Supporting Information for:

Water-Driven Ligations Using Cyclic Amino Squarates: Making SN1-Like Reactions Useful

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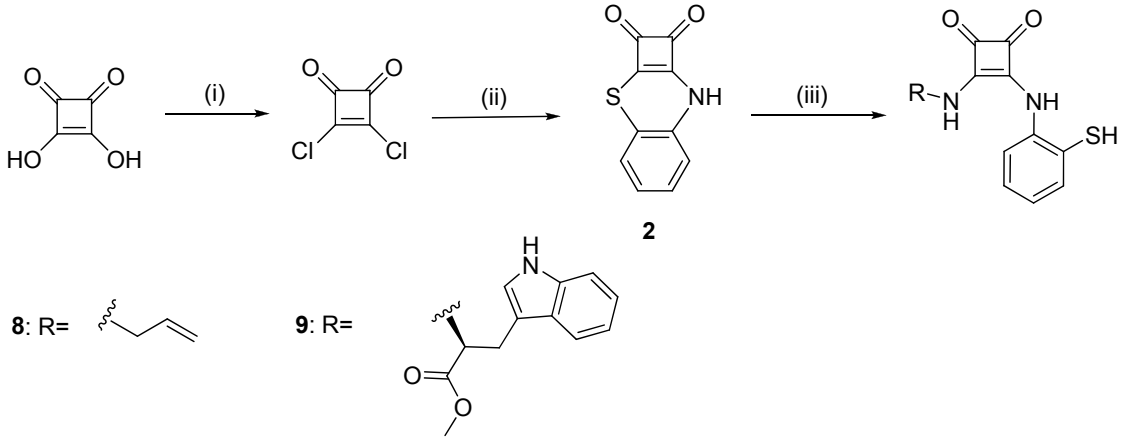
Contents

1. General Information of the synthesis and characterization of the new compounds.

General Experimental Methods. ^{1}H NMR and ^{13}C NMR spectra were recorded on a 300 MHz spectrometer and the chemical shifts were measured from the solvent peak as an internal standard. Chemical shifts and coupling constants (*J*-value) are reported in ppm and Hertz respectively. The following abbreviations are used for spin multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. High Resolution Mass spectra (HRMS) samples were analyzed by positive ion electrospray. Analytical thin-layer chromatography (TLC) was performed using silica gel 60 F254 pre-coated plates (0.25-mm thickness). Preparative TLC separations were made on precoated analytical plates, 0.50 mm thick, silica gel 60 F254. Flash column chromatography was performed with Silica-P Flash Silica Gel (ultra pure 40-63μm). Reagents and solvents purchased from Aldrich Chemical Co., Acros Organics, VWR International Inc., Pfaltz & Bauer and Pharmco and used without any further purification. All reactions were carried out in oven-dried glassware under argon or nitrogen atmosphere unless otherwise specified.

1.1 Synthesis of 1H-benzo[e]cyclobuta[b][1,4]thiazine-1,2(8H)-dione (**2**)



Reagents and conditions: (i) oxalyl chloride, DMF, CCl_4, -78^{o}C to reflux, 3hr; (ii) 2-aminothiophenol, TEA, benzene, 0^{o}C to reflux, 16hr; (iii) RNH_2, TEA, THF, r.t., 16hr.

(1) Synthesis of **2**

Finely powdered 1,2-Dihydroxycyclobutene-3,4-dione (0.57 g, 5 mmol), carbon tetrachloride (10 mL), and DMF (0.05mL), were introduced into a 25 mL round-bottomed flask previously filled with argon. The mixture was frozen by immersion in dry ice-acetone and oxalyl chloride (0.85 mL, 10 mmol) was introduced at once through the condenser's outlet. Then the flask was allowed to warm to room temperature, agitation started at 300 rpm, and the flask was then lowered into an oil bath thermostatted to 50-60 ^{o}C. Regular gas evolution started immediately and was no more apparent after 50 min. After further 2hr, heating and agitation were discontinued. The solvent was removed to give 1,2-dichlorocyclobutene -3,4-dione (0.6g) as yellow crystal.

1,2-Dichlorocyclobutene-3,4-dione (0.6g) was dissolved in dry benzene (50mL) and cooled down to 0 ^{0}C under argon atmosphere. 1 eq of thiophenol was added in one portion, followed by addition of excess of triethylamine (2.2eq) in anhydrous benzene (5mL). Reaction mixture was stirred at 0^{o}C for 0.5hr, at r.t. for 0.5hr and at reflux overnight. The mixture was filtered and the solid was washed with water and methanol to give a yellow solid. Yield: 0.45g (55%). ^{1}HNMR (300 MHz, DMSO-d_6) : δ 7.35-6.89 (m, 3H), 6.73-6.70 (m, 1H). ^{13}CNMR (75 MHz, DMSO-d_6) : δ 183.84, 183.39, 179.66, 164.62, 134.48, 129.57, 128.39, 126.63, 119.14, 115.03. MS: cacld. for M^{+}: 203, found: 203.

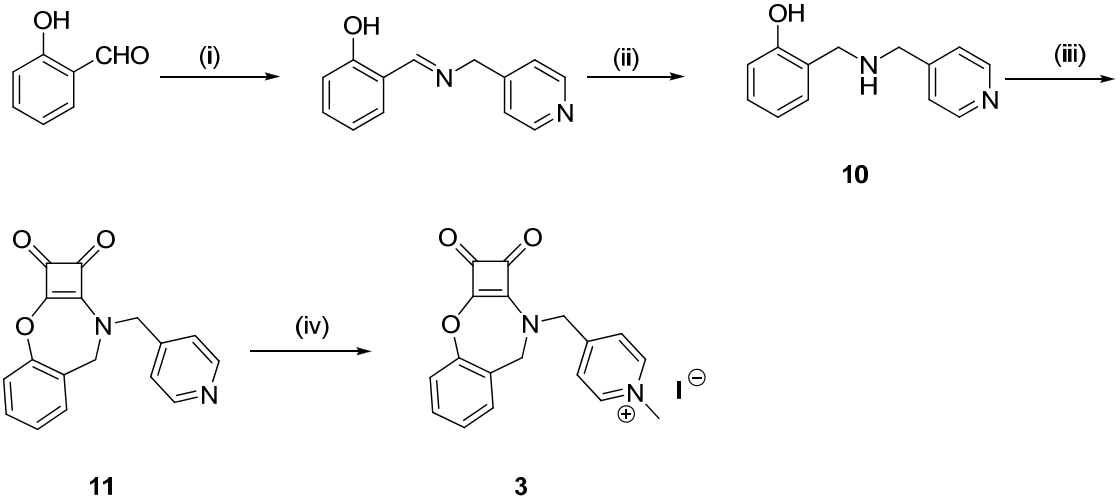
(2) General procedures for the synthesis of **8** and **9**

To a mixture of **2** (1mmol, 1eq) and TEA (2mmol, 2eq) in dry THF (20mL) at r.t. under Ar was added substituted amine (3mmol, 1.5eq). The mixture was stirred at r.t. overnight. The mixture was concentrated and the residue was partitioned between water and ethyl acetate. The organic layer was separated, washed with brine, dried and concentrated. The residue was purified by flash chromatography using PE:EA (1:1) as eluent to give a yellow solid.
3-(Allylamino)-4-(2-mercaptophenylamino)cyclobut-3-ene-1,2-dione (**8**): Yield 0.17g (65%). MS *m/z* (%): cacld. for $[M+H]^{+}$: 261.1, found: 261.1; ^{1}HNMR (300 MHz, DMSO-d_6) : δ 9.59 (brs, 1H), 8.70-8.66 (1H), 8.19 (s, 1H), 6.91-6.74 (m, 3H), 6.54 (dd, *J* = 7.5 Hz, 3Hz, 1H), 5.85-5.72 (m, 1H), 5.11-5.01 (m, 2H), 3.73-3.68 (m, 2H).

^{13}CNMR (75MHz, DMSO-d_6) : δ 177.52, 163.26, 147.03, 135.38, 135.27, 128.19, 127.72, 126.83, 120.97, 117.66, 116.26, 102.40, 41.64.

(S)-methyl-3-(1H-indol-3-yl)-2-(2-(2-mercaptophenylamino)-3,4-dioxocyclobut-1-enylamino)propanoate (**9**). Yield 0.23g (55%). MS *m/z* (%): cacld. for $[M+H]^+$: 422.1, found: 422.1; ^{1}HNMR (300 MHz, DMSO-d_6) : δ 10.86 (s, 1H), 9.73 (s, 1H), 8.73 (d, *J* = 7.8 Hz, 1H), 8.03 (s, 1H), 7.46 (d, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 7.8 Hz, 1H), 7.13 (s, 1H), 7.06-6.78 (m, 5H), 6.56 (d, *J* = 7.8 Hz, 1H), 4.53 (m, 1H), 3.62 (s, 3H), 3.21 (m, 2H); ^{13}CNMR (75MHz, DMSO-d_6) : δ 176.24, 171.77, 162.83, 146.36, 136.21, 134.43, 127.48, 127.09, 126.99, 126.20, 123.92, 121.14, 120.14, 118.59, 118.08, 117.02, 111.62, 109.38, 101.63, 53.04, 52.23, 26.43.

1.2 Synthesis of 4-((1,2-dioxo-1,2-dihydrobenzo[f]cyclobuta[b][1,4]oxazepin-9(8H)-yl)-methyl) -1-methyl -pyridinium iodide (**3**)



Reagents and conditions: (i) 4-aminomethyl pyridine, EtOH, r.t. 1hr; (ii) $NaBH_4$, EtOH, 0°C to r.t., 1hr; (iii) 3,4-dichlorocyclobutene-1,2-dione, TEA, benzene, 0°C to reflux, 16hr; (iv) CH_3I, CH_2Cl_2, r.t., 16hr

(1) Synthesis of 2-((pyridin-4-ylmethylamino)methyl)phenol (**10**)

A mixture of salicyaldehyde (10mmol) and amine (1eq) in EtOH (10mL) was stirred at r.t. for 2hr. The mixture was cooled to 0°C and $NaBH_4$ (1.2 eq) was added in portions. The mixture was then stirred at that temp. for 2hr. The reaction was quenched by water and concentrated. The residue was partitioned between water and DCM. The organic layers were combined, dried and concentrated. The residue was purified by flash chromatography using DCM:MeOH (20:1) as eluent to give a colorless sticky oil. Yield 1.9g (90%). ^{1}HNMR (300 MHz, $CDCl_3$) : δ 8.61-8.56 (m, 2H), 7.26-7.01 (m, 3H), 7.01-6.78 (m, 3H), 4.06 (s, 2H), 3.85 (s, 2H).

(2) Synthesis of 9-(pyridin-4-ylmethyl)-8,9-dihydrobenzo[f]cyclobuta[b][1,4]oxa -zepine-1,2-dione (**11**)

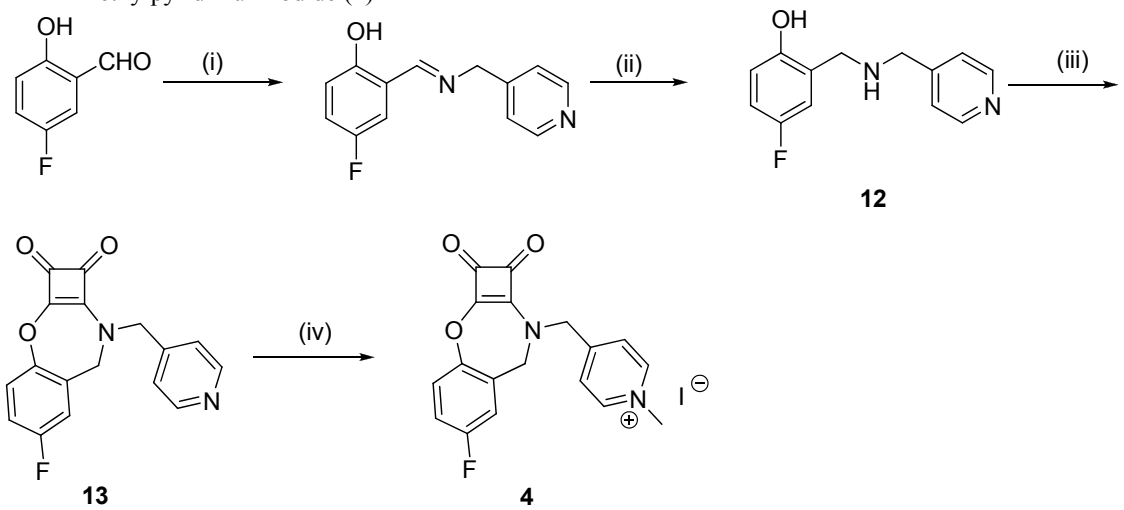
1,2-Dichlorocyclobutene-3,4-dione (0.6g) was dissolved in dry benzene (50mL) and cooled down to 0 °C under argon atmosphere. 1 eq of 2-((pyridin-4-ylmethylamino)-methyl)phenol in anhydrous benzene (25mL) was added dropwise, accompanied by addition of excess of triethyl amine (2.2eq) in anhydrous benzene (25mL). Reaction mixture was stirred at 0°C for 0.5hr, at r.t. for 0.5hr and at reflux overnight. The reaction mixture was then filtered and concentrated. Flash chromatography using Hexanes (100mL), DCM (100mL), DCM:MeOH (100:1, 200mL), DCM:MeOH (80:1, 200mL), DCM:MeOH (60:1) as eluent afforded a brown sticky oil, which was dried under high vacuum to give a foamy solid. Yield: 0.19g (16.3%). MS *m/z* (%): cacld. for $[M+H]^+$: 293.1, found: 293.1; ^{1}HNMR (300 MHz, DMSO-d_6) : δ 8.57 (dd, *J* = 1.5, 4.5Hz, 2H), 7.48-7.31 (m, 5H), 7.22-7.16 (m, 1H), 4.89 (s, 2H), 4.67 (s, 2H).

(3) Synthesis of 4-((1,2-dioxo-1,2-dihydrobenzo[f]cyclobuta[b][1,4]oxazepin-9(8H)-yl)methyl)-1-methyl -pyridinium iodide (**3**)

A mixture of 9-(pyridin-4-ylmethyl)-8,9-dihydrobenzo[f]cyclobuta[b][1,4]oxazepine -1,2-dione (20mg) and iodomethane (excess) in anhydrous DCM (0.4mL) was stirred at r.t. for overnight. The mixture was filtered and the solid was dried under high vacuum to give a pale white solid with rather good water solubility. Yield : 18.9mg

(90%). HRMS *m/z*: cacld. for $[M+H-I]^+$: 307.1082, found: 307.1083; ^{1}HNMR (300 MHz, DMSO-d_6) : δ 8.96 (d, *J* = 6.3Hz, 2H), 8.11 (d, *J* = 6.0Hz, 2H), 7.51-7.19 (m, 4H), 5.16 (s, 2H), 4.74 (s, 2H), 4.32 (s, 3H). ^{13}CNMR (75MHz, DMSO-d_6) : δ 188.44, 181.18, 175.26, 173.81, 155.30, 154.41, 145.69, 132.27, 130.86, 125.91, 125.37, 123.61, 120.50, 53.86, 52.73, 47.65.

1.3 Synthesis of 4-((6-fluoro-1,2-dioxo-1,2-dihydrobenzo[f]cyclobuta[b][1,4]oxazepin -9(8H)-yl)methyl) -1-methylpyridinium iodide (**4**)



Reagents and conditions: (i) 4-aminomethyl pyridine, EtOH, r.t. 1hr; (ii) $NaBH_4$, EtOH, 0^{o}C to r.t., 1hr; (iii) 3,4-dichlorocyclobutene-1,2-dione, TEA, benzene, 0^{o}C to reflux, 16hr; (iv) CH_3I, CH_2Cl_2, r.t., 16hr

(1) Synthesis of 4-fluoro-2-((pyridin-4-ylmethylamino)methyl)phenol (**12**)

A mixture of aldehyde (10mmol) and amine (1eq) in EtOH (10mL) was stirred at r.t. for 2hr. The mixture was then cooled to 0^{o}C and $NaBH_4$ (1.2 eq) was added in portions. The mixture was then stirred at that temp. for 2hr. The reaction solution was quenched by water at 0^{o}C and concentrated. The residue was extracted by DCM. The organic layers were combined, dried and concentrated. The residue was purified by flash chromatography using DCM:MeOH (20:1) as eluent to give a colorless sticky oil. Yield 1.44g (65%). ^{1}HNMR (300 MHz, DMSO-d_6) : δ 8.50 (d, *J* = 6 Hz, 2H), 7.35 (d, *J* = 6Hz, 2H), 7.03 (dd, *J* = 9, 3 Hz, 1H), 6.89 (m, 1H), 6.74-6.69 (m, 1H), 3.73 (s, 2H), 3.70 (s, 2H).

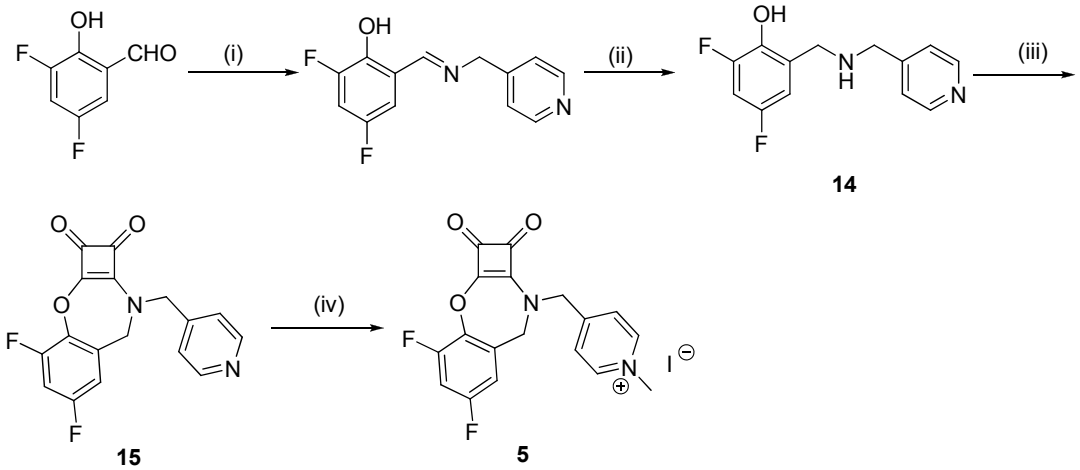
(3) Synthesis of 6-fluoro-9-(pyridin-4-ylmethyl)-8,9-dihydrobenzo[f]cyclobuta[b][1,4]–oxazepine-1,2 -dione (**13**)

1,2-Dichlorocyclobutene-3,4-dione (0.6g) was dissolved in dry benzene (40mL) and cooled down to 0 ^{0}C under argon atmosphere. 1 eq of phenol in anhydrous benzene (30 mL) was added dropwise, accompanied by addition of excess of triethyl amine (2.2eq) in anhydrous benzene (30 mL). Reaction mixture was stirred at 0^{o}C for 0.5hr, at r.t. for 0.5hr and at reflux overnight. The reaction mixture was then filtered and concentrated. Flash chromatography using PE (100mL), DCM (100mL), DCM:MeOH (100:1, 200mL), DCM:MeOH (80:1, 200mL), DCM:MeOH (60:1) as eluent afforded a brown sticky oil, which was dried under high vacuum to give a foamy solid. Yield 0.31g (25%). HRMS *m/z*: cacld. for $[M+H]^+$: 311.0832, found: 311.0822; ^{1}HNMR (300 MHz, DMSO-d_6) : 8.59-8.58 (m, 2H), 7.46-7.26 (m, 5H), 4.98 (s, 2H), 4.66 (s, 2H).

(3) Synthesis of 4-((6-fluoro-1,2-dioxo-1,2-dihydrobenzo[f]cyclobuta[b][1,4]oxazepin -9(8H)-yl)methyl) -1-methylpyridinium iodide (**4**)

A mixture of 6-fluoro-9-(pyridin-4-ylmethyl)-8,9-dihydrobenzo[f]cyclobuta[b][1,4]–oxazepine-1,2 -dione (20mg) and iodomethane (excess) in anhydrous DCM (0.4mL) was stirred at r.t. overnight. The mixture was filtered and the solid was washed with large amount of DCM, dried under high vacuum to give a pale white solid with rather good water solubility. Yield: 18.8 mg (90%). HRMS *m/z*: cacld. for $[M+H-I]^+$: 325.0988, found: 325.0996; ^{1}HNMR (300 MHz, DMSO-d_6) : δ 8.97 (m, 2H), 8.13-8.11 (m, 2H), 7.50-7.23 (m, 3H), 5.15 (s, 2H), 4.72 (s, 2H), 4.33 (3H). ^{13}CNMR (75MHz, DMSO-d_6) : δ 188.33, 181.08, 175.32, 173.72, 159.47, 156.26, 155.05, 150.82, 150.78, 145.68, 126.02, 125.62, 125.51, 122.59,122.48, 118.58, 118.26, 117.47, 117.16, 53.02, 52.71, 47.72.

1.4 Synthesis of 4-((4,6-difluoro-1,2-dioxo-1,2-dihydrobenzo[f]cyclobuta[b][1,4]-oxazepin-9(8H)-yl) -methyl)-1-methylpyridinium iodide **5**



Reagents and conditions: (i) 4-aminomethyl pyridine, EtOH, r.t. 1hr; (ii) $NaBH_4$, EtOH, 0°C to r.t., 1hr; (iii) 3,4-dichlorocyclobutene-1,2-dione, TEA, benzene, 0°C to reflux, 16hr; (iv) CH_3I, CH_2Cl_2, r.t., 16hr

(1) Synthesis of 4,6-difluoro-2-((pyridin-4-ylmethylamino)methyl)phenol (**14**)

A mixture of aldehyde (10mmol) and amine (1eq) in EtOH (10mL) was stirred at r.t. for 2hr. The mixture was then cooled to 0°C and $NaBH_4$ (1.2 eq) was added in portions. The mixture was then stirred at that temp. for 2hr. The reaction solution was quenched by water at 0°C and concentrated. The residue was extracted by DCM. The organic layers were combined, dried and concentrated. The residue was purified by flash chromatography using DCM:MeOH (20:1) as eluent to give a colorless sticky oil. Yield 1.63g (65%). ^{1}HNMR (300 MHz, DMSO-d_6) : δ 8.61 (d, *J* = 4 Hz, 2H), 7.27-6.84 (m, 2H), 6.84-6.76 (m, 1H), 6.56-6.53 (m, 1H), 4.04 (s, 3H), 3.85 (s, 3H).

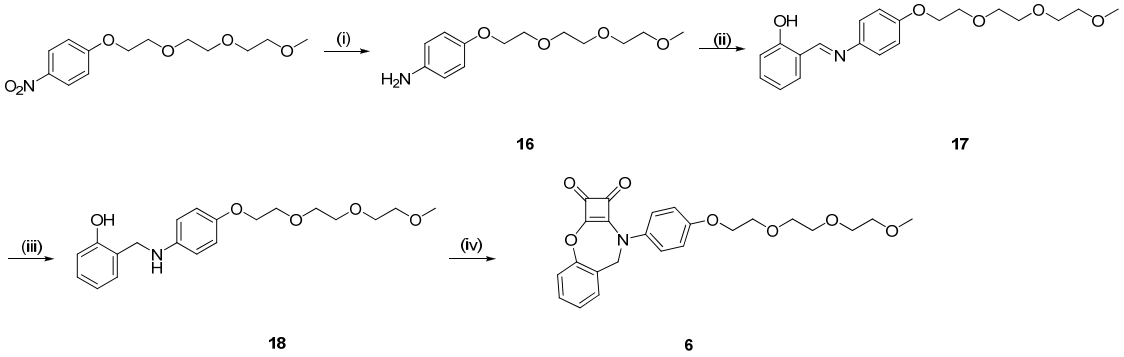
(2) Synthesis of 4,6-difluoro-9-(pyridin-4-ylmethyl)-8,9-dihydrobenzo[f]cyclobuta-[b][1,4]oxazepine- -1,2-dione (**15**)

1,2-Dichlorocyclobutene-3,4-dione (0.6g) was dissolved in dry benzene (40mL) and cooled down to 0 ^{0}C under argon atmosphere. 1 eq of phenol in anhydrous benzene (30 mL) was added dropwise, accompanied by addition of excess of triethyl amine (2.2eq) in anhydrous benzene (30 mL). Reaction mixture was stirred at 0°C for 0.5hr, at r.t. for 0.5hr and at reflux overnight. The reaction mixture was then filtered and concentrated. Flash chromatography using PE (100mL), DCM (100mL), DCM:MeOH (100:1, 200mL), DCM:MeOH (80:1, 200mL), DCM:MeOH (60:1) as eluent afforded a brown sticky oil, which was dried under high vacuum to give a foamy solid. Yield 0.33g (25%). HRMS *m/z*: cacld. for $[M+H]^+$: 329.0738, found: 329.0737; ^{1}HNMR (300 MHz, DMSO-d_6) : 8.70-8.69 (m, 2H), 7.27-7.23 (m, 2H), 7.08-7.01 (m, 1H), 6.64-6.60 (m, 1H), 4.94 (s, 2H), 4.74 (s, 2H).

(3) Synthesis of 4-((4,6-difluoro-1,2-dioxo-1,2-dihydrobenzo[f]cyclobuta[b][1,4] -oxazepin-9(8H)-yl) -methyl)-1-methylpyridinium iodide (**5**)

A mixture of 6-fluoro-9-(pyridin-4-ylmethyl)-8,9-dihydrobenzo[f]cyclobuta[b][1,4]–oxazepine-1,2 -dione (20mg) and iodomethane (excess) in anhydrous DCM (0.4mL) was stirred at r.t. for overnight. The mixture was filtered and the solid was washed with large amount of DCM, dried under high vacuum to give a pale white solid with rather good water solubility. Yield : 19mg (90%). HRMS *m/z*: cacld. for $[M+H-I]^+$: 343.0894, found: 343.0903; ^{1}HNMR (300 MHz, DMSO-d_6) : δ 8.98 (d, *J* = 4Hz, 2H), 8.12 (d, *J* = 4Hz, 2H), 7.67-7.60 (m, 1H), 7.15-7.11 (m, 1H), 5.16 (s, 2H), 4.78 (s, 2H), 4.33 (3H). ^{13}CNMR (75MHz, DMSO-d_6) : δ 188.37, 180.68, 175.31, 172.86, 158.61, 158.45, 155.37, 155.20, 155.03, 153.80, 150.49,150.31, 145.67, 139.74, 139.69, 139.58, 139.52, 127.13, 127.00, 125.92, 113.82, 113.77, 113.49, 113.45, 106.54, 106.23, 106.18, 105.87, 53.00, 52.83, 47.68.

1.5 Synthesis of 9-(4-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)phenyl)-8,9-dihydrobenzo[f]cyclobuta[b][1, -4]oxazepine-1,2-dione (**6**)



Reagents and conditions: (i) Pd/C, H2, MeOH; (ii) salicy aldehyde, EtOH, r.t. 1hr; (iii) $NaBH_4$, EtOH, 0°C to r.t., 1hr; (iv) 3,4-dichlorocyclobutene-1,2-dione, TEA, benzene, 0°C to reflux, 16hr.

(1) Synthesis of 4-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)aniline (**16**)

A mixture of **1** (10 mmol) and 10% Pd/C (10%wt) in MeOH (50 mL) was hydrogenated at room temperature under a hydrogen balloon, with vigorous stirring, overnight. The catalyst was filtered through Celite, and the filtrate was concentrated to give a brown sticky oil. The oil was used directly for the next step without further purification.

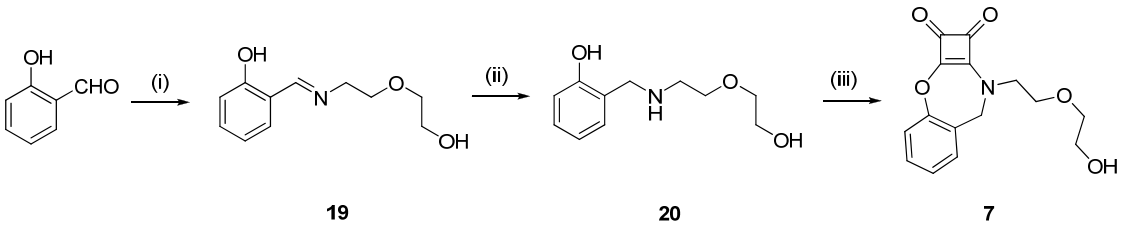
(2) Synthesis of 2-((4-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)phenylamino)methyl)phenol (**18**)

A mixture of aldehyde (10mmol) and amine (1eq) in EtOH was stirred at r.t. for 2hr. The mixture was cooled to 0°C and $NaBH_4$ (1.2eq) was added in portions. The mixture was then stirred at that temp. for 2hr. The reaction was solution was quenched by water and concentrated. The residue was extracted by DCM. The organic layers were combined, dried and concentrated to give a brown sticky oil. The oil was used directly for the next step without further purification.

(3) Synthesis of 9-(4-(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)phenyl)-8,9-dihydrobenzo[f]cyclobuta[b][1,-4]oxazepine-1,2-dione (**6**)

1,2-Dichlorocyclobutene-3,4-dione (0.6g) was dissolved in dry benzene (50mL) and cooled down to 0 ^{0}C under argon atmosphere. 1 eq of phenol in anhydrous benzene (5mL) was added dropwise, accompanied by addition of excess of triethyl amine (2.2eq) in anhydrous benzene (5mL). Reaction mixture was stirred at 0°C for 0.5hr, at r.t. for 0.5hr and at reflux overnight. Flash chromatography using Hexanes:EtOAc (1:1) as eluent afforded a yellow sticky oil, which was dried under high vacuum to give semi solid. Total yield (three steps): 52.7%. HRMS *m/z*: cacld.: 440.1709 $[M+H]^+$, found: 440.1700; ^{1}HNMR (300 MHz, $CDCl_3$) : δ 7.50-7.15 (m, 6H), 6.98 (d, J = 9.0 Hz, 2H), 4.99 (s, 2H), 4.16-4.14 (m, 2H), 3.89-3.86 (m, 2H), 3.76-3.57 (m, 8H), 3.39 (s, 3H).

1.6 Synthesis of 9-(2-(2-hydroxyethoxy)ethyl)-8,9-dihydrobenzo[f]cyclobuta[b][1,4]-oxazepine-1,2-dione (**7**)



(1)Synthesis of 2-((2-(2-hydroxyethoxy)ethylamino)methyl)phenol (**20**)

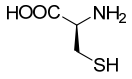
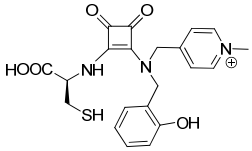
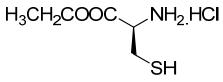
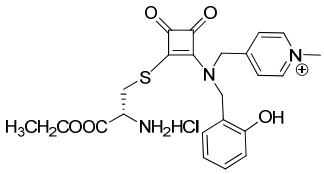
A mixture of aldehyde (10mmol) and amine (1eq) in EtOH was stirred at r.t. for 2hr. The mixture was cooled to 0°C and $NaBH_4$ (1.2eq) was added in portions. The mixture was then stirred at that temp. for 2hr. The reaction was solution was quenched by water and concentrated. The residue was extracted by DCM. The organic layers were combined, dried and concentrated to give a yellow sticky oil. The oil was used directly for the next step without further purification.

(2)Synthesis of 9-(2-(2-hydroxyethoxy)ethyl)-8,9-dihydrobenzo[f]cyclobuta[b][1,4]-oxazepine-1,2-dione (**7**)

1,2-Dichlorocyclobutene-3,4-dione (0.6g) was dissolved in dry benzene (50mL) and cooled down to 0 ^{0}C under argon atmosphere. 1 eq of phenol in anhydrous benzene (5mL) was added dropwise, accompanied by addition of excess of triethyl amine (2.2eq) in anhydrous benzene (5mL). Reaction mixture was stirred at 0^{o}C for 0.5hr, at r.t. for 0.5hr and at reflux overnight. Flash chromatography using Hexanes:EtOAc (1:1) as eluent afforded a yellow sticky oil. Total yield (two steps): 23.1%. HRMS m/z (%): cacld.: 290.1023 $[M+H]^+$, found: 290.1026; ^{1}HNMR (300 MHz, $CDCl_3$) : δ 7.42-6.89 (m, 4H), 4.68 (s, 2H), 3.96-3.56 (m, 8H).

2. Ligation of cyclic squarate derivative **3** with L-cysteine and L-cysteine ethyl ester

Table S1. Ligation of cyclic squarate derivative **3** with L-cysteine and L-cysteine ethyl ester (Scheme **1**).[a]

Entry	Reactant	Product	Time (Conv >95%)
1			5 min
2			5 min

[a]Reaction conditions: H_2O, r.t.

3. UV spectra of **3**, NHS-Biotin and SQ-Biotin

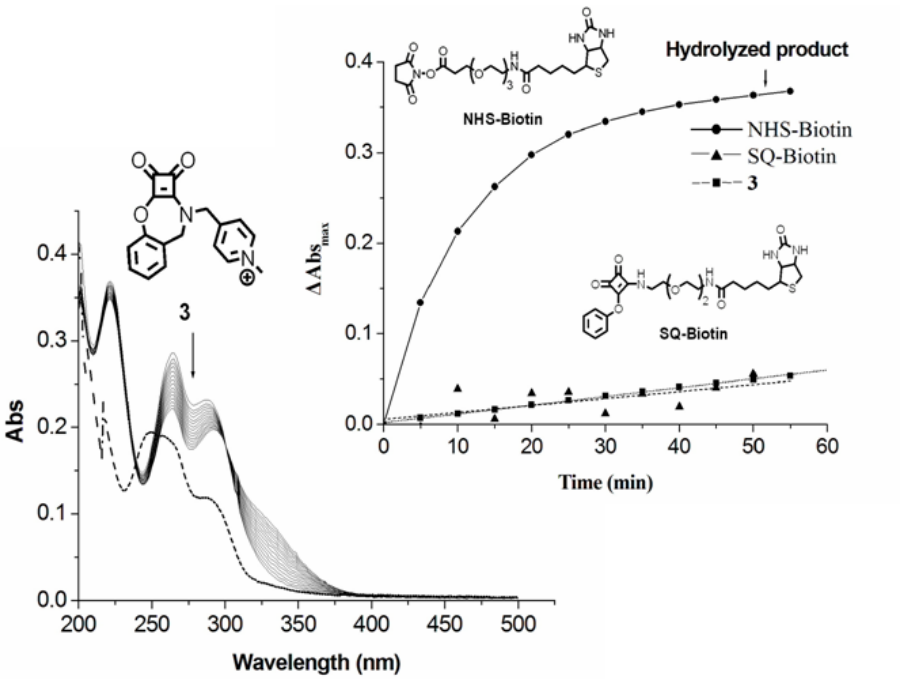


Fig1 UV spectra of 10 μM of **3** in PBS buffer (10 mM, pH 8.02) at increment of 5 min from 0 to 70 min (solid line), and in CH_3CN (dash line). The insert shows the plot of ($Abs_{max(0\ min)}$-$Abs_{max(t)}$) at 264 nm of **3**, NHS-Biotin and SQ-Biotin versus time in PBS buffer (10 mM, pH 8.02).

4. The Maldi-Tof spectra for the ligation of the proteins using squarate pyridium salts.

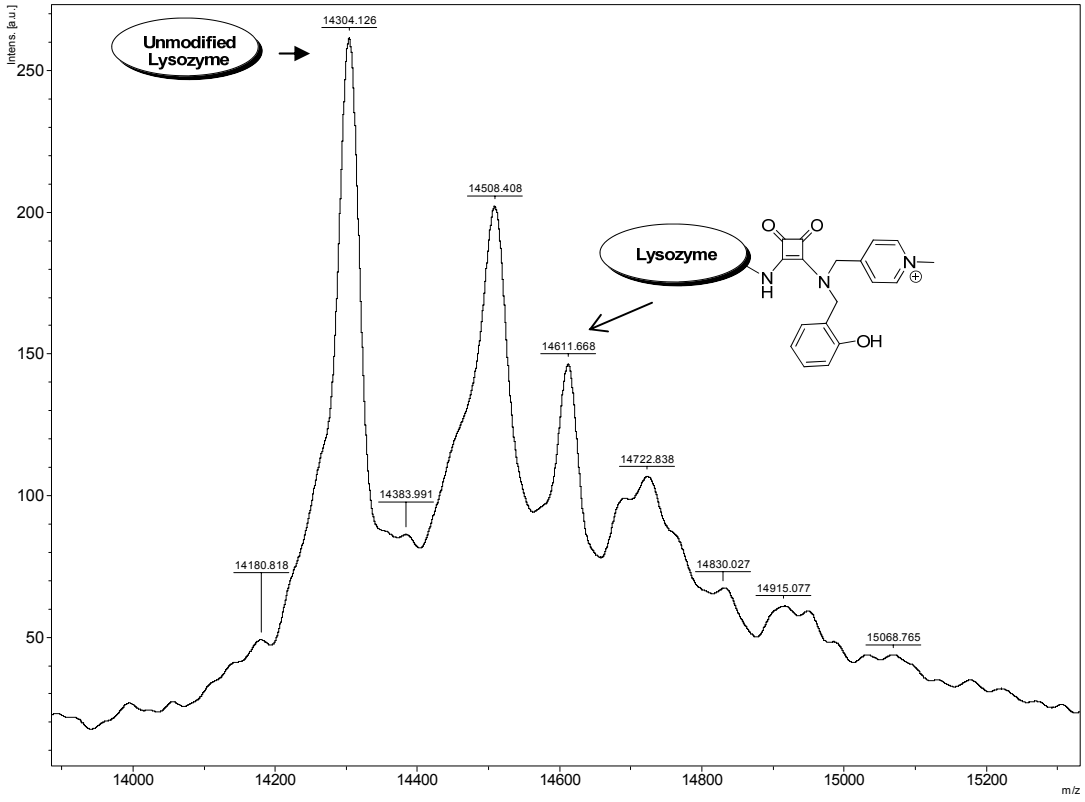


Figure 2. The spectrum of Maldi-Tof of modified Lysozyme by **3** at pH 7.75 in 10mM PBS buffer.

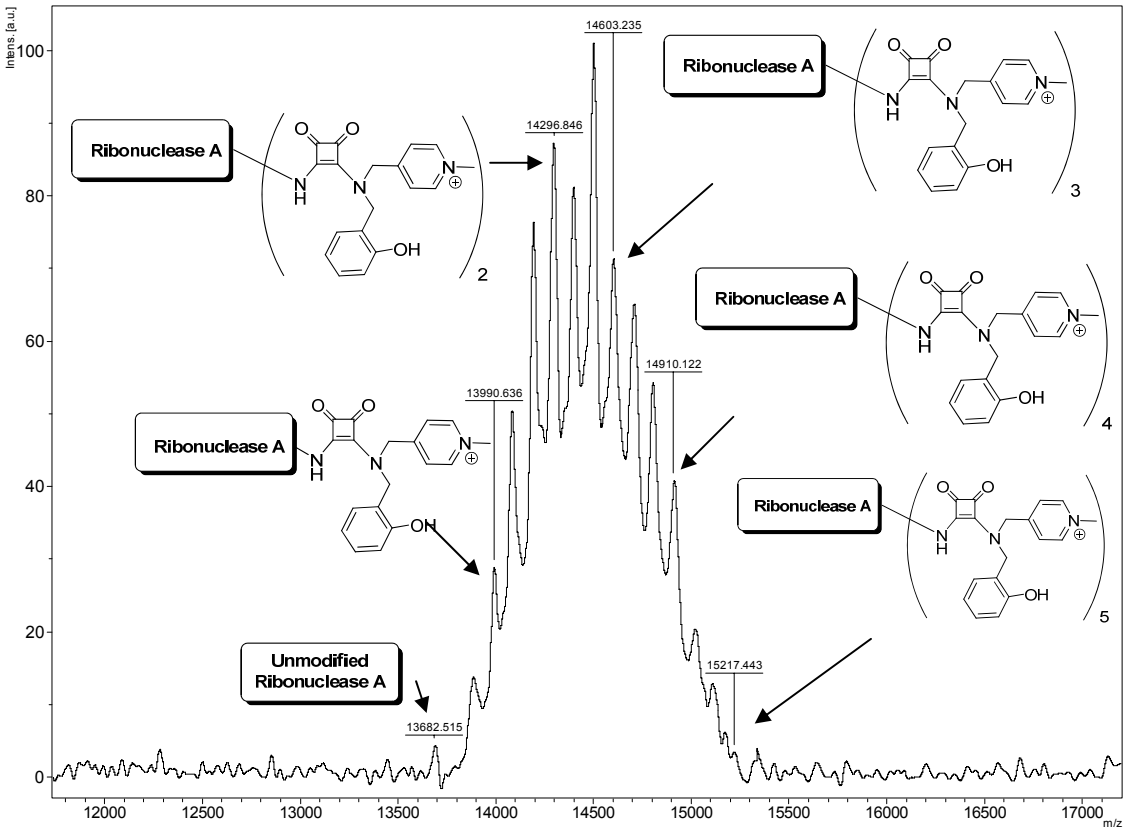


Figure 3. The spectrum of Maldi-Tof of modified Ribonuclease A by **3** at pH 7.75 in 10mM PBS buffer.

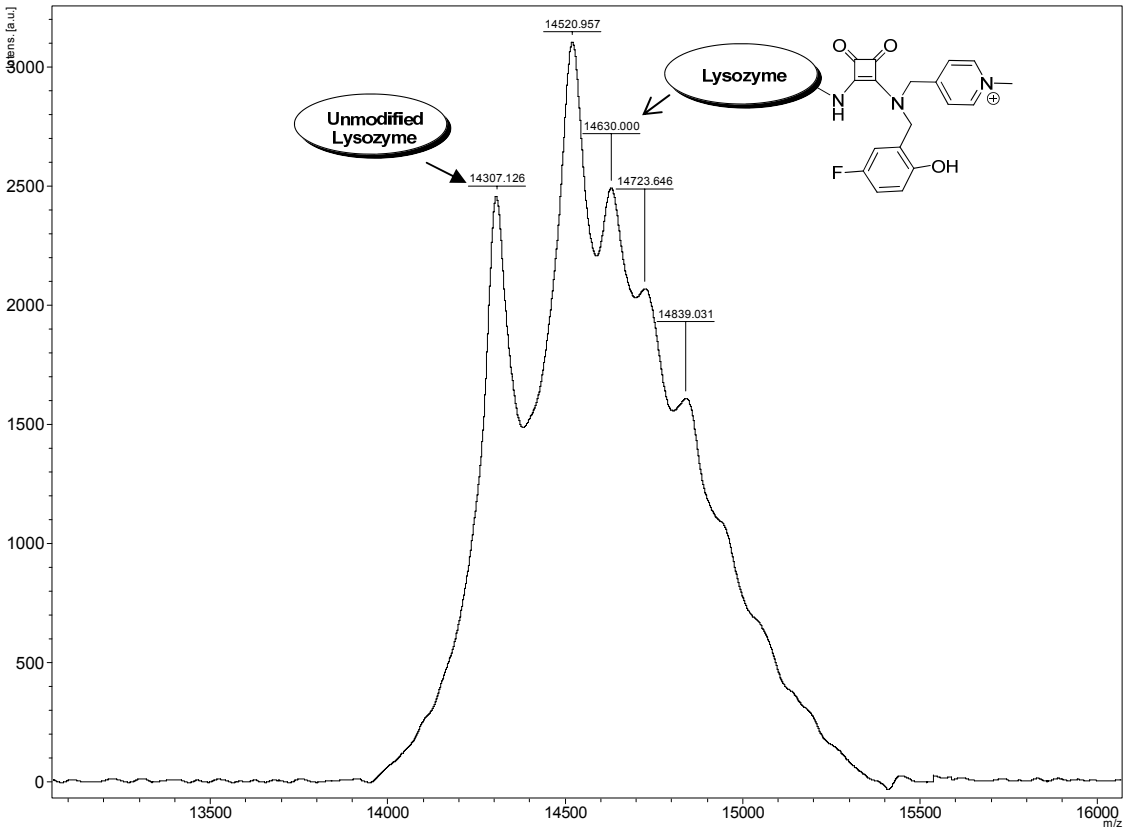


Figure 4. The spectrum of Maldi-Tof of modified Lysozyme by **4** at pH 7.75 in 10mM PBS buffer.

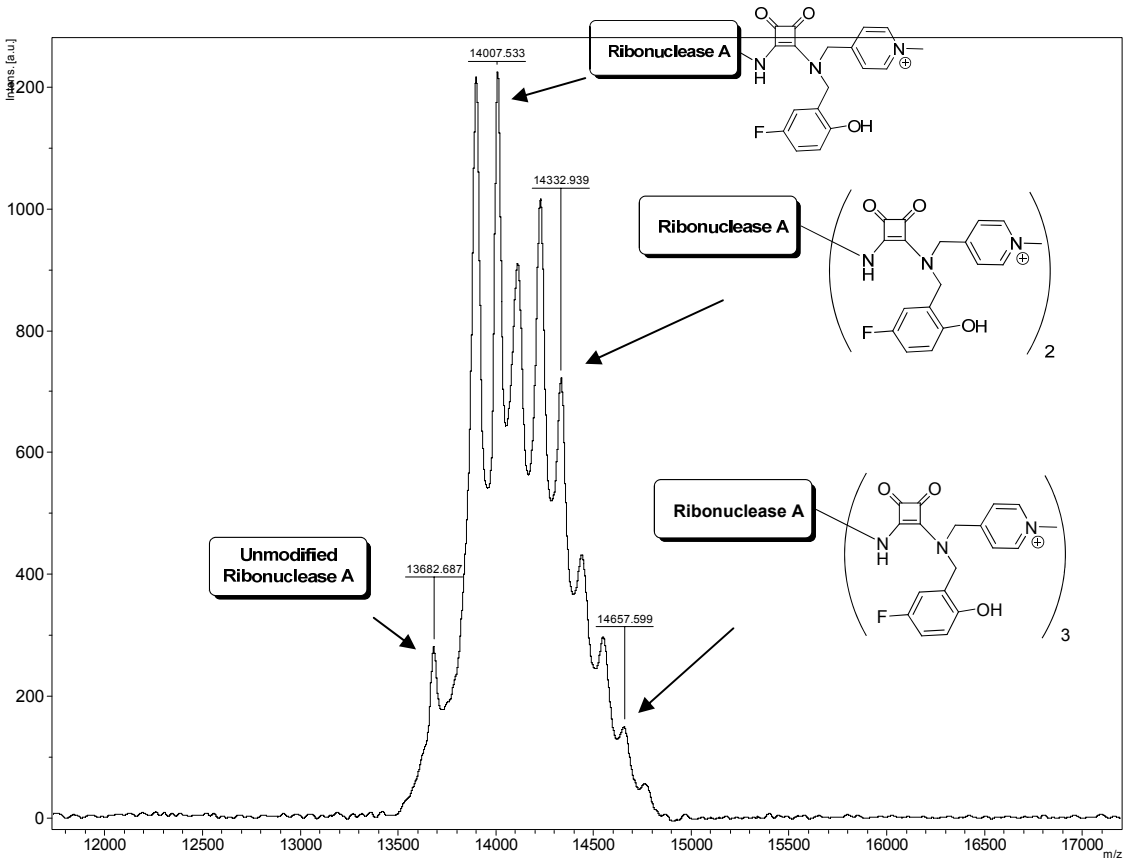


Figure 5. The spectrum of Maldi-Tof of modified Ribonuclese A by **4** at pH 7.75 in 10mM PBS buffer.

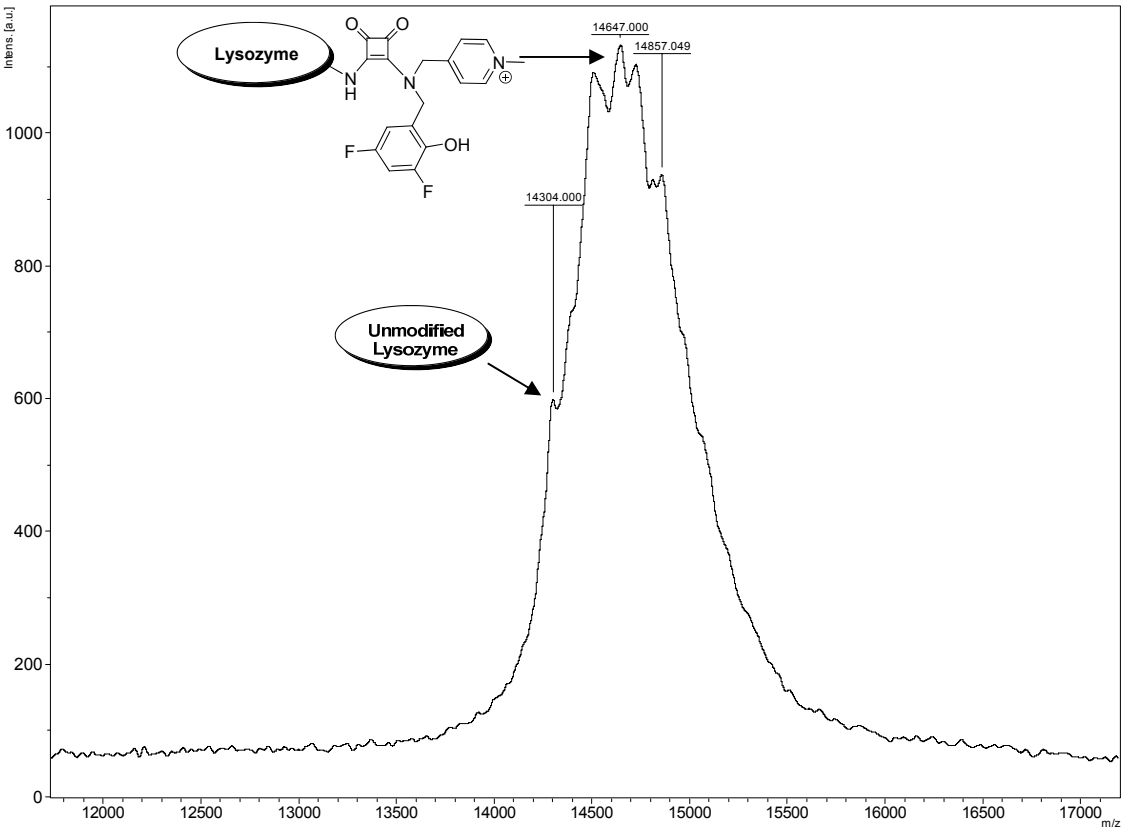


Figure 6. The spectrum of Maldi-Tof of modified Lysozyme by **5** at pH 7.75 in 10mM PBS buffer.

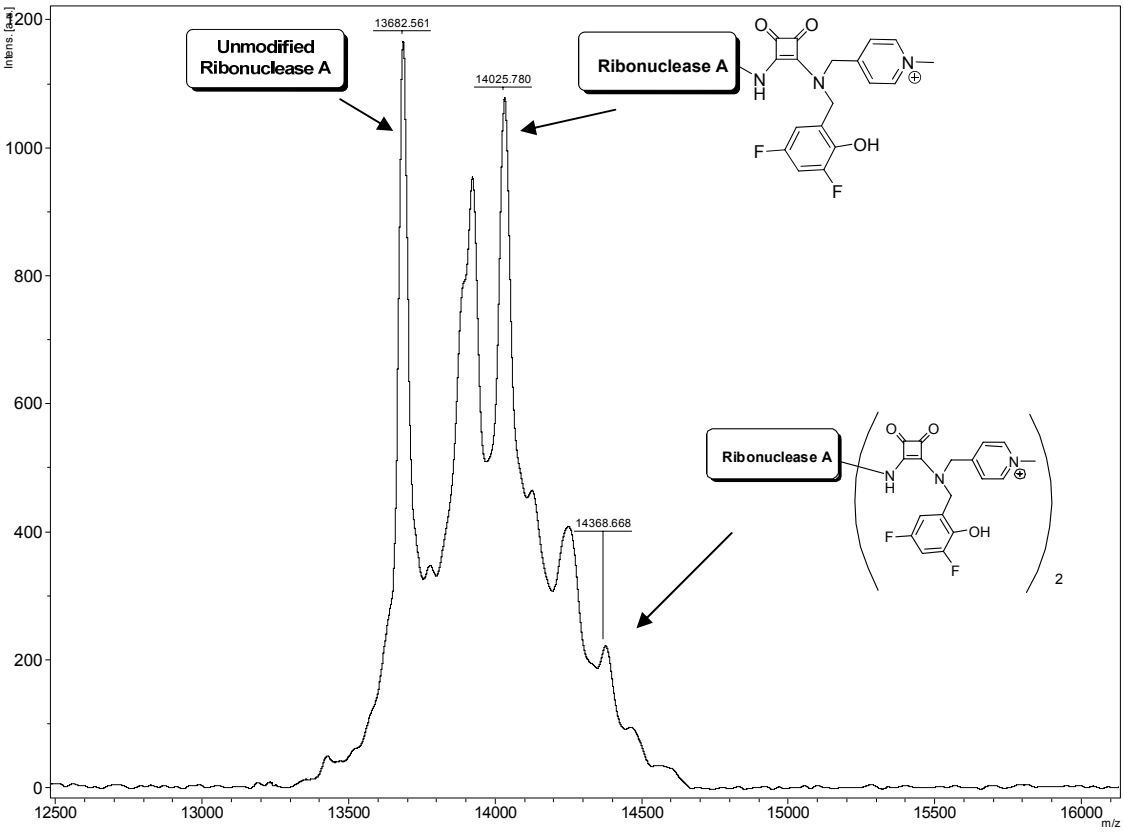


Figure 7. The spectrum of Maldi-Tof of modified Ribonuclese A by **5** at pH 7.75 in 10mM PBS buffer.

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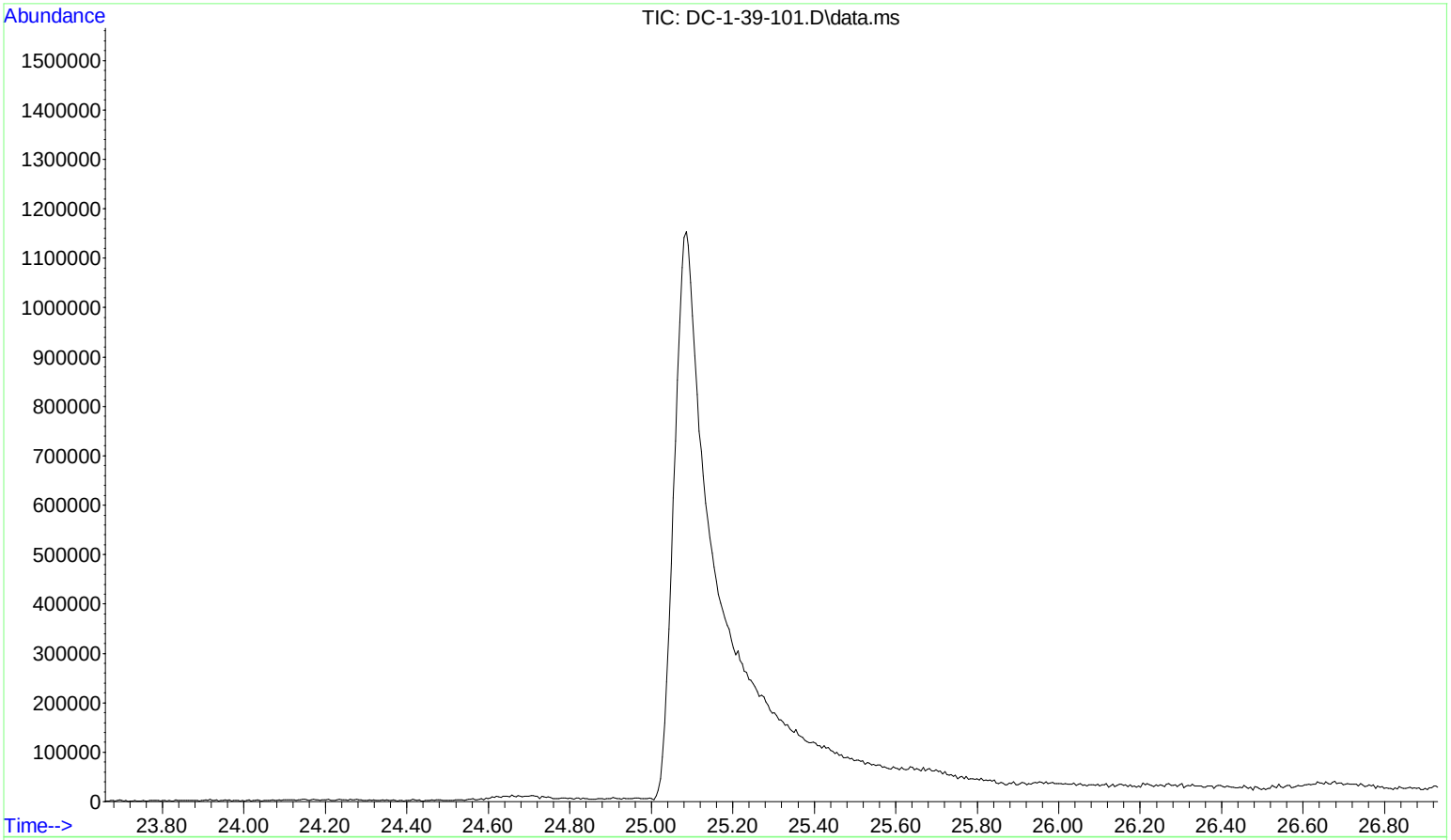
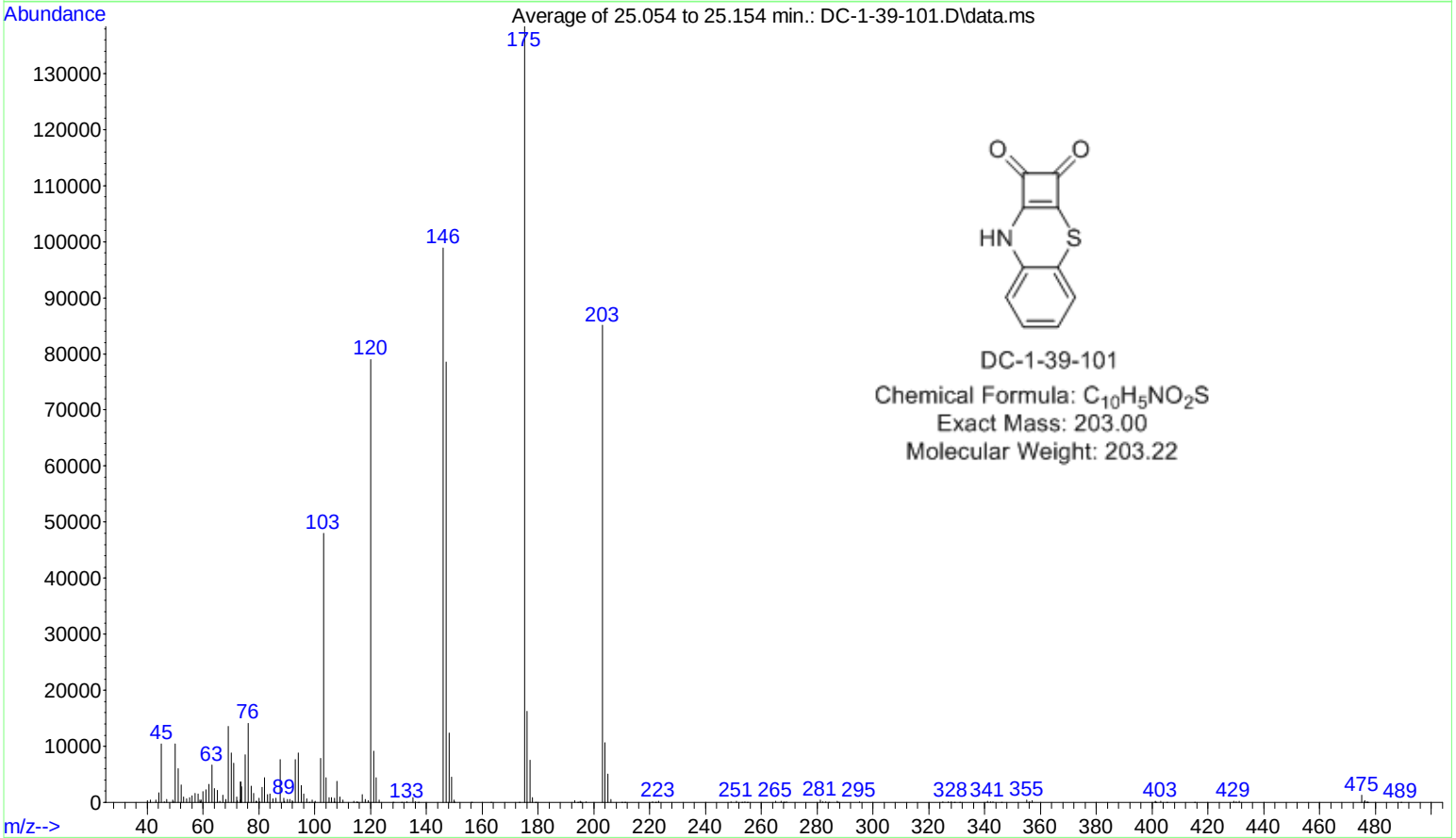
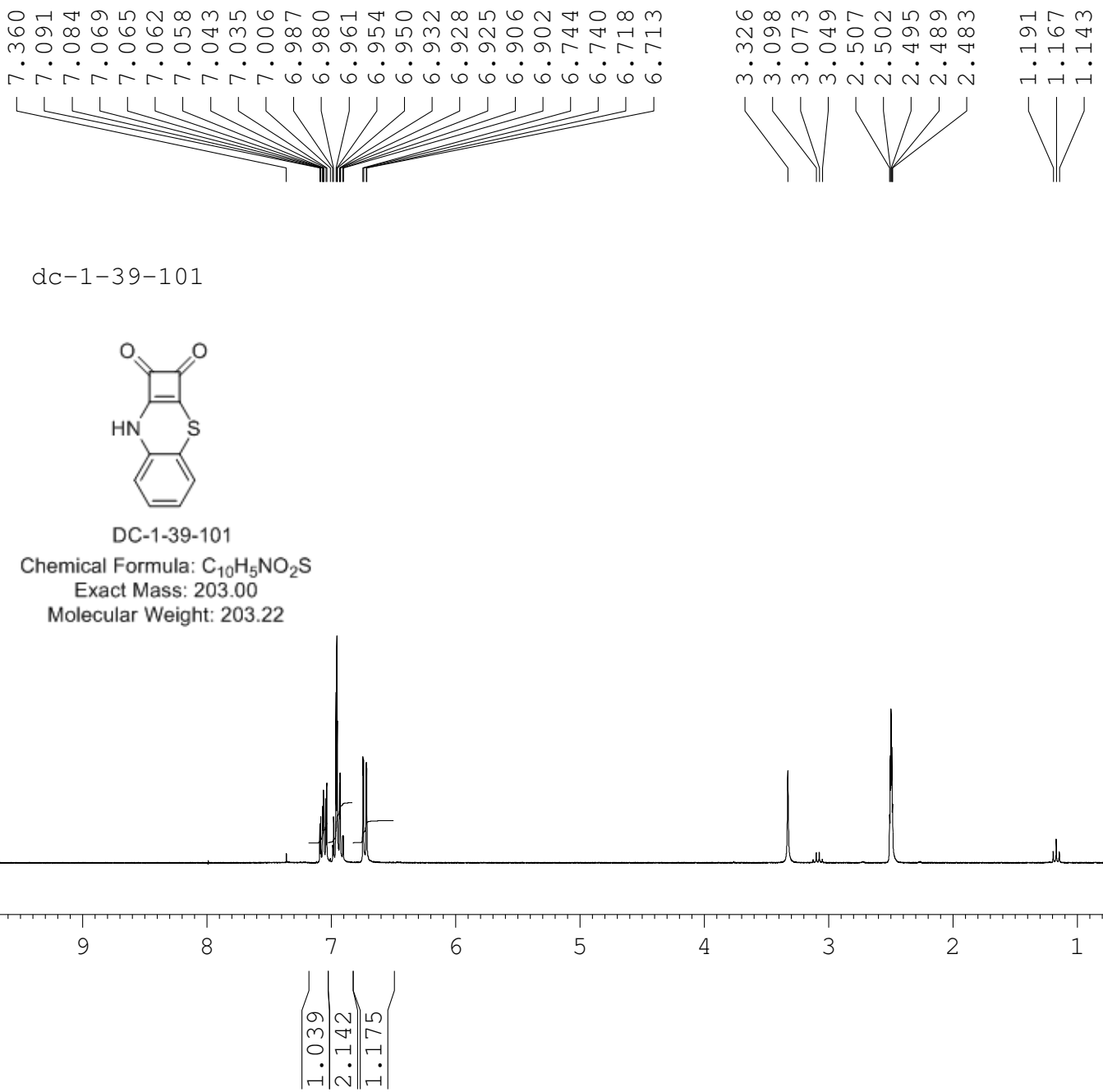
Operator   : amh
Acquired   : 11 Dec 2009  11:30     using AcqMethod 00_GEN 30_1 SPLIT ALS_INJ_EI 40C 40_500DA.M
Instrument :    Instrument #1
Sample Name: dc-1-39-101
Misc Info  :
Vial Number: 11
```





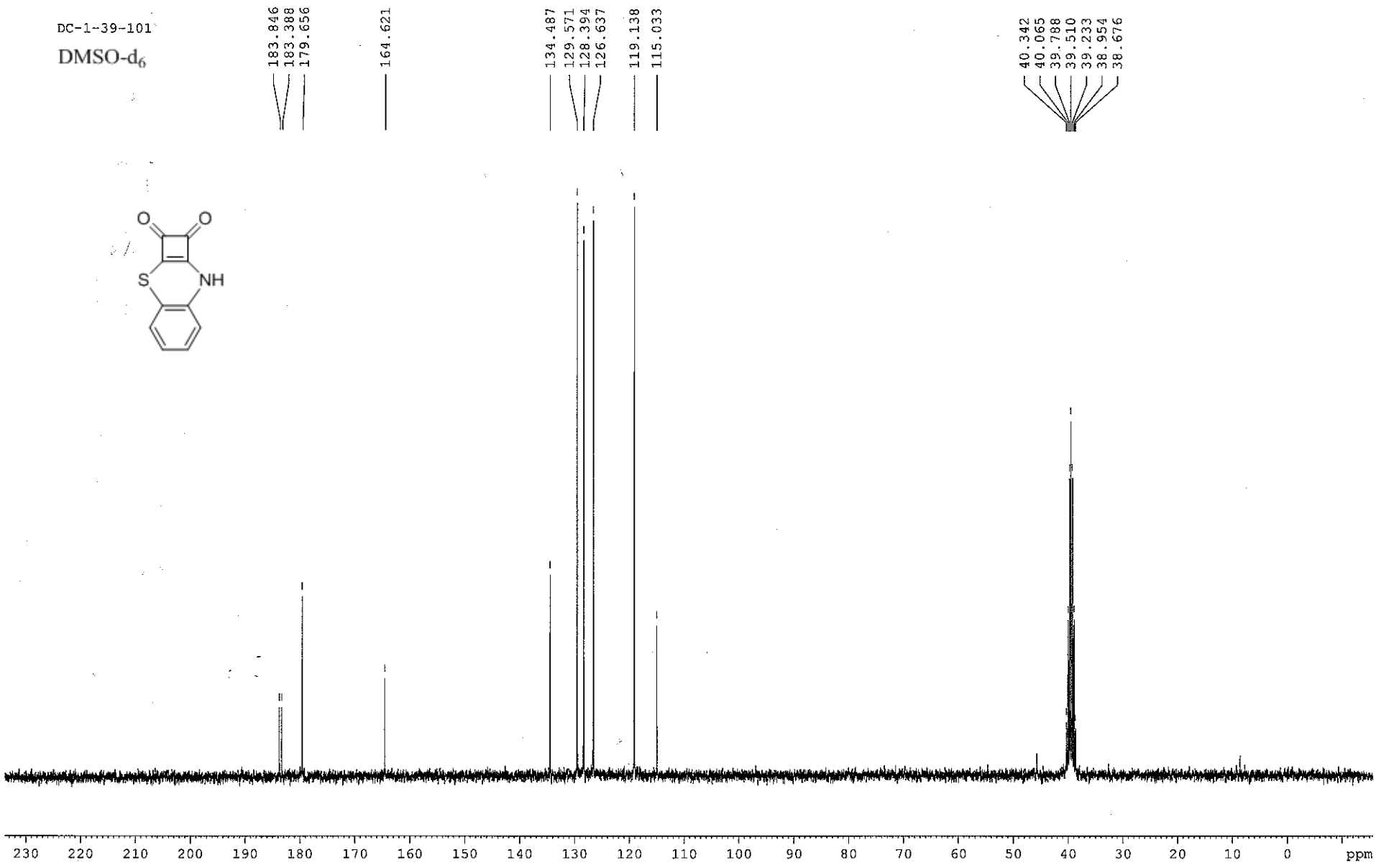


Current Data Parameters
NAME dc−1−39−101
EXPNO 1
PROCNO 1

F2 − Acquisition Parameters
Date_ 20100622
Time 17.43
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 812.7
DW 104.400 usec
DE 54.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 =====
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1324000 MHz

F2 − Processing parameters
SI 32768
SF 300.1300024 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00


DC-1-39-101
DMSO-d6
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183.388
179.656
164.621
134.487
129.571
128.394
126.637
119.138
115.033
40.342
40.065
39.788
39.510
39.233
38.954
38.676
O
O
S
NH
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

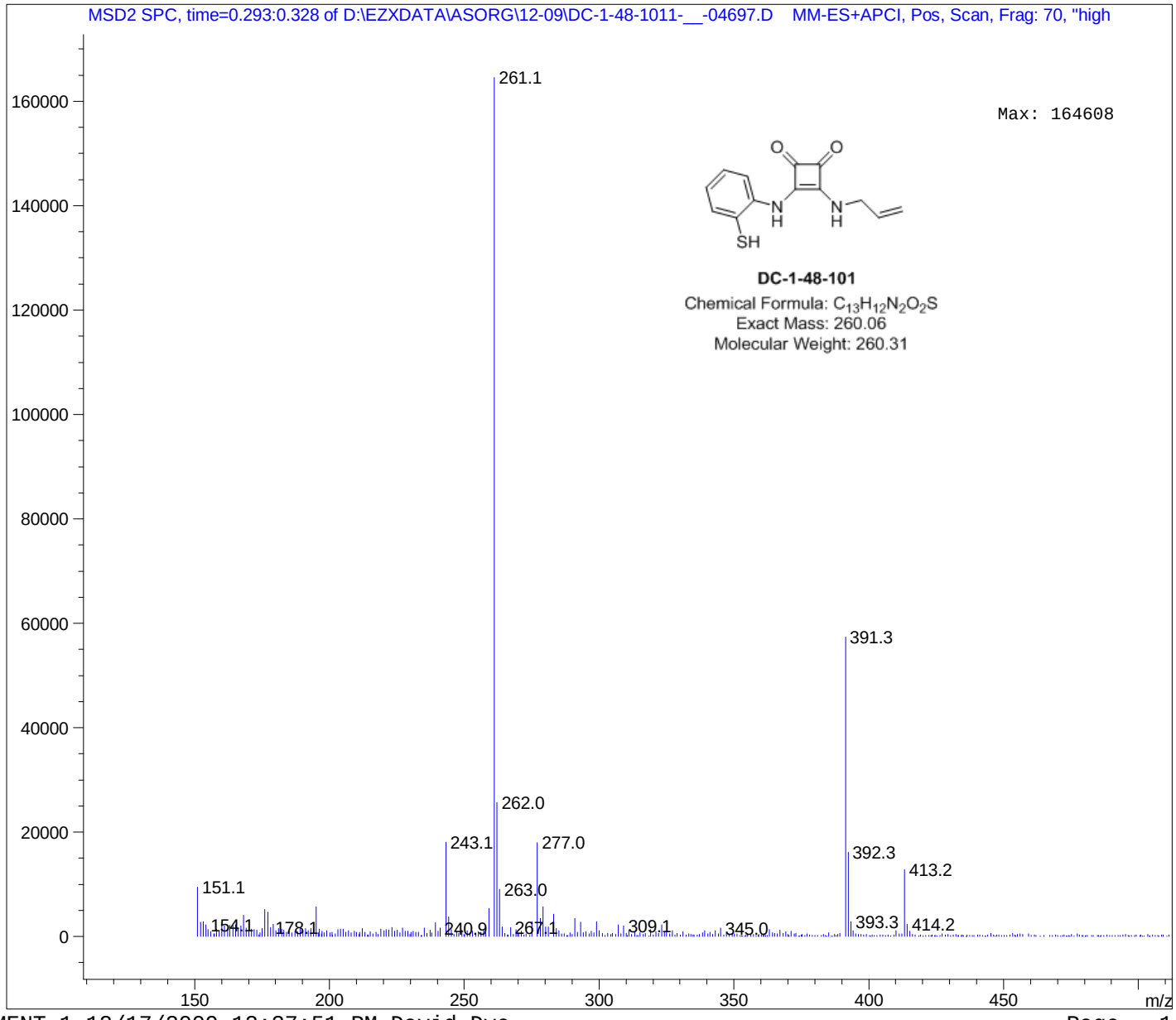
Print of window 80: MS Spectrum
Data File : D:\EZXDATA\ASORG\12-09\DC-1-48-1011-__-04697.D
Sample Name : dc-1-48-101

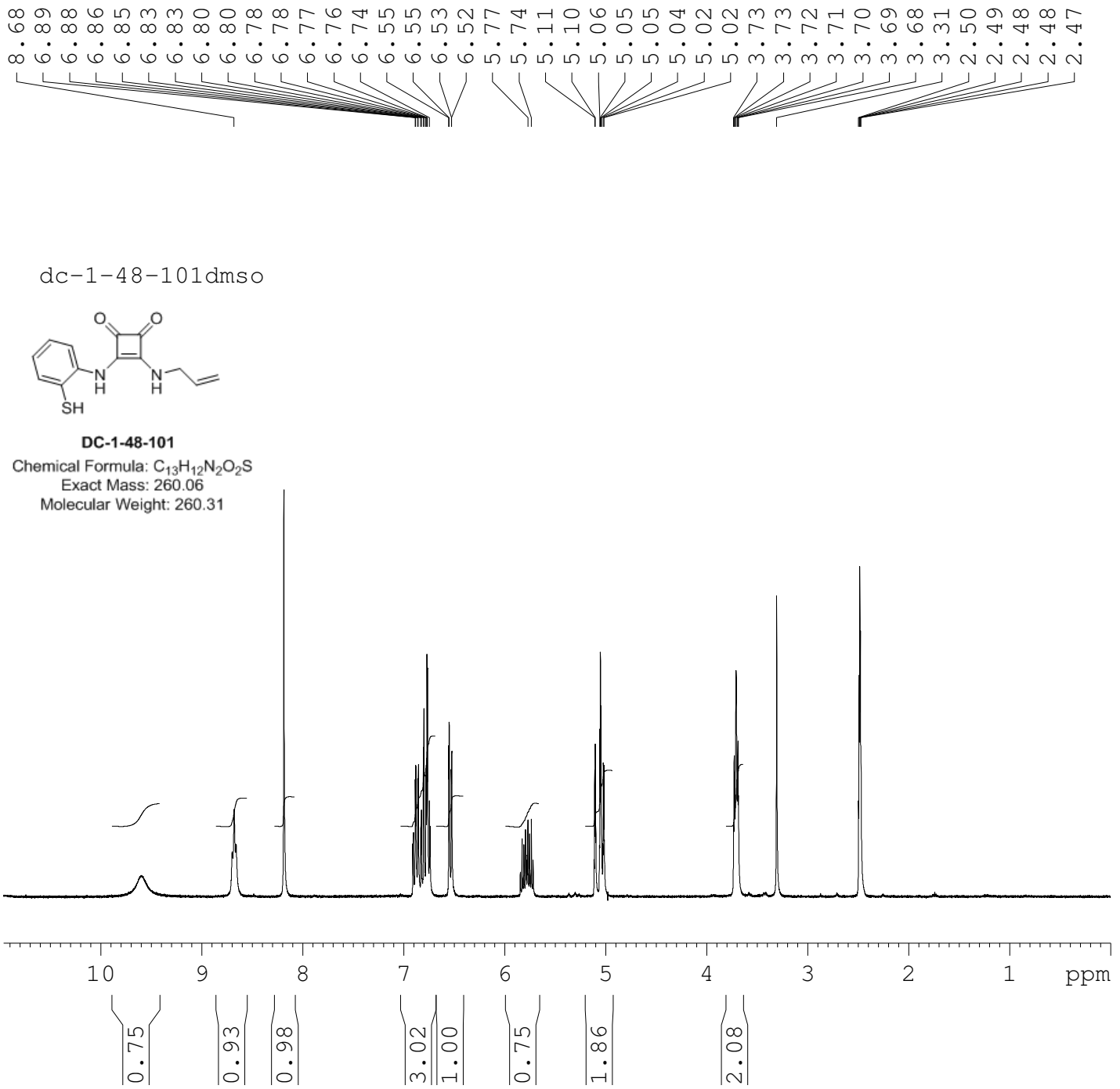
==========

Acq. Operator : Angela Hansen
Acq. Instrument : INSTRUMENT 1 Location : Vial 2
Injection Date : 12/17/2009 12:23:56 PM Inj : 1
Inj Volume : 10 µl
Acq. Method : D:\METHODS\POS_MIXED_LOOP_150_
Last changed : 12/17/2009 12:23:35 PM by Angela Hansen
(modified after loading)
Analysis Method : D:\METHODS\16MIN_150_1000_LCMS_BOTH_POS.M
Last changed : 12/15/2009 2:38:59 PM by David Dye
(modified after loading)
Method Info : Method for loop analysis of small organics

Sample Info : test
Easy-Access Method: 'pos_mixed_loop_150_1000'
Database #:

MS Spectrum



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PROCNO                  1

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Time               12.08
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT            DMSO
NS                   16
DS                    0
SWH            3591.954 Hz
FIDRES        0.109618 Hz
AQ            4.5613556 sec
RG                574.7
DW              139.200 usec
DE               54.00 usec
TE               298.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1        300.1315007 MHz

F2 - Processing parameters
SI                32768
SF          300.1300063 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```

dc-1-48-101

177.521
163.263
147.030
135.383
135.270
128.187
127.721
126.825
120.967
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102.404

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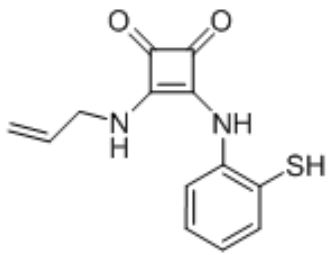
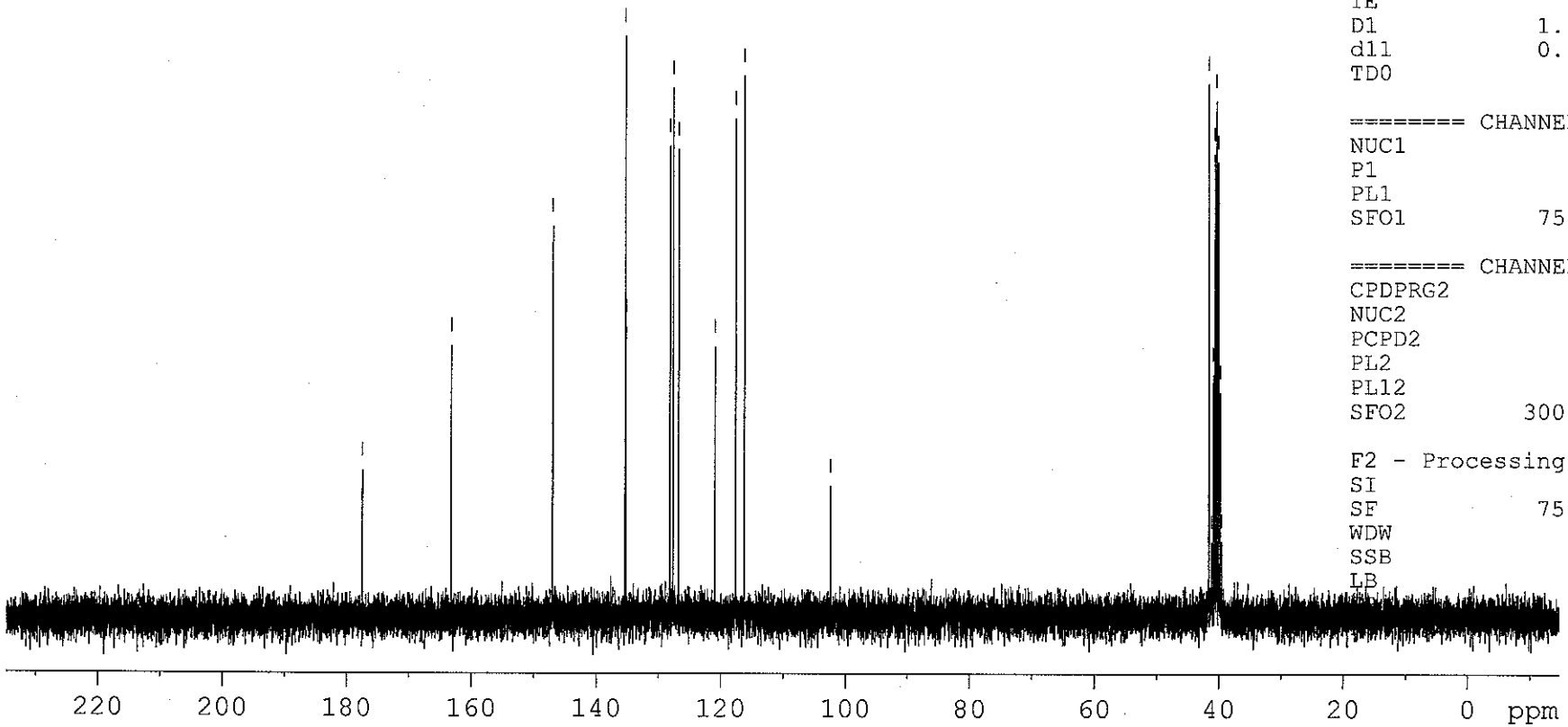
40.654
40.375
40.098
39.821
39.540





Current Data Parameters

NAME	dc-1-48-101
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

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Time	16.40
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PROBHD	5 mm QNP 1H/1
PULPROG	zgdc
TD	65536
SOLVENT	DMSO
NS	300
DS	0
SWH	18832.393 Hz
FIDRES	0.287360 Hz
AQ	1.7400308 sec
RG	1448.2
DW	26.550 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
d11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	13C
P1	11.80 usec
PL1	0.00 dB
SFO1	75.4760200 MHz

======== CHANNEL f2 ========

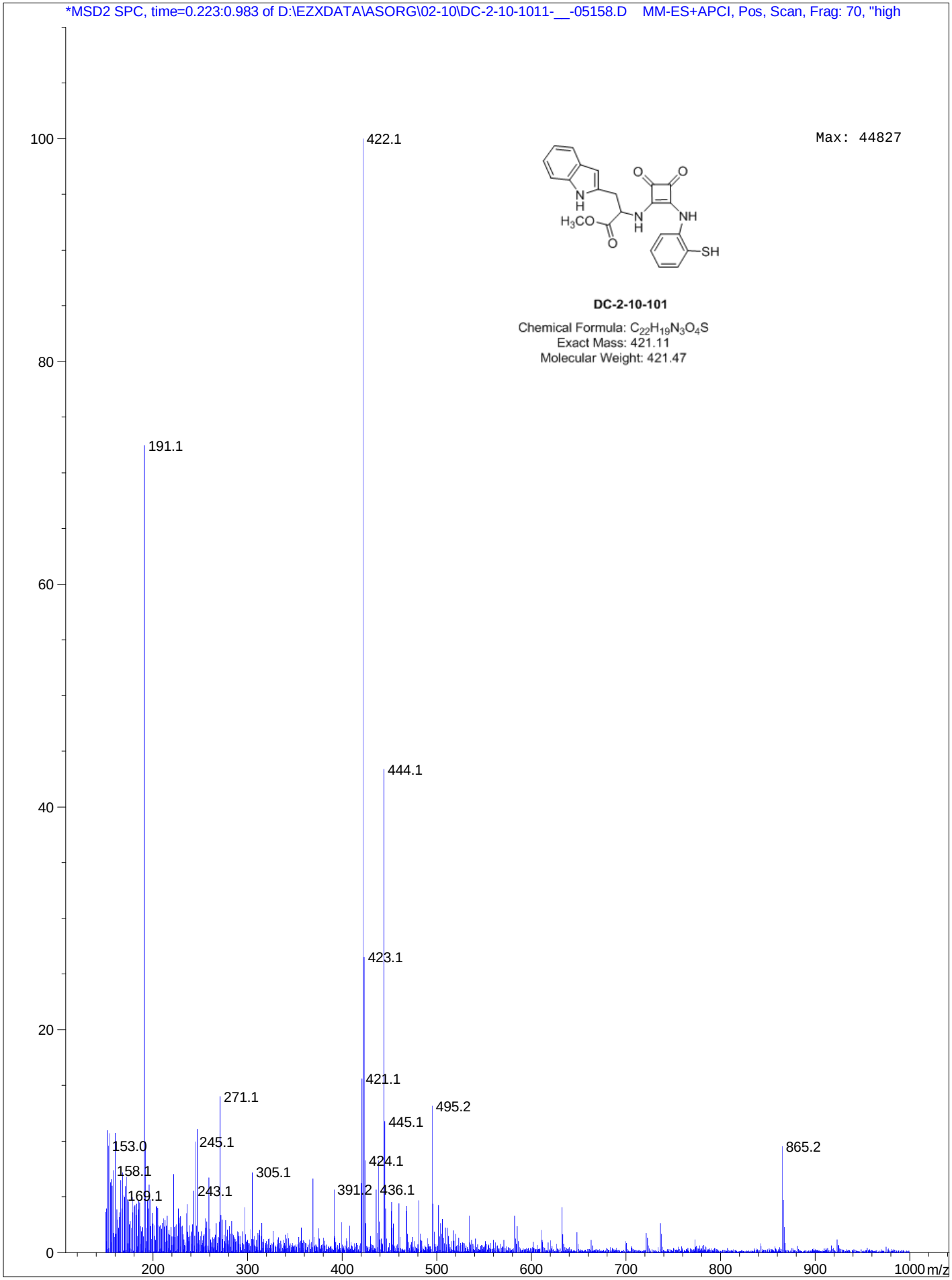
CPDPRG2	waltz16
NUC2	1H
PCPD2	95.00 usec
PL2	0.00 dB
PL12	18.00 dB
SFO2	300.1312005 MHz

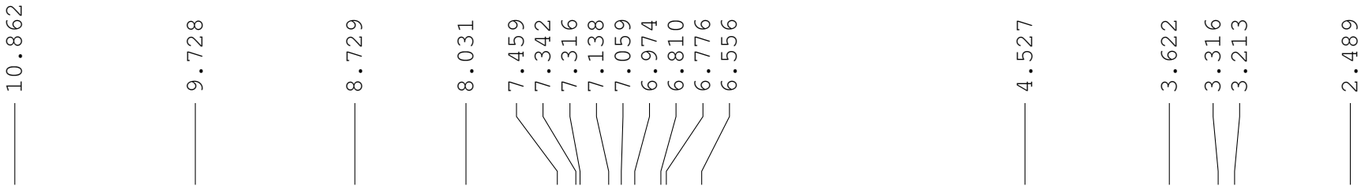
F2 - Processing parameters

SI	32768
SF	75.4677190 MHz
WDW	no
SSB	0
LB	0.00 Hz
	0
	1.40

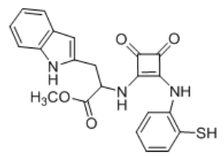
Supplementary Material (ESI) for Chemical Communications

MS Spectrum





dc-2-10-101



DC-2-10-101

Chemical Formula: $C_{22}H_{19}N_3O_4S$
Exact Mass: 421.11
Molecular Weight: 421.47

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NAME         dc-2-10-101
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100130
Time               15.02
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT            DMSO
NS                   16
DS                    0
SWH            3591.954 Hz
FIDRES         0.109618 Hz
AQ             4.5613556 sec
RG                 456.1
DW               139.200 usec
DE                54.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300042 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
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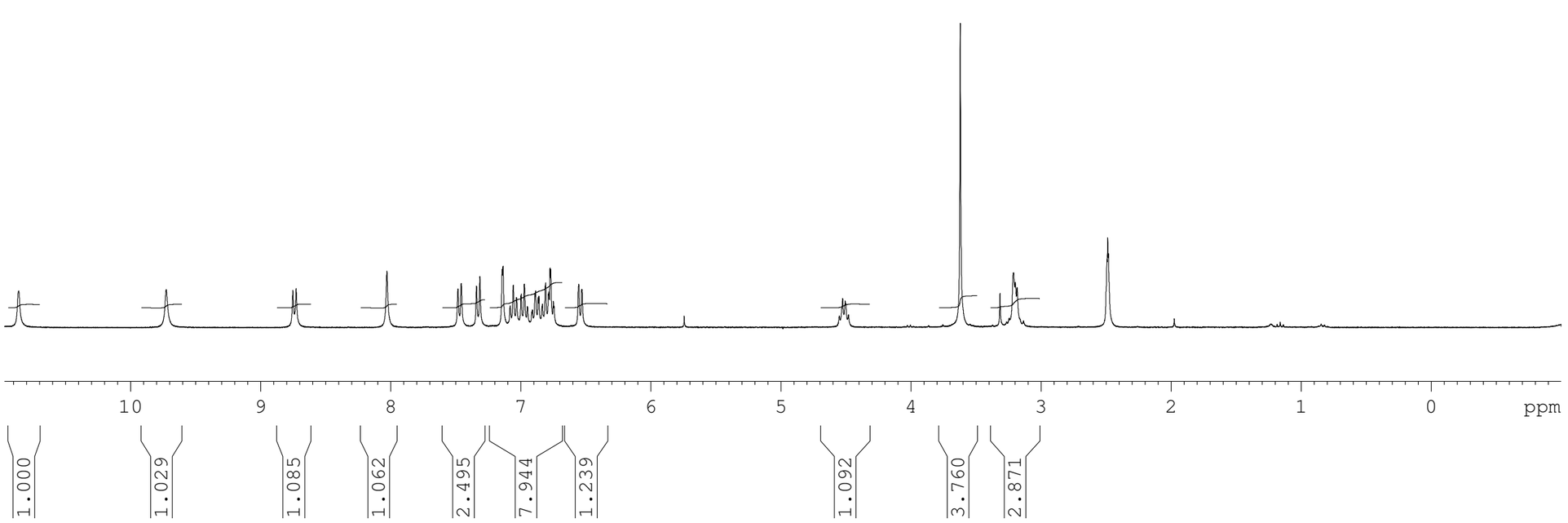
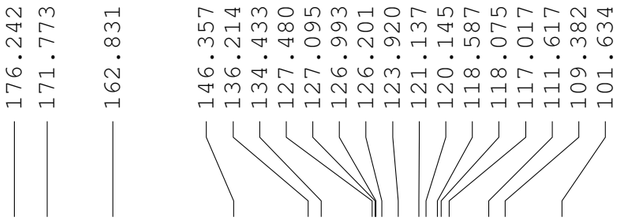
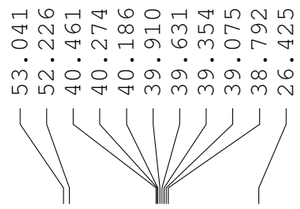
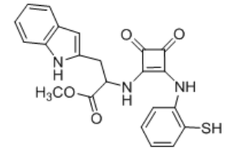




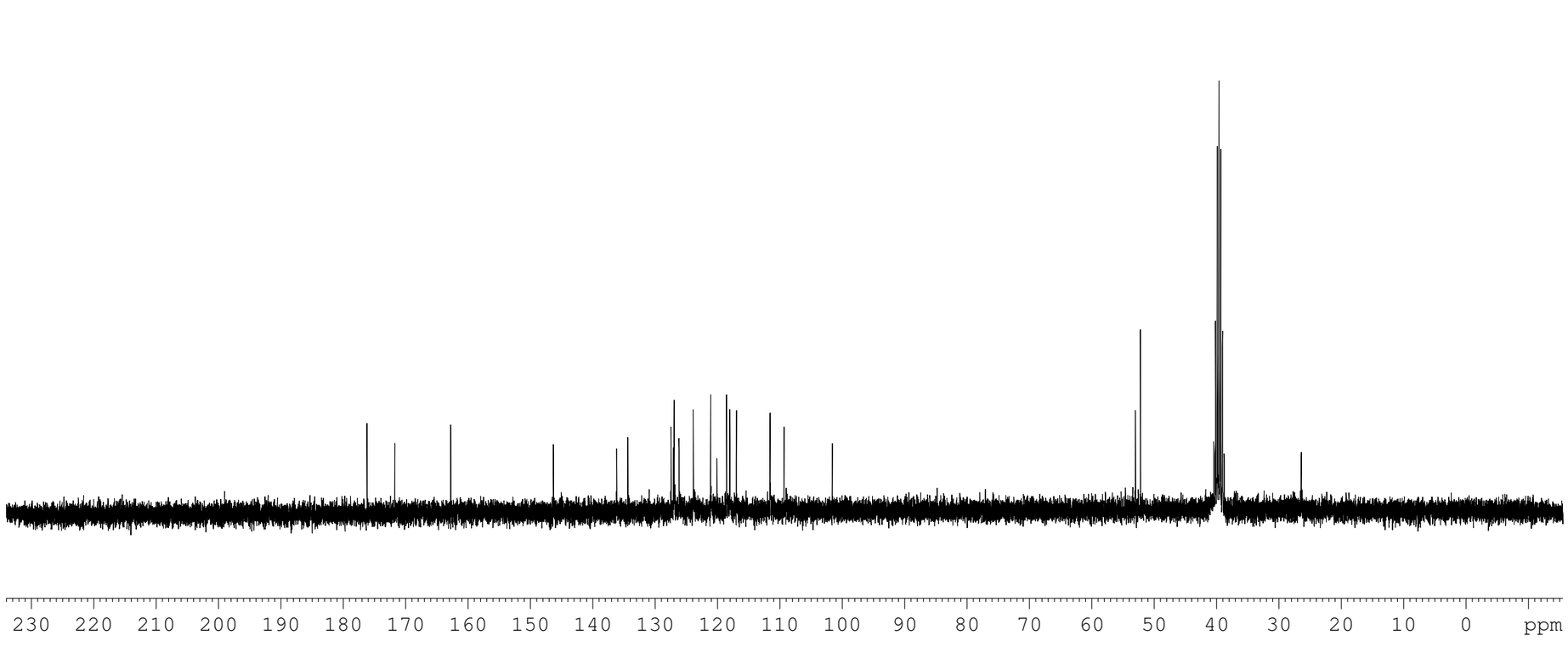


DC-2-10-carbon2



DC-2-10-101

Chemical Formula: $C_{22}H_{19}N_3O_4S$
Exact Mass: 421.11
Molecular Weight: 421.47

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Current Data Parameters
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PROCNO                  1

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INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG             zgdc
TD                 65536
SOLVENT             DMSO
NS                   800
DS                     0
SWH            18832.393 Hz
FIDRES          0.287360 Hz
AQ              1.7400308 sec
RG                 4597.6
DW                 26.550 usec
DE                  6.00 usec
TE                 298.2 K
D1            1.00000000 sec
d11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 11.80 usec
PL1                 0.00 dB
SFO1          75.4760200 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              95.00 usec
PL2                 0.00 dB
PL12               18.00 dB
SFO2         300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF            75.4677751 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
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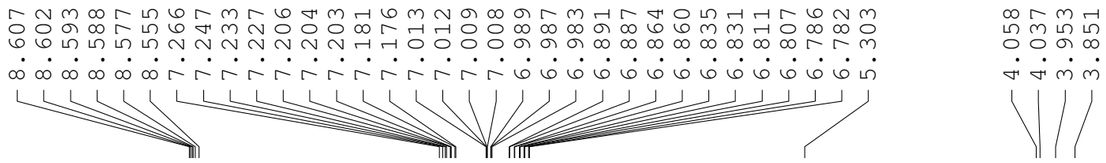
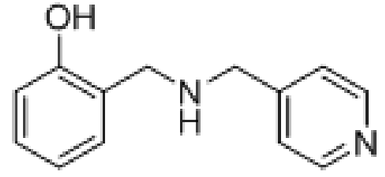
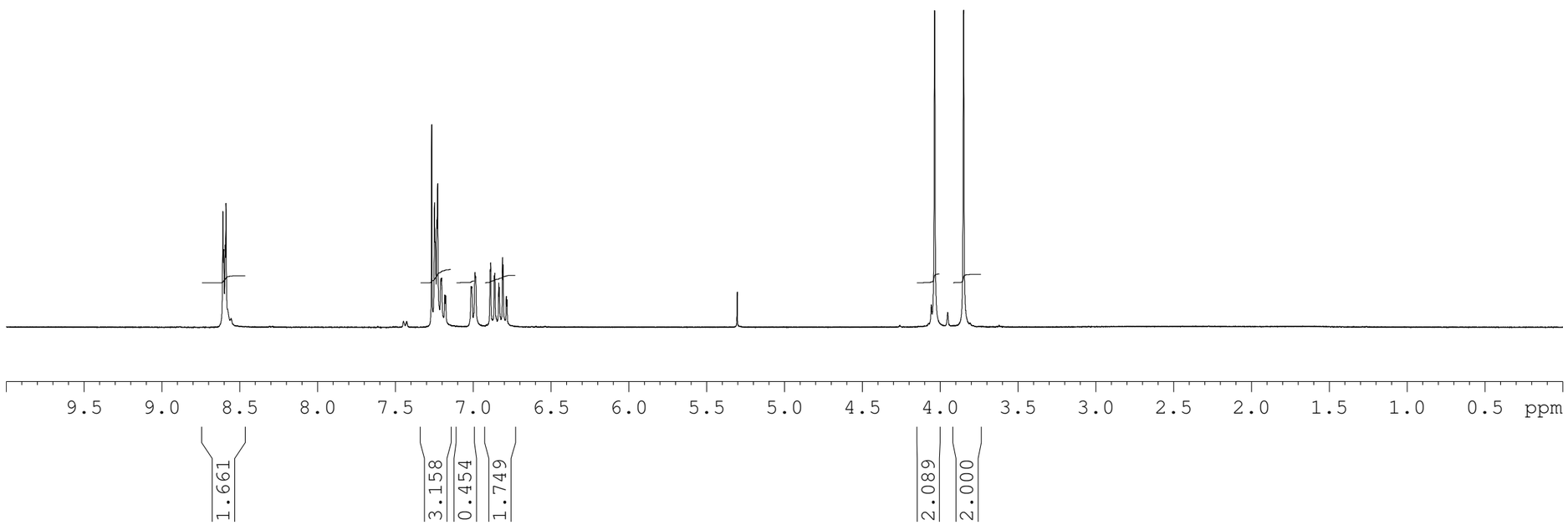


DC-2-32-102



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PROCNO                 1

F2 - Acquisition Parameters
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Time               18.41
INSTRUM            spect
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PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            3591.954 Hz
FIDRES          0.109618 Hz
AQ             4.5613556 sec
RG                574.7
DW                139.200 usec
DE                 54.00 usec
TE                 298.2 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF         300.1300042 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
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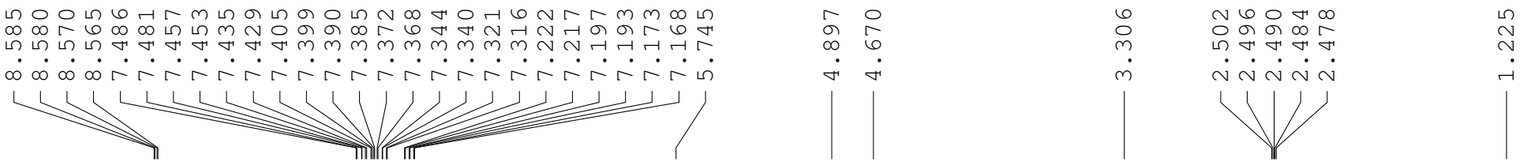
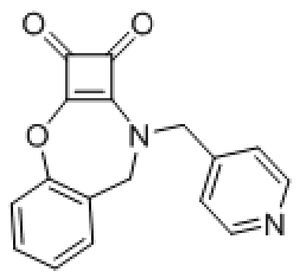
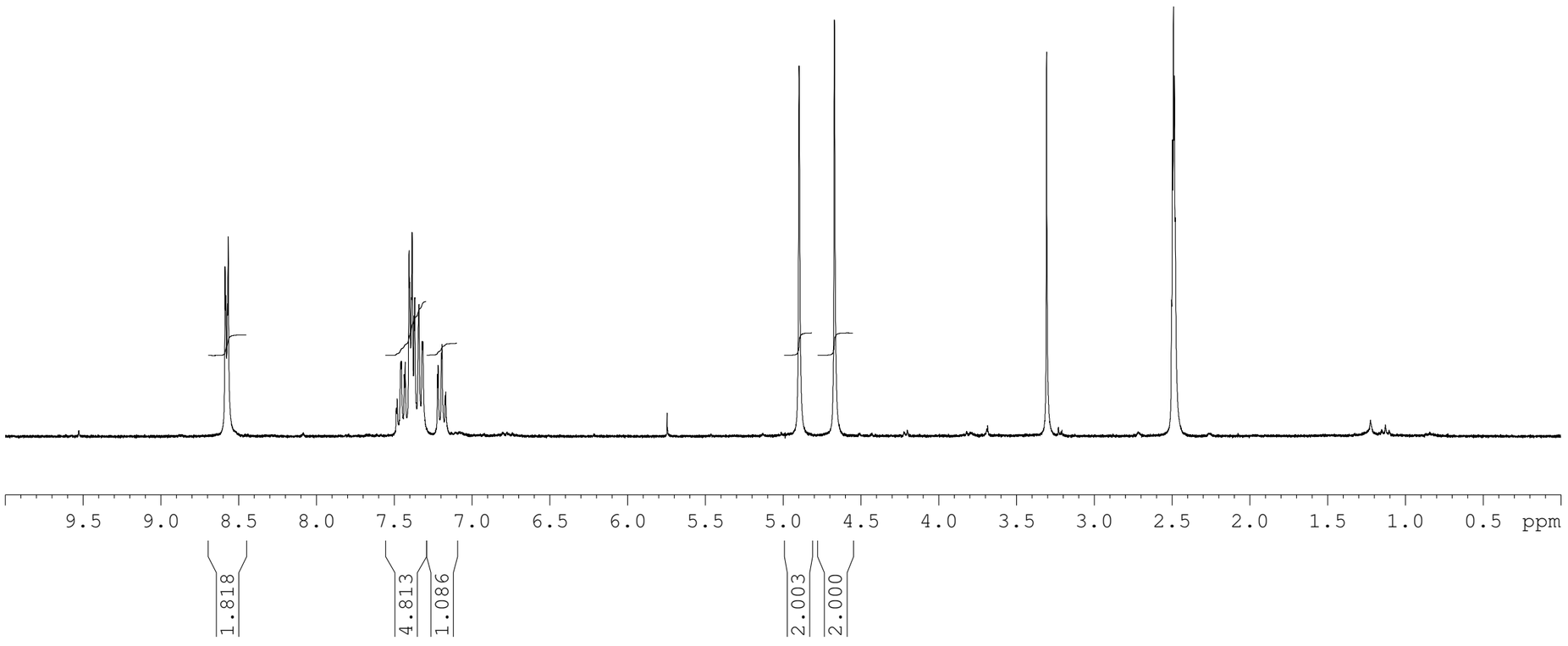


Dc-2-119-101



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EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100312
Time               16.03
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            3591.954 Hz
FIDRES        0.109618 Hz
AQ            4.5613556 sec
RG                 1024
DW              139.200 usec
DE               54.00 usec
TE               299.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1        300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300042 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
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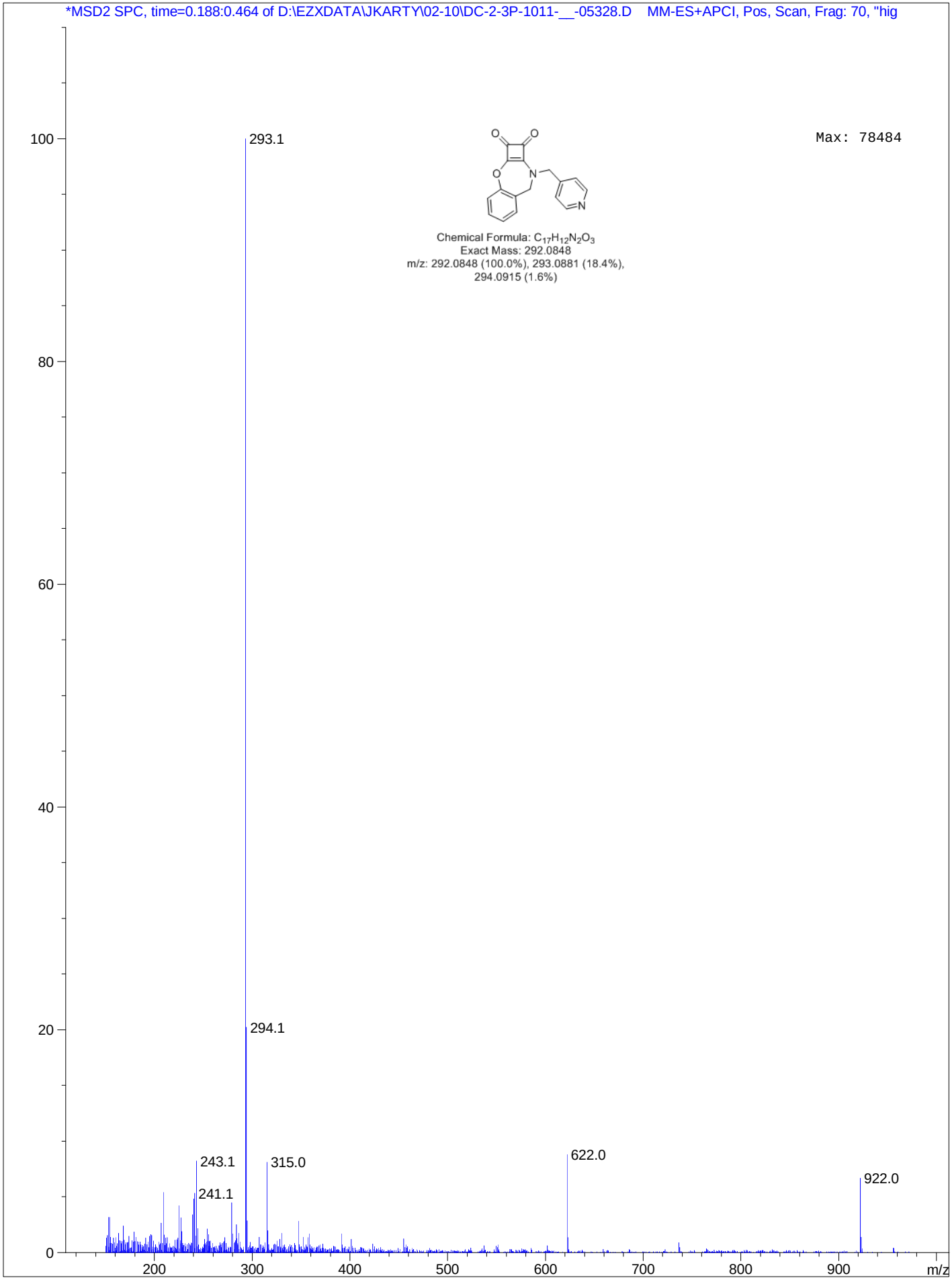
Supplementary Material (ESI) for Chemical Communications

MS Spectrum



8.978
8.957
8.128
8.108
7.512
7.486
7.461
7.410
7.386
7.383
7.346
7.320
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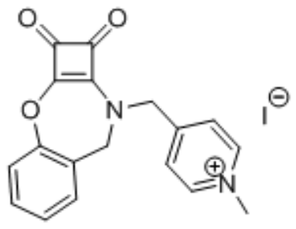
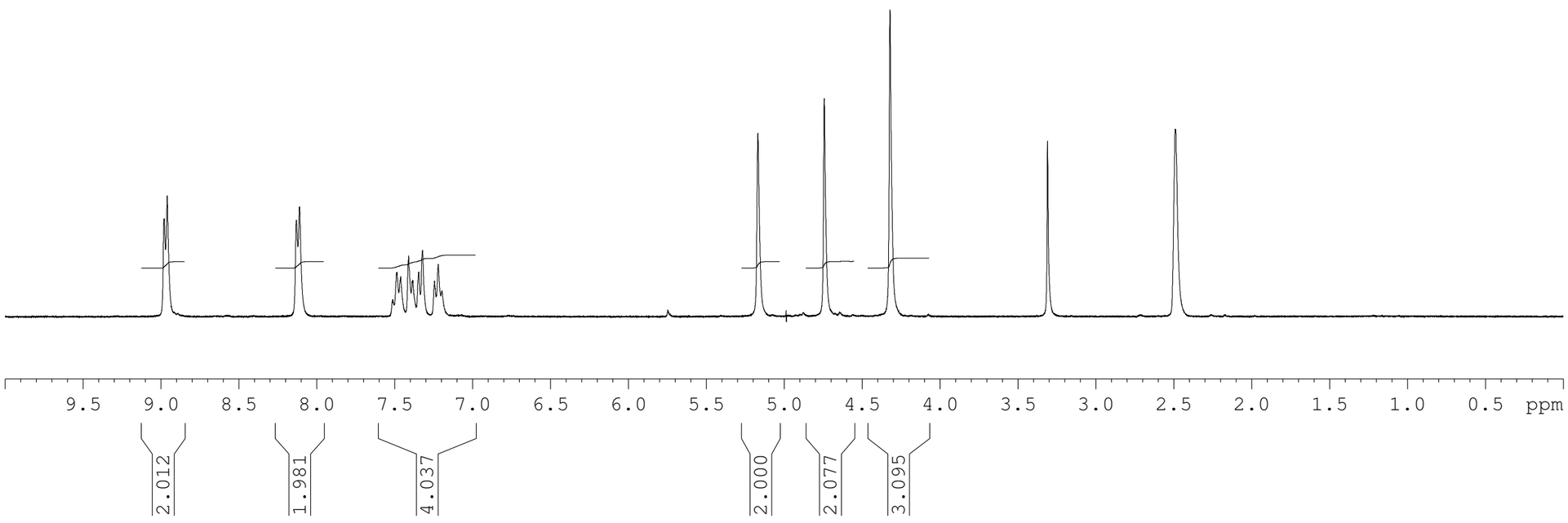
4.321
3.309
2.491
2.486

DC-2-45-103



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PROCNO                 1

F2 - Acquisition Parameters
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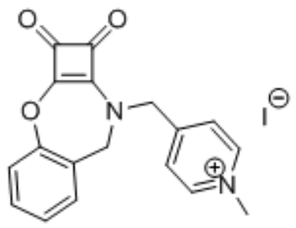
Time               15.04
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT            DMSO
NS                   32
DS                    0
SWH             3591.954 Hz
FIDRES          0.109618 Hz
AQ             4.5613556 sec
RG                645.1
DW                139.200 usec
DE                 54.00 usec
TE                 299.2 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300042 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```

DC-2-45-103-H2O

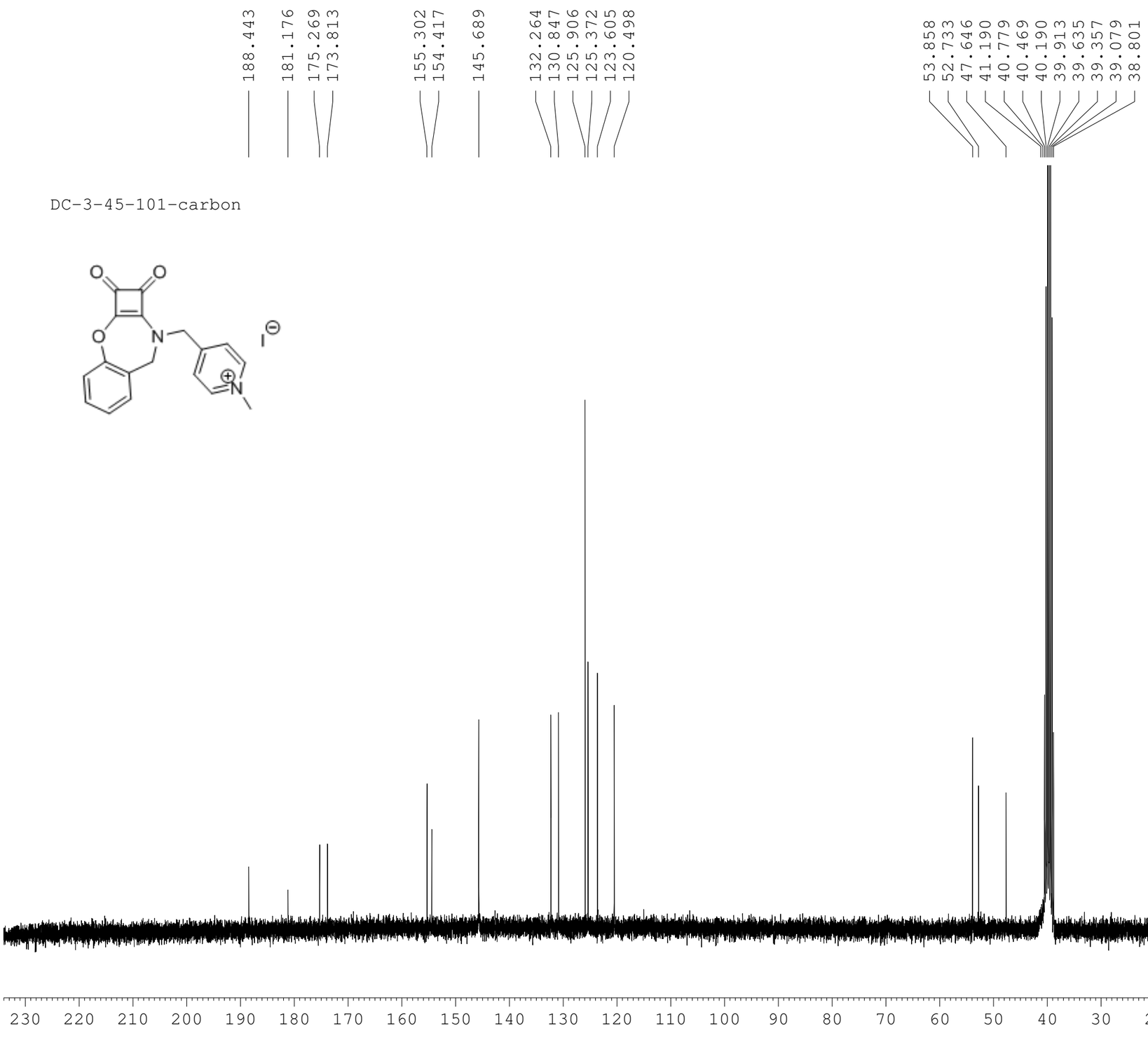



```
Current Data Parameters
NAME         DC-2-45-103-H2O
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20100218
Time               10.05
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT             D2O
NS                   32
DS                    0
SWH            3591.954 Hz
FIDRES          0.109618 Hz
AQ             4.5613556 sec
RG                  256
DW               139.200 usec
DE                54.00 usec
TE                298.2 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                9.75 usec
PL1               0.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI                32768
SF           300.1300042 MHz
WDW                  no
SSB                   0
LB                0.00 Hz
GB                    0
PC                1.00
```




```
Current Data Parameters
NAME      DC-3-45-101-carbon
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20100513
Time                7.53
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG             zgdc
TD                 65536
SOLVENT             DMSO
NS                 13000
DS                     0
SWH           18832.393 Hz
FIDRES         0.287360 Hz
AQ            1.7400308 sec
RG                  2048
DW                26.550 usec
DE                 6.00 usec
TE                297.2 K
D1           1.00000000 sec
d11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                11.80 usec
PL1                0.00 dB
SFO1         75.4760200 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             95.00 usec
PL2                0.00 dB
PL12              18.00 dB
SFO2        300.1312005 MHz

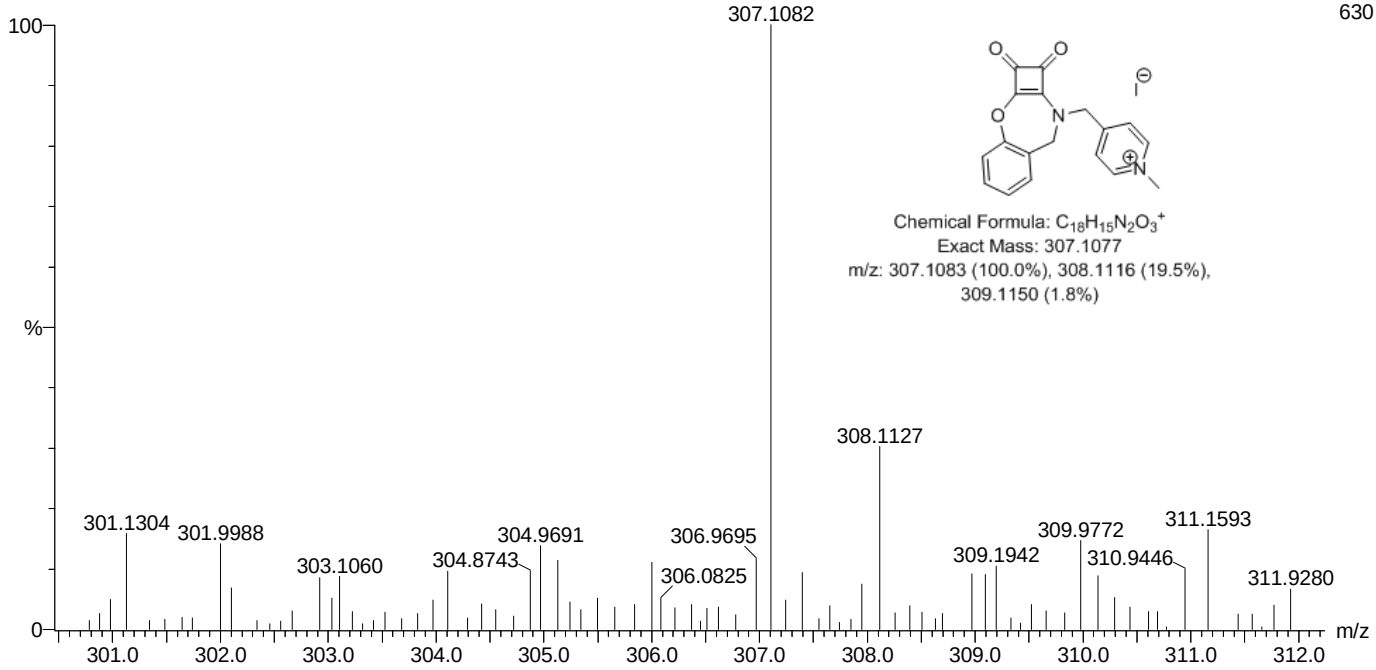
F2 - Processing parameters
SI                 32768
SF           75.4677751 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```

Elemental Composition Report

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

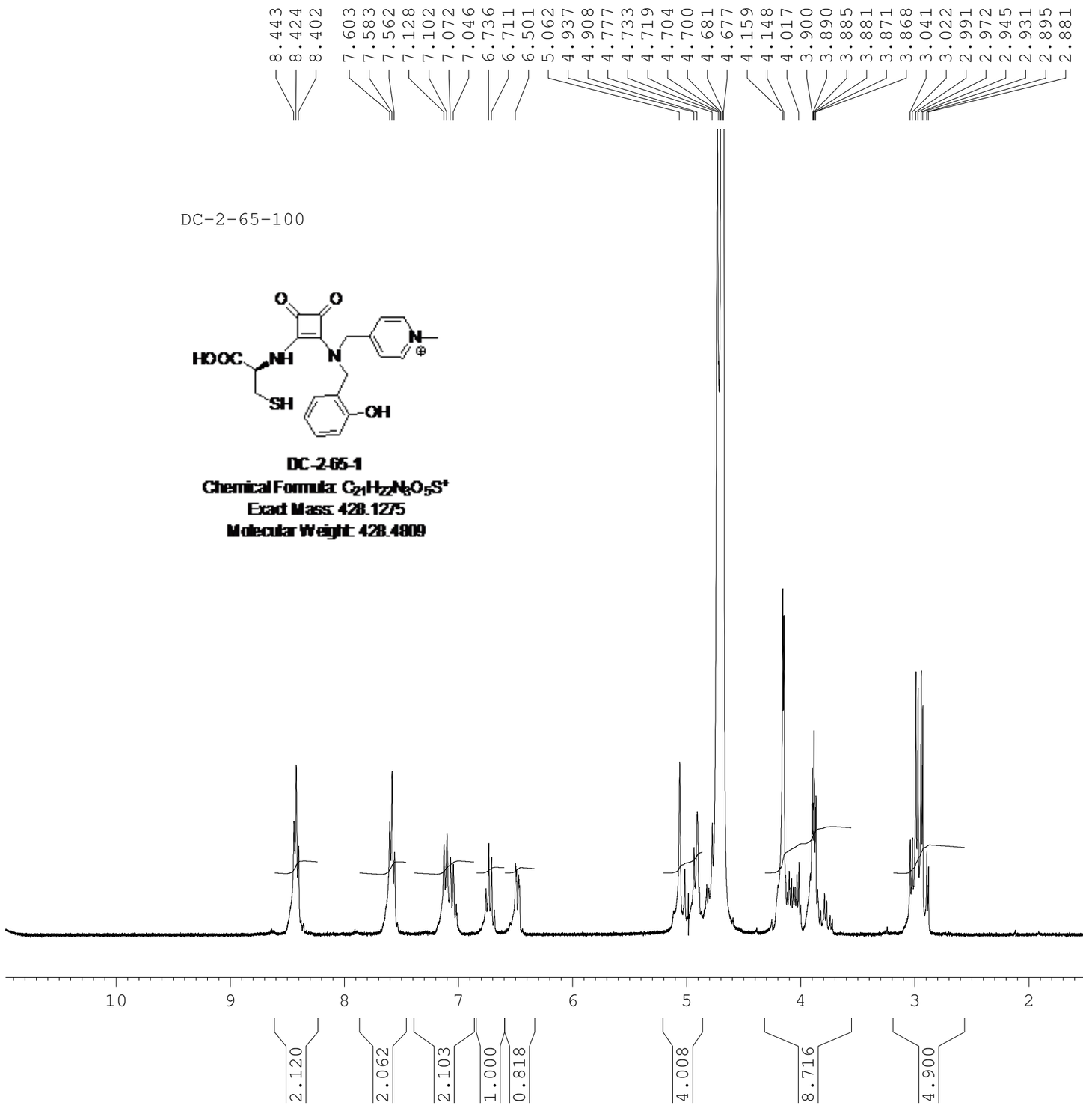
Monoisotopic Mass, Even Electron Ions
13 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-18 H: 0-15 N: 0-2 O: 0-3 Na: 0-1

File name:dc-2-45-103 Notebook Ref: IU Database#:
Instrument: LCT KC366 Test name: Accurate Mass 26-Feb-2010 14:45:30
Method: ESI-TOF Base Peak Mass: 307.10818481
dc-2-45-103c 28 (0.702) AM (Cen,6, 90.00, Ar,6000.0,1222.14,0.70,LS 4); Sm (SG, 2x6.00); Cm (27:28) 1: TOF MS ES+
630



Chemical Formula: $C_{18}H_{15}N_2O_3^+$
Exact Mass: 307.1077
m/z: 307.1083 (100.0%), 308.1116 (19.5%), 309.1150 (1.8%)

Minimum:	80.00				-1.5		
Maximum:	100.00		5.0	50.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
307.1082	100.00	307.1083	-0.1	-0.3	12.5	21.1	C18 H15 N2 O3




```
Current Data Parameters
NAME         DC-2-65-100
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100218
Time               11.07
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT             D2O
NS                   32
DS                    0
SWH            3591.954 Hz
FIDRES        0.109618 Hz
AQ            4.5613556 sec
RG                 362
DW               139.200 usec
DE                54.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1       300.1315007 MHz

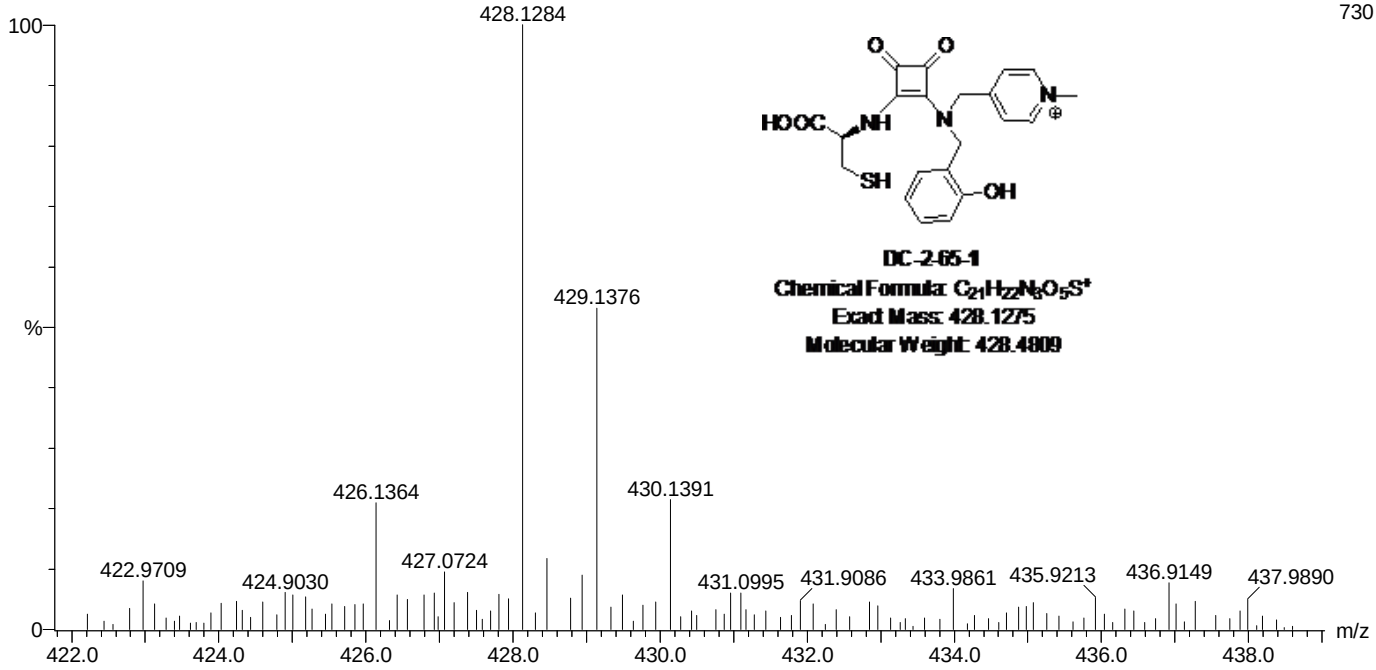
F2 - Processing parameters
SI                 32768
SF         300.1300042 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.00
```

Elemental Composition Report

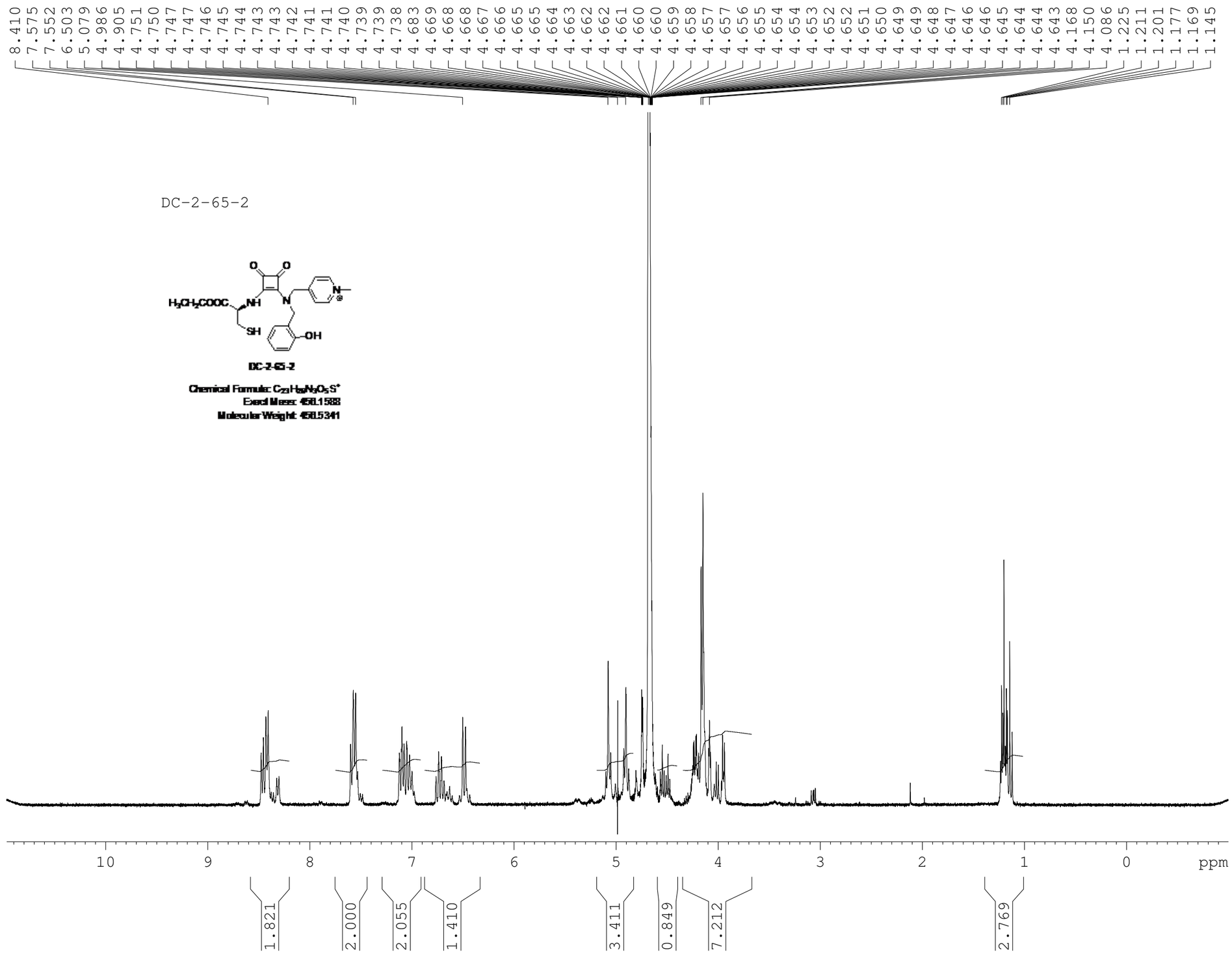
Tolerance = 50.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
81 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-21 H: 0-22 N: 0-3 O: 0-5 Na: 0-1 S: 0-1

File name:dc-2-65-1 Notebook Ref: IU Database#:
Instrument: LCT KC366 Test name: Accurate Mass 26-Feb-2010 12:25:12
Method: ESI-TOF Base Peak Mass: 428.12838745
dc-2-65-1 32 (0.802) AM (Cen,6, 90.00, Ar,6000.0,1222.14,0.70,LS 4); Sm (SG, 2x6.00); Cm (31:32) 1: TOF MS ES+
730



Minimum:	80.00				-1.5		
Maximum:	100.00		5.0	50.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
428.1284	100.00	428.1280	0.4	0.9	12.5	68.7	C21 H22 N3 O5 S




```
Current Data Parameters
NAME            DC-2-65-2
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100222
Time                9.07
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG               zg
TD                 32768
SOLVENT              D2O
NS                    32
DS                     0
SWH             3591.954 Hz
FIDRES          0.109618 Hz
AQ              4.5613556 sec
RG                   362
DW                139.200 usec
DE                 54.00 usec
TE                 298.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1          300.1315007 MHz

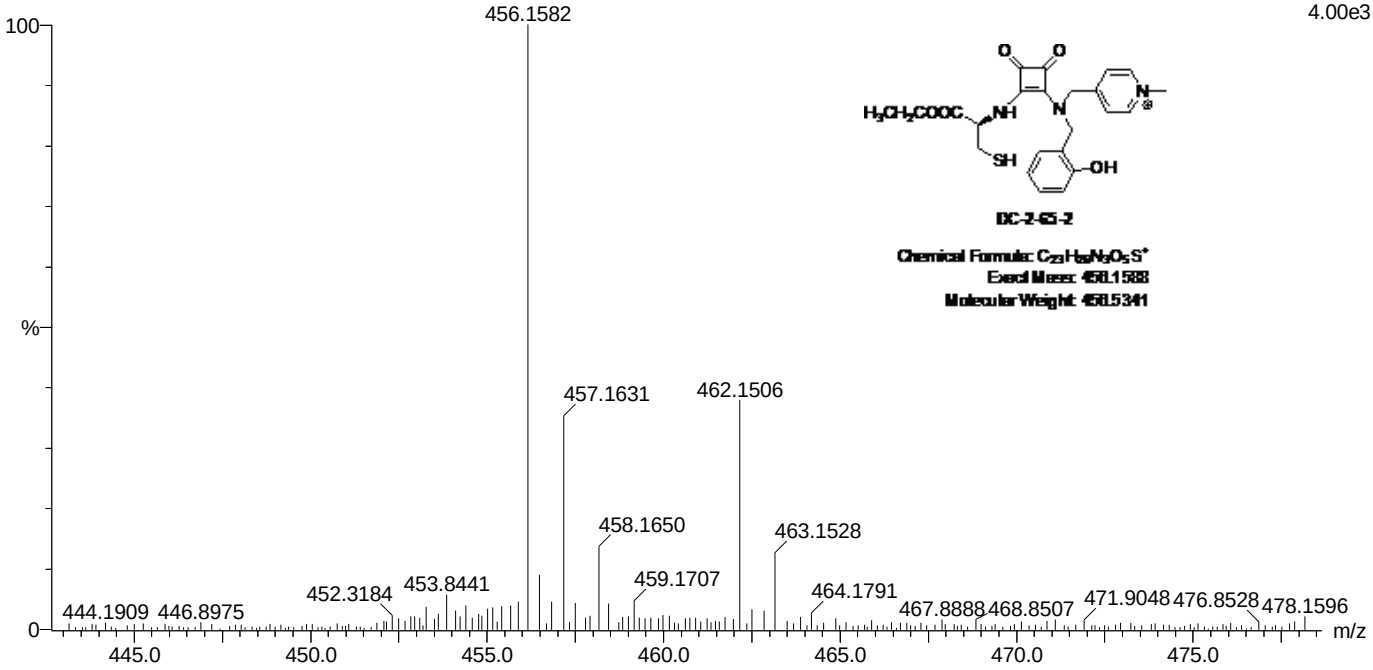
F2 - Processing parameters
SI                 32768
SF            300.1300042 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.00
```

Elemental Composition Report

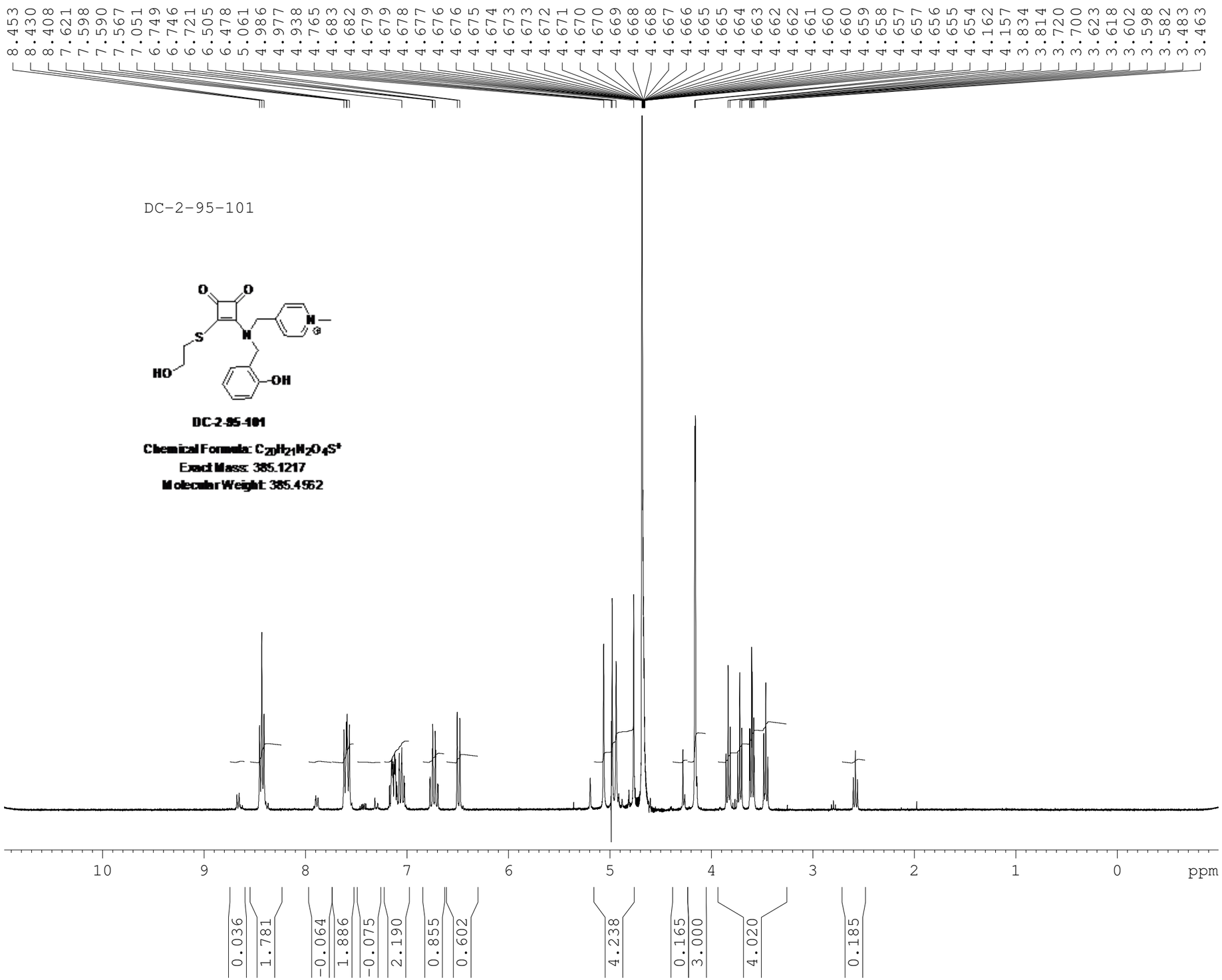
Tolerance = 50.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
81 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-23 H: 0-26 N: 0-3 O: 0-5 Na: 0-1 S: 0-1

File name:dc-2-65-2 Notebook Ref: IU Database#:
Instrument: LCT KC366 Test name: Accurate Mass 26-Feb-2010 12:21:02
Method: ESI-TOF Base Peak Mass: 456.15817261
dc-2-65-2 30 (0.752) AM (Cen,6, 90.00, Ar,6000.0,1222.14,0.70,LS 4); Sm (SG, 2x6.00); Cm (29:30) 1: TOF MS ES+
4.00e3



Minimum:	80.00				-1.5		
Maximum:	100.00		5.0	50.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
456.1582	100.00	456.1593	-1.1	-2.4	12.5	44.9	C23 H26 N3 O5 S




```
Current Data Parameters
NAME         DC-2-95-101
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100226
Time               18.36
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT             D2O
NS                   32
DS                    0
SWH           3591.954 Hz
FIDRES        0.109618 Hz
AQ            4.5613556 sec
RG                645.1
DW              139.200 usec
DE               54.00 usec
TE               299.2 K
D1           1.00000000 sec
TD0                   1

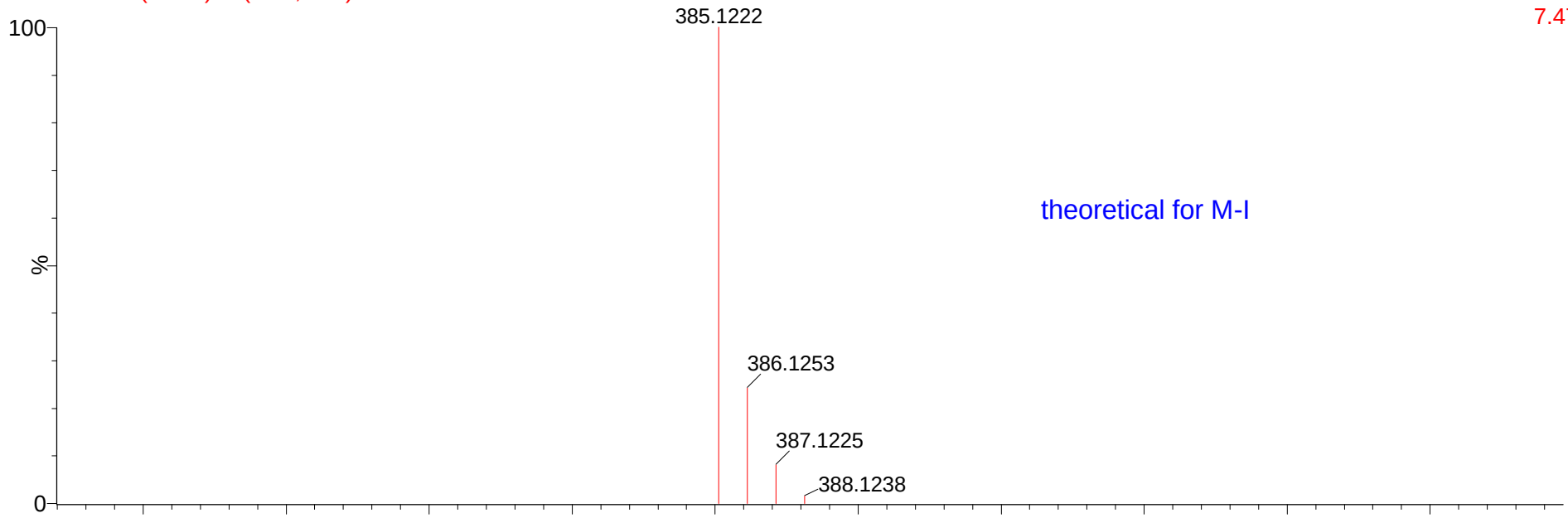
======== CHANNEL f1 ========
NUC1                 1H
P1                9.75 usec
PL1               0.00 dB
SFO1        300.1315007 MHz

F2 - Processing parameters
SI                32768
SF          300.1300042 MHz
WDW                  no
SSB                   0
LB                0.00 Hz
GB                    0
PC                 1.00
```

File name:dc-2-95-101 **Notebook Ref: 24821** **IU Database#: 24821**
Instrument: LCT KC366 **Test name: Accurate Mass** **06-May-2010 11:34:47**
Method: ESI-TOF **Base Peak Mass: 387.13412476**

dc-2-94-201 (0.035) Is (0.50,0.50) C20H21N2O4S

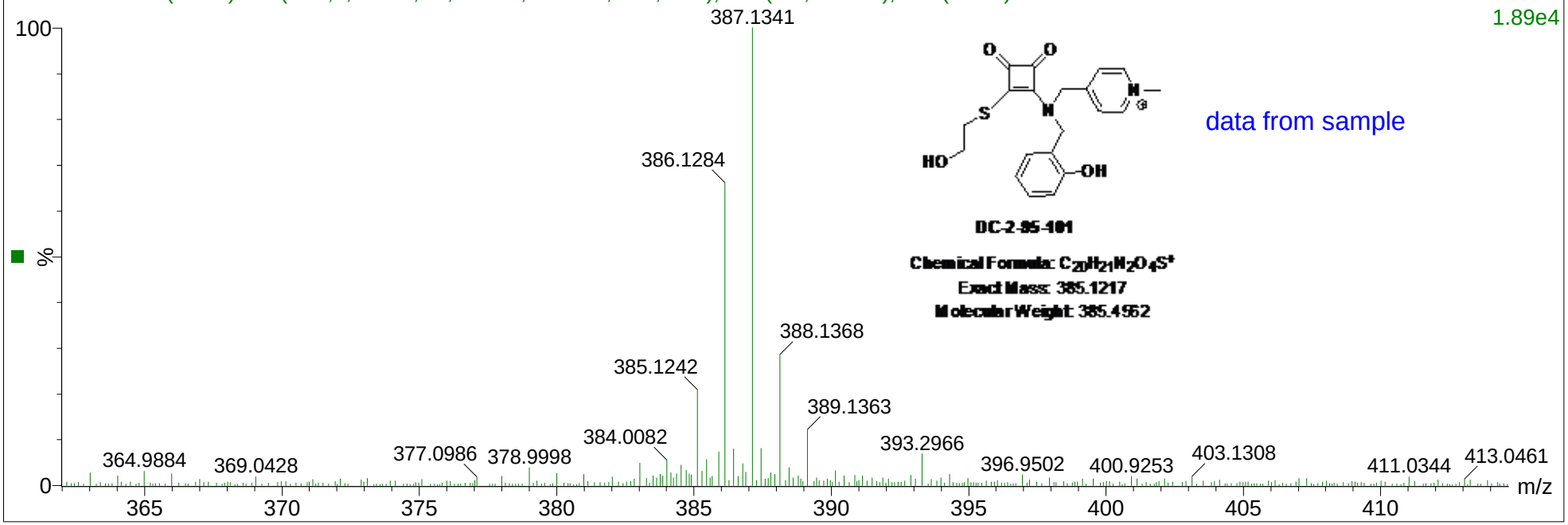
1: TOF MS ES+
7.47e12



theoretical for M-I

dc-2-95-101 64 (1.603) AM (Cen,8, 90.00, Ar,4000.0,1222.14,0.70,LS 6); Sm (SG, 2x4.00); Cm (63:71)

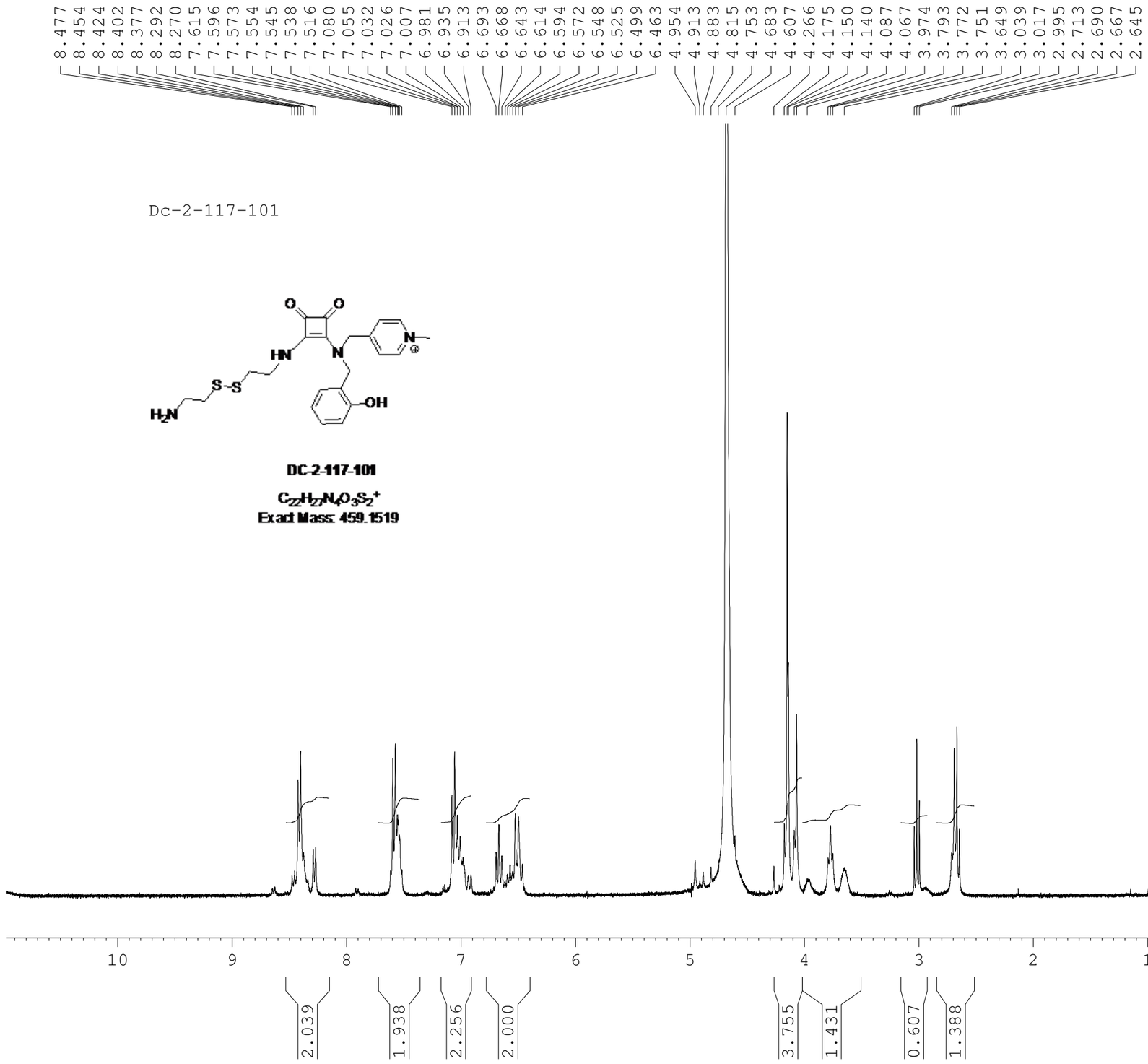
1: TOF MS ES+
1.89e4



DC-2-95-101

Chemical Formula: $C_{20}H_{21}N_2O_4S^+$
Exact Mass: 385.1217
Molecular Weight: 385.4562

data from sample




```
Current Data Parameters
NAME        Dc-2-117-101
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100310
Time               15.18
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT             D2O
NS                   32
DS                    0
SWH            3591.954 Hz
FIDRES        0.109618 Hz
AQ           4.5613556 sec
RG                 256
DW              139.200 usec
DE               54.00 usec
TE               299.2 K
D1          1.00000000 sec
TD0                    1

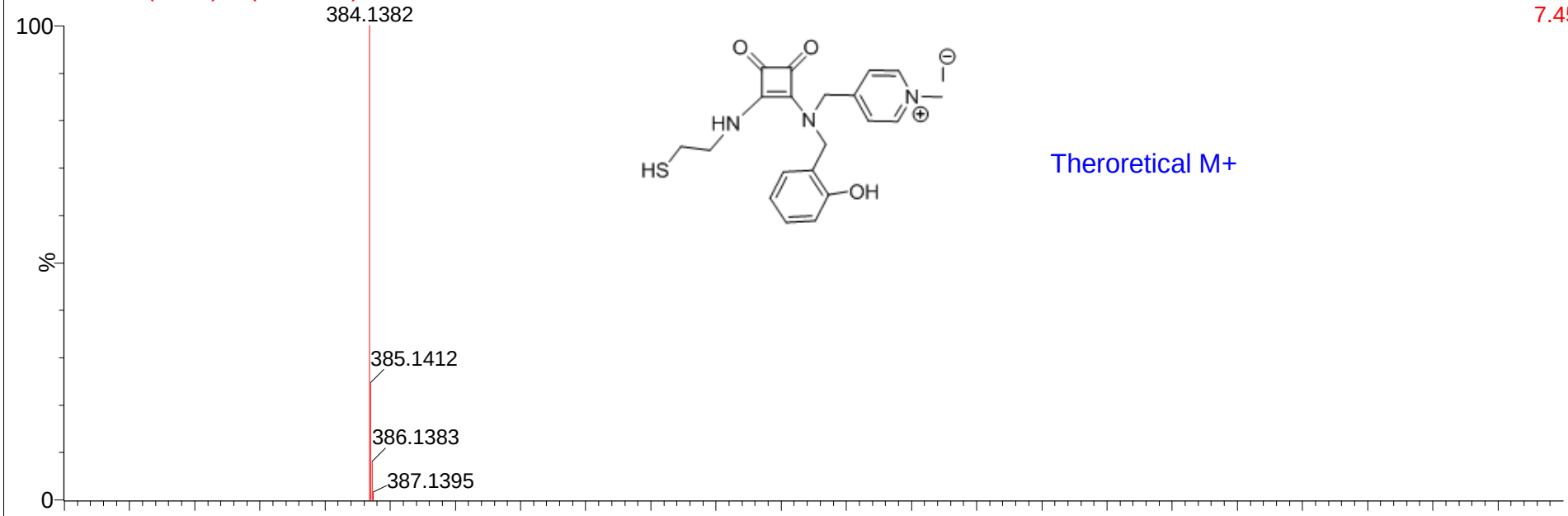
======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1        300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300042 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```

File name:dc-2-117-101B **Notebook Ref: 24952** **IU Database#: 24952**
Instrument: LCT KC366 **Test name: Accurate Mass** **13-May-2010 12:39:59**
Method: ESI-TOF **Base Peak Mass: 459.15127563**

dc-3-86-101 (0.035) Is (0.50,0.50) C20H22N3O3S

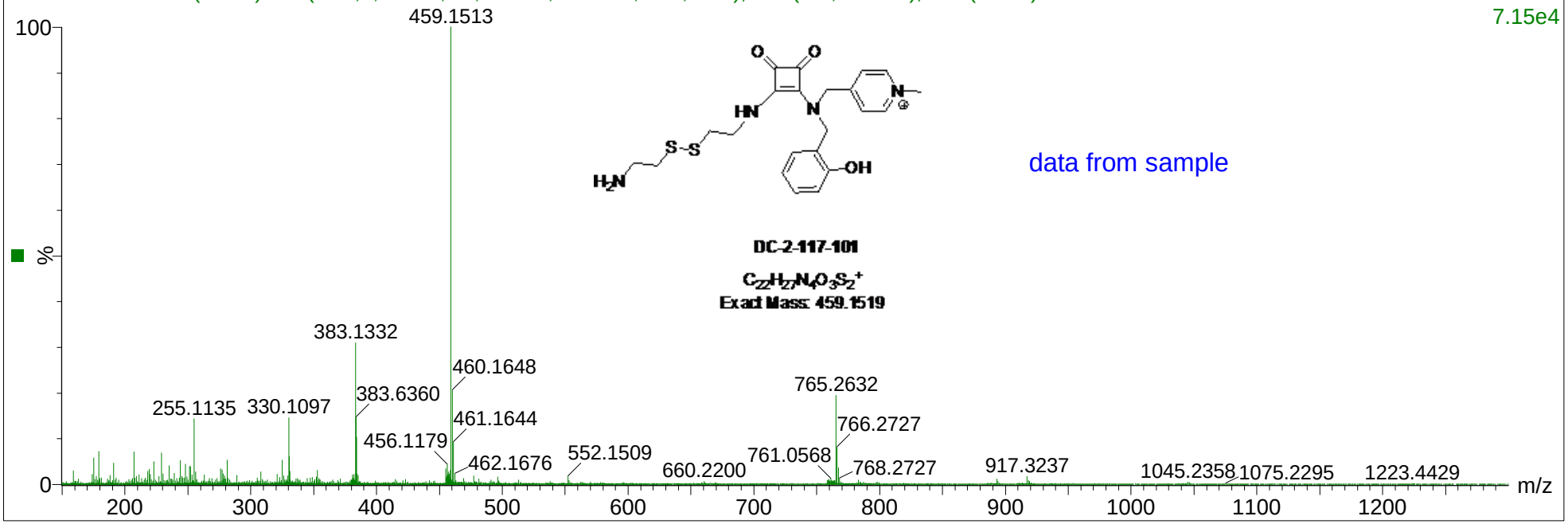
1: TOF MS ES+
7.45e12



Theroretical M+

dc-2-117-101B 34 (0.852) AM (Cen,8, 90.00, Ar,5000.0,1222.14,0.70,LS 6); Sm (SG, 2x4.00); Cm (23:40)

1: TOF MS ES+
7.15e4



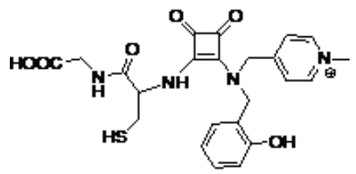
data from sample

DC-2-117-101

$C_{22}H_{27}N_4O_3S_2^+$

Exact Mass: 459.1519

DC-2-92-101



DC-2-92-1

Chemical Formula: $C_{23}H_{25}N_4O_5S^+$
Exact Mass: 485.1489
Molecular Weight: 485.5323

```
Current Data Parameters
NAME          DC-2-92-101
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20100226
Time                14.48
INSTRUM             spect
PROBHD   5 mm QNP  1H/1
PULPROG               zg
TD                 32768
SOLVENT              D2O
NS                    32
DS                     0
SWH              3591.954 Hz
FIDRES           0.109618 Hz
AQ              4.5613556 sec
RG                   256
DW                139.200 usec
DE                 54.00 usec
TE                 299.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.75 usec
PL1                 0.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300042 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```

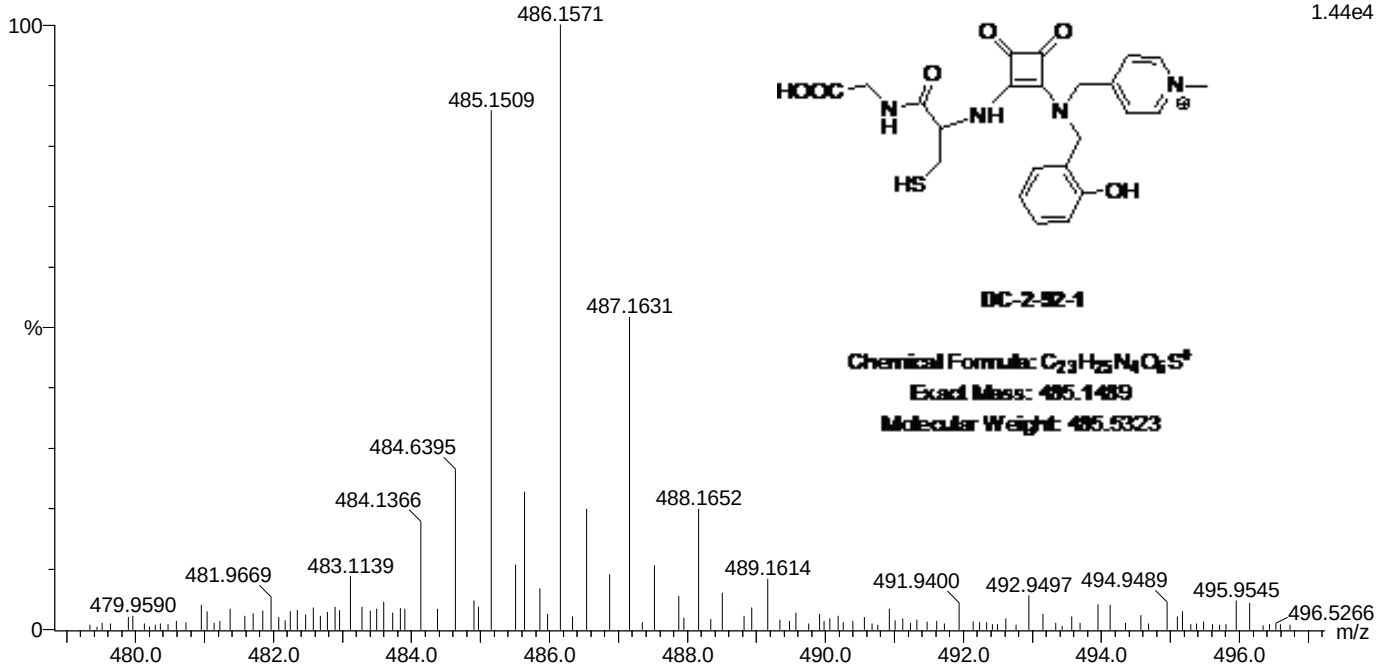
Elemental Composition Report

Multiple Mass Analysis: 2 mass(es) processed

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

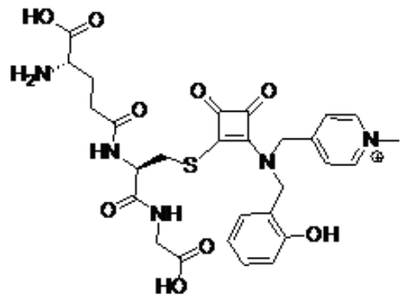
Monoisotopic Mass, Even Electron Ions
31200 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 23-23 N: 0-4 O: 0-6 Na: 0-1 S: 0-1 2H: 1-5 1H: 1-29

File name:dc-2-92-101 Notebook Ref: 24694 IU Database#: 24694
Instrument: LCT KC366 Test name: Accurate Mass 28-Apr-2010 12:17:42
Method: ESI-TOF Base Peak Mass: 486.15707397
dc-2-92-101 31 (0.785) AM (Cen,6, 90.00, Ar,4000.0,922.36,0.70,LS 4); Sm (SG, 2x4.00); Cm (29:45) 1: TOF MS ES+
1.44e4



Minimum:	80.00				-1.5		
Maximum:	100.00		5.0	10.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
485.1509	85.78	485.1464	4.5	9.3	14.5	8216.9	C23 N4 O6 S 2H2 1H21
486.1571	100.00	486.1558	1.3	2.7	13.5	953.5	C23 N4 O6 S 2H 1H24
		486.1527	4.4	9.1	14.5	2915.8	C23 N4 O6 S 2H3 1H20

DC-3-82-101-5min



DC-3-82-101

Chemical Formula: $C_{28}H_{32}N_5O_9S^+$
Exact Mass: 614.1915
Molecular Weight: 614.6462

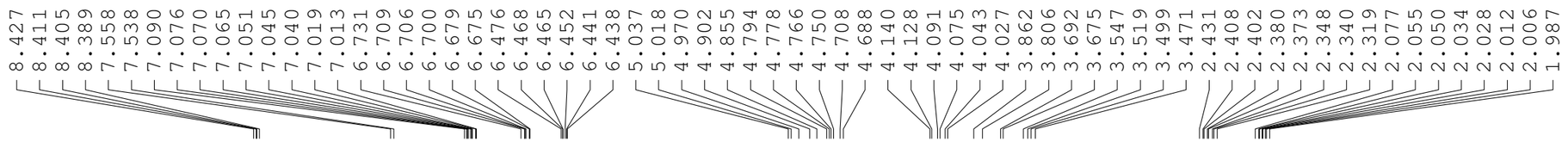
8.656 8.634 8.435 8.419 8.398 7.878 7.857 7.569 7.547 7.284 7.146 7.133 7.099 7.094 7.081 7.075 7.056 7.051 7.024 6.740 6.715 6.684 6.484 6.473 6.458 6.446 5.171 5.045 5.021 4.974 4.905 4.865 4.801 4.784 4.773 4.755 4.700 4.688 4.635 4.259 4.149 4.137 4.096 4.080 4.048 4.033 3.865 3.808 3.721 3.695 3.677 3.553 3.526 3.506 3.478 2.838 2.819 2.813 2.438 2.414 2.387 2.353 2.325 2.084 2.066 2.044 2.019 1.994


```
Current Data Parameters
NAME         DC-3-82-101-5min
EXPNO                       1
PROCNO                      1

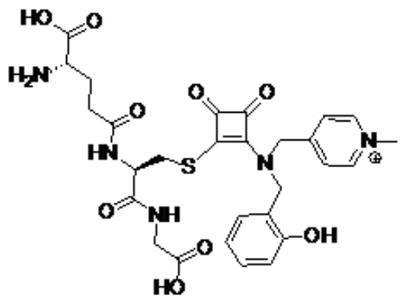
F2 - Acquisition Parameters
Date_              20100503
Time                  17.31
INSTRUM               spect
PROBHD    5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT                D2O
NS                      32
DS                       0
SWH               4789.272 Hz
FIDRES            0.146157 Hz
AQ                3.4210291 sec
RG                     512
DW                 104.400 usec
DE                  54.00 usec
TE                  296.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.75 usec
PL1                  0.00 dB
SFO1          300.1324000 MHz

F2 - Processing parameters
SI                   32768
SF            300.1300024 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```



DC-3-82-101-1h



DC-3-82-101

Chemical Formula: $C_{28}H_{32}N_5O_9S^+$
Exact Mass: 614.1915
Molecular Weight: 614.6462


```
Current Data Parameters
NAME      DC-3-82-101-1h
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100504
Time                8.55
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT             D2O
NS                   32
DS                    0
SWH            4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                 512
DW               104.400 usec
DE                54.00 usec
TE                683.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1        300.1324000 MHz

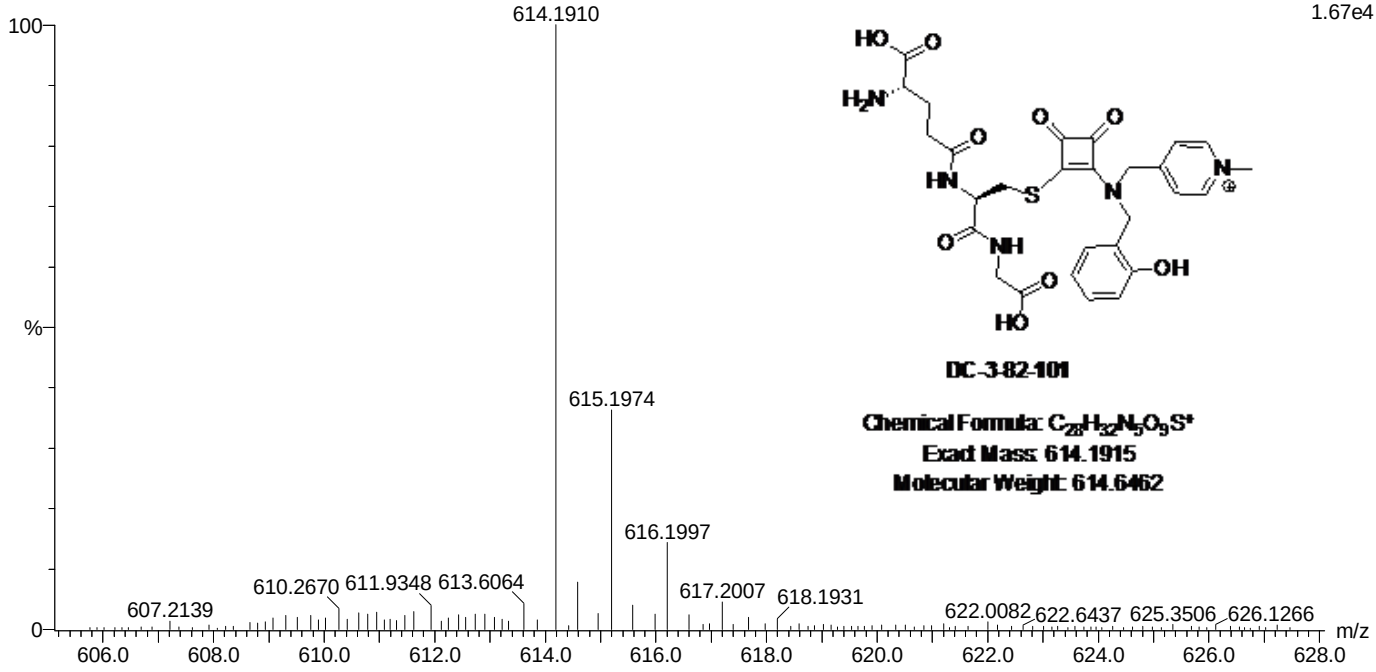
F2 - Processing parameters
SI                 32768
SF          300.1300024 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```

Elemental Composition Report

Tolerance = 20.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

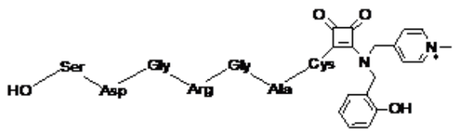
Monoisotopic Mass, Even Electron Ions
114 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-28 H: 0-32 N: 0-5 O: 0-9 S: 0-1

File name:dc-3-82-101 Notebook Ref: 24823 IU Database#: 24823
Instrument: LCT KC366 Test name: Accurate Mass 06-May-2010 11:43:09
Method: ESI-TOF Base Peak Mass: 614.19097900
dc-3-82-101 57 (1.436) AM (Cen,8, 90.00, Ar,4000.0,1222.14,0.70,LS 6); Sm (SG, 2x4.00); Cm (57:60) 1: TOF MS ES+
1.67e4



Minimum:	80.00				-1.5		
Maximum:	100.00		5.0	20.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
614.1910	100.00	614.1921	-1.1	-1.8	15.5	30.9	C28 H32 N5 O9 S

DC-3-86-101-5min



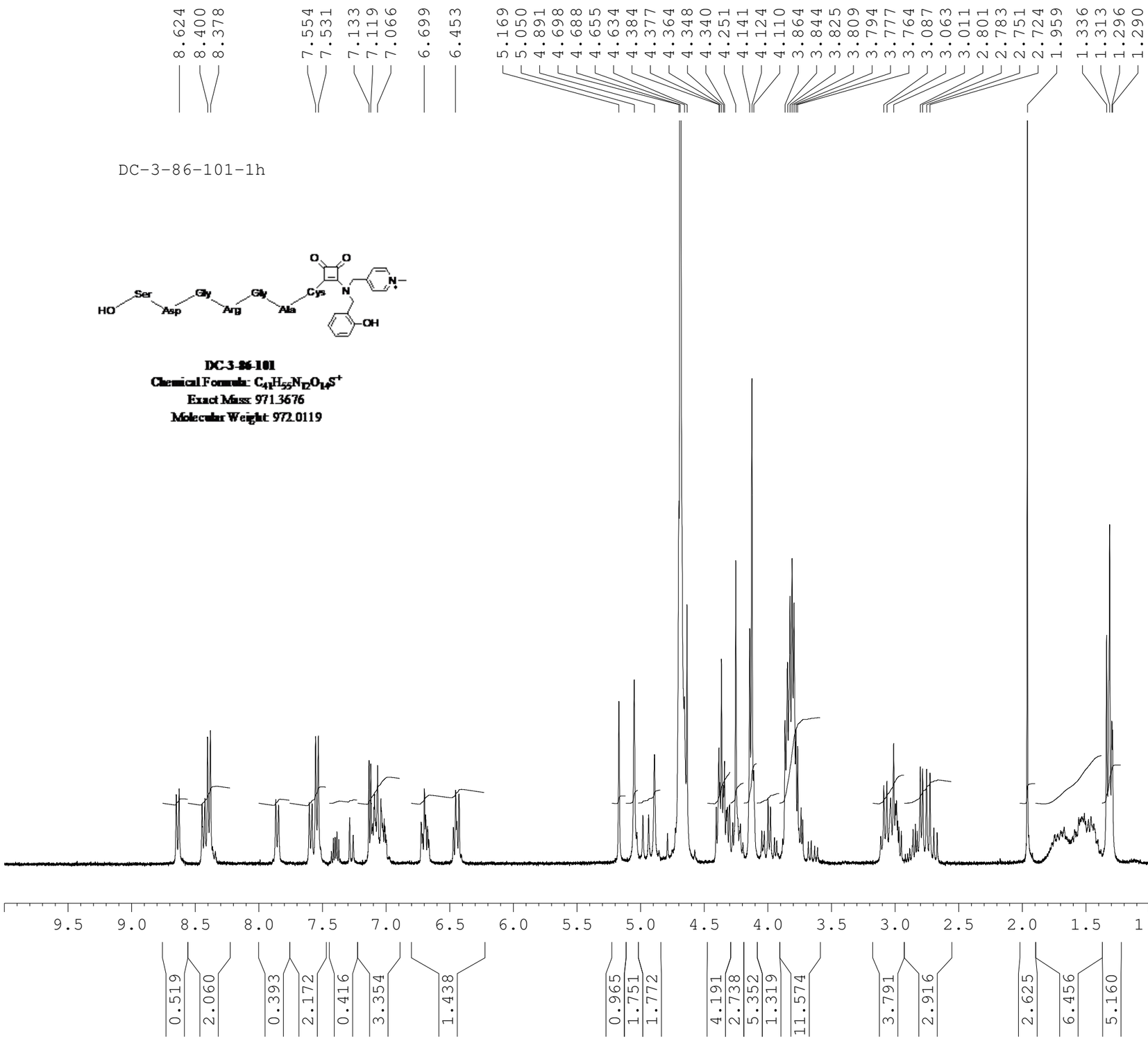
DC-3-86-101
Chemical Formula: $C_{41}H_{55}N_{12}O_{14}S^+$
Exact Mass: 971.3676
Molecular Weight: 972.0119

```
Current Data Parameters
NAME         DC-3-86-101-5min
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20100505
Time                  15.45
INSTRUM               spect
PROBHD    5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT                D2O
NS                      64
DS                       0
SWH                4789.272 Hz
FIDRES             0.146157 Hz
AQ                 3.4210291 sec
RG                     512
DW                   104.400 usec
DE                    54.00 usec
TE                    683.2 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      9.75 usec
PL1                     0.00 dB
SFO1            300.1324000 MHz

F2 - Processing parameters
SI                      32768
SF              300.1300024 MHz
WDW                        no
SSB                         0
LB                      0.00 Hz
GB                          0
PC                      1.00
```

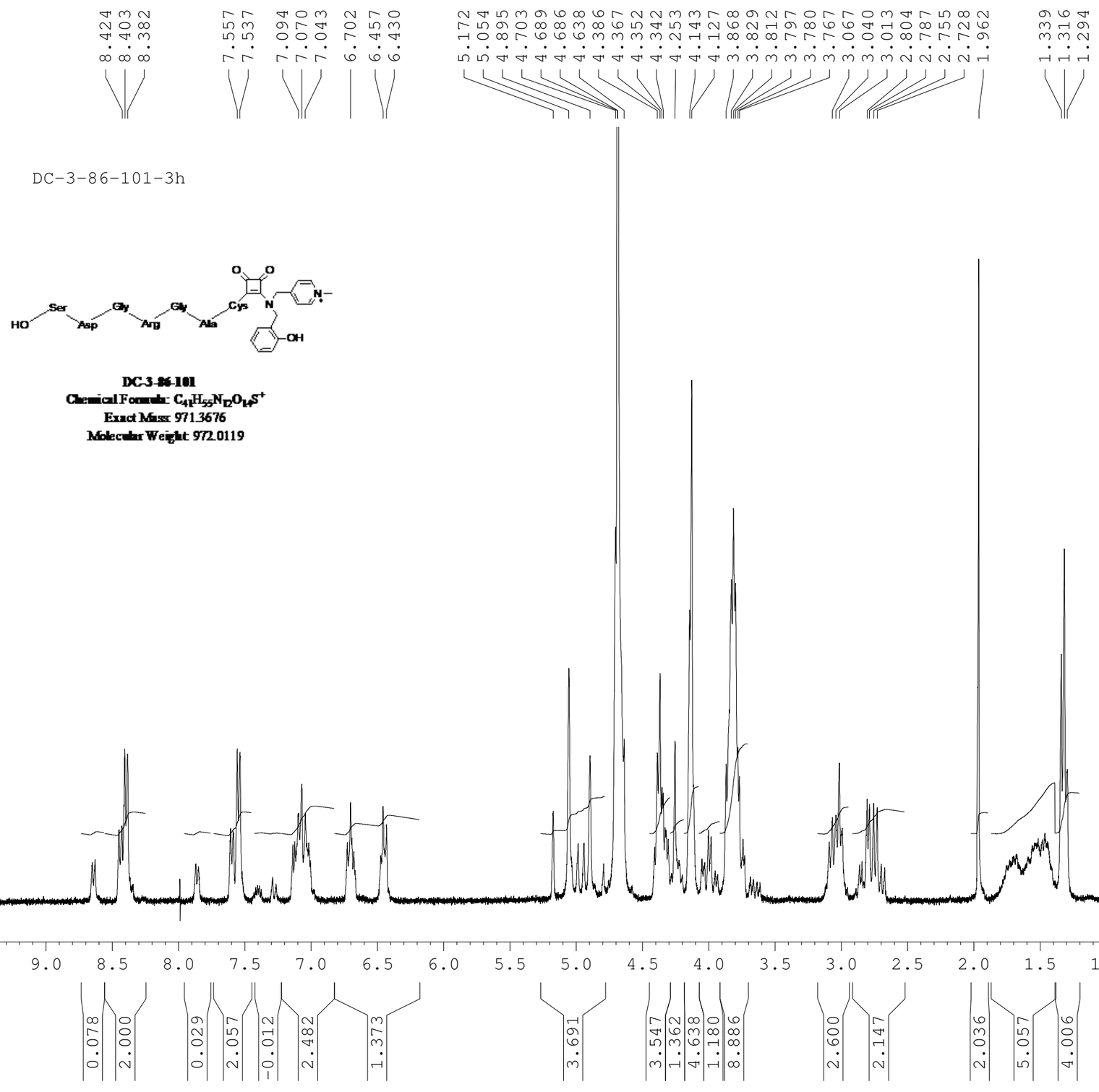



```
Current Data Parameters
NAME      DC-3-86-101-1h
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100505
Time               16.33
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT             D2O
NS                   64
DS                    0
SWH            4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                 512
DW             104.400 usec
DE              54.00 usec
TE              683.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1       300.1324000 MHz

F2 - Processing parameters
SI                 32768
SF       300.1300024 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                  1.00
```

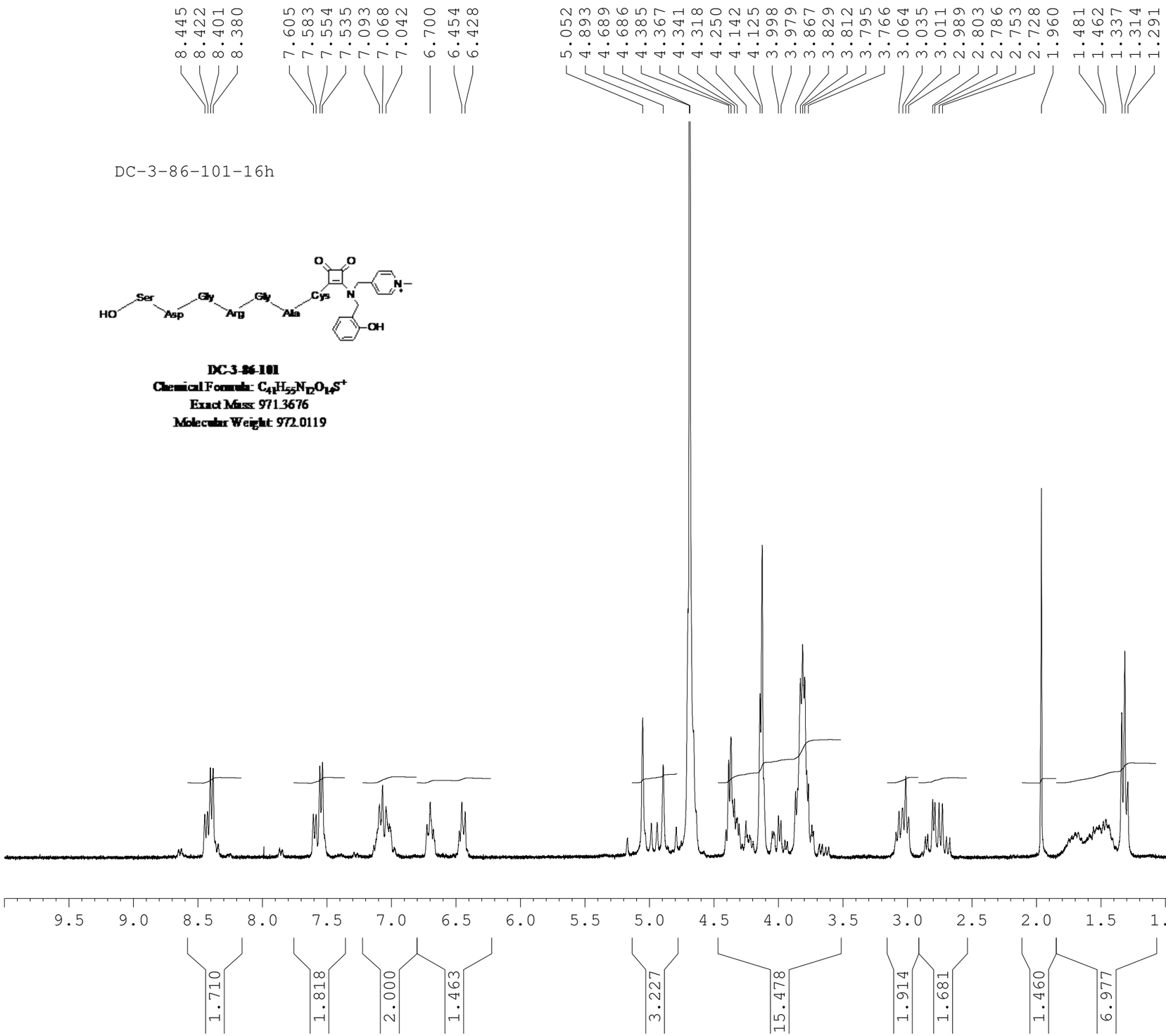



```
Current Data Parameters
NAME      DC-3-86-101-3h
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100505
Time               18.32
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT             D2O
NS                   32
DS                    0
SWH            4789.272 Hz
FIDRES        0.146157 Hz
AQ            3.4210291 sec
RG                 512
DW               104.400 usec
DE                54.00 usec
TE                683.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1        300.1324000 MHz

F2 - Processing parameters
SI                 32768
SF          300.1300024 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME      DC-3-86-101-16h
EXPNO                   1
PROCNO                  1

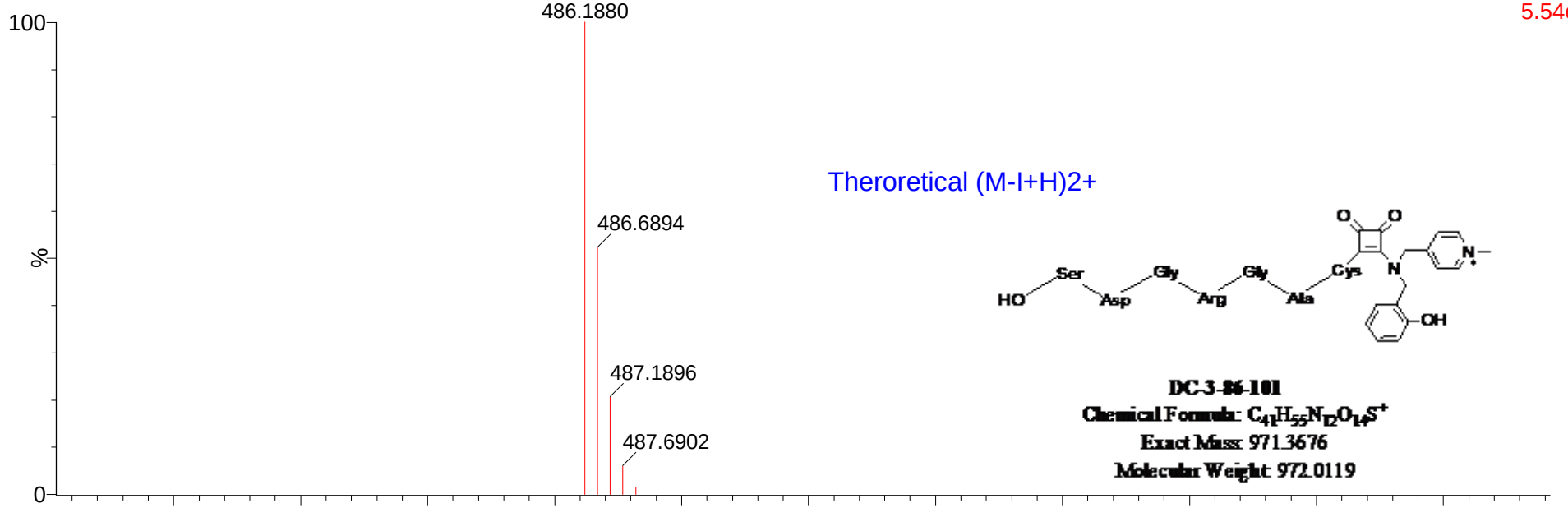
F2 - Acquisition Parameters
Date_           20100506
Time                9.39
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG               zg
TD                 32768
SOLVENT              D2O
NS                    32
DS                     0
SWH              4789.272 Hz
FIDRES           0.146157 Hz
AQ               3.4210291 sec
RG                   512
DW                 104.400 usec
DE                  54.00 usec
TE                  683.2 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.75 usec
PL1                 0.00 dB
SFO1         300.1324000 MHz

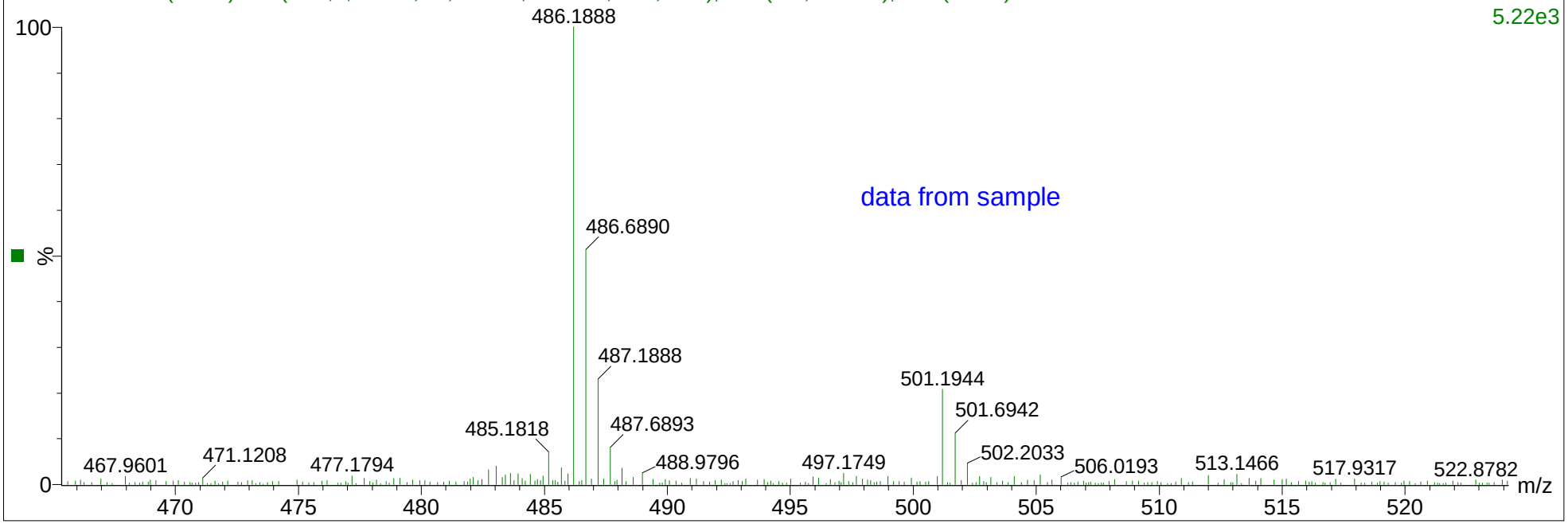
F2 - Processing parameters
SI                 32768
SF           300.1300024 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```

File name:dc-3-86-101 **Notebook Ref: 24947** **IU Database#: 24947**
Instrument: LCT KC366 **Test name: Accurate Mass** **13-May-2010 12:23:17**
Method: ESI-TOF **Base Peak Mass: 486.18878174**

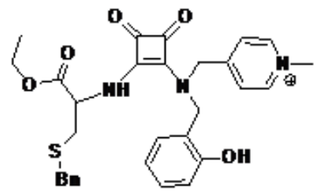
dc-3-86-101 (0.035) Is (0.50,0.50) C41H54N12O14S — 1: TOF MS ES+ 5.54e12



dc-3-86-101 48 (1.202) AM (Cen,8, 90.00, Ar,5000.0,1971.61,0.70,LS 6); Sm (SG, 2x4.00); Cm (48:54) — 1: TOF MS ES+ 5.22e3



DC-2-92-201-5min



DC-2-92-201

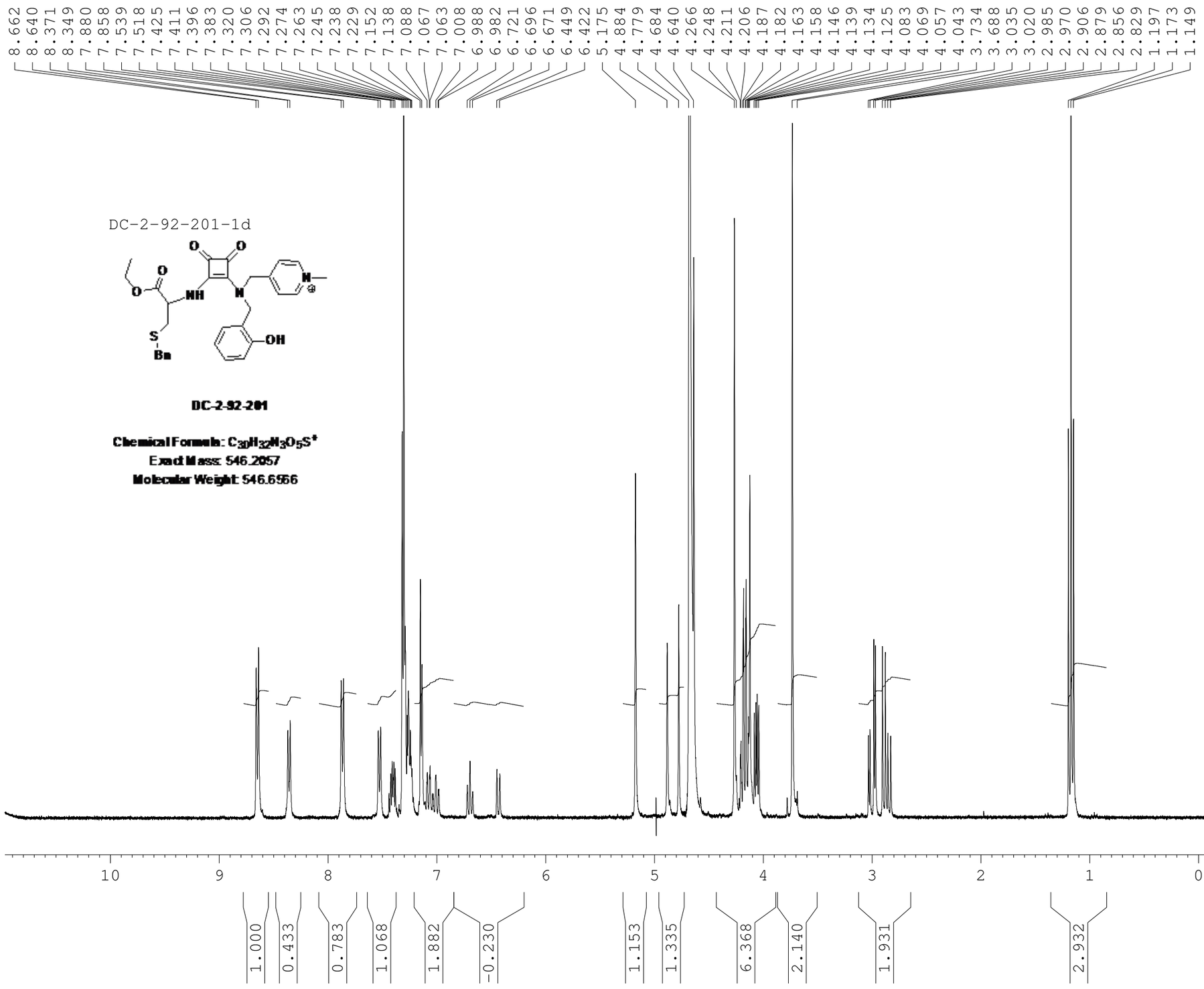
Chemical Formula: $C_{30}H_{32}N_3O_5S^+$
Exact Mass: 546.2057
Molecular Weight: 546.6566


```
Current Data Parameters
NAME         DC-2-92-201-5min
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100226
Time                  14.42
INSTRUM               spect
PROBHD    5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT                D2O
NS                      32
DS                       0
SWH               3591.954 Hz
FIDRES           0.109618 Hz
AQ               4.5613556 sec
RG                     256
DW                 139.200 usec
DE                  54.00 usec
TE                  299.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.75 usec
PL1                  0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                   32768
SF            300.1300042 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

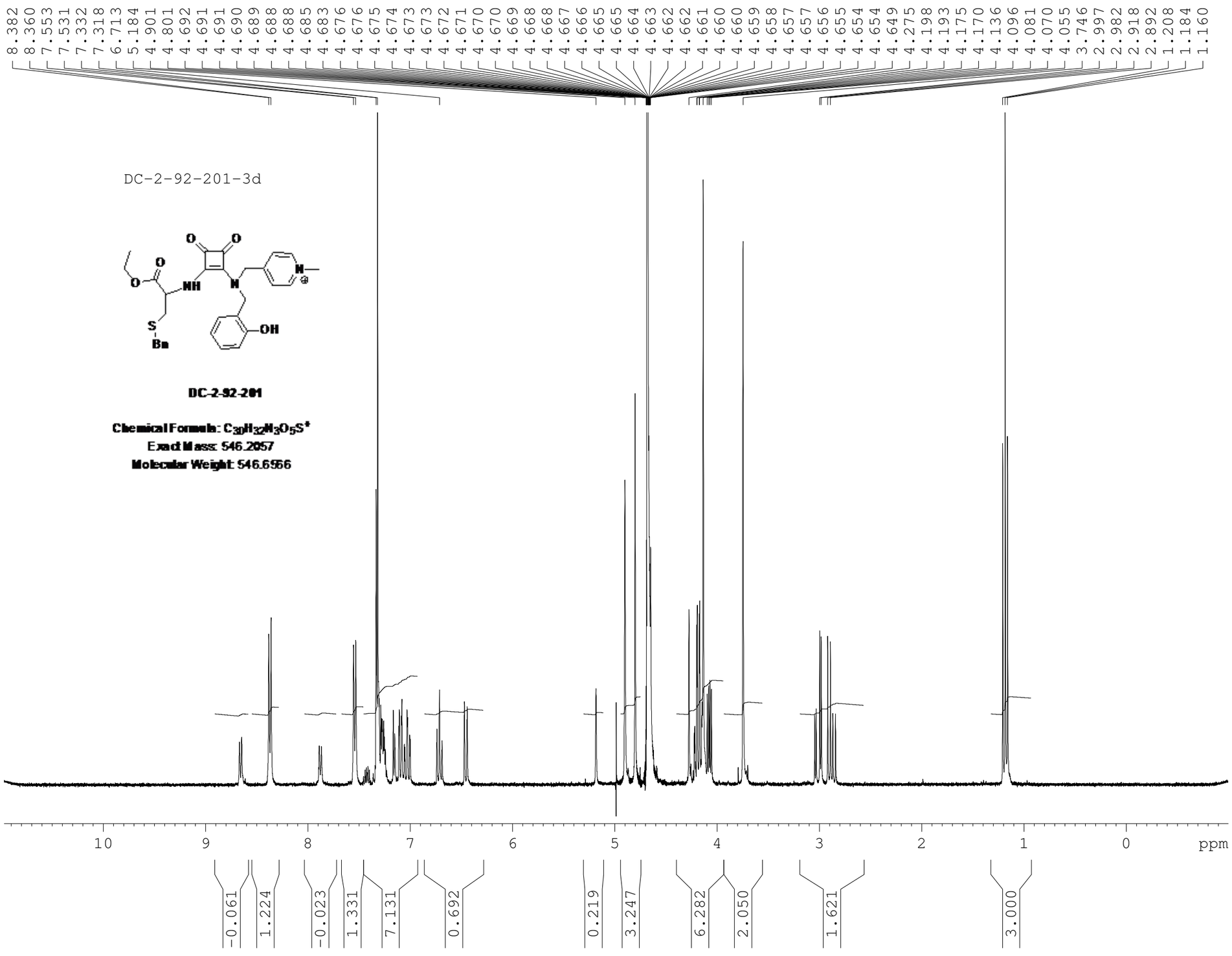



```
Current Data Parameters
NAME         DC-2-92-201-1d
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20100227
Time                 11.31
INSTRUM              spect
PROBHD    5 mm QNP  1H/1
PULPROG                zg
TD                  32768
SOLVENT               D2O
NS                     32
DS                      0
SWH               3591.954 Hz
FIDRES            0.109618 Hz
AQ               4.5613556 sec
RG                    512
DW                139.200 usec
DE                 54.00 usec
TE                 298.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.75 usec
PL1                 0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300042 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                   1.00
```

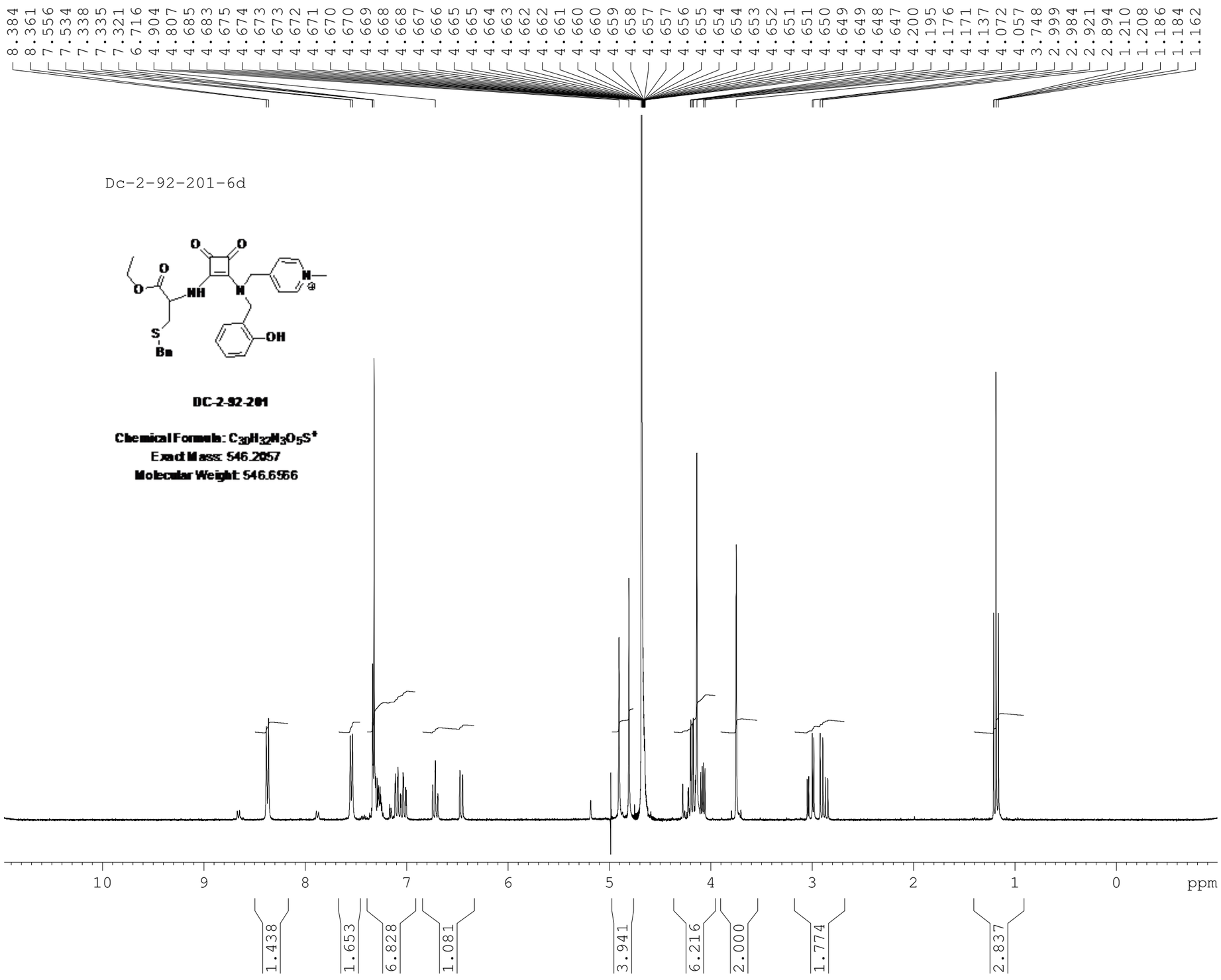



```
Current Data Parameters
NAME         DC-2-92-201-3d
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20100301
Time                  18.23
INSTRUM               spect
PROBHD    5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT                D2O
NS                        16
DS                         0
SWH                3591.954 Hz
FIDRES            0.109618 Hz
AQ                4.5613556 sec
RG                    456.1
DW                  139.200 usec
DE                   54.00 usec
TE                   299.2 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.75 usec
PL1                    0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                    32768
SF            300.1300042 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00
```



Current Data Parameters

NAME	Dc-2-92-201-6d
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20100303
Time	18.12
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg
TD	32768
SOLVENT	D2O
NS	16
DS	0
SWH	3591.954 Hz
FIDRES	0.109618 Hz
AQ	4.5613556 sec
RG	456.1
DW	139.200 usec
DE	54.00 usec
TE	299.2 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	9.75 usec
PL1	0.00 dB
SFO1	300.1315007 MHz

F2 - Processing parameters

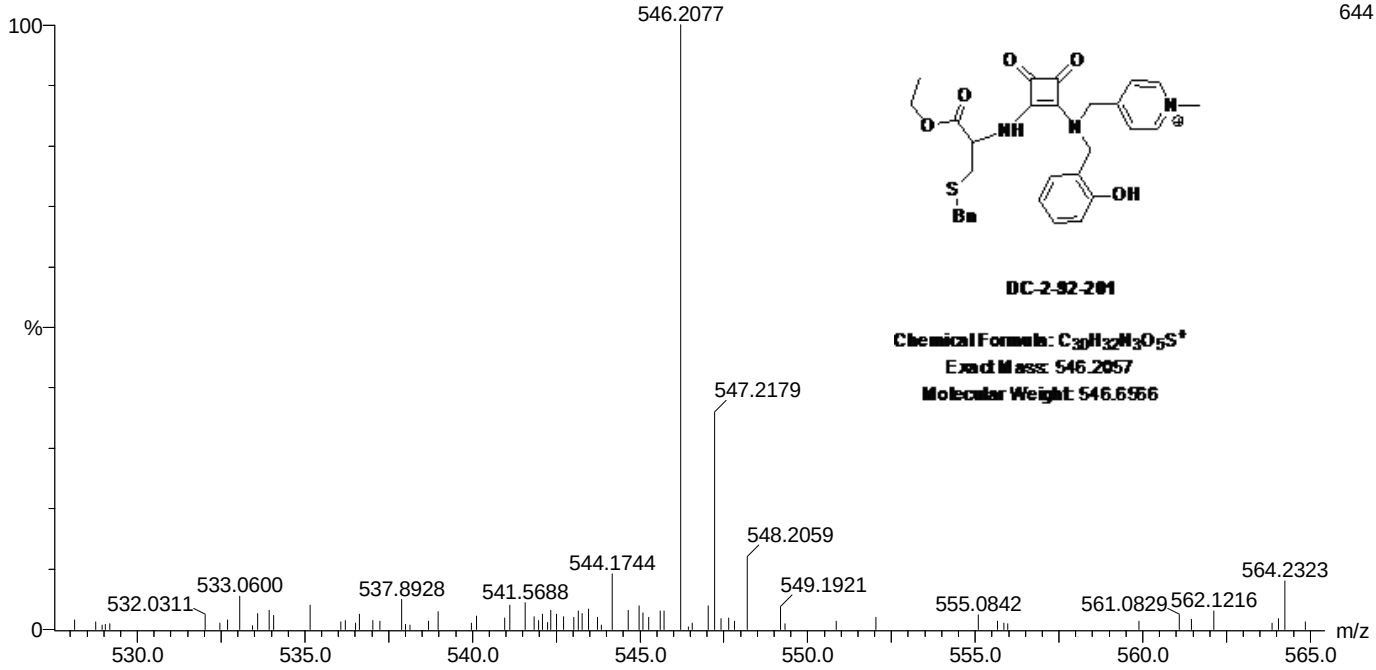
SI	32768
SF	300.1300042 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

Elemental Composition Report

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
74 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-30 H: 0-32 N: 0-3 O: 0-5 Na: 0-1 S: 0-1

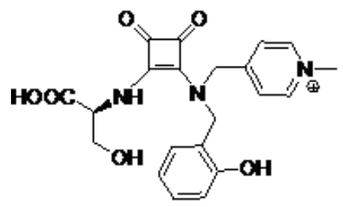
File name:dc-2-92-201b Notebook Ref: 25563 IU Database#: 25563
Instrument: LCT KC366 Test name: Accurate Mass 15-Jun-2010 17:23:10
Method: ESI-TOF Base Peak Mass: 546.20770264
dc-2-92-201b-rerun5 67 (1.686) AM (Cen,8, 90.00, Ar,5000.0,922.36,0.70,LS 7); Sm (SG, 2x4.00); Cm (67:68) 1: TOF MS ES+
644



Minimum:	70.00				-1.5		
Maximum:	100.00		5.0	50.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
546.2077	100.00	546.2063	1.4	2.6	16.5	0.2	C30 H32 N3 O5 S

8.679 8.657 7.900 7.878 7.438 7.426 7.409 7.396 7.308 7.281 7.167 7.152 5.197 4.718 4.716 4.714 4.712 4.712 4.711 4.710 4.710 4.709 4.708 4.707 4.707 4.706 4.705 4.704 4.704 4.703 4.702 4.702 4.701 4.700 4.699 4.699 4.698 4.697 4.696 4.696 4.695 4.694 4.693 4.683 4.668 4.668 4.664 4.283 3.917 3.903 3.876 3.864 3.847 3.824 3.806 3.756 3.743 3.739 3.725

DC-2-69-100-5min



DC-2-69-1

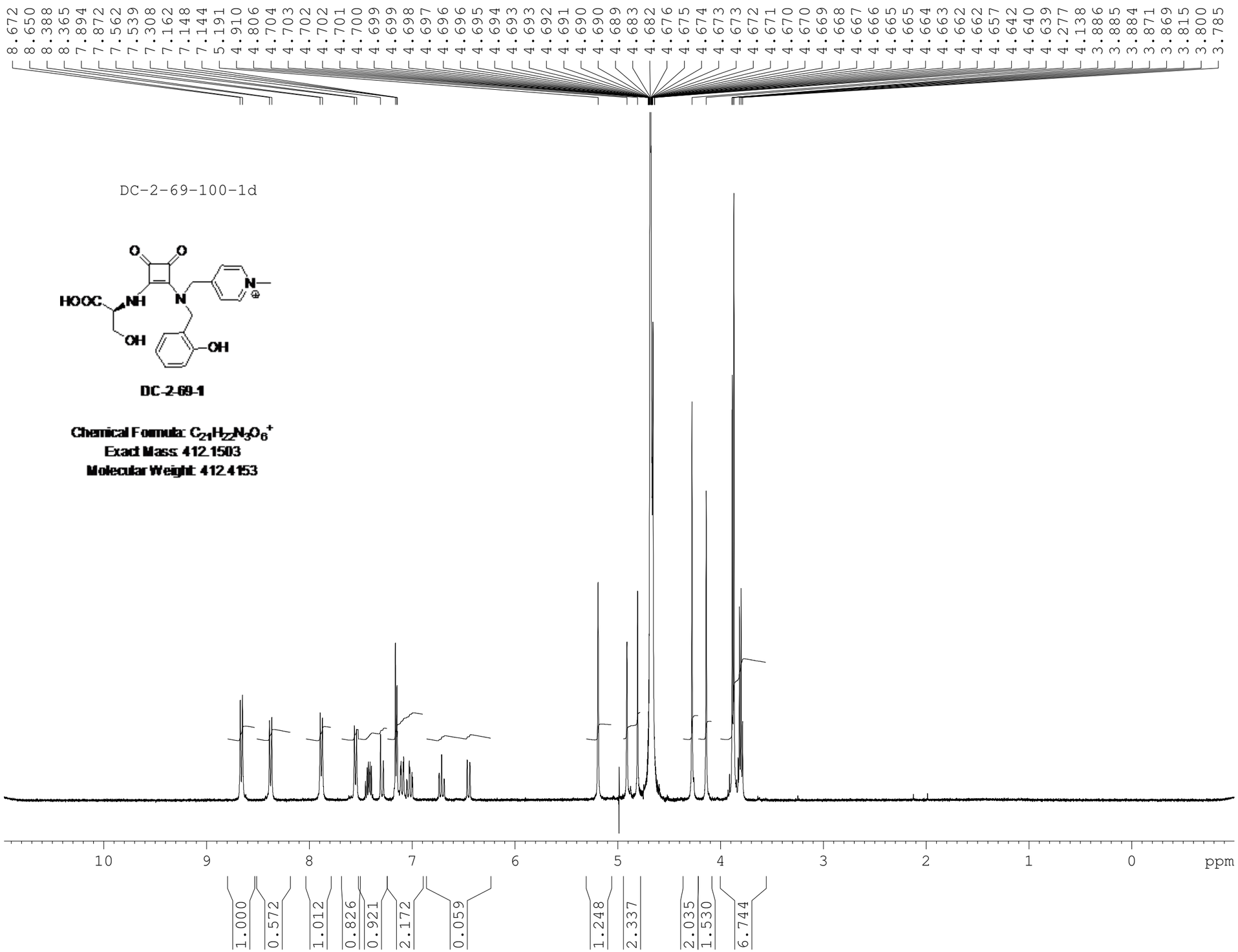
Chemical Formula: $C_{21}H_{22}N_3O_6^+$
Exact Mass: 412.1503
Molecular Weight: 412.4153


```
Current Data Parameters
NAME         DC-2-69-100-5min
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_             20100219
Time                 10.08
INSTRUM              spect
PROBHD    5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT                D2O
NS                      32
DS                       0
SWH               3591.954 Hz
FIDRES            0.109618 Hz
AQ               4.5613556 sec
RG                     256
DW                 139.200 usec
DE                  54.00 usec
TE                  298.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.75 usec
PL1                  0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                   32768
SF            300.1300042 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```



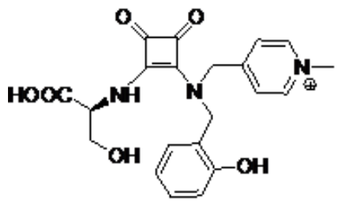

```
Current Data Parameters
NAME         DC-2-69-100-1d
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20100220
Time                 10.42
INSTRUM              spect
PROBHD    5 mm QNP  1H/1
PULPROG                zg
TD                  32768
SOLVENT               D2O
NS                     32
DS                      0
SWH              3591.954 Hz
FIDRES           0.109618 Hz
AQ               4.5613556 sec
RG                   574.7
DW                 139.200 usec
DE                  54.00 usec
TE                  298.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.75 usec
PL1                 0.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI                  32768
SF           300.1300042 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

DC-2-69-100-3d



DC-2-69-1

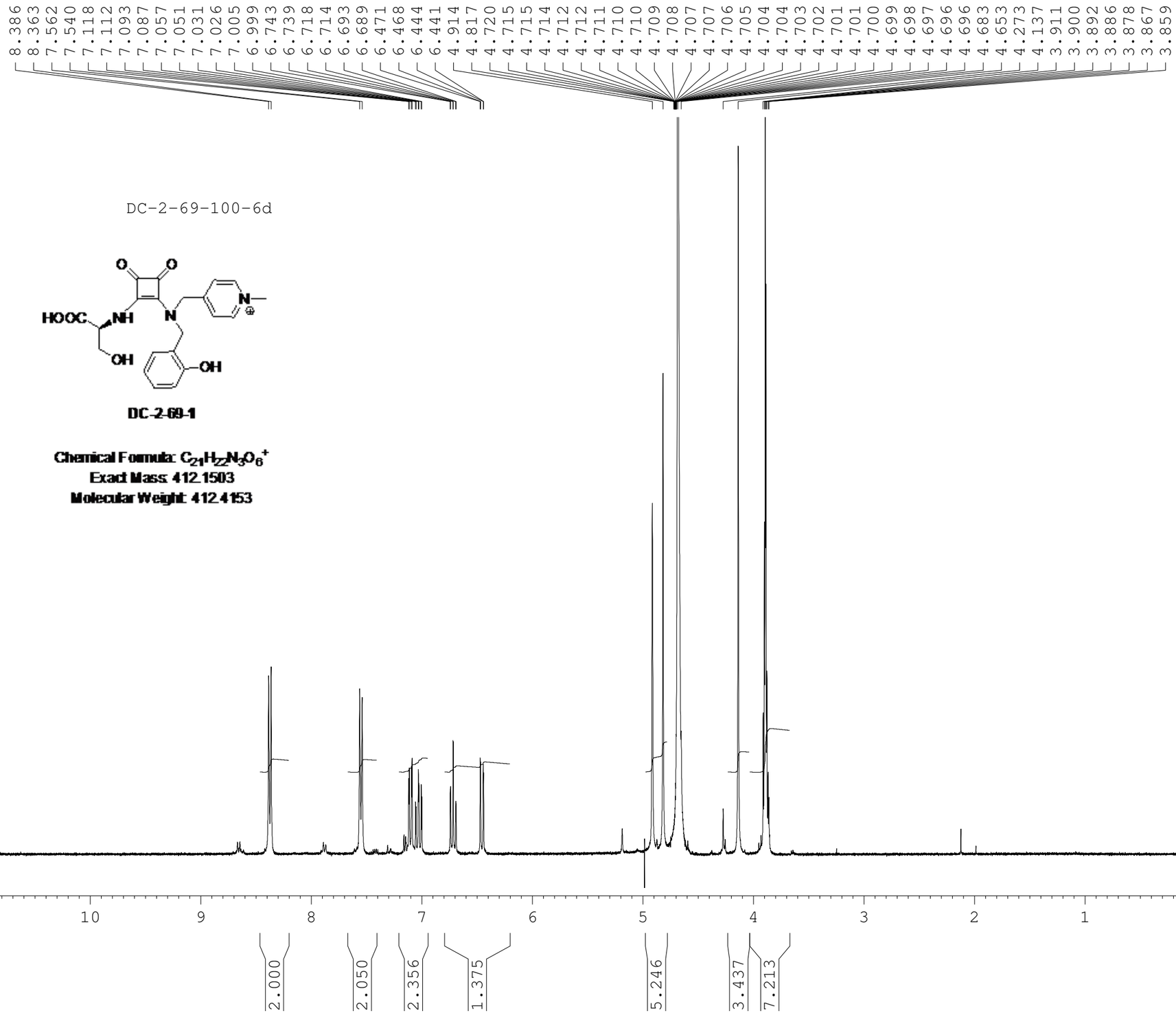
Chemical Formula: $C_{21}H_{22}N_3O_6^+$
Exact Mass: 412.1503
Molecular Weight: 412.4153


```
Current Data Parameters
NAME            DC-2-69-100-3d
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20100222
Time                      9.14
INSTRUM                  spect
PROBHD    5 mm QNP  1H/1
PULPROG                    zg
TD                      32768
SOLVENT                   D2O
NS                         32
DS                          0
SWH                  3591.954 Hz
FIDRES               0.109618 Hz
AQ                 4.5613556 sec
RG                        362
DW                    139.200 usec
DE                     54.00 usec
TE                     298.2 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       9.75 usec
PL1                      0.00 dB
SFO1             300.1315007 MHz

F2 - Processing parameters
SI                       32768
SF               300.1300042 MHz
WDW                         no
SSB                          0
LB                       0.00 Hz
GB                           0
PC                        1.00
```




```
Current Data Parameters
NAME          DC-2-69-100-6d
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100225
Time                  10.34
INSTRUM               spect
PROBHD    5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT                D2O
NS                      32
DS                       0
SWH               3591.954 Hz
FIDRES           0.109618 Hz
AQ               4.5613556 sec
RG                   574.7
DW                 139.200 usec
DE                  54.00 usec
TE                  298.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.75 usec
PL1                  0.00 dB
SFO1          300.1315007 MHz

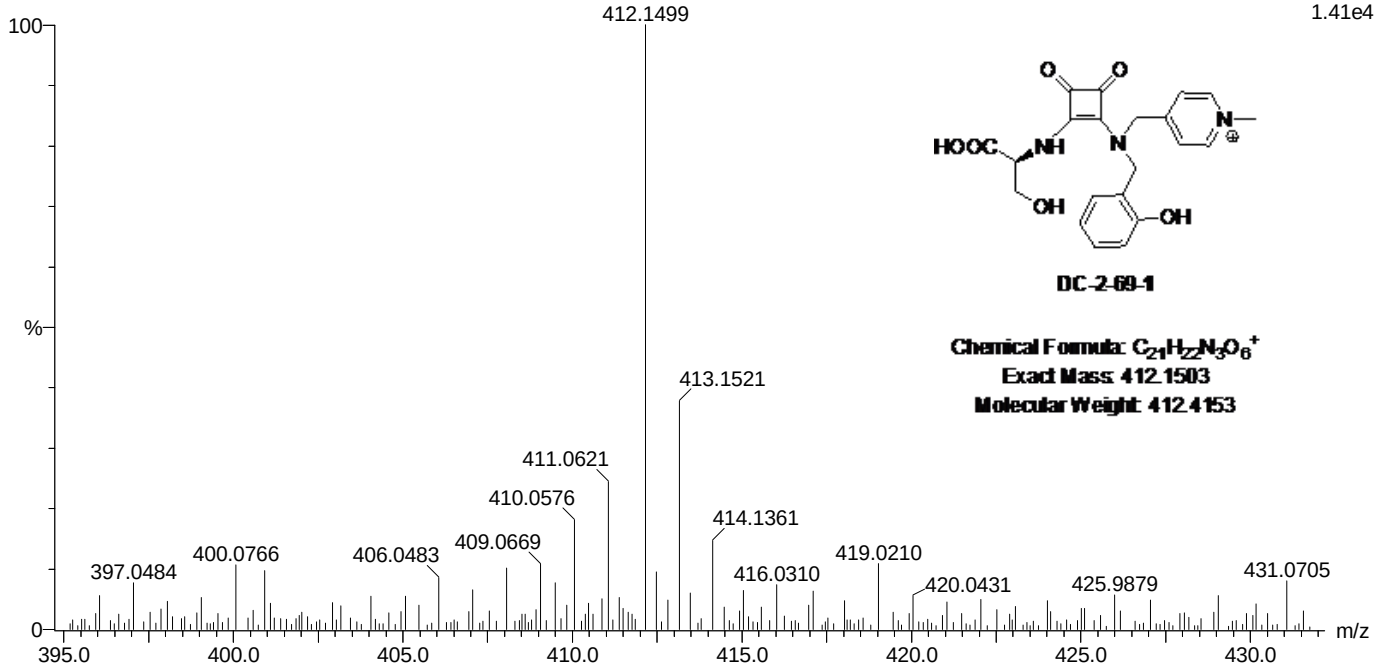
F2 - Processing parameters
SI                   32768
SF            300.1300042 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

Elemental Composition Report

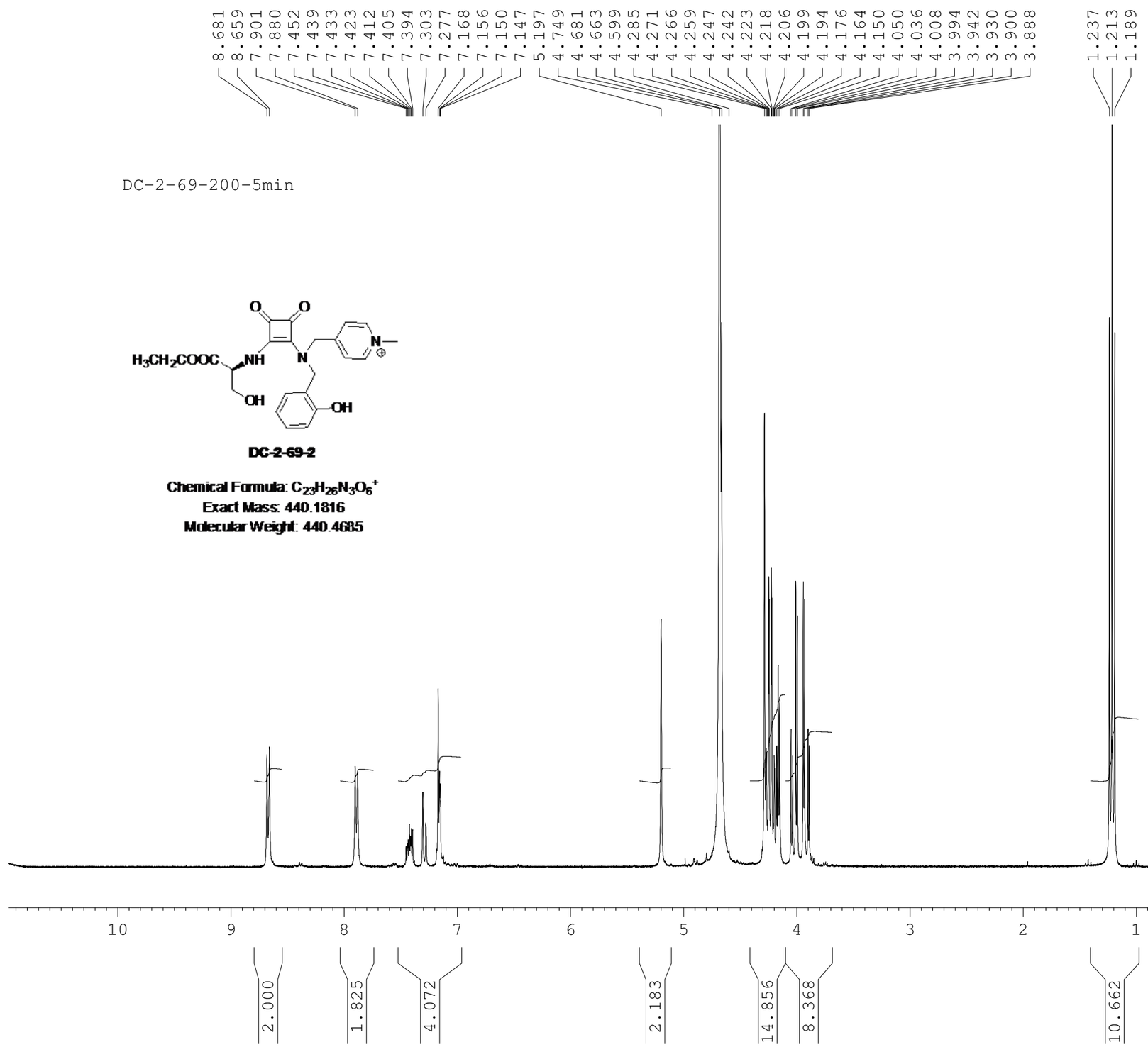
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

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: 0-21 H: 0-22 N: 0-3 O: 0-6

File name:dc-2-69-100 Notebook Ref: 24824 IU Database#: 24824
Instrument: LCT KC366 Test name: Accurate Mass 06-May-2010 11:47:20
Method: ESI-TOF Base Peak Mass: 412.14990234
dc-2-69-100 48 (1.203) AM (Cen,8, 90.00, Ar,4000.0,1222.14,0.70,LS 6); Sm (SG, 2x4.00); Cm (48:74) 1: TOF MS ES+
1.41e4



Minimum:	80.00				-1.5		
Maximum:	100.00		5.0	5.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
412.1499	100.00	412.1509	-1.0	-2.4	12.5	761.1	C21 H22 N3 O6

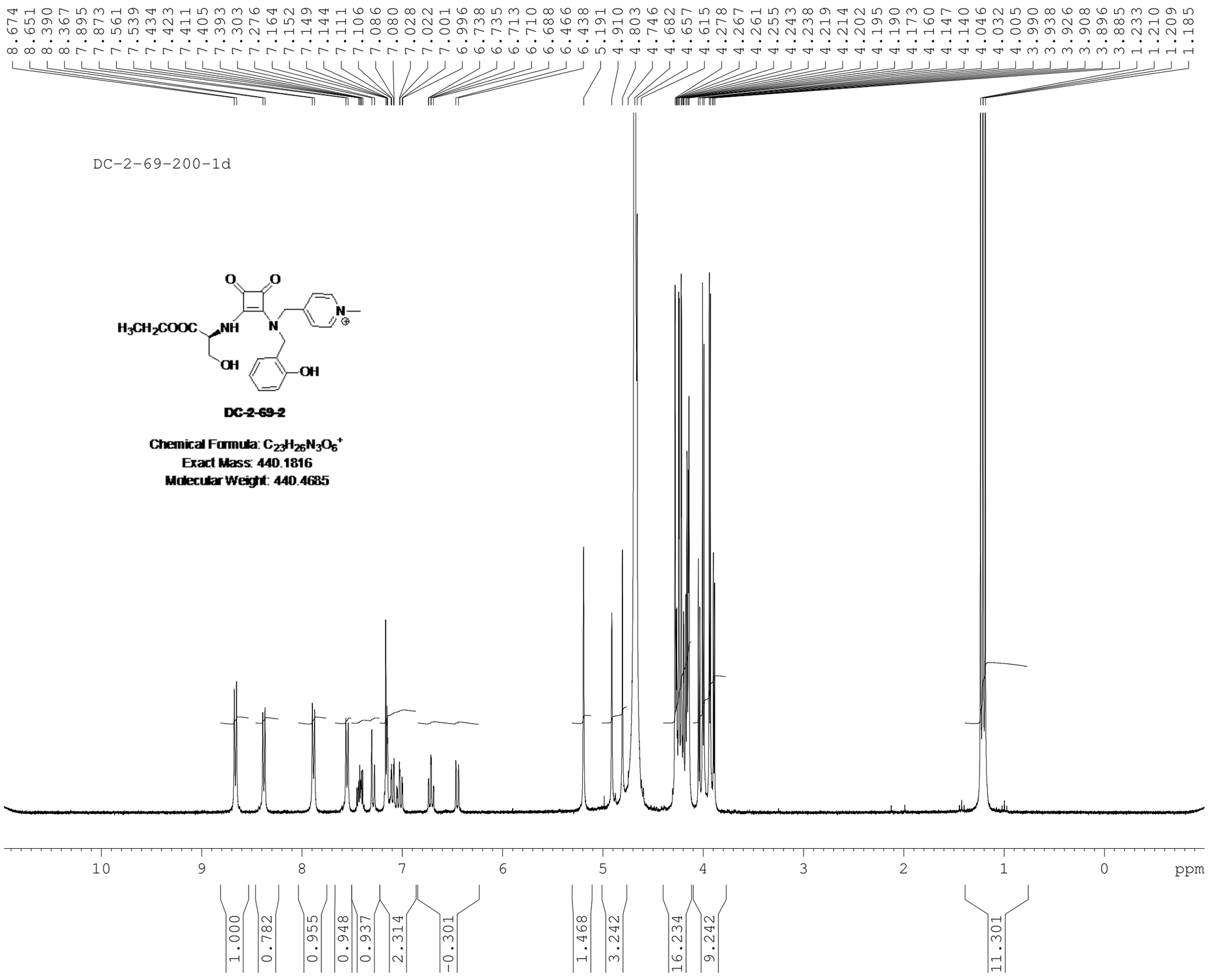



```
Current Data Parameters
NAME         DC-2-69-200-5min
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20100219
Time              10.15
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG             zg
TD                32768
SOLVENT             D2O
NS                   32
DS                    0
SWH            3591.954 Hz
FIDRES        0.109618 Hz
AQ            4.5613556 sec
RG                  256
DW              139.200 usec
DE               54.00 usec
TE               298.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                9.75 usec
PL1               0.00 dB
SFO1        300.1315007 MHz

F2 - Processing parameters
SI                32768
SF          300.1300042 MHz
WDW                  no
SSB                   0
LB                0.00 Hz
GB                    0
PC                 1.00
```

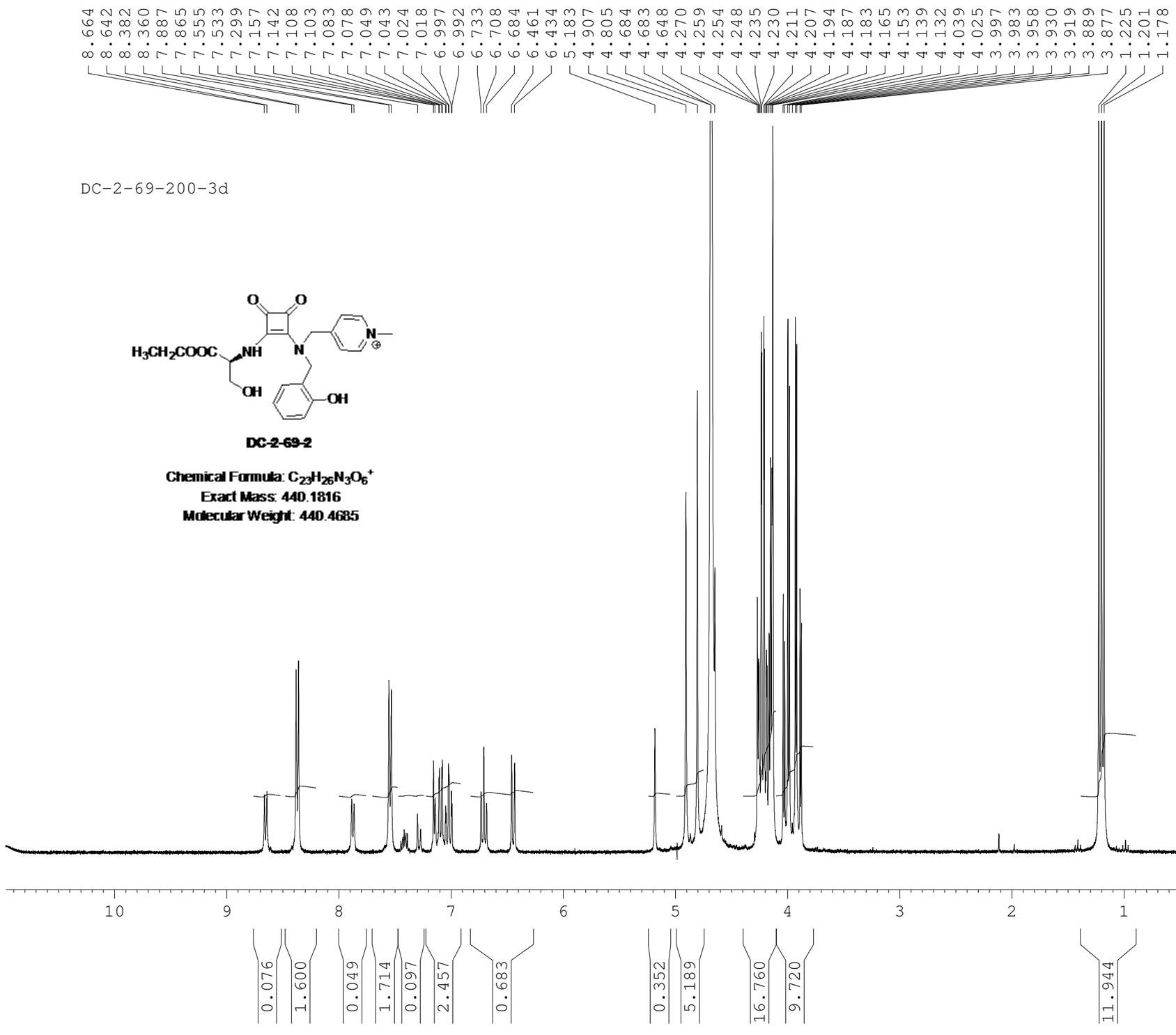



```
Current Data Parameters
NAME         DC-2-69-200-1d
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20100220
Time                  10.50
INSTRUM               spect
PROBHD    5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT                D2O
NS                      32
DS                       0
SWH               3591.954 Hz
FIDRES            0.109618 Hz
AQ               4.5613556 sec
RG                     362
DW                 139.200 usec
DE                  54.00 usec
TE                  298.2 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    9.75 usec
PL1                   0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                    32768
SF            300.1300042 MHz
WDW                      no
SSB                       0
LB                    0.00 Hz
GB                        0
PC                     1.00
```

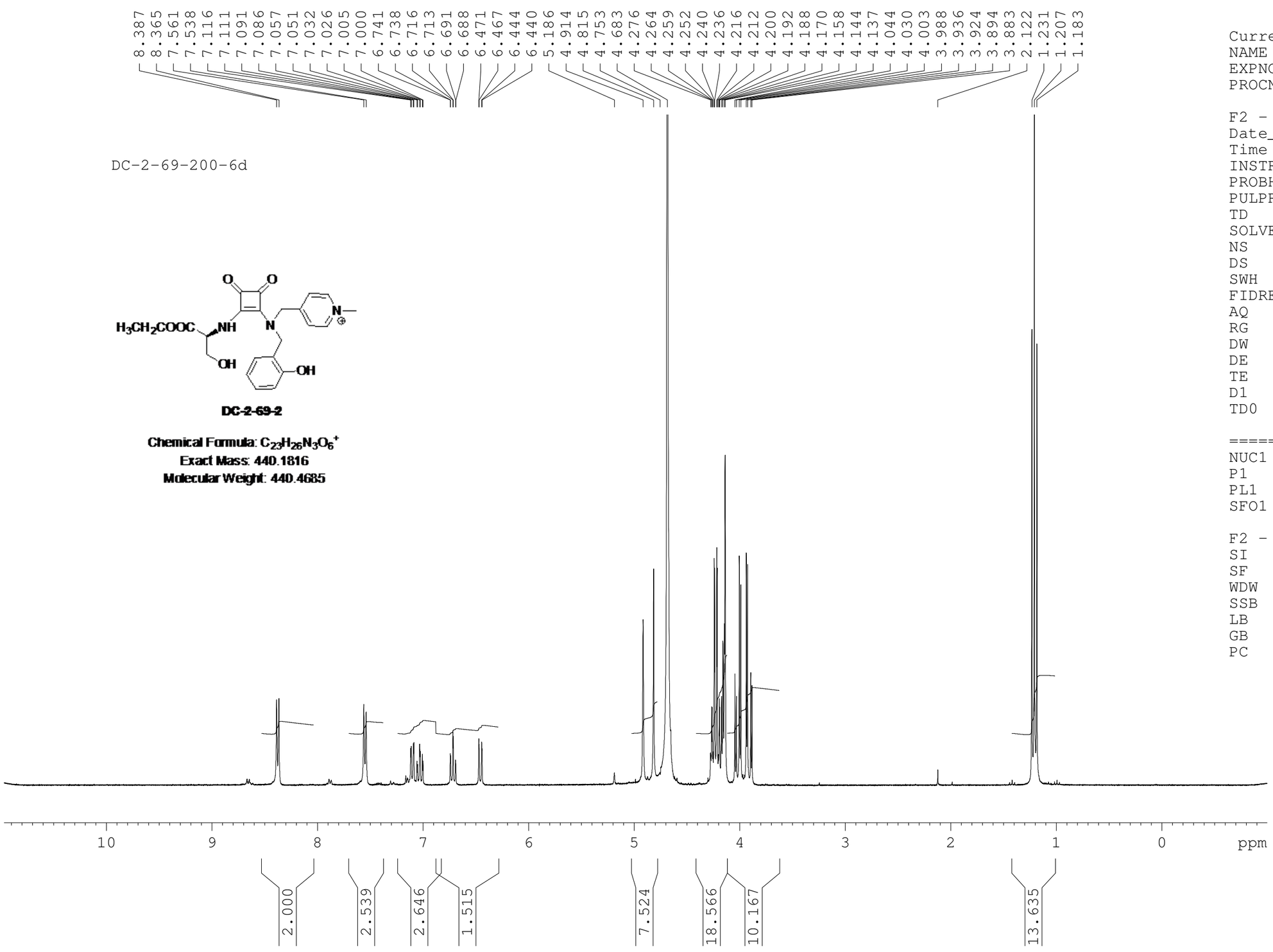



```
Current Data Parameters
NAME         DC-2-69-200-3d
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20100222
Time                   9.21
INSTRUM               spect
PROBHD    5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT                D2O
NS                      32
DS                       0
SWH               3591.954 Hz
FIDRES           0.109618 Hz
AQ               4.5613556 sec
RG                   322.5
DW                 139.200 usec
DE                  54.00 usec
TE                  298.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.75 usec
PL1                  0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                   32768
SF            300.1300042 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```




```
Current Data Parameters
NAME         DC-2-69-200-6d
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20100225
Time                 10.42
INSTRUM              spect
PROBHD    5 mm QNP  1H/1
PULPROG                zg
TD                  32768
SOLVENT               D2O
NS                     32
DS                      0
SWH              3591.954 Hz
FIDRES           0.109618 Hz
AQ               4.5613556 sec
RG                   406.4
DW                 139.200 usec
DE                  54.00 usec
TE                  298.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.75 usec
PL1                 0.00 dB
SFO1         300.1315007 MHz

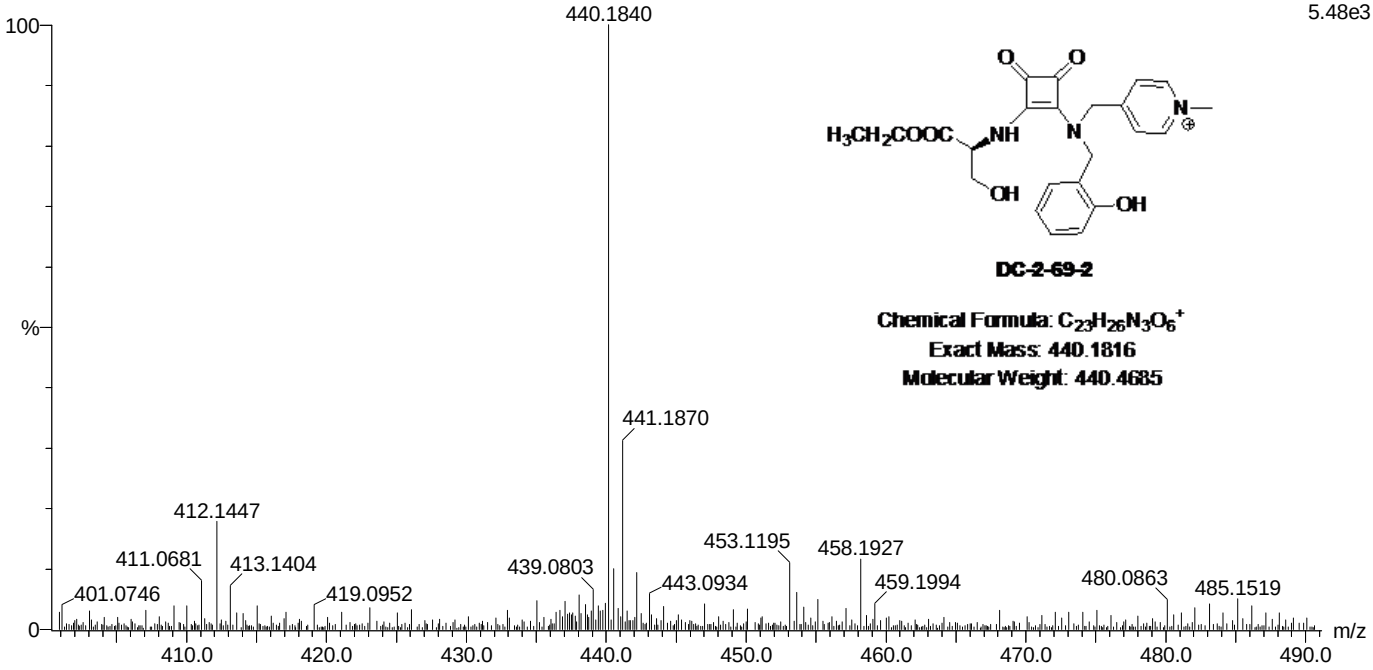
F2 - Processing parameters
SI                  32768
SF           300.1300042 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

Elemental Composition Report

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

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: 0-23 H: 0-26 N: 0-3 O: 0-6

File name:dc-2-69-200 Notebook Ref: 24825 IU Database#: 24825
Instrument: LCT KC366 Test name: Accurate Mass 06-May-2010 11:51:31
Method: ESI-TOF Base Peak Mass: 440.18399048
dc-2-69-200 44 (1.102) AM (Cen,8, 90.00, Ar,4000.0,1222.14,0.70,LS 6); Sm (SG, 2x4.00); Cm (44:49) 1: TOF MS ES+
5.48e3

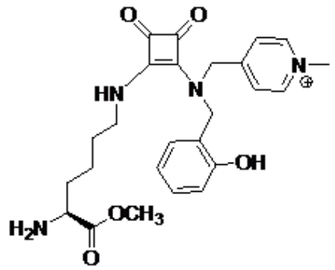


DC-2-69-2

Chemical Formula: $C_{23}H_{26}N_3O_6^+$
Exact Mass: 440.1816
Molecular Weight: 440.4685

Minimum:	80.00				-1.5		
Maximum:	100.00		5.0	5.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
440.1840	100.00	440.1822	1.8	4.1	12.5	66.5	C23 H26 N3 O6

DC-2-94-201-5min



DC-2-94-201

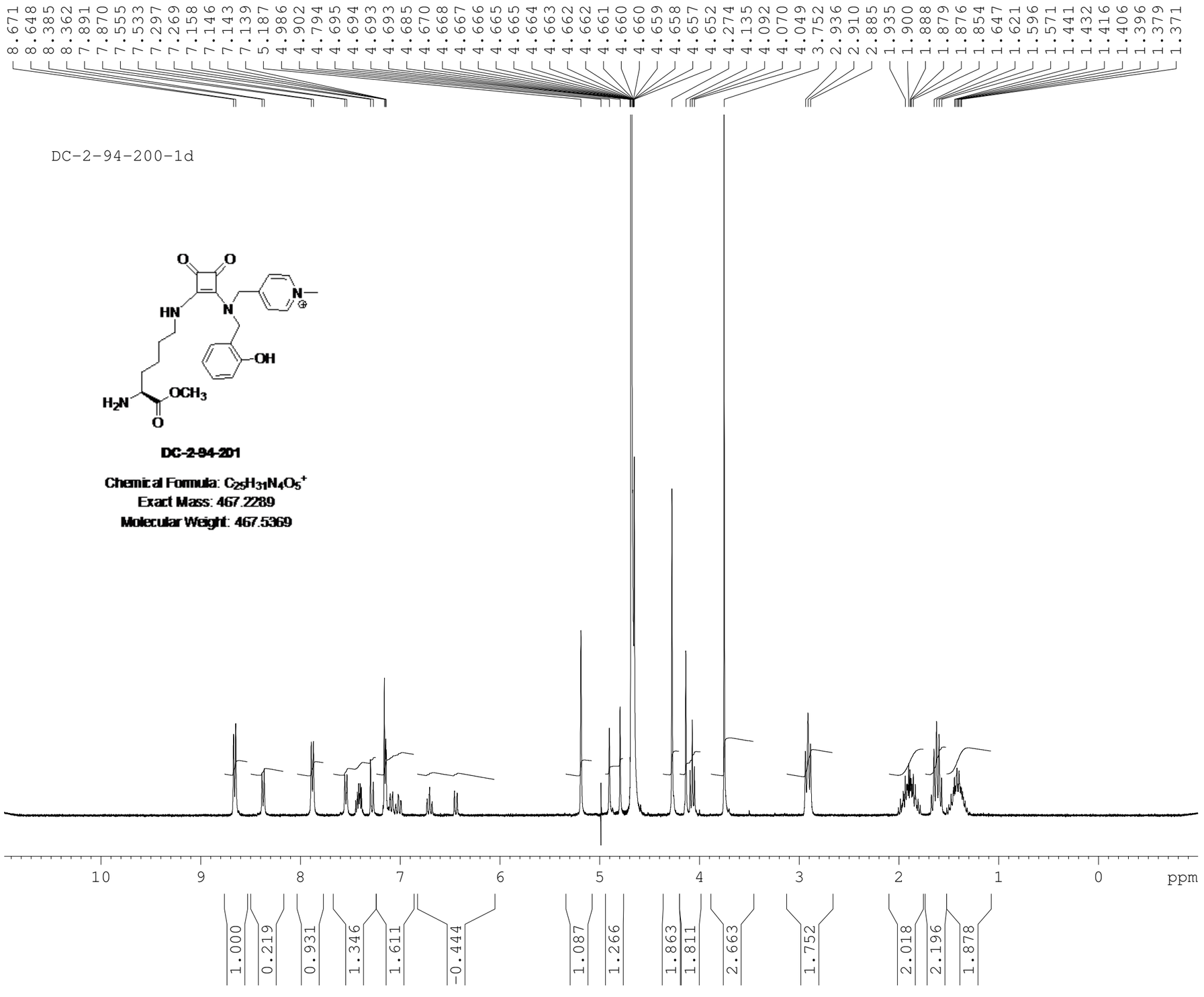
Chemical Formula: $C_{25}H_{31}N_4O_5^+$
Exact Mass: 467.2289
Molecular Weight: 467.5369


```
Current Data Parameters
NAME          DC-2-94-201-5min
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20100226
Time                 16.13
INSTRUM              spect
PROBHD    5 mm QNP  1H/1
PULPROG                zg
TD                  32768
SOLVENT               D2O
NS                     32
DS                      0
SWH              3591.954 Hz
FIDRES           0.109618 Hz
AQ               4.5613556 sec
RG                    256
DW                139.200 usec
DE                 54.00 usec
TE                 299.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.75 usec
PL1                 0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                  32768
SF            300.1300042 MHz
WDW                    no
SSB                     0
LB                  0.00 Hz
GB                      0
PC                   1.00
```

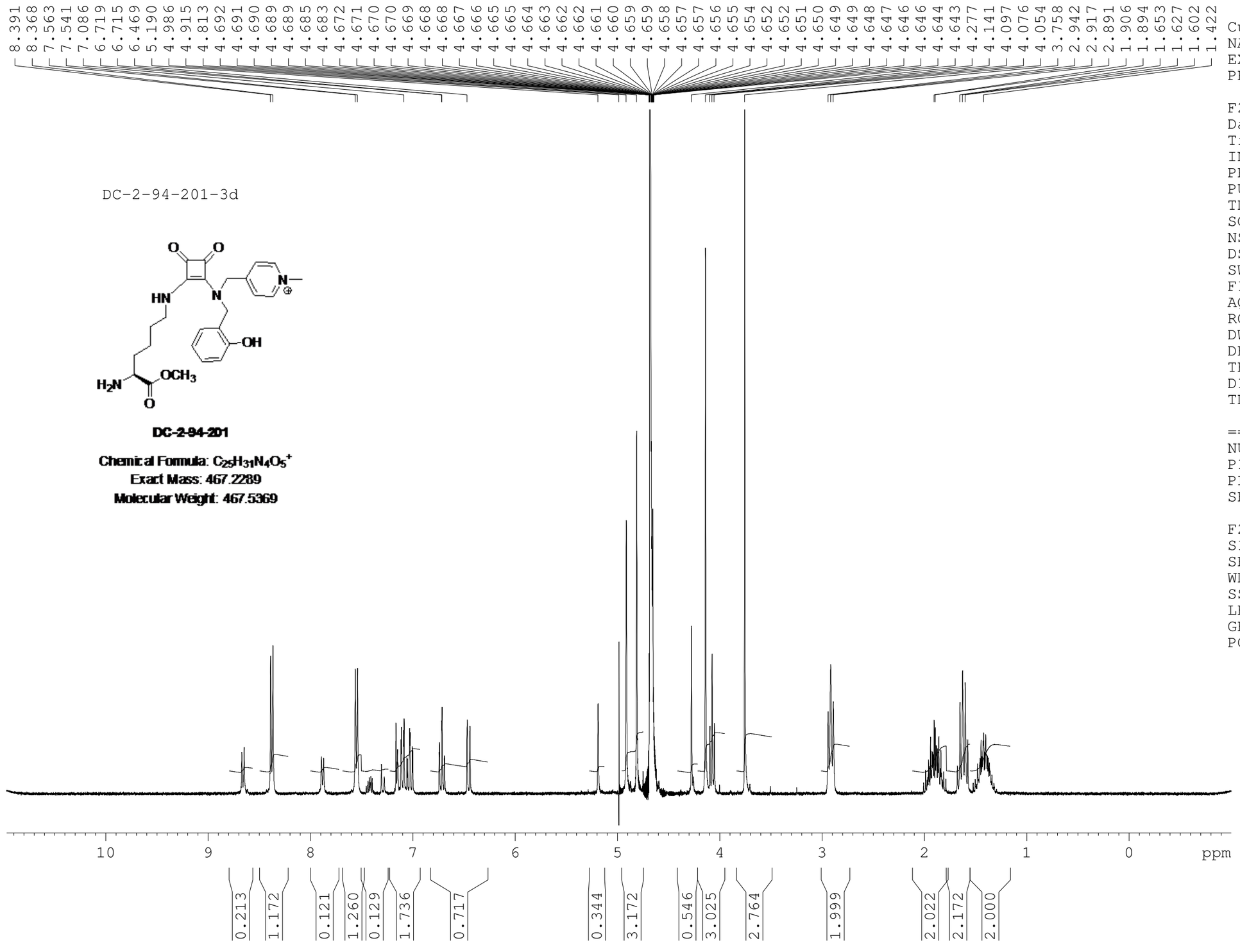



```
Current Data Parameters
NAME        DC-2-94-201-1d
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20100227
Time                 11.41
INSTRUM              spect
PROBHD    5 mm QNP  1H/1
PULPROG                zg
TD                  32768
SOLVENT               D2O
NS                     16
DS                      0
SWH              3591.954 Hz
FIDRES           0.109618 Hz
AQ               4.5613556 sec
RG                    512
DW                 139.200 usec
DE                  54.00 usec
TE                  298.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.75 usec
PL1                  0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                   32768
SF            300.1300042 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```



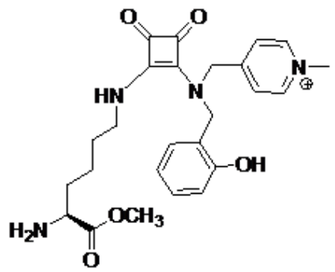
Current Data Parameters
NAME DC-2-94-201-3d
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100301
Time 18.32
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT D2O
NS 16
DS 0
SWH 3591.954 Hz
FIDRES 0.109618 Hz
AQ 4.5613556 sec
RG 456.1
DW 139.200 usec
DE 54.00 usec
TE 299.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1315007 MHz

F2 - Processing parameters
SI 32768
SF 300.1300042 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

Dc-2-94-201-6d



DC-2-94-201

Chemical Formula: $C_{25}H_{31}N_4O_5^+$
Exact Mass: 467.2289
Molecular Weight: 467.5369


```
Current Data Parameters
NAME           Dc-2-94-201-6d
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20100303
Time                   18.31
INSTRUM                spect
PROBHD    5 mm QNP  1H/1
PULPROG                  zg
TD                    32768
SOLVENT                 D2O
NS                       16
DS                        0
SWH                3591.954 Hz
FIDRES            0.109618 Hz
AQ                4.5613556 sec
RG                     406.4
DW                   139.200 usec
DE                    54.00 usec
TE                    299.2 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.75 usec
PL1                    0.00 dB
SFO1          300.1315007 MHz

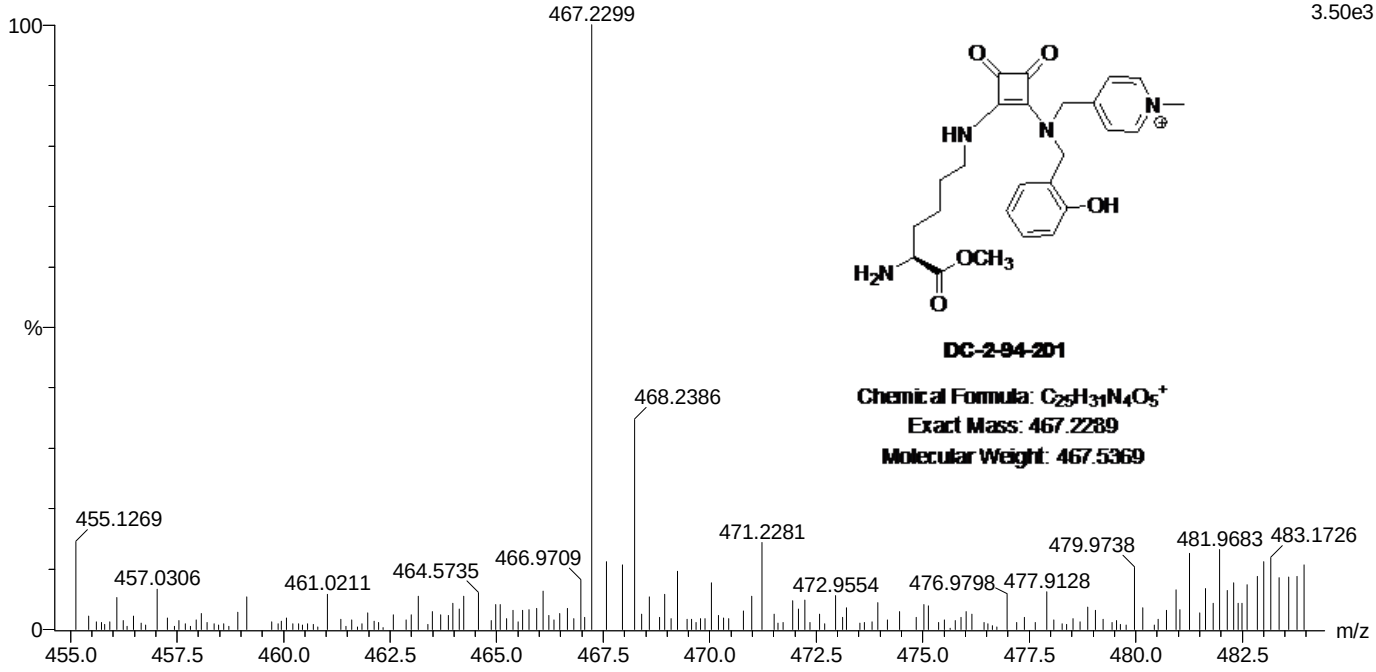
F2 - Processing parameters
SI                    32768
SF            300.1300042 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00
```

Elemental Composition Report

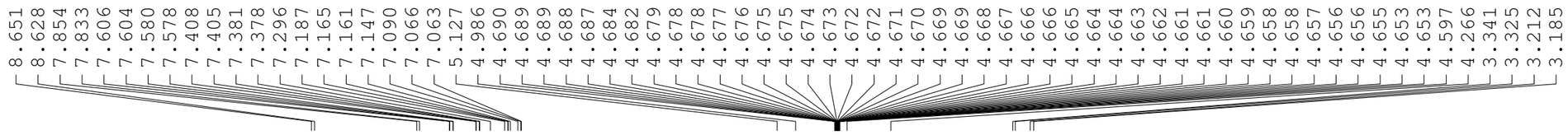
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

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: 0-25 H: 0-31 N: 0-4 O: 0-5

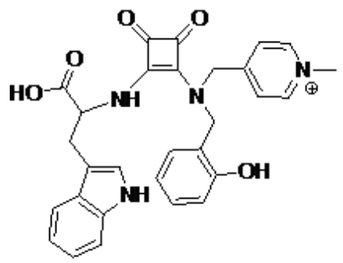
File name:dc-2-94-201 Notebook Ref: 24820 IU Database#: 24820
Instrument: LCT KC366 Test name: Accurate Mass 06-May-2010 11:30:37
Method: ESI-TOF Base Peak Mass: 467.22994995
dc-2-94-201 30 (0.752) AM (Cen,8, 90.00, Ar,4000.0,1222.14,0.70,LS 6); Sm (SG, 2x4.00); Cm (29:36) 1: TOF MS ES+
3.50e3



Minimum:	80.00				-1.5		
Maximum:	100.00		5.0	5.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
467.2299	100.00	467.2294	0.5	1.1	12.5	38.6	C25 H31 N4 O5

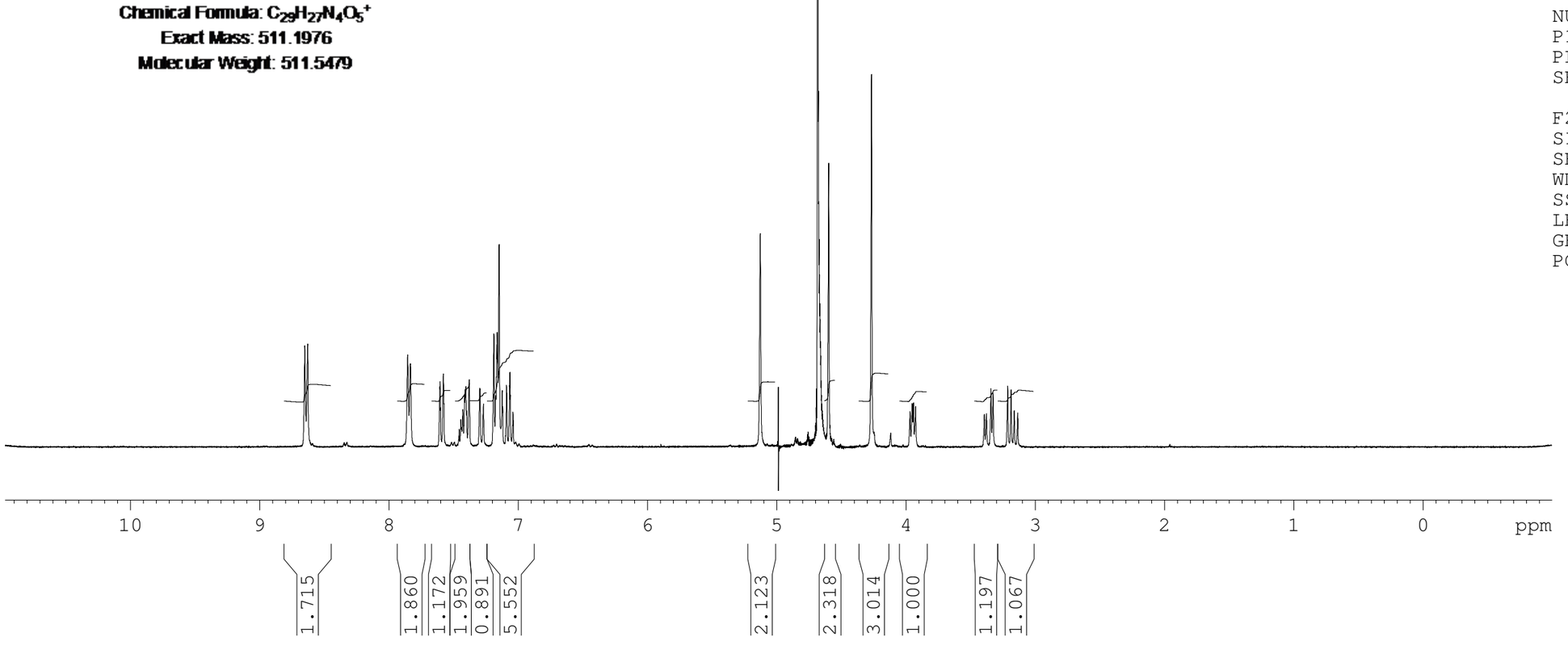


DC-2-91-101-5min



DC-2-91-1

Chemical Formula: $C_{29}H_{27}N_4O_5^+$
Exact Mass: 511.1976
Molecular Weight: 511.5479

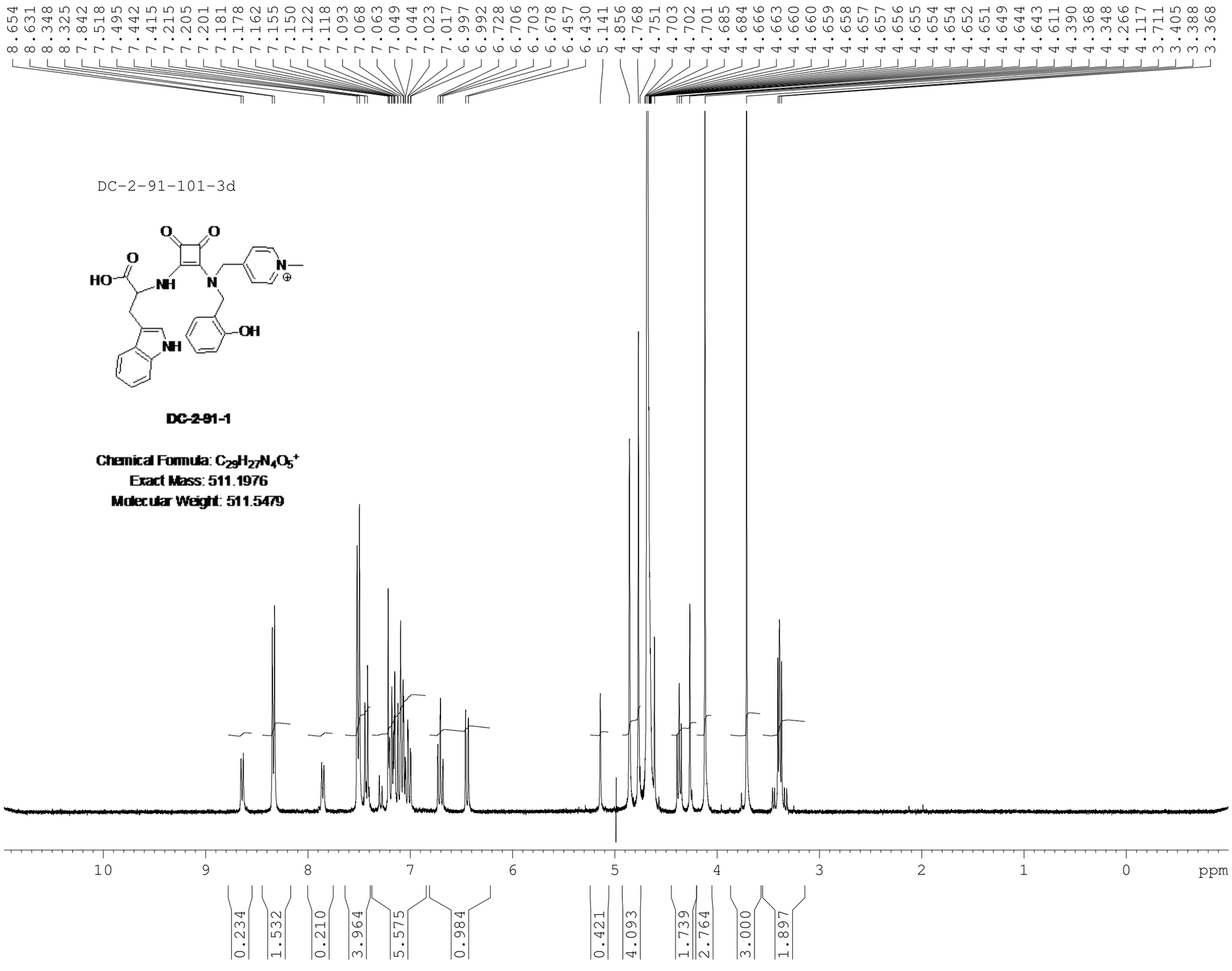



```
Current Data Parameters
NAME         DC-2-91-101-5min
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20100226
Time                   9.14
INSTRUM               spect
PROBHD    5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT                D2O
NS                      32
DS                       0
SWH               3591.954 Hz
FIDRES           0.109618 Hz
AQ               4.5613556 sec
RG                   406.4
DW                 139.200 usec
DE                  54.00 usec
TE                  298.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.75 usec
PL1                  0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                   32768
SF            300.1300042 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

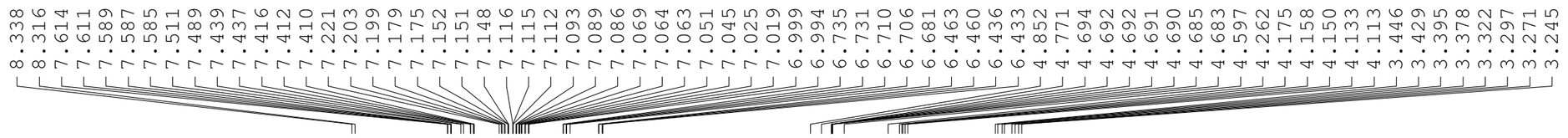



```
Current Data Parameters
NAME         DC-2-91-101-3d
EXPNO                     1
PROCNO                    1

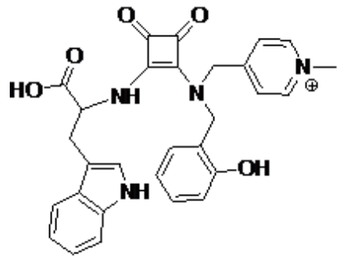
F2 - Acquisition Parameters
Date_              20100301
Time                  18.18
INSTRUM               spect
PROBHD    5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT                D2O
NS                      16
DS                       0
SWH               3591.954 Hz
FIDRES           0.109618 Hz
AQ               4.5613556 sec
RG                     512
DW                 139.200 usec
DE                  54.00 usec
TE                  299.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.75 usec
PL1                  0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                   32768
SF            300.1300042 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

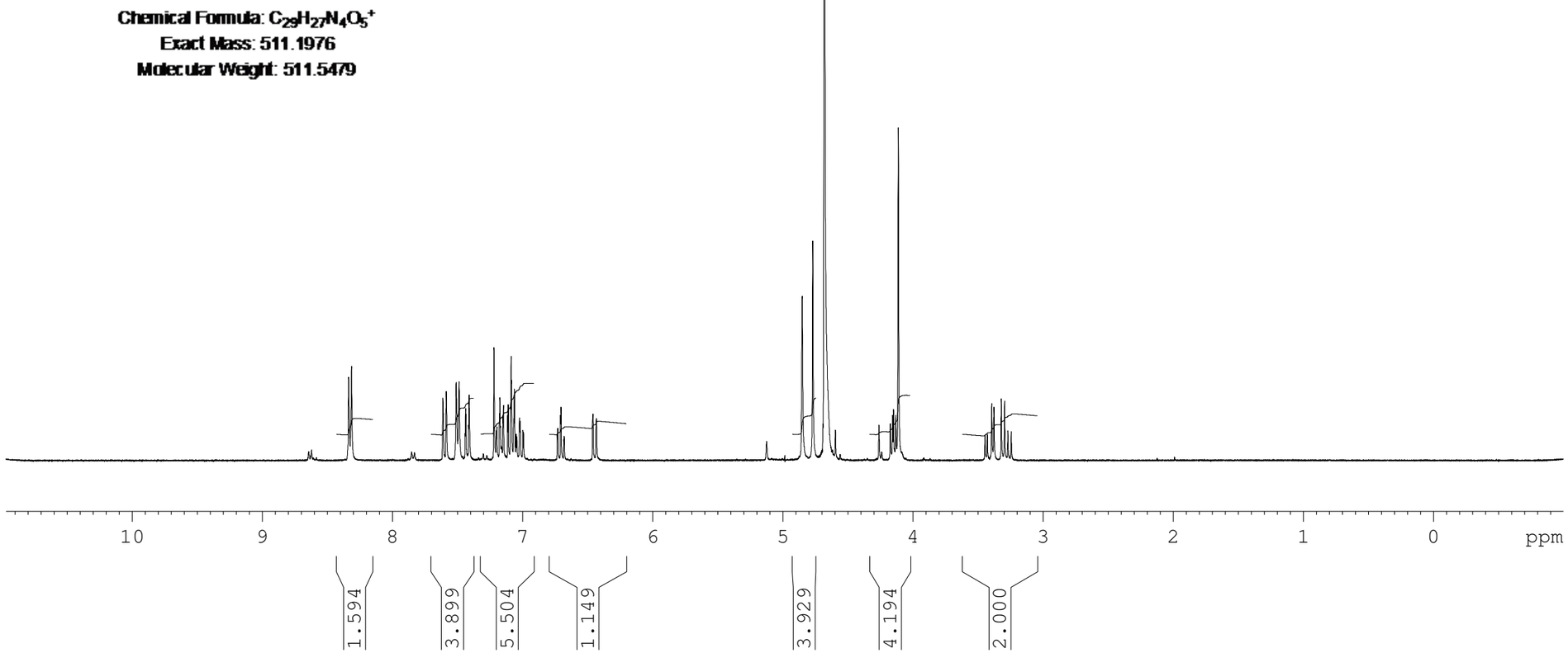


Dc-2-91-101-6d



DC-2-91-1

Chemical Formula: $C_{29}H_{27}N_4O_5^+$
Exact Mass: 511.1976
Molecular Weight: 511.5479




```
Current Data Parameters
NAME            Dc-2-91-101-6d
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_             20100303
Time                 18.18
INSTRUM              spect
PROBHD   5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT                D2O
NS                        16
DS                         0
SWH               3591.954 Hz
FIDRES           0.109618 Hz
AQ               4.5613556 sec
RG                   574.7
DW                 139.200 usec
DE                  54.00 usec
TE                  299.2 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.75 usec
PL1                    0.00 dB
SFO1          300.1315007 MHz

F2 - Processing parameters
SI                     32768
SF            300.1300042 MHz
WDW                       no
SSB                        0
LB                    0.00 Hz
GB                         0
PC                      1.00
```

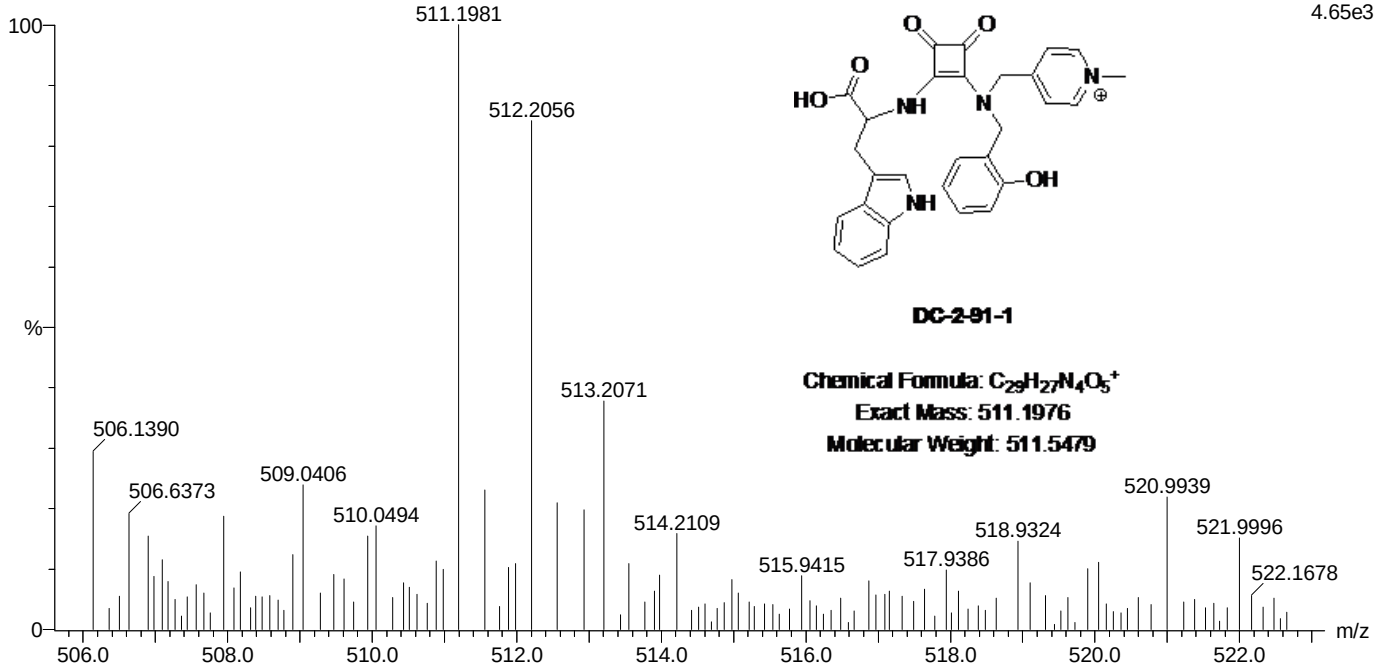
Elemental Composition Report

Multiple Mass Analysis: 2 mass(es) processed

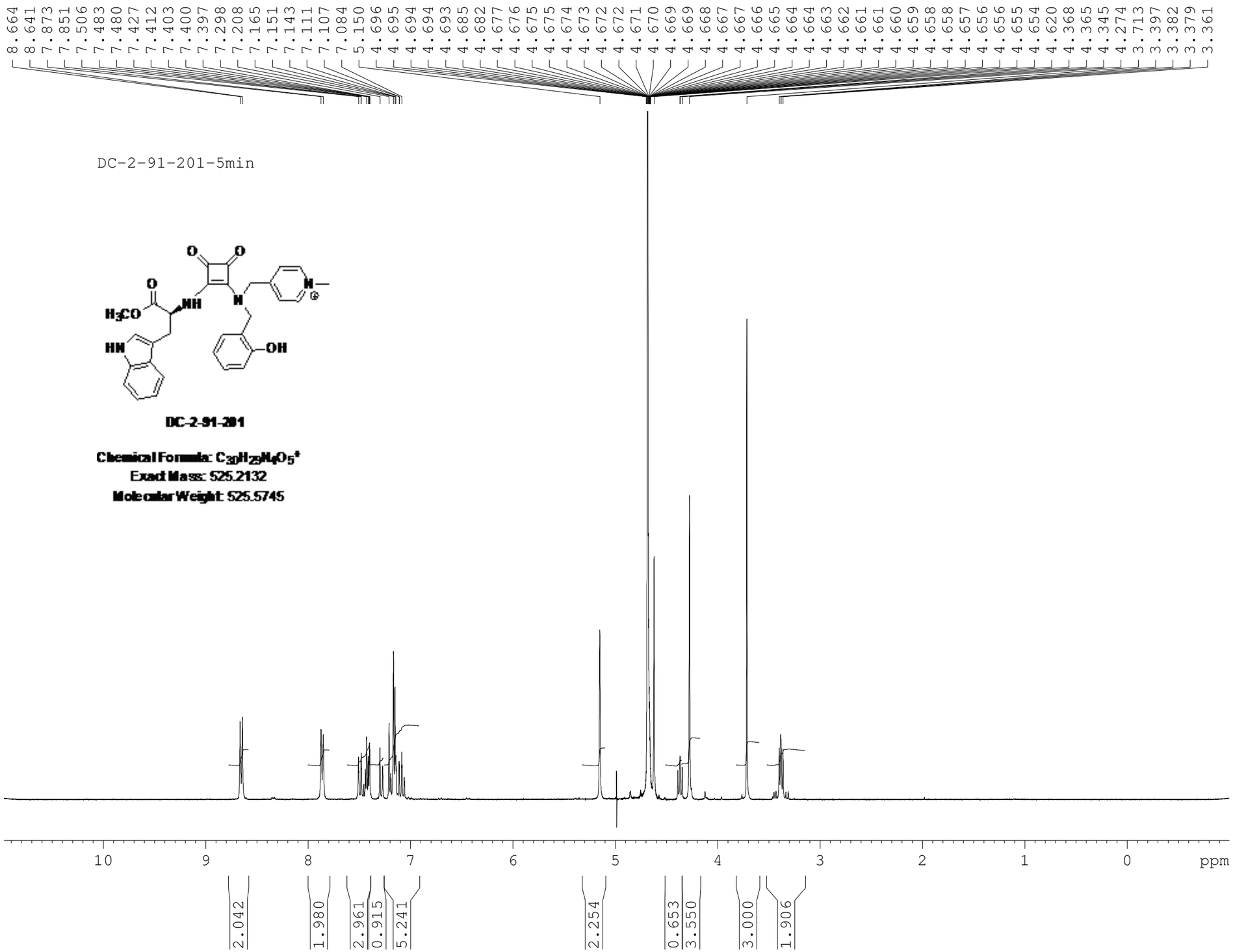
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
82 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-29 H: 0-27 N: 0-4 O: 0-5 Na: 0-1

File name:dc-2-91-101 Notebook Ref: 24689 IU Database#: 24689
Instrument: LCT KC366 Test name: Accurate Mass 28-Apr-2010 11:56:47
Method: ESI-TOF Base Peak Mass: 511.19808960
dc-2-91-101 31 (0.786) AM (Cen,6, 90.00, Ar,4000.0,922.36,0.70,LS 4); Sm (SG, 2x4.00); Cm (27:50) 1: TOF MS ES+
4.65e3



Minimum:	80.00				-1.5		
Maximum:	100.00		5.0	10.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
511.1981	100.00	511.1981	0.0	0.0	18.5	1145.0	C29 H27 N4 O5
512.2056	84.11	---					

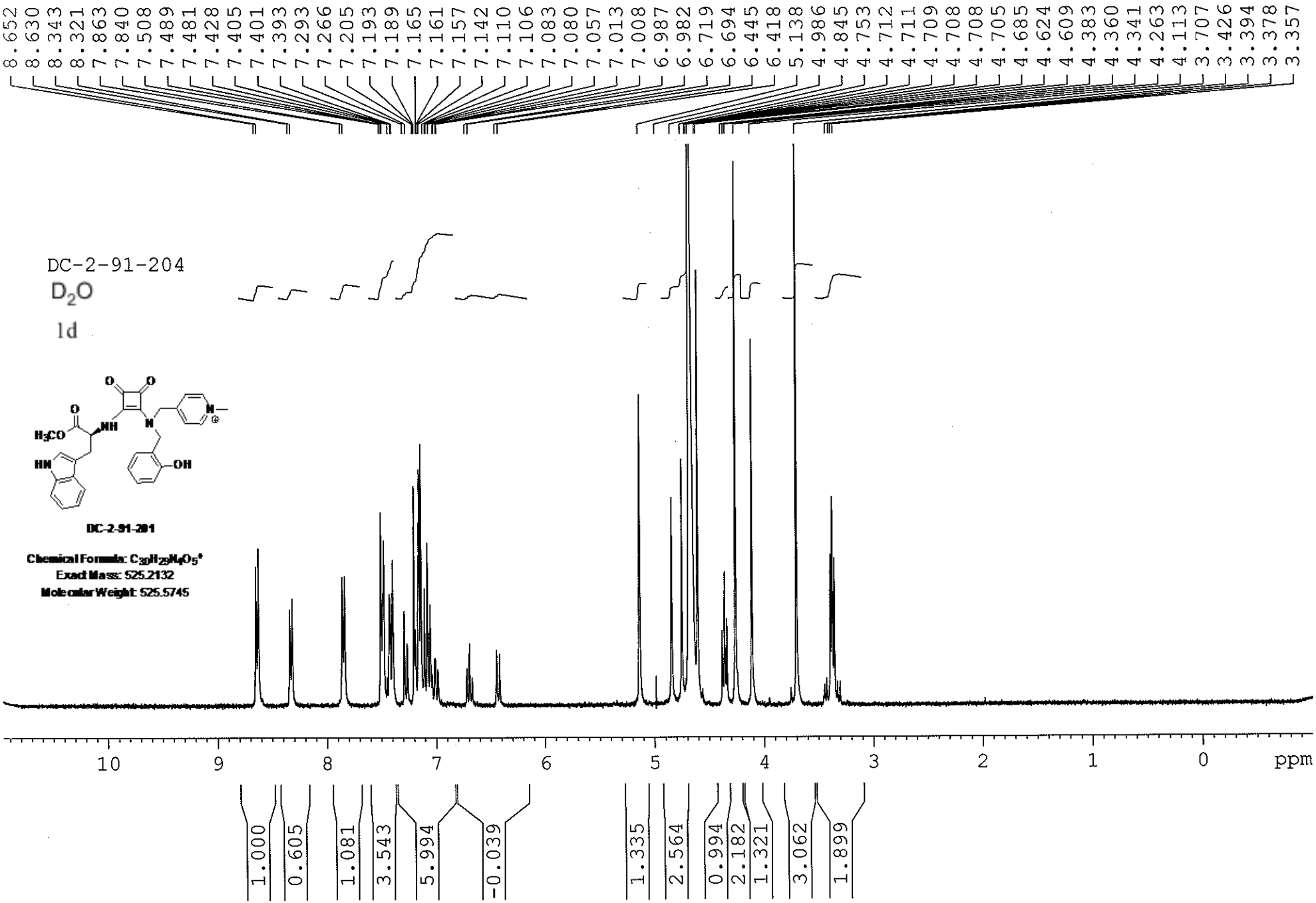


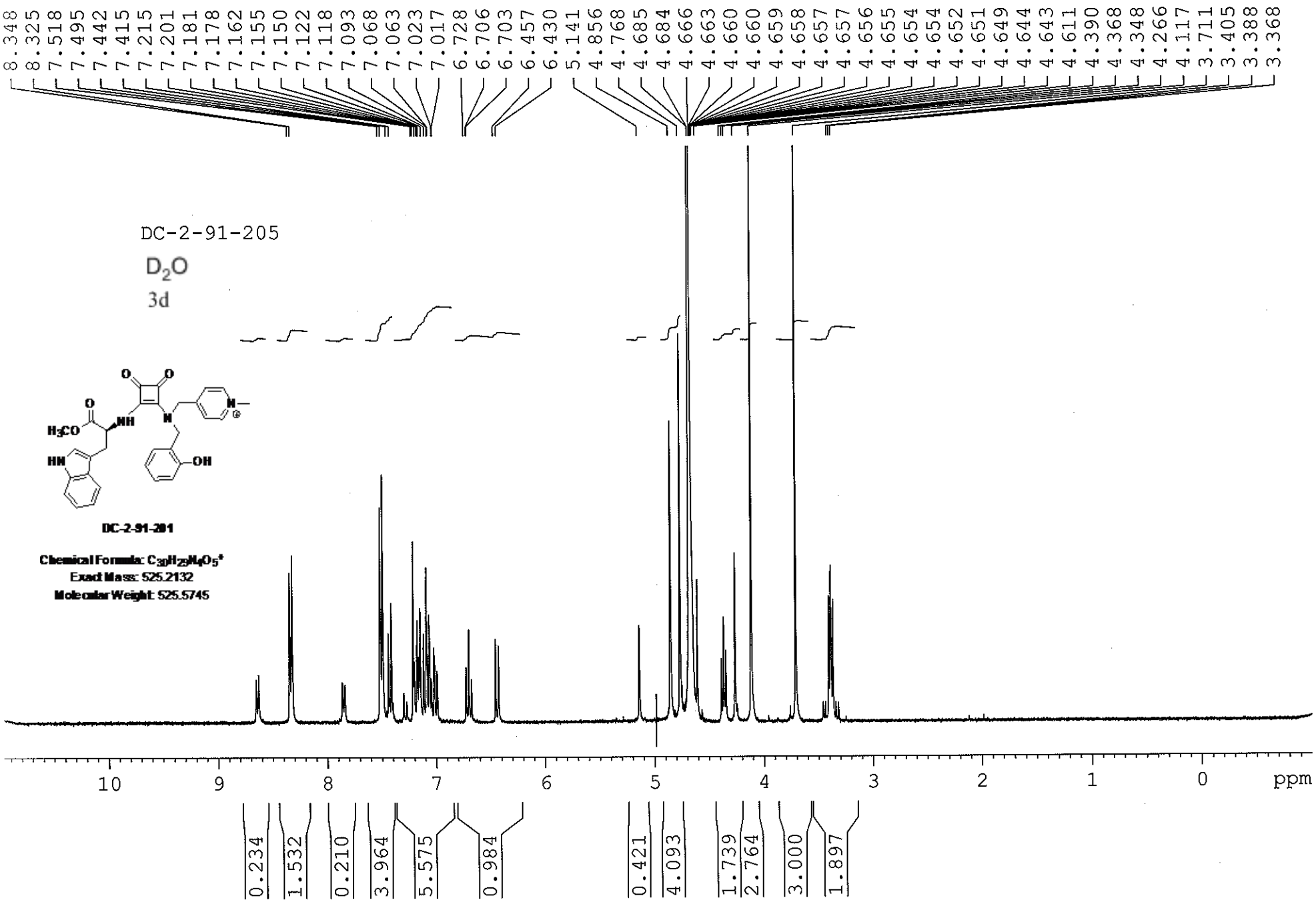

```
Current Data Parameters
NAME         DC-2-91-201-5min
EXPNO                      1
PROCNO                     1

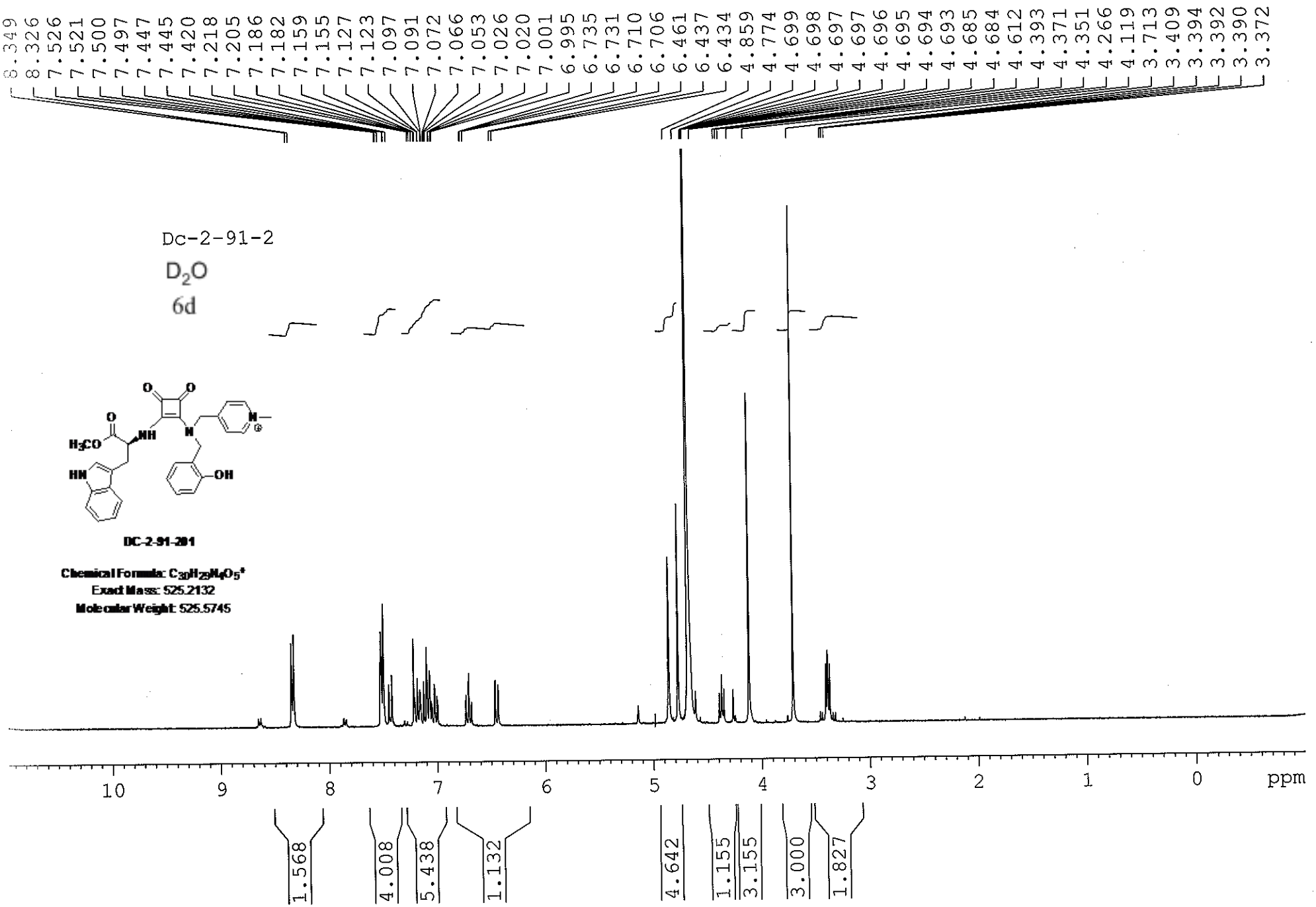
F2 - Acquisition Parameters
Date_              20100226
Time                   9.21
INSTRUM               spect
PROBHD    5 mm QNP  1H/1
PULPROG                  zg
TD                    32768
SOLVENT                D2O
NS                        32
DS                         0
SWH                3591.954 Hz
FIDRES            0.109618 Hz
AQ                4.5613556 sec
RG                    574.7
DW                  139.200 usec
DE                   54.00 usec
TE                   298.2 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                      9.75 usec
PL1                     0.00 dB
SFO1           300.1315007 MHz

F2 - Processing parameters
SI                     32768
SF             300.1300042 MHz
WDW                       no
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.00
```


8.652
8.630
8.343
8.321
7.863
7.840
7.508
7.489
7.481
7.428
7.405
7.401
7.393
7.293
7.266
7.205
7.193
7.189
7.165
7.161
7.157
7.142
7.110
7.106
7.083
7.080
7.057
7.013
7.008
6.987
6.982
6.719
6.694
6.445
6.418
5.138
4.986
4.845
4.753
4.712
4.711
4.709
4.708
4.708
4.705
4.685
4.624
4.609
4.383
4.360
4.341
4.263
4.113
3.707
3.426
3.394
3.378
3.357
DC-2-91-204
D2O
1d
DC-2-91-201
Chemical Formula: C30H29N4O5+
Exact Mass: 525.2132
Molecular Weight: 525.5745
10
9
8
7
6
5
4
3
2
1
0
ppm
1.000
0.605
1.081
3.543
5.994
-0.039
1.335
2.564
0.994
2.182
1.321
3.062
1.899


8.348
8.325
7.518
7.495
7.442
7.415
7.215
7.201
7.181
7.178
7.162
7.155
7.150
7.122
7.118
7.093
7.068
7.063
7.023
7.017
6.728
6.706
6.703
6.457
6.430
5.141
4.856
4.768
4.685
4.684
4.666
4.663
4.660
4.660
4.659
4.658
4.657
4.657
4.656
4.655
4.654
4.654
4.652
4.651
4.649
4.644
4.643
4.611
4.390
4.368
4.348
4.266
4.117
3.711
3.405
3.388
3.368
DC-2-91-205
D2O
3d
DC-2-91-201
Chemical Formula: C30H29N4O5+
Exact Mass: 525.2132
Molecular Weight: 525.5745
10
9
8
7
6
5
4
3
2
1
0
ppm
0.234
1.532
0.210
3.964
5.575
0.984
0.421
4.093
1.739
2.764
3.000
1.897

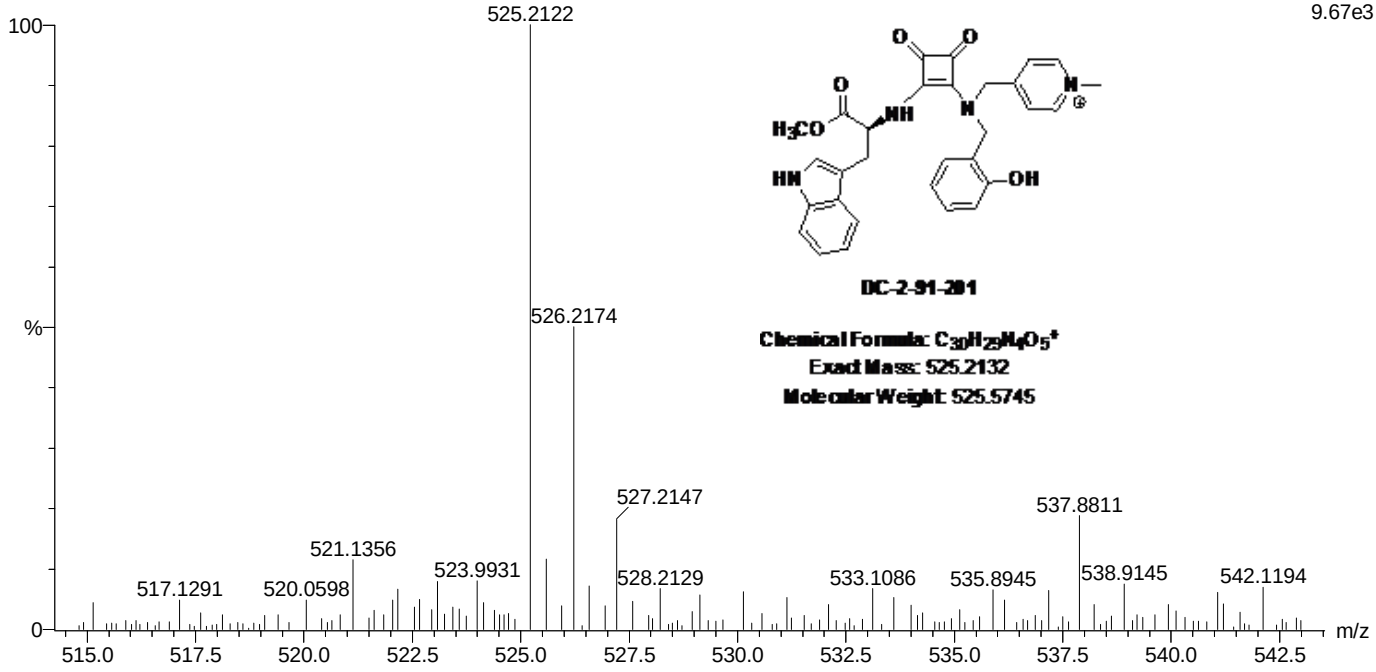

8.349
8.326
7.526
7.521
7.500
7.497
7.447
7.445
7.420
7.218
7.205
7.186
7.182
7.159
7.155
7.127
7.123
7.097
7.091
7.072
7.066
7.053
7.026
7.020
7.001
6.995
6.735
6.731
6.710
6.706
6.461
6.437
6.434
4.859
4.774
4.699
4.698
4.697
4.697
4.696
4.695
4.694
4.693
4.685
4.684
4.612
4.393
4.371
4.351
4.266
4.119
3.713
3.409
3.394
3.392
3.390
3.372
Dc-2-91-2
D2O
6d
DC-2-91-2D1
Chemical Formula: C30H29N4O5+
Exact Mass: 525.2132
Molecular Weight: 525.5745
10
9
8
7
6
5
4
3
2
1
0
ppm
1.568
4.008
5.438
1.132
4.642
1.155
3.155
3.000
1.827

Elemental Composition Report

Tolerance = 20.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

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: 0-30 H: 0-29 N: 0-4 O: 0-5

File name:dc-2-91-201 Notebook Ref: 24822 IU Database#: 24822
Instrument: LCT KC366 Test name: Accurate Mass 06-May-2010 11:38:59
Method: ESI-TOF Base Peak Mass: 525.21215820
dc-2-91-201 34 (0.853) AM (Cen,8, 90.00, Ar,4000.0,1222.14,0.70,LS 6); Sm (SG, 2x4.00); Cm (27:42) 1: TOF MS ES+
9.67e3

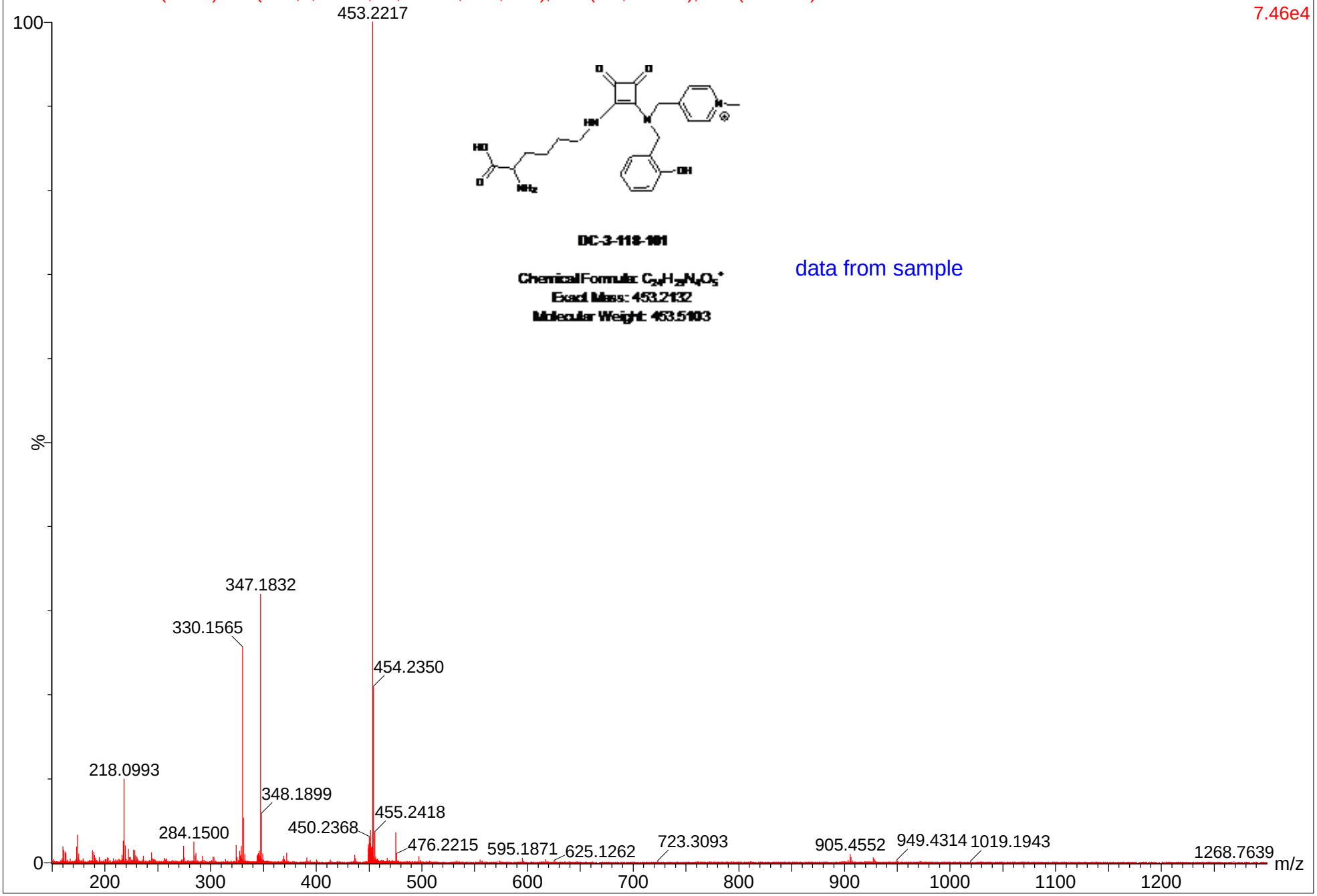


Minimum:	80.00				-1.5		
Maximum:	100.00		5.0	20.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
525.2122	100.00	525.2138	-1.6	-3.0	18.5	447.6	C30 H29 N4 O5

File name:dc-3-118-101 **Notebook Ref: 25174** **IU Database#: 25174**
Instrument: LCT KC366 **Test name: Accurate Mass** **01-Jun-2010 15:51:53**
Method: ESI-TOF **Base Peak Mass: 453.22174072**

dc-3-118-101 146 (3.254) AM (Cen,8, 80.00, Ar,6000.0,0.00,0.70); Sm (SG, 2x4.00); Cm (140:159)

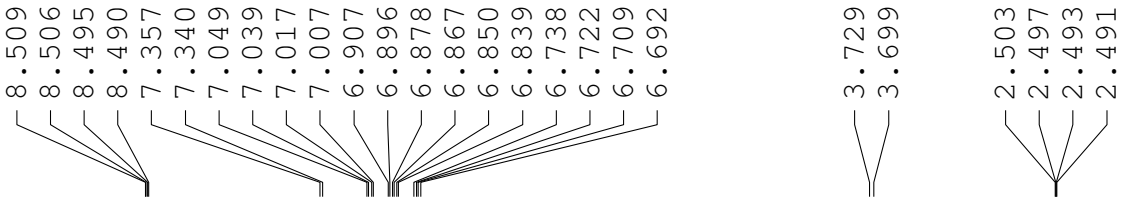
1: TOF MS ES+
7.46e4



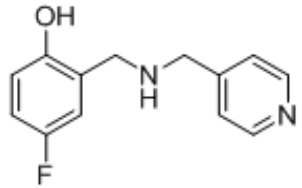
DC-3-118-101

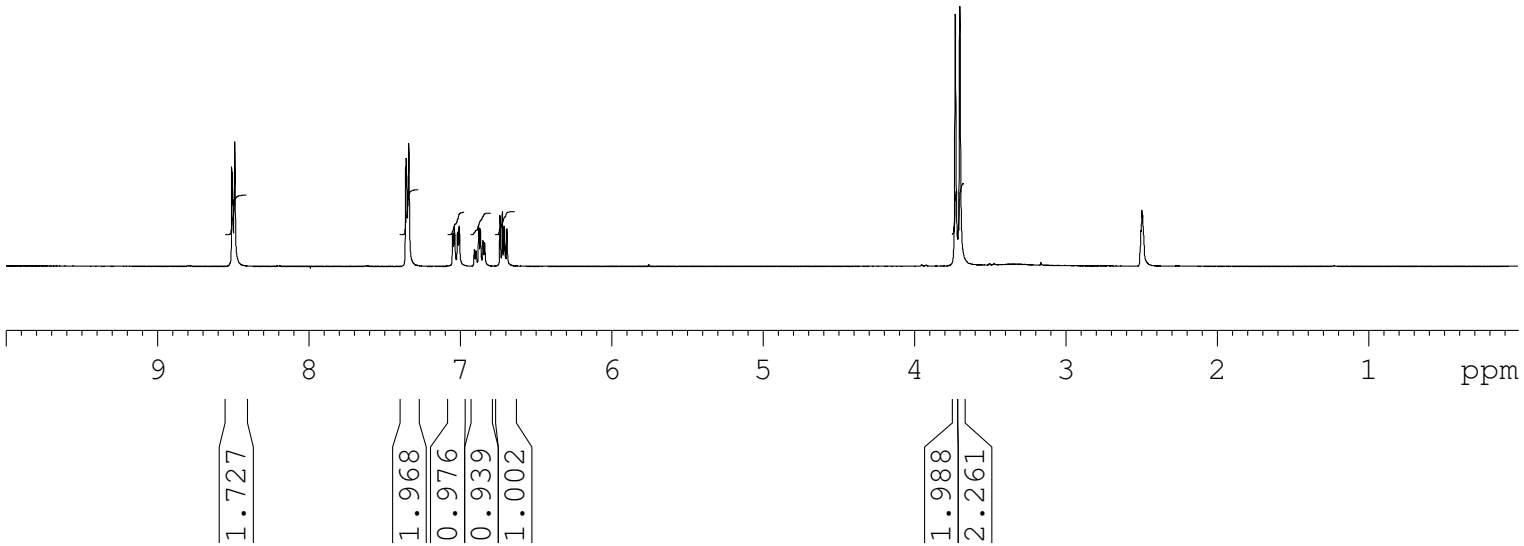
Chemical Formula: $C_{24}H_{29}N_4O_5^+$
Exact Mass: 453.2132
Molecular Weight: 453.5103

data from sample



dc-3-127-101



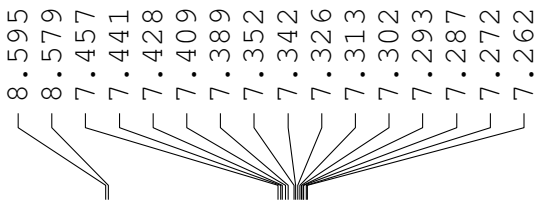


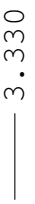
Current Data Parameters
NAME dc−3−127−101
EXPNO 1
PROCNO 1

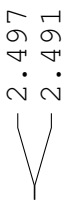
F2 − Acquisition Parameters
Date_ 20100604
Time 10.38
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 406.4
DW 104.400 usec
DE 54.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

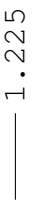
======== CHANNEL f1 =====
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1324000 MHz

F2 − Processing parameters
SI 32768
SF 300.1300024 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

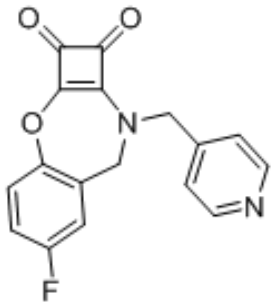


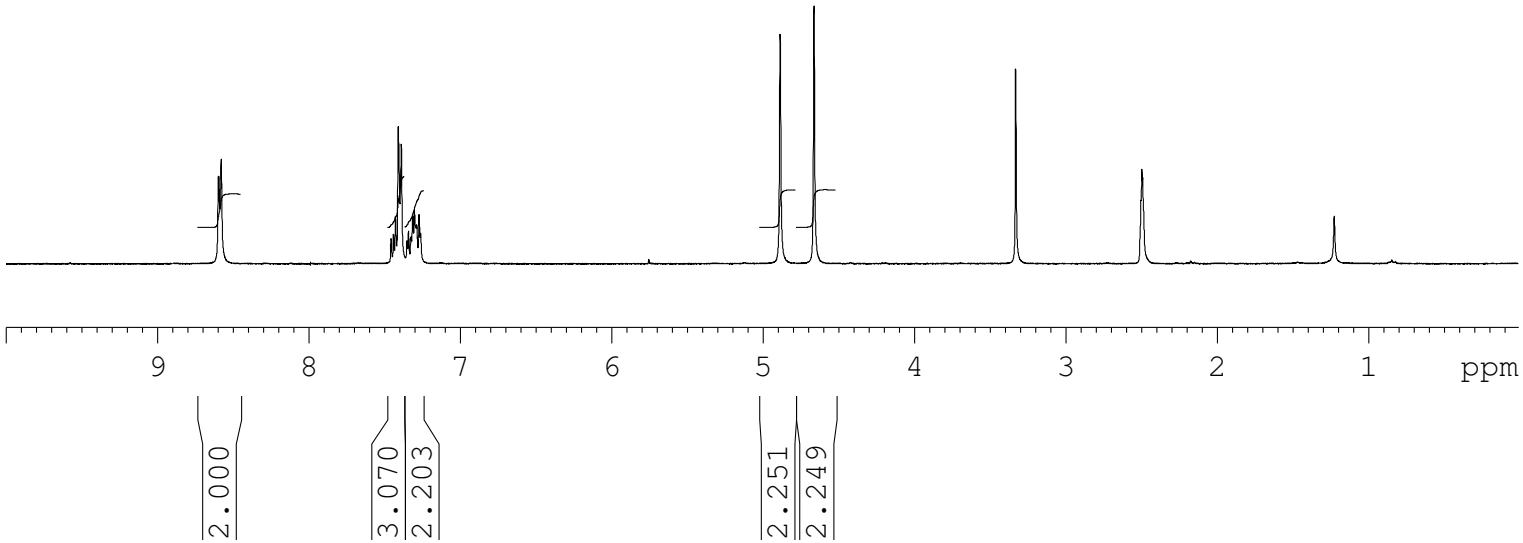






dc-3-134-101





Current Data Parameters
NAME dc−3−134−101
EXPNO 1
PROCNO 1

F2 − Acquisition Parameters
Date_ 20100604
Time 10.33
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 645.1
DW 104.400 usec
DE 54.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 =====
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1324000 MHz

F2 − Processing parameters
SI 32768
SF 300.1300024 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

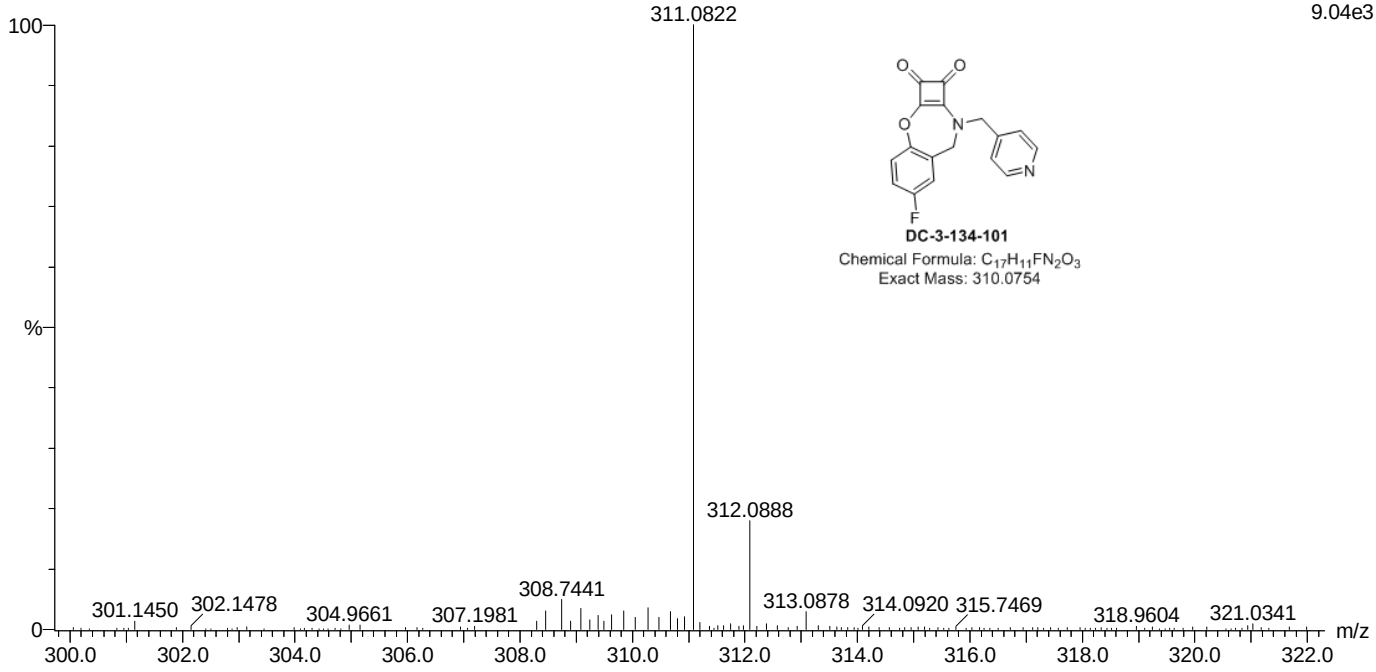
Elemental Composition Report

Multiple Mass Analysis: 2 mass(es) processed

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

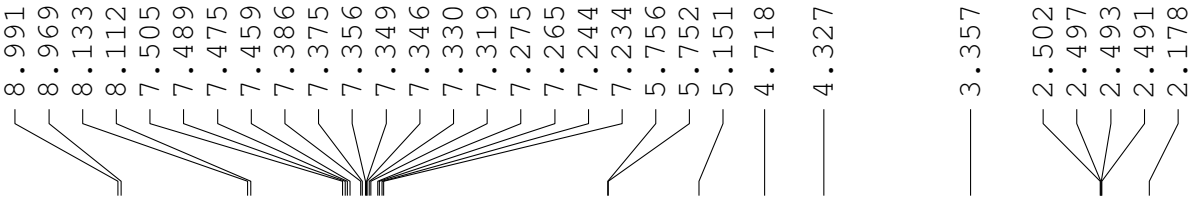
Monoisotopic Mass, Odd and Even Electron Ions
29 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-17 H: 0-12 N: 0-2 O: 0-3 F: 0-1 Na: 0-1

File name:dc-3-134-101 Notebook Ref: 25561 IU Database#: 25561
Instrument: LCT KC366 Test name: Accurate Mass 15-Jun-2010 16:24:54
Method: ESI-TOF Base Peak Mass: 311.08215332
dc-3-134-101 70 (1.753) AM (Cen,8, 90.00, Ar,5000.0,1222.14,0.70,LS 7); Sm (SG, 2x4.00); Cm (70:82) 1: TOF MS ES+
9.04e3

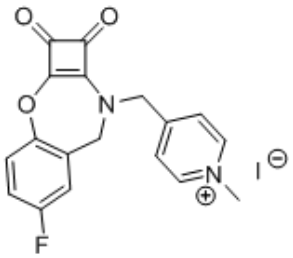


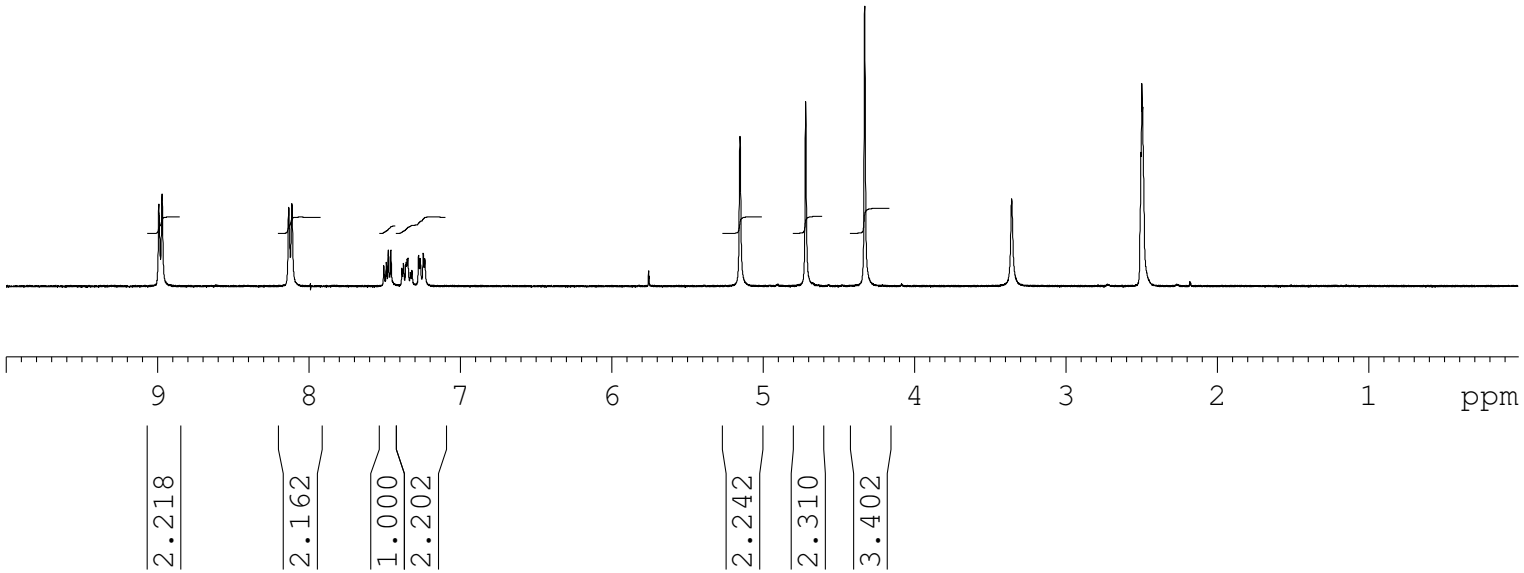
DC-3-134-101
Chemical Formula: $C_{17}H_{11}FN_2O_3$
Exact Mass: 310.0754

Minimum:	70.00				-1.5		
Maximum:	100.00		5.0	10.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
311.0822	100.00	311.0832	-1.0	-3.2	12.5	12.2	C17 H12 N2 O3 F



dc-3-137-101



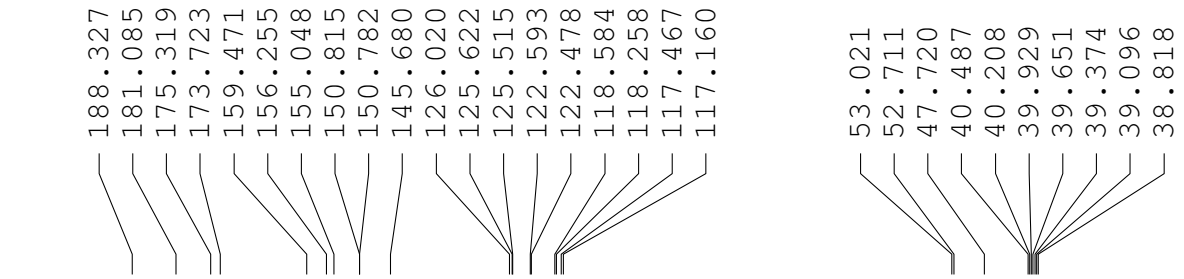


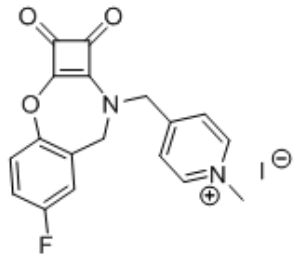
Current Data Parameters
NAME dc−3−137−101
EXPNO 1
PROCNO 1

F2 − Acquisition Parameters
Date_ 20100604
Time 10.28
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 724.1
DW 104.400 usec
DE 54.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 =====
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1324000 MHz

F2 − Processing parameters
SI 32768
SF 300.1300024 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00





Current Data Parameters
NAME DC−3−137−carbon−
EXPNO 1
PROCNO 1

F2 − Acquisition Parameters
Date_ 20100730
Time 15.48
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgdc
TD 65536
SOLVENT DMSO
NS 250
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 3251
DW 26.550 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1

======== CHANNEL f1 ===
NUC1 13C
P1 11.80 usec
PL1 0.00 dB
SFO1 75.4760200 MHz

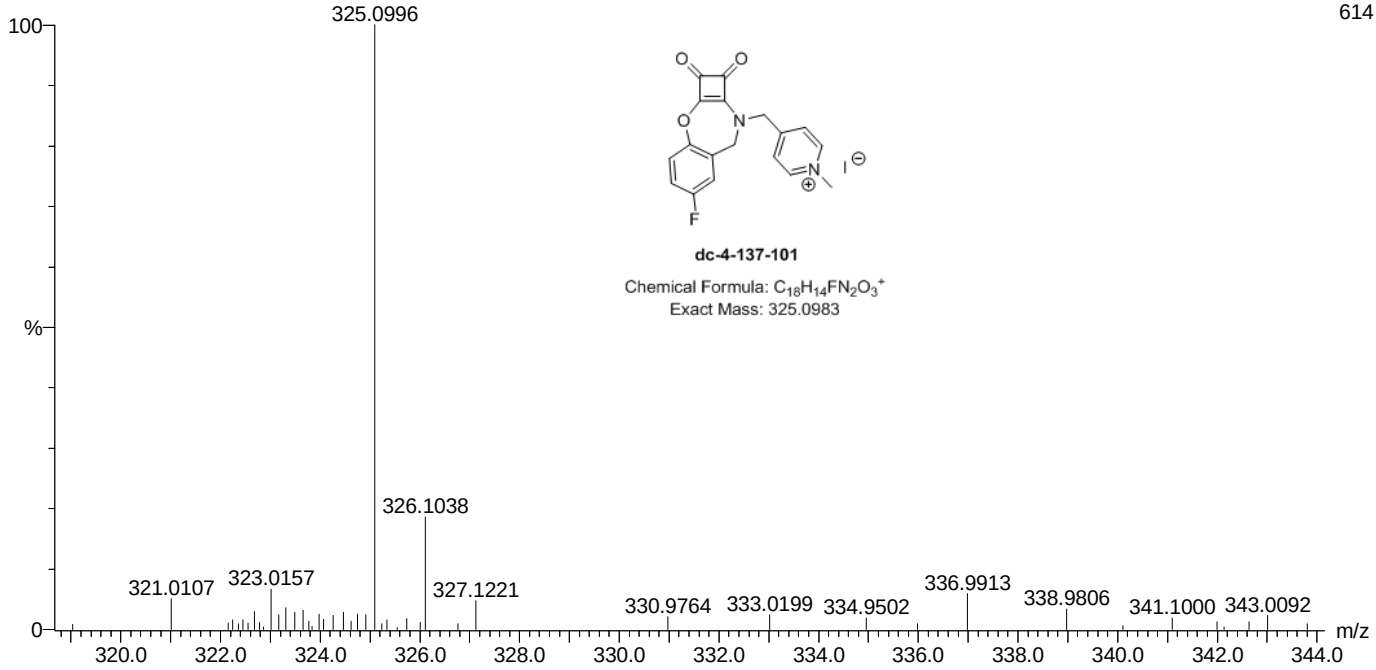
======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz

Elemental Composition Report

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

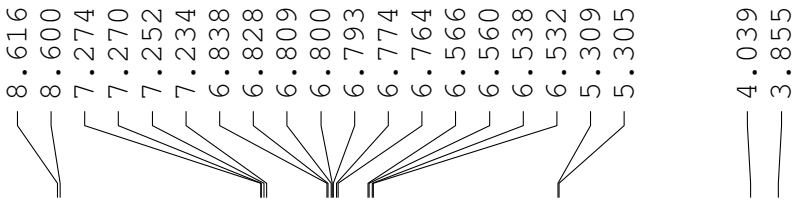
Monoisotopic Mass, Even Electron Ions
29 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-18 H: 0-14 N: 0-2 O: 0-3 F: 0-1 Na: 0-1

File name:dc-3-137-101 Notebook Ref: 25562 IU Database#: 25562
Instrument: LCT KC366 Test name: Accurate Mass 15-Jun-2010 17:19:02
Method: ESI-TOF Base Peak Mass: 325.09957886
dc-3-137-101-rerun5 42 (1.053) AM (Cen,8, 90.00, Ar,5000.0,922.36,0.70,LS 7); Sm (SG, 2x4.00); Cm (42) 1: TOF MS ES+
614

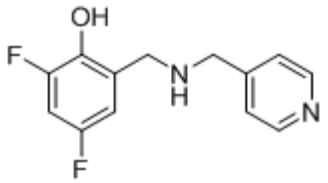


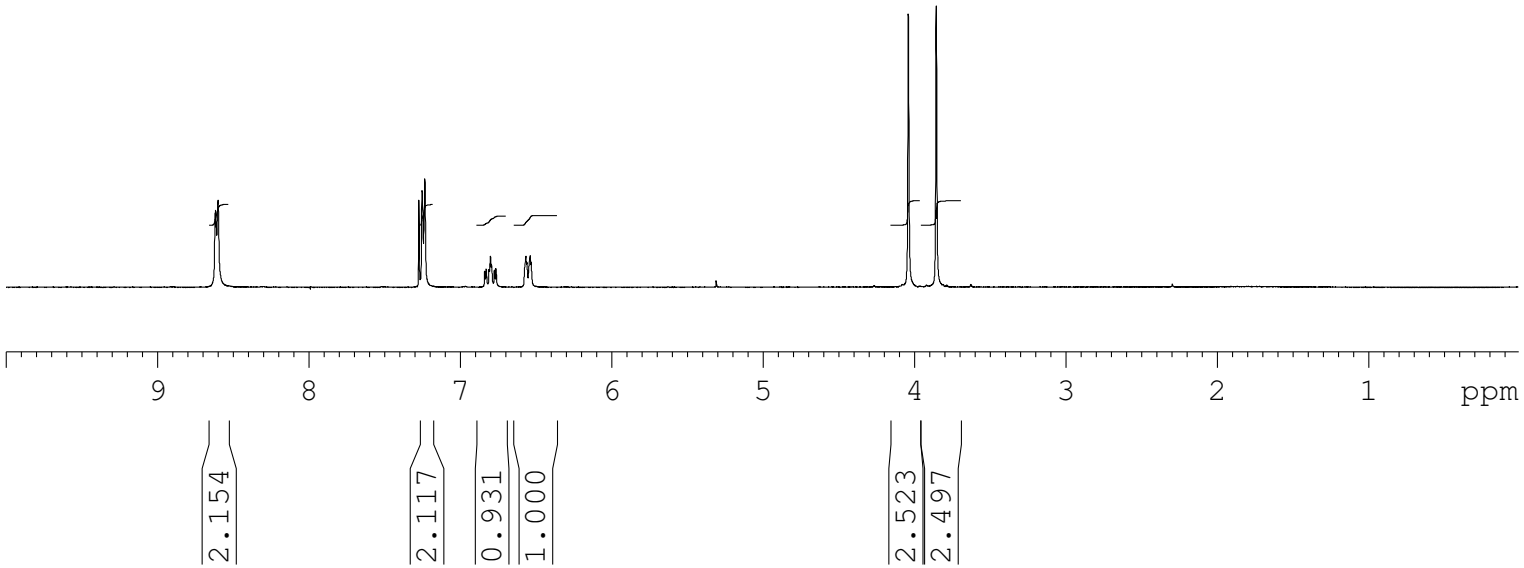
dc-4-137-101
Chemical Formula: $C_{18}H_{14}FN_2O_3^+$
Exact Mass: 325.0983

Minimum:	70.00				-1.5		
Maximum:	100.00		5.0	50.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
325.0996	100.00	325.0988	0.8	2.5	12.5	3.7	C18 H14 N2 O3 F



dc-4-23-101



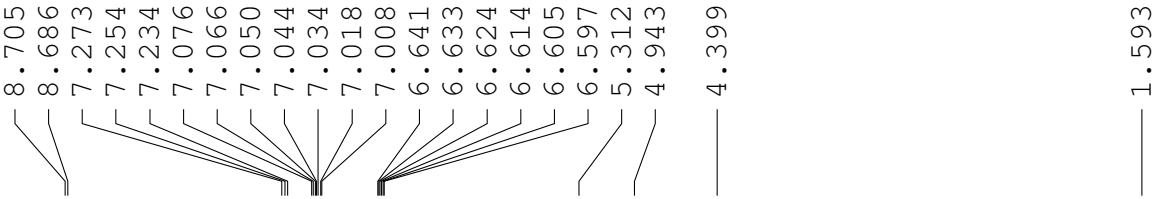


Current Data Parameters
NAME dc−4−23−101
EXPNO 1
PROCNO 1

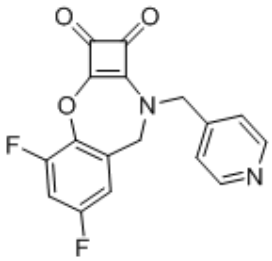
F2 − Acquisition Parameters
Date_ 20100628
Time 11.43
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 362
DW 104.400 usec
DE 54.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

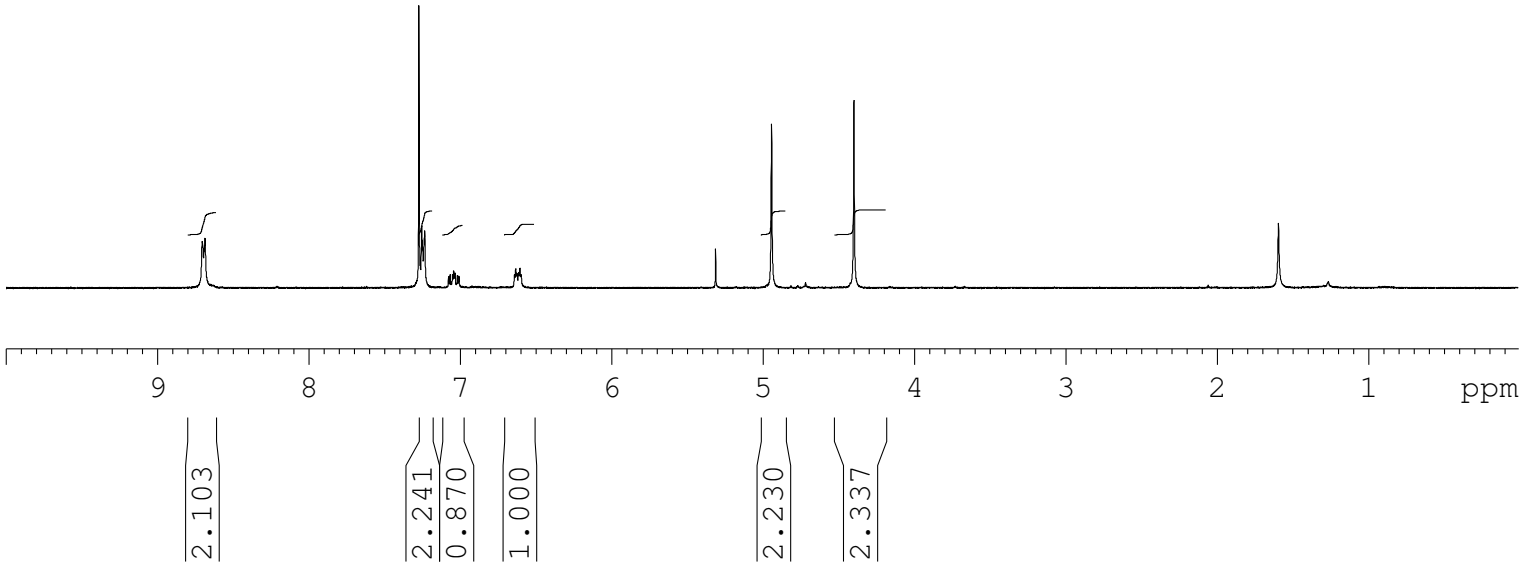
======== CHANNEL f1 ===
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1324000 MHz

F2 − Processing parameters
SI 32768
SF 300.1300024 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



dc-4-25-101





Current Data Parameters
NAME dc−4−25−101
EXPNO 1
PROCNO 1

F2 − Acquisition Parameters
Date_ 20100629
Time 15.46
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 1149.4
DW 104.400 usec
DE 54.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ===
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1324000 MHz

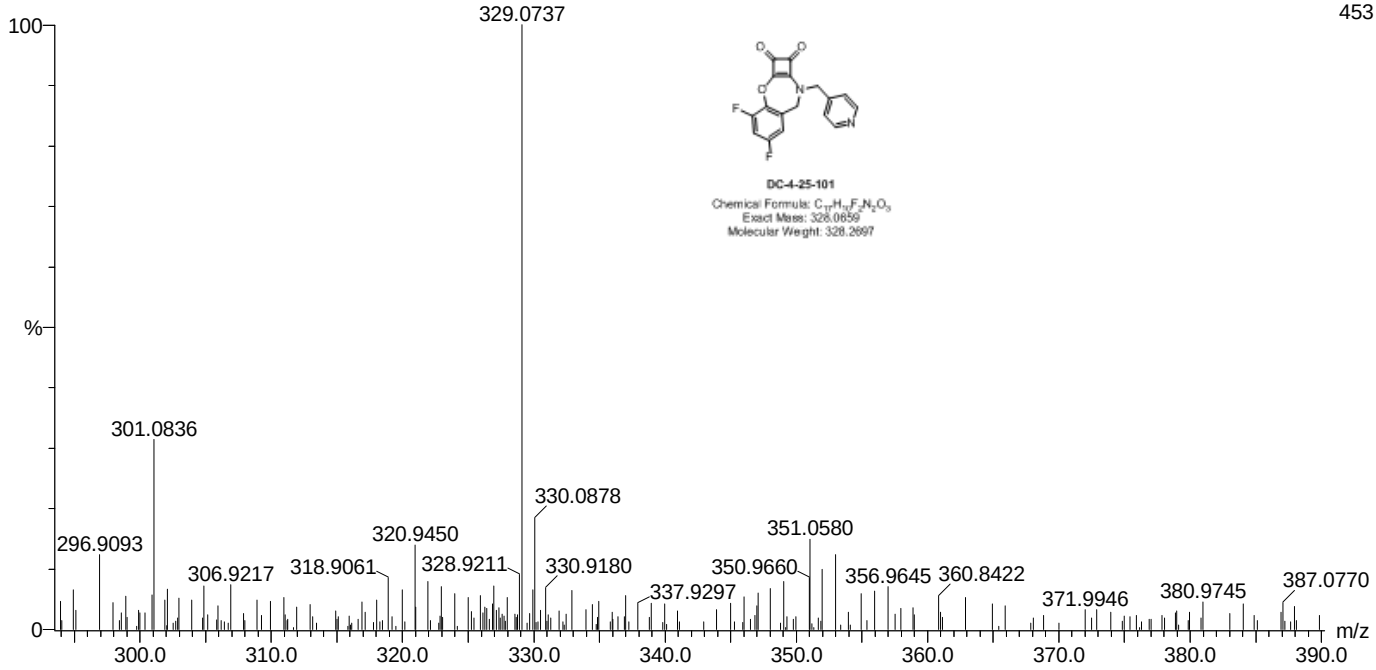
F2 − Processing parameters
SI 32768
SF 300.1300024 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

Elemental Composition Report

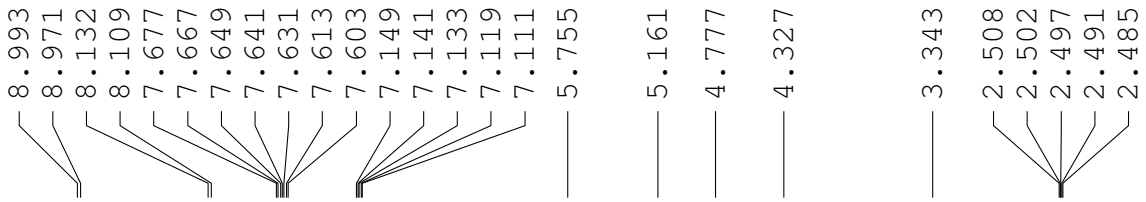
Tolerance = 100.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
28 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-17 H: 0-11 N: 0-2 O: 0-3 F: 0-2

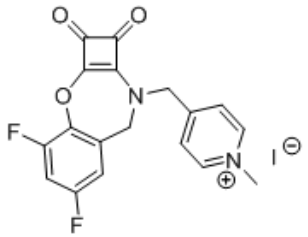
File name:dc-4-25-101 Notebook Ref: 25942 IU Database#: 25942
Instrument: LCT KC366 Test name: Accurate Mass 09-Jul-2010 14:37:47
Method: ESI-TOF Base Peak Mass: 329.07366943
dc-4-25-101 115 (2.887) AM (Cen,8, 80.00, Ar,5000.0,1222.14,0.70,LS 7); Sm (SG, 2x4.00); Cm (115:117) 1: TOF MS ES+
453

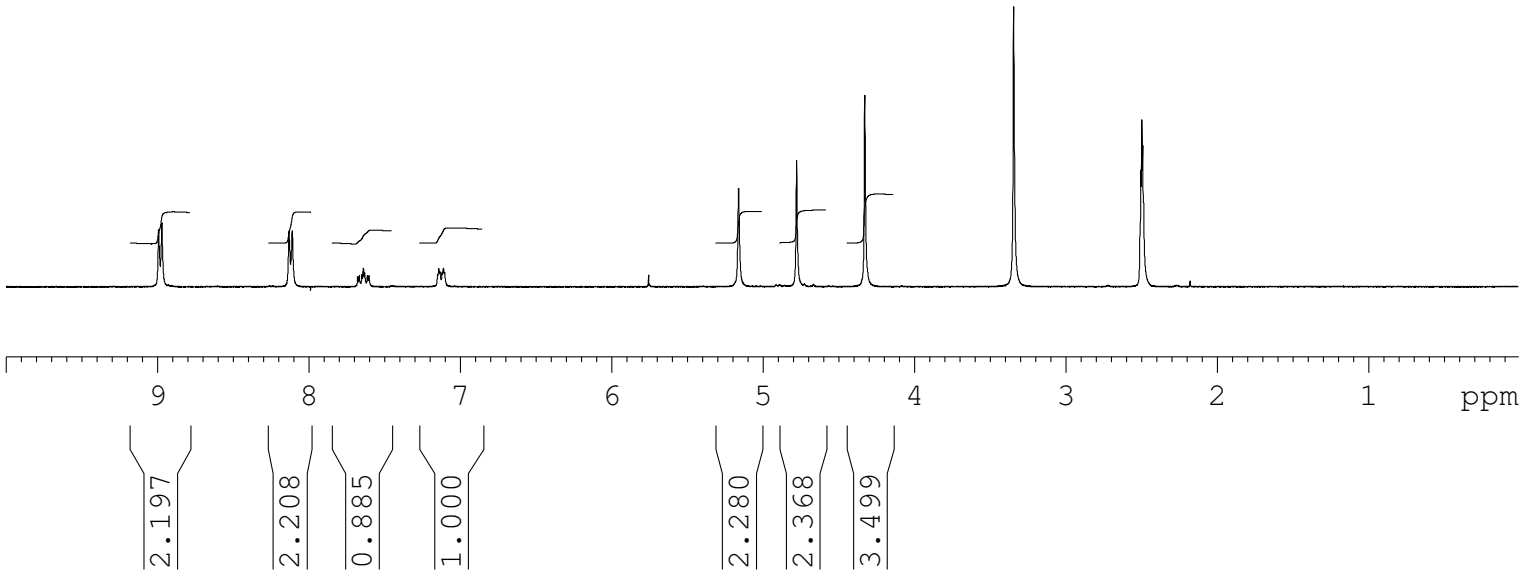


Minimum:	60.00				-1.5		
Maximum:	100.00		5.0	100.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
329.0737	100.00	329.0738	-0.1	-0.3	12.5	0.7	C17 H11 N2 O3 F2



dc-4-33-101c



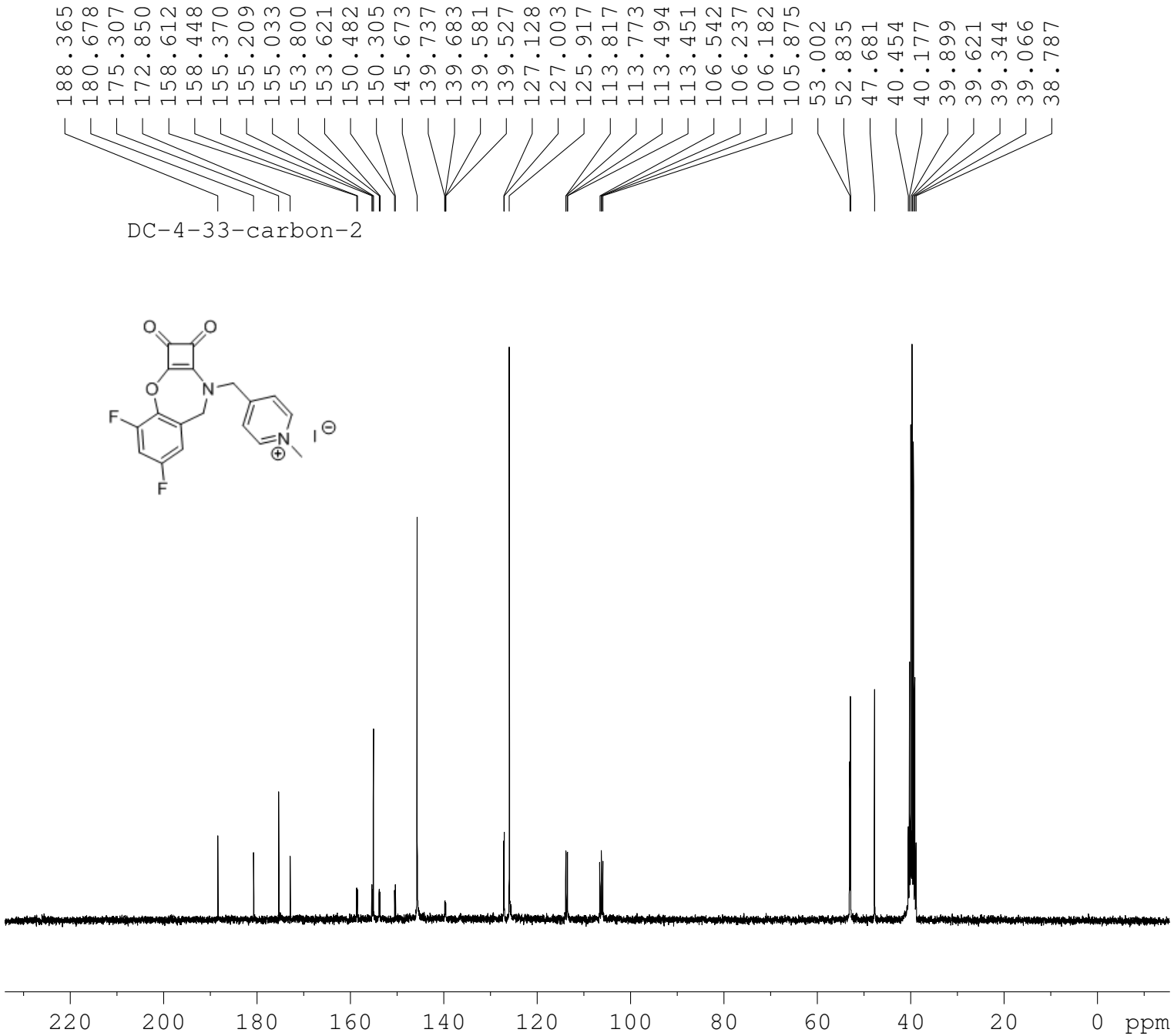


Current Data Parameters
NAME dc−4−33−101c
EXPNO 1
PROCNO 1

F2 − Acquisition Parameters
Date_ 20100709
Time 10.49
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT D2O
NS 32
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 1024
DW 104.400 usec
DE 54.00 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ===
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1324000 MHz

F2 − Processing parameters
SI 32768
SF 300.1300024 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



Current Data Parameters
NAME DC−4−33−carbon−2
EXPNO 1
PROCNO 1

F2 − Acquisition Parameters
Date_ 20100731
Time 17.53
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgdc
TD 65536
SOLVENT DMSO
NS 2400
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 5792.6
DW 26.550 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
d11 0.03000000 sec
TD0 1

======== CHANNEL f1 ===
NUC1 13C
P1 11.80 usec
PL1 0.00 dB
SFO1 75.4760200 MHz

======== CHANNEL f2 ===
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 0.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz

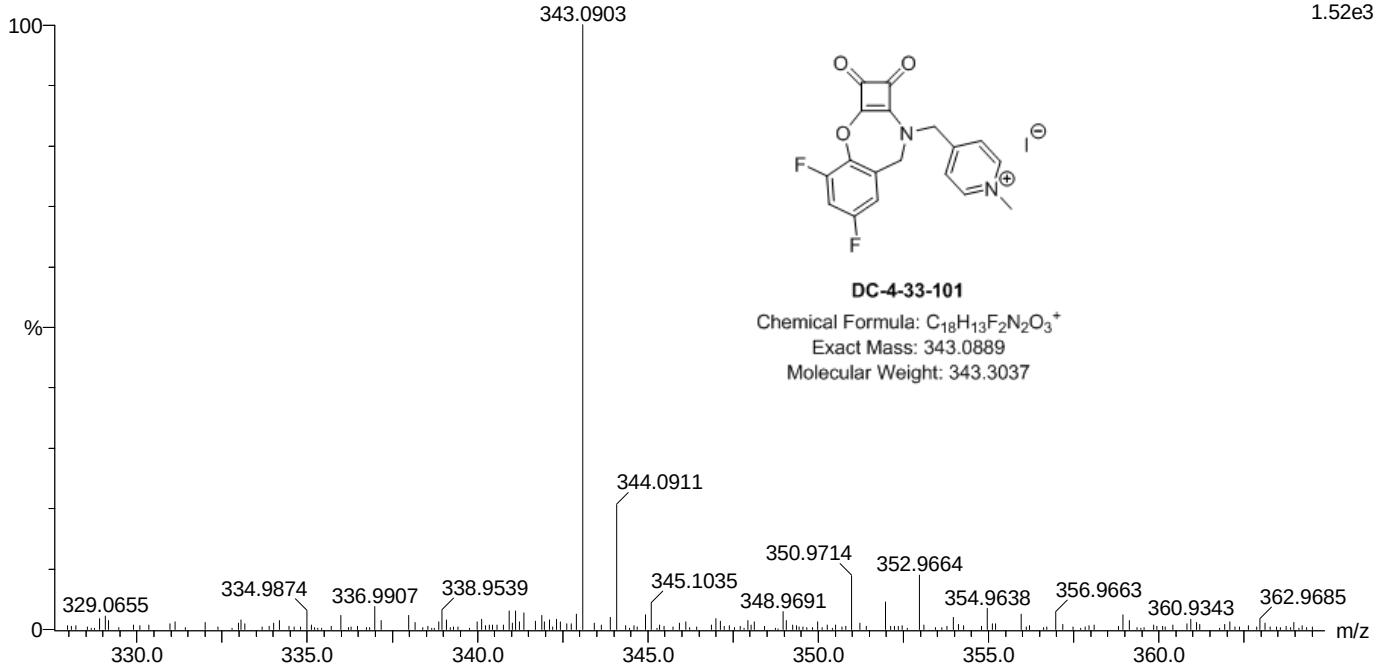
Elemental Composition Report

Multiple Mass Analysis: 2 mass(es) processed

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

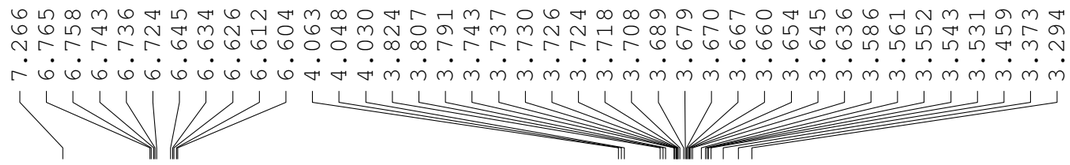
Monoisotopic Mass, Odd and Even Electron Ions
28 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-18 H: 0-13 N: 0-2 O: 0-3 F: 0-2

File name:dc-4-33-101 Notebook Ref: 26284 IU Database#: 26284
Instrument: LCT KC366 Test name: Accurate Mass 30-Jul-2010 12:34:30
Method: ESI-TOF Base Peak Mass: 343.09027100
dc-4-33-101b 30 (0.752) AM (Cen,8, 80.00, Ar,5000.0,1222.14,0.70,LS 6); Sm (SG, 2x6.00); Cm (29:30) 1: TOF MS ES+
1.52e3

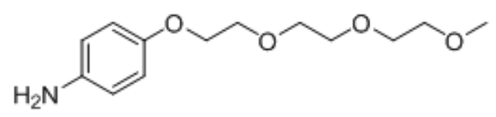


DC-4-33-101
Chemical Formula: $C_{18}H_{13}F_2N_2O_3^+$
Exact Mass: 343.0889
Molecular Weight: 343.3037

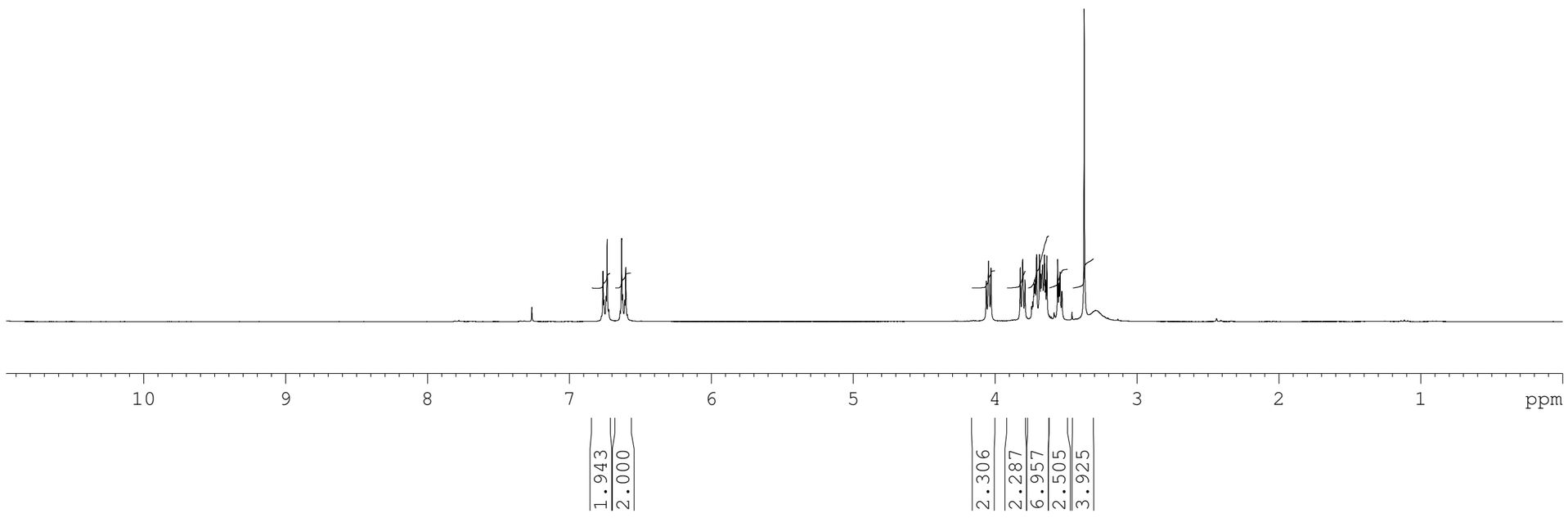
Minimum:	50.00				-1.5		
Maximum:	100.00		5.0	5.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
343.0903	100.00	343.0894	0.9	2.6	12.5	4.6	C18 H13 N2 O3 F2



DC-2-46-101



DC-2-46-101

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Current Data Parameters
NAME         DC-2-46-101
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100211
Time               10.11
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            3591.954 Hz
FIDRES        0.109618 Hz
AQ            4.5613556 sec
RG                 90.5
DW               139.200 usec
DE                54.00 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI                 32768
SF         300.1300042 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```
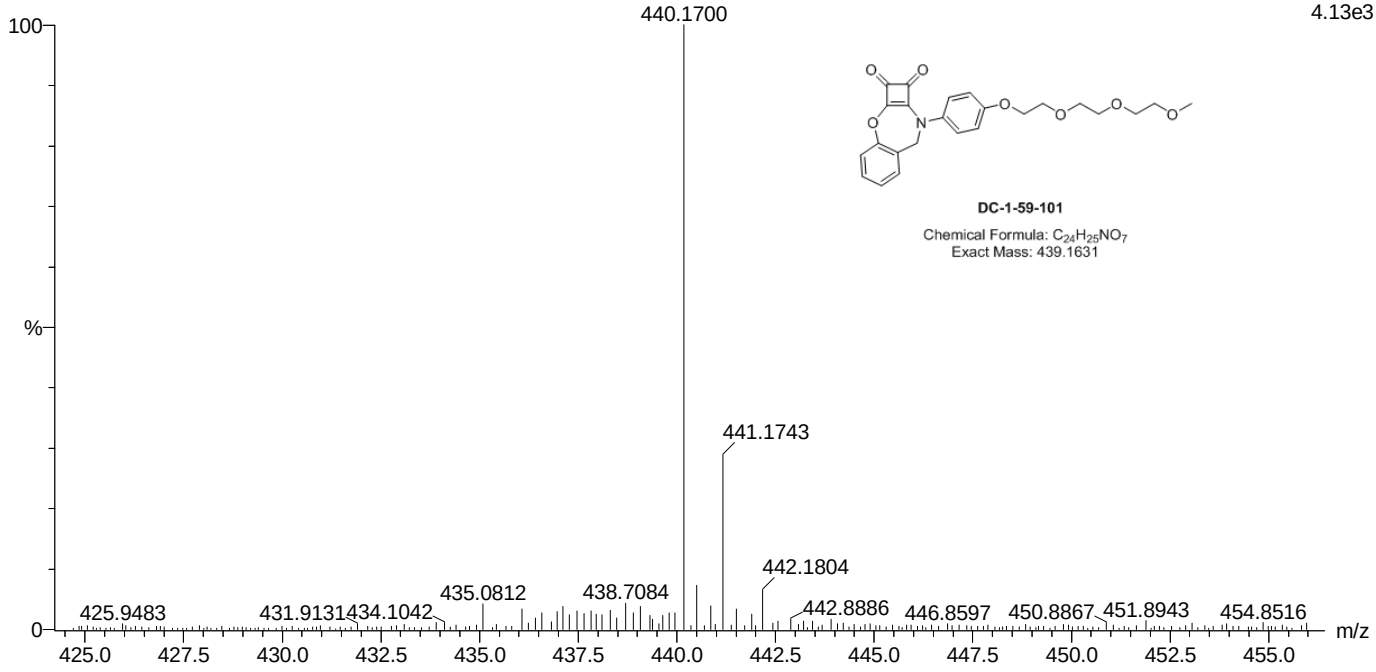

Elemental Composition Report

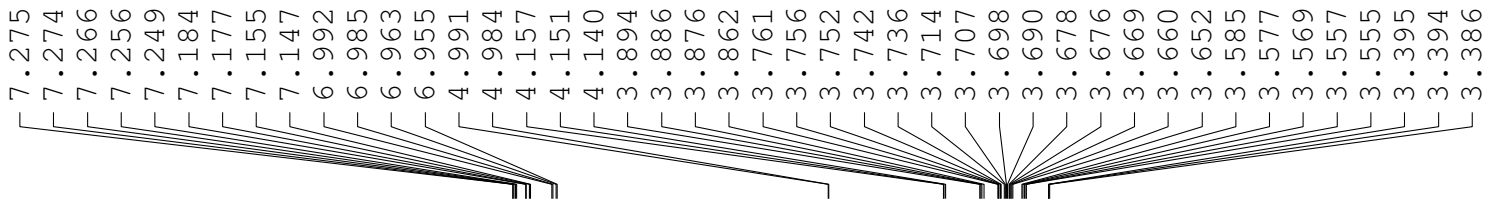
Tolerance = 15.0 PPM / DBE: min = -1.5, max = 80.0
Selected filters: None

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: 0-24 H: 0-26 N: 0-1 O: 0-7 Na: 0-1

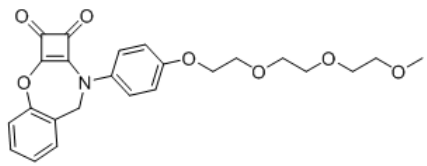
File name:dc-2-59-101 Notebook Ref: IU Database#:
Instrument: LCT KC366 Test name: Accurate Mass 26-Feb-2010 12:12:41
Method: ESI-TOF Base Peak Mass: 440.16995239
dc-2-59-101 23 (0.586) AM (Cen,6, 90.00, Ar,6000.0,1222.14,0.70,LS 4); Sm (SG, 2x6.00); Cm (23) 1: TOF MS ES+
4.13e3



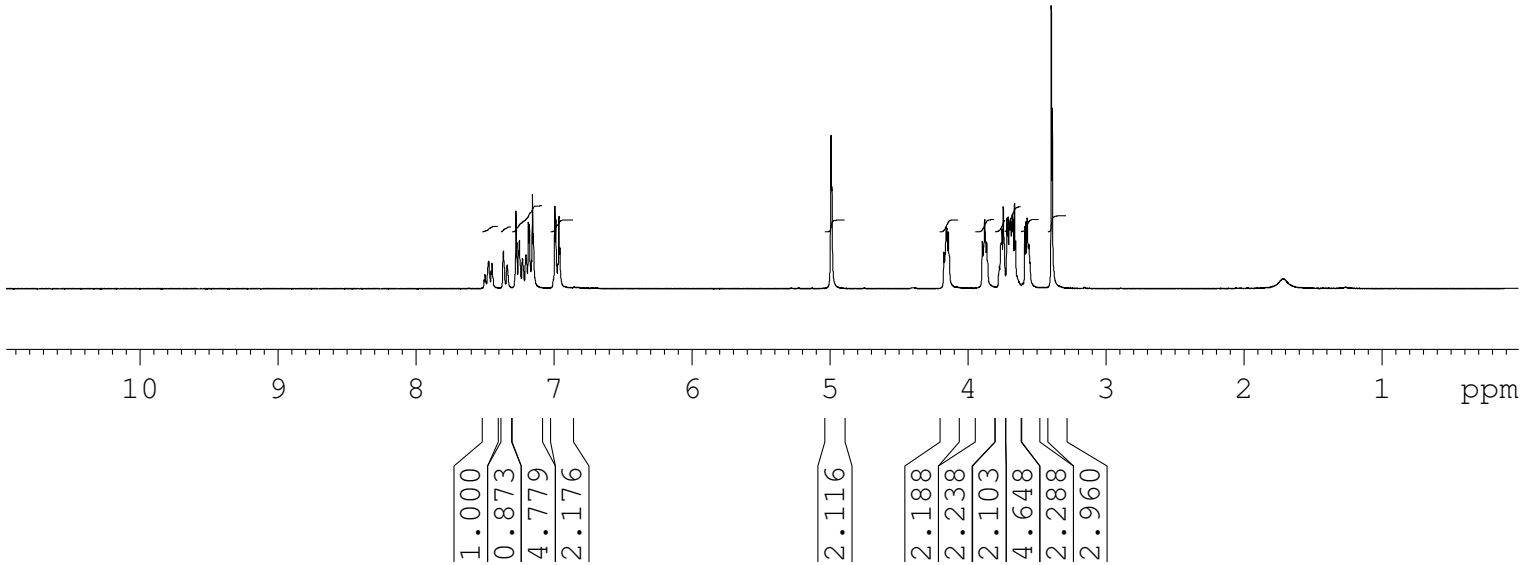
Minimum:	80.00				-1.5		
Maximum:	100.00		5.0	15.0	80.0		
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
440.1700	100.00	440.1709	-0.9	-2.0	12.5	5.8	C24 H26 N O7



dc-2-59-201b



D-2-59-201

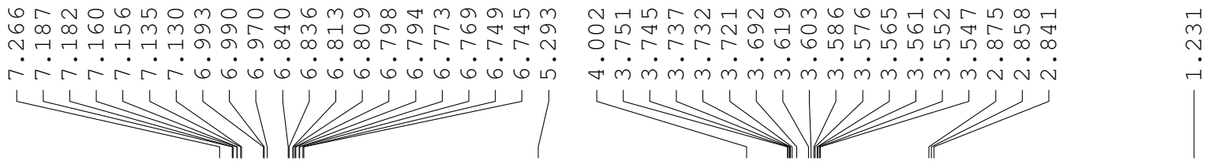


Current Data Parameters
NAME dc−2−59−201b
EXPNO 1
PROCNO 1

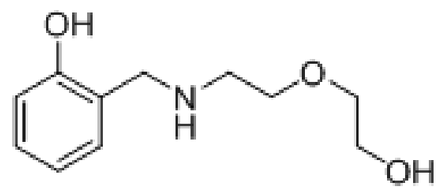
F2 − Acquisition Parameters
Date_ 20100814
Time 10.50
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 362
DW 104.400 usec
DE 54.00 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ===
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1324000 MHz

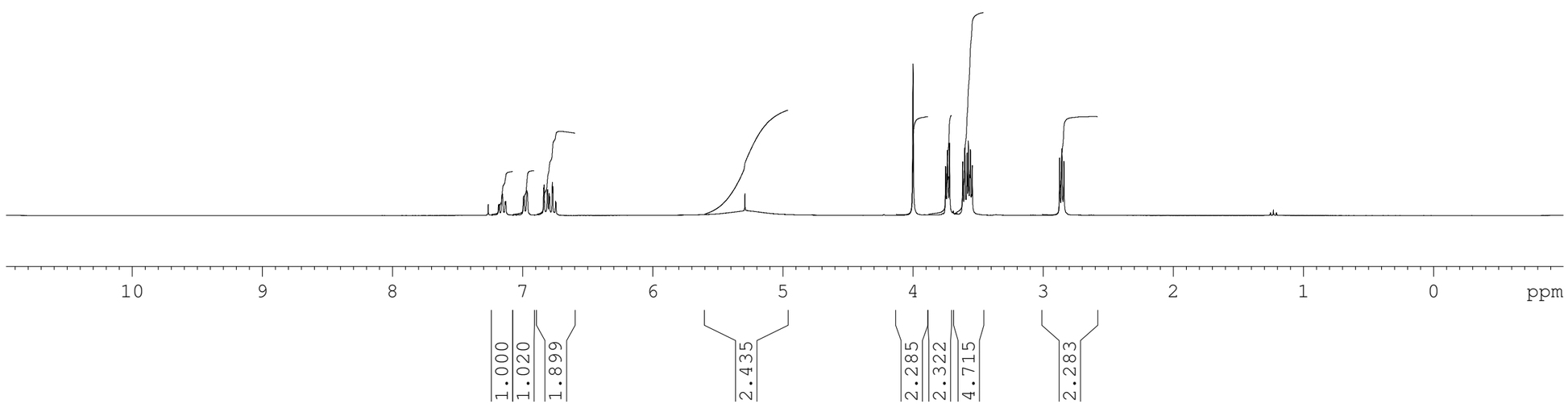
F2 − Processing parameters
SI 32768
SF 300.1300024 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



DC-2-61-101b

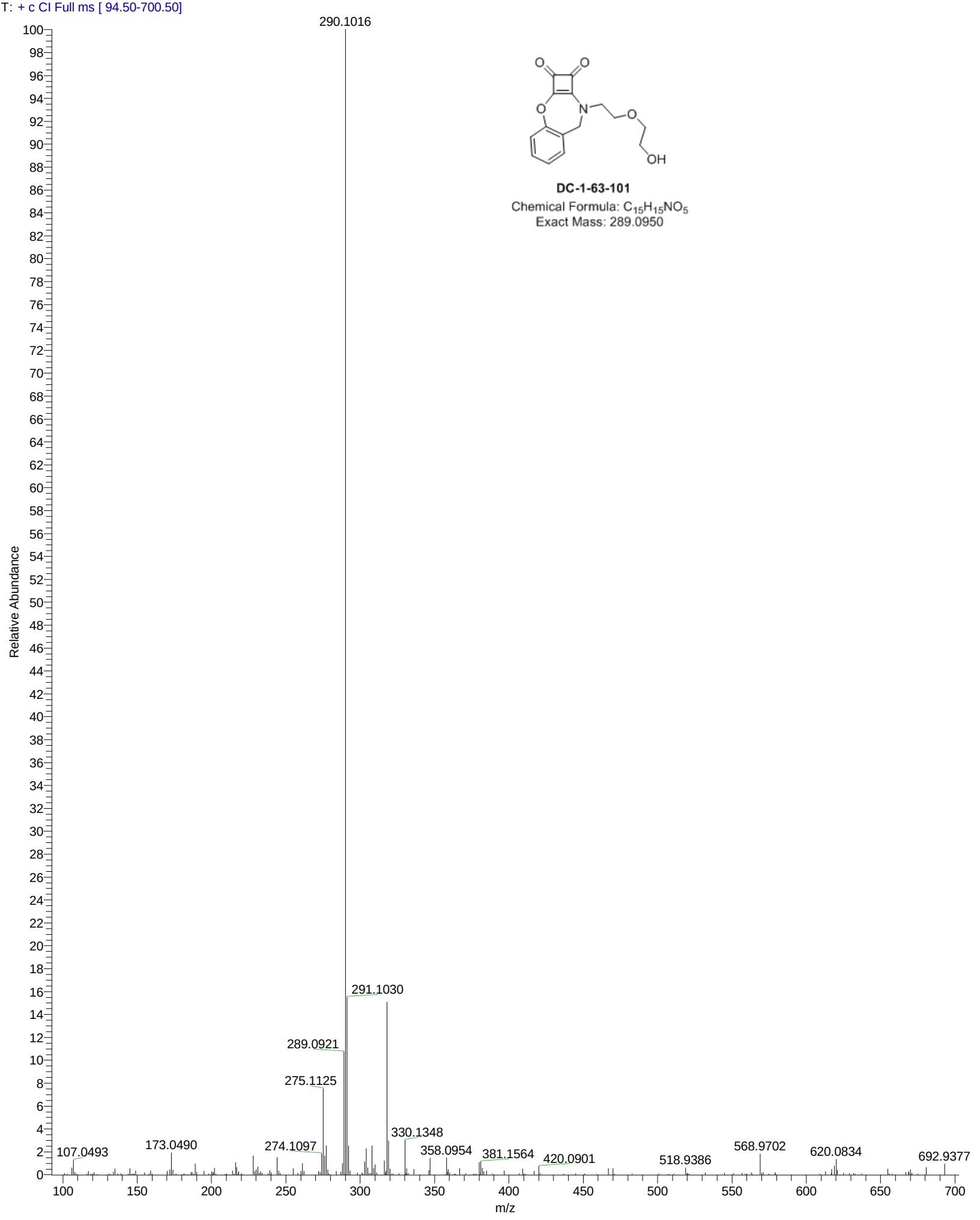


DC-2-61-101b




```
Current Data Parameters
NAME        DC-2-61-101b
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100216
Time               14.13
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT            DMSO
NS                   16
DS                    0
SWH            3591.954 Hz
FIDRES        0.109618 Hz
AQ            4.5613556 sec
RG                 80.6
DW              139.200 usec
DE               54.00 usec
TE               299.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 9.75 usec
PL1                0.00 dB
SFO1        300.1315007 MHz

F2 - Processing parameters
SI                32768
SF        300.1300042 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```

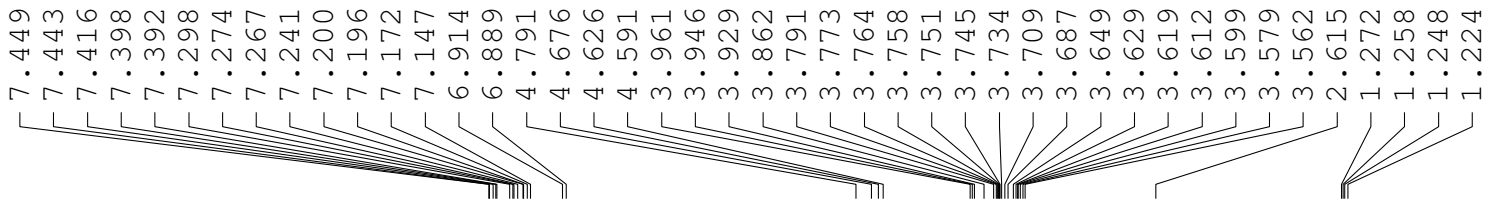
dc-2-63-101-c1 #88-91 RT: 4.14-4.28 AV: 4 NL: 3.19E7
T: + c CI Full ms [94.50-700.50]



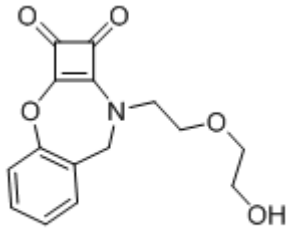
DC-1-63-101

Chemical Formula: $C_{15}H_{15}NO_5$

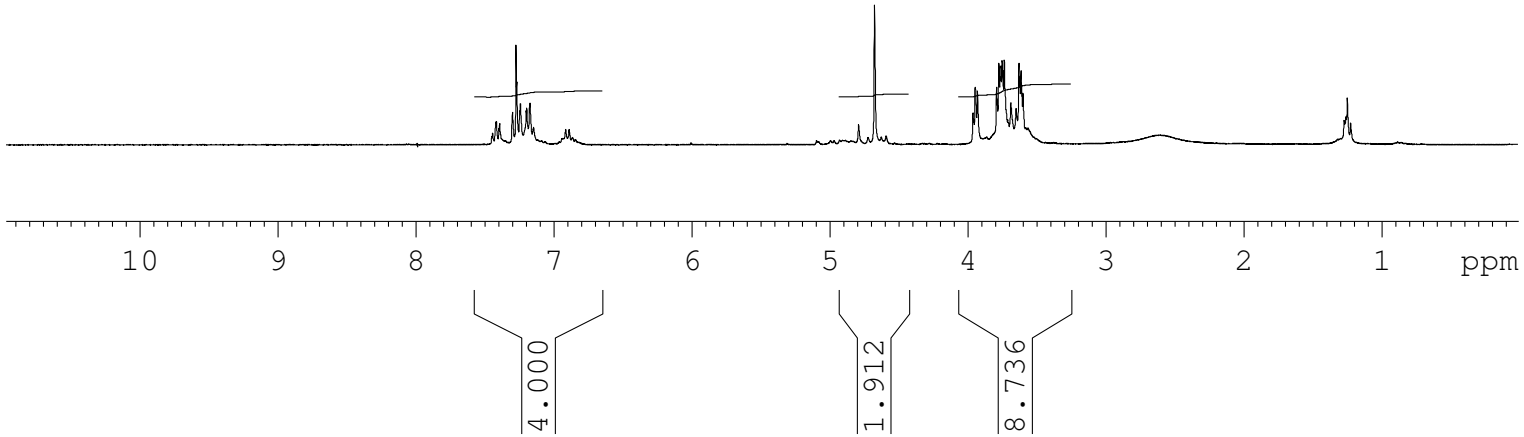
Exact Mass: 289.0950



dc-2-63-101b



dc-2-63-101b



Current Data Parameters
NAME dc−2−63−101b
EXPNO 1
PROCNO 1

F2 − Acquisition Parameters
Date_ 20100814
Time 10.55
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 362
DW 104.400 usec
DE 54.00 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ===
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1324000 MHz

F2 − Processing parameters
SI 32768
SF 300.1300024 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00