Supporting Information

Microwave-Enhanced Carbonylative Generation of Indanones and 3-Acylaminoindanones

*Xiongyu Wu, Peter Nilsson, and Mats Larhed**

Organic Pharmaceutical Chemistry, Department of Medicinal Chemistry, Uppsala University, BMC, Box-574, SE-751 23 Uppsala, Sweden.

mats@orgfarm.uu.se

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EXPERIMENTAL SECTION

Procedures

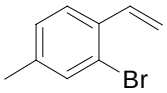
Analytical TLC was performed using Merck glass-backed 0.2 mm silica gel 60 F-254 plates. Visualization was done with UV light. Infrared spectra for neat samples were recorded on a FTIR instrument. NMR spectra were recorded for ^{1}H at 270 MHz or 400 MHz, for ^{13}C NMR at 67.9 MHz or 100 MHz at 25 ^{o}C. Chemical shifts for ^{1}H and ^{13}C were referenced to $CHCl_3$ and $CDCl_3$, respectively. Low-resolution mass spectra were recorded on a GC-MS instrument equipped with a CP-Sil 8 CB capillary column (30 m x 0.25 mm, 0.25 μm) operating at an ionization energy of 70 eV. The oven temperature was 70-300 ^{o}C or 40-300 ^{o}C. High resolution MS was obtained using FAB+ ionization and the indicated values are calculated from the produced $(M^{+}+H)$ ion.

Materials

Palladium(II) acetate was purchased from Merck, tri-*t*-butylphosphonium tetrafluoroborate from Strem, tetrabutylammoniumchloride and 1,3-bis(diphenylphosphino)propane from Fluka. Anhydrous DMF, pyridine, and 1,4-dioxane were obtained from Aldrich. All other reagents were purchased from commercial sources and used as received. Substituted *o*-bromostyrene **1c-f** and *o*-chlorostyrene **4b-e** were synthesized according to the literature.[1] Olefins **1g**,**h**[2,3] were prepared by Heck coupling of *o*-bromophenyl iodide.[2] The triflates **5b-d** were synthesized according to the method for **5a**.[4] Compounds **1a**,**b**, **2a**,[1]**b**,[1] **2c**,[5]**d**,[6] **2f**,[1]**g**,[6] **2j**[7], **4a** are commercially available and **1e**,[8] **1f**,[9] **1g**,**h**, **2e**,[10] **2i**,[11] **3g**,**h**,[12] **4b**,[13] **4e,**[14] **5a**, **5c**,[15,16] **5d**,[17] are all known structures. The data of ^{1}H NMR for **4b** was found not consistent with the literature report[13] but the ^{13}C NMR was in accordance with the report. We have included the NMR and GC-MS of **4b** and also of **4e,** which was reported without detailed data in a patent[14]. All other known products gave satisfactory analytical data corresponding to the reported literature values. The spectroscopic and analytical data are provided below for all new compounds.

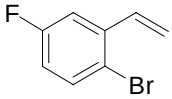
Spectroscopic and analytical data for new compounds

2-Bromo-4-methyl-1-vinyl-benzene (1c)



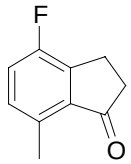
Following the literature method,[1] the product was purified over silica gel (pentane as eluent) to afford the title product as a colorless oil (93%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.32 (s, 3H), 5.32 (dd, J = 10.8, 1.1 Hz, 1H), 5.67 (dd, J = 17.4, 1.1 Hz, 1H), 7.04 (dd, J = 17.4, 10.8 Hz, 1H), 7.07-7.12 (m, 1H), 7.39 (d, J = 0.8 Hz, 1H), 7.45 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.7, 115.7, 123.4, 126.4, 128.3, 133.2, 134.5, 135.5, 139.3; MS (*m/z*, relative intensity) 198 ((M^++2), 98), 196 (M^+, 100); Anal. Calcd for C_9H_9Br: C, 54.85; H, 4.60; Found: C, 54.82; H, 4.61.

2-Bromo-5-fluoro-1-vinyl-benzene (1d)



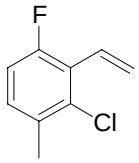
Following the literature method, the product was purified over silica gel (pentane as eluent) to afford the title product as a colorless oil (60%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.42 (d, J = 11.0 Hz, 1H), 5.70 (d, J = 17.4 Hz, 1H), 6.86 (ddd, J = 8.8, 7.9, 3.1 Hz, 1H), 7.00 (ddd, J = 17.4, 11.0, 1.5 Hz, 1H), 7.25 (dd, J = 9.7, 3.1 Hz, 1H), 7.49 (dd, J = 8.8, 5.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 113.5 (d, $J_{C\text{-}F}$ = 23.7 Hz), 116.4 (d, $J_{C\text{-}F}$ = 22.2 Hz), 117.7, 117.9, 134.1 (d, $J_{C\text{-}F}$ = 8.4 Hz), 135.2, 139.3 (d, $J_{C\text{-}F}$ = 6.9 Hz), 162.2 (d, $J_{C\text{-}F}$ = 244.7 Hz); MS (*m/z*, relative intensity) 202 ((M^++2), 100), 200 (M^+, 98); Anal. Calcd for C_8H_6BrF: C, 47.80; H, 3.01; Found: C, 47.96; H, 3.17.

4-Fluoro-7-methyl-indanone (2h)



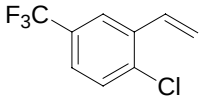
Following the general procedure and purification over silica gel gave a white solid (56.2 mg, 34% yield), ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.57 (s, 3H), 2.65-2.71 (m, 2H), 3.05-3.10 (m, 2H), 7.03-7.15 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 17.5, 20.9, 36.4, 119.9 (d, $J_{C\text{-}F}$ = 19.9 Hz), 130.7 (d, $J_{C\text{-}F}$ = 6.1 Hz), 134.3 (d, $J_{C\text{-}F}$ = 3.1 Hz), 136.4 (d, $J_{C\text{-}F}$ = 3.1 Hz), 141.2 (d, $J_{C\text{-}F}$ = 19.1 Hz), 158.2 (d, $J_{C\text{-}F}$ = 245.4 Hz), 206.7; IR: 1716 cm^{-1}; MS (*m/z*, relative intensity) 164 (M^+, 100), 135 (63); Anal. Calcd for $C_{10}H_9FO$: C, 73.16; H, 5.53. Found: C, 72.91; H, 5.56.

2-Chloro-4-fluoro-1-methyl-3-vinyl-benzene (4c)



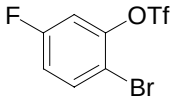
Following the literature method, the product was purified over silica gel (pentane as eluent) to afford the title product as a colorless oil (78%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.35 (s, 3H), 5.64 (m, 1H), 5.93 (m, 1H), 6.84 (dd, J = 18.0, 11.9 Hz, 1H), 6.91 (dd, J = 10.4, 8.5 Hz, 1H), 7.07 (dd, J = 8.5, 5.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.4, 113.9 (d, $J_{C\text{-}F}$ = 22.9 Hz), 122.0 (d, $J_{C\text{-}F}$ = 10.7 Hz), 124.3 (d, $J_{C\text{-}F}$ = 14.5 Hz), 128.4, 129.5 (d, $J_{C\text{-}F}$ = 9.2 Hz), 132.3, 134.2, 159.6 (d, $J_{C\text{-}F}$ = 248.5 Hz). MS (*m/z*, relative intensity) 172 ((M^++2), 35), 170 (M^+, 100); Anal. Calcd for C_9H_8ClF: C, 63.36; H, 4.73. Found: C, 63.40; H, 4.69.

2-Chloro-5-trifluoromethyl-1-vinyl-benzene (4d)



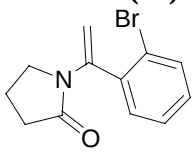
Following the literature method, the product was purified over silica gel (pentane as eluent) to afford the title product as a colorless oil (89%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.50 (d, J = 11.0, 0.6 Hz, 1H), 5.82 (d, J = 17.4, 0.6 Hz, 1H), 7.09 (d, J = 17.4, 11.0 Hz, 1H), 7.38-7.50 (m, 2H), 7.79 (br s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 118.4, 123.5, 123.7 (q, $J_{C\text{-}F}$ = 271.5 Hz, CF_3), 125.2, 129.4 (q, $J_{C\text{-}F}$ = 32.1 Hz), 130.1, 132.1, 136.48, 136.52; MS (*m/z*, relative intensity) 208 ((M^++2), 35), 206 (M^+, 100); Anal. Calcd for $C_9H_6ClF_3$: C, 52.32; H, 2.93. Found: C, 52.55; H, 3.06.

Trifluoro-methanesulfonic acid 2-bromo-5-fluoro-phenyl ester (5b)



Following the literature method[4] and purification over silica gel with CH_2Cl_2 to give the product as a colorless oil, yield 95%. ^{1}H NMR (270 MHz, $CDCl_3$): δ 7.05 (ddd, J = 8.9, 7.6, 2.8 Hz, 1H), 7.14 (dd, J = 8.2, 2.8 Hz, 1H), 7.67 (dd, J = 8.9, 5.6 Hz, 1H); ^{13}C NMR (67.9 MHz, $CDCl_3$): δ 110.7 (d, $J_{C\text{-}F}$ = 3.7 Hz), 111.4 (d, $J_{C\text{-}F}$ = 26.8 Hz), 117.1 (d, $J_{C\text{-}F}$ = 23.2 Hz), 118.5 (q, $J_{C\text{-}F}$ = 320.9 Hz), 134.8 (d, $J_{C\text{-}F}$ = 8.5 Hz), 147.0 (d, $J_{C\text{-}F}$ = 11.0 Hz), 161.6 (d, $J_{C\text{-}F}$ = 251.3 Hz); MS (*m/z*, relative intensity) 324 ((M^++2), 98), 322 (M^+, 100); Anal. Calcd for $C_7H_7BrF_4O_3S$: C, 26.02; H, 0.94; Found: C, 26.15; H, 1.05.

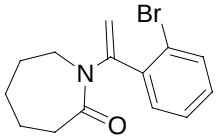
1-[1-(2-Bromo-phenyl)-vinyl]-pyrrolidin-2-one (7a)



Following the general procedure and subsequent purification over silica gel with eluent of 60:40:5 hexane: diethyl ether: triethylamine furnished a pale yellow liquid (356 mg, 44% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.98-2.10 (m, 2H), 2.47 (t, J = 8.1 Hz, 2H), 3.49 (t, J = 7.0 Hz, 2H), 4.85 (s, 1H), 5.31 (s, 1H), 7.14-7.21 (m, 1H), 7.24-7.35 (m, 2H), 7.51 (dd, J = 8.0,

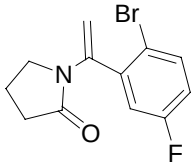
0.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.0, 31.9, 48.5, 106.4, 121.8, 127.3, 129.5, 131.0, 132.4, 138.4, 143.1, 173.6; IR: 1708 cm^{-1}; MS (*m/z*, relative intensity) 267 ((M^++2), 5), 265 (M^+, 5), 186 (100); HRMS (FAB+) Calcd for $C_{12}H_{13}BrNO$ (M^++H): 266.0181; Found: 266.0175; Anal. Calcd for $C_{12}H_{12}BrNO$: C, 73.16; H, 5.53; N, 5.26. Found: C, 72.91; H, 5.56; N, 5.37.

1-[1-(2-Bromo-phenyl)-vinyl]-azepan-2-one (7b)



Following the general procedure and purification over silica gel with eluent of 65:35:5 hexane: diethyl ether: triethylamine furnished a pale yellow liquid (423 mg, 48% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.74 (br s, 6H), 2.50-265 (m, 2H), 3.55-3.65 (m, 2H), 5.19 (s, 1H), 5.34 (s, 1H), 7.10-7.16 (m, 1H), 7.28 (ddd, J = 7.5, 7.5, 1.2 Hz, 1H), 7.42 (dd, J =7.5, 1.2 Hz, 1H), 7.51 (dd, J = 7.5, 1.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.3, 28.6, 29.7, 37.6, 51.9, 113.0, 121.0, 127.2, 129.2, 131.6, 132.9, 139.3, 148.2, 175.1; IR: 1666 cm^{-1}; MS (*m/z*, relative intensity) 295 ((M^++2), 10), 293 (M^+, 9), 214 (100); HRMS (FAB+) Calcd for $C_{14}H_{17}BrNO$ (M^++H): 294.0494; Found: 294.0490; Anal. Calcd for $C_{14}H_{16}BrNO$: C, 57.16; H, 5.48; N, 4.76. Found: C, 56.98; H, 5.54; N, 4.83.

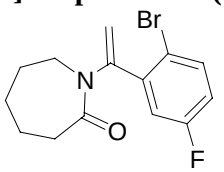
1-[1-(2-Bromo-5-fluoro-phenyl)-vinyl]-pyrrolidin-2-one (7c)



Following the general procedure and purification over silica gel with eluent of 65:35:5 hexane: diethyl ether: triethylamine furnished a pale yellow liquid (222 mg, 26% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.02-2.14 (m, 2H), 2.46 (t, J = 8.1 Hz, 2H), 3.55 (t, J = 7.2 Hz, 2H), 4.81 (s,

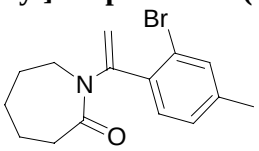
1H), 5.21 (s, 1H), 6.87-6.96 (m, 1H), 7.06 (dd, J = 8.8, 3.1 Hz, 1H), 7.45 (dd, J = 8.8, 5.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.0, 31.7, 48.5, 106.1, 116.0, 116.5 (d, $J_{C\text{-}F}$ = 22.2 Hz), 118.1 (d, $J_{C\text{-}F}$ = 23.7 Hz), 133.6 (d, $J_{C\text{-}F}$ = 8.4 Hz), 140.3 (d, $J_{C\text{-}F}$ = 7.6 Hz), 142.5, 161.6 (d, $J_{C\text{-}F}$ = 246.2 Hz), 173.4; IR: 1708 cm^{-1}; MS (*m/z*, relative intensity) 285 ((M^++2), 2), 283 (M^+, 2), 204 (100); HRMS (FAB+) Calcd for $C_{12}H_{11}BrFNO$ (M^++H): 284.0086; Found: 284.0063; Anal. Calcd for $C_{12}H_{12}BrNO$: C, 50.73; H, 3.90, N, 4.93. Found: C, 50.58; H, 4.00; N, 5.02.

1-[1-(2-Bromo-5-fluoro-phenyl)-vinyl]-azepan-2-one (7d)



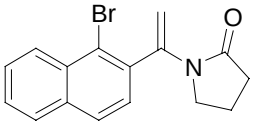
Following the general procedure and purification over silica gel with eluent of 65:35:5 hexane: diethyl ether: triethylamine furnished a pale yellow liquid (415 mg, 33% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.75 (br s, 6H), 2.50-2.65 (m, 2H), 3.55-3.70 (m, 2H), 5.16 (d, J = 0.6 Hz, 1H), 5.34 (d, J = 0.6 Hz, 1H), 6.81-6.89 (m, 1H), 7.14 (dd, J = 9.2, 2.9 Hz, 1H), 7.44 (dd, J = 8.8, 5.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.2, 28.7, 29.7, 37.4, 52.4, 113.0, 115.0 (d, $J_{C\text{-}F}$ = 2.1 Hz), 116.4 (d, $J_{C\text{-}F}$ = 22.2 Hz), 118.5 (d, $J_{C\text{-}F}$ = 23.7 Hz), 134.1 (d, $J_{C\text{-}F}$ = 8.4 Hz), 141.3 (d, $J_{C\text{-}F}$ = 7.7 Hz), 148.0, 161.7 (d, $J_{C\text{-}F}$ = 245.5 Hz), 175.0; IR: 1665 cm^{-1}; MS (*m/z*, relative intensity) 313 ((M^++2), 10), 311 (M^+, 10), 232 (100); HRMS (FAB+) Calcd for $C_{14}H_{16}BrFNO$ (M^++H): 312.0399; Found: 312.0401; Anal. Calcd for $C_{14}H_{15}BrFNO$: C, 53.86; H, 4.84; N, 4.49. Found: C, 54.01; H, 4.95; N, 4.67.

1-[1-(2-Bromo-4-methyl-phenyl)-vinyl]-azepan-2-one (7f)



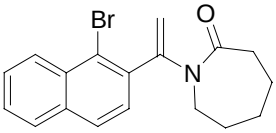
Following the general procedure and purification over silica gel with gradient eluent of 20:80:5 to 65:35:5 hexane: diethyl ether: triethylamine furnished a pale yellow liquid (487 mg, 53%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.66-1.78 (m, 6H), 2.29 (s, 3H), 2.52-2.60 (m, 2H), 3.54-3.62 (m, 2H), 5.16 (s, 1H), 5.30 (s, 1H), 7.07 (dd, J = 7.8, 0.6 Hz, 1H), 7.29 (d, J = 7.8 Hz, 1H), 7.33 (d, J = 0.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.7, 23.3, 28.6, 29.8, 37.6, 51.8, 112.8, 120.7, 128.1, 131.4, 133.4, 136.3, 139.5, 148.0, 175.1; IR: 1664 cm^{-1}; MS (*m/z*, relative intensity) 309 ((M^++2), 7), 307 (M^+, 6), 228 (100); HRMS (FAB+) Calcd for $C_{15}H_{19}BrNO$ (M^++H): 308.0650; Found: 308.0647; Anal. Calcd for $C_{15}H_{18}BrNO$: C, 58.45; H, 5.89; N, 4.54. Found: C, 58.68; H, 5.77; N, 4.47.

1-[1-(1-Bromo-naphthalen-2-yl)-vinyl]-pyrrolidin-2-one (7g)



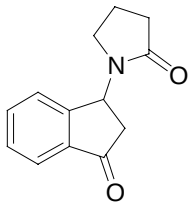
Following the general procedure and purification over silica gel with 50:50:5 hexane: diethyl ether: triethylamine furnished a pale yellow liquid (576 mg, 61 %). ^{1}H NMR (270 MHz, $CDCl_3$): δ 1.90-2.15 (m, 2H), 2.50 (t, J = 8.0 Hz, 2H), 3.52 (t, J = 7.0 Hz, 2H), 4.95 (s, 1H), 5.50 (s, 1H), 7.43 (d, J = 8.3 Hz, 1H), 7.46-7.64 (m, 2H), 7.76-7.88 (m, 2H), 8.33 (d, J = 8.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.0, 31.9, 48.5, 106.7, 122.2, 126.8, 127.5, 127.62, 127.68, 128.01, 128.08, 132.0, 133.9, 136.5, 143.7, 173.7; IR: 1702 cm^{-1}; MS (*m/z*, relative intensity) 318 (((M^++2)+H), 1), 316 ((M^++H), 1), 236 (100); HRMS (FAB+) Calcd for $C_{16}H_{15}BrNO$ (M^++H): 316.0337; Found: 316.0344; Anal. Calcd for $C_{16}H_{14}BrNO$: C, 60.68; H, 4.46; N, 4.43. Found: C, 60.78; H, 4.46; N, 4.43.

1-[1-(1-Bromo-naphthalen-2-yl)-vinyl]-azepan-2-one (7h)



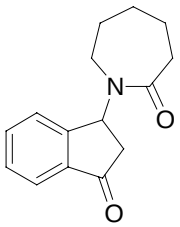
Following the general procedure and purification over silica gel with 50:45:5 hexane: diethyl ether: triethylamine furnished a white solid (807 mg, 78 %). ^{1}H NMR (270 MHz, $CDCl_3$): δ 1.74 (br s, 6H), 2.50-2.65 (m, 2H), 3.55-3.75 (m, 2H), 5.26 (s, 1H), 5.47 (s, 1H), 7.40-7.63 (m, 3H), 7.79 (m, 2H), 8.34 (d, J = 8.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.3, 28.6, 29.7, 37.6, 51.8, 113.5, 121.3, 126.6, 127.4, 127.7, 128.0, 128.7, 132.1, 133.9, 137.7, 148.7, 175.1; IR: 1663 cm^{-1}; MS (*m/z*, relative intensity) 346 (((M^{+}+2)+H), 1), 316 ((M^{+}+H), 1), 264 (M^{+}), 100); HRMS (FAB+) Calcd for $C_{18}H_{19}BrNO$ (M^{+}+H): 344.0650; Found: 344.0661; Anal. Calcd for $C_{16}H_{14}BrNO$: C, 62.80; H, 5.27; N, 4.07. Found: C, 62.84; H, 5.39; N, 4.13.

1-(3-Oxo-indan-1-yl)-pyrrolidin-one (8a)



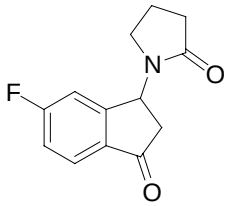
Following the general procedure at a 0.64 mmol scale and purification over silica gel with 1:99 methanol : dichloromethane furnished a white solid (76 mg, 58% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.90-2.10 (m, 2H), 2.50 (d, J = 7.6 Hz, 1H), 2.52 (dd, J = 7.6, 0.8 Hz, 1H), 2.57 (dd, J = 19.2, 3.6 Hz, 1H), 2.88 (ddd, J = 9.5, 8.2, 5.7 Hz, 1H), 3.04 (dd, J = 19.2, 8.0 Hz, 1H), 3.13 (ddd, J = 9.5, 8.1, 6.3 Hz, 1H), 6.01 (dd, J = 8.0, 3.6 Hz, 1H), 7.43-7.51 (m, 2H), 7.66 (ddd, J = 8.8, 7.5, 1.3 Hz, 1H), 7.78 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 17.9, 31.1, 40.2, 42.1, 48.9, 123.6, 125.8, 129.2, 135.3, 137.2, 152.0, 175.3, 202.8. IR: 1716, 1686 cm^{-1}; MS (*m/z*, relative intensity) 215 (M^{+}, 100); Anal. Calcd for $C_{13}H_{13}NO_2$: C, 72.54; H, 6.09; N, 6.51. Found: C, 72.34; H, 6.17; N, 6.66.

1-(3-Oxo-indan-1-yl)-azepan-one (8b)



Following the general procedure at a 0.50 mmol scale and purification over silica gel with 3:97 methanol: dichloromethane, then 10:90 ethyl acetate: diethyl ether furnished a white solid (58 mg, 47% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.34-1.54 (m, 2H), 1.58-1.80 (m, 4H), 2.44 (dd, J = 19.4, 3.6 Hz, 1H), 2.56-2.68 (m, 2H), 2.94 (t, J = 5.1 Hz, 2H), 3.04 (dd, J = 19.4, 7.8 Hz, 1H), 6.46 (dd, J = 7.8, 3.6 Hz, 1H), 7.40-7.50 (m, 2H), 7.60-7.67 (m, 1H), 7.77 (d, J = 7.7 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.4, 29.5, 29.9, 37.5, 40.9, 44.0, 51.4, 123.5, 126.0, 129.0, 135.2, 137.8, 153.2, 176.6, 203.4. IR: 1716, 1643 cm^{-1}; MS (*m/z*, relative intensity) 243 (M^+, 100); Anal. Calcd for $C_{15}H_{17}NO_2$: C, 74.05; H, 7.04; N, 5.76. Found: C, 73.84; H, 7.04; N, 5.71.

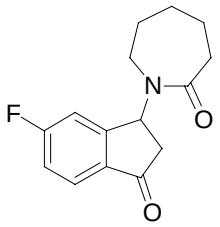
1-(6-Fluoro-3-oxo-indan-1-yl)-pyrrolidin-one (8c)



Following the general procedure at a 0.61 mmol scale and purification over silica gel with gradient eluent of 70:30 ethyl acetate: petroleum ether to 100% ethyl acetate furnished a white solid (70 mg, 48% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.92-2.10 (m, 2H), 2.44-2.52 (m, 2H), 2.57 (dd, J = 19.2, 3.7 Hz, 1H), 2.85-2.95 (m, 1H), 3.03 (dd, J = 19.2, 8.1 Hz, 1H), 3.11-3.20 (m, 1H), 5.92-6.00 (m, 1H), 7.10 (d, J = 8.1 Hz, 1H), 7.16 (ddd, J = 10.2, 8.4, 1.7 Hz, 1H), 7.77 (dd, J = 8.4, 5.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 17.8, 30.9, 40.2, 42.0, 48.5, 112.4 (d, $J_{C\text{-}F}$ = 22.2 Hz), 117.6 (d, $J_{C\text{-}F}$ = 23.7 Hz), 126.0 (d, $J_{C\text{-}F}$ = 9.9 Hz), 133.5, 155.1 (d,

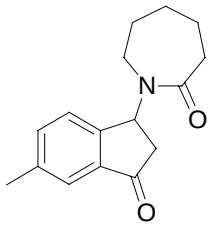
$J_{C\text{-}F}$ = 9.2 Hz), 167.4 (d, $J_{C\text{-}F}$ = 257.0 Hz), 175.3, 200.7. IR: 1716, 1696 cm^{-1}; MS (*m/z*, relative intensity) 233 (M^+, 100); Anal. Calcd for $C_{13}H_{12}FNO_2$: C, 66.94; H, 5.19; N, 6.01. Found: C, 66.84; H, 5.28; N, 6.00.

1-(6-Fluoro-3-oxo-indan-1-yl)-azepan-one (8d)



Following the general procedure at a 0.82 mmol scale and purification over silica gel with gradient eluent of 30:70 to 60:40 ethyl acetate: petroleum ether furnished a white solid (111 mg, 52% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.40-1.58 (m, 2H), 1.66-1.82 (m, 4H), 2.49 (dd, J = 19.4, 3.8 Hz, 1H), 2.60-2.68 (m, 2H), 2.94-3.00 (m, 2H), 3.06 (dd, J = 19.4, 7.9 Hz, 1H), 6.38-6.47 (m, 1H), 7.10-7.20 (m, 2H), 7.79 (dd, J = 8.4, 5.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.3, 29.5, 29.8, 37.4, 41.0, 44.1, 51.2, 112.5 (d, $J_{C\text{-}F}$ = 22.9 Hz), 117.4 (d, $J_{C\text{-}F}$ = 23.7 Hz), 125.9 (d, $J_{C\text{-}F}$ = 9.9 Hz), 134.1, 156.4 (d, $J_{C\text{-}F}$ = 9.2 Hz), 167.4 (d, $J_{C\text{-}F}$ = 256.9 Hz), 176.6, 201.3. IR: 1718, 1644 cm^{-1}; MS (*m/z*, relative intensity) 261 (M^+, 100); Anal. Calcd for $C_{15}H_{16}FNO_2 \cdot 1/2H_2O$: C, 66.65; H, 6.34; N, 5.18. Found: C, 66.63; H, 5.97; N, 5.21.

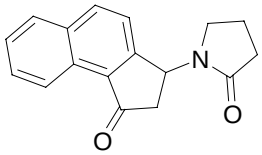
1-(5-Methyl-3-oxo-indan-1-yl)-azepan-one (8f)



Following the general procedure at a 0.80 mmol scale and purification over silica gel with 70:30 ethyl acetate: petroleum ether and purification furnished a white solid (113 mg, 55%

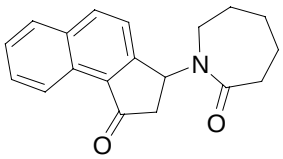
yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.38-1.48 (m, 2H), 1.62-1.76 (m, 4H), 2.40 (s, 3H), 2.41 (dd, J = 19.2, 3.5 Hz, 1H), 2.57-2.64 (m, 2H), 2.88-2.96 (m, 2H), 3.02 (dd, J = 19.2, 7.3 Hz, 1H), 6.36-6.43 (m, 1H), 7.34 (d, J = 7.9 Hz, 1H), 7.44 (d, J = 7.9 Hz, 1H), 7.54 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.9, 23.1, 29.2, 29.6, 37.3, 41.0, 43.6, 50.9, 123.1, 125.4, 136.2, 137.7, 138.9, 150.3, 176.2, 203.2. IR: 1718, 1636 cm^{-1}; MS (*m/z*, relative intensity) 257 (M^+, 100); Anal. Calcd for $C_{16}H_{19}NO_2$: C, 74.68; H, 7.44; N, 5.44. Found: C, 74.51; H, 7.44; N, 5.44.

1-(1-Oxo-2,3-dihydro-1*H*-cyclopenta[*a*]naphthalen-3-yl)-pyrrolidin-2-one (8g)



Following the general procedure at a 0.80 mmol scale and purification over silica gel with ethyl acetate furnished a white solid (179 mg, 84% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.90-2.06 (m, 2H), 2.51 (t, J = 8.0 Hz, 2H), 2.66 (dd, J = 18.8, 3.2 Hz, 1H), 2.80-2.90 (m, 1H), 3.08-3.20 (m, 2H), 6.07 (dd, J = 7.7, 3.2 Hz, 1H), 7.46 (d, J = 8.2 Hz, 1H), 7.54-7.64 (m, 1H), 7.65-7.72 (m, 1H), 7.90 (d, J = 8.2 Hz, 1H), 8.10 (d, J = 8.4 Hz, 1H), 9.11 (d, J = 8.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 17.8, 31.0, 40.6, 42.1, 48.6, 122.1, 124.3, 127.4, 128.1, 128.7, 129.3, 131.6, 133.4, 136.6, 154.7, 175.4, 203.2. IR: 1701, 1685 cm^{-1}; MS (*m/z*, relative intensity) 265 (M^+, 100); Anal. Calcd for $C_{17}H_{15}NO_2 \cdot H_2O$: C, 72.07; H, 6.04; N, 4.94. Found: C, 72.07; H, 6.04; N, 5.09.

1-(1-Oxo-2,3-dihydro-1*H*-cyclopenta[*a*]naphthalen-3-yl)-azepan-2-one (8h)

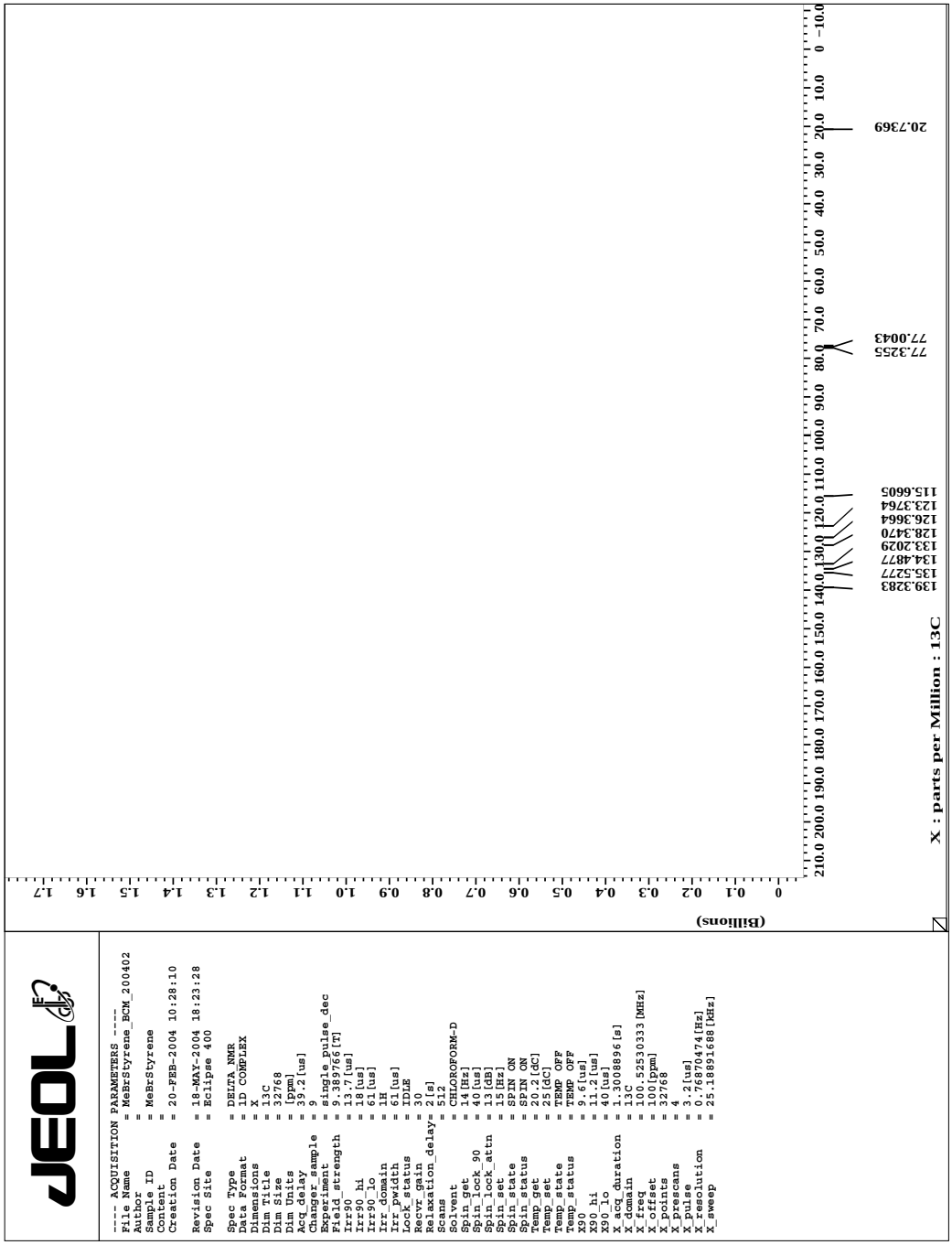


Following the general procedure at a 0.80 mmol scale and purification over silica gel with 65:35 ethyl acetate: petroleum ether furnished a white solid (207 mg, 88% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.38-1.50 (m, 2H), 1.62-1.82 (m, 4H), 2.56 (dd, J = 19.1, 3.2 Hz, 1H), 2.62-2.72 (m, 2H), 2.92-3.02 (m, 2H), 3.16 (dd, J = 19.1, 7.6 Hz, 1H), 6.52-6.60 (m, 1H), 7.50 (d, J = 8.4 Hz, 1H), 7.60 (ddd, J = 8.1, 7.0, 1.1 Hz, 1H), 7.66-7.74 (m, 1H), 7.91 (d, J = 8.2 Hz, 1H), 8.09 (d, J = 8.4 Hz, 1H), 9.14 (d, J = 8.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 23.4, 29.5, 29.9, 37.6, 41.4, 43.7, 51.1, 122.6, 124.4, 127.4, 128.2, 128.9, 129.4, 132.3, 133.4, 136.5, 156.0, 176.7, 204.0. IR: 1699, 1643 cm^{-1}; MS (*m/z*, relative intensity) 293 (M^+, 100); Anal. Calcd for $C_{19}H_{19}NO_2$: C, 77.79; H, 6.53; N, 4.77. Found: C, 77.97; H, 6.58; N, 4.86.

APPENDIX

^{13}C NMR spectra and GC-MS chromatograms for new compounds 1c,**d**, **2h, 4c**,**d**, **5b**, **7a-h**, **8a-h**

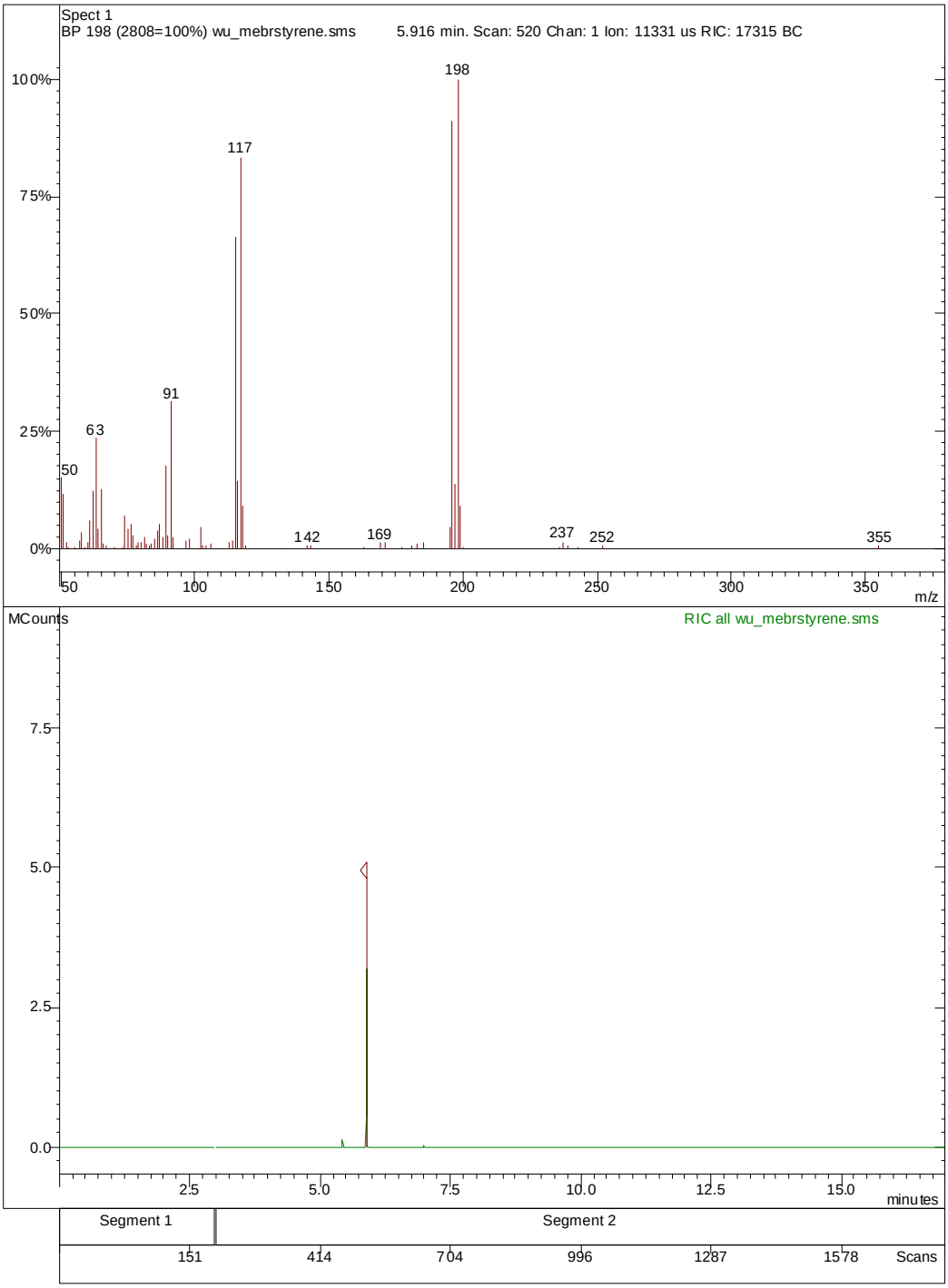
2-Bromo-4-methyl-1-vinyl-benzene (1c)



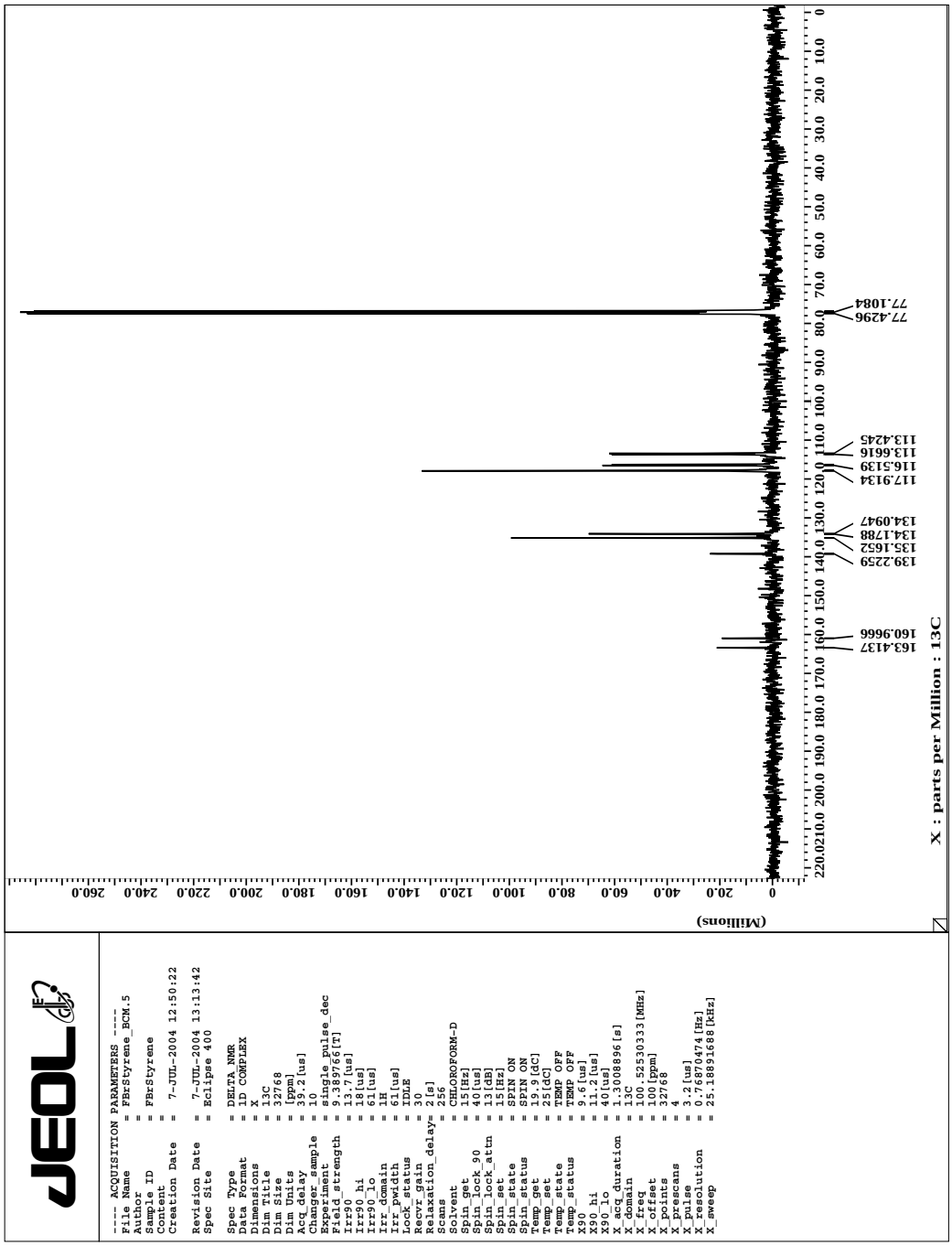
Chromatogram Plot

File: i:\wu_mebrstyrene.sms
Sample: wu_MeBrStyrene
Scan Range: 1 - 1809 Time Range: 0.00 - 16.98 min.
Sample Notes: ROUTINE

Operator: Andrei
Date: 02/20/04 16:35



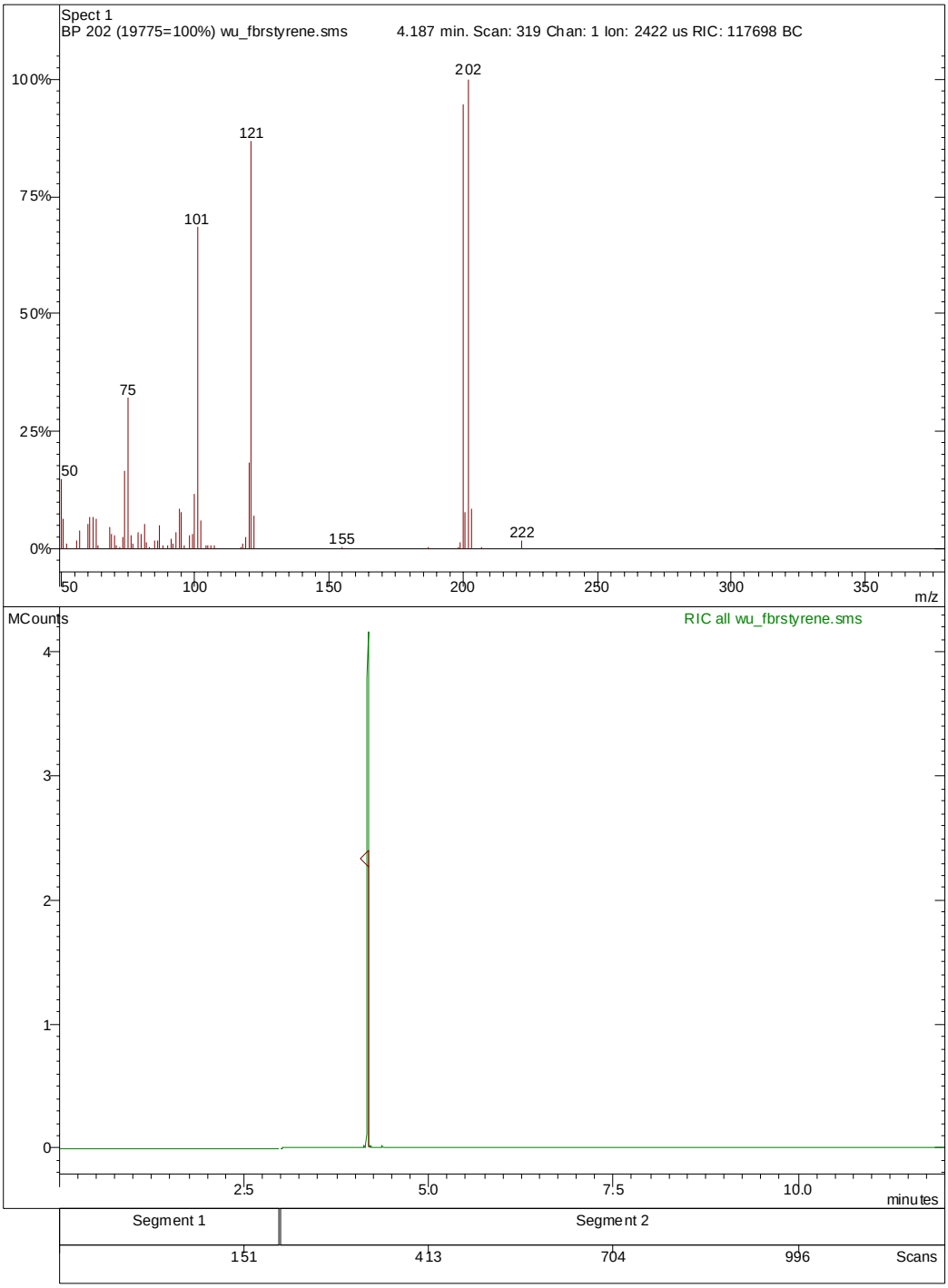
2-Bromo-5-fluoro-1-vinyl-benzene (1d)



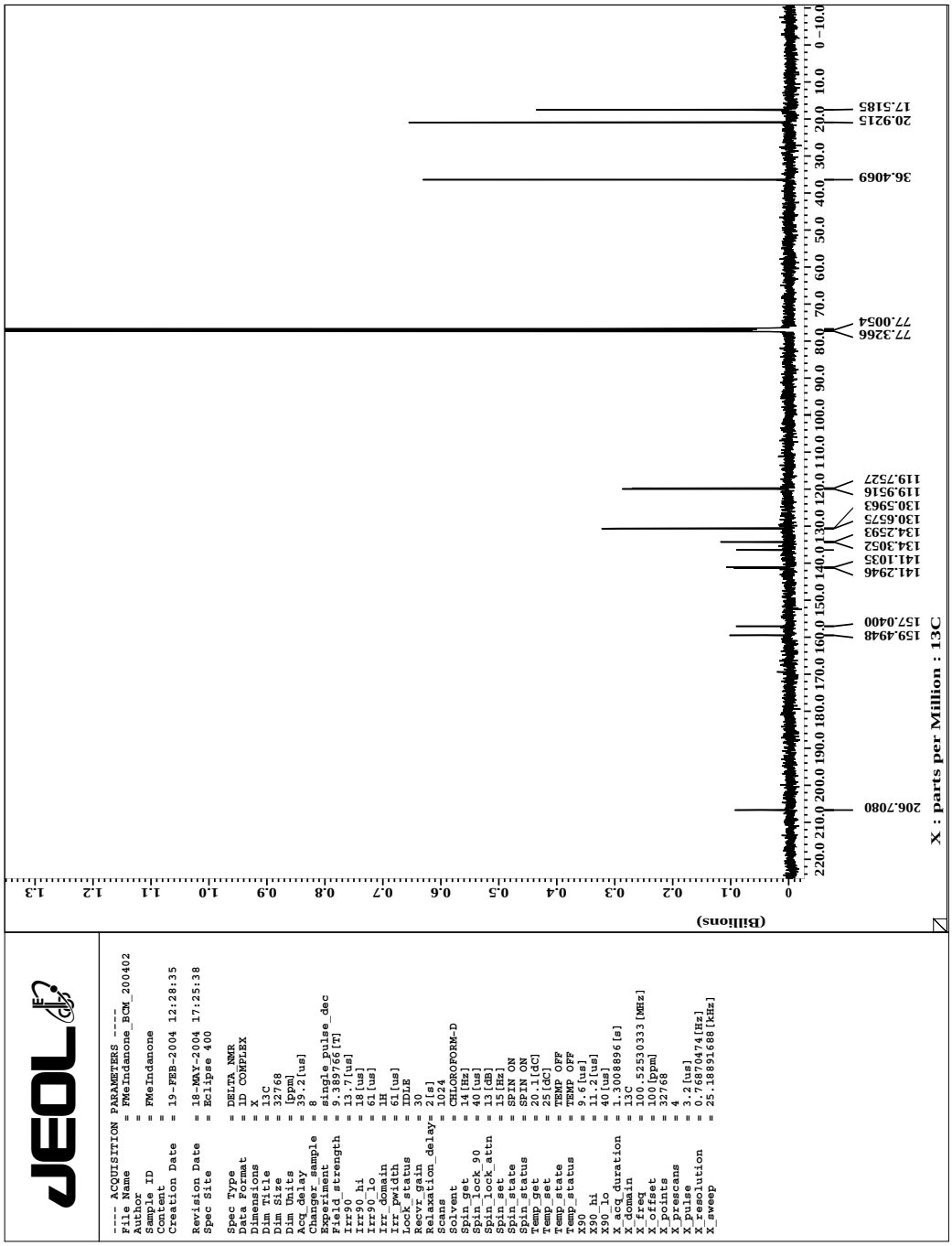
Chromatogram Plot

File: i:\wu_fbrstyrene.sms
Sample: wu_FBrstyrene
Scan Range: 1 - 1227 Time Range: 0.00 - 11.98 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 05/12/04 23:03



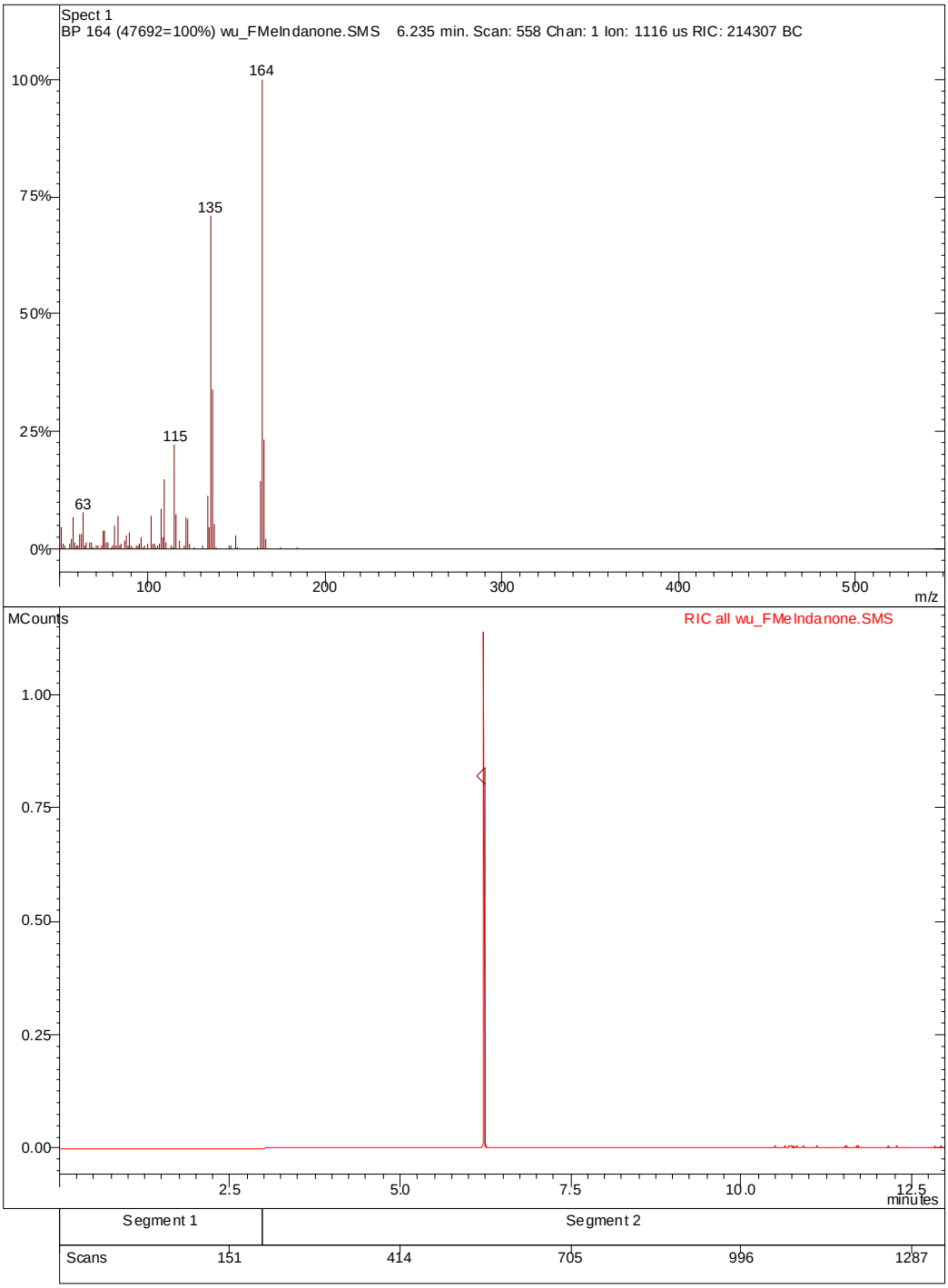
4-Fluoro-8-methyl-indan-1-one (2h)



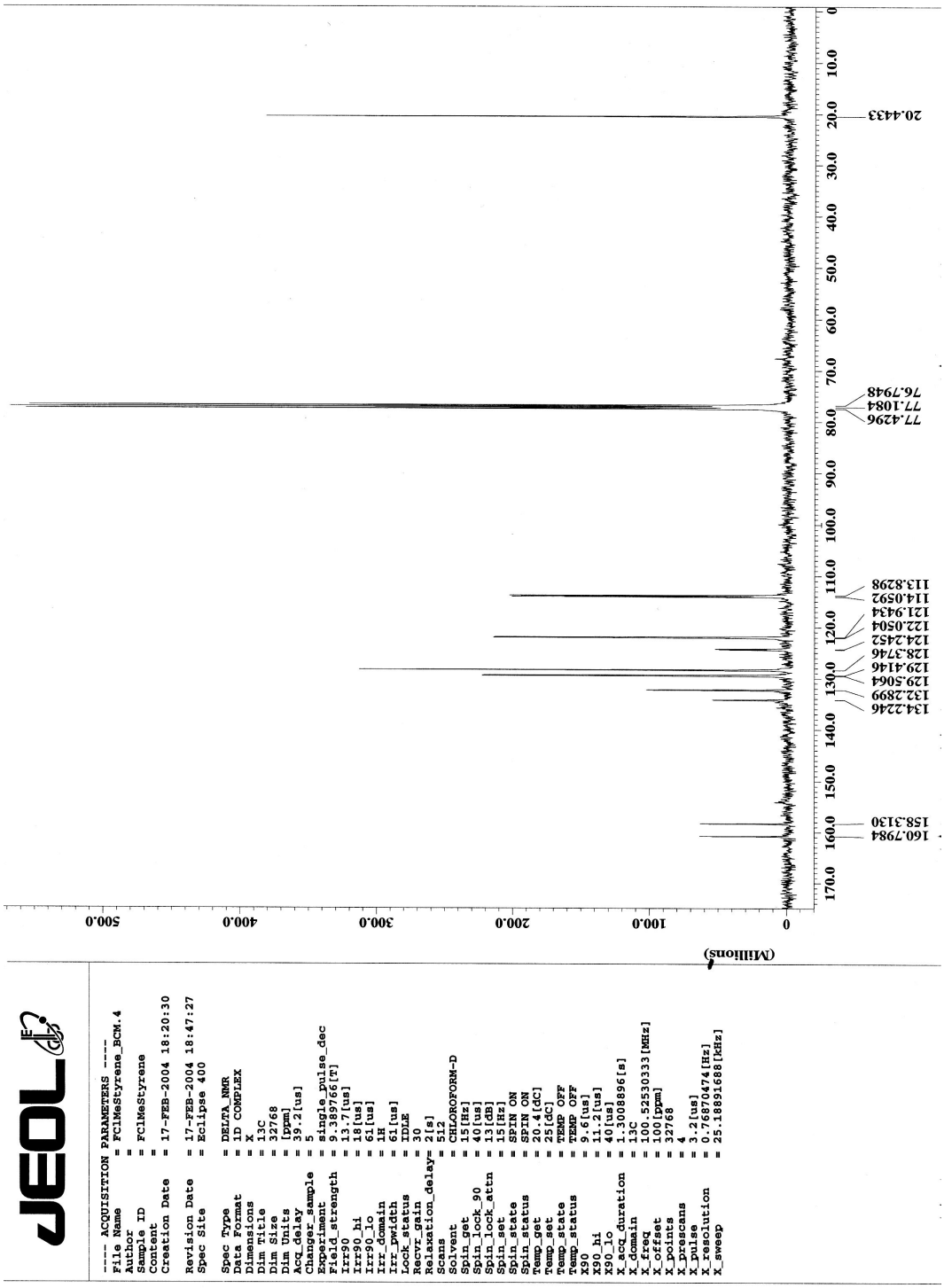
Chromatogram Plot

File: i:\wu_fmeindanone.sms
Sample: wu_FMeIndanone
Scan Range: 1 - 1344 Time Range: 0.00 - 12.99 min.
Sample Notes: ROUTINE

Operator: Andrei
Date: 02/22/04 20:01



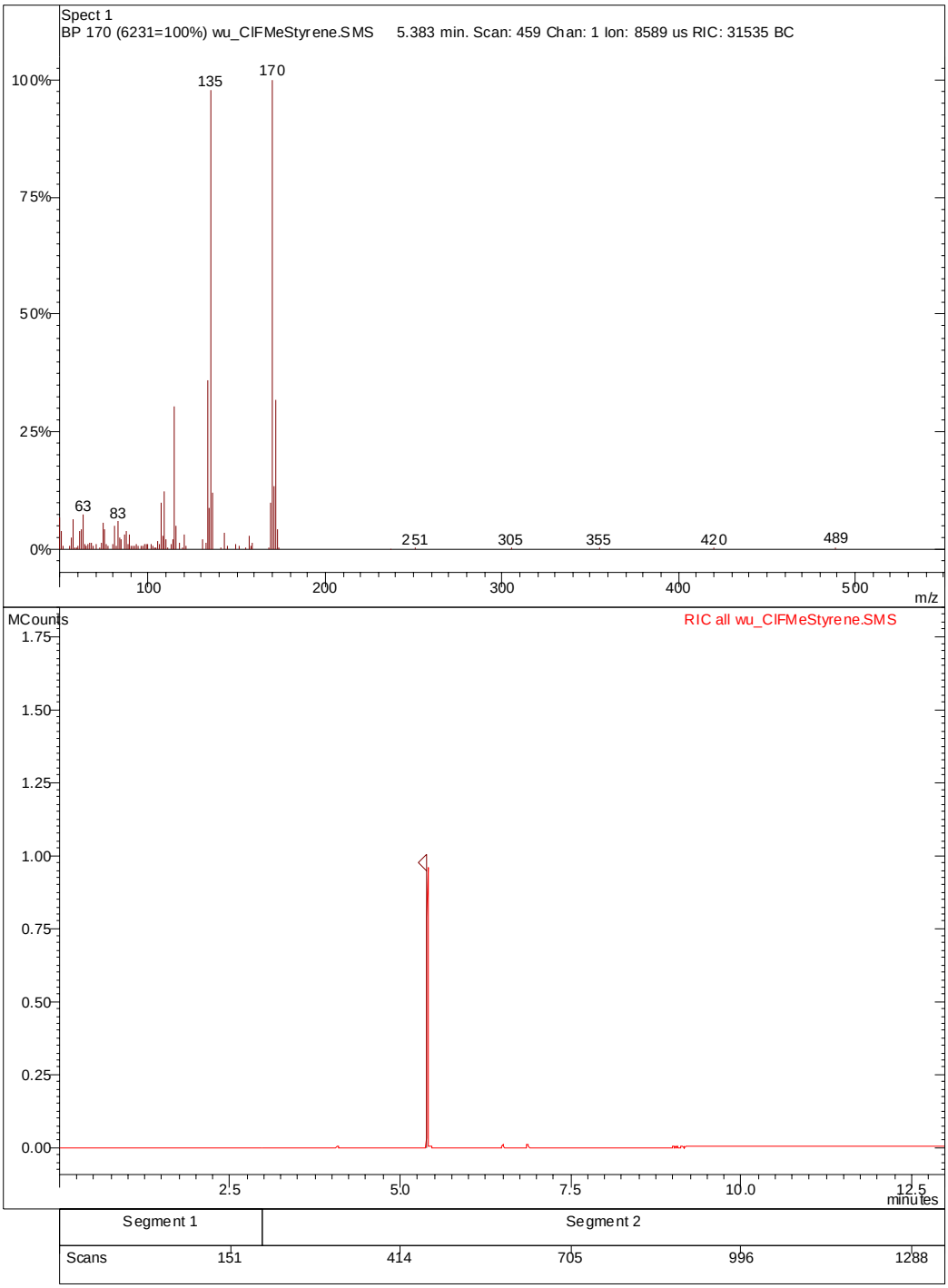
2-Chloro-4-fluoro-1-methyl-3-vinyl-benzene (4c)



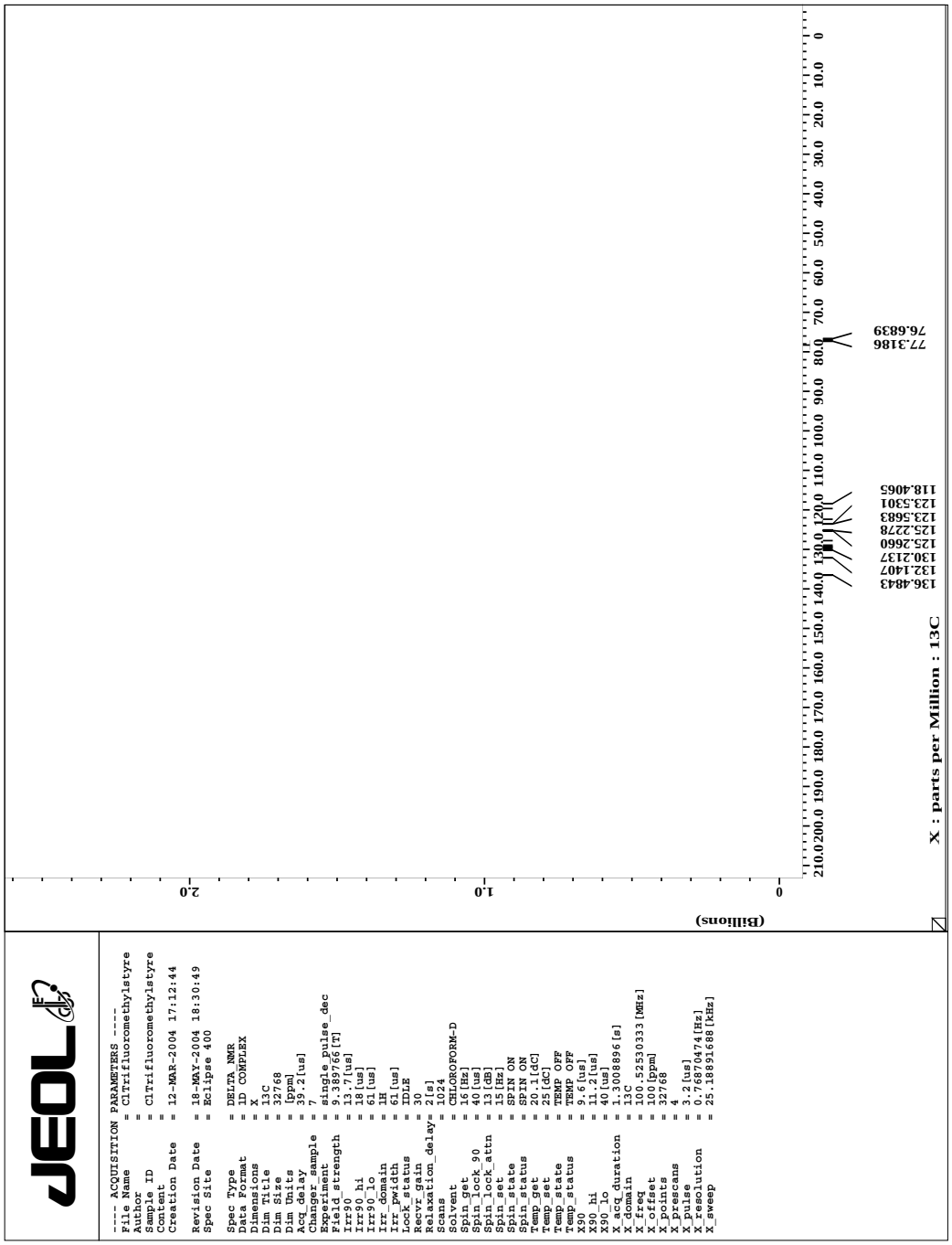
Chromatogram Plot

File: i:\wu_clfmestyrene.sms
Sample: wu_ClFMeStyrene
Scan Range: 1 - 1344 Time Range: 0.00 - 12.98 min.
Sample Notes: ROUTINE

Operator: Andrei
Date: 02/22/04 18:55



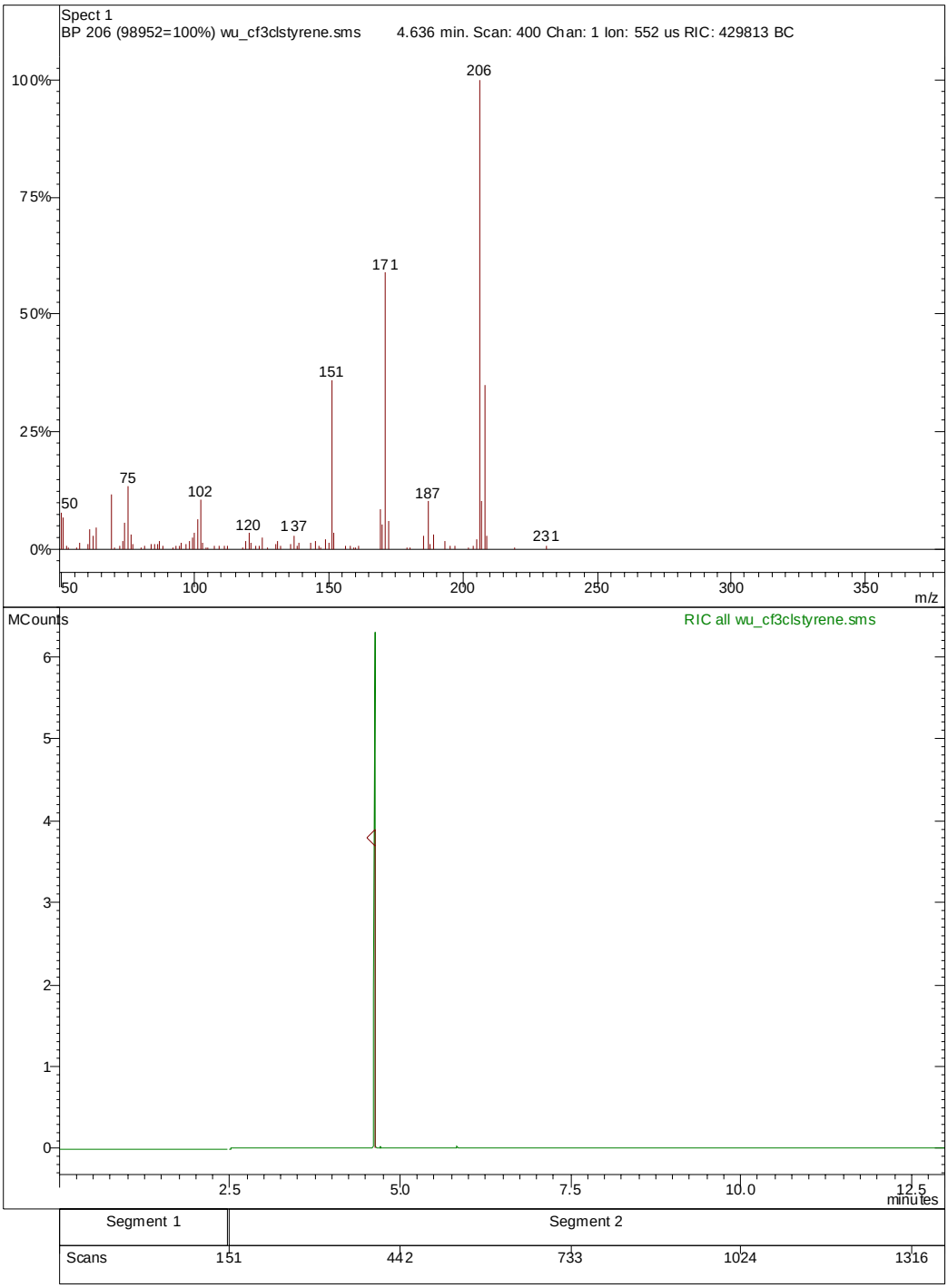
2-Chloro-5-trifluoromethyl-1-vinyl-benzene (4d)



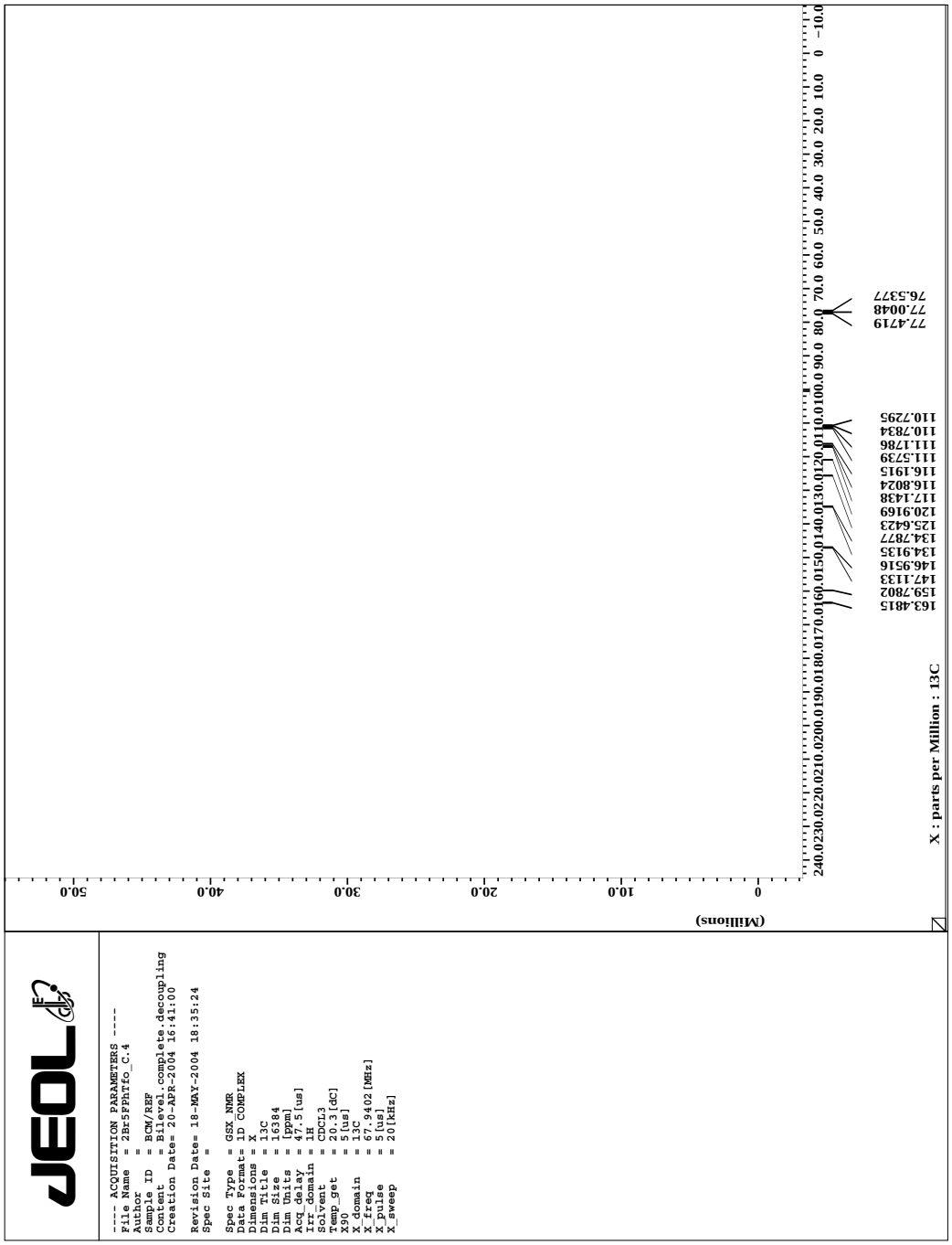
Chromatogram Plot

File: i:\wu_cf3clstyrene.sms
Sample: wu_CF3ClStyrene
Scan Range: 1 - 1373 Time Range: 0.00 - 12.99 min.
Sample Notes: today

Operator: Operator
Date: 05/12/04 13:28



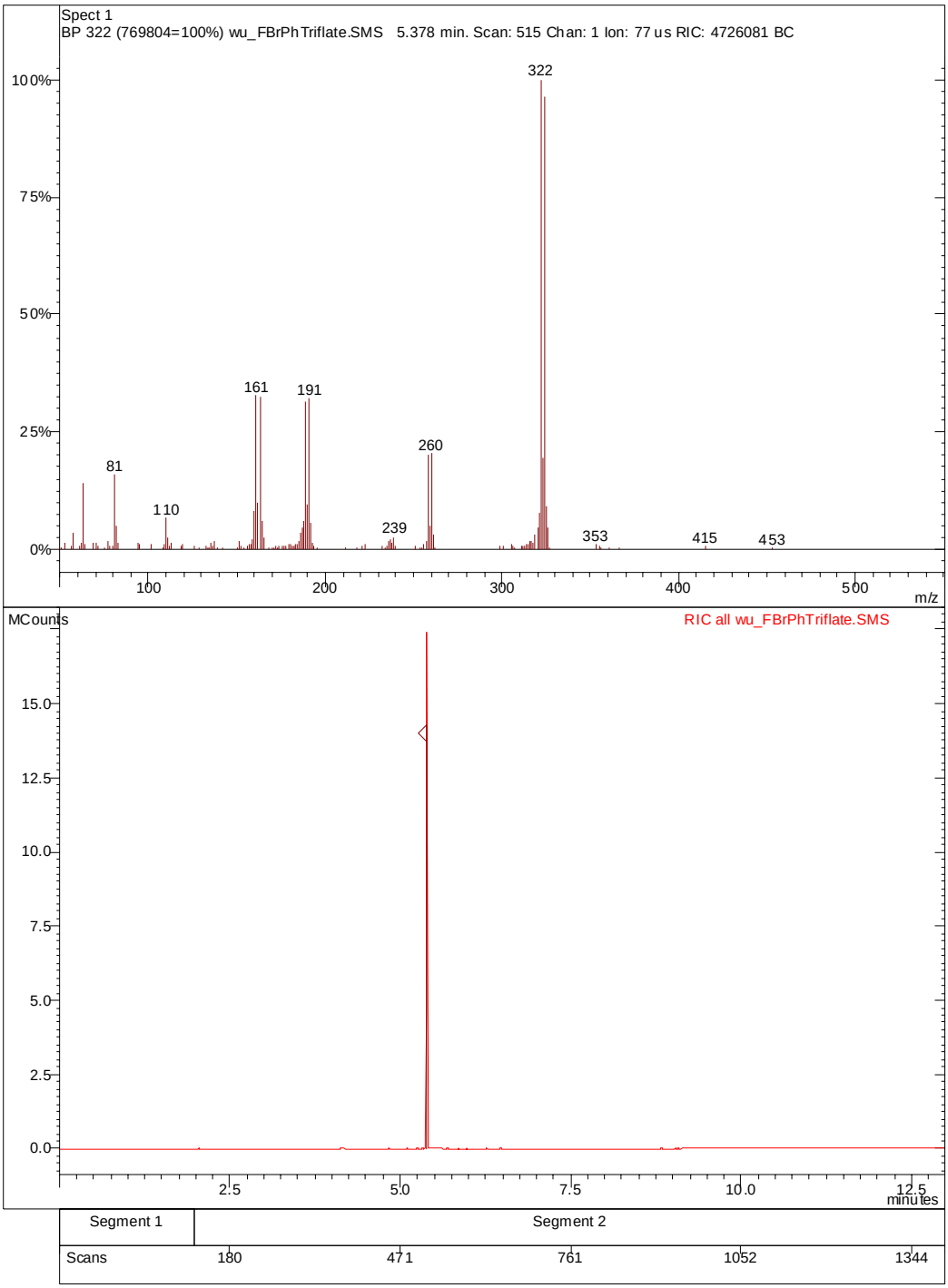
Trifluoro-methanesulfonic acid 2-bromo-5-fluoro-phenyl ester (5b)



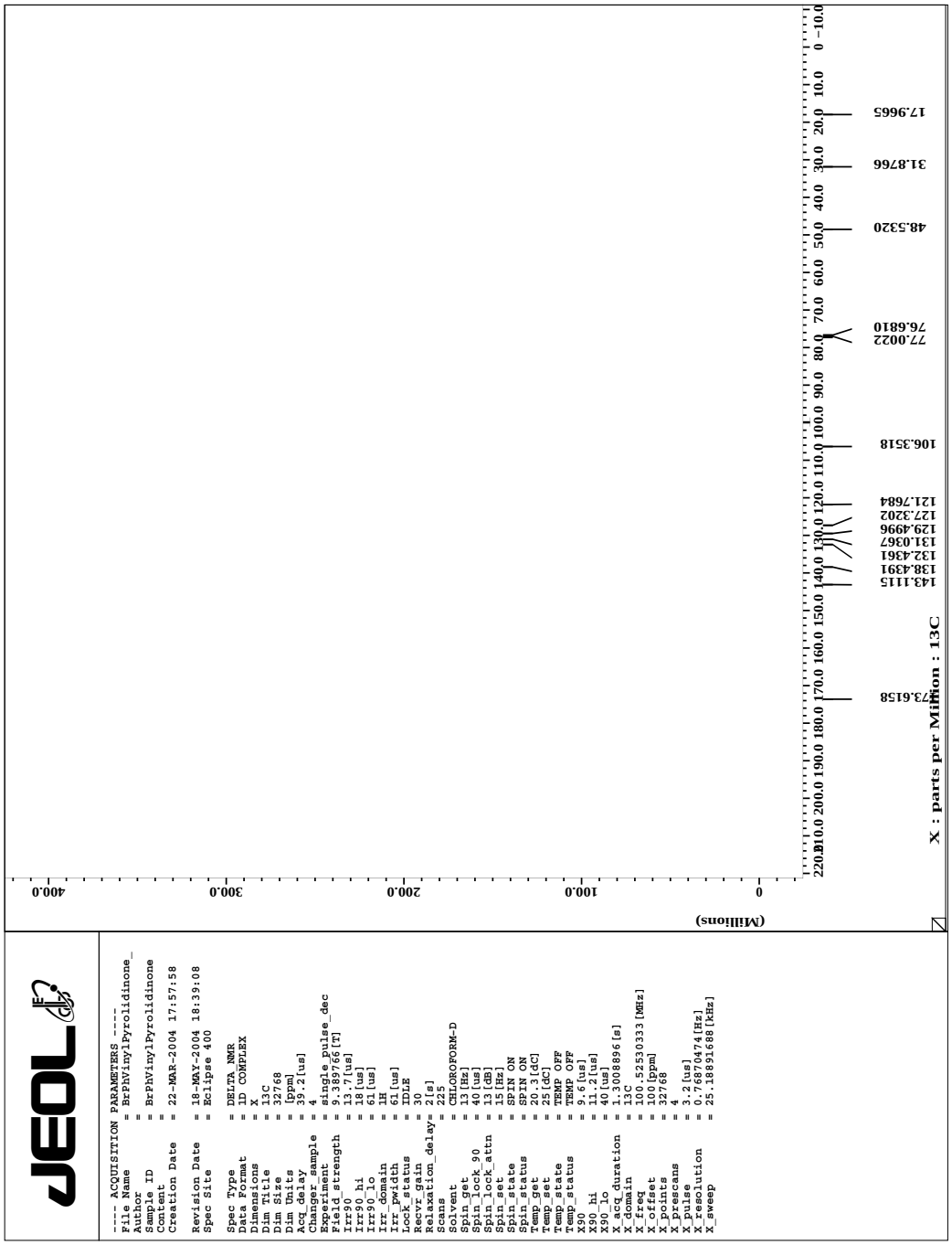
Chromatogram Plot

File: i:\wu_fbrphtriflate.sms
Sample: wu_FBrPhTriflate
Scan Range: 1 - 1400 Time Range: 0.00 - 12.98 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 04/20/04 21:48



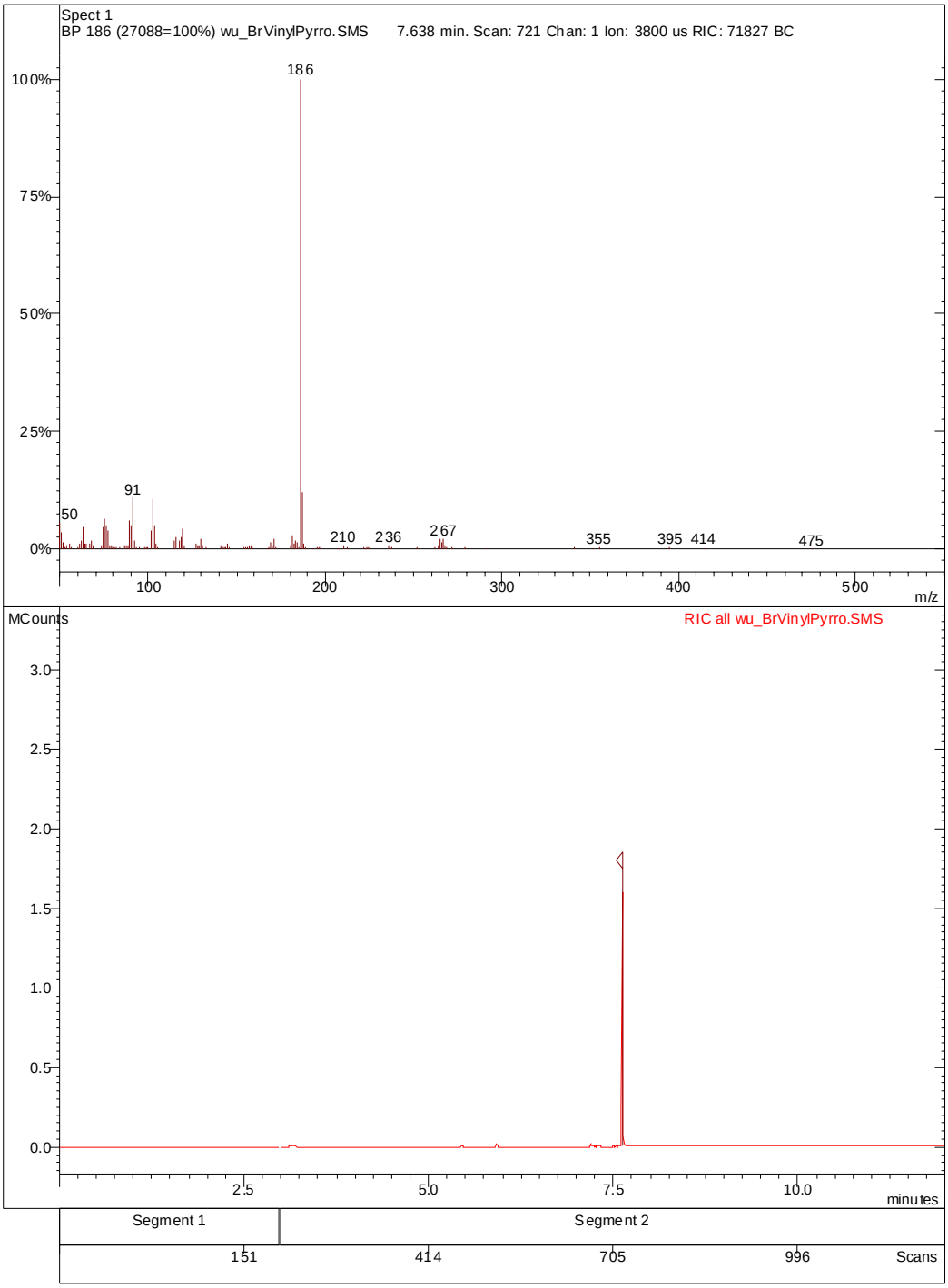
1-[1-(2-Bromo-phenyl)-vinyl]-pyrrolidin-2-one (7a)



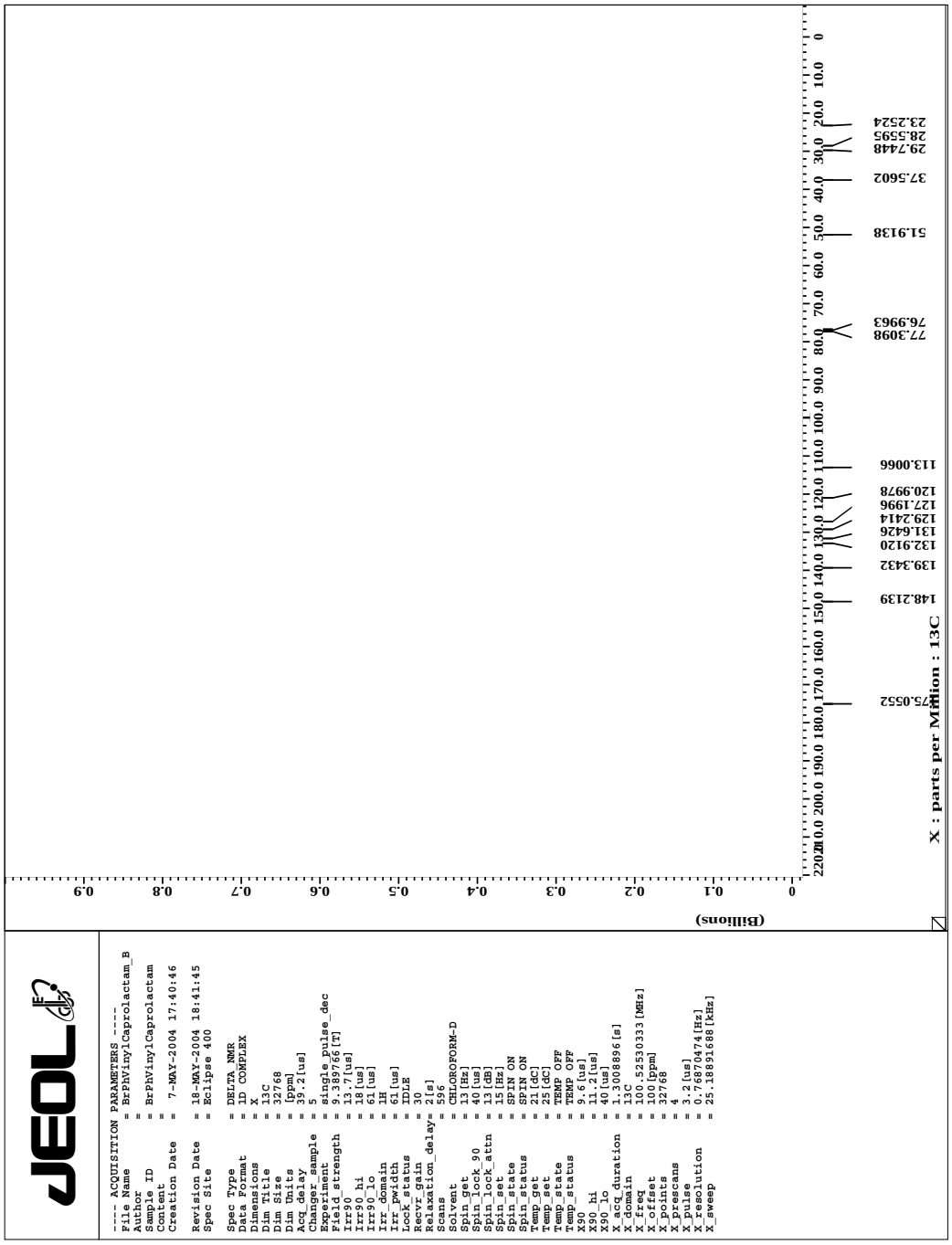
Chromatogram Plot

File: i:\wu_brvinylpyrro.sms
Sample: wu
Scan Range: 1 - 1228 Time Range: 0.00 - 11.99 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 03/22/04 11:11



1-[1-(2-Bromo-phenyl)-vinyl]-azepan-2-one (7b)



Chromatogram Plot

File: i:\wu_brphvinylcaprola.sms
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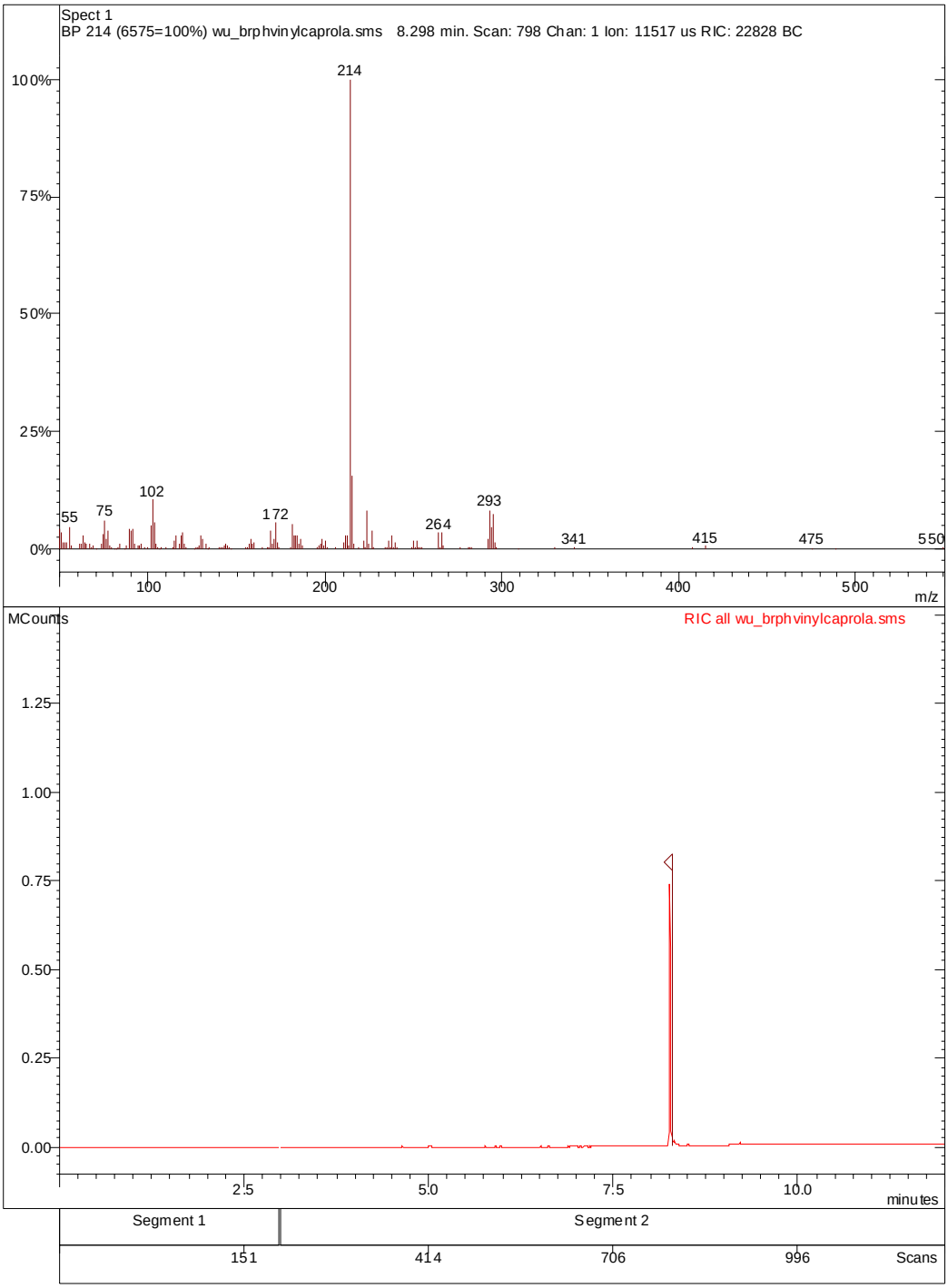
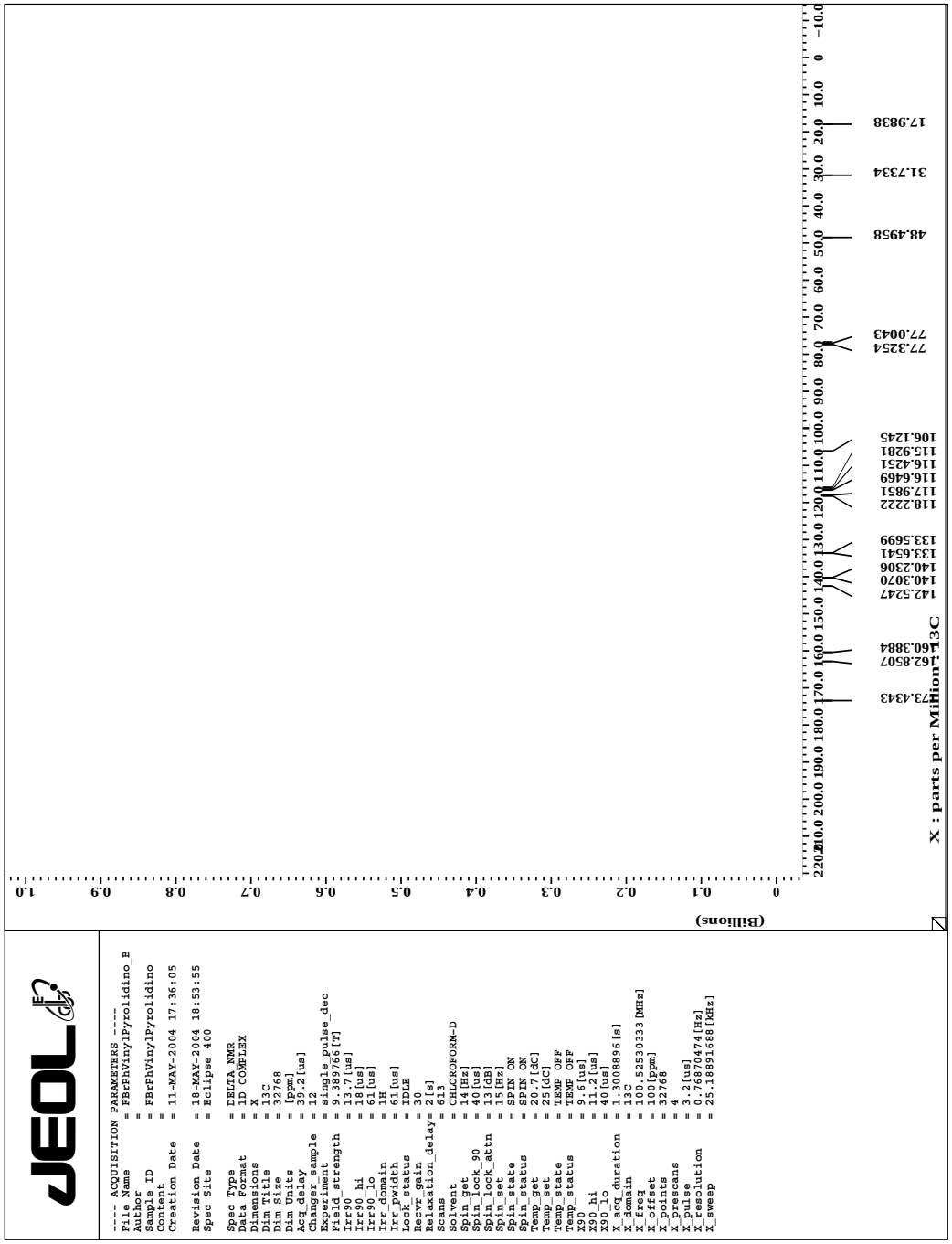
Scan Range: 1 - 1228 Time Range: 0.00 - 11.99 min.
Sample Notes: today

Operator: Operator
Date: 05/07/04 17:45



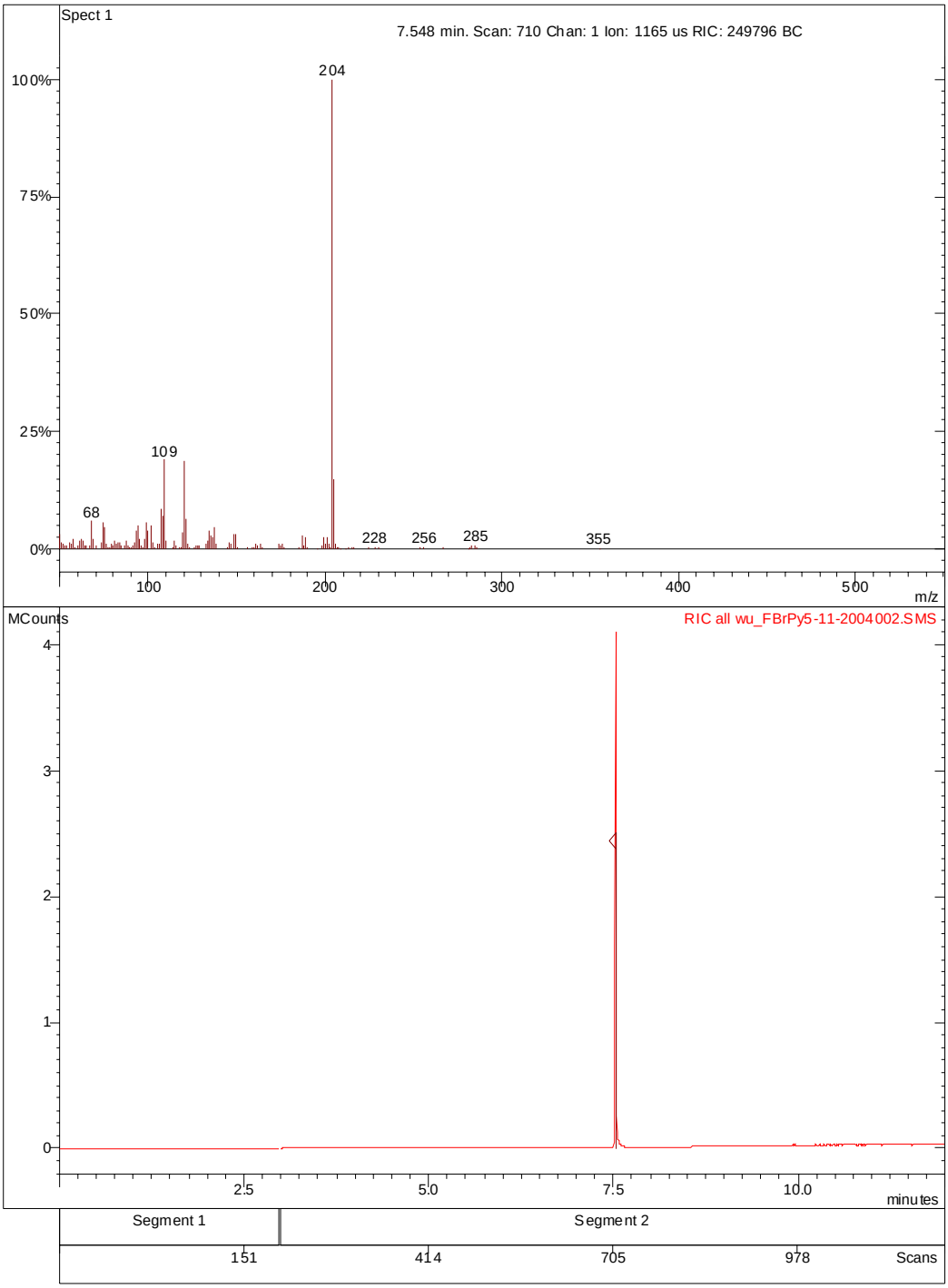
1-[1-(2-Bromo-5-fluoro-phenyl)-vinyl]-pyrrolidin-2-one (7c)



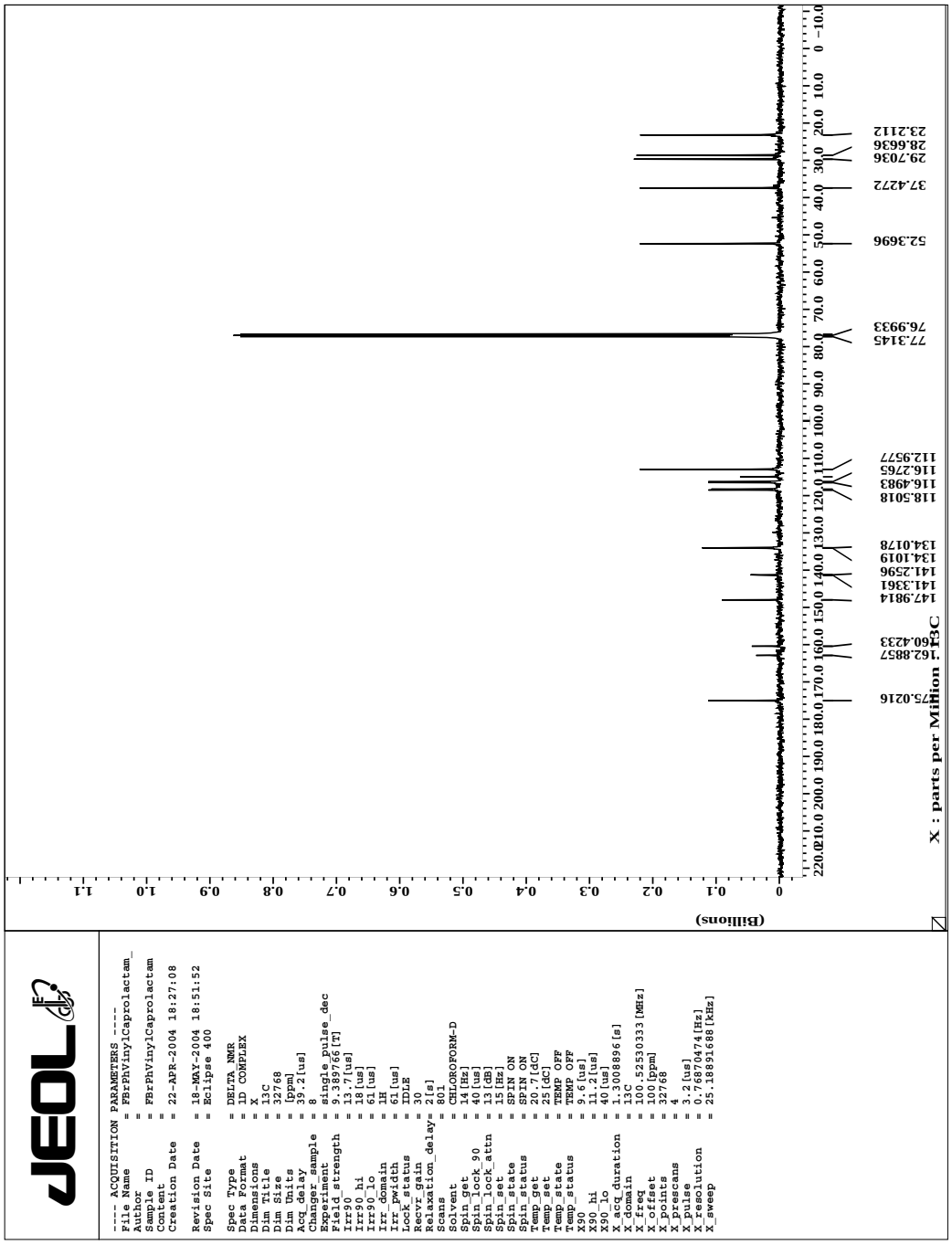
Chromatogram Plot

File: i:\wu_fbrpy5-11-2004002.sms
Sample: wu_FBrPyf1
Scan Range: 1 - 1188 Time Range: 0.00 - 11.99 min.
Sample Notes: Routine

Operator: Org Farm Kemi
Date: 05/11/04 16:00



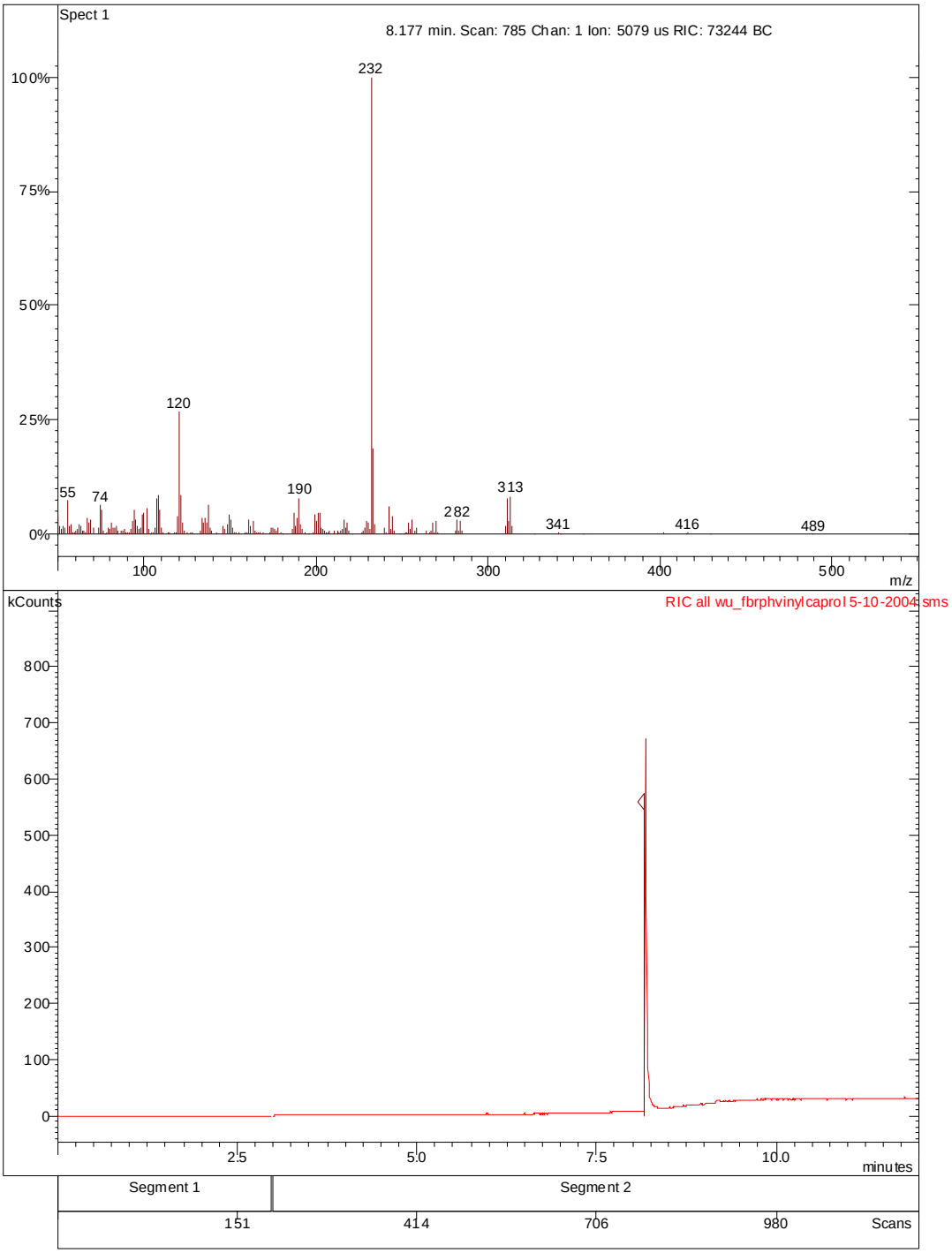
1-[1-(2-Bromo-5-fluoro-phenyl)-vinyl]-azepan-2-one (7d)



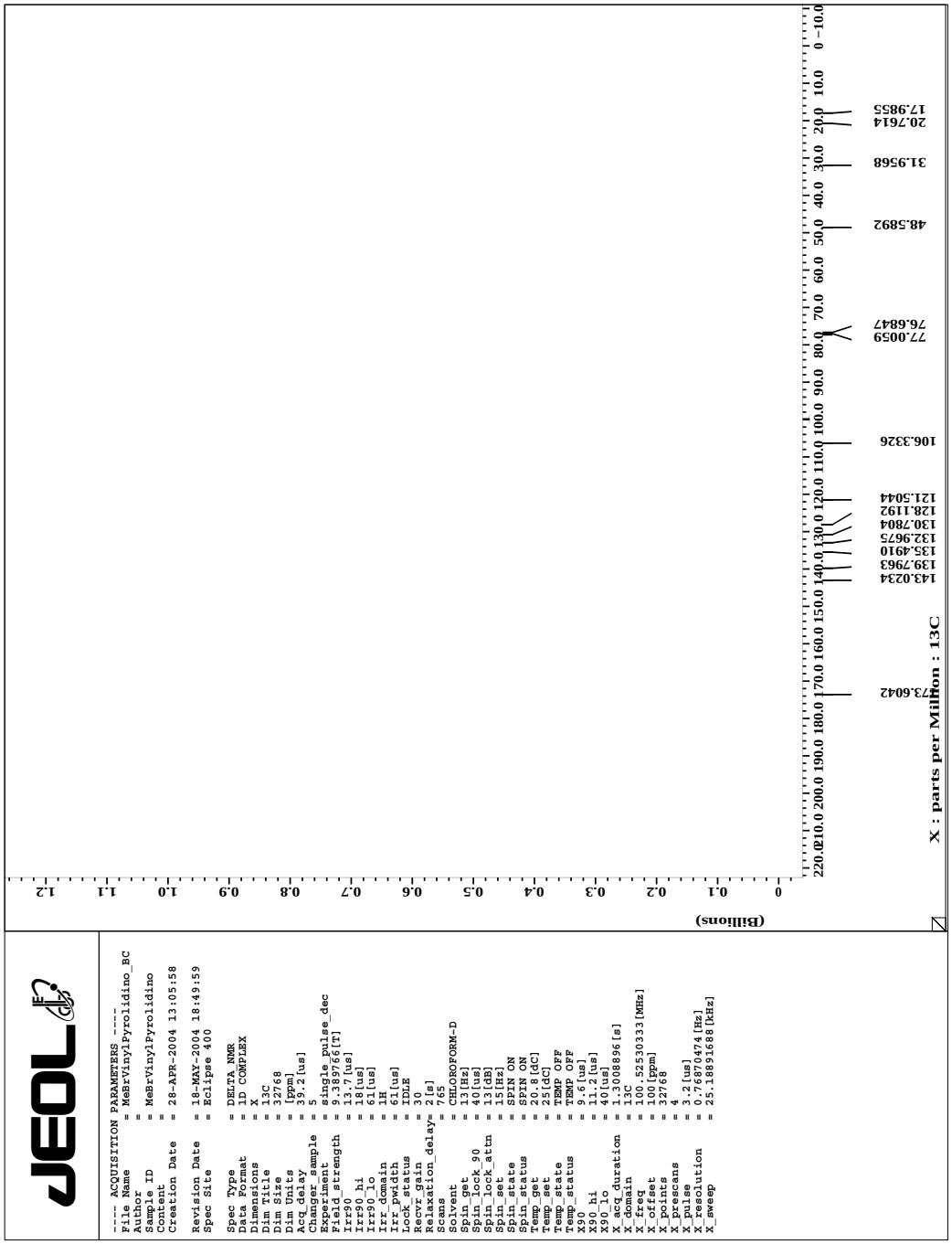
Chromatogram Plot

File: i:\wu_fbrphvinylcaprol 5-10-2004.sms
Sample: wu_FBrPhVinylCaprol
Scan Range: 1 - 1193 Time Range: 0.00 - 11.98 min.
Sample Notes: Routine

Operator: Org Farm Kemi
Date: 05/10/04 22:14



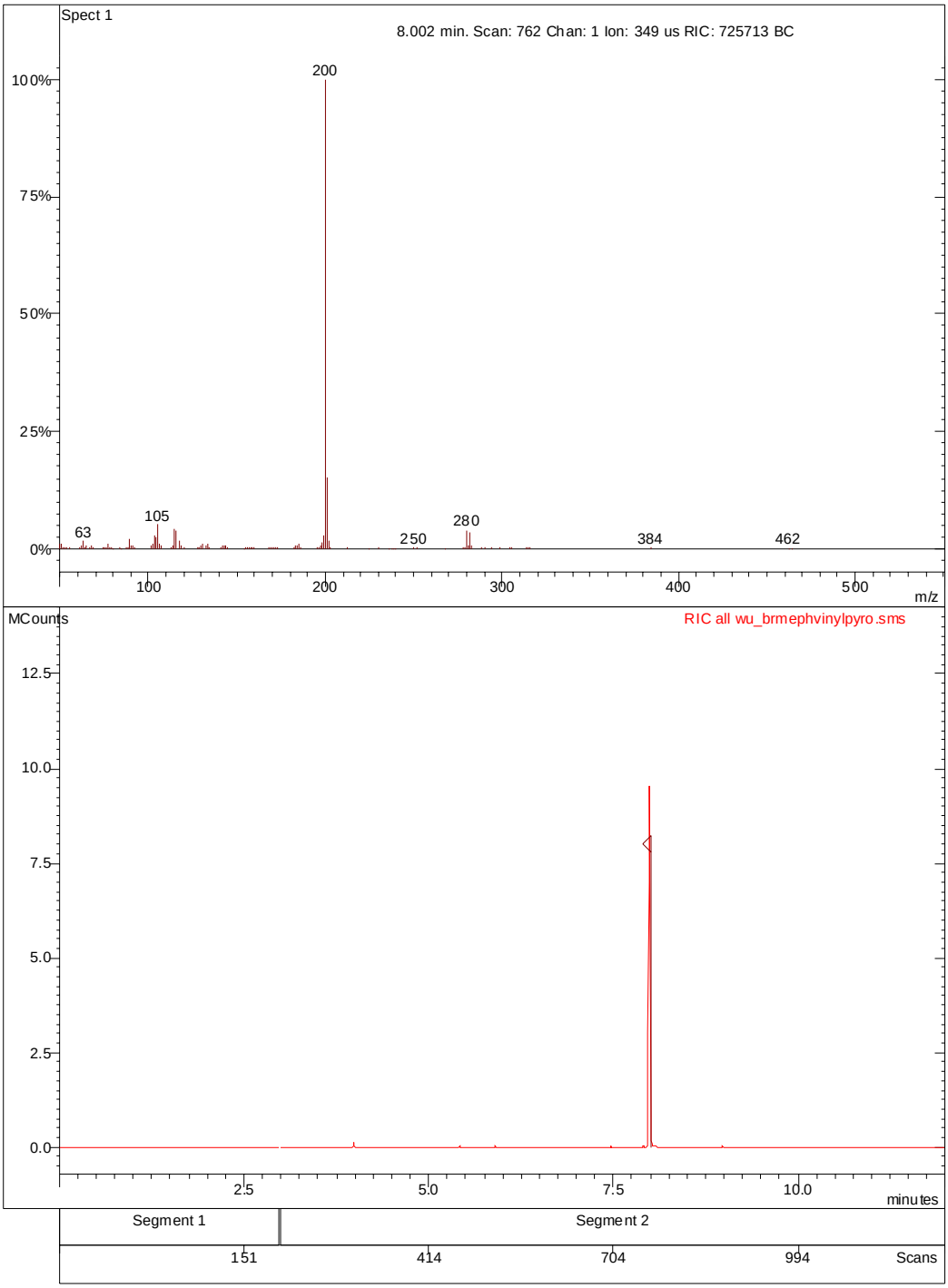
1-[1-(2-Bromo-4-methyl-phenyl)-vinyl] pyrrolidin-2-one (7e)



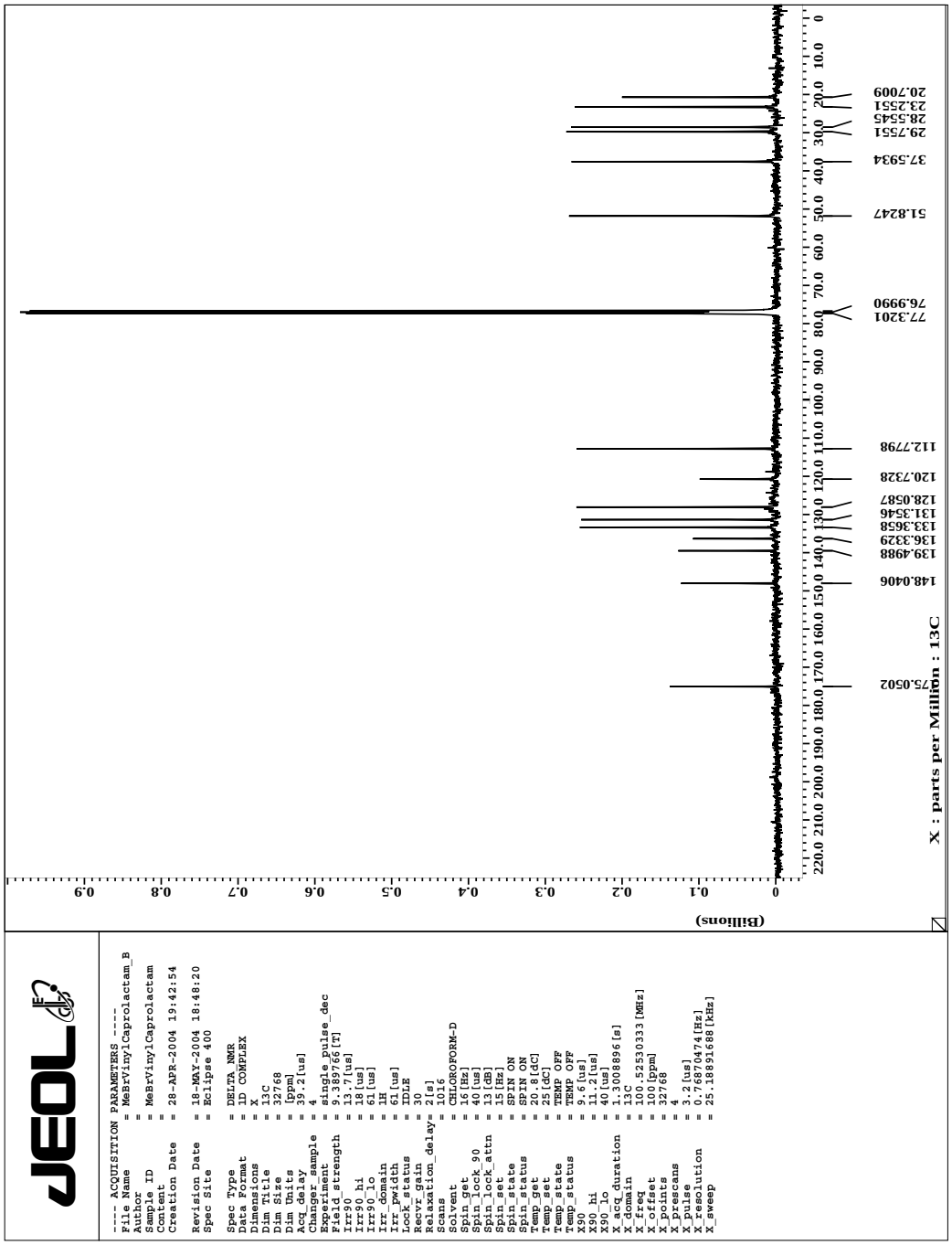
Chromatogram Plot

File: i:\wu_brmephvinylpyro.sms
Sample: wu_BrMePhVinylPyro
Scan Range: 1 - 1225 Time Range: 0.00 - 11.98 min.
Sample Notes: today

Operator: Operator
Date: 04/27/04 22:06



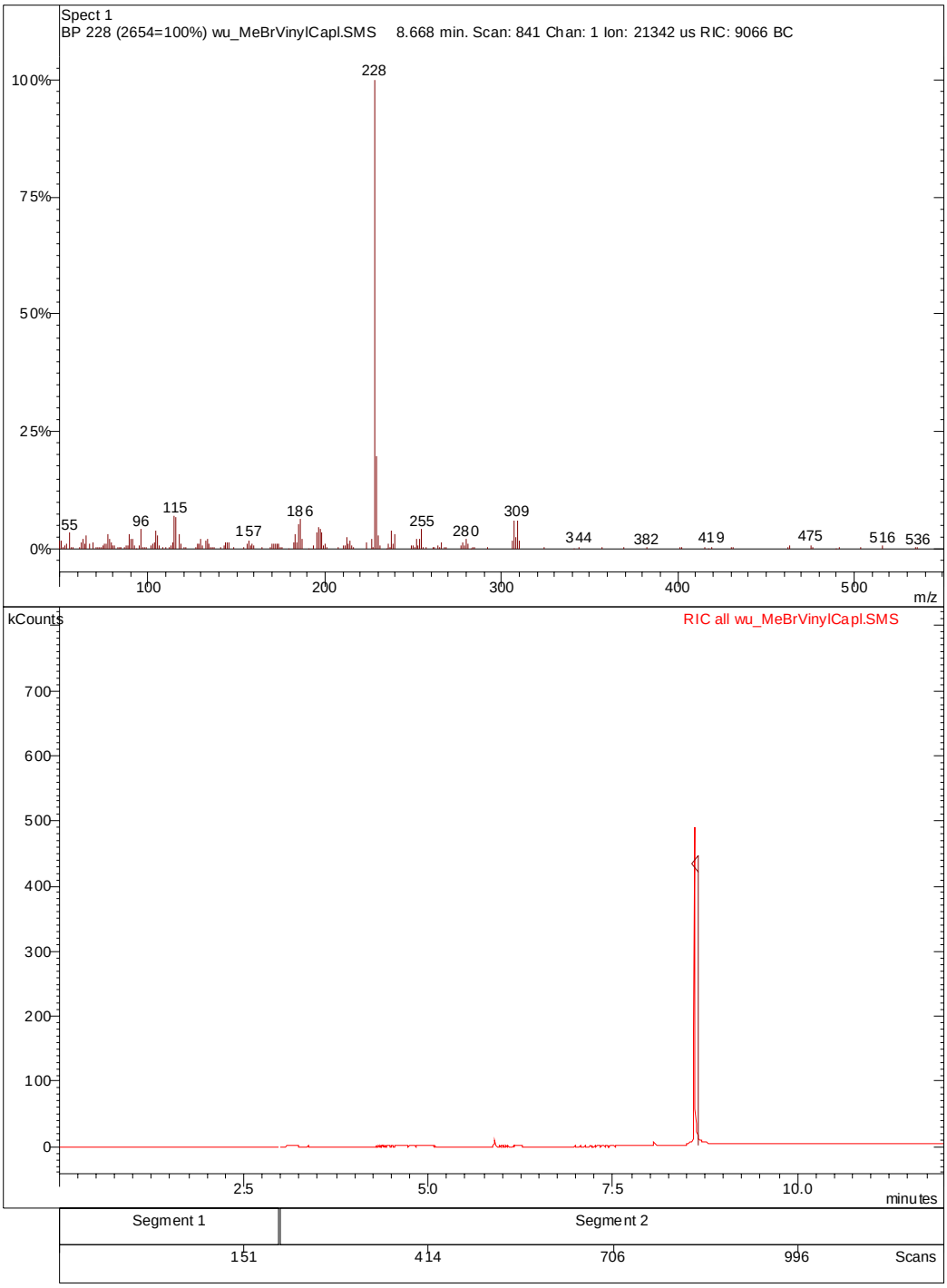
1-[1-(2-Bromo-4-methyl-phenyl)-vinyl]-azepan-2-one (7f)



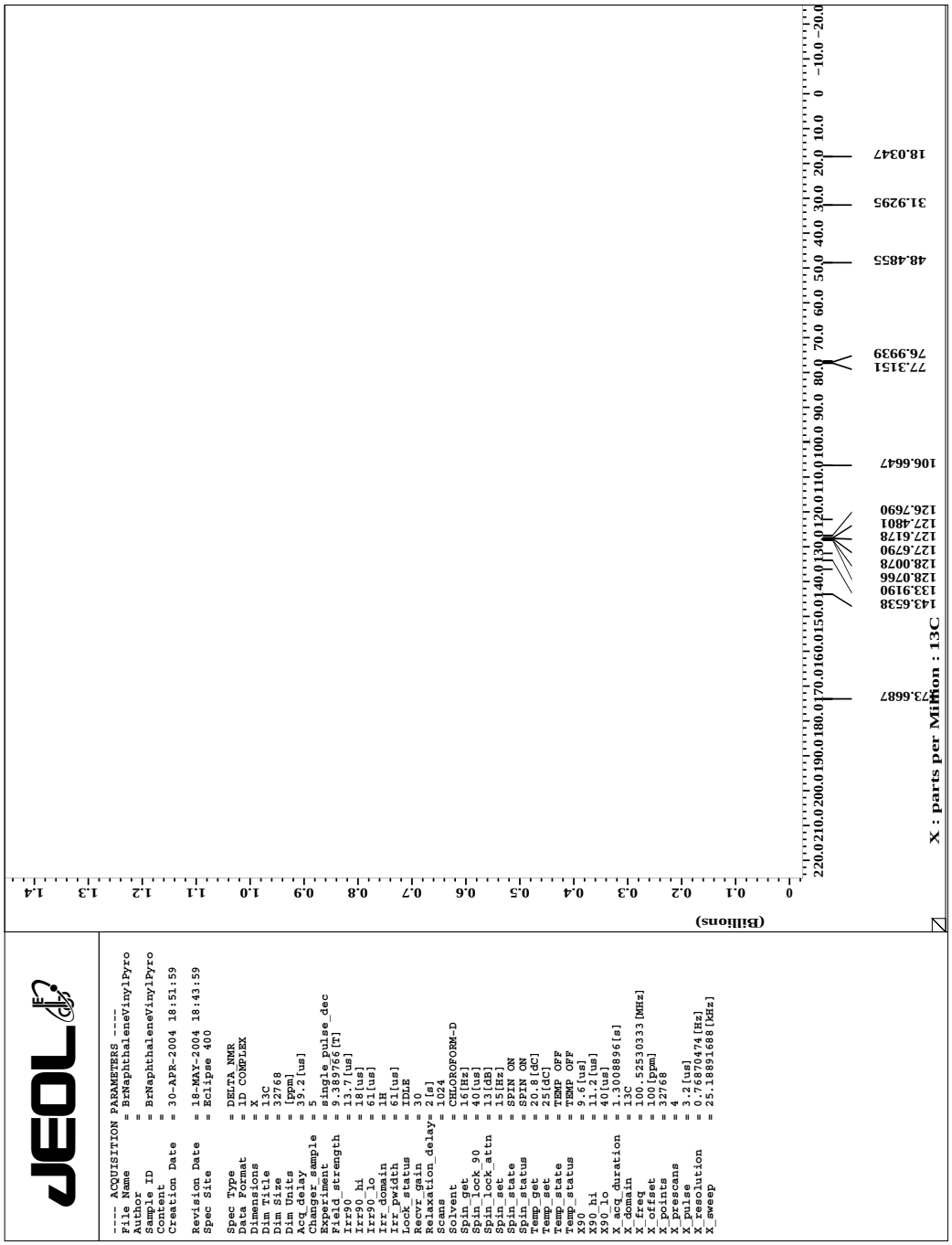
Chromatogram Plot

File: i:\wu_mebrvinylcapl.sms
Sample: wu_MeBrVinylCapl
Scan Range: 1 - 1227 Time Range: 0.00 - 11.98 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 04/28/04 16:00



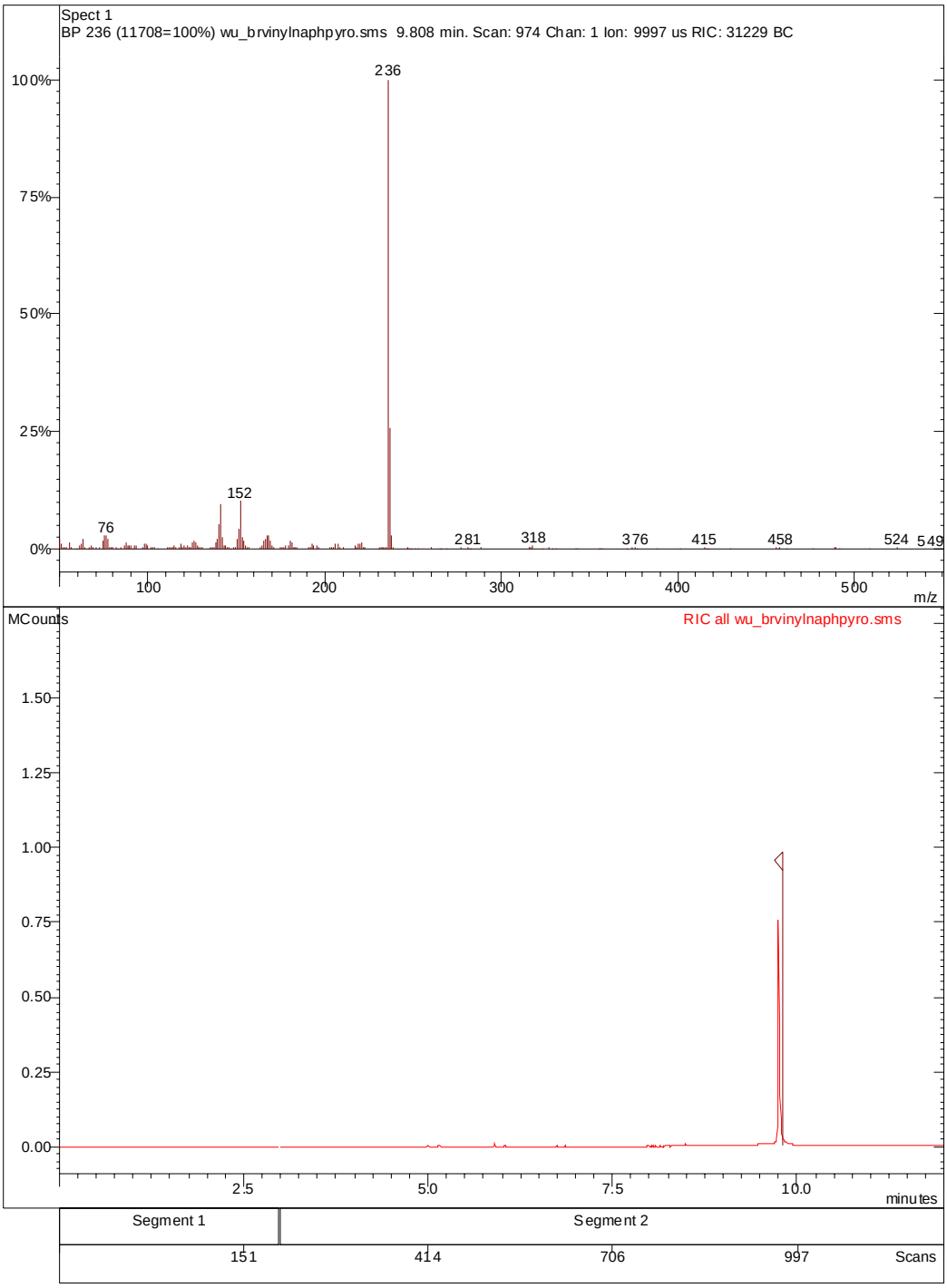
1-[1-(1-Bromo-naphthalen-2-yl)-vinyl]-pyrrolidin-2-one (7g)



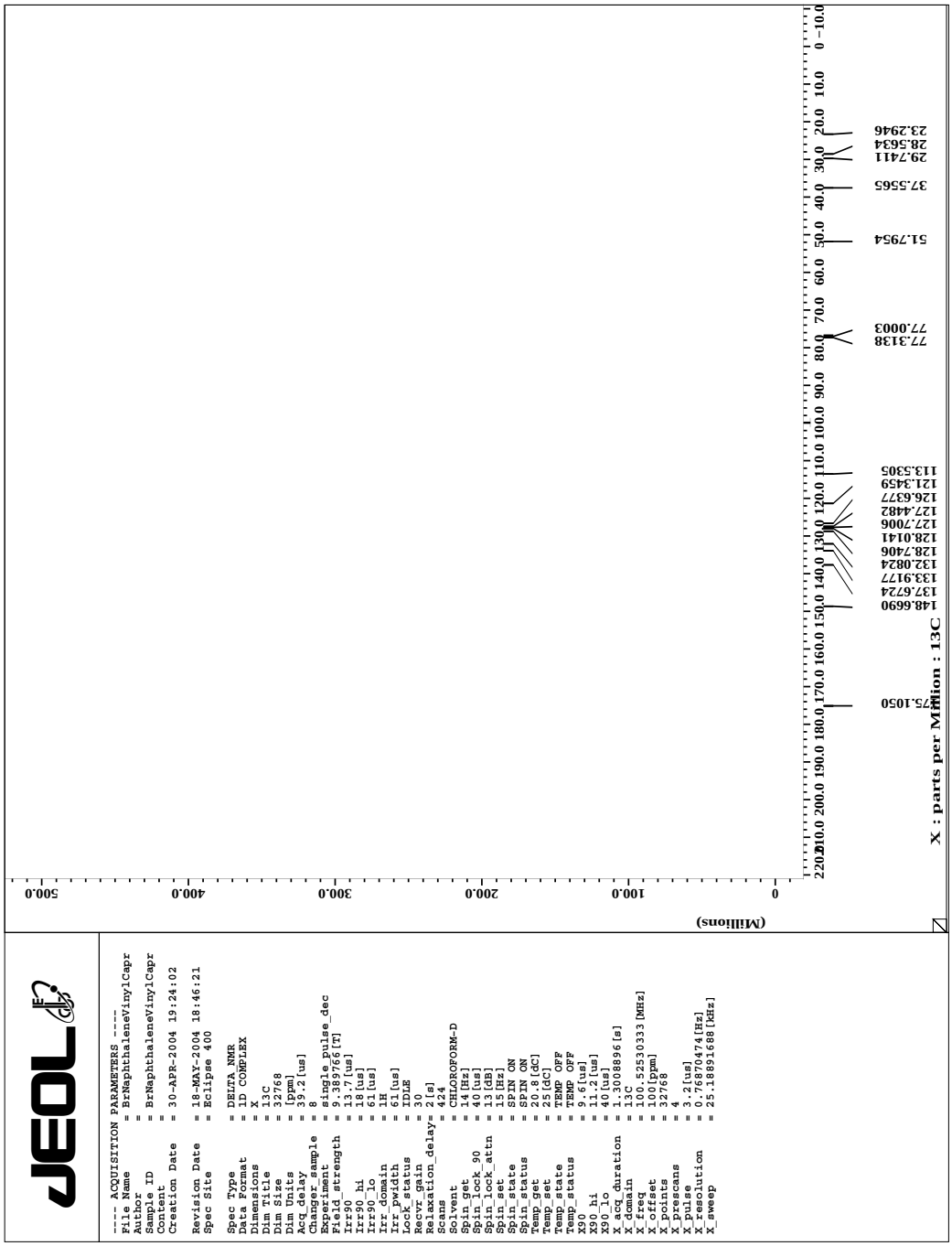
Chromatogram Plot

File: i:\wu_brvinylnaphpyro.sms
Sample: wu_BrVinylNaphPyro
Scan Range: 1 - 1228 Time Range: 0.00 - 11.99 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 04/29/04 23:34



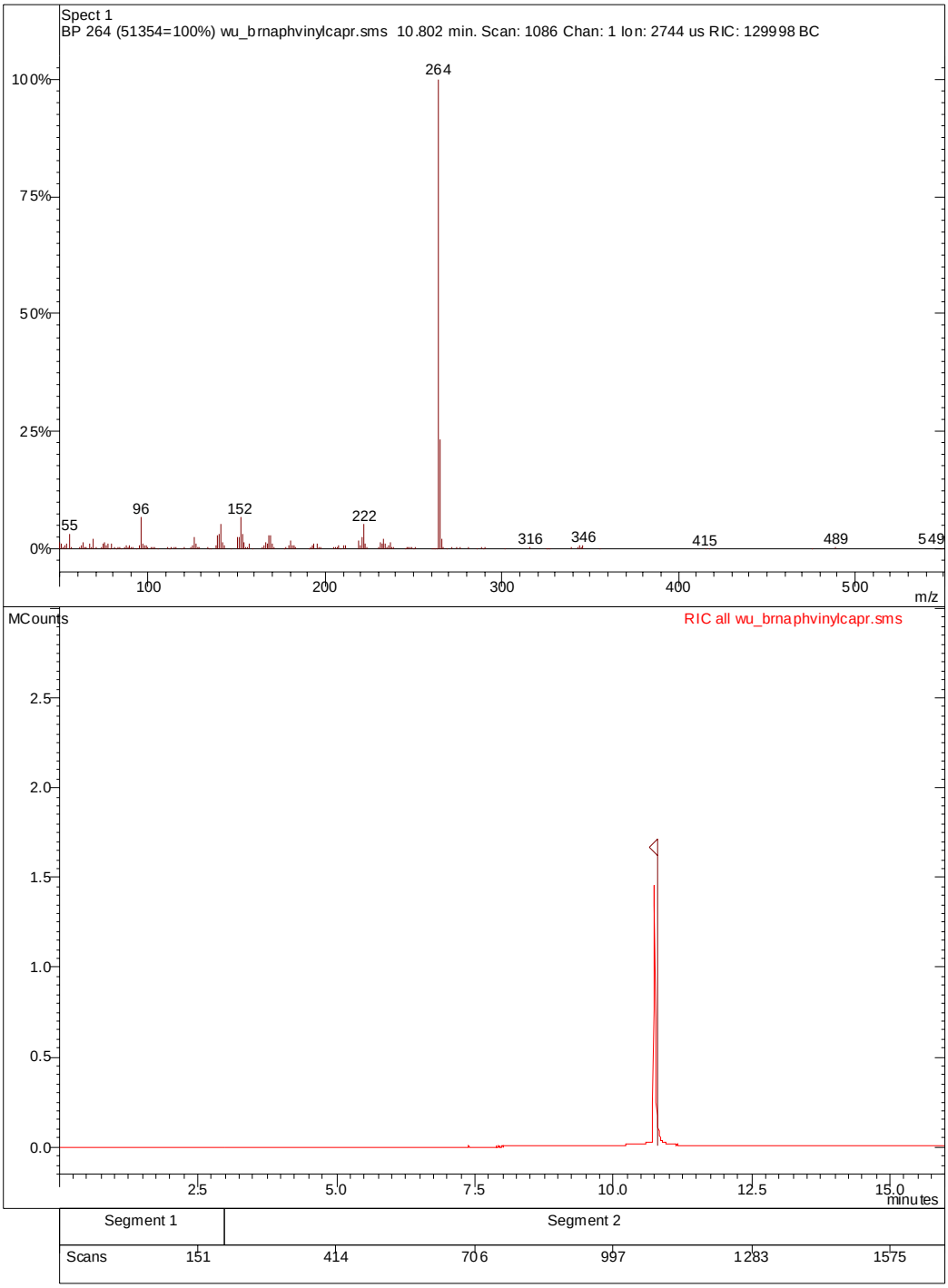
1-[1-(1-Bromo-naphthalen-2-yl)-vinyl]-azepan-2-one (7h)



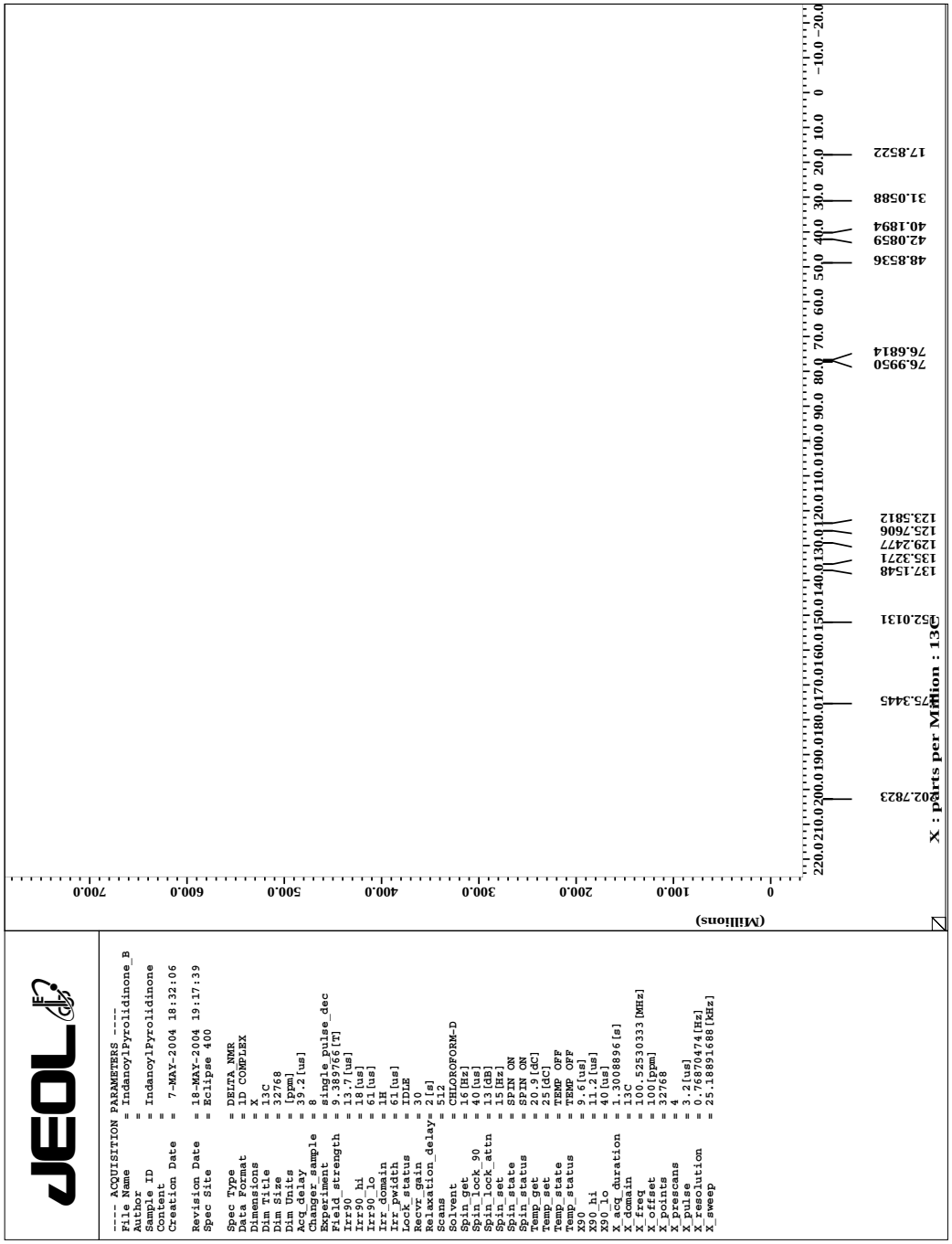
Chromatogram Plot

File: i:\wu_brnaphvinylcapr.sms
Sample: wu_BrNaphVinylCapr
Scan Range: 1 - 1689 Time Range: 0.00 - 15.98 min.
Sample Notes: today

Operator: Operator
Date: 05/06/04 18:16



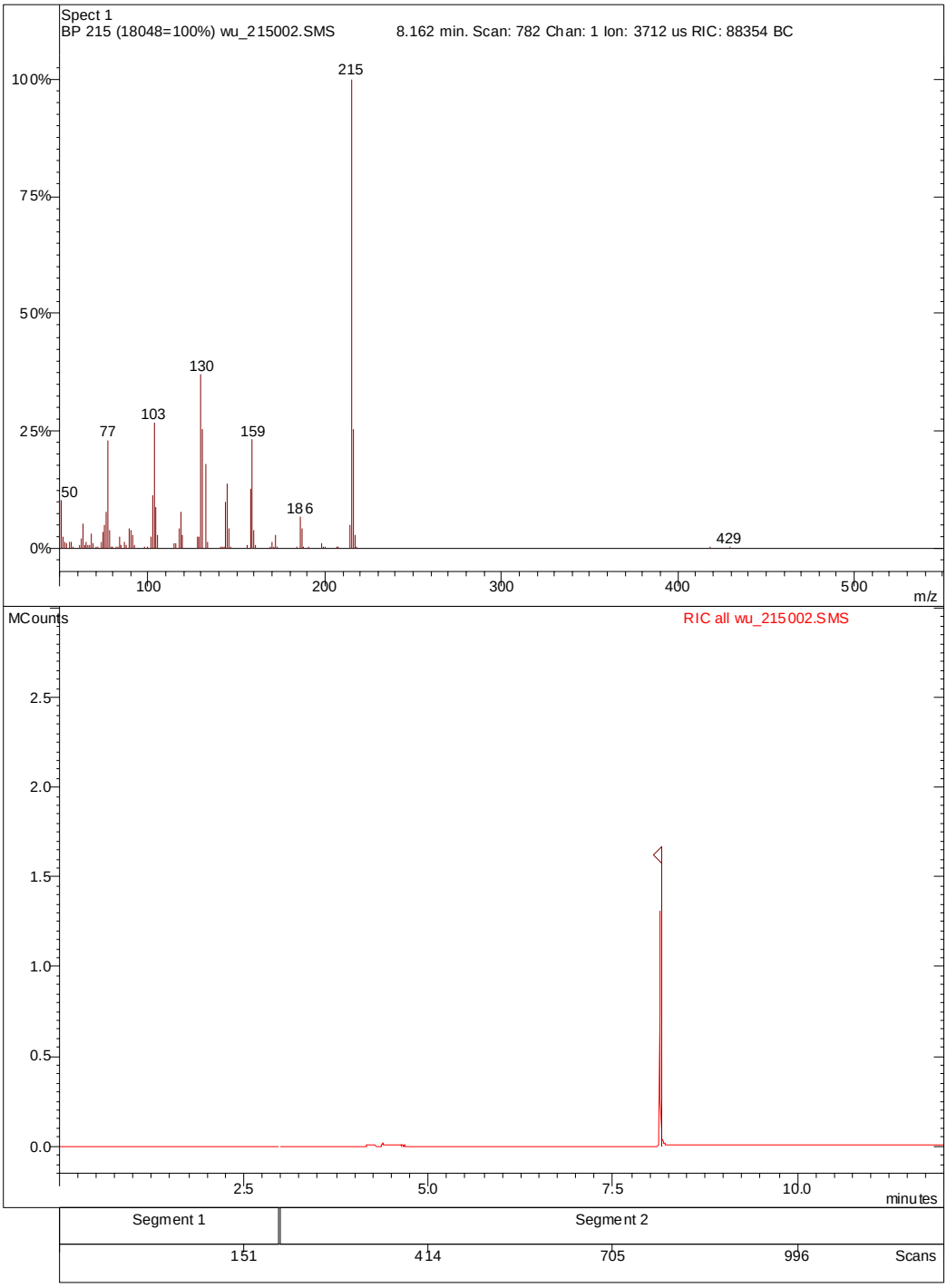
1-(3-Oxo-indan-1-yl)-pyrrolidin-one (8a)



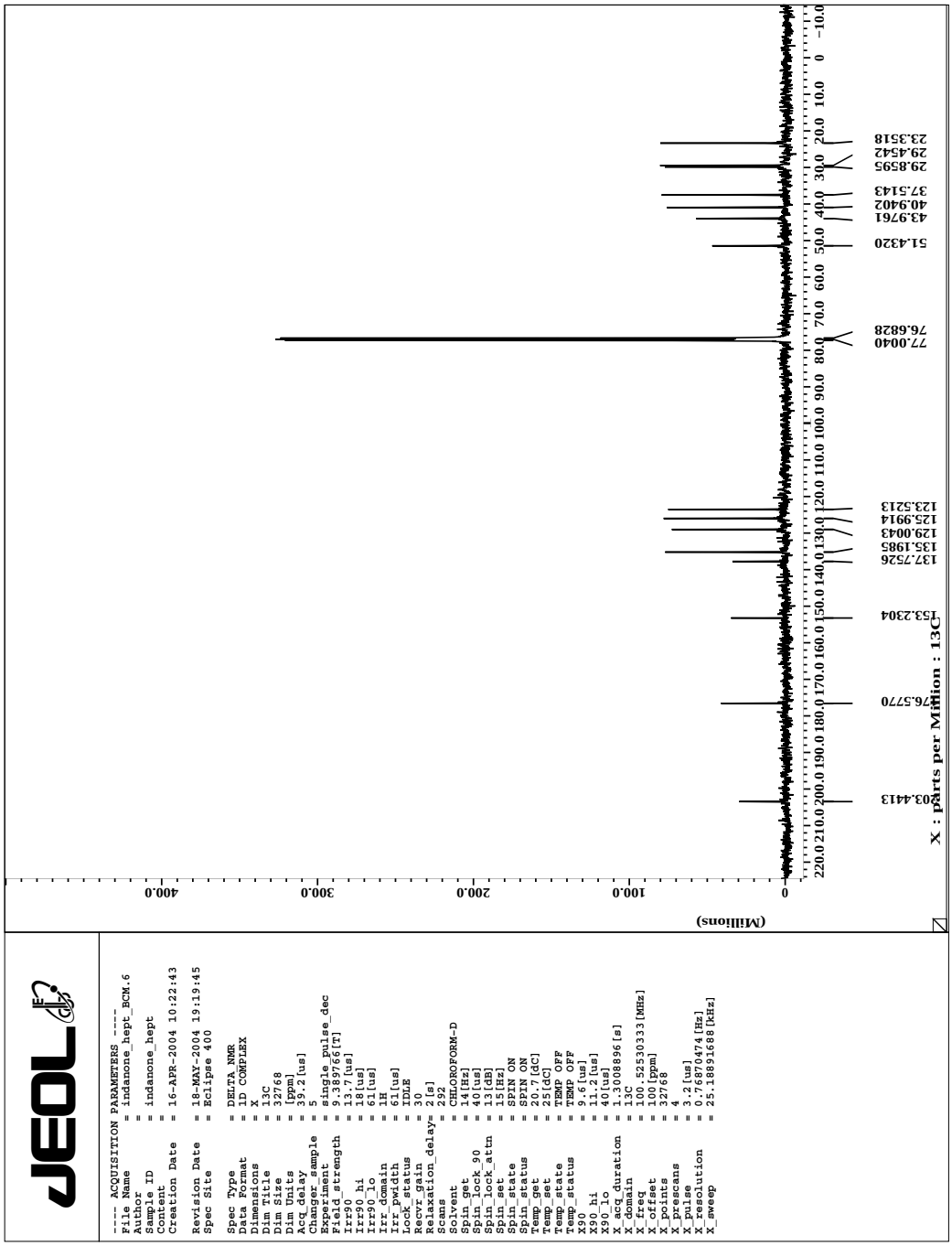
Chromatogram Plot

File: i:\wu_215002.sms
Sample: wu_215
Scan Range: 1 - 1227 Time Range: 0.00 - 11.98 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 03/24/04 13:15



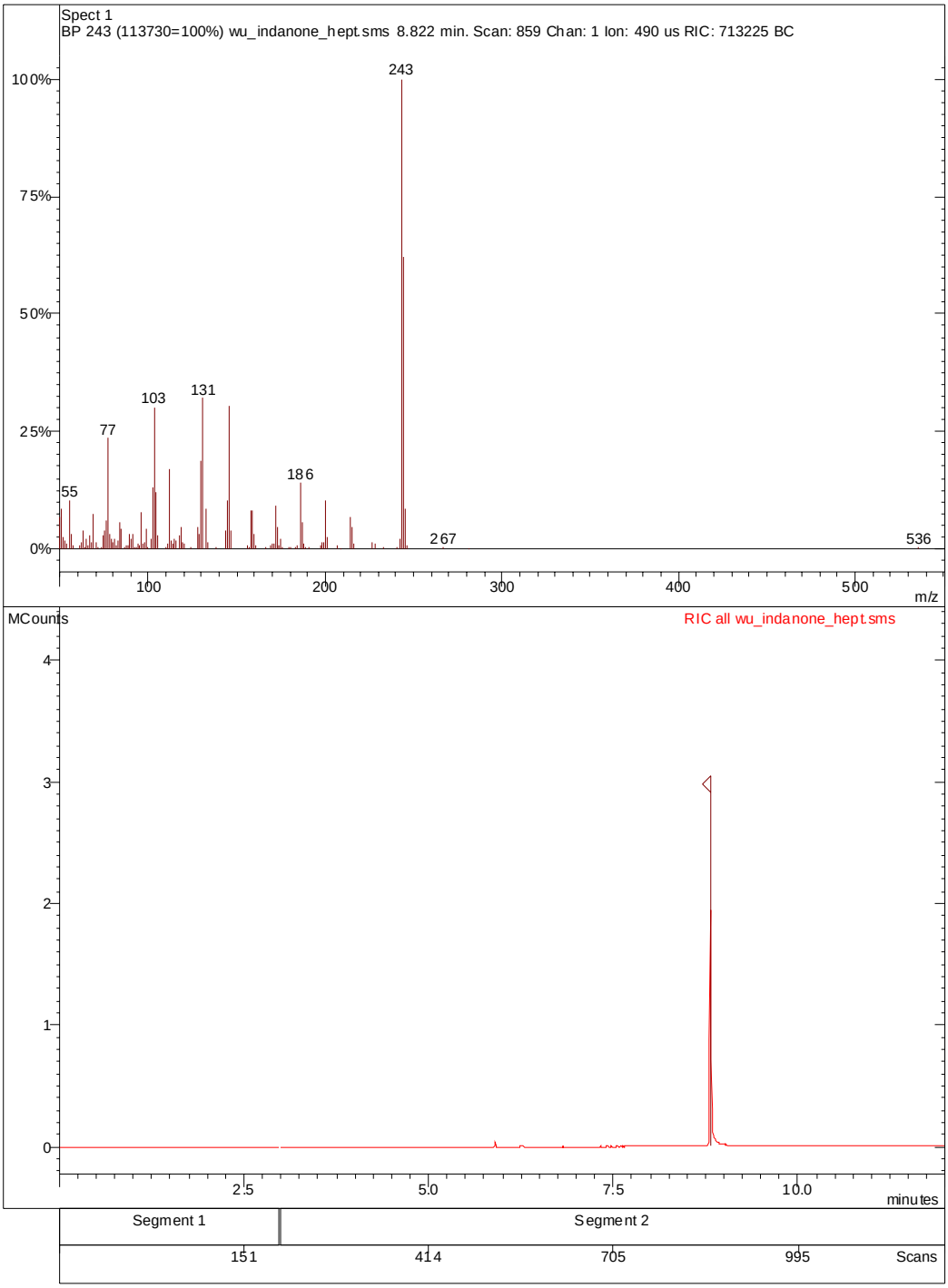
1-(3-Oxo-indan-1-yl)-azepan-one (8b)



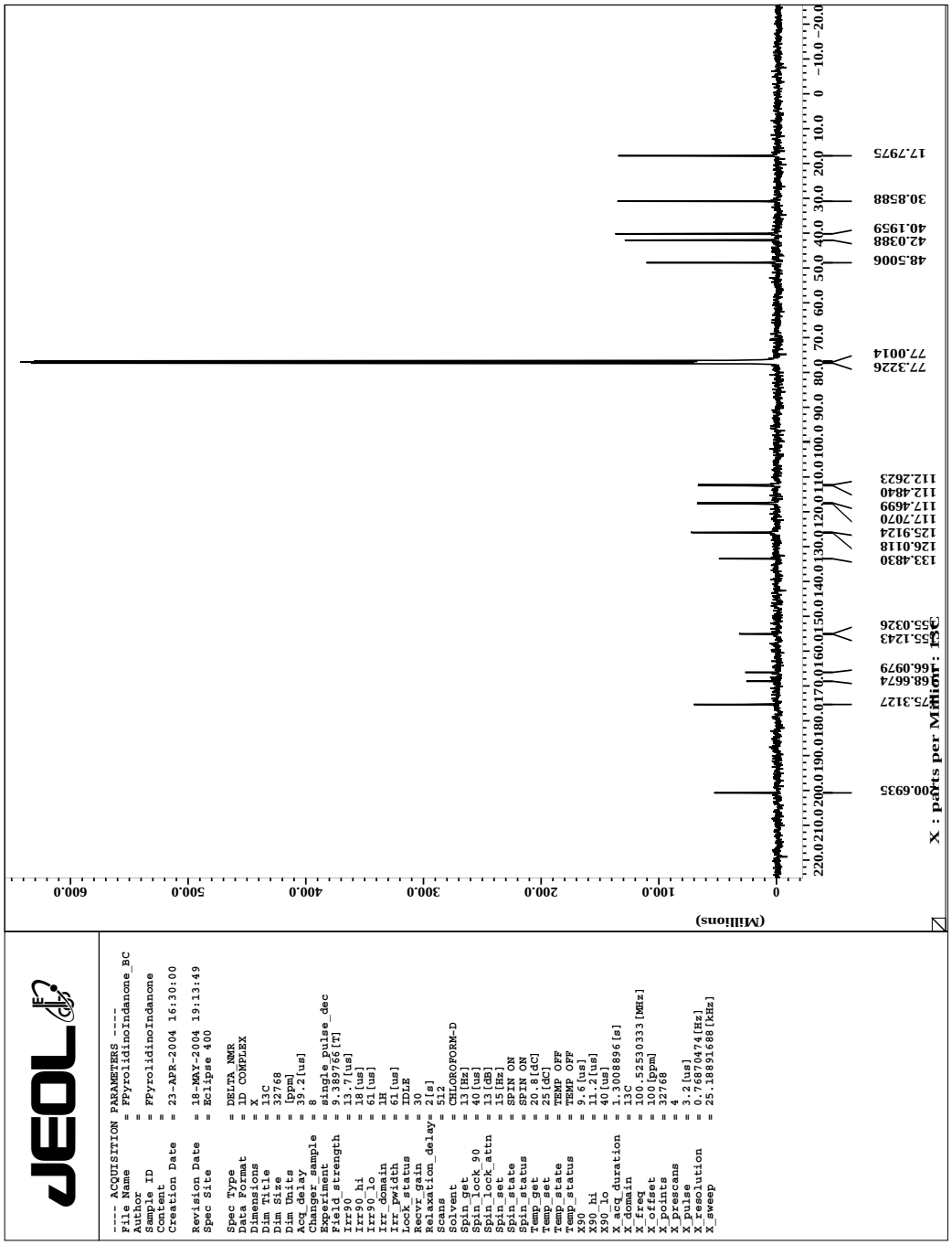
Chromatogram Plot

File: i:\wu_indanone_hept.sms
Sample: wu_indanone_hept
Scan Range: 1 - 1227 Time Range: 0.00 - 11.99 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 04/16/04 13:01



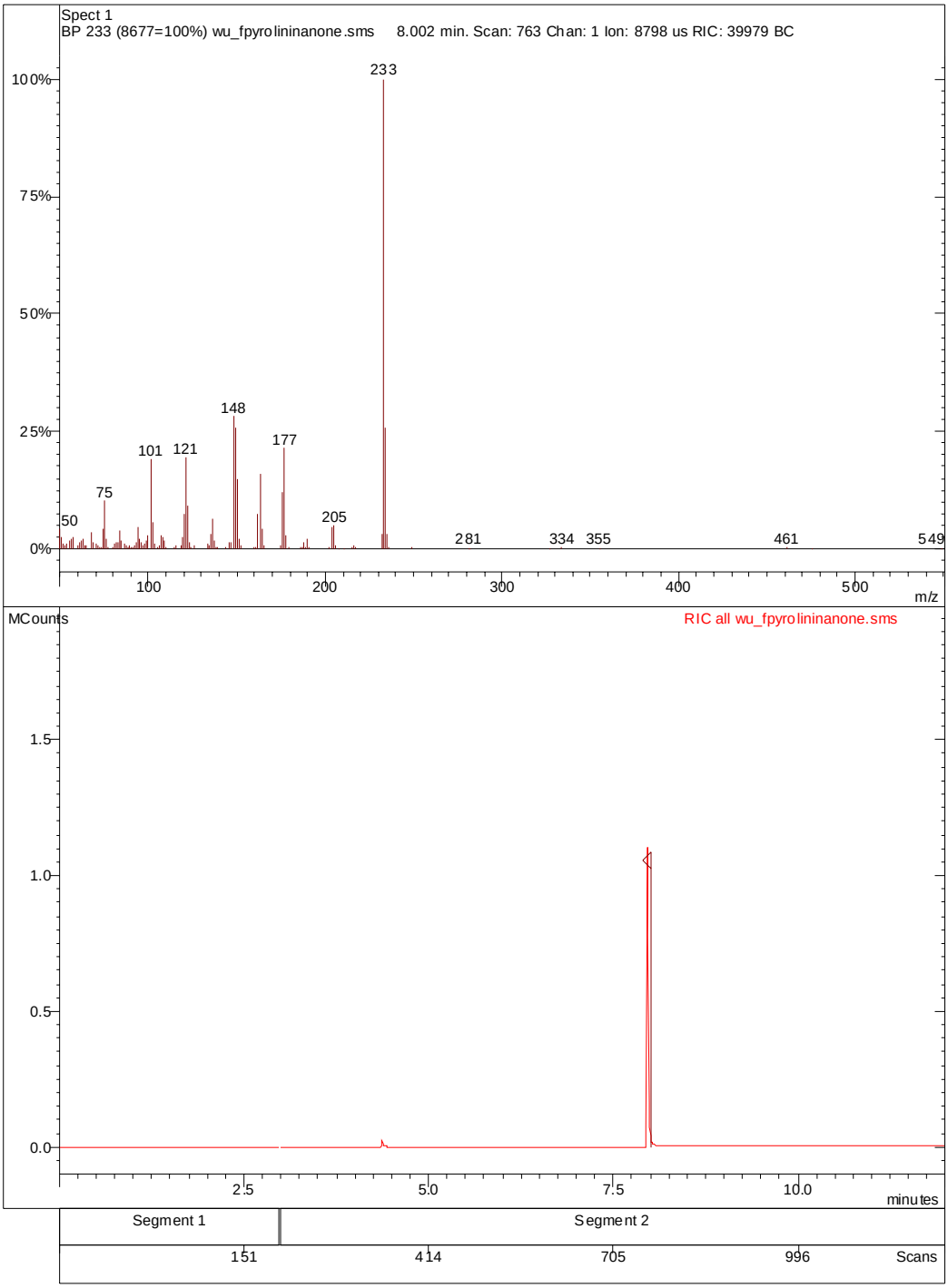
1-(6-Fluoro-3-oxo-indan-1-yl)-pyrrolidin-one (8c)



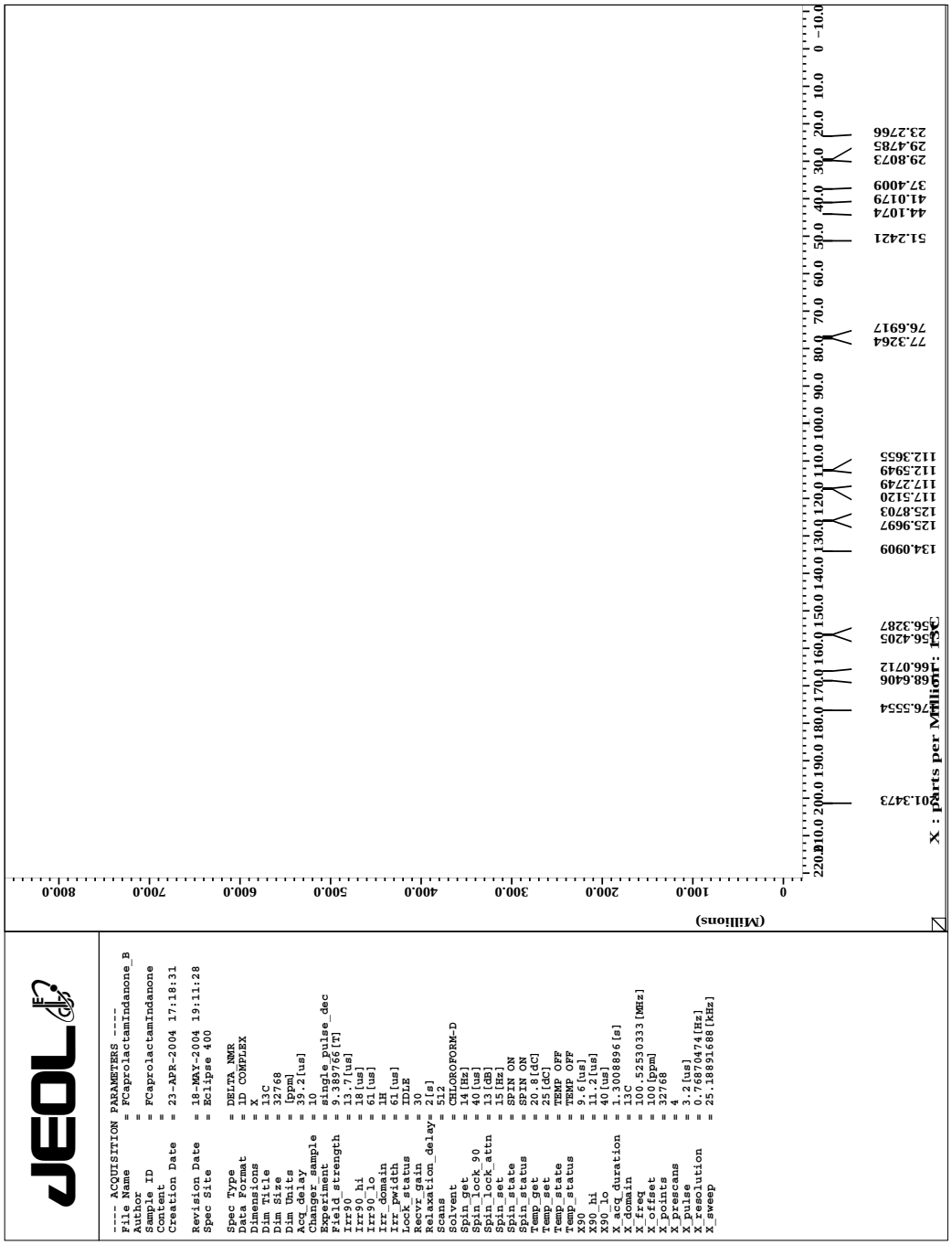
Chromatogram Plot

File: i:\wu_fpyrolininanone.sms
Sample: wu_FPyrolinInanone
Scan Range: 1 - 1227 Time Range: 0.00 - 11.99 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 04/23/04 19:59



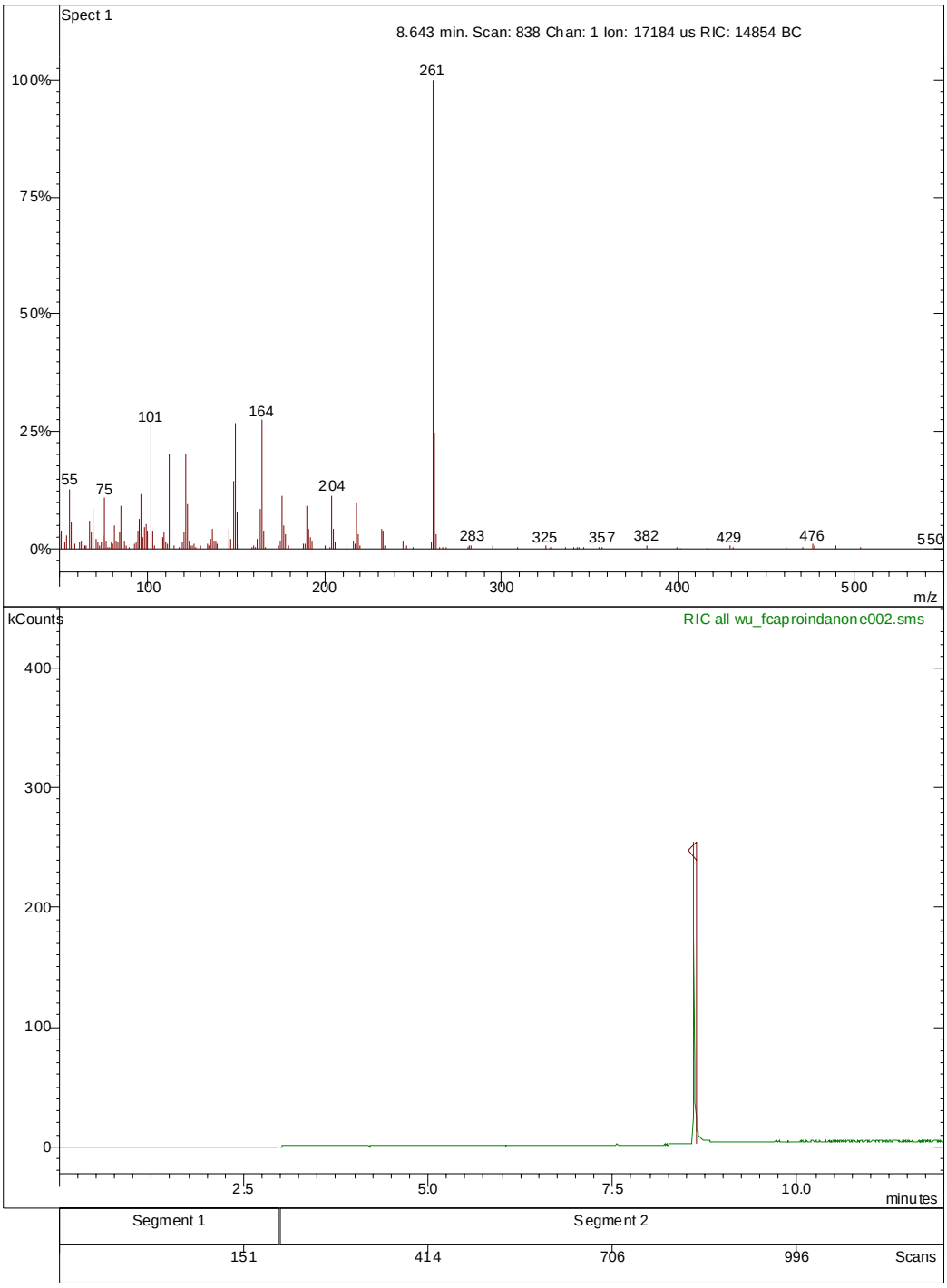
1-(6-Fluoro-3-oxo-indan-1-yl)-azepan-one (8d)



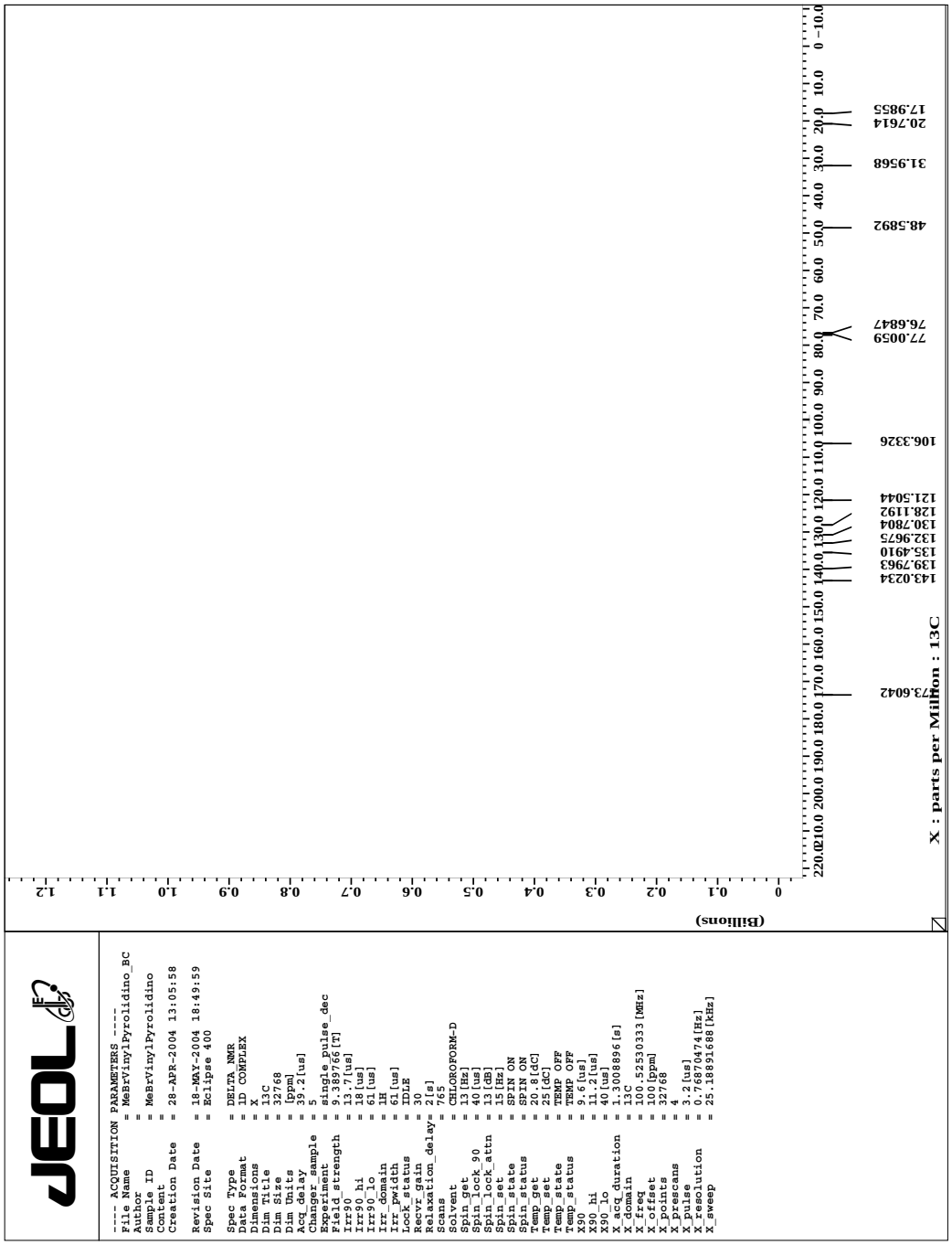
Chromatogram Plot

File: i:\wu_fcaproindanone002.sms
Sample: wu_FCaproIndanone
Scan Range: 1 - 1228 Time Range: 0.00 - 11.99 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 04/25/04 12:46



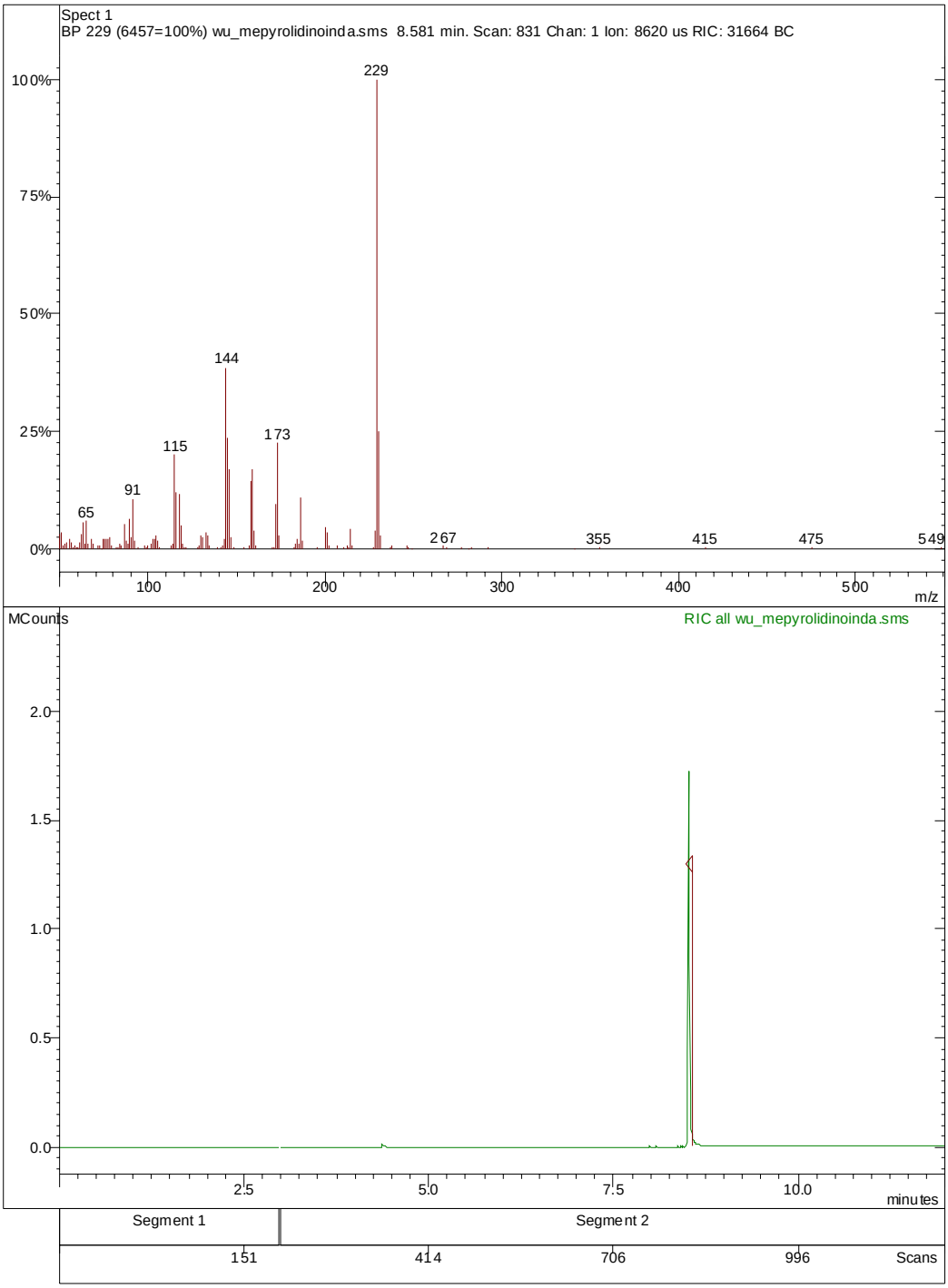
1-(5-methyl-3-oxo-indan-1-yl)-pyrrolidin-one (8e)



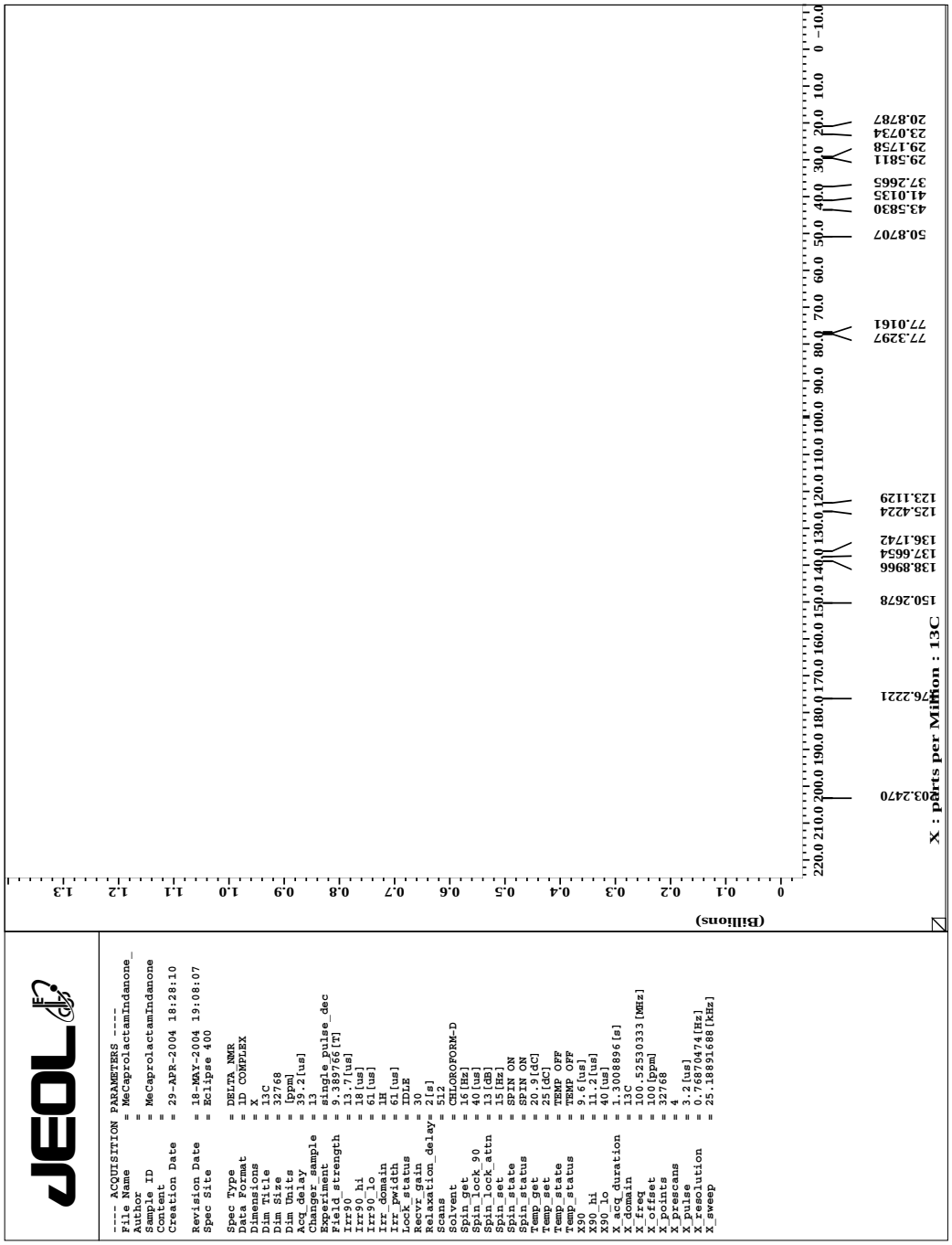
Chromatogram Plot

File: i:\wu_mepyrolidinoinda.sms
Sample: wu_MePyrolidinoInda
Scan Range: 1 - 1227 Time Range: 0.00 - 11.98 min.
Sample Notes: today

Operator: Operator
Date: 04/29/04 20:35



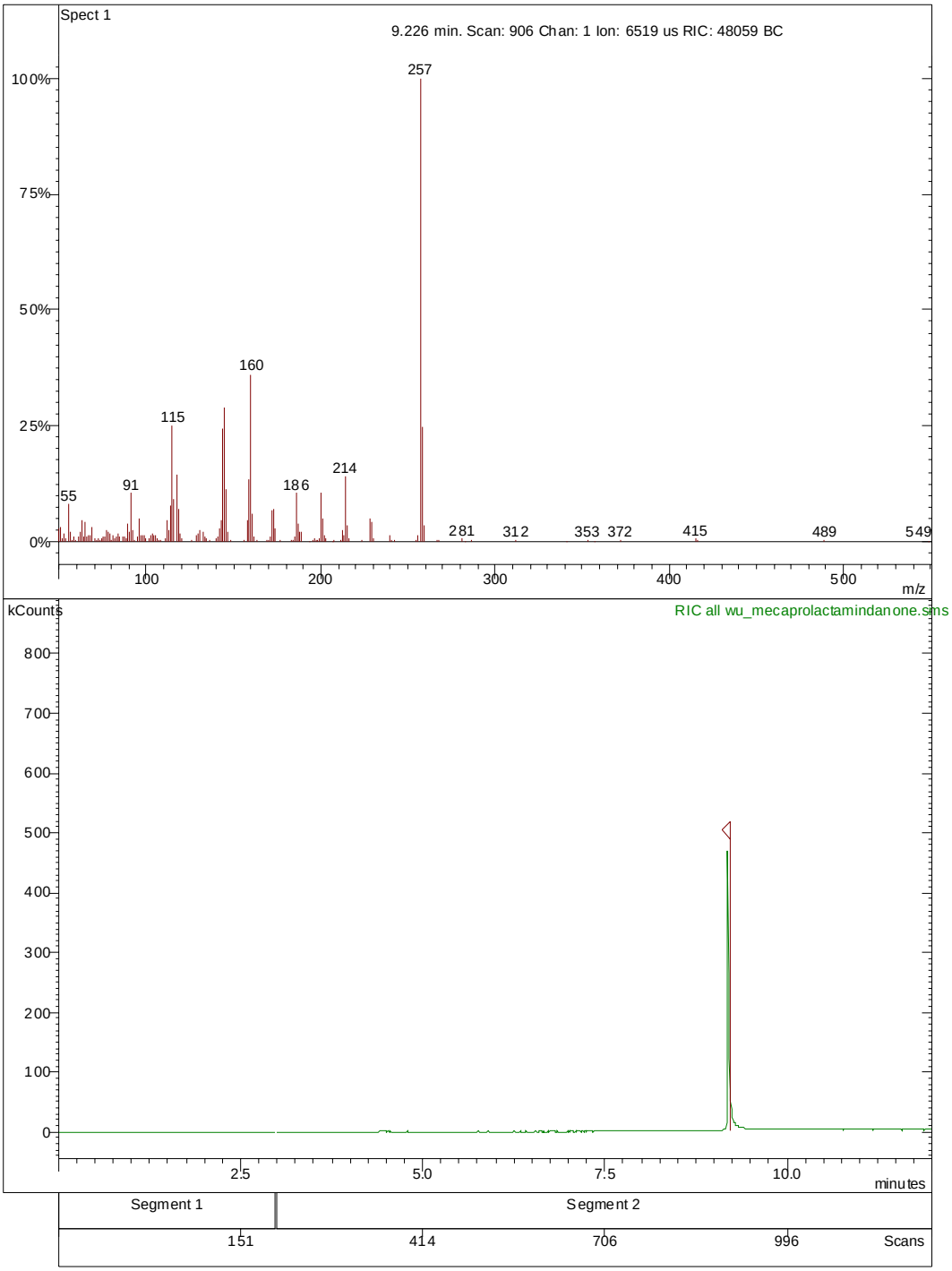
1-(5-methyl-3-oxo-indan-1-yl)-azepan-one (8f)



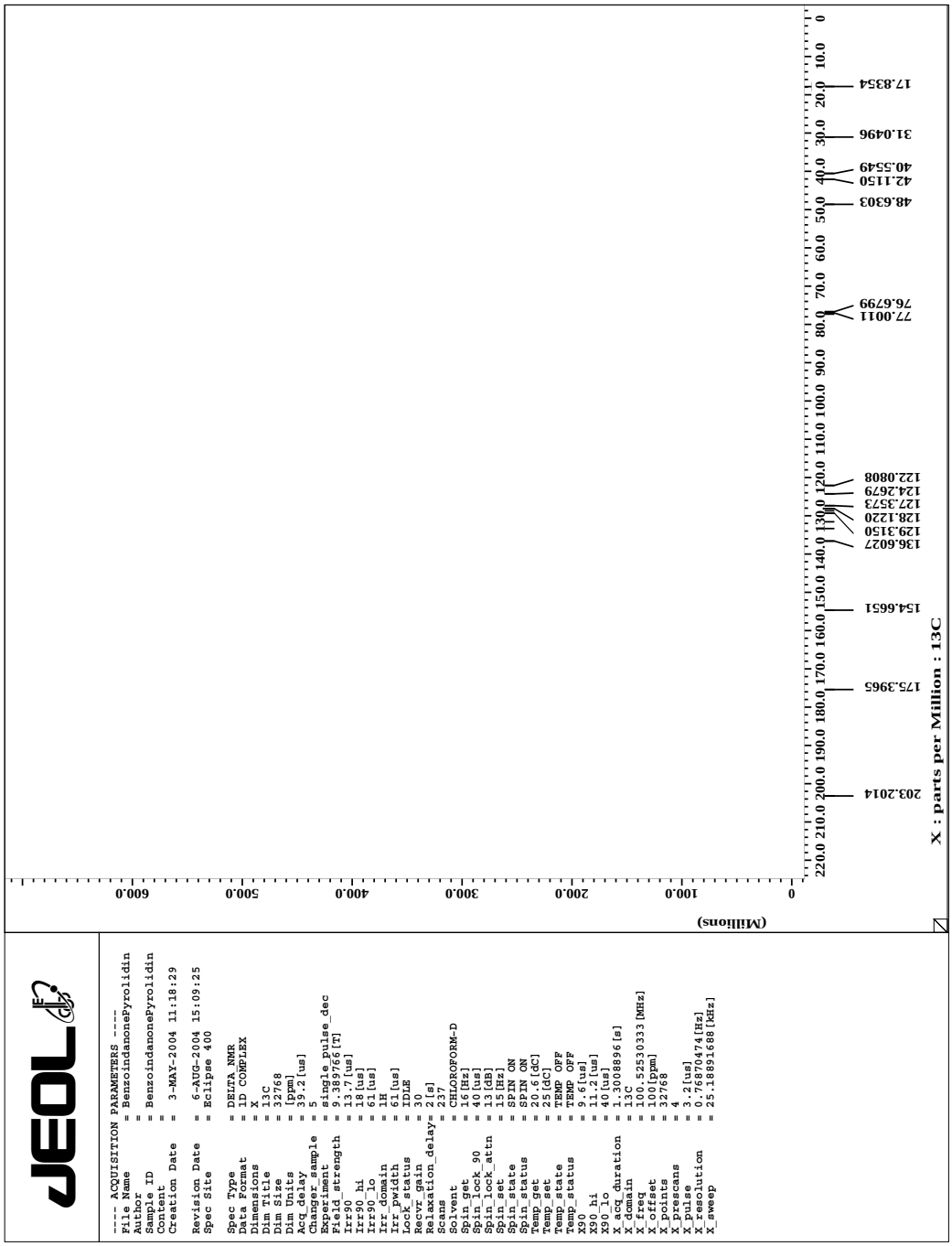
Chromatogram Plot

File: i:\wu_mecaprolactamindanone.sms
Sample: wu_MePyIndanone
Scan Range: 1 - 1227 Time Range: 0.00 - 11.99 min.
Sample Notes: today

Operator: Operator
Date: 04/29/04 17:02



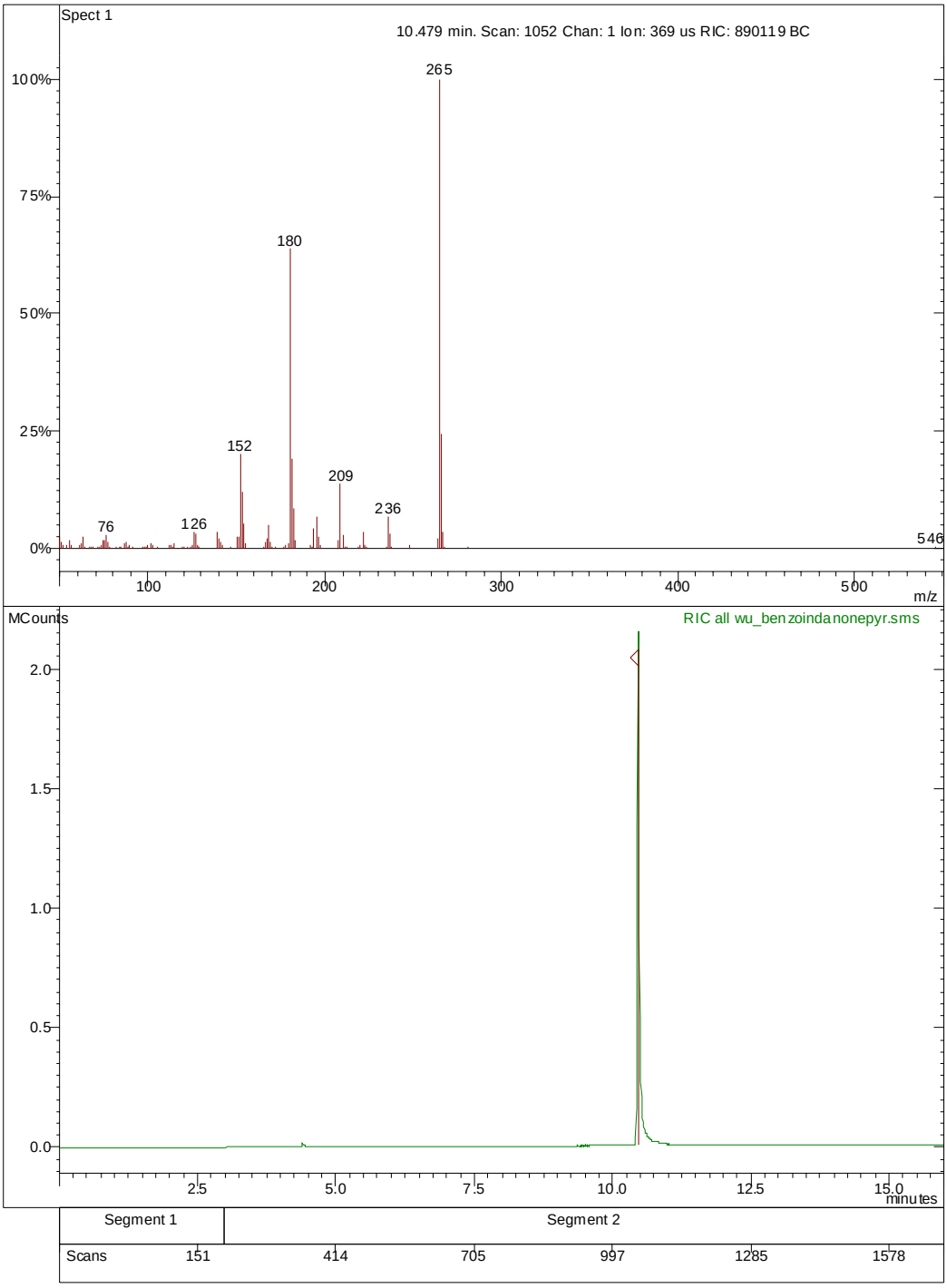
1-(1-Oxo-2,3-dihydro-1*H*-cyclopenta[*a*]naphthalen-3-yl)-pyrrolidin-2-one (8g)



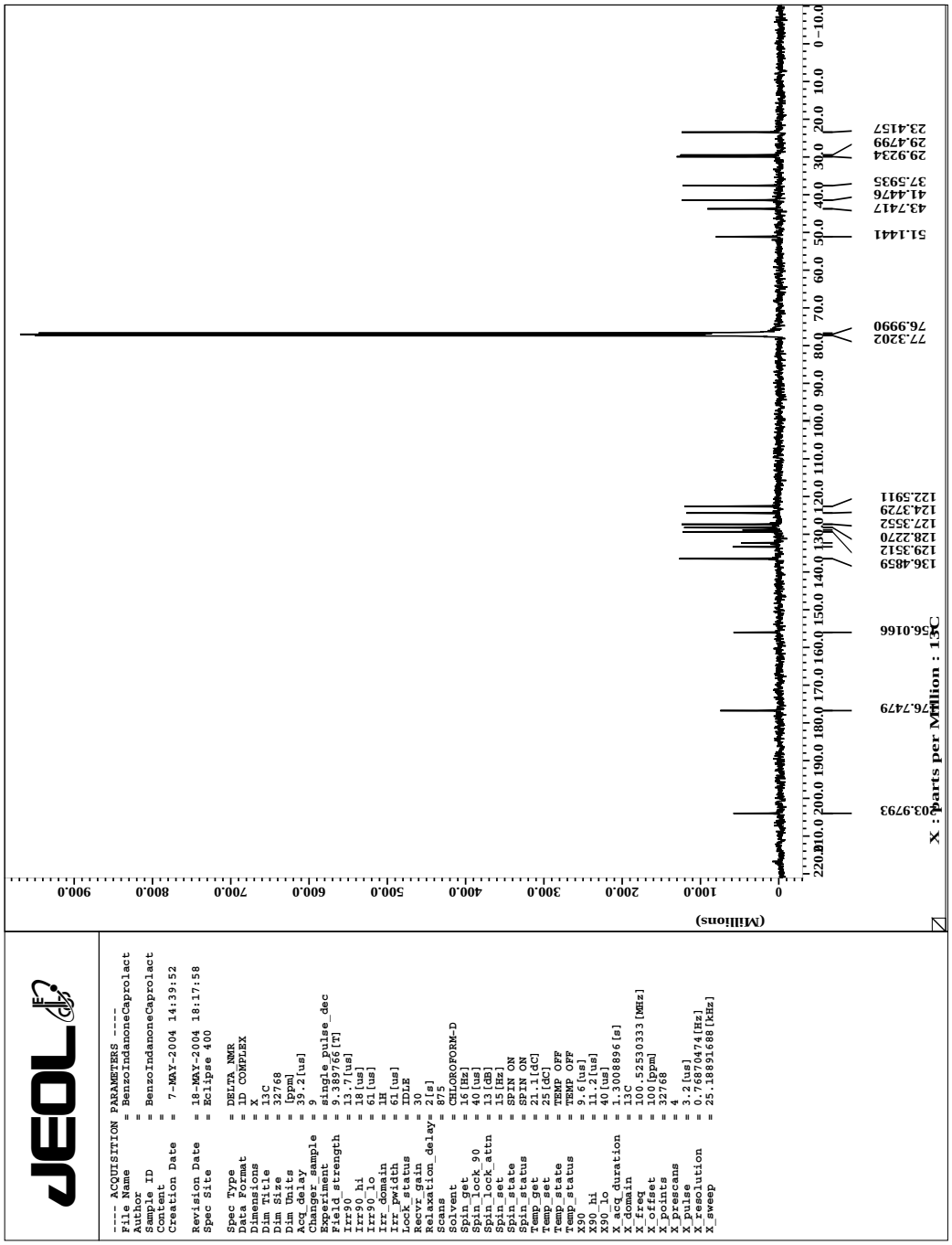
Chromatogram Plot

File: i:\wu_benzoindanonepyr.sms
Sample: wu_benzoindanonepyr
Scan Range: 1 - 1693 Time Range: 0.00 - 15.98 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 05/03/04 12:14



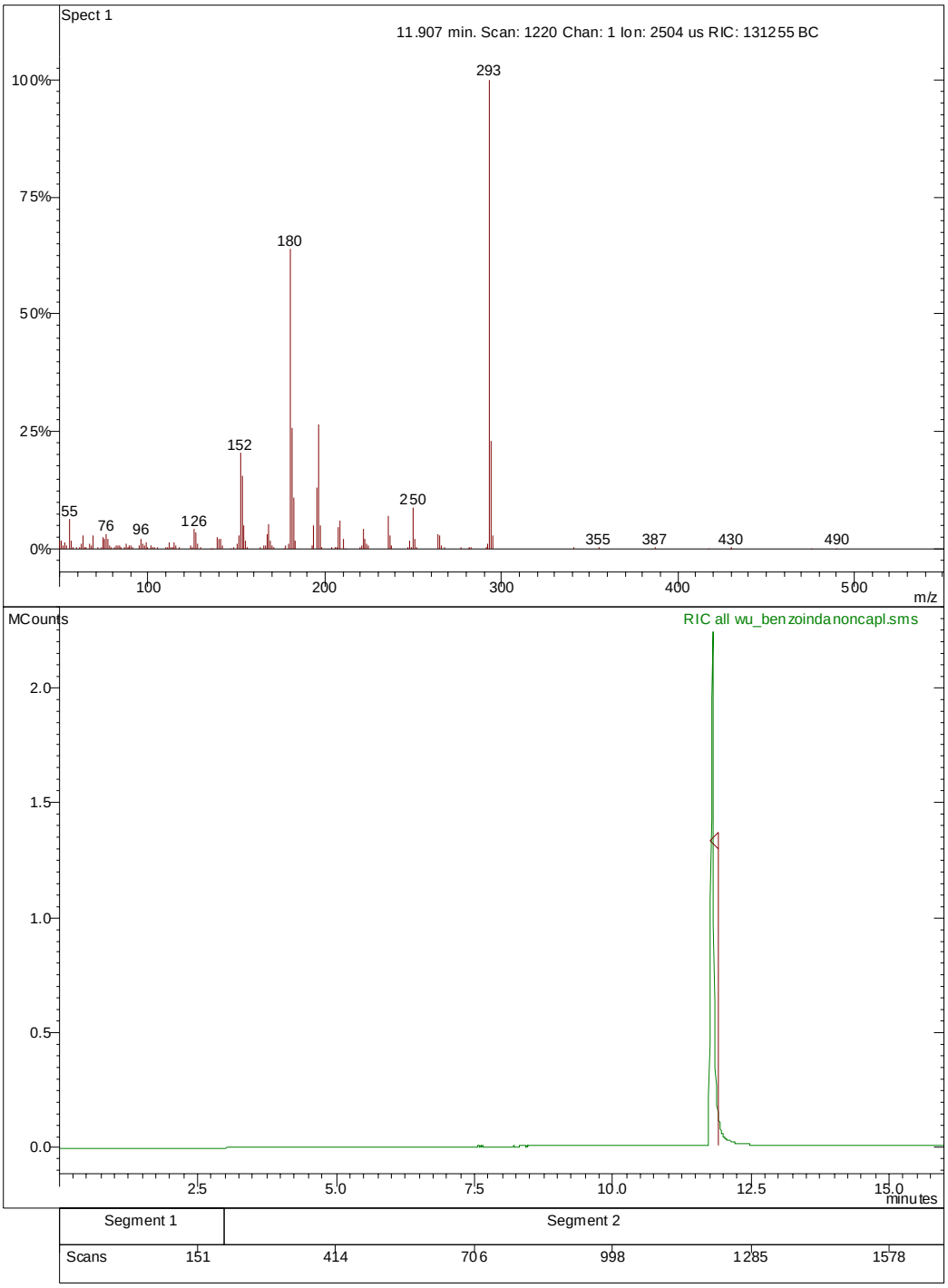
1-(1-Oxo-2,3-dihydro-1*H*-cyclopenta[*a*]naphthalen-3-yl)-azepan-2-one (8h)



Chromatogram Plot

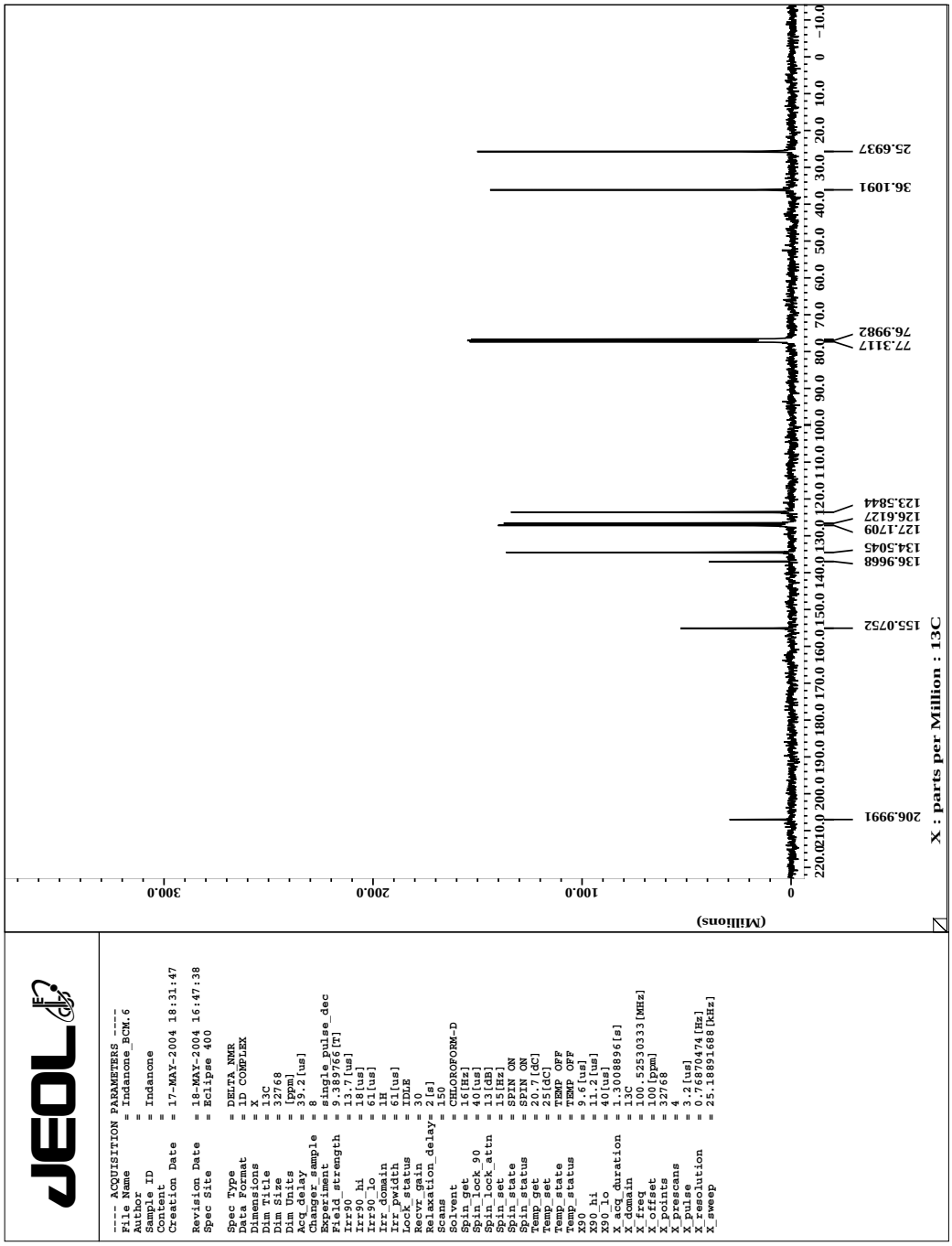
File: i:\wu_benzoindanoncapl.sms
Sample: wu_BenzoindanonCapl
Scan Range: 1 - 1693 Time Range: 0.00 - 15.98 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 05/06/04 11:35



^{13}C NMR spectra and GC-MS chromatograms for known compounds 2a-g, 2i,j and 3g,h, 4b, 4e

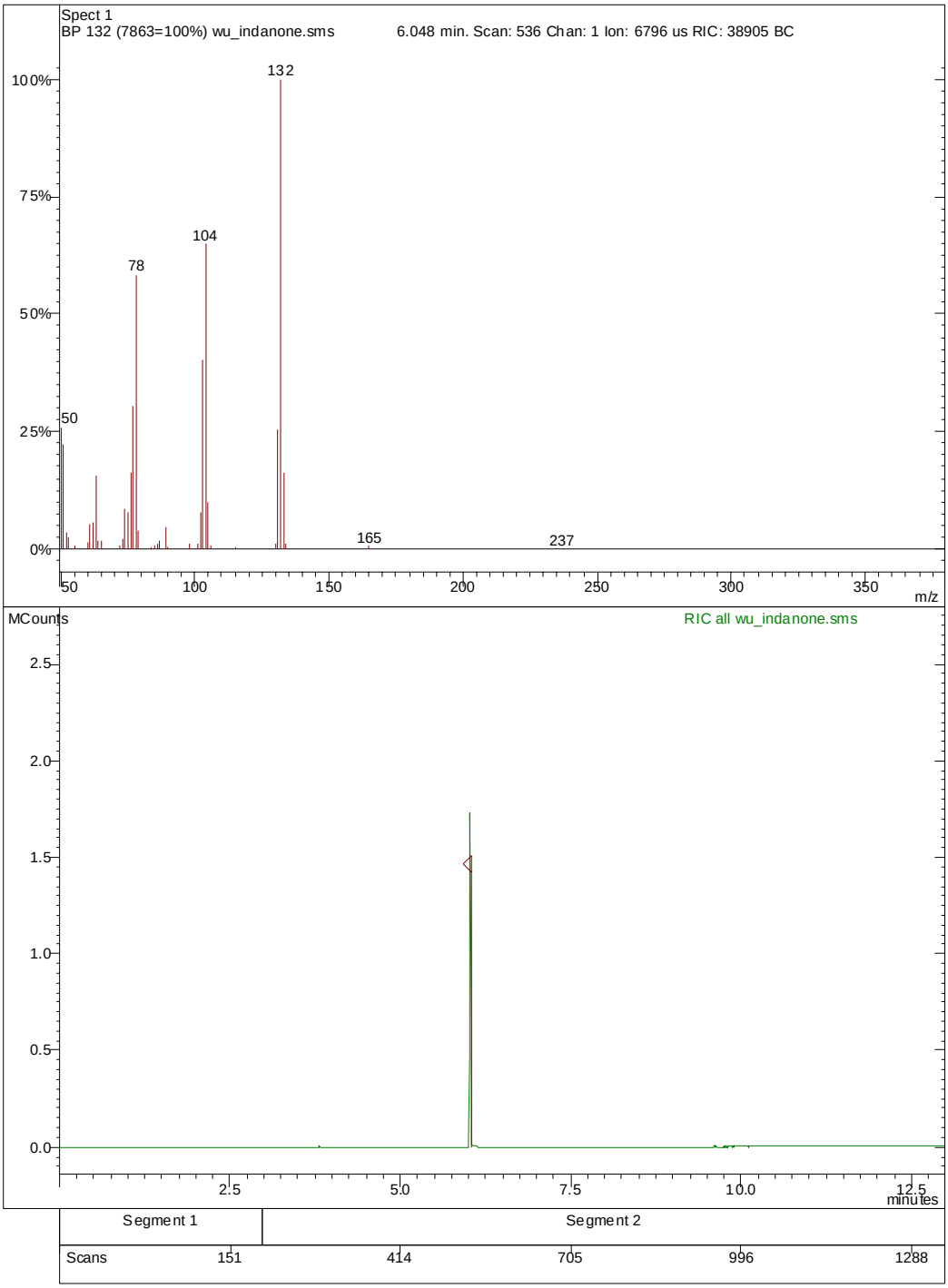
Indan-1-one (2a)



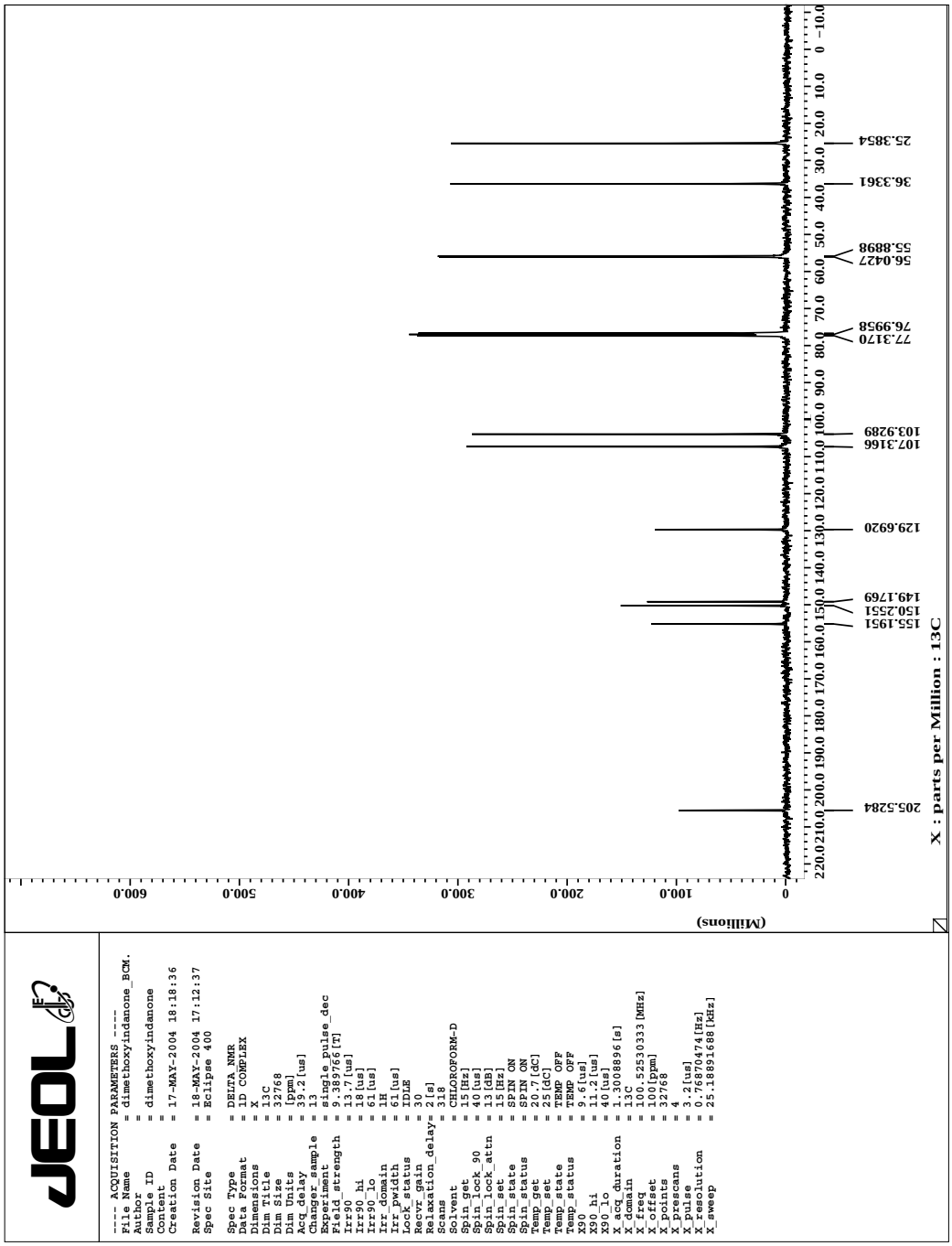
Chromatogram Plot

File: i:\wu_indanone.sms
Sample: wu_Indanone
Scan Range: 1 - 1344 Time Range: 0.00 - 12.98 min.
Sample Notes: ROUTINE

Operator: Andrei
Date: 02/22/04 20:45



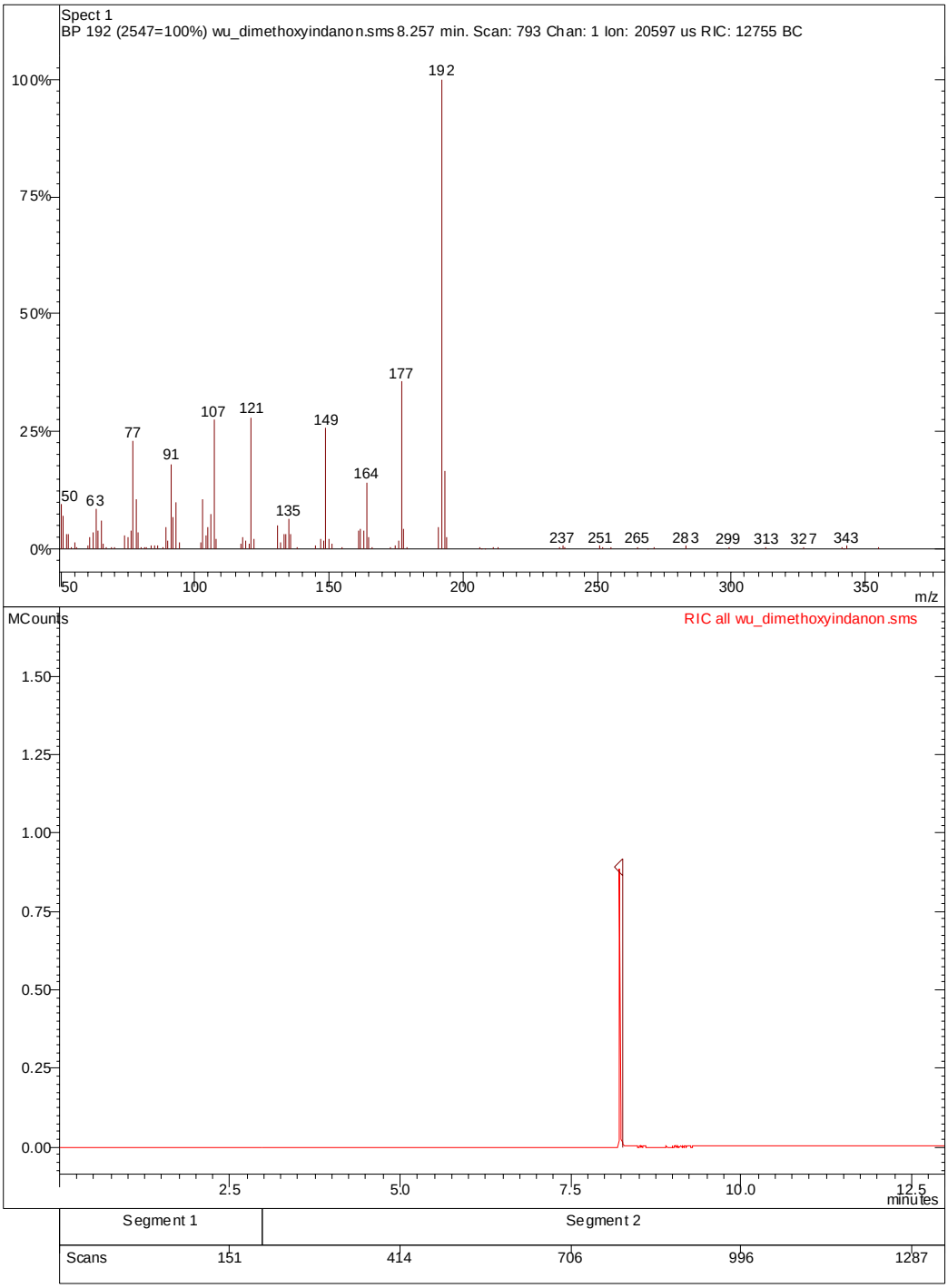
5,6-Dimethoxy-indan-1-one (2b)



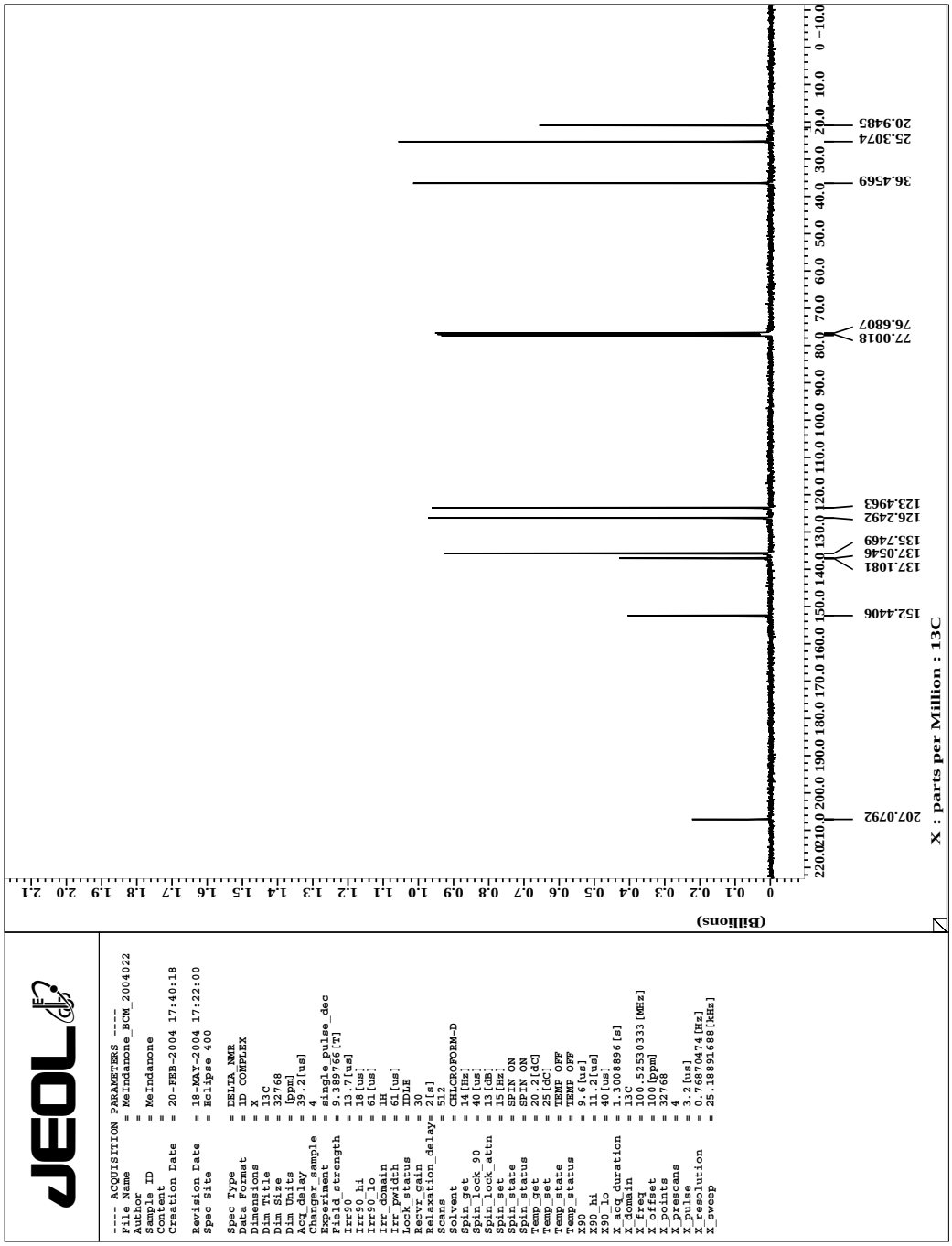
Chromatogram Plot

File: i:\wu_dimethoxyindanon.sms
Sample: wu_DiMethoxyIndanon
Scan Range: 1 - 1344 Time Range: 0.00 - 12.99 min.
Sample Notes: ROUTINE

Operator: Andrei
Date: 02/22/04 19:39



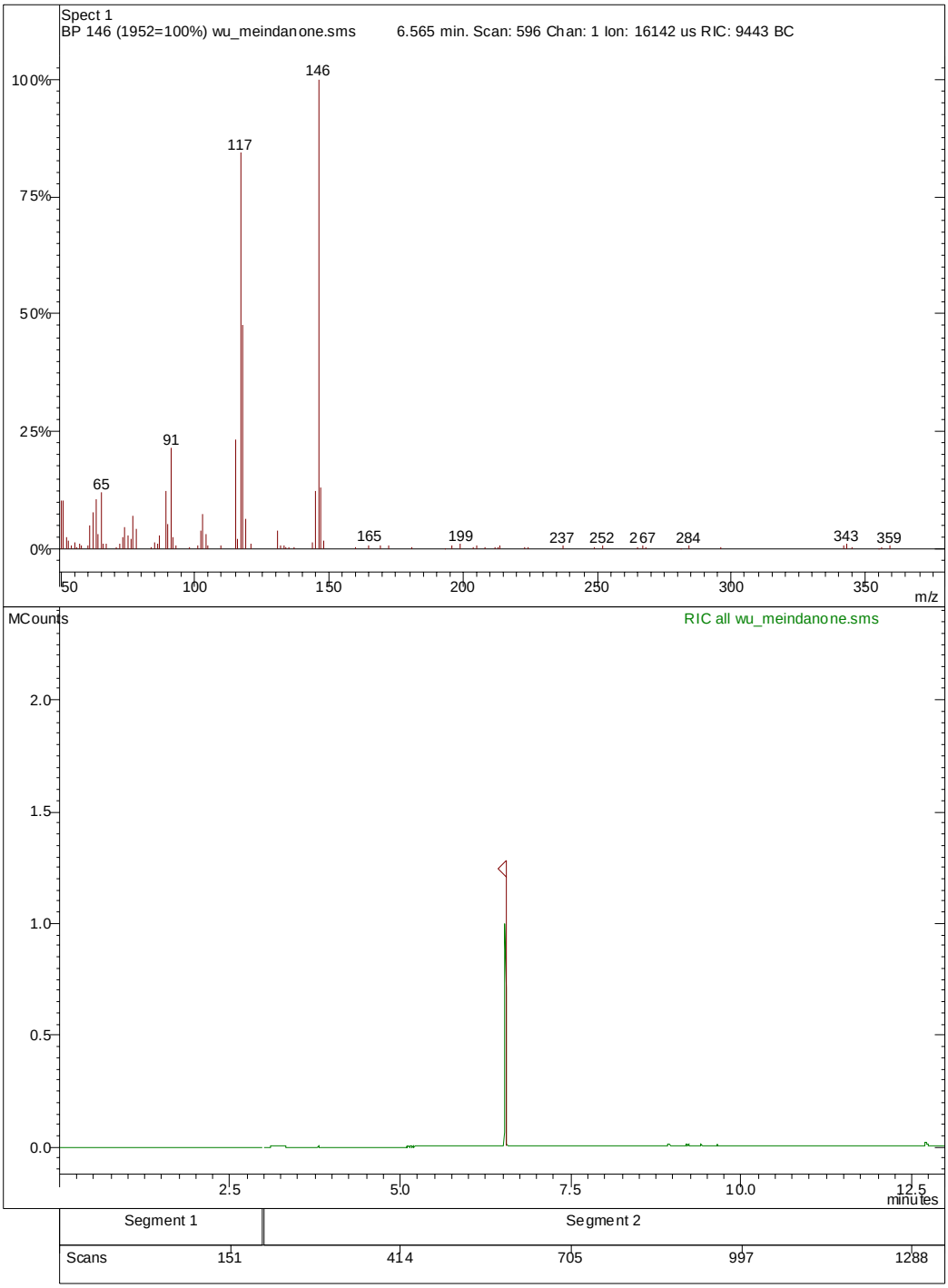
6-Methyl-indan-1-one (2c)



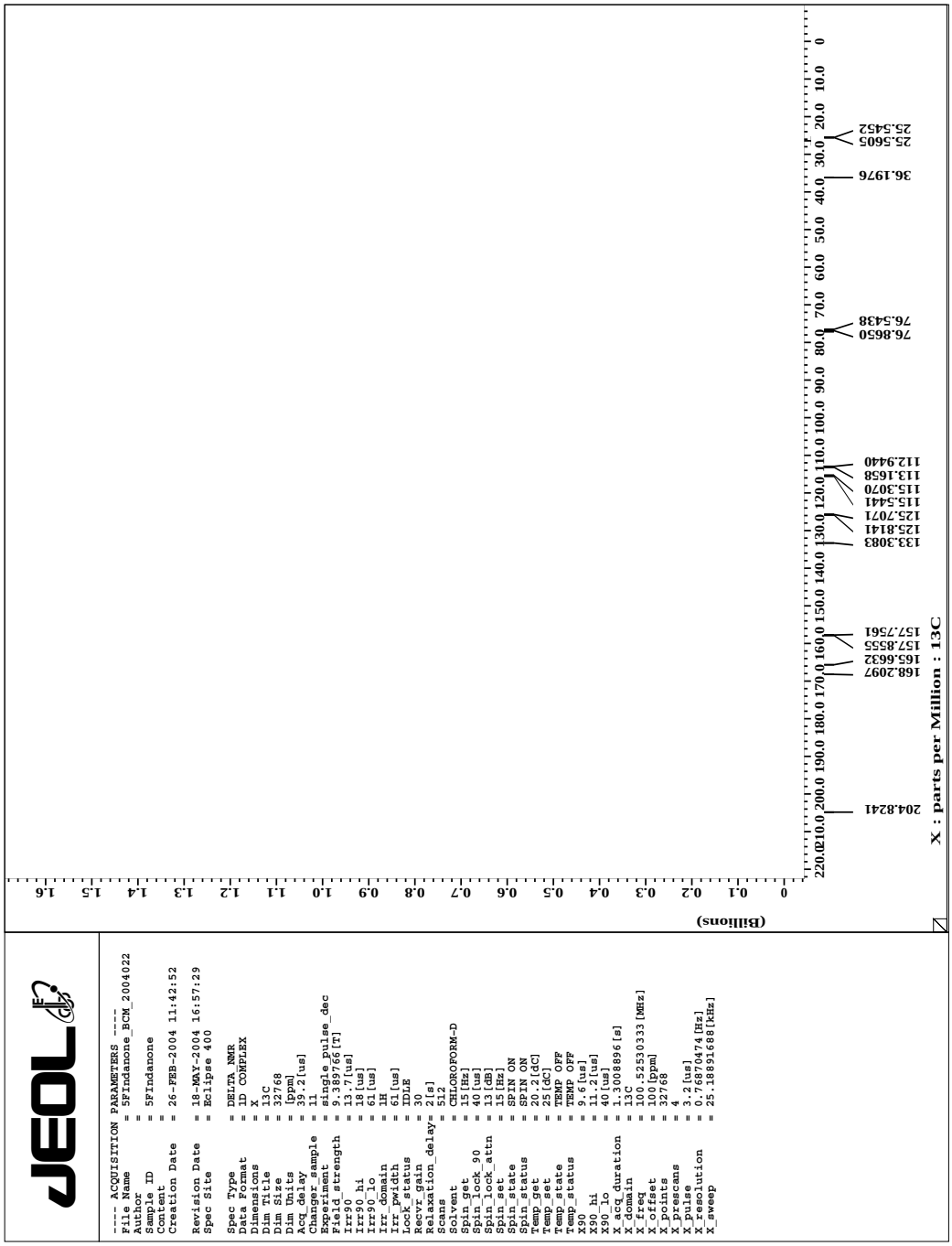
Chromatogram Plot

File: i:\wu_meindanone.sms
Sample: wu_MeIndanone
Scan Range: 1 - 1344 Time Range: 0.00 - 12.98 min.
Sample Notes: ROUTINE

Operator: Andrei
Date: 02/22/04 18:33



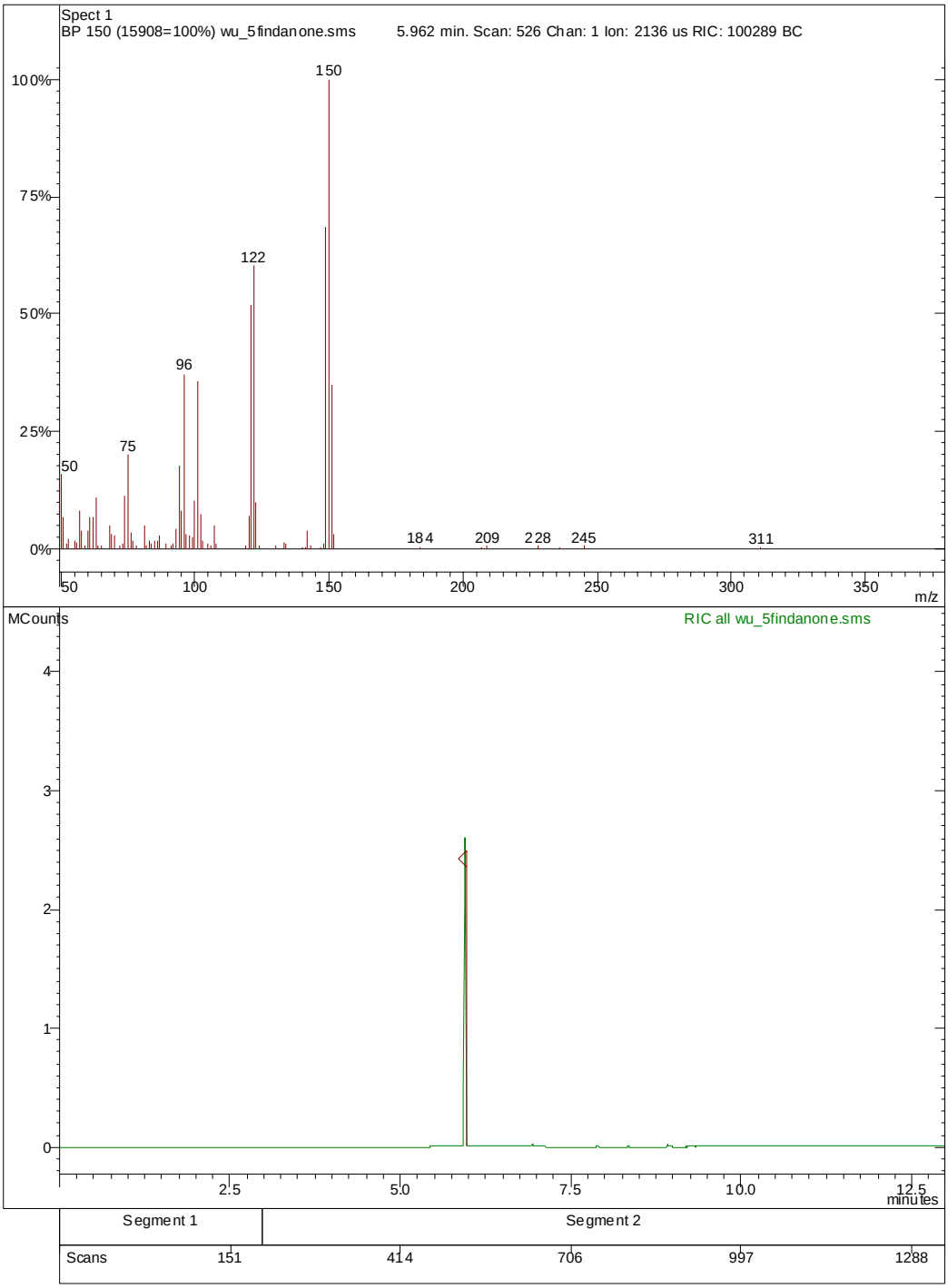
5-Fluoro-indan-1-one (2d)



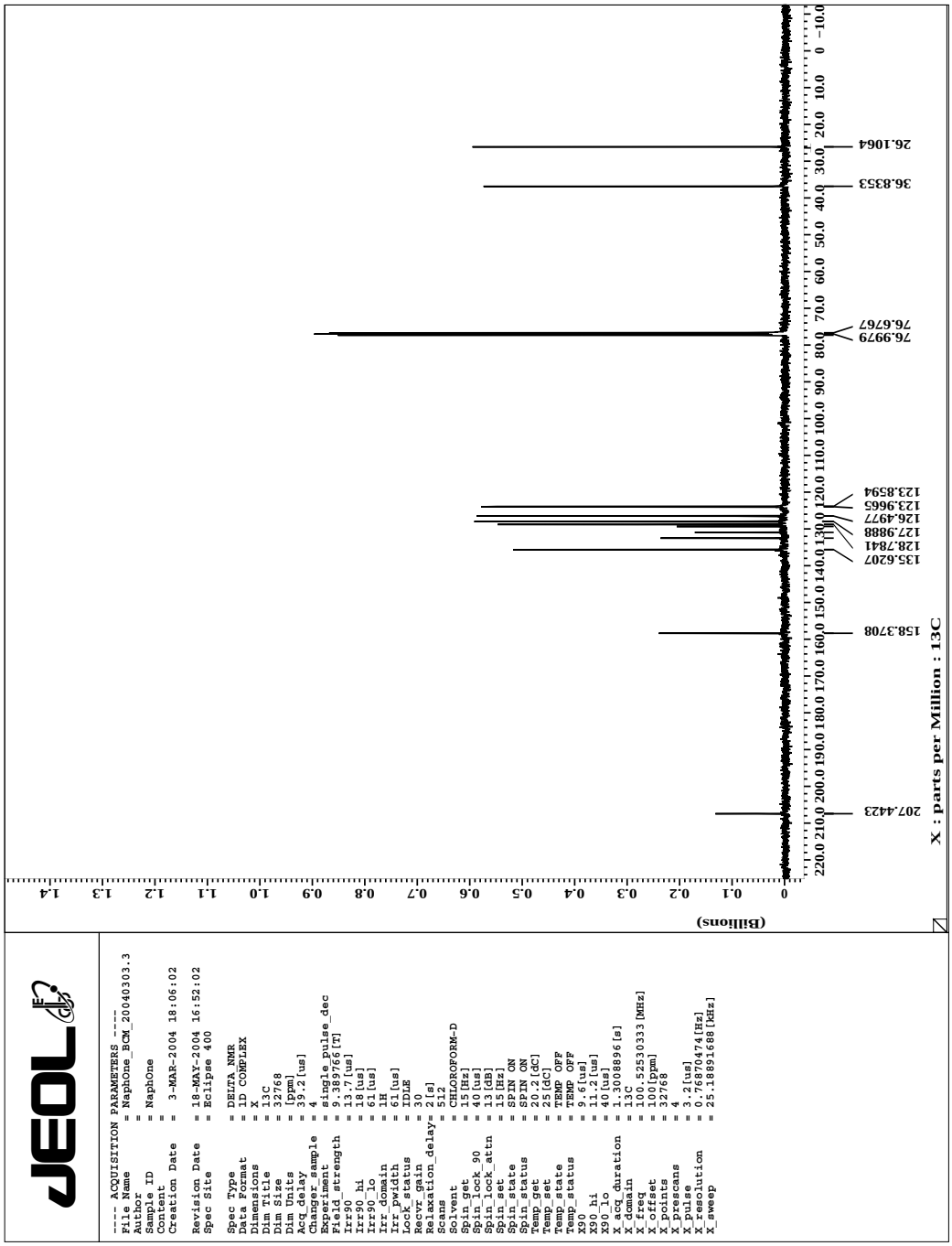
Chromatogram Plot

File: i:\wu_5findanone.sms
Sample: wu_5FIndanone
Scan Range: 1 - 1344 Time Range: 0.00 - 12.98 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 02/27/04 16:56



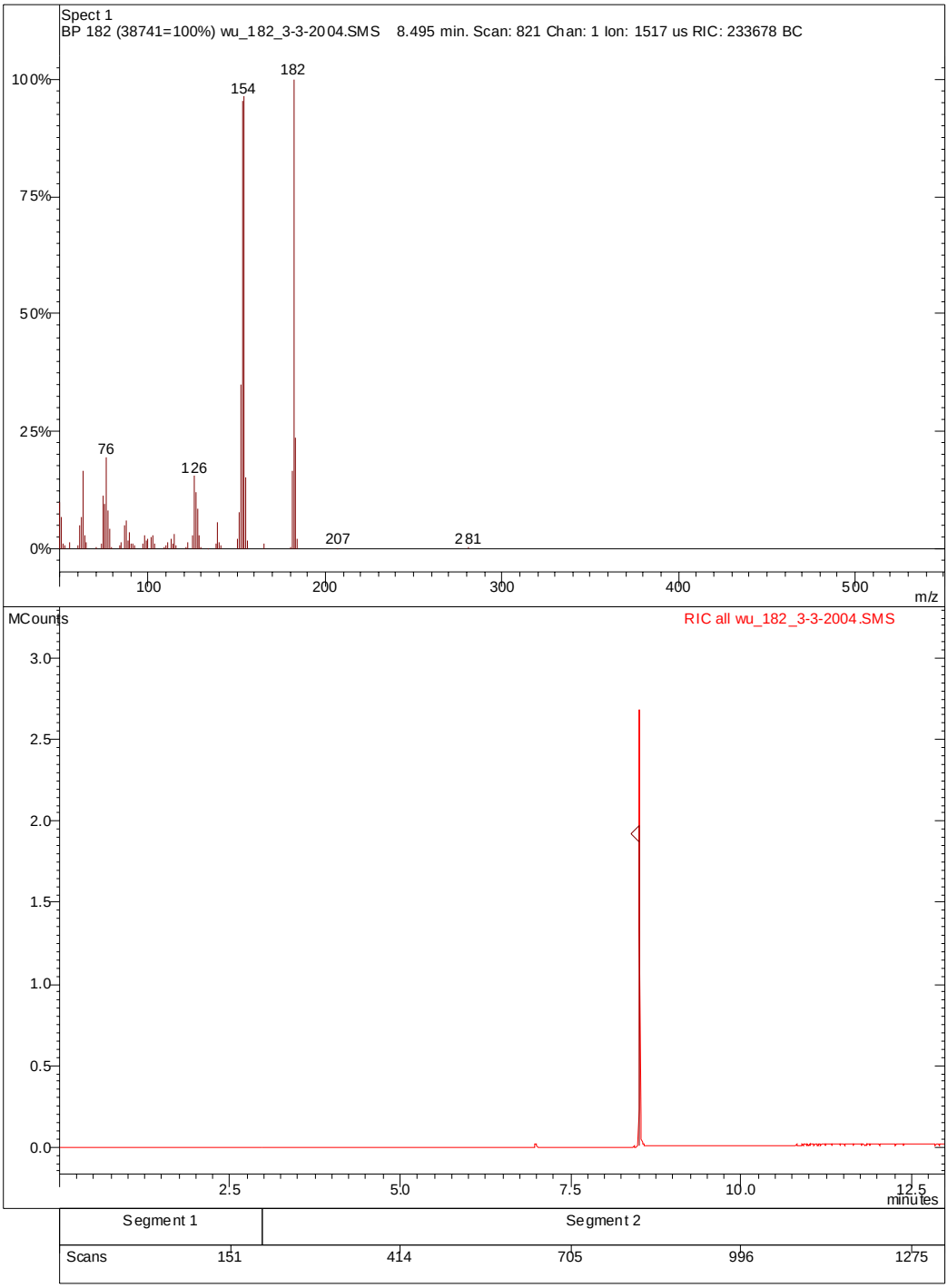
2,3-Dihydro-cyclopenta[]naphthalene-1-one (2e)



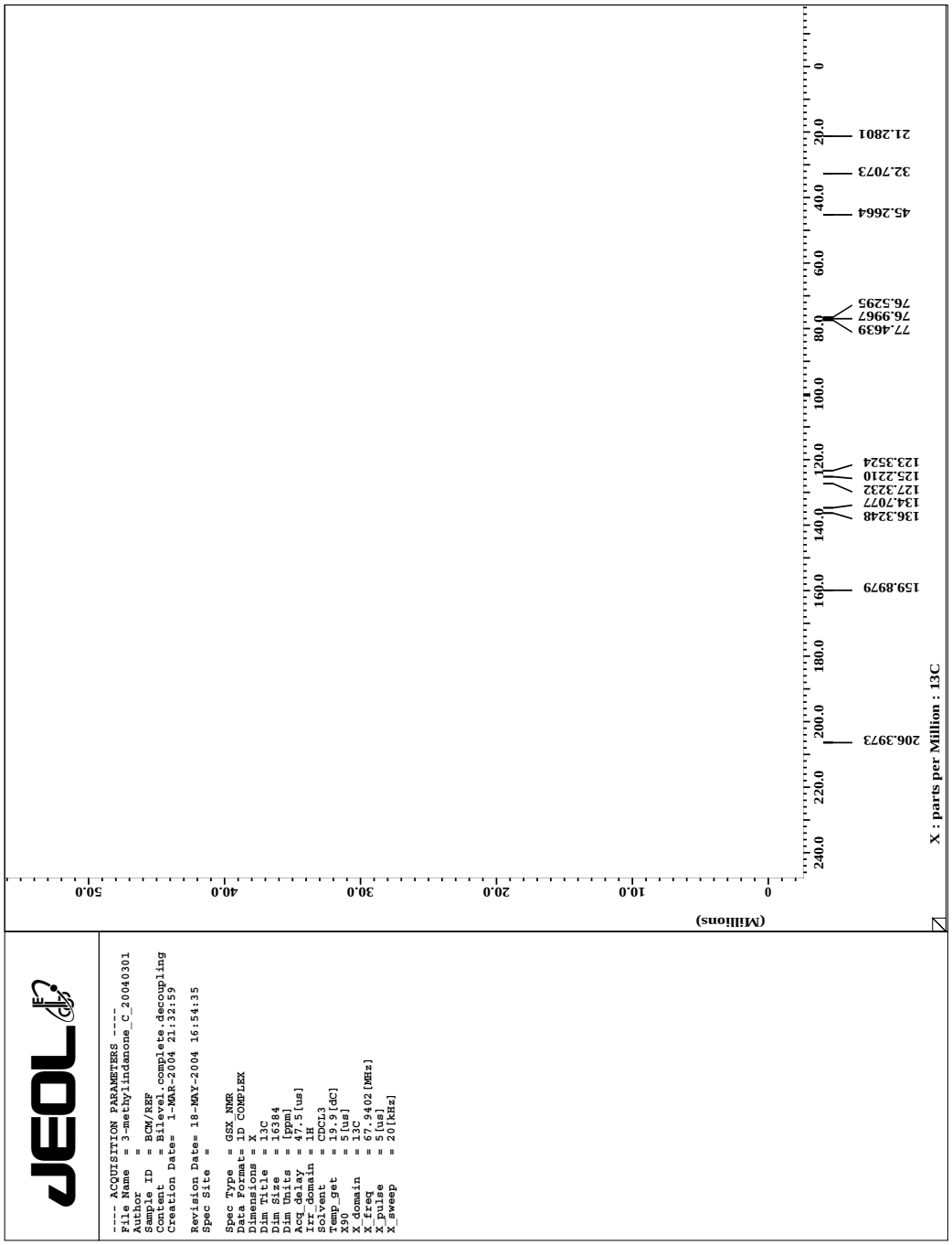
Chromatogram Plot

File: i:\wu_182_3-3-2004.sms
Sample: wu_182
Scan Range: 1 - 1327 Time Range: 0.00 - 12.98 min.
Sample Notes: today

Operator: Org Farm Kemi
Date: 03/03/04 17:25



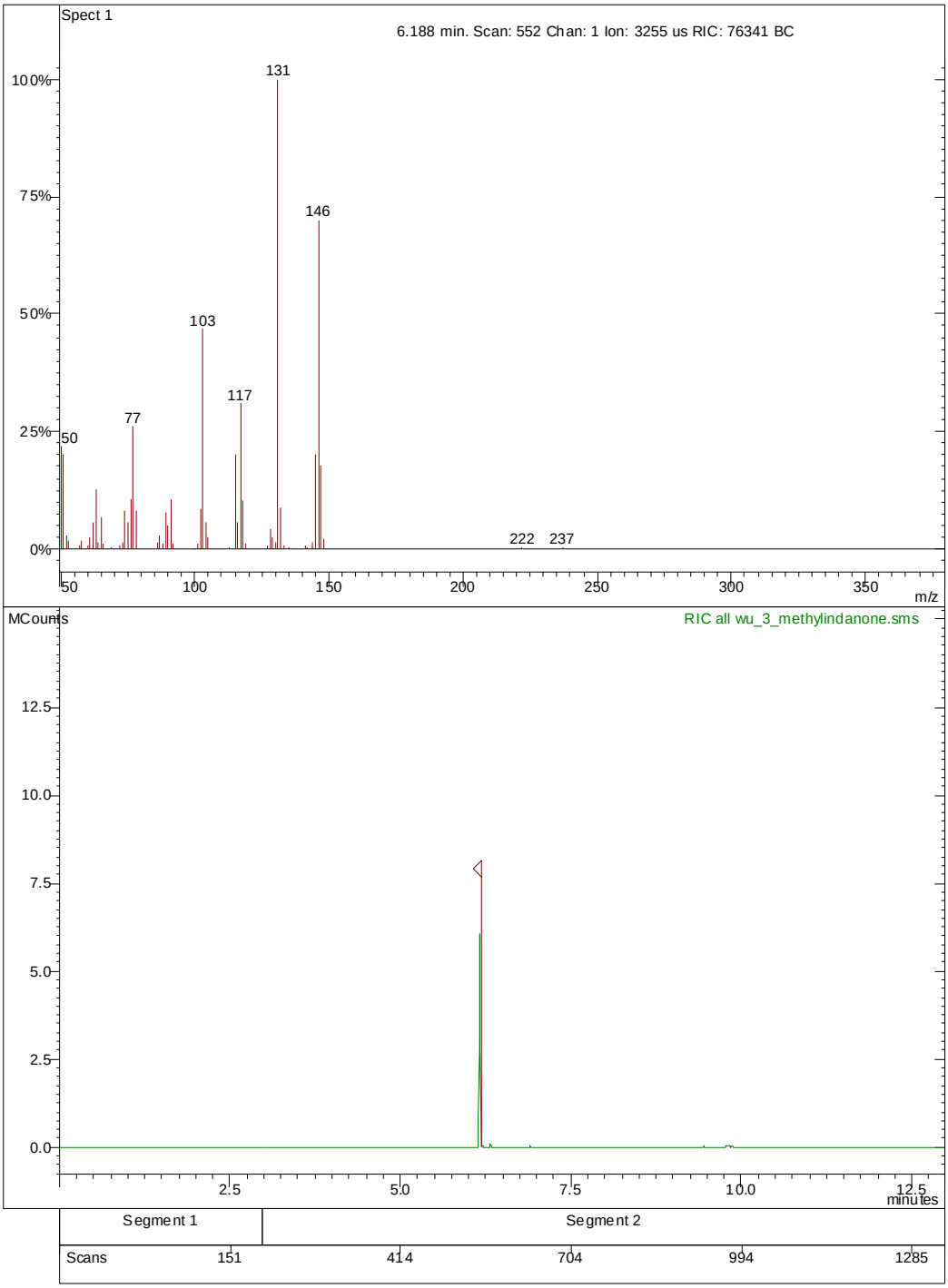
3-Methyl-indan-1-one (2f)



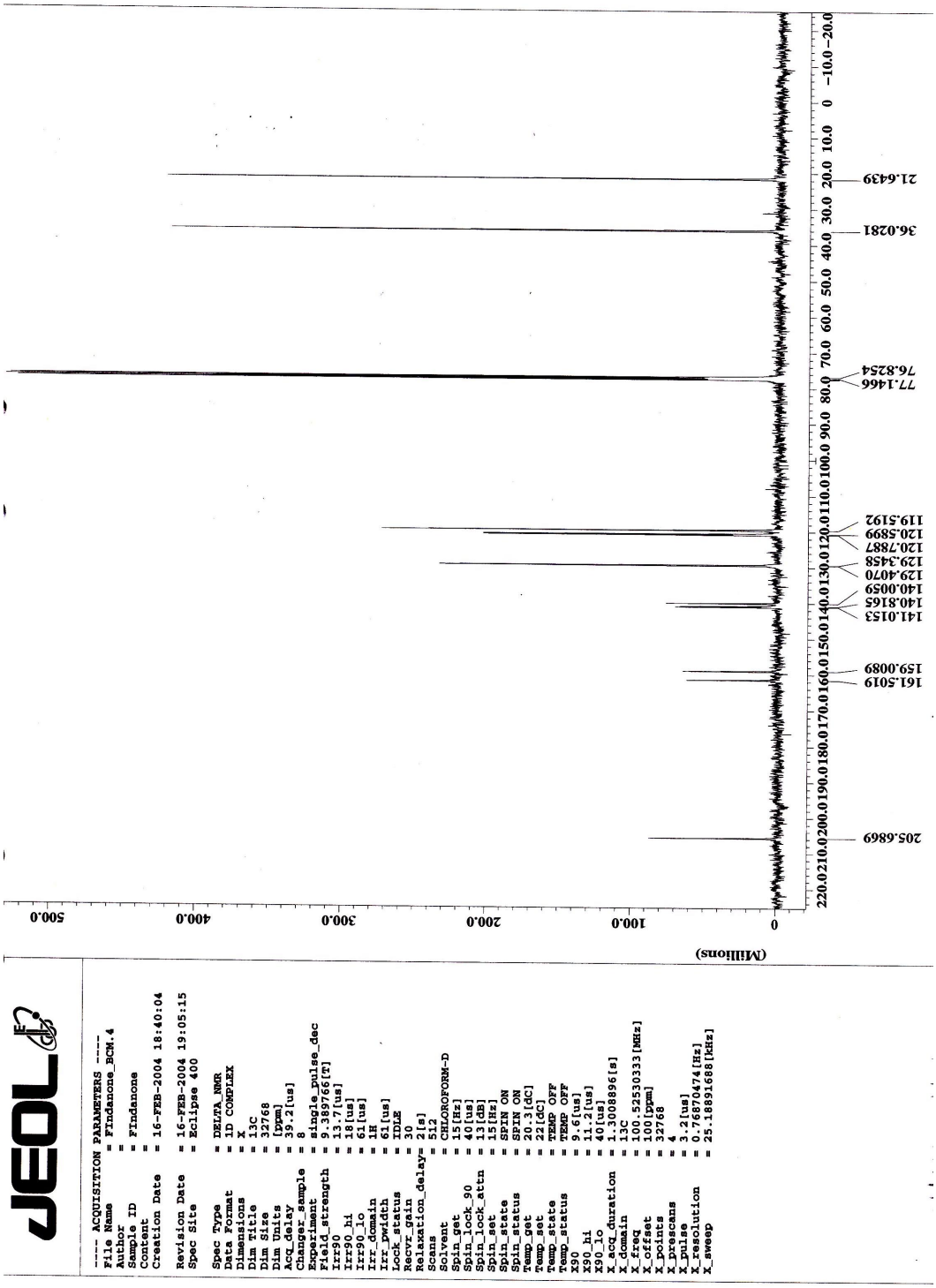
Chromatogram Plot

File: i:\wu_3_methylindanone.sms
Sample: wu_3_methylIndanone
Scan Range: 1 - 1341 Time Range: 0.00 - 12.98 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 03/04/04 14:44



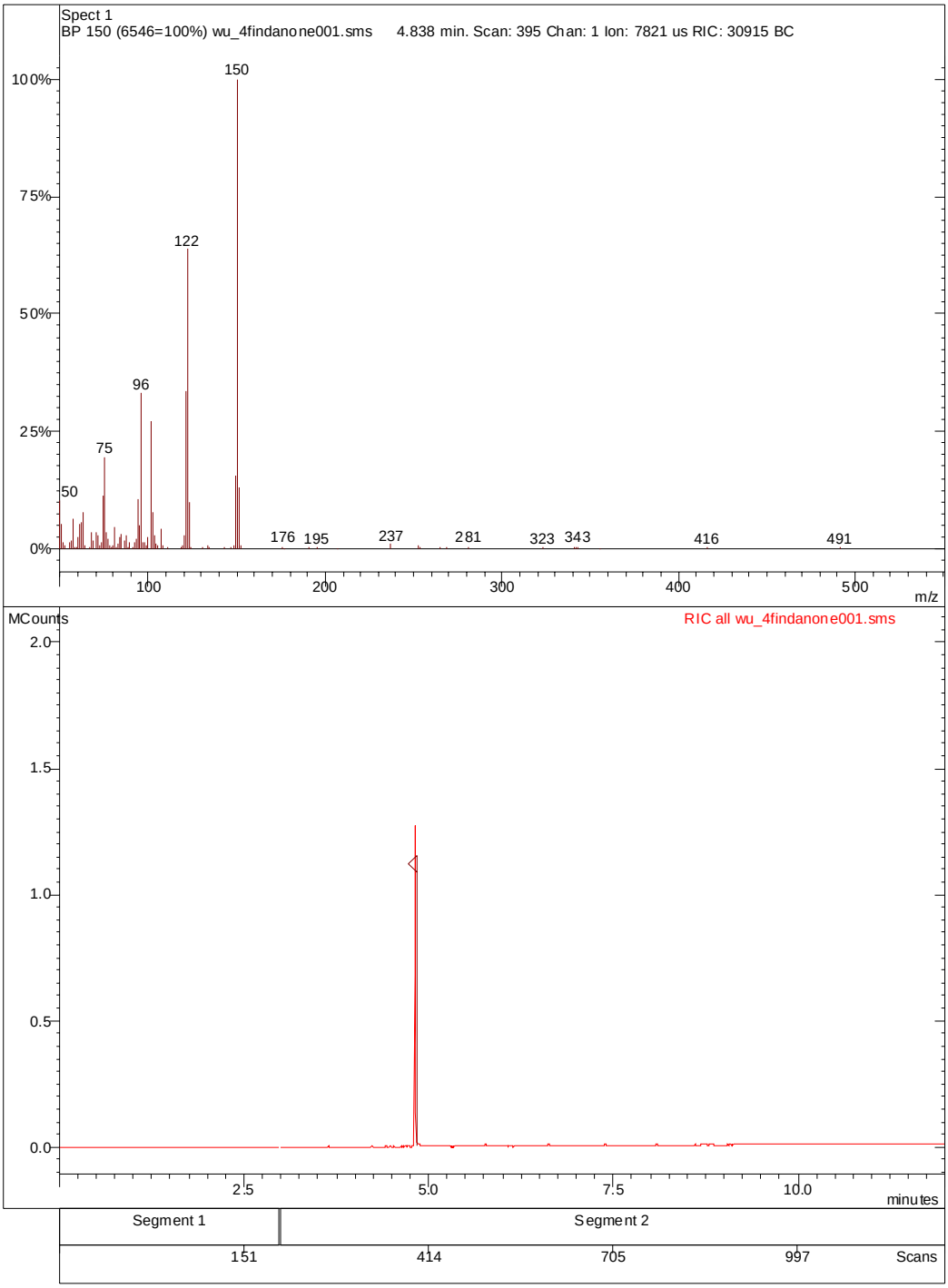
4-Fluoro-indan-1-one (2g)



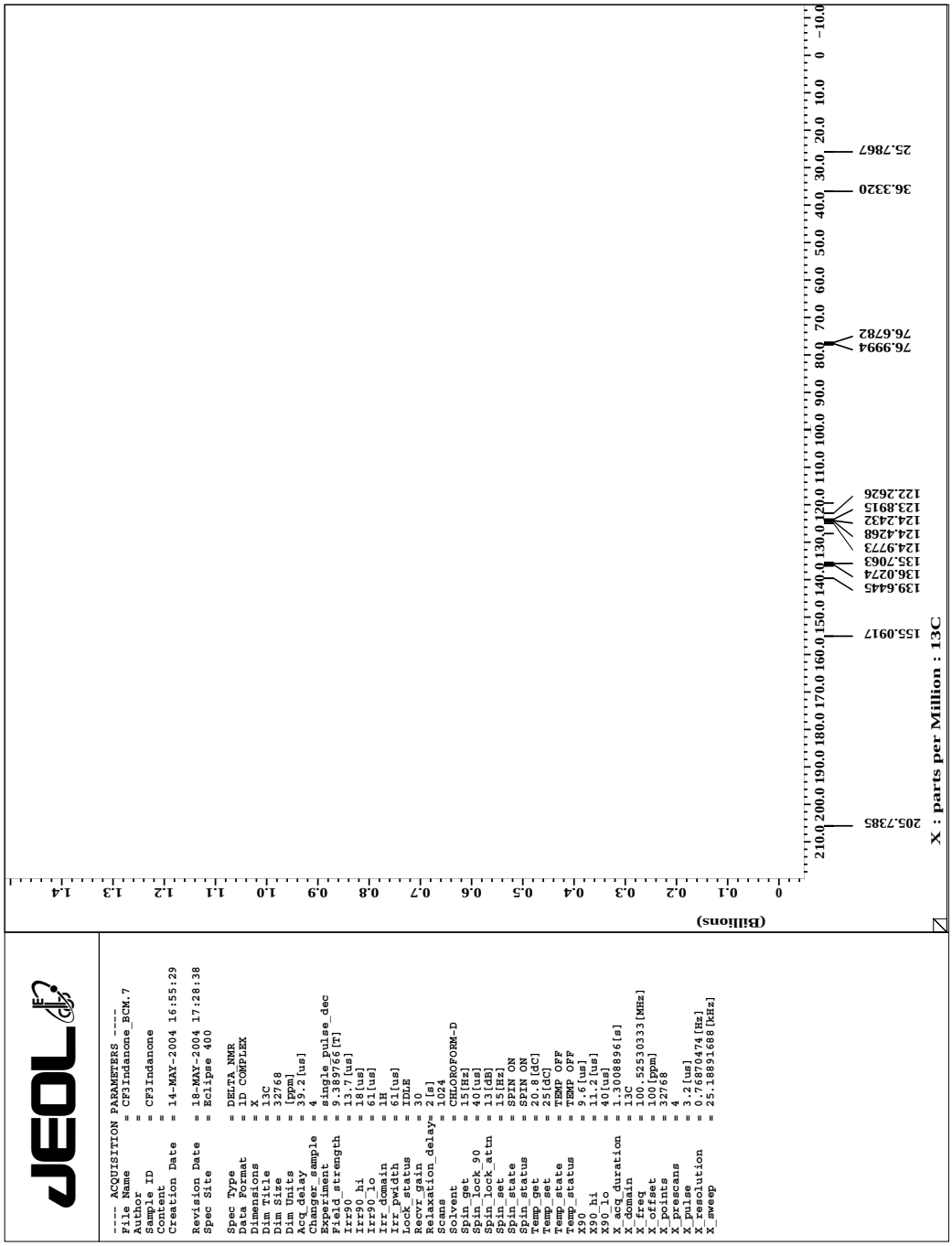
Chromatogram Plot

File: i:\wu_4findanone001.sms
Sample: wu_4FIndanone
Scan Range: 1 - 1232 Time Range: 0.00 - 11.99 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 05/17/04 15:19



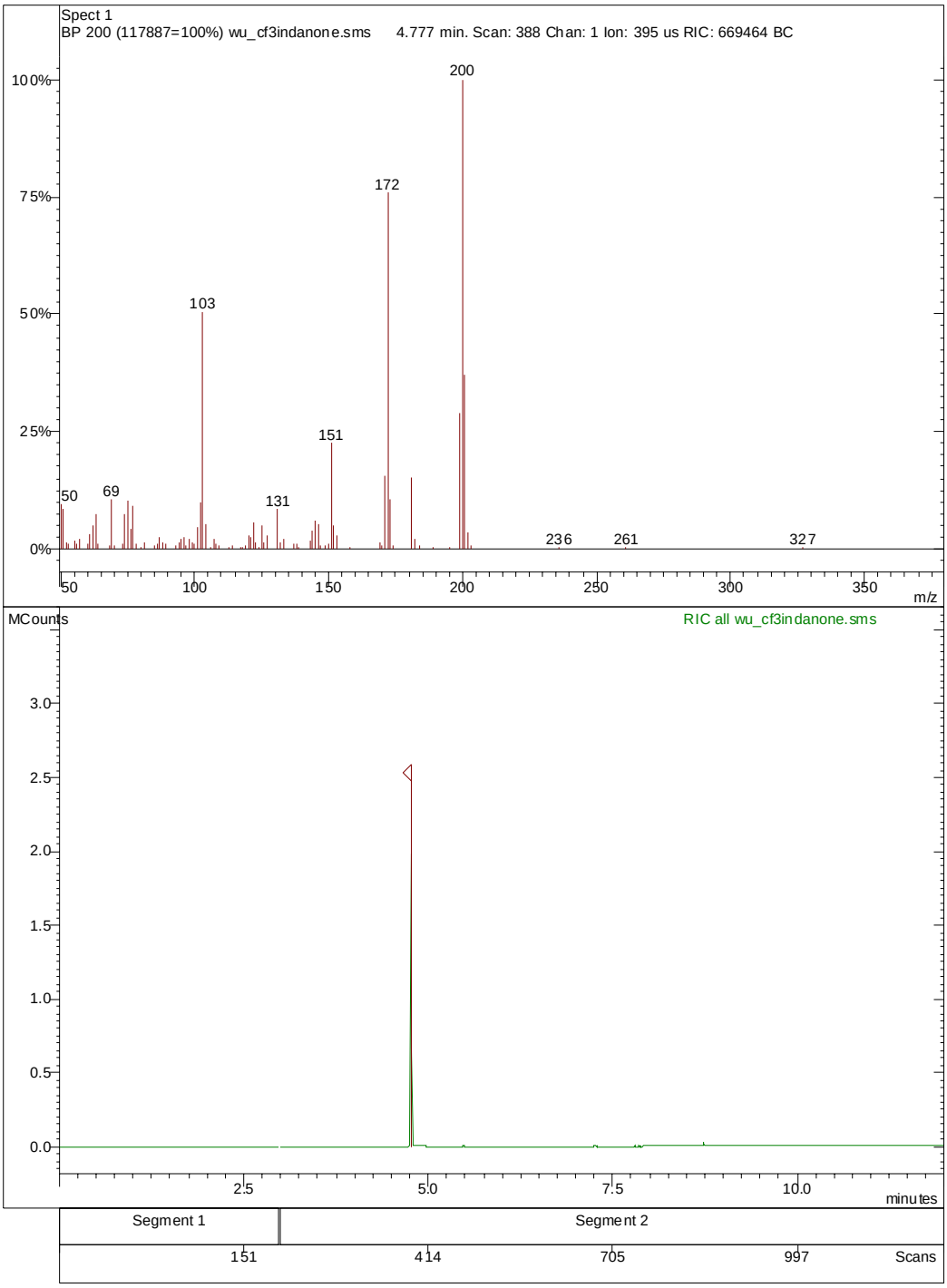
5-Trifluoromethyl-indan-1-one (2i)



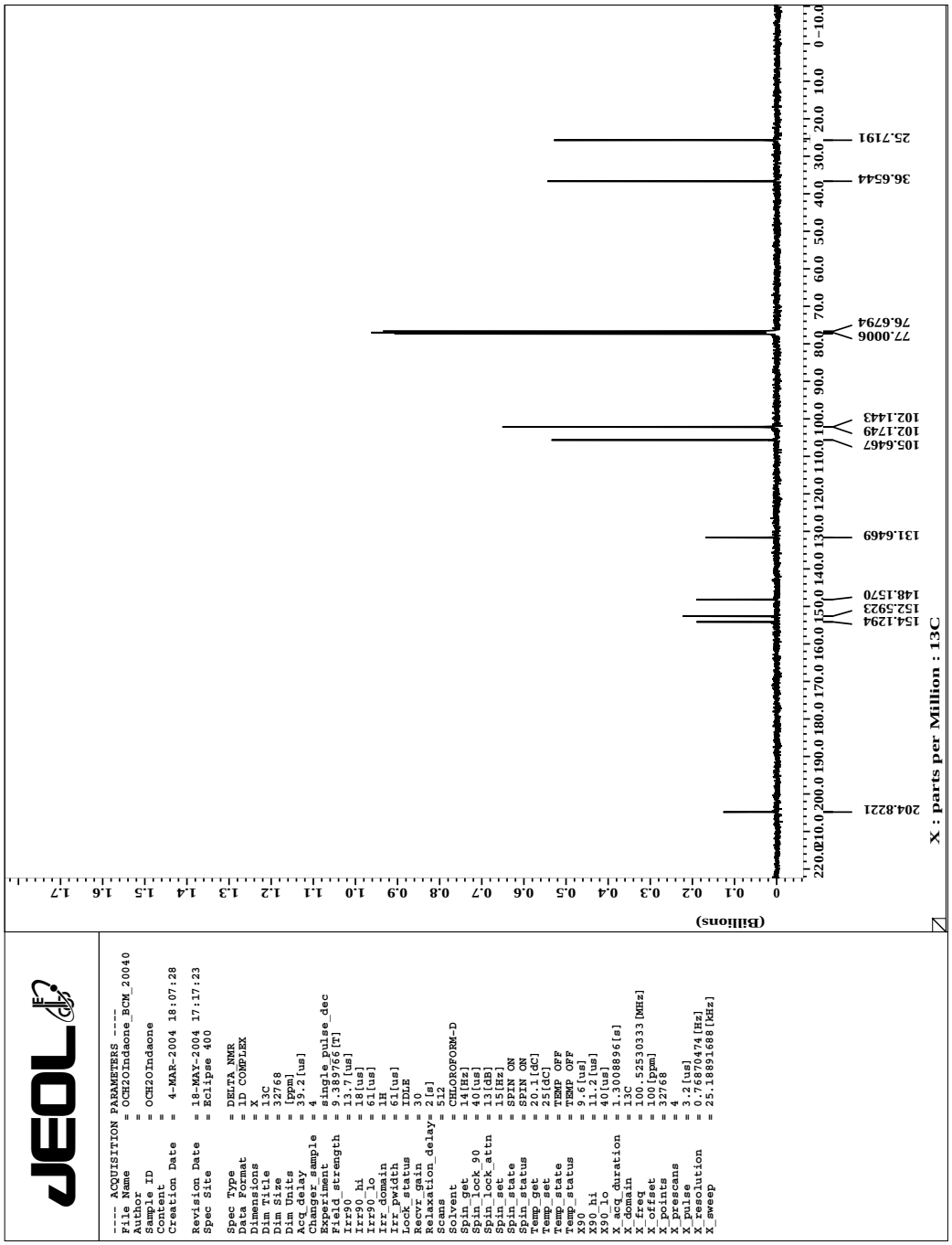
Chromatogram Plot

File: i:\wu_cf3indanone.sms
Sample: wu_CF3Indanone
Scan Range: 1 - 1230 Time Range: 0.00 - 11.98 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 05/14/04 17:59



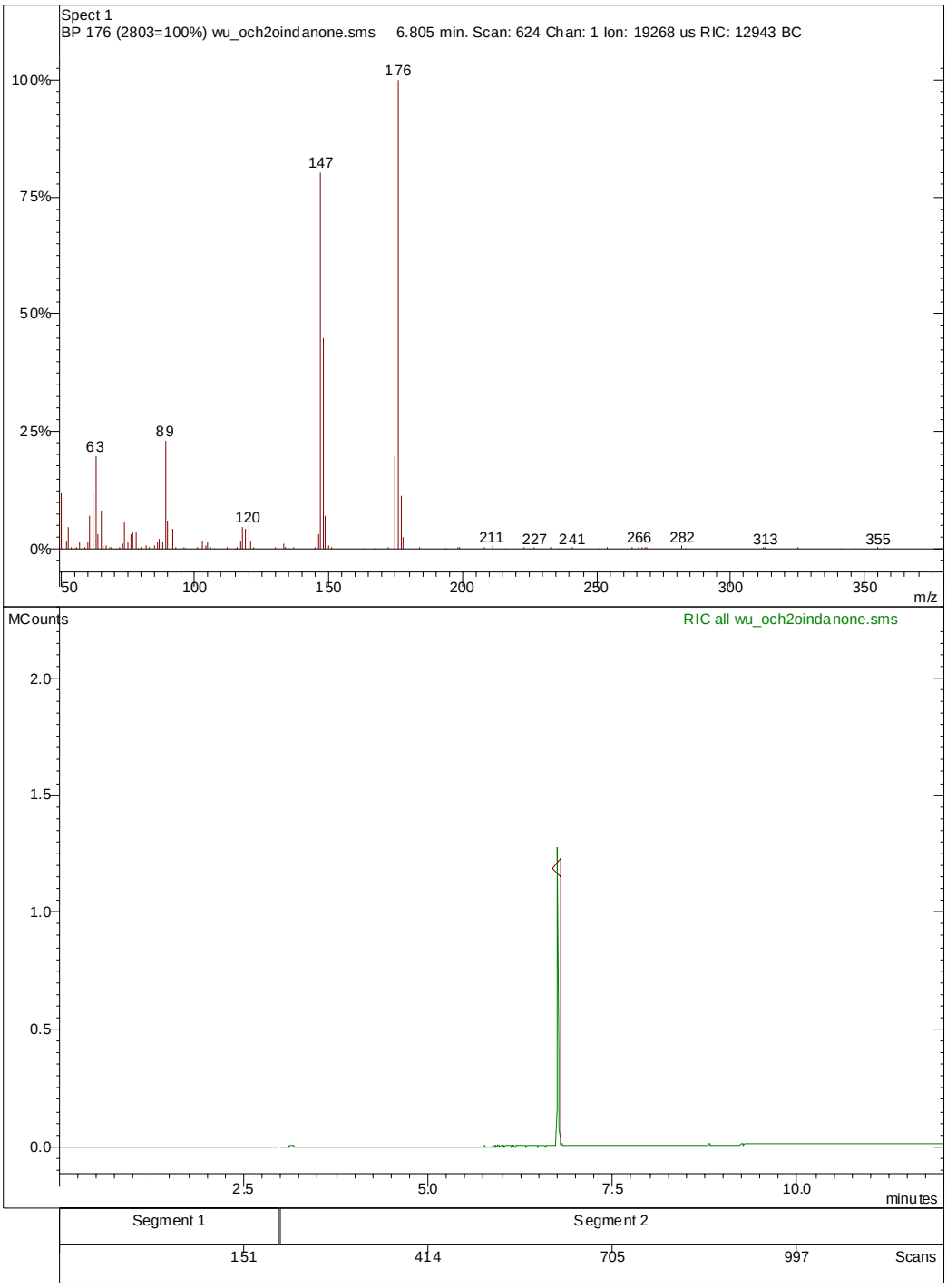
6,7-Dihydro-indeno[5,6-*d*][1,3]dioxol-5-one (2j)



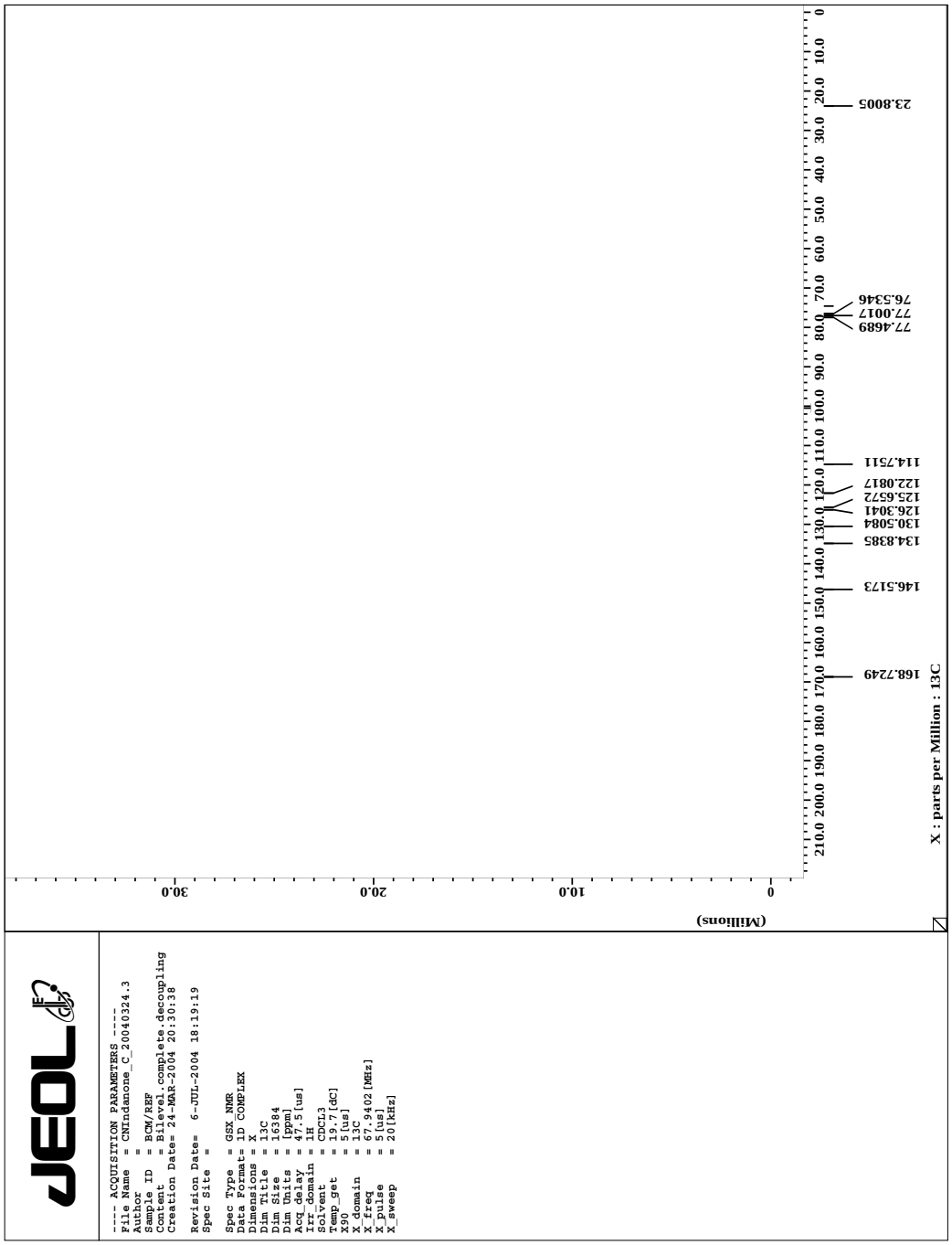
Chromatogram Plot

File: i:\wu_och2oindanone.sms
Sample: wu_OCH2OIndanone
Scan Range: 1 - 1232 Time Range: 0.00 - 11.99 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 05/17/04 13:13

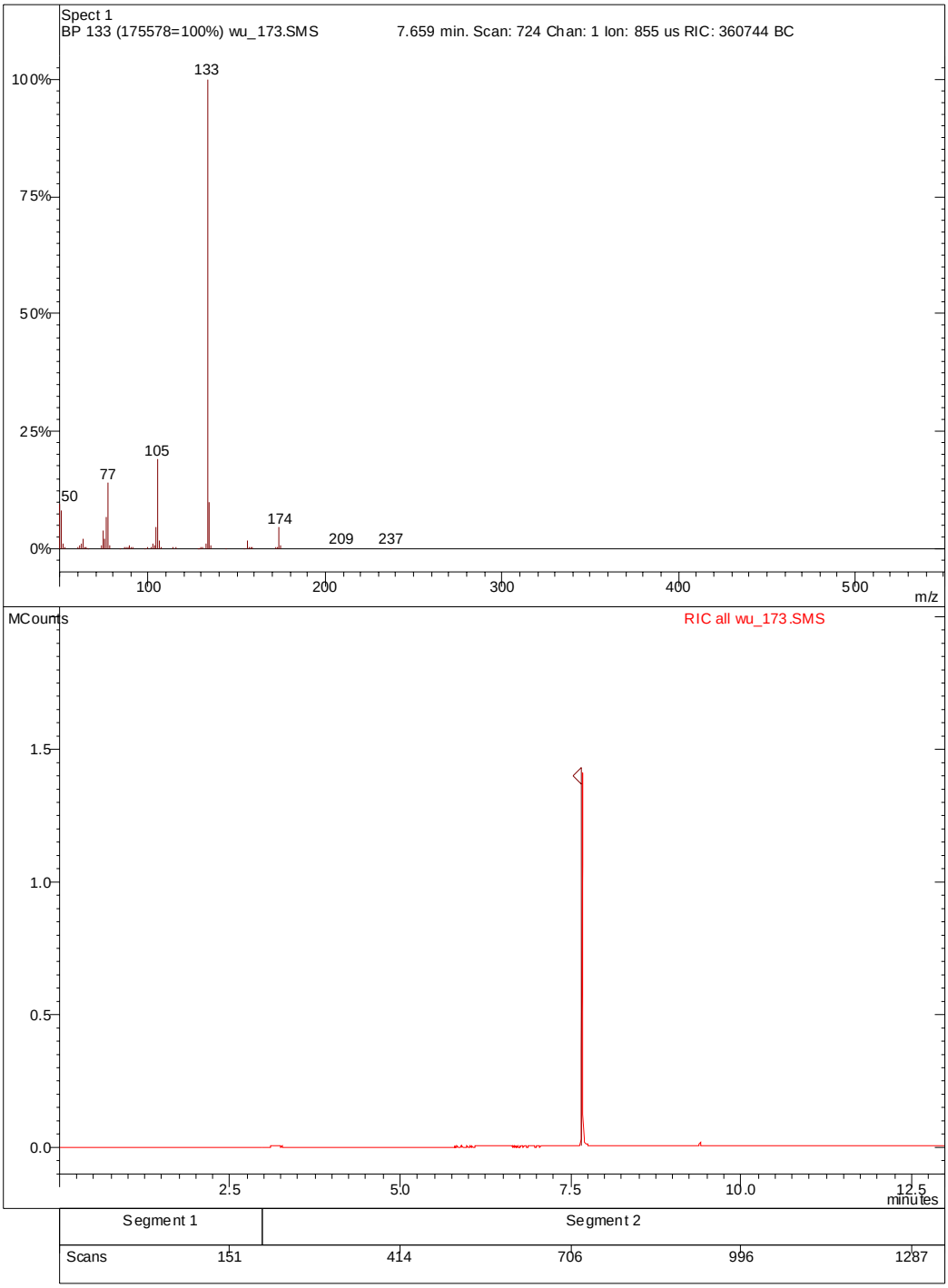


(3-Oxo-1,3-dihydro-isobenzofuran-1-yl)acetonitrile (3g)

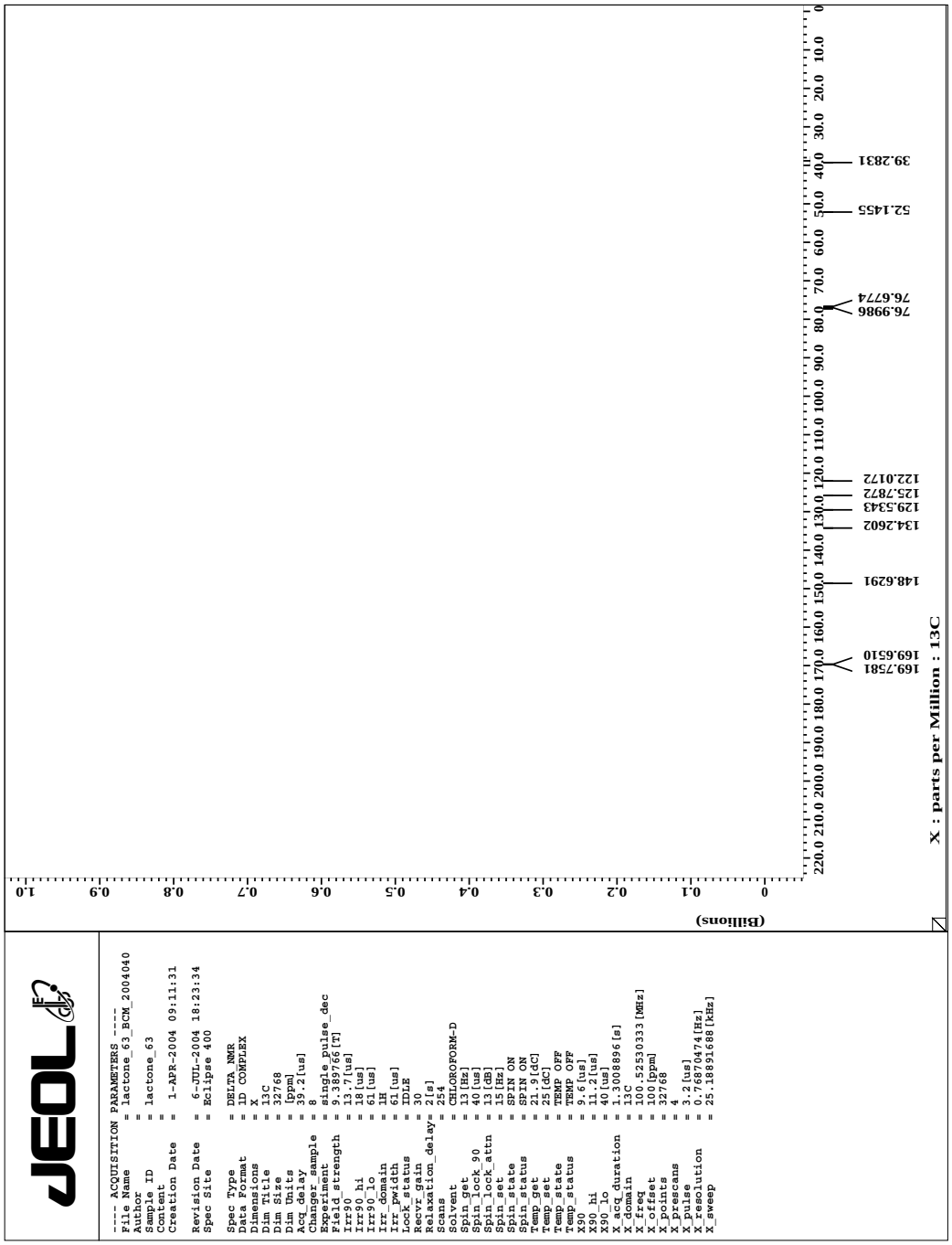


Chromatogram Plot

File: i:\wu_173.sms
Sample: wu_173 Operator: Operator
Scan Range: 1 - 1344 Time Range: 0.00 - 12.99 min. Date: 03/25/04 10:39
Sample Notes: ROUTINE



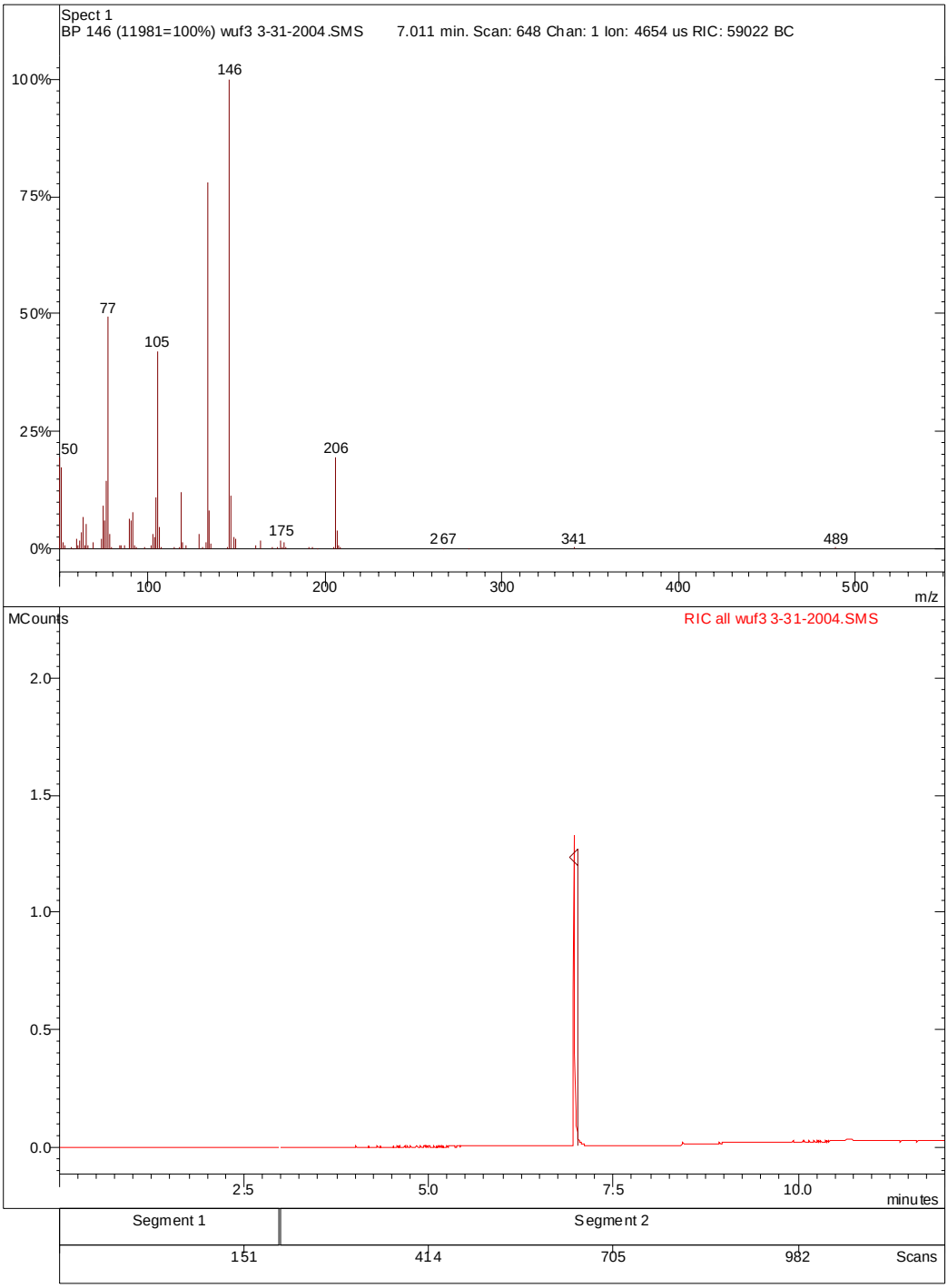
(3- Oxo-1,3-dihydro-isobenzofuran-1-yl)-acetic acid methyl ester (3h)



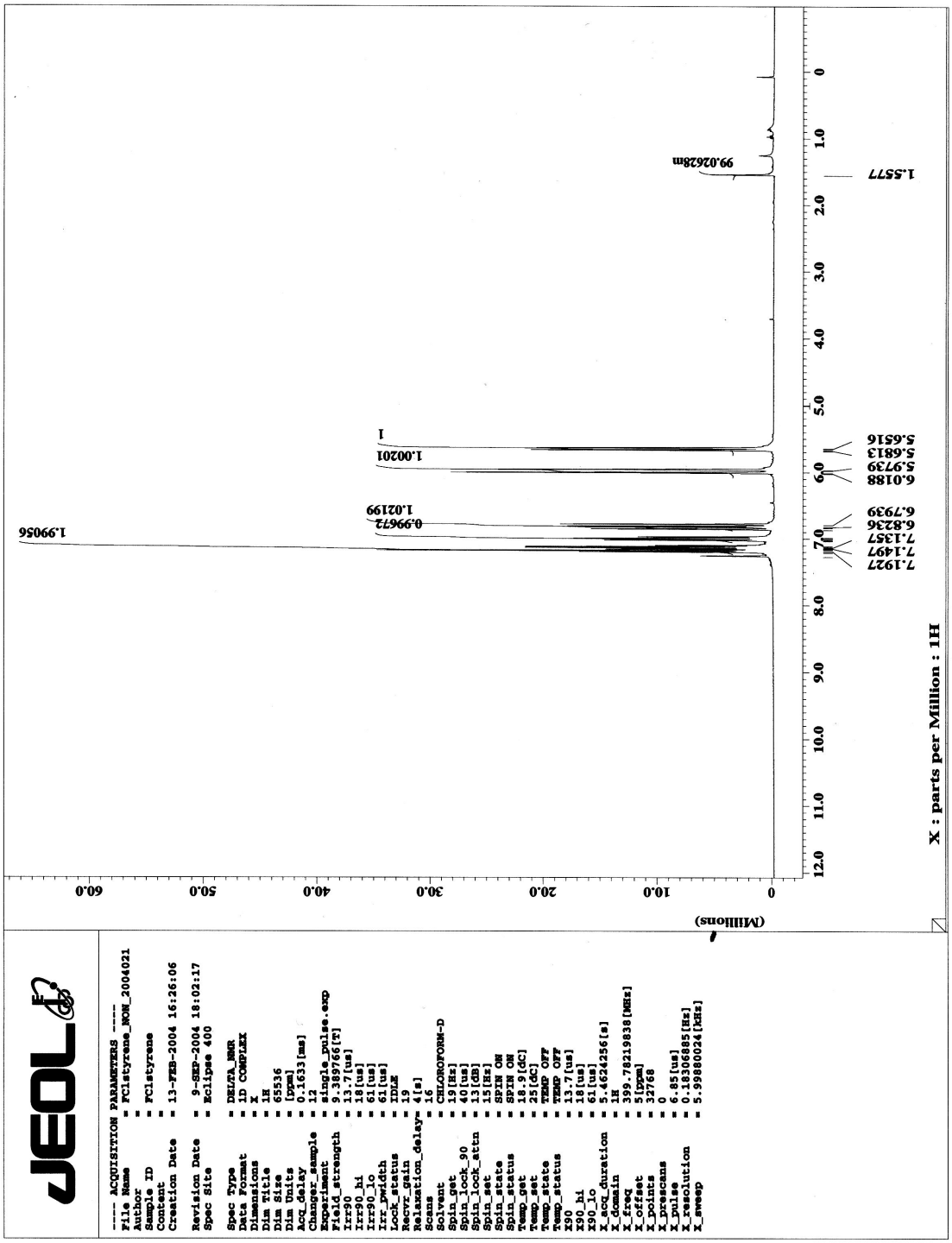
Chromatogram Plot

File: i:\wuf3 3-31-2004.sms
Sample: wuf3
Scan Range: 1 - 1191 Time Range: 0.00 - 11.99 min.
Sample Notes: Routine

Operator: Org Farm Kemi
Date: 03/31/04 19:41



1-Chloro-3-fluoro-2-vinyl-benzene (4b)



JEOL

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---- ACQUISITION PARAMETERS ----
File Name   = FClstyrene_C_20040212.4
Author      =
Sample ID   = BCM/REF
Content     = Bilevel.complete.decoupling
Creation Date= 12-FEB-2004 19:29:37

Revision Date=  9-SEP-2004 17:55:44
Spec Site   =

Spec Type   = GSX_NMR
Data Format= 1D COMPLEX
Dimensions = X
Dim Title   = 13C
Dim Size    = 16384
Dim Units   = [ppm]
Acq_delay   = 47.5[us]
Irr_domain  = 1H
Solvent     = CDCL3
Temp_get    = 19.5[dC]
X90         = 5[us]
X_domain    = 13C
X_freq      = 67.9402[MHz]
X_pulse     = 5[us]
X_sweep     = 20[kHz]
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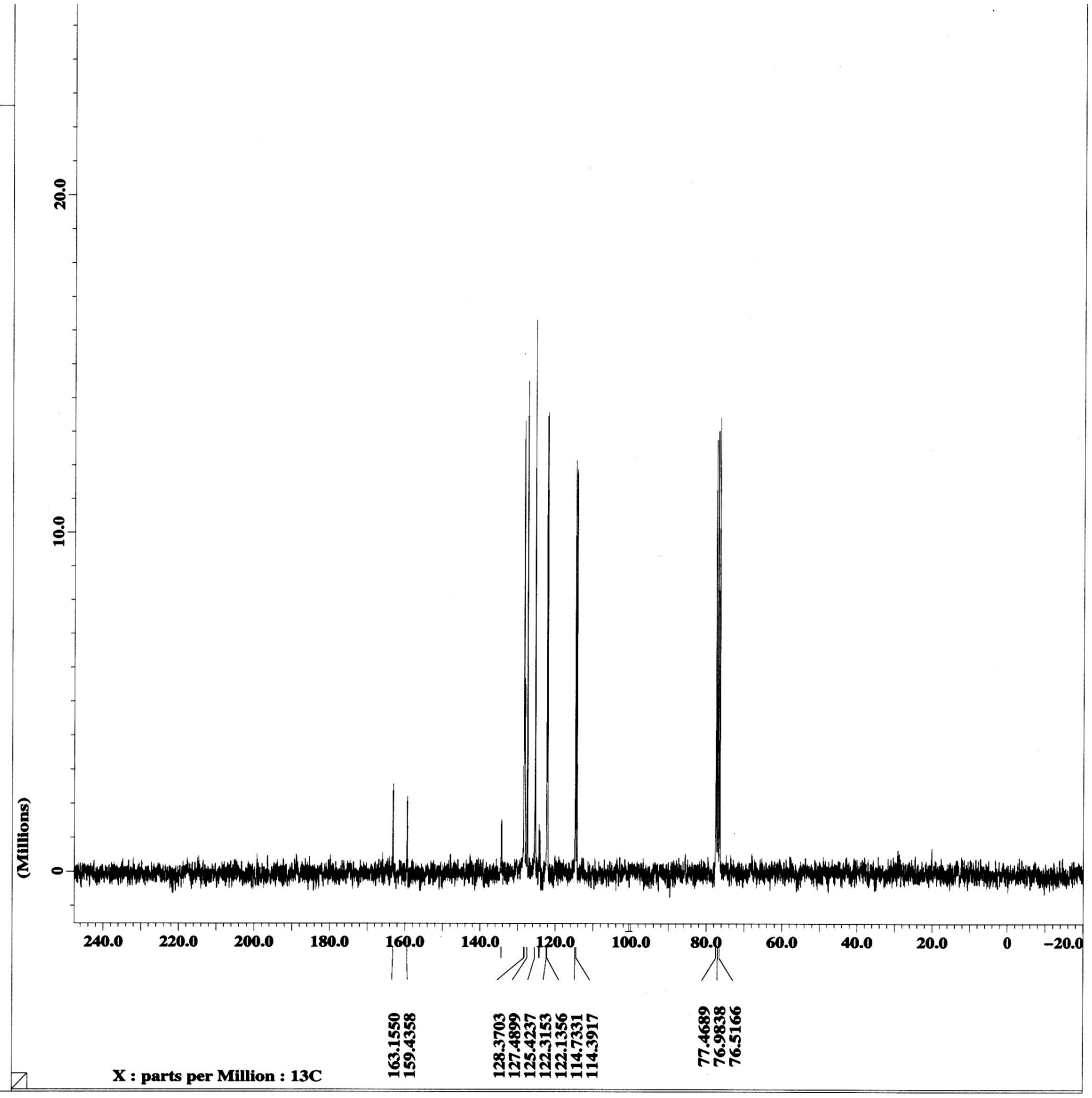
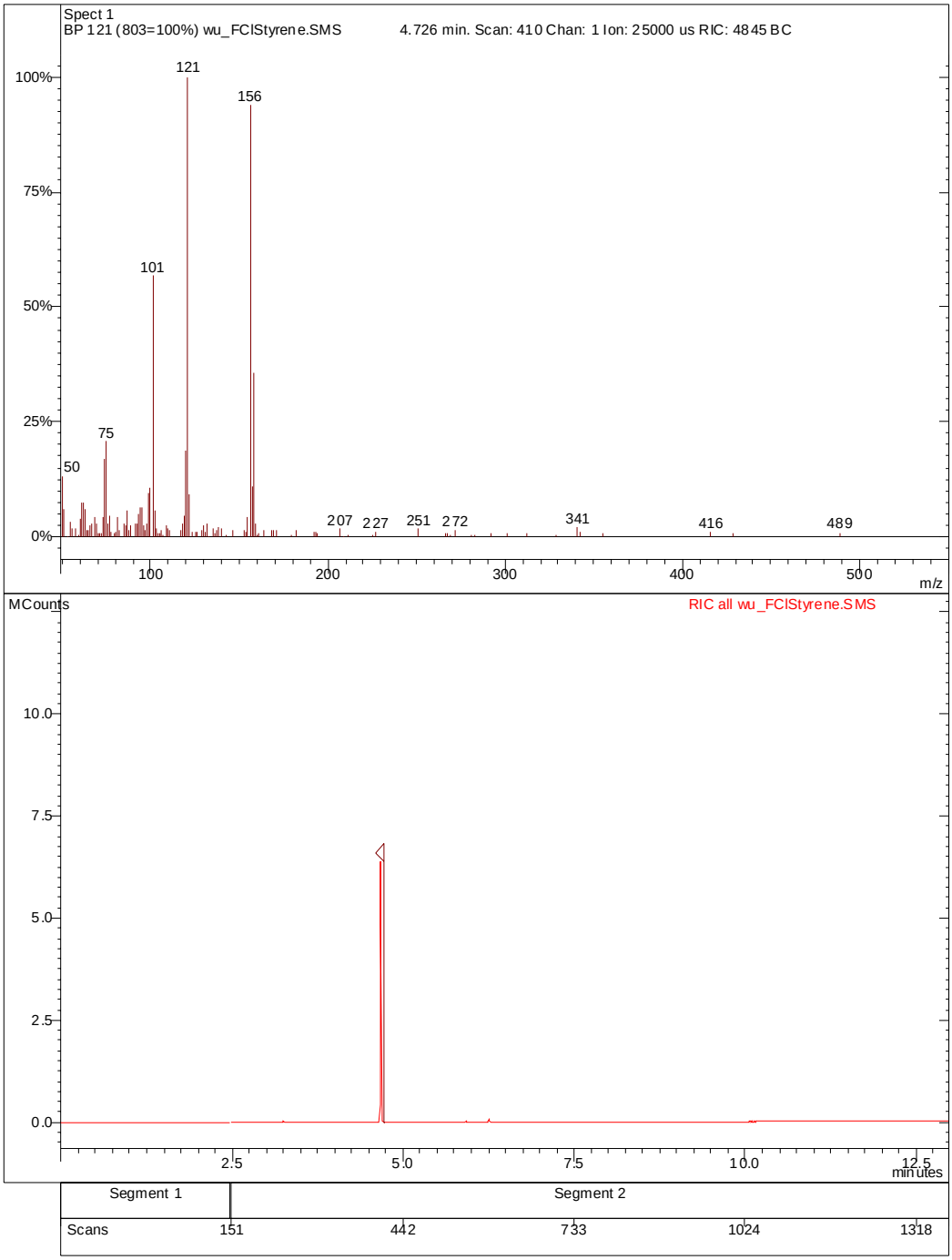


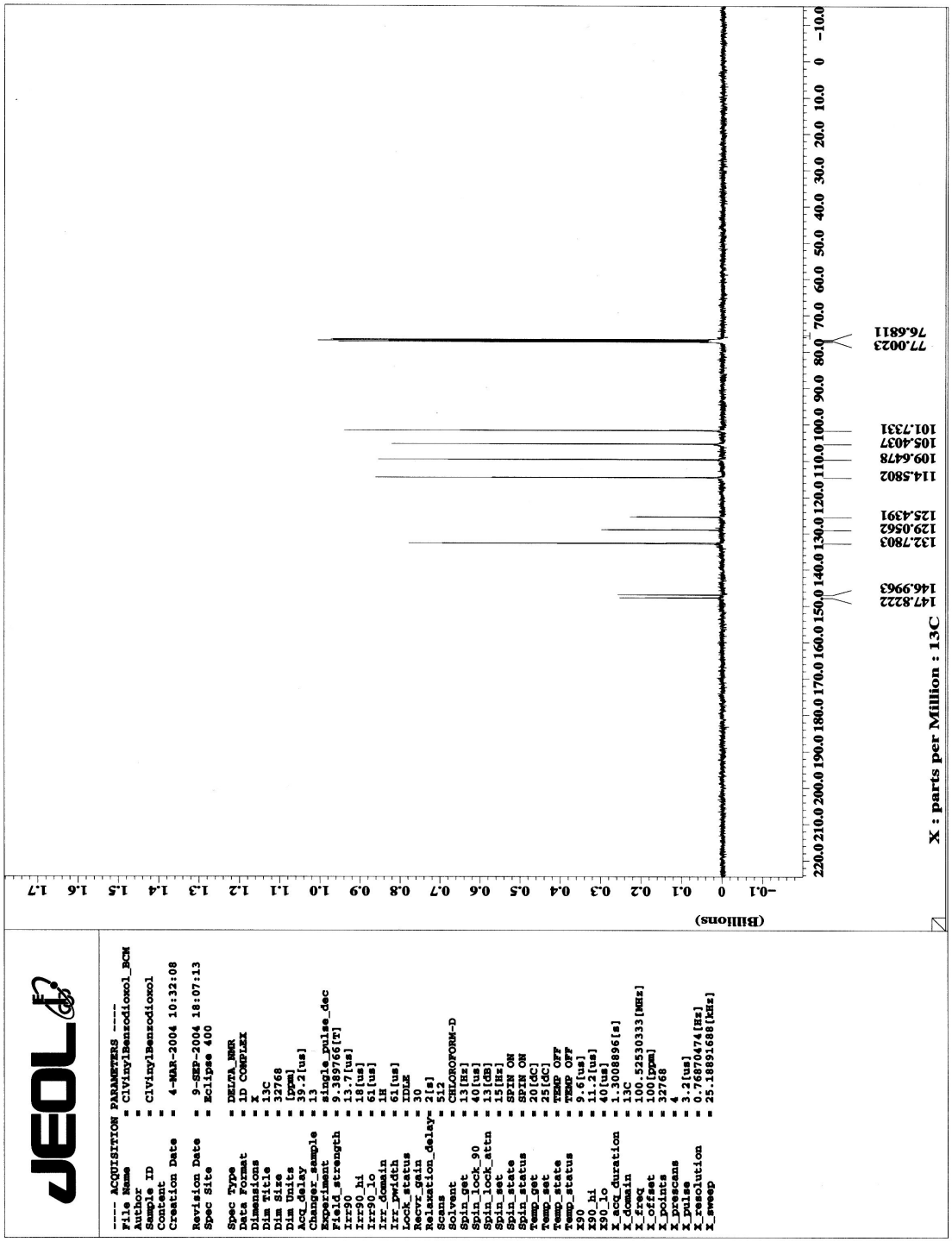
Chromatogram Plot

File: i:\wu_fclstyrene.sms
Sample: wu_FClStyrene
Scan Range: 1 - 1374 Time Range: 0.00 - 12.98 min.
Sample Notes: today

Operator: Operator
Date: 09/09/04 19:18



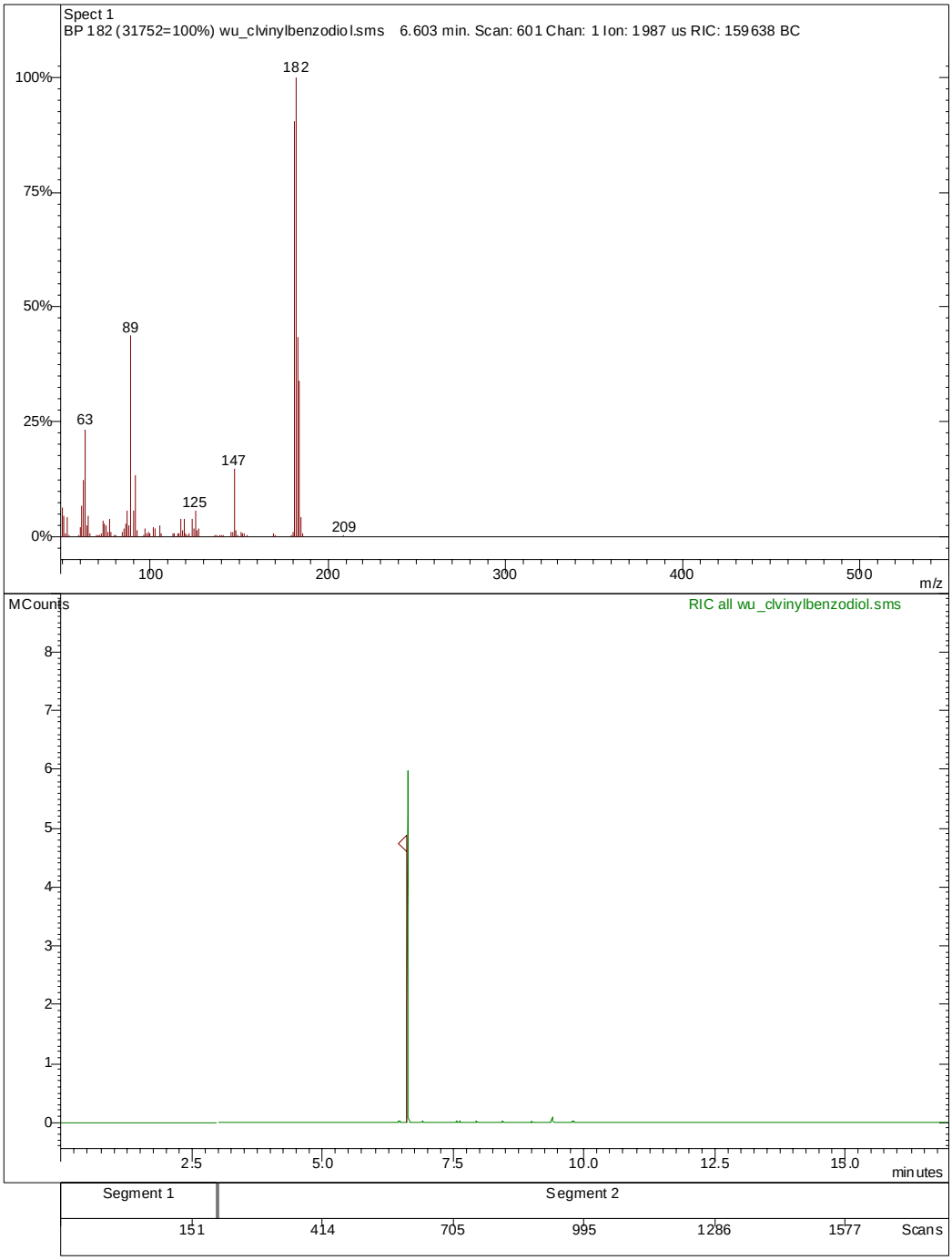
5-Chloro-6-vinyl-benzo[1,3]dioxole (4e)



Chromatogram Plot

File: i:\wu_clvinylbenzodiol.sms
Sample: wu_ClVinylBenzodiol
Scan Range: 1 - 1808 Time Range: 0.00 - 16.98 min.
Sample Notes: ROUTINE

Operator: Operator
Date: 03/04/04 11:34



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