Electronic Supplementary Material (ESI) for Materials Advances.

Exploiting α-benzylated 1,4-butanesultones to expedite the discovery of small-molecule, LCST-type sulfobetaine zwitterionic materials

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Measurements of critical temperature (T_c) for sulfobetaine zwitterionic materials in water

The critical temperatures were determined by the standard method of visual observation of the disappearance (homogeneous one-phase) and reappearance (heterogeneous two-phase) of the meniscus between the ZM-rich and water phases upon heating and cooling solutions of ZMs in water (1:3, w/w). A representative video (for **ZM 3c**) is provided in the Supporting Information-2 (ESI-2).

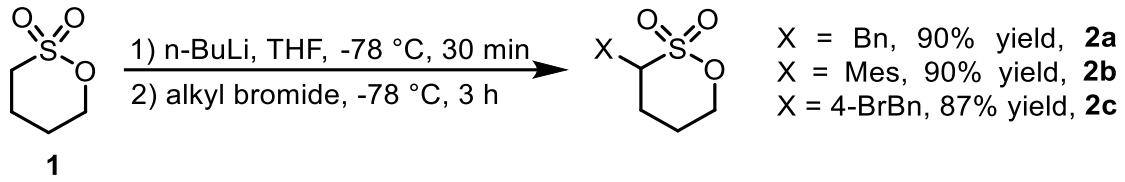
Thermogravimetric analysis (TGA) measurements

A PerkinElmer STA 6000 thermogravimetric analyzer (PerkinElmer, Waltham, MA, USA) was applied to investigate the thermal stability of zwitterionic sulfobetaine materials (**ZM 1c**, **ZM 2c**, **ZM 3c**, and **ZM 4c**) under nitrogen atmosphere. The thermal stability of ZMs was measured at a heating speed of 20 ^{o}C min^{-1} (up to 600 °C).

Measurements for protein enrichment and separation

As a proof-of-concept application, the method of temperature-dependent phase transition was used for the enrichment of diluted solutions containing Coomassie blue dye (37.5 μg/mL, 45 μM) and human hemoglobin protein (1 mg/mL, 15 μM), respectively. In this application, aqueous solutions (1:20, w/w) of **ZM 4c** in water were employed. UV-vis spectrophotometric measurements by a NanoDrop 2000 spectrophotometer (Thermo Scientific, Waltham, MA) were used for quantitatively determining degree of enrichment in Coomassie blue dye (λ_{max} at 597 nm) and human hemoglobin protein (Soret band at 406 nm), respectively, before and after temperature-triggered phase transitions. In this specific but preliminary applications, only minute amount of **ZM 4c** (4 mg in 80 μL water) was needed to achieve the sample enrichment.

1. Synthesis of α-benzylated 1,4-butanesultones (2a, 2b, and 2c)



To a stirred solution of 1,4-butane sultone (1.0 equiv) in THF (0.5 M) was slowly added *n*-BuLi (1.1 equiv) at -78 ℃. The reaction mixture was stirred at –78 ºC for 30 minutes. A solution of alkyl bromide (benzyl bromide, mesityl bromide, and 4-bromobenzyl bromide; 1.2 equiv in THF) was added at -78 ºC. The reaction mixture was stirred for 3 h, and quenched with water. The resulting mixture was extracted with ethyl acetate. The combined organic layers were concentrated and purified by column chromatography (silica gel; ethyl acetate/hexane = 1:4 ~ 1:2, v/v) to afford the desired products **2a-2c**.

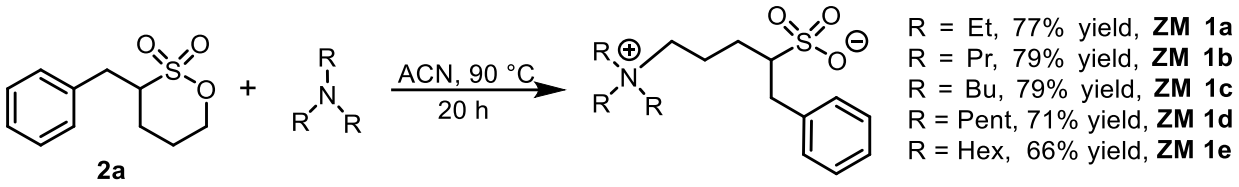
3-benzyl-1,2-oxathiane 2,2-dioxide (2a): 90% yield, white solid (m.p. 136 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.77-2.05 (m, $SOCH_2CH_2$ + $SCHCH_2CH_2$, 4 H), 2.73-2.79 (m, $SCHCH_2Ar$, 1H), 3.24-3.31 (m, SC*H*, 1H), 3.50-3.54 (m, $SCHCH_2Ar$, 1H), 4.44-4.61 (m, $SOCH_2CH_2$, 2H), 7.18-7.35 (m, aryl H, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.13, 27.56, 34.16, 60.81, 73.84, 127.39, 129.02, 129.40, 135.97; FAB-HRMS m/z $[M+H]^+$ calculated for $C_{11}H_{15}O_3S^+$ 227.0736, found 227.0736.

3-(3,5-dimethylbenzyl)-1,2-oxathiane 2,2-dioxide (2b): 90% yield, white solid (m.p. 86 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.77-2.08 (m, $SOCH_2CH_2$ + $SCHCH_2CH_2$, 4H), 2.30 (s, $ArCH_3$, 6H), 2.63-2.69 (m, $SCHCH_2Ar$, 1H), 3.21-3.29 (m, SC*H*, 1H), 3.42-3.47 (m, $SCHCH_2Ar$, 1H), 4.44-4.60 (m, $SOCH_2CH_2$, 2H), 6.80 (s, aryl H, 2H), 6.90 (s, aryl H, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.35, 24.13, 27.56, 33.90, 60.86, 73.83, 127.17, 128.94, 135.82, 138.56; FAB-HRMS m/z $[M+H]^+$ calculated for $C_{13}H_{19}O_3S^+$ 255.1049, found 255.1051.

3-(4-bromobenzyl)-1,2-oxathiane 2,2-dioxide (2c): 87% yield, white solid (m.p. 122 ℃); ^{1}H NMR

(400 MHz, $CDCl_3$) δ 1.79-2.05 (m, $SOCH_2CH_2$ + $SCHCH_2CH_2$, 4H), 2.73-2.77 (m, $SCHCH_2Ar$, 1H), 3.20-3.27 (m, SC*H*, 1H), 3.43-3.47 (m, $SCHCH_2Ar$, 1H), 4.45--4.61 (m, $SOCH_2CH_2$, 2H), 7.08 (d, *J* = 8.3 Hz, aryl H, 2H), 7.46 (d, *J* = 8.3 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.13, 27.66, 33.75, 60.53, 73.85, 121.40, 131.06, 132.14, 134.97; FAB-HRMS m/z $[M+H]^+$ calculated for $C_{11}H_{14}BrO_3S^+$ 304.9842 (100.0%), 306.9821 (97.3%), found 304.9855 (100.0%), 306.9827 (91.0%).

2. Synthesis of trialkylammonium α-benzyl-substituted sulfonate ZMs (ZM 1a-e, ZM 2a-e, and ZM 3a-e)



2a

To a 4 mL sample bottle containing **2a** (1.0 equiv), trialkyl amine (triethyl amine, tripropyl amine, tributyl amine, tripentyl amine, and trihexyl amine; 1.5 equiv) and acetonitrile (3.0 M). The reaction mixture was carried out at 90 ºC for 20 h. The reaction mixtures were purified by column chromatography (silica gel; dichloromethane/methanol) to afford the desired **ZM 1a-1e**.

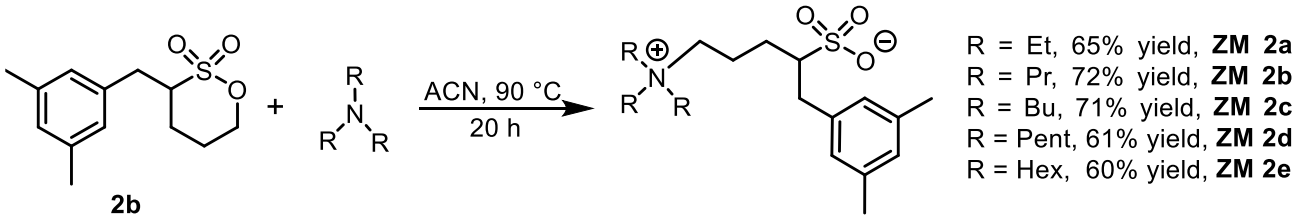
1-Phenyl-5-(triethylammonio)pentane-2-sulfonate (ZM 1a): 77% yield, white solid (m.p. 156 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.28 (t, *J* = 7.2 Hz, NCH_2CH_3 9H), 1.53-1.59 (m, $SCHCH_2CH_2$, 1H), 1.77-1.99 (m, $SCHCH_2CH_2$ + $SCHCH_2CH_2$, 3H), 2.68-2.74 (m, $SCHCH_2Ar$, 1H), 2.96-3.05 (m, NCH_2CH_2, 2H), 3.12-3.17 (m, SCH, 1H), 3.19-3.32 (m, NCH_2CH_3, 6H), 3.63-3.68 (m, $SCHCH_2Ar$, 1H), 7.17-7.30 (m, aryl H, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.80, 19.46, 26.43, 37.37, 53.13, 57.79, 19.94, 126.21, 128.62, 129.49, 140.33; ESI-HRMS m/z $[M+H]^+$ calculated for $C_{17}H_{30}NO_3S^+$ 328.1941, found 328.1938 ($[M+H]^+$), 350.1751 ($[M+Na]^+$), 366.1501 ($[M+K]^+$).

1-Phenyl-5-(tripropylammonio)pentane-2-sulfonate (ZM 1b): 79% yield, white solid (m.p. 176 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.01 (t, *J* = 7.2 Hz, $N(CH_2)_2CH_3$, 9H), 1.61-1.67 (m, $NCH_2CH_2CH_3$ + $SCHCH_2CH_2$, 7H), 1.84-1.85 (m, $SCHCH_2CH_2$ + $SCHCH_2CH_2$, 3H), 2.69-2.76 (m, $SCHCH_2Ar$, 1H), 2.99-3.26 (m, NCH_2 + $SCHCH_2Ar$, 9H), 3.67-3.71 (m, $SCHCH_2Ar$, 1H), 7.17-7.30 (m, aryl H, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 10.86, 15.59, 19.60, 26.37, 37.30, 59.59, 59.83, 60.46, 126.01, 128.43, 129.37, 140.34; ESI-HRMS m/z $[M+H]^+$ calculated for $C_{20}H_{36}NO_3S^+$ 370.2410, found 370.2405 ($[M+H]^+$), 392.2224 ($[M+Na]^+$), 408.1971 ($[M+K]^+$).

1-Phenyl-5-(tributylammonio)pentane-2-sulfonate (ZM 1c): 79% yield, white solid (m.p. 181 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, *J* = 7.3 Hz, $N(CH_2)_3CH_3$, 9H), 1.37-1.42 (m, $N(CH_2)_2CH_2CH_3$, 6H), 1.52-1.60 (m, $NCH_2CH_2CH_2CH_3$ + $SCHCH_2CH_2$, 7H), 1.85 (m, $SCHCH_2CH_2$ + $SCHCH_2CH_2$, 3H), 2.69-2.76 (m, $SCHCH_2Ar$, 1H), 2.99-3.25 (m, NCH_2 + $SCHCH_2Ar$, 9H), 3.68-3.72 (m, $SCHCH_2Ar$, 1H), 7.17-7.30 (m, aryl H, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.78, 19.72, 19.88, 24.03, 26.52, 37.46, 58.90, 59.80, 59.95, 126.09, 128.53, 129.50, 140.66; ESI-HRMS m/z $[M+H]^+$ calculated for $C_{23}H_{42}NO_3S^+$ 412.2880, found 412.2866 ($[M+H]^+$), 434.2697 ($[M+Na]^+$), 450.2439 ($[M+K]^+$).

1-Phenyl-5-(tripentylammonio)pentane-2-sulfonate (ZM 1d): 71% yield, white solid (m.p. 187 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.92 (t, *J* = 6.9 Hz, $N(CH_2)_4CH_3$, 9H), 1.25-1.42 (m, $NCH_2CH_2(CH_2)_2CH_3$, 12H), 1.56-1.60(m, $NCH_2CH_2(CH_2)_2CH_3$ + $SCHCH_2CH_2$, 7H), 1.81-1.87 (m, $SCHCH_2CH_2$ + $SCHCH_2CH_2$, 3H), 2.69-2.75 (m, $SCHCH_2Ar$, 1H), 2.98-3.30 (m, NCH_2 + $SCHCH_2Ar$, 9H), 3.70-3.74 (m, $SCHCH_2Ar$, 1H), 7.16-7.27 (m, aryl H, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.95, 19.74, 21.83, 22.32, 26.51, 28.56, 37.50, 59.09, 59.82, 59.94, 126.06, 128.51, 129.50, 140.71; ESI-HRMS m/z $[M+H]^+$ calculated for $C_{26}H_{48}NO_3S^+$ 454.3349, found 454.3350 ($[M+H]^+$), 476.3159 ($[M+Na]^+$).

1-Phenyl-5-(trihexylammonio)pentane-2-sulfonate (ZM 1e): 66% yield, white solid (m.p. 174 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.90 (t, *J* = 6.6 Hz, $N(CH_2)_5CH_3$, 9H), 1.32 (m, $NCH_2CH_2(CH_2)_3CH_3$, 18H), 1.57 (m, $NCH_2CH_2(CH_2)_3CH_3$ + $SCHCH_2CH_2$, 7H), 1.84-1.85 (m, $SCHCH_2CH_2$ + $SCHCH_2CH_2$, 3H), 2.69-2.76 (m, $SCHCH_2Ar$, 1H), 2.98-3.30 (m, NCH_2 + $SCHCH_2Ar$, 9H), 3.69-3.74 (m, $SCHCH_2Ar$, 1H), 7.17-7.29 (m, aryl H, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.00, 19.71, 22.09, 22.55, 26.19, 26.48, 31.30, 37.47, 59.13, 59.87, 59.94, 126.06, 128.51, 129.50, 140.70; ESI-HRMS m/z $[M+H]^+$ calculated for $C_{29}H_{54}NO_3S^+$ 496.3819, found 496.3810 ($[M+H]^+$), 518.3629 ($[M+Na]^+$).



To a 4 mL sample bottle containing **2b** (1.0 equiv), trialkyl amine (triethyl amine, tripropyl amine, tributyl amine, tripentyl amine, and trihexyl amine; 1.5 equiv) and acetonitrile (3.0 M). The reaction mixture was carried out at 90 ºC for 20 h. The reaction mixtures were purified by column chromatography (silica gel; dichloromethane/methanol) to afford the desired **ZM 2a-2e**.

1-(3,5-Dimethylphenyl)-5-(triethylammonio)pentane-2-sulfonate (ZM 2a): 65% yield, white solid (m.p. 215 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.29 (t, *J* = 7.2 Hz, NCH_2CH_3, 9H), 1.55-1.82 (m, $SCHCH_2CH_2$, 2H), 1.88-2.02 (m, $SCHCH_2CH_2$, 2H), 2.27 (s, $ArCH_3$, 6H), 2.57-2.63 (m, $SCHCH_2Ar$, 1H), 2.93-3.04 (m, NCH_2CH_2, 2H), 3.12-3.19 (m, SCH, 1H), 3.27-3.33 (m, NCH_2CH_3, 6H), 3.54-3.58 (m, $SCHCH_2Ar$, 1H), 6.82 (s, aryl H, 1H), 6.85 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.79, 19.38, 21.41, 26.40, 37.10, 53.19, 57.79, 60.08, 127.26, 127.84, 138.01, 140.10; ESI-HRMS m/z $[M+H]^+$ calculated for $C_{19}H_{34}NO_3S^+$ 356.2254, found 356.2250 ($[M+H]^+$), 378.2068 ($[M+Na]^+$), 394.1811 ($[M+K]^+$).

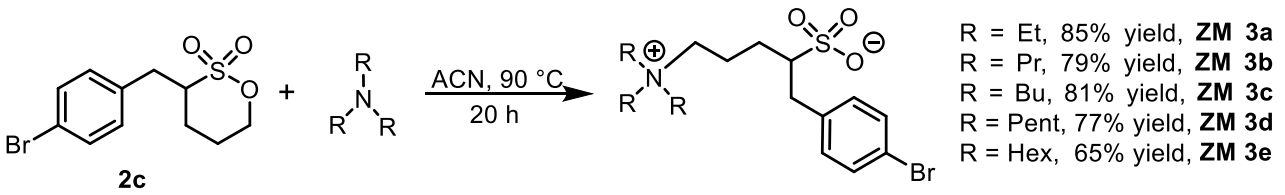
1-(3,5-Dimethylphenyl)-5-(tripropylammonio)pentane-2-sulfonate (ZM 2b): 72% yield, white solid (m.p. 215 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.01 (t, *J* = 7.2 Hz, $N(CH_2)_2CH_3$, 9H), 1.61-1.68 (m, $NCH_2CH_2CH_3$ + $SCHCH_2CH_2$, 7H), 1.80-1.93 (m, $SCHCH_2CH_2$ + $SCHCH_2CH_2$, 3H), 2.28 (s, $ArCH_3$, 6H), 2.57-2.64 (m, $SCHCH_2Ar$, 1H) , 2.92-2.97 (m, SCH, 1H), 3.09-3.28 (m, NCH_2, 8H), 3.59-3.63 (m, $SCHCH_2Ar$, 1H), 6.82 (s, aryl H, 1H), 6.86 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.01, 15.74, 19.74, 21.43, 26.52, 37.29, 59.80, 60.07, 60.59, 127.29, 127.74, 137.93, 140.42; ESI-HRMS m/z $[M+H]^+$ calculated for $C_{22}H_{40}NO_3S^+$ 398.2723, found 398.2715 ($[M+H]^+$), 420.2536 ($[M+Na]^+$).

1-(3,5-Dimethylphenyl)-5-(tributylammonio)pentane-2-sulfonate (ZM 2c): 71% yield, white solid (m.p. 216 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, *J* = 7.3 Hz, $N(CH_2)_3CH_3$, 9H), 1.36-1.45 (m, $N(CH_2)_2CH_2CH_3$, 6H), 1.54-1.60 (m, $NCH_2CH_2CH_2CH_3$ + $SCHCH_2CH_2$, 7H), 1.77-1.86 (m, $SCHCH_2CH_2$, 1H), 1.91-1.97 (m, $SCHCH_2CH_2$, 2H), 2.27 (s, $ArCH_3$, 6H), 2.57-2.64 (m, $SCHCH_2Ar$, 1H) , 2.94-2.99 (m, SC*H*, 1H), 3.10-3.30 (m, NCH_2, 8H), 3.61-3.66 (m, $SCHCH_2Ar$, 1H), 6.82 (s, aryl H, 1H), 6.86 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.78, 19.69, 19.90, 21.43, 24.05, 26.46, 37.32, 58.93, 59.95, 60.01, 127.33, 127.69, 137.89, 140.56; ESI-HRMS m/z $[M+H]^+$ calculated for $C_{25}H_{46}NO_3S^+$ 440.3193, found 440.3193 ($[M+H]^+$), 462.3011 ($[M+Na]^+$), 478.2752 ($[M+K]^+$).

1-(3,5-Dimethylphenyl)-5-(tripentylammonio)pentane-2-sulfonate (ZM 2d): 61% yield, white solid (m.p. 174 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.92 (t, *J* = 6.9 Hz, $N(CH_2)_4CH_3$, 9H), 1.29-1.41 (m, $NCH_2CH_2(CH_2)_2CH_3$, 12H), 1.58-1.62 (m, $NCH_2CH_2(CH_2)_2CH_3$ + $SCHCH_2CH_2$, 7H), 1.79-1.85 (m, $SCHCH_2CH_2$, 1H), 1.91-1.97 (m, $SCHCH_2CH_2$, 2H), 2.27 (s, $ArCH_3$, 6H), 2.57-2.64 (m, $SCHCH_2Ar$, 1H) , 2.94-2.99 (m, SC*H*, 1H), 3.07-3.33 (m, NCH_2, 8H), 3.63-3.67 (m, $SCHCH_2Ar$, 1H), 6.82 (s, aryl H, 1H), 6.86 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.95, 19.69, 21.43, 21.84, 22.32, 26.44, 28.57, 37.31, 59.11, 59.95, 60.01, 127.32, 127.67, 137.87, 140.56; ESI-HRMS m/z $[M+H]^+$ calculated for $C_{28}H_{52}NO_3S^+$ 482.3662, found 482.3671 ($[M+H]^+$), 504.3490 ($[M+Na]^+$), 520.3229 ($[M+K]^+$).

1-(3,5-Dimethylphenyl)-5-(trihexylammonio)pentane-2-sulfonate (ZM 2e): 60% yield, white solid (m.p. 155 °C); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.90 (t, *J* = 6.7 Hz, $N(CH_2)_5CH_3$, 9H), 1.33 (m, $NCH_2CH_2(CH_2)_3CH_3$, 18H), 1.59 (m, $NCH_2CH_2(CH_2)_3CH_3$ + $SCHCH_2CH_2$, 7H), 1.77-1.81 (m, $SCHCH_2CH_2$, 1H), 1.92-1.94 (m, $SCHCH_2CH_2$, 2H), 2.27 (s, $ArCH_3$, 6H), 2.57-2.64 (m, $SCHCH_2Ar$, 1H) , 2.94-2.99 (m, SC*H*, 1H), 3.07-3.29 (m, NCH_2, 8H), 3.62-3.66 (m, $SCHCH_2Ar$, 1H), 6.82 (s, aryl H, 1H), 6.86 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.98, 19.62, 21.42, 22.09, 22.54, 26.21, 26.36, 31.29, 37.31, 59.16, 59.96, 60.02, 127.32, 127.66, 137.86, 140.54; ESI-HRMS m/z $[M+H]^+$

calculated for $C_{31}H_{58}NO_3S^+$ 524.4132, found 524.4125 ([M+H]$^+$), 546.3943 ([M+Na]$^+$).



To a 4 mL sample bottle containing **2c** (1.0 equiv), trialkyl amine (triethyl amine, tripropyl amine, tributyl amine, tripentyl amine, and trihexyl amine; 1.5 equiv) and acetonitrile (3.0 M). The reaction was carried out at 90 ºC for 20 h. The reaction mixtures were purified by column chromatography (silica gel; dichloromethane/methanol) to afford the desired **ZM 3a-3e**.

1-(4-Bromophenyl)-5-(triethylammonio)pentane-2-sulfonate (ZM 3a): 85% yield, white solid (m.p. 224 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.29 (t, *J* = 7.2 Hz, NCH_2CH_3 9H), 1.52-1.53 (m, $SCHCH_2CH_2$, 1H), 1.78-1.97 (m, $SCHCH_2CH_2$ + $SCHCH_2CH_2$, 3H), 2.66-2.72 (m, $SCHCH_2Ar$, 1H), 2.94-3.07 (m, NCH_2CH_2, 2H), 3.14-3.33 (m, SCH + NCH_2CH_3, 7H), 3.54-3.59 (m, $SCHCH_2Ar$, 1H), 7.16 (d, *J* = 4.1 Hz, aryl H, 2H), 7.40 (d, *J* = 4.1 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.67, 19.37, 26.39, 36.72, 53.05, 57.59, 59.60, 119.89, 131.17, 131.54, 139.18; ESI-HRMS m/z [M+H]$^+$ calculated for $C_{17}H_{29}BrNO_3S^+$ 406.1046, 408.1026, found 406.1041 ([M+H]$^+$), 408.1025 ([M+H]$^+$), 428.0862 ([M+Na]$^+$) , 430.0841 ([M+Na]$^+$).

1-(4-Bromophenyl)-5-(tripropylammonio)pentane-2-sulfonate (ZM 3b): 79% yield, white solid (m.p. 226 ℃); ^{1}H NMR (400 MHz, D_2O) δ 0.90 (t, *J* = 7.2 Hz, $N(CH_2)_2CH_3$ 9H), 1.41-1.86 (m, $SCHCH_2CH_2$ + $SCHCH_2CH_2$ + $NCH_2CH_2CH_3$, 10H), 2.68-2.74 (m, $SCHCH_2Ar$, 1H), 2.89-3.10 (m, SCH + NCH_2, 9H), 3.34-339 (m, $SCHCH_2Ar$, 1H), 7.28 (d, *J* = 4.1 Hz, aryl H, 2H), 7.56 (d, *J* = 4.1 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, D_2O) δ 9.85, 14.78, 19.40, 25.79, 35.59, 57.68, 59.93, 60.42, 120.07, 131.24, 131.73, 137.68; ESI-HRMS m/z [M+H]$^+$ calculated for $C_{20}H_{35}BrNO_3S^+$ 448.1516,

450.1495, found 448.1509 ([M+H]$^+$), 450.1491 ([M+H]$^+$), 470.1328 ([M+Na]$^+$), 472.1307 ([M+Na]$^+$).

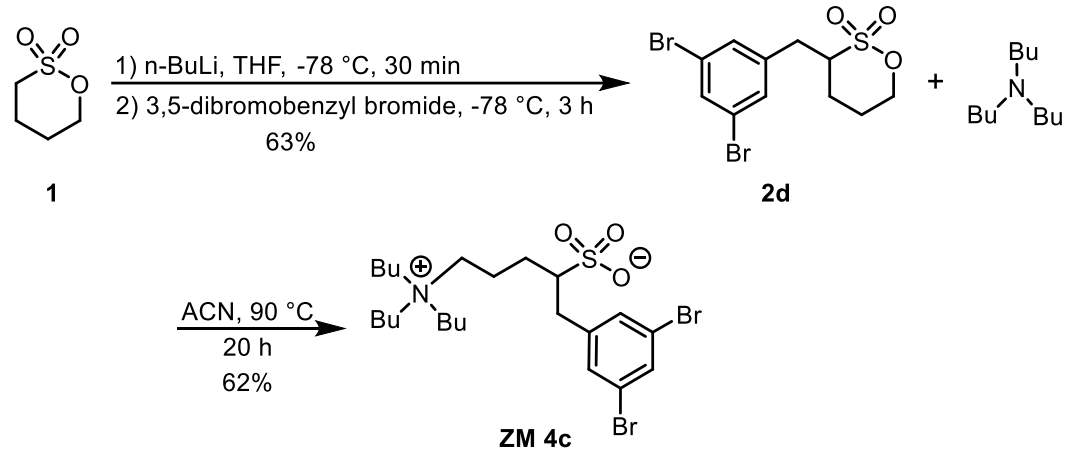
1-(4-Bromophenyl)-5-(tributylammonio)pentane-2-sulfonate (ZM 3c): 81% yield, white solid (m.p. 189 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, *J* = 7.3 Hz, $N(CH_2)_3C\mathit{H}_3$ 9H), 1.36-1.45 (m, $NCH_2CH_2C\mathit{H}_2CH_3$, 6H), 1.53-1.59 (m, $SCHC\mathit{H}_2CH_2$ + $NCH_2C\mathit{H}_2CH_2CH_3$, 7H), 1.87-1.92 (m, $SCHC\mathit{H}_2CH_2$ + $SCHCH_2C\mathit{H}_2$, 3H), 2.67-2.73 (m, $SCHC\mathit{H}_2Ar$, 1H), 2.95-3.30 (m, $SC\mathit{H}$ + $NC\mathit{H}_2$, 9H), 3.61-3.65 (m, $SCHC\mathit{H}_2Ar$, 1H), 7.15 (d, *J* = 4.1 Hz, aryl H, 2H), 7.40 (d, *J* = 4.1 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.65, 19.58, 19.74, 23.91, 26.39, 36.78, 58.80, 59.56, 59.62, 119.78, 131.15, 131.46, 139.45; ESI-HRMS m/z [M+H]$^+$ calculated for $C_{23}H_{41}BrNO_3S^+$ 490.1985, 492.1965, found 490.1983 ([M+H]$^+$), 492.1960 ([M+H]$^+$), 512.1798 ([M+Na]$^+$) , 514.1785 ([M+Na]$^+$).

1-(4-Bromophenyl)-5-(tripentylammonio)pentane-2-sulfonate (ZM 3d): 77% yield, white solid (m.p. 184 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.92 (t, *J* = 6.9 Hz, $N(CH_2)_4C\mathit{H}_3$ 9H), 1.31-1.39 (m, $NCH_2CH_2(C\mathit{H}_2)_2CH_3$, 12H), 1.55-1.59 (m, $SCHC\mathit{H}_2CH_2$ + $NCH_2C\mathit{H}_2(CH_2)_2CH_3$, 7H), 1.81-1.89 (m, $SCHC\mathit{H}_2CH_2$ + $SCHCH_2C\mathit{H}_2$, 3H), 2.67-2.73 (m, $SCHC\mathit{H}_2Ar$, 1H), 2.94-3.31 (m, $SC\mathit{H}$ + $NC\mathit{H}_2$, 9H), 3.61-3.66 (m, $SCHC\mathit{H}_2Ar$, 1H), 7.15 (d, *J* = 4.1 Hz, aryl H, 2H), 7.39 (d, *J* = 4.1 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.95, 19.73, 21.85, 22.33, 26.53, 28.56, 36.99, 59.15, 59.67, 59.82, 119.89, 131.28, 131.59, 139.68; ESI-HRMS m/z [M+H]$^+$ calculated for $C_{26}H_{47}BrNO_3S^+$ 532.2455, 534.2434, found 532.2462 ([M+H]$^+$), 534.2440 ([M+H]$^+$), 554.2283 ([M+Na]$^+$), 556.2262 ([M+Na]$^+$).

1-(4-Bromophenyl)-5-(trihexylammonio)pentane-2-sulfonate (ZM 3e): 65% yield, white solid (m.p. 167 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.90 (t, *J* = 6.7 Hz, $N(CH_2)_5C\mathit{H}_3$ 9H), 1.33 (m, $NCH_2CH_2(C\mathit{H}_2)_3CH_3$, 18H), 1.58 (m, $SCHC\mathit{H}_2CH_2$ + $NCH_2C\mathit{H}_2(CH_2)_3CH_3$, 7H), 1.75-1.98 (m, $SCHC\mathit{H}_2CH_2$ + $SCHCH_2C\mathit{H}_2$, 3H), 2.66-2.73 (m, $SCHC\mathit{H}_2Ar$, 1H), 2.94-3.35 (m, $SC\mathit{H}$ + $NC\mathit{H}_2$, 9H), 3.63-3.67 (m, $SCHC\mathit{H}_2Ar$, 1H), 7.14 (d, *J* = 4.1 Hz, aryl H, 2H), 7.39 (d, *J* = 4.1 Hz, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.00, 19.69, 22.11, 22.55, 26.20, 26.51, 31.31, 36.97, 59.17, 59.63, 59.84, 119.87, 131.28, 131.58, 139.67; ESI-HRMS m/z [M+H]$^+$ calculated for $C_{29}H_{53}BrNO_3S^+$ 574.2924,

576.2904, found 574.2920 ([M+H]$^+$), 576.2912 ([M+H]$^+$), 596.2742 ([M+Na]$^+$), 598.2723 ([M+Na]$^+$).

3. Synthesis of tributylammonium α-(3,5-dibromobenzyl)-substituted sulfonate ZM 4c



To a stirred solution of 1,4-butane sultone (0.35 mL, 1.0 equiv) in THF (0.5 M) was slowly added *n*-BuLi (1.1 equiv) at -78 ℃. The reaction mixture was stirred at –78 ºC for 30 minutes. A solution of 3,5-dibromobenzyl bromide (1.2 equiv in THF) was added at -78 ºC. The reaction mixture was carried out for 3 h, and quenched with water. The resulting reaction mixture was extracted with ethyl acetate. The combined organic layers were concentrated and purified by column chromatography (silica gel; ethyl acetate/hexane = 1:10 ~ 1:2, v/v) to afford the desired **2d.**

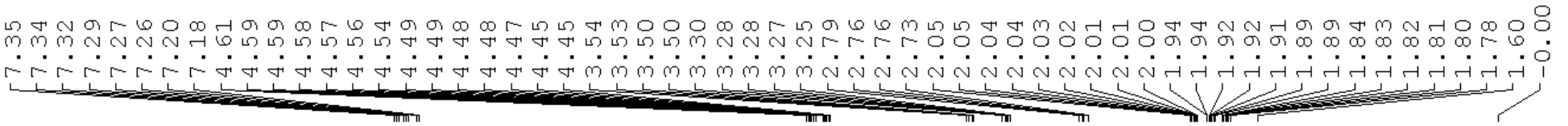
3-(3,5-Dibromobenzyl)-1,2-oxathiane 2,2-dioxide (2d): 63% yield, colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ1.83-2.07 (m, $SOCH_2CH_2$ + $SCHCH_2CH_2$, 4H), 2.68-2.75 (m, $SCHCH_2Ar$, 1H), 3.21-3.28 (m, SCH, 1H), 3.41-3.46 (m, $SCHCH_2Ar$, 1H), 4.46-4.61 (m, $SOCH_2CH_2$, 2H), 7.30 (d, *J* = 1.6 Hz, aryl H, 1H), 7.30 (d, *J* = 1.6 Hz, aryl H, 1H), 7.59 (dd, *J* = 1.6, 1.6 Hz, aryl H, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.04, 27.77, 33.69, 60.18, 73.94, 123.44, 131.14, 133.21, 139.96; FAB-HRMS m/z [M+H]$^+$ calculated for $C_{11}H_{13}Br_2O_3S^+$ 384.8926 (100.0%), 382.8947 (51.4%), 386.8906 (48.6%), found 384.8943 (87.9%), 382.8953 (41.6%), 386.8925 (50.3%).

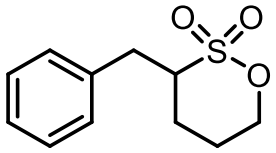
To a 4 mL sample bottle containing **2d** (0.27 g, 1.0 equiv), tributyl amine (1.5 equiv) and acetonitrile (3.0 M), The reaction mixture was carried out at 90 ºC for 20 h. The crude reaction products were

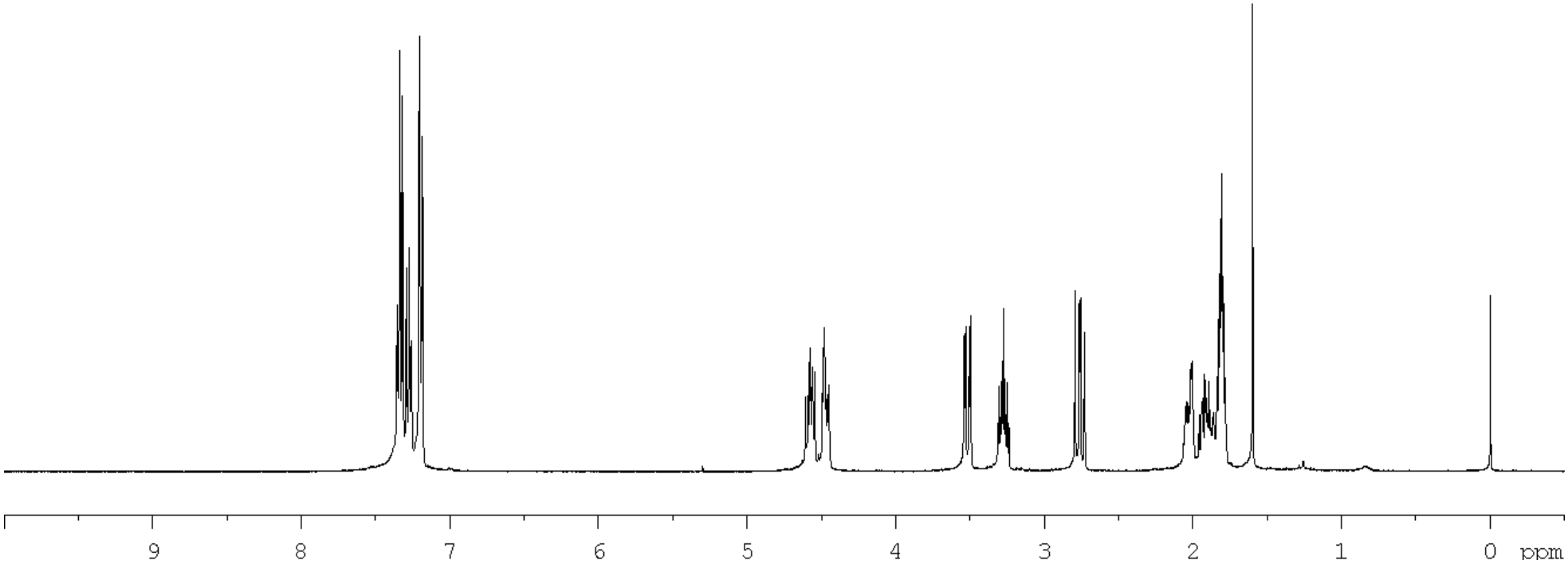
purified by column chromatography (silica gel; dichloromethane/methanol = 30:1 ~ 6:1, v/v) to afford the desired product **ZM 4c**.

1-(3,5-Dibromophenyl)-5-(tributylammonio)pentane-2-sulfonate (ZM 4c): 62% yield, white solid (m.p. 191 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.00 (t, *J* = 7.3 Hz, $N(CH_2)_3CH_3$, 9H), 1.38-1.47 (m, $N(CH_2)_2CH_2CH_3$, 6H), 1.57-1.63 (m, $NCH_2CH_2CH_2CH_3$ + $SCHCH_2CH_2$, 7H), 1.79-1.85 (m, $SCHCH_2CH_2$, 1H), 1.97-1.99 (m, $SCHCH_2CH_2$, 2H), 2.63-2.69 (m, $SCHCH_2Ar$, 1H) , 2.90-3.34 (m, SCH + NCH_2, 9H), 3.61-3.65 (m, $SCHCH_2Ar$, 1H), 7.35 (s, aryl H, 1H), 7.50 (s, aryl H, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.78, 19.62, 19.90, 24.06, 26.54, 37.15, 59.03, 59.44, 59.82, 122.98, 131.25, 131.87, 144.89; ESI-HRMS m/z $[M+H]^+$ calculated for $C_{23}H_{40}Br_2NO_3S^+$ 570.1070, 568.1090, 572.1049, found 570.1062 ($[M+H]^+$), 568.1082 ($[M+H]^+$), 572.1040 ($[M+H]^+$), 592.0879 ($[M+Na]^+$), 590.0901 ($[M+Na]^+$), 594.0859 ($[M+Na]^+$).

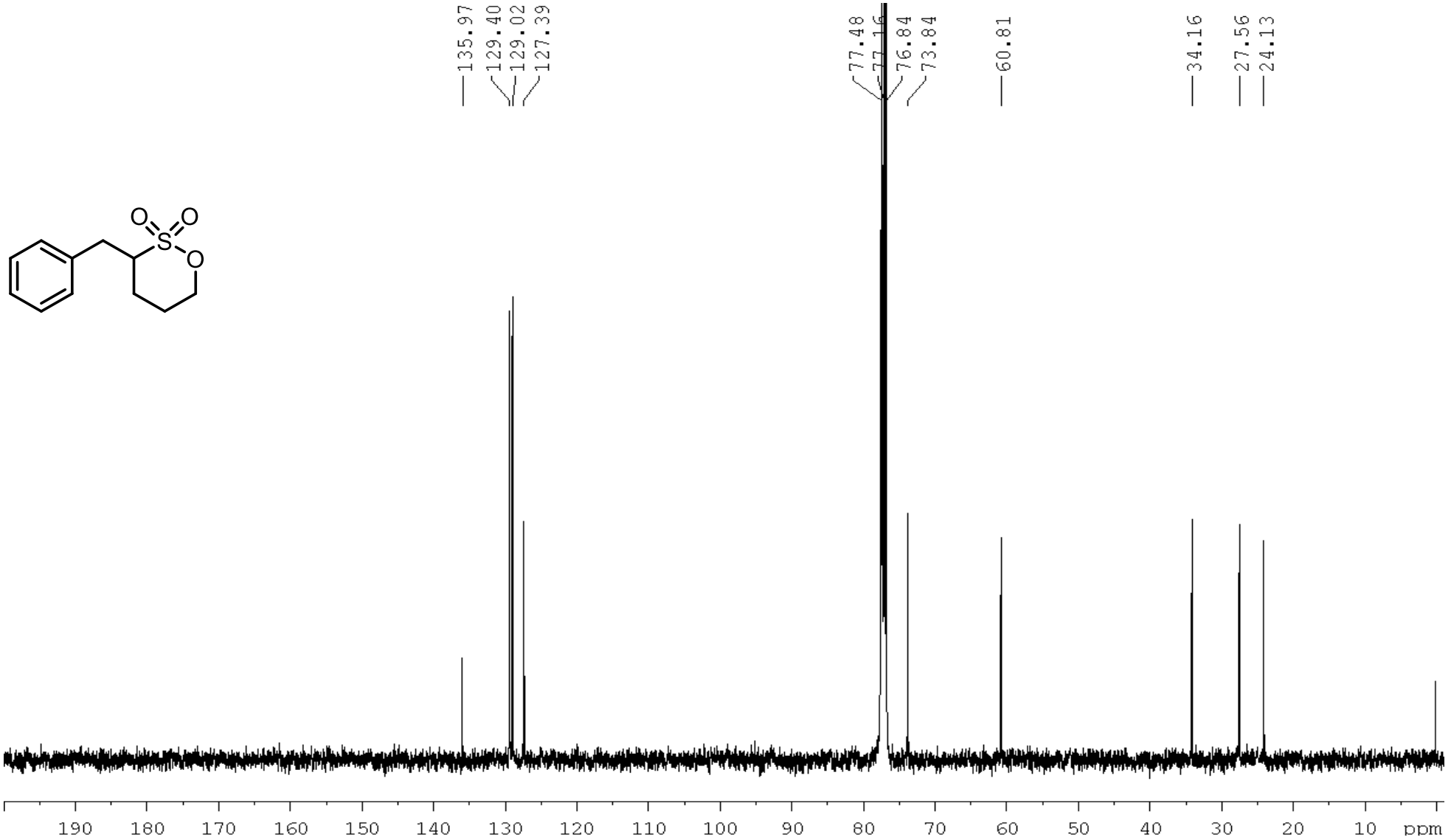
^{1}H NMR spectrum of **2a**





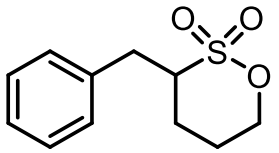


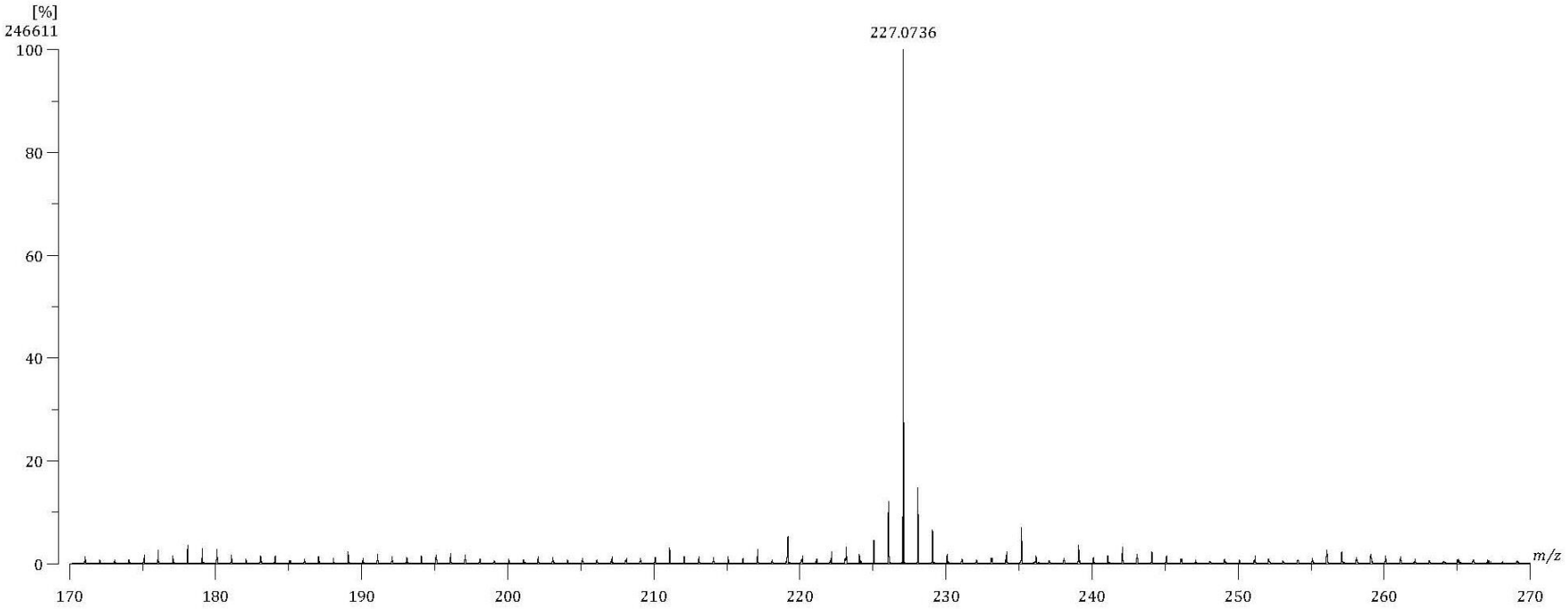
^{13}C NMR spectrum of **2a**



HRMS spectrum of **2a**

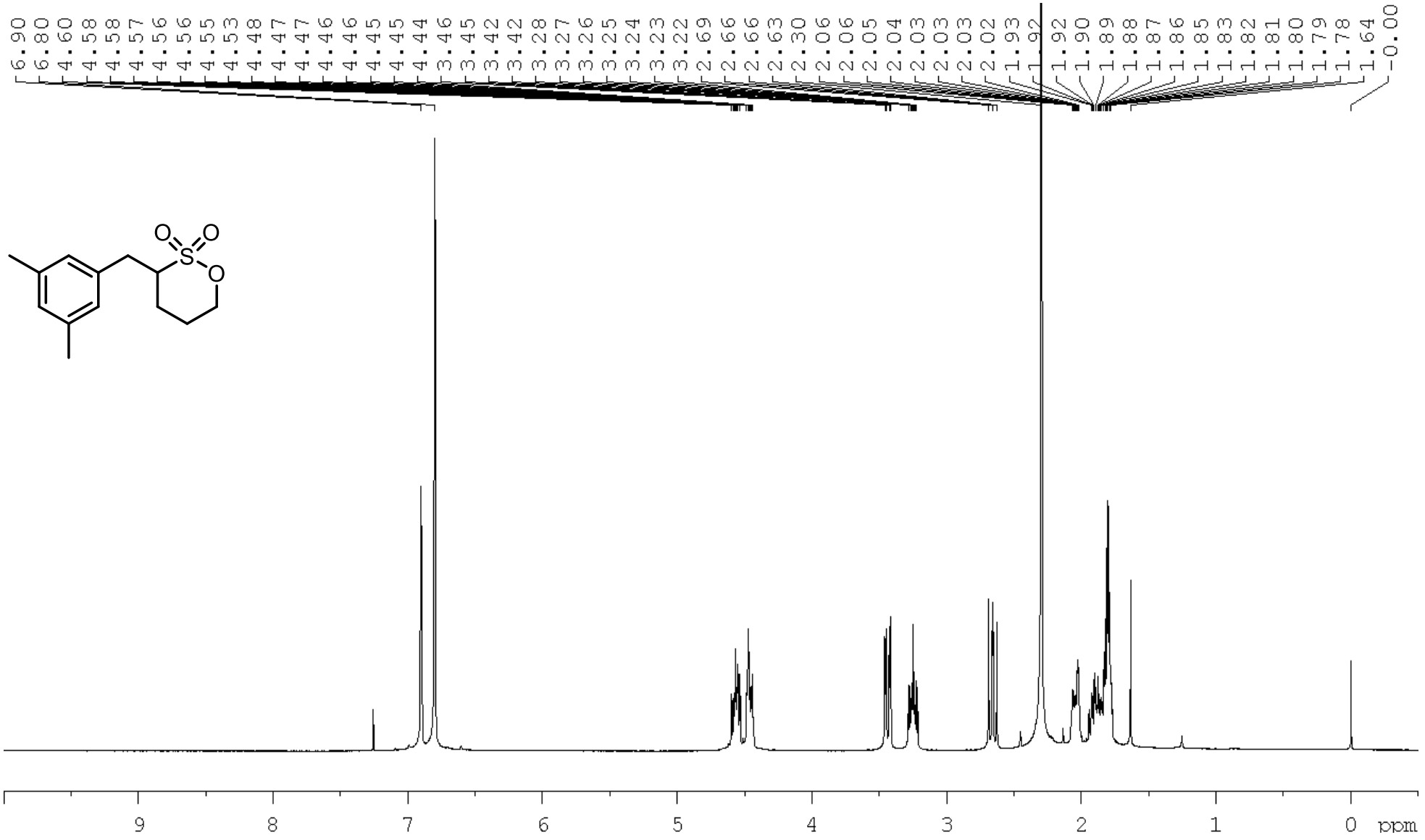
[Mass Spectrum]
Data : 20220819_BnC4-HR-001 Date : 19-Aug-2022 17:21
Sample : BnC4
Note : NBA
Ion Mode : FAB+
RT : 0.41 min Scan# : 5
Elements : C 1000/0, H 1000/0, O 3/3, S 1/1
Mass Tolerance : 50mmu
Unsaturation (U.S.) : -0.5 - 1000.0



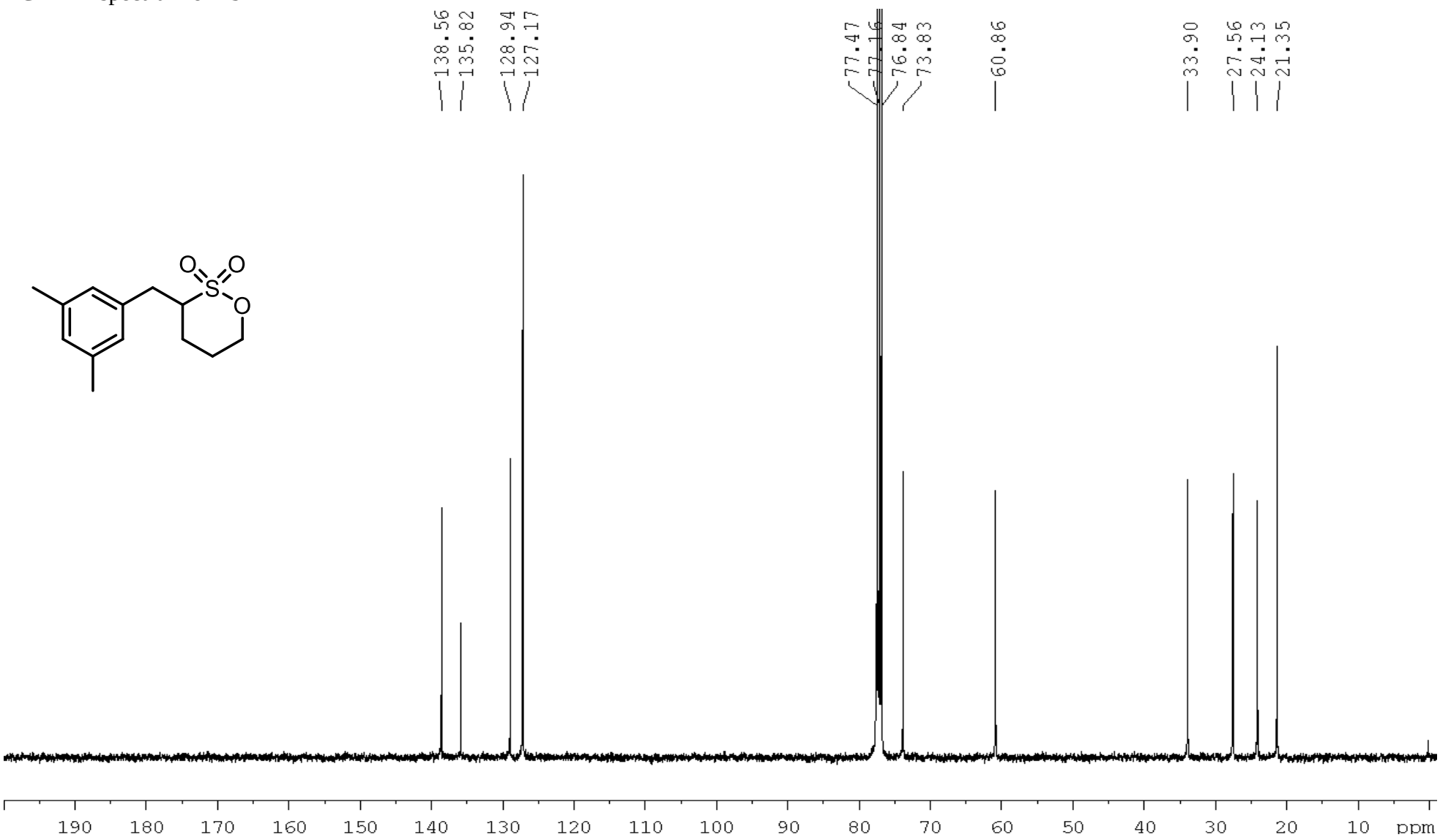


	Observed m/z	Int%					
	227.0736	100.00					
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	O	S
1	227.0742	-2.6 / -0.6	5.5	11	15	3	1

^{1}H NMR spectrum of **2b**

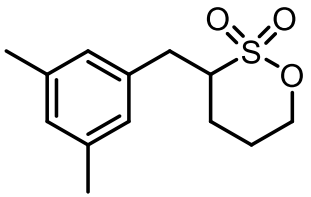


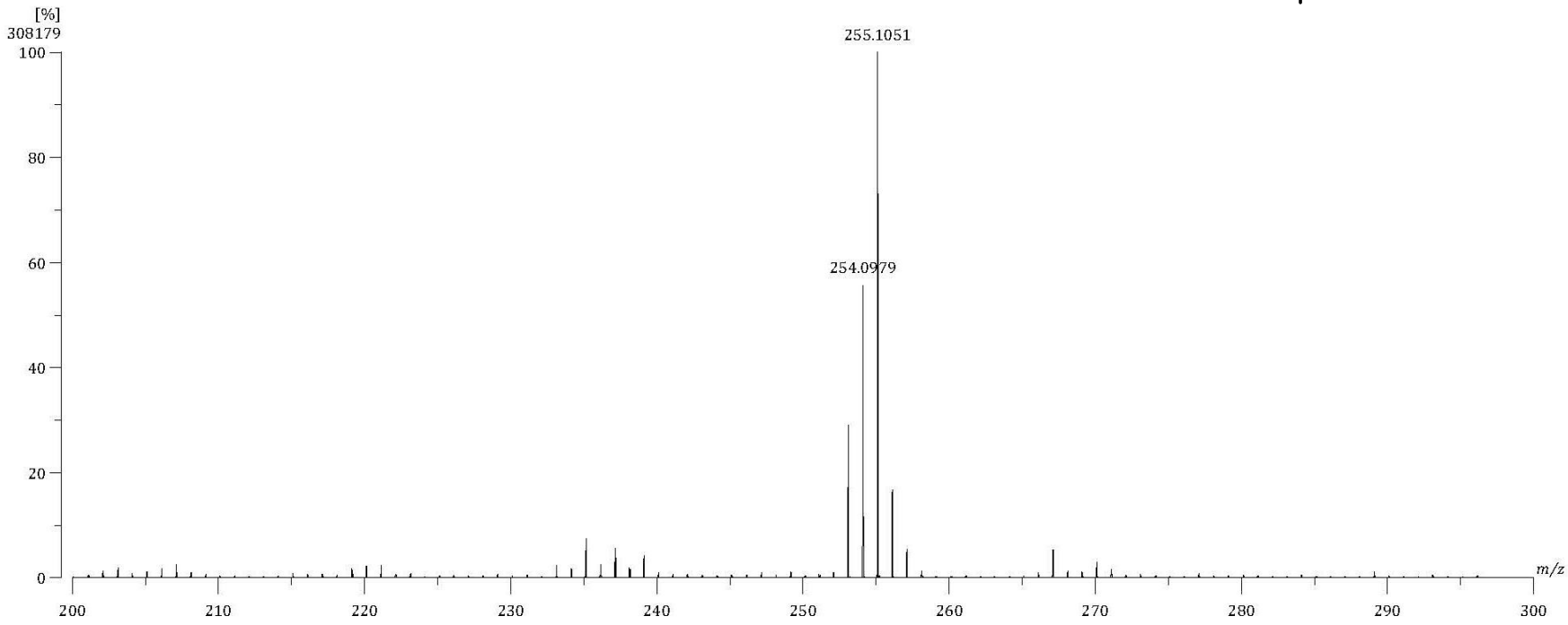
^{13}C NMR spectrum of **2b**



HRMS spectrum of **2b**

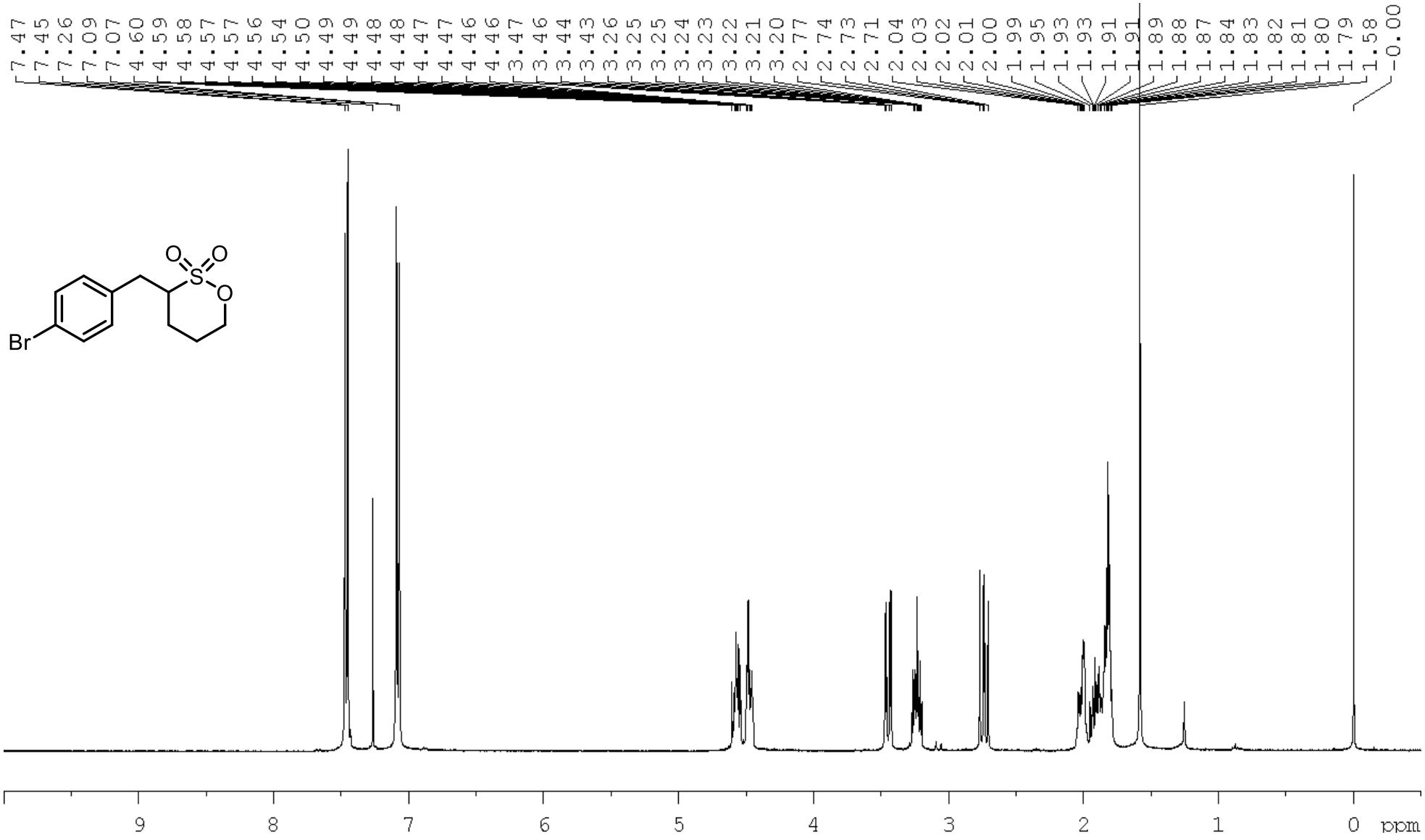
[Mass Spectrum]
Data : 20220819_MesC4-HR-001 Date : 19-Aug-2022 17:14
Sample : MesC4
Note : NBA
Ion Mode : FAB+
RT : 0.10 min Scan# : 2
Elements : C 1000/0, H 1000/0, O 3/3, S 1/1
Mass Tolerance : 50mmu
Unsaturation (U.S.) : -0.5 - 1000.0



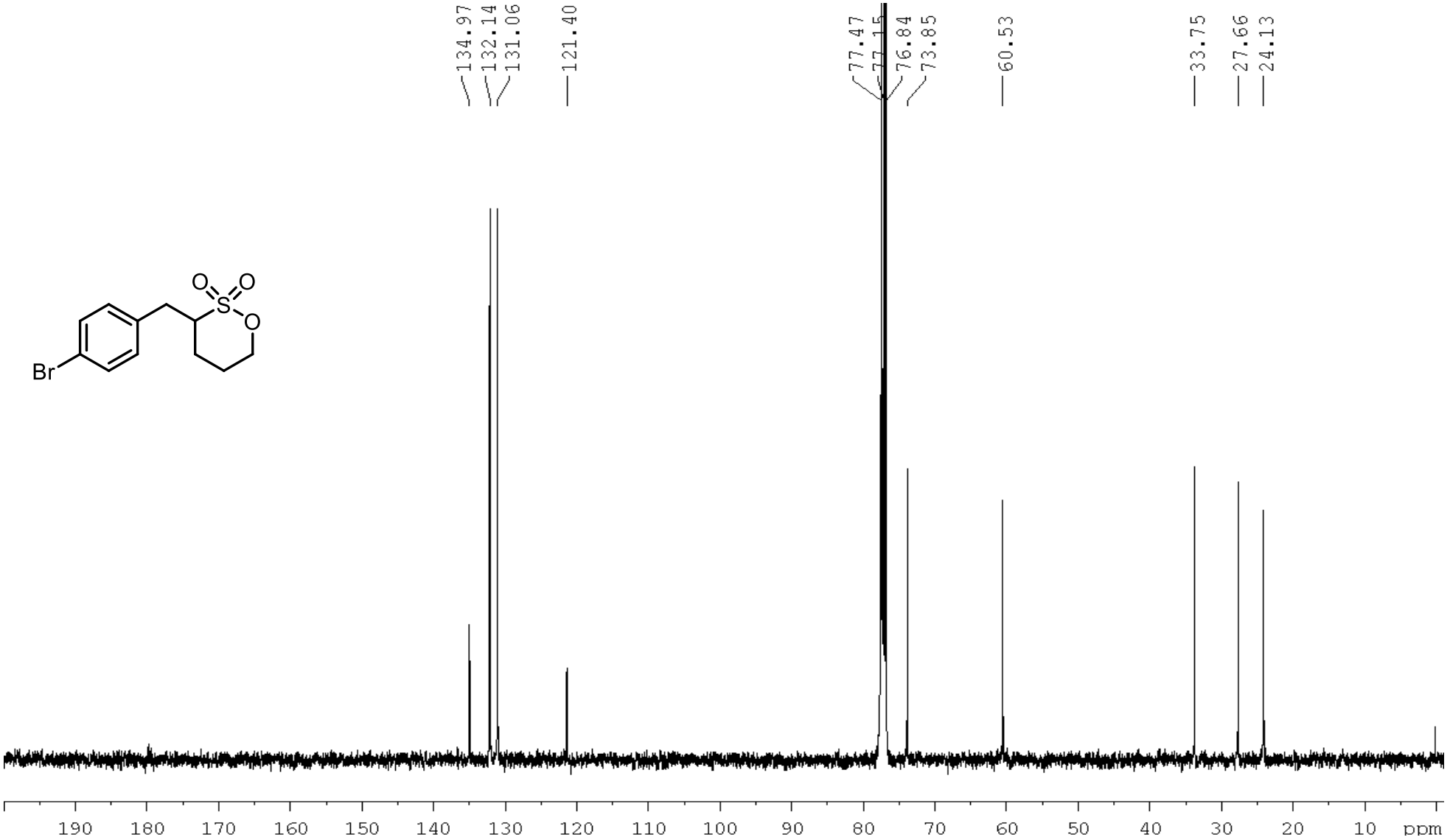


	Observed m/z	Int%					
	255.1051	100.00					
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	O	S
1	255.1055	-1.5 / -0.4	5.5	13	19	3	1

^{1}H NMR spectrum of **2c**

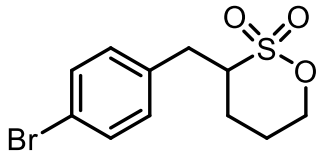


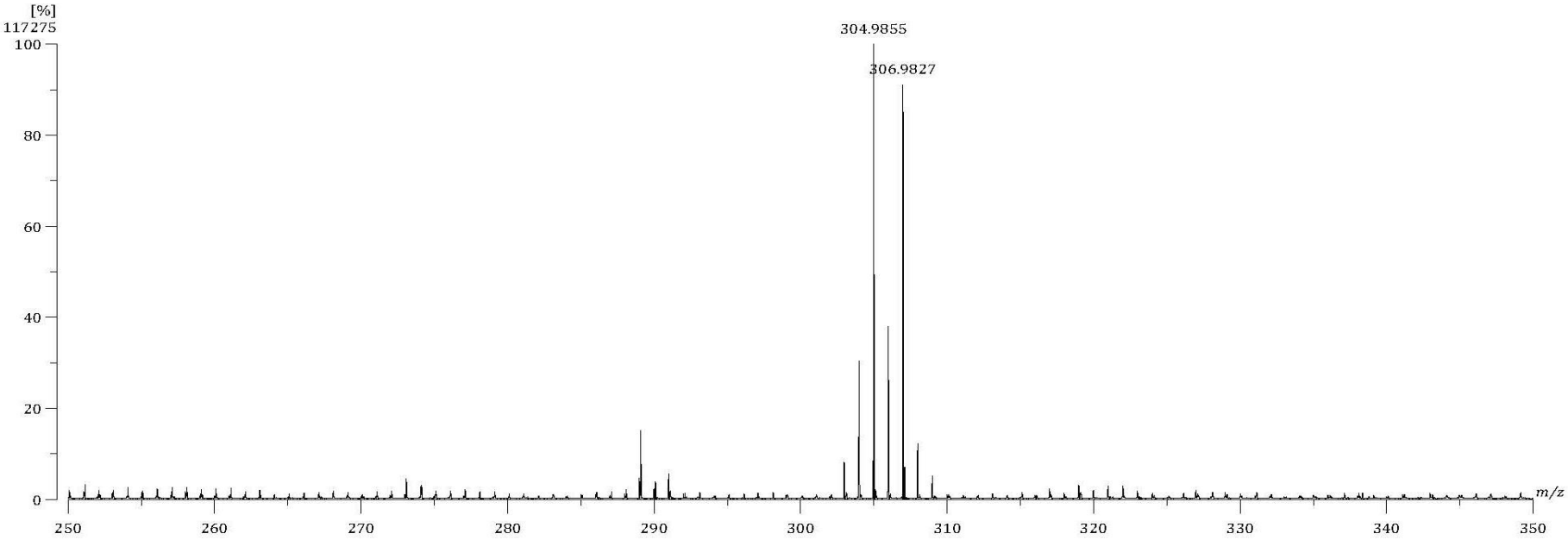
^{13}C NMR spectrum of **2c**



HRMS spectrum of **2c**

[Mass Spectrum]
Data : 20220819_4-BrBnC4-HR-002 Date : 19-Aug-2022 16:58
Sample : 4-BrBnC4
Note : NBA
Ion Mode : FAB+
RT : 0.43 min Scan# : (6,7)
Elements : C 1000/0, H 1000/0, 79Br 1/0, 81Br 1/0, O 3/3, S 1/1
Mass Tolerance : 5mmu
Unsaturation (U.S.) : -0.5 - 1000.0

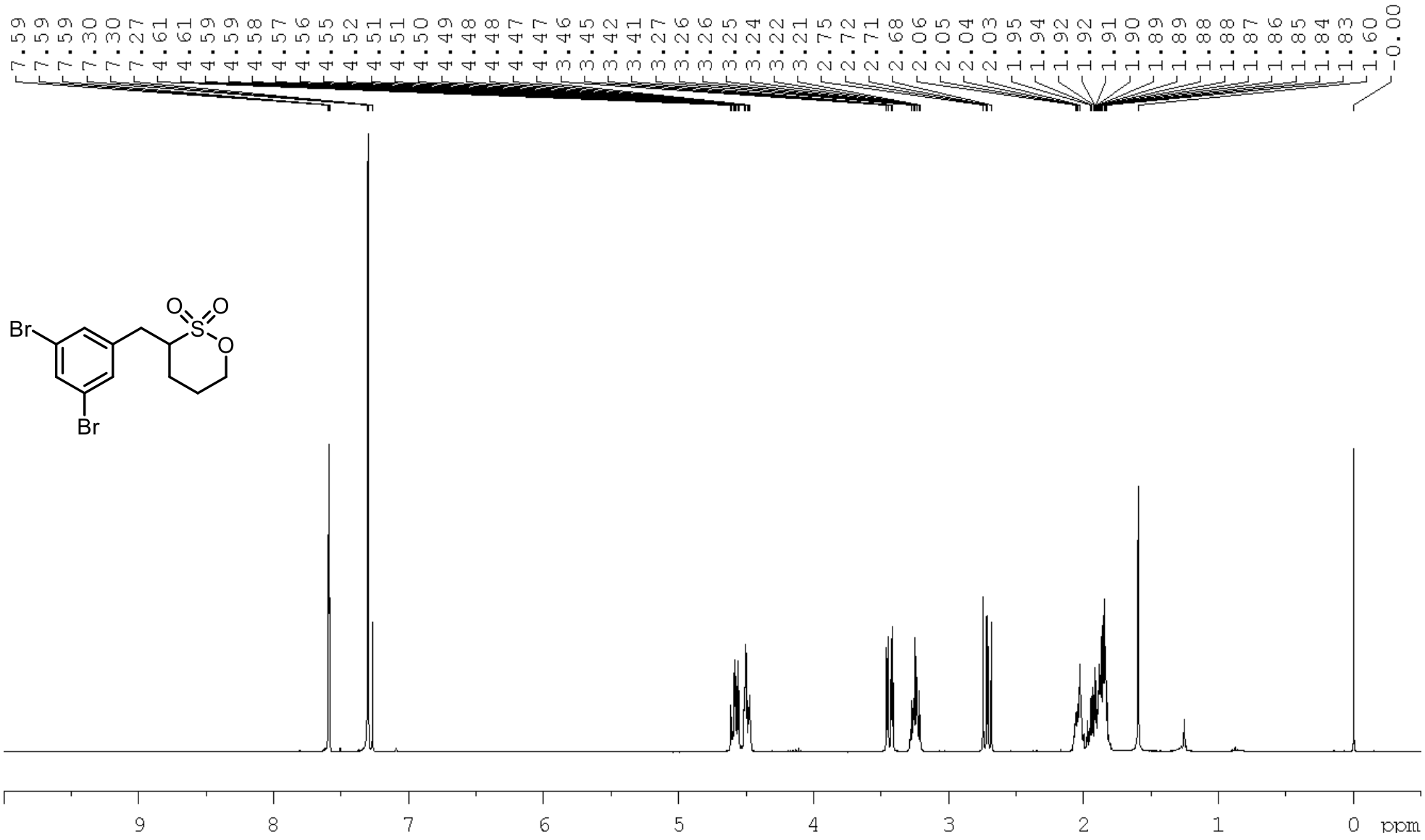




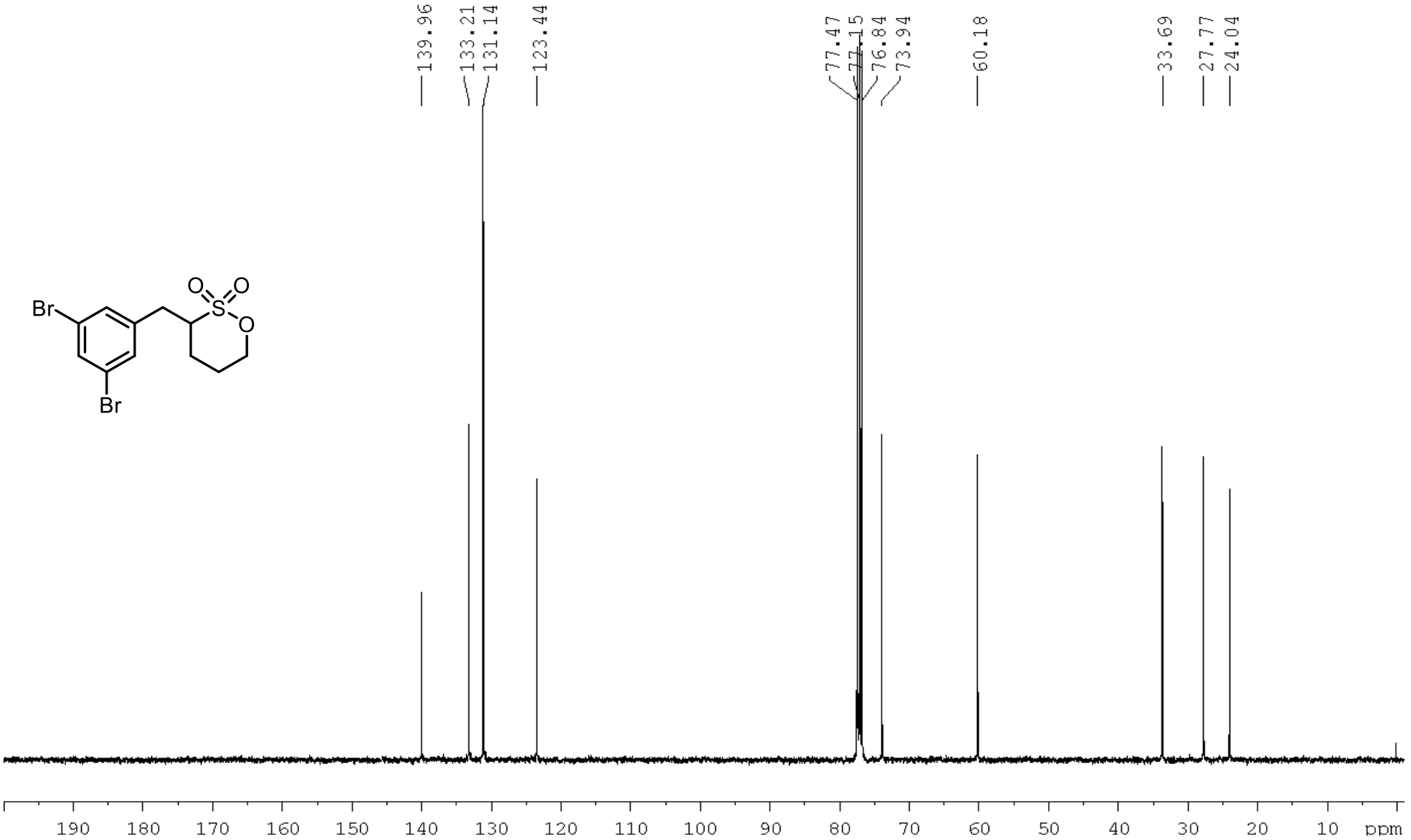
	Observed m/z	Int%								
	304.9855	100.00								
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	O	S	
1	304.9847	+2.6 / +0.8	5.5	11	14	1	-	3	1	

	Observed m/z	Int%								
	306.9827	91.01								
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	O	S	
2	306.9827	+0.1 / +0.0	5.5	11	14	-	1	3	1	

^{1}H NMR spectrum of **2d**

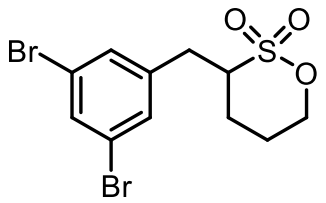


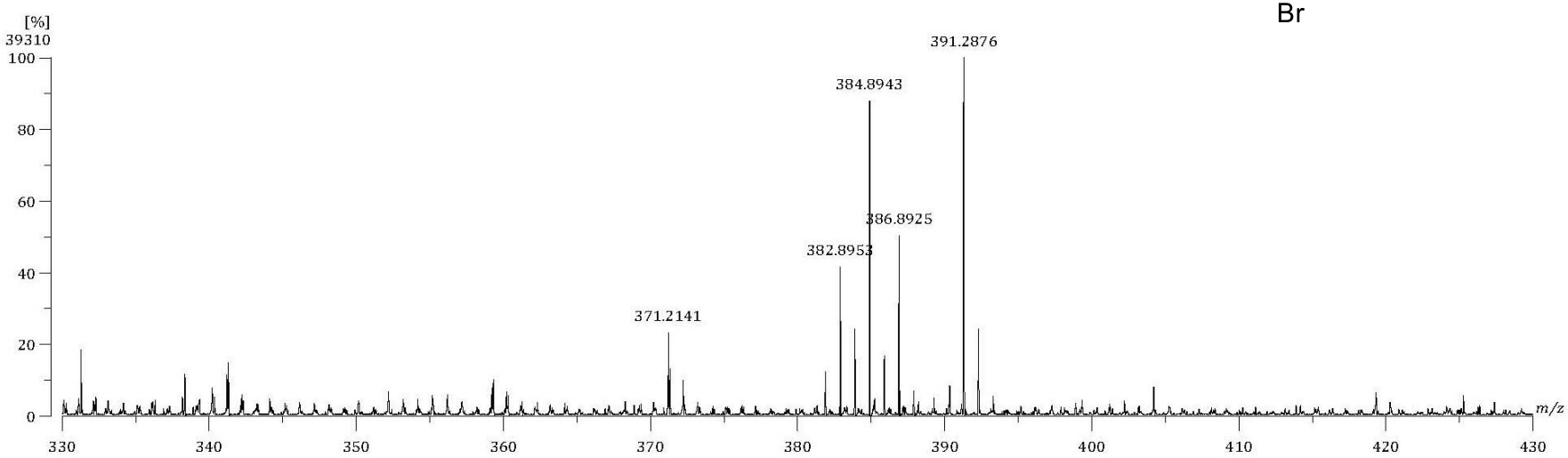
^{13}C NMR spectrum of **2d**



HRMS spectrum of **2d**

[Mass Spectrum]
Data : 20221011_3,5-diBrBN-HR-001 Date : 11-Oct-2022 17:07
Sample : 3,5-diBrBN
Note : NBA
Ion Mode : FAB+
RT : 0.60 min Scan# : (9,10)
Elements : C 1000/0, H 1000/0, 79Br 2/0, 81Br 2/0, O 3/3, S 1/1
Mass Tolerance : 5mmu
Unsaturation (U.S.) : -0.5 - 1000.0



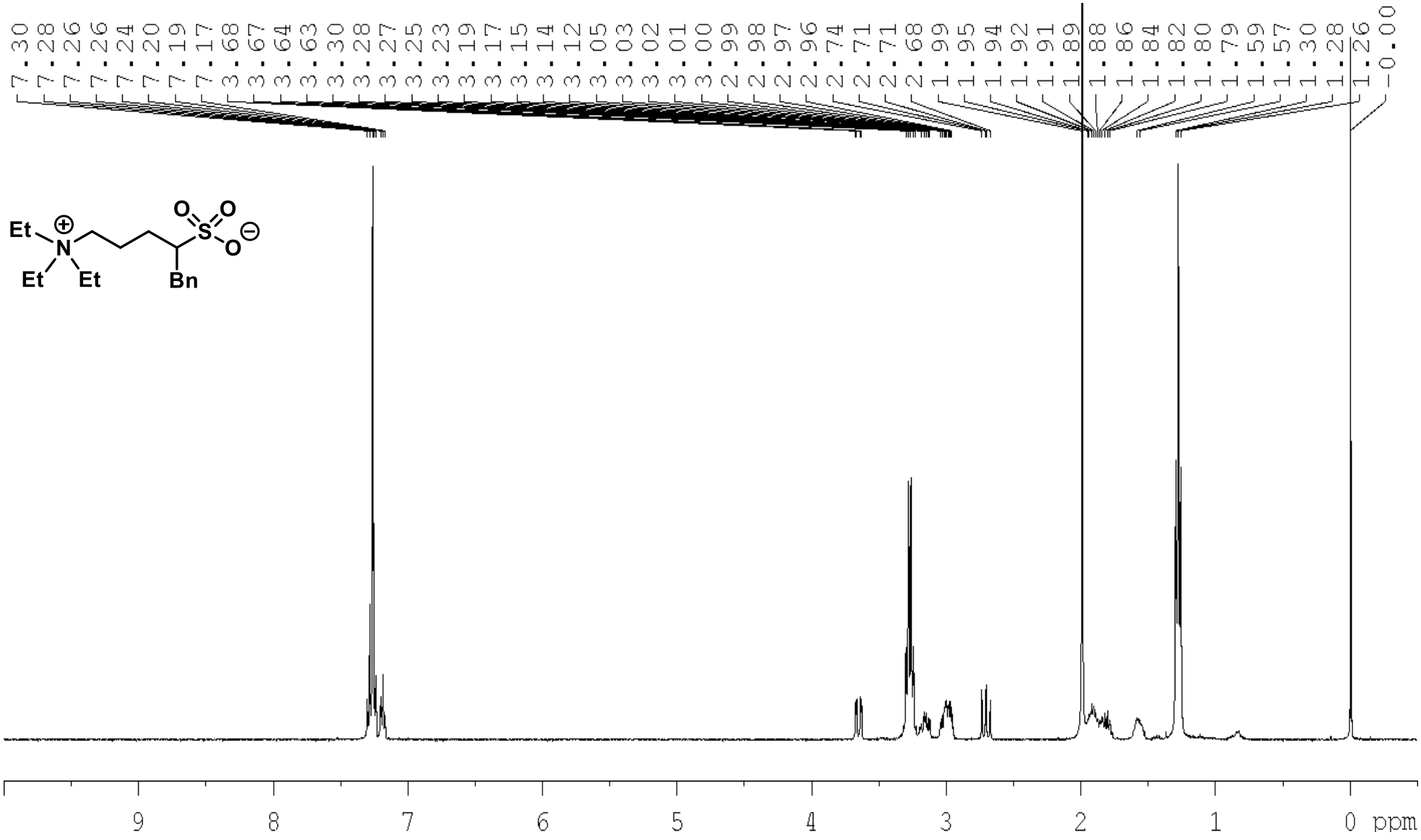


	Observed m/z	Int%							
	382.8953	41.56							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	O	S
1	382.8952	+0.2 / +0.1	5.5	11	13	2	-	3	1

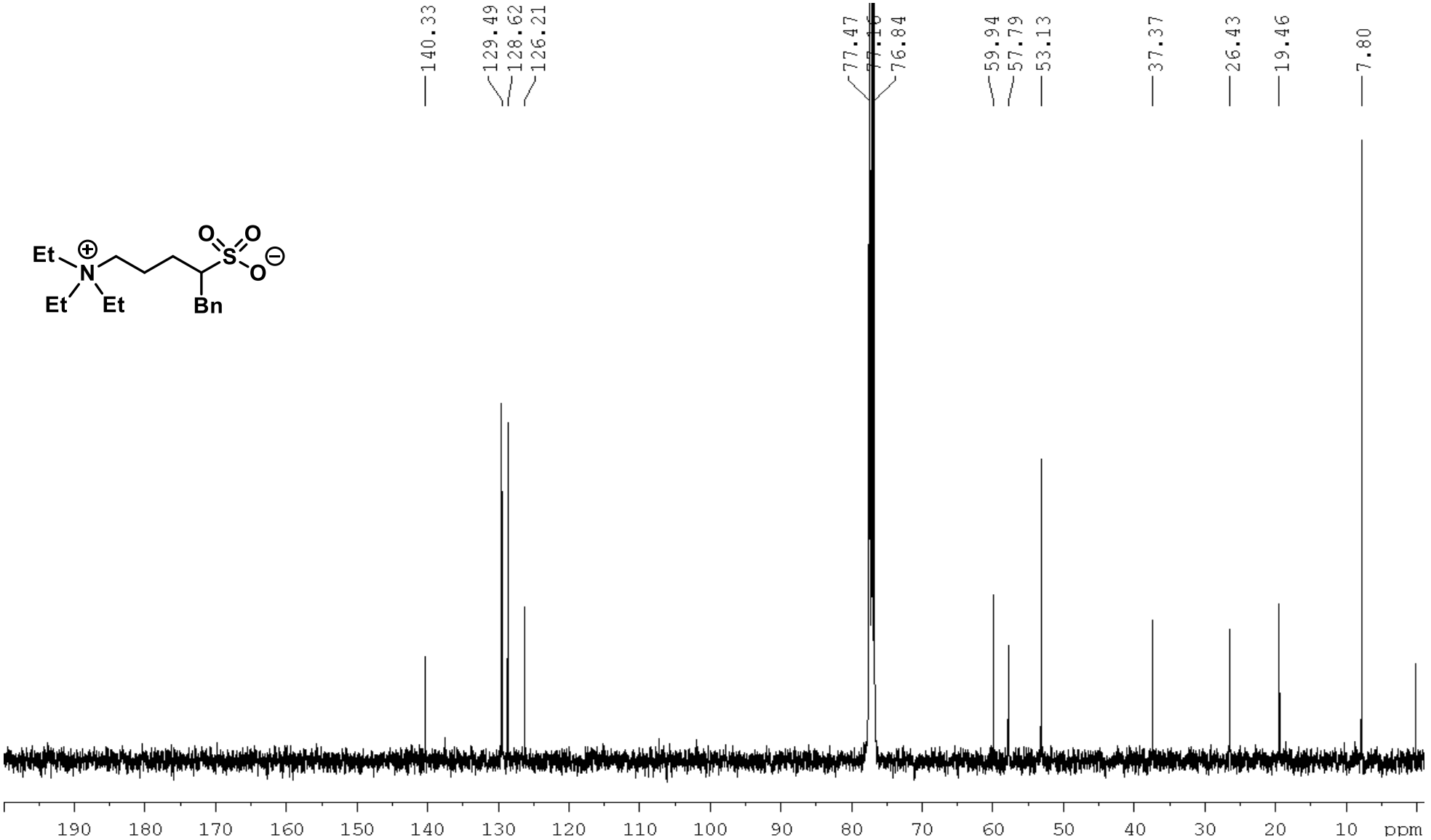
	Observed m/z	Int%							
	384.8943	87.94							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	O	S
2	384.8932	+2.9 / +1.1	5.5	11	13	1	1	3	1

	Observed m/z	Int%							
	386.8925	50.30							
	Estimated m/z	Err[ppm / mmu]	U.S.	C	H	79Br	81Br	O	S
3	386.8911	+3.6 / +1.4	5.5	11	13	-	2	3	1

^{1}H NMR spectrum of **ZM 1a**

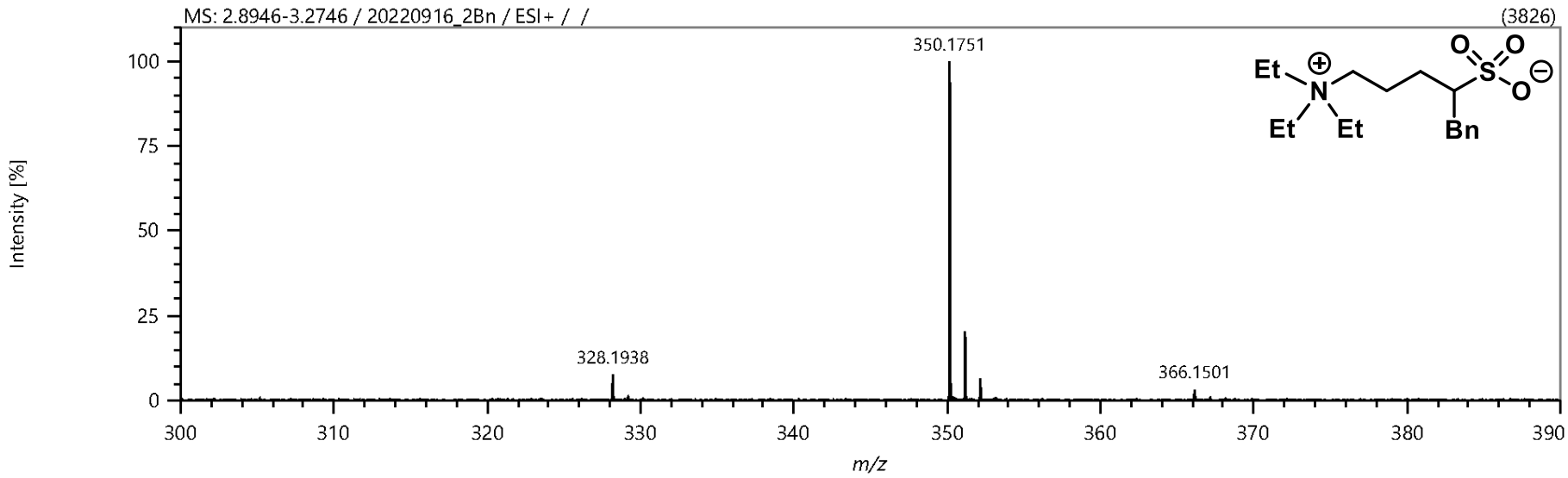


^{13}C NMR spectrum of **ZM 1a**



HRMS spectrum of **ZM 1a**

Spectrum



Elemental Composition

Parameters	
Tolerance:	±3.00 ppm
Electron:	Odd/Even
Charge:	+1
DBE:	-99.0 - 999.0

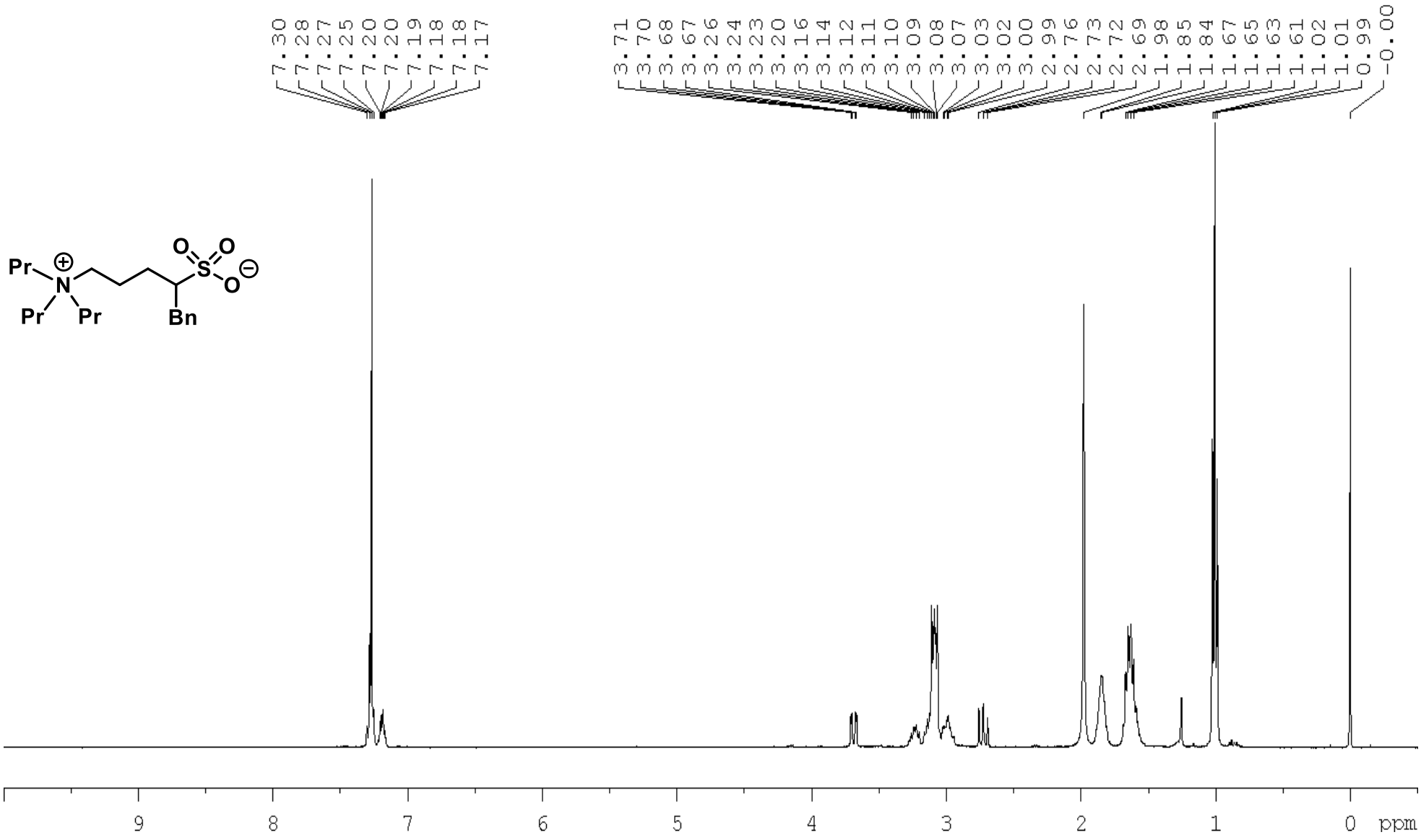
Elements Set 1:

Symbol	C	H	N	O	S	Na	K
Min	0	0	1	3	1	0	0
Max	400	1000	1	3	1	1	1

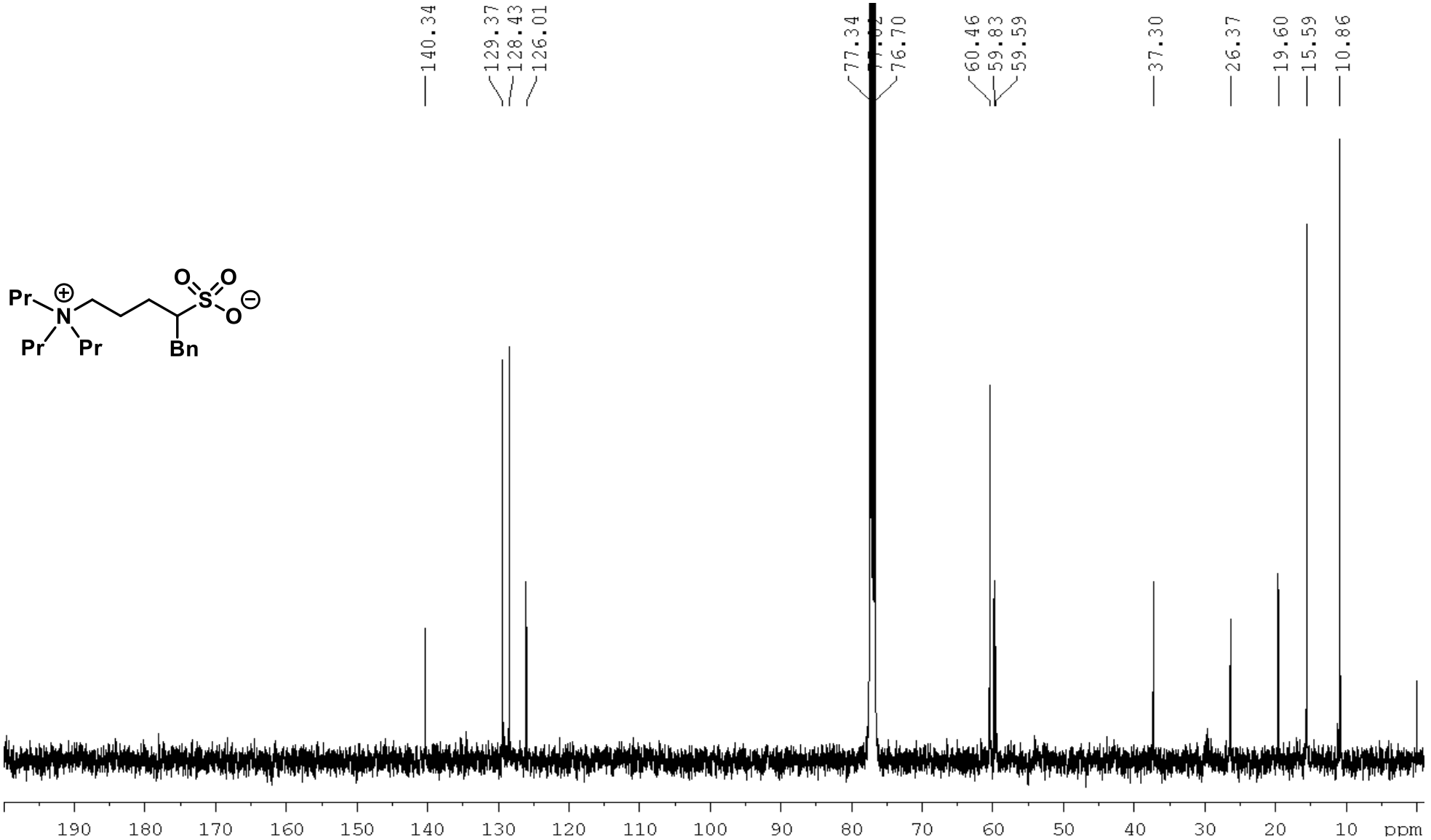
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
328.19376	C17 H30 N O3 S	328.19409	-0.33	-1.01	3.5
350.17508	C17 H29 N O3 Na S	350.17604	-0.96	-2.74	3.5
366.15011	C17 H29 N O3 S K	366.14997	0.13	0.36	3.5

^{1}H NMR spectrum of **ZM 1b**

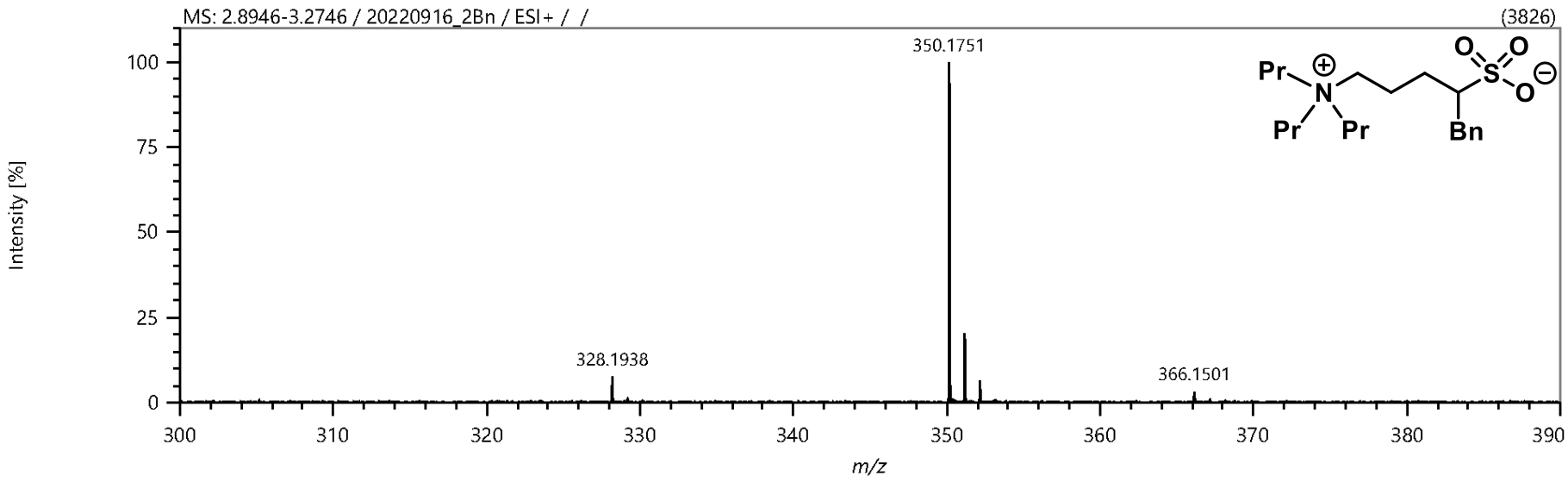


^{13}C NMR spectrum of **ZM 1b**



HRMS spectrum of **ZM 1b**

Spectrum



Elemental Composition

Parameters	
Tolerance:	±3.00 ppm
Electron:	Odd/Even
Charge:	+1
DBE:	-99.0 - 999.0

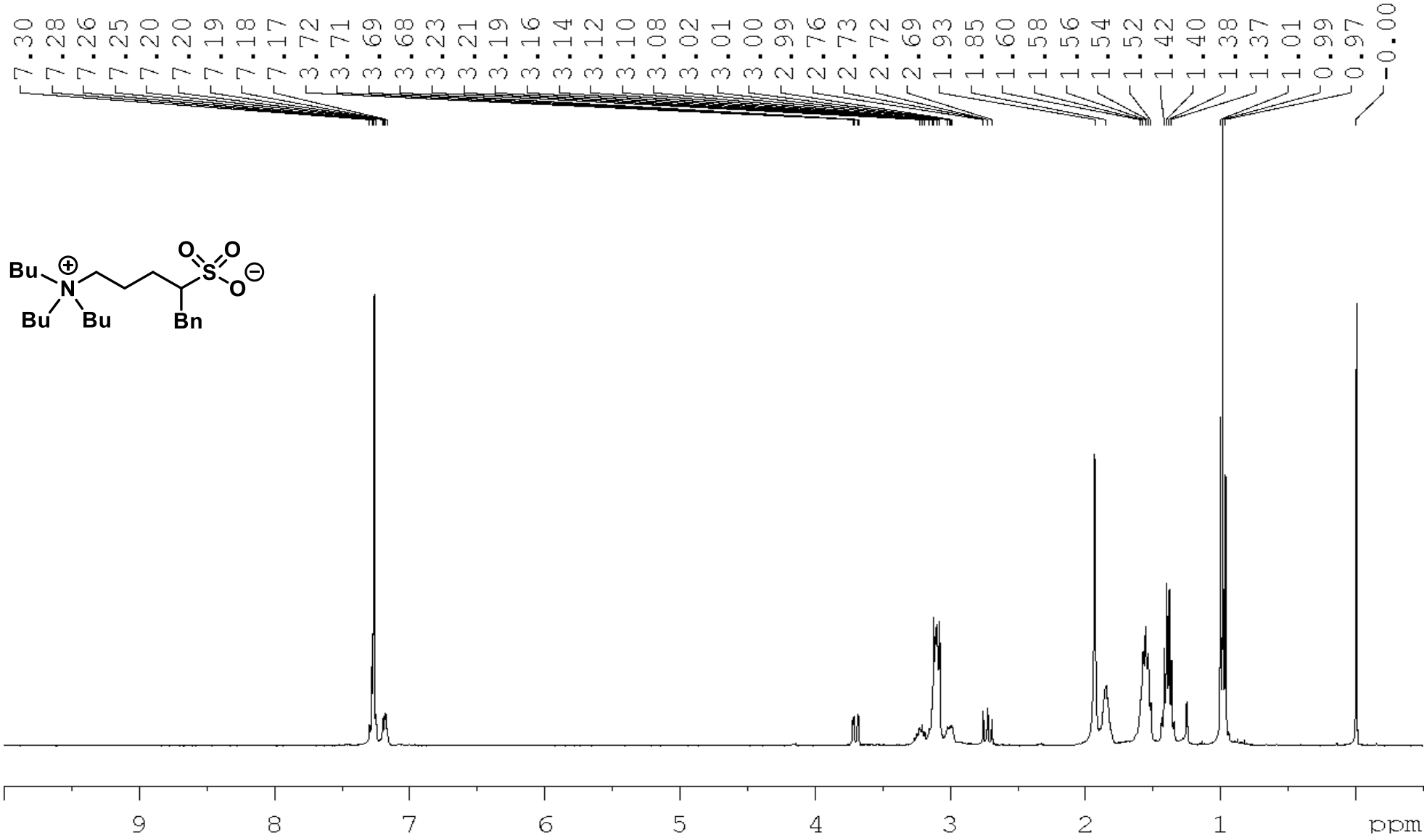
Elements Set 1:

Symbol	C	H	N	O	S	Na	K
Min	0	0	1	3	1	0	0
Max	400	1000	1	3	1	1	1

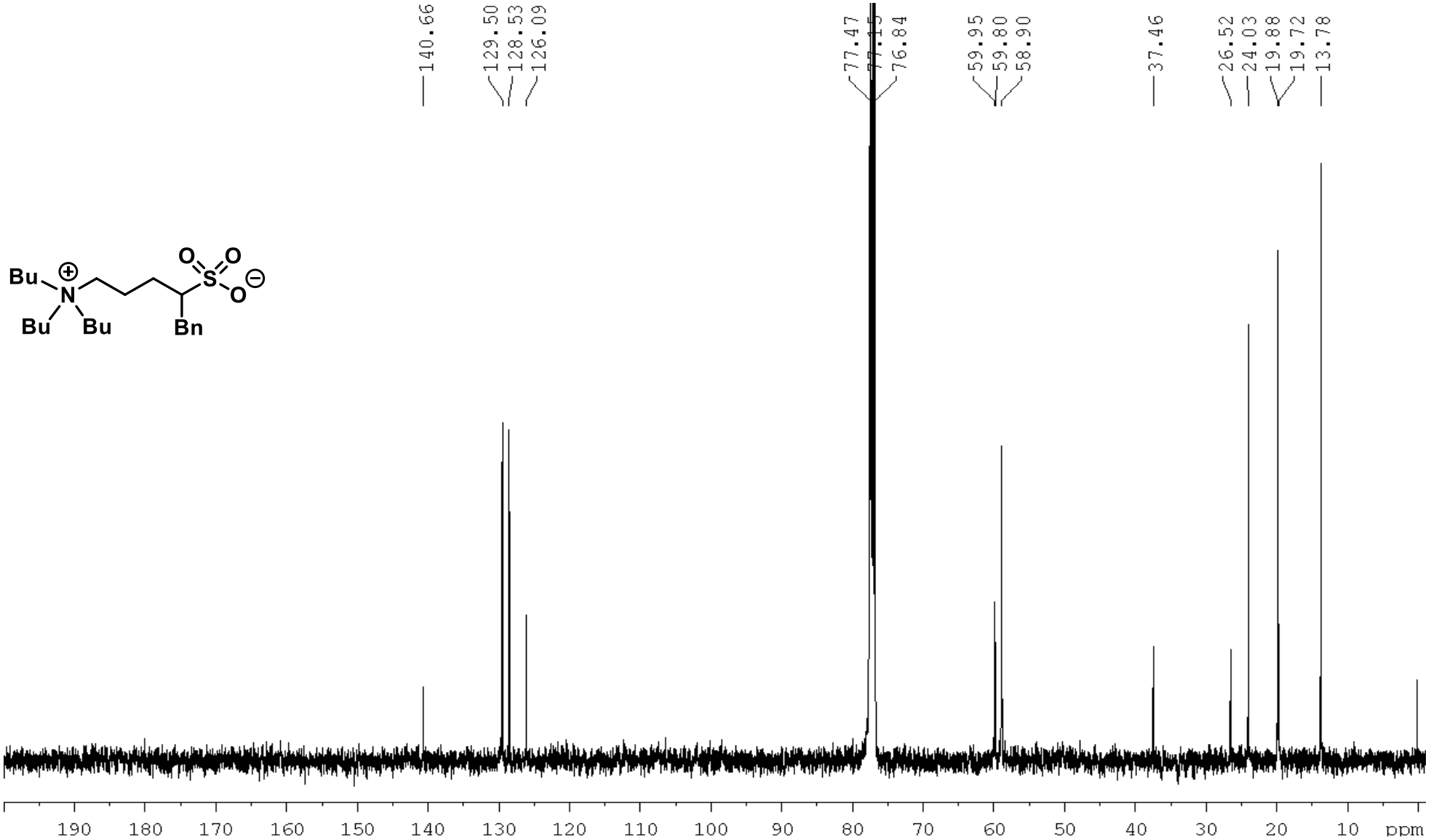
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
328.19376	C17 H30 N O3 S	328.19409	-0.33	-1.01	3.5
350.17508	C17 H29 N O3 Na S	350.17604	-0.96	-2.74	3.5
366.15011	C17 H29 N O3 S K	366.14997	0.13	0.36	3.5

^{1}H NMR spectrum of **ZM 1c**

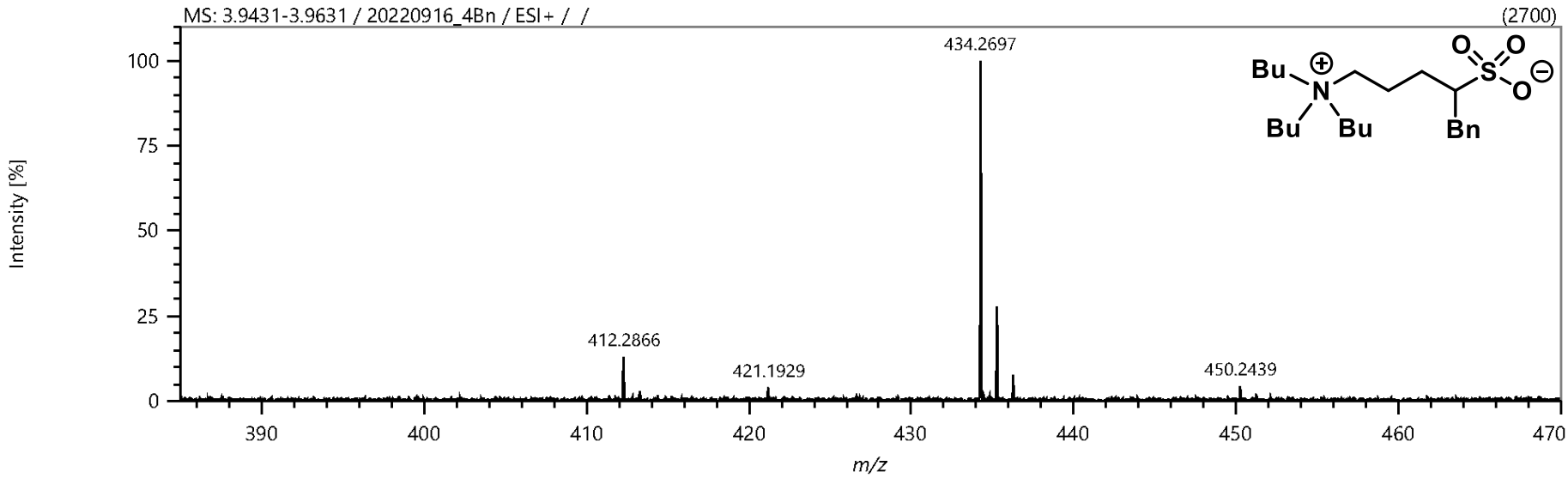


^{13}C NMR spectrum of **ZM 1c**



HRMS spectrum of **ZM 1c**

Spectrum



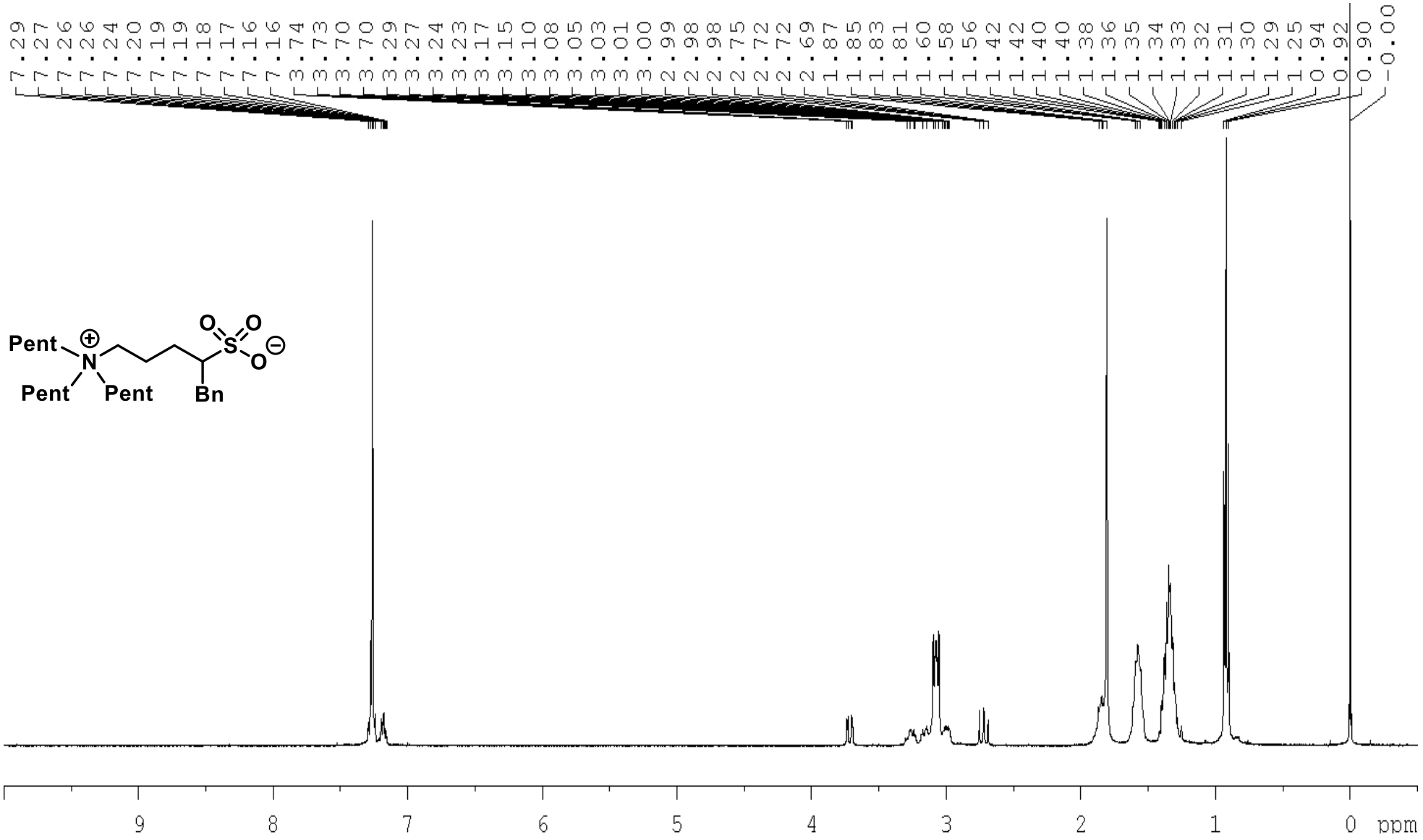
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±3.00 ppm	Symbol	C	H	N	O	S	Na	K
Electron:	Odd/Even	Min	0	0	1	3	1	0	0
Charge:	+1	Max	400	1000	1	3	1	1	1
DBE:	-99.0 - 999.0								

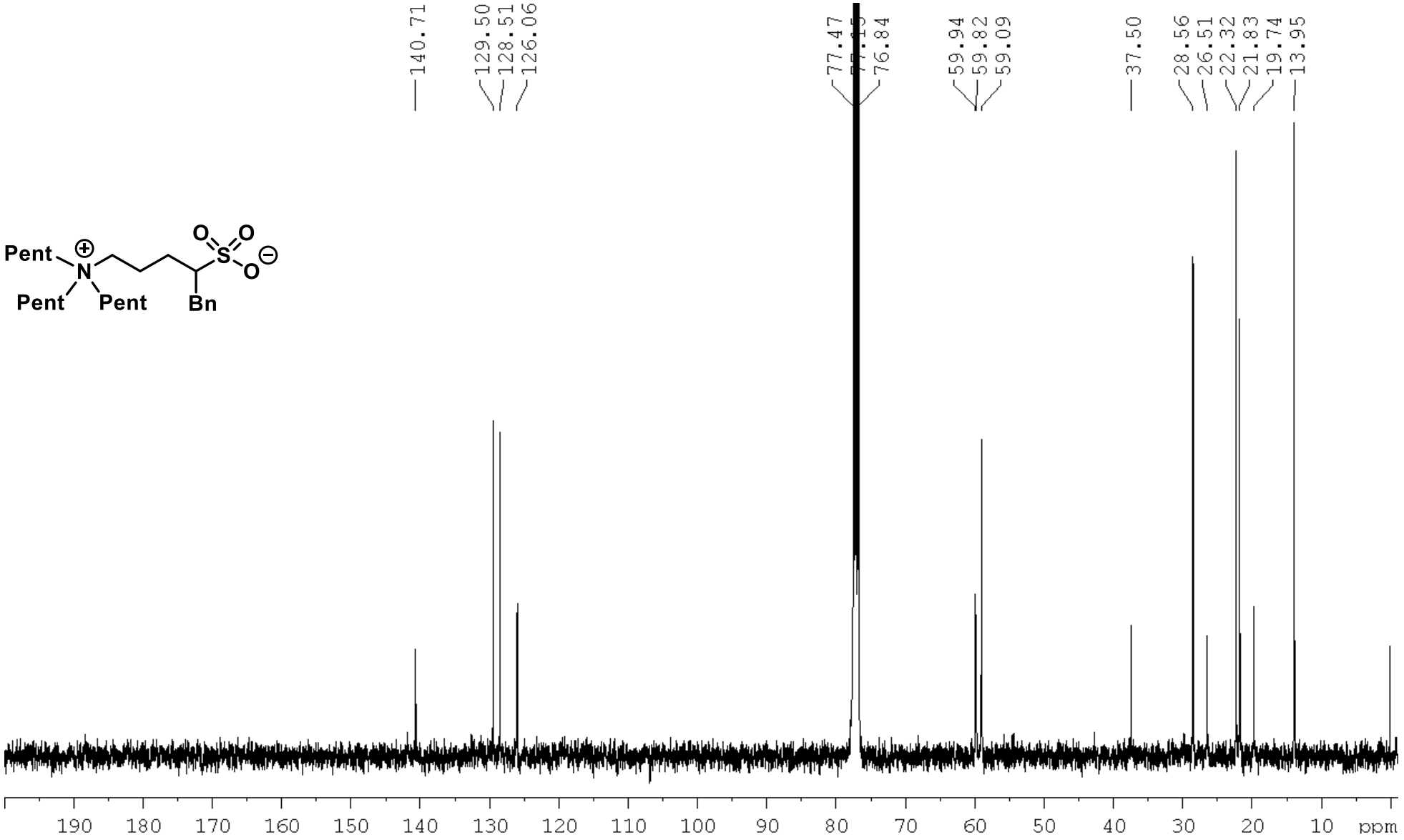
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
412.28657	C21 H43 N O3 Na S	412.28559	0.98	2.38	0.5
434.26972	C23 H41 N O3 Na S	434.26994	-0.21	-0.49	3.5
450.24391	C23 H41 N O3 S K	450.24387	0.03	0.07	3.5

^{1}H NMR spectrum of **ZM 1d**

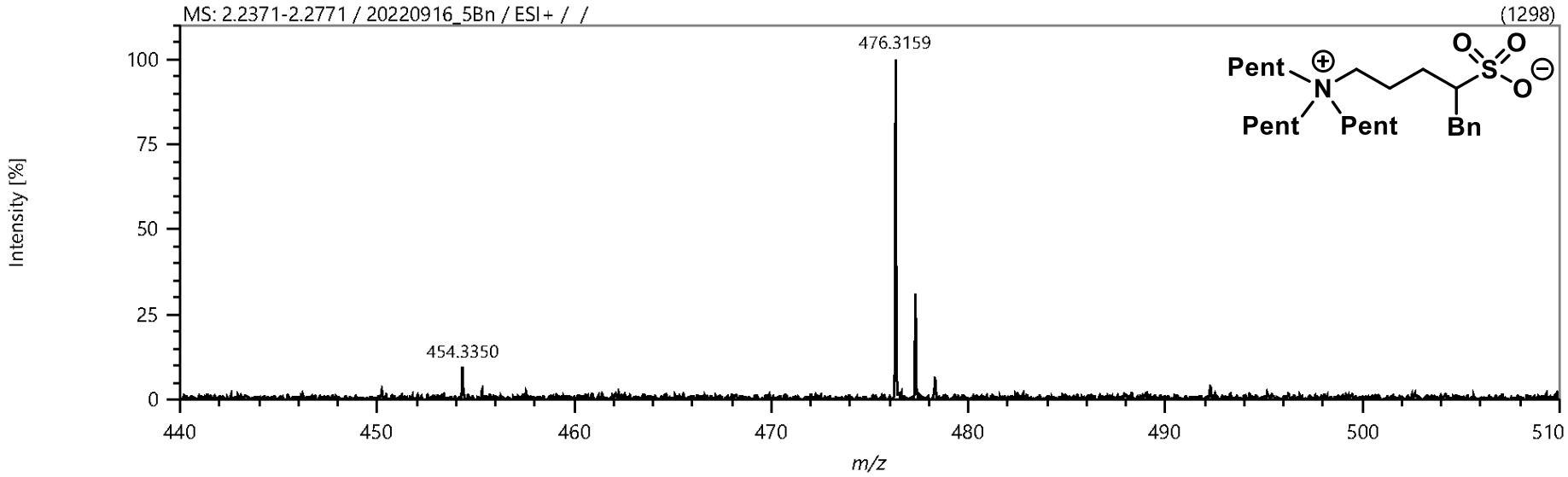


^{13}C NMR spectrum of **ZM 1d**



HRMS spectrum of **ZM 1d**

Spectrum



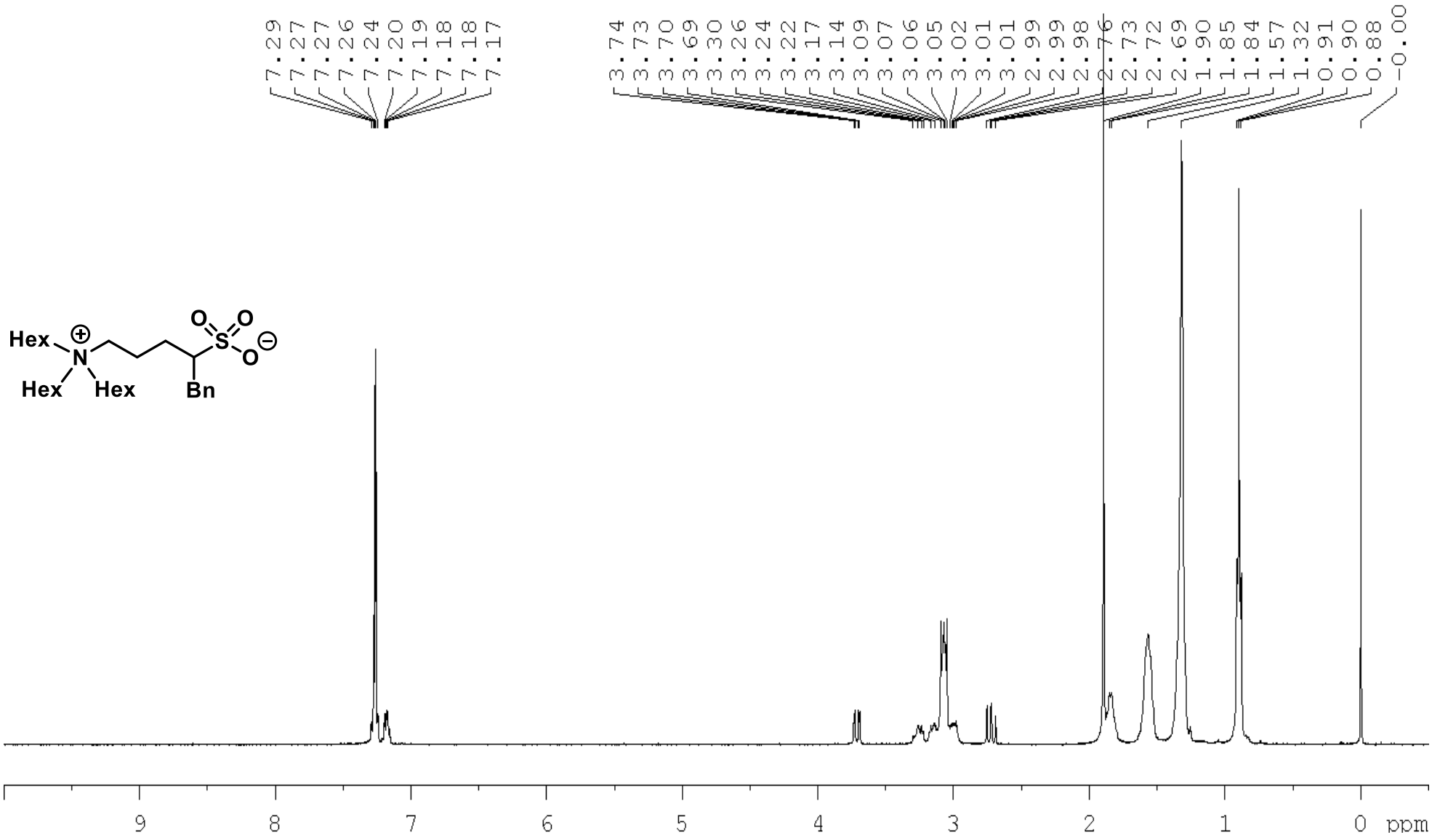
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±5.00 ppm	Symbol	C	H	N	O	S	Na	K
Electron:	Odd/Even	Min	0	0	1	3	1	0	0
Charge:	+1	Max	400	1000	1	3	1	1	1
DBE:	-99.0 - 999.0								

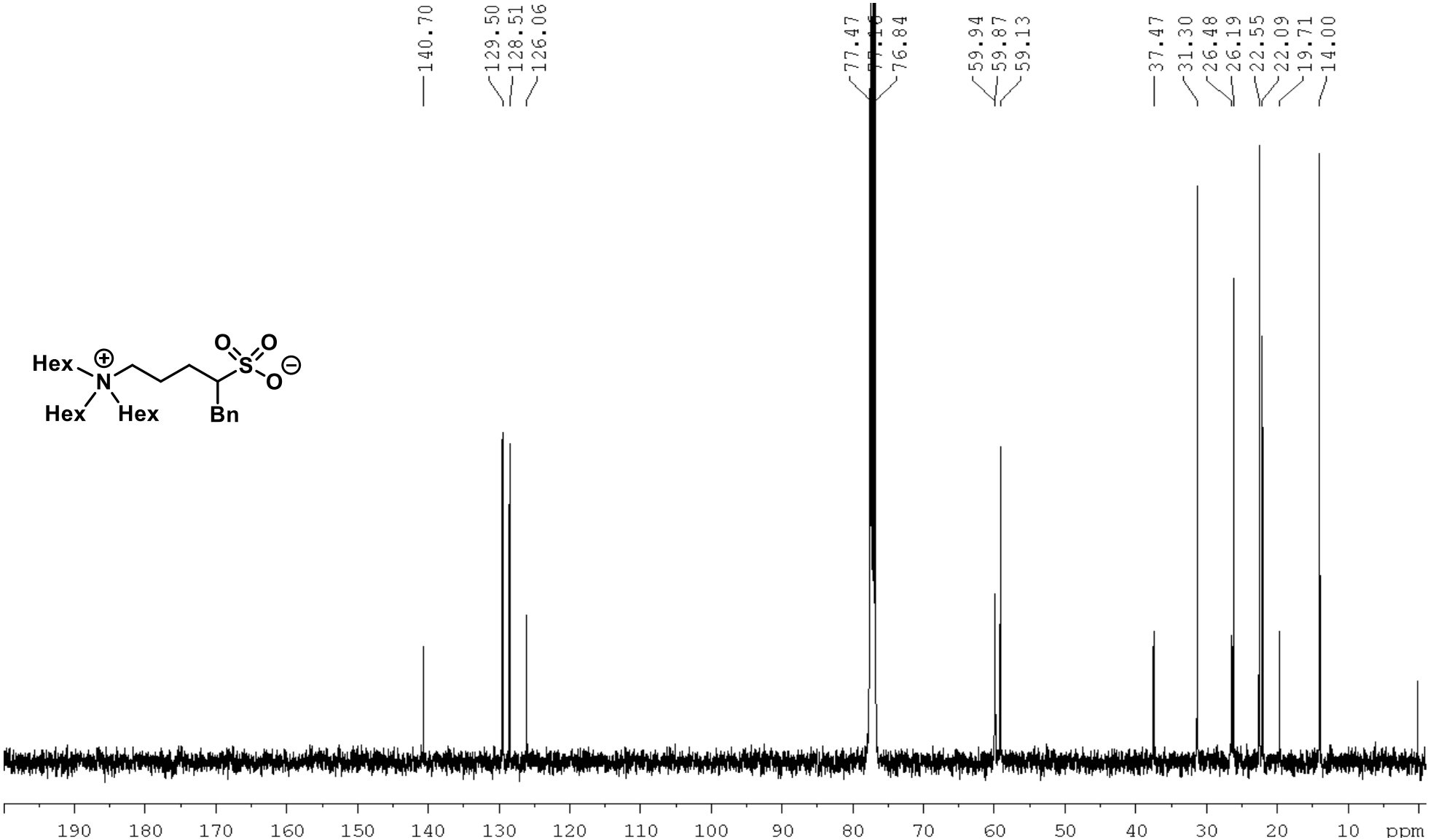
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
454.33504	C26 H48 N O3 S	454.33494	0.10	0.23	3.5
476.31592	C26 H47 N O3 Na S	476.31689	-0.97	-2.04	3.5

^{1}H NMR spectrum of **ZM 1e**

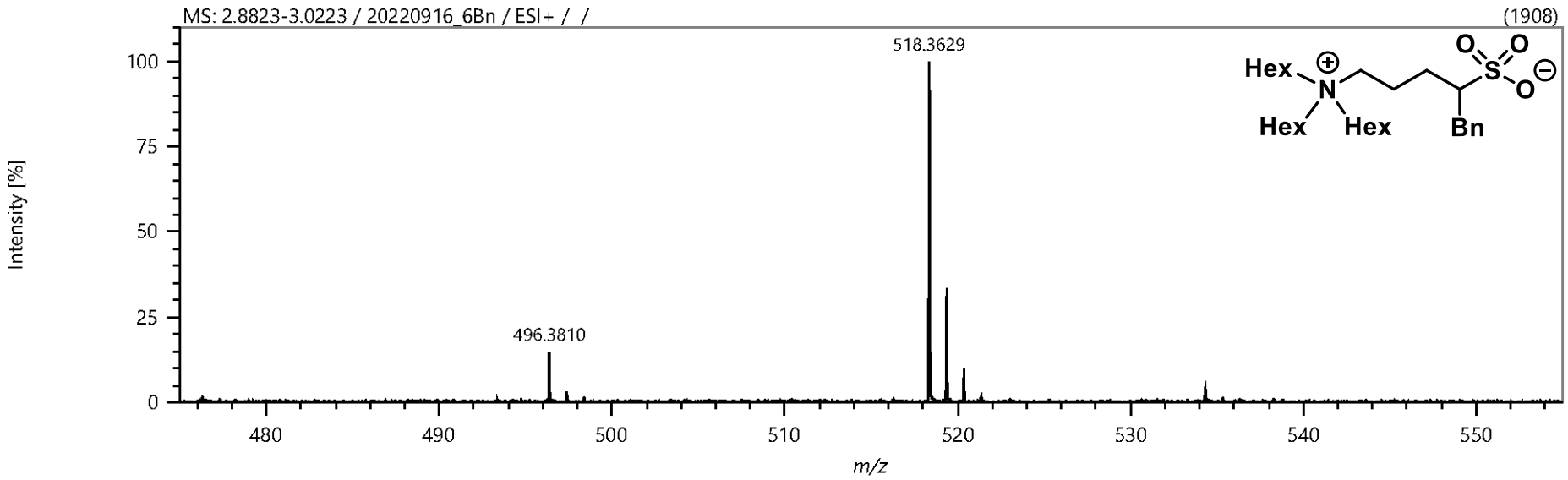


^{13}C NMR spectrum of **ZM 1e**



HRMS spectrum of **ZM 1e**

Spectrum



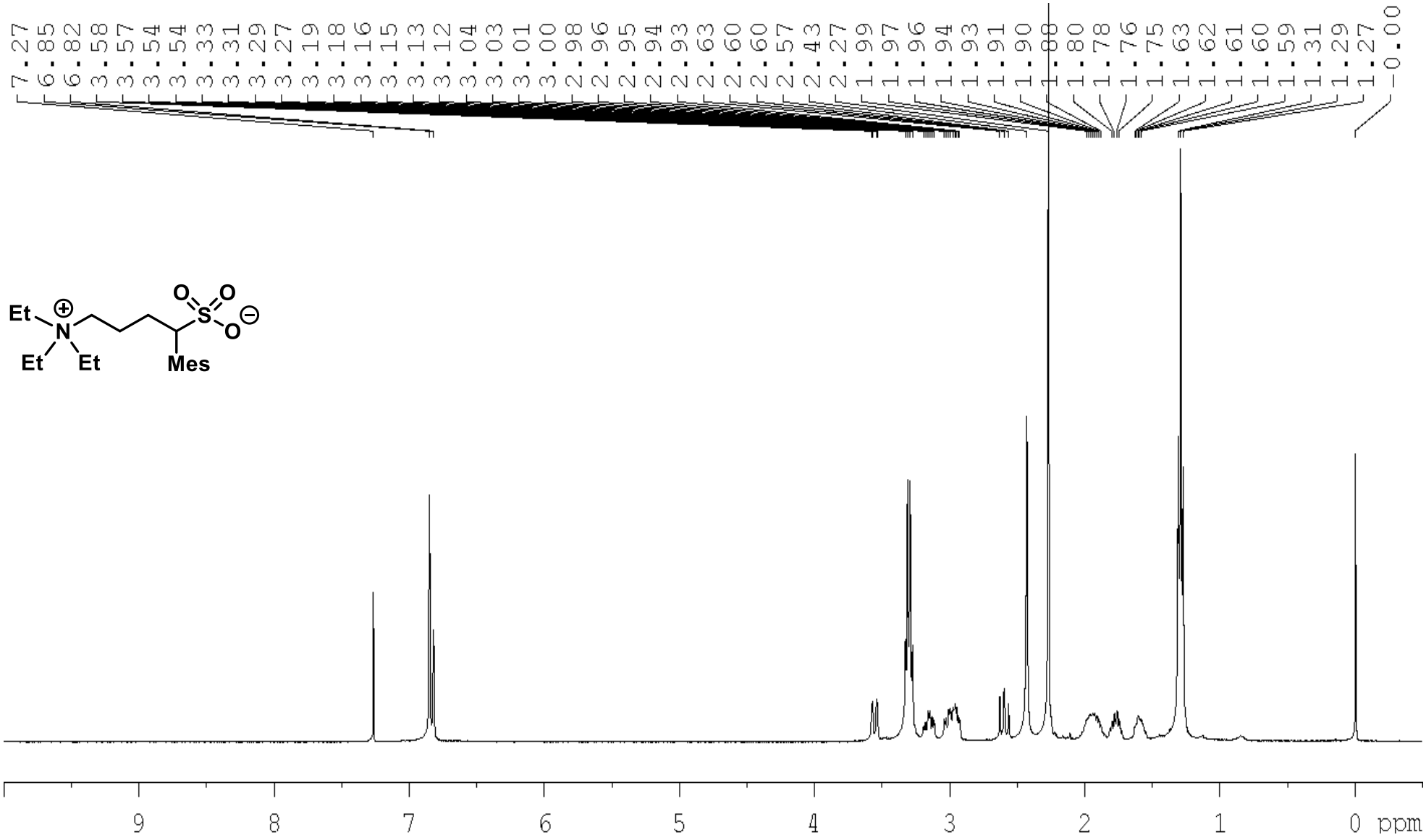
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±3.00 ppm	Symbol	C	H	N	O	S	Na	K
Electron:	Odd/Even	Min	0	0	1	3	1	0	0
Charge:	+1	Max	400	1000	1	3	1	1	1
DBE:	-99.0 - 999.0								

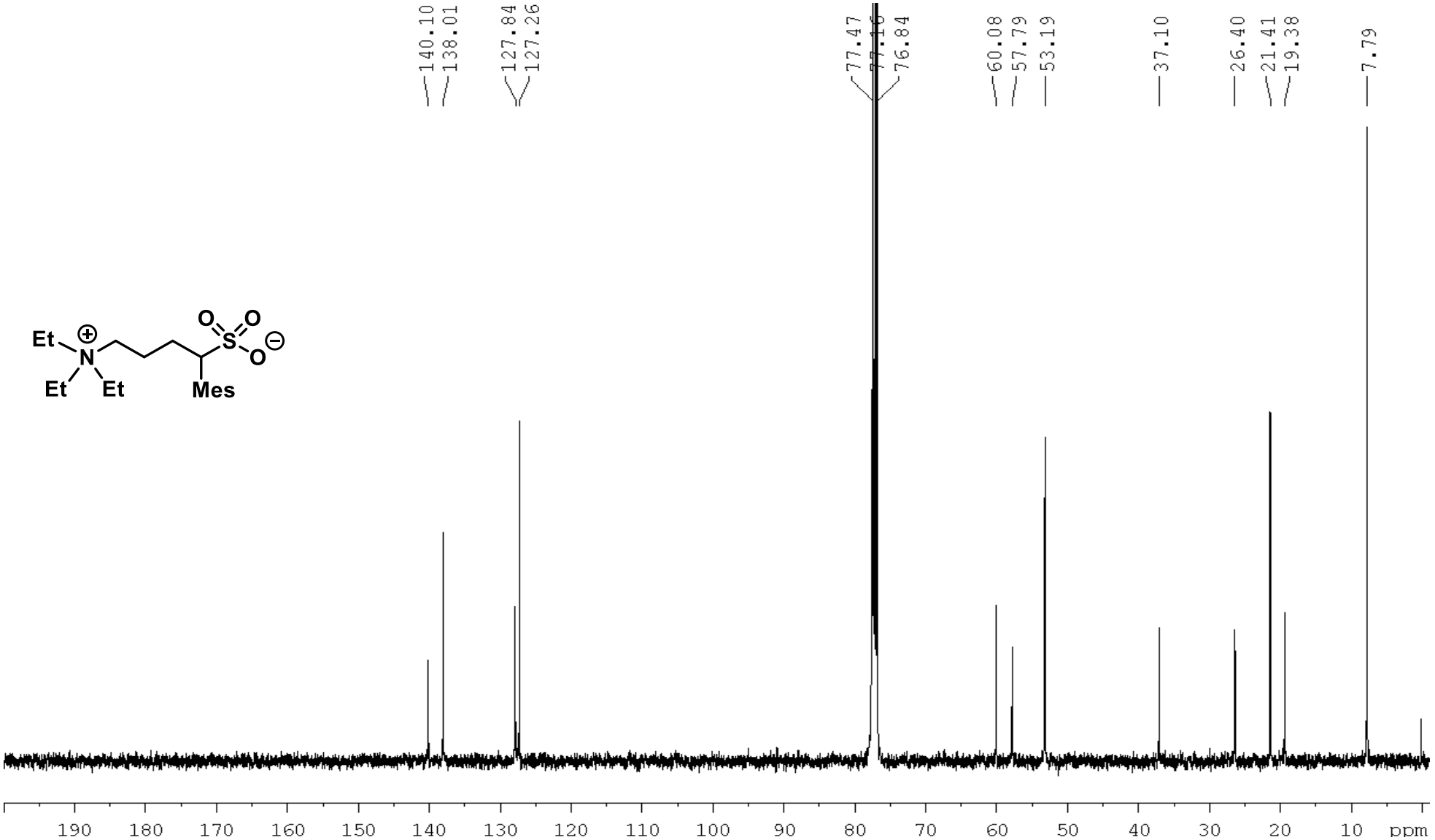
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
496.38098	C29 H54 N O3 S	496.38189	-0.91	-1.83	3.5
518.36292	C29 H53 N O3 Na S	518.36384	-0.92	-1.78	3.5

^{1}H NMR spectrum of **ZM 2a**

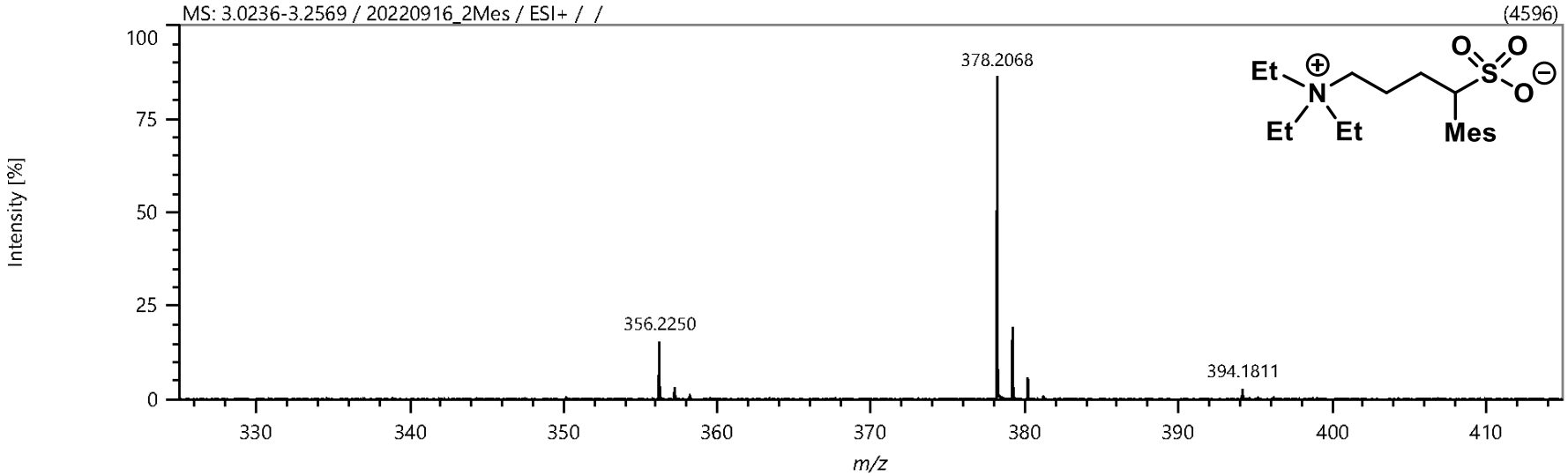


^{13}C NMR spectrum of **ZM 2a**



HRMS spectrum of **ZM 2a**

Spectrum



Elemental Composition

Parameters	
Tolerance:	±3.00 ppm
Electron:	Odd/Even
Charge:	+1
DBE:	-99.0 - 999.0

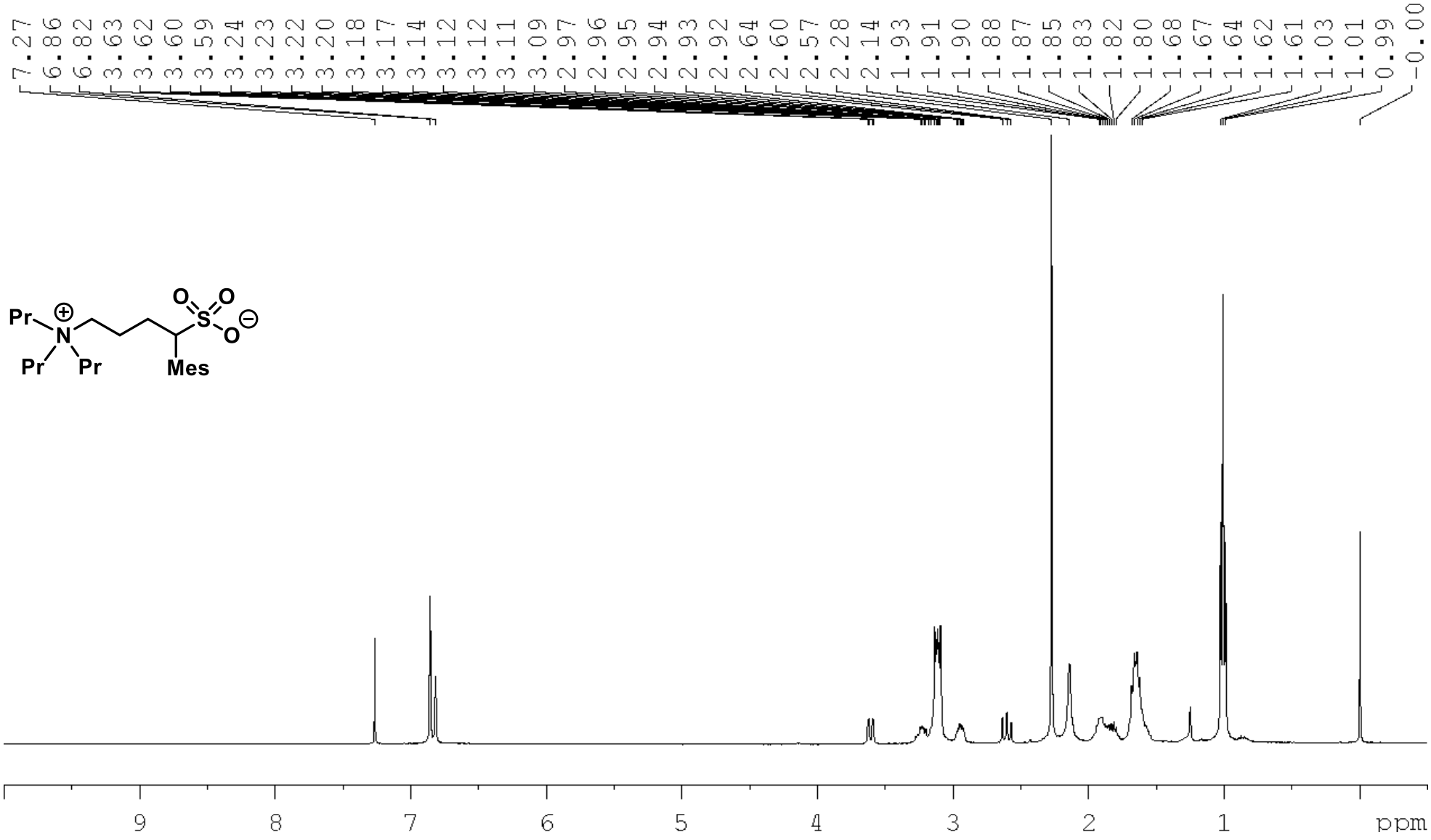
Elements Set 1:

Symbol	C	H	N	O	S	Na	K
Min	0	0	1	3	1	0	0
Max	400	1000	1	3	1	1	1

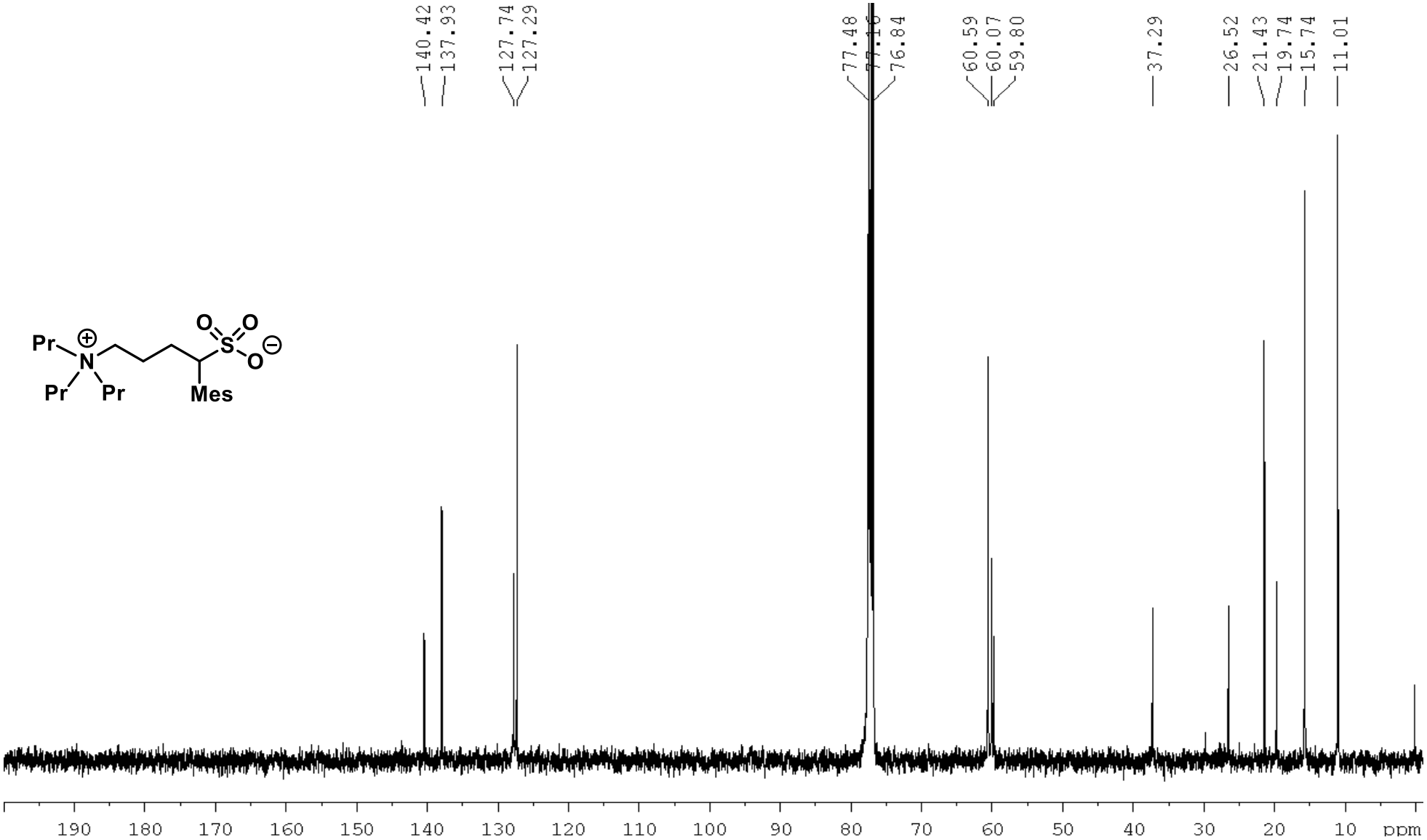
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
356.22500	C19 H34 N O3 S	356.22539	-0.39	-1.08	3.5
378.20683	C19 H33 N O3 Na S	378.20734	-0.51	-1.34	3.5
394.18107	C19 H33 N O3 S K	394.18127	-0.21	-0.52	3.5

^{1}H NMR spectrum of **ZM 2b**

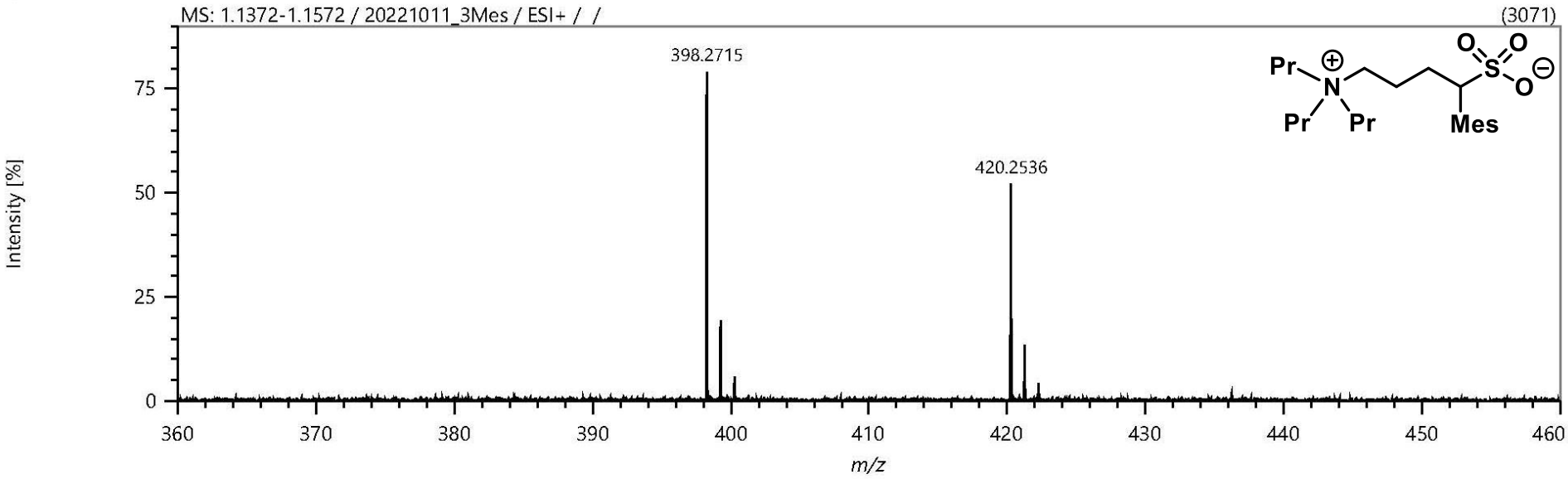


^{13}C NMR spectrum of **ZM 2b**



HRMS spectrum of **ZM 2b**

Spectrum



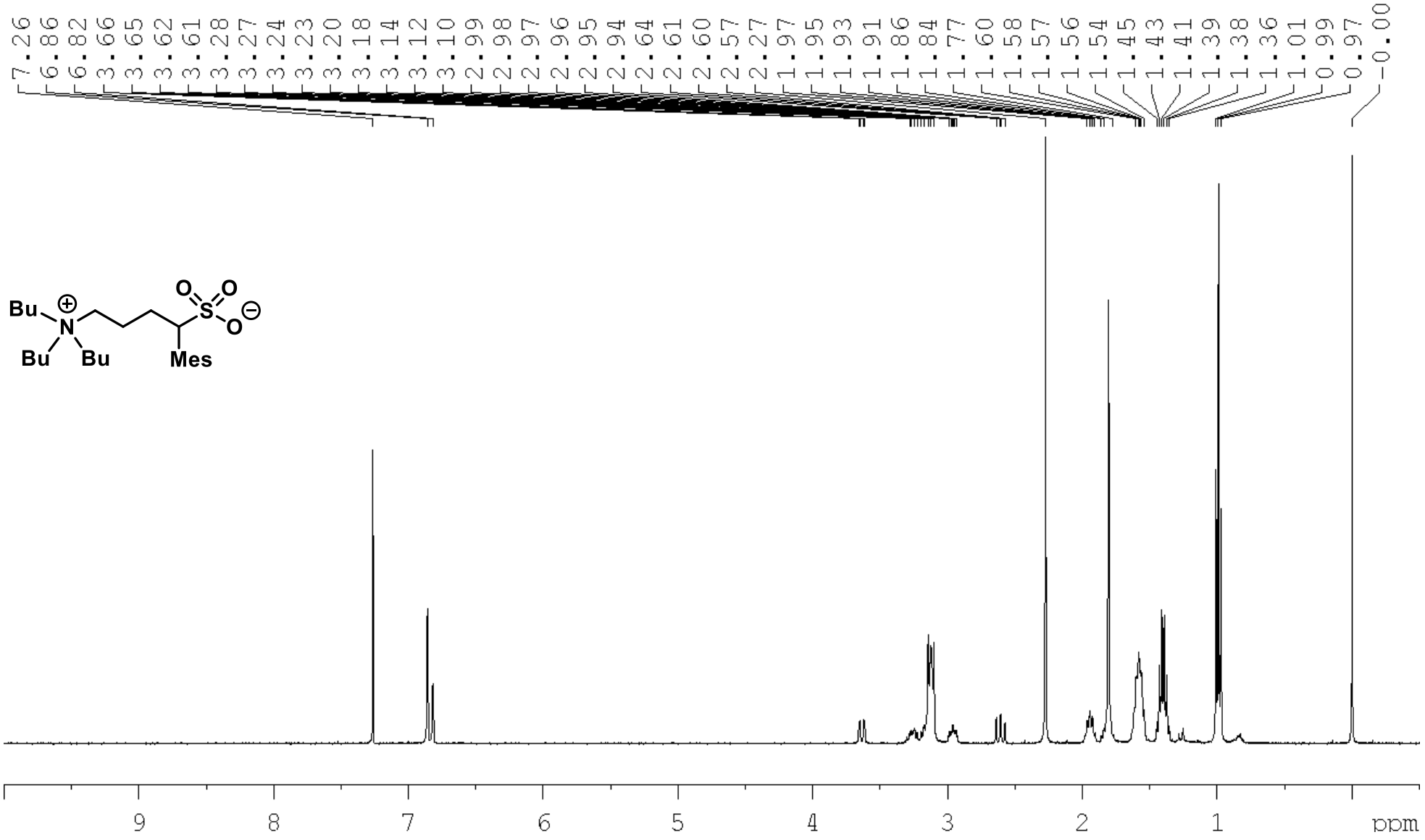
Elemental Composition

Parameters		Elements Set 1:						
Tolerance:	±3.00 ppm	Symbol	C	H	O	N	S	Na
Electron:	Odd/Even	Min	0	0	3	1	1	0
Charge:	+1	Max	400	1000	3	1	1	1
DBE:	-99.0 - 999.0							

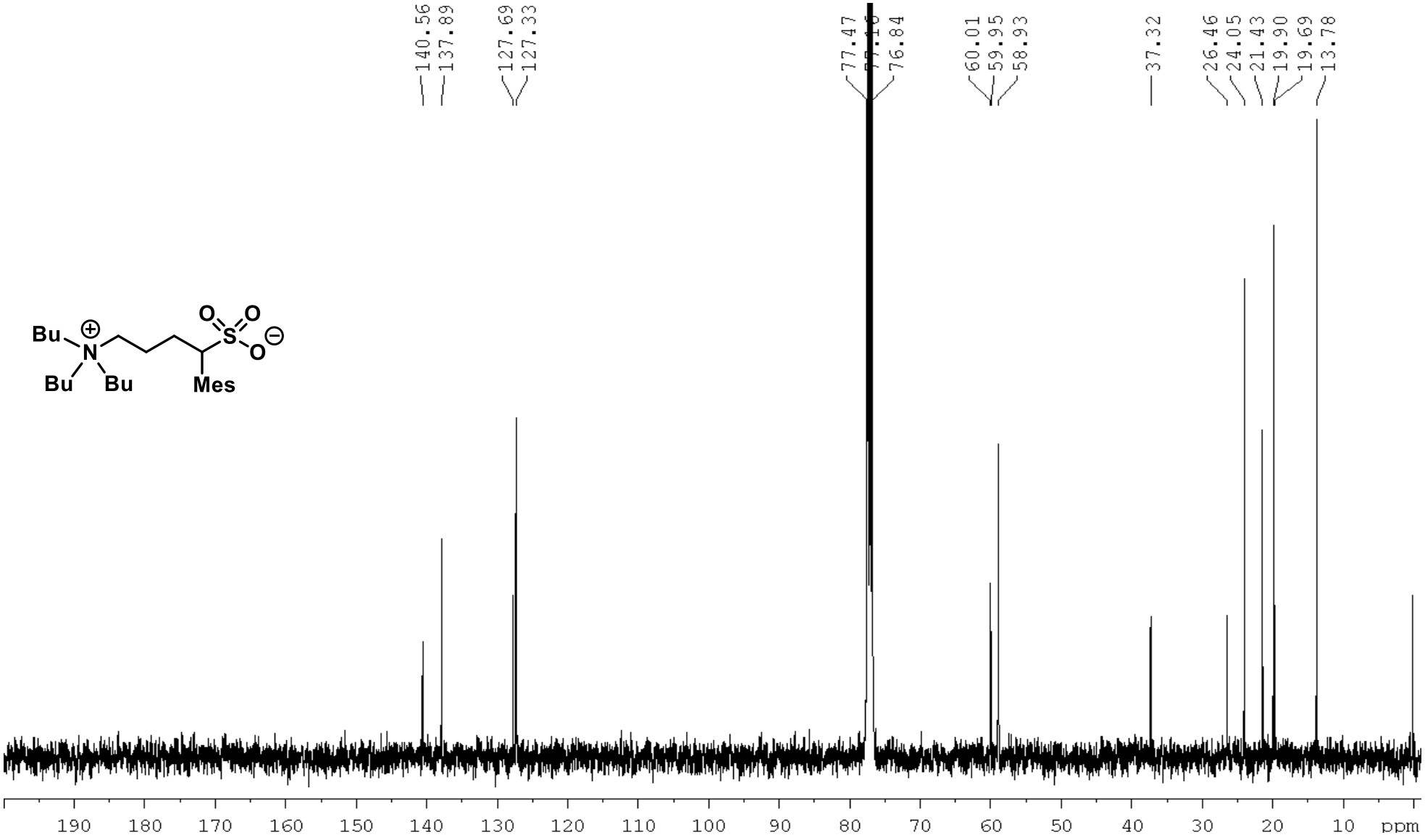
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
398.27150	C22 H40 N O3 S	398.27234	-0.84	-2.11	3.5
420.25362	C22 H39 N O3 Na S	420.25429	-0.67	-1.58	3.5

^{1}H NMR spectrum of **ZM 2c**

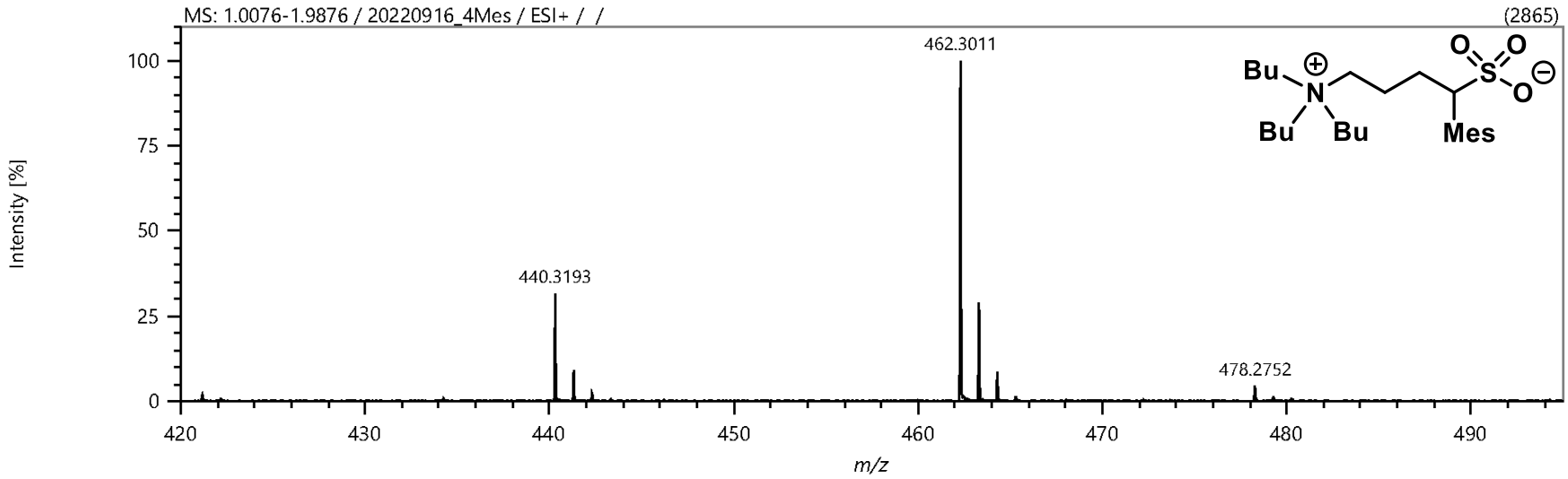


^{13}C NMR spectrum of **ZM 2c**



HRMS spectrum of **ZM 2c**

Spectrum



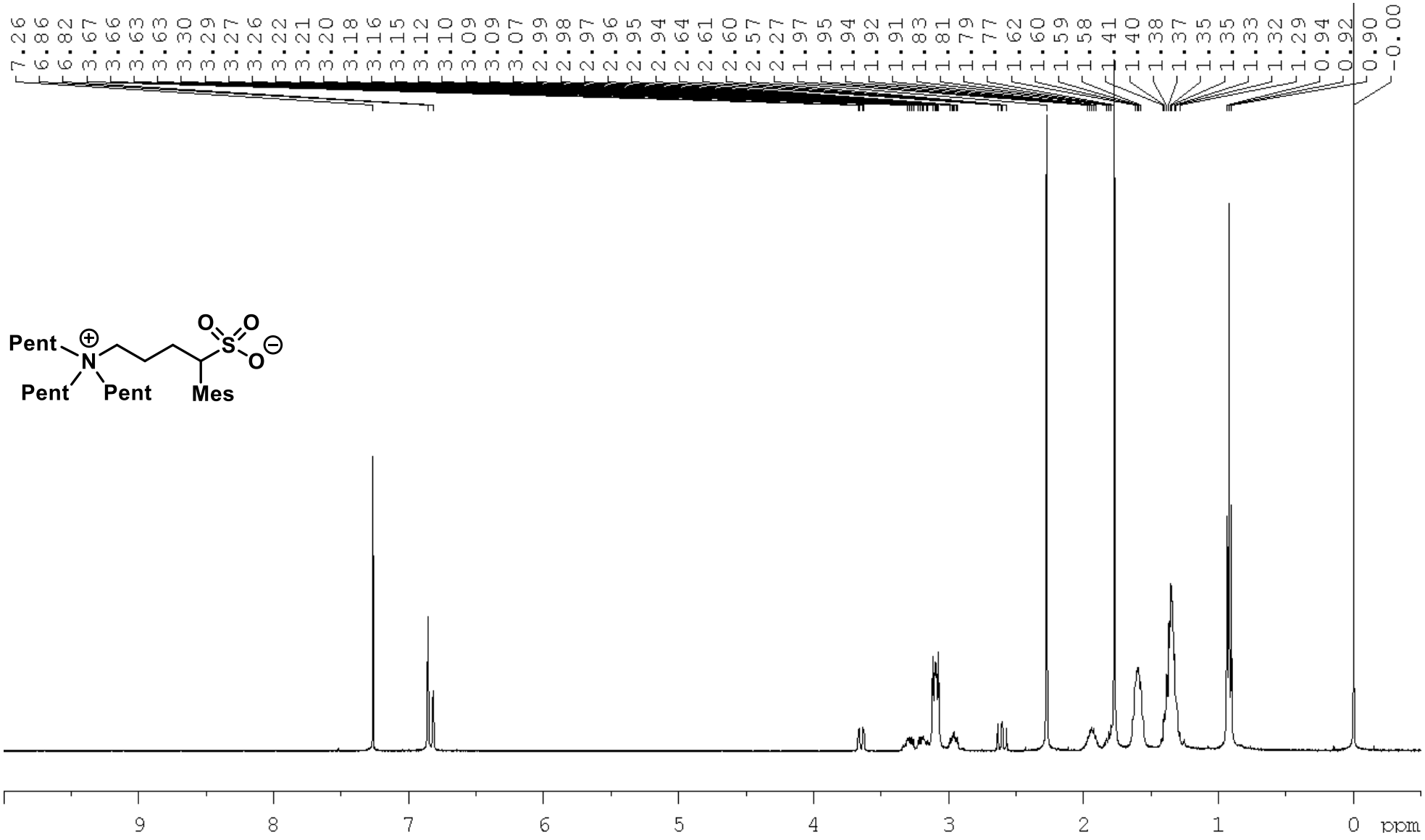
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±3.00 ppm	Symbol	C	H	N	O	S	Na	K
Electron:	Odd/Even	Min	0	0	1	3	1	0	0
Charge:	+1	Max	400	1000	1	3	1	1	1
DBE:	-99.0 - 999.0								

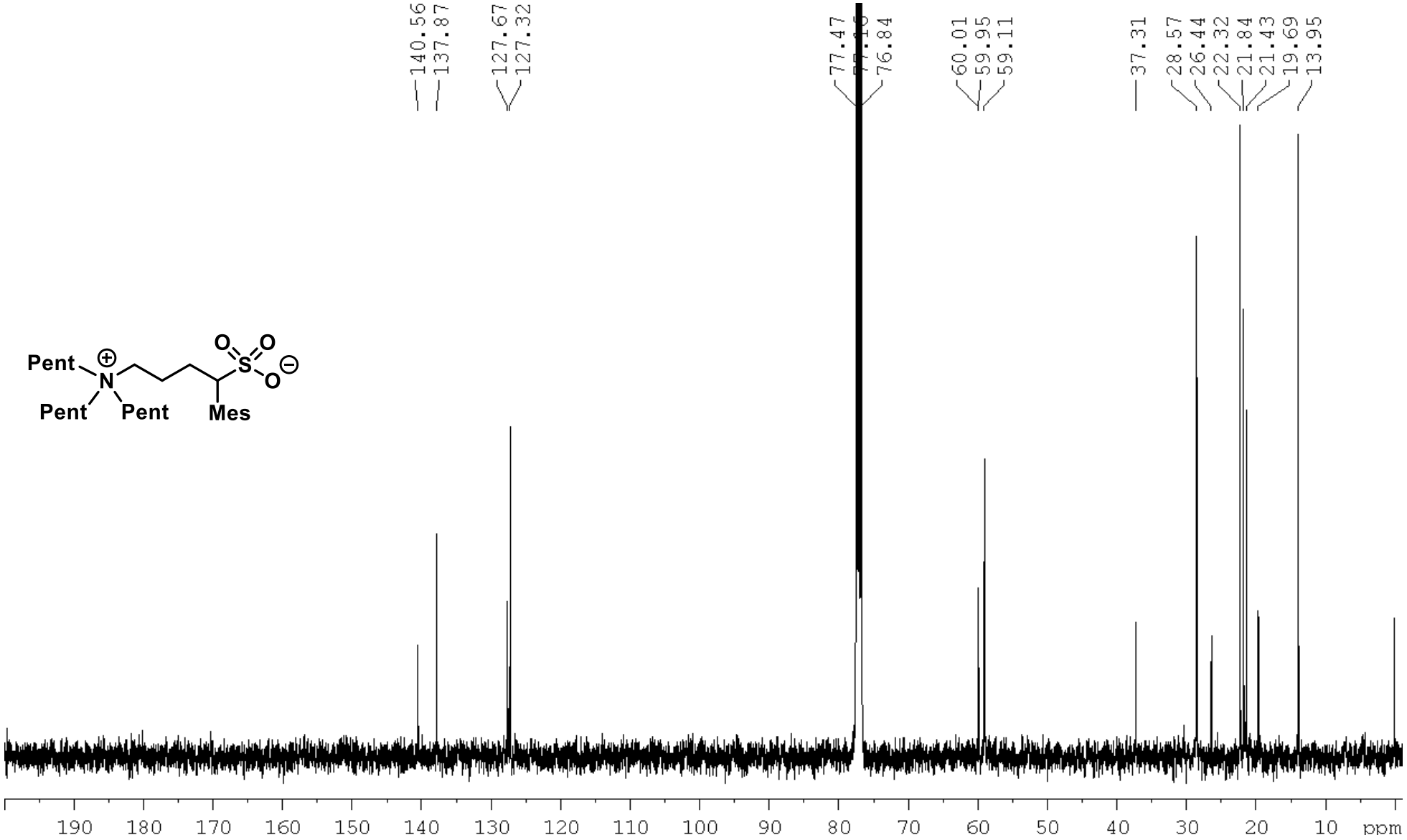
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
440.31929	C25 H46 N O3 S	440.31929	0.00	-0.01	3.5
462.30114	C25 H45 N O3 Na S	462.30124	-0.10	-0.22	3.5
478.27520	C25 H45 N O3 S K	478.27517	0.03	0.06	3.5

^{1}H NMR spectrum of **ZM 2d**

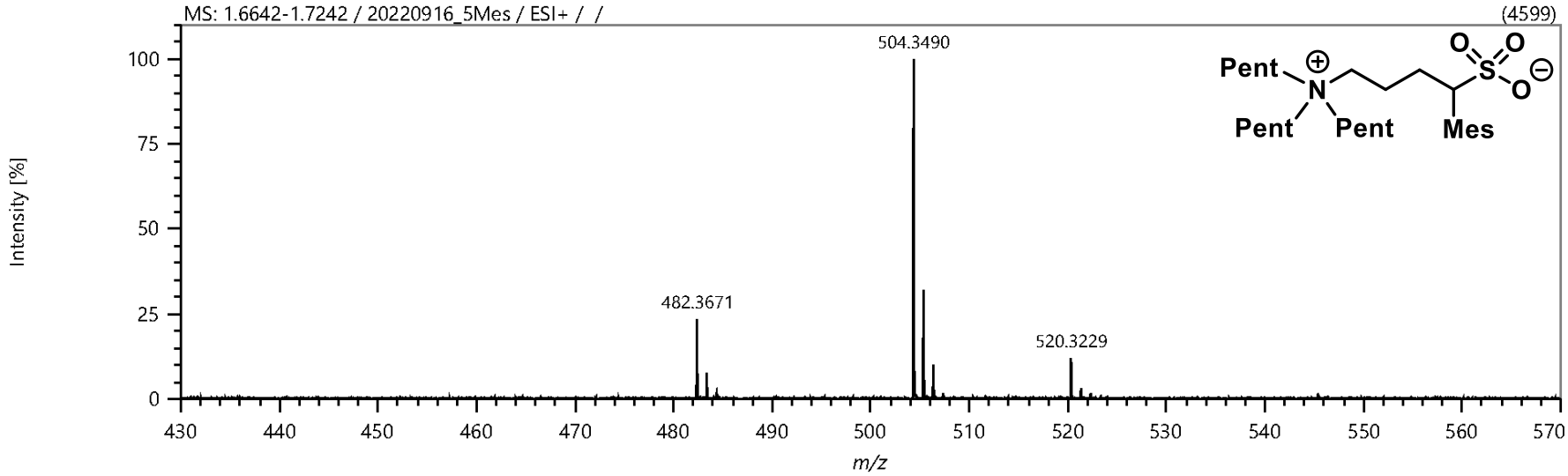


^{13}C NMR spectrum of **ZM 2d**



HRMS spectrum of **ZM 2d**

Spectrum



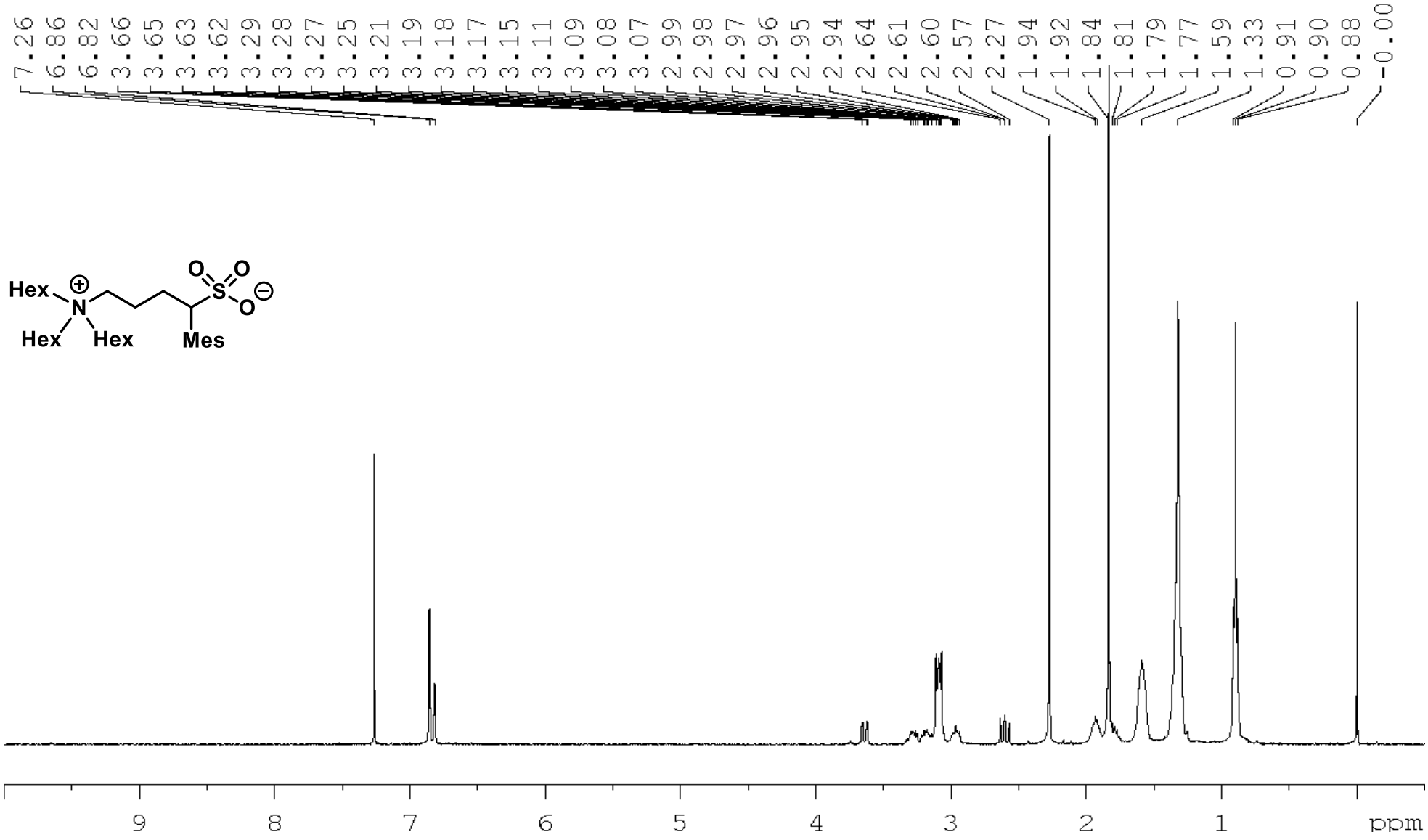
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±3.00 ppm	Symbol	C	H	N	O	S	Na	K
Electron:	Odd/Even	Min	0	0	1	3	1	0	0
Charge:	+1	Max	400	1000	1	3	1	1	1
DBE:	-99.0 - 999.0								

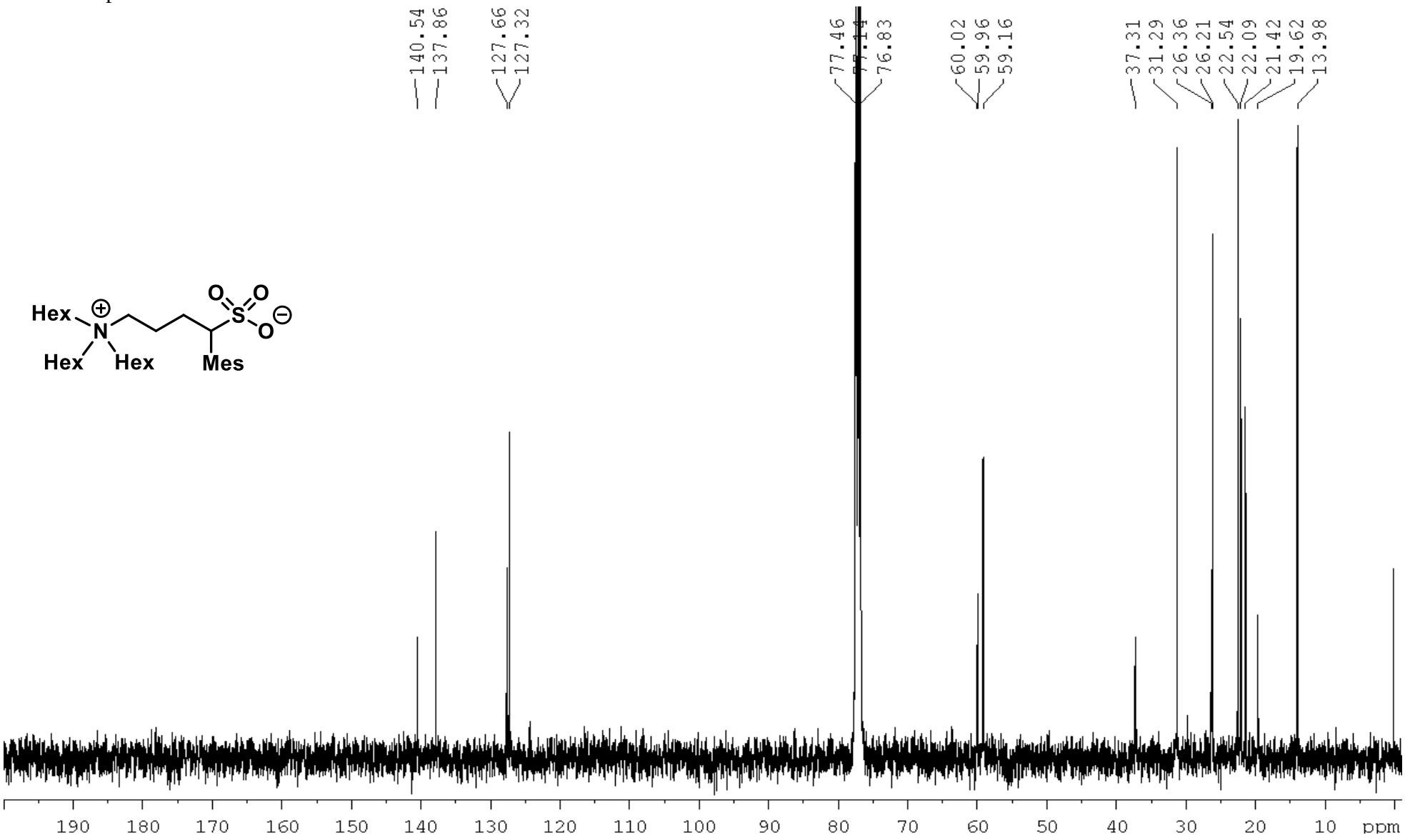
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
482.36712	C28 H52 N O3 S	482.36624	0.88	1.83	3.5
504.34896	C28 H51 N O3 Na S	504.34819	0.77	1.54	3.5
520.32291	C28 H51 N O3 S K	520.32212	0.78	1.50	3.5

^{1}H NMR spectrum of **ZM 2e**

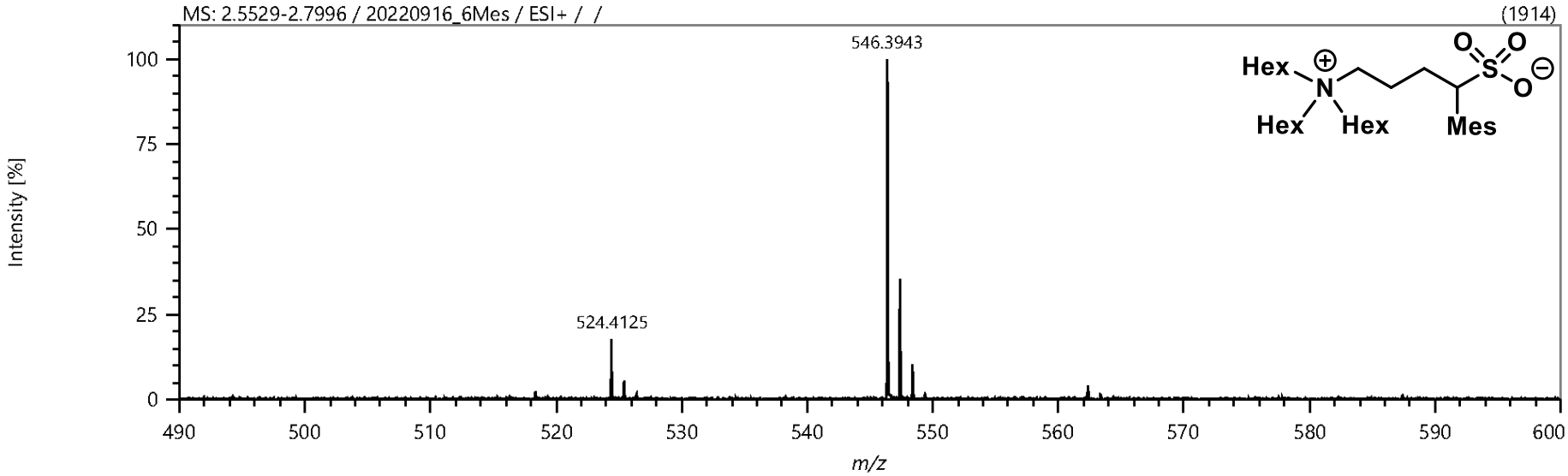


^{13}C NMR spectrum of **ZM 2e**



HRMS spectrum of **ZM 2e**

Spectrum



Elemental Composition

Parameters	
Tolerance:	±3.00 ppm
Electron:	Odd/Even
Charge:	+1
DBE:	-99.0 - 999.0

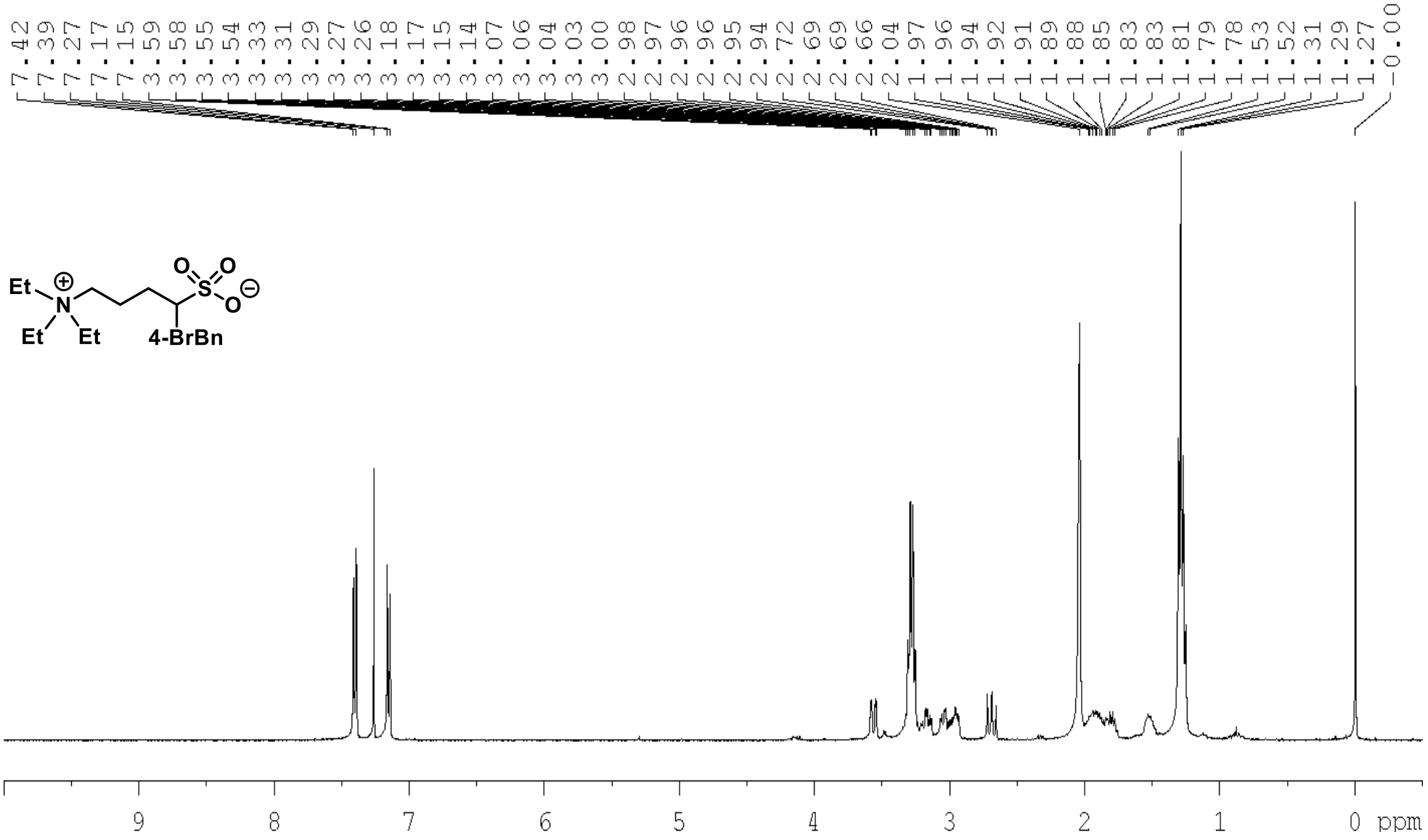
Elements Set 1:

Symbol	C	H	N	O	S	Na	K
Min	0	0	1	3	1	0	0
Max	400	1000	1	3	1	1	1

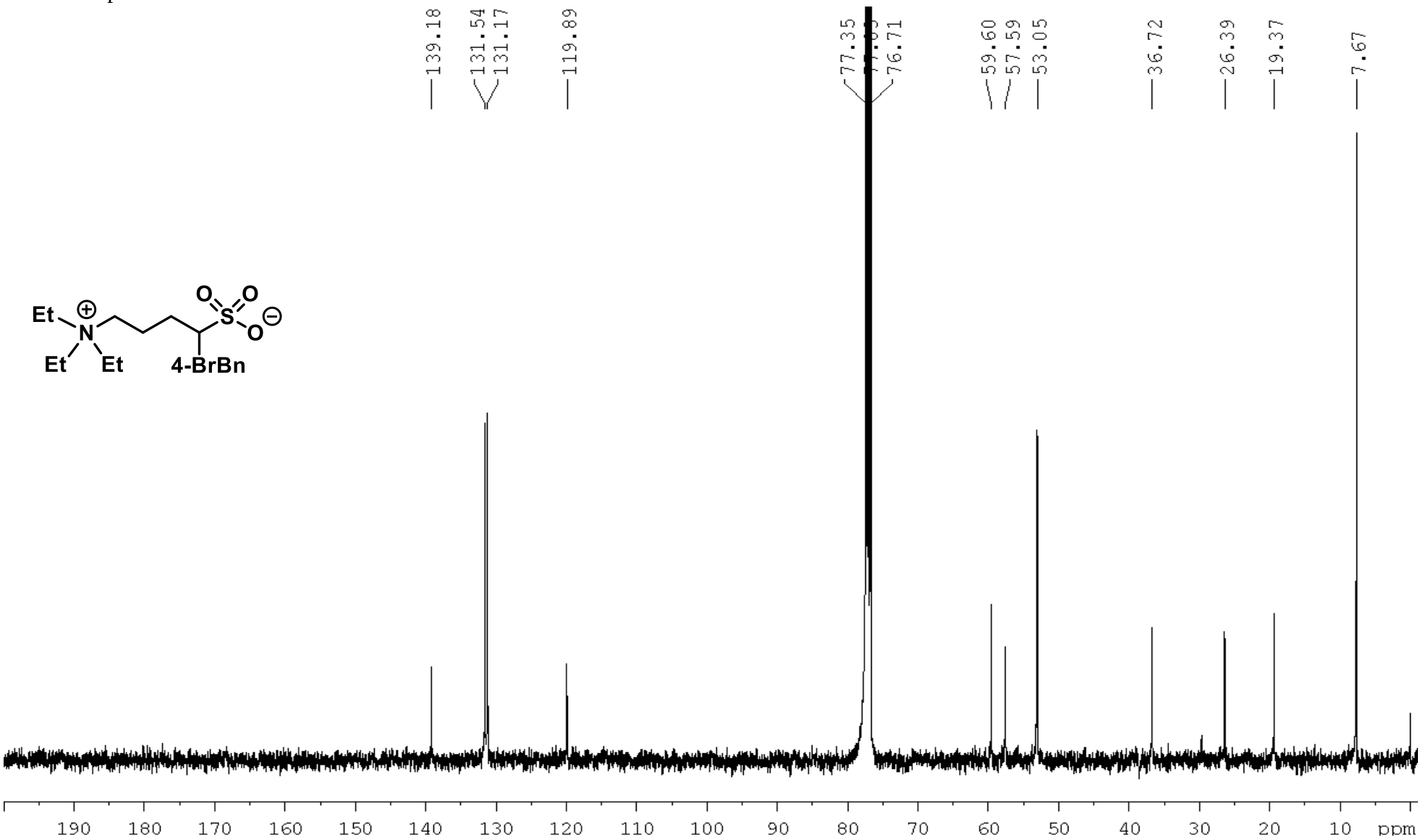
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
524.41245	C31 H58 N O3 S	524.41319	-0.74	-1.41	3.5
546.39432	C31 H57 N O3 Na S	546.39514	-0.82	-1.49	3.5

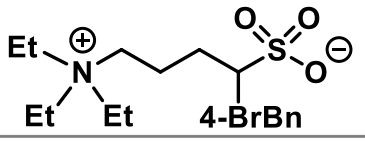
^{1}H NMR spectrum of **ZM 3a**



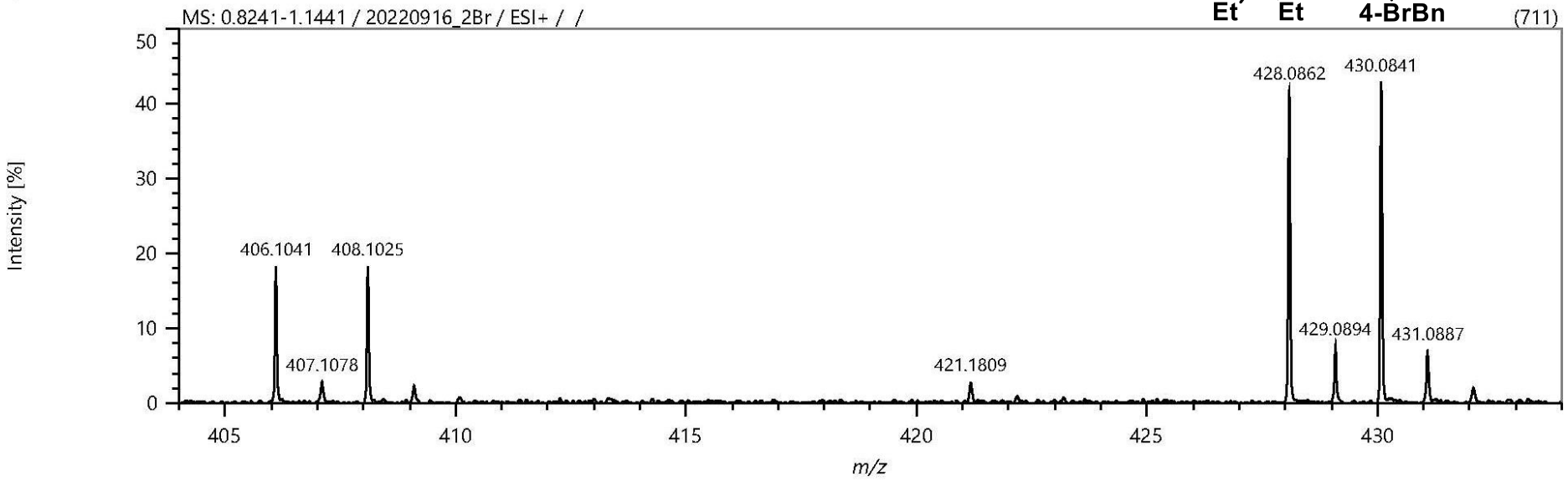
^{13}C NMR spectrum of **ZM 3a**



HRMS spectrum of **ZM 3a**



Spectrum



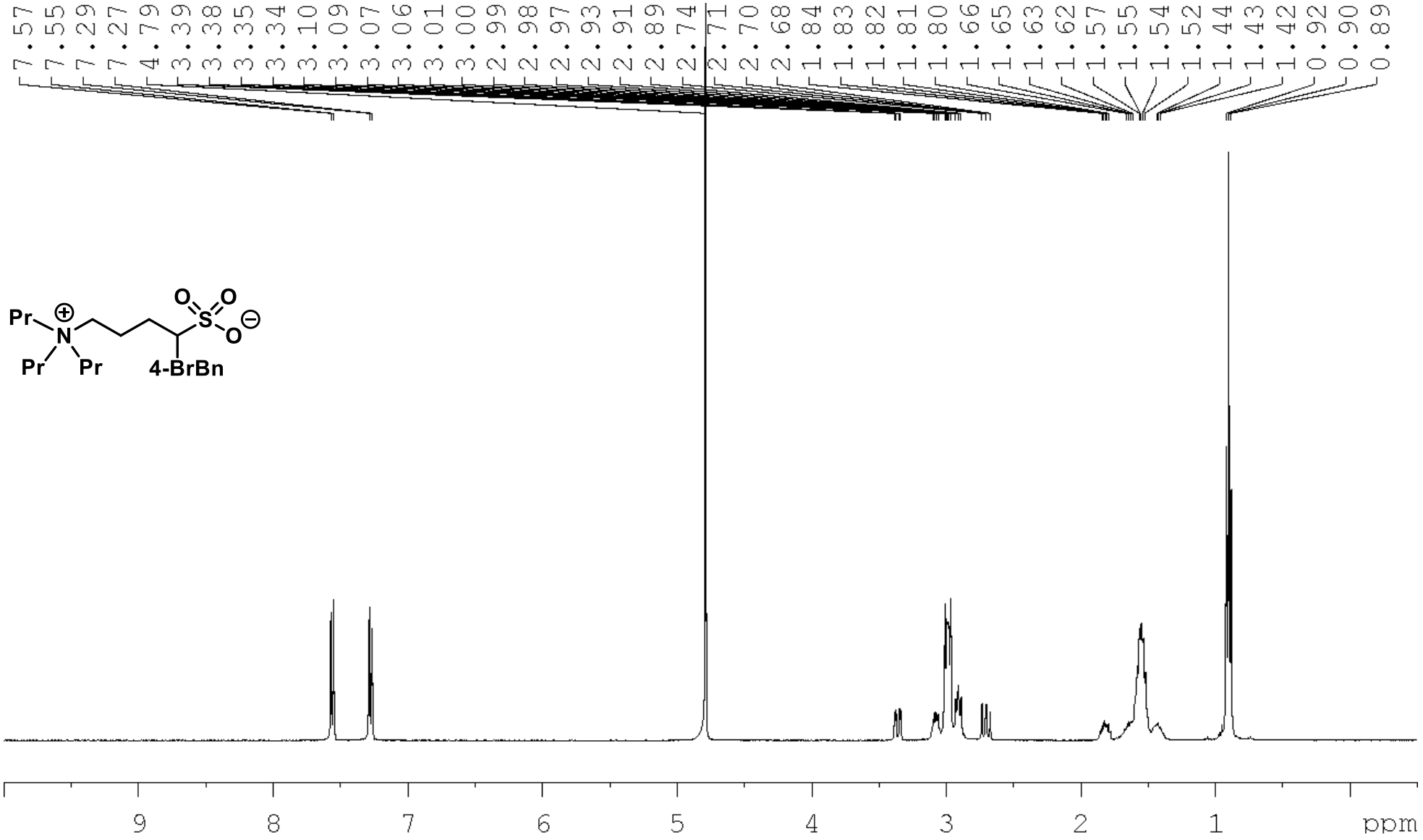
Elemental Composition

Parameters		Elements Set 1:								
Tolerance:	±3.00 ppm	Symbol	C	H	N	O	S	Na	K	Br
Electron:	Odd/Even	Min	0	0	1	3	1	0	0	1
Charge:	+1	Max	400	1000	1	3	1	1	1	1
DBE:	-99.0 - 999.0									

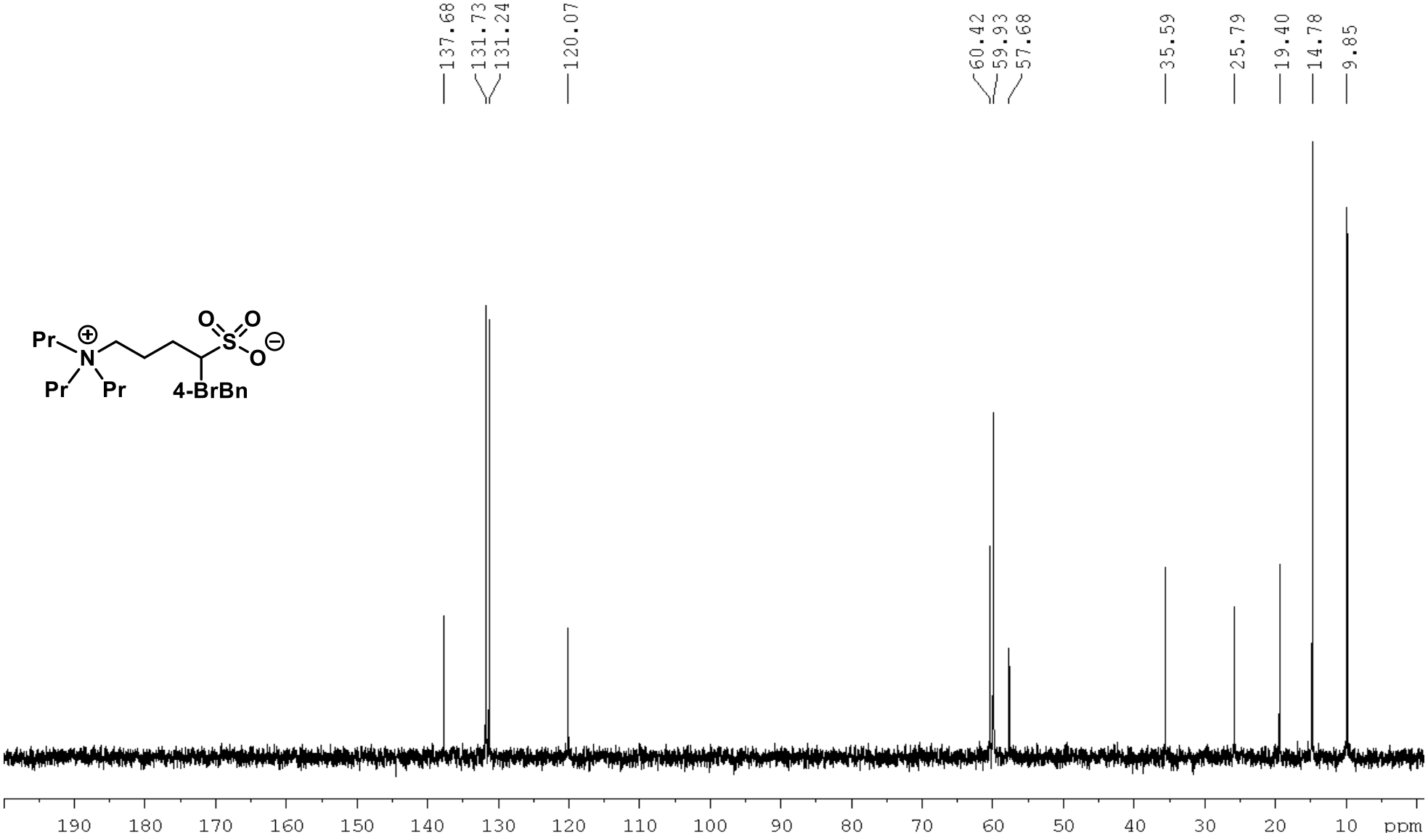
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
406.10410	C17 H29 N O3 S Br	406.10460	-0.50	-1.24	3.5
428.08619	C17 H28 N O3 Na S Br	428.08655	-0.36	-0.83	3.5
444.06108	C17 H28 N O3 S K Br	444.06049	0.59	1.34	3.5

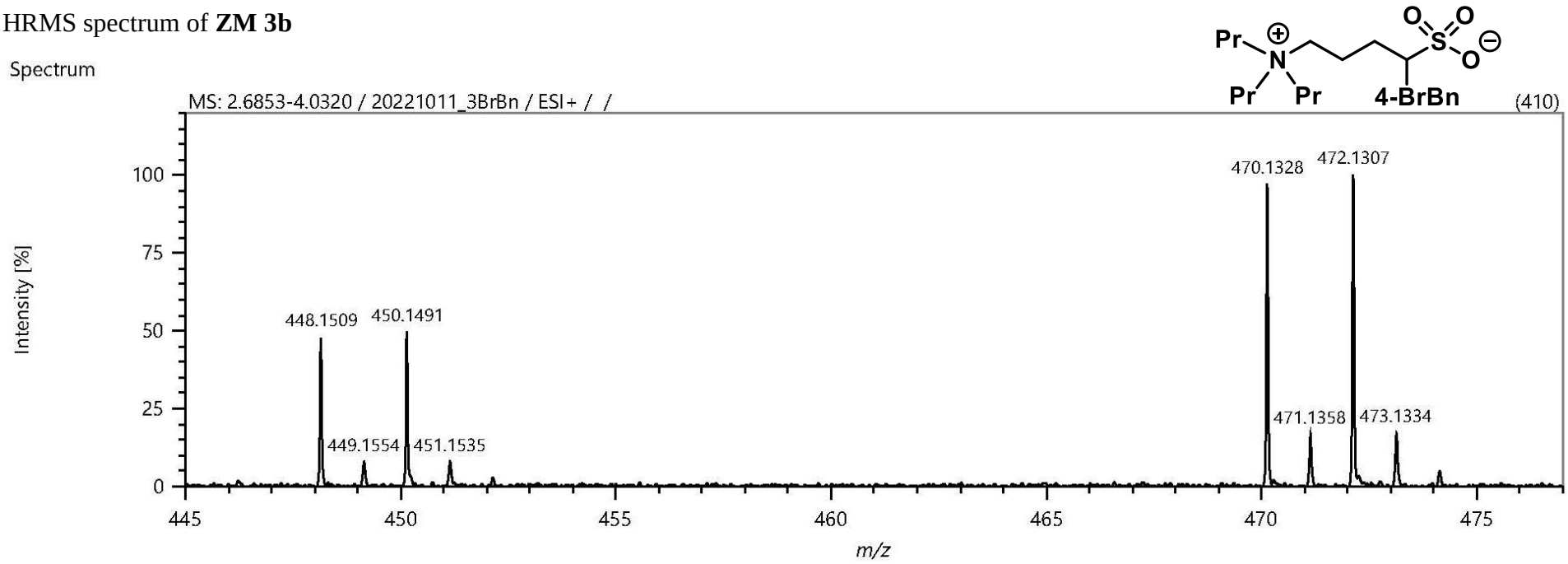
^{1}H NMR spectrum of **ZM 3b**



^{13}C NMR spectrum of **ZM 3b**



HRMS spectrum of **ZM 3b**



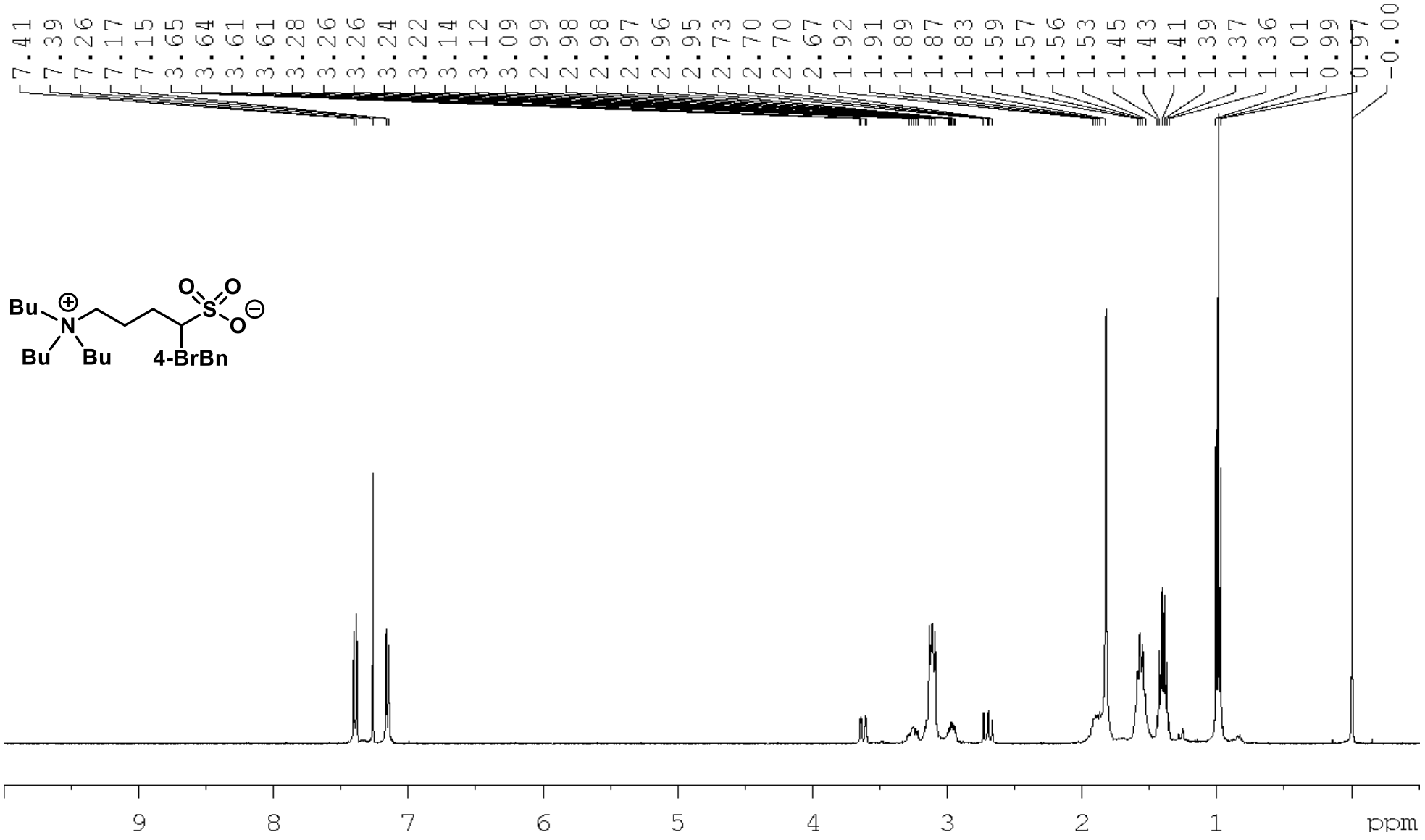
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±3.00 ppm	Symbol	C	H	O	N	S	Na	Br
Electron:	Odd/Even	Min	0	0	3	1	1	0	1
Charge:	+1	Max	400	1000	3	1	1	1	1
DBE:	-99.0 - 999.0								

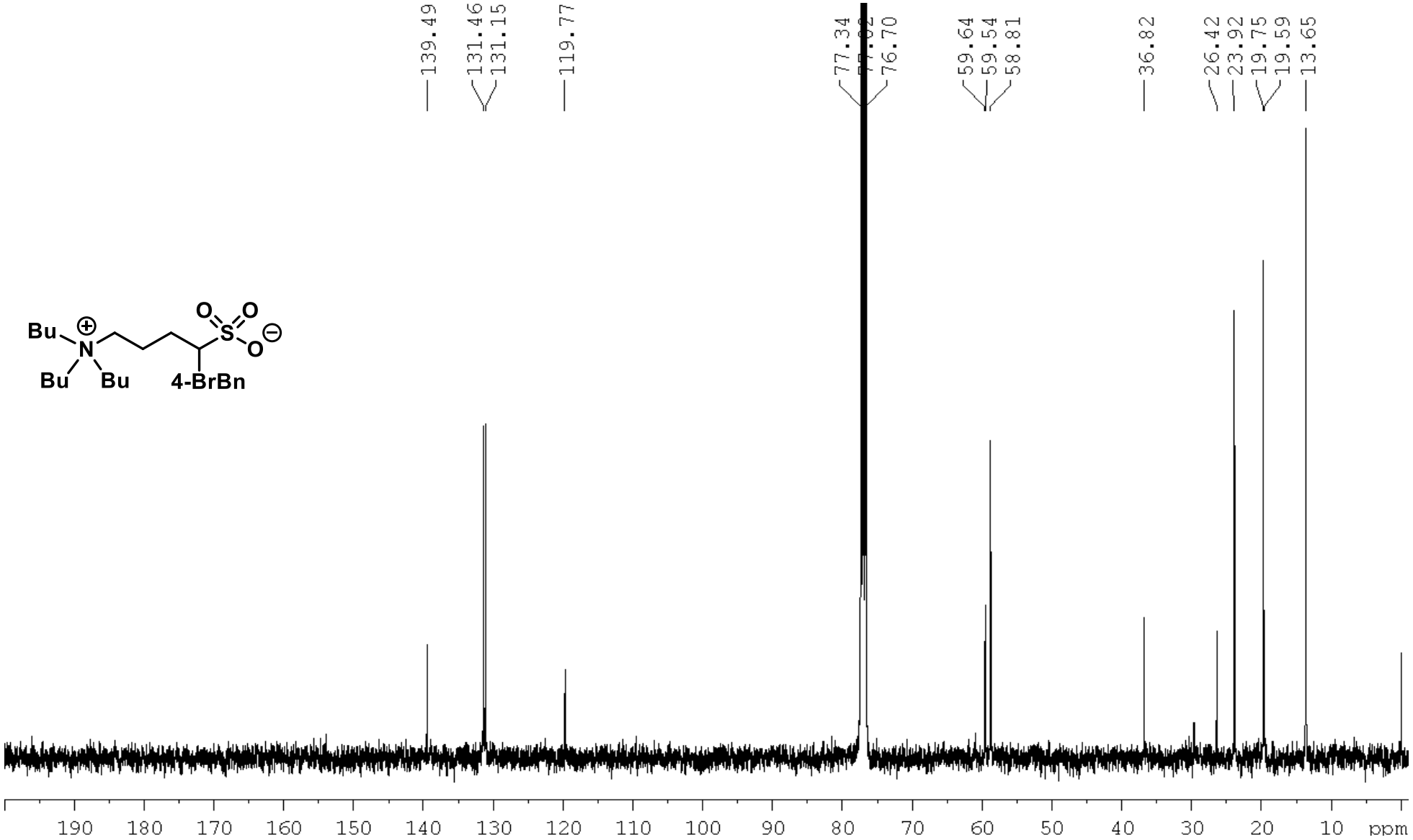
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
448.15092	C20 H35 N O3 S Br	448.15155	-0.63	-1.41	3.5
470.13275	C20 H34 N O3 Na S Br	470.13350	-0.75	-1.59	3.5

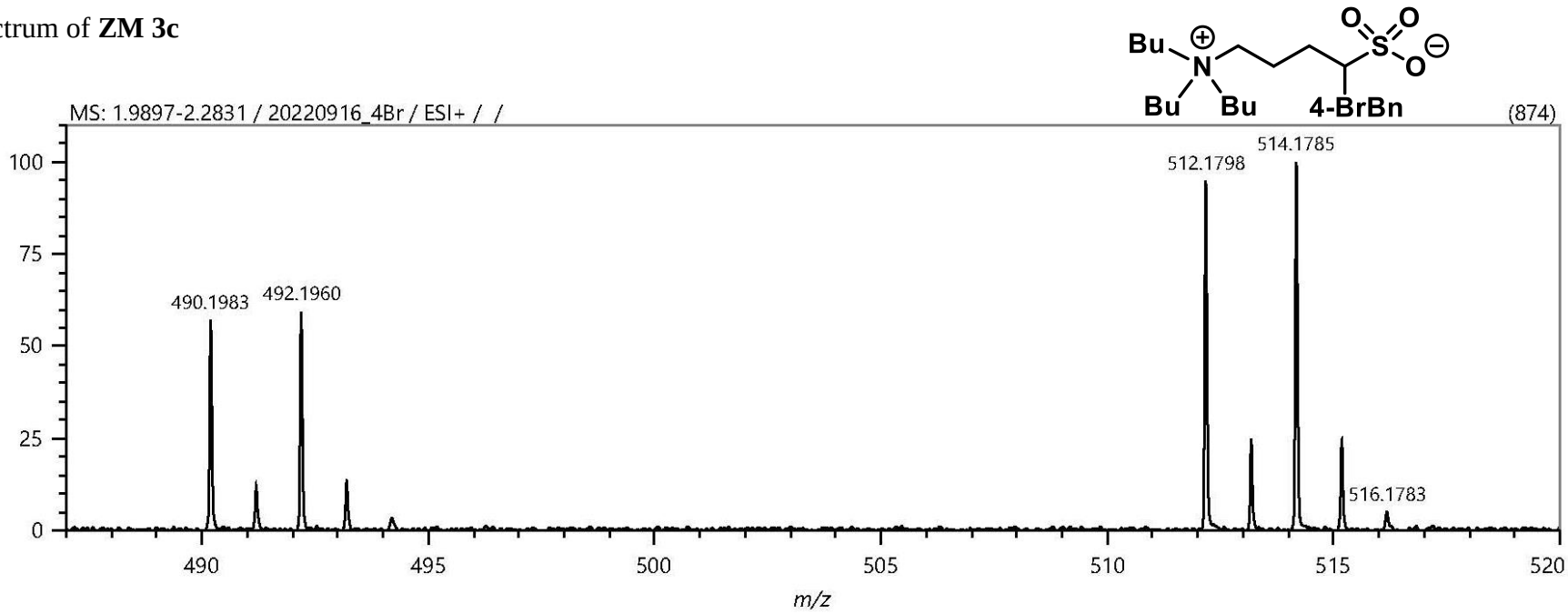
^{1}H NMR spectrum of **ZM 3c**



^{13}C NMR spectrum of **ZM 3c**



HRMS spectrum of **ZM 3c**



Spectrum

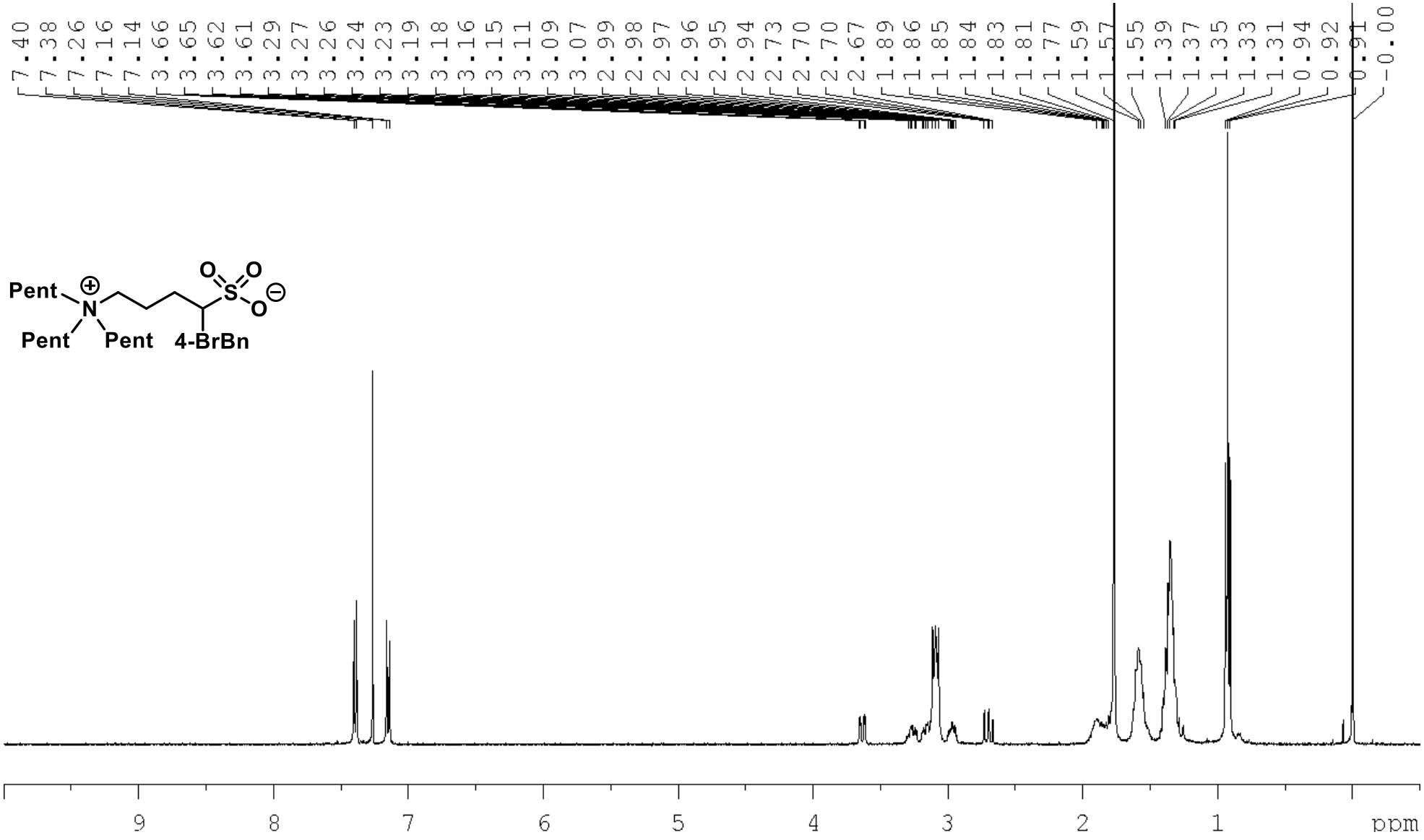
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±3.00 ppm	Symbol	C	H	N	O	S	Na	K	Br
Electron:	Odd/Even	Min	0	0	1	3	1	0	0	1
Charge:	+1	Max	400	1000	1	3	1	1	1	1
DBE:	-99.0 - 999.0									

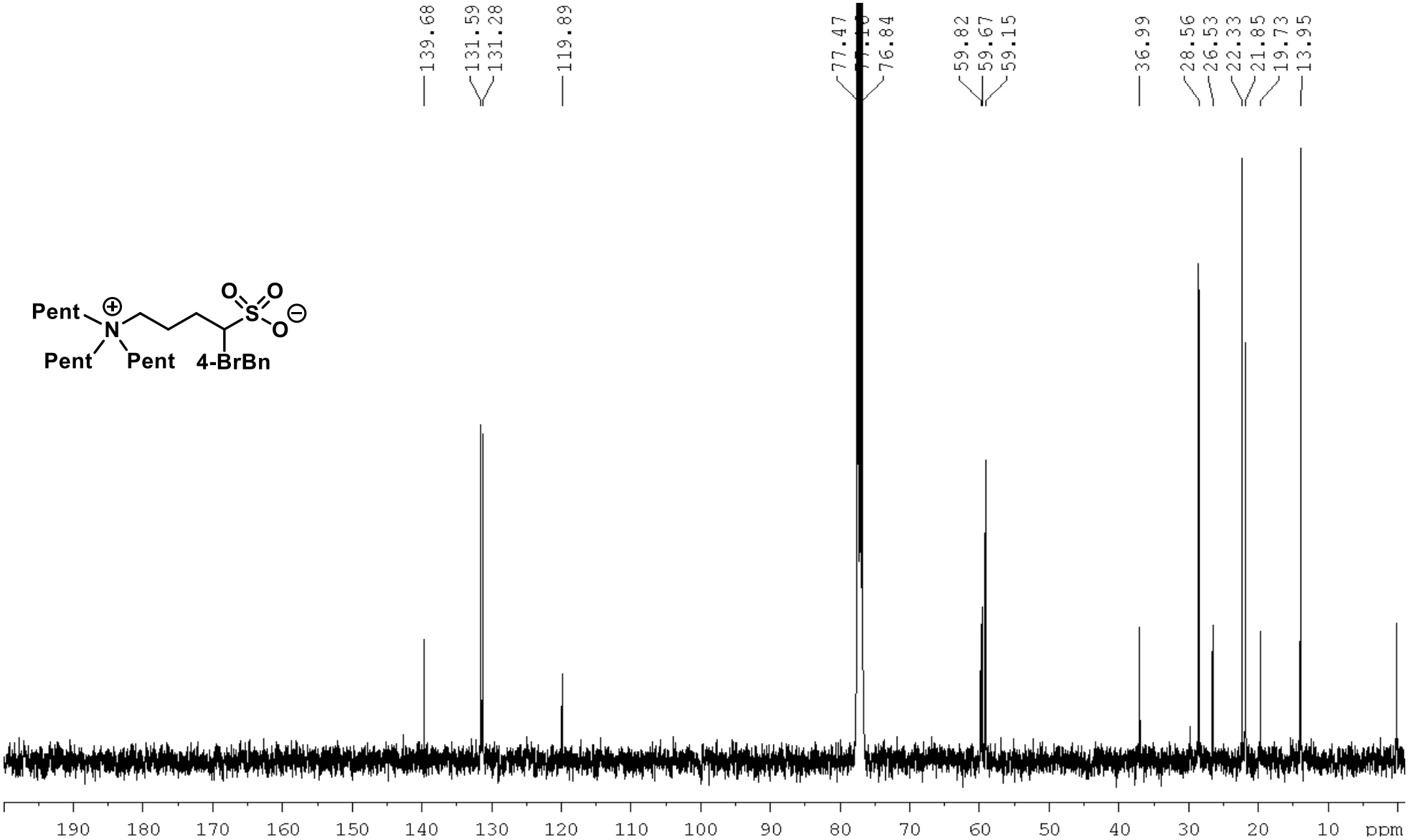
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
490.19831	C23 H41 N O3 S Br	490.19850	-0.19	-0.40	3.5
512.17982	C23 H40 N O3 Na S Br	512.18045	-0.63	-1.23	3.5
528.15495	C23 H40 N O3 S K Br	528.15439	0.56	1.07	3.5

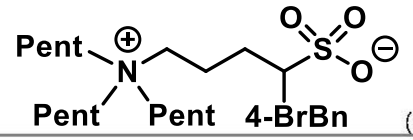
^{1}H NMR spectrum of **ZM 3d**



^{13}C NMR spectrum of **ZM 3d**

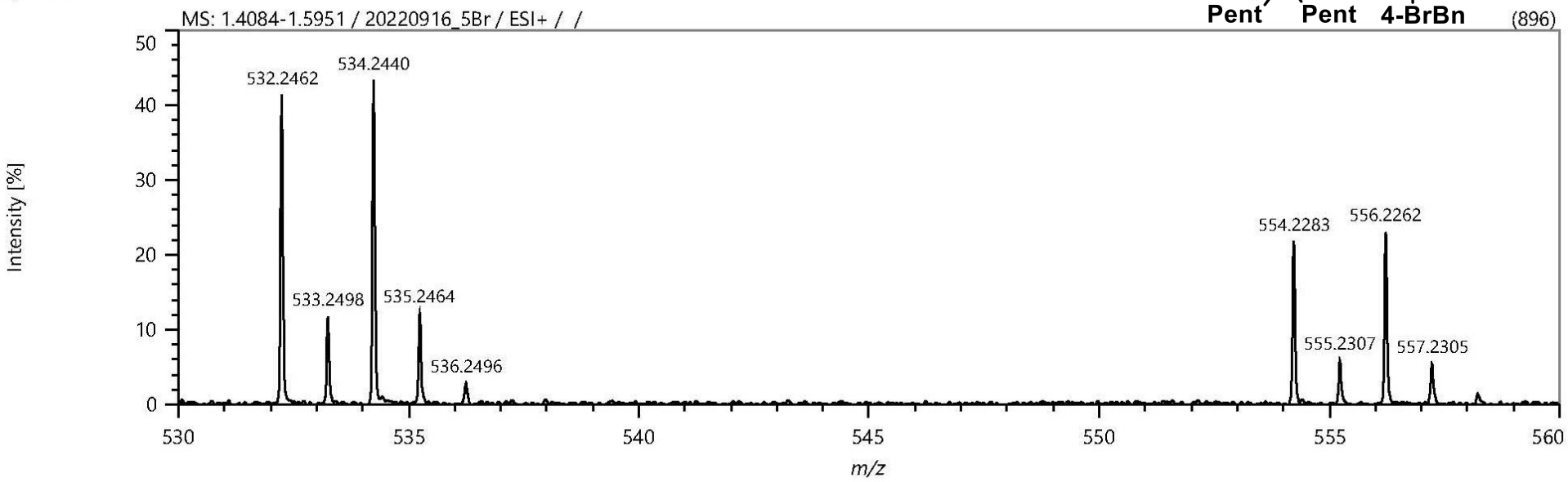


HRMS spectrum of **ZM 3d**



(896)

Spectrum



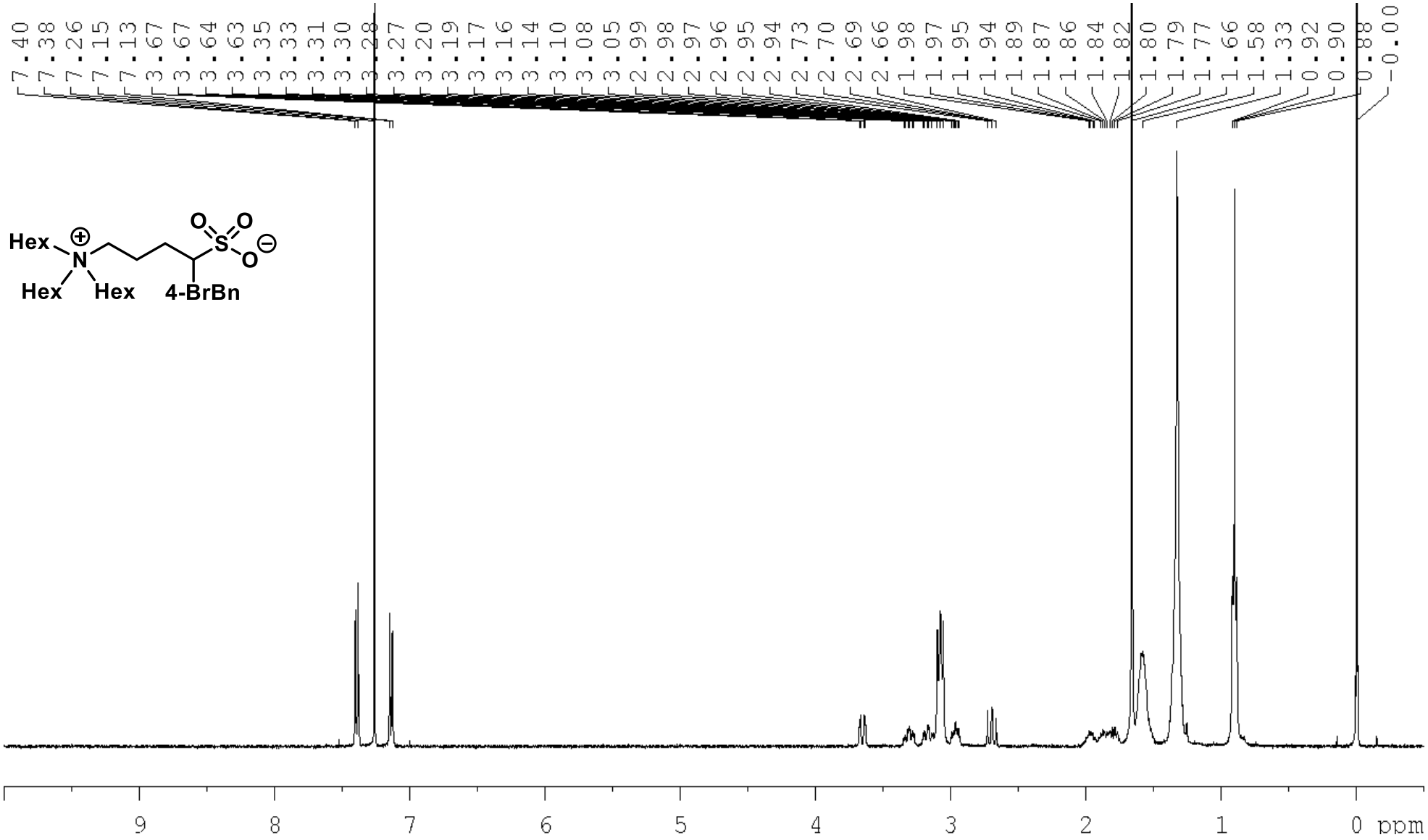
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±2.00 ppm	Symbol	C	H	N	O	S	Na	K	Br
Electron:	Odd/Even	Min	0	0	1	3	1	0	0	1
Charge:	+1	Max	400	1000	1	3	1	1	1	1
DBE:	-99.0 - 999.0									

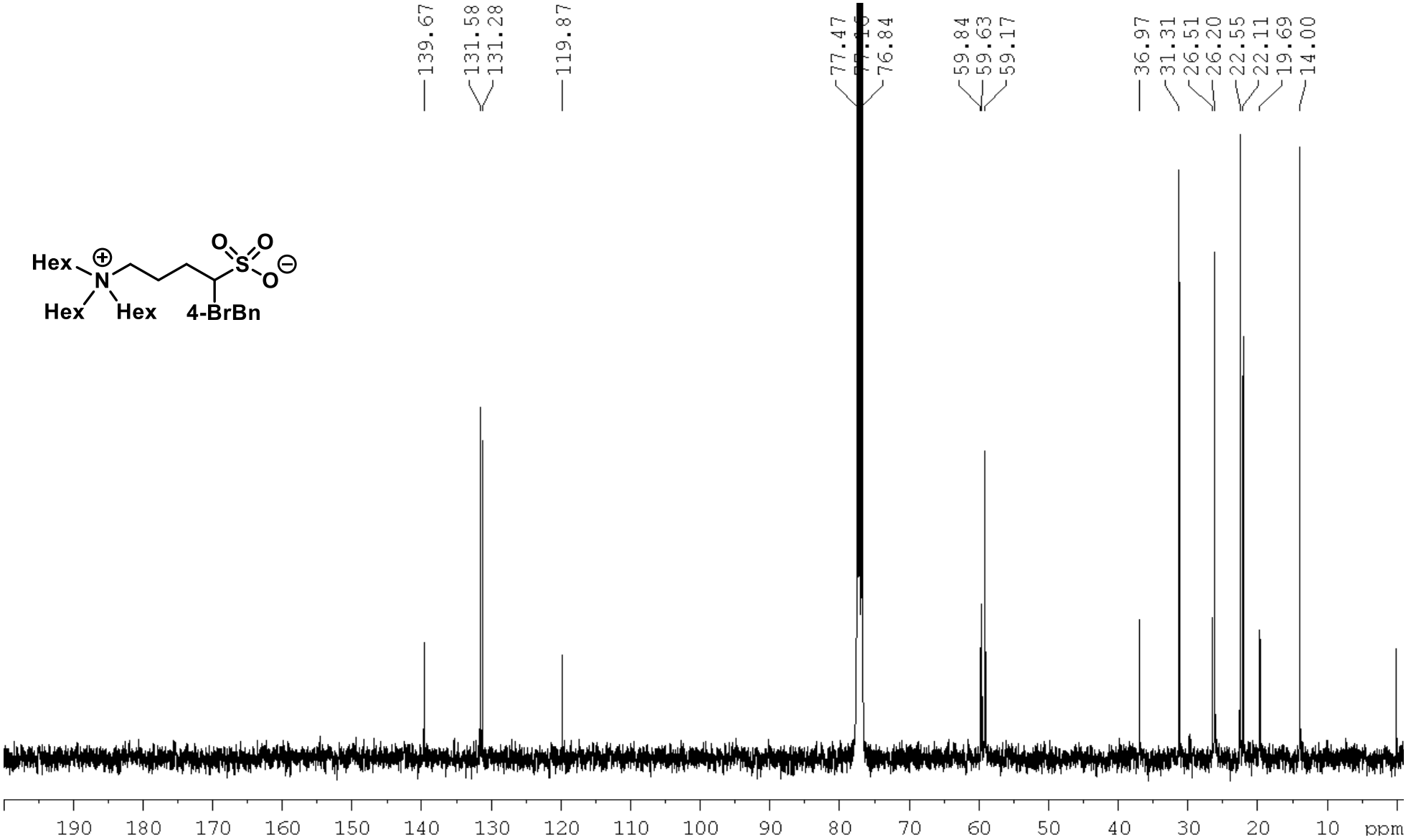
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
532.24620	C26 H47 N O3 S Br	532.24545	0.75	1.40	3.5
554.22831	C26 H46 N O3 Na S Br	554.22740	0.91	1.65	3.5

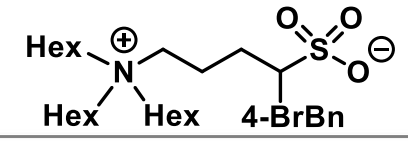
^{1}H NMR spectrum of **ZM 3e**



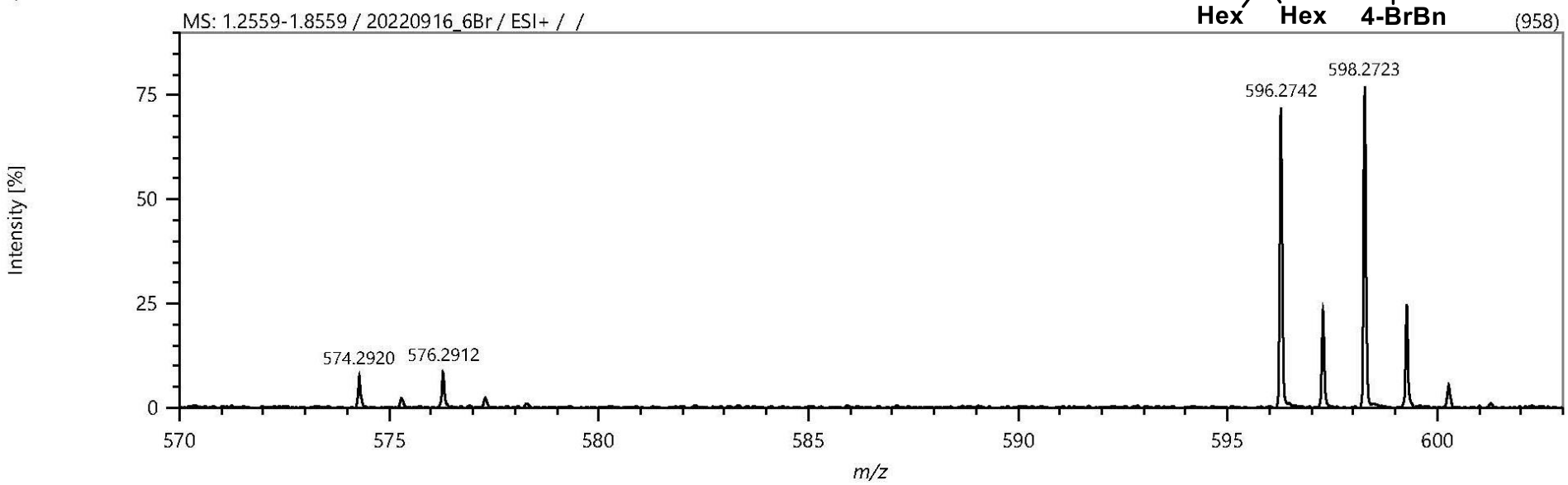
^{13}C NMR spectrum of **ZM 3e**



HRMS spectrum of **ZM 3e**



Spectrum



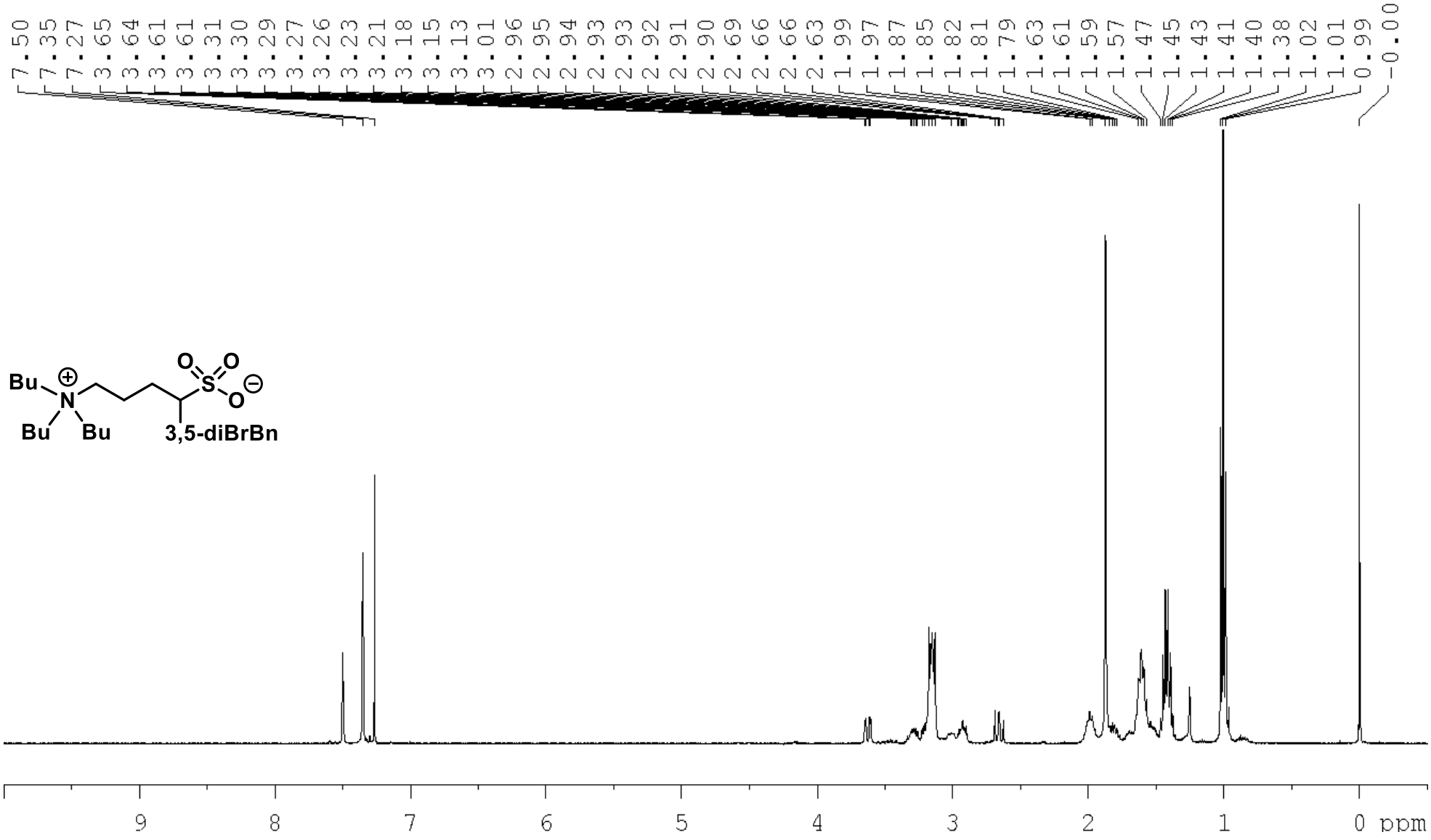
Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±3.00 ppm	Symbol	C	H	N	O	S	Na	K	Br
Electron:	Odd/Even	Min	0	0	1	3	1	0	0	1
Charge:	+1	Max	400	1000	1	3	1	1	1	1
DBE:	-99.0 - 999.0									

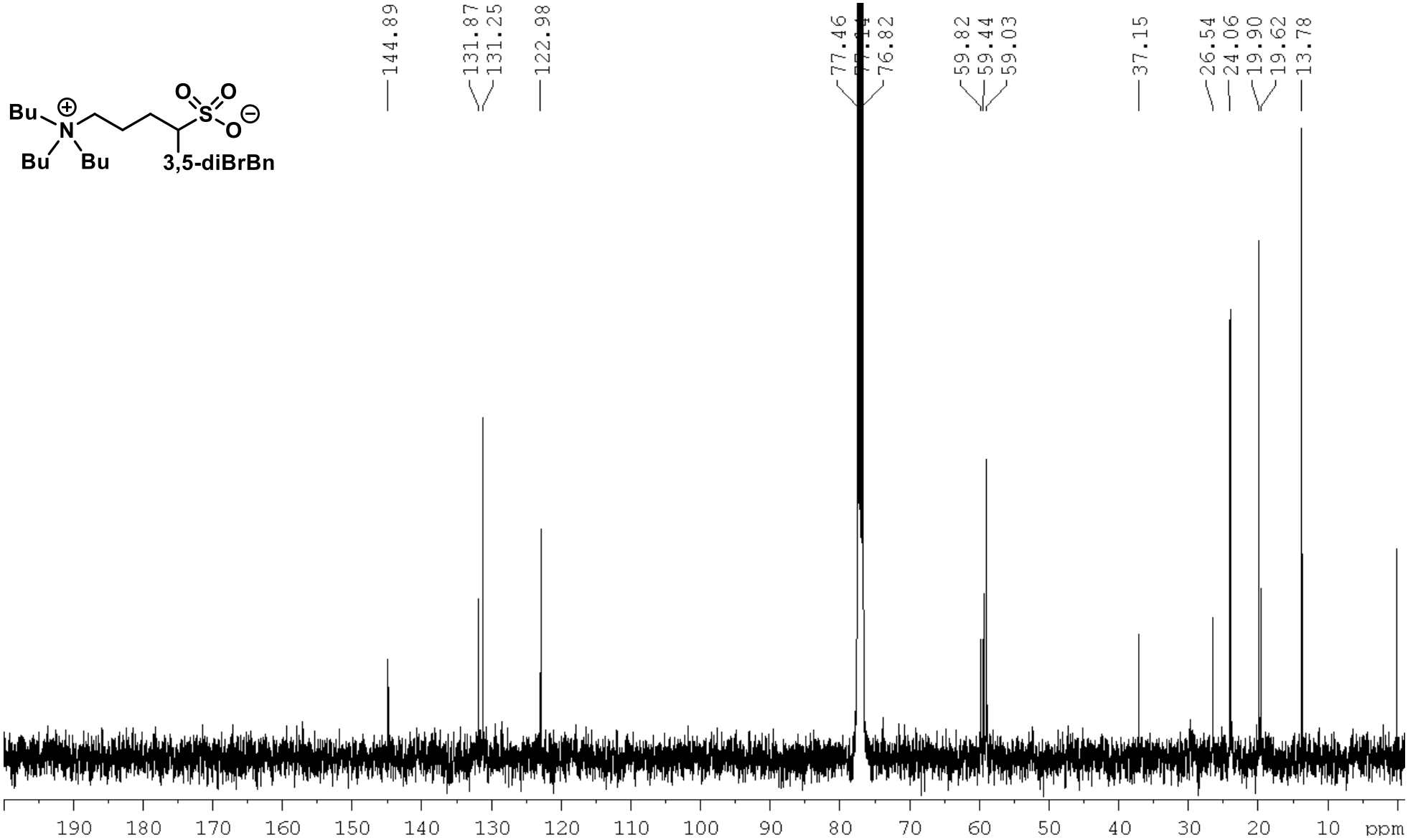
Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
574.29197	C29 H53 N O3 S Br	574.29240	-0.43	-0.76	3.5
596.27419	C29 H52 N O3 Na S Br	596.27435	-0.16	-0.27	3.5
612.24862	C29 H52 N O3 S K Br	612.24829	0.33	0.55	3.5

^{1}H NMR spectrum of **ZM 4c**

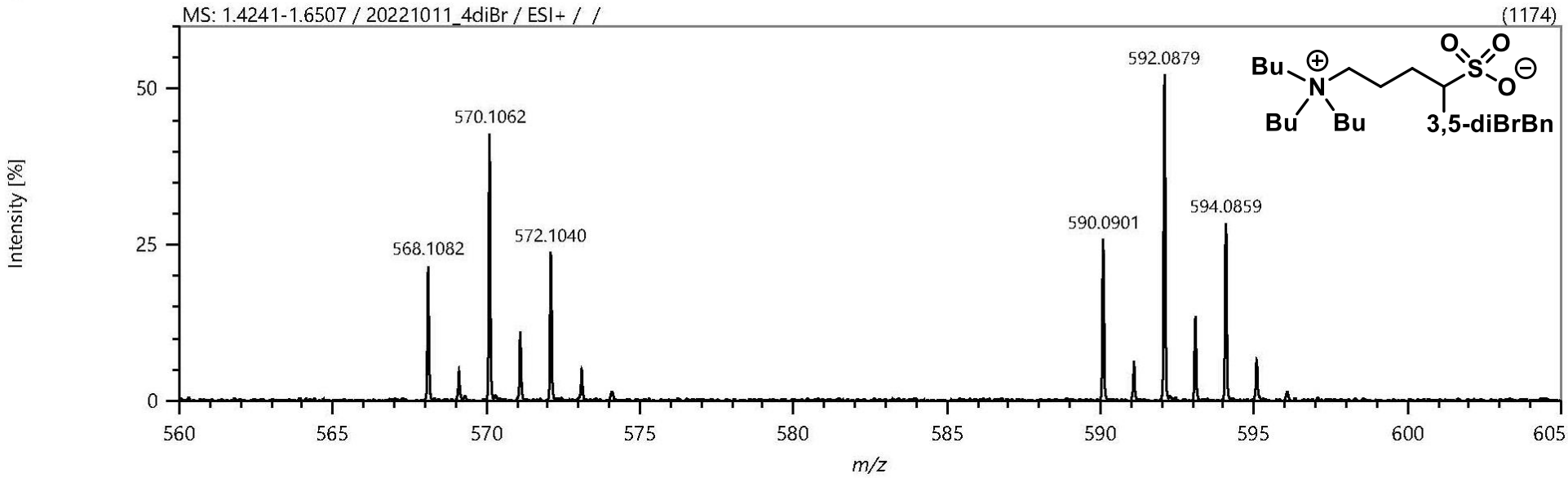


^{13}C NMR spectrum of **ZM 4c**



HRMS spectrum of **ZM 4c**

Spectrum



Elemental Composition

Parameters		Elements Set 1:							
Tolerance:	±2.00 ppm	Symbol	C	H	O	N	Br	S	Na
Electron:	Odd/Even	Min	0	0	3	1	2	1	0
Charge:	+1	Max	400	1000	3	1	2	1	1
DBE:	-99.0 - 999.0								

Results

Mass	Formula	Calculated Mass	Mass Difference [mDa]	Mass Difference [ppm]	DBE
568.10818	C23 H40 N O3 S Br2	568.10902	-0.84	-1.48	3.5
590.09006	C23 H39 N O3 Na S Br2	590.09096	-0.90	-1.53	3.5