First Asymmetric Synthesis of Orthoquinone Monoketal Enantiomers via Anodic Oxidation

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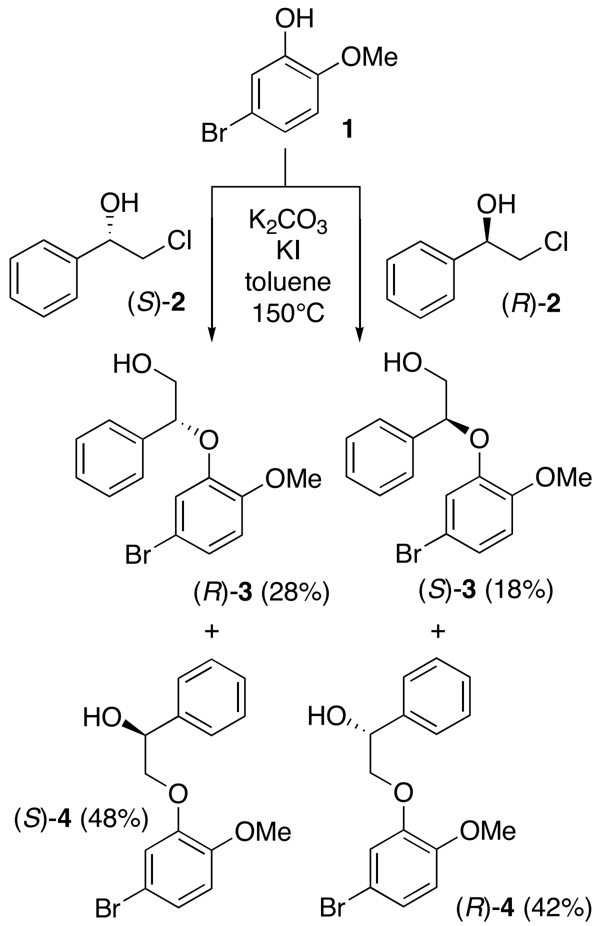
Supporting Information

Experimental Section

General Procedure. Acetonitrile (MeCN), dichloromethane (CH_2Cl_2), diethyl ether (Et_2O), and methanol (MeOH) were distilled under N_2 immediately before use from P_2O_5, CaH_2, and $CaCl_2$, respectively. Tetrahydrofuran (THF) was purified by distillation from sodium/benzophenone under N_2 immediately before use. Synthesis grade ethyl acetate was used as received. Light petroleum refers to the fraction boiling in the 40-60 °C boiling range. Moisture and oxygen sensitive reactions were carried out in flame-dried glassware under N_2. Evaporations were conducted under reduced pressure at temperatures less than 45°C unless otherwise noted. Column chromatography was carried out under positive pressure using 40-60 μm silica gel (Merck) and the indicated solvents. Further drying of the residues was accomplished under high vacuum. Melting points are uncorrected. NMR spectra of samples in the indicated solvent were run at either 200, 250 or 300 MHz. Carbon multiplicities and stereochemical assignments were determined by DEPT135 and NOESY experiments, respectively. Electron impact (50-70 eV) and liquid secondary ion mass spectrometry low- and high-resolution data (EIMS, and LSIMS, HRMS) were obtained from the mass spectrometry laboratory of the CESAMO at the University of Bordeaux, Talence, France. Atmospheric pressure chemical ionization (APCI) mass spectrometry fragmentation data and MALDI-ToF high-resolution data (APCIMS and MALDI, HRMS) were obtained from the mass spectrometry laboratory at the European Institute of Chemistry and Biology, Pessac, France.

Preparation of the chiral alcohols 3 and 4. The Williamson-type etherification reaction between 5-bromoguaiacol (**1**) and (*S*)-2-chloro-1-phenylethanol (**2**) or its enantiomer *R*-(**2)** furnished mixtures of primary and secondary alcohol enantiomers in good yields (see Scheme below), probably via the styrene oxide intermediates derived from *S*-(**2)** or *R*-(**2)** – Both styrene oxides are commercially available under enantiomerically pure forms at lower costs than those of the chloroethanols **2**, but lower yields and lower regioselectivity were observed in our first series of attempts to use them – Chromatographic separation of the mixtures afforded *R*-(**3)** and *S*-(**4)** in 28% and 48% yields from *S*-(**2)**, and *S*-(**3)** and *R*-(**4)** in 18% and 42% yields from *R*-(**2)**. The lack of better regioselectivity was somewhat disappointing, but the facility with which the primary and secondary alcohol products were separated offered us a convenient access to two pairs of enantiomers as starting materials for asymmetric anodic oxidation.

5-Bromoguaiacol (1). A 39% solution of peracetic acid in acetic acid (30 mL, 446 mmol) was added dropwise to a stirring ice-cooled solution of 5-bromo-2-methoxybenzaldehyde (15.0 g, 70.0 mmol) in EtOAc (160 mL). The reaction mixture was allowed to warm up slowly to room temperature while stirred overnight, after which time it was diluted in H_2O (80 mL). After separation, the aqueous layer was extracted with EtOAc (3 × 20 mL), and the combined organic layers were washed with saturated aqueous $NaHCO_3$ (3 × 10 mL), dried over Na_2SO_4, filtered and evaporated. The yellow residue was dissolved in MeOH (160 mL), then KOH pellets (4.12 g, 73.6 mmol) were added portionwise at 0°C under stirring. The reaction mixture was stirred at 0°C for an additional 30 min, after which time it was diluted in H_2O (80 mL), slowly acidified with 10% aqueous HCl, extracted with EtOAc (3 × 20 mL), washed with brine (2 × 10 mL), dried over Na_2SO_4, filtered and evaporated to give crude **1** as a yellow oil, which was purified by column chromatography, eluting with light petroleum/Et_2O (6:1 → 3:1) to furnish **1** (9.2 g, 65%) as an off-white solid. All characterization data were identical to those previously reported.[1,2]



2-(5-Bromo-2-methoxyphenoxy)-(2*R*)-phenylethanol [(*R*)-3] and 2-(5-Bromo-2-methoxyphenoxy)-(1*S*)-phenylethanol [(*S*)-4]. To a stirred ice-cold solution of bromoguaiacol (**1,** 1.39 g, 6.9 mmol) in toluene (30 mL) were added powdered potassium carbonate (4.60 g, 33.3 mmol) and potassium iodine (459 mg, 2.7 mmol). After stirring for 15 min, (*S*)-(+)-2-chloro-1-phenylethanol [*S*-(**2**), 1.42 g, 9.1 mmol] was added. The reaction mixture was refluxed for 5 days, after which time toluene was evaporated. The residue was taken up with water (20 mL) and CH_2Cl_2 (30 mL). After separation, the aqueous phase was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and evaporated to give a crude mixture of (*R*)-**3** and (*S*)-**4,** which was submitted to column chromatography, eluting with light petroleum/Et_2O (3:2), to furnish (*R*)-**3** (622 mg, 28 %) and (*S*)-**4** (1.07 g, 48 %). (*R*)-**3**:

$[\alpha]^{21}_D$ = -44.68° (c = 0.82, CH_2Cl_2); IR (NaCl) 3449, 3053, 2920, 1588, 1502 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 3.11 (bs, 1H), 3.75-3.99 (m, 2H), 3.87 (s, 3H), 5.10 (dd, *J* = 8.5, 3.4 Hz, 1H), 6.75 (d, *J* = 8.5 Hz, 1H), 6.85 (d, *J* = 2.2 Hz, 1H), 7.04 (dd, *J* =8.7, 2.3 Hz, 1H), 7.32-7.39 (m, 5H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 149.6, 148.3, 137.4, 128.7, 128.4, 126.3, 125.2, 120.9, 113.1, 112.5, 85.0, 67.3, 56.0; EIMS *m/z* (rel intensity) 324 (M^+, 4), 322 (M^+, 4), 293 (2), 291 (2), 277 (2), 275 (2), 204 (94), 202 (100), 189 (20), 187 (22); HRMS (EIMS) calcd for $C_{15}H_{15}O_3Br$ 322.0206 found 322.0201. (*S*)-**4**: $[\alpha]^{21}_D$ = +14.02° (c = 1.07, CH_2Cl_2); IR (NaCl) 3402, 3054, 2990, 1508, 1420 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 3.78 (s, 3H), 3.98 (bt, *J* = 9.5 Hz, 1H), 4.11 (dd, *J* = 9.8, 2.8 Hz, 1H), 5.14 (dd, *J* = 8.9, 2.8 Hz, 1H), 6.73 (d, *J* = 8.6 Hz, 1H), 7.00 (dd, *J* = 1.8 Hz, 1H), 7.05 (dd, *J* = 8.5, 2.1 Hz, 1H), 7.32-7.46 (m, 5H); ^{13}C NMR ($CDCl_3$, 50.3 MHz) δ 149.0, 148.6, 139.3, 128.5, 128.1, 126.2, 124.6, 118.2, 113.0, 111.8, 75.7, 72.2, 56.0; EIMS *m/z* (rel intensity) 324 (M^+, 20), 322 (M^+, 20), 218 (20), 216 (20), 204 (92), 202 (100), 189 (26), 187 (27); HRMS (EIMS) calcd for $C_{15}H_{15}O_3Br$ 322.0206 found 322.0208.

2-(5-Bromo-2-methoxy-phenoxy)-(2*S*)-phenylethanol [(*S*)-3] and 2-(5-Bromo-2-methoxy-phenoxy)-(1*R*)-phenylethanol [(*R*)-4]. To a stirred ice-cold solution of bromoguaiacol (**1,** 1.51 g, 7.5 mmol) in toluene (50 mL) were added powdered potassium carbonate (4.49 g, 32.5 mmol) and potassium iodine (626 mg, 3.8 mmol). After stirring for 15 min, (*R*)-(–)-2-chloro-1-phenylethanol [*R*-(**2**), 1.19 g, 7.6 mmol] was added. The reaction mixture was refluxed for 5 days, after which time toluene was evaporated. The residue was taken up with water (20 mL) and CH_2Cl_2 (30 mL). After separation, the aqueous phase was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and evaporated to give a crude mixture of (*S*)-**3** and (*R*)-**4,** which was submitted to column chromatography, eluting with light petroleum/Et_2O (3:2), to furnish (*S*)-**3** (413 mg, 18 %) and (*R*)-**4** (1.02 g, 42 %). (*S*)-**3**: $[\alpha]^{21}_D$ = +43.02° (c = 0.86, CH_2Cl_2); IR (NaCl) 3373, 2943, 2830, 1467, 1420 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 3.32 (bs, 1H), 3.84 (s, 3H), 3.74-4.00 (m, 2H), 5.14 (dd, *J* = 8.4, 2.9 Hz, 1H), 6.73 (d, *J* = 8.5 Hz, 1H), 6.85 (s, 1H), 7.02 (d, *J* = 8.9 Hz, 1H), 7.33-7.37 (m, 5H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 149.4, 148.2, 137.3, 128.6, 128.3, 126.2, 125.0, 120.4, 112.9, 112.4, 84.5, 67.2, 55.9; EIMS *m/z* (rel intensity) 324 (M^+, 17), 322 (M^+, 18), 218 (26), 216 (27), 204 (90), 202 (100), 189 (26), 187 (27); HRMS

(EIMS) calcd for $C_{15}H_{15}O_3Br$ 322.0206 found 322.0195. (*R*)-**4**: $[\alpha]^{21}_D$ = -20.49° (c = 1.22, CH_2Cl_2); IR (NaCl) 3304, 2946, 2827, 1450 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 3.81 (s, 3H), 3.98 (bt, *J* = 9.5 Hz, 1H), 4.12 (dd, *J* = 9.6, 2.6 Hz, 1H), 5.14 (dd, *J* = 9.0, 2.6 Hz, 1H), 6.74 (d, *J* = 8.6 Hz, 1H), 7.01 (s, 1H), 7.07 (d, *J* =8.6 Hz, 1H), 7.32-7.46 (m, 5H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 148.8, 148.6, 139.3, 128.4, 128.0, 126.2, 124.5, 117.9, 112.9, 112.6, 75.5, 72.1, 55.9; EIMS *m/z* (rel intensity) 324 (M^+, 3), 322 (M^+, 3), 293 (2), 291 (2), 204 (97), 202 (100), 189 (27), 187 (28); HRMS (EIMS) calcd for $C_{15}H_{15}O_3Br$ 322.0206 found 322.0201.

General Procedure for Anodic Oxidation. Electrolyses were carried out in a 100 mL divided cylindrical cell, equipped with a platinum grid as the anode and a platinum wire as the cathode. KOH pellets (1.1 g, 19.6 mmol) was added as a supporting electrolyte into 100 mL of methanol. The starting alcohol was introduced into the anodic compartment, and the electrolysis was then performed on an Autolab PGSTAT 100 potentiostat using an Ag/AgCl reference electrode. Electrolysis was carried out, under vigorous stirring, at a constant current of *ca*. 100 mA, which was maintained at this value by increasing gradually the potential until TLC monitoring indicated the complete disappearance of the starting alcohol. The reaction mixture was then evaporated, and the residue was taken up in CH_2Cl_2 (50 mL) and H_2O (20 mL). After separation, the aqueous layer was extracted with CH_2Cl_2 (3 × 30 mL) and the combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and evaporated.

9-Bromo-6,6-dimethoxy-(2*R*)-phenyl-1,4-dioxaspiro[4.(5*S*)]deca-7,9-diene [(*RS*)-5]. Anodic oxidation of a solution of the primary alcohol (*R*)-**3** (194 mg, 0.6 mmol) in MeOH was performed according to the general procedure described above. Electrolysis was carried out at 100 mA by increasing potential from 2.80 to 3.90 V/Ag/0.1 M AgCl. The reaction mixture was then processed as described above, and the residue was further dried overnight to give an oily crude product, which was purified by column chromatography, eluting with light petroleum/Et_2O (3:2), to furnish the pure *spiro*-bisketal (*RS*)-**5** as a yellow oil (71 mg, 34 %): $[\alpha]^{21}_D$ = -6.67° (c = 0.48, CH_2Cl_2); IR (NaCl) 2929, 2850, 1749, 1501 cm^{-1}; ^1H NMR ($CDCl_3$, 200 MHz) δ 3.45 (s, 3H), 3.47 (s, 3H), 3.82-3.88 (m, 1H), 4.40 (bt, *J* = 7.2 Hz, 1H), 5.22 (dd, *J* = 9.0, 6.7 Hz, 1H), 5.89 (d, *J* =10.3 Hz, 1H), 6.11 (dd, $J = 10.3, 1.6$ Hz, 1H), 6.35 (d, $J = 1.4$ Hz, 1H), 7.34-7.44 (m, 5H); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 136.6, 132.3, 131.5, 130.4, 128.6, 128.5, 126.4, 119.0, 108.5, 97.0, 78.2, 71.1, 51.2, 51.0; EIMS *m/z* (rel intensity) 354 (M^+, 2), 352 (M^+, 2), 273 (2), 250 (18), 248 (21); HRMS (EIMS) calcd for $C_{16}H_{17}O_4Br$ 352.0310 found 352.0302.

9-Bromo-6,6-dimethoxy-(2*S*)-phenyl-1,4-dioxaspiro[4.(5*R*)]deca-7,9-diene [(*SR*)-5]. Anodic oxidation of a solution of the primary alcohol (*S*)-**3** (278 mg, 0.9 mmol) in MeOH was performed according to the general procedure described above. Electrolysis was carried at 110 mA by increasing potential from 1.90 to 2.20 V/Ag/0.1 M AgCl. The reaction mixture was then processed as described above, and the residue was further dried overnight to give an oily crude product, which was purified by column chromatography, eluting with light petroleum/Et_2O (3:2→1:1), to furnish the pure *spiro*-bisketal (*SR*)-**5** as a yellow oil (83 mg, 27 %): $[\alpha]^{21}_D = +0.88°$ (c = 0.68, CH_2Cl_2); IR (NaCl) 2938, 2844, 1694, 1206 cm^{-1}; ^{1}H NMR ($CDCl_3$, 250 MHz) δ 3.45 (s, 3H), 3.47 (s, 3H), 3.81 (bt, $J = 8.2$ Hz, 1H), 4.40 (bt, $J = 7.0$ Hz, 1H), 5.22 (dd, $J = 9.2, 6.7$ Hz, 1H), 5.89 (d, $J = 10.1$ Hz, 1H), 6.11 (dd, $J = 10.4, 1.5$ Hz, 1H), 6.35 (d, $J = 1.5$ Hz, 1H), 7.31-7.43 (m, 5H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 136.5, 132.2, 131.4, 130.3, 128.5, 128.4, 126.3, 118.9, 108.4, 96.9, 78.1, 71.0, 51.1, 50.9; EIMS *m/z* (rel intensity) 354 (M^+, 3), 352 (M^+, 3), 273 (4), 250 (20), 248 (23); HRMS (EIMS) calcd for $C_{16}H_{17}O_4Br$ 352.0310 found 352.0304.

9-Bromo-6,6-dimethoxy-(2R)-phenyl-1,4-dioxaspiro[4.(5R)]deca-7,9-diene [(*RR*)-5]. Anodic oxidation of a solution of the secondary alcohol (*R*)-**4** (285 mg, 0.9 mmol) in MeOH was performed according to the general procedure described above. Electrolysis was carried out at 100 mA by increasing potential from 1.40 to 2.40 V/Ag/0.1 M AgCl. The reaction mixture was then processed as described above, and the residue was further dried overnight to give an oily crude product, which was purified by column chromatography, eluting with light petroleum/Et_2O (1:1), to furnish the pure *spiro*-bisketal (*RR*)-**5** as a yellow oil (47 mg, 15 %): $[\alpha]^{21}_D = -20.6°$ (c = 1.40, CH_2Cl_2); IR (NaCl) 2937, 2848, 1786, 1651, 1463, 1384 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.45 (s, 3H), 3.51 (s, 3H), 3.71-3.89 (m, 1H + *impurities integrating for about 1H*), 4.52 (bt, $J = 7.4$ Hz, 1H), 5.25 (dd, $J = .8.5, 6.6$ Hz, 1H), 5.91 (d, $J = 10.2$ Hz, 1H), 6.14 (d, $J = 10.2$ Hz, 1H), 6.27 (s, 1H), 7.35

(bs, 5H); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 138.0, 132.5, 130.8, 128.6, 128.3, 126.2, 119.0, 109.2, 97.6, 79.0, 72.1, 51.4, 51.1; EIMS *m/z* (rel intensity) 354 (M^+, 4), 352 (M^+, 4), 273 (22), 250 (20), 248 (20); HRMS (EIMS) calcd for $C_{16}H_{17}O_4Br$ 352.0310 found 352.0300.

9-Bromo-6,6-dimethoxy-(2*S*)-phenyl-1,4-dioxaspiro[4.(5*S*)]deca-7,9-diene [(*SS*)-5]. Anodic oxidation of a solution of the secondary alcohol (*S*)-**4** (416 mg, 1.3 mmol) in MeOH was performed according to the general procedure described. Electrolysis was carried out a 100 mA by increasing potential from 1.90 to 2.90 V/Ag/0.1 M AgCl. The reaction mixture was then processed as described above, and the residue was further dried overnight to give an oily crude product, which was purified by column chromatography, eluting with light petroleum/Et_2O (4:1→3:2), to furnish the pure *spiro*-bisketal (*SS*)-**5** as a yellow oil (65 mg, 14 %): $[\alpha]^{21}_D$ = +31.5° (c = 1.00, CH_2Cl_2); IR (NaCl) 2937, 2848, 1786, 1651, 1463, 1384 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 3.45 (s, 3H), 3.51 (s, 3H), 3.76-3.88 (m, 1H 1H + *impurities integrating for about 1H*), 4.52 (dd, *J* = 8.5, 6.4 Hz, 1H), 5.25 (dd, *J* = 8.5, 6.4 Hz, 1H), 5.90 (d, *J* = 10.4 Hz, 1H), 6.14 (dd, *J* = 10.4, 1.5 Hz, 1H), 6.27 (d, *J* = 1.8 Hz 1H), 7.35 (bs, 5H); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 138.0, 132.5, 130.8, 128.6, 128.3, 126.2, 119.0, 109.1, 97.5, 78.9, 72.1, 51.4, 51.1; EIMS *m/z* (rel intensity) 354 (M^+, 0.5), 352 (M^+,0.6), 273 (3), 250 (14), 248 (14); HRMS (EIMS) calcd for $C_{16}H_{17}O_4Br$ 352.0310 found 352.0310.

9-Bromo-(2*R*)-phenyl-1,4-dioxaspiro[4.(5*S*)]deca-7,9-dien-6-one [(*RS*)-6]. Selective monohydrolysis of the *spiro*-bisketal (*RS*)-**5** (37 mg, 0.1 mmol) in Et_2O (5 mL) was carried out at 0°C by treatment with TFA (3 mL) and water (1 mL). The reaction mixture was stirred for 1 h, after which time it was neutralized with $NaHCO_3$ (3 × 10 mL), dried over Na_2SO_4, filtered, evaporated and submitted to column chromatography, eluting with CH_2Cl_2, to give the orthoquinone *spiro*-monoketal (*RS*)-**6** as a yellow oil (17 mg, 53%): $[\alpha]^{21}_D$ + 6.8° (c = 0.43, $CHCl_3$); IR (NaCl) 2926, 1740, 1659, 1636 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 4.21 (bt, *J* = 8.9 Hz, 1H), 4.43 (dd, *J* = 8.1, 5.7 Hz, 1H), 5.17 (dd, *J* = 9.8, 5.5 Hz, 1H), 6.00 (d, *J* = 10.4 Hz, 1H), 6.55 (d, *J* = 1.9 Hz, 1H), 6.92 (dd, *J* = 10.2, 2.0 Hz, 1H), 7.37-7.53 (m, 5H); ^{13}C NMR ($CDCl_3$, 50.3 MHz) δ 196.1, 143.8, 137.1, 135.4, 128.8, 128.6, 127.4, 125.5, 119.9, 97.8, 80.1, 72.7; MALDI *m/z* 306 (M^+), 308 (M^+); HRMS (MALDI) calcd for $C_{14}H_{11}O_3Br$ 306.9969 found 306.9835.

9-Bromo-(2*S*)-phenyl-1,4-dioxaspiro[4.(5*R*)]deca-7,9-dien-6-one [(*SR*)-6]. Selective monohydrolysis of the *spiro*-bisketal (*SR*)-**5** (82 mg, 0.2 mmol) in Et_2O (10 mL) was carried out at 0°C by treatment with TFA (6 mL) and water (2 mL). The reaction mixture was stirred for 1 h, after which time it was neutralized with $NaHCO_3$ (3 × 10 mL), dried over Na_2SO_4, filtered, evaporated and submitted to column chromatography, eluting with CH_2Cl_2, to give the orthoquinone *spiro*-monoketal (*SR*)-**6** as a yellow oil (41 mg, 60%): $[\alpha]^{21}_D$ – 7.6° (c = 0.52, $CHCl_3$); IR (NaCl) 2927, 2848, 1792, 1695, 1628 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 4.20, (bt, *J* = 9.0 Hz, 1H), 4.43 (dd, *J* = 8.2, 5.5 Hz, 1 H), 5.16 (dd, *J* = 9.8, 5.8 Hz, 1H), 6.00 (d, *J* = 10.4 Hz, 1H), 6.55 (d, *J* = 2.4 Hz, 1H), 6.92 (dd, *J* = 10.4, 2.4 Hz, 1H), 7.36-7.53 (m, 5H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 196.1, 143.8, 137.1, 135.4, 128.8, 128.6, 127.4, 125.5, 119.9, 97.8, 80.1, 72.7; MALDI *m/z* 306 (M^+), 308 (M^+); HRMS (MALDI) calcd for $C_{14}H_{11}O_3Br$ 306.9969 found 306.9776.

9-Bromo-(2*R*)-phenyl-1,4-dioxaspiro[4.(5*R*)]deca-7,9-dien-6-one [(*RR*)-6]. Selective monohydrolysis of the *spiro*-bisketal (*RR*)-**5** (94 mg, 0.3 mmol) in Et_2O (10 mL) was carried out at 0°C by treatment with TFA (6 mL) and water (2 mL). The reaction mixture was stirred for 1 h, after which time it was neutralized with $NaHCO_3$ (3 × 10 mL), dried over Na_2SO_4, filtered, evaporated and submitted to column chromatography, eluting with CH_2Cl_2, to give the orthoquinone *spiro*-monoketal (*RR*)-**6** as a yellow oil (44 mg, 49%): $[\alpha]^{21}_D$ – 14.2° (c = 1.09, $CHCl_3$); IR (NaCl) 2929, 2852, 1731, 1681, 1652 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 3.83-3.96 (m, 1H + *impurities integrating for about 1H*), 4.64 (bt, *J* = 7.2 Hz, 1 H), 5.58 (bt, *J* = 6.9 Hz, 1H), 5.97 (d, *J* = 10.4 Hz, 1H), 6.58 (d, *J* = 2.1 Hz, 1H), 6.90 (dd, *J* = 10.2, 2.3 Hz, 1H), 7.37 (bs, 5H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 195.9, 143.7, 137.6, 136.3, 128.7, 128.6, 126.4, 125.5, 120.7, 98.7, 79.1, 72.3; APCIMS/MS *m/z* 309 (MH^+, 100), 307 (MH^+, 96), 227 (13), 189 (92), 187 (100); HRMS (MALDI) calcd for $C_{14}H_{11}O_3Br$ 306.9969 found 306.9817.

9-Bromo-(2*S*)-phenyl-1,4-dioxaspiro[4.(5*S*)]deca-7,9-dien-6-one [(*SS*)-6]. Selective monohydrolysis of the *spiro*-bisketal (*SS*)-**5** (65 mg, 0.2 mmol) in Et_2O (10 mL) was carried out at 0°C by treatment with TFA (6 mL) and water (2 mL). The reaction mixture was stirred for 1 h, after which time it was neutralized with $NaHCO_3$ (3 × 10 mL), dried over Na_2SO_4, filtered, evaporated and submitted to column chromatography, eluting with CH_2Cl_2, to give the orthoquinone *spiro*-monoketal (*SS*)-**6** as a yellow oil (25 mg, 43%): $[\alpha]^{21}_D$ + 7.9° (c = 1.23, $CHCl_3$); IR (NaCl) 2929, 2852, 1731, 1681, 1652 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 3.83-3.96 (m, 1H + *impurities integrating for about 1H*), 4.64 (bt, *J* = 7.3 Hz, 1 H), 5.58 (bt, *J* = 6.7 Hz, 1H), 5.97 (d, *J* = 10.1 Hz, 1H), 6.58 (s, 1H), 6.90 (dd, *J* = 10.4, 2.1 Hz, 1H), 7.37 (bs, 5H); ^{13}C NMR ($CDCl_3$, 50.3 MHz) δ 195.9, 143.7, 137.7, 136.4, 128.7, 128.6, 126.4, 125.6, 120.7, 98.8, 79.1, 72.4; MALDI *m/z* 306 (M^+), 308 (M^+); HRMS (MALDI) calcd for $C_{14}H_{11}O_3Br$ 306.9969 found 306.9751.

NB: The four enantiopure orthoquinone *spiro*-monoketals described above are stable in 0.02 M solution in Et_2O. Their 4-bromine substituent do retard dimerization, but they still tend to dimerize at room temperature upon standing as oily residues and in concentrated solutions. The fact that no or very little racemization occurred at the *spiro*-center during monohydrolysis was clearly evidenced by ^1H NMR analysis, since the diastereoisomers (*RR*)- and (*RS*)-**6**, as well as their enantiomeric counterparts, display different ^1H NMR characteristics. Their optical rotations are in agreement with this analysis (*vide supra*). Examination of further synthetic transformations of these enantiopure orthoquinone monoketals was performed only using the (*RS*)-**6** enantiomer (*vide infra*).

Cyclohexa-2,4-dienols 7. $LiAlH_4$ (17 mg, 0.5 mmol) and LiBr (50 mg, 0.6 mmol) were dried beforehand at 140°C for 3 h under reduced pressure.[3] After cooling, a solution of (*RS*)-**6** (26 mg, 0.09 mmol) in anhydrous Et_2O (5mL) was then added and the resulting mixture was stirred at -78°C for 1 h, after which time the excess reducing agent was quenched with saturated aqueous NH_4Cl. The mixture was diluted in H_2O (5mL), separated and the aqueous layer was extracted with Et_2O (3 × 10 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered and evaporated to give a residue, which was submitted to column chromatography, eluting with light petroluem/Et_2O (1:1), to afford **7** as diastereoisomeric mixture (10 mg, 38%) in *ca.* 30% *de*: IR

(NaCl) 3397, 2926, 1490, 1444 cm^{-1}; diastereoisomer I: ^1H NMR ($CDCl_3$, 250 MHz) δ 3.79-4.05 (m, 3H), 5.04 (dd, J = 7.6, 3.4 Hz, 1H), 6.79-7.04 (m, 3H), 7.38-7.40 (m, 5H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 145.9, 136.7, 128.9, 128.8, 126.3, 126.2, 120.5, 117.2, 110.9, 84.5, 77.2, 66.9; EIMS *m/z* (rel intensity) 310 (M^+, 5), 308 (M^+, 5), 190 (79), 188 (82), 121 (100); diastereoisomer II: ^1H NMR ($CDCl_3$, 250 MHz) δ 4.09-4.18 (m, 3H), 5.13 (dd, J = 8.4, 3.2 Hz, 1H), 6.79-7.04 (m, 3H), 7.38-7.40 (m, 5H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 146.5, 139.2, 129.0, 128.7, 126.3, 126.2, 125.5, 116.9, 111.2, 84.5, 74.8, 72.9; EIMS *m/z* (rel intensity) 310 (M^+, 5), 308 (M^+, 5), 190 (79), 188 (82), 121 (100); HRMS (EIMS) calcd for $C_{14}H_{13}O_3Br$ 308.0048 found 308.0047.

Cyclohex-3-enone 8. A 1.0 M solution of L-selectride in THF (780 μL, 0.8 mmol) was added dropwise at -78°C to a solution of (*RS*)-**6** (95 mg, 0.3 mmol) in anhydrous THF (3.5 mL). The resulting mixture was stirred –78°C for 1 h, after which time the excess reducing agent was quenched with a aqueous 10% NaOH (8 mL). The mixture was extracted with EtOAc (3 × 5 mL) and the combined organic layers were washed with brine, dried over Na_2SO_4, filtered and evaporated to give a residue, which was submitted to column chromatography, eluting with light petroluem/Et_2O (1:1), to afford **8** (32 mg, 33%). $[\alpha]^{21}_D$ – 8.2° (c = 1.08, $CHCl_3$); IR (NaCl) 2896, 1746, 1646, 1456 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 2.80-3.00 (m, 4H), 3.90 (bt, J = 9.2 Hz, 1H), 4.42 (dd, J = 8.4, 5.6 Hz, 1H), 5.11 (dd, J = 9.6, 5.6 Hz, 1H), 6.19 (s, 1H), 7.34-7.40 (m, 5H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 202.1, 136.2, 130.3, 130.1, 128.7, 128.6, 126.9, 101.3, 79.8, 73.1, 37.1, 35.7; EIMS *m/z* (rel intensity) 310 (M^+, 1), 308 (M^+, 1), 282 (2), 280 (2), 229 (3), 201 (100).

1-phenylcyclohexa-2,4-diene-1-ol 9. A 1.0 M solution of PhMgBr in THF (550 μL, 0.6 mmol) was added at -78°C to a solution of (*RS*)-**6** (68 mg, 0.2 mmol) in anhydrous THF (5 mL). The resulting mixture was stirred at -78°C for 1 h, after which time the excess Grignard reagent was quenched with saturated aqueous $NaHCO_3$. The mixture was diluted in H_2O (5mL), separated and the aqueous layer was extracted with Et_2O (3 × 10 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered and evaporated to give a residue, which was submitted to column chromatography, eluting with light petroluem/Et_2O (1:1), to afford **9** (74 mg, 88%) in 90% *de*. Major diastereoisomer only: $[\alpha]^{21}_D$ + 83.1° (c = 1.29, $CHCl_3$); IR (NaCl) 3450, 2930, 2848, 1742, 1496, 1450 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 3.40 (s, 1H), 3.59 (bt, J = 8.4 Hz, 1H), 4.23 (dd, J = 8.1, 6.2 Hz, 1H), 5.14 (dd, J = 8.7, 6.2 Hz, 1H), 5.93 (d, J = 10.0 Hz, 1H), 6.15 (dd, J = 10.0, 1.8 Hz, 1H), 6.23 (d, J = 1.6 Hz, 1H), 7.39-7.66 (m, 10H); ^{13}C NMR ($CDCl_3$, 50.3 MHz) δ 138.3, 137.2, 135.9, 129.2, 129.0, 128.9, 128.1, 127.9, 127.4, 127.1, 126.7, 121.0, 109.8, 78.5, 71.3; EIMS *m/z* (rel intensity) 386 (M$^+$, 0.2), 384 (M$^+$, 0.2), 368 (0.5), 366 (0.5), 305 (2), 282 (4), 280 (4); HRMS (EIMS) calcd for $C_{20}H_{17}O_3Br$ 384.0361 found 384.0355.

2-(4-Allyl-5-bromo-2-hydroxyphenoxy)-(2*R*)-phenylethanol (10a) and 2-(4-allyl-5-bromo-2-hydroxyphenoxy)-(1*R*)-phenylethanol (10b). A 1.0 M solution of allylmagnesium bromide in ether (2.2 mL, 2.2 mmol) was added at -78°C to a solution of (*RS*)-**6** (68 mg, 0.2 mmol) in anhydrous THF (5 mL). The resulting mixture was stirred at -78°C for 1 h, after which time the excess Grignard reagent was quenched with saturated aqueous $NaHCO_3$. The mixture was diluted in H_2O (5mL), separated and the aqueous layer was extracted with Et_2O (3 × 10 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered and evaporated to give a residue, which was submitted to column chromatography, eluting with light petroluem/Et_2O (1:1), to afford **10a** (22 mg, 29%) and **10b** (25 mg, 32%) as yellow oils. **10a**: IR (NaCl) 3340, 2928, 1490 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.27 (d, J = 6.4 Hz, 2H), 3.80-3.93 (m, 2H), 4.93-5.02 (m, 3H), 5.75-5.88 (m, 1H), 6.76 (s, 2H), 7.19-7.32 (m, 5H); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 147.0, 144.4, 136.8, 135.6, 134.5, 128.9, 128.7, 126.3, 121.4, 117.4, 116.4, 112.6, 84.6, 67.0, 39.6; EIMS *m/z* (rel intensity) 350 (M$^+$, 1), 348 (M$^+$, 1), 332 (2), 330 (2), 230 (49), 228 (54). **10b**: IR (NaCl) 3340, 2928, 1490 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.39 (d, J = 6.03 Hz, 2H), 3.83-4.15 (m, 2H), 5.04-5.13 (m, 3H), 5.85-5.98 (m, 1H), 6.84 (s, 1H), 7.03 (s, 1H), 7.38-7.41 (m, 5H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 146.1, 144.8, 139.2, 135.7, 133.7, 128.8, 128.7, 126.2, 118.5, 117.2, 116.4, 112.9, 75.2, 72.9, 39.6; EIMS *m/z* (rel intensity) 350 (M$^+$, 11), 348 (M$^+$, 12), 332 (27), 330 (29), 230 (65), 228 (70).

Cyclohex-3-enone 11. To a –50°C cooled solution of dimethylmalonate (30 μL, 0.25 mmol) and KHMDS (43 mg, 0.21 mmol) in anhydrous THF (2 mL) was added (*RS*)-**6** (57 mg, 0.2 mmol) in anhydrous THF (3 mL). The resulting mixture was stirred –50°C for 1 hour, after which time it was diluted in Et_2O (10 mL) and H_2O (5 mL). After separation, the aqueous phase was extracted with Et_2O (2 × 10 mL), acidified with aqueous HCl 10% and further extracted with Et_2O (2 × 10 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, filtered and evaporated to give a residue, which was submitted to column chromatography, eluting with light petroluem/Et_2O (1:1), to afford **11** as a diastereoisomeric mixture (28 mg, 34%) in 30% *de*: IR (NaCl) 2958, 2900, 1748, 1502, 1444 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 3.07 (bt, J = 5.95 Hz, 2H), 3.63-3.87 (m, 8H), 4.11 (bt, J = 3.51 Hz, 1H), 4.35 (dd, J = 8.4, 5.6 Hz, 1H), 4.46 (dd, J = 8.2, 5.8 Hz, 1H), 5.10 (dd, J = 9.8, 5.8 Hz, 1H), 6.31 (d, J = 3.35 Hz, 1H), 7.36-7.58 (m, 5H); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 200.8, 200.2, 167.9, 166.9, 136.2, 136.0, 133.0, 132.8, 130.5, 130.3, 128.7, 128.6, 128.5, 127.1, 127.0, 100.2, 99.8, 80.3, 79.9, 73.9, 72.7, 56.1, 53.0, 52.7, 52.6, 52.5, 44.6, 41.1, 39.7, 39.5; EIMS *m/z* (rel intensity) 440 (M^+, 0.02), 438 (M^+, 0.02), 359 (12), 331 (29); HRMS (EIMS) calcd for $C_{19}H_{19}O_7Br$ 438.0314 found 438.022.

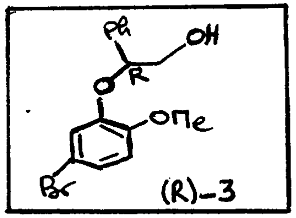
Additional References

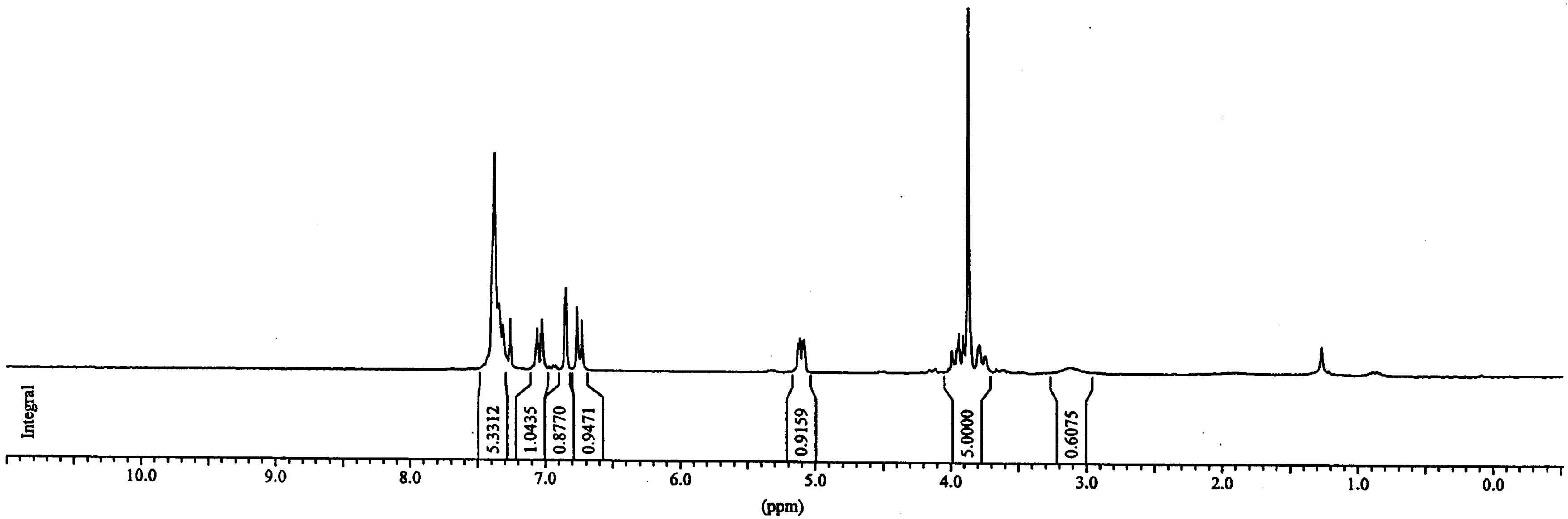
[1] A. I. Meyers, L. Snyder, *J. Org. Chem.* **1993**, *58*, 36.

[2] M. Van der Mey, A. Hatzelmann, G. P. M. Van Klink, I. J. Van der Laan, G. J. Sterk, U. Thibaut, W. R. Ulrich, H. Timmerman, *J. Med. Chem.* **2001**, *44*, 2523.

[3] H. Fujioka, H. Annoura, K. Murano, Y. Kita, Y. Tamura, *Chem. Pharm. Bull.* **1989**, *37*, 2047.

IFB153.3/CDCl3/JT10-104/AC 250





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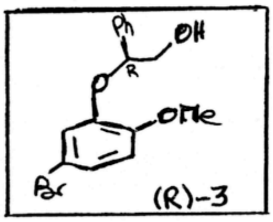

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7.09/ 1774.5	7.00/ 1750.4	6.40	19.14
6.88/ 1720.1	6.81/ 1704.3	17.56	27.02
6.78/ 1695.1	6.69/ 1672.2	11.26	20.06
5.18/ 1295.6	4.97/ 1244.4	4.55	12.79
4.05/ 1012.7	3.49/ 873.0	81.08	117.70
4.08/ 1021.6	3.62/ 905.6	4.01	20.32
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Peak Picking results

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4	6915	1835.80	7.3393	2024	18.0
5	6934	1830.00	7.3161	1385	12.3
6	6980	1815.96	7.2600	1562	13.9
7	7140	1767.14	7.0648	969	8.6
8	7147	1765.00	7.0563	1278	11.4
9	7168	1758.59	7.0306	1123	10.0
10	7176	1756.15	7.0209	1549	13.8
11	7313	1714.34	6.8537	2195	19.6
12	7320	1712.20	6.8452	2517	22.4
13	7386	1692.06	6.7647	1924	17.1
14	7414	1683.52	6.7305	1538	13.7
15	8729	1282.21	5.1261	876	7.8
16	8740	1278.86	5.1127	1051	9.4
17	8757	1273.67	5.0920	964	8.6
18	8768	1270.31	5.0785	989	8.8
19	9662	997.48	3.9878	701	6.2
20	9691	988.63	3.9524	771	6.9
21	9702	985.28	3.9390	1216	10.8
22	9730	976.73	3.9048	1152	10.3
23	9759	967.88	3.8695	11229	100.0
24	9776	962.69	3.8487	1407	12.5
25	9828	946.82	3.7853	893	7.9
26	9860	937.06	3.7462	529	4.7
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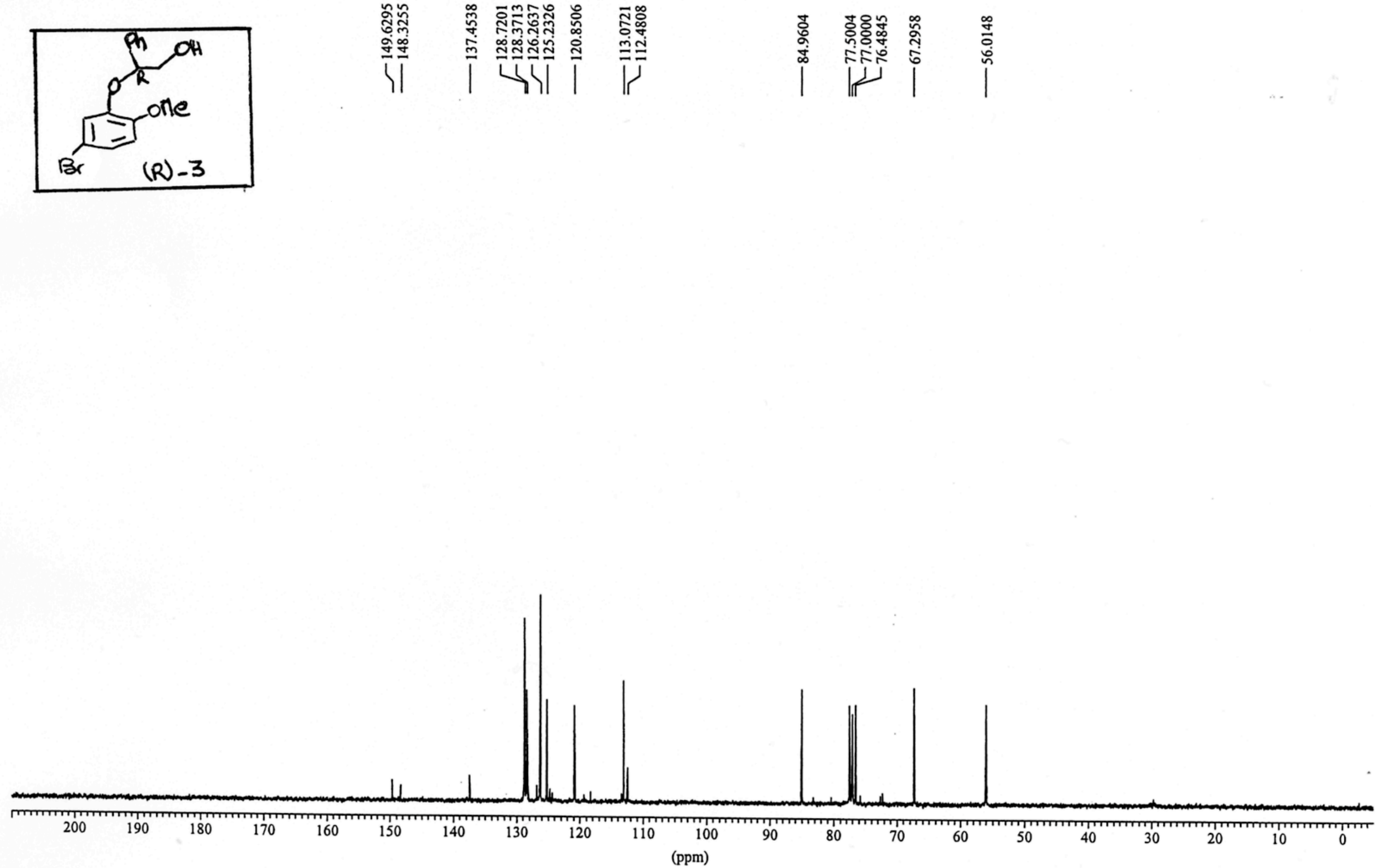
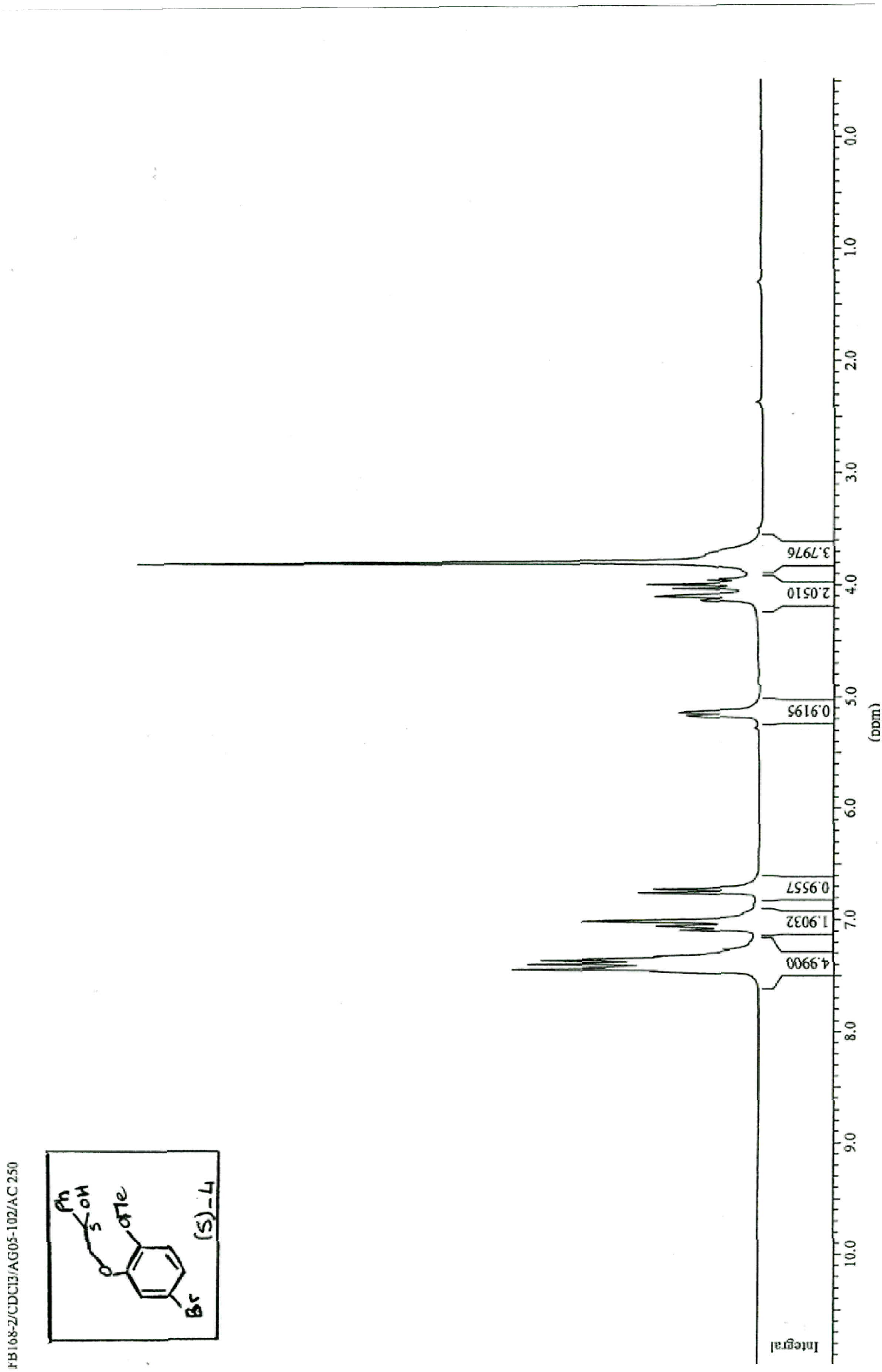
IFB153.3/CDCl3/JT10-104/AC 250

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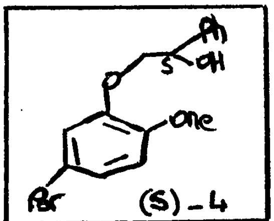

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3.93/ 982.2	3.57/ 893.7	76.37	117.67

Peak Picking results

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5	6881	1846.48	7.3820	1070	33.9
6	6906	1838.85	7.3515	956	30.3
7	6932	1830.92	7.3198	433	13.7
8	6981	1815.96	7.2600	146	4.6
9	7124	1772.32	7.0855	329	10.4
10	7131	1770.19	7.0770	365	11.5
11	7152	1763.78	7.0514	416	13.2
12	7159	1761.64	7.0428	473	15.0
13	7189	1752.49	7.0062	856	27.1
14	7195	1750.66	6.9989	671	21.3
15	7400	1688.10	6.7488	620	19.6
16	7428	1679.55	6.7146	532	16.8
17	8696	1292.59	5.1676	303	9.6
18	8705	1289.84	5.1566	330	10.5
19	8725	1283.74	5.1322	375	11.9
20	8734	1280.99	5.1212	361	11.4
21	9543	1034.10	4.1342	219	6.9
22	9552	1031.36	4.1232	242	7.7
23	9575	1024.34	4.0952	463	14.7
24	9584	1021.59	4.0842	440	13.9
25	9636	1005.72	4.0208	434	13.7
26	9666	996.57	3.9842	567	17.9
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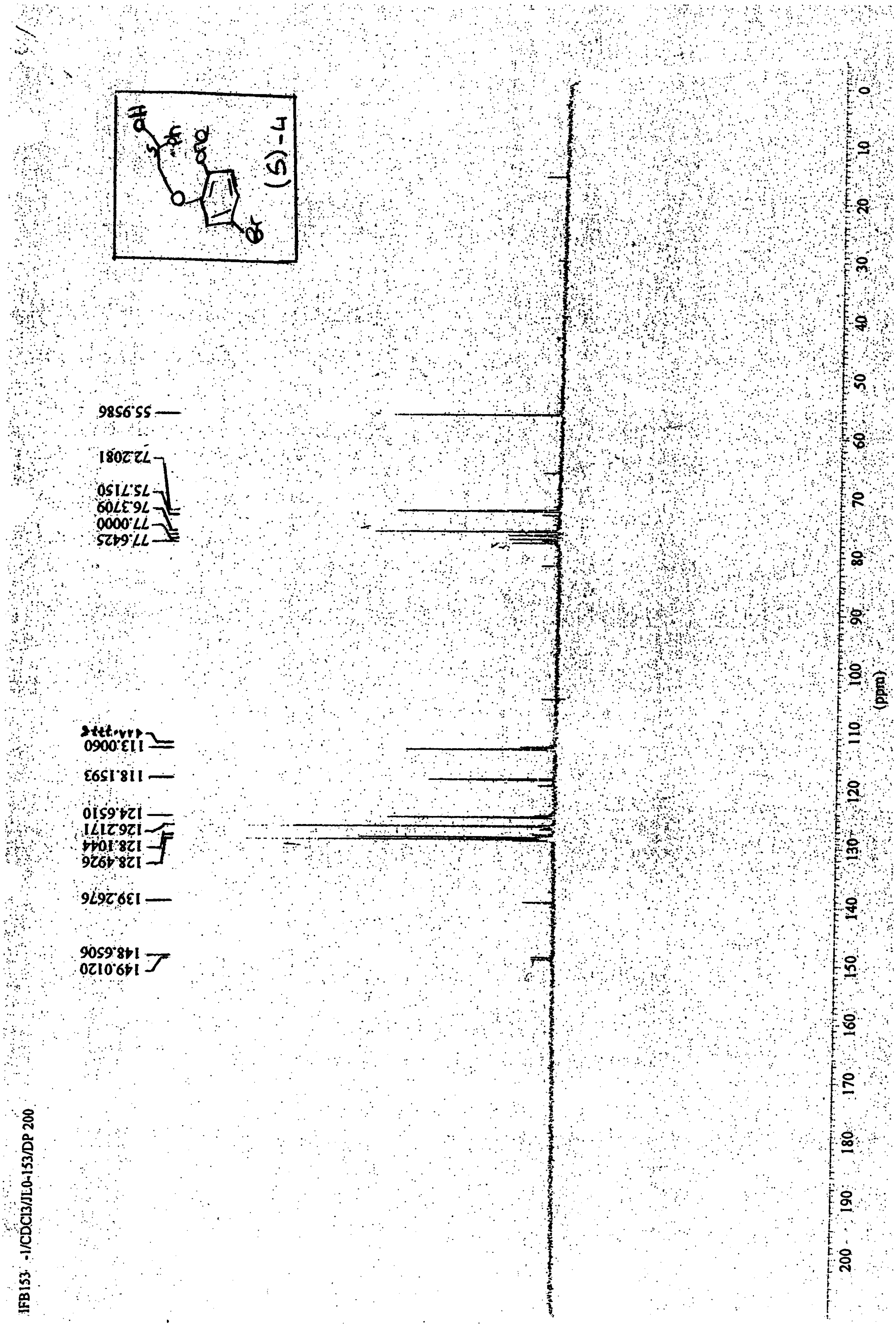
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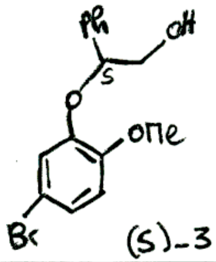

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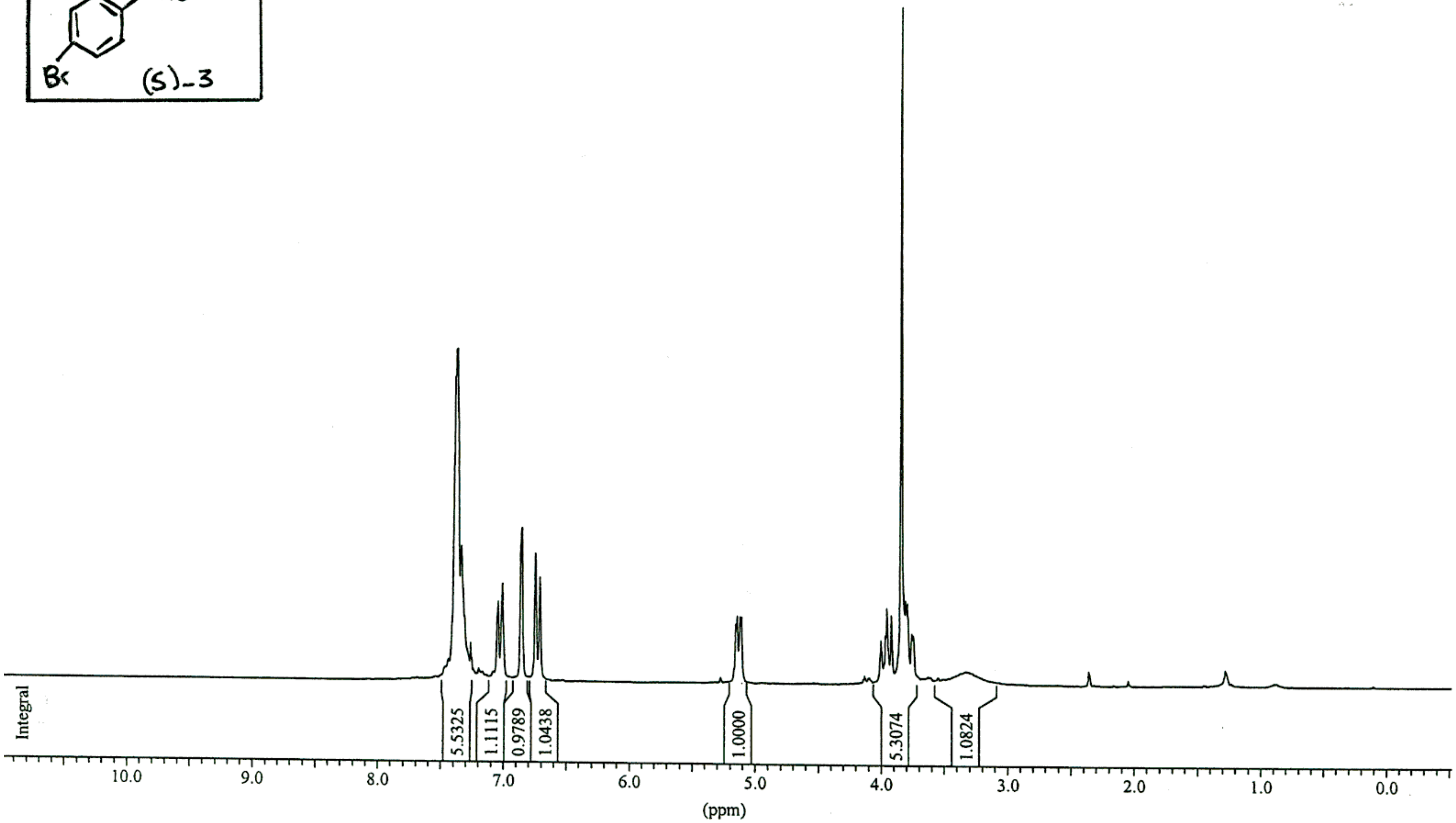

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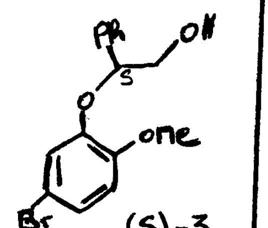

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4.08/ 1019.8	3.46/ 866.3	91.91	108.37
3.39/ 847.0	3.28/ 821.4	-0.00	6.39
6.88/ 1721.7	6.78/ 1696.0	20.89	26.29
7.11/ 1777.2	6.93/ 1732.3	10.32	18.43
6.78/ 1696.0	6.65/ 1664.0	12.78	22.61
7.54/ 1885.8	7.21/ 1802.8	17.45	53.57

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
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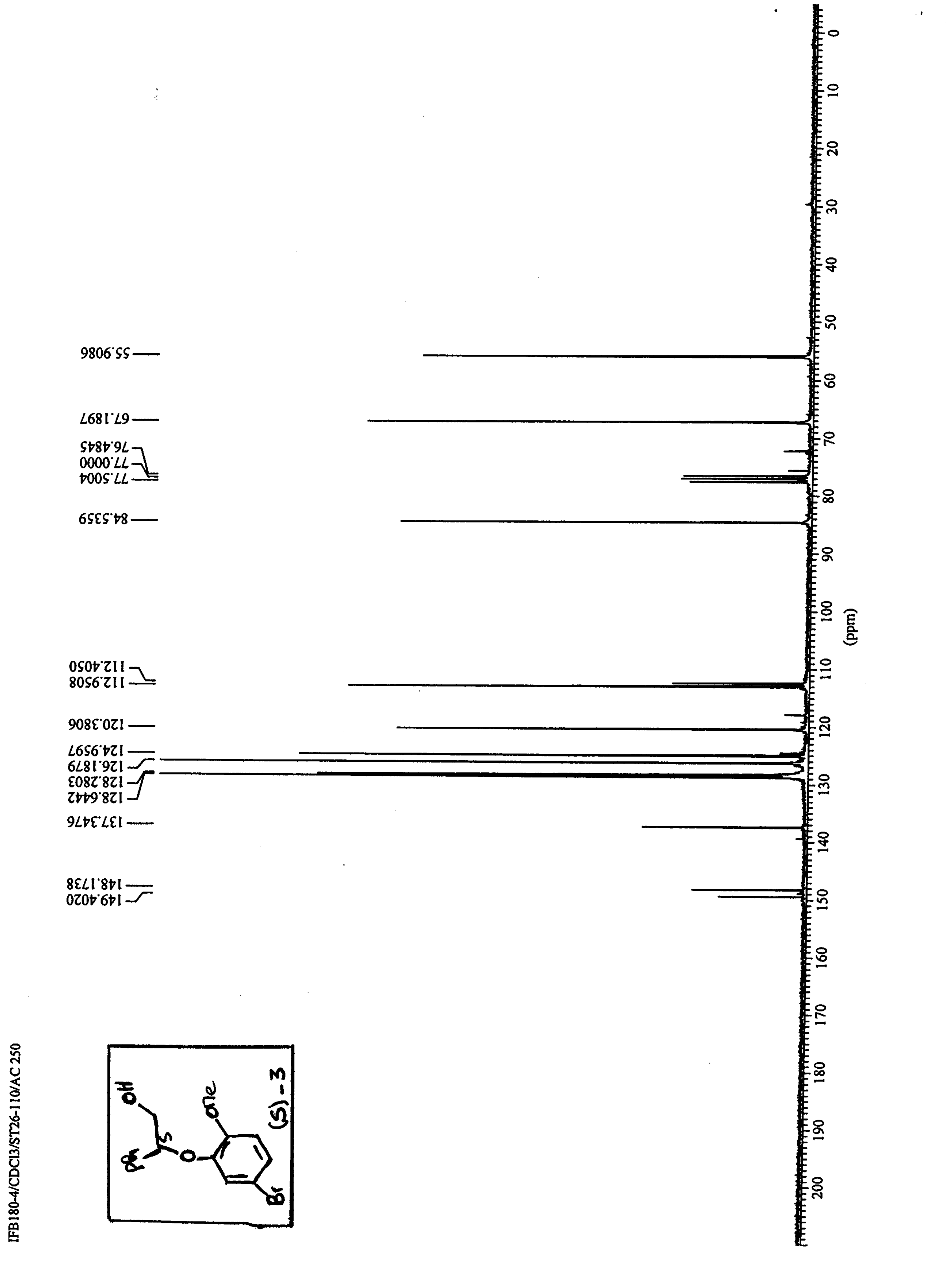
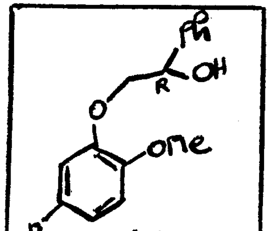
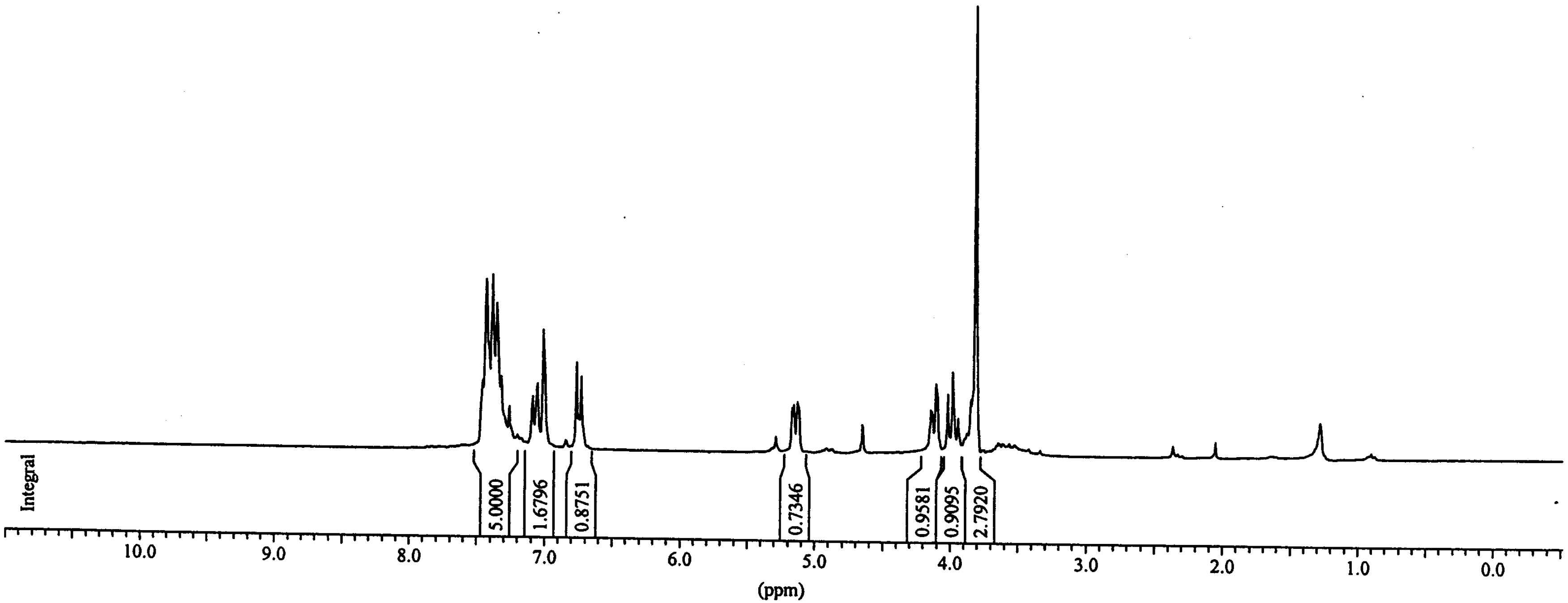
4	7158	1761.34	7.0416	389	11.6
5	7187	1752.49	7.0062	480	14.3
6	7313	1714.04	6.8525	753	22.4
7	7401	1687.18	6.7451	630	18.7
8	7430	1678.33	6.7098	511	15.2
9	8706	1288.93	5.1530	290	8.6
10	8715	1286.18	5.1420	331	9.8
11	8733	1280.69	5.1200	327	9.7
12	8743	1277.63	5.1078	326	9.7
13	9651	1000.53	4.0000	218	6.5
14	9679	991.99	3.9659	242	7.2
15	9690	988.63	3.9524	376	11.2
16	9718	980.09	3.9183	340	10.1
17	9781	960.86	3.8414	3363	100.0
18	9799	955.37	3.8194	392	11.7
19	9810	952.01	3.8060	415	12.3
20	9821	948.65	3.7926	402	11.9
21	9850	939.80	3.7572	252	7.5
22	9860	936.75	3.7450	242	7.2
23	10203	832.08	3.3265	69	2.0


IFB180-4/CDCl3/ST26-110/AC 250
55.9086
67.1897
76.4845
77.0000
77.5004
84.5359
112.4050
112.9508
120.3806
124.9597
126.1879
128.2803
128.6442
137.3476
148.1738
149.4020
(ppm)
(S)-3

IFB180-2/CDCl3/SE25-110/AC 250

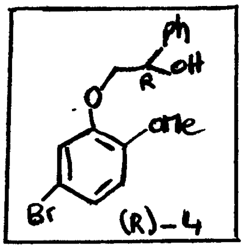


(R)-4



Peak List Date: 22.02.2004 Time: 15:08

File Name : C:\WIN1D\SPC\ASP3000\SE25\110001.1R
Peak Results saved in File :
Nucleus : 1H
SF : 250.132854 MHz
OFFSET : 15.7760 ppm
SW_p : 5000.00 Hz
SI : 16384



Peak Picking Parameter

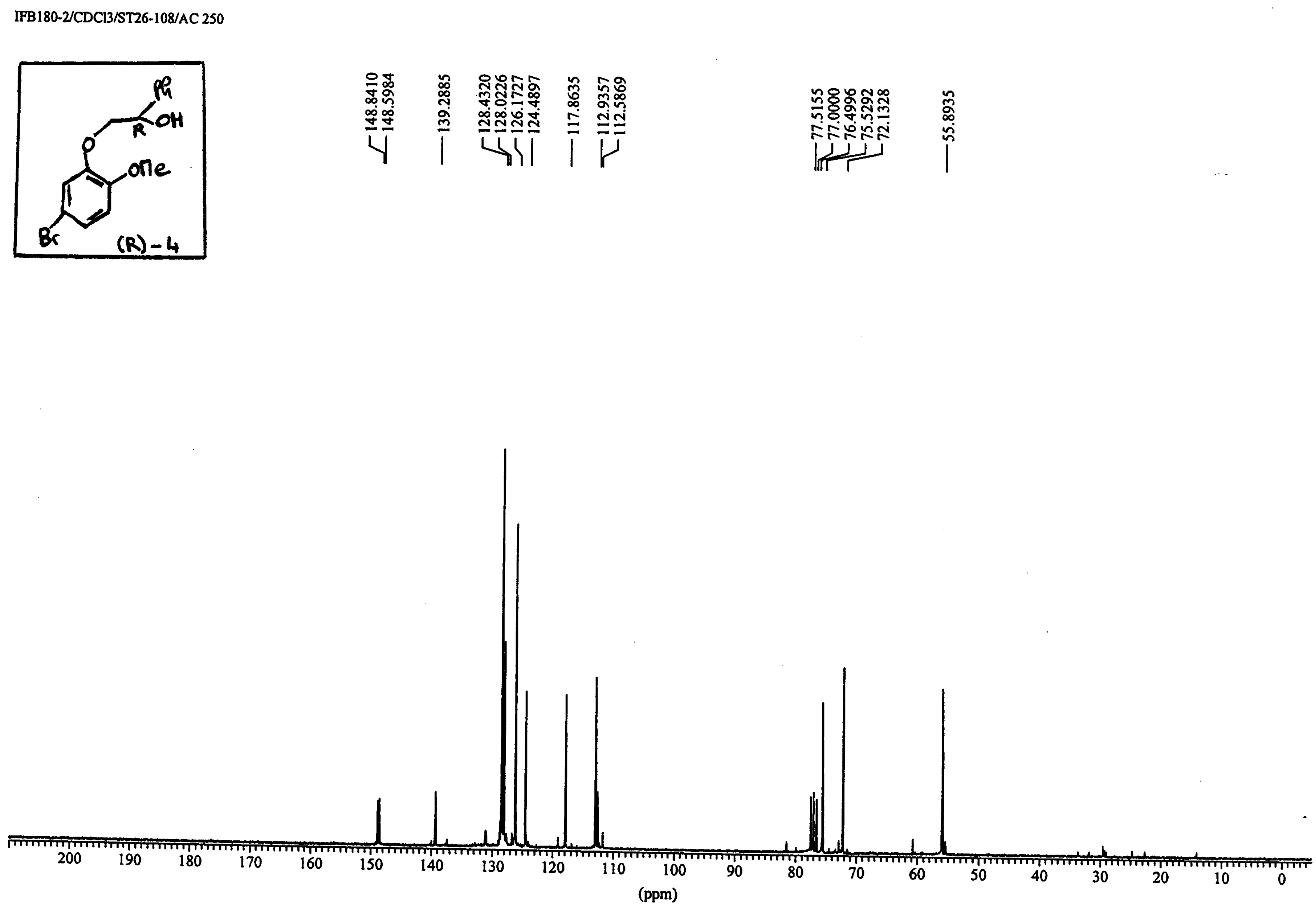
Peak constant PC = 1.00
Noise = 1
Sens. level = 5

Peak Picking region

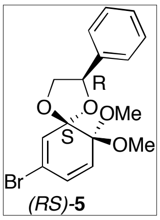
Start(ppm/Hz)	End(ppm/Hz)	MI(%)	MAXI(%)
5.25/ 1314.3	5.02/ 1255.4	8.80	11.86
4.17/ 1042.3	4.07/ 1017.9	7.83	19.39
4.03/ 1008.5	3.90/ 974.6	6.63	19.09
3.83/ 957.2	3.69/ 923.9	94.51	106.72
6.78/ 1695.1	6.62/ 1655.7	10.28	23.68
7.02/ 1755.2	6.92/ 1731.1	19.08	32.49
7.14/ 1786.4	7.04/ 1761.9	10.28	22.48
7.47/ 1868.5	7.25/ 1812.6	7.88	49.69

Peak Picking results

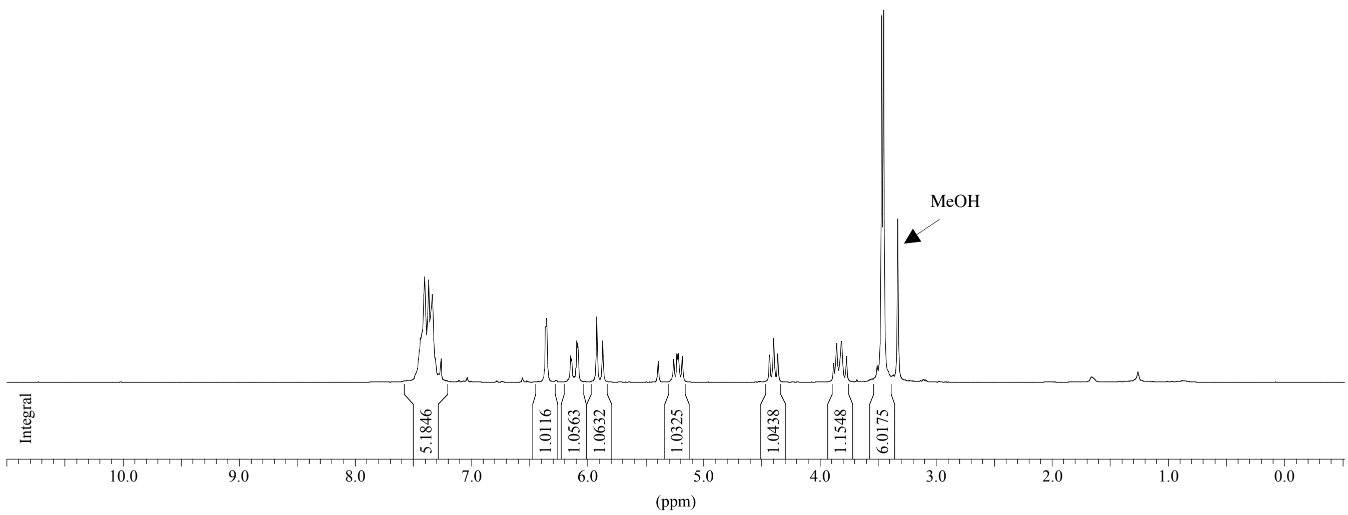
Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6817	1865.71	7.4589	509	15.3
2	6840	1858.69	7.4308	1260	37.9
3	6878	1847.09	7.3844	1293	38.9
4	6903	1839.46	7.3539	1077	32.4
5	6929	1831.53	7.3222	538	16.2
6	6980	1815.96	7.2600	314	9.5
7	7123	1772.32	7.0855	393	11.8
8	7151	1763.78	7.0514	490	14.7
9	7185	1753.40	7.0099	887	26.7
10	7388	1691.45	6.7622	642	19.3
11	7416	1682.91	6.7281	540	16.3
12	8698	1291.67	5.1639	335	10.1
13	8706	1289.23	5.1542	357	10.7
14	8727	1282.82	5.1286	376	11.3
15	8736	1280.08	5.1176	352	10.6
16	9535	1036.24	4.1428	330	9.9
17	9544	1033.49	4.1318	314	9.5
18	9567	1026.47	4.1037	523	15.7
19	9575	1024.03	4.0940	482	14.5
20	9639	1004.50	4.0159	446	13.4
21	9670	995.04	3.9781	607	18.3
22	9701	985.58	3.9402	270	8.1
23	9810	952.32	3.8072	3321	100.0


IFB180-2/CDCl3/ST26-108/AC 250
Ph
OH
R
OMe
Br
(R)-4
148.8410
148.5984
139.2885
128.4320
128.0226
126.1727
124.4897
117.8635
112.9357
112.5869
77.5155
77.0000
76.4996
75.5292
72.1328
55.8935
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

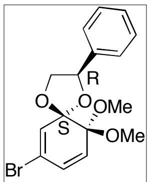
IFC115-1/CDCl3/JU04-ifc115-1/DPX 200



(RS)-**5**

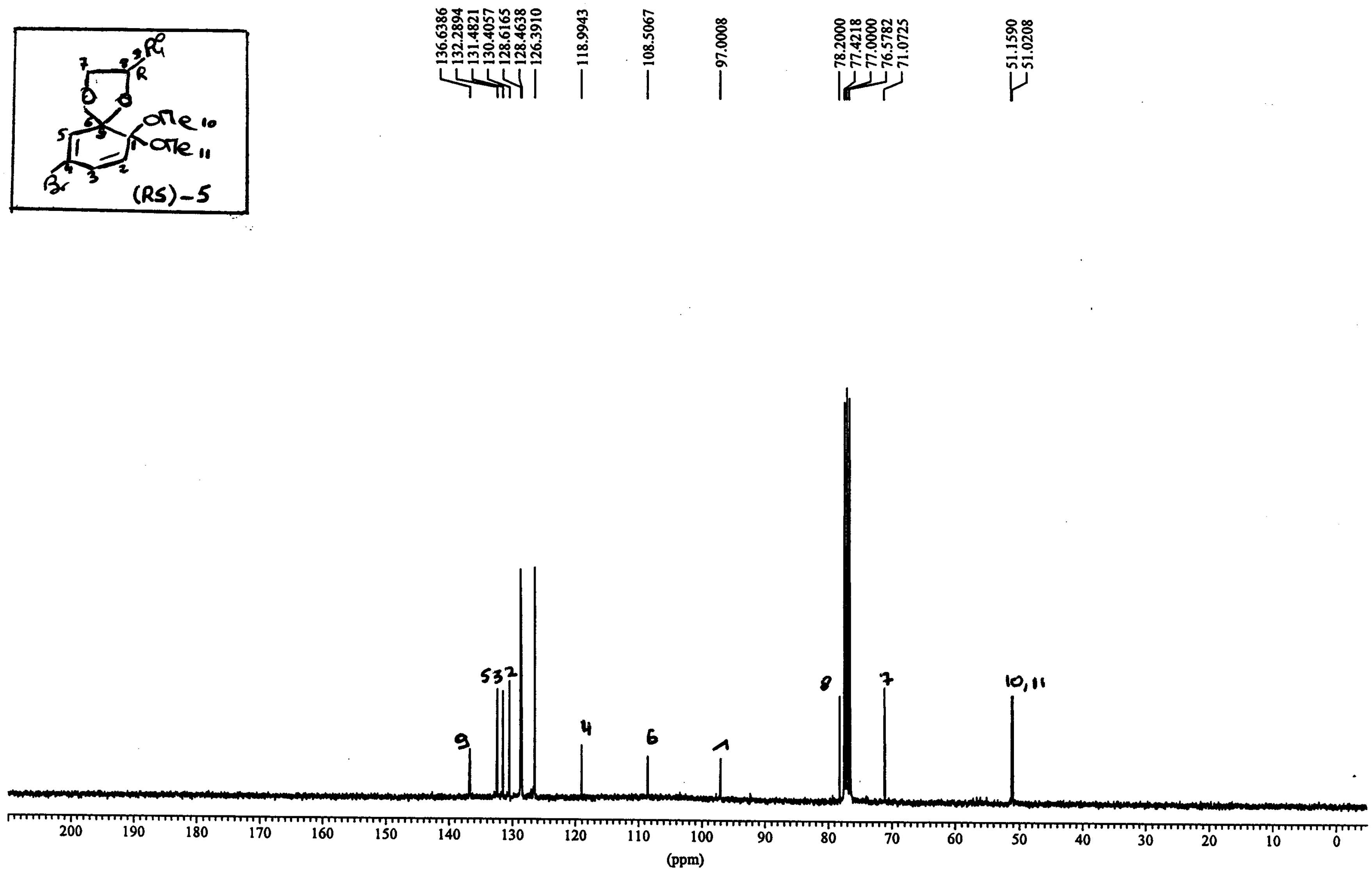


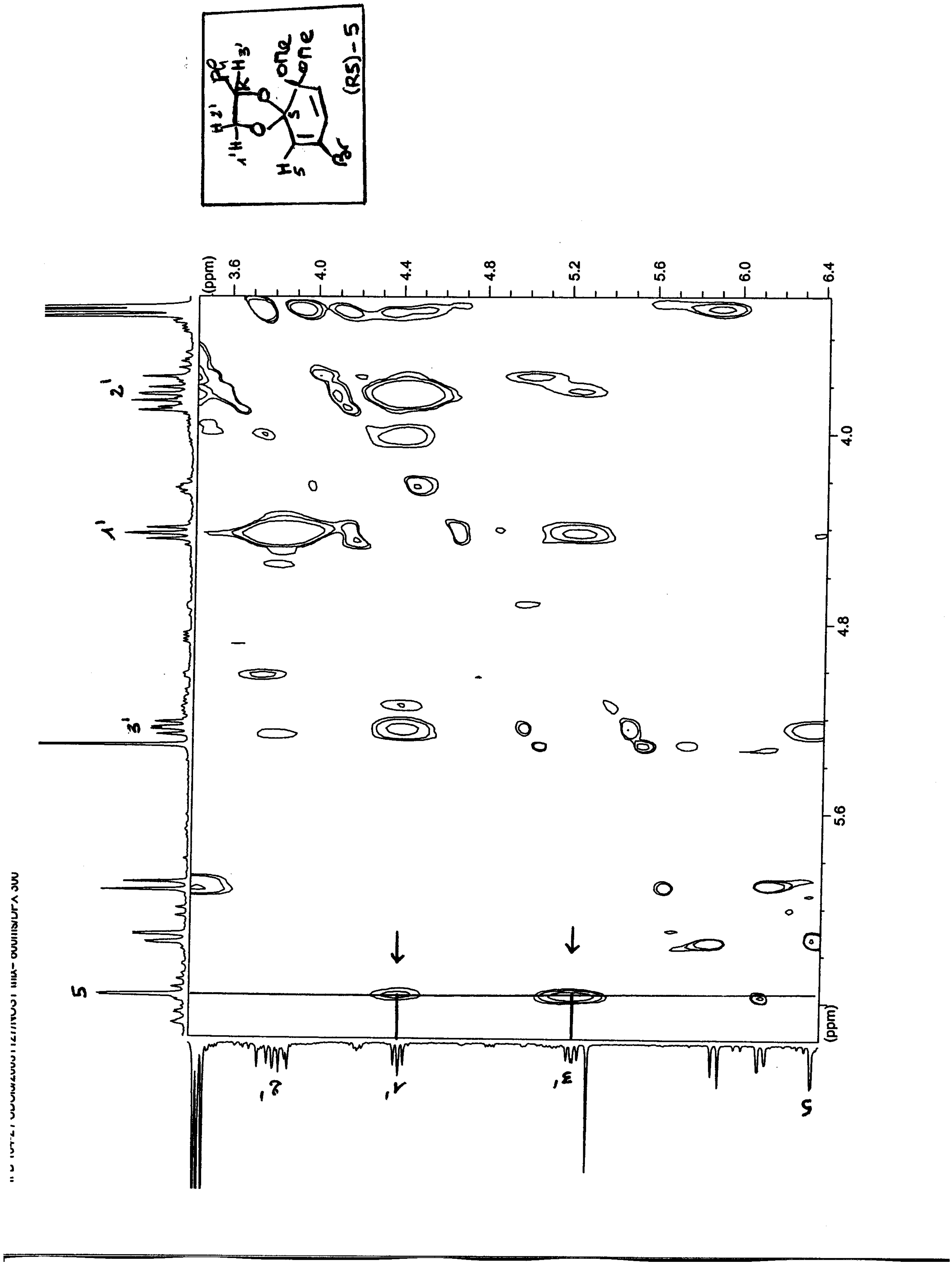
Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	5632	1488.61	7.4371	19608	12.0
2	5677	1481.45	7.4013	46272	28.4
3	5719	1474.78	7.3680	44857	27.5
4	5760	1468.26	7.3354	38537	23.6
5	5855	1453.16	7.2600	10062	6.2
6	6988	1273.08	6.3603	26895	16.5
7	6997	1271.65	6.3531	28084	17.2
8	7260	1229.84	6.1443	11507	7.1
9	7270	1228.25	6.1364	10830	6.6
10	7325	1219.51	6.0927	18176	11.1
11	7335	1217.92	6.0847	17265	10.6
12	7542	1185.02	5.9204	28740	17.6
13	7607	1174.69	5.8687	18264	11.2
14	8372	1053.10	5.2613	9896	6.1
15	8414	1046.42	5.2279	12419	7.6
16	8429	1044.04	5.2160	12639	7.7
17	8471	1037.36	5.1827	11200	6.9
18	9415	887.32	4.4330	11850	7.3
19	9461	880.00	4.3965	19410	11.9
20	9505	873.01	4.3616	12366	7.6
21	10110	776.85	3.8811	8223	5.0
22	10144	771.44	3.8541	16978	10.4
23	10193	763.66	3.8152	18010	11.0
24	10633	693.72	3.4658	160796	98.6
25	10653	690.54	3.4499	163117	100.0



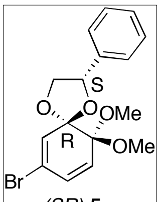
(RS)-**5**

IFB164-2/CDCl3/26070313/DPX 300

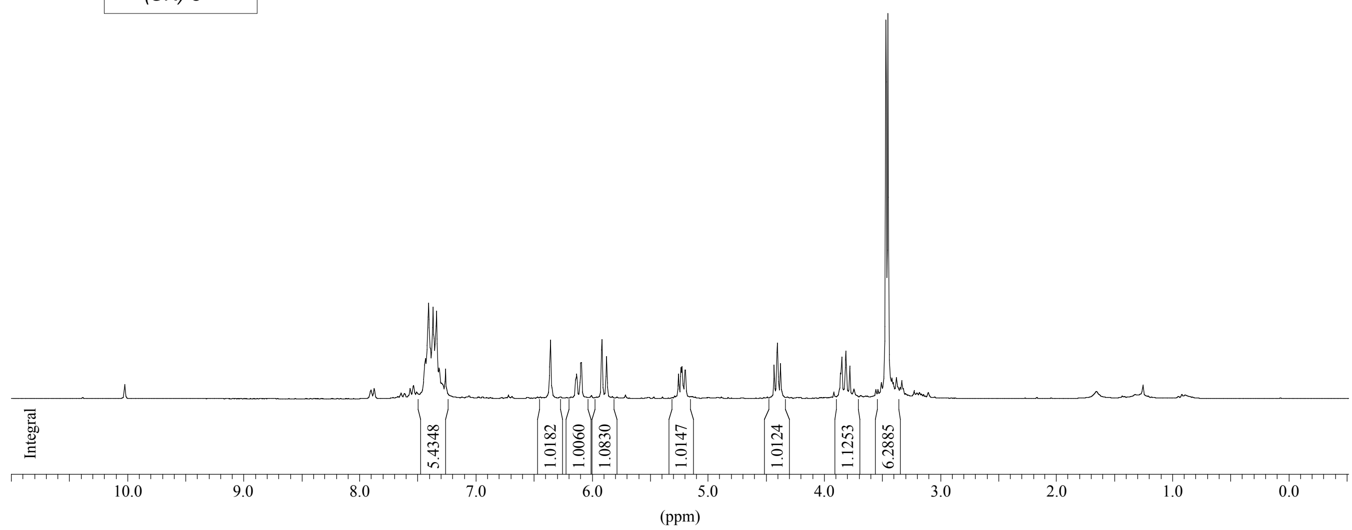



(RS)-5
(ppm)
3.6
4.0
4.4
4.8
5.2
5.6
6.0
6.4

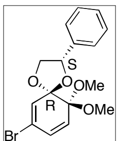
IFC30-1/CDCl3/DB16-110/AC 250



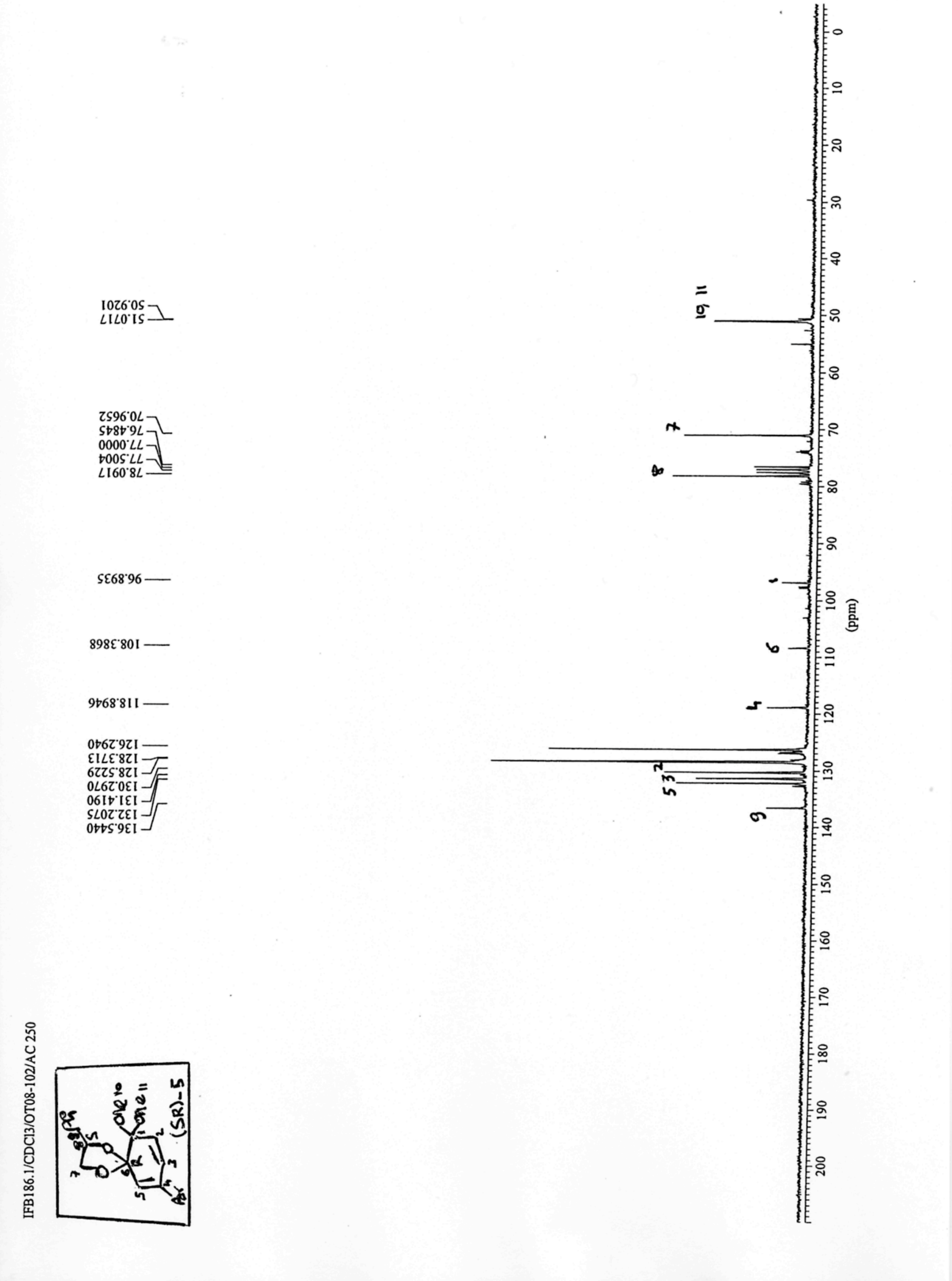
(SR)-**5**

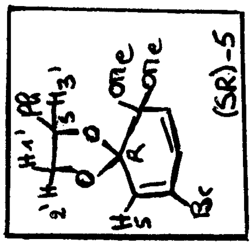


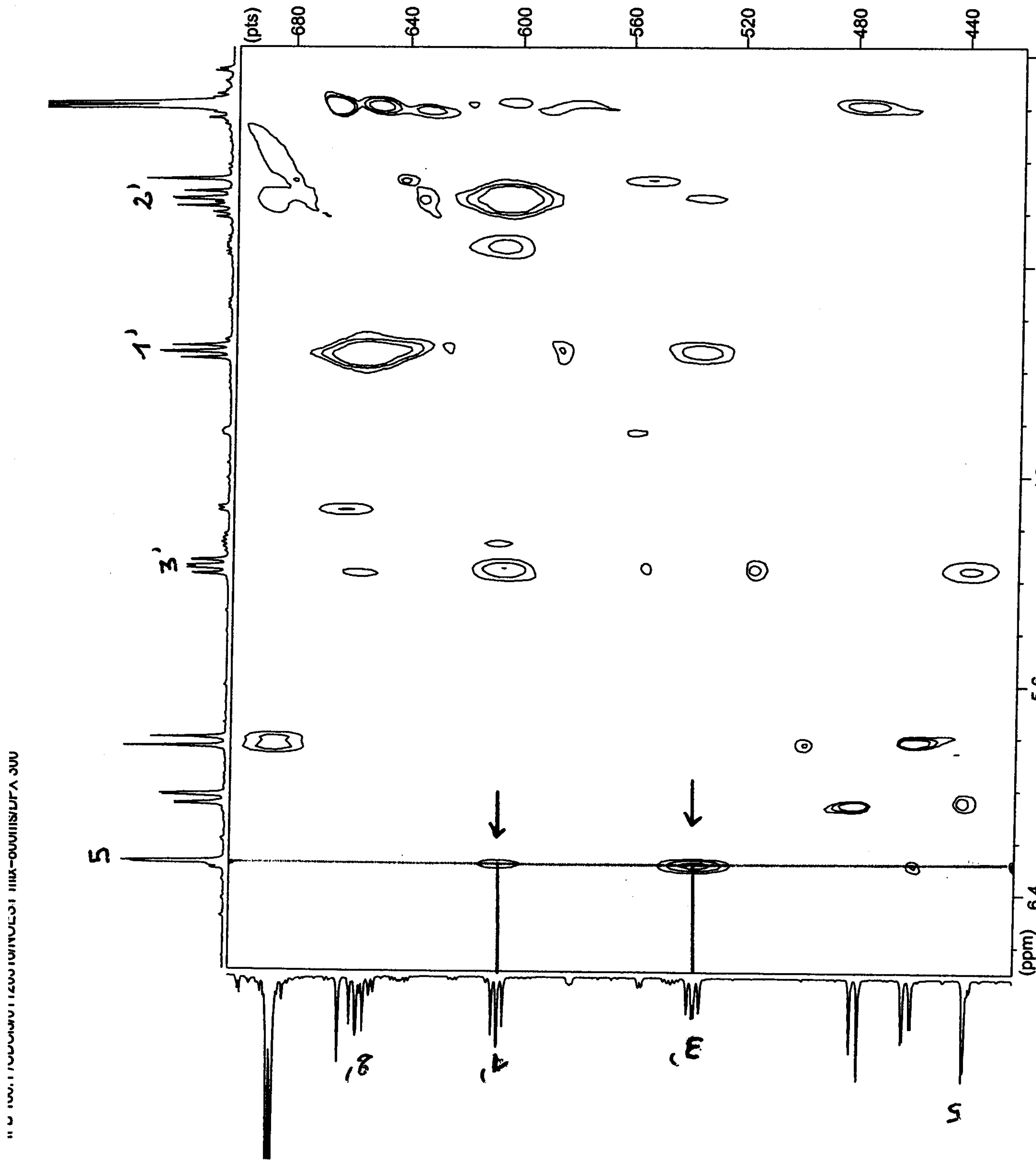
Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6837	1859.30	7.4332	498	10.3
2	6861	1851.98	7.4040	1200	24.8
3	6891	1842.82	7.3674	1153	23.8
4	6916	1835.19	7.3369	1097	22.7
5	6935	1829.39	7.3137	380	7.8
6	6979	1815.96	7.2600	368	7.6
7	7716	1591.05	6.3608	682	14.1
8	7721	1589.52	6.3547	735	15.2
9	7898	1535.51	6.1388	301	6.2
10	7903	1533.98	6.1327	304	6.3
11	7932	1525.13	6.0973	439	9.1
12	7937	1523.61	6.0912	446	9.2
13	8082	1479.36	5.9143	742	15.3
14	8115	1469.28	5.8740	527	10.9
15	8621	1314.87	5.2567	304	6.3
16	8643	1308.15	5.2298	385	7.9
17	8651	1305.71	5.2201	395	8.2
18	8673	1299.00	5.1932	354	7.3
19	9299	1107.96	4.4295	416	8.6
20	9323	1100.63	4.4002	697	14.4
21	9345	1093.92	4.3734	435	9.0
22	9777	962.08	3.8463	514	10.6
23	9806	953.23	3.8109	593	12.2
24	9832	945.30	3.7792	403	8.3
25	10087	867.48	3.4681	4763	98.3
26	10101	863.21	3.4510	4843	100.0



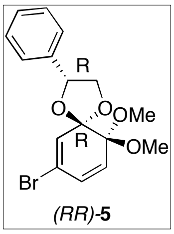
(SR)-**5**

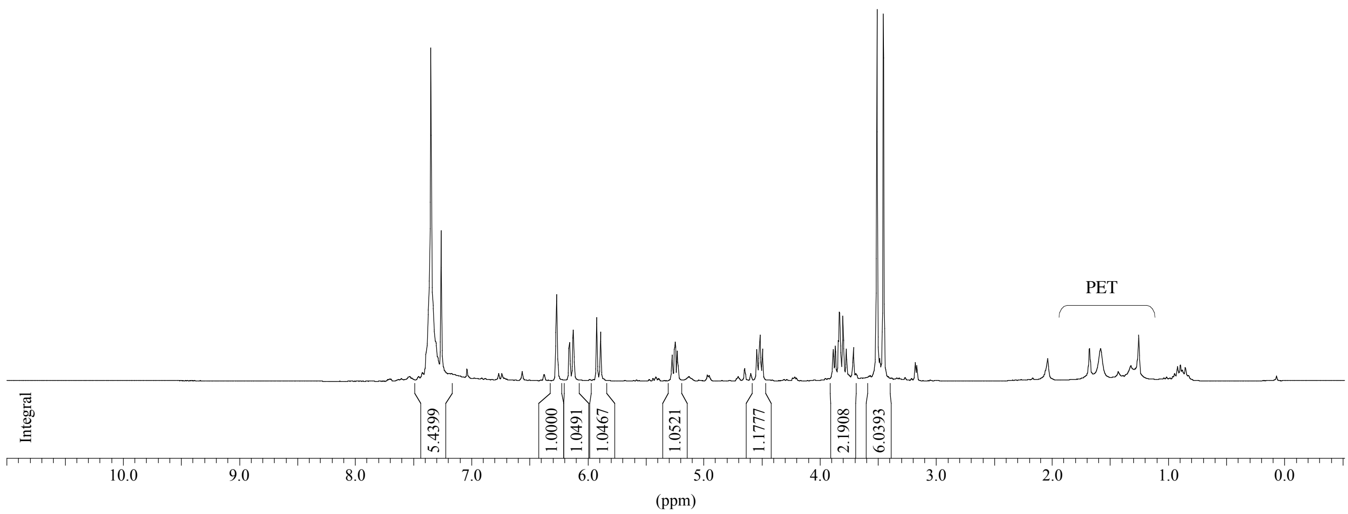

IFB186.1/CDCl3/OT08-102/AC 250
(SR)-5
51.0717
50.9201
78.0917
77.5004
77.0000
76.4845
70.9652
96.8935
108.3868
118.8946
126.2940
128.3713
128.5229
130.2970
131.4190
132.2075
136.5440
(ppm)



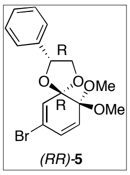


IFC34-2-B/CDCl3/12010424/DPX 300

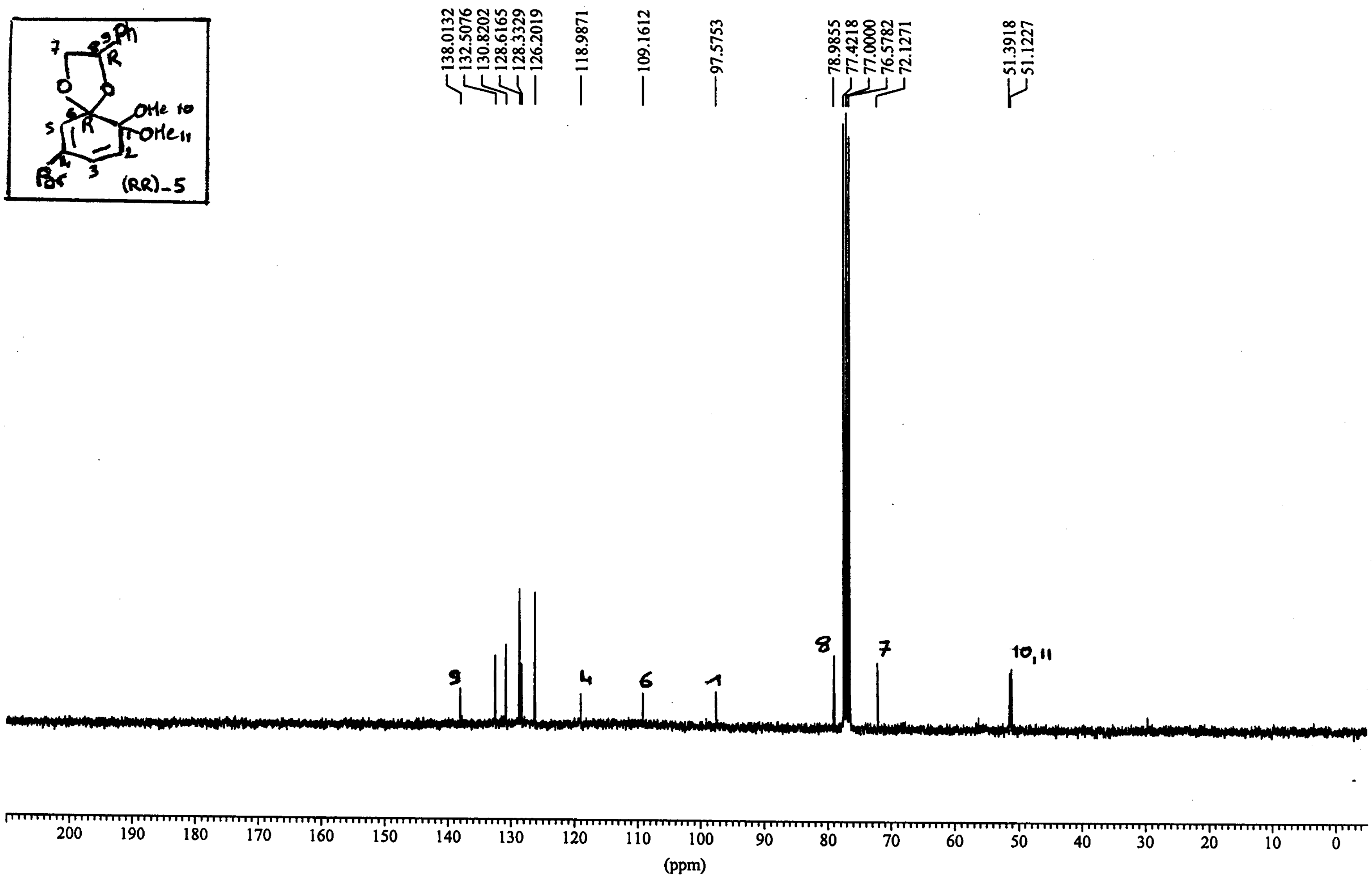




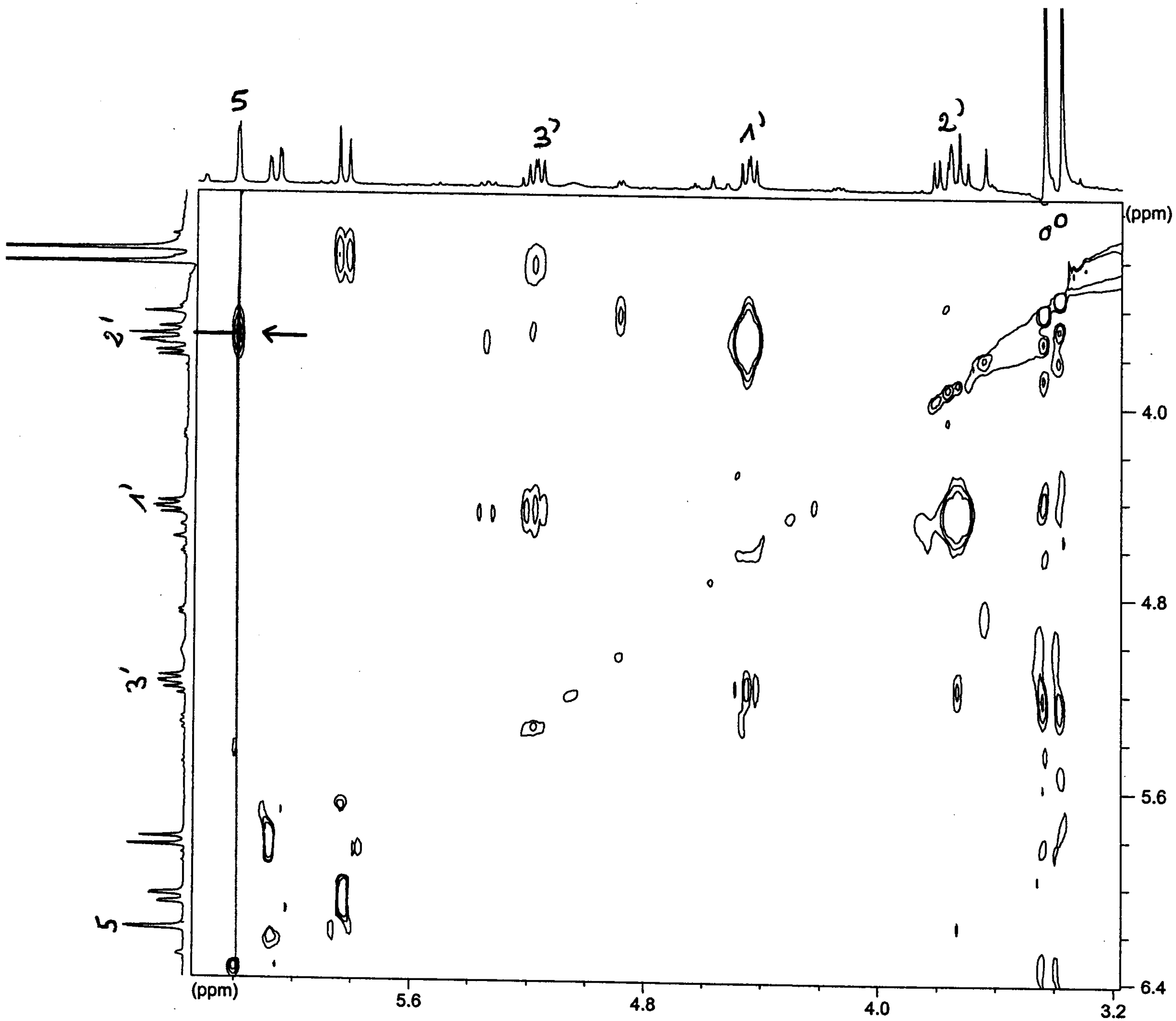
Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	9889	2205.69	7.3491	260032	89.7
2	9960	2178.94	7.2600	117446	40.5
3	10752	1880.55	6.2658	67472	23.3
4	10839	1847.77	6.1566	29818	10.3
5	10866	1837.60	6.1227	39751	13.7
6	11025	1777.69	5.9231	49682	17.1
7	11052	1767.52	5.8892	38401	13.2
8	11541	1583.29	5.2753	20181	7.0
9	11563	1575.00	5.2477	30213	10.4
10	11581	1568.22	5.2251	23057	8.0
11	12125	1363.26	4.5422	24373	8.4
12	12142	1356.85	4.5209	29225	10.1
13	12147	1354.97	4.5146	35659	12.3
14	12164	1348.56	4.4933	24822	8.6
15	12647	1166.59	3.8869	24435	8.4
16	12662	1160.94	3.8681	27123	9.4
17	12691	1150.01	3.8317	53867	18.6
18	12715	1140.97	3.8016	50788	17.5
19	12737	1132.68	3.7740	24605	8.5
20	12787	1113.84	3.7112	25671	8.9
21	12948	1053.18	3.5091	289976	100.0
22	12992	1036.61	3.4539	286469	98.8

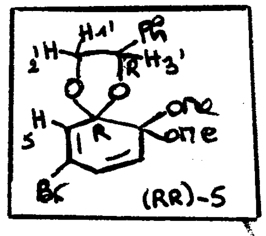


IFC 34-2-B/CDCl3/12010424/DPX 300

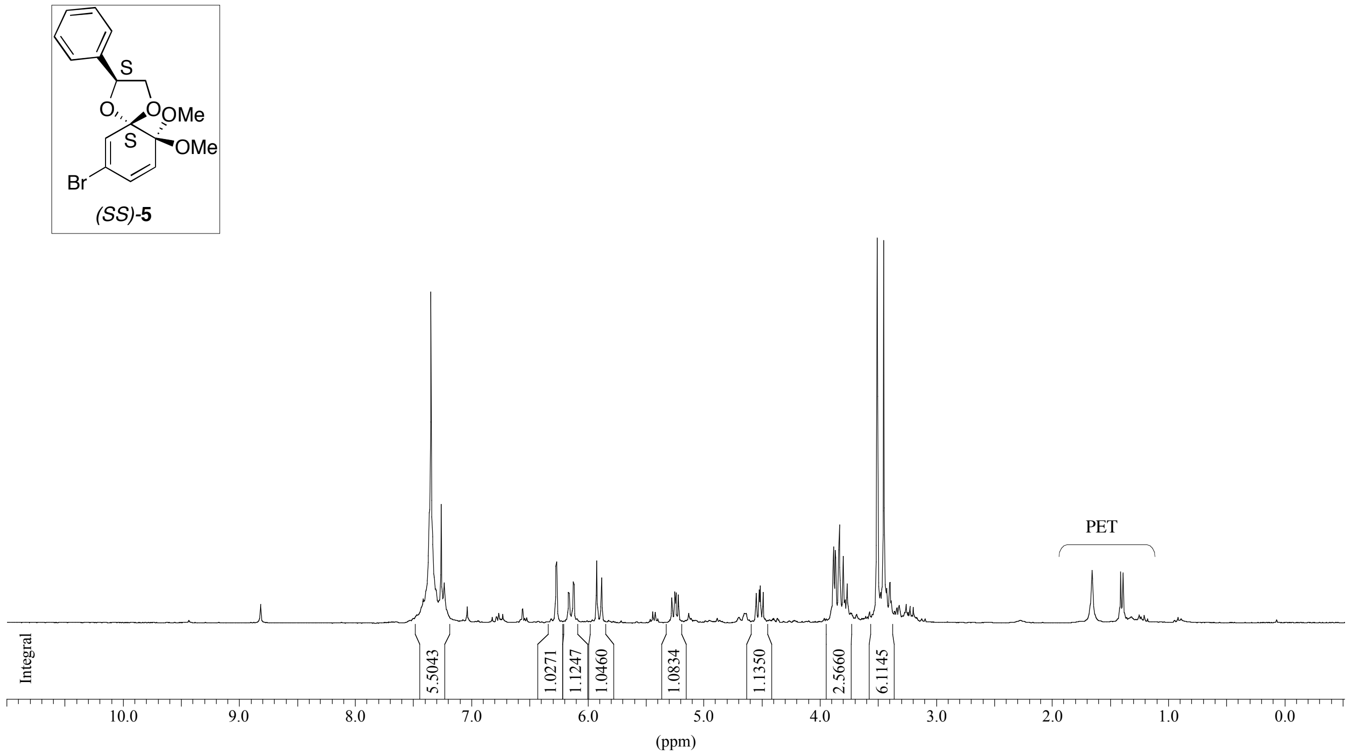


IFC34 1/2 B /CDCl3/30010413/ DPX 300/NOESY mix=800ms

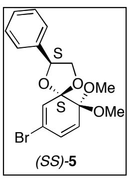


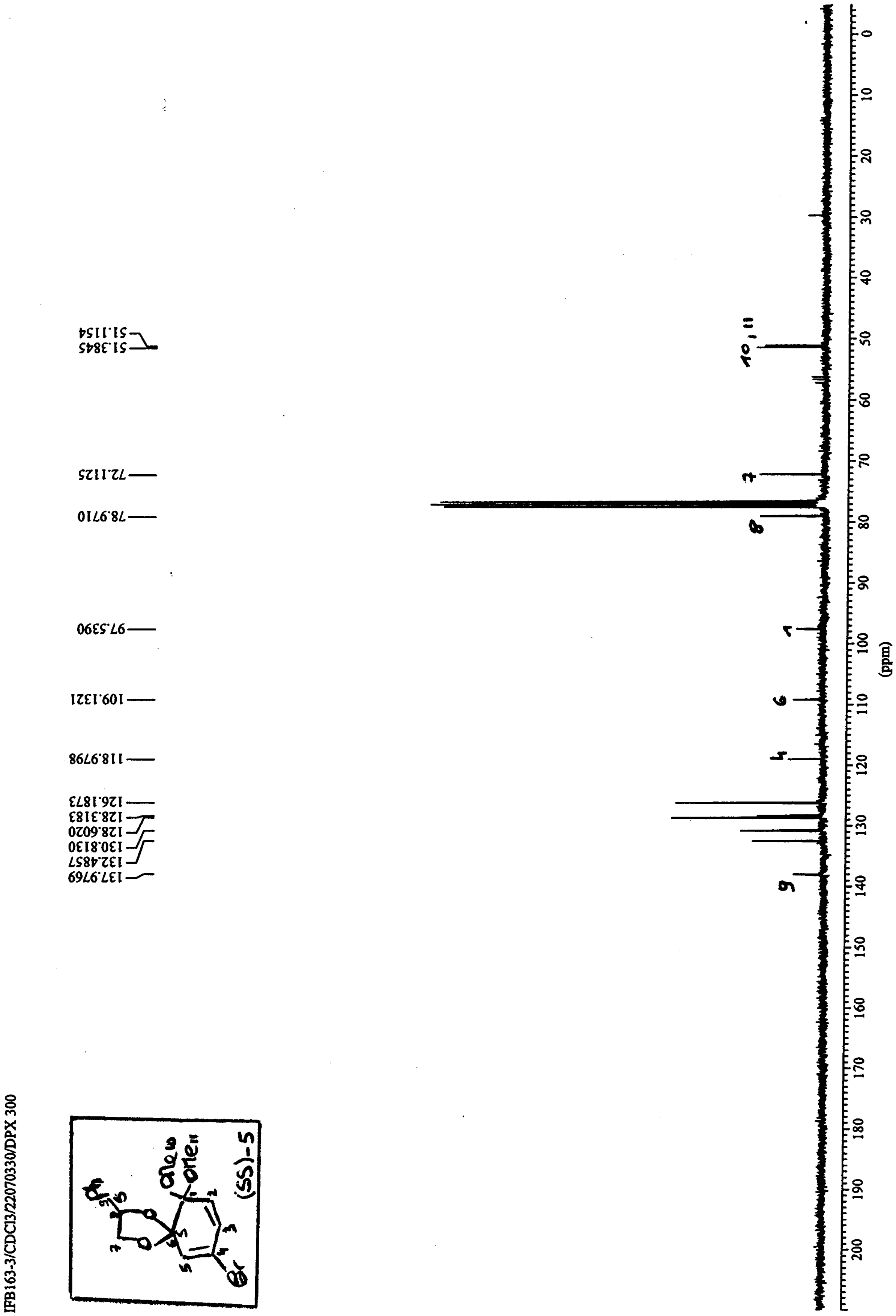


IFC126-2/CDCl3/OC13-105/AC 250



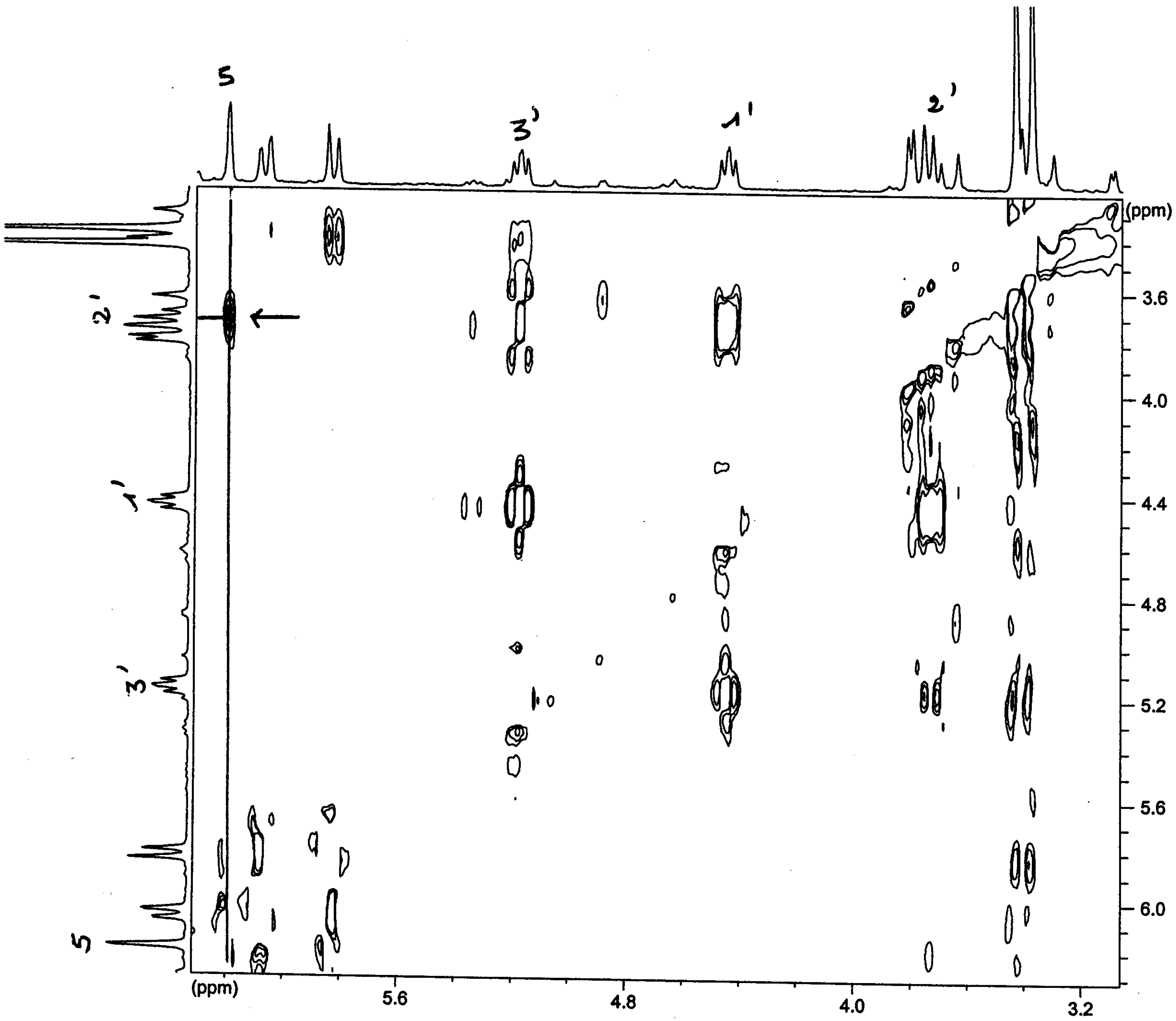
Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6907	1837.94	7.3478	3103	85.8
2	6917	1834.89	7.3356	959	26.5
3	6979	1815.96	7.2600	1109	30.7
4	7788	1569.08	6.2730	532	14.7
5	7794	1567.25	6.2657	572	15.8
6	7876	1542.22	6.1656	285	7.9
7	7881	1540.70	6.1595	274	7.6
8	7909	1532.15	6.1253	385	10.7
9	7915	1530.32	6.1180	355	9.8
10	8075	1481.49	5.9228	587	16.2
11	8109	1471.12	5.8813	420	11.6
12	8605	1319.75	5.2762	234	6.5
13	8626	1313.34	5.2506	291	8.1
14	8634	1310.90	5.2408	279	7.7
15	8654	1304.80	5.2164	261	7.2
16	9202	1137.56	4.5478	279	7.7
17	9223	1131.15	4.5222	311	8.6
18	9230	1129.01	4.5137	347	9.6
19	9251	1122.61	4.4880	284	7.8
20	9748	970.93	3.8817	718	19.9
21	9760	967.27	3.8670	685	18.9
22	9786	959.34	3.8353	880	24.3
23	9789	958.42	3.8316	920	25.5
24	9817	949.88	3.7975	629	17.4
25	9845	941.33	3.7633	365	10.1
26	10055	877.24	3.5071	3615	100.0
27	10101	863.21	3.4510	3590	99.3

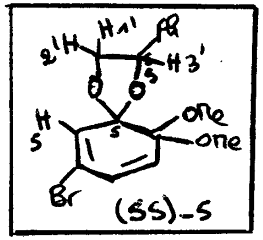




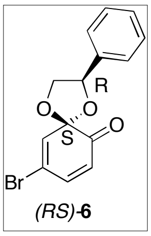
IFB163-3/CDCl3/22070330/DPX 300
51.1154
51.3845
72.1125
78.9710
97.5390
109.1321
118.9798
126.1873
128.3183
128.6020
130.8130
132.4857
137.9769
(ppm)
(SS)-5

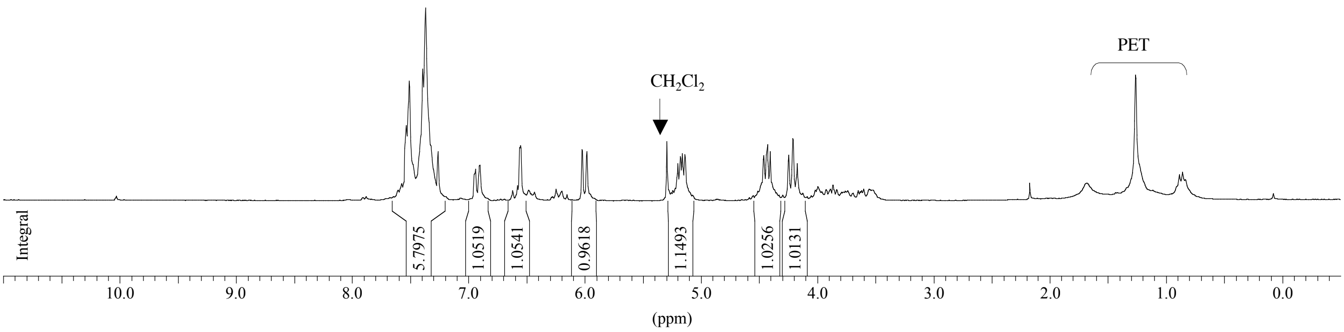
IFB163.3 /CDCl3/08120314/NOESY mix=600ms/DPX 300



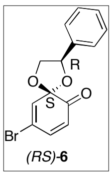


IFB175-1/CDCl3/SE01-109/AC 250

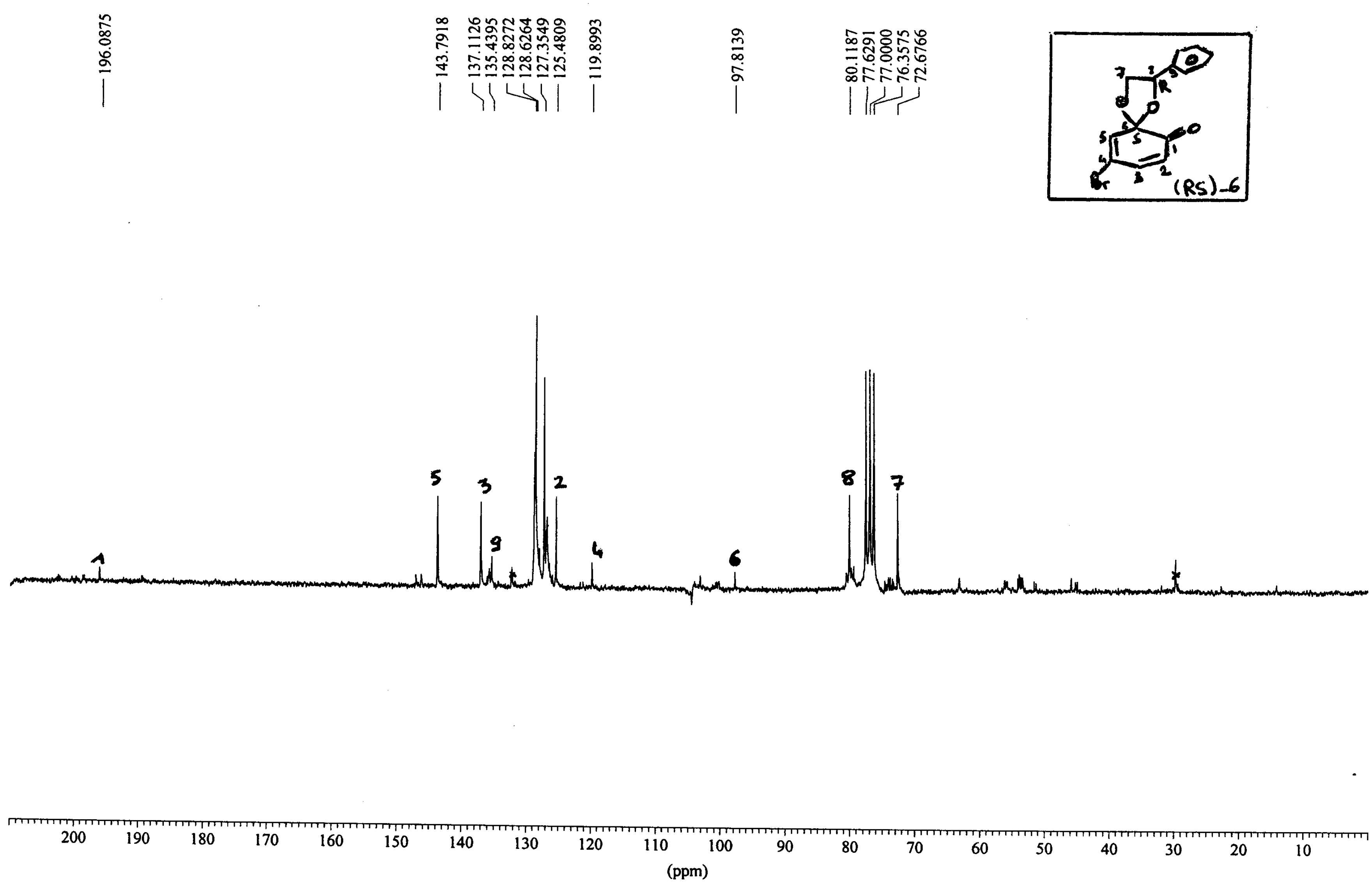




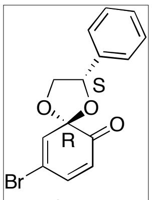
Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6754	1884.93	7.5357	380	39.3
2	6776	1878.22	7.5089	601	62.2
3	6870	1849.53	7.3942	661	68.4
4	6892	1842.82	7.3674	966	100.0
5	6980	1815.96	7.2600	248	25.6
6	7235	1738.14	6.9489	148	15.3
7	7242	1736.01	6.9403	158	16.4
8	7269	1727.77	6.9074	173	18.0
9	7275	1725.94	6.9001	180	18.6
10	7554	1640.79	6.5597	274	28.4
11	7560	1638.96	6.5524	277	28.7
12	7993	1506.82	6.0241	256	26.5
13	8027	1496.45	5.9826	246	25.4
14	8670	1300.22	5.1981	186	19.2
15	8688	1294.72	5.1761	222	23.0
16	8702	1290.45	5.1591	236	24.4
17	8720	1284.96	5.1371	232	24.0
18	9275	1115.59	4.4600	226	23.4
19	9294	1109.79	4.4368	228	23.6
20	9302	1107.35	4.4270	280	29.0
21	9320	1101.85	4.4051	246	25.5
22	9451	1061.88	4.2452	228	23.6
23	9481	1052.72	4.2086	313	32.4
24	9510	1043.87	4.1733	186	19.2



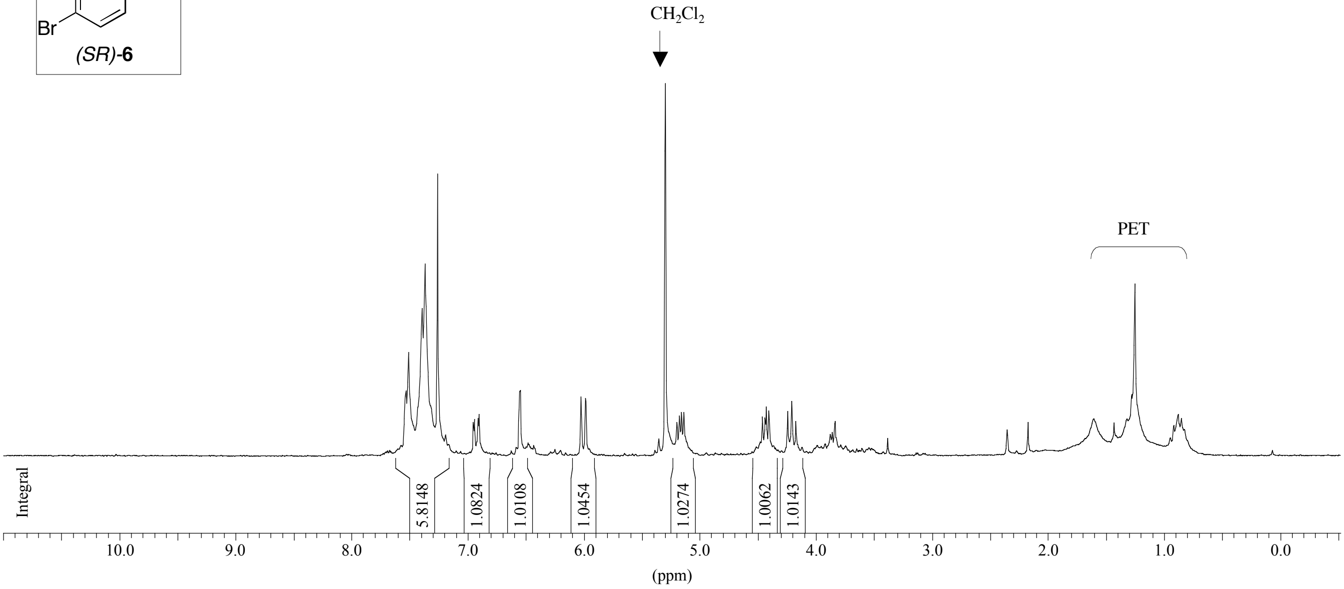
IFB175-1/CDCl3/SE01-ifb1751/DPX 200



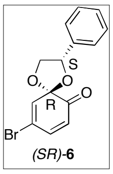
IFC25-1/CDCl3/DB10-109/AC 250



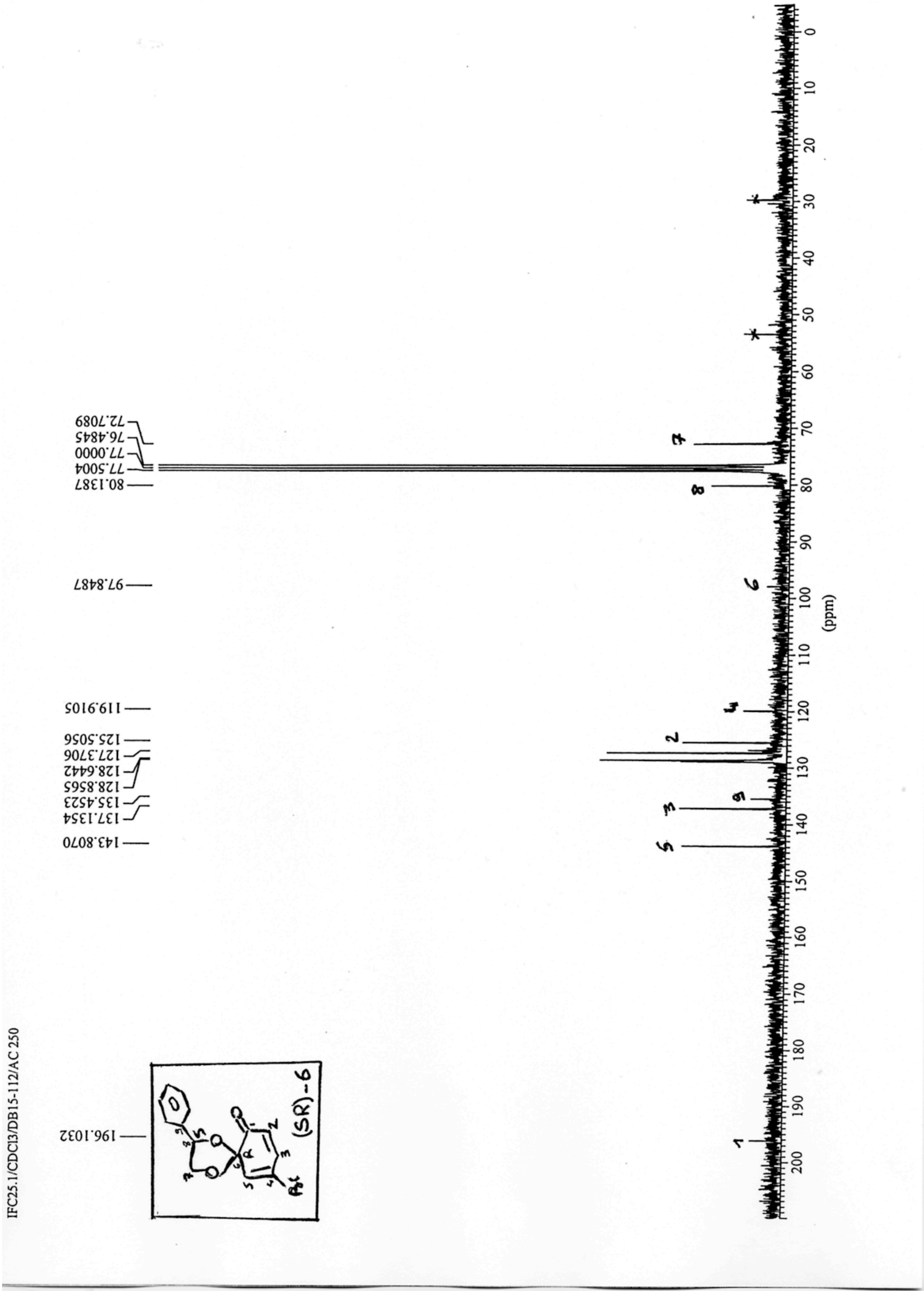
(SR)-**6**



Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6756	1884.02	7.5321	328	23.0
2	6775	1878.22	7.5089	520	36.6
3	6869	1849.53	7.3942	744	52.3
4	6892	1842.51	7.3661	969	68.1
5	6979	1815.96	7.2600	1423	100.0
6	7231	1739.06	6.9525	165	11.6
7	7239	1736.62	6.9428	181	12.7
8	7265	1728.68	6.9111	197	13.9
9	7272	1726.55	6.9025	205	14.4
10	7556	1639.88	6.5560	323	22.7
11	7563	1637.74	6.5475	328	23.1
12	7990	1507.43	6.0265	298	20.9
13	8024	1497.06	5.9850	289	20.3
14	8671	1299.61	5.1957	166	11.7
15	8689	1294.11	5.1737	199	14.0
16	8703	1289.84	5.1566	217	15.2
17	8721	1284.35	5.1347	215	15.1
18	9274	1115.59	4.4600	194	13.6
19	9292	1110.09	4.4380	188	13.2
20	9301	1107.35	4.4270	246	17.3
21	9319	1101.85	4.4051	223	15.7
22	9451	1061.57	4.2440	220	15.5
23	9482	1052.11	4.2062	275	19.3
24	9510	1043.56	4.1720	171	12.0

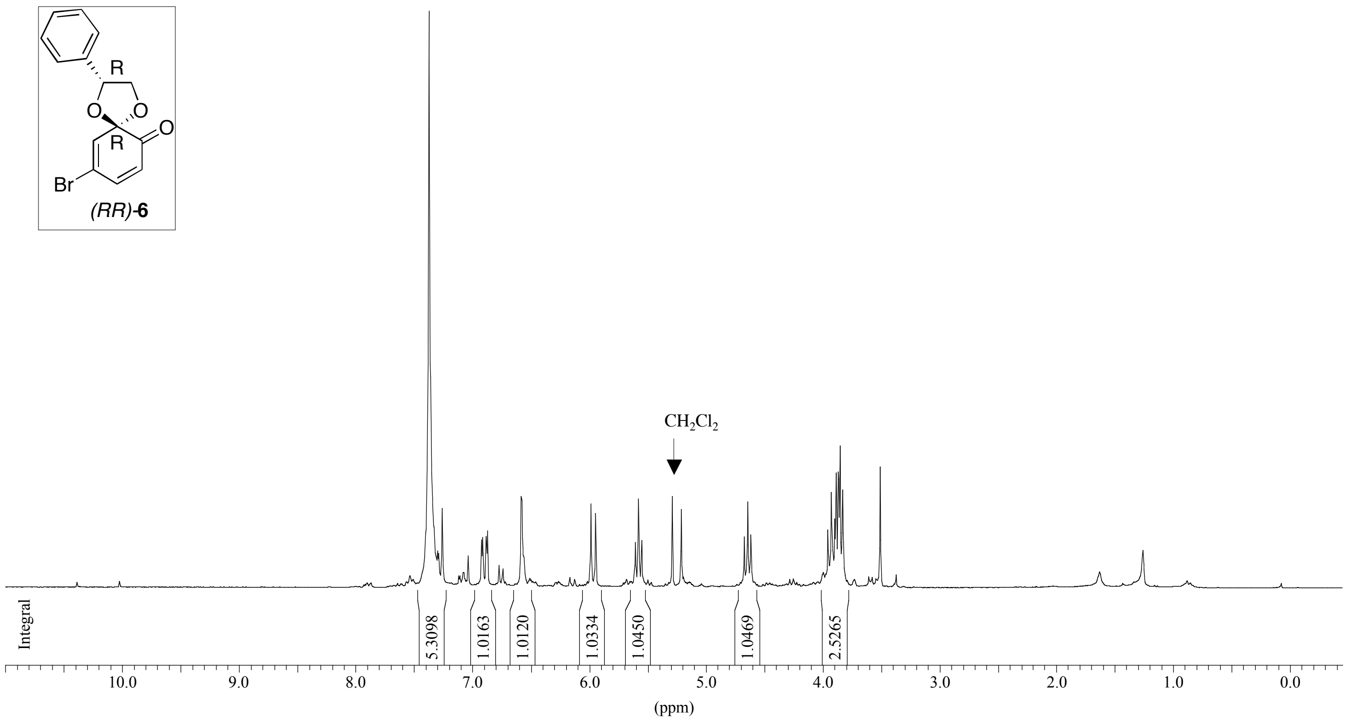


(SR)-**6**

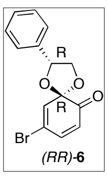


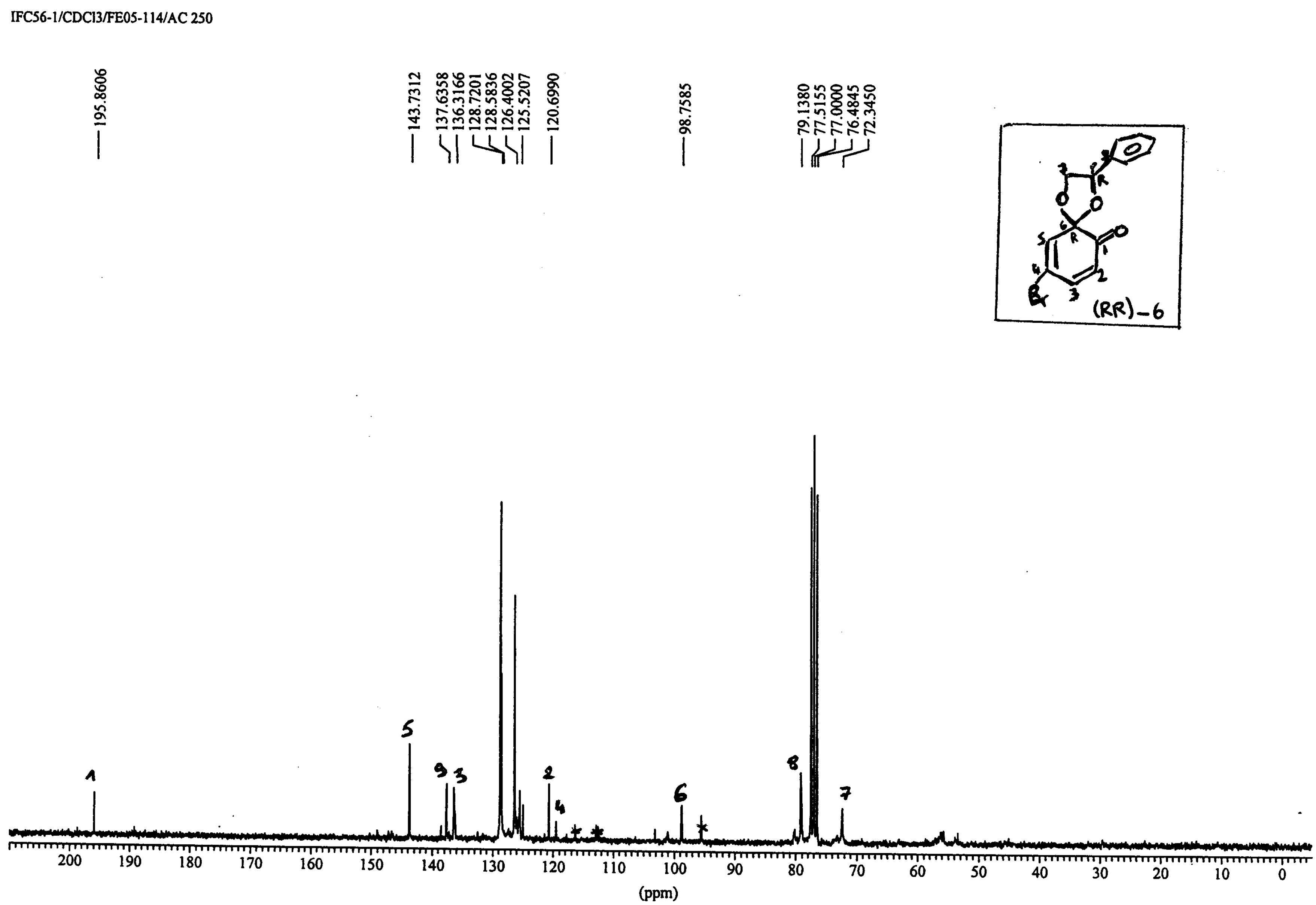
IFC25.1/CDCl3/DB15-112/AC 250
196.1032
143.8070
137.1354
135.4523
128.8565
128.6442
127.3706
125.5056
119.9105
97.8487
80.1387
77.5004
77.0000
76.4845
72.7089
(SR)-6
(ppm)

IFC56-2/CDCl3/FE05-113/AC 250

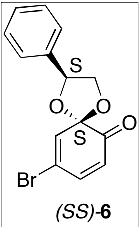


Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6887	1844.04	7.3722	2013	100.0
2	6979	1815.96	7.2600	275	13.6
3	7255	1731.74	6.9233	163	8.1
4	7262	1729.60	6.9147	173	8.6
5	7288	1721.67	6.8830	176	8.7
6	7296	1719.22	6.8732	195	9.7
7	7533	1646.90	6.5841	317	15.7
8	7540	1644.76	6.5755	302	15.0
9	8021	1497.97	5.9887	290	14.4
10	8055	1487.60	5.9472	257	12.8
11	8333	1402.76	5.6080	154	7.7
12	8355	1396.04	5.5812	308	15.3
13	8378	1389.02	5.5531	160	8.0
14	9100	1168.69	4.6723	175	8.7
15	9124	1161.36	4.6430	297	14.7
16	9148	1154.04	4.6137	176	8.7
17	9686	989.85	3.9573	200	9.9
18	9710	982.53	3.9280	331	16.5
19	9719	979.78	3.9170	145	7.2
20	9734	975.21	3.8987	238	11.8
21	9744	972.15	3.8865	399	19.8
22	9745	971.85	3.8853	391	19.4
23	9760	967.27	3.8670	402	20.0
24	9772	963.61	3.8524	482	23.9
25	9790	958.12	3.8304	340	16.9

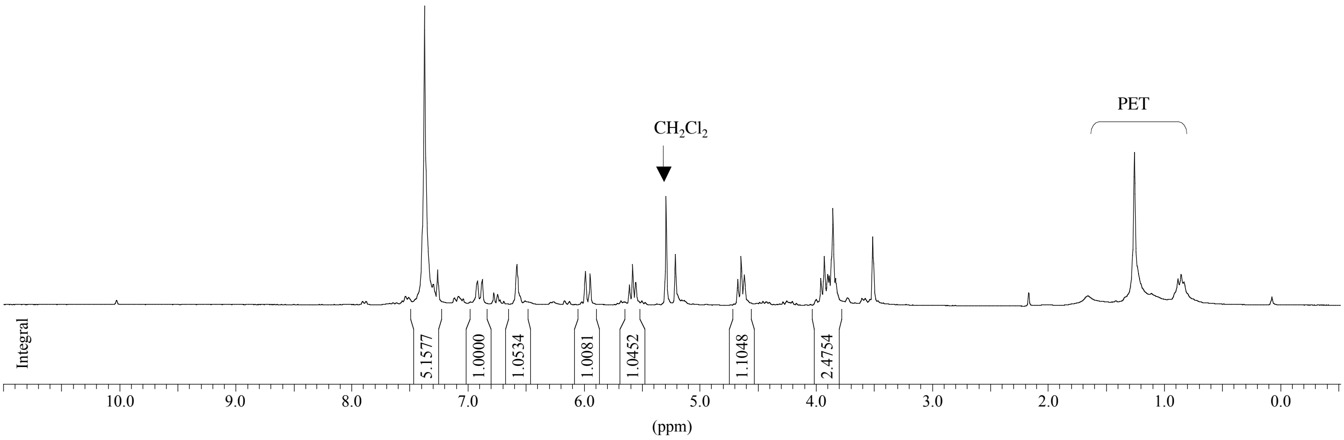



IFC56-1/CDCl3/FE05-114/AC 250
195.8606
143.7312
137.6358
136.3166
128.7201
128.5836
126.4002
125.5207
120.6990
98.7585
79.1380
77.5155
77.0000
76.4845
72.3450
(RR)-6
(ppm)

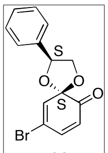
IFB176-1/CDCl3/SE02-117/AC 250



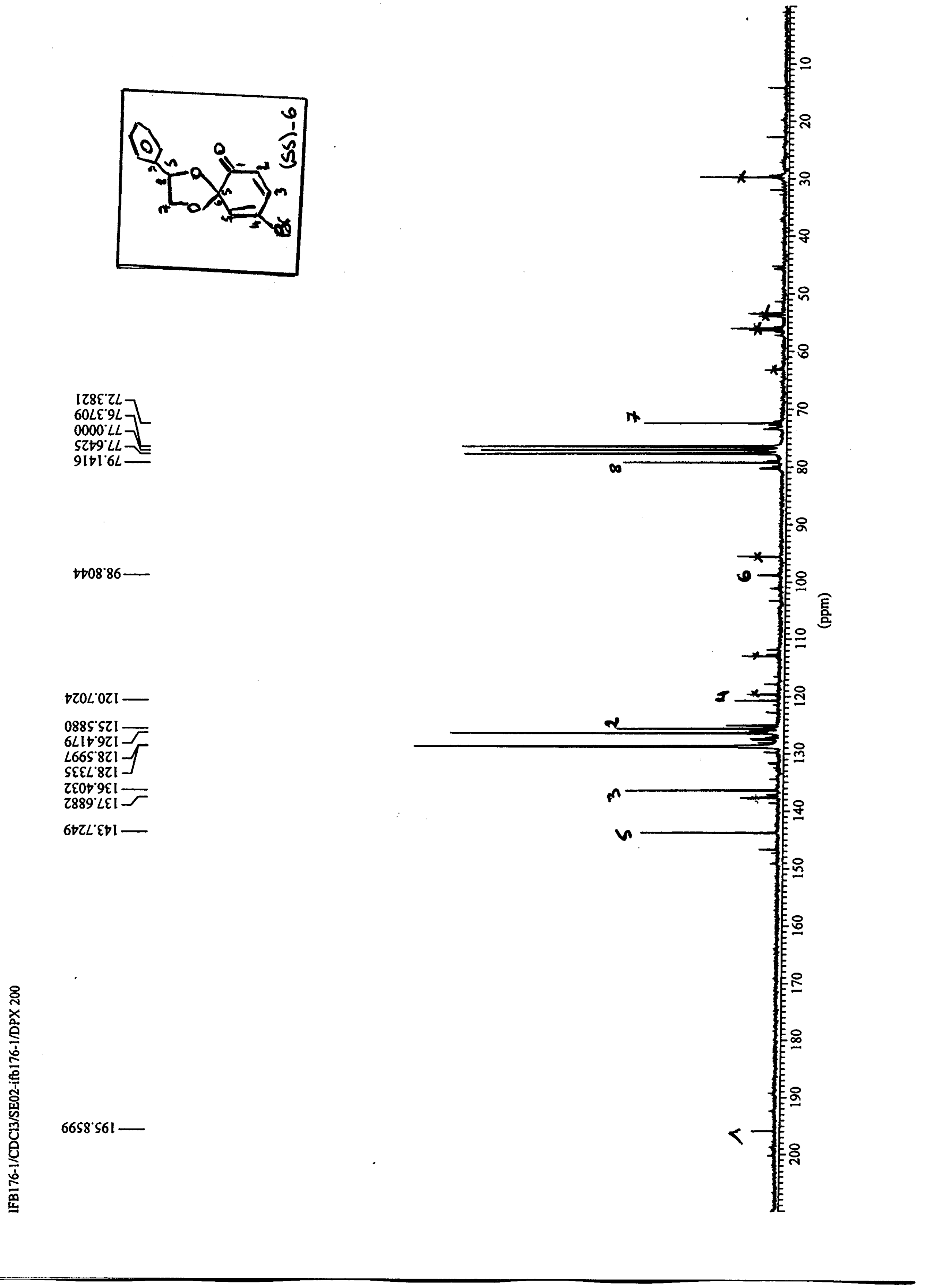
(SS)-**6**



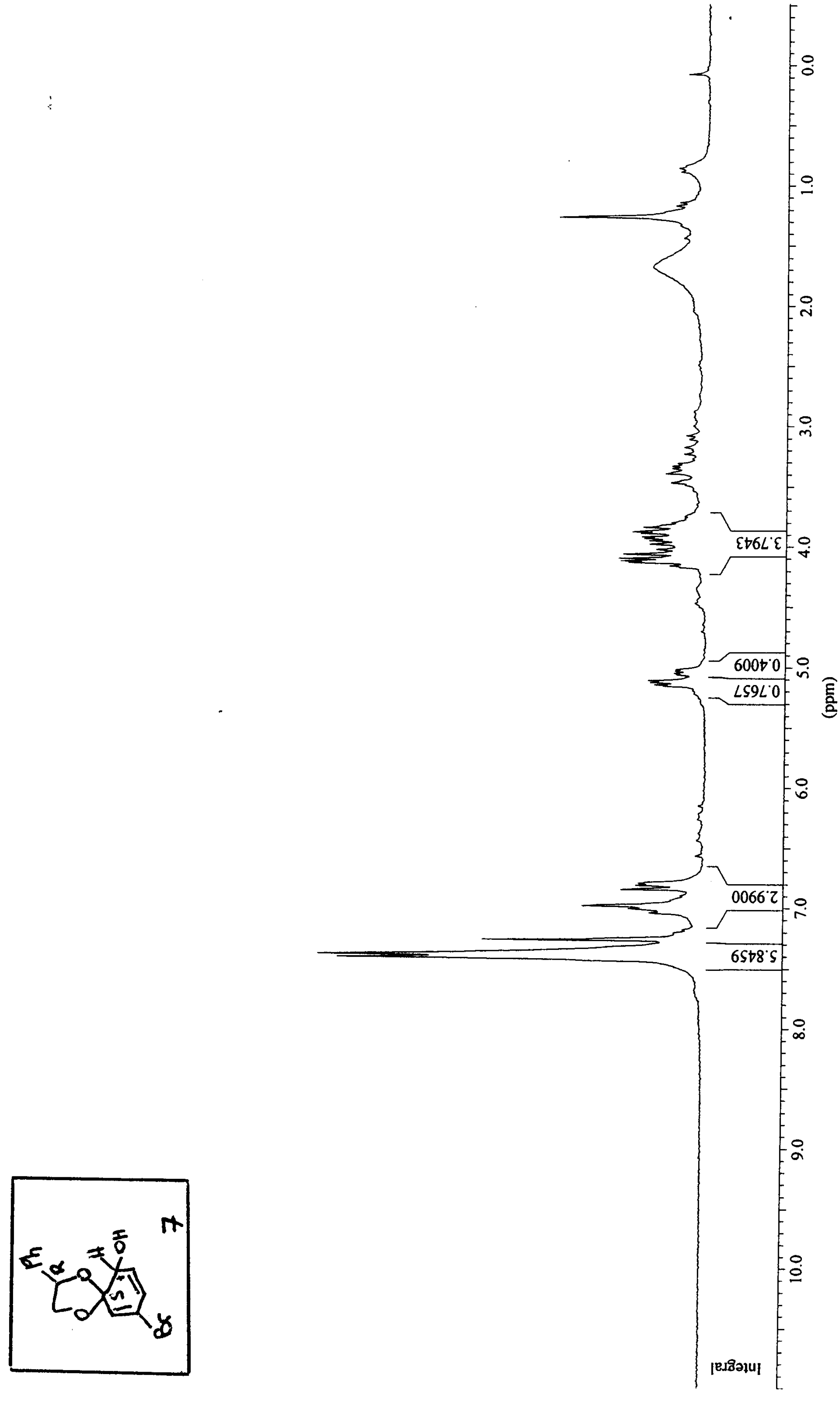
Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6887	1844.04	7.3722	2168	100.0
2	6947	1825.73	7.2990	150	6.9
3	6953	1823.90	7.2917	149	6.9
4	6979	1815.96	7.2600	254	11.7
5	7254	1732.04	6.9245	152	7.0
6	7261	1729.90	6.9159	175	8.1
7	7288	1721.67	6.8830	161	7.4
8	7295	1719.53	6.8745	186	8.6
9	7534	1646.59	6.5829	271	12.5
10	7539	1645.07	6.5768	296	13.6
11	8021	1497.97	5.9887	244	11.3
12	8054	1487.90	5.9484	226	10.4
13	8332	1403.06	5.6093	147	6.8
14	8354	1396.35	5.5824	295	13.6
15	8377	1389.33	5.5544	163	7.5
16	9099	1168.99	4.6735	186	8.6
17	9123	1161.67	4.6442	355	16.4
18	9147	1154.34	4.6149	217	10.0
19	9685	990.16	3.9585	195	9.0
20	9709	982.83	3.9293	356	16.4
21	9733	975.51	3.9000	221	10.2
22	9744	972.15	3.8865	210	9.7
23	9771	963.91	3.8536	701	32.3
24	9789	958.42	3.8316	194	9.0



(SS)-**6**

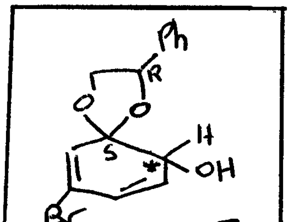

IFB176-1/CDCl3/SE02-ifb176-1/DPX 200
195.8599
143.7249
137.6882
136.4032
128.7335
128.5997
126.4179
125.5880
120.7024
98.8044
79.1416
77.6425
77.0000
76.3709
72.3821
(S)-6
(ppm)

IFB190-1/CDCl3/OC15-115/AC 250



Peak List   Date: 3.05.2004   Time: 10:51

File Name : C:\WIN1D\SPC\ASP3000\OC15\115001.1R
Peak Results saved in File :
Nucleus : 1H
SF : 250.132854 MHz
OFFSET : 15.7760 ppm
SW_p : 5000.00 Hz
SI : 16384



7

Peak Picking Parameter

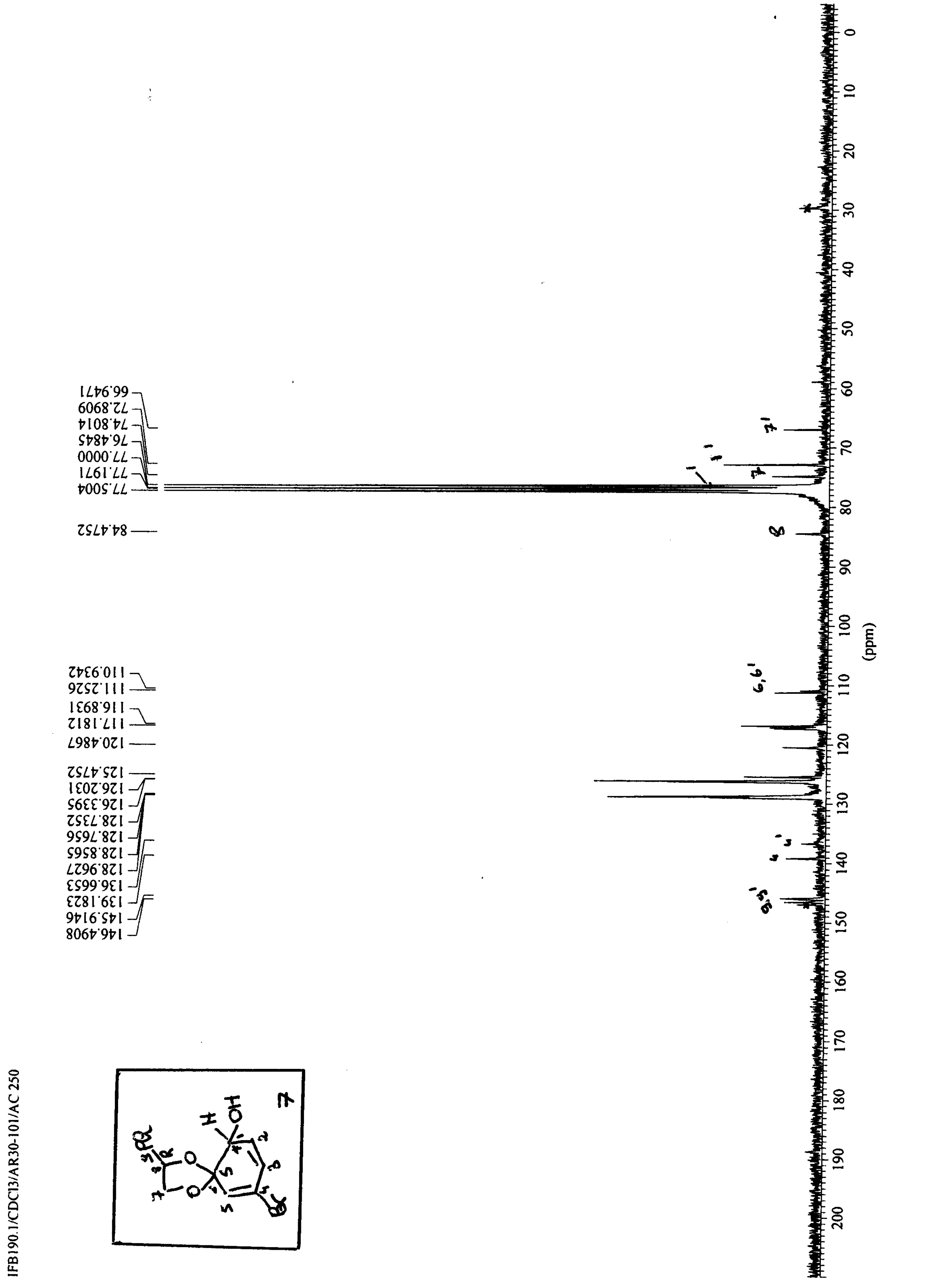
Peak constant PC = 1.00
Noise = 1
Sens. level = 5

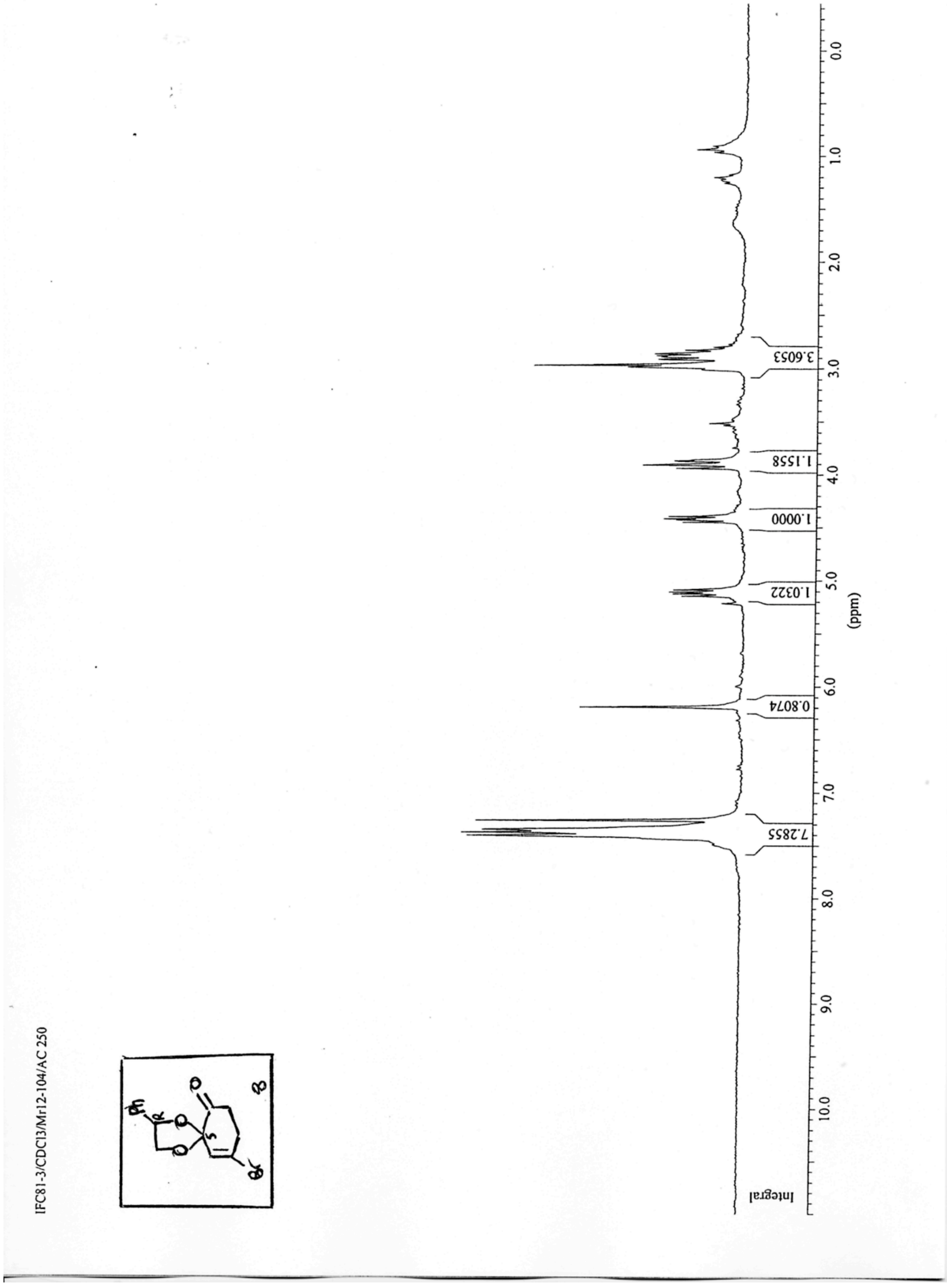
Peak Picking region

Start(ppm/Hz)	End(ppm/Hz)	MI(%)	MAXI(%)
7.47/ 1869.1	7.06/ 1765.6	43.73	106.64
7.14/ 1786.1	6.69/ 1673.4	11.09	39.41
5.22/ 1305.7	4.94/ 1236.4	5.77	16.57
4.40/ 1100.3	3.74/ 936.4	7.25	30.38

Peak Picking results

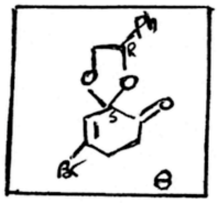
Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6862	1851.98	7.4040	1049	94.9
2	6883	1845.57	7.3783	1106	100.0
3	6980	1815.96	7.2600	632	57.1
4	7163	1760.12	7.0367	156	14.1
5	7192	1751.27	7.0013	215	19.5
6	7211	1745.47	6.9782	348	31.4
7	7322	1711.59	6.8427	238	21.5
8	7350	1703.05	6.8086	195	17.6
9	7363	1699.08	6.7927	188	17.0
10	8706	1289.23	5.1542	122	11.0
11	8716	1286.18	5.1420	147	13.3
12	8733	1280.99	5.1212	159	14.4
13	8744	1277.63	5.1078	164	14.8
14	8784	1265.43	5.0590	82	7.5
15	8795	1262.07	5.0456	90	8.2
16	8809	1257.80	5.0285	87	7.9
17	8820	1254.44	5.0151	85	7.7
18	9515	1042.34	4.1672	82	7.4
19	9525	1039.29	4.1550	106	9.6
20	9548	1032.27	4.1269	224	20.3
21	9558	1029.22	4.1147	246	22.3
22	9576	1023.73	4.0927	251	22.7
23	9604	1015.18	4.0586	238	21.6
24	9637	1005.11	4.0183	144	13.1
25	9671	994.74	3.9768	164	14.8
26	9696	987.11	3.9463	165	14.9
27	9710	982.83	3.9293	178	16.1
28	9722	979.17	3.9146	186	16.8
29	9745	972.15	3.8865	190	17.2
30	9758	968.19	3.8707	214	19.3
31	9789	958.73	3.8329	182	16.5
32	9821	948.96	3.7938	102	9.2


IFB190.1/CDCl3/AR30-101/AC 250
66.9471
72.8909
74.8014
76.4845
77.0000
77.1971
77.5004
84.4752
110.9342
111.2526
116.8931
117.1812
120.4867
125.4752
126.2031
126.3395
128.7352
128.7656
128.8565
128.9627
136.6653
139.1823
145.9146
146.4908
(ppm)


IFC81-3/CDCl3/Mr12-104/AC 250
B
Integral
7.2855
0.8074
1.0322
1.0000
1.1558
3.6053
(ppm)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

Peak List   Date: 9.04.2004   Time: 12:29

File Name : C:\WIN1D\SPC\ASP3000\MR120F\104001.1R
Peak Results saved in File :
Nucleus : 1H
SF : 250.132854 MHz
OFFSET : 15.7748 ppm
SW_p : 5000.00 Hz
SI : 16384



Peak Picking Parameter

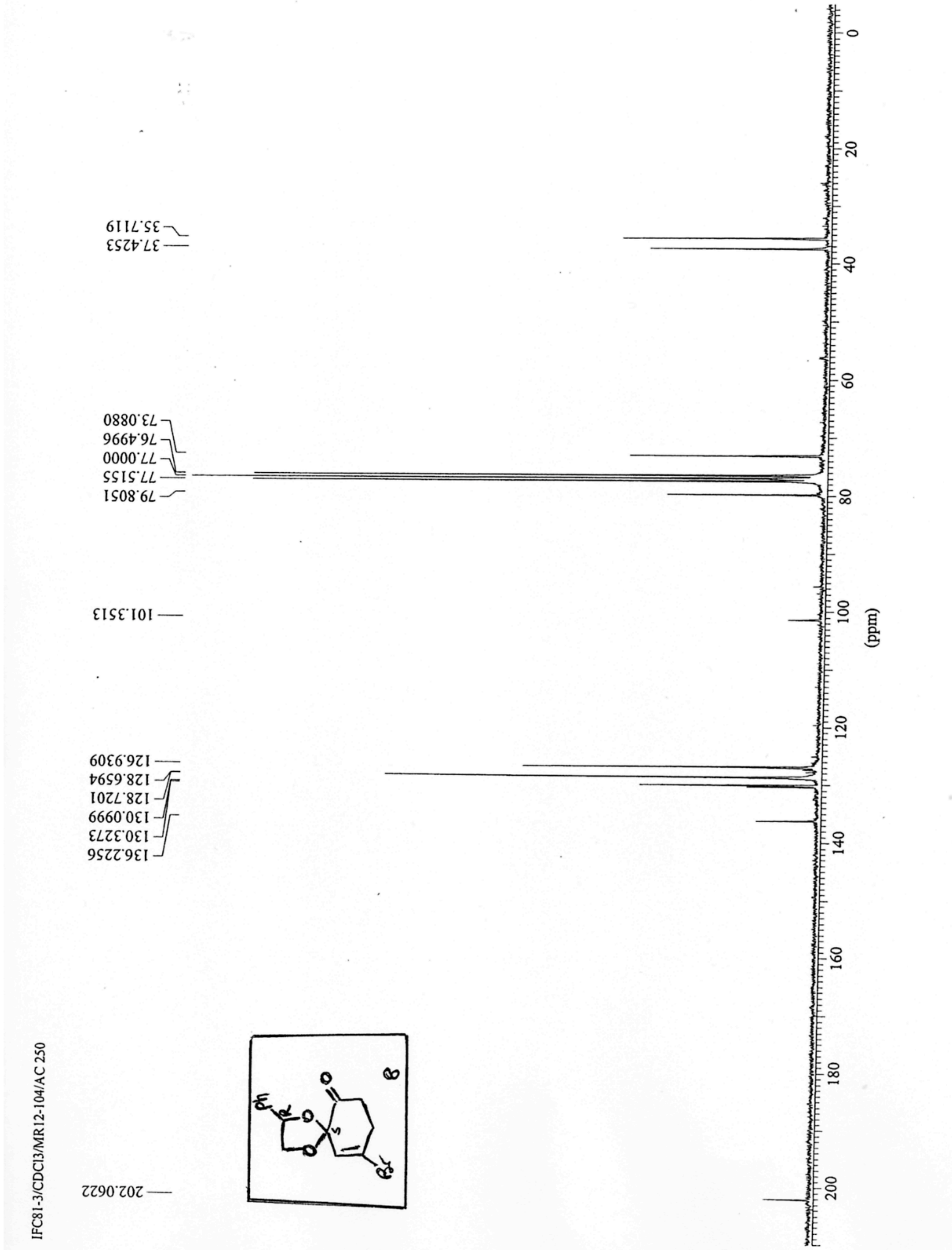
Peak constant PC = 1.00
Noise = 7
Sens. level = 28

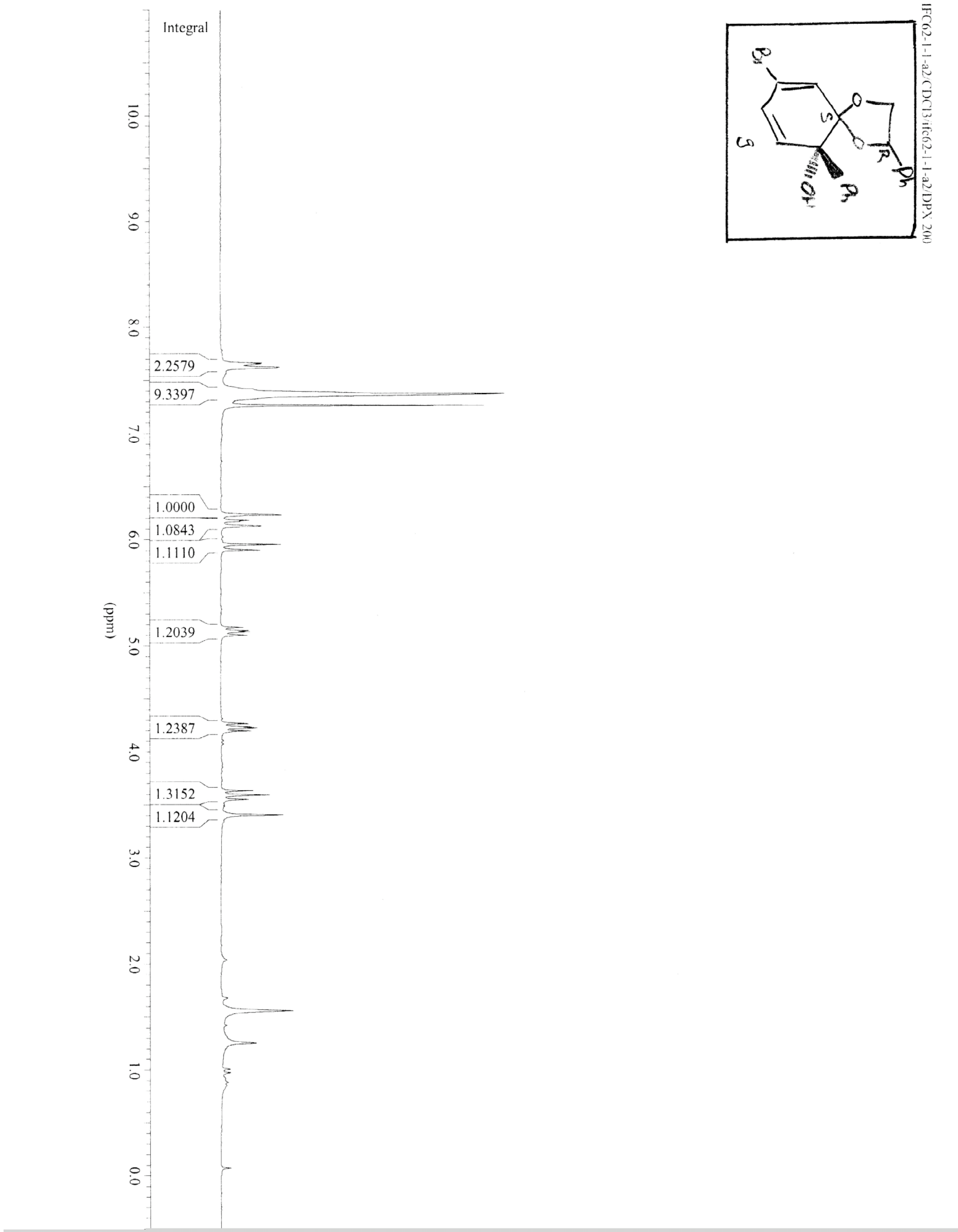
Peak Picking region

Start(ppm/Hz)	End(ppm/Hz)	MI(%)	MAXI(%)
15.77/ 3945.8	-4.21/-1054.2	-0.98	100.00

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6862	1851.67	7.4027	4710	98.0
2	6888	1843.74	7.3710	4807	100.0
3	6909	1837.33	7.3454	4441	92.4
4	6979	1815.96	7.2600	4562	94.9
5	7856	1548.33	6.1900	2783	57.9
6	8716	1285.87	5.1408	1066	22.2
7	8734	1280.38	5.1188	1211	25.2
8	8747	1276.41	5.1029	1273	26.5
9	8766	1270.62	5.0798	1205	25.1
10	9286	1111.92	4.4453	1063	22.1
11	9305	1106.13	4.4222	1171	24.4
12	9314	1103.38	4.4112	1379	28.7
13	9332	1097.89	4.3892	1298	27.0
14	9702	984.97	3.9378	1180	24.5
15	9732	975.82	3.9012	1743	36.3
16	9762	966.66	3.8646	1208	25.1
17	9768	964.83	3.8573	940	19.5
18	10466	751.82	3.0057	756	15.7
19	10488	745.10	2.9788	1989	41.4
20	10505	739.91	2.9581	3618	75.3
21	10546	727.40	2.9081	1489	31.0
22	10567	720.99	2.8824	1426	29.7
23	10574	718.86	2.8739	1519	31.6
24	10590	713.97	2.8544	1553	32.3
25	10615	706.35	2.8239	1033	21.5
26	10636	699.94	2.7983	547	11.4

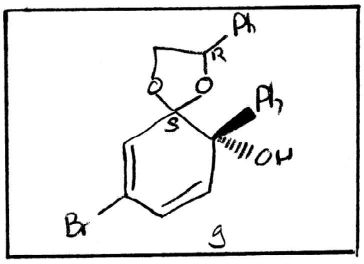

IFC81-3/CDCl3/MR12-104/AC 250
202.0622
136.2256
130.3273
130.0999
128.7201
128.6594
126.9309
101.3513
79.8051
77.5155
77.0000
76.4996
73.0880
37.4253
35.7119
(ppm)
200
180
160
140
120
100
80
60
40
20
0
8


IFC62-1-1-a2/CDCl3/ifc62-1-1-a2/DPX 200
Integral
2.2579
9.3397
1.0000
1.0843
1.1110
1.2039
1.2387
1.3152
1.1204
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

Peak List

Date: 5.06.2004 Time: 18:23

File Name	:	c:\cesamo\lcsv\sasn\isabelle\rmnthe~1\juin20~1\04
Peak Results saved in File	:	
Nucleus	:	off
SF	:	200.160019 MHz
OFFSET	:	11.9086 ppm
SW_p	:	2604.17 Hz
SI	:	16384



Peak Picking Parameter

Peak constant PC	=	1.00
Noise	=	259
Sens. level	=	1037

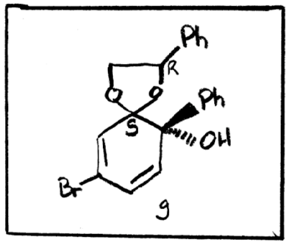
Peak Picking region

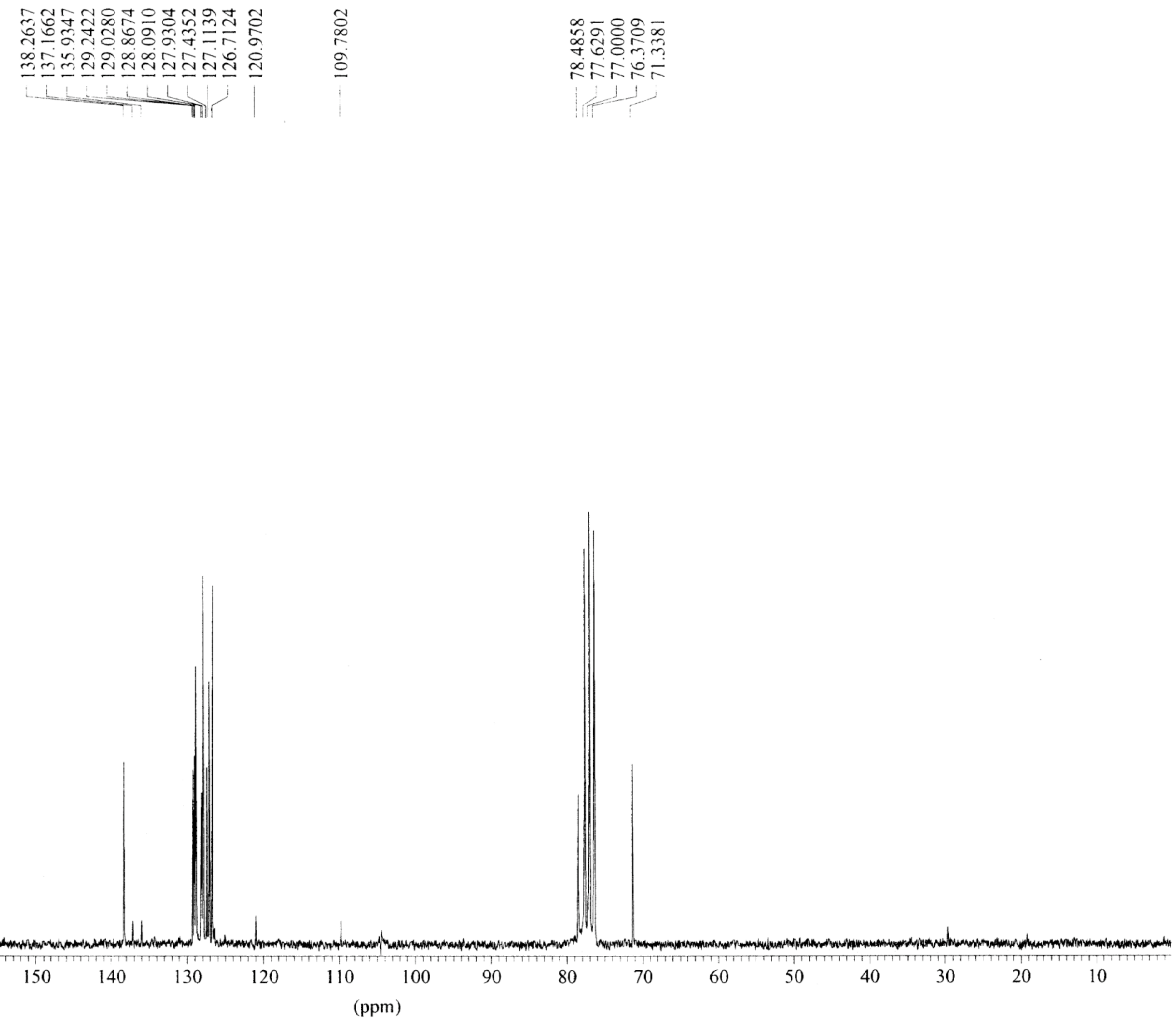
Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
7.91/ 1583.3	7.58/ 1517.2	11.54	33.66
7.45/ 1490.2	7.08/ 1416.9	57.59	107.26
6.28/ 1257.3	6.21/ 1242.2	16.16	26.82
6.21/ 1242.7	6.07/ 1214.3	6.71	16.16
6.00/ 1201.7	5.84/ 1168.5	5.31	25.21
5.25/ 1051.8	4.95/ 991.4	6.31	18.58
4.29/ 858.9	4.16/ 833.0	6.31	16.77
3.72/ 745.5	3.52/ 703.6	5.71	24.41
3.46/ 692.1	3.36/ 673.4	16.97	25.42

Peak Picking results

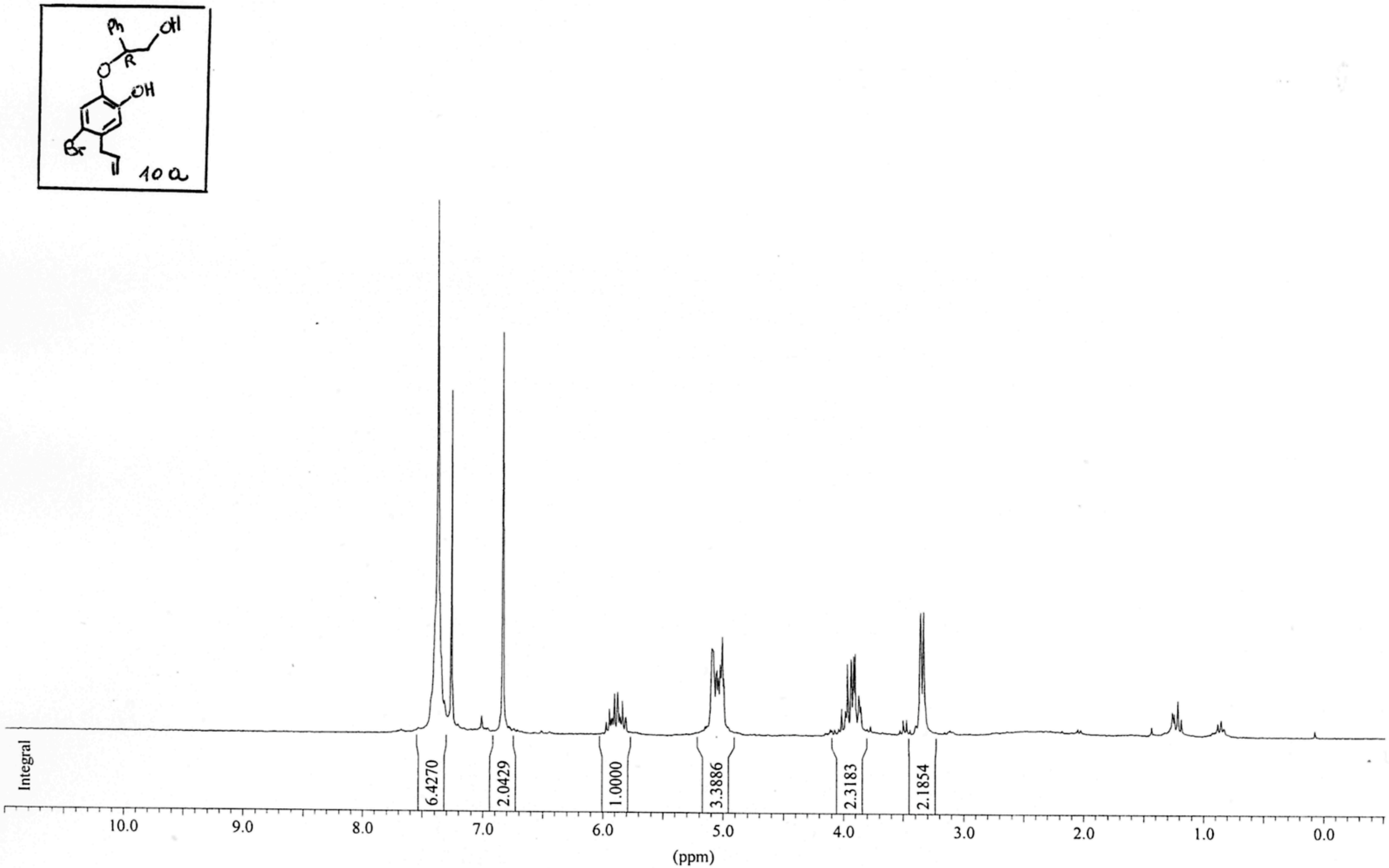
Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	5348	1533.59	7.6618	25623	14.6
2	5360	1531.68	7.6523	25303	14.4
3	5398	1525.64	7.6221	36690	20.9
4	5408	1524.05	7.6142	34147	19.5
5	5717	1474.94	7.3688	175287	100.0
6	5854	1453.16	7.2600	162455	92.7
7	7144	1248.12	6.2356	32676	18.6
8	7154	1246.53	6.2277	37850	21.6
9	7214	1237.00	6.1800	17546	10.0
10	7225	1235.25	6.1713	14226	8.1
11	7277	1226.98	6.1300	25350	14.5
12	7288	1225.23	6.1213	23139	13.2
13	7501	1191.38	5.9521	37195	21.2
14	7564	1181.36	5.9021	24349	13.9
15	8482	1035.45	5.1731	13989	8.0
16	8521	1029.25	5.1422	17767	10.1
17	8537	1026.71	5.1294	17944	10.2
18	8576	1020.51	5.0985	16587	9.5
19	9626	853.62	4.2647	17018	9.7
20	9665	847.42	4.2337	19149	10.9
21	9677	845.51	4.2242	22495	12.8
22	9716	839.31	4.1932	18823	10.7
23	10421	727.26	3.6334	19890	11.3
24	10474	718.83	3.5913	30188	17.2
25	10527	710.41	3.5492	17094	9.8
26	10710	681.32	3.4039	38560	22.0

IFC62-1-1-a2/CDCl3/IFC62-1-1-a2/DPX 200



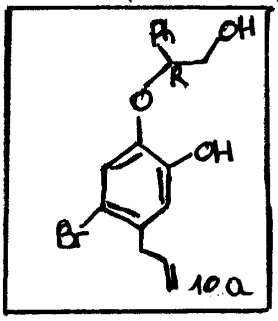


IFC54.5/CDCl3/FV07-101/AC 250



Peak List Date: 25.04.2004 Time: 13:13

File Name : c:\cesamo\lcsv\sasn\isabelle\rmnthe~1\fevrie~1\fv(
Peak Results saved in File :
Nucleus : 1H
SF : 250.132854 MHz
OFFSET : 15.7760 ppm
SW_p : 5000.00 Hz
SI : 16384



Peak Picking Parameter

Peak constant PC = 1.00
Noise = 5
Sens. level = 22

Peak Picking region

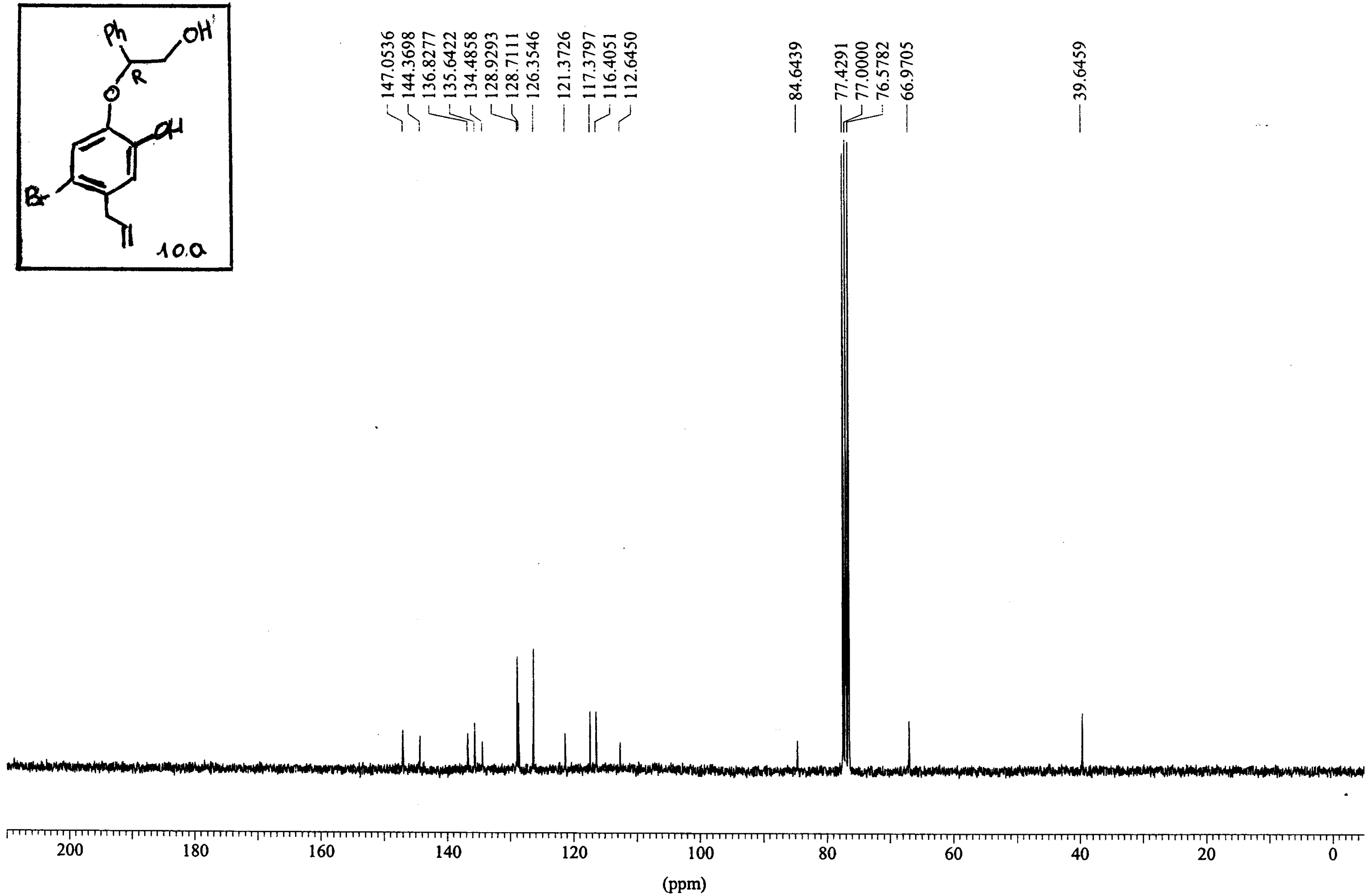
Start (ppm/Hz)	End (ppm/Hz)	MI (%)	MAXI (%)
9.16/ 2292.3	4.82/ 1206.5	25.31	124.84
6.26/ 1565.4	5.71/ 1427.8	1.18	11.81
5.27/ 1319.1	4.76/ 1190.0	9.41	21.24
3.54/ 884.9	3.11/ 778.4	15.56	25.47
4.10/ 1024.6	3.76/ 941.3	3.89	16.44

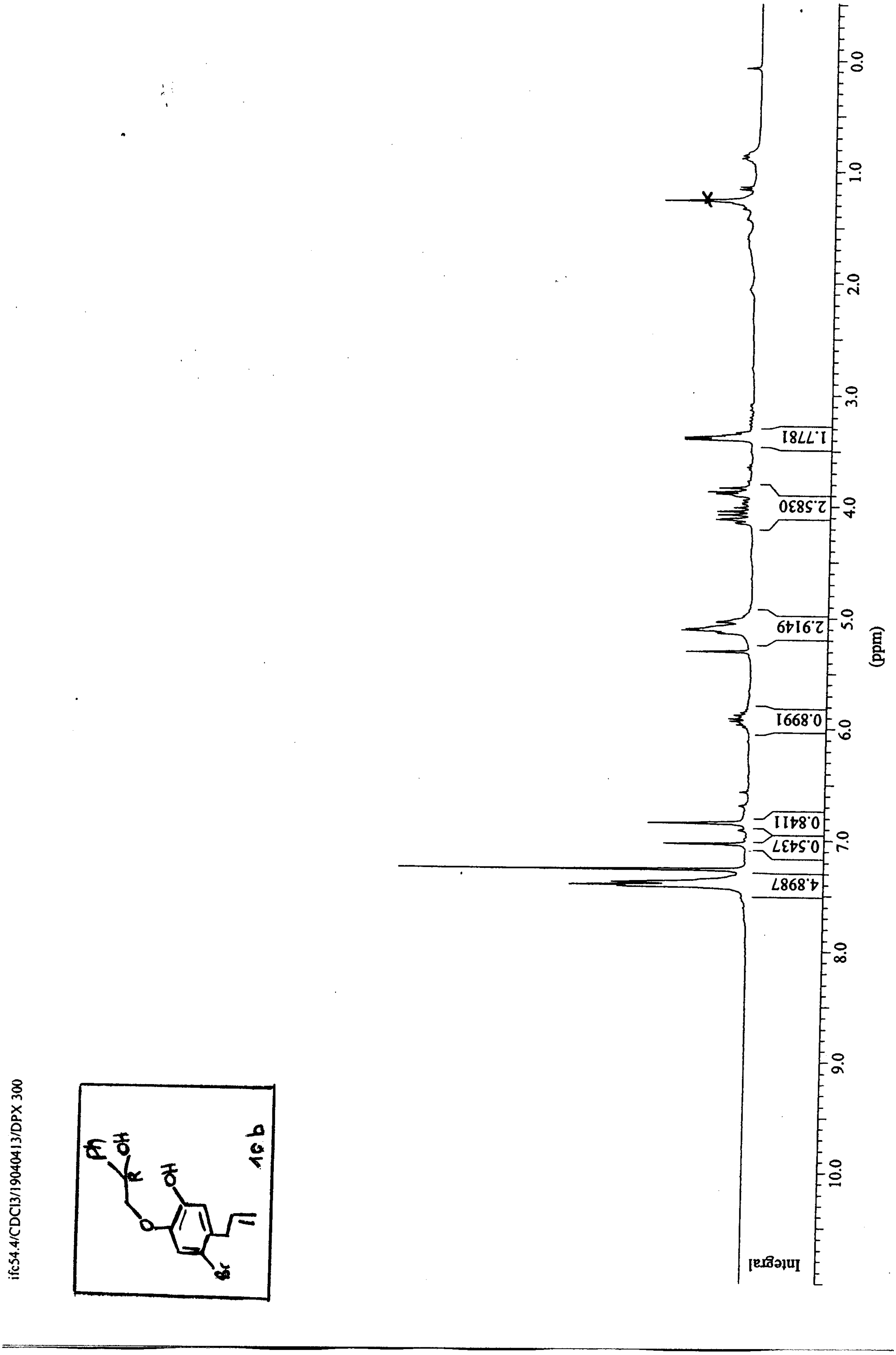
Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6887	1844.35	7.3735	13803	100.0
2	6980	1815.96	7.2600	8884	64.4
3	7329	1709.46	6.8342	10416	75.5
4	8038	1493.09	5.9692	320	2.3
5	8060	1486.37	5.9423	657	4.8
6	8072	1482.71	5.9277	427	3.1
7	8081	1479.97	5.9167	420	3.0
8	8093	1476.30	5.9021	1062	7.7
9	8115	1469.59	5.8752	1098	8.0
10	8127	1465.93	5.8606	545	3.9
11	8136	1463.18	5.8496	461	3.3
12	8148	1459.52	5.8350	868	6.3
13	8170	1452.81	5.8081	443	3.2
14	8757	1273.67	5.0920	2242	16.2
15	8760	1272.75	5.0883	2129	15.4
16	8765	1271.23	5.0822	2172	15.7
17	8770	1269.70	5.0761	1712	12.4
18	8785	1265.12	5.0578	1583	11.5
19	8790	1263.60	5.0517	1680	12.2
20	8800	1260.54	5.0395	1473	10.7
21	8813	1256.58	5.0236	1826	13.2
22	8821	1254.14	5.0139	1988	14.4
23	8826	1252.61	5.0078	2544	18.4
24	8837	1249.25	4.9944	1465	10.6
25	9640	1004.20	4.0147	697	5.0
26	9666	996.26	3.9829	655	4.7
27	9679	992.30	3.9671	1861	13.5
28	9705	984.36	3.9354	1987	14.4
29	9720	979.78	3.9171	2067	15.0
30	9731	976.43	3.9036	2137	15.5
31	9758	968.19	3.8707	1050	7.6

2	9770	964.52	3.8560	764	5.5
3	10178	840.01	3.3583	3191	23.1
4	10200	833.30	3.3314	3214	23.3

IFC54.5/CDCl3/0020413/DXP 200





ifc54.4/CDCl3/19040413/DPX 300
10 b
Br
OH
R
Ph
Integral
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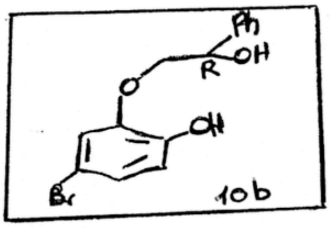
0.5437
0.8411
0.8991
2.9149
2.5830
1.7781
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

Peak List Date: 20.04.2004 Time: 11:53

File Name : c:\cesamo\lcsv\sasn\isabelle\dpx300~1\avil04r\isa
Peak Results saved in File :
Nucleus : off
SF : 300.130007 MHz
OFFSET : 19.7618 ppm
SW_p : 6172.84 Hz
SI : 16384



10b

Peak Picking Parameter

Peak constant PC = 1.00
Noise = 330
Sens. level = 1318

Peak Picking region

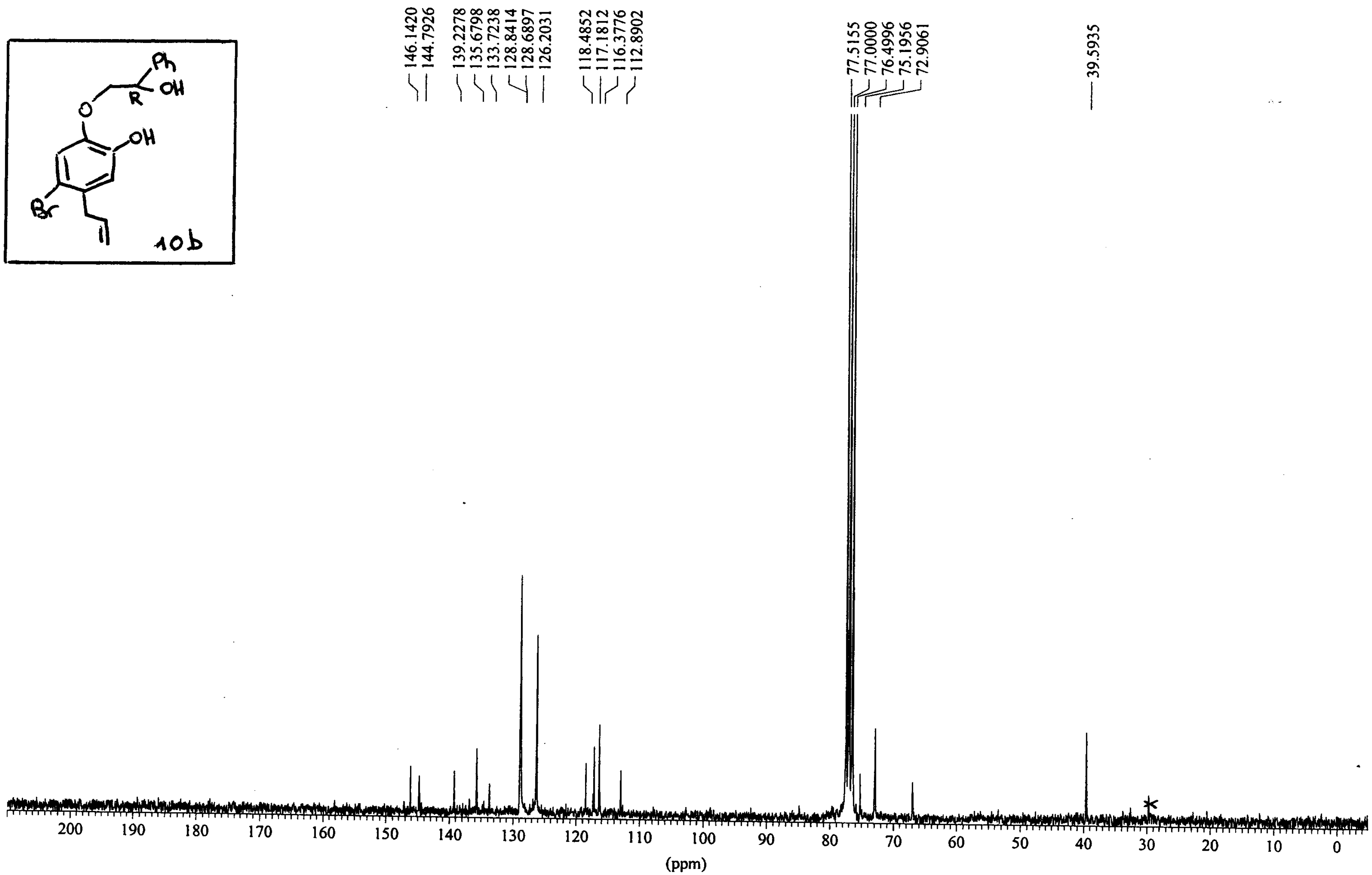
Start(ppm/Hz)	End(ppm/Hz)	MI(%)	MAXI(%)
4.26/ 1277.7	3.68/ 1104.1	2.76	25.29
3.52/ 1056.9	3.26/ 977.8	14.61	25.44
4.03/ 1209.6	4.01/ 1202.8	21.49	22.95
7.53/ 2258.8	7.24/ 2174.4	27.28	57.57
7.13/ 2141.3	6.69/ 2007.5	11.61	39.60
6.11/ 1832.3	5.73/ 1720.0	2.16	11.64
5.15/ 1544.9	4.91/ 1474.4	7.23	23.87

Peak Picking results

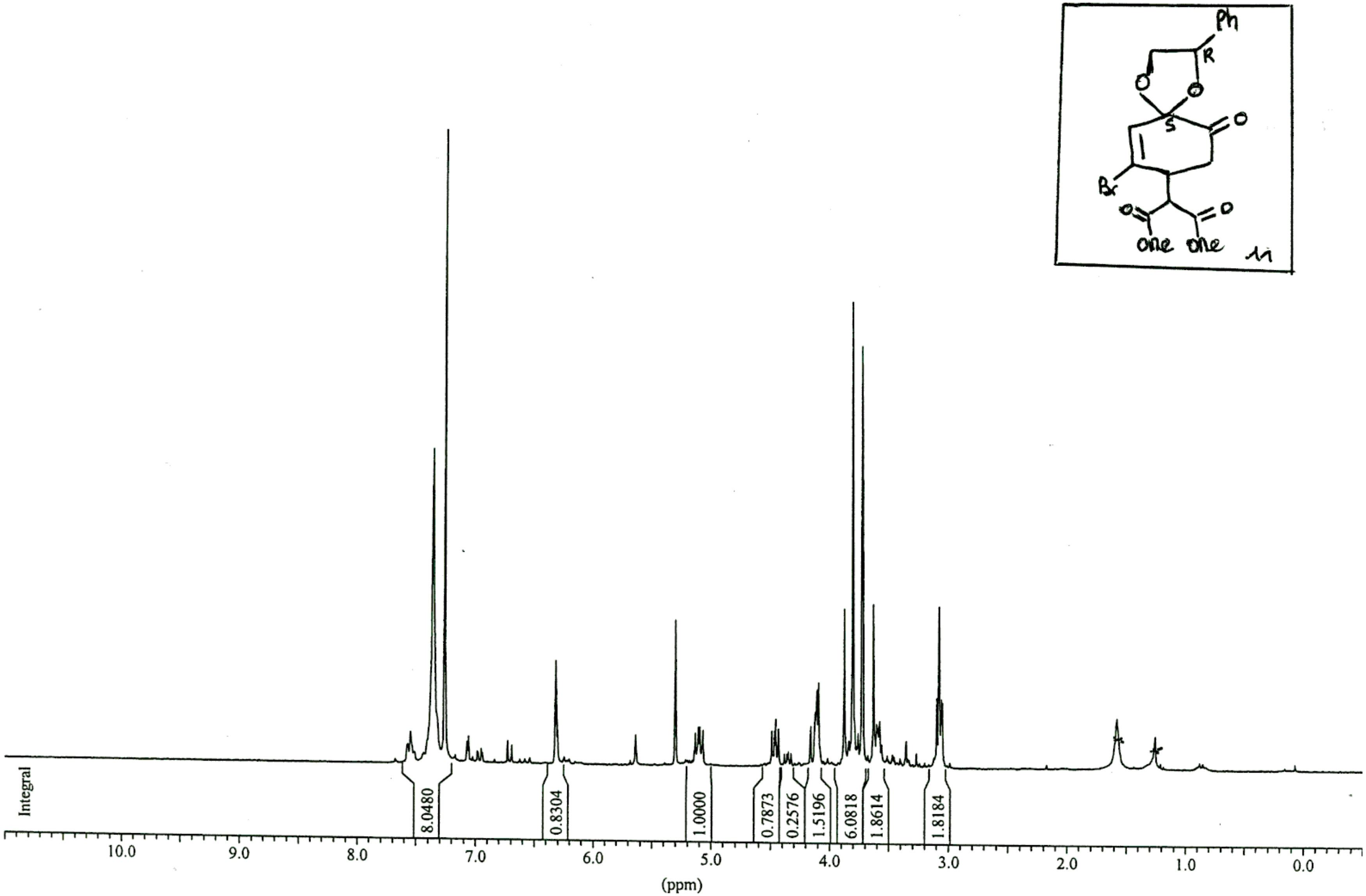
Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	9837	2224.91	7.4132	69063	37.4
2	9848	2220.76	7.3993	94363	51.1
3	9867	2213.61	7.3755	72171	39.1
4	9959	2178.94	7.2600	184552	100.0
5	10143	2109.62	7.0290	44872	24.3
6	10292	2053.48	6.8420	53420	28.9
7	10975	1796.16	5.9846	4372	2.4
8	10992	1789.75	5.9633	7788	4.2
9	11020	1779.20	5.9281	11069	6.0
10	11037	1772.80	5.9068	11660	6.3
11	11064	1762.62	5.8729	8996	4.9
12	11081	1756.22	5.8515	5476	3.0
13	11652	1541.09	5.1347	18494	10.0
14	11660	1538.07	5.1247	19894	10.8
15	11674	1532.80	5.1071	37834	20.5
16	11684	1529.03	5.0946	35140	19.0
17	11697	1524.13	5.0782	24996	13.5
18	11731	1511.32	5.0356	19240	10.4
19	12435	1246.09	4.1518	9675	5.2
20	12443	1243.07	4.1418	10489	5.7
21	12462	1235.91	4.1179	20232	11.0
22	12470	1232.90	4.1079	18335	9.9
23	12498	1222.35	4.0727	19385	10.5
24	12521	1213.68	4.0439	19733	10.7
25	12548	1203.51	4.0100	10952	5.9
26	12572	1194.47	3.9798	6366	3.4
27	12581	1191.08	3.9685	6894	3.7
28	12596	1185.43	3.9497	5634	3.1
29	12603	1182.79	3.9409	6480	3.5

30	12630	1172.62	3.9070	10243	5.6
31	12640	1168.85	3.8945	12825	6.9
32	12646	1166.59	3.8869	20610	11.2
33	12660	1161.31	3.8694	25254	13.7
34	12689	1150.39	3.8330	19122	10.4
35	13036	1019.65	3.3974	37469	20.3
36	13041	1017.77	3.3911	37706	20.4
37	13052	1013.62	3.3773	37820	20.5

IFC54.4/CDCl3/AR20-102/AC 250



IFC67-2/CDCL3/FV24-101/AC 250



Peak List Date: 25.02.2004 Time: 14:20

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Peak Results saved in File	:	
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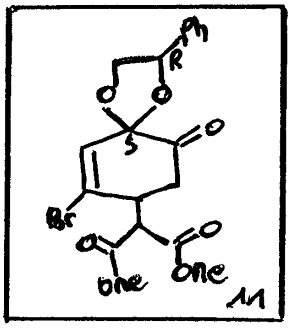
OFFSET	:	15.7748 ppm
SW_p	:	5000.00 Hz
SI	:	16384



Peak Picking Parameter

Peak constant PC	=	1.00
Noise	=	7
Sens. level	=	26

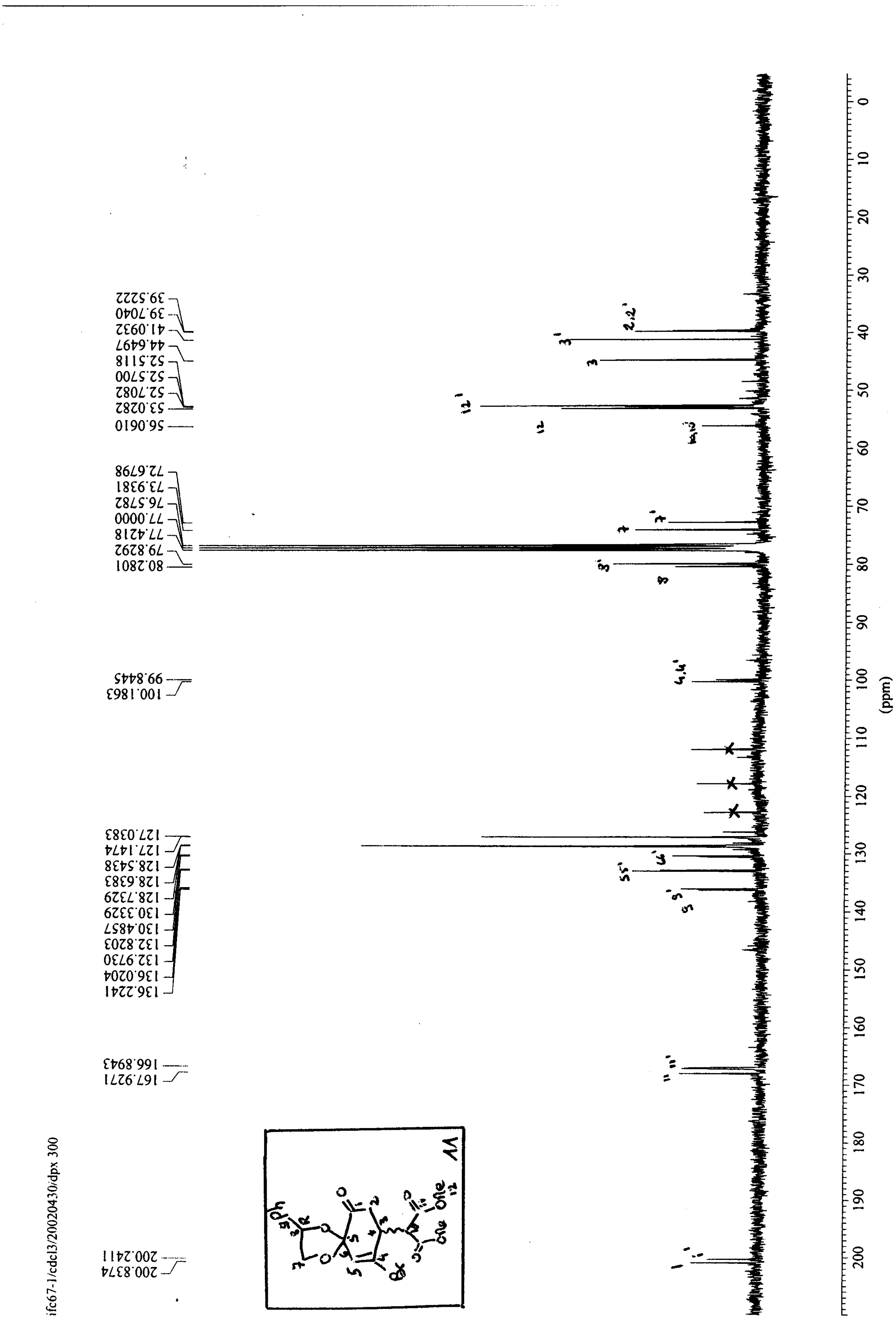
Peak Picking region

Start(ppm/Hz)	End(ppm/Hz)	MI(%)	MAXI(%)
7.37/ 1844.7	7.17/ 1794.0	80.35	109.98
7.44/ 1860.8	7.28/ 1821.8	35.06	57.07
7.61/ 1903.2	7.51/ 1877.9	1.83	8.18
6.40/ 1601.1	6.21/ 1553.8	3.95	20.24
5.21/ 1304.2	4.94/ 1235.8	3.81	10.14
5.38/ 1346.3	5.23/ 1307.2	22.21	26.04
4.51/ 1128.4	4.33/ 1084.2	3.91	9.47
4.39/ 1099.1	4.29/ 1074.1	1.32	3.24
3.96/ 989.9	3.67/ 918.4	58.94	85.77

Peak Picking results

Peak Nr.	Data Point	Frequency	PPM	Intensity	%Int.
1	6718	1895.62	7.5784	772	2.7
2	6725	1893.48	7.5699	839	2.9
3	6744	1887.68	7.5467	1390	4.8
4	6749	1886.15	7.5406	1339	4.6
5	6900	1840.07	7.3564	14379	49.6
6	6979	1815.96	7.2600	28999	100.0
7	7750	1580.67	6.3193	4719	16.3
8	7761	1577.32	6.3059	2023	7.0
9	8586	1325.55	5.2994	6653	22.9
10	8724	1283.43	5.1310	1490	5.1
11	8743	1277.63	5.1078	1760	6.1
12	8756	1273.67	5.0920	1760	6.1
13	8775	1267.87	5.0688	1618	5.6
14	9252	1122.30	4.4868	1615	5.6
15	9271	1116.50	4.4636	1615	5.6
16	9279	1114.06	4.4539	2157	7.4
17	9298	1108.26	4.4307	1716	5.9
18	9339	1095.75	4.3807	555	1.9
19	9358	1089.95	4.3575	575	2.0
20	9367	1087.20	4.3465	676	2.3
21	9385	1081.71	4.3245	597	2.1
22	9553	1030.44	4.1196	2562	8.8
23	9564	1027.09	4.1062	3523	12.1
24	9576	1023.42	4.0915	3841	13.2
25	9755	968.80	3.8731	7228	24.9
26	9813	951.10	3.8024	21306	73.5
27	9880	930.65	3.7206	19293	66.5

8	9957	907.15	3.6267	7480	25.8
9	10392	774.40	3.0960	3217	11.1
0	10409	769.21	3.0752	7389	25.5
1	10424	764.63	3.0569	3134	10.8
2	10431	762.50	3.0484	2966	10.2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128.6383
128.5438
127.1474
127.0383
100.1863
99.8445
80.2801
79.8292
77.4218
77.0000
76.5782
73.9381
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56.0610
53.0282
52.7082
52.5700
52.5118
44.6497
41.0932
39.7040
39.5222
(ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
2,2'
3'
3
12'
12
4,4'
11,11'
OMe
Ph