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Combinatorial Discovery of Thermoresponsive Cycloammonium Ionic Liquids

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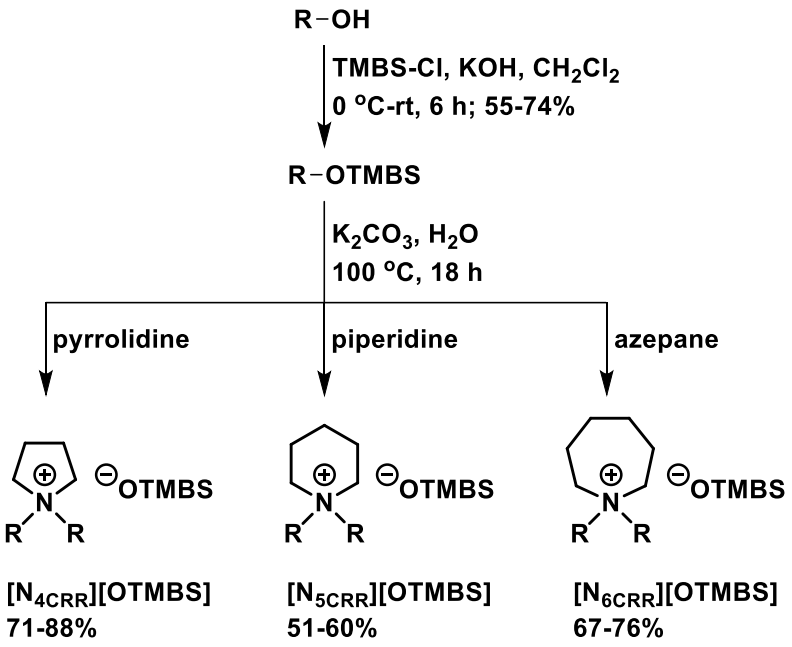
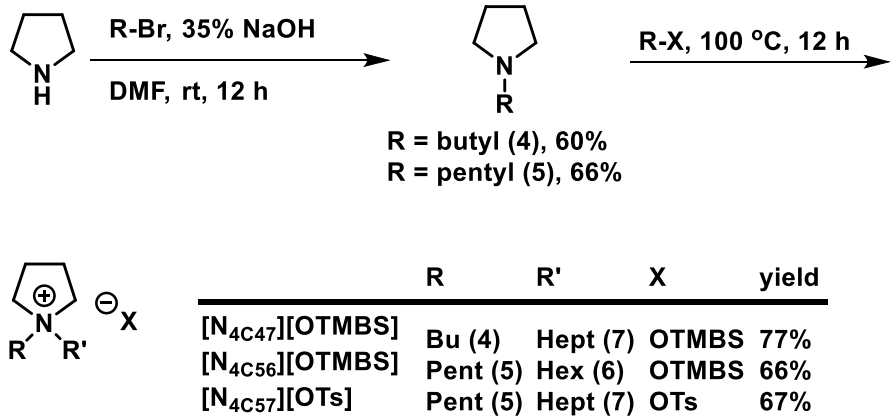


Scheme S1. Synthesis of thermoresponsive cycloammonium ionic liquids [N_{4CRR}][OTMBS], [N_{5CRR}][OTMBS], and [N_{6CRR}][OTMBS].



Scheme S2. Synthesis of cycloammonium ionic liquids [N_{4C56}][OTMBS], [N_{4C47}][OTMBS], and [N_{4C57}][OTs].

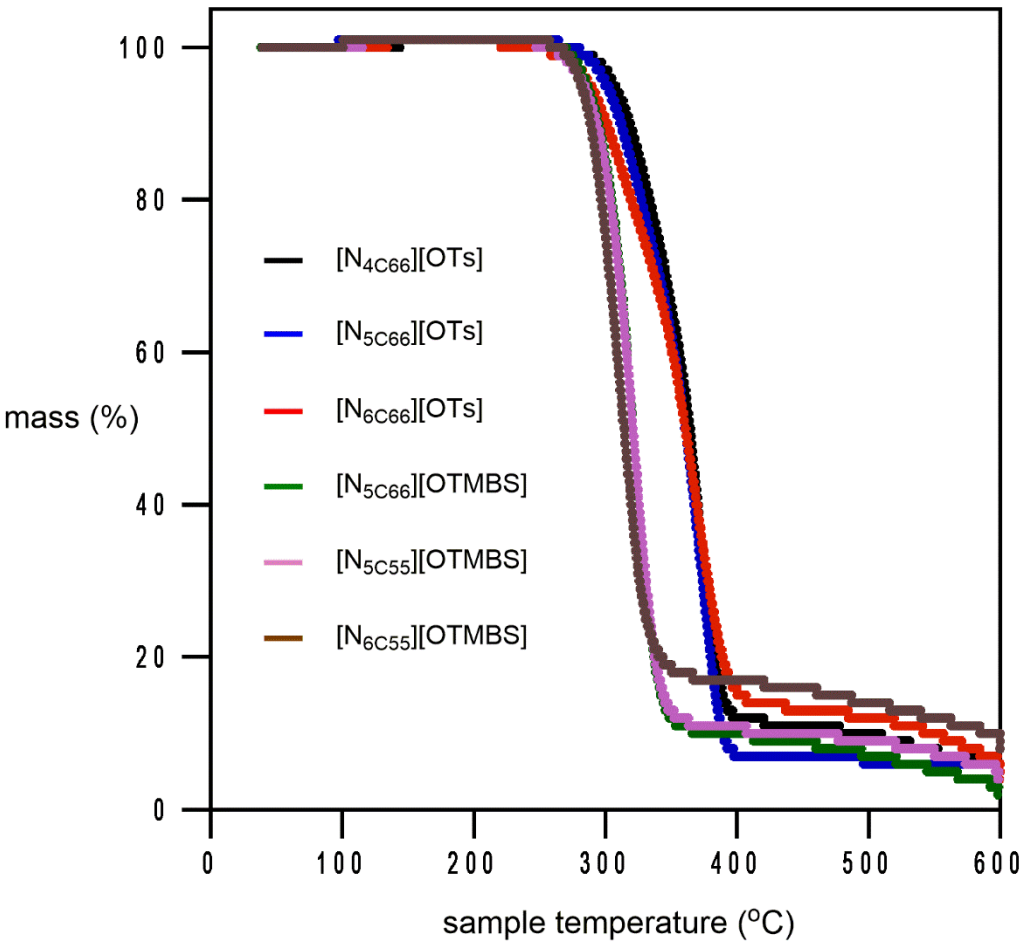
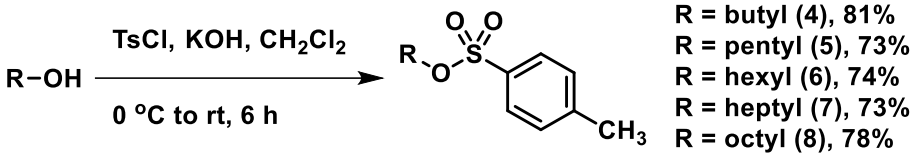


Figure S1. Thermogravimetric analysis (TGA) of six cycloammonium ionic salts that were identified as TILs and measured under nitrogen at a scanning speed of 20 °C min^{-1}. T_{dcp} reflects the temperature at which a weight loss of 10% was observed: 319, 313, 302, 295, 294 and 289 °C for $[N_{4C66}][OTs]$, $[N_{5C66}][OTs]$, $[N_{6C66}][OTs]$, $[N_{5C55}][OTMBS]$, $[N_{5C66}][OTMBS]$, and $[N_{6C55}][OTMBS]$, respectively.

General procedure for synthesis of alkyl 4-methylbenzenesulfonates, R-OTs



To a round-bottomed flask containing an alcohol (1.05 equiv) in dichloromethane was added potassium hydroxide (4 equiv) with stirring at 0 °C for 30 min. To this mixture, a solution of 4-methylbenzenesulfonyl chloride (5.003-9.050 g, 26.24-47.47 mmol) in dichloromethane was added slowly at 0 °C, then to room temperature. The reaction was performed and monitored by TLC until completion (6 h). The resulting solution was diluted with water and extracted with dichloromethane. The combined organic layers were washed with brine. The organic layer was dried over anhydrous sodium sulfate, filtered, concentrated under reduced pressure, and dried under vacuum, affording pure alkyl tosylate as a colorless liquid. When needed, silica gel column chromatography (ethyl acetate : hexane = 1 : 10, v/v) was carried out to afford the pure alkyl tosylate product as colorless liquid.

Butyl 4-methylbenzenesulfonate: 81% isolated yield; colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, *J* = 7.4 Hz, C*H*$_3$CH$_2$, 3H), 1.35 (m, CH$_3$C*H*$_2$, 2H), 1.64 (m, OCH$_2$C*H*$_2$, 2H), 2.45 (s, CH$_3$, 3H), 4.03 (t, *J* = 6.5 Hz, OCH$_2$, 2H), 7.34 (d, *J* = 8.3 Hz, aryl H, 2H), 7.79 (d, *J* = 8.3 Hz, aryl H, 2H).

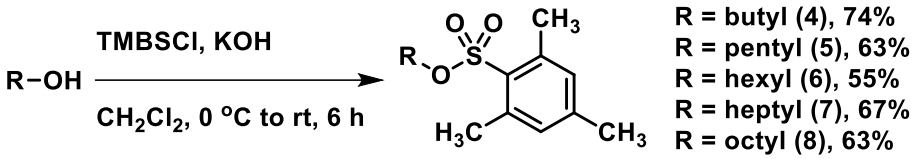
Pentyl 4-methylbenzenesulfonate: 73% isolated yield; colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.85 (t, *J* = 7.0 Hz, C*H*$_3$ CH$_2$, 3H), 1.21-1.33 (m, 2 × CH$_2$, 4H), 1.64 (m, OCH$_2$C*H*$_2$, 2H), 2.45 (s, CH$_3$, 3H), 4.02 (t, *J* = 6.6 Hz, OCH$_2$, 2H), 7.34 (d, *J* = 8.2 Hz, aryl H, 2H), 7.79 (d, *J* = 8.2 Hz, aryl H, 2H).

Hexyl 4-methylbenzenesulfonate: 74% isolated yield; colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.85 (t, *J* = 6.9 Hz, CH$_3$, 3H), 1.16-1.34 (m, 3 × CH$_2$, 6H), 1.63 (m, OCH$_2$C*H*$_2$, 2H), 2.45 (s, CH$_3$, 3H), 4.02 (t, *J* = 6.5 Hz, OCH$_2$, 2H), 7.34 (d, *J* = 8.2 Hz, aryl H, 2H), 7.79 (d, *J* = 8.2 Hz, aryl H, 2H).

Heptyl 4-methylbenzenesulfonate: 73% isolated yield; colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, *J* = 6.9 Hz, CH$_3$, 3H), 1.17-1.33 (m, 4 × CH$_2$, 8H), 1.63 (m, OCH$_2$C*H*$_2$, 2H), 2.45 (s, C*H*$_3$C, 3H), 4.02 (t, *J* = 6.5 Hz, OC*H*$_2$CH$_2$, 2H), 7.34 (d, *J* = 8.1 Hz, aryl H, 2H), 7.79 (d, *J* = 8.1 Hz, aryl H, 2H).

Octyl 4-methylbenzenesulfonate: 78% isolated yield; colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.87 (t, *J* = 7.7 Hz, CH$_3$, 3H), 1.17-1.33 (m, 5 × CH$_2$, 10H), 1.63 (m, OCH$_2$C*H*$_2$, 2H), 2.45 (s, CH$_3$, 3H), 4.02 (t, *J* = 6.5 Hz, OCH$_2$, 2H), 7.34 (d, *J* = 7.8 Hz, aryl H, 2H), 7.79 (d, *J* = 7.8 Hz, aryl H, 2H).

General procedure for synthesis of alkyl 2,4,6-trimethylbenzenesulfonates, R-OTMBS



To a round-bottomed flask containing an aliphatic alcohol (1.05 equiv) in dichloromethane was added potassium hydroxide (4 equiv) with stirring at 0 °C for 30 min. To this mixture, a solution of 2,4,6-trimethylbenzenesulfonyl chloride (4.001-10.062 g, 18.29-46.01 mmol) in dichloromethane was slowly added at 0 °C, then to room temperature. The reaction was carried out for 6 h to reach its completion. The resulting solution was diluted with water and extracted with dichloromethane. The combined organic layers were washed with brine. The organic layer was dried over anhydrous sodium sulfate, filtered, concentrated under reduced pressure, dried in vacuum, and finally affording the desired product as a colorless liquid. When needed, silica gel column chromatography (ethyl acetate : hexane = 1 : 10, v/v) was carried out to afford the pure R-OTMBS product as colorless liquid.

Butyl 2,4,6-trimethylbenzenesulfonate: 74% isolated yield; colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, J = 7.4 Hz, C*H*$_3$CH$_2$, 3H), 1.37 (m, CH$_3$C*H*$_2$, 2H), 1.64 (m, OCH$_2$C*H*$_2$, 2H), 2.31 (s, *p*-CH$_3$, 3H), 2.63 (s, *o*- & *m*-CH$_3$, 6H), 3.97 (t, J = 6.5 Hz, OCH$_2$, 2H), 6.97 (s, aryl H, 2H).

Pentyl 2,4,6-trimethylbenzenesulfonate: 63% isolated yield; colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, J = 7.0 Hz, C*H*$_3$CH$_2$, 3H), 1.23-1.35 (m, 2 × CH$_2$, 4H), 1.66 (m, OCH$_2$C*H*$_2$, 2H), 2.31 (s, *p*-CH$_3$, 3H), 2.63 (s, *o*- & *m*-CH$_3$, 6H), 4.00 (t, J = 6.6 Hz, OCH$_2$, 2H), 6.97 (s, aryl H, 2H).

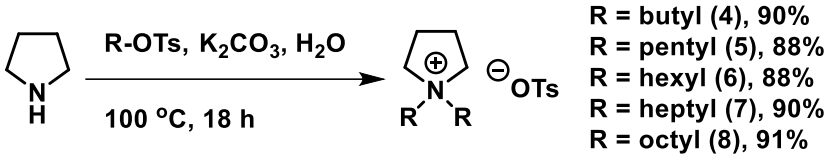
Hexyl 2,4,6-trimethylbenzenesulfonate: 55% isolated yield; colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, J = 7.0 Hz, CH$_3$, 3H), 1.17-1.37 (m, 3 × CH$_2$, 6H), 1.65 (m, OCH$_2$C*H*$_2$, 2H), 2.31 (s, *p*-CH$_3$, 3H), 2.63 (s, *o*- & *m*-CH$_3$, 6H), 4.00 (t, J = 6.6 Hz, OCH$_2$, 2H), 6.97 (s, aryl H, 2H).

Heptyl 2,4,6-trimethylbenzenesulfonate: 67% isolated yield; colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, J = 6.8 Hz, CH$_3$, 3H), 1.18-1.36 (m, 4 × CH$_2$, 8H), 1.65 (m, OCH$_2$C*H*$_2$, 2H), 2.31 (s, *p*-CH$_3$, 3H), 2.63 (s, *o*- & *m*-CH$_3$, 6H), 4.00 (t, J = 6.6 Hz, OCH$_2$, 2H), 6.97 (s, aryl H, 2H).

Octyl 2,4,6-trimethylbenzenesulfonate: 63% isolated yield; colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.87 (t, J = 6.8 Hz, CH$_3$, 3H), 1.18-1.36 (m, 5 × CH$_2$, 10H), 1.65

(m, OCH_2CH_2, 2H), 2.31 (s, *p*-CH_3, 3H), 2.63 (s, *o*- & *m*-CH_3, 6H), 4.00 (t, J = 6.5 Hz, OCH_2, 2H), 6.97 (s, aryl H, 2H).

General procedure for synthesis of *N*,*N*-dialkylpyrrolidinium 4-methylbenzenesulfonates, [N_{4CRR}][OTs]



To a solution of pyrrolidine (0.21-0.26 g, 2.85-3.70 mmol) in water (1 M) was added potassium carbonate (4 equiv) and an alkyl 4-methylbenzenesulfonate (2.05 equiv). The solution mixture was stirred and heated to reflux for 18 h. The reaction progress could be conveniently monitored by TLC. The resulting solution was cooled to room temperature, diluted with water, and extracted with dichloromethane. The organic layer was dried over anhydrous sodium sulfate, filtered, concentrated under reduced pressure, and dried in vacuum. The crude product was exhaustedly washed by diethyl ether to remove residual reactants, then concentrated under reduced pressure, and dried in vacuum, finally affording the desired product as a white solid. When needed, silica gel column chromatography (methanol : dichloromethane = 1 : 10, v/v) was carried out to afford the desired product.

***N*,*N*-Dibutylpyrrolidinium 4-methylbenzenesulfonate, [N_{4C44}][OTs]:** 90% isolated yield; white solid; mp 104-105 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.98 (t, J = 7.3 Hz, 2 × CH_3CH_2, 6H), 1.42 (m, 2 × CH_3CH_2, 4H), 1.64 (m, 2 × NCH_2CH_2, 4H), 2.26 (m, 2 × NCH_2CH_2 ring, 4H), 2.33 (s, *p*-CH_3, 3H), 3.33 (m, 2 × NCH_2, 4H), 3.75 (m, 2 × NCH_2 ring, 4H), 7.13 (d, J = 8.0 Hz, aryl H, 2H), 7.79 (d, J = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.54, 19.57, 21.15, 21.68, 25.21, 59.08, 62.66, 125.82, 128.36, 138.78, 144.29; EI-HRMS *m/z* $[M]^+$ calcd for $C_{12}H_{26}N$ 184.2065, found 184.2068.

***N*,*N*-Dipentylpyrrolidinium 4-methylbenzenesulfonate, [N_{4C55}][OTs]:** 88% isolated yield; white solid; mp 147-148 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.91 (t, J = 6.8 Hz, 2 × CH_3CH_2, 6H), 1.30-1.41 (m, 4 × CH_2, 8H), 1.65 (m, 2 × NCH_2CH_2, 4H), 2.27 (m, 2 × NCH_2CH_2 ring, 4H), 2.33 (s, *p*-CH_3, 3H), 3.32 (m, 2 × NCH_2, 4H), 3.74 (m, 2 × NCH_2 ring, 4H), 7.13 (d, J = 8.0 Hz, aryl H, 2H), 7.79 (d, J = 8.0 Hz, aryl H, 2H);^{13}C NMR (100 MHz, $CDCl_3$) δ 13.75, 21.22, 21.74, 22.18, 23.10, 28.35, 59.25, 62.75, 125.92, 128.43, 138.82, 144.23; EI-HRMS *m/z* $[M]^+$ calcd for $C_{14}H_{30}N$ 212.2378, found 212.2381.

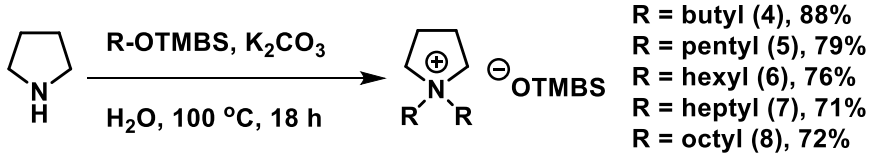
***N*,*N*-Dihexylpyrrolidinium 4-methylbenzenesulfonate, [N_{4C66}][OTs]:** 88% isolated

yield; white solid; mp 169-170 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) 0.89 (t, J = 6.9 Hz, 2 × CH_3, 6H), 1.26-1.40 (m, 6 × CH_2, 12H), 1.64 (m, 2 × NCH_2CH_2, 4H), 2.26 (m, 2 × NCH_2CH_2 ring, 4H), 2.33 (s, p-CH_3, 3H), δ 3.31 (m, 2 × NCH_2, 4H), 3.74 (m, 2 × NCH_2 ring, 4H), 7.13 (d, J = 8.0 Hz, aryl H, 2H), 7.79 (d, J = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.77, 21.14, 21.68, 22.29, 23.26, 25.90, 31.13, 59.22, 62.66, 125.83, 128.35, 138.72, 144.32; EI-HRMS *m/z* $[M]^+$ calcd for $C_{16}H_{34}N$ 240.2691, found 240.2693.

***N*,*N*-Diheptylpyrrolidinium 4-methylbenzenesulfonate, [N_{4C77}][OTs]:** 90% isolated yield; white solid; mp 166-167 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, J = 6.9 Hz, 2 × CH_3, 6H), 1.20-1.40 (m, 8 × CH_2, 16H), 1.64 (m, 2 × NCH_2CH_2, 4H), 2.23 (m, 2 × NCH_2CH_2 ring, 4H), 2.33 (s, p-CH_3, 3H), 3.30 (m, 2 × NCH_2, 4H), 3.73 (m, 2 × NCH_2 ring, 4H), 7.13 (d, J = 8.0 Hz, aryl H, 2H), 7.79 (d, J = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.92, 21.17, 21.70, 22.38, 23.33, 26.22, 28.72, 31.44, 59.22, 62.69, 125.86, 128.37, 138.73, 144.31; EI-HRMS *m/z* $[M]^+$ calcd for $C_{18}H_{38}N$ 268.3004, found 268.3009.

***N*,*N*-Dioctylpyrrolidinium 4-methylbenzenesulfonate, [N_{4C88}][OTs]:** 91% isolated yield; white solid; mp 168-169 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, J = 6.8 Hz, 2 × CH_3, 6H), 1.20-1.41 (m, 10 × CH_2, 20H), 1.65 (m, 2 × NCH_2CH_2, 4H), 2.27 (m, 2 × NCH_2CH_2 ring, 4H), 2.33 (s, p-CH_3, 3H), 3.32 (m, 2 × NCH_2, 4H), 3.75 (m, 2 × NCH_2 ring, 4H), 7.13 (d, J = 8.0 Hz, aryl H, 2H), 7.80 (d, J = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.94, 21.15, 21.69, 22.48, 23.31, 26.24, 28.93, 29.00, 31.55, 59.21, 62.69, 125.84, 128.37, 138.73, 144.27; EI-HRMS *m/z* $[M]^+$ calcd for $C_{20}H_{42}N$ 296.3317, found 296.3319.

General procedure for synthesis of *N*,*N*-dialkylpyrrolidinium 2,4,6-trimethylbenzenesulfonates, [N_{4CRR}][OTMBS]



To a solution of pyrrolidine (0.19-0.26 g, 2.70-3.63 mmol) in water (1 M) was added potassium carbonate (4 equiv) and an alkyl 2,4,6-trimethylbenzenesulfonate (2.05 equiv). The solution mixture was stirred and heated to reflux for 18 h. The reaction progress could be conveniently monitored by TLC. After cooling to room temperature, the resulting solution was diluted with water and extracted with dichloromethane three times. The organic layer was dried over anhydrous sodium sulfate, filtered,

concentrated under reduced pressure, and dried in vacuum. The crude product was washed by diethyl ether to remove residual reactants, then concentrated under reduced pressure, and dried in vacuum, finally affording the desired product as white solid. When needed, silica gel column chromatography (methanol : dichloromethane = 1 : 10, v/v) was carried out to afford the desired product.

***N*,*N*-Dibutylpyrrolidinium 2,4,6-trimethylbenzenesulfonate, [N_{4C44}][OTMBS]:** 88% isolated yield; white solid; mp 87-88 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, *J* = 7.3 Hz, 2 × C*H*$_3$CH$_2$, 6H), 1.42 (m, 2 × CH$_3$C*H*$_2$, 4H), 1.65 (m, 2 × NCH$_2$C*H*$_2$, 4H), 2.21 (s, *p*-CH$_3$, 3H), 2.27 (m, 2 × NCH$_2$C*H*$_2$ ring, 4H), 2.70 (s, *o*- & *m*-CH$_3$, 6H), 3.34 (m, 2 × NCH$_2$, 4H), 3.77 (m, 2 × NCH$_2$ ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.55, 19.59, 20.63, 21.66, 23.04, 25.23, 58.98, 62.62, 130.30, 136.71, 137.34, 141.59; EI-HRMS *m/z* $[M]^+$ calcd for $C_{12}H_{26}N$ 184.2065, found 184.2067.

***N*,*N*-Dipentylpyrrolidinium 2,4,6-trimethylbenzenesulfonate, [N_{4C55}][OTMBS]:** 79% isolated yield; white solid; mp 66-67 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.91 (t, *J* = 6.8 Hz, 2 × CH$_3$, 6H), 1.30-1.41 (m, 4 × CH$_2$, 8H), 1.65 (m, 2 × NCH$_2$C*H*$_2$, 4H), 2.21 (s, *p*-CH$_3$, 3H), 2.27 (m, 2 × NCH$_2$C*H*$_2$ ring, 4H), 2.70 (s, *o*- & *m*-CH$_3$, 6H), 3.32 (m, 2 × NCH$_2$, 4H), 3.75 (m, 2 × NCH$_2$ ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.67, 20.60, 21.65, 22.09, 22.97, 23.03, 28.23, 59.10, 62.59, 130.29, 136.68, 137.31, 141.58; EI-HRMS *m/z* $[M]^+$ calcd for $C_{14}H_{30}N$ 212.2378, found 212.2374.

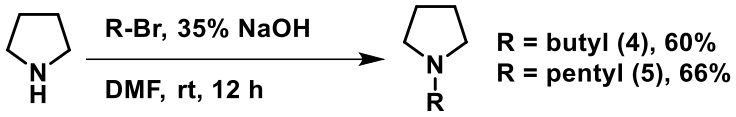
***N*,*N*-Dihexylpyrrolidinium 2,4,6-trimethylbenzenesulfonate, [N_{4C66}][OTMBS]:** 76% isolated yield; white solid; mp 59-60 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 2 × CH$_3$, 6H), 1.24-1.41 (m, 6 × CH$_2$, 12H), 1.64 (m, 2 × NCH$_2$C*H*$_2$, 4H), 2.21 (s, *p*-CH$_3$, 3H), 2.27 (m, 2 × NCH$_2$C*H*$_2$ ring, 4H), 2.70 (s, *o*- & *m*-CH$_3$, 6H), 3.31 (m, 2 × NCH$_2$, 4H), 3.75 (m, 2 × NCH$_2$ ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.75, 20.59, 21.64, 22.27, 23.04, 23.23, 25.87, 31.11, 59.12, 62.60, 130.29, 136.67, 137.30, 141.54; EI-HRMS *m/z* $[M]^+$ calcd for $C_{16}H_{34}N$ 240.2691, found 240.2695.

***N*,*N*-Diheptylpyrrolidinium 2,4,6-trimethylbenzenesulfonate, [N_{4C77}][OTMBS]:** 71% isolated yield; white solid; mp 56-57 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.9 Hz, 2 × CH$_3$, 6H), 1.21-1.40 (m, 8 × CH$_2$, 16H), 1.65 (m, 2 × NCH$_2$C*H*$_2$, 4H), 2.21 (s, *p*-CH$_3$, 3H), 2.27 (m, 2 × NCH$_2$C*H*$_2$ ring, 4H), 2.70 (s, *o*- & *m*-CH$_3$, 6H), 3.32 (m, 2 × NC*H*$_2$, 4H), 3.76 (m, 2 × NC*H*$_2$ ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.88, 20.58, 21.63, 22.32, 23.03, 23.25, 26.14, 28.66, 31.39, 59.11, 62.61, 130.29, 136.66, 137.32, 141.46; EI-HRMS *m/z* $[M]^+$ calcd for $C_{18}H_{38}N$ 268.3004, found 268.2998.

***N*,*N*-Dioctylpyrrolidinium 2,4,6-trimethylbenzenesulfonate, [N_{4C88}][OTMBS]:** 72% isolated yield; white solid; mp 51-52 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 2 × CH$_3$, 6H), 1.20-1.41 (m, 10 × CH$_2$, 20H), 1.64 (m, 2 × NCH$_2$C*H*$_2$, 4H),

2.21 (s, *p*-CH_3, 3H), 2.27 (m, 2 × NCH_2CH_2 ring, 4H), 2.70 (s, *o*- & *m*-CH_3, 6H), 3.32 (m, 2 × NCH_2, 4H), 3.76 (m, 2 × NCH_2 ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.92, 20.60, 21.64, 22.46, 23.04, 23.26, 26.19, 28.90, 28.97, 31.51, 59.10, 62.61, 130.29, 136.67, 137.29, 141.50; EI-HRMS *m/z* $[M]^+$ calcd for $C_{20}H_{42}N$ 296.3317, found 296.3310.

Synthesis of *N*-butylpyrrolidine and *N*-pentylpyrrolidine

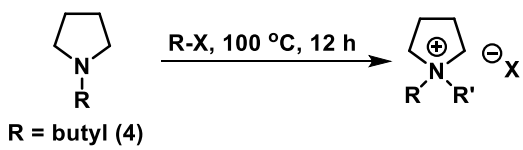


To a round-bottomed flask containing pyrrolidine (0.409 g, 5.75 mmol for the reaction with butylbromide; 1.012 g, 14.23 mmol for the reaction with pentylbromide) was added 35% aqueous sodium hydroxide (0.85 and 2.1 mL, respectively) in dimethylformamide (1.3 M) with stirring for 1 h at room temperature. To the solution mixture, 1-bromoalkane (1.3 equiv) was then added slowly. The reaction was carried out at room temperature for 12 h. The progress of the reaction could be conveniently monitored by TLC. The resulting solution was diluted with water and extracted with ethyl acetate three times. The combined organic layers were washed with water. The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure, finally affording the desired products.

***N*-Butylpyrrolidine:** 60% isolated yield; yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.92 (t, *J* = 7.3 Hz, CH_3, 3H), 1.34 (m, CH_3CH_2, 2H), 1.51 (m, NCH_2CH_2, 2H), 1.78 (m, 2 × NCH_2CH_2 ring, 4H), 2.43 (m, NCH_2, 2H), 2.50 (m, 2 × NCH_2 ring, 4H).

***N*-Pentylpyrrolidine:** 66% isolated yield; yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.90 (t, *J* = 6.9 Hz, CH_3, 3H), 1.24-1.38 (m, 2 × CH_2, 4H), 1.52 (m, NCH_2CH_2, 2H), 1.77 (m, 2 × NCH_2CH_2 ring, 4H), 2.41 (m, NCH_2, 2H), 2.48 (m, 2 × NCH_2 ring, 4H).

Synthesis of *N*-butyl-*N*-heptylpyrrolidinium 2,4,6-trimethylbenzenesulfonate [N_{4C47}][OTMBS], *N*-hexyl-*N*-pentylpyrrolidinium 2,4,6-trimethylbenzenesulfonate [N_{4C56}][OTMBS], and *N*-heptyl-*N*-pentylpyrrolidinium 4-methylbenzenesulfonate [N_{4C57}][OTs]



	R	R'	X	yield
[N_{4C47}][OTMBS]	Bu (4)	Hept (7)	OTMBS	77%
[N_{4C56}][OTMBS]	Pent (5)	Hex (6)	OTMBS	66%
[N_{4C57}][OTs]	Pent (5)	Hept (7)	OTs	67%

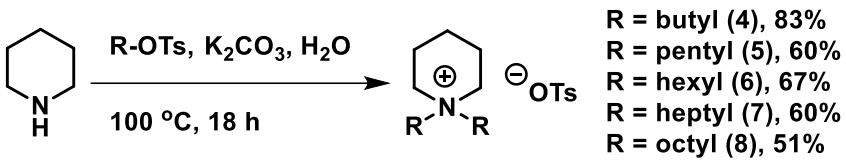
To a round-bottomed flask containing *N*-alkylpyrrolidine (0.061-0.438 g, 0.43-3.44 mmol) was added alkyl 2,4,6-trimethylbenzenesulfonate (1 equiv) for [N_{4C47}][OTMBS] and [N_{4C56}][OTMBS] synthesis, and alkyl 4-methylbenzenesulfonate (1 equiv) for [N_{4C57}][OTs] synthesis. Under neat conditions, the mixture was stirred and heated at 100 °C for 12 h. After cooling to room temperature, the solution was evaporated and the crude product was purified by column chromatography (methanol : dichloromethane = 1 : 10, v/v) to afford the desired pure product as white solid.

***N*-Butyl-*N*-heptylpyrrolidinium 2,4,6-trimethylbenzenesulfonate [N_{4C47}][OTMBS]:** 77% isolated yield; white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.9 Hz, heptyl CH_3, 3H), 0.96 (t, *J* = 7.3 Hz, butyl CH_3, 3H), 1.19-1.35 (m, 4 × CH_2, 8H), 1.39 (m, CH_3CH_2, 2H), 1.61 (m, 2 × NCH_2CH_2, 4H), 2.21 (s, *p*-CH_3, 3H), 2.23 (m, 2 × NCH_2CH_2 ring, 4H), 2.69 (s, *o*- & *m*-CH_3, 6H), 3.29 (m, 2 × NCH_2, 4H), 3.71 (m, 2 × NCH_2 ring, 4H), 6.79 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.76, 14.13, 19.78, 20.83, 21.87, 22.57, 23.26, 23.50, 25.44, 26.40, 28.92, 31.64, 59.20, 59.36, 62.85, 130.53, 136.92, 137.60, 141.68; EI-HRMS *m/z* $[M]^+$ calcd for $C_{15}H_{32}N$ 226.2529, found 226.2531.

***N*-Hexyl-*N*-pentylpyrrolidinium 2,4,6-trimethylbenzenesulfonate [N_{4C56}][OTMBS]:** 66% isolated yield; white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (m, 2 × CH_3, 6H), 1.25-1.40 (m, 5 × CH_2, 10H), 1.62 (m, 2 × NCH_2CH_2, 4H), 2.21 (s, p-CH_3, 3H), 2.24 (m, 2 × NCH_2CH_2 ring, 4H), 2.69 (s, *o*- & *m*-CH_3, 6H), 3.29 (m, 2 × NCH_2, 4H), 3.71 (m, 2 × NCH_2 ring, 4H), 6.79 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.90, 13.98, 20.83, 21.87, 22.33, 22.51, 23.20, 23.20, 23.26, 23.47, 26.11, 28.46, 31.35, 59.35, 62.86, 130.54, 136.92, 137.61, 141.66; EI-HRMS *m/z* $[M]^+$ calcd for $C_{15}H_{32}N$ 226.2529, found 226.2530.

***N*-Heptyl-*N*-pentylpyrrolidinium 4-methylbenzenesulfonate [N_{4C57}][OTs]:** 67% isolated yield; white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (m, 2 × CH_3, 6H), 1.21-1.39 (m, 6 × CH_2, 12H), 1.64 (m, 2 × NCH_2CH_2, 4H), 2.25 (bs, 2 × NCH_2CH_2 ring, 4H), 2.33 (s, *p*-CH_3, 3H), 3.30 (m, 2 × NCH_2, 4H), 3.72 (m, 2 × NCH_2 ring, 4H), 7.13 (d, *J* = 8.0 Hz, aryl H, 2H), 7.78 (d, *J* = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.89, 14.11, 21.34, 21.88, 21.88, 22.30, 22.55, 23.18, 23.48, 26.38, 28.44, 28.89, 31.62, 59.41, 62.85, 126.02, 128.56, 138.95, 144.50; EI-HRMS *m/z* $[M]^+$ calcd for $C_{16}H_{34}N$ 240.2686, found 240.2696.

General procedure for synthesis of *N*,*N*-dialkylpiperidinium 4-methylbenzenesulfonate, [N_{5CRR}][OTs]



To a solution of piperdine (0.285-0.338 g, 3.35-3.97 mmol) in water (1 M) was added potassium carbonate (4 equiv) and alkyl 4-methylbenzenesulfonate (2.05 equiv). The mixture solution was stirred and heated to reflux for 18 h. After cooling to room temperature, the resulting solution was diluted with water and extracted with dichloromethane three times. The organic layer was dried over anhydrous sodium sulfate, filtered, concentrated under reduced pressure, and dried in vacuum. The crude product was washed using diethyl ether to remove residual reactants, concentrated under reduced pressure, and dried in vacuum, finally affording the desired product as white solid.

***N*,*N*-Dibutylpiperidinium 4-methylbenzenesulfonate, [N_{5C44}][OTs]:** 83% isolated yield; white solid; mp 111-112 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, J = 7.2 Hz, 2 × CH_3, 6H), 1.43 (m, 2 × CH_3CH_2, 4H), 1.61 (m, 2 × NCH_2CH_2, 4H), 1.84 (bs, $CH_2CH_2CH_2$ ring, 6H), 2.33 (s, *p*-CH_3, 3H), 3.38 (m, 2 × NCH_2, 4H), 3.64 (bs, 2 × NCH_2 ring, 4H), 7.13 (d, J = 7.6 Hz, aryl H, 2H), 7.81 (d, J = 7.6 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.54, 19.57, 19.69, 20.54, 21.12, 23.33, 57.87, 58.85, 125.85, 128.29, 138.66, 144.42; EI-HRMS *m/z* $[M]^+$ calcd for $C_{13}H_{28}N$ 198.2222, found 198.2222.

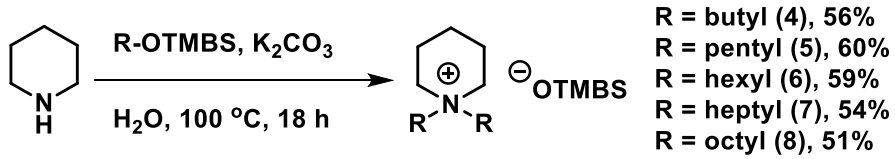
***N*,*N*-Dipentylpiperidinium 4-methylbenzenesulfonate, [N_{5C55}][OTs]:** 60% isolated yield; white solid; mp 114-115 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.92 (t, J = 6.8 Hz, 2 × CH_3, 6H), 1.31-1.42 (m, 4 × CH_2, 8H), 1.62 (m, 2 × NCH_2CH_2, 4H), 1.85 (bs, 2 × $CH_2CH_2CH_2$ ring, 6H), 2.33 (s, *p*-CH_3, 3H), 3.36 (m, 2 × NCH_2, 4H), 3.65 (bs, 2 × NCH_2 ring, 4H), 7.13 (d, J = 8.0 Hz, aryl H, 2H), 7.81 (d, J = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.69, 19.71, 20.54, 21.12, 21.13, 22.10, 28.26, 58.03, 58.84, 125.87, 128.30, 138.65, 144.41; EI-HRMS *m/z* $[M]^+$ calcd for $C_{15}H_{32}N$ 226.2535, found 226.2531.

***N*,*N*-Dihexylpiperidinium 4-methylbenzenesulfonate, [N_{5C66}][OTs]:** 67% isolated yield; white solid; mp 125-126 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, J = 6.7 Hz, 2 × CH_3, 6H), 1.27-1.42 (m, 6 × CH_2, 12H), 1.61 (m, 2 × NCH_2CH_2, 4H), 1.84 (bs, $CH_2CH_2CH_2$ ring, 6H), 2.33 (s, *p*-CH_3, 3H), 3.36 (m, 2 × NCH_2, 4H), 3.64 (bs, 2 × NCH_2 ring, 4H), 7.13 (d, J = 8.0 Hz, aryl H, 2H), 7.81 (d, J = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.76, 19.73, 20.54, 21.13, 21.39, 22.29, 25.91, 31.13, 58.03, 58.85, 125.87, 128.30, 138.64, 144.40; EI-HRMS *m/z* $[M]^+$ calcd for $C_{17}H_{36}N$ 254.2848, found 254.2848.

***N*,*N*-Diheptylpiperidinium 4-methylbenzenesulfonate, [N_{5C77}][OTs]:** 60% isolated yield; white solid; mp 123-124 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, *J* = 6.8 Hz, 2 × CH_3, 6H), 1.21-1.41 (m, 8 × CH_2, 16H), 1.61 (m, 2 × NCH_2CH_2, 4H), 1.84 (bs, $CH_2CH_2CH_2$ ring, 6H), 2.33 (s, *p*-CH_3, 3H), 3.35 (m, 2 × NCH_2, 4H), 3.63 (bs, 2 × NCH_2 ring, 4H), 7.13 (d, *J* = 8.0 Hz, aryl H, 2H), 7.81 (d, *J* = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.89, 19.72, 20.53, 21.12, 21.42, 22.34, 26.19, 28.69, 31.41, 58.01, 58.85, 125.86, 128.29, 138.62, 144.40; EI-HRMS *m/z* $[M]^+$ calcd for $C_{19}H_{40}N$ 282.3161, found 282.3155.

***N*,*N*-Dioctylpiperidinium 4-methylbenzenesulfonate, [N_{5C88}][OTs]:** 51% isolated yield; white solid; mp 125-126 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 2 × CH_3, 6H), 1.20-1.41 (m, 8 × CH_2, 16H), 1.60 (m, 2 × NCH_2CH_2, 4H), 1.85 (bs, $CH_2CH_2CH_2$ ring, 6H), 2.33 (s, *p*-CH_3, 3H), 3.35 (m, 2 × NCH_2, 4H), 3.64 (bs, 2 × NCH_2 ring, 4H), 7.13 (d, *J* = 8.0 Hz, aryl H, 2H), 7.81 (d, *J* = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.94, 19.75, 20.54, 21.15, 21.44, 22.47, 26.25, 28.93, 29.01, 31.54, 58.02, 58.87, 125.88, 128.32, 138.66, 144.34; EI-HRMS *m/z* $[M]^+$ calcd for $C_{21}H_{44}N$ 310.3474, found 310.3470.

General procedure for synthesis of *N*,*N*-dialkylpiperidinium 2,4,6-trimethylbenzenesulfonate, [N_{5CRR}][OTMBS]



To a solution of piperdine (0.308-0.391 g, 3.62-4.59 mmol) in water (1 M) was added potassium carbonate (4 equiv) and an alkyl 2,4,6-trimethylbenzenesulfonate (2.05 equiv). The mixture solution was stirred and heated to reflux for 18 h. After cooling to room temperature, the resulting solution was diluted with water and extracted with dichloromethane three times. The organic layer was dried over anhydrous sodium sulfate, filtered, concentrated under reduced pressure, and dried in vacuum. The crude product was washed using diethyl ether to remove residual reactants, then concentrated under reduced pressure, and dried in vacuum, finally affording the desired product. When needed, product could be further purified by silica gel column chromatography (methanol : dichloromethane = 1 : 10, v/v) to afford the pure product.

***N*,*N*-Dibutylpiperidinium 2,4,6-trimethylbenzenesulfonate, [N_{5C44}][OTMBS]:** 56% isolated yield; white solid; mp 101-102 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, *J* = 7.2 Hz, 2 × CH_3, 6H), 1.43 (m, 2 × CH_3CH_2, 4H), 1.60 (m, 2 × NCH_2CH_2, 4H),

1.84 (bs, $CH_2CH_2CH_2$ ring, 6H), 2.21 (s, *p*-CH_3, 3H), 2.71 (s, *o*- & *m*-CH_3, 6H), 3.38 (m, 2 × NCH_2, 4H), 3.65 (bs, 2 × NCH_2 ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.55, 19.56, 19.70, 20.55, 20.59, 23.03, 23.33, 57.79, 58.85, 130.24, 136.71, 137.27, 141.64; EI-HRMS *m/z* $[M]^+$ calcd for $C_{13}H_{28}N$ 198.2222, found 198.2220.

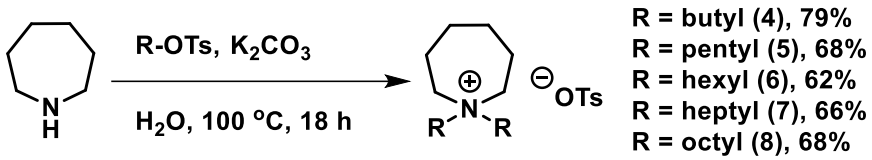
***N*,*N*-Dipentylpiperidinium 2,4,6-trimethylbenzenesulfonate, [N_{5C55}][OTMBS]:** 60% isolated yield; white solid; mp 97-98 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.92 (t, *J* = 6.8 Hz, 2 × CH_3, 6H), 1.30-1.42 (m, 4 × CH_2, 8H), 1.61 (m, 2 × NCH_2CH_2, 4H), 1.84 (bs, $CH_2CH_2CH_2$ ring, 6H), 2.21 (s, *p*-CH_3, 3H), 2.71 (s, *o*- & *m*-CH_3, 6H), 3.36 (m, 2 × NCH_2, 4H), 3.64 (bs, 2 × NCH_2 ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.65, 19.69, 20.53, 20.57, 21.08, 22.08, 23.03, 28.22, 57.92, 58.82, 130.23, 136.69, 137.21, 141.65; EI-HRMS *m/z* $[M]^+$ calcd for $C_{15}H_{32}N$ 226.2535, found 226.2539.

***N*,*N*-Dihexylpiperidinium 2,4,6-trimethylbenzenesulfonate, [N_{5C66}][OTMBS]:** 59% isolated yield; white solid; mp 74-75 ºC; ^{1}H NMR (400M Hz, $CDCl_3$) δ 0.89 (t, *J* = 6.9 Hz, 2 × CH_3, 6H), 1.27-1.41 (m, 6 × CH_2, 12H), 1.60 (m, 2 × NCH_2CH_2, 4H), 1.84 (bs, $CH_2CH_2CH_2$ ring, 6H), 2.21 (s, *p*-CH_3, 3H), 2.71 (s, *o*- & *m*-CH_3, 6H), 3.36 (m, 2 × NCH_2, 4H), 3.64 (bs, 2 × NCH_2 ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.76, 19.76, 20.55, 20.61, 21.40, 22.30, 23.07, 25.91, 31.14, 57.97, 58.88, 130.28, 136.73, 137.27, 141.59; EI-HRMS *m/z* $[M]^+$ calcd for $C_{17}H_{36}N$ 254.2848, found 254.2852.

***N*,*N*-Diheptylpiperidinium 2,4,6-trimethylbenzenesulfonate, [N_{5C77}][OTMBS]:** 54% isolated yield; white solid; mp 75-76 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, *J* = 6.8 Hz, 2 × CH_3, 6H), 1.22-1.40 (m, 8 × CH_2, 16H), 1.60 (m, 2 × NCH_2CH_2, 4H), 1.84 (bs, $CH_2CH_2CH_2$ ring, 6H), 2.21 (s, *p*-CH_3, 3H), 2.71 (s, *o*- & *m*-CH_3, 6H), 3.35 (m, 2 × NCH_2, 4H), 3.64 (bs, 2 × NCH_2 ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.88, 19.71, 20.54, 20.59, 21.39, 22.33, 23.05, 26.16, 28.67, 31.39, 57.94, 58.86, 130.25, 136.70, 137.23, 141.58; EI-HRMS *m/z* $[M]^+$ calcd for $C_{19}H_{40}N$ 282.3161, found 282.3164.

***N*,*N*-Dioctylpiperidinium 2,4,6-trimethylbenzenesulfonate, [N_{5C88}][OTMBS]:** 51% isolated yield; white solid; mp 86-87 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 2 × CH_3, 6H), 1.20-1.41 (m, 10 × CH_2, 20H), 1.60 (m, 2 × NCH_2CH_2, 4H), 1.85 (bs, $CH_2CH_2CH_2$ ring, 6H), 2.21 (s, *p*-CH_3, 3H), 2.71 (s, *o*- & *m*-CH_3, 6H), 3.36 (m, 2 × NCH_2, 4H), 3.65 (bs, 2 × NCH_2 ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.94, 19.76, 20.56, 20.62, 21.43, 22.47, 23.08, 26.24, 28.92, 29.00, 31.53, 57.96, 58.90, 130.29, 136.73, 137.27, 141.55; EI-HRMS *m/z* $[M]^+$ calcd for $C_{21}H_{44}N$ 310.3474, found 310.3468.

General procedure for synthesis of *N*,*N*-dialkylazepanium 4-methylbenzenesulfonate, [N_{6CRR}][OTs]



To a solution of azepane (0.271-0.405 g, 2.73-4.08 mmol) in water (1 M) was added potassium carbonate (4 equiv) and an alkyl 4-methylbenzenesulfonate (2.05 equiv). The mixture solution was stirred and heated to reflux for 18 h. After cooling to room temperature, the resulting solution was diluted with water and extracted with dichloromethane three times. The organic layer was dried over anhydrous sodium sulfate, filtered, concentrated under reduced pressure, and dried in vacuum. The crude product was washed by diethyl ether to remove residual reactants, concentrated under reduced pressure, and dried in vacuum, finally affording the desired product.

***N*,*N*-Dibutylazepanium 4-methylbenzenesulfonate, [N_{6C44}][OTs]:** 79% isolated yield; white solid; mp 125-126 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, J = 7.3 Hz, 2 × C*H*$_3$CH$_2$, 6H), 1.42 (m, 2 × CH$_3$C*H*$_2$, 4H), 1.68 (m, 2 × NCH$_2$CH$_2$C*H*$_2$ ring, 4H), 1.75 (bs, 2 × NCH$_2$C*H*$_2$, 4H), 1.95 (bs, 2 × NCH$_2$C*H*$_2$ ring, 4H), 2.33 (s, *p*-CH$_3$, 3H), 3.37 (m, 2 × NCH$_2$, 4H), 3.57 (m, 2 × NCH$_2$ ring, 4H), 7.13 (d, J = 8.0 Hz, aryl H, 2H), 7.80 (d, J = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.54, 19.61, 21.12, 21.72, 24.24, 27.36, 60.90, 62.75, 125.88, 128.24, 138.57, 144.55; EI-HRMS *m/z* $[M]^+$ calcd for $C_{14}H_{30}N$ 212.2378, found 212.2375.

***N*,*N*-Dipentylazepanium 4-methylbenzenesulfonate, [N_{6C55}][OTs]:** 68% isolated yield; white solid; mp 157-158 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.91 (t, J = 6.8 Hz, 2 × CH$_3$, 6H), 1.31-1.40 (m, 4 × CH$_2$, 8H), 1.67 (m, 2 × NCH$_2$CH$_2$C*H*$_2$ ring, 4H), 1.75 (bs, 2 × NCH$_2$C*H*$_2$, 4H), 1.94 (bs, 2 × NCH$_2$C*H*$_2$ ring, 4H), 2.32 (s, *p*-CH$_3$, 3H), 3.35 (m, 2 × NCH$_2$, 4H), 3.57 (m, 2 × NCH$_2$ ring, 4H), 7.12 (d, J = 8.0 Hz, aryl H, 2H), 7.80 (d, J = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.77, 21.12, 21.75, 22.30, 25.95, 27.35, 31.14, 61.04, 62.75, 125.90, 128.24, 138.53, 144.56; EI-HRMS *m/z* $[M]^+$ calcd for $C_{16}H_{34}N$ 240.2697, found 240.2688.

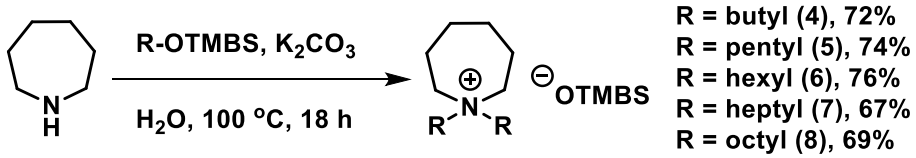
***N*,*N*-Dihexylazepanium 4-methylbenzenesulfonate, [N_{6C66}][OTs]:** 62% isolated yield; white solid; mp 175-176 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, J = 7.0 Hz, 2 × CH$_3$, 6H), 1.27-1.42 (m, 6 × CH$_2$, 12H), 1.67 (m, 2 × NCH$_2$CH$_2$C*H*$_2$ ring, 4H), 1.75 (bs, 2 × NCH$_2$C*H*$_2$, 4H), 1.95 (bs, 2 × NCH$_2$C*H*$_2$ ring, 4H), 2.32 (s, *p*-CH$_3$, 3H), 3.34 (m, 2 × NCH$_2$, 4H), 3.57 (m, 2 × NCH$_2$ ring, 4H), 7.12 (d, J = 8.0 Hz, aryl H, 2H), 7.80 (d, J = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.71, 21.12, 21.74, 22.04,

22.11, 27.35, 27.35 28.30, 61.02, 62.73, 125.90, 128.24, 138.56, 144.55; EI-HRMS *m/z* $[M]^+$ calcd for $C_{18}H_{38}N$ 268.3004, found 268.3003.

***N*,*N*-Diheptylazepanium 4-methylbenzenesulfonate, [N$_{6C77}$][OTs]:** 66% isolated yield; white solid; mp 169-170 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.9 Hz, 2 × CH_3, 6H), 1.21-1.40 (m, 8 × CH_2, 16H), 1.66 (m, 2 × $NCH_2CH_2CH_2$ ring, 4H), 1.75 (bs, 2 × NCH_2CH_2, 4H), 1.95 (bs, 2 × NCH_2CH_2 ring, 4H), 2.32 (s, *p*-CH_3, 3H), 3.34 (m, 2 × NCH_2, 4H), 3.57 (m, 2 × NCH_2 ring, 4H), 7.12 (d, *J* = 8.0 Hz, aryl H, 2H), 7.80 (d, *J* = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.91, 21.14, 21.77, 22.37, 26.24, 27.36, 28.72, 28.72, 31.45, 61.03, 62.77, 125.92, 128.25, 138.53, 144.55; EI-HRMS *m/z* $[M]^+$ calcd for $C_{20}H_{42}N$ 296.3317, found 296.3322.

***N*,*N*-Dioctylazepanium 4-methylbenzenesulfonate, [N$_{6C88}$][OTs]:** 68% isolated yield; white solid; mp 172-173 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.9 Hz, 2 × CH_3, 6H), 1.21-1.40 (m, 10 × CH_2, 20H), 1.66 (m, 2 × $NCH_2CH_2CH_2$ ring, 4H), 1.75 (bs, 2 × NCH_2CH_2, 4H), 1.95 (bs, 2 × NCH_2CH_2 ring, 4H), 2.32 (s, *p*-CH_3, 3H), 3.34 (m, 2 × NCH_2, 4H), 3.57 (m, 2 × NCH_2 ring, 4H), 7.12 (d, *J* = 8.0 Hz, aryl H, 2H), 7.80 (d, *J* = 8.0 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.94, 21.13, 21.74, 22.35, 22.47, 26.28, 27.35, 28.95, 29.01, 31.55, 61.01, 62.75, 125.90, 128.26, 138.55, 144.49; EI-HRMS *m/z* $[M]^+$ calcd for $C_{22}H_{46}N$ 324.3630, found 324.3638.

General procedure for synthesis of *N*,*N*-dialkylazepanium 2,4,6-trimethylbenzenesulfonate, [N$_{6CRR}$][OTMBS]



To a solution of azepane (0.292-0.456 g, 2.94-4.60 mmol) in water (1 M) was added potassium carbonate (4 equiv) and alkyl 2,4,6-trimethylbenzenesulfonate (2.05 equiv). The mixture solution was stirred and heated to reflux for 18 h. After cooling down, the resulting solution was diluted with water and extracted with dichloromethane three times. The organic layer was dried over anhydrous sodium sulfate, filtered, concentrated under reduced pressure, and dried in vacuum. The crude product was washed using diethyl ether to remove residual reactants, then concentrated under reduced pressure, and dried in vacuum, finally affording the desire product.

***N*,*N*-Dibutylazepanium 2,4,6-trimethylbenzenesulfonate, [N$_{6C44}$][OTMBS]:** 72% isolated yield; white solid; mp 102-103ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.98 (t, *J* = 7.3 Hz, 2 × CH_3CH_2, 6H), 1.42 (m, 2 × CH_3CH_2, 4H), 1.66 (m, 2 × $NCH_2CH_2CH_2$ ring,

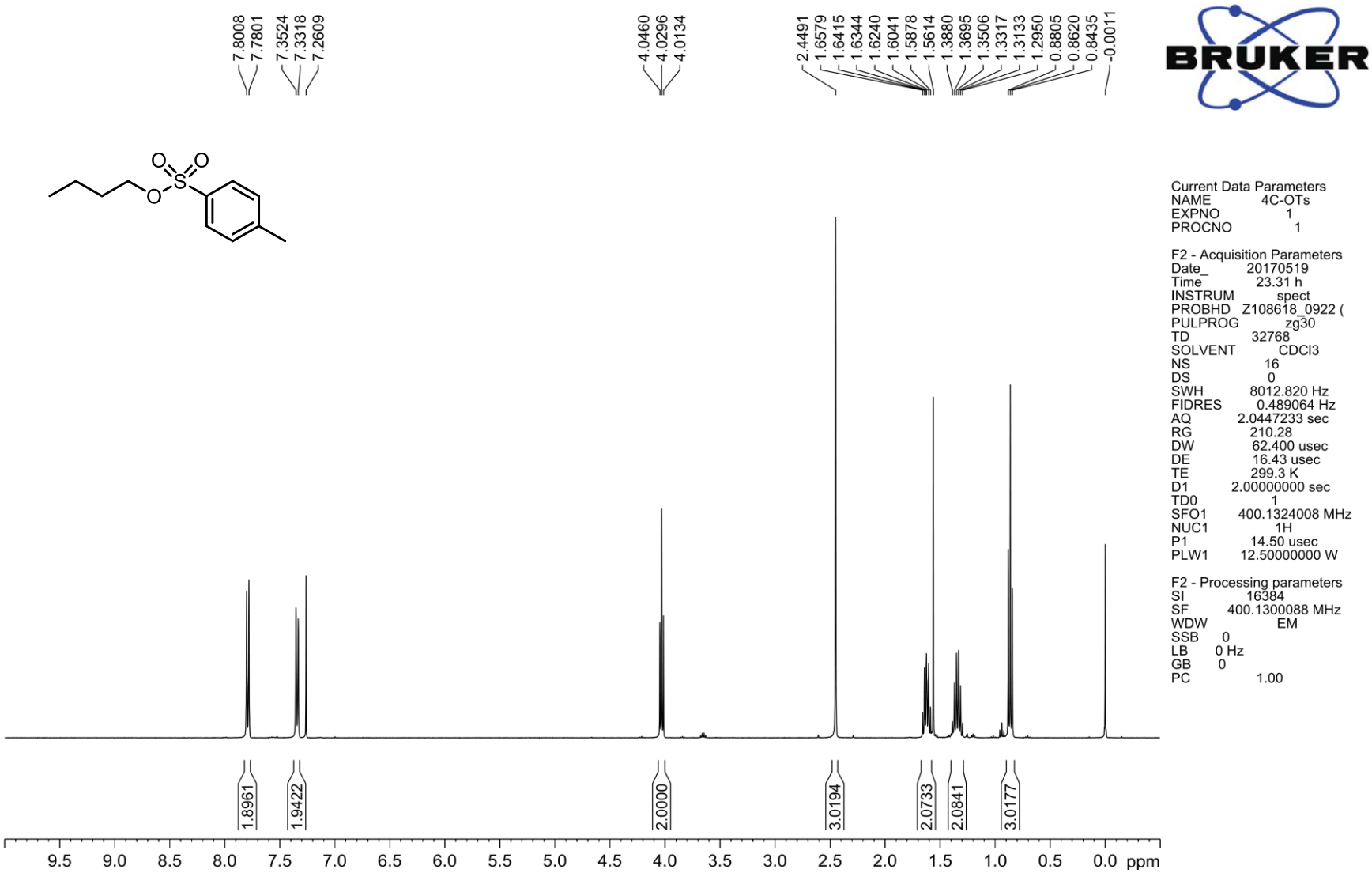
4H), 1.78 (bs, 2 × NCH_2CH_2, 4H), 1.94 (bs, 2 × NCH_2CH_2 ring, 4H), 2.21 (s, *p*-CH_3, 3H), 2.70 (s, *o*- & *m*-CH_3, 6H), 3.37 (m, 2 × NCH_2, 4H), 3.57 (m, 2 × NCH_2 ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.55, 19.62, 20.60, 21.74, 23.06, 24.26, 27.32, 60.83, 62.68, 130.22, 136.73, 137.19, 141.76; EI-HRMS *m/z* $[M]^+$ calcd for $C_{14}H_{30}N$ 212.2378, found 212.2373.

***N*,*N*-Dipentylazepanium 2,4,6-trimethylbenzenesulfonate, [N$_{6C55}$][OTMBS]:** 74% isolated yield; white solid; mp 120-121 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.91 (t, *J* = 6.8 Hz, 2 × CH_3, 6H), 1.31-1.42 (m, 4 × CH_2, 8H), 1.67 (m, 2 × $NCH_2CH_2CH_2$ ring, 4H), 1.74 (bs, 2 × NCH_2CH_2, 4H), 1.94 (bs, 2 × NCH_2CH_2 ring, 4H), 2.21 (s, *p*-CH_3, 3H), 2.71 (s, *o*- & *m*-CH_3, 6H), 3.36 (m, 2 × NCH_2, 4H), 3.58 (m, 2 × NCH_2 ring, 4H), 6.79 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.70, 20.60, 21.76, 22.05, 22.12, 23.08, 27.31, 28.30, 60.95, 62.66, 130.23, 136.73, 137.16, 141.79; EI-HRMS *m/z* $[M]^+$ calcd for $C_{16}H_{34}N$ 240.2697, found 240.2694.

***N*,*N*-Dihexylazepanium 2,4,6-trimethylbenzenesulfonate, [N$_{6C66}$][OTMBS]:** 76% isolated yield; white solid; mp 123-124 ºC; ^{1}H NMR (400MHz, $CDCl_3$) δ 0.89 (t, *J* = 6.9 Hz, CH_3, 6H), 1.27-1.41 (m, 3 × CH_2, 12H), 1.67 (m, $NCH_2CH_2CH_2$ ring, 4H), 1.75 (bs, NCH_2CH_2, 4H), 1.95 (bs, NCH_2CH_2 ring, 4H), 2.21 (s, CH_3C, 3H), 2.71 (s, CH_3CCSO_3, 6H), 3.35 (m, NCH_2, 4H), 3.58 (m, NCH_2 ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.76, 20.59, 21.75, 22.30, 23.09, 25.94, 27.30, 27.30, 31.13, 60.95, 62.66, 130.22, 136.71, 137.12, 141.80; EI-HRMS *m/z* $[M]^+$ calcd for $C_{18}H_{38}N$ 268.3004, found 268.3002.

***N*,*N*-Diheptylazepanium 2,4,6-trimethylbenzenesulfonate, [N$_{6C77}$][OTMBS]:** 67% isolated yield; white solid; mp 98-99 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.9 Hz, 2 × CH_3, 6H), 1.21-1.40 (m, 8 × CH_2, 16H), 1.66 (m, 2 × $NCH_2CH_2CH_2$ ring, 4H), 1.74 (bs, 2 × NCH_2CH_2, 4H), 1.94 (bs, 2 × NCH_2CH_2 ring, 4H), 2.21 (s, *p*-CH_3, 3H), 2.71 (s, *o*- & *m*-CH_3, 6H), 3.35 (m, 2 × NCH_2, 4H), 3.57 (m, 2 × NCH_2 ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.91, 20.61, 21.77, 22.36, 23.10, 26.23, 27.31, 28.71, 28.71, 31.43, 60.95, 62.68, 130.23, 136.73, 137.13, 141.78; EI-HRMS *m/z* $[M]^+$ calcd for $C_{20}H_{42}N$ 296.3317, found 296.3321.

***N*,*N*-Dioctylazepanium 2,4,6-trimethylbenzenesulfonate, [N$_{6C88}$][OTMBS]:** 69% isolated yield; white solid; mp 103-104 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.9 Hz, 2 × CH_3, 6H), 1.21-1.40 (m, 10 × CH_2, 20H), 1.67 (m, 2 × $NCH_2CH_2CH_2$ ring, 4H), 1.75 (bs, 2 × NCH_2CH_2, 4H), 1.95 (bs, 2 × NCH_2CH_2 ring, 4H), 2.21 (s, *p*-CH_3, 3H), 2.71 (s, *o*- & *m*-CH_3, 6H), 3.36 (m, 2 × NCH_2, 4H), 3.59 (m, 2 × NCH_2 ring, 4H), 6.80 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.95, 20.61, 21.77, 22.36, 22.48, 23.11, 26.27, 27.31, 28.94, 29.00, 31.55, 60.94, 62.69, 130.23, 136.73, 137.11, 141.79; EI-HRMS *m/z* $[M]^+$ calcd for $C_{22}H_{46}N$ 324.3630, found 324.3631.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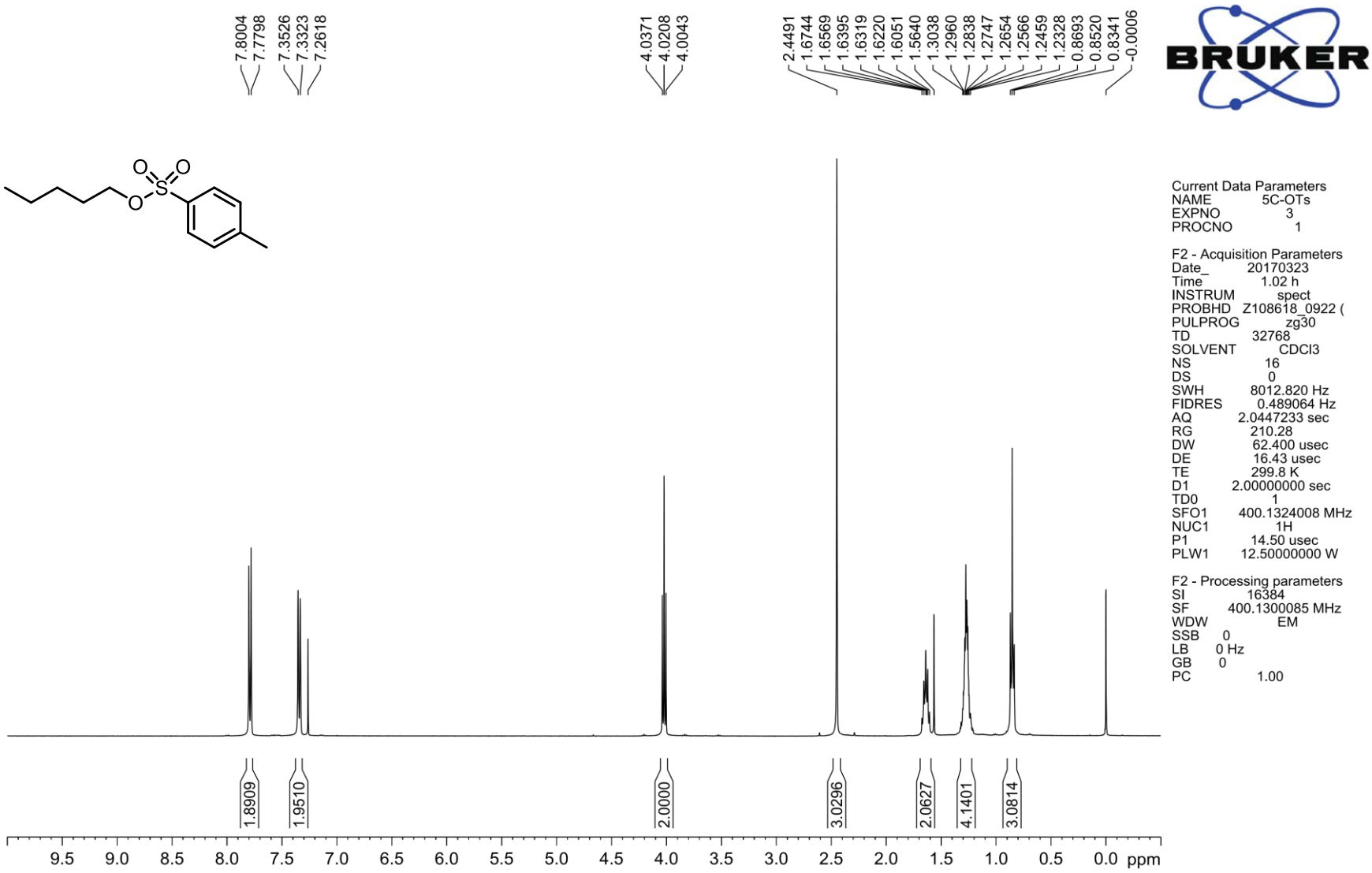
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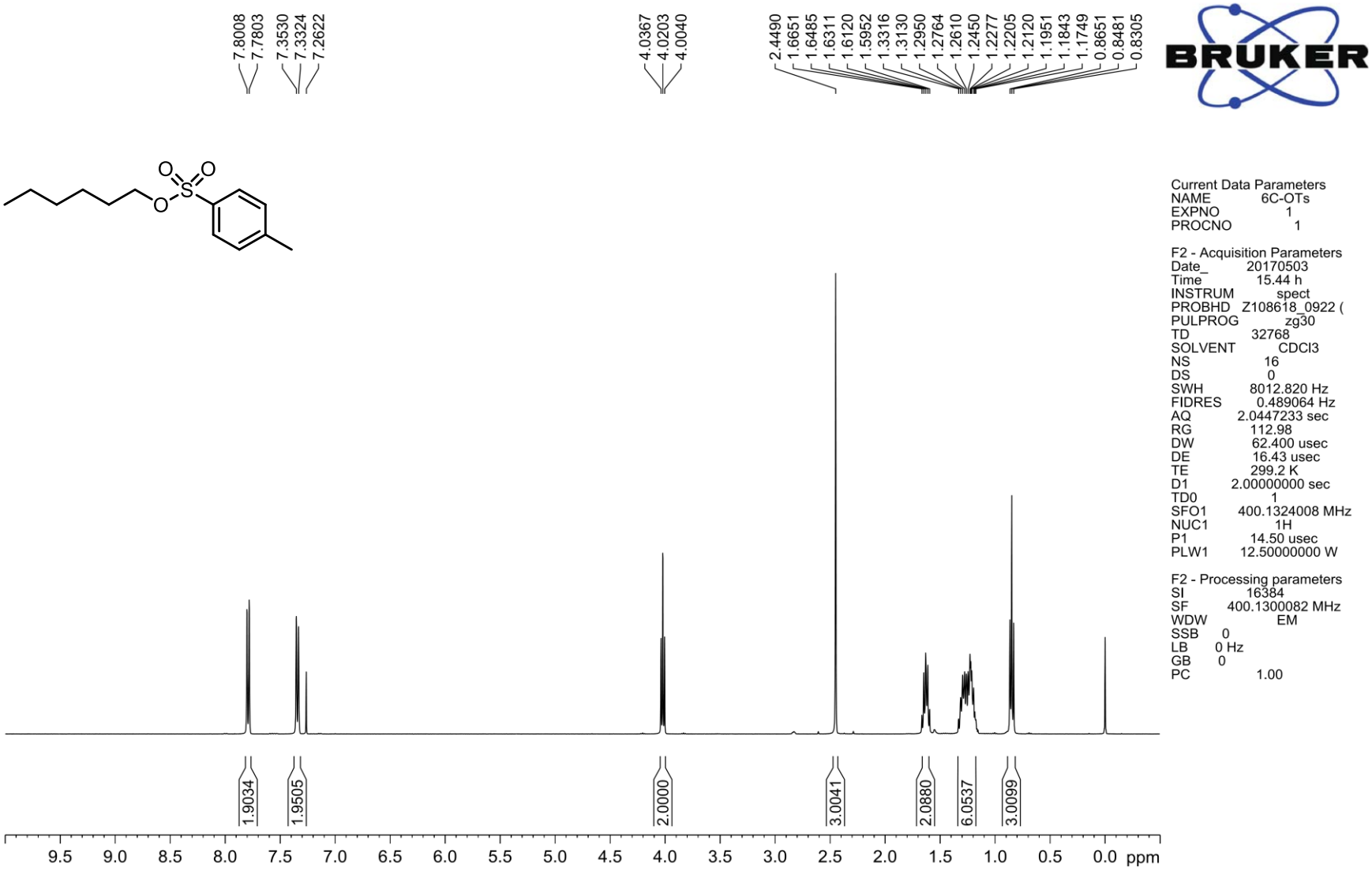
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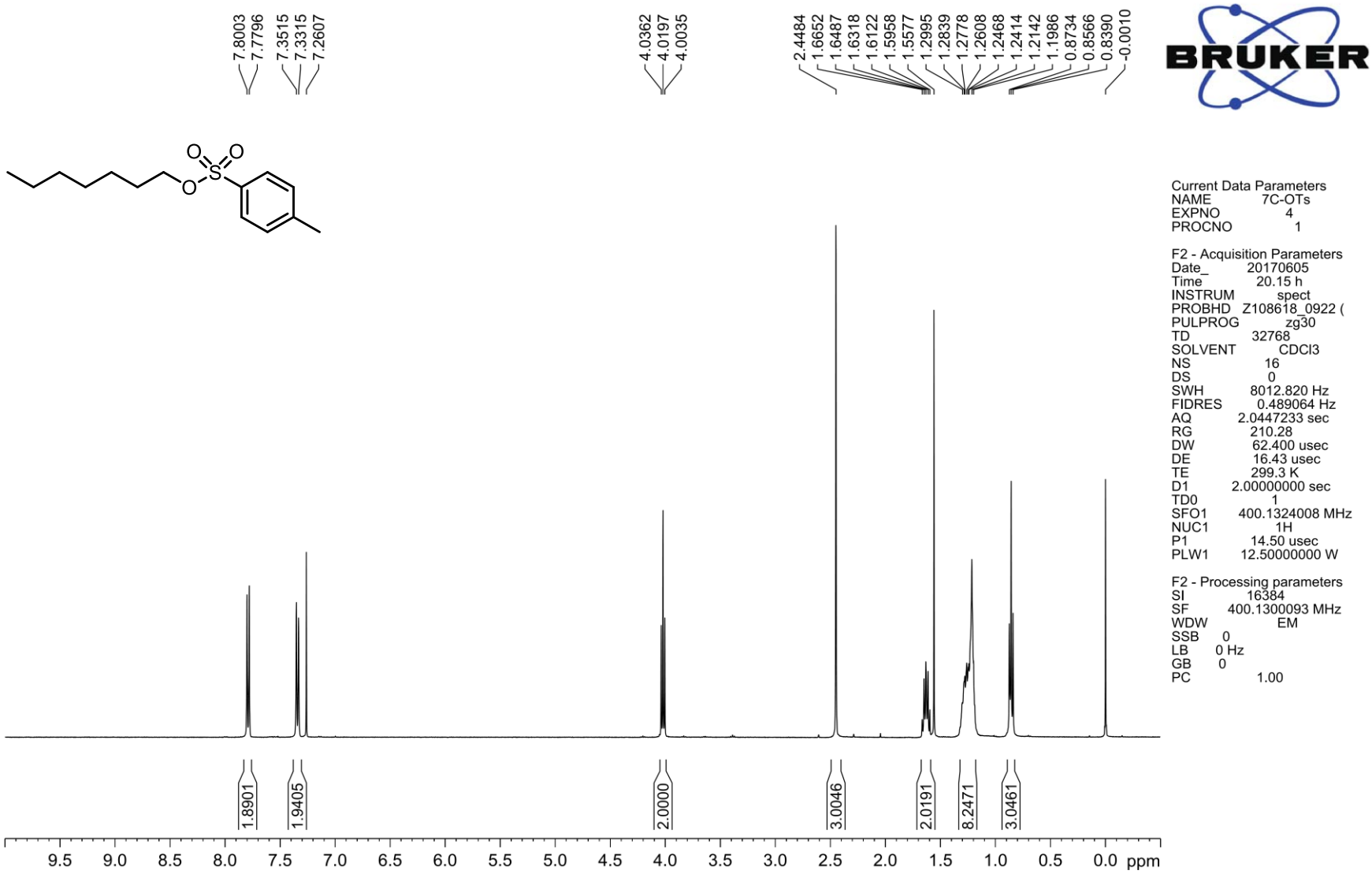
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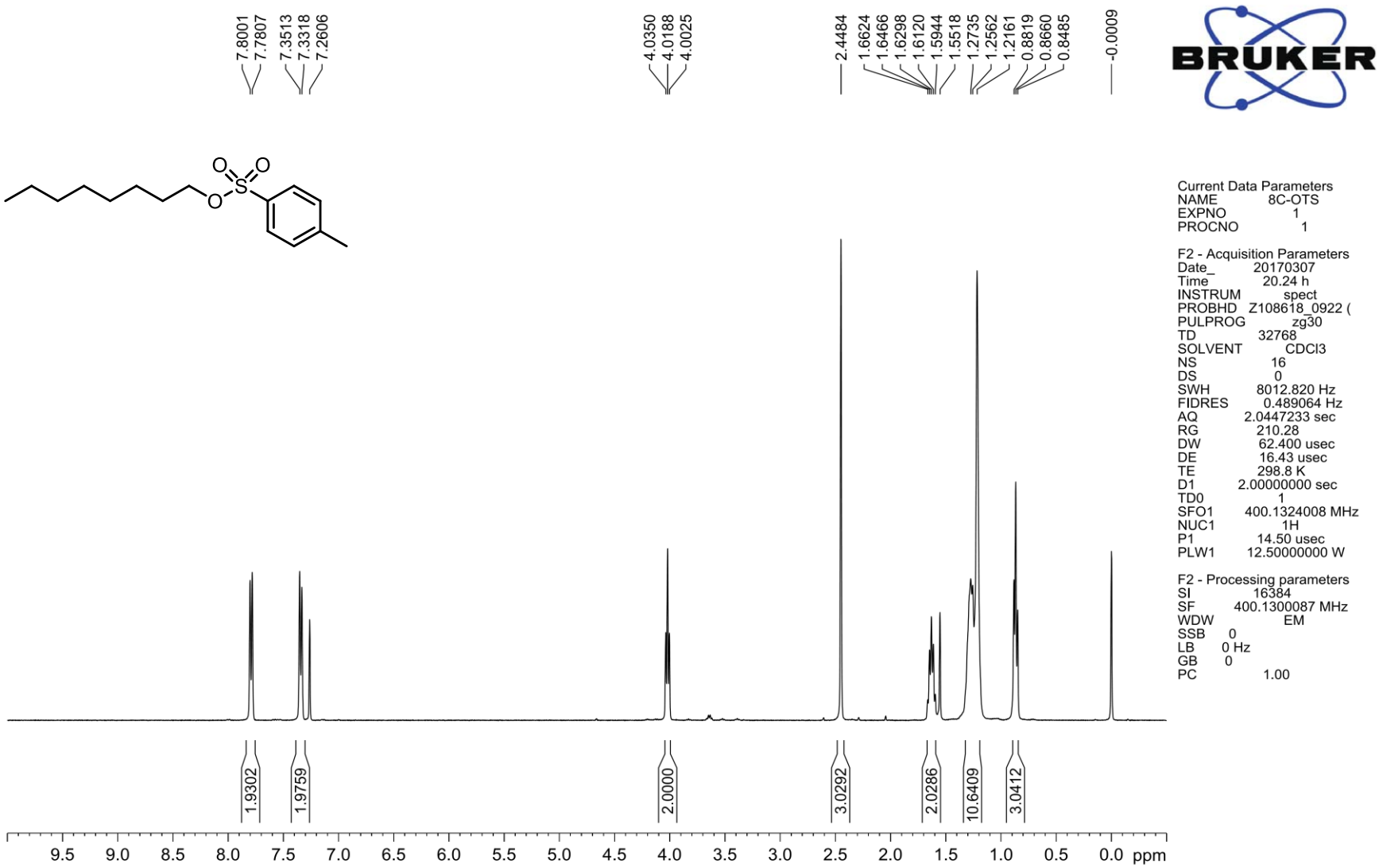
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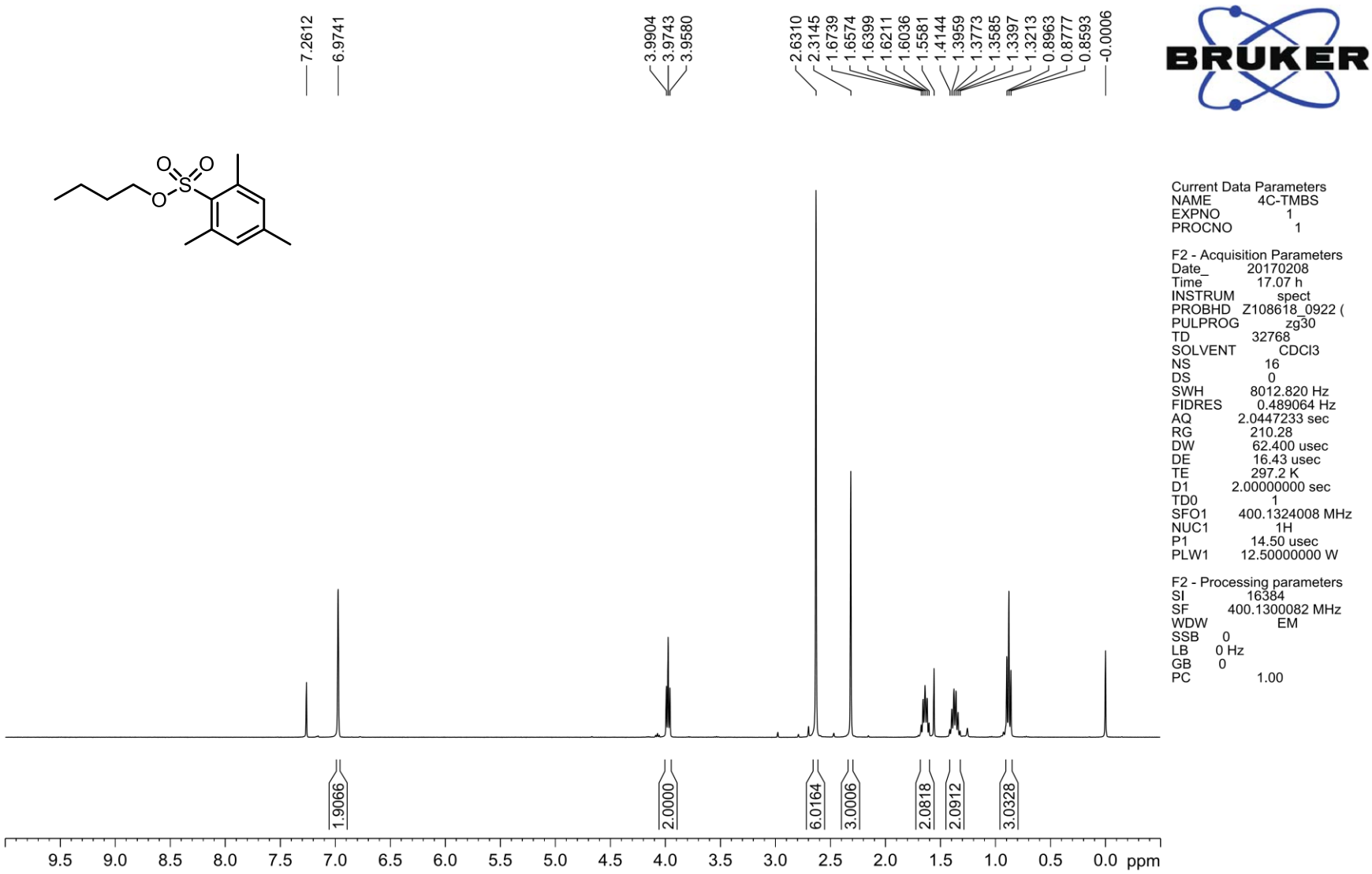
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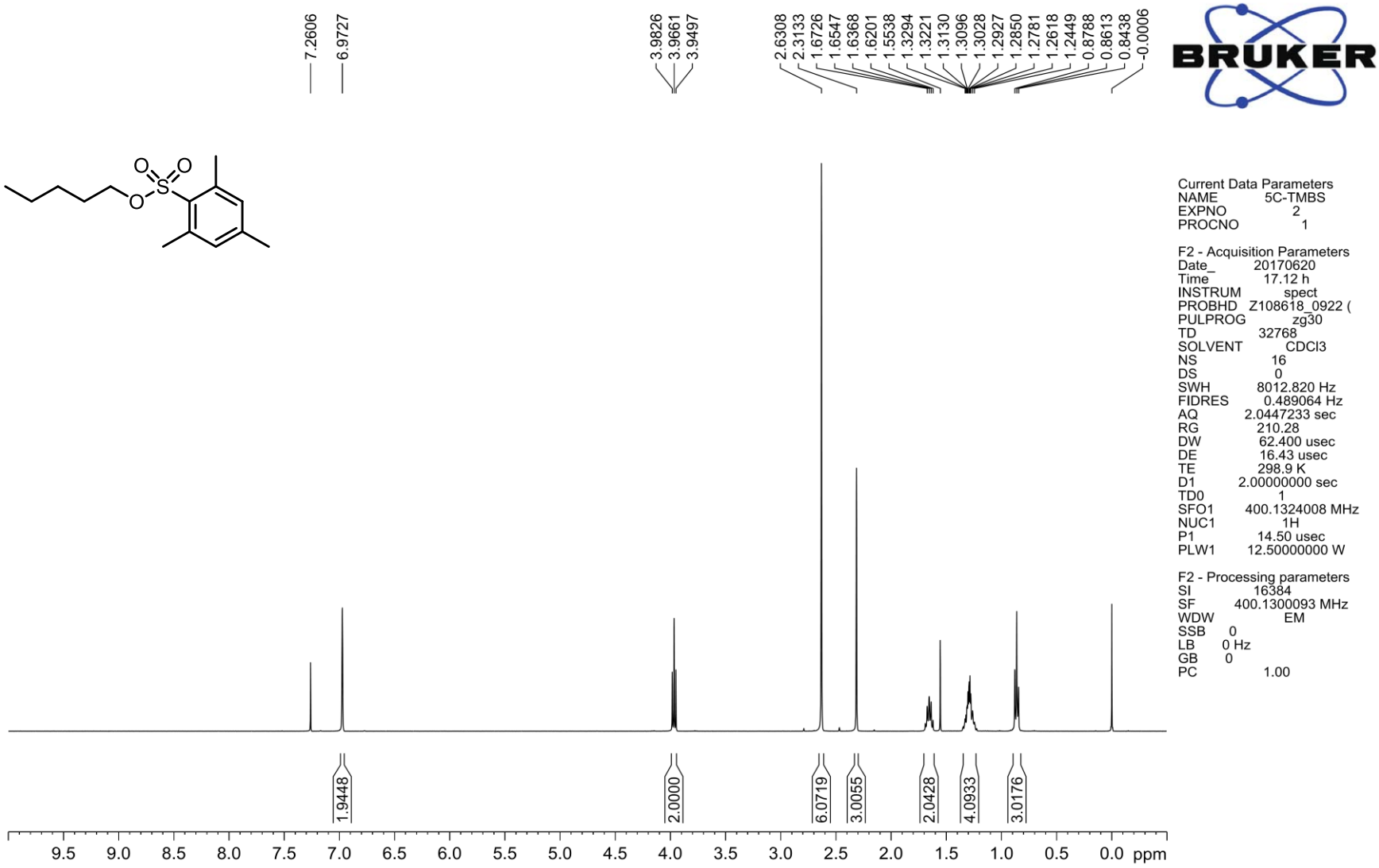
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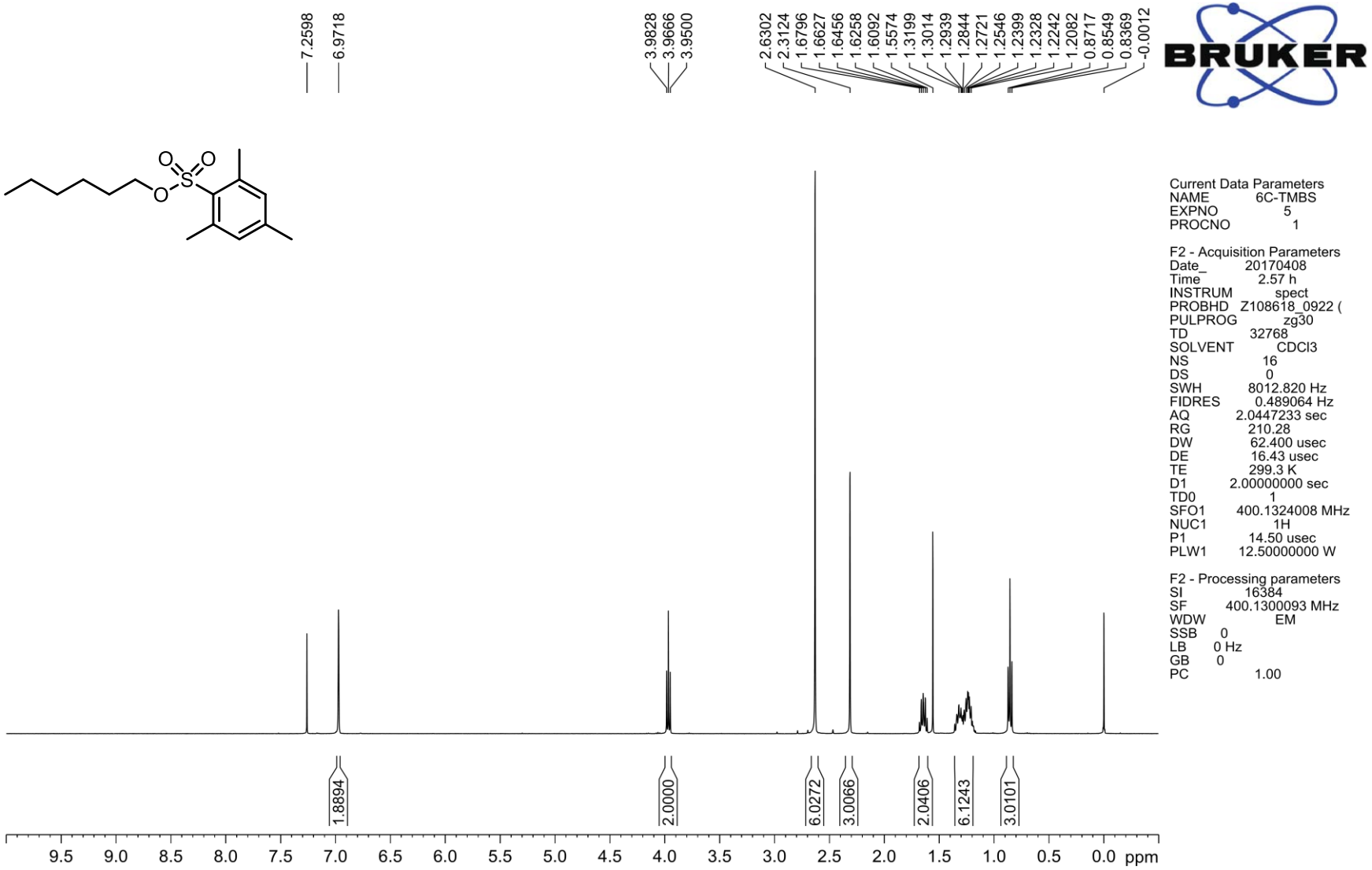
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NUC1 1H
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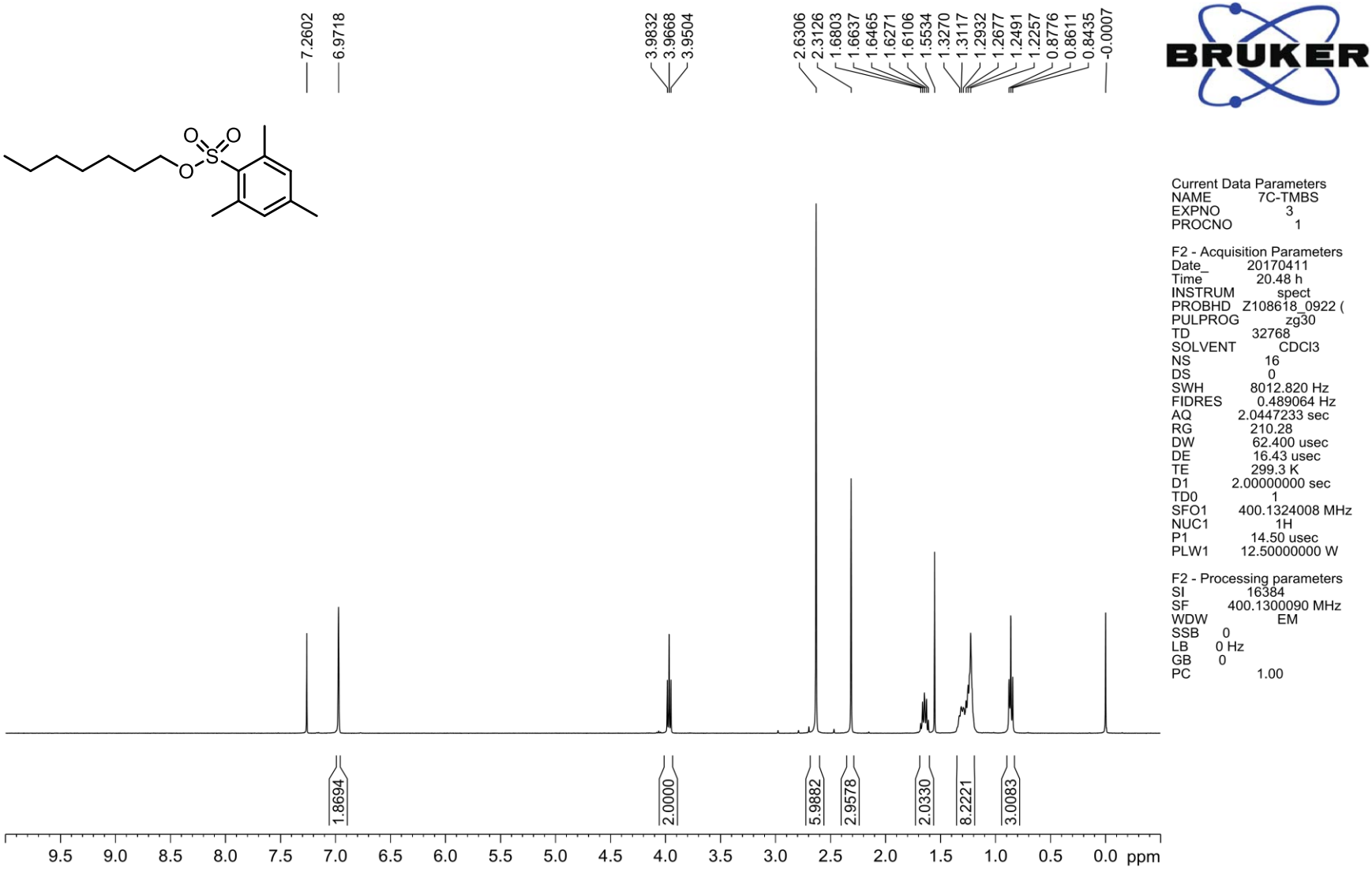
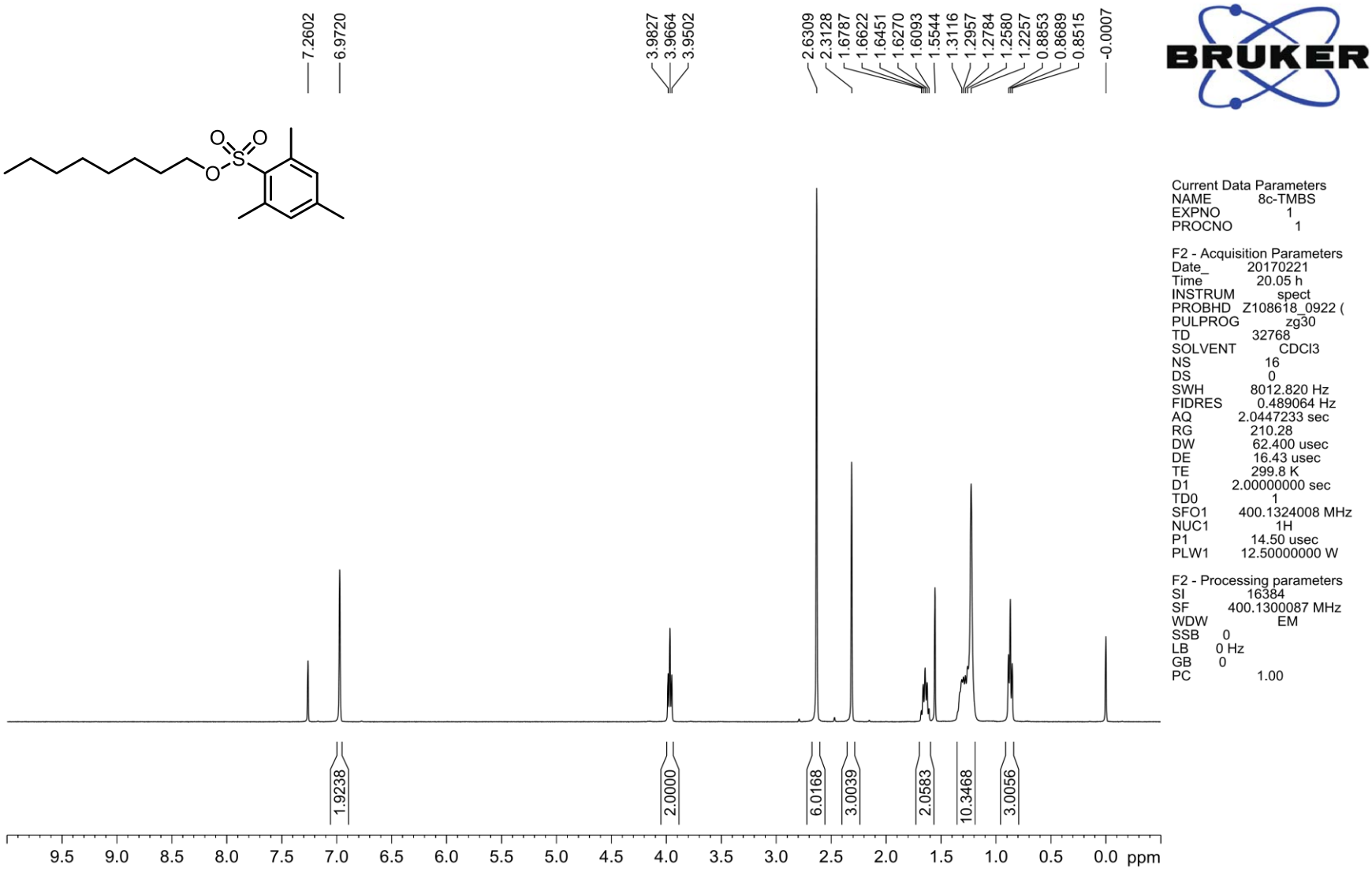
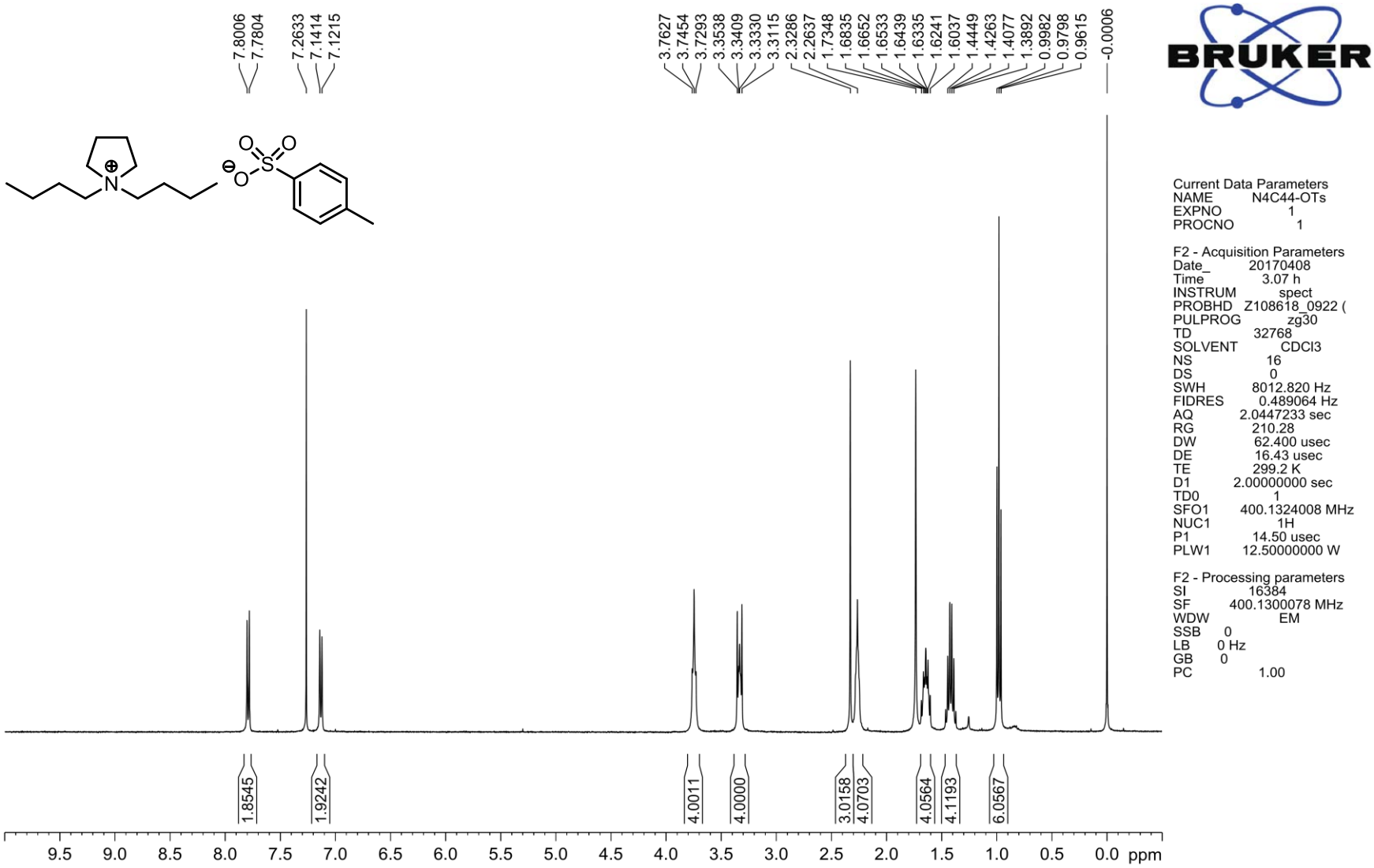
3.0101
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm


7.2602
6.9718
3.9832
3.9668
3.9504
2.6306
2.3126
1.6803
1.6637
1.6465
1.6271
1.6106
1.5534
1.3270
1.3117
1.2932
1.2677
1.2491
1.2257
0.8776
0.8611
0.8435
-0.0007
BRUKER
Current Data Parameters
NAME 7C-TMBS
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170411
Time 20.48 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.3 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.8694
2.0000
5.9882
2.9578
2.0330
8.2221
3.0083
9.5 9.0 8.5 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 ppm

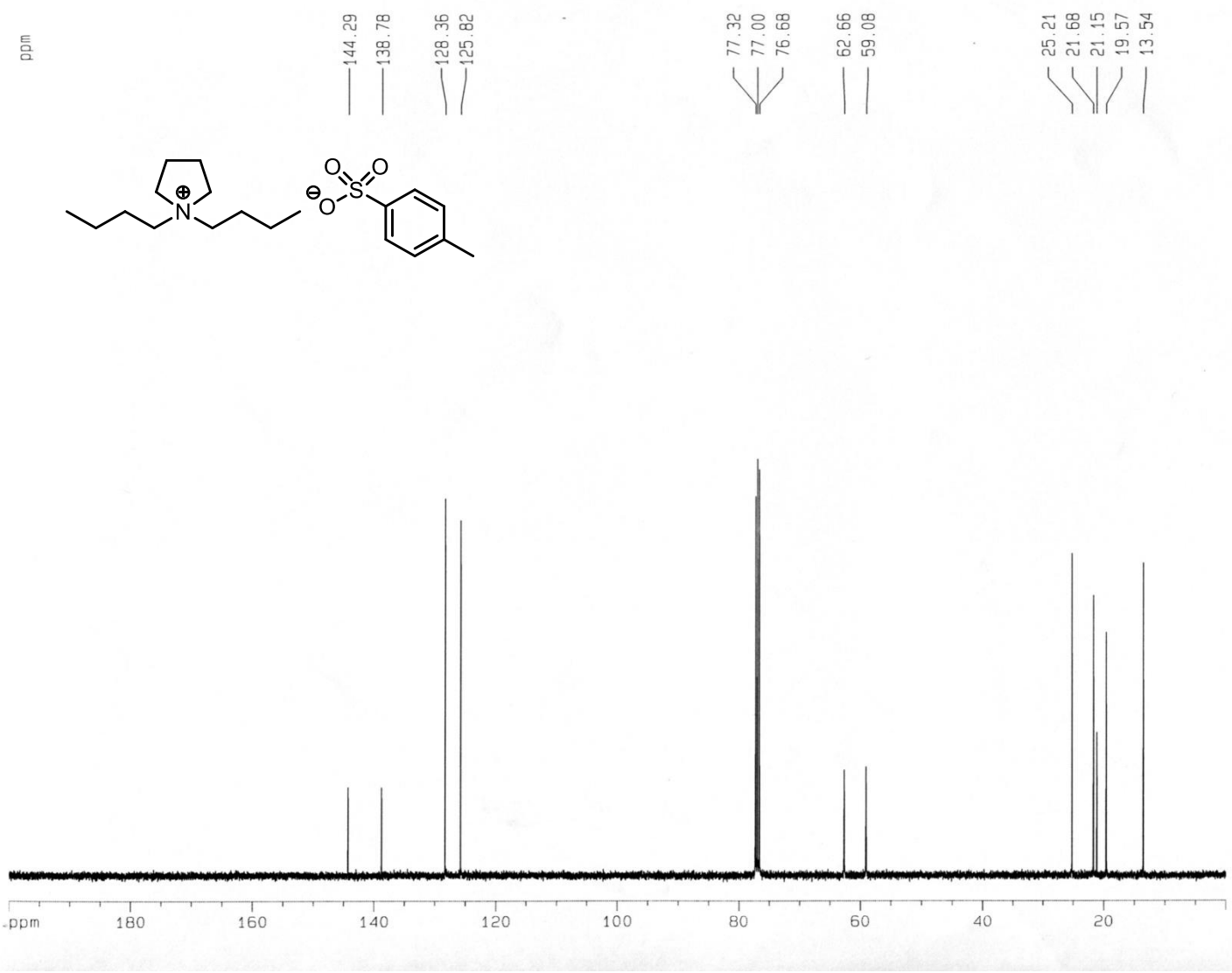

7.2602
6.9720
3.9827
3.9664
3.9502
2.6309
2.3128
1.6787
1.6622
1.6451
1.6270
1.6093
1.5544
1.3116
1.2957
1.2784
1.2580
1.2257
0.8853
0.8689
0.8515
-0.0007
BRUKER
Current Data Parameters
NAME 8c-TMBS
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170221
Time 20.05 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.8 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300087 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9238
2.0000
6.0168
3.0039
2.0583
10.3468
3.0056
9.5 9.0 8.5 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 ppm



Current Data Parameters
NAME N4C44-OTs
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170408
Time 3.07 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.2 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME                  N4CXXC13
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20170704
Time                     11.18
INSTRUM                  spect
PROBHD       5 mm QNP  1H
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         752
DS                           4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                         256
DW                    19.900 usec
DE                     6.50 usec
TE                     300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 =============
NUC1                      13C
P1                      10.20 usec
PL1                      0.00 dB
SFO1              100.6237959 MHz

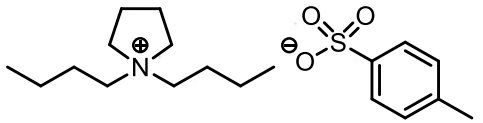
============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                     -3.00 dB
PL12                    14.50 dB
PL13                    17.50 dB
SFO2              400.1326008 MHz

F2 - Processing parameters
SI                        32768
SF                100.6127784 MHz
WDW                          EM
SSB                           0
LB                       0.30 Hz
GB                            0
PC                       1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     200.000 ppm
F1                    20122.55 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM                1006.12775 Hz/cm
```

1 (HR-ESI)

Elemental Composition Report



Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

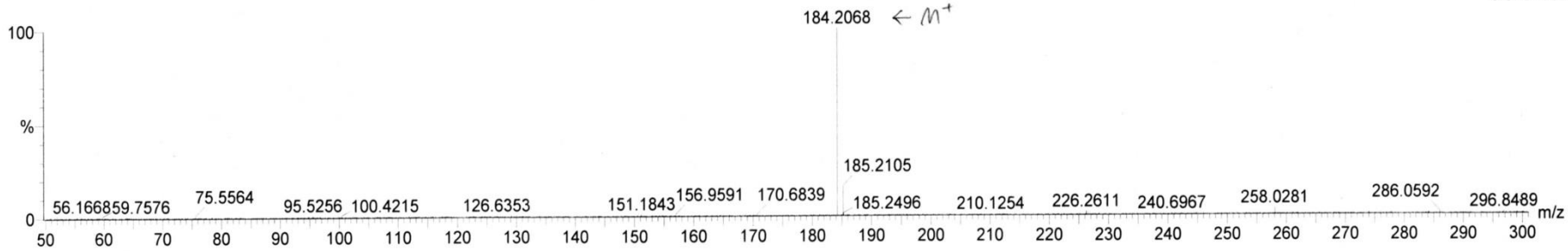
Monoisotopic Mass, Even Electron Ions
15 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

1

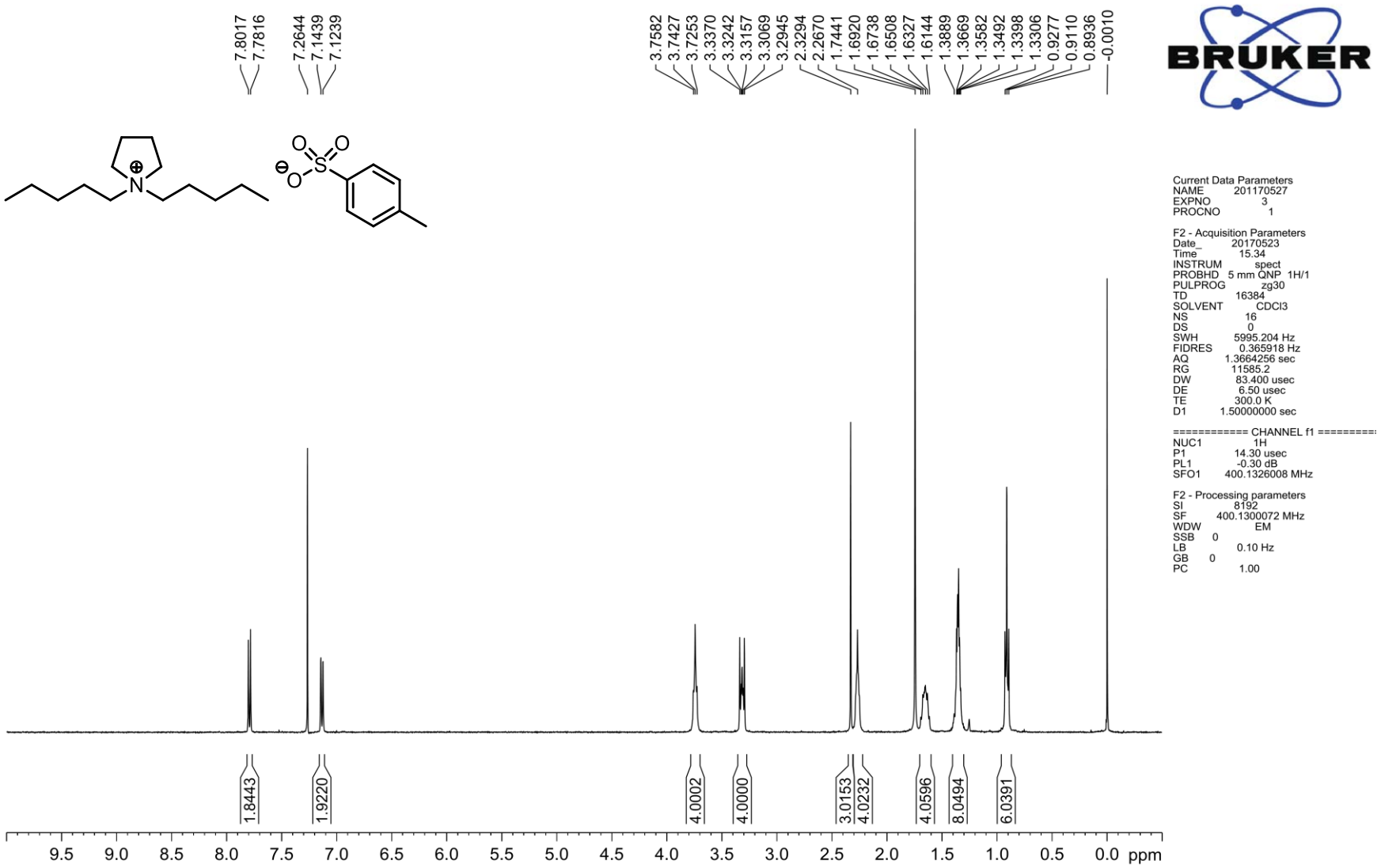
KE267

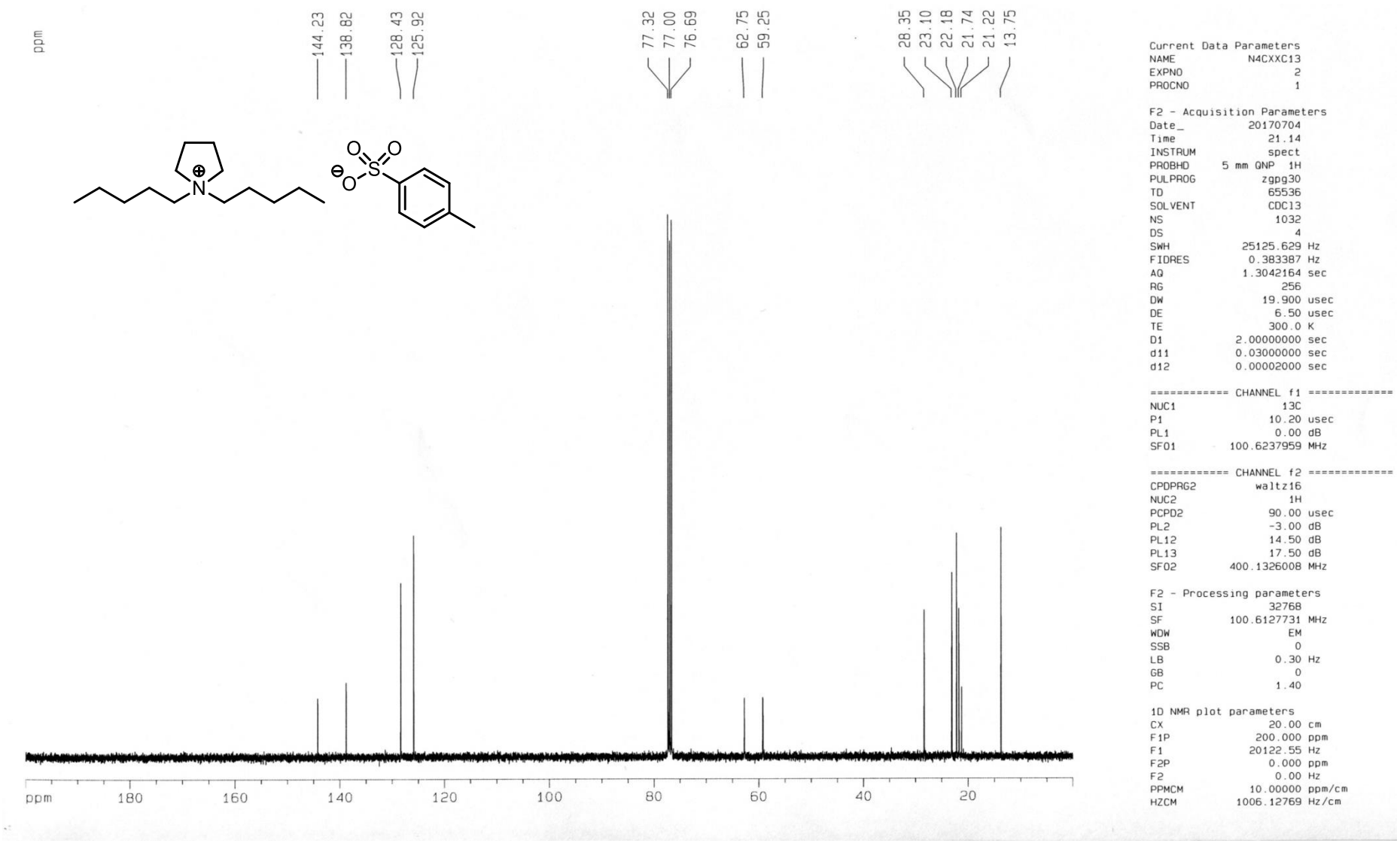
29-Jun-2017
13:43:18
1: TOF MS ES+
6.61e+002

0629_1 45 (1.642) Cm (45-1)



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
184.2068	184.2065	0.3	1.6	0.5	45.8	0.0	C12 H26 N


7.8017
7.7816
7.2644
7.1439
7.1239
3.7582
3.7427
3.7253
3.3370
3.3242
3.3157
3.3069
3.2945
2.3294
2.2670
1.7441
1.6920
1.6738
1.6508
1.6327
1.6144
1.3889
1.3669
1.3582
1.3492
1.3398
1.3306
0.9277
0.9110
0.8936
-0.0010
BRUKER
Current Data Parameters
NAME 201170527
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170523
Time 15.34
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 11585.2
DW 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 14.30 usec
PL1 -0.30 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 8192
SF 400.1300072 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1.8443
1.9220
4.0002
4.0000
3.0153
4.0232
4.0596
8.0494
6.0391
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                 N4CXXC13
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170704
Time                   21.14
INSTRUM                spect
PROBHD     5 mm QNP  1H
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1032
DS                          4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                        256
DW                   19.900 usec
DE                    6.50 usec
TE                   300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                      13C
P1                     10.20 usec
PL1                     0.00 dB
SFO1              100.6237959 MHz

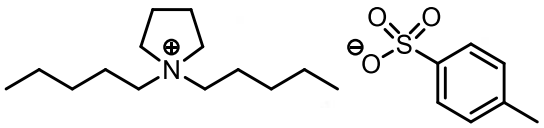
============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  90.00 usec
PL2                    -3.00 dB
PL12                   14.50 dB
PL13                   17.50 dB
SFO2              400.1326008 MHz

F2 - Processing parameters
SI                       32768
SF                100.6127731 MHz
WDW                         EM
SSB                          0
LB                     0.30 Hz
GB                           0
PC                     1.40

1D NMR plot parameters
CX                     20.00 cm
F1P                   200.000 ppm
F1                  20122.55 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM                 1006.12769 Hz/cm
```

2 (HR-ESI)

Elemental Composition Report

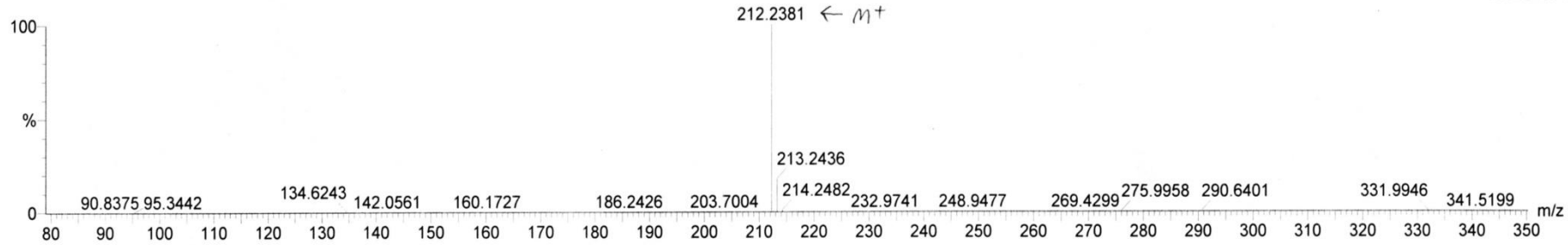


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

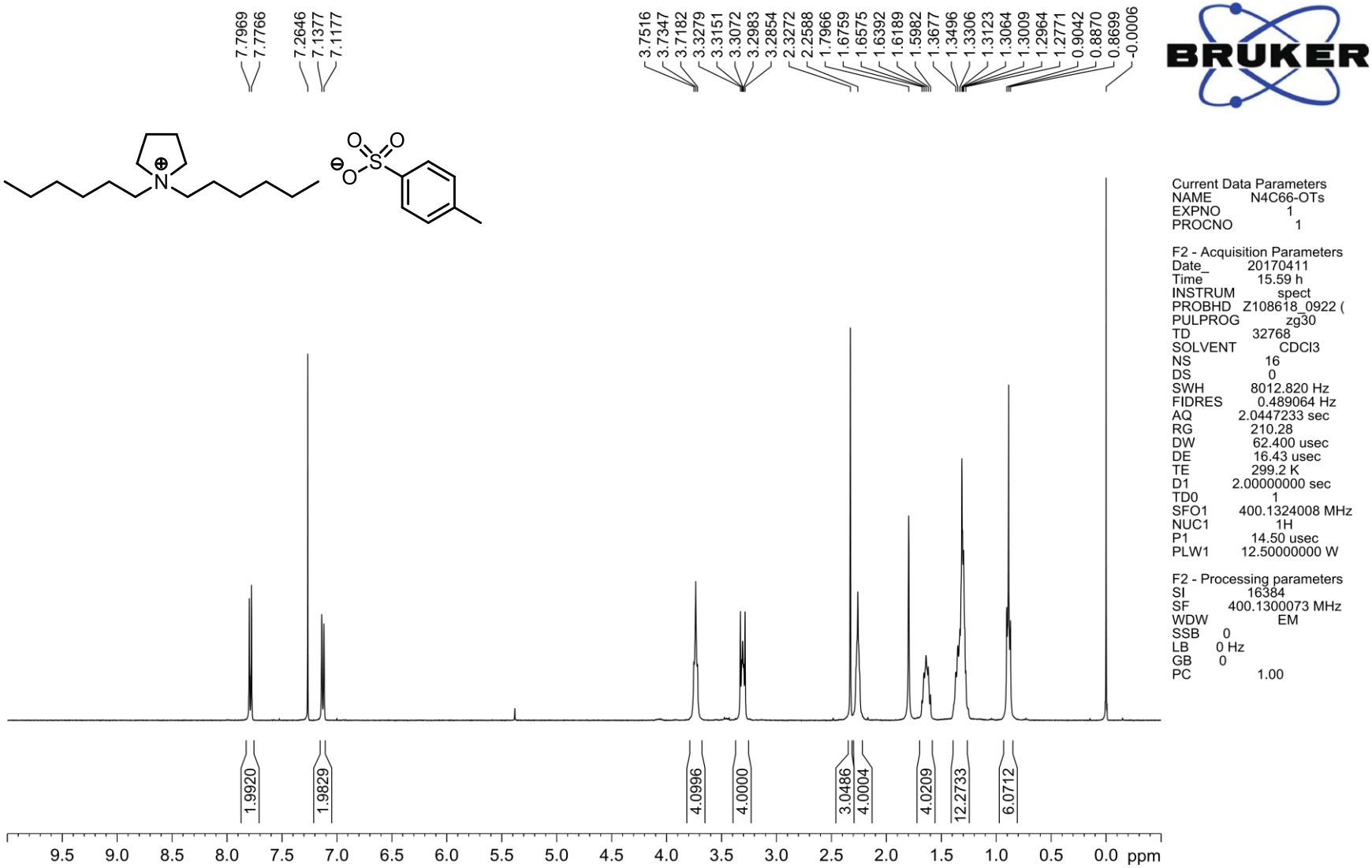
Monoisotopic Mass, Even Electron Ions
17 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

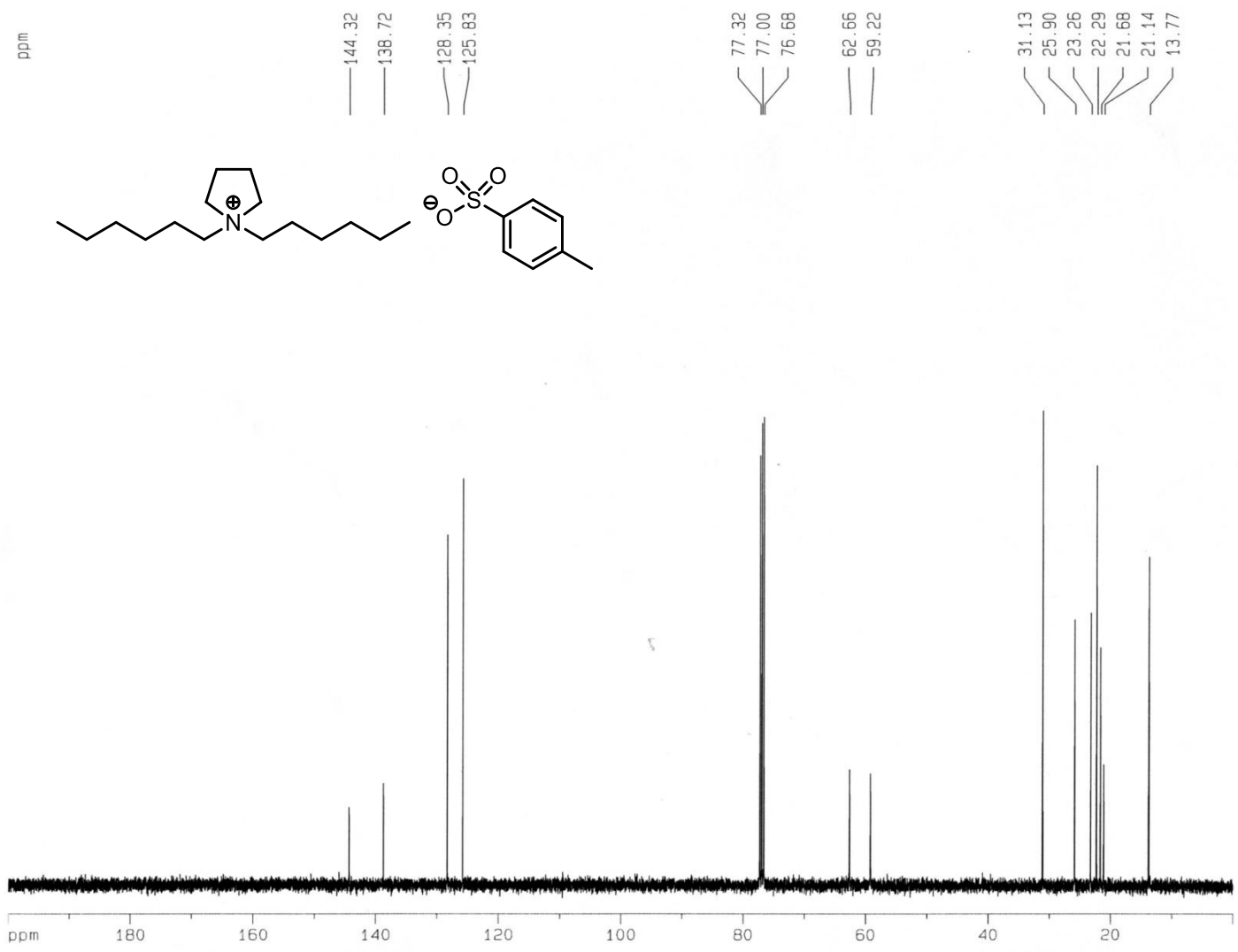
2 KE267 29-Jun-2017 13:02:47

0629_2 35 (1.266) Cm (35-1x200.000) 1: TOF MS ES+ 9.02e+004



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
212.2381	212.2378	0.3	1.4	0.5	80.6	0.0	C14 H30 N


7.7969
7.7766
7.2646
7.1377
7.1177
3.7516
3.7347
3.7182
3.3279
3.3151
3.3072
3.2983
3.2854
2.3272
2.2588
1.7966
1.6759
1.6575
1.6392
1.6189
1.5982
1.3677
1.3496
1.3306
1.3123
1.3064
1.3009
1.2964
1.2771
0.9042
0.8870
0.8699
-0.0006
BRUKER
Current Data Parameters
NAME N4C66-OTs
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170411
Time 15.59 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.2 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300073 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9920
1.9829
4.0996
4.0000
3.0486
4.0004
4.0209
12.2733
6.0712
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                 N4CXXC13
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170704
Time                   21.42
INSTRUM                spect
PROBHD     5 mm QNP  1H
PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                        274
DS                          4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                        256
DW                   19.900 usec
DE                    6.50 usec
TE                   300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                      13C
P1                     10.20 usec
PL1                     0.00 dB
SFO1               100.6237959 MHz

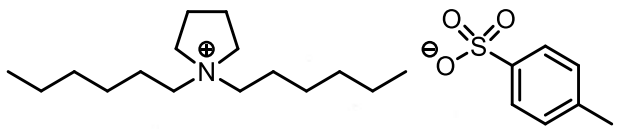
============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  90.00 usec
PL2                    -3.00 dB
PL12                   14.50 dB
PL13                   17.50 dB
SFO2               400.1326008 MHz

F2 - Processing parameters
SI                        32768
SF                 100.6127784 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
F1P                  200.000 ppm
F1                 20122.55 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM               1006.12775 Hz/cm
```

3 (HR-ESI)

Elemental Composition Report



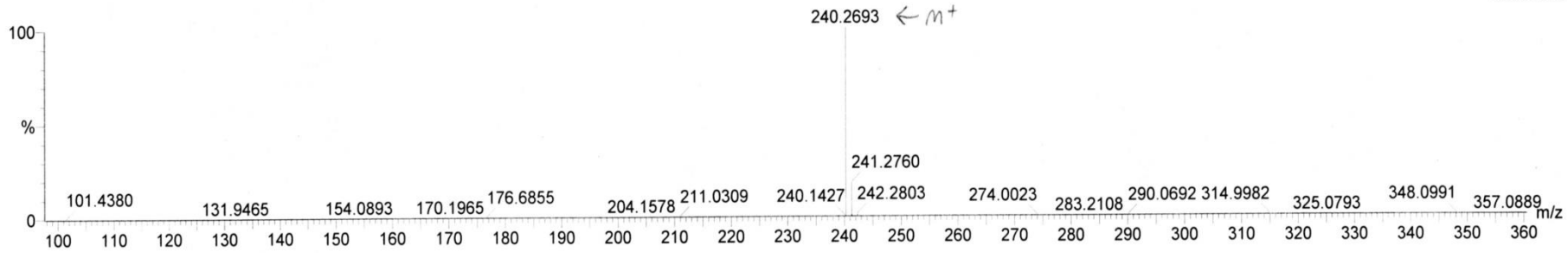
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
19 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1
3

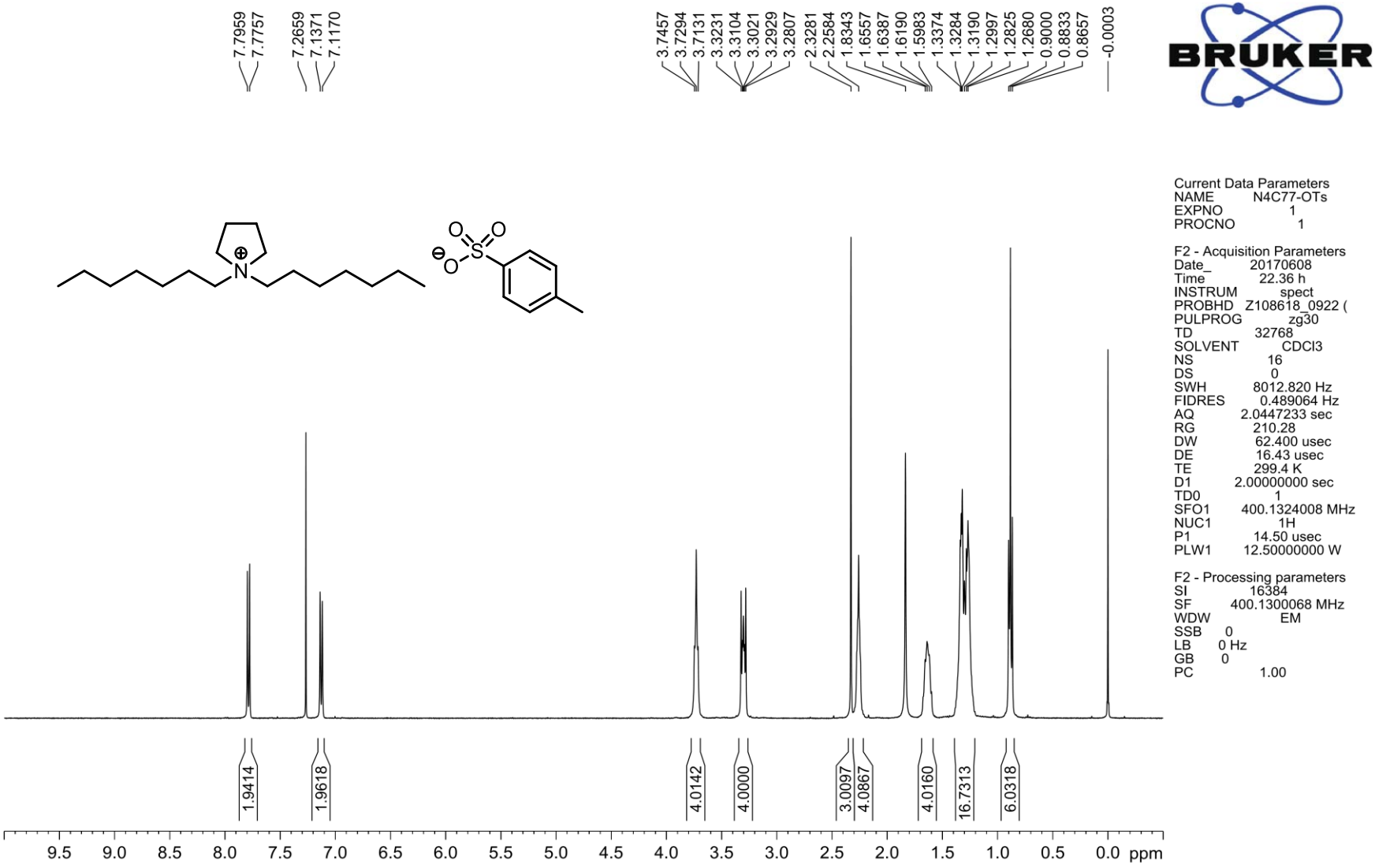
KE267

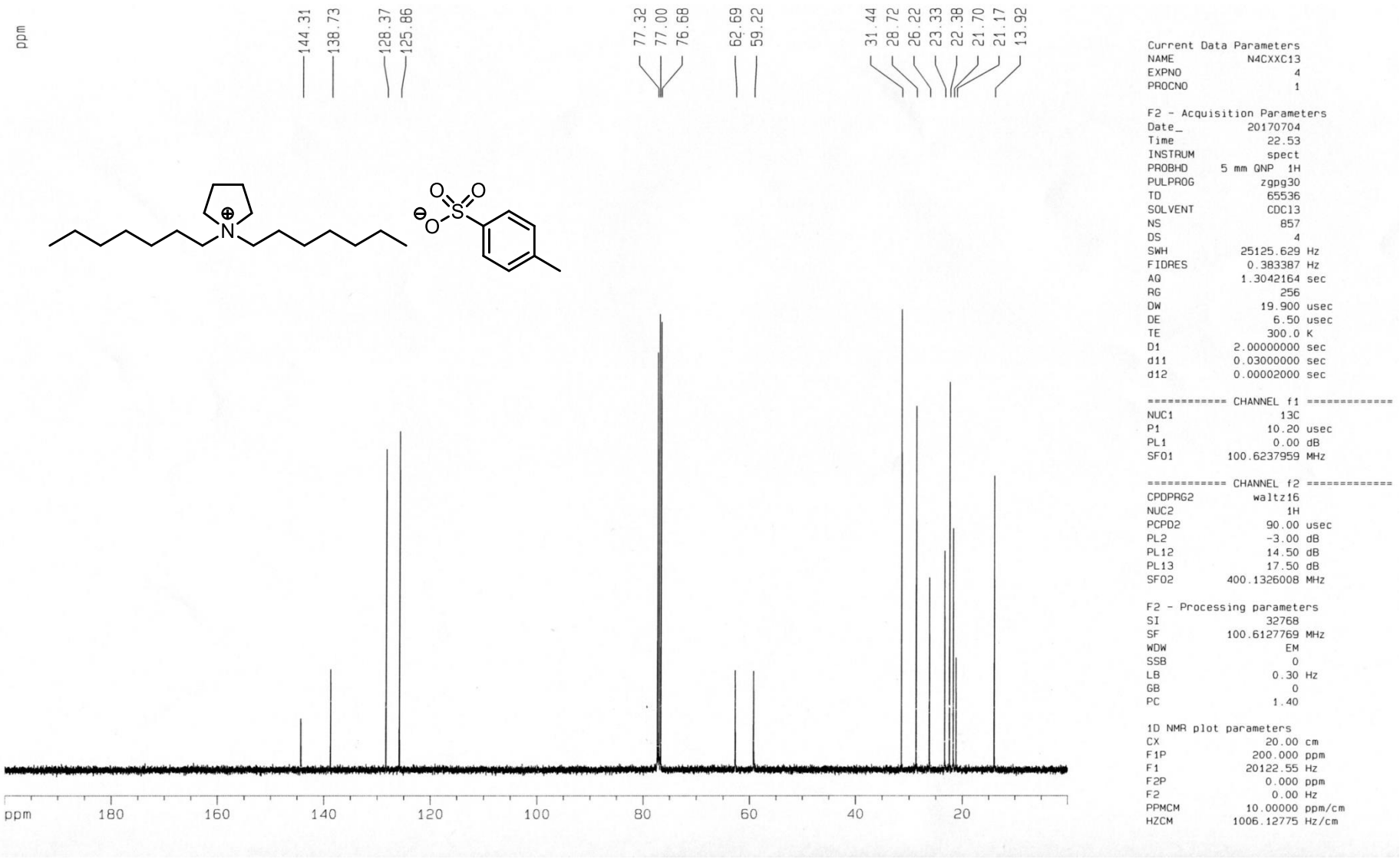
29-Jun-2017
12:38:31
1: TOF MS ES+
1.83e+005

0629_3 84 (3.035) Cm (84-1x200.000)



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
240.2693	240.2691	0.2	0.8	0.5	93.8	0.0	C16 H34 N


7.7959
7.7757
7.2659
7.1371
7.1170
3.7457
3.7294
3.7131
3.3231
3.3104
3.3021
3.2929
3.2807
2.3281
2.2584
1.8343
1.6557
1.6387
1.6190
1.5983
1.3374
1.3284
1.3190
1.2997
1.2825
1.2680
0.9000
0.8833
0.8657
-0.0003
BRUKER
Current Data Parameters
NAME N4C77-OTs
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170608
Time 22.36 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.4 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300068 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9414
1.9618
4.0142
4.0000
3.0097
4.0867
4.0160
16.7313
6.0318
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME            N4CXXC13
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170704
Time               22.53
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  857
DS                    4
SWH           25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1        100.6237959 MHz

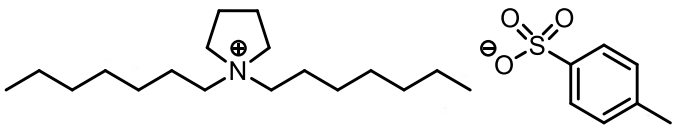
============ CHANNEL f2 =============
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2        400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127769 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.40

1D NMR plot parameters
CX                20.00 cm
F1P              200.000 ppm
F1             20122.55 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1006.12775 Hz/cm
```

4 (HR-ESI)

Elemental Composition Report



Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

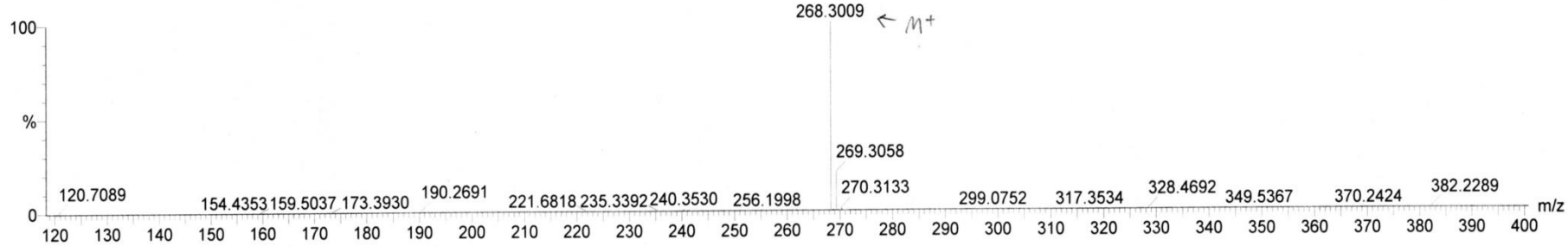
Monoisotopic Mass, Even Electron Ions
22 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

4

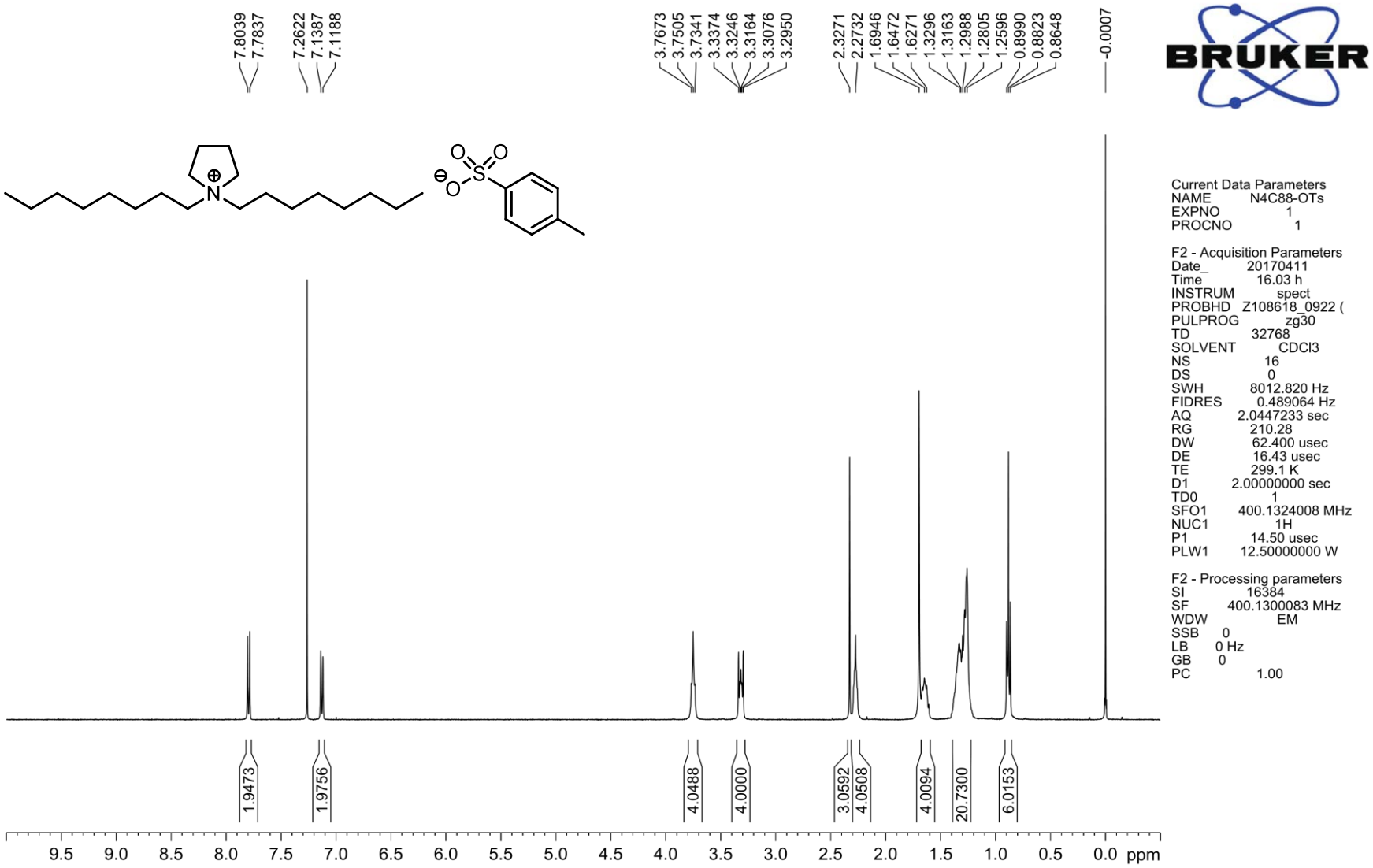
KE267

29-Jun-2017
12:46:36
1: TOF MS ES+
5.33e+004

0629_4 62 (2.255) Cm (62-1x200.000)



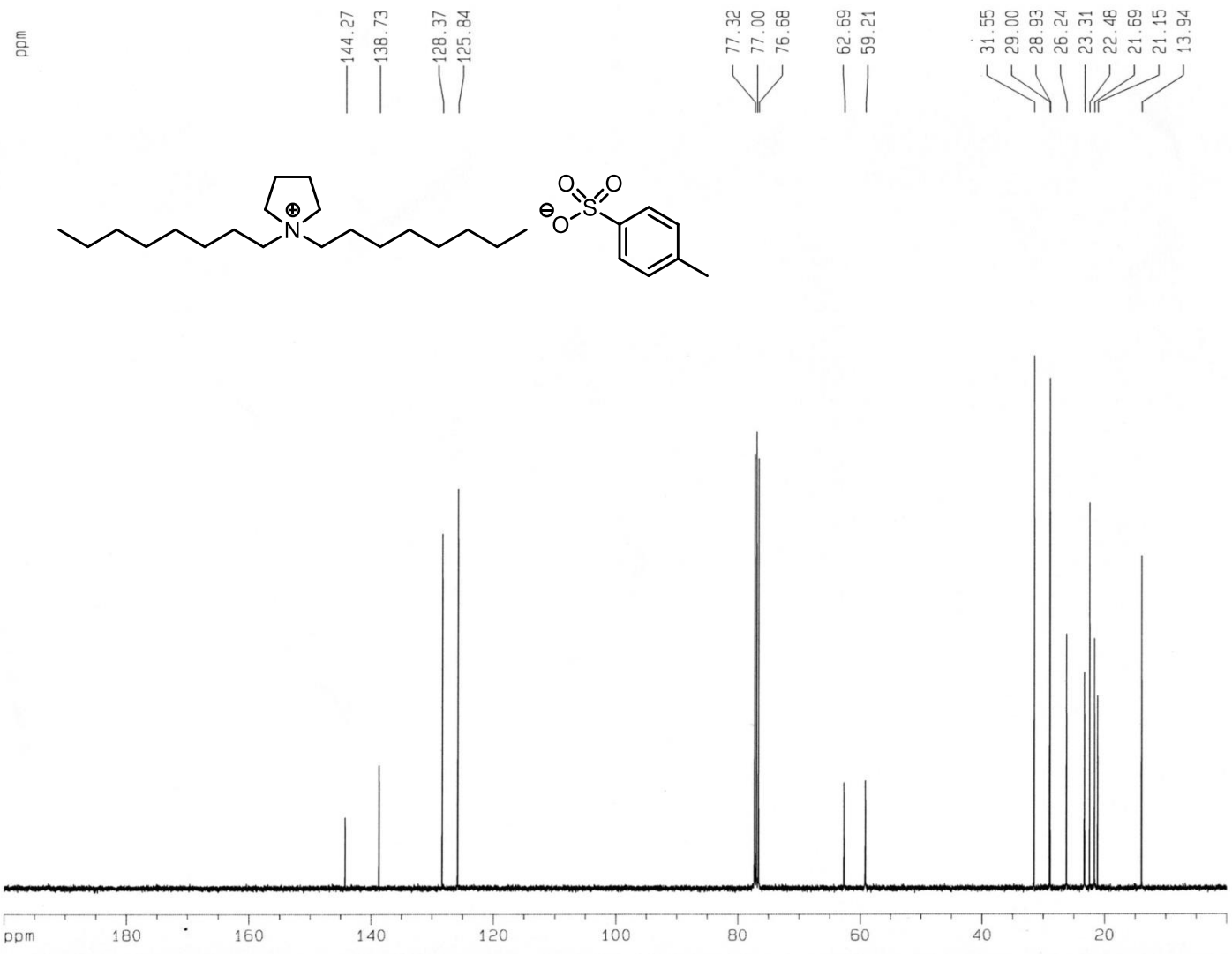
Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
268.3009	268.3004	0.5	1.9	0.5	84.2	0.0	C18 H38 N



Current Data Parameters
NAME N4C88-OTs
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170411
Time 16.03 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.1 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

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Current Data Parameters
NAME                 N4CXXC13
EXPNO                       5
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170704
Time                   23.23
INSTRUM                spect
PROBHD      5 mm QNP  1H
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      1257
DS                         4
SWH               25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                       256
DW                    19.900 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                      13C
P1                      10.20 usec
PL1                      0.00 dB
SF01              100.6237959 MHz

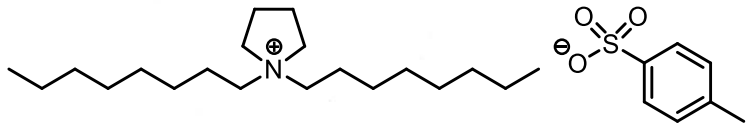
============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                     -3.00 dB
PL12                    14.50 dB
PL13                    17.50 dB
SF02              400.1326008 MHz

F2 - Processing parameters
SI                        32768
SF                100.6127777 MHz
WDW                          EM
SSB                           0
LB                       0.30 Hz
GB                            0
PC                       1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     200.000 ppm
F1                    20122.55 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM               1006.12775 Hz/cm
```

5 (HR-ESI)

Elemental Composition Report



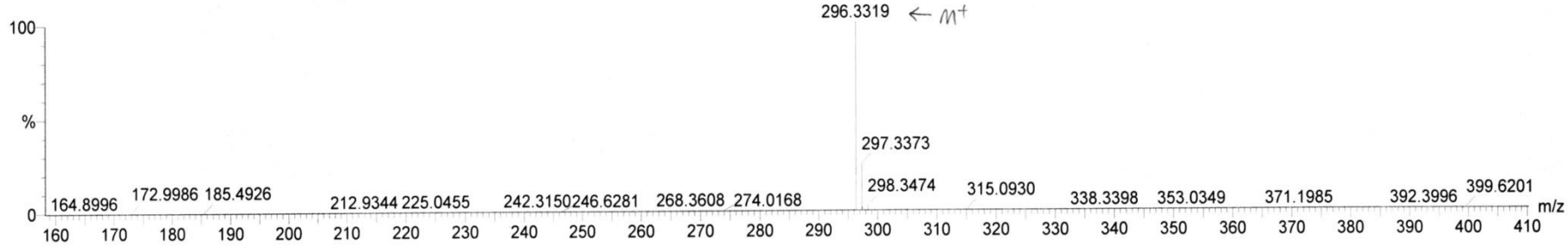
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
24 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1
5

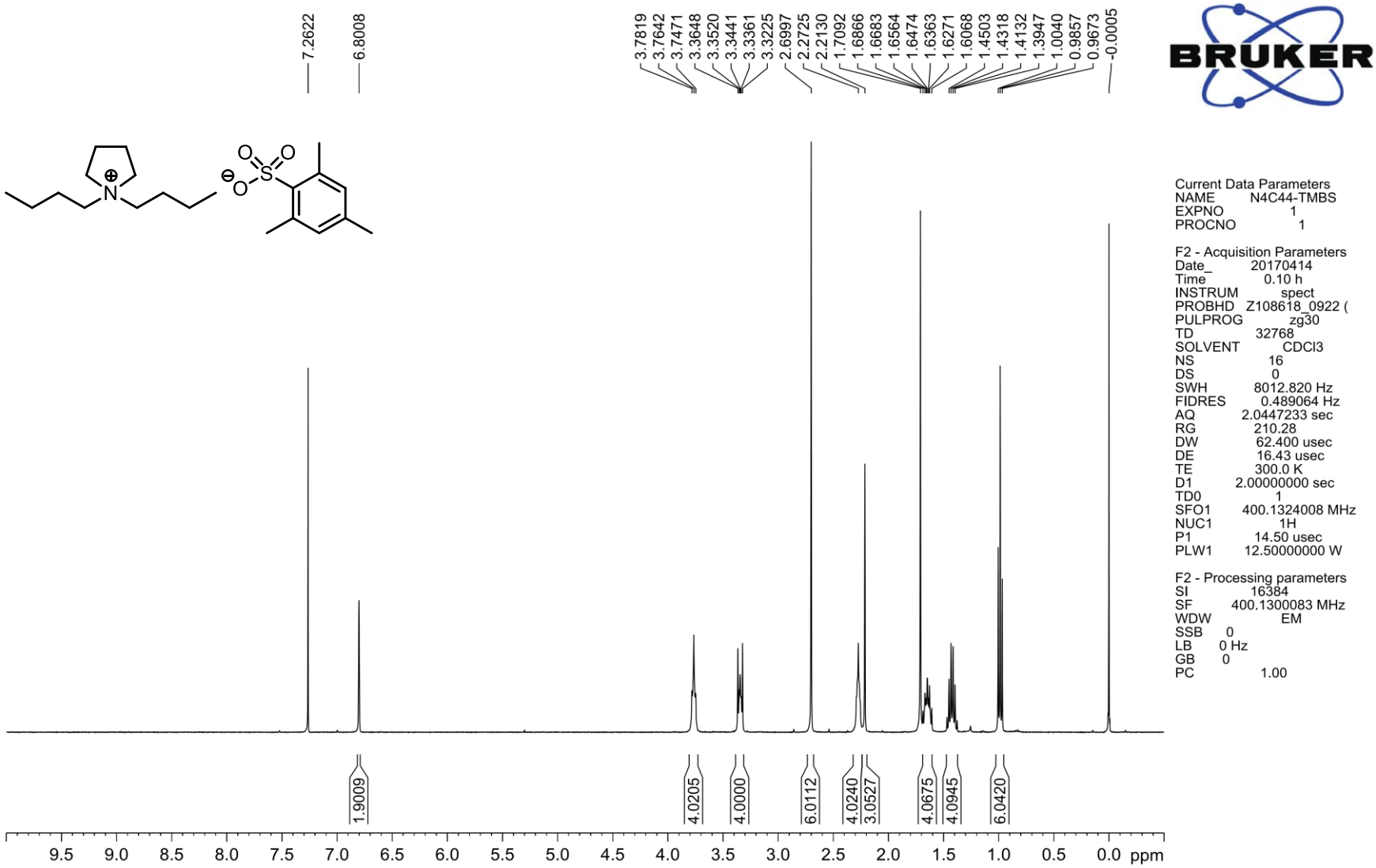
KE267

29-Jun-2017
12:54:40
1: TOF MS ES+
7.21e+004

0629_5 97 (3.532) Cm (97-1x200.000)



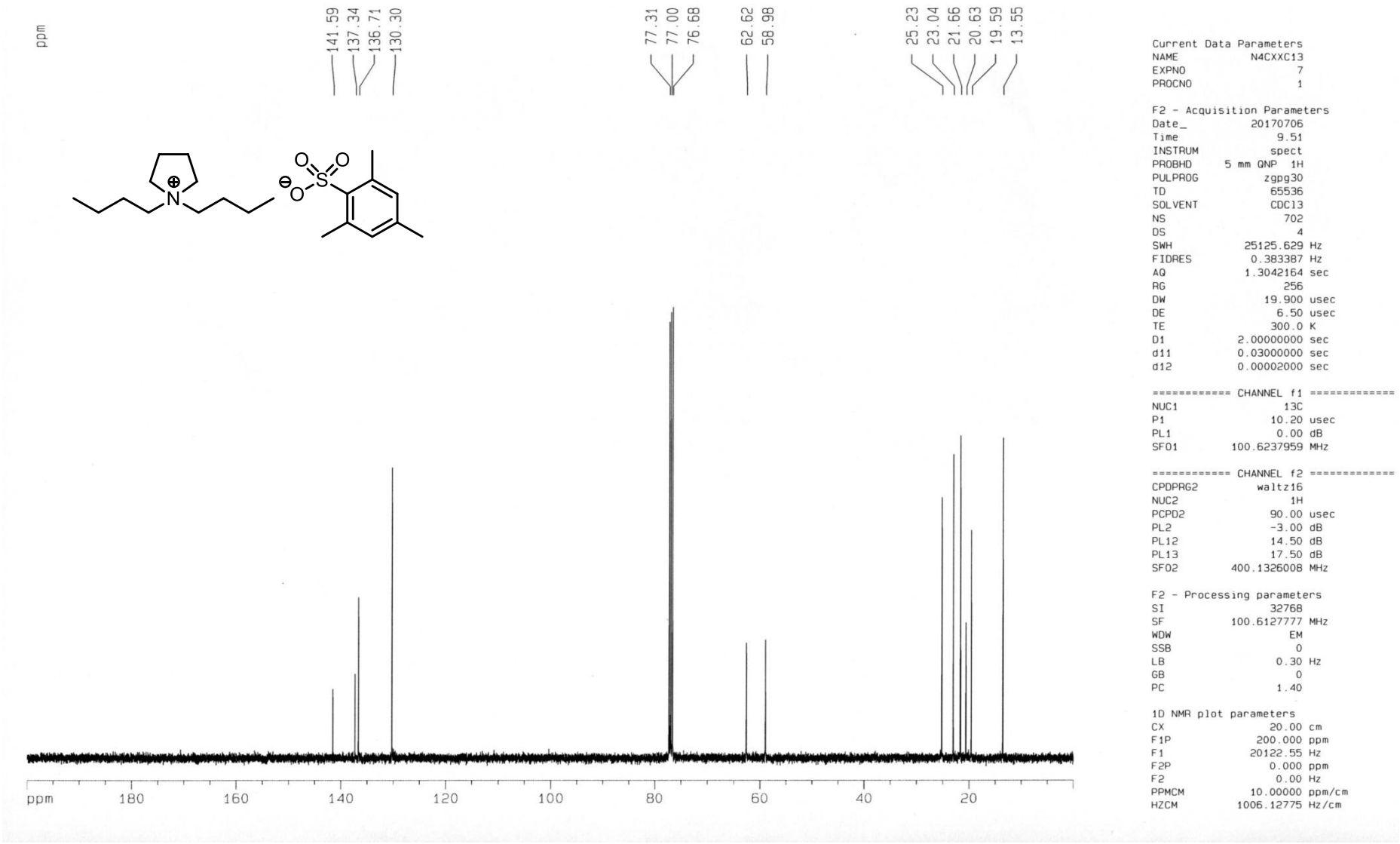
Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
296.3319	296.3317	0.2	0.7	0.5	45.0	0.0	C20 H42 N



Current Data Parameters
NAME N4C44-TMBS
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170414
Time 0.10 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME            N4CXXC13
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170706
Time                9.51
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  702
DS                    4
SWH           25125.629 Hz
FIDRES            0.383387 Hz
AQ              1.3042164 sec
RG                  256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

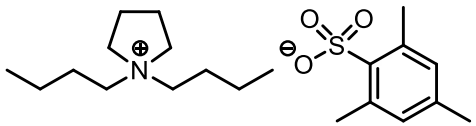
============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127777 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              200.000 ppm
F1             20122.55 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1006.12775 Hz/cm
```

1A (HR-ESI)

Elemental Composition Report

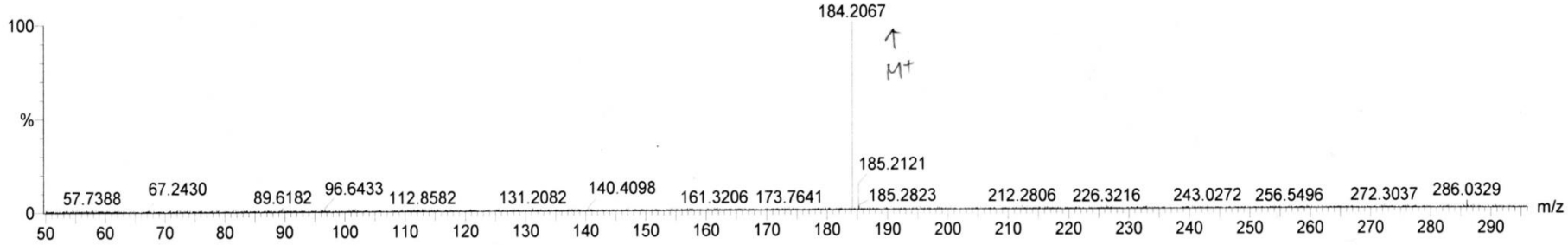


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

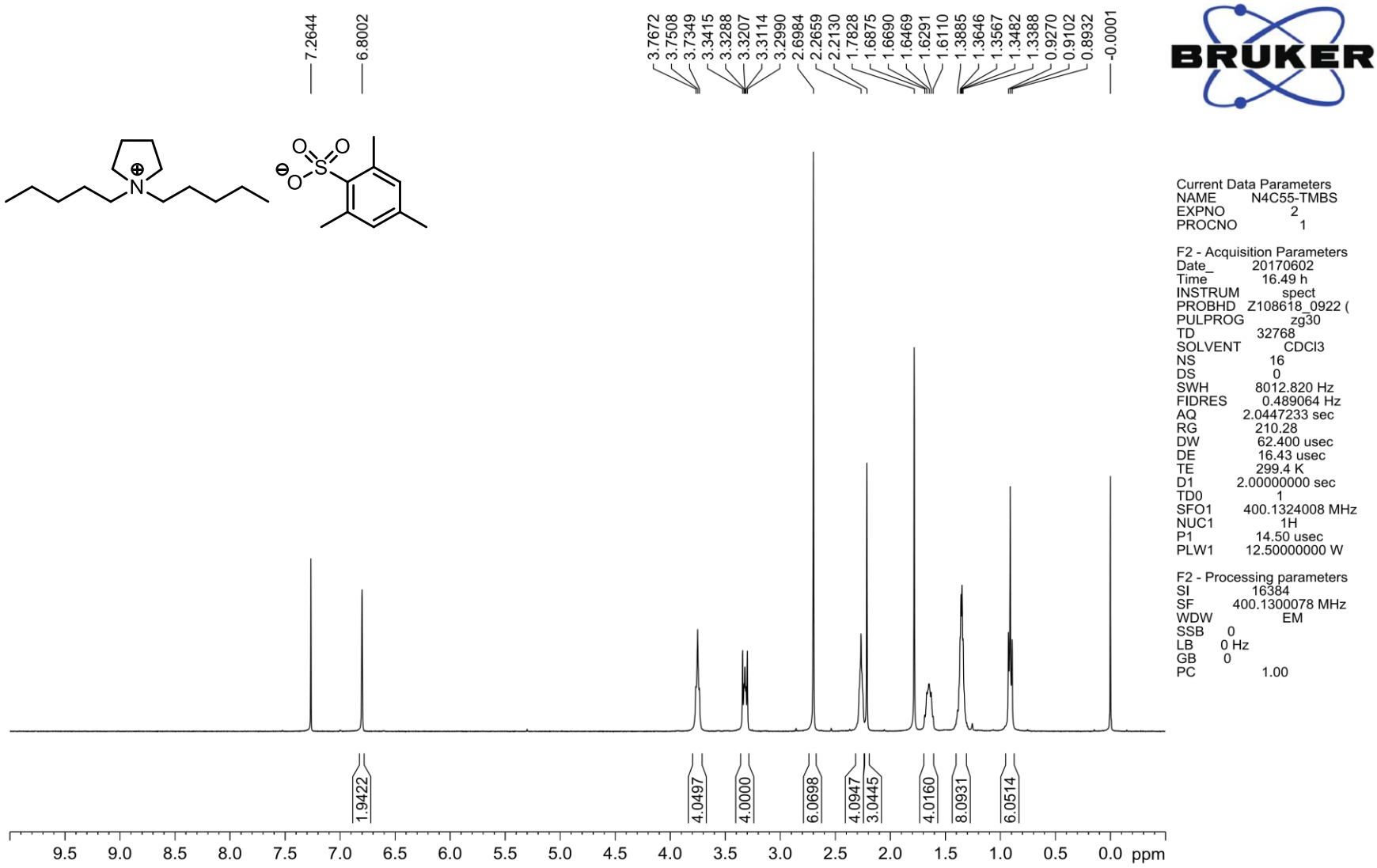
Monoisotopic Mass, Even Electron Ions
15 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

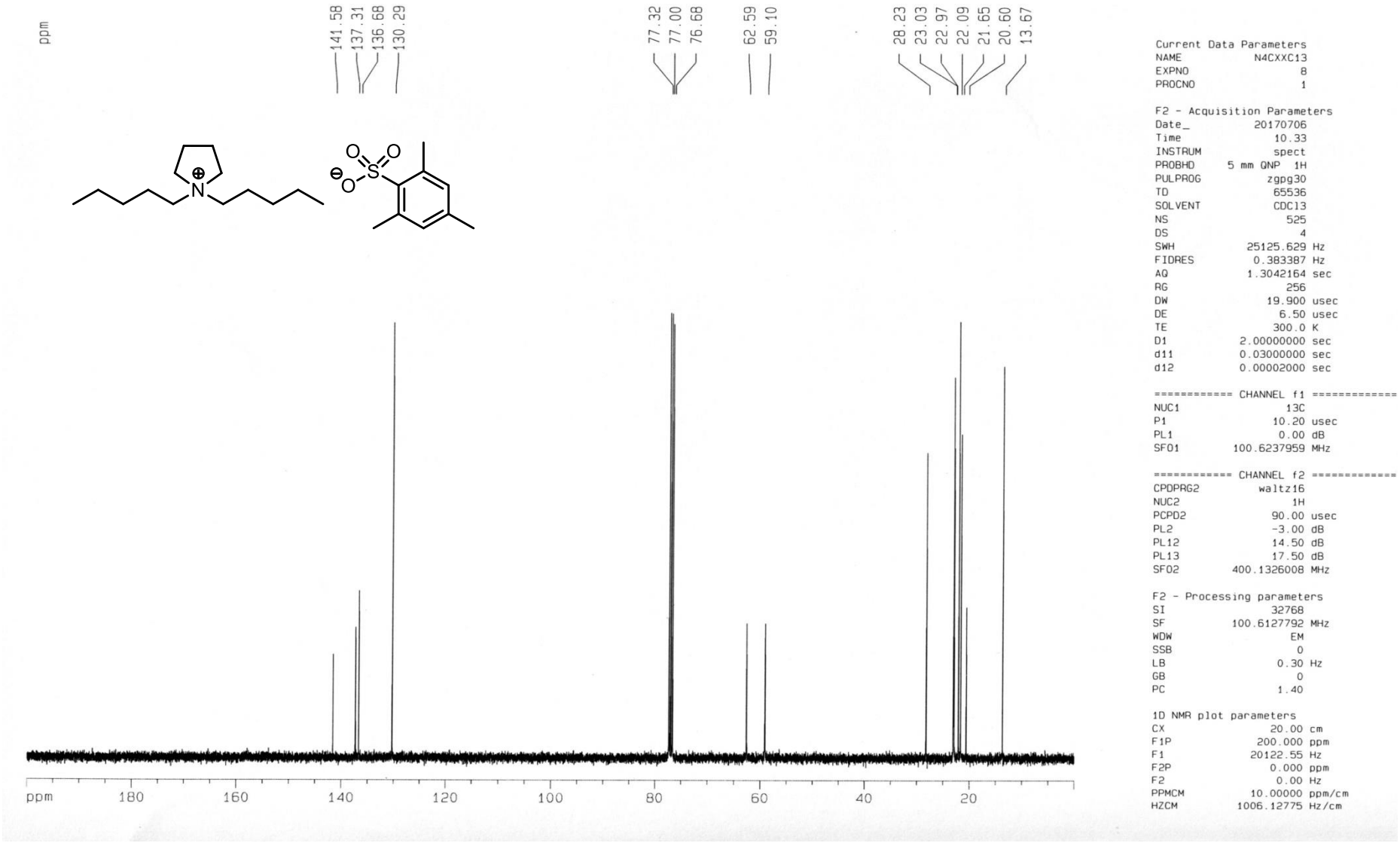
1A KE267 29-Jun-2017 13:51:26

0629_1A 43 (1.556) Cm (43-1) 1: TOF MS ES+ 3.54e+002



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
184.2067	184.2065	0.2	1.1	0.5	38.0	0.0	C12 H26 N


7.2644
6.8002
3.7672
3.7508
3.7349
3.3415
3.3288
3.3207
3.3114
3.2990
2.6984
2.2659
2.2130
1.7828
1.6875
1.6690
1.6469
1.6291
1.6110
1.3885
1.3646
1.3567
1.3482
1.3388
0.9270
0.9102
0.8932
-0.0001
BRUKER
Current Data Parameters
NAME N4C55-TMBS
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170602
Time 16.49 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.4 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9422
4.0497
4.0000
6.0698
4.0947
3.0445
4.0160
8.0931
6.0514
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                 N4CXXC13
EXPNO                       8
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170706
Time                   10.33
INSTRUM                spect
PROBHD     5 mm QNP  1H
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       525
DS                         4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                       256
DW                    19.900 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                      13C
P1                     10.20 usec
PL1                     0.00 dB
SFO1               100.6237959 MHz

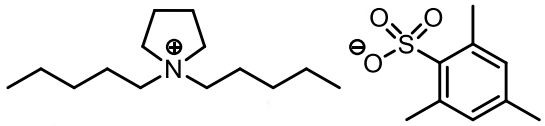
============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                      1H
PCPD2                  90.00 usec
PL2                    -3.00 dB
PL12                   14.50 dB
PL13                   17.50 dB
SFO2               400.1326008 MHz

F2 - Processing parameters
SI                        32768
SF                 100.6127792 MHz
WDW                          EM
SSB                           0
LB                     0.30 Hz
GB                            0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
F1P                  200.000 ppm
F1                 20122.55 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM               1006.12775 Hz/cm
```

2A (HR-ESI)

Elemental Composition Report



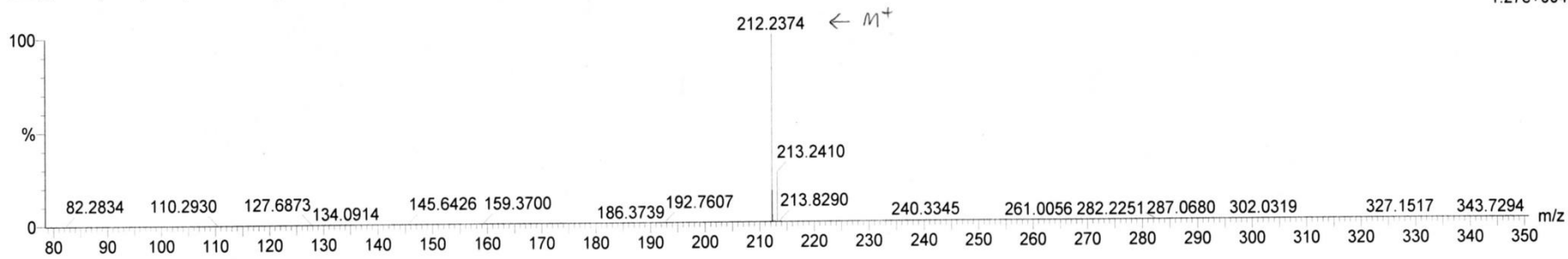
Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

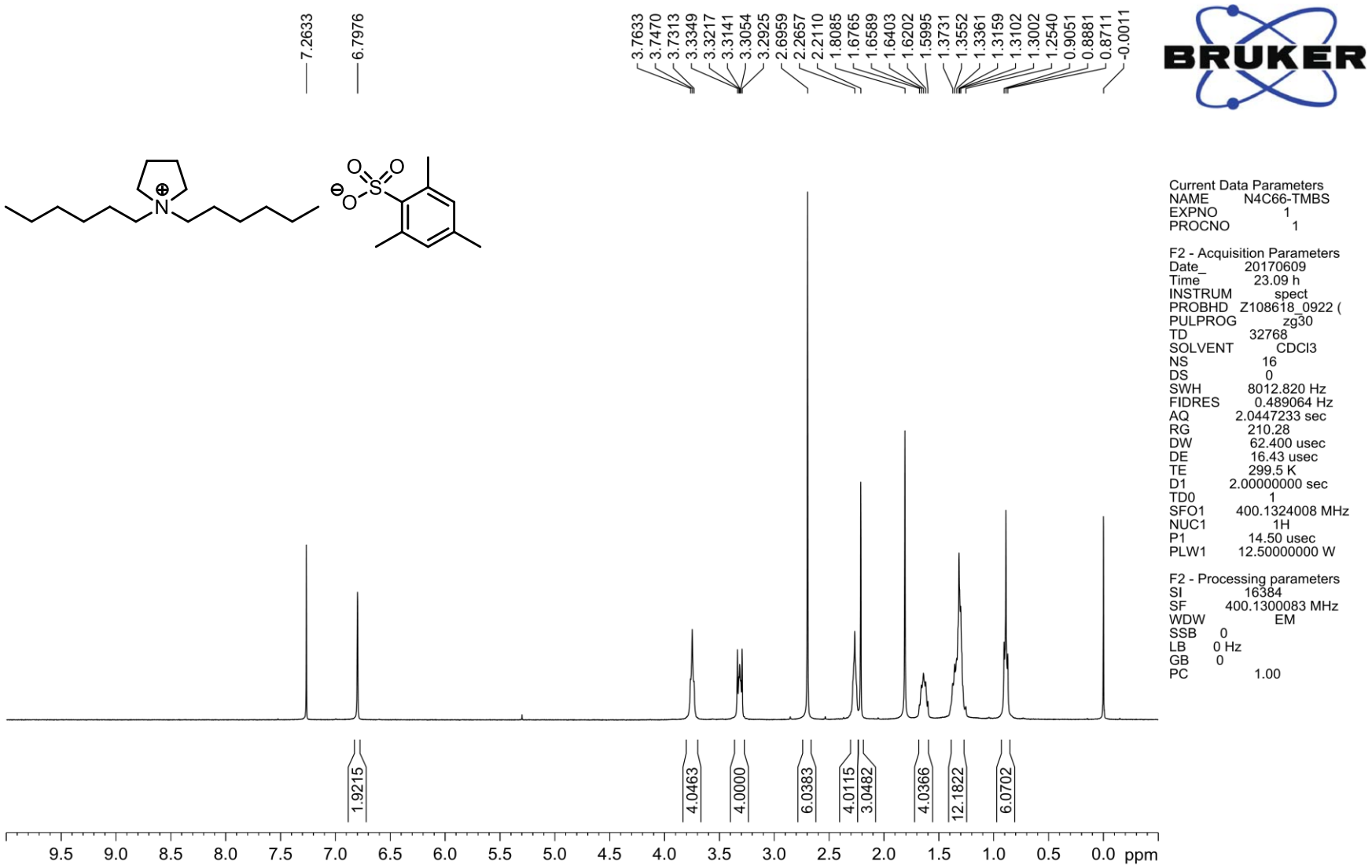
Monoisotopic Mass, Even Electron Ions
17 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

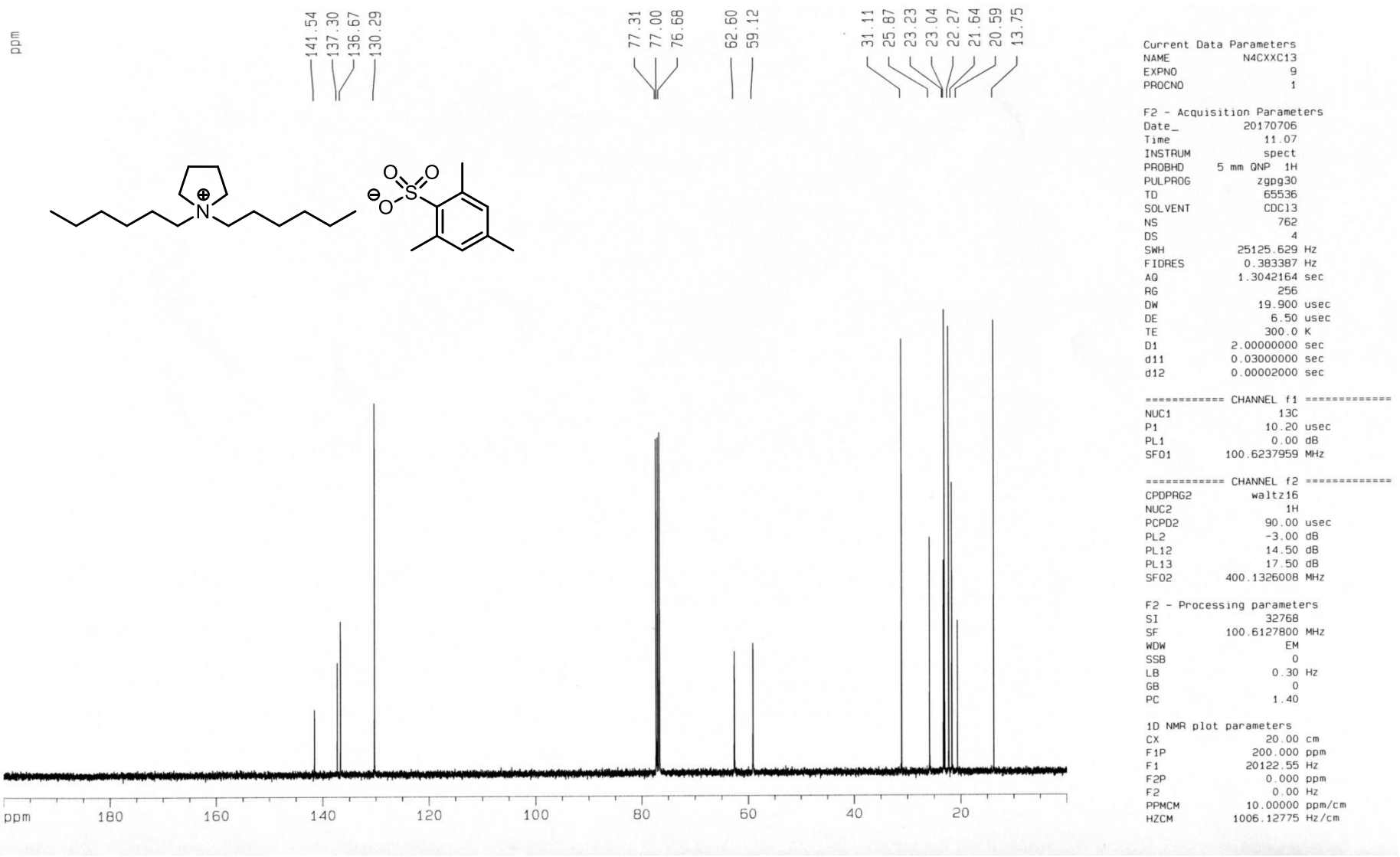
2A KE267 29-Jun-2017 13:10:57

0629_2A 49 (1.789) Cm (49-1x20.000) 1: TOF MS ES+ 1.27e+004



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
212.2374	212.2378	-0.4	-1.9	0.5	99.9	0.0	C14 H30 N


7.2633
6.7976
3.7633
3.7470
3.7313
3.3349
3.3217
3.3141
3.3054
3.2925
2.6959
2.2657
2.2110
1.8085
1.6765
1.6589
1.6403
1.6202
1.5995
1.3731
1.3552
1.3361
1.3159
1.3102
1.3002
1.2540
0.9051
0.8881
0.8711
-0.0011
BRUKER
Current Data Parameters
NAME N4C66-TMBS
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170609
Time 23.09 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.5 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9215
4.0463
4.0000
6.0383
4.0115
3.0482
4.0366
12.1822
6.0702
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                  N4CXXC13
EXPNO                        9
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20170706
Time                     11.07
INSTRUM                  spect
PROBHD       5 mm QNP  1H
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          762
DS                            4
SWH                 25125.629 Hz
FIDRES                0.383387 Hz
AQ                   1.3042164 sec
RG                          256
DW                     19.900 usec
DE                      6.50 usec
TE                     300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
d12                 0.00002000 sec

============ CHANNEL f1 =============
NUC1                        13C
P1                       10.20 usec
PL1                       0.00 dB
SFO1                100.6237959 MHz

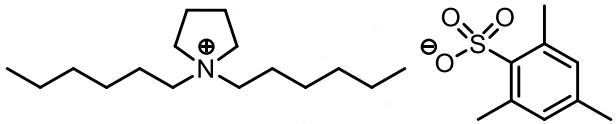
============ CHANNEL f2 =============
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    90.00 usec
PL2                      -3.00 dB
PL12                     14.50 dB
PL13                     17.50 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                         32768
SF                  100.6127800 MHz
WDW                           EM
SSB                            0
LB                        0.30 Hz
GB                             0
PC                        1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     200.000 ppm
F1                    20122.55 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM                1006.12775 Hz/cm
```

3A (HR-ESI)

Elemental Composition Report



Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

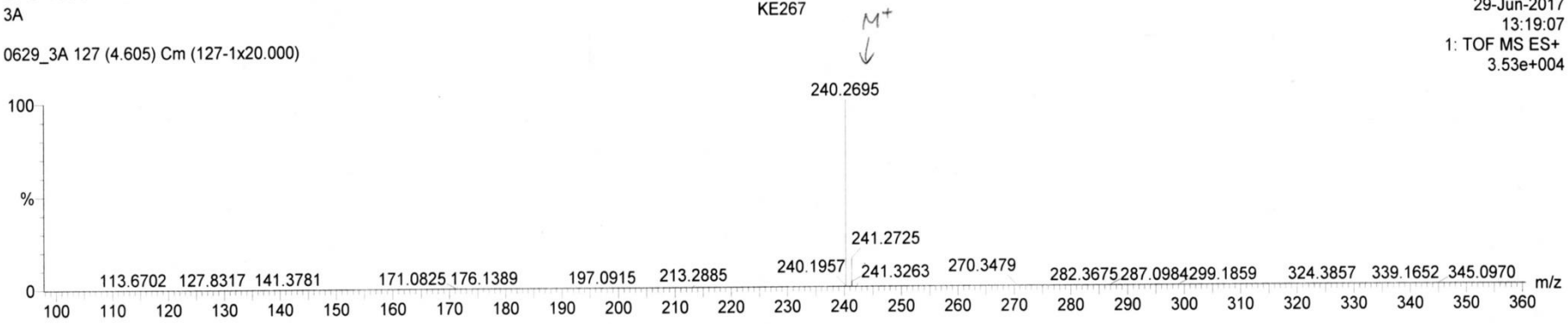
Monoisotopic Mass, Even Electron Ions
19 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

3A

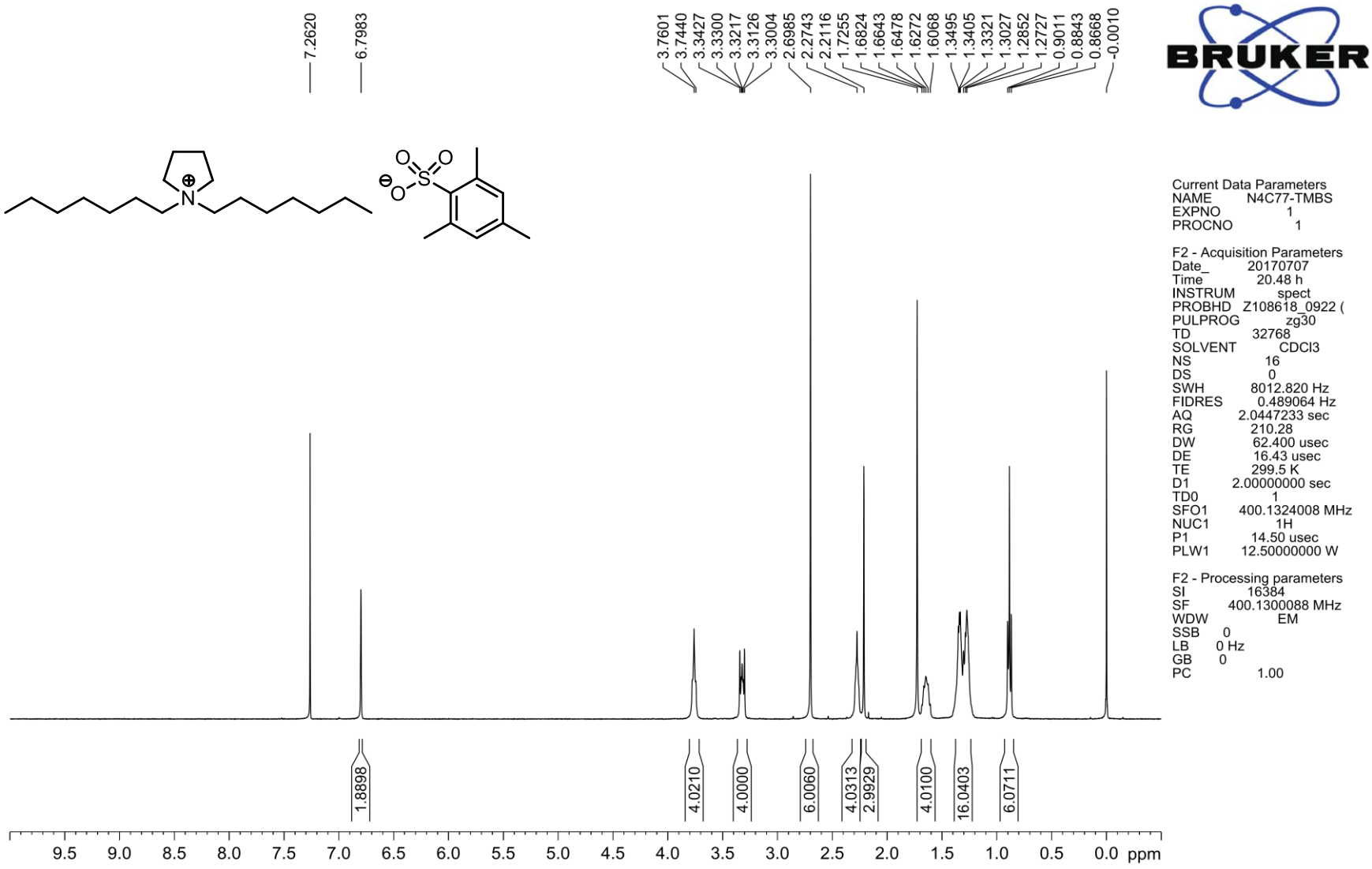
KE267

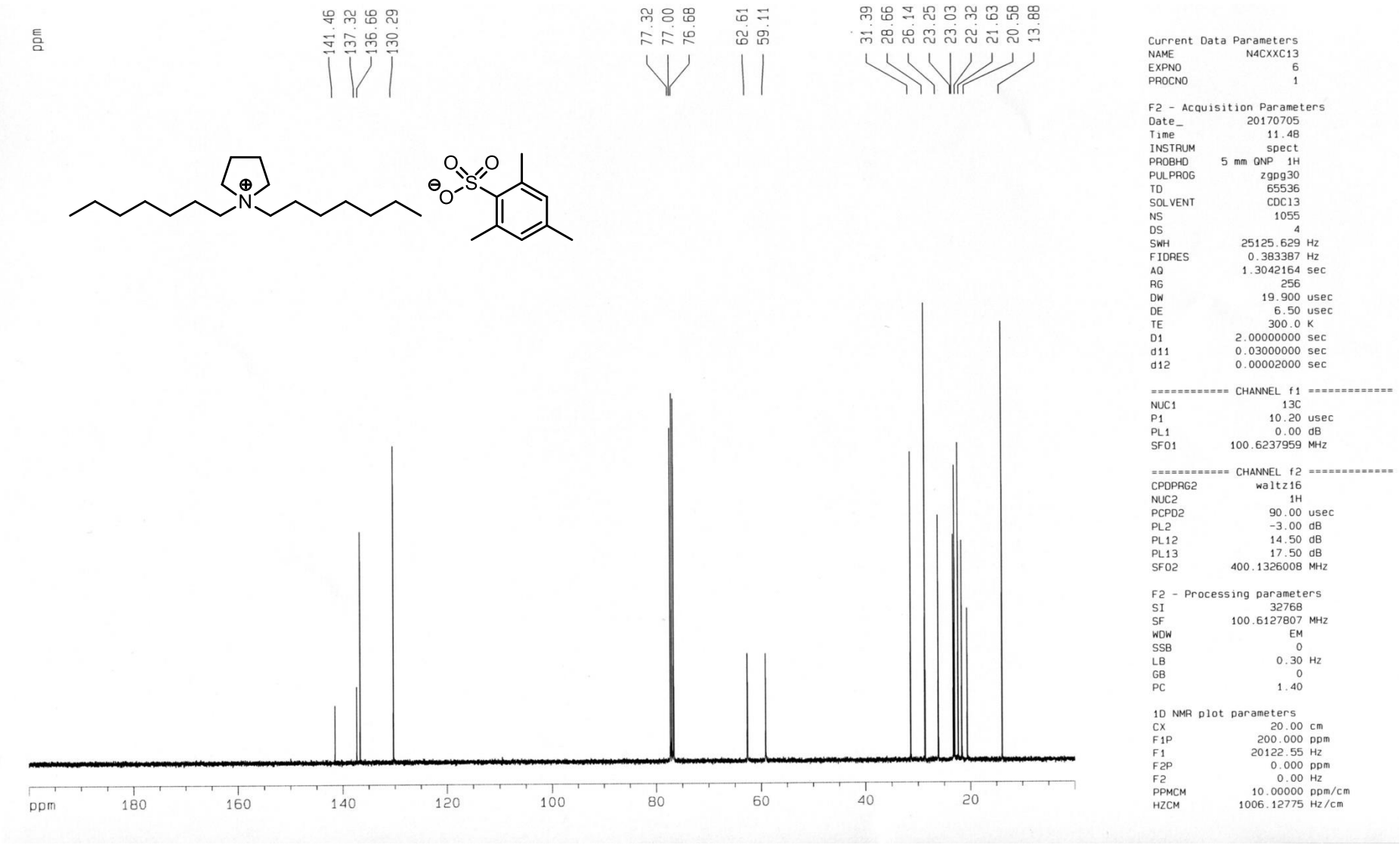
29-Jun-2017
13:19:07
1: TOF MS ES+
3.53e+004

0629_3A 127 (4.605) Cm (127-1x20.000)



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
240.2695	240.2691	0.4	1.7	0.5	66.4	0.0	C16 H34 N


7.2620
6.7983
3.7601
3.7440
3.3427
3.3300
3.3217
3.3126
3.3004
2.6985
2.2743
2.2116
1.7255
1.6824
1.6643
1.6478
1.6272
1.6068
1.3495
1.3405
1.3321
1.3027
1.2852
1.2727
0.9011
0.8843
0.8668
-0.0010
BRUKER
Current Data Parameters
NAME N4C77-TMBS
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170707
Time 20.48 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.5 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.8898
4.0210
4.0000
6.0060
4.0313
2.9929
4.0100
16.0403
6.0711
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                  N4CXXC13
EXPNO                        6
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20170705
Time                     11.48
INSTRUM                  spect
PROBHD       5 mm QNP  1H
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1055
DS                          4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                        256
DW                   19.900 usec
DE                    6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                      13C
P1                   10.20 usec
PL1                   0.00 dB
SFO1              100.6237959 MHz

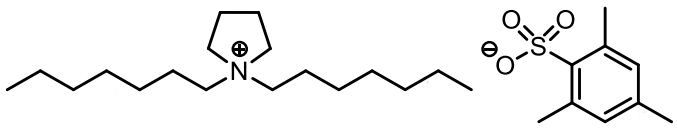
============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                90.00 usec
PL2                  -3.00 dB
PL12                 14.50 dB
PL13                 17.50 dB
SFO2              400.1326008 MHz

F2 - Processing parameters
SI                        32768
SF                100.6127807 MHz
WDW                          EM
SSB                           0
LB                    0.30 Hz
GB                            0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                 200.000 ppm
F1                20122.55 Hz
F2P                   0.000 ppm
F2                    0.00 Hz
PPMCM             10.00000 ppm/cm
HZCM              1006.12775 Hz/cm
```

4A (HR-ESI)

Elemental Composition Report



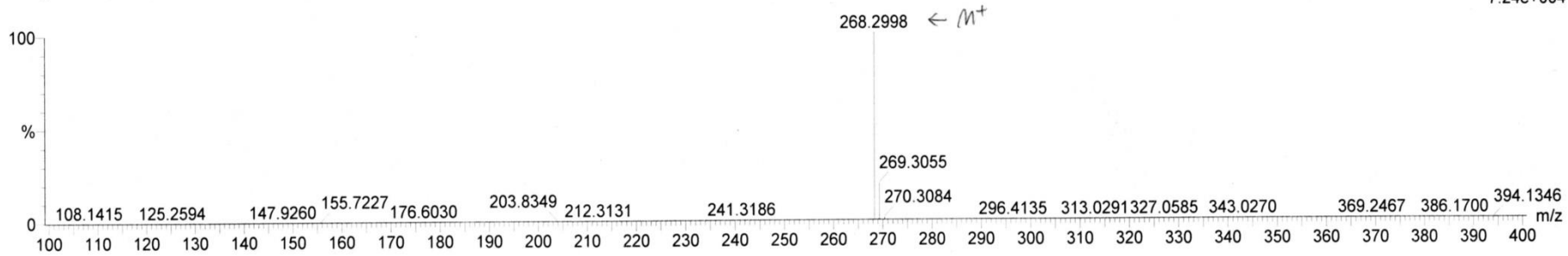
Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

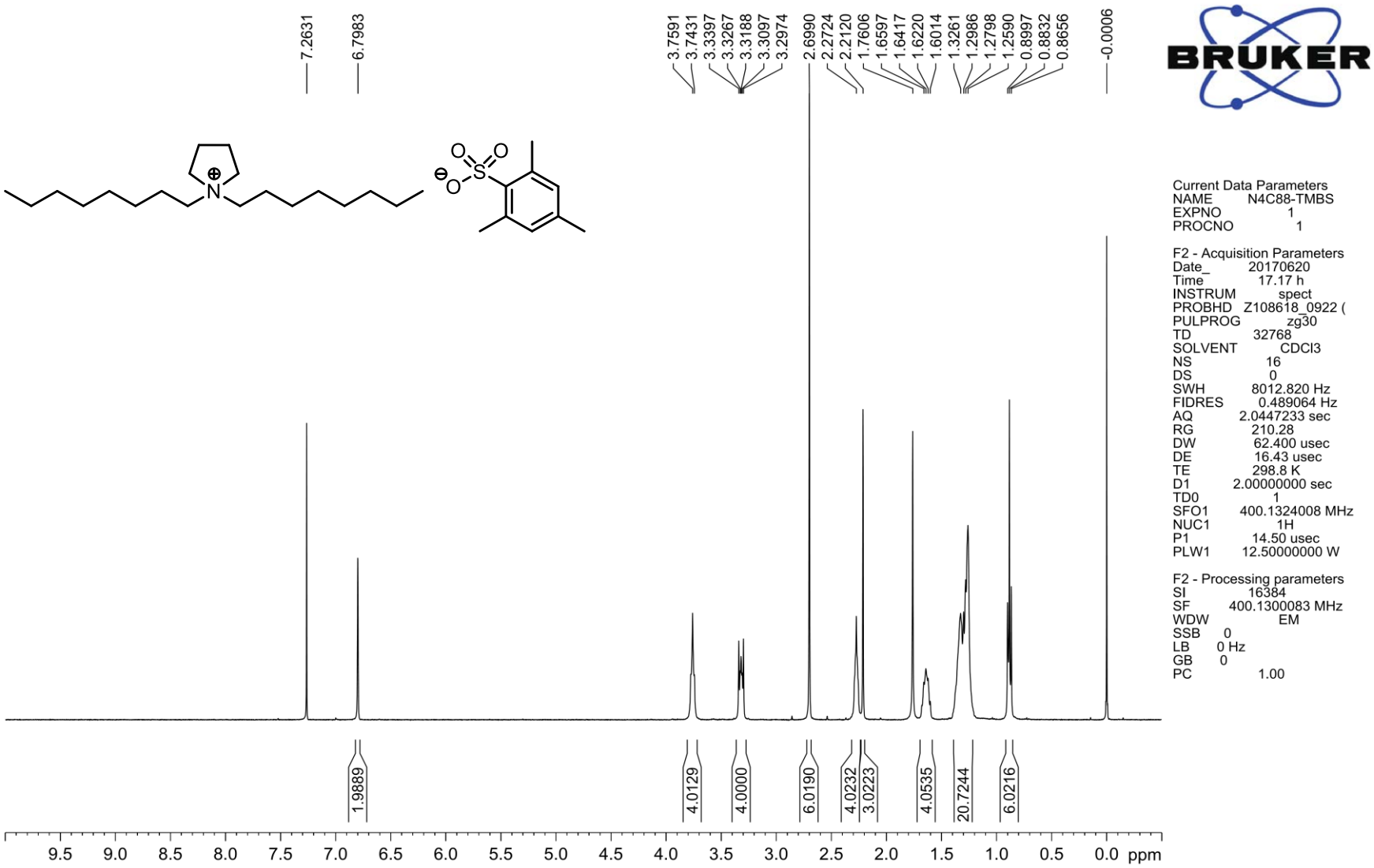
Monoisotopic Mass, Even Electron Ions
22 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

4A KE267 29-Jun-2017 13:27:11

0629_4A 141 (5.129) Cm (140:141-1x50.000) 1: TOF MS ES+ 7.24e+004



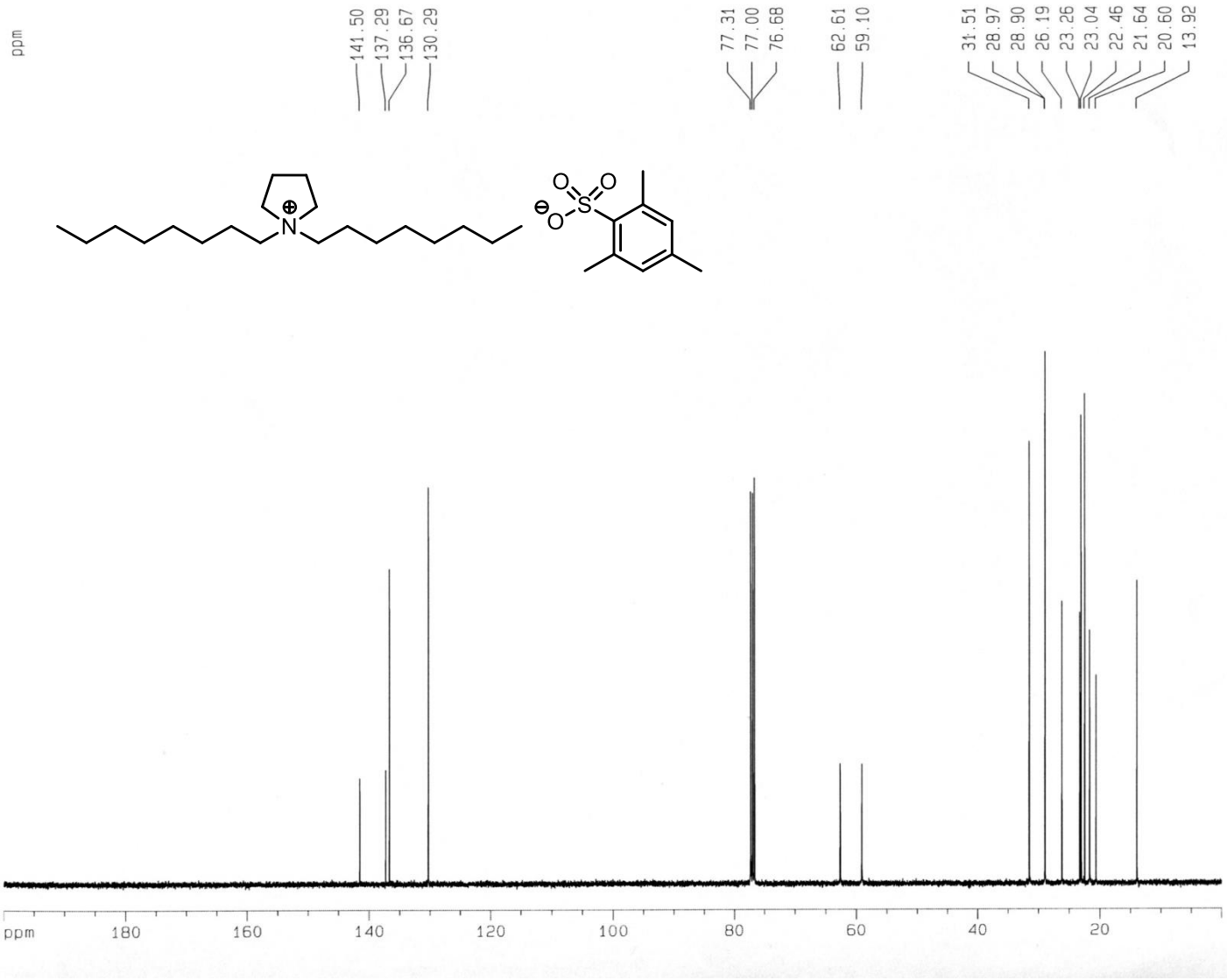
Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
268.2998	268.3004	-0.6	-2.2	0.5	115.2	0.0	C18 H38 N



Current Data Parameters
NAME N4C88-TMBS
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170620
Time 17.17 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 298.8 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

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Current Data Parameters
NAME                  N4CXXC13
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20170706
Time                     11.51
INSTRUM                  spect
PROBHD       5 mm QNP  1H
PULPROG                zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1162
DS                            4
SWH                 25125.629 Hz
FIDRES                0.383387 Hz
AQ                   1.3042164 sec
RG                          256
DW                       19.900 usec
DE                        6.50 usec
TE                       300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
d12                 0.00002000 sec

============ CHANNEL f1 =============
NUC1                        13C
P1                        10.20 usec
PL1                        0.00 dB
SFO1                100.6237959 MHz

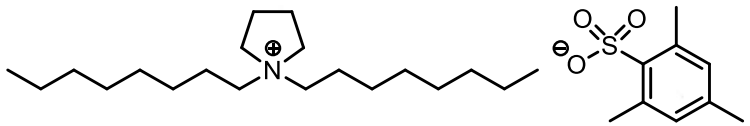
============ CHANNEL f2 =============
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     90.00 usec
PL2                       -3.00 dB
PL12                      14.50 dB
PL13                      17.50 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                         32768
SF                  100.6127800 MHz
WDW                           EM
SSB                            0
LB                        0.30 Hz
GB                             0
PC                         1.40

1D NMR plot parameters
CX                        20.00 cm
F1P                      200.000 ppm
F1                     20122.55 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                 10.00000 ppm/cm
HZCM                1006.12775 Hz/cm
```

5A (HR-ESI)

Elemental Composition Report

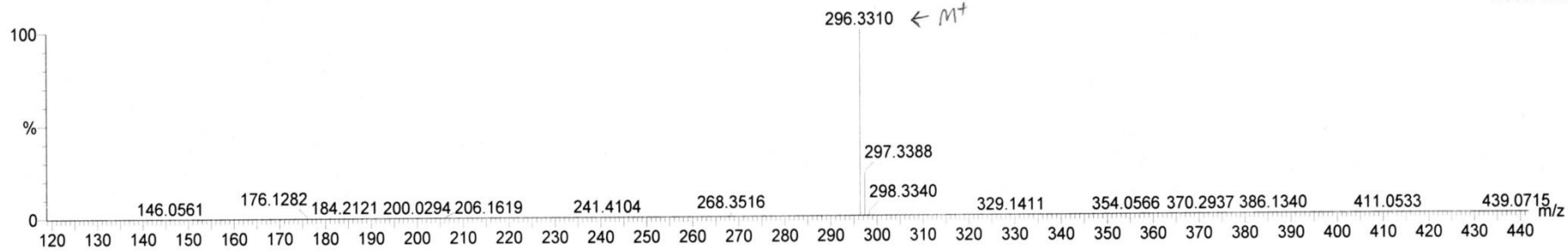


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

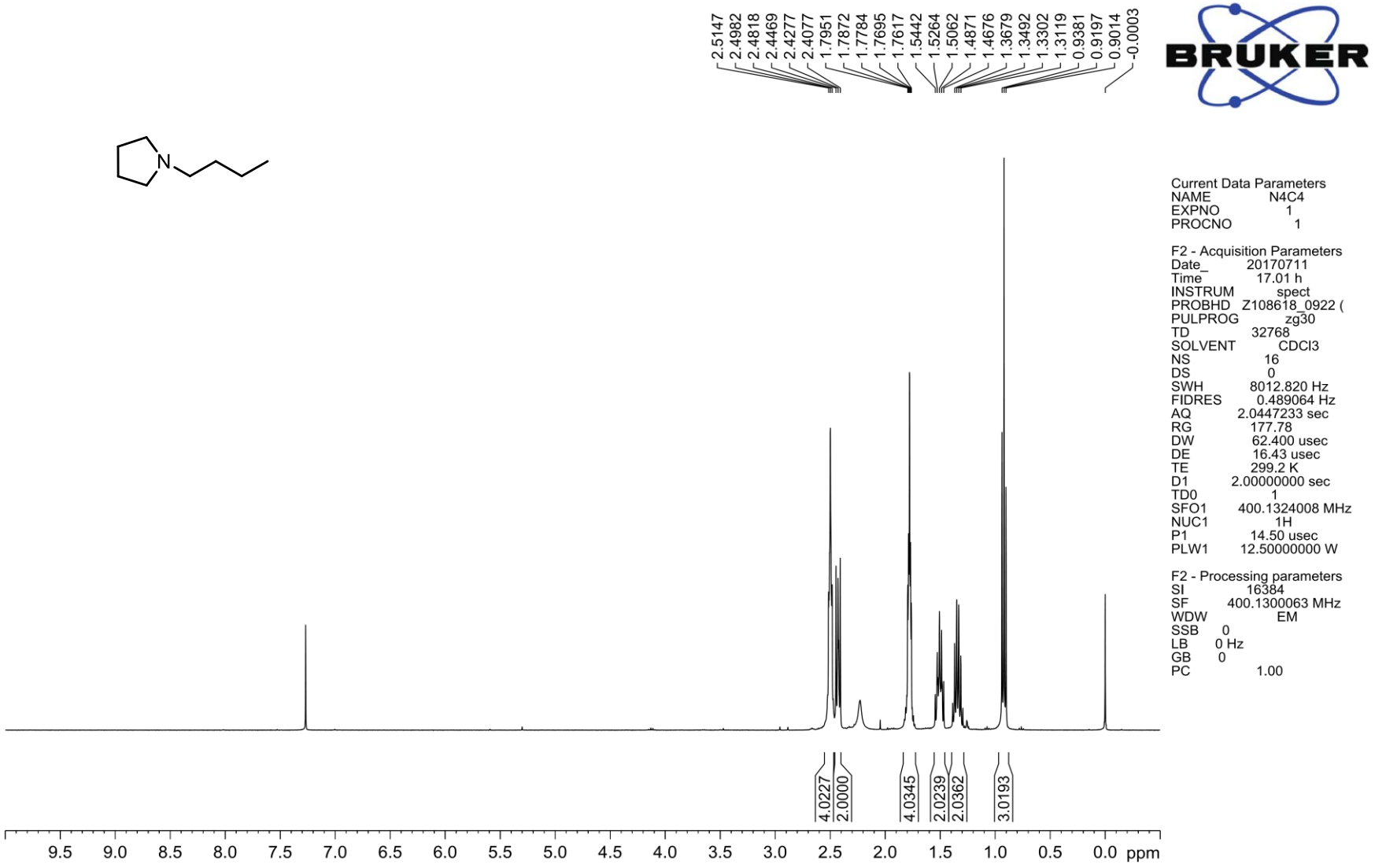
Monoisotopic Mass, Even Electron Ions
24 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

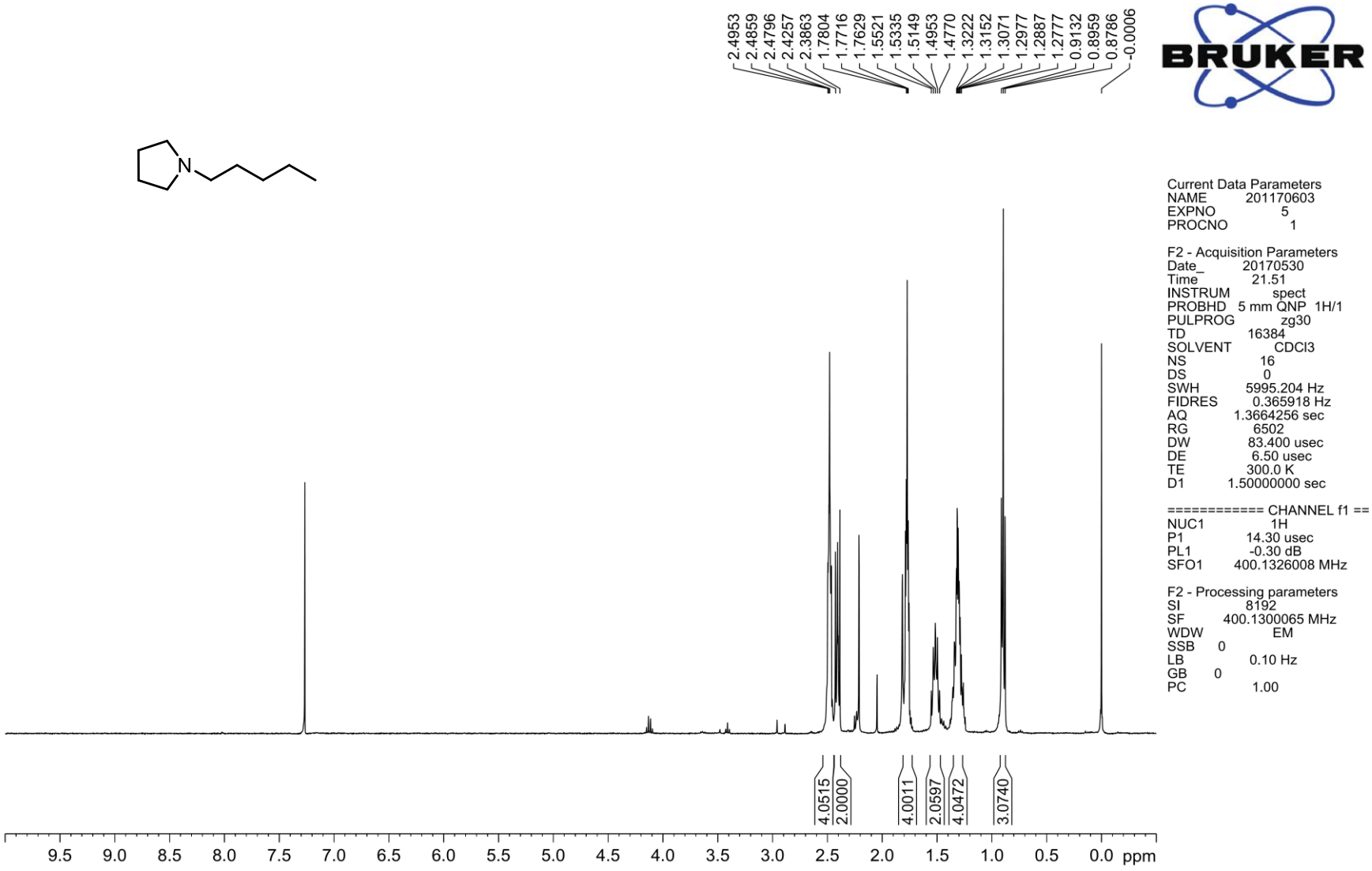
5A KE267 29-Jun-2017 13:35:14

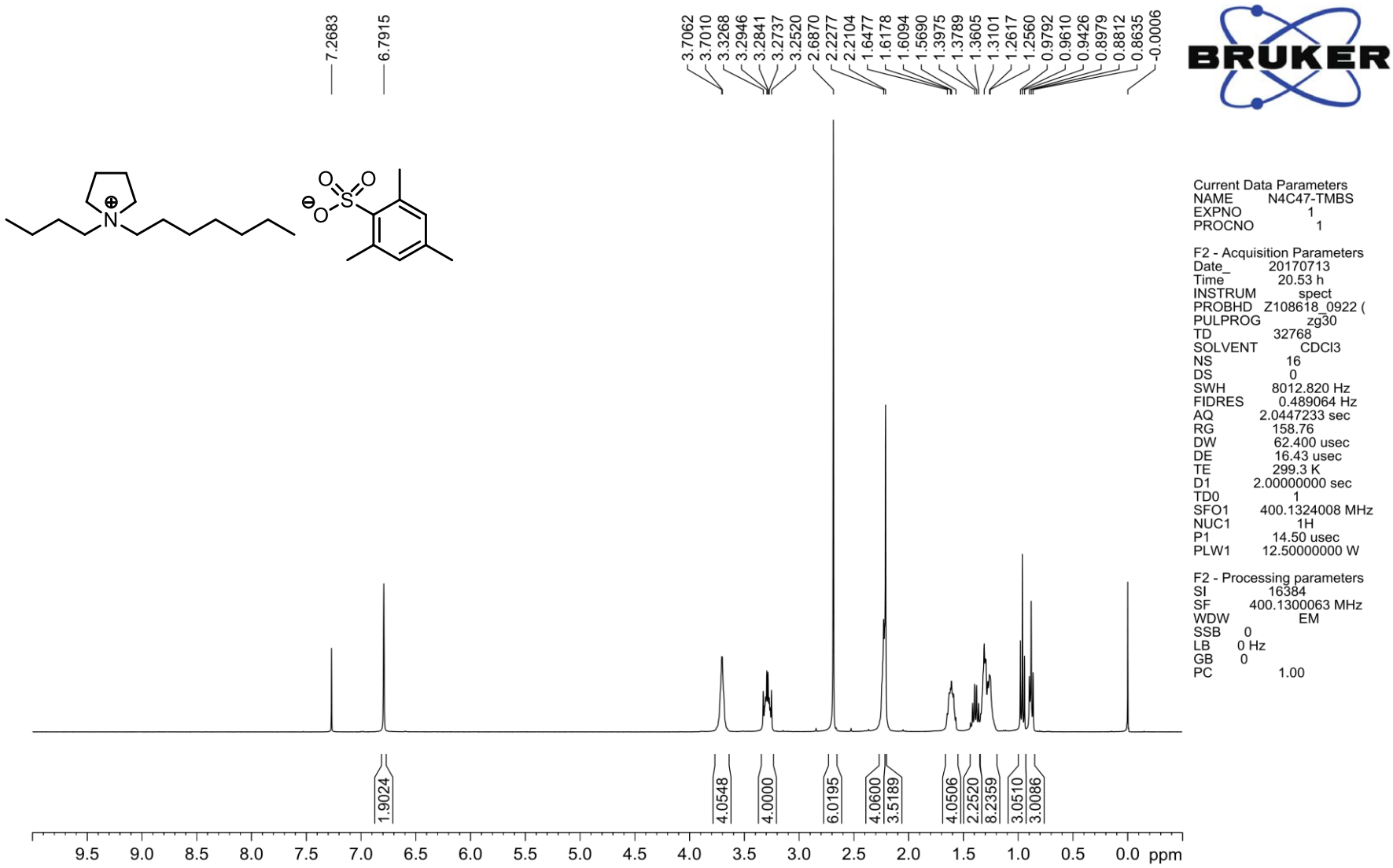
0629_5A 118 (4.288) Cm (118-1x20.000) 1: TOF MS ES+ 7.56e+004

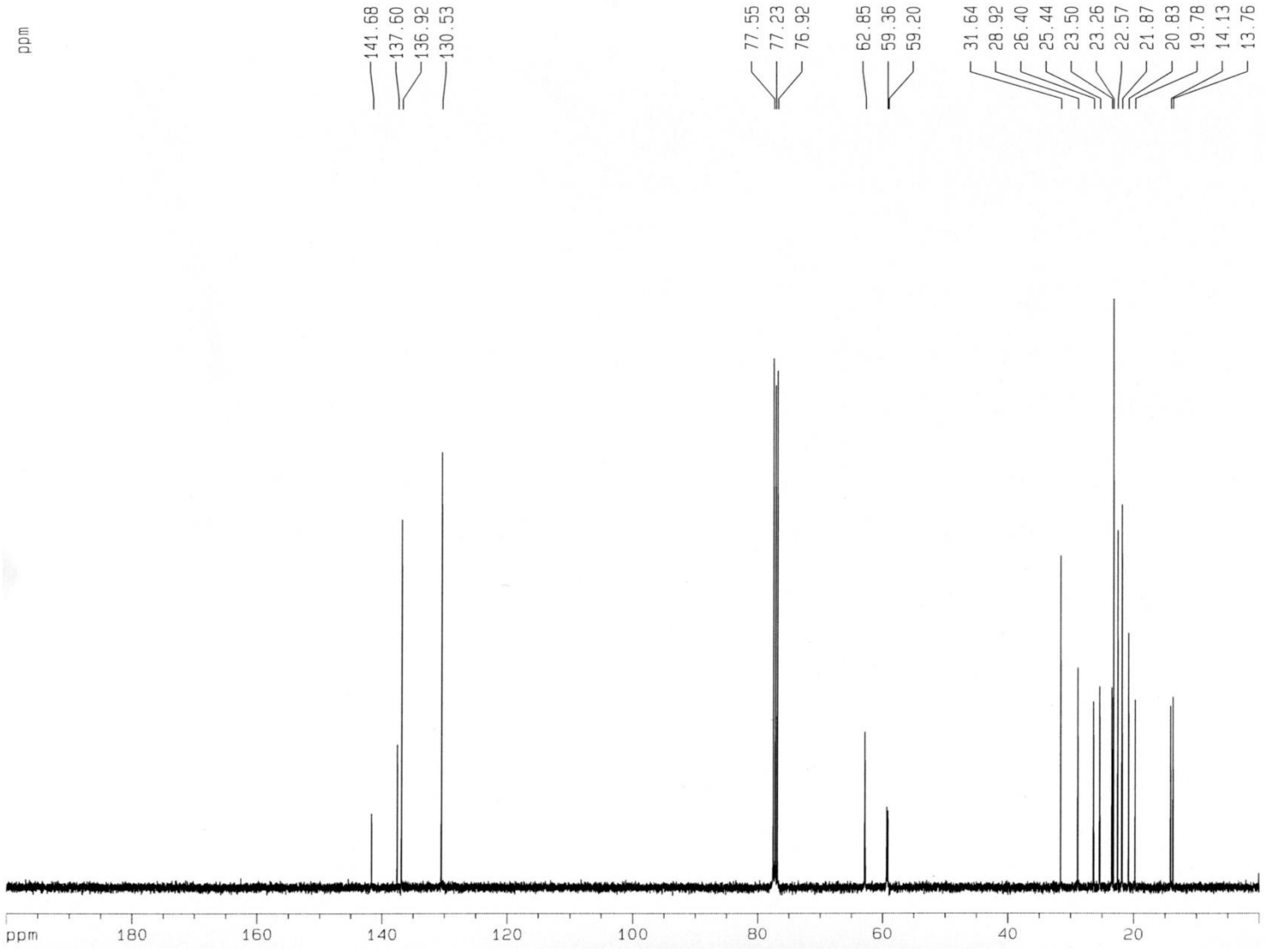


Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
296.3310	296.3317	-0.7	-2.4	0.5	102.0	0.0	C20 H42 N


2.5147
2.4982
2.4818
2.4469
2.4277
2.4077
1.7951
1.7872
1.7784
1.7695
1.7617
1.5442
1.5264
1.5062
1.4871
1.4676
1.3679
1.3492
1.3302
1.3119
0.9381
0.9197
0.9014
-0.0003
BRUKER
N
Current Data Parameters
NAME N4C4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170711
Time 17.01 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 177.78
DW 62.400 usec
DE 16.43 usec
TE 299.2 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300063 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
4.0227
2.0000
4.0345
2.0239
2.0362
3.0193
9.5 9.0 8.5 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 ppm


2.4953
2.4859
2.4796
2.4257
2.3863
1.7804
1.7716
1.7629
1.5521
1.5335
1.5149
1.4953
1.4770
1.3222
1.3152
1.3071
1.2977
1.2887
1.2777
0.9132
0.8959
0.8786
-0.0006
BRUKER
N
Current Data Parameters
NAME 201170603
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170530
Time 21.51
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664256 sec
RG 6502
DW 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec
============ CHANNEL f1 ==
NUC1 1H
P1 14.30 usec
PL1 -0.30 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 8192
SF 400.1300065 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
4.0515
2.0000
4.0011
2.0597
4.0472
3.0740
9.5 9.0 8.5 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 ppm


7.2683
6.7915
3.7062
3.7010
3.3268
3.2946
3.2841
3.2737
3.2520
2.6870
2.2277
2.2104
1.6477
1.6178
1.6094
1.5690
1.3975
1.3789
1.3605
1.3101
1.2617
1.2560
0.9792
0.9610
0.9426
0.8979
0.8812
0.8635
-0.0006
BRUKER
Current Data Parameters
NAME N4C47-TMBS
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170713
Time 20.53 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 158.76
DW 62.400 usec
DE 16.43 usec
TE 299.3 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300063 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9024
4.0548
4.0000
6.0195
4.0600
3.5189
4.0506
2.2520
8.2359
3.0510
3.0086
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME          N4CXXsingle
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170803
Time                10.54
INSTRUM             spect
PROBHD      5 mm QNP  1H
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   751
DS                     4
SWH            25125.629 Hz
FIDRES             0.383387 Hz
AQ              1.3042164 sec
RG                 1290.2
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
d12          0.00002000 sec

============ CHANNEL f1 =============
NUC1                  13C
P1                  10.20 usec
PL1                  0.00 dB
SF01          100.6237959 MHz

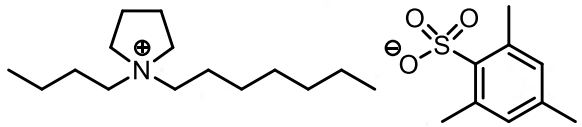
============ CHANNEL f2 =============
CPDPRG2           waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -3.00 dB
PL12                14.50 dB
PL13                17.50 dB
SF02          400.1326008 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127561 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
F1P               200.000 ppm
F1              20122.55 Hz
F2P                 0.000 ppm
F2                  0.00 Hz
PPMCM           10.00000 ppm/cm
HZCM          1006.12756 Hz/cm
```

1 (HR-APCI)

Elemental Composition Report



Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

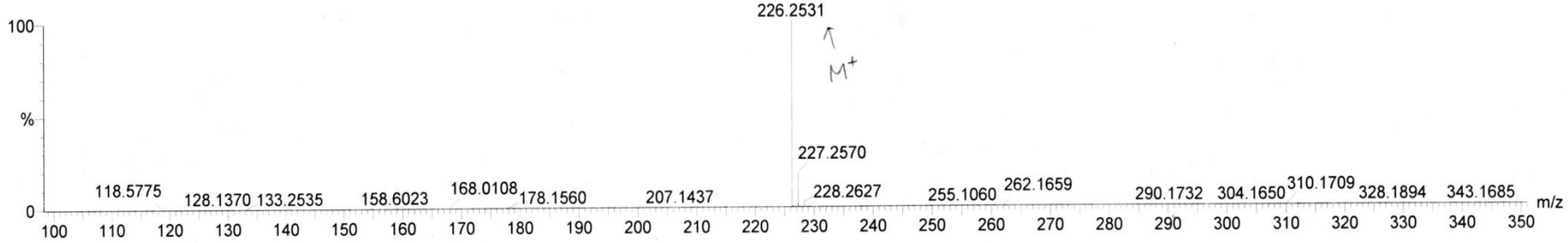
Monoisotopic Mass, Even Electron Ions
18 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

1

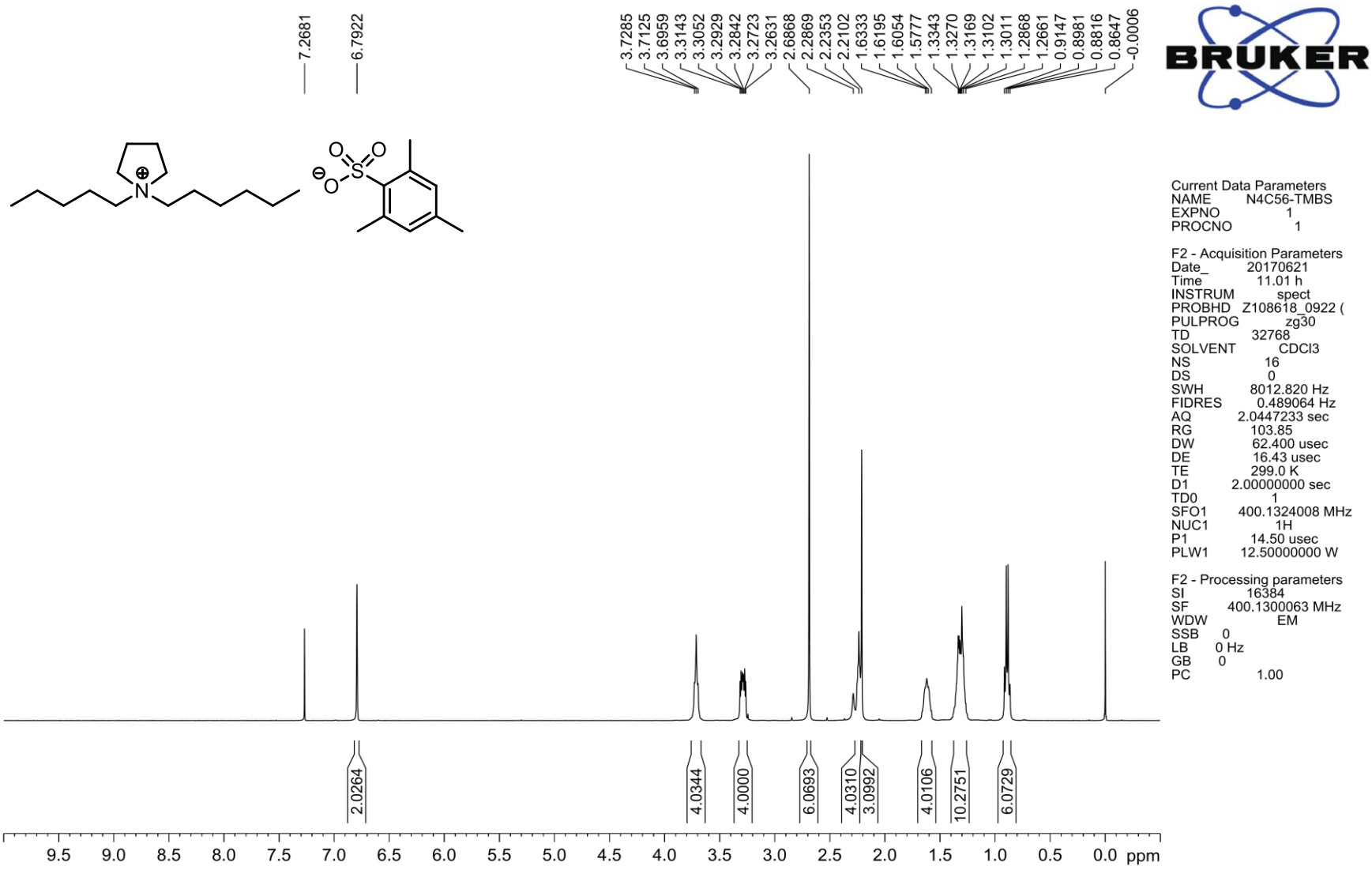
KE267

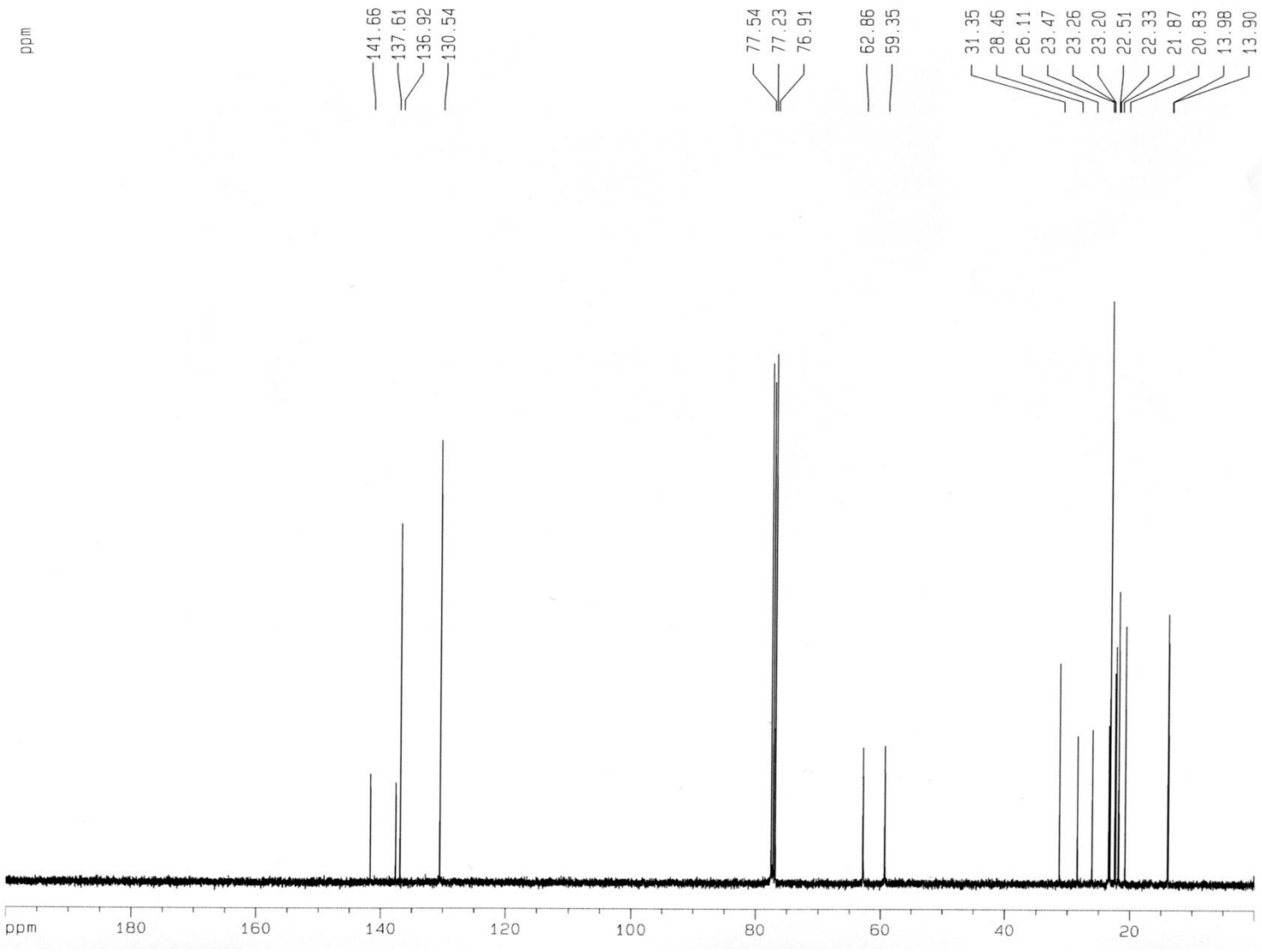
11-Aug-2017
12:16:08
2: TOF MS AP+
1.27e+004

0811_1 45 (1.305) Cm (44:45-1x5.000)



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
226.2531	226.2535	-0.4	-1.8	0.5	74.3	0.0	C15 H32 N


7.2681
6.7922
3.7285
3.7125
3.6959
3.3143
3.3052
3.2929
3.2842
3.2723
3.2631
2.6868
2.2869
2.2353
2.2102
1.6333
1.6195
1.6054
1.5777
1.3343
1.3270
1.3169
1.3102
1.3011
1.2868
1.2661
0.9147
0.8981
0.8816
0.8647
-0.0006
BRUKER
Current Data Parameters
NAME N4C56-TMBS
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170621
Time 11.01 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 103.85
DW 62.400 usec
DE 16.43 usec
TE 299.0 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300063 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
2.0264
4.0344
4.0000
6.0693
4.0310
3.0992
4.0106
10.2751
6.0729
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME          N4CXXsingle
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170803
Time                11.45
INSTRUM             spect
PROBHD      5 mm QNP  1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   862
DS                     4
SWH           25125.629 Hz
FIDRES            0.383387 Hz
AQ              1.3042164 sec
RG                2580.3
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 =============
NUC1                  13C
P1                 10.20 usec
PL1                 0.00 dB
SF01          100.6237959 MHz

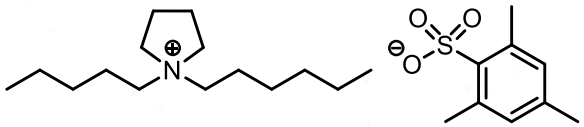
============ CHANNEL f2 =============
CPDPRG2           waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -3.00 dB
PL12               14.50 dB
PL13               17.50 dB
SF02          400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127561 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               200.000 ppm
F1              20122.55 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1006.12756 Hz/cm
```

2 (HR-ESI)

Elemental Composition Report



Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

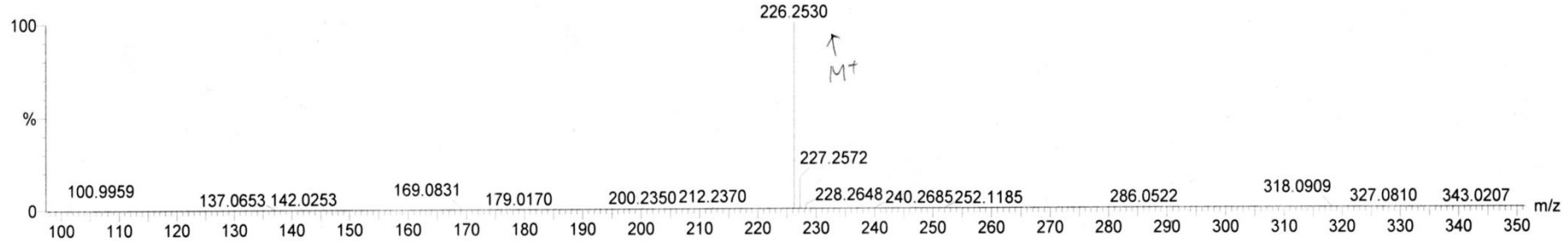
Monoisotopic Mass, Even Electron Ions
18 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

2

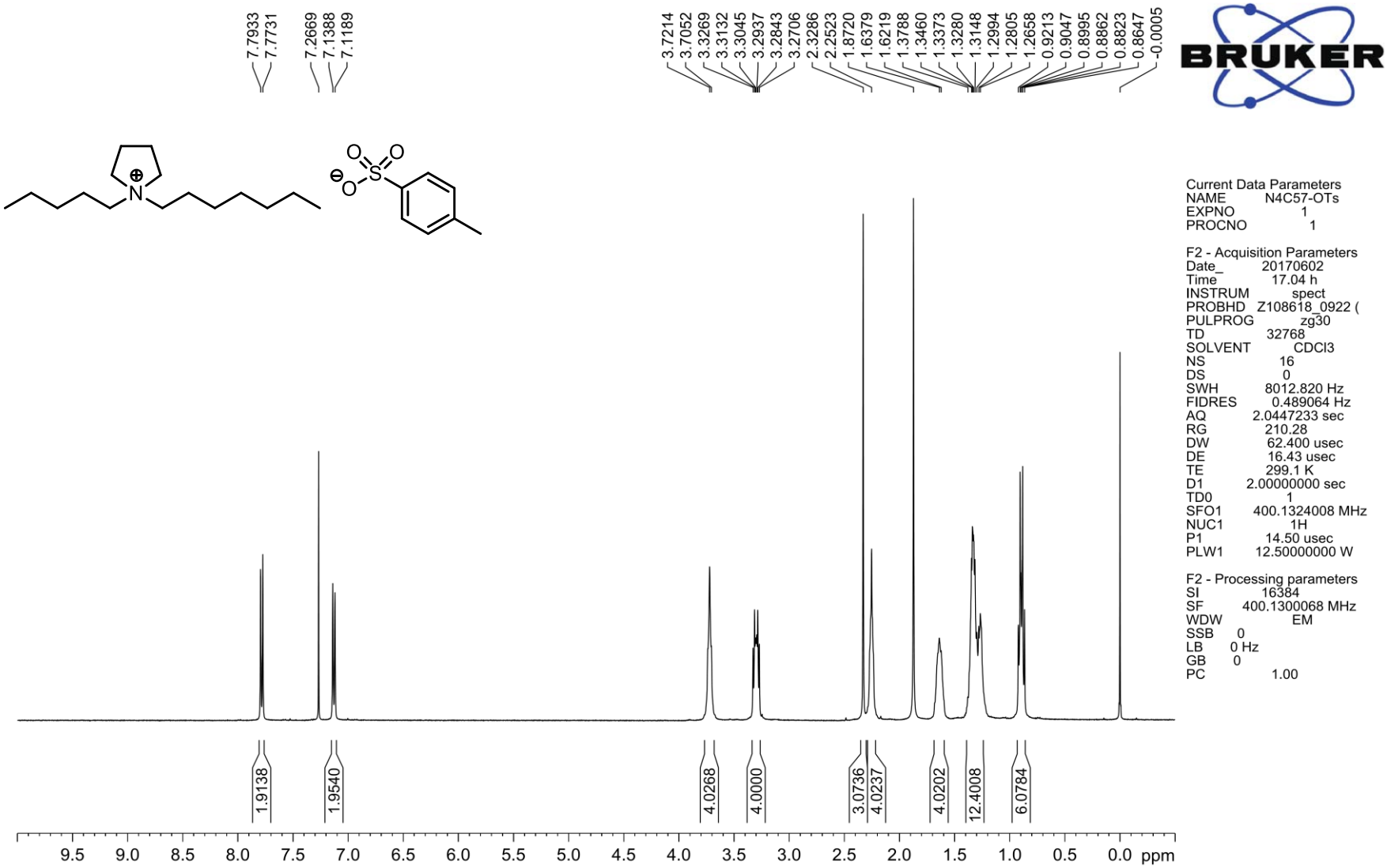
KE267

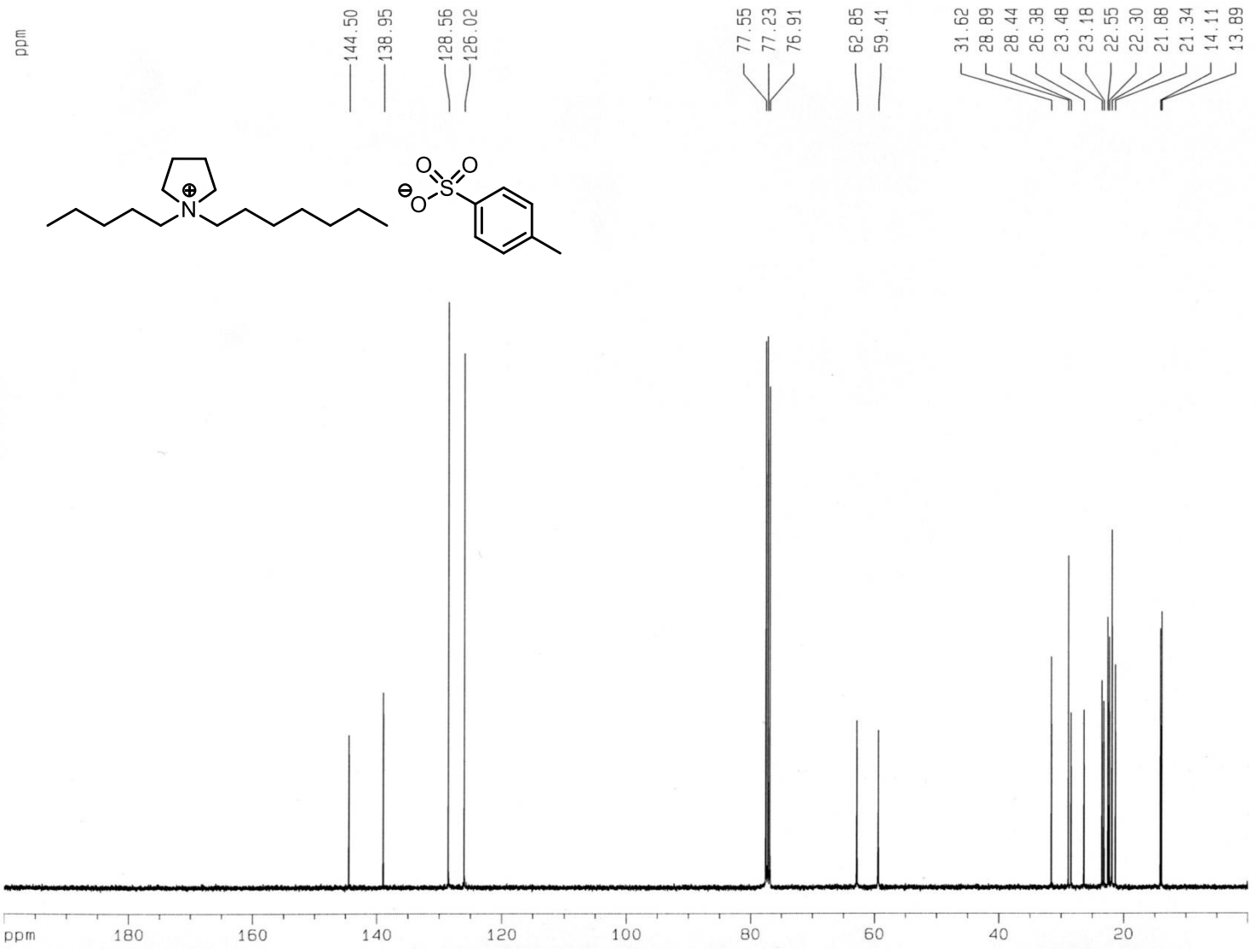
11-Aug-2017
15:06:58
1: TOF MS ES+
2.40e+004

0811_2_2 115 (4.169)



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
226.2530	226.2535	-0.5	-2.2	0.5	57.9	0.0	C15 H32 N


7.7933
7.7731
7.2669
7.1388
7.1189
3.7214
3.7052
3.3269
3.3132
3.3045
3.2937
3.2843
3.2706
2.3286
2.2523
1.8720
1.6379
1.6219
1.3788
1.3460
1.3373
1.3280
1.3148
1.2994
1.2805
1.2658
0.9213
0.9047
0.8995
0.8862
0.8823
0.8647
-0.0005
BRUKER
Current Data Parameters
NAME N4C57-OTs
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170602
Time 17.04 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.1 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300068 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9138
1.9540
4.0268
4.0000
3.0736
4.0237
4.0202
12.4008
6.0784
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME          N4CXXsingle
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170803
Time                12.37
INSTRUM             spect
PROBHD      5 mm QNP  1H
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    3072
DS                       4
SWH             25125.629 Hz
FIDRES              0.383387 Hz
AQ                1.3042164 sec
RG                   2896.3
DW                  19.900 usec
DE                   6.50 usec
TE                  300.0 K
D1              2.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

============ CHANNEL f1 =============
NUC1                   13C
P1                   10.20 usec
PL1                   0.00 dB
SF01           100.6237959 MHz

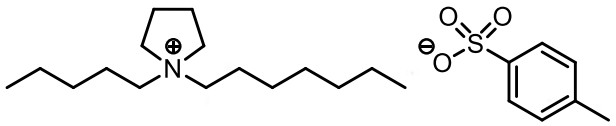
============ CHANNEL f2 =============
CPDPRG2            waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                  -3.00 dB
PL12                 14.50 dB
PL13                 17.50 dB
SF02           400.1326008 MHz

F2 - Processing parameters
SI                    32768
SF             100.6127576 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                200.000 ppm
F1               20122.55 Hz
F2P                  0.000 ppm
F2                   0.00 Hz
PPMCM             10.00000 ppm/cm
HZCM           1006.12756 Hz/cm
```

3 (HR-APCI)

Elemental Composition Report



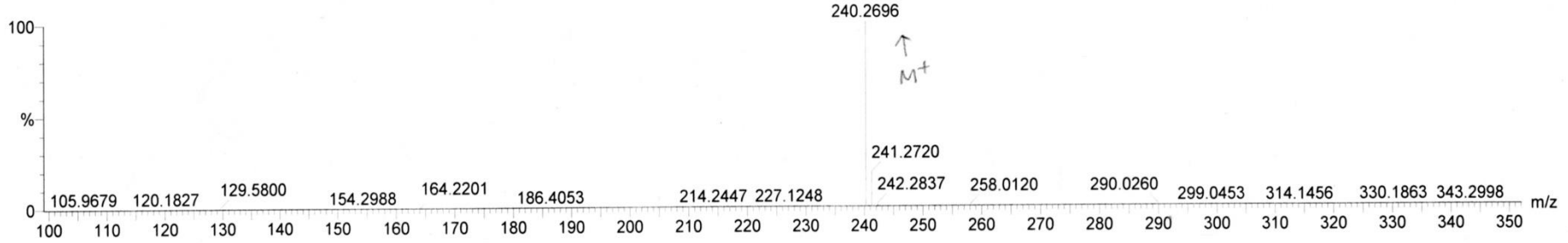
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
19 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1
3

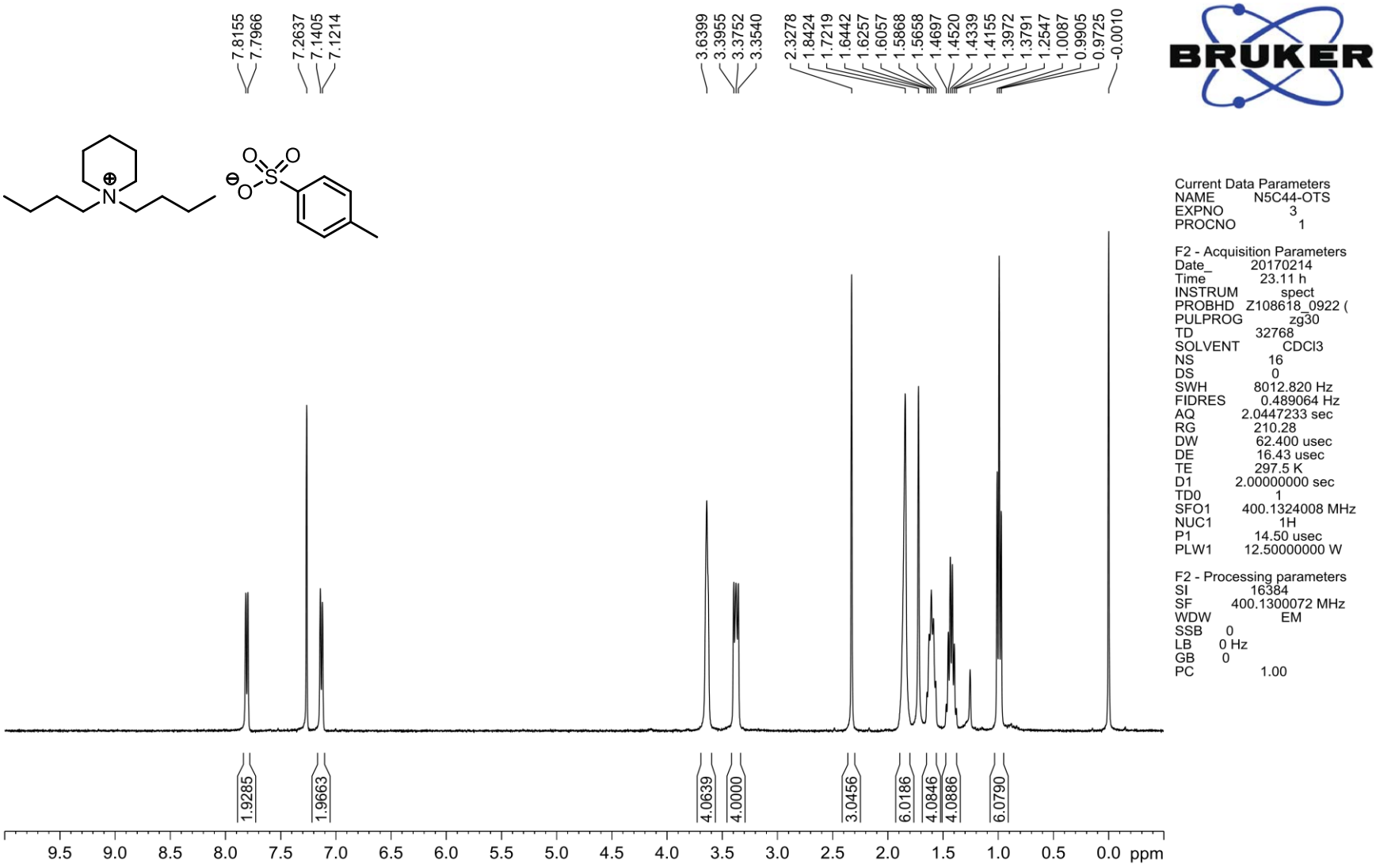
KE267

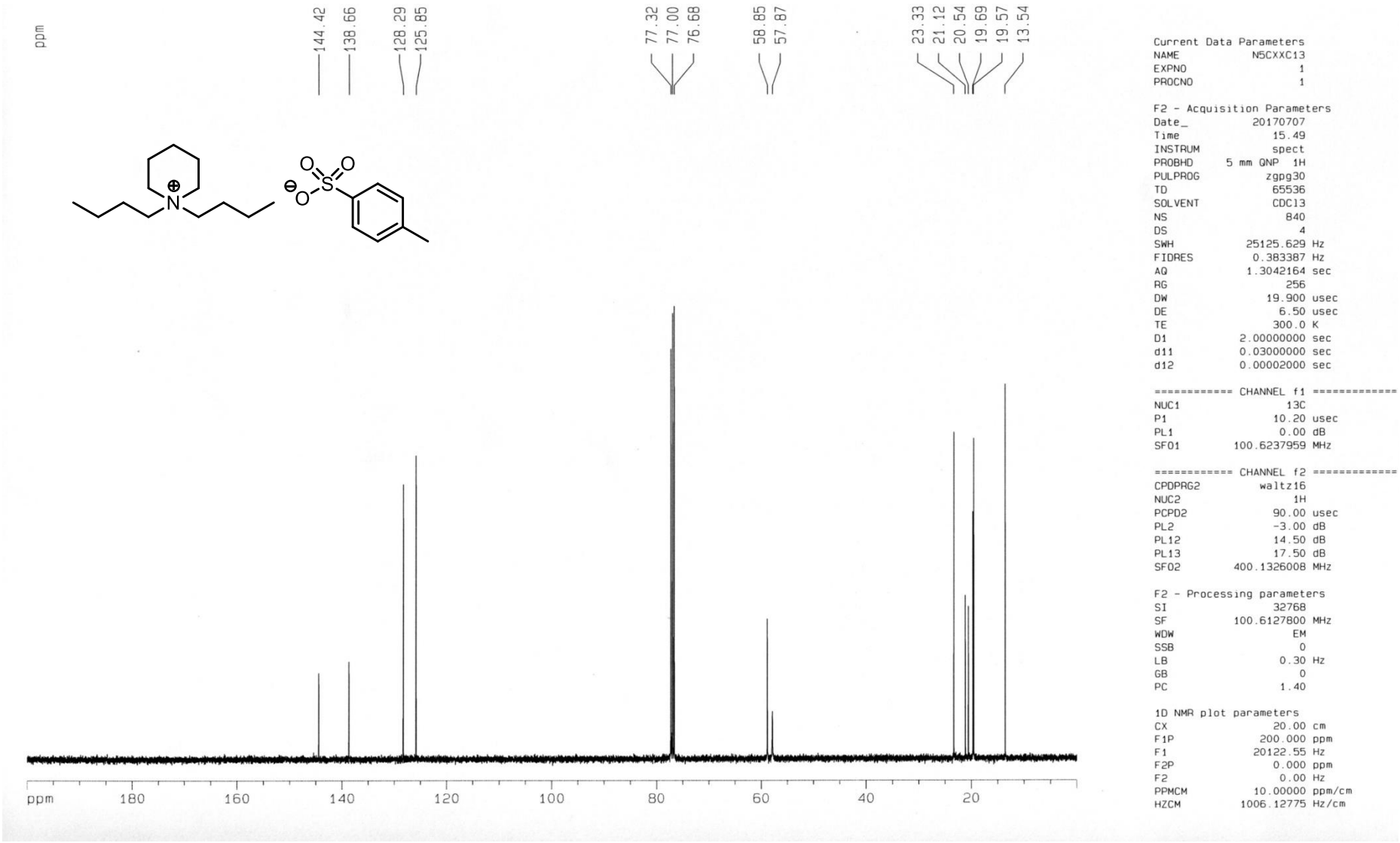
11-Aug-2017
11:59:50
2: TOF MS AP+
1.26e+004

0811_3 122 (3.547) Cm (122-1x5.000)



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
240.2696	240.2691	0.5	2.1	0.5	43.0	0.0	C16 H34 N


7.8155
7.7966
7.2637
7.1405
7.1214
3.6399
3.3955
3.3752
3.3540
2.3278
1.8424
1.7219
1.6442
1.6257
1.6057
1.5868
1.5658
1.4697
1.4520
1.4339
1.4155
1.3972
1.3791
1.2547
1.0087
0.9905
0.9725
-0.0010
BRUKER
Current Data Parameters
NAME N5C44-OTS
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170214
Time 23.11 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 297.5 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300072 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9285
1.9663
4.0639
4.0000
3.0456
6.0186
4.0846
4.0886
6.0790
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                 N5CXXC13
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170707
Time                   15.49
INSTRUM                spect
PROBHD     5 mm QNP  1H
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       840
DS                         4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                       256
DW                    19.900 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                     13C
P1                    10.20 usec
PL1                    0.00 dB
SFO1              100.6237959 MHz

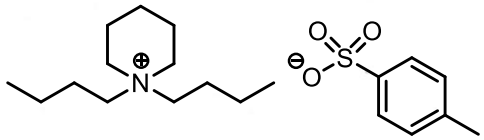
============ CHANNEL f2 =============
CPDPRG2               waltz16
NUC2                     1H
PCPD2                 90.00 usec
PL2                   -3.00 dB
PL12                  14.50 dB
PL13                  17.50 dB
SFO2              400.1326008 MHz

F2 - Processing parameters
SI                       32768
SF                100.6127800 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                    1.40

1D NMR plot parameters
CX                    20.00 cm
F1P                  200.000 ppm
F1                 20122.55 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM               1006.12775 Hz/cm
```

1 (HR-ES2)

Elemental Composition Report



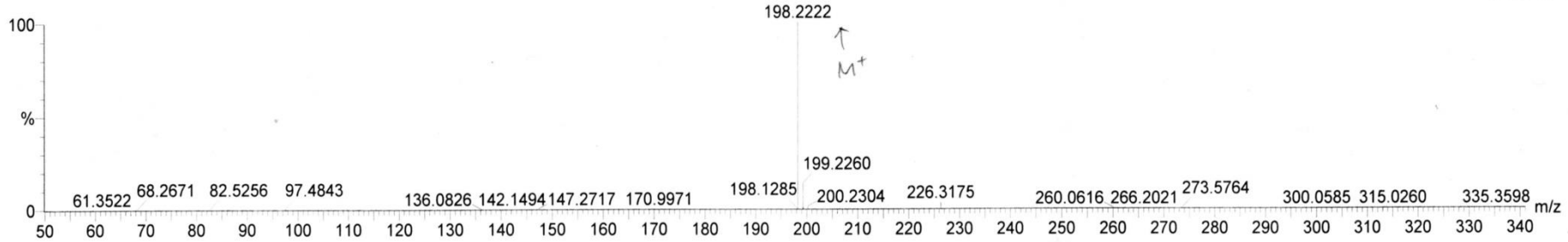
Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

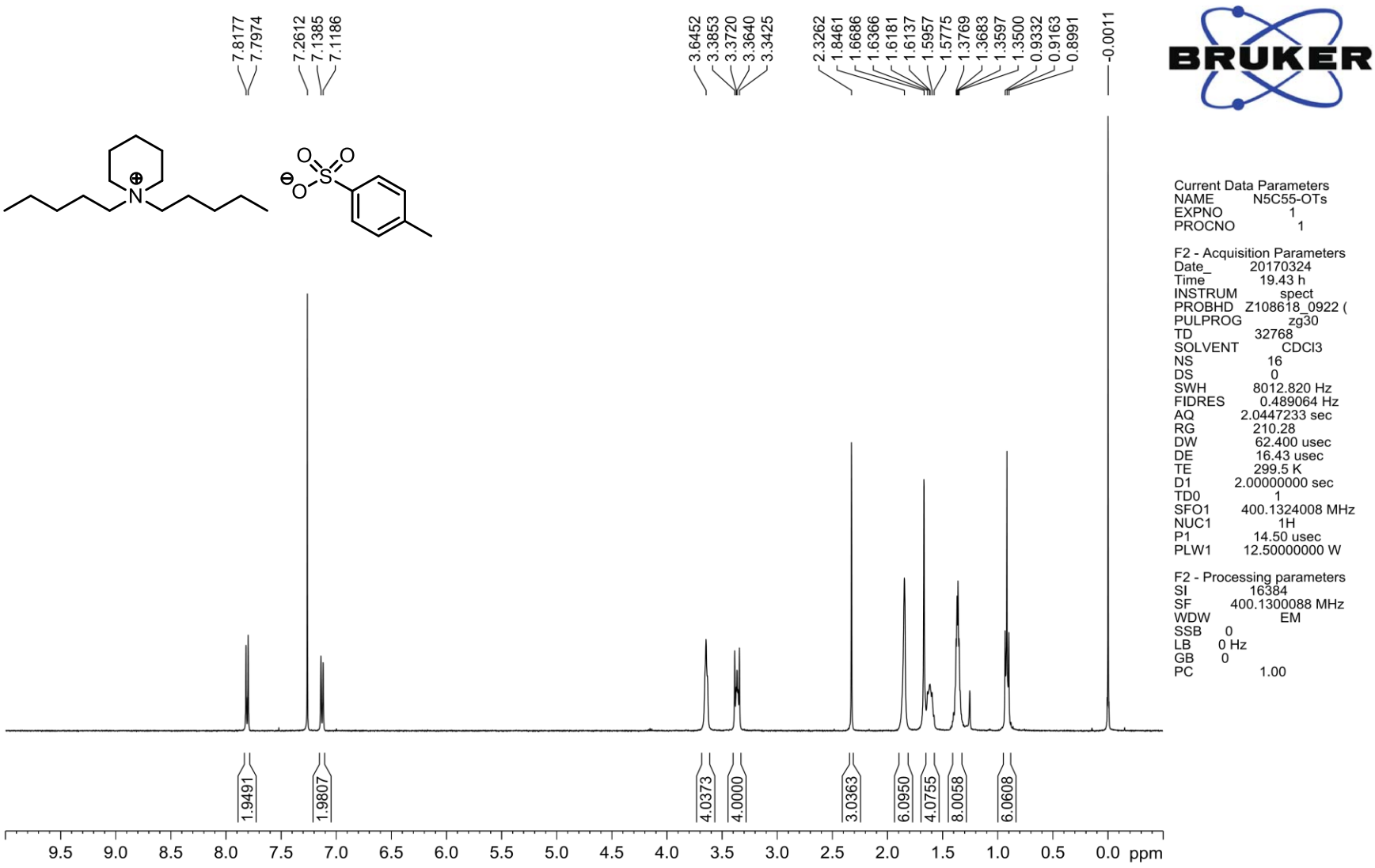
Monoisotopic Mass, Even Electron Ions
16 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

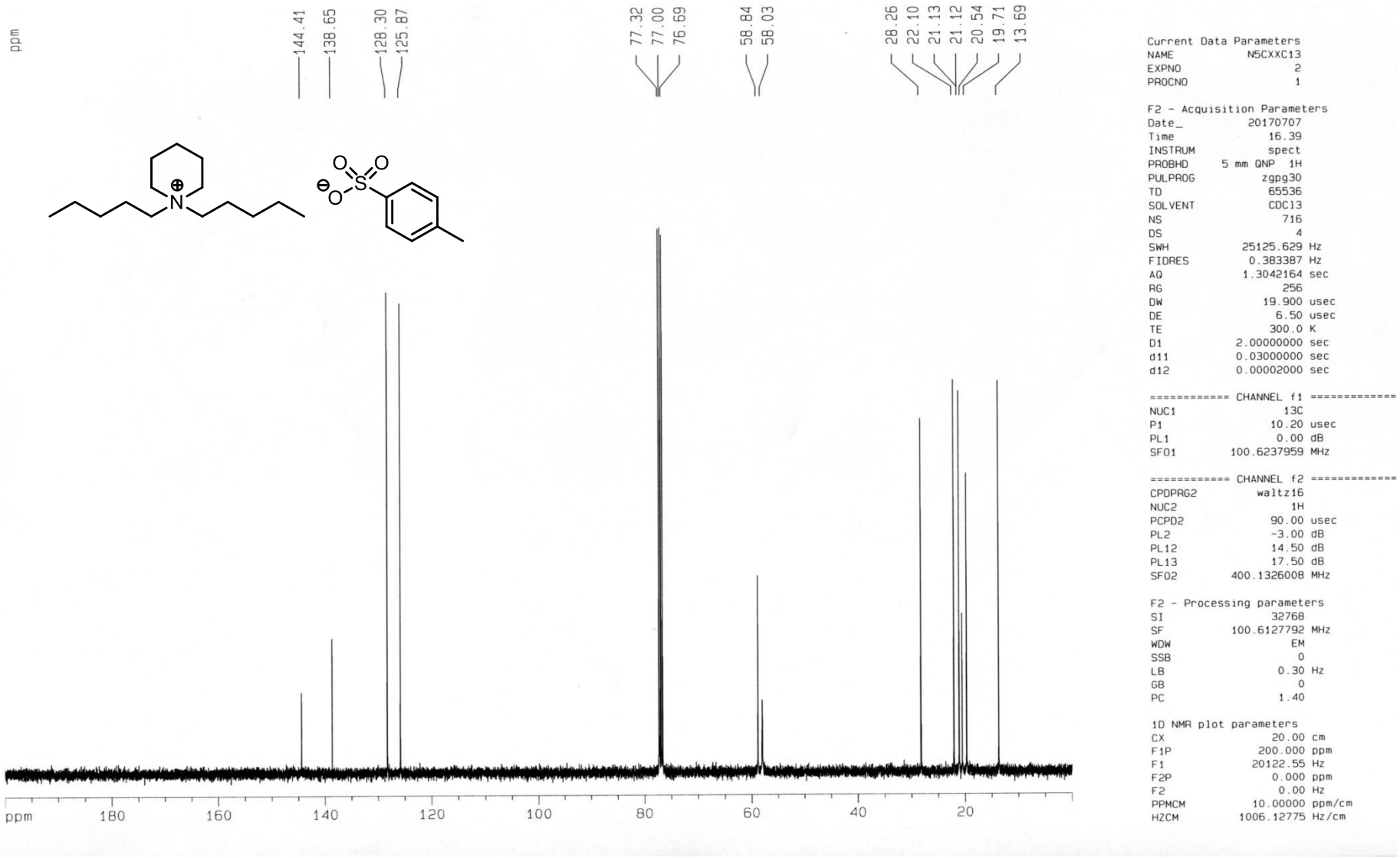
1 KE267 14-Jul-2017 13:33:01

0714_1 80 (2.890) Cm (80-1x100.000) 1: TOF MS ES+ 3.46e+004



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
198.2222	198.2222	0.0	0.0	0.5	50.9	0.0	C13 H28 N


7.8177
7.7974
7.2612
7.1385
7.1186
3.6452
3.3853
3.3720
3.3640
3.3425
2.3262
1.8461
1.6686
1.6366
1.6181
1.6137
1.5957
1.5775
1.3769
1.3683
1.3597
1.3500
0.9332
0.9163
0.8991
-0.0011
BRUKER
Current Data Parameters
NAME N5C55-OTs
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170324
Time 19.43 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.5 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9491
1.9807
4.0373
4.0000
3.0363
6.0950
4.0755
8.0058
6.0608
9.5 9.0 8.5 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 ppm



Current Data Parameters
NAME N5CXXC13
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170707
Time 16.39
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 716
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 256
DW 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 10.20 usec
PL1 0.00 dB
SF01 100.6237959 MHz

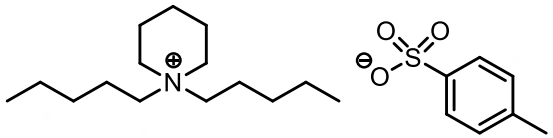
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.00 dB
PL12 14.50 dB
PL13 17.50 dB
SF02 400.1326008 MHz

F2 - Processing parameters
SI 32768
SF 100.6127792 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 20122.55 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1006.12775 Hz/cm

2 (HR-ESI)

Elemental Composition Report



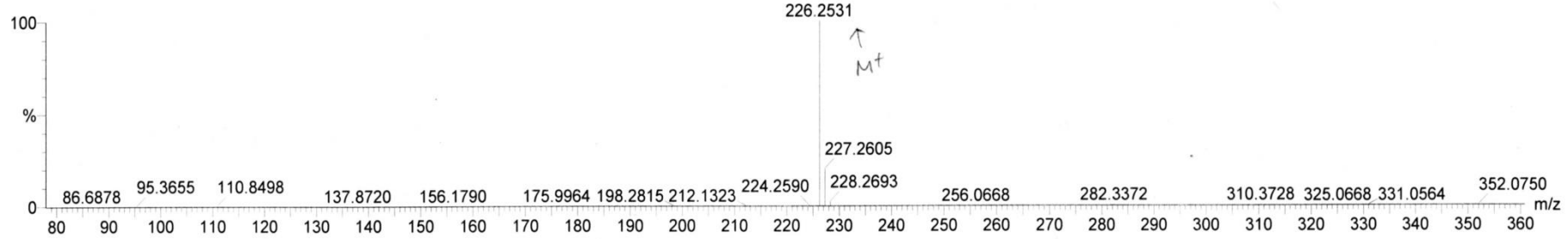
Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

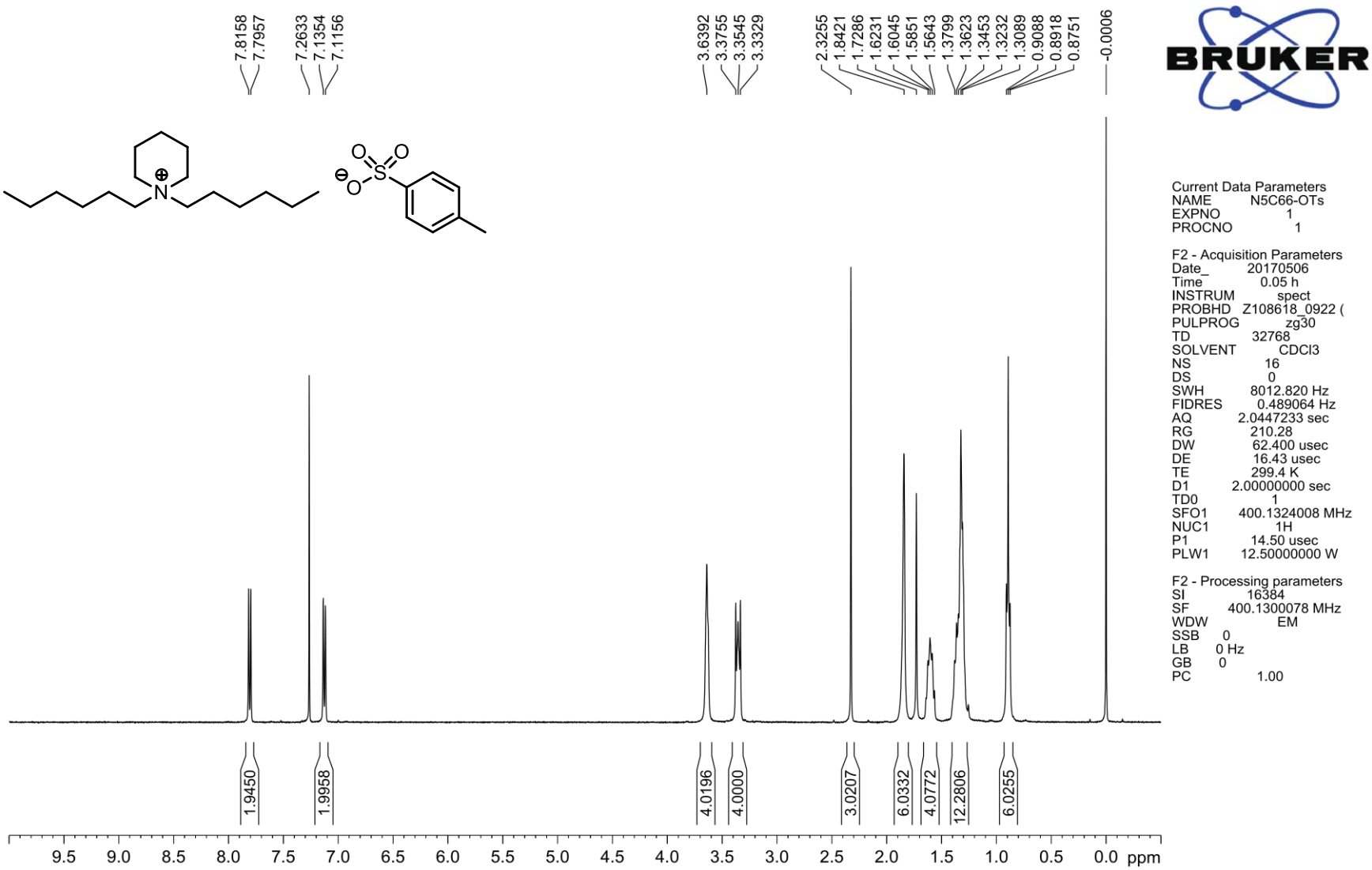
Monoisotopic Mass, Even Electron Ions
18 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

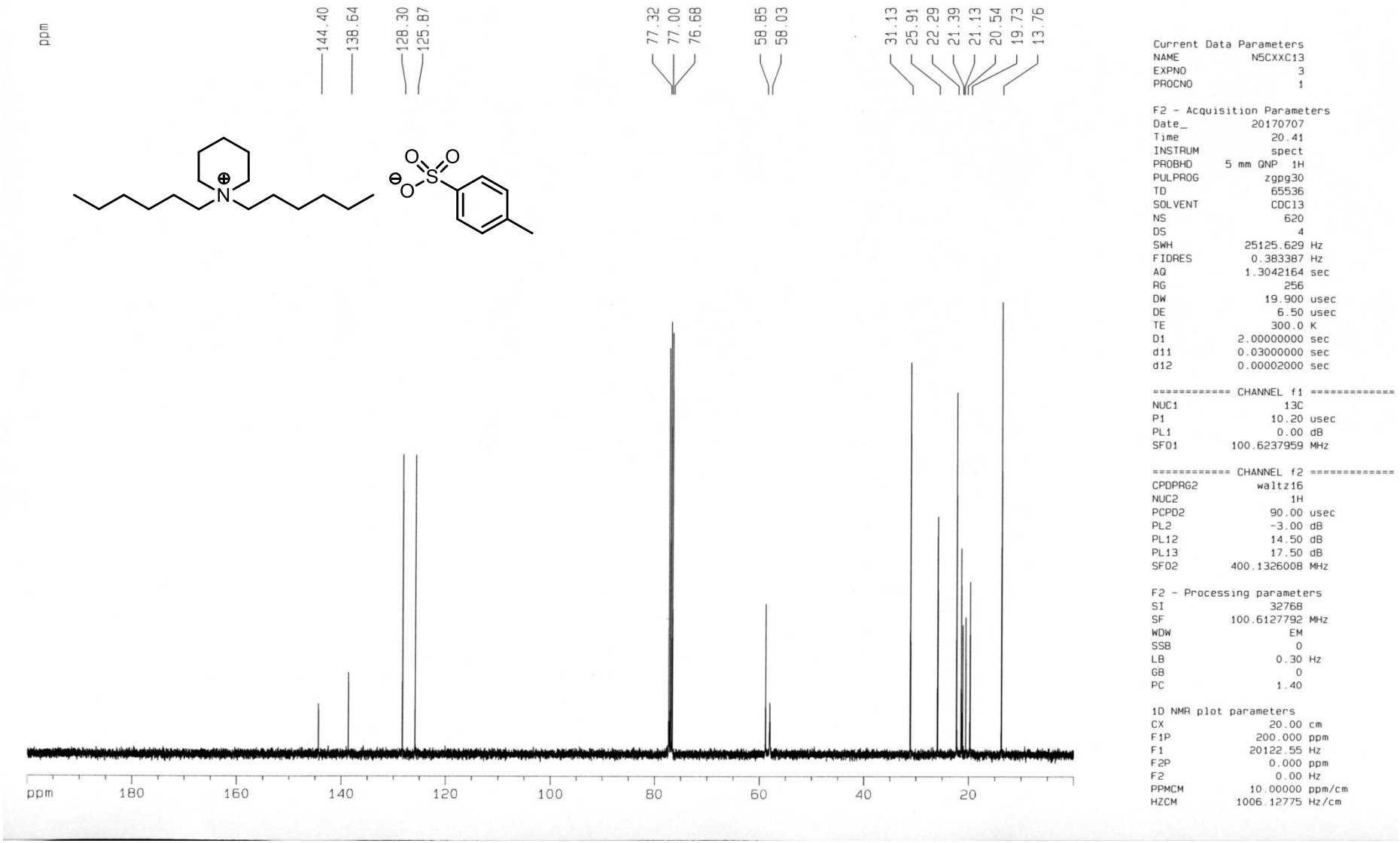
2 KE267 14-Jul-2017 13:24:52

0714_2 32 (1.147) Cm (32-1x100.000) 1: TOF MS ES+ 1.29e+005



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
226.2531	226.2535	-0.4	-1.8	0.5	89.5	0.0	C15 H32 N


7.8158
7.7957
7.2633
7.1354
7.1156
3.6392
3.3755
3.3545
3.3329
2.3255
1.8421
1.7286
1.6231
1.6045
1.5851
1.5643
1.3799
1.3623
1.3453
1.3232
1.3089
0.9088
0.8918
0.8751
-0.0006
1.9450
1.9958
4.0196
4.0000
3.0207
6.0332
4.0772
12.2806
6.0255
9.5 9.0 8.5 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 ppm
Current Data Parameters
NAME N5C66-OTs
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170506
Time 0.05 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.4 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME              N5CXXC13
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_              20170707
Time                 20.41
INSTRUM              spect
PROBHD     5 mm QNP  1H
PULPROG              zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     620
DS                       4
SWH              25125.629 Hz
FIDRES               0.383387 Hz
AQ                1.3042164 sec
RG                     256
DW                  19.900 usec
DE                   6.50 usec
TE                  300.0 K
D1               2.00000000 sec
d11              0.03000000 sec
d12              0.00002000 sec

============ CHANNEL f1 =============
NUC1                   13C
P1                  10.20 usec
PL1                  0.00 dB
SF01           100.6237959 MHz

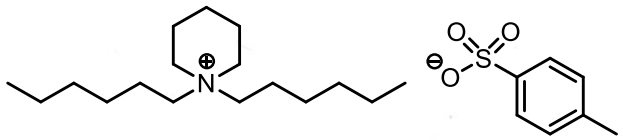
============ CHANNEL f2 =============
CPDPRG2            waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -3.00 dB
PL12                14.50 dB
PL13                17.50 dB
SF02           400.1326008 MHz

F2 - Processing parameters
SI                     32768
SF             100.6127792 MHz
WDW                    EM
SSB                    0
LB                   0.30 Hz
GB                     0
PC                   1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                 200.000 ppm
F1                20122.55 Hz
F2P                   0.000 ppm
F2                    0.00 Hz
PPMCM             10.00000 ppm/cm
HZCM           1006.12775 Hz/cm
```

3 (HR-ESI)

Elemental Composition Report

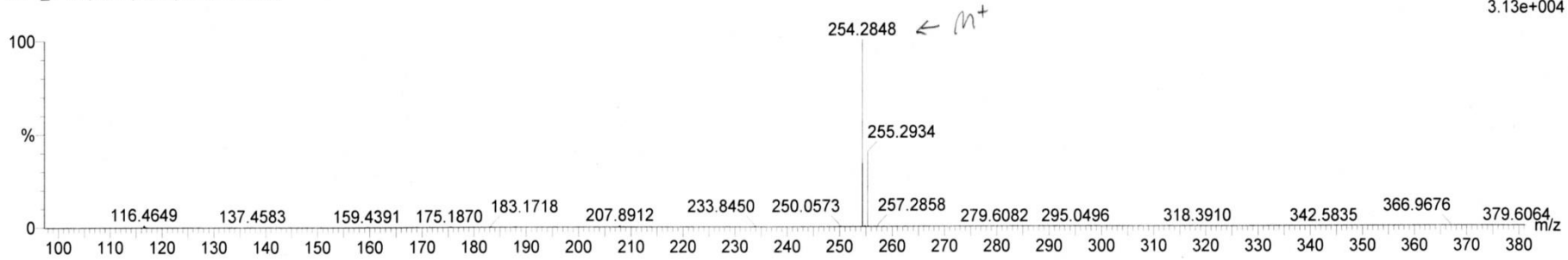


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

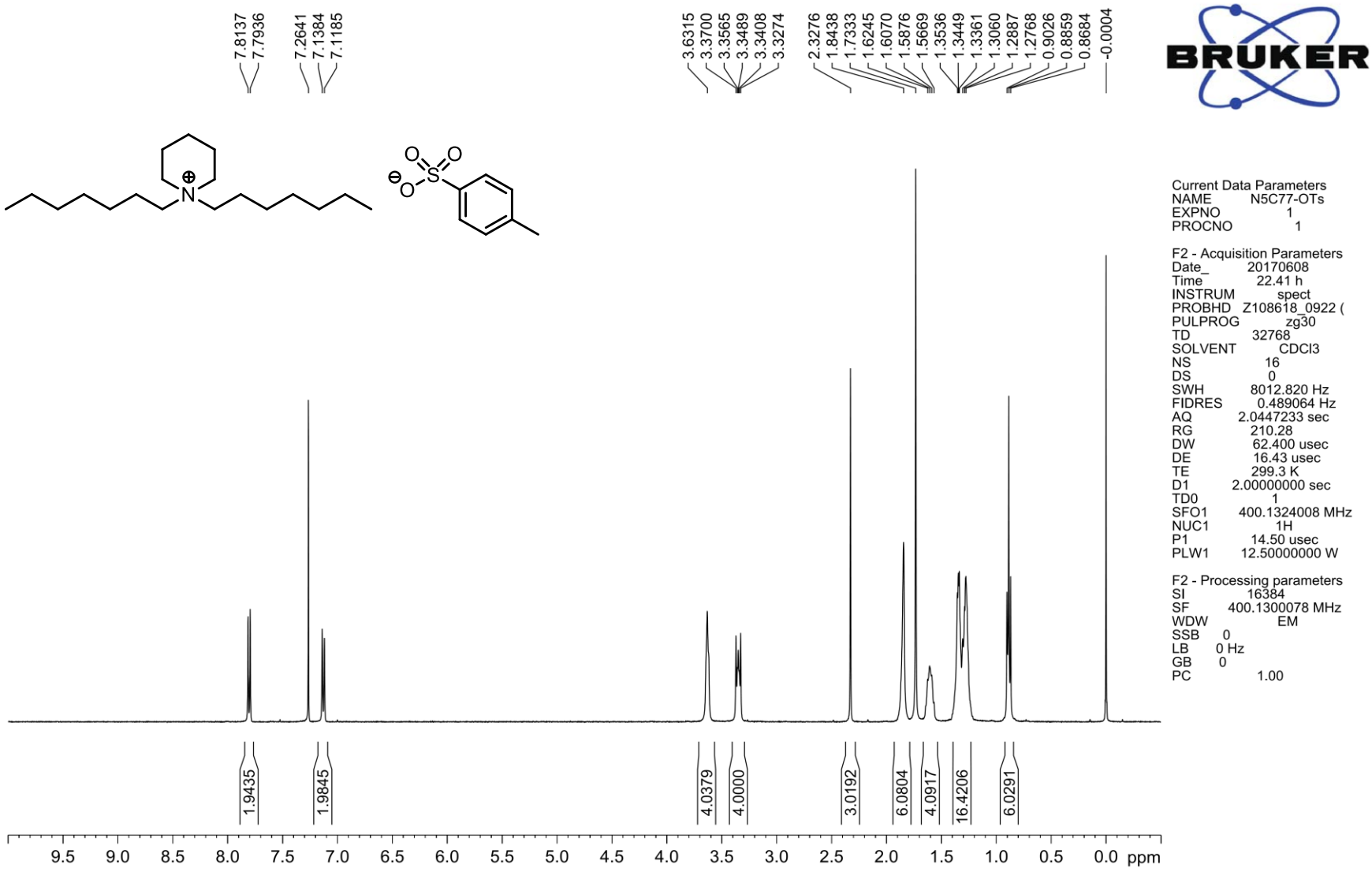
Monoisotopic Mass, Even Electron Ions
21 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

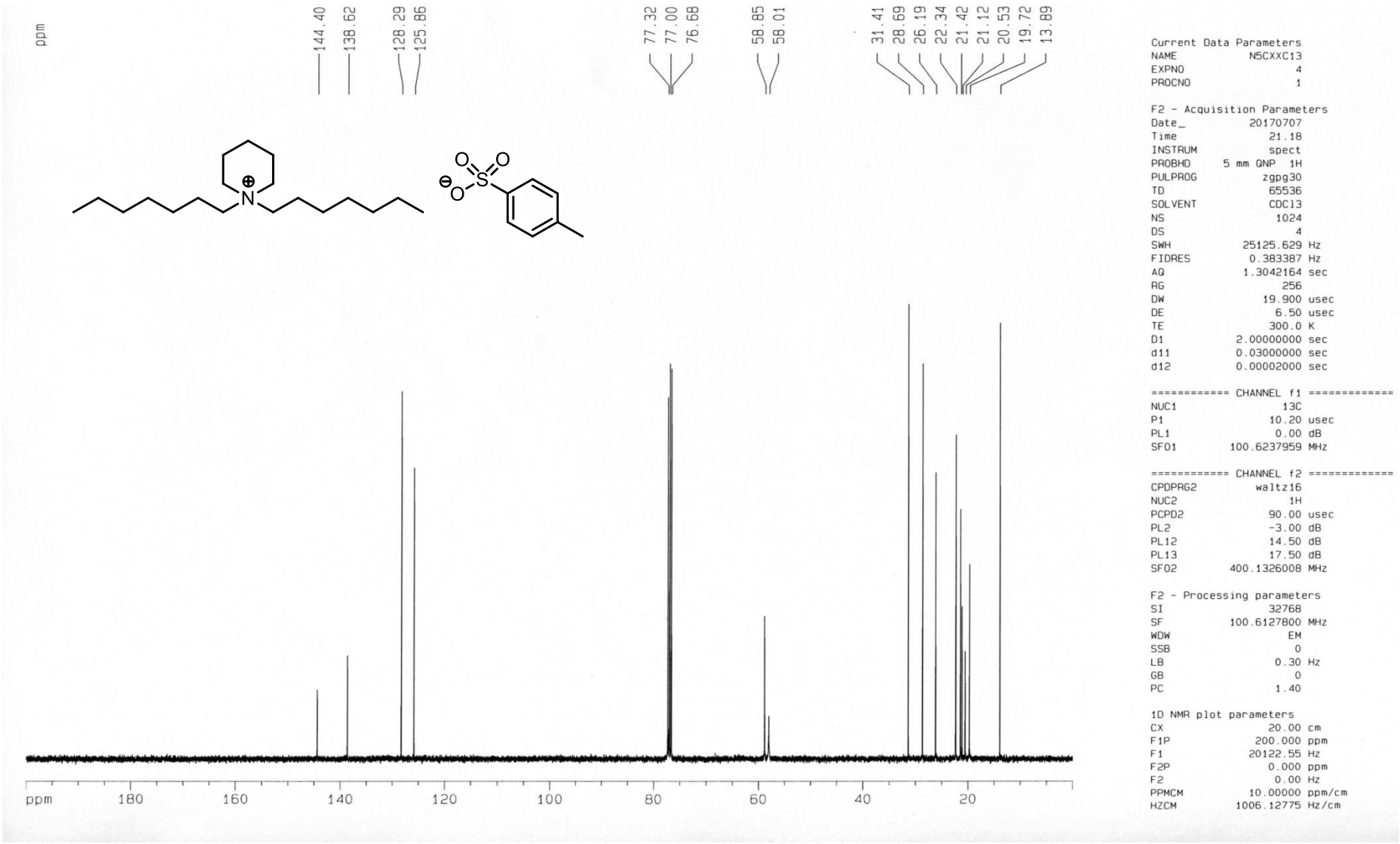
3 KE267 14-Jul-2017 13:16:42

0714_3 11 (0.391) Cm (11-1x200.000) 1: TOF MS ES+ 3.13e+004



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
254.2848	254.2848	0.0	0.0	0.5	97.3	0.0	C17 H36 N


7.8137
7.7936
7.2641
7.1384
7.1185
3.6315
3.3700
3.3565
3.3489
3.3408
3.3274
2.3276
1.8438
1.7333
1.6245
1.6070
1.5876
1.5669
1.3536
1.3449
1.3361
1.3060
1.2887
1.2768
0.9026
0.8859
0.8684
-0.0004
BRUKER
Current Data Parameters
NAME N5C77-OTs
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170608
Time 22.41 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.3 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9435
1.9845
4.0379
4.0000
3.0192
6.0804
4.0917
16.4206
6.0291
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                 N5CXXC13
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170707
Time                   21.18
INSTRUM                spect
PROBHD      5 mm QNP  1H
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      1024
DS                         4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                       256
DW                    19.900 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                     13C
P1                    10.20 usec
PL1                    0.00 dB
SFO1              100.6237959 MHz

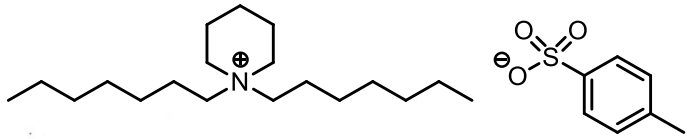
============ CHANNEL f2 =============
CPDPRG2               waltz16
NUC2                     1H
PCPD2                 90.00 usec
PL2                   -3.00 dB
PL12                  14.50 dB
PL13                  17.50 dB
SFO2              400.1326008 MHz

F2 - Processing parameters
SI                       32768
SF                100.6127800 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                    1.40

1D NMR plot parameters
CX                    20.00 cm
F1P                  200.000 ppm
F1                 20122.55 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM               1006.12775 Hz/cm
```

4 (HR-ESI)

Elemental Composition Report

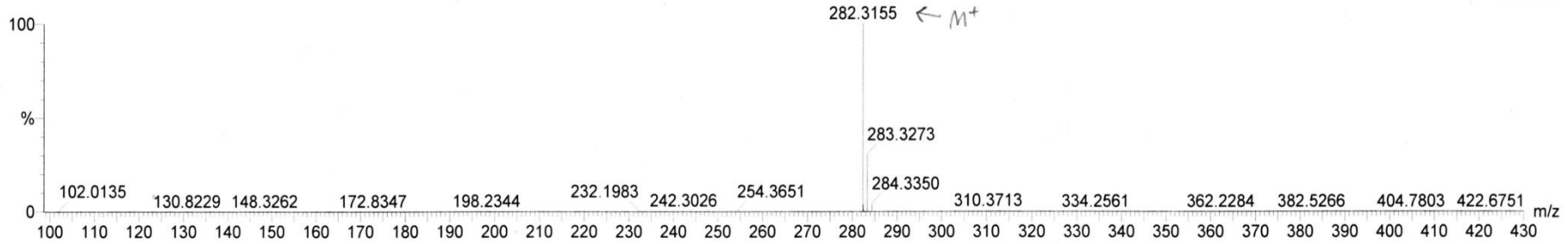


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

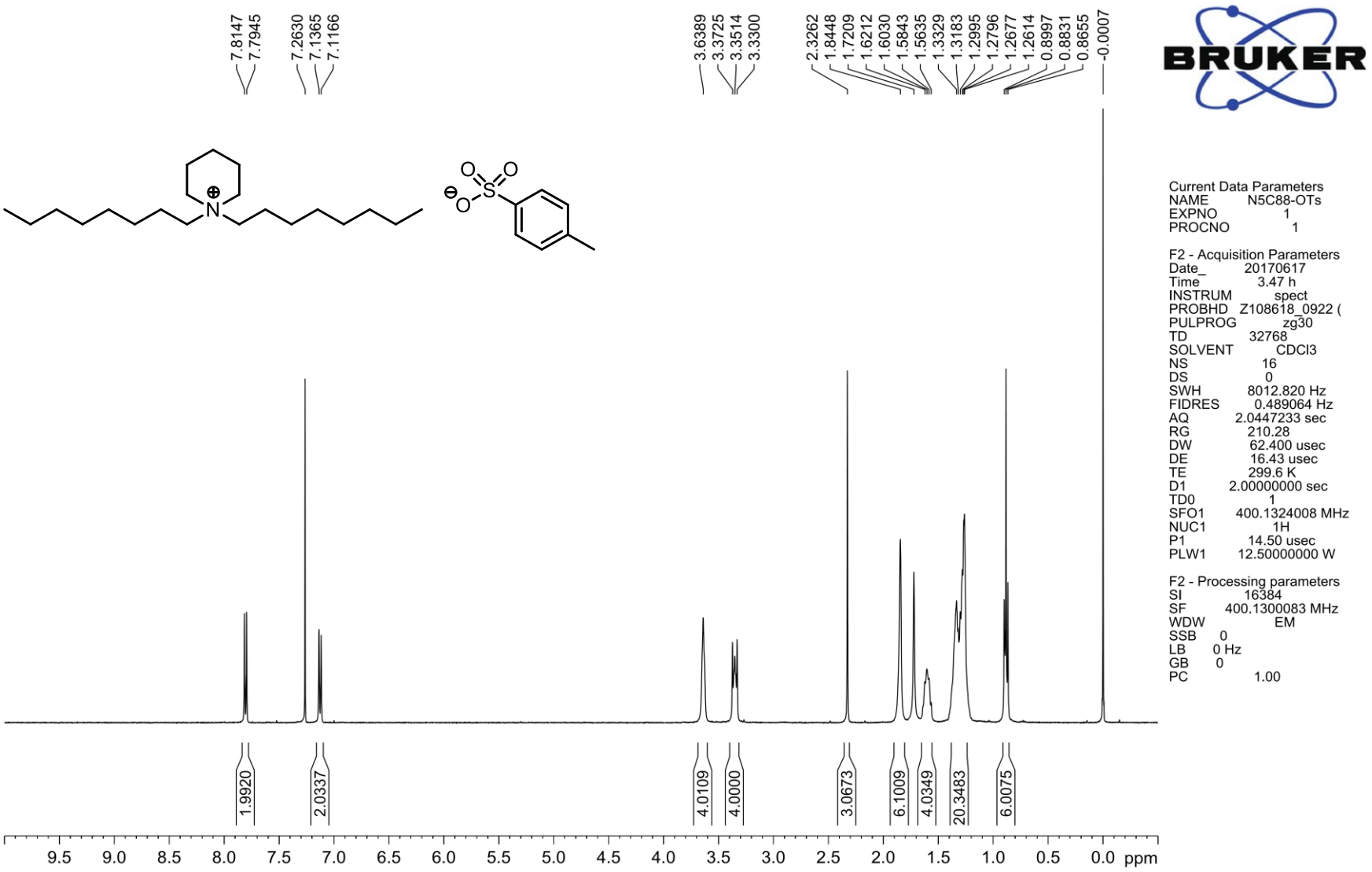
Monoisotopic Mass, Even Electron Ions
23 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

4 KE267 14-Jul-2017 13:08:34

0714_4 20 (0.711) Cm (20-1x200.000) 1: TOF MS ES+ 7.53e+004



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
282.3155	282.3161	-0.6	-2.1	0.5	81.0	0.0	C19 H40 N



Current Data Parameters
NAME N5C88-OTs
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170617
Time 3.47 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.6 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00


```
Current Data Parameters
NAME            N5CXXC13
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170707
Time               22.30
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  537
DS                    4
SWH           25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1         100.6237959 MHz

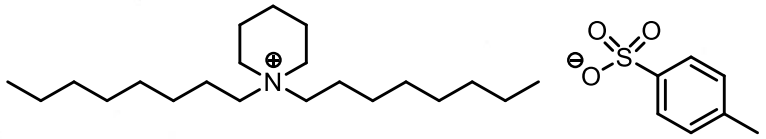
============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127784 MHz
WDW                   EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              200.000 ppm
F1             20122.55 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1006.12775 Hz/cm
```

5 (HR-ESI)

Elemental Composition Report



Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
25 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

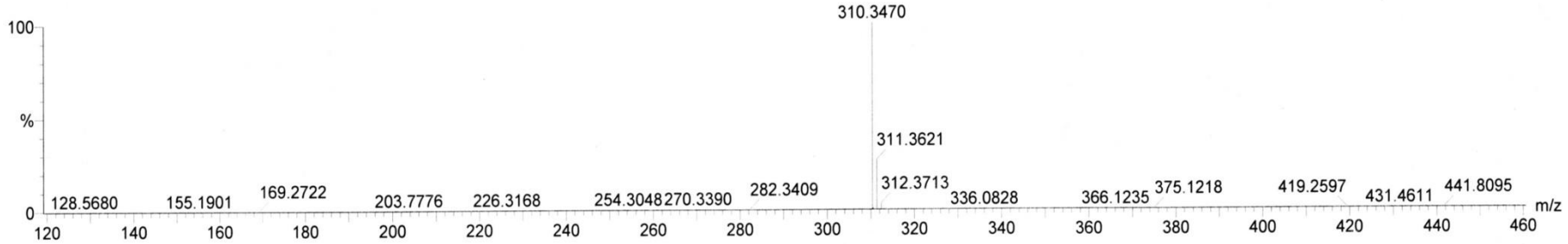
5

KE267

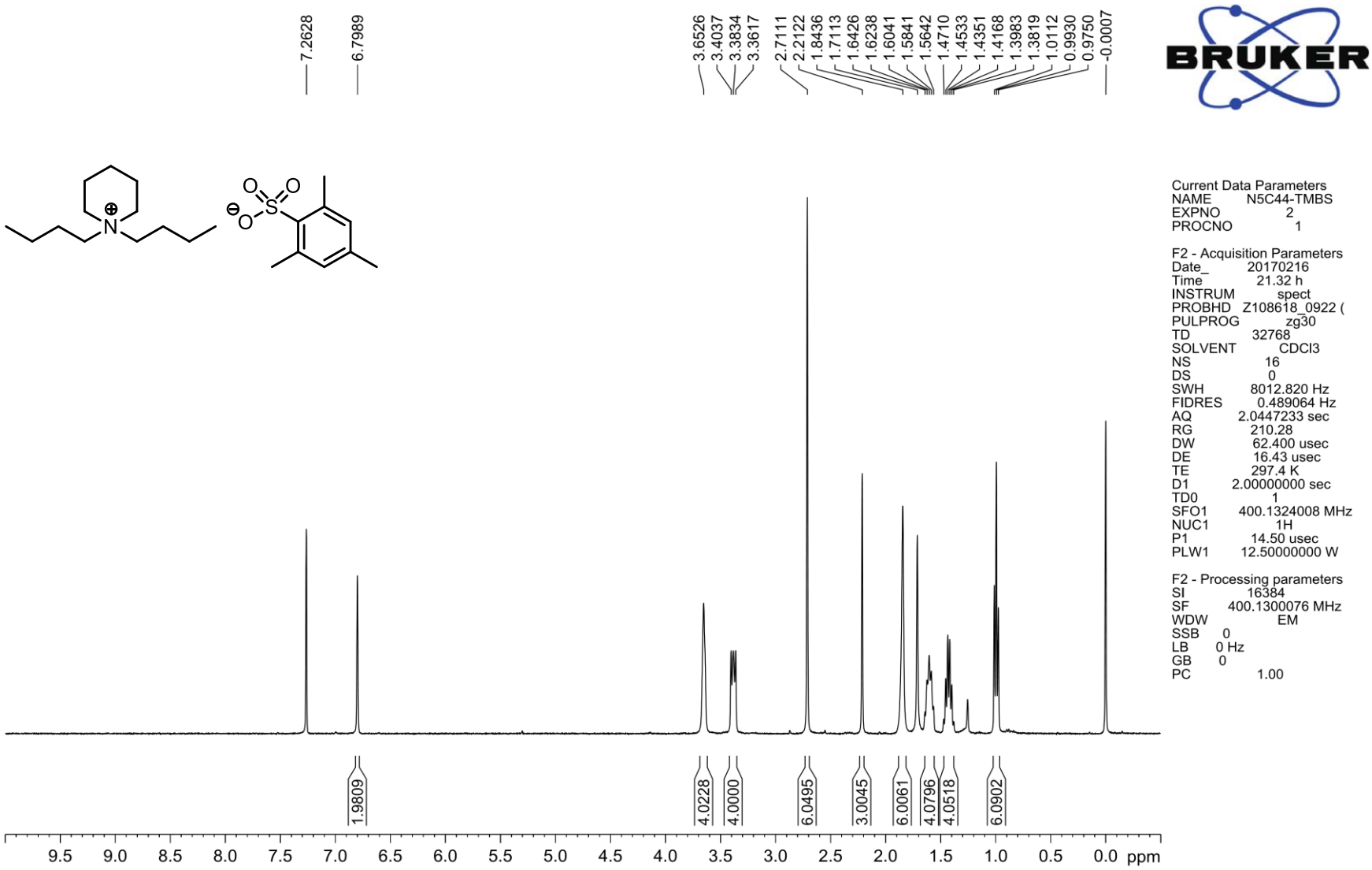
M^+

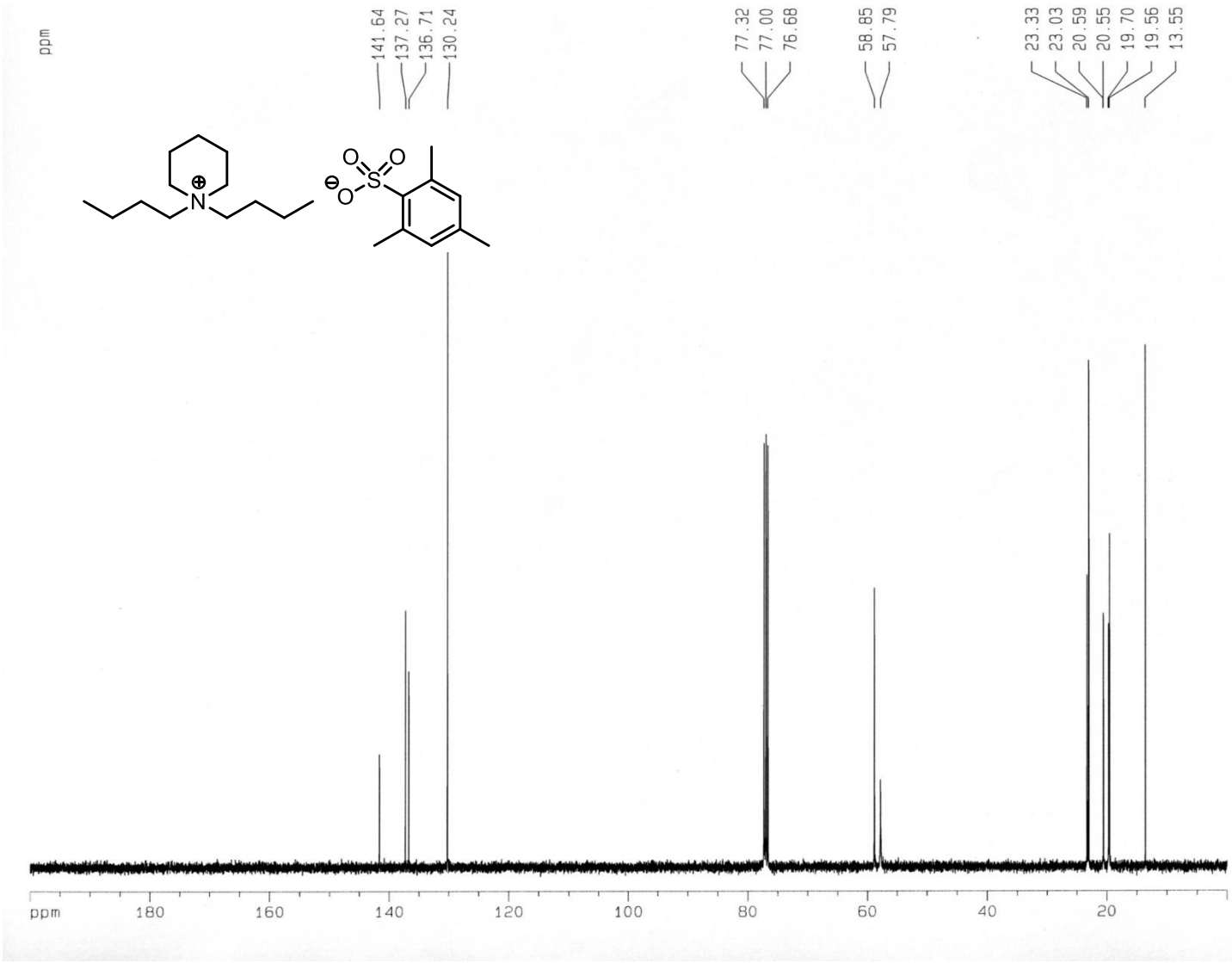
14-Jul-2017
13:00:31
1: TOF MS ES+
3.16e+005

0714_5 26 (0.946) Cm (26-1x200.000)



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
310.3470	310.3474	-0.4	-1.3	0.5	119.3	0.0	C21 H44 N


7.2628
6.7989
3.6526
3.4037
3.3834
3.3617
2.7111
2.2122
1.8436
1.7113
1.6426
1.6238
1.6041
1.5841
1.5642
1.4710
1.4533
1.4351
1.4168
1.3983
1.3819
1.0112
0.9930
0.9750
-0.0007
BRUKER
Current Data Parameters
NAME N5C44-TMBS
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170216
Time 21.32 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 297.4 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300076 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9809
4.0228
4.0000
6.0495
3.0045
6.0061
4.0796
4.0518
6.0902
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                 N5CXXC13
EXPNO                       6
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170709
Time                   10.55
INSTRUM                spect
PROBHD      5 mm QNP  1H
PULPROG              zgpg30
TD                      65536
SOLVENT                CDCl3
NS                        975
DS                          4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                        256
DW                   19.900 usec
DE                    6.50 usec
TE                   300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                      13C
P1                     10.20 usec
PL1                     0.00 dB
SFO1               100.6237959 MHz

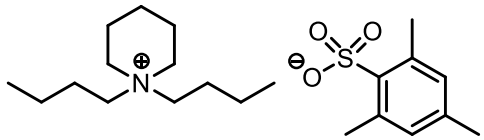
============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  90.00 usec
PL2                    -3.00 dB
PL12                   14.50 dB
PL13                   17.50 dB
SFO2               400.1326008 MHz

F2 - Processing parameters
SI                        32768
SF                 100.6127800 MHz
WDW                          EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.40

1D NMR plot parameters
CX                     20.00 cm
F1P                   200.000 ppm
F1                  20122.55 Hz
F2P                     0.000 ppm
F2                      0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM                1006.12775 Hz/cm
```

1A (HR-ESI)

Elemental Composition Report



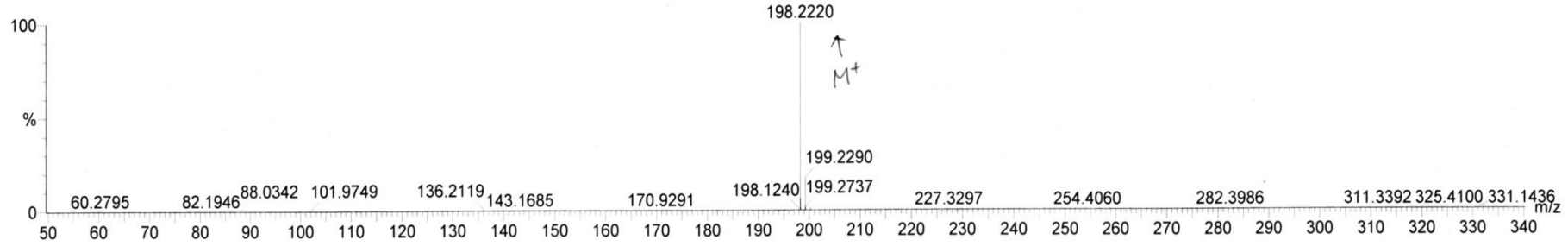
Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

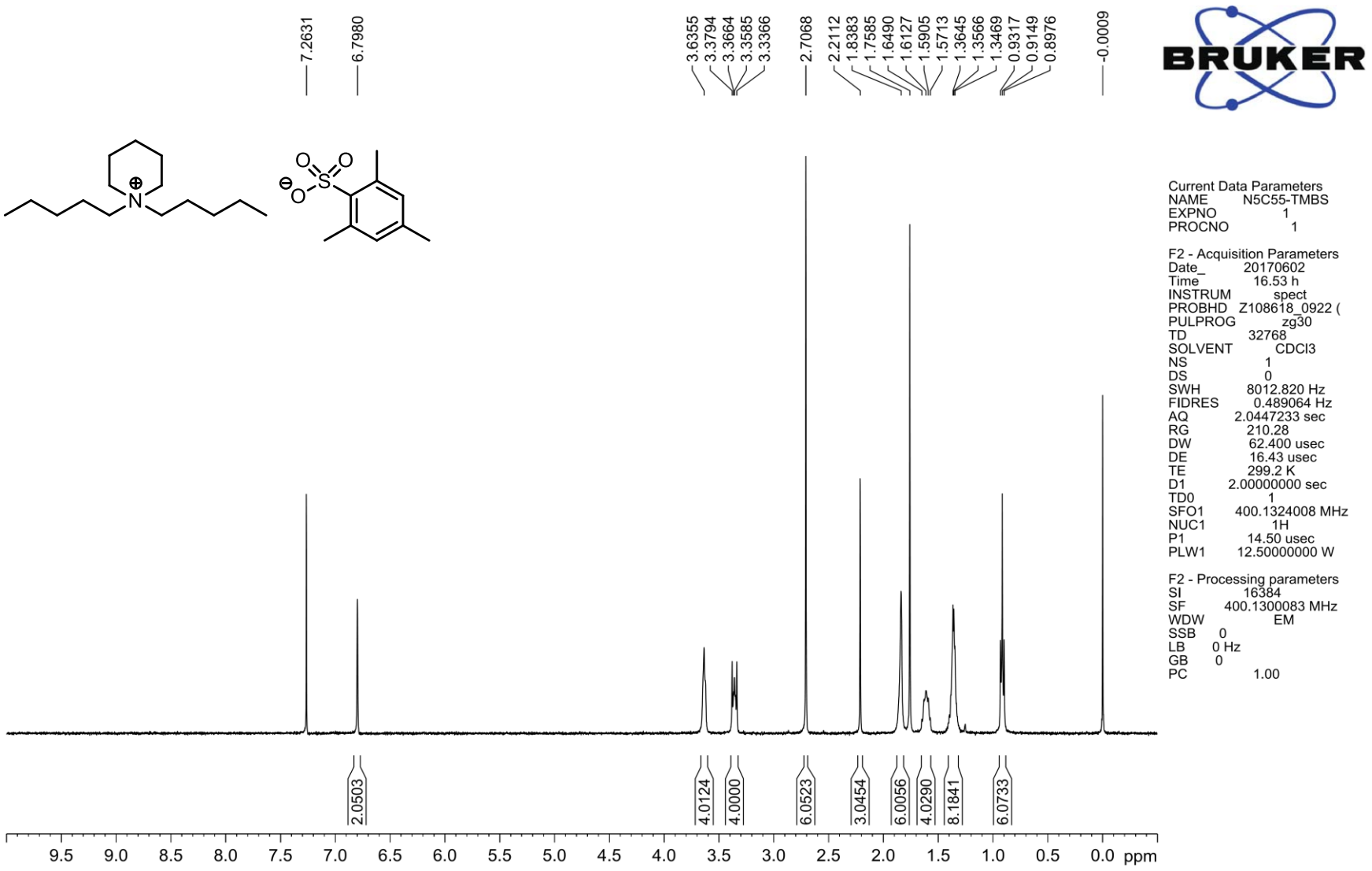
Monoisotopic Mass, Even Electron Ions
16 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

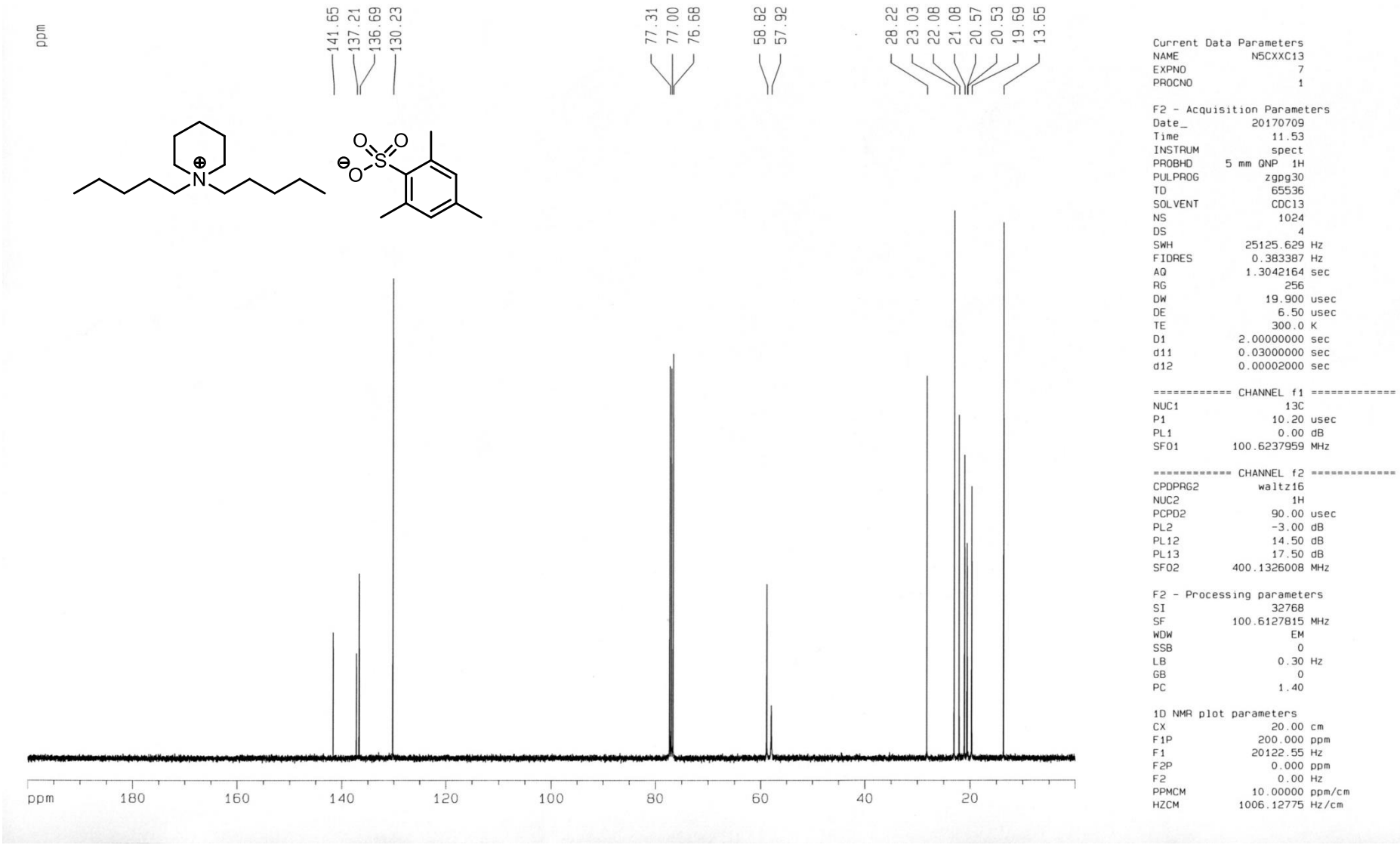
1A KE267 14-Jul-2017 12:52:28

0714_1A 35 (1.263) Cm (35-1x200.000) 1: TOF MS ES+ 4.34e+004



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
198.2220	198.2222	-0.2	-1.0	0.5	82.5	0.0	C13 H28 N


7.2631
6.7980
3.6355
3.3794
3.3664
3.3585
3.3366
2.7068
2.2112
1.8383
1.7585
1.6490
1.6127
1.5905
1.5713
1.3645
1.3566
1.3469
0.9317
0.9149
0.8976
-0.0009
BRUKER
Current Data Parameters
NAME N5C55-TMBS
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170602
Time 16.53 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 1
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.2 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
2.0503
4.0124
4.0000
6.0523
3.0454
6.0056
4.0290
8.1841
6.0733
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME            N5CXXC13
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170709
Time               11.53
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1         100.6237959 MHz

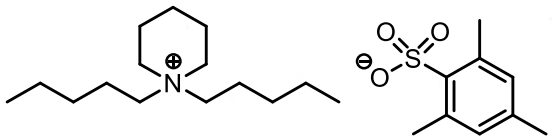
============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127815 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               200.000 ppm
F1              20122.55 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1006.12775 Hz/cm
```

2A (HR-ESI)

Elemental Composition Report



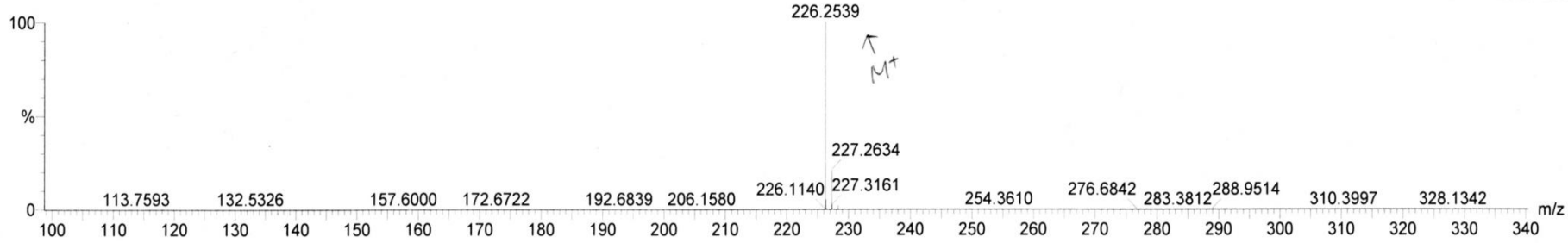
Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

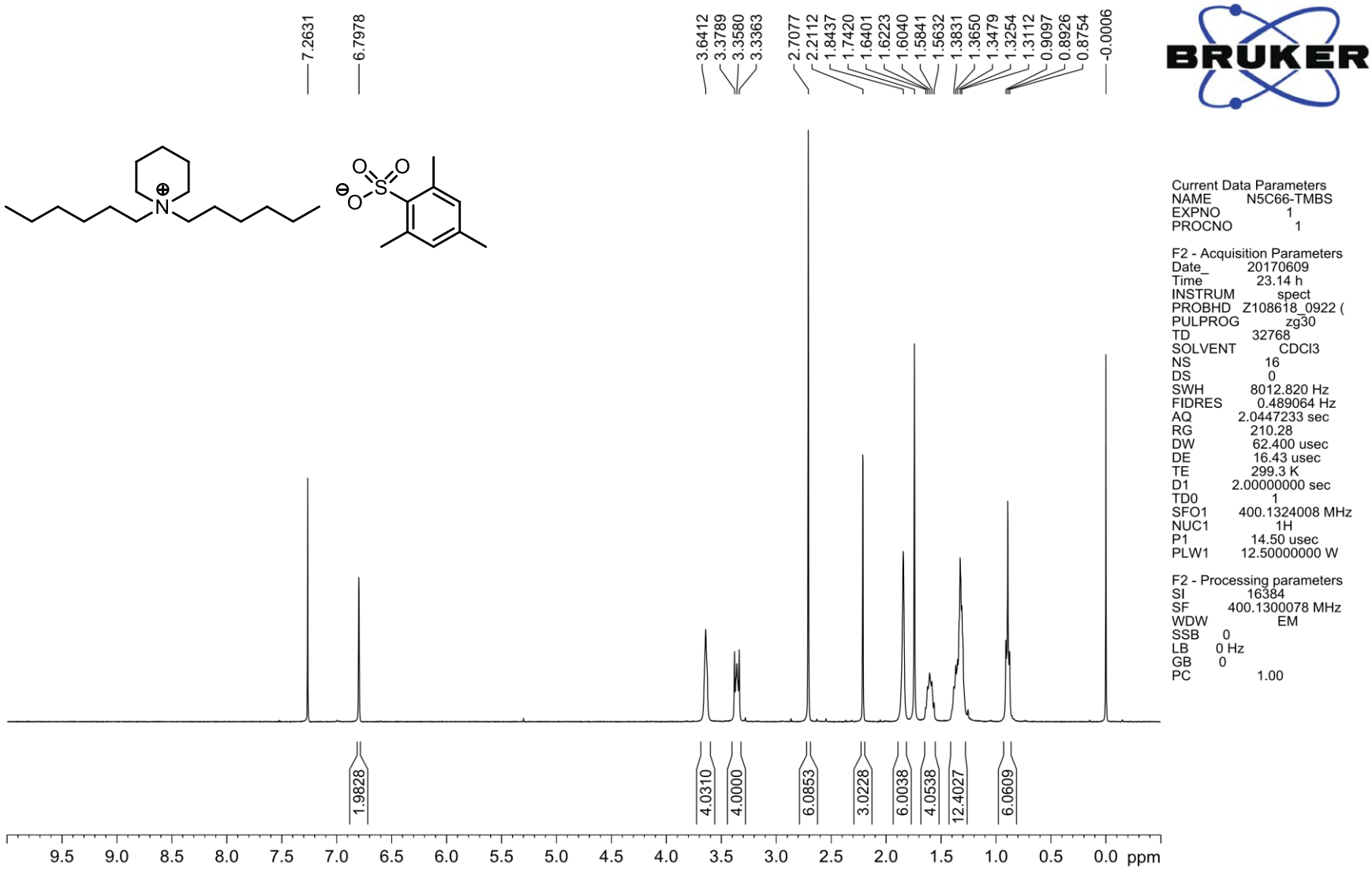
Monoisotopic Mass, Even Electron Ions
18 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

2A KE267 14-Jul-2017 12:44:25

0714_2A 28 (1.001) Cm (28-1x200.000) 1: TOF MS ES+ 1.50e+005



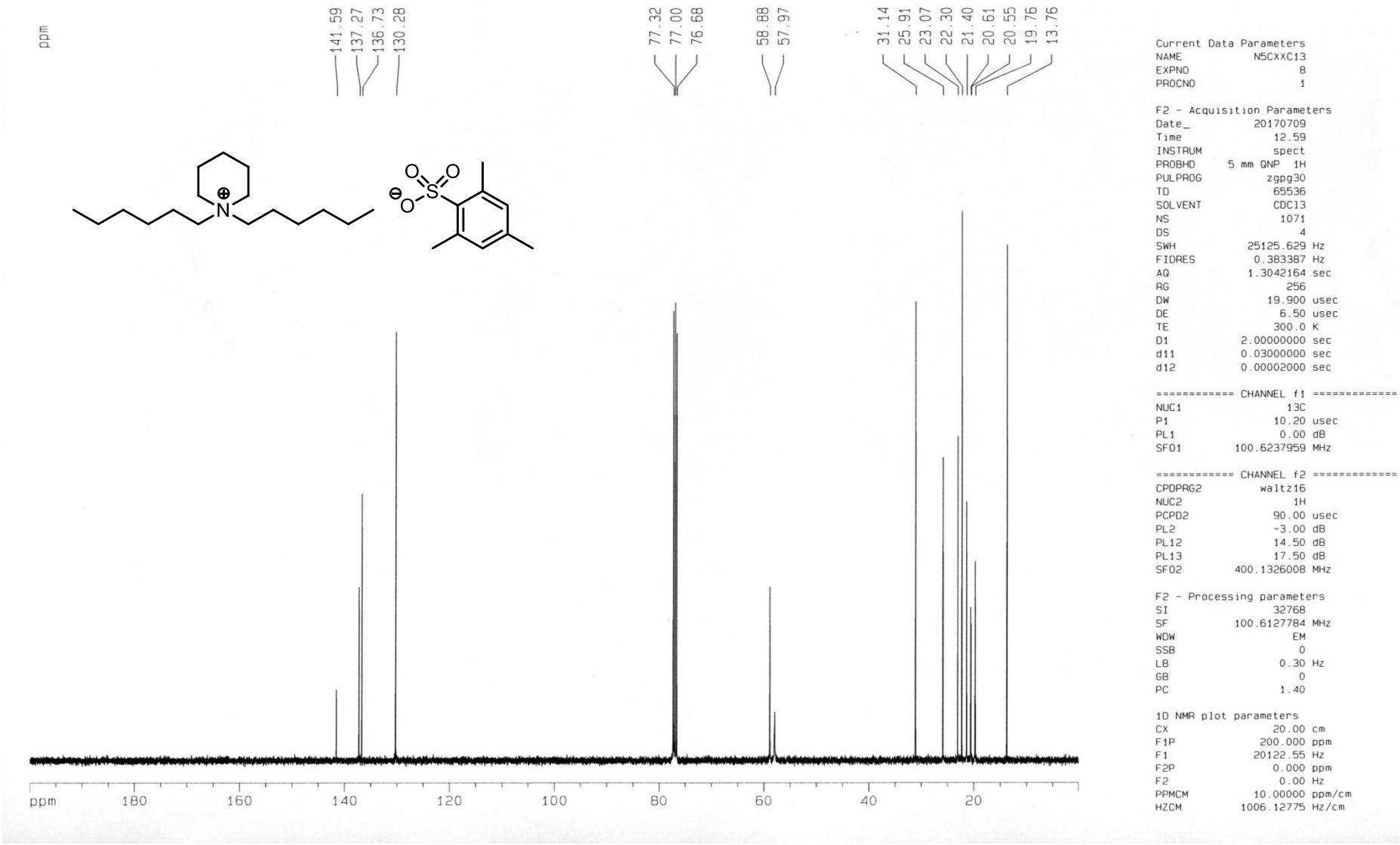
Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
226.2539	226.2535	0.4	1.8	0.5	95.0	0.0	C15 H32 N



Current Data Parameters
NAME N5C66-TMBS
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170609
Time 23.14 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.3 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME                 N5CXXC13
EXPNO                       8
PROCNO                      1

F2 - Acquisition Parameters
Date_                20170709
Time                    12.59
INSTRUM                 spect
PROBHD       5 mm QNP  1H
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1071
DS                          4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                        256
DW                   19.900 usec
DE                    6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                      13C
P1                      10.20 usec
PL1                      0.00 dB
SFO1               100.6237959 MHz

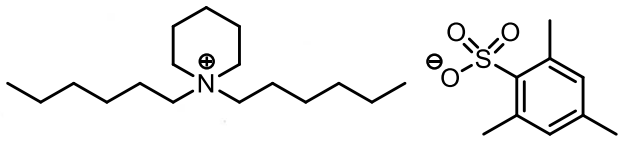
============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                     -3.00 dB
PL12                    14.50 dB
PL13                    17.50 dB
SFO2               400.1326008 MHz

F2 - Processing parameters
SI                       32768
SF                 100.6127784 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                      1.40

1D NMR plot parameters
CX                      20.00 cm
F1P                    200.000 ppm
F1                   20122.55 Hz
F2P                     0.000 ppm
F2                      0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM                1006.12775 Hz/cm
```

3A (HR-ES2)

Elemental Composition Report

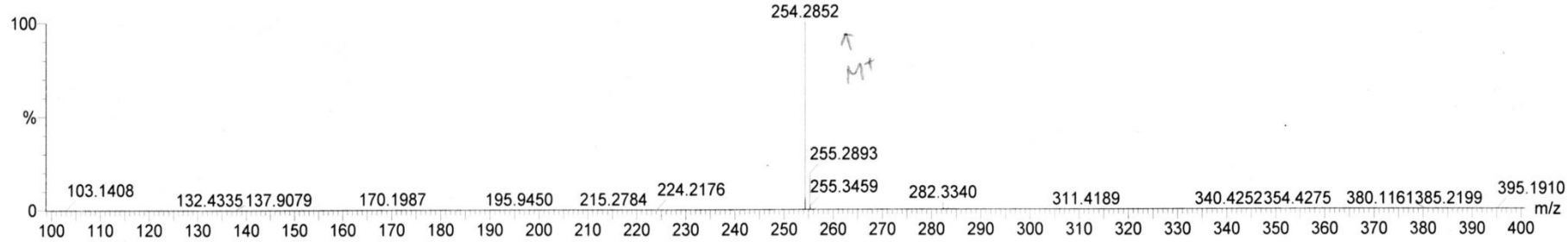


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

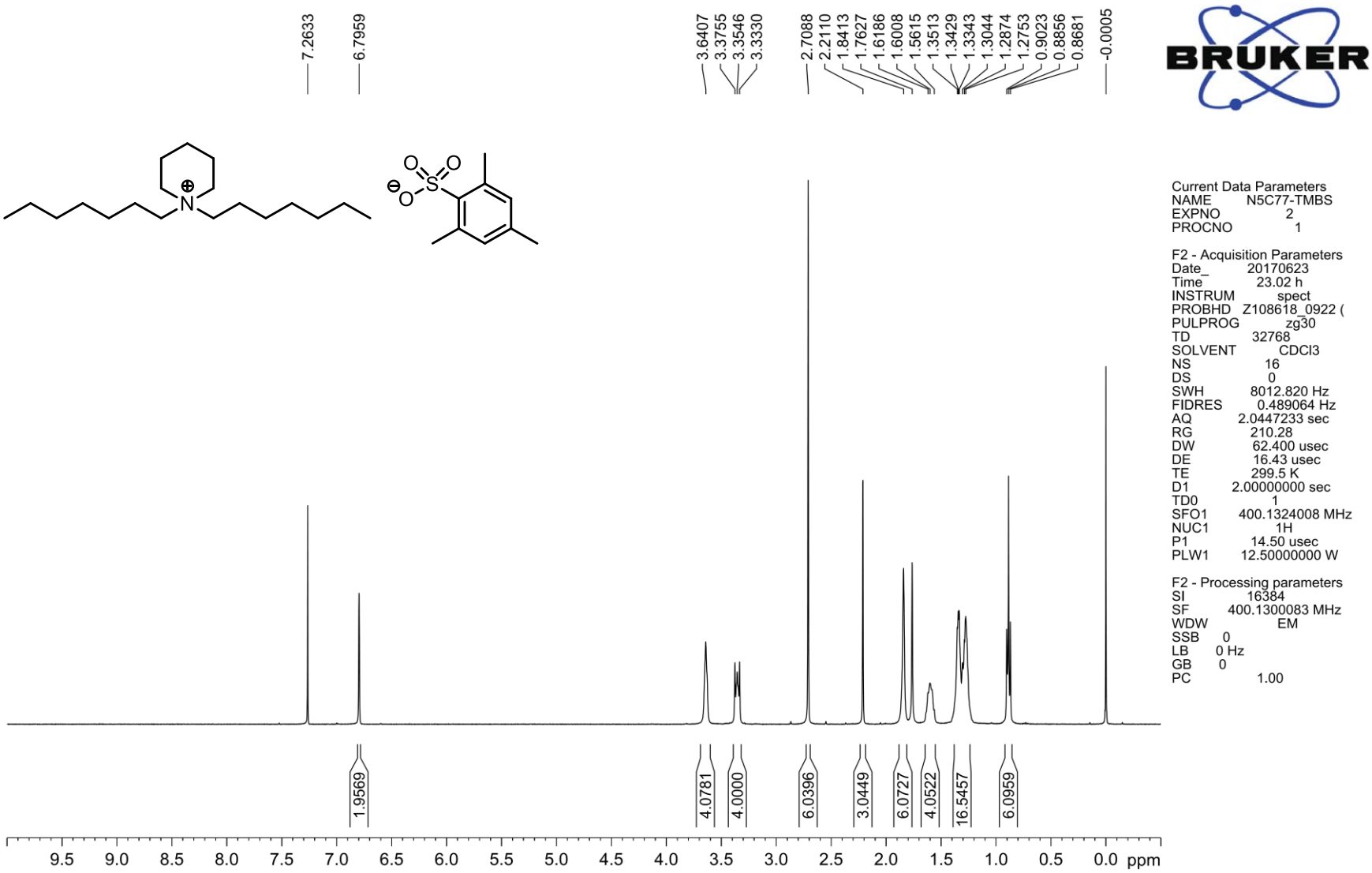
Monoisotopic Mass, Even Electron Ions
21 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

3A KE267 14-Jul-2017 12:36:19

0714_3A 90 (3.271) Cm (90-1x200.000) 1: TOF MS ES+ 6.48e+004



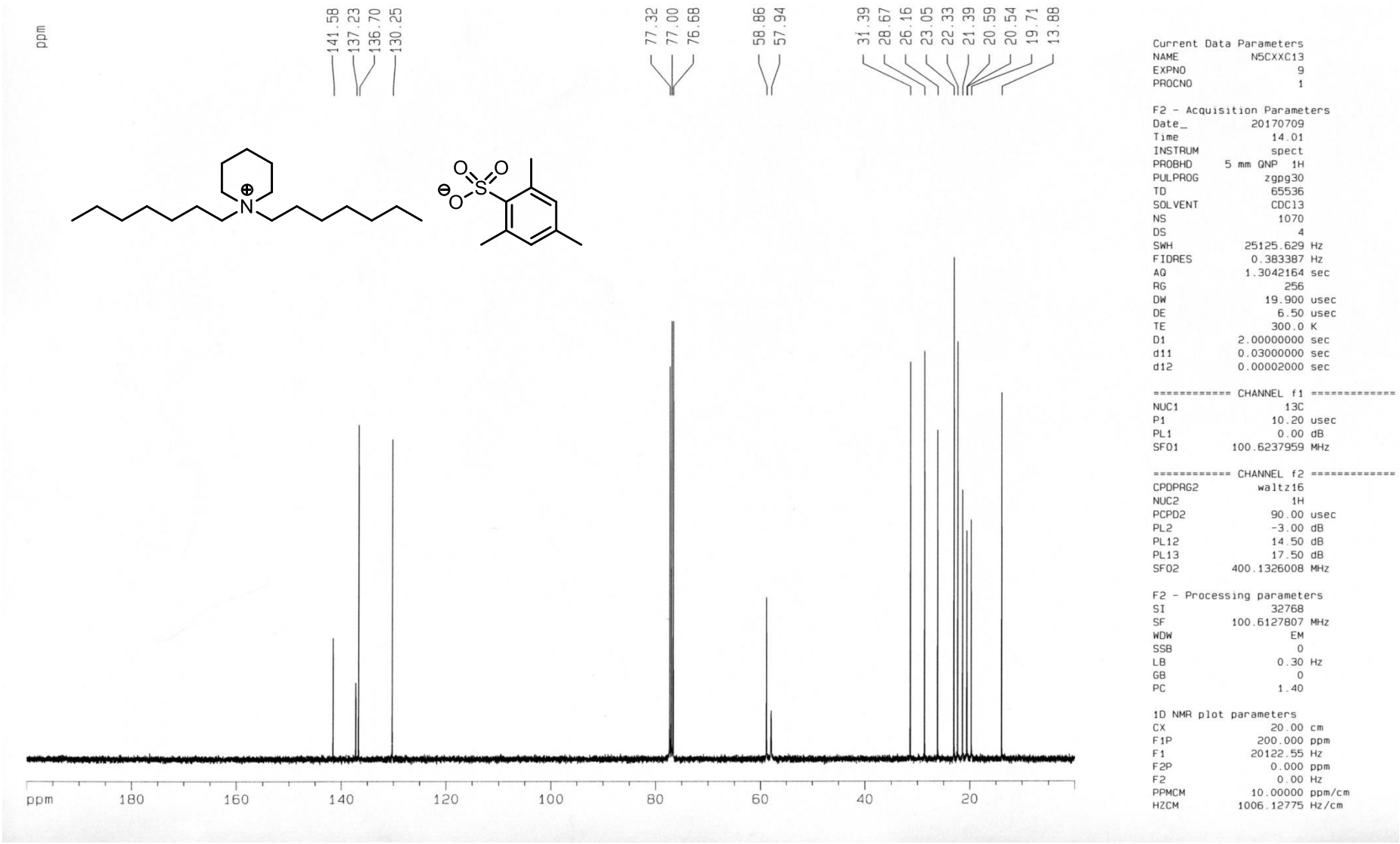
Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
254.2852	254.2848	0.4	1.6	0.5	103.0	0.0	C17 H36 N



Current Data Parameters
NAME N5C77-TMBS
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170623
Time 23.02 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.5 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME                 N5CXXC13
EXPNO                       9
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170709
Time                   14.01
INSTRUM                spect
PROBHD      5 mm QNP  1H
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      1070
DS                         4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                       256
DW                    19.900 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                      13C
P1                      10.20 usec
PL1                      0.00 dB
SFO1               100.6237959 MHz

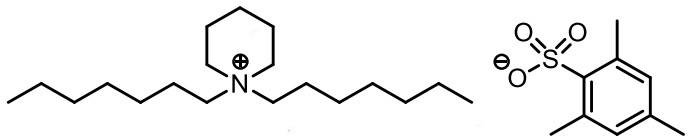
============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                     -3.00 dB
PL12                    14.50 dB
PL13                    17.50 dB
SFO2               400.1326008 MHz

F2 - Processing parameters
SI                       32768
SF                 100.6127807 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                      1.40

1D NMR plot parameters
CX                      20.00 cm
F1P                    200.000 ppm
F1                   20122.55 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM                 1006.12775 Hz/cm
```

4A (HR-ESI)

Elemental Composition Report

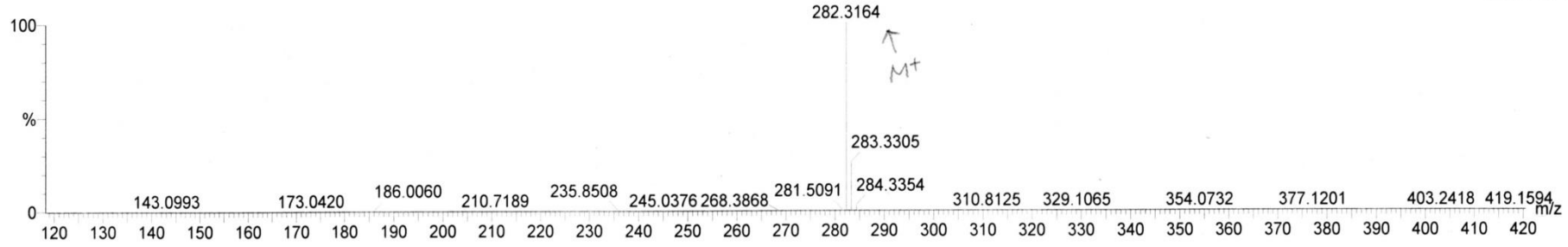


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

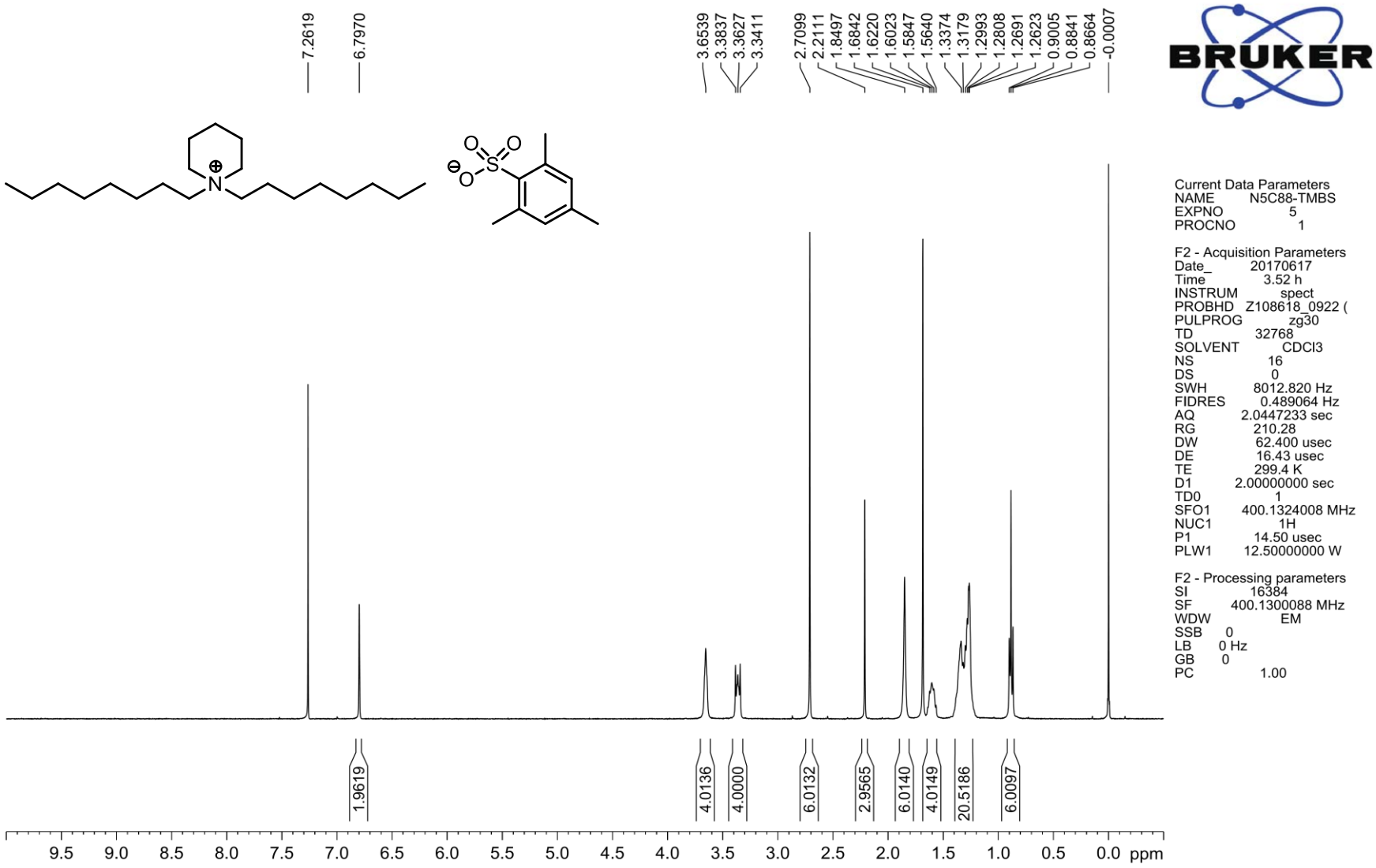
Monoisotopic Mass, Even Electron Ions
23 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

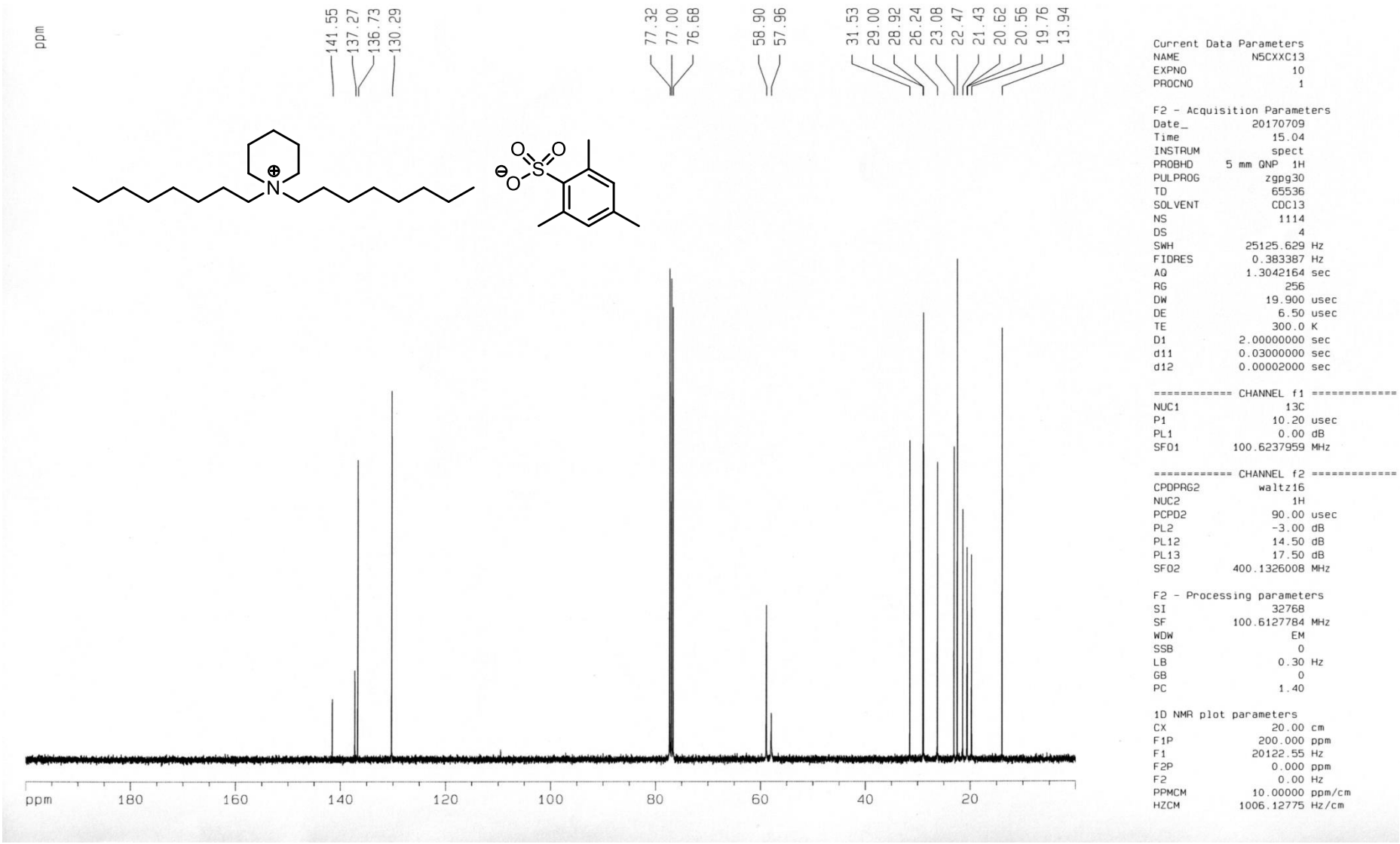
4A KE267 14-Jul-2017 12:28:11

0714_4A 69 (2.516) Cm (69-1x200.000) 1: TOF MS ES+ 3.33e+005



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
282.3164	282.3161	0.3	1.1	0.5	118.9	0.0	C19 H40 N


7.2619
6.7970
3.6539
3.3837
3.3627
3.3411
2.7099
2.2111
1.8497
1.6842
1.6220
1.6023
1.5847
1.5640
1.3374
1.3179
1.2993
1.2808
1.2691
1.2623
0.9005
0.8841
0.8664
-0.0007
1.9619
4.0136
4.0000
6.0132
2.9565
6.0140
4.0149
20.5186
6.0097
9.5 9.0 8.5 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 ppm
Current Data Parameters
NAME N5C88-TMBS
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170617
Time 3.52 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.4 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

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Current Data Parameters
NAME                 N5CXXC13
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_                20170709
Time                    15.04
INSTRUM                 spect
PROBHD       5 mm QNP  1H
PULPROG                 zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1114
DS                          4
SWH                25125.629 Hz
FIDRES              0.383387 Hz
AQ                  1.3042164 sec
RG                        256
DW                     19.900 usec
DE                      6.50 usec
TE                     300.0 K
D1                  2.00000000 sec
d11                 0.03000000 sec
d12                 0.00002000 sec

============ CHANNEL f1 =============
NUC1                       13C
P1                      10.20 usec
PL1                      0.00 dB
SFO1               100.6237959 MHz

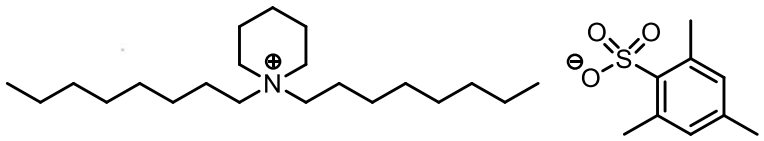
============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                     -3.00 dB
PL12                    14.50 dB
PL13                    17.50 dB
SFO2               400.1326008 MHz

F2 - Processing parameters
SI                       32768
SF                 100.6127784 MHz
WDW                         EM
SSB                          0
LB                      0.30 Hz
GB                           0
PC                      1.40

1D NMR plot parameters
CX                      20.00 cm
F1P                    200.000 ppm
F1                   20122.55 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM                 1006.12775 Hz/cm
```

5A (HR-ESI)

Elemental Composition Report

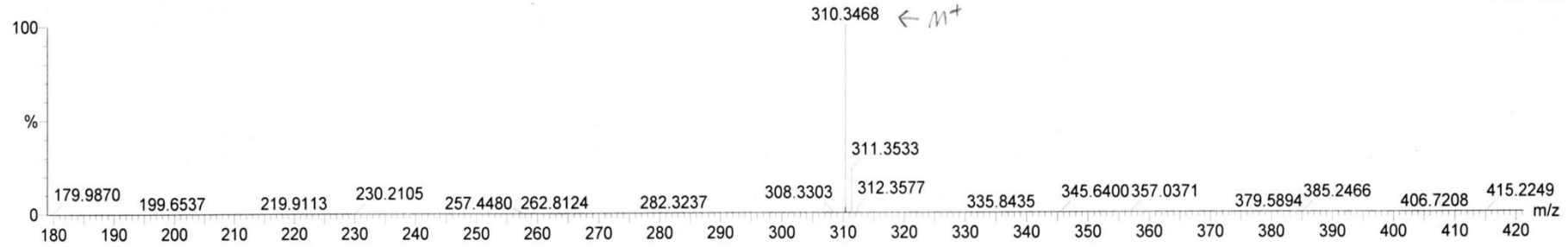


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

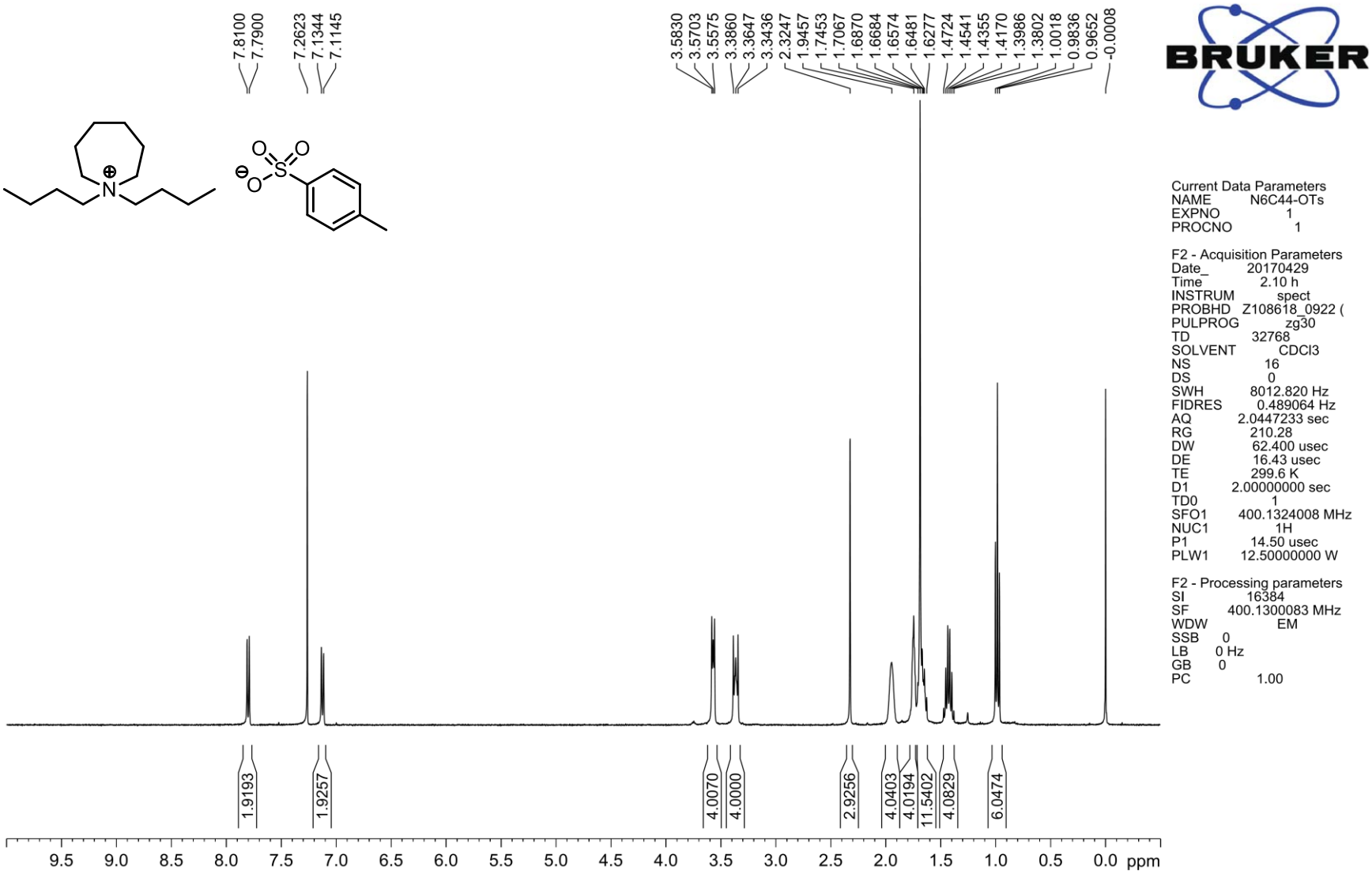
Monoisotopic Mass, Even Electron Ions
25 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-1000 H: 0-1000 N: 1-1

5A KE267 14-Jul-2017 12:20:06

0714_5A 8 (0.274) Cm (8-1x200.000) 1: TOF MS ES+ 2.48e+005



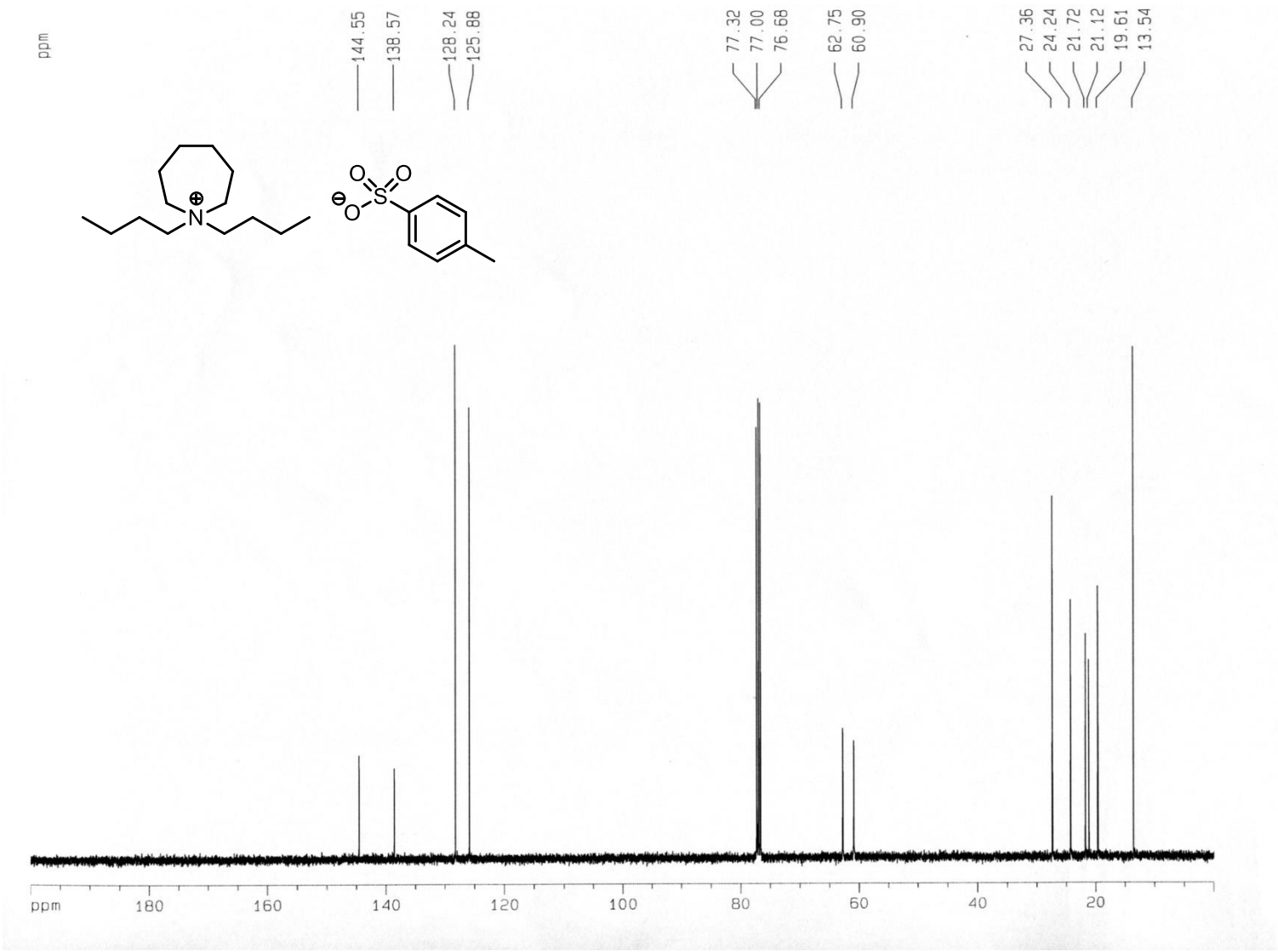
Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
310.3468	310.3474	-0.6	-1.9	0.5	83.5	0.0	C21 H44 N



Current Data Parameters
NAME N6C44-OTs
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170429
Time 2.10 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.6 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

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Current Data Parameters
NAME                 N6CXXC13
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170706
Time                   14.12
INSTRUM                spect
PROBHD      5 mm QNP  1H
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       768
DS                         4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                       256
DW                    19.900 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                     13C
P1                     10.20 usec
PL1                     0.00 dB
SFO1              100.6237959 MHz

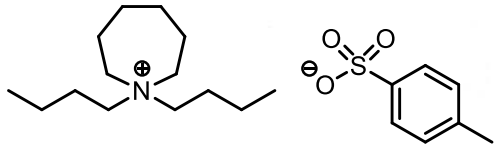
============ CHANNEL f2 =============
CPDPRG2              waltz16
NUC2                     1H
PCPD2                 90.00 usec
PL2                   -3.00 dB
PL12                  14.50 dB
PL13                  17.50 dB
SFO2              400.1326008 MHz

F2 - Processing parameters
SI                       32768
SF                100.6127800 MHz
WDW                         EM
SSB                          0
LB                     0.30 Hz
GB                           0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
F1P                  200.000 ppm
F1                 20122.55 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM              1006.12775 Hz/cm
```

1 (HR-APCI)

Elemental Composition Report

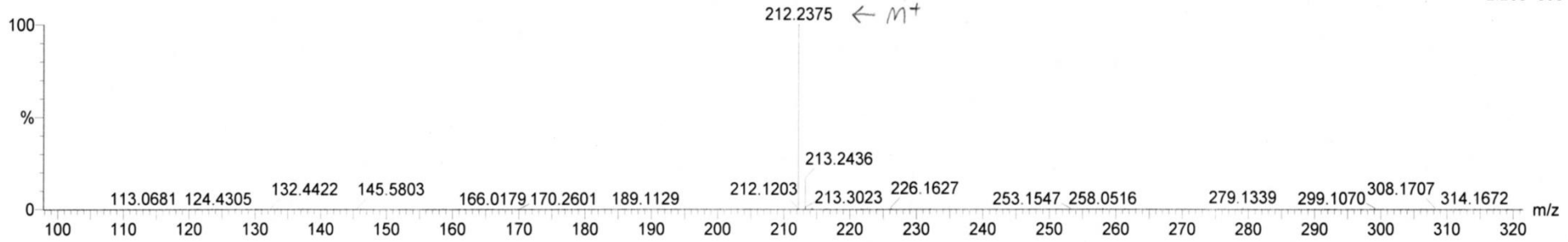


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

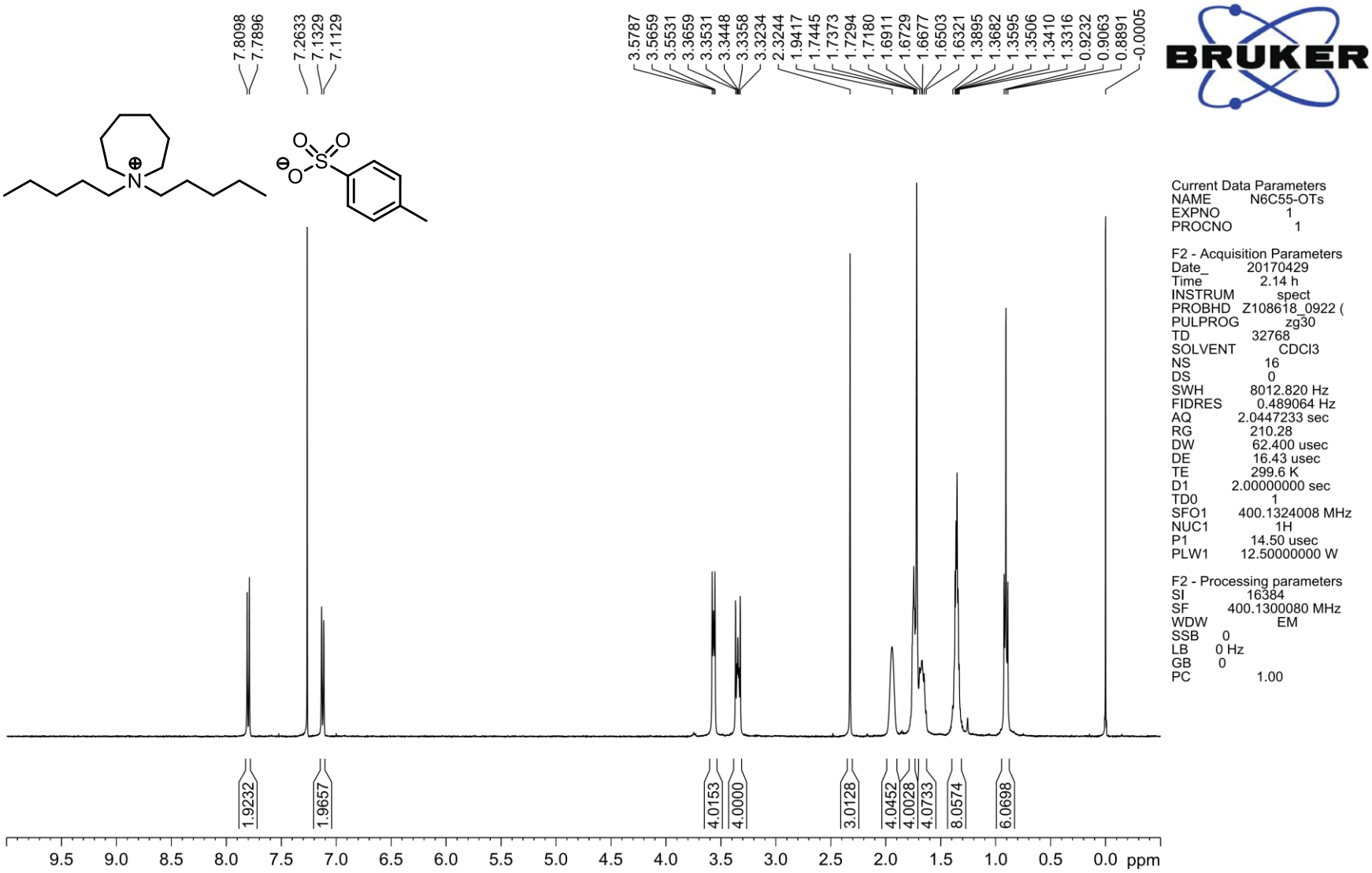
Monoisotopic Mass, Even Electron Ions
16 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 1-1000 H: 0-1000 N: 1-1

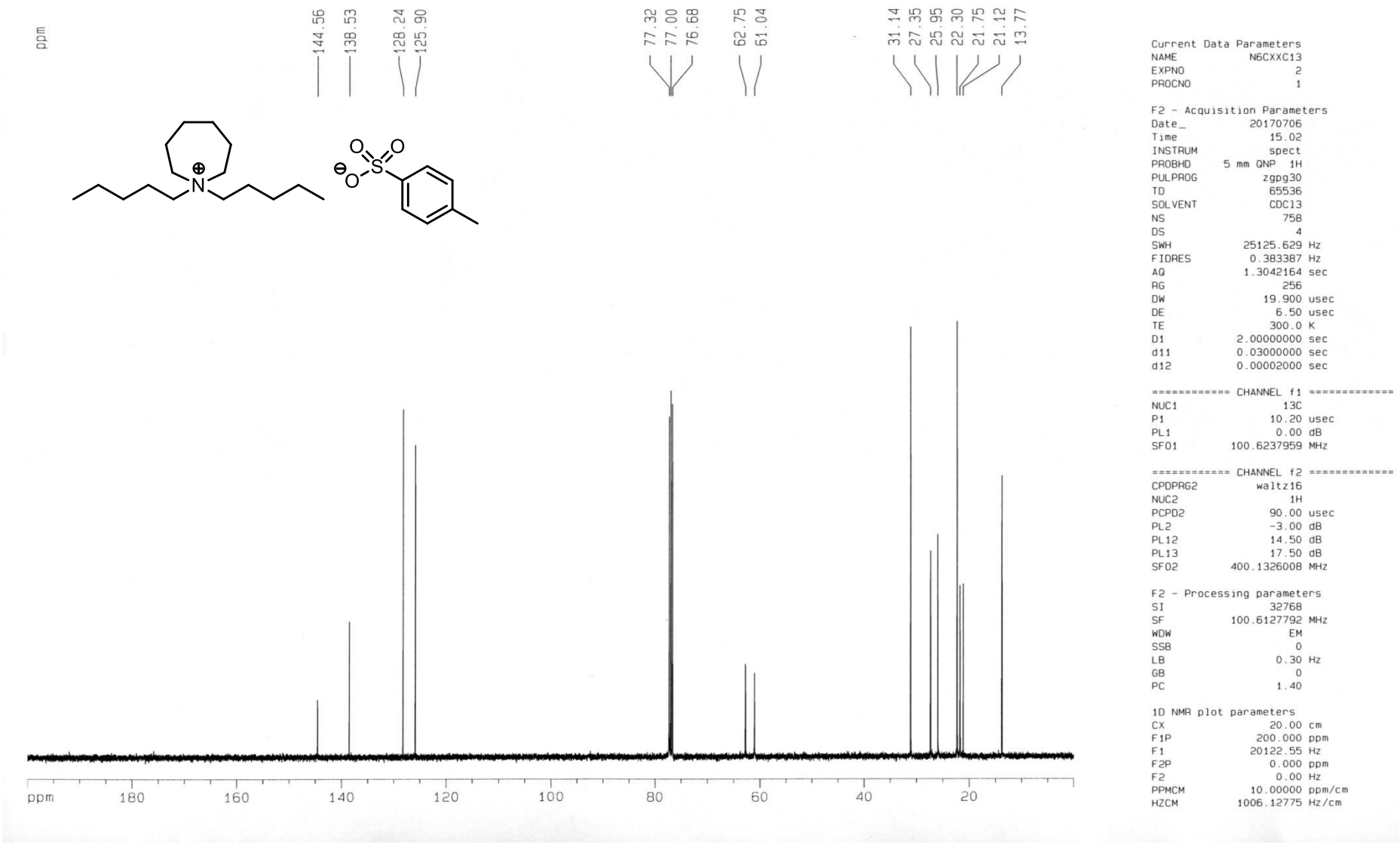
1 KE267 25-Jul-2017 14:17:52

0725_1 148 (4.300) Cm (148-1x10.000) 2: TOF MS AP+ 2.20e+003



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
212.2375	212.2378	-0.3	-1.4	0.5	32.2	0.0	C14 H30 N


7.8098
7.7896
7.2633
7.1329
7.1129
3.5787
3.5659
3.5531
3.3659
3.3531
3.3448
3.3358
3.3234
2.3244
1.9417
1.7445
1.7373
1.7294
1.7180
1.6911
1.6729
1.6677
1.6503
1.6321
1.3895
1.3682
1.3595
1.3506
1.3410
1.3316
0.9232
0.9063
0.8891
-0.0005
BRUKER
Current Data Parameters
NAME N6C55-OTs
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170429
Time 2.14 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.6 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300080 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9232
1.9657
4.0153
4.0000
3.0128
4.0452
4.0028
4.0733
8.0574
6.0698
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME            N6CXXC13
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170706
Time               15.02
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  758
DS                    4
SWH           25125.629 Hz
FIDRES         0.383387 Hz
AQ             1.3042164 sec
RG                  256
DW               19.900 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1               10.20 usec
PL1               0.00 dB
SFO1         100.6237959 MHz

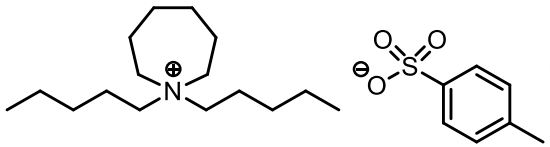
============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127792 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
F1P               200.000 ppm
F1              20122.55 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1006.12775 Hz/cm
```

2 (HR-ESI)

Elemental Composition Report



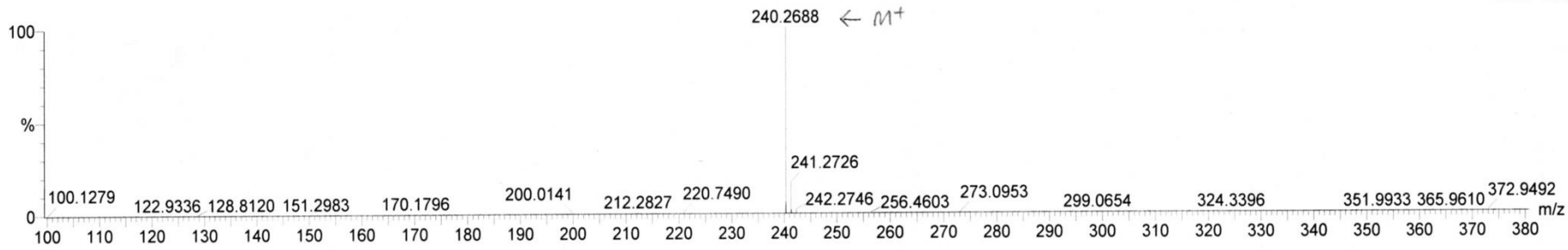
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
18 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 1-1000 H: 0-1000 N: 1-1
2

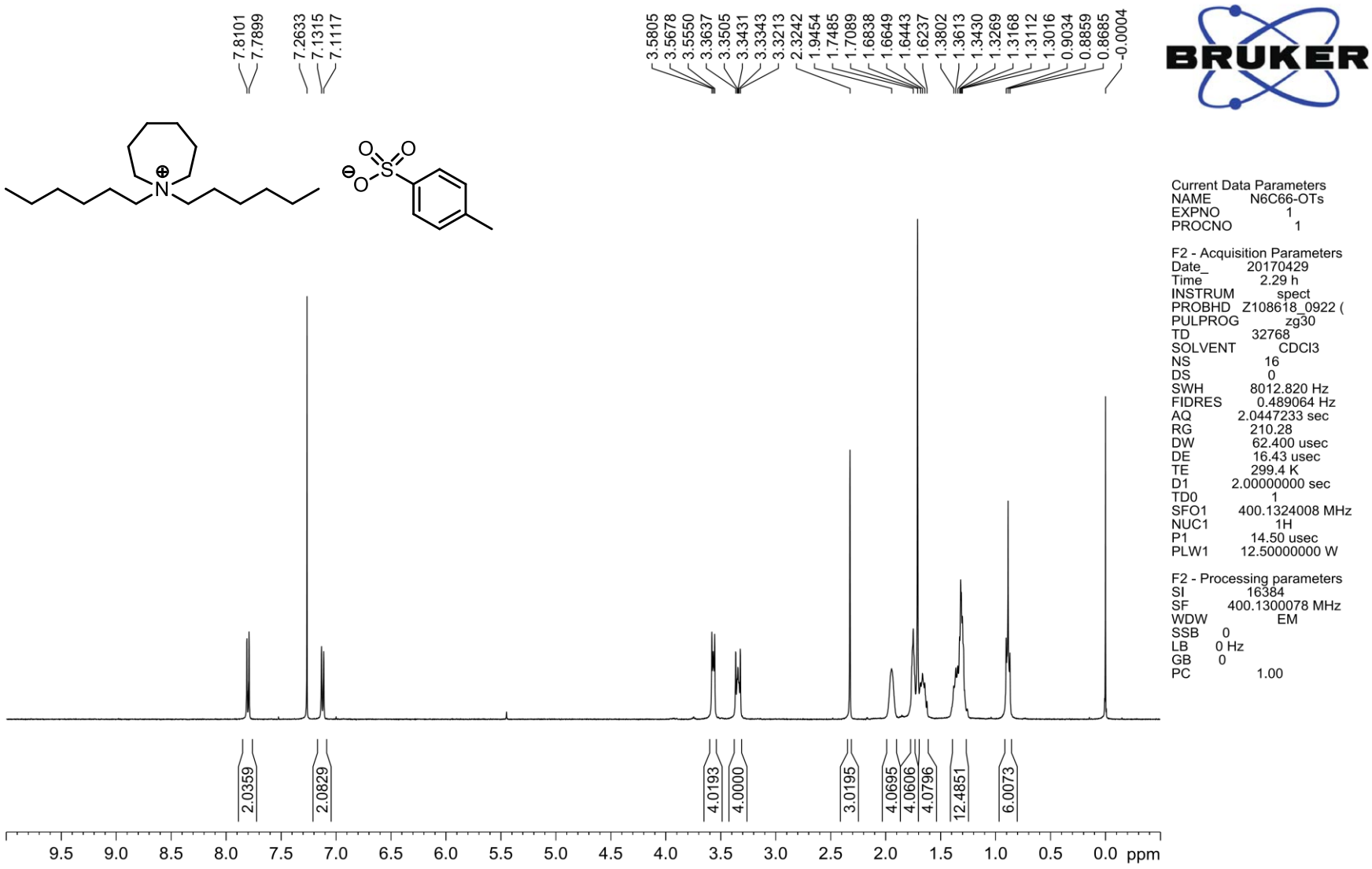
KE267

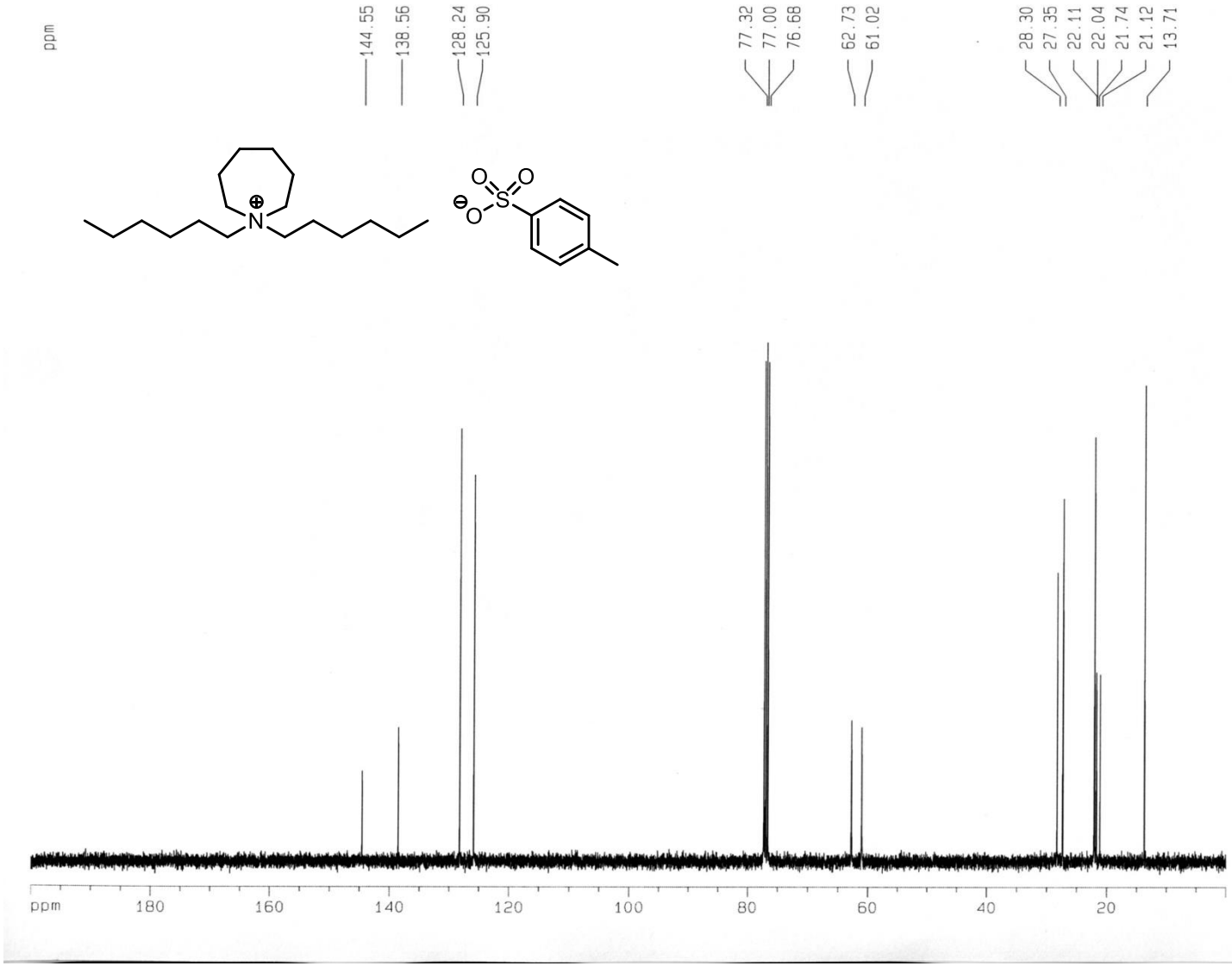
25-Jul-2017
14:25:58
1: TOF MS ES+
1.36e+004

0725_2 72 (2.599) Cm (72-1x10.000)



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
240.2688	240.2691	-0.3	-1.2	0.5	69.4	0.0	C16 H34 N


7.8101
7.7899
7.2633
7.1315
7.1117
3.5805
3.5678
3.5550
3.3637
3.3505
3.3431
3.3343
3.3213
2.3242
1.9454
1.7485
1.7089
1.6838
1.6649
1.6443
1.6237
1.3802
1.3613
1.3430
1.3269
1.3168
1.3112
1.3016
0.9034
0.8859
0.8685
-0.0004
BRUKER
Current Data Parameters
NAME N6C66-OTs
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170429
Time 2.29 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.4 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
2.0359
2.0829
4.0193
4.0000
3.0195
4.0695
4.0606
4.0796
12.4851
6.0073
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                 N6CXXC13
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170706
Time                   15.48
INSTRUM                spect
PROBHD      5 mm QNP  1H
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        4
SWH               25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                      256
DW                   19.900 usec
DE                    6.50 usec
TE                   300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 =============
NUC1                    13C
P1                   10.20 usec
PL1                   0.00 dB
SFO1             100.6237959 MHz

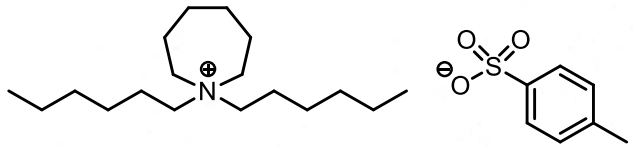
============ CHANNEL f2 =============
CPDPRG2              waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -3.00 dB
PL12                 14.50 dB
PL13                 17.50 dB
SFO2             400.1326008 MHz

F2 - Processing parameters
SI                      32768
SF               100.6127792 MHz
WDW                        EM
SSB                         0
LB                    0.30 Hz
GB                          0
PC                     1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                200.000 ppm
F1                 20122.55 Hz
F2P                  0.000 ppm
F2                    0.00 Hz
PPMCM             10.00000 ppm/cm
HZCM              1006.12775 Hz/cm
```

3 (HR-ESI)

Elemental Composition Report

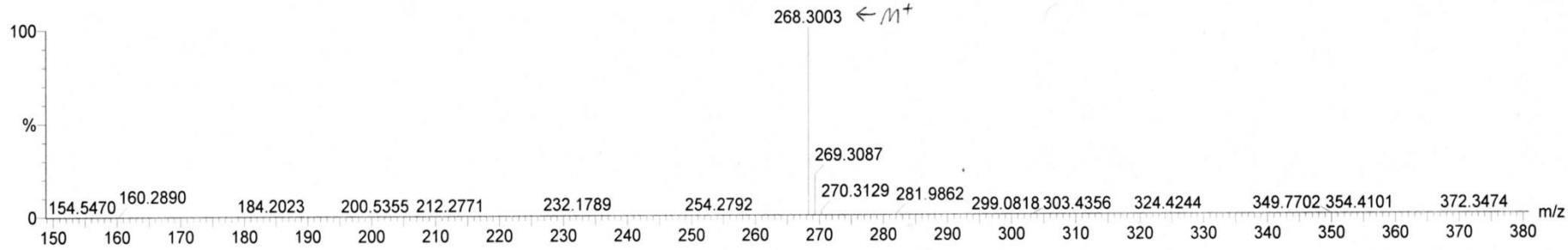


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

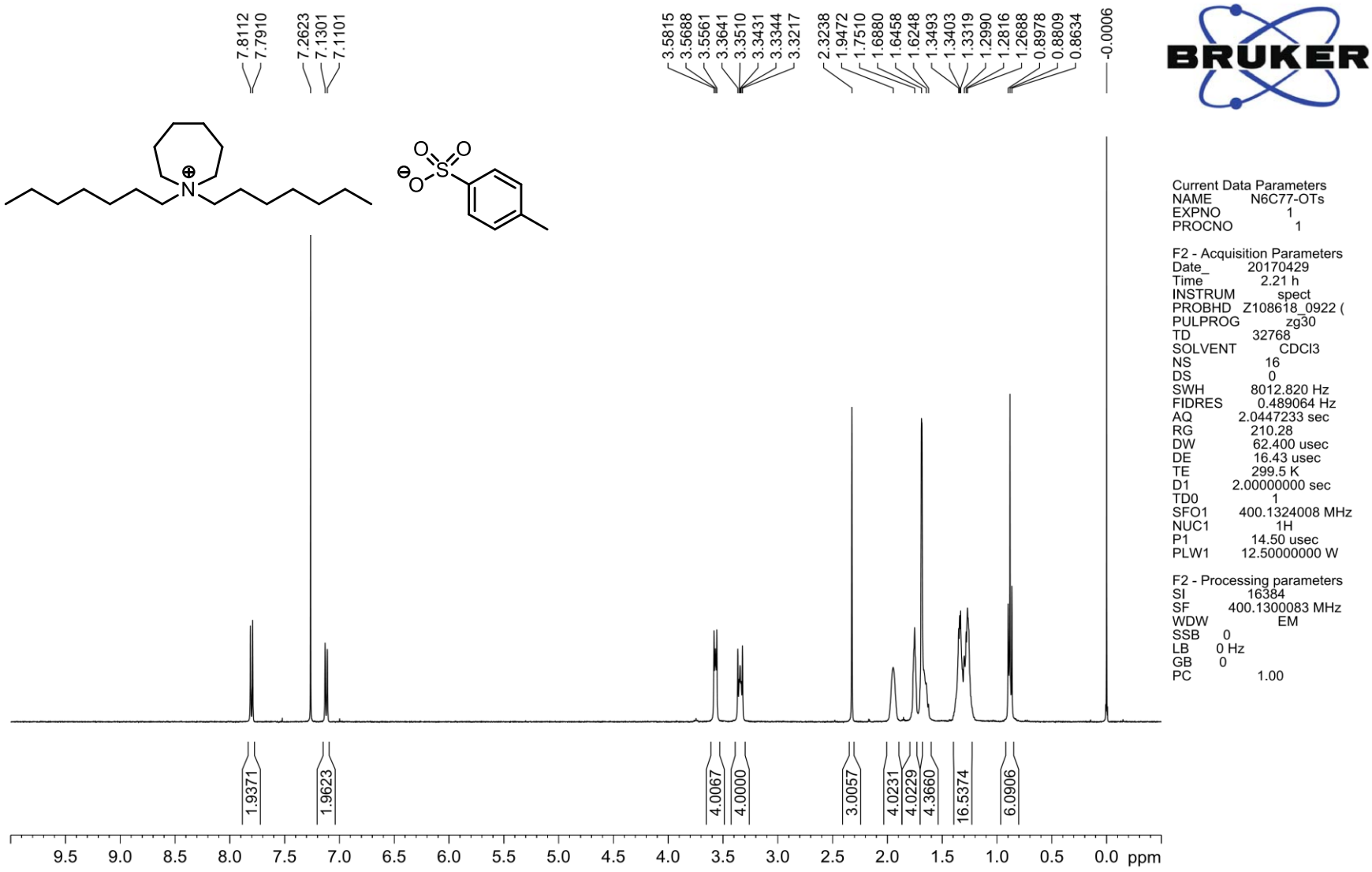
Monoisotopic Mass, Even Electron Ions
21 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 1-1000 H: 0-1000 N: 1-1

3 KE267 25-Jul-2017 14:34:08

0725_3 37 (1.352) Cm (37-1x10.000) 1: TOF MS ES+ 9.77e+004



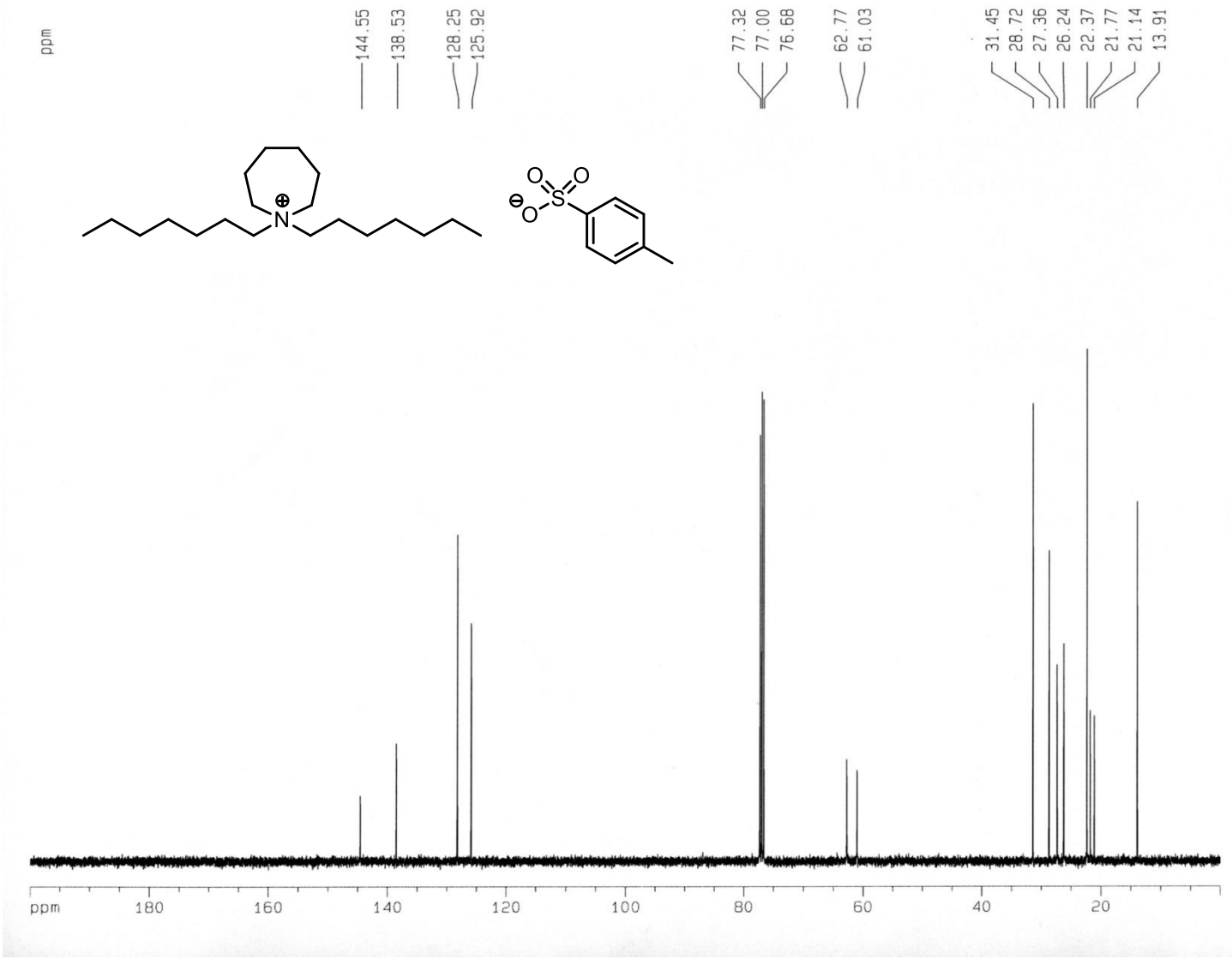
Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
268.3003	268.3004	-0.1	-0.4	0.5	61.3	0.0	C18 H38 N



Current Data Parameters
NAME N6C77-OTs
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170429
Time 2.21 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.5 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME            N6CXXC13
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170706
Time               16.49
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  487
DS                    4
SWH           25125.629 Hz
FIDRES            0.383387 Hz
AQ             1.3042164 sec
RG                  256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1         100.6237959 MHz

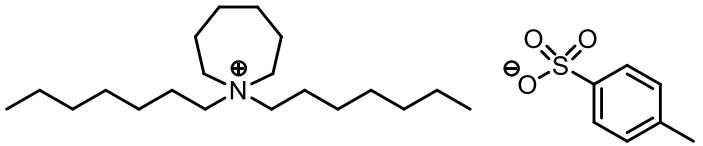
============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2         400.1326008 MHz

F2 - Processing parameters
SI                32768
SF           100.6127784 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              200.000 ppm
F1             20122.55 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1006.12775 Hz/cm
```

4 (HR-ESI)

Elemental Composition Report

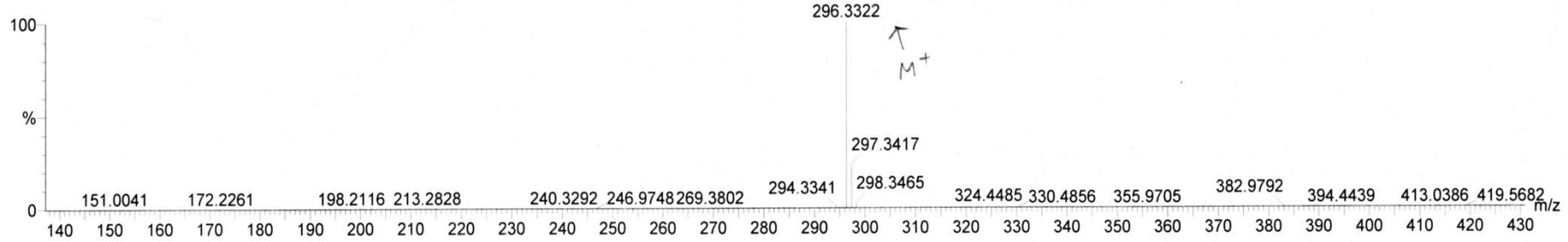


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

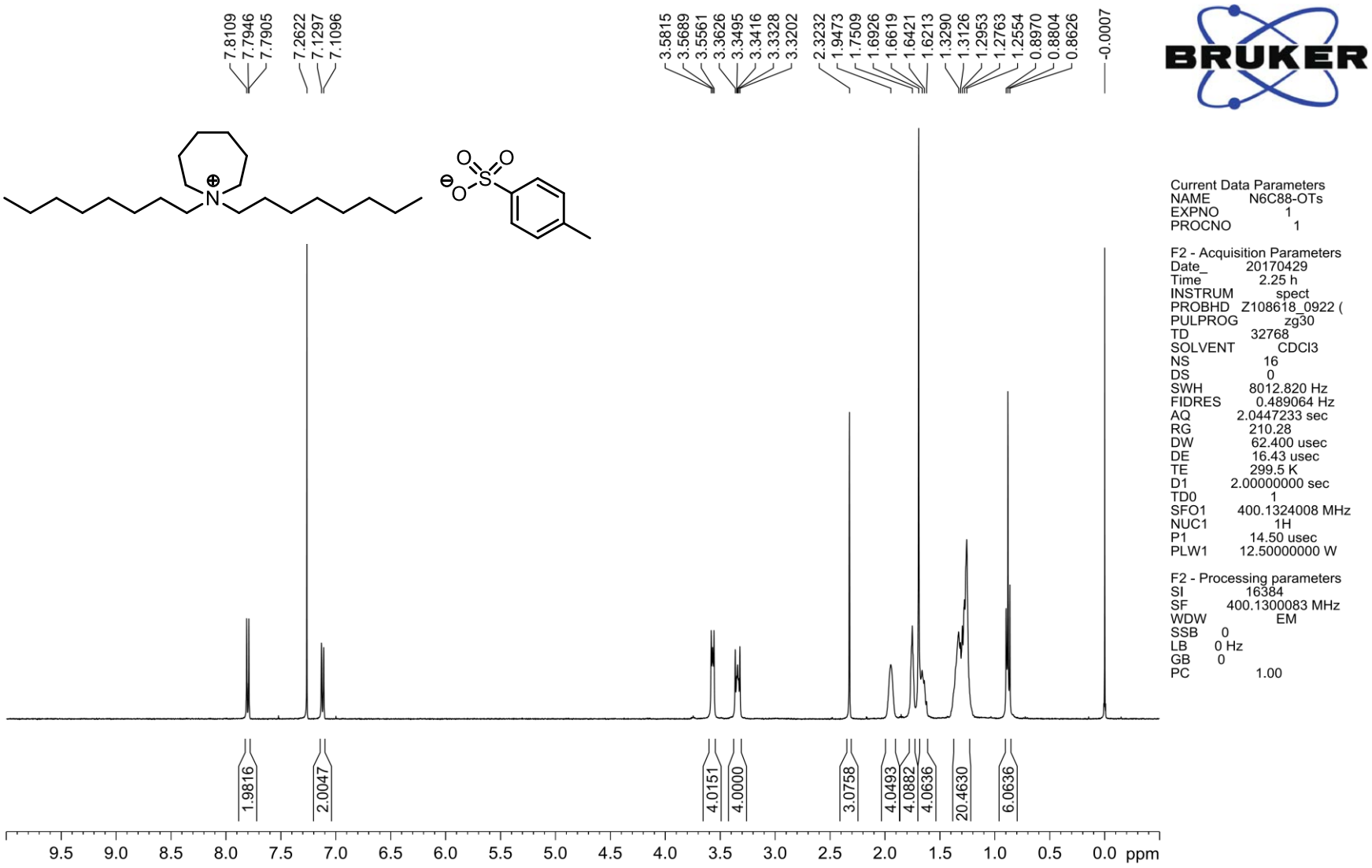
Monoisotopic Mass, Even Electron Ions
23 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 1-1000 H: 0-1000 N: 1-1

4 KE267 25-Jul-2017 14:58:30

0725_4 124 (4.487) Cm (124-1x10.000) 1: TOF MS ES+ 1.03e+005



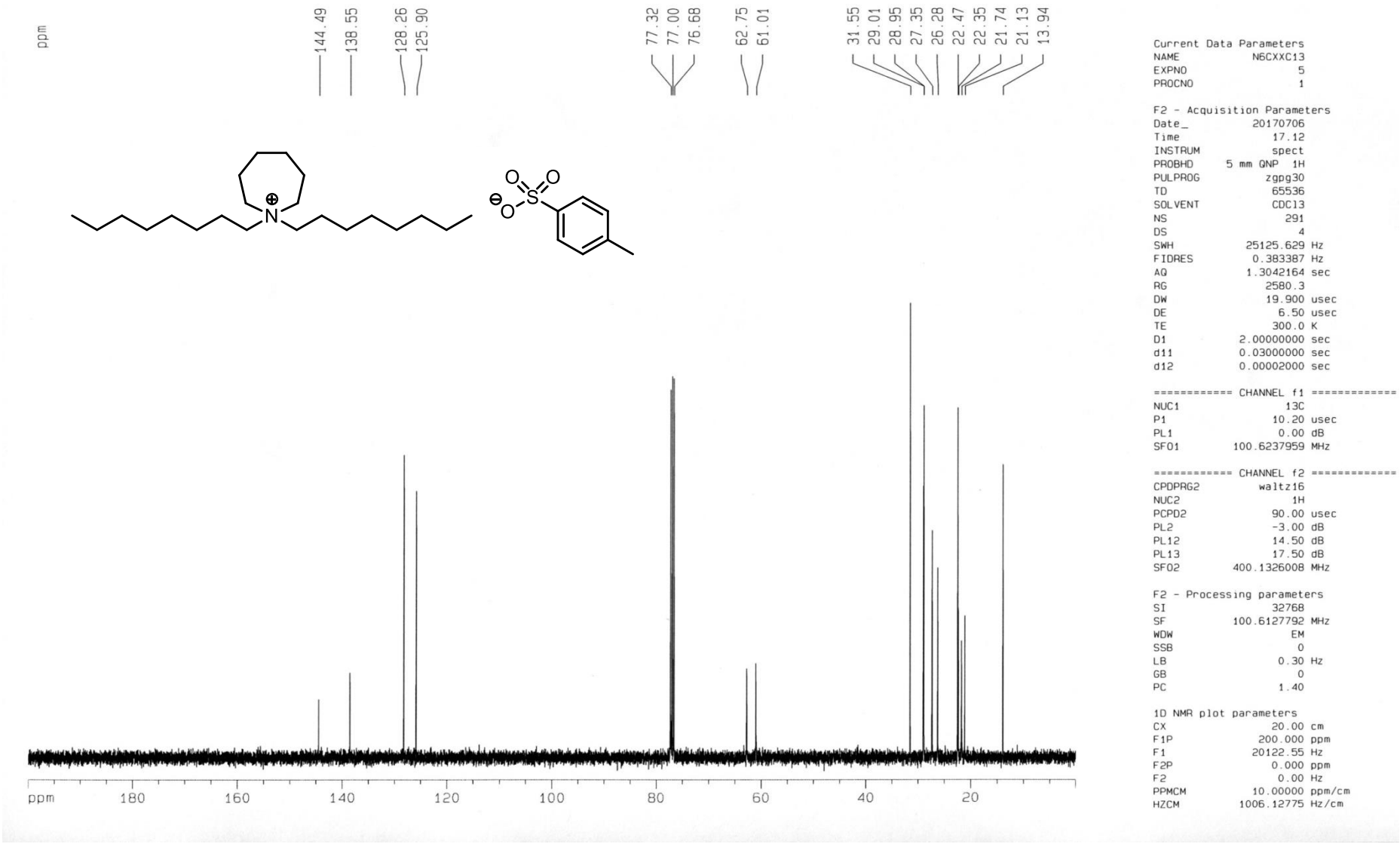
Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
296.3322	296.3317	0.5	1.7	0.5	107.7	0.0	C20 H42 N



Current Data Parameters
NAME N6C88-OTs
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170429
Time 2.25 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.5 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME                 N6CXXC13
EXPNO                       5
PROCNO                      1

F2 - Acquisition Parameters
Date_                20170706
Time                    17.12
INSTRUM                 spect
PROBHD       5 mm QNP  1H
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        291
DS                          4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                    2580.3
DW                   19.900 usec
DE                    6.50 usec
TE                   300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                      13C
P1                      10.20 usec
PL1                      0.00 dB
SFO1               100.6237959 MHz

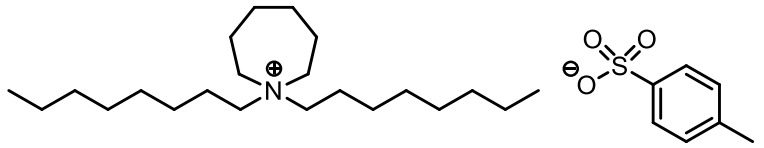
============ CHANNEL f2 =============
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                     -3.00 dB
PL12                    14.50 dB
PL13                    17.50 dB
SFO2               400.1326008 MHz

F2 - Processing parameters
SI                        32768
SF                 100.6127792 MHz
WDW                          EM
SSB                           0
LB                       0.30 Hz
GB                            0
PC                       1.40

1D NMR plot parameters
CX                      20.00 cm
F1P                    200.000 ppm
F1                   20122.55 Hz
F2P                     0.000 ppm
F2                      0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM                 1006.12775 Hz/cm
```

5 (HR-ESI)

Elemental Composition Report



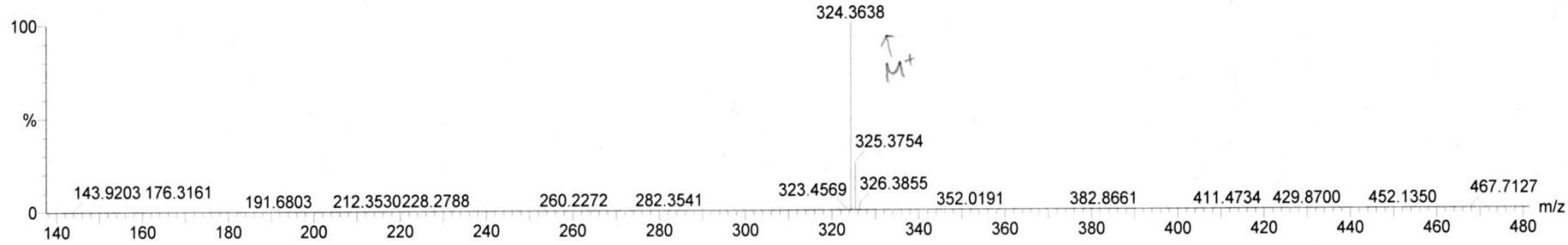
Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
25 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 1-1000 H: 0-1000 N: 1-1
5

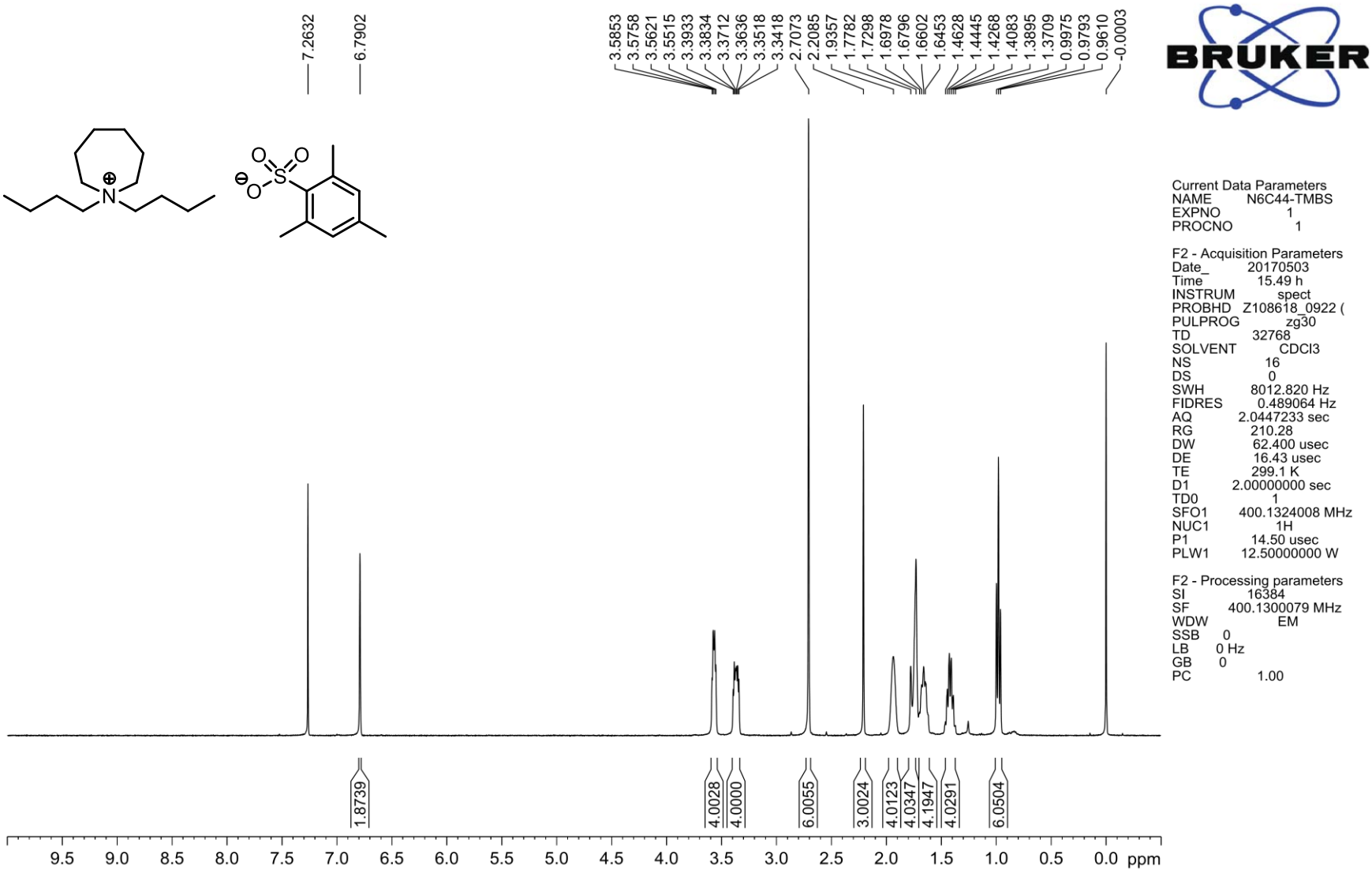
KE267

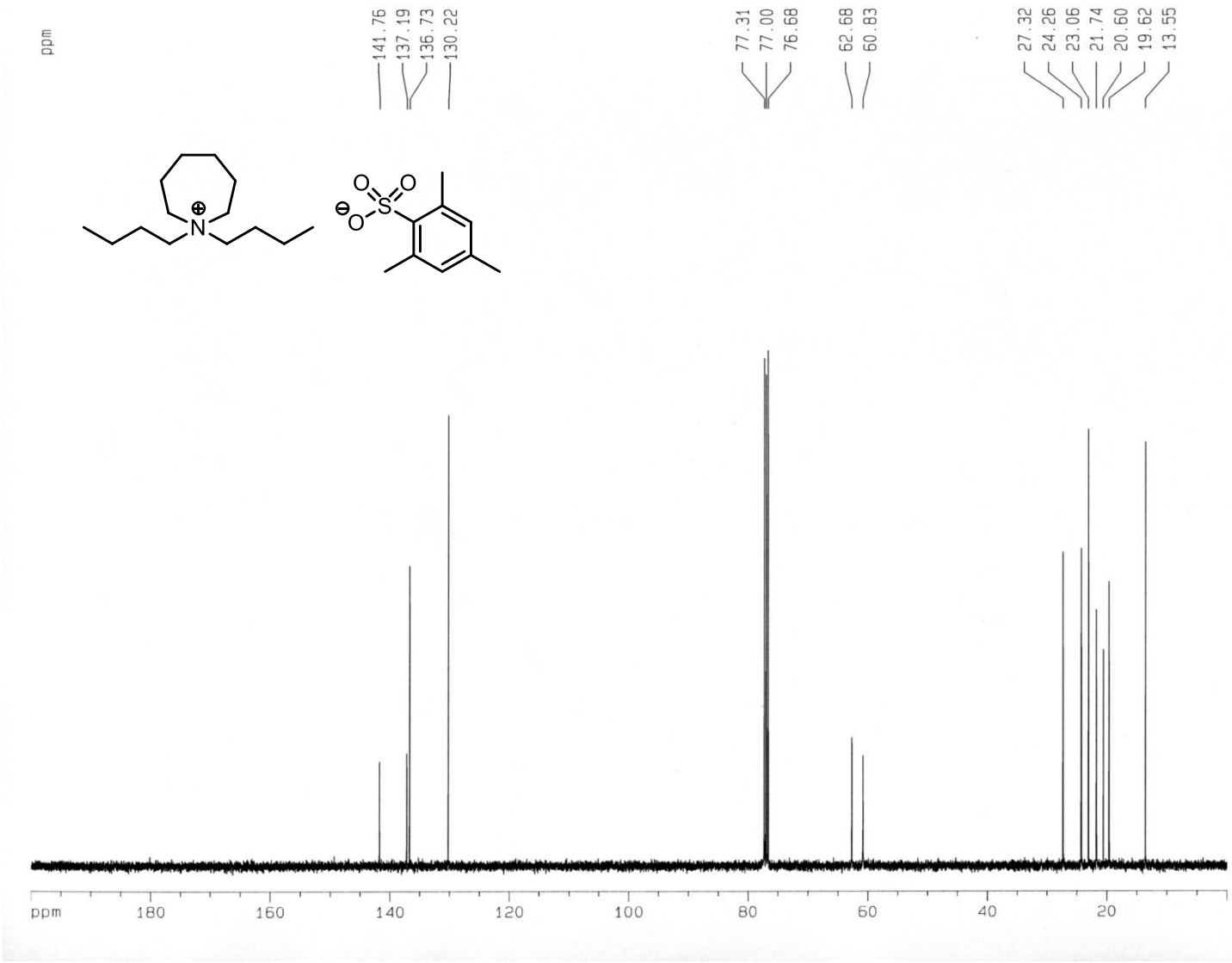
25-Jul-2017
14:50:26
1: TOF MS ES+
1.28e+005

0725_5 43 (1.555) Cm (43-1x10.000)



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
324.3638	324.3630	0.8	2.5	0.5	104.6	0.0	C22 H46 N


7.2632
6.7902
3.5853
3.5758
3.5621
3.5515
3.3933
3.3834
3.3712
3.3636
3.3518
3.3418
2.7073
2.2085
1.9357
1.7782
1.7298
1.6978
1.6796
1.6602
1.6453
1.4628
1.4445
1.4268
1.4083
1.3895
1.3709
0.9975
0.9793
0.9610
-0.0003
BRUKER
Current Data Parameters
NAME N6C44-TMBS
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170503
Time 15.49 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.1 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300079 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.8739
4.0028
4.0000
6.0055
3.0024
4.0123
4.0347
4.1947
4.0291
6.0504
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                 N6CXXC13
EXPNO                       6
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170707
Time                   12.07
INSTRUM                spect
PROBHD      5 mm QNP  1H
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       569
DS                         4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                       256
DW                    19.900 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                     13C
P1                    10.20 usec
PL1                    0.00 dB
SFO1              100.6237959 MHz

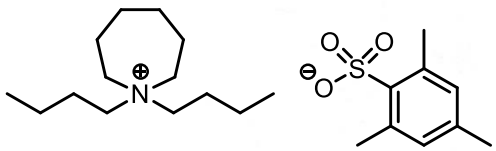
============ CHANNEL f2 =============
CPDPRG2              waltz16
NUC2                     1H
PCPD2                 90.00 usec
PL2                   -3.00 dB
PL12                  14.50 dB
PL13                  17.50 dB
SFO2              400.1326008 MHz

F2 - Processing parameters
SI                       32768
SF                100.6127792 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                    1.40

1D NMR plot parameters
CX                    20.00 cm
F1P                  200.000 ppm
F1                 20122.55 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM               1006.12775 Hz/cm
```

1A (HR-ESI)

Elemental Composition Report



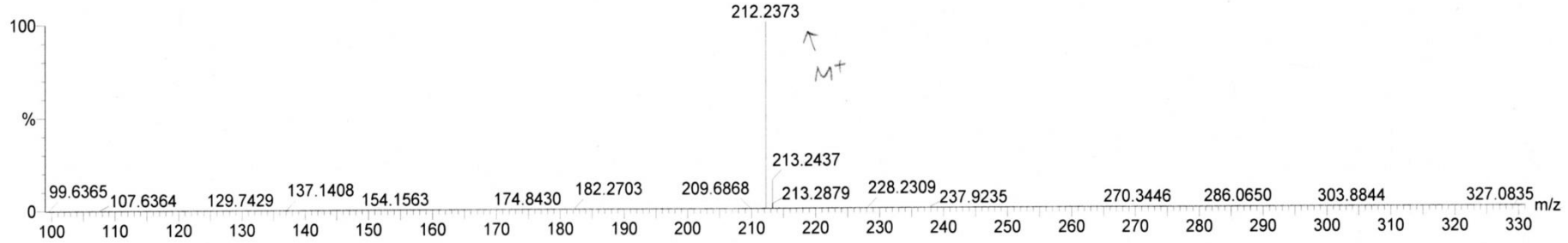
Single Mass Analysis

Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

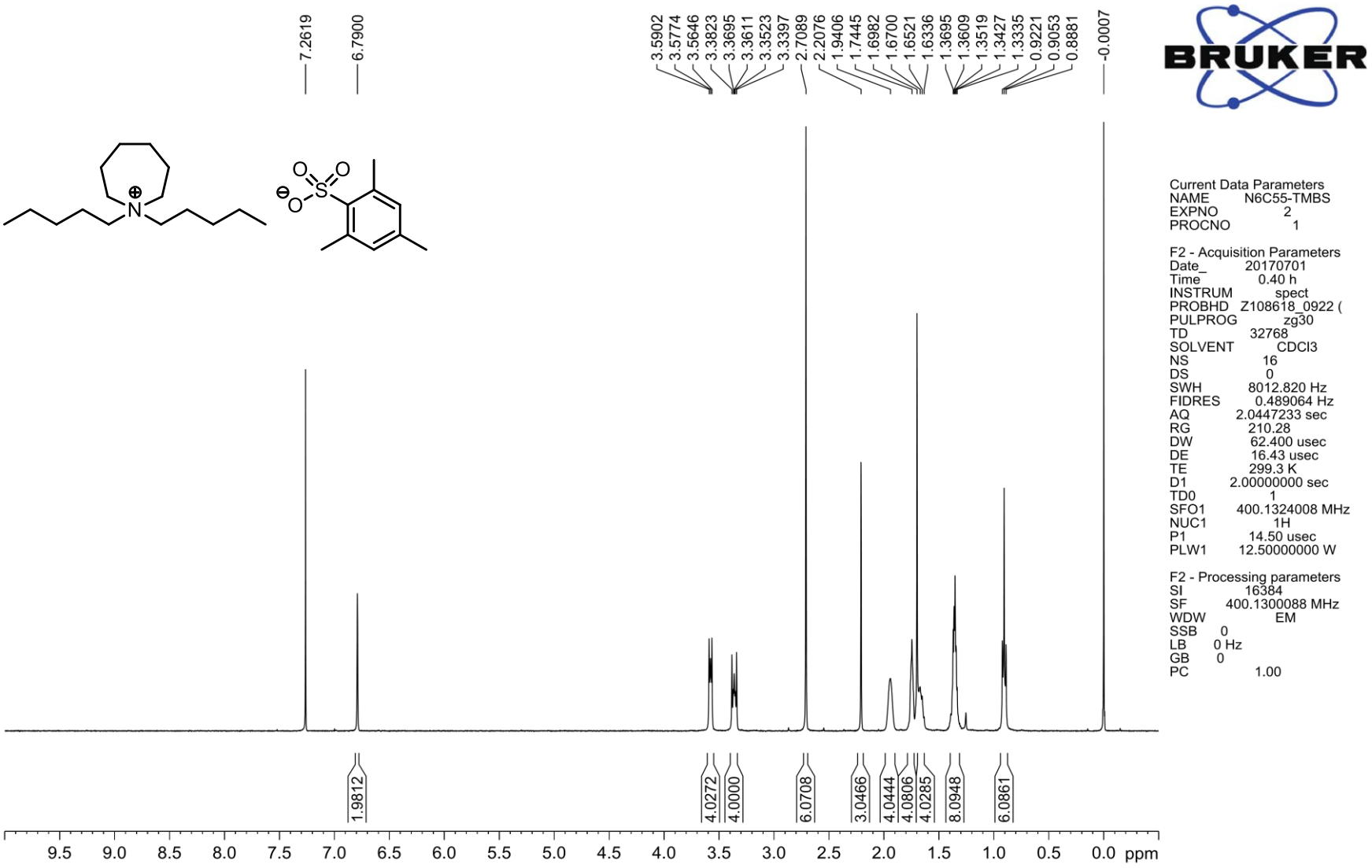
Monoisotopic Mass, Even Electron Ions
16 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 1-1000 H: 0-1000 N: 1-1

1A KE267 25-Jul-2017 14:42:17

0725_1A 32 (1.148) Cm (32-1) 1: TOF MS ES+ 1.26e+004



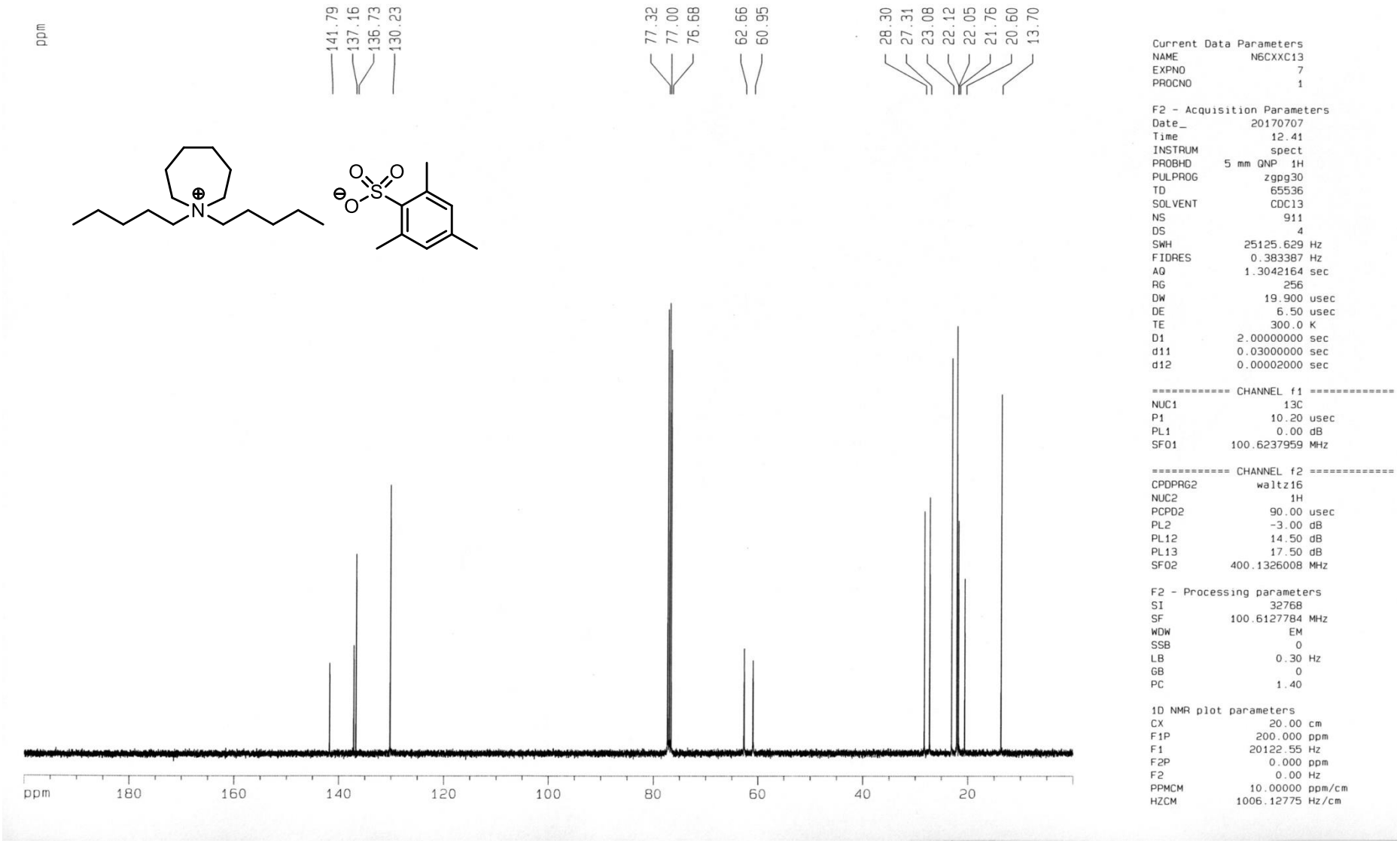
Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
212.2373	212.2378	-0.5	-2.4	0.5	56.9	0.0	C14 H30 N



Current Data Parameters
NAME N6C55-TMBS
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170701
Time 0.40 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.3 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W

F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00




```
Current Data Parameters
NAME              N6CXXC13
EXPNO                    7
PROCNO                   1

F2 - Acquisition Parameters
Date_              20170707
Time                 12.41
INSTRUM              spect
PROBHD     5 mm QNP  1H
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     911
DS                       4
SWH              25125.629 Hz
FIDRES           0.383387 Hz
AQ               1.3042164 sec
RG                     256
DW                19.900 usec
DE                 6.50 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 =============
NUC1                   13C
P1                  10.20 usec
PL1                  0.00 dB
SF01          100.6237959 MHz

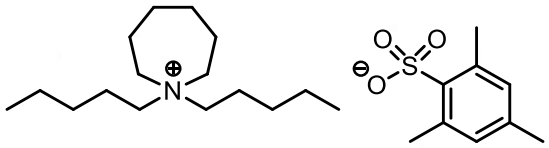
============ CHANNEL f2 =============
CPDPRG2             waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -3.00 dB
PL12                14.50 dB
PL13                17.50 dB
SF02          400.1326008 MHz

F2 - Processing parameters
SI                     32768
SF            100.6127784 MHz
WDW                       EM
SSB                        0
LB                   0.30 Hz
GB                         0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                200.000 ppm
F1               20122.55 Hz
F2P                  0.000 ppm
F2                   0.00 Hz
PPMCM             10.00000 ppm/cm
HZCM           1006.12775 Hz/cm
```

2A (HR-APCI)

Elemental Composition Report

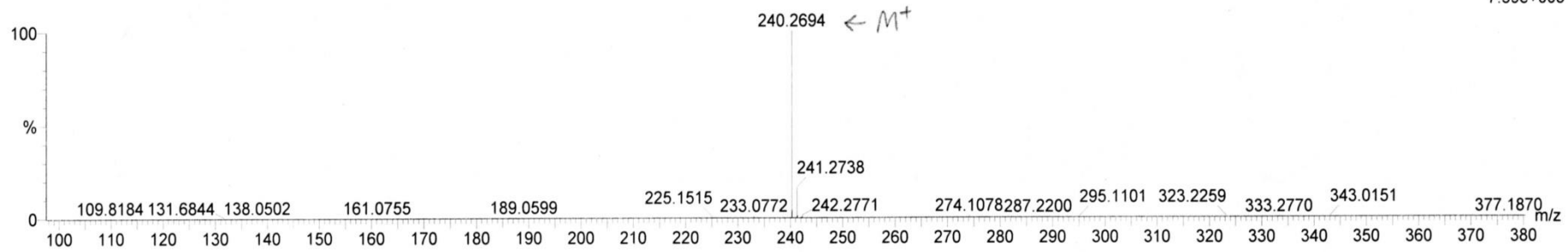


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

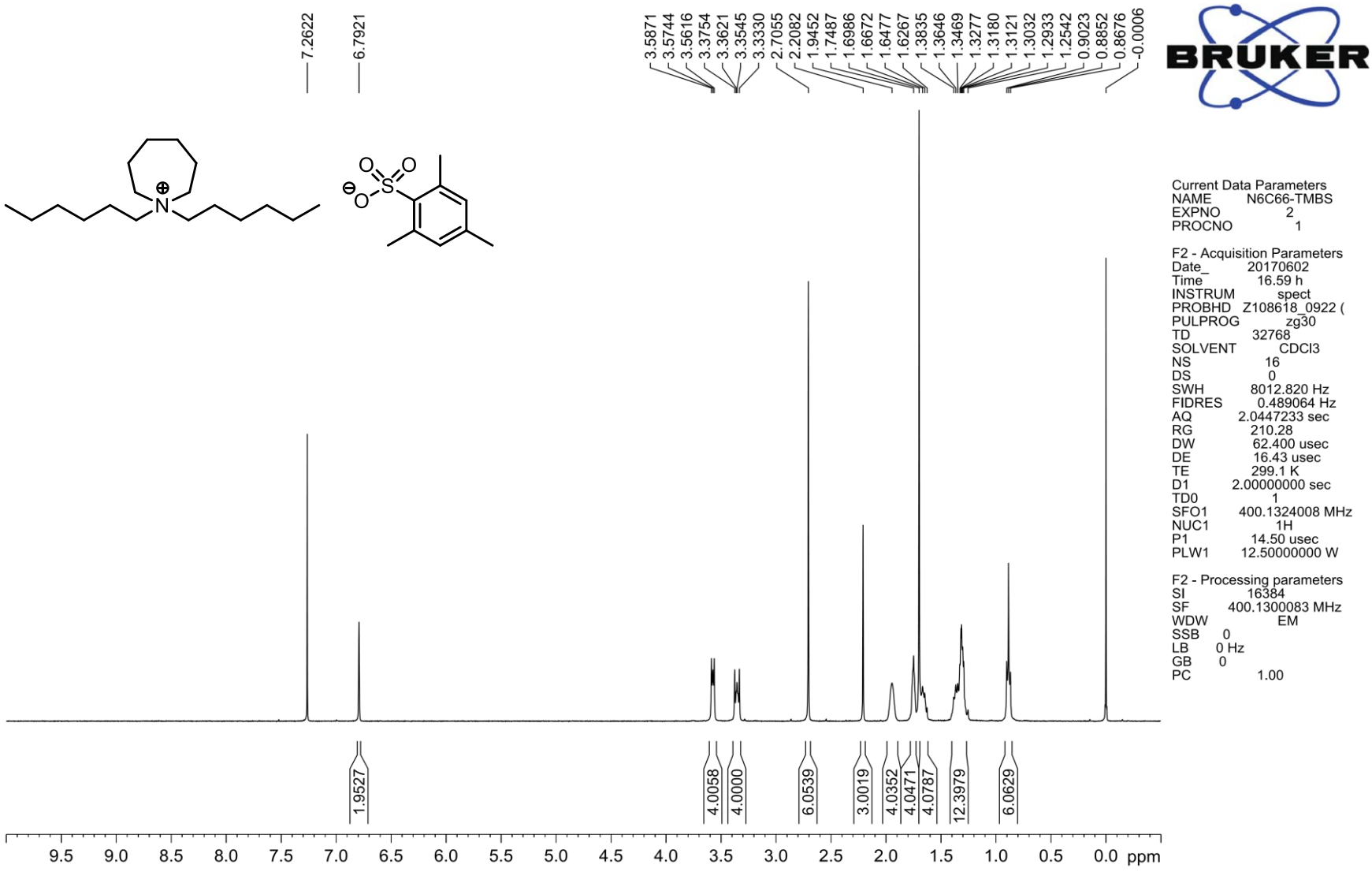
Monoisotopic Mass, Even Electron Ions
18 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 1-1000 H: 0-1000 N: 1-1

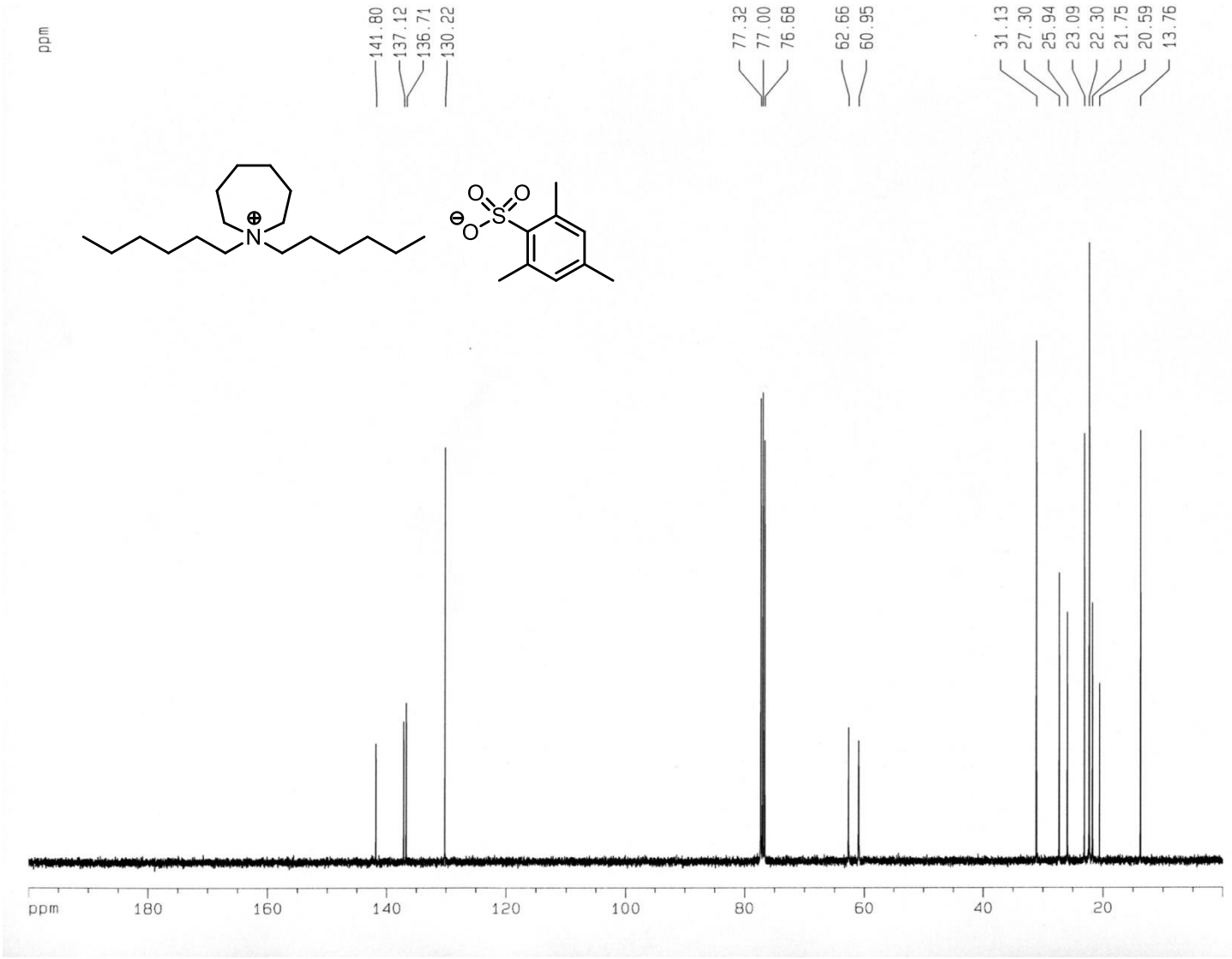
2A KE267 25-Jul-2017 12:40:51

0725_2A 59 (1.715) Cm (59-1x10.000) 2: TOF MS AP+ 7.59e+003



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
240.2694	240.2691	0.3	1.2	0.5	45.5	0.0	C16 H34 N


7.2622
6.7921
3.5871
3.5744
3.5616
3.3754
3.3621
3.3545
3.3330
2.7055
2.2082
1.9452
1.7487
1.6986
1.6672
1.6477
1.6267
1.3835
1.3646
1.3469
1.3277
1.3180
1.3121
1.3032
1.2933
1.2542
0.9023
0.8852
0.8676
-0.0006
BRUKER
Current Data Parameters
NAME N6C66-TMBS
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170602
Time 16.59 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.1 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9527
4.0058
4.0000
6.0539
3.0019
4.0352
4.0471
4.0787
12.3979
6.0629
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                 N6CXXC13
EXPNO                       8
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170707
Time                   13.35
INSTRUM                spect
PROBHD     5 mm QNP  1H
PULPROG                zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       778
DS                         4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                       256
DW                    19.900 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
d12                0.00002000 sec

============ CHANNEL f1 =============
NUC1                     13C
P1                    10.20 usec
PL1                    0.00 dB
SFO1              100.6237959 MHz

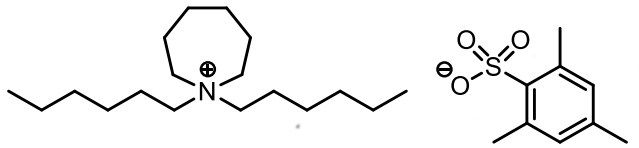
============ CHANNEL f2 =============
CPDPRG2              waltz16
NUC2                     1H
PCPD2                 90.00 usec
PL2                   -3.00 dB
PL12                  14.50 dB
PL13                  17.50 dB
SFO2              400.1326008 MHz

F2 - Processing parameters
SI                       32768
SF                100.6127792 MHz
WDW                         EM
SSB                          0
LB                     0.30 Hz
GB                           0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
F1P                  200.000 ppm
F1                 20122.55 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM               1006.12775 Hz/cm
```

3A (HR-ESI)

Elemental Composition Report

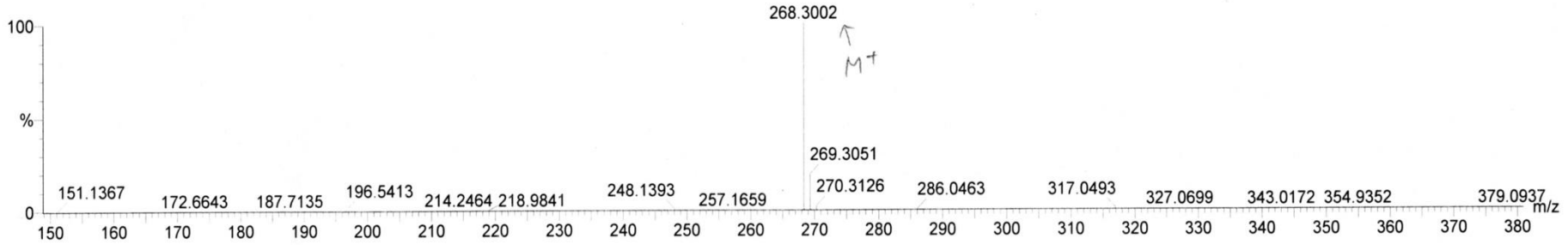


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

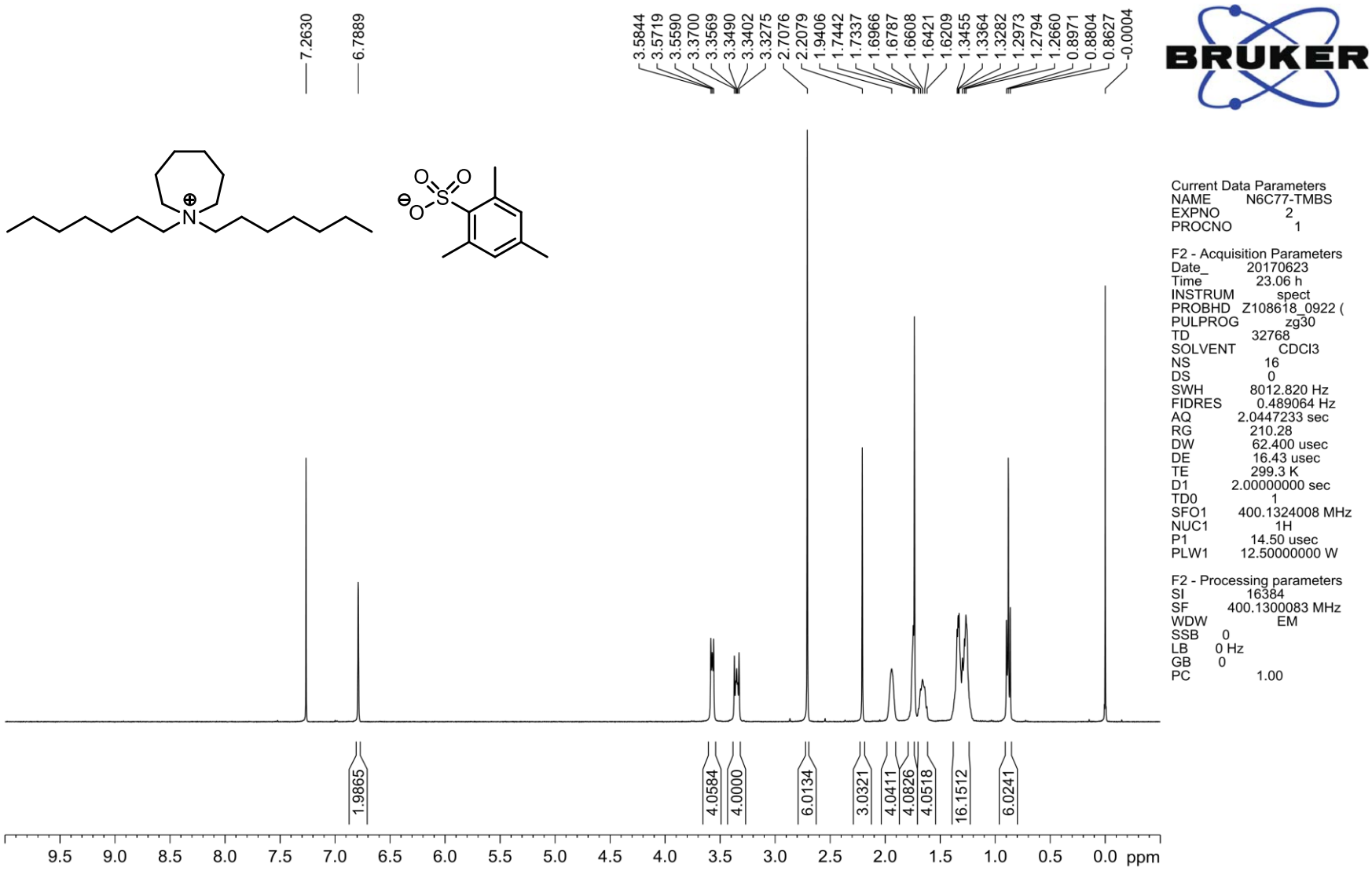
Monoisotopic Mass, Even Electron Ions
21 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 1-1000 H: 0-1000 N: 1-1

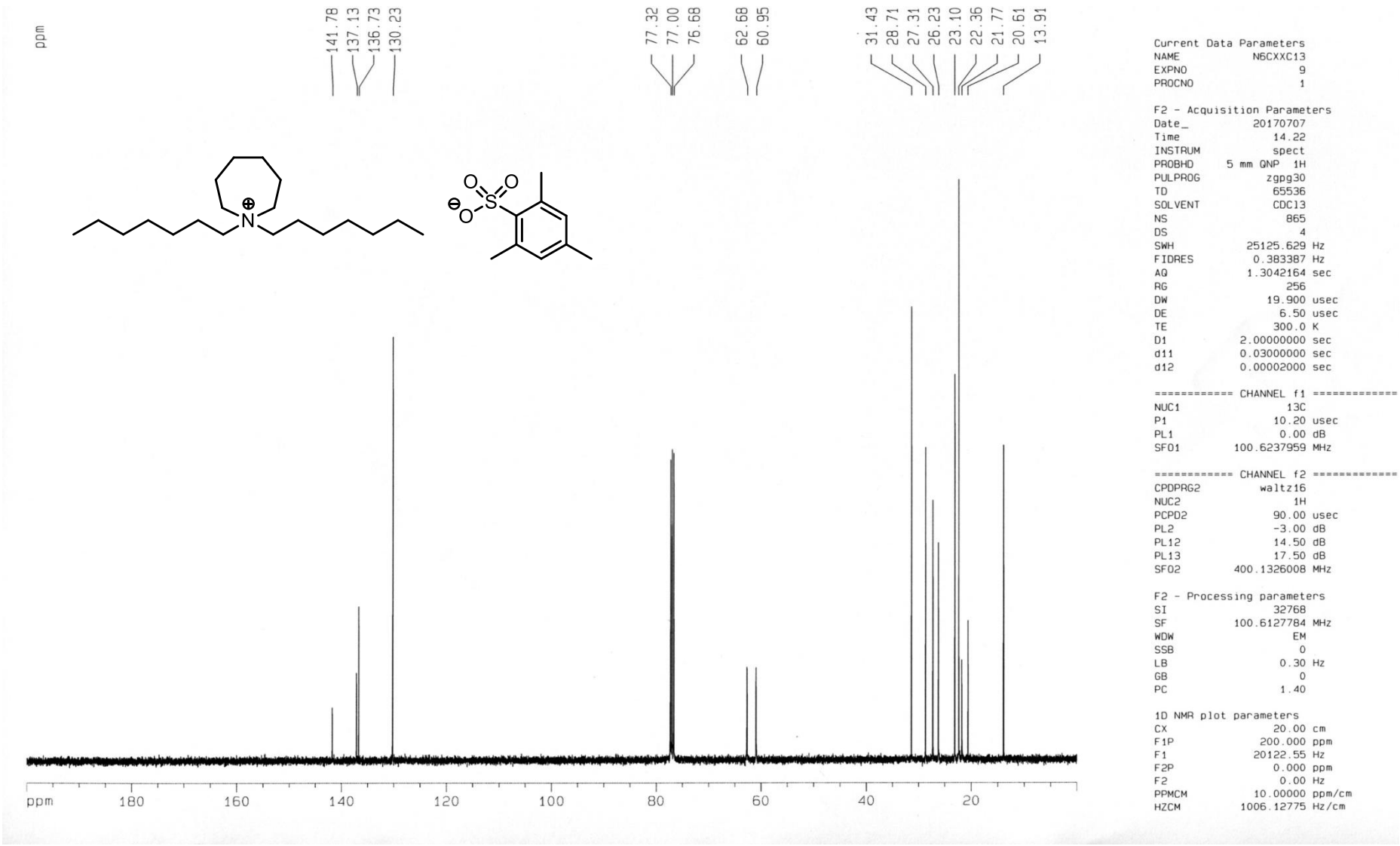
3A KE267 25-Jul-2017 12:32:41

0725_3A 165 (6.002) Cm (162:165-1x100.000) 1: TOF MS ES+ 1.25e+005



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
268.3002	268.3004	-0.2	-0.7	0.5	123.2	0.0	C18 H38 N


7.2630
6.7889
3.5844
3.5719
3.5590
3.3700
3.3569
3.3490
3.3402
3.3275
2.7076
2.2079
1.9406
1.7442
1.7337
1.6966
1.6787
1.6608
1.6421
1.6209
1.3455
1.3364
1.3282
1.2973
1.2794
1.2660
0.8971
0.8804
0.8627
-0.0004
BRUKER
Current Data Parameters
NAME N6C77-TMBS
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170623
Time 23.06 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 299.3 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
1.9865
4.0584
4.0000
6.0134
3.0321
4.0411
4.0826
4.0518
16.1512
6.0241
9.5 9.0 8.5 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 ppm




```
Current Data Parameters
NAME                 N6CXXC13
EXPNO                       9
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170707
Time                   14.22
INSTRUM                spect
PROBHD      5 mm QNP  1H
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      865
DS                        4
SWH                25125.629 Hz
FIDRES             0.383387 Hz
AQ                 1.3042164 sec
RG                      256
DW                   19.900 usec
DE                    6.50 usec
TE                   300.0 K
D1                2.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 =============
NUC1                    13C
P1                    10.20 usec
PL1                    0.00 dB
SFO1             100.6237959 MHz

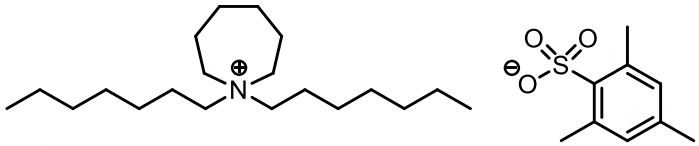
============ CHANNEL f2 =============
CPDPRG2             waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -3.00 dB
PL12                 14.50 dB
PL13                 17.50 dB
SFO2             400.1326008 MHz

F2 - Processing parameters
SI                      32768
SF               100.6127784 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.40

1D NMR plot parameters
CX                     20.00 cm
F1P                   200.000 ppm
F1                  20122.55 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM              1006.12775 Hz/cm
```

4A (HR-ESI)

Elemental Composition Report

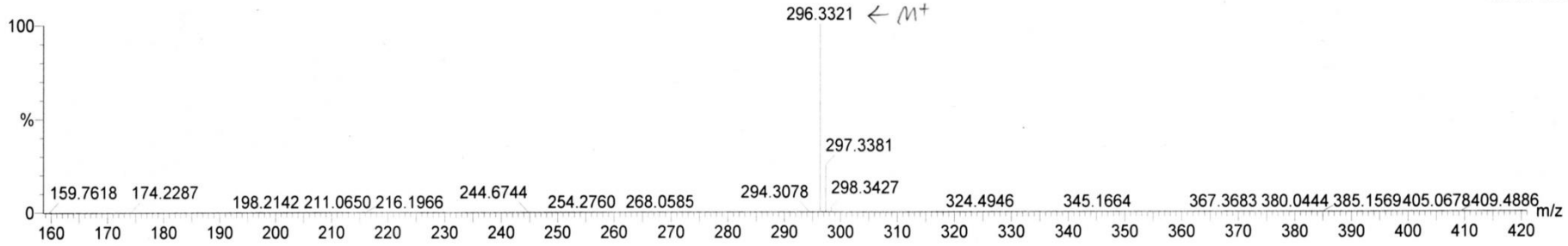


Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

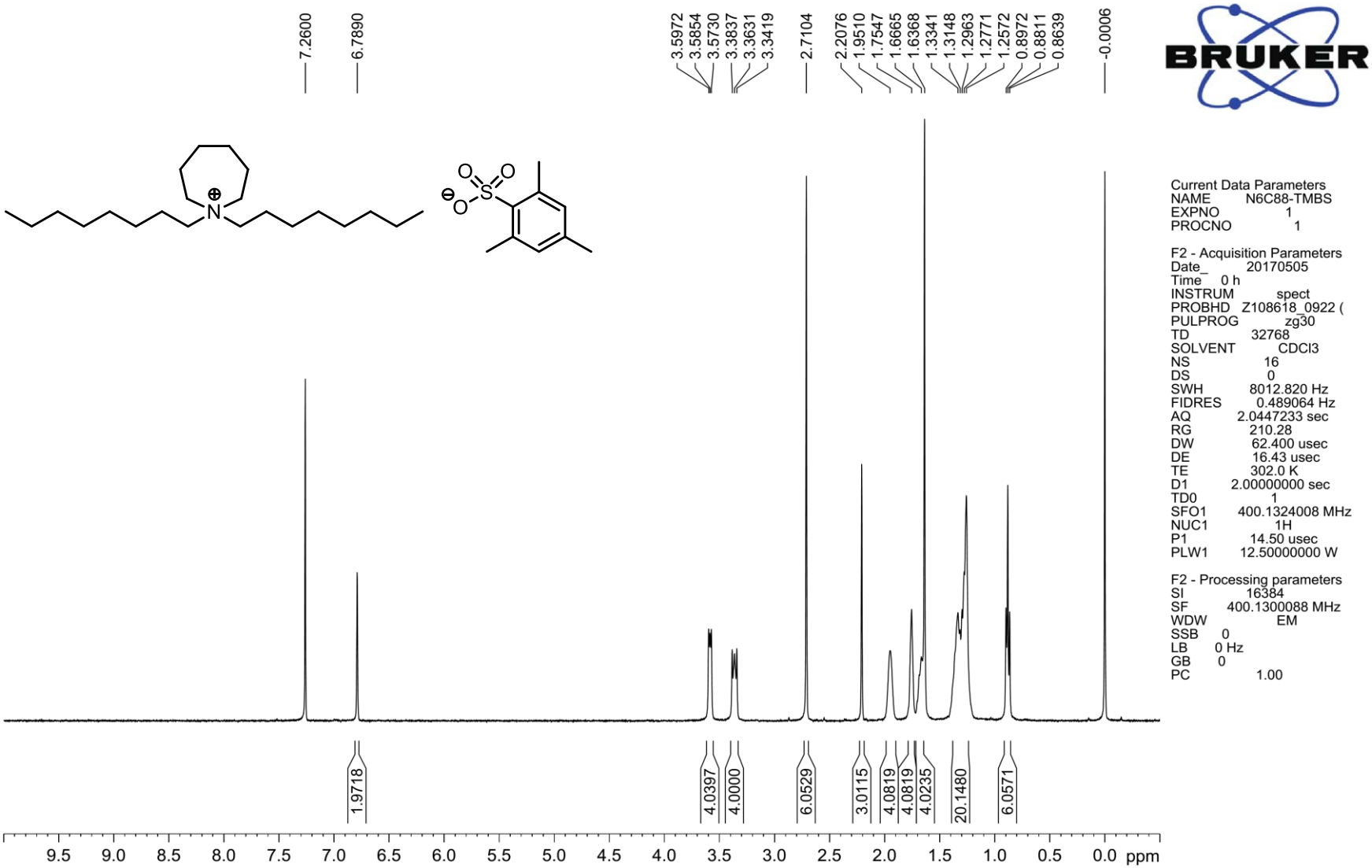
Monoisotopic Mass, Even Electron Ions
23 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 1-1000 H: 0-1000 N: 1-1

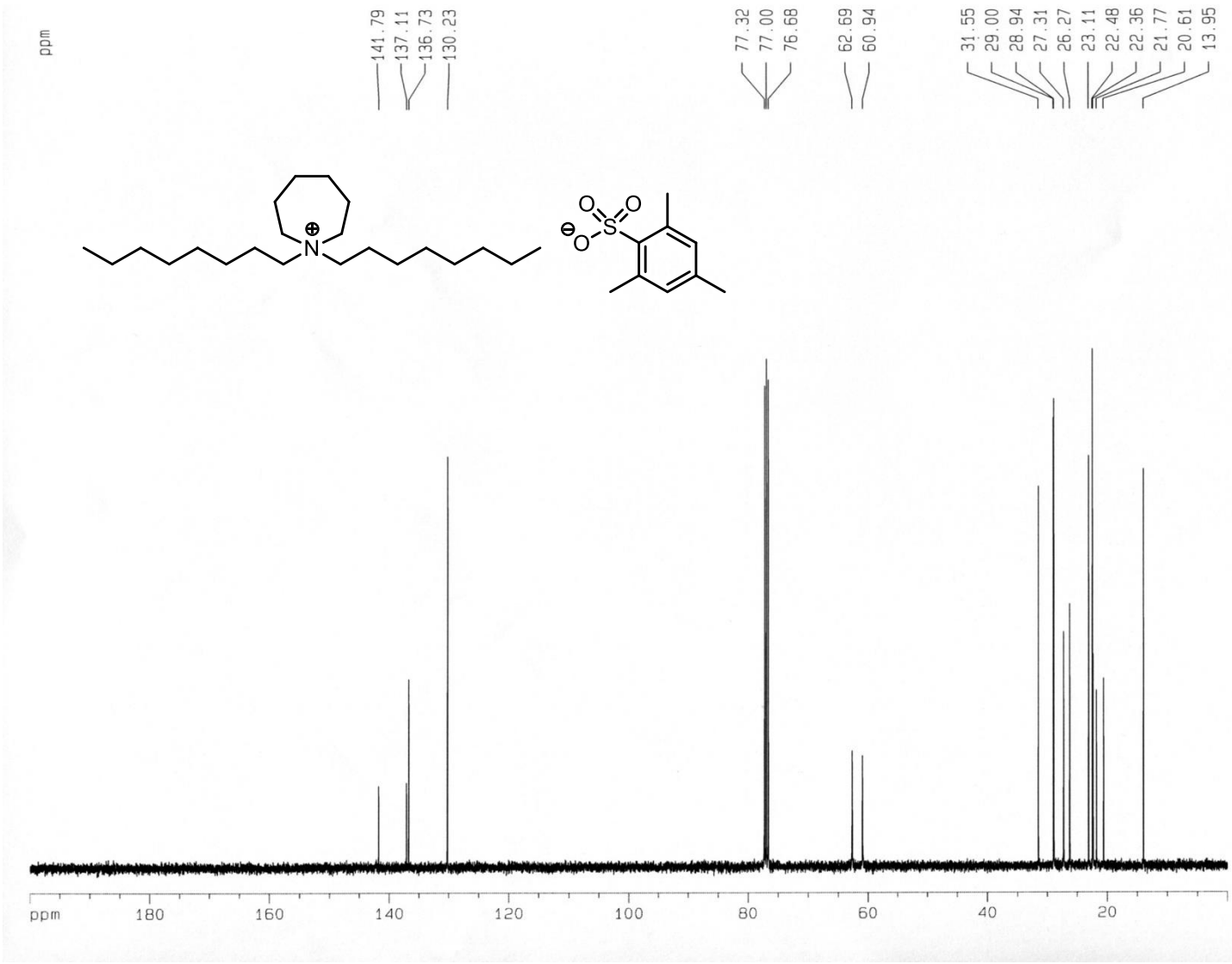
4A KE267 25-Jul-2017 13:45:39

0725_4A 90 (3.270) Cm (90-1x10.000) 1: TOF MS ES+ 6.91e+004



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
296.3321	296.3317	0.4	1.3	0.5	97.9	0.0	C20 H42 N


7.2600
6.7890
3.5972
3.5854
3.5730
3.3837
3.3631
3.3419
2.7104
2.2076
1.9510
1.7547
1.6665
1.6368
1.3341
1.3148
1.2963
1.2771
1.2572
0.8972
0.8811
0.8639
-0.0006
1.9718
4.0397
4.0000
6.0529
3.0115
4.0819
4.0819
4.0235
20.1480
6.0571
9.5 9.0 8.5 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 ppm
Current Data Parameters
NAME N6C88-TMBS
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170505
Time 0 h
INSTRUM spect
PROBHD Z108618_0922 (
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 210.28
DW 62.400 usec
DE 16.43 usec
TE 302.0 K
D1 2.00000000 sec
TD0 1
SFO1 400.1324008 MHz
NUC1 1H
P1 14.50 usec
PLW1 12.50000000 W
F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

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Current Data Parameters
NAME            N6CXXC13
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170707
Time               15.14
INSTRUM            spect
PROBHD     5 mm QNP  1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  584
DS                    4
SWH           25125.629 Hz
FIDRES            0.383387 Hz
AQ              1.3042164 sec
RG                  256
DW                19.900 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 =============
NUC1                13C
P1                10.20 usec
PL1                0.00 dB
SFO1          100.6237959 MHz

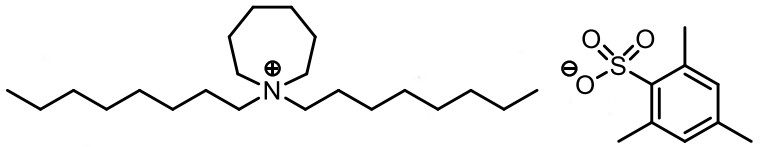
============ CHANNEL f2 =============
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PL2               -3.00 dB
PL12              14.50 dB
PL13              17.50 dB
SFO2          400.1326008 MHz

F2 - Processing parameters
SI                32768
SF           100.6127784 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              200.000 ppm
F1             20122.55 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1006.12775 Hz/cm
```

5A (HR-ESI)

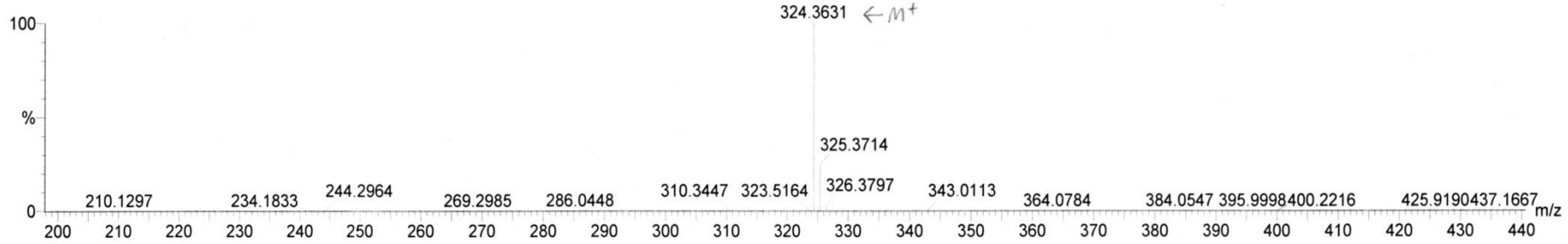
Elemental Composition Report



Single Mass Analysis
Tolerance = 50.0 PPM / DBE: min = -1000.0, max = 1000.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
25 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 1-1000 H: 0-1000 N: 1-1

5A KE267 25-Jul-2017 13:37:35
0725_5A 51 (1.844) Cm (51-1x10.000) 1: TOF MS ES+ 1.07e+005



Minimum:				-1000.0			
Maximum:		5.0	50.0	1000.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
324.3631	324.3630	0.1	0.3	0.5	84.3	0.0	C22 H46 N