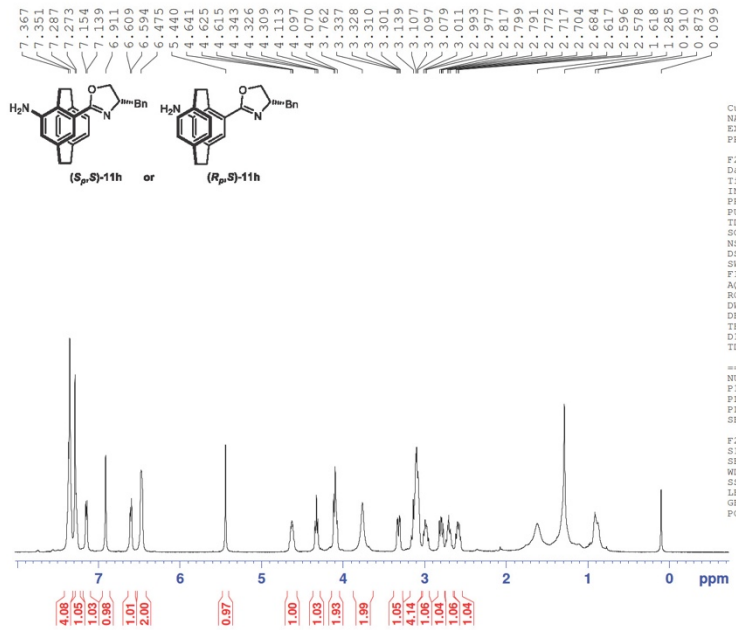
F2 - Processing parameters
SI                      32768
SF             500.1300000 MHz
WDW                        EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```




```
Current Data Parameters
NAME                     4
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_              20210817
Time                  8.37 h
INSTRUM              Avance
PROBHD   Z168386_0003 (
PULPROG              zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  18432
DS                      4
SWH              41666.668 Hz
FIDRES           1.271566 Hz
AQ               0.7864320 sec
RG                     101
DW                  12.000 usec
DE                  18.00 usec
TE                  298.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                     18
SFO1           176.0654333 MHz
NUC1                   13C
P0                   4.00 usec
P1                  12.00 usec
PLW1            132.53999329 W
SFO2            700.1328005 MHz
NUC2                    1H
CPDPRG[2             waltz65
PCPD2                80.00 usec
PLW2             7.66239977 W
PLW12            0.07662400 W
PLW13            0.03000000 W

F2 - Processing parameters
SI                      32768
SF             176.0478285 MHz
WDW                        EM
SSB                     0
LB                   2.00 Hz
GB                      0
PC                   1.40
```



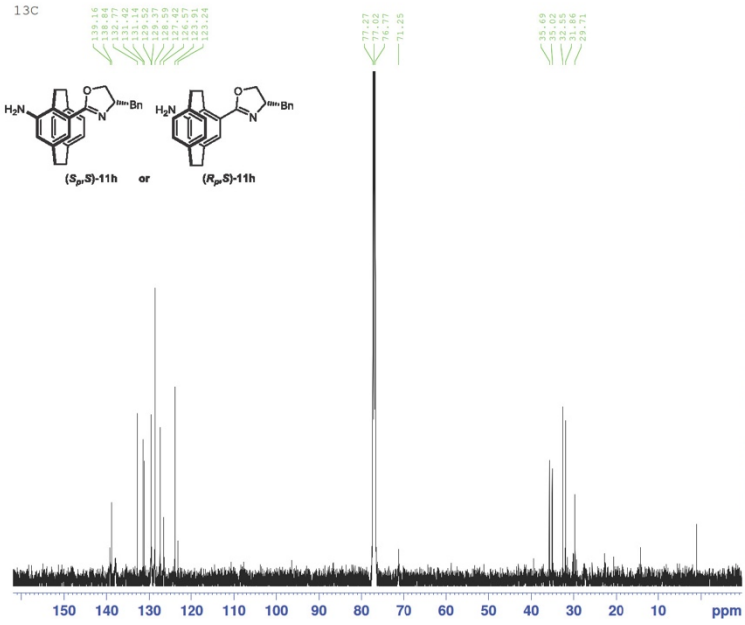
BRUKER

```
Current Data Parameters
NAME                  11hb
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20201207
Time                 10.21
INSTRUM              spect
PROBHD   5 mm PAQXI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     64
DS                      2
SWH             10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1719425 sec
RG                    181
DW                48.400 usec
DE                 6.50 usec
TE                298.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.50 usec
PL1                 4.00 dB
PL1W          12.10000038 W
SFO1          500.1330885 MHz

F2 - Processing parameters
SI                  32768
SF            500.1300000 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



BRUKER

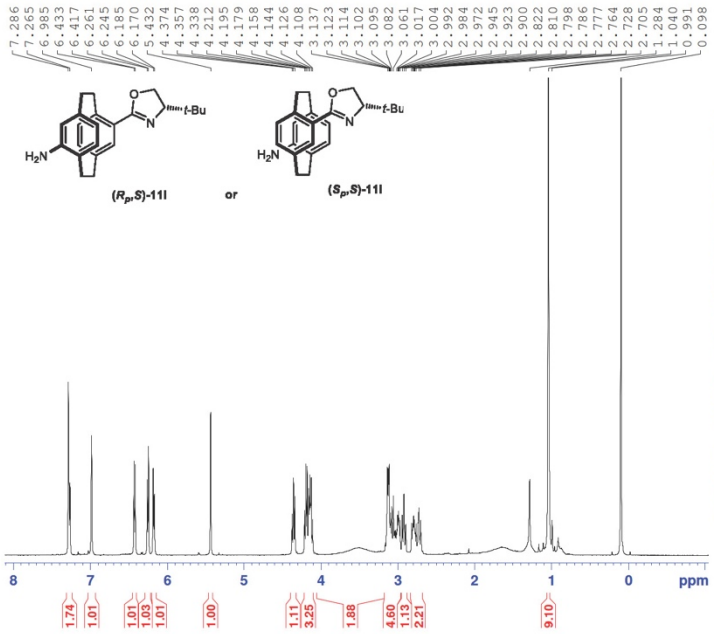
```
Current Data Parameters
NAME                  11hb
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_              20210813
Time                  7.57
INSTRUM              spect
PROBHD   5 mm PAQXI 1H/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  16384
DS                      4
SWH             30030.029 Hz
FIDRES           0.458222 Hz
AQ               1.0911744 sec
RG                  32768
DW                16.650 usec
DE                 6.50 usec
TE                298.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.00 usec
PL1                -4.00 dB
PL1W         172.88230896 W
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 4.00 dB
PL12               22.51 dB
PL13               25.00 dB
PL2W          12.10000038 W
PL12W          0.17052394 W
PL13W          0.09611372 W
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                  32768
SF            125.7577890 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

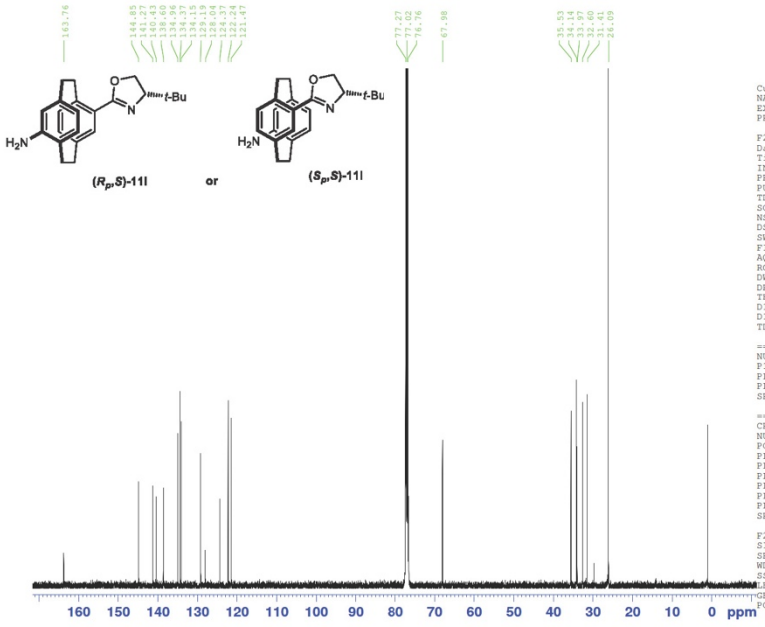


```
Current Data Parameters
NAME                  111a
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20210922
Time                 17.12
INSTRUM              spect
PROBHD    5 mm PAQXI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     64
DS                      2
SWH             10330.578 Hz
FIDRES              0.157632 Hz
AQ                3.1719425 sec
RG                     128
DW                  48.400 usec
DE                   6.50 usec
TE                  298.2 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.50 usec
PL1                  4.00 dB
PL1W            12.10000038 W
SFO1           500.1330885 MHz

F2 - Processing parameters
SI                   32768
SF              500.1300000 MHz
WDW                     EM
SSB          0
LB                   0.30 Hz
GB           0
PC                   1.00
```



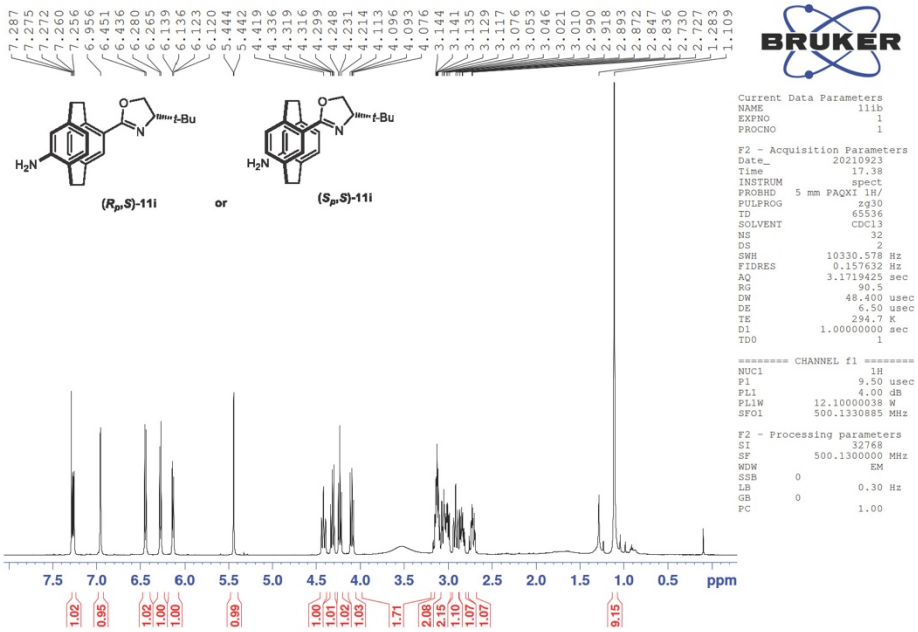
```
Current Data Parameters
NAME                  111a
EXPNO                    2
PROCNO                   1

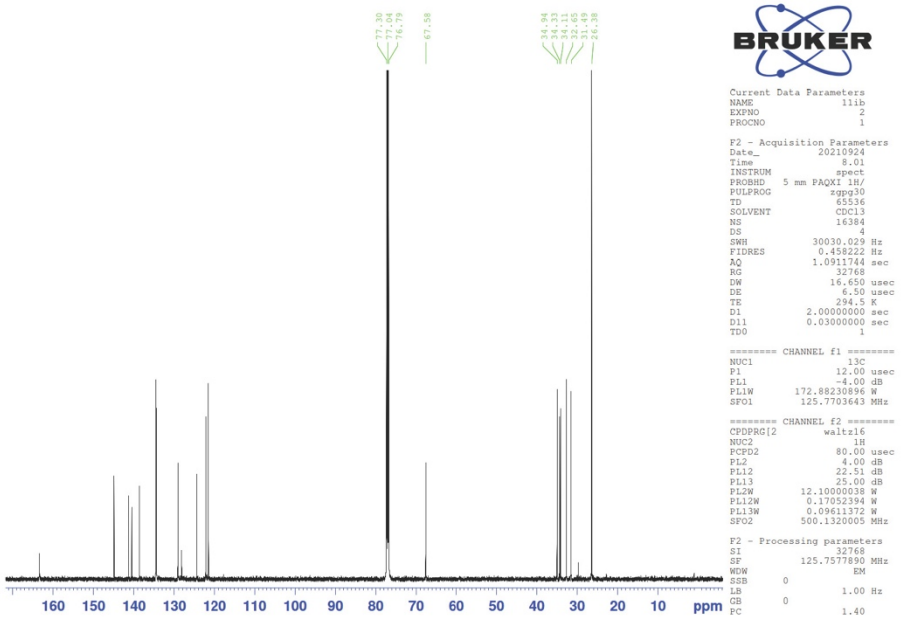
F2 - Acquisition Parameters
Date_              20210923
Time                  7.37
INSTRUM              spect
PROBHD    5 mm PAQXI 1H/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  16384
DS                      4
SWH             30030.029 Hz
FIDRES              0.458222 Hz
AQ                1.0911744 sec
RG                   32768
DW                  16.650 usec
DE                   6.50 usec
TE                  298.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

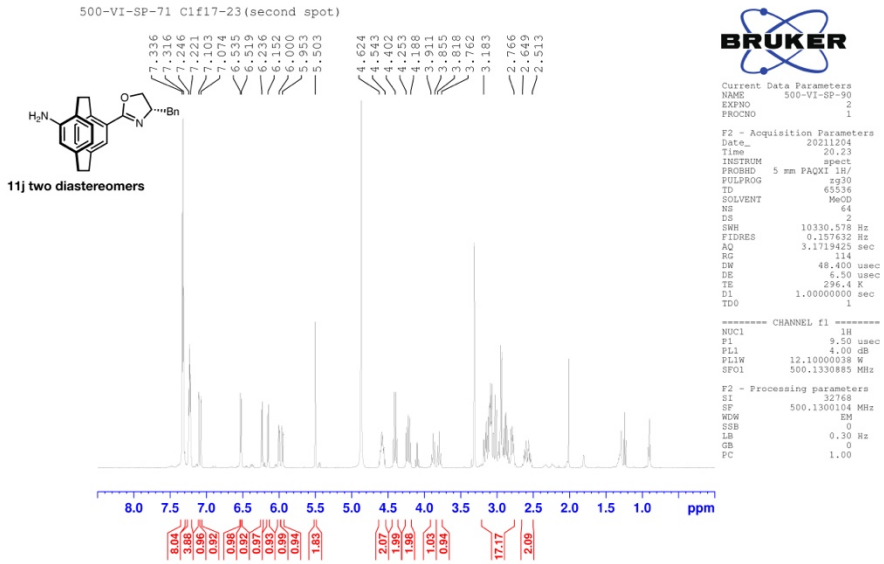
======== CHANNEL f1 ========
NUC1                   13C
P1                  12.00 usec
PL1                  -4.00 dB
PL1W           172.88230896 W
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                  4.00 dB
PL12                22.51 dB
PL13                25.00 dB
PL2W            12.10000038 W
PL12W           0.17052394 W
PL13W           0.09611372 W
SFO2           500.1320005 MHz

F2 - Processing parameters
SI                   32768
SF              125.7577890 MHz
WDW                     EM
SSB          0
LB                   1.00 Hz
GB           0
PC                   1.40
```


7.287
7.275
7.272
7.260
7.256
6.456
6.451
6.436
6.280
6.265
6.139
6.136
6.123
6.120
5.444
5.442
4.419
4.336
4.319
4.316
4.299
4.248
4.231
4.214
4.113
4.096
4.093
4.076
3.144
3.141
3.135
3.129
3.117
3.076
3.053
3.046
3.021
3.010
2.990
2.918
2.893
2.872
2.847
2.836
2.730
2.727
1.283
1.109
t-Bu
H2N
(Rpr,S)-11i
or
(Spr,S)-11i
BRUKER
Current Data Parameters
NAME 11ib
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210923
Time 17.38
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 90.5
DW 48.400 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.02 0.95 1.02 1.00 1.00 0.99 1.00 1.01 1.02 1.03 1.71 2.08 2.15 1.10 1.07 1.07 9.15


77.30
77.04
76.79
67.58
34.94
34.33
34.11
33.65
31.49
26.38
BRUKER
Current Data Parameters
NAME 11ib
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210924
Time 8.01
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 294.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



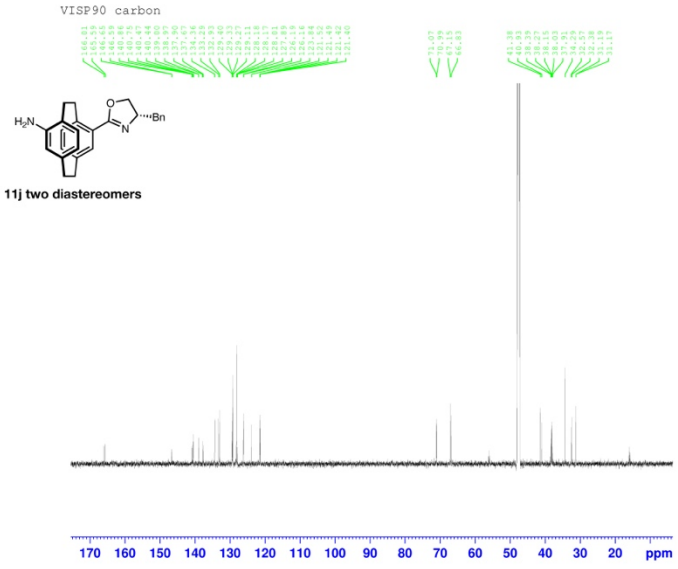
BRUKER

```
Current Data Parameters
NAME        500-VI-SP-90
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20211204
Time               20.23
INSTRUM            spect
PROBHD   5 mm PAQXI 1H/
PULPROG            zg30
TD                65536
SOLVENT            MeOD
NS                   64
DS                    2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719425 sec
RG                  114
DW               48.400 usec
DE                6.50 usec
TE               296.4 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.50 usec
PL1                4.00 dB
PL1W        12.10000038 W
SFO1        500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF          500.1300104 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

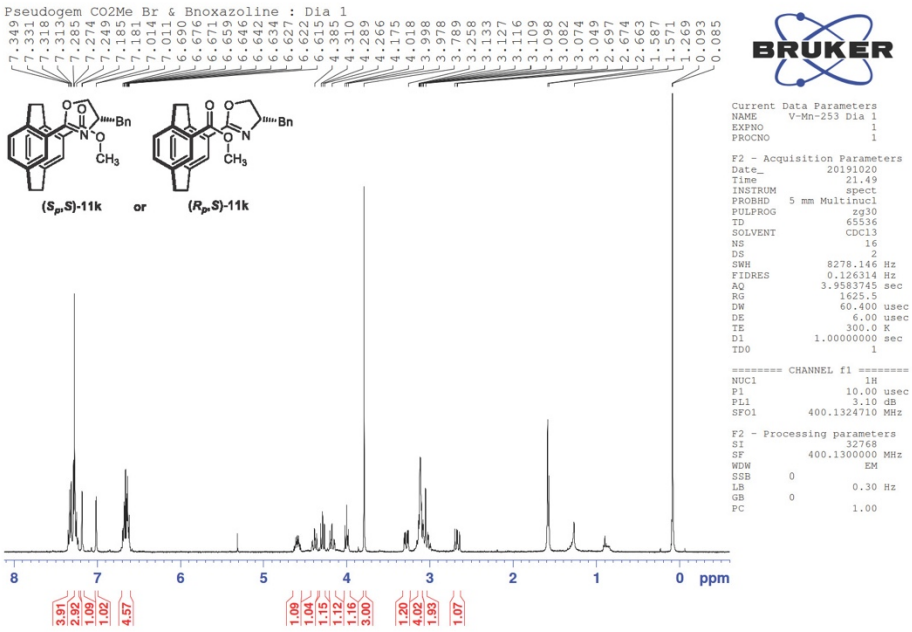


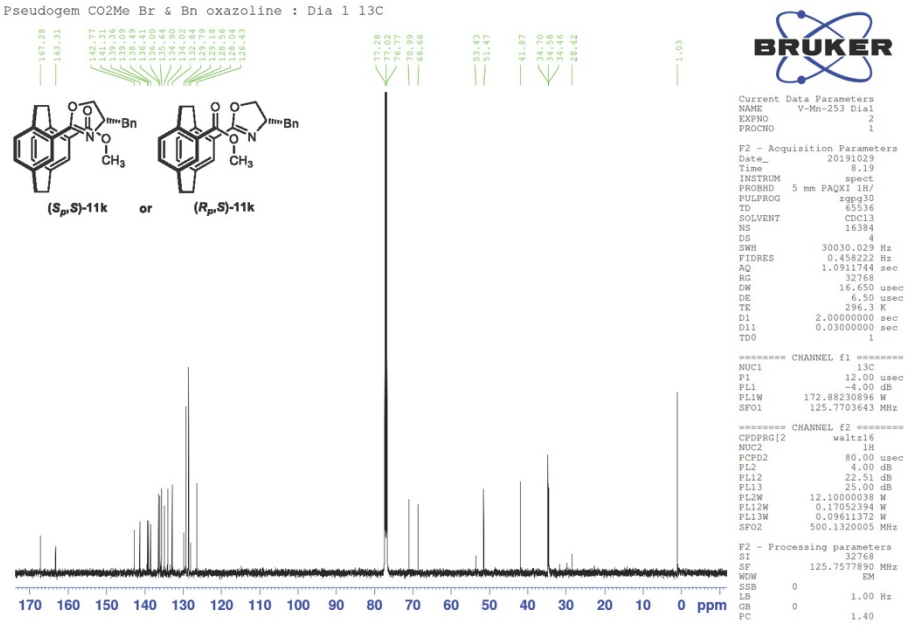
BRUKER

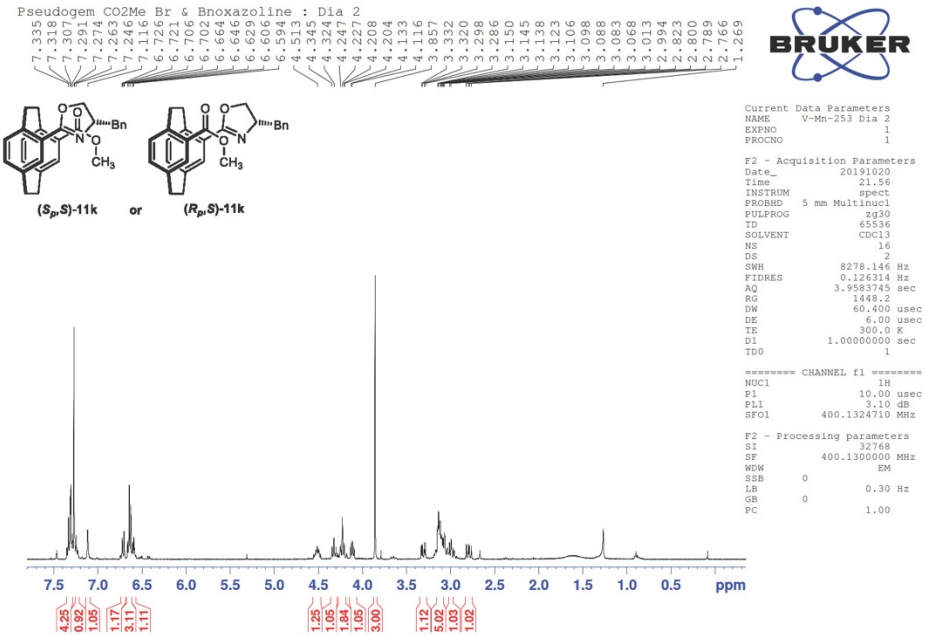
```
Current Data Parameters
NAME        suraj_ViSP90
EXPNO                  2
PROCNO                 1

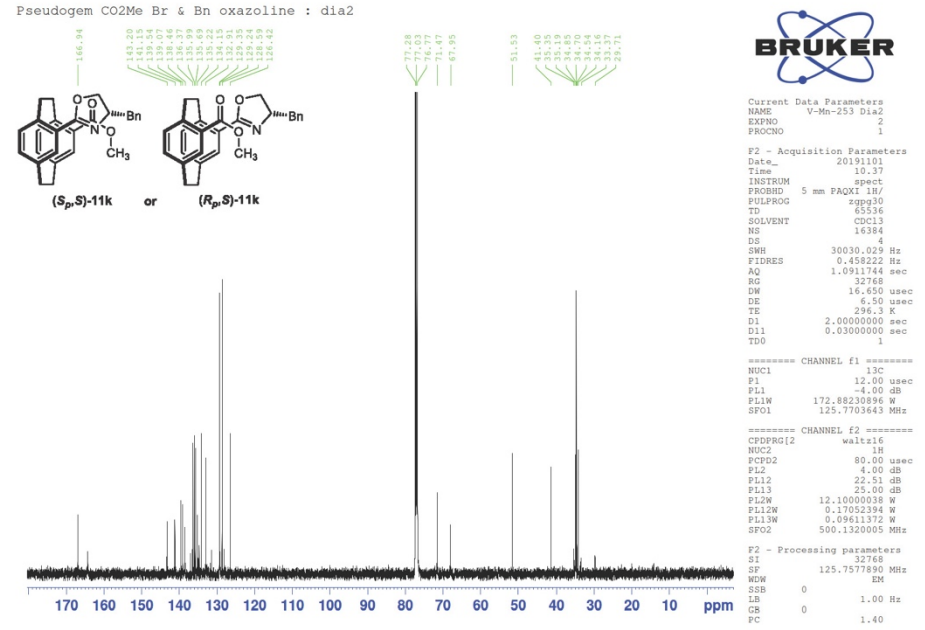
F2 - Acquisition Parameters
Date_           20211209
Time              15.38 h
INSTRUM           Avance
PROBHD   Z44908_0010 (C
PULPROG          zgpg30
TD                65536
SOLVENT            MeOD
NS                 4972
DS                    4
SWH           41666.668 Hz
FIDRES        1.271566 Hz
AQ            0.7864320 sec
RG                  101
DW               12.000 usec
DE                18.00 usec
TE               298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   24
SFO1        176.0654333 MHz
NUC1                  13C
P0                 5.00 usec
P1                15.00 usec
PLW1       141.05000305 W
SFO2        700.1328005 MHz
NUC2                  1H
CPDPRG[2          waltz65
PCPD2             65.00 usec
PLW2         8.93029976 W
PLW12        0.11835570 W
PLW13        0.05931989 W

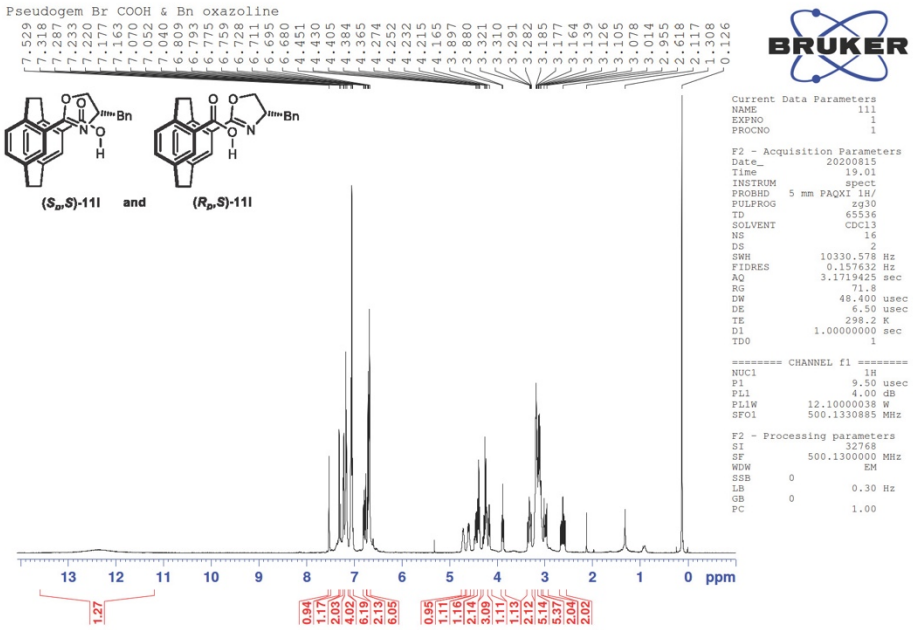
F2 - Processing parameters
SI                 32768
SF          176.0478285 MHz
WDW                   EM
SSB                    0
LB                  1.50 Hz
GB                     0
PC                  1.40
```

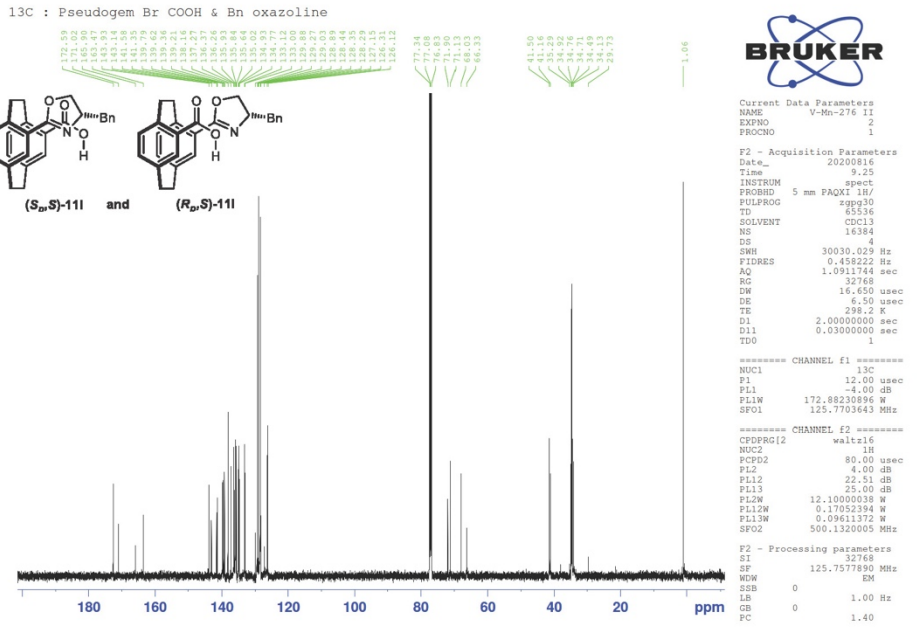

Pseudogem CO2Me Br & Bnoxazoline : Dia 1
7.349 7.331 7.318 7.313 7.285 7.274 7.249 7.185 7.181 7.011 7.014 6.690 6.676 6.671 6.659 6.646 6.642 6.634 6.627 6.622 6.615 4.385 4.310 4.289 4.266 4.175 4.018 3.998 3.978 3.789 3.258 3.133 3.127 3.116 3.109 3.098 3.082 3.074 3.049 2.697 2.674 2.663 1.587 1.571 1.269 0.093 0.085
(Sp,S)-11k or (Rp,S)-11k
8 7 6 5 4 3 2 1 0 ppm
3.91 2.92 1.09 1.02 4.57 1.09 1.04 1.15 1.12 1.16 3.00 1.20 4.02 1.93 1.07
Current Data Parameters
NAME V-Mn-253 Dia 1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191020
Time 21.49
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 1625.5
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.10 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

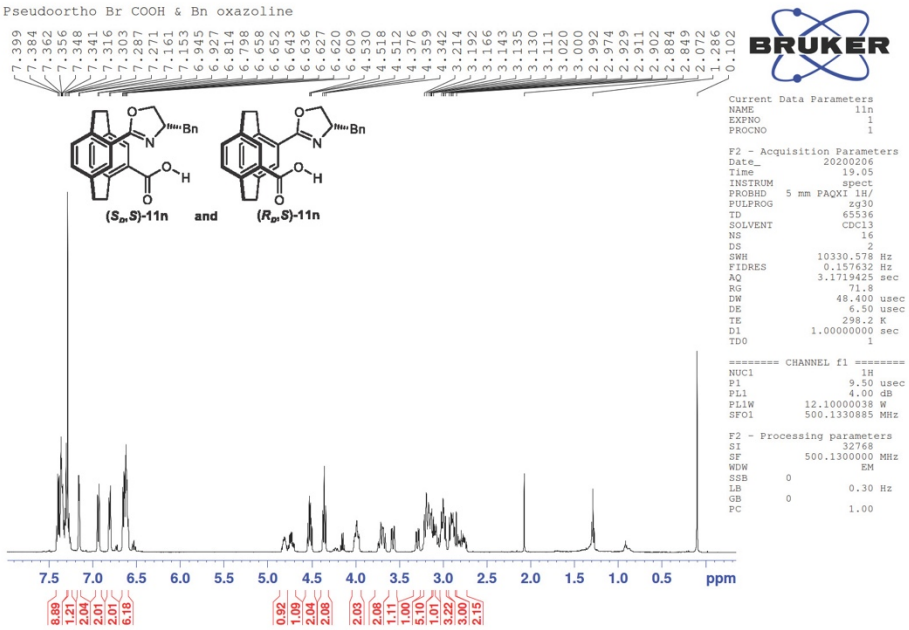

Pseudogem CO2Me Br & Bn oxazoline : Dia 1 13C
167.28 163.31 140.77 140.31 139.36 139.09 138.40 136.41 136.00 135.66 134.46 134.02 132.84 132.79 129.18 128.56 128.04 126.43 77.28 77.02 76.77 70.99 68.66 53.43 51.47 41.87 34.78 34.58 34.46 28.42 1.03
(Sp,S)-11k or (Rp,S)-11k
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Current Data Parameters
NAME V-Mn-253 Dia1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191029
Time 8.19
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

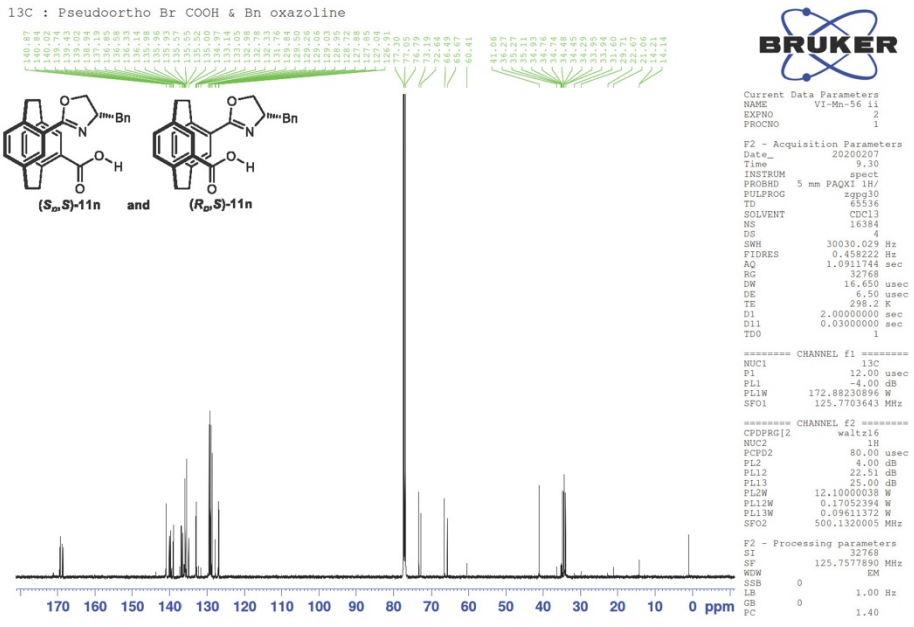

Pseudogem CO2Me Br & Bnoxazoline : Dia 2
(Sp,S)-11k or (Rp,S)-11k
Current Data Parameters
NAME V-Mn-253 Dia 2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191020
Time 21.56
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9583745 sec
RG 1448.2
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 3.10 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
4.25 0.92 1.05 1.17 3.11 1.11 1.25 1.05 1.84 1.05 3.00 1.12 5.02 1.03 1.02

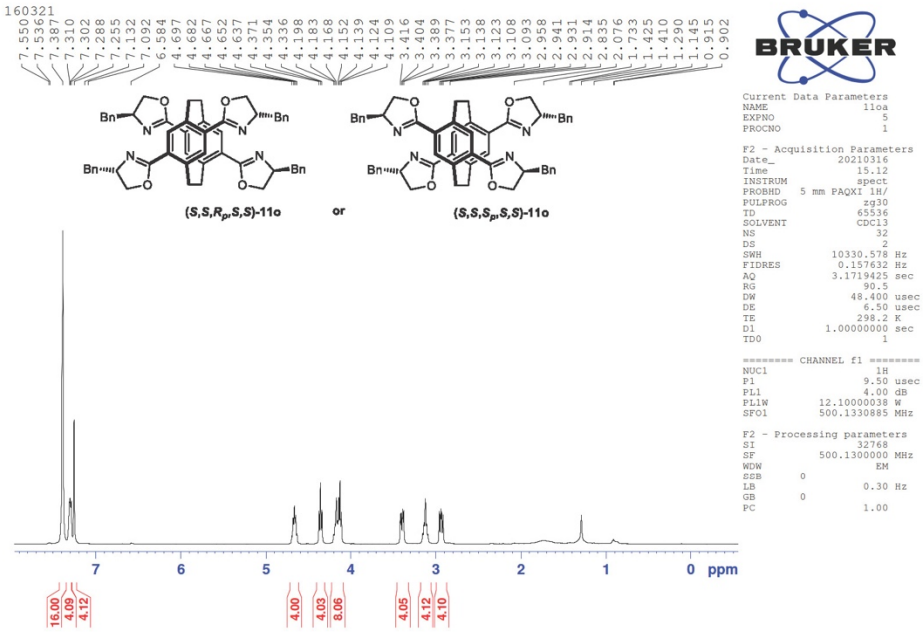

Pseudogem CO2Me Br & Bn oxazoline : dia2
(Sp,S)-11k or (Rp,S)-11k
Current Data Parameters
NAME V-Mn-253 Dia2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20191101
Time 10.37
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Pseudogem Br COOH & Bn oxazoline
(S_D,S)-11l and (R_D,S)-11l
Current Data Parameters
NAME 111
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200815
Time 19.01
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 71.8
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm


13C : Pseudogem Br COOH & Bn oxazoline
(S_D,S)-11l and (R_D,S)-11l
Current Data Parameters
NAME V-Mn-276 II
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200816
Time 9.25
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.50 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm


Pseudoortho Br COOH & Bn oxazoline
(S,S)-11n and (R,S)-11n
Current Data Parameters
NAME 11n
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200206
Time 19.05
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 71.8
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


13C : Pseudoortho Br COOH & Bn oxazoline
(S,S)-11n and (R,S)-11n
Current Data Parameters
NAME VI-Mn-56 ii
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200207
Time 9.30
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



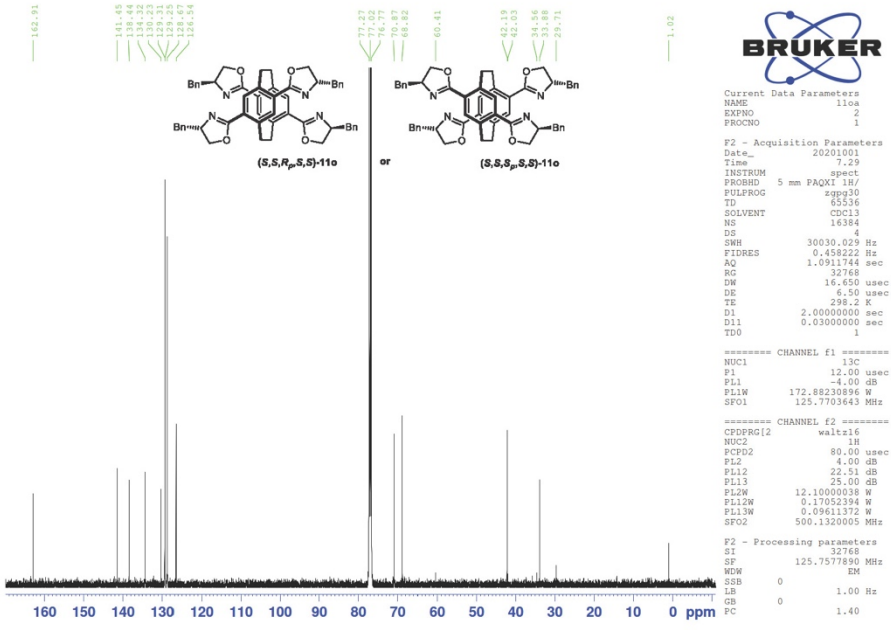
BRUKER

```
Current Data Parameters
NAME                  11oa
EXPNO                    5
PROCNO                   1

F2 - Acquisition Parameters
Date_              20210316
Time                 15.12
INSTRUM              spect
PROBHD    5 mm PAQXI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      2
SWH             10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1719425 sec
RG                   90.5
DW                48.400 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.50 usec
PL1                 4.00 dB
PL1W         12.10000038 W
SFO1        500.1330885 MHz

F2 - Processing parameters
SI                   32768
SF          500.1300000 MHz
WDW                     EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



BRUKER

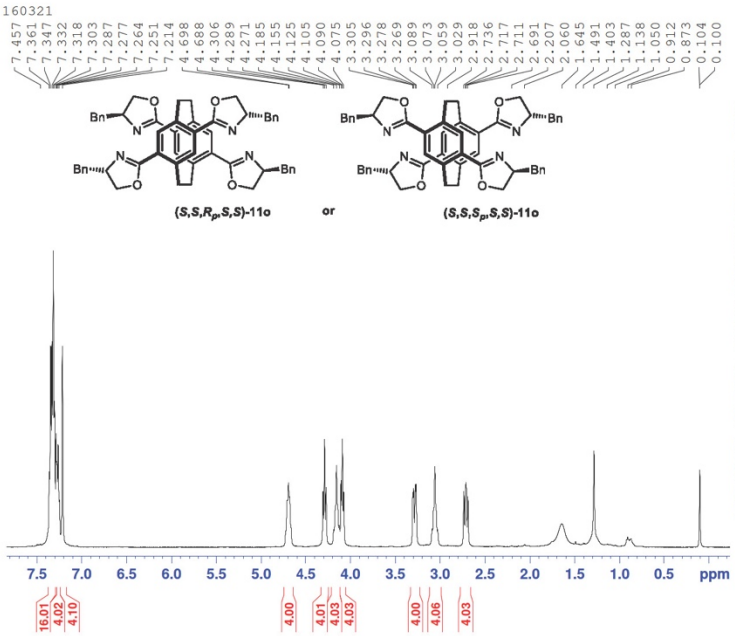
```
Current Data Parameters
NAME                  11oa
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20201001
Time                  7.29
INSTRUM              spect
PROBHD    5 mm PAQXI 1H/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  16384
DS                      4
SWH             30030.029 Hz
FIDRES          0.458222 Hz
AQ              1.0911744 sec
RG                   32768
DW                16.650 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.00 usec
PL1                -4.00 dB
PL1W        172.88230896 W
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 4.00 dB
PL12               22.51 dB
PL13               25.00 dB
PL2W         12.10000038 W
PL12W         0.17052394 W
PL13W         0.09611372 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                   32768
SF          125.7577890 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



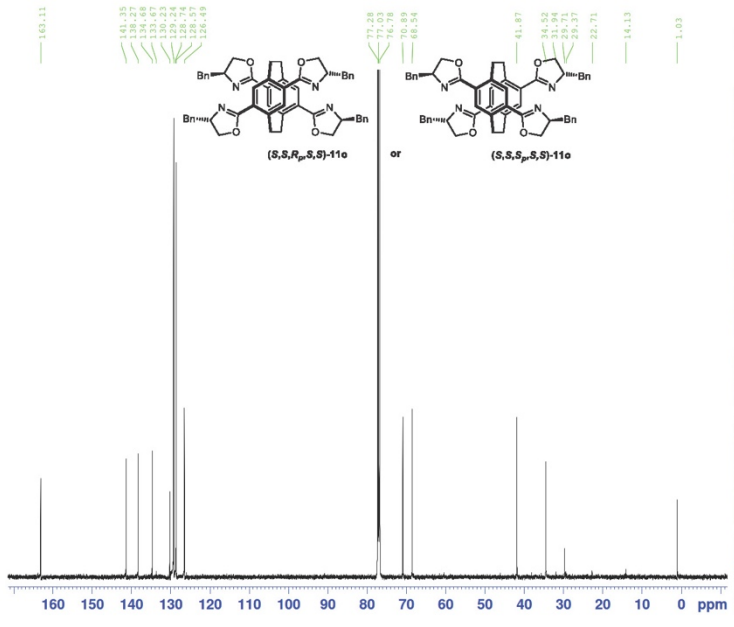
BRUKER

```
Current Data Parameters
NAME                  11ob
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_              20210316
Time                 15.21
INSTRUM              spect
PROBHD    5 mm PAQXI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      32
DS                       2
SWH             10330.578 Hz
FIDRES              0.157632 Hz
AQ                3.1719425 sec
RG                     128
DW                  48.400 usec
DE                   6.50 usec
TE                  298.2 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.50 usec
PL1                  4.00 dB
PL1W            12.10000038 W
SFO1           500.1330885 MHz

F2 - Processing parameters
SI                   32768
SF              500.1300000 MHz
WDW                     EM
SSB          0
LB                   0.30 Hz
GB           0
PC                   1.00
```



BRUKER

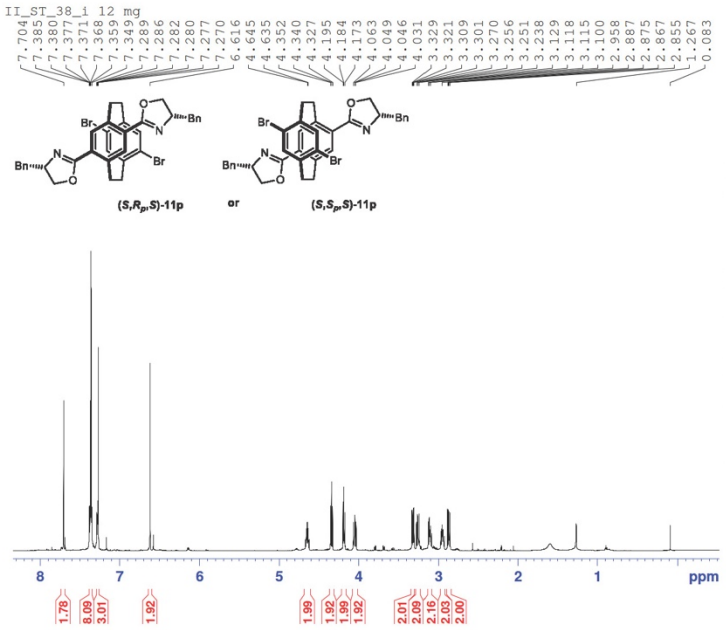
```
Current Data Parameters
NAME                  11ob
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20201003
Time                  7.07
INSTRUM              spect
PROBHD    5 mm PAQXI 1H/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   16384
DS                       4
SWH             30030.029 Hz
FIDRES              0.458222 Hz
AQ                1.0911744 sec
RG                   32768
DW                  16.650 usec
DE                   6.50 usec
TE                  298.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                  12.00 usec
PL1                 -4.00 dB
PL1W           172.88230896 W
SFO1           125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                  4.00 dB
PL12                22.51 dB
PL13                25.00 dB
PL2W            12.10000038 W
PL12W            0.17052394 W
PL13W            0.09611372 W
SFO2           500.1320005 MHz

F2 - Processing parameters
SI                   32768
SF              125.7577890 MHz
WDW                     EM
SSB          0
LB                   1.00 Hz
GB           0
PC                   1.40
```

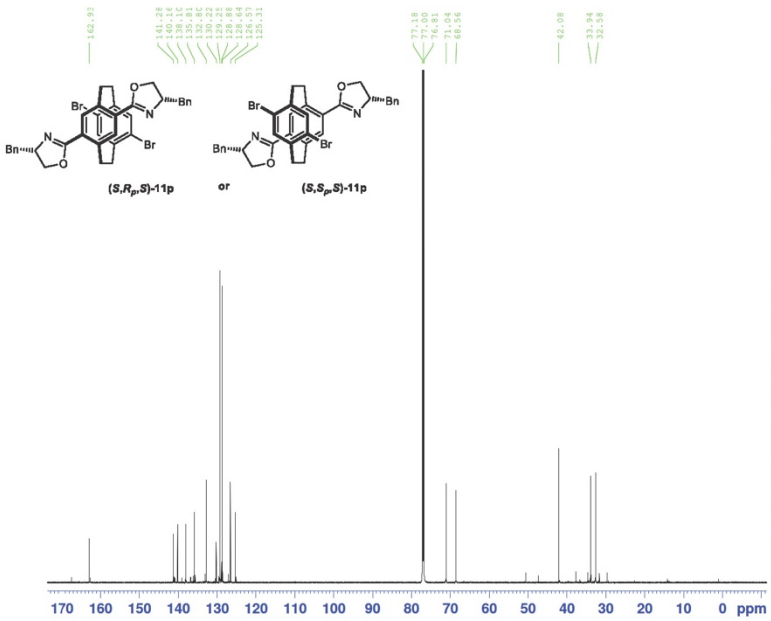



```
Current Data Parameters
NAME                  11pa
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20200806
Time                 16.39
INSTRUM              spect
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                       4
DS                       2
SWH              14492.754 Hz
FIDRES           0.221142 Hz
AQ               2.2609921 sec
RG                    35.9
DW                34.500 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.00 usec
PL1                 6.90 dB
PL1W          10.71880245 W
SFO1         700.1343236 MHz

F2 - Processing parameters
SI                   32768
SF            700.1300133 MHz
WDW                     EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



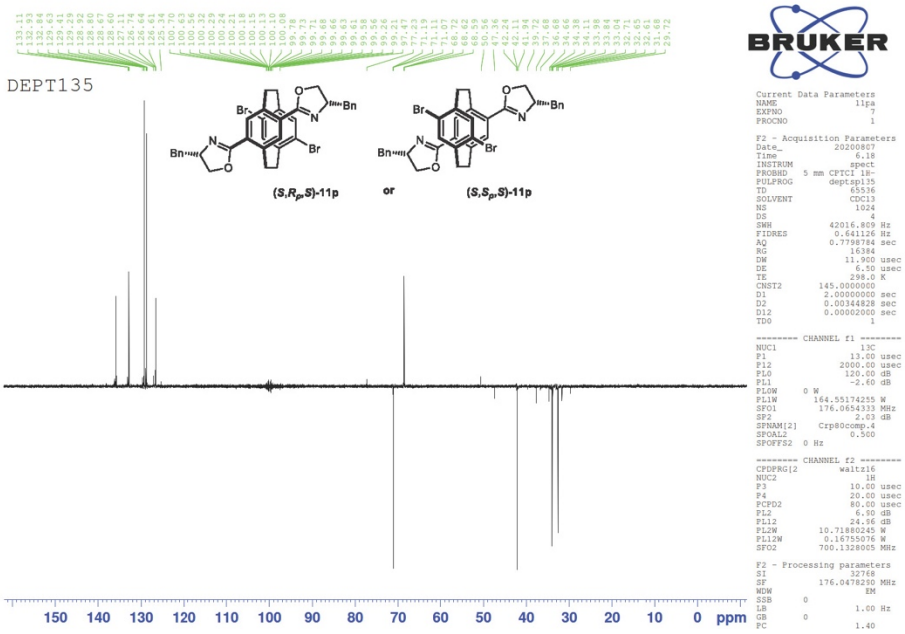

```
Current Data Parameters
NAME                  11pa
EXPNO                    6
PROCNO                   1

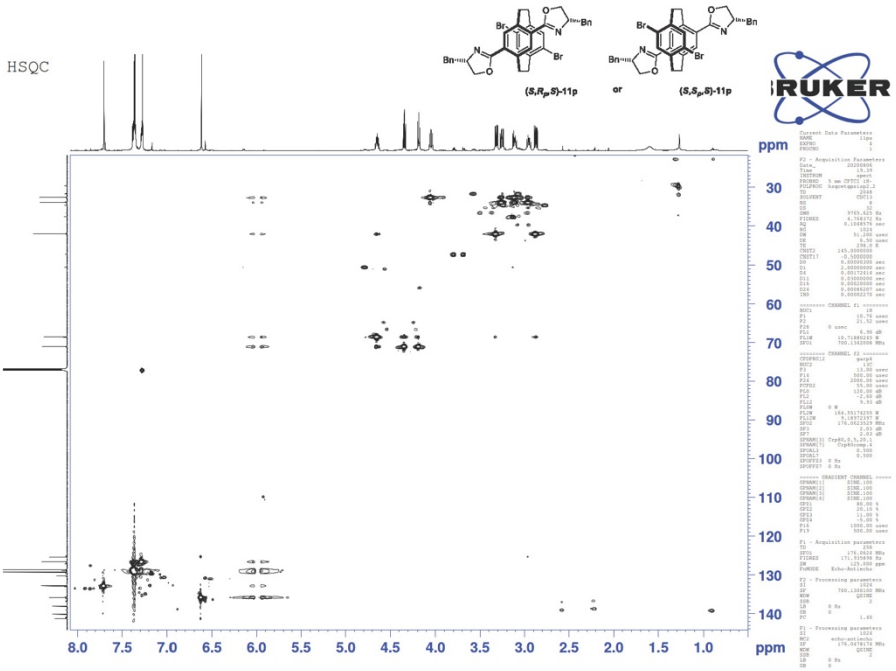
F2 - Acquisition Parameters
Date_              20200807
Time                  2.15
INSTRUM              spect
PROBHD    5 mm CPTCI 1H-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    3192
DS                       4
SWH              42016.809 Hz
FIDRES           0.641126 Hz
AQ               0.7798784 sec
RG                   32768
DW                11.900 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      2

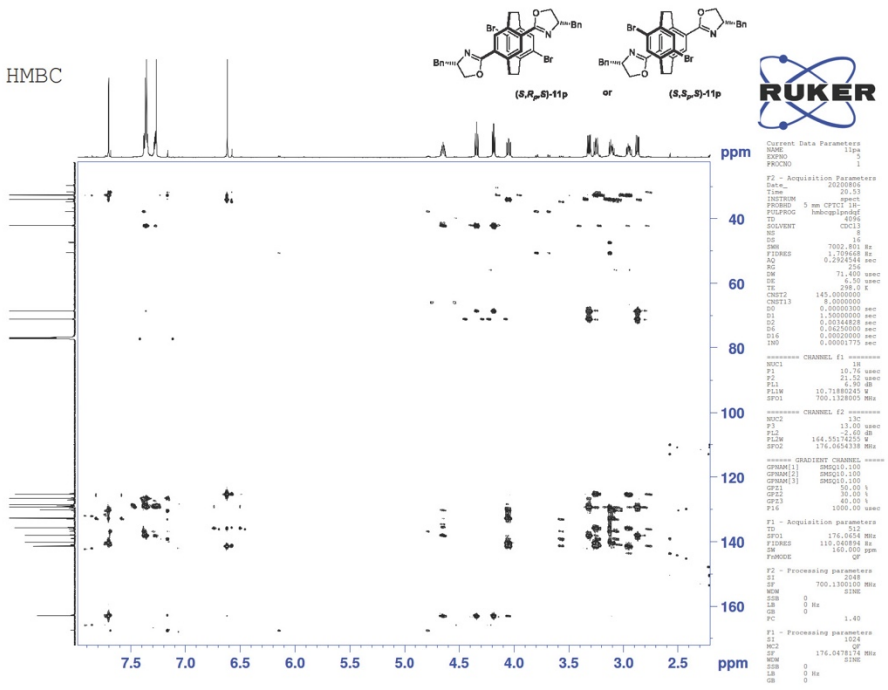
======== CHANNEL f1 ========
NUC1                  13C
P1                 13.00 usec
PL1                -2.60 dB
PL1W         164.55174255 W
SFO1         176.0654333 MHz

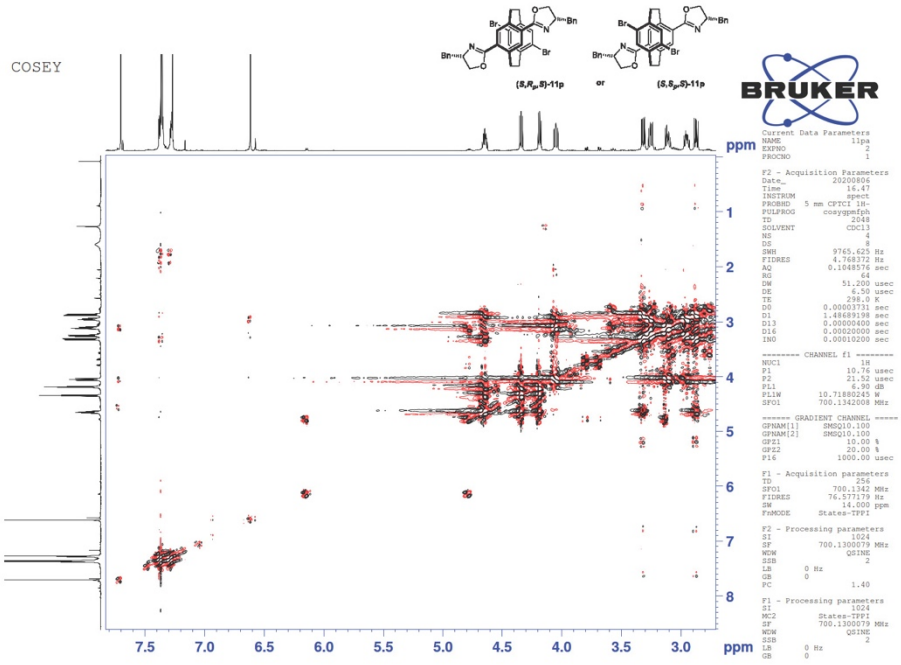
======== CHANNEL f2 ========
CPDPRG[2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                 6.90 dB
PL12               24.96 dB
PL13               19.50 dB
PL2W          10.71880245 W
PL12W          0.16755076 W
PL13W          0.58904201 W
SFO2         700.1323005 MHz

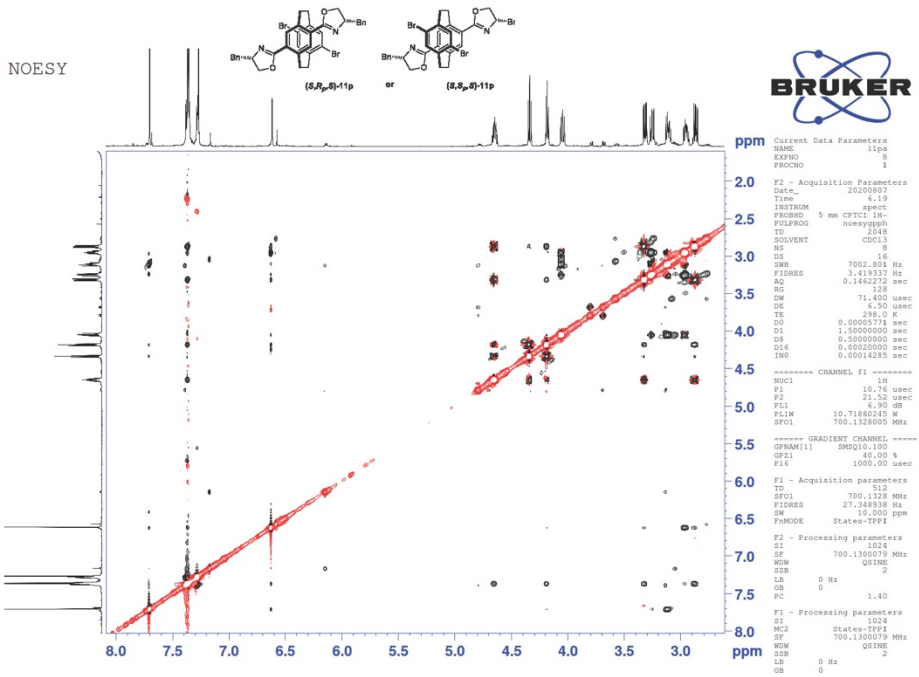
F2 - Processing parameters
SI                   32768
SF            176.0478348 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

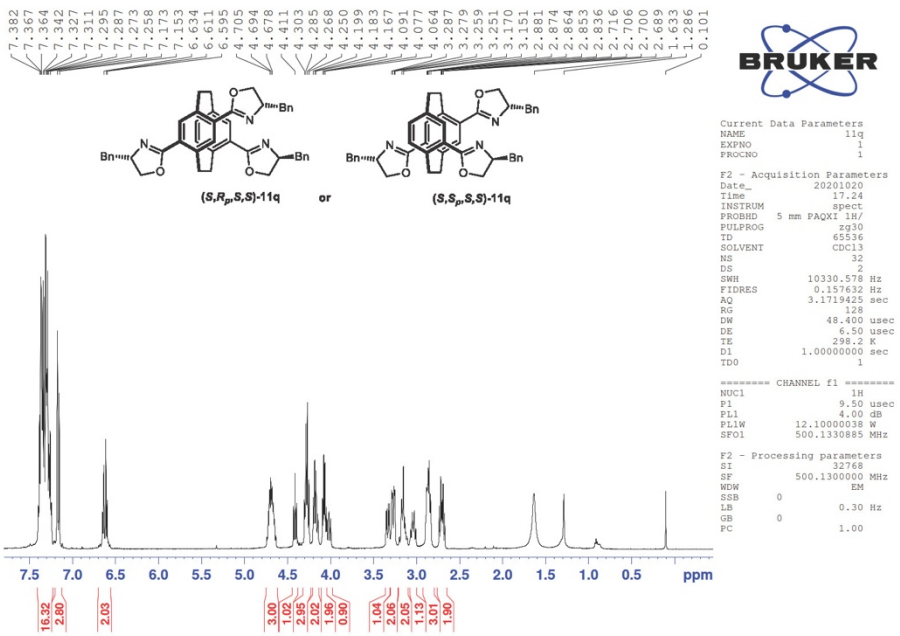

DEPT135
(S,Rp,S)-11p
or
(S,Sp,S)-11p
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

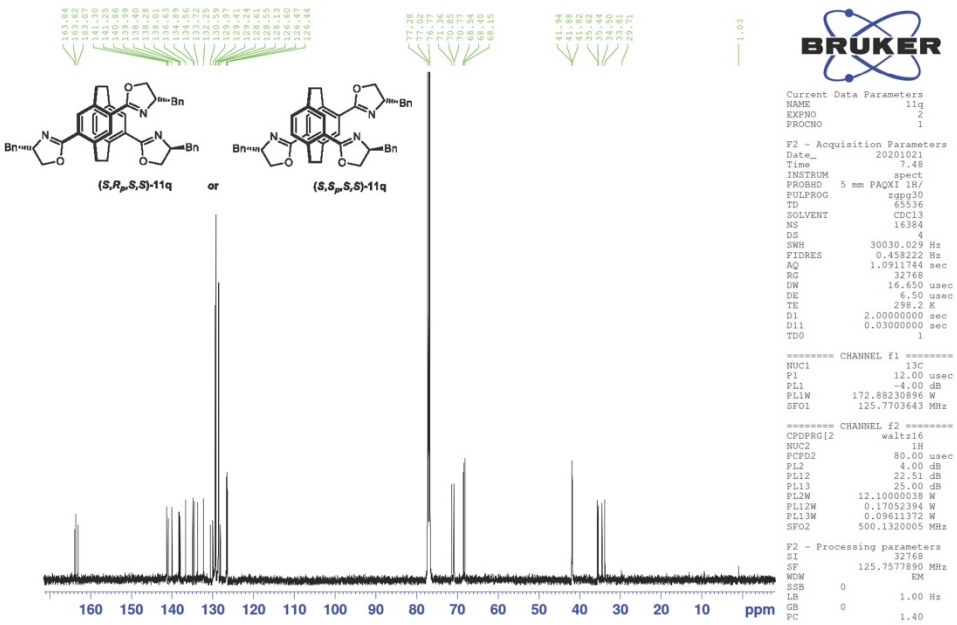

HSQC
(S,Rp,S)-11p
or
(S,Sp,S)-11p
ppm
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

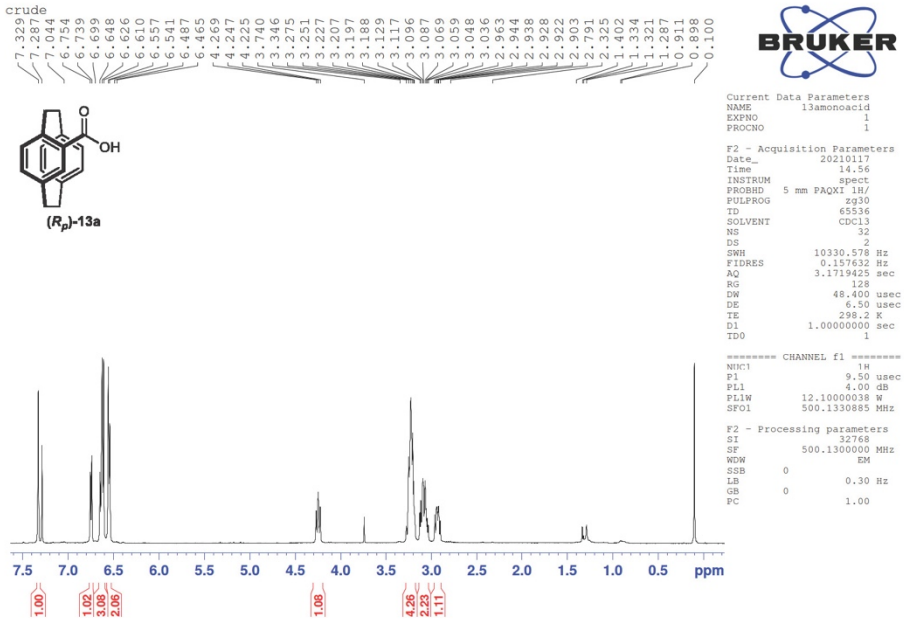

HMBC
(S,Rp,S)-11p
or
(S,Sp,S)-11p
BRUKER
ppm
40
60
80
100
120
140
160
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm

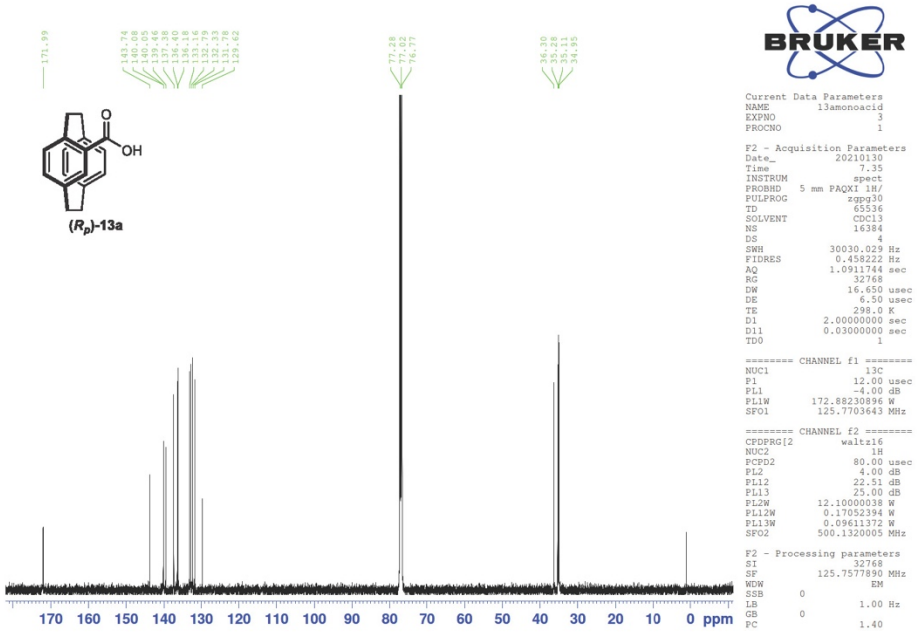

COSEY
(S,Rp,S)-11p
or
(S,Sp,S)-11p
BRUKER
ppm
1
2
3
4
5
6
7
8
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm

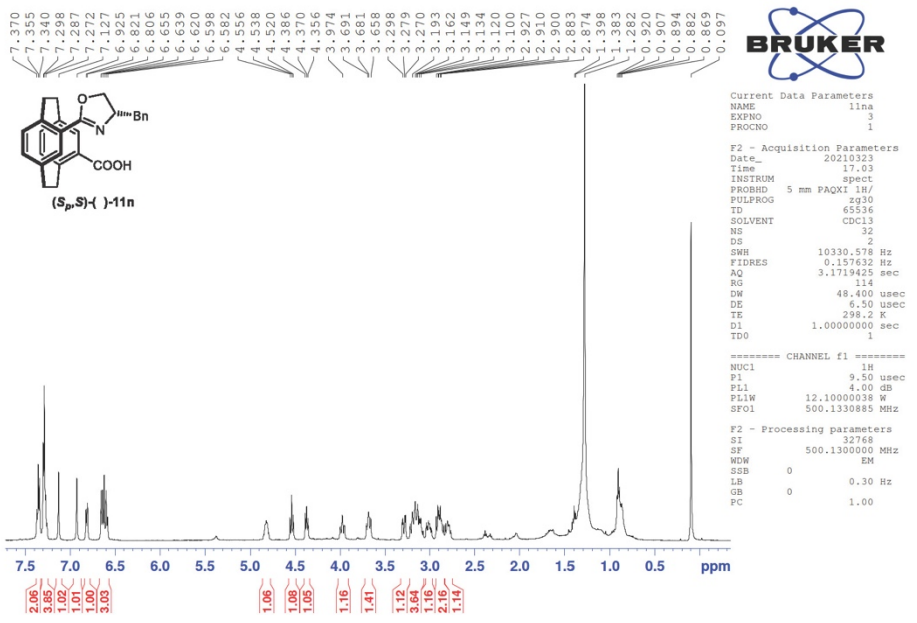

NOESY
(S,Rp,S)-11p
or
(S,Sp,S)-11p
BRUKER
ppm
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 ppm
Current Data Parameters
NAME 11pa
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200807
Time 6.19
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG noesygpph
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 7002.801 Hz
FIDRES 3.419337 Hz
AQ 0.1462272 sec
RG 128
DW 71.400 usec
DE 6.50 usec
TE 298.0 K
D0 0.00005771 sec
D1 1.50000000 sec
D8 0.50000000 sec
D16 0.00020000 sec
IN0 0.00014285 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.76 usec
P2 21.52 usec
PL1 6.90 dB
PL1W 10.71880245 W
SFO1 700.1328005 MHz
====== GRADIENT CHANNEL =====
GPNAM[1] SMSQ10.100
GPZ1 40.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 512
SFO1 700.1328 MHz
FIDRES 27.348938 Hz
SW 10.000 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 1024
SF 700.1300079 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 700.1300079 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0

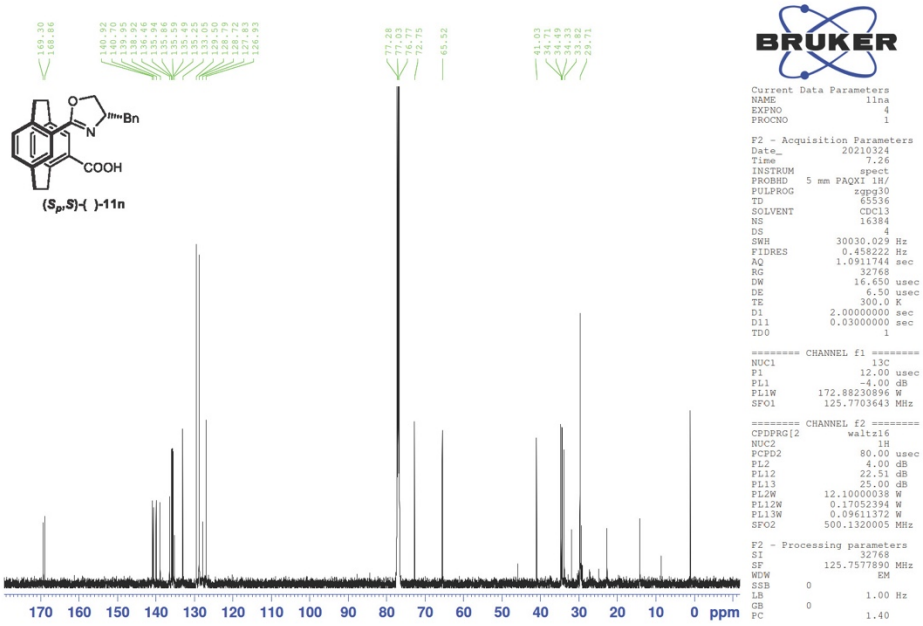

7.382 7.367 7.364 7.342 7.327 7.311 7.295 7.287 7.273 7.258 7.173 7.153 6.634 6.611 6.595 4.705 4.694 4.678 4.411 4.303 4.285 4.268 4.250 4.199 4.183 4.167 4.091 4.077 4.064 3.287 3.279 3.259 3.251 3.170 3.151 2.881 2.874 2.864 2.853 2.836 2.716 2.706 2.700 2.689 1.633 1.286 0.101
(S,Rp,S,S)-11q
or
(S,Sp,S,S)-11q
BRUKER
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
16.32 2.80 2.03 3.00 1.02 2.95 2.02 1.96 0.90 1.04 2.06 2.05 1.13 3.01 1.90
Current Data Parameters
NAME 11q
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20201020
Time 17.24
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 128
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

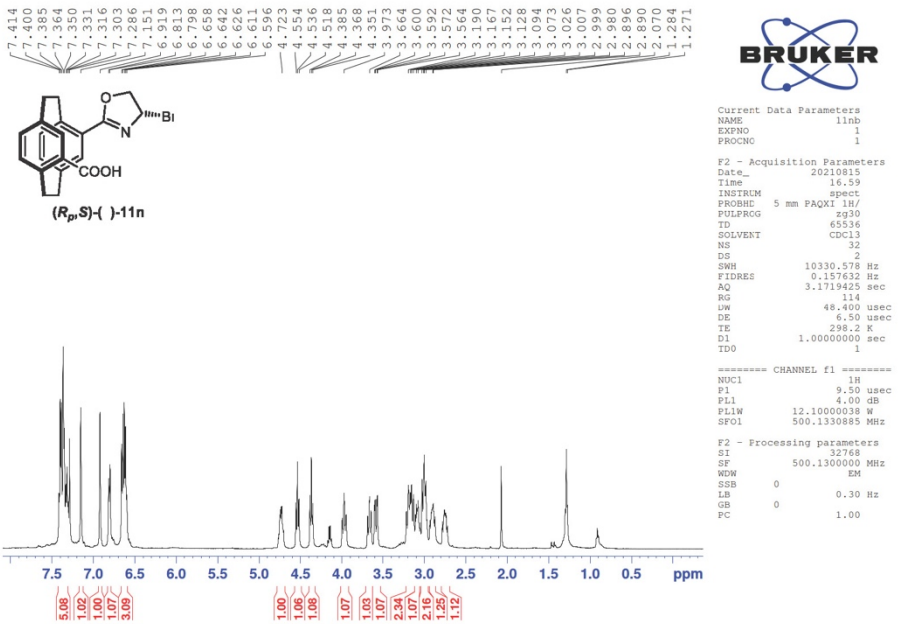

(S,Rp,S,S)-11q
or
(S,Sp,S,S)-11q
ppm

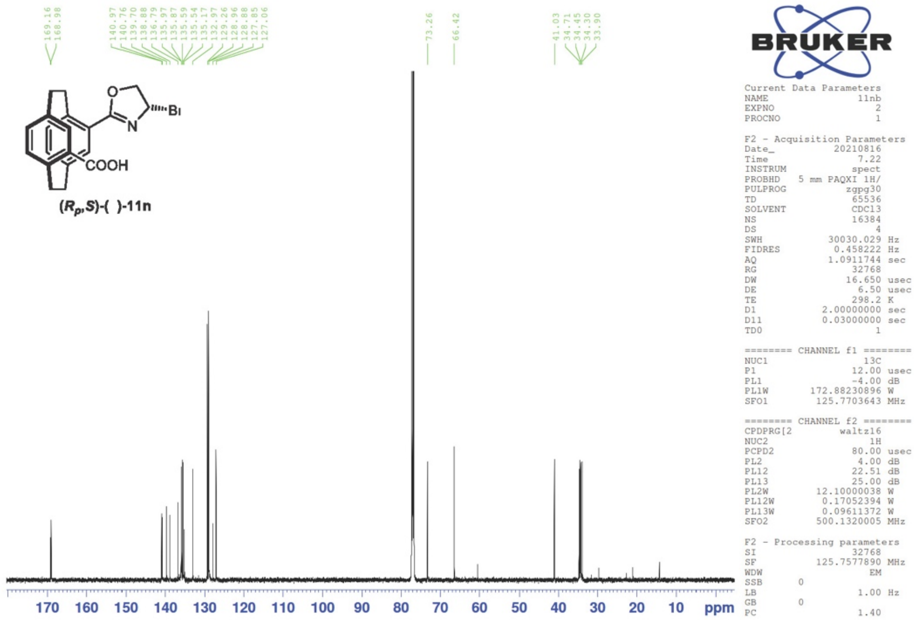

crude
(Rp)-13a
ppm

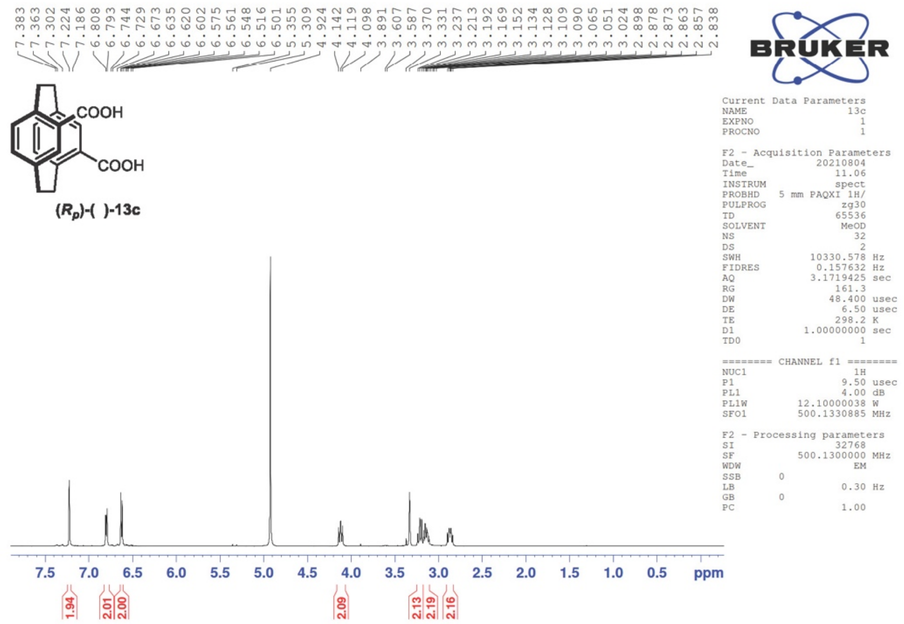

171.99
143.74
140.68
140.05
139.46
137.38
136.60
136.18
133.16
132.79
132.33
131.78
129.62
77.28
77.02
76.77
36.30
35.38
35.11
34.95
O
OH
(Rp)-13a
BRUKER
Current Data Parameters
NAME 13amonoacid
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210130
Time 7.35
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

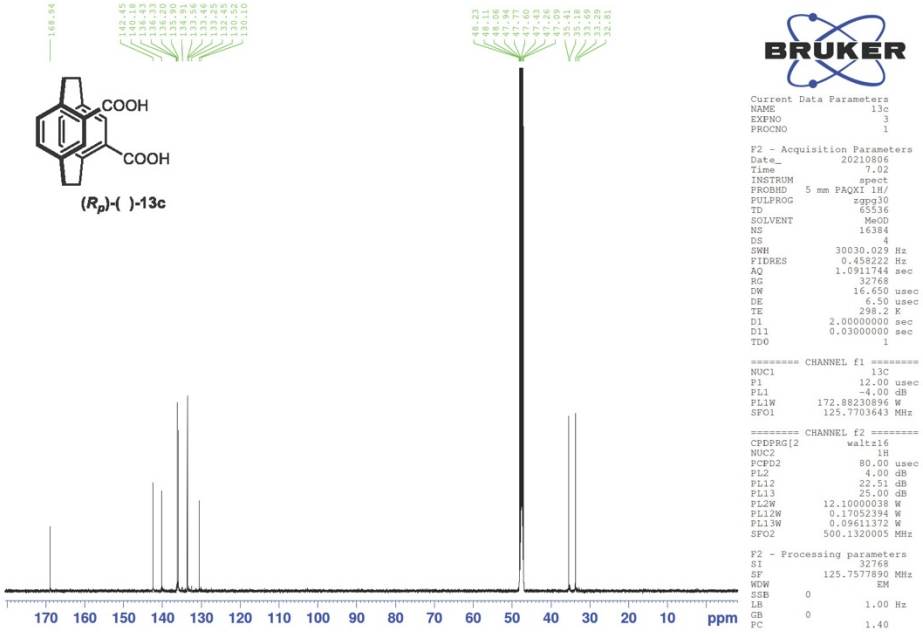

7.370
7.355
7.340
7.298
7.287
7.272
7.127
6.925
6.821
6.806
6.655
6.639
6.620
6.598
6.582
4.556
4.538
4.520
4.386
4.370
4.356
3.974
3.691
3.681
3.658
3.298
3.279
3.270
3.193
3.162
3.149
3.134
3.120
3.100
2.927
2.910
2.900
2.883
2.874
1.398
1.383
1.282
0.920
0.907
0.894
0.882
0.869
0.097
O
N
Bn
COOH
(Sp,S)-( )-11n
BRUKER
Current Data Parameters
NAME 11na
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210323
Time 17.03
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 114
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.06
3.85
1.02
1.01
1.00
3.03
1.06
1.08
1.05
1.16
1.41
1.12
3.64
1.16
2.16
1.14

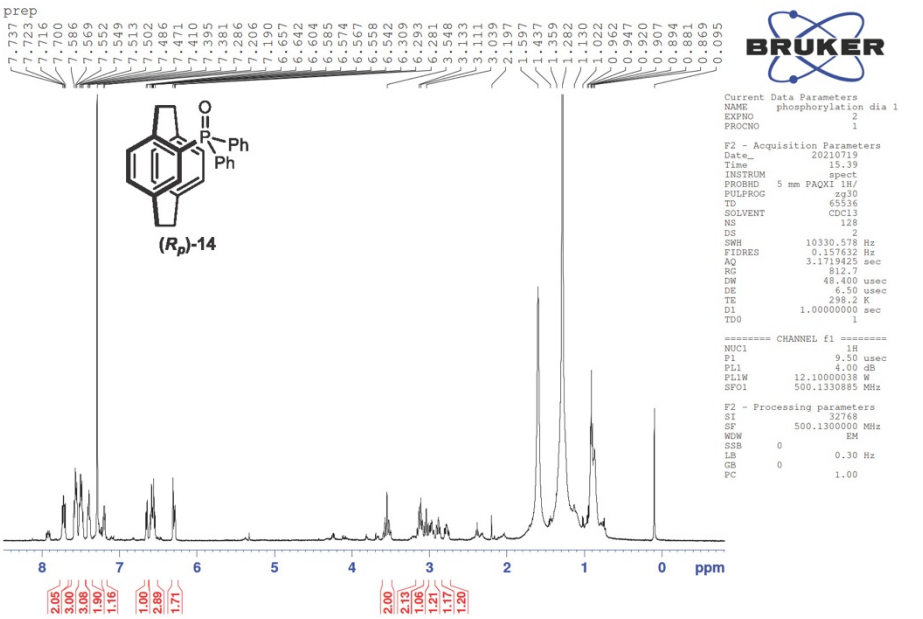

169.30
168.86
140.92
140.70
139.95
138.42
136.46
135.94
135.86
135.59
135.49
133.25
133.05
132.60
128.79
128.72
127.83
126.93
77.28
77.03
76.77
72.75
65.52
41.03
34.71
34.49
34.33
33.82
29.71
Bn
COOH
(Sp,S)-( )-11n
BRUKER
Current Data Parameters
NAME 11na
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210324
Time 7.26
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

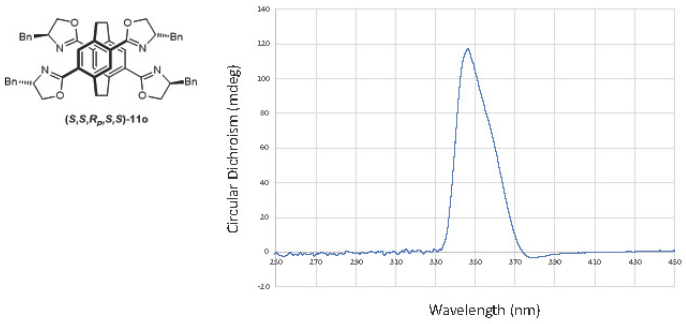

7.414
7.400
7.385
7.364
7.350
7.331
7.316
7.303
7.286
7.151
6.919
6.813
6.798
6.658
6.642
6.626
6.611
6.596
4.723
4.554
4.536
4.518
4.385
4.368
4.351
3.973
3.664
3.600
3.592
3.572
3.564
3.190
3.167
3.152
3.128
3.094
3.073
3.026
3.007
2.999
2.980
2.896
2.890
2.070
1.284
1.271
Bl
COOH
(Rp,S)-( )-11n
BRUKER
Current Data Parameters
NAME 11nb
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210815
Time 16.59
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 114
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
5.08
1.02
1.00
1.07
3.09
1.00
1.06
1.08
1.07
1.03
1.07
2.34
1.07
2.16
1.25
1.12


169.16
168.98
140.97
140.76
139.70
138.88
136.79
135.97
135.87
135.59
135.54
135.17
132.97
129.26
128.96
128.88
127.85
127.06
73.26
66.42
41.03
34.71
34.45
34.30
33.90
COOH
(Rp,S)-( )-11n
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
BRUKER
Current Data Parameters
NAME 11nb
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210816
Time 7.22
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

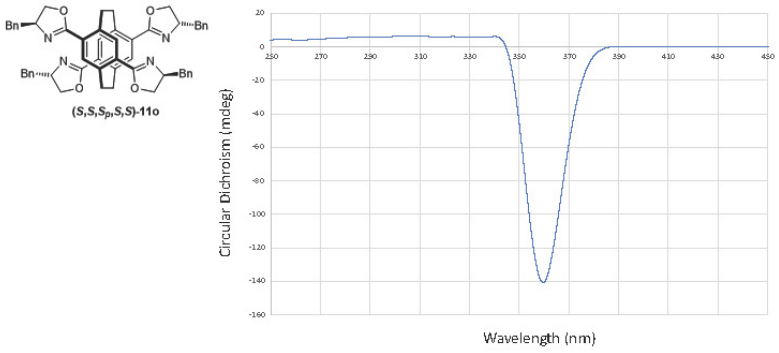

7.383
7.363
7.302
7.224
7.186
6.808
6.793
6.744
6.729
6.673
6.635
6.620
6.602
6.575
6.561
6.548
6.516
6.501
5.355
5.309
4.924
4.142
4.119
4.098
3.891
3.607
3.587
3.370
3.331
3.237
3.213
3.192
3.169
3.152
3.134
3.128
3.109
3.090
3.065
3.051
3.024
2.898
2.878
2.873
2.863
2.857
2.838
COOH
COOH
(Rp)-( )-13c
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.94
2.01
2.00
2.09
2.13
2.19
2.16
BRUKER
Current Data Parameters
NAME 13c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210804
Time 11.06
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 32
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 161.3
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


168.94
142.45
140.18
136.43
136.33
136.20
135.90
134.91
133.56
133.46
133.26
132.45
130.52
130.10
48.23
48.11
48.06
47.94
47.77
47.60
47.43
47.26
47.09
36.41
35.18
33.69
33.29
32.81
COOH
COOH
(Rp)-( )-13c
BRUKER
Current Data Parameters
NAME 13c
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210806
Time 7.02
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 16384
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0911744 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
PL1W 172.88230896 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 22.51 dB
PL13 25.00 dB
PL2W 12.10000038 W
PL12W 0.17052394 W
PL13W 0.09611372 W
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


prep
7.737
7.723
7.716
7.700
7.586
7.569
7.552
7.549
7.513
7.502
7.486
7.471
7.410
7.395
7.381
7.286
7.206
7.190
6.657
6.642
6.604
6.585
6.574
6.567
6.558
6.542
6.309
6.293
6.281
3.548
3.133
3.111
3.039
2.197
1.597
1.437
1.359
1.282
1.130
1.022
0.962
0.947
0.920
0.907
0.894
0.881
0.869
0.095
O
P
Ph
Ph
(Rp)-14
BRUKER
Current Data Parameters
NAME phosphorylation dia 1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20210719
Time 15.39
INSTRUM spect
PROBHD 5 mm PAQXI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 128
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719425 sec
RG 812.7
DW 48.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 4.00 dB
PL1W 12.10000038 W
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8 7 6 5 4 3 2 1 0 ppm
2.05
3.00
3.08
1.90
1.16
1.00
2.89
1.71
2.00
2.13
1.06
1.21
1.17
1.20



CD spectrum of (*S, S,R*$_p$,*S,S*)-4,7,12,15-Tetra-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane



CD spectrum of (*S, S,S*$_p$,*S,S*)-4,7,12,15-Tetra-(4′-benzyl-oxazolin-2′-yl)[2.2]paracyclophane