Supporting Information

General experimental procedures

Melting points were measured in Melt-Temp apparatus and were uncorrected. ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded in $CDCl_3$ (unless indicated elsewhere) using Bruker AM-500 instruments with Me_4Si and $CFCl_3$ (with upfield negative) as the internal and external standards, respectively. IR spectra were obtained with a Nicolet AV-360 spectrophotometer. Lower resolution mass spectra were determined with Agilent 1100 LC/MSD SL instrument using ESI technique. High Resolution mass spectra were run on Ionspec 4.7 Tesla FTMS using MALDI/DHB. All the solvents were not purified before use. X-ray diffraction crystal structure analysis was obtained on Bruker P4 instrument using graphite monochromatized Mo Kα radiation (λ = 0.71073 Å) at 293(2) K.

General procedure for the preparation of 4g:

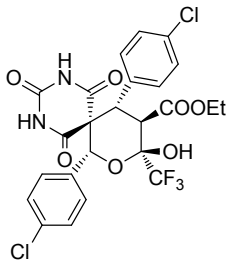
To a mixture of pyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione **1** (128.0 mg, 1.0mmol) benzaldehyde **2g** (212.0 mg, 2.0 mmol), and ethyl trifluoroacetoacetate **3** (184.0 mg, 1.0 mmol) in DMSO (6 mL) was added 0.5 mmol of Et_3N as catalyst. The resultant mixture was stirred at room temperature for as indicated hour. After completion of the reaction, the mixture was poured into water (50 mL) and extracted with ethyl acetate (3X50 mL). The organic phase was washed with water for several times, then dried over anhydrous magnesium sulfate. After evaporation of solvent on under reduced pressure, the residue was purified by column chromatography on a silica gel using petroleum ether/ethyl acetate (4/1, v/v) as eluent to afford the pure product **4g** 364.2mg, 72% yield.

Typical experimental procedure for preparation of 5d

To a stir CH_3CN solution (10.0 mL) containing compound **4g** (506 mg, 1.0 mmol) and pyridine (395 mg, 5.0 mmol) was added dropwise $SOCl_2$ (595 mg, 5.0 mmol) at room temperature. The resultant mixture was stirred at room temperature for indicated hours until completion of reaction (monitored by TLC). The mixture was concentrated under reduced pressure, the residue was purified by column chromatography on a silica gel using petroleum ether/ethyl acetate 6:1 (v/v) as eluent to afford the pure product **5d** 248.5 mg, 51% yield.

Characterization data for all compounds

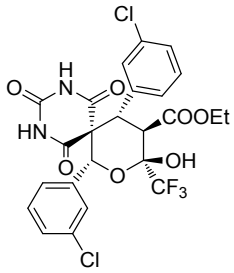
Ethyl-7,11-bis(4-chlorophenyl)-9-hydroxy-1,3,5-trioxo-9-(trifluoromethyl) -8-oxa-2,4-diazaspiro[5.5]undecane-10-carboxylate (4a)



The product was recrystallized form PE-EtOAc contained one molecule of EtOAc.

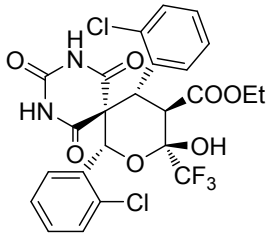
White solid; mp 290.2-290.4 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 0.86 (t, J = 7.5 Hz, 3H), 3.92-3.94 (m, 2H), 4.33 (d, J = 13.0 Hz ,1H), 4.44 (d, J = 13.0 Hz, 1H), 5.80 (s, 1H), 6.18 (s,1H), 7.14-7.27 (m, 8H), 7.97 (s, 1H), 8.21 (s, 1H); three crystallization solvent peaks were also observed: 1.25 (t, J = 7.0 Hz, 3H, solvent), 2.03 (s, 3H), 4.10 (q, J = 7.0 Hz ,2H), 4.10 (q, J = 7.0 Hz ,2H), ^{19}F NMR ($CDCl_3$, 470 MHz): δ -85.10 (s, 3F); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.3, 41.8, 46.2, 60.0, 62.6, 75.9, 94.1 (q, $^2J_{C\text{-}F}$ = 33.4 Hz), 122.1 (q, $^1J_{C\text{-}F}$ = 248.1 Hz), 128.0, 129.0, 129.3, 130.6, 131.7, 132.1, 135.4, 135.7, 147.2, 166.6, 168.6, 173.2; four crystallization solvent peaks were also observed: 14.1, 21.0, 60.6, 171.6; IR (KBr) ν_{max}: 3384.5, 3107.6, 2855.2, 1712.8, 1492.8, 1184.4, 1016.6, 834.5 cm^{-1}; MS [ESI(-)] *m/z*: [(M-H)$^+$, 573]; HRMS Calcd. for $C_{24}H_{19}Cl_2F_3N_2O_7Na$ [M+Na]$^+$: 597.0419, Found: 597.0414.

Ethyl-7,11-bis(3-chlorophenyl)-9-hydroxy-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undecane-10-carboxylate (4b)



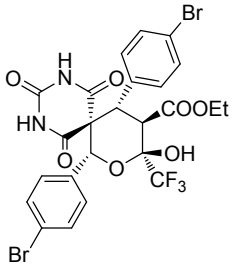
White solid; mp 265.8-266.0 °C; ^{1}H NMR (Acetone-d_6, 500 MHz): δ 0.90 (t, J = 7.5 Hz, 3H), 3.87-3.99 (m, 2H), 4.60 (d, J = 13.0 Hz, 1H), 4.63 (d, J = 13.0 Hz, 1H), 5.88 (s, 1H),7.06 (s, 1H), 7.18-7.42 (m, 8H), 10.30 (s, 1H), 10.50 (s, 1H); ^{19}F NMR (Acetone-d_6, 470 MHz): δ -84.56 (s, 3F); ^{13}C NMR (Acetone-d_6, 100 MHz): δ 13.0, 43.2, 45.6, 59.4, 61.0, 75.4, 94.7 (q, $^2J_{C\text{-}F}$ = 32.7 Hz), 122.5 (q, $^1J_{C\text{-}F}$ = 285.2 Hz), 125.3, 126.8, 127.5, 128.8, 128.9, 129.5, 130.3, 130.6, 134.1, 136.8, 137.0, 147.4, 167.4, 168.8, 169.4; IR(KBr) ν_{max}: 3221.5, 3080.3, 2869.1, 1716.4, 1373.9, 1210.7, 1122.7, 767.7 cm^{-1}; MS [ESI(-)] m/z: [(M-H)$^+$, 573]; HRMS Calcd. for $C_{24}H_{19}Cl_2F_3N_2O_7Na$ [M+Na]$^+$: 597.0419, Found: 597.0416.

Ethyl-7,11-bis(2-chlorophenyl)-9-hydroxy-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undecane-10-carboxylate (4c)



White solid; mp 203.1-203.2 °C; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 0.82 (t, J = 7.0 Hz, 3H), 3.80-3.88 (m, 2H), 4.38 (d, J = 12.5 Hz, 1H), 4.45 (d, J = 12.5 Hz, 1H), 5.67 (s, 1H), 7.02-7.47 (m, 8H), 8.52 (s, 1H), 11.46 (s, 1H), 11.79 (s, 1H); ^{19}F NMR (DMSO-d_6, 470 MHz): δ -82.25 (s, 3F); ^{13}C NMR (Acetone-d_6, 100 MHz): δ13.6, 41.7, 45.3, 58.3, 62.0, 73.2, 95.4 (q, $^{2}J_{C\text{-}F}$ = 32.4 Hz), 123.3 (q, $^{1}J_{C\text{-}F}$ = 285.5 Hz), 128.1, 128.2, 130.3, 130.5, 130.7, 130.8, 131.3, 131.8, 133.0, 133.5, 133.7, 136.2, 148.4, 167.0, 169.4, 170.5; IR(KBr) ν_{max}: 3407.0, 3247.8, 3127.1, 2998.4, 1707.1, 1328.9, 1184.9, 752.3 cm^{-1}; MS [ESI(-)] m/z: [(M-H)$^{+}$, 573]; HRMS Calcd. for $C_{24}H_{19}Cl_2F_3N_2O_7Na$ [M+Na]$^{+}$: 597.0419, Found: 597.0428.

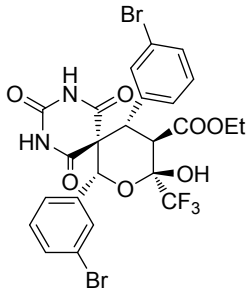
Ethyl-7,11-bis(4-bromophenyl)-9-hydroxy-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undecane-10-carboxylate (4d)



The product was recrystallized form PE-EtOAc contained one molecule of EtOAc.

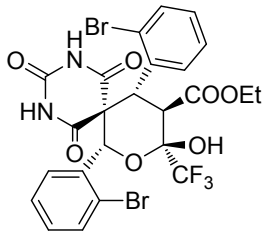
White solid; mp 288.3-288.7 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 0.87 (t, J = 7.5 Hz, 3H), 3.91-3.96 (m, 2H), 4.32 (d, J = 13.0 Hz, 1H), 4.45 (d, J = 13.0 Hz, 1H), 5.79 (s, 1H), 6.18 (s, 1H), 7.08-7.42 (m, 8H), 8.02 (s, 1H), 8.28 (s, 1H); three crystallization solvent peaks were also observed: 1.25 (t, J = 7.5 Hz, 3H), 2.03 (s, 3H), 4.11 (q, J = 7.5 Hz, 2H, solvent); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -85.08 (s, 3F); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.3, 41.8, 46.2, 59.8, 62.6, 94.1 (q, $^{2}J_{C\text{-}F}$ = 33.3 Hz), 122.0 (q, $^{1}J_{C\text{-}F}$ = 285.4 Hz), 123.6, 123.9, 128.2, 131.9, 132.2, 132.3, 132.6, 147.2, 166,6, 168.6, 173.1; four crystallization solvent peaks were also observed: 14.1, 21.0, 60.5, 171.6; IR(KBr) ν_{max}: 3384.7, 3099.9, 2981.0, 1715.9, 1326.5, 1168.7, 1012.9, 786.6 cm^{-1}; MS (ESI) m/z: [(M+Na)$^{+}$, 685]; HRMS Calcd. for $C_{24}H_{19}Br_2F_3N_2O_7Na$ [M+Na]$^{+}$: 684.9409, Found: 684.9407.

Ethyl-7,11-bis(3-bromophenyl)-9-hydroxy-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undecane-10-carboxylate (4e)



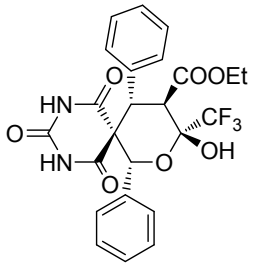
White solid; mp 271.9-272.8 °C; ^{1}H NMR (Acetone-d_6, 500 MHz): δ 0.91 (t, J = 7.0 Hz, 3H), 3.87-3.99 (m, 2H), 4.58 (d, J = 13.0 Hz , 1H), 4.60 (d, J = 13.0 Hz, 1H), 5.85 (s, 1H), 7.06 (s, 1H), 7.22-7.57 (m, 8H), 10.31 (s, 1H), 10.51 (s, 1H); ^{19}F NMR (Acetone-d_6, 470 MHz): δ -84.33 (s, 3F); ^{13}C NMR (Acetone-d_6, 100 MHz): δ 13.0, 43.2, 45.5, 59.4, 75.3, 94.7 (q, $^{2}J_{C\text{-}F}$ = 32.7 Hz), 122.1, 122.2, 122.5 (q, $^{1}J_{C\text{-}F}$ = 285.3 Hz), 125.7, 128.0, 129.6, 130.6, 130.8, 131.8, 131.9, 132.4, 137.0, 137.3, 147.4, 167.4, 168.8, 169.4; IR(KBr) ν_{max}: 3224.5, 3071.6, 2868.7, 1712.0, 1372.5, 1209.3, 1121.6, 765.2 cm^{-1}; MS [ESI(-)] m/z: [(M-H)$^{+}$, 663]; HRMS Calcd. for $C_{24}H_{19}Br_2F_3N_2O_7Na$ [M+Na]$^{+}$: 684.9409, Found: 684.9397.

Ethyl-7,11-bis(2-bromophenyl)-9-hydroxy-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undecane-10-carboxylate (4f)



White solid; mp 221.9-222.2 °C; ^{1}H NMR (Acetone-d_6, 500 MHz): δ 0.73 (t, J = 7.0 Hz, 3H), 3.73-3.90 (m, 2H), 4.54 (d, J = 12.5 Hz, 1H), 5.29 (d, J = 12.5 Hz, 1H), 6.34 (s, 1H), 6.98 (s, 1H), 7.16-7.57 (m, 8H), 10.16 (s, br, 1H), 10.39 (s, br, 1H); ^{19}F NMR (Acetone-d_6, 470 MHz): δ -84.60 (s, 3F); ^{13}C NMR (DMSO-d_6, 100 MHz): 13.5, 45.4, 57.1, 60.3, 74.0, 94.5 (q, $^{2}J_{C\text{-}F}$ = 31.7 Hz), 124.6 (q, $^{1}J_{C\text{-}F}$ = 180.7 Hz), 128.1, 130.1, 133.3, 134.0, 134.1, 134.9, 148.5, 166.5, 167.4, 169.1, 191.8; IR(KBr) ν_{max}: 3407.3, 3229.3, 2990.4, 1715.7, 1332.6, 1186.6, 1027.4, 756.44 cm^{-1};MS [ESI(-)] m/z: [(M-H)$^{+}$, 663]; HRMS Calcd. for $C_{24}H_{19}Br_2F_3N_2O_7Na$ [M+Na]$^{+}$: 684.9409, Found: 684.9395.

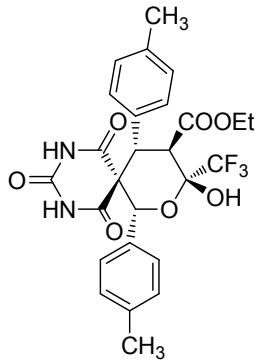
Ethyl-9-hydroxy-1,3,5-trioxo-7,11-diphenyl-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undecane-10-carboxylate (4g)



The product was recrystallized form PE-EtOAc contained one molecule of EtOAc.

White solid; mp 263.4-263.7 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 0.82 (t, J = 7.5 Hz, 3H), 3.89-3.98 (m, 2H), 4.40 (d, J = 13.0 Hz, 1H), 4.56 (d, J = 13.0 Hz,, 1H), 5.89 (s, 1H), 6.28 (s, 1H), 7.24-7.35 (m, 10H), 7.75 (s, 1H), 7.95 (s, 1H); three crystallization solvent peaks were also observed: 1.30 (t, J = 7.5 Hz, 3H), 2.08 (s, 3H), 4.16 (q, J = 7.5 Hz, 2H); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -85.14 (s, 3F); ^{13}C NMR (Acetone-d_6, 100 MHz): δ 13.7, 44.0, 46.9, 60.6, 61.6, 76.9, 95.5 (q, $^2J_{C\text{-}F}$ = 30.3 Hz), 123.5 (q, $^1J_{C\text{-}F}$ = 255.5 Hz), 127.6, 129.3, 129.4, 129.5, 129.8, 130.0, 135.6, 135.7, 148.4, 168.5, 170.4, 170.6,; four crystallization solvent peaks were also observed: 14.4, 20.7, 60.5, 170.9; IR(KBr) ν_{max}: 3225.9, 3111.3, 2882.3, 1701.9, 1340.1, 1206.1, 1122.6, 739.8 cm^{-1}; MS (ESI) m/z: [(M+Na)$^+$, 529]; HRMS Calcd. for $C_{24}H_{21}F_3N_2O_7Na$ [M+Na]$^+$: 529.1199, Found: 529.1194.

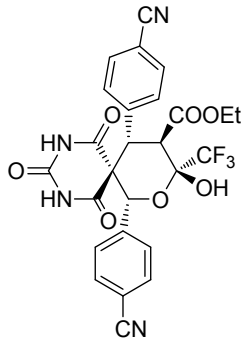
Ethyl-9-hydroxy-1,3,5-trioxo-7,11-di-p-tolyl-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undecane-10-carboxylate (4h)



The product was recrystallized form PE-EtOAc contained one molecule of EtOAc.

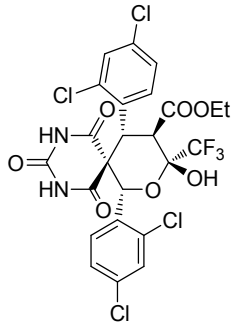
White solid; mp 247.2-248.0 °C;^{1}H NMR (Acetone-d_6, 500 MHz): δ 0.80 (t, J = 7.0 Hz, 3H), 2.16 (s, 3H), 2.20 (s, 3H), 3.78-3.83 (m,2H), 4.43 (d, J = 13.0 Hz 1H), 4.51 (d, J = 13.0 Hz, 1H), 5.72 (s, 1H), 6.74 (s, 1H), 7.02-7.08 (m, 8H), 10.04 (s, br, 2H); three crystallization solvent peaks were also observed: 1.12 (t, J = 7.0 Hz, 3H), 1.89 (s, 3H), 3.98 (q, J = 7.0 Hz, 2H); ^{19}F NMR (Acetone-d_6, 470 MHz): δ -84.49 (s, 3F); ^{13}C NMR (DMSO-d_6, 100 MHz): 13.9, 21.2, 44.0, 45.0, 60.0, 60.2, 75.6, 94.8 (q, $^2J_{C\text{-}F}$ = 31.8 Hz), 123.0 (q, $^1J_{C\text{-}F}$ = 342.5 Hz), 126.9, 128.7, 129.4, 129.6, 130.0, 130.2, 132.3, 132.7, 134.4, 137.9, 138.8, 149.1, 150.3, 168.3, 168.5, 170.9; four crystallization solvent peaks were also observed: 14.5, 21.0, 60.5, 170.8; IR(KBr) ν_{max}: 3379.0, 3202.9, 3095.2, 1709.2, 1327.1, 1208.2, 1110.7, 779.6 cm^{-1}; MS (ESI) m/z: [(M+Na)$^+$, 557]; HRMS Calcd. for $C_{26}H_{25}F_3N_2O_7Na$ [M+Na]$^+$: 557.1512, Found: 557.1496.

Ethyl-7,11-bis(4-cyanophenyl)-9-hydroxy-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undecane-10-carboxylate (4i)



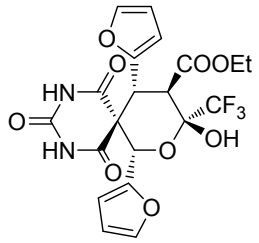
White solid; mp 263.9-264.1 °C; ^{1}H NMR (Acetone-d_6, 500 MHz): δ 0.79 (t, J = 7.2 Hz, 3H), 3.77-3.81 (m, 2H), 4.50 (d, J = 12.5 Hz, 1H), 4.60 (d, J = 12.5 Hz, 1H), 5.83 (s, 1H), 7.06 (s, 1H), 7.29-7.71 (m, 8H), 10.14 (s, br, 2H); ^{19}F NMR (Acetone-d_6, 470 MHz): δ -84.19 (s, 3F); ^{13}C NMR (Acetone-d_6, 100 MHz): 13.0, 43.3, 45.9, 59.1, 61.1, 75.5, 94.8 (q, $^{2}J_{C\text{-}F}$ = 32.6 Hz), 112.6, 113.2, 117.8, 122.5 (q, $^{1}J_{C\text{-}F}$ = 353.7 Hz), 127.8, 130.1, 132.6, 132.7, 139.5, 140.0, 147.1, 167.1, 168.4, 169.1; IR(KBr) ν_{max}: 3241.7, 3121.1, 2239.8, 1701.6, 1333.5, 1178.9, 1103.8, 771.6 cm^{-1}; MS [ESI(-)] m/z: [(M-H)$^{+}$,555]; HRMS Calcd. for $C_{26}H_{19}F_3N_4O_7Na$ [M+Na]$^{+}$: 579.1104, Found: 579.1098.

Ethyl-7,11-bis(2,4-dichlorophenyl)-9-hydroxy-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undecane-10-carboxylate (4j)



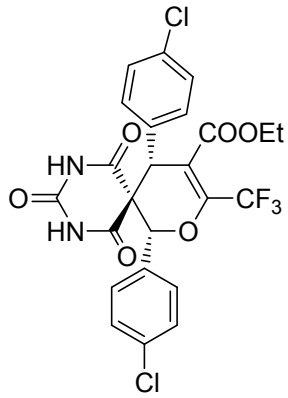
White solid; mp 178.6-180 °C; ^{1}H NMR (Acetone-d_6, 500 MHz): δ 0.87 (t, J = 7.0 Hz, 3H), 3.83-3.89 (m, 1H), 3.92-3.98 (m, 1H), 4.57 (d, J = 12.5 Hz, 1H), 5.38 (d, J = 12.5 Hz, 1H), 6.35 (s, 1H), 7.20 (s, 1H), 7.39-7.60 (m, 6H), 10.39 (d, 2H); ^{19}F NMR (Acetone-d_6, 470 MHz): δ -84.39 (s, 3F); ^{13}C NMR (Acetone-d_6, 100 MHz): δ 13.7, 41.2, 45.5, 58.2, 62.0, 72.8, 95.5 (q, $^{2}J_{C\text{-}F}$ = 32.7 Hz), 123.2 (q, $^{1}J_{C\text{-}F}$ = 285.5 Hz), 128.5, 128.6, 130.0, 130.8, 131.7, 132.0, 132.1, 132.8, 134.4, 135.4, 136.5, 137.4, 148.3, 166.9, 169.2, 169.8; IR(KBr) ν_{max}: 3390.9, 3251.8, 2978.1, 1713.2, 1330.9, 1185.4, 1105.8, 777.4 cm^{-1}; MS [ESI(-)] m/z: [(M-H)$^{+}$, 643]; HRMS Calcd. for $C_{24}H_{17}Cl_4F_3N_2O_7Na$ [M+ Na]$^{+}$: 664.9640, Found: 664.9641.

Ethyl-7,11-di(furan-2-yl)-9-hydroxy-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undecane-10-carboxylate (4k)



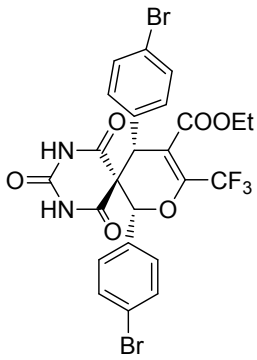
Yellow solid; mp 221.1-221.8 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.05 (t, J = 7.0 Hz, 3H), 4.05 (q, J = 7.0 Hz, 2H), 4.32 (d, J = 12.5 Hz, 1H), 4.40 (d, J = 12.5 Hz, 1H), 5.83 (s, 1H), 6.15 (s, 1H), 6.22-6.42 (m, 4H), 7.32 (s, 2H), 7.62 (s, br, 1H), 7.72 (s, br, 1H); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -84.96 (s, 3F); ^{13}C NMR (DMSO-d_6, 100 MHz): 14.1, 43.4, 55.7, 61.0, 69.8, 94.5 (q, $^{2}J_{C\text{-}F}$ = 32.3 Hz), 108.6, 110.3, 111.1, 111.3, 118.4 (q, $^{1}J_{C\text{-}F}$ = 229.1 Hz), 143.7, 144.4, 148.2, 149.5, 150.1, 167.9, 168.2, 170.1; IR(KBr) ν_{max}: 3358.2, 3238.9, 3129.4, 2857.7, 1712.9, 1373.2, 1014.8, 738.8 cm^{-1}; MS (ESI) m/z: [(M+Na)$^{+}$, 509]; HRMS Calcd. for $C_{20}H_{17}F_3N_2O_9Na$ [M+Na]$^{+}$: 509.0784, Found: 509.0773.

Ethyl-7,11-bis(4-chlorophenyl)-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undec-9-ene-10-carboxylate (5a)



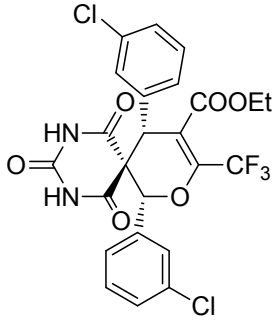
White solid; mp 205.6-206.7 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.29 (t, J = 7.0 Hz, 3H), 4.70 (d, J = 5.0 Hz, 1H), 4.19-4.34 (m, 2H), 4.55 (d, J = 5.0 Hz, 1H), 6.04 (s, 1H), 7.06-7.32 (m, 8H), 7.79 (s, 1H); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -79.88 (s, 3F); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.6, 46.9, 50.8, 53.9, 63.4, 80.8, 99.8 (q, $^{2}J_{C\text{-}F}$ = 36.0 Hz), 119.2 (q, $^{1}J_{C\text{-}F}$ = 281.9 Hz), 128.0, 129.3, 129.8, 131.1, 132.3, 135.7, 136.4, 152.9, 164.8, 167.3, 174.9; IR(KBr) ν_{max}: 3205.7, 3096.2, 2994.0, 1709.3, 1375.7, 1145.2, 1040.0, 768.0 cm^{-1}; MS (ESI) m/z: [(M+H)$^{+}$, 557]; HRMS Calcd. for $C_{24}H_{17}C_{l2}F_3N_2O_6Na$ [M+Na]$^{+}$: 579.0313, Found: 579.0314.

Ethyl-7,11-bis(4-bromophenyl)-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undec-9-ene-10-carboxylate (5b)



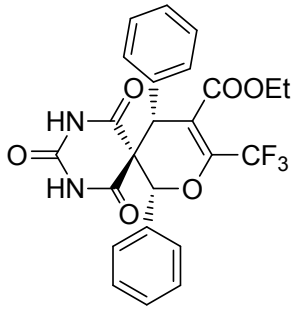
White solid; mp 214.9-216.1 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.30 (t, J = 7.0 Hz, 3H), 3.71 (d, J = 5.0 Hz, 1H), 4.20-4.36 (m, 2H), 4.55 (d, J = 5.0 Hz, 1H), 6.04 (s, 1H), 7.01-7.50 (m, 8H), 7.80 (s, 1H); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -79.86 (s, 3F); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.6, 47.1, 50.8, 53.8, 63.4, 81.0, 99.9 (q, $^{2}J_{C\text{-}F}$ = 35.9 Hz), 119.3 (q, $^{1}J_{C\text{-}F}$ = 281.7 Hz), 124.1, 124.8, 128.3, 129.6, 131.6, 132.4, 132.9, 152.8, 164.5, 167.3, 174.9; IR(KBr) ν_{max}: 3345.2, 3097.6, 2996.4, 1713.2, 1371.3, 1254.2, 1145.5, 767.9 cm^{-1}; MS (ESI) m/z: [(M+H)$^{+}$, 645]; HRMS Calcd. for $C_{24}H_{17}Br_2F_3N_2O_6Na$ [M+Na]$^{+}$: 666.9303, Found: 666.9289.

Ethyl-7,11-bis(3-chlorophenyl)-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undec-9-ene-10-carboxylate (5c)



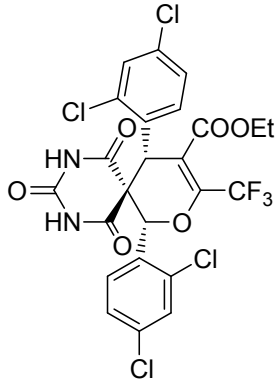
White solid; mp 175.3-175.8 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.32 (t, J = 7.0 Hz, 3H), 3.75 (s, J = 5.0 Hz, 3H), 4.22-4.37 (m, 2H), 4.56 (d, J = 4.5 Hz, 1H), 6.05 (s, 1H), 7.02-7.37 (m, 8H), 7.97 (s, 1H); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -79.80 (s, 3F); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 13.5, 47.0, 50.7, 53.6, 63.4, 80.6, 99.7 (q, $^{2}J_{C\text{-}F}$ = 36.1 Hz), 119.2 (q, $^{1}J_{C\text{-}F}$ = 281.8 Hz), 124.7, 125.6, 126.8, 128.6, 129.8, 130.3, 130.5, 134.5, 134.9, 135.3, 135.7, 152.9, 164.7, 167.1, 174.6; IR(KBr) ν_{max}: 3206.4, 3111.8, 2987.6, 1715.6, 1373.4, 1251.9, 1146.2, 785.2 cm^{-1}; MS (ESI) m/z: [(M+H)$^{+}$, 557]; HRMS Calcd. for $C_{24}H_{17}Cl_2F_3N_2O_6Na$ [M+Na]$^{+}$: 579.0313, Found: 579.0315.

Ethyl-1,3,5-trioxo-7,11-diphenyl-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undec-9-ene-10-carboxylate (5d)



White solid; mp 217.0-217.6 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.30 (t, J = 7.0 Hz, 3H), 3.80 (d, J = 5.0 Hz, 1H), 4.20-4.35 (m, 2H), 4.60 (d, J = 5.0 Hz, 1H), 6.08 (s, 1H), 7.13-7.36 (m, 10H), 7.84 (s, 1H); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -79.91 (s, 3F); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.7, 47.7, 51.0, 54.3, 63.2, 81.7, 100.0 (q, $^{2}J_{C\text{-}F}$ = 35.8 Hz), 119.5 (q, $^{1}J_{C\text{-}F}$ = 281.8 Hz), 126.8, 128.1, 129.0, 129.2, 129.5, 129.6, 130.2, 132.9, 134.1, 153.4, 165.2, 167.6, 175.5; IR(KBr) ν_{max}: 3200.5, 3106.5, 2982.6, 1712.2, 1375.1, 1222.0, 1043.7, 757.3 cm^{-1}; MS (ESI) m/z: [(M+H)$^{+}$, 489]; HRMS Calcd. for $C_{24}H_{19}F_3N_2O_6Na$ [M+Na]$^{+}$: 511.1093, Found: 511.1089.

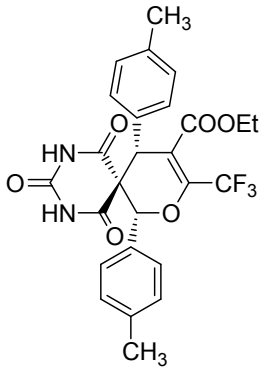
Ethyl-7,11-bis(2,4-dichlorophenyl)-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undec-9-ene-10-carboxylate (5e)



The product was recrystallized form PE-EtOAc contained one molecule of EtOAc.

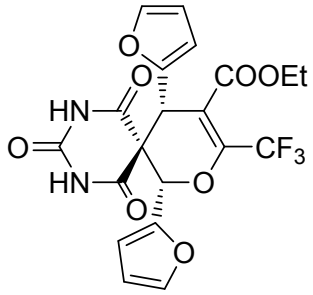
White solid; mp 181.2-183.3 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.23 (t, J = 7.0 Hz, 3H), 3.51 (s, 1H), 4.26-4.38 (m, 2H), 5.41 (s, 1H), 6.69 (s, 1H), 7.06-7.43 (m, 6H), 8.41 (s, 1H); three crystallization solvent peaks were also observed: 1.32 (t, J = 7.5 Hz, 3H), 2.00 (s, 3H), 4.08 (q, J = 7.5 Hz, 2H); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -79.92 (s, 3F); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.6, 42.4, 51.8, 63.4, 99.8 (q, $^{2}J_{C\text{-}F}$ = 36.0 Hz), 119.1 (q, $^{1}J_{C\text{-}F}$ = 282.2 Hz), 127.8, 128.3, 128.7, 128.8, 128.9, 130.3, 130.6, 131.4, 134.8, 135.7, 136.0, 137.2, 153.0, 162.5, 166.6, 175.1; four crystallization solvent peaks were also observed: 14.0, 20.9, 60.4, 171.3; IR(KBr) ν_{max}: 3453.7, 3039.9, 2904.9, 1724.9, 1374.4, 1248.7, 1104.2, 771.2 cm^{-1}; MS (ESI) m/z: [(M+H)$^{+}$, 625]; HRMS Calcd. for $C_{24}H_{15}Cl_4F_3N_2O_6Na$ [M+Na]$^{+}$: 646.9534, Found: 646.9536.

Ethyl-1,3,5-trioxo-7,11-di-p-tolyl-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undec-9-ene-10-carboxylate (5f)



White solid; mp 198.9-199.3 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.28 (t, J = 7 Hz, 3H), 2.28 (s, 3H), 2.29 (s, 3H),3.76 (d, J = 5.0 Hz, 1H), 4.18-4.34 (m, 2H), 4.54 (d, J = 5.0 Hz, 1H), 6.02 (s, 1H), 7.01-7.15 (m, 8H), 7.72 (s, 1H); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -79.97 (s, 3F); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.5, 20.9, 21.0, 47.2, 51.0, 54.4, 63.0, 81.5, 99.9 (q, $^{2}J_{C\text{-}F}$ = 35.7 Hz), 119.4 (q, $^{1}J_{C\text{-}F}$ = 281.8 Hz), 126.5, 127.8, 129.6, 129.9, 130.1, 131.1, 139.3, 140.1, 153.4, 165.0, 167.5, 175.8; IR(KBr) ν_{max}: 3205.2, 3106.5, 2988.1, 1711.4, 1374.7, 1253.3, 1147.2, 769.7 cm^{-1}; MS (ESI) m/z: [(M+H)$^{+}$, 517]; HRMS Calcd. for $C_{26}H_{23}F_3N_2O_6Na$ [M+Na]$^{+}$: 539.1406, Found: 539.1392.

Ethyl-7,11-di(furan-2-yl)-1,3,5-trioxo-9-(trifluoromethyl)-8-oxa-2,4-diazaspiro[5.5]undec-9-ene-10-carboxylate (5g)

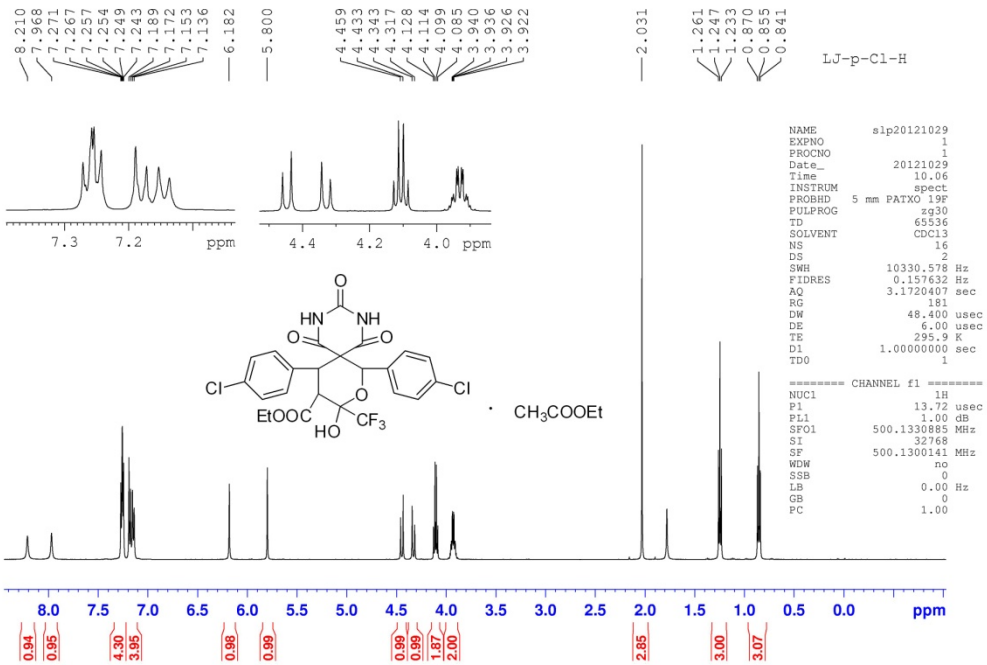


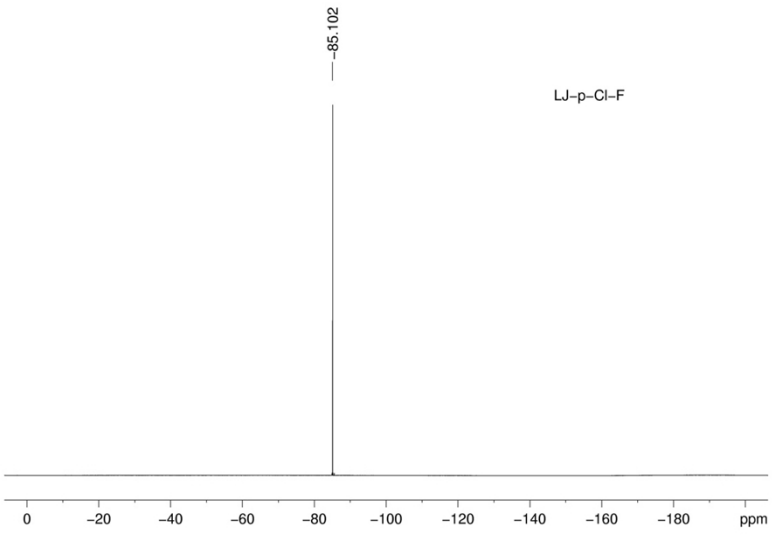
The product was recrystallized form PE-EtOAc contained one molecule of EtOAc.

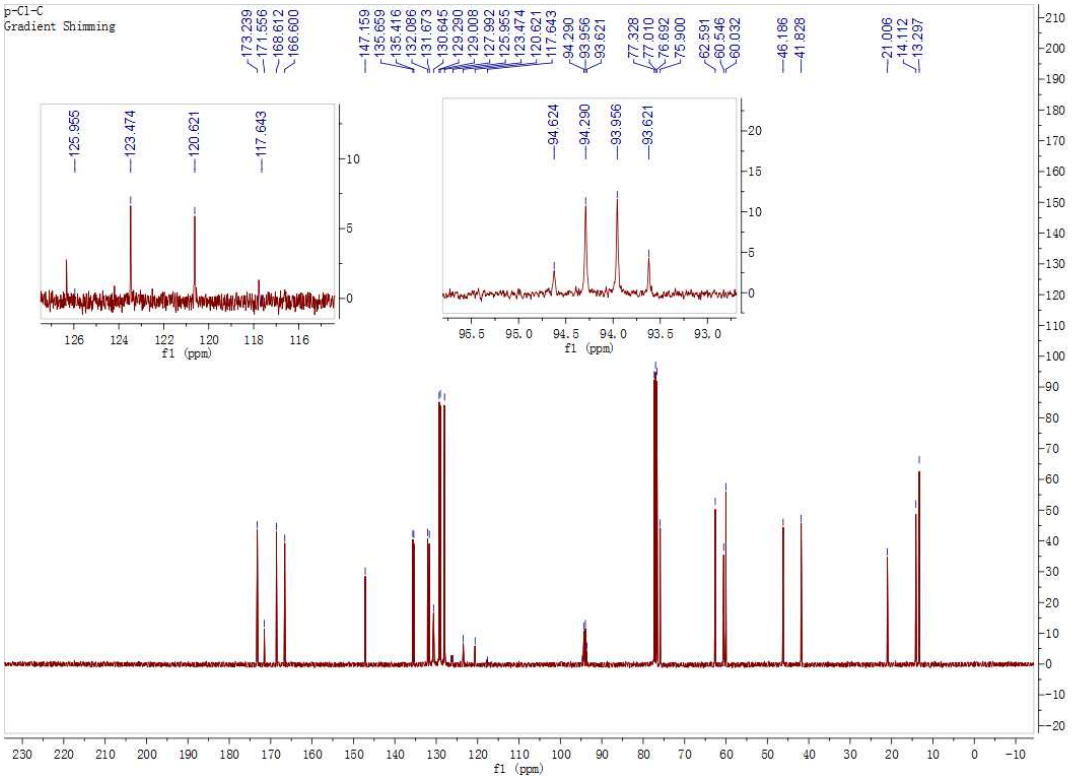
White solid; mp 199.4-200.0 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.23 (t, J = 7.0 Hz, 3H), 3.82 (d, J = 5.0 Hz, 1H), 4.21-4.34 (m, 2H), 4.62 (d, J = 5.0 Hz, 1H), 5.97 (s, 1H), 6.28-7.36 (m, 6H), 8.08 (s, 1H); three crystallization solvent peaks were also observed: 1.30 (t, J= 7.0 Hz, 3H), 2.02 (s, 3H), 4.10 (q, J = 7.0 Hz, 2H), ^{19}F NMR ($CDCl_3$, 470 MHz): δ -79.96 (s, 3F); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.6, 41.4, 48.8, 49.7, 63.3, 99.4 (q, $^{2}J_{C\text{-}F}$ = 35.8 Hz), 110.6, 110.9, 111.0, 112.7, 119.1 (q, $^{1}J_{C\text{-}F}$ = 282.1 Hz), 144.4, 145.0, 145.6, 146.4, 153.9, 164.6, 167.2, 175.7; four crystallization solvent peaks were also observed: 14.1, 20.9, 60.4, 171.2; IR(KBr) ν_{max}: 3347.0, 3155.7, 2994.8, 1719.9, 1372.2, 1247.5, 1147.7, 768.8 cm^{-1}; MS (ESI) m/z: [(M+H)$^{+}$, 469]; HRMS Calcd. for $C_{20}H_{15}F_3N_2O_8Na$ [M+Na]$^{+}$: 491.0678, Found: 491.0668.

^{1}H, ^{19}F and ^{13}C NMR Spectra, Mass spectra and HRMS spectra of compounds **4** and **5** in sequence

4a







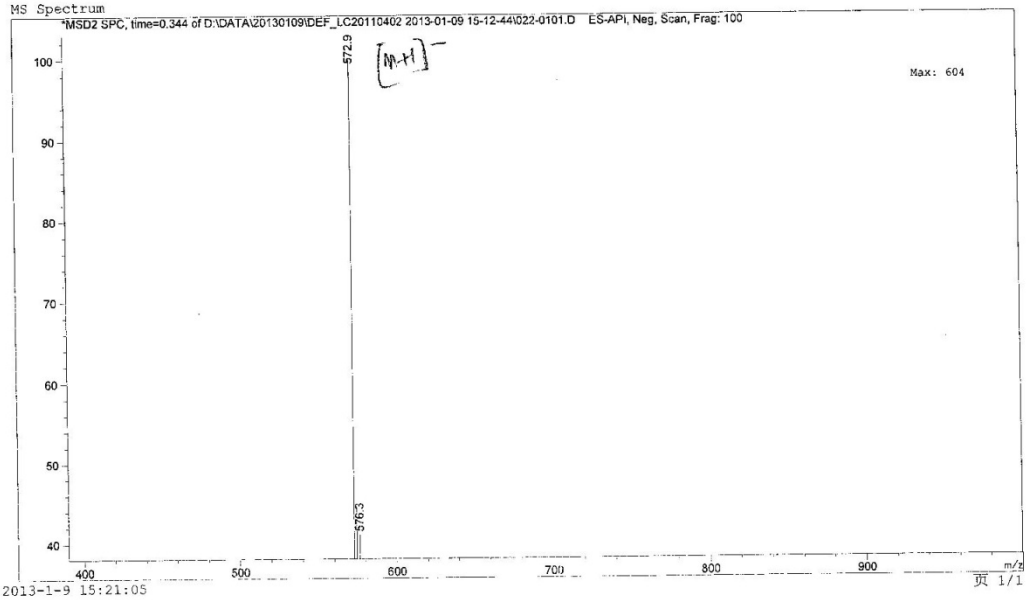
打印窗口 80: MS Spectrum
数据文件: : D:\DATA\20130109\DEF_LC20110402 2013-01-09 15-12-44\022-0101.D
样品名称 : 0104-4

P-Cl
4J-1

操作者 :
仪器 : 仪器 1
进样日期 : 2013-1-9 15:13:37

序列行 : 1
位置 : 样品瓶 22
进样次数 : 1
进样量 : 0.1 µl

采集方法 : D:\DATA\20130109\DEF_LC20110402 2013-01-09 15-12-44\DEF_LC20110310.M
最后修改 : 2013-1-9 15:12:42
分析方法 : C:\CHEM32\1\METHODS\DEF_LC20110310.M
最后修改 : 2013-1-9 15:20:42
(调用后修改)



仪器 1 2013-1-9 15:21:05

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1297

Sample Serial Number: LJ-1

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 597.0414 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
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H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	7
F	18.998403	1	3
Cl	34.968853	1	2
Na	22.989770	1	1

Additional Search Restrictions:

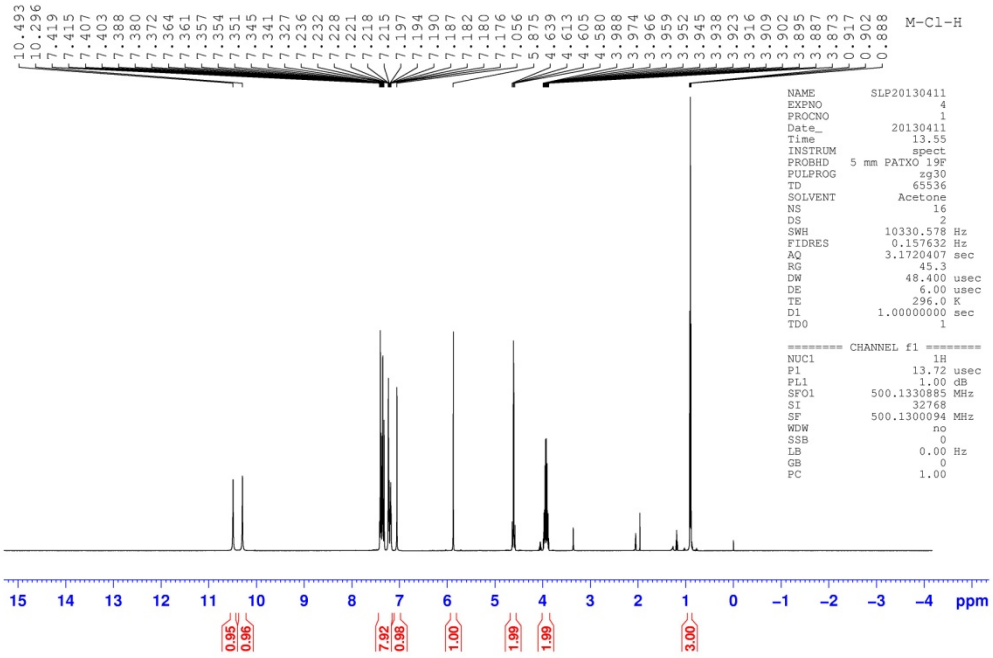
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

Search Results:

Number of Hits = 7

m/z	Delta m/z	DBE	Formula
597.04136	0.00004	16.5	$C_{24}H_{19}N_2O_7F_3Cl_2Na^{+1}$
597.04127	0.00013	32.0	$C_{36}H_{12}N_2O_3FClNa^{+1}$
597.04241	-0.00101	28.5	$C_{33}H_{13}N_2O_4F_2ClNa^{+1}$
597.04022	0.00118	20.0	$C_{27}H_{18}N_2O_6F_2Cl_2Na^{+1}$
597.03973	0.00167	24.0	$C_{30}H_{15}NO_7F_2ClNa^{+1}$
597.04310	-0.00170	27.5	$C_{35}H_{17}O_3FCl_2Na^{+1}$
597.03951	0.00189	29.5	$C_{36}H_{15}F_3Cl_2Na^{+1}$

4b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NAME SLP20130411
EXPNO 4
PROCNO 1
Date_ 20130411
Time 13.55
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT Acetone
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DS 2
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FIDRES 0.157632 Hz
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RG 45.3
DW 48.400 usec
DE 6.00 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
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P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300094 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3 −4 ppm
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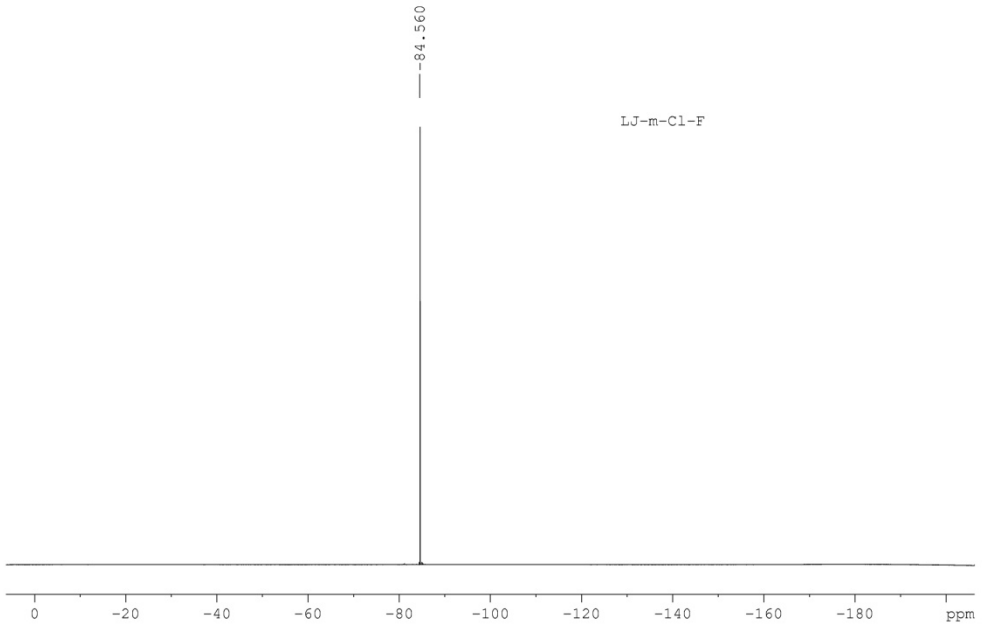
0.96
7.92
0.98
1.00
1.99
1.99
3.00


−84.560
LJ-m-Cl-F
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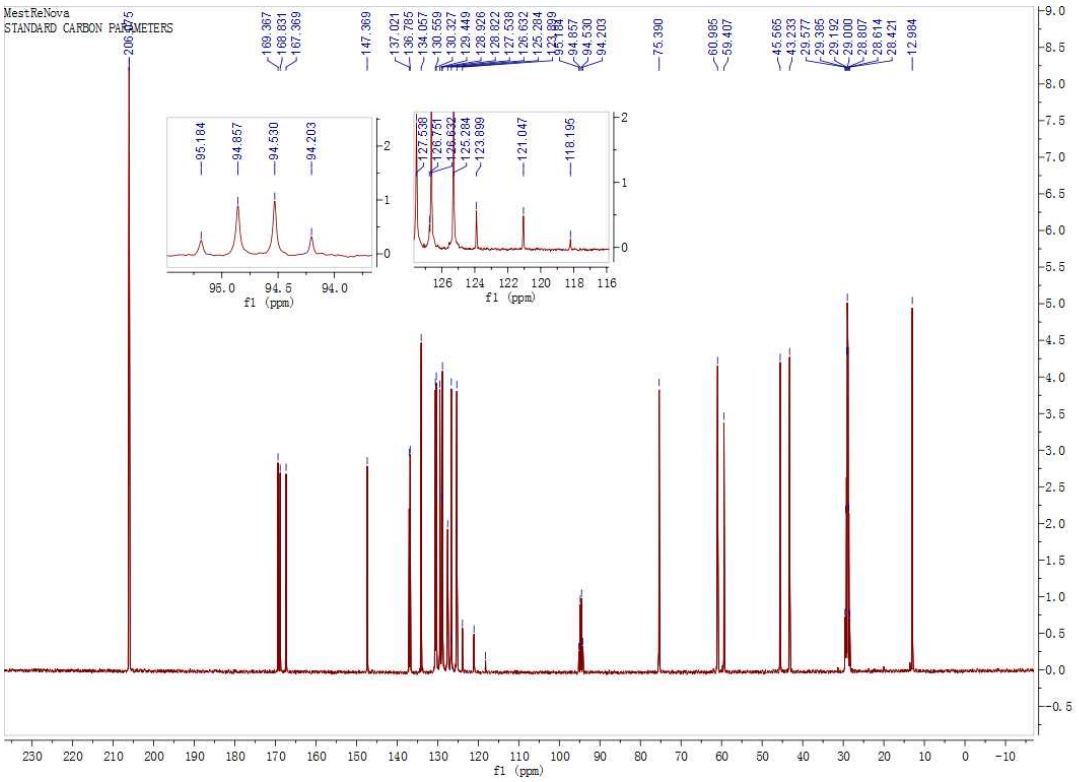

Print of window 80: MS Spectrum

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Inj : 1
Inj Volume : 0.3 µl

Acq. Method : C:\HPCHEM\1\METHODS\AGILENT1.M
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(modified after loading)
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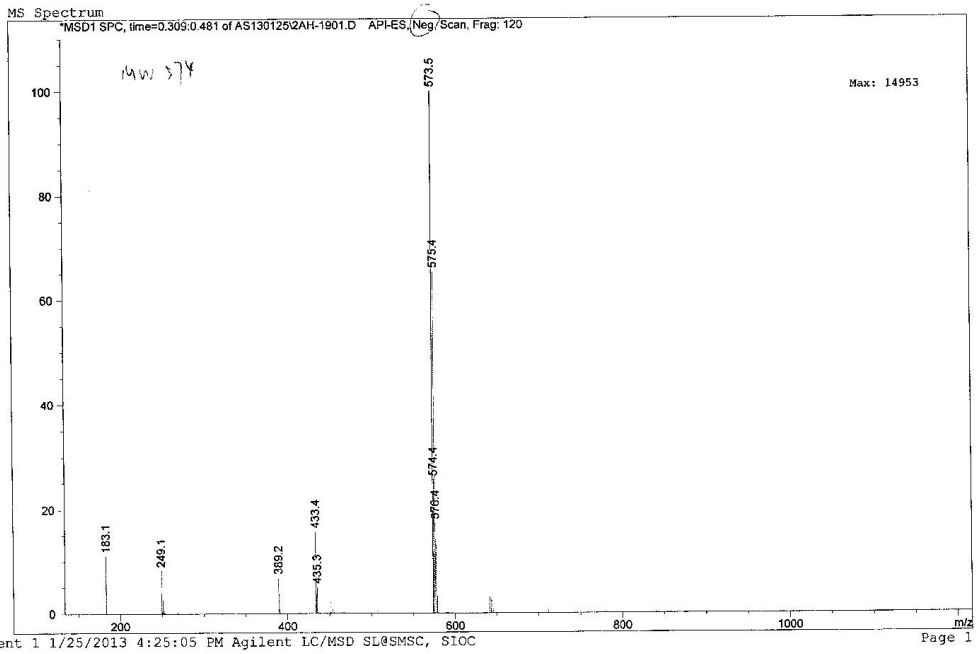
Test

m-cl

LJ-2

MS Spectrum



Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1298

Sample Serial Number: LJ-2

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 597.0416 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	7
F	18.998403	1	3
Cl	34.968853	1	2
Na	22.989770	1	1

Additional Search Restrictions:

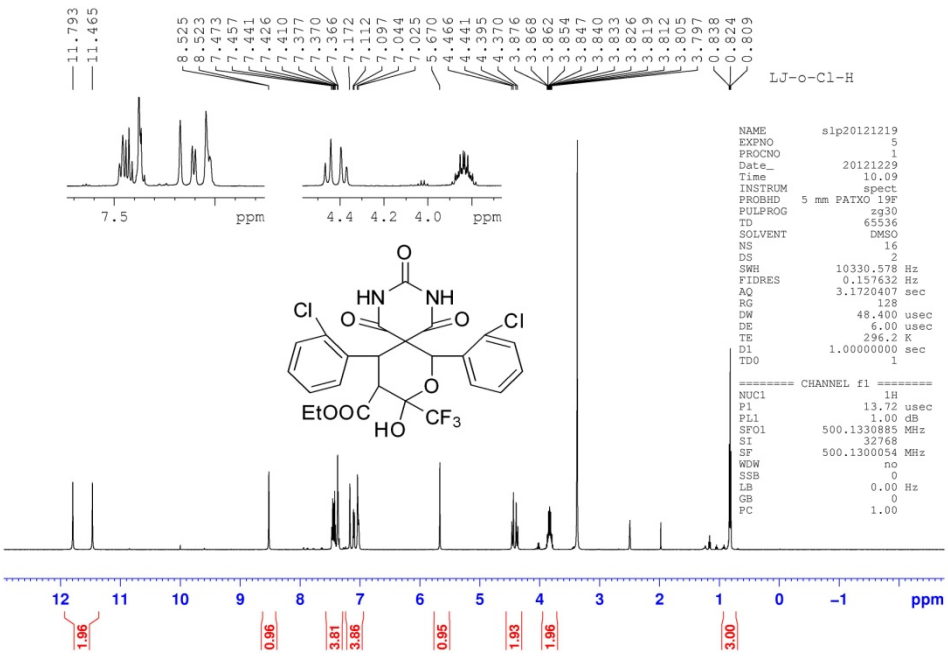
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

Search Results:

Number of Hits = 7

m/z	Delta m/z	DBE	Formula
597.04136	0.00024	16.5	$C_{24}H_{19}N_2O_7F_3Cl_2Na^{+1}$
597.04127	0.00033	32.0	$C_{36}H_{12}N_2O_3FClNa^{+1}$
597.04241	-0.00081	28.5	$C_{33}H_{13}N_2O_4F_2ClNa^{+1}$
597.04022	0.00138	20.0	$C_{27}H_{18}N_2O_6F_2Cl_2Na^{+1}$
597.04310	-0.00150	27.5	$C_{35}H_{17}O_3FCl_2Na^{+1}$
597.03973	0.00187	24.0	$C_{30}H_{15}NO_7F_2ClNa^{+1}$
597.04356	-0.00196	25.0	$C_{30}H_{14}N_2O_5F_3ClNa^{+1}$

4c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PROBHD 5 mm PATXO 19F
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NS 16
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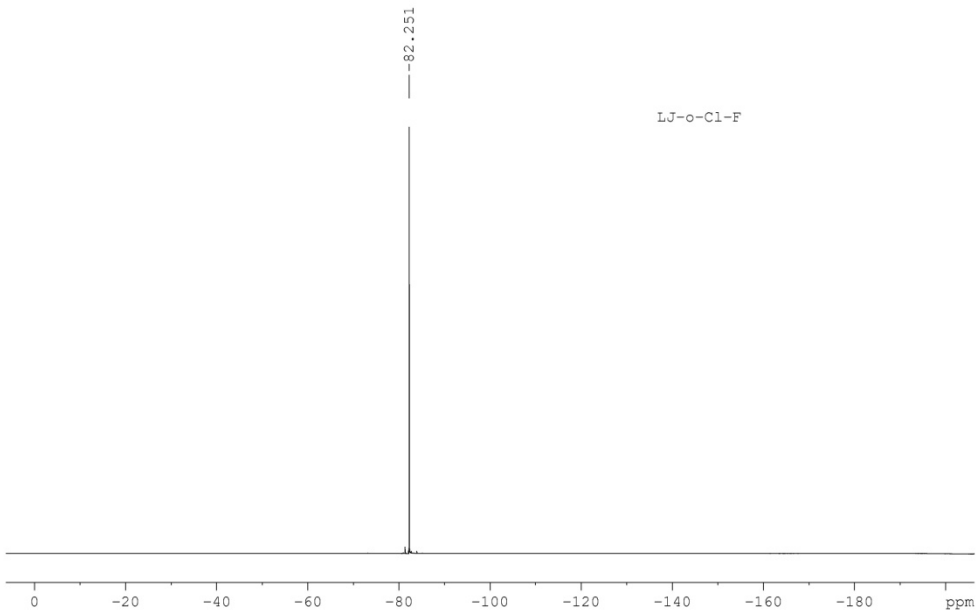
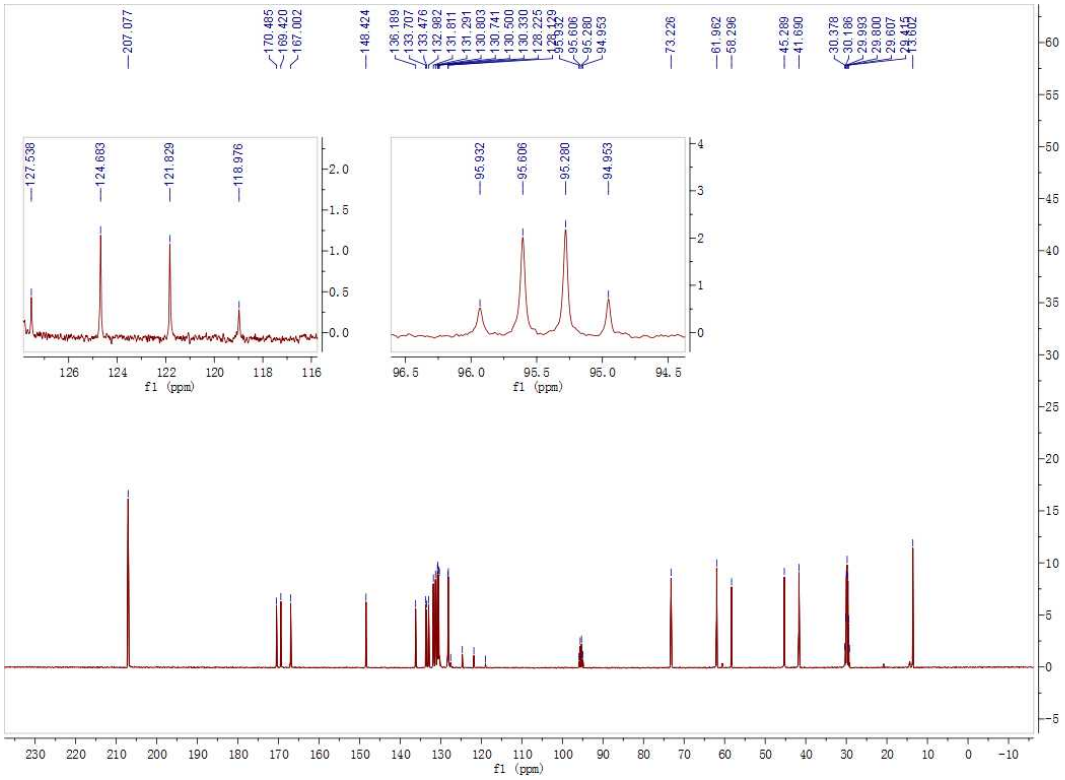
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300054 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
12 11 10 9 8 7 6 5 4 3 2 1 0 −1 ppm
1.96
0.96
3.81
3.86
0.95
1.93
1.96
3.00

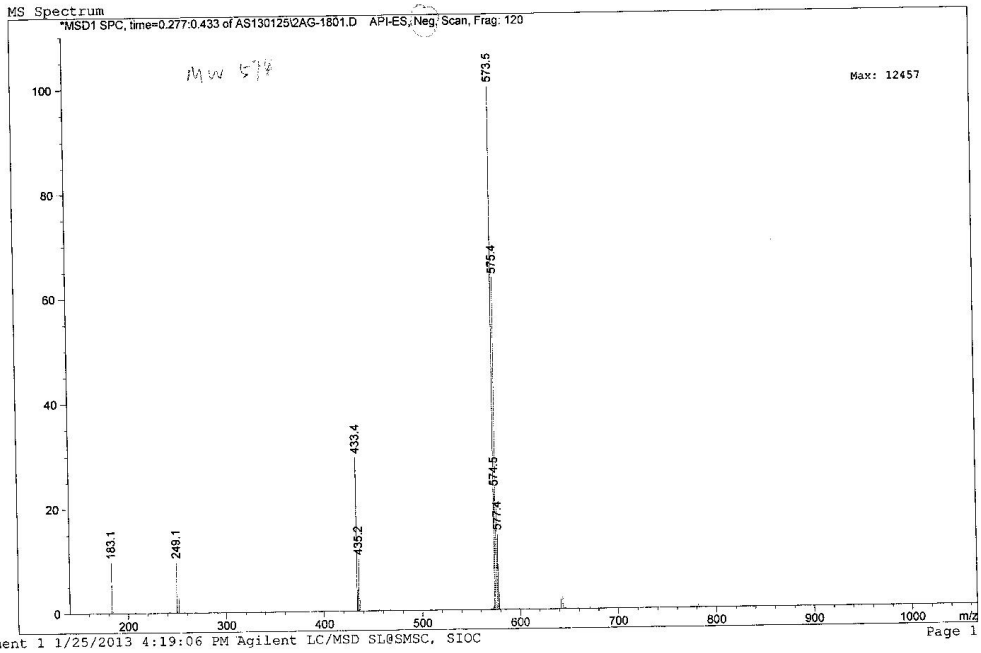

-82.251
LJ-o-Cl-F
0 -20 -40 -60 -80 -100 -120 -140 -160 -180 ppm



Injection Date : 1/25/2013 3:56:00 PM
Sample Name : LJ-4
Acq. Operator : Agilent LC/MSD SL@SMSC, SIOC

Seq. Line : 10
Location : P2-A-07
Inj : 1
Inj Volume : 0.1 µl

Acq. Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 1/25/2013 3:56:22 PM by Agilent LC/MSD SL@SMSC, SIOC
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\AANAL3'.M
Last changed : 1/25/2013 3:59:53 PM by Agilent LC/MSD SL@SMSC, SIOC
Test



Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1299

Sample Serial Number: LJ-3

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 597.0428 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	7
F	18.998403	1	3
Cl	34.968853	1	2
Na	22.989770	1	1

Additional Search Restrictions:

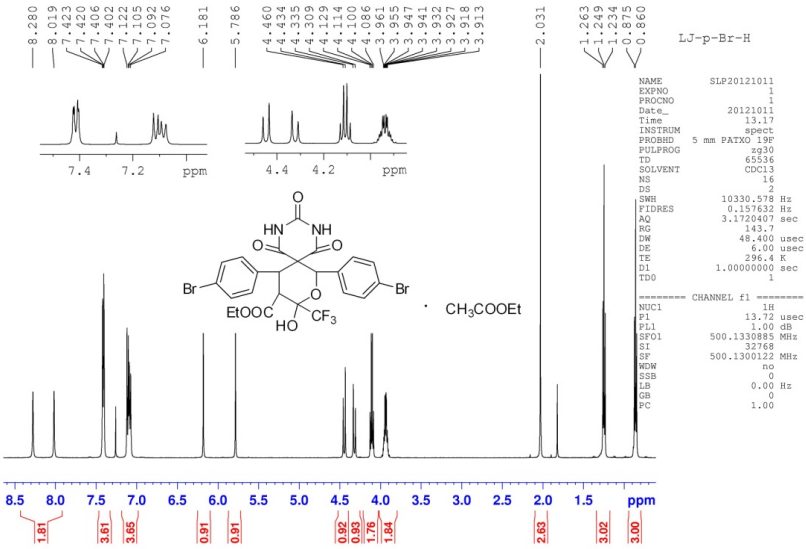
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

Search Results:

Number of Hits = 6

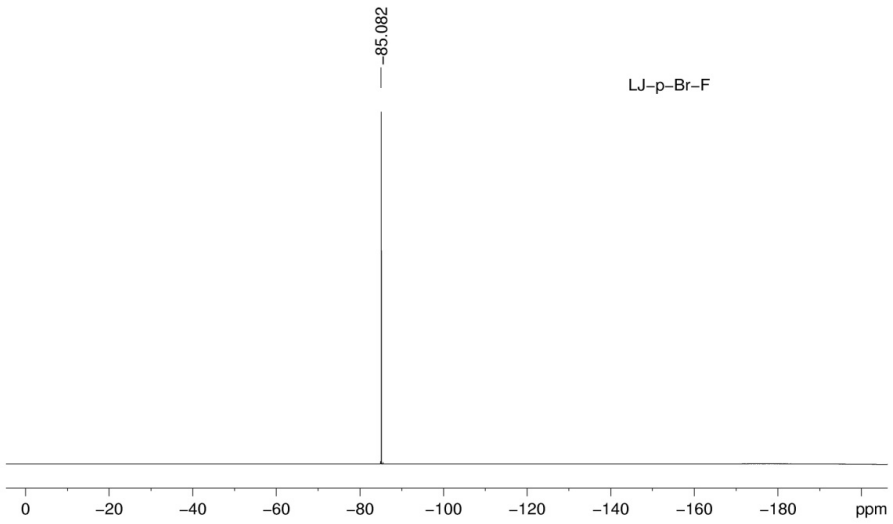
m/z	Delta m/z	DBE	Formula
597.04310	-0.00030	27.5	$C_{35}H_{17}O_3FCl_2Na^{+1}$
597.04241	0.00039	28.5	$C_{33}H_{13}N_2O_4F_2ClNa^{+1}$
597.04356	-0.00076	25.0	$C_{30}H_{14}N_2O_5F_3ClNa^{+1}$
597.04136	0.00144	16.5	$C_{24}H_{19}N_2O_7F_3Cl_2Na^{+1}$
597.04424	-0.00144	24.0	$C_{32}H_{18}O_4F_2Cl_2Na^{+1}$
597.04127	0.00153	32.0	$C_{36}H_{12}N_2O_3FClNa^{+1}$

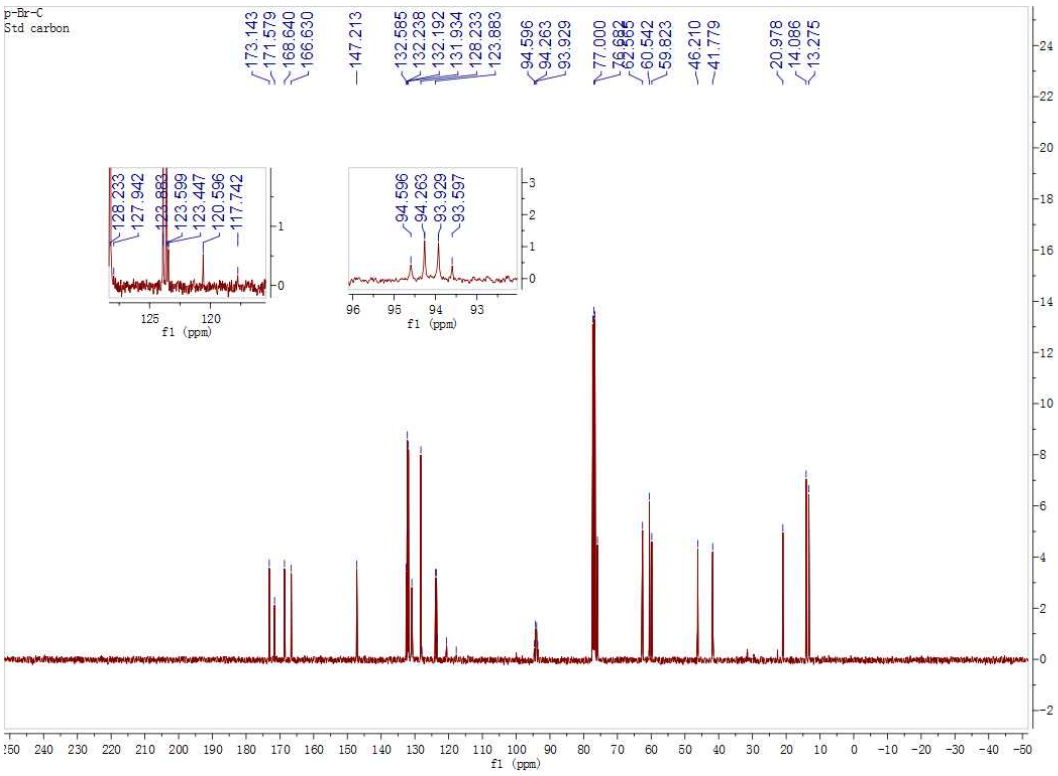
4d



NAME SLP20121011
EXPNO 1
PROCNO 1
Date_ 20121011
Time 13.17
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 143.7
DW 48.400 usec
DE 6.00 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300122 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00





Print of Window 80: MS Spectrum
Data File : D:\DATA\CHEMDATA\AT20130114\DEF_LC 2013-01-14 16-47-36\1HD-0101.D
Sample Name : 0104-3

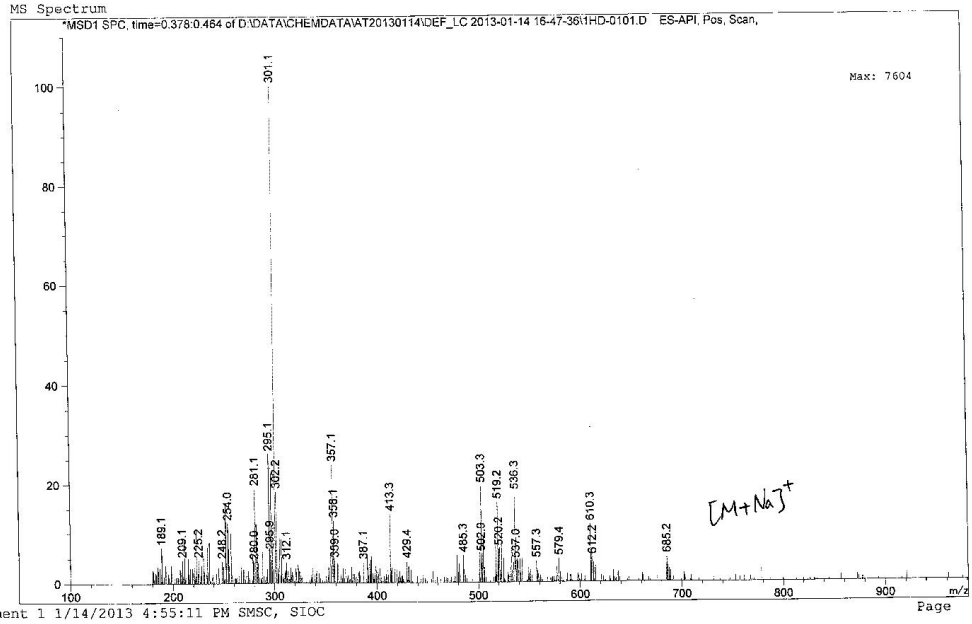
Acq. Operator : SMSC, SIOC
Acq. Instrument : Instrument 1
Injection Date : 1/14/2013 4:48:35 PM
Seq. Line : 1
Location : P1-H-04
Inj : 1
Inj Volume : 1.000 µl

Acq. Method : D:\DATA\CHEMDATA\AT20130114\DEF_LC 2013-01-14 16-47-36\AANAL1.M
Last changed : 1/14/2013 4:47:36 PM by SMSC, SIOC
Analysis Method : C:\CHEM32\1\METHODS\AANAL-NEG.M
Last changed : 1/14/2013 4:54:16 PM by SMSC, SIOC
(modified after loading)

p-Br

LJ-4

MS Spectrum



Instrument 1 1/14/2013 4:55:11 PM SMSC, SIOC

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1300

Sample Serial Number: LJ-4

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 684.9407 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	7
F	18.998403	1	3
Br	78.918338	1	2
Na	22.989770	1	1

Additional Search Restrictions:

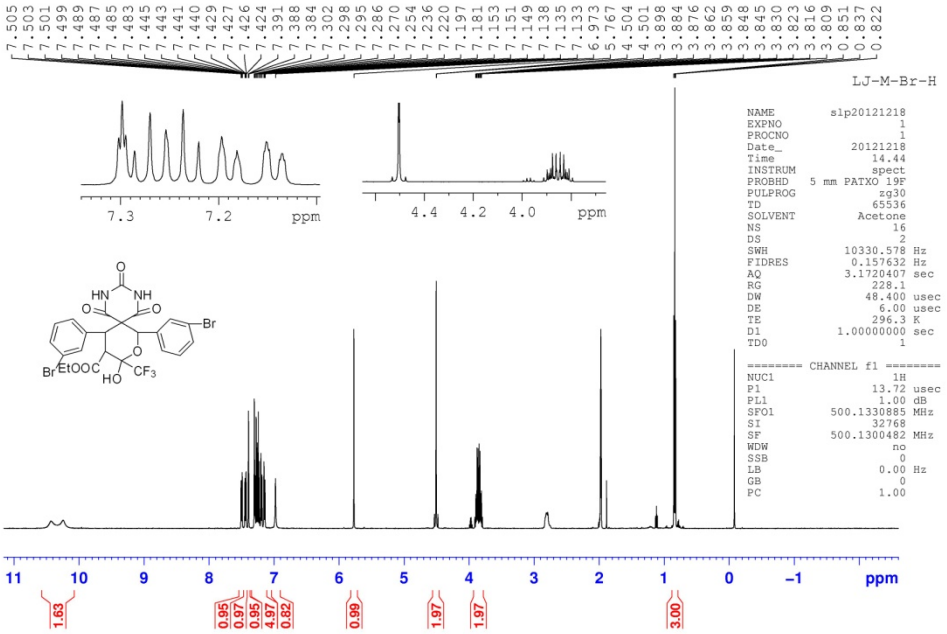
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

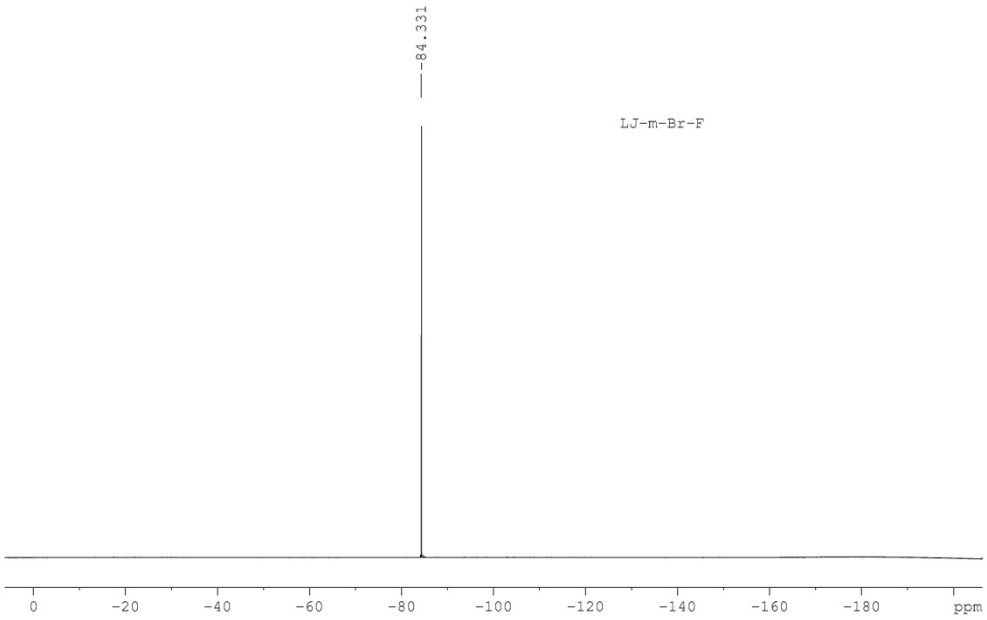
Search Results:

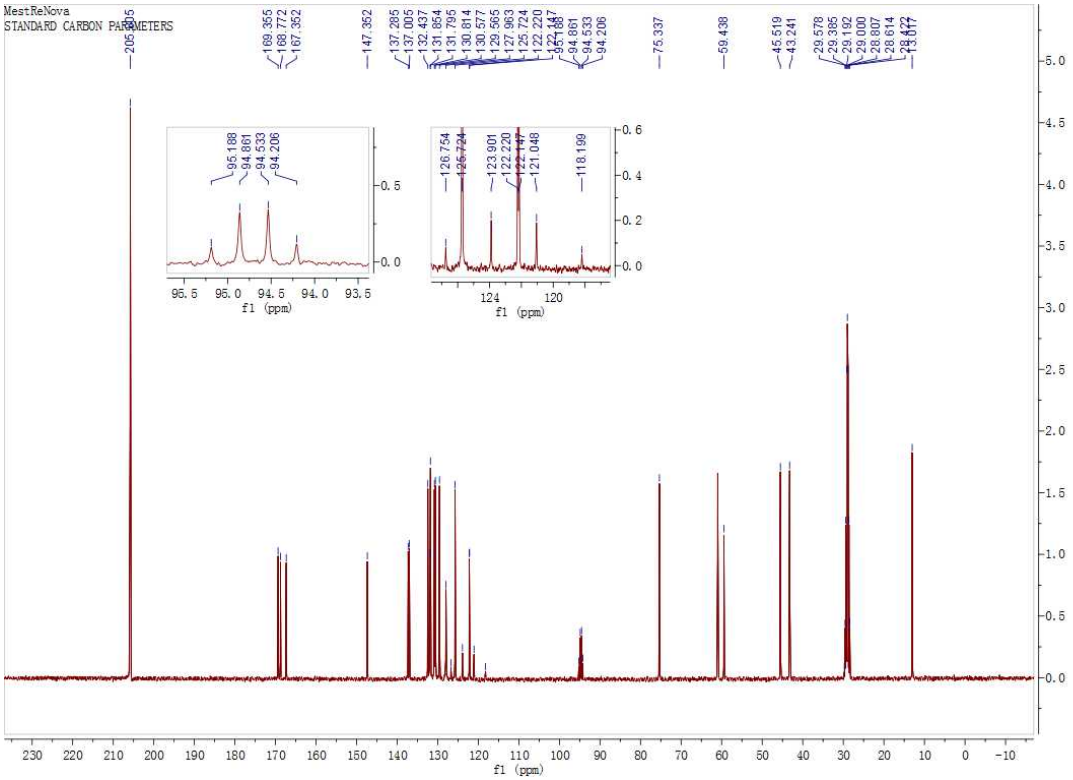
Number of Hits = 5

m/z	Delta m/z	DBE	Formula
684.94061	0.00009	36.0	$C_{37}H_6N_2O_3F_3BrNa^{+1}$
684.94033	0.00037	16.5	$C_{24}H_{19}N_2O_7F_3Br_2Na^{+1}$
684.93947	0.00123	39.5	$C_{40}H_5N_2O_2F_2BrNa^{+1}$
684.94207	-0.00137	27.5	$C_{35}H_{17}O_3FBr_2Na^{+1}$
684.93919	0.00151	20.0	$C_{27}H_{18}N_2O_6F_2Br_2Na^{+1}$

4e


LJ-M-Br-H
NAME s1p20121218
EXPNO 1
PROCNO 1
Date_ 20121218
Time 14.44
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 228.1
DW 48.400 usec
DE 6.00 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300482 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
1.63
0.95
0.97
0.95
4.97
0.82
0.99
1.97
1.97
3.00


-84.331
LJ-m-Br-F
ppm



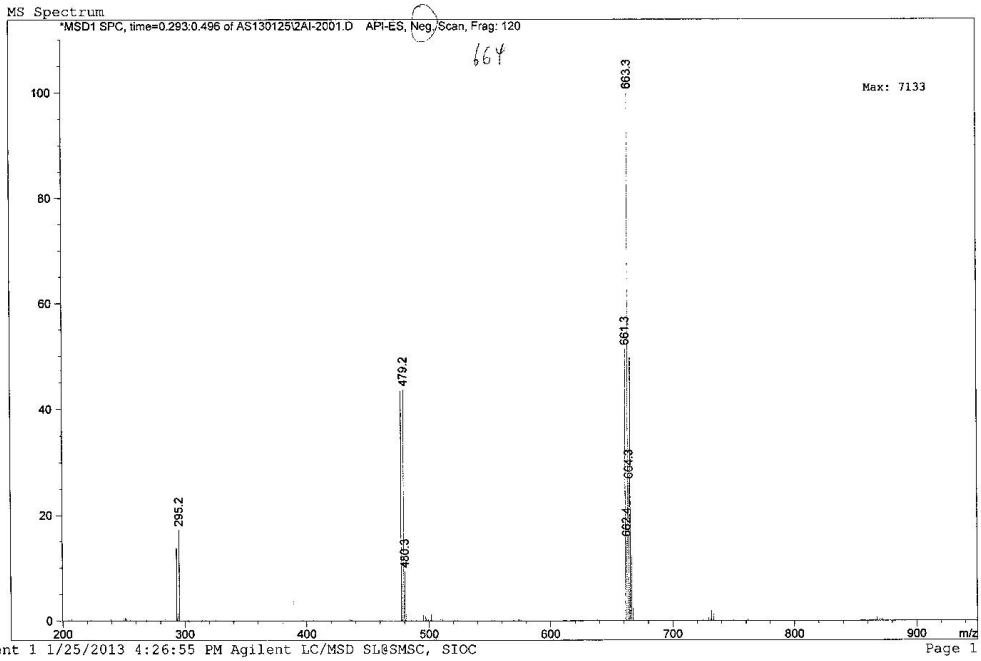
Print of Window 80: MS Spectrum

Injection Date : 1/25/2013 4:21:53 PM Seq. Line : 20
Sample Name : LJ-3 Location : P2-A-09
Acq. Operator : Agilent LC/MSD SL@SMSC, SIOC Inj : 1
Inj Volume : 0.1 µl
Acq. Method : C:\HPCHEM\1\METHODS\AANAL3'.M
Last changed : 1/25/2013 4:11:59 PM by Agilent LC/MSD SL@SMSC, SIOC
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\AANAL3'.M
Last changed : 1/25/2013 3:59:53 PM by Agilent LC/MSD SL@SMSC, SIOC
Test

m-Br

LJ-5

MS Spectrum



Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1301

Sample Serial Number: LJ-5

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 684.9397 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	7
F	18.998403	1	3
Br	78.918338	1	2
Na	22.989770	1	1

Additional Search Restrictions:

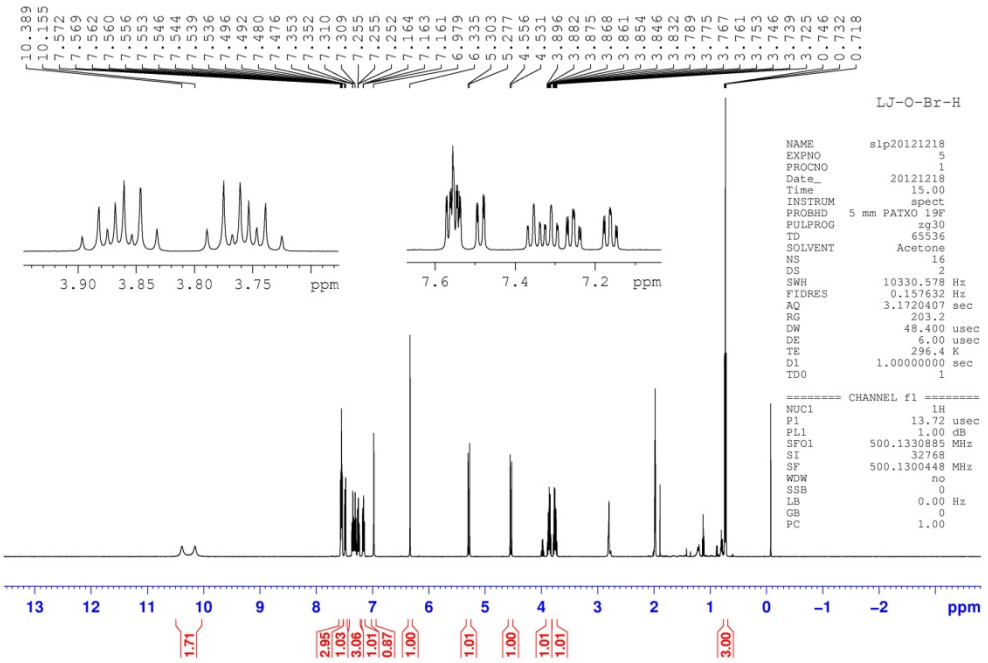
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

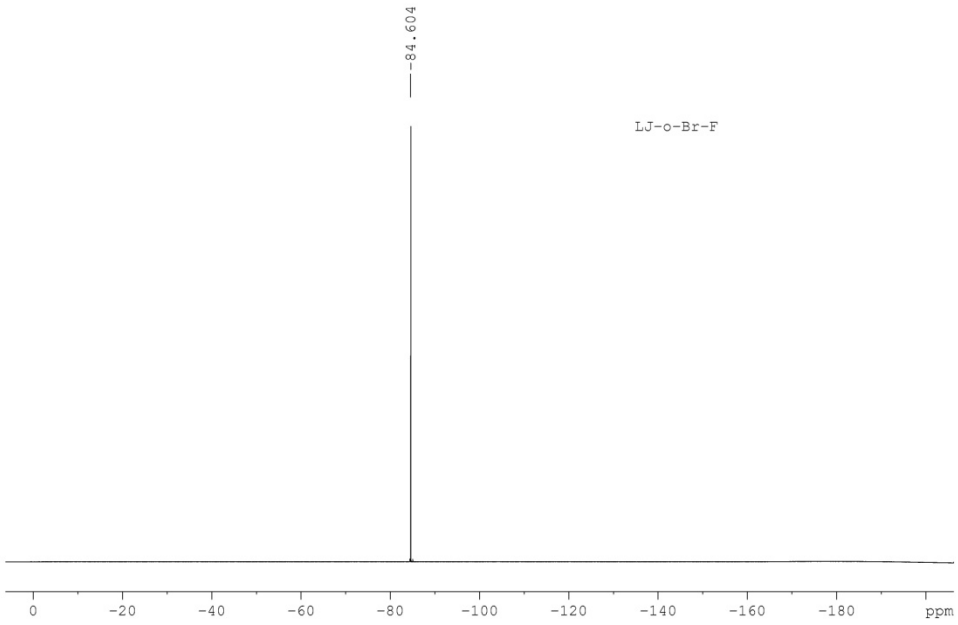
Search Results:

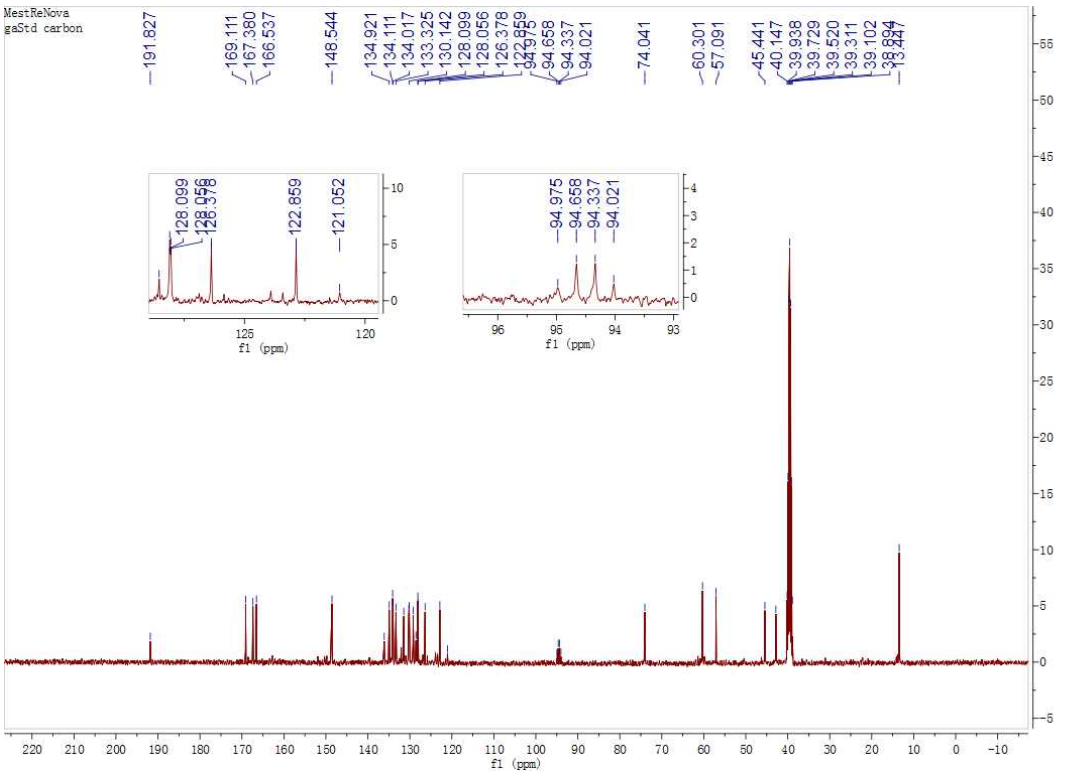
Number of Hits = 8

m/z	Delta m/z	DBE	Formula
684.93947	0.00023	39.5	$C_{40}H_5N_2O_2F_2BrNa^{+1}$
684.93919	0.00051	20.0	$C_{27}H_{18}N_2O_6F_2Br_2Na^{+1}$
684.94033	-0.00063	16.5	$C_{24}H_{19}N_2O_7F_3Br_2Na^{+1}$
684.94061	-0.00091	36.0	$C_{37}H_6N_2O_3F_3BrNa^{+1}$
684.93848	0.00122	29.5	$C_{36}H_{15}F_3Br_2Na^{+1}$
684.93833	0.00137	43.0	$C_{43}H_4N_2OFBrNa^{+1}$
684.93805	0.00165	23.5	$C_{30}H_{17}N_2O_5FBr_2Na^{+1}$
684.93793	0.00177	31.5	$C_{34}H_8NO_6F_3BrNa^{+1}$

4f


10.389
10.155
7.572
7.569
7.562
7.560
7.556
7.553
7.546
7.544
7.539
7.536
7.496
7.492
7.480
7.476
7.353
7.352
7.310
7.309
7.255
7.255
7.252
7.164
7.163
7.161
6.979
6.335
5.303
5.277
4.556
4.531
3.896
3.882
3.875
3.868
3.861
3.854
3.846
3.832
3.789
3.775
3.767
3.761
3.753
3.746
3.739
3.725
0.746
0.732
0.718
LJ-O-Br-H
3.90 3.85 3.80 3.75 ppm
7.6 7.4 7.2 ppm
NAME slp20121218
EXPNO 5
PROCNO 1
Date_ 20121218
Time 15.00
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 203.2
DW 48.400 usec
DE 6.00 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300448 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 ppm
1.71
2.95
1.03
3.06
1.01
0.87
1.00
1.01
1.00
1.01
1.01
3.00


-84.604
LJ-o-Br-F
0 -20 -40 -60 -80 -100 -120 -140 -160 -180 ppm

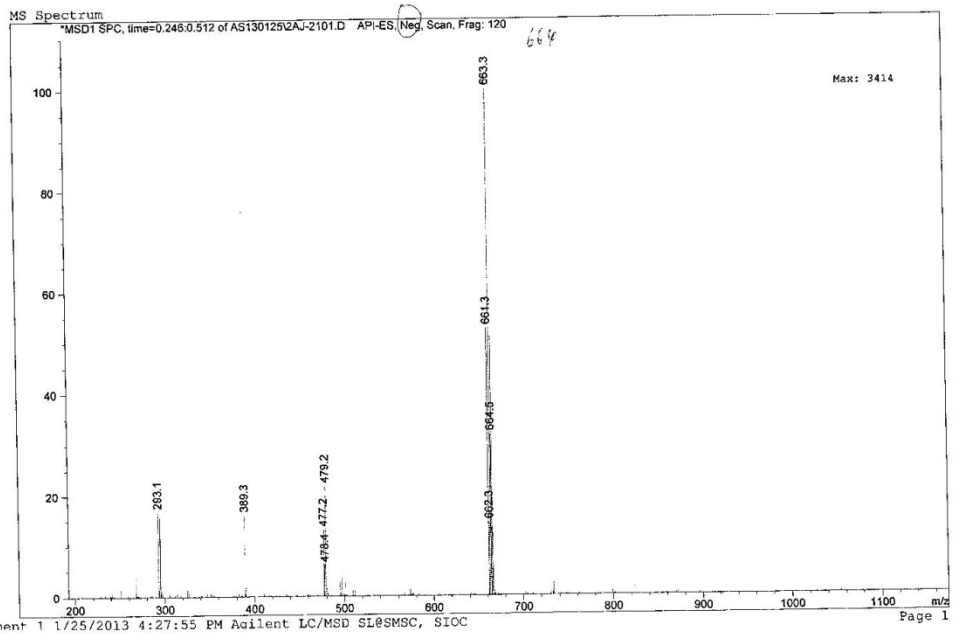


Print of Window 80: MS Spectrum

Injection Date : 1/25/2013 4:06:23 PM
Sample Name : LJ-2
Acq. Operator : Agilent LC/MSD SL@SMSC, SIOC
Seq. Line : 14
Location : P2-A-10
Inj : 1
Inj Volume : 0.1 µl

Acq. Method : C:\HPCHEM\1\METHODS\AGILENT1.M
Last changed : 1/25/2013 4:01:41 PM by Agilent LC/MSD SL@SMSC, SIOC
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\AANAL3'.M
Last changed : 1/25/2013 3:59:53 PM by Agilent LC/MSD SL@SMSC, SIOC
Test

LJ-6



Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1302

Sample Serial Number: LJ-6

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 684.9395 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	7
F	18.998403	1	3
Br	78.918338	1	2
Na	22.989770	1	1

Additional Search Restrictions:

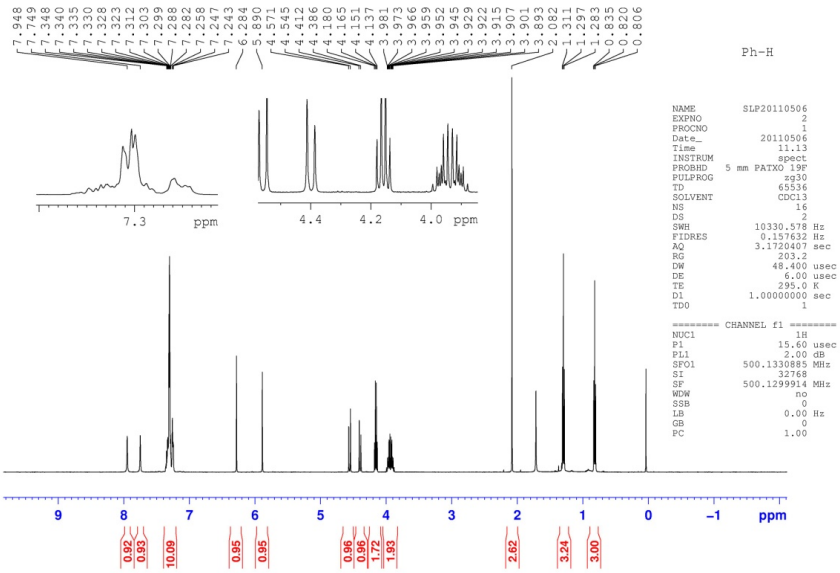
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

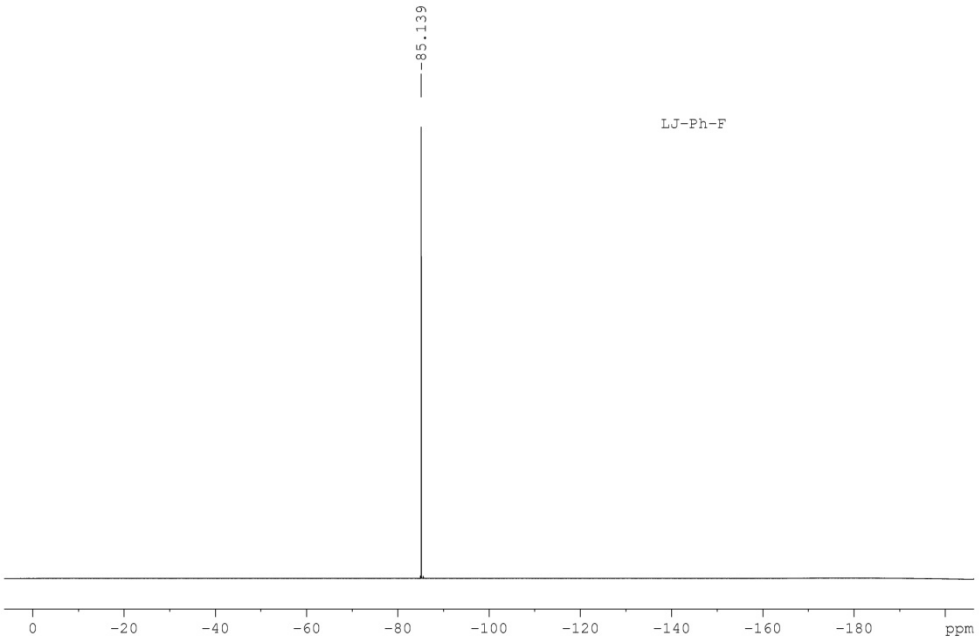
Search Results:

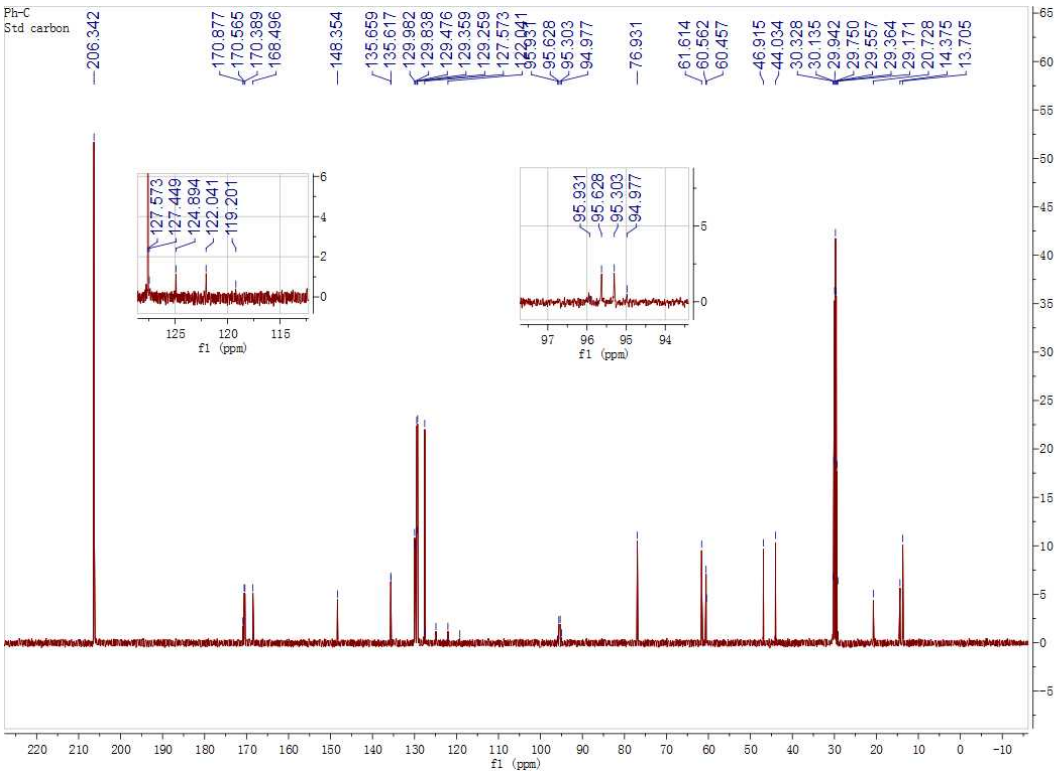
Number of Hits = 8

m/z	Delta m/z	DBE	Formula
684.93947	0.00003	39.5	$C_{40}H_5N_2O_2F_2BrNa^{+1}$
684.93919	0.00031	20.0	$C_{27}H_{18}N_2O_6F_2Br_2Na^{+1}$
684.94033	-0.00083	16.5	$C_{24}H_{19}N_2O_7F_3Br_2Na^{+1}$
684.93848	0.00102	29.5	$C_{36}H_{15}F_3Br_2Na^{+1}$
684.94061	-0.00111	36.0	$C_{37}H_6N_2O_3F_3BrNa^{+1}$
684.93833	0.00117	43.0	$C_{43}H_4N_2OFBrNa^{+1}$
684.93805	0.00145	23.5	$C_{30}H_{17}N_2O_5FBr_2Na^{+1}$
684.93793	0.00157	31.5	$C_{34}H_8NO_6F_3BrNa^{+1}$

4g


7.948
7.749
7.348
7.340
7.335
7.330
7.328
7.323
7.312
7.303
7.299
7.288
7.282
7.258
7.247
7.243
6.284
5.890
4.571
4.545
4.412
4.386
4.180
4.165
4.151
4.137
3.981
3.973
3.966
3.959
3.952
3.945
3.929
3.922
3.915
3.907
3.901
3.893
2.082
1.311
1.297
1.283
0.835
0.820
0.806
Ph-H
7.3 ppm
4.4 4.2 4.0 ppm
NAME SLP20110506
EXPNO 2
PROCNO 1
Date_ 20110506
Time 11.13
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 203.2
DW 48.400 usec
DE 6.00 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.60 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1299914 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 −1 ppm
0.92
0.93
10.09
0.95
0.95
0.96
0.96
1.72
1.93
2.62
3.24
3.00


−85.139
LJ-Ph-F
0 −20 −40 −60 −80 −100 −120 −140 −160 −180 ppm

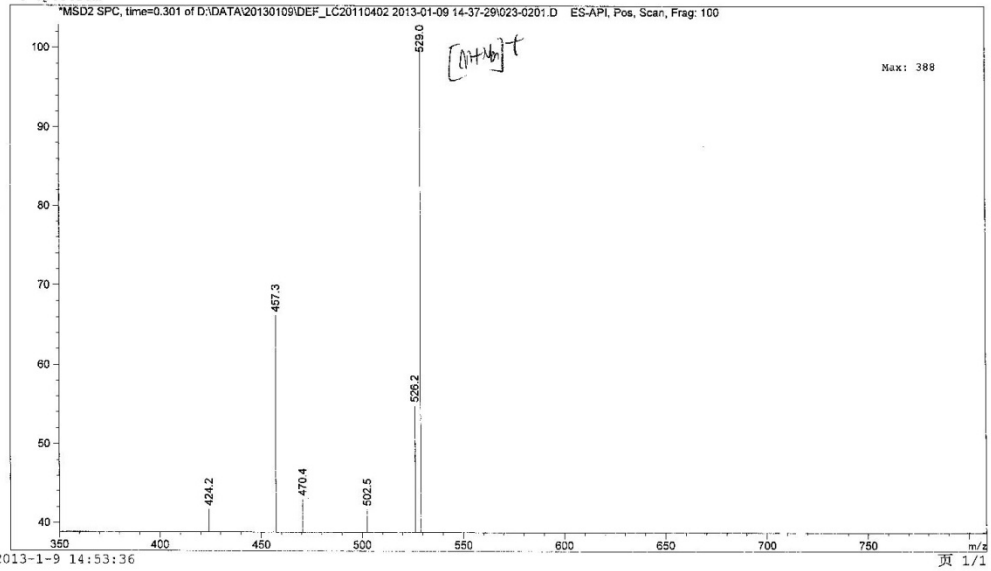


打印窗口 80: MS Spectrum
数据文件: : D:\DATA\20130109\DEF_LC20110402 2013-01-09 14-37-29\023-0201.D
样品名称 : 0104-5

PL
4J-7

操作者 :
仪器 : 仪器 1
进样日期 : 2013-1-9 14:42:13
序列行 : 2
位置 : 样品瓶 23
进样次数 : 1
进样量 : 0.1 µl
采集方法 : D:\DATA\20130109\DEF_LC20110402 2013-01-09 14-37-29\DEF_LC20110310.M
最后修改 : 2013-1-9 14:37:28
分析方法 : C:\CHEM32\1\METHODS\DEF_LC20110310.M
最后修改 : 2013-1-9 14:53:25
(调用后修改)

MS Spectrum



Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1303

Sample Serial Number: LJ-7

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 529.1194 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	7
F	18.998403	1	3
Na	22.989770	1	1

Additional Search Restrictions:

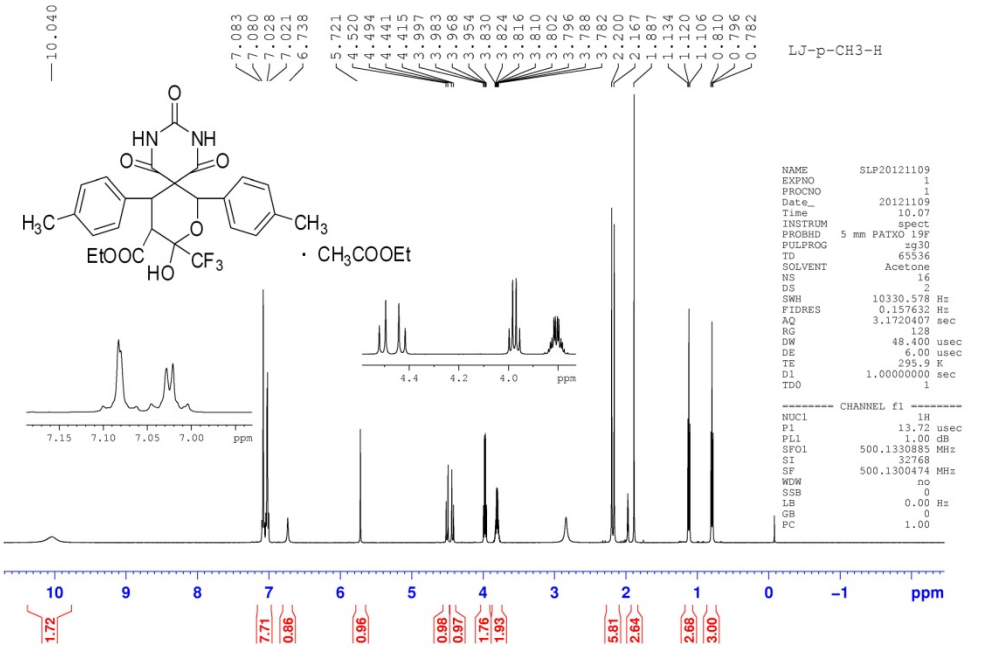
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

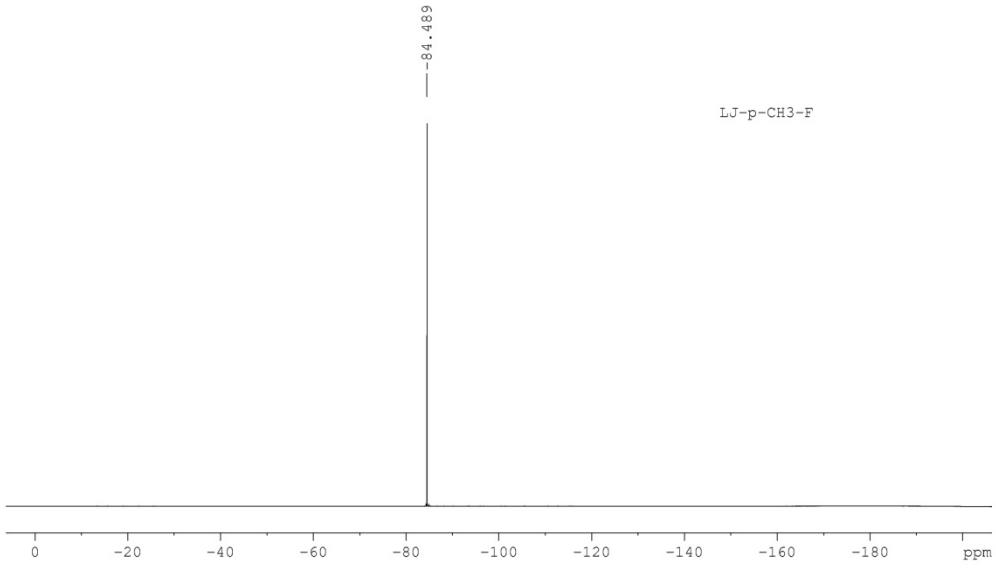
Search Results:

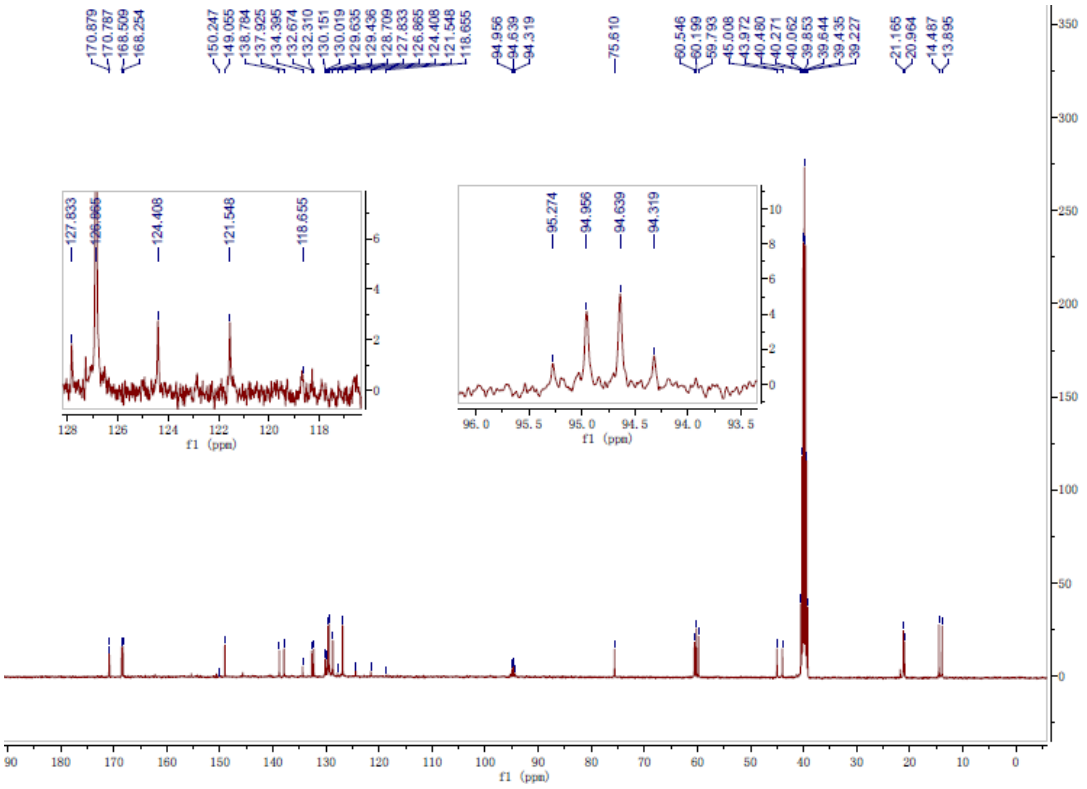
Number of Hits = 4

m/z	Delta m/z	DBE	Formula
529.11931	0.00009	15.5	$C_{24}H_{21}N_2O_7F_3Na^{+1}$
529.11817	0.00123	19.0	$C_{27}H_{20}N_2O_6F_2Na^{+1}$
529.12104	-0.00164	26.5	$C_{35}H_{19}O_3FNa^{+1}$
529.11746	0.00194	28.5	$C_{36}H_{17}F_3Na^{+1}$

4h


10.040
7.083
7.080
7.028
7.021
6.738
5.721
4.520
4.494
4.441
4.415
3.997
3.983
3.968
3.954
3.830
3.824
3.816
3.810
3.802
3.796
3.788
3.782
2.200
2.167
1.887
1.134
1.120
1.106
0.810
0.796
0.782
LJ-p-CH3-H
HN
NH
O
O
O
O
H3C
CH3
EtOOC
CF3
HO
· CH3COOEt
NAME SLP20121109
EXPNO 1
PROCNO 1
Date_ 20121109
Time 10.07
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 128
DW 48.400 usec
DE 6.00 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300474 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
1.72
7.71
0.86
0.96
0.98
0.97
1.76
1.93
5.81
2.64
2.68
3.00


-84.489
LJ-p-CH3-F
ppm



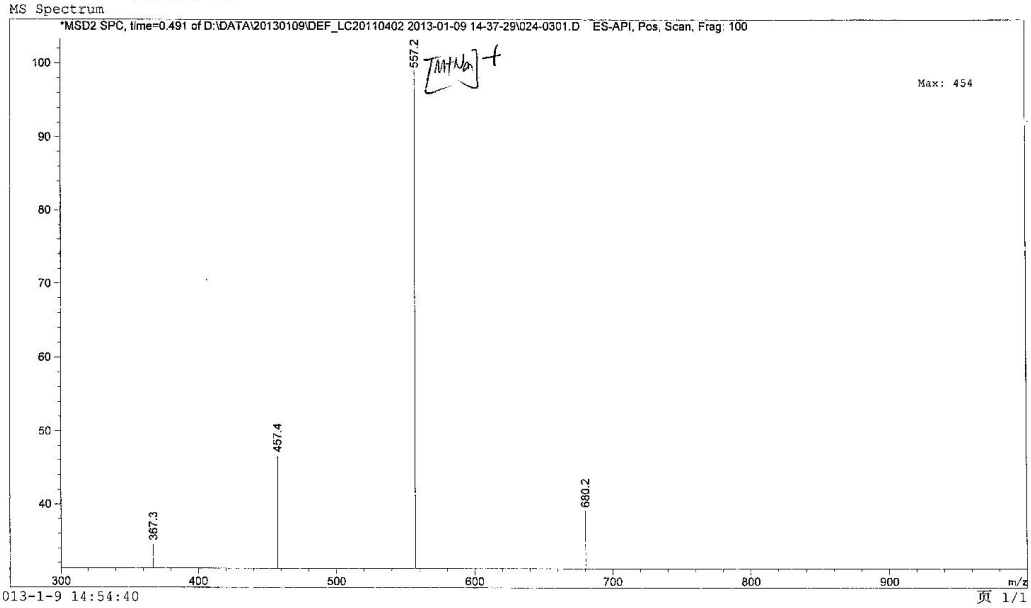
打印窗口 80: MS Spectrum
数据文件: : D:\DATA\20130109\DEF_LC20110402 2013-01-09 14-37-29\024-0301.D
样品名称 : 0104-6

p-CH3

LJ-8

操作者 : 序列行 : 3
仪器 : 仪器 1 位置 : 样品瓶 24
进样日期 : 2013-1-9 14:46:04 进样次数 : 1
进样量 : 0.1 µl
采集方法 : D:\DATA\20130109\DEF_LC20110402 2013-01-09 14-37-29\DEF_LC20110310.M
最后修改 : 2013-1-9 14:37:28
分析方法 : C:\CHEM32\1\METHODS\DEF_LC20110310.M
最后修改 : 2013-1-9 14:54:12
(调用后修改)

MS Spectrum



仪器 1 2013-1-9 14:54:40

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1304

Sample Serial Number: LJ-8

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 557.1496 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	7
F	18.998403	1	3
Na	22.989770	1	1

Additional Search Restrictions:

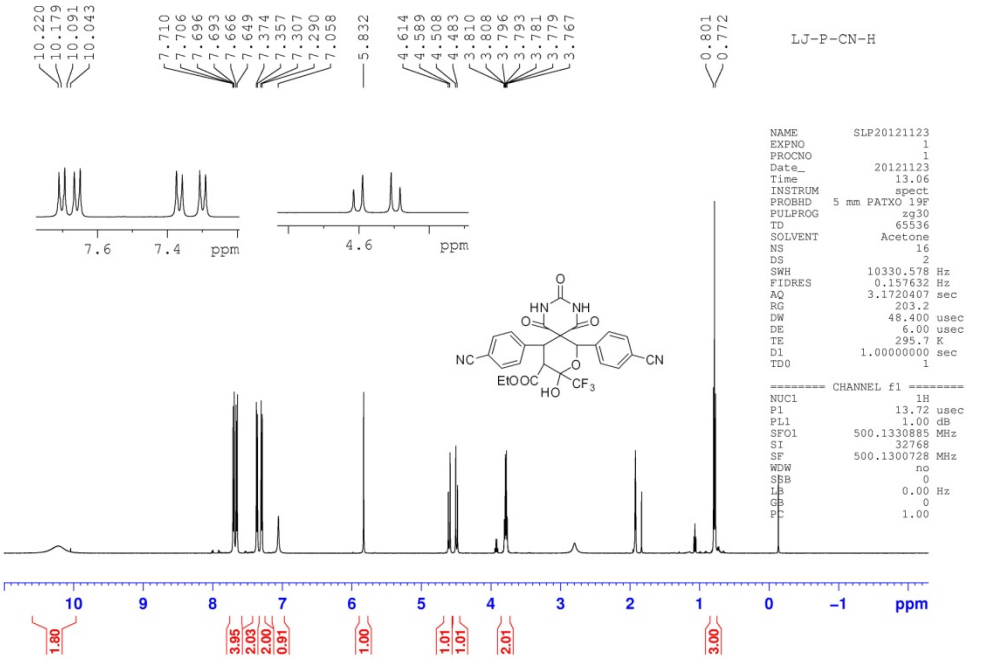
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

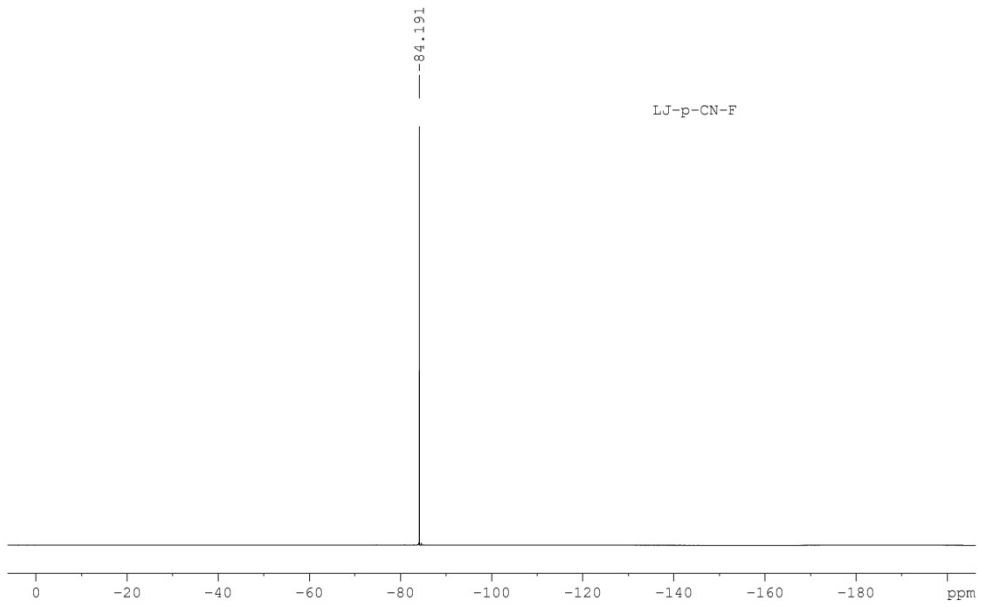
Search Results:

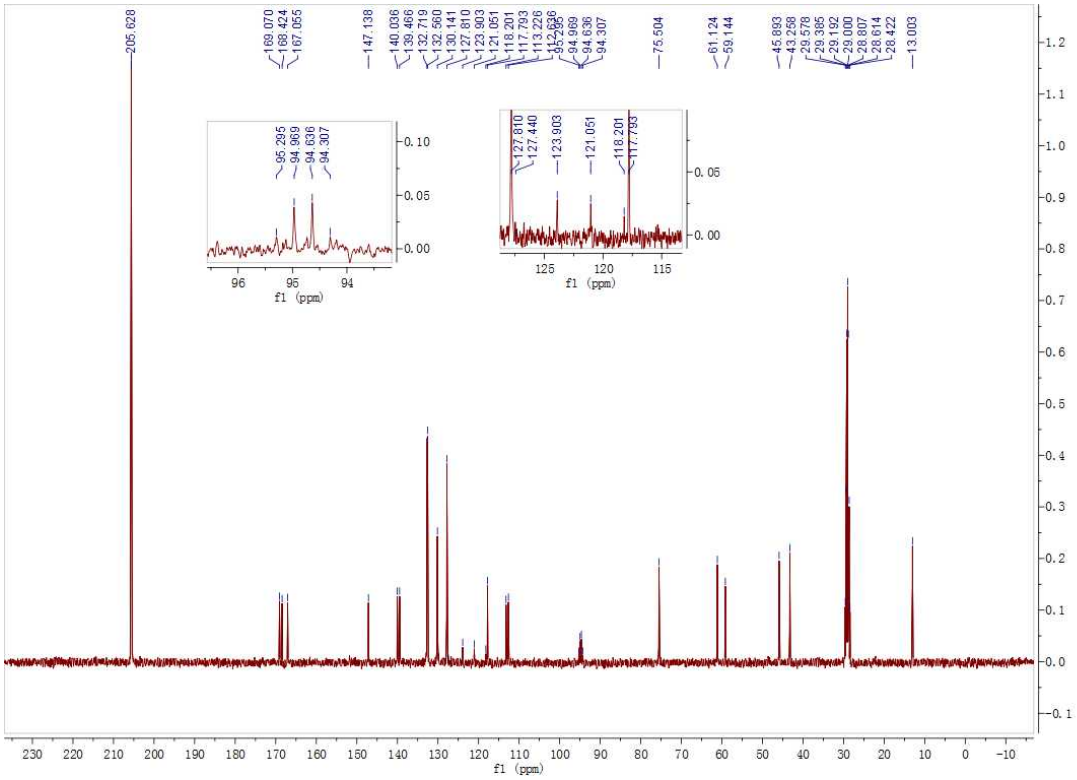
Number of Hits = 4

m/z	Delta m/z	DBE	Formula
557.14947	0.00013	19.0	$C_{29}H_{24}N_2O_6F_2Na^{+1}$
557.14876	0.00084	28.5	$C_{38}H_{21}F_3Na^{+1}$
557.15061	-0.00101	15.5	$C_{26}H_{25}N_2O_7F_3Na^{+1}$
557.14832	0.00128	22.5	$C_{32}H_{23}N_2O_5FNa^{+1}$

4i


10.220
10.179
10.091
10.043
7.710
7.706
7.696
7.693
7.666
7.649
7.374
7.357
7.307
7.290
7.058
5.832
4.614
4.589
4.508
4.483
3.810
3.808
3.796
3.793
3.781
3.779
3.767
0.801
0.772
LJ-P-CN-H
7.6
7.4
ppm
4.6
ppm
NAME SLP20121123
EXPNO 1
PROCNO 1
Date_ 20121123
Time 13.06
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 203.2
DW 48.400 usec
DE 6.00 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300728 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
HN
NH
O
O
O
NC
CN
EtOOC
HO
CF3
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
1.80
3.95
2.03
2.00
0.91
1.00
1.01
1.01
2.01
3.00


−84.191
LJ-p-CN-F
0
−20
−40
−60
−80
−100
−120
−140
−160
−180
ppm

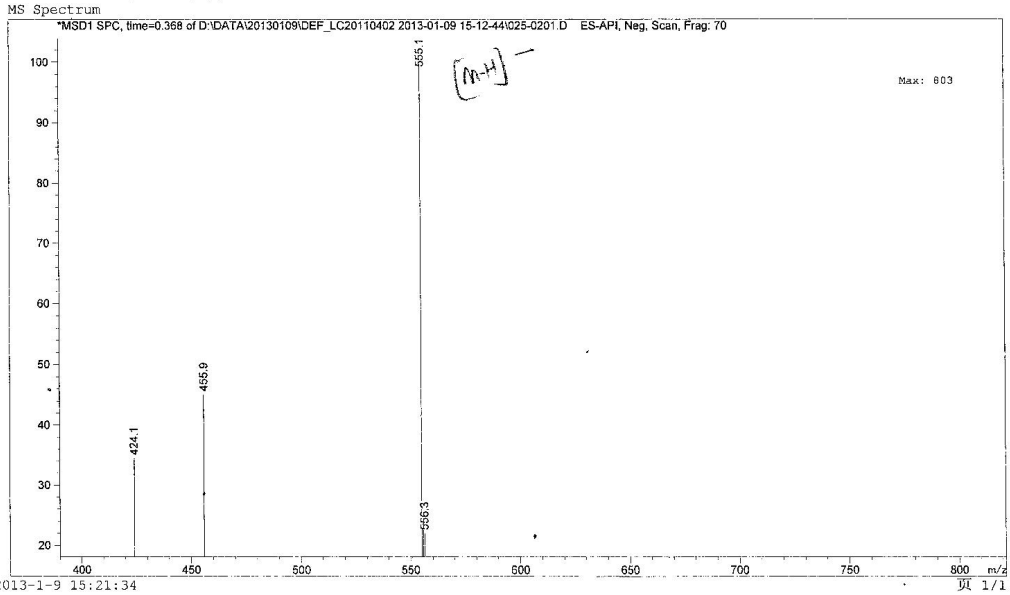


打印窗口 80: MS Spectrum
数据文件: : D:\DATA\20130109\DEF_LC20110402 2013-01-09 15-12-44\025-0201.D
样品名称 : 0104-7

P-CN
LJ-9

操作者 :
仪器 : 仪器 1
进样日期 : 2013-1-9 15:17:30
序列行 : 2
位置 : 样品瓶 25
进样次数 : 1
进样量 : 0.1 µl
采集方法 : D:\DATA\20130109\DEF_LC20110402 2013-01-09 15-12-44\DEF_LC20110310.M
最后修改 : 2013-1-9 15:12:42
分析方法 : C:\CHEM32\1\METHODS\DEF_LC20110310.M
最后修改 : 2013-1-9 15:20:42
(调用后修改)

MS Spectrum



仪器 1 2013-1-9 15:21:34

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academy of Sciences
High Resolution MS Data Report

Instrument

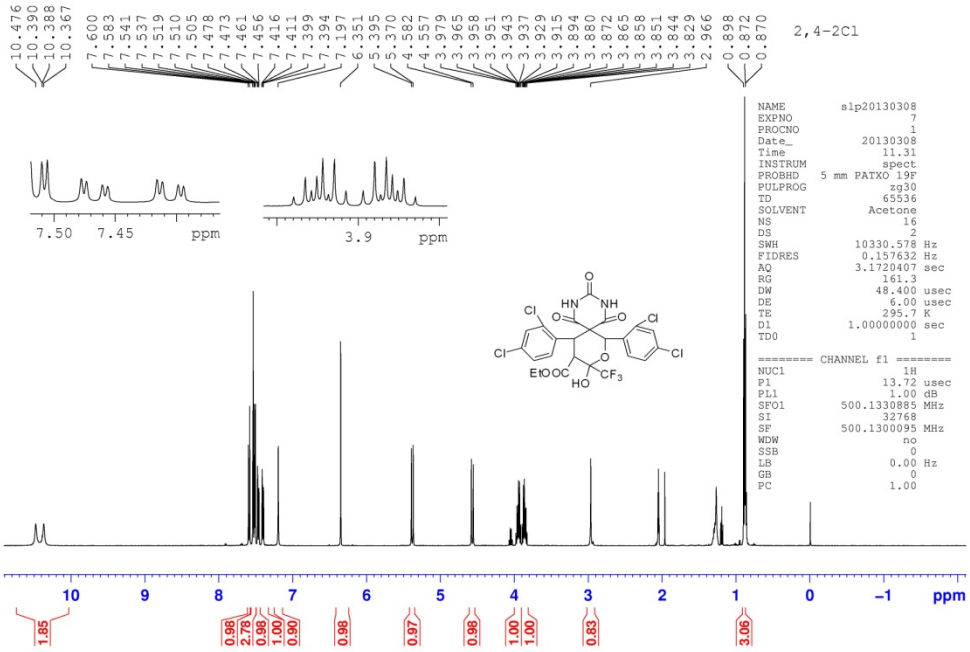
BRUKER

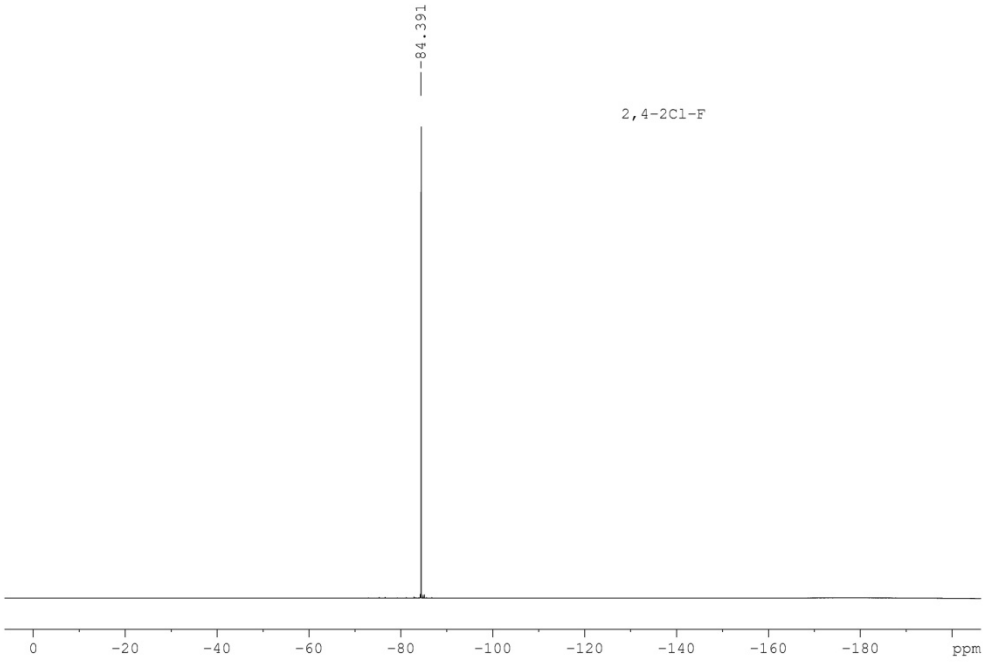
Bruker Daltonics, Inc. APEXIII 7.0 TESLA FTMS

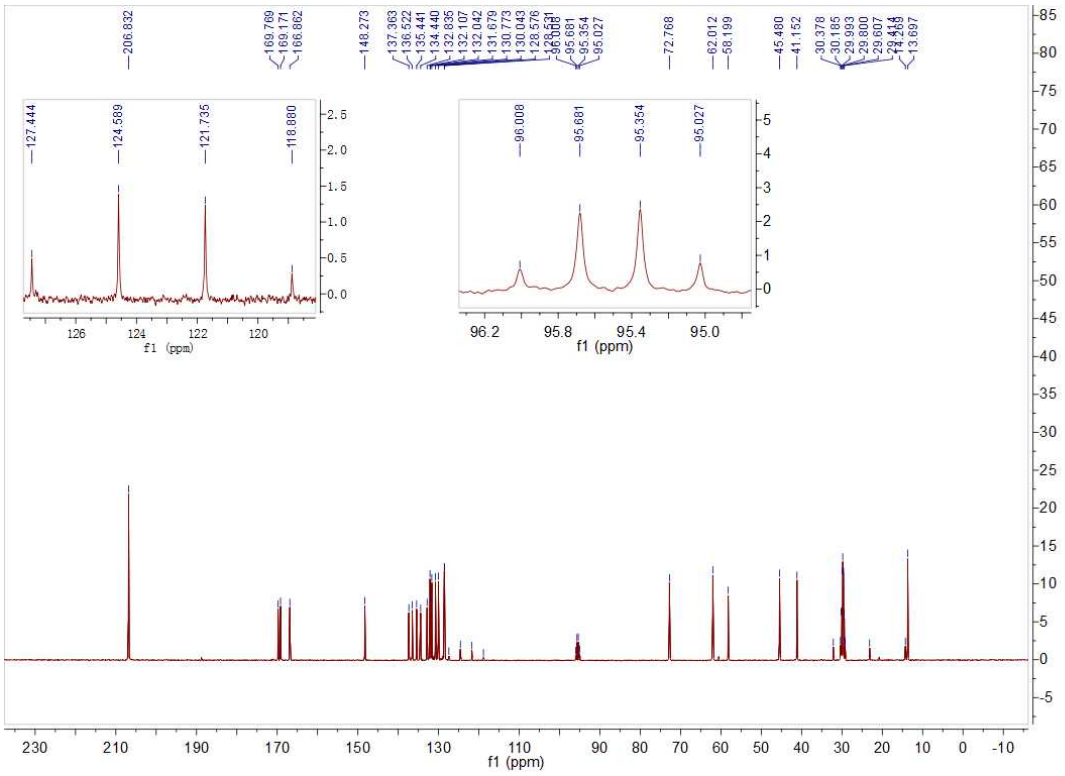
Card Serial Number	E132003
Analysis Name	D:\Data\LHB\20131022_000004.d
Sample Name	LJ-9
Acquisition Date	10/22/2013 11:04:43 AM
Operator:	zfj
Ionization Mode	ESI-Positive
Ion Mass (Measured)	579.1098

Sum Formula	Sigma	m/z	Err [ppm]	Mean Err [ppm]	Err [mDa]	rdb	N Rule	e⁻
C 23 H 21 F 3 N 3 Na 1 O 10	0.007	579.1071	-4.70	-5.02	-2.72	13.00	ok	odd
C 25 H 23 F 3 Na 1 O 11	0.007	579.1085	-2.38	-2.58	-1.38	12.50	ok	even
✓ C 26 H 19 F 3 N 4 Na 1 O 7	0.017	579.1098	-0.07	-0.41	-0.04	17.50	ok	even

4j


10.476
10.390
10.388
10.367
7.600
7.583
7.541
7.537
7.519
7.510
7.505
7.478
7.473
7.461
7.456
7.416
7.411
7.399
7.394
7.197
6.351
5.395
5.370
4.582
4.557
3.979
3.965
3.958
3.951
3.943
3.937
3.929
3.915
3.894
3.880
3.872
3.865
3.858
3.851
3.844
3.829
2.966
0.898
0.872
0.870
2,4-2Cl
7.50
7.45
ppm
3.9
ppm
NAME slp20130308
EXPNO 7
PROCNO 1
Date_ 20130308
Time 11.31
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 161.3
DW 48.400 usec
DE 6.00 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300095 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
HN
NH
O
Cl
EtOOC
HO
CF3
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
1.85
0.98
2.78
0.98
1.00
0.90
0.98
0.97
0.98
1.00
1.00
0.83
3.06


−84.391
2,4-2Cl-F
0
−20
−40
−60
−80
−100
−120
−140
−160
−180
ppm

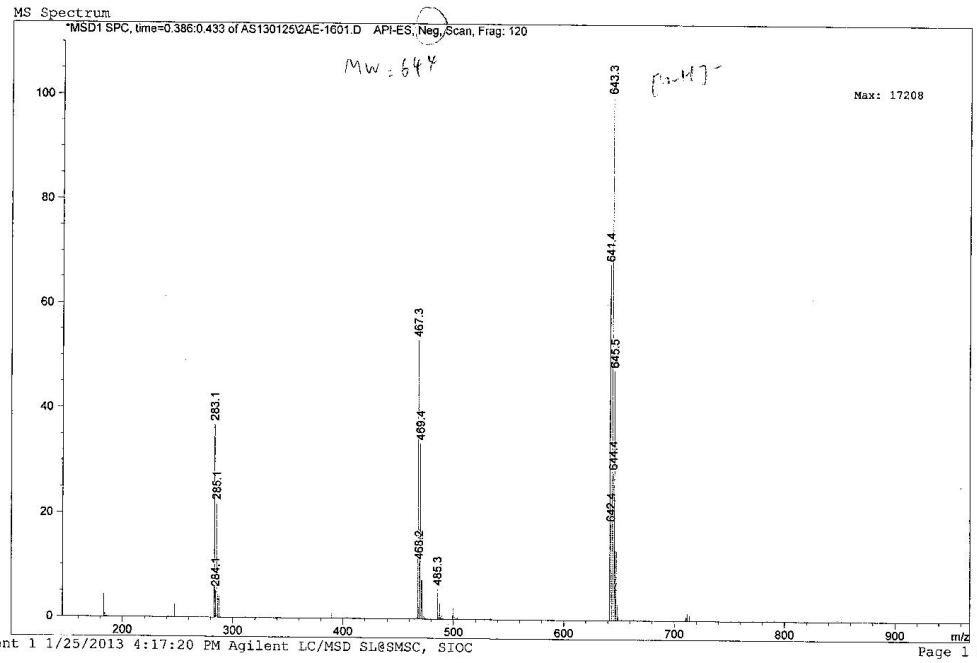


Print of Window 60: MS Spectrum

Injection Date	: 1/25/2013 4:11:49 PM	Seq. Line	: 16
Sample Name	: LJ-6	Location	: P2-A-05
Acq. Operator	: Agilent LC/MSD SL@SMSC, SIOC	Inj	: 1
		Inj Volume	: 0.3 µl
Acq. Method	: C:\HPCHEM\1\METHODS\AANAL3'.M		
Last changed	: 1/25/2013 4:11:59 PM by Agilent LC/MSD SL@SMSC, SIOC (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\AANAL3'.M		
Last changed	: 1/25/2013 3:59:53 PM by Agilent LC/MSD SL@SMSC, SIOC		
Test			

2,4-2d LJ-10

MS Spectrum



Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1296

Sample Serial Number: LJ-10

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 664.9641 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	7
F	18.998403	1	3
Cl	34.968853	2	4
Na	22.989770	1	1

Additional Search Restrictions:

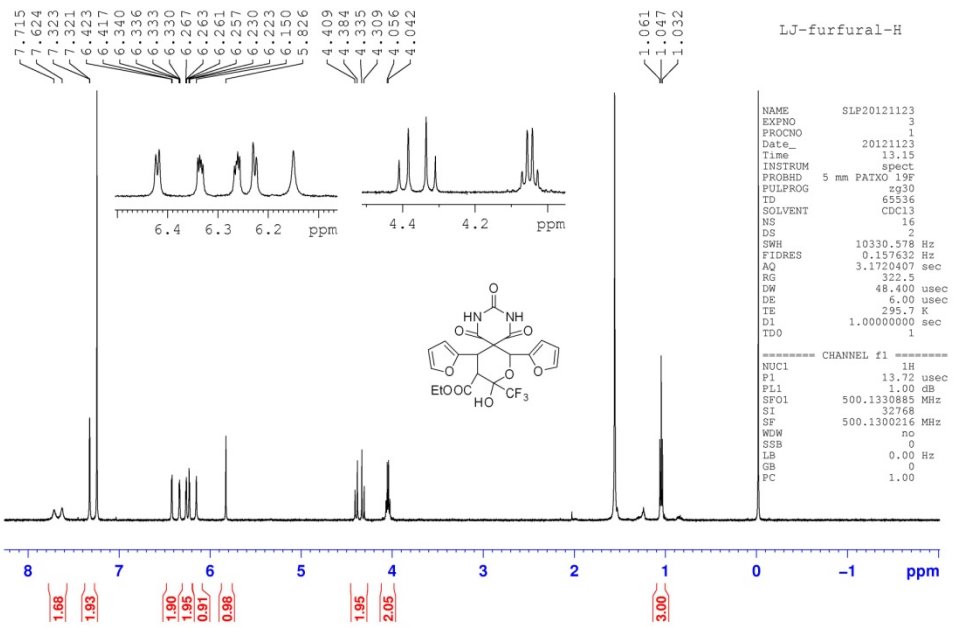
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

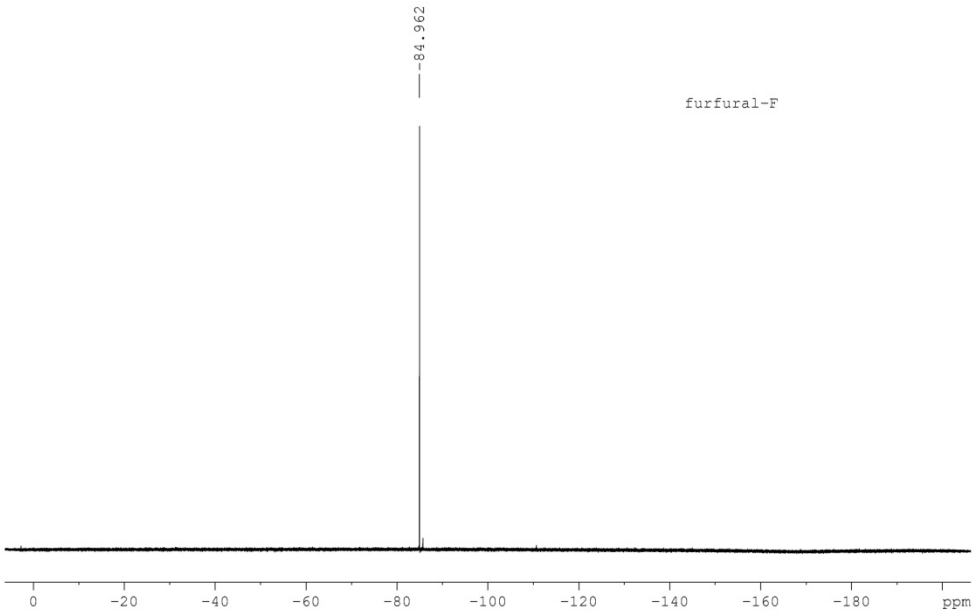
Search Results:

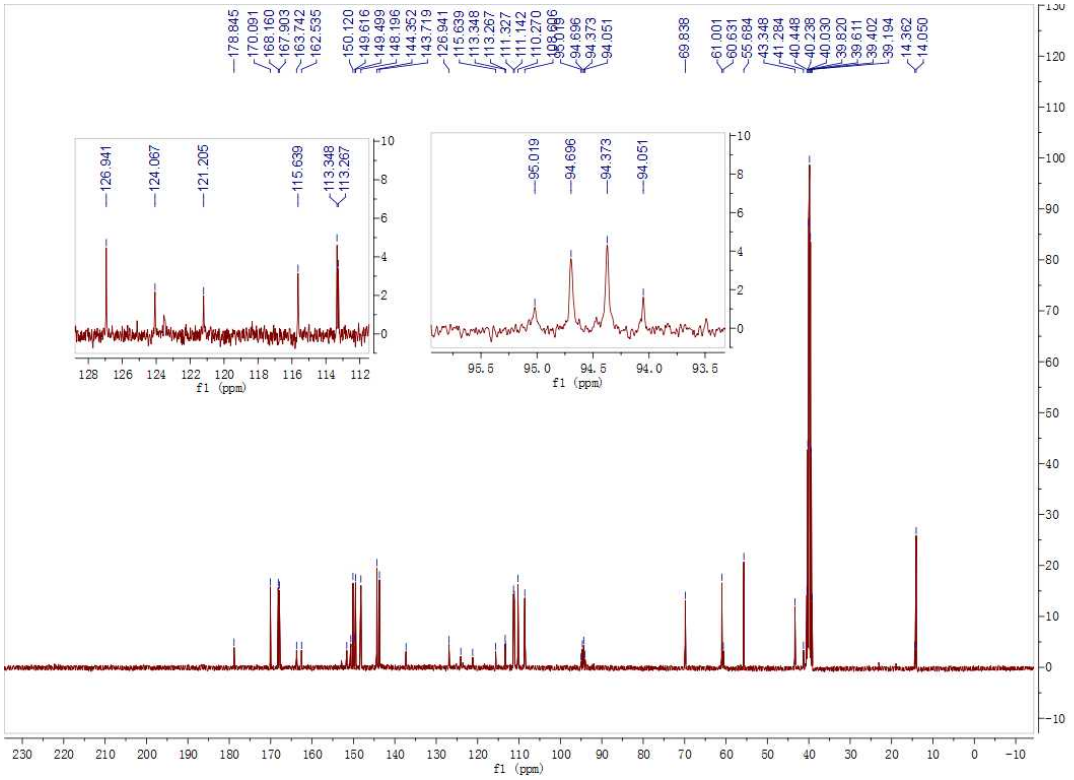
Number of Hits = 10

m/z	Delta m/z	DBE	Formula
664.96398	0.00012	33.5	$C_{36}H_8NO_5F_2Cl_2Na^{+1}$
664.96447	-0.00037	29.5	$C_{33}H_{11}N_2O_4F_2Cl_3Na^{+1}$
664.96342	0.00068	17.5	$C_{24}H_{17}N_2O_7F_3Cl_4Na^{+1}$
664.96333	0.00077	33.0	$C_{36}H_{10}N_2O_3FCl_3Na^{+1}$
664.96513	-0.00103	30.0	$C_{33}H_9NO_6F_3Cl_2Na^{+1}$
664.96516	-0.00106	28.5	$C_{35}H_{15}O_3FCl_4Na^{+1}$
664.96284	0.00126	37.0	$C_{39}H_7NO_4FCl_2Na^{+1}$
664.96552	-0.00142	41.5	$C_{42}H_5N_2OFCl_2Na^{+1}$
664.96561	-0.00151	26.0	$C_{30}H_{12}N_2O_5F_3Cl_3Na^{+1}$
664.96228	0.00182	21.0	$C_{27}H_{16}N_2O_6F_2Cl_4Na^{+1}$

4k


7.715
7.624
7.323
7.321
6.423
6.417
6.340
6.336
6.333
6.330
6.267
6.263
6.261
6.257
6.230
6.223
6.150
5.826
4.409
4.384
4.335
4.309
4.056
4.042
1.061
1.047
1.032
LJ-furfural-H
6.4 6.3 6.2 ppm
4.4 4.2 ppm
HN NH
EtOOC HO CF3
NAME SLP20121123
EXPNO 3
PROCNO 1
Date_ 20121123
Time 13.15
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 322.5
DW 48.400 usec
DE 6.00 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300216 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8 7 6 5 4 3 2 1 0 −1 ppm
1.68
1.93
1.90
1.95
0.91
0.98
1.95
2.05
3.00


−84.962
furfural-F
0 −20 −40 −60 −80 −100 −120 −140 −160 −180 ppm



数据文件: D:\DATA\20130109\DEF_LC20110402 2013-01-09 14-37-29\026-0501.D
样品名称 : 0104-8

K

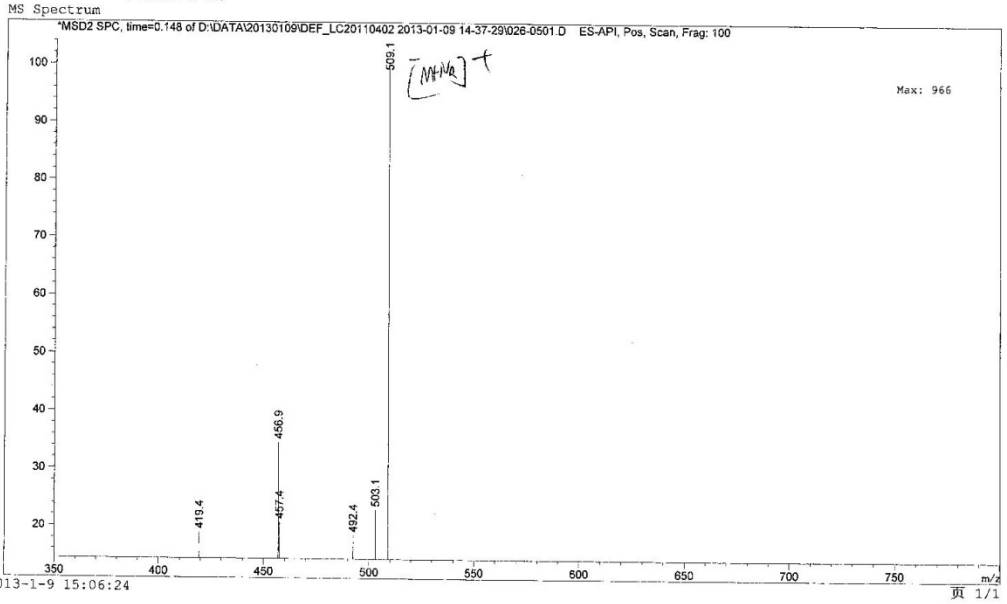
LJ-12

操作者 :
仪器 : 仪器 1
进样日期 : 2013-1-9 14:53:50

序列行 : 5
位置 : 样品瓶 26
进样次数 : 1
进样量 : 0.1 µl

采集方法 : D:\DATA\20130109\DEF_LC20110402 2013-01-09 14-37-29\DEF_LC20110310.M
最后修改 : 2013-1-9 14:37:28
分析方法 : C:\CHEM32\1\METHODS\DEF_LC20110310.M
最后修改 : 2013-1-9 14:55:22
(调用后修改)

MS Spectrum



仪器 1 2013-1-9 15:06:24

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1305

Sample Serial Number: LJ-12

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 509.0773 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	9
F	18.998403	1	3
Na	22.989770	1	1

Additional Search Restrictions:

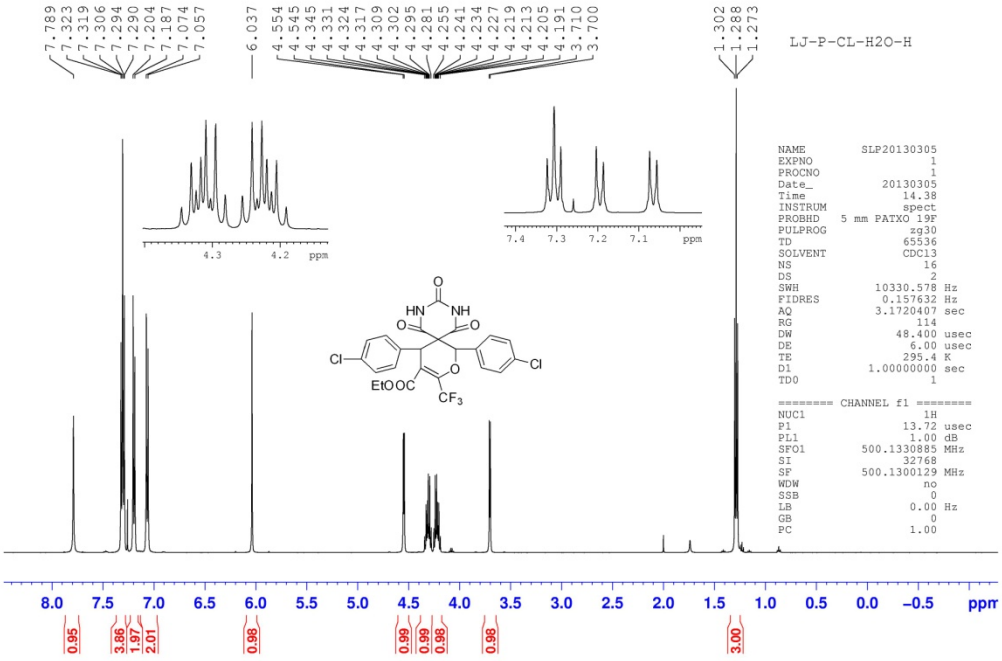
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

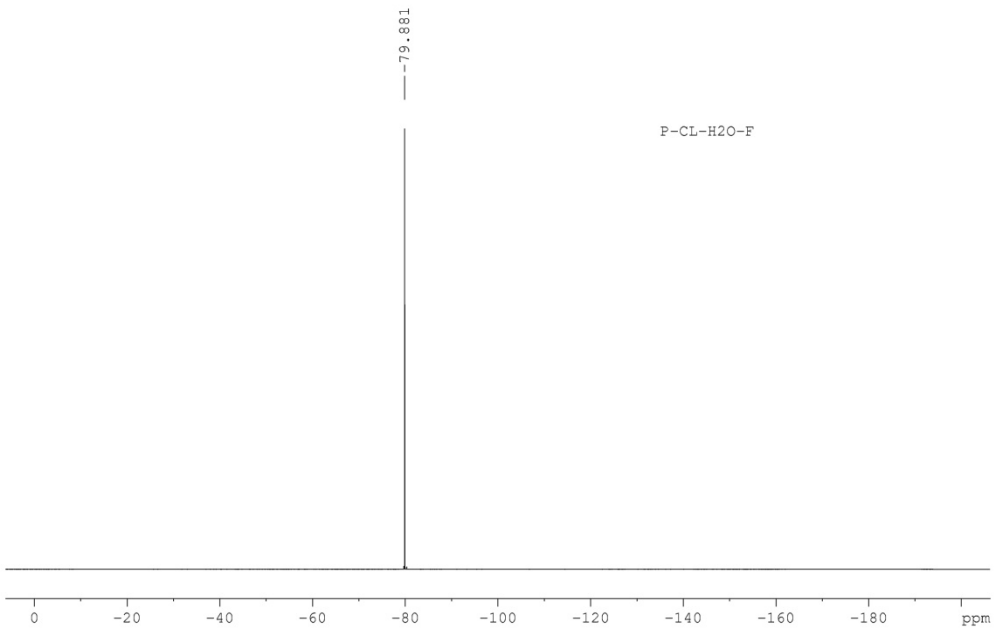
Search Results:

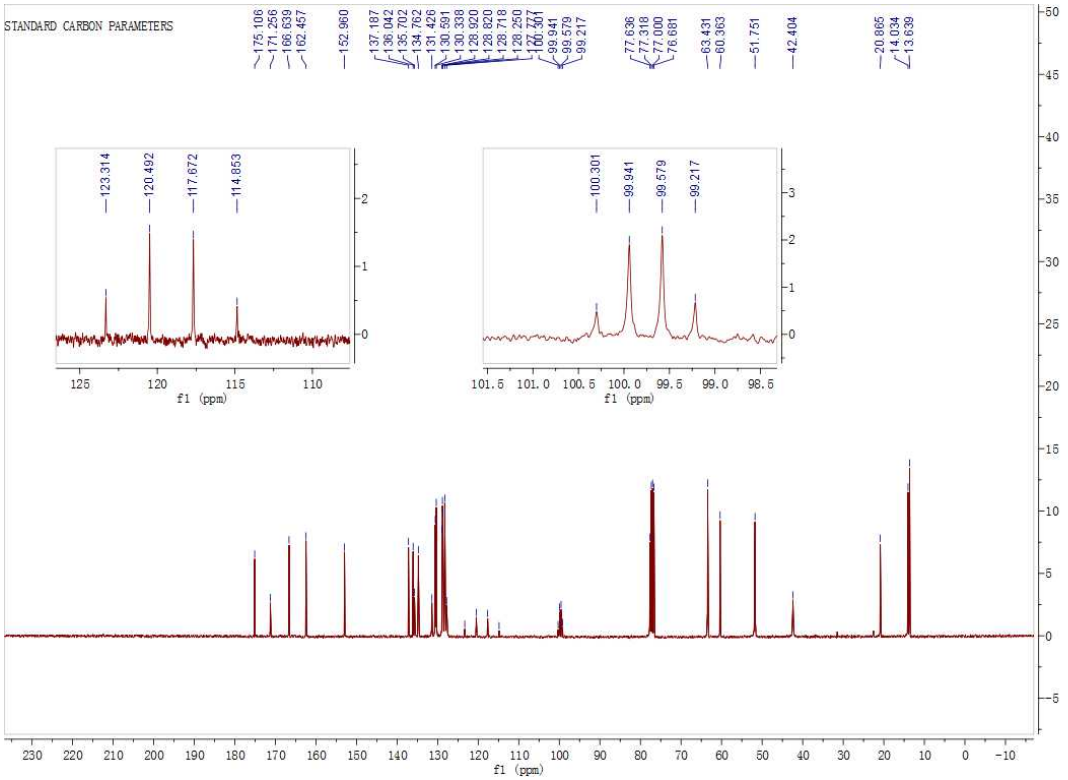
Number of Hits = 4

m/z	Delta m/z	DBE	Formula
509.07784	-0.00054	13.5	$C_{20}H_{17}N_2O_9F_3Na^{+1}$
509.07670	0.00060	17.0	$C_{23}H_{16}N_2O_8F_2Na^{+1}$
509.07599	0.00131	26.5	$C_{32}H_{13}O_2F_3Na^{+1}$
509.07555	0.00175	20.5	$C_{26}H_{15}N_2O_7FNa^{+1}$

5a


7.789
7.323
7.319
7.306
7.294
7.290
7.204
7.187
7.074
7.057
6.037
4.554
4.545
4.345
4.331
4.324
4.317
4.309
4.302
4.295
4.281
4.255
4.241
4.234
4.227
4.219
4.213
4.205
4.191
3.710
3.700
1.302
1.288
1.273
LJ-P-CL-H2O-H
NAME SLP20130305
EXPNO 1
PROCNO 1
Date_ 20130305
Time 14.38
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 114
DW 48.400 usec
DE 6.00 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
HN
NH
O
Cl
EtOOC
CF3
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm
0.95
3.86
1.97
2.01
0.98
0.99
0.99
0.98
0.98
3.00


-79.881
P-CL-H2O-F
0 -20 -40 -60 -80 -100 -120 -140 -160 -180 ppm



Print of window 80: MS Spectrum

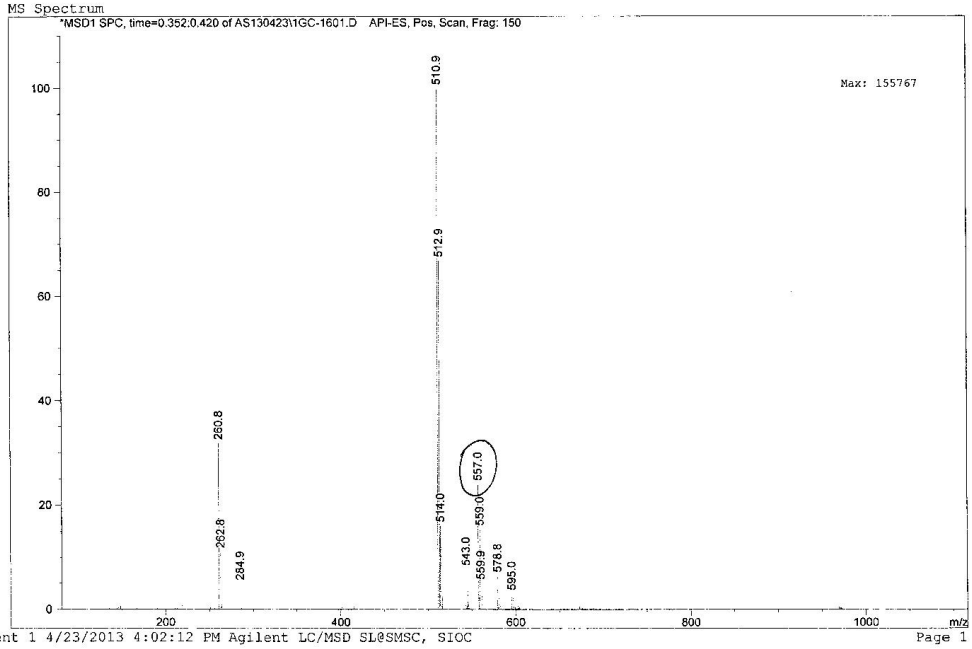
Injection Date : 4/23/2013 3:59:16 PM Seq. Line : 16
Sample Name : LJ-1 Location : P1-G-03
Acq. Operator : Agilent LC/MSD SL@SMSC, SIOC Inj : 1
Inj Volume : 0.1 µl

Acq. Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/23/2013 3:21:20 PM by Agilent LC/MSD SL@SMSC, SIOC
Analysis Method : C:\HPCHEM\1\METHODS\LC_A.M
Last changed : 4/23/2013 3:59:26 PM by Agilent LC/MSD SL@SMSC, SIOC
(modified after loading)

Test

p-Cl-H20
LJ-13

MS Spectrum



Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1306

Sample Serial Number: LJ-13

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 579.0314 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	6
F	18.998403	1	3
Cl	34.968853	1	2
Na	22.989770	1	1

Additional Search Restrictions:

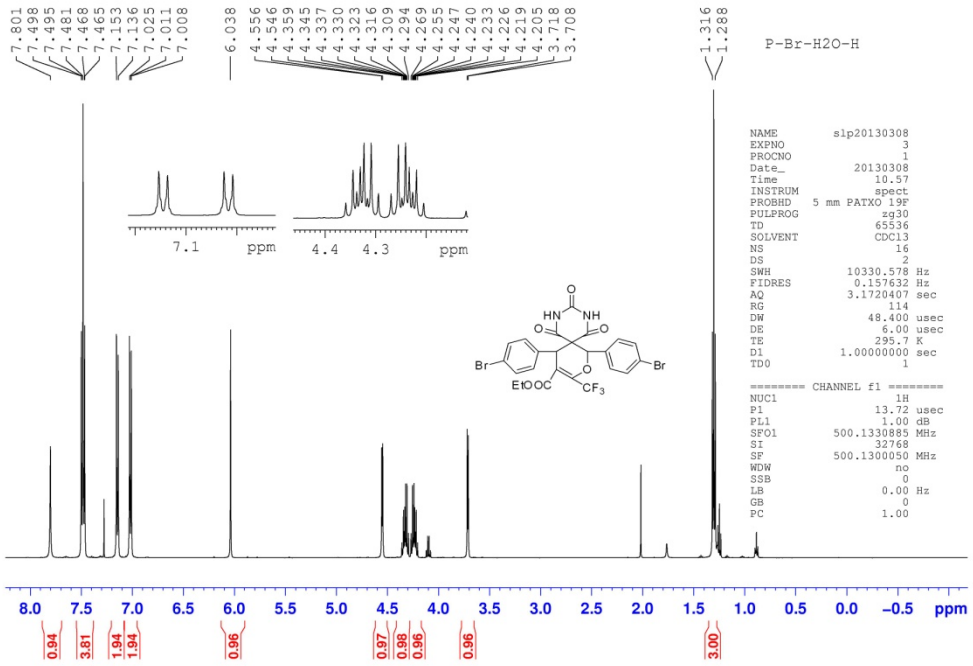
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

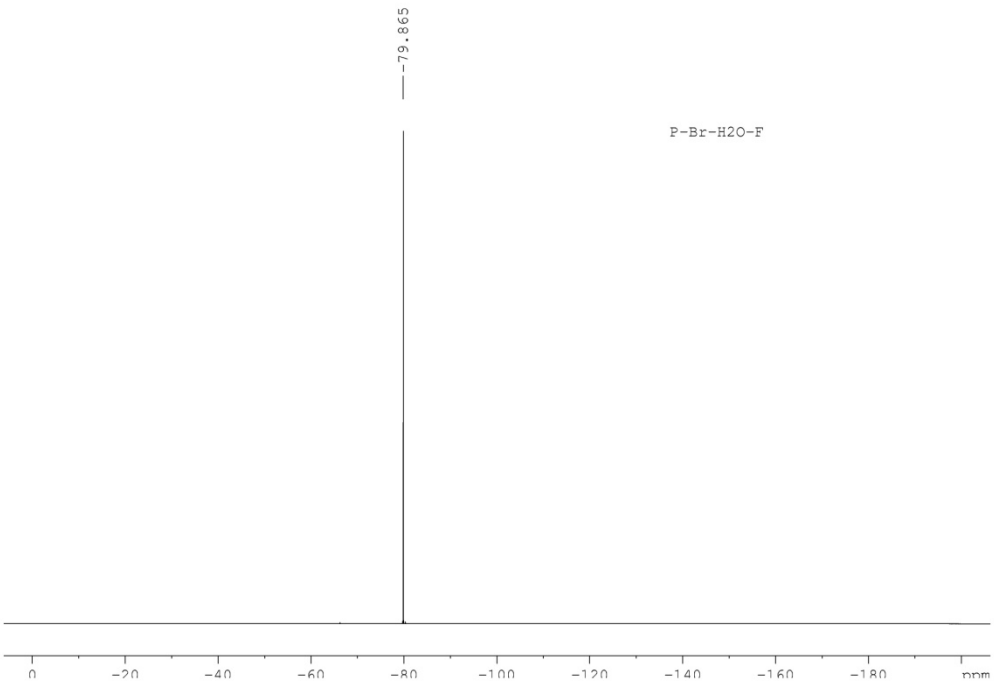
Search Results:

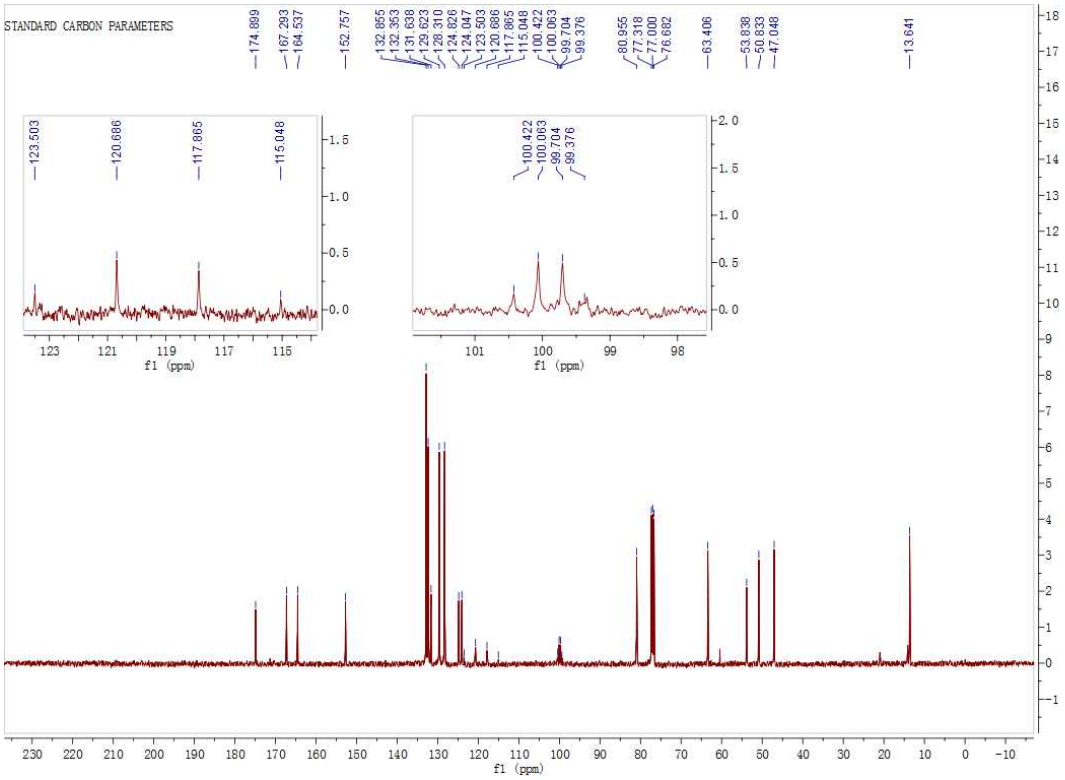
Number of Hits = 6

m/z	Delta m/z	DBE	Formula
579.03185	-0.00045	29.5	$C_{33}H_{11}N_2O_3F_2ClNa^{+1}$
579.03080	0.00060	17.5	$C_{24}H_{17}N_2O_6F_3Cl_2Na^{+1}$
579.03071	0.00069	33.0	$C_{36}H_{10}N_2O_2FClNa^{+1}$
579.03254	-0.00114	28.5	$C_{35}H_{15}O_2FCl_2Na^{+1}$
579.03299	-0.00159	26.0	$C_{30}H_{12}N_2O_4F_3ClNa^{+1}$
579.02966	0.00174	21.0	$C_{27}H_{16}N_2O_5F_2Cl_2Na^{+1}$

5b


7.801
7.498
7.495
7.481
7.468
7.465
7.153
7.136
7.025
7.011
7.008
6.038
4.556
4.546
4.359
4.345
4.337
4.330
4.323
4.316
4.309
4.294
4.269
4.255
4.247
4.240
4.233
4.226
4.219
4.205
3.718
3.708
1.316
1.288
P-Br-H2O-H
7.1 ppm
4.4 4.3 ppm
HN
NH
O
O
O
Br
Br
EtOOC
CF3
NAME s1p20130308
EXPNO 3
PROCNO 1
Date_ 20130308
Time 10.57
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 114
DW 48.400 usec
DE 6.00 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300050 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
0.94
3.81
1.94
1.94
0.96
0.97
0.98
0.96
0.96
3.00


−79.865
P-Br-H2O-F
0 −20 −40 −60 −80 −100 −120 −140 −160 −180 ppm



Print of window 80: MS Spectrum

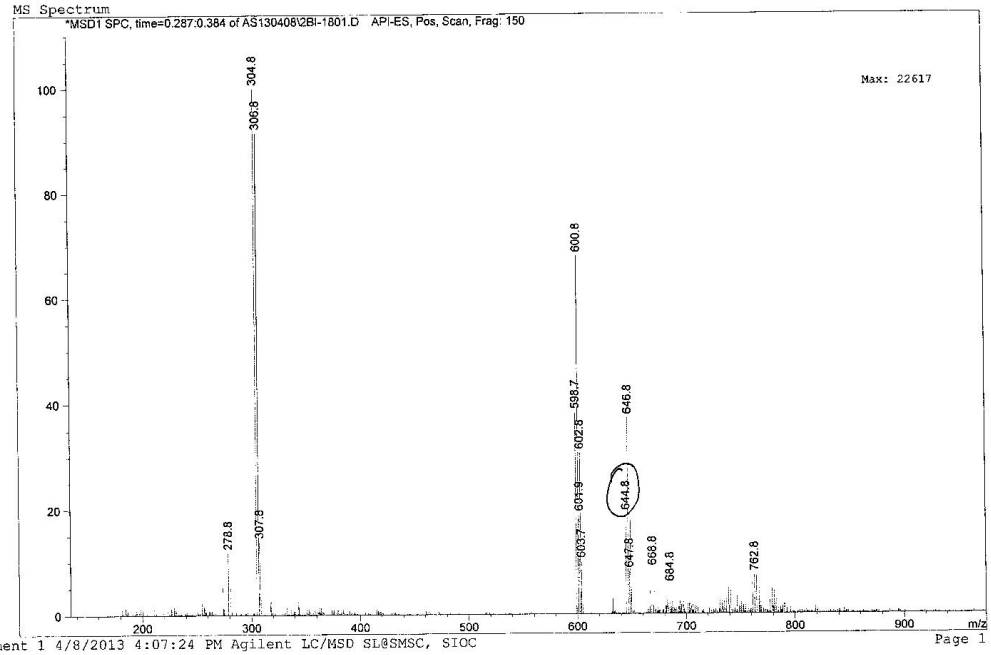
Injection Date : 4/8/2013 3:32:53 PM
Sample Name : LJ-4
Acq. Operator : Agilent LC/MSD SL@SMSC, SIOC
Seq. Line : 18
Location : P2-B-09
Inj : 1
Inj Volume : 0.1 µl

Acq. Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:09:35 PM by Agilent LC/MSD SL@SMSC, SIOC
Analysis Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:33:25 PM by Agilent LC/MSD SL@SMSC, SIOC
(modified after loading)

Test

p-Br-H2O
LJ-14

MS Spectrum



Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1307

Sample Serial Number: LJ-14

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 666.9289 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	6
F	18.998403	1	3
Br	78.918338	1	2
Na	22.989770	1	1

Additional Search Restrictions:

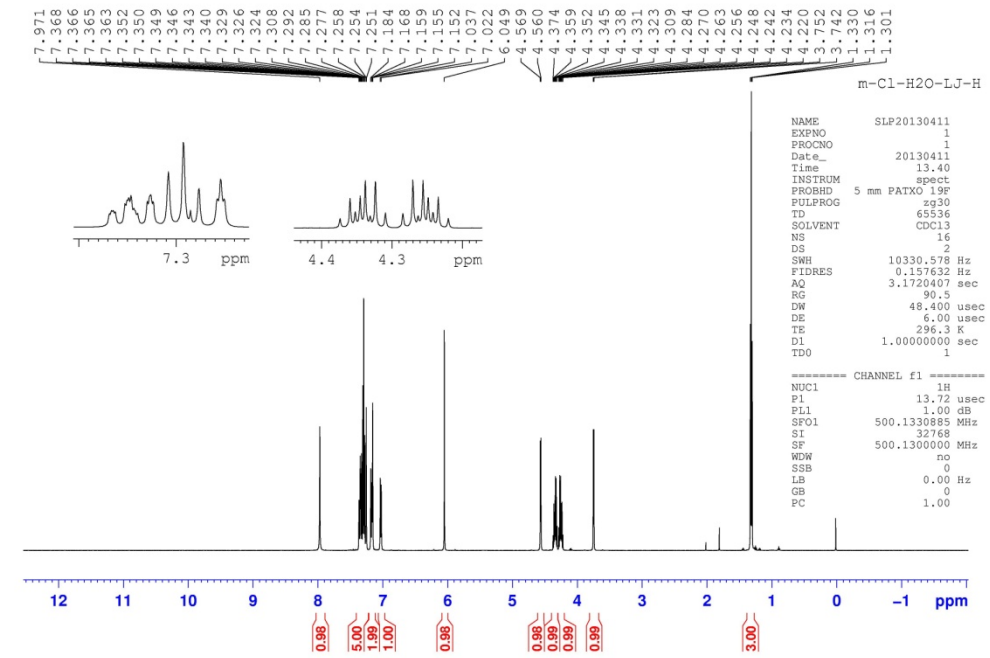
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

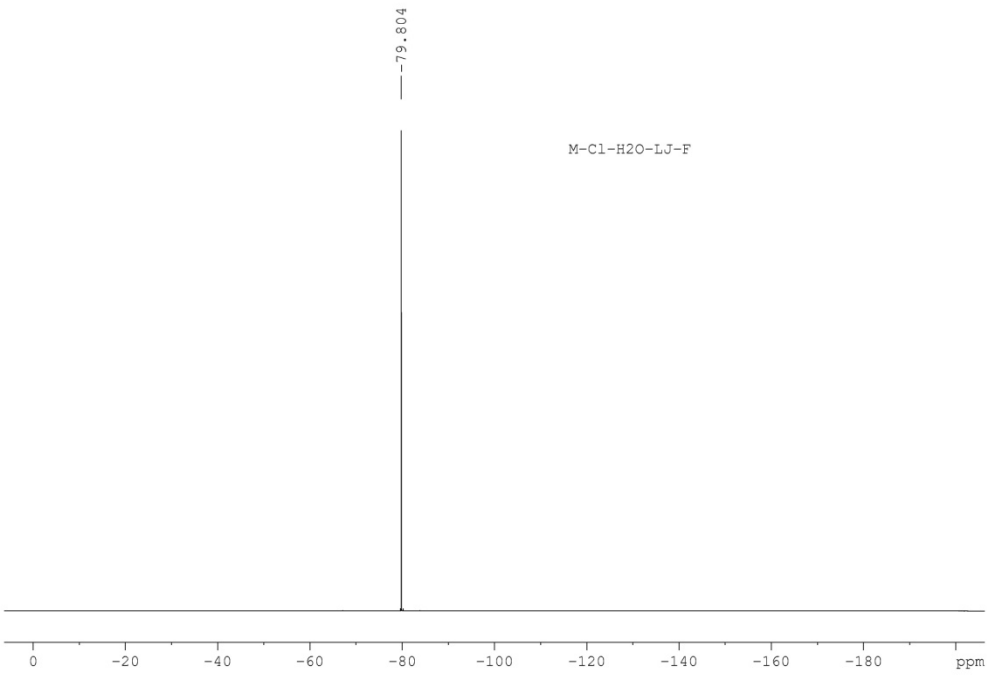
Search Results:

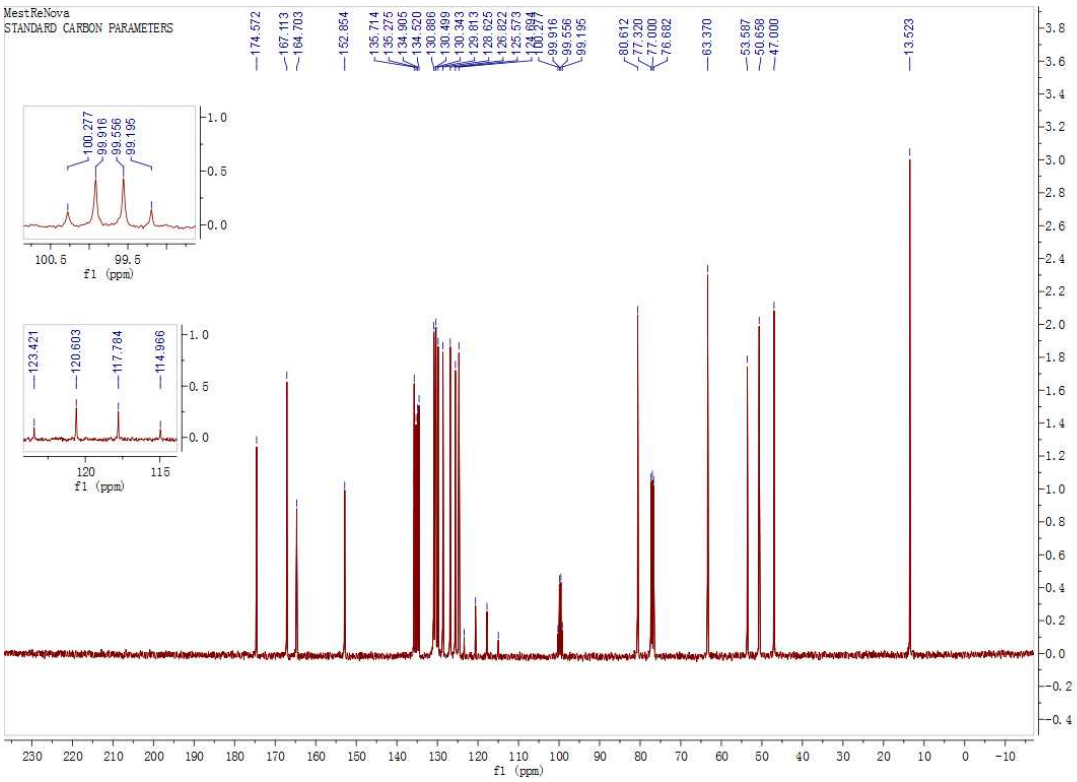
Number of Hits = 7

m/z	Delta m/z	DBE	Formula
666.92890	0.00000	40.5	$C_{40}H_3N_2OF_2BrNa^{+1}$
666.92863	0.00027	21.0	$C_{27}H_{16}N_2O_5F_2Br_2Na^{+1}$
666.92977	-0.00087	17.5	$C_{24}H_{17}N_2O_6F_3Br_2Na^{+1}$
666.92776	0.00114	44.0	$C_{43}H_2N_2FBrNa^{+1}$
666.93005	-0.00115	37.0	$C_{37}H_4N_2O_2F_3BrNa^{+1}$
666.92748	0.00142	24.5	$C_{30}H_{15}N_2O_4FBr_2Na^{+1}$
666.92737	0.00153	32.5	$C_{34}H_6NO_5F_3BrNa^{+1}$

5c


7.971
7.368
7.366
7.365
7.363
7.352
7.350
7.349
7.346
7.343
7.340
7.329
7.326
7.324
7.308
7.292
7.285
7.277
7.258
7.254
7.251
7.184
7.168
7.159
7.155
7.152
7.037
7.022
6.049
4.569
4.560
4.374
4.359
4.352
4.345
4.338
4.331
4.323
4.309
4.284
4.270
4.263
4.256
4.248
4.242
4.234
4.220
3.752
3.742
1.330
1.316
1.301
m-Cl-H2O-LJ-H
7.3 ppm
4.4 4.3 ppm
NAME SLP20130411
EXPNO 1
PROCNO 1
Date_ 20130411
Time 13.40
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 90.5
DW 48.400 usec
DE 6.00 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
12 11 10 9 8 7 6 5 4 3 2 1 0 −1 ppm
0.98 5.00 1.99 1.00 0.98 0.98 0.99 0.99 0.99 3.00

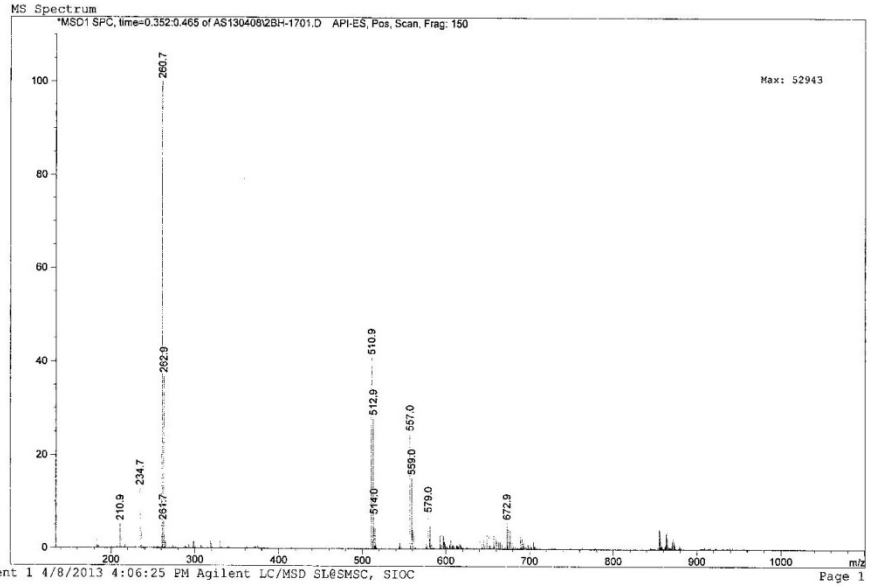

−79.804
M-Cl-H2O-LJ-F
0 −20 −40 −60 −80 −100 −120 −140 −160 −180 ppm



Injection Date : 4/8/2013 3:30:31 PM Seq. Line : 17
Sample Name : LJ-3 Location : P2-B-08
Acq. Operator : Agilent LC/MSD SL@SMSC, SIOC Inj : 1
Inj Volume : 0.1 µl
Acq. Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:09:35 PM by Agilent LC/MSD SL@SMSC, SIOC
Analysis Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:33:25 PM by Agilent LC/MSD SL@SMSC, SIOC
(modified after loading)
Test

LJ-15

MS Spectrum



Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1308

Sample Serial Number: LJ-15

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 579.0315 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	6
F	18.998403	1	3
Cl	34.968853	1	2
Na	22.989770	1	1

Additional Search Restrictions:

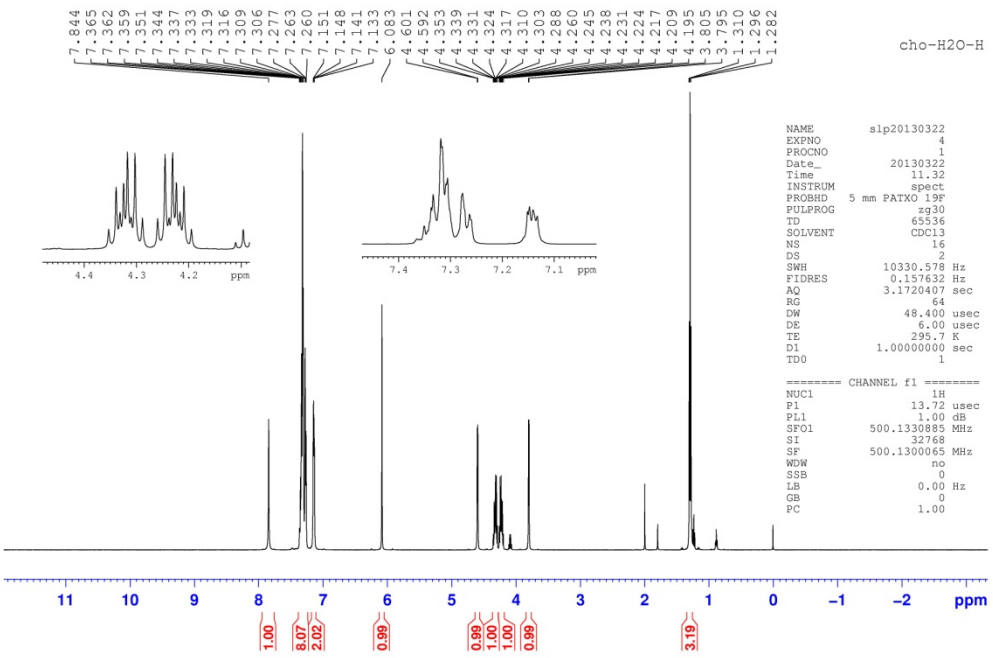
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

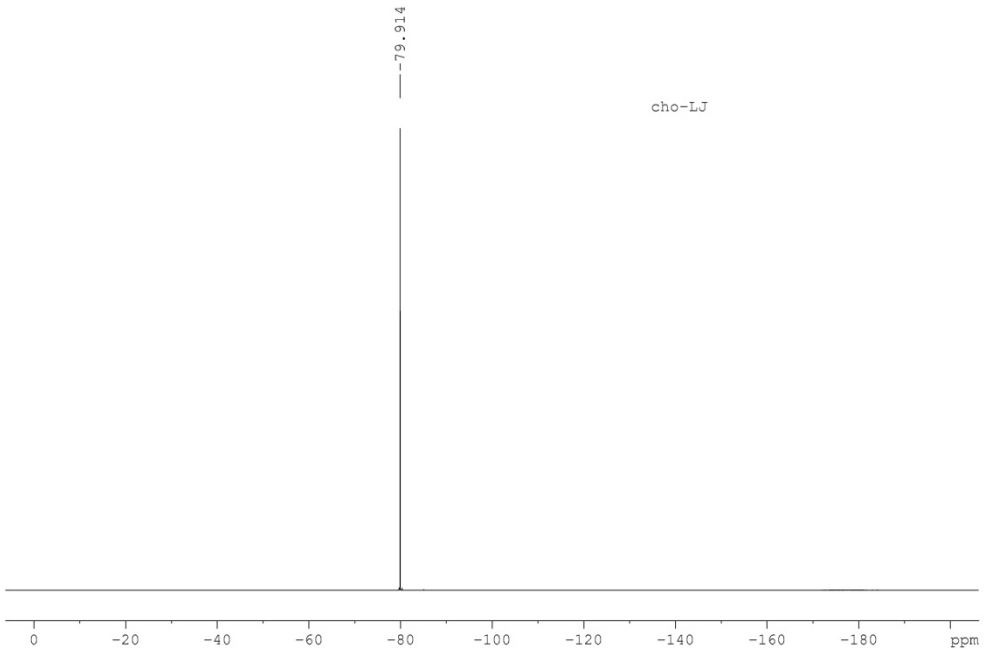
Search Results:

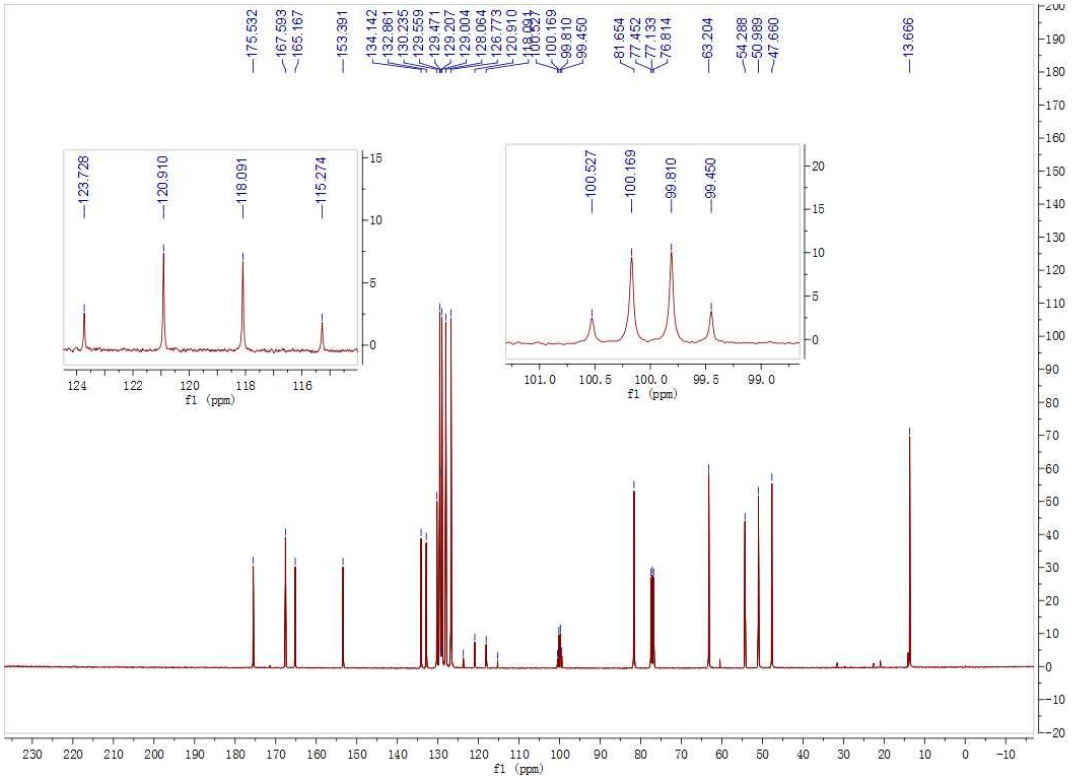
Number of Hits = 6

m/z	Delta m/z	DBE	Formula
579.03185	-0.00035	29.5	$C_{33}H_{11}N_2O_3F_2ClNa^{+1}$
579.03080	0.00070	17.5	$C_{24}H_{17}N_2O_6F_3Cl_2Na^{+1}$
579.03071	0.00079	33.0	$C_{36}H_{10}N_2O_2FClNa^{+1}$
579.03254	-0.00104	28.5	$C_{35}H_{15}O_2FCl_2Na^{+1}$
579.03299	-0.00149	26.0	$C_{30}H_{12}N_2O_4F_3ClNa^{+1}$
579.02966	0.00184	21.0	$C_{27}H_{16}N_2O_5F_2Cl_2Na^{+1}$

5d


7.844
7.365
7.362
7.359
7.351
7.344
7.337
7.333
7.319
7.316
7.309
7.306
7.277
7.263
7.260
7.151
7.148
7.141
7.133
6.083
4.601
4.592
4.353
4.339
4.331
4.324
4.317
4.310
4.303
4.288
4.260
4.245
4.238
4.231
4.224
4.217
4.209
4.195
3.805
3.795
1.310
1.296
1.282
cho-H2O-H
NAME slp20130322
EXPNO 4
PROCNO 1
Date_ 20130322
Time 11.32
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 64
DW 48.400 usec
DE 6.00 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300065 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4.4 4.3 4.2 ppm
7.4 7.3 7.2 7.1 ppm
11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 ppm
1.00
8.07
2.02
0.99
0.99
1.00
1.00
0.99
3.19


-79.914
cho-LJ
0 -20 -40 -60 -80 -100 -120 -140 -160 -180 ppm



Print of window 80: MS Spectrum

Injection Date : 4/8/2013 3:25:47 PM
Sample Name : LWJ-5
Acq. Operator : Agilent LC/MSD SL@SMSC, SIOC

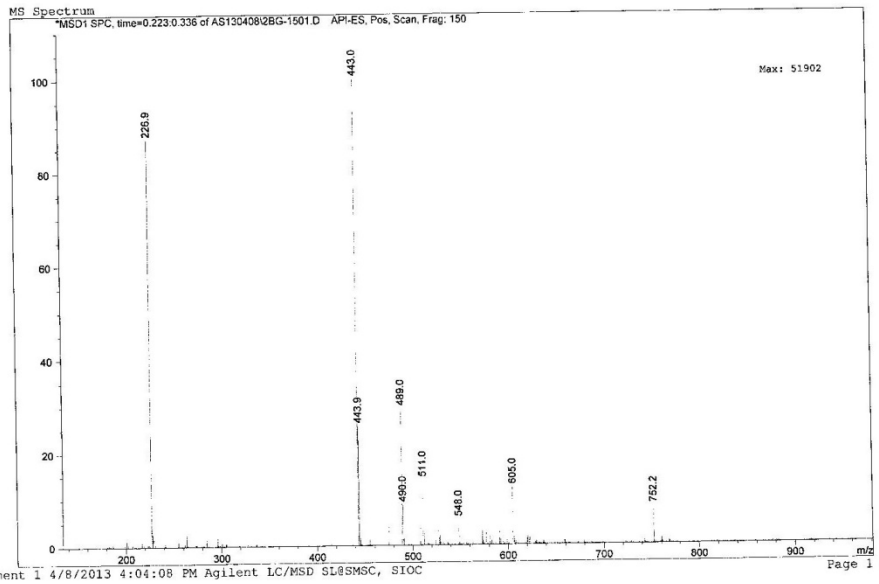
Seq. Line : 15
Location : P2-B-07
Inj : 1
Inj Volume : 0.1 µl

Acq. Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:09:35 PM by Agilent LC/MSD SL@SMSC, SIOC
Analysis Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:33:25 PM by Agilent LC/MSD SL@SMSC, SIOC
(modified after loading)

Test

CHO-H2O

LJ-16



Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1309

Sample Serial Number: LJ-16

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 511.1089 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	6
F	18.998403	1	3
Na	22.989770	1	1

Additional Search Restrictions:

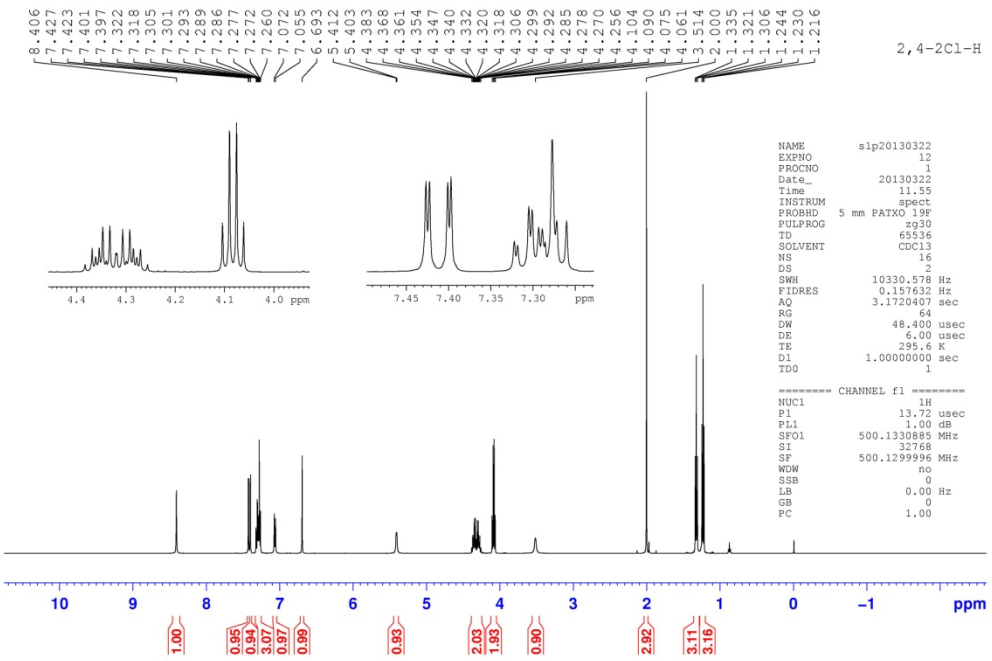
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

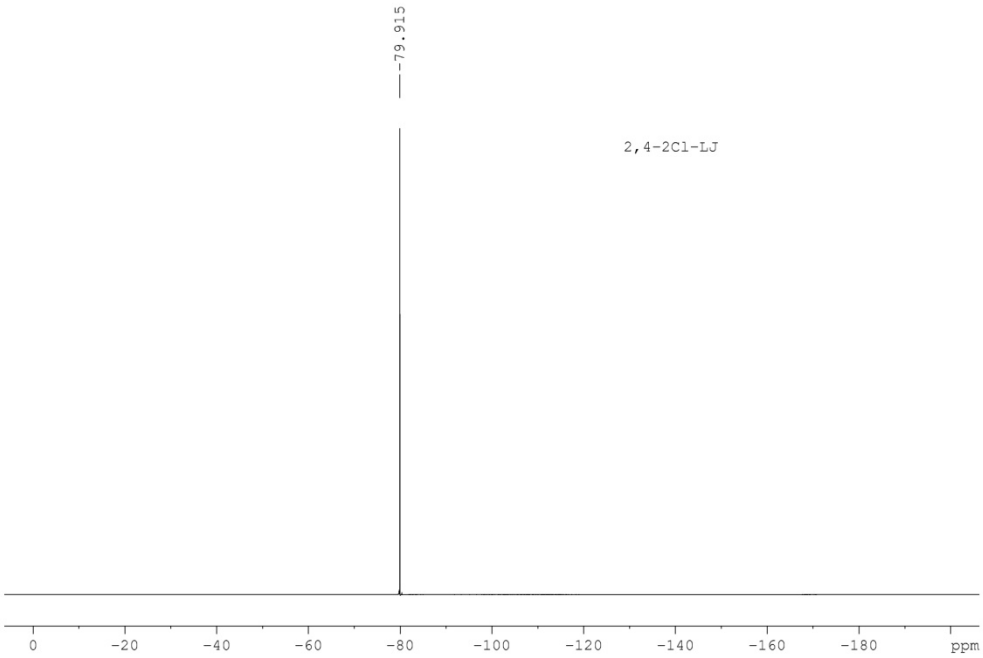
Search Results:

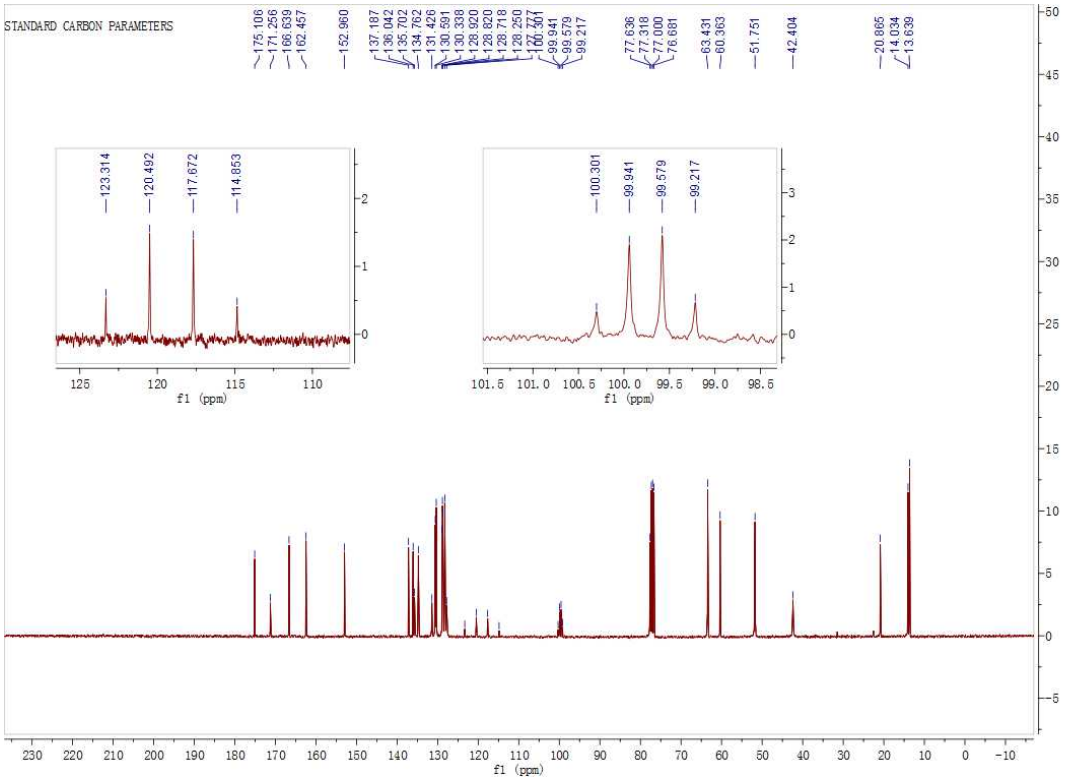
Number of Hits = 3

m/z	Delta m/z	DBE	Formula
511.10874	0.00016	16.5	$C_{24}H_{19}N_2O_6F_3Na^{+1}$
511.10760	0.00130	20.0	$C_{27}H_{18}N_2O_5F_2Na^{+1}$
511.11048	-0.00158	27.5	$C_{35}H_{17}O_2FNa^{+1}$

5e


8.406
7.427
7.423
7.401
7.397
7.322
7.318
7.305
7.301
7.293
7.289
7.286
7.277
7.272
7.260
7.072
7.055
6.693
5.412
5.403
4.383
4.368
4.361
4.354
4.347
4.340
4.332
4.320
4.318
4.306
4.299
4.292
4.285
4.278
4.270
4.256
4.104
4.090
4.075
4.061
3.514
2.000
1.335
1.321
1.306
1.244
1.230
1.216
2,4-2Cl-H
NAME slp20130322
EXPNO 12
PROCNO 1
Date_ 20130322
Time 11.55
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 64
DW 48.400 usec
DE 6.00 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1299996 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
4.4 4.3 4.2 4.1 4.0 ppm
7.45 7.40 7.35 7.30 ppm
10 9 8 7 6 5 4 3 2 1 0 −1 ppm
1.00
0.95
0.94
3.07
0.97
0.99
0.93
2.03
1.93
0.90
2.92
3.11
3.16


−79.915
2,4-2Cl-LJ
0 −20 −40 −60 −80 −100 −120 −140 −160 −180 ppm



Print of window 80: MS Spectrum

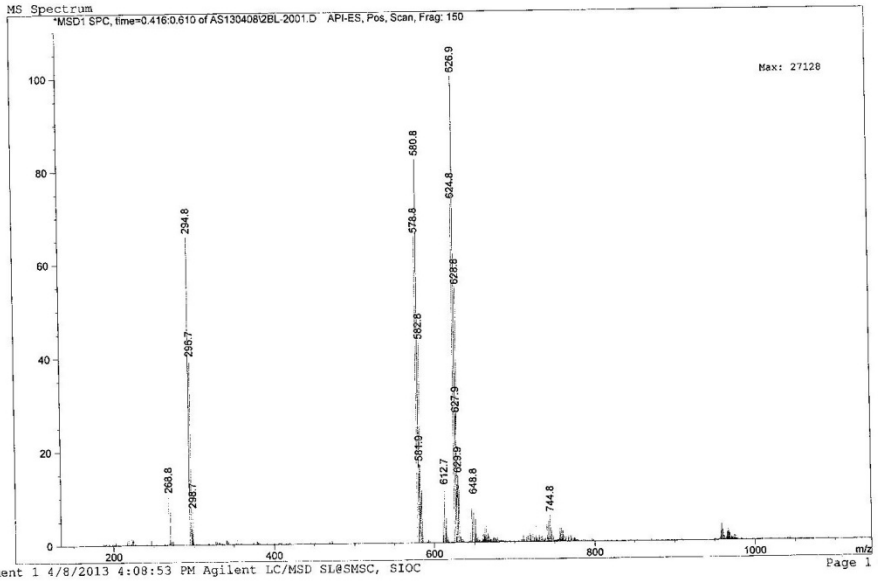
Injection Date : 4/8/2013 3:37:38 PM
Sample Name : LJ-8
Acq. Operator : Agilent LC/MSD SL@SMSC, SIOC
Seq. Line : 20
Location : P2-B-12
Inj : 1
Inj Volume : 0.1 µl

Acq. Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:09:35 PM by Agilent LC/MSD SL@SMSC, SIOC
Analysis Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:33:25 PM by Agilent LC/MSD SL@SMSC, SIOC
(modified after loading)

Test

2,4-Cl-Ph

LJ-17



Instrument 1 4/8/2013 4:08:53 PM Agilent LC/MSD SL@SMSC, SIOC

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1310

Sample Serial Number: LJ-17

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 646.9536 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	6
F	18.998403	1	3
Cl	34.968853	2	4
Na	22.989770	1	1

Additional Search Restrictions:

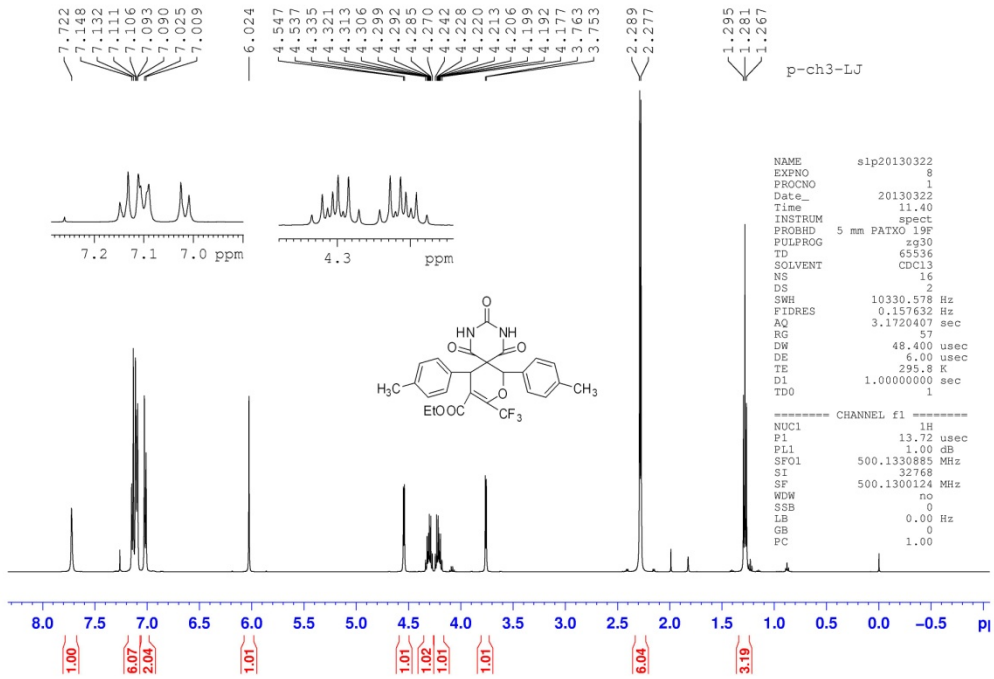
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

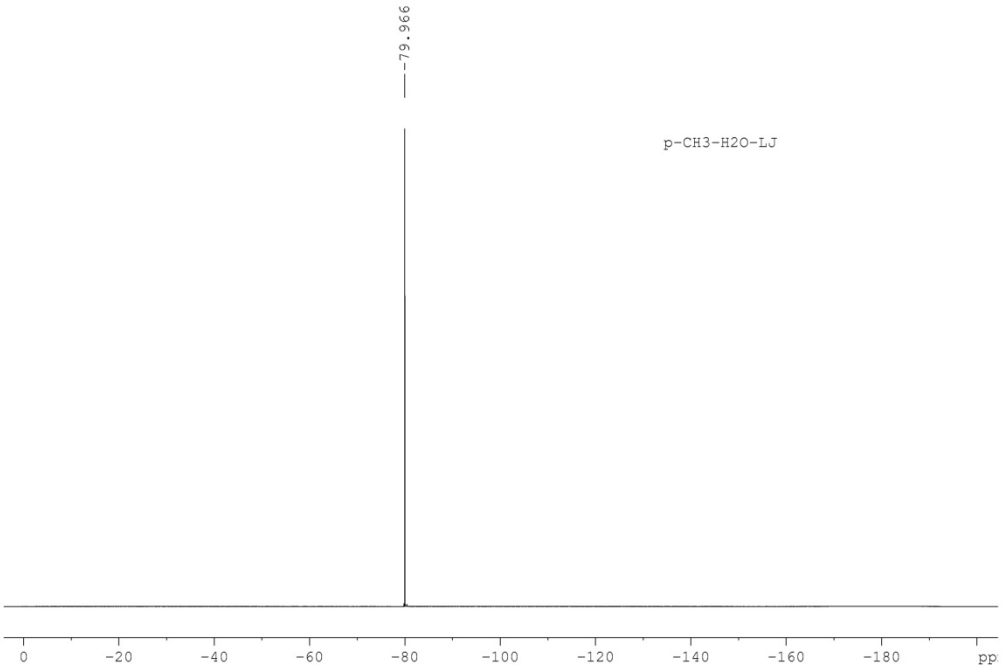
Search Results:

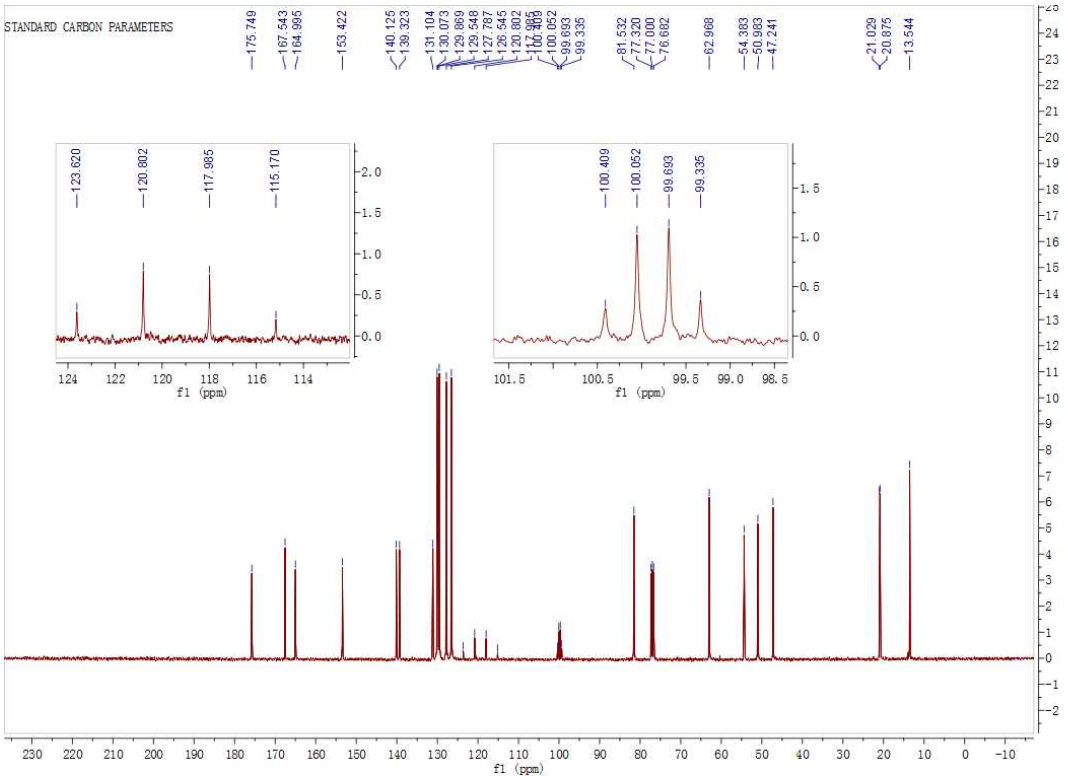
Number of Hits = 10

m/z	Delta m/z	DBE	Formula
646.95342	0.00018	34.5	$C_{36}H_6NO_4F_2Cl_2Na^{+1}$
646.95390	-0.00030	30.5	$C_{33}H_9N_2O_3F_2Cl_3Na^{+1}$
646.95286	0.00074	18.5	$C_{24}H_{15}N_2O_6F_3Cl_4Na^{+1}$
646.95276	0.00084	34.0	$C_{36}H_8N_2O_2FCl_3Na^{+1}$
646.95456	-0.00096	31.0	$C_{33}H_7NO_5F_3Cl_2Na^{+1}$
646.95459	-0.00099	29.5	$C_{35}H_{13}O_2FCl_4Na^{+1}$
646.95227	0.00133	38.0	$C_{39}H_5NO_3FCl_2Na^{+1}$
646.95495	-0.00135	42.5	$C_{42}H_3N_2FCl_2Na^{+1}$
646.95505	-0.00145	27.0	$C_{30}H_{10}N_2O_4F_3Cl_3Na^{+1}$
646.95171	0.00189	22.0	$C_{27}H_{14}N_2O_5F_2Cl_4Na^{+1}$

5f


7.722
7.148
7.132
7.111
7.106
7.093
7.090
7.025
7.009
6.024
4.547
4.537
4.335
4.321
4.313
4.306
4.299
4.292
4.285
4.270
4.242
4.228
4.220
4.213
4.206
4.199
4.192
4.177
3.763
3.753
2.289
2.277
1.295
1.281
1.267
p-ch3-LJ
7.2 7.1 7.0 ppm
4.3 ppm
HN
NH
O
O
O
O
H3C
CH3
EtOOC
CF3
NAME s1p20130322
EXPNO 8
PROCNO 1
Date_ 20130322
Time 11.40
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 57
DW 48.400 usec
DE 6.00 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300124 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
1.00
6.07
2.04
1.01
1.01
1.02
1.01
1.01
6.04
3.19


-79.966
p-CH3-H2O-LJ
0 -20 -40 -60 -80 -100 -120 -140 -160 -180



Print of window 80: MS Spectrum

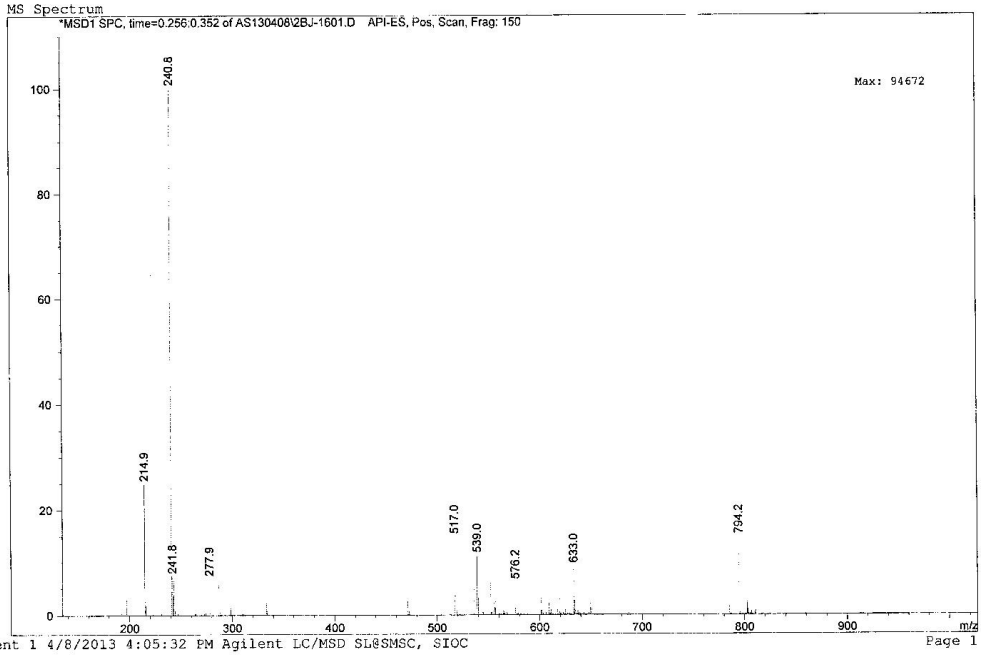
Injection Date : 4/8/2013 3:28:09 PM
Sample Name : LJ-7
Acq. Operator : Agilent LC/MSD SL@SMSC, SIOC
Seq. Line : 16
Location : P2-B-10
Inj : 1
Inj Volume : 0.1 µl

Acq. Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:09:35 PM by Agilent LC/MSD SL@SMSC, SIOC
Analysis Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:33:25 PM by Agilent LC/MSD SL@SMSC, SIOC
(modified after loading)

Test

P-CH3-Ph0 LJ-18

MS Spectrum



Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1311

Sample Serial Number: LJ-18

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 539.1392 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	6
F	18.998403	1	3
Na	22.989770	1	1

Additional Search Restrictions:

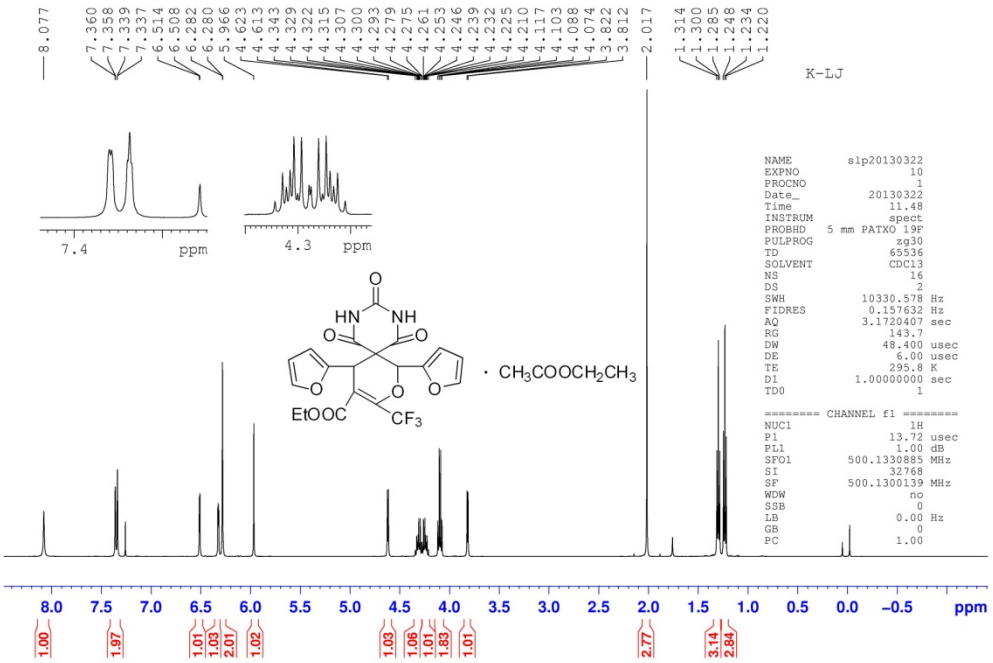
DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

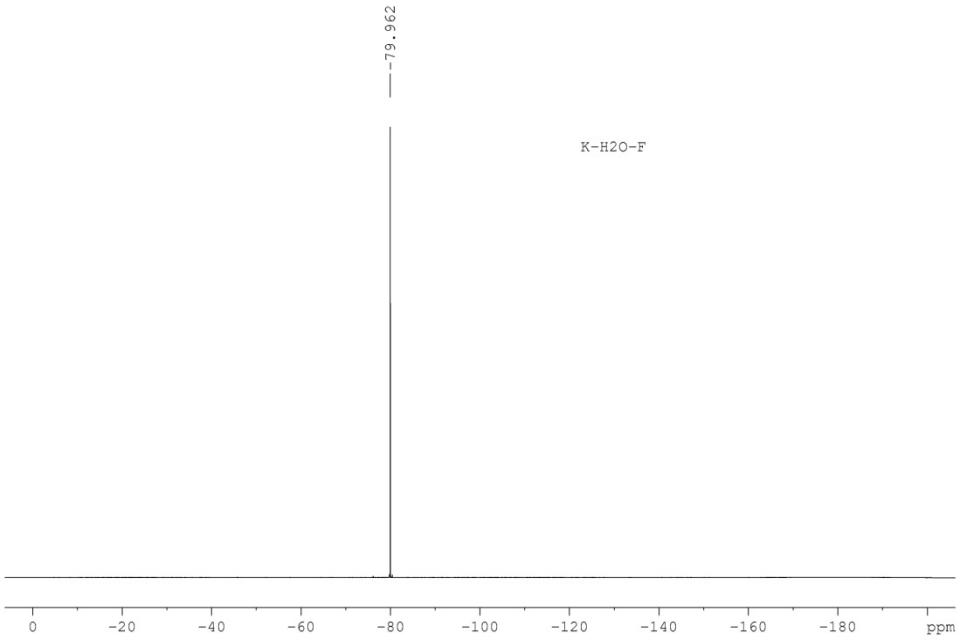
Search Results:

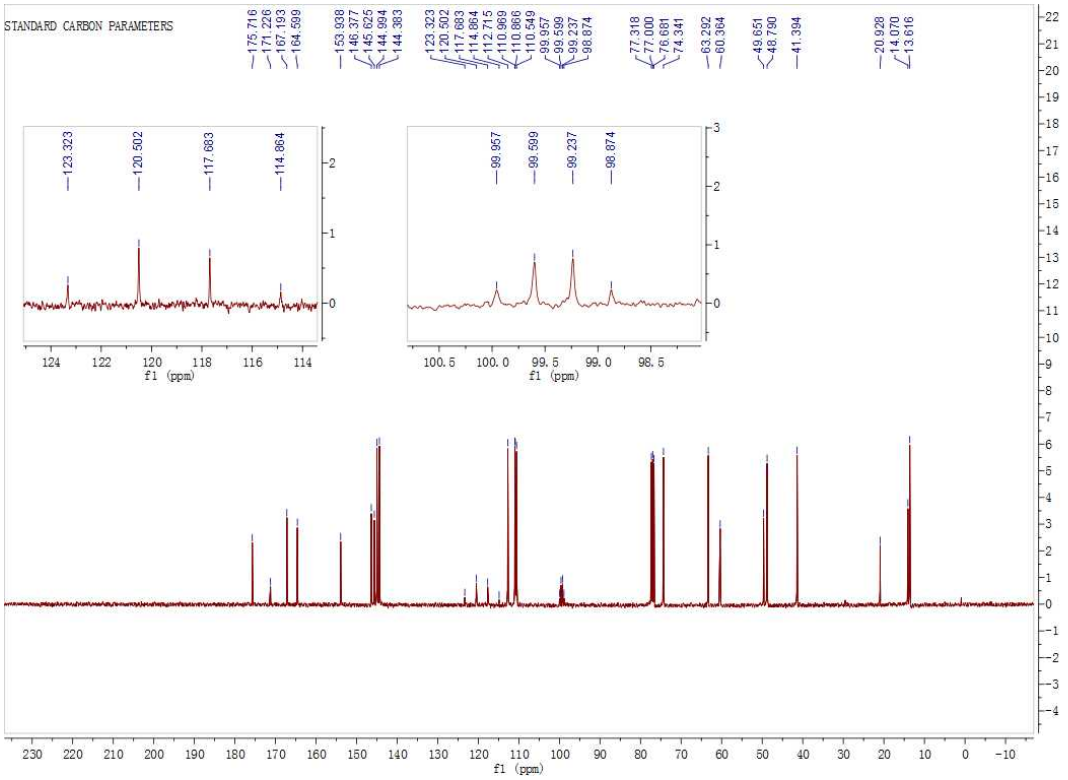
Number of Hits = 3

m/z	Delta m/z	DBE	Formula
539.13890	0.00030	20.0	$C_{29}H_{22}N_2O_5F_2Na^{+1}$
539.14004	-0.00084	16.5	$C_{26}H_{23}N_2O_6F_3Na^{+1}$
539.13776	0.00144	23.5	$C_{32}H_{21}N_2O_4FNa^{+1}$

5g


8.077
7.360
7.358
7.339
7.337
6.514
6.508
6.282
6.280
5.966
4.623
4.613
4.343
4.329
4.322
4.315
4.307
4.300
4.293
4.279
4.275
4.261
4.253
4.246
4.239
4.232
4.225
4.210
4.117
4.103
4.088
4.074
3.822
3.812
2.017
1.314
1.300
1.285
1.248
1.234
1.220
K-LJ
7.4 ppm
4.3 ppm
HN
NH
O
EtOOC
CF3
· CH3COOCH2CH3
NAME s1p20130322
EXPNO 10
PROCNO 1
Date_ 20130322
Time 11.48
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 143.7
DW 48.400 usec
DE 6.00 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.72 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300139 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 ppm
1.00 1.97 1.01 1.03 2.01 1.02 1.03 1.06 1.01 1.83 1.01 2.77 3.14 2.84


−79.962
K-H2O-F
0 −20 −40 −60 −80 −100 −120 −140 −160 −180 ppm



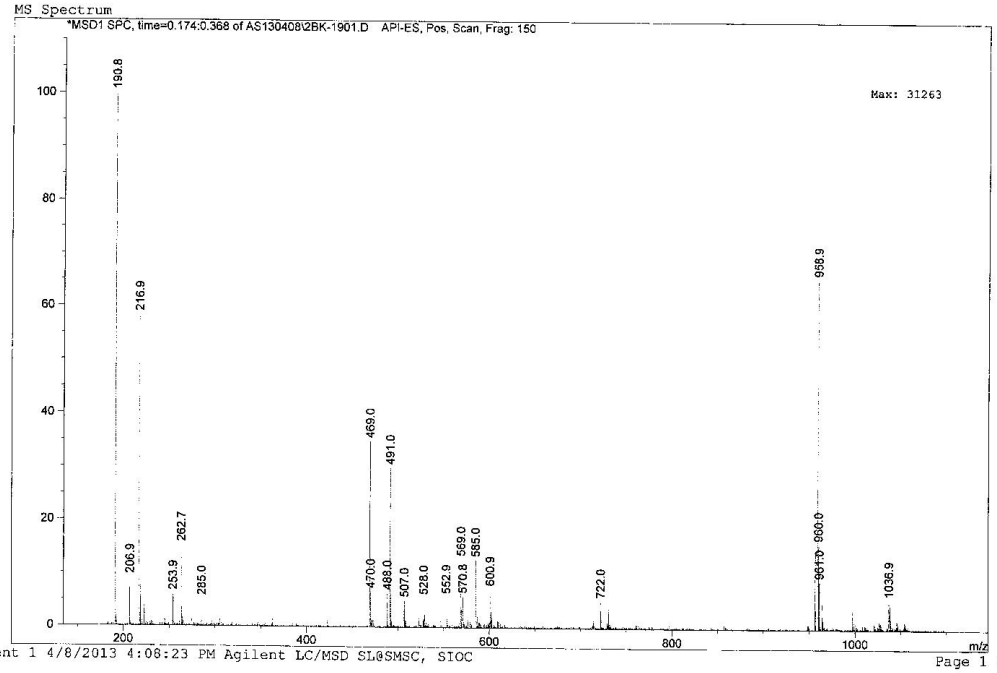
Injection Date : 4/8/2013 3:35:14 PM Seq. Line : 19
Sample Name : LJ-6 Location : P2-B-11
Acq. Operator : Agilent LC/MSD SL@SMSC, SIOC Inj : 1
Inj Volume : 0.1 µl

Acq. Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:09:35 PM by Agilent LC/MSD SL@SMSC, SIOC
Analysis Method : C:\HPCHEM\1\METHODS\AANAL1.M
Last changed : 4/8/2013 3:33:25 PM by Agilent LC/MSD SL@SMSC, SIOC
(modified after loading)

Test

K-HW 19

MS Spectrum



Instrument 1 4/8/2013 4:08:23 PM Agilent LC/MSD SL@SMSC, SIOC

Shanghai Mass Spectrometry Center
Shanghai Institute of Organic Chemistry
Chinese Academic of Sciences
High Resolution MS DATA REPORT

Instrument: IonSpec 4.7 Tesla FTMS

Card Serial Number : I13 1312

Sample Serial Number: LJ-19

Operator : HuaQin Date: 2013/09/16

Operation Mode: MALDI/DHB

Elemental Composition Search Report:

Target Mass:

Target m/z = 491.0668 ± 0.002
Charge = +1

Possible Elements:

Element:	Exact Mass:	Min:	Max:
C	12.000000	0	100
H	1.007825	0	100
N	14.003074	0	2
O	15.994915	0	8
F	18.998403	1	3
Na	22.989770	1	1

Additional Search Restrictions:

DBE Limit Mode = Both Integer and Half-Integer
Minimum DBE = 0

Search Results:

Number of Hits = 4

m/z	Delta m/z	DBE	Formula
491.06727	-0.00047	14.5	$C_{20}H_{15}N_2O_8F_3Na^{+1}$
491.06613	0.00067	18.0	$C_{23}H_{14}N_2O_7F_2Na^{+1}$
491.06542	0.00138	27.5	$C_{32}H_{11}OF_3Na^{+1}$
491.06499	0.00181	21.5	$C_{26}H_{13}N_2O_6FNa^{+1}$