Bispyridinium Dienes, Histone Deacetylase Inhibitors With Selective Activities

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2-(11-Bromoundecyloxy)-tetrahydro-2*H*-pyran (4a). To a stirred solution of 11-bromoundecanol (10.0 g, 33.80 mmol) in dichloromethane (100 mL) was added pyridinum *para*-toluenesulfonate (100 mg, 0.39 mmol, 1 mol%) and dihydropyran (5.45 mL, 59.70 mmol, 1.5 equiv). The resulting colourless solution was stirred at 25 ºC for 14 h. Then it was washed with a 2M aqueous Na_2CO_3 solution (2 x 50 mL). The combined organic layers were dried over Na_2SO_4 and the solvents removed under reduced pressure. The residue was purified by flash chromatography (96:4, hexane-EtOAc) to afford 12.85 g (96% yield) of the title compound as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.58 (broad s, 1H), 3.9-3.8 (m, 1H), 3.8-3.7 (m, 1H), 3.5-3.4 (m, 1H), 3.41 (t, J = 6.9 Hz, 2H), 3.4-3.3 (m, 1H), 1.9-1.8 (m, 3H), 1.71 (t, J = 10.7 Hz, 1H), 1.6-1.5 (m, 5H), 1.5-1.2 (m, 15H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 98.8, 67.6, 62.3, 34.0, 32.8, 30.7, 29.7, 29.5, 29.4, 29.3, 28.7, 28.1, 26.2, 25.5, 19.7 ppm. IR (NaCl) ν 2924 (s, C–H), 2852 (s, C–H), 1635 (w), 1459 (m), 1350 (m), 1258 (w), 1120 (s), 1075 (s), 1028 (s). MS (EI$^+$) *m/z* (%) 335 ([M-1]$^+$ [^{81}Br], 2), 333 ([M-1]$^+$ [^{79}Br], 2), 164 (33), 162 (38), 150 (57), 147 (69), 101 (80), 97 (88), 85 (100). HRMS (EI$^+$) calcd for $C_{16}H_{30}{}^{81}BrO_2$, 335.1409 and $C_{16}H_{30}{}^{79}BrO_2$, 333.1429; found 335.1410 and 333.1443.

2-(12-Bromododecyloxy)-tetrahydro-2*H*-pyran (4b). Following the same procedure described for **4a**, protected alcohol **4b** was obtained in 93% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.57 (broad s, 1H), 3.9-3.8 (m, 1H), 3.8-3.7 (m, 1H), 3.5-3.4 (m, 1H), 3.40 (t, J = 6.9 Hz, 2H), 3.4-3.3 (m, 1H), 1.9-1.8 (m, 3H), 1.7-1.6 (m, 1H), 1.6-1.4 (m, 6H), 1.4-1.2 (m, 16H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 98.8, 67.6, 62.3, 34.0, 32.8, 30.7, 29.7, 29.6, 29.5, 29.4, 29.3, 28.7, 28.1, 26.2, 25.5, 19.7 ppm. IR (NaCl) ν 2913 (s, C–H), 2849 (s, C–H), 1458 (m), 1349 (m), 1257 (w), 1200 (w), 1120 (s), 1075 (s), 1027 (s). MS (EI$^+$) *m/z* (%) 349 ([M-1]$^+$ [^{81}Br], 1), 347 ([M-1]$^+$ [^{79}Br], 1), 150 (21), 148 (22), 111 (13), 101 (24), 97 (74), 85 (100). HRMS (EI^+) calcd for $C_{17}H_{32}{}^{81}BrO_2$, 349.1565 and $C_{17}H_{32}{}^{79}BrO_2$, 347.1586; found 349.1573 and 347.1601.

3-[12-(Tetrahydro-2*H*-pyran-2-yloxy)dodecyl]pyridine (5a). *n*-BuLi (16.8 mL, 26.8 mmol, 1.6 M in hexanes) was added dropwise to a stirred solution of diisopropylamine (3.8 mL, 26.80 mmol) in THF (17 mL) at 0 ºC. The slightly yellow solution was stirred at 0 ºC for 30 min. DMPU (3.2 mL, 26.8 mmol) was added and stirring at 0 ºC was continued for another 20 min. A solution of 3-methylpyridine (2.6 mL, 26.8 mmol) in THF (8 mL) was added dropwise to the reaction mixture. The resulting deep red solution was stirred at 0 ºC for further 30 min. Then it was cooled down to -78 ºC and a solution of **4a** (3.0 g, 8.95 mmol) in THF (8 mL) was added dropwise. The reaction mixture was stirred at -78ºC for 1 h and then it was allowed to reach slowly room temperature. The excess organolithium was quenched by the addition of a saturated NH_4Cl solution (15 mL) and H_2O (10 mL). The mixture was extracted with EtOAc (3 x 70 mL), the combined organic layers were dried over Na_2SO_4 and the solvents removed under reduced pressure. The residue was purified by flash chromatography (60:40, hexane-EtOAc) to afford 2.53 g (76% yield) of the title compound as a colourless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 8.42-8.41 (m, 2H), 7.48 (d, J = 7.7 Hz, 1H), 7.19 (dd, J = 7.7, 4.8 Hz, 1H), 4.57 (broad s, 1H), 3.9-3.8 (m, 1H), 3.7-3.6 (m, 1H), 3.5-3.4 (m, 1H), 3.4-3.3 (m, 1H), 2.7-2.6 (m, 1H), 1.9-1.8 (m, 1H), 1.7-1.6 (m, 1H), 1.6-1.4 (m, 8H), 1.4-1.12 (m, 16H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.9, 147.1, 137.9, 135.6, 123.1, 98.8, 67.6, 62.3, 32.9, 31.1, 30.7, 29.7, 29.5, 29.4 (2x), 29.3, 29.1, 26.2, 25.4, 19.6 ppm. IR (NaCl) ν 2924 (s, C–H), 2851 (s, C–H), 1582 (m), 1461 (m), 1371 (m), 1119 (s), 1075 (s), 1026 (s), 982 (m), 712 (m). MS (EI^+) *m/z* (%) 346 ($[M-1]^+$, 2), 318 (7), 262 (97), 246 (100), 232 (38), 218 (32), 204 (51), 190 (38), 176

(25), 148 (30), 106 (96), 93 (95). HRMS (EI$^+$) calcd for $C_{22}H_{36}NO_2$, 346.2746; found 346.2739.

3-[13-(Tetrahydro-2*H*-pyran-2-yloxy)tridecyl]pyridine (5b). Following the same procedure described for **5a**, pyridine **5b** was obtained in 70% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45-8.41 (m, 2H), 7.48 (d, *J* = 7.7 Hz, 1H), 7.19 (dd, *J* = 7.7, 4.8 Hz, 1H), 4.57 (broad s, 1H), 3.9-3.8 (m, 1H), 3.8-3.7 (m, 1H), 3.5-3.4 (m, 1H), 3.4-3.3 (1H), 2.7-2.6 (m, 1H), 1.9-1.8 (m, 1H), 1.7-1.6 (m, 1H), 1.6-1.5 (m, 8H), 1.42-1.2 (m, 18H) ppm ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.8, 147.0, 137.7, 135.5, 123.0, 98.6, 67.5, 62.1, 32.8, 30.9, 30.6, 29.6, 29.4, 29.3 (2x), 29.2, 29.0, 26.1, 25.3, 19.5 ppm. IR (NaCl) ν 2912 (s, C–H), 2849 (s, C–H), 1574 (w), 1459 (m), 1422 (m), 1349 (m), 1120 (s), 1074 (s), 1026 (s), 987 (m), 713 (m). MS (FAB$^+$) *m/z* (%) 362 ([M+1]$^+$, 45), 278 (100), 260 (27). HRMS (EI$^+$) calcd for $C_{23}H_{38}NO_2$, 360.2903; found 360.2911.

1-(Oct-7-enyl)-3-[12-(tetrahydro-2*H*-pyran-2-yloxy)dodecyl]pyridinium Bromide (6a). 8-Bromo-1-octene (2 mL, 11.93 mmol, 1 equiv) was added to a solution of **5a** (4.15 g, 11.93 mmol) in acetonitrile (20 mL). The resulting solution was heated at reflux and the disappearance of the starting material was monitored by TLC. After completion of the reaction (usually 24 h), the solvent was removed under reduced pressure. The residue was purified by flash chromatography (90:10, CH_2Cl_2-MeOH) to afford 6.16 g (96% yield) of the title compound as a viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.41 (d, *J* = 5.9 Hz, 1H), 9.29 (s, 1H), 8.17 (d, *J* = 8.0 Hz, 1H), 8.03 (dd, *J* = 8.0, 5.9 Hz, 1H), 5.7-5.6 (m, 1H), 4.94 (t, *J* = 7.4 Hz, 2H), 4.9-4.8 (m, 2H), 4.5-4.4 (m, 1H), 3.80-3.7- (m, 1H), 3.7-3.6 (m, 1H), 3.5-3.4 (m, 1H), 3.3-3.2 (m, 1H), 2.83 (t, *J* = 6.9 Hz, 2H), 2.0-1.9 (m, 4H), 1.8-1.7 (m, 1H), 1.7-1.6 (m, 3H), 1.5-1.4 (m, 6H), 1.4-1.1 (m, 22H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.3, 143.8, 143.6, 142.6, 138.3, 127.9, 114.2, 98.6, 67.4, 62.1, 61.4, 33.3, 32.2, 31.6, 30.5, 30.1, 29.4, 29.2, 29.1, 28.9, 28.7, 28.2, 28.1, 25.9, 25.5, 25.1, 19.4 ppm. IR (NaCl) ν 2924 (s, C–H), 2852 (s, C–H), 1635 (m), 1503 (m), 1461 (m), 1351 (w), 1120 (s), 1075 (s), 1026 (s), 906 (m), 690 (m). MS (EI^+) *m/z* (%) 458 ($[M-Br^-]^+$, 4), 374 (1), 262 (56), 246 (95), 106 (100).

1-(Oct-7-enyl)-3-[13-(tetrahydro-2*H*-pyran-2-yloxy)tridecyl]pyridinium Bromide (6b). Following the same procedure described for **6a**, pyridinium salt **6b** was obtained in 98% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.37 (d, J = 5.2 Hz, 1H), 9.14 (s, 1H), 8.21 (d, J = 7.7 Hz, 1H) 8.01 (dd, J = 7.7, 6.3 Hz, 1H), 5.7-5.6 (m, 1H), 5.02 (t, J = 7.4 Hz, 2H), 5.0-4.9 (m, 2H), 3.9-3.8 (m, 1H), 3.7-3.6 (m, 1H), 3.5-3.4 (m, 1H), 3.4-3.3 (m, 1H), 2.89 (d, J = 7.8 Hz, 2H), 2.1-1.9 (m, 4H), 1.9-1.8 (m, 1H), 1.7-1.6 (m, 3H), 1.6-1.5 (m, 6H), 1.4-1.2 (m, 24H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 144.1, 143.8, 142.7, 138.5, 127.8, 114.5, 98.8, 67.6, 62.3, 61.8, 33.4, 32.7, 31.9, 30.7, 30.4, 29.7, 29.5, 29.4, 29.2, 29.0, 28.4 (2x), 26.1, 25.8, 25.4, 19.7 ppm. IR (NaCl) ν 2925 (s, C–H), 2853 (s, C–H), 1637 (m), 1505 (m), 1465 (m), 1352 (w), 1136 (m), 1120 (s), 1032 (s), 906 (m), 691 (m). MS (FAB^+) *m/z* (%) 472 ($[M-Br^-]^+$, 100). HRMS (FAB^+) calcd for $C_{31}H_{54}NO_2^+$, 472.4155; found 472.4144.

3-(12-Hydroxydodecyl)-1-(oct-7-enyl)pyridinium Bromide (7a). A 3M aqueous solution of HCl (4.4 mL, 1.1 equiv) was added to a solution of **6a** (6.0 g, 11.14 mmol) in MeOH (44 mL). The reaction mixture was stirred at 25 ºC for 16 h. Most of the solvents were removed under reduced pressure to give a viscous oil. The residue was disolved in dichloromethane (80 mL) and washed with a saturated $NaHCO_3$ aqueous solution (80 mL). The organic layer was dried with Na_2SO_4 and the solvents were removed under reduced pressure. The residue was purified by flash chromatography (90:10, CH_2Cl_2-MeOH) to afford 4.45 g (88% yield) of the title compound as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.33 (s, 1H), 9.28 (d, J = 5.8 Hz, 1H), 8.20 (d, J = 8.0 Hz, 1H), 8.04 (dd, J = 8.0, 5.8 Hz, 1H), 5.67 (tdd, J = 13.2, 10.0, 6.7 Hz, 1H), 4.9-

4.8 (m, 4H), 3.52 (t, J = 6.7 Hz, 2H), 2.83 (t, J = 7.8 Hz, 2H), 2.4-2.3 (m, 1H), 2.0-1.9 (m, 4H), 1.7-1.56 (m, 2H), 1.5-1.4 (m, 2H), 1.4-1.1 (m, 23H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 144.0, 143.9, 142.4, 138.4, 127.9, 114.3, 62.4, 61.4, 33.3, 32.5, 32.4, 31.7, 30.2, 29.2 (2x), 28.9, 28.7, 28.3, 28.2, 25.6, 25.5 ppm. IR (NaCl) ν 3500-3100 (br, O-H), 2918 (s, C–H), 2847 (s, C–H), 1634 (m), 1513 (m), 1465 (m), 1058 (s), 922 (m). MS (EI^+) m/z (%) 374 ($[M\text{-}Br^-]^+$, 12), 262 (10), 246 (15), 232 (11), 204 (12), 190 (12), 120 (11), 106 (100). HRMS (EI^+) calcd for $C_{25}H_{44}NO_2^+$, 374.3423; found 374.3430.

3-(13-Hydroxytridecyl)-1-(oct-7-enyl)pyridinium Bromide (7b). Following the same procedure described for **7a**, alcohol **7b** was obtained in 82% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.35 (d, J = 6.0 Hz, 1H), 9.29 (s, 1H), 8.21 (d, J = 8.0 Hz, 1H), 8.05 (dd, J = 8.0, 6.0 Hz, 1H), 5.72 (tdd, J = 16.9, 10.2, 6.7 Hz, 1H), 4.95 (t, J = 7.4, Hz, 2H), 4.93-4.86 (m, 2H), 3.59 (t, J = 6.6 Hz, 1H), 2.9-2.8 (m, 2H), 2.1-1.9 (m, 6H), 1.7-1.6 (m, 2H), 1.6-1.5 (m, 2H), 1.4-1.1 (m, 25H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 144.0, 143.9, 142.4, 138.4, 127.9, 114.3, 62.4, 61.4, 33.3, 32.5, 32.4, 31.7, 30.2, 29.2 (2x), 28.9, 28.7, 28.3, 28.2, 25.6, 25.5 ppm. IR (NaCl) ν 3500-3100 (br, O-H), 2922 (s, C–H), 2848 (s, C–H), 1636 (m), 1513 (m), 1466 (m), 1306 (w), 1172 (w), 1056 (s). MS (EI^+) m/z (%) 388 ($[M\text{-}Br^-]^+$, 11), 260 (10), 258 (12), 218 (16), 216 (11), 204 (15), 150 (40), 148 (41), 106 (100). HRMS (EI^+) calcd for $C_{26}H_{46}NO_2^+$, 388.3579; found 374.3585.

3-(12-Chlorododecyl)-1-(oct-7-enyl)pyridinium Chloride (8a). Thionyl chloride (0.95 mL, 13.02 mmol) was added to a solution of **7a** (4.1 g, 10.02 mmol) in dichloromethane (37 mL) at 0 ºC. The mixture was maintained at 0 ºC for 30 min and then it was stirred at 25 ºC for 6h. Dichloromethane (60 mL) was added and the resulting solution was washed with a 0.2 N NaOH solution (2 x 50 mL) and brine (2 x

50 mL). The organic layer was dried over Na_2SO_4 and the solvents were removed under reduced pressure. The crude residue was purified by flash chromatography (90:10, CH_2Cl_2-MeOH) to afford 3.9 g (91%) yield of the title compound as a viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.44 (d, *J* = 5.9 Hz, 1H), 9.17(s, 1H), 8.19 (d, *J* = 7.9 Hz, 1H), 8.01 (dd, *J* = 7.9, 5.9 Hz, 1H), 5.75 (tdd, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.01 (t, *J* = 7.4 Hz, 2H), 5.0-4.9 (m, 2H), 3.52 (t, *J* = 6.8, Hz, 2H), 2.87 (t, *J* = 6.9 Hz, 2H), 2.1-1.9 (m, 4H), 1.9-1.7 (m, 4H), 1.5-1.1 (m, 22H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.4, 144.0, 143.9, 142.7, 138.4, 127.9, 114.4, 61.6, 45.1, 33.3, 32.6, 32.4, 31.9, 30.3, 29.3 (2x), 29.2, 29.1, 28.9, 28.7, 28.3 (2x), 26.7, 25.7 ppm. IR (NaCl) ν 2924 (s, C–H), 2853 (s, C–H), 1637 (m), 1504 (m), 1462 (m), 1154 (m), 925 (m), 726 (s). MS (EI^+) *m/z* (%) 394 ($[M\text{-}Cl^-]^+[^{37}Cl]$, 8) 392 ($[M\text{-}Cl^-]^+[^{35}Cl]$, 48), 356 (7), 246 (94), 218 (15), 204 (18), 106 (100). HRMS (EI^+) calcd for $C_{25}H_{43}{}^{35}ClN^+$, 392.3084 and $C_{25}H_{43}{}^{37}ClN^+$, 394.3055; found 392.3082 and 394.3073.

3-(13-Chlorotridecyl)-1-(oct-7-enyl)pyridinium Chloride (8b). Following the same procedure, pyridinium chloride **8b** was obtained in 95% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.37 (d, *J* = 5.9 Hz, 1H), 9.27(s, 1H), 8.21 (d, *J* = 7.8 Hz, 1H), 8.1-8.0 (m, 1H), 5.8-5.7 (m, 1H), 5.0-4.9 (m, 2H), 4.9-4.8 (m, 2H), 3.5-3.4 (m, 2H), 2.9-2.8 (m, 2H), 2.1-1.9 (m, 4H), 1.8-1.6 (m, 4H), 1.5-1.1 (m, 24H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.2, 143.8, 143.7, 142.2, 138.1, 127.6, 114.0, 61.1, 44.8, 33.0, 32.1, 31.5, 30.0, 29.0 (2x), 28.9, 28.8, 28.6, 28.3, 28.0 (2x), 26.3, 25.4 ppm. IR (NaCl) ν 2925 (s, C–H), 2853 (s, C–H), 1633 (m), 1504 (m), 1463 (m), 1154 (m), 726 (s). MS (EI^+) *m/z* (%) 408 ($[M\text{-}Cl^-]^+[^{37}Cl]$, 33), 406 ($[M\text{-}Cl^-]^+[^{35}Cl]$, 100), 370 (18), 260 (73), 232 (23), 174 (21), 150 (52), 148 (50). HRMS (EI^+) calcd for $C_{26}H_{45}{}^{35}ClN^+$, 406.3241 and $C_{26}H_{45}{}^{37}ClN^+$, 408.3211; found 406.3235 and 408.3209.

Preparation of pyridines 9a-c. See procedure for pyridines **5a,b**

3-(Pent-4-enyl)pyridine (9a). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.5-8.40 (m, 2H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.19 (dd, *J* = 7.8, 4.9 Hz, 1H), 5.81 (tdd, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.0-4.9 (m, 2H), 2.7-2.6 (m, 2H), 2.1-2.0 (m, 2H), 1.8-1.7 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.9, 147.2, 138.0, 137.4, 135.7, 123.2, 115.0, 33.0, 32.2, 30.1 ppm. IR (NaCl) ν 2932 (s, C-H), 2858 (w, C–H), 1640 (m), 1575 (m), 1478 (m), 1422 (s), 912 (s). MS (EI^+) *m/z* (%) 147 ($[M]^{+\cdot}$, 9), 146 ($[M-1]^+$, 4), 118 (18), 105 (100), 93, (20), 92 (57), 78 (14). HRMS (EI^+) calcd for $C_{10}H_{13}N$, 147.1048; found 147.1050.

3-(Hex-5-enyl)pyridine (9b). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.5-8.4 (m, 2H), 7.46 (d, *J* = 7.8 Hz, 1H), 7.17 (dd, *J* = 7.8, 4.9 Hz, 1H), 5.77 (tdd, *J* = 16.9, 10.1, 6.7 Hz, 1H), 4.98 (dd, *J* = 17.1, 1.4 Hz, 1H), 4.93 (dd, *J* = 10.2, 1.2 Hz, 1H), 2.59 (t, *J* = 7.5 Hz, 2H), 2.1-2.0 (m, 2H), 1.7-1.6 (m, 2H), 1.5-1.4 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.8, 147.1, 138.4, 137.6, 135.6, 123.1, 114.5, 33.4, 32.7, 30.4, 28.2 ppm. IR (NaCl) ν 2907 (w, C–H), 2858 (w, C–H), 1639 (m), 1575 (m), 1477 (m), 1421 (s), 1189 (w), 1025 (m), 992 (m), 909 (s), 795 (m), 712 (s). MS (EI^+) *m/z* (%) 161 ($[M]^{+\cdot}$, 3), 160 (8), 137 (11), 133 (65), 118 (19), 105 (29), 92 (47), 81 (11), 69 (100). HRMS (EI^+) calcd for $C_{11}H_{15}N$ 161.1204; found 161.1210.

3-(Oct-7-enyl)pyridine (9c). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.5-8.4 (m, 2H), 7.47 (d, *J* = 7.6 Hz, 1H), 7.19 (dd, *J* = 7.6, 4.9 Hz, 1H), 5.79 (tdd, *J* = 17.0, 10.2, 6.7 Hz, 1H), 4.98 (dd, *J* = 17.1, 1.3 Hz, 1H), 4.92 (ddd, *J* = 10.2, 2.0, 1.0 Hz, 1H), 2.59 (t, *J* = 7.1 Hz, 2H), 2.1-2.0 (m, 2H), 1.7-1.6 (m, 1H), 1.4-1.3 (m, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.9, 147.1, 139.0, 137.9, 135.7, 123.2, 114.2, 33.7, 32.9, 31.0, 28.9, 28.8, 28.7 ppm. IR (NaCl) ν 2925 (s, C–H), 2854 (w, C–H), 1639 (m), 1574 (m), 1477 (m), 1421 (s), 1188 (w), 1026 (m), 994 (m), 907 (s), 793 (m), 712 (s). MS (EI^+) *m/z* (%) 189 ($[M]^{+\cdot}$, 21), 188 (25), 146 (100), 133 (27), 106 (27), 105 (19), 93 (29), 92 (63).

1-(Oct-7-enyl)-3-{12-[3-(oct-7-enyl)pyridinium-1-yl]dodecyl}pyridinium Chloride Iodide (10c). ^{1}H NMR (400 MHz, CD_3OD) δ 9.13 (br s, 2H), 9.0-8.9 (m, 2H), 8.53 (d, *J* = 8.0 Hz, 2H), 8.07 (dd, *J* = 8.0, 6.3 Hz, 2H), 5.8-5.7 (m, 2H), 4.98 (d, *J* = 17.1 Hz, 2H), 4.91 (d, *J* = 10.1 Hz, 2H), 4.73 (t, *J* = 7.6 Hz, 4H), 2.93 (t, *J* = 7.7 Hz, 4H), 2.1-2.0 (m, 8H), 1.8-1.7 (m, 4H), 1.5-1.2 (m, 28H) ppm. ^{13}C NMR (100 MHz, CD_3OD) δ 146.7, 145.6, 145.5, 145.3, 143.2, 139.9, 139.8, 129.0, 115.0, 114.9, 62.7, 34.7, 34.6, 33.4 (2x), 32.5, 31.5, 31.4, 30.7, 30.6 (2x), 30.5, 30.1, 29.9, 29.8 (2x), 29.7, 29.5, 26.9 ppm. IR (NaCl) ν 3015 (s), 2924 (s, C–H), 2853 (s, C–H), 1633 (m), 1504 (s), 1457 (s), 1153 (m), 910 s), 732 (s), 688 (s). HRMS (ESI$^+$) *m/z* calcd for $C_{38}H_{62}IN_2^+$ 673.3951 (found 673.3945). $C_{38}H_{62}N_2^{2+}$ 273.2451 (found 273.2447).

1-(Oct-7-enyl)-3-{13-[3-(pent-4-enyl)pyridinium-1-yl]tridecyl}pyridinium Chloride Iodide (10d). ^{1}H NMR (400 MHz, CD_2Cl_2) δ 9.36 (br s, 1H), 9.34 (br s, 1H), 9.19 (d, *J* = 6.0 Hz, 1H), 9.16 (d, *J* = 6.0 Hz, 1H), 8.32 (d, *J* = 8.0 Hz, 2H), 8.05 (dd, *J* = 8.0, 6.0 Hz, 2H), 5.9-5.7 (m, 2H), 5.1-4.9 (m, 4H), 4.85 (t, *J* = 7.4 Hz, 4H), 2.9-2.8 (m, 4H), 2.1-2.0 (m, 2H), 2.06-1.9 (m, 6H), 1.8-1.7 (m, 4H), 1.5-1.1 (m, 24H) ppm. ^{13}C NMR (100 MHz, CD_2Cl_2) δ 145.2, 144.2, 144.0, 143.9, 143.8, 142.2, 142.1, 138.8, 137.5, 128.0, 115.4, 114.2, 61.5, 61.4, 33.5, 32.8, 32.4, 31.8, 31.7, 30.3, 29.4, 29.3, 29.2, 29.1, 29.0, 28.8 (2x), 28.4, 28.3, 25.8, 25.7 ppm. IR (NaCl) ν 3016 (m), 2925 (s, C–H), 2853 (w, C–H), 1631 (m), 1503 (s), 1460 (m), 1152 (m), 911 (m), 688 (s). MS (ESI$^+$) (%) 645 ([M-Cl$^-$]$^{+\cdot}$, 5). HRMS (ESI$^+$) *m/z* calcd for $C_{36}H_{58}IN_2^+$ 645.3639 (found 645.3626), $C_{36}H_{58}N_2^{2+}$ 259.2294 (found 259.2290).

3-(Hex-5-enyl)-1-{13-[1-(oct-7-enyl)pyridinium-3-yl]tridecyl}pyridinium Chloride Iodide (10e). ^{1}H NMR (400 MHz, CD_3OD) δ 9.05 (br s, 2H), 8.90 (t, *J* = 5.3 Hz, 2H), 8.49 (d, *J* = 8.0 Hz, 2H), 8.04 (dd, *J* = 7.8, 6.2 Hz, 2H), 5.8-5-7 (m, 2H), 5.0-4.9 (m, 4H), 4.66 (t, *J* = 7.6 Hz, 4H), 2.9-2.8 (m, 4H), 2.2-2.1 (m, 2H), 2.0-1.9 (m, 6H), 1.8-1.7 (m, 4H), 1.5-1.4 (m, 2H), 1.4-1.2 (m, 24H) ppm. ^{13}C NMR (100 MHz, CD_3OD) δ 146.7, 145.7, 145.6, 145.3, 143.4, 143.3, 139.9, 139.6, 129.0, 115.4, 115.0, 62.8 (2x), 34.7, 34.5, 33.5, 33.3, 32.6, 32.5, 31.6, 31.0, 30.7, 30.6 (2x), 30.5, 30.4, 30.1 (2x), 29.8, 29.6, 29.4, 27.2, 27.0 ppm. IR (NaCl) ν 3016 (m), 2925 (s, C–H), 2853 (s, C–H), 1631 (m), 1505 (s), 1460 (m), 1152 (m), 911 (m), 688 (s). HRMS (ESI$^+$) *m/z* calcd for $C_{37}H_{60}IN_2^+$ 659.3795 (found 659.3787), $C_{37}H_{60}N_2^{2+}$ 266.2372 (found 266.2369).

1-(Oct-7-enyl)-3-{13-[3-(oct-7-enyl)pyridinium-1-yl]tridecyl}pyridinium Chloride Iodide (10f). ^{1}H NMR (400 MHz, CD_3OD) δ 9.02 (br s, 2H), 8.9-8.8 (m, 2H), 8.48 (d, *J* = 7.7 Hz, 2H), 8.03 (dd, *J* = 7.7, 6.4 Hz, 2H), 5.8-5.7 (m, 2H), 4.98 (d, *J* = 17.1 Hz, 2H), 4.91 (d, *J* = 10.2 Hz, 2H), 4.65 (t, *J* = 7.5 Hz, 4H), 2.9-2.8 (m, 4H), 2.1-2.0 (m, 8H), 1.8-1.7 (m, 4H), 1.5-1.2 (m, 30H) ppm. ^{13}C NMR (100 MHz, CD_3OD) δ 146.7, 145.8, 145.7, 145.3, 143.3, 140.0, 139.9, 129.0, 115.0, 114.9, 62.9, 34.8, 34.7, 33.5, 32.5, 32.4, 31.5 (2x), 30.6 (2x), 30.4 (2x), 30.1, 30.0, 29.9 (2x) 29.8, 29.5, 27.1, 26.9 ppm. IR (NaCl) ν 3016 (m), 2925 (s, C–H), 2853 (s, C–H), 1631 (m), 1503 (s), 1460 (m), 1152 (m), 911 (m), 688 (s). HRMS (ESI$^+$) *m/z* calcd for $C_{39}H_{64}IN_2^+$ 687.4109 (found 687.4097), $C_{39}H_{64}N_2^{2+}$ 280.2529 (found 280.2524).

Cyclostellettamine A (1a). ^{1}H NMR (400 MHz, CD_3OD) δ 8.95 (br s, 2H, H2/2'), 8.85 (d, *J* = 5.5 Hz, 2H, H6/6'), 8.46 (d, *J* = 7.9 Hz, 2H, H4/4'), 8.02 (dd, *J* = 7.9, 5.5 Hz, 2H, H5/5'), 4.61 (t, *J* = 7.5 Hz, 4H, H7/7'), 2.9-2.8 (m, 4H, H18/18'), 2.1-2.0 (m, 4H, H8/8'), 1.7-1.6 (m, 4H, H17/17'), 1.5-1.3 (m, 32H, H9-16/9'-16') ppm. ^{13}C NMR (100 MHz, CD_3OD) δ 146.6 (C4/4'), 145.7 (C3/3'), 145.3 (C2/2'), 143.4 (C6/6'), 129.0 (C5/5'), 62.9 (C7/7'), 33.6 (C18/18'), 32.6 (C8/8'), 31.6 (C17/17'), 30.7, 30.6, 30.5 (C10-15/10'-15'), 30.1 (C16/16'), 27.1 (C9/9') ppm. IR (NaCl) ν 3020 (m), 2924 (s, C–H), 2853 (s, C–H), 1632 (m), 1505 (s), 1463 (m), 1155 (m), 732 (m), 692 (s). HRMS (ESI$^+$)

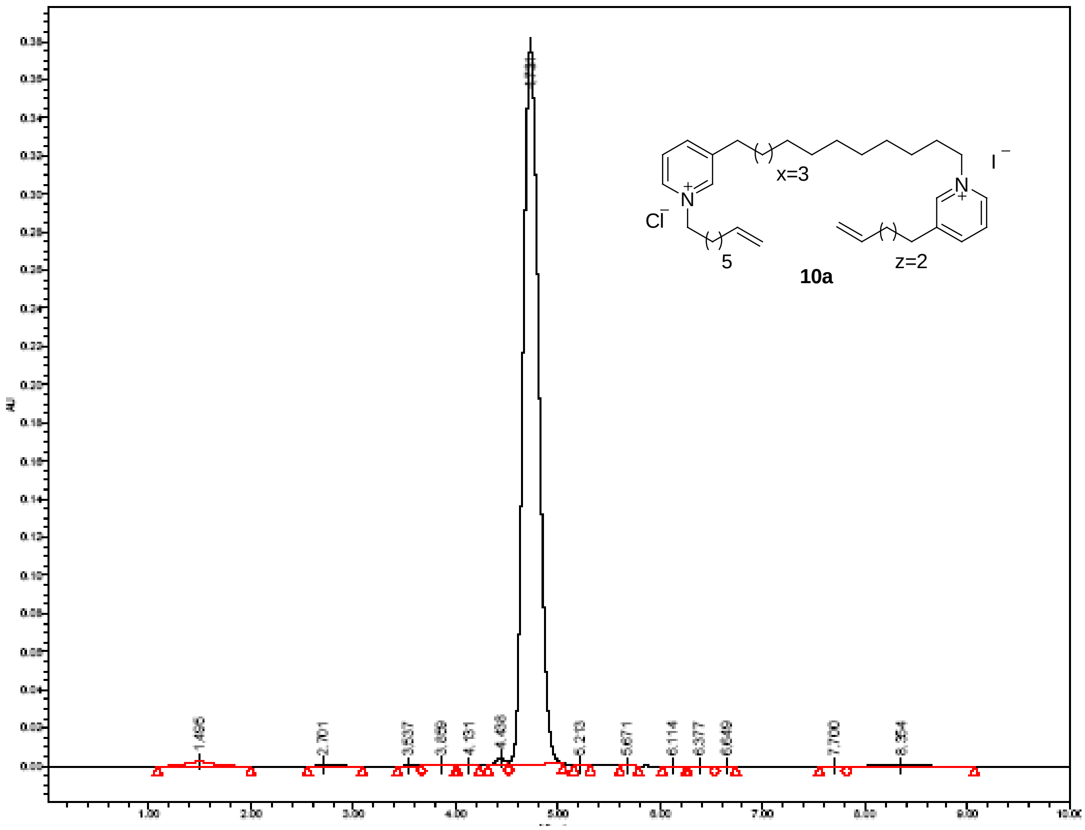
m/z calcd for $C_{34}H_{56}IN_2^+$, 619.3483 (found 619.3475), $C_{34}H_{56}ClN_2^+$ 527.4126 (found 527.4120), $C_{34}H_{56}N_2^{2+}$ 246. 2216 (found 246.2221).

Cyclostellettamine B (1b). ^{1}H NMR (400 MHz, CD_3OD) δ 8.99 (br s, 2H, H2/2'), 8.88 (d, *J* = 5.5 Hz, 2H, H6/6'), 8.48 (d, *J* = 7.8 Hz, 2H, H4/4'), 8.04 (dd, *J* = 7.8, 5.5 Hz 2H, H5/5'), 4.65 (t, *J* = 7.4 Hz, 4H, H7/7'), 2.90 (t, *J* = 7.7 Hz, 4H, H18/19'), 2.1-1.9 (m, 4H, H8/8'), 1.8-1.6 (m, 4H, H17/18'), 1.5-1.2 (m, 34H, H9-16/9'-17') ppm. ^{13}C NMR (100 MHz, CD_3OD) δ 146.6 (C4/4'), 145.7 (C3/3'), 145.3 (C2/2'), 143.4(C6/6'), 129.0 (C5/5'), 62.9 (C7/7'), 33.5 (C18/19'), 32.6 (C8/8'), 31.6 (C17/18'), 30.7, 30.6, 30.5 (C10-15/10'-16'), 30.2 (C16/17'), 27.2 (C9/9') ppm. IR (NaCl) ν 3020 (m), 2924 (s, C–H), 2853 (s, C–H), 1632 (m), 1505 (s), 1464 (m), 1155 (m), 691 (s). HRMS (ESI$^+$) *m/z* calcd for $C_{35}H_{58}IN_2^+$ 633.3639 (found 633.3632), $C_{35}H_{58}ClN_2^+$ 541.4283 (found 541.4275), $C_{35}H_{58}N_2^{2+}$ 253.2294 (found 253.2295).

Cyclostellettamine E (1e). ^{1}H NMR (400 MHz, CD_3OD) δ 8.96 (br s, 2H, H2/2'), 8.86 (d, *J* = 5.5 Hz, 2H, H6/6'), 8.46 (d, *J* = 7.8 Hz, 2H, H4/4'), 8.0-7.9 (m, 2H, H5/5'), 4.62 (t, *J* = 7.5 Hz, 4H, H7/7'), 2.89 (t, *J* = 7.8 Hz, 4H, H19/20'), 2.0-1.9 (m, 4H, H8/8'), 1.74 (t, *J* = 7.2 Hz, 4H, H18/19'), 1.5-1.2 (m, 36H, H9-17/9'-18') ppm. ^{13}C NMR (100 MHz, CD_3OD) δ 146.7 (C4/4'), 145.8 (C3/3'), 145.3 (C2/2'), 143.4 (C6/6'), 129.0 (C5/5'), 62.9 (C7/7'), 33.6 (C19/20'), 32.6 (C8/8'), 31.7 (C18/19'), 30.7, 30.6, 30.5, 30.3 (C10-16/10'-17'), 30.2 (C17/18'), 27.2 (C9/9') ppm. IR (NaCl) ν 3021 (m), 2925 (s, C–H), 2853 (s, C–H), 1632 (m), 1505 (s), 1456 (s), 1155 (m), 1035 (m), 692 (s). HRMS (ESI$^+$) *m/z* calcd for $C_{37}H_{62}IN_2^+$ 661.3952 (found 661.3949), $C_{37}H_{62}ClN_2^+$ 569.4596 (found 569.4590), $C_{37}H_{62}N_2^{2+}$ 267.2451 (found 267.2452).

Cyclostellettamine G (1g). ^{1}H NMR (400 MHz, CD_3OD) δ 8.96 (br s, 2H, H2/2'), 8.85 (d, *J* = 6.2 Hz, 2H, H6/6'), 8.47 (d, *J* = 8.1 Hz, 2H, H4/4'), 8.02 (dd, *J*= 8.1, 6.2 Hz, 2H, H5/5'), 4.62 (t, *J* = 7.3 Hz, 4H, H7/7'), 2.9-2.8 (m, 4H, H17/18'), 2.1-1.9 (m, 4H, H8/8'), 1.73 (t, *J*= 7.2 Hz, 4H, H16/17'), 1.3-1.2 (m, 30H, H9-14/9'-15') ppm. ^{13}C NMR (100 MHz, CD_3OD) δ 146.7 (C4/4'), 145.8 (C3/3'), 145.3 (C2/2'), 143.4 (C6/6'), 129.0 (C5/5'), 62.9 (C7/7'), 33.6 (C17/18'), 32.6 (C8/8'), 31.6 (C16/17'), 30.7, 3.06, 30.5, 30.4 (C10-14/C10'-15'), 30.2 (C15/16'), 27.2 (C9/9') ppm. IR (NaCl) ν 3022 (m), 2922 (s, C–H), 2852 (s, C–H), 1632 (m), 1505 (s), 1464 (m), 1156 (m), 691 (s). HRMS (ESI^+) *m/z* calcd for $C_{33}H_{54}IN_2^+$, 605.3326 (found 605.3317), $C_{33}H_{54}ClN_2^+$ 513.3970 (found 513.3964), $C_{33}H_{54}N_2^{2+}$ 239.2138 (found 239.2140).

Cyclostellettamine analogue (12). ^1H NMR (400 MHz, CD_3OD) δ 8.97 (br s, 2H, H2/2'), 8.85 (d, *J* = 5.8 Hz, 2H, H6/6'), 8.46 (d, *J* = 8.0 Hz, 2H, H4/4'), 8.02 (dd, *J* = 8.0, 5.8 Hz, 2H, H5/5'), 4.62 (t, *J* = 7.5 Hz, 4H, H7/7'), 2.9-2.8 (m, 4H, H19/17'), 2.0-1.9 (m, 4H, H8/8'), 1.74 (t, *J* = 7.0 Hz, 4H, H18/16'), 1.4-1.2 (m, 36H, H9-17/9'-15') ppm. ^{13}C NMR (100 MHz, CD_3OD) δ 146.7 (C4/4'), 145.8 (C3/3'), 145.3 (C2/2'), 143.4 (C6/6'), 129.0 (C5/5'), 62.9 (C7/7'), 33.6 (C19/17'), 32.6 (C8/8'), 31.7 (C18/16'), 30.8, 30.7, 30. 6, 30.5, 30.4, 30.3, 30.2 (C10-16/10'-14'), 30.1 (C17/15'), 27.2 (C9/9') ppm. IR (NaCl) ν 3021 (m), 2923 (s, C–H), 2852 (s, C–H), 1632 (m), 1505 (s), 1464 (m), 1155 (m), 692 (s). HRMS (ESI^+) *m/z* calcd for $C_{34}H_{56}IN_2^+$, 619.3483 (found 619.3474), $C_{34}H_{56}ClN_2^+$ 527.4126 (found 527.4121), $C_{34}H_{56}N_2^{2+}$ 246. 2216 (found 246.2219).



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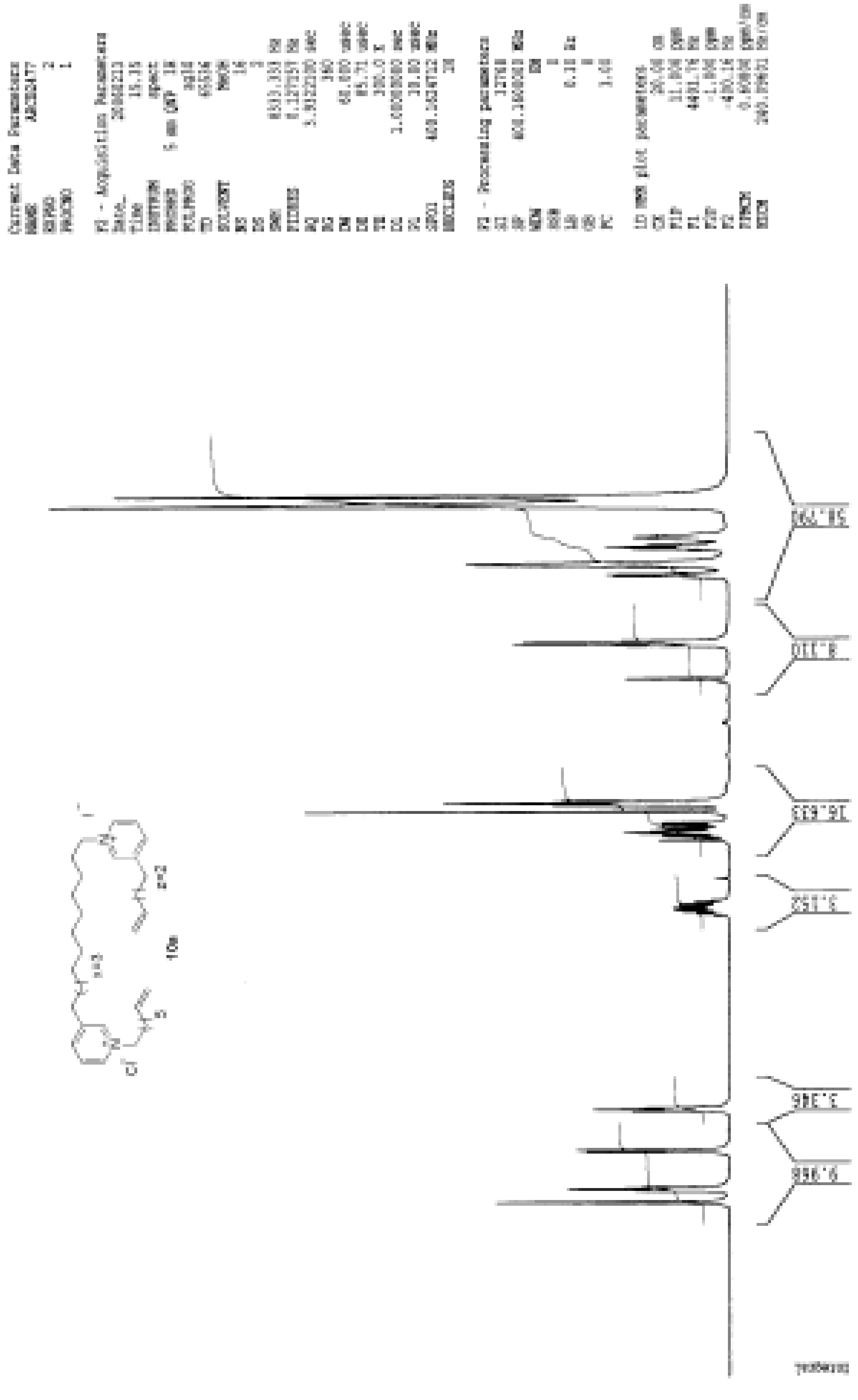
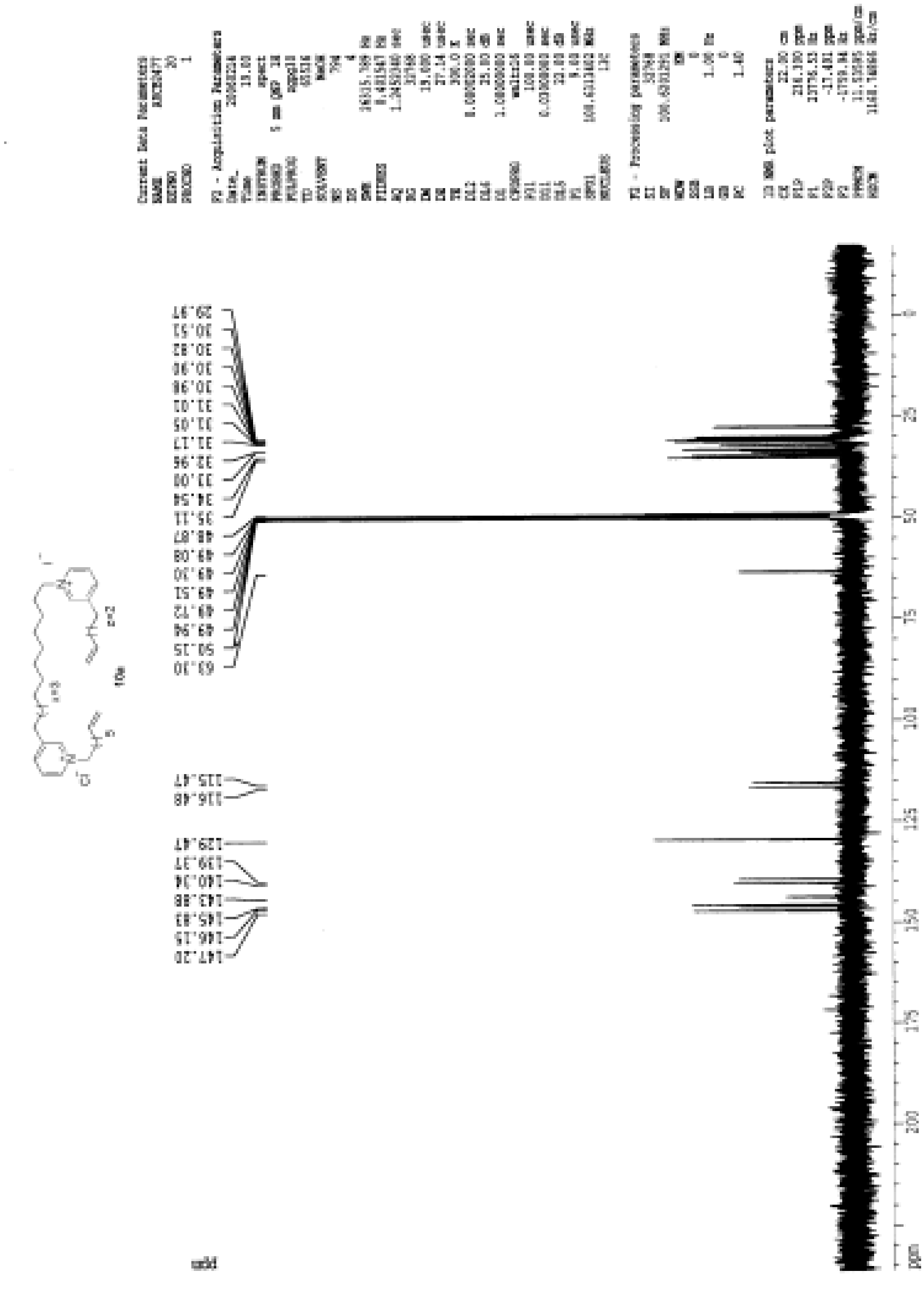
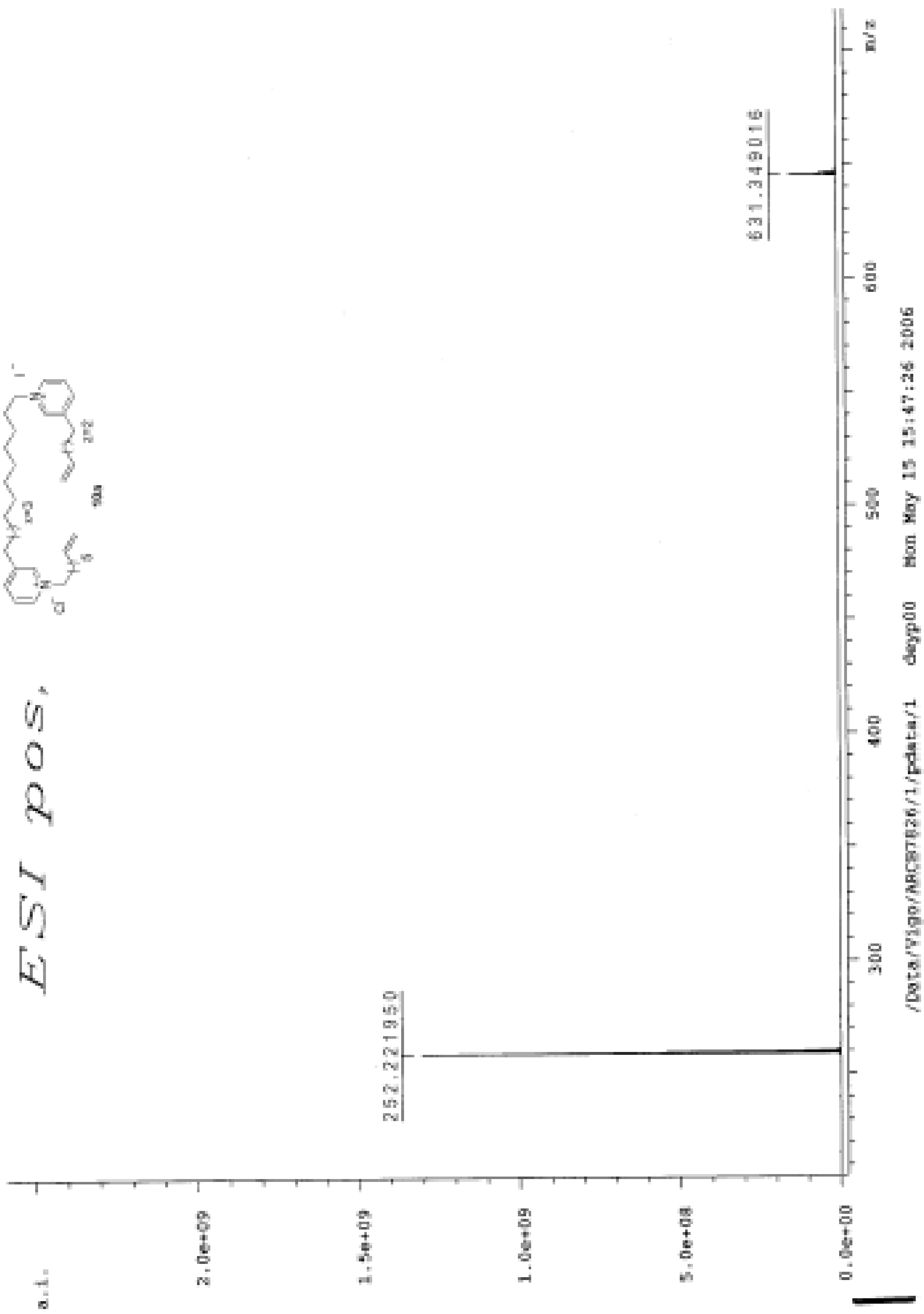
4		3.859	5585	0.14	528	VB			Unknown	
5		4.131	868	0.02	157	BB			Unknown	
6		4.438	21834	0.54	3427	BV			Unknown	
7		4.731	3974704	97.78	377434	VB			Unknown	
8		5.213	624	0.02	130	BB			Unknown	
9		5.671	143	0.00	26	BB			Unknown	
10		6.114	210	0.01	28	BB			Unknown	
11		6.377	183	0.00	21	BV			Unknown	
12		6.649	247	0.01	39	VB			Unknown	
13		7.700	678	0.02	66	BV			Unknown	
14		8.354	10499	0.26	337	VB			Unknown	

Develosil C-30
H_20/CH_3CN:17/83 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm







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m/z
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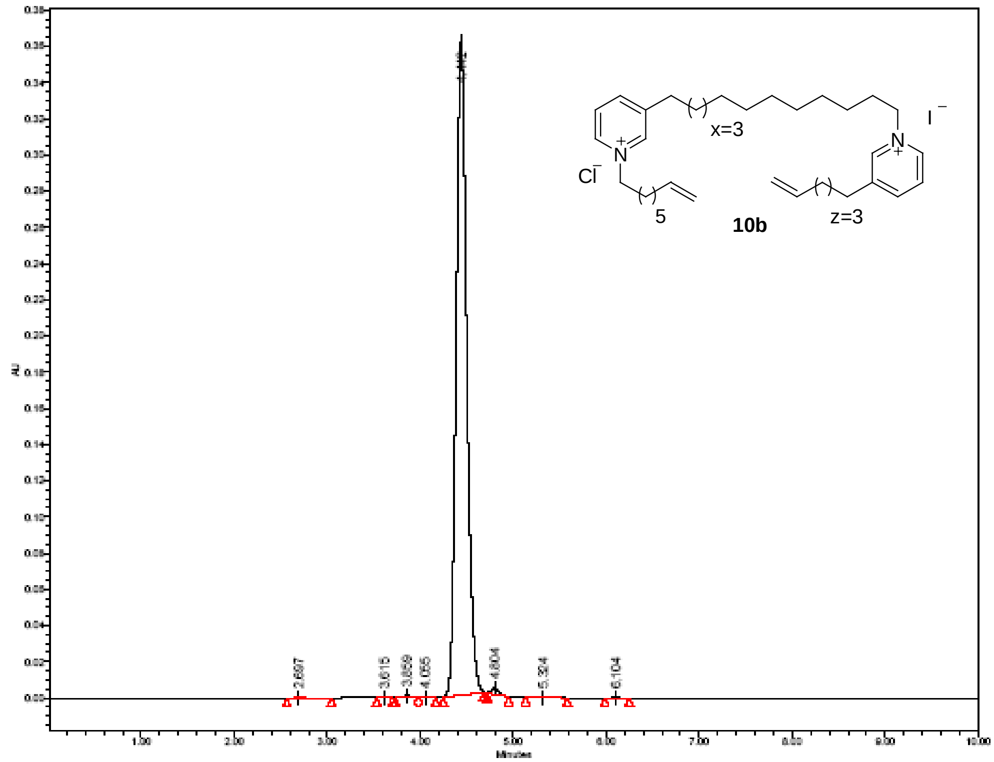

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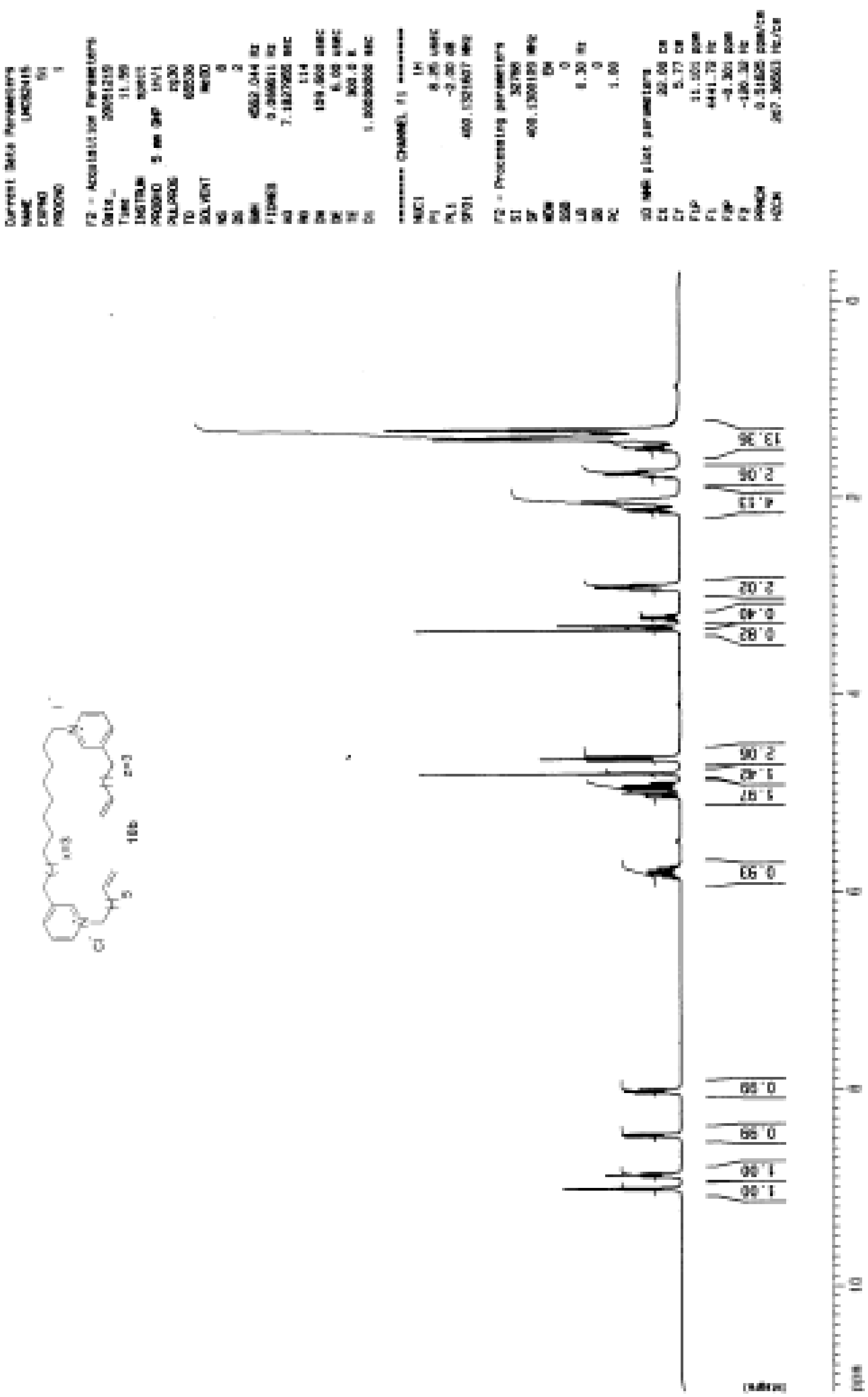
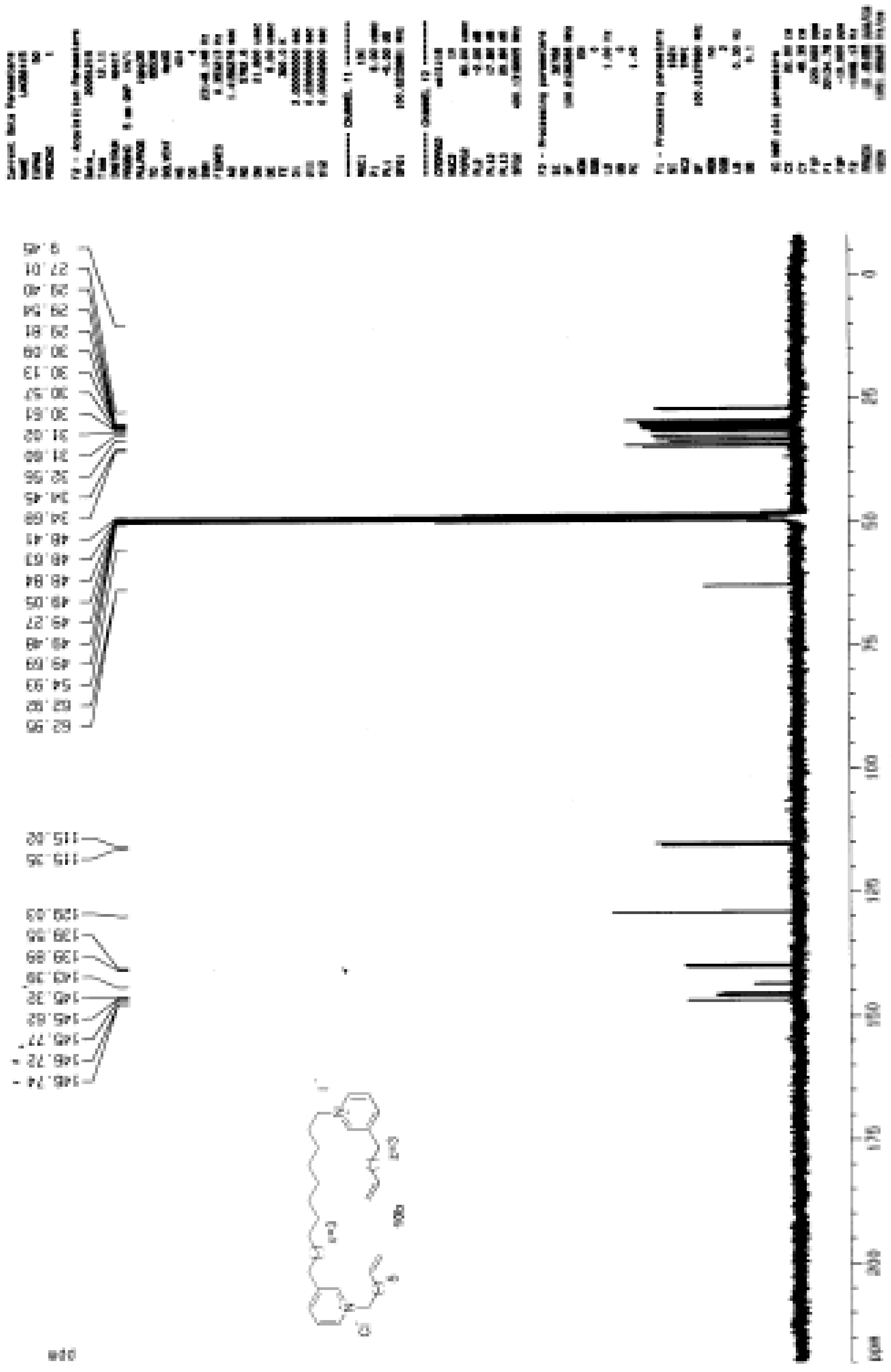
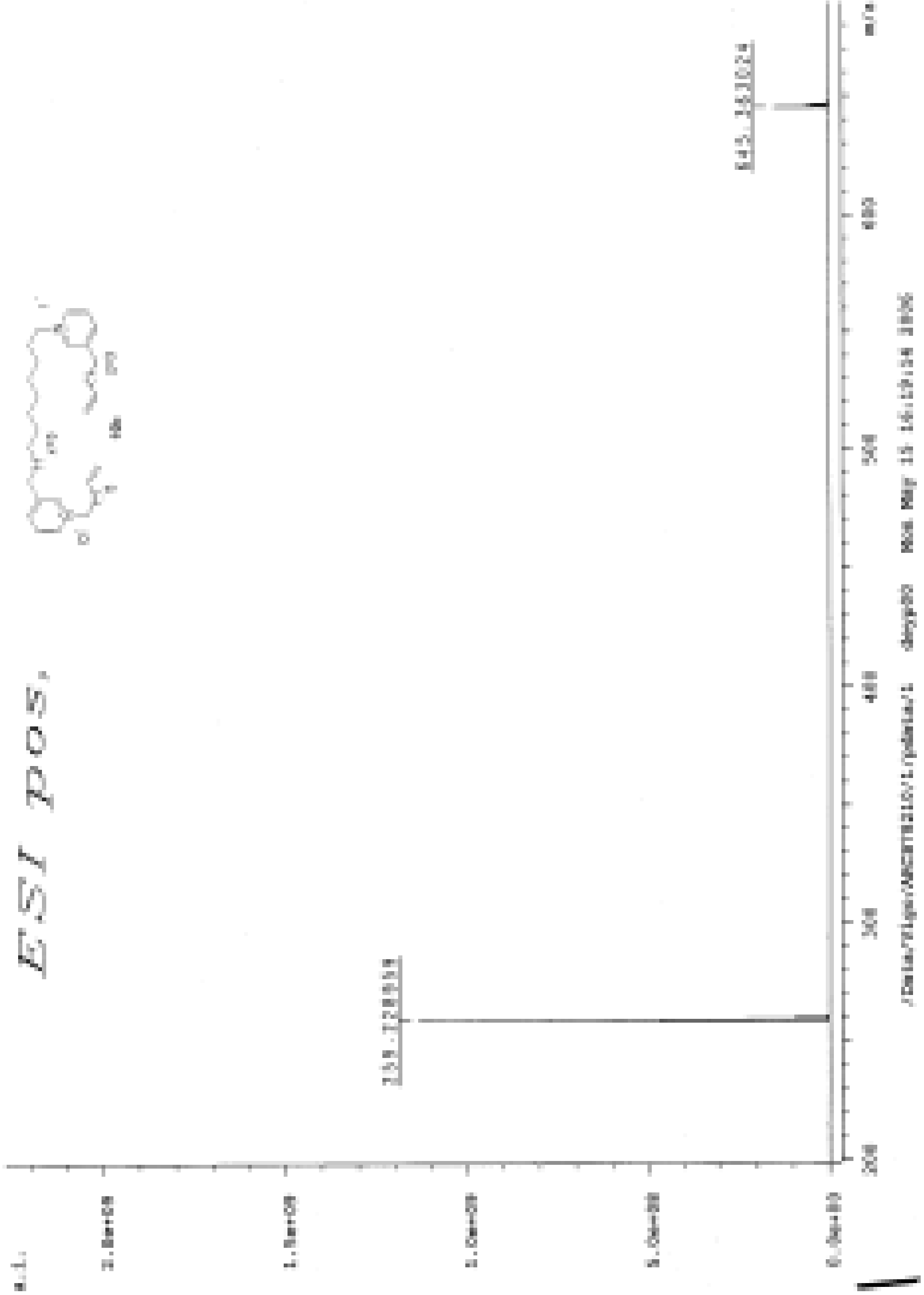
3		3.859	7526	0.26	1067	BV			Unknown	
4		4.055	1347	0.05	190	VB			Unknown	
5		4.442	2863353	98.81	362979	BB			Unknown	
6		4.804	19824	0.68	3412	BB			Unknown	
7		5.324	3498	0.12	325	BB			Unknown	
8		6.104	752	0.03	97	BB			Unknown	

Develosil C-30
H_20/CH_3CN:17/83 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm



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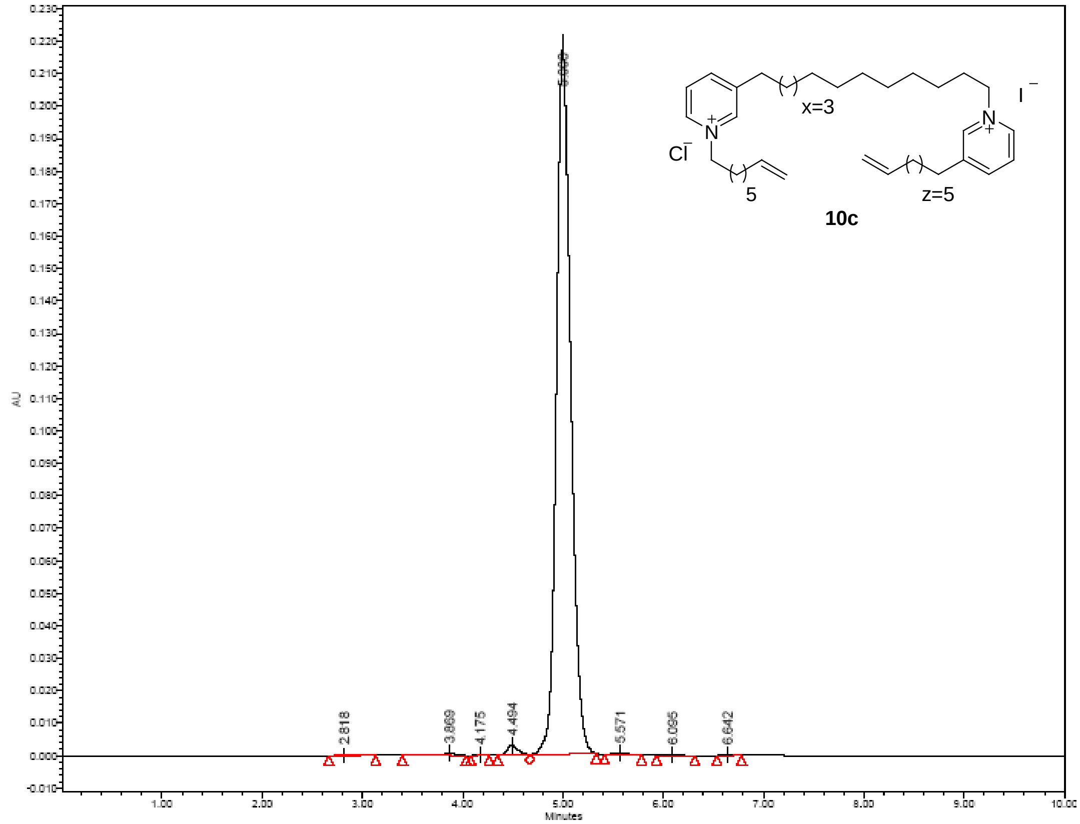

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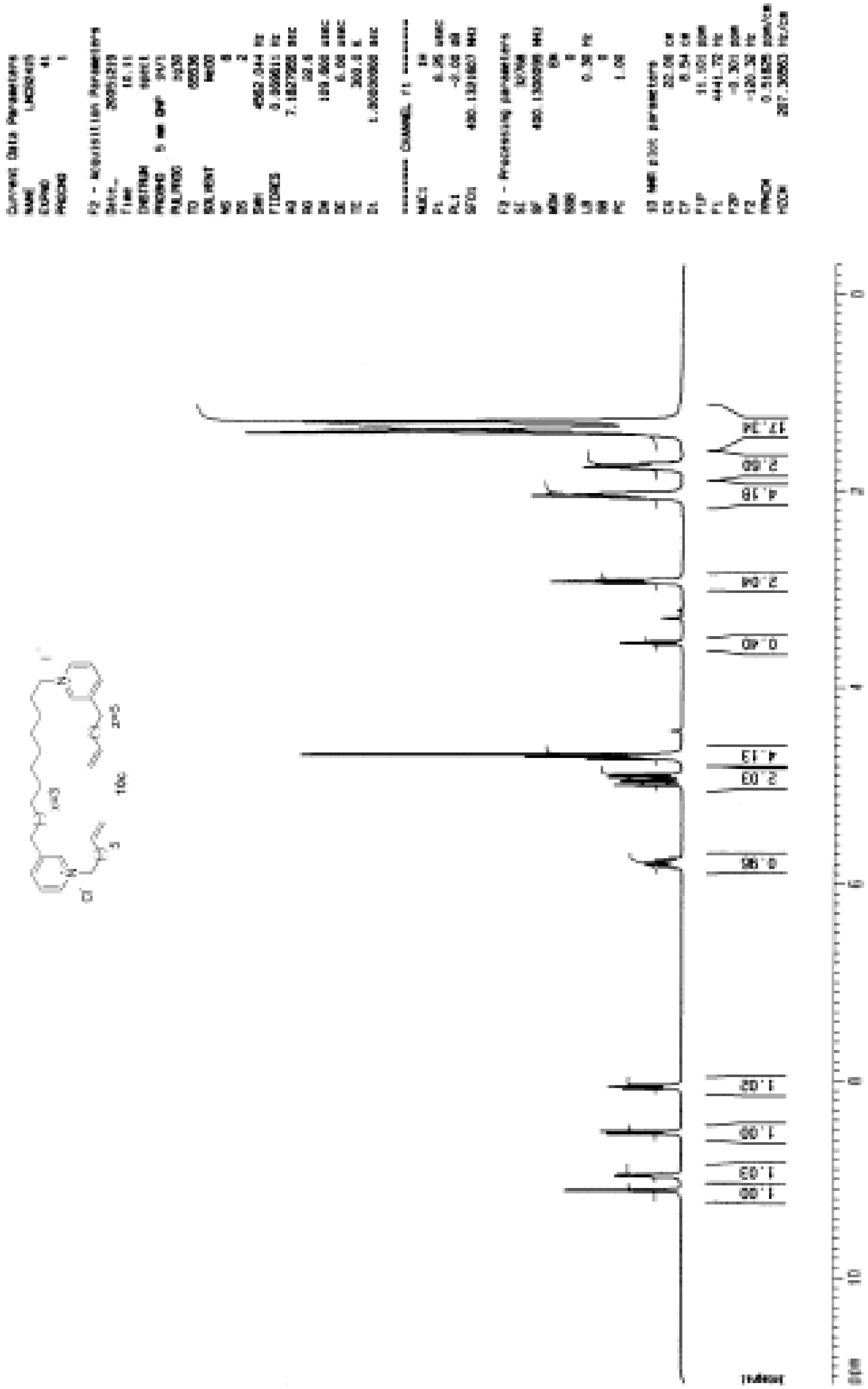
4		4.494	23084	1.05	3021	BV			Unknown	
5		5.000	2158065	98.27	218843	VB			Unknown	
6		5.571	4686	0.21	405	BB			Unknown	
7		6.095	1321	0.06	159	BB			Unknown	
8		6.642	928	0.04	131	BB			Unknown	

Develosil C-30
H_20/CH_3CN:17/83 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm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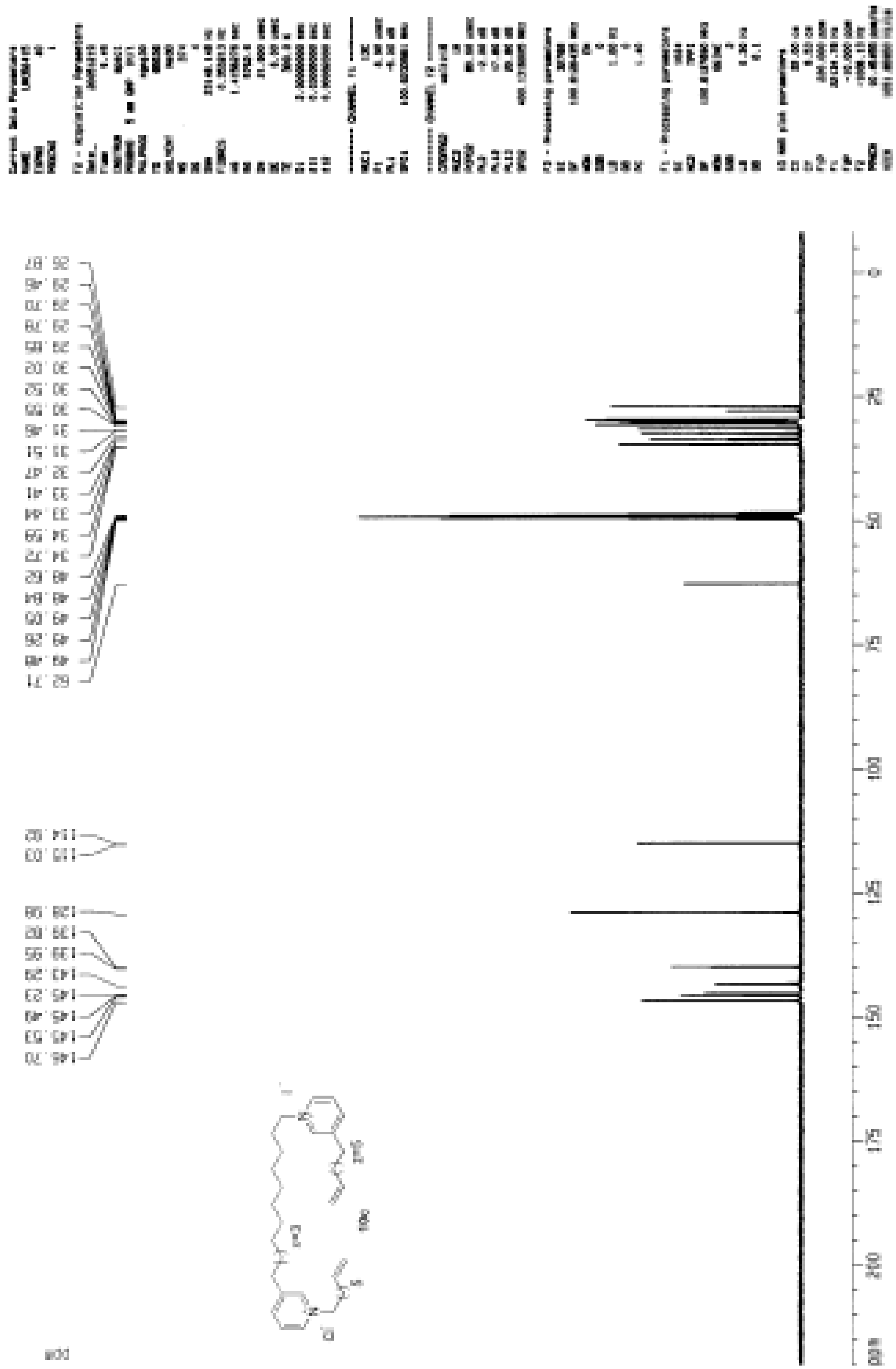

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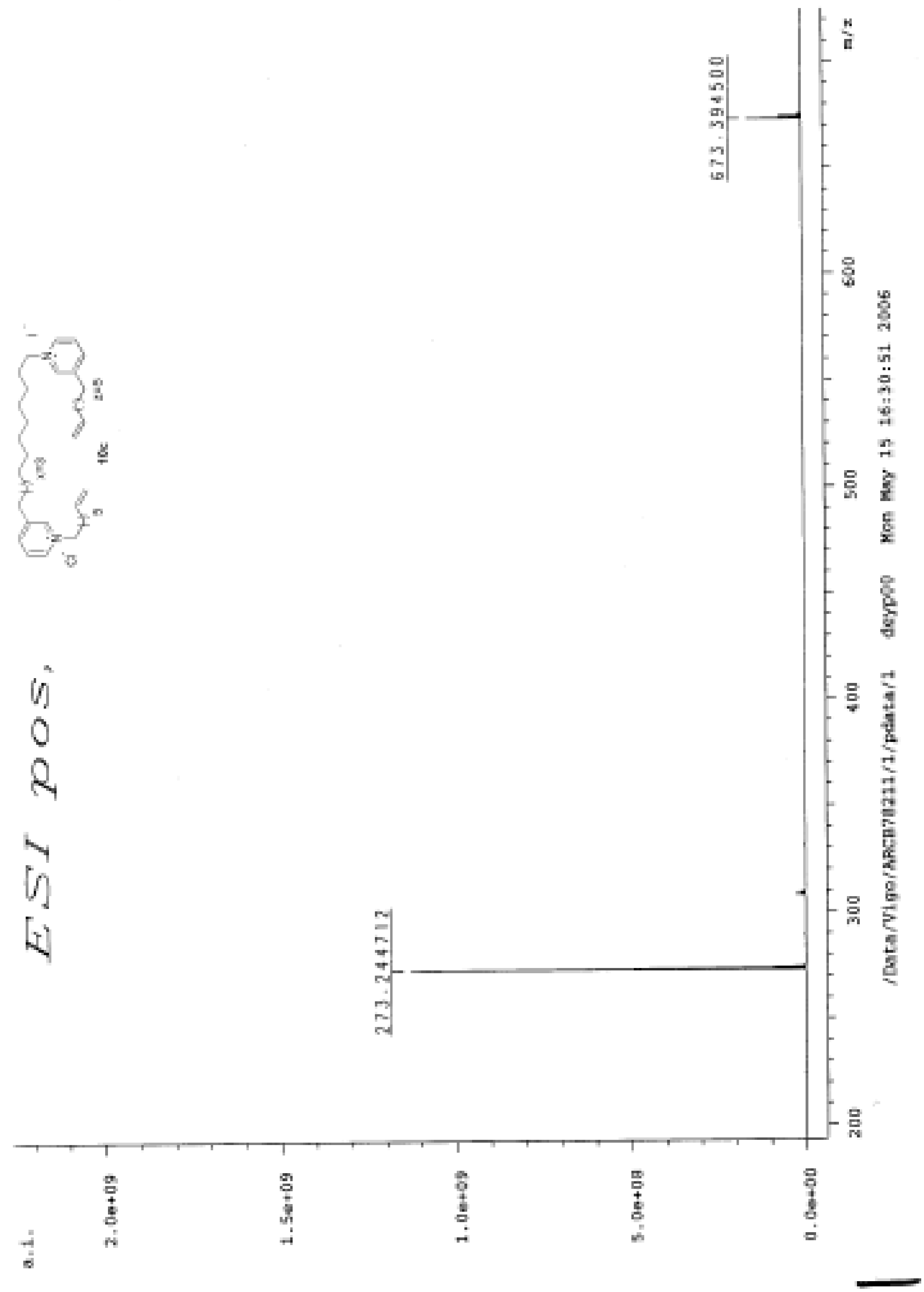
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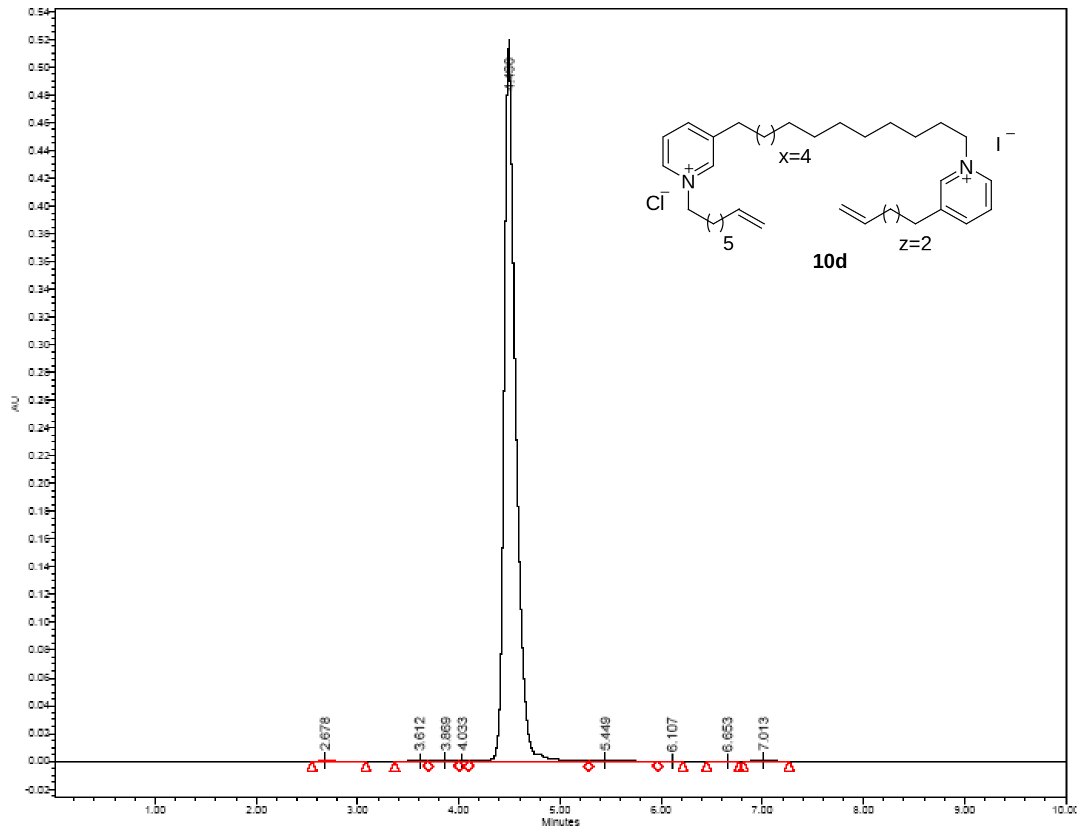

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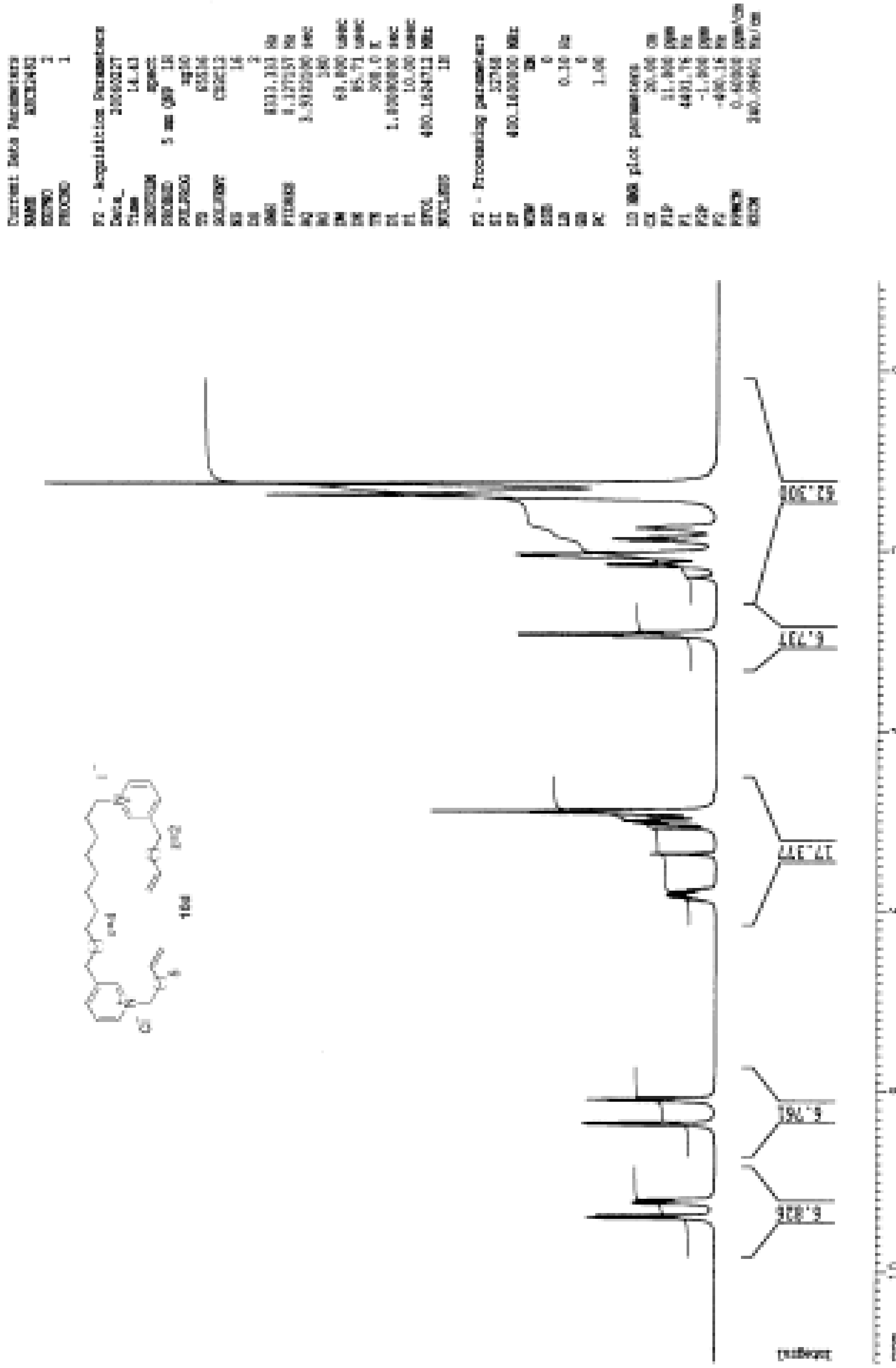
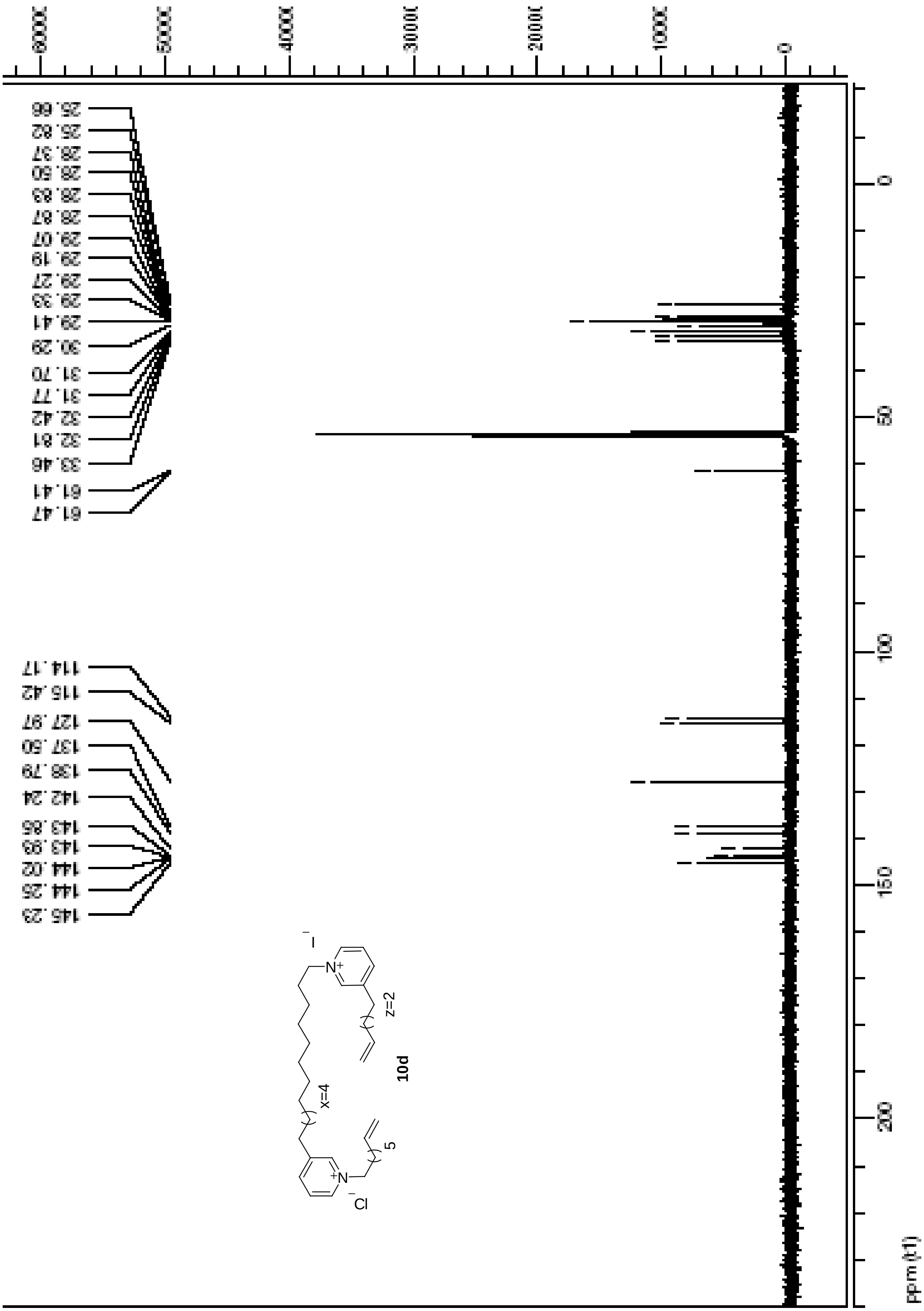
3		3.869	6528	0.16	810	VV			Unknown	
4		4.033	685	0.02	129	VV			Unknown	I19
5		4.496	4138283	99.34	515653	VV			Unknown	
6		5.449	7733	0.19	441	VV			Unknown	
7		6.107	425	0.01	52	VB			Unknown	
8		6.653	563	0.01	80	BB			Unknown	
9		7.013	6867	0.16	726	BB			Unknown	

Develosil C-30
H_20/CH_3CN:17/83 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm





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25.82
28.37
28.50
28.63
28.67
29.07
29.19
29.27
29.33
29.41
30.29
31.70
31.77
32.42
32.81
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61.41
61.47
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115.42
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137.50
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144.02
144.25
145.23
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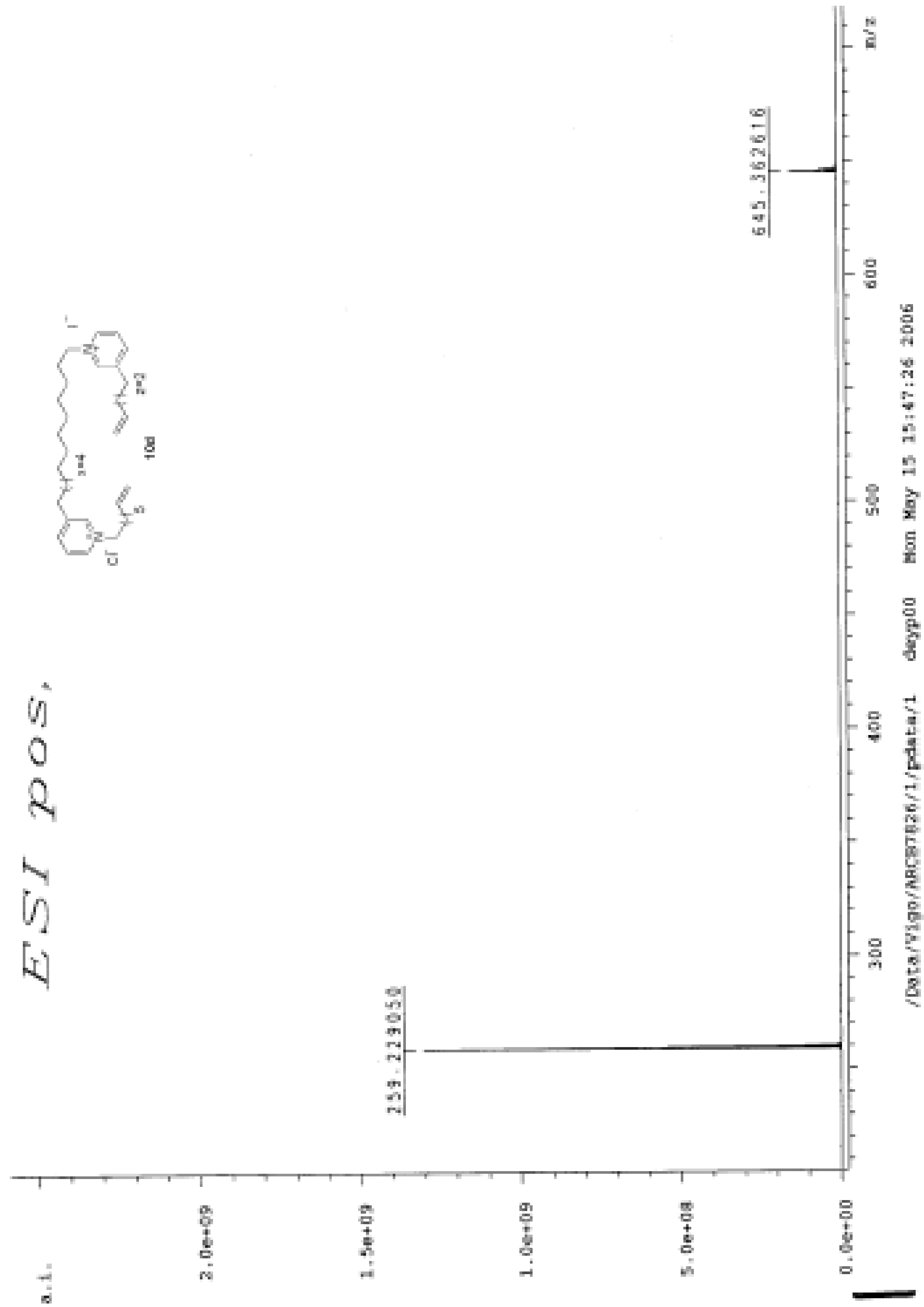
z=2
5
10d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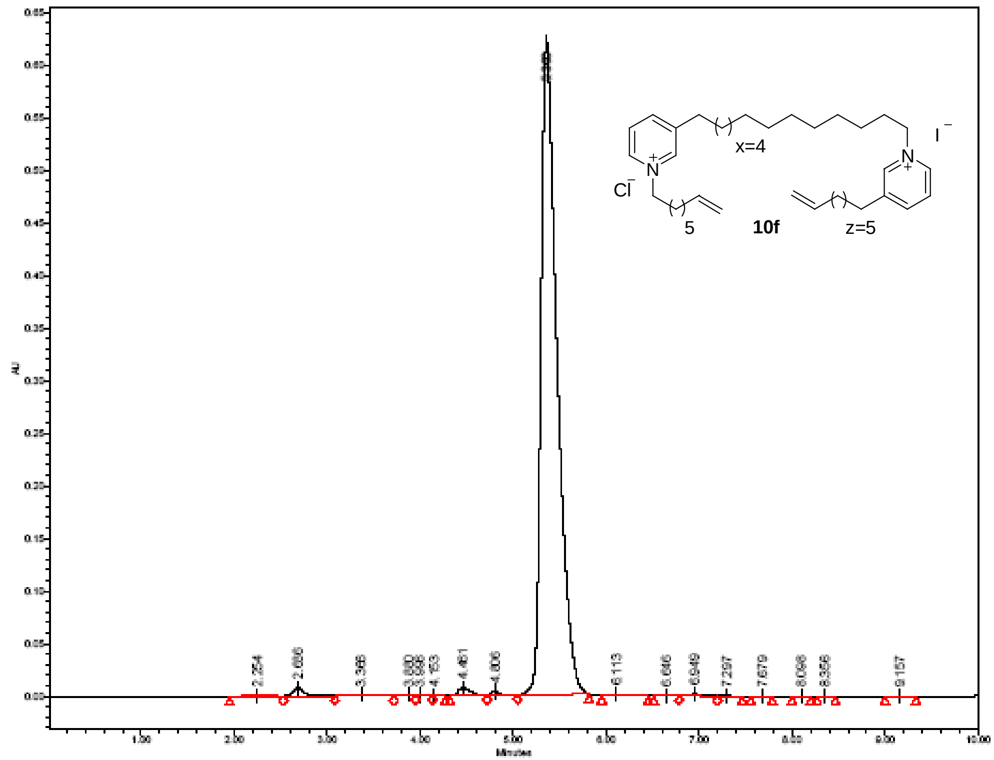
m/z



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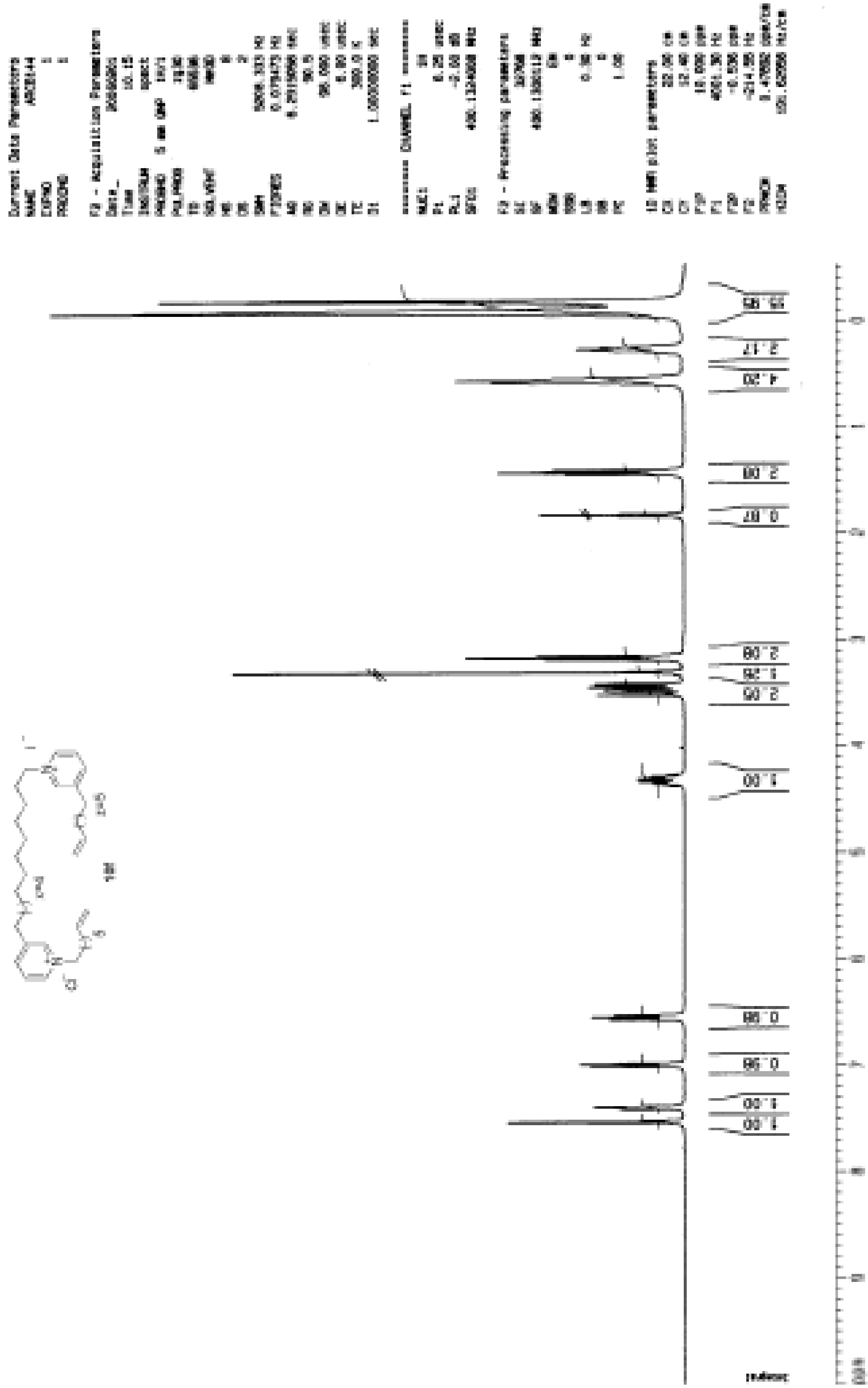
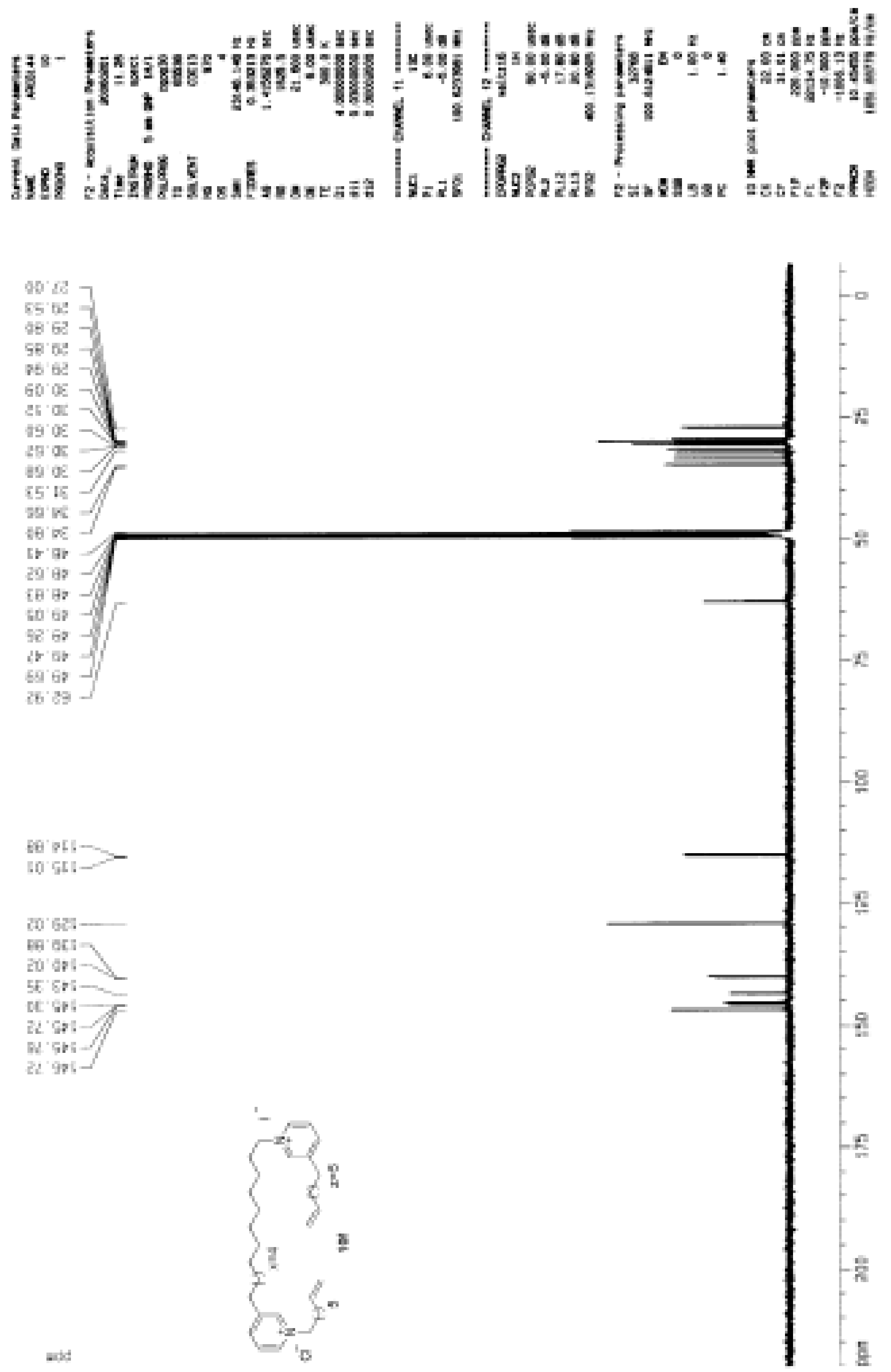
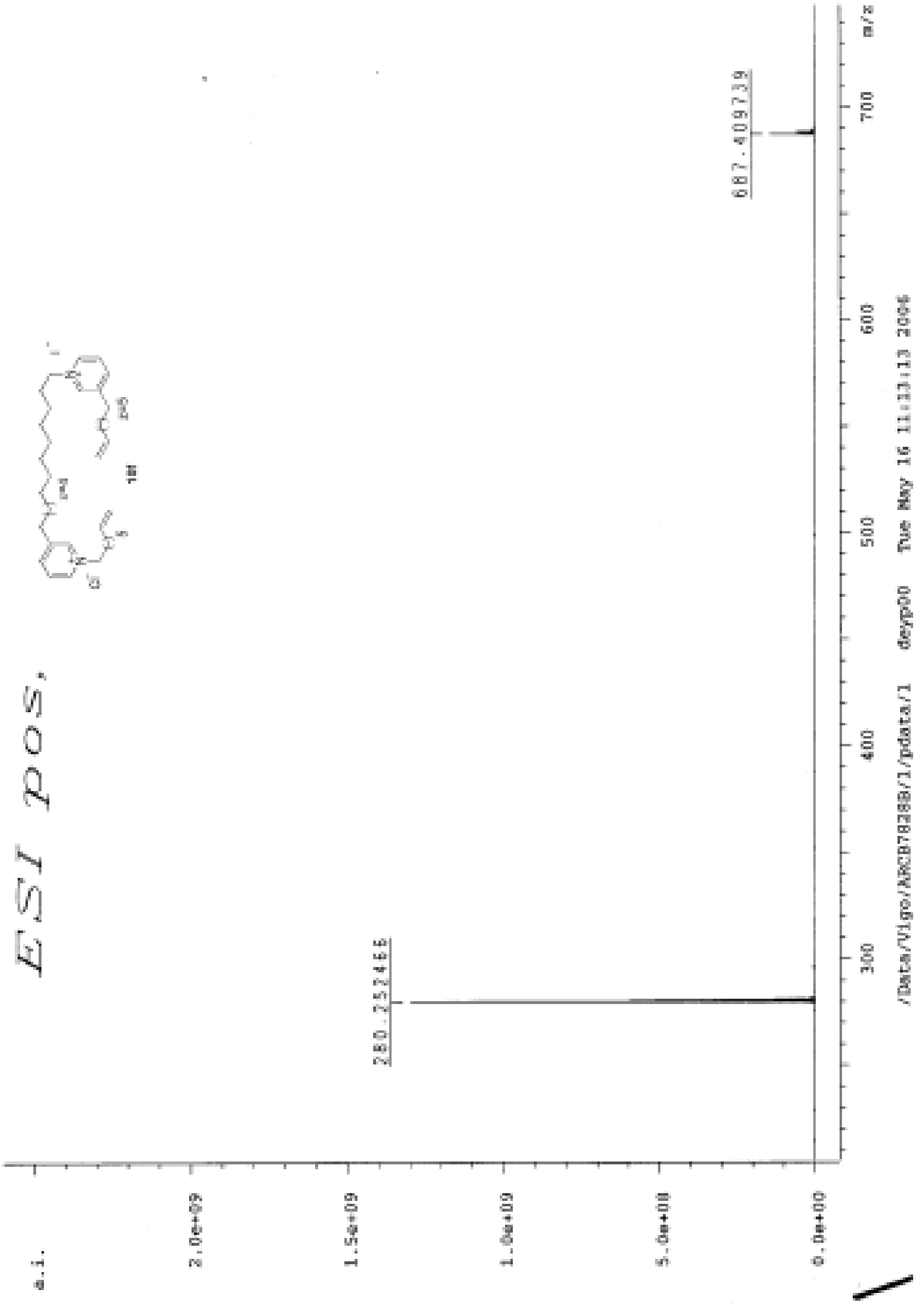
1		2.254	10347	0.13	711	BV			Unknown	
2		2.686	65421	0.85	7619	VV			Unknown	
3		3.366	26442	0.34	1069	VV			Unknown	
4		3.880	9274	0.12	977	VV			Unknown	
5		3.996	5414	0.07	873	VV			Unknown	
6		4.153	495	0.01	91	VB			Unknown	
7		4.461	69571	0.90	7078	BV			Unknown	
8		4.806	26058	0.34	3625	VV			Unknown	
9		5.360	7488265	96.78	628273	VB			Unknown	
10		6.113	12209	0.16	880	BB			Unknown	
11		6.646	4336	0.06	586	BV			Unknown	
12		6.949	17785	0.23	1901	VV			Unknown	
13		7.297	417	0.01	54	VB			Unknown	
14		7.679	288	0.00	41	BB			Unknown	
15		8.098	196	0.00	27	BB			Unknown	
16		8.356	181	0.00	25	BB			Unknown	
17		9.157	369	0.00	37	BB			Unknown	

Develosil C-30
H_20/CH_3CN:17/83 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm



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m/z
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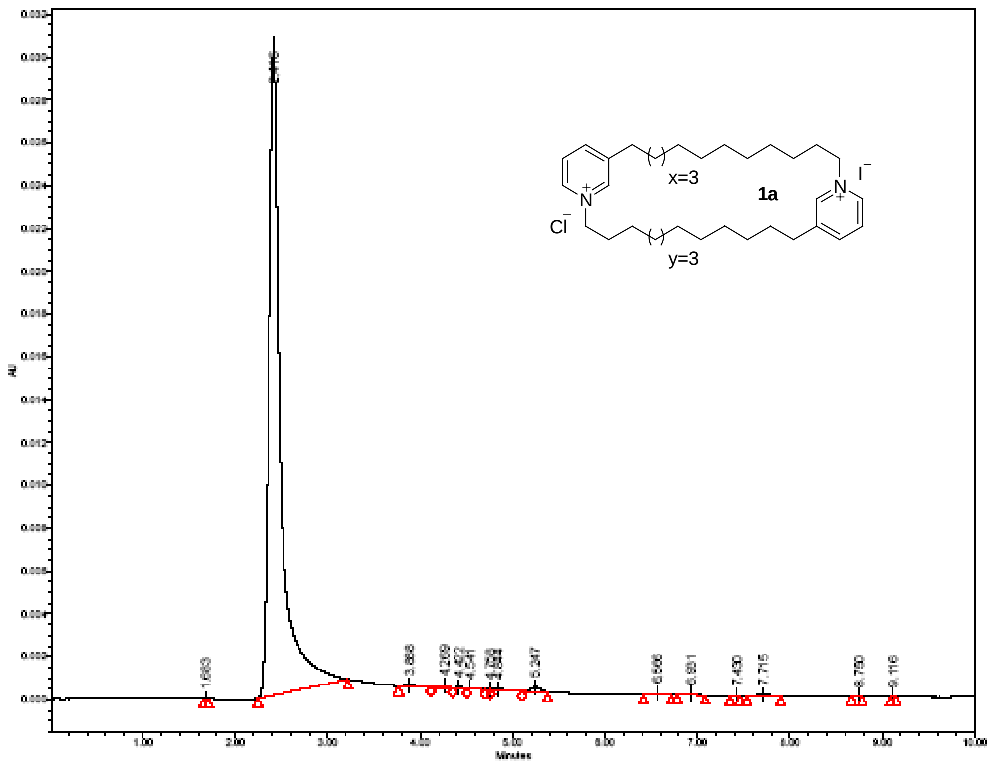
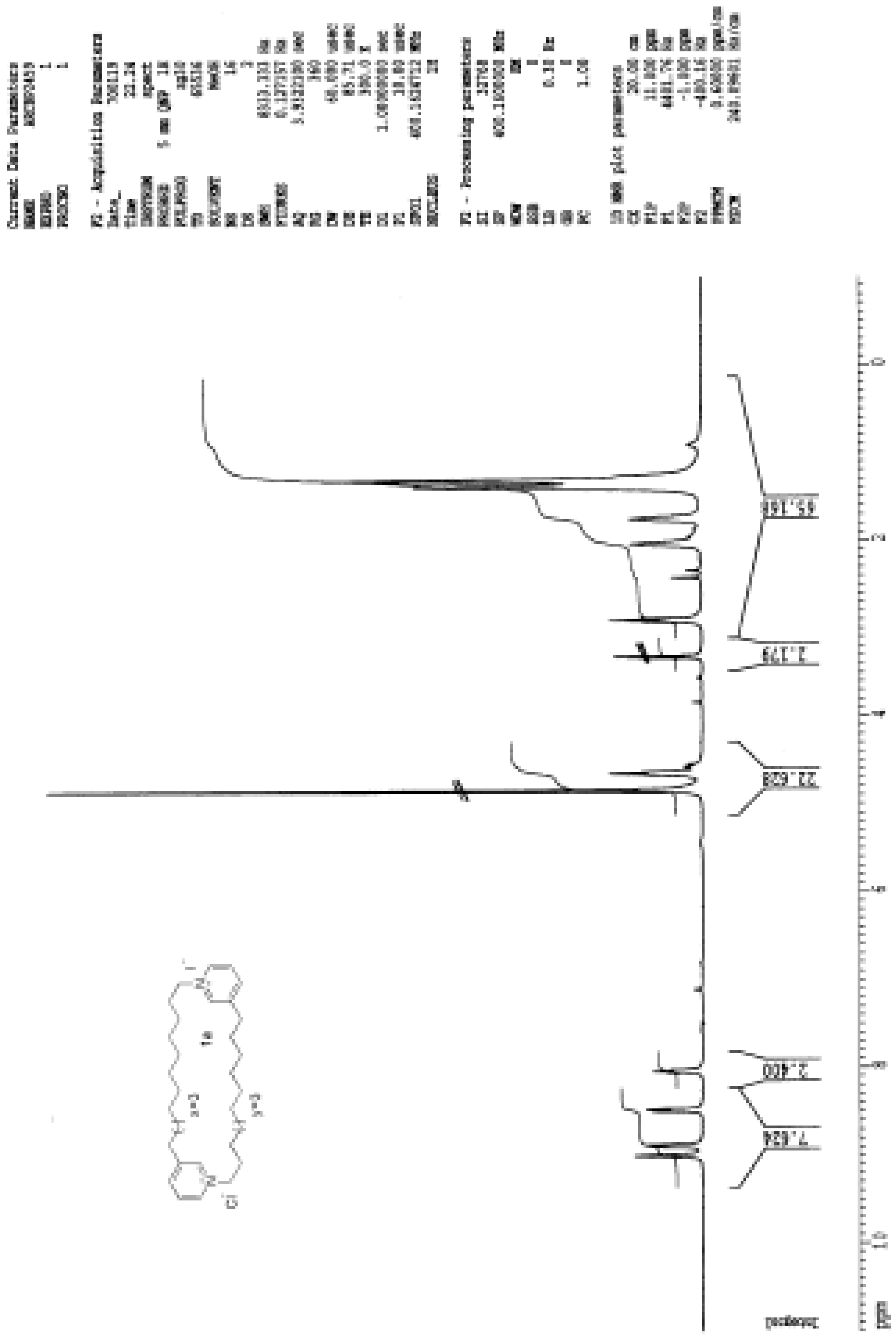


	Name	Retention Time (min)	Area (µV*sec)	% Area	Height (µV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		1.683	17	0.01	8	BB			Unknown	I19
2		2.416	270483	97.89	30294	BB			Unknown	
3		3.888	670	0.24	70	BV			Unknown	
4		4.269	702	0.25	96	VV			Unknown	
5		4.422	382	0.14	66	VV			Unknown	
6		4.541	277	0.10	28	VV			Unknown	I19
7		4.758	113	0.04	38	VV			Unknown	I19 I38
8		4.844	690	0.25	54	VV			Unknown	
9		5.247	1611	0.58	223	VB			Unknown	
10		6.566	233	0.08	28	BB			Unknown	
11		6.931	296	0.11	29	BB			Unknown	
12		7.430	30	0.01	9	BB			Unknown	I19
13		7.715	759	0.27	74	BB			Unknown	
14		8.750	21	0.01	8	BB			Unknown	I19
15		9.116	15	0.01	8	BB			Unknown	I19

Develosil C-30
$H_2$0/MeOH:7/93 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm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F2 - Acquisition Parameters
F2 - Processing parameters
1D NMR plot parameters
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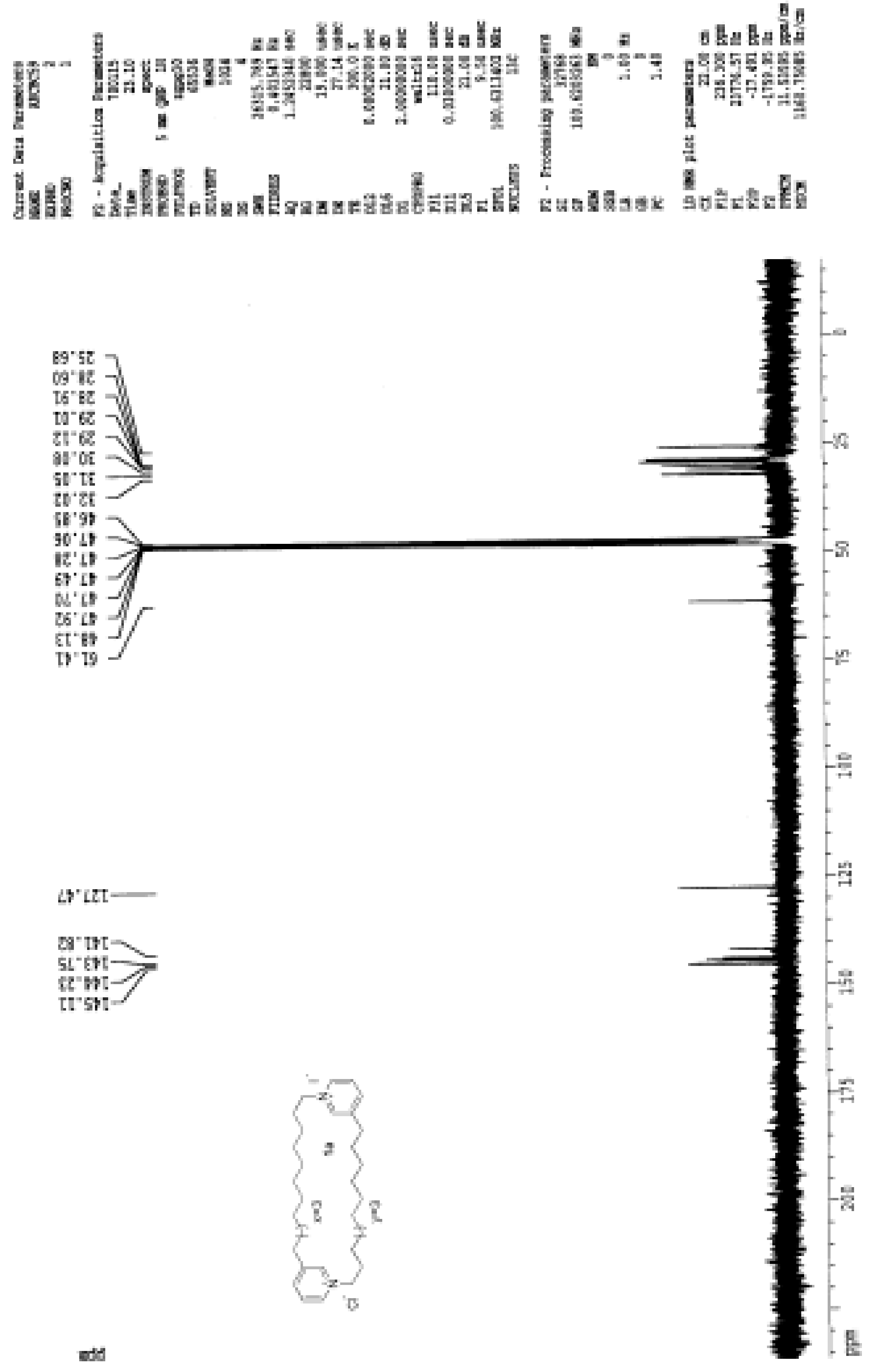
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28.60
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30.08
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32.02
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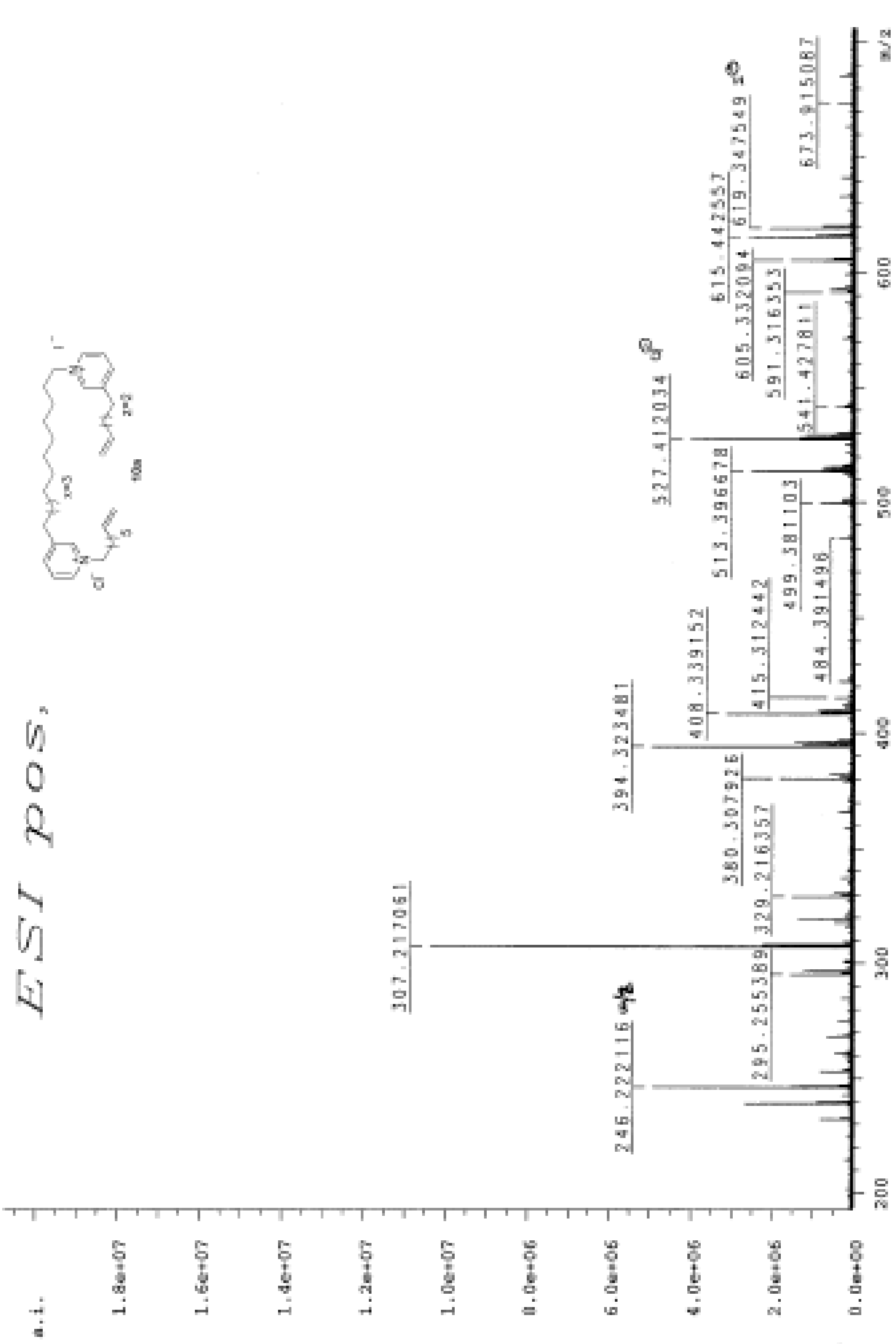

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6.0e+06
4.0e+06
2.0e+06
0.0e+00
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m/z
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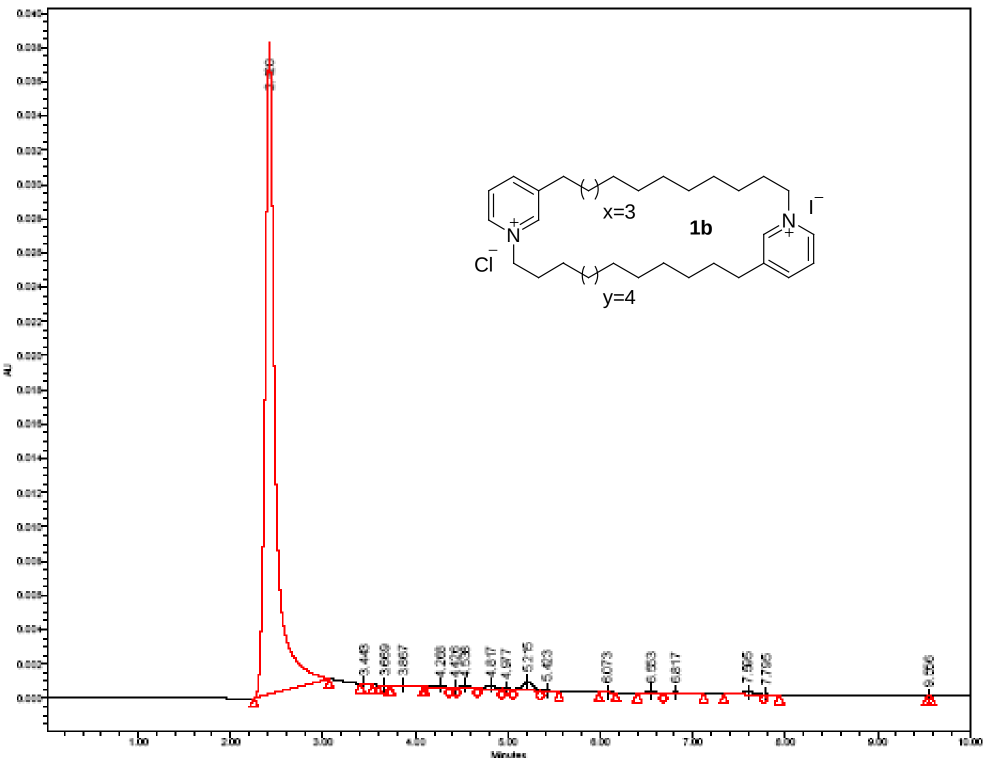
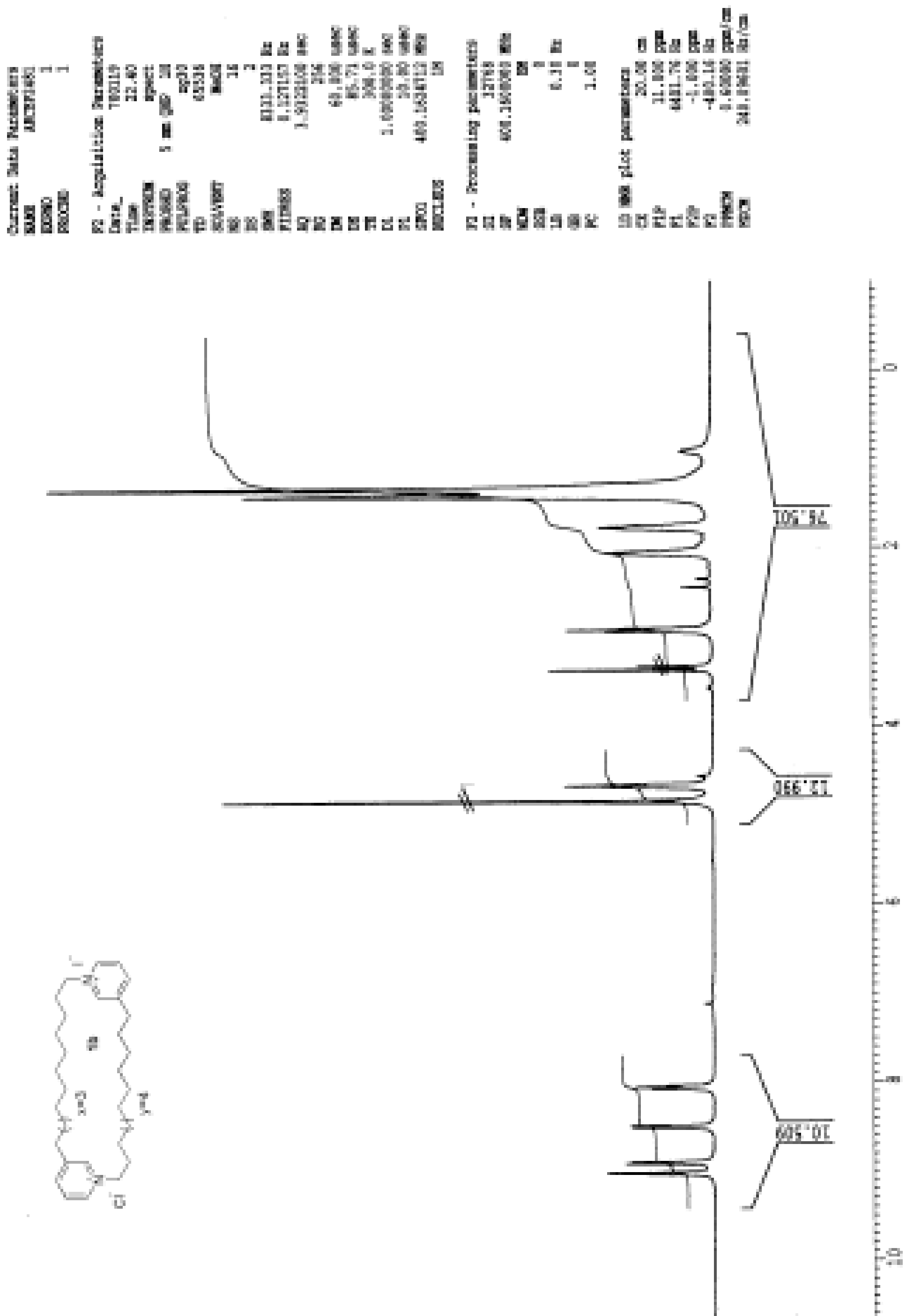
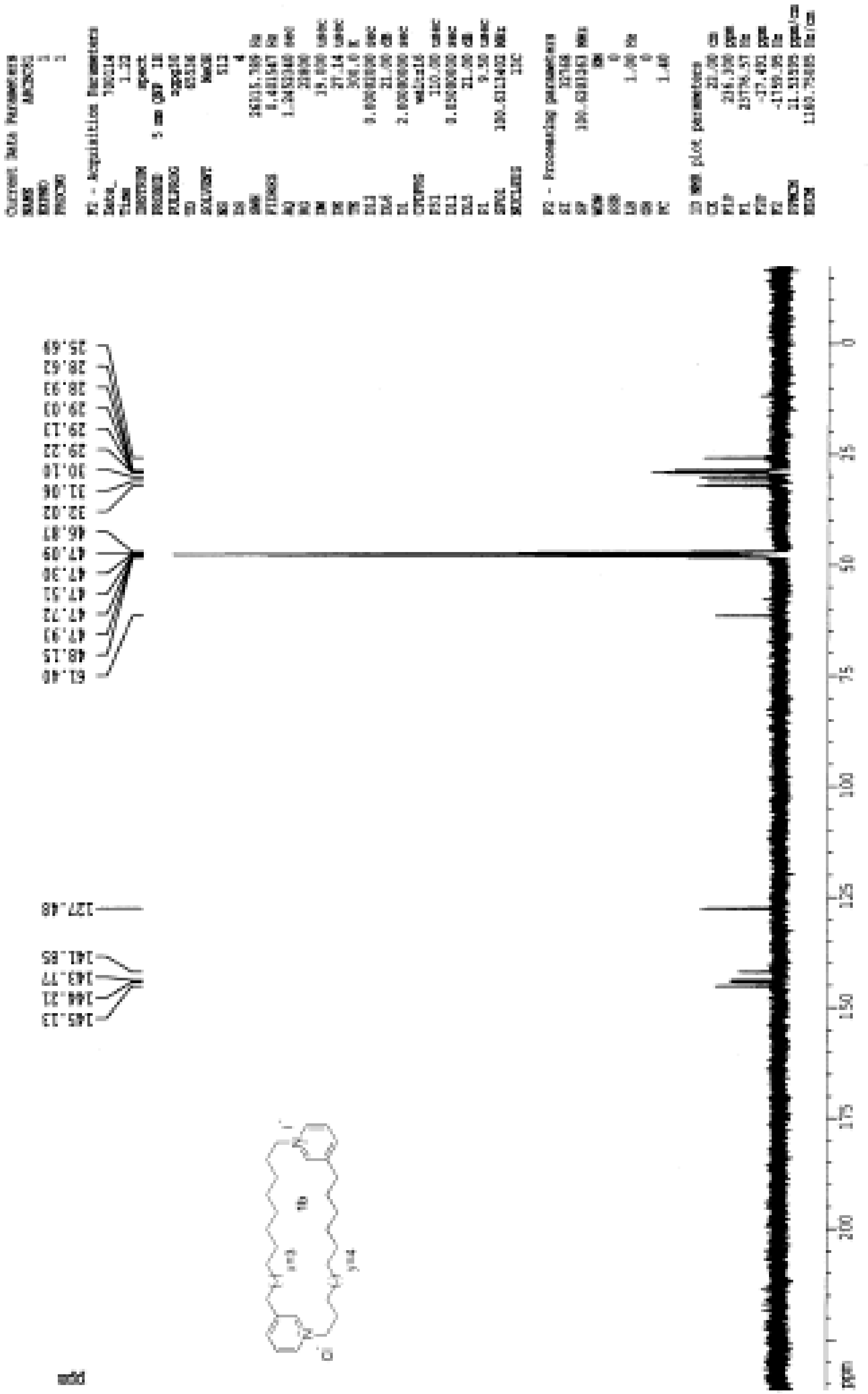


	Name	Retention Time (min)	Area (µV*sec)	% Area	Height (µV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		2.420	293340	95.82	37855	BB			Unknown	
2		3.443	45	0.01	10	BB			Unknown	
3		3.669	49	0.02	14	BB			Unknown	I19
4		3.867	555	0.18	59	BB			Unknown	
5		4.268	720	0.24	96	BV			Unknown	
6		4.426	167	0.05	43	VV			Unknown	I19
7		4.538	1201	0.39	157	VV			Unknown	
8		4.817	1846	0.60	206	VV			Unknown	
9		4.977	474	0.15	83	VV			Unknown	I19
10		5.215	4068	1.33	508	VV			Unknown	
11		5.423	282	0.09	42	VB			Unknown	
12		6.073	82	0.03	14	BB			Unknown	
13		6.553	826	0.27	100	BV			Unknown	
14		6.817	815	0.27	74	VB			Unknown	
15		7.595	1593	0.52	150	BV			Unknown	
16		7.795	70	0.02	17	VB			Unknown	I19
17		9.556	8	0.00	5	BB			Unknown	I19

Develosil C-30
$H_2$0/MeOH:7/93 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm

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29.03
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29.22
30.10
31.06
32.02
46.87
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47.93
48.15
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141.85
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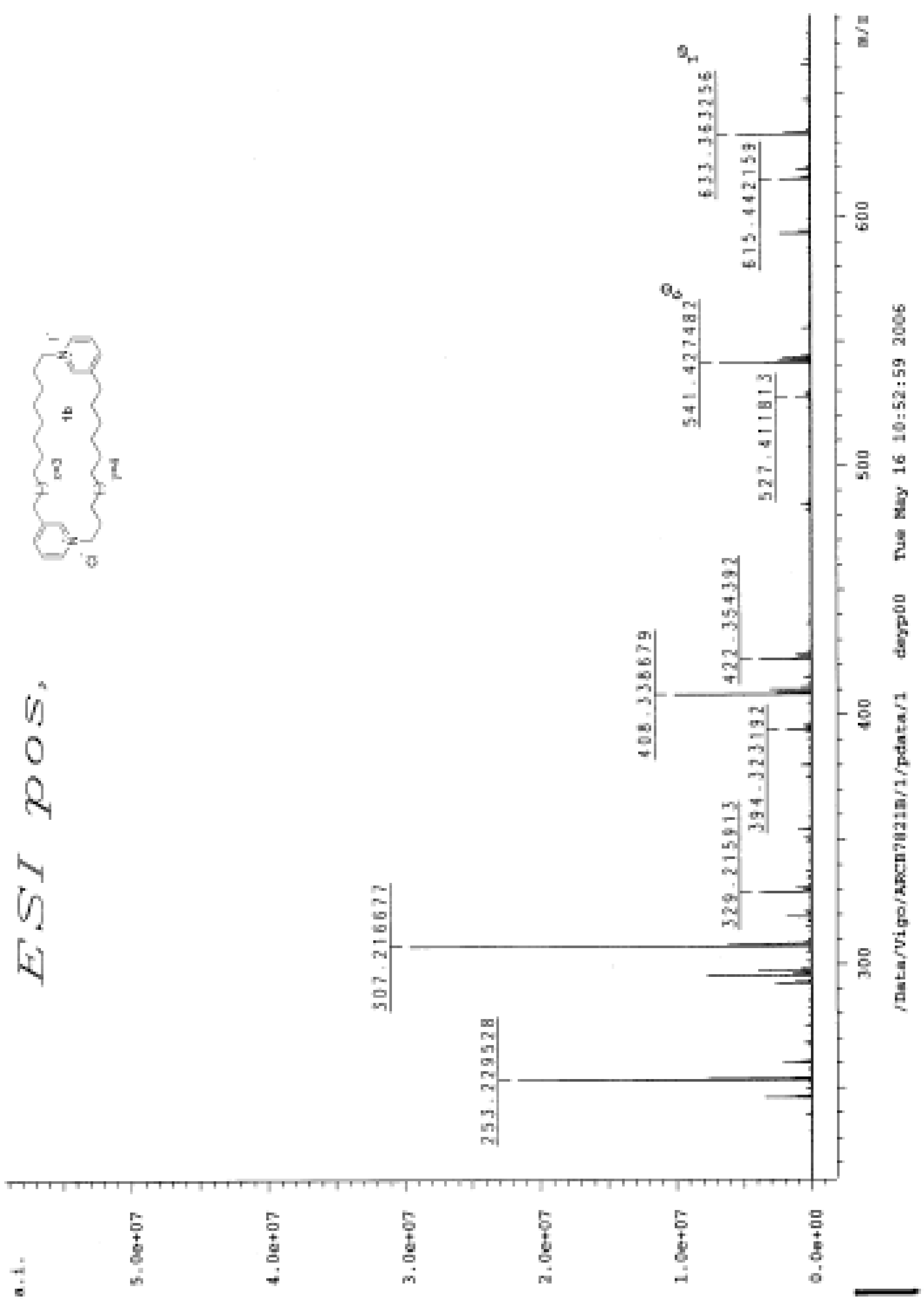
144.21
145.13
ppm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1.0e+07
0.0e+00
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307.216677
329.215913
394.323192
408.338679
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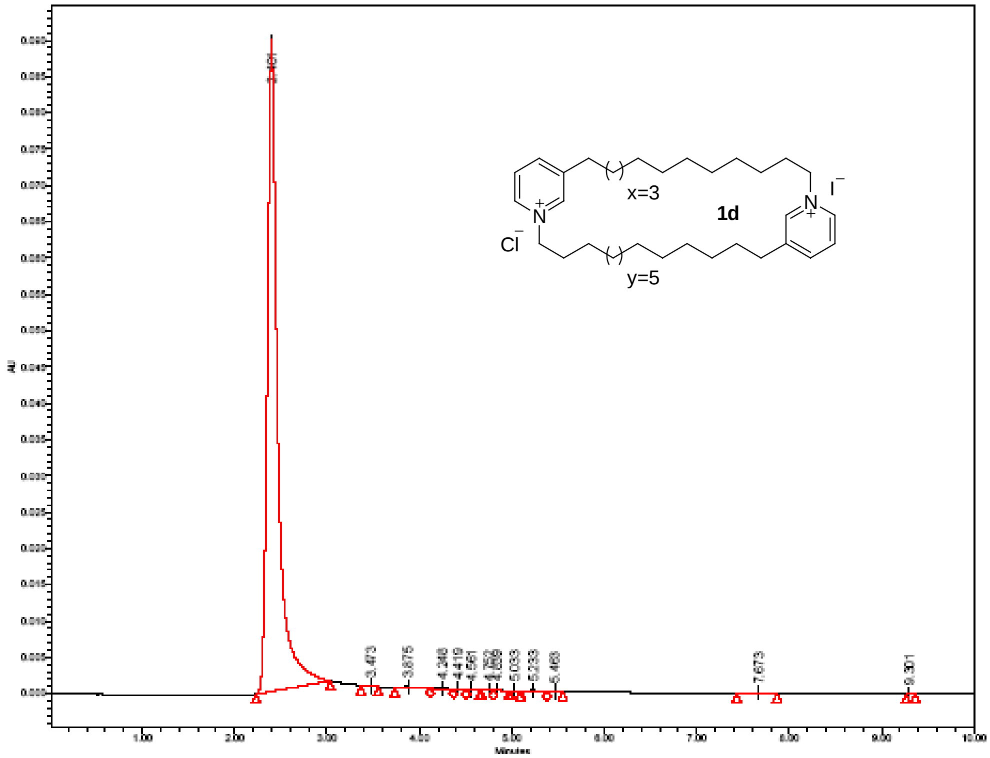
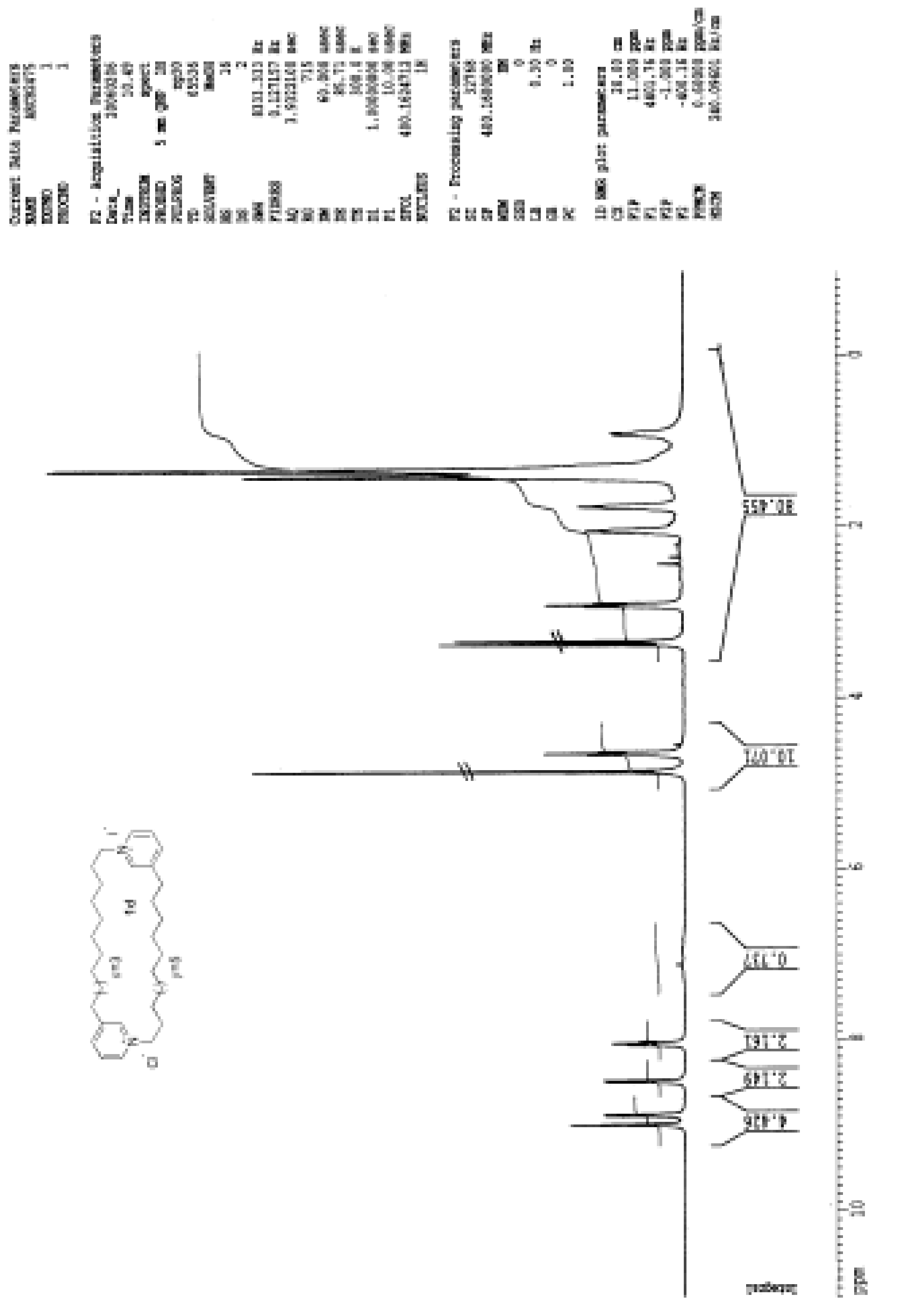
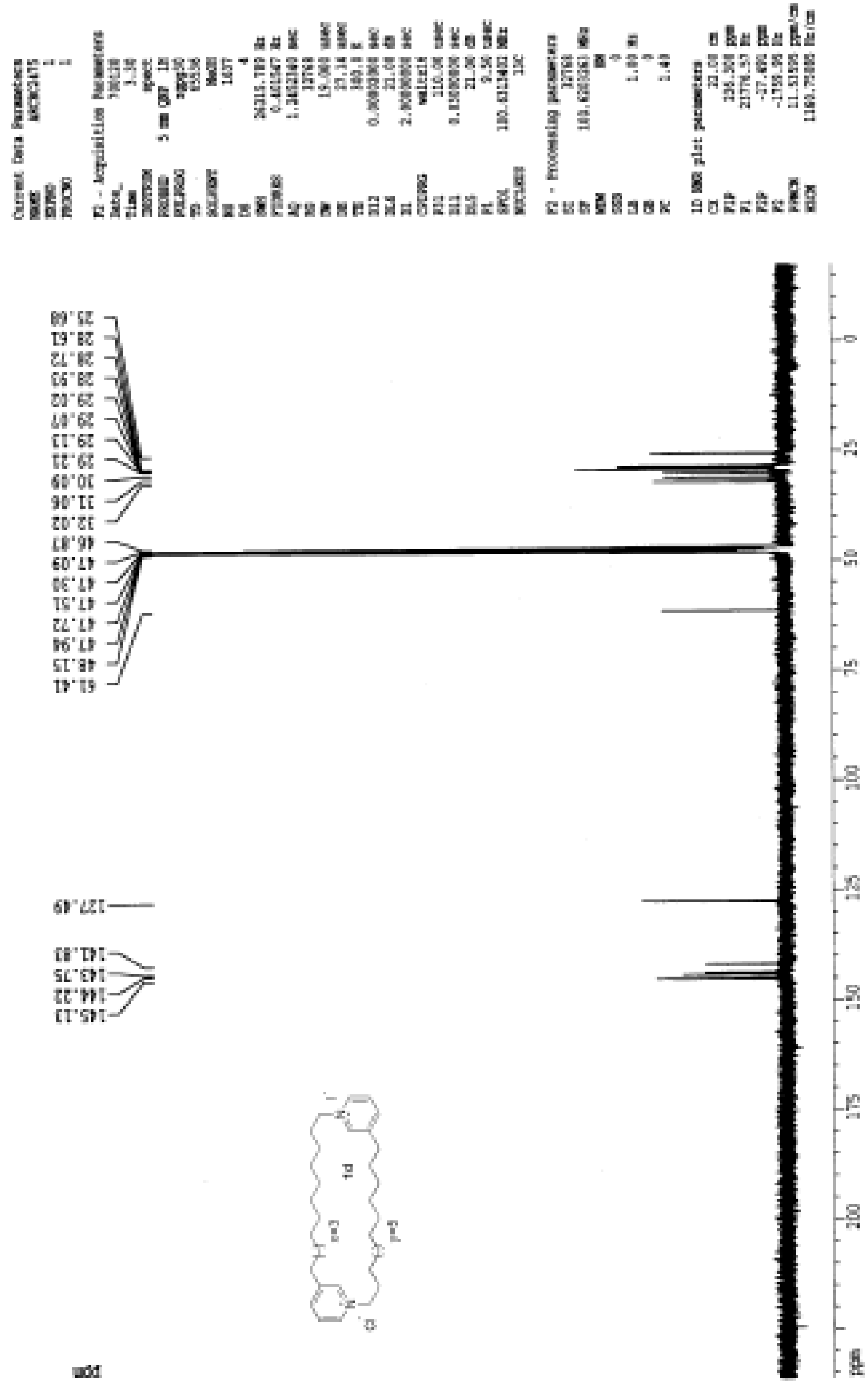
400
500
600
m/z
Tue May 16 10:52:59 2006



	Name	Retention Time (min)	Area (µV*sec)	% Area	Height (µV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		2.401	710569	99.27	89255	BB			Unknown	
2		3.473	89	0.01	16	BB			Unknown	
3		3.875	1716	0.24	176	BV			Unknown	
4		4.248	927	0.13	107	VV			Unknown	
5		4.419	258	0.04	57	VV			Unknown	I19
6		4.561	175	0.02	30	VB			Unknown	
7		4.752	185	0.03	35	BV			Unknown	
8		4.839	170	0.02	35	VB			Unknown	I19
9		5.033	33	0.00	12	BB			Unknown	I19
10		5.233	670	0.09	90	BV			Unknown	
11		5.463	80	0.01	15	VB			Unknown	
12		7.673	885	0.12	84	BB			Unknown	
13		9.301	24	0.00	8	BB			Unknown	I19

Develosil C-30
H_20/MeOH:7/93 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm

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28.93
29.02
29.07
29.13
29.21
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31.06
32.02
46.87
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47.72
47.94
48.15
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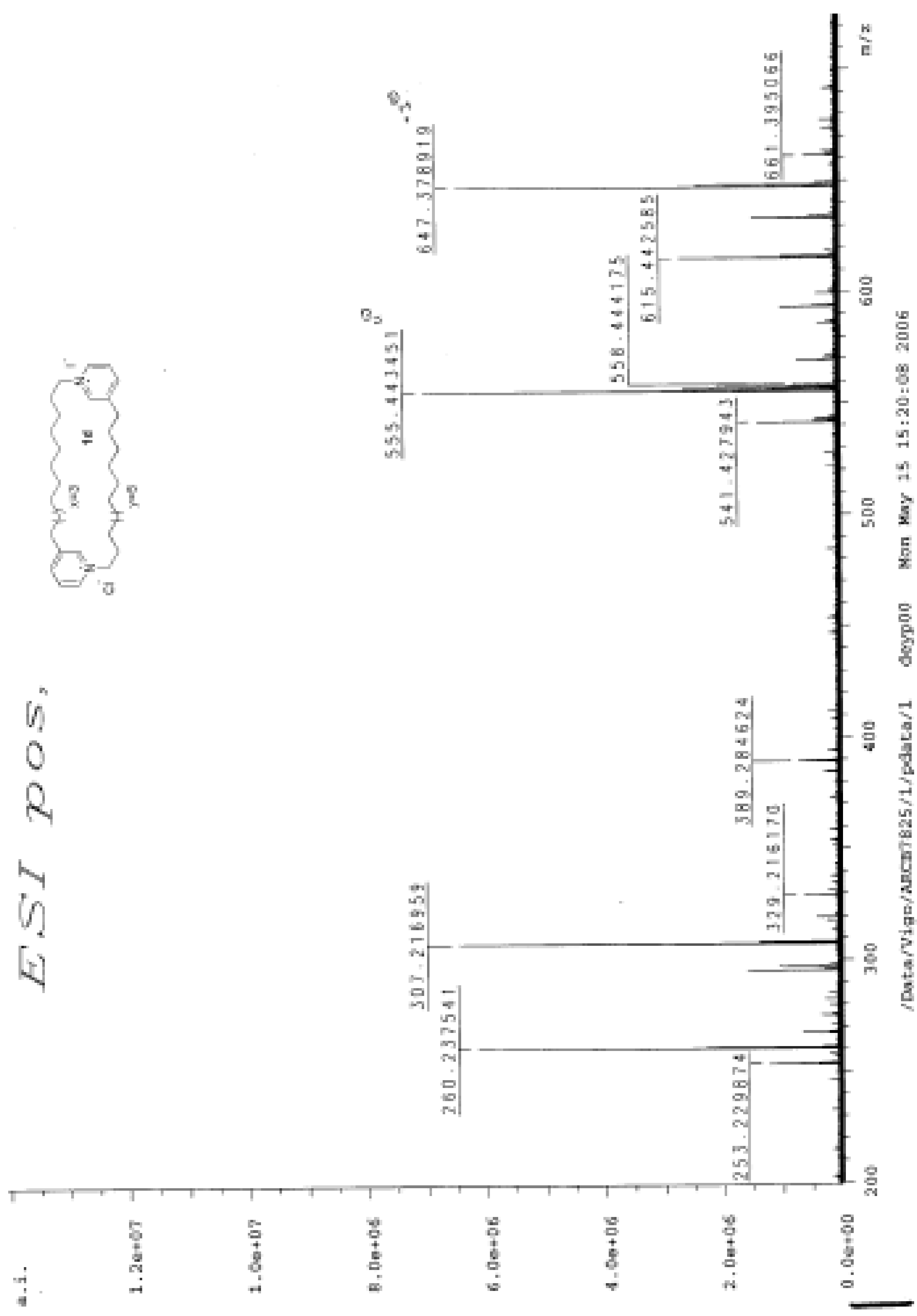
144.32
145.13
ppm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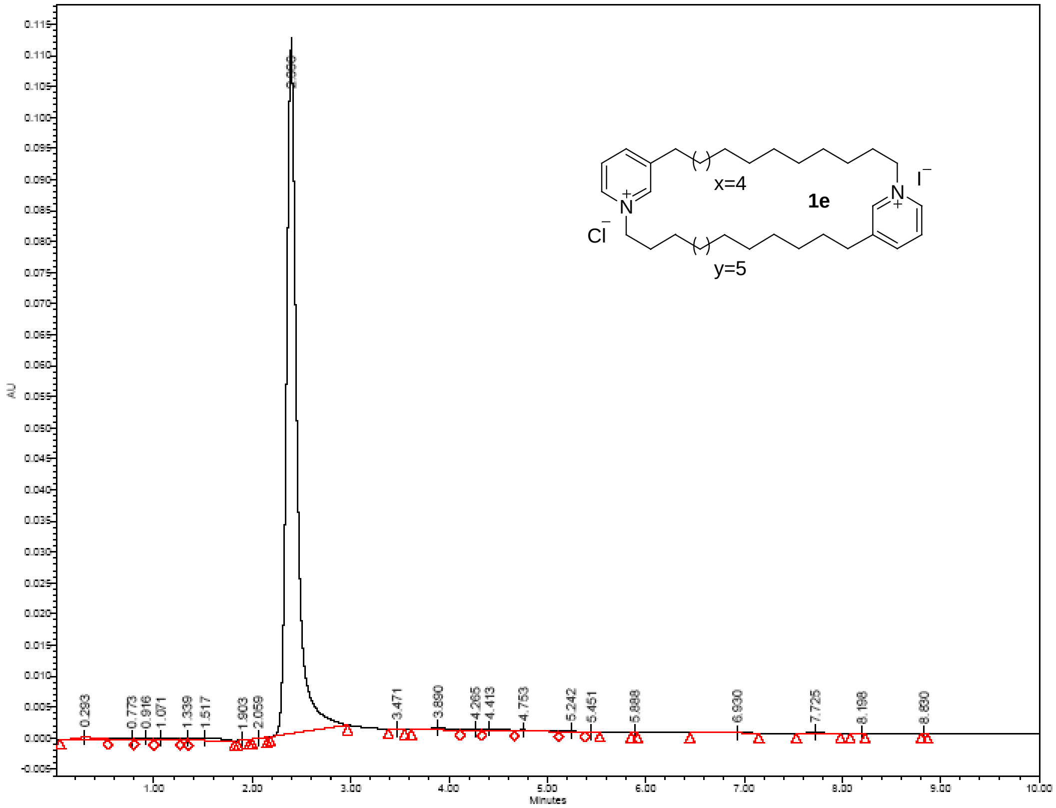
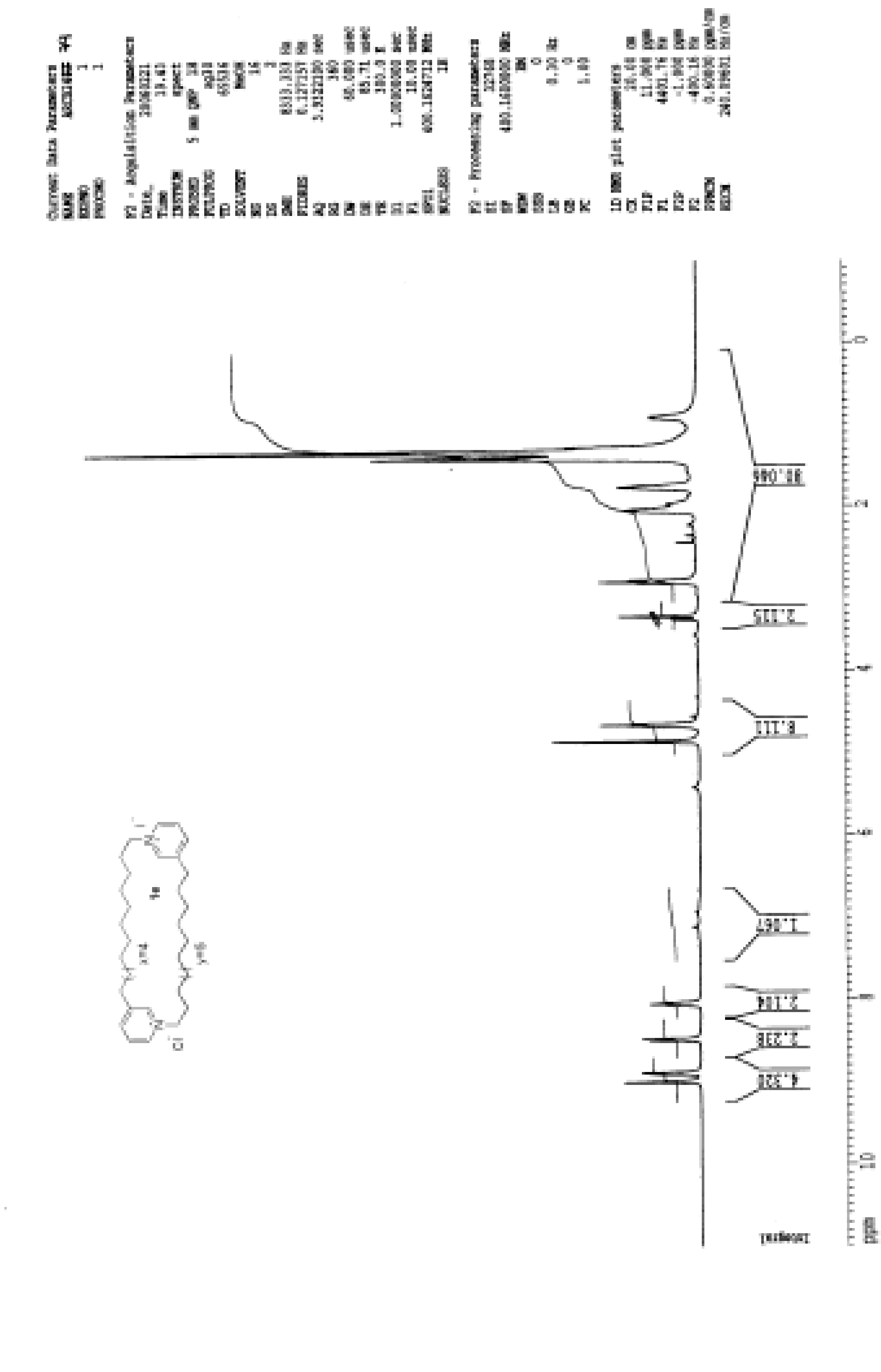
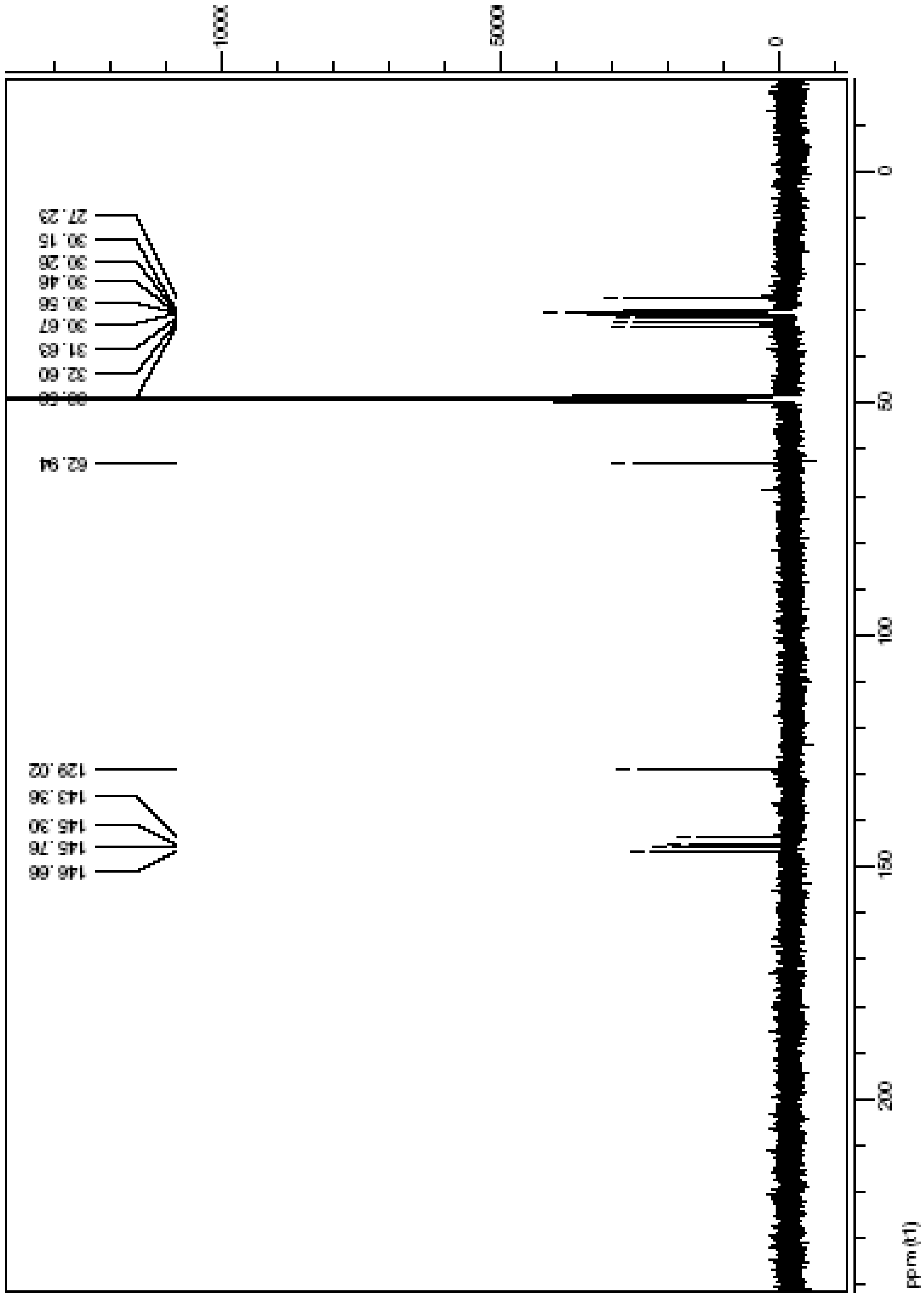
m/z
Mon May 15 15:20:08 2006



	Name	Retention Time (min)	Area (µV*sec)	% Area	Height (µV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		0.293	6357	0.72	363	BV			Unknown	
2		0.773	4641	0.53	339	VV			Unknown	
3		0.916	4283	0.49	372	VV			Unknown	
4		1.071	5506	0.63	381	VV			Unknown	
5		1.339	1619	0.18	332	VV			Unknown	I19
6		1.517	7608	0.87	354	VB			Unknown	
7		1.903	202	0.02	57	BB			Unknown	I19
8		2.059	62	0.01	14	BB			Unknown	
9		2.396	828709	94.48	111345	BB			Unknown	
10		3.471	126	0.01	26	BB			Unknown	
11		3.890	5093	0.58	319	BV			Unknown	
12		4.265	2959	0.34	251	VV			Unknown	
13		4.413	4145	0.47	306	VV			Unknown	
14		4.753	3034	0.35	183	VV			Unknown	
15		5.242	1050	0.12	129	VV			Unknown	
16		5.451	46	0.01	10	VB			Unknown	I19
17		5.888	11	0.00	6	BB			Unknown	I19
18		6.930	759	0.09	38	BB			Unknown	I19
19		7.725	884	0.10	83	BB			Unknown	
20		8.198	46	0.01	9	BB			Unknown	I19
21		8.830	12	0.00	6	BB			Unknown	I19

Develosil C-30
H_20/MeOH:7/93 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm





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32.60
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0
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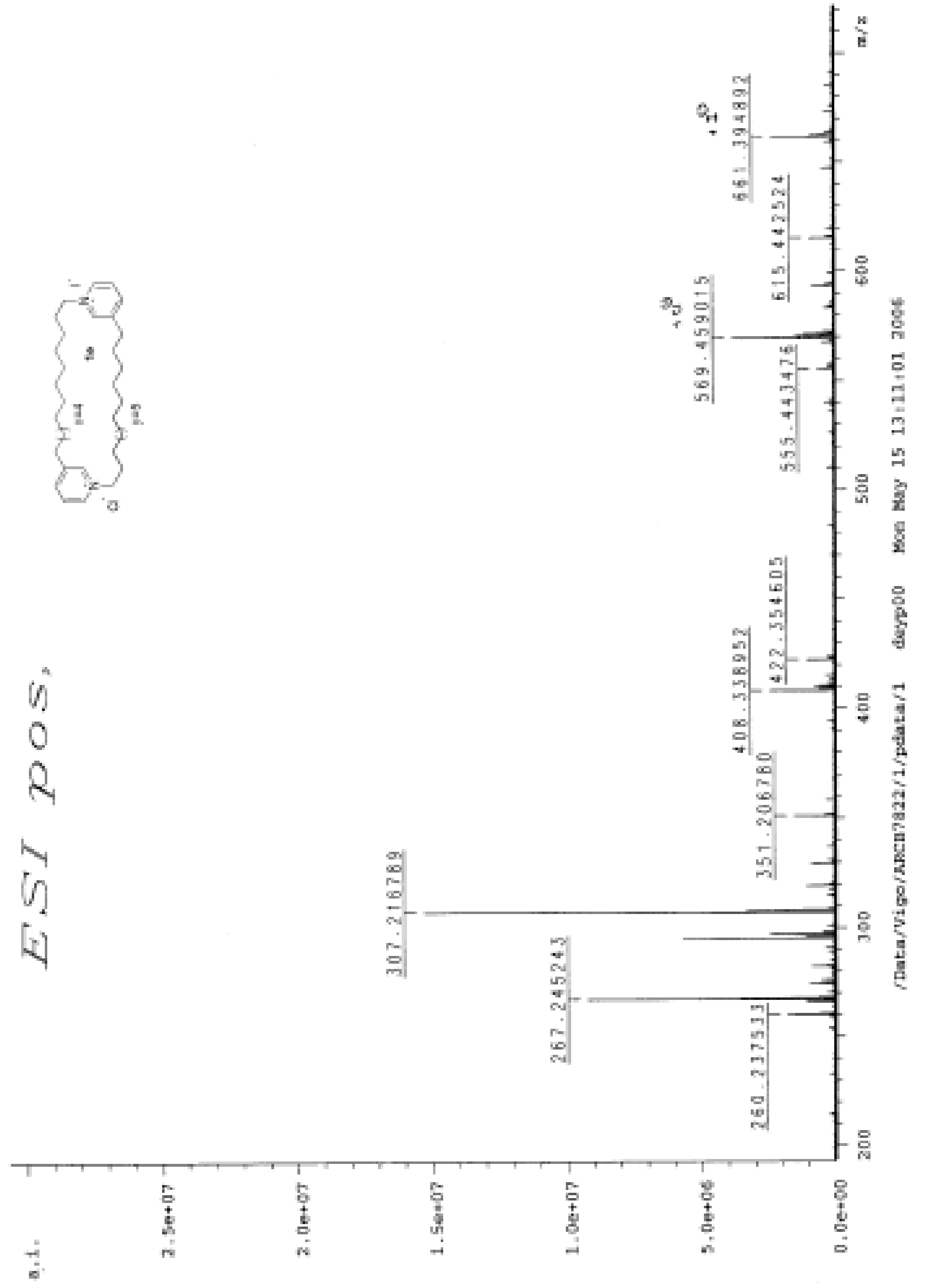
100
150
200
ppm (t1)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422.354605
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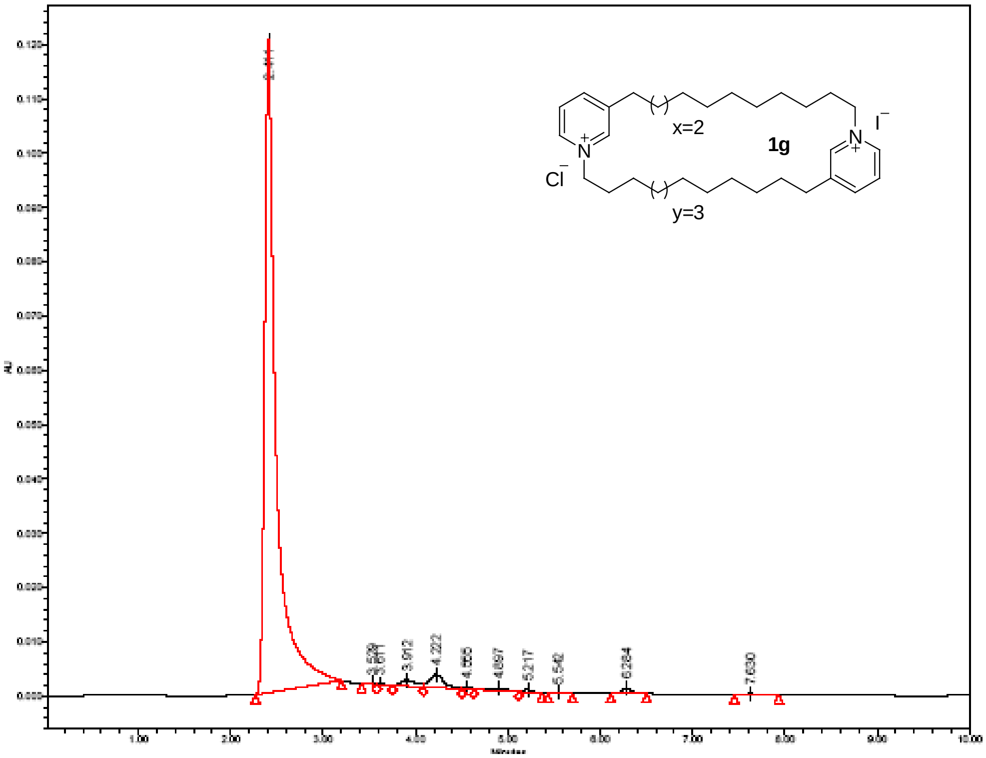
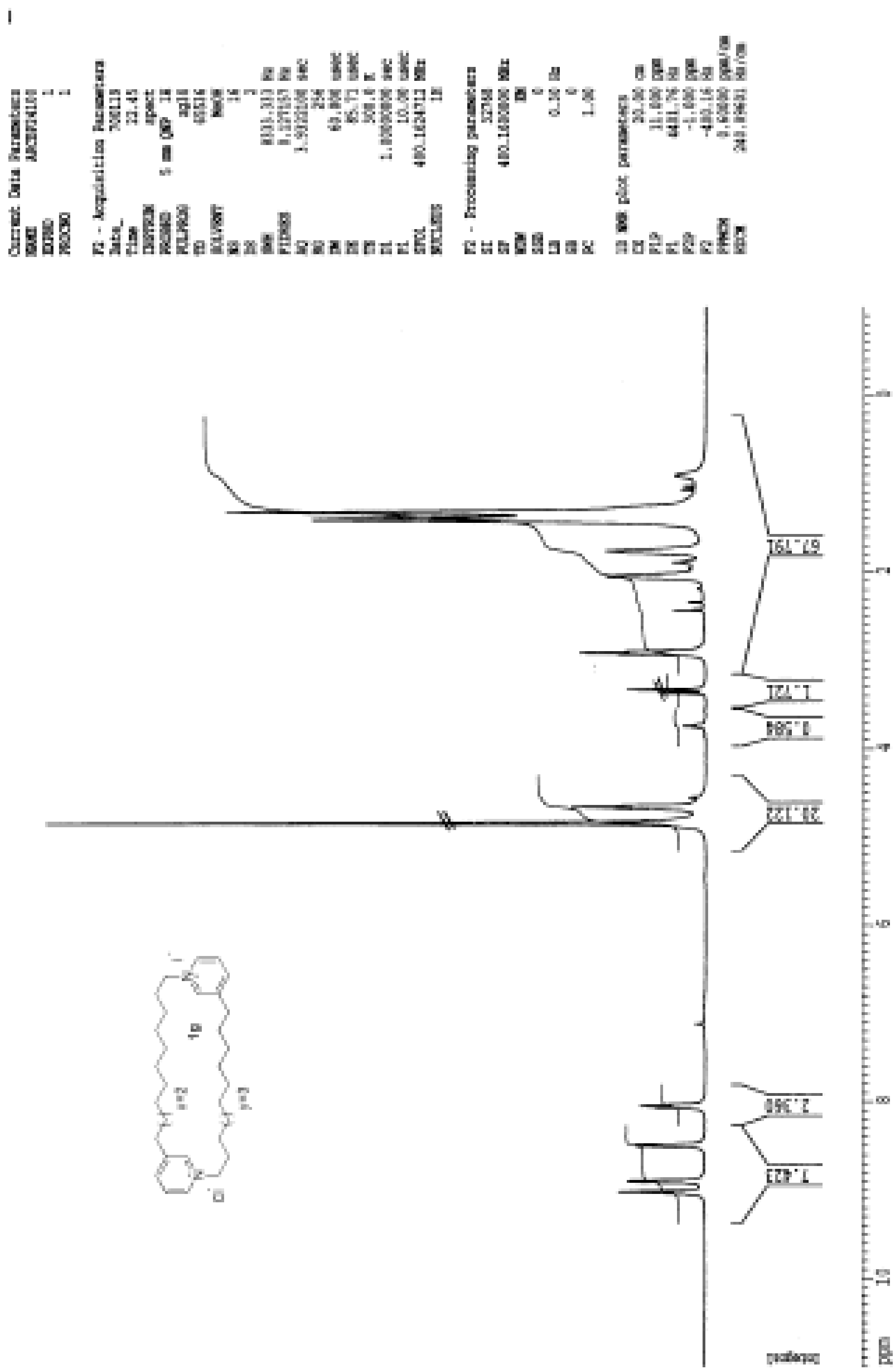
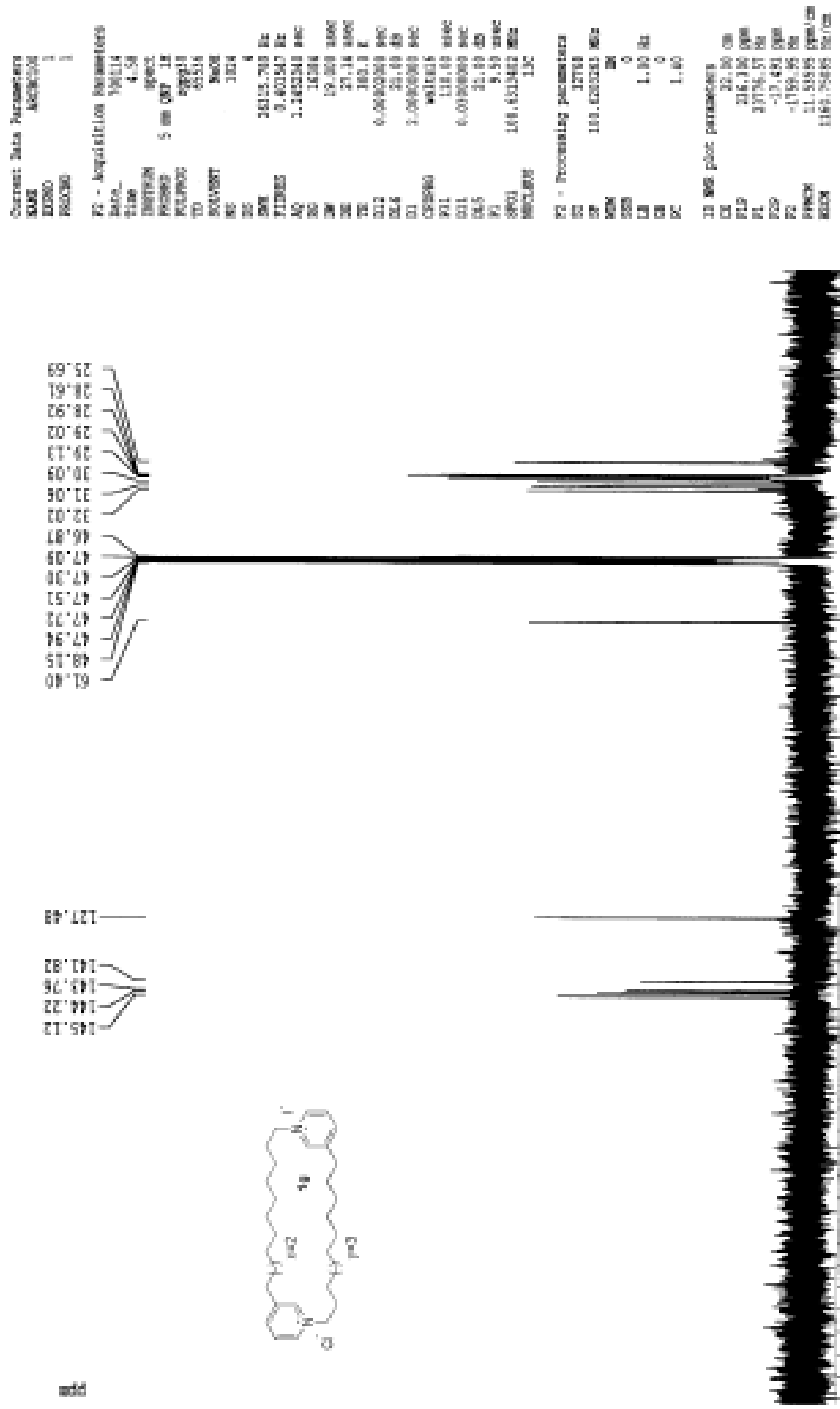
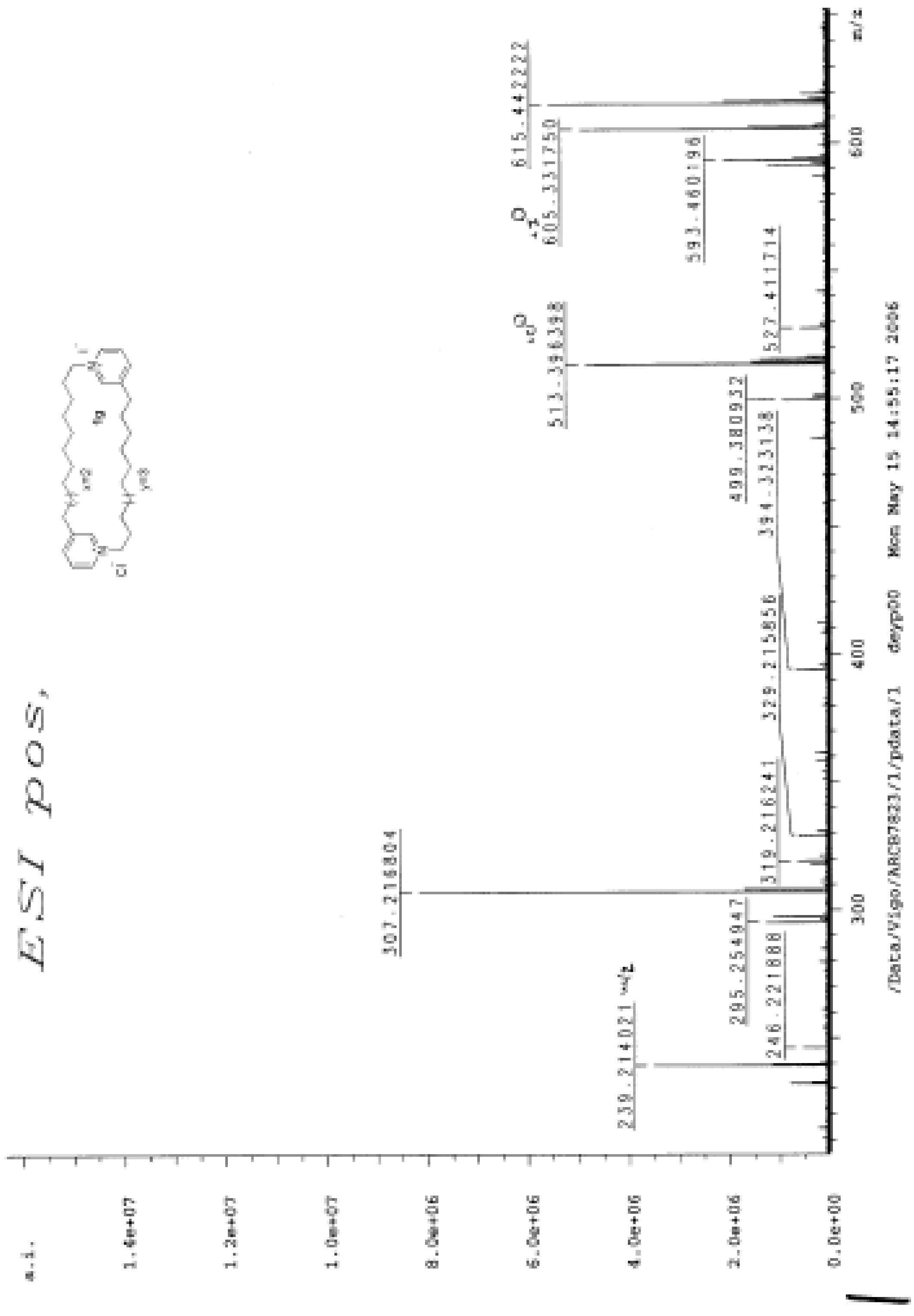


	Name	Retention Time (min)	Area (µV*sec)	% Area	Height (µV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		2.411	1054936	95.06	120471	BB			Unknown	
2		3.529	1187	0.11	193	BV			Unknown	
3		3.611	1185	0.11	163	VV			Unknown	
4		3.912	12890	1.15	1089	VV			Unknown	
5		4.222	26471	2.36	2472	VV			Unknown	
6		4.555	2016	0.18	265	VV			Unknown	
7		4.897	8289	0.74	454	VV			Unknown	
8		5.217	3275	0.29	472	VB			Unknown	
9		5.542	778	0.07	114	BB			Unknown	
10		6.284	7870	0.70	1006	BB			Unknown	
11		7.630	2620	0.23	236	BB			Unknown	

Develosil C-30
H_20/MeOH:7/93 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm



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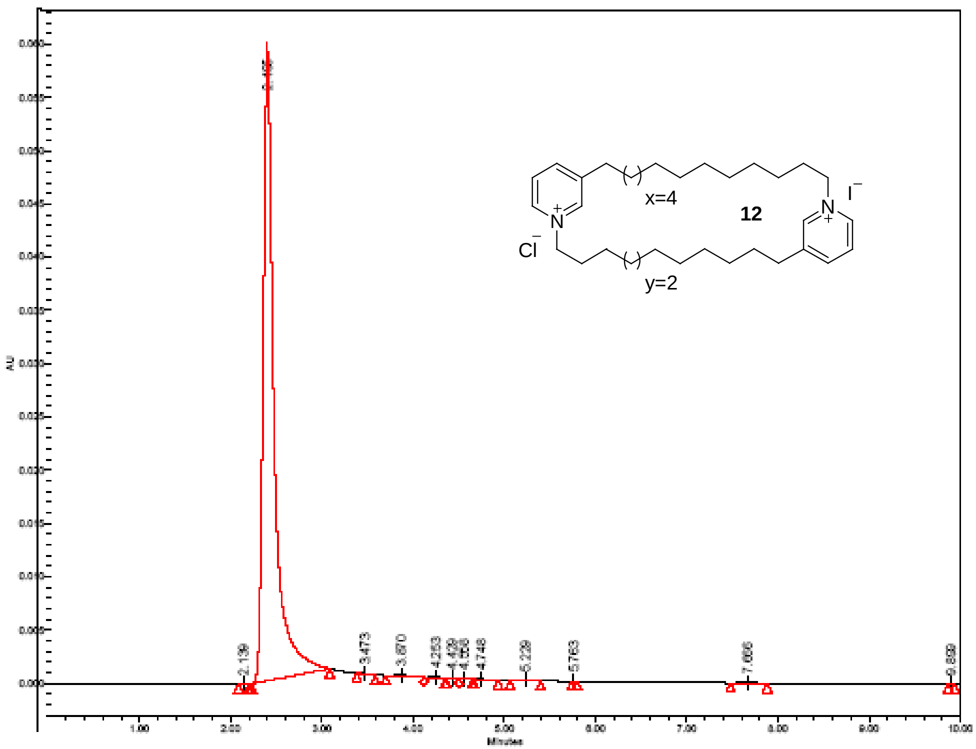
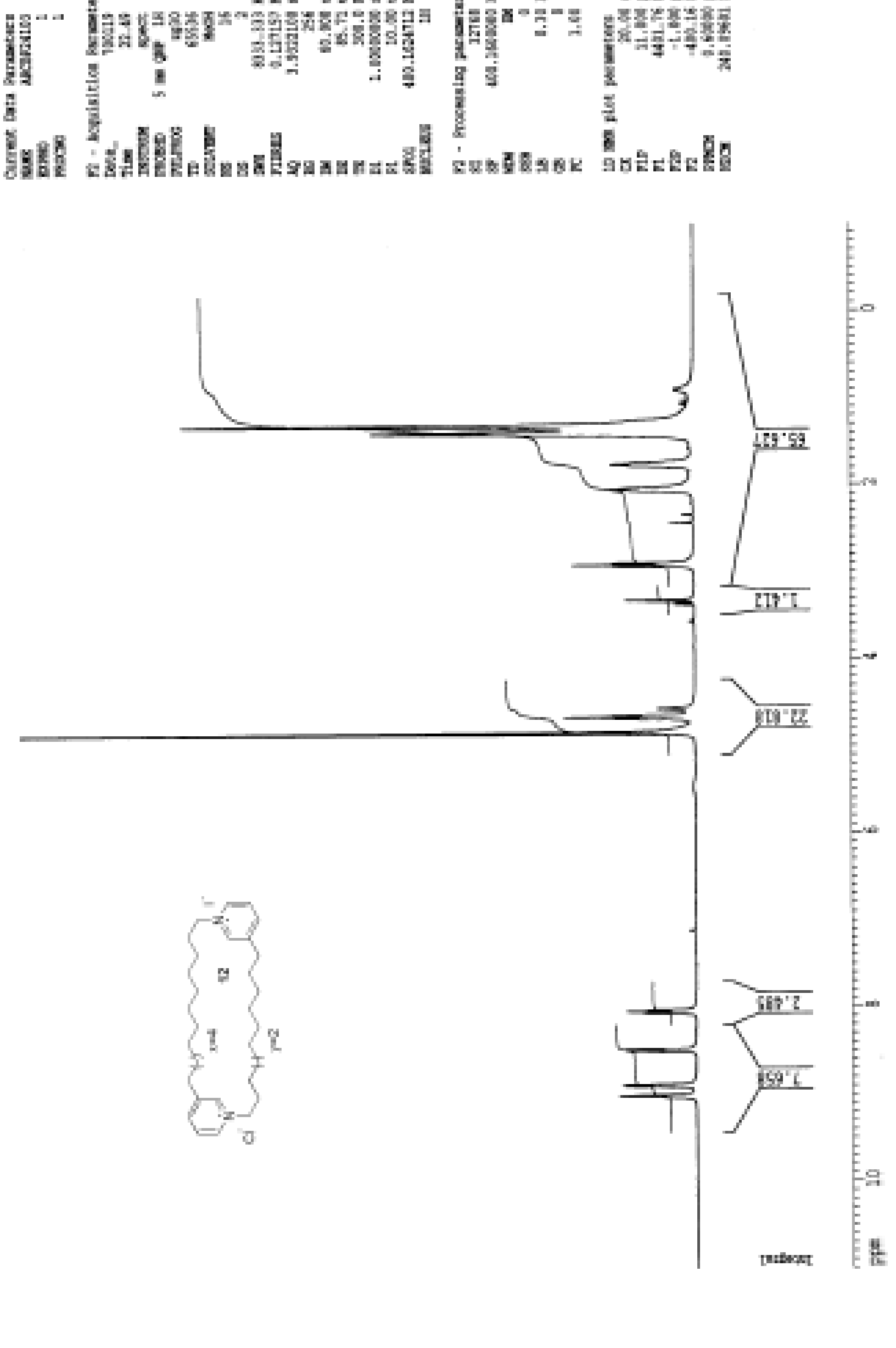
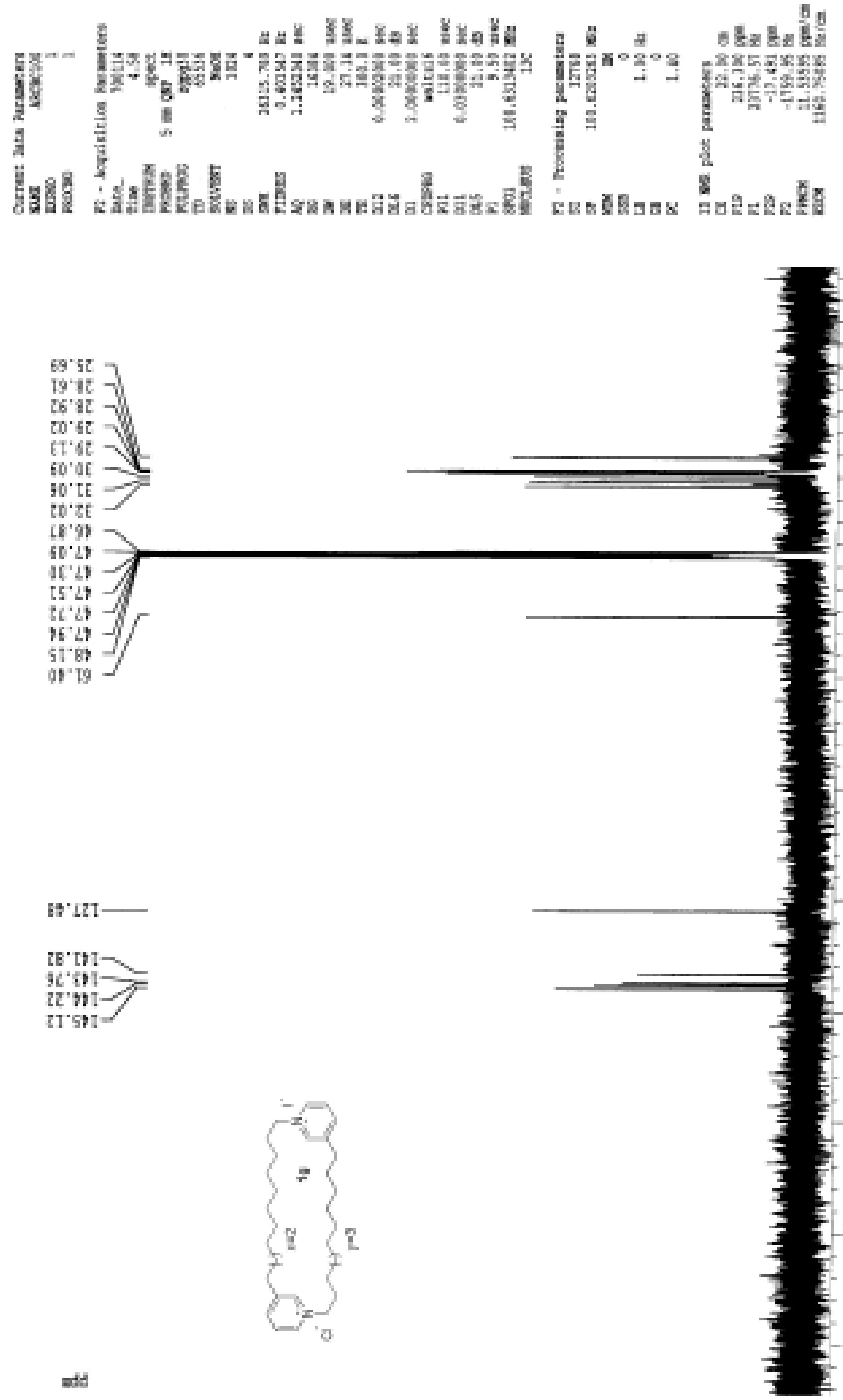


	Name	Retention Time (min)	Area (µV*sec)	% Area	Height (µV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		2.139	37	0.01	10	BB			Unknown	I19
2		2.405	499472	99.26	59774	BB			Unknown	
3		3.473	111	0.02	20	BB			Unknown	
4		3.870	1275	0.25	140	BV			Unknown	
5		4.253	644	0.13	82	VB			Unknown	
6		4.429	223	0.04	43	BV			Unknown	
7		4.558	139	0.03	24	VB			Unknown	
8		4.748	220	0.04	32	BB			Unknown	
9		5.229	455	0.09	58	BB			Unknown	
10		5.763	13	0.00	6	BB			Unknown	I19
11		7.666	616	0.12	57	BB			Unknown	
12		9.899	15	0.00	7	BB			Unknown	I19

Develosil C-30
$H_2$0/MeOH:7/93 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm





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29.02
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30.09
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32.03
46.87
47.09
47.30
47.51
47.73
47.94
48.15
61.40
127.48
141.82
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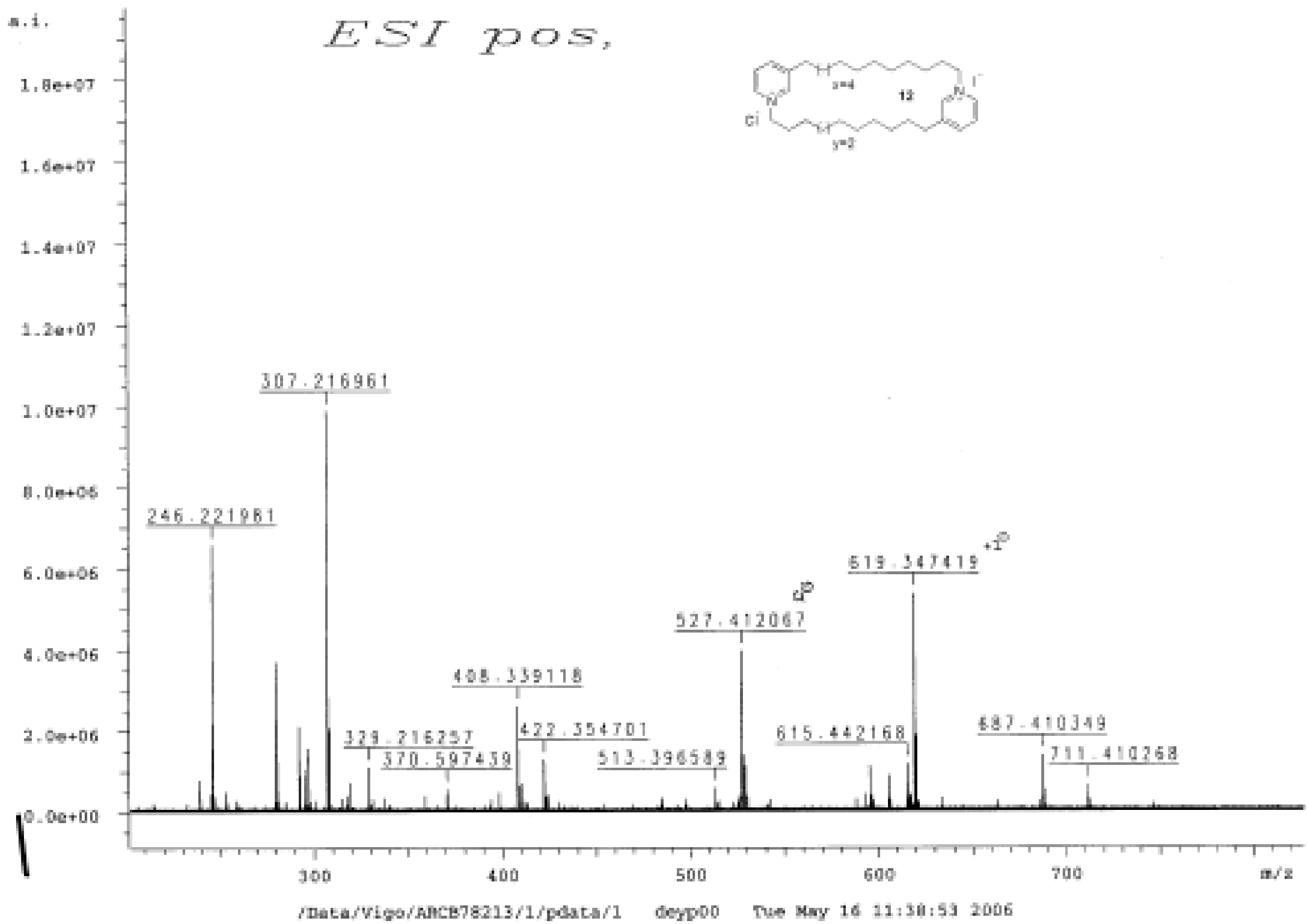
144.22
145.12
ppm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513.396589
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Cl
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700
m/z
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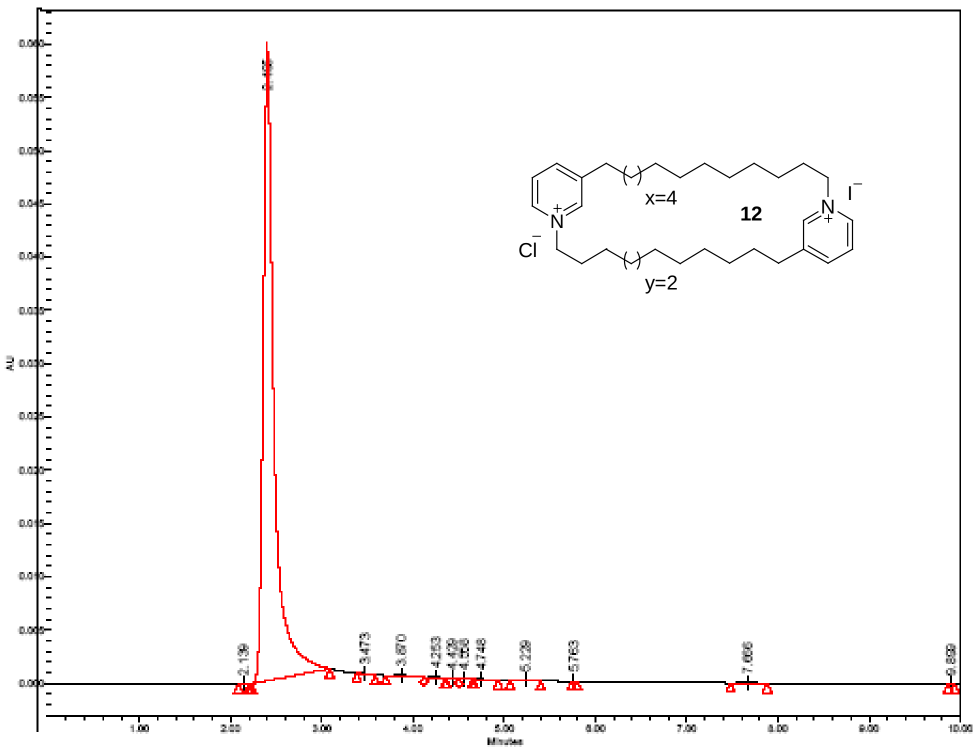
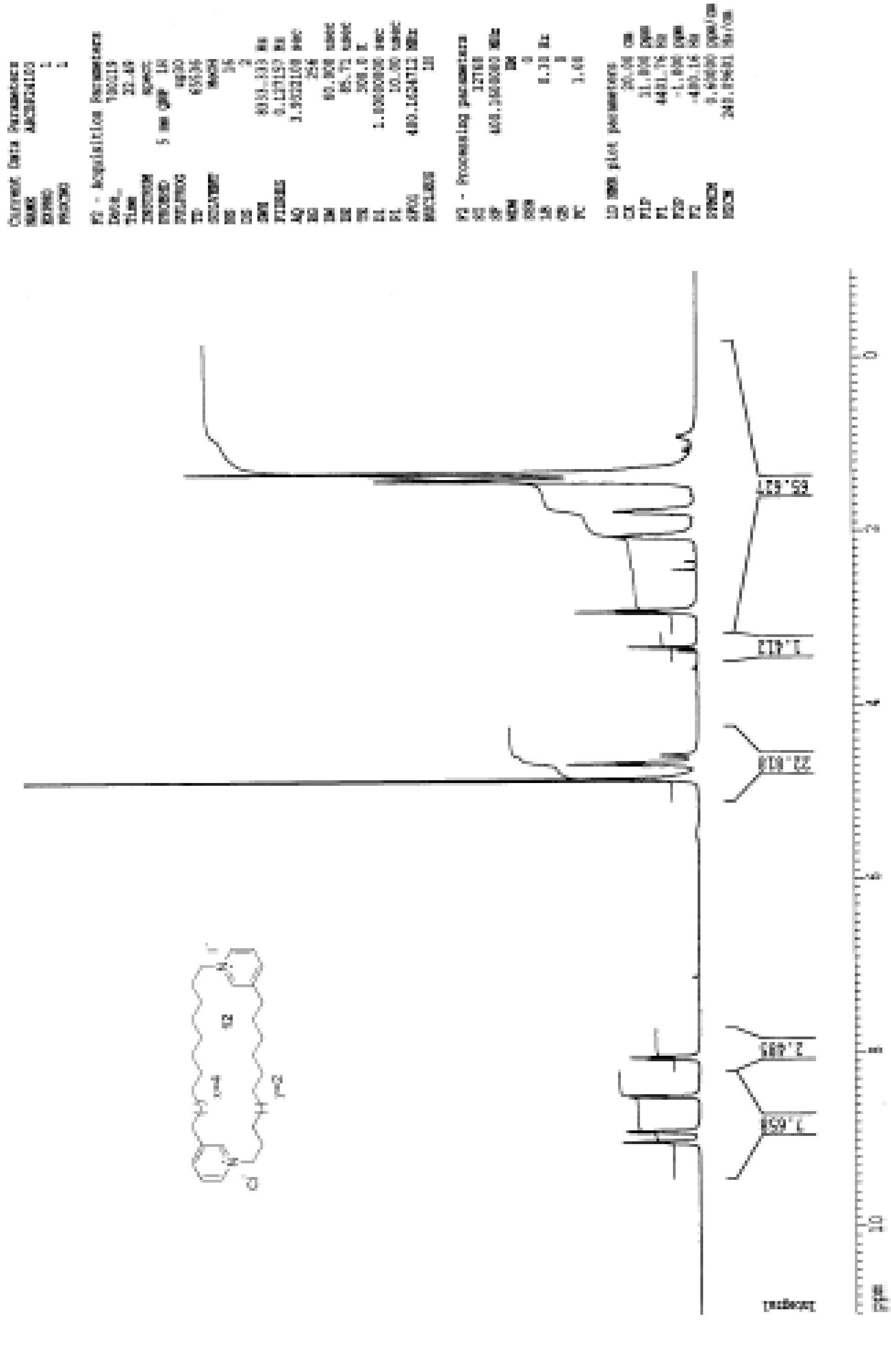
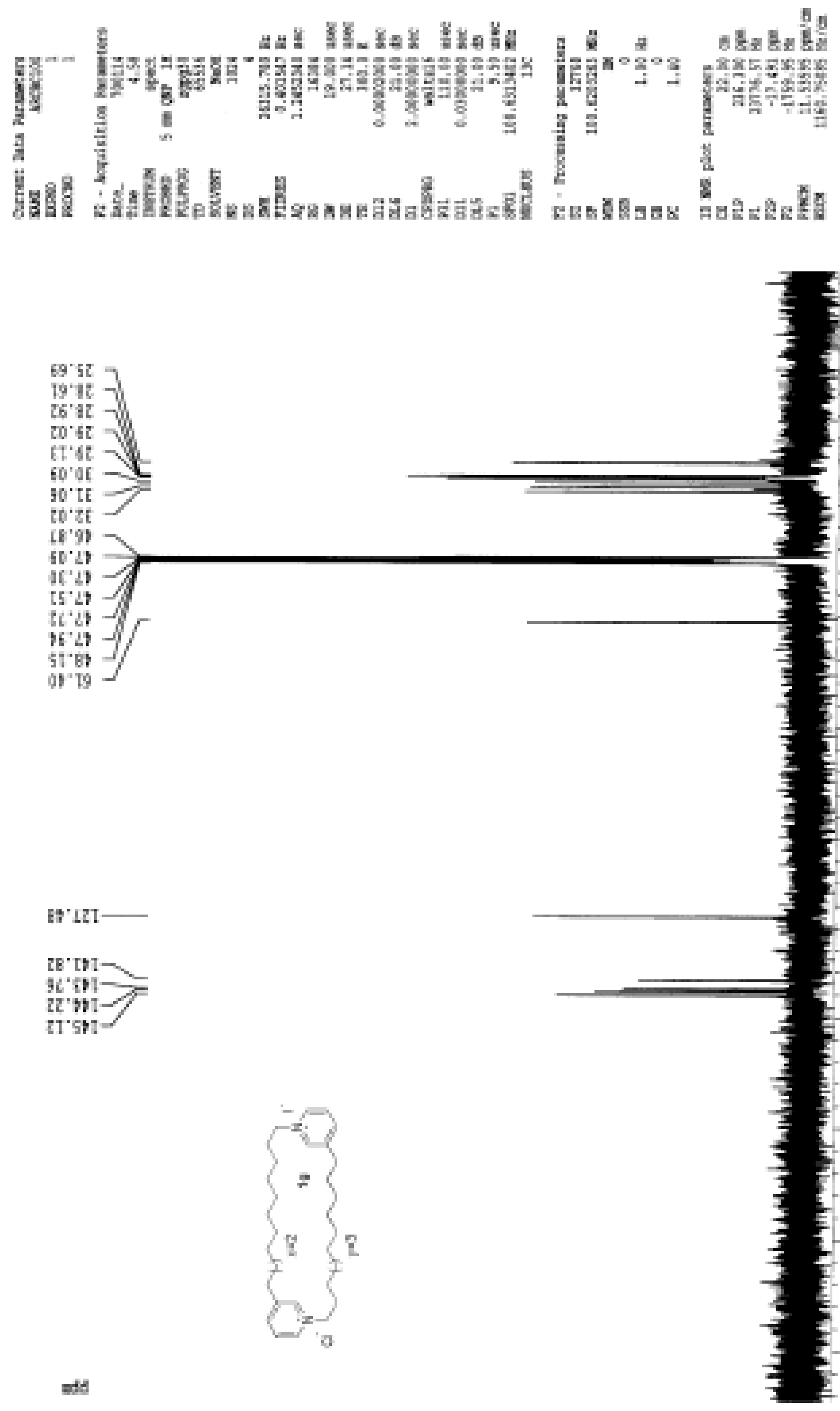


	Name	Retention Time (min)	Area (μV*sec)	% Area	Height (μV)	Int Type	Amount	Units	Peak Type	Peak Codes
1		2.139	37	0.01	10	BB			Unknown	I19
2		2.405	499472	99.26	59774	BB			Unknown	
3		3.473	111	0.02	20	BB			Unknown	
4		3.870	1275	0.25	140	BV			Unknown	
5		4.253	644	0.13	82	VB			Unknown	
6		4.429	223	0.04	43	BV			Unknown	
7		4.558	139	0.03	24	VB			Unknown	
8		4.748	220	0.04	32	BB			Unknown	
9		5.229	455	0.09	58	BB			Unknown	
10		5.763	13	0.00	6	BB			Unknown	I19
11		7.666	616	0.12	57	BB			Unknown	
12		9.899	15	0.00	7	BB			Unknown	I19

Develosil C-30
$H_2$0/MeOH:7/93 (0.1% TFA)
Flow: 3.2 mL/min
υ=267 nm

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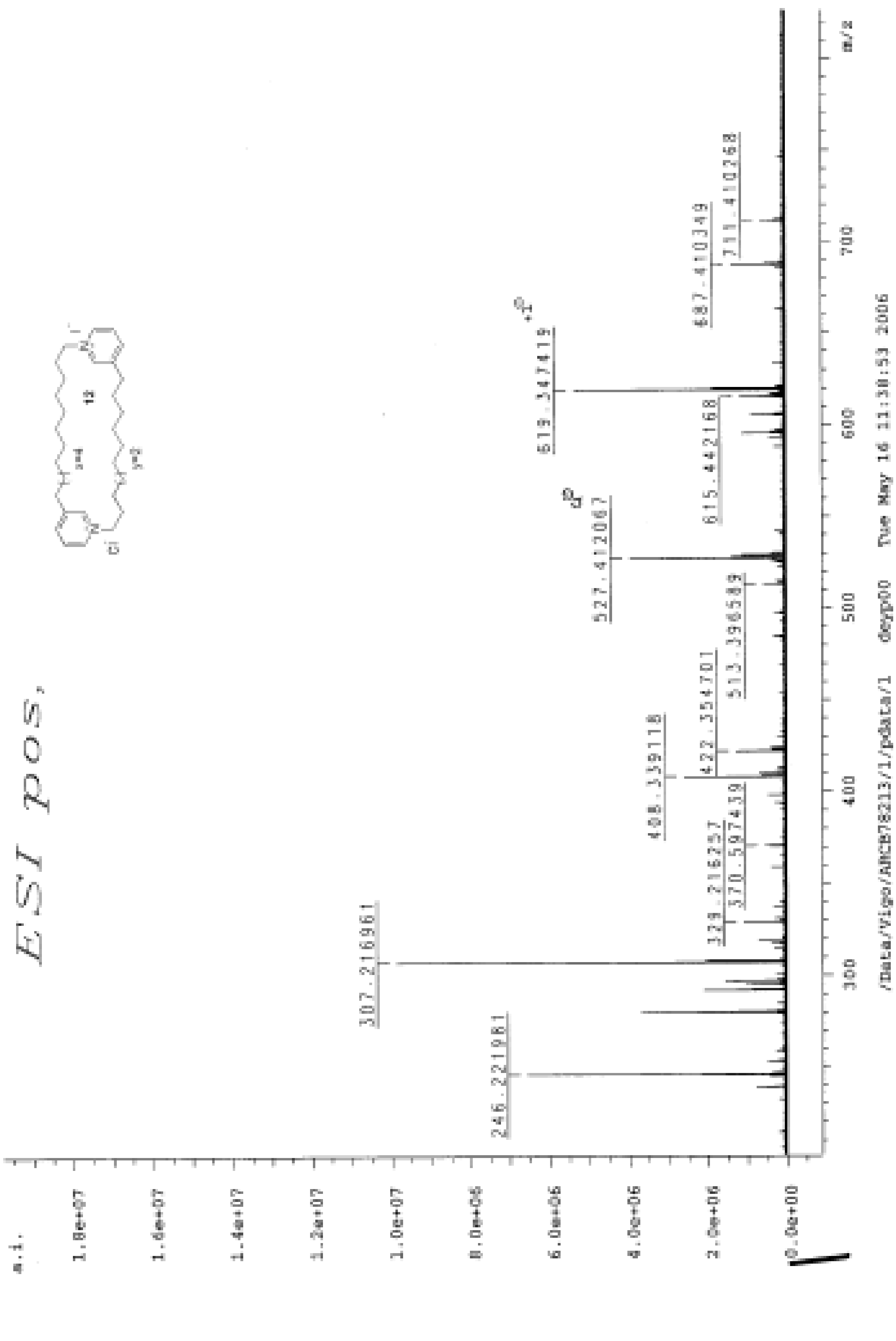
ppm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