Rational Design of Chiral Selenium-π-Acid Catalysts

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Supporting Information

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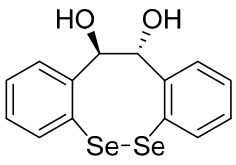
2 General Remarks

Chemicals were obtained from commercial sources and were used without further purification. Yields correspond to isolated compounds unless indicated otherwise. Purity is estimated to be ≥95% based on ^{1}H-NMR spectroscopic analysis. Irradiation experiments were performed at λ = 465 nm using commercially available blue LED strips (see experimental setup picture below). The light intensity applied was in the range of 3500-4500 lx. TLC: MACHEREY-NAGEL, TLC plates Alugram® Sil G/UV254. Visualization of the developed chromatogram was performed by fluorescence quenching at 254 nm and staining with potassium permanganate. Chromatography: Separations were carried out on Merck Silica 60 (0.063–0.200 mm, 70–230 mesh ASTM) using forced flow. GPC: Japan Analytical Industries (JAI) LC-92XX II Series, UV- and RI-detector, column: JAIGEL HH series; IR: Bruker FT-IR Alpha-spectrometer and JASCO FT/IR-4600 with ATR sampling module; High resolution mass spectrometry (HR-MS): APEX IV 7T FTICR, BRUKER Daltonic. NMR (^{1}H, ^{13}C, ^{77}Se, ^{11}B, ^{31}P) spectra were recorded at 300, 400, 500 MHz (^{1}H) and 75, 101, 126 MHz (^{13}C, APT (Attached Proton Test)), respectively, on VARIAN Unity-300, AMX 300, Inova 400 and Inova 500 instruments in $CDCl_3$ solutions at 298 K, if not specified otherwise. Chemical shifts (δ) are given in ppm. Multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sex = sextet, sept = septet, m = multiplet). Melting point: KRÜSS Melting Point Meter M5000; HPLC: Agilent Technologies 1290 Infinity; Kontron A.

3 Synthetic Procedures

3.1 Synthesis of diselenocines

3.1.1 (11*R*,12*R*)-11,12-Dihydrodibenzo[c,g][1,2]diselenocin-11,12-diol (6)[1]

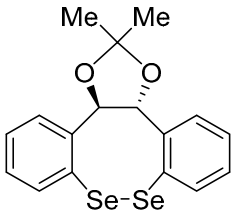


To a suspension of (*R*,*R*)-hydrobenzoin (**5**) (1.00 g, 4.66 mmol, 1.00 equiv.) in *n*-hexane (25 mL) and Et_2O (18 mL) *n*-BuLi (1.92 M in hexane, 14.6 mL, 1.79 g, 28.0 mmol, 6.00 equiv.) was added dropwise at rt. The resulting mixture was refluxed for 16 h at 50 °C. After cooling to rt selenium (1.84 g, 23.3 mmol, 5.00 equiv.) and THF (18 mL) were added and the mixture was stirred for further 1.5 h at 50 °C. After cooling to rt the mixture was poured into ice water (100 mL) and stirred for 1 h under air. The phases were separated and the aqueous phase was extracted with DCM (3 x 20 mL). The combined org. phases were washed with H_2O (3 x 20 mL), dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 5:1 PE/EtOAc) provided the title product as a yellow solid (581 mg, 1.57 mmol, 34 %).

TLC: R_f = 0.19 (PE/EtOAc, 5:1); **T_m:** 210-213 °C; **IR** (ATR): $\tilde{\nu}$ = 3430, 3236, 2543, 2430, 1441, 1329, 1247, 1191, 1111, 1056, 898, 759, 734, 695 cm^{-1}; **^{1}H-NMR** (300 MHz, DMSO-D_6): δ (ppm) =7.77 (dd, ^{3}J = 7.6 Hz, ^{4}J = 1.3 Hz, 2 H), 7.62 (dd, ^{3}J = 7.6 Hz, ^{4}J = 1.6 Hz, 2 H), 7.50 (ddd, ^{3}J = 7.6,

7.6 Hz, ^4J = 1.3 Hz, 2 H), 7.27 (ddd, ^3J = 7.6, 7.6 Hz, ^4J = 1.6 Hz, 2 H), 5.67 (d, ^3J = 6.6 Hz, 2 H), 5.22 (d, ^3J = 6.6 Hz, 2 H); **^{13}C-NMR** (75 MHz, DMSO-D_6): δ (ppm) = 152.3, 135.4, 130.0, 127.4, 126.7, 125.0, 73.9; **^{77}Se NMR** (76 MHz, DMSO-D_6) δ = 461; **HR-MS** (ESI): calc. for $C_{14}H_{12}NaO_2Se_2$ ([M + Na]$^+$): 394.9063, found: 394.9056; **optical rotation:** α^{D}_{20} = –208° (c = 1.00, MeOH).

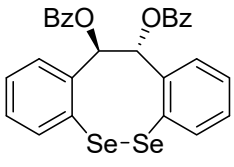
3.1.2 (3a*R*,13b*R*)-2,2-Dimethyl-3a,13b-dihydrodibenzo[3,4:7,8][1,2]diselenocino-[5,6-d][1,3]dioxol (7a) [2]



To a suspension of (11*R*,12*R*)-11,12-Dihydrodibenzo[c,g][1,2]diselenocin-11,12-diol (**6**) (595 mg, 1.61 mmol, 1.00 equiv.) in 2,2-dimethoxypropane (1.98 mL, 1.68 g, 16.1 mmol, 10.0 equiv.) a drop of aq. HCl (37%) was added and the resulting mixture was stirred for 16 h at rt. One drop of NEt_3 was added and the solvent was evaporated. The residue was dissolved in $CHCl_3$ (10 mL), filtered through celite and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 50:1 PE/Et_2O) provided the title product as a yellow solid (427 mg, 1.04 mmol, 65 %).

TLC: R_f = 0.13 (PE/Et_2O, 50:1); **T_m:** 106-109 °C; **IR** (ATR): $\tilde{\nu}$ = 3047, 2979, 1454, 1369, 1240, 1205, 1061, 1025, 872, 753 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) =7.85 (dd, ^3J = 7.8 Hz, ^4J = 1.5 Hz, 2 H), 7.72 (dd, ^3J = 7.5 Hz, ^4J = 1.4 Hz, 2 H), 7.42 (ddd, ^3J = 7.8, 7.5 Hz, ^3J = 1.4 Hz, 2 H), 7.19 (ddd, ^3J = 7.5, 7.5 Hz, ^3J = 1.5 Hz, 2 H), 5.85 (s, 2 H), 1.75 (s, 6 H) ; **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 148.2, 136.2, 130.3, 129.6, 127.8, 127.6, 111.6, 84.7, 28.3 ; **^{77}Se NMR** (76 MHz, $CDCl_3$) δ = 472.95; **HR-MS** (ESI): calc. for $C_{17}H_{16}NaO_2Se_2$ ([M + Na]$^+$): 434.9376, found: 434.9367; **optical rotation:** α^{D}_{20} = –100° (c = 1.00, $CHCl_3$).

3.1.3 (11*R*,12*R*)-11,12-Dihydrodibenzo[c,g][1,2]diselenocin-11,12-diyldibenzoat (7d)[3]

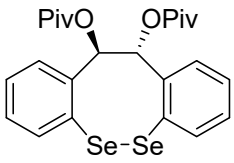


To a solution of (11*R*,12*R*)-11,12-dihydrodibenzo[c,g][1,2]diselenocin-11,12-diol (**6**)(50 mg, 0.14 mmol, 1.0 equiv.) and DMAP (1.6 mg, 14 µmol, 0.10 equiv.) in pyridine (1.5 mL), benzoyl chloride (156 µL, 190 mg, 1.35 mmol, 10.0 equiv.) was added at 0 °C. The solution was warmed to 40 °C and stirred for 24 h. Sat. aq. $NaHCO_3$-sol. (2.5 mL) was added and the solution was extracted with DCM (3 x 5 mL). The combined org. phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (PE/EtOAc, 10:1) provided the title product as a yellow solid (36 mg, 62 µmol, 46 %).

TLC: R_f = 0.49 (PE/EtOAc, 10:1); **T_m:** 180-185 °C; **IR** (ATR): $\tilde{\nu}$ = 3060, 1722, 1451, 1246, 1094, 1068, 1025, 761, 706 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 8.20 (d, ^3J = 7.6 Hz, 4 H), 7.85 (d, ^3J = 7.5 Hz, 2 H), 7.16-7.67 (m, 12 H), 7.09 (s, 2 H) ; **^{13}C-NMR** (125 MHz, $CDCl_3$): δ (ppm) = 164.9, 147.1, 136.4, 133.4, 130.2, 129.9, 129.7, 128.6, 128.5, 128.4, 128.3, 127.6, 124.8, 76.1; **HR-**

MS (ESI): calc. for $C_{28}H_{20}NaO_4Se_2$ ([M + Na]$^+$): 602.9590, found: 602.9525; **optical rotation:** $\alpha^{D_{20}}$ = +135° (c = 1.00, $CHCl_3$).

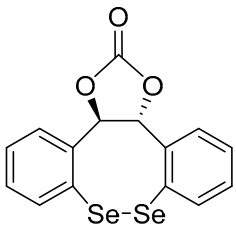
3.1.4 (11*R*,12*R*)-11,12-Dihydrodibenzo[c,g][1,2]diselenocin-11,12-diylbis(2,2-dimethylpropanoat) (7b)[3]



To a solution of (11*R*,12*R*)-11,12-dihydrodibenzo[c,g][1,2]diselenocin-11,12-diol (**6**) (70 mg, 0.19 mmol, 1.00 equiv.) and DMAP (1.6 mg, 14 µmol, 0.10 equiv.) in pyridine (1.5 mL), pivaloyl chloride (228 mg, 1.89 mmol, 10.0 equiv.) was added at 0 °C. The solution was warmed to 40 °C and stirred for 24 h. Sat. aq. $NaHCO_3$-sol. (2.5 mL) was added and the solution was extracted with DCM (3 x 5 mL). The combined org. phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (PE/EtOAc, 10:1) provided the title product as a yellow solid (100 mg, 186 µmol, 98 %).

TLC: R_f = 0.37 (PE/EtOAc, 10:1); **T_m:** 140-150 °C (decomposition); **IR** (ATR): $\tilde{\nu}$ = 2973, 1734, 1278, 1129, 1114, 1038, 759, 735, 448 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.80 (dd, ^3J = 7.6 Hz, ^4J = 1.3 Hz, 2 H), 7.16-7.40 (m, 6 H), 6.71 (s, 2 H), 1.31 (s, 18 H); **^{13}C-NMR** (125 MHz, $CDCl_3$): δ (ppm) = 176.7, 147.5, 136.4, 130.1, 128.3, 128.2, 123.4, 75.6, 39.1, 27.4; **HR-MS** (ESI): calc. for $C_{24}H_{32}NO_4Se_2$ ([M+NH_4]$^+$): 558.0661, found: 558.0630; **optical rotation:** $\alpha^{D_{20}}$ = +3° (c = 1.00, $CHCl_3$).

3.1.5 (3a*R*,13b*R*)-3a,13b-Dihydrodibenzo[3,4:7,8][1,2]diselenocino[5,6-d][1,3]dioxol-2-on (7c)[4]

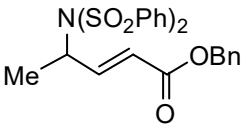


To a solution of (11*R*,12*R*)-11,12-Dihydrodibenzo[c,g][1,2]diselenocin-11,12-diol (**6**) (50 mg, 0.14 mmol, 1.0 equiv.) in DCM (1 mL) bis(trichlormethyl) carbonate (44 mg, 0.15 mmol, 1.1 equiv.) and NEt_3 (41 µL, 30 mg, 300 µmol, 2.30 equiv.) were added and the resulting mixture was stirred for 2 h at RT. H_2O (2 mL) was added and the mixture was extracted with DCM (3 x 5 mL). The combined org. phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (PE/EtOAc, 10:1) provided the title product as a yellow solid (32 mg, 81 µmol, 60 %).

TLC: R_f = 0.39 (PE/EtOAc, 10:1); **T_m:** 245-252 °C (decomposition); **IR** (ATR): = $\tilde{\nu}$ = 3051, 2922, 1822, 1798, 1143, 1067, 989, 744, 449 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.81 (m, 2 H), 7.50-7.54 (m, 4 H), 7.34 (m, 2 H), 6.21 (s, 2 H); **^{13}C-NMR** (125 MHz, $CDCl_3$): δ (ppm) = 154.8, 143.8, 136.9, 130.7, 129.3, 128.1, 124.4, 82.4; **^{77}Se NMR** (76 MHz, $CDCl_3$) δ (ppm) = 465.16; **HR-MS** (ESI): calc for $C_{15}H_{14}NO_3Se_2$ ([M+NH_4]$^+$): 415.9302, found.: 415.9281; **optical rotation:** $\alpha^{D_{20}}$ = –430° (c = 1.00, $CHCl_3$).

3.2 Asymmetric imidation

3.2.1 (*E*)-Benzyl-4-(*N*-(phenylsulfonyl)phenylsulfonamid)pent-2-enoat (3)[5]



To a solution of (*E*)-benzylpent-3-enoate (**1**) (50 mg, 0.26 mmol, 1.00 equiv.), NFSI (**2**) (83 mg, 260 µmol, 1.0 equiv.), and 4 Å molecular sieves (spatula tip) in the corresponding solvent (1.5 mL), the catalyst (13 µmol, 5 mol%) was added. The resulting suspension was stirred for 16 h at rt. The solvent was removed under reduced pressure and column chromatography (SiO_2, 10:1→3:1 PE/Et_2O) provided the title product as a colorless solid.

Table 1: Conditions used in the asymmetric imidation

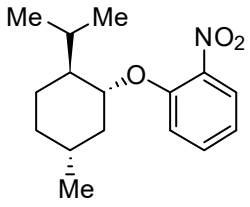
entry	solvent	catalyst	m (product)	n (product)	yield	*ee*
1	THF	**7a**	37 mg	76 µmol	29 %	19 %
2	1,4-dioxane	**7a**	22 mg	45 µmol	17 %	15 %
3	DCM	**7a**	23 mg	47 µmol	18 %	18 %
4	$MeNO_2$	**7a**	20 mg	42 µmol	16 %	8 %
5	MeCN	**7a**	64 mg	0.13 mmol	50 %	3 %
6	Toluol	**7a**	20 mg	42 µmol	16 %	14 %
7	THF/MeCN (9:1)	**7a**	47 mg	97 µmol	37 %	7 %
8	MTBE	**7a**	36 mg	74 µmol	28 %	16 %
9	Et_2O	**7a**	34 mg	71 µmol	27 %	14 %
10	cyclohexane	**7a**	-	-	0 %	-
11	THF	**7d**	66 mg	0.14 mmol	52 %	16 %
12	THF	**7d**	63 mg	0.13 mmol	49 %	8 %
13	THF	**7c**	103 mg	213 µmol	81 %	50 %

TLC: R_f = 0.11 (PE/ Et_2O, 3:1); **IR** (ATR): $\tilde{\nu}$ = 3067, 2937, 1721, 1448, 1377, 1354, 1084, 1165, 850, 720, 684, 546 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.93-8.12 (m, 4 H), 7.61 (m, 2 H), 7.45-7.57 (m, 4 H), 7.29-7.44 (m, 5 H), 7.00 (dd, ^3J = 15.9 Hz, 5.6 Hz, 1 H), 5.79 (dd, ^3J = 15.9 Hz, ^4J = 1.8 Hz, 1 H), 4.91 (qdd, ^3J = 7.0, 5.6 Hz, ^4J = 1.8 Hz, 1 H), 5.17 (s, 2 H), 1.54 (d, ^3J = 6.9 Hz, 3

H); **^{13}C-NMR** (125 MHz, $CDCl_3$): δ (ppm) = 18.8, 58.1, 66.4, 122.8, 128.3, 128.5, 128.6, 128.9, 129.0, 133.9, 135.7, 139.9, 146.0, 165.3; **HR-MS** (ESI): calc. for $C_{24}H_{23}NO_6S_2$ ([M+H]$^+$): 486.1040, found: 486.1038; **HPLC:** 22.734 min., 25.738 min. (Daicel Chiralpak IA; eluent *n*-hexane/*i*-PrOH, 90:10; flow rate: 0.8 mL/min.).

3.3 Synthesis of alkoxycatalysts

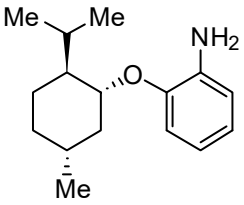
3.3.1 1-(((1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl)oxy)-2-nitrobenzene [6]



Sodium hydride (60 w% in mineral oil, 1.91 g, 47.7 mmol, 1.50 equiv.) was suspended in dry THF (20 mL) under argon-atmosphere at 0 °C, treated with 2-fluoronitrobenzene (3.00 g, 21.0 mmol, 1.00 equiv.). A solution of (–)-menthol (4.98 g, 31.8 mmol, 1.50 equiv.) in dry THF (16 mL) was slowly added, and the mixture was allowed to warm to rt and stirred for 16 h at 60 °C. After cooling to rt, sat. aq. NH_4Cl-sol. (45 mL) was added, the aqueous phase was extracted with DCM (3 x 25 mL), the combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 20:1 pentane/DCM) provided the title product as a yellow solid (4.48 g, 16.0 mmol, 76%).

TLC: R_f = 0.71 (pentane/EtOAc: 30:1); **IR** (neat): $\tilde{\nu}$ = 2953, 2929, 2870, 2360, 1602, 1524, 1485, 1456, 1355, 1277, 1256, 1163, 984, 851, 747, 669 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.78 (dd, ^3J = 8.1 Hz, ^4J = 1.7 Hz, 1 H), 7.49 (ddd, ^3J = 8.5, 7.4, ^4J = 1.8 Hz 1 H), 7.10 (dt, ^3J = 8.5, ^4J = 0.9 Hz, 1 H), 6.98 (ddd, ^3J = 8.1, 7.4, ^4J = 1.2 Hz, 1 H), 4.22 (td, ^3J = 10.6, 4.2, 1 H), 2.30-2.18 (m, 1 H), 2.18-2.10 (m, 1 H), 1.76 (ddt, ^3J = 11.5, 4.9 Hz, ^4J = 2.8 Hz, 2 H), 1.63 (ddt, ^3J = 13.3, 10.2 Hz, ^4J = 3.2 Hz, 1 H), 1.50 (tdd, ^3J = 12.0, 6.5 Hz, ^4J = 3.3 Hz, 1 H), 1.3-1.2 (m, 2 H), 0.95 (dd, ^3J = 6.8, 1.3 Hz, 6 H), 0.77 (d, ^3J = 7.0 Hz, 3 H); **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 151.6, 133.6, 125.5, 119.6, 115.1, 79.3, 47.6, 39.7, 34.2, 31.5, 25.8, 23.5, 22.0, 20.7, 16.4) **HR-MS (ESI)**: calc. for.: $C_{16}H_{23}NO_3Na$ ([M+Na]$^+$): 300.1570 found: 300.1572; **optical rotation** $\alpha^{D_{20}}$ = –87°(c = 0.52, $CHCl_3$).

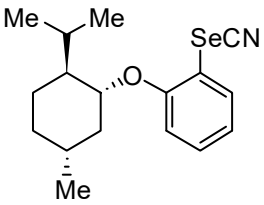
3.3.2 2-(((1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl)oxy)aniline (9b)[7]



1-(((1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl)oxy)-2-nitrobenzene (4.33 g, 15.6 mmol, 1.00 equiv.) was dissolved in ethanol/acetic acid (250 mL, 1:1), treated with iron powder (2.62 g, 46.0 mmol, 3.00 equiv.) and stirred for 3 h at 100 °C. After cooling to rt the mixture was diluted with EtOAc (275 mL) and the *pH* value was adjusted to *pH*=10 using aq. NaOH (1 M) and sat. aq. Na_2CO_3-sol. The phases were separated and the organic phase was washed with sat. aq. $NaHCO_3$-sol. (3 x10 mL). The combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 50:1 pentane/EtOAc) provided the title product as a yellow oil (2.50 g, 10.1 mmol, 65%).

TLC: R_f= 0.19 (Pent/EtOAc: 30:1); **IR** (neat): $\tilde{\nu}$ = 2955, 2925, 2867, 1612, 1503, 1456, 1275, 1217, 1038, 1012, 991, 739 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 6.84-6.64 (m, 4 H), 4.06 (d, ^3J = 4.1 Hz, 1 H), 3.76 (s, 2 H), 2.27 (qd, ^3J = 7.0, ^4J = 2.8 Hz, 1 H), 2.18 (dtd, ^3J = 12.4, 3.8, ^4J = 2.1 Hz, 1 H), 1.81-1.66 (m, 2 H), 1.66-1.36 (m, 2 H), 1.13 (m, 1 H), 1.01 (m, 1 H), 0.92 (dd, ^3J = 10.3, 6.8Hz, 7 H), 0.80 (d, ^3J = 6.9 Hz, 3 H). **^{13}C-NMR** (75 MHz, $CDCl_3$): δ (ppm) = 145.6, 137.3, 120.8, 118.4, 115.4, 113.1, 77.8, 48.1, 40.5, 34.6, 31.4, 26.1, 23.7, 22.2, 20.9, 16.7); **HR-MS** (ESI): calc. for: $C_{16}H_{26}Na$ $[M+Na]^+$: 248.2009, found: 248.2013; **optical rotation** $\alpha^{D_{20}}$ = –115° (c = 1.00, $CHCl_3$).

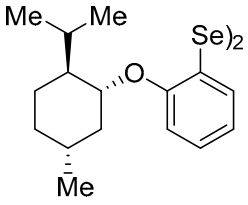
3.3.3 1-(((1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl)oxy)-2-selenocyanatobenzol (10b)



$BF_3{\cdot}OEt_2$ (4.24 mL, 4.79 g, 34.0 mmol, 3.50 equiv.) was dissolved in dry THF (65 mL) under an argon atmosphere at -30 °C (((1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl)oxy)aniline (2.50 g, 10.1 mmol, 1.00 equiv.) in dry THF (20 mL) and *tert*-butylnitrite (4.59 mL, 3.98 g, 39 mmol, 4.00 equiv.) were slowly added and the mixture was warmed to rt within 30 min and stirred for further 30 min at rt. The resulting solid was filtered off and washed with diethyl ether until it was completely white (ATTENTION: USE EXPLOSION SHIELD!). The filtrate was also treated with diethyl ether (40 mL) and the resulting solid was also filtered off and washed with diethyl ether. The combined solids were dried *in vacuo* and then dissolved in dry acetonitrile (50 mL). The solution was cooled to –20 °C and a solution of potassium selenocyanate (1.39 g, 9.64 mmol, 1.00 equiv.) in dry acetonitrile (25 mL) was slowly added. The mixture was slowly warmed to 0 °C (ice bath) and warmed to rt over 16 h. The mixture was diluted with DCM/water (100 mL, 1:1) and the phases were separated. The aqueous phase was extracted with diethyl ether (3 x 50 mL) and the combined organic phases were dried over Na_2SO_4. Removal of the solvent under reduced pressure provided the title product as an orange-red oil (2.98 g, 8.86 mmol, 88%). The crude product was used without further purification.

TLC: R_f = 0.44 (Pent/EtOAc: 30:1); **IR** (neat): $\tilde{\nu}$ = 2955, 2925, 2865, 1471, 1243, 991, 749, 679, 669, 656 cm^{-1}; **^{1}H NMR** (300 MHz, $CHCl_3$) δ (ppm) = 7.61 (dd, ^3J = 7.9 Hz, ^4J =1.5 Hz, 1 H), 7.30 (m, 1 H), 6.99 (m, 1 H), 6.88 (m, 1 H), 4.14 (td, ^3J = 10.5, 4.2 Hz, 1 H), 2.17 – 2.04 (m, 2 H), 1.79 – 1.66 (m, 2 H), 1.62 – 0.84 (m, 12 H), 0.75 (d, ^3J = 6.9 Hz, 2 H). **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 154.0, 129.6, 129.3, 122.4, 112.6, 101.8, 79.2, 47.8, 40.2, 34.3, 31.5, 26.3, 23.7, 22.1, 20.8, 16.7); **HR-MS (ESI)**: calc. for: $C_{17}H_{23}NOSeNa$ ([M+Na]$^+$): 360.0838; found: 360.0841.

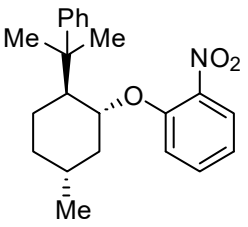
3.3.4 1,2-Bis(2-(((1*S*,2*R*,5*S*)-2-isopropyl-5-methylcyclohexyl)oxy)-phenyl)diselane (11b)[8]



1-(((1*S*,2*R*,5*S*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)-2-selenocyan-atobenzene (2.98 g, 8.86 mmol, 1.00 equiv.) was dissolved in ethanol (50 mL), treated with aq. NaOH sol. (2.4 M, 4 mL, 10.0 mmol, 1.10 equiv.) and stirred for 1 h at rt. A mixture of DCM/water (120 mL, 1:1) was added, the phases were separated, and the aqueous phase was extracted with DCM (3 x 60 mL). The combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 20:1 pentane/DCM) provided the title product as a yellow oil (900 mg, 1.45 mmol, 33%).

TLC: R_f = 0.50 (pentane/EtOAc: 30:1); **IR** (neat): $\tilde{\nu}$ = 2948, 2921, 2866, 1572, 1463, 1441, 1275, 1264, 1234, 1046, 1028, 1009, 992, 747, 668, 655 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) = 7.51 (dd, ^3J = 8.0 Hz, ^4J = 1.6 Hz, 1 H), 7.15 (ddd, ^3J = 8.2, 7.4 Hz, ^4J = 1.6 Hz, 1 H), 6.84-6.79 (m, 2 H), 4.15 (dt, ^3J = 10.5, 4.1 Hz, 1 H), 2.36 (quintd, ^3J = 7.0, ^4J = 2.8 Hz, 1 H), 2.18 (m,1 H), 1.81-1.68 (m, 2 H), 1.67-1.58 (m, 2H), 1.48 (dddd, ^3J = 15.2, 12.0, 5.8 Hz, ^4J = 3.3 Hz, 1 H), 1.21-1.05 (m, 2 H), 0.95 (dd, ^3J = 15.8, 6.8 Hz, 6 H), 0,81 (d, ^3J = 7.0 Hz, 3 H); **^{13}C-NMR** (101 MHz, $CDCl_3$): δ (ppm) = 155.3, 130.2, 127.6, 121.5, 120.2, 112.1, 78.6, 47.9, 40.3, 34.4, 31.5, 26.1, 23.6, 22.1, 20.9, 16.7; **^{77}Se-NMR** (95 MHz, $CDCl_3$): δ (ppm) =324.79; **HR-MS (ESI)**: calc. for: $C_{32}H_{46}O_2Se_2K$ ([M+K]$^+$): 661.1466, found: 661.1422; **optical rotation** $\alpha^{D_{20}}$ = –93° (c = 1.10, $CHCl_3$).

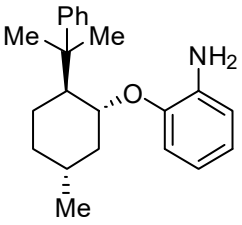
3.3.5 1-(((1*R*,2*S*,5*R*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)-2-nitrobenzene[6]



Sodium hydride (60 w% in mineral oil, 103 mg, 2.58 µmol, 1.50 equiv.) was suspended in dry THF (6 mL) under an argon atmosphere at 0 °C, treated with 2-fluoronitrobenzene (243 mg, 1.72 mmol, 1.00 equiv.). (–)-8-phenylmenthol (600 mg, 2.58 mg, 1.50 equiv.) in dry THF (2 mL) was slowly added and the mixture was allowed to warm to rt and stirred for 16 h at 60 °C. After cooling to rt, sat. aq. NH_4Cl-sol. (10 mL) was added, the aqueous phase was extracted with DCM (3 x 25 mL), the combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 20:1 pentane:DCM) provided the title product as a yellow solid (553 mg, 1.71 mmol, 99%).

TLC: R_f = 0.26 (15:1 Hex:EtOAc); **T_m:** 82 °C; **IR** (ATR): $\tilde{\nu}$ = 2925, 1604, 1525, 1483, 1353, 1279, 989, 767, 701 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) =7.73 (dd, ^3J = 8.0 Hz, ^4J = 1.8 Hz, 1 H), 7.42 (ddd, ^3J = 8.4, 7.3 Hz,^4J = 1.8 Hz, 1 H), 7.26 – 7.18 (m, 4 H), 7.12 (m, 1 H), 6.93 (ddd, ^3J = 8.1, 7.4 Hz, ^4J = 1.1 Hz, 1 H), 6.88 (d, ^3J = 8.4 Hz, 1 H), 4.22 (td, ^3J = 10.4, 4.2 Hz, 1 H), 2.08 – 1.90 (m, 2 H), 1.60 – 1.48 (m, 2 H), 1.36 (s, 7 H), 1.12 (td, ^3J = 12.6, 10.8 Hz, 1 H), 1.02 (tdd, ^3J = 13.5, 12.1, 3.8 Hz, 1 H), 0.91 – 0.78 (m, 4 H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) =150.2, 149.5, 141.0, 133.5, 127.8, 126.0, 125.6, 125.3, 119.5, 114.5, 79.0, 51.3, 40.4, 40.0, 34.5, 31.3, 29.6, 27.2, 25.6, 21.7; **HR-ESI-MS** (m/z) calc. for $C_{22}H_{27}O_3NNa$ $[M+Na]^+$: 376.1883, found: 376.1883; **optical rotation:** α^{D}_{20} = –158° (0.99, $CHCl_3$).

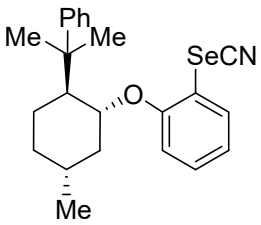
3.3.6 2-(((1*R*,2*S*,5*R*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)aniline (9c) [7]



1-(((1*S*,2*R*,5*S*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)-2-nitrobenzene (200 mg, 566 µmol, 1.00 equiv.) was dissolved in ethanol/acetic acid (9 mL, 1:1), treated with iron powder (95 mg, 1.70 mmol, 3.00 equiv.) and stirred for 3 h at 100 °C. After cooling to rt, the mixture was diluted with EtOAc (10 mL) and sat. aq. Na_2CO_3-sol. (10 mL) was added. The phases were separated and the organic phase was washed with sat. aq. $NaHCO_3$-sol. (3 x10 mL). The combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 20:1 → 15:1 pentane/EtOAc) provided the title product as a yellow oil (110 mg, 340 µmol, 61%).

TLC: R_f = 0.18 (15:1 Hex:EtOAc); **IR** (ATR): $\tilde{\nu}$ = 2951, 2922, 2867, 1611, 1501, 1457, 1278, 1213, 1008, 764, 735, 700 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$): δ (ppm) =7.37 – 7.20 (m, 4 H), 7.19 – 7.08 (m, 1 H), 6.74 – 6.55 (m, 4 H), 4.19 (td, ^3J = 10.4, 3.9 Hz, 1 H), 3.00 (sbr, 2 H), 2.27 – 2.00 (m, 2 H), 1.84 – 1.55 (m, 2 H), 1.37 (s, 4 H), 1.27 (s, 3 H), 1.12 (m, 1 H), 1.03 – 0.78 (m, 5 H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 152.1, 144.1, 137.1, 127.9, 125.5, 124.8, 120.2, 118.0, 115.2, 111.0, 76.8, 51.4, 40.1, 39.9, 35.0, 31.3, 28.1, 26.8, 25.7, 21.8; **HR-ESI-MS** (m/z) calc. for $C_{22}H_{30}ON$ $[M+H]^+$: 324.2322, found: 324.2322; **optical rotation:** α^{D}_{20}= –78° (0.92, DCM).

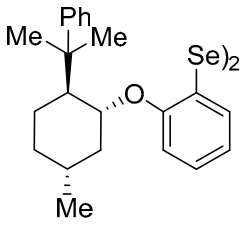
3.3.7 1-(((1*R*,2*S*,5*R*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)-2-selenocyanato-benzene (10c)



$BF_3{\cdot}Et_2O$ (564 µL, 638 mg, 5.06 mmol, 3.50 equiv.) was dissolved in dry THF (2.5 mL) under an argon atmosphere at -30 °C. A solution of 2-(((1*S*,2*R*,5*S*)-5-methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)aniline (**9c**) (468 mg, 1.45 mmol, 1.00 equiv.) in dry THF (10 mL) and *tert*-butylnitrite (688 µL, 597 mg, 5.79 mmol, 4.00 equiv.) were slowly added and the mixture was warmed to rt within 30 min and stirred for further 30 min at rt. The resulting solid was filtered off and washed with diethylether until it was completely white (ATTENTION: USE EXPLOSION SHIELD!). The filtrate was also treated with diethyl ether (40 mL) and the resulting solid was also filtered off and washed with diethyl ether. The combined solids were dried *in vacuo* and dissolved in dry acetonitrile (10 mL). The solution was cooled to –20 °C and a solution of potassium selenocyanate (418 mg, 2.90 mmol, 2.00 equiv.) in dry acetonitrile (5 mL) was slowly added. The mixture was slowly warmed to 0 °C (ice bath) and warmed to rt over 16 h. Then the mixture was diluted with DCM/water (20 mL, 1:1) and the phases were separated. The aqueous phase was extracted with diethyl ether (3 x 10 mL) and the combined organic phases were dried over Na_2SO_4. Removal of the solvent under reduced pressure provided the title product as orange-red oil (433 mg, 1.04 mmol, 72%). The crude product was used without further purification.

IR (ATR): $\tilde{\nu}$ = 2956, 2924, 2869, 2151, 1585, 1494, 1470, 1445, 1239, 1030, 993, 749, 700 cm-1; **H-NMR** (300 MHz, $CDCl_3$): δ (ppm) =7.62 (dd, ^3J = 7.9 Hz, ^4J = 1.5 Hz, 1 H), 7.43 – 7.09 (m, 6 H), 7.01 (td, ^3J = 7.6 Hz, ^4J = 1.2 Hz, 1 H), 6.82 (dd, ^3J = 8.4, ^4J = 1.2 Hz, 1 H), 4.28 (m, 1 H), 2.17 – 1.91 (m, 2 H), 1.77 – 0.78 (m, 17 H); **HR-ESI-MS** (m/z) calc. for: $C_{23}H_{27}ONSeNa$ $[M+Na]^+$: 436.1151, found: 436.1151.

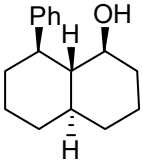
3.3.8 1,2-Bis(2-(((1*R*,2*S*,5*R*)-5-methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)phenyl) diselane (11c)[8]



1-(((1*S*,2*R*,5*S*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)-2-seleno-cyanatobenzene (420 mg, 1.01 mmol, 1.00 equiv.) was dissolved in ethanol (12 mL), treated with aq. NaOH-sol. (4.5 M, 161 µL, 725 µmol, 0.50 equiv.) and stirred for 1 h at rt. A mixture of DCM/water (20 mL, 1:1) was added, the phases were separated, and the aqueous phase was extracted with DCM (3 x 10 mL). The combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 20:1→5:1 pentane/DCM) provided the title product as yellow oil (214 mg, 277 µmol, 54%).

TLC: R_f = 0.18 (15:1 Hex:EtOAc); **IR** (ATR): $\tilde{\nu}$ = 2952, 2921, 2868, 1571, 1464, 1441, 1227, 1030, 996, 908, 746, 700, 409 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ (ppm) = 7.50 (dd, ^3J = 7.8 Hz, ^4J = 1.6 Hz, 1 H), 7.37 – 7.32 (m, 2 H), 7.26 (m, 2 H), 7.20 – 7.10 (m, 2 H), 6.83 (ddd, ^3J = 7.8, 7.3 Hz, ^4J = 1.1 Hz, 1 H), 6.72 (m, 1 H), 4.25 (td, ^3J = 10.4, 4.1 Hz, 1 H), 2.12 – 2.00 (m, 2 H), 1.61 – 1.29 (m, 9 H), 1.09 (td, ^3J = 12.5, 10.7 Hz, 1H), 1.00 (m, 1 H), 0.92 – 0.81 (m, 4 H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ (ppm) = 153.9, 150.1, 130.4, 127.9, 127.5, 126.1, 125.3, 121.5, 120.6, 111.8, 78.4, 51.5, 40.7, 40.4, 34.7, 31.4, 30.6, 27.3, 25.1, 21.8; **^{77}Se-NMR** (95 MHz, $CDCl_3$) δ (ppm) = 331.86. **HR-ESI-MS** (m/z) calc. for: $C_{44}H_{54}O_2Se_2Na$ $[M+Na]^+$: 797.2356 found: 797.2338. **optical rotation:** $\alpha^{D_{20}}$= –84 (0.60, $CHCl_3$).

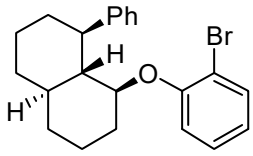
3.3.9 (1*S*,4a*R*,8*R*,8a*R*)-8-Phenyldecahydronaphthalen-1-ol [9]



The compound was synthesized according to a literature-known procedure. The spectra are in accordance with the literature.

TLC: R_f (30:1 Pent/EtOAc) = 0.13; **IR** (ATR) $\tilde{\nu}$ = 3591, 2921, 2853, 1714, 1493, 1449, 1048, 759, 701 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$) δ (ppm) =7.50 – 7.13 (m, 5 H), 3.49 (dddd, ^3J = 10.6, 9.1, 4.5 Hz, ^4J = 1.9 Hz, 1 H), 2.41 (ddd, ^3J = 11.9, 10.3, ^4J = 3.3 Hz, 1 H), 2.04 – 0.92 (m, 13 H); **^{13}C-NMR** (126 MHz,$CDCl_3$) δ (ppm) =146.8, 129.1, 127.4, 126.8, 75.6, 54.8, 50.5, 41.8, 37.1, 35.1, 33.9, 33.8, 26.5, 23.9; **HR-ESI-MS** m/z calc. for $C_{16}H_{22}ONa$ ($[M+Na]^+$): 253.1563, found: 253.1563; **optical rotation:** $\alpha^{D_{20}}$= 9.9° (c = 1.00, $CHCl_3$).

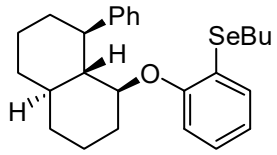
3.3.10 (1*S*,4a*R*,8*R*,8a*R*)-1-(2-Bromophenoxy)-8-phenyldecahydronaphthalene (16)[10]



(1*S*,4a*R*,8*R*,8a*R*)-8-Phenyldecahydronaphthalen-1-ol (275 mg, 1.19 mmol, 1.00 equiv.) and 1-bromo-2-fluorobenzene (189 mg, 1.08 mmol, 0.90 equiv.) were dissolved in dry DMF (3 mL) and potassium *tert*-butoxide (1 M in THF, 1.37 mL, 1.37 mmol, 1.15 equiv.) was added dropwise. The mixture was stirred for 16 h at 100 °C and another portion of 1-bromo-2-fluorobenzene (100 mg, 570 µmol, 0.48 equiv.) and potassium *tert*-butoxide (1 M in THF, 1.00 mL, 1.00 mmol, 0.90 equiv.) were added. The reaction was stirred 3 h at 100 °C and, after cooling to rt quenched with H_2O (5 mL). The mixture was extracted with Et_2O (3 x 10 mL), the combined org. phases were washed with water (2 x 10 mL) and brine (10 mL), dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, pentane → 4:1 pentane:DCM) provided the title product as yellow oil (261 mg, 677 µmol, 57%).

TLC: R_f (4:1 Pent/DCM) = 0.74; **IR** (ATR) $\tilde{\nu}$ = 2925, 2852, 1585, 1474, 1441, 1272, 1245, 1031, 744, 697 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$) δ (ppm) =7.23 (dd, ^3J = 7.8 Hz, ^4J = 1.7 Hz, 1 H), 7.12 – 6.99 (m, 3 H), 6.99 (t, ^3J = 7.4 Hz, 2H), 6.89 (m, 1 H), 6.65 (dd, ^3J = 8.4 Hz, ^4J = 1.3 Hz, 1 H), 6.60 (td, ^3J = 7.6 Hz, ^4J = 1.4 Hz, 1 H), 4.13 (td, ^3J = 9.6, 4.7 Hz, 1 H), 2.45 – 2.30 (m, 2 H), 1.99 – 1.13 (m, 15 H).; **^{13}C-NMR** (125 MHz,$CDCl_3$) δ (ppm) = 153.4, 146.9, 133.0, 127.6, 127.0, 125.0, 120.5, 113.6, 113.0, 80.5, 51.7, 50.8, 42.9, 37.3, 34.0, 32.0, 26.6, 23.6; **HR-ESI-MS** m/z calc. for $C_{22}H_{25}OBrNa$ ([M+Na]$^+$): 407.0981, found: 407.0980; **optical rotation:** α^{D}_{20}= –41.9° (c = 1.04, $CHCl_3$).

3.3.11 Butyl(2-(((1*S*,4a*R*,8*R*,8a*R*)-8-phenyldecahydronaphthalen-1-yl)oxy)phenyl)selane (11d)

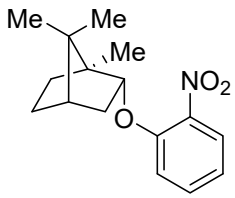


(1*S*,4a*R*,8*R*,8a*R*)-1-(2-bromophenoxy)-8-phenyldecahydronaphthalene (**16**) (260 mg, 677 µmol, 1.00 equiv.) was dissolved in dry Et_2O (12 mL) and *n*-butyllithium (2.5 M in hexane, 298 µL, 745 mmol, 1.10 equiv.) was added dropwise. The mixture was stirred for 1 h at 45°C and selenium (160 mg, 2.03 mmol, 3.00 equiv.) was added. The mixture was stirred for another 16 h at 45 °C and quenched with NH_4Cl (10 mL). The mixture was extracted with DCM (3 x 20 mL), the combined org. phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 20:1 Pent/DCM) followed by gel-permeation chromatography ($CHCl_3$) provided the title product as yellow oil (48.5 mg, 110 µmol, 16%).

TLC: R_f = 0.21 (pentane:DCM) ; **IR** (ATR) $\tilde{\nu}$ = 2922, 2852, 1574, 1467, 1440, 1268, 1233, 1123, 1036, 1012, 965, 753, 697 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ (ppm) = 7.11 – 7.01 (m, 3 H), 7.05 – 6.93 (m, 3 H), 6.86 (tt, ^3J = 7.4 Hz, ^4J = 1.2 Hz, 1 H), 6.69 (td, ^3J = 7.4 Hz, ^4J = 1.2 Hz, 1 H), 6.60

(dt, ^3J = 8.0 Hz, ^4J = 1.0 Hz, 1 H), 4.09 (td, ^3J = 9.7, 4.5 Hz, 1 H), 2.67 – 2.56 (m, 2 H), 2.41 (ddd, ^3J = 12.0, 10.3, ^4J = 3.5 Hz, 1 H), 1.92 – 1.11 (m, 18 H), 0.90 (t, ^3J = 7.4 Hz, 3 H); **^{13}C-NMR** (126 MHz,$CDCl_3$) δ (ppm) = 155.3, 147.2, 130.0, 127.6, 127.0, 126.0, 124.8, 122.6, 120.5, 113.1, 81.2, 53.4, 51.6, 50.6, 42.3, 37.4, 33.9, 33.5, 32.1, 31.6, 26.5, 24.7, 23.5, 23.1, 13.6; **^{77}Se-NMR** (95 MHz, $CDCl_3$) δ (ppm) = 232.35; **HR-ESI-MS** m/z calc. for $C_{26}H_{35}OSe$ ($[M+H]^+$): 443.1849, found: 443.1854; **optical rotation:** α^{D}_{20}= –65.8° (c = 0.96, $CHCl_3$).

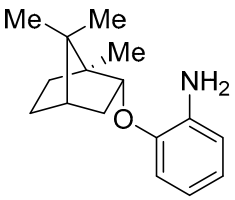
3.3.12 (1*S*)-1,7,7-Trimethyl-2-(2-nitrophenoxy)bicyclo[2.2.1]heptane[6]



Sodium hydride (60 w% in mineral oil, 2.00 g, 13.0 mmol, 1.25 equiv.) was suspended in dry THF (32 mL) under an argon atmosphere at 0 °C, treated with 2-fluoronitrobenzene (1.46 mg, 10.37 mmol, 1.00 equiv.). A solution of (–)-borneol (2.00 g, 13.0 mmol, 1.25 Äq equiv.) in dry THF (12 mL) was slowly added and the mixture was allowed to warm to rt and stirred for 16 h at 60 °C. After cooling to rt sat. aq. NH_4Cl-sol. (30 mL) was added, the aqueous phase was extracted with DCM (3 x 50 mL), the combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 15:1 pentane/EtOAc) provided the title product as an orange solid (2.36 g, 8.57 mmol, 83%).

TLC: R_f = 0.41 (30:1 pentane:EtOAc); **T_m**: 68 °C; **IR** (ATR) $\tilde{\nu}$ = 2953, 1606, 1523, 1482, 1351, 1274, 1164, 1021, 867, 840, 743 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$) δ (ppm) =7.82 (dd, ^3J = 8.1 Hz, ^4J = 1.7 Hz, 1 H), 7.47 (ddd, ^3J = 8.3, 7.4 Hz, ^4J = 1.7 Hz, 1 H), 6.99 – 6.89 (m, 2 H), 4.43 (ddd, ^3J = 9.3, 3.3 Hz, ^4J = 1.7 Hz, 1 H), 2.40 (ddt, ^3J = 13.3, 9.2, 3.8 Hz, 1 H), 2.27 (m, 1 H), 1.87 – 1.70 (m, 2 H), 1.46 – 1.21 (m, 2 H), 1.16 (dd, ^3J = 13.3, 3.4 Hz, 1 H), 0.94 (s, 6 H), 0.93 (s, 3 H); **^{13}C-NMR** (76 MHz, $CDCl_3$) δ (ppm) = 152.5, 140.1, 133.9, 125.6, 119.5, 115.4, 85.1, 49.8, 47.6, 45.1, 36.6, 27.8, 26.8, 19.6, 18.9, 13.6; **HR-ESI-MS** m/z calc. for $C_{16}H_{21}NO_3Na$ ($[M+Na]^+$): 298.1414, found: 298.1418; **optical rotation** α^{D}_{20}= –136 ° (c = 0.997, $CHCl_3$).

3.3.13 2-(((1*S*,2*R*,4*S*)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl)oxy)-aniline (9a) [7]

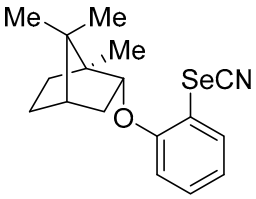


(1*S*)-1,7,7-Trimethyl-2-(2-nitrophenoxy)bicyclo[2.2.1]heptane (2.00 g, 7.26 mmol, 1.00 equiv.) was dissolved in ethanol/acetic acid (140 mL, 1:1), treated with iron powder (1.22 g, 21.8 mmol, 3.00 equiv.) and stirred for 3 h at 100 °C. After cooling to rt the mixture was diluted with EtOAc (10 mL) and brought to pH=10 by the addition of aq. NaOH-sol. (1 M). The phases were separated and the organic phase was washed with sat. aq. $NaHCO_3$-sol. (3 x100 mL). The combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 30:1 pentane:EtOAc) provided the

title product as a red solid (1.33 g, 5.42 mmol, 75%). (1*S*)-1,7,7-Trimethyl-2-(2-nitrophenoxy)bicyclo[2.2.1]heptane (222 mg, 806 μmol, 11%) could be reisolated.

TLC: R_f = 0.34 (15:1 Hex:EtOAc); **T_m**:66 °C; **IR** (ATR) $\tilde{\nu}$ = 2951, 1612, 1504, 1457, 1273, 1216, 1114, 1053, 735 cm^{-1}; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ (ppm) = 6.81 – 6.63 (m, 4H), 4.34 (ddd, $^3J_{HH}$ = 9.2, 3.3 Hz, $^4J_{HH}$ = 1.9 Hz, 1H), 3.80 (s_{br}, 2H), 2.39 (dddd, $^3J_{HH}$ = 13.6, 9.2, 4.7, 3.3 Hz, 1H), 2.27 – 2.17 (m, 1H), 1.85 – 1.72 (m, 2H), 1.45 – 1.34 (m, 1H), 1.29 (m, 1H), 1.17 (dd, $^3J_{HH}$ = 13.4, 3.4 Hz, 1H), 0.95 (s, 3H), 0.95 (s, 3H), 0.92 (s, 3H); **^{13}C-NMR** (101 MHz,$CDCl_3$) δ (ppm) = 146.5, 136.6, 120.6, 118.4, 115.0, 112.5, 83.1, 49.6, 47.6, 45.2, 37.0, 28.0, 27.1, 19.7, 18.9, 13.9; **HR-ESI-MS** m/z calc. for: $C_{16}H_{24}NO$ ($[M+H]^+$): 246.1852,found: 246.1860; **optical rotation** $\alpha^{D_{20}}$= –117 ° (c = 1.00, $CHCl_3$, 3mm).

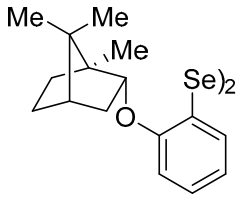
3.3.14 (1*S*)-1,7,7-Trimethyl-2-(2-selenocyanatophenoxy)bicyclo[2.2.1]-heptane (10a)



$BF_3{\cdot}OEt_2$ (796 µL, 899 mg, 7.14 mmol, 3.50 equiv.) was dissolved in dry THF (15 mL) under argon atmosphere at -30 °C. A solution of 2-(((1*S*,2*R*,4*S*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)oxy)-aniline (**9a**) (500 mg, 2.04 mmol, 1.00 equiv.) in dry THF (15 mL) and *tert*-butyl nitrite (841 mg, 8.16 mmol, 4.00 equiv.) were slowly added and the mixture was slowly warmed to rt within 30 min and stirred for further 30 min at rt. The resulting solid was filtered off and washed with diethyl ether until it was completely white (ATTENTION: USE EXPLOSION SHIELD!). The filtrate was treated with pentane (15 mL) and the resulting solid was filtered off. The combined solids were dried *in vacuo* and then dissolved in dry acetonitrile (10 mL). The solution was cooled to –20 °C and potassium selenocyanate (293 mg, 2.04 mmol, 1.00 equiv.) in dry acetonitrile (5 mL) was slowly added. The mixture was slowly warmed to 0 °C (ice-bath) and warmed to rt over 16 h. Then the mixture was diluted with DCM/water (20 mL, 1:1) and the phases were separated. The aqueous phase was extracted with DCM (2 x 20 mL) and the combined organic phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 4:1 Pent/DCM) provided the title product as an brown oil (409 mg, 1.22 mmol, 60%).

TLC: R_f (4:1 Pent:DCM) = 0.15; **IR** (ATR) $\tilde{\nu}$ = 2953, 1574, 1472, 1446, 1305, 1278, 1245, 1054, 1022, 993, 746 cm^{-1}; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ (ppm) = 7.63 (dd, ^3J = 7.9 Hz, ^4J = 1.5 Hz, 1 H), 7.29 (ddd, ^3J = 8.2, 7.5 Hz, ^4J = 1.5 Hz, 1 H), 7.00 (ddd, ^3J = 7.9, 7.5 Hz, ^4J = 1.2 Hz, 1 H), 6.76 (dd, ^3J = 8.2 Hz, ^4J = 1.2 Hz, 1 H), 4.41 (ddd, ^3J = 9.3, 3.3 Hz, ^4J = 1.9 Hz, 1 H), 2.39 (dddd, ^3J = 13.7, 9.2, 4.6, 3.3 Hz, 1 H), 2.10 (ddd, ^3J = 13.4, 9.3, 3.9 Hz, 1 H), 1.91 – 1.71 (m, 2 H), 1.42 (m, 1 H), 1.27 (m, 1 H), 1.13 (dd, ^3J = 13.5, 3.3 Hz, 1 H), 0.96 (s, 3 H), 0.94 (s, 3 H), 0.93 (s, 3 H); **^{13}C-NMR** (101 MHz,$CDCl_3$) δ (ppm) = 154.9, 129.7, 129.6, 122.4, 113.7, 112.8, 101.6, 84.9, 49.8, 47.7, 45.1, 36.7, 27.8, 27.0, 19.6, 18.9, 13.8; **^{77}Se-NMR** (76 MHz, $CDCl_3$) δ (ppm) = 281.0; **HR-ESI-MS** m/z calc. for: $C_{17}H_{21}NOSeNa$ ($[M+Na]^+$): 358.0681, found: 358.0688.

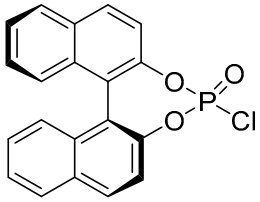
3.3.15 1,2-Bis(2-(((1*S*,2*R*,4*S*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)oxy)phenyl)diselenide (11a)[8]



(1*S*)-1,7,7-Trimethyl-2-(2-selenocyanatophenoxy)bicyclo[2.2.1]-heptane (**10a**) (370 mg, 1.11 mmol, 1.00 equiv.) was dissolved in ethanol (12 mL), treated with aq. NaOH-sol. (2.5 M in water, 221 µL, 553 µmol, 0.50 equiv.) and stirred for 1 h at rt. Filtration yielded the title product as a yellow solid (270 mg, 438 µmol, 79%).

T_m: 162 °C; **IR** (ATR) $\tilde{\nu}$ = 2951, 2876, 1572, 1466, 1442, 1390, 1364, 1304, 1271, 1238, 1054, 1022, 744 cm^{-1}; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ (ppm) =7.53 (dd, ^3J = 7.5 Hz, ^4J = 1.6 Hz, 1 H), 7.15 (ddd, ^3J = 8.1, 7.5 Hz, ^4J = 1.6 Hz, 1 H), 6.84 (td, ^3J = 7.5 Hz, ^4J = 1.2 Hz, 1 H), 6.68 (dd, ^3J = 8.1 Hz, ^4J = 1.2 Hz, 1 H), 4.44 (ddd, ^3J = 9.3, 3.2 Hz, ^4J = 1.6 Hz, 1 H), 2.40 (dddd, ^3J = 13.5, 9.9, 4.8, 2.5 Hz, 2 H), 1.92 – 1.71 (m, 2 H), 1.50 – 1.30 (m, 2 H), 1.22 (dd, ^3J = 13.3, 3.3 Hz, 1 H), 1.03 (s, 3 H), 0.97 (s, 3 H), 0.95 (s, 3 H); **^{13}C-NMR** (101 MHz,$CDCl_3$) δ (ppm) =156.0, 130.0, 130.0, 127.7, 127.6, 121.5, 121.5, 121.4, 119.8, 112.1, 111.9, 84.2, 84.1, 49.9, 47.6, 45.3, 45.2, 37.0, 36.9, 36.8, 27.9, 27.9, 27.8, 27.2, 27.1, 27.1, 19.7, 19.7, 19.0, 19.0, 19.0, 13.9, 13.9, 13.9, 13.8; **^{77}Se-NMR** (76 MHz, $CDCl_3$) δ (ppm) = 281; **HR-ESI-MS** m/z calc. for $C_{32}H_{42}O_2Se_2Na$ ($[M+Na]^+$): 657.1153, found.: 657.1150; **optical rotation** $\alpha^{D_{20}}$ = –91° (c = 1.005%, $CHCl_3$, 3mm).

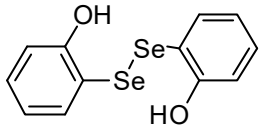
3.3.16 (*R*)-4-Chlorodinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepine 4-oxide (18)[11]



(*R*)-BINOL (500 mg, 1.75 mmol, 1.00 equiv.) and triethyl amine (975 µL, 707 mg, 6.98 mmol, 4.00 equiv.) were dissolved in dry toluene (10 mL), cooled to 0 °C and $POCl_3$ (175 µL, 294 mg, 1.92 mmol, 1.10 equiv.) was added slowly. The mixture was stirred for 16 h at 0 °C and the solvent was removed under reduced pressure. Column chromatography (SiO_2, DCM) provided the title compound as colorless solid (484 mg, 1.32 mmol, 75%).

TLC: R_f = 0.60 (DCM); **T_m:** 188 °C; **IR** (ATR) $\tilde{\nu}$ = 2956, 2923, 2853, 1591, 1508, 1463, 1227, 1029, 963, 815, 748, 597, 483, 400 cm^{-1}; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ (ppm) = 8.13 – 8.04 (m, 2 H), 8.04 – 7.95 (m, 2 H), 7.63 (dd, ^3J = 8.9, 1.1 Hz, 1 H), 7.59 – 7.48 (m, 3 H), 7.45 – 7.29 (m, 4 H); **^{13}C-NMR** (101 MHz,$CDCl_3$) δ (ppm) =146.6 (d, $^3J_{CP}$ = 12.7 Hz), 146.3 (d, $^3J_{CP}$ = 11.3 Hz), 132.2 (d, $^5J_{CP}$ = 1.9 Hz), 132.1 , 132.0 (d, $^5J_{CP}$ = 1.8 Hz), 131.9 (d, $^5J_{CP}$ = 1.5 Hz), 131.6 (d, $^5J_{CP}$ = 1.6 Hz), 128.8 – 128.4 (m), 127.3 – 127.0 (m), 126.3 – 126.3 (m), 121.6 (d, $^4J_{CP}$ = 3.0 Hz), 121.5 (d, $^4J_{CP}$ = 2.5 Hz), 120.3 (d, $^4J_{CP}$ = 2.8 Hz), 119.9 (d, $^4J_{CP}$ = 3.8 Hz); **^{31}P-NMR** (162 MHz, $CDCl_3$) δ (ppm) = 10.9; **HR-ESI-MS** m/z calc. for: $C_{20}H_{13}O_3PCl$ ($[M+H]^+$): 367.0285, found: 367.0277; the results are in accordance with literature.

3.3.17 2,2'-Diphenol diselenide (19)[12]

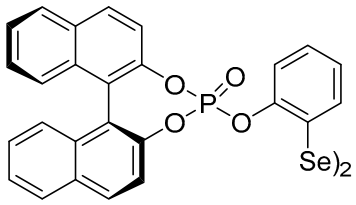


n-Butyllithium (2.5 M in hexan, 21 mL, 52.0 mmol, 1.50 equiv.) was added to dry hexane at -78 °C (20 mL) and TMEDA (5.2 mL, 4.03 g, 34.7 mmol, 2.00 equiv.) was added slowly. 2-Bromophenol (2.01 mL, 3.00 g, 17.3 mmol 0.50 equiv.) was then added to the cloudy solution at -78 °C and the mixture was stirred for further 2 h at rt. Selenium (1.38 g, 17.3 mmol, 0.50 equiv.) was added at 0 °C and the mixture was stirred for further 16 h at rt. Aq. HCl (1 M, 10 mL), water (30 mL) and EtOAc (20 mL) were added. The phases were separated, aq. HCl (5 M, 10 mL) was added to the aqueous phase, and it was extracted with EtOAc (3 x 20 mL). The combined org.

phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 15:1→2:1 Pent:EtOAc) provided the title compound as a red solid (893 mg, 2.58 mmol, 30%) as an inseperatable mixture with 10 mol% 2-bromophenol.

TLC: R_f =0.15 (5:1 Hex:EtOAc); **IR** (ATR) $\tilde{\nu}$ = 3424, 1574, 1463, 1443, 1334, 1287, 1236, 1180, 1022, 826, 750, 472, 446 cm^{-1}; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ (ppm) = 7.37 (dd, ^3J = 7.7 Hz, ^4J = 1.7 Hz, 2 H), 7.32 (ddd, ^3J = 8.2, 7.3 Hz, ^4J = 1.7 Hz, 2 H), 7.01 (dd, ^3J = 8.2 Hz, ^4J = 1.4 Hz, 2 H), 6.79 (td, ^3J = 7.5 Hz, ^4J = 1.4 Hz, 2 H), 6.11 (s_{br}, 2 H); **^{13}C-NMR** (101 MHz,$CDCl_3$) δ (ppm) = 156.7, 137.5, 133.0, 121.1, 115.2; **^{77}Se-NMR** (76 MHz, $CDCl_3$) δ (ppm) = 377; **HR-ESI-MS** m/z calc. for $C_{12}H_{10}O_4Se_2Na$ ([M+Na]$^+$): 368.8906, found: 368.8900.

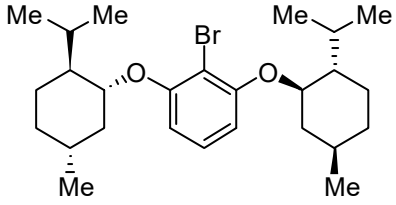
3.3.18 (*R*)-4,4'-((Diseleniddiylbis(2,1-phenylene))bis(oxy))bis(dinaphtho-[2,1-d:1',2'-f][1,3,2]dioxaphosphepine 4-oxid) (20)



(*R*)-4-Chlorodinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepine 4-oxide (**18**) (100 mg, 273 µmol, 2.00 equiv.), 2,2'-diphenoldiselenid (**19**) (50 mg, 138 µmol, 1.00 equiv.) and triethyl amine (152 µL, 110 mg, 1.09 mmol, 4.00 equiv.) were dissolved in dry DCM (5 mL). The mixture was stirred for 16 h at rt and sat. aq. NH_4Cl-Lsg. (5 mL) was added. The aqueous phase was extracted with DCM (3 x 15 mL) the combined organic phases were dried over Na_2SO_4 and column chromatography (SiO_2, DCM) provided the title compound as yellow oil (37 mg, 28.4 µmol, 21%).

TLC: R_f (DCM) =0.70; **IR** (ATR) $\tilde{\nu}$ = 1508, 1312, 1200, 1187, 1156, 967, 951, 899, 815, 750 cm^{-1}; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ (ppm) =8.07 (d, $^3J_{HH}$ = 8.8 Hz, 2 H), 8.03 – 7.89 (m, 6 H), 7.64 (m, 2 H), 7.57 – 7.27 (m, 18 H), 7.23 – 7.13 (m, 2 H), 6.96 (t, $^3J_{HH}$ = 7.6 Hz, 2 H); **^{13}C-NMR** (101 MHz,$CDCl_3$) δ (ppm) =148.3 (d, $^3J_{CP}$ = 6.0 Hz), 147.3 (d, $^2J_{CP}$ = 11.6 Hz), 146.0 (d, $^3J_{CP}$ = 8.6 Hz), 132.4 , 132.2 (d, $^5J_{CP}$ = 1.0 Hz), 132.2 (d, $^5J_{CP}$ = 1.1 Hz), 132.0 (d, $^5J_{CP}$ = 1.4 Hz), 131.8 (d, $^5J_{CP}$ = 1.1 Hz), 129.0 – 128.9 (m), 128.5 (d, $^4J_{CP}$ = 4.3 Hz), 127.1 (d, $^2J_{CP}$ = 9.1 Hz), 126.9 (d, $^4J_{CP}$ = 2.2 Hz), 126.6, 126.0 (d, $^3J_{CP}$ = 6.4 Hz), 121.5 (d, $^3J_{CP}$ = 6.3 Hz), 121.1 (d, $^4J_{CP}$ = 2.2 Hz), 120.5 (d, $^4J_{CP}$ = 3.0 Hz), 120.2 (d, $^4J_{CP}$ = 3.4 Hz), 119.3 (d, $^4J_{CP}$ = 2.0 Hz); **^{31}P-NMR** (203 MHz, $CDCl_3$) δ (ppm) = -3.75; **^{77}Se-NMR** (95 MHz, $CDCl_3$) δ (ppm) = 364; **HR-ESI-MS** m/z calc. for $C_{52}H_{32}O_8P_2Se_2Na$ ([M+Na]$^+$): 1028.9809, found: 1028.9782. **optical rotation:** α^{D}_{20}= –45.5 (c = 1.44, $CHCl_3$).

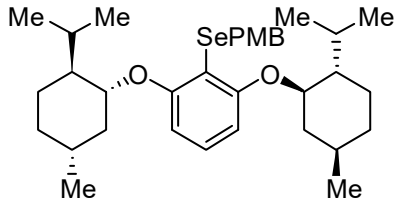
3.3.19 (1*S*,1'*S*,2*R*,2'*R*,4*R*,4'*R*)-2,2'-((2-Bromo-1,3-phenylene)bis(oxy))bis(1-isopropyl-4-methylcyclohexane)(13a)[10]



(–)-Menthol (810 mg, 5.18 mmol, 2.00 equiv.) and 1-bromo-2,6-difluorobenzene (189 mg, 1.08 mmol, 0.90 equiv.) were dissolved in dry DMF (10 mL) and sodium hydride (60 w% in mineral oil, 248 mg, 6.22 mmol, 2.40 equiv.) was added to the solution. The mixture was stirred for 19 h at 100 °C and quenched by the addition of aq. sat. NH_4Cl-sol. The mixture was extracted with EtOAc (3 x 20 mL), the combined org. phases were washed with water (2 x 10 mL) and brine (10 mL), dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, pentane → 143:1 pentane/DCM) provided the title product as a colorless solid (467 mg, 1.12 mmol, 43%).

TLC: R_f (DCM) =0.20 (pentane/DCM, 143:1); **T_m**: 104 °C; **IR** (ATR) $\tilde{\nu}$ =2954, 2929, 2669, 1582, 1459, 1367,1331, 1249, 1272, 1250, 1183, 1100, 1054, 1035, 981, 946, 923, 878, 844, 756, 703, 664 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$) δ (ppm) =7.12 (t, ^3J = 8.3 Hz, 1 H), 6.51 (d, ^3J = 8.4 Hz, 2 H), 4.07 (td, ^3J = 10.5, 4.1 Hz, 2 H), 2.31 (heptd, ^3J = 6.9, 2.7 Hz, 2 H), 2.18, 2.09 (m, 2 H), 1.80, 1.58 (m, 6 H), 1.45 (ddtd, ^3J = 19.3, 9.7, 6.5 Hz, ^4J = 3.3 Hz, 2 H), 1.18, 0.99 (m, 6 H), 0.93 (t, ^3J = 6.9 Hz, 12 H), 0.76 (d, ^3J = 7.0 Hz, 6 H); **^{13}C-NMR** (101 MHz,$CDCl_3$) δ (ppm) =156.3, 127.7, 106.3, 104.3, 79.1, 48.1, 40.5, 34.7, 31.7, 26.2, 23.9, 22.4, 21.1, 16.9; **HR-ESI-MS** m/z calc. for $C_{26}H_{42}O_2Br$ ([M+H]$^+$): 465.2363, found: 465.2365; **optical rotation:** α^{D}_{20} = –111° (1.00, $CHCl_3$).

3.3.20 Bis-2,6-bis(((1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl)oxy)benzene diselenide (14a)

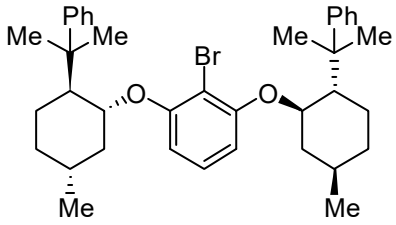


(1*S*,1'*S*,2*R*,2'*R*,4*R*,4'*R*)-2,2'-((2-Bromo-1,3-phenylene)bis(oxy))bis-(1-isopropyl-4-methylcyclohexane) (**13a**) (467 mg, 0.32 mmol, 1.00 equiv.) was dissolved in dry diethyl ether (1.1 mL) and cooled to -78°. *t*-Butyllithium (1.9 M in pentane, 425 µL, 820 µmol, 2.52 equiv.) was slowly added and the mixture was stirred for 1 h at 0 °C. A solution of PMBSeCN (98 mg, 430 µmol, 1.33 equiv.) in THF (2 mL) was added to the solution and the mixture was stirred for further 15 min. Then the reaction is quenched by the addition of sat. aq. NH_4Cl-sol. (4 mL) and extracted with EtOAc (2 x 5 mL). The organic phase was washed with brine (3 mL), dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, pentane → 50:1 pentane/EtOAc) provided the title product as yellow oil (66 mg, 0.126 mmol, 35%).

TLC: R_f (50:1 Pent/Et_2O) = 0.38; **IR** (ATR) $\tilde{\nu}$ =2952, 2923, 2867, 1609, 1578, 1509, 1453, 1369, 1299, 1246, 1231, 1173, 1098, 1068, 1053, 829, 764, 741, 712 cm^{-1}; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ (ppm) = 7.19 - 7.09 (m, 3 H), 6.78 - 6.73 (m, 2 H), 6.49 (d, ^3J = 8.4 Hz, 2 H), 4.16 (d, ^3J = 10.9 Hz, 1 H), 4.11-4.01 (m, 3 H), 3.76 (s,3 H), 2.32 (dqd, ^3J = 13.7, 6.9, 3.5 Hz, 2 H), 2.16-2.03 (m, 2 H), 1.72 (ddt, ^3J = 11.1, 8.3, 3.9 Hz, 4 H), 1.65 - 1.56 (m, 2 H), 1.43 (dddd, ^3J = 15.3, 12.3, 6.3 Hz, ^4J = 3.2 Hz, 2 H), 1.17 - 0.96 (m, 6 H), 0.96 - 0.88 (m, 12 H), 0.75 (d, ^3J = 6.9Hz, 6 H); **^{13}C-NMR**

(101 MHz, $CDCl_3$) δ (ppm) =159.4, 158.3, 132.1, 130.1, 128.8, 113.8, 105.5, 78.3, 77.2, 55.3, 48.1, 40.4, 34.7, 31.6, 29.9, 26.1, 23.6, 22.3, 21.1, 16.6; **^{77}Se-NMR** (76 MHz, $CDCl_3$): δ (ppm) = 231.8; **HR-ESI-MS** m/z calc. for $C_{52}H_{32}O_8P_2Se_2Na$ ([M+Na]$^+$): 587.3001, found: 587.2983; **optical rotation:** α^{D}_{20} = 40° (c = 0.37, $CHCl_3$).

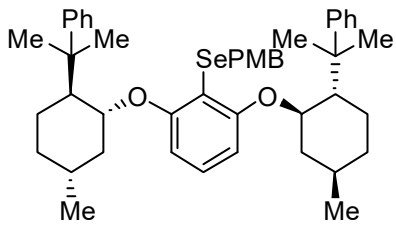
3.3.21 (((1*S*,1'*S*,2*R*,2'*R*,4*R*,4'*R*)-((2-Bromo-1,3-phenylene)bis(oxy))bis(4-methylcyclohexane-2,1-diyl))bis(propane-2,2-diyl))dibenzene (13b)[10]



(–)-8-Phenylmenthol (1.18 g, 5.01 mmol, 2.56 equiv.) and 1-bromo-2,6-difluorobenzene (376 mg, 1.96 mmol, 1.00 equiv.) were dissolved in dry DMF (7.5 mL) and potassium *tert*-butoxide (1 M in THF, 5 mL, 5.00 mmol, 2.30 equiv.) was added dropwise to the solution. The mixture was stirred for 16 h at 100 °C. After cooling to rt quenched with H_2O (5 mL), the mixture was extracted with Et_2O (3 x 20 mL), the combined org. phases were washed with water (2 x 10 mL) and brine (10 mL), dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, pentane → 4:1 pentane:DCM) provided the title product as colorless solid (618 mg, 1.00 mmol, 51%). The mono-substituted product was also isolated (137 mg, 338 µmol, 17%).

TLC: R_f (4:1 Pent/DCM) = 0.71; **T_m**: 209°C; **IR** (ATR) $\tilde{\nu}$ = 2951, 2923, 2869, 1586, 1461, 1251, 1092, 1064, 1034, 907, 760, 734, 700 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$) δ (ppm) = 7.37 – 7.21 (m, 8H), 7.19 – 7.05 (m, 3H), 6.39 (d, J = 8.4 Hz, 2H), 4.18 (td, J = 10.3, 4.1 Hz, 2H), 2.19 – 1.94 (m, 4H), 1.52 (d, J = 10.4 Hz, 8H), 1.42 (s, 10H), 1.22 – 0.74 (m, 10H); **^{13}C-NMR** (126 MHz,$CDCl_3$) δ (ppm) = 155.0, 150.0, 127.7, 127.4, 126.2, 125.1, 105.2, 103.9, 78.4, 51.5, 40.8, 40.5, 34.8, 31.5, 31.4, 27.5, 24.5, 21.9; **HR-ESI-MS** m/z calc. for $C_{38}H_{50}O_2Br$ ([M+H]$^+$): 617.2989, found: 617.2986; **optical rotation:** α^{D}_{20} = –54° (c = 0.09, $CHCl_3$)

3.3.22 (2,6-Bis(((1*R*,2*S*,5*R*)-5-methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)phenyl)(4-methoxybenzyl)selane (14b)

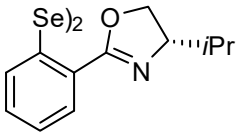


(((1*S*,1'*S*,2*R*,2'*R*,4*R*,4'*R*)-((2-Bromo-1,3-phenylene)bis(oxy))bis(4-methylcyclohexane-2,1-diyl))bis(propane-2,2-diyl))dibenzene (**13b**) (100 mg, 162 µmol, 1.00 equiv.) is dissolved in dry diethyl ether (2 mL) and *n*-butyllithium (2.5 M in hexane, 71 µL, 178 µmol, 1.10 equiv.) was added at rt. The mixture was stirred for 1 h at 45 °C and a solution of PMBSeCN (68 mg, 243 µmol, 1.50 equiv.) in dry diethyl ether (1.5 mL) was added to the solution. The mixture was stirred for 16 h at 40 °C. The reaction was quenched by the addition of sat. aq. NH_4Cl, the mixture was extracted with DCM

(3 x 10 mL), the combined org. phases were dried over Na_2SO_4 and the solvent was removed under reduced pressure. Column chromatography (SiO_2, 4:1 pentane/DCM) provided the title product as a yellow oil (27 mg, 36.0 µmol, 22%).

TLC: R_f = 0.37 (4:1 Pent:DCM); **IR** (ATR) $\tilde{\nu}$ = 2953, 2923, 2869, 2369, 2359, 2342, 1579,1510, 1453, 1246, 1226, 1092, 1061, 1036, 801, 763, 700 cm^{-1}; **^{1}H-NMR** (500 MHz, $CDCl_3$) δ (ppm) =7.37 – 7.31 (m, 4 H), 7.31 – 7.24 (m, 4 H), 7.24 – 7.20 (m, 2 H), 7.19 – 7.11 (m, 3 H), 6.80 (d, ^3J = 8.6 Hz, 2 H), 6.43 (d, ^3J = 8.4 Hz, 2 H), 4.25 (d, ^3J = 10.8 Hz, 1 H), 4.17 (td, ^3J = 10.3, 4.0 Hz, 2 H), 4.00 (d, ^3J = 10.7 Hz, 1 H), 3.77 (s, 3 H), 2.11 – 1.98 (m, 4 H), 1.62 – 1.19 (m, 24 H), 1.13 – 0.74 (m, 16 H); **^{13}C-NMR** (126 MHz,$CDCl_3$) δ (ppm) =158.2, 150.1, 131.9, 130.0, 128.7, 127.8, 126.3, 125.2, 113.7, 111.5, 105.2, 78.4, 55.2, 51.6, 40.8, 40.5, 34.7, 31.5, 31.4, 30.2, 27.5, 24.4, 21.8, 1.0; **HR-ESI-MS** m/z calc. for $C_{42}H_{59}O_2Se$ ($[M+H]^+$): 675.3679, found: 675.3671; **optical rotation:** $\alpha^{D_{20}}$ = –34° (c = 0.53, $CHCl_3$).

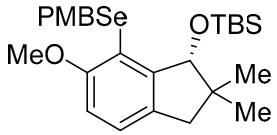
3.3.23 1,2-Bis(2-((*S*)-4-isopropyl-4,5-dihydrooxazol-2-yl)phenyl)diselane(4)[13]



The compound was synthesized according to literature. The spectroscopic data are in accordance with literature.

IR (ATR) $\tilde{\nu}$ = 2956, 2929, 2872, 1643, 1463, 1354, 1247, 1019, 967, 732 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$) δ (ppm) =7.83 (m, 4 H), 7.24 (m, 4 H), 4.48 (dd, ^2J = 8.7 Hz, ^3J = 7.7 Hz, 2 H), 1.86 (hept, ^3J = 7.7 Hz, 2 H), 4.22 (m, 4 H), 1.12 (m, 6 H), 1.03 (m, 6 H); **HR-ESI-MS** m/z calc. for $C_{24}H_{29}O_2N_2Se_2$ ($[M+H]^+$): 537.0558, found: 537.0543.

3.3.24 (*R*)-*tert*-Butyl((6-methoxy-7-((4-methoxybenzyl)selanyl)-2,2-dimethyl-2,3-dihydro-1H-inden-1-yl)oxy)dimethylsilane (23)[14]

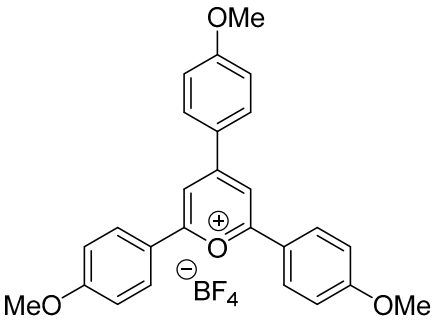


The compound was synthesized according to the literature: spectoscopic data are in accordance with literature

TLC: R_f (1:1 DCM:Pent)= 0.65; **IR** (ATR) $\tilde{\nu}$ = 2953, 2928, 2855, 1609, 1510, 1460, 1434, 1247, 1173, 1063, 1039, 834, 774 cm^{-1}; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ (ppm) = 7.06 (d, ^3J = 8.2 Hz, 1 H), 6.96 (d, ^3J = 8.6 Hz, 1 H), 6.73 (d, ^3J = 8.1 Hz, 1 H), 6.66 (d, ^3J = 8.6 Hz, 2 H), 4.40 (s, 1 H), 4.09 (d, ^3J = 11.5 Hz, 1 H), 3.90 (s, 3 H), 3.86 (d, ^3J = 11.6 Hz, 1 H), 3.71 (s, 3 H), 2.95 (d, ^3J = 14.3 Hz, 1 H), 2.27 (d, ^3J = 14.7 Hz, 1 H), 1.13 (s, 3 H), 0.80 (s, 9 H), 0.50 (s, 3 H), 0.10 (s, 3 H), -0.04 (s, 3 H); **^{13}C-NMR** (101 MHz,$CDCl_3$) δ (ppm) = 158.2, 158.1, 152.1, 136.8, 131.9, 129.6, 125.7, 115.4, 113.6, 110.2, 85.2, 56.3, 55.2, 44.8, 44.7, 30.3, 26.2, 26.1, 23.5, 18.6, -3.2, -3.5; **^{77}Se-NMR** (76 MHz, $CDCl_3$) δ (ppm) = 269.61; **HR-ESI-MS** m/z calc for $[C_{26}H_{38}O_3SeSiNa]^+$ $[M+Na]^+$:529.1649, found: 529.1648. **optical rotation** $\alpha^{D_{20}}$ = 155° (c = 1.01, $CHCl_3$).

3.4 Photocatalysts

3.4.1 2,4,6-Tris(4-methoxyphenyl)pyrylium tetrafluoroborate (TAPT)[15]

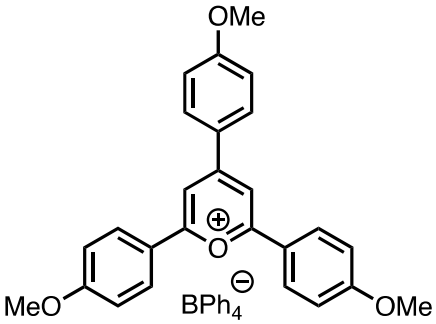


This compound was synthesized according to literature:

Spectra are in accordance to literature.

IR (ATR) $\tilde{\nu}$ = 2941, 2841, 1585, 1482, 1457, 1434, 1258, 1235, 1174, 1016, 829, 562, 518 cm^{-1}; **^{1}H-NMR** (300 MHz, DMSO-D$_6$) δ (ppm) = 8.54 (s, 2 H), 8.43 (d, *J* = 9.1 Hz, 2 H), 8.29 (d, *J* = 9.0 Hz, 4 H), 7.04-7.21 (m, 6 H), 3.94 (s, 3 H), 3.91 (s, 6 H); **^{13}C-NMR** (101 MHz, DMSO-D$_6$) δ (ppm) = 167.4, 165.2, 164.4, 161.5, 132.2, 130.4, 124.2, 121.0, 115.2, 115.1, 110.3, 55.9, 55.8; **HR-ESI-MS** m/z calc for $[C_{26}H_{23}O_4]^+$ [M]$^+$: 399.1591, found.: 399.1587.

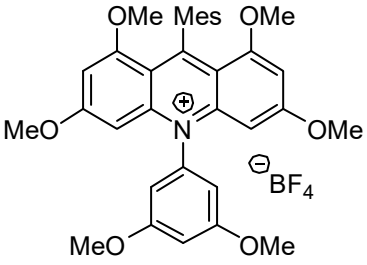
3.4.2 2,4,6-Tris(4-methoxyphenyl)pyrylium tetraphenylborate (TAPTP)



2,4,6-Tris(4-methoxyphenyl)pyrylium tetraphenylborate (300 mg, 617 µmol, 1.00 equiv.) was dissolved in dry diethyl ether (10 mL), potassium tetraphenylborate was added (321 mg, 617 µmol, 1.00 equiv.) and the mixture was stirred for 16 h at rt. THF (10 mL) was added and the suspension was filtered. The filtrate was collected and evaporation of the solvent provided the title compound as red solid (391 mg, 544 µmol, 88%).

T$_m$= 85.2 °C; **IR** (ATR) $\tilde{\nu}$ = 1584, 1569, 1509, 1478, 1457, 1436, 1304, 1257, 1240, 1171, 1121, 1018, 830, 732, 703 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$) δ (ppm) = 7.76 (d, ^3J = 8.8 Hz, 4 H), 7.58 – 7.44 (m, 12 H), 6.96 (t, ^3J = 7.4 Hz, 12 H), 6.82 (q, ^3J = 8.2, 7.2 Hz, 6 H), 3.87 (s, 6 H), 3.85 (s, 3 H); **^{13}C-NMR** (75 MHz, $CDCl_3$) δ (ppm) = 168.0, 165.9, 165.3, 164.6, 163.9, 163.3, 162.4, 136.2, 131.6, 130.2, 128.8, 127.2, 125.7, 125.7, 125.6, 125.6, 124.1, 121.7, 120.6, 115.9, 115.8, 110.3, 56.2, 56.1; **^{11}B-NMR** (96 MHz, $CDCl_3$) δ = -6.42; **HR-ESI-MS** m/z Cation: calc for $[C_{26}H_{23}O_4]^+$ [M]$^+$: 399.1591, found.: 399.1589, Anion: calc for $[C_{24}H_{20}B]^-$ [M]$^-$: 319.1700, found.: 318.1693.

3.4.3 10-(3,5-Dimethoxyphenyl)-9-mesityl-1,3,6,8-tetramethoxyacridin-10-ium tetrafluoroborate (DMTA)[16]

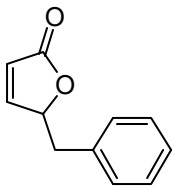


This compound was synthesized according to literature:

Spectra are in accordance to literature.

IR (ATR) $\tilde{\nu}$ = 3030, 2968, 2937, 2878, 2251, 1655, 1461, 1417, 1287, 1072, 969, 907, 865, 793, 730 cm^{-1}; **^{1}H-NMR** (300 MHz, $CDCl_3$) δ (ppm) =6.90 (dd, ^{3}J = 1.3, 0.7 Hz, 2 H), 6.83 (t, ^{3}J = 2.2 Hz, 1 H), 6.60 (d, ^{3}J = 2.2 Hz, 2 H), 6.50 (dd, ^{3}J = 2.3, 0.5 Hz, 2 H), 6.18 (d, ^{3}J = 2.3 Hz, 2 H), 3.92 (s, 6 H), 3.85 (s, 6 H), 3.53 – 3.45 (m, 6 H), 2.37 (s, 3 H), 1.83 (t, ^{4}J = 0.6 Hz, 6 H); **^{13}C-NMR** (101 MHz,$CDCl_3$) δ (ppm) = 168.3, 163.2, 162.3, 160.7, 144.7, 139.8, 137.5, 136.4, 132.0, 127.0, 113.3, 105.6, 102.8, 97.5, 92.8, 57.1, 56.5, 56.2, 21.1, 20.2; **HR-ESI-MS** m/z calc for $[C_{34}H_{36}O_6]^+$ $[M]^+$: 554.2537, found.: 554.2538.

3.5 Asymmetric lactonization



(*E*)-5-Phenylpent-3-enoic acid (1.00 equiv.), the photocatalyst (0.05 equiv.) and the selenium catalyst (0.05 equiv. for diselenides, 0.10 equiv. for monoselenides) were dissolved in acetonitrile (0.1 M). The mixture was stirred vigorously at rt and irradiated with blue light (465 nm, 4500 lx). The solvent was removed under reduced pressure and column chromatography (SiO_2, 1:2 pentane/DCM) provided the title product as light yellow oil.

Table 2: Conditions used in the asymmetric aerobic lactonization.

entry	Se-catalyst	photocatalyst	solvent	*T*	*t*	yield	*ee*
1	**11b**	TAPT	MeCN	35°C	16h	70%	19%
2	**11c**	TAPT	MeCN	35°C	16h	68%	49%
3	**11c**	TAPT	acetone	35°C	16h	10%	nd
4	**11c**	TAPT	DCE	35°C	16h	61%	25%
5	**11c**	TAPT	MeCN	0°C	20h	65%	47%
6	**11d**	TAPT	MeCN	20°C	16h	40%	55%
7	**11d**	TAPT	DCE	20°C	19 h	38%	50%
8	**11a**	TAPT	MeCN	35°C	16h	81%	5%
9	**20**	TAPT	MeCN	35°C	16h	78%	10%
10	**14b**	TAPT	MeCN	35°C	20h	24%	48%
11[a]	**14b**	TAPT	MeCN	35°C	40h	59%	33%
12	**14b**	-	PhMe	35°C	16h	n.d.	37%
13	**14b**	DMTA	PhMe	35°C	16h	33%	8%
14	**14b**	TAPT	MeCN	50°C	96h	99%	24%
15	**14b**	TAPT	MeCN	35°C	6h	23%	15%

16	**14b**	TAPT	MeCN	0°C	16h	21%	20%
17	**14b**	Rhodamin G	MeCN	35°C	16h	0%	nd
18	**14b**	Rhodamin G	MeCN	35°C	16h	0%	nd
19	**14b**	$Ru(bpz)_3PF_6$	MeCN	45°C	16h	19%	4%
20	**14b**	TAPTP	MeCN	35°C	18h	10%	12%
21	**14b**	TAPTP	MeCN	35°C	17h	13%	12%
22	**14b**	TAPT	MeCN dry	35°C	16h	35%	16%
23	**14b**	TAPT	MeCN/H_2O 10:1	35°C	16h	0%	nd
24	**14a**	TAPT	MeCN	35°C	16h	40%	55%
25	**4**	TAPT	MeCN	35°C	16h	0%	nd
26	**23**	TAPT	MeCN	0°C	48h	10%	65%
27	**23**	TAPT	MeCN	0°C	88h	44%	67%
28	**23**	DMTA	PhMe	35°C	16h	0%	nd
30	**23**	$NO[BF_4]$	DCM	25°C	21h	0%	nd
31	**23**	$NO[BF_4]$	DCM	25°C	21h	12%	0%
32	**7a**	TAPT	MeCN	35°C	16h	44%	22%
33	**7c**	TAPT	MeCN	35°C	16h	11%	0%

[a]Instead of the respective aryl-PMB-selenide **14b** its butyl-substituted analogue was used.

R_f(Pent:Et_2O)= 0.21; **IR** (ATR): $\tilde{\nu}$ = 3030, 1748, 1602, 1496, 1455, 1337, 1160, 1099, 1023, 924, 900, 812, 748, 701 cm^{-1}; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ = 7.40 (dd, ^3J = 5.7 Hz, ^4J = 1.5 Hz, 1 H), 7.37 – 7.24 (m, 3 H), 7.24 – 7.19 (m, 1 H), 6.08 (dd, ^3J = 5.7 Hz, ^4J = 2.0 Hz, 1 H), 5.27 – 5.20 (m, 1 H), 3.16 (dd, ^3J = 13.9, 6.4 Hz, 1 H), 2.96 (dd, ^3J = 13.9, 7.1 Hz, 1 H); **^{13}C-NMR** (101 MHz, $CDCl_3$): δ = 172.7, 155.5, 134.8, 129.4, 128.7, 127.3, 122.1, 83.4, 39.6; **HR-ESI-MS** (m/z) calculated for $C_{11}H_{11}O_2$ $[M+H]^+$: 175.0754 found: 175.0755; **HPLC:** Daicel OD, 0.9 mL/min, 99:1 Hex:*i*PrOH R_T = 49.160 min, 51.825 min; Daicel ID, 1.0 mL/min, 90.1:9.9 Hex: *i*-PrOH R_T= 16.557 min, 17.644 min; Daicel ID, 0.8 mL/min, 90.1:9.9 Hex: *i*-PrOH *RT*= 23.469 min, 24.491 min.

4 Literature

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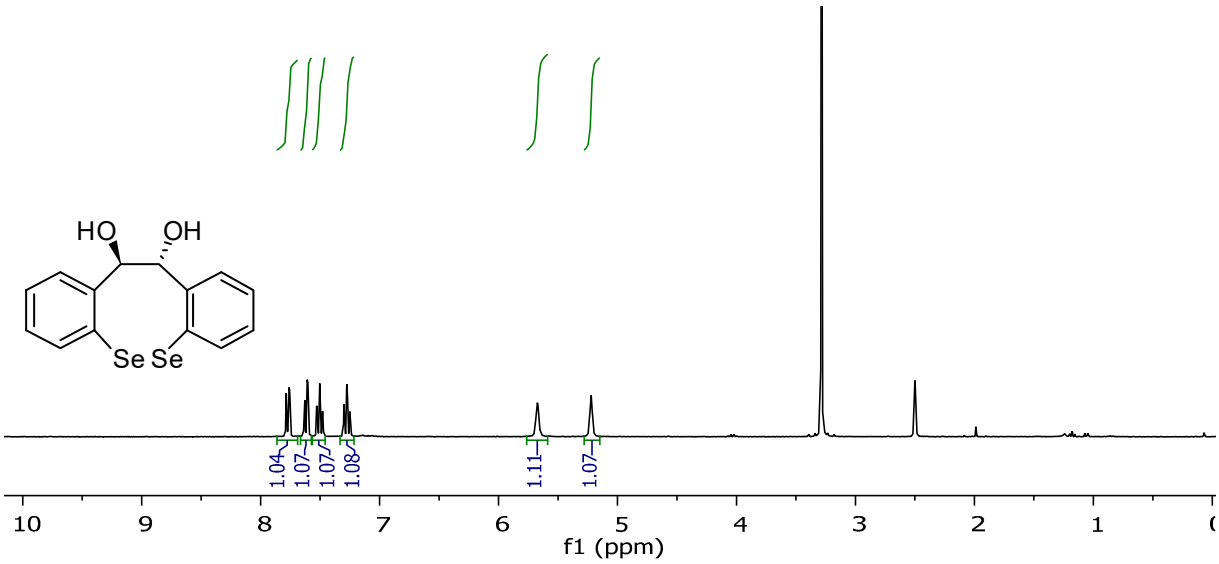
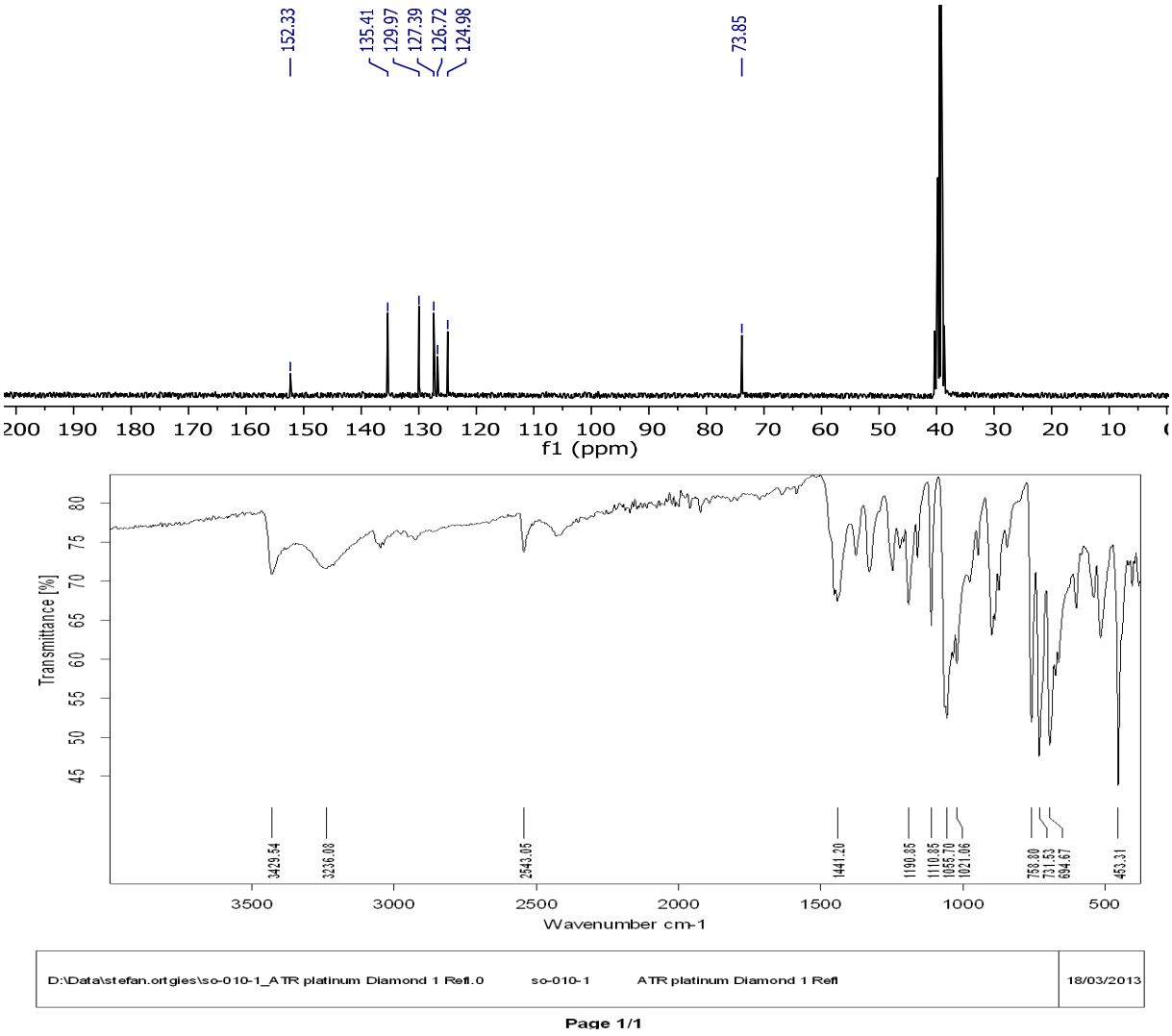
5 NMR Spectra

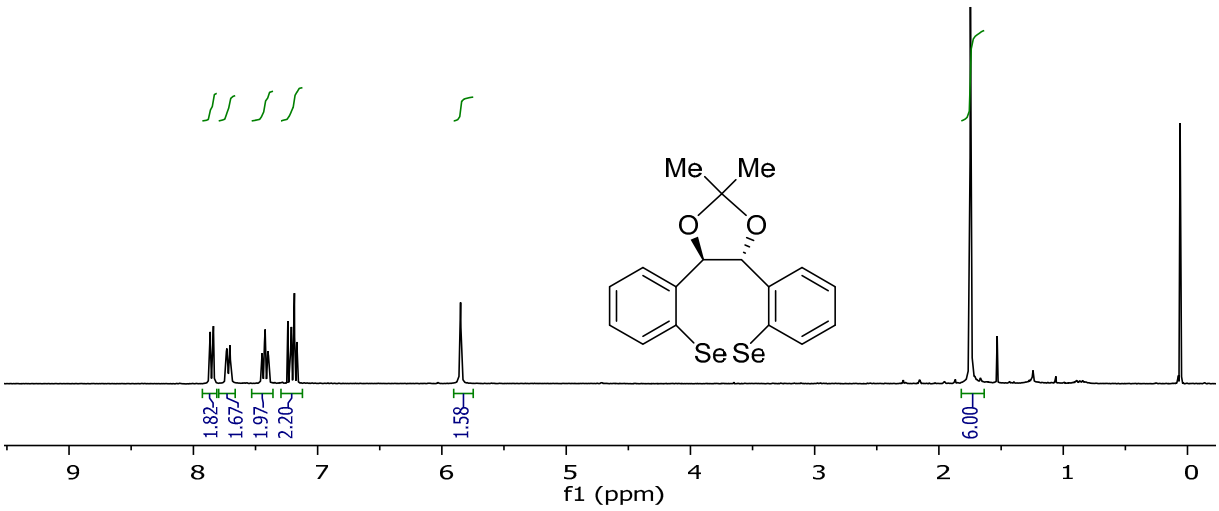
5.1 Diselenocine catalysts

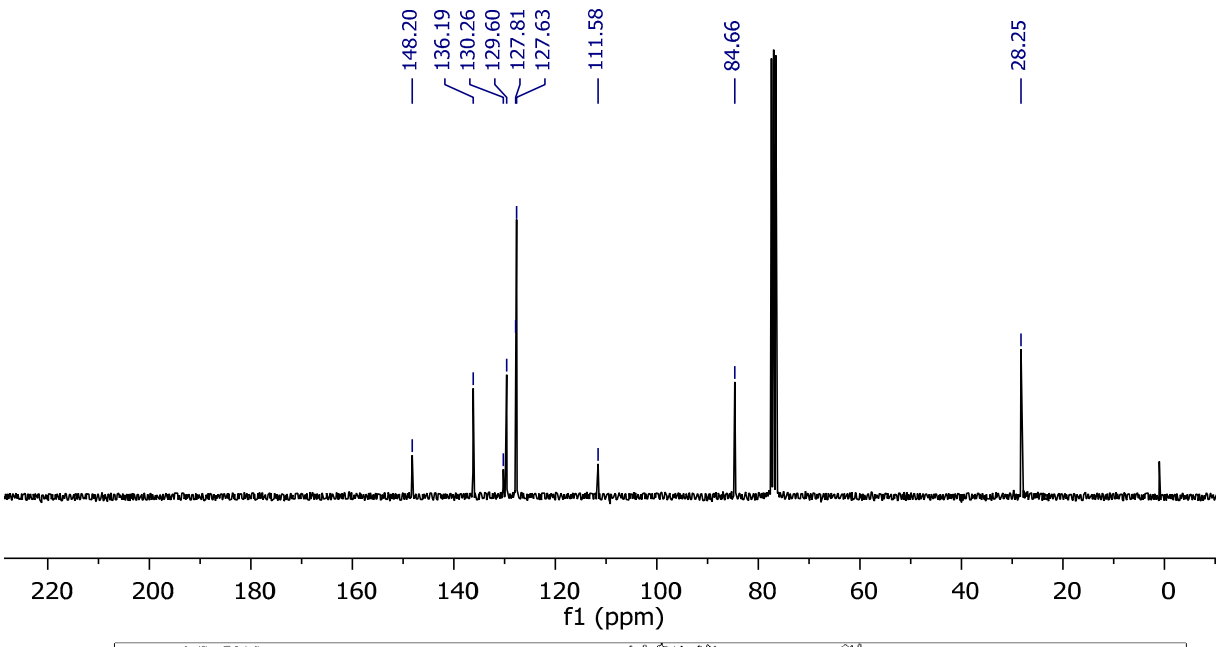
5.1.1 (11*R*,12*R*)-11,12-Dihydrodibenzo[c,g][1,2]diselenocin-11,12-diol (6)

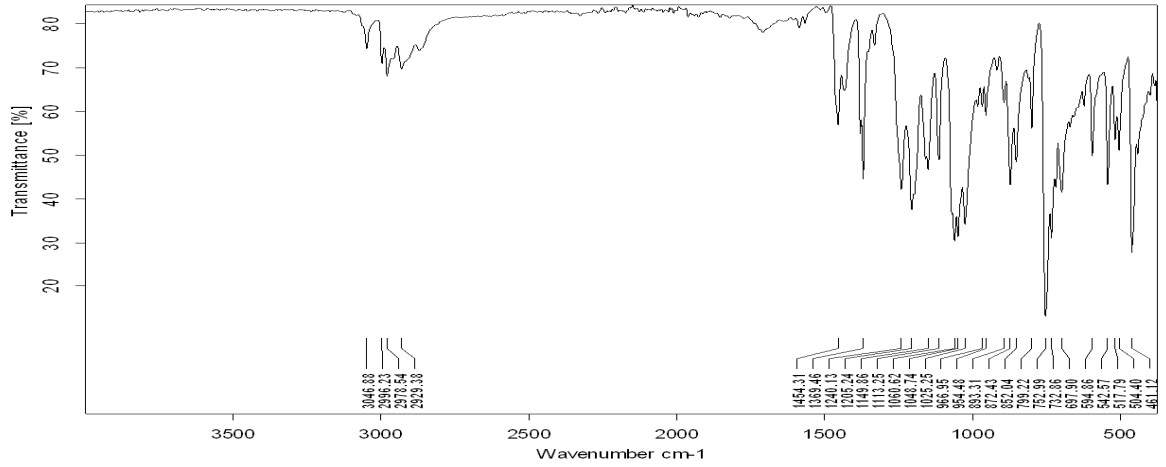




5.1.2 (3a*R*,13b*R*)-2,2-Dimethyl-3a,13b-dihydrodibenzo[3,4:7,8][1,2]diselenocino-[5,6-d][1,3]dioxol (7a)







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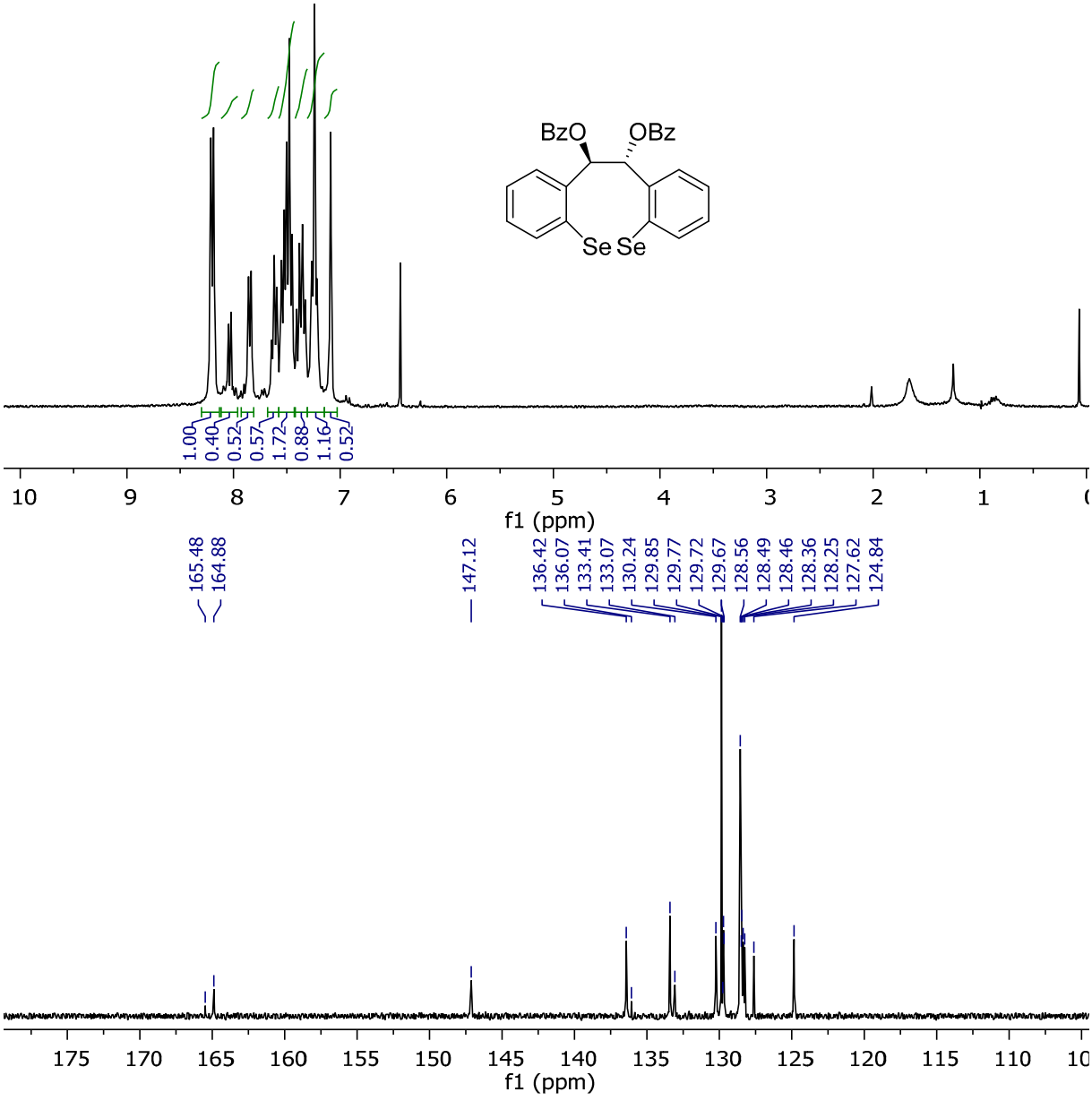
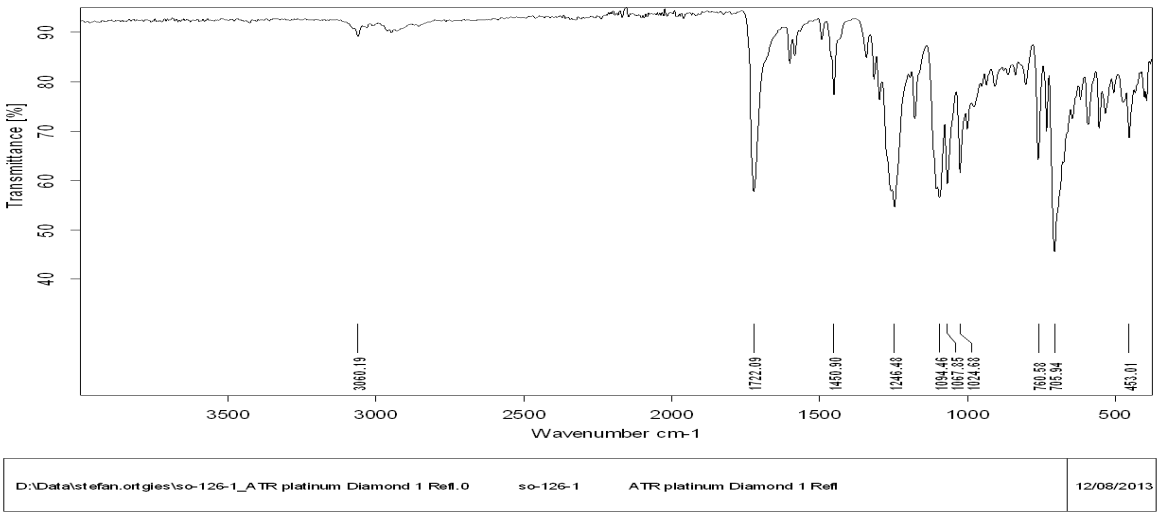
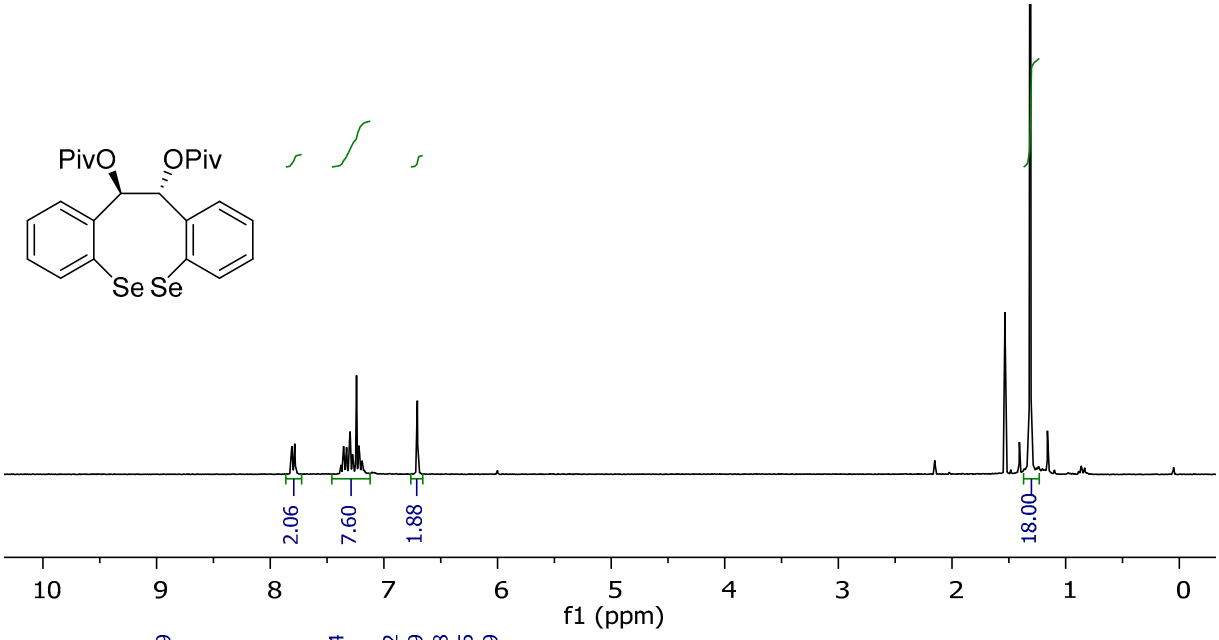
5.1.3 (11*R*,12*R*)-11,12-Dihydrodibenzo[c,g][1,2]diselenocin-11,12-diyldibenzoat (7d)

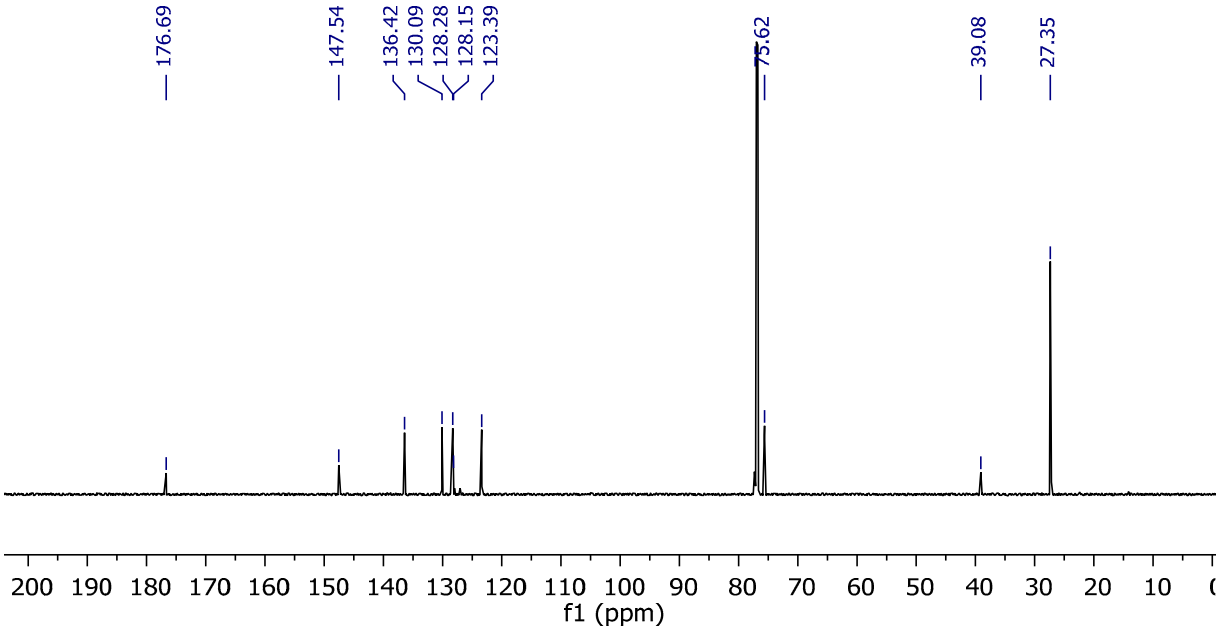


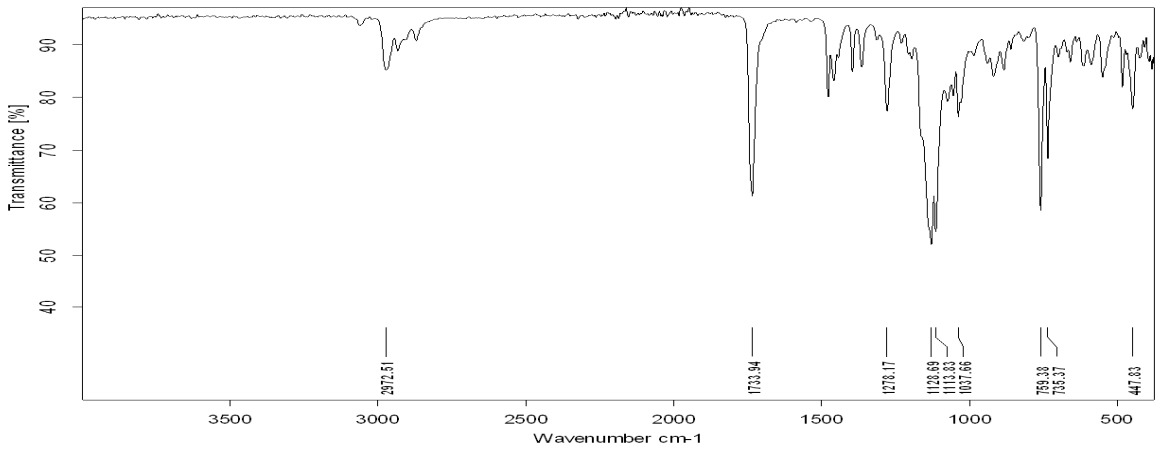
c



5.1.4 (11*R*,12*R*)-11,12-Dihydrodibenzo[c,g][1,2]diselenocin-11,12-diylbis(2,2- dimethyl-propanoat) (7b)







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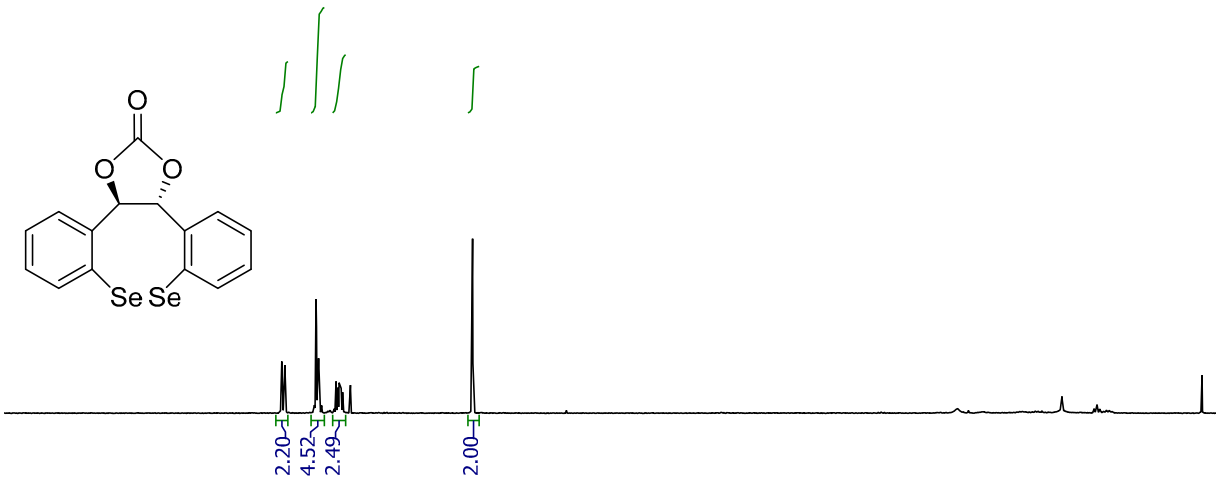
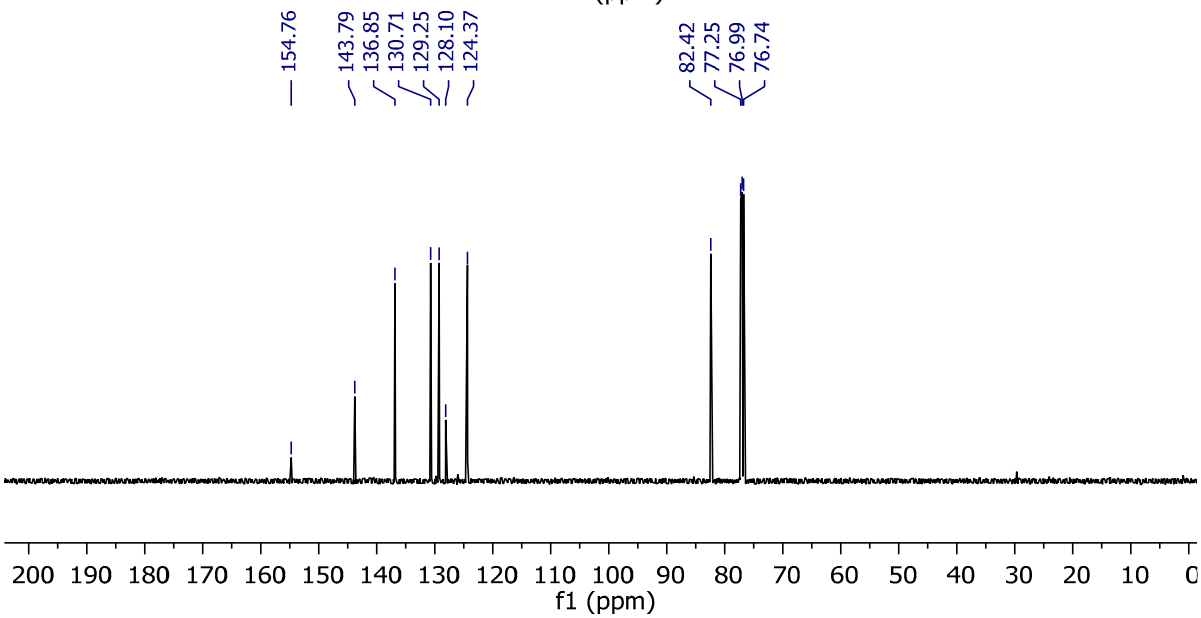
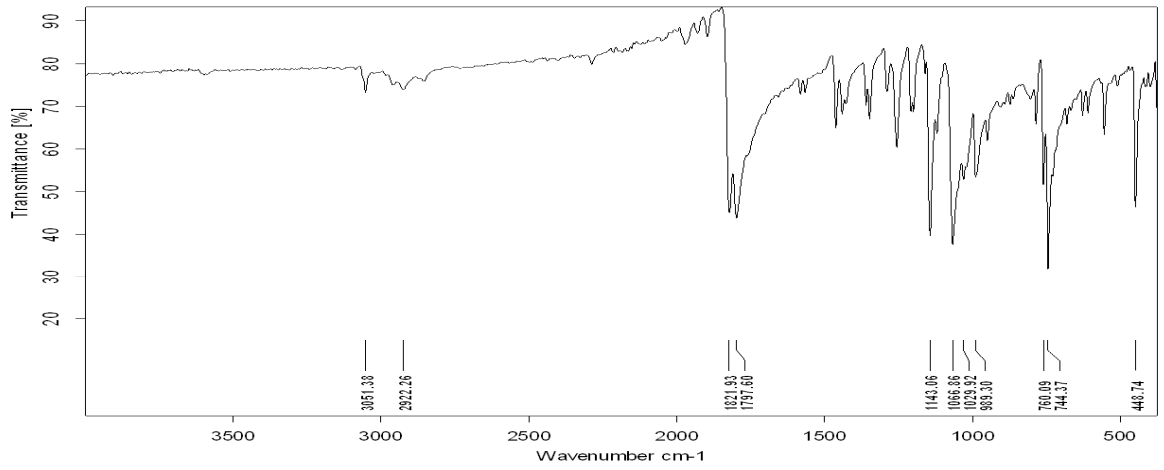
5.1.5 (3a*R*,13b*R*)-3a,13b-Dihydrodibenzo[3,4:7,8][1,2]diselenocino[5,6-d][1,3]dioxol-2-on (7c)







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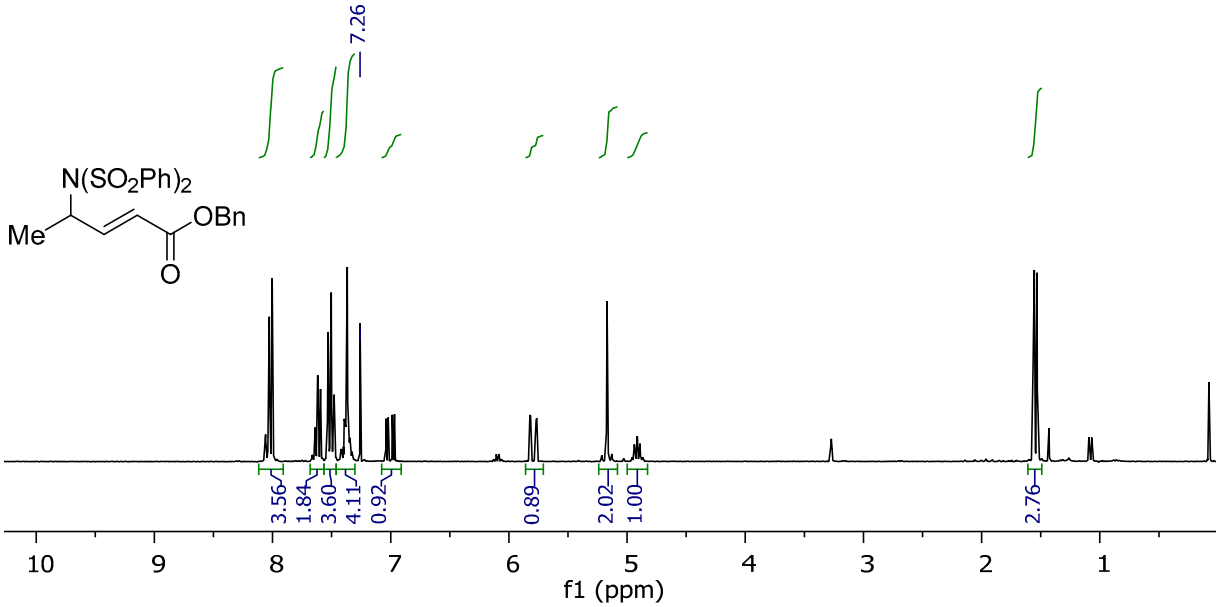
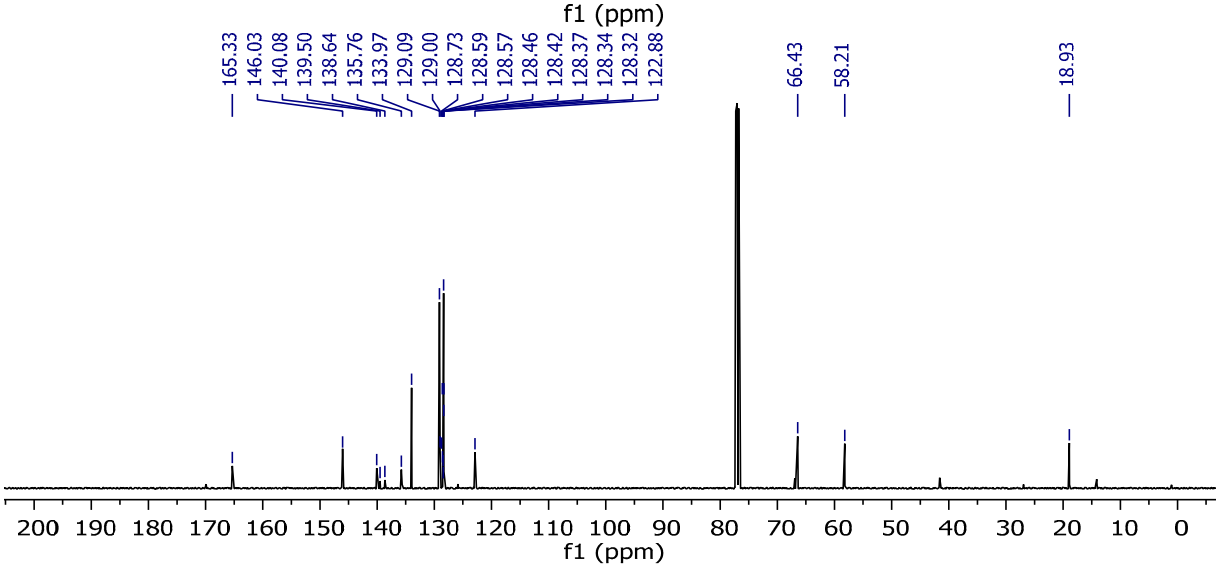
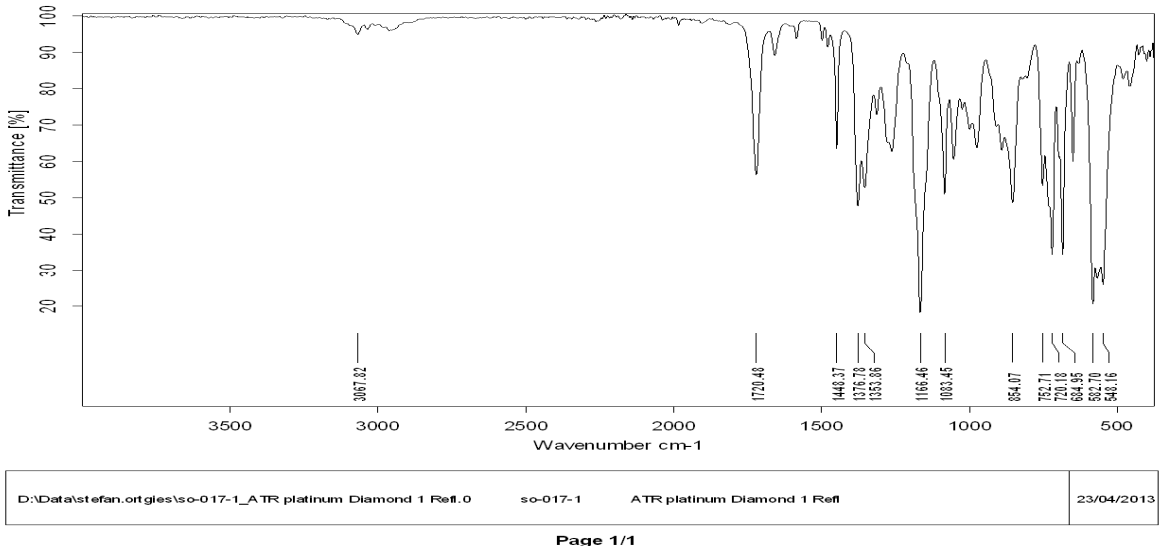
5.2 Imidation

5.2.1 (*E*)-Benzyl-4-(*N*-(phenylsulfonyl)phenylsulfonamid)pent-2-enoat (3)







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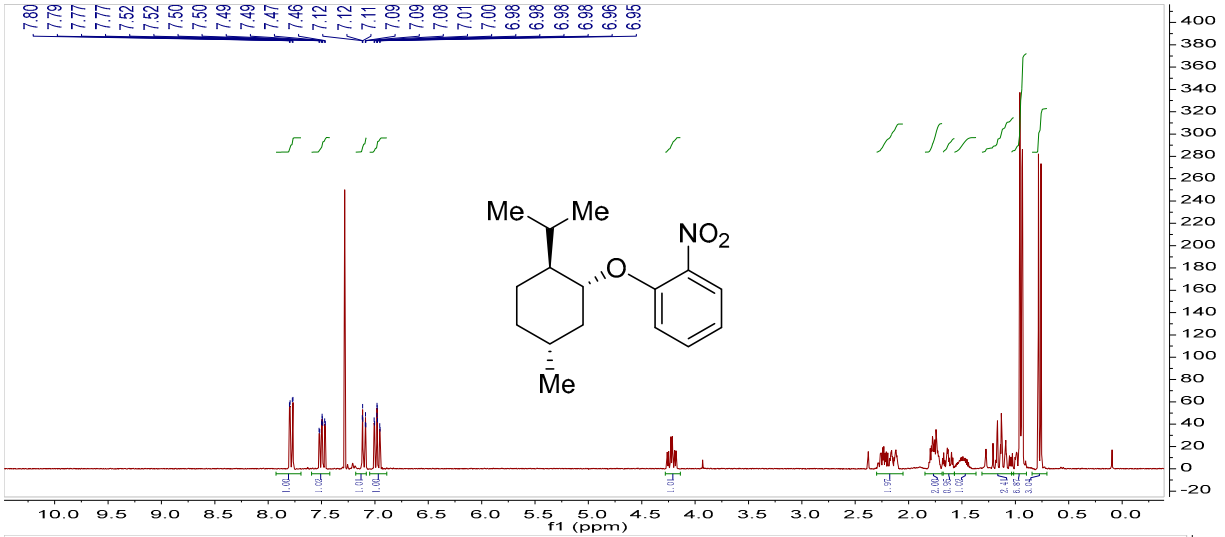
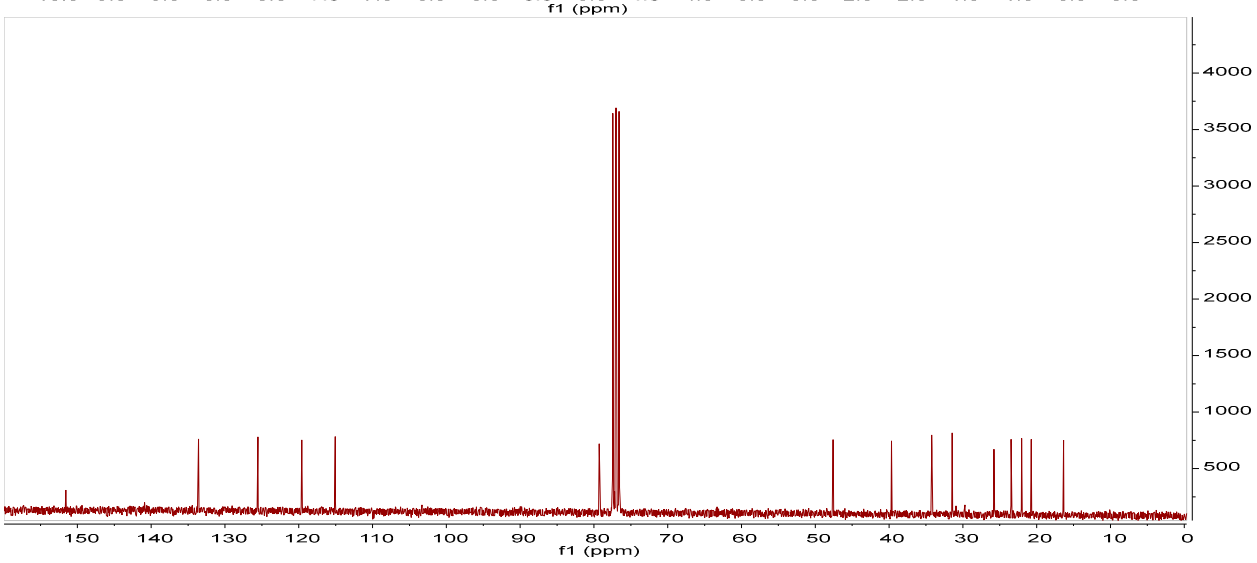
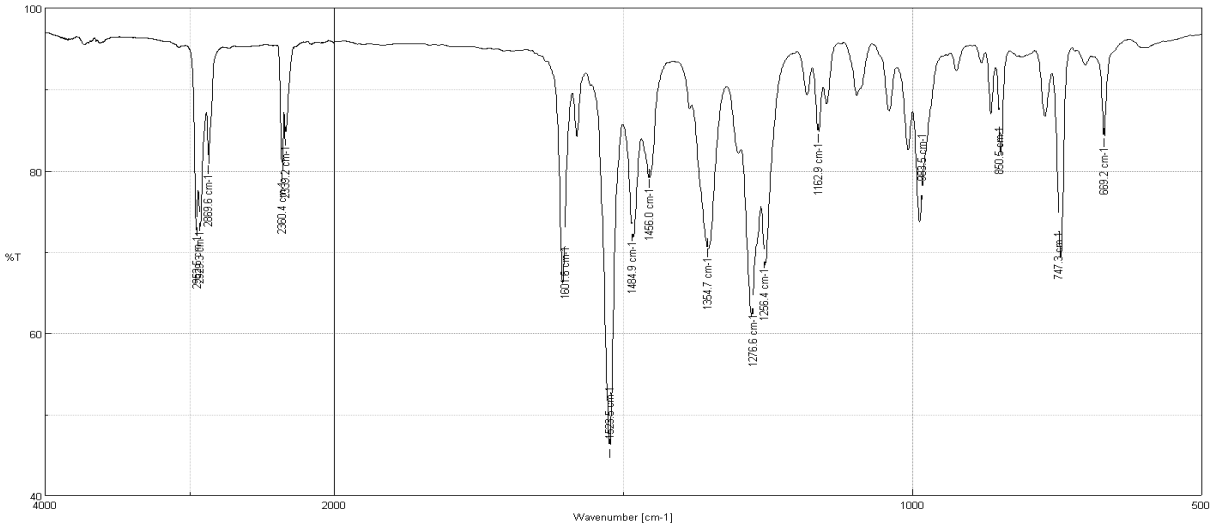
5.3 Alkoxy-catalysts

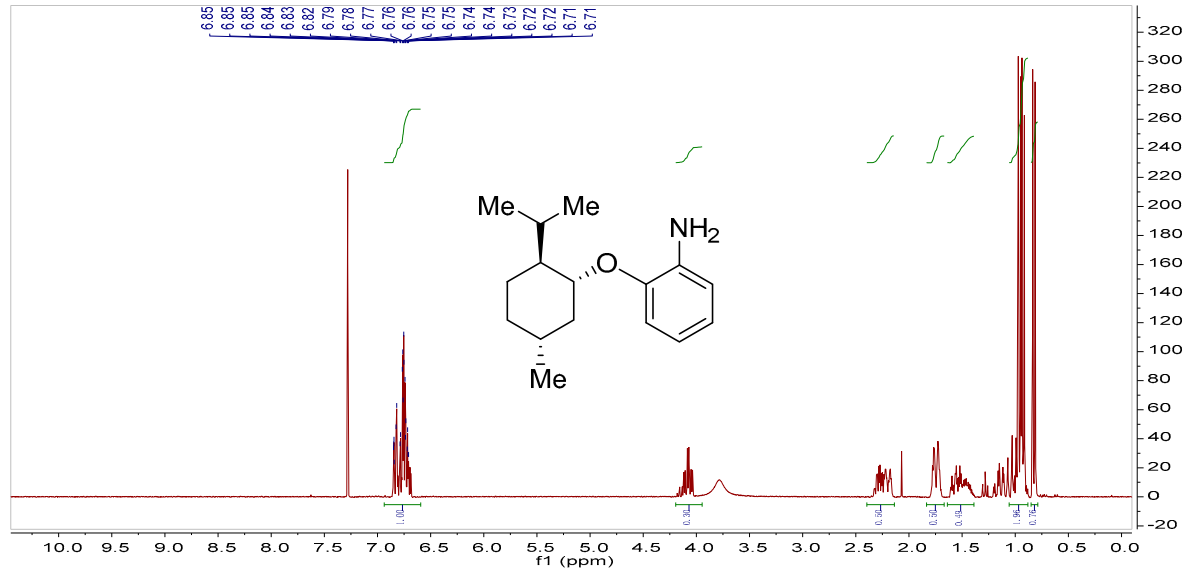
5.3.1 1-(((1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl)oxy)-2-nitrobenzene

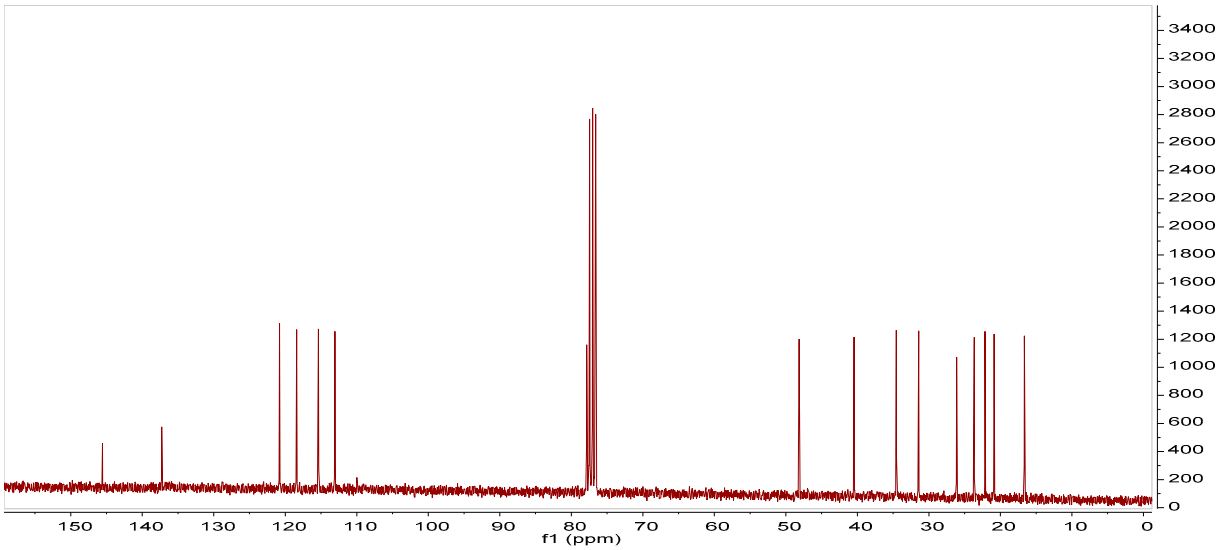


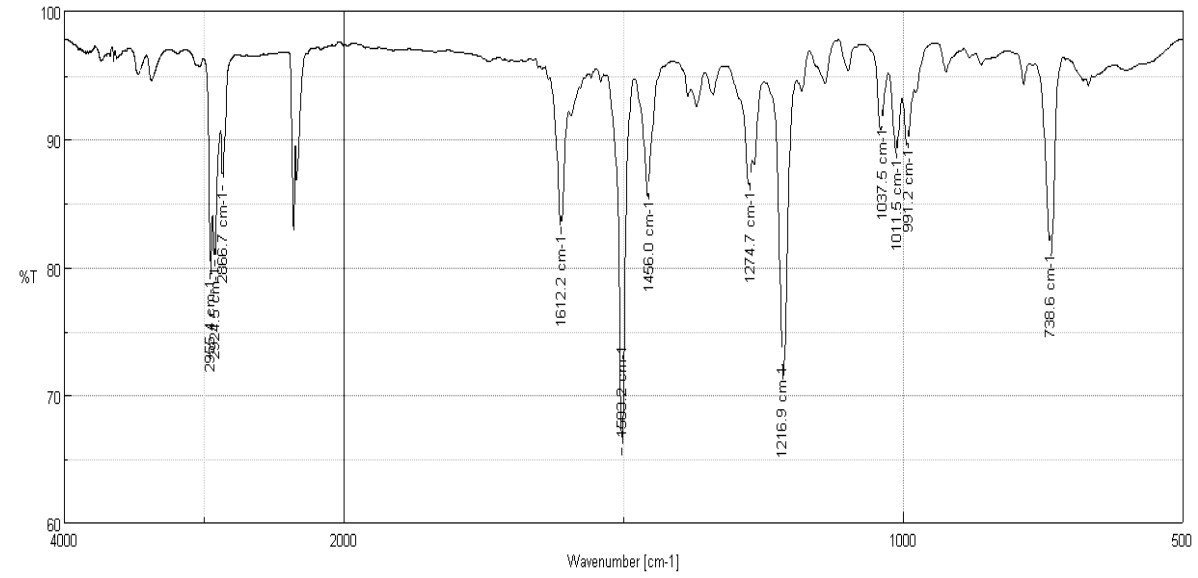




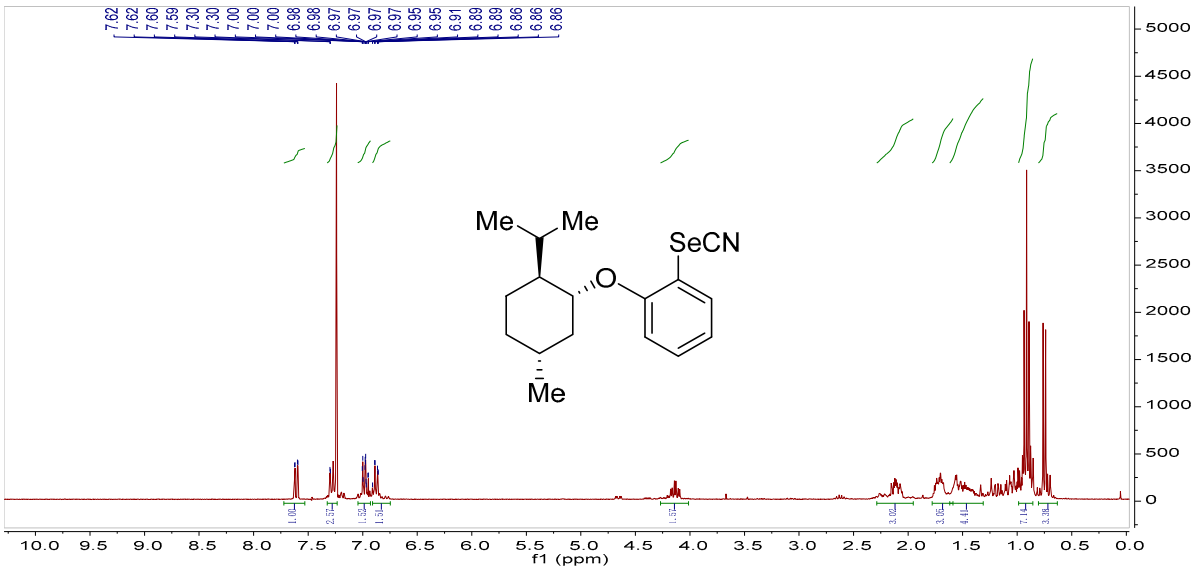
5.3.2 2-(((1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl)oxy)aniline (9b)

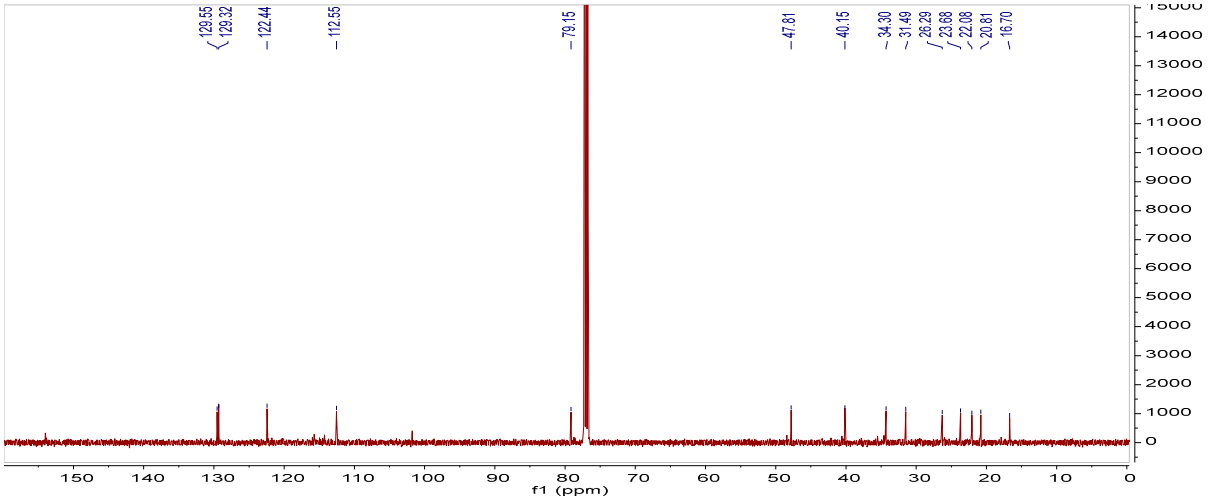


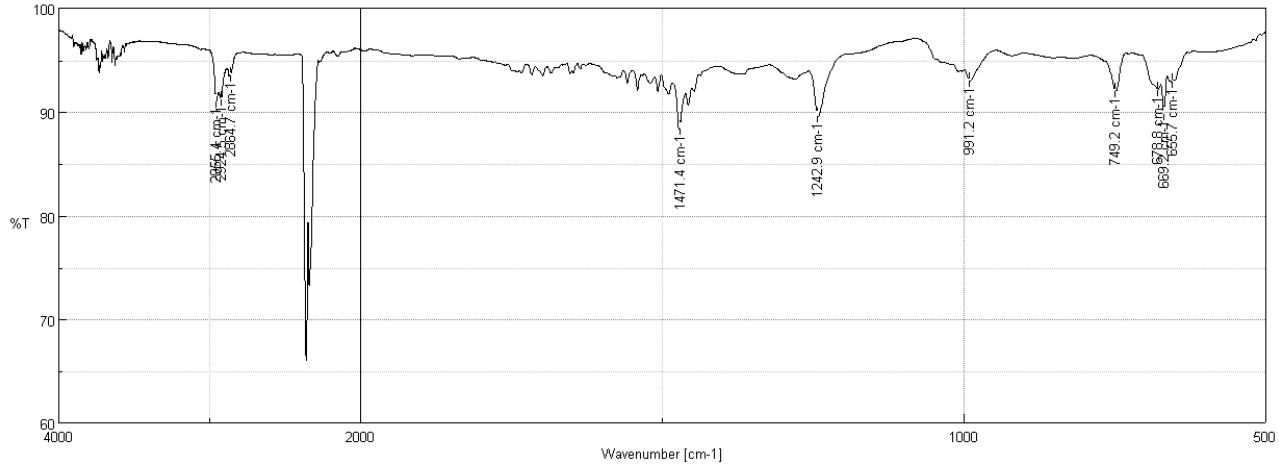




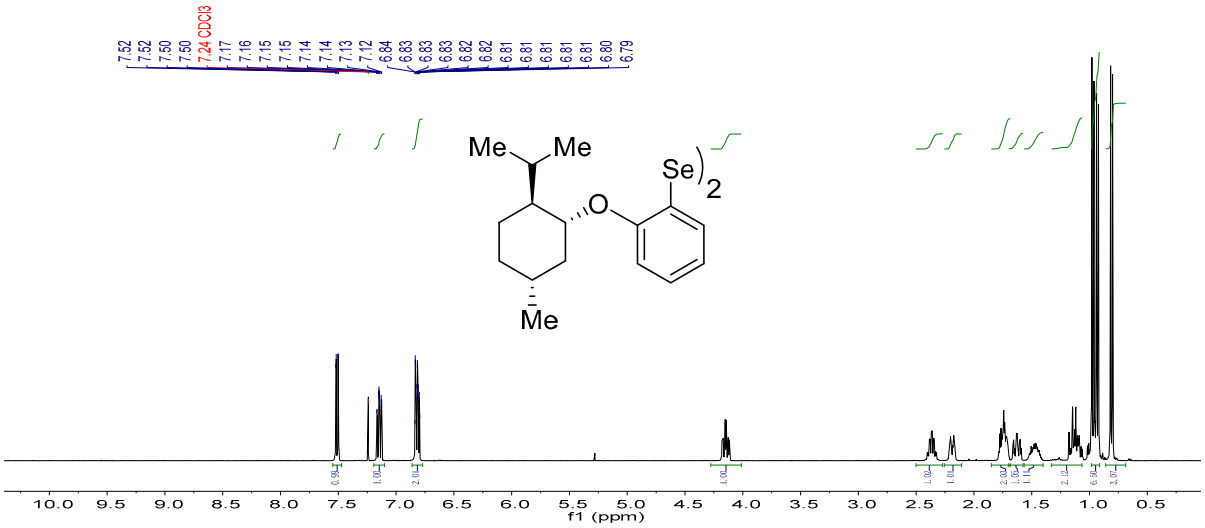
5.3.3 1-(((1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl)oxy)-2-selenocyanatobenzene (10b)

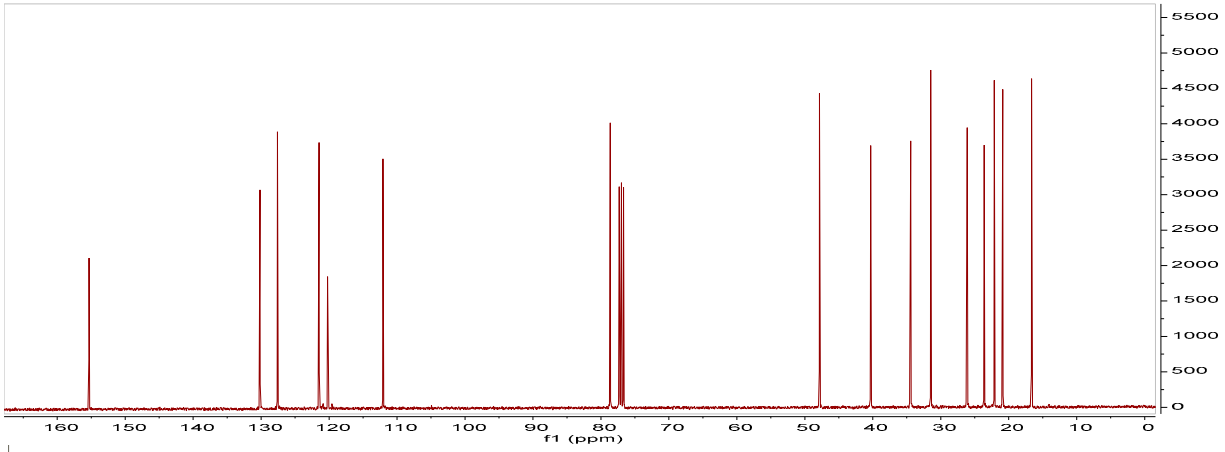


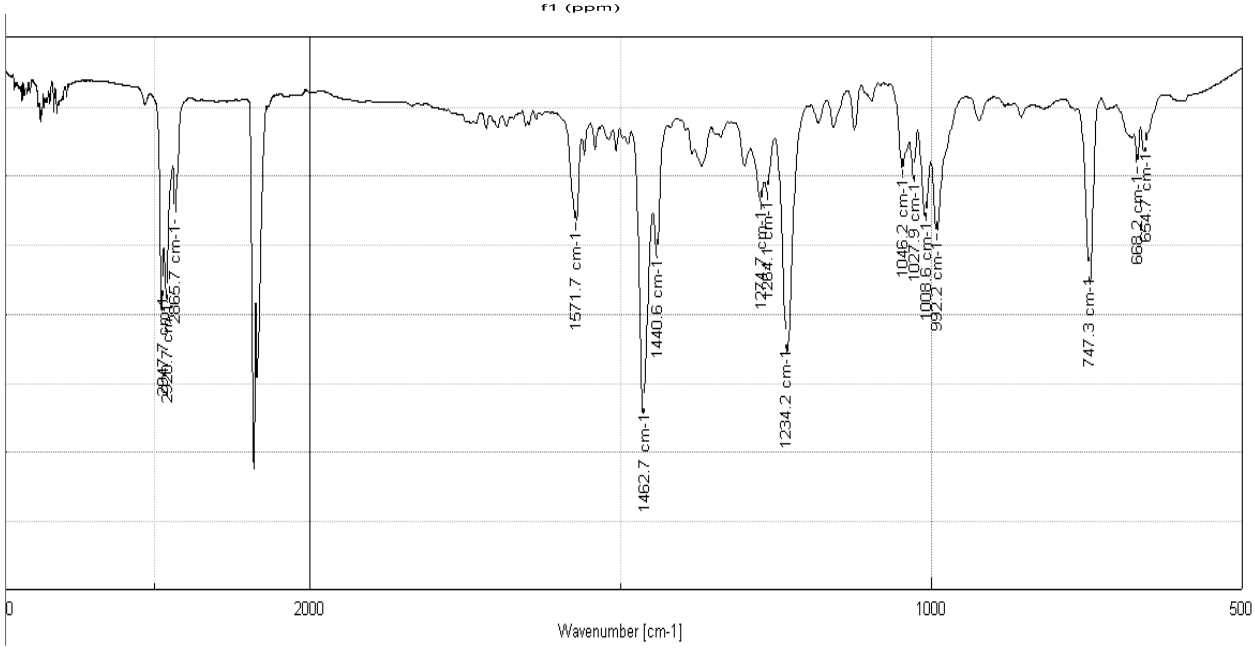




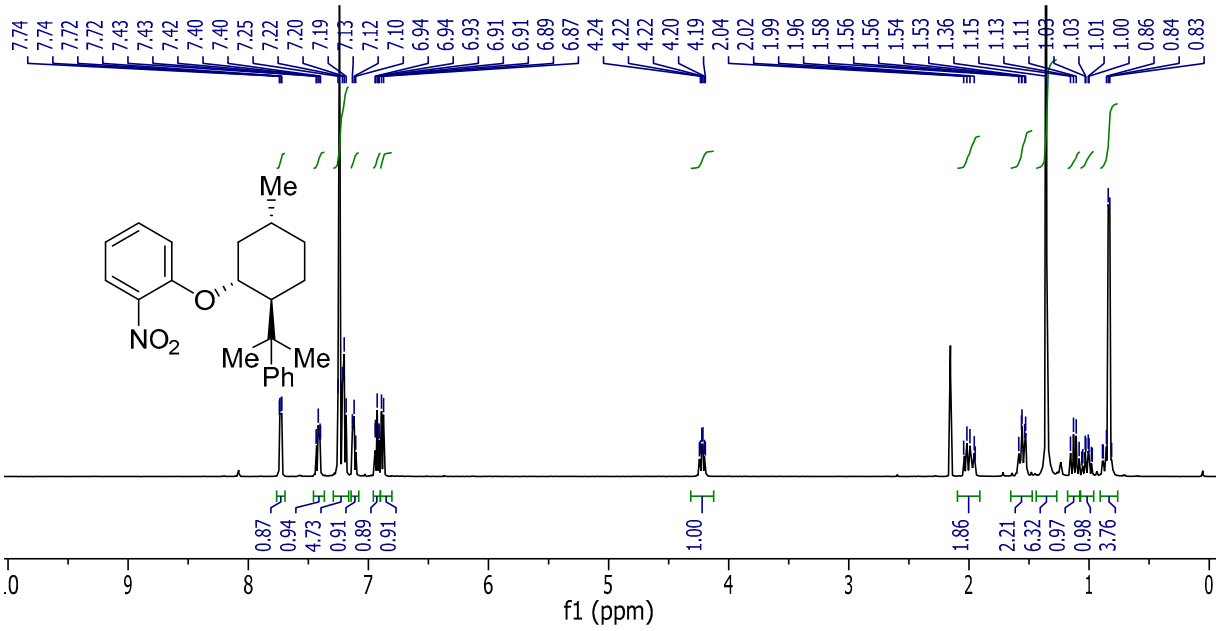
5.3.4 1,2-Bis(2-(((1*S*,2*R*,5*S*)-2-isopropyl-5-methylcyclohexyl)oxy)-phenyl)diselane (11b)

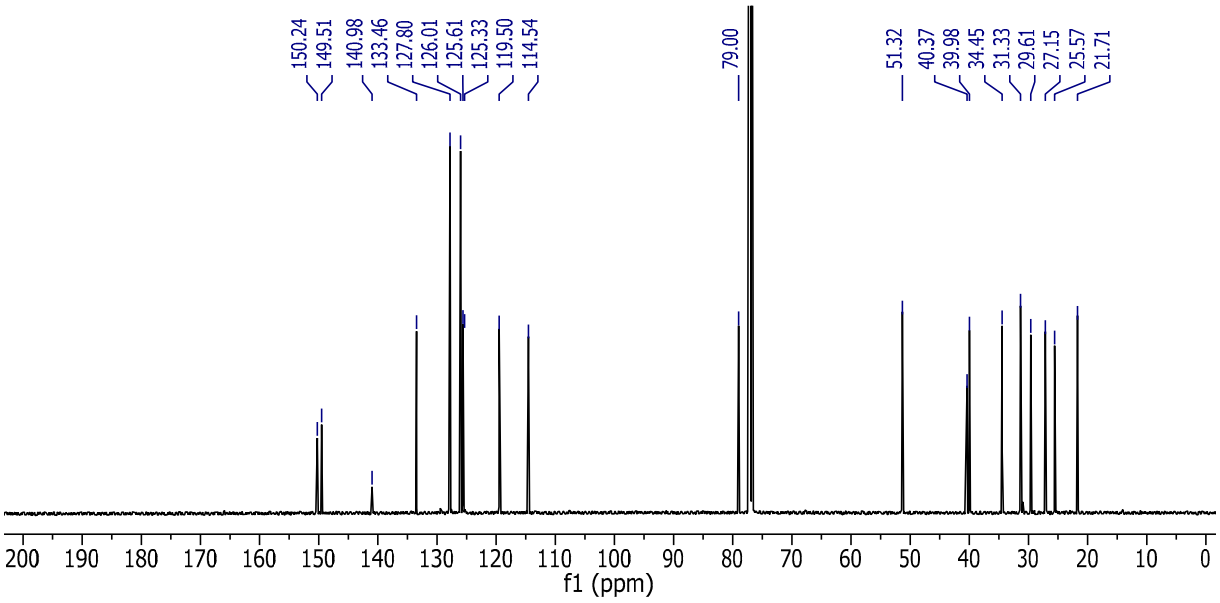


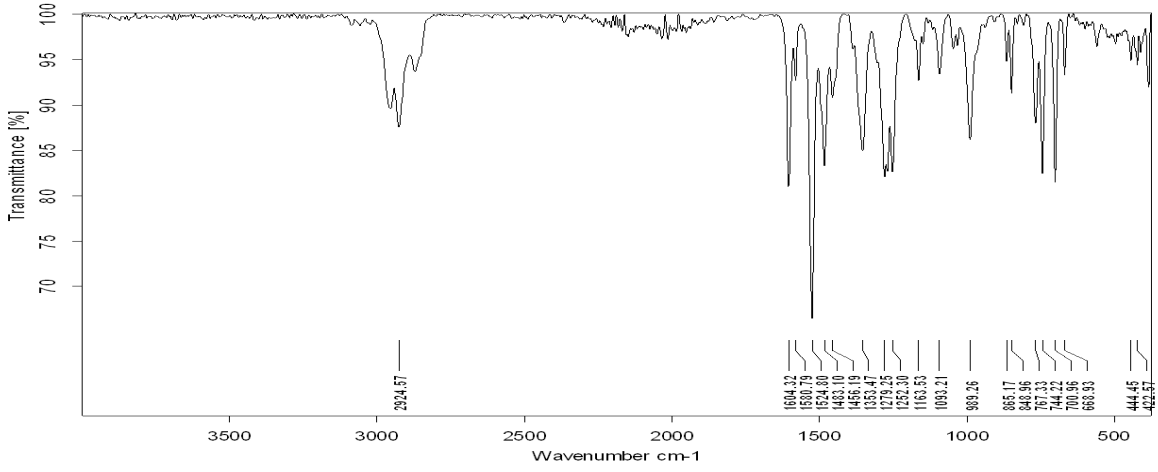




5.3.5 1-(((1*S*,2*R*,5*S*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)-2-nitrobenzol







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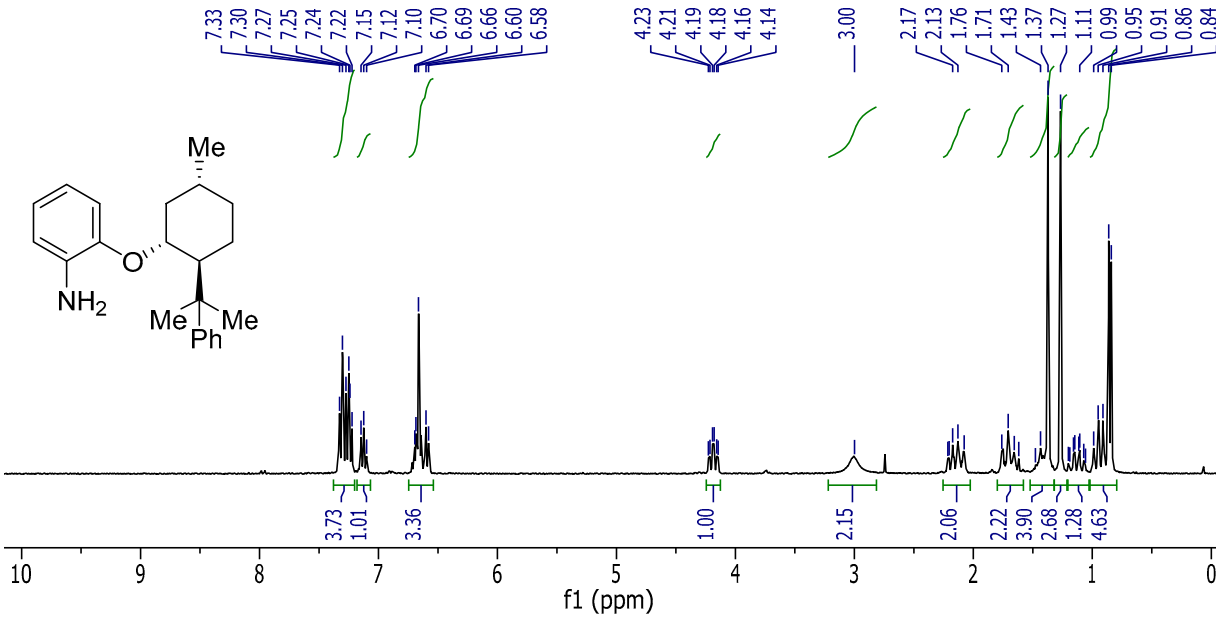
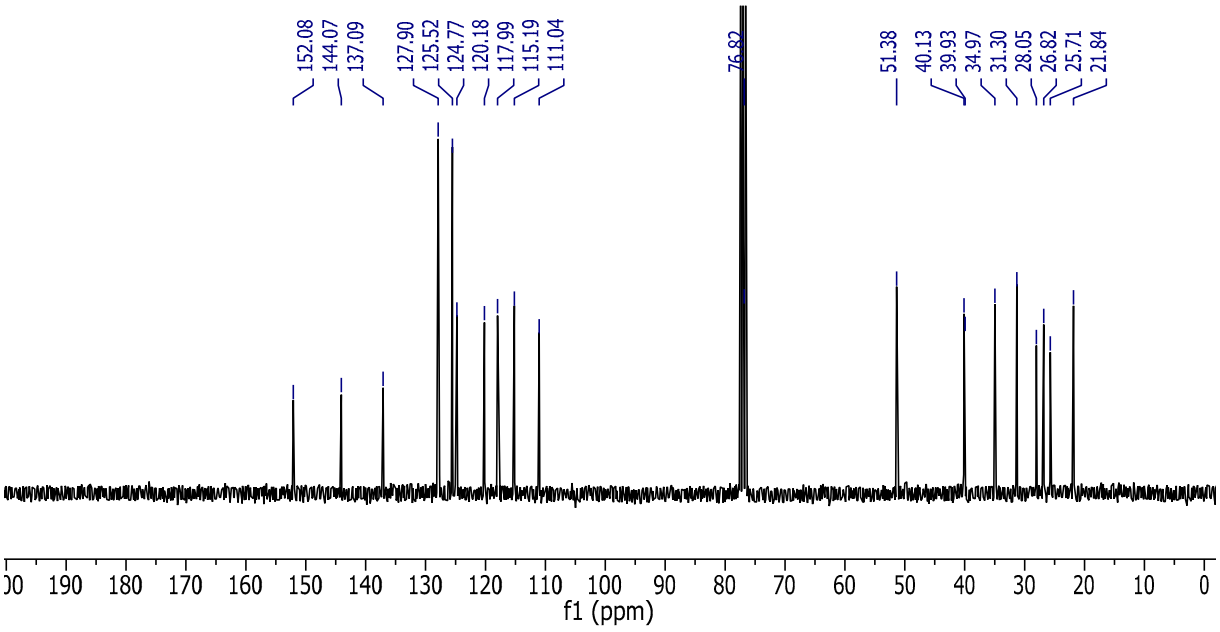
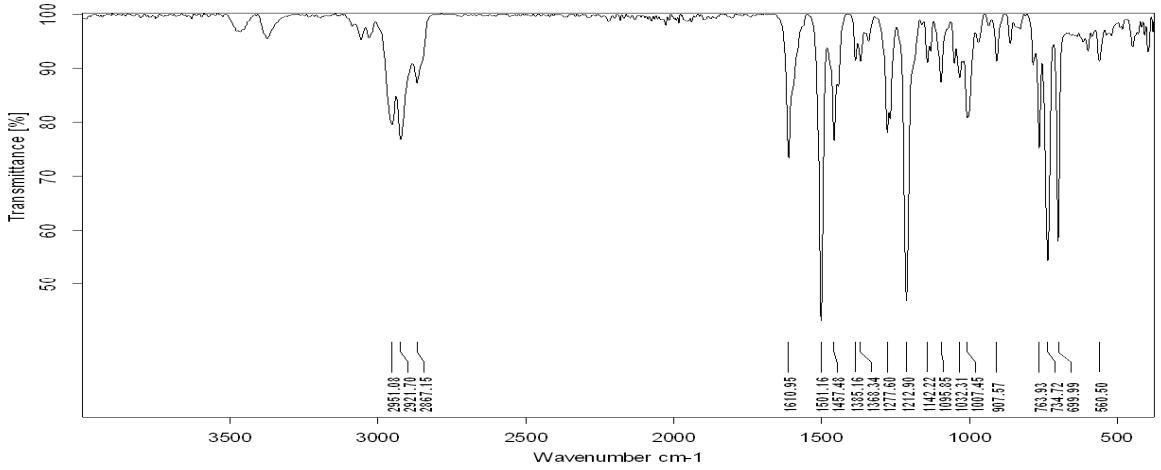
5.3.6 2-(((1S,2R,5S)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)aniline (9c)







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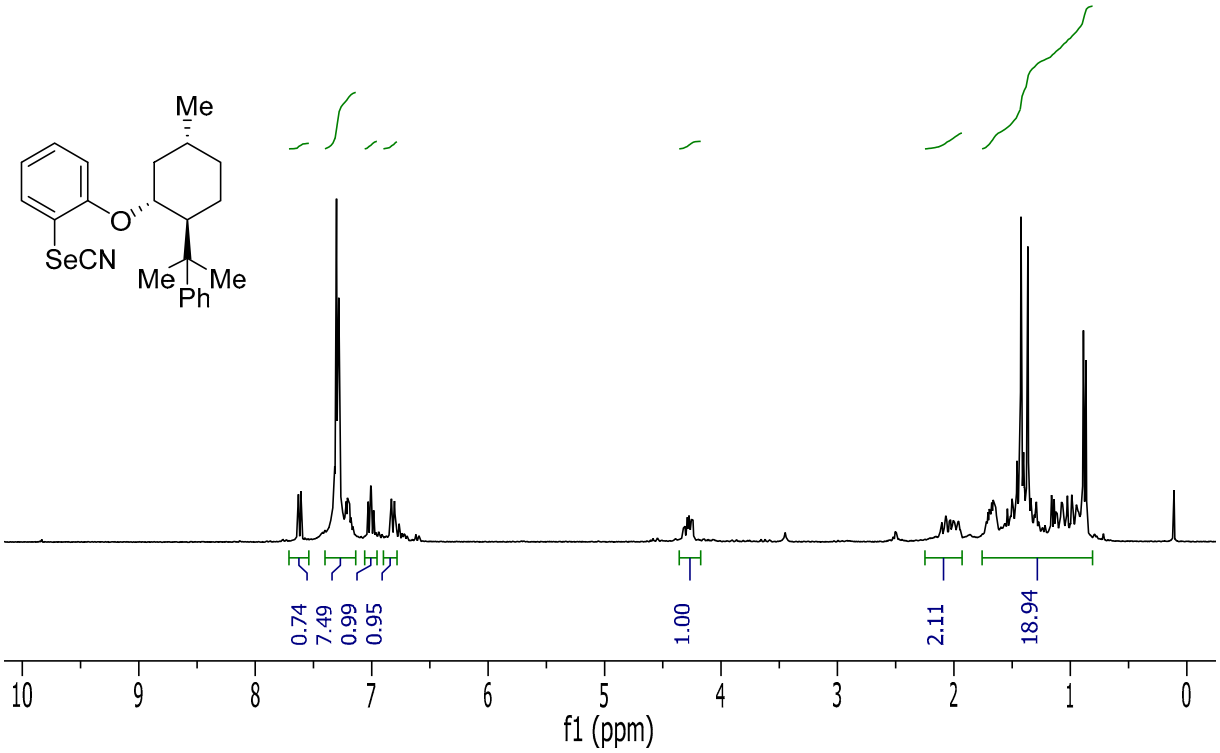
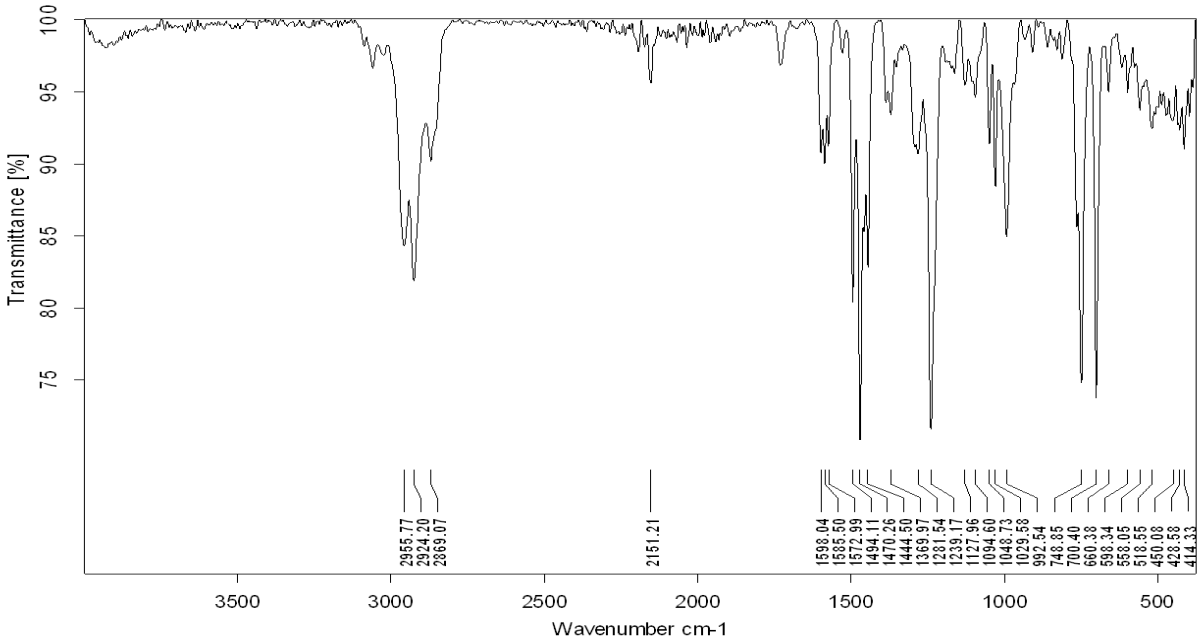
5.3.7 1-(((1*S*,2*R*,5*S*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)-2-selenocyanatobenzene (10c)





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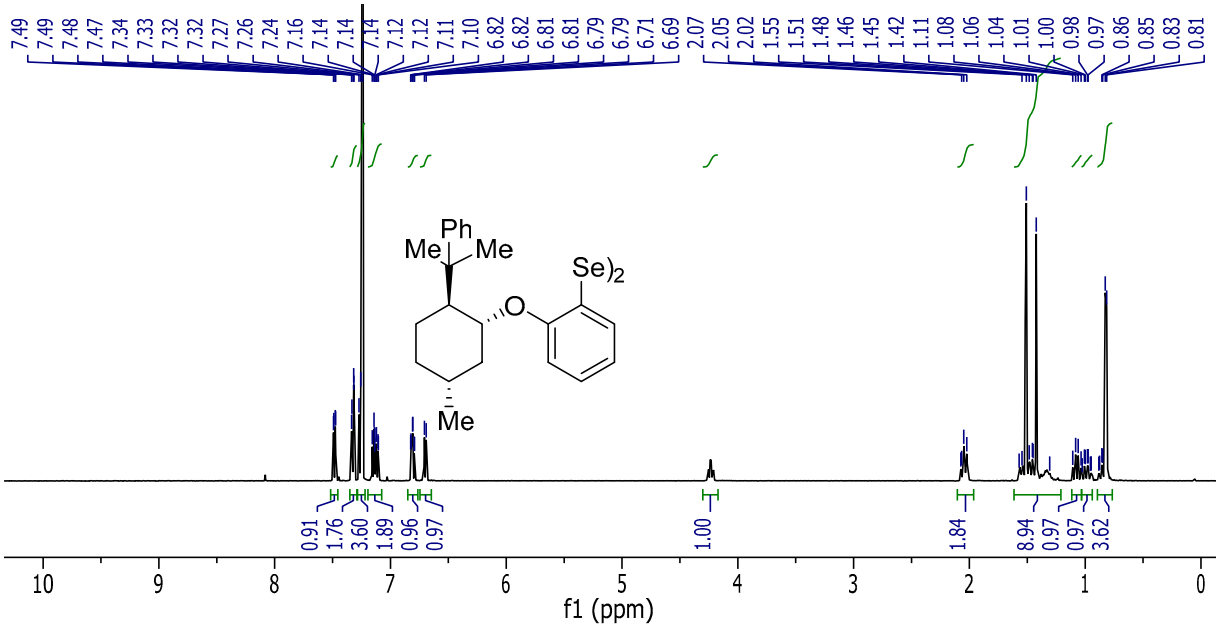
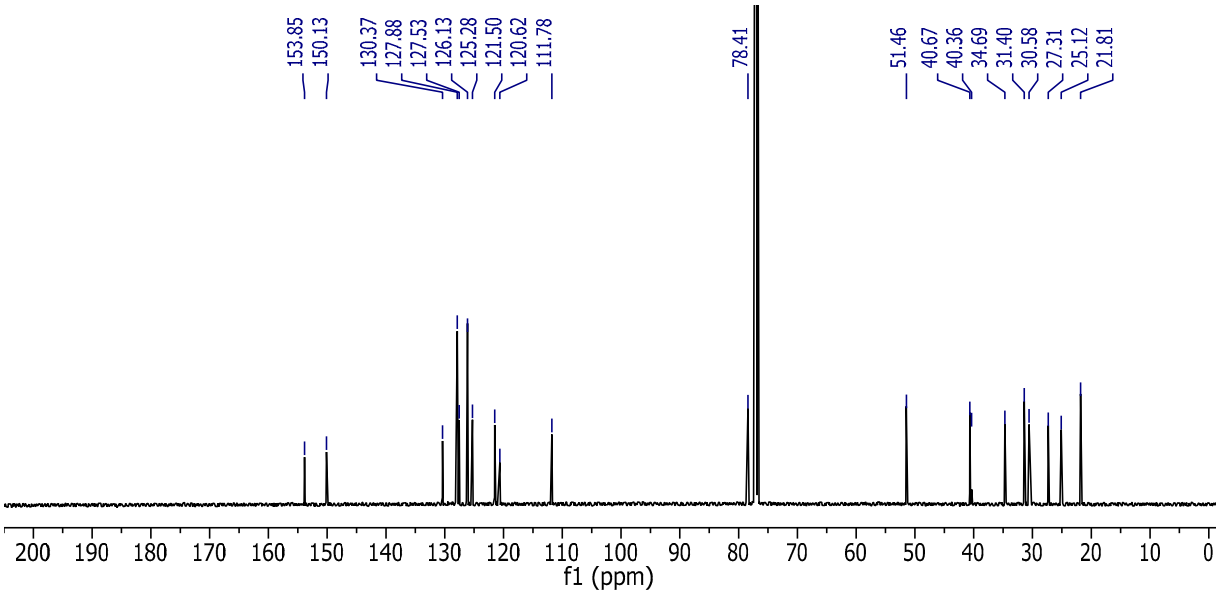
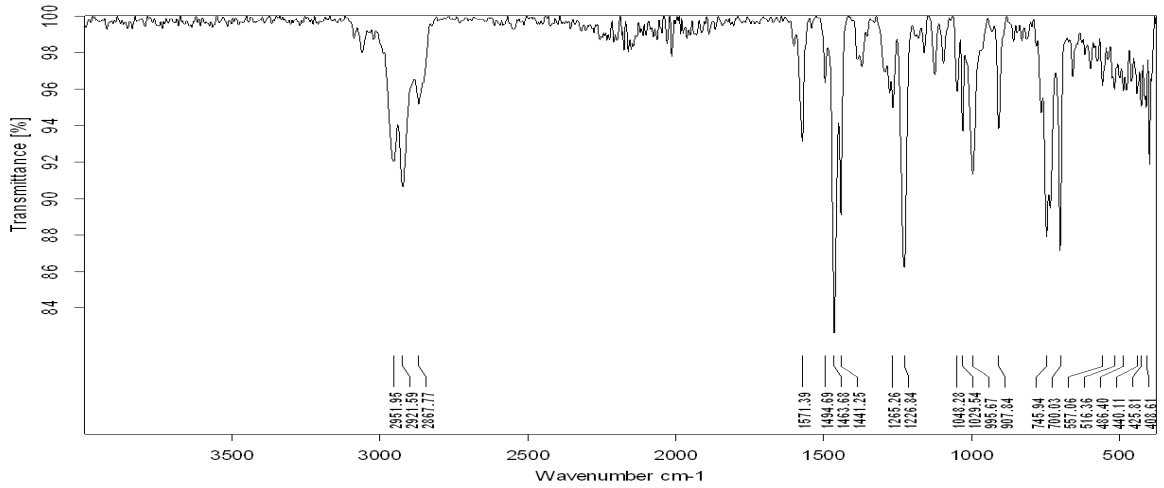
5.3.8 1,2-Bis(2-(((1*S*,2*R*,5*S*)-5-methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)-phenyl)diselane (11c)







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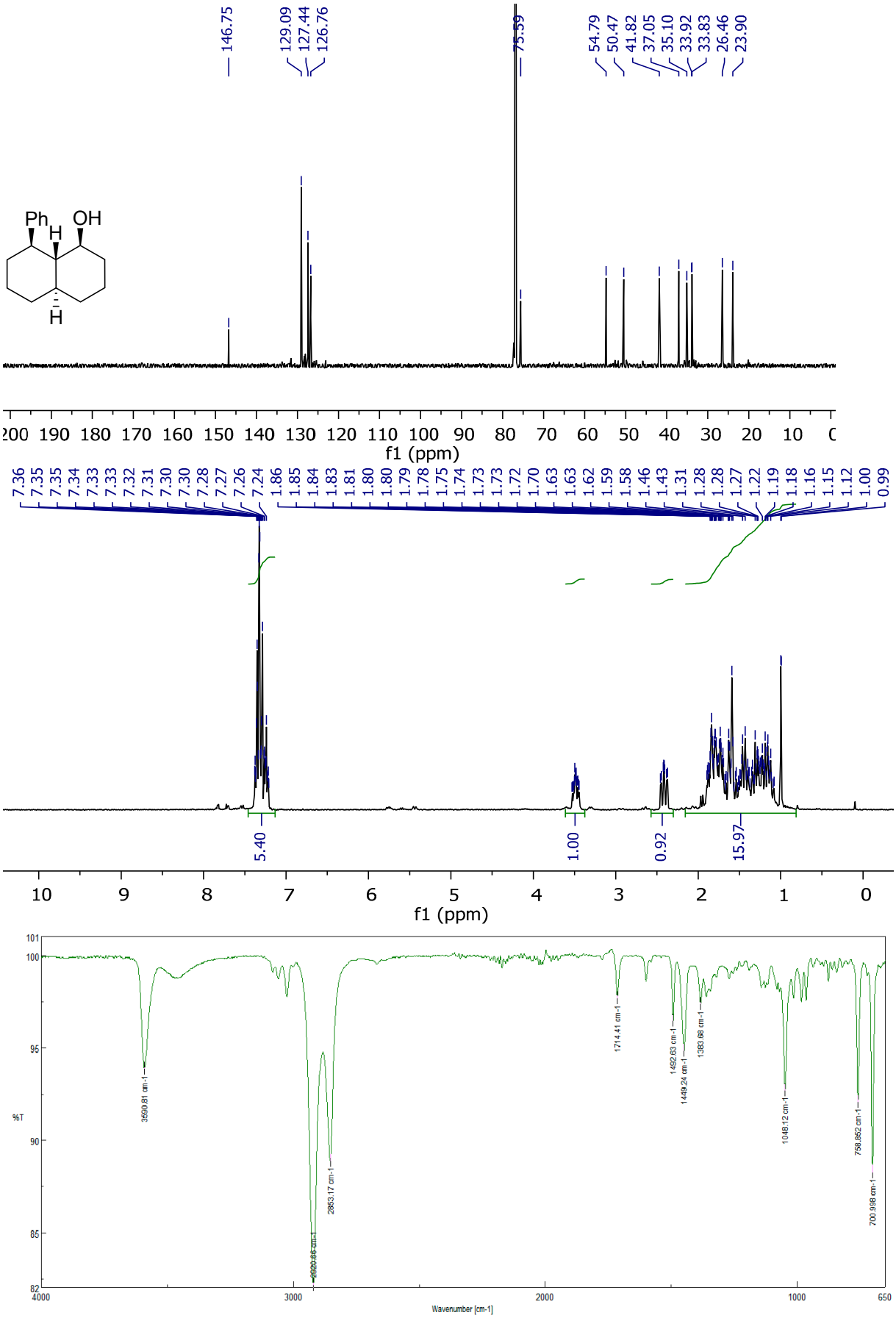
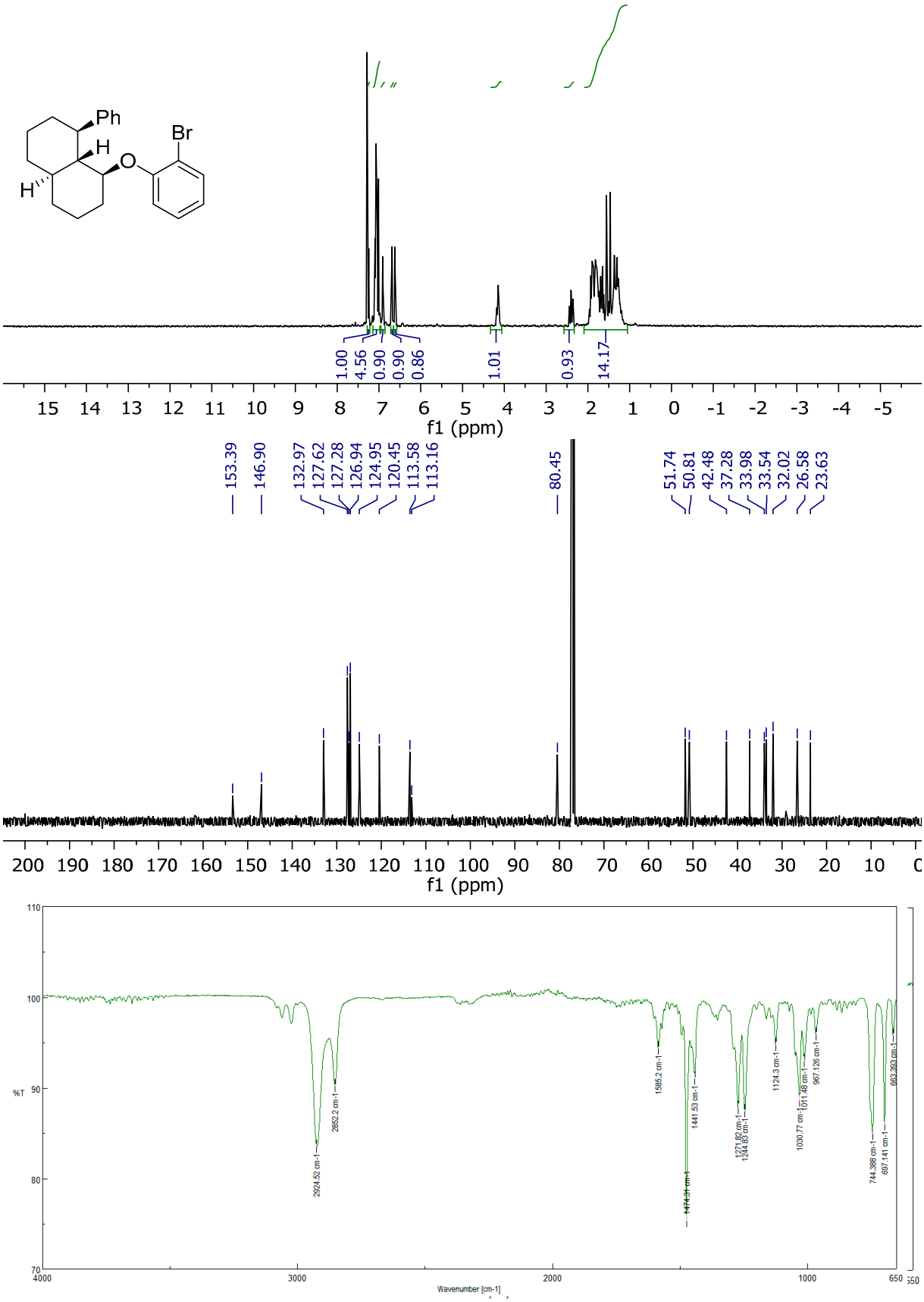
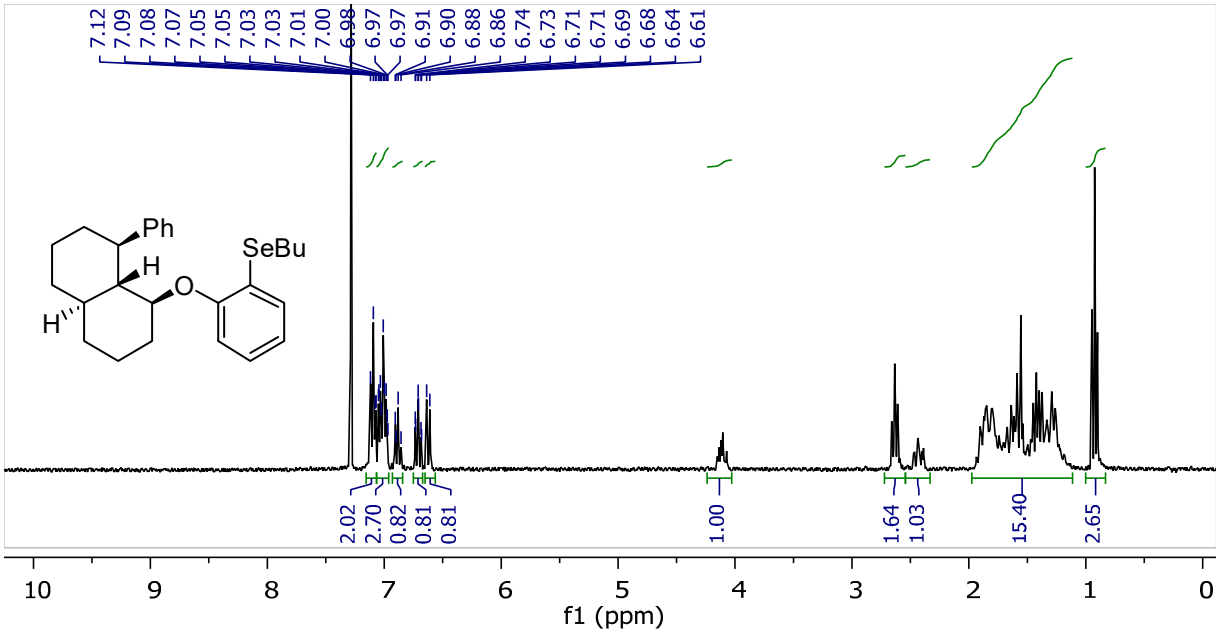
5.3.9 (1*S*,4a*R*,8*R*,8a*R*)-8-phenyldecahydronaphthalen-1-ol

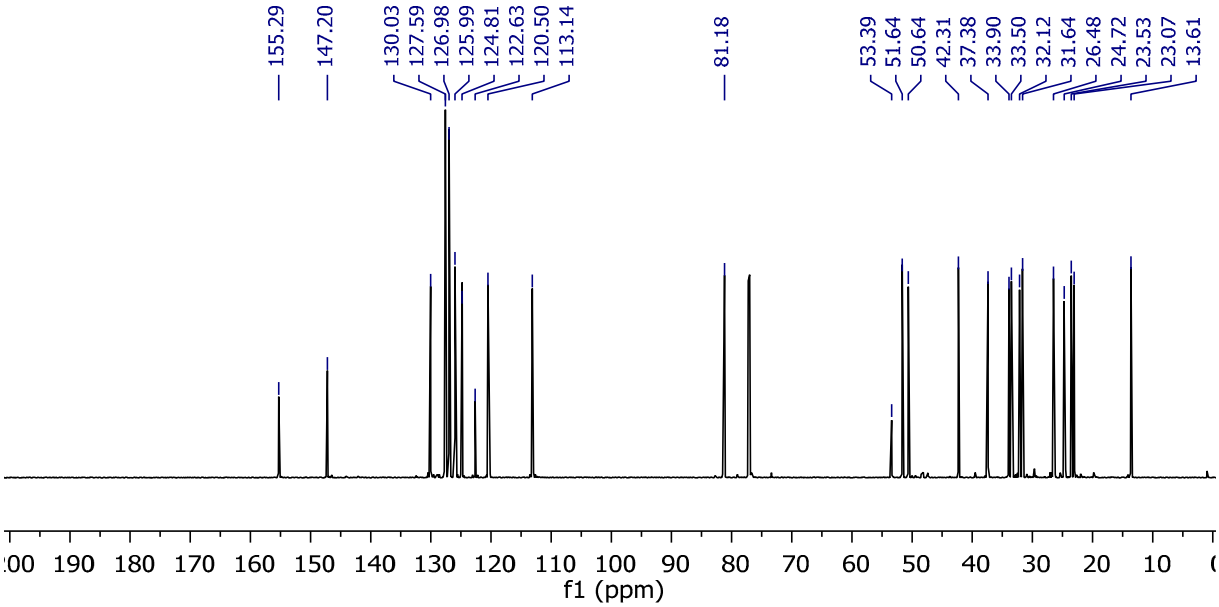


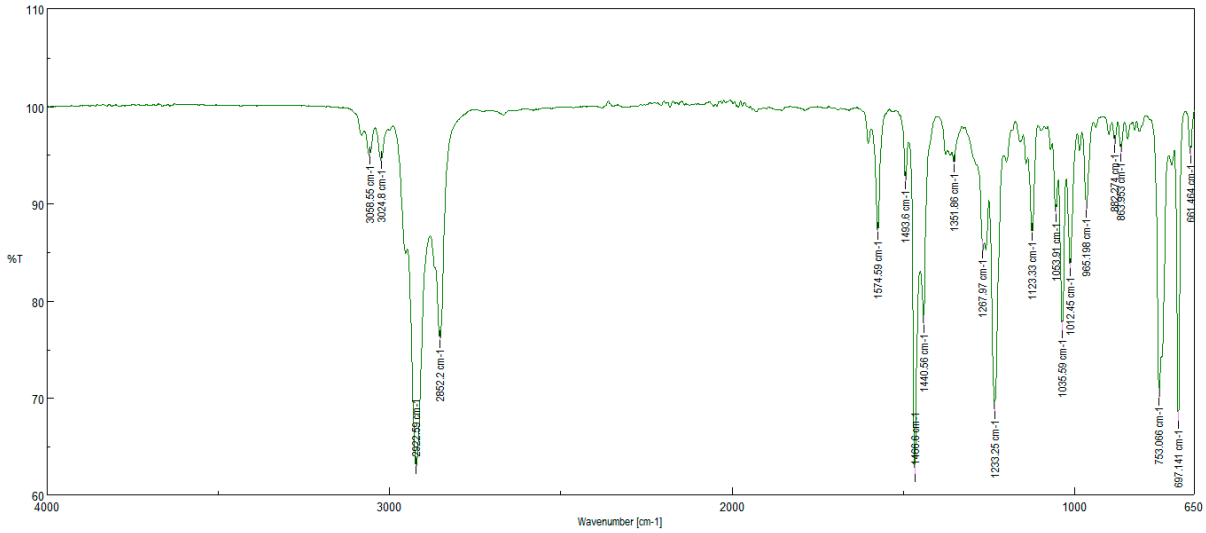
5.3.10 (1*S*,4a*R*,8*R*,8a*R*)-1-(2-bromophenoxy)-8-phenyldecahydronaphthalene (16)



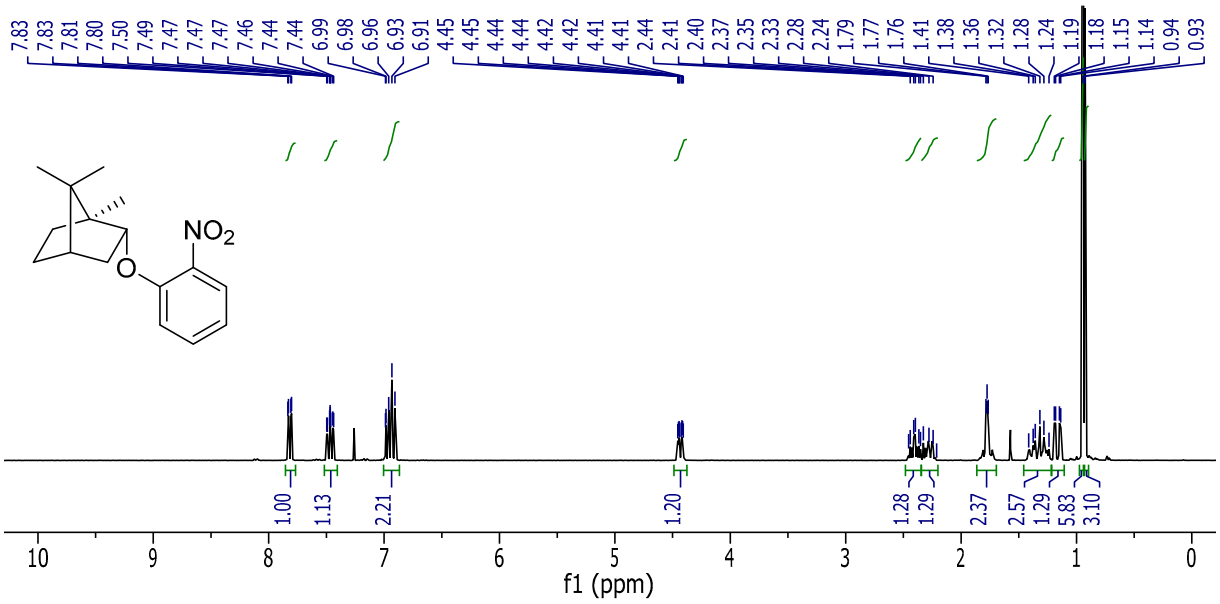
5.3.11 Butyl(2-(((1*S*,4a*R*,8*R*,8a*R*)-8-phenyldecahydronaphthalen-1-yl)oxy)phenyl)selane (11d)

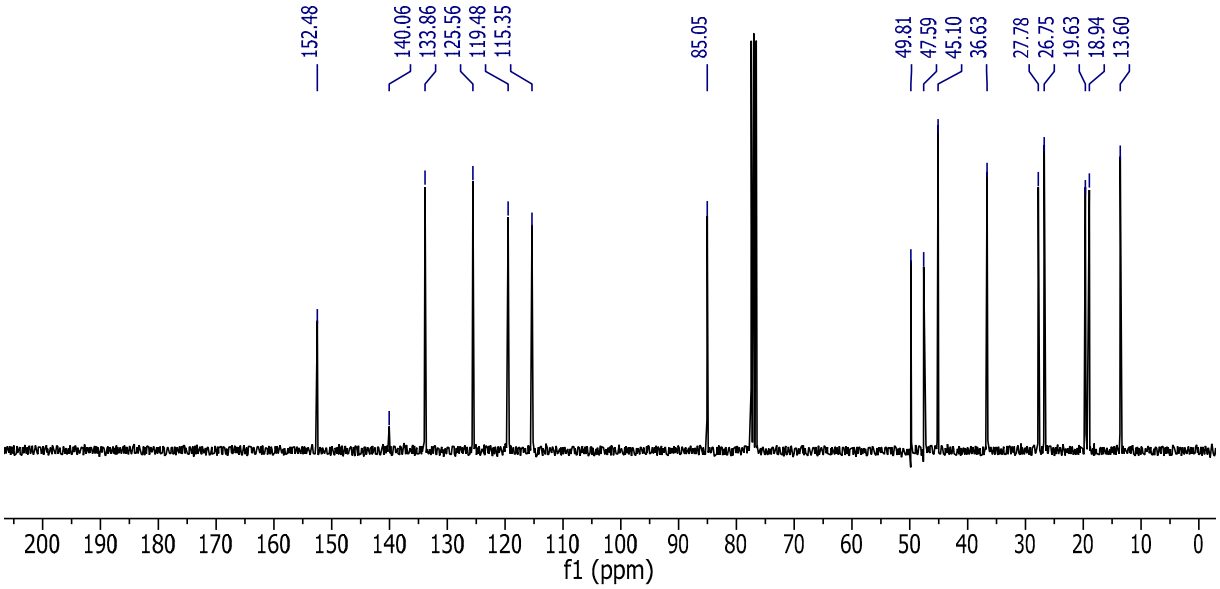


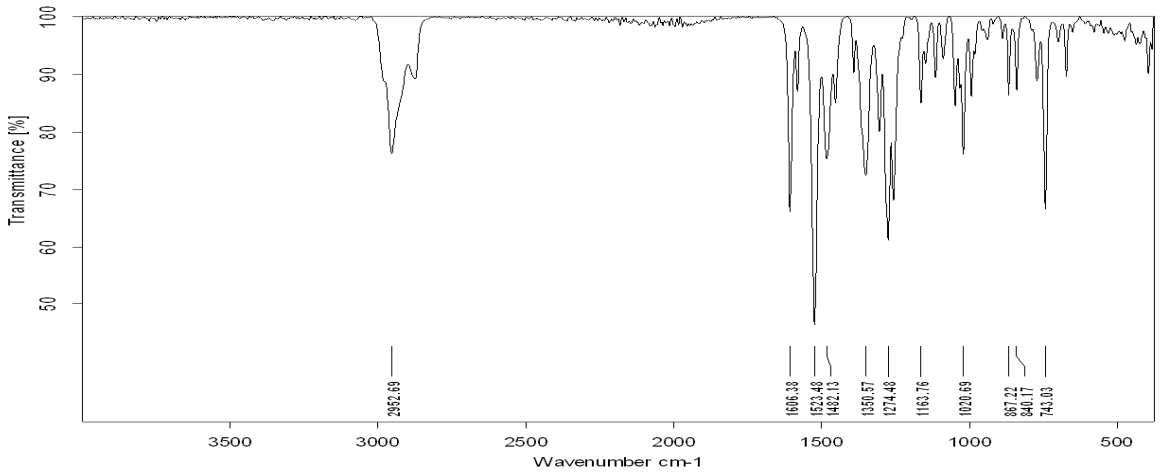

155.29
147.20
130.03
127.59
126.98
125.99
124.81
122.63
120.50
113.14
81.18
53.39
51.64
50.64
42.31
37.38
33.90
33.50
32.12
31.64
26.48
24.72
23.53
23.07
13.61
f1 (ppm)


%T
Wavenumber [cm-1]
3068.55 cm-1
3024.8 cm-1
2852.2 cm-1
1574.59 cm-1
1493.6 cm-1
1440.56 cm-1
1351.86 cm-1
1297.97 cm-1
1233.25 cm-1
1123.33 cm-1
1063.91 cm-1
1035.59 cm-1
1012.45 cm-1
965.198 cm-1
863.303 cm-1
753.066 cm-1
697.141 cm-1
661.464 cm-1

5.3.12 (1*S*)-1,7,7-Trimethyl-2-(2-nitrophenoxy)bicyclo[2.2.1]heptane







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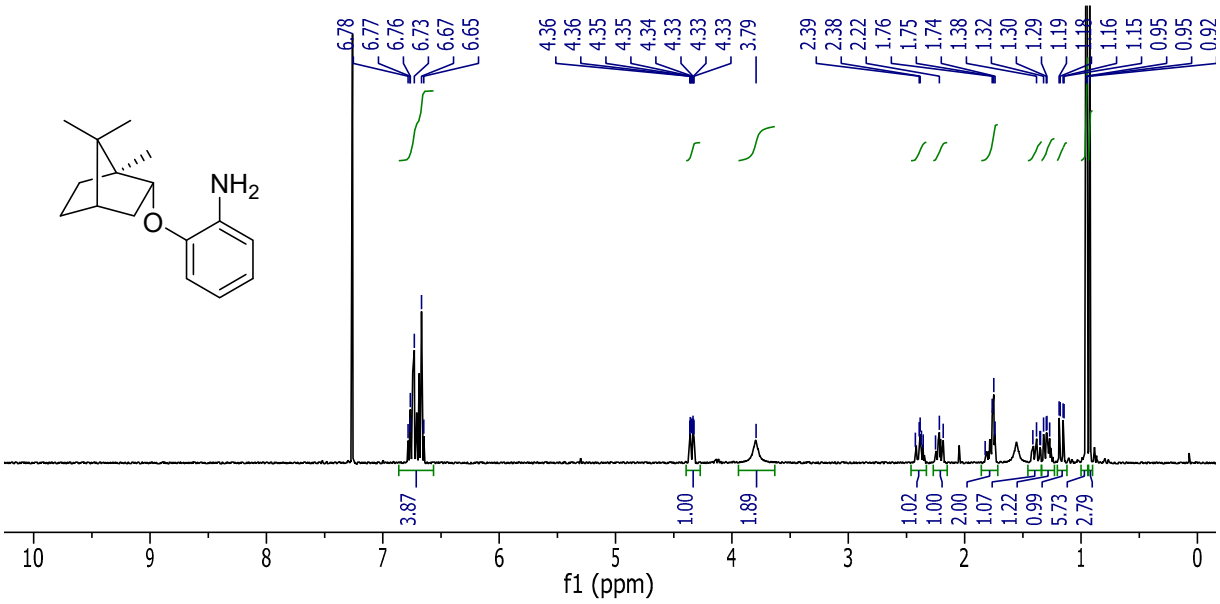
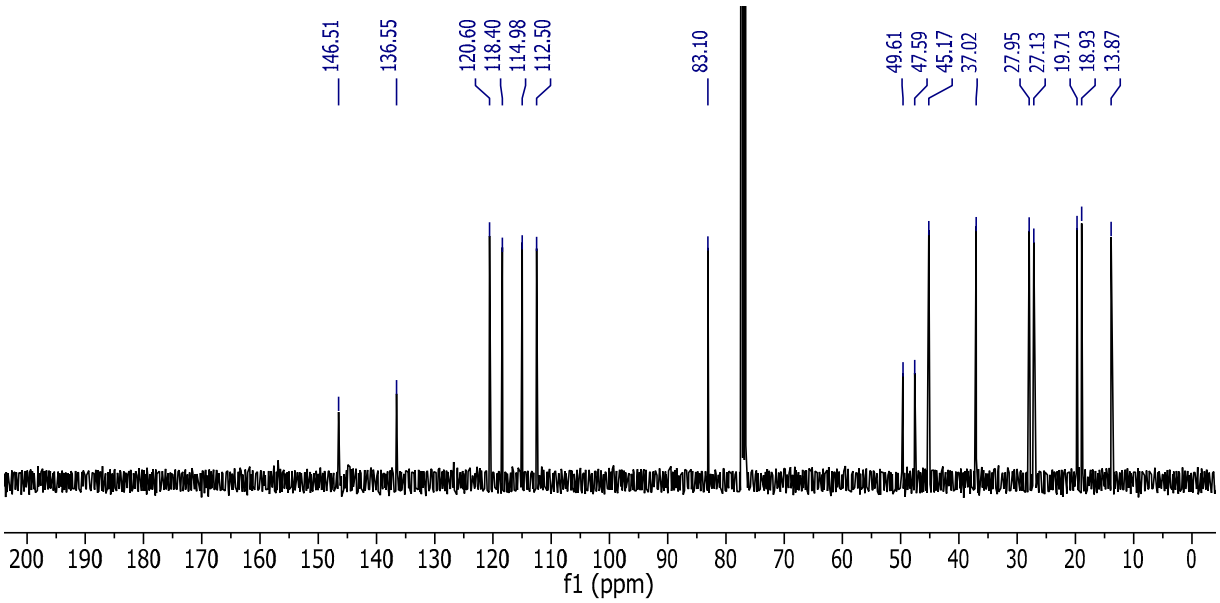
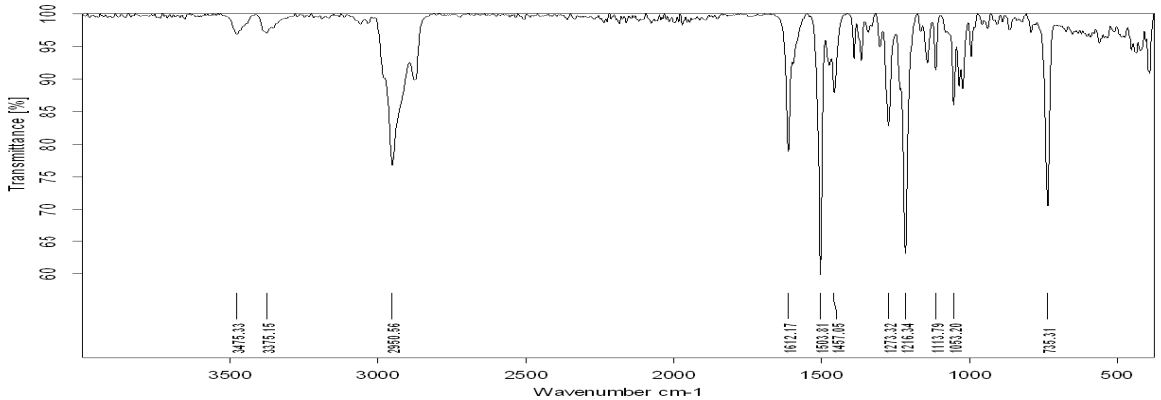
5.3.13 2-(((1*S*,2*R*,4*S*)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl)oxy)-aniline (9a)







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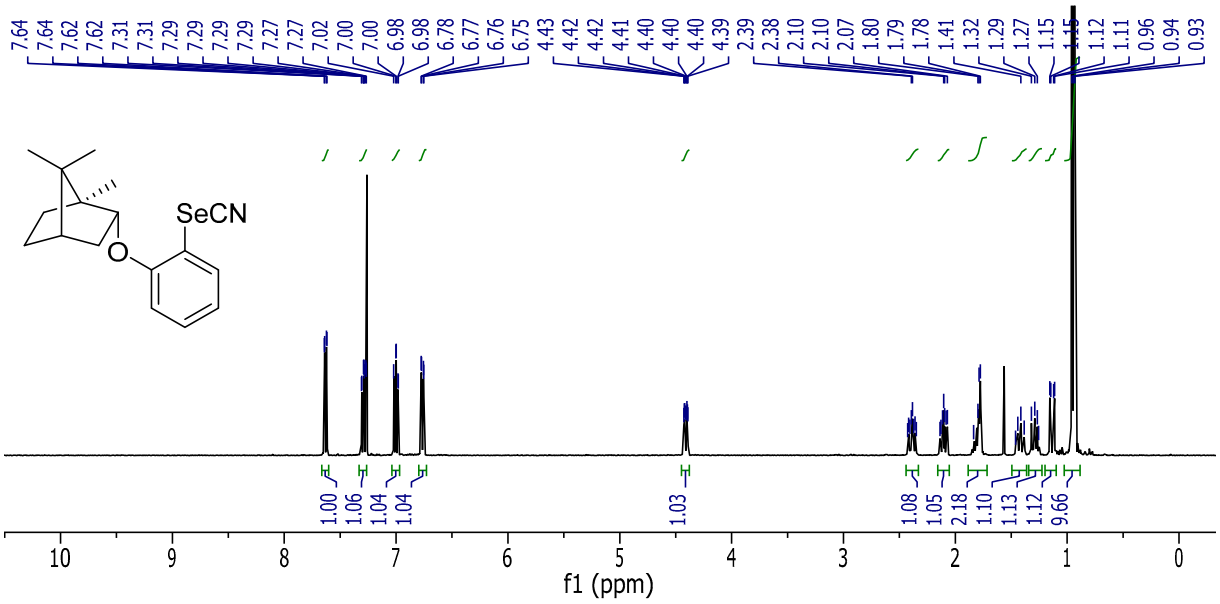
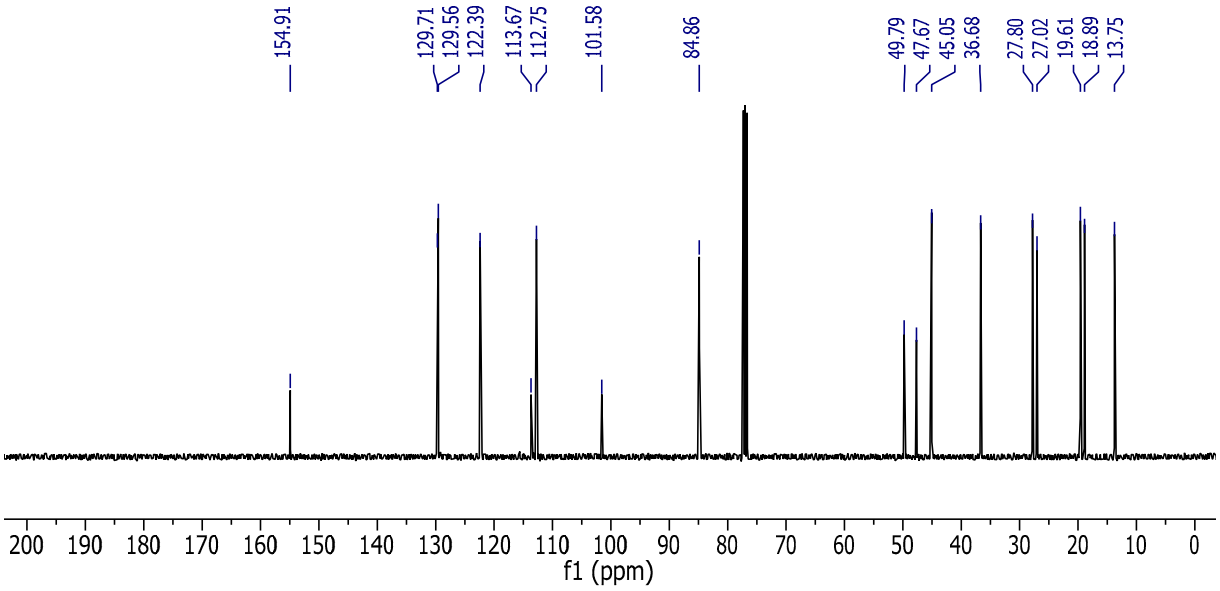
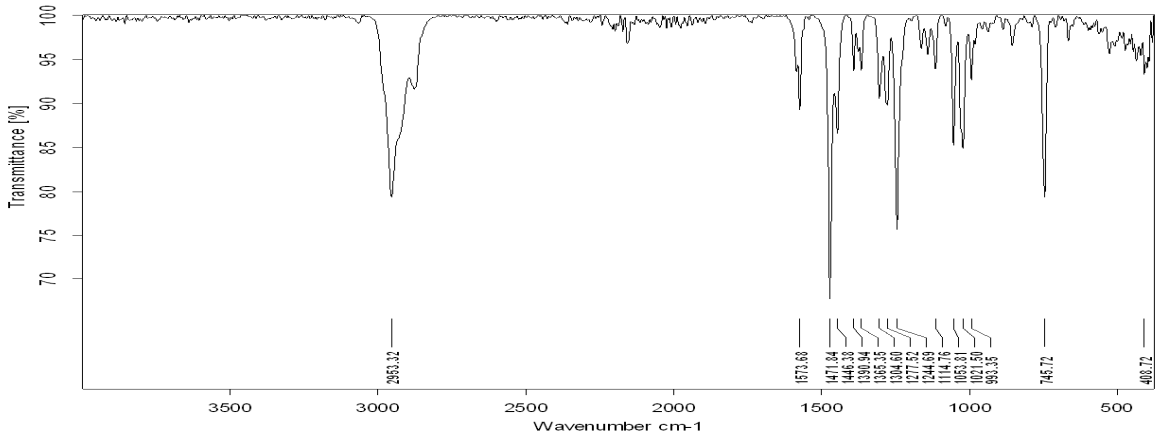
5.3.14 (1*S*)-1,7,7-trimethyl-2-(2-selenocyanatophenoxy)bicyclo[2.2.1]-heptane (10a)







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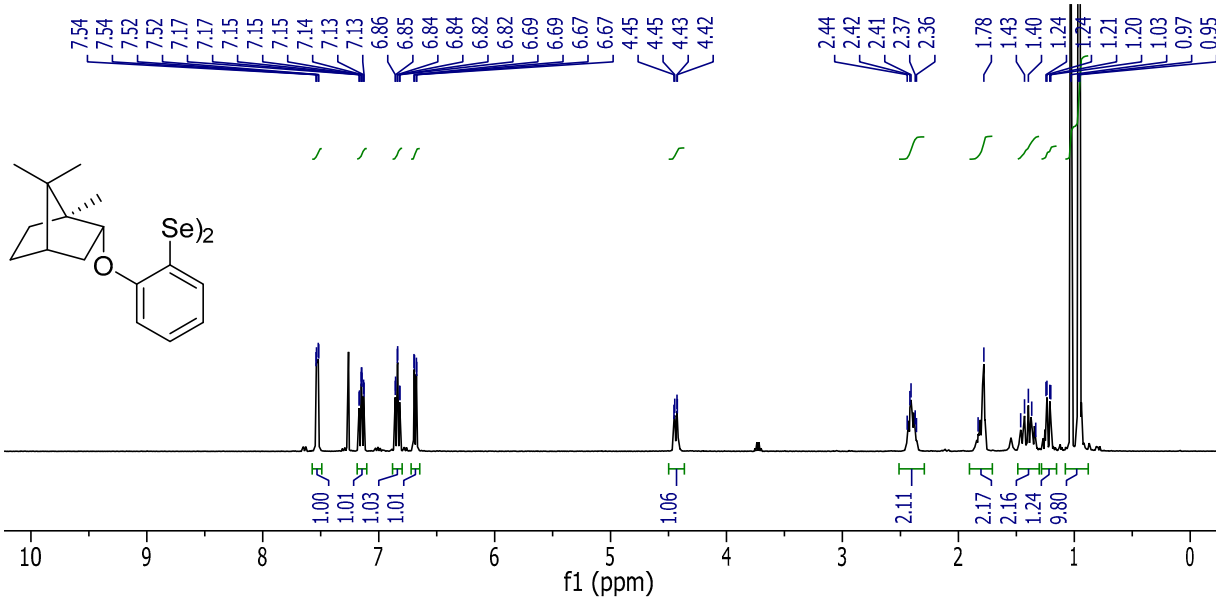
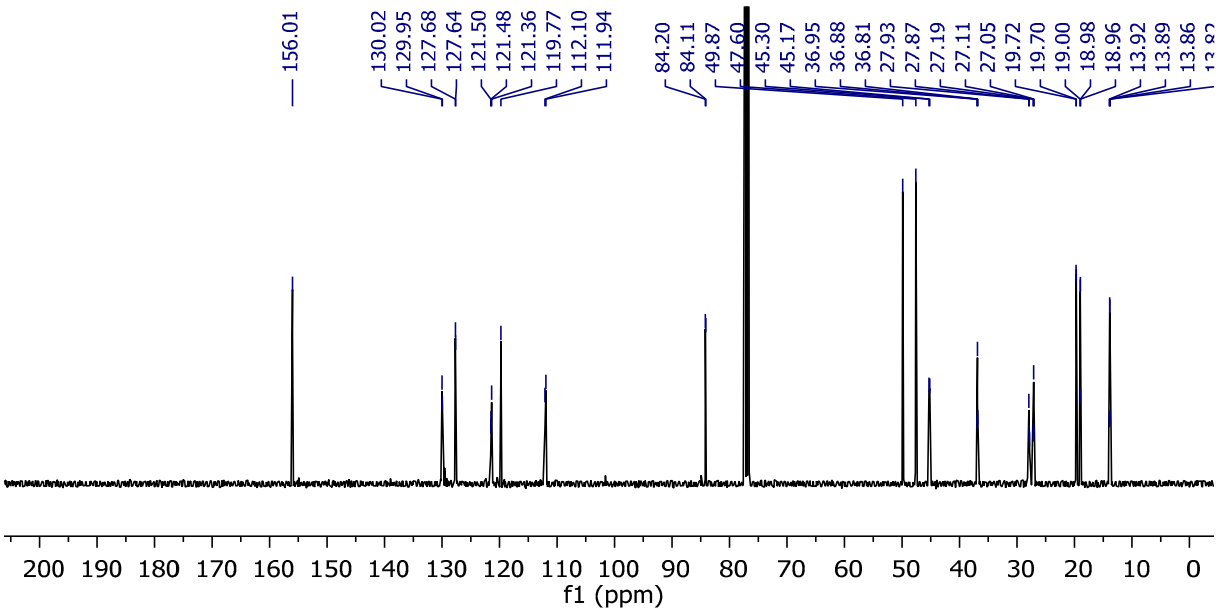
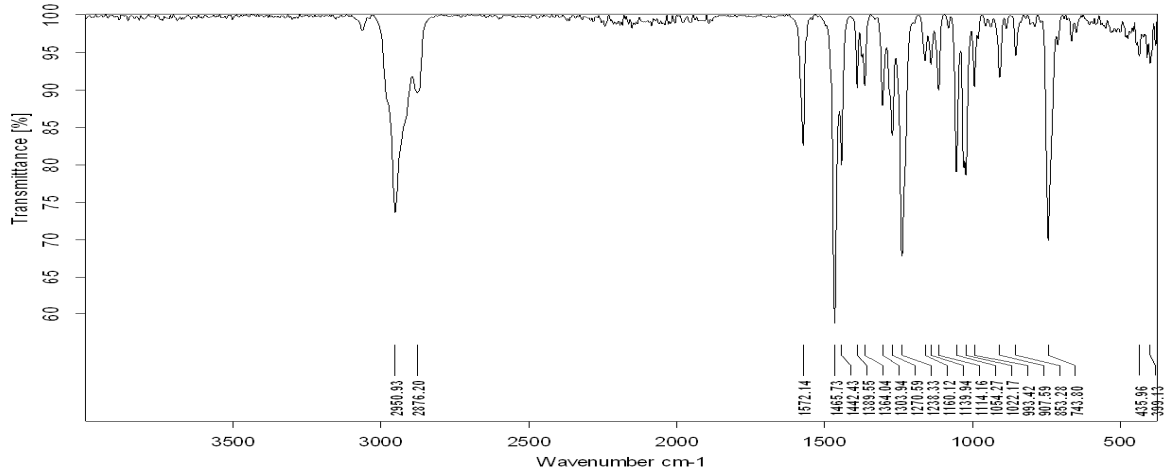
5.3.15 1,2-Bis(2-(((1*S*,2*R*,4*S*)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)oxy)phenyl) diselenide (11a)







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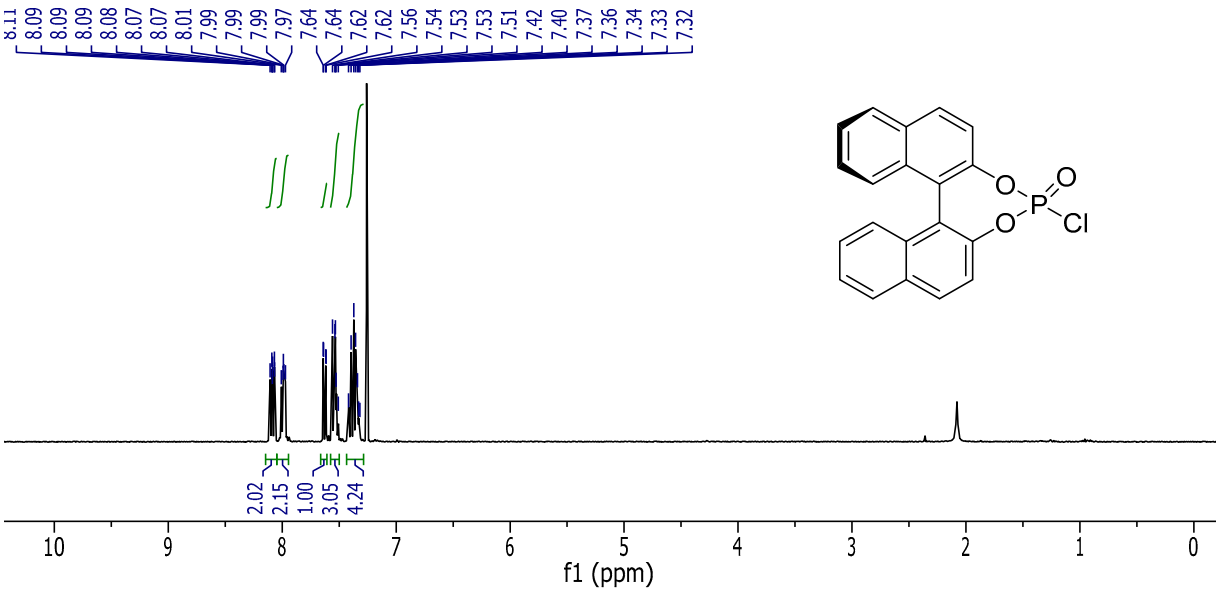
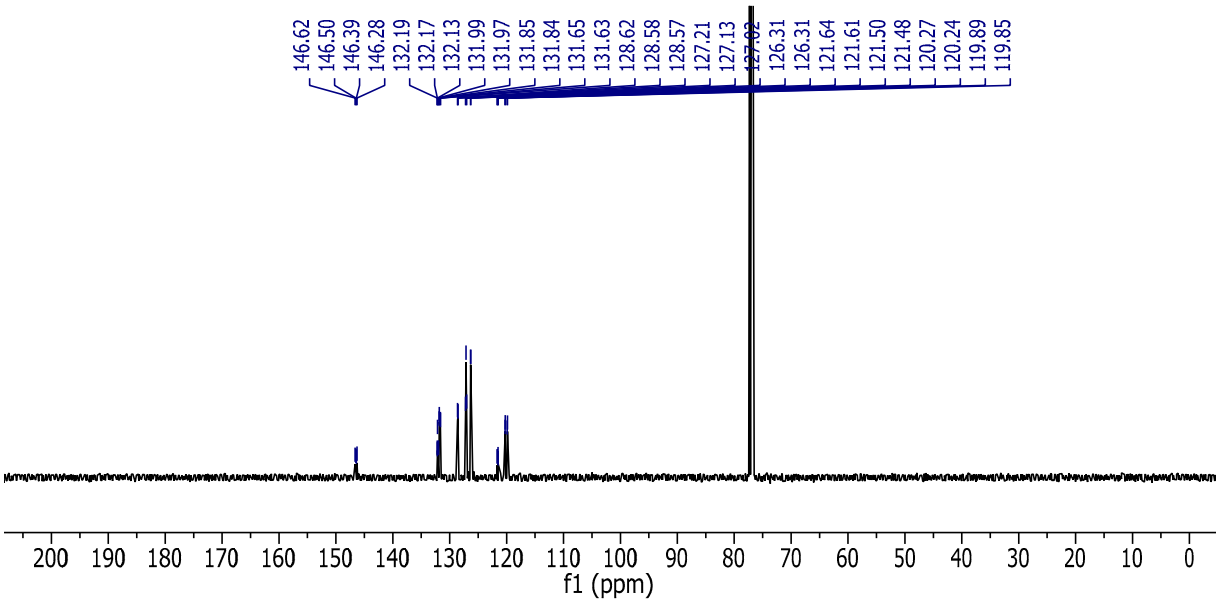
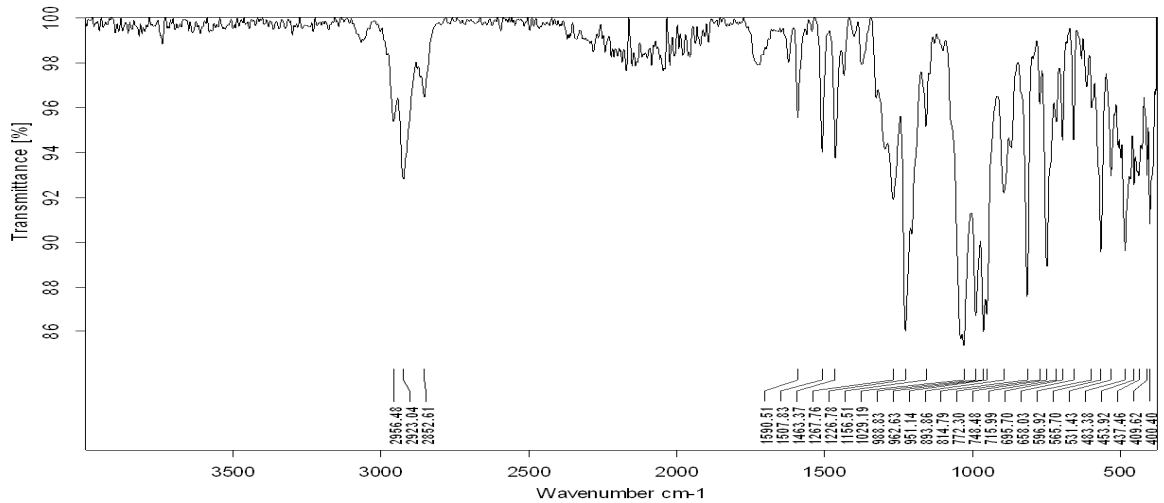
5.3.16 (*R*)-4-Chlorodinaphtho[2,1-d:1',2'-f][1,3,2]dioxaphosphepine 4-oxide (18)







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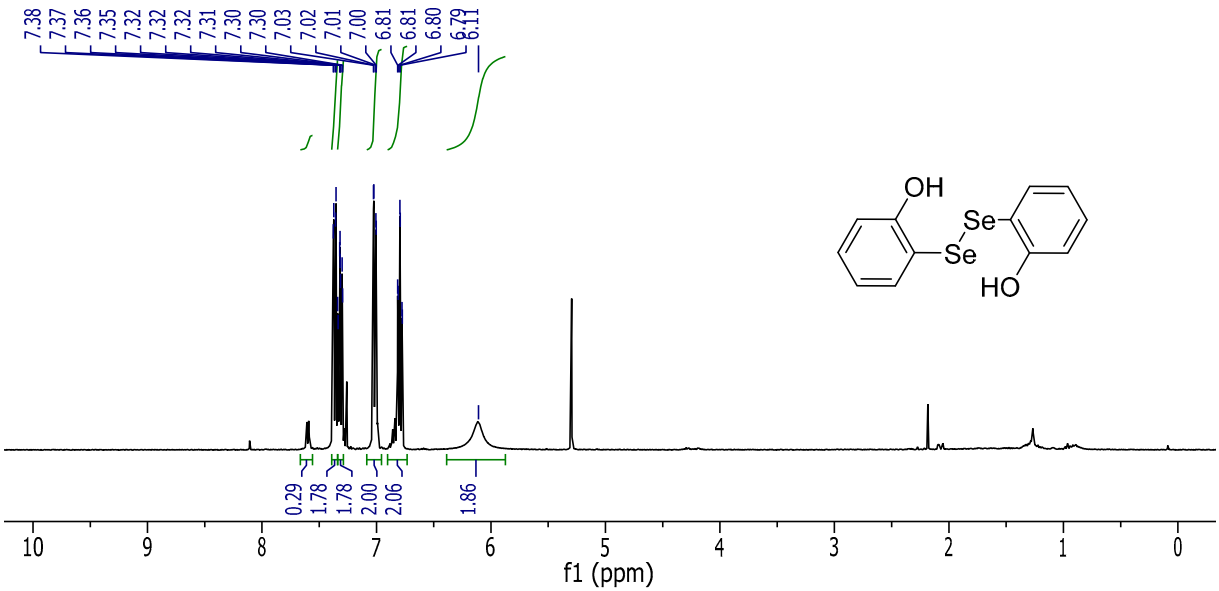
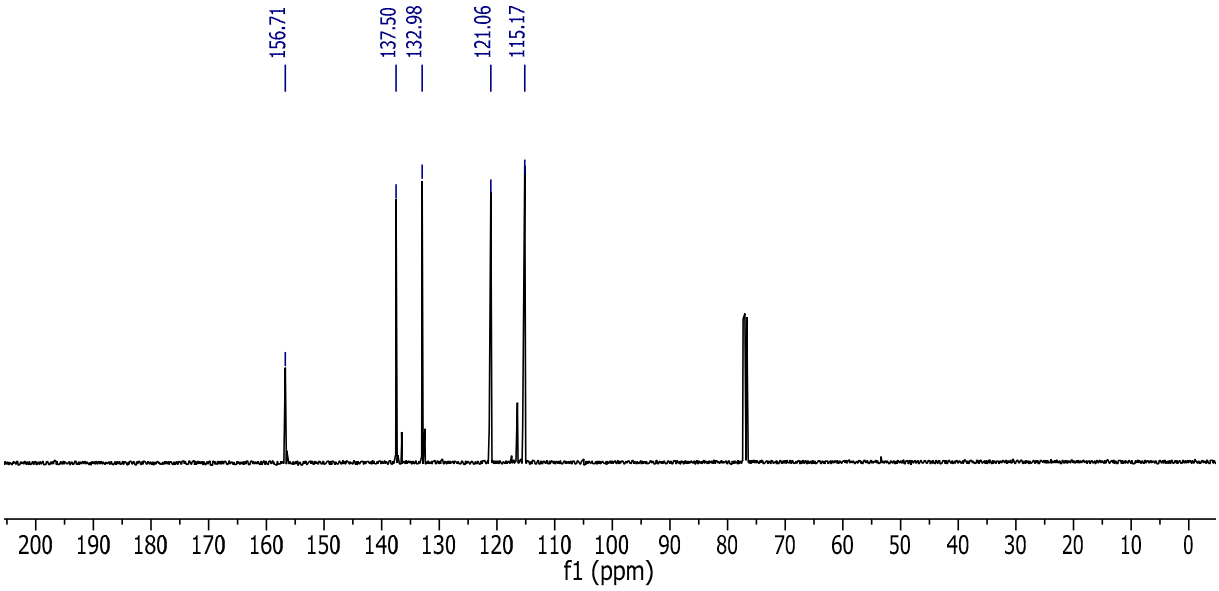
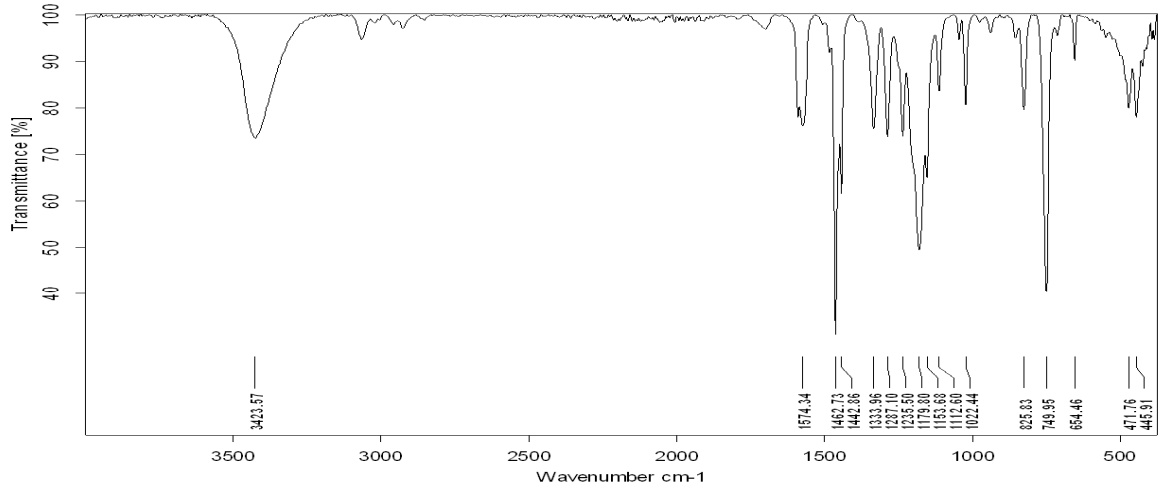
5.3.17 2,2'-Diphenol diselenide (19)







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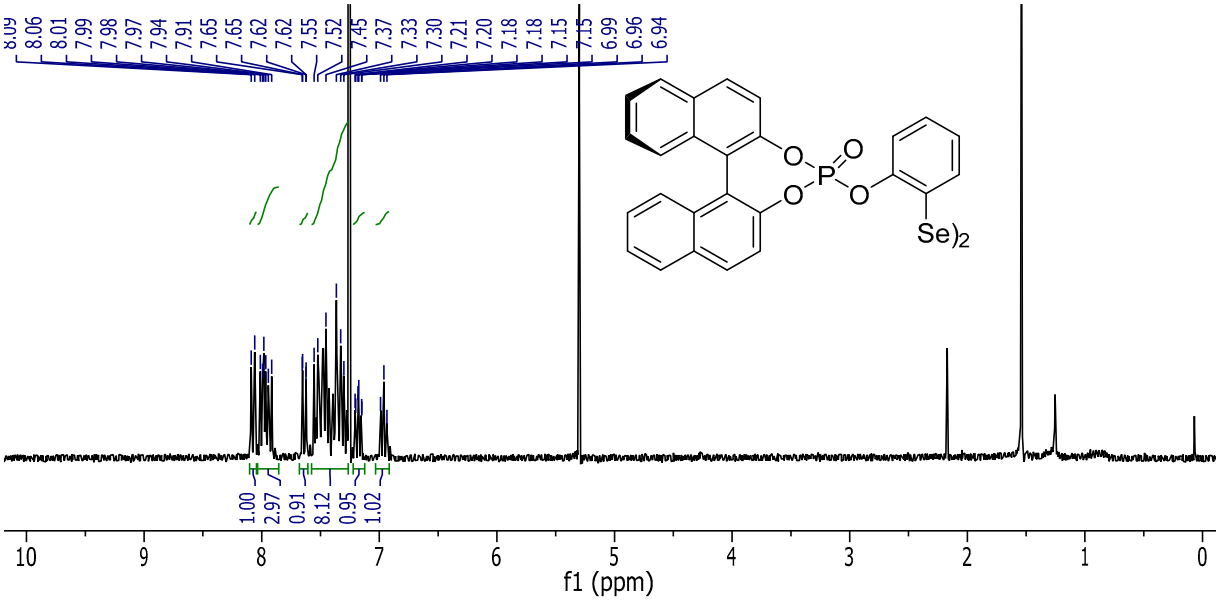
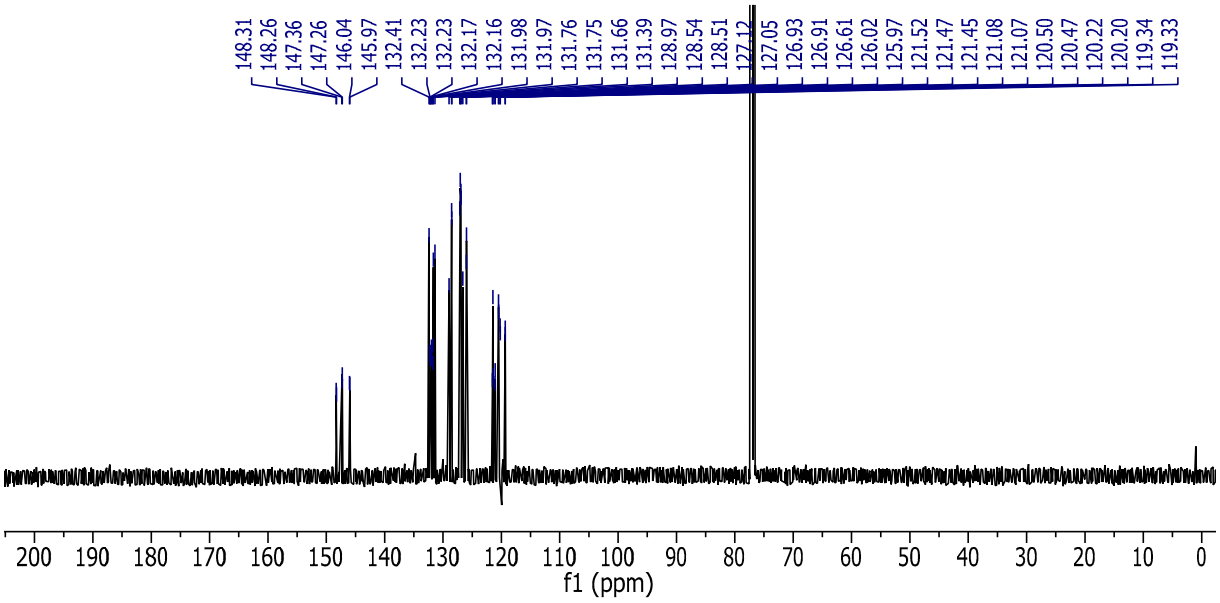
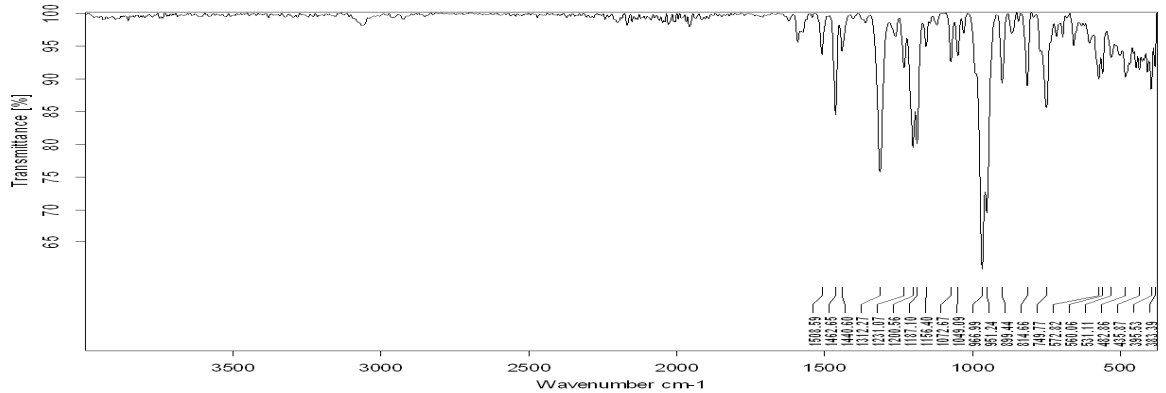
5.3.18 (*R*)-4,4'-((Diseleniddiylbis(2,1-phenylene))bis(oxy))bis-(dinaphtho-[2,1-d:1',2'-f][1,3,2]dioxaphosphepine 4-oxid) (20)







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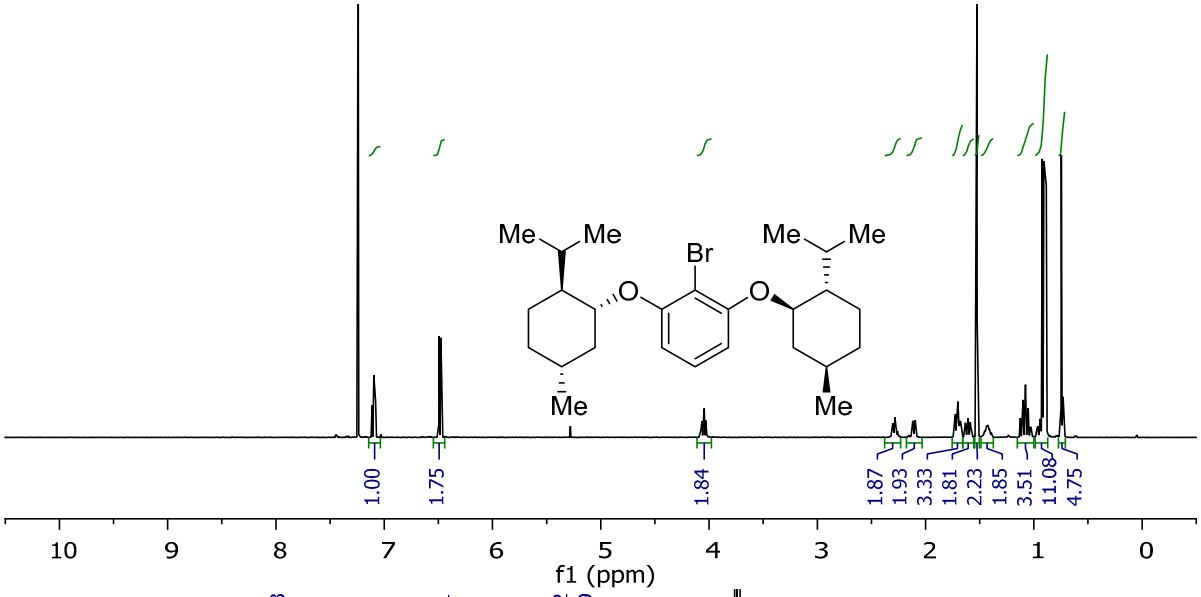
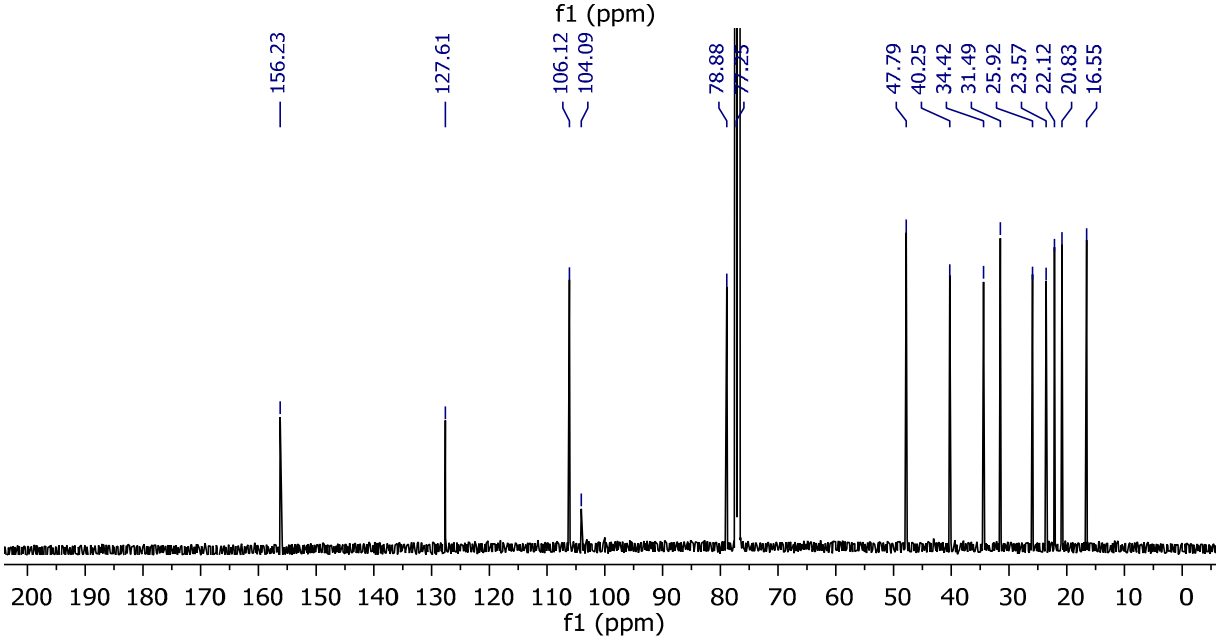
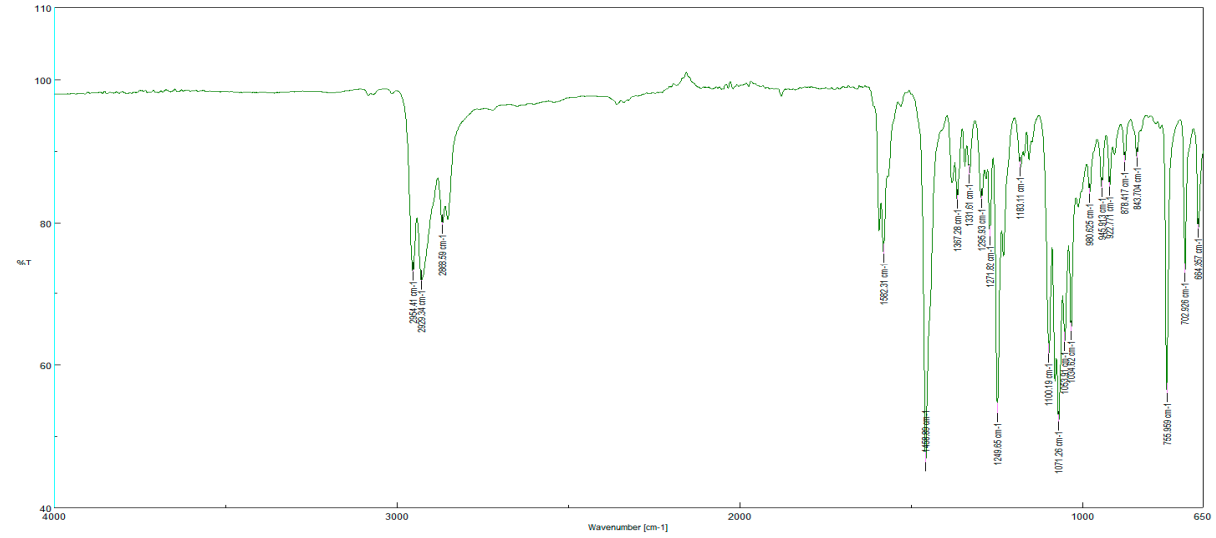
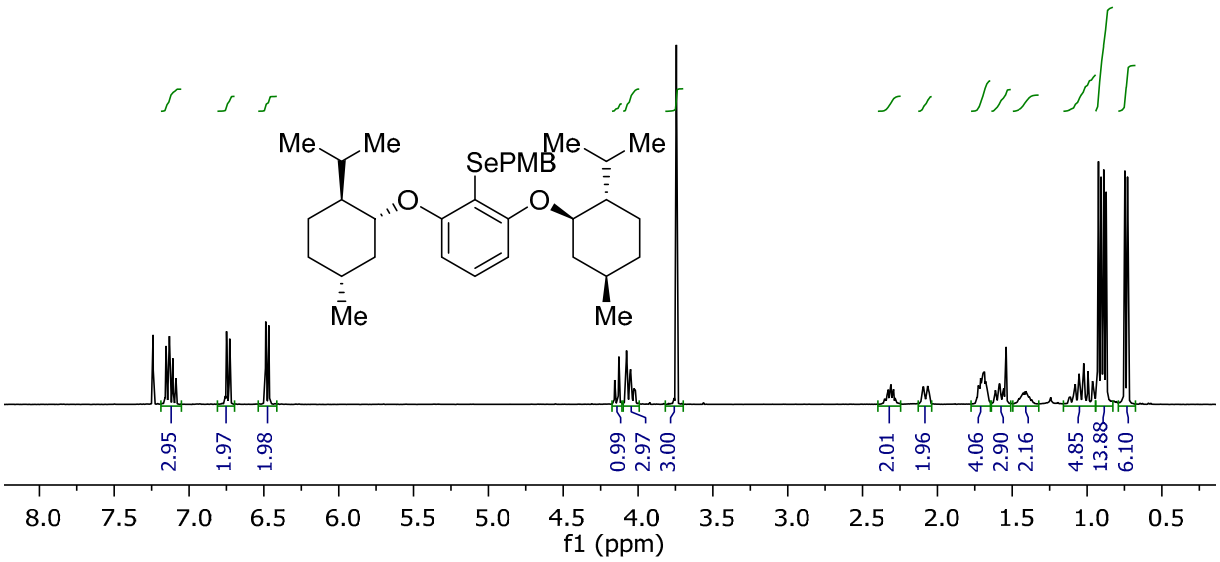
5.3.19 (1*S*,1'*S*,2*R*,2'*R*,4*R*,4'*R*)-2,2'-((2-bromo-1,3-phenylene)bis(oxy))bis(1-isopropyl-4-methylcyclohexane) (13a)

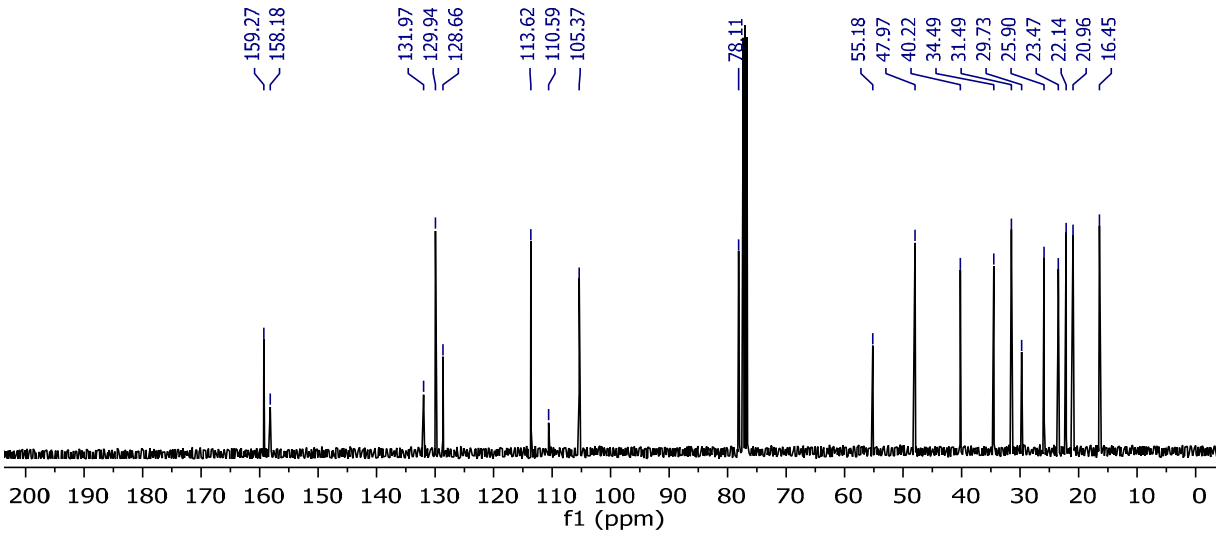


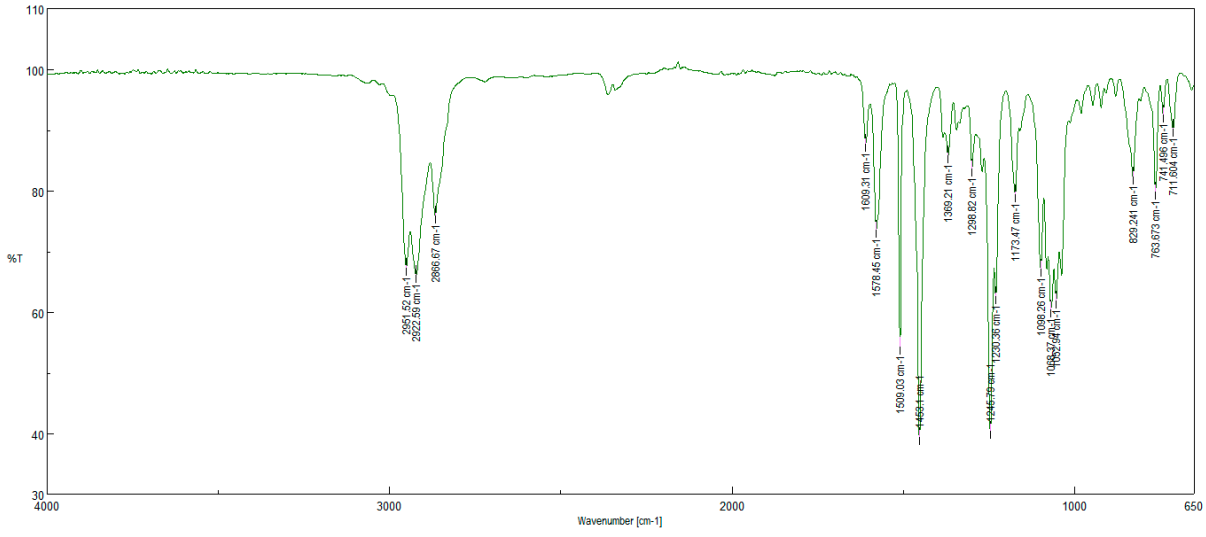




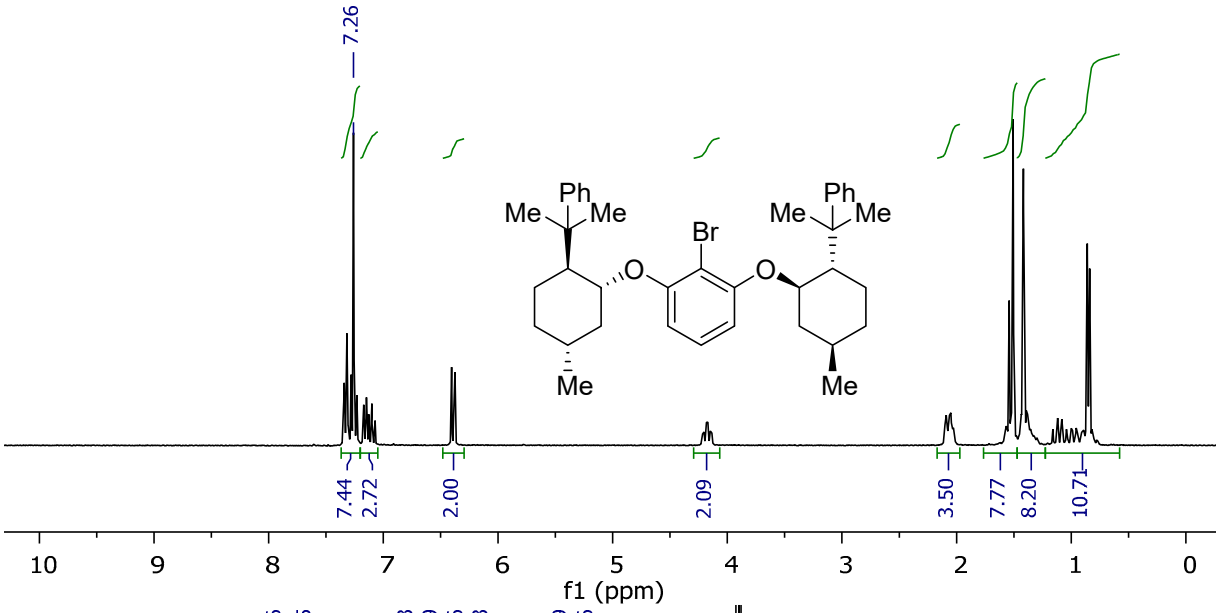
5.3.20 Bis-2,6-bis(((1R,2S,5R)-2-isopropyl-5-methylcyclohexyl)oxy)benzene diselenide (14a)

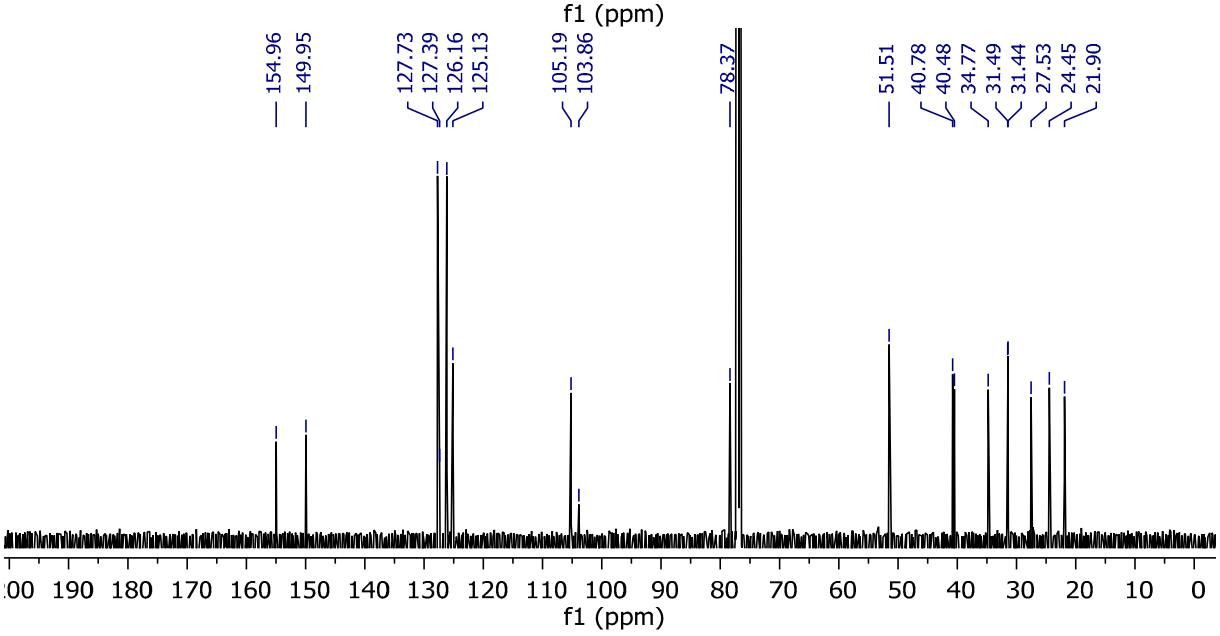


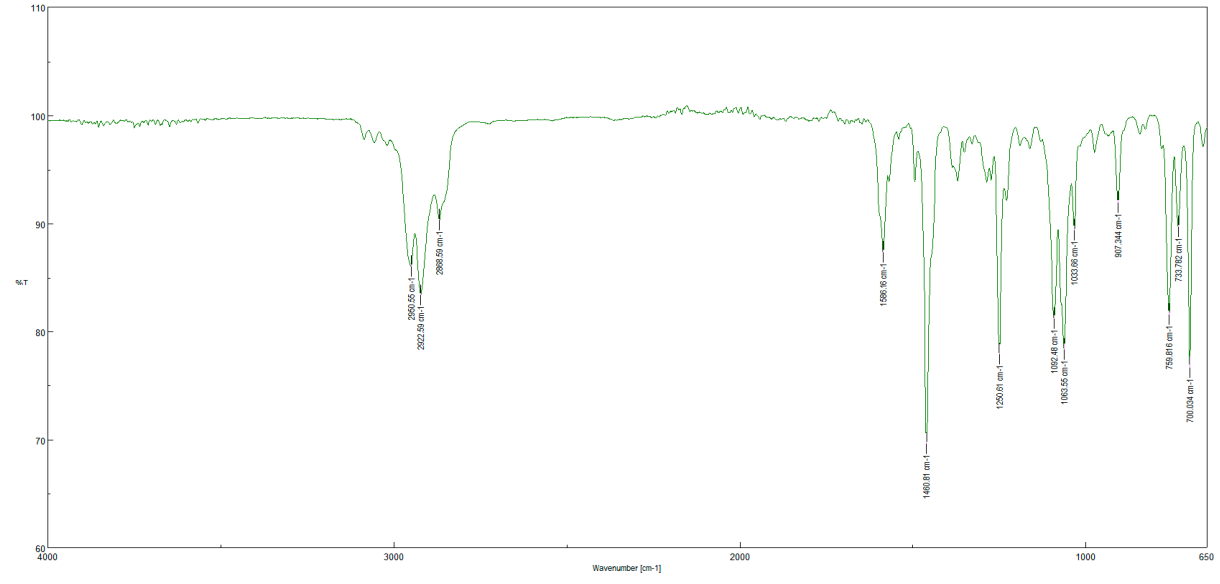




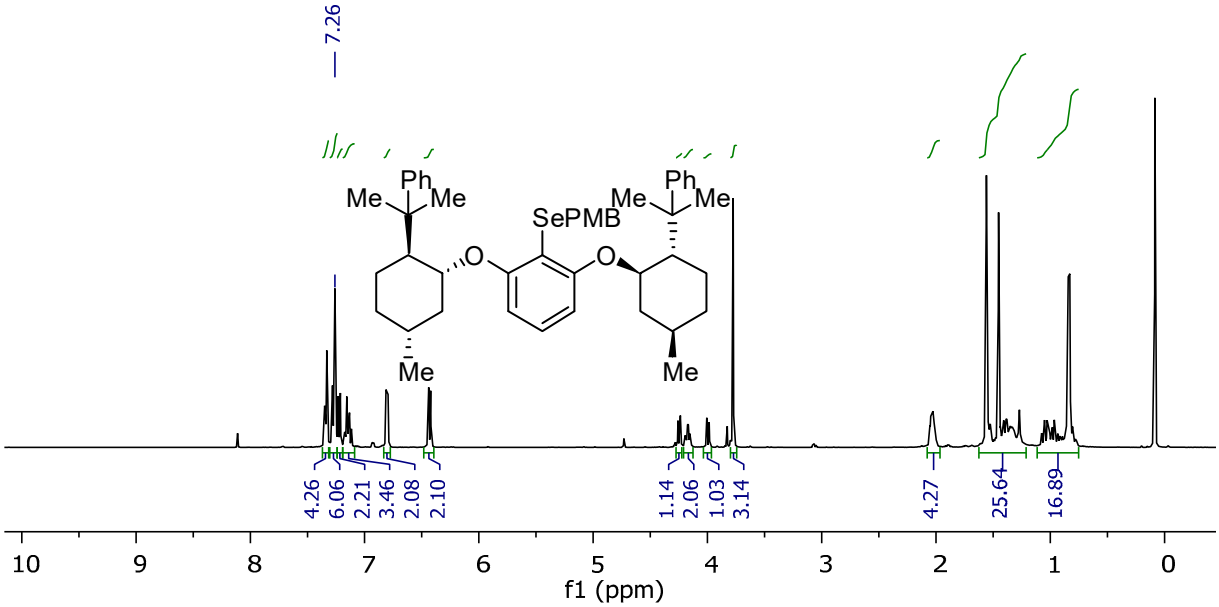
5.3.21 (((1*S*,1'*S*,2*R*,2'*R*,4*R*,4'*R*)-((2-bromo-1,3-phenylene)bis(oxy))bis(4-methylcyclohexane-2,1-diyl))bis(propane-2,2-diyl))dibenzene (13b)

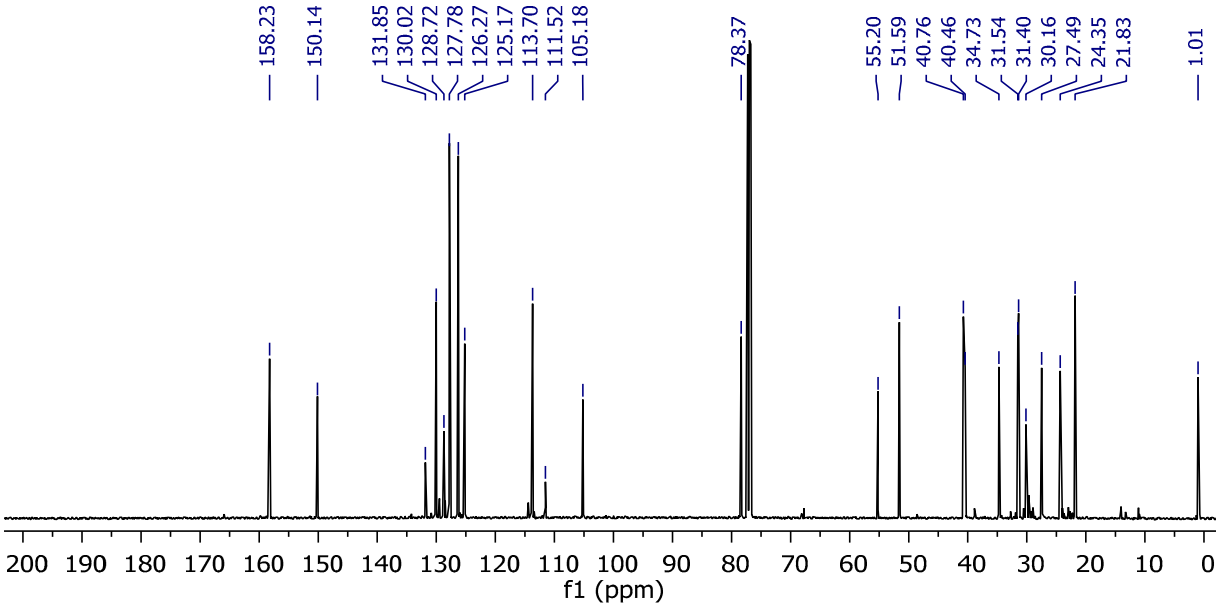


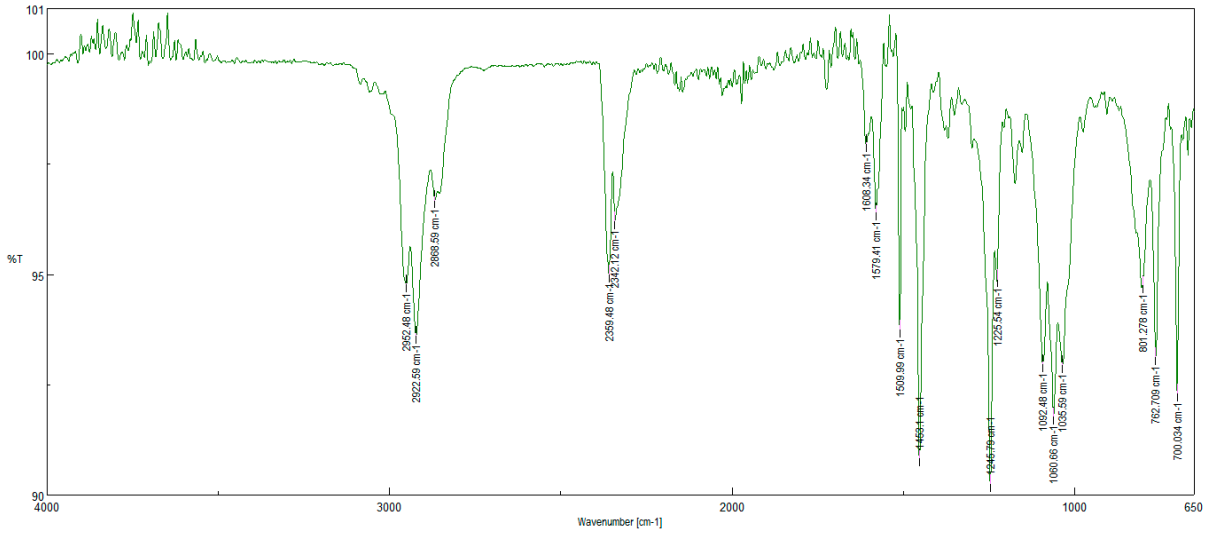




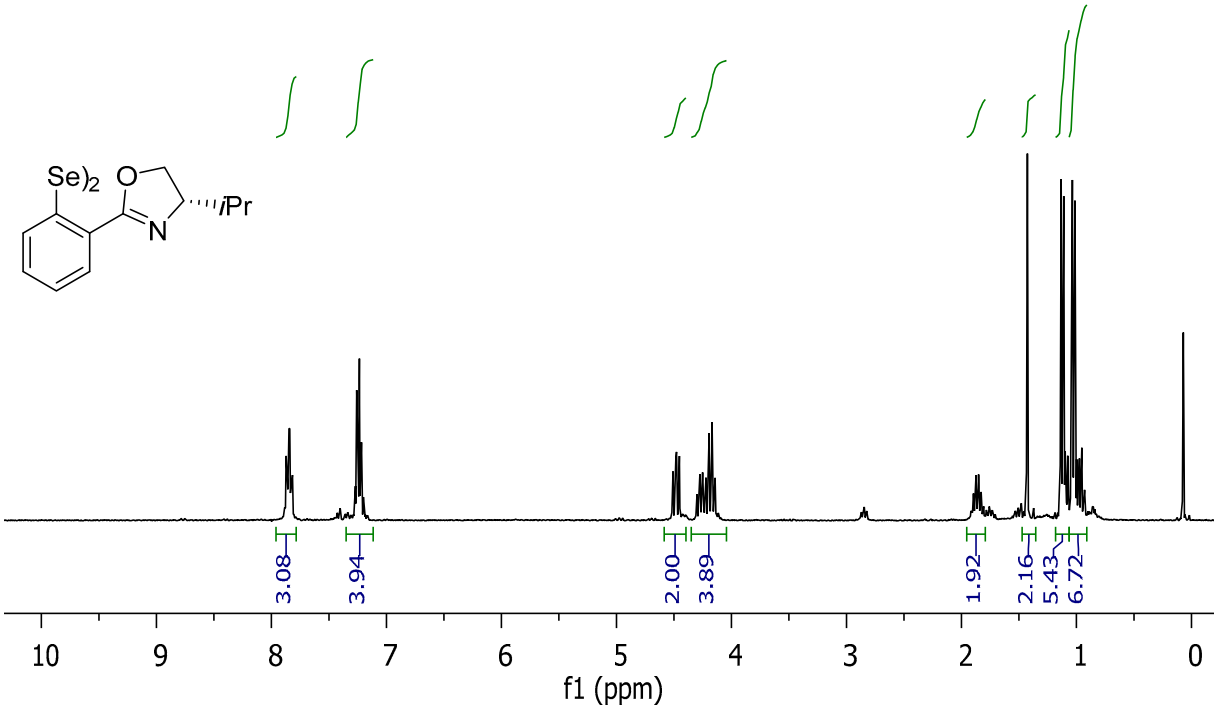
5.3.22 (2,6-bis(((1R,2S,5R)-5-methyl-2-(2-phenylpropan-2-yl)cyclohexyl)oxy)phenyl)(4-methoxybenzyl)selane (14b)

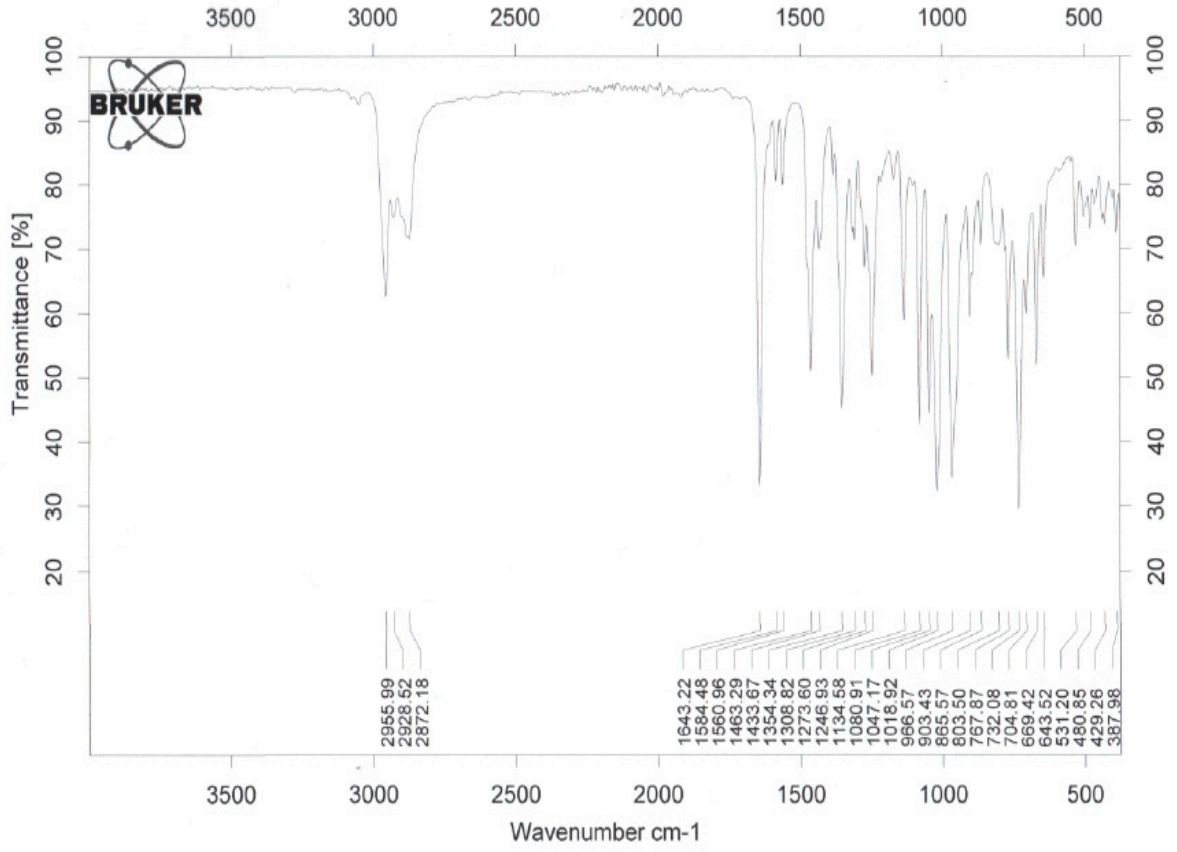



158.23
150.14
131.85
130.02
128.72
127.78
126.27
125.17
113.70
111.52
105.18
78.37
55.20
51.59
40.76
40.46
34.73
31.54
31.40
30.16
27.49
24.35
21.83
1.01
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

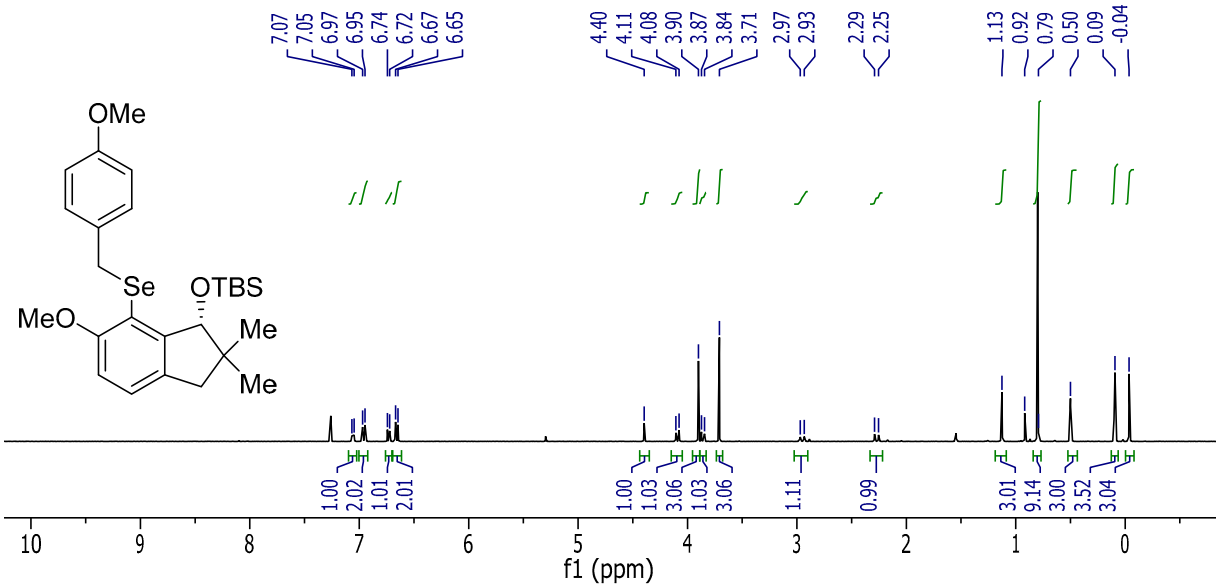

%T
101
100
95
90
4000
3000
2000
1000
650
Wavenumber [cm-1]
2962.48 cm-1
2922.59 cm-1
2868.59 cm-1
2359.48 cm-1
2342.12 cm-1
1608.34 cm-1
1579.41 cm-1
1509.99 cm-1
1463.1 cm-1
1245.79 cm-1
1225.54 cm-1
1092.48 cm-1
1060.66 cm-1
1055.59 cm-1
801.278 cm-1
762.709 cm-1
700.034 cm-1

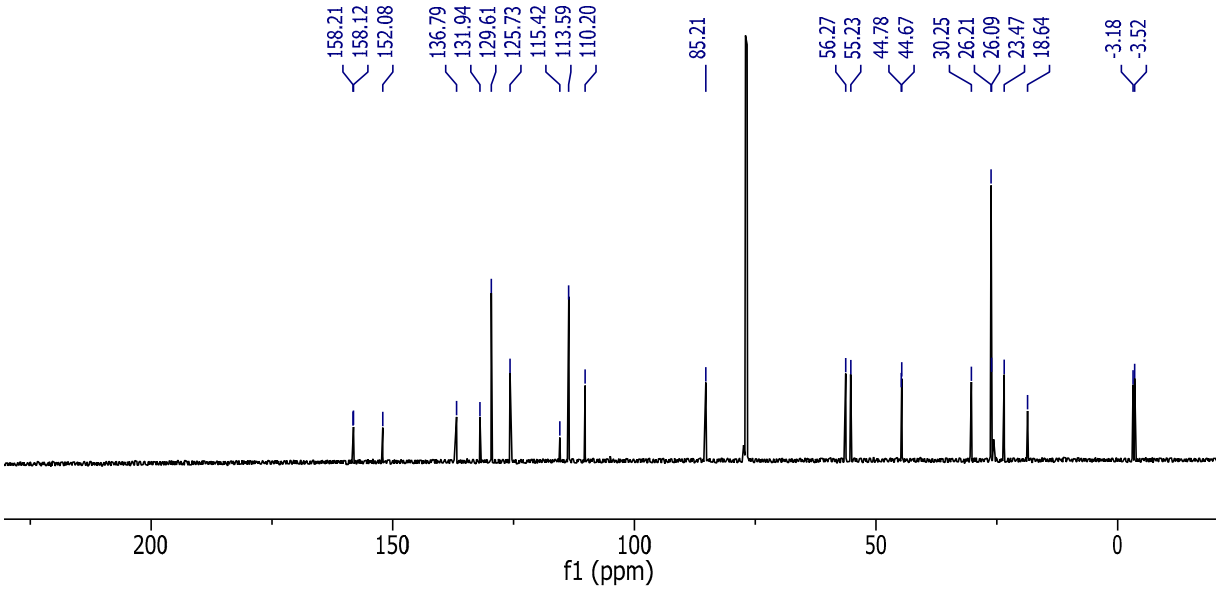
5.3.23 1,2-Bis(2-((*S*)-4-isopropyl-4,5-dihydrooxazol-2-yl)phenyl)diselane(4)

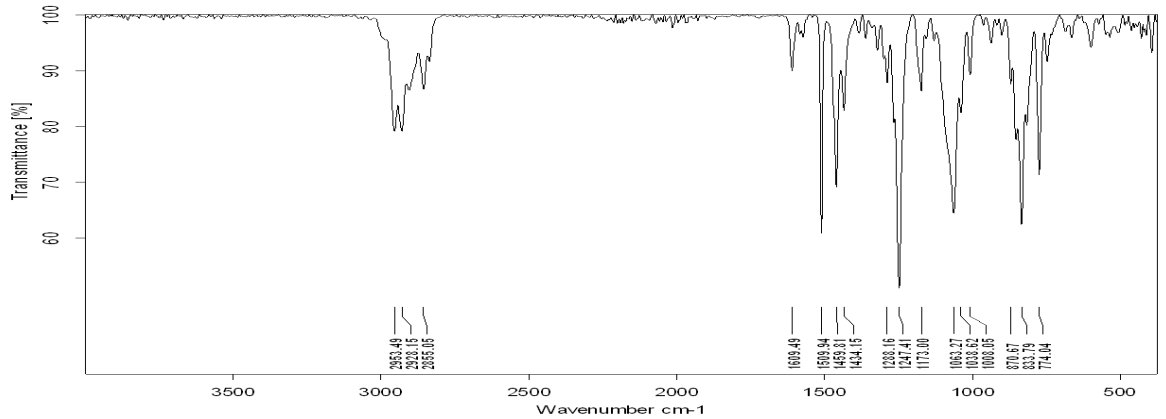




5.3.24 (*R*)-tert-butyl((6-methoxy-7-((4-methoxybenzyl)selanyl)-2,2-dimethyl-2,3-dihydro-1H-inden-1-yl)oxy)dimethylsilane (23)







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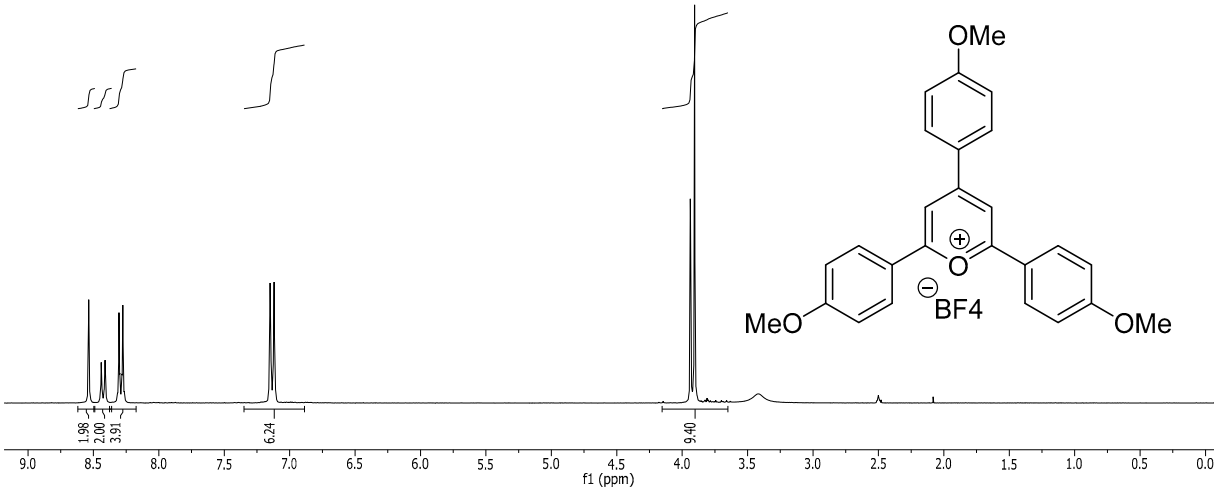
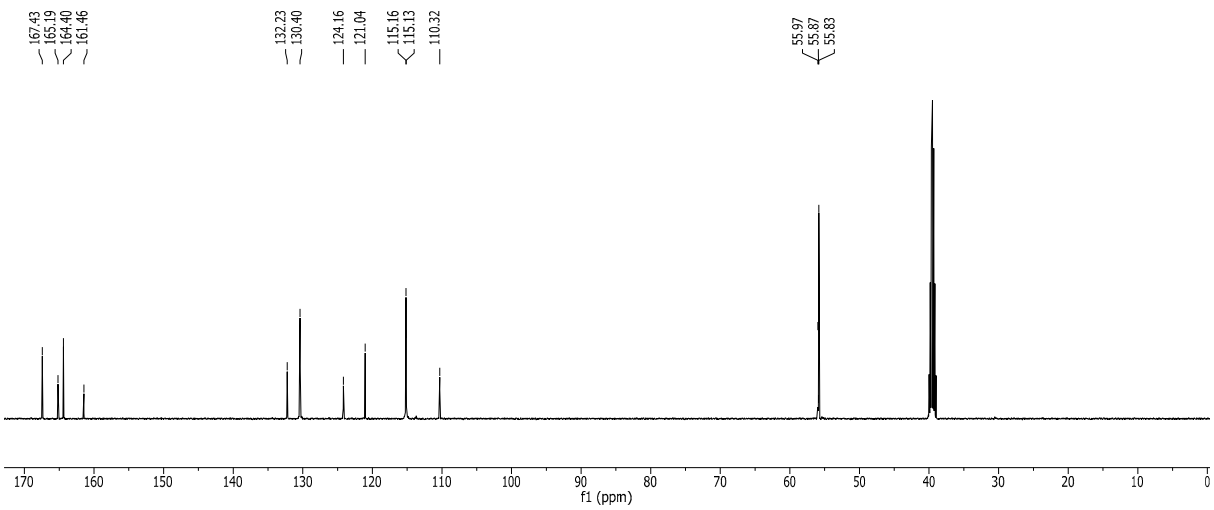
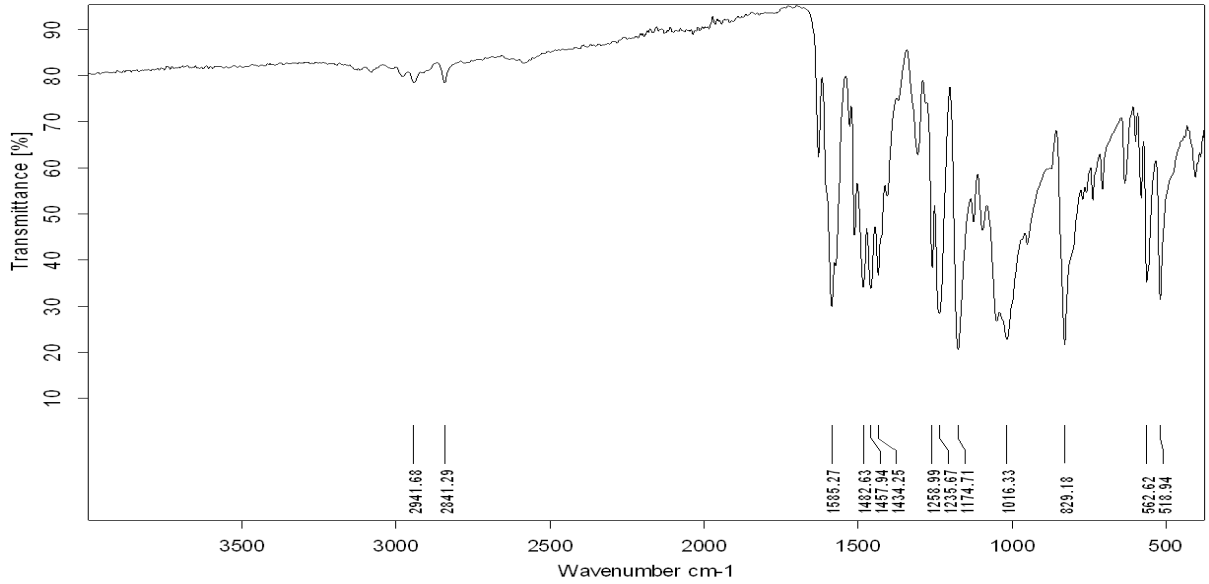
5.4 Photocatalysts

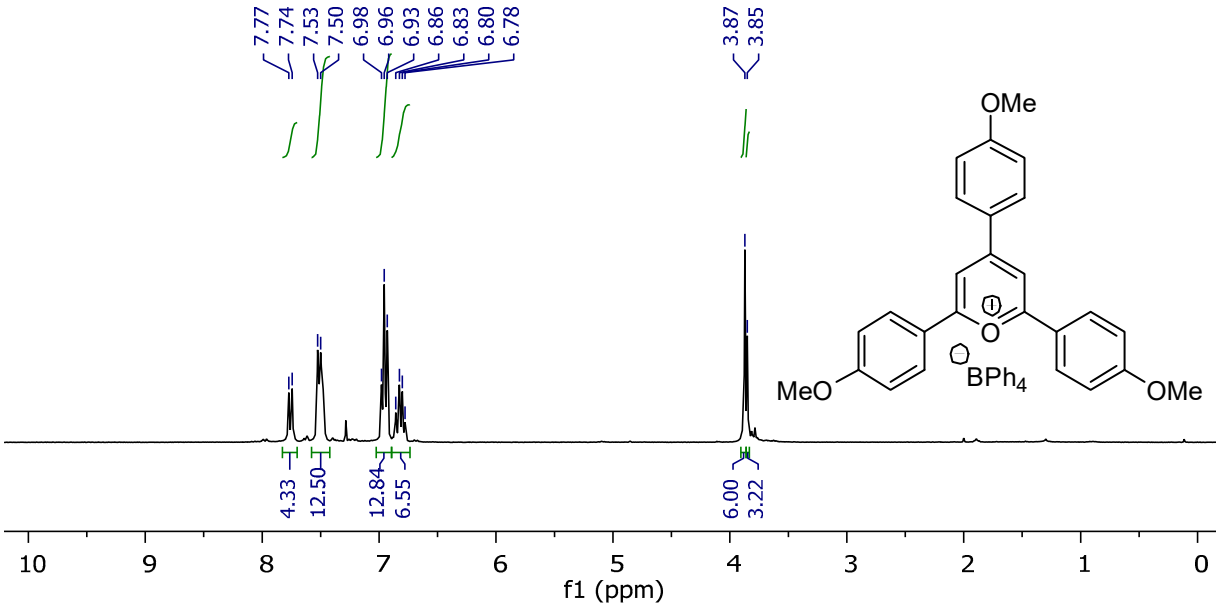
5.4.1 2,4,6-tris(4-methoxyphenyl)pyrylium tetrafluoroborate (TAPT)

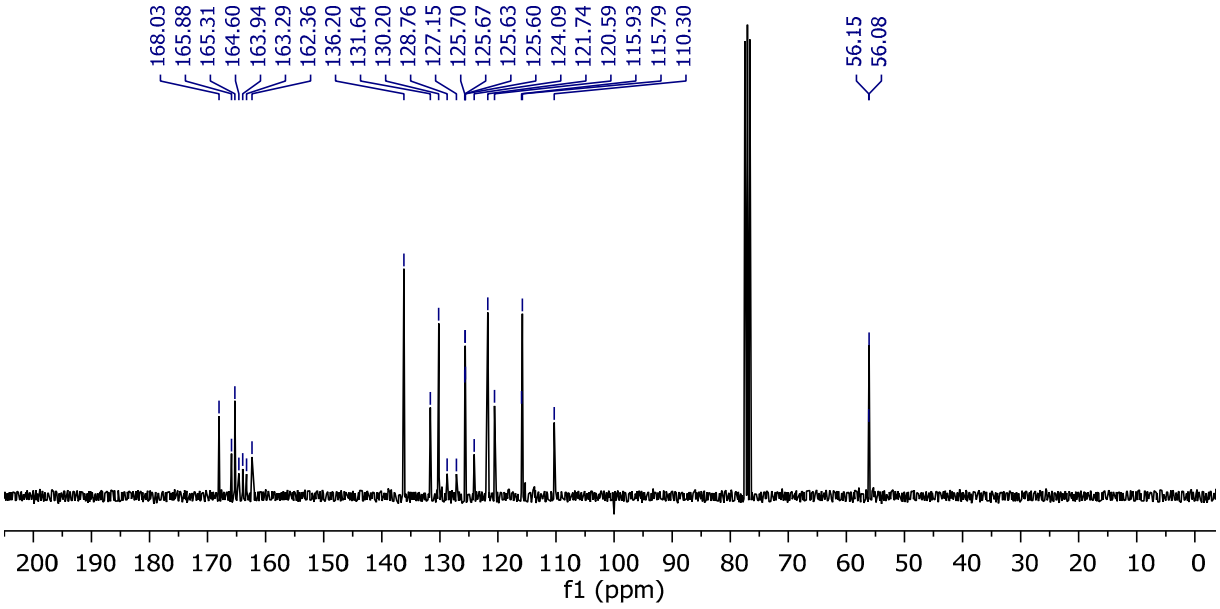


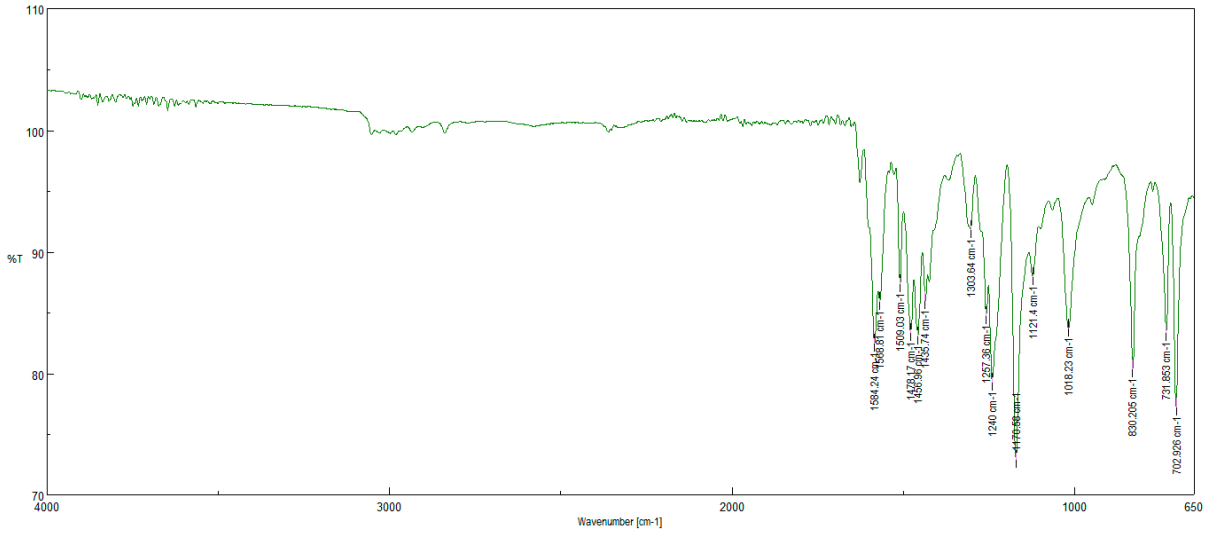




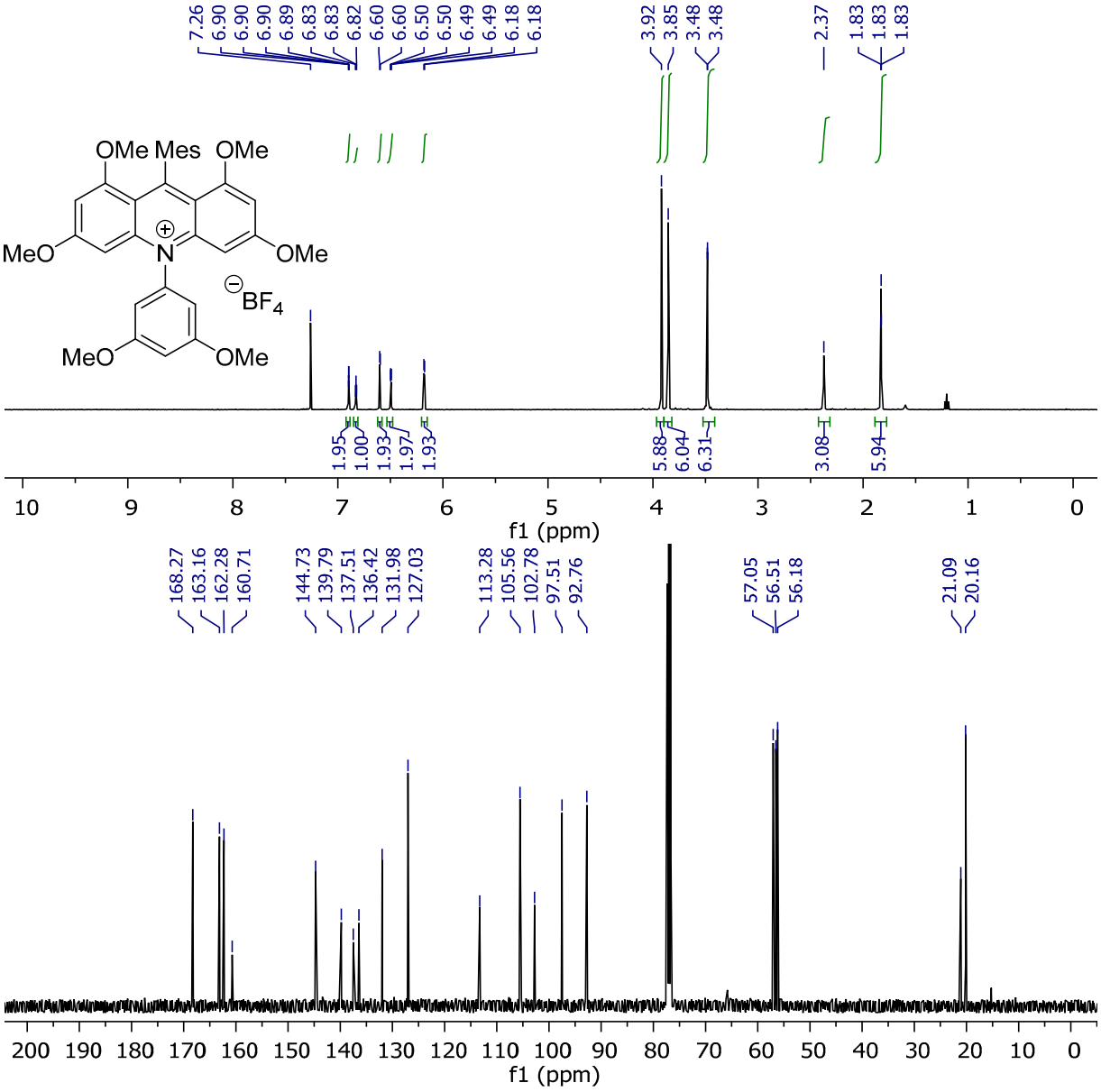
5.4.2 2,4,6-tris(4-methoxyphenyl)pyrylium tetraphenylborate (TABTP)

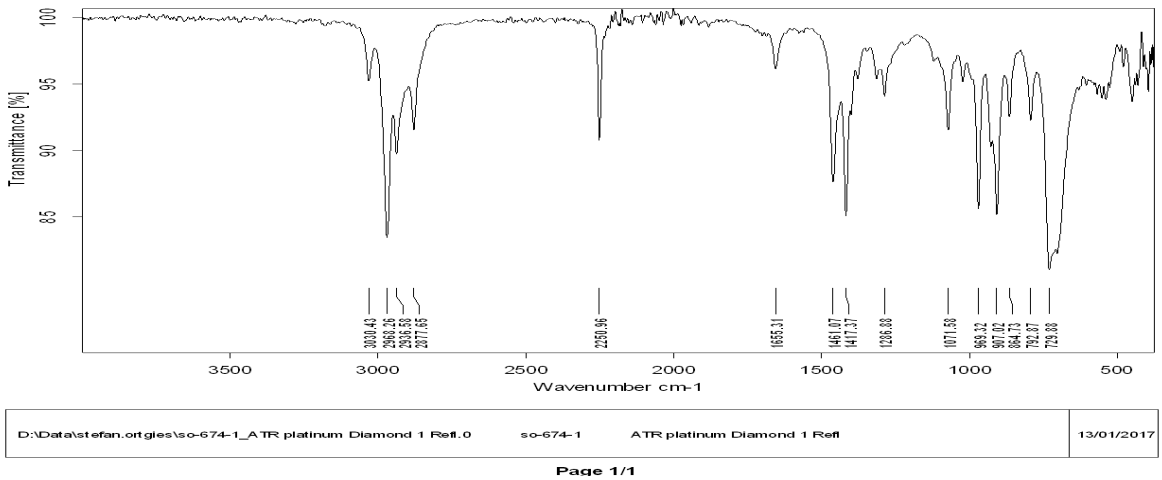





110
100
90
80
70
%T
4000
3000
2000
1000
650
Wavenumber [cm-1]
1584.24 cm-1
1509.03 cm-1
1478.17 cm-1
1456.96 cm-1
1435.74 cm-1
1303.64 cm-1
1257.36 cm-1
1240 cm-1
1121.4 cm-1
1018.23 cm-1
830.205 cm-1
731.853 cm-1
702.926 cm-1

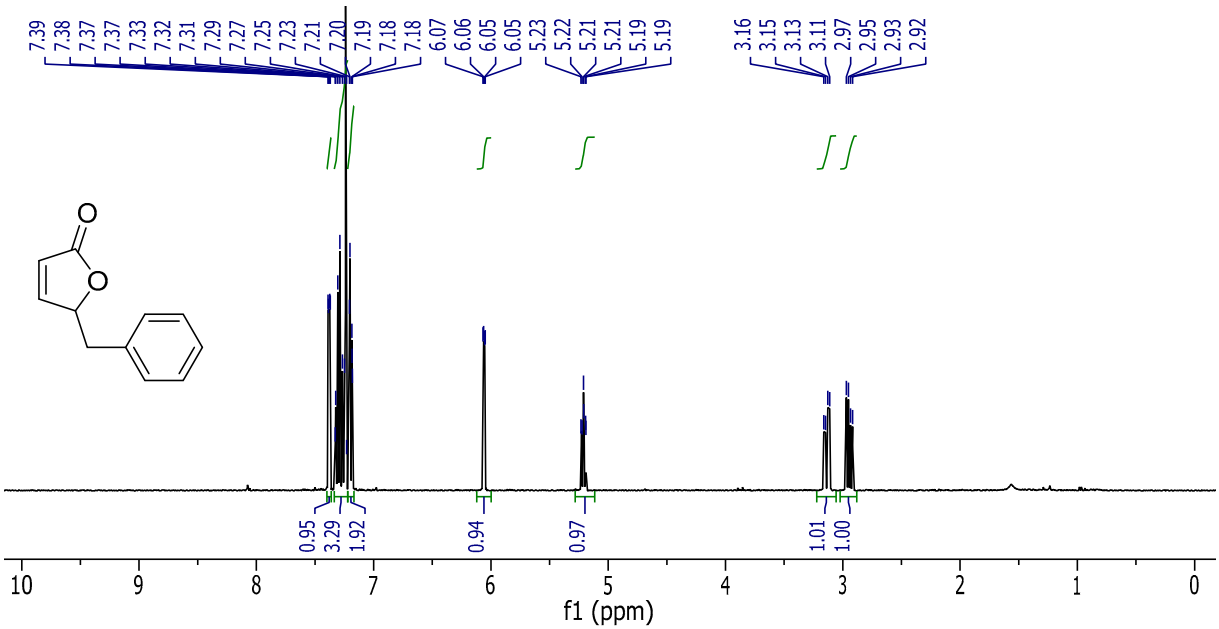
5.4.3 10-(3,5-dimethoxyphenyl)-9-mesityl-1,3,6,8-tetramethoxyacridin-10-ium tetrafluoroborate (DMTA)

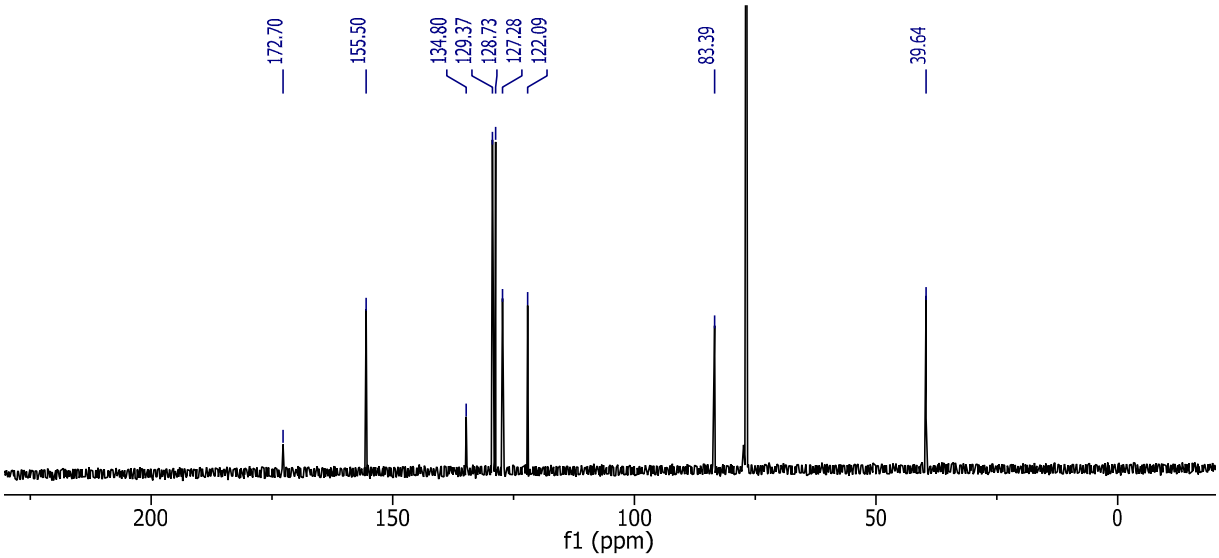


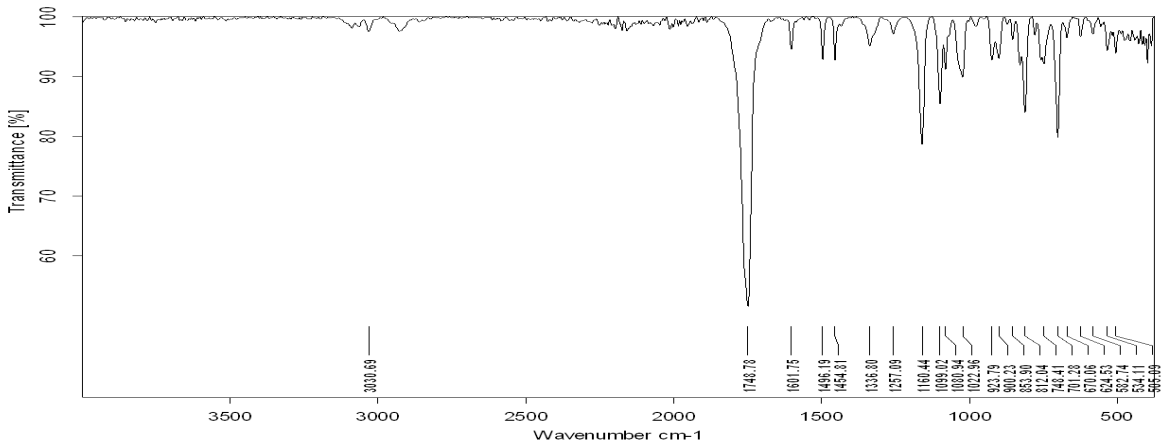


5.5 Asymmetric lactonization

5.5.1 5-Benzylfuran-2(5H)-on (22)







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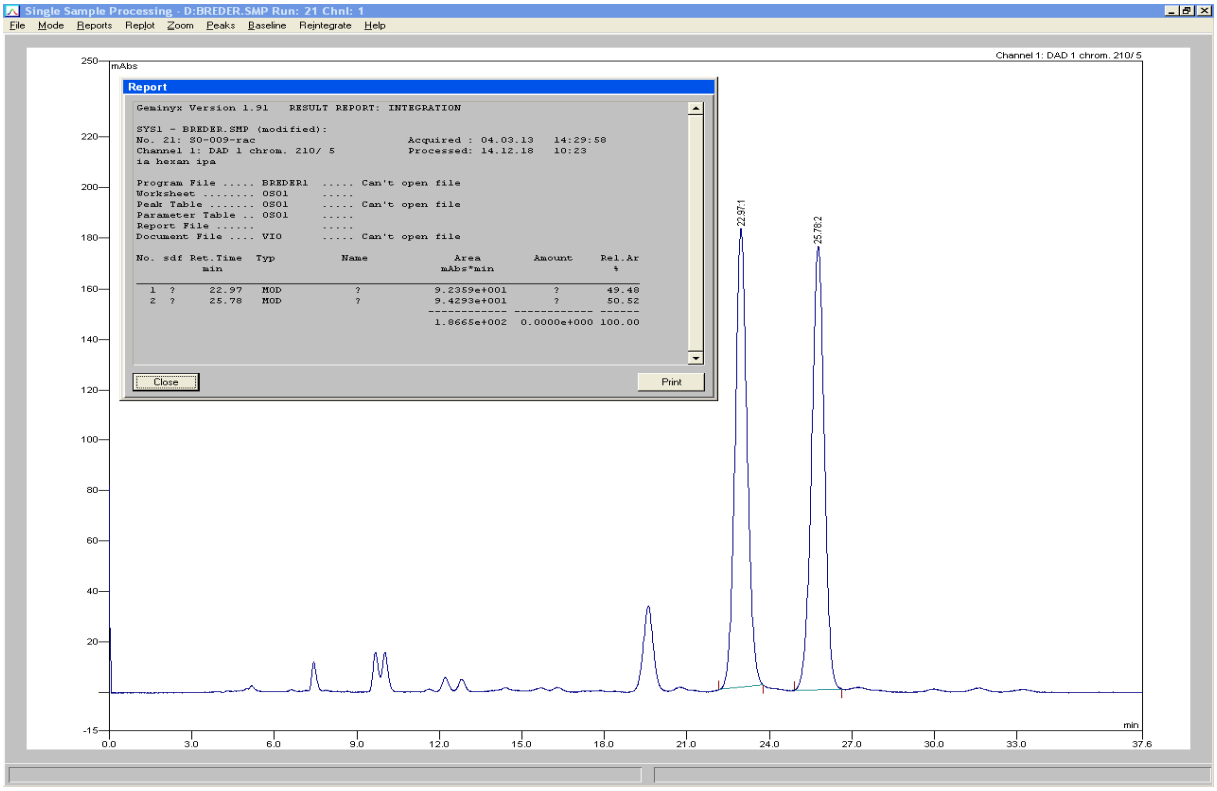
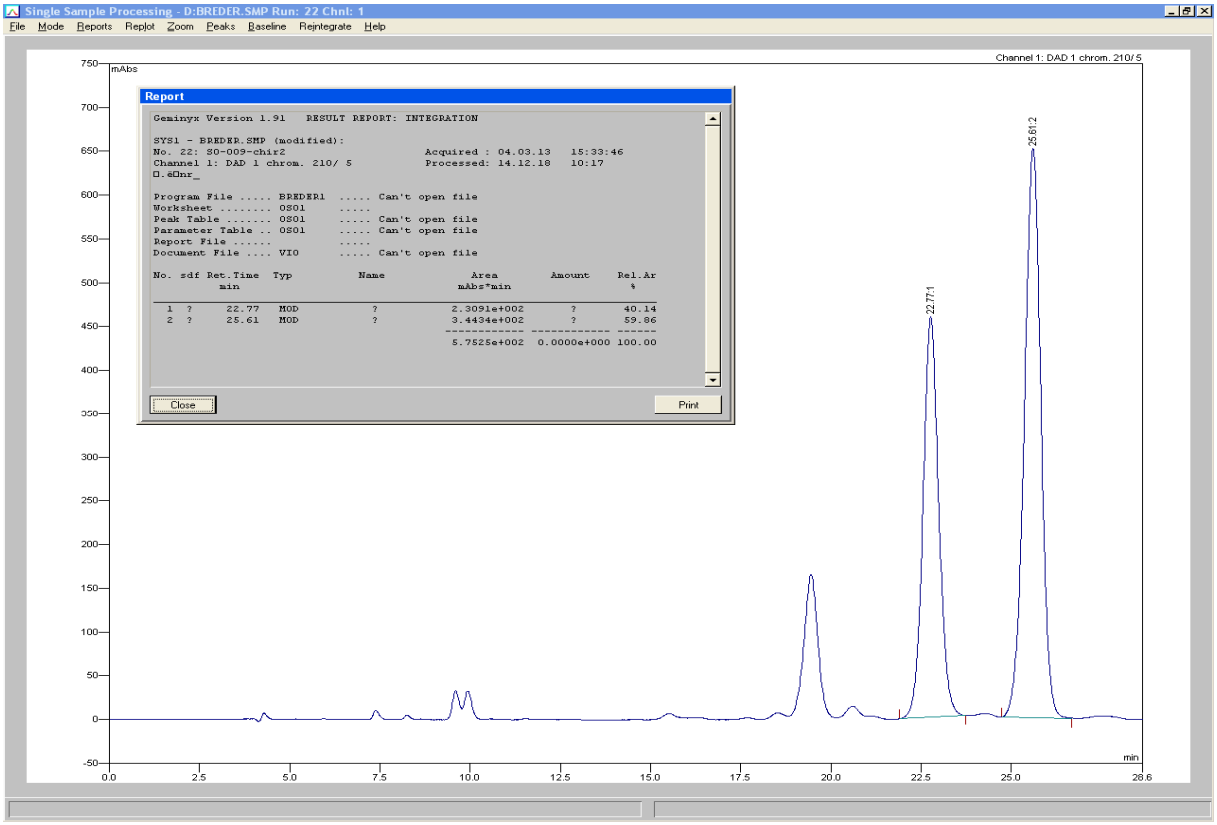
6 HPLC Chromatograms

6.1 Imidation

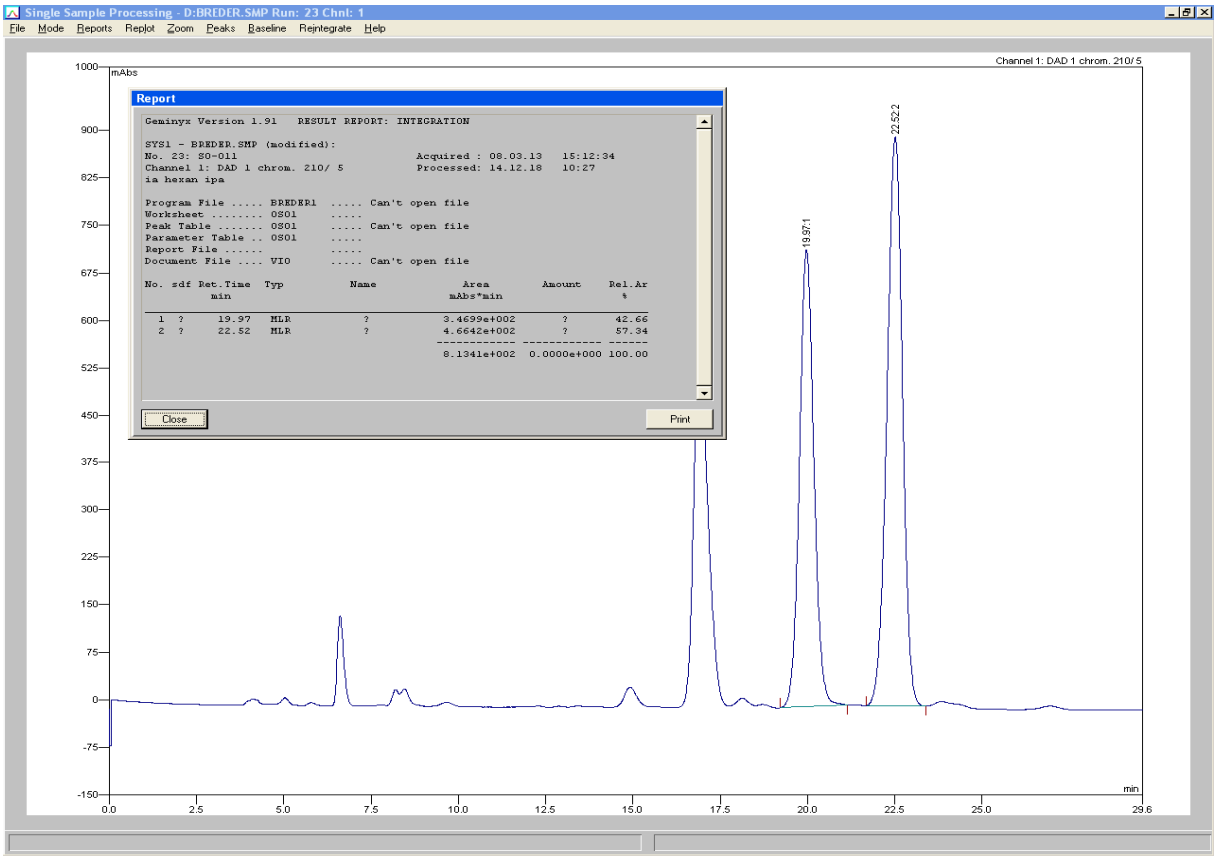
6.1.1 Racemate



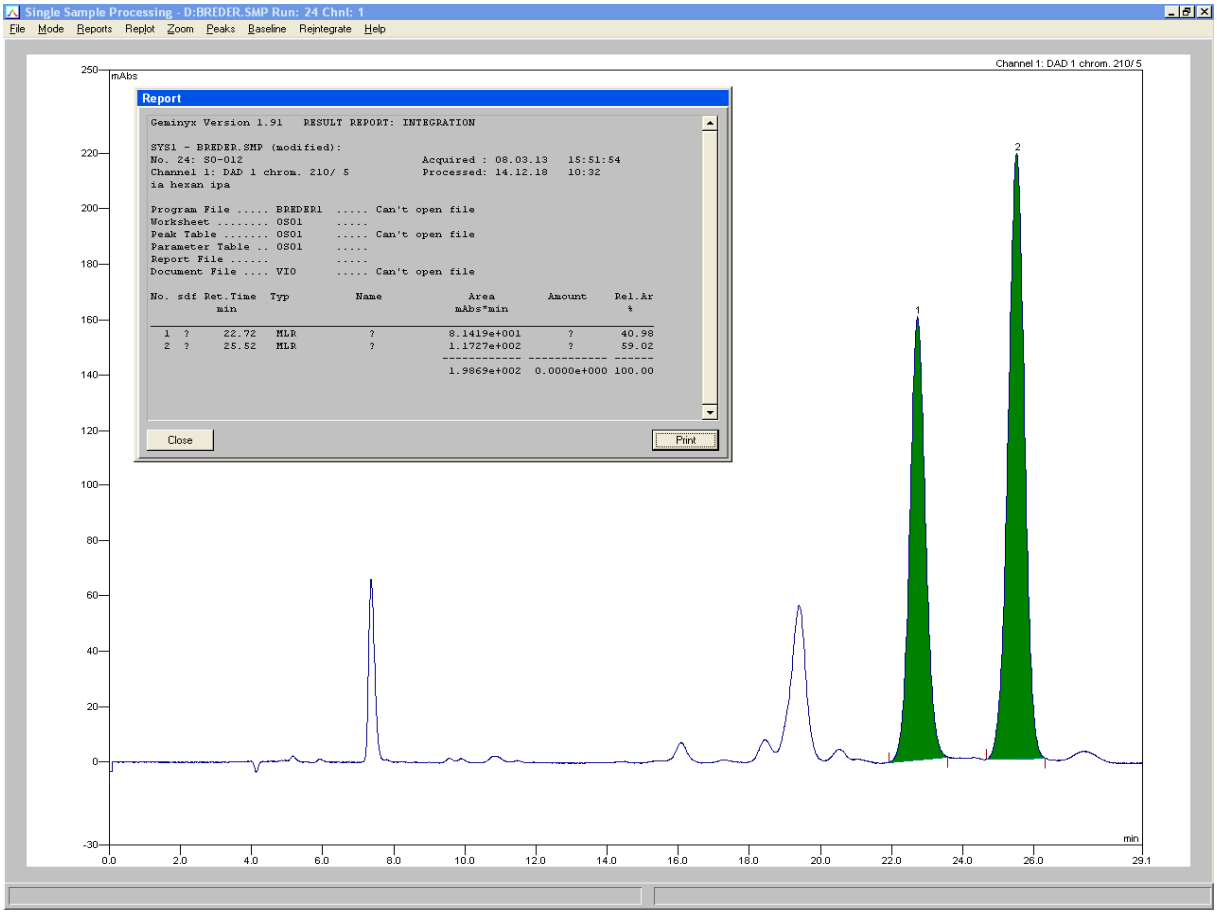
6.1.2 Entry 1



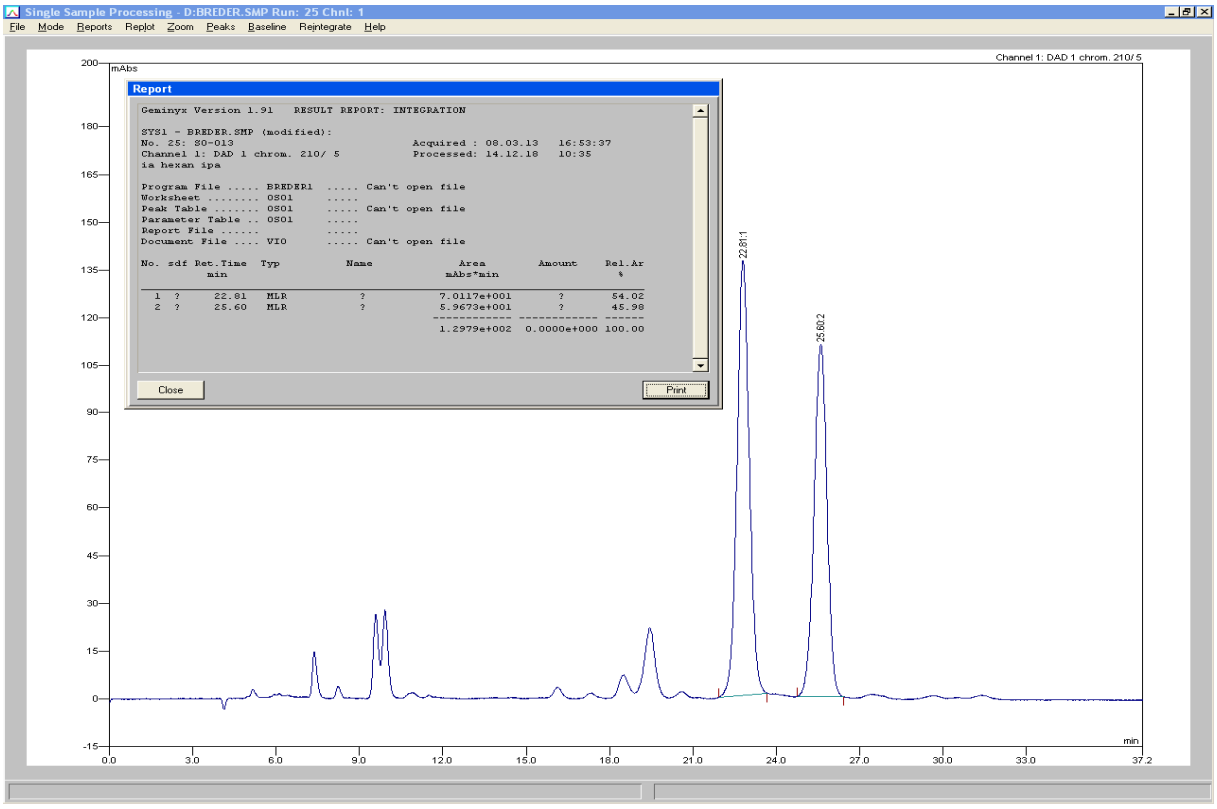
6.1.3 Entry 2



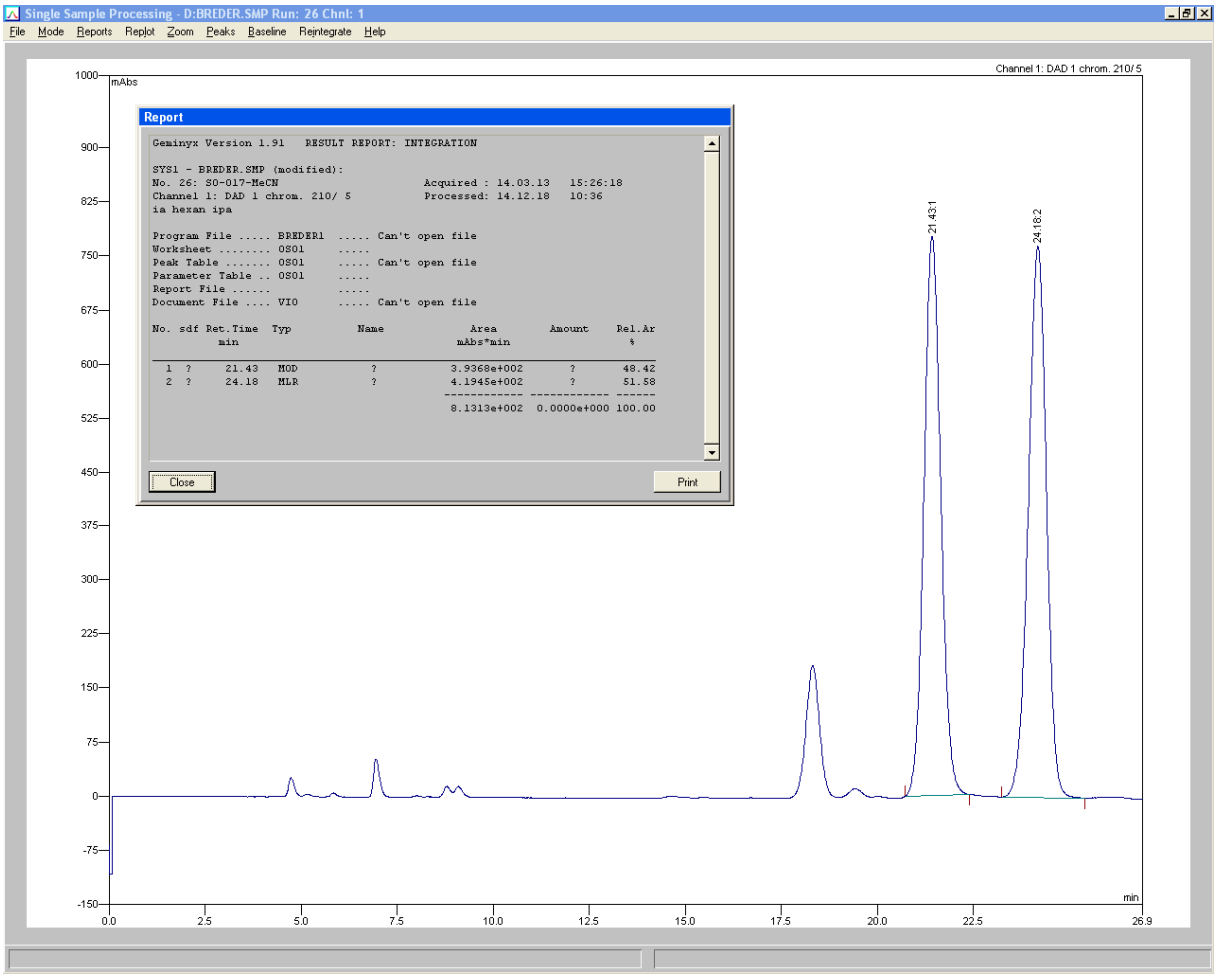
6.1.4 Entry 3



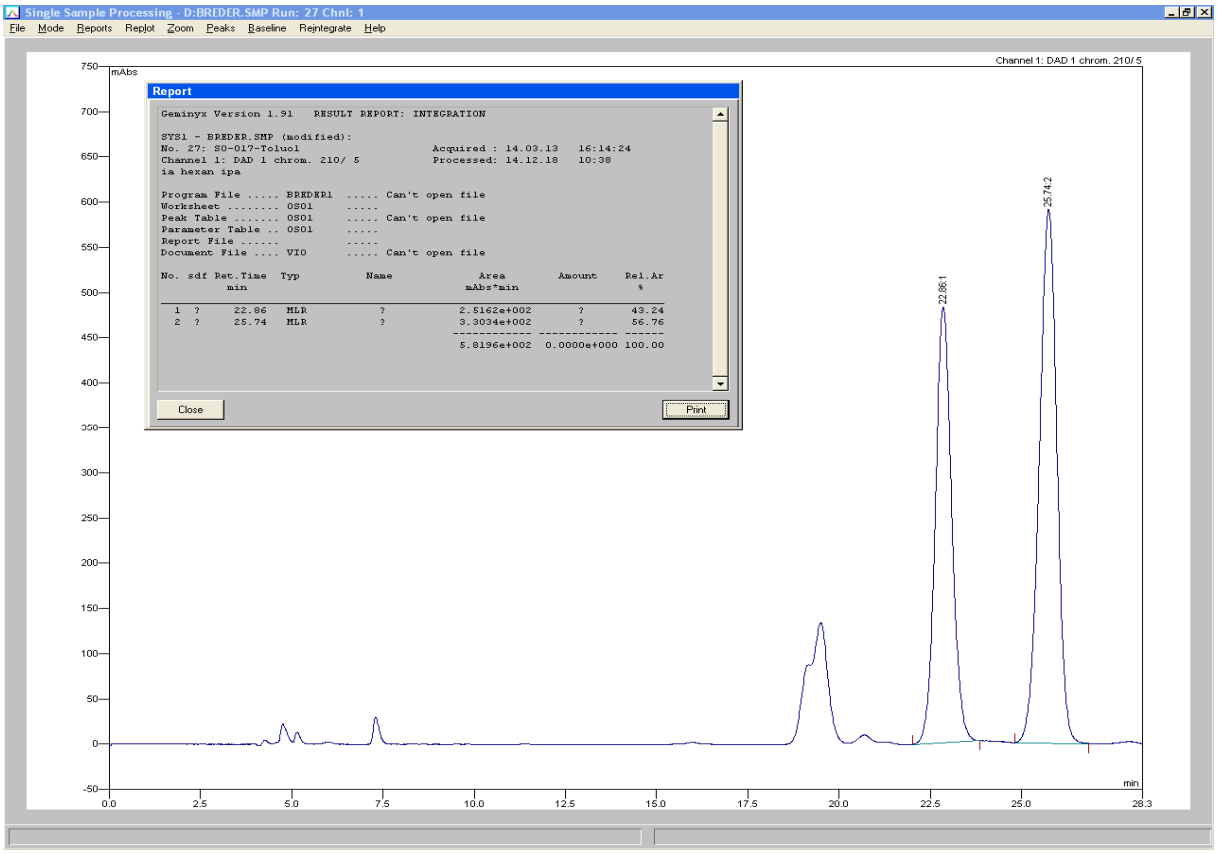
6.1.5 Entry4



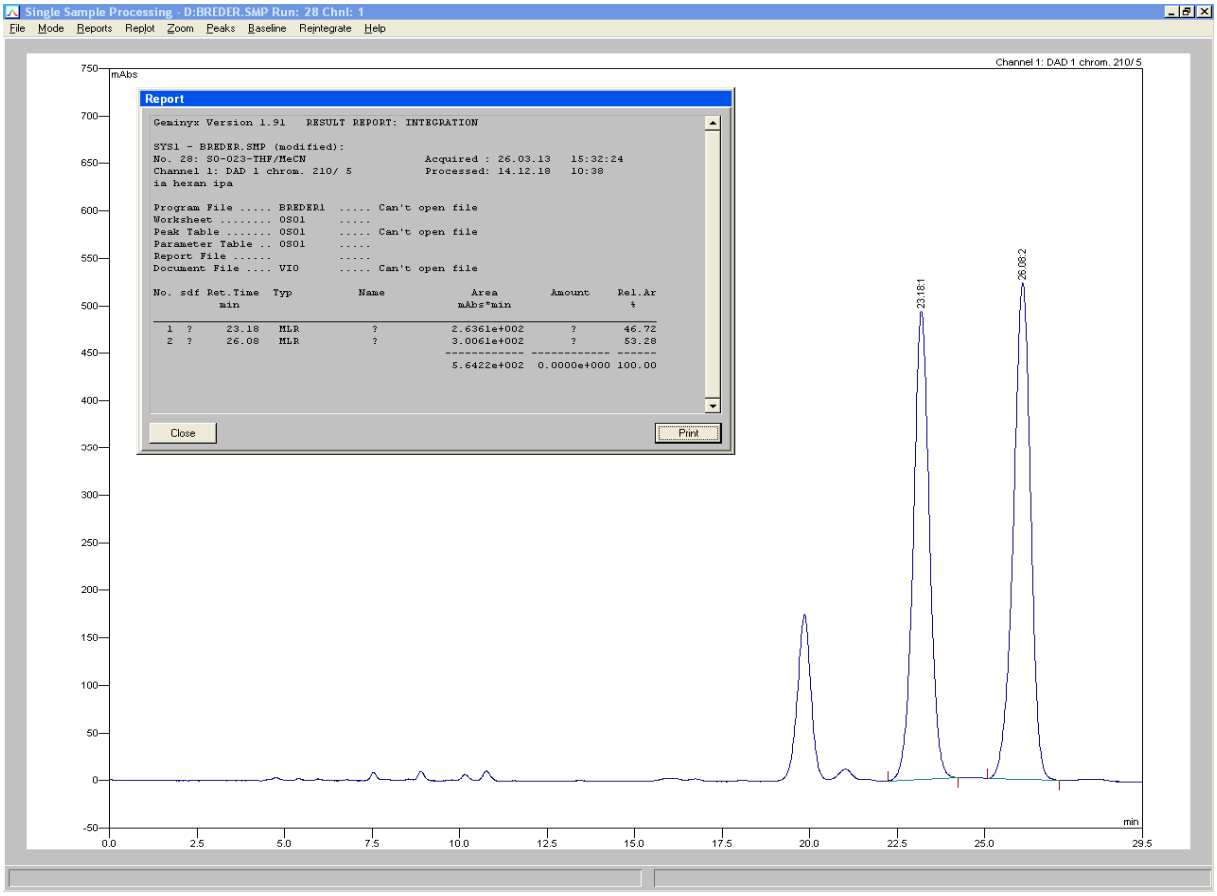
6.1.6 Entry 5



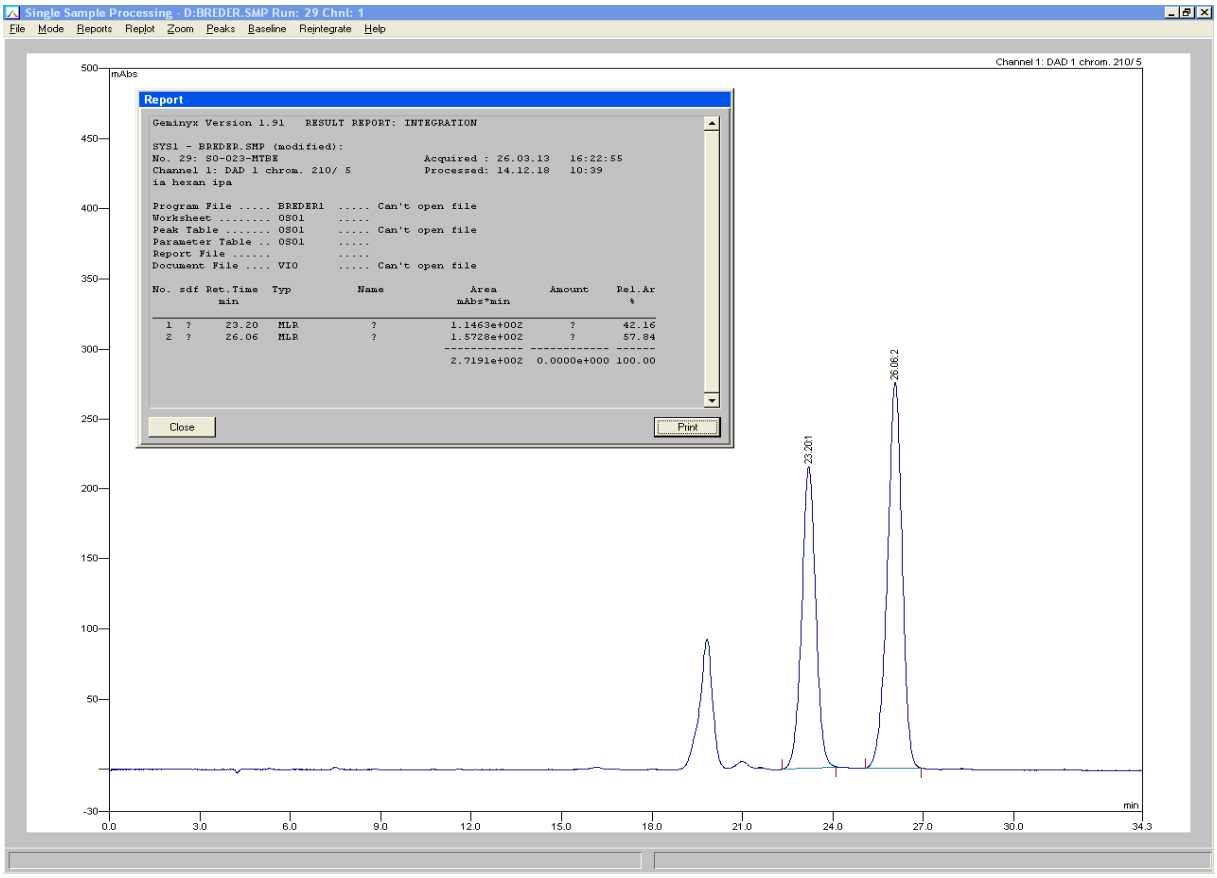
6.1.7 Entry 6



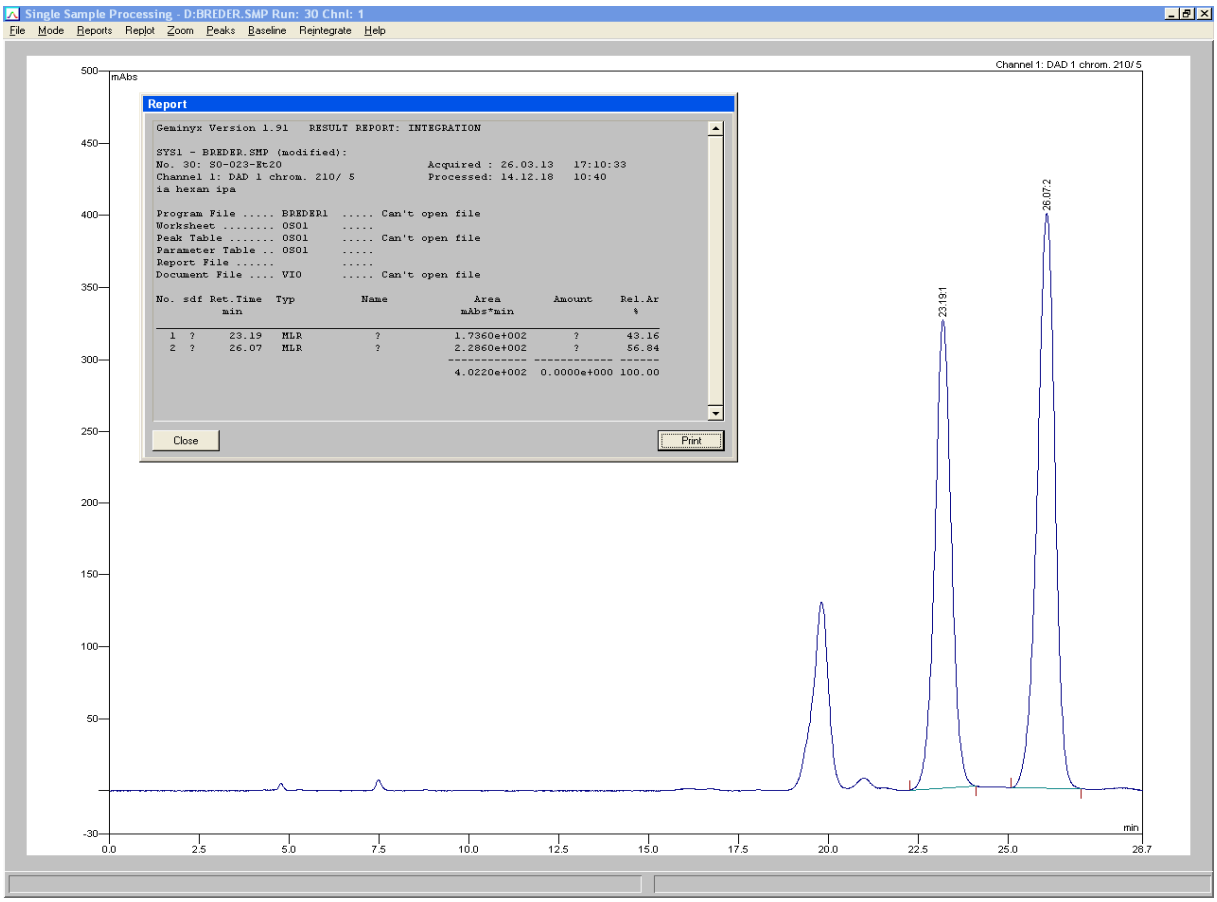
6.1.8 Entry 7



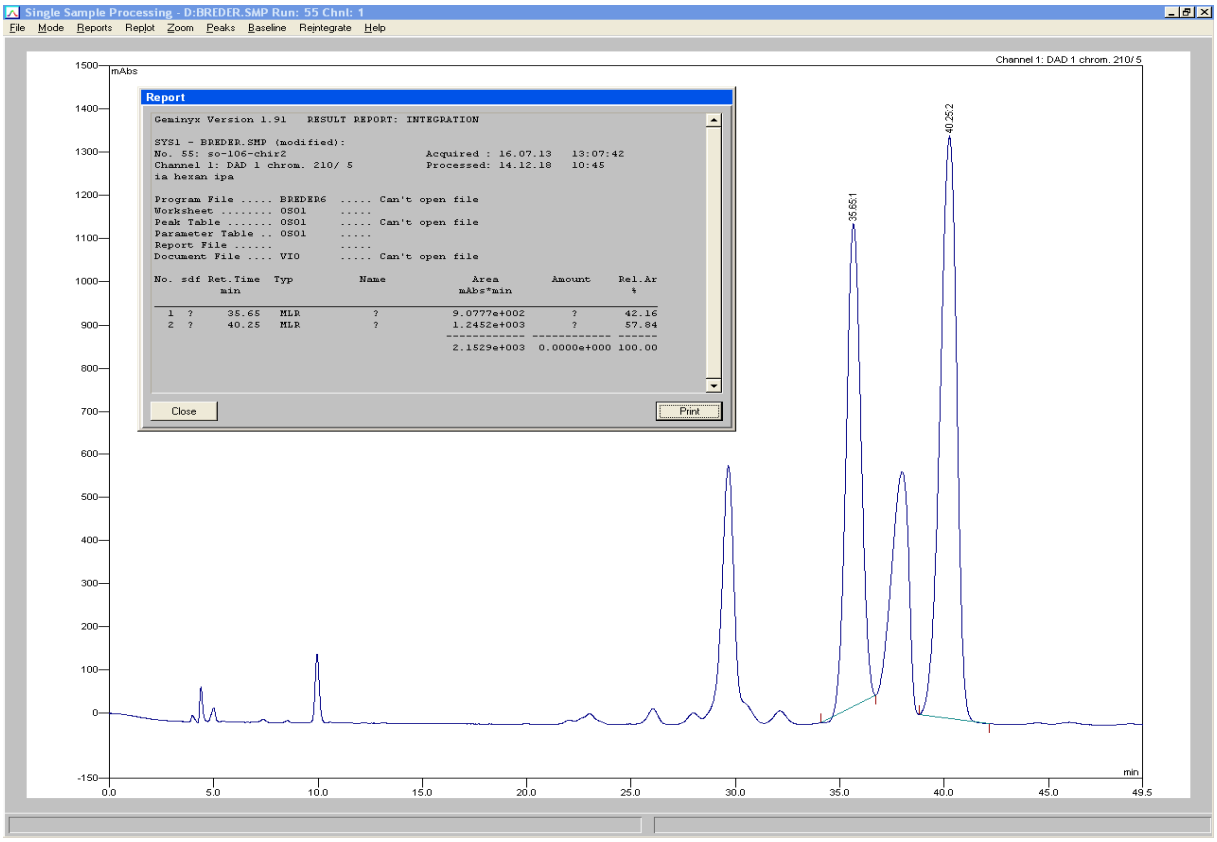
6.1.9 Entry 8



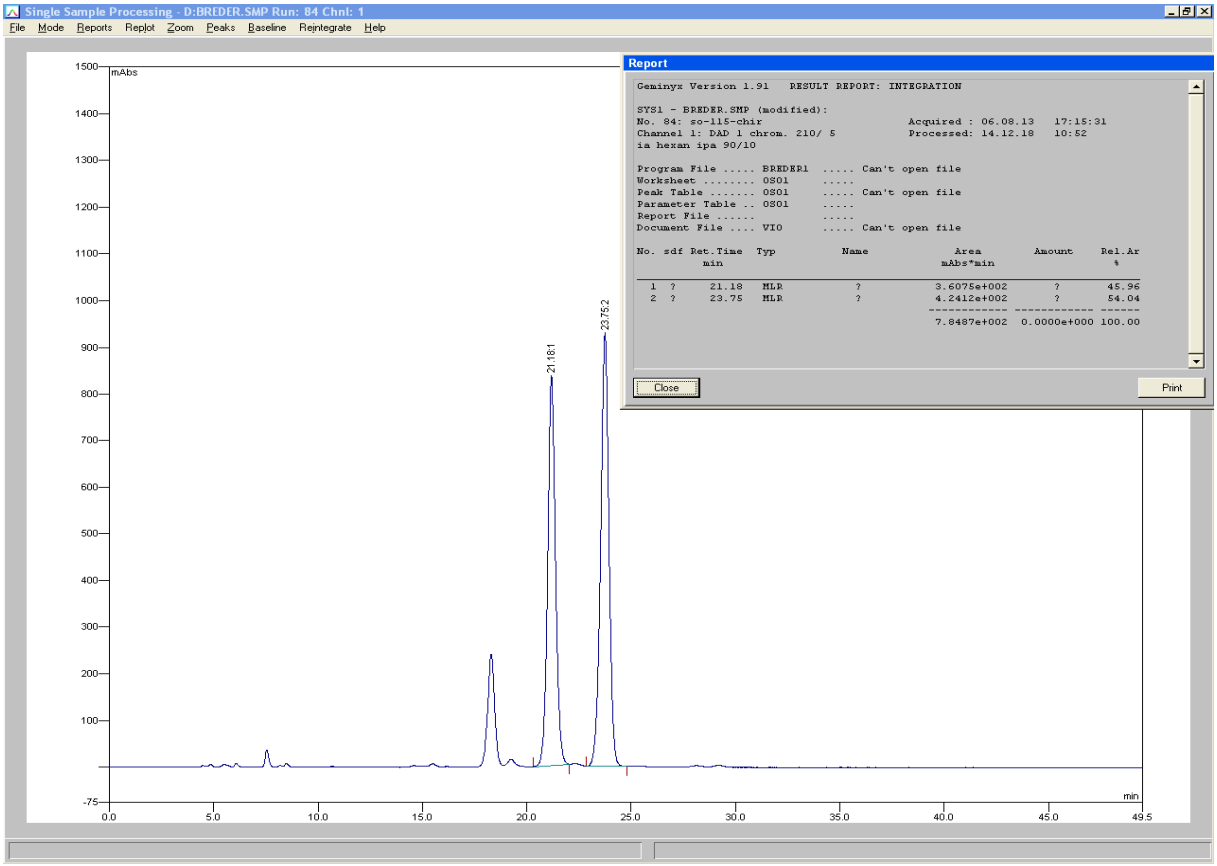
6.1.10 Entry 9



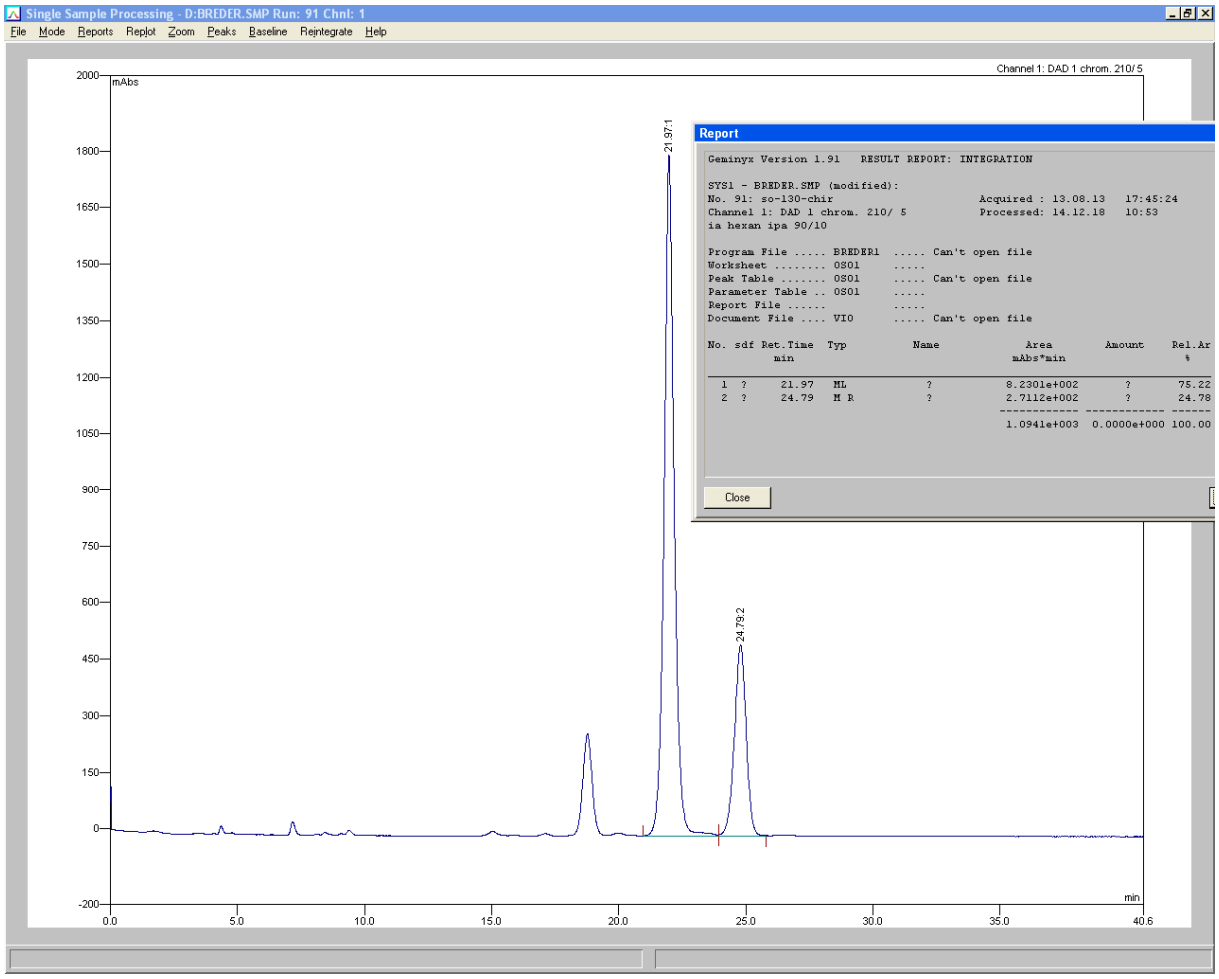
6.1.11 Entry 11



6.1.12 Entry 12



6.1.13 Entry 13



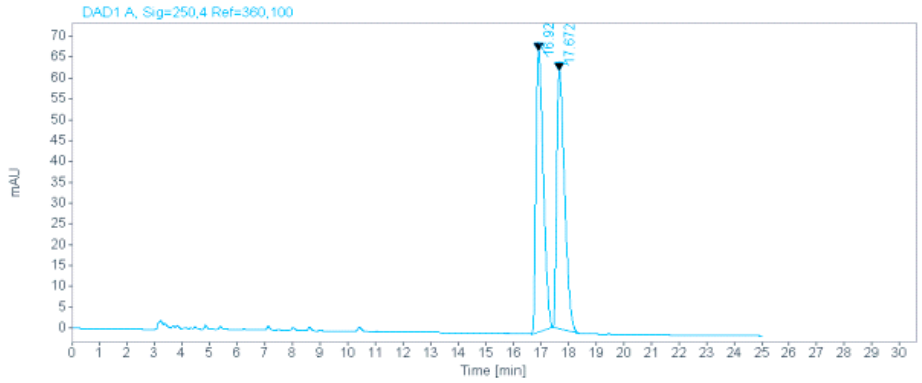
6.2 Lactonisation

6.2.1 Racemate

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Praktikum\FK-Bre04.D
Sample name:	FK-Bre04
Description:	FK-Bre04_Racemat
Instrument:	LC1260
Injection date:	11/2/2017 2:40:54 PM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_90.1_1.0_25_I D.M
Last changed:	9/16/2016 3:57:01 PM
Column name:	CHIRALPAK ID-3
Serial #:	555
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=250,4 Ref=360,100

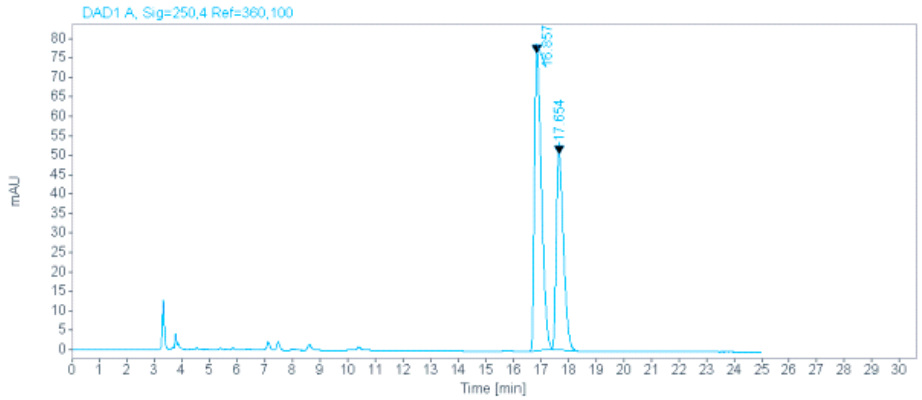
RT [min]	Type	Width [min]	Area	Height	Area%	Name
16.920	MM	0.3071	1242.6899	67.4498	50.2540	
17.672	MM	0.3316	1230.1281	61.8351	49.7460	
		Sum	2472.8180			

6.2.2 Entry 1

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Praktikum\FK-Bre04_Gruppe12.D
Sample name:	FK-Bre04_Gruppe12
Description:	FK-Bre04_Menthol-Kat
Instrument:	LC1260
Injection date:	11/17/2017 3:15:14 PM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_90.1_1.0_25_I D.M
Last changed:	9/16/2016 3:57:01 PM
Column name:	CHIRALPAK ID-3
Serial #:	555
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=250,4 Ref=360,100

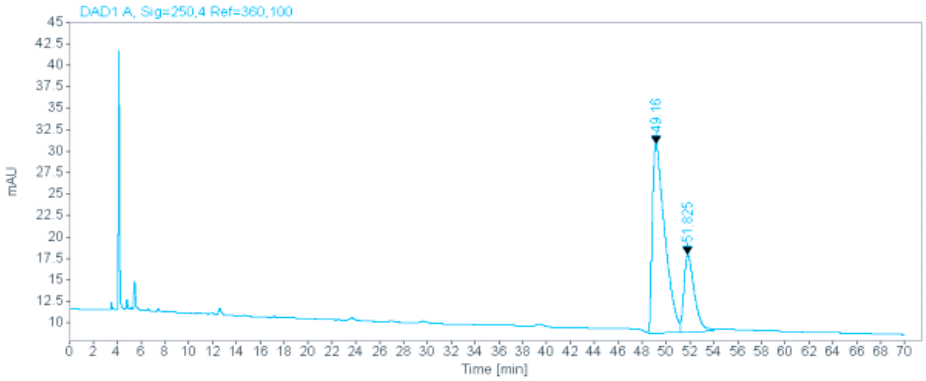
RT [min]	Type	Width [min]	Area	Height	Area%	Name
16.857	MM	0.2969	1362.2036	76.4608	59.6493	
17.654	MM	0.3042	921.4839	50.4820	40.3507	
		Sum	2283.6875			

6.2.3 Entry 2

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\Induktion_neuerKat_221 2016-03-01 18-13-37\FK221-01.D
Sample name:	FK221-01
Description:	
Instrument:	LC1260
Injection date:	3/1/2016 6:14:37 PM
Acq. method:	20_99_0.9_70_OD.M
Analysis method:	20_99_0.9_70_OD.M
Last changed:	3/1/2016 5:50:23 PM
Column name:	CHIRALCEL OD-3
Serial #:	444
Injection volume:	20.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=250,4 Ref=360,100

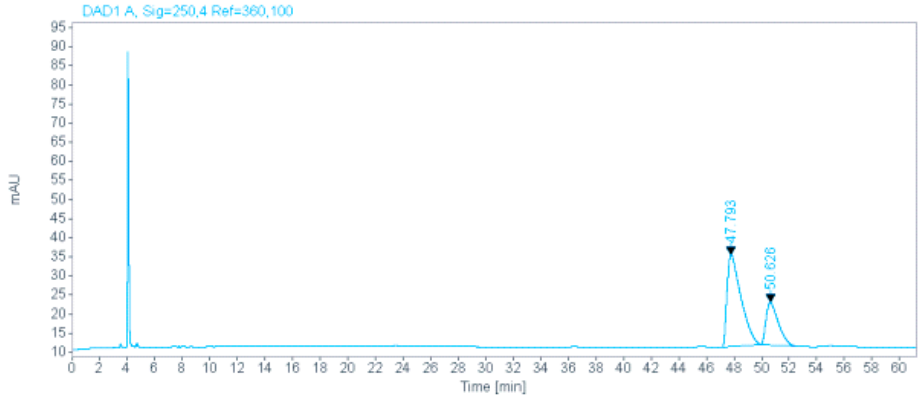
RT [min]	Type	Width [min]	Area	Height	Area%	Name
49.160	MF	1.2046	1590.5411	22.0060	74.2714	
51.825	FM	1.0229	550.9844	8.9779	25.7286	
		Sum	2141.5256			

6.2.4 Entry 4

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\Induktion_neuerKat_221 2016-03-01 18-13-37\FK221-03.D
Sample name:	FK221-03
Description:	
Instrument:	LC1260
Injection date:	3/1/2016 8:36:43 PM
Acq. method:	20_99_0.9_70_OD.M
Analysis method:	20_99_0.9_70_OD.M
Last changed:	3/1/2016 5:50:23 PM
Column name:	CHIRALCEL OD-3
Serial #:	444
Injection volume:	20.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=250,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
47.793	BB	1.0049	1689.7697	24.1333	71.0134	
50.626	BB	0.8932	689.7393	11.0654	28.9866	
		Sum	2379.5090			

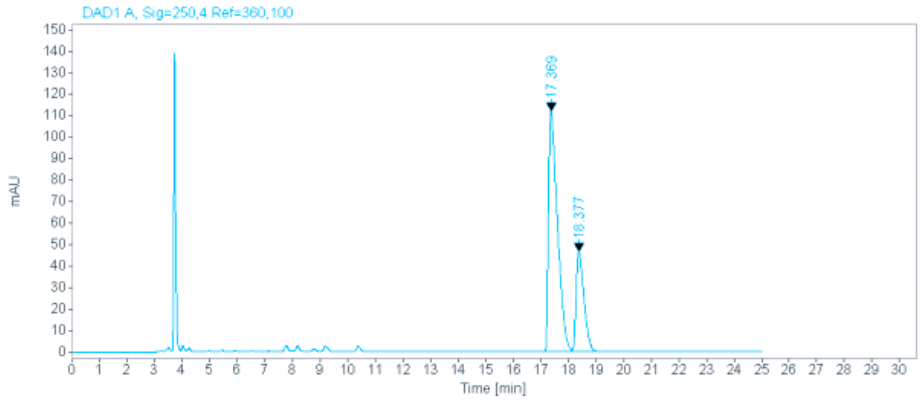
6.2.5 Entry 5

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-15_Ph-Menth_0°C.D
Sample name:	FK221-15_Ph-Menth_0°C
Description:	10_90.1_1.0_25_ID FK221-15_Ph-Menth_0°C

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	9/16/2016 6:03:34 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_ID.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:	CHIRALPAK ID-3		
Serial #:	555		



Signal: DAD1 A, Sig=250,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
17.369	BV	0.3374	2534.8894	112.2577	73.2480	
18.377	VB	0.3059	925.8031	46.5747	26.7520	
		Sum	3460.6925			

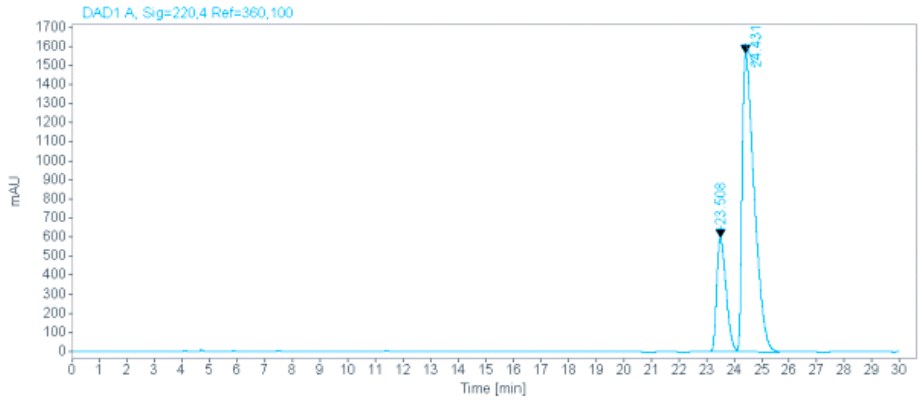
6.2.6 Entry 6

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-41.D
Sample name:	FK221-41
Description:	FK221-40 Decalinol Kat

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	11/2/2018 4:29:30 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_ID.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:	CHIRALPAK ID-3		
Serial #:	555		



Signal: DAD1 A, Sig=220,4 Ref=360,100

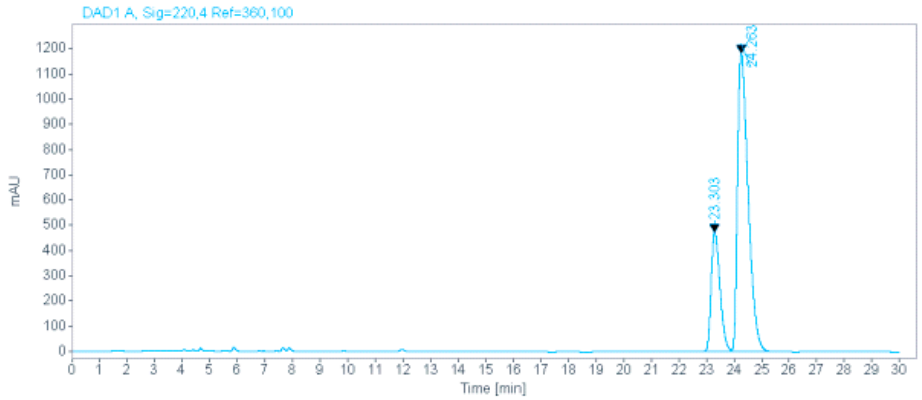
RT [min]	Type	Width [min]	Area	Height	Area%	Name
23.508	MM	0.3921	14020.5664	595.9428	22.7235	
24.431	MM	0.5080	47680.1875	1564.3646	77.2765	
		Sum	61700.7539			

6.2.7 Entry 7

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-42.D
Sample name:	FK221-42
Description:	FK221-42 Decalinol Kat DCE
Instrument:	LC1260
Injection date:	11/8/2018 10:59:48 AM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_90.1_1.0_25_ID.M
Last changed:	6/5/2018 5:13:20 PM
Column name:	CHIRALPAK ID-3
Serial #:	555
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=220,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
23.303	BV	0.3480	10534.7627	472.6717	25.0152	
24.263	VB	0.4141	31578.7324	1180.6094	74.9848	
		Sum	42113.4951			

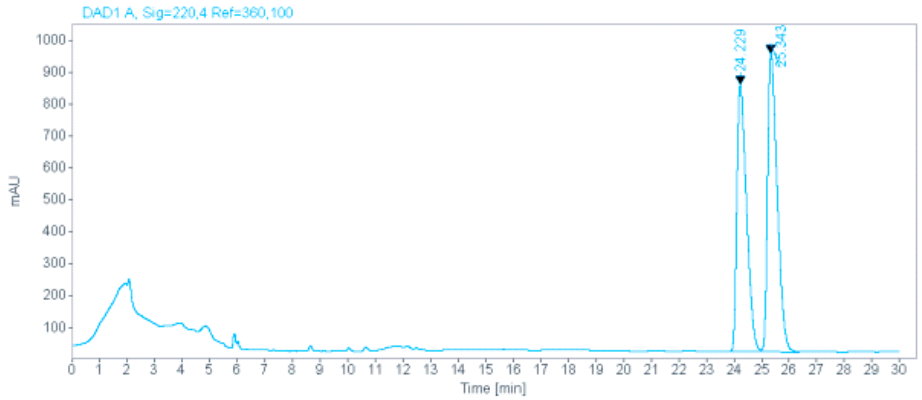
6.2.8 Entry 8

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-46.D
Sample name:	FK221-46
Description:	FK221-46 Borneol Kat. entspricht FK221-02

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	12/17/2018 5:03:16 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_ID.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:			
Serial #:			



Signal: DAD1 A, Sig=220,4 Ref=360,100

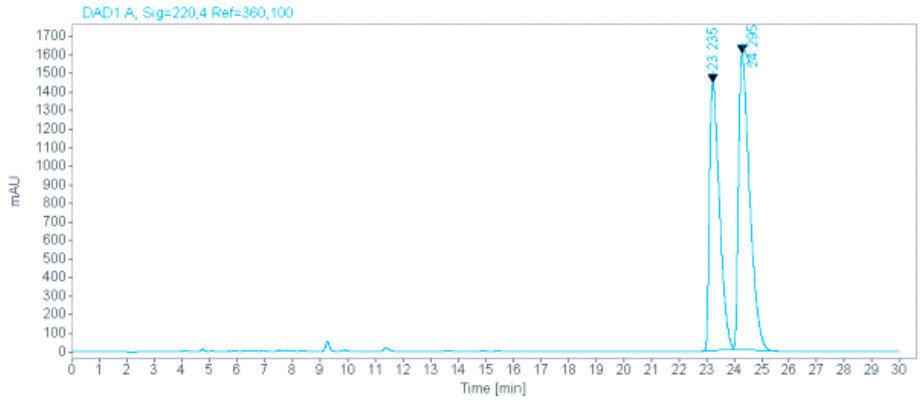
RT [min]	Type	Width [min]	Area	Height	Area%	Name
24.229	MM	0.4034	20262.0078	837.0413	47.2880	
25.343	MM	0.4032	22586.0664	933.6463	52.7120	
		Sum	42848.0742			

6.2.9 Entry 9

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-45.D
Sample name:	FK221-45
Description:	FK221-46 BINOL-Kat.
Instrument:	LC1260
Injection date:	12/17/2018 6:15:56 PM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_90.1_1.0_25_ID.M
Last changed:	6/5/2018 5:13:20 PM
Column name:	
Serial #:	
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=220,4 Ref=360,100

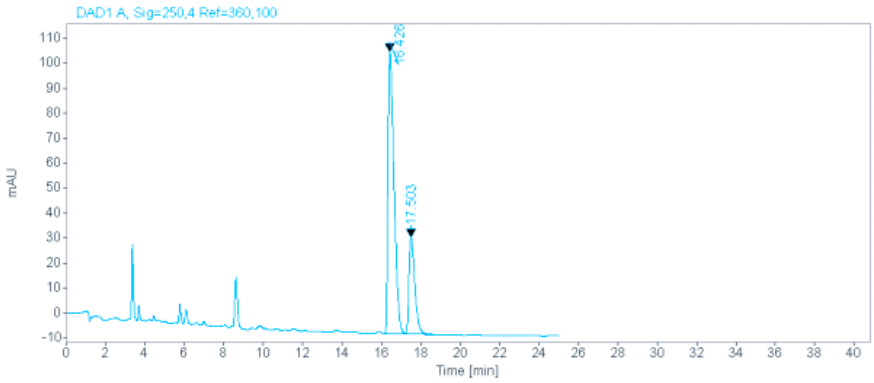
RT [min]	Type	Width [min]	Area	Height	Area%	Name
23.235	MM	0.4202	36434.2930	1445.1360	44.9293	
24.295	MM	0.4658	44658.2227	1597.7968	55.0707	
		Sum	81092.5156			

6.2.10 Entry 10

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\so-918 2018-05-16 17-36-03\FK221-23.D
Sample name:	FK221-23
Description:	
Instrument:	LC1260
Injection date:	5/16/2018 5:38:12 PM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_90.1_1.0_25_ID.M
Last changed:	9/16/2016 3:57:01 PM
Column name:	CHIRALPAK ID-3
Serial #:	555
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=250,4 Ref=360,100

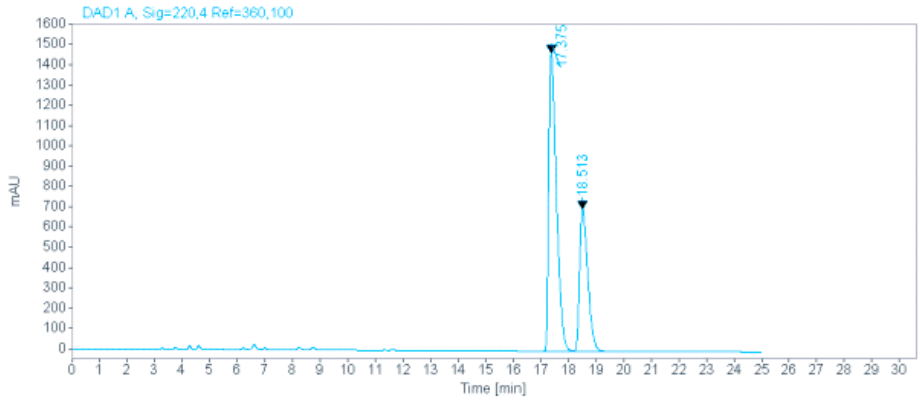
RT [min]	Type	Width [min]	Area	Height	Area%	Name
16.426	BV	0.3317	2397.2534	112.9012	74.7810	
17.503	VB	0.3170	808.4459	38.8092	25.2190	
		Sum	3205.6993			

6.2.11 Entry 11

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-24.D
Sample name:	FK221-24
Description:	FK221-24_DiPhenylmethyl
Instrument:	LC1260
Injection date:	5/24/2018 3:12:35 PM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_90.1_1.0_25_I D.M
Last changed:	6/5/2018 5:13:20 PM
Column name:	CHIRALPAK ID-3
Serial #:	555
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=220,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
17.375	BV	0.2983	28718.0859	1467.6029	66.4654	
18.513	VB	0.3197	14489.4551	699.3735	33.5346	
		Sum	43207.5410			

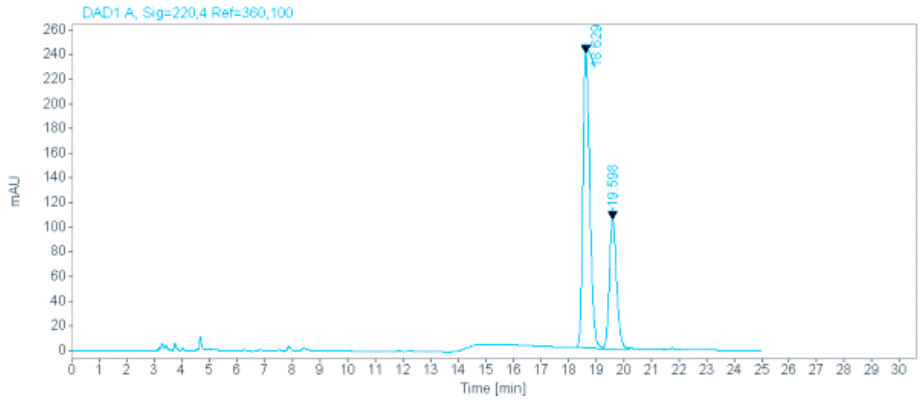
6.2.12 Entry 12

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-25.D
Sample name:	FK221-25
Description:	FK221-25_DiPhenylmethyl_Maruoka Cond.

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	5/25/2018 5:02:13 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_ID.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:	CHIRALPAK ID-3		
Serial #:	555		



Signal: DAD1 A, Sig=220,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
18.629	MM	0.2907	4160.9102	238.5581	68.2703	
19.598	MM	0.3081	1933.8499	104.6171	31.7297	
		Sum	6094.7600			

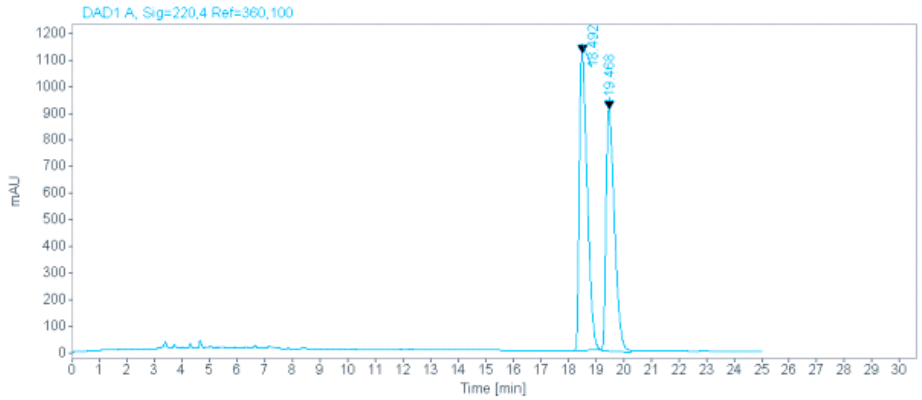
6.2.13 Entry 13

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-26.D
Sample name:	FK221-24
Description:	FK221-26_DiPhenylmethyl_PhMe

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	5/25/2018 3:21:05 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_ID.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:	CHIRALPAK ID-3		
Serial #:	555		



Signal: DAD1 A, Sig=220,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
18.492	MM	0.3337	22314.7383	1114.5094	53.7668	
19.468	MM	0.3526	19188.0918	906.8666	46.2332	
		Sum	41502.8301			

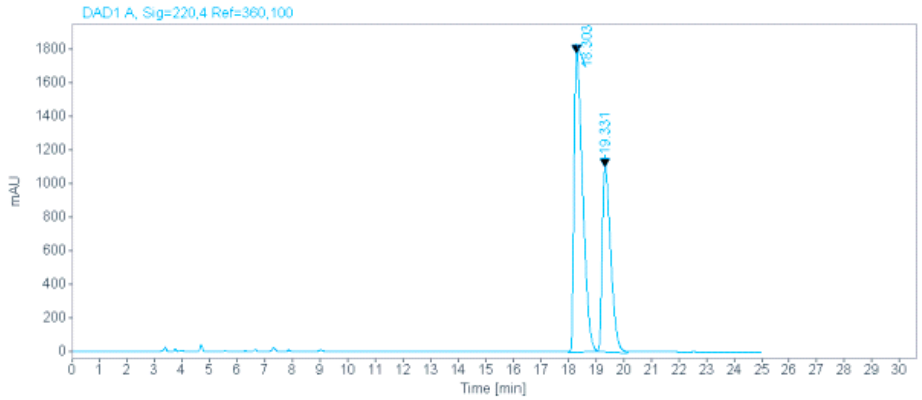
6.2.14 Entry 14

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-27.D
Sample name:	FK221-27
Description:	FK221-27_DiPhenylmethyl_Lange_Bestrahlung

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	5/28/2018 4:49:10 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_ID.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:	CHIRALPAK ID-3		
Serial #:	555		



Signal: DAD1 A, Sig=220,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
18.303	MM	0.3625	38601.9414	1774.8201	62.1891	
19.331	MM	0.3560	23469.9395	1098.8024	37.8109	
		Sum	62071.8809			

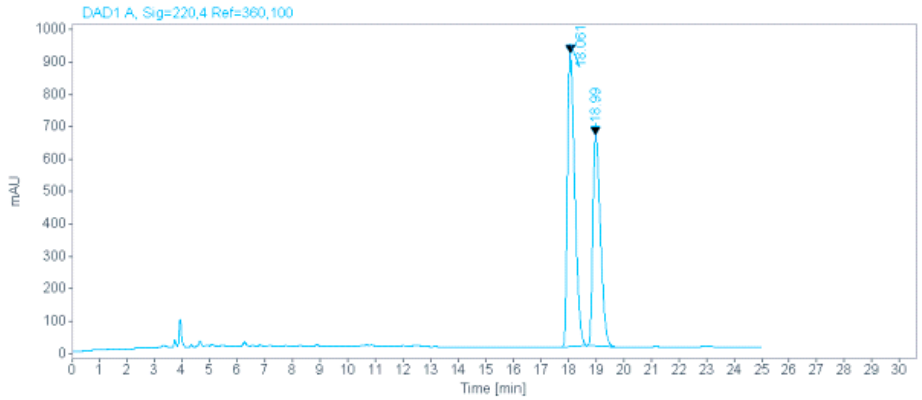
6.2.15 Entry 15

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-29.D
Sample name:	FK221-27
Description:	FK221-27_DiPhenylmethyl_MeCN_6h

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	5/31/2018 1:41:51 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_ID.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:	CHIRALPAK ID-3		
Serial #:	555		



Signal: DAD1 A, Sig=220,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
18.061	MM	0.3098	16799.9395	903.8807	57.3758	
18.990	MM	0.3194	12480.6104	651.3352	42.6242	
		Sum	29280.5498			

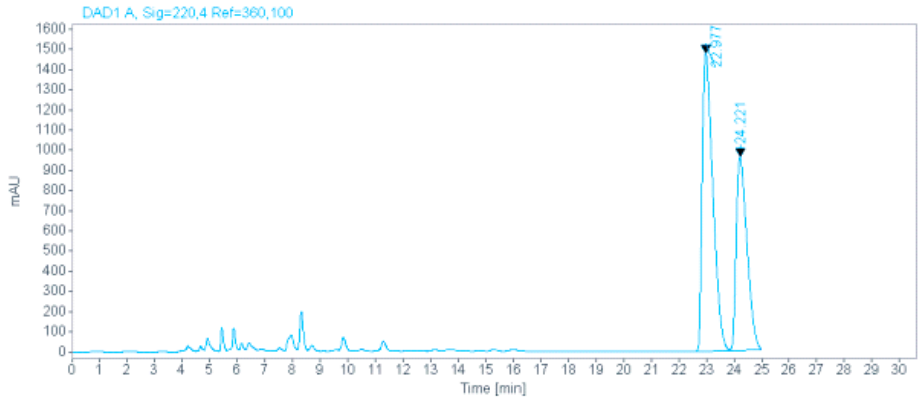
6.2.16 Entry 16

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-30.D
Sample name:	FK221-30
Description:	FK221-30_Tiefe Temperatur

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	6/5/2018 2:52:27 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_ID.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:	CHIRALPAK ID-3		
Serial #:	555		



Signal: DAD1 A, Sig=220,4 Ref=360,100

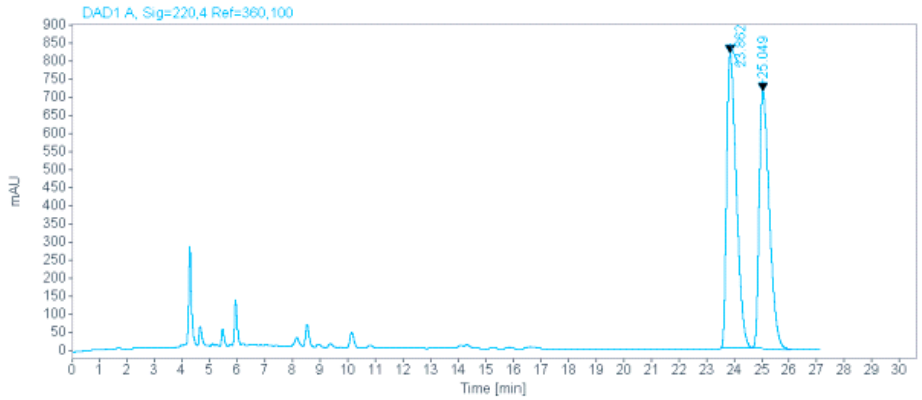
RT [min]	Type	Width [min]	Area	Height	Area%	Name
22.977	BV	0.4129	39589.2891	1475.9030	60.4868	
24.221	VBA	0.4172	25861.8711	957.3465	39.5132	
		Sum	65451.1602			

6.2.17 Entry 19

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-34.D
Sample name:	FK221-34
Description:	FK221-34_FK431-Ru
Instrument:	LC1260
Injection date:	6/8/2018 3:41:57 PM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_90.1_1.0_25_ID.M
Last changed:	6/5/2018 5:13:20 PM
Column name:	CHIRALPAK ID-3
Serial #:	555
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=220,4 Ref=360,100

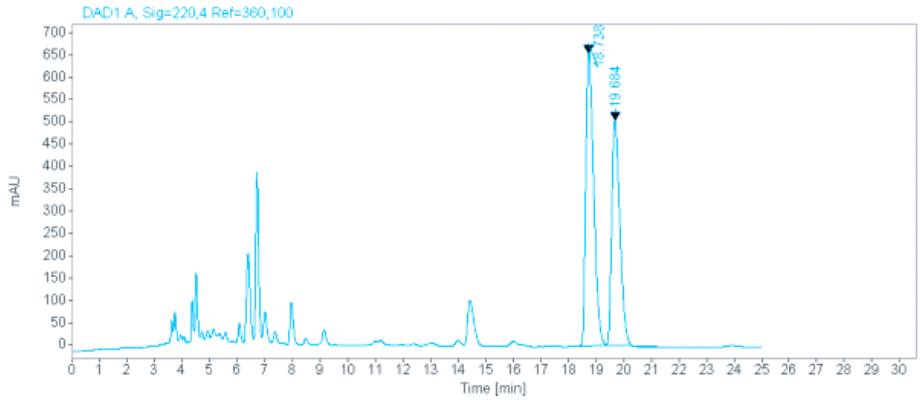
RT [min]	Type	Width [min]	Area	Height	Area%	Name
23.862	MM	0.4080	19926.8262	813.9940	52.1906	
25.049	MM	0.4278	18254.0508	711.2316	47.8094	
		Sum	38180.8770			

6.2.18 Entry 20

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-34_BPh4.D
Sample name:	FK221-34
Description:	FK221-34_FK431-PyrrOMeBPh4
Instrument:	LC1260
Injection date:	6/13/2018 4:42:17 PM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_90.1_1.0_25_I D.M
Last changed:	6/5/2018 5:13:20 PM
Column name:	CHIRALPAK ID-3
Serial #:	555
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=220,4 Ref=360,100

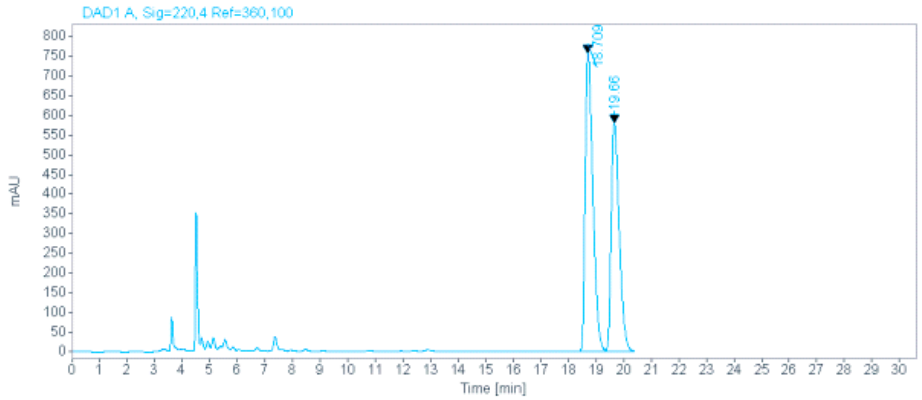
RT [min]	Type	Width [min]	Area	Height	Area%	Name
18.738	MM	0.3314	13023.4590	654.9884	55.8723	
19.684	MM	0.3406	10285.8906	503.2816	44.1277	
		Sum	23309.3496			

6.2.19 Entry 21

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-35_BPh4_dry.D
Sample name:	FK221-35
Description:	FK221-35_FK431-PyrrOMeBPh4_dry
Instrument:	LC1260
Injection date:	6/13/2018 5:28:30 PM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_90.1_1.0_25_I D.M
Last changed:	6/5/2018 5:13:20 PM
Column name:	CHIRALPAK ID-3
Serial #:	555
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=220,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
18.709	BV	0.3018	14767.6328	756.3871	55.7442	
19.660	VBA	0.3119	11724.1719	579.9187	44.2558	
		Sum	26491.8047			

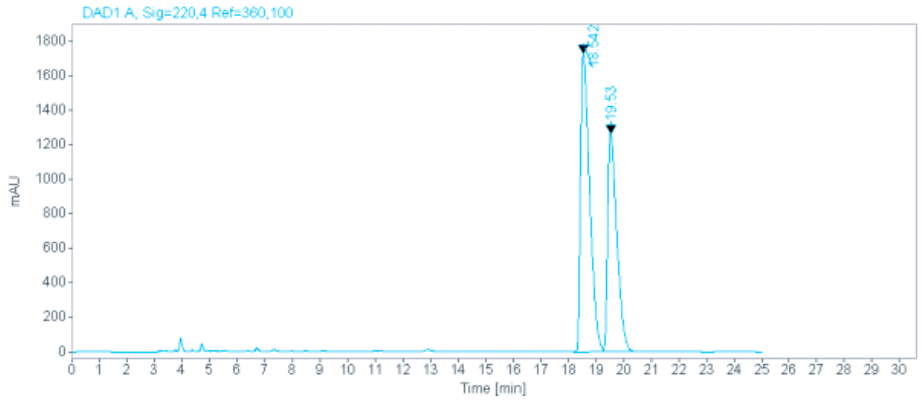
6.2.20 Entry 22

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-36.D
Sample name:	FK221-36
Description:	FK221-36_FK431_dry_conditions

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	6/14/2018 3:13:10 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_ID.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:	CHIRALPAK ID-3		
Serial #:	555		



Signal: DAD1 A, Sig=220,4 Ref=360,100

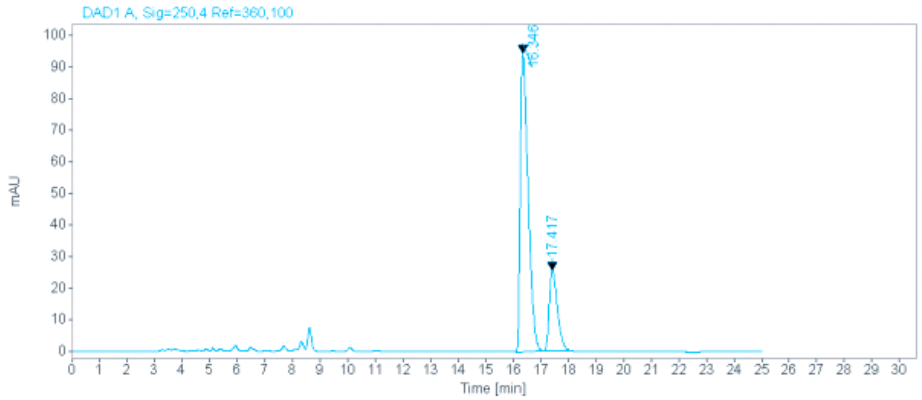
RT [min]	Type	Width [min]	Area	Height	Area%	Name
18.542	MM	0.3844	39879.4492	1729.0916	57.8361	
19.530	MM	0.3840	29073.1152	1261.9829	42.1639	
		Sum	68952.5645			

6.2.21 Entry 24

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Katha\RW-6-FR7-8.D
Sample name:	RW-6-FR7-8
Description:	RW-6-FR7-8
Instrument:	LC1260
Injection date:	3/15/2018 3:20:59 PM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_99.5_0.8_20_OD.M
Last changed:	2/28/2018 2:20:13 PM
Column name:	CHIRALPAK ID-3
Serial #:	555
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=250,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
16.346	MM	0.3293	1865.0796	94.3985	77.9591	
17.417	MM	0.3435	527.3024	25.5813	22.0409	
		Sum	2392.3820			

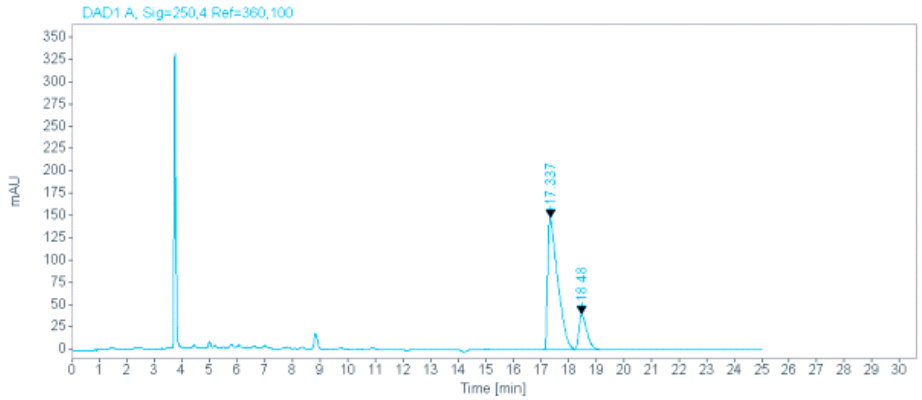
6.2.22 Entry 26

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-13_Maruoka_0°C.D
Sample name:	CD-329OJ-III
Description:	10_90.1_1.0_25_ID FK221-13_Maruoka_0°C

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	9/16/2016 3:59:29 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_I D.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:	CHIRALPAK ID-3		
Serial #:	555		



Signal: DAD1 A, Sig=250,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
17.337	MM	0.4216	3744.0320	148.0126	82.5616	
18.480	MM	0.3366	790.8032	39.1546	17.4384	
		Sum	4534.8352			

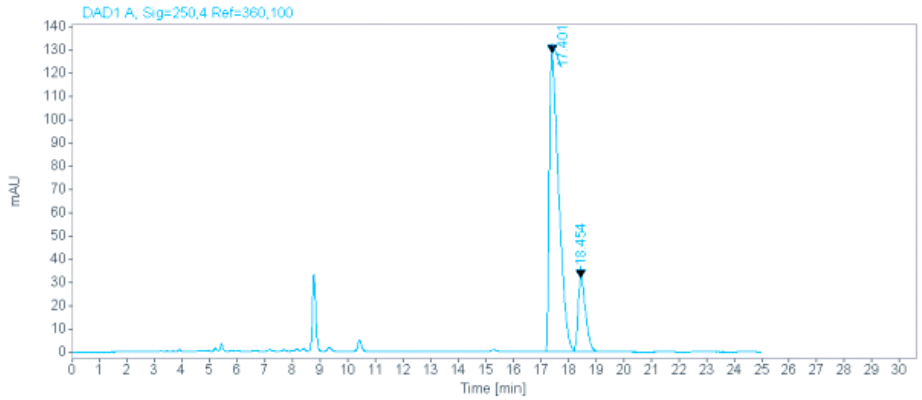
6.2.23 Entry 27

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-14_Maruoka_0°C.D
Sample name:	FK221-14_Maruoka_0°C
Description:	10_90.1_1.0_25_IDFK221-14_Maruoka_0°C

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	9/19/2016 4:00:16 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_I D.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:	CHIRALPAK ID-3		
Serial #:	555		



Signal: DAD1 A, Sig=250,4 Ref=360,100

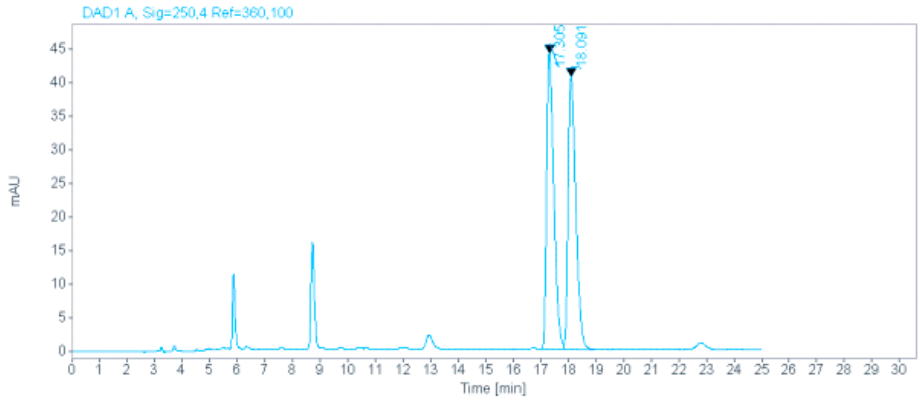
RT [min]	Type	Width [min]	Area	Height	Area%	Name
17.401	BV	0.3529	3043.9487	128.2518	83.1920	
18.454	VB	0.3006	614.9948	31.6648	16.8080	
		Sum	3658.9435			

6.2.24 Entry 31

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-19_NOBF4_Maruoka.D
Sample name:	FK221-19
Description:	FK221-19_NOBF4_Maruoka
Instrument:	LC1260
Injection date:	6/12/2017 3:47:25 PM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_90.1_1.0_25_I D.M
Last changed:	6/5/2018 5:13:20 PM
Column name:	CHIRALPAK ID-3
Serial #:	555
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=250,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
17.305	MM	0.3036	800.9833	43.9698	50.3888	
18.091	MM	0.3241	788.6212	40.5596	49.6112	
		Sum	1589.6045			

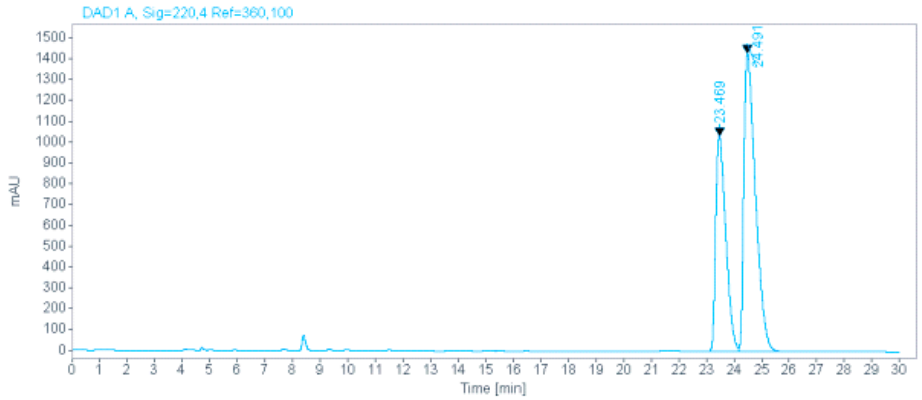
6.2.25 Entry 32

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-43.D
Sample name:	FK221-43
Description:	FK221-43 Diselenocin Acetal

Instrument:	LC1260	**Injection volume:**	10.000
Injection date:	12/14/2018 4:28:34 PM	**Acq. operator:**	SYSTEM
Acq. method:	10_90.1_1.0_25_ID.M	**Sample type:**	Sample
Analysis method:	10_90.1_1.0_25_ID.M	**Dilution:**	1
Last changed:	6/5/2018 5:13:20 PM		
Column name:	CHIRALPAK ID-3		
Serial #:	555		



Signal: DAD1 A, Sig=220,4 Ref=360,100

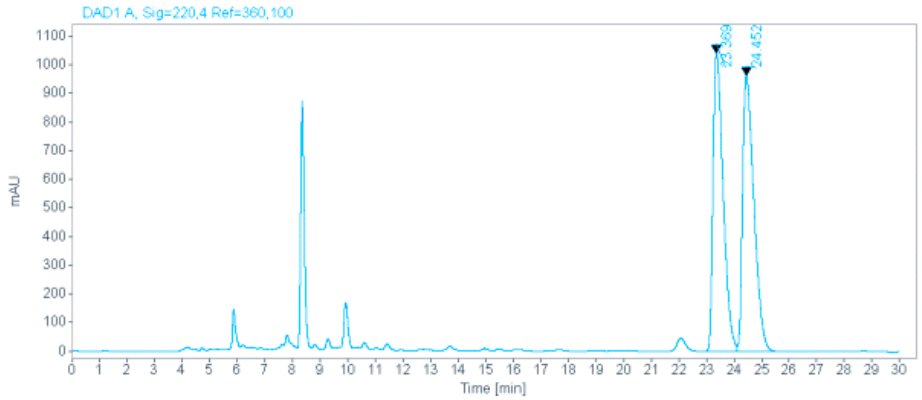
RT [min]	Type	Width [min]	Area	Height	Area%	Name
23.469	MM	0.4227	26266.6113	1035.7816	38.5517	
24.491	MM	0.4878	41866.8594	1430.5667	61.4483	
		Sum	68133.4707			

6.2.26 Entry 33

Single Injection Report

Agilent Technologies

Data file:	C:\Data\Felix\Induktion_neuerKat_221\FK221-44.D
Sample name:	FK221-44
Description:	FK221-44 Diselenocin Carbonat
Instrument:	LC1260
Injection date:	12/14/2018 5:12:58 PM
Acq. method:	10_90.1_1.0_25_ID.M
Analysis method:	10_90.1_1.0_25_ID.M
Last changed:	6/5/2018 5:13:20 PM
Column name:	CHIRALPAK ID-3
Serial #:	555
Injection volume:	10.000
Acq. operator:	SYSTEM
Sample type:	Sample
Dilution:	1



Signal: DAD1 A, Sig=220,4 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%	Name
23.369	BV	0.3969	26648.8867	1040.2244	49.2414	
24.452	VB	0.4365	27470.0293	963.8536	50.7586	
		Sum	54118.9160			