Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Straightforward Synthesis of Enantiopure (*R*)- and (*S*)-trifluoroalaninol

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General informations

Unless otherwise mentioned, all the reagents were purchased from commercial source. All glassware was dried in an oven prior to use. Ether and THF were distilled under nitrogen from sodium/benzophenone prior to use. CH_2Cl_2 was distilled under nitrogen from CaH_2 prior to use. ^1H NMR (400,00 MHz or 250.00 MHz), ^{13}C NMR (100,50 MHz or 62.90 MHz) and ^{19}F NMR (376,20 MHz or 235.36 MHz) were measured on a JEOL 400 spectrometer or a BRÜCKER ADVANCE 250 DPX. Chemical shifts of ^1H NMR were expressed in parts per million downfield from tetramethylsilane (δ = 0 ppm) in $CDCl_3$. Chemical shifts of ^{13}C NMR were expressed in parts per million downfield from $CDCl_3$ as internal standard (δ = 77.0 ppm). Chemical shifts of ^{19}F NMR were expressed in parts per million downfield from C_6F_6 as internal standard (δ = –164.9 ppm). Coupling constants are reported in hertz. Column chromatography was performed on Merck Kieselgel 60 (0,040–0,063 mm), employing mixture of specified solvent as eluent. Thin-layer chromatography (TLC) was performed on Merck silica gel (Merck 60 PF_{254}) plates. Silica TLC plates were visualized under UV light, by a 10% solution of phosphomolybdic acid in ethanol followed by heating. Gas chromatography was performed on Agilent 6890 N (detector with ionization of flame) and with a polydimethylsiloxane column HP ultra I (25 m x 3.2 mm x 0.52 µm thickness of layer). Mass spectra (MS) were obtained on a GC/MS apparatus HP 5973 MSD with an HP 6890 Series GC. Ionization was obtained by electronic impact (EI 70 eV). Infrared spectra (IR) were obtained by Fourier-transformation on BRÜCKER TENSOR 27, wave numbers are given in cm^{-1}. Elemental analyses were performed by the CNRS analysis central service. Optical rotations are reported as their specific rotations at 25 ºC in g/100 mL, determined using a JASCO P1010 polarimeter. Melting points were obtained on a Büchi apparatus and are uncorrected.

Synthesis of α-Tfm-aminonitriles: α-Tfm-aminonitriles (*R*)-**2** and (*S*)-**2** were prepared according to our previously reported procedure.[1]

2-((1*R*)-2-hydroxy-1-phenylethylamino)-3,3,3-trifluoromethylpropanoate (3) : To a 83:17 mixture of aminonitriles (*R*)-**2** and (*S*)-**2** (4.07 g, 16.7 mmol, 1 equiv) was added dropwise a solution of saturated hydrochloric acid in methanol (200 mL) at 0°C. The reaction was refluxed for 5 days, cooled down to room temperature, poured into a saturated solution of sodium bicarbonate (250 mL) and concentrated under reduced pressure. The crude material was purified by flash chromatography (80/20 cyclohexane/ethyl acetate) to afford a 67:33 diastereomeric mixture of (*R*)-**3** and (*S*)-**3** as a yellow oil (3.92 g, 85%). Pure (*R*)-**3** and (*S*)-**3** samples were isolated for analytical studies. Major diastereomer (*R*)-**3**: $[\alpha]_D$ –141.3 (*c* 1.3, $CHCl_3$). IR (neat): 3330, 1746, 1128, 701 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ 2.26 (s, 1H, OH), 2.96 (s, 1H, NH), 3.47-3.72 (m, 4H), 3.74 (s, 3H, CH_3O), 7.18-7.29 (m, 5H, Ar) ppm. ^{13}C NMR (62.9 MHz, $CDCl_3$): δ 52.3 (s, CH_3), 60.3 (q, *J* = 30.1 Hz, CH), 62.0 (s, CH), 66.7 (s, CH_2), 122.4 (q, *J* = 281.0 Hz, CF_3), 126.6 (s, Ar), 127.3 (s, Ar), 128.0 (s, Ar), 137.3 (s, Ar), 167.7 (s, CO) ppm. ^{19}F NMR (235 MHz, $CDCl_3$): δ –73.2 (d, *J* = 7.1 Hz, 3F, CF_3) ppm. MS (EI): *m*/*z* (%) = 246 [M^+-OMe] (100), 186, 159, 118. Minor diastereomer (*S*)-**3**: $[\alpha]_D$ –24.8 (*c* 2.2, $CHCl_3$). IR (neat): 3348, 1747, 1122, 700 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ 2.52 (s, 1H, OH), 2.72 (s, 1H, NH), 3.55-3.81 (m, 3H), 3.63 (s, 3H, CH_3O), 3.90 (dd, *J* = 8.4 Hz, *J* = 4.0 Hz, CH-Ph), 7.24-7.36 (m, 5H, Ar) ppm. ^{13}C NMR (62.9 MHz, $CDCl_3$): δ 52.6 (s, CH_3), 60.3 (q, *J* = 29.8 Hz, CH), 63.5 (s, CH), 66.4 (s, CH_2), 123.6 (q, *J* = 283.4 Hz, CF_3), 127.2 (s, Ar), 127.8 (s, Ar), 128.6 (s, Ar),

(1) F. Huguenot and T. Brigaud, *J. Org. Chem.*, 2006, **71**, 7075–7078.

138.8 (s, Ar), 167.5 (s, CO) ppm. ^{19}F NMR (235 MHz, $CDCl_3$): δ –72.8 (d, J = 7.1 Hz, 3F, CF_3) ppm. MS (EI): m/z (%) = 246 [M^+-OMe] (100), 186, 159, 118.

2-((1*R*)-2-hydroxy-1-phenylethylamino)-3,3,3-trifluoropropan-1-ol (4): To a 67:33 solution of (*R*)-**3** and (*S*)-**3** (1.38 g, 4.98 mmol) in anhydrous diethyl ether (75 mL) was added lithium aluminum hydride (754 mg, 19.9 mmol, 4 equiv) carefully at 0°C. The reaction was stirred under argon atmosphere at room temperature for 24 hours and quenched with successive addition of water (4 mL), sodium hydroxide (15% W, 4 mL) and water (8 mL). The white precipitate was filtered over a pad of celite and washed with diethyl ether (100 mL). The filtrate was dried over magnesium sulfate, concentrated under reduced pressure, purified by flash chromatography (70/30 cyclohexane/ethyl acetate) to afford pure (*R*)-**4** (744 mg, 60%) and (*S*)-**4** (310 mg, 25%) as white solids. Major diastereomer (*R*)-**4**: mp = 65-67°C. $[\alpha]_D$ –68.1 (*c* 2.0, $CHCl_3$). IR (neat): 3328 (b), 2248, 2163 (w), 1456, 1388, 1280, 1247, 1129, 1079, 1019, 923, 758, 727, 698, 603 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.50 (s, 1H, NH), 3.08 (qdd, J = 7.9 Hz, J = 4.1 Hz, J = 3.7 Hz, 1H, CH-CF_3), 3.61 (dd, J = 10.5 Hz, J = 9.1 Hz, 1H, CH_2-OH), 3.73 (dd, J = 12.0 Hz, J = 5.1 Hz, 1H, CH_2-OH), 3.80 (dd, J = 10.5 Hz, J = 3.7 Hz, 1H, CH_2-OH), 3.90 (dd, J = 12.0 Hz, J = 3.7 Hz, 1H, CH_2-OH), 4.03 (dd, J = 9.1 Hz, J = 3.7 Hz, 1H, CHPh-CH_2), 4.13 (bs, 1H, OH), 4.25 (bs, 1H, OH), 7.20-7.50 (m, 5H, Ar) ppm. ^{13}C NMR (100.5 MHz, $CDCl_3$): δ 57.7 (s, CH_2), 57.8 (q, J = 26.8 Hz, CH-CF_3), 61.8 (s, CH), 67.3 (s, CH_2), 125.6 (q, J = 282.8 Hz, CF_3), 127.5 (s, Ar), 128.0 (s, Ar), 128.7 (s, Ar), 139.1 (s, Ar) ppm. ^{19}F NMR (376.2 MHz, $CDCl_3$): δ –76.7 (d, J = 7.9 Hz, 3F, CF_3) ppm. Anal. Calcd for $C_{11}H_{14}F_3NO_2$ (249.10): C, 53.01; H, 5.66; N, 5.62. found: C, 52.82; H, 5.70; N, 5.49. Minor diastereomer (*S*)-**4**: mp = 75-80°C. $[\alpha]_D$ –77.0 (*c* 2.0, $CHCl_3$). IR (neat): 3234 (b), 2875, 2162 (w), 1455, 1365, 1284, 1237, 1123, 1080, 1031, 993, 924, 753, 727, 695, 632 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$): δ 2.20-2.60 (bs, 3H, NH, OH, OH), 3.08 (qdd, J = 7.3 Hz, J = 4.6 Hz, J = 2.7 Hz, 1H, CH-CF_3), 3.53-3.70 (m, 3H, CH2-OH), 3.72

(dd, J = 10.9 Hz, J = 4.0 Hz, 1H, CH_2-OH), 4.12 (dd, J = 8.7 Hz, J = 4.0 Hz, 1H, CHPh-CH_2), 7.20-7.40 (m, 5H, Ar) ppm. ^{13}C NMR (62.9 MHz, $CDCl_3$): δ 57.8 (q, J = 26.4 Hz, CH-CF_3), 60.1 (s, 2C, CH_2, CH), 67.9 (s, CH_2), 126.6 (q, J = 282.1 Hz, CF_3), 128.0 (s, Ar), 128.7 (s, Ar), 129.3(s, Ar), 139.3 (s, Ar) ppm. ^{19}F NMR (376.2 MHz, $CDCl_3$): δ –75.5 (d, J = 7.2 Hz, 3F, CF_3) ppm.

(*E*)-1-Ethoxycarbonyl-2,2,2-trifluoroethylidene-((1*R*)-2-*tert*-butyldimethylsilyloxy-1-phenylethylamine (5):

Multigram quantity of **5** was prepared using our already reported procedure.[2]

Synthesis of aminodiols (*R*)-**4 and** (*S*)-**4 from the imine 5:**

To a solution of imine **5** (403 mg, 1 mmol) in anhydrous diethyl ether (25 mL) was added lithium aluminum hydride (152 mg, 4 mmol, 4 equiv) carefully at 0°C. The reaction was stirred under argon atmosphere at reflux for 24 hours. The reaction mixture was cool down to room temperature and quenched with successive addition of water (0.16 mL), sodium hydroxide (15% W, 0.16 mL) and water (0.48 mL). The white precipitate was filtered over a pad of celite and washed with diethyl ether. The filtrate was dried over magnesium sulfate, concentrated under reduced pressure, purified by flash chromatography (70/30 cyclohexane/ethyl acetate) to afford a 72:28 diastereomeric mixture of (*S*)-**4** and (*R*)-**4** (179 mg, 73%).

(4*R*)-2-carboethoxy-2-trifluoromethyl-4-phenyl-1,3-oxazolidine (6):

Multigram quantity of **(6)** was prepared using our already reported procedure.[2,3]

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(2) G. Chaume, M.-C. Van Severen, S. Marinkovic and T. Brigaud, *Org. Lett.*, 2006, **8**, 6123–6126.

(3) (a) C. Caupene, G. Chaume, L. Ricard and T. Brigaud, *Org. Lett.*, 2009, **11**, 209–212, (b) G. Chaume, M.-C. Van Severen, L. Ricard and T. Brigaud, *J. Fluorine Chem.*, 2008, **129**, 1104–

Synthesis of aminodiols (*R*)-**4 and** (*S*)-**4 from the oxazolidines 6:**

To a 83:17 mixture of **6** (1.0 g, 3.45 mmol) in anhydrous diethyl ether (50 mL) was added lithium aluminum hydride (524 mg, 13.8 mmol, 4 equiv) carefully at 0°C. The reaction was stirred under argon atmosphere at room temperature for 18 hours and quenched with successive addition of water (0.6 mL), sodium hydroxide (15% W, 0.6 mL) and water (1.8 mL). The white precipitate was filtered over a pad of celite and washed with diethyl ether (80 mL). The filtrate was dried over magnesium sulfate, concentrated under reduced pressure, purified by flash chromatography (70/30 to 50/50 cyclohexane/ethyl acetate) to afford pure (*R*)-**4** (509 mg, 60%) and (*S*)-**4** (211 mg, 24%) as white solids.

(*R*)-Trifluoroalaninol hydrochloride salt ((*R*)-7):

To a solution of **(*R*)-4** (400 mg, 1.6 mmol) in methanol (4 mL) was added successively palladium hydroxide (63 mg, 20% W, 0.08 mmol, 5 mol%) and a 3N hydrochloride solution (2.4 mL). The mixture was placed in an autoclave under hydrogen pressure (5.5 bar) and vigorously stirred for 48 hours. The suspension was filtered and evaporated to dryness under reduced pressure. The solid was washed with diethyl ether (3x3 mL) to remove 2-phenylethanol and evaporated under reduced pressure to give pure hydrochloride salt of **(*R*)-7** (202 mg, 76%). mp = 202°C. $[\alpha]_D$ +8.1 (*c* 0.8, EtOH). IR (neat): 3354, 2913, 2490, 1584, 1156, 1143 cm^{-1}. ^1H NMR (400 MHz, MeOH): δ 3.83 (A from ABX, J_{AB} = 13.5 Hz, J_{AX} = 5.0 Hz, 1H, CH_2-OH), 3.86 (B from ABX, J_{AB} = 13.5 Hz, J_{BX} = 4.6 Hz, 1H, CH_2-OH), 4.02 (qdd, J_{HF} = 7.6 Hz, J_{AX} = 5.0 Hz, J_{BX} = 4.6 Hz, 1H, CH-CF_3) ppm. ^{13}C NMR (62.9 MHz, MeOH): δ 55.2 (q, *J* = 30.2 Hz, CH-CF_3), 58.0 (s, CH_2), 125.3 (q, *J* = 280.8 Hz, CF_3) ppm. ^{19}F NMR (376.2 MHz, $CDCl_3$): δ –75.7

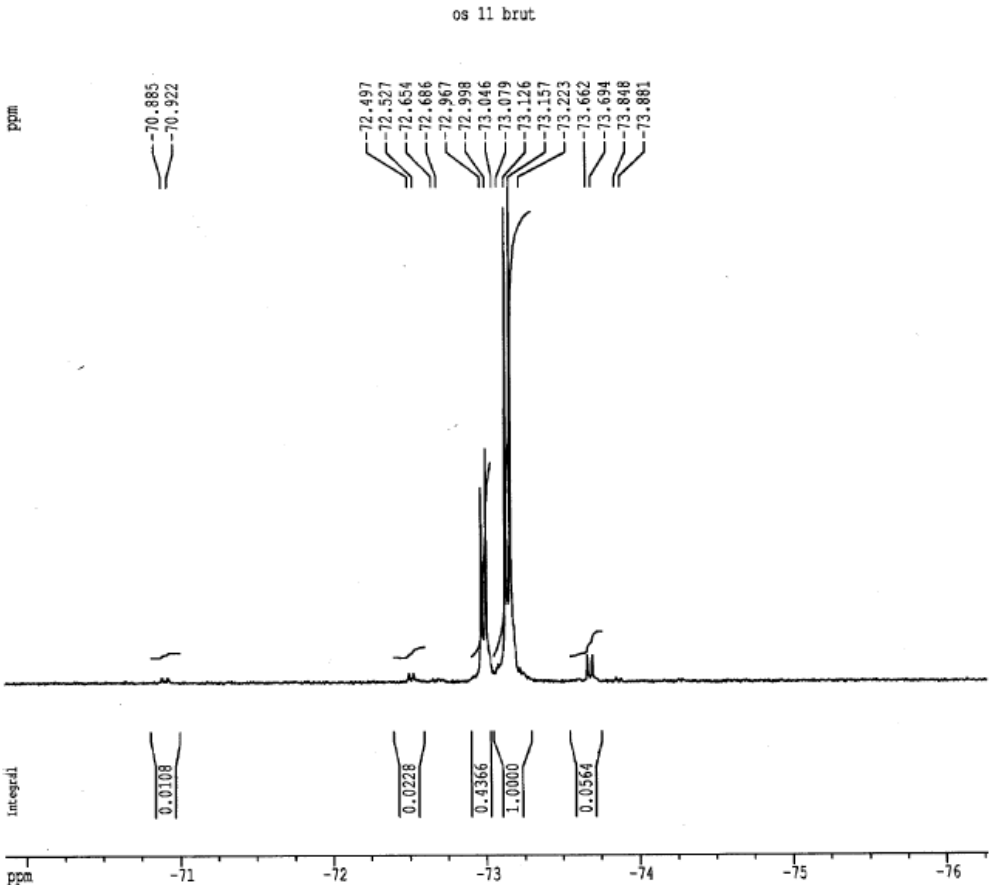
1109; (c) G. Chaume, N. Lensen, C. Caupene and T. Brigaud, *Eur. J. Org. Chem.*, 2009, 5717–5724.

(d, J = 7.6 Hz, 3F, CF_3) ppm. Anal. Calcd for $C_3H_7ClF_3NO$ (165.02): C, 21.77; H, 4.26; N, 8.46. found: C, 21.35, H, 4.22, N, 7.85.

(*S*)-Trifluoroalaninol hydrochloride salt ((*S*)-7):

Obtained from **(*S*)-4** (185 mg, 0.74 mmol), following the procedure used for **(*R*)-7**, as a white solid (110 mg, 90%). **(*S*)-7**: $[\alpha]_D$ –8.0 (*c* 0.6, EtOH). Other spectral and analytical properties are strictly identical to **(*R*)-7**.

^{19}F NMR: crude mixture of (*R*)-3 and (*S*)-3

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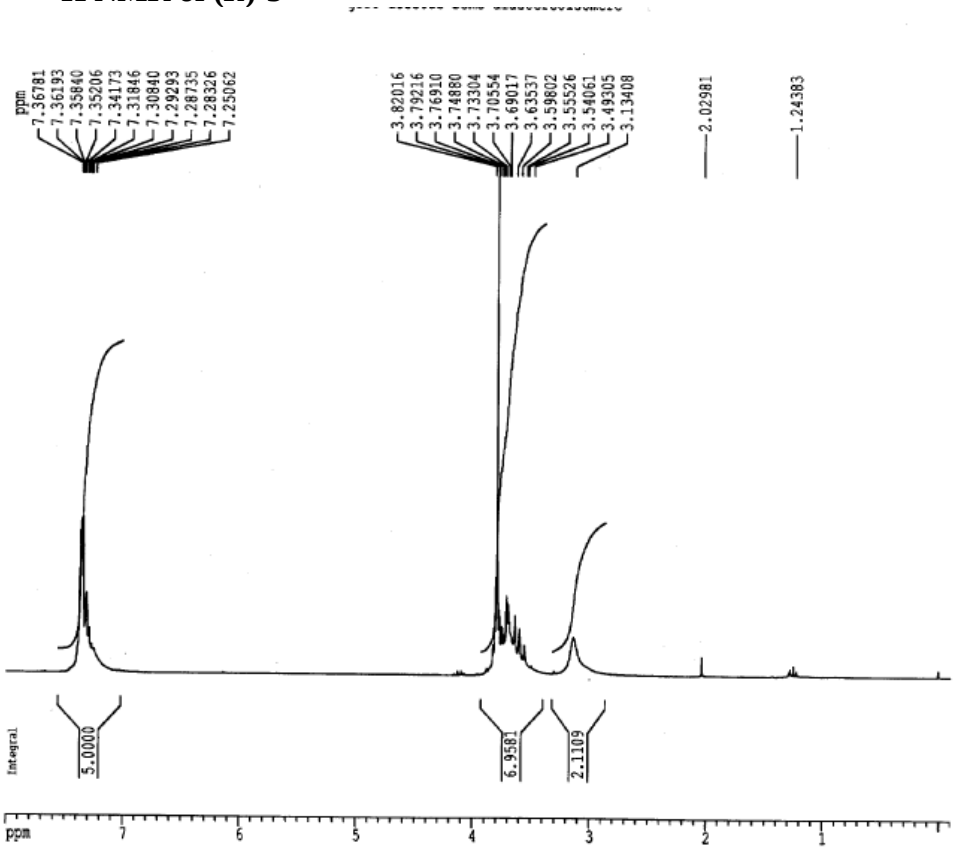
^{1}H NMR of (*R*)-3

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F1              2001.04 Hz
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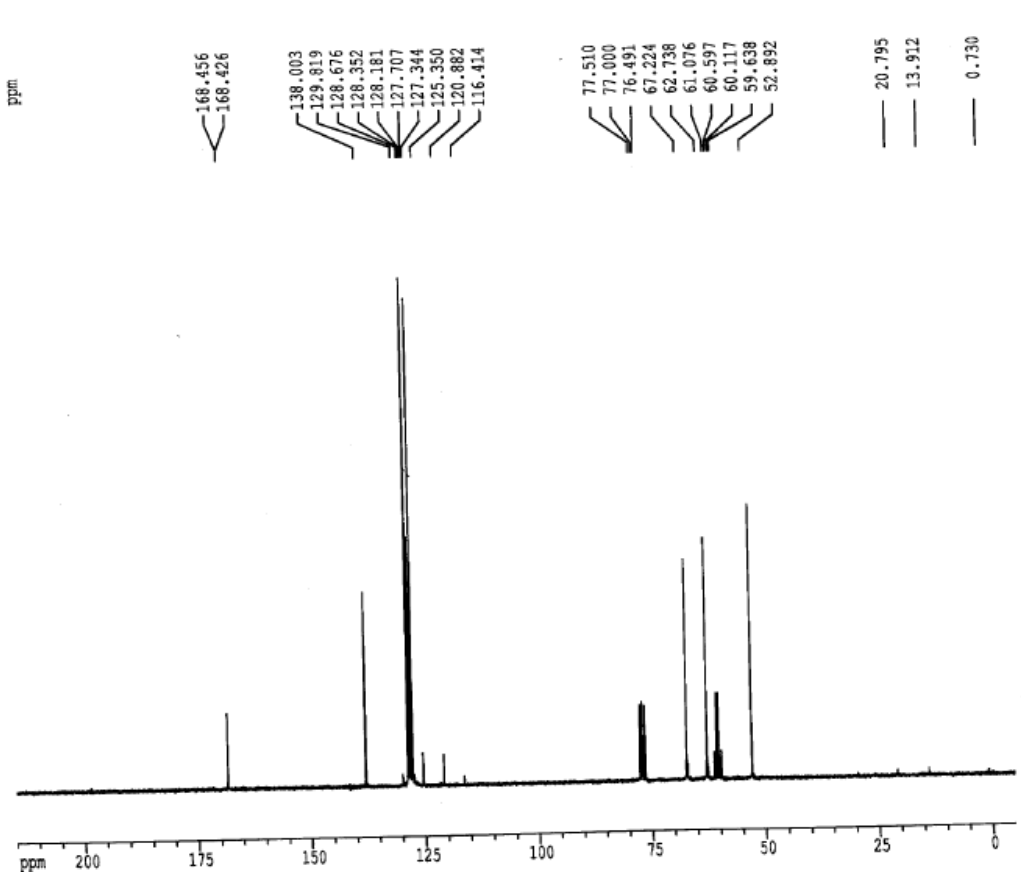
^{13}C NMR of (*R*)-3

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1D NMR plot parameters
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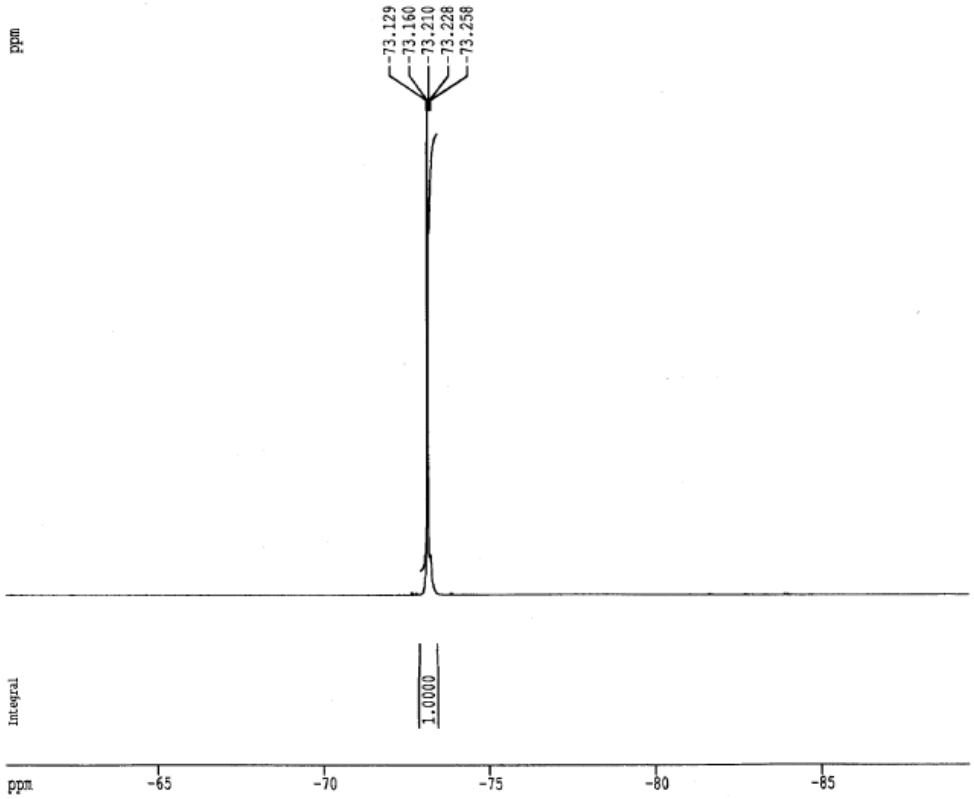
^{19}F NMR of (*R*)-3

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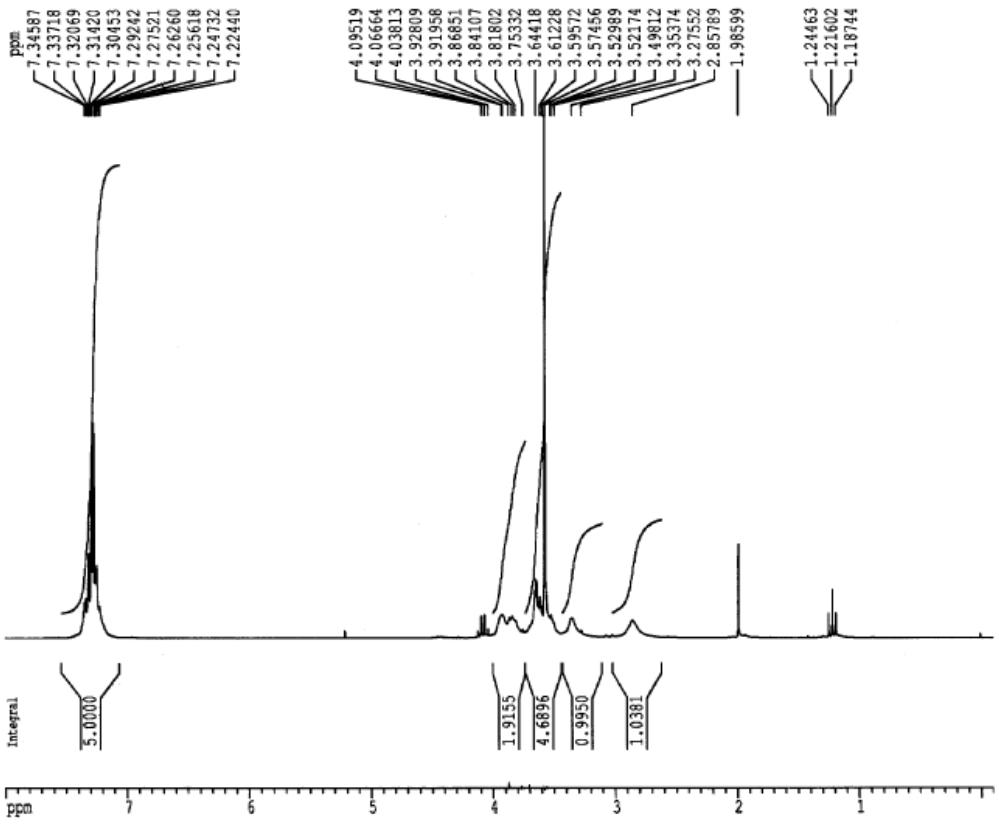
^{1}H NMR of (*S*)-3

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F1               2001.04 Hz
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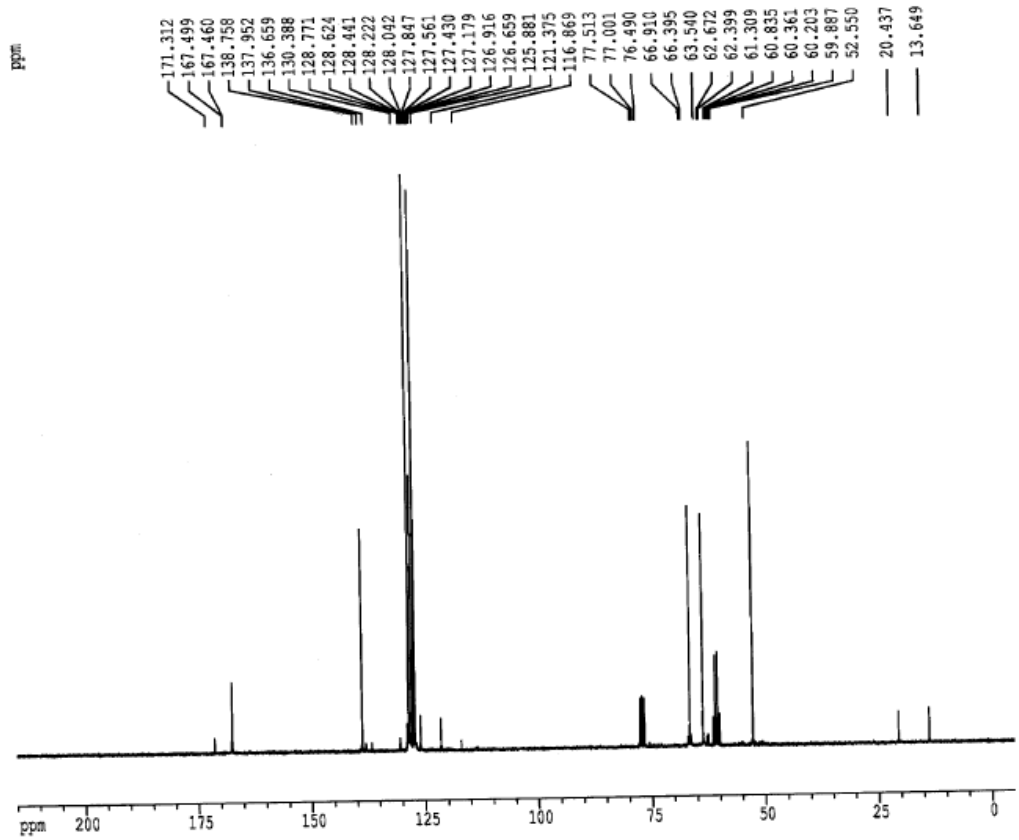
^{13}C NMR of (*S*)-3

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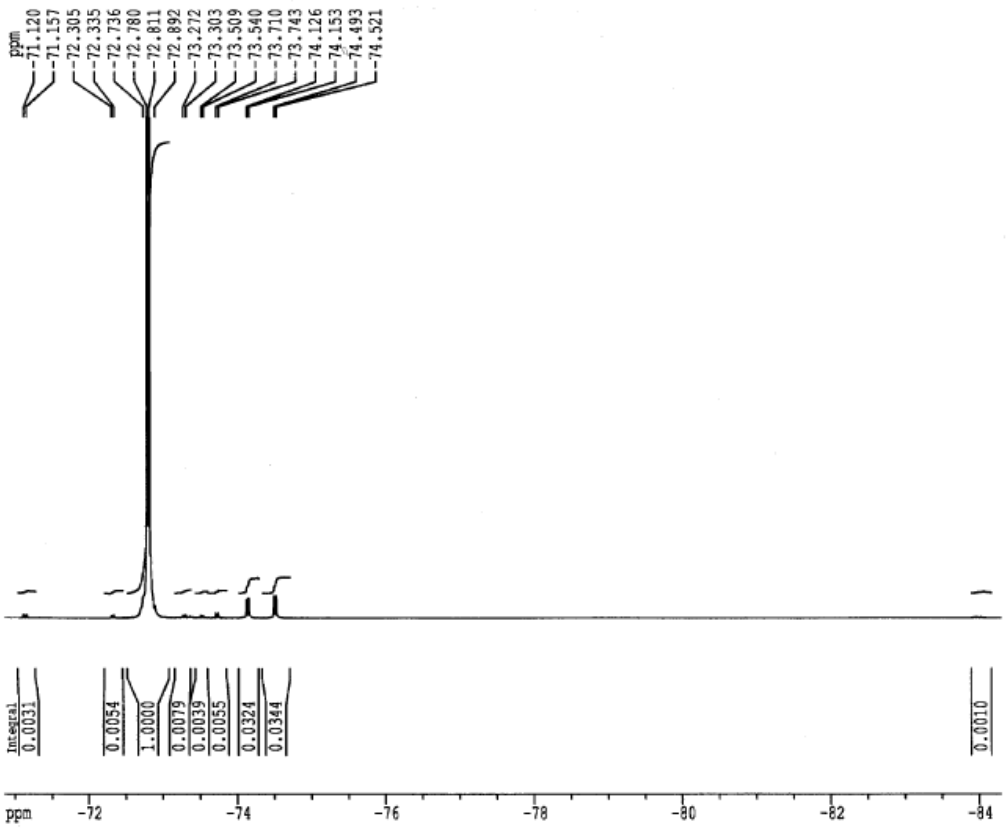
^{19}F NMR of (*S*)-3

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RG                  512
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TE                300.0 K
D1           1.00000000 sec

========== CHANNEL f1 =====
NUC1                19F
P1                19.50 usec
PL1               -4.00 dB
SFO1        235.3339190 MHz

F2 - Processing parameters
SI                 65536
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1D NMR plot parameters
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F2P              -84.297 ppm
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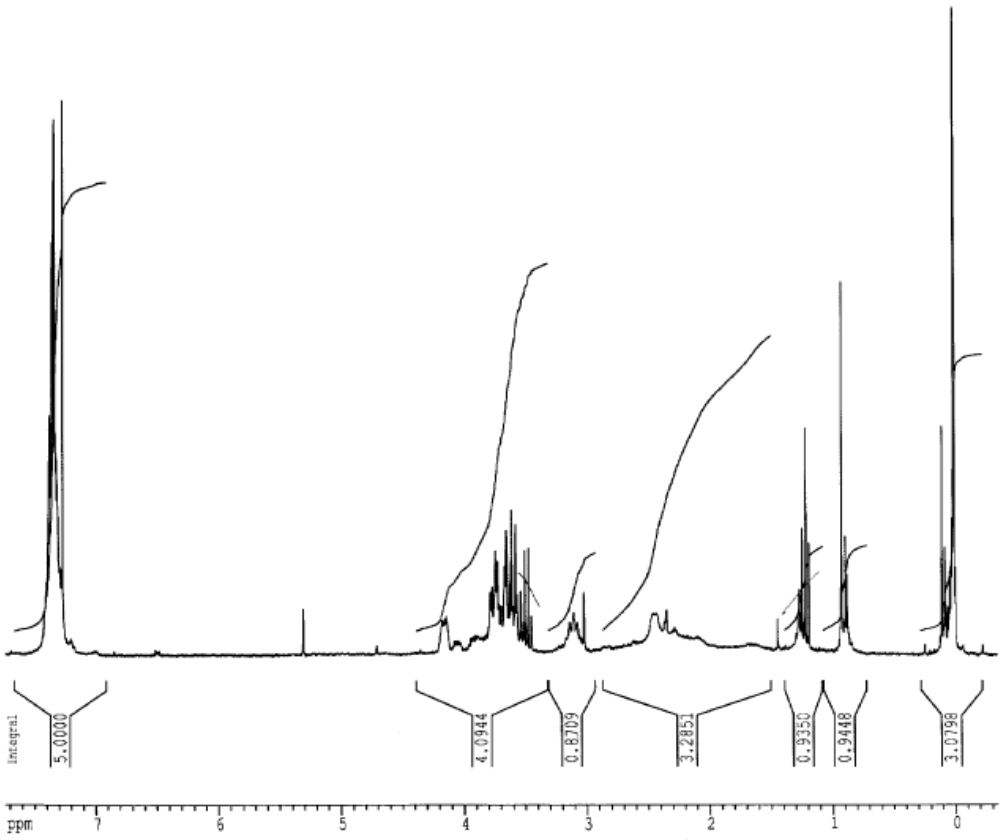
PPMCM              0.67201 ppm/c
HZCM          158.16312 Hz/cm
```

^{1}H NMR: crude mixture of (*R*)-4 and (*S*)-4 from imine 5

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Current Data Parameters
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EXPNO                2160
PROCNO                  1

F2 - Acquisition Parameters
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Time               18.04
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                 65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            5175.983 Hz
FIDRES        0.078979 Hz
AQ            6.3308277 sec
RG                912.3
DW                96.600 usec
DE                 6.50 usec
TE                300.0 K
D1           1.00000000 sec

========== CHANNEL f1 ======
NUC1                 1H
P1                13.50 usec
PL1               -6.00 dB
SFO1        250.1315447 MHz

F2 - Processing parameters
SI                 32768
SF          250.1300048 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
F1P                7.738 ppm
F1             1935.47 Hz
F2P              -0.343 ppm
F2              -85.88 Hz
PPMCM              0.40406 ppm/cm
HZCM          101.06743 Hz/cm
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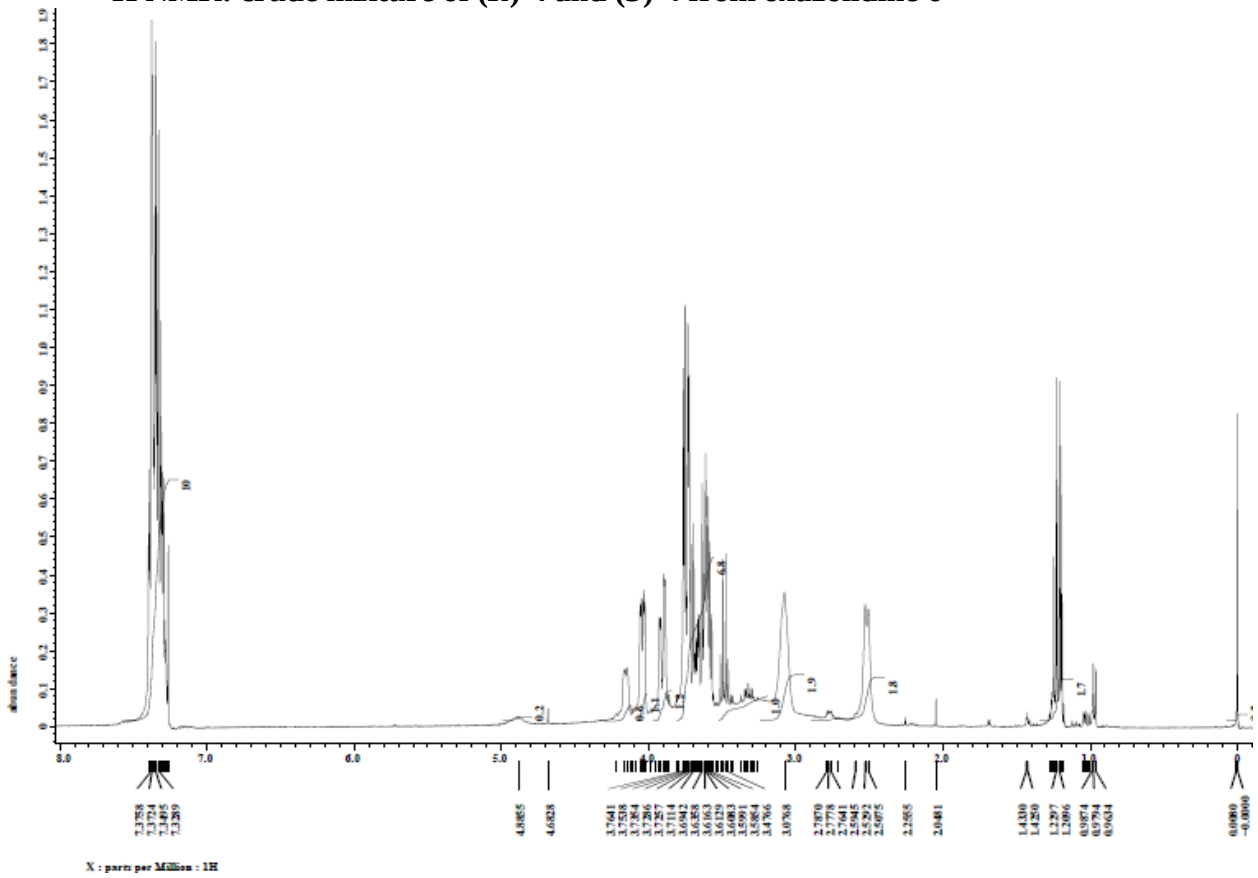
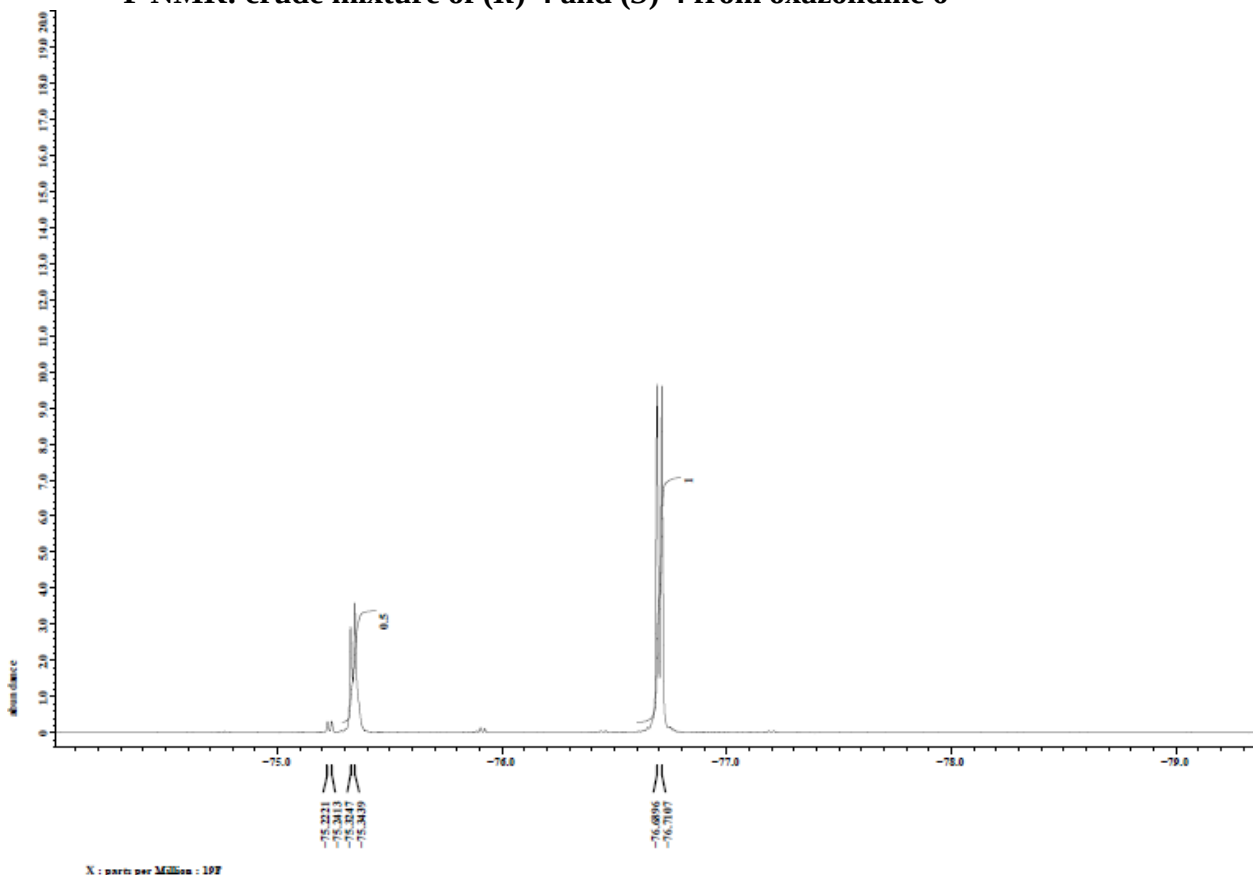
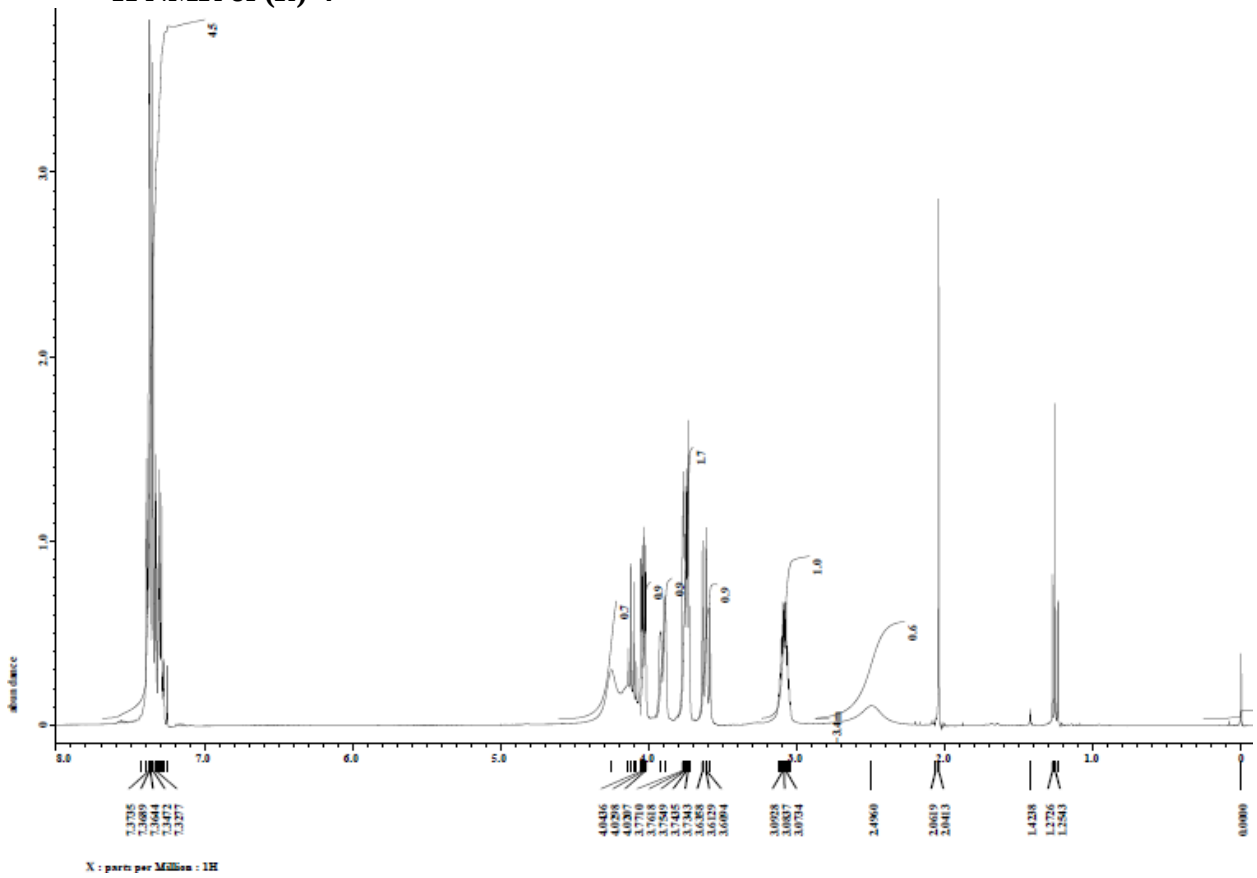
^{1}H NMR: crude mixture of (*R*)-4 and (*S*)-4 from oxazolidine 6



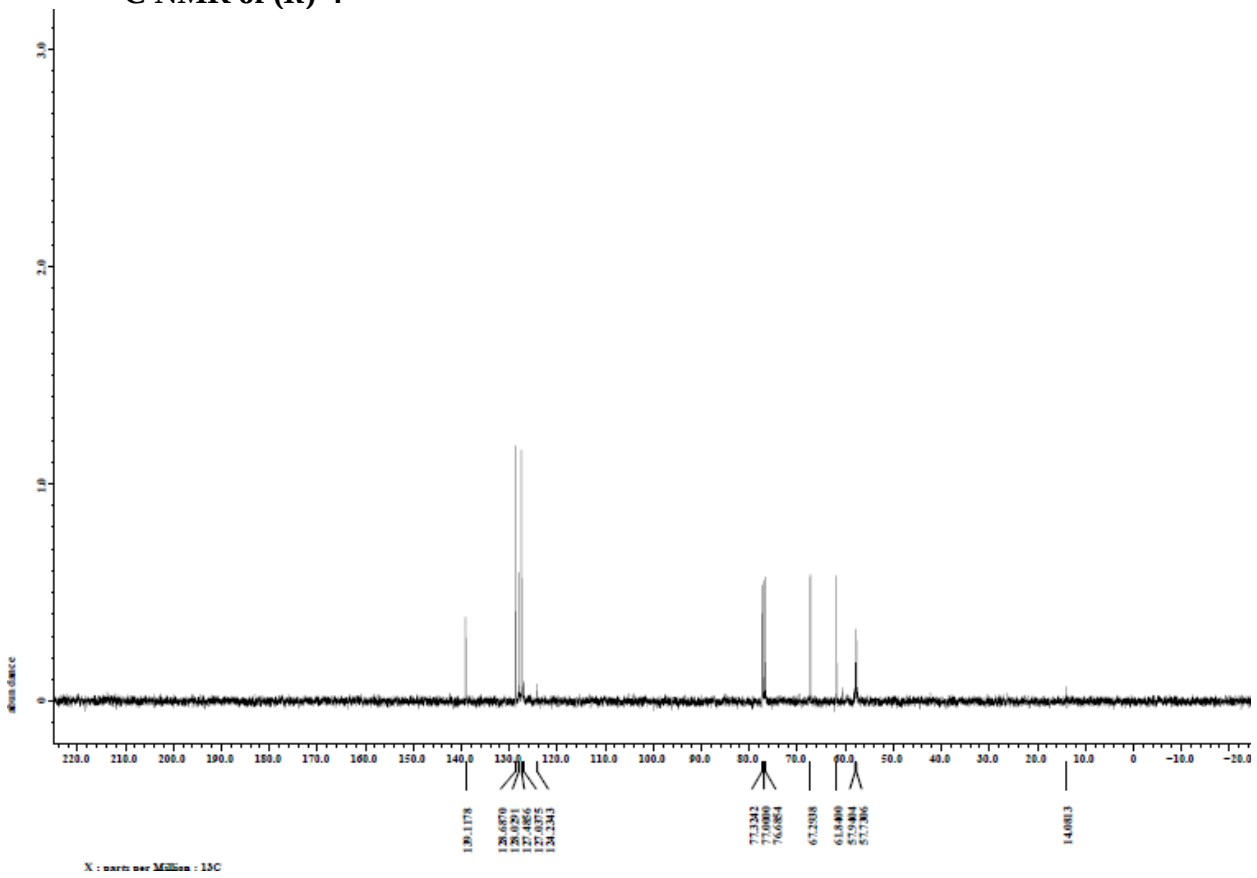
^{19}F NMR: crude mixture of (*R*)-4 and (*S*)-4 from oxazolidine 6



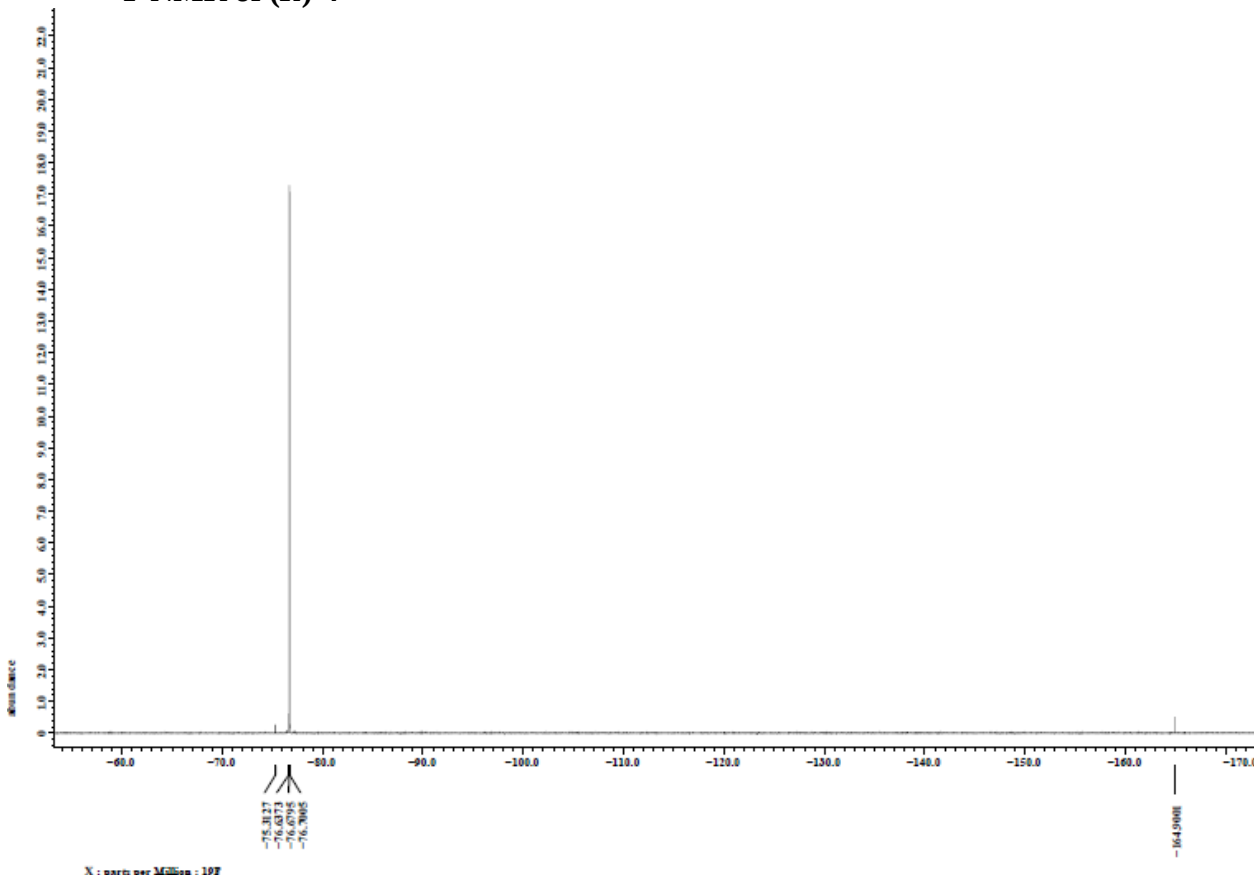
^{1}H NMR of (*R*)-4



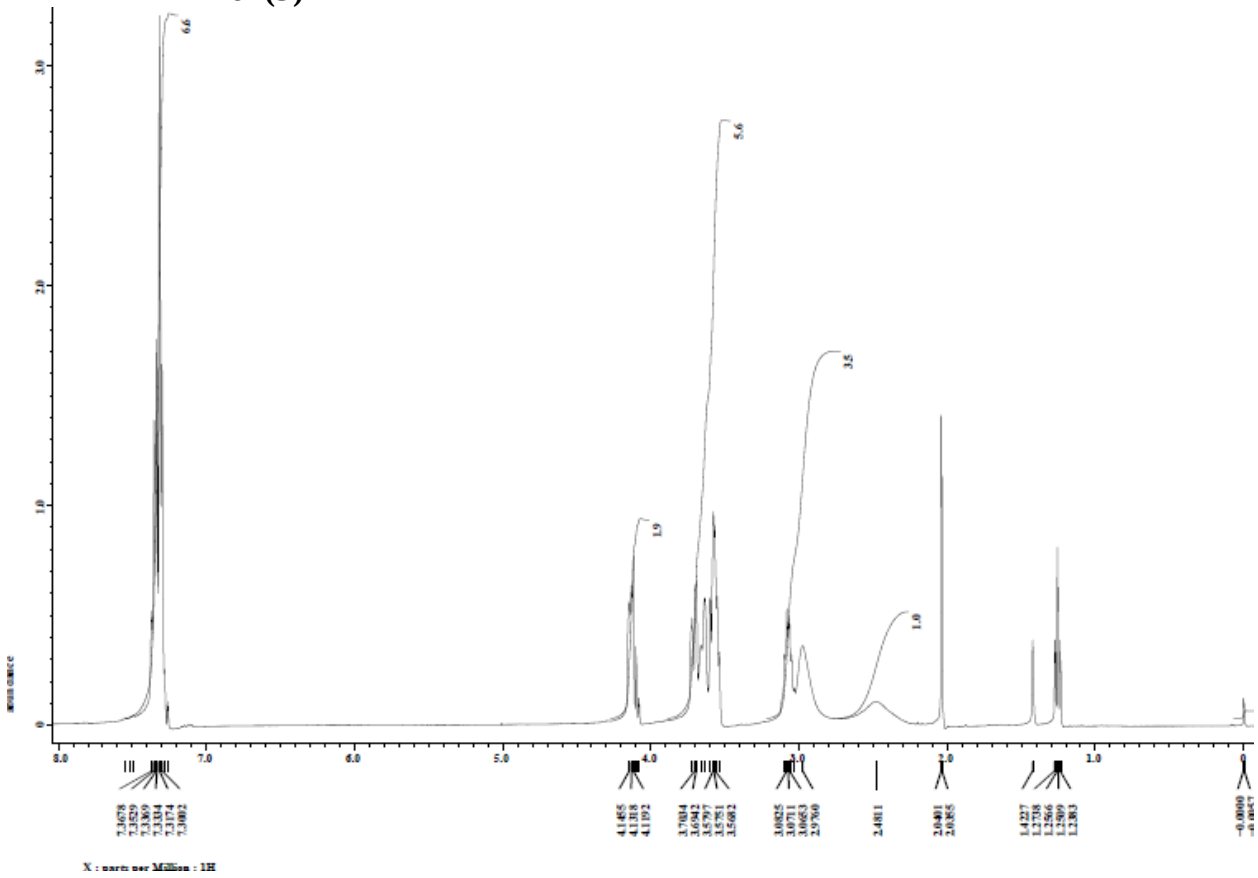
^{13}C NMR of (*R*)-4



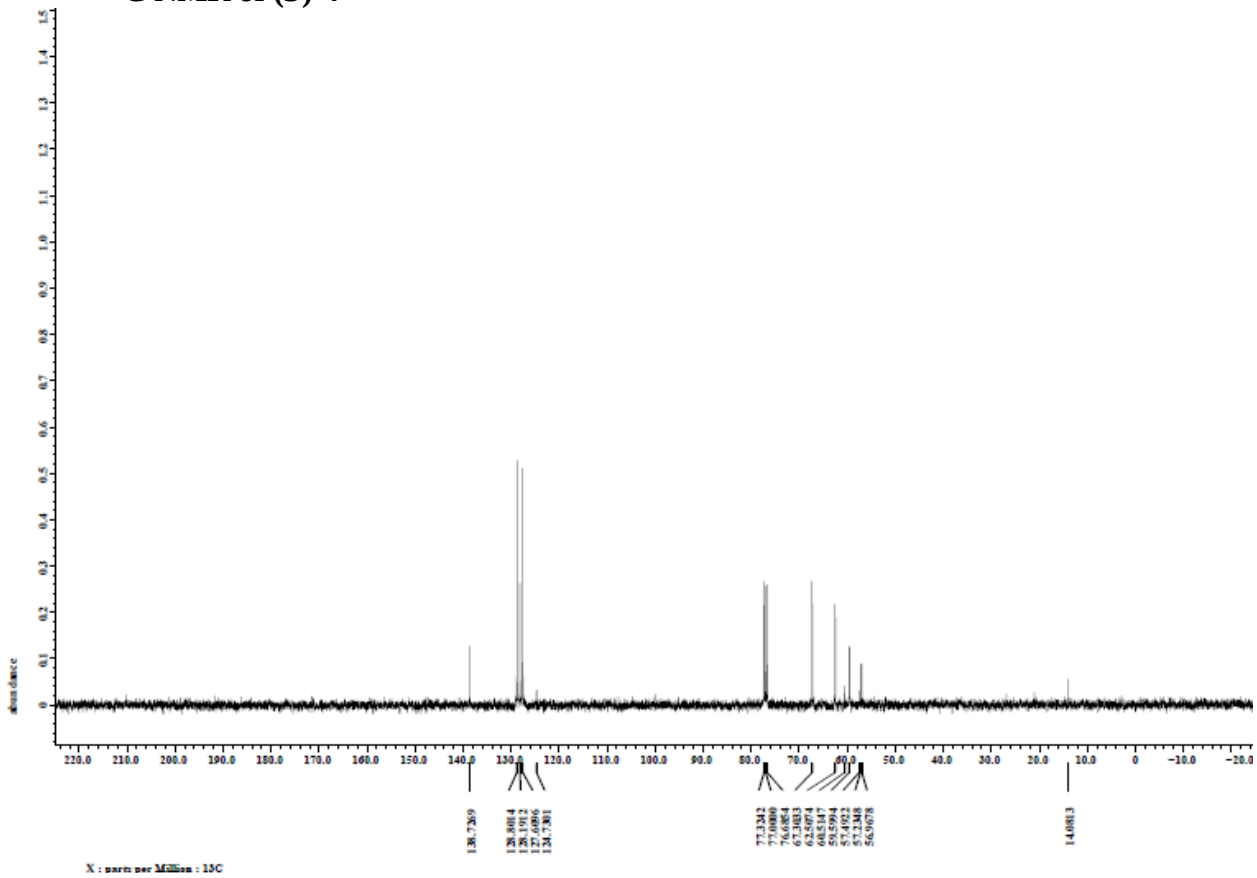
^{19}F NMR of (*R*)-4



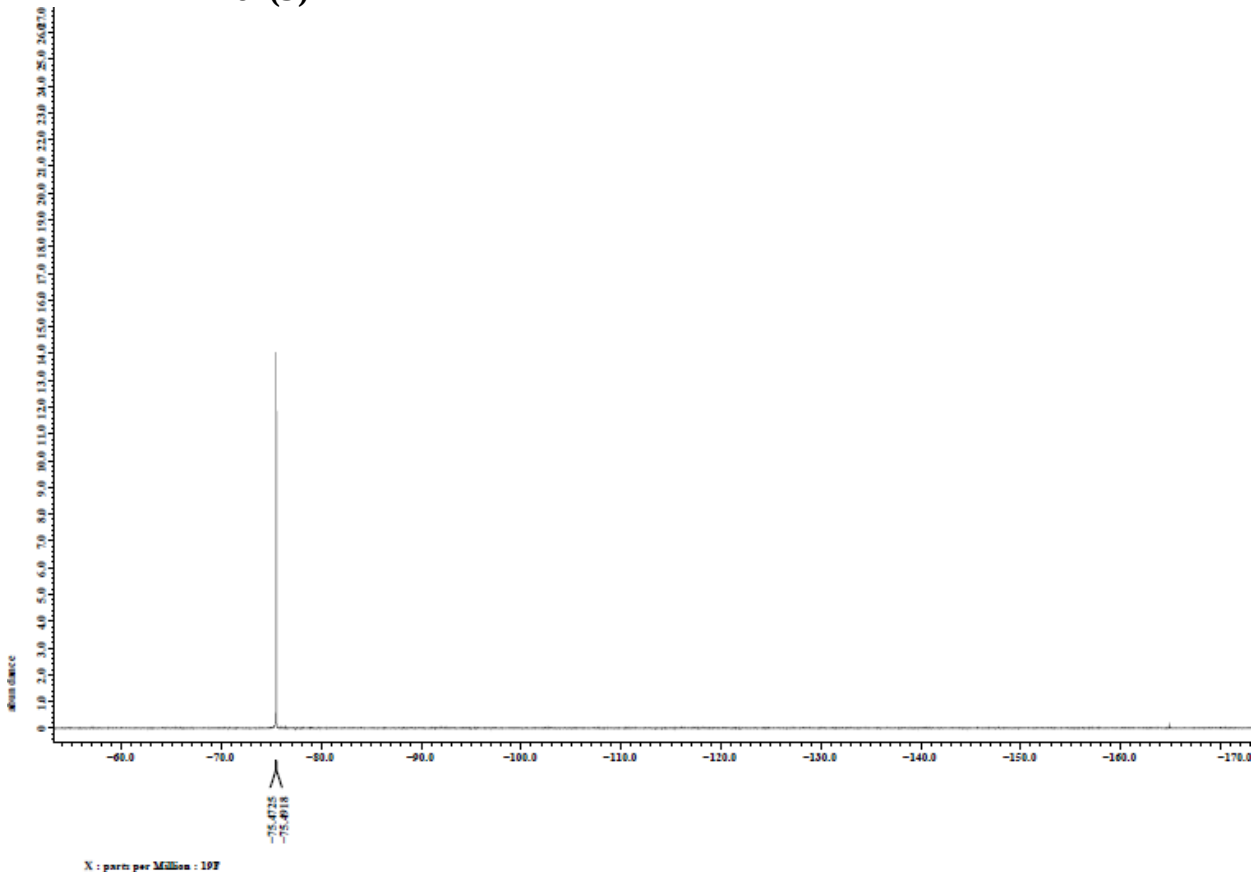
^{1}H NMR of (*S*)-4



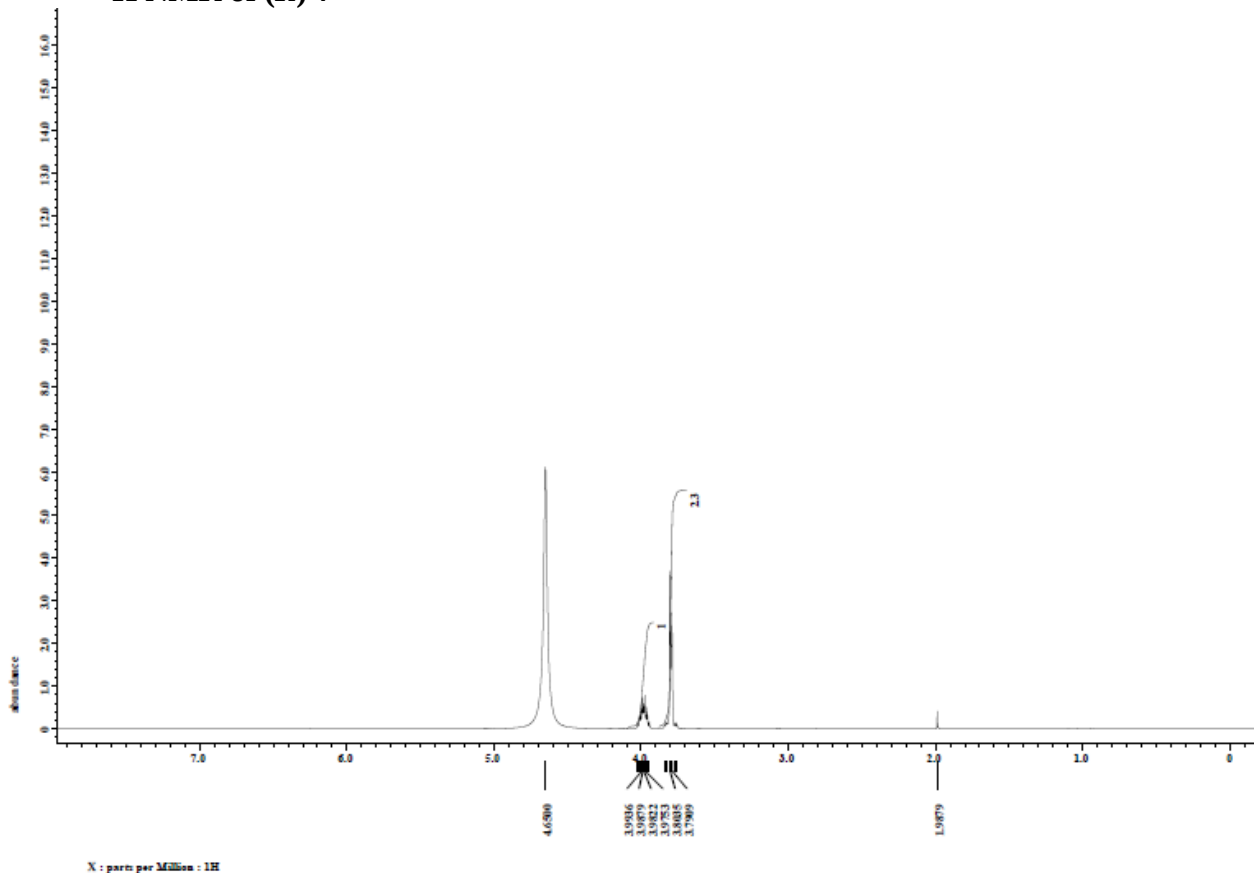
^{13}C NMR of (*S*)-4



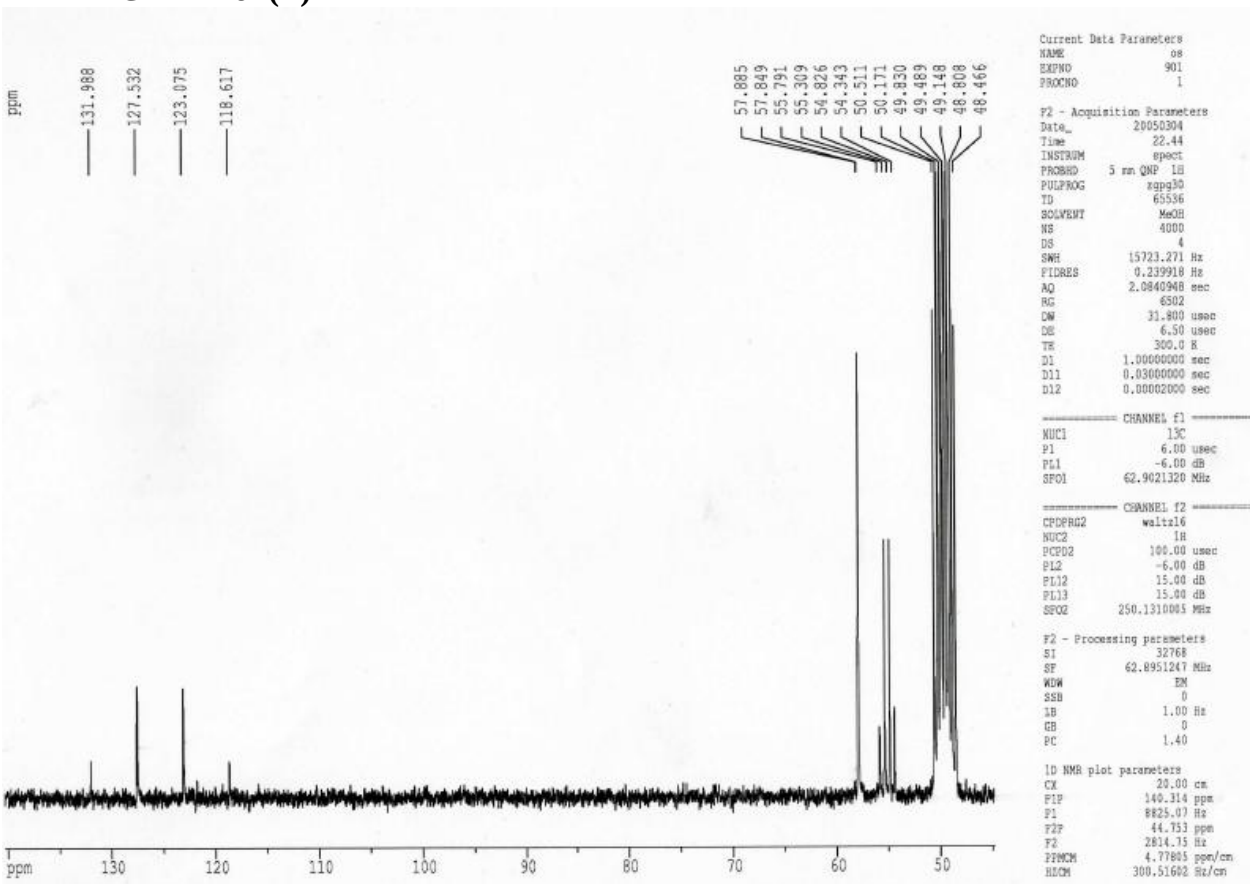
^{19}F NMR of (*S*)-4



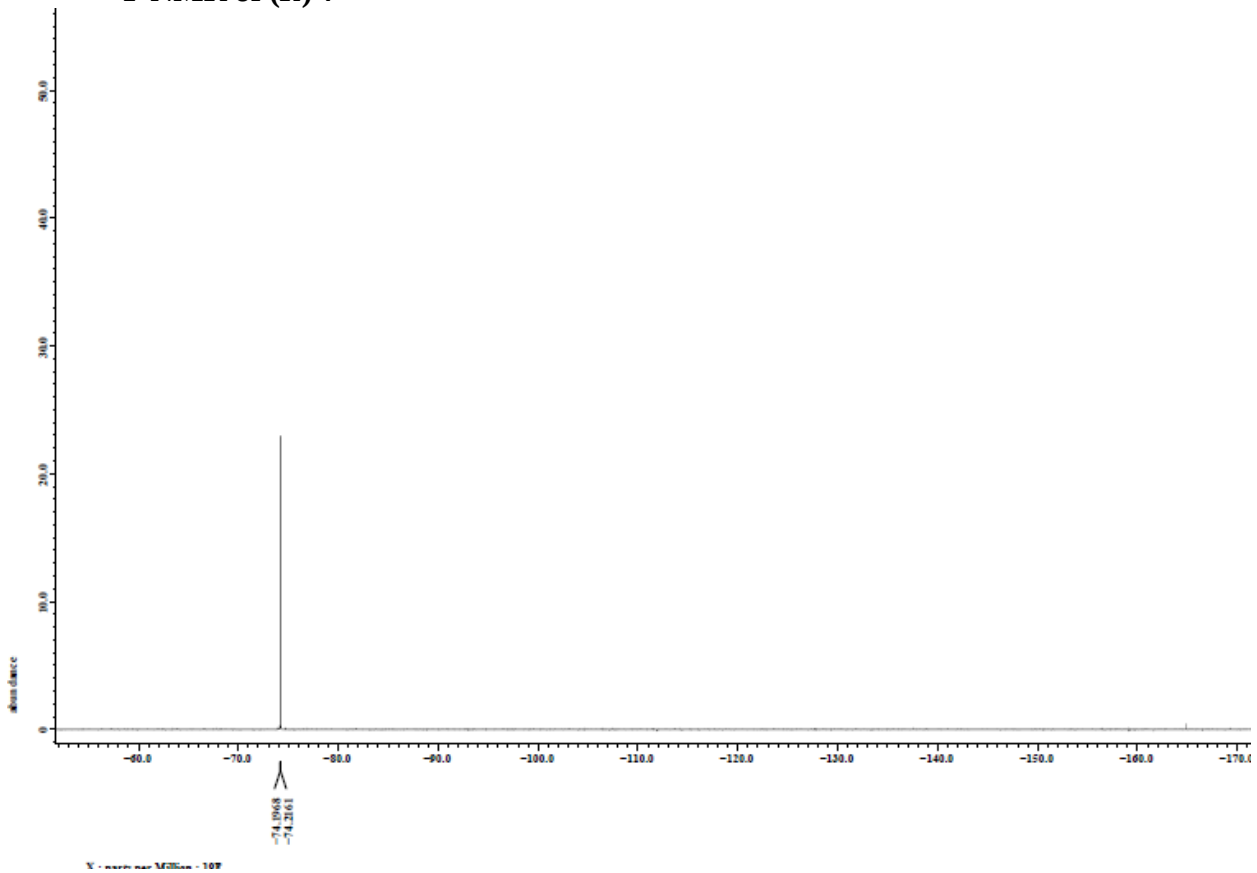
^{1}H NMR of (*R*)-7



^{13}C NMR of (*R*)-7



^{19}F NMR of (*R*)-7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-140.0
-150.0
-160.0
-74.1968
-74.2161