Supporting Information

Microwave-Accelerated Spiro-Cyclizations of *o*-Halobenzyl Cyclohexenyl Ethers by Palladium(0) Catalysis

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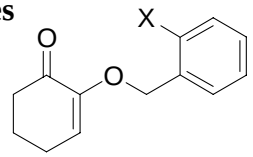
General Section

^{1}H and ^{13}C NMR spectra were obtained in $CDCl_3$ solution at 400 MHz and 100 MHz respectively. Overlapping signals in spectra were assigned as multiplets. Low resolution mass spectra were recorded on a GC-MS instrument equipped with a CP-SIL 8 CB capillary column (30 m × 0.25 mm) operating at an ionization potential of 70 eV. The oven temperature was generally 40-300 °C (gradient 30 °C/min). Melting points were determined on a capillary melting point apparatus and are uncorrected. All Heck reactions were performed in septa-sealed process vials. Silica gel 60 (0.040-0.063 mm, E. Merck, no. 9385) or aluminiumoxide was used for column chromatography. Microwave heating was performed by a microwave reactor of model Smith Synthesizer operating at a frequency of 2450 MHz. The temperature was measured via an internal IR-sensor. Specified reaction times refer to a total hold time at given temperature. All reagents obtained from commercial sources were used as received. Reported spectral data were in agreement with the proposed structures. Synthesized compounds lacking elemental analysis in the literature exhibited spectral and analytical properties as summarized below.

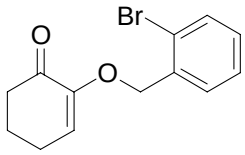
General Procedure for Preparation of 2-(2-Halobenzyloxy)-2-cyclohexenones (1 and 2).



The following chemicals were added to a dry 100 mL three-necked round-bottomed flask: either *o*-bromobenzyl alcohol (4.68 g, 25 mmol) or *o*-iodobenzyl alcohol (5.85 g, 25 mmol), 1,2-cyclohexandione (4.20 g, 37.5 mmol), *p*-toluenesulfonic acid monohydrate (0.28 g, 1.25 mmol) and benzene (50 mL). A soxlett device filled with K_2CO_3 (s) was connected to the flask with a condenser on the top. Upon heating, condensed vapors rinsed through the soxlett device, trapping the released water. The contents of the flask were magnetically stirred and heated using an oil-bath (120 °C) until no further conversion of the *o*-halobenzyl alcohol was detected. A second portion of 1,2-cyclohexandione (1.40 g, 12.5 mmol) was added and the reaction was continued until not more than traces of *o*-

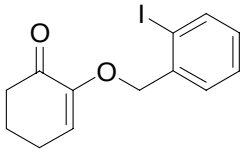
halobenzyl alcohol remained in the reaction mixture. The reaction mixture was cooled to 0 °C and 0.5 M Na_2CO_3 (100 mL) was added and the layers separated. The organic phase was washed with additional portions of 0.5 M Na_2CO_3 until no traces of 1,2-cyclohexandione was left. The organic phase was dried ($MgSO_4$) and concentrated under reduced pressure. The crude product was purified by silica chromatography (ether/toluene) to give the title products.

2-(2-Bromobenzyloxy)-cyclohex-2-enone (1)



The compound was prepared according to the General Procedure for Preparation of 2-(2-Halobenzyloxy)-2-cyclohexenones. White crystalline solid, 83% yield (5.83 g, 20.7 mmol, >95% by GC-MS), mp = 51 °C. Compound **1** was also produced in 52% yield with $CHCl_3$ as the solvent. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54-7.48 (m, 2H), 7.33-7.28 (m, 1H), 7.17-7.12 (m, 1H), 5.91 (t, *J* = 4.6 Hz, 1H), 4.90 (s, 2H), 2.53 (t, *J* = 6.7 Hz, 2H), 2.39 (td, *J* = 6.0, 4.6 Hz, 2H), 1.96 (tt, *J* = 6.7, 6.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.2, 150.3, 136.0, 132.6, 129.4, 128.9, 127.8, 122.1, 119.8, 69.0, 39.1, 24.7, 23.1. IR (KBr) 1692 cm^{-1}. MS m/z (relative intensity 70 eV) 253 (10), 251 (10), 202 (16), 201 (100), 173 (19), 171 (98), 170 (13), 169 (94), 90 (38), 89 (37), 63 (23), 55 (24). Anal. Calcd for $C_{13}H_{13}BrO_2$: C, 55.54; H, 4.66. Found: C, 55.40; H, 4.56.

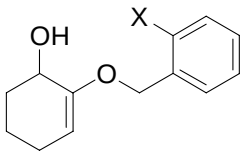
2-(2-Iodobenzyloxy)-cyclohex-2-enone (2)



The compound was prepared according to the General Procedure for Preparation of 2-(2-Halobenzyloxy)-2-cyclohexenones. White crystalline solid, 77% yield (6.32 g, 19.3 mmol, >95% by GC-MS), mp = 35 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (dd, *J* = 8.0, 1.3 Hz,1H), 7.37 (dd, *J* = 7.7, 1.3 Hz ,1H), 7.23 (ddd, *J* = 7.7, 7.3, 1.3 Hz, 1H), 6.88 (ddd, *J* = 8.0, 7.3, 1.7 Hz, 1H), 5.81 (t, *J* = 4.6 Hz, 1H), 4.68 (s, 2H), 2.41 (t, *J* = 6.8 Hz, 2H), 2.28 (td, *J* = 6.0, 4.6 Hz, 2H), 1.85 (tt, *J* = 6.8, 6.0 Hz,

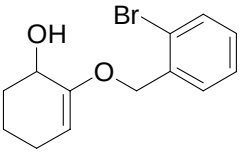
2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 150.0, 138.9, 138.5, 129.4, 128.365, 128.350, 119.6, 96.8, 73.3, 38.8, 24.5, 22.8. IR (KBr) 1692 cm^{-1}. MS m/z (relative intensity 70 eV) 328 (2), 311 (10), 299 (10), 218 (13), 217 (100), 202 (41), 201 (46), 173 (11), 90 (41), 89 (58), 63 (20), 55 (17). Anal. Calcd for $C_{13}H_{13}IO_2$: C, 47.58; H, 3.99. Found: C, 47.56; H, 3.99.

General Procedure for Preparation of 2-(2-Halobenzyloxy)-cyclohex-2-enol (3 and 4).



1 (7.03 g, 25 mmol) or **2** (8.20 g, 25 mmol) was dissolved in a mixture of THF (50 mL) and methanol (50 mL) in a 250 mL round bottomed flask and cooled to 0 °C. $NaBH_4$ (0.95 g, 25 mmol) was added in portions under continuous cooling and stirring. The mixture was allowed to stir for another 10 minutes, before 0.5M citric acid (50 mL) was added and finally the mixture was concentrated to <20 mL under reduced pressure. The remaining mixture was extracted with diethyl ether, and the combined etheral phases were dried ($MgSO_4$), concentrated and the residue was purified by chromatography (aluminium oxide with 6% (w/w) water added, ether/toluene eluent) to furnish the desired products.

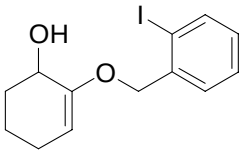
2-(2-Bromobenzyloxy)-cyclohex-2-enol (3)



The compound was prepared according to the General Procedure for Preparation of 2-(2-halobenzyloxy)-2-cyclohexenol. White solid, 92% yield (6.51 g, 23.0 mmol, >95% by GC-MS), mp = 54 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.46 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.32 (ddd, *J* = 7.7, 7.5, 1.3 Hz, 1H), 7.17 (ddd, *J* = 8.0, 7.5, 1.8 Hz, 1H), 4.86-4.84 (m, 1H), 4.81 (s, 2H), 4.26-4.24 (m, 1H), 2.42 (s, 1H), 2.20-2.10 (m, 1H), 2.08-1.99 (m, 1H), 1.92-1.77 (m, 2H), 1.76-1.66 (m, 1H), 1.61-1.52 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.6, 136.7, 133.0, 129.6, 129.5, 127.8, 123.1, 98.4, 68.7, 66.7, 31.3, 24.2, 19.0. IR (thin film) 3422, 3364 cm^{-1}. MS m/z (relative intensity 70 eV) 266

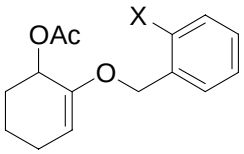
(12), 264 (12), 186 (11), 185 (18), 172 (11), 171 (100), 170 (12), 169 (93), 90 (30), 89 (34), 67 (11), 63 (14). Anal. Calcd for $C_{13}H_{15}BrO_2$: C, 55.14; H, 5.34. Found: C, 55.01; H, 5.21. Compound unstable in solution.

2-(2-Iodobenzyloxy)-cyclohex-2-enol (4)



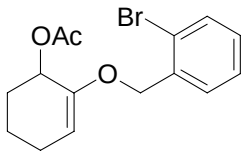
The compound was prepared according to the General Procedure for Preparation of 2-(2-halobenzyloxy)-2-cyclohexenol. White solid, 87% yield (7.20 g, 21.8 mmol, >95% by GC-MS), mp = 55 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.39 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.30 (ddd, *J* = 7.5, 7.3, 1.3 Hz, 1H), 6.96 (ddd, *J* = 7.9, 7.3, 1.8 Hz, 1H), 4.82-4.79 (m, 1H), 4.69-4.68 (m, 2H), 4.23-4.19 (m, 1H), 2.79 (s, 1H), 2.16-1.46 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.4, 139.34, 139.25, 129.5, 128.9, 128.4, 98.2, 97.8, 72.7, 66.3, 31.2, 24.1, 18.7. IR (KBr) 3422, 3378 cm^{-1}. MS m/z (relative intensity 70 eV) 312 (22), 217 (100), 186 (49), 185 (20), 90 (27), 89 (23). Anal. Calcd for $C_{13}H_{15}IO_2$: C, 47.29; H, 4.58. Found: C, 47.15; H, 4.51. Compound unstable in solution.

General Procedure for Preparation of 2-(2-Halobenzyloxy)-cyclohex-2-enyl acetate (5 and 6).



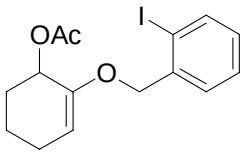
Compound **3** (5.66 g, 20 mmol) or **4** (7.44 g, 20 mmol) was added to a dry 50 mL round-bottomed flask. The compound was dissolved in acetic anhydride (20 mL) and *N*,*N*-diisopropylethylamine (4.1 mL, 25 mmol). Once a solution was formed, DMAP (122 mg, 1 mmol) was added under stirring. After 5 minutes, the solution was transferred to an extraction funnel with 100 mL 1M Na_2CO_3 and left for the acetic anhydride to dissolve. The aqueous fraction was extracted with diethyl ether, dried (Na_2SO_4), concentrated, and the residue purified by chromatography (aluminium oxide with 6% (w/w) water added, toluene/isohexane eluent) to obtain the title compounds.

2-(2-Bromobenzyloxy)-cyclohex-2-enyl acetate (5)



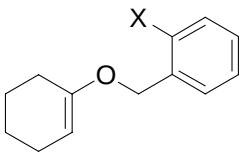
Colorless oil, 90% yield (5.85 g, 18.0 mmol, >95% by GC-MS). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.45 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.31 (ddd, *J* = 7.7, 7.3, 1.3 Hz, 1H), 7.14 (ddd, *J* = 8.0, 7.3, 1.8 Hz, 1H), 5.46-5.44 (m, 1H), 5.03-5.01 (m, 1H), 4.80-4.79 (m, 2H), 2.24-2.14 (m, 1H), 2.12-2.02 (m, 1H), 2.08 (s, 3H), 2.07-1.89 (m, 1H), 1.85-1.75 (m, 1H), 1.72-1.58 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 151.3, 136.8, 132.6, 129.1, 128.6, 127.7, 122.1, 101.4, 68.4, 68.3, 29.4, 23.7, 21.6, 18.3. IR (thin film) 1733 cm^{-1}. MS m/z (relative intensity 70 eV) 266 (24), 264 (24), 248 (17), 246 (16), 186 (92), 185 (100), 171 (61), 169 (59), 90 (27). Anal. Calcd for $C_{15}H_{17}BrO_3$: C, 55.40; H, 5.27. Found: C, 55.63; H, 5.23.

2-(2-Iodobenzyloxy)-cyclohex-2-enyl acetate (6)



Colorless oil, 89% yield (6.63 g, 17.8 mmol, >95% by GC-MS). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.40 (dd, *J* = 7.5, 1.9 Hz, 1H), 7.34 (ddd, *J* = 7.5, 7.3, 1.3 Hz, 1H), 6.97 (ddd, *J* = 7.9, 7.3, 1.9 Hz, 1H), 5.45-5.44 (m, 1H), 5.01-4.99 (m, 1H), 4.70 (s, 2H), 2.25-2.14 (m, 1H), 2.10 (s, 3H), 2.13-2.00 (m, 1H), 1.98-1.90 (m, 1H), 1.85-1.75 (m, 1H), 1.71-1.58 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 151.3, 139.5, 139.2, 129.4, 128.5, 128.4, 101.5, 97.0, 72.9, 68.3, 29.4, 23.8, 21.7, 18.4. IR (thin film) 1735 cm^{-1}. MS m/z (relative intensity 70 eV) 373 (3), 312 (29), 294 (38), 217 (100), 187 (17), 186 (92), 185 (73), 113 (12), 90 (31), 89 (28), 63 (10). Anal. Calcd for $C_{15}H_{17}IO_3$: C, 48.40; H, 4.60. Found: C, 48.24; H, 4.57.

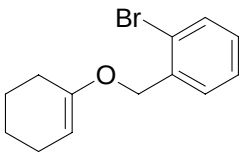
General Procedure for Preparation of 1-Halo-2-(cyclohex-1-enyloximetyl)-



benzene (7 and 8).

The following chemicals were added to a dry 100 mL round-bottomed flask: *o*-bromobenzyl alcohol (4.68 g, 25 mmol) or *o*-iodobenzylalcohol (5.85 g, 25 mmol), 1-methoxy cyclohexen (4.6 mL, 37.5 mmol), benzene (50 mL). The mixture was cooled on ice until a slurry was formed. *p*-Toluenesulfonic acid monohydrate (48 mg, 0.25 mmol) was added and the reaction was cooled and stirred for 1 h. An oven dried distillation device connected through a vigreux column was mounted on top of the flask and the solvent was distilled of from the flask and successively refilled with fresh benzene. Once the vapor temperature reached 80 °C, additional *p*-toluenesulfonic acid monohydrate (48 mg, 0.25 mmol) was added to facilitate the methanol elimination ensuring no traces of the methyl/*o*-halobensyl ketal intermediate was to be detected. A maximum of 3 x 48 mg of *p*-toluenesulfonic acid monohydrate was added. The reaction mixture was washed with 2M NaOH and the organic phase dried (K_2CO_3), concentrated at reduced pressure, and the residue purified by chromatography (aluminium oxide with 6% water added, ether/isohexane eluent). If the product containing fractions were contaminated with *o*-halobenzyl ketals, the product mixture was dissolved in dimethylacetamide (10 mL/g substance) and microwave heated to 220 °C for 5 minutes in a sealed vessel, cooled, diluted with water, extracted with diethyl ether, dried (K_2CO_3), concentrated, and the residue purified by chromatography (aluminium oxide with 6% water added, ether/isohexane mobile phase) to give **7** or **8**.

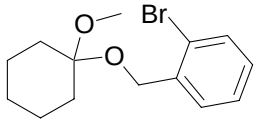
1-Bromo-2-(cyclohex-1-enyloximetyl)-benzene (7)



The compound was prepared according to the General Procedure for Preparation of (2-Halobenzyloxy)-1-cyclohexen. Colorless liquid, 85% yield (5.68 g, 21.3 mmol, >95% by GC-MS). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.50 (dd, *J* = 7.9, 1.8 Hz, 1H), 7.33 (ddd, *J* = 7.9, 7.2, 1.3 Hz, 1H), 7.15 (ddd, *J* = 8.0, 7.2, 1.8 Hz, 1H), 4.81 (s, 2H), 4.76-4.75 (m, 1H), 2.22-2.17 (m, 2H), 2.12-2.07 (m, 2H), 1.77-1.70 (m, 2H), 1.62-1.56 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.5, 137.3, 132.7,

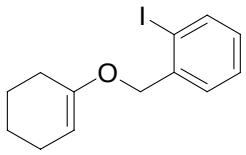
129.08, 129.07, 127.6, 122.6, 95.3, 68.0, 28.0, 23.8, 23.2, 23.0. IR (thin film, ATR diamond crystal) 1668 cm^{-1} MS m/z (relative intensity 70 eV) 268 (42), 266 (42), 187 (31), 171 (100), 170 (28), 169 (84), 119 (11), 90 (37), 89 (32), 63 (15). Anal. Calcd for $C_{13}H_{15}BrO$: C, 58.44; H, 5.66. Found: C, 58.53; H, 5.62.

1-Bromo-2-(1-methoxy-cyclohexyloxymethyl)-benzene



Intermediate isolated from the synthesis of **7**; see Scheme 2. Colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (dd, *J* = 7.5, 1.8 Hz, 1H), 7.53 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.32 (td, *J* = 7.5, 1.3 Hz, 1H), 7.13 (ddd, *J* = 7.9, 7.5, 1.8 Hz, 1H), 4.53 (s, 2H), 3.22 (s, 3H), 1.85-1.68 (m, 4H), 1.63-1.50 (m, 4H), 1.48-1.39 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.8, 132.6, 128.9, 128.6, 127.6, 122.7, 100.9, 61.6, 48.0, 33.5, 25.8, 23.2. MS m/z (relative intensity 70 eV) 267 (10), 220 (19), 219 (100), 171 (36), 169 (39), 113 (47), 90 (19), 81 (17), 69 (13). Anal. Calcd for $C_{14}H_{19}BrO_2$: C, 56.20; H, 6.40. Found: C, 56.30; H, 6.33.

1-Iodo-2-(cyclohex-1-enyloximetyl)-benzene (8)



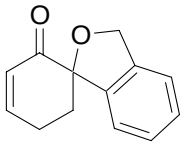
The compound was prepared according to the General Procedure for Preparation of (2-Halobenzyloxy)-1-cyclohexen. Colorless liquid, 77% yield (6.05 g, 19.3 mmol, >95% by GC-MS). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.37 (dd, *J* = 7.7, 1.9 Hz, 1H), 7.28 (ddd, *J* = 7.7, 7.3, 1.3 Hz, 1H), 6.91 (ddd, *J* = 7.9, 7.3, 1.9 Hz, 1H), 4.66 (tt, *J* = 3.9, 1.3 Hz, 1H), 4.63 (s, 2H), 2.09 (tdd, *J* = 6.4, 3.3, 2.1 Hz, 2H), 2.04-1.98 (m, 2H), 1.68-1.61 (m, 2H), 1.54-1.46 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.6, 140.3, 139.4, 129.5, 128.9, 128.6, 97.7, 95.5, 72.7, 28.1, 23.9, 23.3, 23.1. IR (thin film) 1666 cm^{-1}. MS m/z (relative intensity 70 eV) 314 (94), 296 (17), 218 (13), 217 (100), 188 (15), 187 (57), 186 (12), 119 (13), 90 (25), 89 (30), 63 (11). Anal. Calcd for $C_{13}H_{15}IO$: C, 49.70; H, 4.81. Found:

C, 49.95; H, 4.84.

General Procedure for Spiro Cyclizations (Table 1).

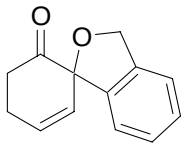
The following chemicals were added to a thick-walled tube: $Pd(OAc)_2$, either as a 0.1 M stock solution (50 μL of solution containing 22.5 mg/mL $Pd(OAc)_2$ in acetonitrile, 0.005 mmol) or as solid (11.2 mg, 0.05 mmol); tetrabutylammonium bromide (322 mg, 1 mmol) or tetrabutylammonium hydrogen sulfate (340 mg, 1 mmol); either of the substrates: **1** (141 mg, 0.5 mmol), **2** (164 mg, 0.5 mmol), **3** (142 mg, 0.5 mmol), **4** (165 mg, 0.5 mmol), **5** (163 mg, 0.5 mmol), **6** (186 mg, 0.5 mmol), **7** (134 mg, 0.5 mmol) or **8** (157 mg, 0.5 mmol); in entry 5 only, Ag_3PO_4 (84 mg, 0.2 mmol); 3 mL of solvent and PMP (0.362 mL, 2 mmol). The reaction mixture was septum sealed in air and magnetically stirred and heated according to specifications. The reaction mixture was cooled, diluted (diethyl ether) and washed with water, dried (K_2CO_3), concentrated under vacuum and the residue was purified by chromatography (silica, ether/isohexane mobile phase) to yield the products **9**-**12** characterized below.

3'*H*-Spiro[cyclohex-3-ene-1,1'-isobenzofuran]-2-one (9a)



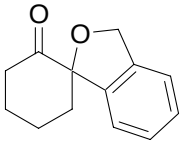
Colorless oil, 91% yield (91.1 mg, 0.455 mmol, Table 1, entry 3, >95% by GC-MS). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.21 (m, 1H), 7.20-7.15 (m, 2H), 7.11-7.08 (m, 1H), 7.01 (dtd, *J* = 10.1, 4.0, 0.7 Hz, 1H), 6.07 (dt, *J* = 10.1, 2.0 Hz, 1H), 5.19 (d, *J* = 12.2 Hz, 1H), 5.09 (d, *J* = 12.2 Hz, 1H), 2.58 (dddd, *J* = 6.7, 5.5, 4.0, 2.0 Hz, 2H), 2.31 (ddd, *J* = 13.6, 5.5, 0.7 Hz, 1H), 2.25 (dtd, *J* = 13.6, 6.7, 5.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.2, 150.7, 140.1, 139.6, 129.0, 128.7, 127.6, 121.9, 121.7, 89.5, 73.2, 34.4, 24.5. IR (thin film) 1682 cm^{-1}. MS m/z (relative intensity 70 eV) 200 (67), 182 (11), 133 (23), 132 (100), 131 (41), 115 (13), 104 (49), 103 (19), 90 (12), 89 (22), 78 (17), 77 (11), 63 (13), 50 (10). Anal. Calcd for $C_{13}H_{12}O_2$: C, 77.98; H, 6.04. Found: C, 77.75; H, 5.94.

3'*H*-Spiro[cyclohex-5-ene-1,1'-isobenzofuran]-2-one (9b)



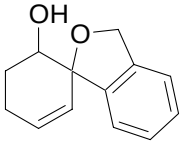
Colorless oil, 26% yield (26.0 mg, 0.13 mmol, Table 1, entry 5, >95% by GC-MS). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.20 (m, 4H), 6.17-6.12 (m, 1H), 5.79-5.75 (m, 1H), 5.35 (d, *J* = 12.0 Hz, 1H), 5.19 (d, *J* = 12.0 Hz, 1H), 2.95-2.85 (m, 1H), 2.77-2.62 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.7, 140.22, 140.20, 131.3, 130.9, 128.8, 127.7, 122.4, 121.7, 89.8, 73.7, 36.6, 27.1. IR (thin film) 1725 cm^{-1}. MS m/z (relative intensity 70 eV) 201 (31), 173 (17), 172 (100), 171 (29), 158 (46), 157 (45), 131 (26), 129 (26), 128 (26), 115 (26), 89 (22). Anal. Calcd for $C_{13}H_{12}O_2$: C, 77.98; H, 6.04. Found: C, 77.77; H, 6.19.

3'*H*-Spiro[cyclohexane-1,1'-isobenzofuran]-2-one (10a)



White crystalline solid, 59% yield (59.6 mg, 0.295 mmol, Table 1, entry 7, >95% by GC-MS), mp = 92 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.30 (m, 3H), 7.23-7.19 (m, 1H), 5.27 (d, *J* = 12.1 Hz, 1H), 5.17 (d, *J* = 12.1 Hz, 1H), 2.93-2.84 (m, 1H), 2.57-2.49 (m, 1H), 2.20-2.01 (m, 4H), 1.96-1.84 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.9, 140.0, 139.8, 128.7, 127.6, 123.3, 121.4, 92.8, 72.7, 40.0, 39.7, 27.6, 21.9. IR (KBr) 1712 cm^{-1}. MS m/z (relative intensity 70 eV) 202 (10), 174 (36), 146 (13), 145 (100), 131 (10), 117 (19), 89 (10). Anal. Calcd for $C_{13}H_{14}O_2$: C, 77.20; H, 6.98. Found: C, 76.96; H, 6.94.

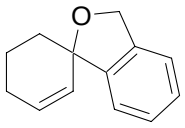
3'*H*-Spiro[cyclohexen-5-ene-1,1'-isobenzofuran]-2-ol (10b + 10c,



diastereomeric mixture, 1:3)

Colorless oil, 15% yield (15.2 mg, 0.075 mmol, Table 1, entry 7, >95% by GC-MS). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.12 (m, 4H), 5.68-5.64 (m, 2H), 5.12-5.02 (m, 2H), 3.97-3.91 (m, 0.75H), 3.93-3.87 (m, 0.25H), 2.65-2.15 (m, 4H), 1.97-1.95 (m, 0.25H), 1.92-1.90 (m, 0.75H). ^{13}C NMR (100 MHz, $CDCl_3$) Major product: δ 141.9, 140.3, 128.2, 127.7, 125.4, 124.8, 122.9, 121.0, 88.6, 72.8, 72.2, 36.9, 32.5, minor product: δ 142.4, 140.1, 128.3, 128.0, 124.9, 124.6, 121.7, 121.2, 88.9, 71.9, 70.4, 36.2, 31.4. IR (thin film) 3027 cm^{-1}. MS m/z (relative intensity 70 eV) 202 (81), 201 (17), 186 (10), 185 (63), 171 (100), 148 (34), 143 (11), 128 (20), 119 (17), 89 (15). Anal. Calcd for $C_{13}H_{14}O_2$: C, 77.20; H, 6.98. Found: C, 77.33; H, 7.07.

3'*H*-Spiro[cyclohexen-2-ene-1,1'-isobenzofuran] (12)

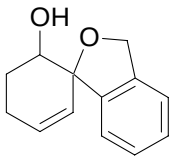


Colorless oil, 77% yield (71.7 mg, 0.385 mmol, Table 1, entry 14, >95% by GC-MS). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20-7.17 (m, 2H), 7.16-7.12 (m, 1H), 7.09-7.06 (m, 1H), 5.82-5.76 (m, 1H), 5.71-5.66 (m, 1H), 5.04-5.02 (m, 2H), 2.40-2.29 (m, 3H), 2.18-2.07 (m, 1H), 1.84-1.77 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.4, 139.4, 127.79, 127.64, 127.40, 125.0, 121.41, 121.31, 85.6, 71.1, 37.5, 32.8, 23.6. IR (thin film) 2844 cm^{-1}. MS m/z (relative intensity 70 eV) 186 (39), 133 (18), 132 (100), 131 (28), 104 (32), 103 (15), 89 (10), 78 (11). Anal. Calcd for $C_{13}H_{14}O$: C, 83.83; H, 7.58. Found: C, 83.66; H, 7.44.

Alternative Synthesis of 10 b, d, e and 11a-d.

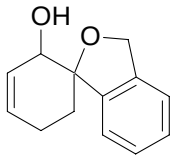
Due to the difficulties to separate the four isomers derived from **6** (**11a-d**) and to obtain unambiguous identification and characterization, the compounds were also prepared by means of other procedures. Conjugated ketone **9a** was reduced by $CeCl_3/NaBH_4$ to give a mixture of **10d** and **10e** that could be isolated and described separately. Further on, the **10d-e** mixture was acetylated using Ac_2O/DMAP to yield the expected products **11c** and **11d**. Also, the **10b-c** mixture was acetylated (Ac_2O/DMAP) to produce **11a** and **11b**. Since the **10b-c** mixture yielded by Heck cyclization of **3** and **4** (entries 6-8) could not be separated by chromatography, **10b** was also produced from **11b** through hydrolysis (2:1 dioxane/2M aq. HCl, 50 °C, 48 h) in order to ascertain a correct structural determination. The *Z*-configuration of **10e** and **11d** was assigned by ^{1}H NMR NOESY experiments, in which a positive NOE was observed between the hydrogen geminal to the hydroxi or acetate moiety, and a proton of the proximal aromatic system.

3'*H*-Spiro[cyclohexen-5-ene-1,1'-isobenzofuran]-2-ol, major isomer (10b)



11b (1.86 mmol, 455 mg, prepared as described below) was dissolved in dioxane (2 mL) and 2M (aq.) HCl (1 mL) and heated at 50 °C over night. Full conversion of **11b** was verified by GC-MS. The reaction mixture was diluted with water and extracted with diethyl ether. The etheral phase was dried (Na_2SO_4), rinsed through a silica plug, and the solvent evaporated to give a yield of 95% (364 mg, >95% by GC-MC) of pure **10b**. Minor product starting from **6**, major product starting from **4** via **10b-c**. Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.12 (m, 4H), 5.68-5.66 (m, 2H), 5.12 (d, *J* = 12.2 Hz, 1H), 5.05 (d, *J* = 12.2 Hz, 1H), 3.98-3.93 (m, 1H), 2.67-2.59 (m, 1H), 2.52-2.38 (m, 2H), 2.26-2.17 (m, 1H), 1.85 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.0, 140.4, 128.4, 127.8, 125.5, 124.9, 123.0, 121.1, 88.7, 72.9, 72.3, 37.0, 32.6. MS m/z (relative intensity 70 eV) 202 (66), 185 (17), 171 (100), 148 (35), 143 (19), 129 (13), 128 (21), 119 (21), 91 (16), 89 (17). IR (thin film, ATR diamond crystal) 3400 cm^{-1}.

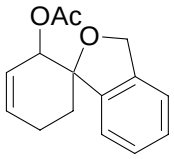
Anal. Calcd for $C_{13}H_{14}O_2$: C, 77.20; H, 6.98. Found: C, 76.94; H, 6.94.



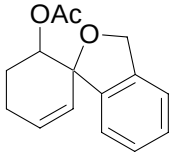
3'*H*-Spiro[cyclohexen-3-ene-1,1'-isobenzofuran]-2-ol (10d, 10e)

9a (4.40 mmol, 0.88 g) was dissolved in dioxane (20 mL) and methanol (20 mL), $CeCl_3 \cdot 7\ H_2O$ was added (2.20 mmol, 0.82 g) and the resulting mixture was cooled to –18 °C (brine ice). $NaBH_4$ (2.20 mmol, 83 mg) was added in portions (voluminous foam raising from surface) under continuous cooling and stirring. The reaction mixture was diluted with water, extracted with diethyl ether, the etheral phase was separated, dried (Na_2SO_4), concentrated and the residue purified by chromatography (diethyl ether/isohexane) to give a yield of 61% **10d** (542 mg, >95% by GC-MS) and 13% **10e** (116 mg, >95% by GC-MS) respectively. **10d** (*E*) - major product, second GC-MS peak, second eluted on chromatography, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.15 (m, 4H), 5.92-5.87 (m, 1H), 5.79-5.74 (m, 1H), 5.06-5.04 (m, 2H), 4.23-4.19 (m, 1H), 2.35-2.24 (m, 1H), 2.24-2.14 (m, 1H), 2.04-1.97 (m, 1H), 1.88-1.81 (m, 1H), 1.65 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.5, 140.8, 130.5, 128.7, 128.4, 127.6, 123.6, 121.5, 89.1, 71.7, 71.0, 30.5, 24.1. IR (thin film, ATR diamond crystal) 3400 cm^{-1}. MS m/z (relative intensity 70 eV) 202 (5), 133 (75), 132 (100), 131 (33), 104 (42), 103 (21), 78 (16). Anal. Calcd for $C_{13}H_{14}O_2$: C, 77.20; H, 6.98. Found: C, 77.21; H, 7.02. **10e** (*Z*) - minor product, first GC-MS peak, first eluted on chromatography, colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.10 (m, 4H), 5.66-5.63 (m, 2H), 5.09 (d, *J* = 12.1 Hz, 1H), 5.01 (d, *J* = 12.1 Hz, 1H), 3.92-3.89 (m, 1H), 2.63-2.55 (m, 1H), 2.50-2.40 (m, 2H), 2.22-2.14 (m, 1H), 2.07-2.06 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.0, 140.4, 128.2, 127.7, 125.4, 124.9, 122.9, 121.0, 88.7, 72.8, 72.2, 36.9, 32.5. IR (thin film, ATR diamond crystal) 3430 cm^{-1}. MS m/z (relative intensity 70 eV) 202 (3), 133 (61), 132 (100), 131 (30), 104 (42), 103 (20), 78 (18). Anal. Calcd for $C_{13}H_{14}O_2$: C, 77.20; H, 6.98. Found: C, 77.38; H, 6.93.

3'*H*-Spiro[cyclohexen-3-ene-1,1'-isobenzofuran]-2-yl acetate (11c, 11d)



A **10d**/**10e** 4:1 mixture (370 mg, 1.84 mmol, achieved through reduction of **9a**) together with DMAP (22 mg, 0.18 mmol) was dissolved in Ac_2O (3 mL) and stirred for 1 h. Saturated aq. $NaHCO_3$ was added to the reaction mixture with stirring until the Ac_2O layer had disappeared, followed by extraction with diethyl ether. The etheral phases was dried ($MgSO_4$), the solvent evaporated at reduced pressure, and the products purified by chromatography (diethyl ether/isohexane) to give a yield of 77% **11c** (346 mg, >95% by GC-MS) and 19% **11d** (85 mg, >95% by GC-MS), respectively. **11c** (*E*) - major product from this route, second GC-MS peak, first eluted on chromatography, white powder. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.12 (m, 4H), 6.07-6.02 (m, 1H), 5.80-5.74 (m, 1H), 5.14-5.10 (m, 1H), 5.06 (d, *J* = 12.4 Hz, 1H), 5.00 (d, *J* = 12.4 Hz, 1H), 2.44-2.33 (m, 1H), 2.24-2.08 (m, 2H), 1.89-1.88 (m, 3H), 1.86-1.79 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 142.4, 140.0, 133.0, 128.3, 127.2, 124.3, 123.3, 121.3, 86.3, 71.9, 70.0, 29.9, 23.2, 21.4. IR (thin film) 1733 cm^{-1}. MS m/z (relative intensity 70 eV) 244 (0, M^+), 185 (14), 184 (57), 133 (20), 132 (100), 131 (23), 104 (30), 103 (11). Anal. Calcd for $C_{15}H_{16}O_3$: C, 73.75; H, 6.60. Found: C, 73.92; H, 6.66. **11b** (*Z*) - minor product from this route, first GC-MS peak, second eluted on chromatography, white powder. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.20 (m, 1H), 7.20-7.16 (m, 1H), 7.15-7.12 (m, 1H), 7.10-7.07 (m, 1H), 6.02-6.01 (m, 1H), 5.61-5.56 (m, 1H), 5.50-5.48 (m, 1H), 5.13 (d, *J* = 12.1 Hz, 1H), 5.05 (d, *J* = 12.1 Hz, 1H), 2.50-2.39 (m, 2H), 2.28-2.13 (m, 2H), 2.04-1.91 (m, 1H), 1.80-1.79 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 142.0, 140.2, 132.0, 128.4, 127.8, 125.4, 121.8, 121.1, 86.6, 73.8, 73.0, 32.1, 23.4, 21.3. IR (thin film, ATR diamond crystal) 1737 cm^{-1}. MS m/z (relative intensity 70 eV) 244 (0, M^+), 185 (28), 184 (71), 133 (31), 132 (100), 131 (27), 104 (29), 103 (12). Anal. Calcd for $C_{15}H_{16}O_3$: C, 73.75; H, 6.60. Found: C, 73.95; H, 6.79.



3'*H*-Spiro[cyclohexen-5-ene-1,1'-isobenzofuran]-2-yl acetate (11a, 11b)

A **10b**/**10c** 3:1 mixture (260 mg, 1.29 mmol, achieved through the Heck arylation of **4**) together with DMAP (0.13 mmol, 16 mg) was dissolved in Ac_2O (3 mL) and stirred for 1 h. The reaction mixture was mixed with an excess of saturated aq. $NaHCO_3$ and stirred until the Ac_2O layer had disappeared, followed by extraction with diethyl ether. The etheral phases was dried ($MgSO_4$), the solvent evaporated at reduced pressure, and the products purified by chromatography (diethyl ether/isohexane) to give a yield of 72% **11b** (227 mg, >95% by GC-MS) and 20% **11a** (63 mg, >95% by GC-MS), respectively. **11b** - Major product through this route, minor product starting from **6**, second GC-MS peak, second eluted on chromatography, white powder. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.13 (m, 4H), 5.75-5.69 (m, 1H), 5.68-5.63 (m, 1H), 5.05-5.03 (m, 2H), 5.02-4.99 (m, 1H), 2.70-2.54 (m, 2H), 2.44-2.36 (m, 1H), 2.27-2.19 (m, 1H), 1.95-1.94 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 142.3, 140.1, 128.3, 127.6, 124.9, 124.0, 122.4, 121.1, 85.6, 73.1, 72.6, 36.2, 29.6, 21.5. IR (thin film, ATR diamond crystal) 1743 cm^{-1}. MS m/z (relative intensity 70 eV) 245 (5), 190 (11), 185 (31), 184 (100), 183 (16), 169 (18), 148 (38), 128 (13), 119 (14). Anal. Calcd for $C_{15}H_{16}O_3$: C, 73.75; H, 6.60. Found: C, 73.79; H, 6.59. **11a** - Minor product through this route, major product starting from **6**, first GC-MS peak, second eluted on chromatography, white powder. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.08 (m, 4H), 5.72-5.65 (m, 2H), 5.25-5.21 (m, 1H), 5.11 (d, *J* = 12.1 Hz, 1H), 5.04 (d, *J* = 12.1 Hz, 1H), 2.56-2.40 (m, 4H), 1.68 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 141.7, 140.6, 128.4, 127.8, 124.9, 124.8, 121.9, 121.0, 87.4, 72.9, 72.6, 37.8, 29.2, 21.1. IR (thin film, ATR diamond crystal) 1745 cm^{-1}. MS m/z (relative intensity 70 eV) 245 (29), 190 (10), 185 (43), 184 (100), 183 (19), 148 (30), 119 (13), 89 (12). Anal. Calcd for $C_{15}H_{16}O_3$: C, 73.75; H, 6.60. Found: C, 73.62; H, 6.42.

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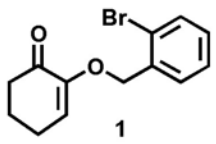

1

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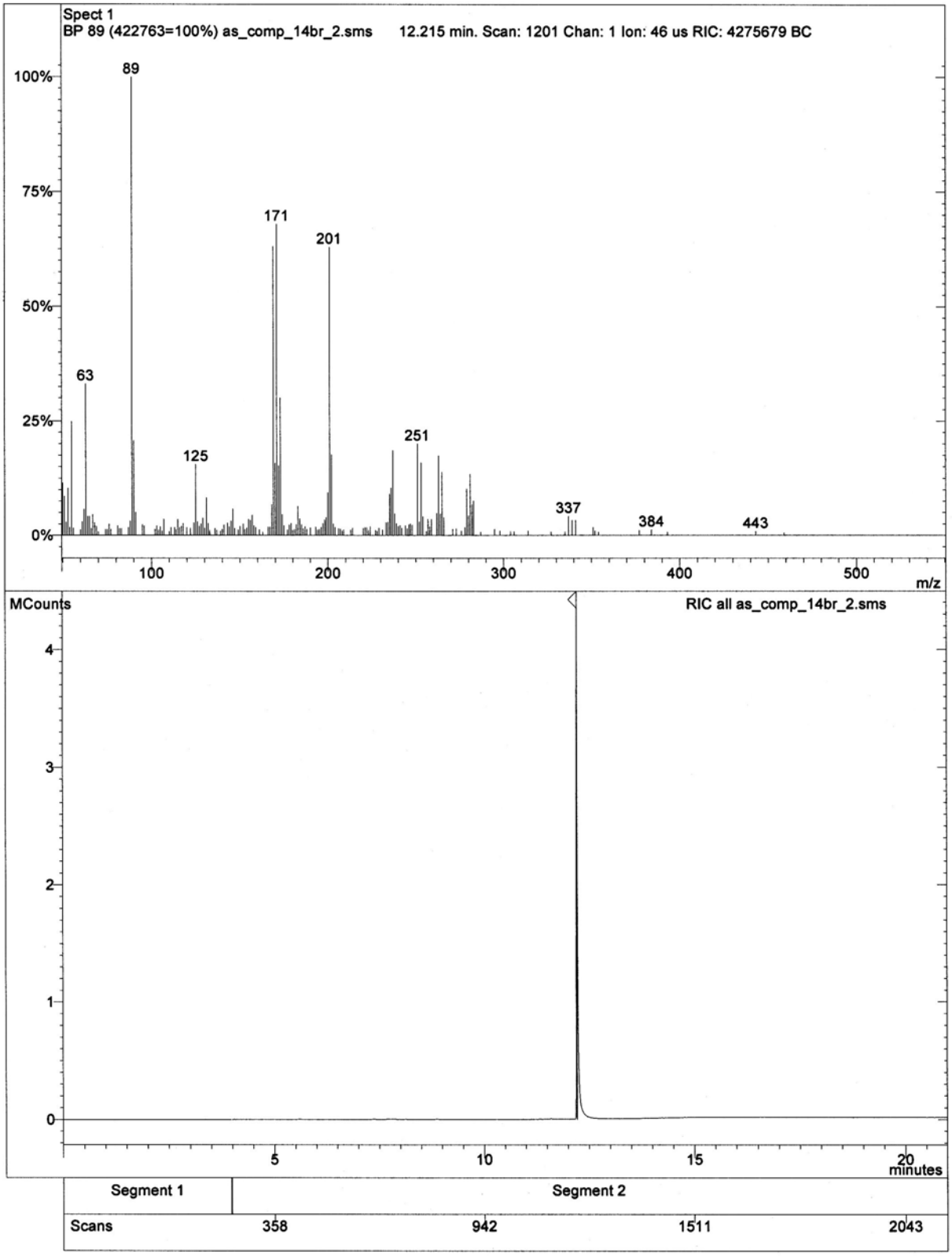

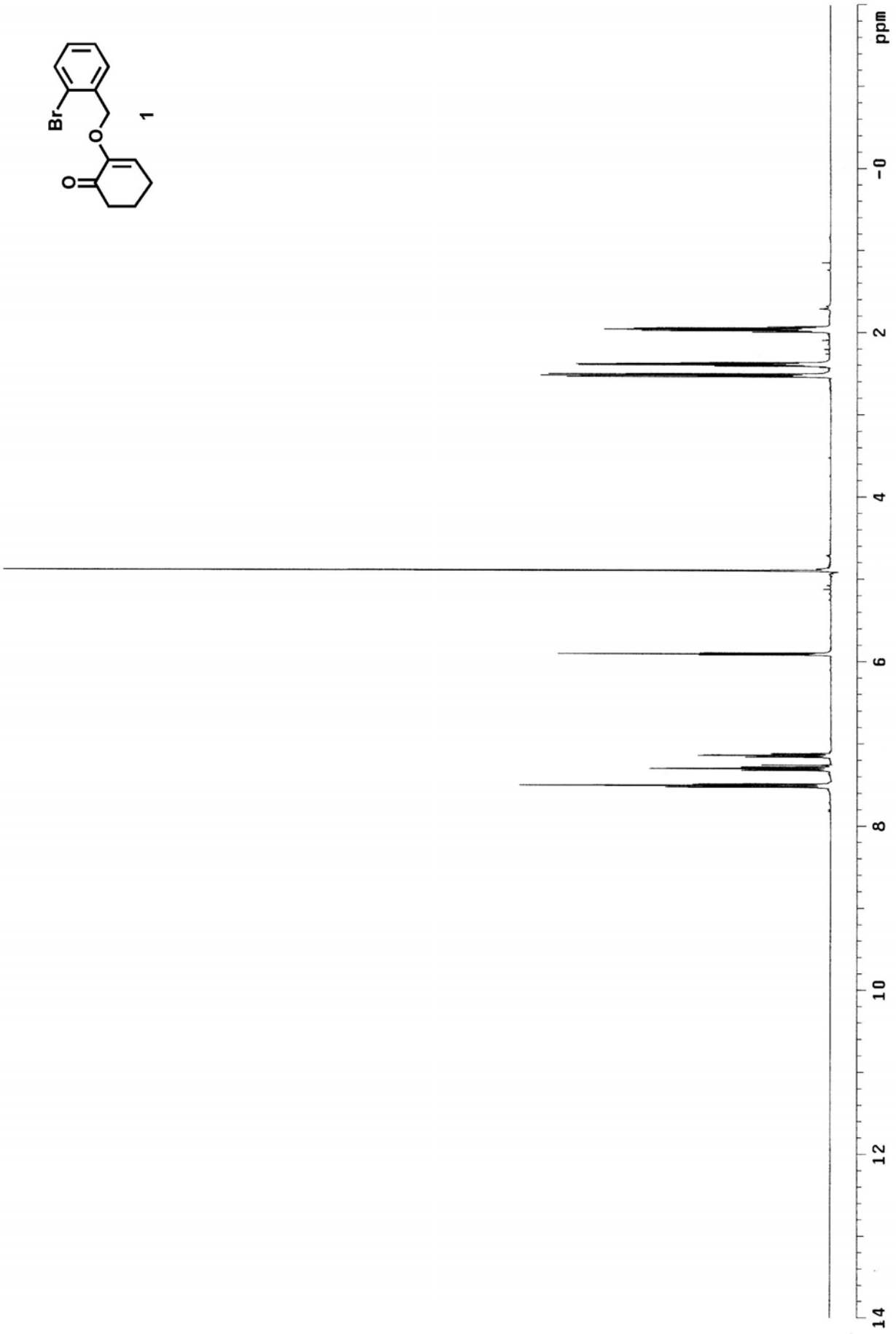
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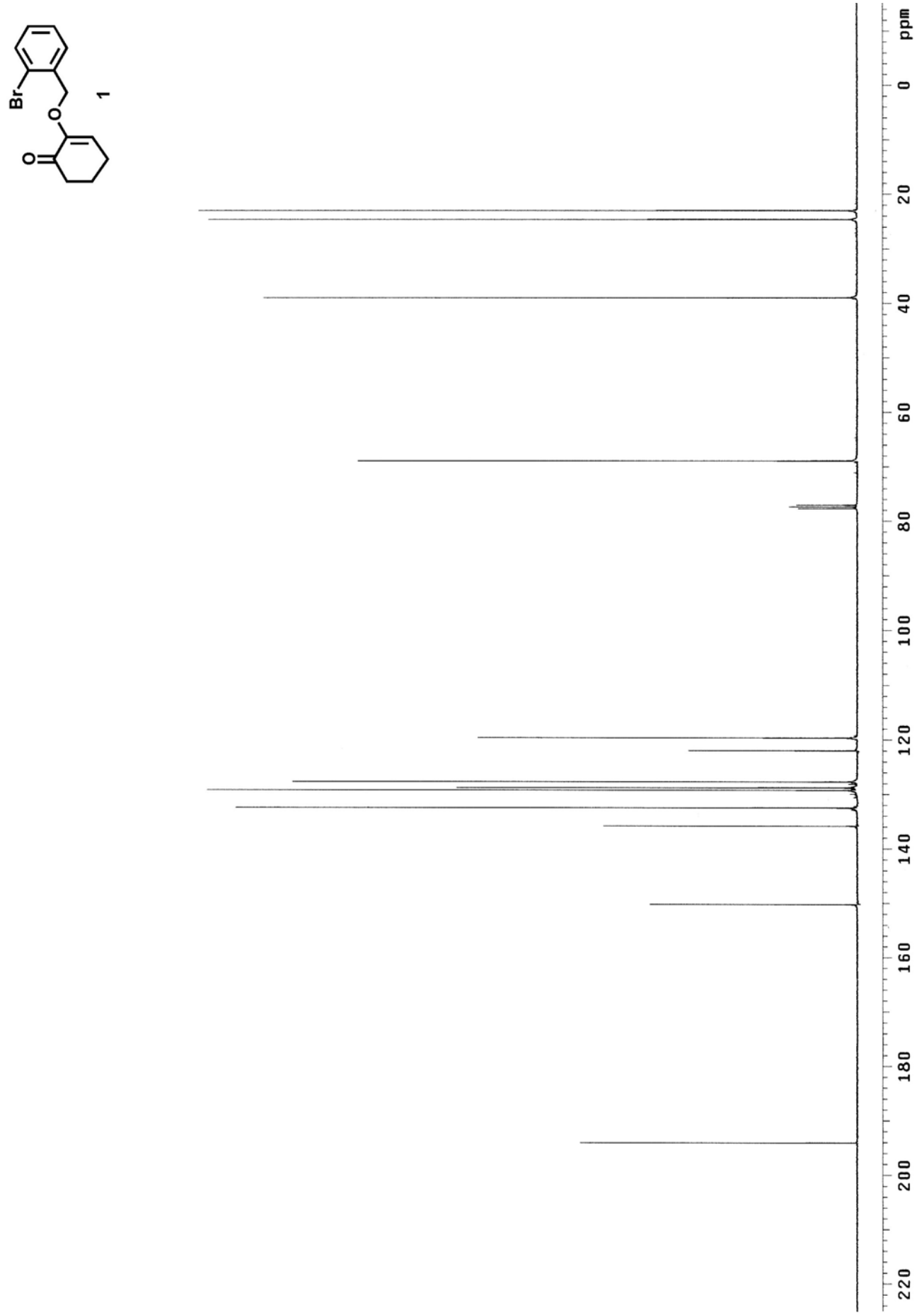
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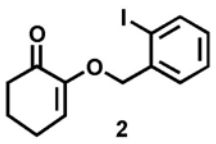

2

Chromatogram Plot

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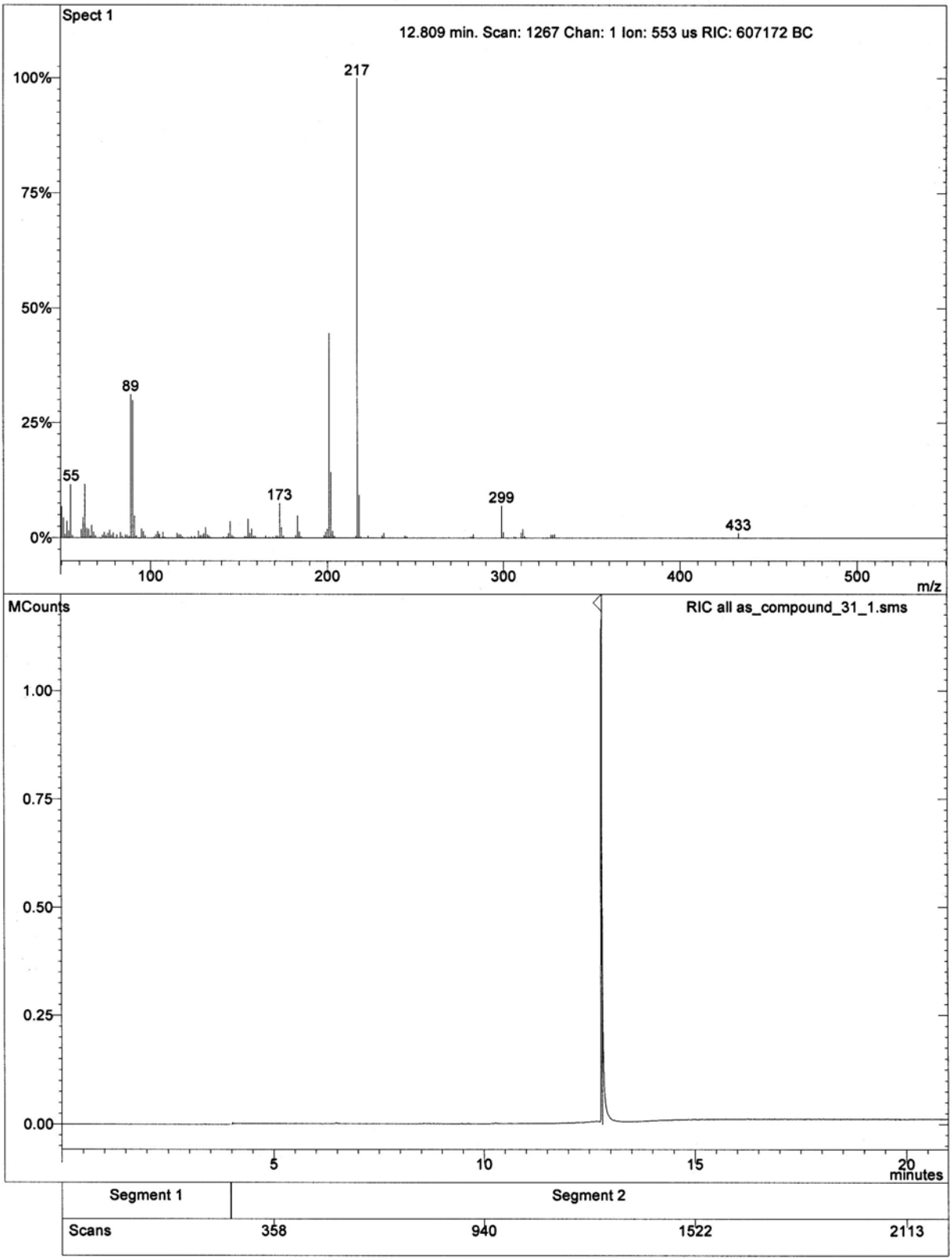

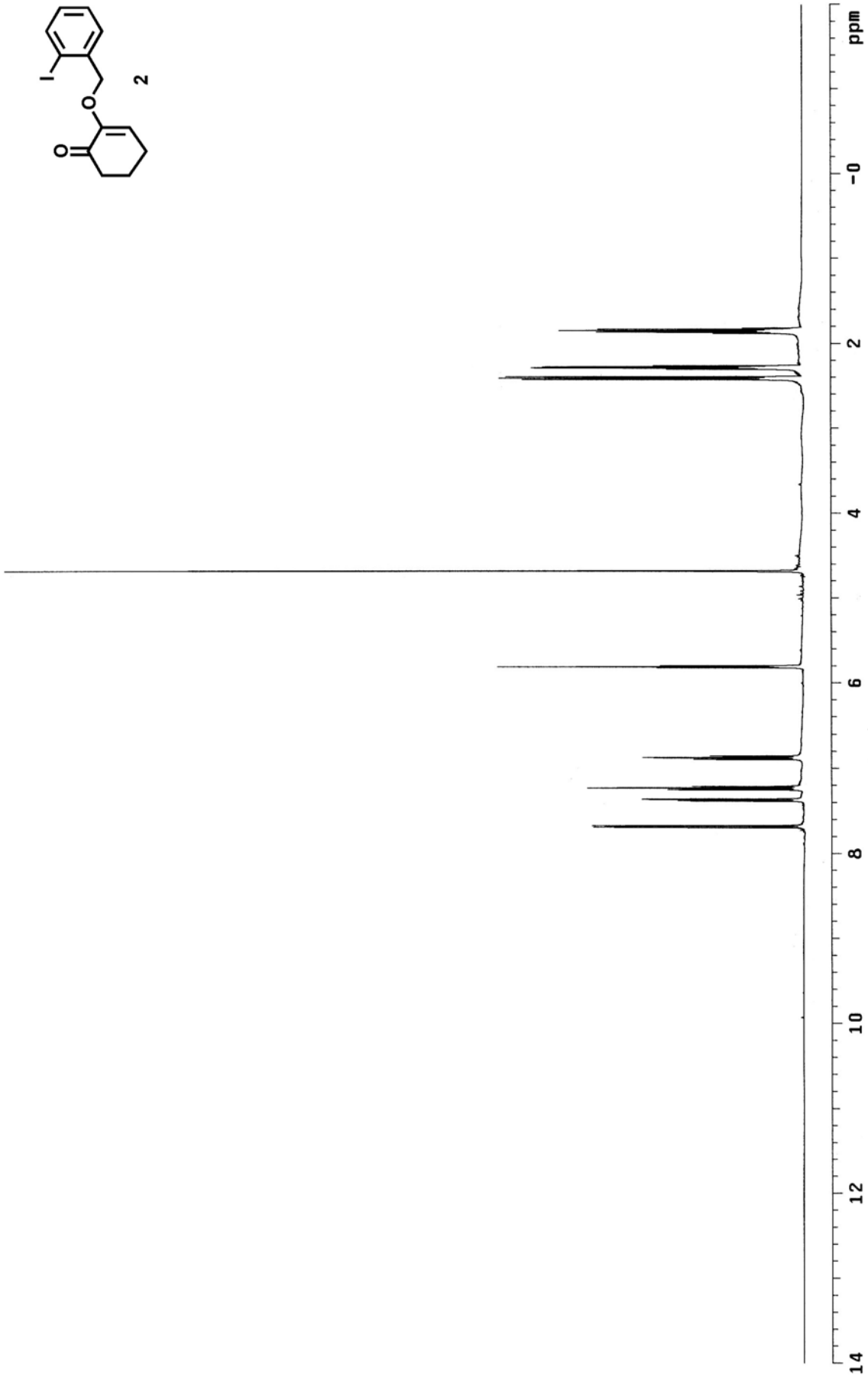
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2
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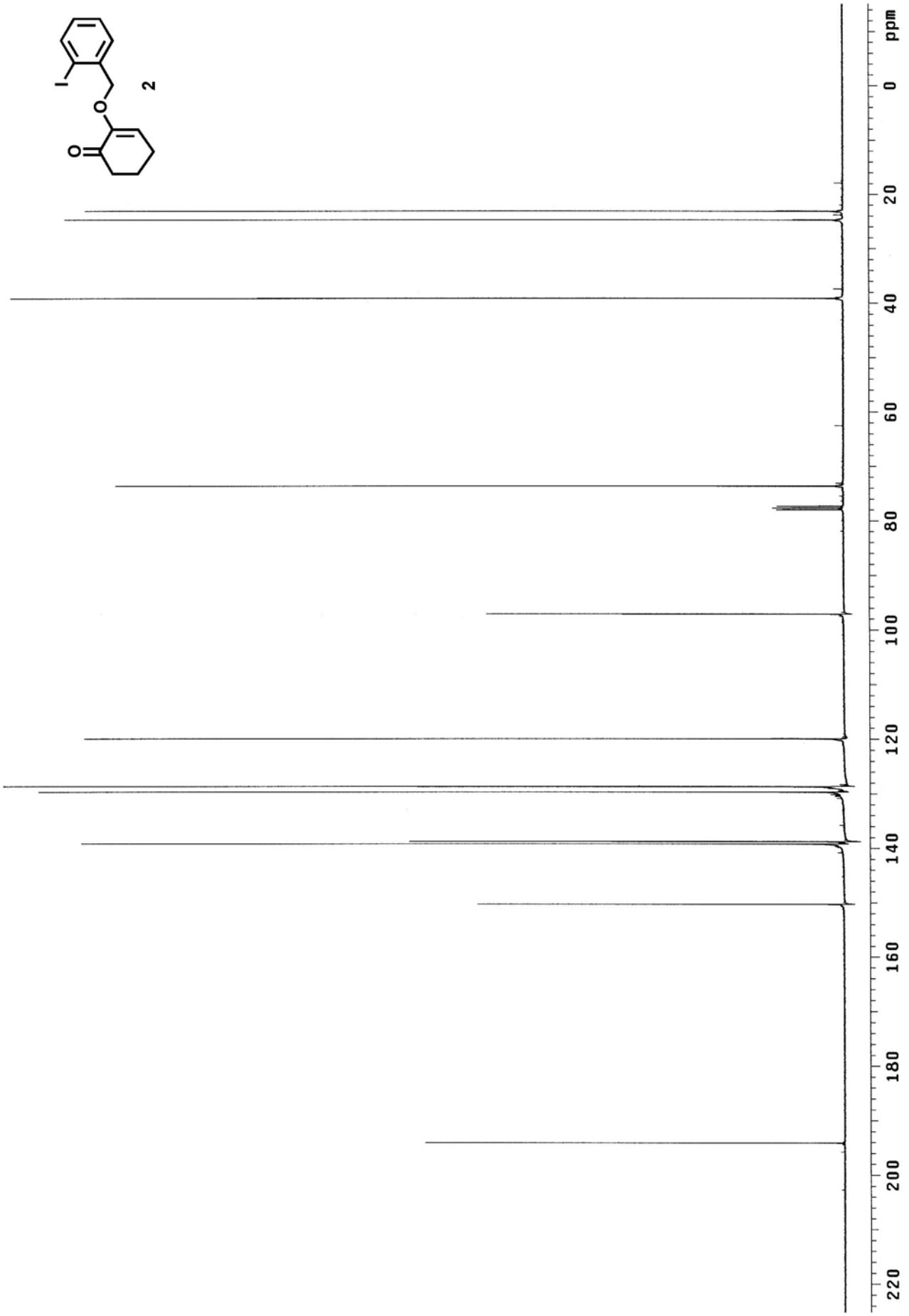
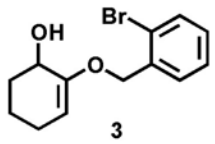

I
O
O
2
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 10 Mar 2007 20:37:10

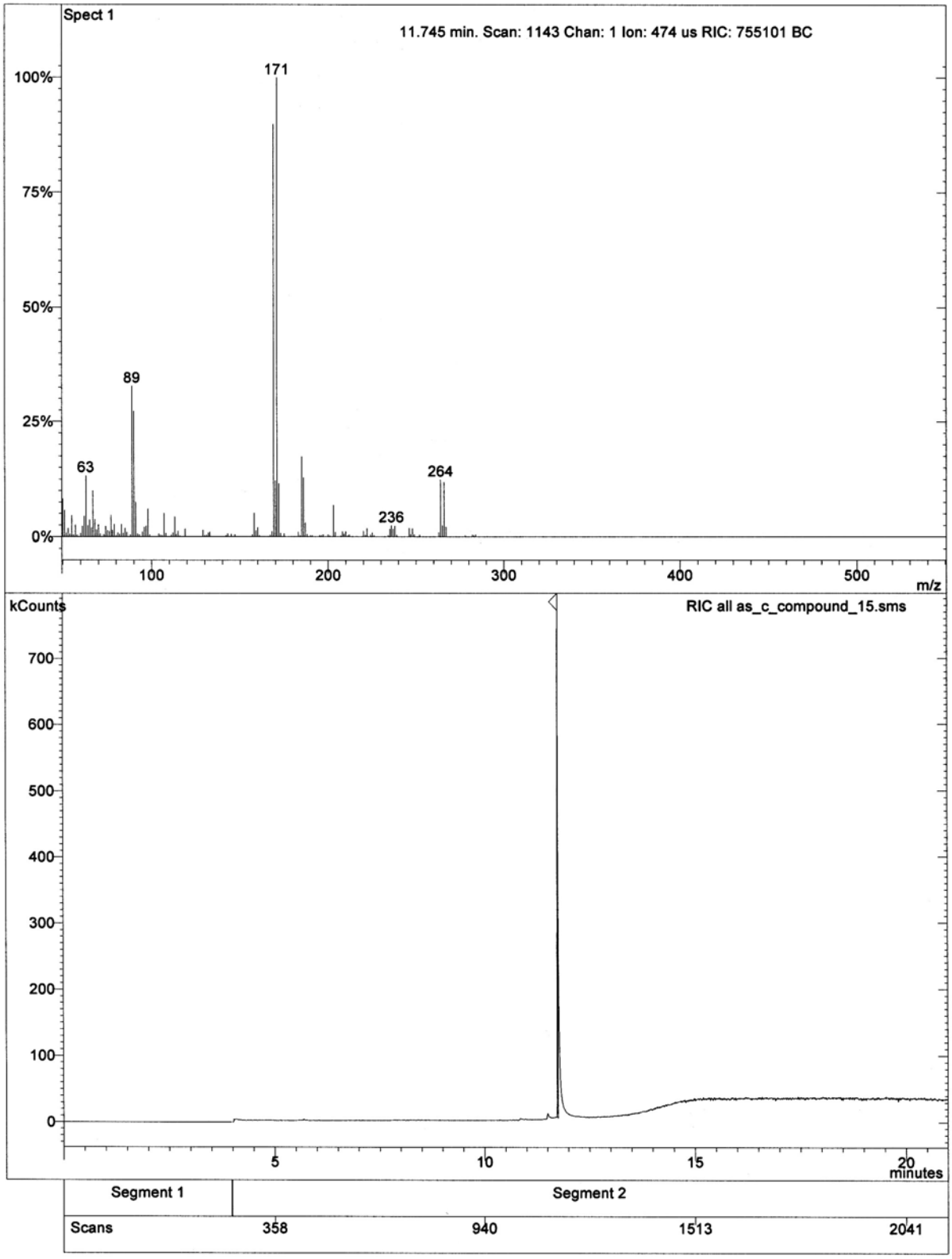


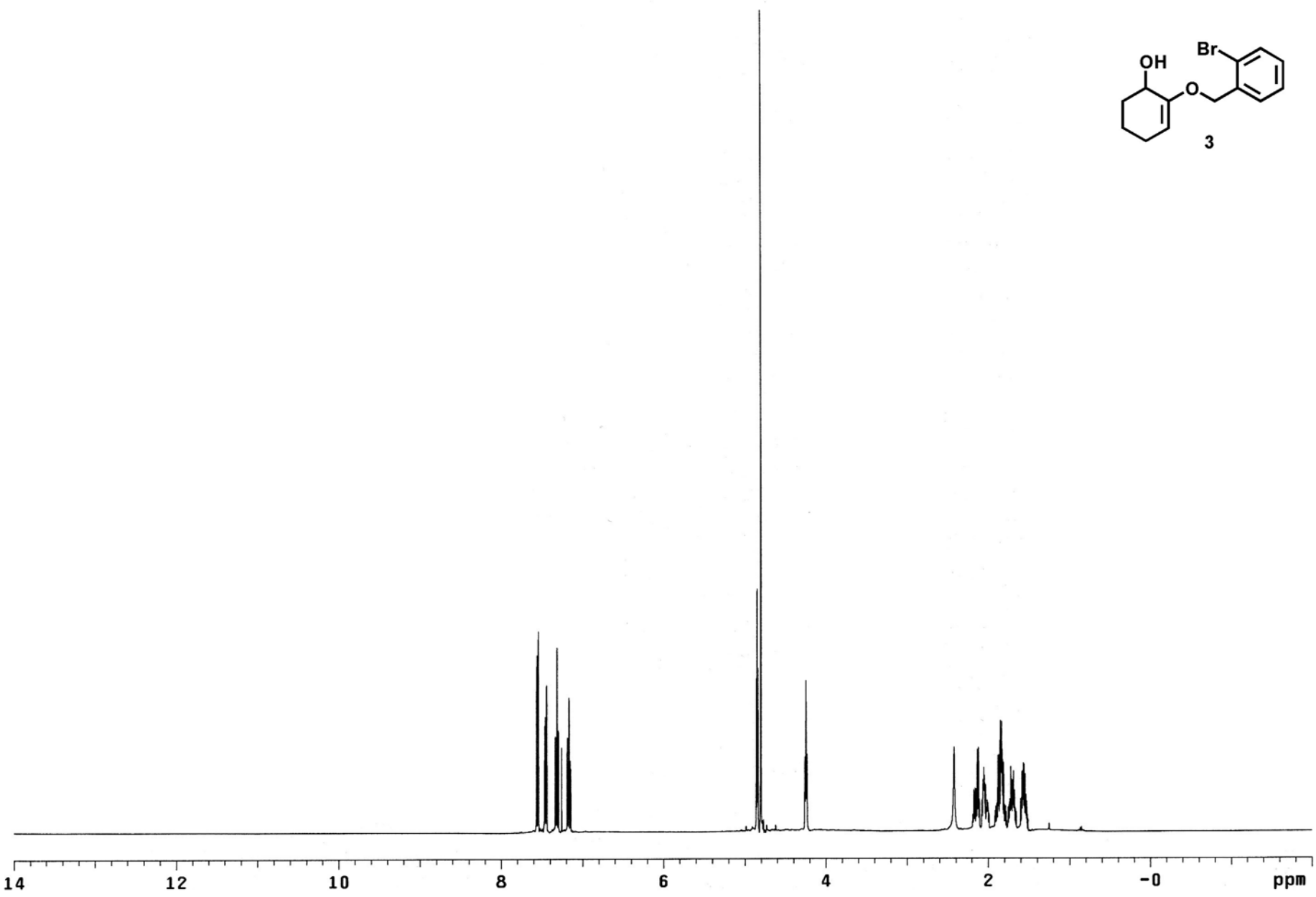
3

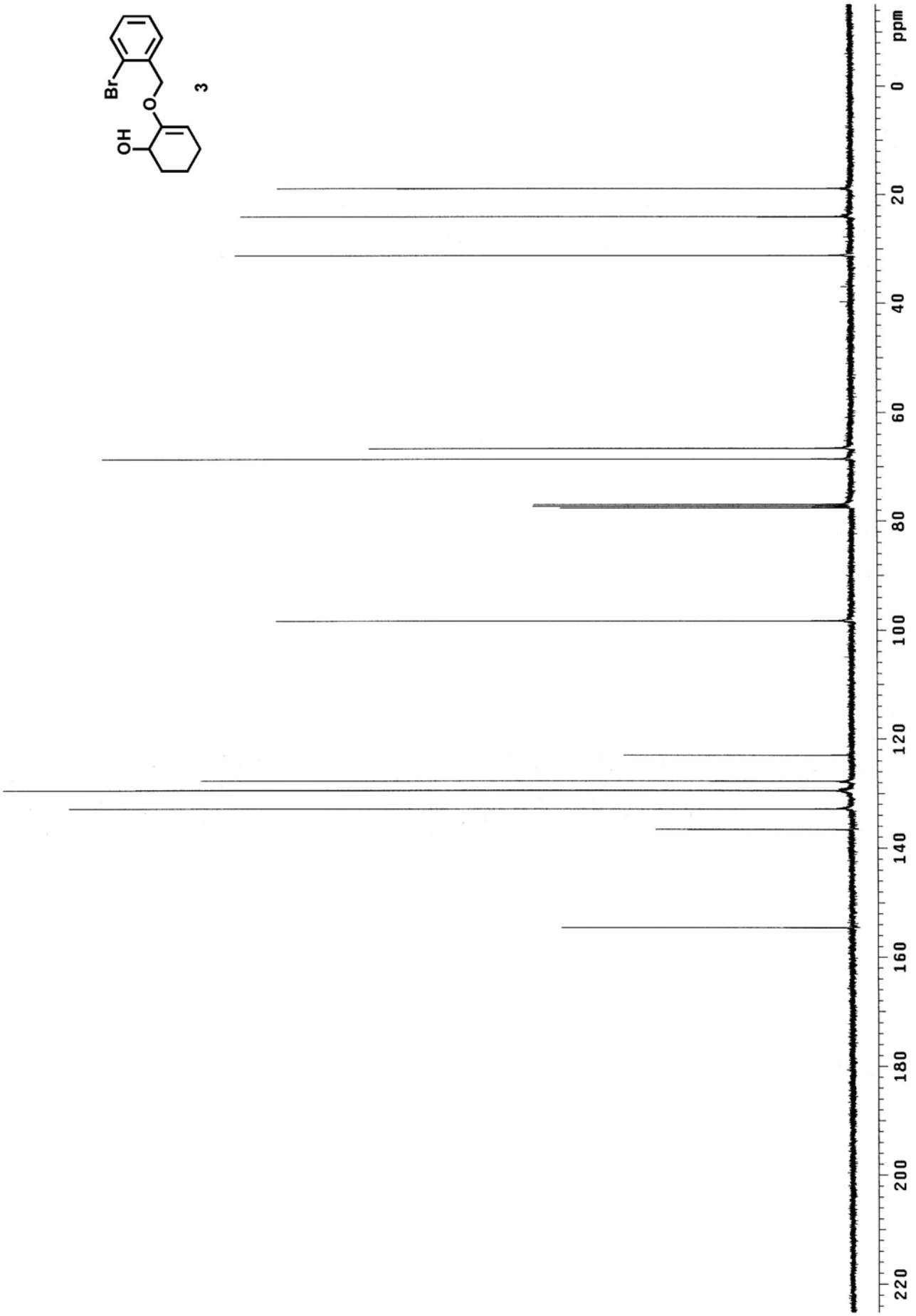
Chromatogram Plot

File: m:\spiro\compounds\as_c_compound_15.sms
Sample: as_C_Compound_15
Scan Range: 1 - 2146 Time Range: 0.00 - 20.99 min.
Sample Notes: ROUTINE

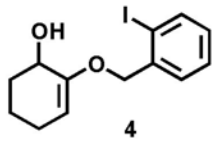
Operator: Operator
Date: 2005-07-07 14:41




OH
Br
O
3
14
12
10
8
6
4
2
-0
ppm


Br
O
OH
3
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 08 Mar 2007 13:14:33

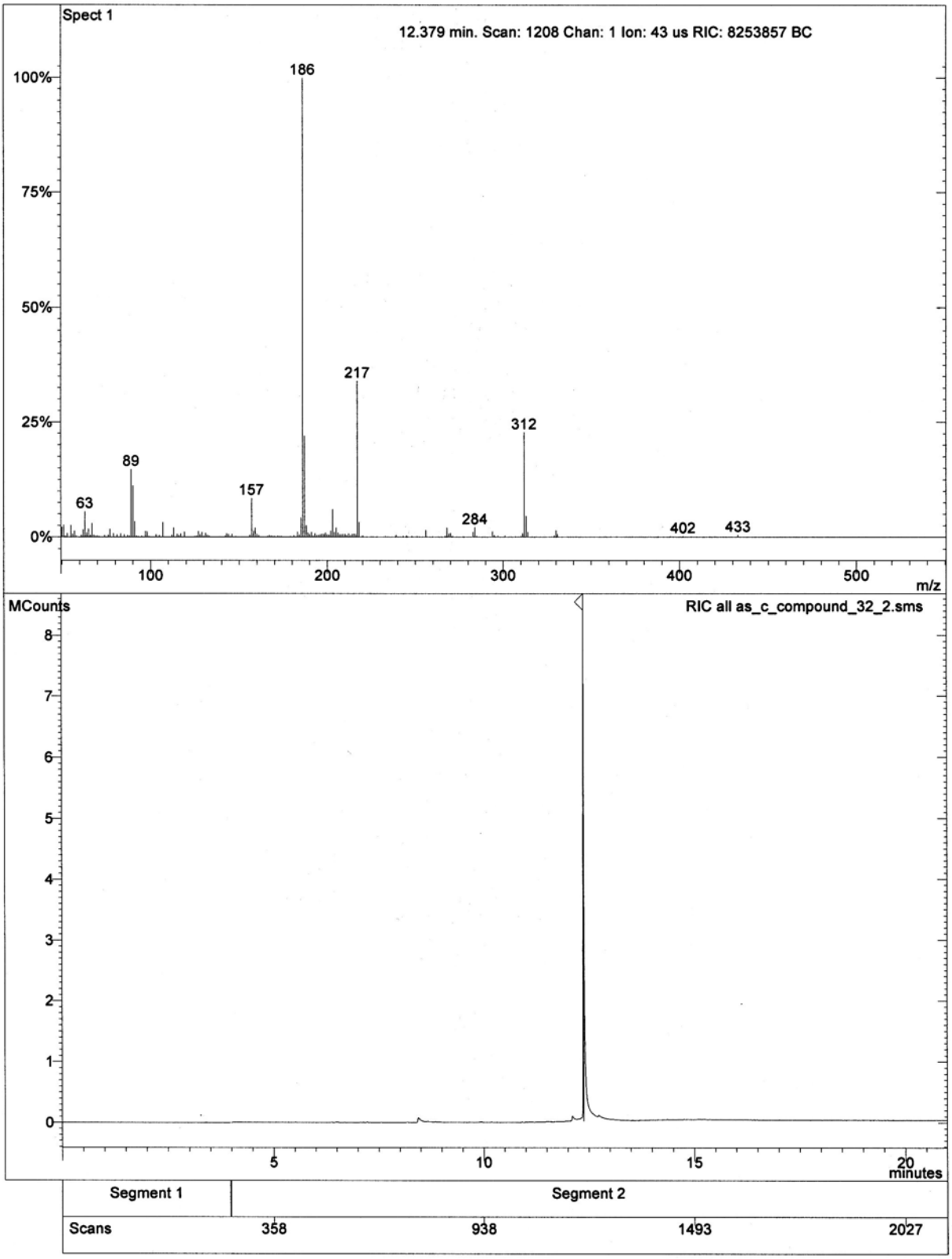


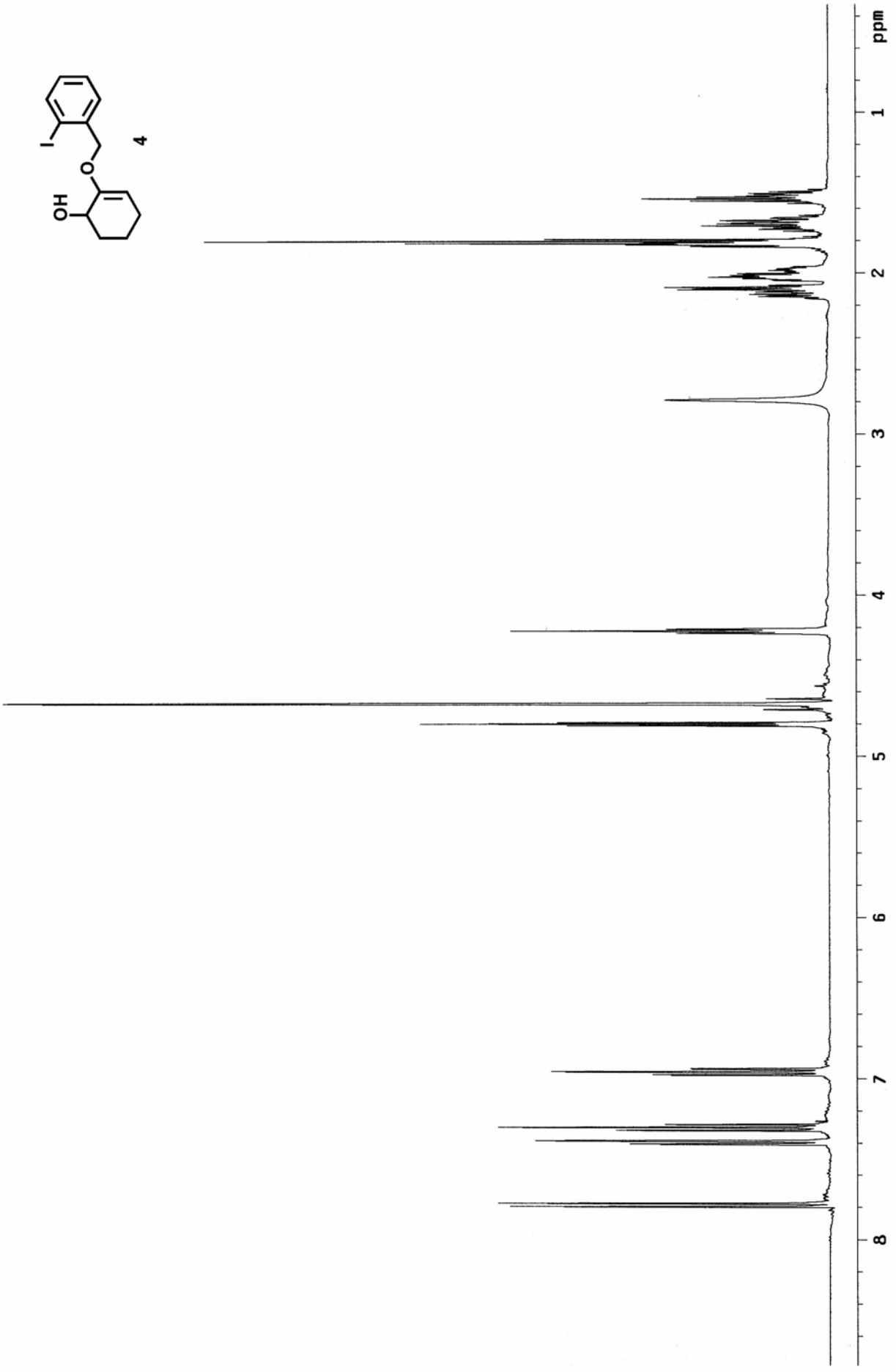
4

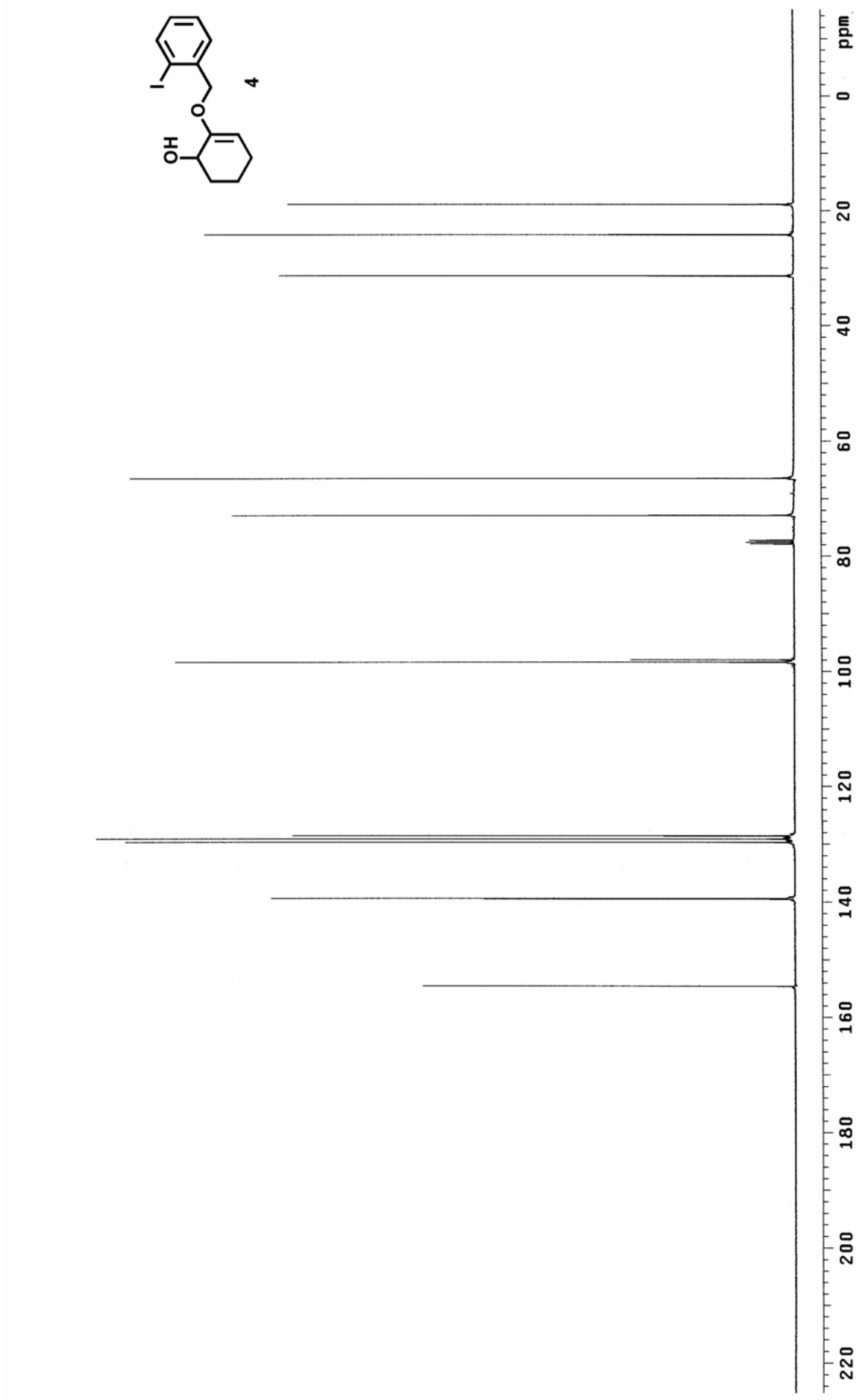
Chromatogram Plot

File: m:\spiro\compounds\as_c_compound_32_2.sms
Sample: as_C_Compound_32_2
Scan Range: 1 - 2131 Time Range: 0.00 - 20.99 min.
Sample Notes: ROUTINE

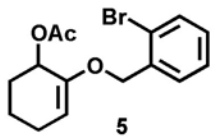
Operator: Operator
Date: 2005-07-07 00:15




I
O
OH
4
ppm
1
2
3
4
5
6
7
8


I
O
OH
4
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 09 Mar 2007 17:23:15

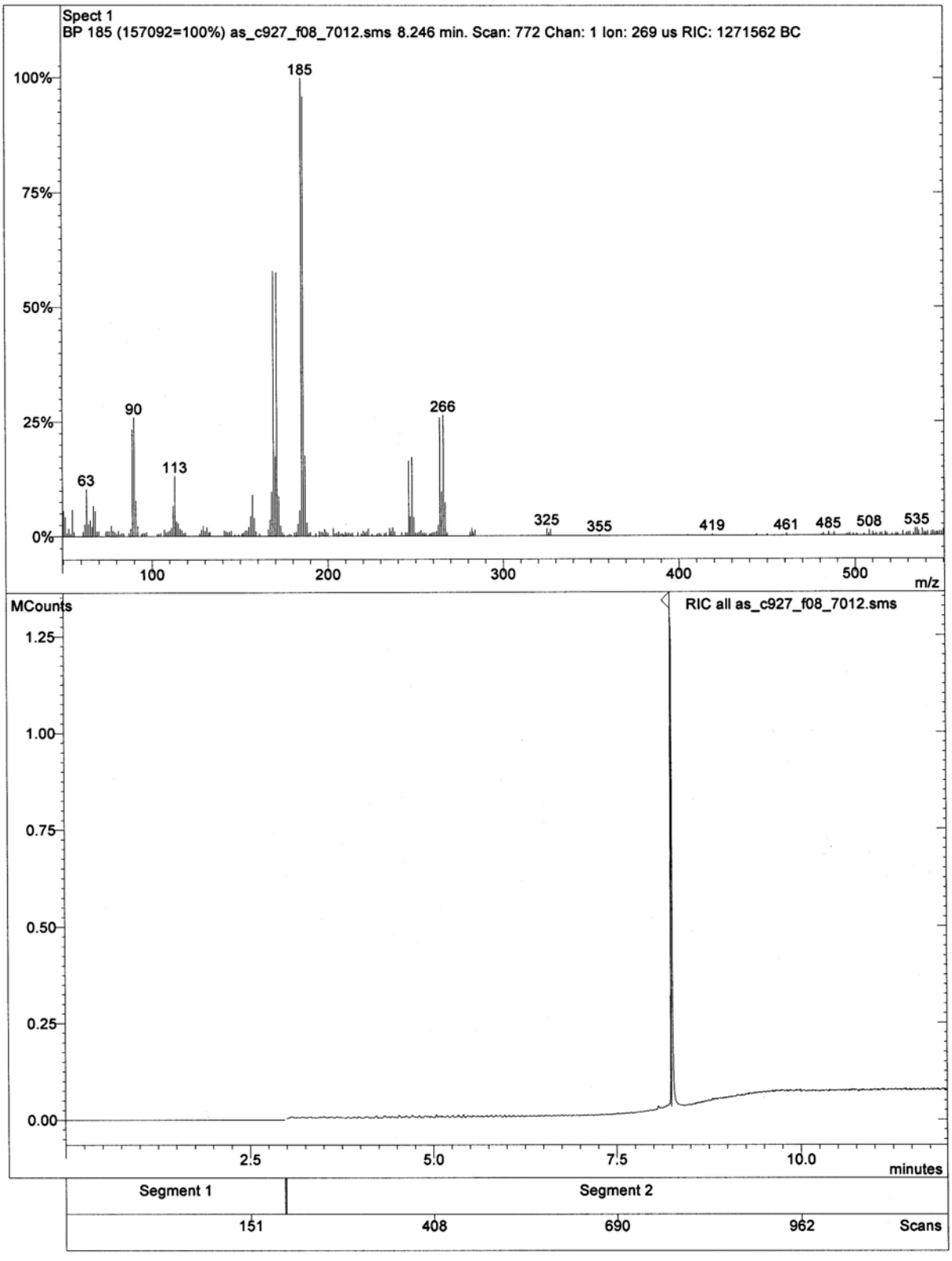


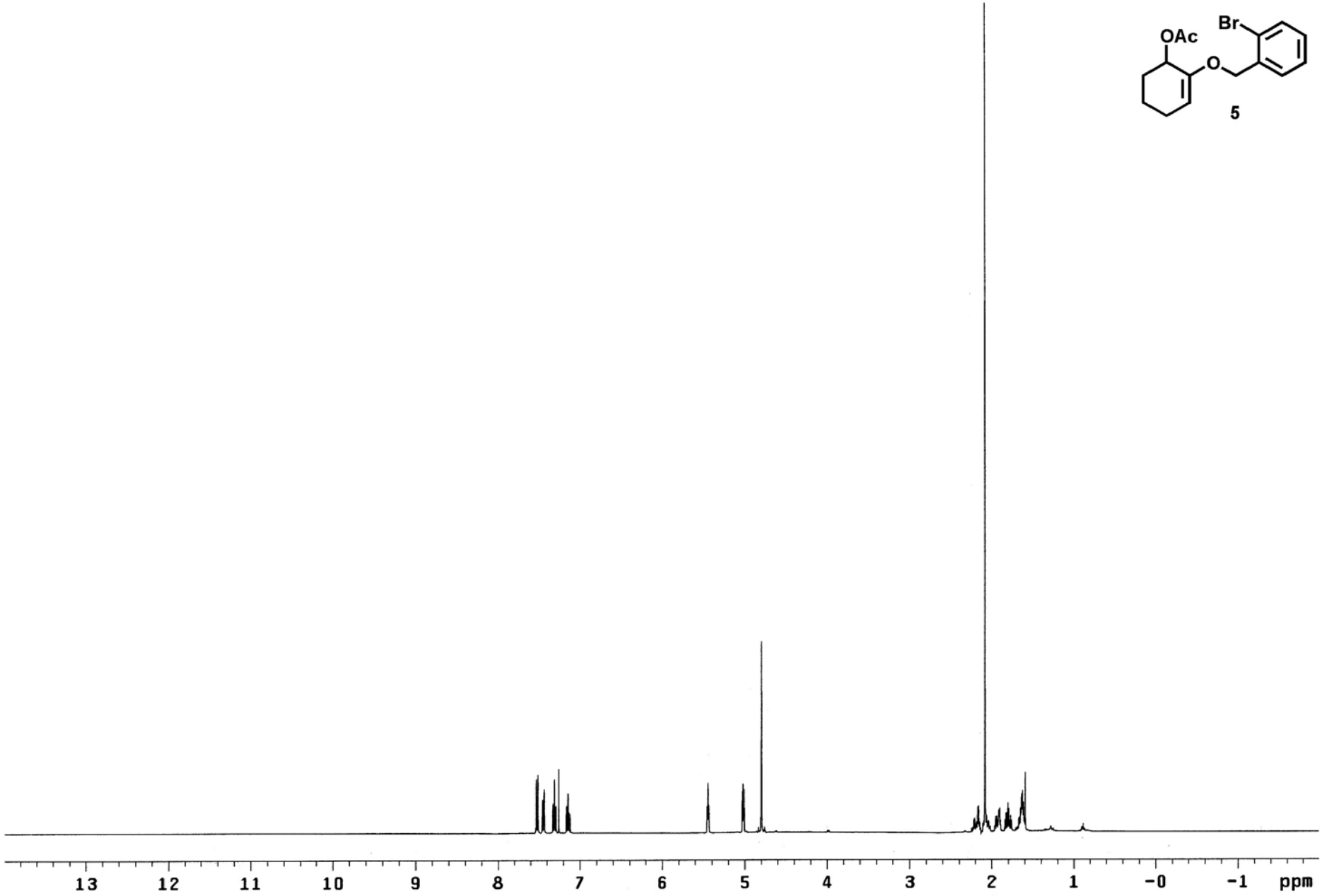
5

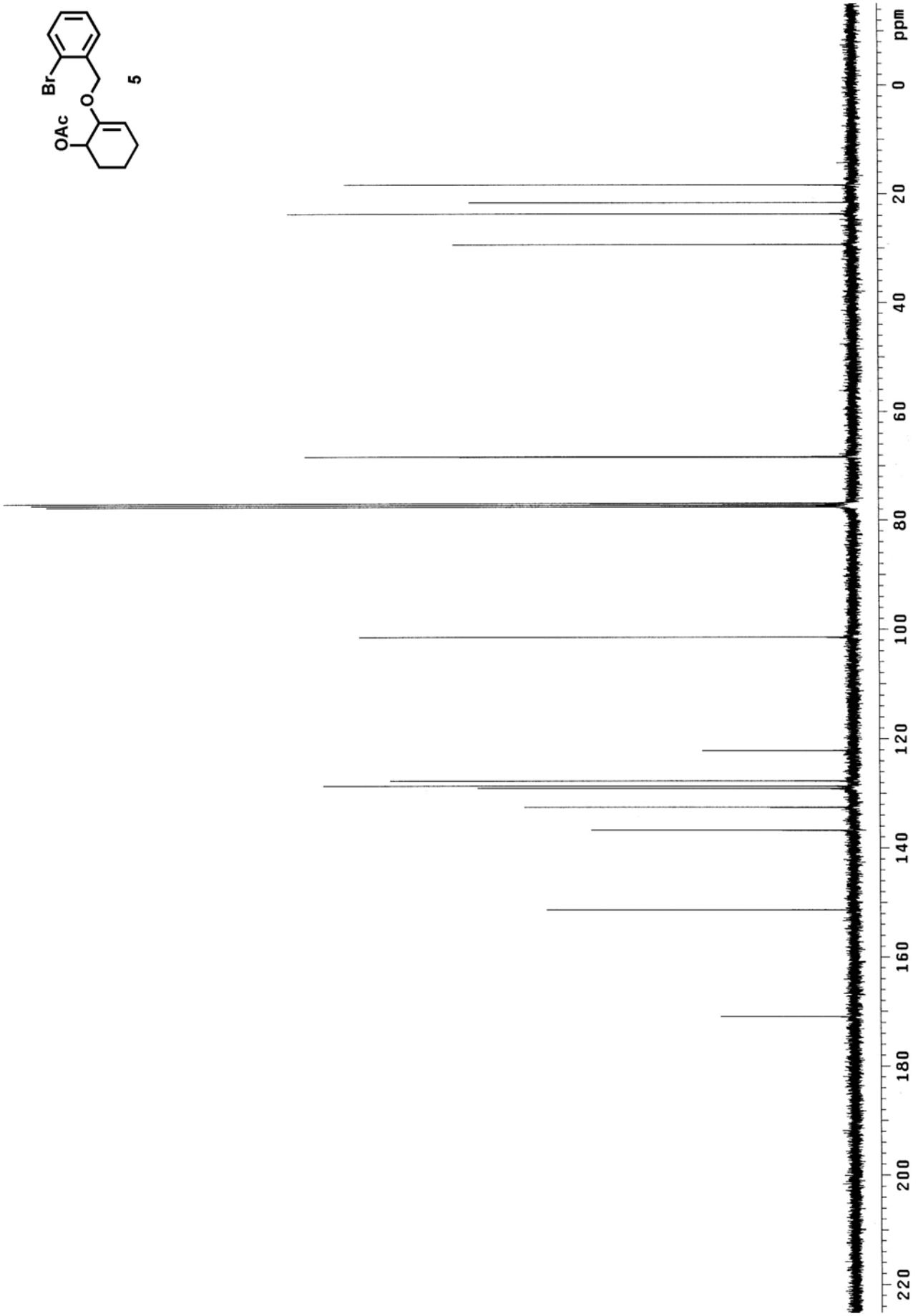
Chromatogram Plot

File: h:\as_c927_f08_7012.sms
Sample: as_S927_F08_7012
Scan Range: 1 - 1175 Time Range: 0.00 - 11.98 min.
Sample Notes: ROUTINE

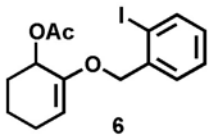
Operator: Operator
Date: 2006-09-09 04:15




OAc
Br
O
5
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm


Br
O
OAc
5
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 08 Mar 2007 13:16:39

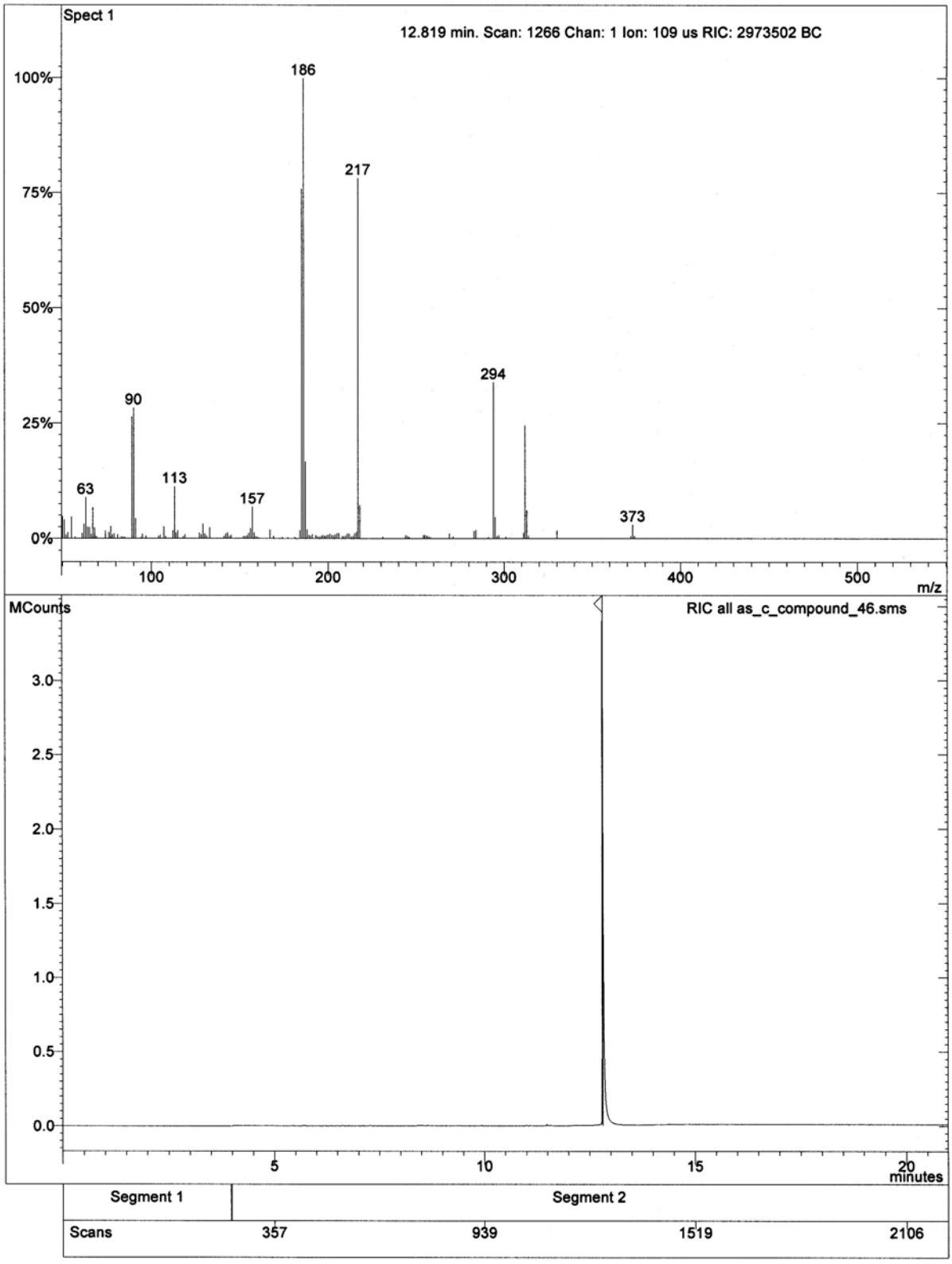


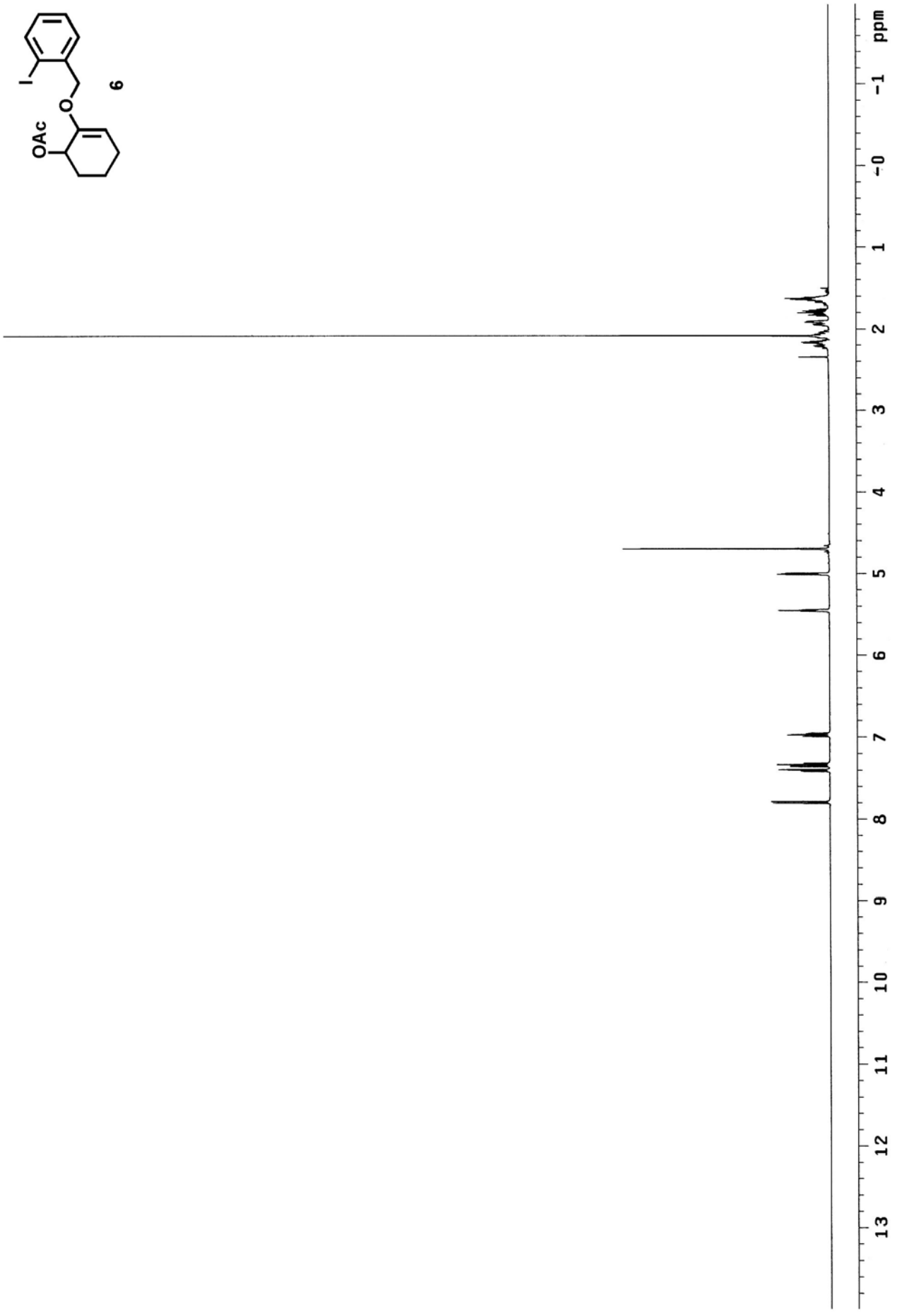
6

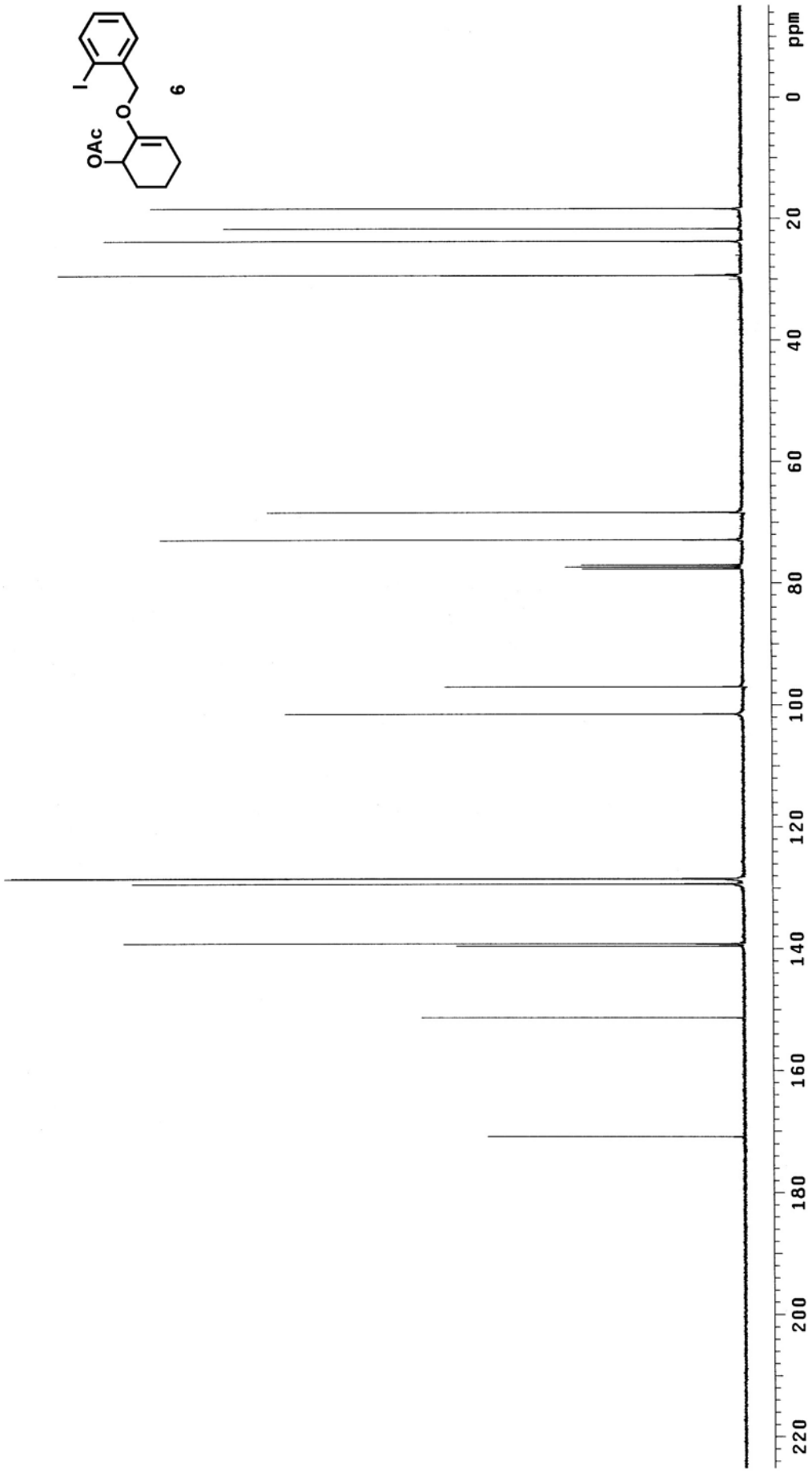
Chromatogram Plot

File: m:\spiro\compounds\as_c_compound_46.sms
Sample: as_C_Compound_46
Scan Range: 1 - 2221 Time Range: 0.00 - 20.98 min.
Sample Notes: ROUTINE

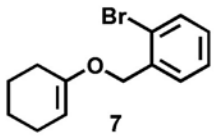
Operator: Operator
Date: 2005-10-06 19:11




I
O
OAc
6
ppm
13
12
11
10
9
8
7
6
5
4
3
2
1
−0
−1


I
O
OAc
6
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 07 Feb 2006 14:43:33

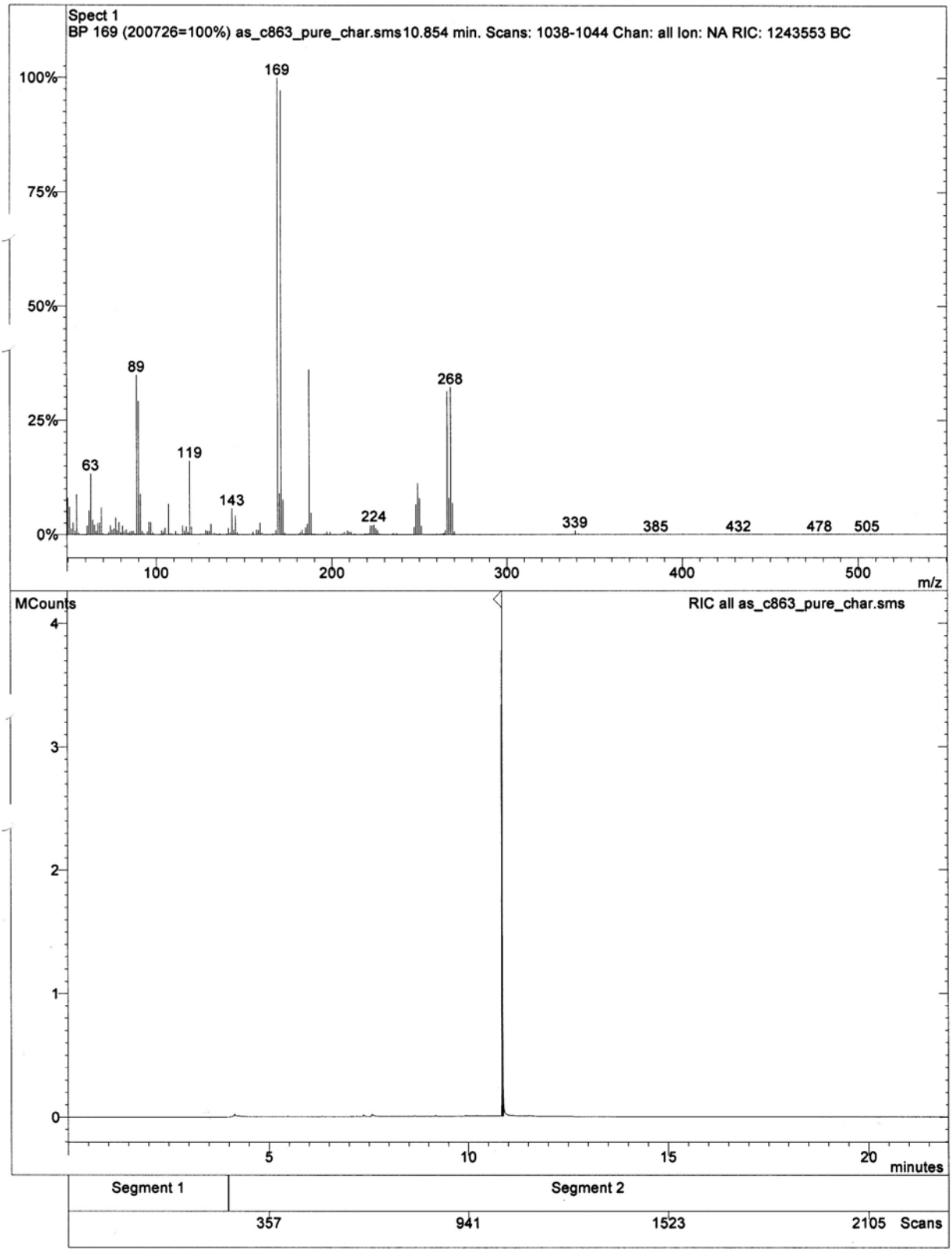


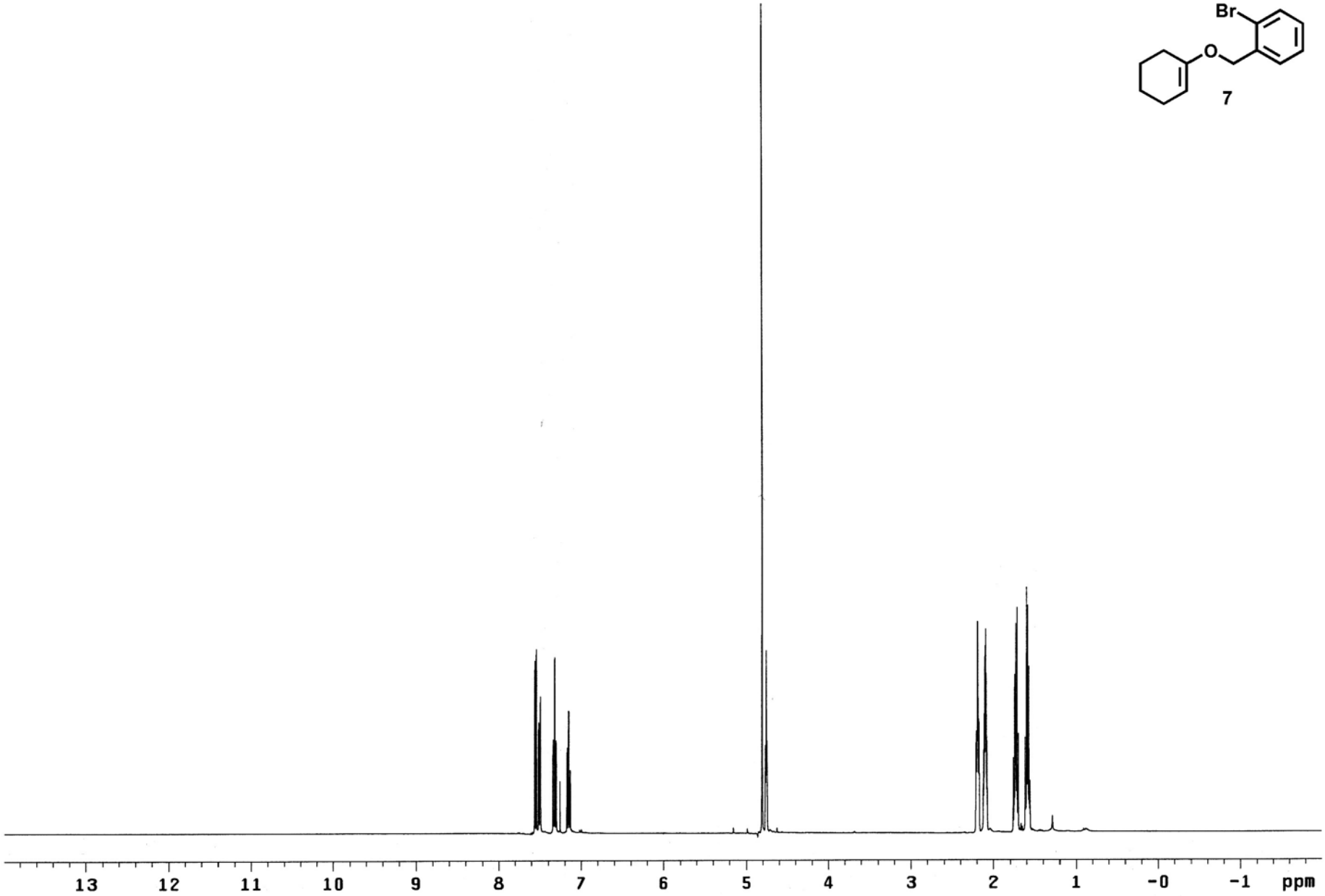
7

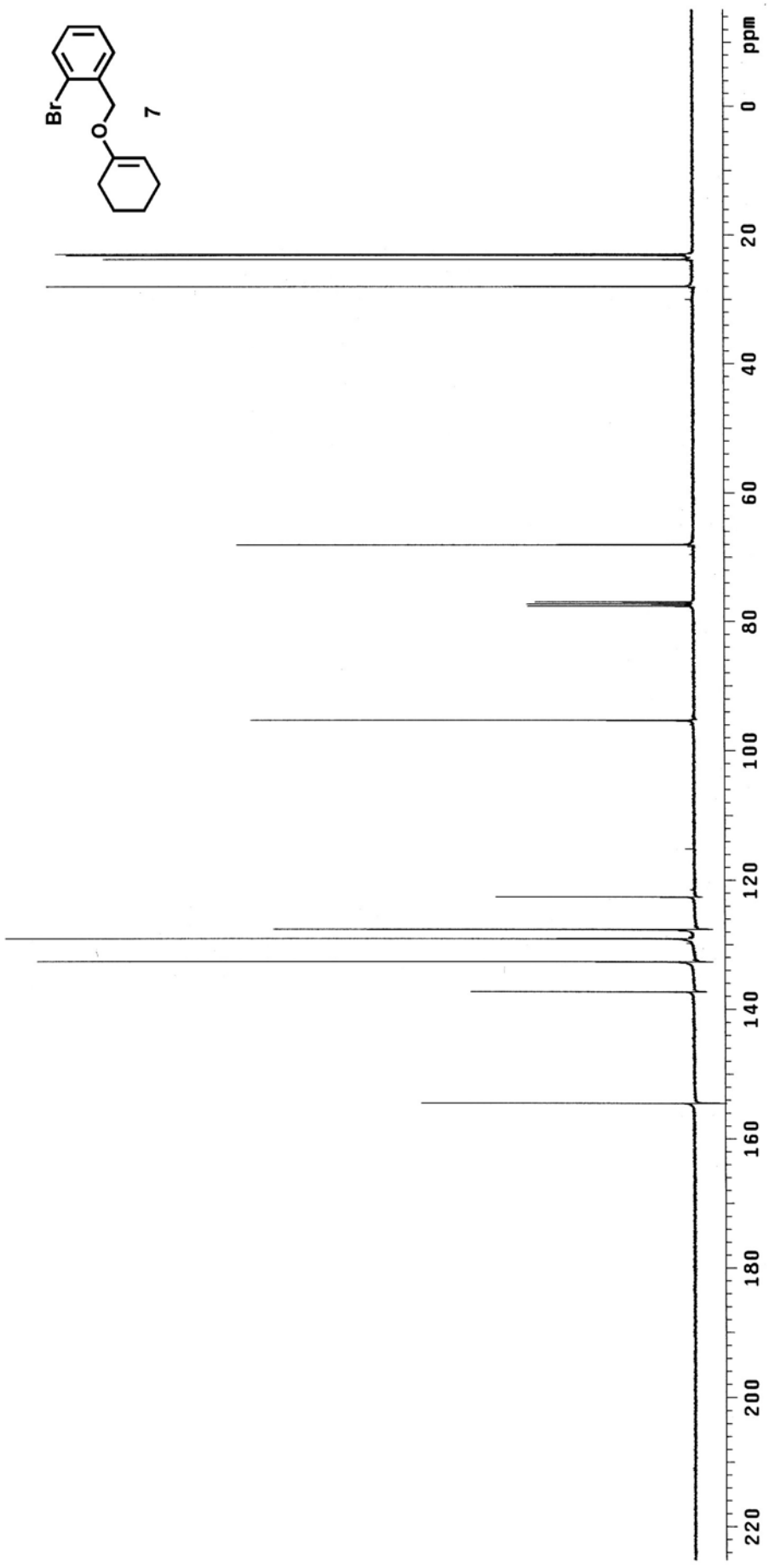
Chromatogram Plot

File: h:\as_c863_pure_char.sms
Sample: as_C863_pure_Char
Scan Range: 1 - 2336 Time Range: 0.00 - 21.98 min.
Sample Notes: ROUTINE

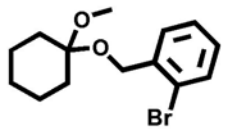
Operator: Operator
Date: 2006-02-07 13:33




Br
O
7
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm


Br
O
7
ppm
0
20
40
60
80
100
120
140
160
180
200
220

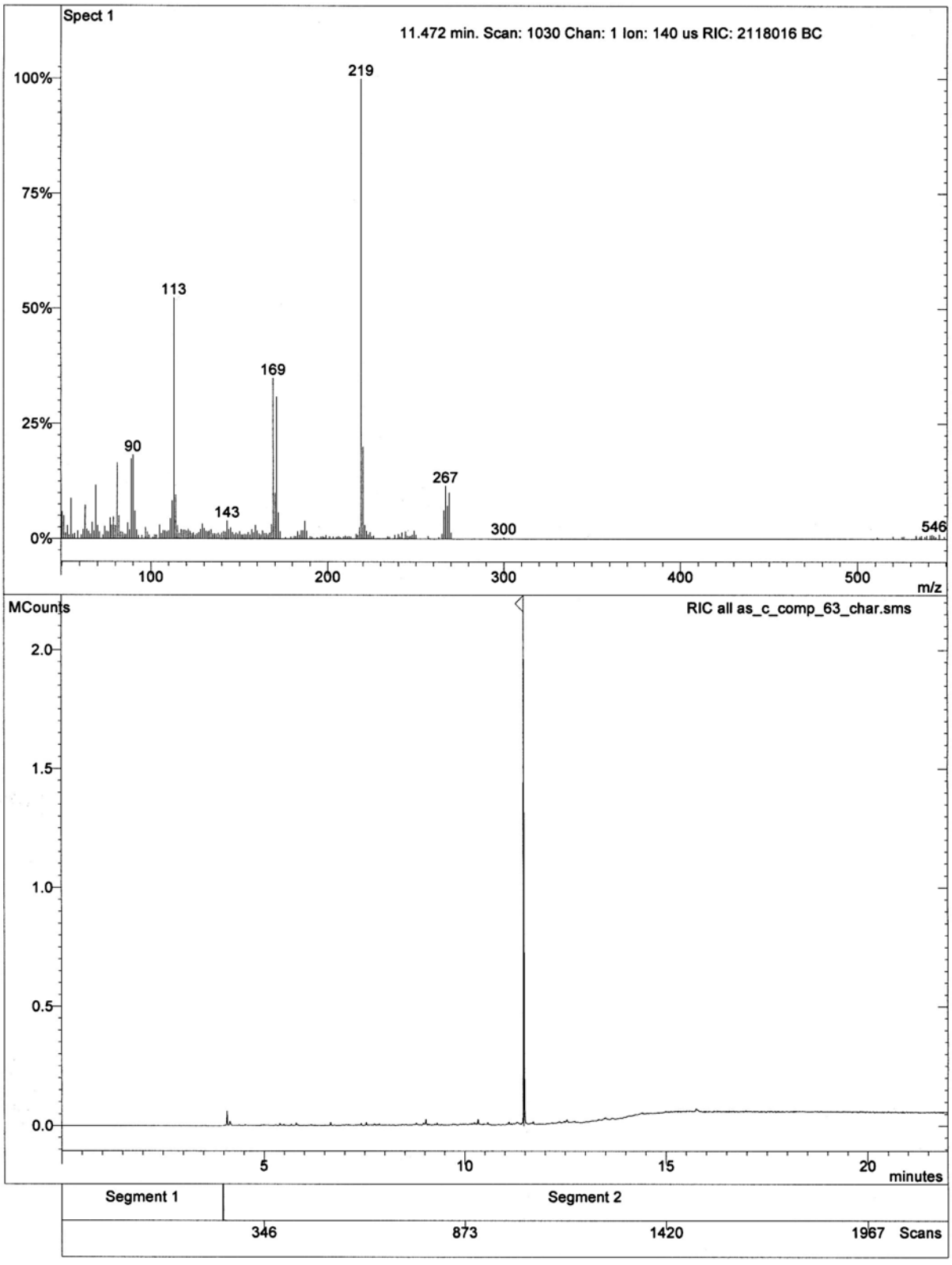
Print Date: 20 Mar 2007 16:09:37

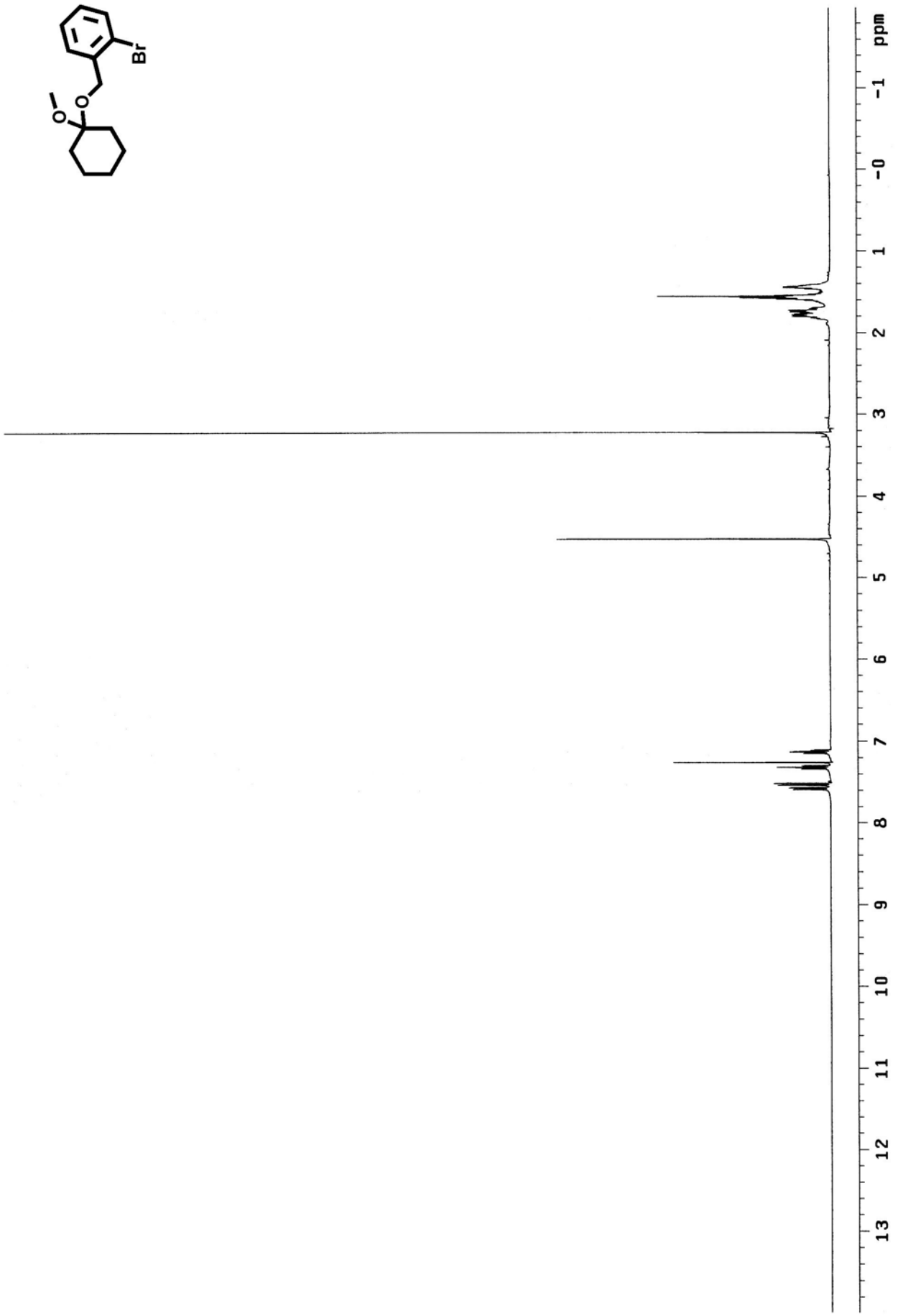


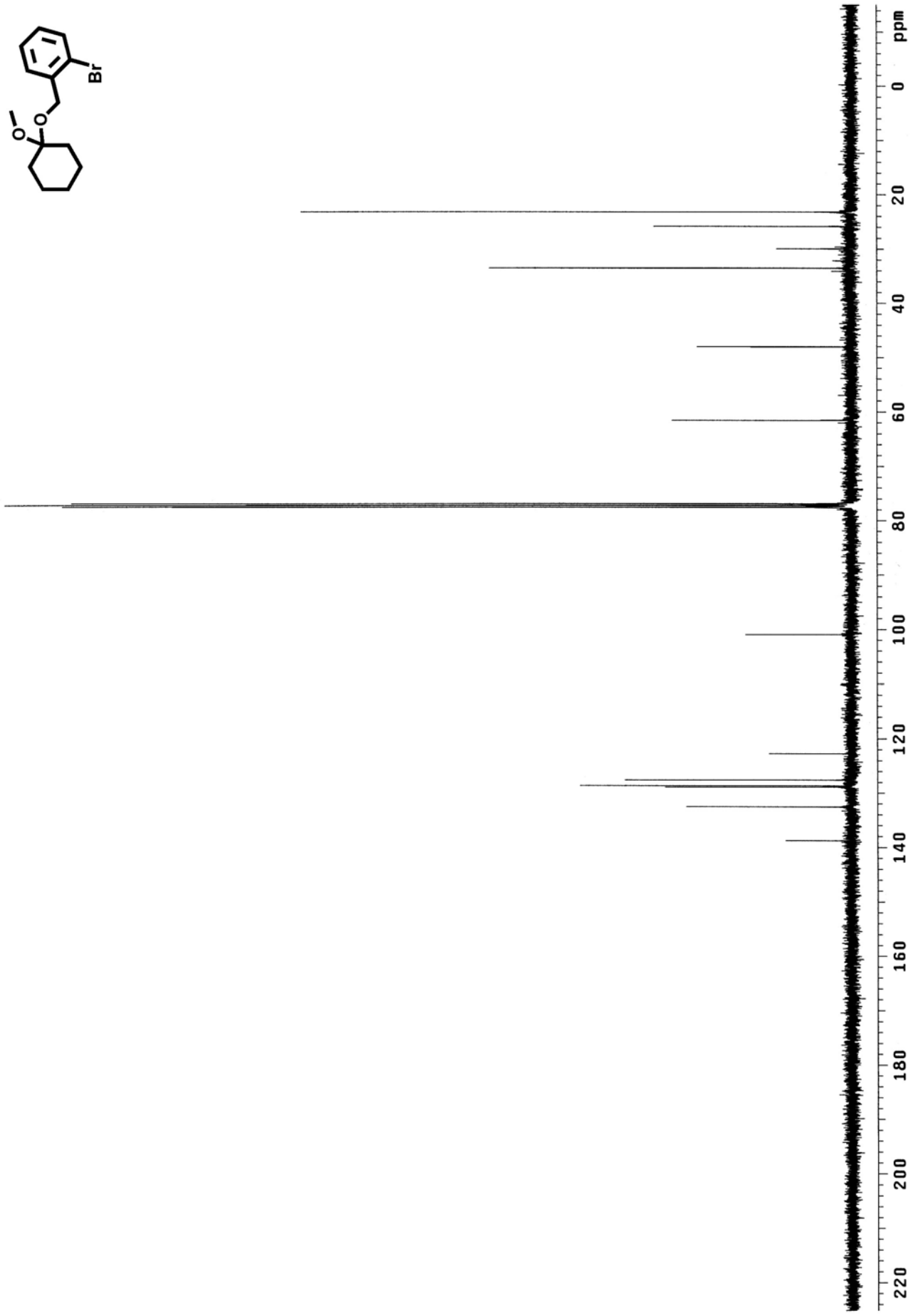
Chromatogram Plot

File: h:\as_c_comp_63_char.sms
Sample: as_C_Comp_63_Char
Scan Range: 1 - 2183 Time Range: 0.00 - 21.98 min.
Sample Notes: ROUTINE

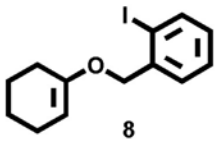
Operator: Operator
Date: 2006-09-17 22:42




Br
O
O
ppm
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1


Br
O
O
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 03 Nov 2005 11:29:39

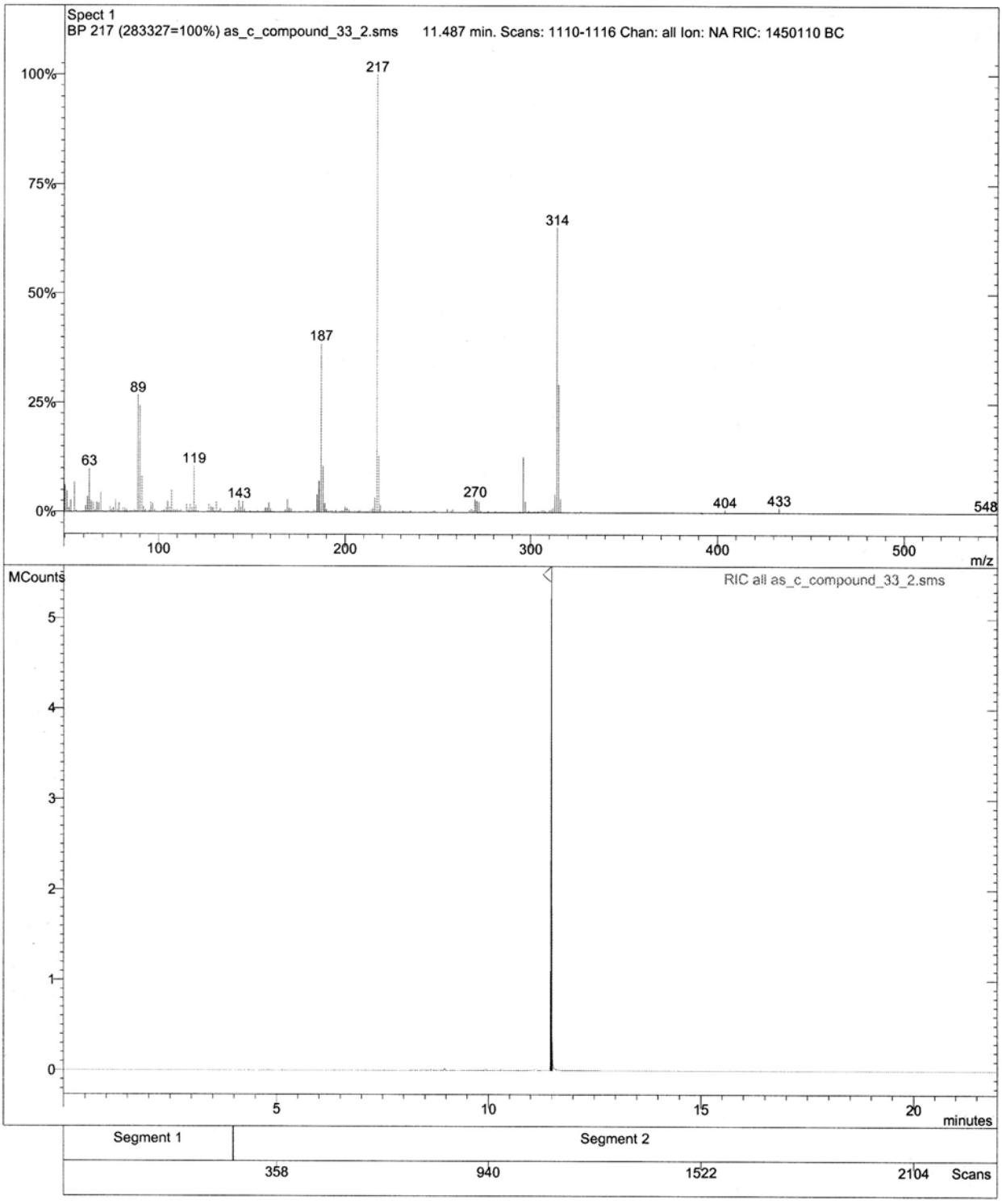


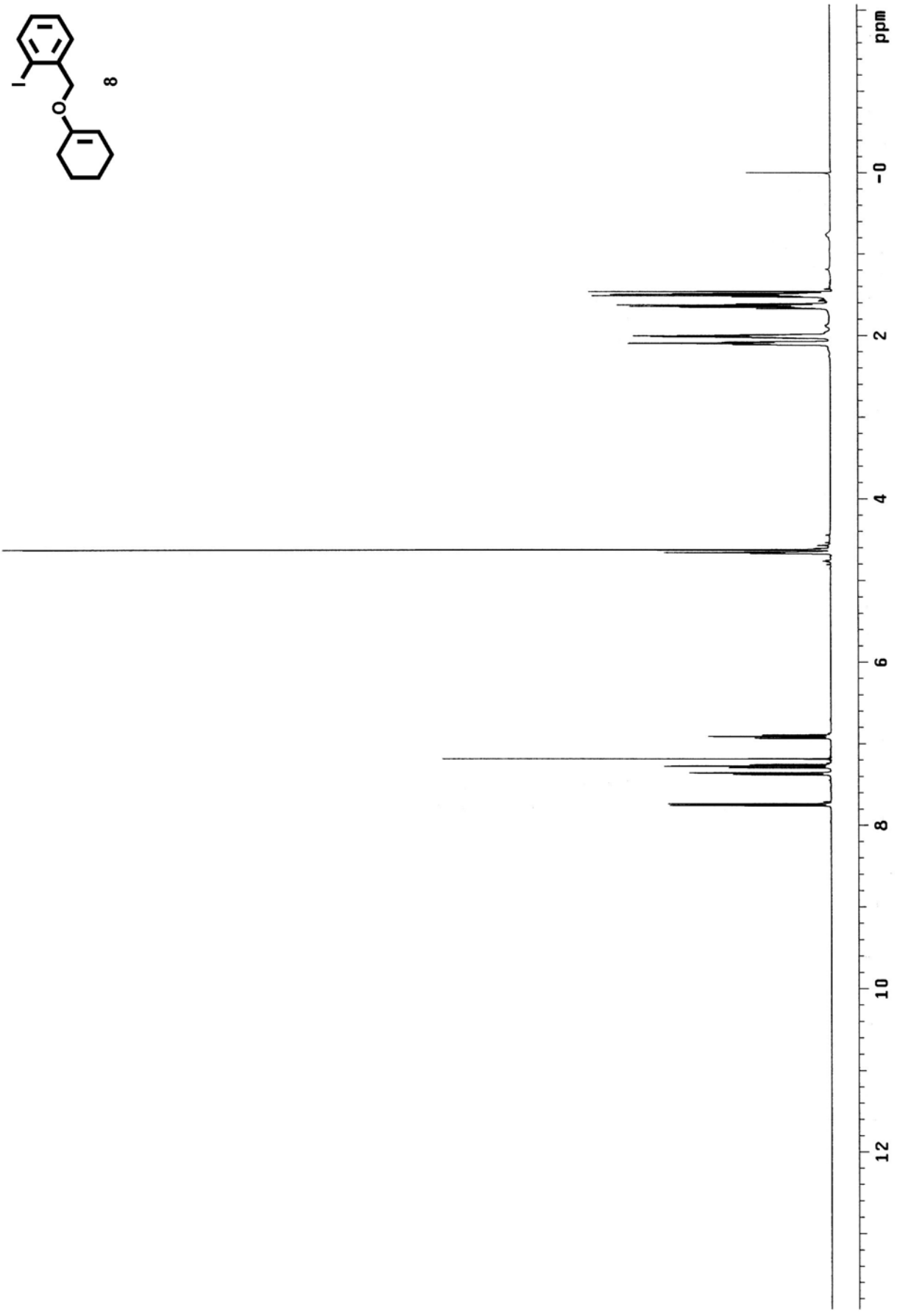
8

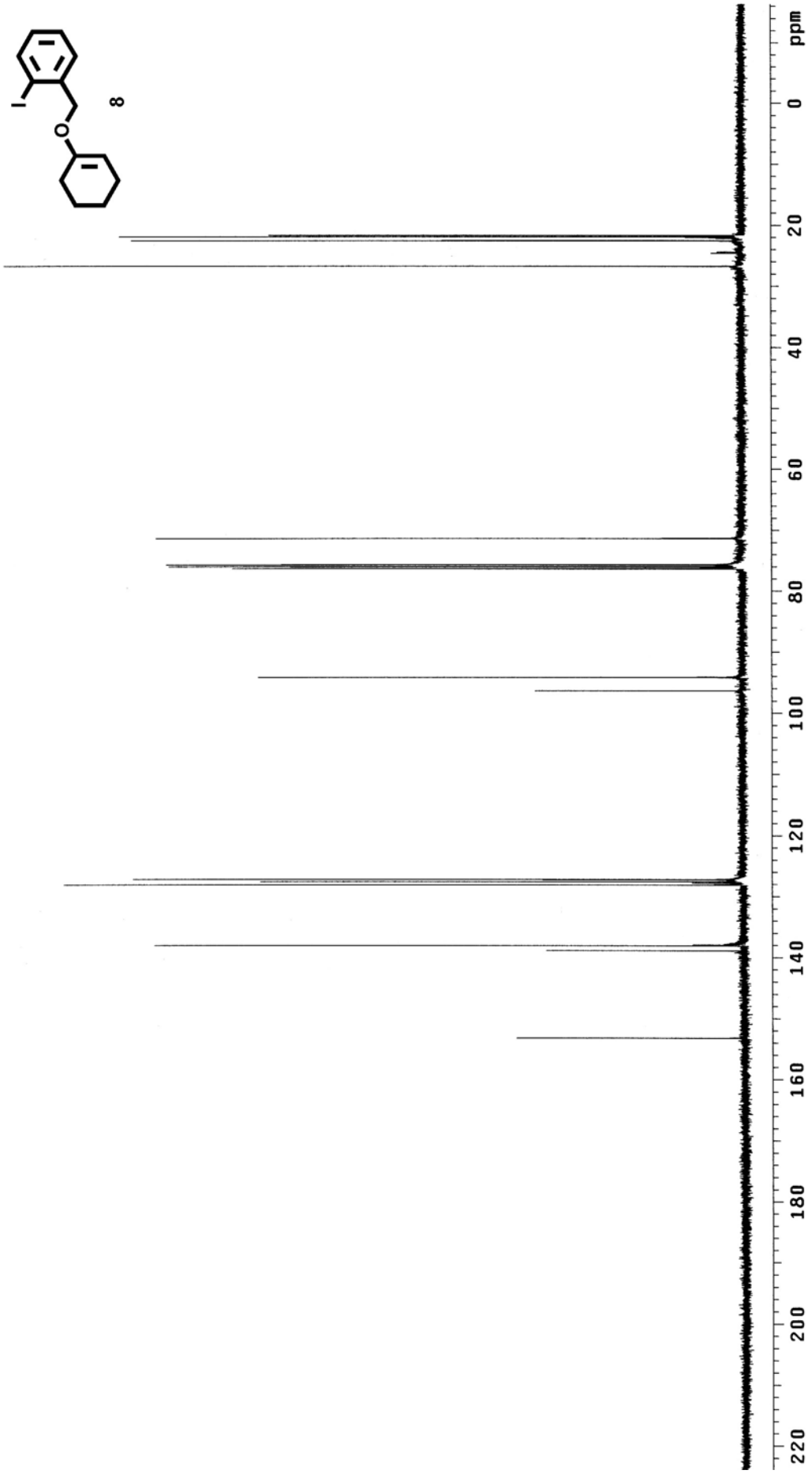
Chromatogram Plot

File: h:\as_c_compound_33_2.sms
Sample: as_C_Compound_33_2
Scan Range: 1 - 2335 Time Range: 0.00 - 21.98 min.
Sample Notes: ROUTINE

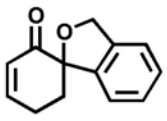
Operator: Operator
Date: 2005-10-24 00:49




I
O
8
ppm
-0
2
4
6
8
10
12


I
O
8
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 09 Mar 2007 15:43:58

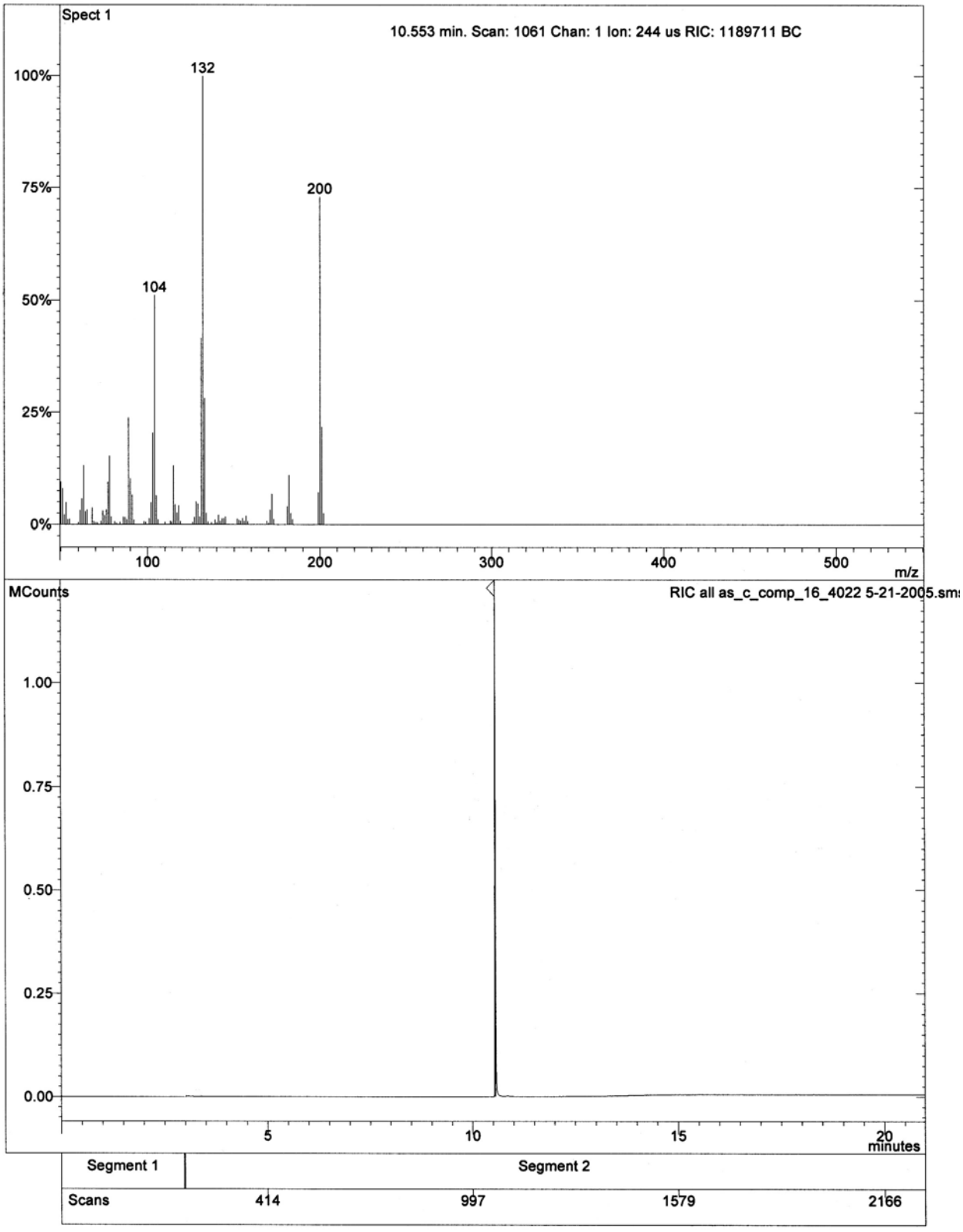


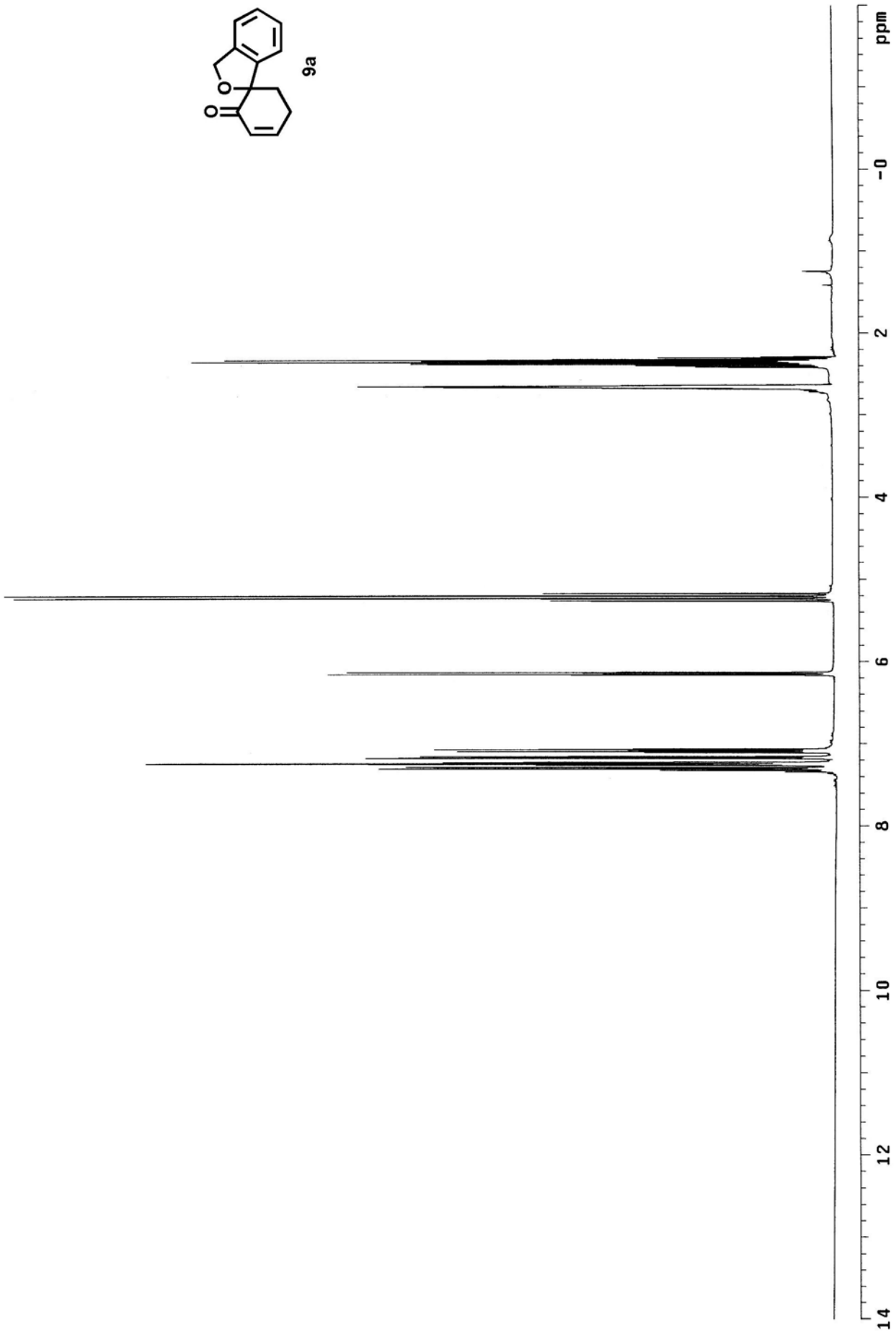
9a

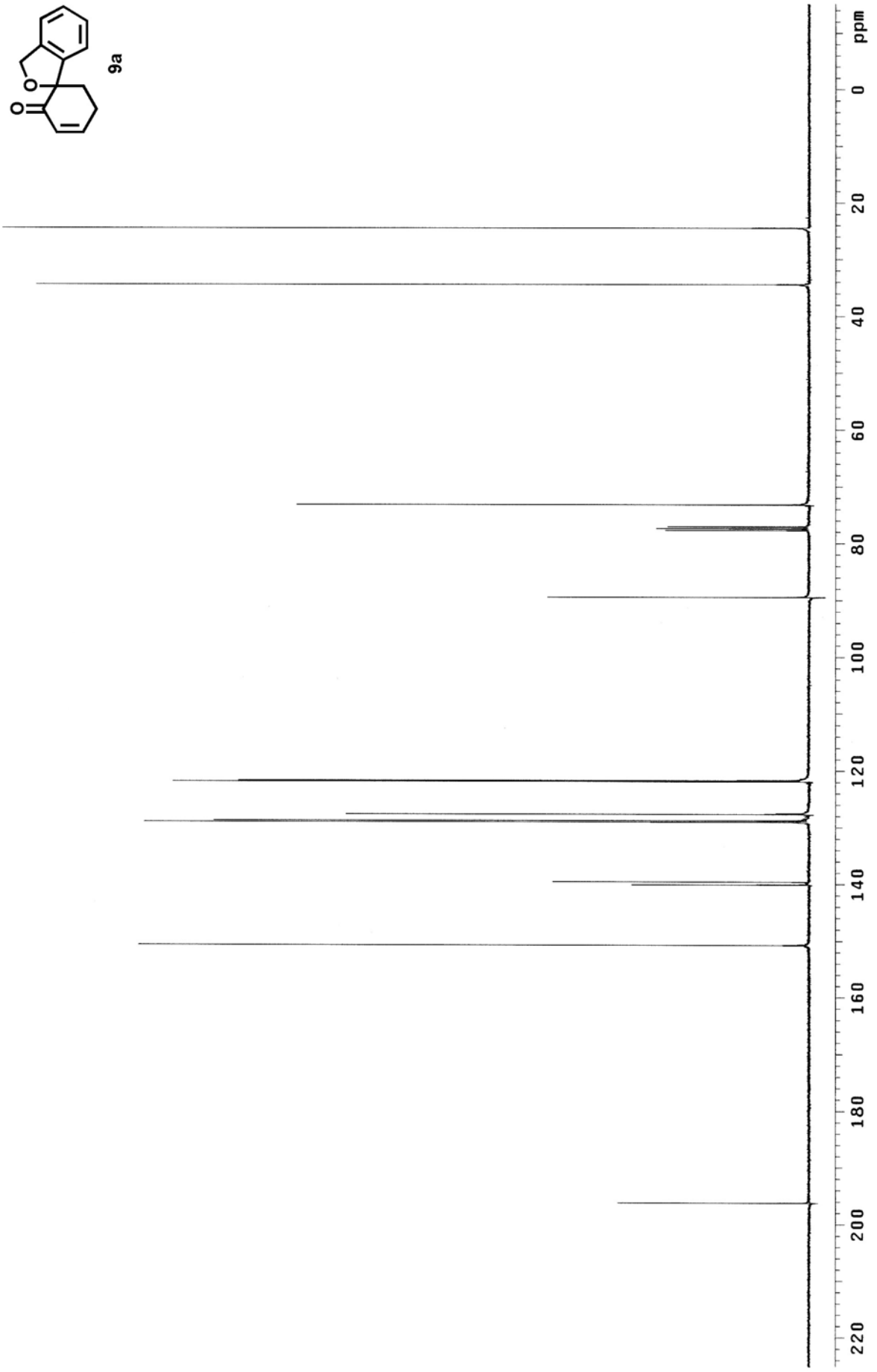
Chromatogram Plot

File: m:\spiro\compounds\as_c_comp_16_4022 5-21-2005.sms
Sample: as_C_Comp_16_4022
Scan Range: 1 - 2281 Time Range: 0.00 - 20.98 min.
Sample Notes: Routine

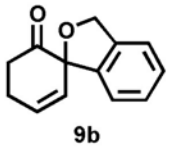
Operator: Org Farm Kemi
Date: 2005-05-21 18:13




9a
O
O
ppm
-0
2
4
6
8
10
12
14


9a
O
O
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 09 Mar 2007 15:44:25

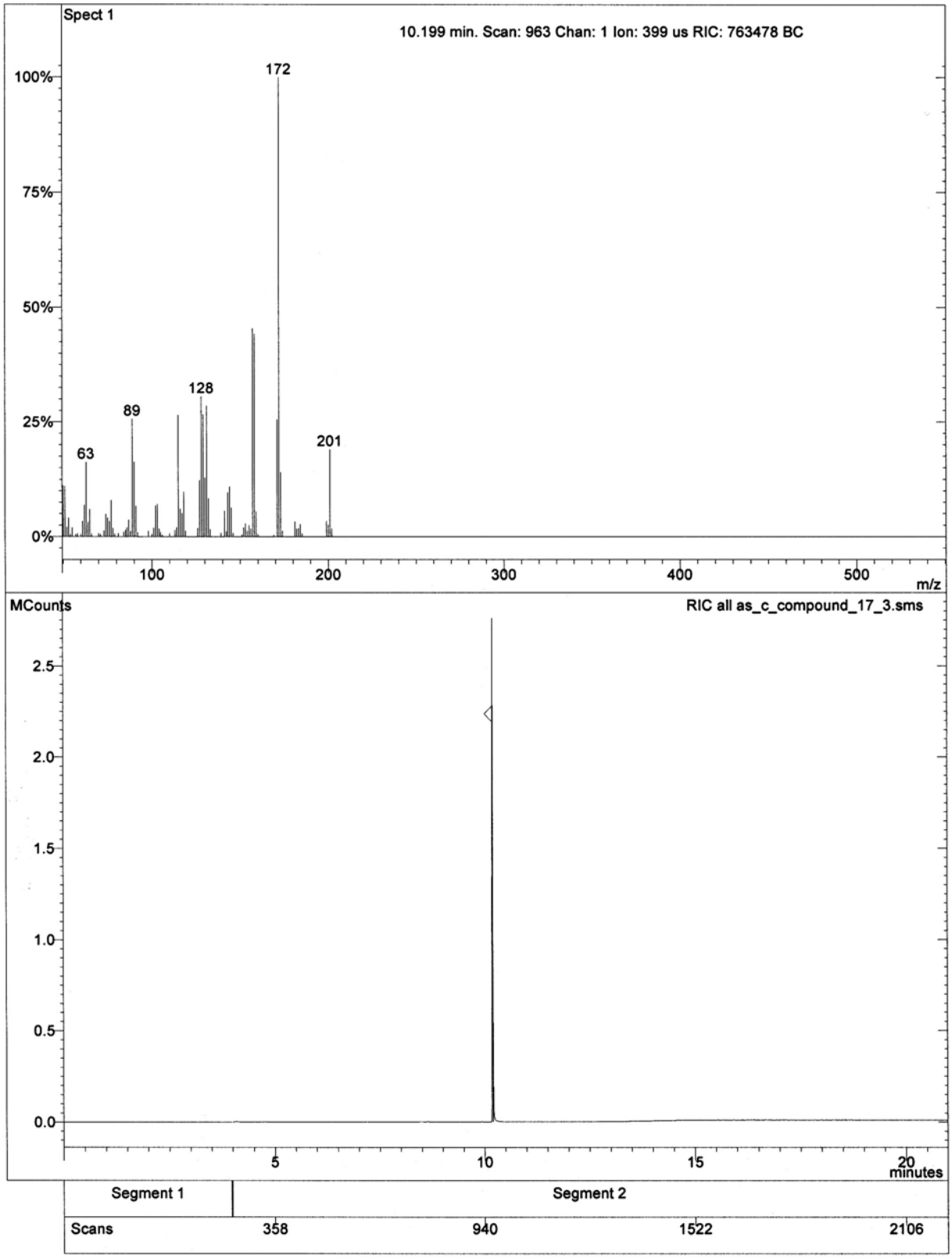


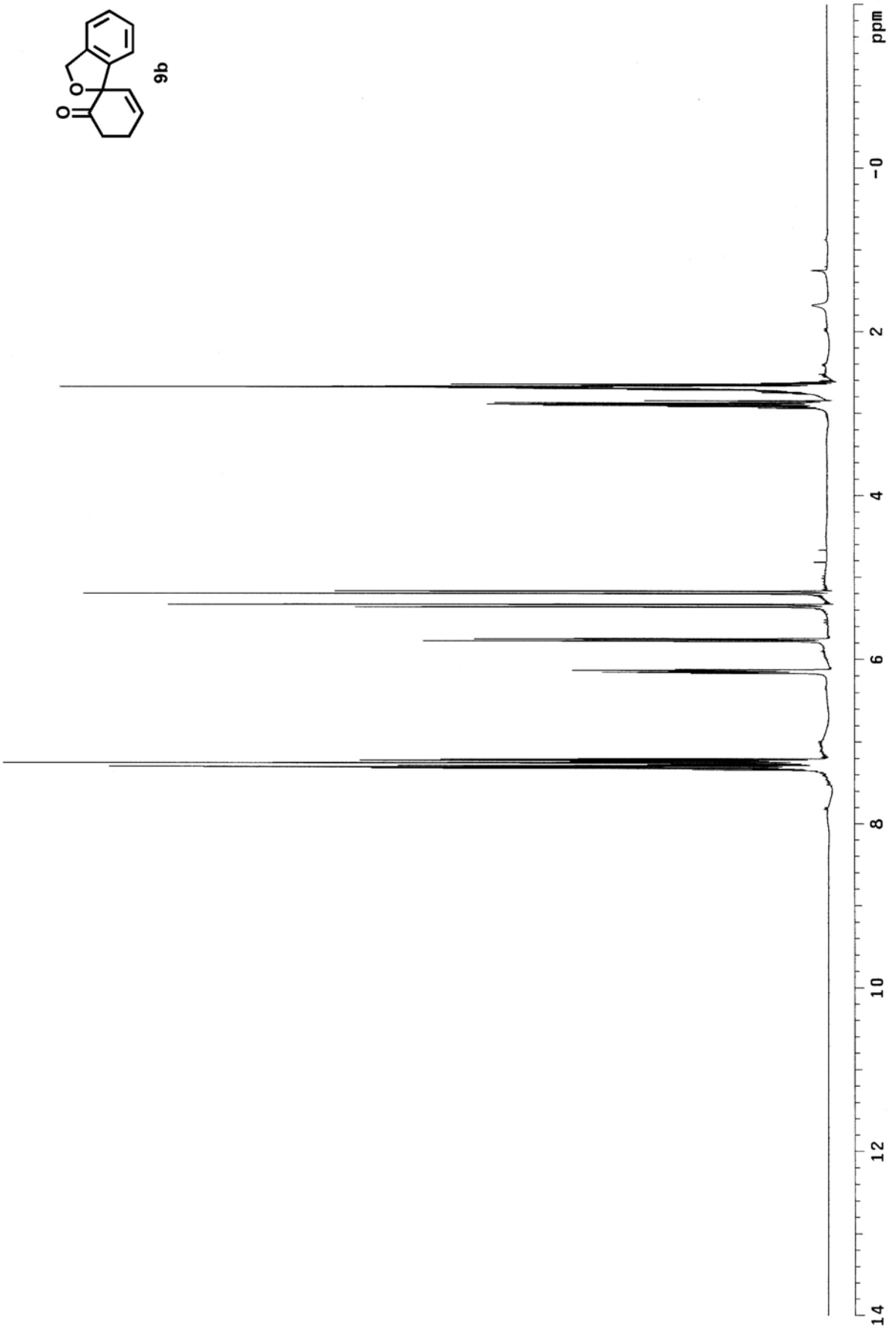
9b

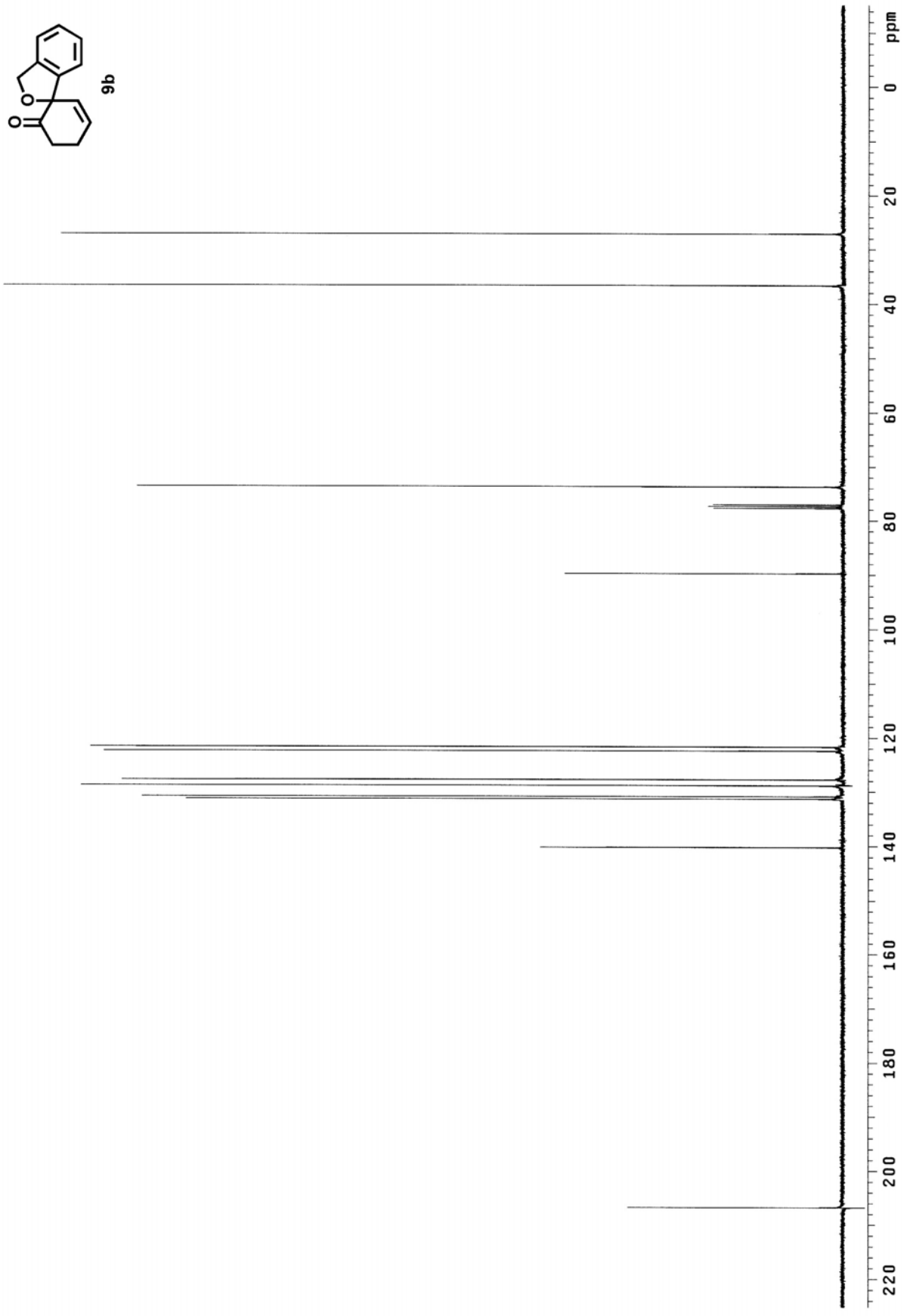
Chromatogram Plot

File: m:\spiro\compounds\as_c_compound_17_3.sms
Sample: as_C_Compound_17_3
Scan Range: 1 - 2221 Time Range: 0.00 - 20.98 min.
Sample Notes: ROUTINE

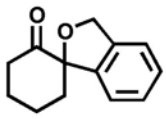
Operator: Operator
Date: 2005-09-19 23:30




9b
O
O
ppm
14
12
10
8
6
4
2
-0


9b
O
O
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 09 Mar 2007 15:44:50

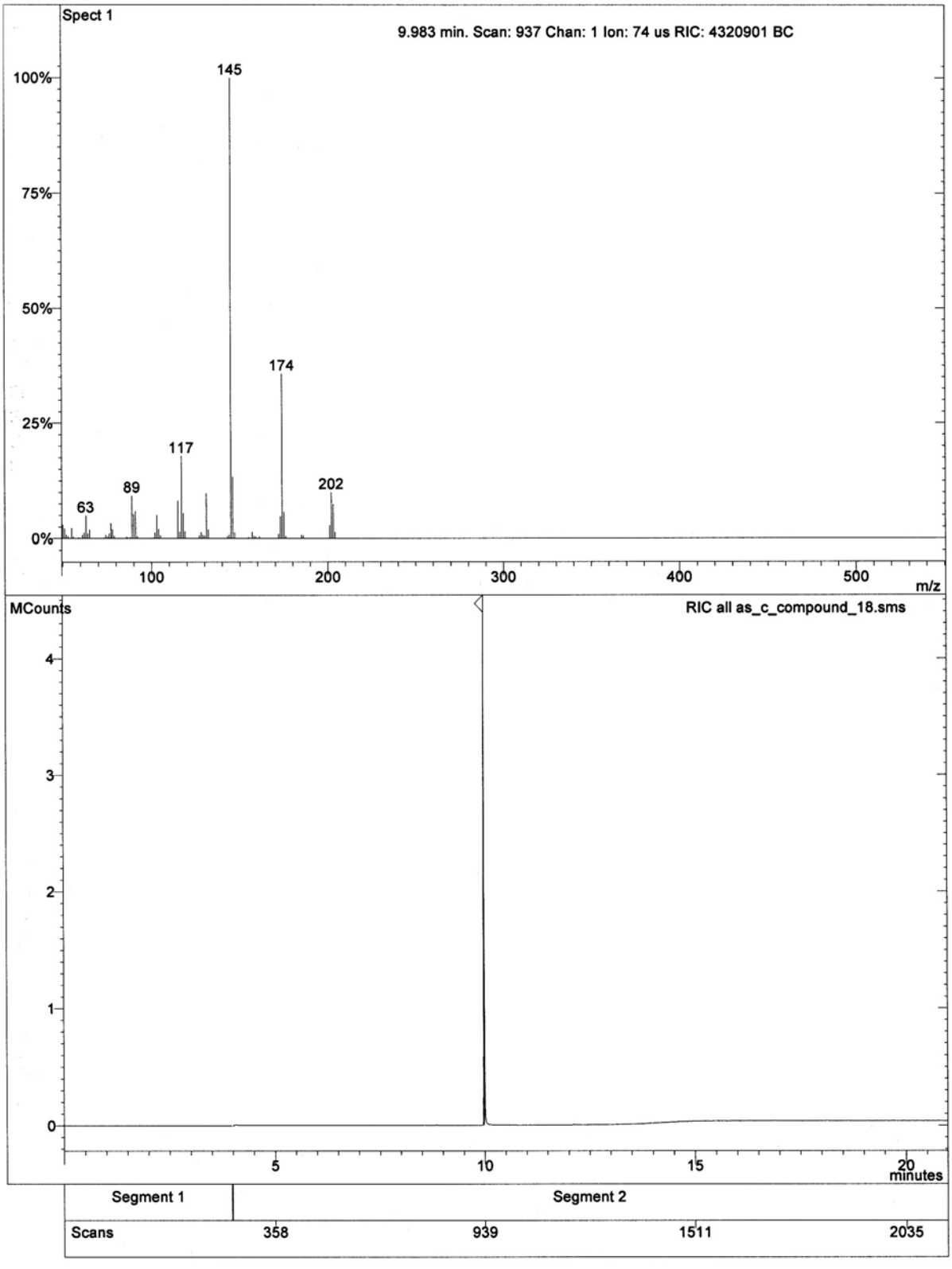


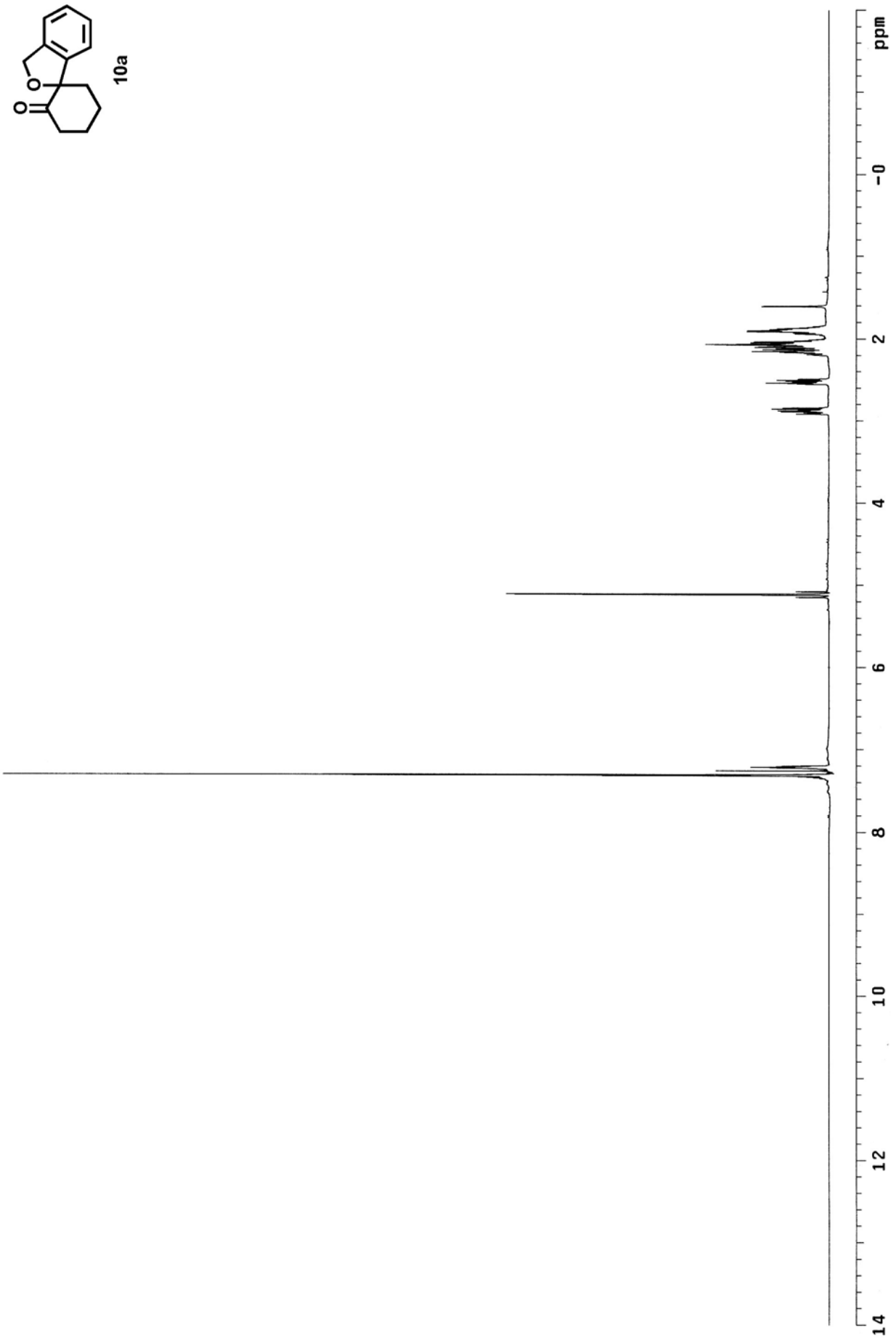
10a

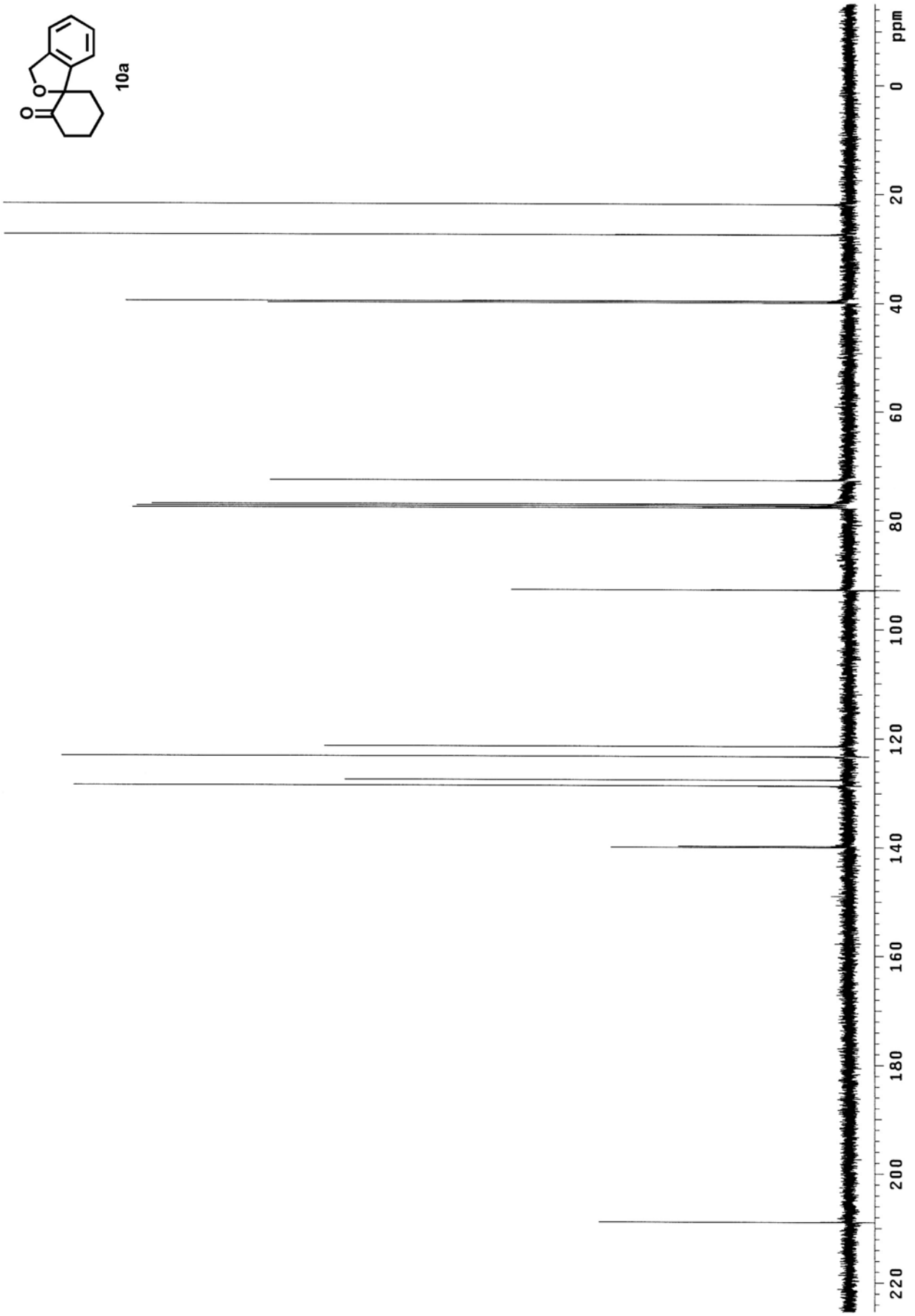
Chromatogram Plot

File: m:\spiro\compounds\as_c_compound_18.sms
Sample: as_Compound_18
Scan Range: 1 - 2138 Time Range: 0.00 - 20.99 min.
Sample Notes: today

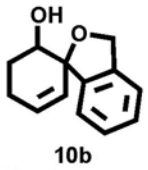
Operator: Operator
Date: 2005-07-07 11:35




O
O
10a
ppm
-0
2
4
6
8
10
12
14


10a
O
O
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 20 Mar 2007 14:14:25

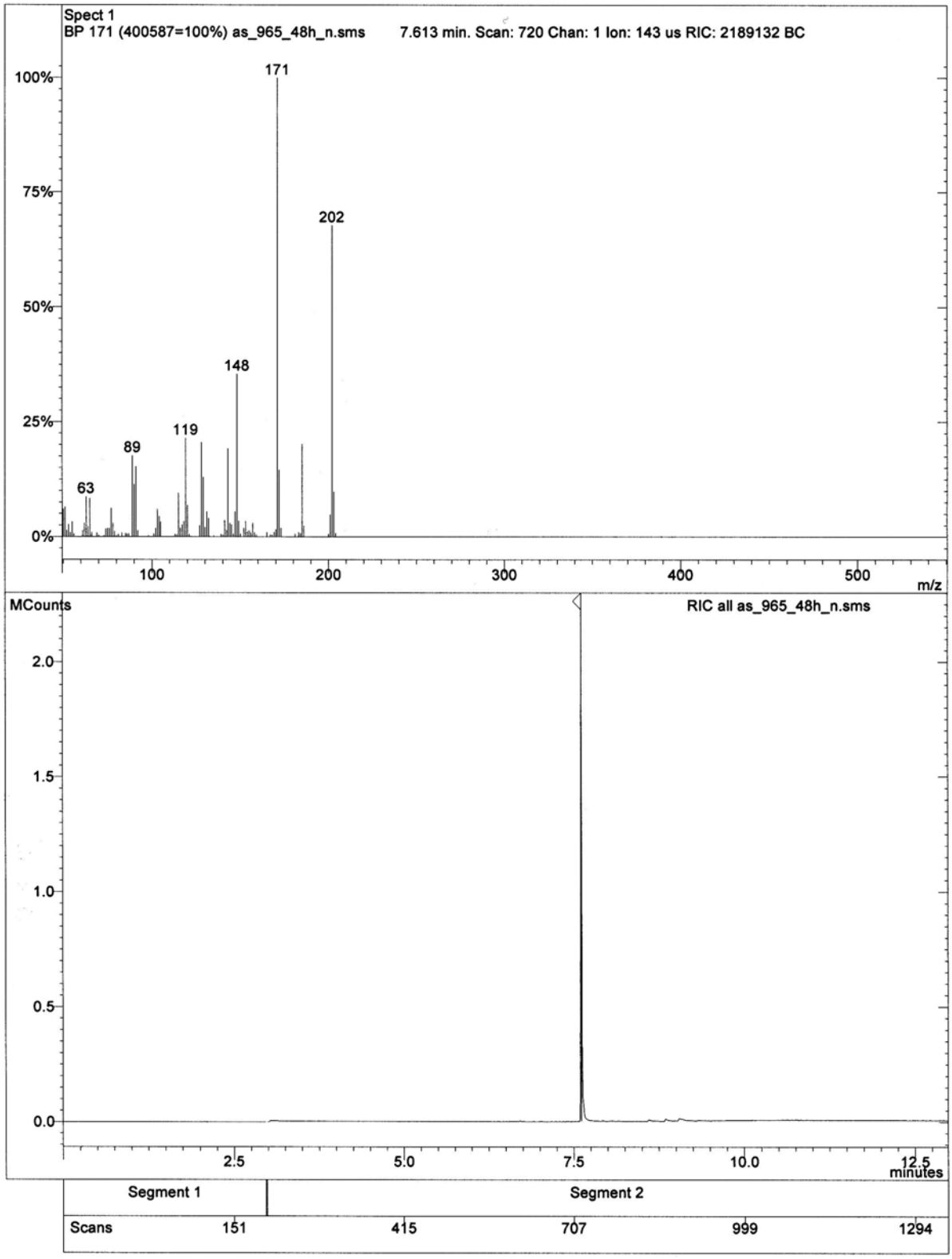


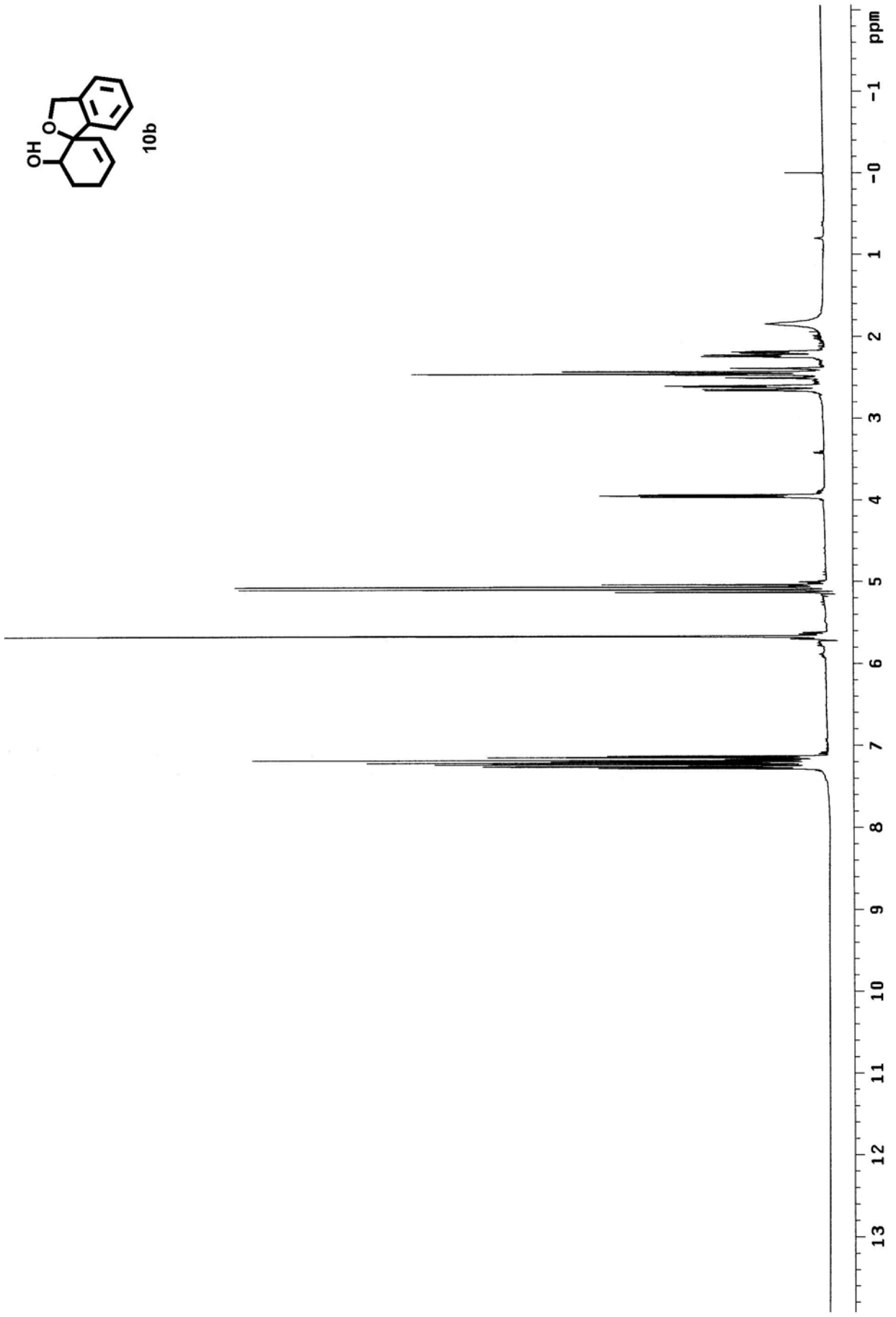
10b

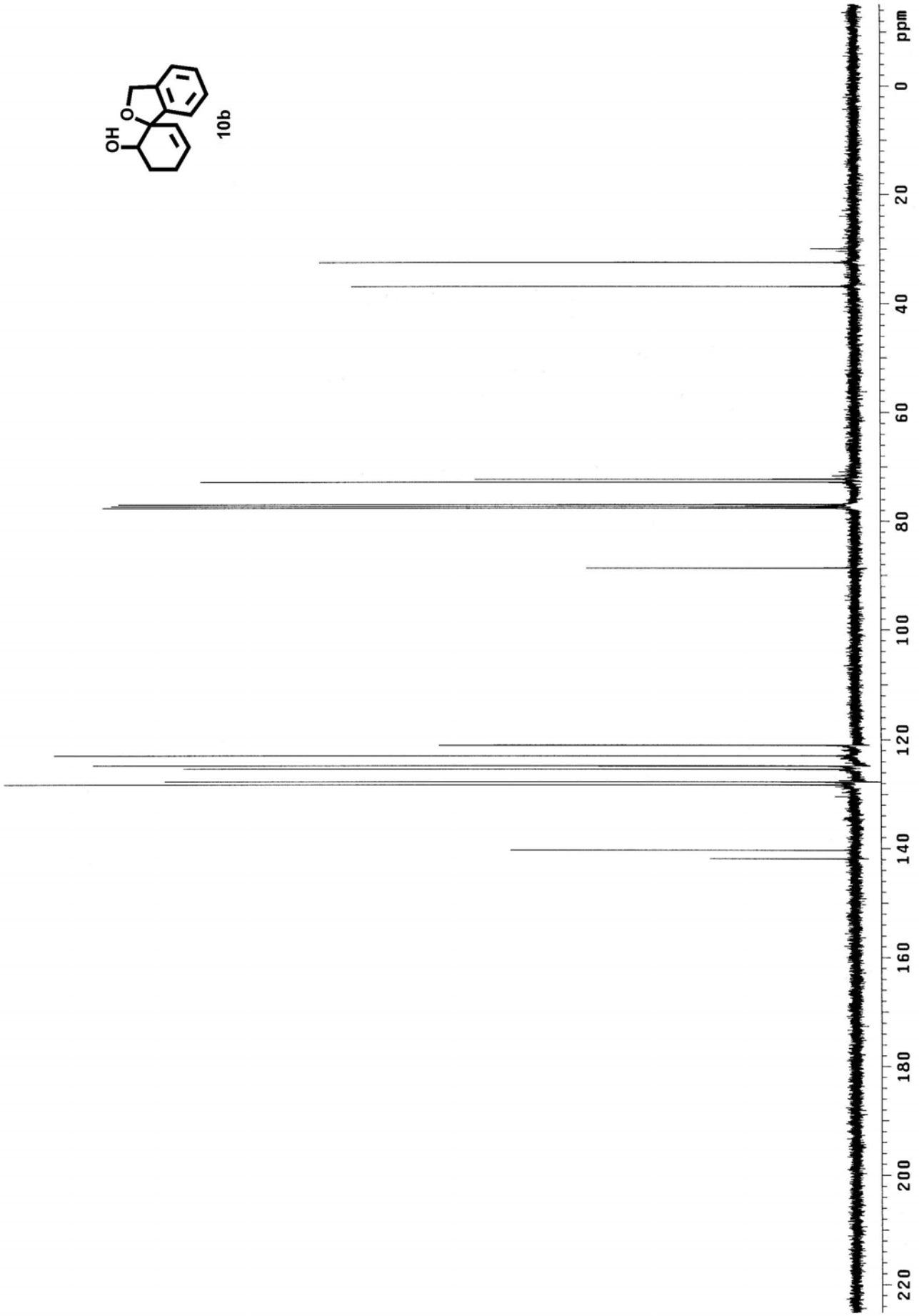
Chromatogram Plot

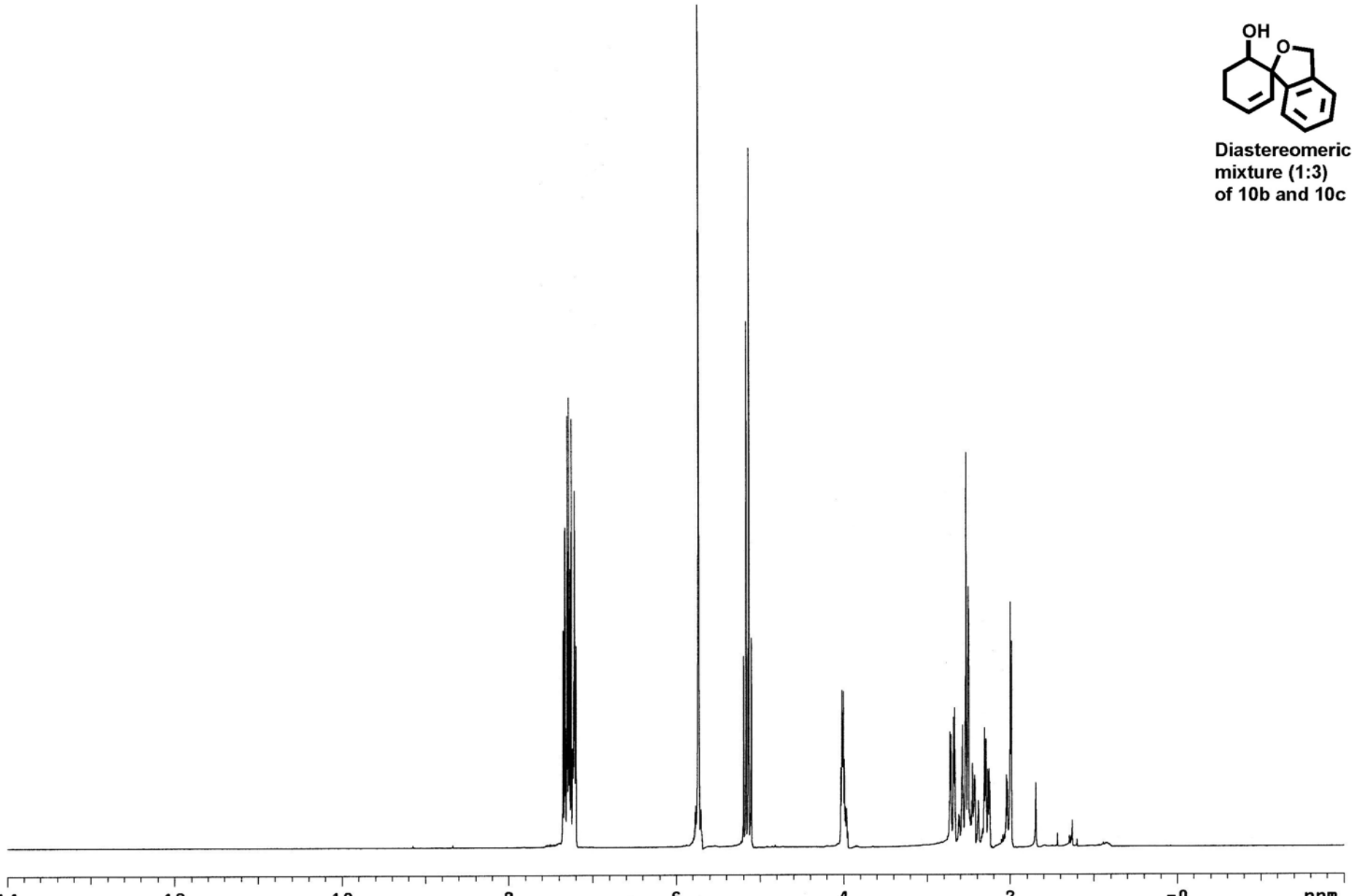
File: d:\users\andreas\spiro_gc\as_965_48h_n.sms
Sample: as_hydrolysis_48h_N
Scan Range: 1 - 1351 Time Range: 0.00 - 12.98 min.
Sample Notes: ROUTINE

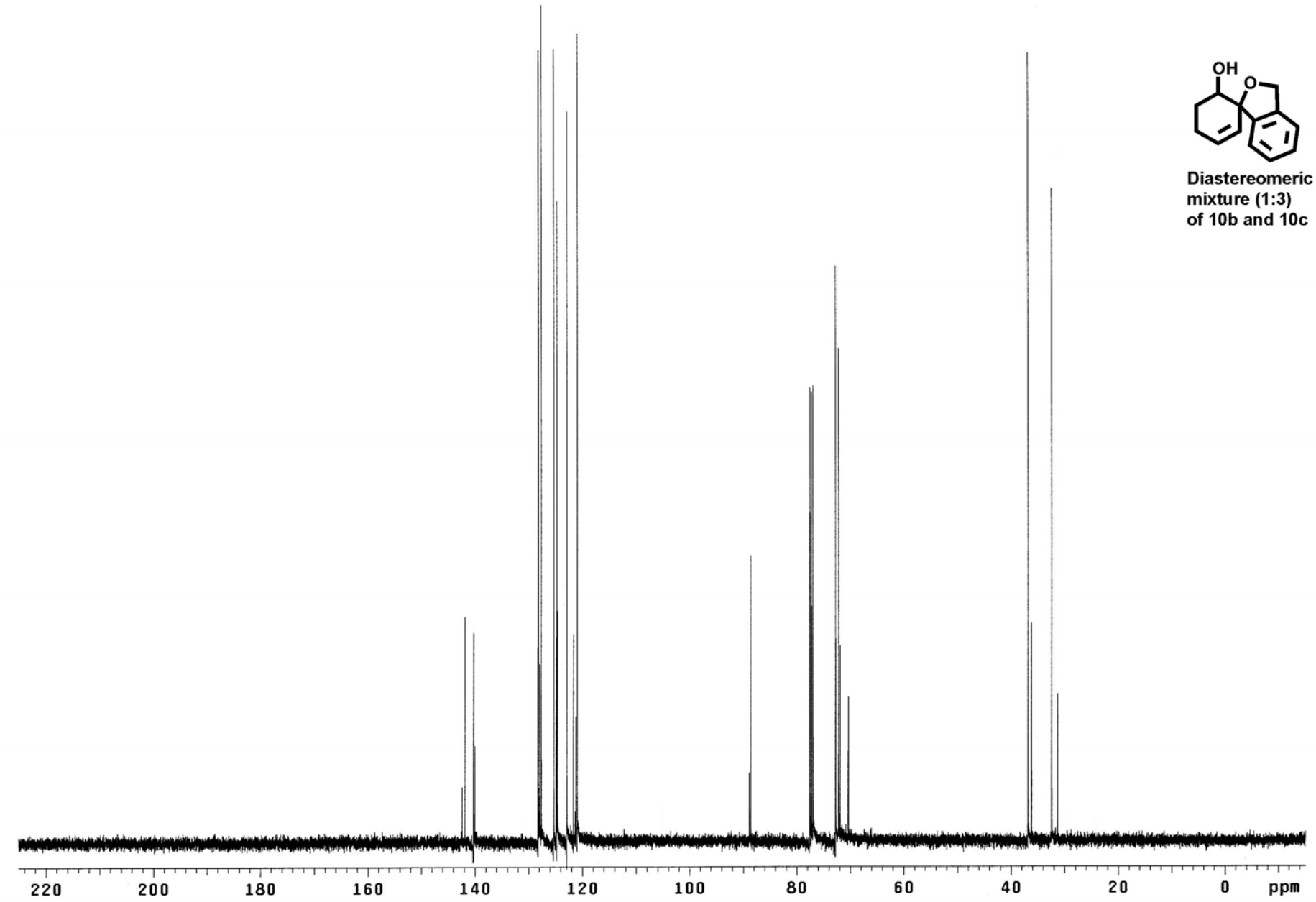
Operator: Operator
Date: 2007-02-13 01:50



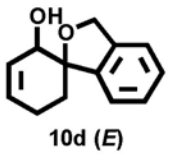

OH
O
10b
ppm
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1


OH
O
10b
ppm
0
20
40
60
80
100
120
140
160
180
200
220


OH
O
Diastereomeric
mixture (1:3)
of 10b and 10c
14
12
10
8
6
4
2
-0
ppm


OH
O
Diastereomeric
mixture (1:3)
of 10b and 10c
220
200
180
160
140
120
100
80
60
40
20
0
ppm

nt Date: 18 Mar 2007 00:38:10

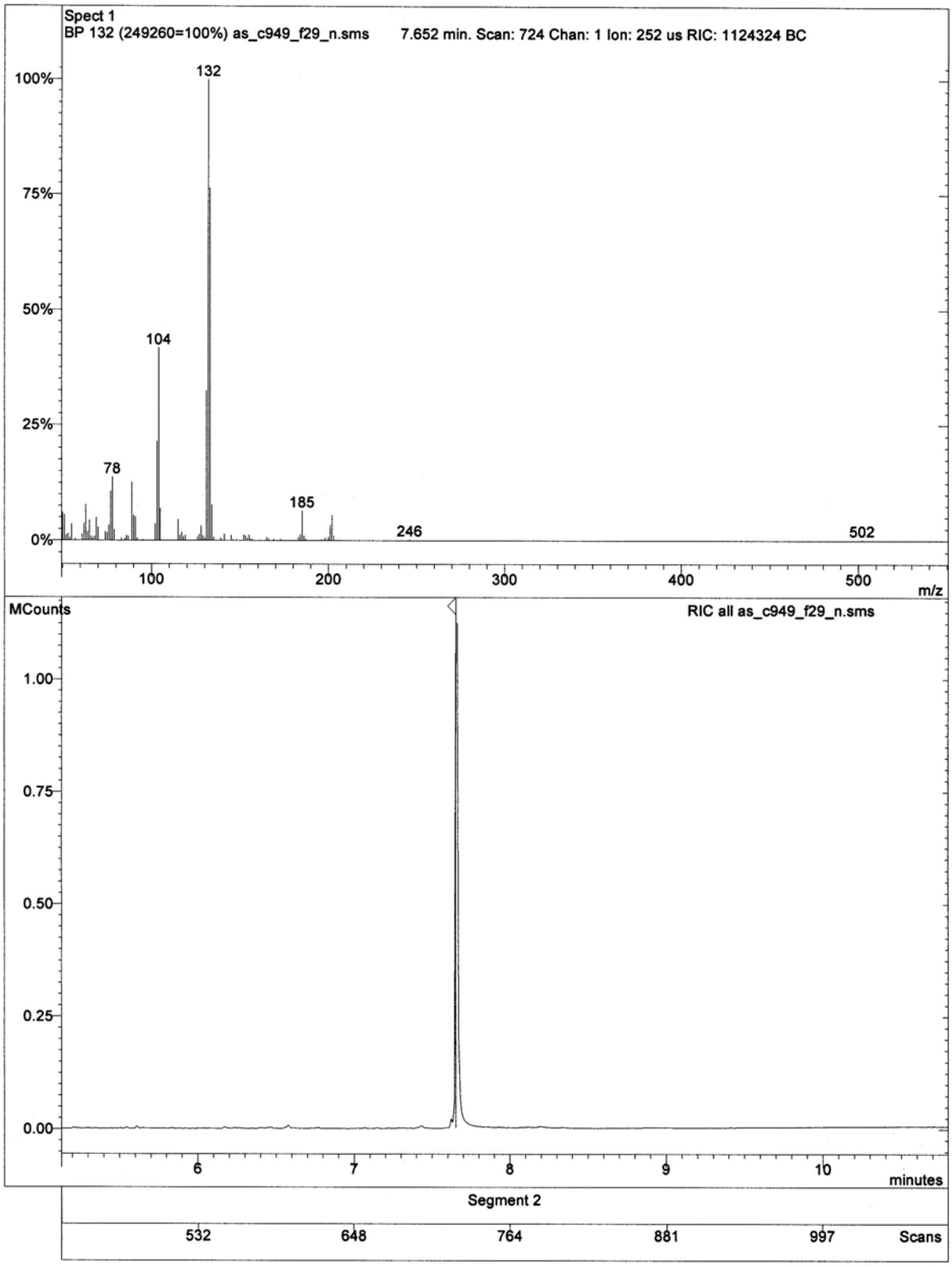


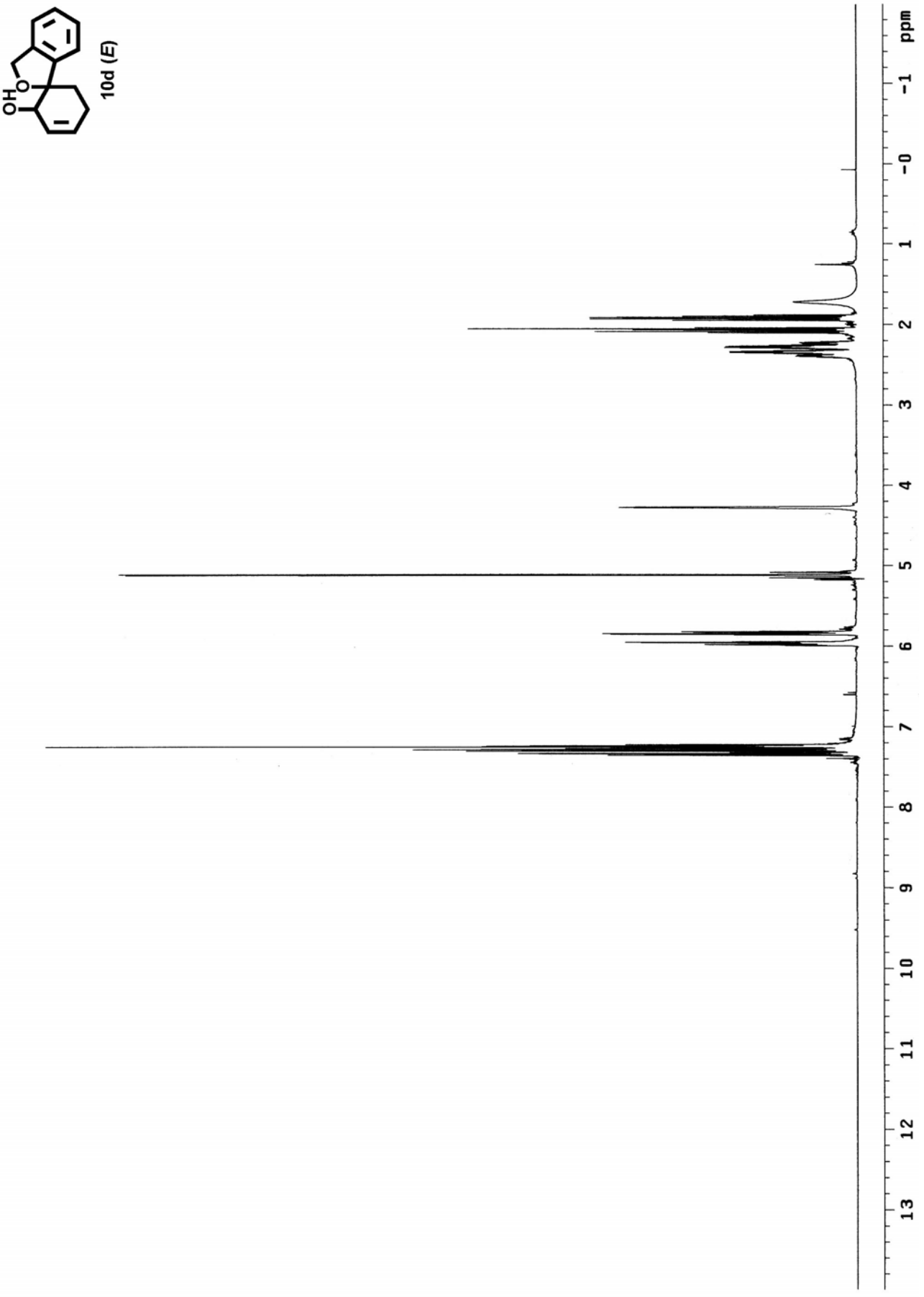
10d (*E*)

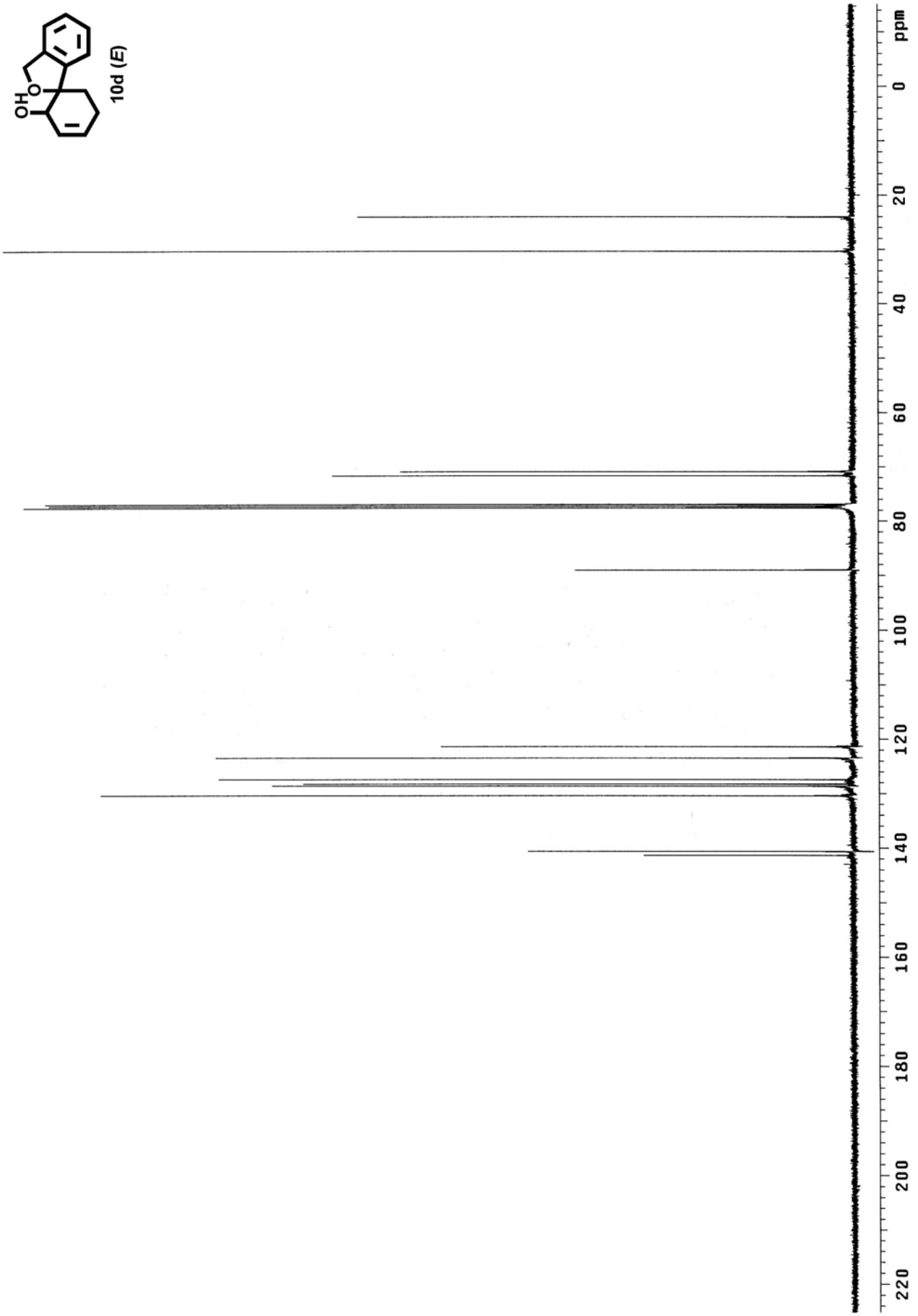
Chromatogram Plot

File: d:\users\andreas\spiro_gc\as_c949_f29_n.sms
Sample: as_C949_F29_N
Scan Range: 1 - 1347 Time Range: 0.00 - 12.98 min.
Sample Notes: ROUTINE

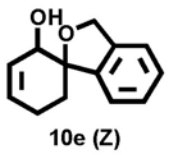
Operator: Operator
Date: 2007-01-13 03:32




OH
O
10d (E)
ppm
-1
-0
1
2
3
4
5
6
7
8
9
10
11
12
13


OH
O
10d (E)
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 18 Mar 2007 00:37:25

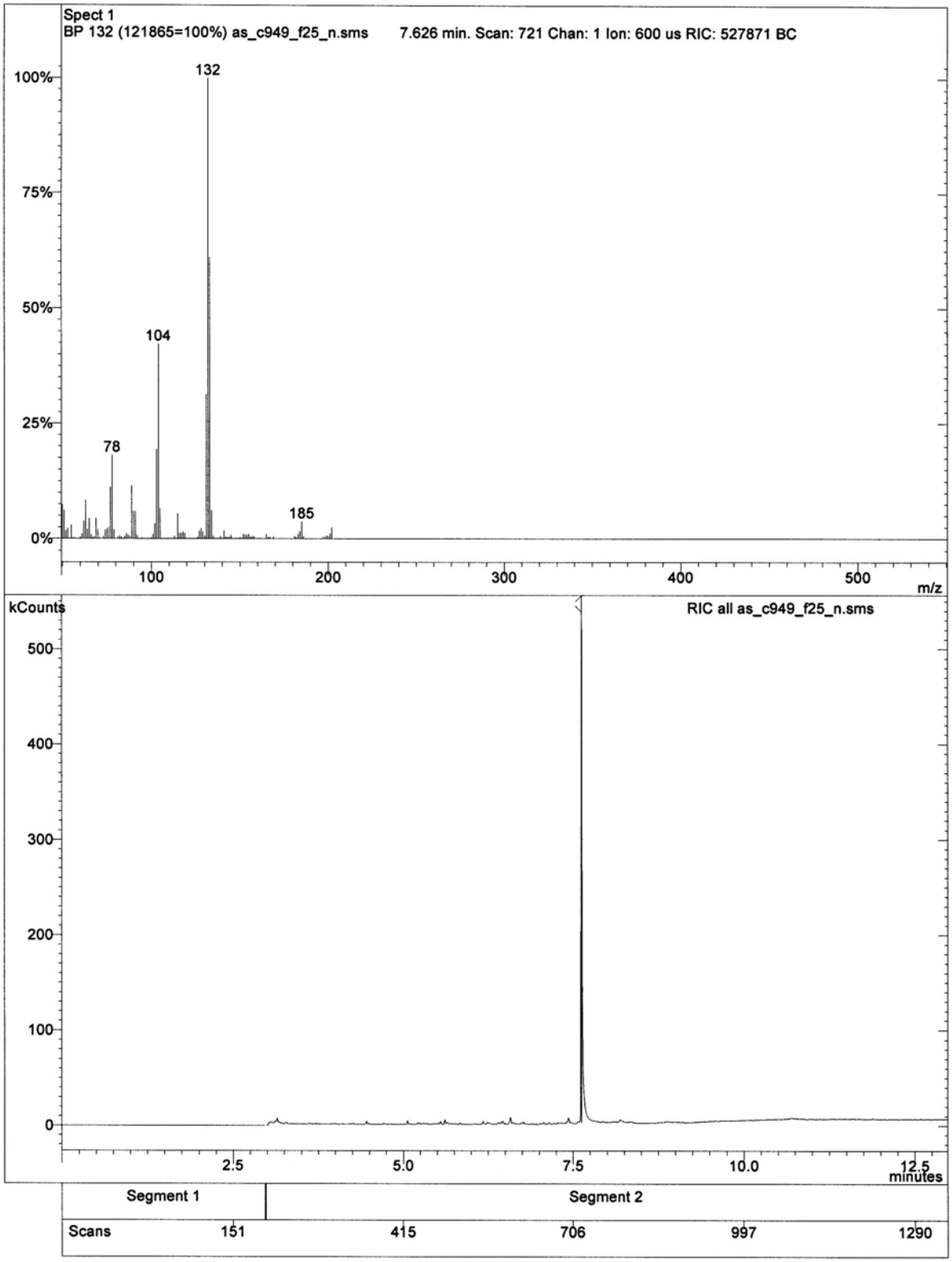


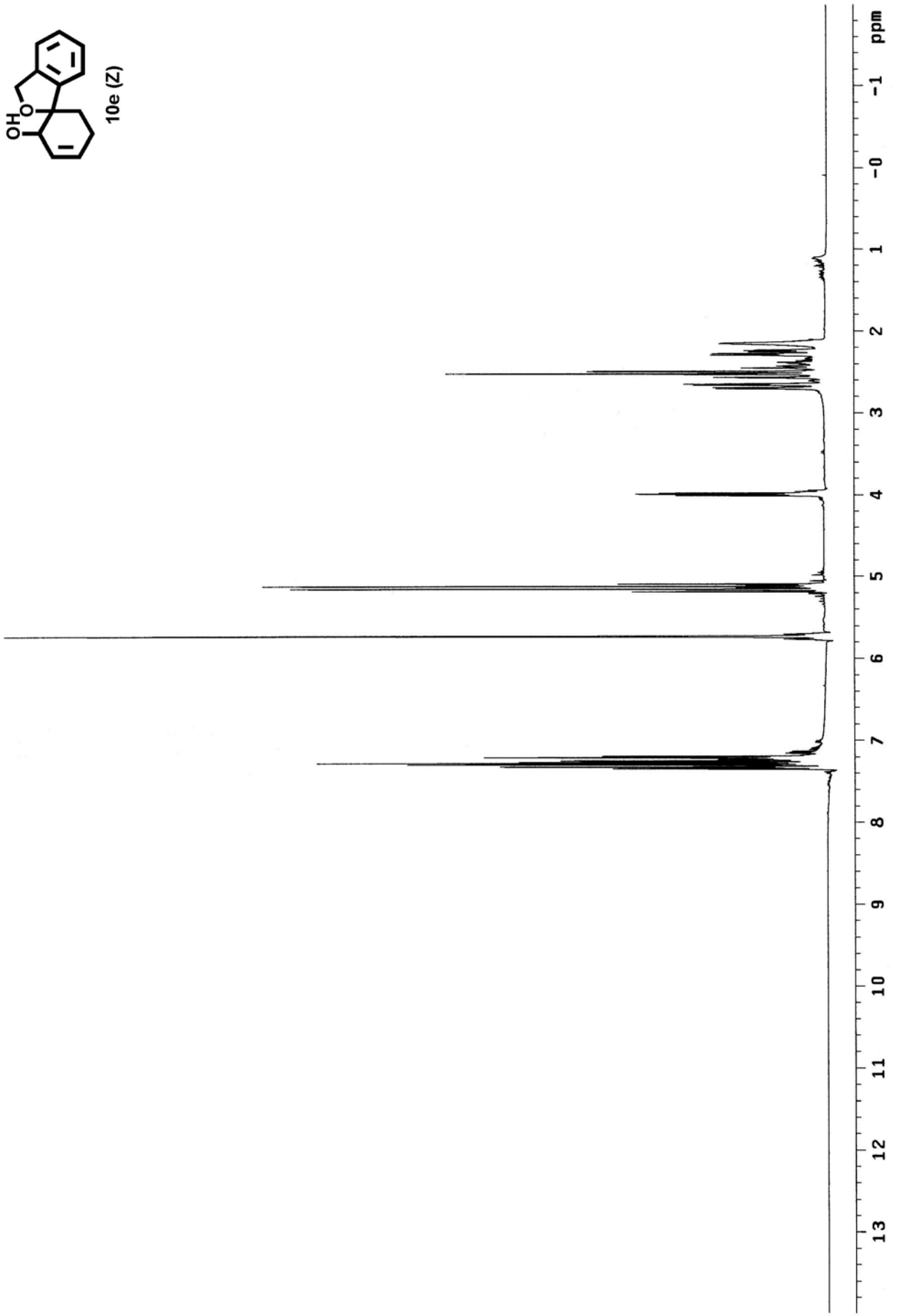
10e (Z)

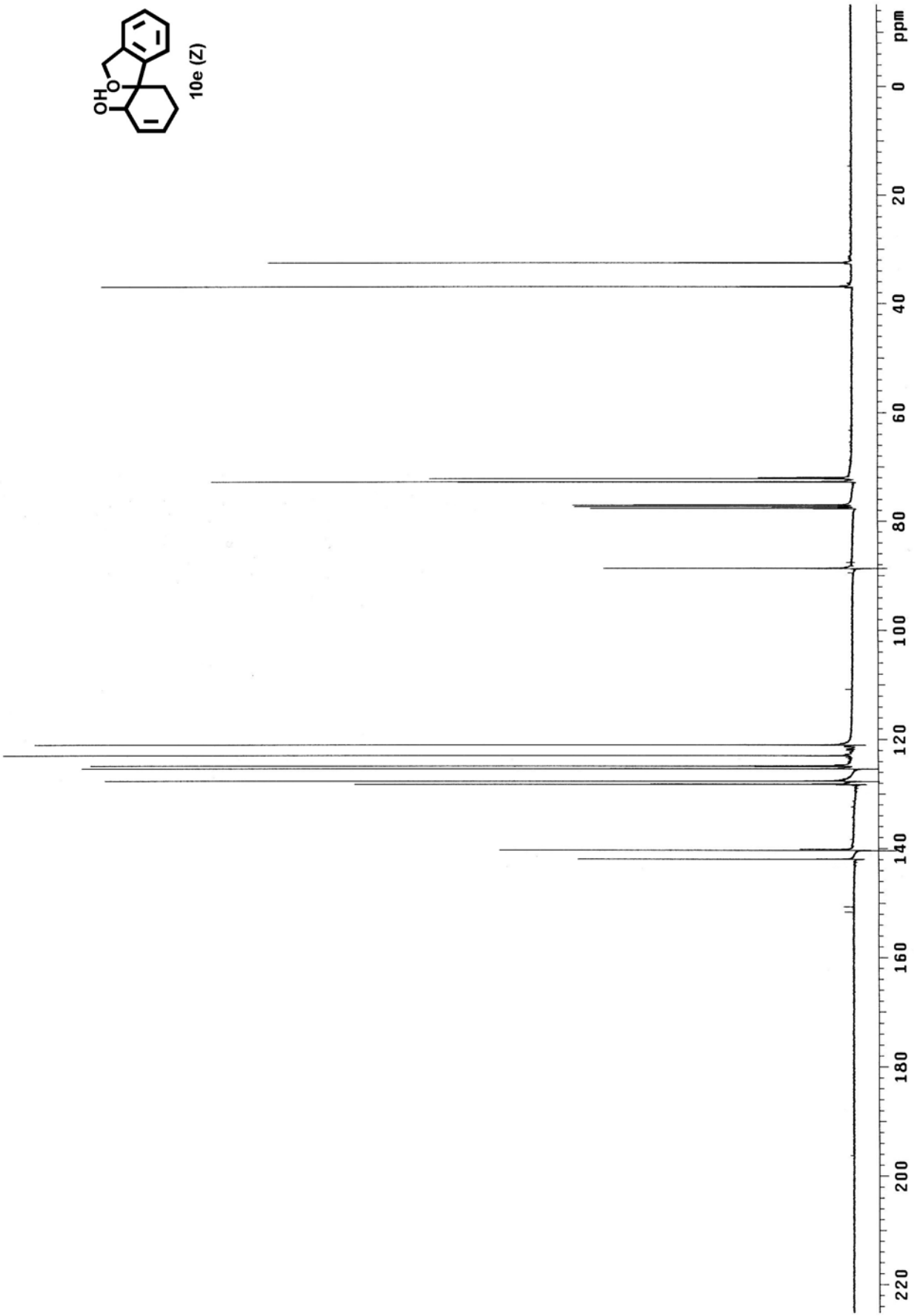
Chromatogram Plot

File: d:\users\andreas\spiro_gc\as_c949_f25_n.sms
Sample: as_C949_F25_N
Scan Range: 1 - 1346 Time Range: 0.00 - 12.98 min.
Sample Notes: ROUTINE

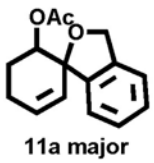
Operator: Operator
Date: 2007-01-13 04:36




OH
O
10e (Z)
ppm
-1
-0
1
2
3
4
5
6
7
8
9
10
11
12
13


10e (Z)
OH
O
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 20 Mar 2007 13:10:30

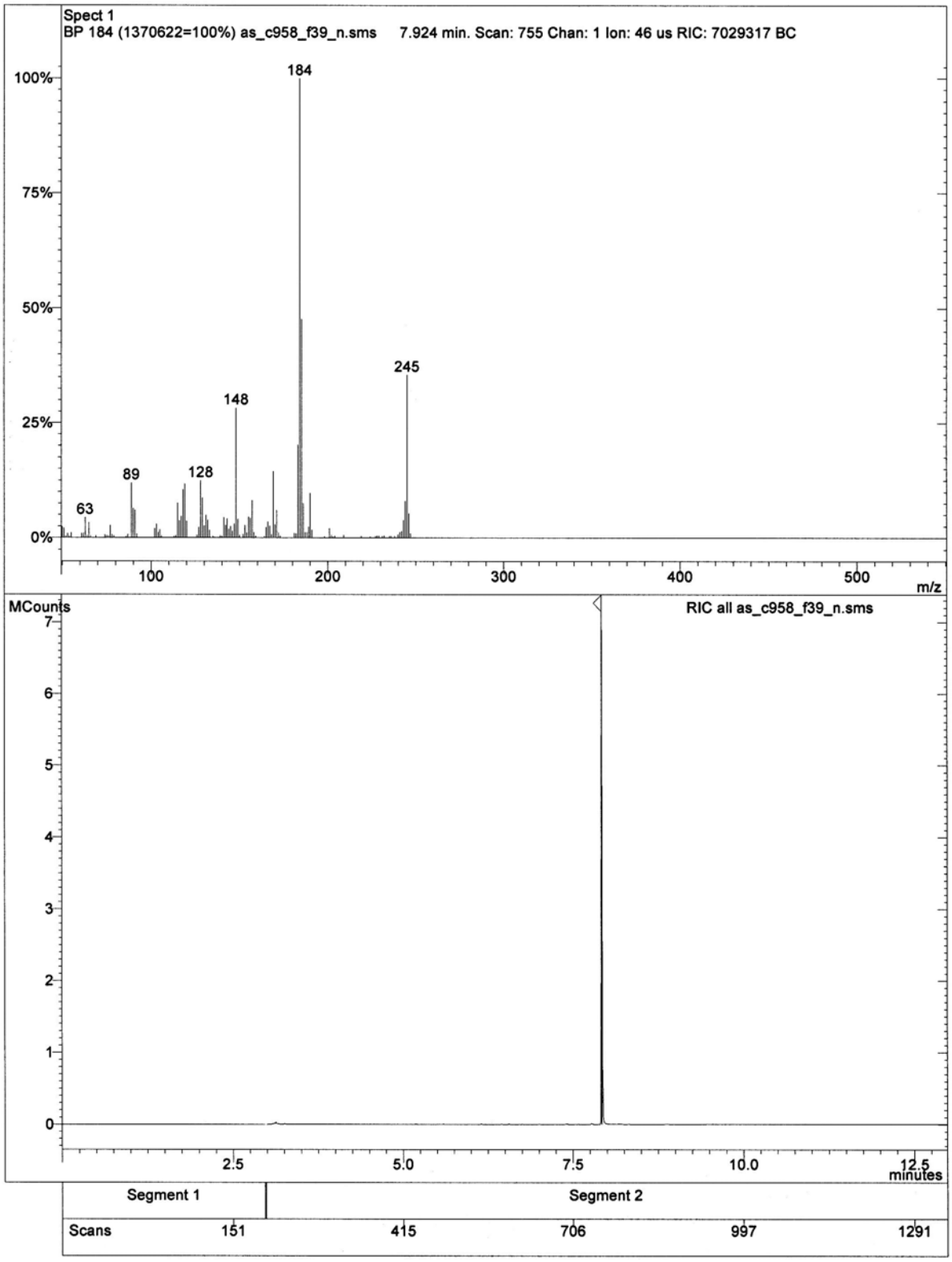


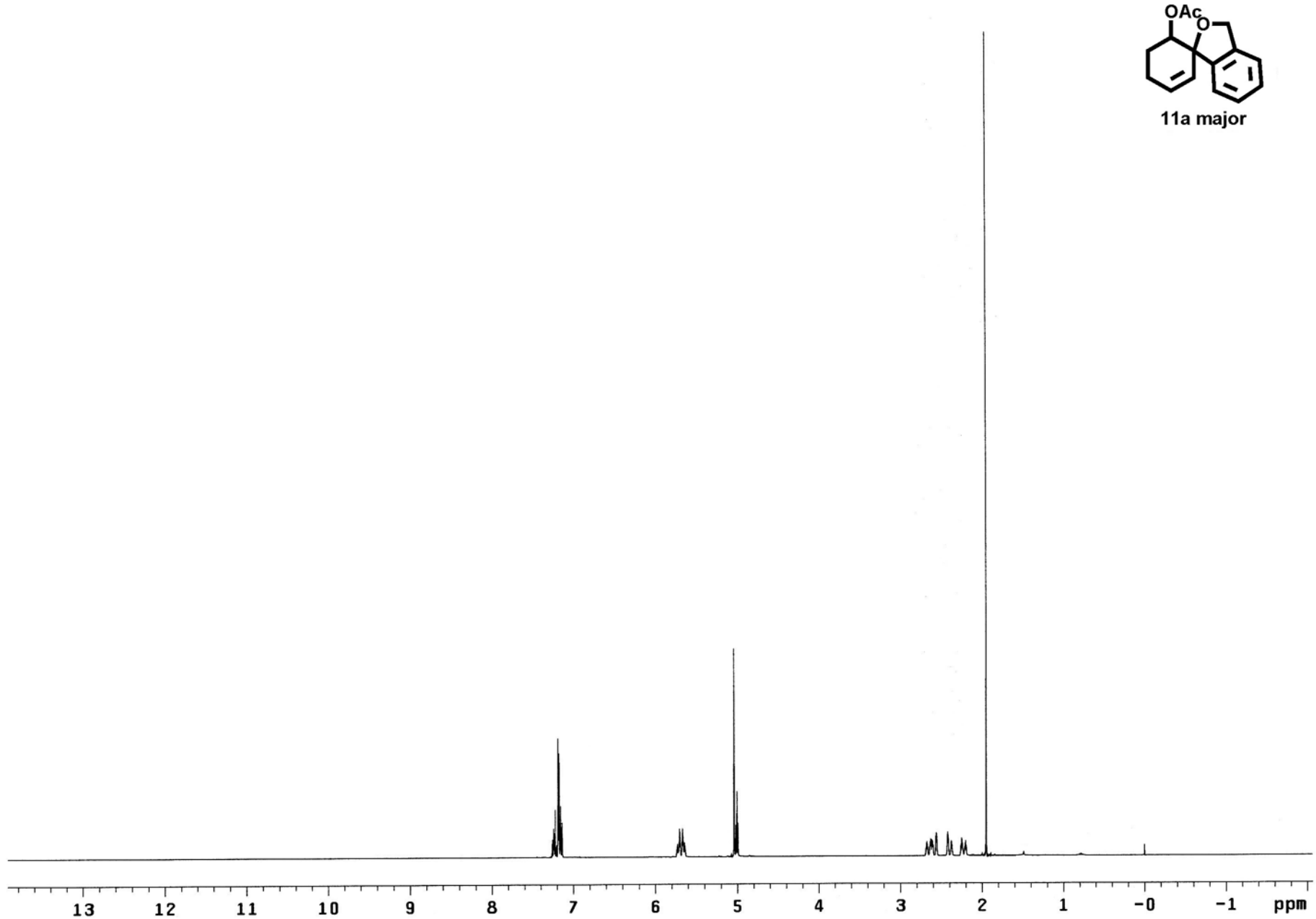
11a major

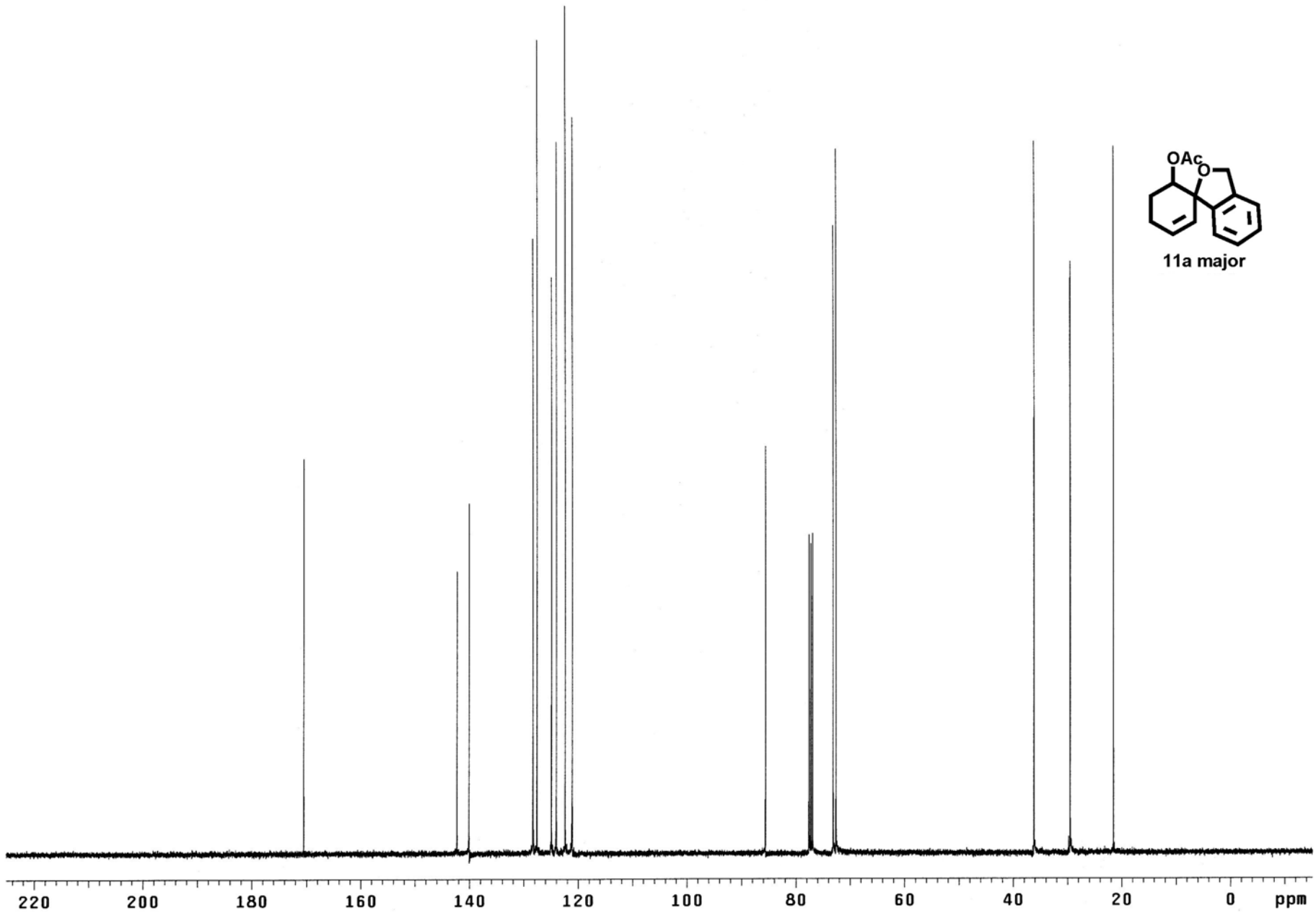
Chromatogram Plot

File: d:\users\andreas\spiro_gc\as_c958_f39_n.sms
Sample: as_C958_F39_N
Scan Range: 1 - 1348 Time Range: 0.00 - 12.98 min.
Sample Notes: today

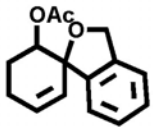
Operator: Operator
Date: 2007-02-05 14:51




OAc
O
11a major
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm


OAc
O
11a major
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Print Date: 20 Mar 2007 13:09:47

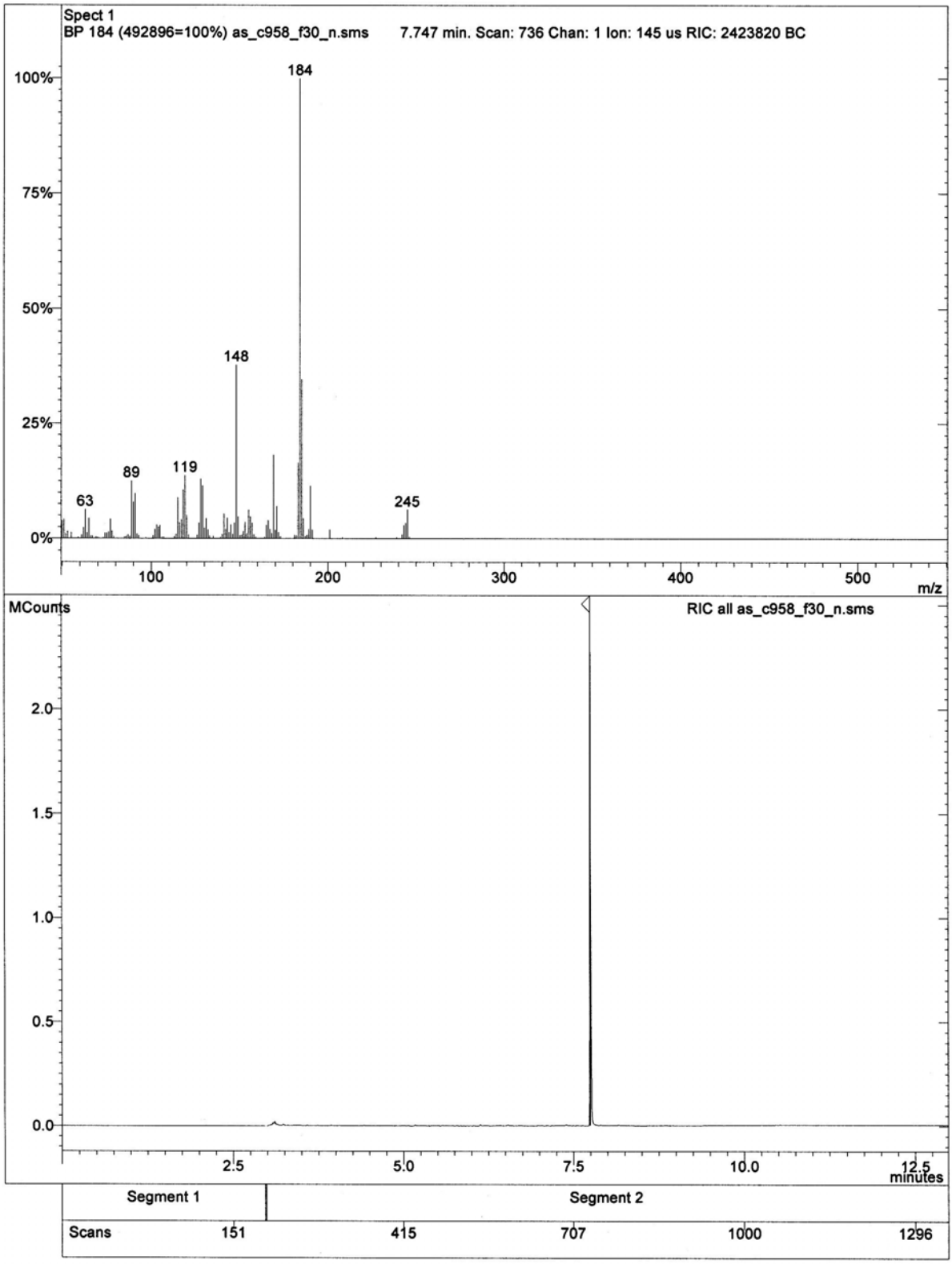


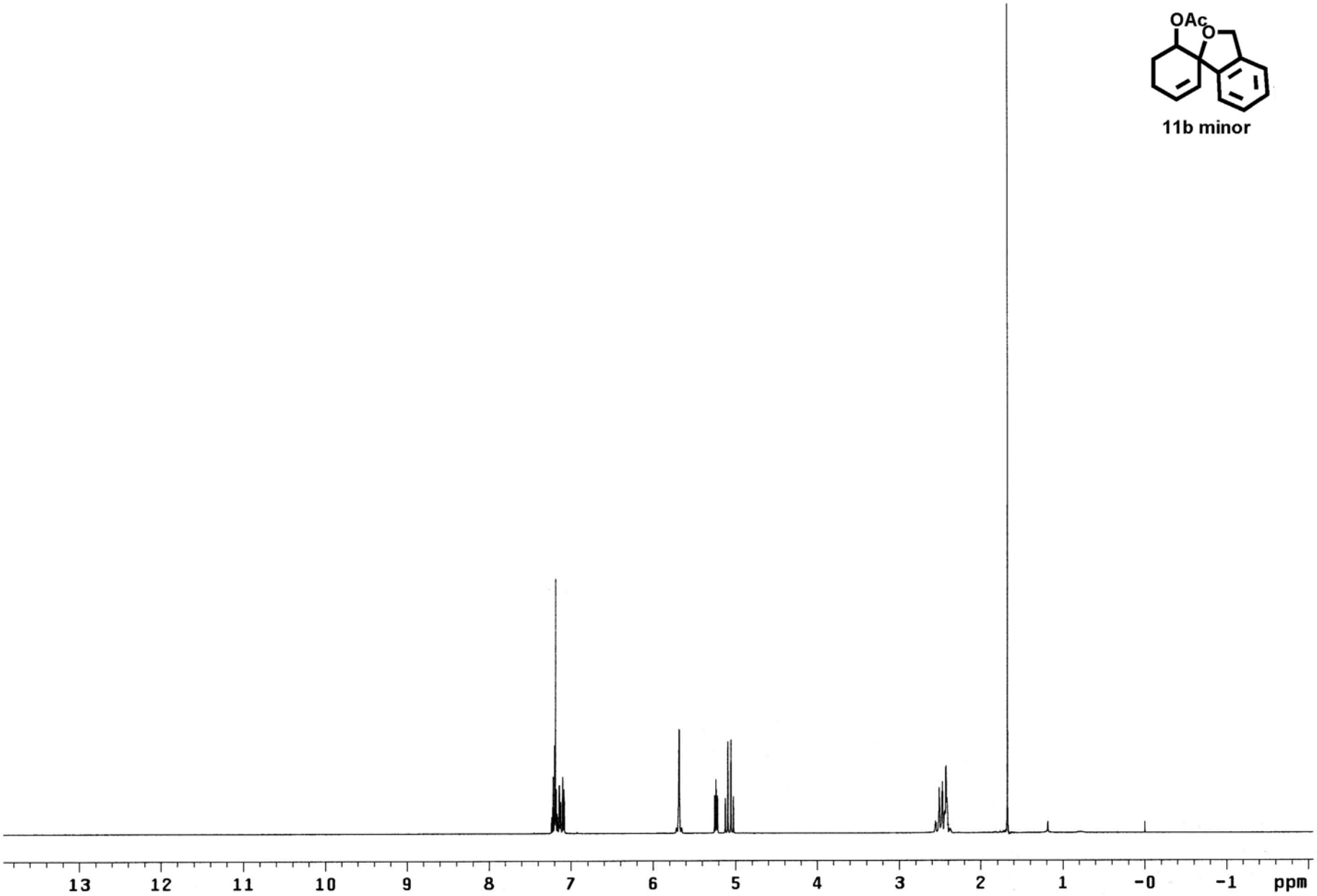
11b minor

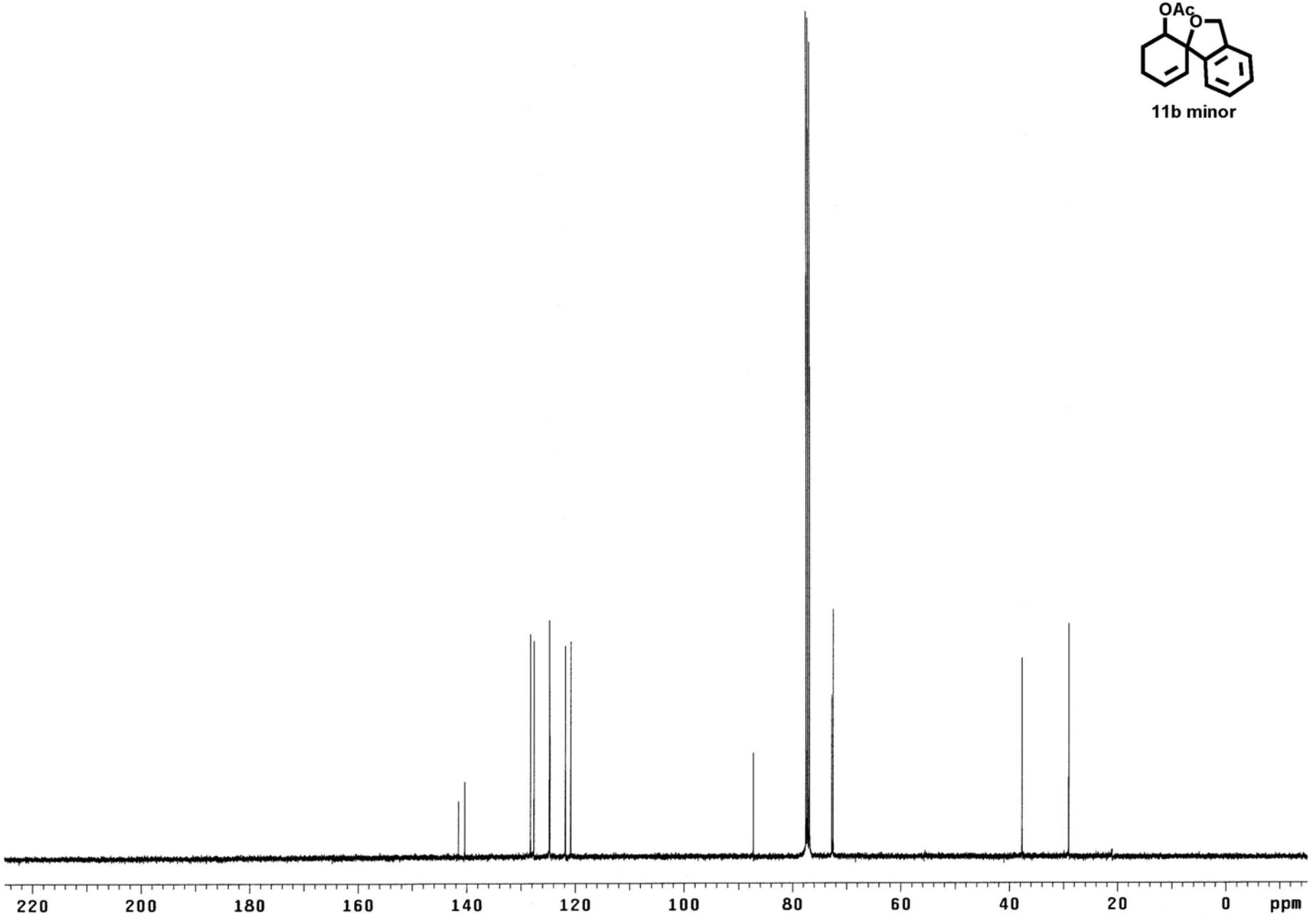
Chromatogram Plot

File: d:\users\andreas\spiro_gc\as_c958_f30_n.sms
Sample: as_C958_F30_N
Scan Range: 1 - 1353 Time Range: 0.00 - 12.98 min.
Sample Notes: ROUTINE

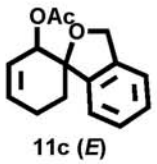
Operator: Operator
Date: 2007-02-03 21:21




OAc
O
11b minor
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm


OAc
O
11b minor
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Print Date: 10 Apr 2007 21:46:56

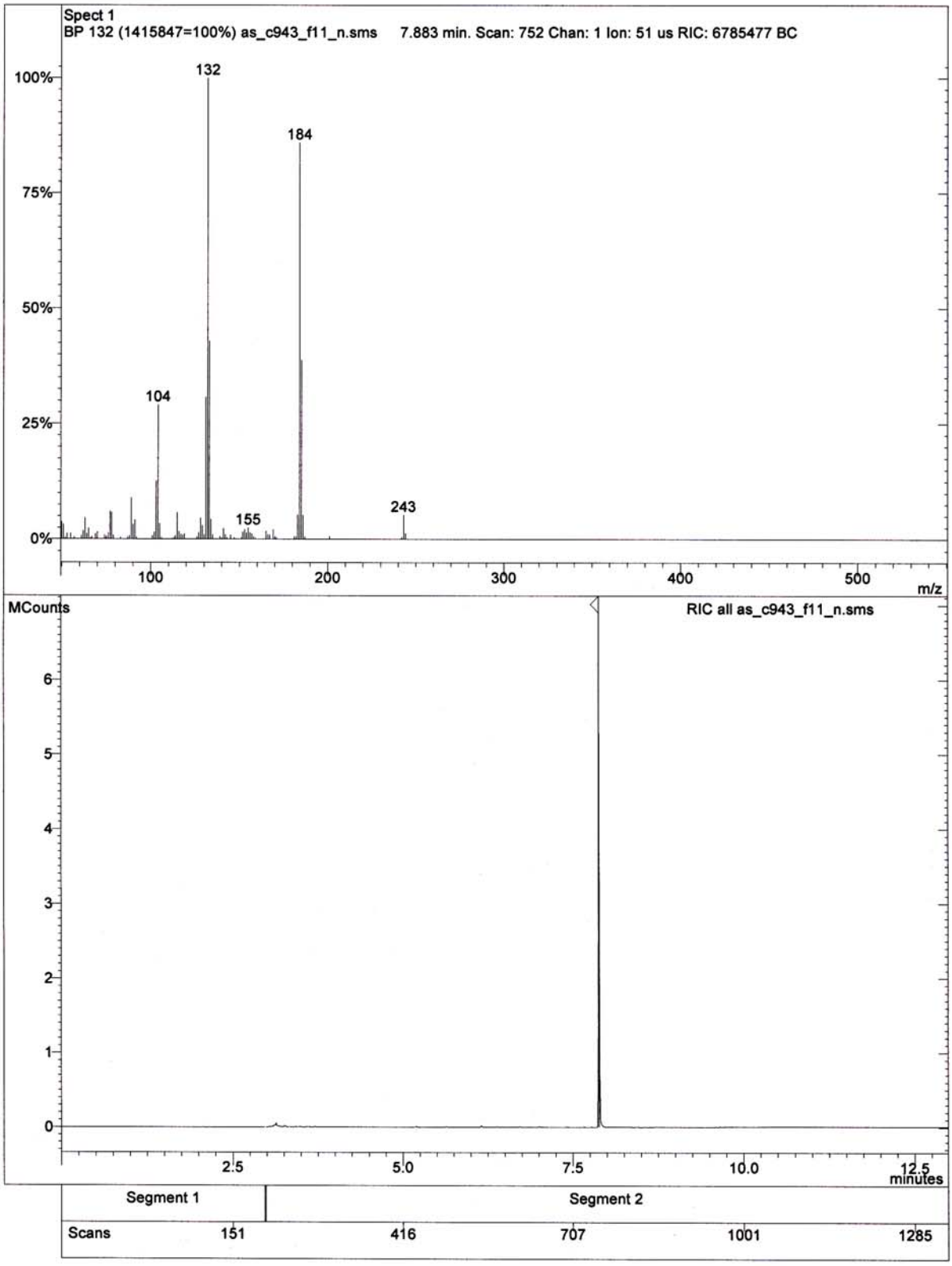


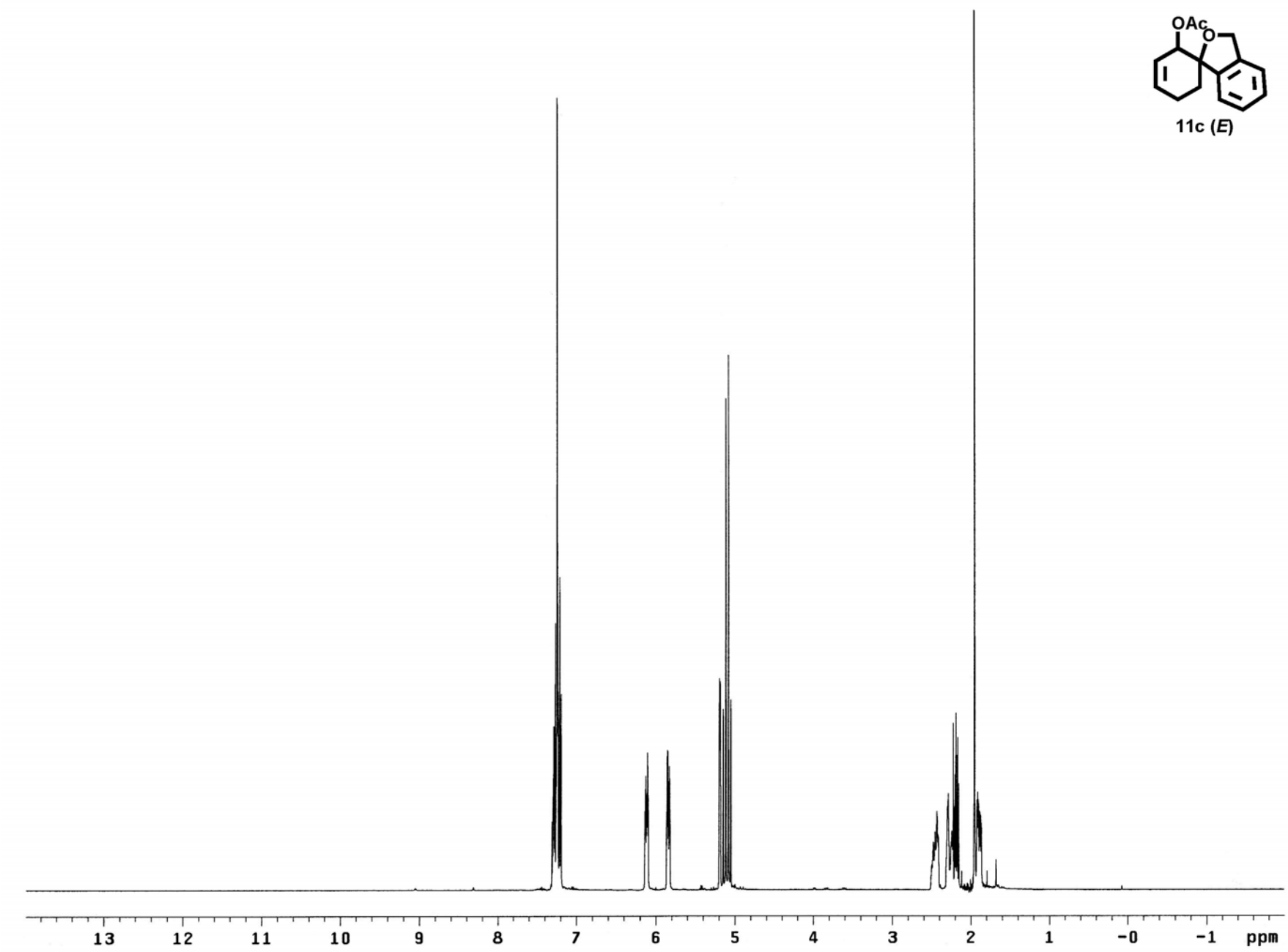
11c (*E*)

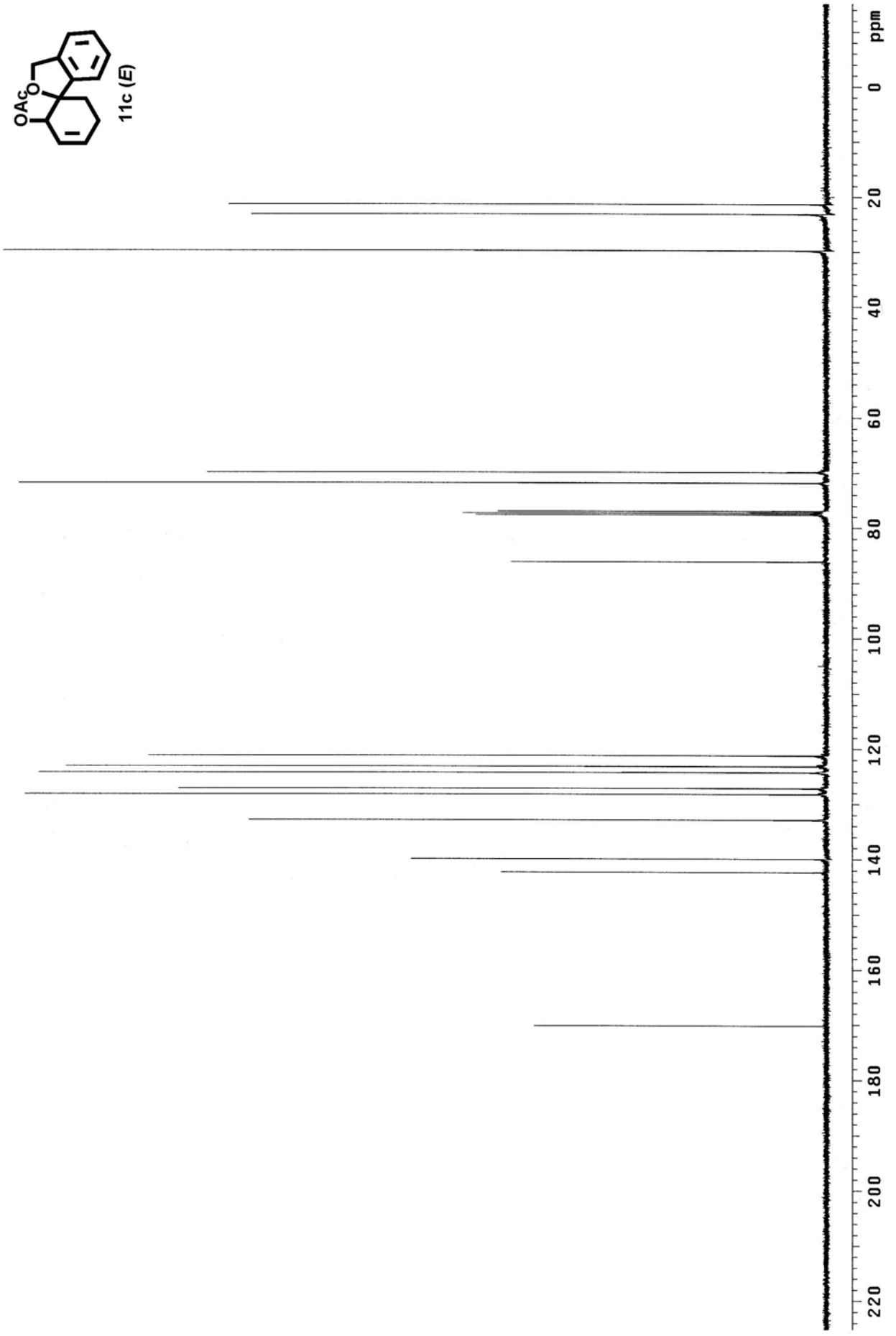
Chromatogram Plot

File: m:\spiro_gc\as_c943_f11_n.sms
Sample: as_C943_F11_N
Scan Range: 1 - 1341 Time Range: 0.00 - 12.99 min.
Sample Notes: ROUTINE

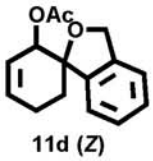
Operator: Operator
Date: 2006-12-31 00:45




OAc
O
11c (E)
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm


OAc
O
11c (E)
ppm
0
20
40
60
80
100
120
140
160
180
200
220

Print Date: 10 Apr 2007 21:45:44

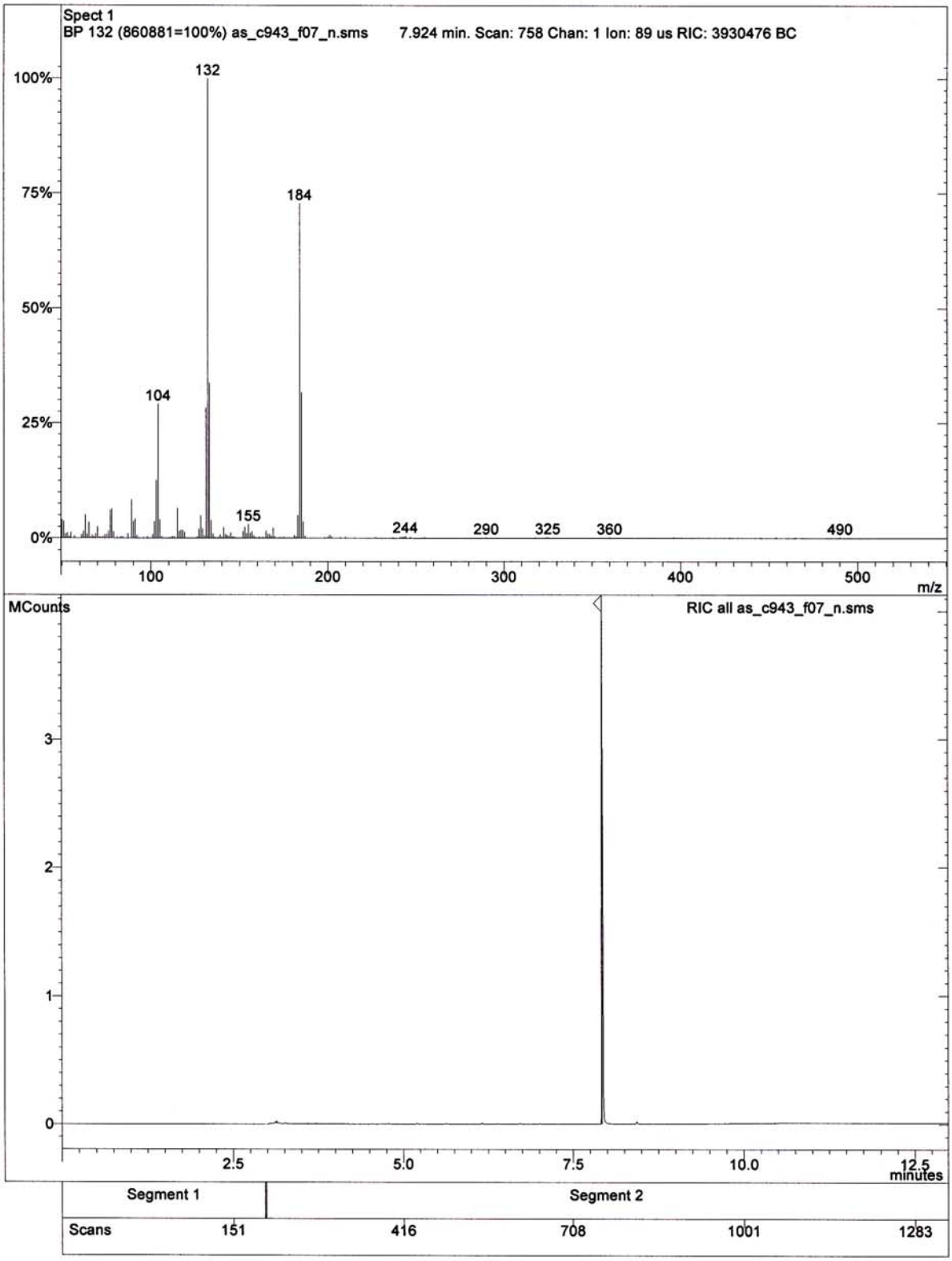


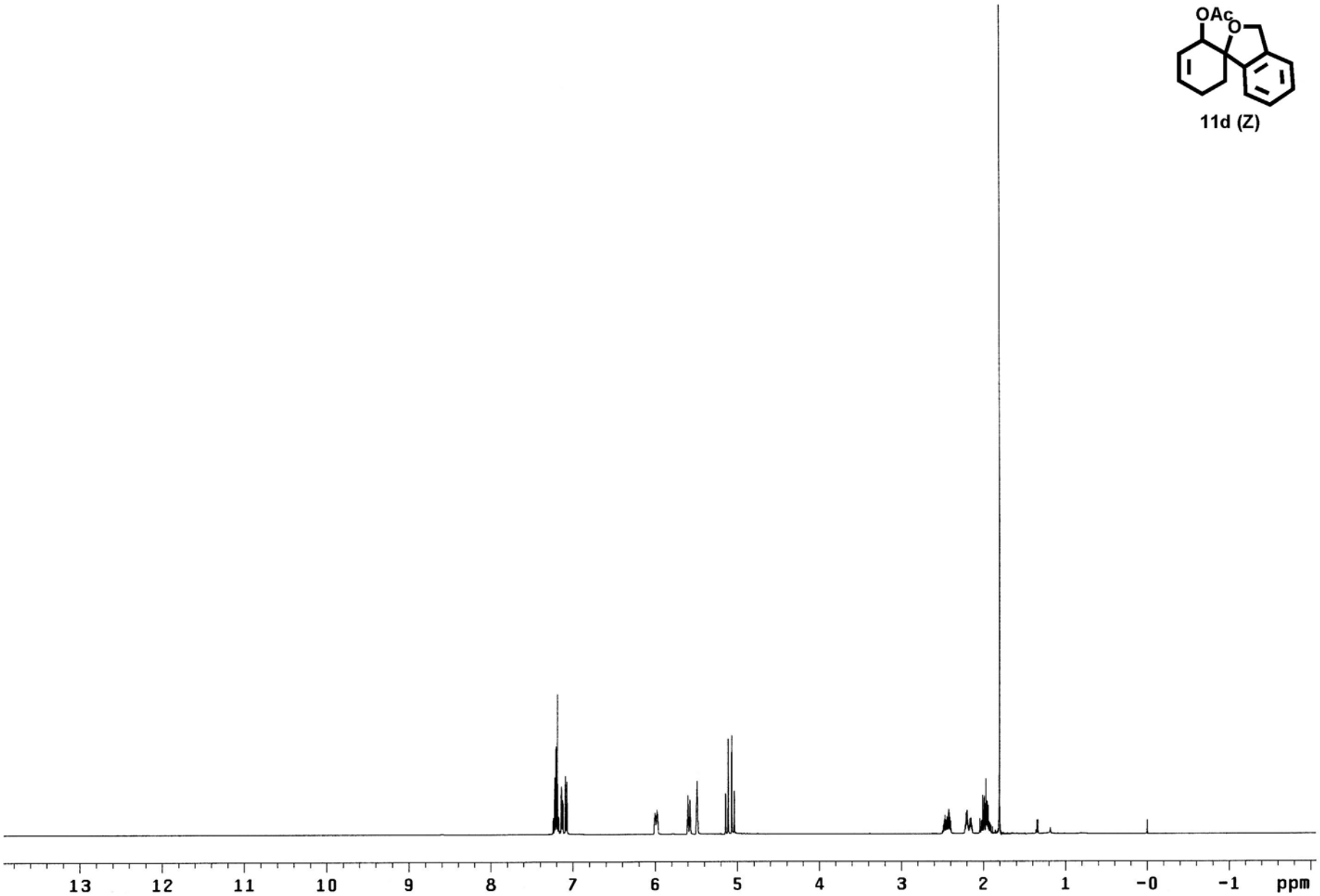
11d (Z)

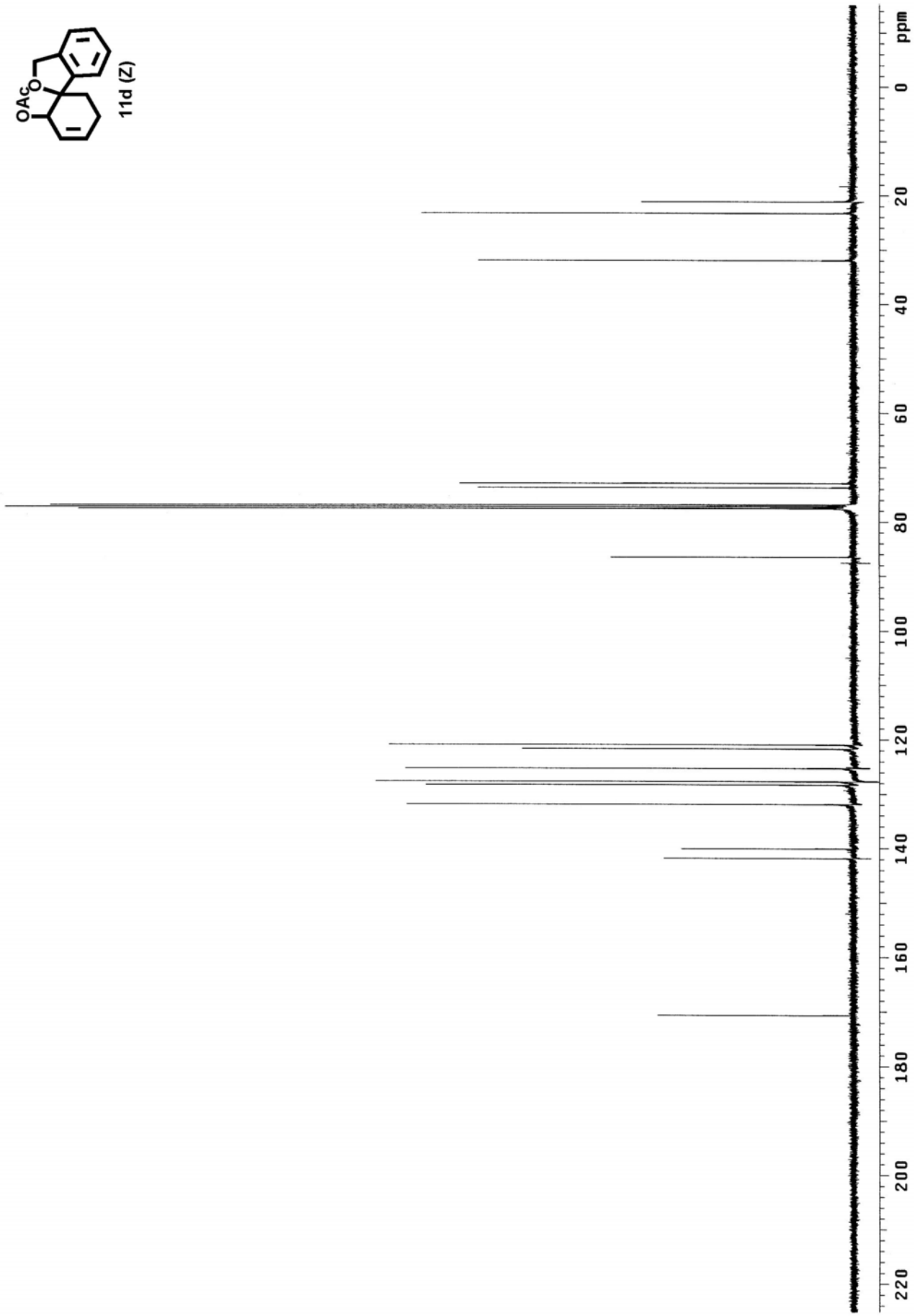
Chromatogram Plot

File: m:\spiro_gc\as_c943_f07_n.sms
Sample: as_C943_F07_N
Scan Range: 1 - 1337 Time Range: 0.00 - 12.99 min.
Sample Notes: ROUTINE

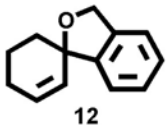
Operator: Operator
Date: 2006-12-30 22:32




OAc
O
11d (Z)
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm


OAc
O
11d (Z)
ppm
0
20
40
60
80
100
120
140
160
180
200
220

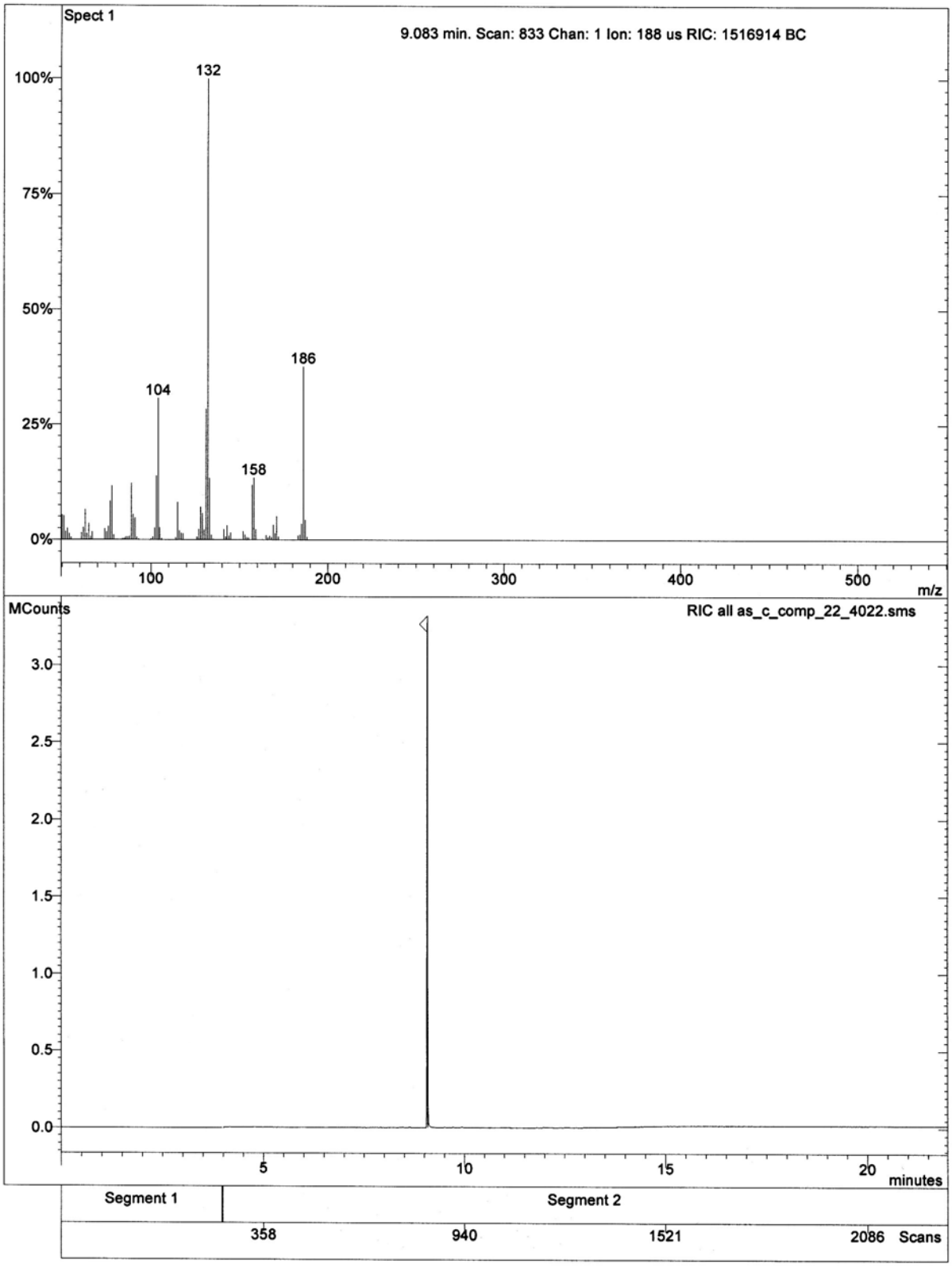
Print Date: 17 Mar 2007 23:54:41

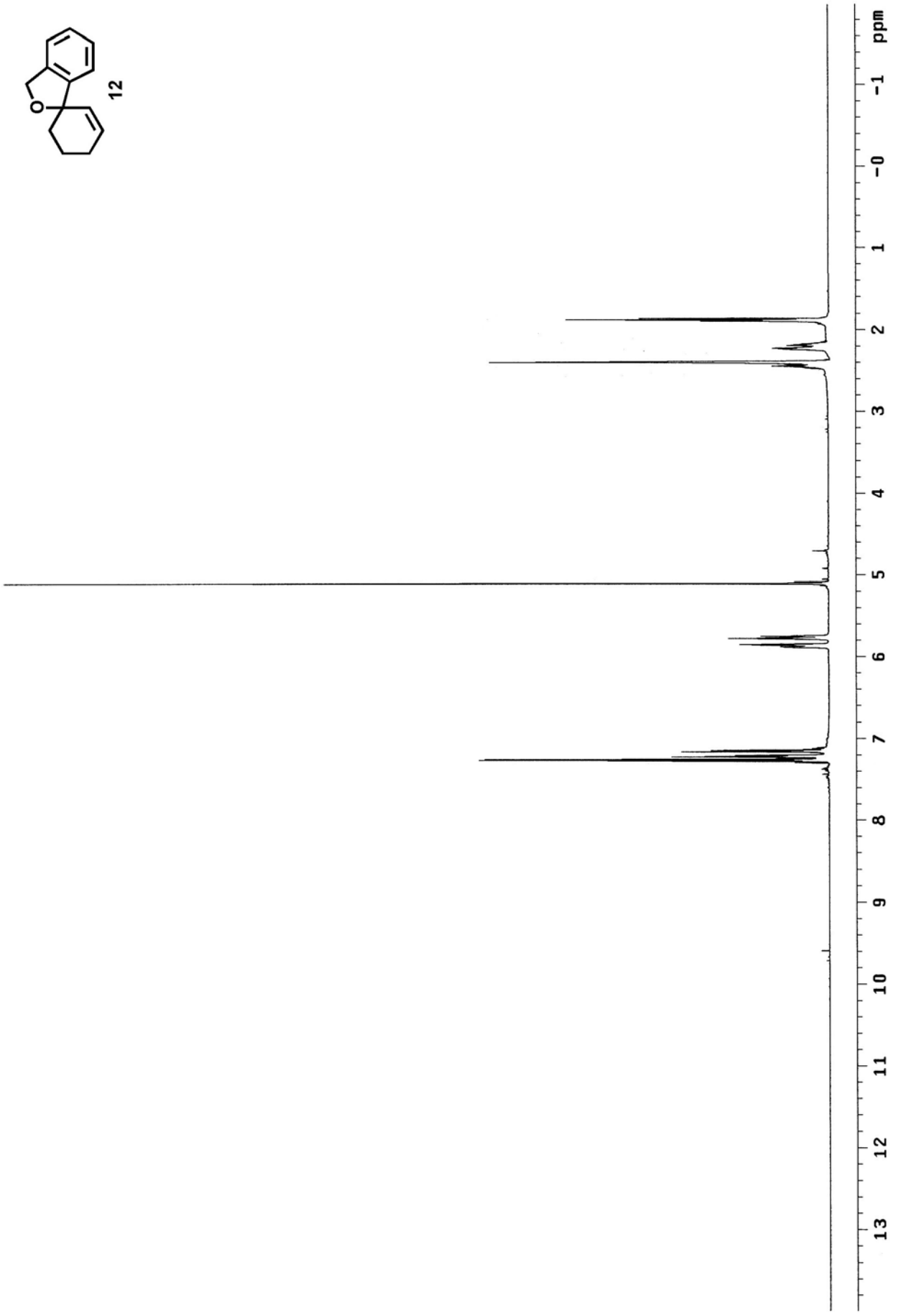


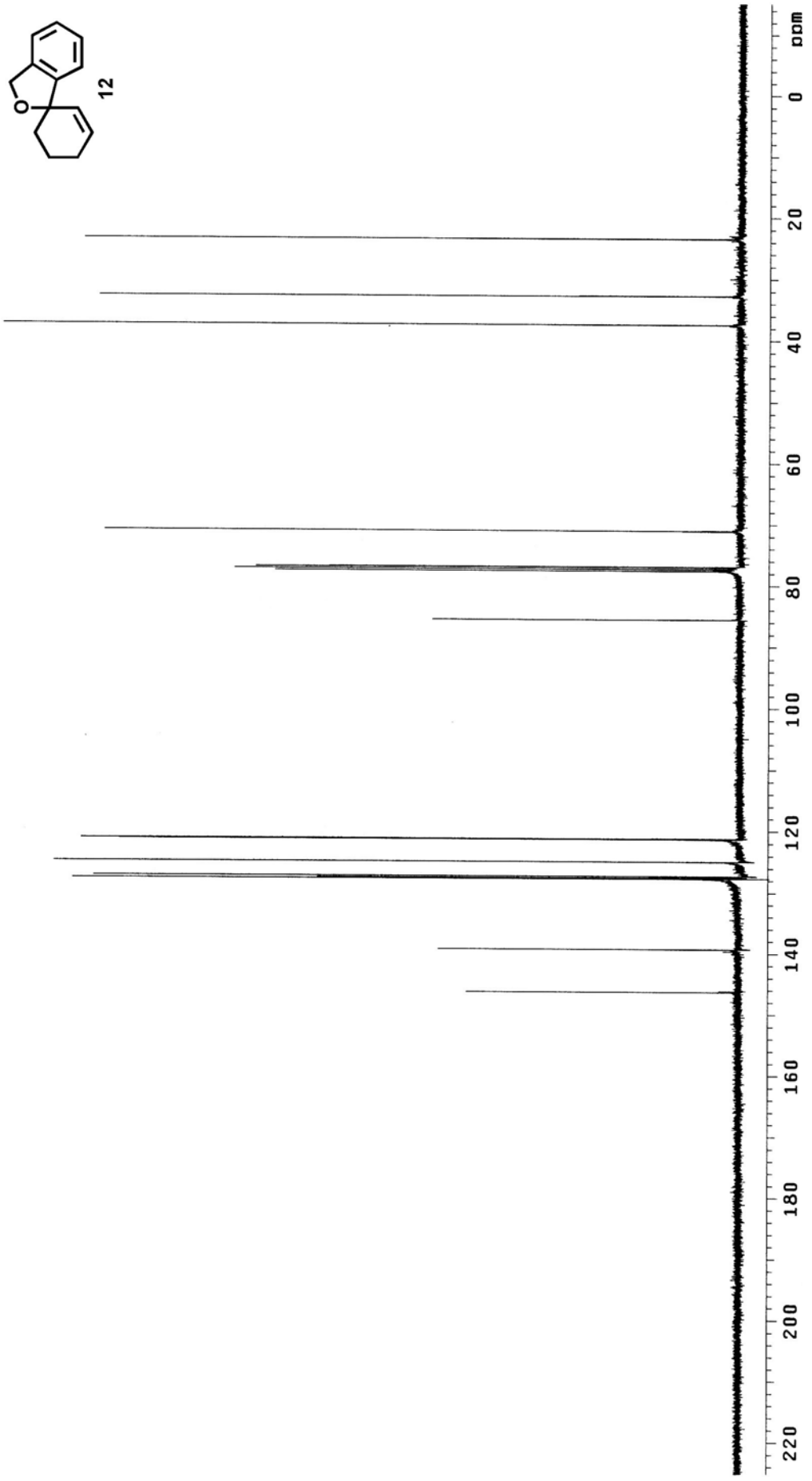
12

Chromatogram Plot

File: d:\users\andreas\spiro\compounds\as_c_comp_22_4022.sms
Sample: as_C_Comp_22_4022
Operator: Operator
Scan Range: 1 - 2309 Time Range: 0.00 - 21.98 min.
Date: 2005-12-11 21:26
Sample Notes: ROUTINE




O
12
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm


12
O
ppm
0
20
40
60
80
100
120
140
160
180
200
220