

Supporting Information

Silver(I)-Catalyzed Regioselective Synthesis of Triazole Fused-1,5-Benzoxazocinones

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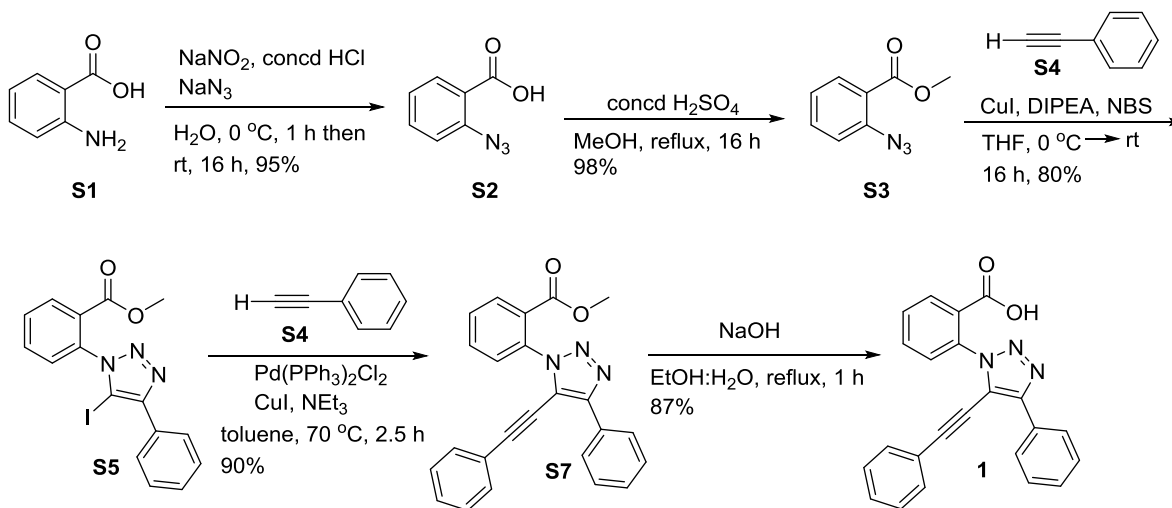
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General Methods

^1H NMR (400 MHz) and ^{13}C NMR (101 MHz) spectra were recorded on 400-MR automated spectrometer. Chemical shifts are reported in parts per million (ppm) on the δ scale from an internal standard (TMS). Analytical thin-layer chromatography (TLC) was performed using 0.25 mm silica gel-coated Kieselgel 60 F₂₅₄ plates. Flash chromatography was performed using the indicated solvent and silica gel 60 (Merck, 230-400 mesh). High-resolution mass spectra (HRMS) were recorded in ESI mode using TOF mass spectrometer. Microwave irradiation experiments were performed in a CEM Discover single-mode microwave reactor equipped with an IR temperature sensor using standard 10 mL CEM process vial sealed with Teflon[®] cap. All materials were purchased from commercial sources and used without further purification.

Experimental Procedures

General procedures for synthesis of 2-(4-phenyl-5-(phenylethynyl)-1*H*-1,2,3-triazol-1-yl)benzoic Acid (**1**)



Synthesis of 2-azido benzoic acid (S2): Compound **S2** was prepared according to literature method.¹

A suspension of anthranilic acid **S1** (3 g, 21.8 mmol) in water (15 mL) was cooled to $-5\text{ }^\circ\text{C}$, then conc. HCl (7.5 mL) and a solution of sodium nitrite (1.81 g, 26.2 mmol) in water (15 mL) was added dropwise. The above solution was stirred at $-5\text{ }^\circ\text{C}$ for 1 h and poured into a solution of sodium azide (1.7 g, 26.2 mmol) in water (25 mL) at $-5\text{ }^\circ\text{C}$. The resulting reaction mixture was stirred for 16 h. After completion of the reaction, the solid was filtered through Buchner funnel, washed

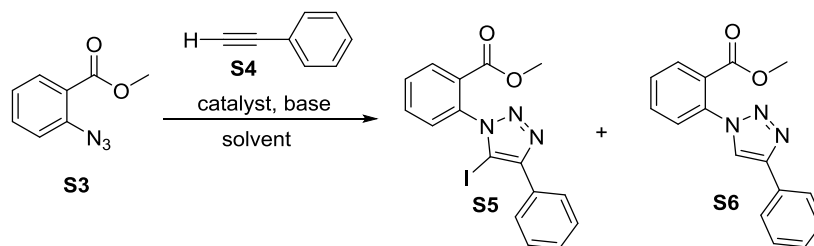
with water (200 mL) and dried to afford 2-azido benzoic acid **S2** as an off-white solid (3.4 g, 95%). ^1H NMR (400 MHz, Acetone- d_6) δ 11.35 (bs, 1H), 7.90 (d, J = 9.3 Hz, 1H), 7.63 (t, J = 8.5 Hz, 1H), 7.36 (d, J = 8.1 Hz, 1H), 7.28 (t, J = 7.6 Hz, 1H)

Synthesis of methyl 2-azidobenzoate (S3): Compound **S3** was prepared according to literature method.²

To a solution of 2-azido benzoic acid **S2** (3 g, 18.3 mmol) in methanol was added conc. H_2SO_4 (1.3 mL) and the reaction mixture was refluxed for 16 h. The solvent was evaporated; the crude reaction mixture was neutralized with sat. NaHCO_3 and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were washed with brine solution, dried over MgSO_4 and concentrated *in vacuo* to afford methyl 2-azidobenzoate **S3** as a brown oil (3.2 g, 98%). ^1H NMR (400 MHz, CDCl_3) δ 7.83 (dd, J = 7.8, 1.6 Hz, 1H), 7.53 – 7.47 (m, 1H), 7.22 (t, J = 7.7 Hz, 1H), 7.16 (t, J = 7.6 Hz, 1H), 3.88 (s, 3H).

Synthesis of methyl 2-(5-iodo-4-phenyl-1*H*-1,2,3-triazol-1-yl)benzoate (S5)

Optimization of the Reaction Conditions for the One-Pot Synthesis of 5-Iodo-1,2,3-Triazole Benzoate S5^a



entry	additives	base	solvent	temp (°C)	Time (h)	product	Yield (%) ^b
1	CuI/I ₂	-	THF	reflux	16	S5	Trace
2	CuI/I ₂	DIPEA	THF	reflux	16	N. R.	0
3	Cu(OTf) ₂ /NaI	NEt ₃	THF	rt	16	N. R.	0
4	Cu(OTf) ₂ /NaI	NEt ₃	THF	reflux	16	N. R.	0
5	CuI/NBS	DIPEA	THF	rt	16	S5 + S6	40:40
6	CuI/NBS	DIPEA	THF	0→rt	16	S5	80

^aReaction conditions: **S3** (0.56 mmol), **S4** (1 equiv), CuI (1 equiv), NBS (1.09 equiv), DIPEA (1 equiv), solvent (5 mL). N. R. = No Reaction. ^bIsolated yield.

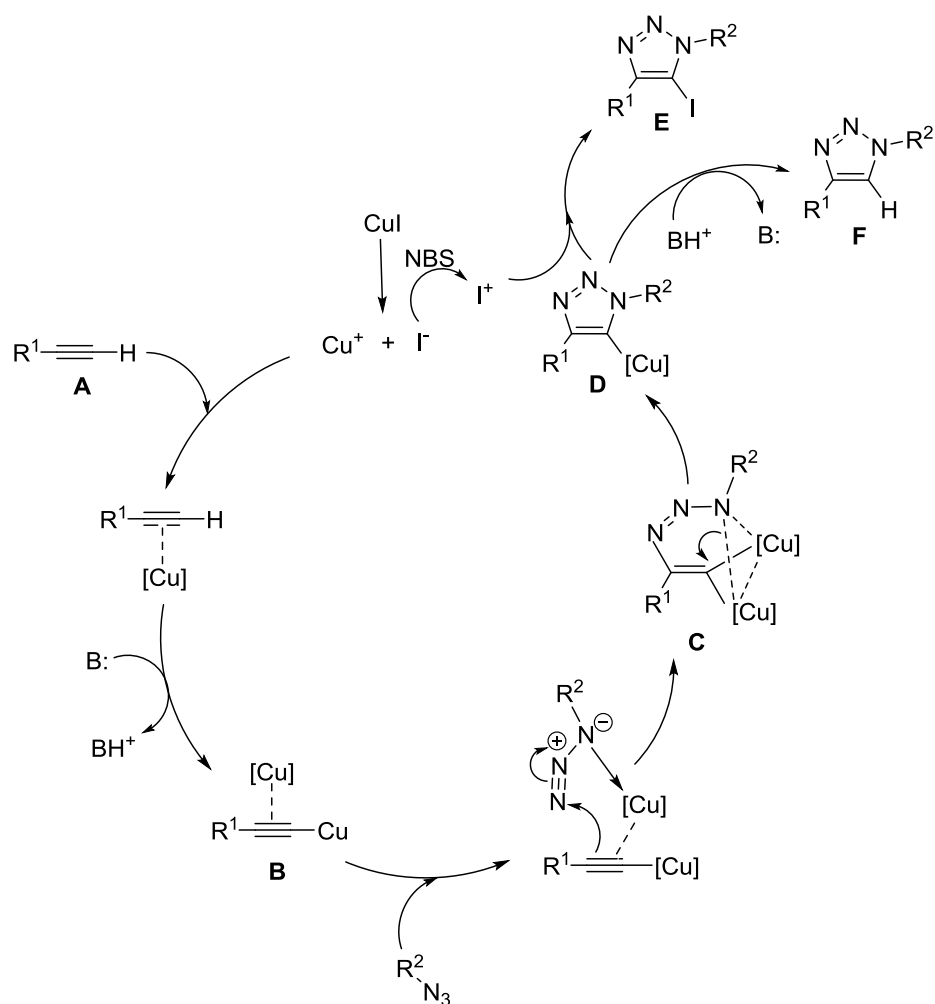
General procedure for the One-Pot Synthesis of 5-Iodo-1,2,3-Triazole Benzoate S5

A solution of NBS (1.09 g, 6.15 mmol) in THF (40 mL) was cooled to 0 °C and CuI (1.07 g, 5.64 mmol), methyl 2-azidobenzoate **S3** (1 g, 5.64 mmol), phenyl

acetylene **S4** (0.57 g, 5.64 mmol) and DIPEA (0.98 mL, 5.64 mmol) were added. The resulting reaction mixture was stirred at room temperature for 16 h. The solvent was evaporated; the reaction mixture was diluted with water (40 mL) and extracted with ethyl acetate (3 x 25 mL). The combined organic layers were washed with brine solution, dried over MgSO₄ and concentrated under reduced pressure. The crude was purified by flash column chromatography (50-70% ethyl acetate in hexanes) to yield methyl 2-(5-iodo-4-phenyl-1*H*-1,2,3-triazol-1-yl)benzoate **S5** as a white solid (1.82 g, 80%).

Plausible Mechanism

The plausible mechanism for CuI/NBS catalyzed 5-iodo-1,2,3-triazole formation is depicted in Scheme S1.^{3,4} In the first step, copper ion is coordinated to alkyne **A** and its deprotonation by base generates terminal copper acetylide **B**. Subsequently, the azide coordinates with dinuclear copper acetylide **B** to form complex **C**. The complex **C** then undergoes ring formation to afford copper triazolide **D**. Finally, trapping of either I⁺ or H⁺ ion by nucleophilic copper triazolide **D** provides either iodotriazole **E** or triazole **F**. The CuI provides Cu⁺ ions for cycloaddition reaction between alkyne and azide as well as I⁺ ions for iodation. The role of NBS is to oxidize I⁻ to I⁺.



Scheme S1. A plausible mechanism for CuI/NBS catalyzed iodotriazole formation

Synthesis of methyl 2-(4-phenyl-5-(phenylethynyl)-1*H*-1,2,3-triazol-1-yl)benzoate (S7)

To a mixture of methyl 2-(5-iodo-4-phenyl-1*H*-1,2,3-triazol-1-yl)benzoate **S5** (1 g, 2.46 mmol) in toluene was added phenyl acetylene **S4** (0.37 g, 3.7 mmol), Pd(PPh₃)₂Cl₂ (0.17 g, 0.24 mmol), CuI (0.093 g, 0.49 mmol) and triethyl amine (2 mL, 14.8 mmol) under nitrogen atmosphere and the reaction mixture was heated at 70 °C for 2.5 h. The solvent was evaporated, the crude reaction mixture was diluted with water (30 mL) and extracted with ethyl acetate (3 x 25 mL). The combined organic layers were washed with brine solution, dried over MgSO₄ and concentrated. The crude product was purified by flash column chromatography (5-7% ethyl acetate in hexanes) to obtain methyl 2-(4-phenyl-5-(phenylethynyl)-1*H*-1,2,3-triazol-1-yl)benzoate **S7** as a brown oil (0.8 g, 90%).

Synthesis of 2-(4-phenyl-5-(phenylethynyl)-1*H*-1,2,3-triazol-1-yl)benzoic acid (1)

To a mixture of methyl 2-(4-phenyl-5-(phenylethynyl)-1*H*-1,2,3-triazol-1-yl)benzoate **S7** (0.4 g, 1.05 mmol) in ethanol : H₂O (1:1; 10 mL) was added NaOH (0.4 g) and the reaction was refluxed for 1 h. The solvent was evaporated. The crude reaction mixture was neutralized with 1N HCl and extracted with ethyl

acetate (3 x 15 mL). The combined organic layers were washed with brine solution, dried over MgSO_4 and concentrated *in vacuo* to afford 2-(4-phenyl-5-(phenylethynyl)-1*H*-1,2,3-triazol-1-yl)benzoic acid **1** as an off-white solid (0.38 g, 98%).

Typical Procedure for the Synthesis of (Z)-3,5-diphenyl-7*H*-benzo[*c*][1,2,3]triazolo[1,5-*e*][1,5]oxazocin-7-one (2a)

A Biotage microwave vial (2-5 mL) was charged with 2-(4-phenyl-5-(phenylethynyl)-1*H*-1,2,3-triazol-1-yl)benzoic acid **1** (0.1 g 0.27 mmol), AgOTf (0.007 g, 0.027 mmol), K_2CO_3 (0.075 g, 0.54 mmol) and CHCl_3 (2 mL). The vial was sealed with a septum, and the mixture was irradiated with microwave (150 W) at 100 °C for 20 min. The reaction mixture was filtered through a thin bed of celite and the filtrate was concentrated under reduced pressure. The crude product was purified by flash column chromatography (10-12 % ethyl acetate in hexanes) to obtain (Z)-3,5-diphenyl-7*H*-benzo[*c*][1,2,3]triazolo[1,5-*e*][1,5]oxazocin-7-one **2a** as an off-white solid (0.087 g, 87%).

Representative procedure at 1 mmol scale for the synthesis of (Z)-3,5-diphenyl-7H-benzo[c][1,2,3]triazolo[1,5-e][1,5]oxazocin-7-one (2a)

A Biotage microwave vial (10-20 mL) was charged with 2-(4-phenyl-5-(phenylethynyl)-1H-1,2,3-triazol-1-yl)benzoic acid **1** (0.5 g 1.36 mmol), AgOTf (0.035 g, 0.13 mmol), K₂CO₃ (0.378 g, 2.73 mmol) and CHCl₃ (10 mL). The vial was sealed with a septum, and the mixture was irradiated with microwave (150 W) at 100 °C for 20 min. The reaction mixture was filtered through a thin bed of celite and the filtrate was concentrated under reduced pressure. The crude product was purified by flash column chromatography (10-12% ethyl acetate in hexanes) to obtain (Z)-3,5-diphenyl-7H-benzo[c][1,2,3]triazolo[1,5-e][1,5]oxazocin-7-one **2a** as an off-white solid (0.425 g, 85%).

References

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- (2) Alcaide, B.; Almendros, P.; Lázaro-Milla, C. *Chem. Commun.* **2015**, 51, 6992-6995.
- (3) Li, L.; Zhang, G.; Zhu, A.; Zhang, L. *J. Org. Chem.* **2008**, 73, 3630-3633.
- (4) Worrell, B. T.; Malik, J. A.; Fokin, V. V. *Science* **2013**, 340, 457-460.

Characterization data of compounds S5, S7, 1 and 2a-t

Methyl 2-(5-iodo-4-phenyl-1*H*-1,2,3-triazol-1-yl)benzoate (S5)

Brown solid (1.82 g, 80%); mp 160-162 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.16 (d, *J* = 7.6 Hz, 1H), 8.03 (d, *J* = 7.8 Hz, 2H), 7.73 (t, *J* = 7.5 Hz, 1H), 7.66 (t, *J* = 7.6 Hz, 1H), 7.50 – 7.37 (m, 4H), 3.65 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 164.4, 149.4, 136.3, 133.1, 131.7, 130.8, 130.0, 129.3, 128.6, 128.5, 128.5, 127.4, 79.9, 52.6; ; MS (ESI) *m/z*: 406.1 (M+H)⁺; HRMS (ESI) calcd for C₁₆H₁₃IN₃O₂ *m/z*: 406.0052; Found 406.0046 (M+H)⁺.

Methyl 2-(4-phenyl-5-(phenylethynyl)-1*H*-1,2,3-triazol-1-yl)benzoate (S7)

Brown oil (0.8 g, 90%); ¹H NMR (400 MHz, CDCl₃) δ 8.28 (d, *J* = 7.8 Hz, 2H), 8.13 (d, *J* = 8.0 Hz, 1H), 7.77 – 7.70 (m, 1H), 7.68 – 7.61 (m, 2H), 7.50 (t, *J* = 7.3 Hz, 2H), 7.44 – 7.31 (m, 6H), 3.68 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 165.4, 147.4, 135.1, 132.8, 131.4, 131.4, 130.2, 130.1, 129.6, 128.7, 128.7, 128.5, 128.0, 127.9, 126.3, 121.2, 118.91, 101.9, 75.4, 52.6; MS (ESI) *m/z*: 380.3 (M+H)⁺; HRMS (ESI) calcd for C₂₄H₁₈N₃O₂ *m/z*: 380.1399; Found 380.1399 (M+H)⁺.

2-(4-phenyl-5-(phenylethynyl)-1H-1,2,3-triazol-1-yl)benzoic acid (1)

Off-white solid (0.38 g, 98%); mp 163-165 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.21 (d, *J* = 7.6 Hz, 2H), 8.14 (d, *J* = 7.7 Hz, 1H), 7.72 (t, *J* = 7.4 Hz, 1H), 7.65 – 7.51 (m, 3H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.37 – 7.24 (m, 5H), 6.40 (s, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 168.1, 147.3, 135.4, 133.2, 132.1, 132.1, 132.0, 131.9, 131.5, 130.3, 130.0, 129.5, 128.6, 128.6, 128.5, 128.4, 128.2, 126.4, 121.2, 119.2, 102.0, 75.4; MS (ESI) *m/z*: 366.3 (M+H)⁺; HRMS (ESI) calcd for C₂₃H₁₆N₃O₂ *m/z*: 366.1242; Found 366.1242 (M+H)⁺.

(Z)-3,5-diphenyl-7H-benzo[*c*][1,2,3]triazolo[1,5-*e*][1,5]oxazocin-7-one (2a):

Off-white solid (87 mg, 87%); mp 167-169 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.92 (d, *J* = 7.4 Hz, 2H), 7.74 – 7.62 (m, 4H), 7.57 – 7.52 (m, 2H), 7.47 – 7.42 (m, 5H), 7.38 (t, *J* = 7.4 Hz, 1H), 6.73 (s, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 165.9, 154.1, 144.5, 133.6, 133.4, 132.2, 130.9, 130.3, 130.2, 130.1, 129.1, 128.9, 128.6, 128.4, 127.0, 126.7, 126.4, 125.5, 102.1; MS (ESI) *m/z*: 366.2 (M+H)⁺; HRMS (ESI) calcd for C₂₃H₁₆N₃O₂ *m/z*: 366.1243; Found 366.1239 (M+H)⁺.

(Z)-3-phenyl-5-propyl-7H-benzo[*c*][1,2,3]triazolo[1,5-*e*][1,5]oxazocin-7-one

(2b): White solid (75 mg, 75%); mp 130-132 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.88 (d, *J* = 7.8 Hz, 2H), 7.69 (t, *J* = 7.2 Hz, 2H), 7.64 – 7.58 (m, 1H), 7.53 (d, *J* = 7.9 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.36 (t, *J* = 7.4 Hz, 1H), 6.01 (s, 1H), 2.36 (t,

$J = 7.4$ Hz, 2H), 1.55 (sext, $J = 7.1$ Hz, 2H), 0.84 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 165.3, 158.3, 143.8, 133.1, 132.9, 131.0, 130.2, 129.6, 128.8, 128.4, 127.4, 127.1, 126.6, 126.5, 103.4, 36.9, 19.3, 13.2; HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{18}\text{N}_3\text{O}_2$ m/z : 332.1399; Found 332.1402 ($\text{M}+\text{H}$) $^+$.

(Z)-3-(4-methoxyphenyl)-5-phenyl-7H-benzo[c][1,2,3]triazolo[1,5-

e][1,5]oxazocin-7-one (2c): Yellow solid (69 mg, 69%); mp 79-81 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.86 (d, $J = 8.5$ Hz, 2H), 7.75 – 7.66 (m, 4H), 7.58 – 7.53 (m, 2H), 7.45 – 7.42 (m, 3H), 7.00 (d, $J = 8.5$ Hz, 2H), 6.71 (s, 1H), 3.85 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 165.9, 159.9, 153.9, 144.5, 133.7, 133.3, 132.3, 130.7, 130.2, 130.1, 129.1, 128.1, 127.0, 126.5, 125.5, 122.7, 114.4, 102.2, 55.3; HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{18}\text{N}_3\text{O}_3$ m/z : 396.1348; Found 396.1344 ($\text{M}+\text{H}$) $^+$.

(Z)-5-cyclohexyl-3-phenyl-7H-benzo[c][1,2,3]triazolo[1,5-e][1,5]oxazocin-7-

one (2d): Pale yellow solid (76 mg, 76%); mp 176-178 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.89 (d, $J = 7.2$ Hz, 2H), 7.72 (t, $J = 7.6$ Hz, 2H), 7.66 – 7.59 (m, 1H), 7.55 (d, $J = 8.0$ Hz, 1H), 7.47 (t, $J = 7.5$ Hz, 2H), 7.38 (t, $J = 7.4$ Hz, 1H), 6.00 (s, 1H), 2.40 – 2.30 (m, 1H), 1.93 – 1.78 (m, 4H), 1.75 – 1.67 (m, 1H), 1.33 – 1.25 (m, 5H); ^{13}C NMR (101 MHz, CDCl_3) δ 165.67, 162.69, 143.77, 133.10, 132.85, 130.32, 130.15, 129.89, 128.86, 128.41, 127.48, 126.56, 101.57, 43.39, 29.97,

25.65, 25.53; MS (ESI) m/z : 372.2 (M+H)⁺; HRMS (ESI) calcd for C₂₃H₂₂N₃O₂ m/z : 372.1712; Found 372.1712 (M+H)⁺.

(Z)-3-cyclohexyl-5-(thiophen-3-yl)-7H-benzo[c][1,2,3]triazolo[1,5-

e][1,5]oxazocin-7-one (2e): Brown solid (72 mg, 72%); mp 195-197 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.68 – 7.65 (m, 3H), 7.57 – 7.45 (m, 2H), 7.37 – 7.34 (m, 1H), 7.25 – 7.22 (m, 1H), 6.45 (s, 1H), 2.84 – 2.76 (m, 1H), 2.02 – 1.99 (m, 2H), 1.86 – 1.83 (m, 2H), 1.75 – 1.66 (m, 3H), 1.44 – 1.30 (m, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 166.1, 150.1, 149.4, 135.1, 134.0, 133.2, 130.2, 130.0, 127.7, 127.1, 126.3, 125.0, 124.2, 100.8, 35.7, 32.3, 26.3, 25.8; MS (ESI) m/z : 378.1(M+H)⁺; HRMS (ESI) calcd for C₂₁H₂₀N₃O₂S m/z : 378.1276; Found 378.1271 (M+H)⁺.

(Z)-5-phenyl-3-(thiophen-3-yl)-7H-benzo[c][1,2,3]triazolo[1,5-e][1,5]oxazocin-

7-one (2f): Pale yellow solid (71 mg, 71%); mp 184-186 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.76 – 7.61 (m, 6H), 7.55 (t, J = 8.5 Hz, 2H), 7.47 – 7.38 (m, 4H), 6.74 (s, 1H); ¹³C NMR (101 MHz, CDCl₃) δ 165.7, 154.3, 141.2, 133.5, 133.4, 132.2, 131.1, 130.9, 130.3, 130.2, 129.1, 127.9, 127.0, 126.6, 126.45, 126.1, 125.5, 122.6, 101.6; MS (ESI) m/z : 372.1 (M+H)⁺; HRMS (ESI) calcd for C₂₁H₁₄N₃O₂S m/z : 372.0807; Found 372.0803 (M+H)⁺.

(Z)-3,5-di(thiophen-3-yl)-7H-benzo[c][1,2,3]triazolo[1,5-e][1,5]oxazocin-7-one

(2g): Pale brown solid (75 mg, 75%); mp 154-156°C; ¹H NMR (400 MHz, CDCl₃)

δ 7.74 – 7.65 (m, 4H), 7.62 (d, J = 4.9 Hz, 1H), 7.54 (t, J = 8.0 Hz, 2H), 7.40 (dd, J = 4.8, 2.9 Hz, 1H), 7.38 – 7.34 (m, 1H), 7.26 (d, J = 3.4 Hz, 1H), 6.56 (s, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 165.7, 150.5, 141.0, 134.9, 133.6, 133.4, 131.1, 130.4, 130.3, 128.0, 127.7, 127.0, 126.6, 126.2, 126.1, 125.6, 124.3, 122.6, 100.6; MS (ESI) m/z : 378.1 ($\text{M}+\text{H}$) $^+$; HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{12}\text{N}_3\text{O}_2\text{S}_2$ m/z : 378.0371; Found 378.0364 ($\text{M}+\text{H}$) $^+$.

(Z)-5-cyclohexyl-3-(thiophen-3-yl)-7H-benzo[*c*][1,2,3]triazolo[1,5-

e][1,5]oxazocin-7-one (2h): Pale brown solid (70 mg, 70%); mp 203-205 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.72 – 7.66 (m, 2H), 7.64 – 7.58 (m, 2H), 7.52 (d, J = 7.9 Hz, 1H), 7.41 (dd, J = 5.0, 2.9 Hz, 1H), 5.97 (s, 1H), 2.40 – 2.26 (m, 1H), 1.93 – 1.74 (m, 4H), 1.69 (d, J = 12.3 Hz, 1H), 1.36 – 1.10 (m, 5H); ^{13}C NMR (101 MHz, CDCl_3) δ 165.5, 162.9, 140.5, 132.9, 132.8, 131.3, 130.1, 129.8, 127.4, 127.0, 126.4, 125.9, 122.1, 101.0, 43.3, 29.9, 25.6; MS (ESI) m/z : 378.2 ($\text{M}+\text{H}$) $^+$; HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{12}\text{N}_3\text{O}_2\text{S}_2$ m/z : 378.1276; Found 378.1270 ($\text{M}+\text{H}$) $^+$.

(Z)-3,5-diphenyl-7H-naphtho[2,3-*c*][1,2,3]triazolo[1,5-*e*][1,5]oxazocin-7-one

(2j): Yellow solid (77 mg, 77%); mp 224-226 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.24 (s, 1H), 8.05 (s, 1H), 7.96 – 7.92 (m, 4H), 7.70 – 7.62 (m, 4H), 7.50 – 7.38 (m, 6H), 6.76 (s, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 165.9, 154.1, 144.6, 134.5, 132.4, 132.3, 131.9, 130.8, 130.2, 130.0, 129.6, 129.1, 128.9, 128.7, 128.6, 128.5, 128.3,

126.7, 125.6, 124.0, 102.2; MS (ESI) m/z : 416.2 (M+H)⁺; HRMS (ESI) calcd for C₂₇H₁₇N₃O₂ m/z : 416.1399; Found 416.1394 (M+H)⁺.

(Z)-5-cyclohexyl-3-pentyl-7H-naphtho[2,3-c][1,2,3]triazolo[1,5-

e][1,5]oxazocin-7-one (2k): Yellow liquid (81 mg, 81%); ¹H NMR (400 MHz, CDCl₃) δ 8.23 (s, 1H), 8.04 – 7.91 (m, 3H), 7.72 – 7.62 (m, 2H), 5.83 (s, 1H), 2.73 (t, J = 7.7 Hz, 2H), 2.31 – 2.21 (m, 1H), 1.88 – 1.82 (m, 2H), 1.81 – 1.65 (m, 6H), 1.42 – 1.34 (m, 4H), 1.27 – 1.22 (m, 6H), 0.93 – 0.87 (m, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 166.0, 162.0, 145.4, 134.3, 132.4, 131.1, 130.1, 129.3, 128.6, 128.3, 125.9, 100.5, 43.4, 31.3, 29.9, 29.7, 28.7, 25.7, 25.5, 25.4, 22.4, 14.0; MS (ESI) m/z : 416.2 (M+H)⁺; HRMS (ESI) calcd for C₂₆H₃₀N₃O₂ m/z : 416.2338; Found 416.2338 (M+H)⁺.

(Z)-5-cyclohexyl-3-phenyl-7H-naphtho[2,3-c][1,2,3]triazolo[1,5-

e][1,5]oxazocin-7-one (2l): Pale yellow solid (76 mg, 76%); mp 211-213 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.27 (s, 1H), 8.05 – 8.01 (m, 2H), 7.99 – 7.94 (m, 1H), 7.91 (d, J = 7.8 Hz, 2H), 7.72 – 7.67 (m, 2H), 7.48 (t, J = 7.6 Hz, 2H), 7.39 (t, J = 7.4 Hz, 1H), 6.01 (s, 1H), 2.39 – 2.32 (m, 1H), 1.90 – 1.89 (m, 2H), 1.80 – 1.79 (m, 2H), 1.70 – 1.67 (m, 2H), 1.34 – 1.23 (m, 4H); ¹³C NMR (101 MHz, CDCl₃) δ 165.7, 162.5, 143.8, 134.2, 132.5, 131.2, 130.4, 129.7, 129.4, 128.8, 128.6, 128.5, 128.3, 127.7, 126.5, 126.1, 125.2, 101.6, 43.4, 29.8, 25.6, 25.5; MS (ESI) m/z :

442.4 (M+H)⁺; HRMS (ESI) calcd for C₂₇H₂₃N₃O₂ *m/z*: 422.1869; Found 422.1873 (M+H)⁺.

(Z)-5-butyl-3-phenyl-7*H*-naphtho[2,3-*c*][1,2,3]triazolo[1,5-*e*][1,5]oxazocin-7-

one (2m): Yellow liquid (75 mg, 75%); ¹H NMR (400 MHz, CDCl₃) δ 8.28 (s, 1H), 8.06 – 7.90 (m, 5H), 7.75 – 7.67 (m, 2H), 7.54 – 7.46 (m, 2H), 7.42 – 7.38 (m, 1H), 6.03 (s, 1H), 2.40 (t, *J* = 7.5 Hz, 2H), 1.30 – 1.21 (m, 4H), 0.86 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 165.3, 158.4, 143.8, 134.3, 132.5, 131.0, 130.3, 129.8, 129.4, 128.9, 128.6, 128.4, 127.7, 126.6, 126.2, 124.9, 103.3, 34.8, 27.9, 21.9, 13.5; MS (ESI) *m/z*: 396.3 (M+H)⁺; HRMS (ESI) calcd for C₂₅H₂₁N₃O₂ *m/z*: 396.1712; Found 396.1600 (M+H)⁺.

(Z)-9-methyl-5-pentyl-3-phenyl-7*H*-benzo[*c*][1,2,3]triazolo[1,5-

***e*][1,5]oxazocin-7-one (2n):** Yellow solid (78 mg, 78%); mp 149-151 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.89 (d, *J* = 7.5 Hz, 2H), 7.51 – 7.42 (m, 5H), 7.37 (t, *J* = 7.3 Hz, 1H), 6.01 (s, 1H), 2.48 (s, 3H), 2.40 (t, *J* = 7.5 Hz, 2H), 1.55 (p, *J* = 7.3 Hz, 2H), 1.27 (p, *J* = 7.2 Hz, 2H), 1.18 (p, *J* = 7.6 Hz, 2H), 0.85 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 165.5, 158.4, 143.6, 140.8, 133.6, 130.8, 130.3, 129.8, 128.8, 128.3, 127.4, 126.8, 126.5, 103.4, 35.1, 30.8, 25.6, 22.2, 21.0, 13.8; MS (ESI) *m/z*: 374.2 (M+H)⁺; HRMS (ESI) calcd for C₂₃H₂₃N₃O₂ *m/z*: 374.1869; Found 374.1870 (M+H)⁺.

(Z)-5-cyclohexyl-9-methyl-3-phenyl-7H-benzo[c][1,2,3]triazolo[1,5-

e][1,5]oxazocin-7-one (2o): Yellow solid (75 mg, 75%); mp 201-203 °C ¹H NMR (400 MHz, CDCl₃) δ 7.88 (d, *J* = 7.5 Hz, 2H), 7.53 – 7.41 (m, 5H), 7.37 (t, *J* = 7.6 Hz, 1H), 5.99 (s, 1H), 2.50 (s, 3H), 2.37 – 2.30 (m, 1H), 1.89 – 1.81 (m, 5H), 1.33 – 1.20 (m, 5H); ¹³C NMR (101 MHz, CDCl₃) δ 165.9, 162.6, 140.8, 133.6, 130.7, 130.3, 130.1, 128.8, 128.3, 127.5, 127.0, 126.5, 126.4, 101.6, 43.4, 29.9, 25.6, 25.5, 21.1; MS (ESI) *m/z*: 386.2 (M+H)⁺; HRMS (ESI) calcd for C₂₄H₂₃N₃O₂ *m/z* : 386.1869; Found 386.1875 (M+H)⁺.

(Z)-3-(4-methoxyphenyl)-9-methyl-5-phenyl-7H-benzo[c][1,2,3]triazolo[1,5-e][1,5]oxazocin-7-one (2p):

Pale yellow solid (68 mg, 68%); 149-151 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, *J* = 8.8 Hz, 2H), 7.68 – 7.66 (m, 2H), 7.48 – 7.44 (m, 5H), 7.00 (d, *J* = 8.7 Hz, 2H), 6.72 (s, 1H), 3.85 (s, 3H), 2.41 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 166.1, 159.9, 153.8, 144.3, 140.8, 134.1, 132.5, 131.3, 130.7, 130.4, 129.0, 128.1, 127.5, 126.9, 126.0, 125.5, 122.7, 114.3, 102.3, 55.3, 21.0; MS (ESI) *m/z*: 410.2 (M+H)⁺; HRMS (ESI) calcd for C₂₅H₁₉N₃O₃ *m/z*: 410.1505; Found 410.1505 (M+H)⁺.

(Z)-9-methyl-3-phenyl-5-(4-(trifluoromethyl)phenyl)-7H-

benzo[c][1,2,3]triazolo[1,5-e][1,5]oxazocin-7-one (2q): Yellow solid (71 mg, 71%); mp 242-244 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.68 (d, *J* = 8.3 Hz, 1H),

8.23 (s, 1H), 7.78 (d, $J = 7.8$ Hz, 1H), 7.33 – 7.17 (m, 5H), 7.09 – 7.02 (s, 5H), 2.61 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 195.1, 163.1, 141.0, 137.9, 135.8, 133.6, 133.3, 132.6, 132.1, 128.8, 128.4, 128.3, 127.6, 125.5, 124.9 (q, $J = 3.6$ Hz), 117.8, 116.6, 102.3, 21.4; HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{17}\text{F}_3\text{N}_3\text{O}_2$ m/z : 448.1273; Found 448.1274 ($\text{M}+\text{H}$) $^+$.

(Z)-9-methyl-3-phenyl-5-(thiophen-3-yl)-7H-benzo[c][1,2,3]triazolo[1,5-*e*][1,5]oxazocin-7-one (2r): Brown solid (73 mg, 73%); mp 75-77 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.92 (d, $J = 7.5$ Hz, 2H), 7.72 (s, 1H), 7.52 – 7.42 (m, 5H), 7.41 – 7.34 (m, 2H), 7.27 (d, $J = 5.3$ Hz, 1H), 6.57 (s, 1H), 2.42 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 166.1, 150.3, 144.2, 135.1, 134.2, 131.3, 130.6, 130.2, 128.9, 128.5, 128.1, 127.8, 126.9, 126.7, 125.8, 125.4, 124.3, 101.3, 21.0; MS (ESI) m/z : 386.1 ($\text{M}+\text{H}$) $^+$; HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{16}\text{N}_3\text{O}_2\text{S}$ m/z : 386.0963; Found 386.0958 ($\text{M}+\text{H}$) $^+$.

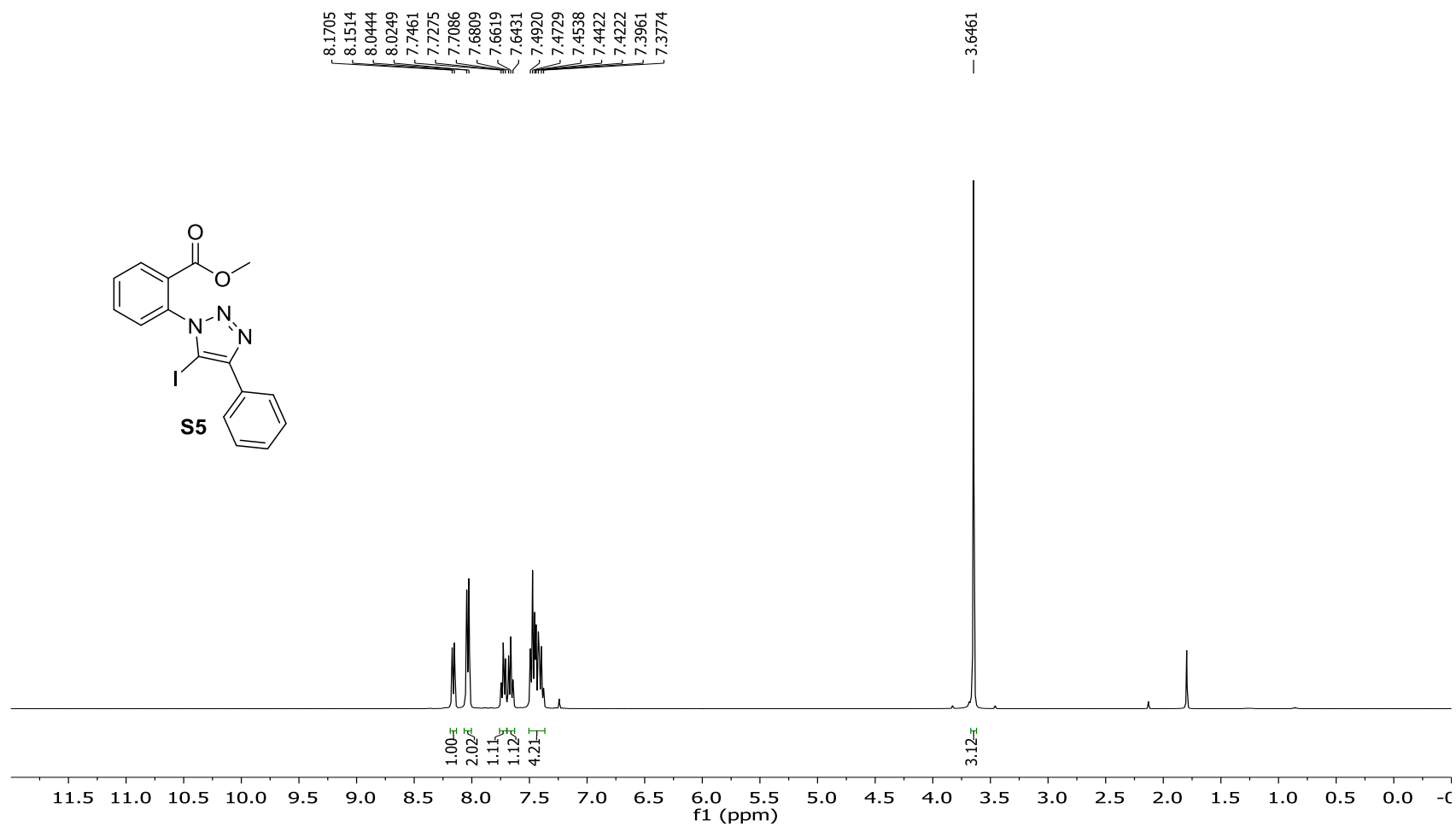
(Z)-10-chloro-5-pentyl-3-phenyl-7H-benzo[c][1,2,3]triazolo[1,5-*e*][1,5]oxazocin-7-one (2t): Yellow solid (74 mg, 74%); mp 151-153 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.87 (d, $J = 6.3$ Hz, 1H), 7.71 – 7.55 (m, 4H), 7.48 – 7.44 (m, 2H), 7.41 – 7.35 (m, 1H), 6.04 (s, 1H), 2.40 (t, $J = 6.4$ Hz, 2H), 1.30 – 1.22 (m, 6H), 0.87 – 0.84 (m, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 164.5, 158.7, 143.9, 139.1, 134.1, 131.0, 130.5, 129.9, 128.9, 128.6, 127.4, 127.0, 126.6, 125.9, 125.5,

103.3, 35.0, 30.9, 25.7, 22.2, 13.9; MS (ESI) m/z : 394.2 (M+H)⁺; HRMS (ESI)

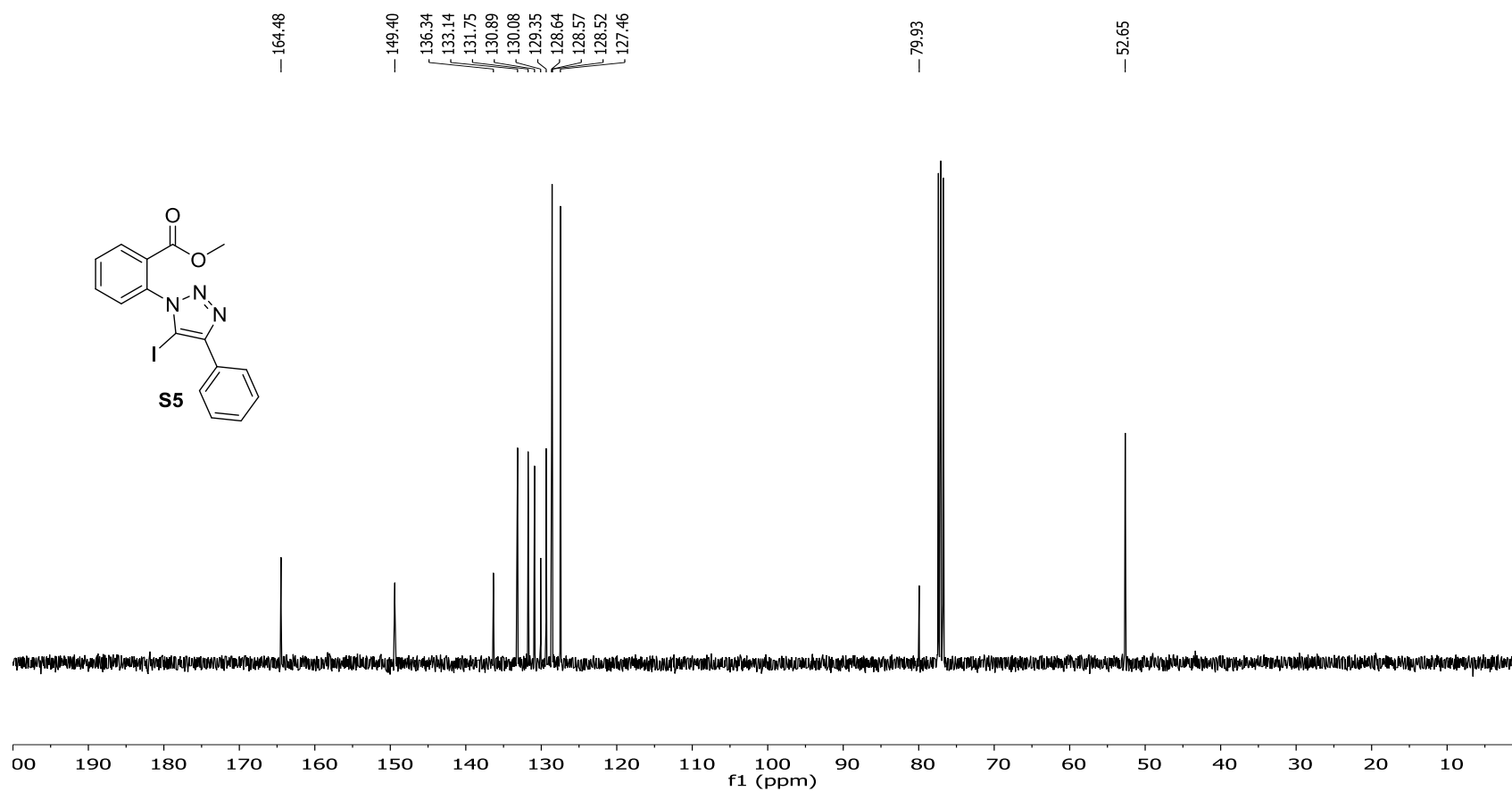
calcd for C₂₂H₂₁ClN₃O₂ m/z : 394.1322; Found 394.1319 (M+H)⁺.

^1H , ^{13}C NMR, LRMS and HRMS Spectra of
compounds **S5**, **S7**, **1** and **2a-t**

^1H , ^{13}C , LRMS and HRMS Spectra of compounds S5, S7, 1, 2a-t



^1H NMR Spectrum (400 MHz) of compound S5 in CDCl_3

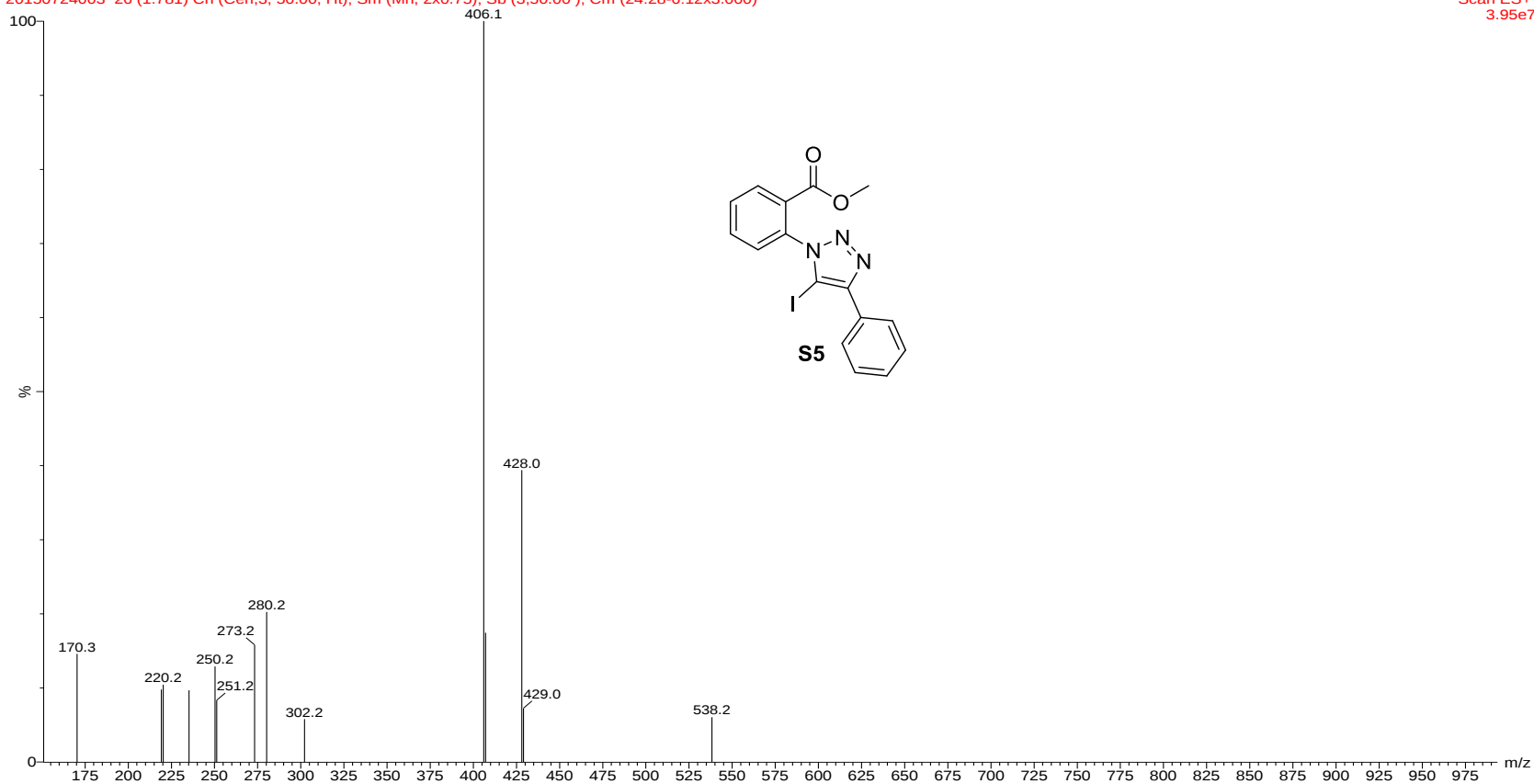


¹³C NMR Spectrum (101 MHz) of compound S5 in CDCl₃

ib-N9-059

20150724003 26 (1.781) Cn (Cen,3, 50.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (24:28-6:12x3.000)

Scan ES+
3.95e7

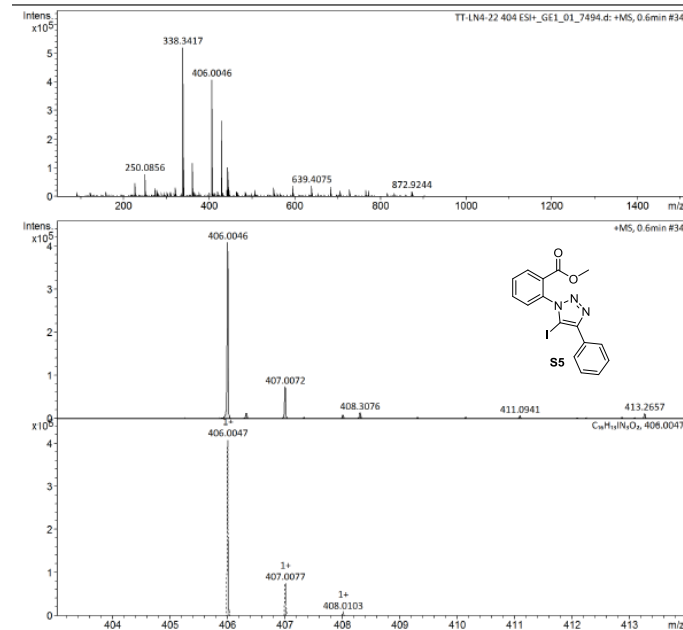


LRMS of compound S5

Display Report

Analysis Info		Acquisition Date	9/30/2015 11:07:54 AM
Analysis Name	D:\Data\ncnu service\data\2015\20150930\TT-LN4-22 404 ESI+_GE1_01_7494.d	Operator	NCTU
Method	Small molecule.m	Instrument	Impact HD
Sample Name	TT-LN4-22 404 ESI+		1819090.00164
Comment			

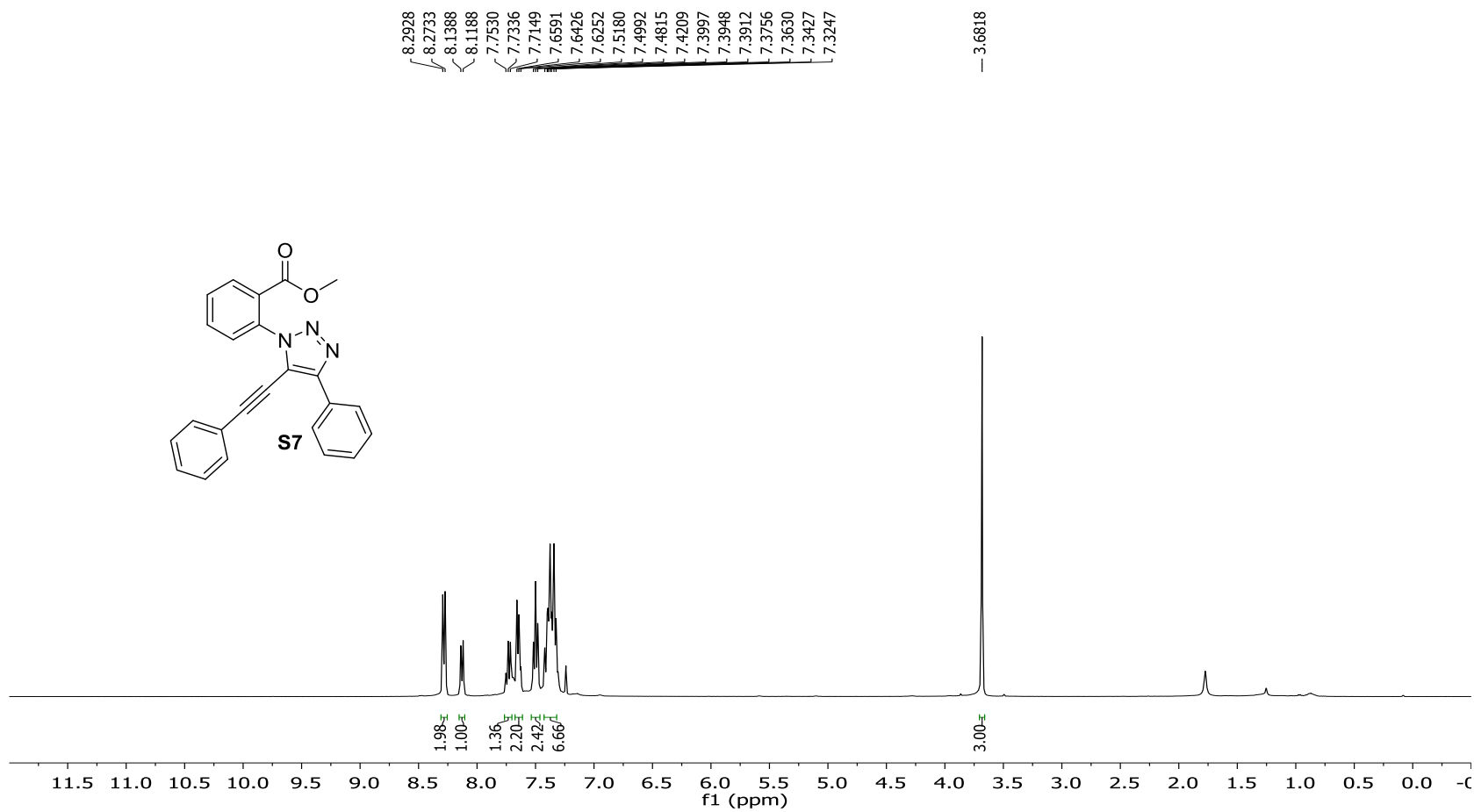
Acquisition Parameter			
Source Type	ESI	Ion Polarity	Positive
Focus	Active	Set Capillary	4500 V
Scan Begin	50 m/z	Set End Plate Offset	-500 V
Scan End	1500 m/z	Set Charging Voltage	2000 V
		Set Corona	0 nA
		Set Nebulizer	1.0 Bar
		Set Dry Heater	200 °C
		Set Dry Gas	6.0 l/min
		Set Divert Valve	Waste
		Set APCI Heater	0 °C



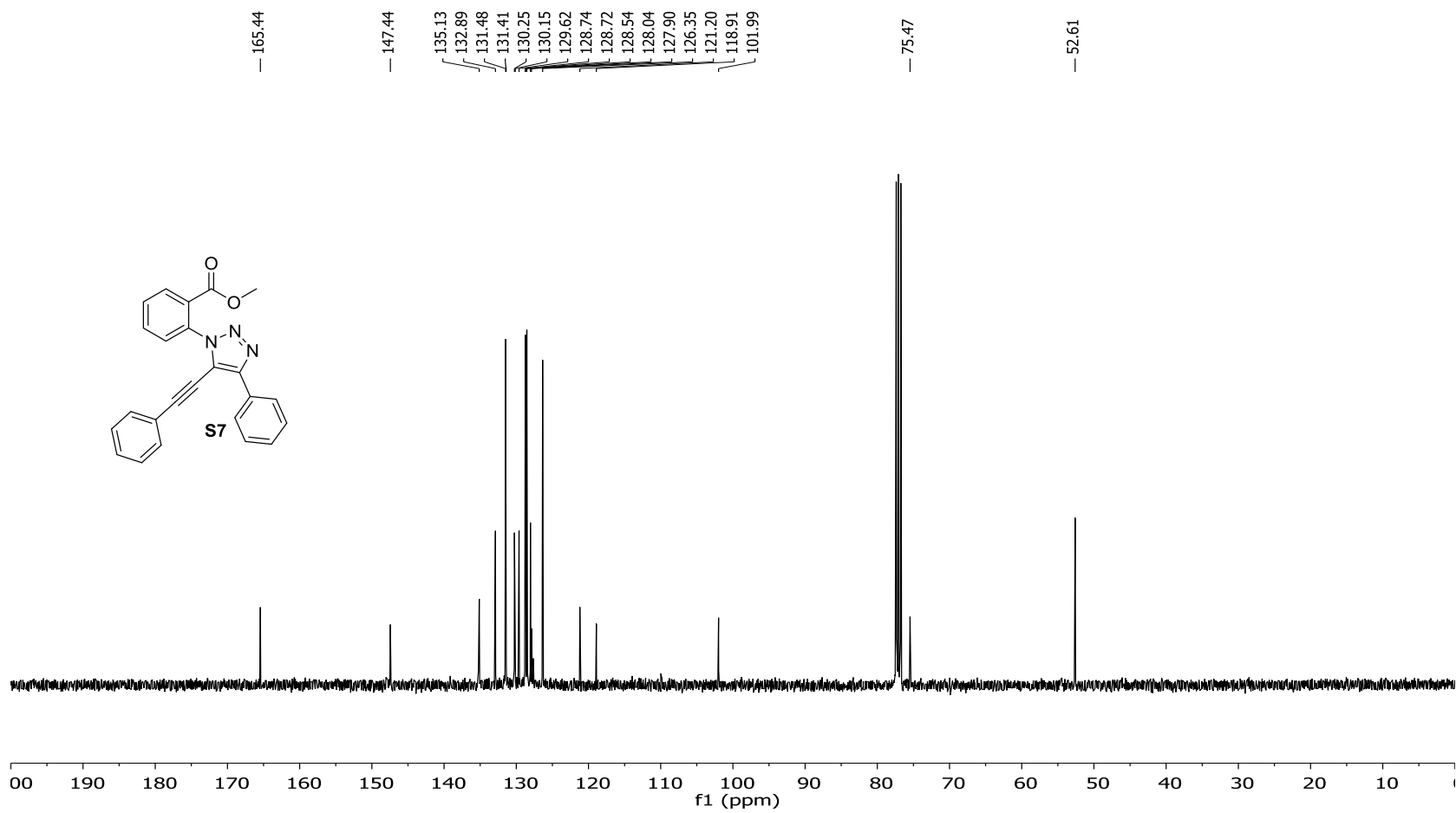
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
406.0046	1	C ₁₆ H ₁₃ IN ₃ O ₂	406.0047	-0.2	4.0	1	100.00	11.5	even	ok	M+H

HRMS of compound S5



¹H NMR Spectrum (400 MHz) of compound **S7** in CDCl₃

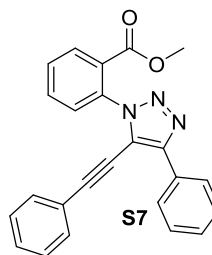
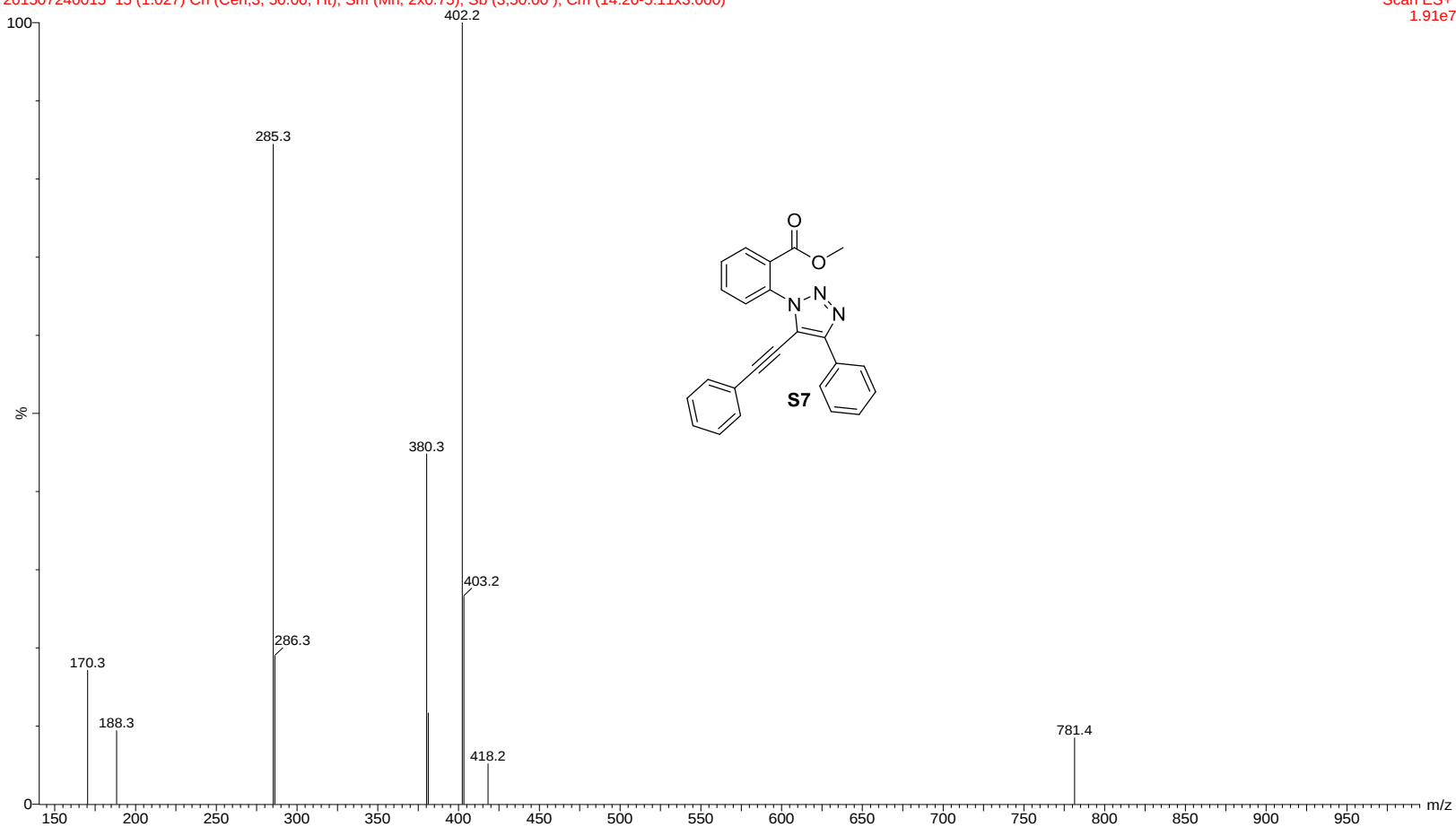


¹³C NMR Spectrum (101 MHz) of compound S7 in CDCl₃

ib-N9-061

201507240015 15 (1.027) Cn (Cen,3, 50.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (14:20-5:11x3.000)

Scan ES+
1.91e7

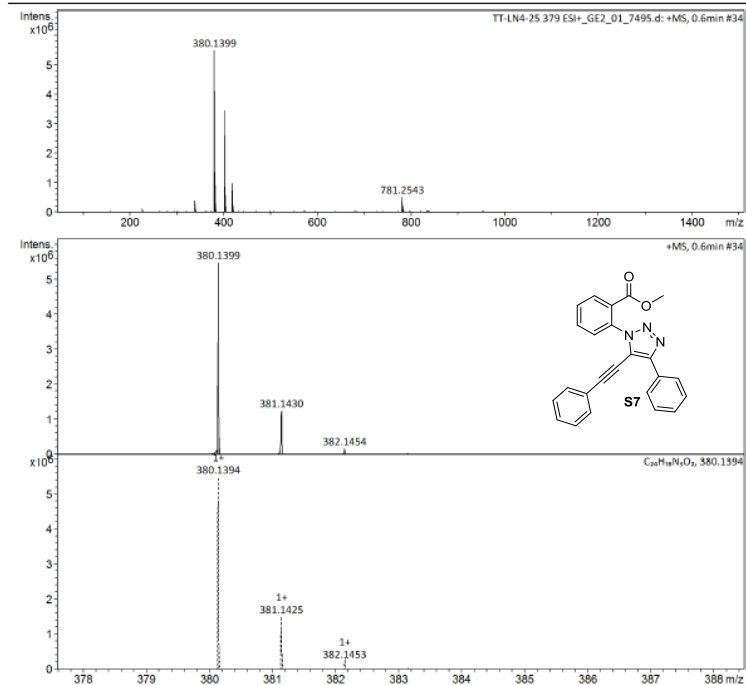


LRMS of compound S7

Display Report

Analysis Info		Acquisition Date	9/30/2015 11:12:09 AM	
Analysis Name	D:\Data\ncnu service\data\2015\20150930\TT-LN4-25 379 ESI+_GE2_01_7496.d			
Method	Small molecule.m		Operator NCTU	
Sample Name	TT-LN4-25 379 ESI+		Instrument	Impact HD 1819090.00164
Comment				

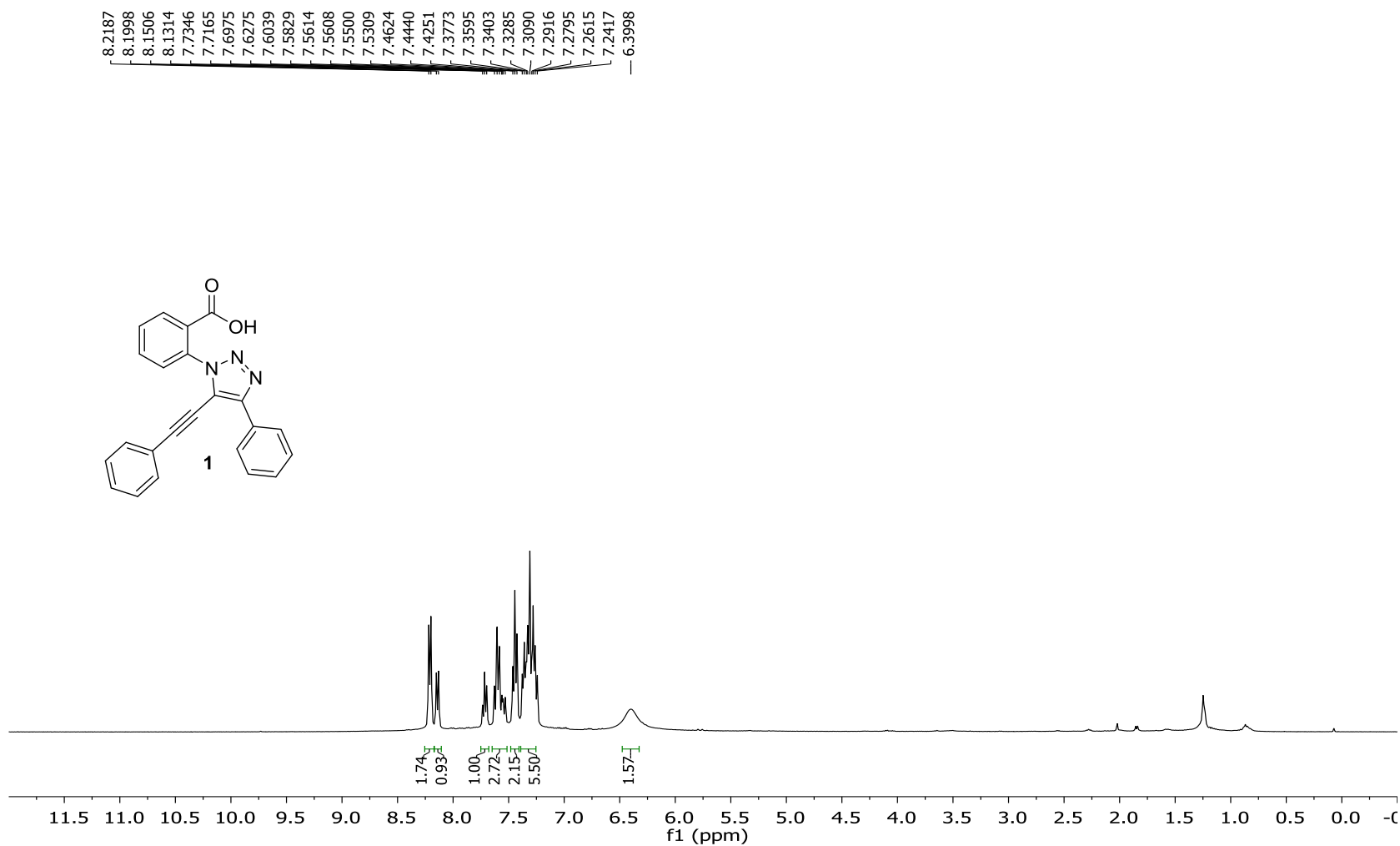
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

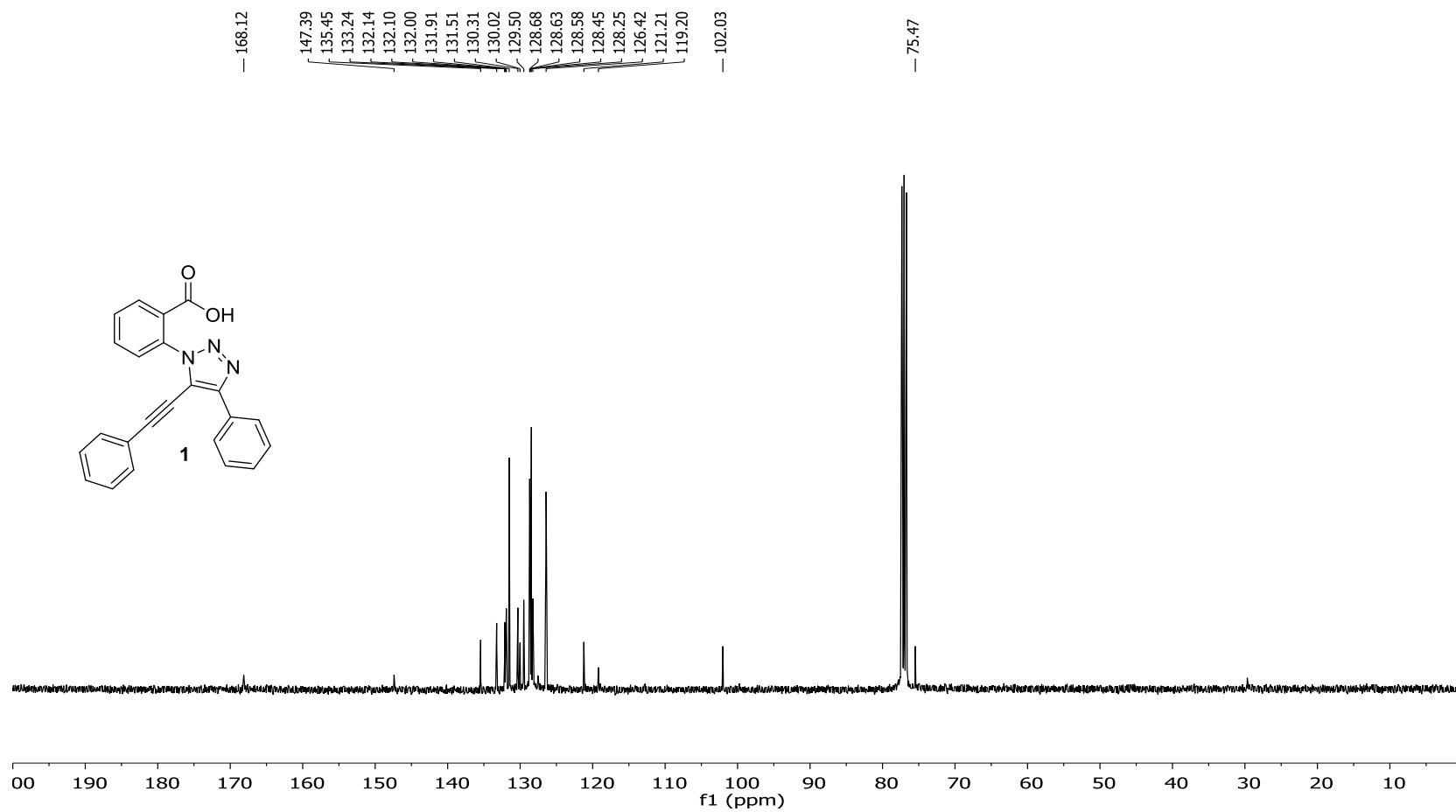


Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
380.1399	1	C ₂₄ H ₁₈ N ₃ O ₂	380.1394	-1.4	24.2	1	100.00	17.5	even	ok	M+H

HRMS of compound S7



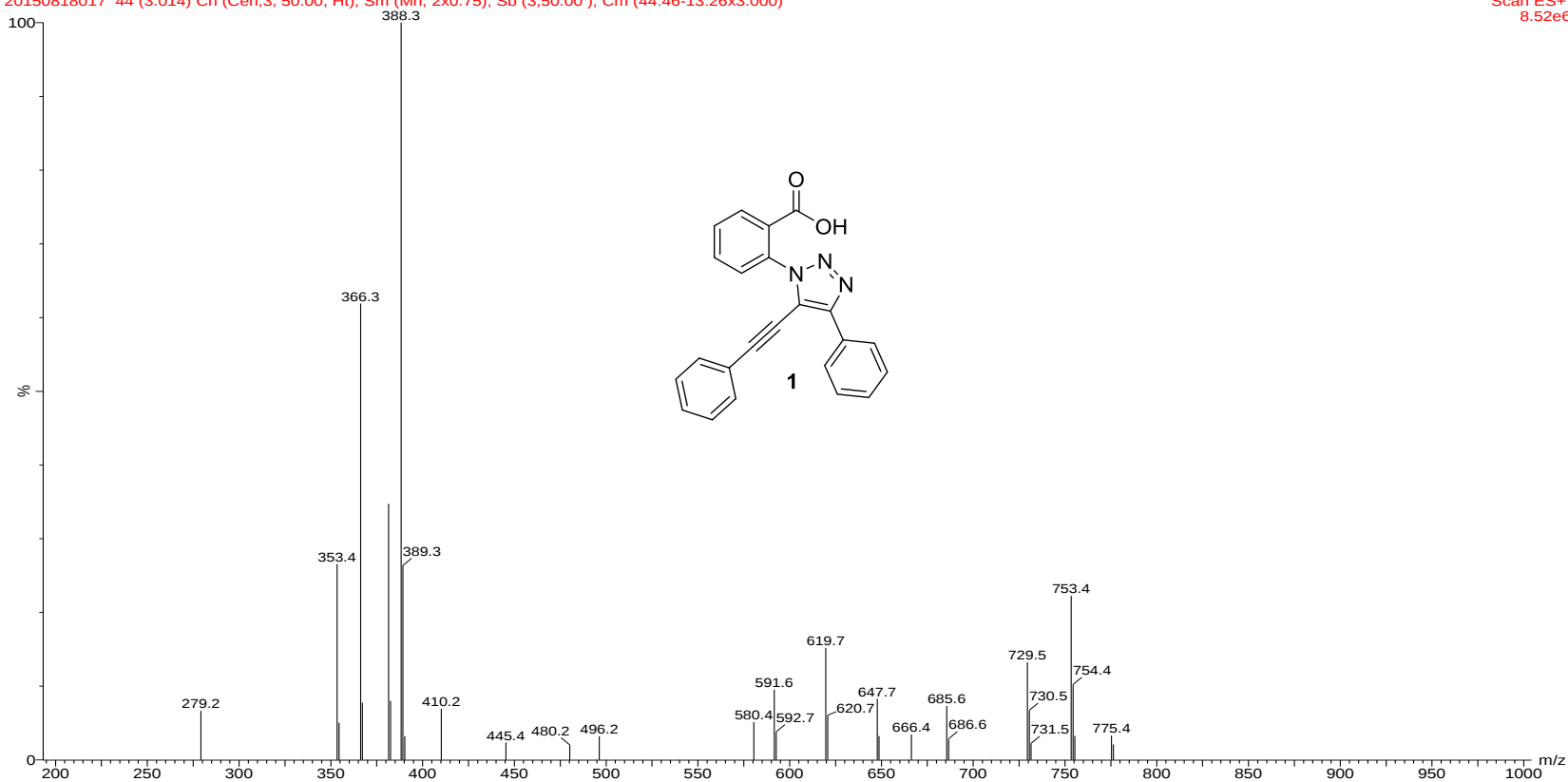


¹³C NMR Spectrum (101 MHz) of compound **1** in CDCl₃

ib-N9-082

20150818017 44 (3.014) Cn (Cen,3, 50.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (44:46-13:26x3.000)

Scan ES+
8.52e6

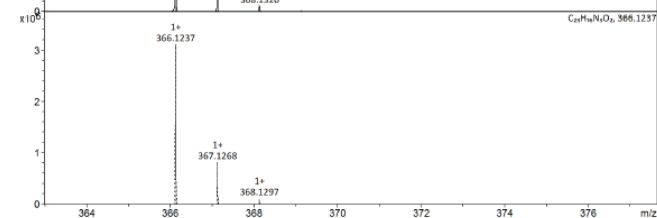
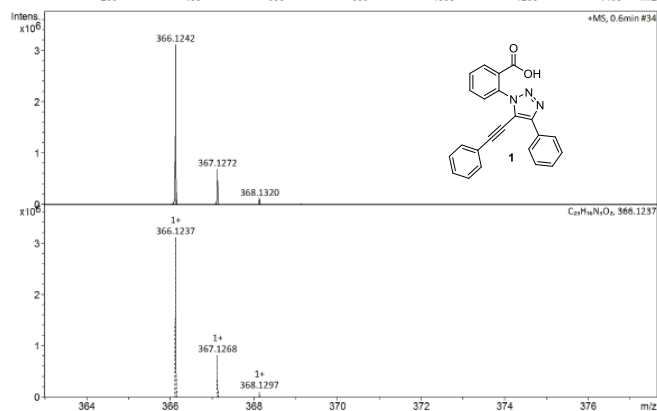
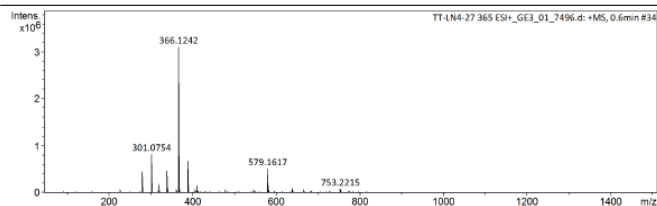


LRMS of compound 1

Display Report

Analysis Info		Acquisition Date	9/30/2015 11:16:26 AM	
Analysis Name	D:\Data\ncu service\data\2015\20150930\TT-LN4-27 365 ESI+-GE3_01_7496.d	Operator	NCTU	
Method	Small molecule.m	Instrument	impact HD	
Sample Name	TT-LN4-27 365 ESI+		1819696.00164	
Comment				

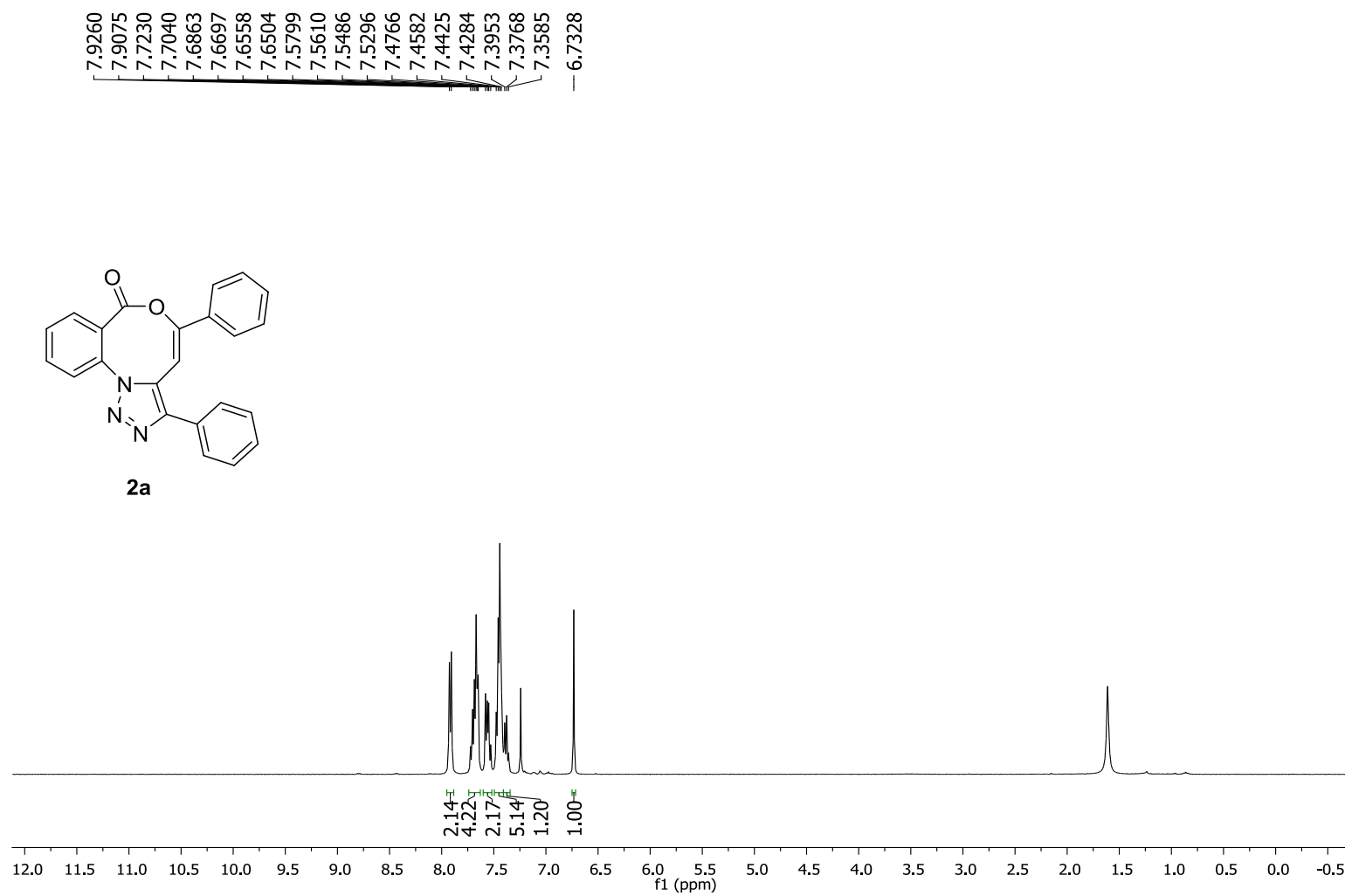
Acquisition Parameter					
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



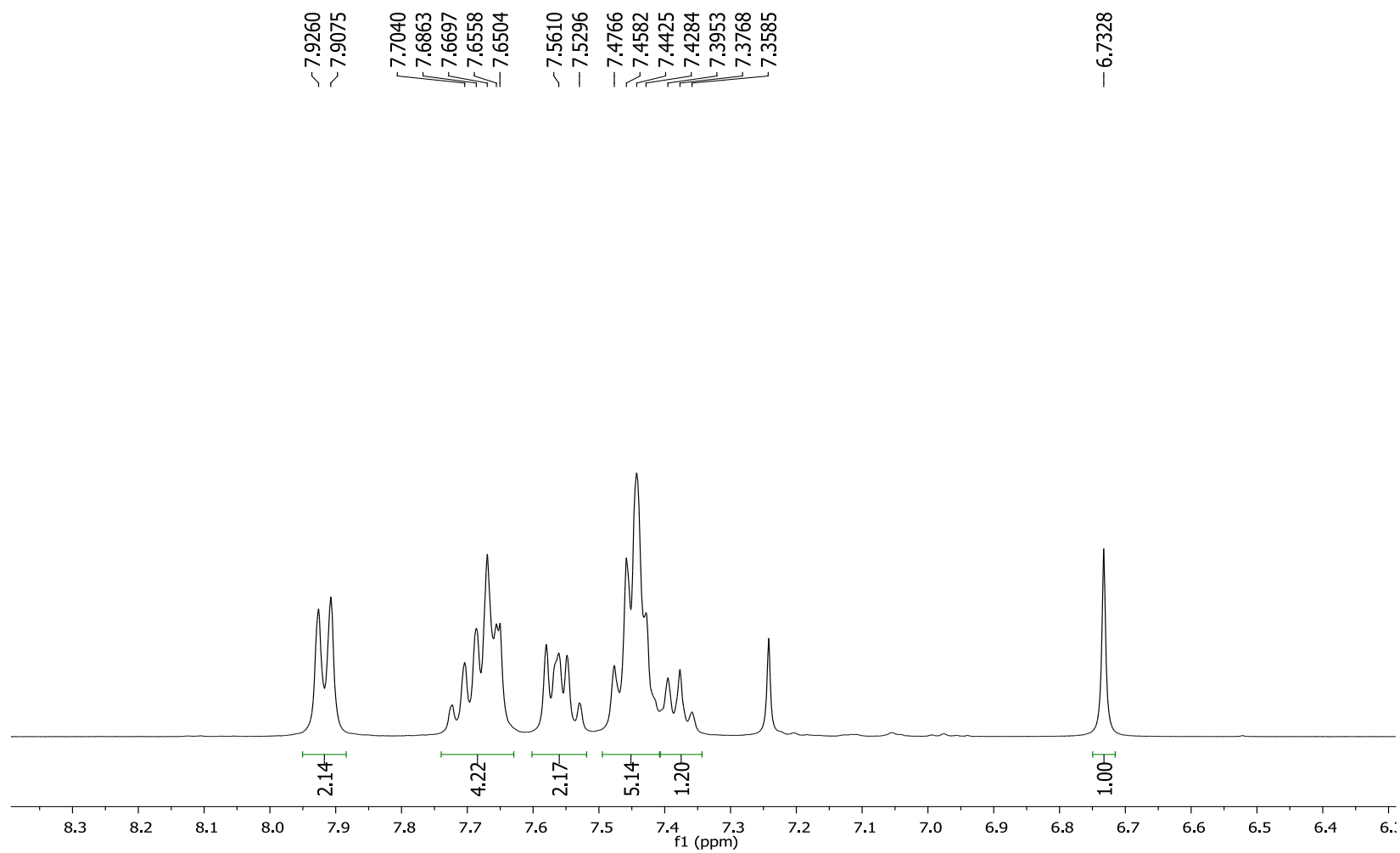
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
366.1242	1	C23H16N3O2	366.1237	1.4	21.4	1	100.00	17.5	even	ok	M+H

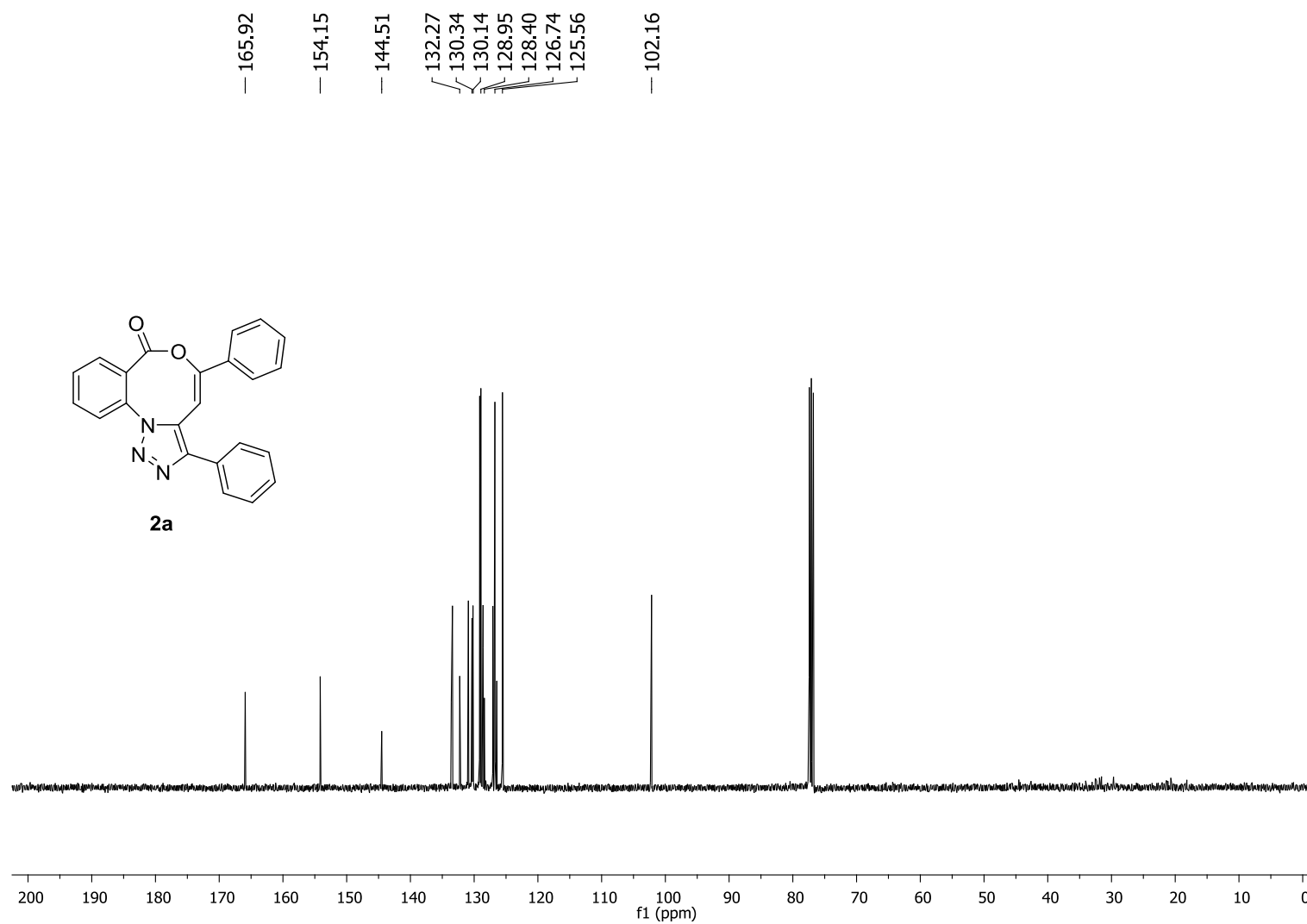
HRMS of compound 1

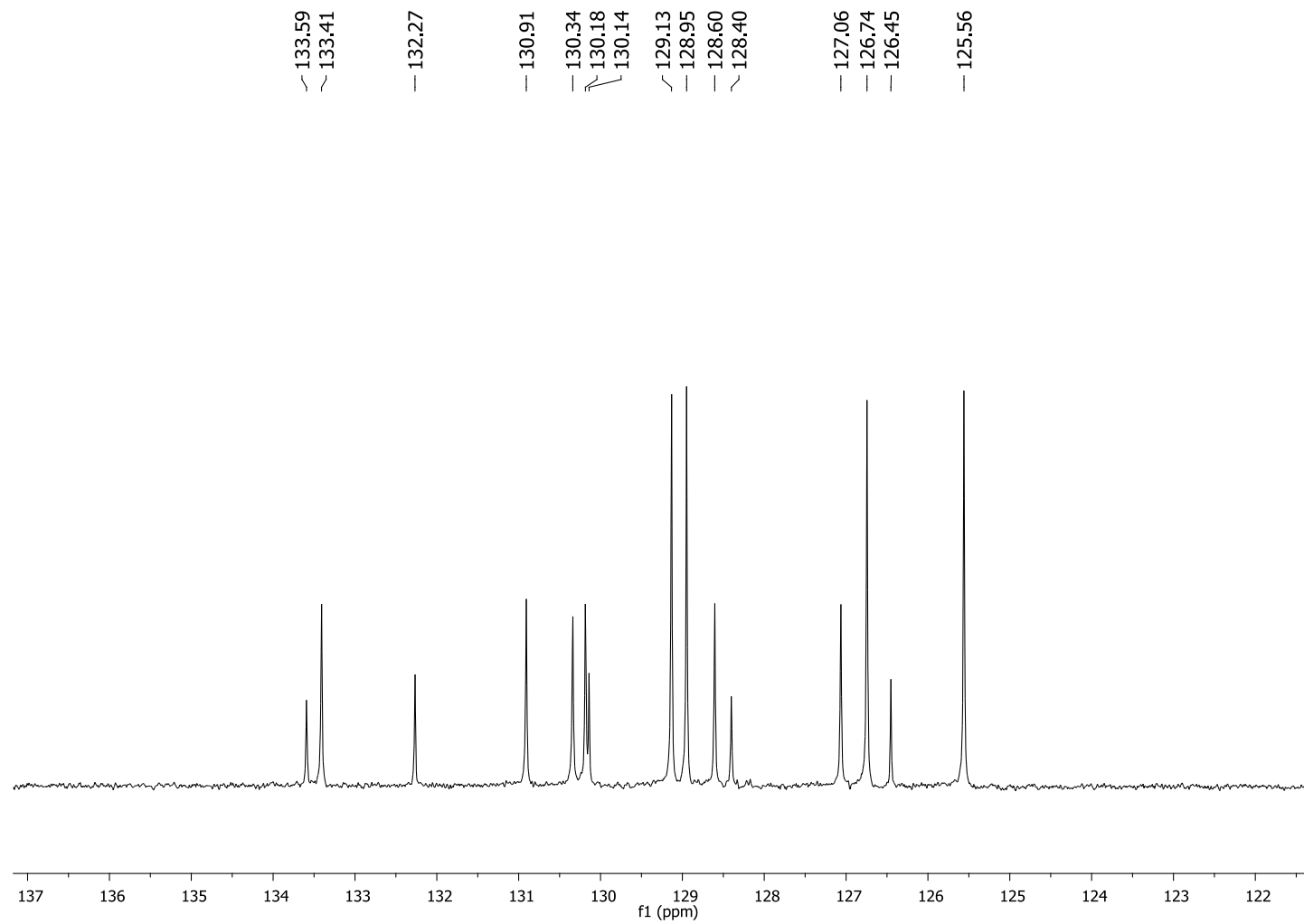


¹H NMR Spectrum (400 MHz) of compound **2a** in CDCl₃



Expansion of ^1H NMR Spectrum (400 MHz) of compound **2a** in CDCl_3



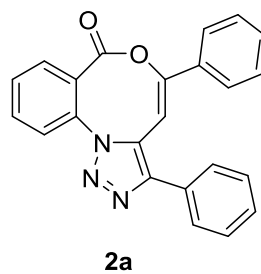
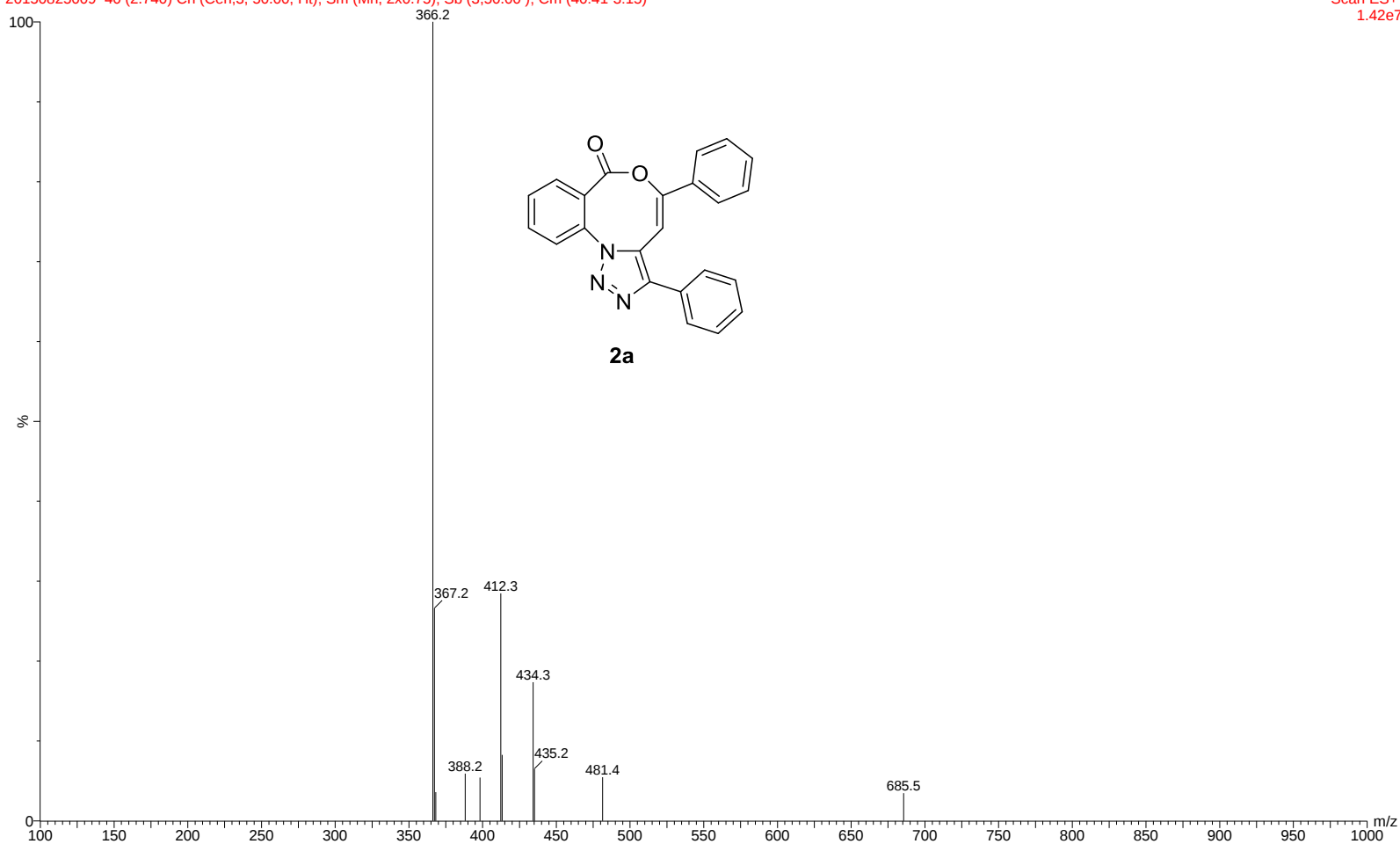


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2a** in CDCl_3

ib-Ng-086

20150825009 40 (2.740) Cn (Cen,3, 50.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (40:41-3:15)

Scan ES+
1.42e7

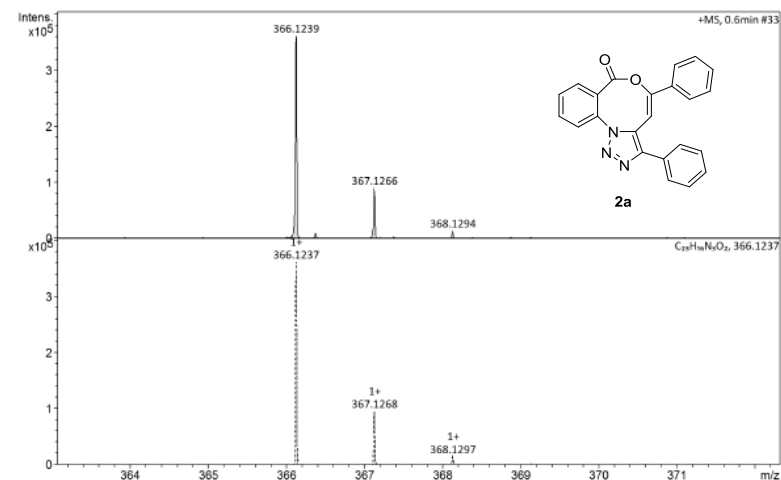
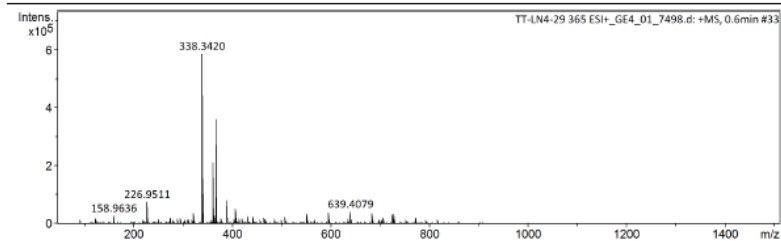


LRMS of compound **2a**

Display Report

Analysis Info		Acquisition Date	9/30/2015 11:25:01 AM	
Analysis Name	D:\Data\nctu_service\data\2015\20150930\TT-LN4-29 365 ESI+_GE4_01_7498.d	Operator	NCTU	
Method	Small molecule.m	Instrument	Impact HD	1819@00.00164
Sample Name	TT-LN4-29 365 ESI+			
Comment				

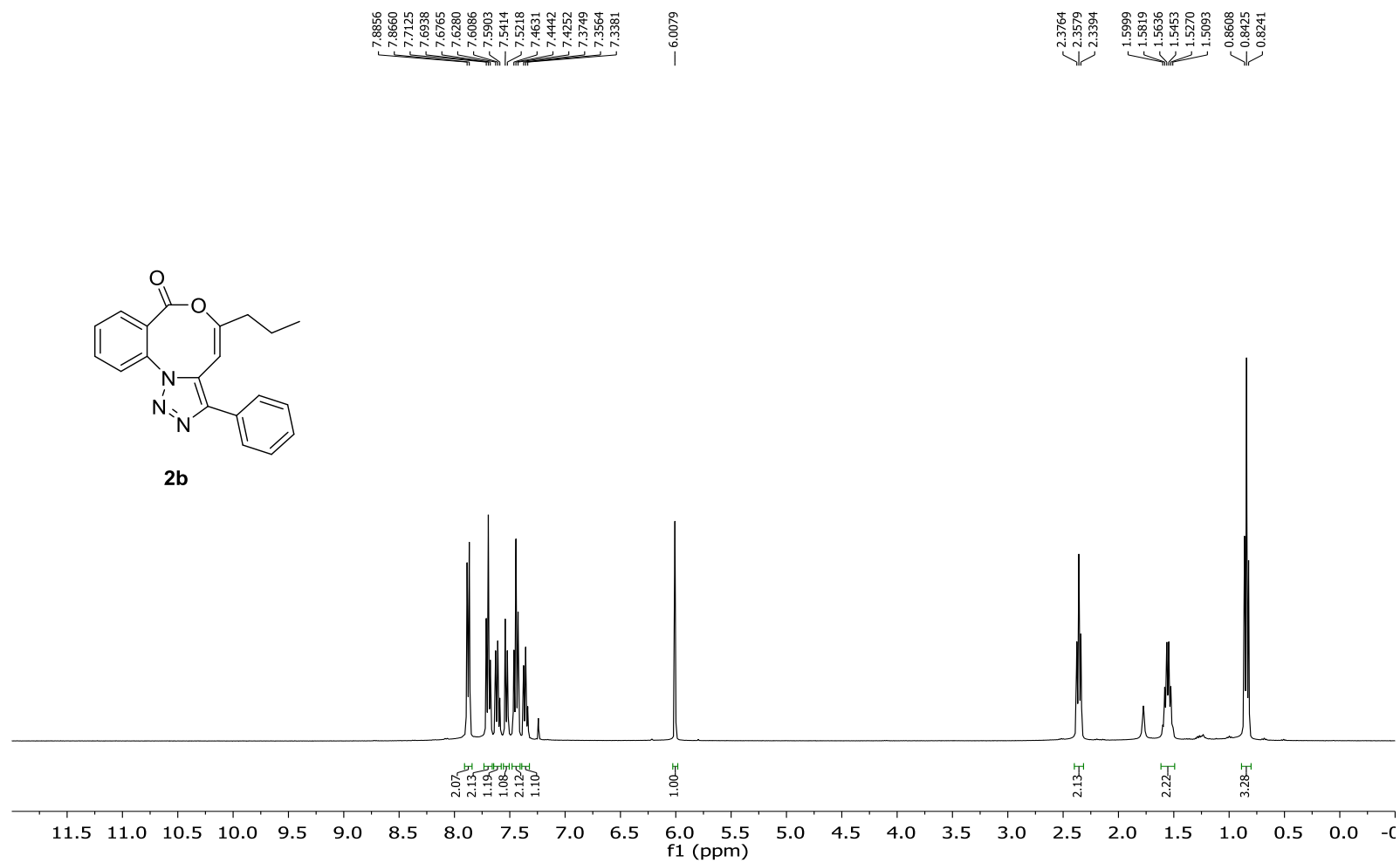
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



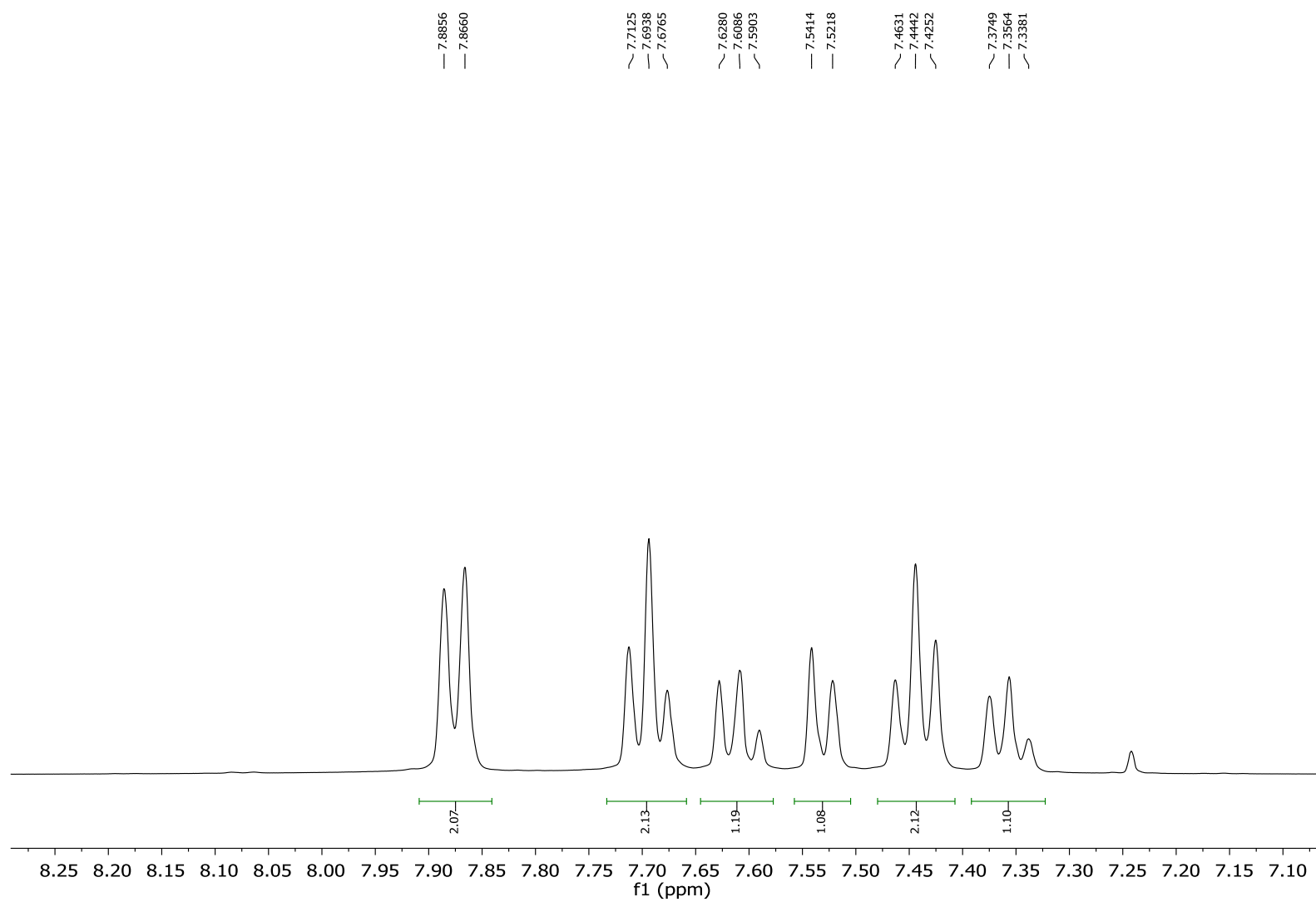
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
366.1239	1	C23H16N3O2	366.1237	0.5	7.1	1	100.00	17.5	even	ok	M+H

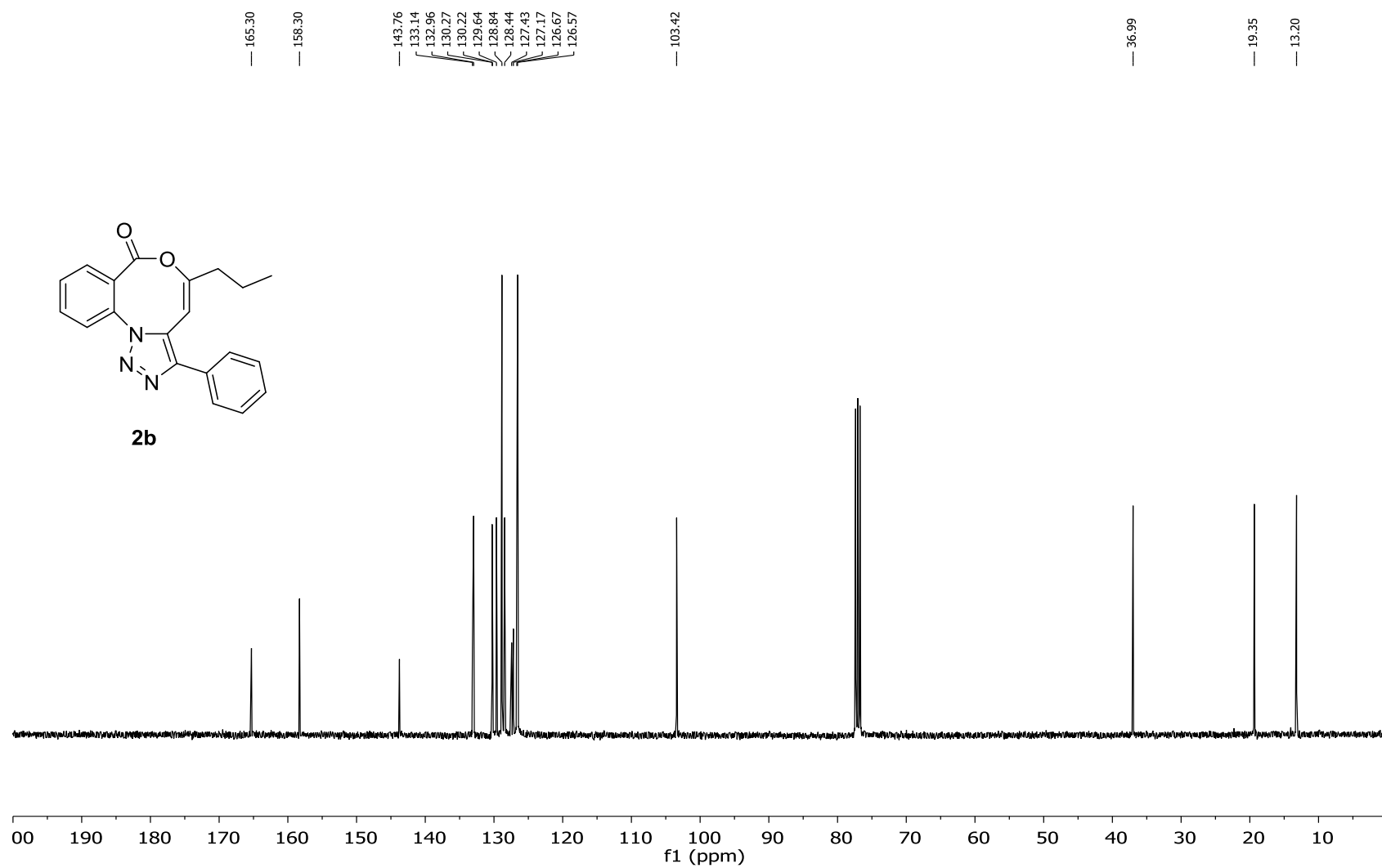
HRMS of compound **2a**



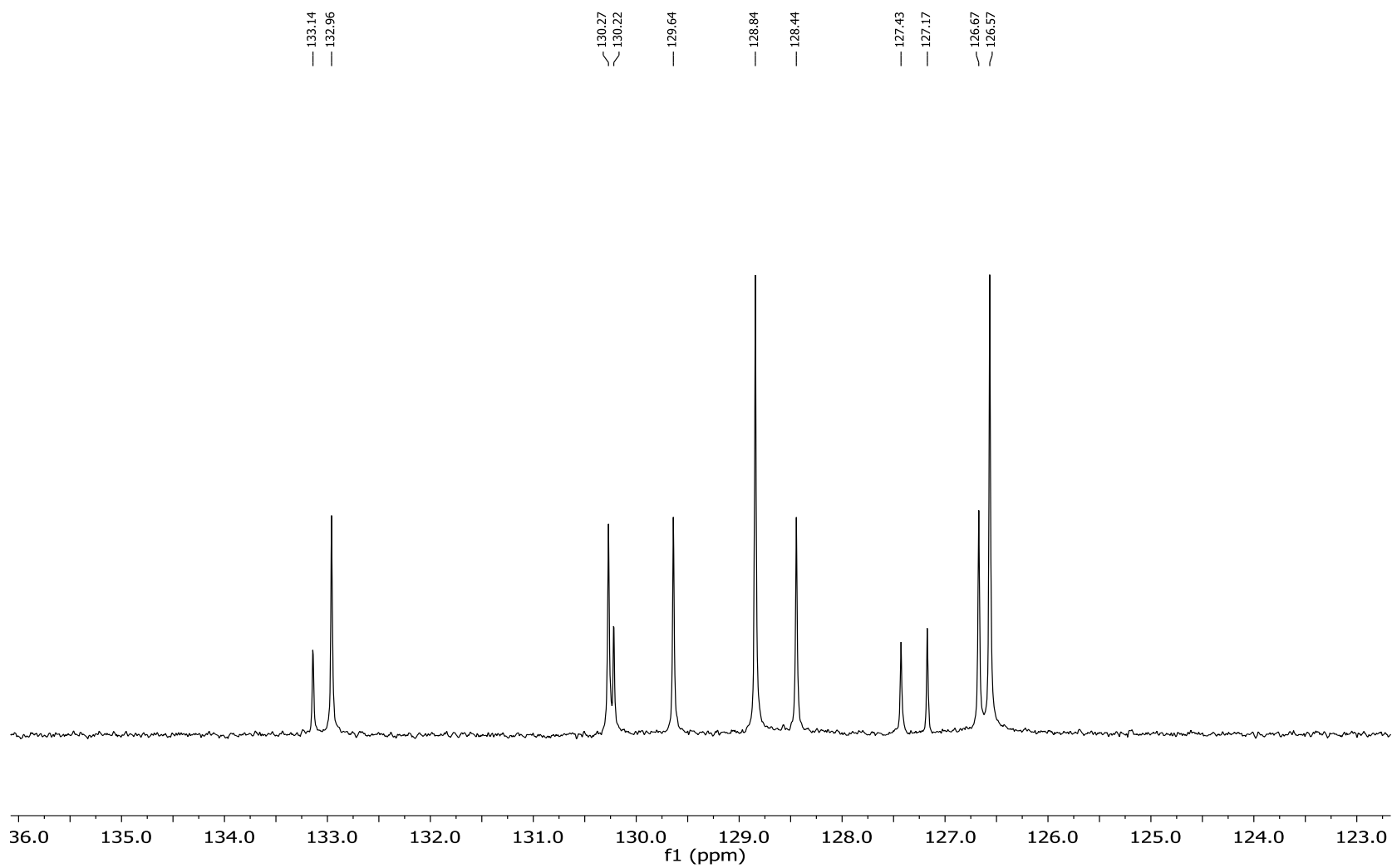
¹H NMR Spectrum (400 MHz) of compound **2b** in CDCl₃



Expansion of ^1H NMR Spectrum (400 MHz) of compound **2b** in CDCl_3



^{13}C NMR Spectrum (101 MHz) of compound **2b** in CDCl_3

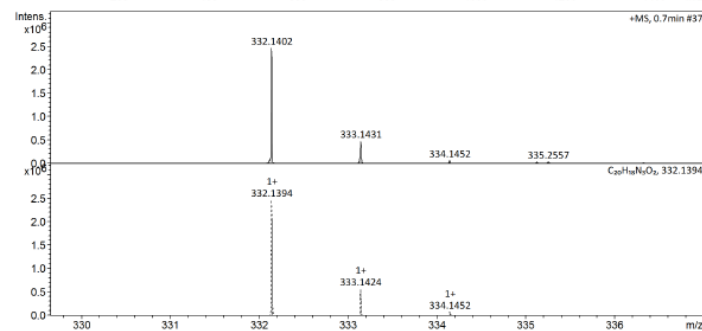
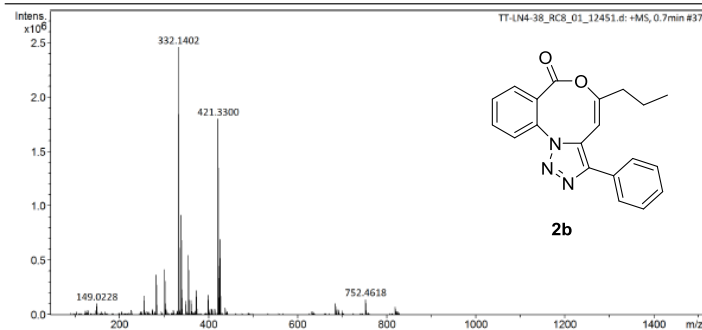


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2b** in CDCl_3

Display Report

Analysis Info		Acquisition Date	2/7/2017 11:58:50 AM	
Analysis Name	D:\Data\ncu service\data\2017\20170207\TT-LN4-38_RC8_01_12451.d	Operator	NCTU	
Method	Small molecule.m	Instrument	Impact HD	1819096.00164
Sample Name	TT-LN4-38			
Comment				

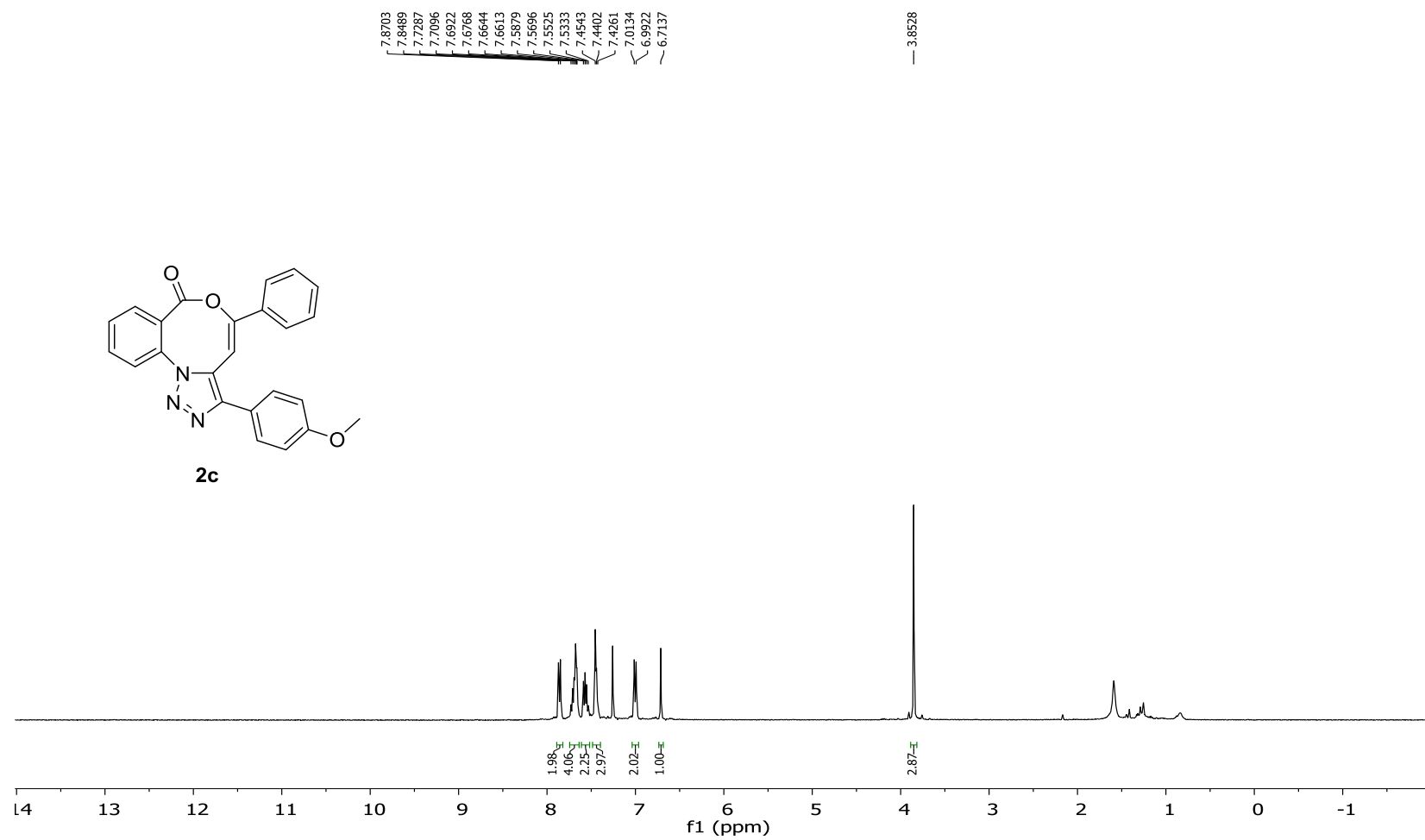
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



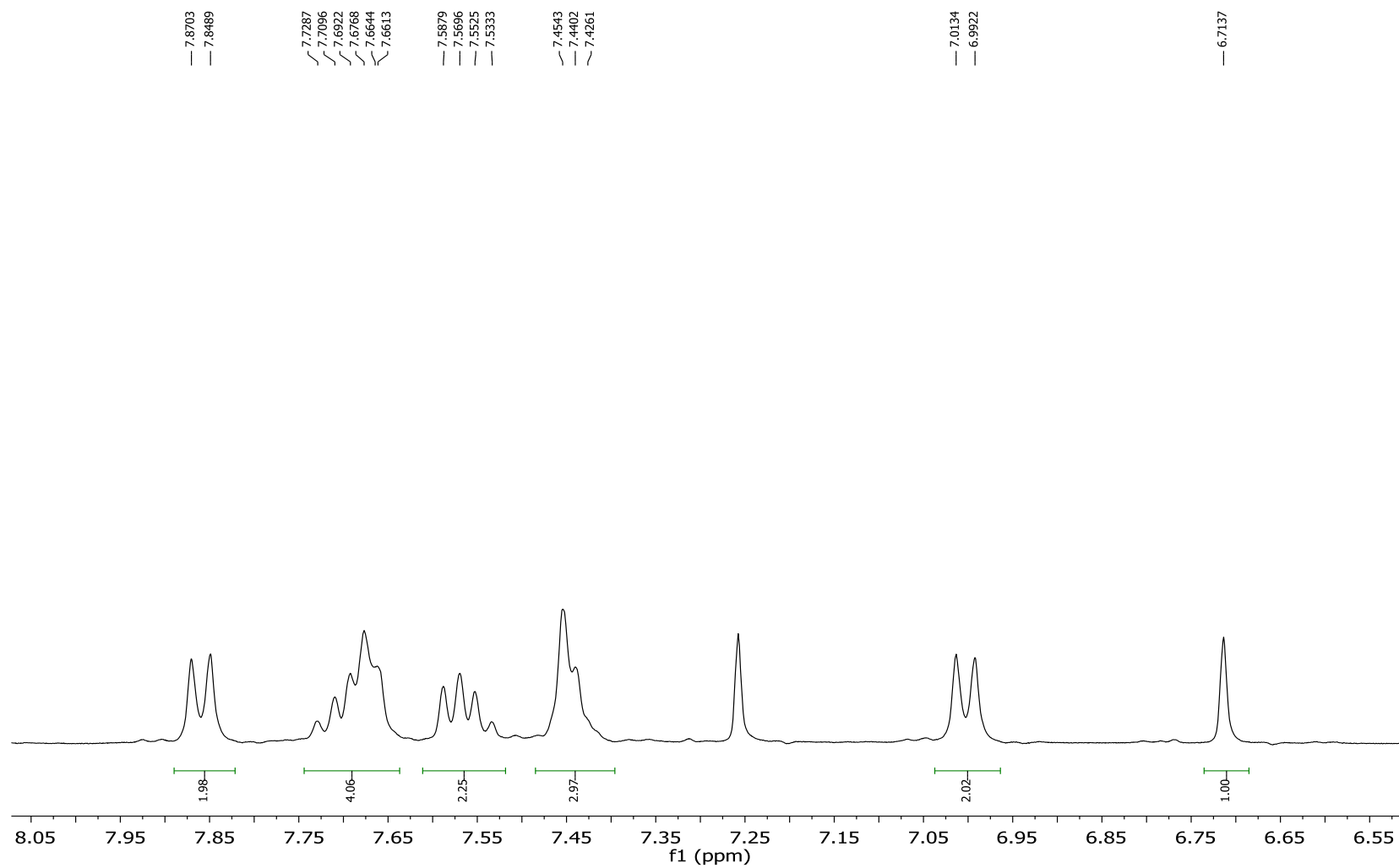
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e ⁻ Conf	N-Rule	Adduct
332.1402	1	C ₂₀ H ₁₈ N ₃ O ₂	332.1394	-2.6	21.4	1	100.00	13.5	even	ok	M+H

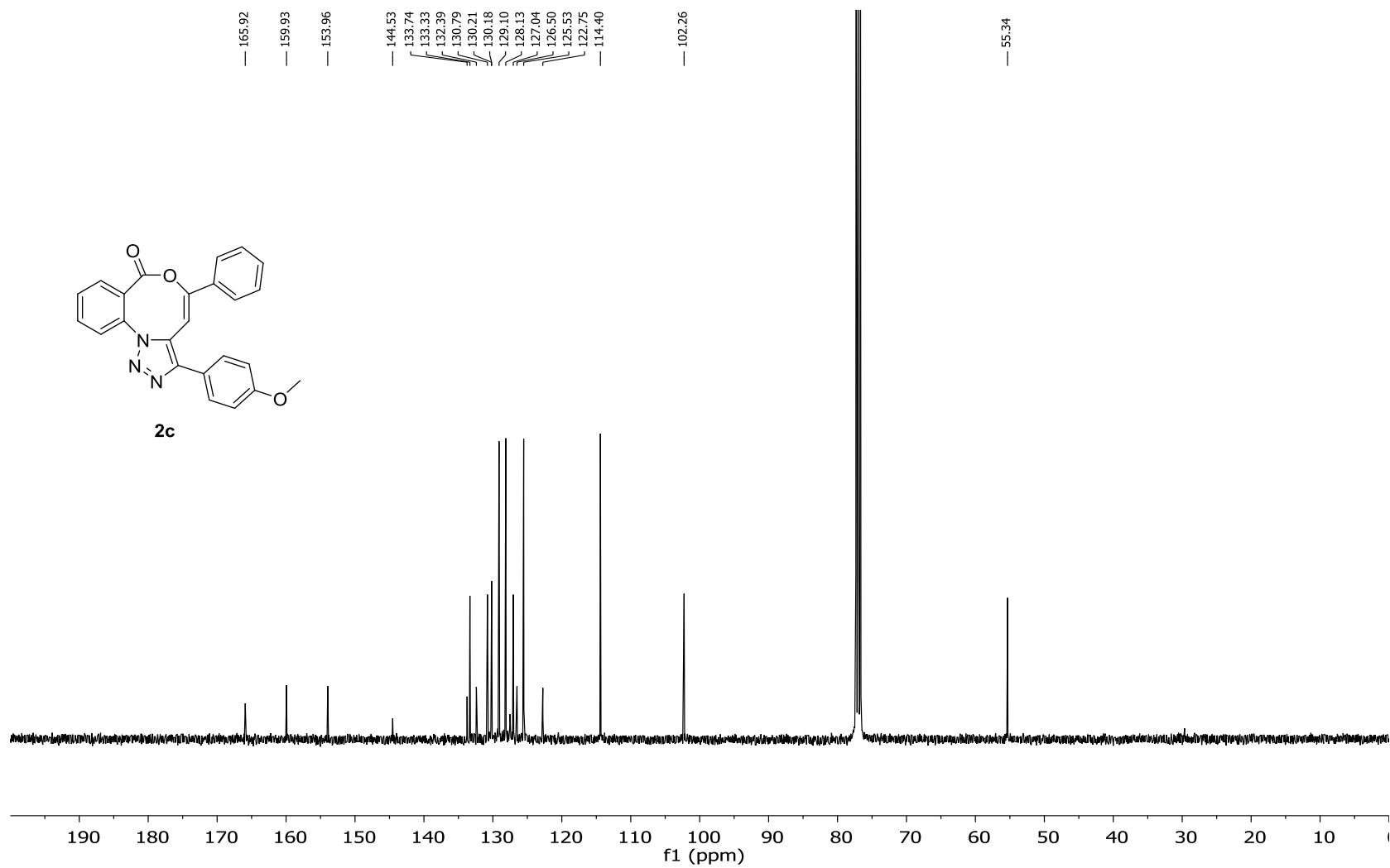
HRMS of compound **2b**



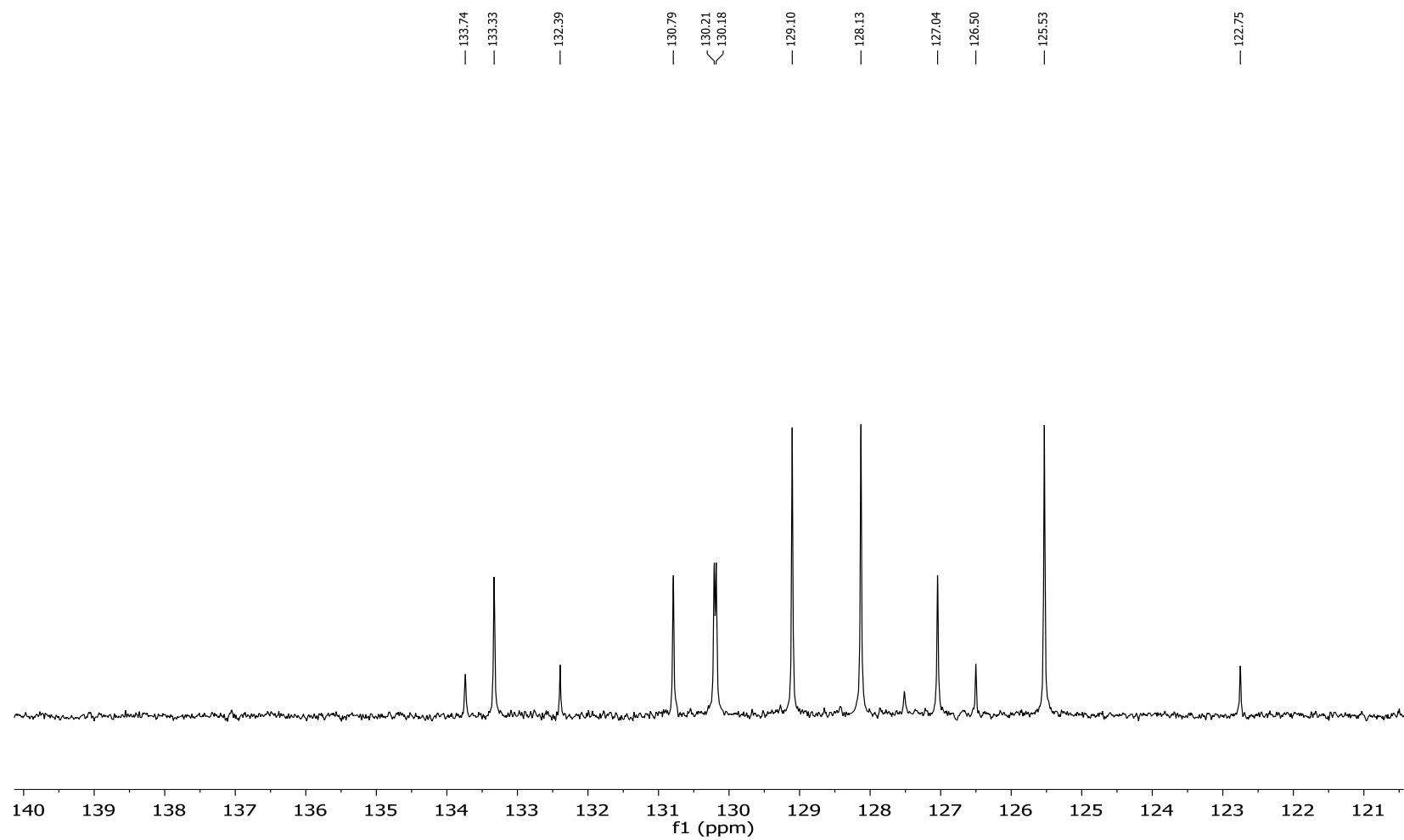
¹H NMR Spectrum (400 MHz) of compound **2c** in CDCl₃



Expansion of ^1H NMR Spectrum (400 MHz) of compound **2c** in CDCl_3



^{13}C NMR Spectrum (101 MHz) of compound **2c** in CDCl_3

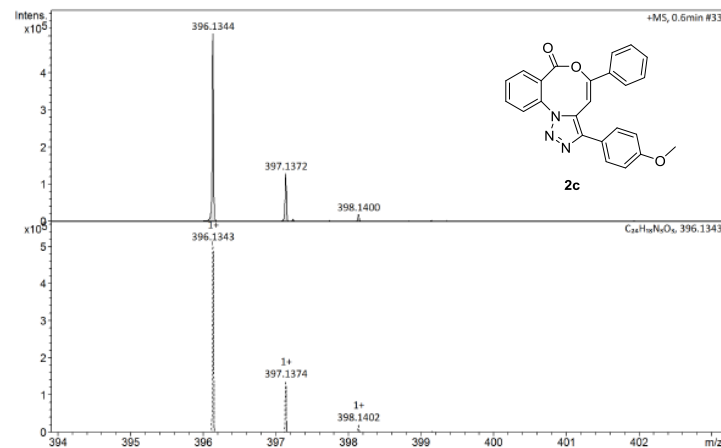
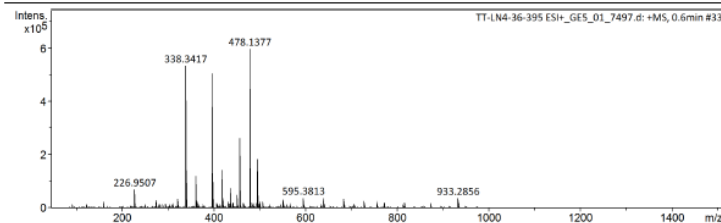


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2c** in CDCl_3

Display Report

Analysis Info		Acquisition Date	9/30/2015 11:20:45 AM	
Analysis Name	D:\Data\Nctu service\data\2015\20150930\TT-LN4-36-395 ESI+_GE5_01_7497.d	Operator	NCTU	
Method	Small molecule.m	Instrument	Impact HD	1819666.00164
Sample Name	TT-LN4-36-395 ESI+			
Comment				

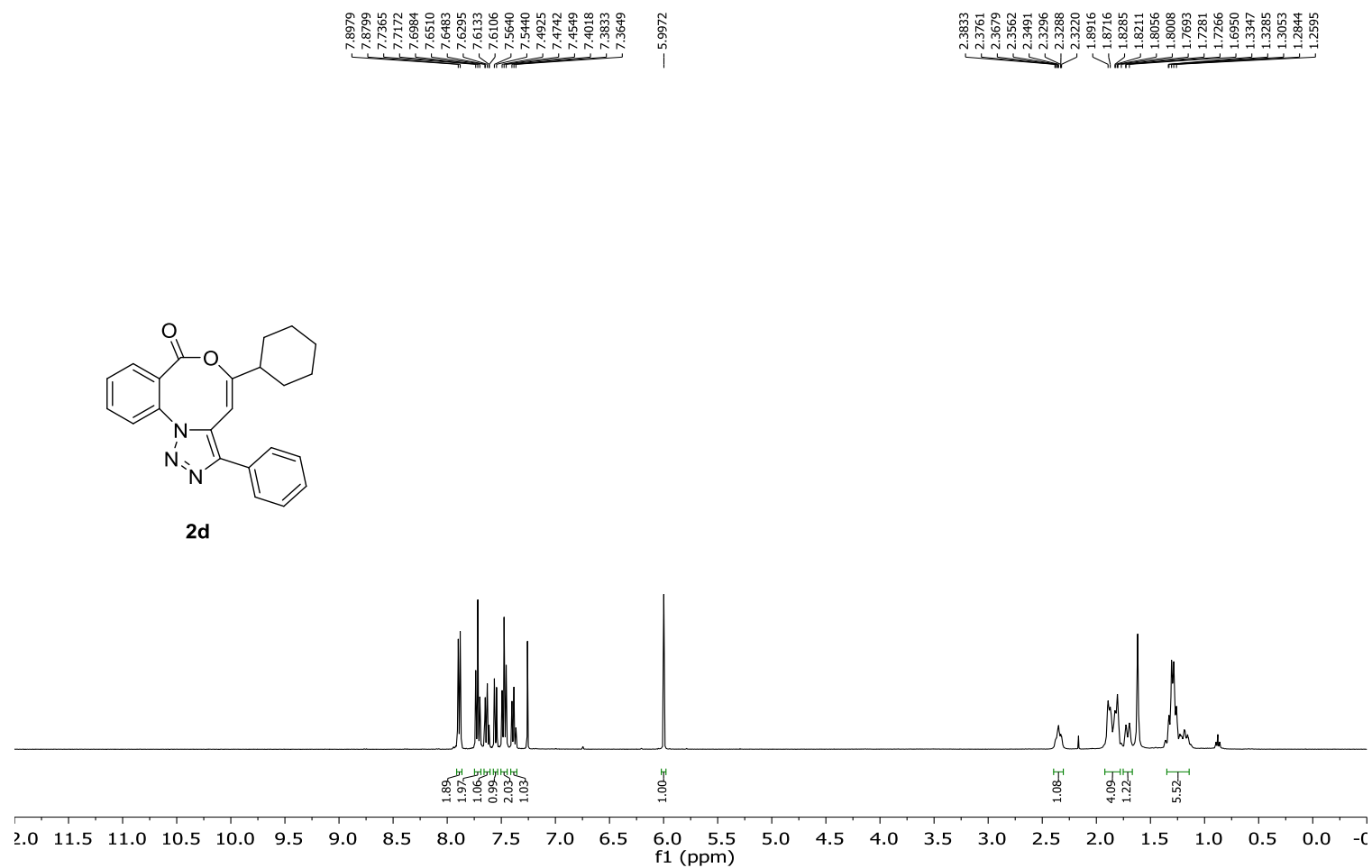
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



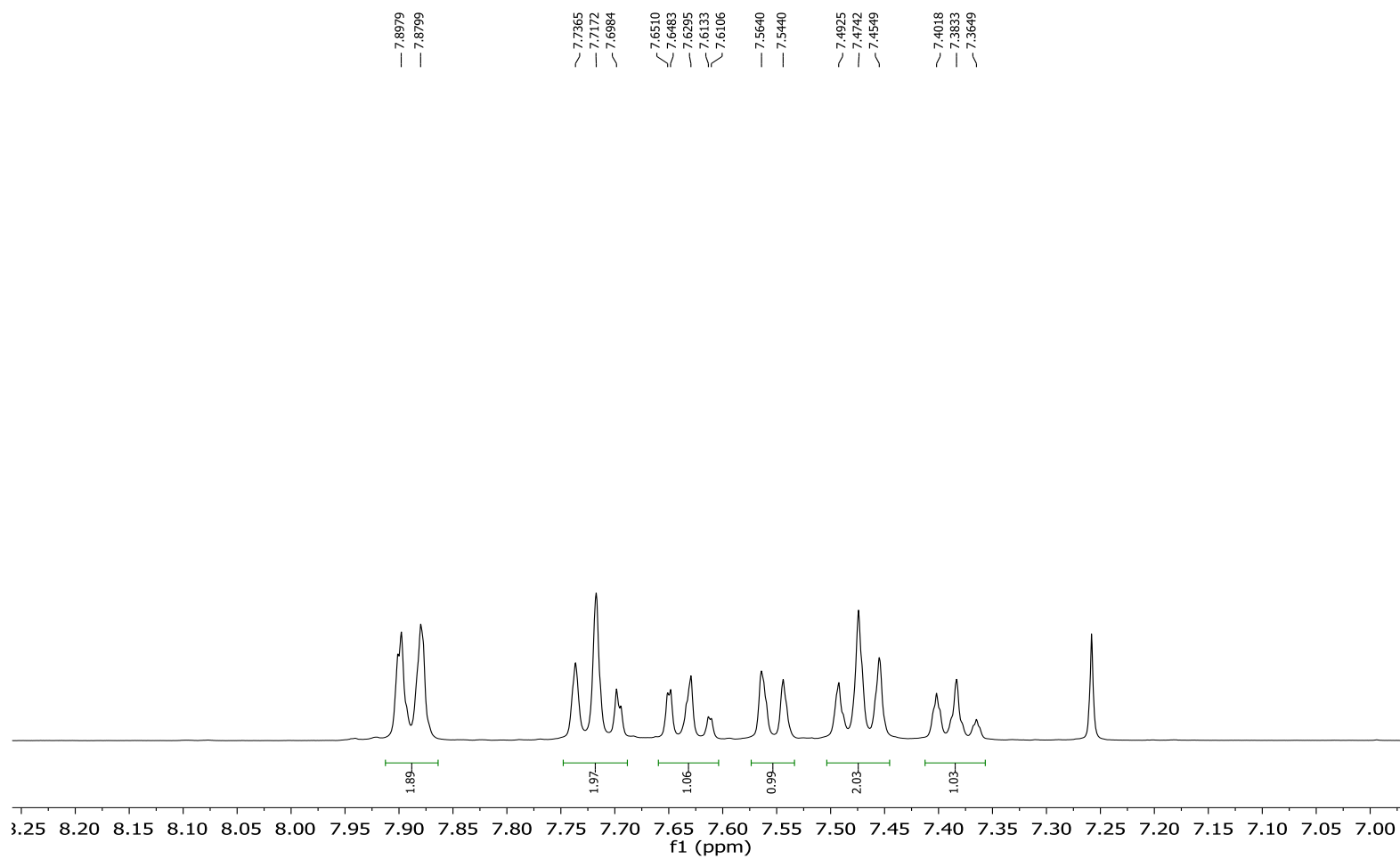
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
396.1344	1	C24H18N3O3	396.1343	-0.4	8.6	1	100.00	17.5	even	ok	M+H

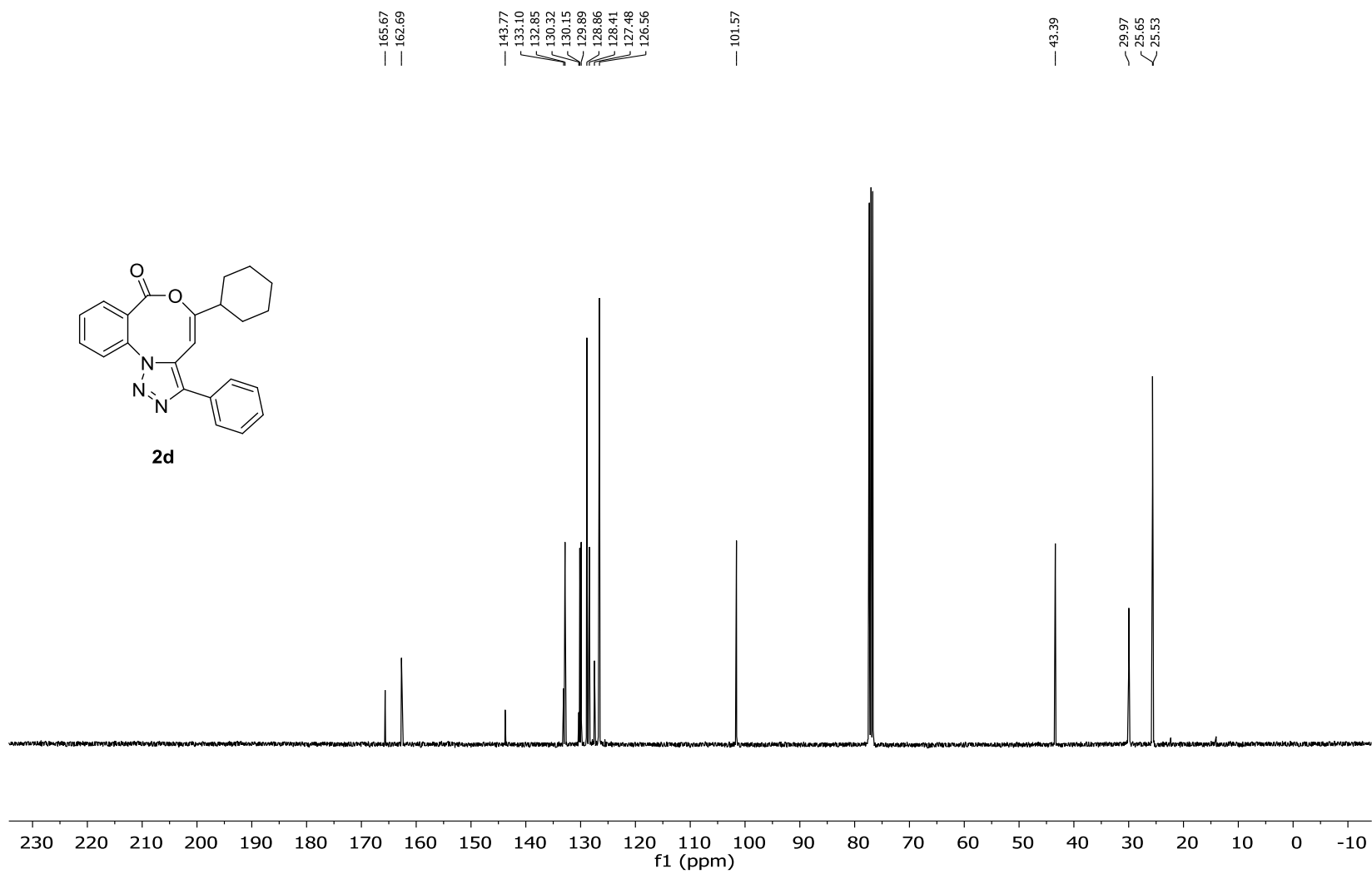
HRMS of compound **2c**

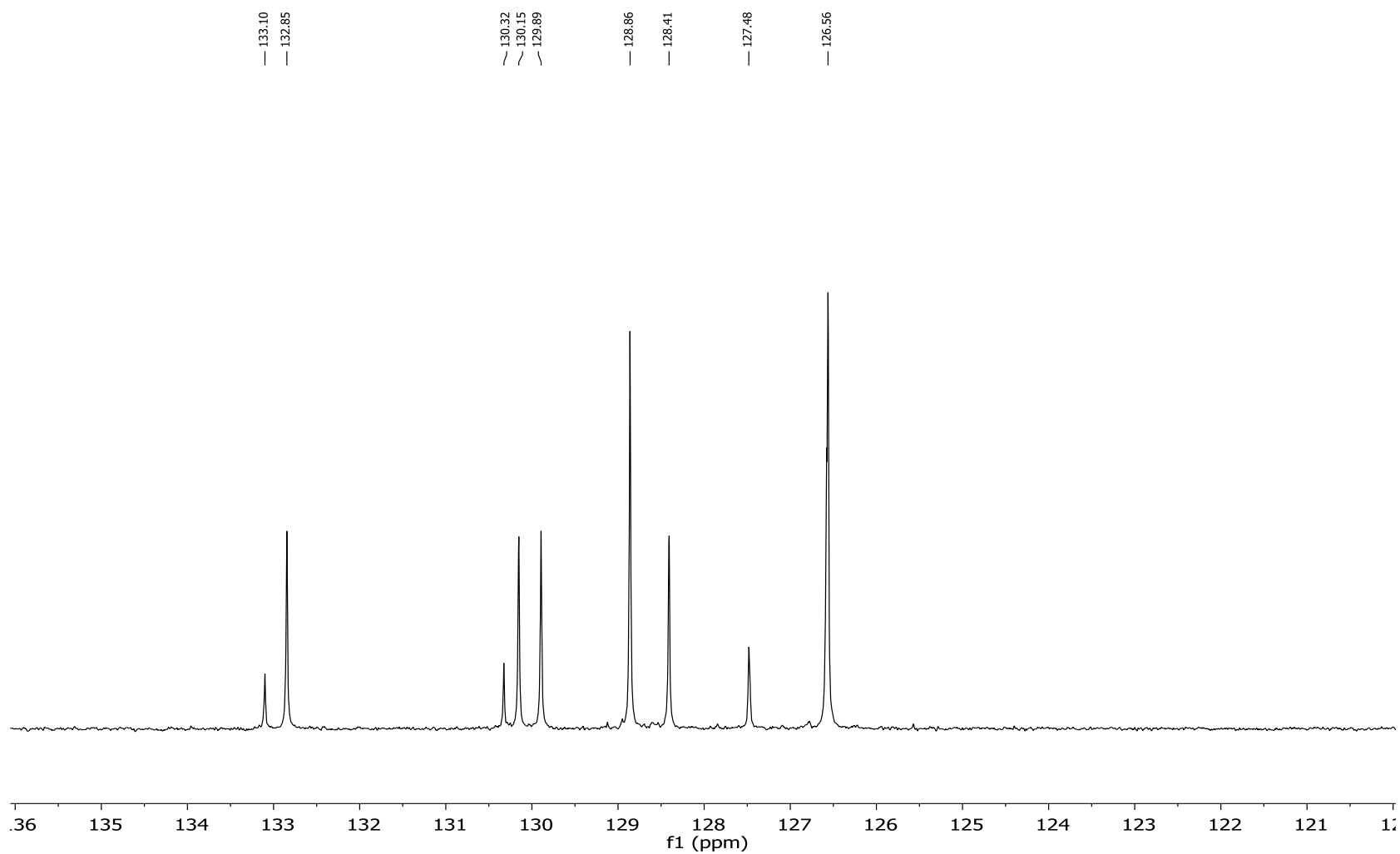


¹H NMR Spectrum (400 MHz) of compound **2d** in CDCl₃



Expansion of ^1H NMR Spectrum (400 MHz) of compound **2d** in CDCl_3



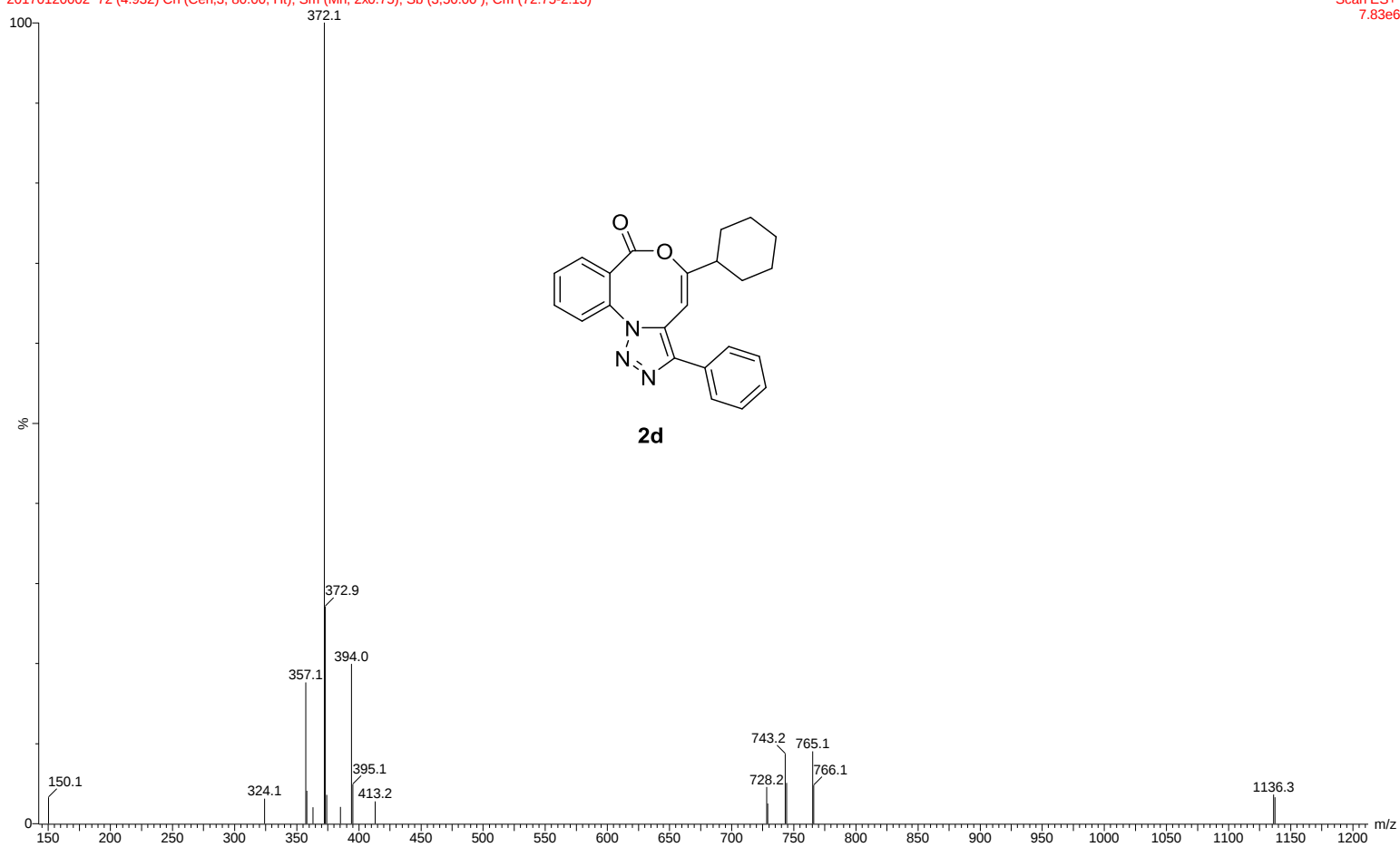


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2d** in CDCl_3

ib-N10-059

20170120002 72 (4.932) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (72:75-2:13)

Scan ES+
7.83e6

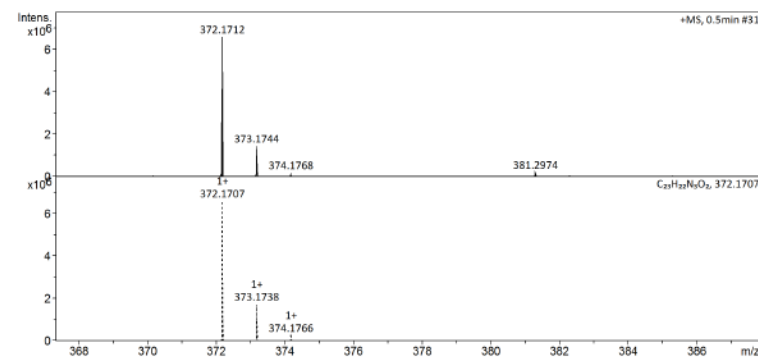
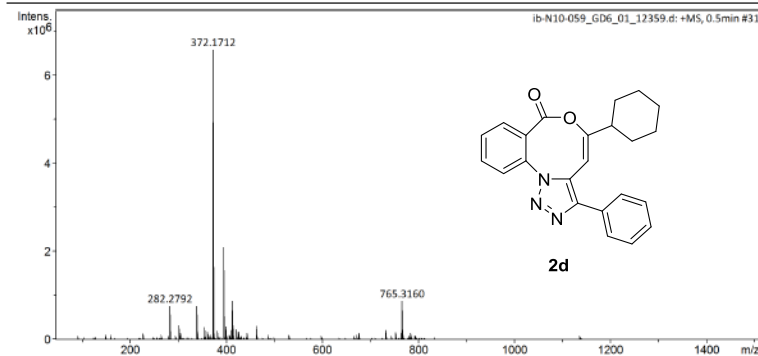


LRMS of compound **2d**

Display Report

Analysis Info		Acquisition Date	1/20/2017 12:33:48 PM	
Analysis Name	D:\Data\ntcu service\data\2017\20170120\ib-N10-059_GD6_01_12359.d	Operator	NCTU	
Method	Small molecule.m	Instrument	impact HD	1819098.00164
Sample Name	ib-N10-059			
Comment				

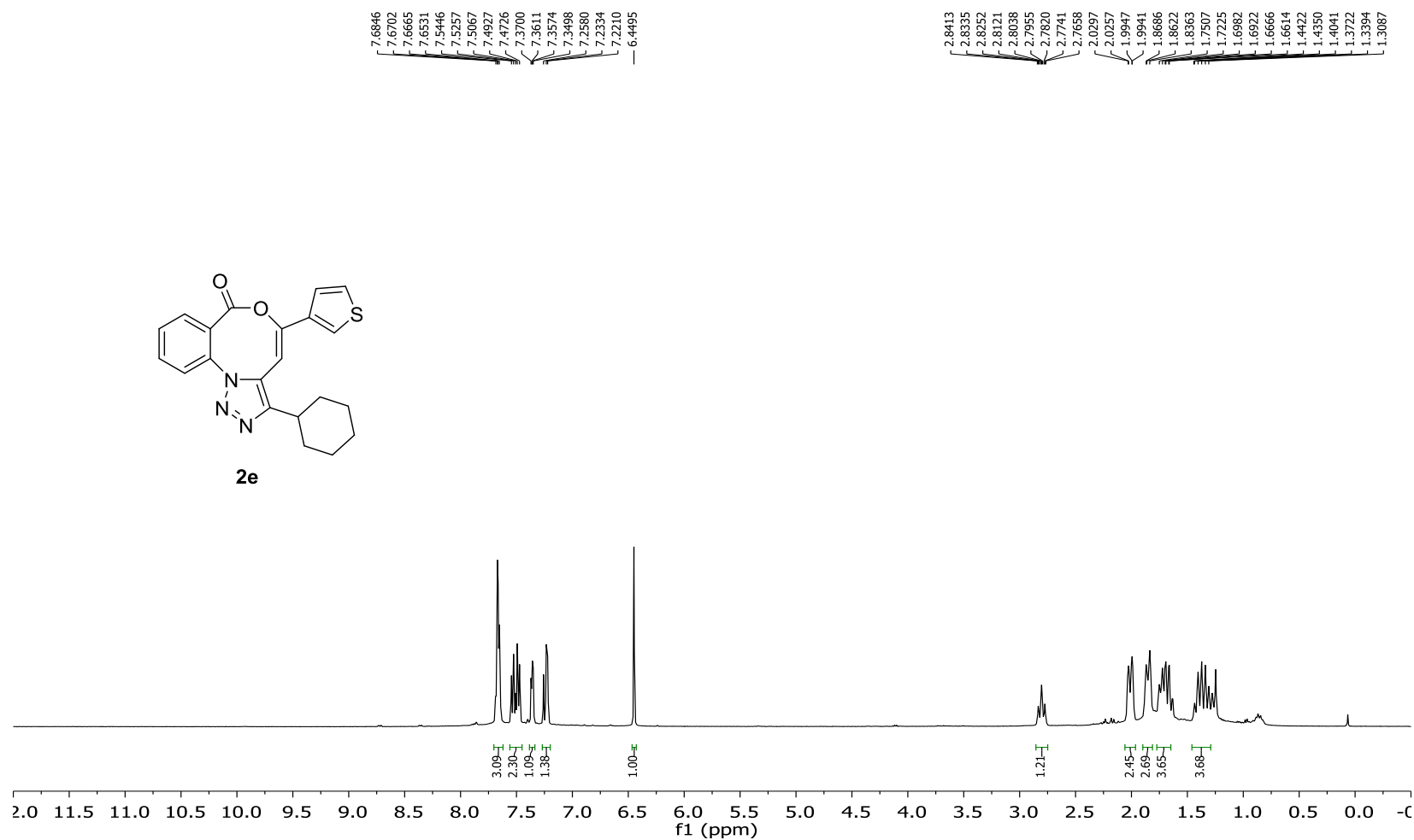
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



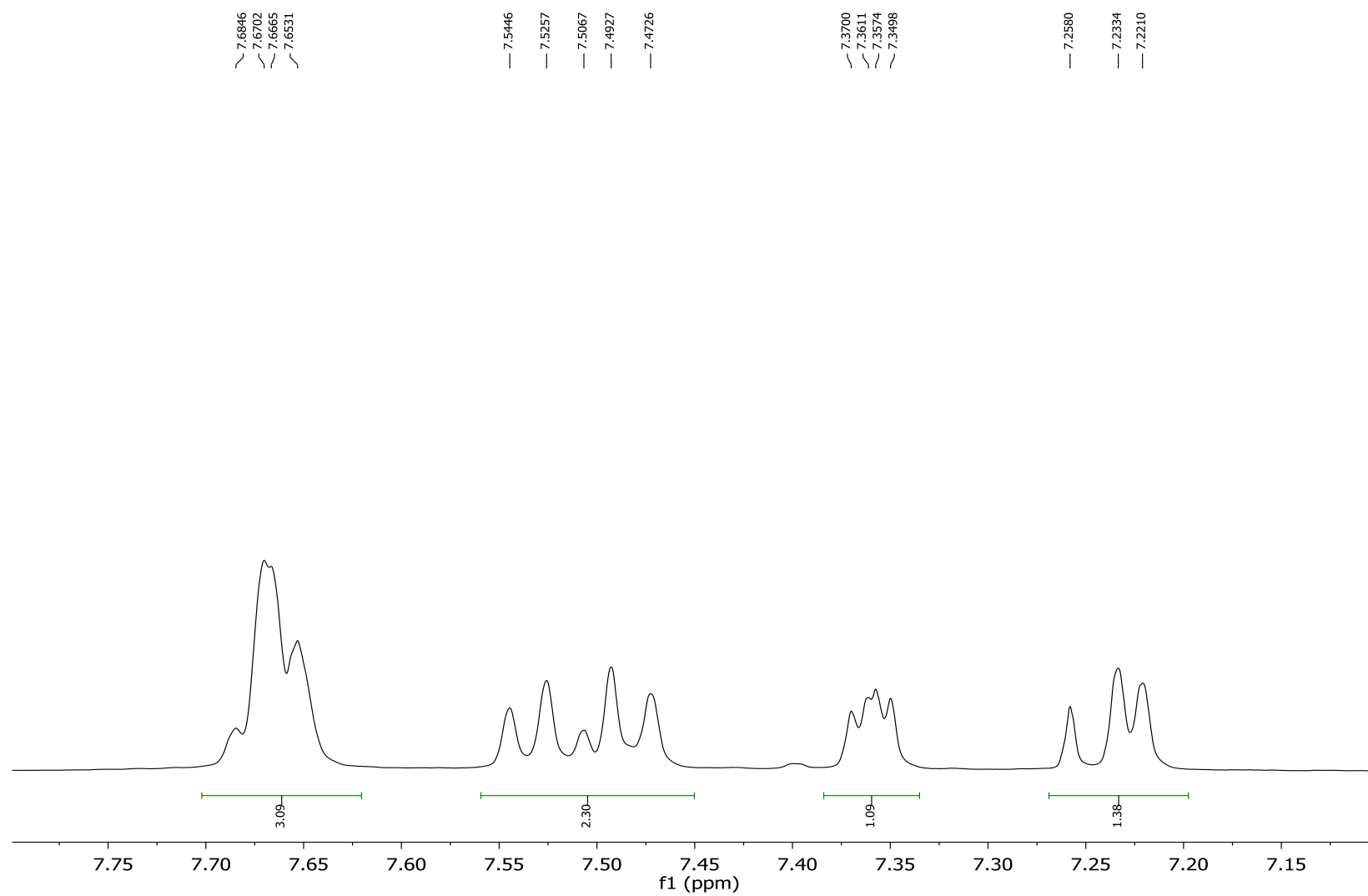
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
372.1712	1	C ₂₃ H ₂₂ N ₃ O ₂	372.1707	-1.5	23.3	1	100.00	14.5	even	ok	M+H

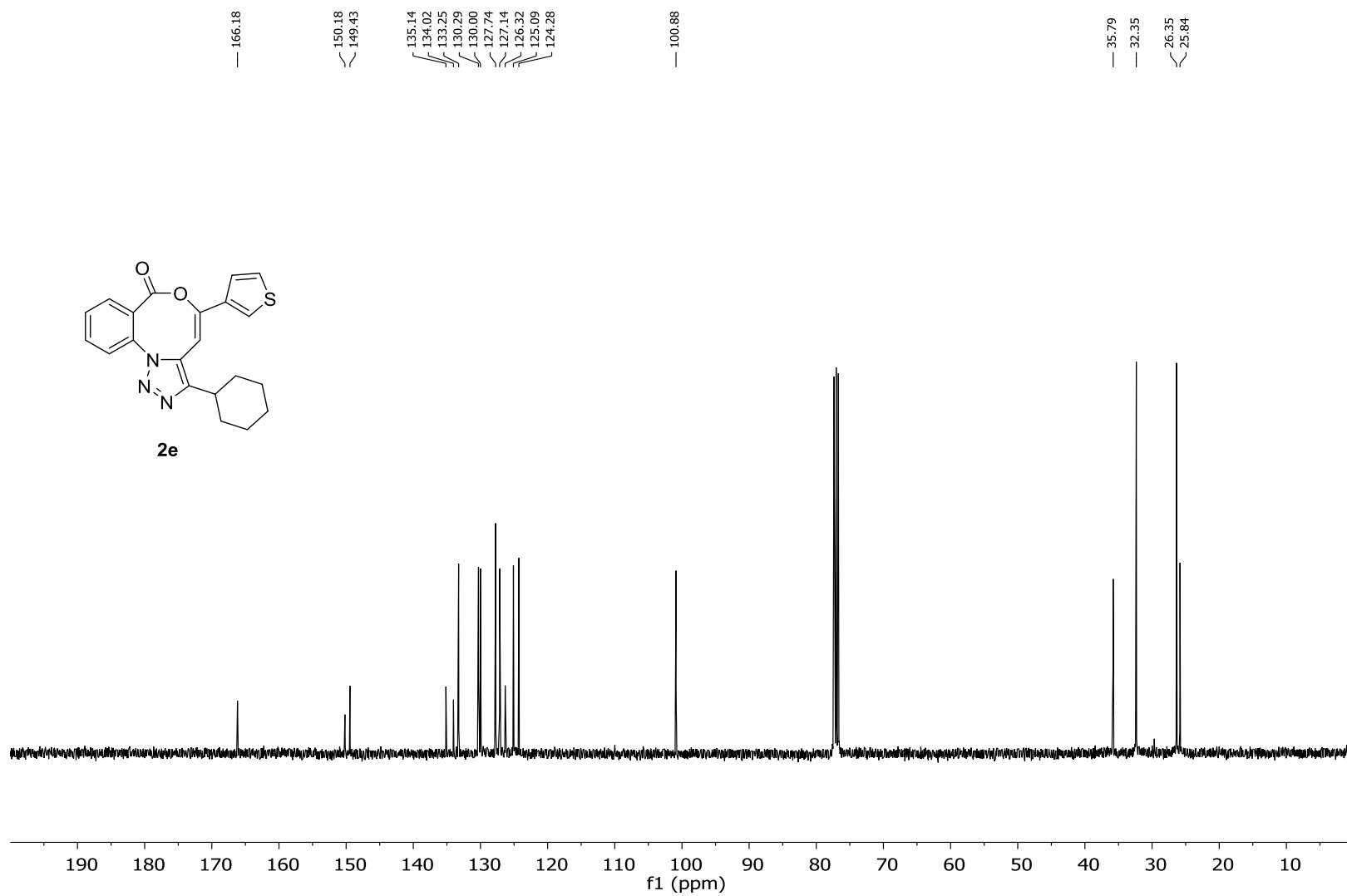
HRMS of compound **2d**

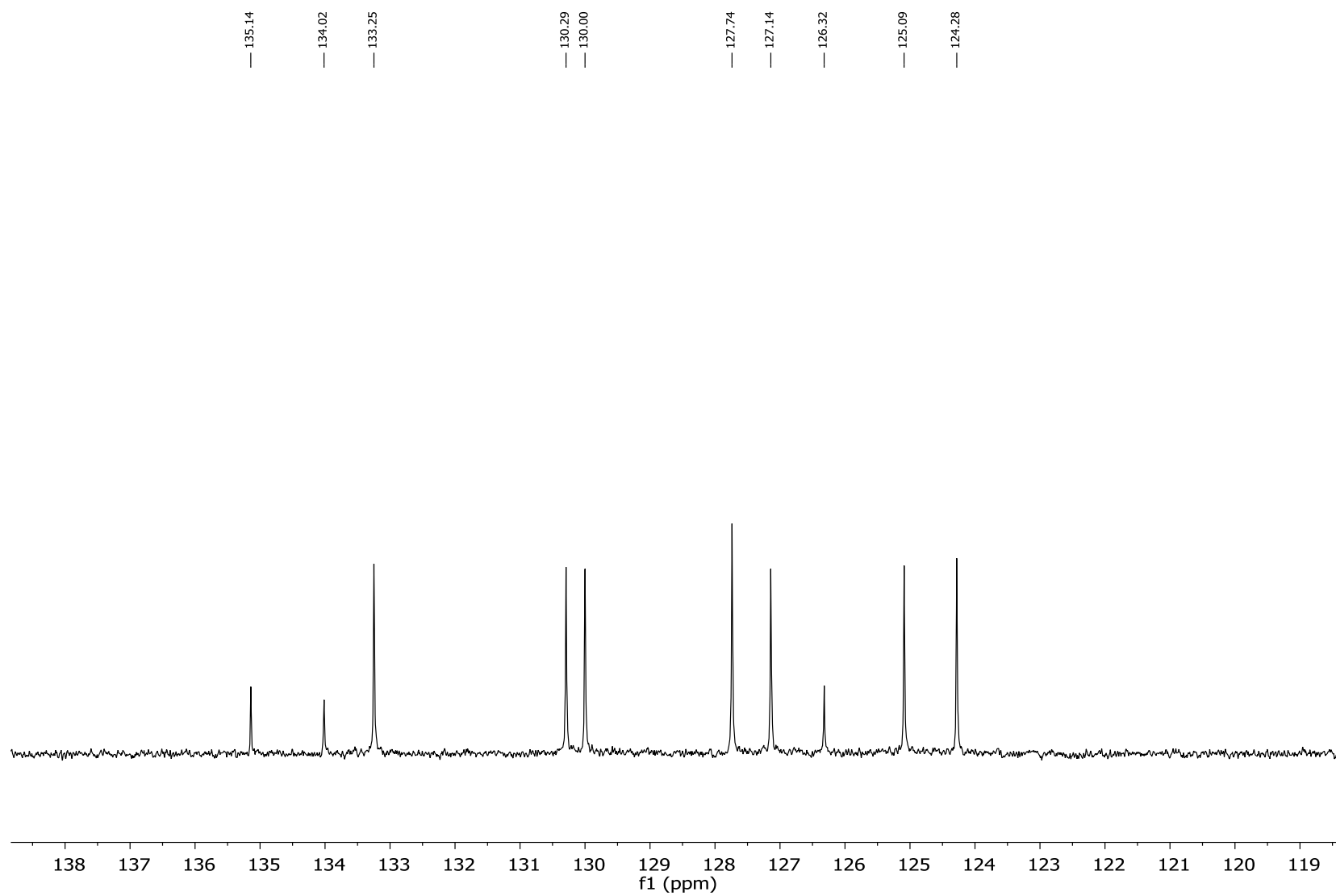


¹H NMR Spectrum (400 MHz) of compound **2e** in CDCl₃



Expansion of ^1H NMR Spectrum (400 MHz) of compound **2e** in CDCl_3



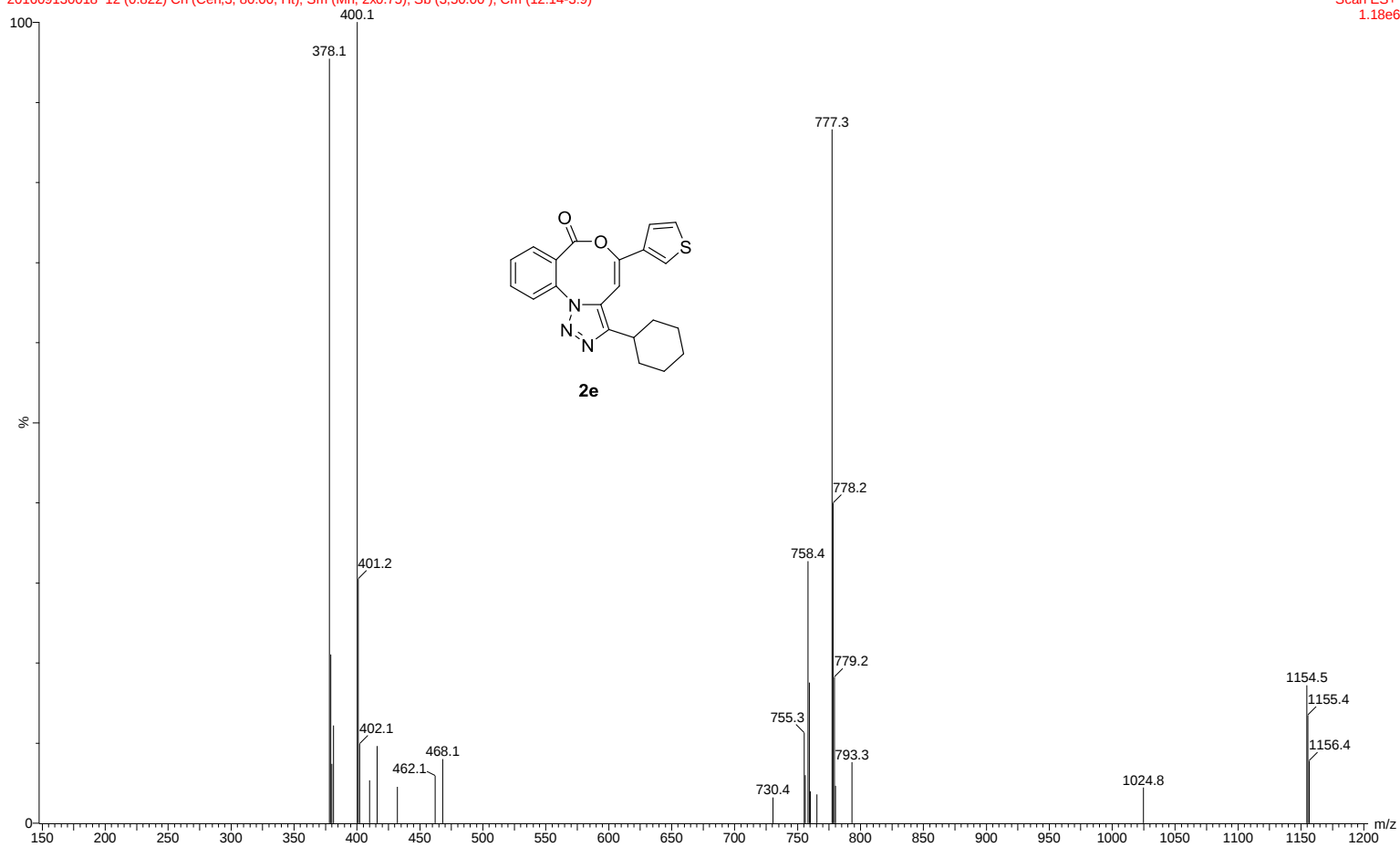


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2e** in CDCl_3

TT-LNS-83

201609130018 12 (0.822) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (12:14-3:9)

Scan ES+
1.18e6

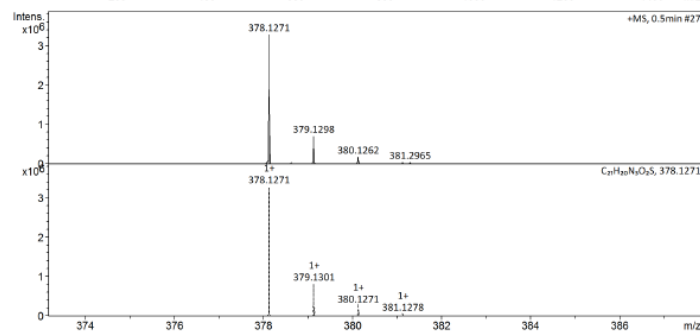
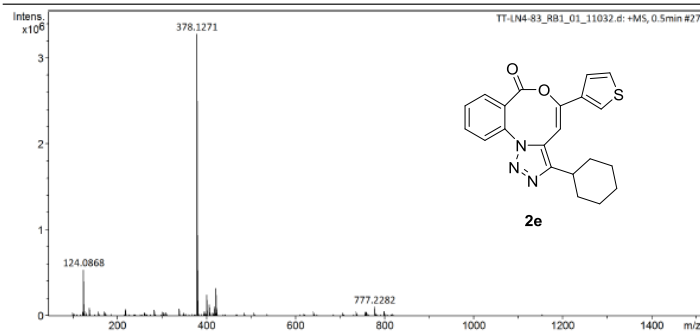


LRMS of compound **2e**

Display Report

Analysis Info		Acquisition Date	9/13/2016 10:56:52 AM	
Analysis Name	D:\Data\ncu service\data\2016\20160912\TT-LN4-83_RB1_01_11032.d	Operator	NCTU	
Method	Small molecule.m	Instrument	Impact HD	1819096.00164
Sample Name	TT-LN4-83			
Comment				

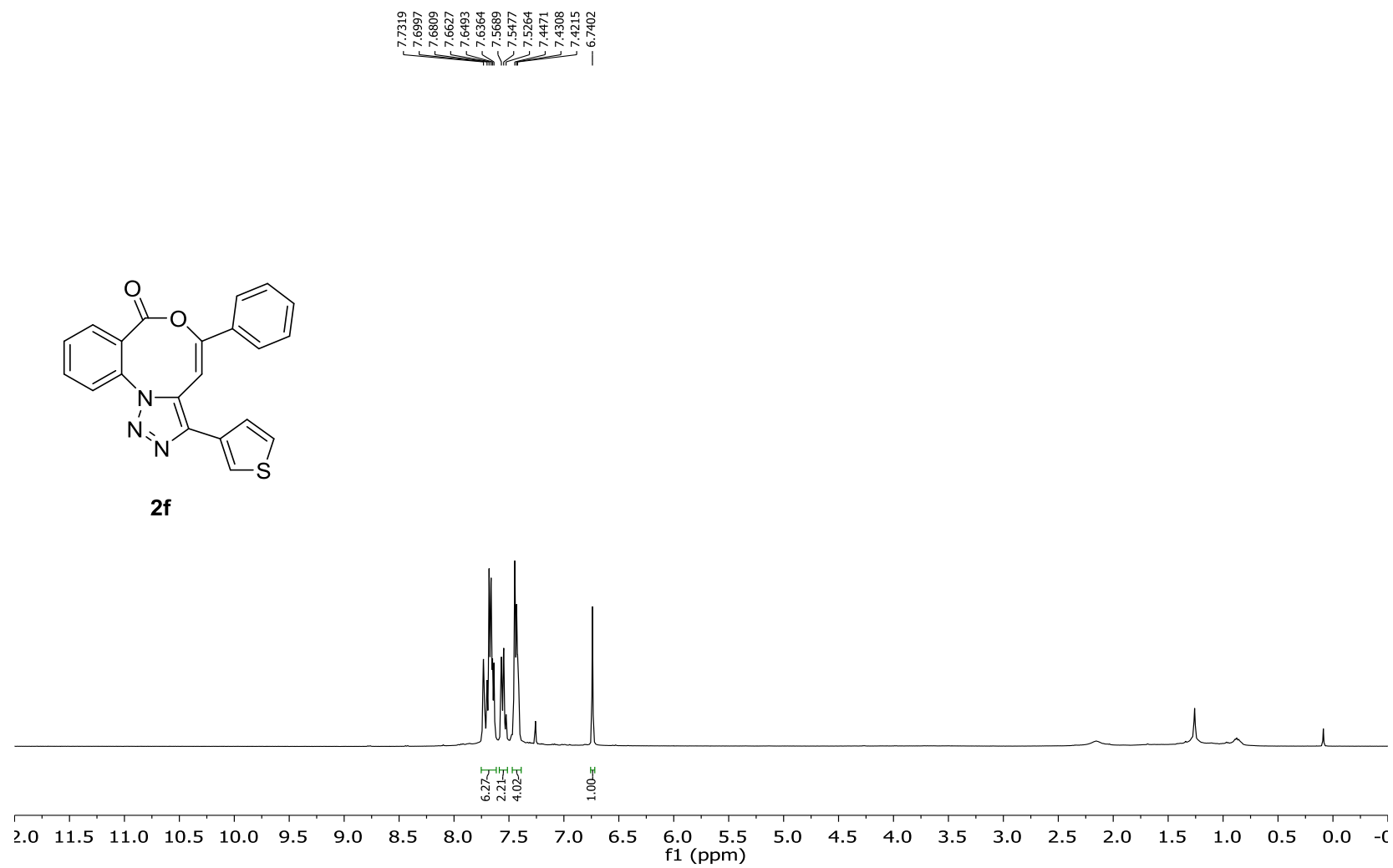
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

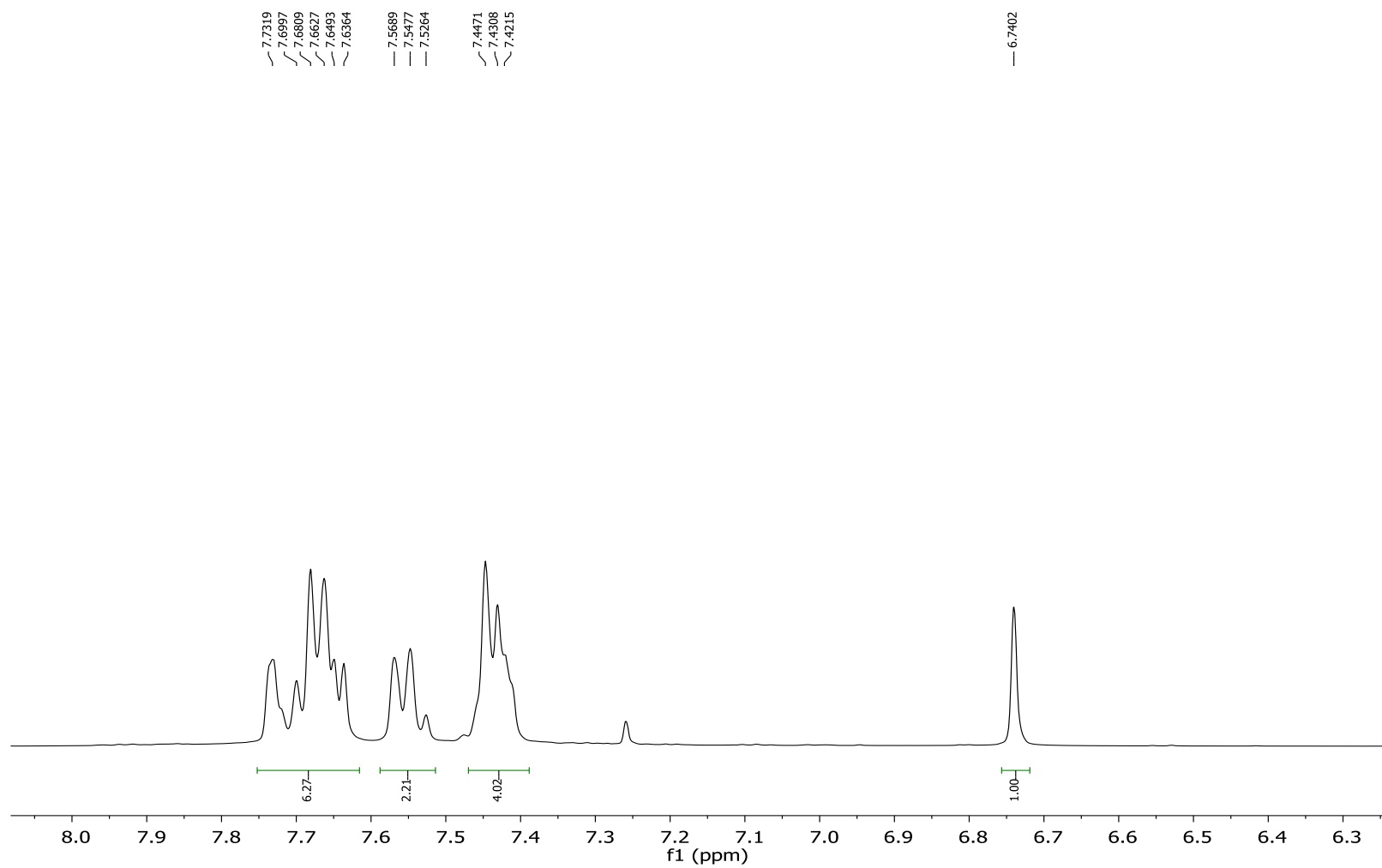


Display Report

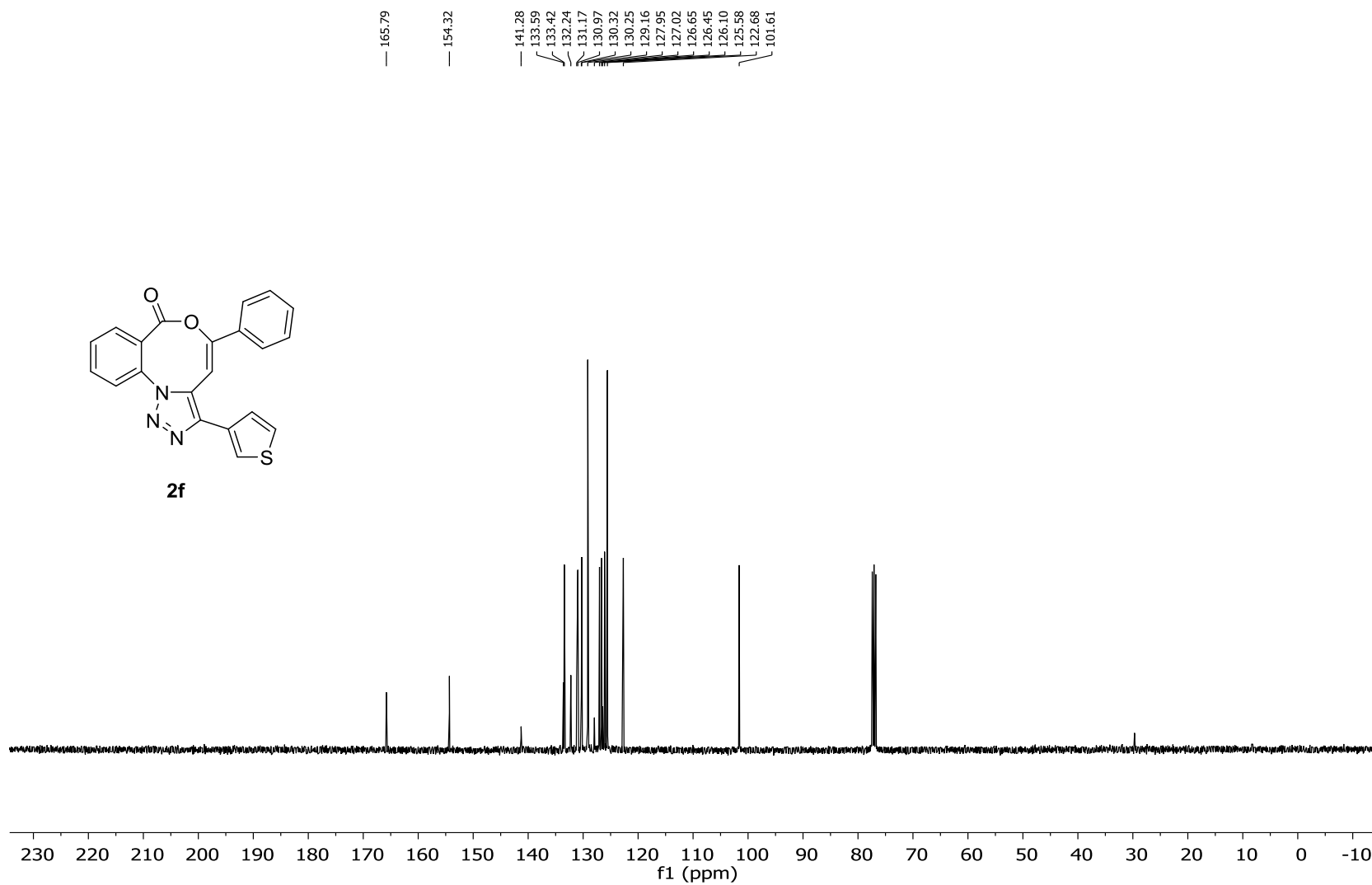
Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e ⁻ Conf	N-Rule	Adduct
378.1271	1	C ₂₁ H ₂₀ N ₃ O ₂ S	378.1271	-0.2	21.1	1	100.00	13.5	even	ok	M+H

HRMS of compound **2e**

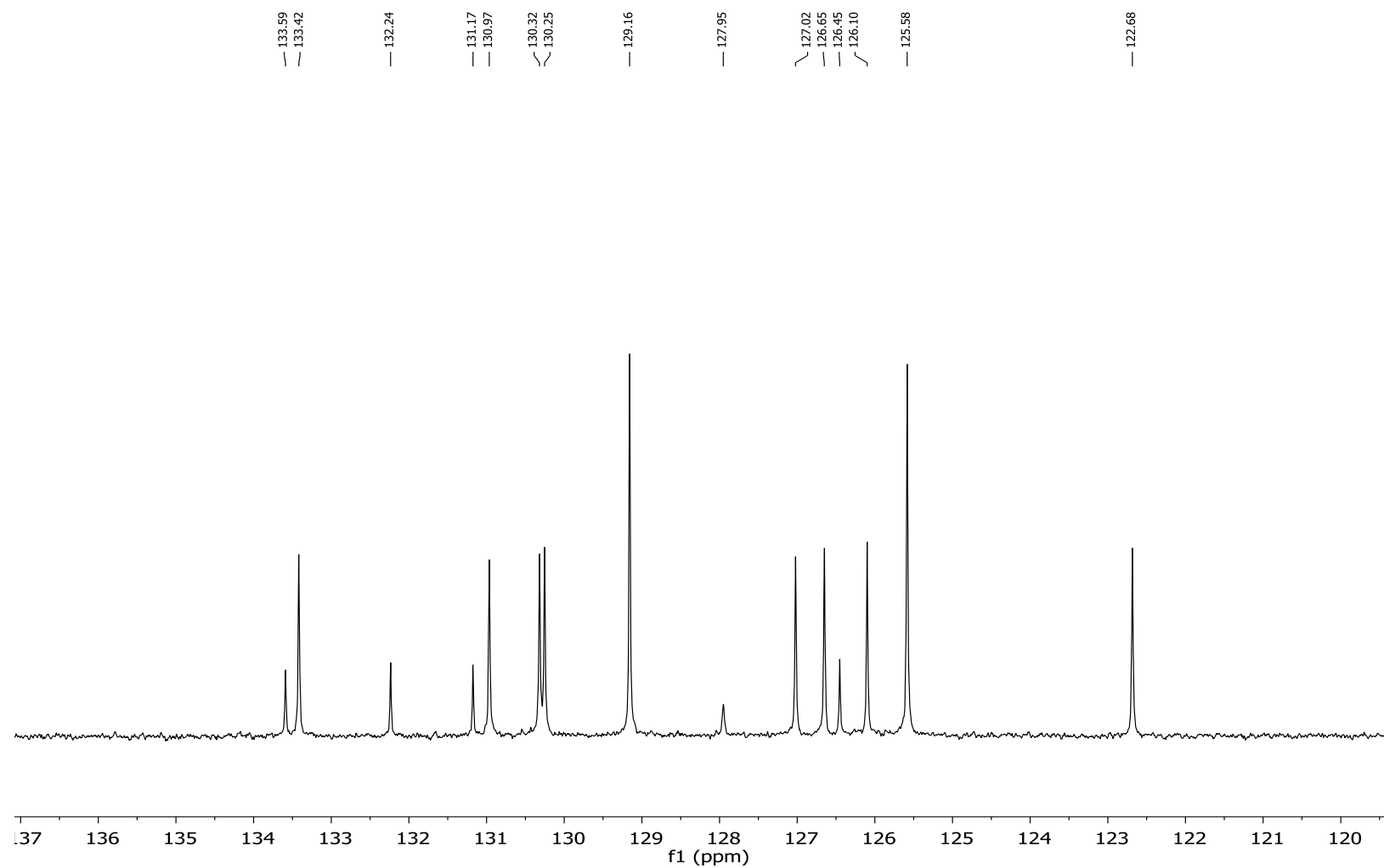




Expansion of ^1H NMR Spectrum (400 MHz) of compound **2f** in CDCl_3



^{13}C NMR Spectrum (101 MHz) of compound **2f** in CDCl_3

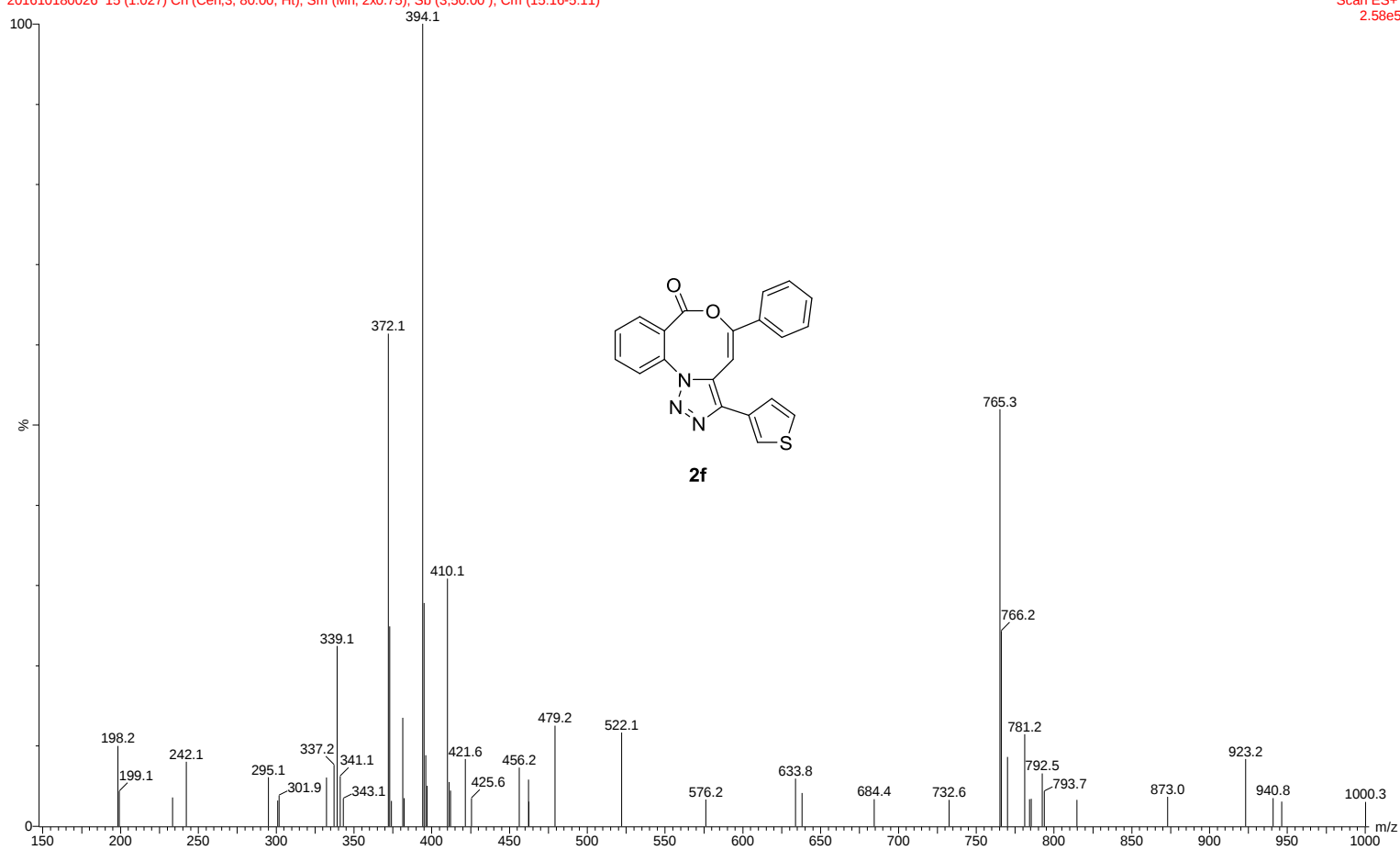


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2f** in CDCl_3

TT-LN4-93

201610180026 15 (1.027) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (15:16-5:11)

Scan ES+
2.58e5

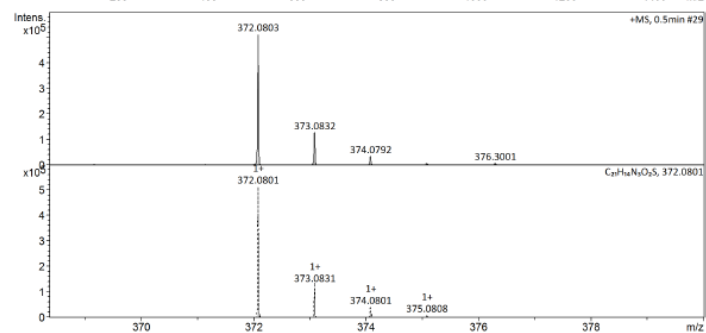
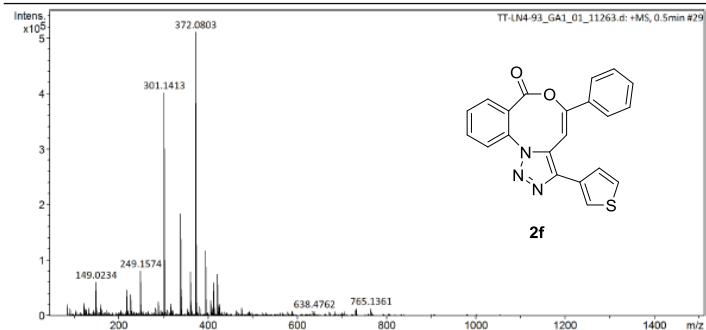


LRMS of compound **2f**

Display Report

Analysis Info		Acquisition Date	10/18/2018 10:25:45 AM
Analysis Name	D:\Data\ncu service\data\2018\20181018\TT-LN4-93_GA1_01_11263.d	Operator	NCTU
Method	Small molecule.m	Instrument	Impact HD
Sample Name	TT-LN4-93		1819098.00164
Comment			

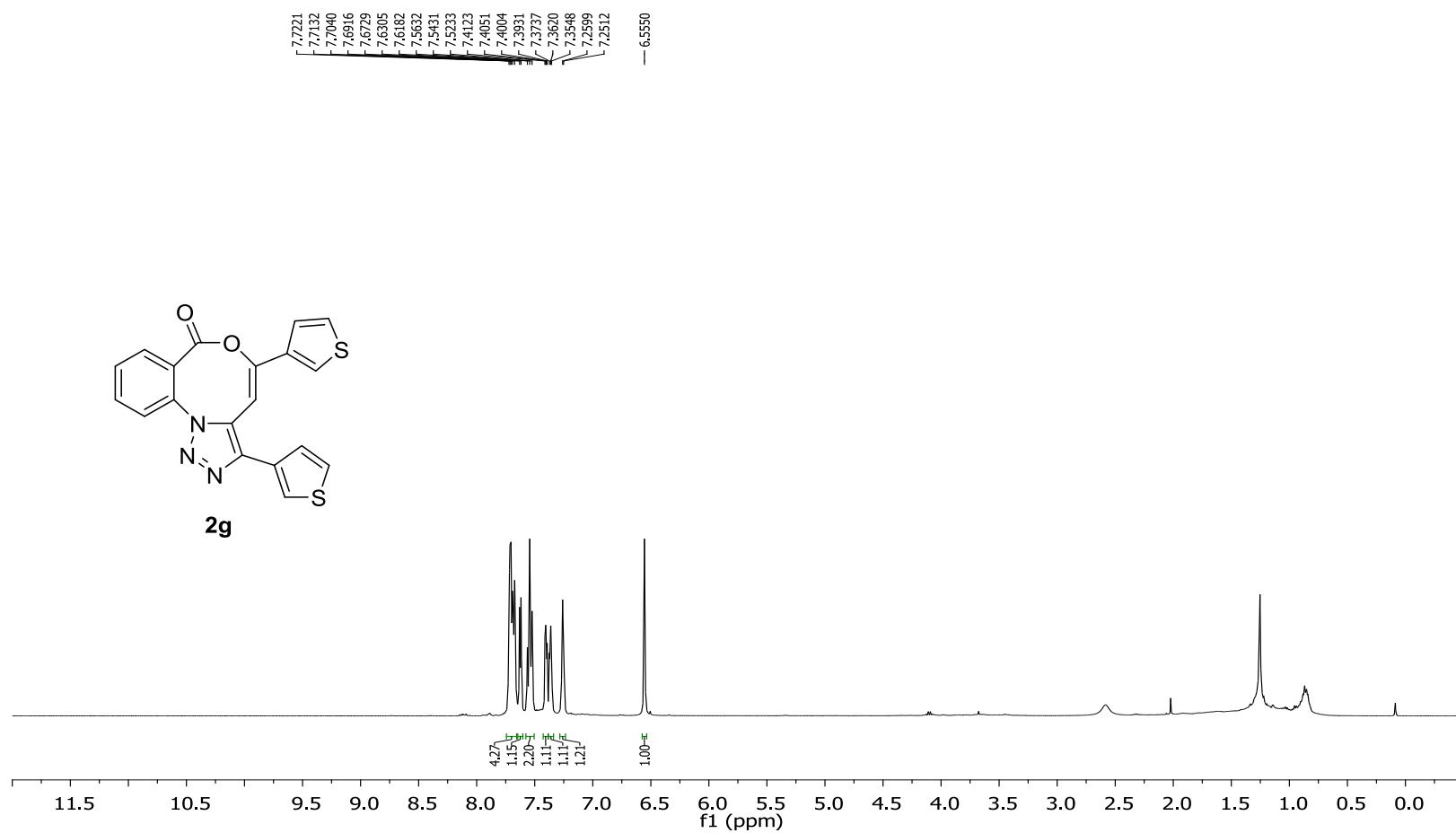
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



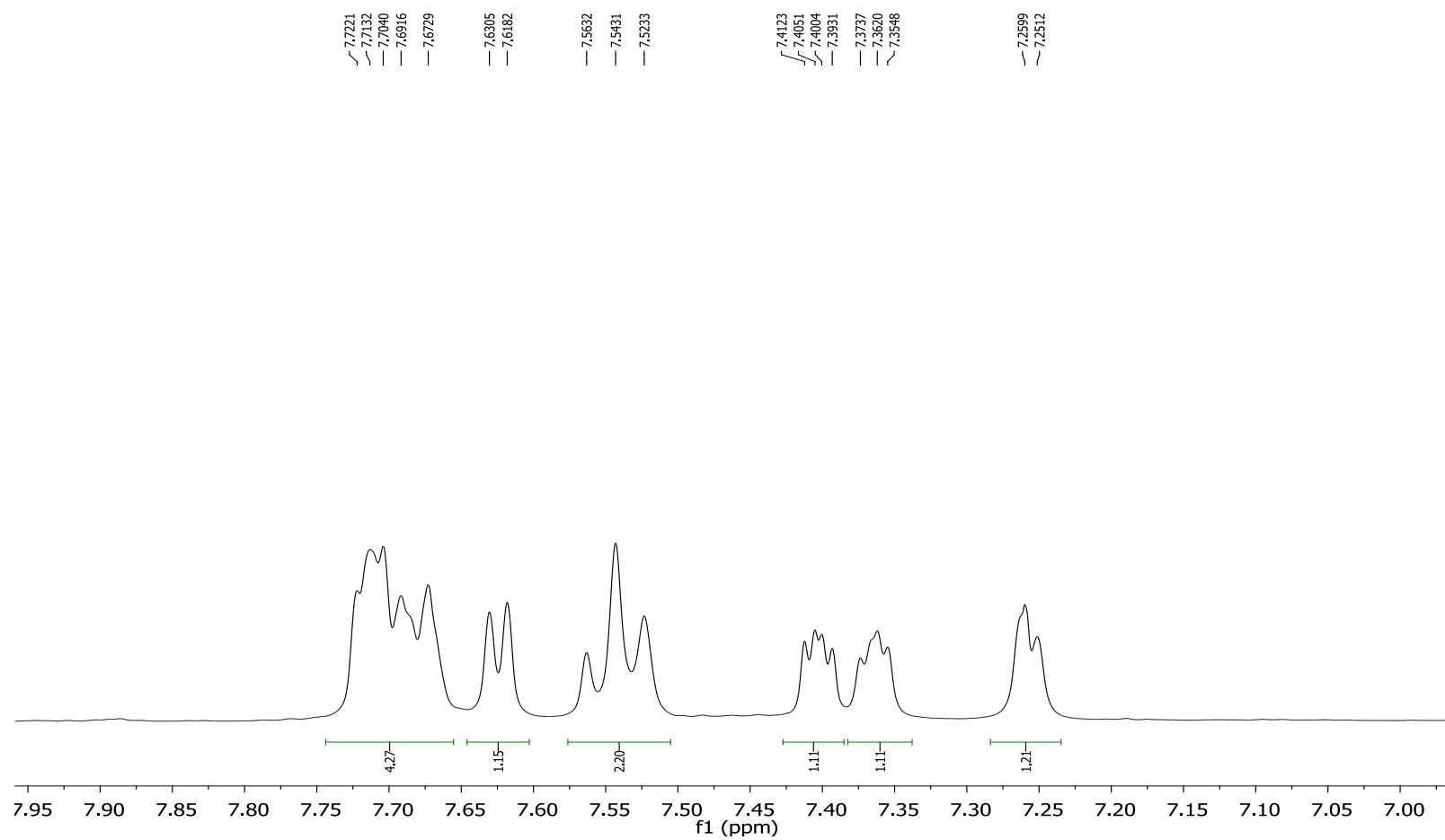
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
372.0803	1	C ₂₁ H ₁₄ N ₃ O ₂ S	372.0801	-0.4	7.4	1	100.00	16.5	even	ok	M+H

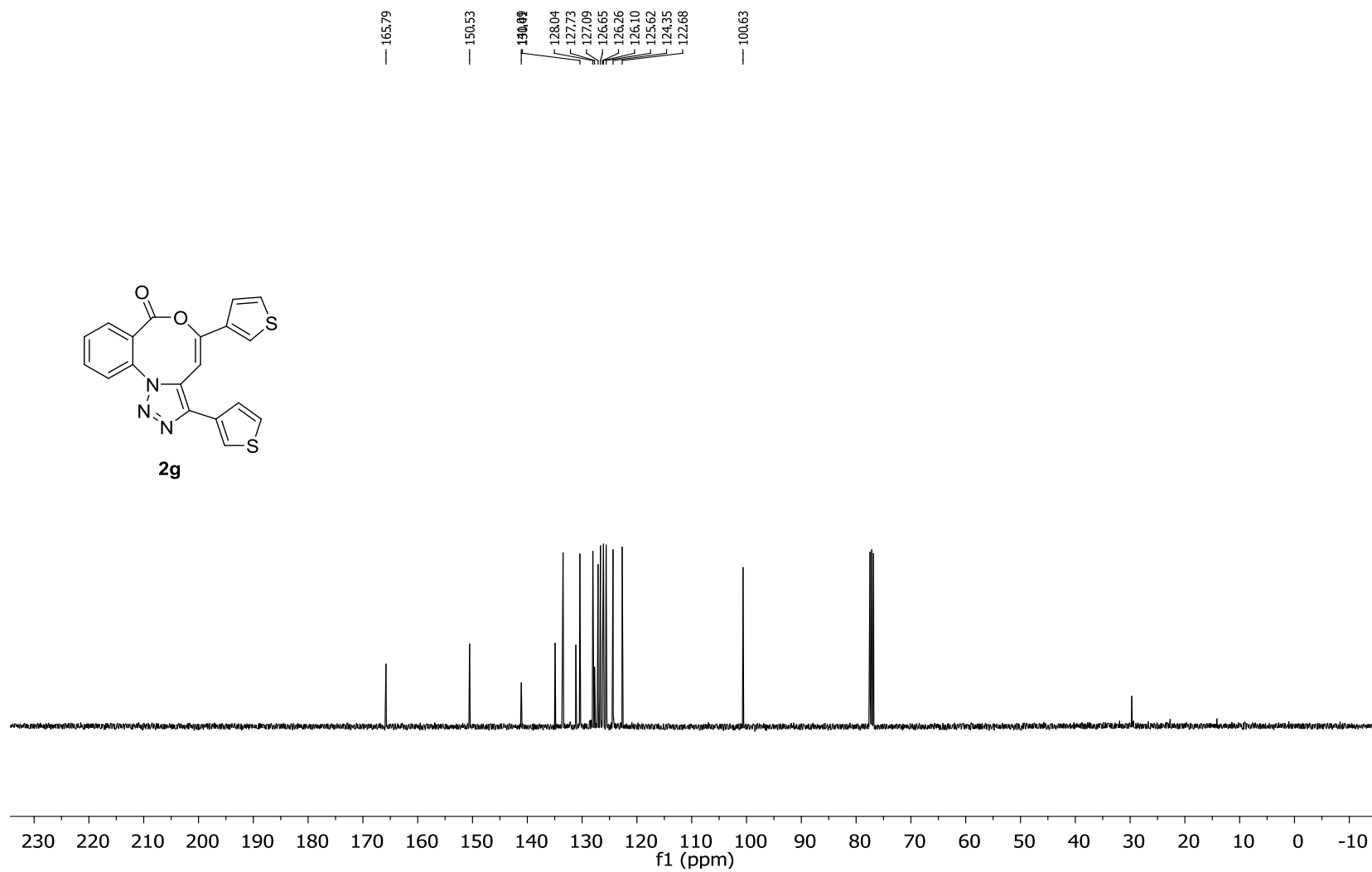
HRMS of compound 2f



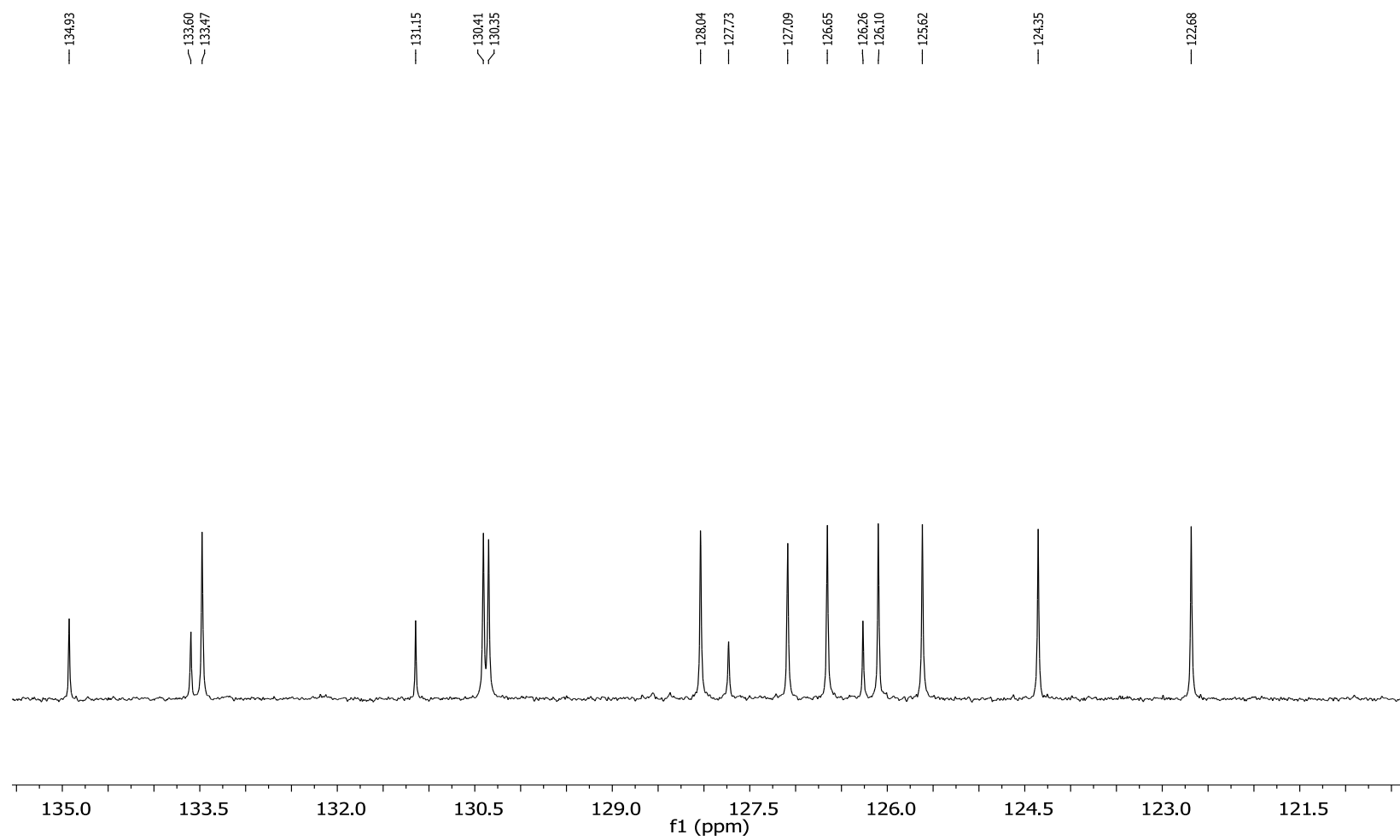
¹H NMR Spectrum (400 MHz) of compound **2g** in CDCl₃



Expansion of ^1H NMR Spectrum (400 MHz) of compound **2g** in CDCl_3



¹³C NMR Spectrum (101 MHz) of compound **2g** in CDCl₃

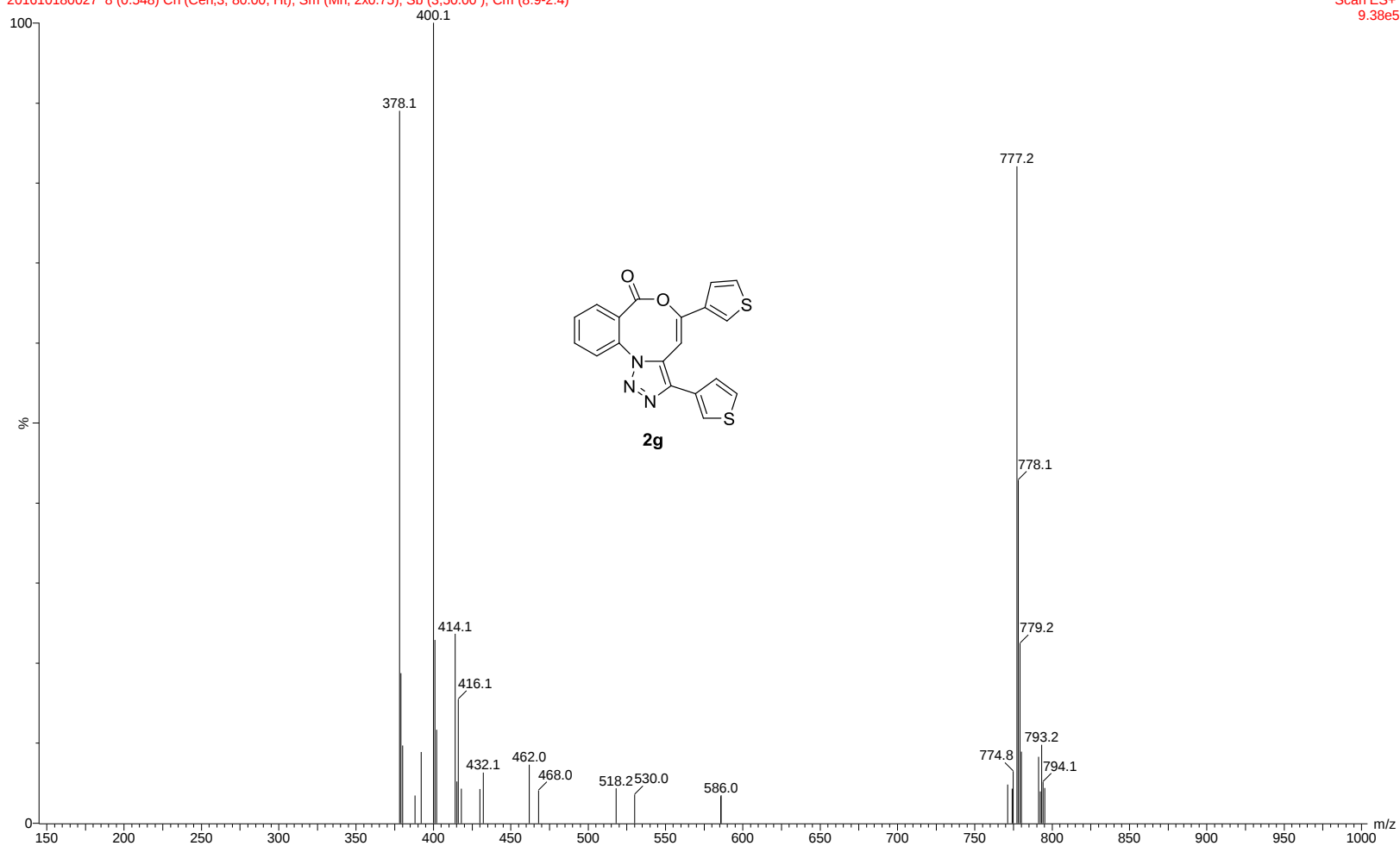


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2g** in CDCl_3

TT-LN4-94

201610180027 8 (0.548) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (8,9-2:4)

Scan ES+
9.38e5

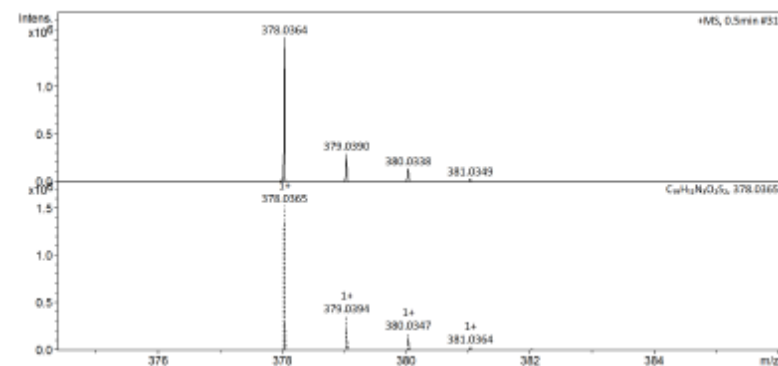
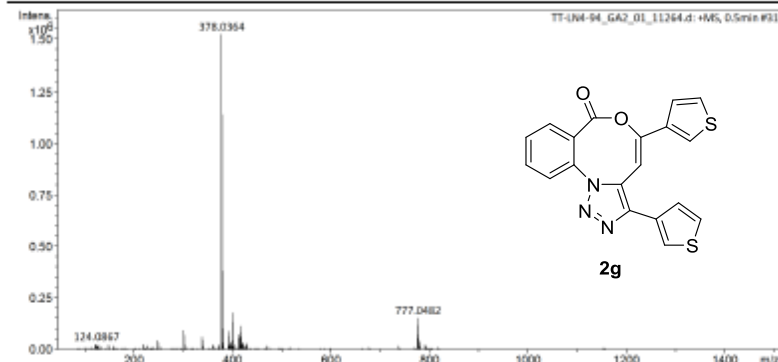


LRMS of compound **2g**

Display Report

Analytic Info		Acquisition Date	10/18/2016 10:30:03 AM	
Analysis Name	D:\Data\ncu service\data\2016\20161018\TT-LN4-94_GA2_01_11264.d			
Method	Small molecule.m		Operator	NCTU
Sample Name	TT-LN4-94		Instrument	Impact HD 1819696.00164
Comment				

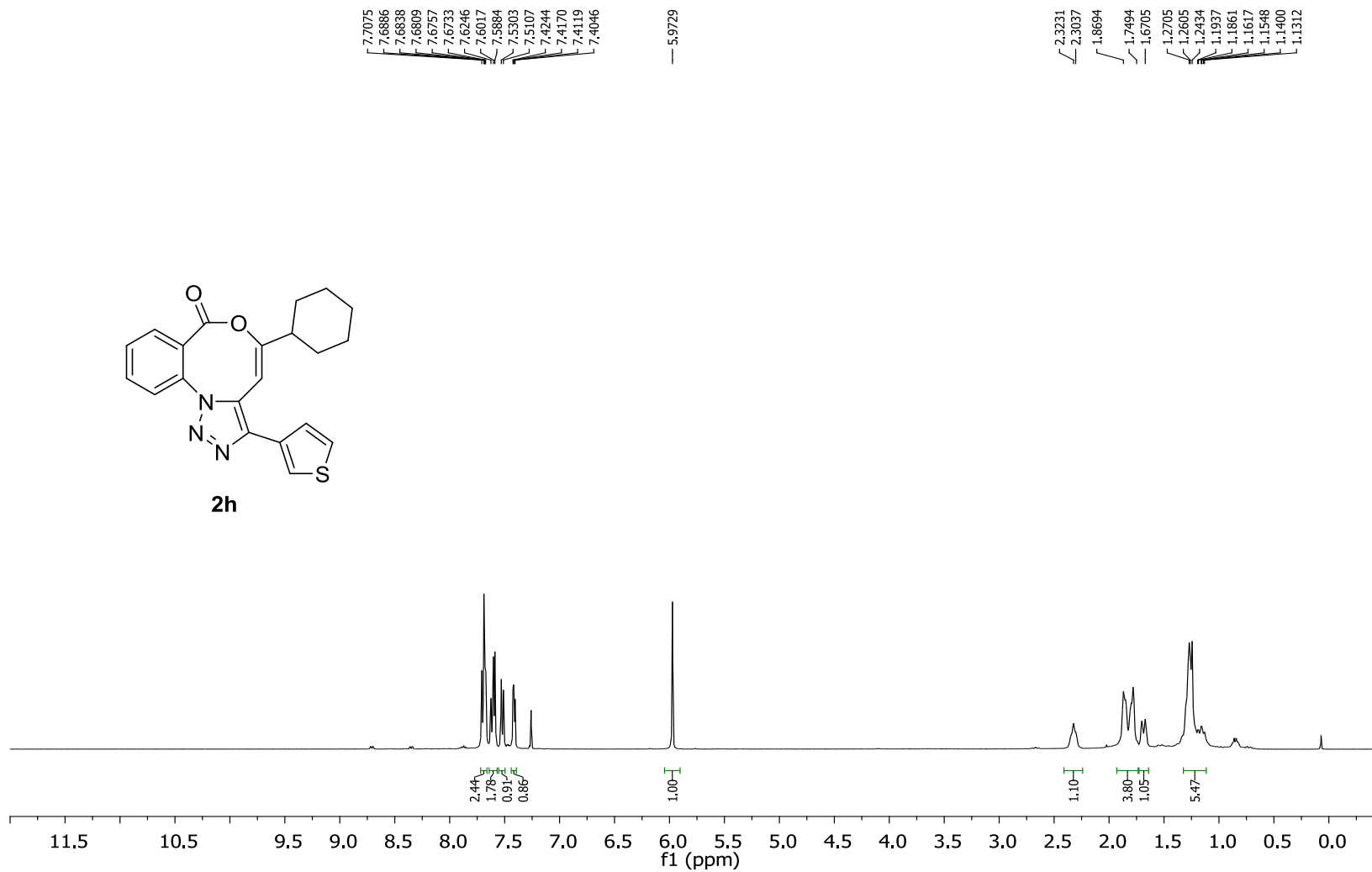
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



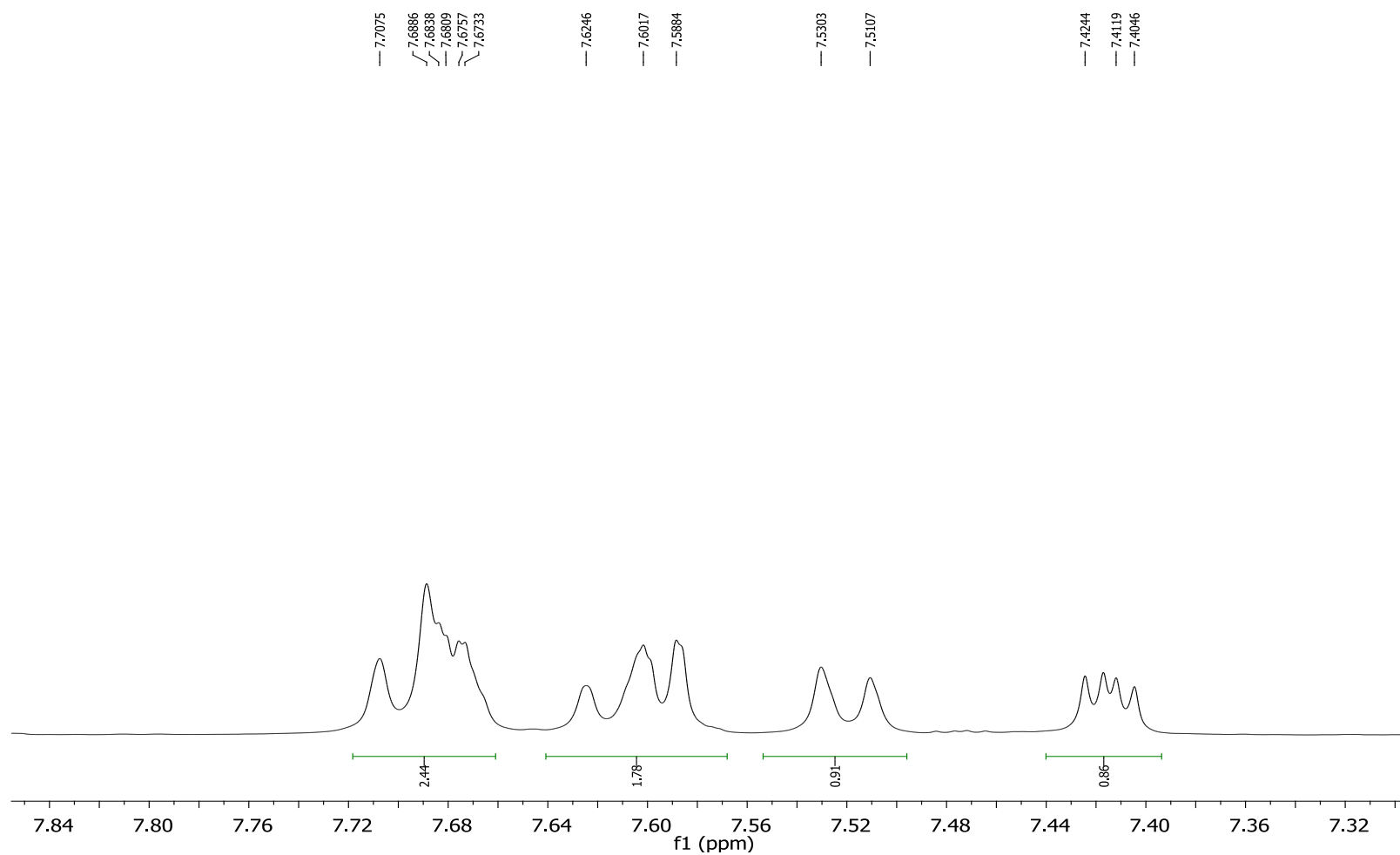
Display Report

Mass. m/z	#	Ion Formula	m/z	em [ppm]	mSigma	#Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
378.0364	1	C ₁₆ H ₁₂ N ₂ O ₂ S ₂	378.0365	0.3	22.8	1	100.00	15.5	even	ok	M+H

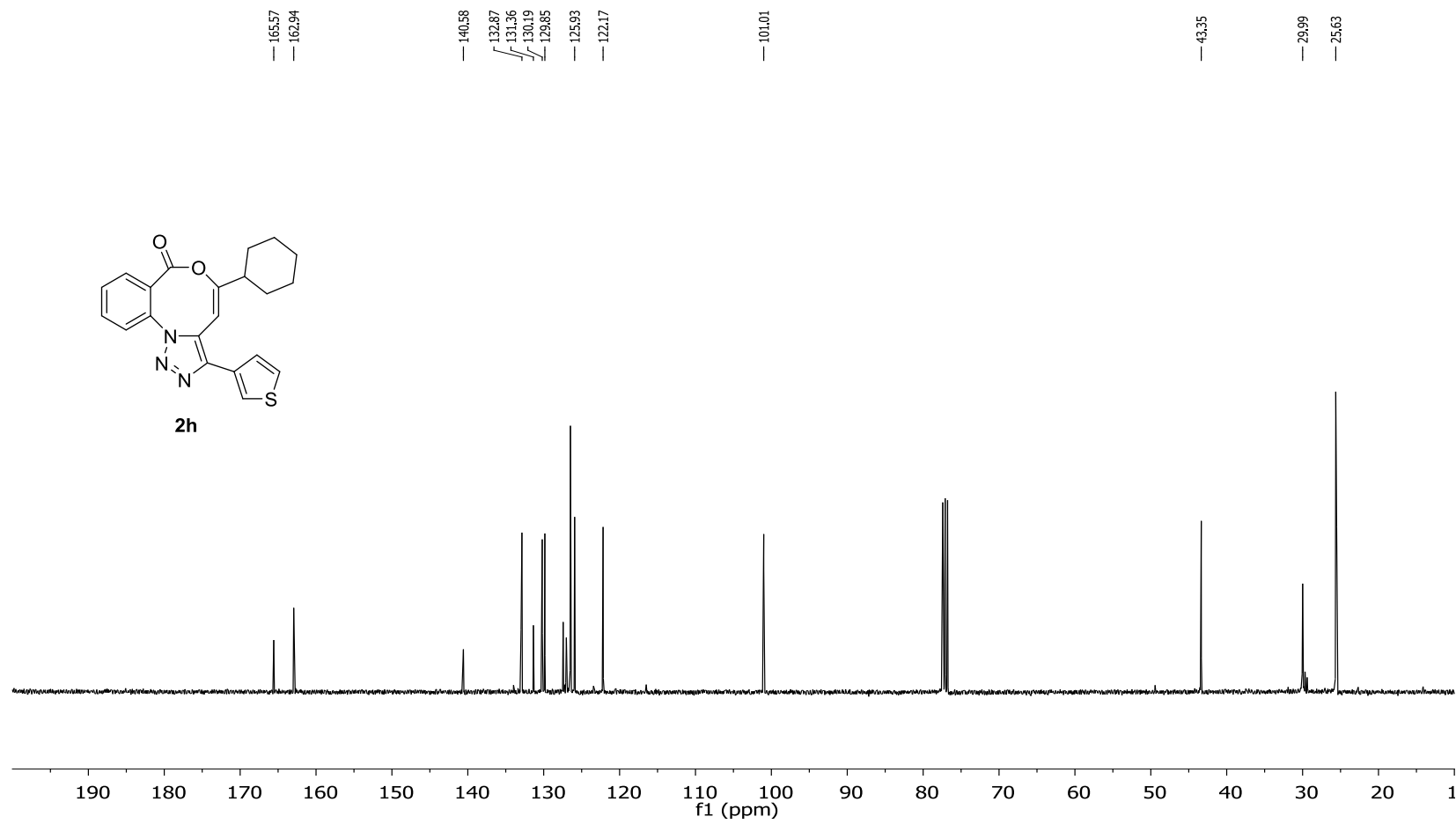
HRMS spectrum of compound **2g**



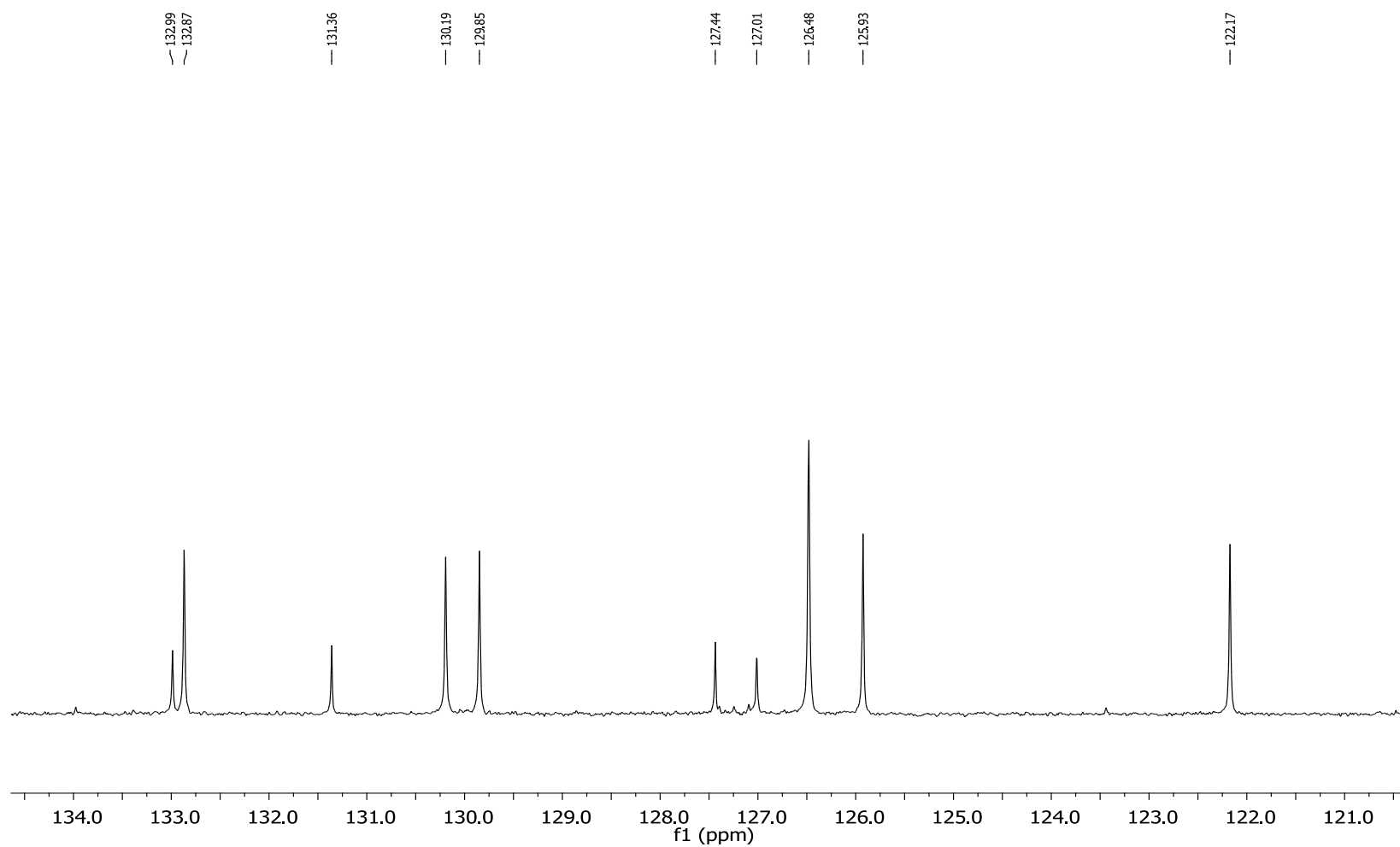
¹H NMR Spectrum (400 MHz) of compound **2h** in CDCl₃



Expansion of ^1H NMR Spectrum (400 MHz) of compound **2h** in CDCl_3



¹³C NMR Spectrum (101 MHz) of compound **2h** in CDCl₃

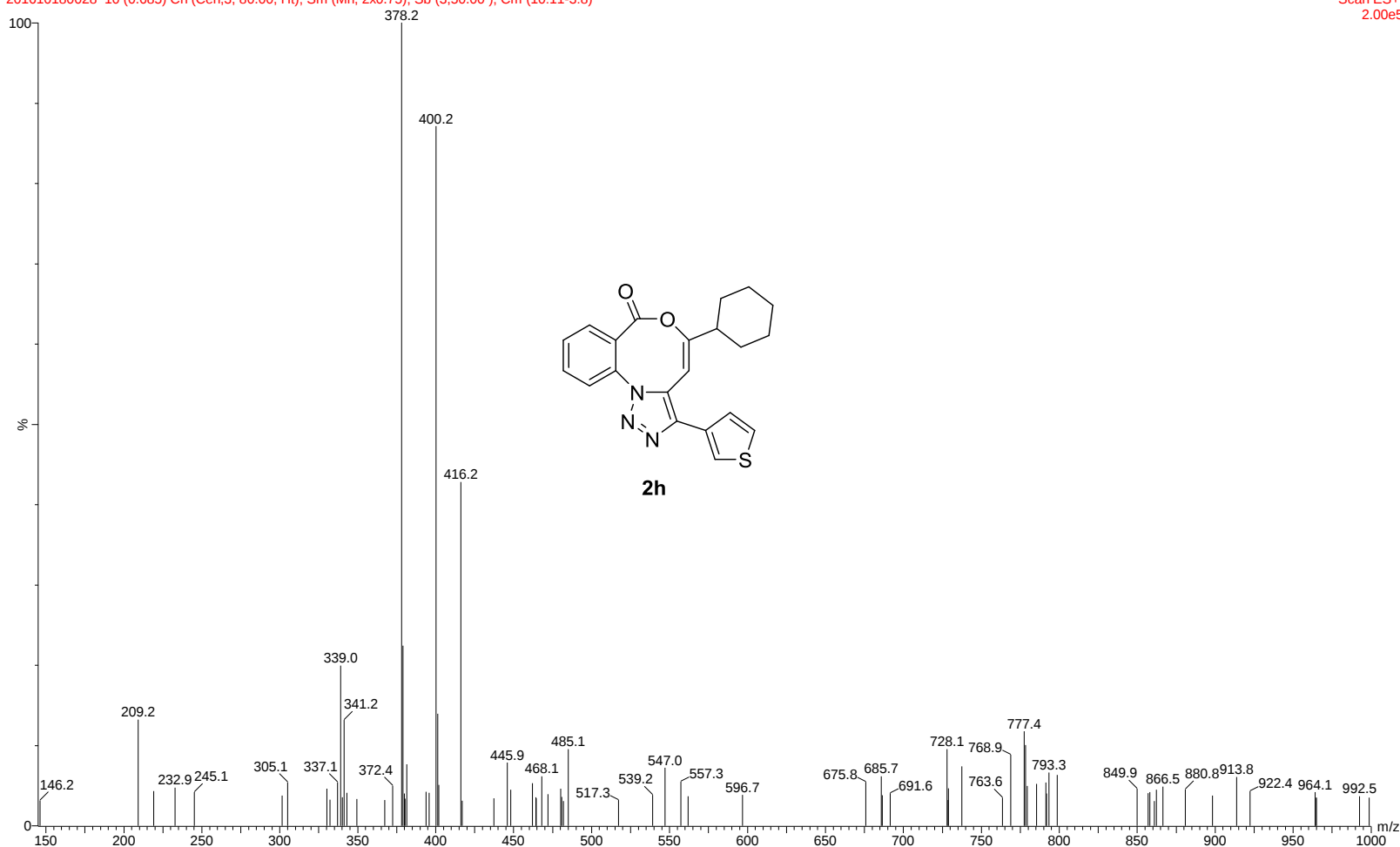


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2h** in CDCl_3

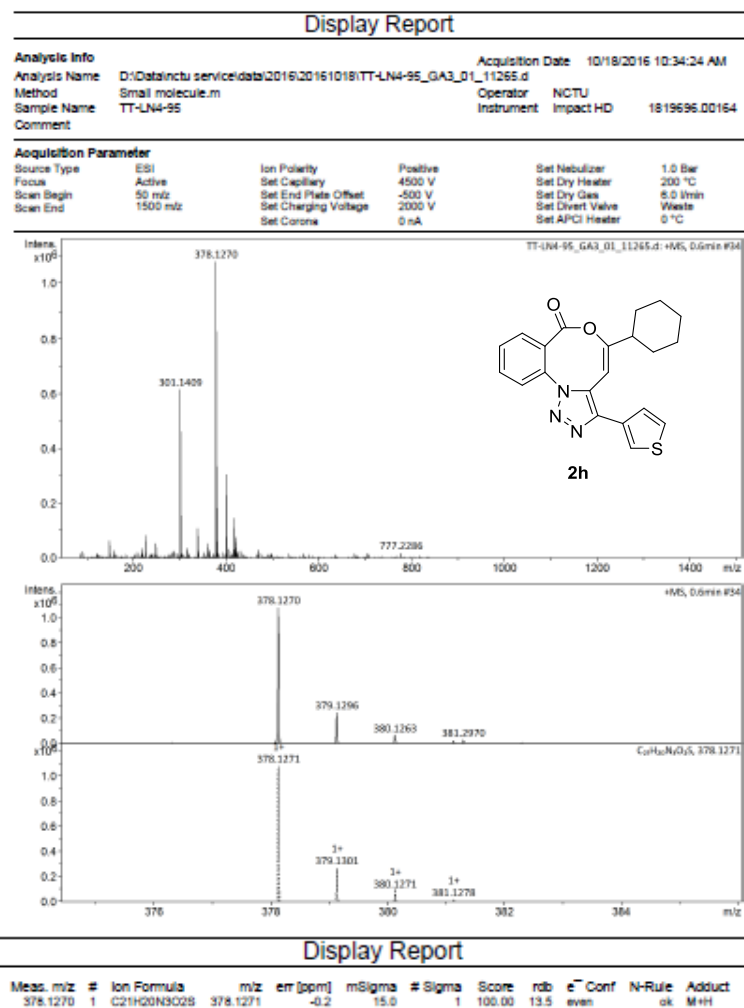
TT-LN4-95

201610180028 10 (0.685) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (10:11:3:8)

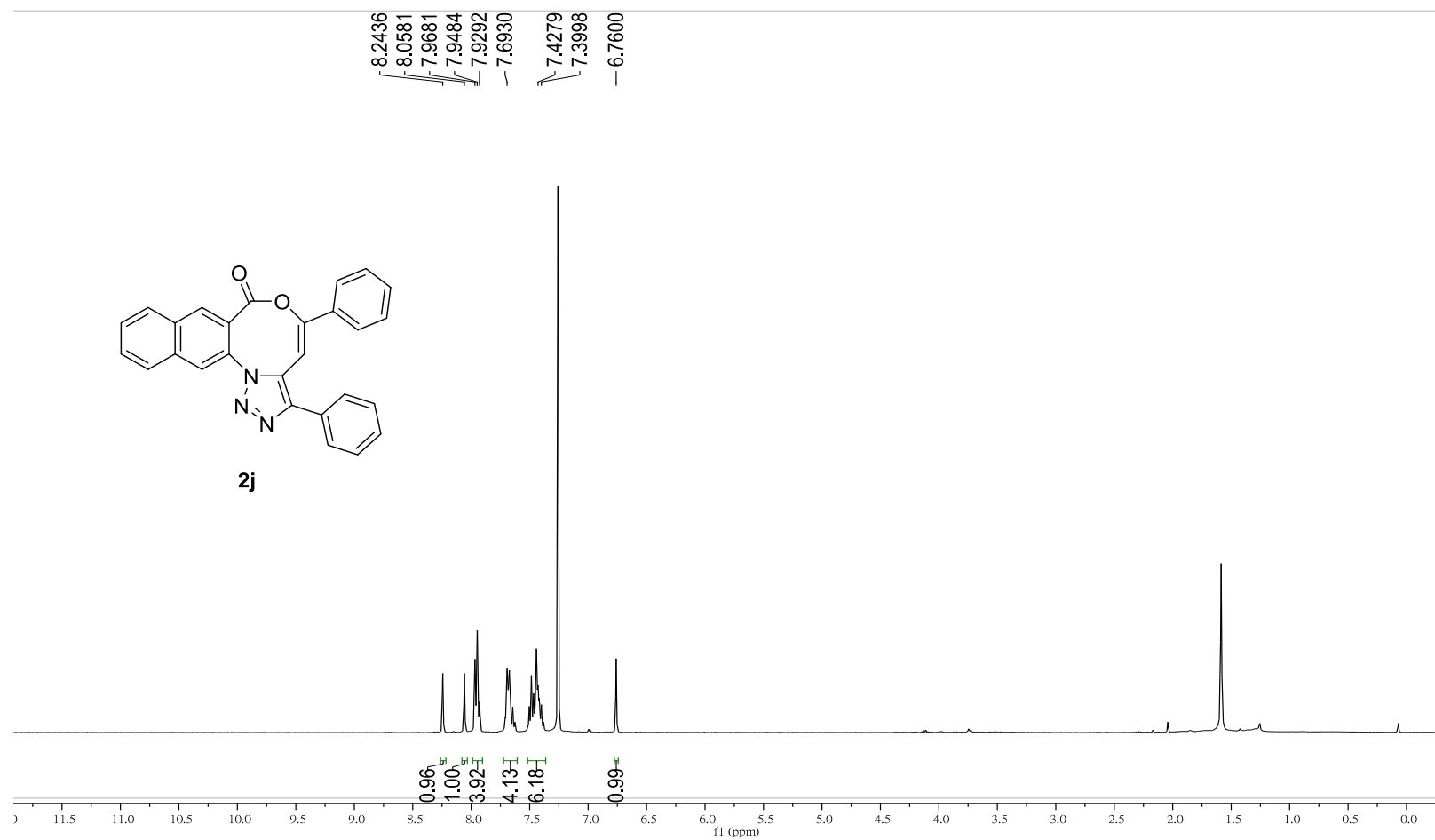
Scan ES+
2.00e5



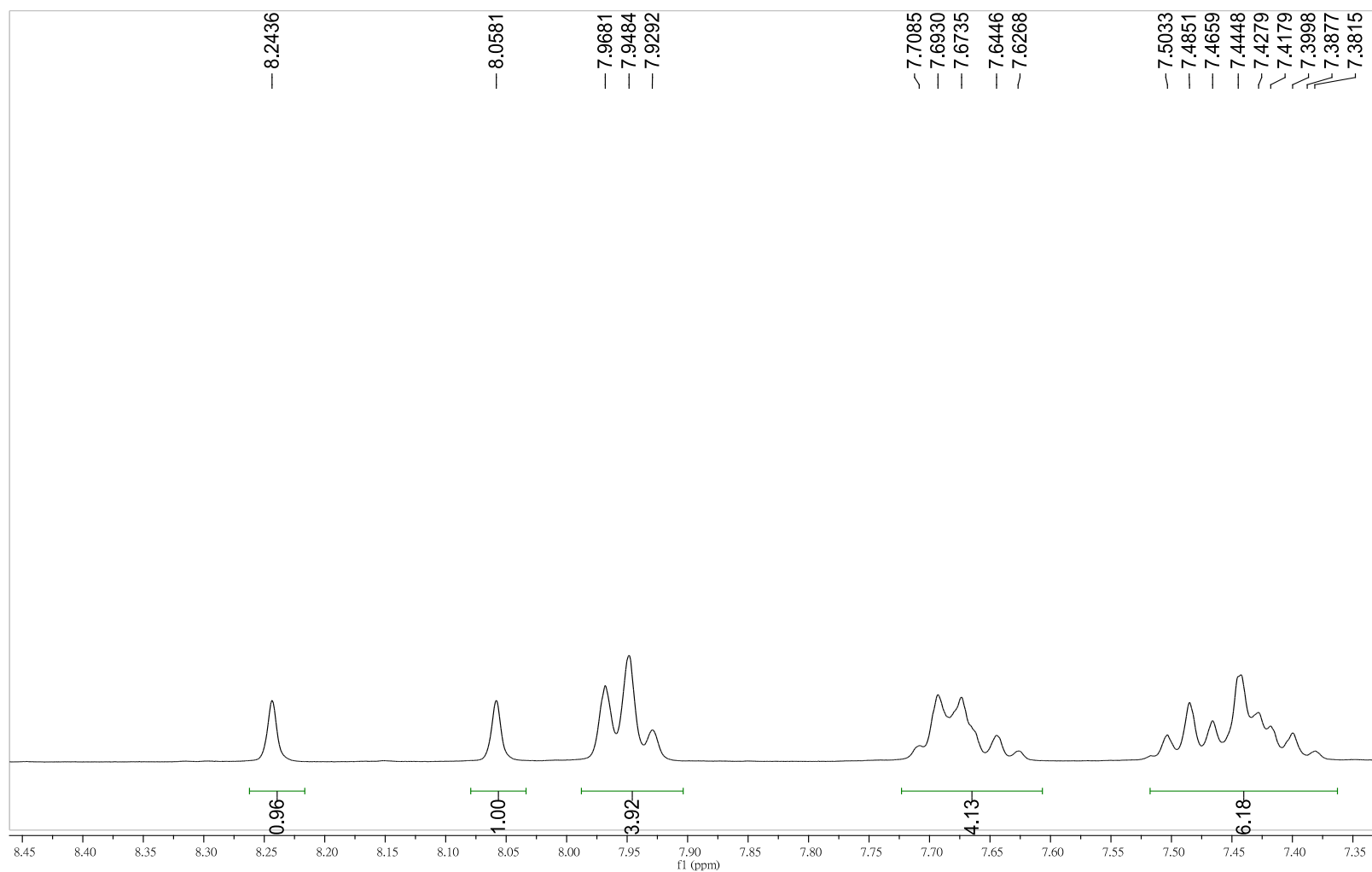
LRMS of compound **2h**



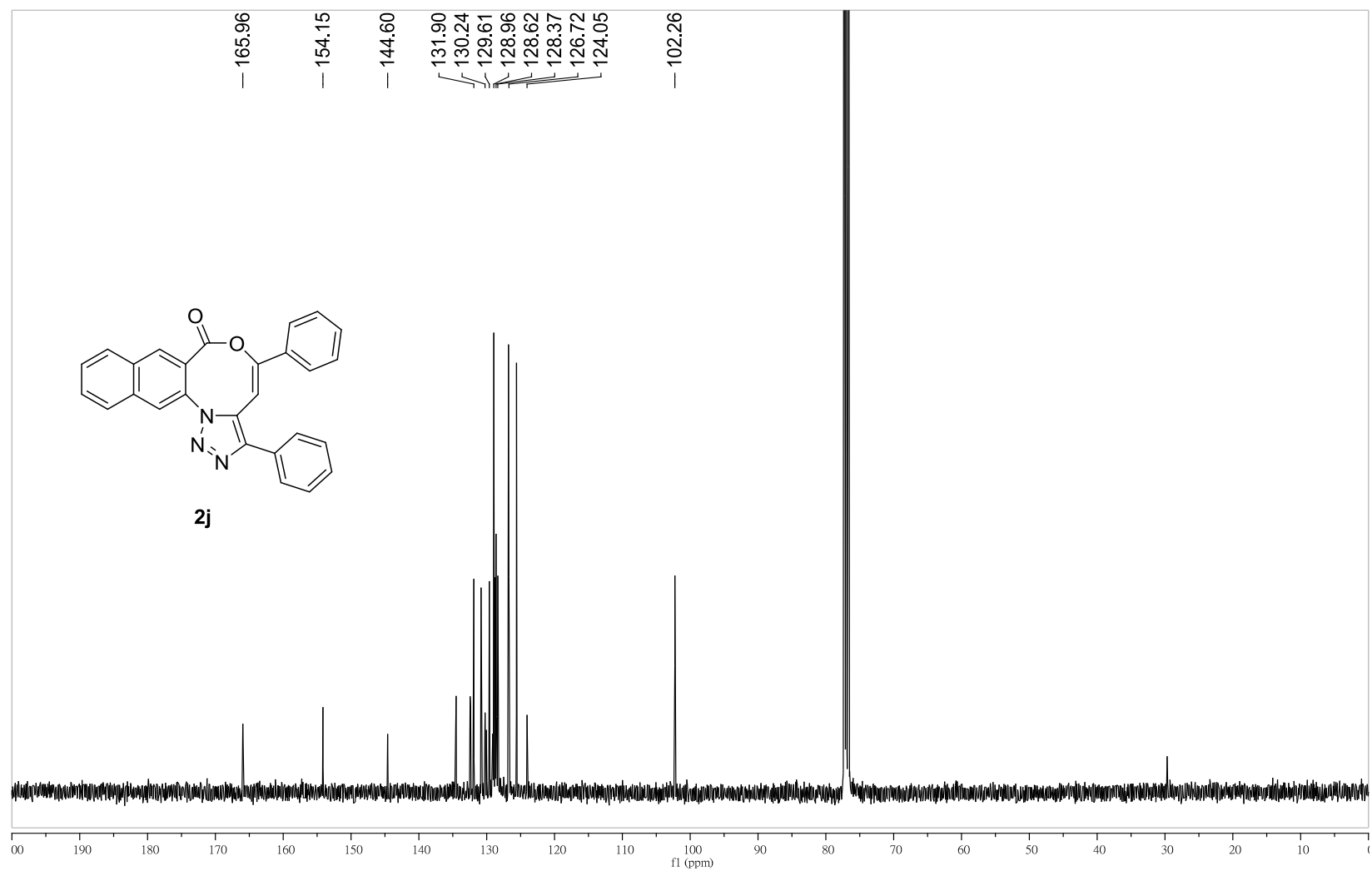
HRMS of compound **2h**



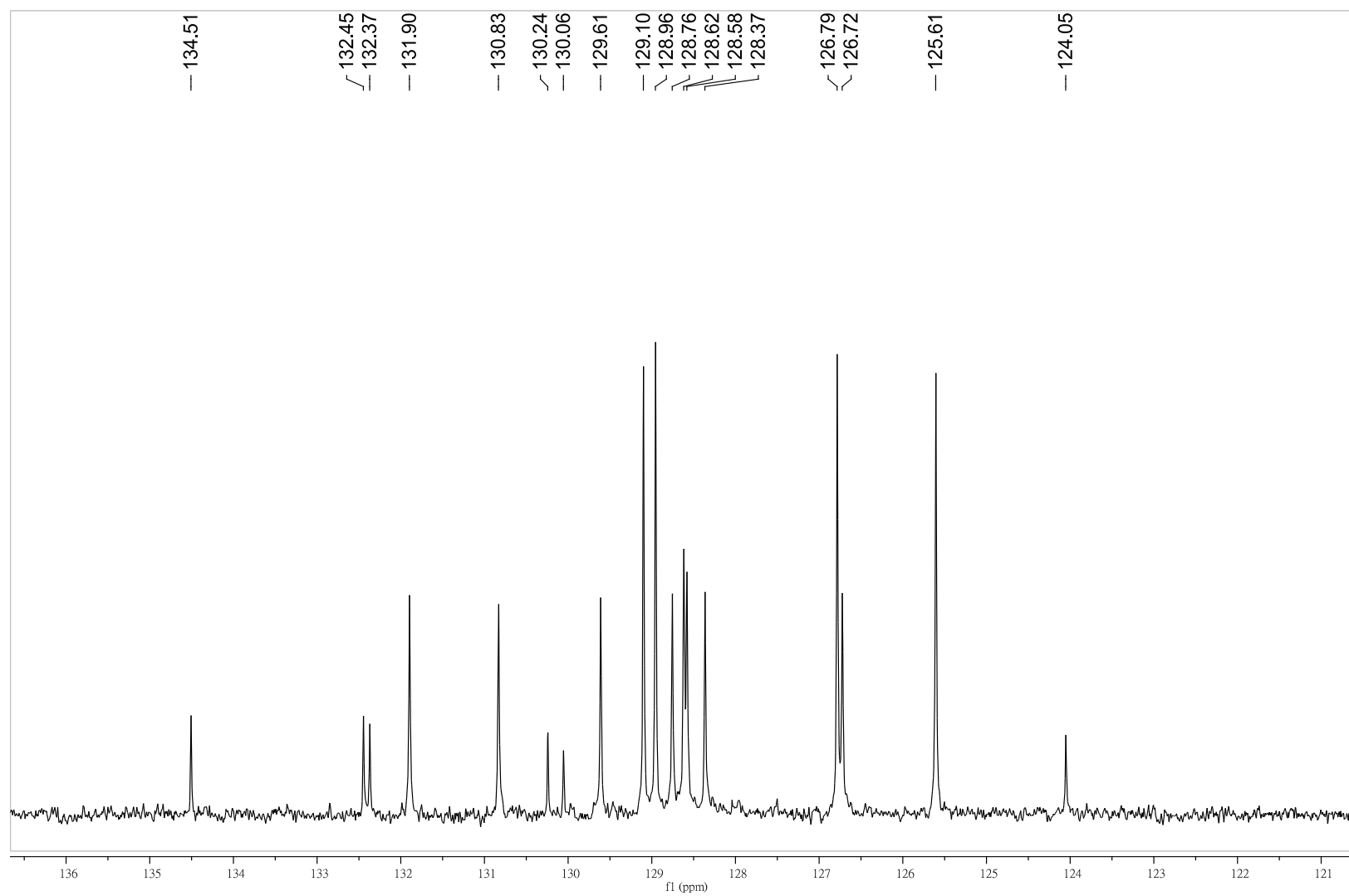
¹H NMR Spectrum (400 MHz) of compound **2j** in CDCl₃



Expansion of ^1H NMR Spectrum (400 MHz) of compound **2j** in CDCl_3



^{13}C NMR Spectrum (101 MHz) of compound **2j** in CDCl_3

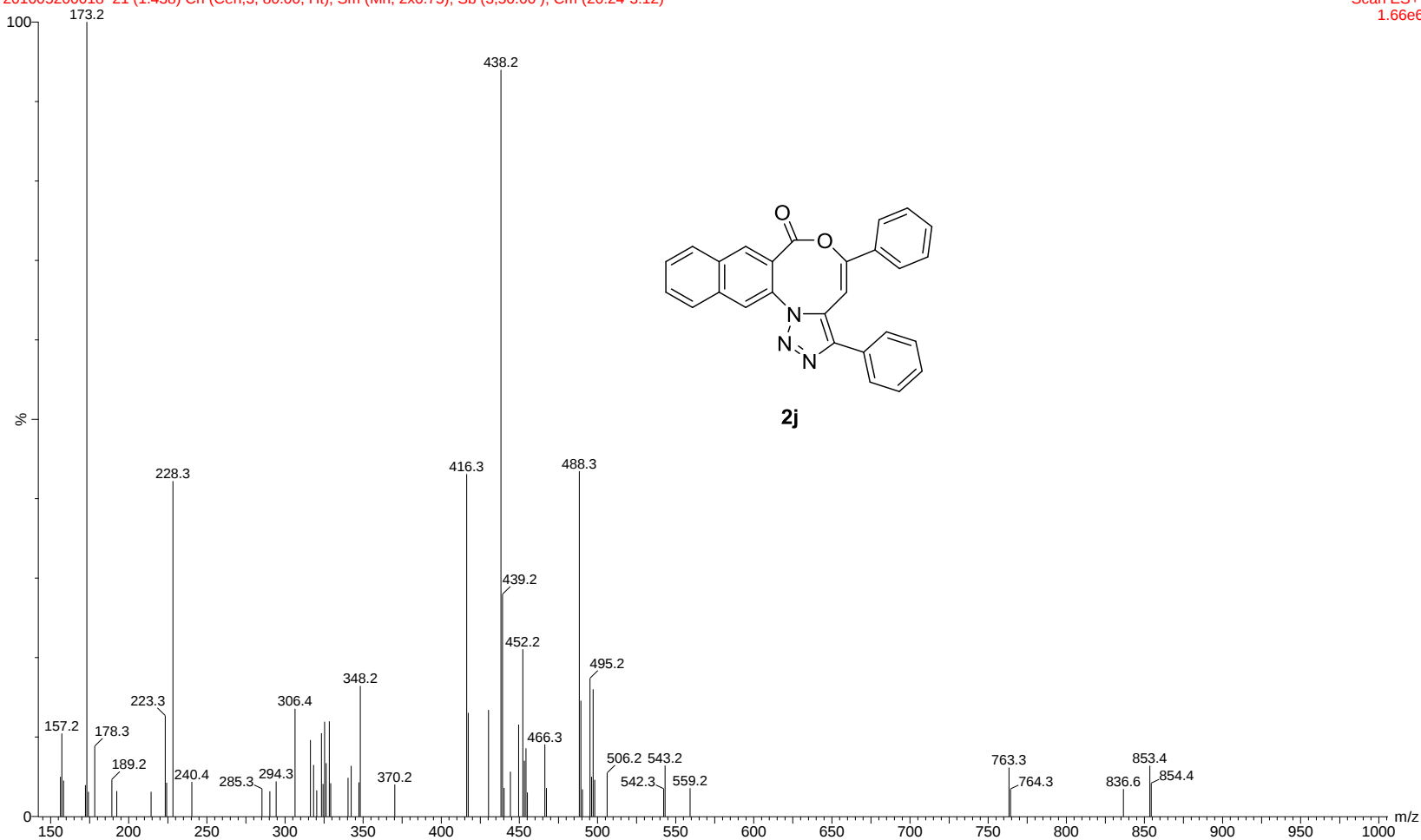


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2j** in CDCl_3

3p12-13

201605200018 21 (1.438) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (20:24-3:12)

Scan ES+
1.66e6



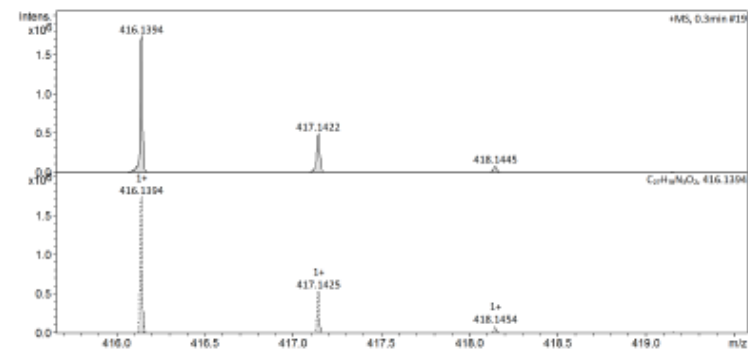
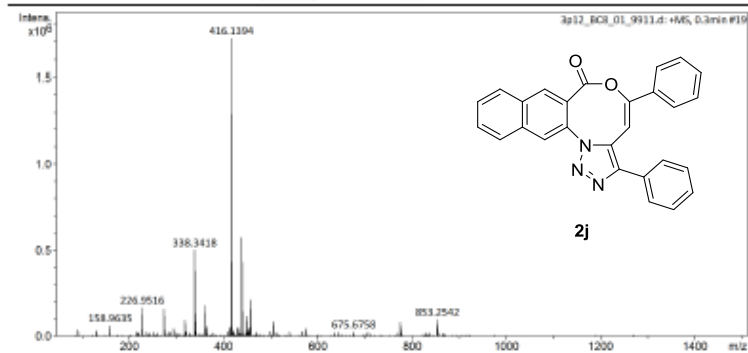
LRMS of compound 2j

Display Report

Analysis Info		Acquisition Date	5/27/2016 1:21:09 PM	
Analysis Name	D:\Data\inctu service\data\2016\20160527\3p12_BC08_01_9911.d		Operator	NCTU
Method	Small molecule.m		Instrument	Impact HD
Sample Name	3p12			1819696.00164
Comment				

Acquisition Parameter

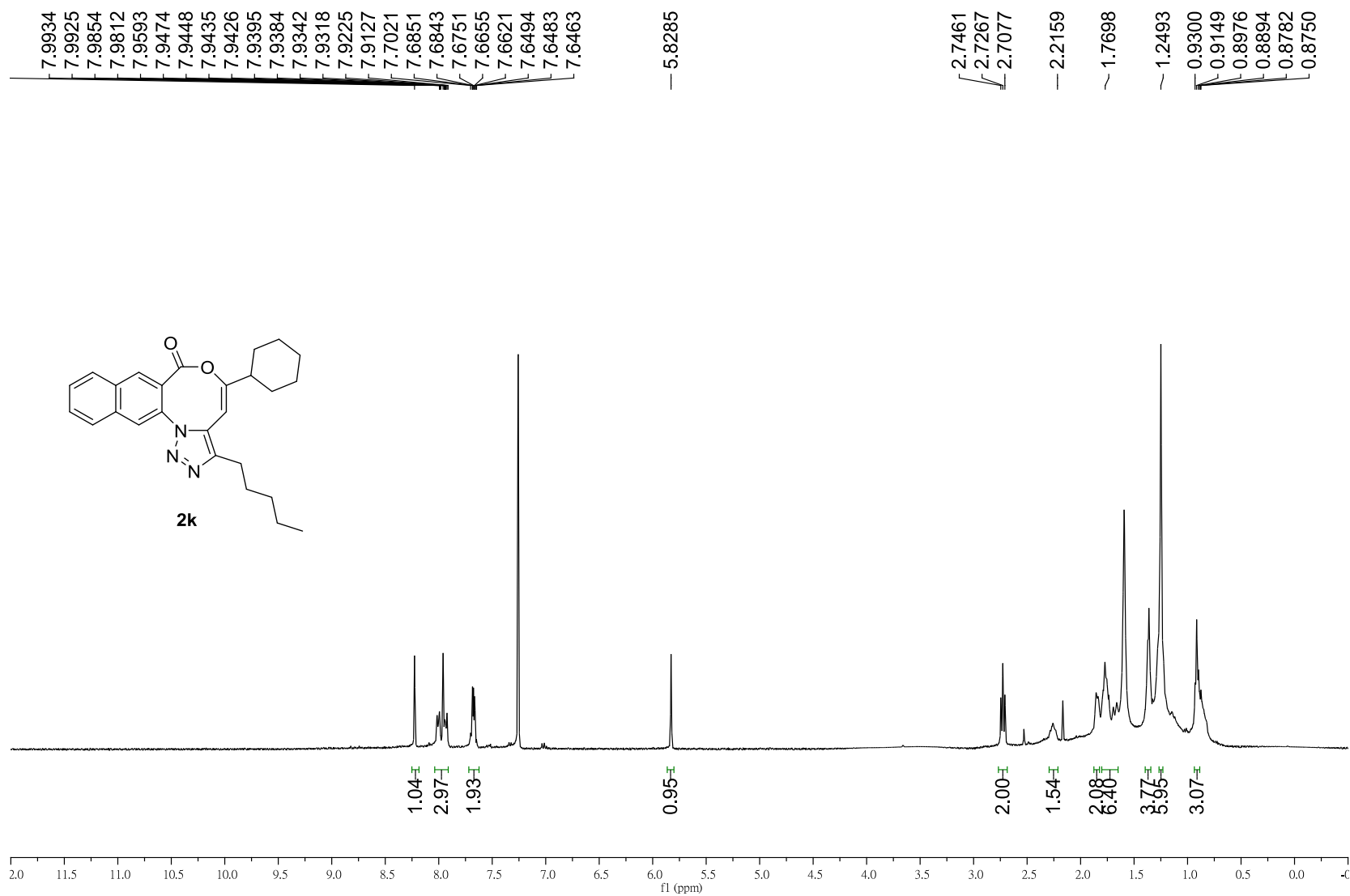
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



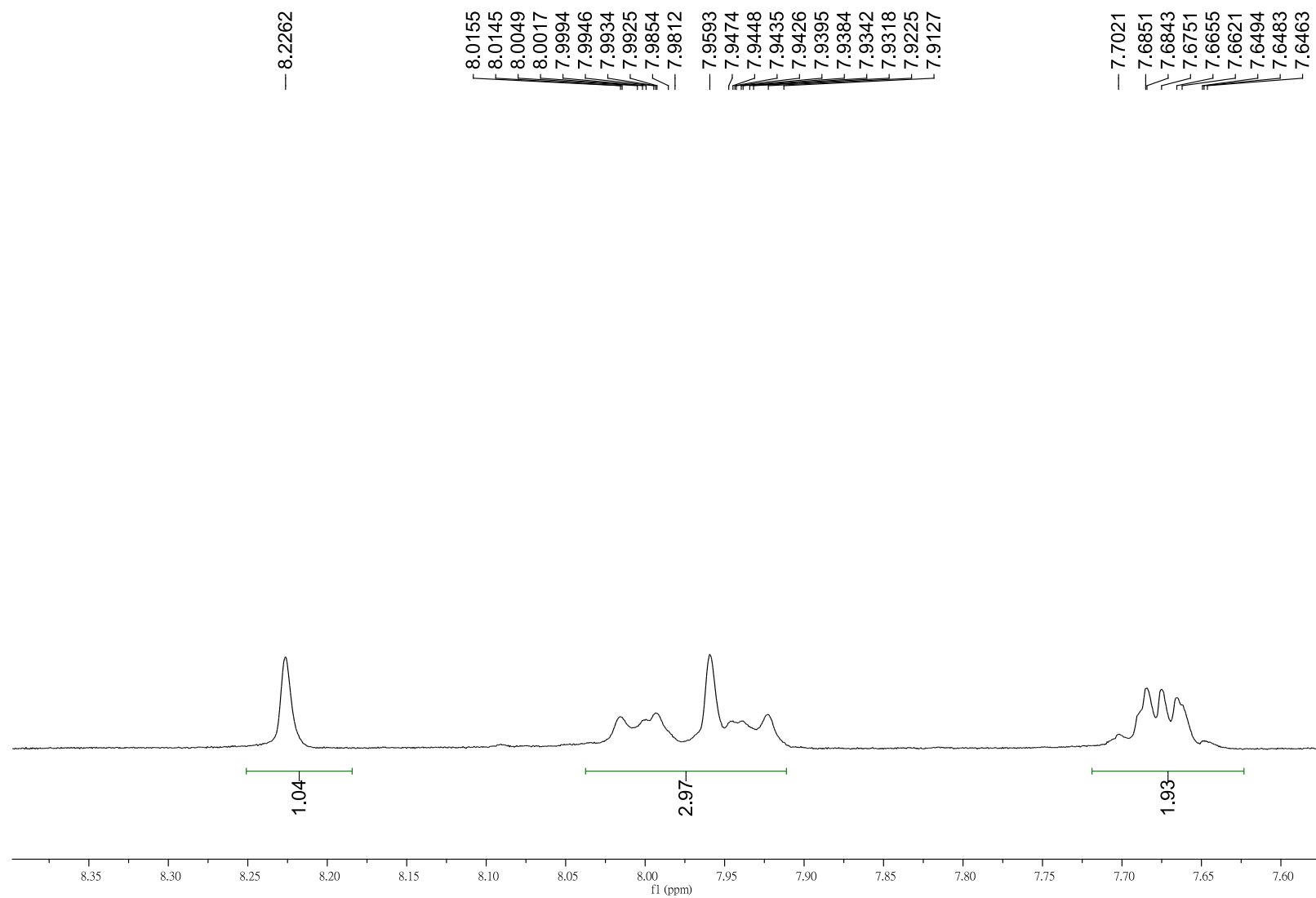
Display Report

Mass. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
416.1394	1	C ₂₇ H ₁₈ N ₃ O ₂	416.1394	-0.1	12.1	1	100.00	20.5	even	ok	M+H

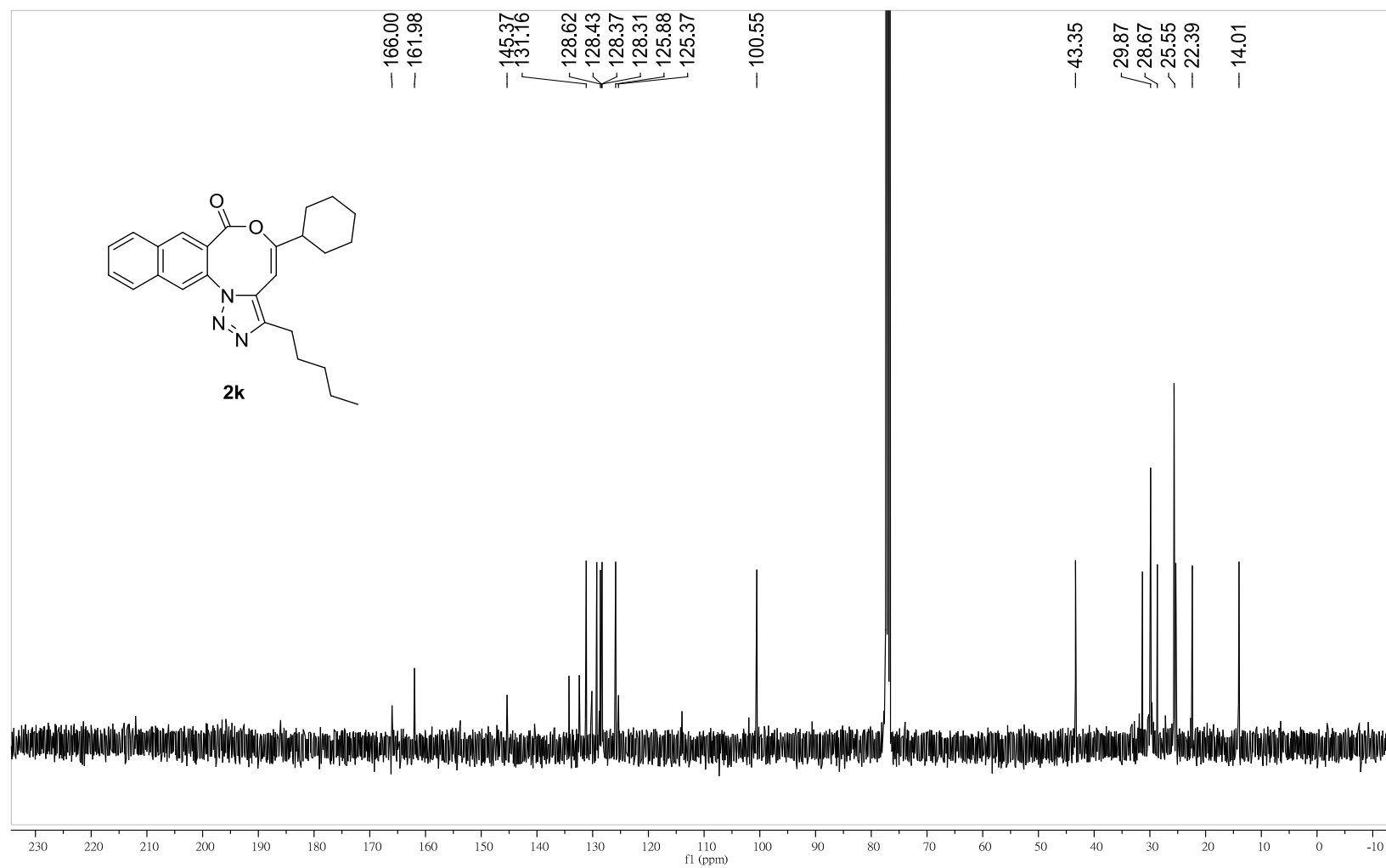
HRMS of compound 2j



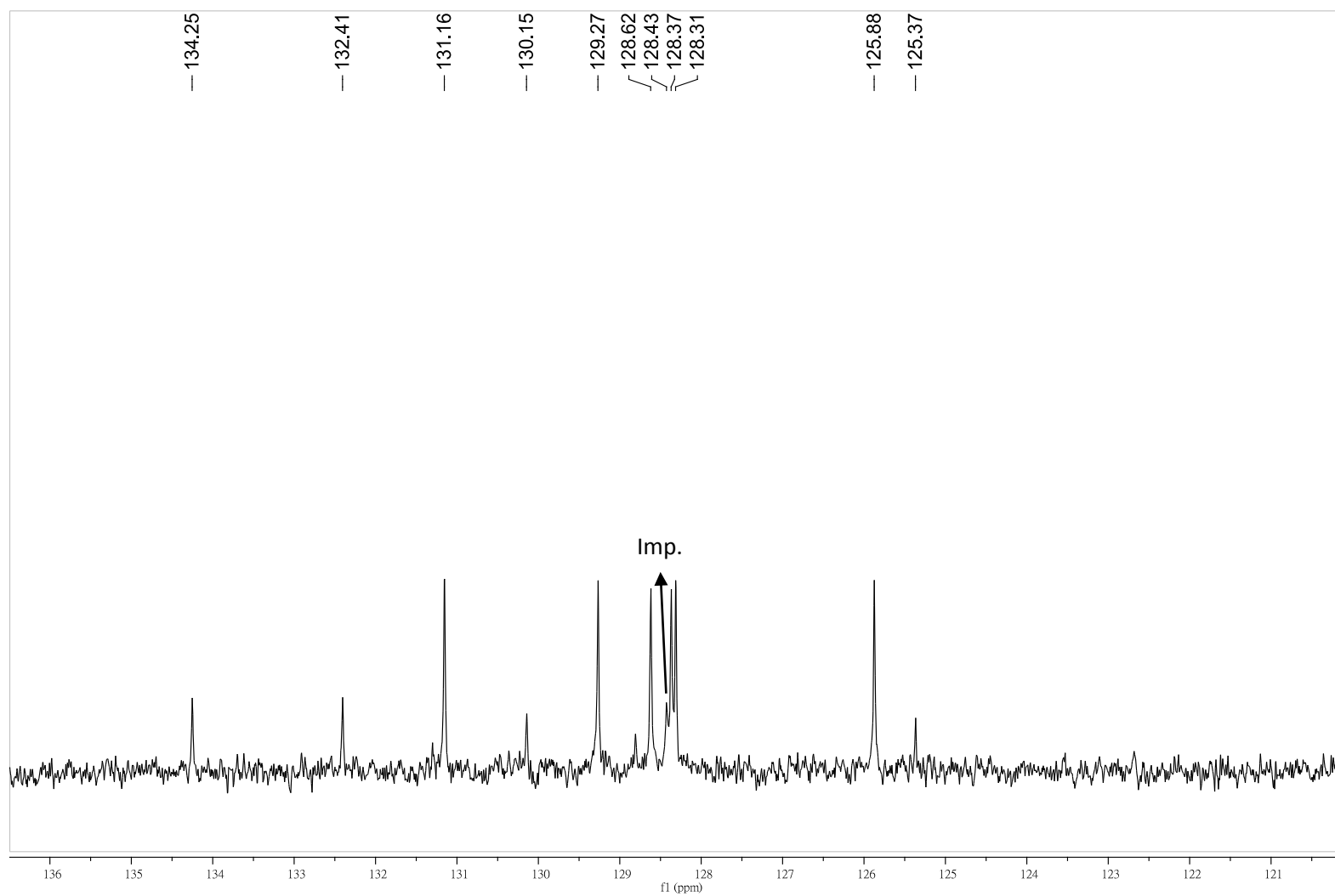
¹H NMR Spectrum (300 MHz) of compound **2k** in CDCl₃



Expansion of ^1H NMR Spectrum (300 MHz) of compound **2k** in CDCl_3



^{13}C NMR Spectrum (75 MHz) of compound **2k** in CDCl_3

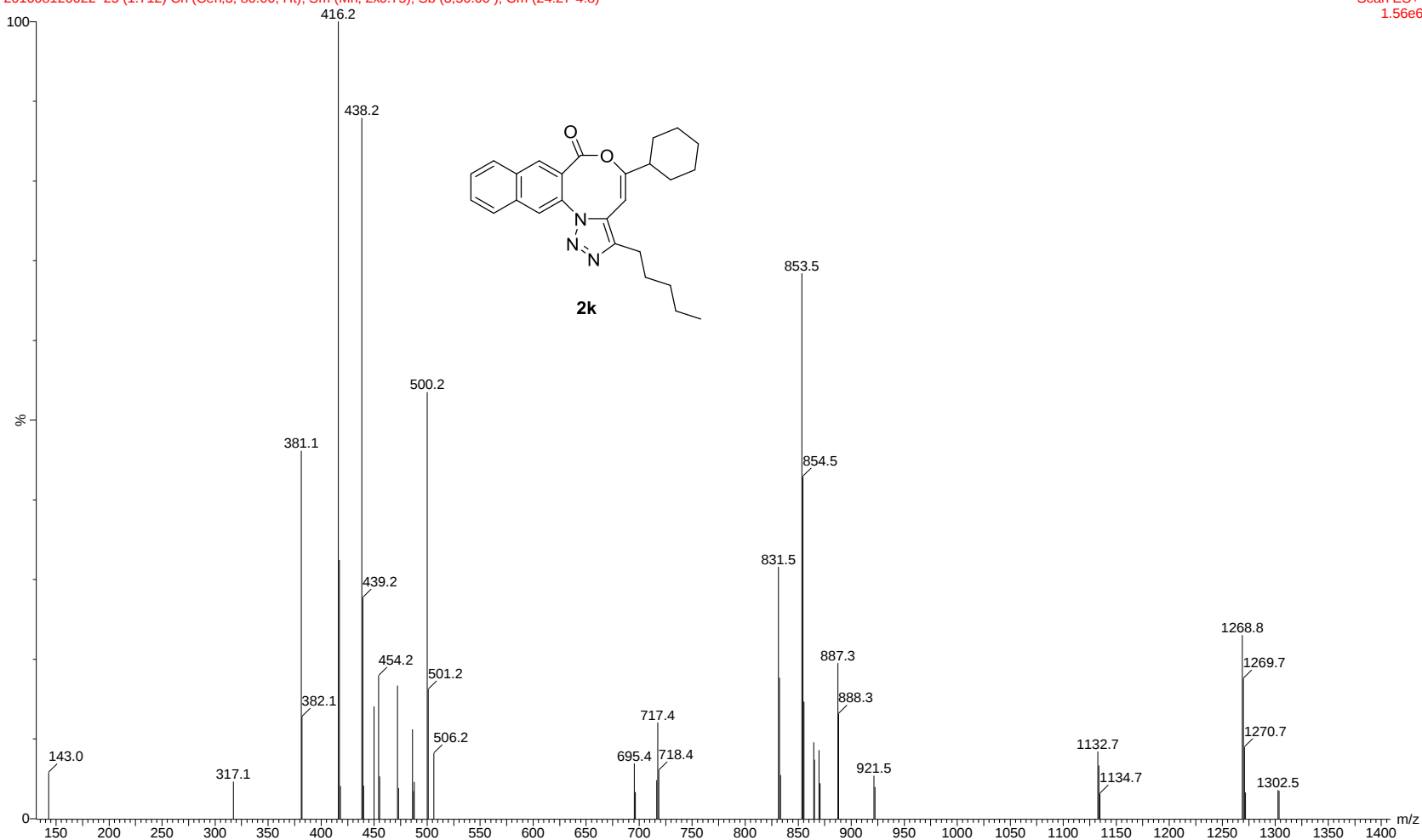


Expansion of ^{13}C NMR Spectrum (75 MHz) of compound **2k** in CDCl_3

3p53-Ag

201608120022 25 (1.712) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (24:27-4:8)

Scan ES+
1.56e6



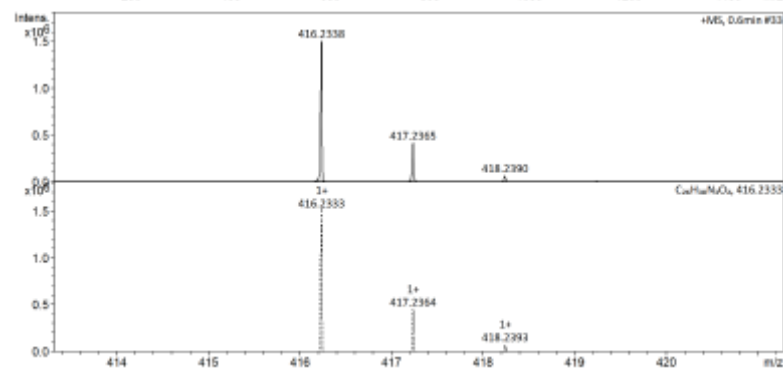
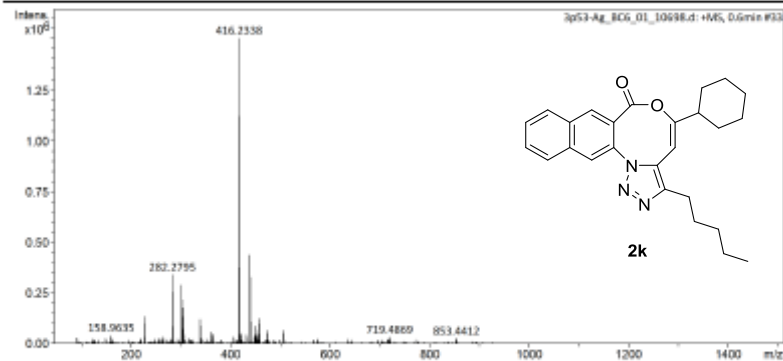
LRMS of compound **2k**

Display Report

Analytic Info		Acquisition Date	8/16/2016 10:26:59 AM	
Analysis Name	D:\Data\ncu_service\data\2016\20160816\3p53-Ag_BO6_01_10698.d		Operator	NCTU
Method	Small molecule.m	Instrument	Impact HD	1819696.00164
Sample Name	3p53-Ag			
Comment				

Acquisition Parameter

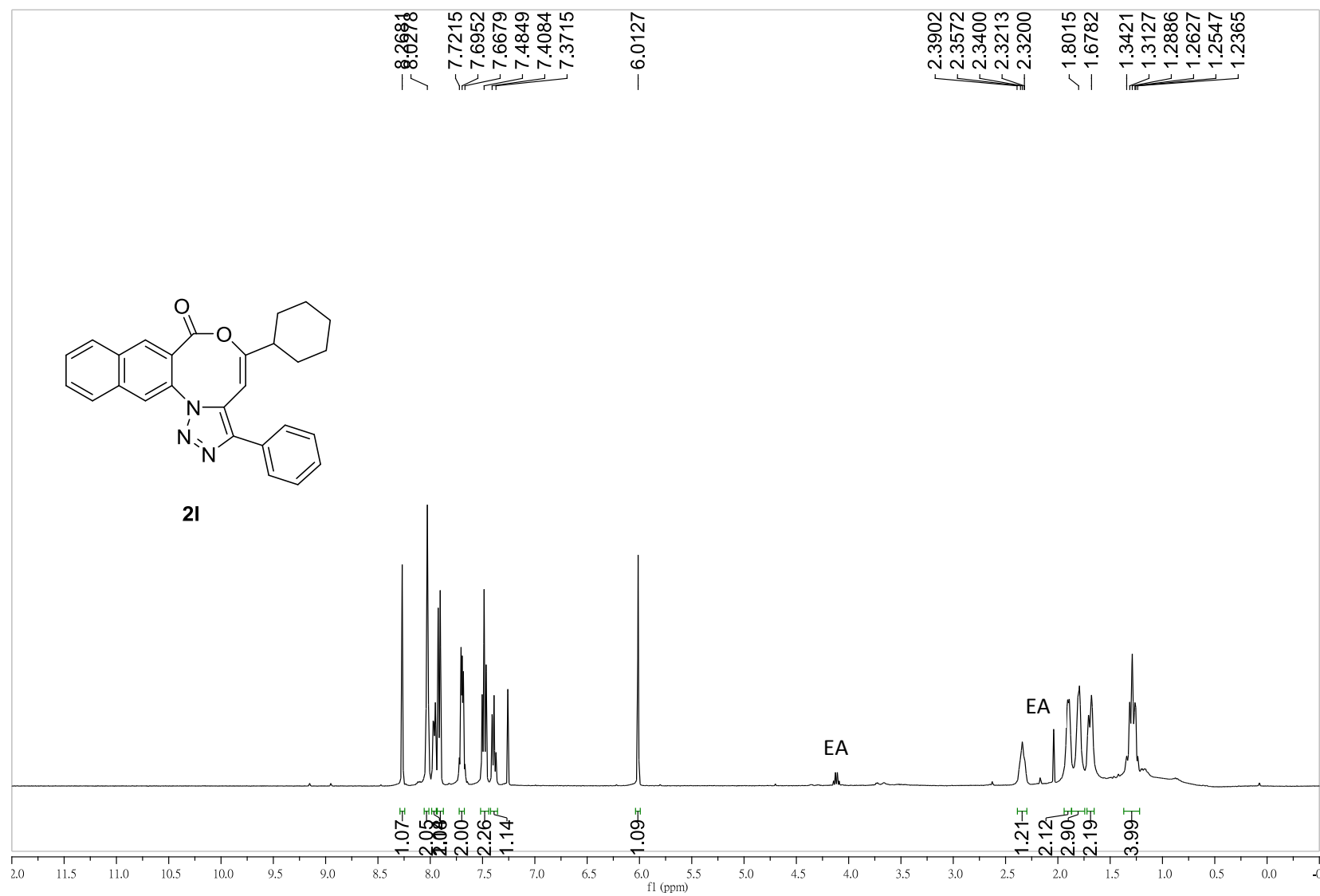
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



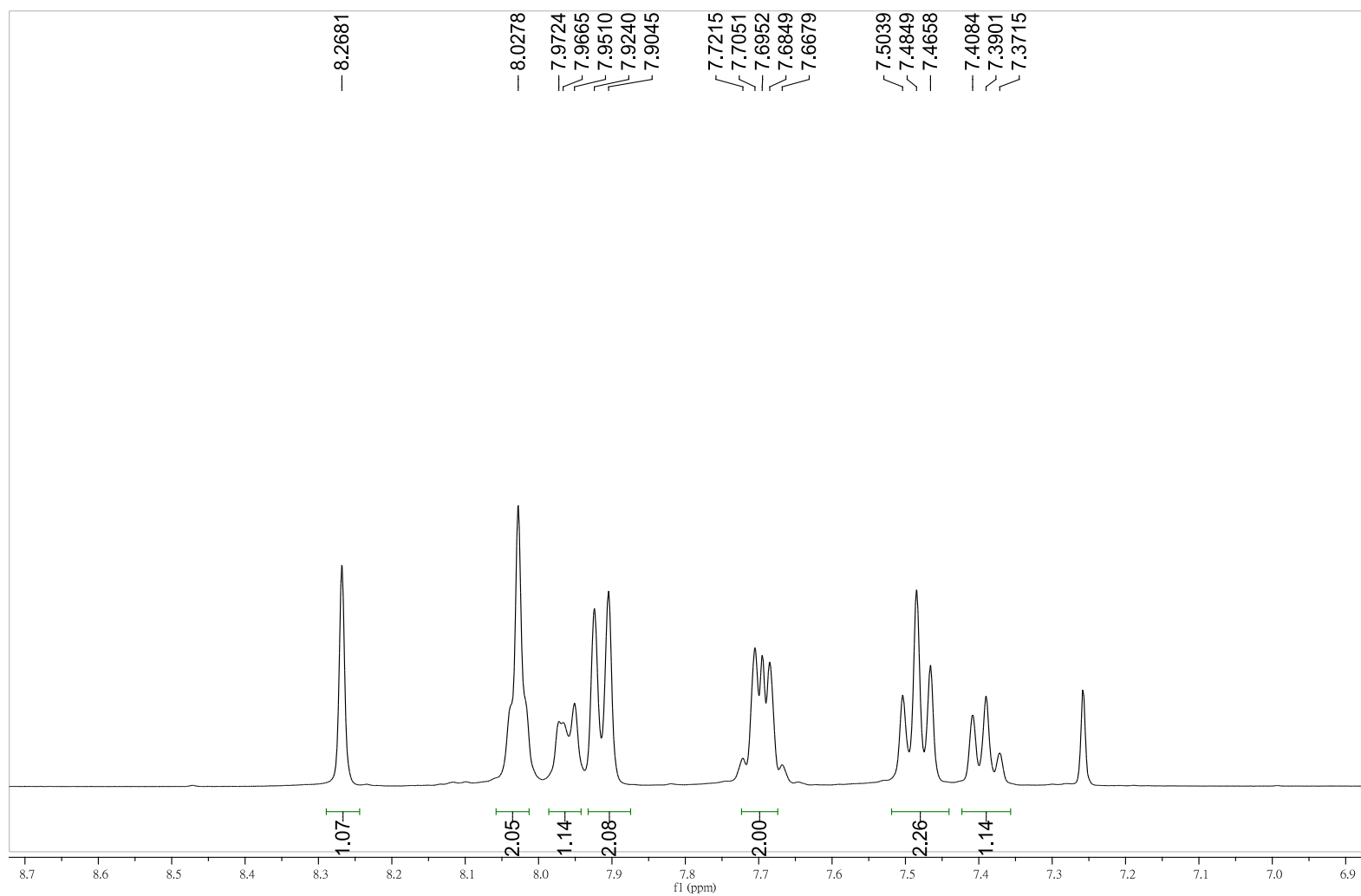
Display Report

Mass. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
416.2338	1	C ₂₈ H ₃₀ N ₃ O ₂	416.2333	-1.2	10.3	1	100.00	13.5	even	ok	M+H

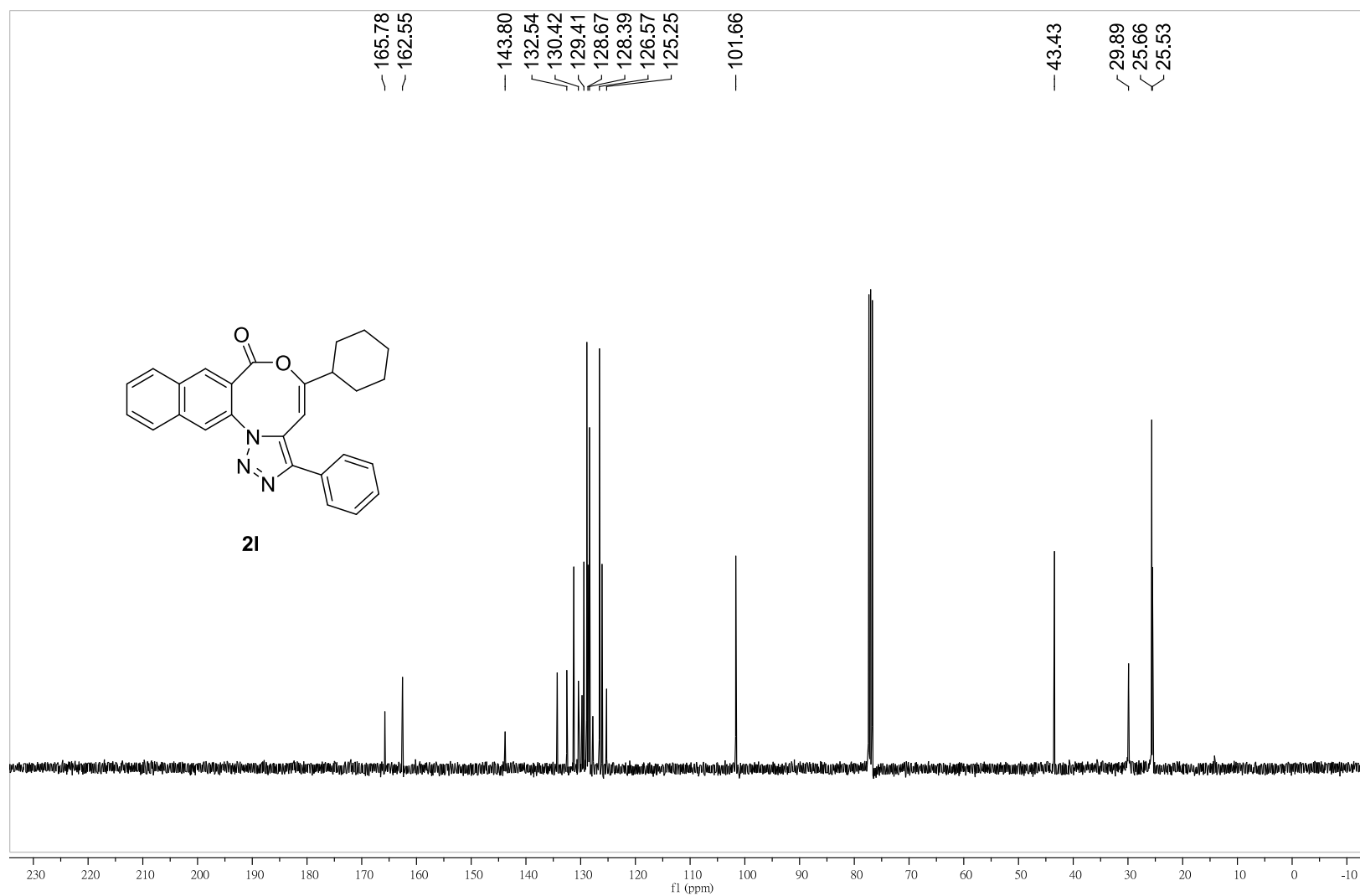
HRMS of compound **2k**

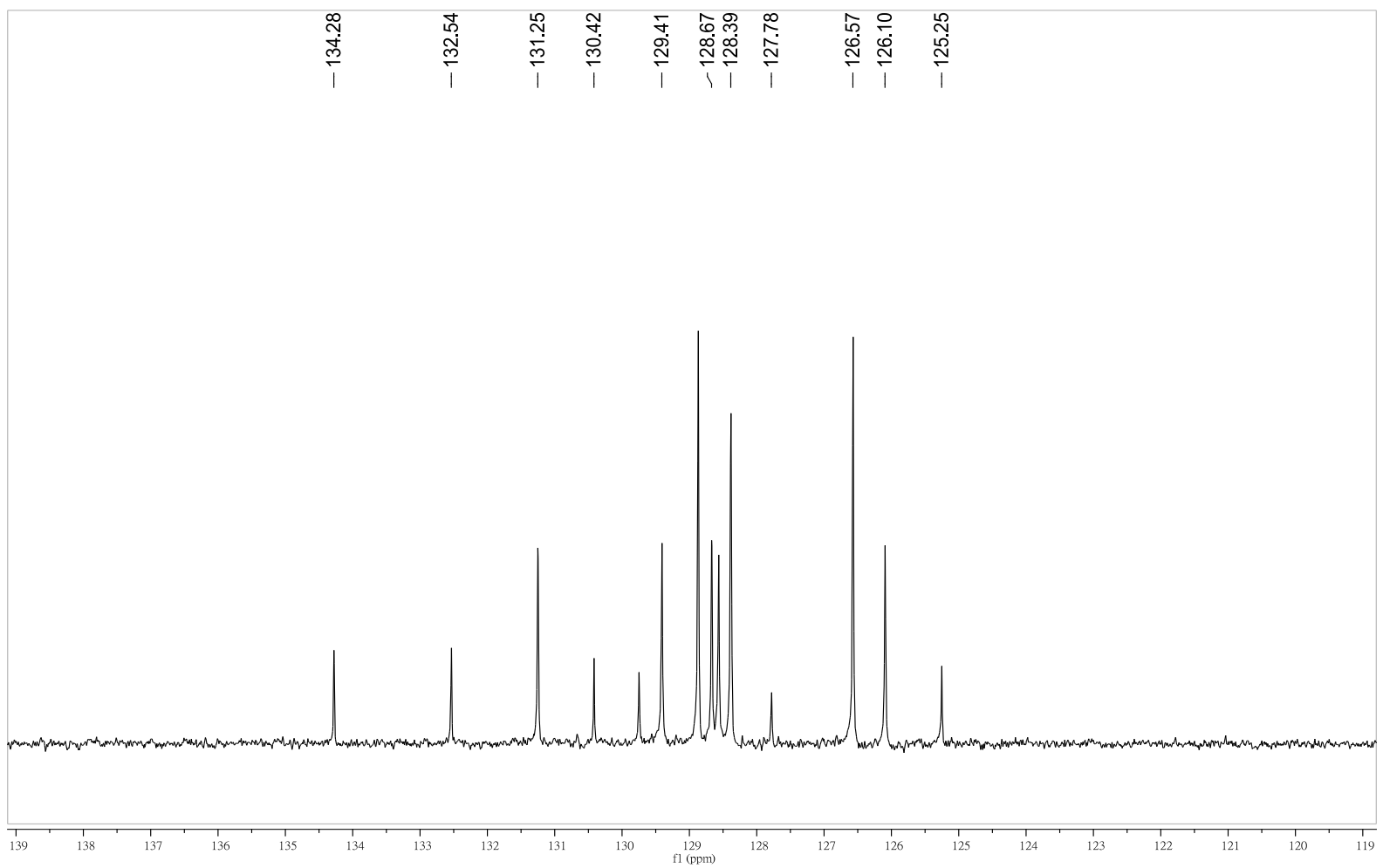


¹H NMR Spectrum (400 MHz) of compound **2I** in CDCl₃



Expansion of ^1H NMR Spectrum (400 MHz) of compound **2I** in CDCl_3



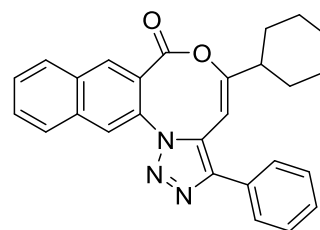
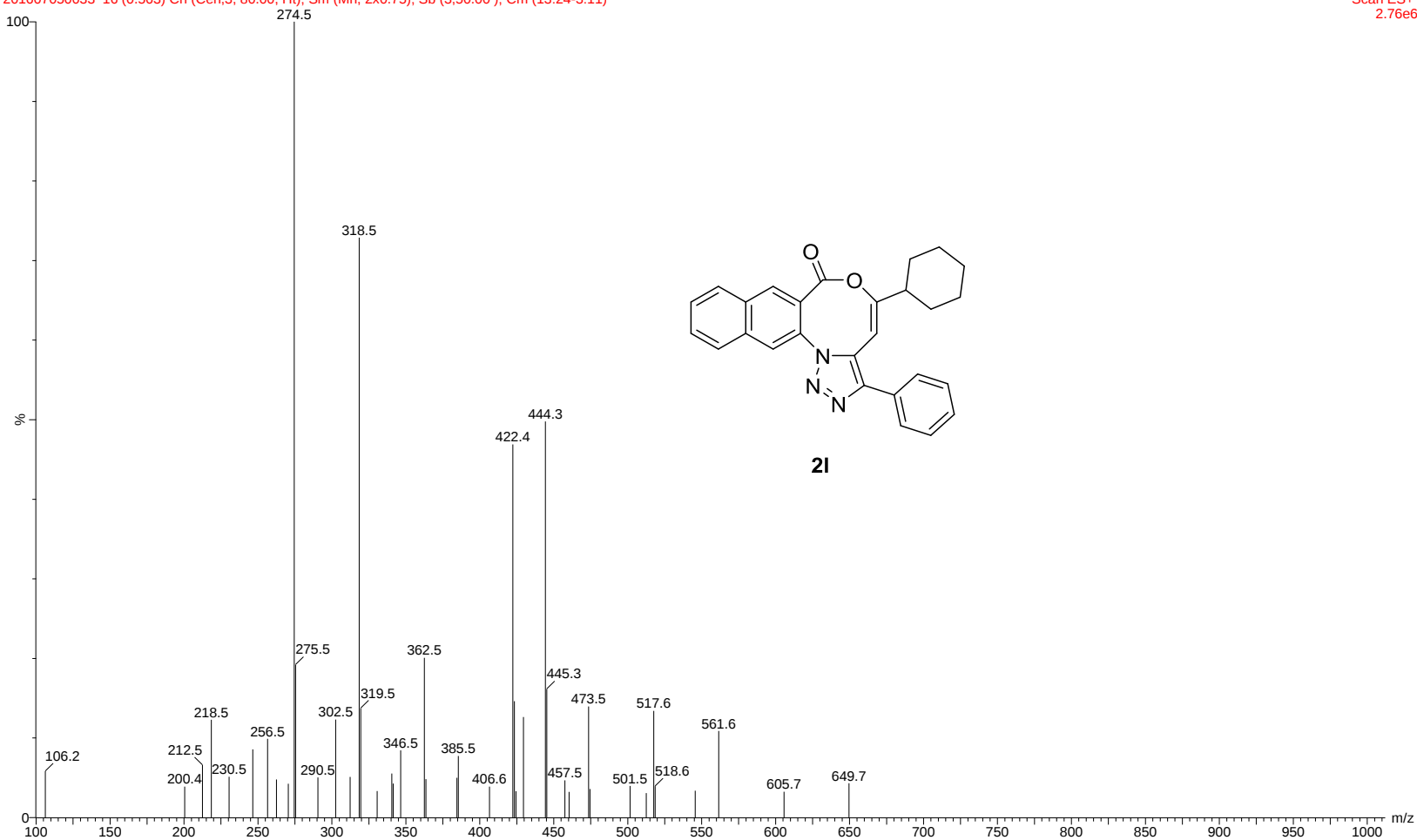


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2I** in CDCl_3

cyclehexane-final-4

201607050033 16 (0.563) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (13:24-3:11)

Scan ES+
2.76e6



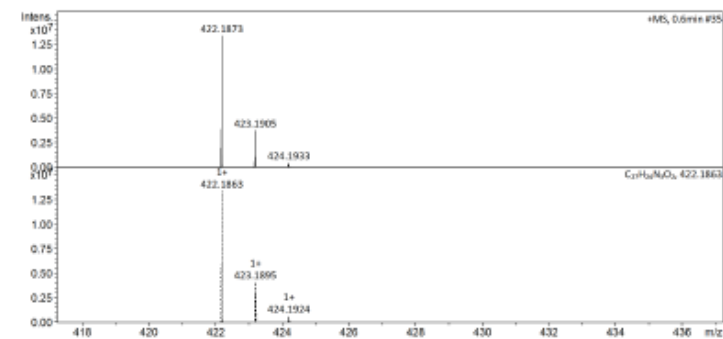
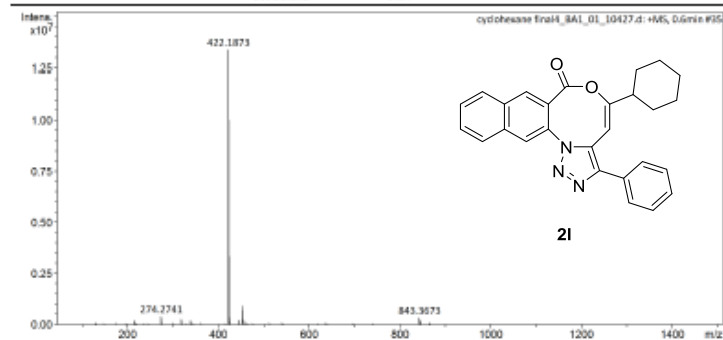
21

LRMS of compound 21

Display Report

Analytic Info		Acquisition Date	7/11/2016 1:29:55 PM	
Analysis Name	D:\Data\ctu service\data\2016\20160711\cyclohexane final4_BA1_01_10427.d		Operator	NCTU
Method	Small molecule.m		Instrument	Impact HD
Sample Name	cyclohexane final4			1819696.00164
Comment				

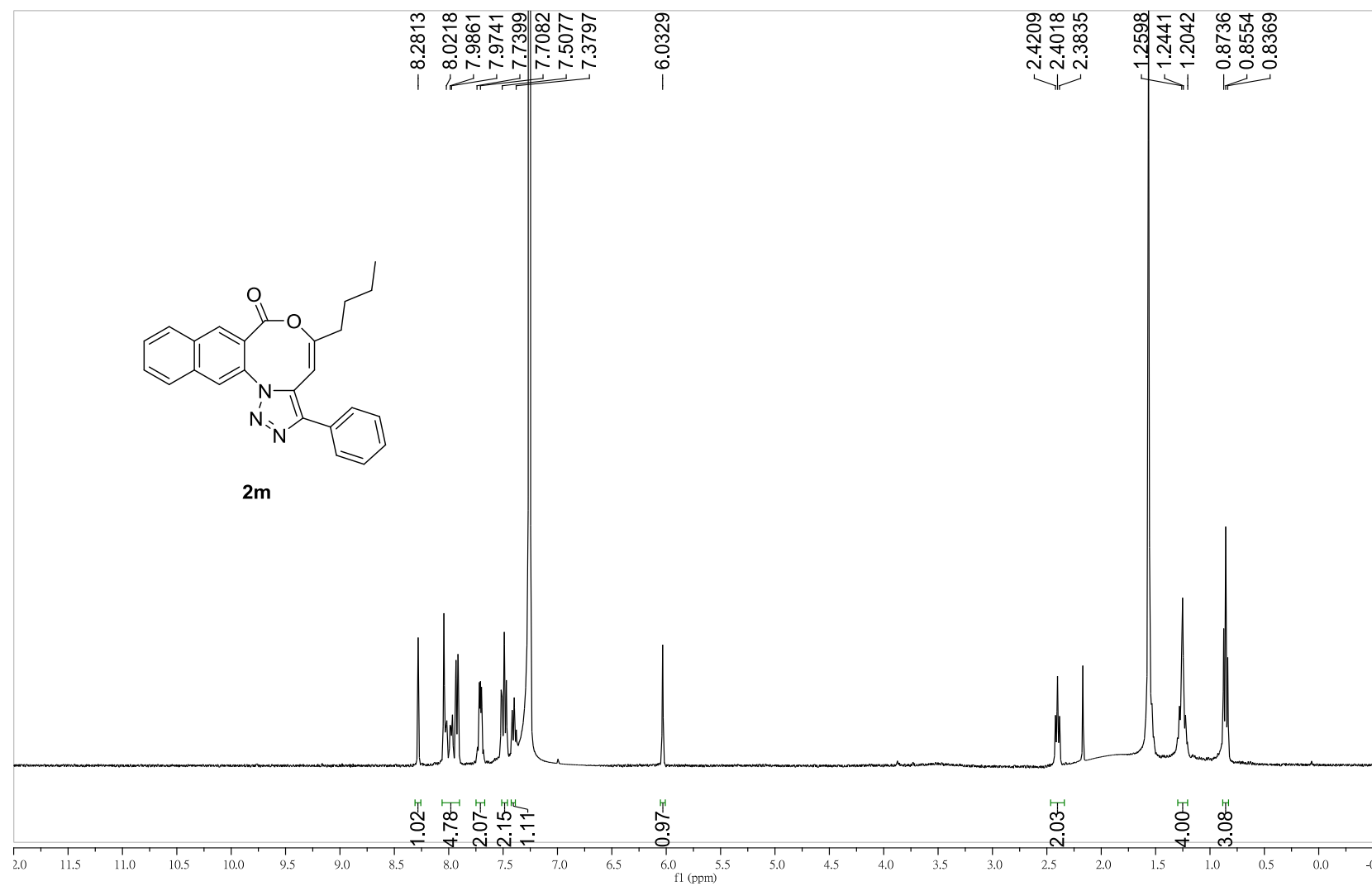
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 L/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



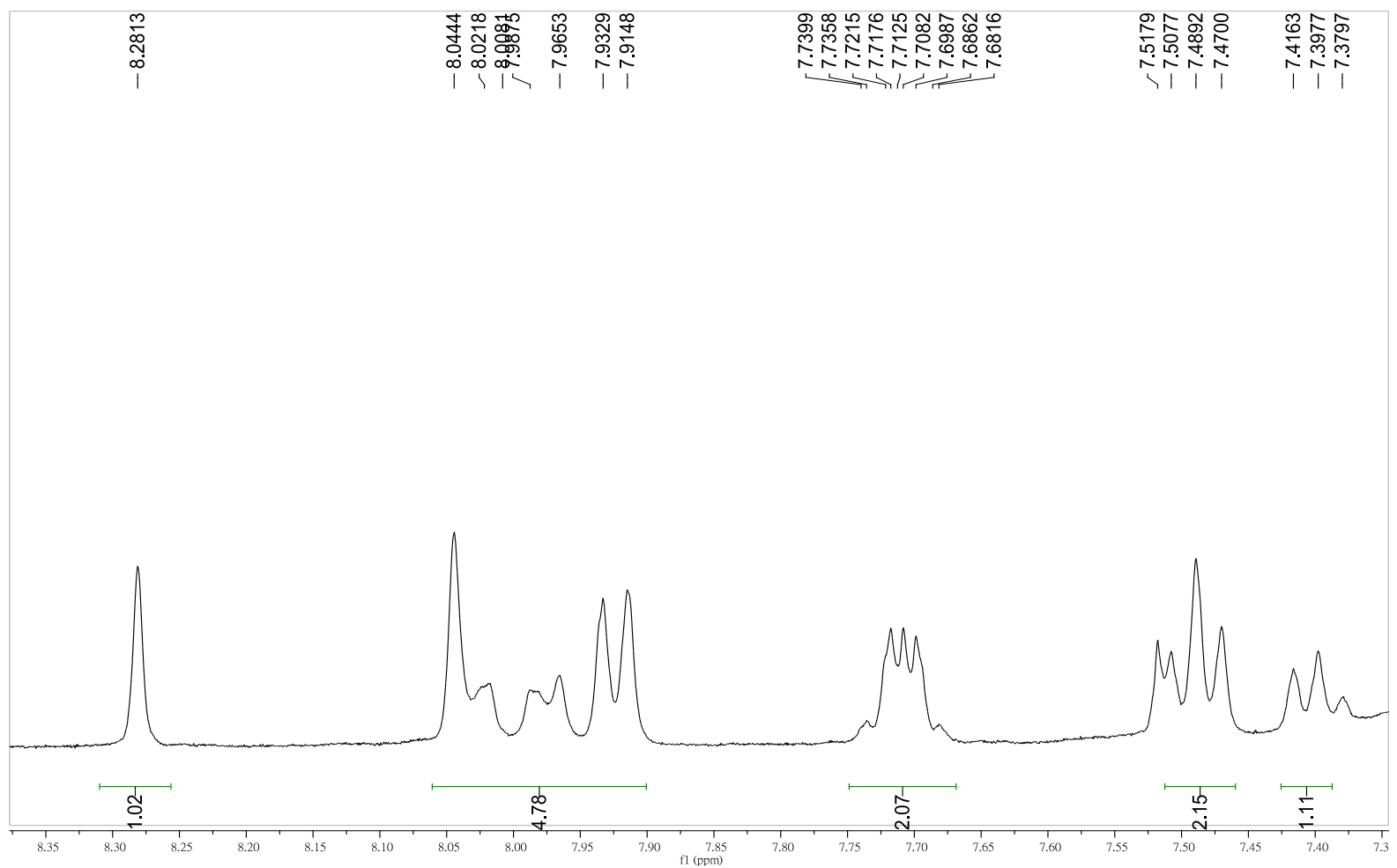
Display Report

Mass, m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	ndb	e ⁻ Conf	N-Rule	Adduct
422.1873	1	C ₂₇ H ₂₀ N ₃ O ₂	422.1863	-2.4	15.7	1	100.00	17.5	even	ok	M+H

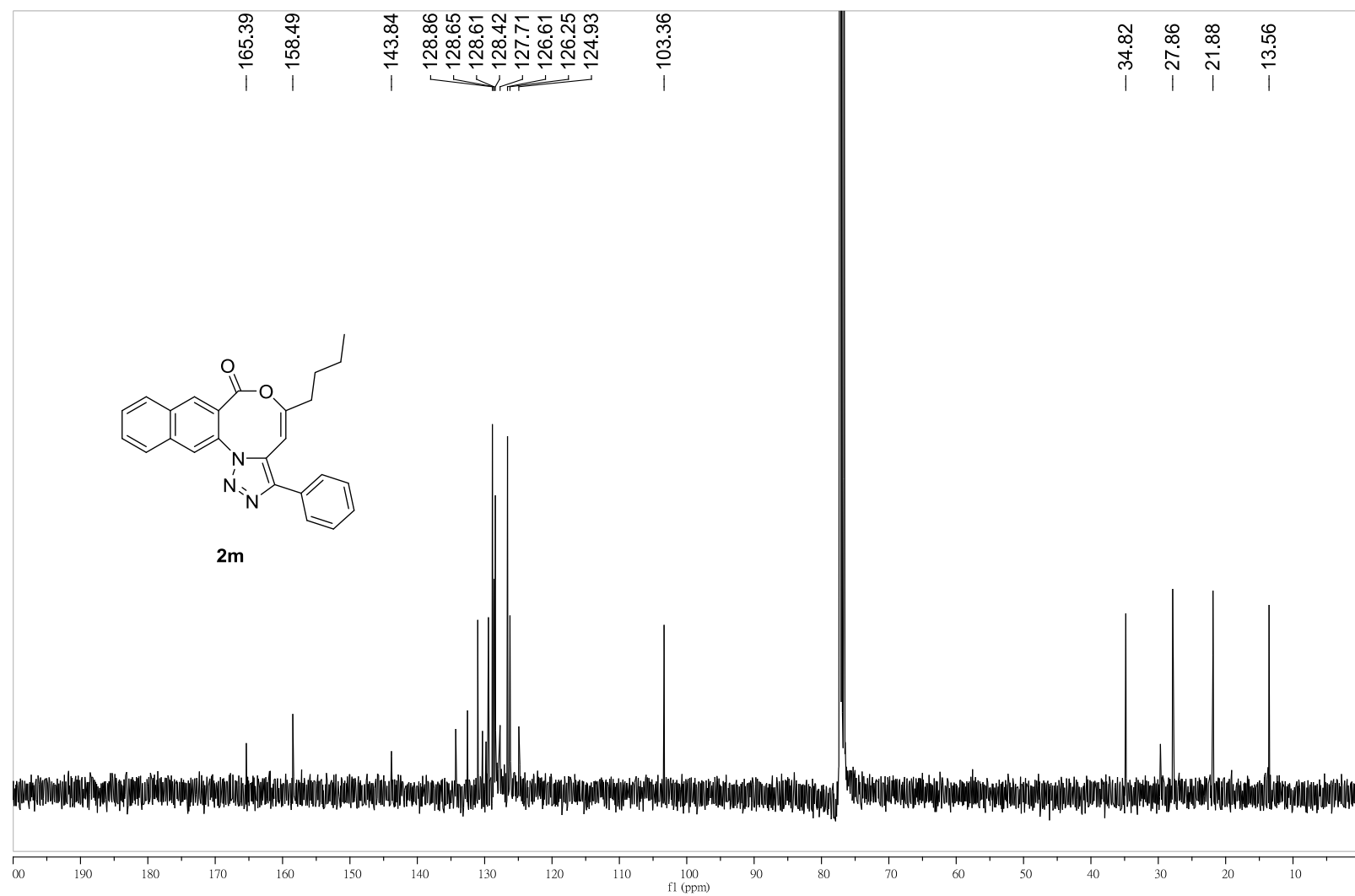
HRMS of compound 2I



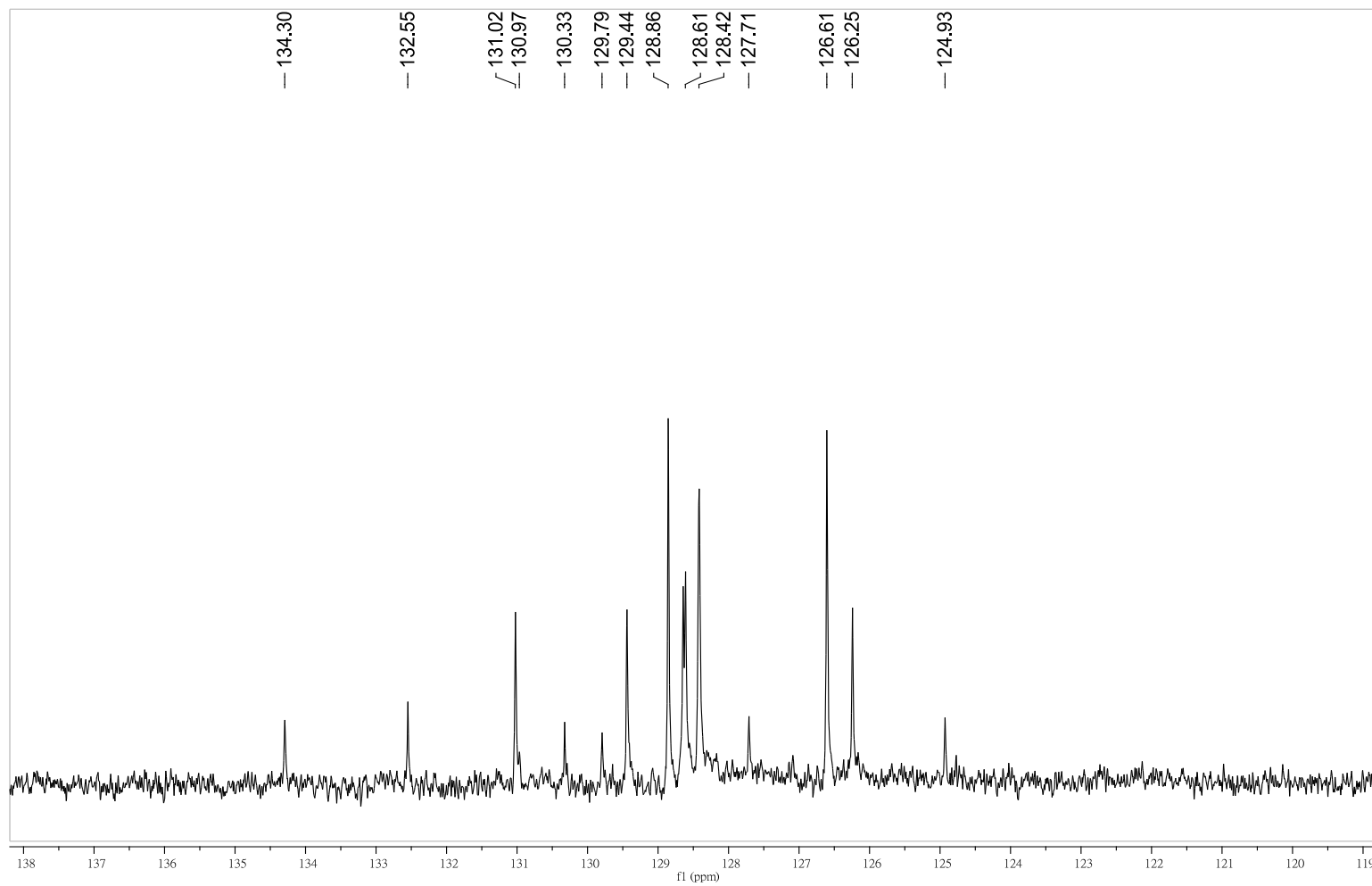
^1H NMR Spectrum (400 MHz) of compound **2m** in CDCl_3



Expansion of ^1H NMR Spectrum (400 MHz) of compound **2m** in CDCl_3



¹³C NMR Spectrum (101 MHz) of compound **2m in CDCl₃**

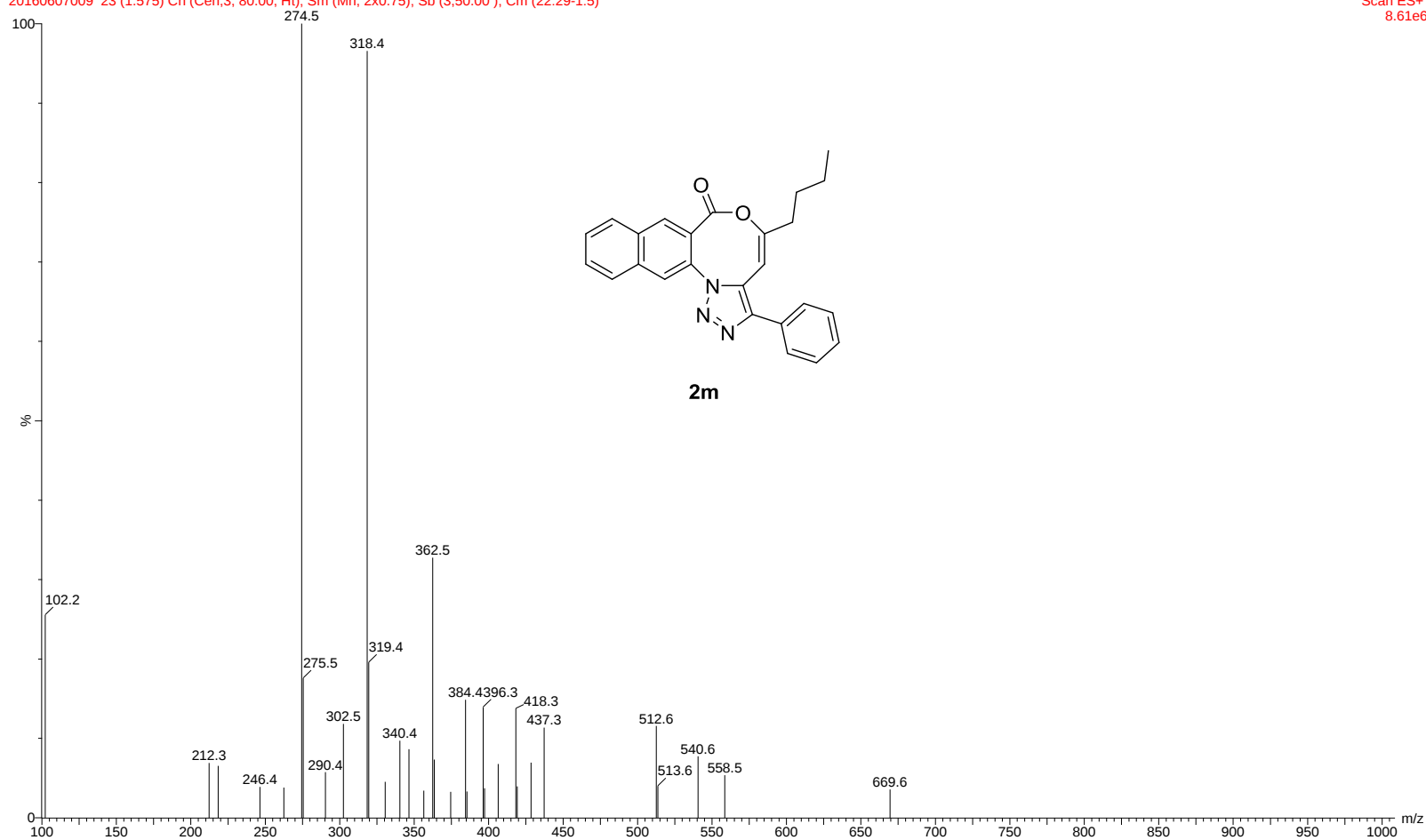


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2m** in CDCl_3

3p22

20160607009 23 (1.575) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (22:29-1:5)

Scan ES+
8.61e6

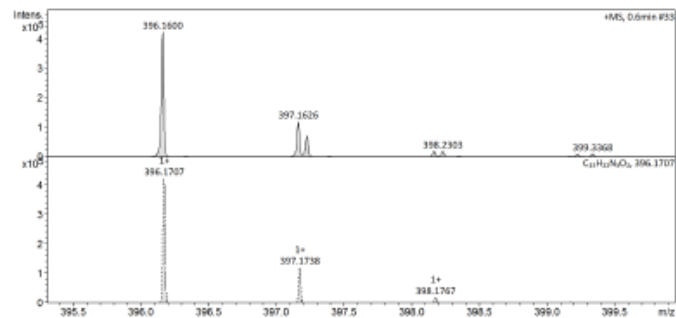
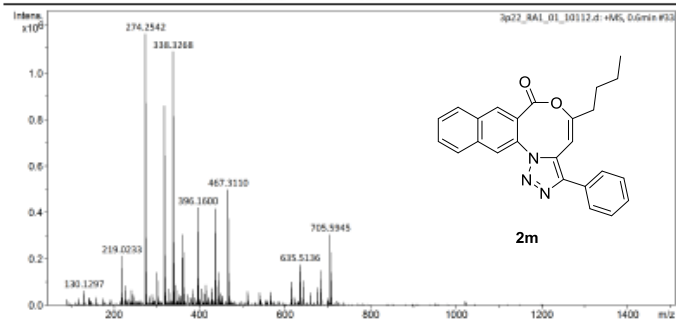


LRMS of compound 2m

Display Report

Analytic Info
 Analysis Name: D:\Data\inctu service\data\2016\20160607\3p22_RA1_01_10112.d
 Method: Small molecule.m
 Sample Name: 3p22
 Comment:
 Acquisition Date: 6/14/2016 10:56:56 AM
 Operator: NCTU
 Instrument: Impact HD
 Impact ID: 1819696.00164

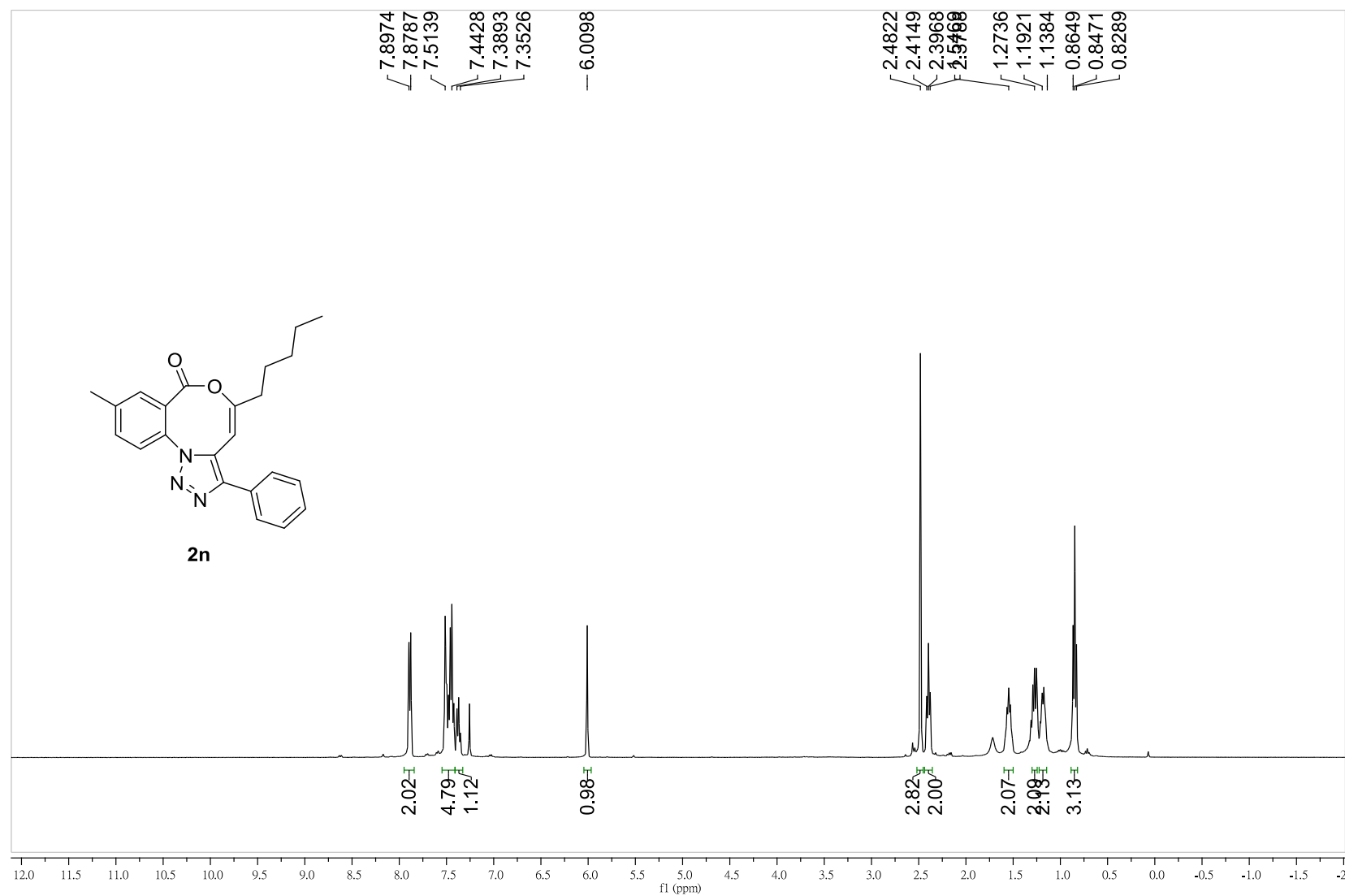
Acquisition Parameter
 Source Type: ESI
 Focus: Active
 Scan Begin: 50 m/z
 Scan End: 1500 m/z
 Ion Polarity: Positive
 Set Capillary: 4500 V
 Set End Plate Offset: -500 V
 Set Charging Voltage: 2000 V
 Set Corona: 0 nA
 Set Nebulizer: 1.0 Bar
 Set Dry Heater: 200 °C
 Set Dry Gas: 6.0 l/min
 Set Divert Valve: Waste
 Set AP/CI Heater: 0 °C



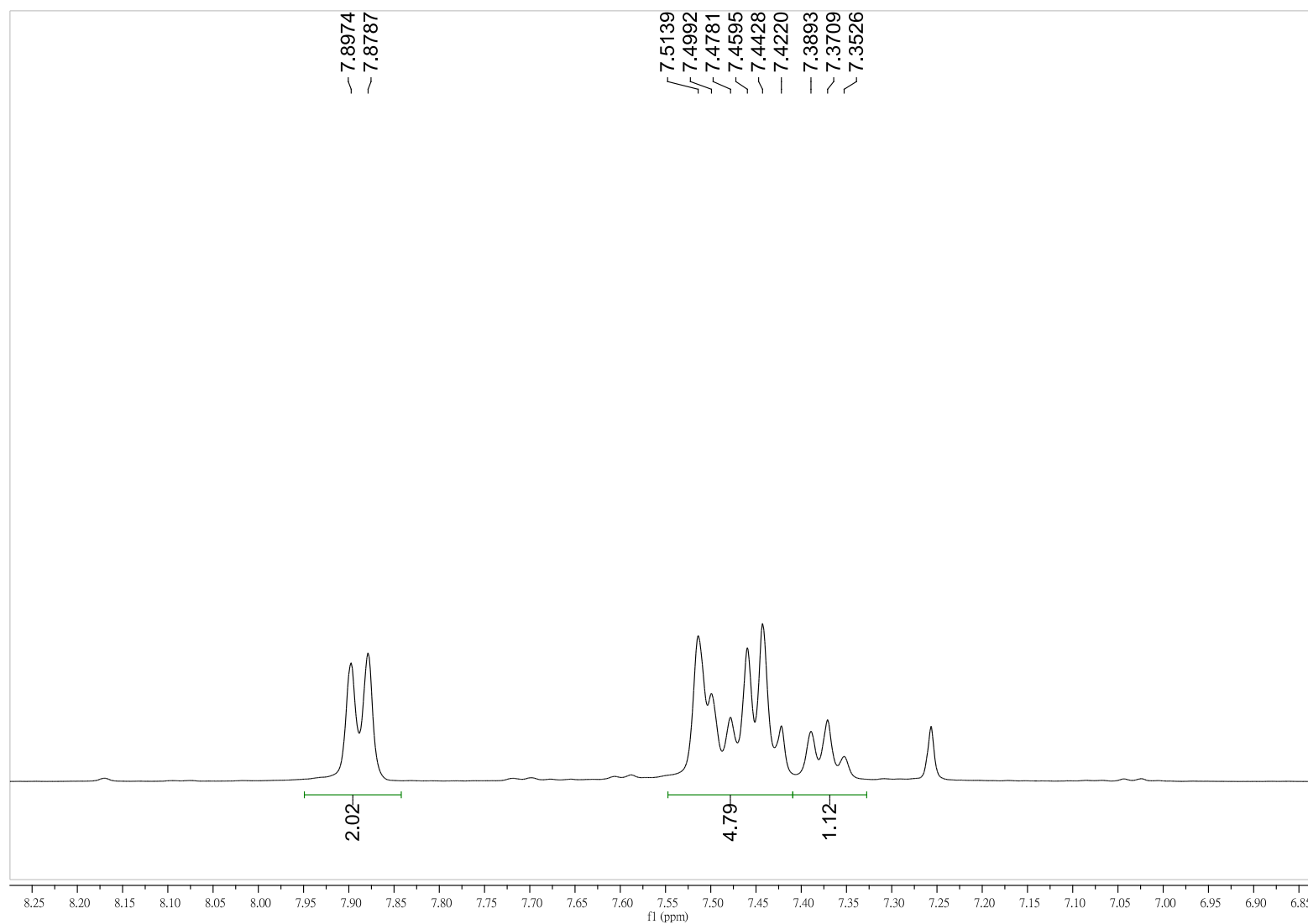
Display Report

Mass, m/z	#	Ion Formula	m/z	em [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
396.1600	1	C ₂₅ H ₂₂ N ₃ O ₂	396.1707	27.0	1.8	1	100.00	18.5	even	ok	M+H

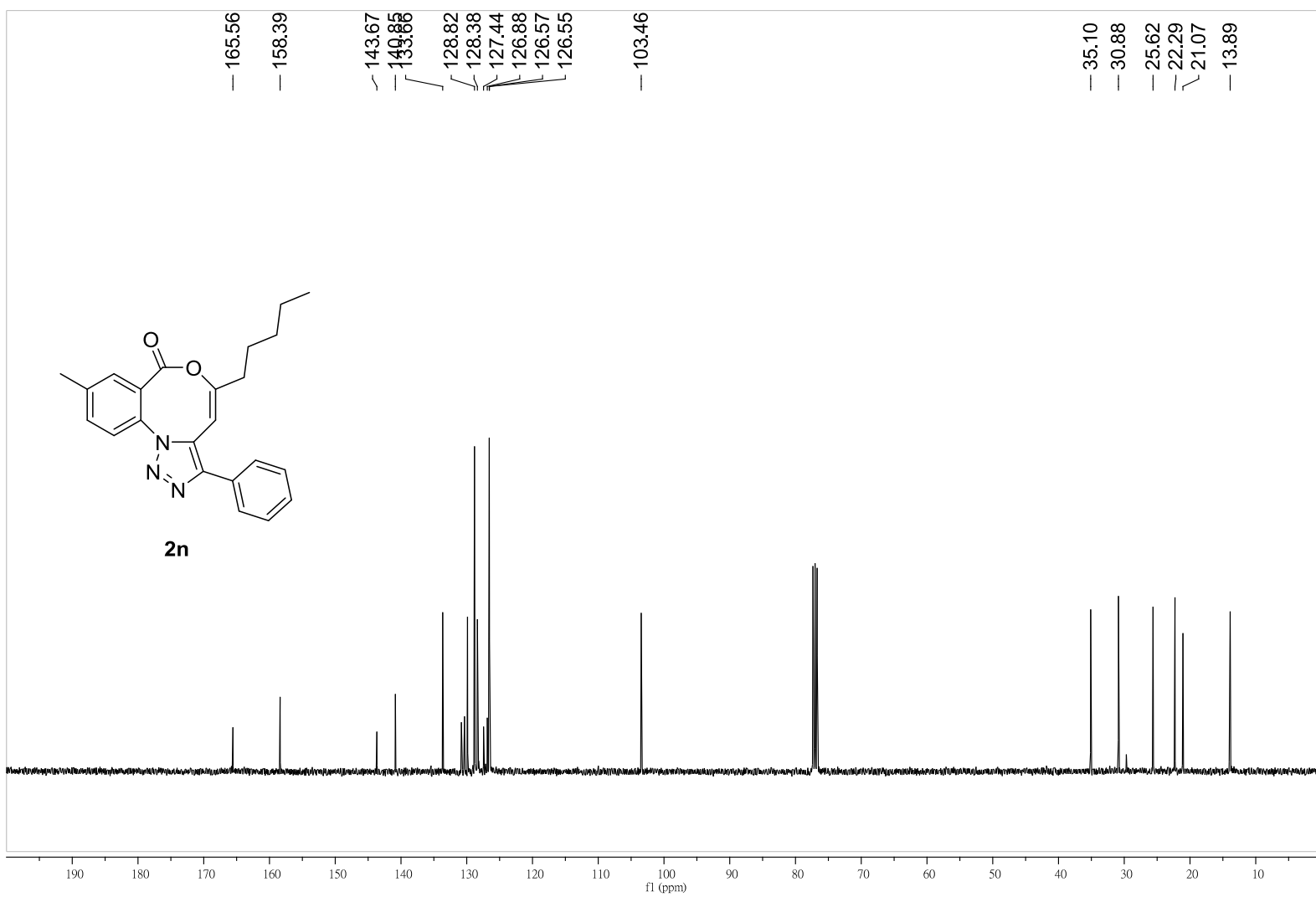
HRMS of compound **2m**



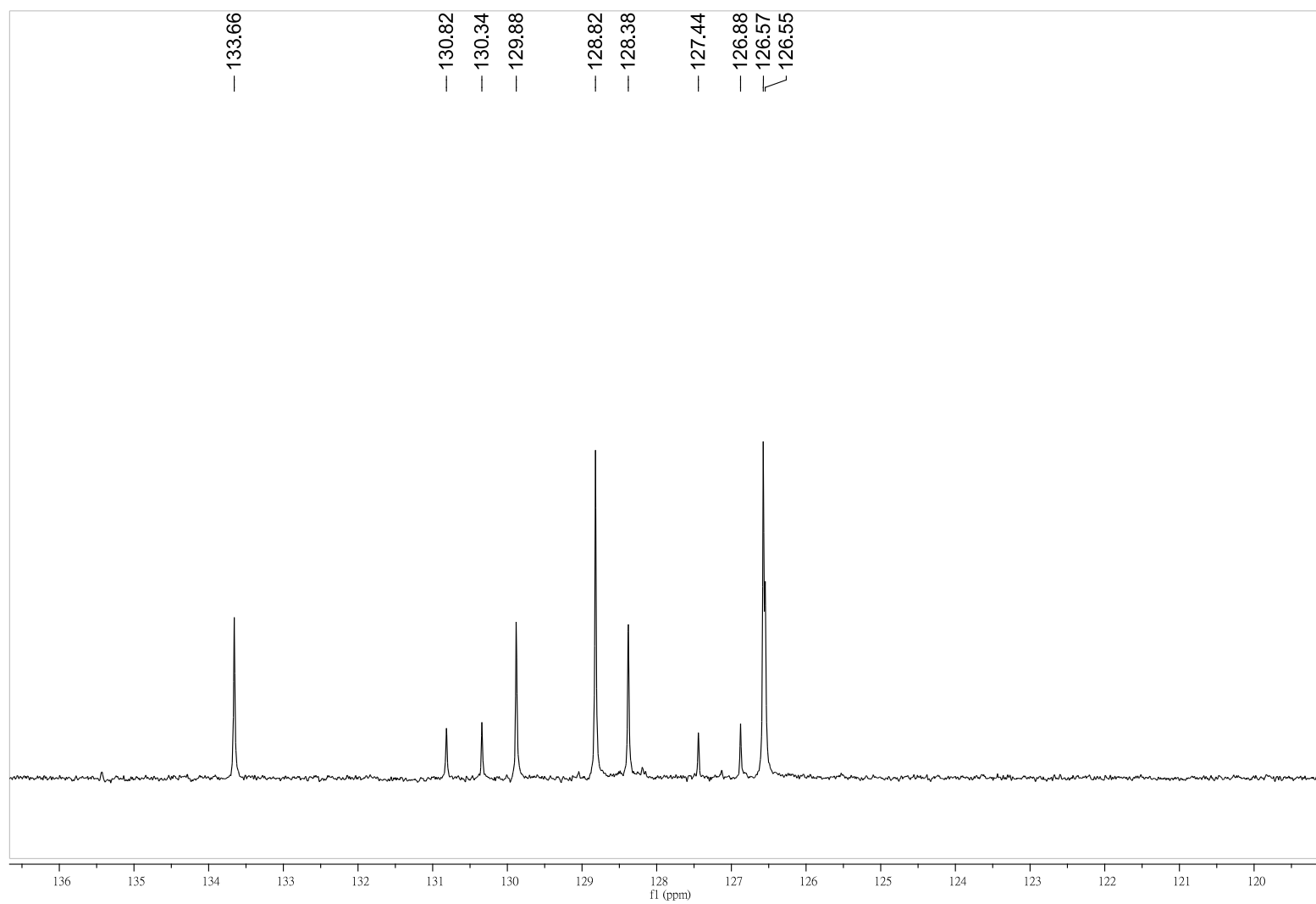
^1H NMR Spectrum (400 MHz) of compound **2n** in CDCl_3



Expansion of ^1H NMR Spectrum (400 MHz) of compound **2n** in CDCl_3



¹³C NMR Spectrum (101 MHz) of compound **2n** in CDCl₃

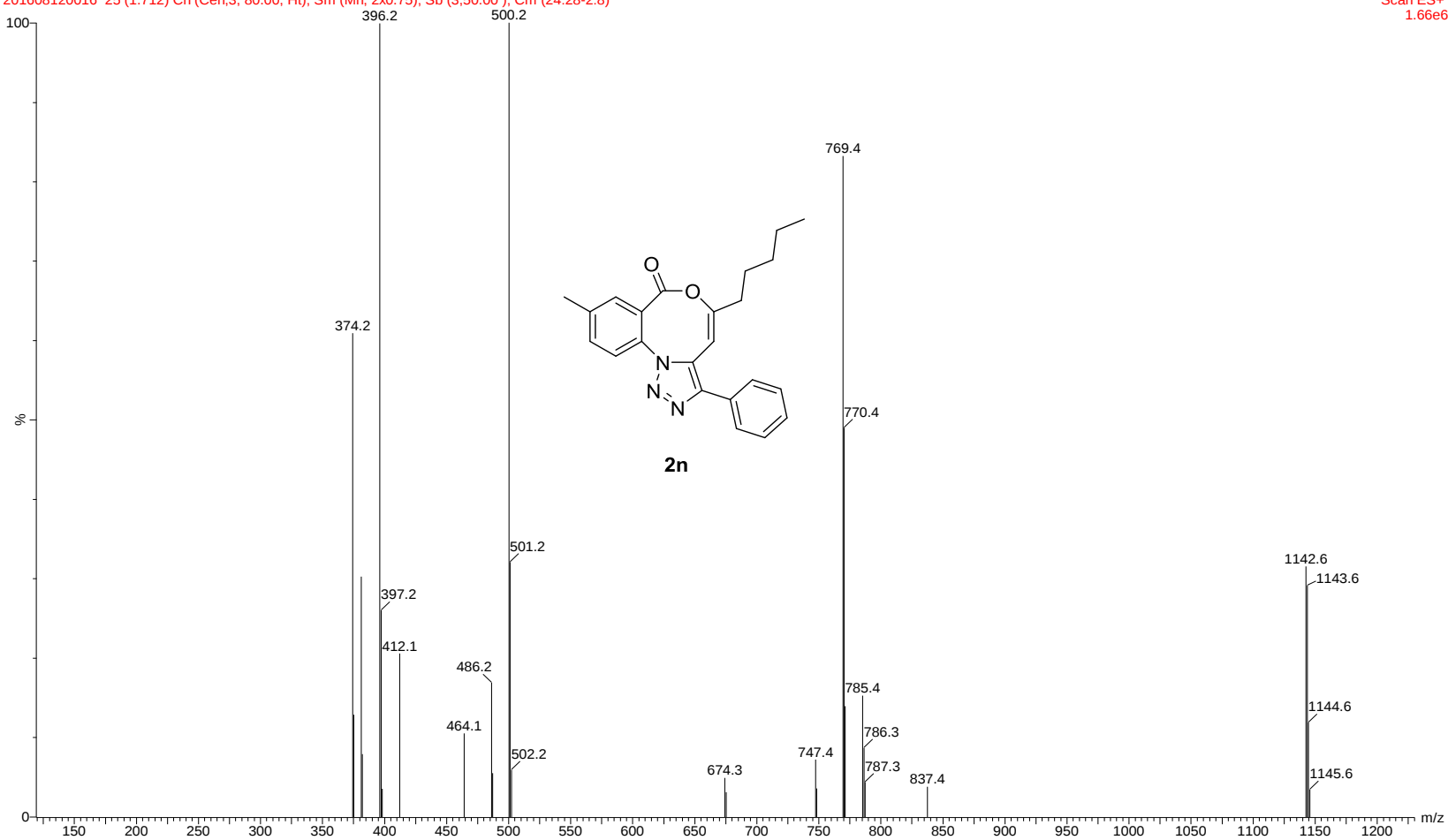


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2n** in CDCl_3

3p52-Ag

201608120016 25 (1.712) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (24:28-2:8)

Scan ES+
1.66e6

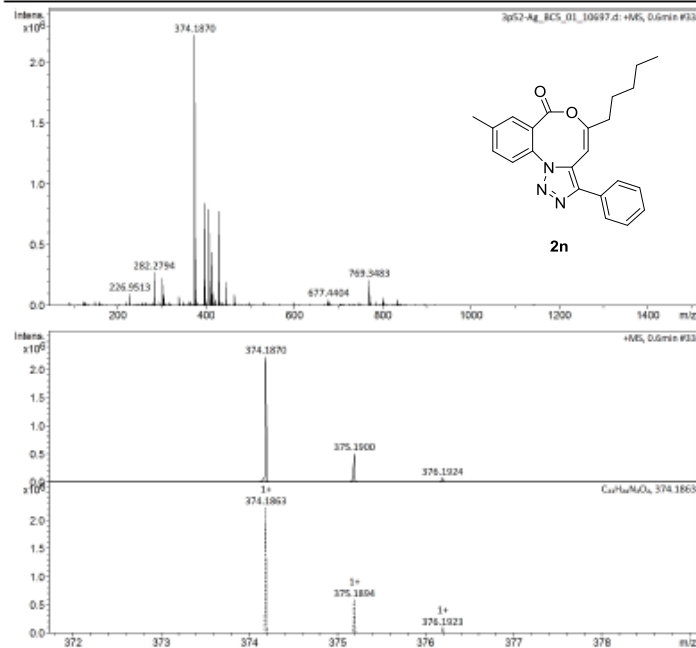


LRMS of compound **2n**

Display Report

Analysis Info		Acquisition Date	8/16/2016 10:22:41 AM	
Analysis Name	D:\Data\inchi service\data\2016\20160816\3p52-Ag_BC5_01_10697.d	Operator	NCTU	
Method	Small molecule.m	Instrument	Impact HD	1819696.00164
Sample Name	3p52-Ag	Comment		

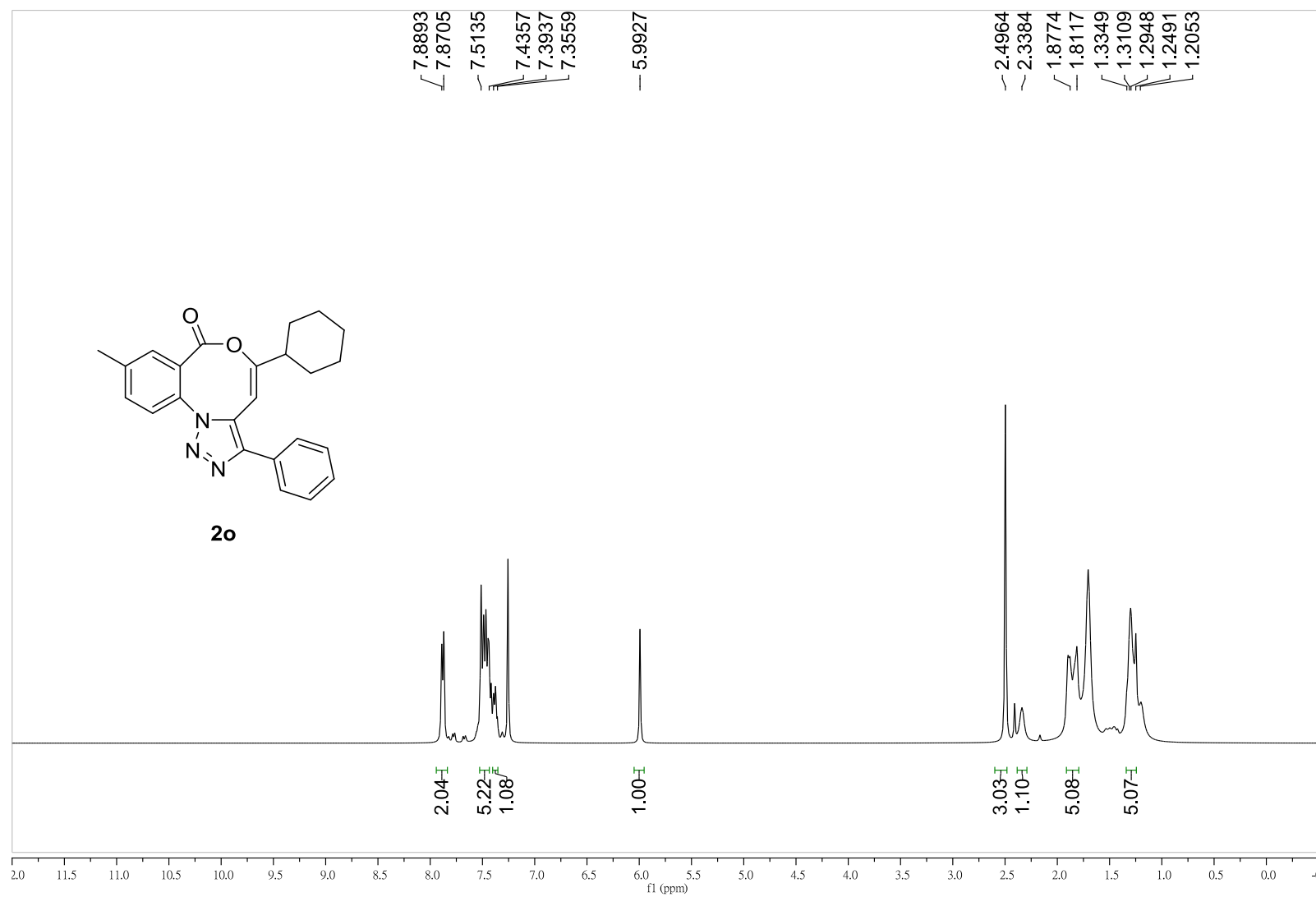
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



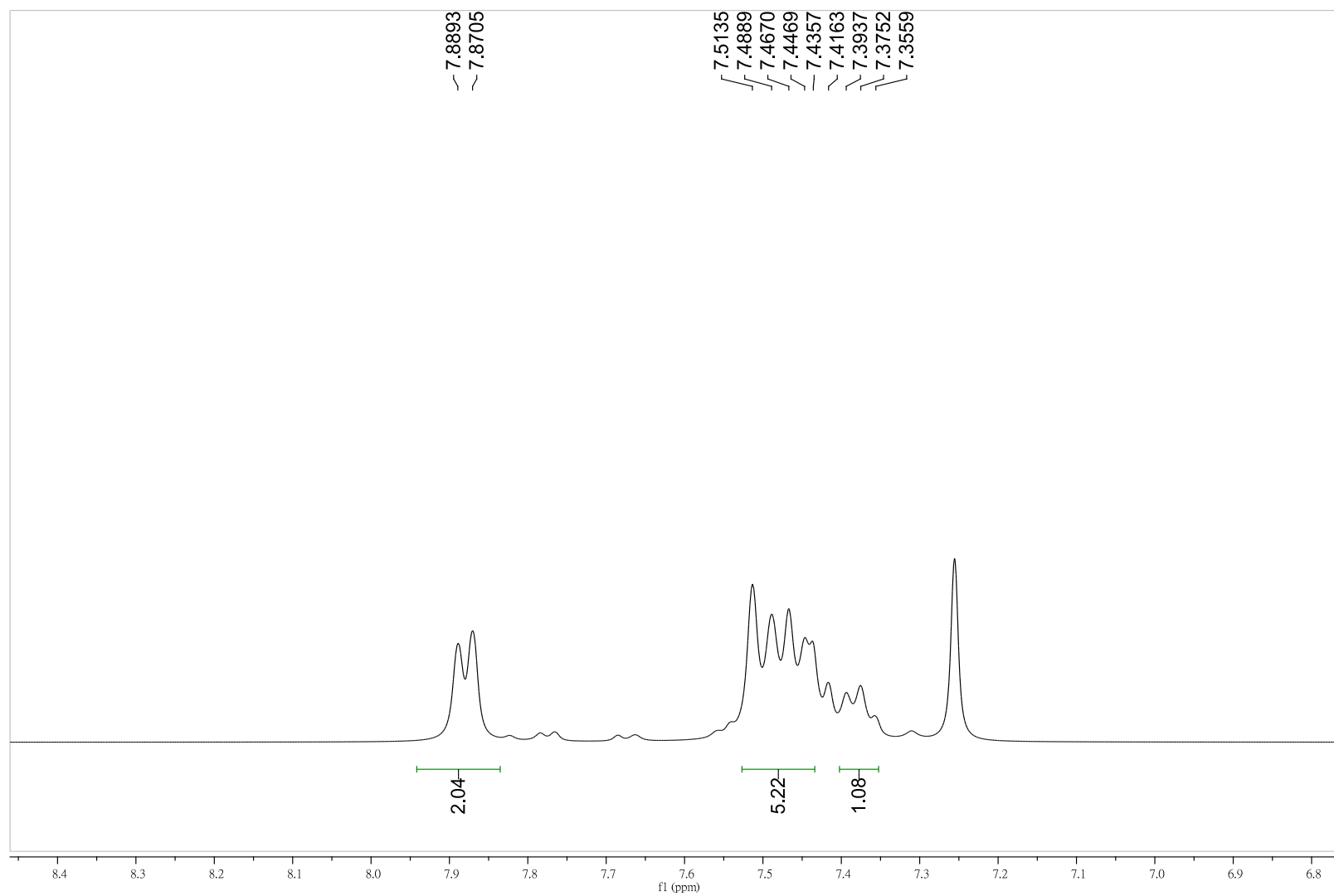
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
374.1870	1	C ₂₃ H ₂₄ N ₃ O ₂	374.1863	1.9	17.9	1	100.00	13.5	even	ok	M+H

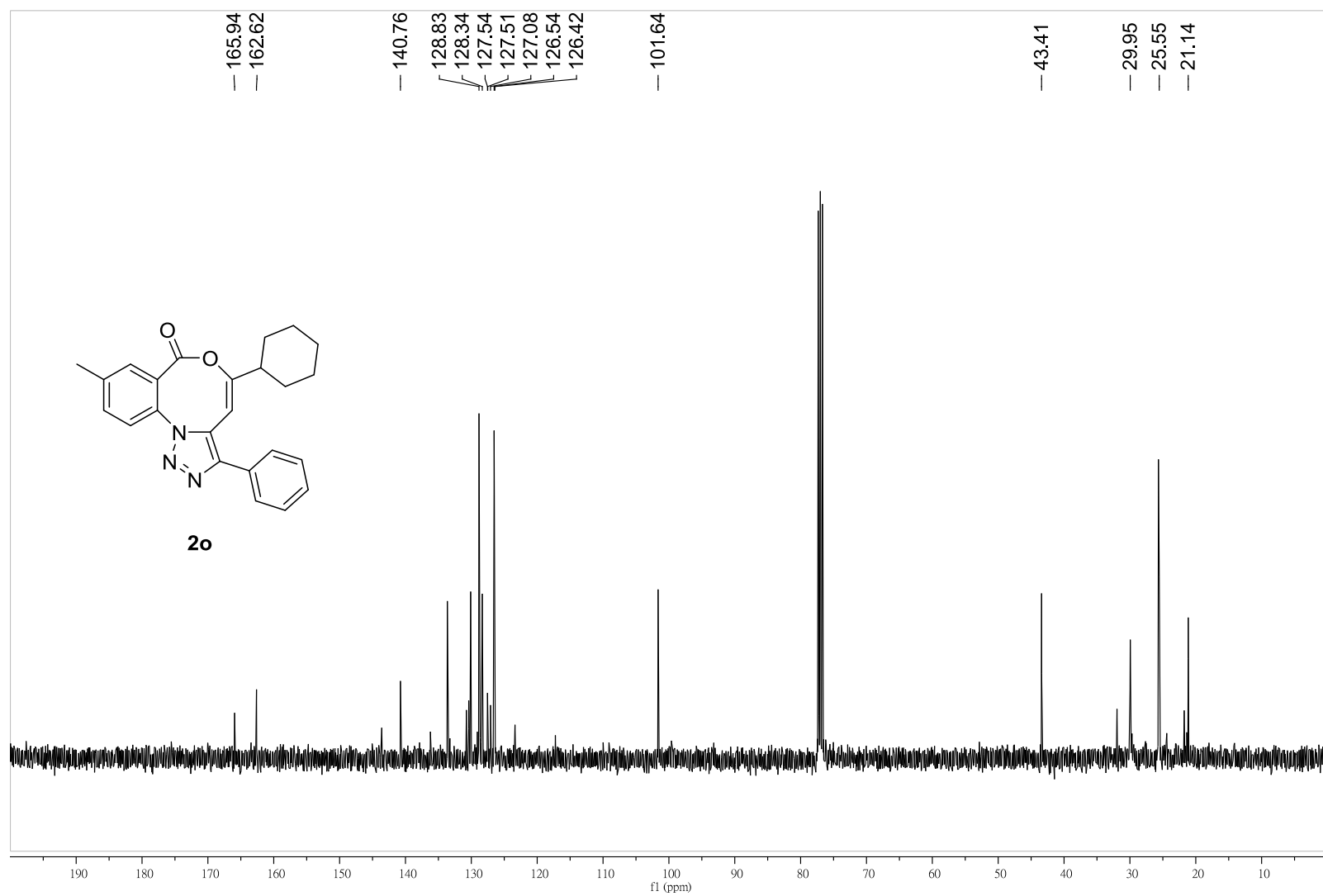
HRMS of compound **2n**

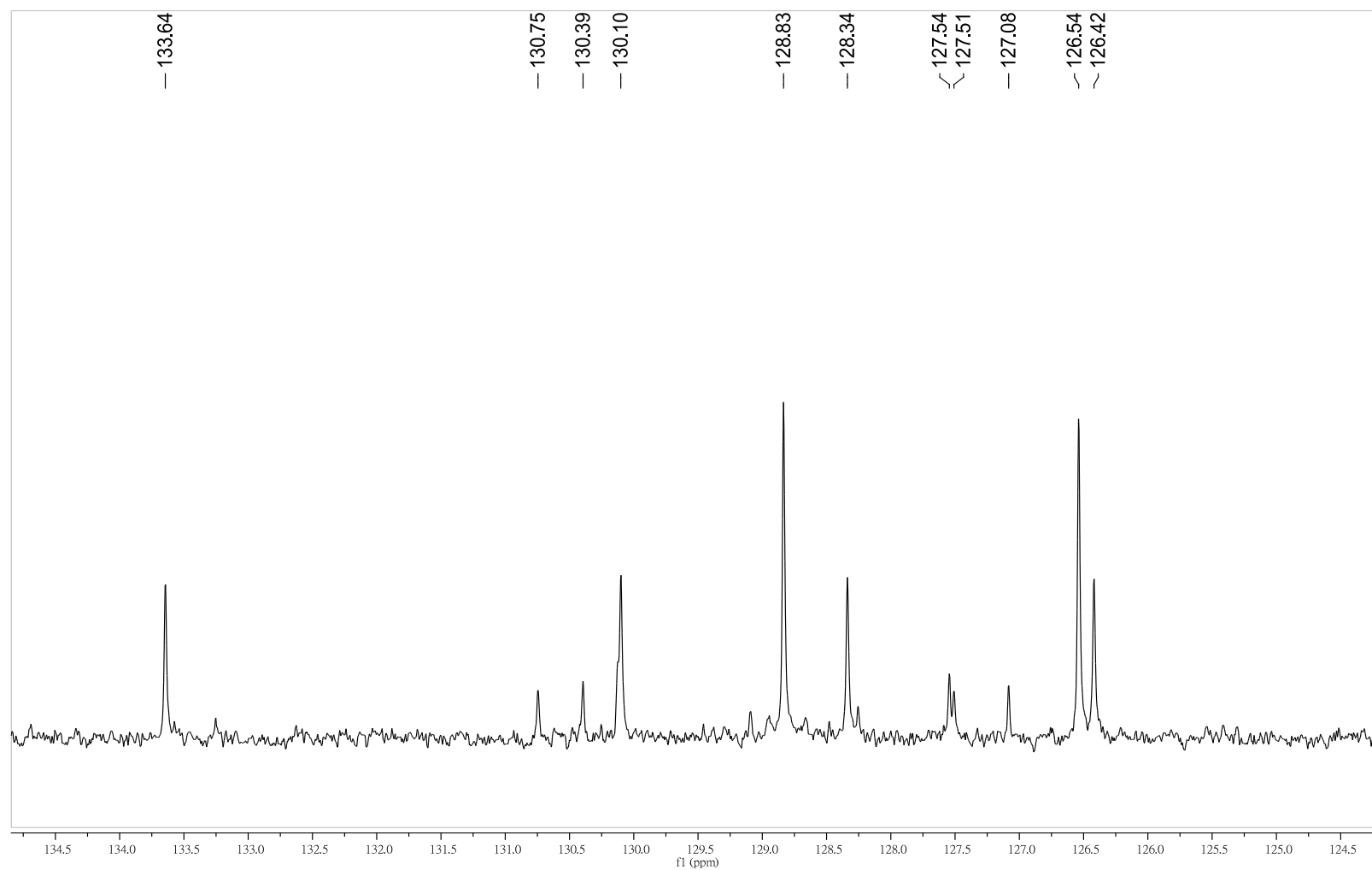


¹H NMR Spectrum (400MHz) of compound **2o** in CDCl₃



Expansion of ^1H NMR Spectrum (400MHz) of compound **2o** in CDCl_3



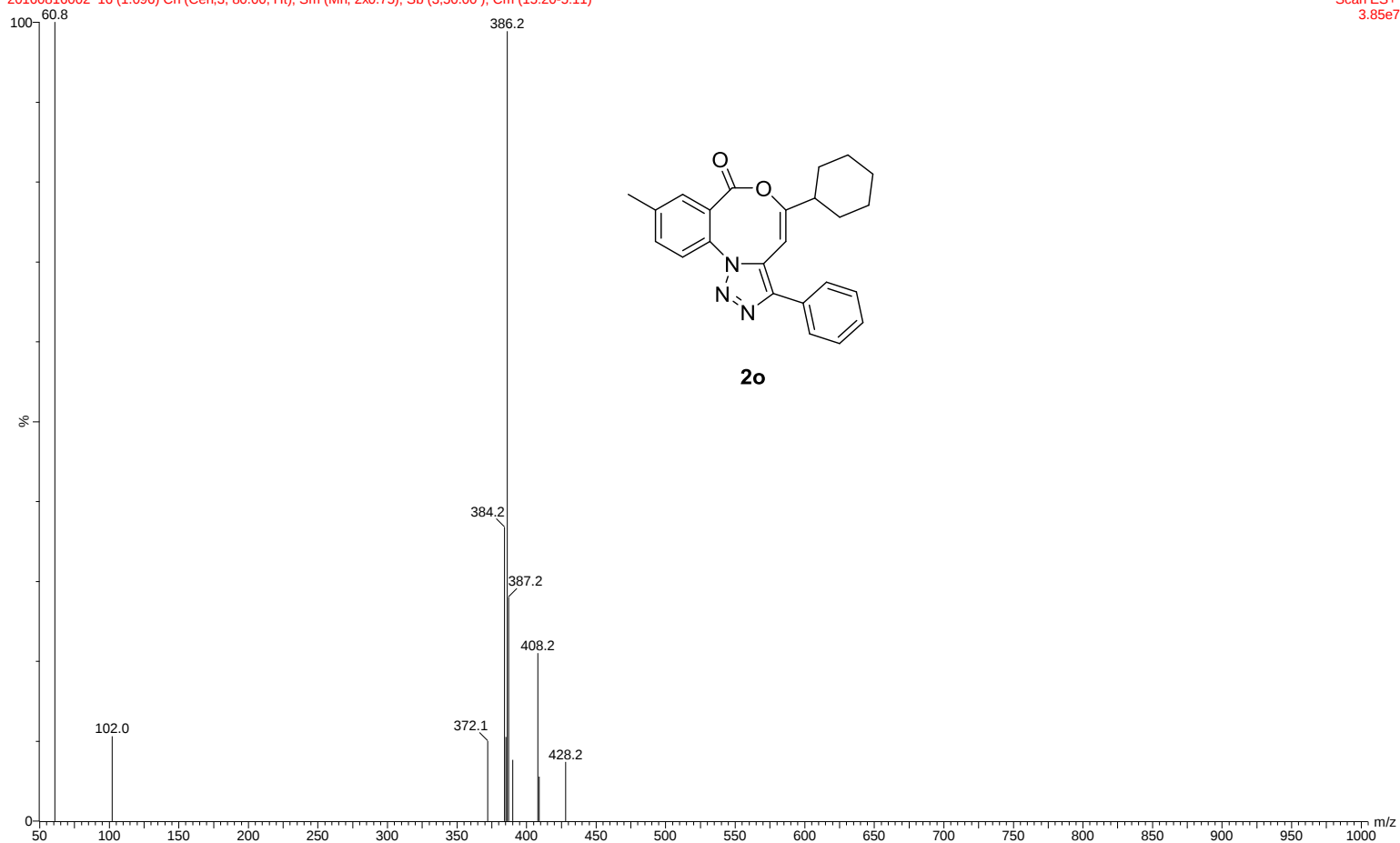


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2o** in CDCl_3

3p56-Ag

20160816002 16 (1.096) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (15:20-5:11)

Scan ES+
3.85e7

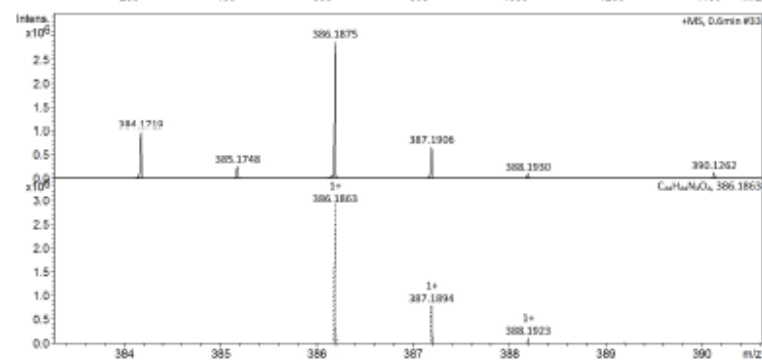
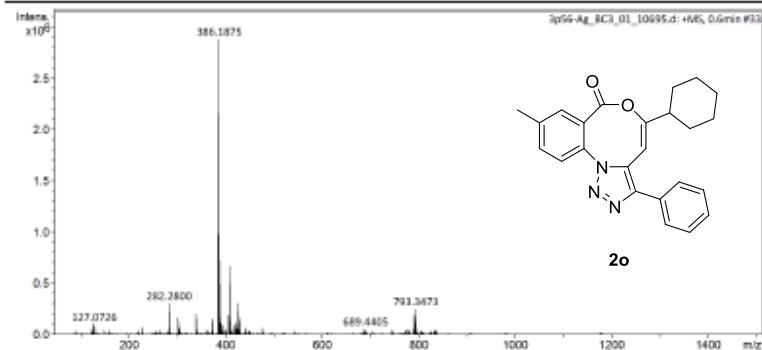


LRMS of compound **2o**

Display Report

Analytic Info		Acquisition Date 8/16/2016 10:14:03 AM	
Analysis Name	D:\Data\ctu service\data\2016\20160816\3p56-Ag_BC3_01_10695.d	Operator	NCTU
Method	Small molecule.m	Instrument	Impact HD
Sample Name	3p56-Ag	Impact HD	1819696.00164
Comment			

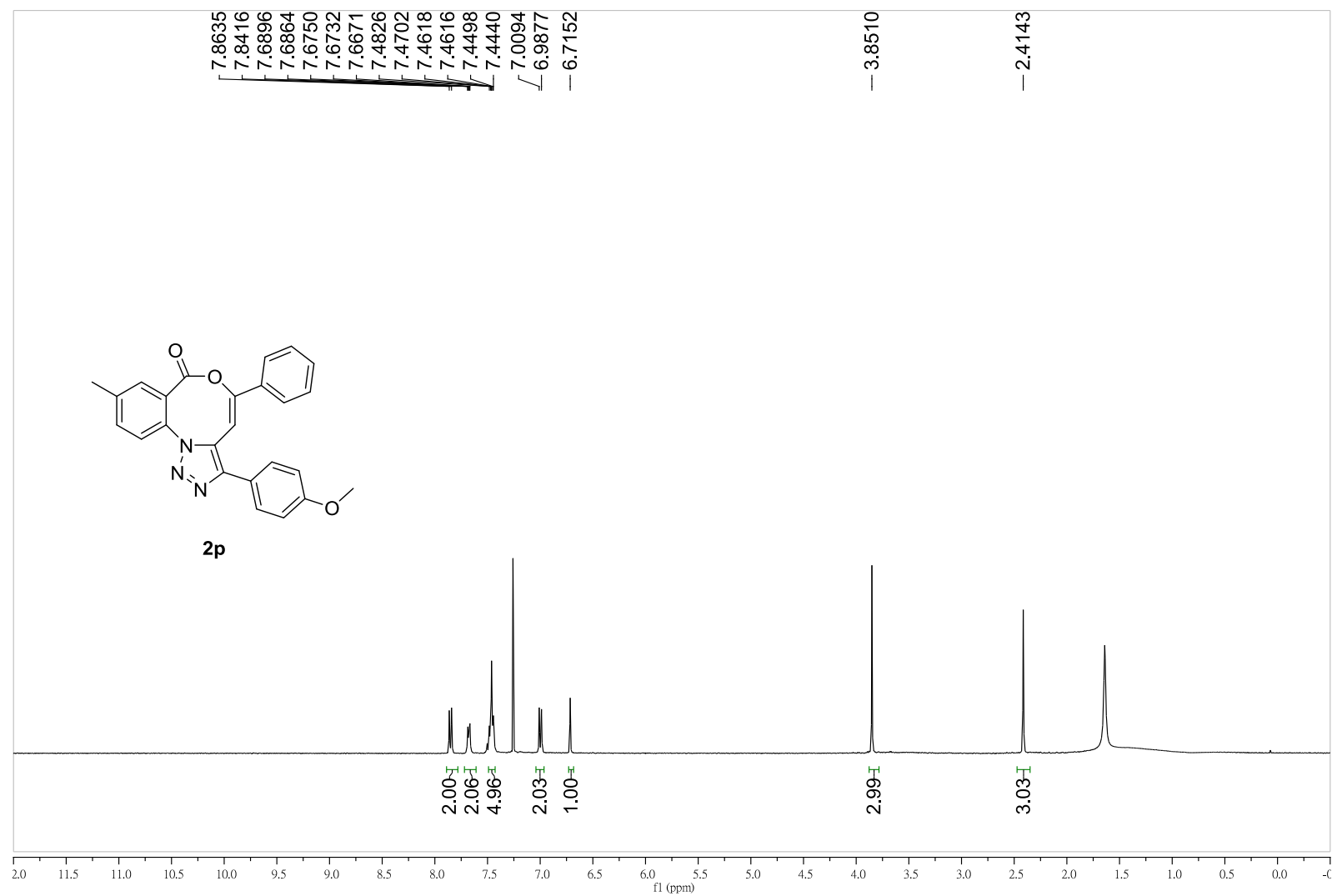
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C



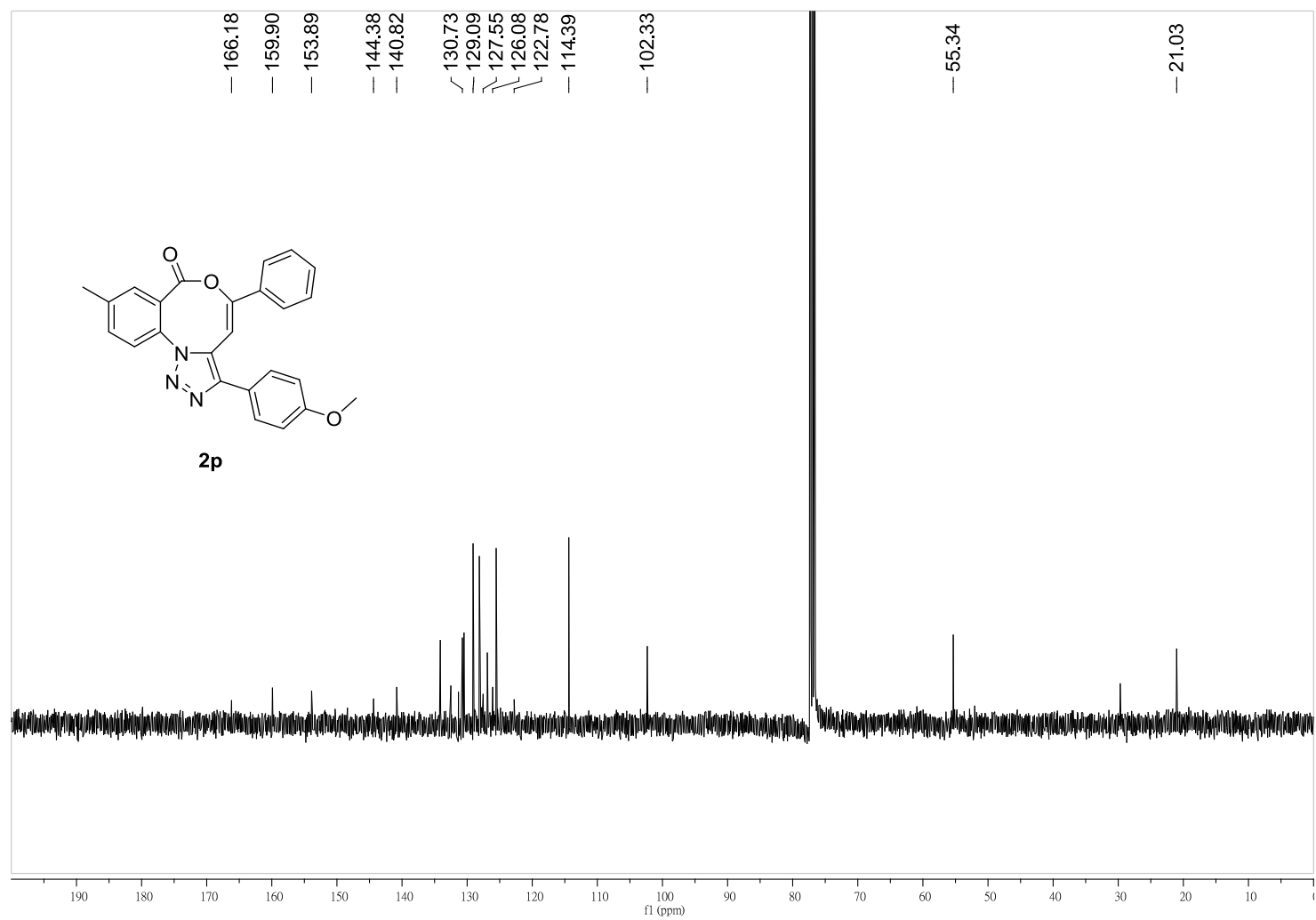
Display Report

Mass, m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdB	e ⁻ Conf	N-Rule	Adduct
386.1875	1	C ₂₄ H ₂₄ N ₂ O ₂	386.1883	-3.1	22.4	1	100.00	14.5	even	ok	M+H

HRMS spectrum of compound **2o**



^1H NMR Spectrum (400 MHz) of compound **2p**

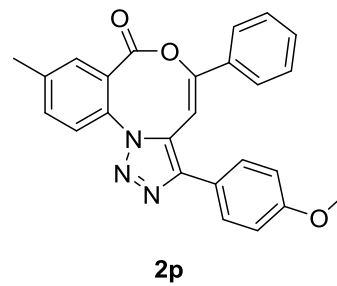
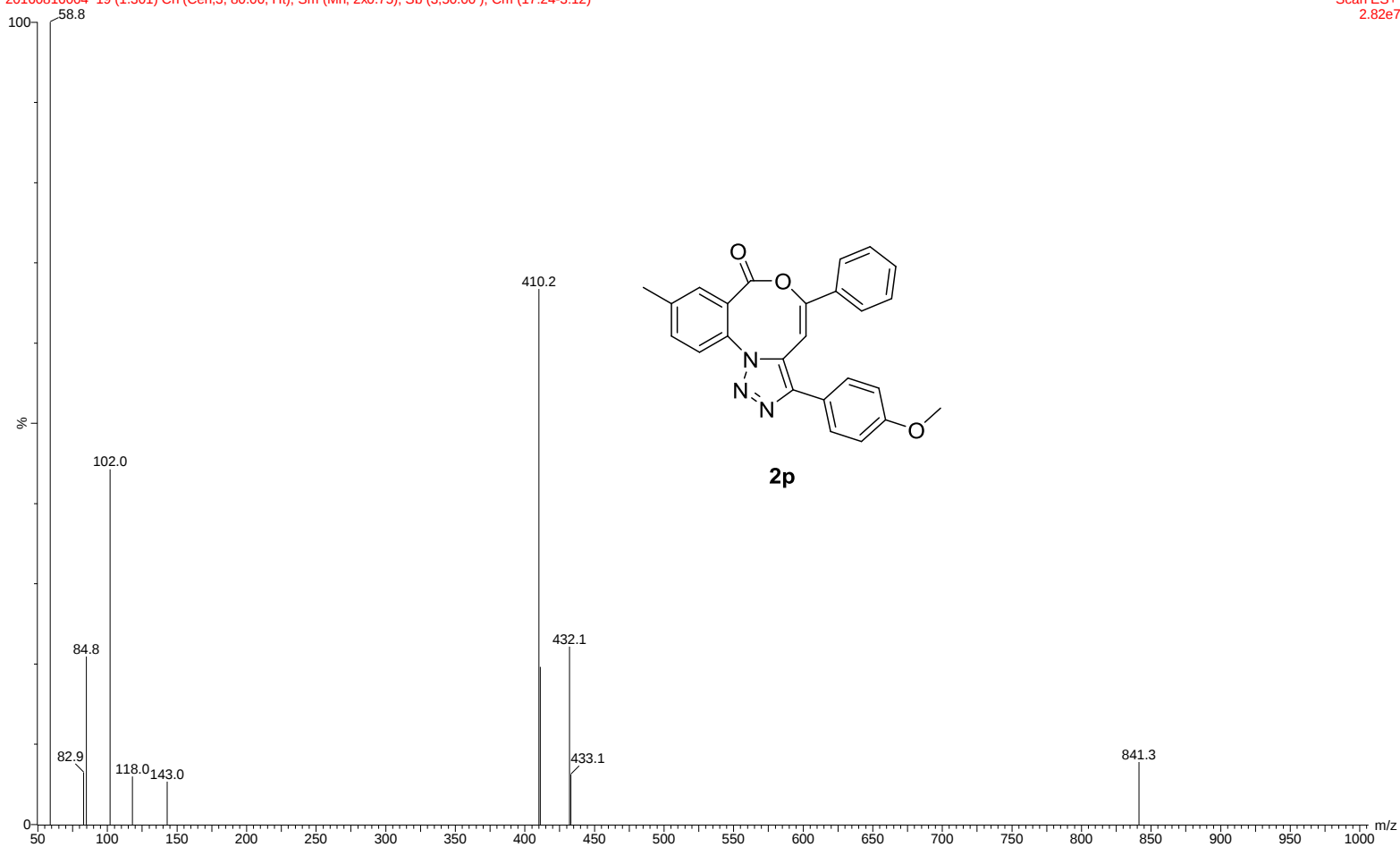


^{13}C NMR Spectrum (101 MHz) of compound **2p** in CDCl_3

3p58-Ag

20160816004 19 (1.301) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (17:24-3:12)

Scan ES+
2.82e7

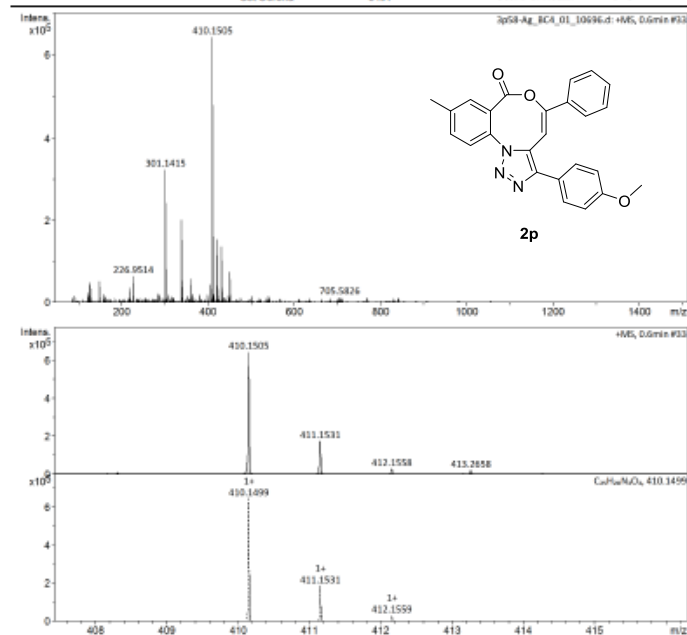


LRMS of compound 2p

Display Report

Analysis Info
 Analysis Name: D:\Data\inctu service\data\2016\20160816\3p58-Ag_BC4_01_10696.d
 Method: Small molecule.m
 Sample Name: 3p58-Ag
 Comment:
 Acquisition Date: 8/16/2016 10:18:22 AM
 Operator: NCTU
 Instrument: Impact HD
 Impact ID: 1819696.00164

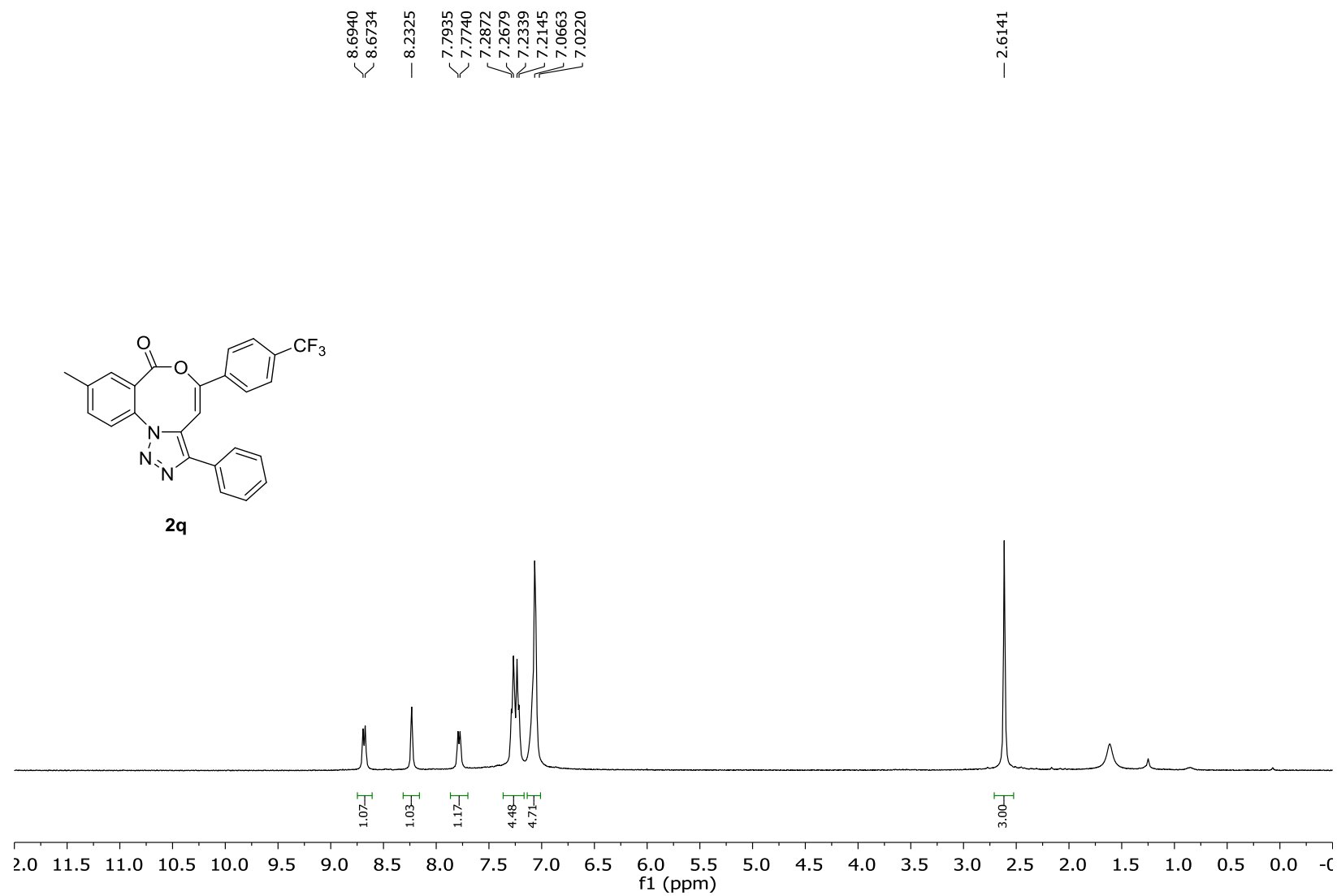
Acquisition Parameter
 Source Type: ESI
 Focus: Active
 Scan Begin: 50 m/z
 Scan End: 1500 m/z
 Ion Polarity: Positive
 Set Capillary: 4500 V
 Set End Plate Offset: -500 V
 Set Charging Voltage: 2000 V
 Set Corona: 0 nA
 Set Nebulizer: 1.0 Bar
 Set Dry Heater: 200 °C
 Set Dry Gas: 6.0 l/min
 Set Divert Valve: Waste
 Set APCI Heater: 0 °C



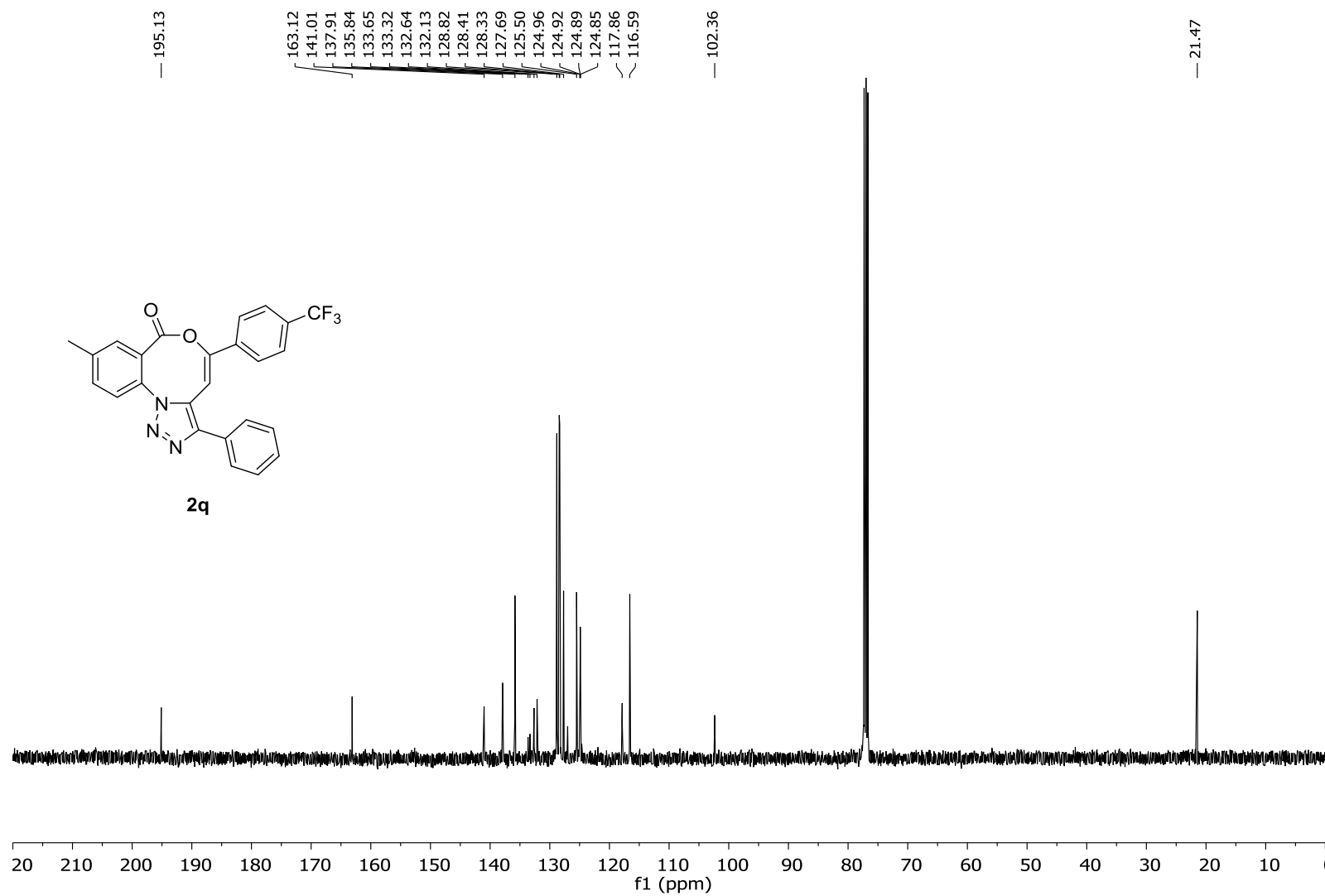
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	ndb	e ⁻ Conf	N-Rule	Adduct
410.1505	1	C ₂₅ H ₂₀ N ₃ O ₃	410.1499	-1.5	7.1	1	100.00	17.5	even	ok	M+H

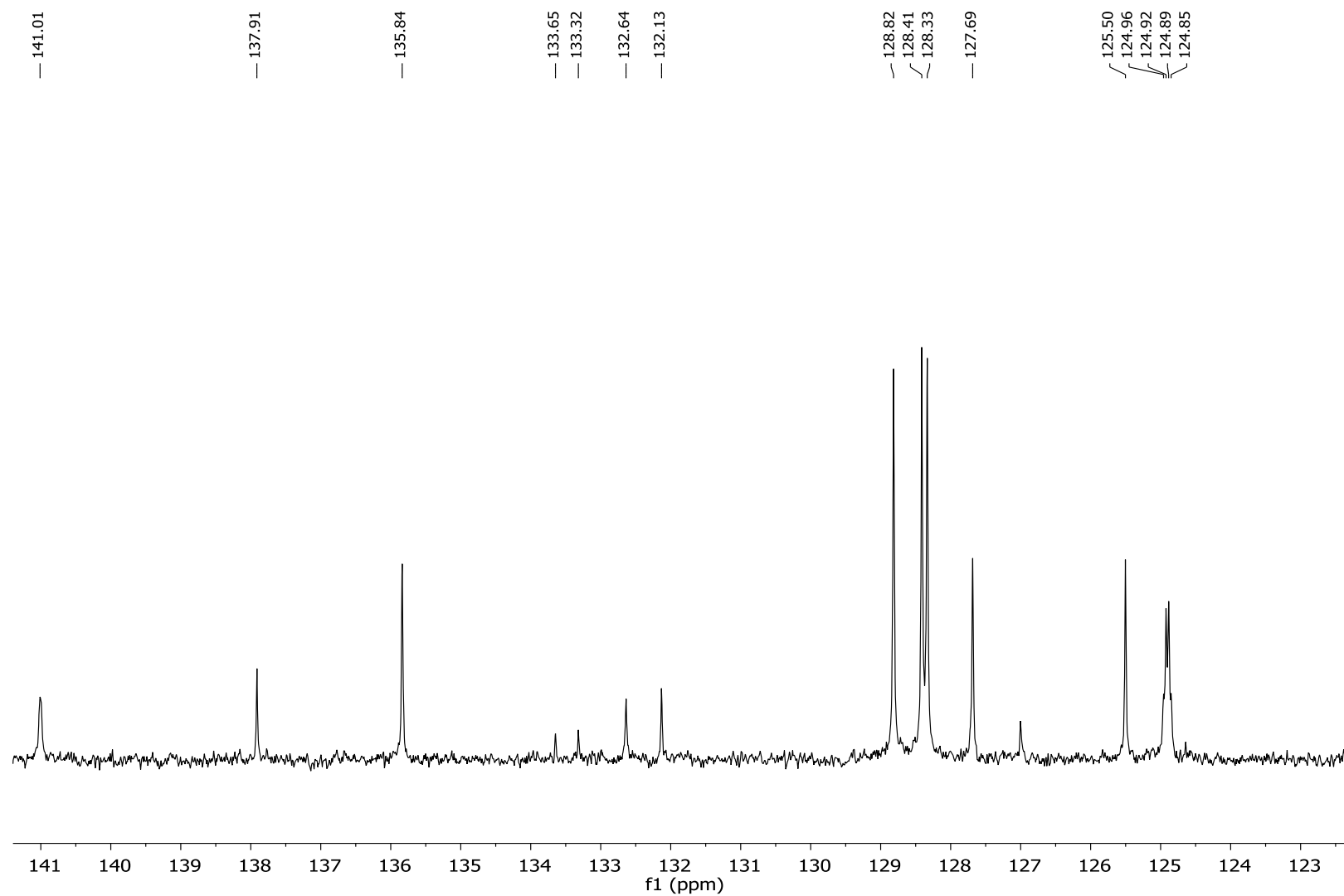
HRMS spectrum of compound **2p**



¹H NMR Spectrum (400MHz) of compound **2q** in CDCl₃



¹³C NMR Spectrum (101 MHz) of compound 2q in CDCl₃

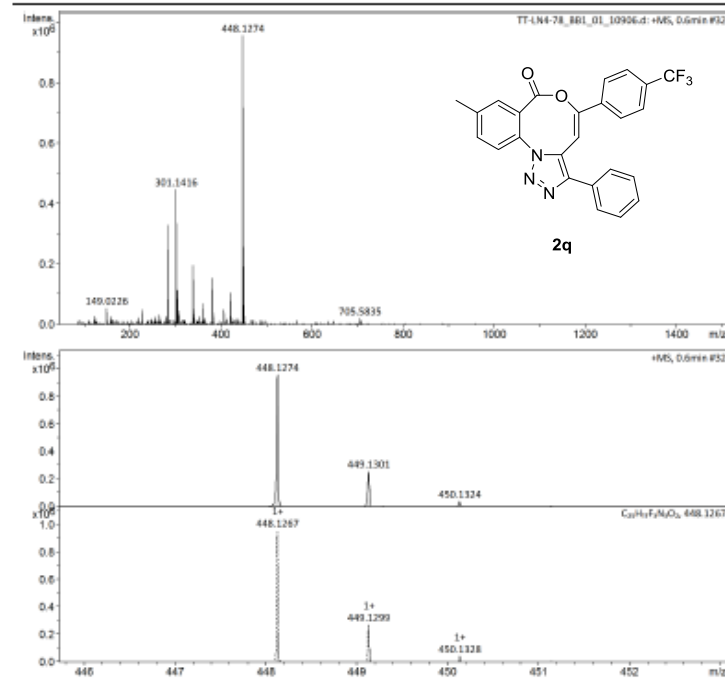


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2q** in CDCl_3

Display Report

Analysis Info		Acquisition Date	9/2/2016 10:53:51 AM	
Analysis Name	D:\Data\ncbu serv\data\2016\20160902\TT-LN4-78_BB1_01_10906.d			
Method	Small molecule.m	Operator	NCTU	
Sample Name	TT-LN4-78	Instrument	Impact HD	1819695.00164
Comment				

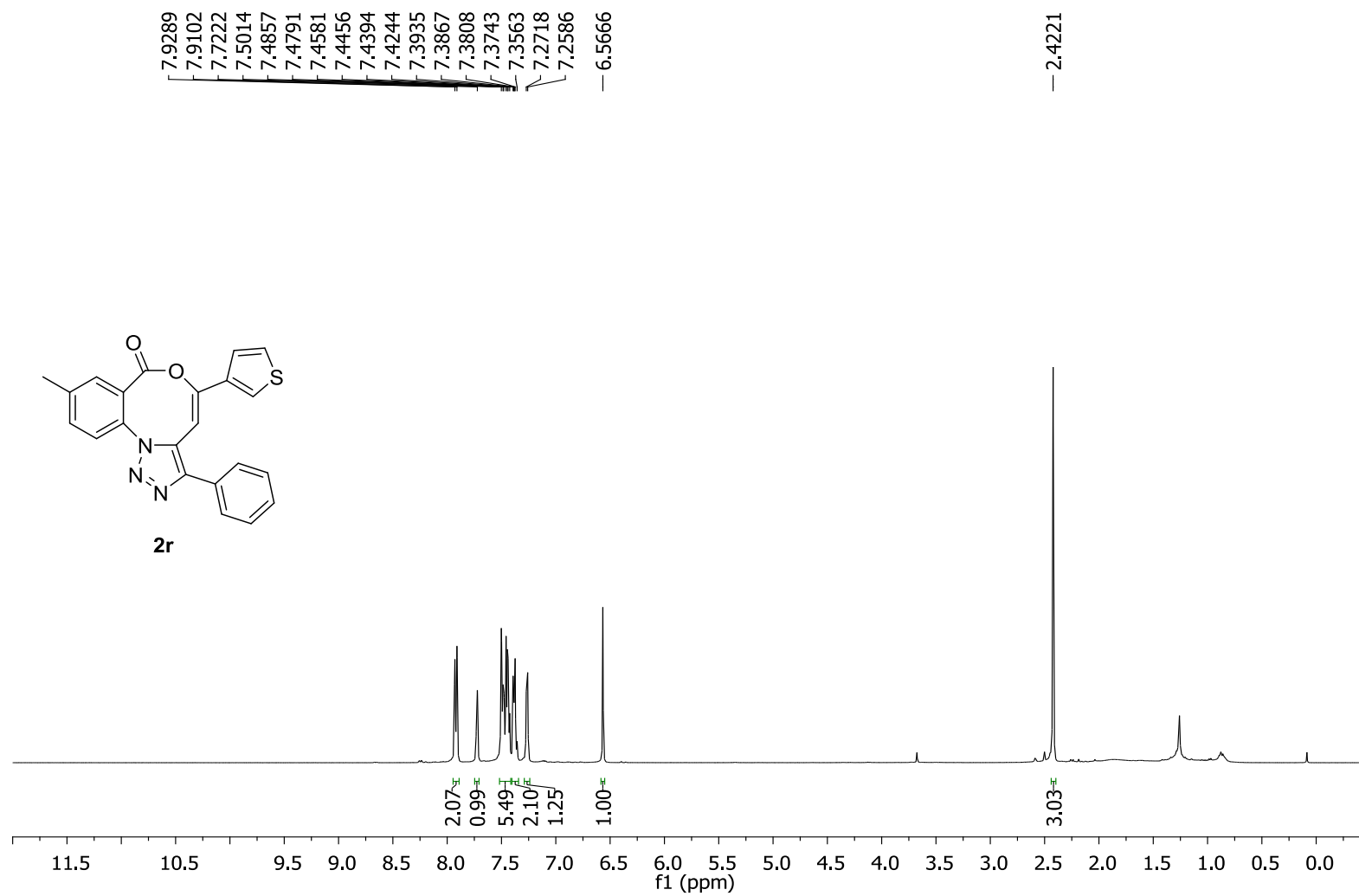
Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	1500 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Header	0 °C



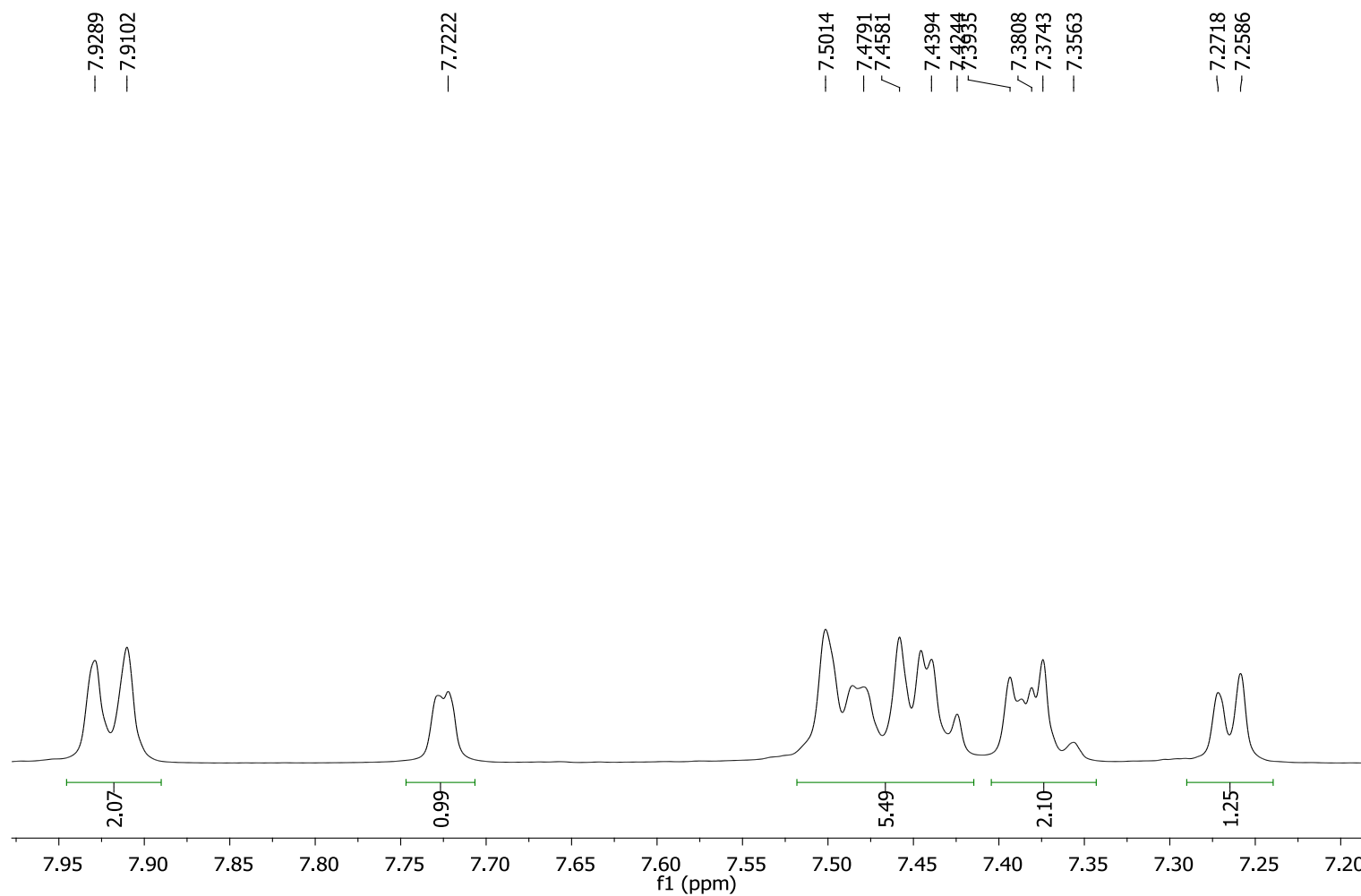
Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	#Sigma	Score	rd	e ⁻ Conf	N-Rule	Adduct
448.1274	1	C ₂₅ H ₁₇ F ₃ N ₃ O ₂	448.1267	1.5	8.5	1	100.00	17.5	even	ok	M+H

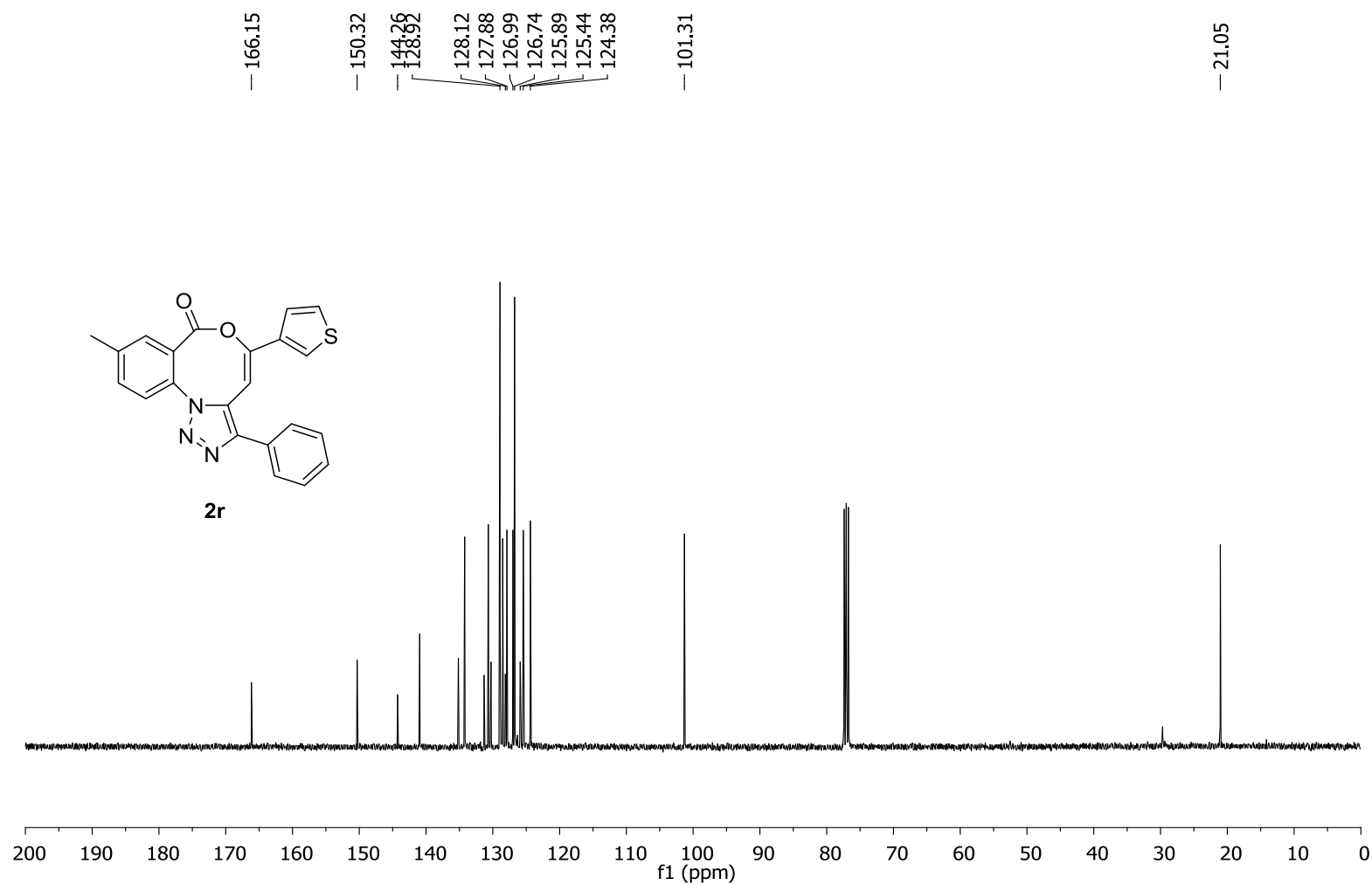
HRMS of compound **2q**



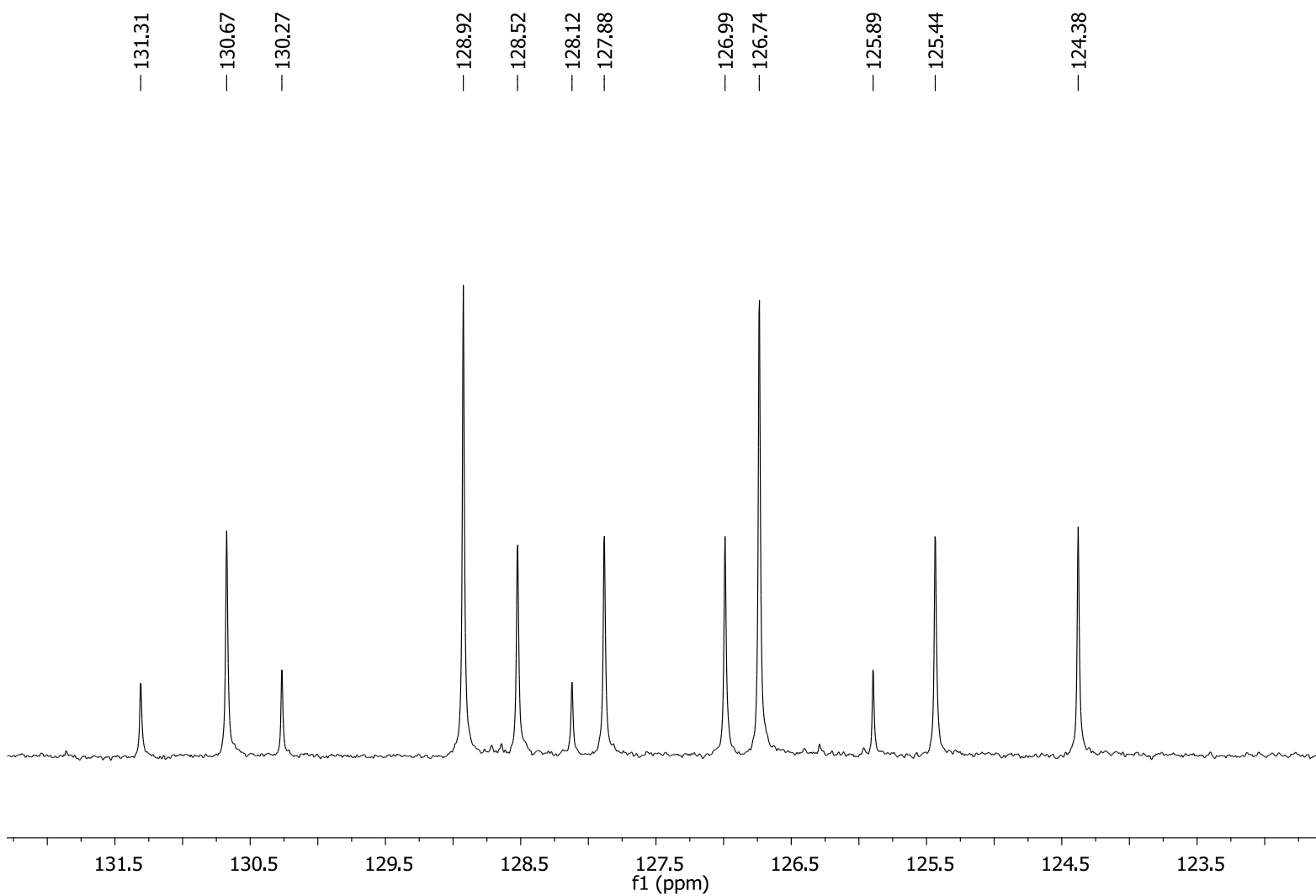
¹H NMR Spectrum (400MHz) of compound **2r** in CDCl₃



Expansion of ^1H NMR Spectrum (400MHz) of compound **2r** in CDCl_3



¹³C NMR Spectrum (101 MHz) of compound **2r** in CDCl₃

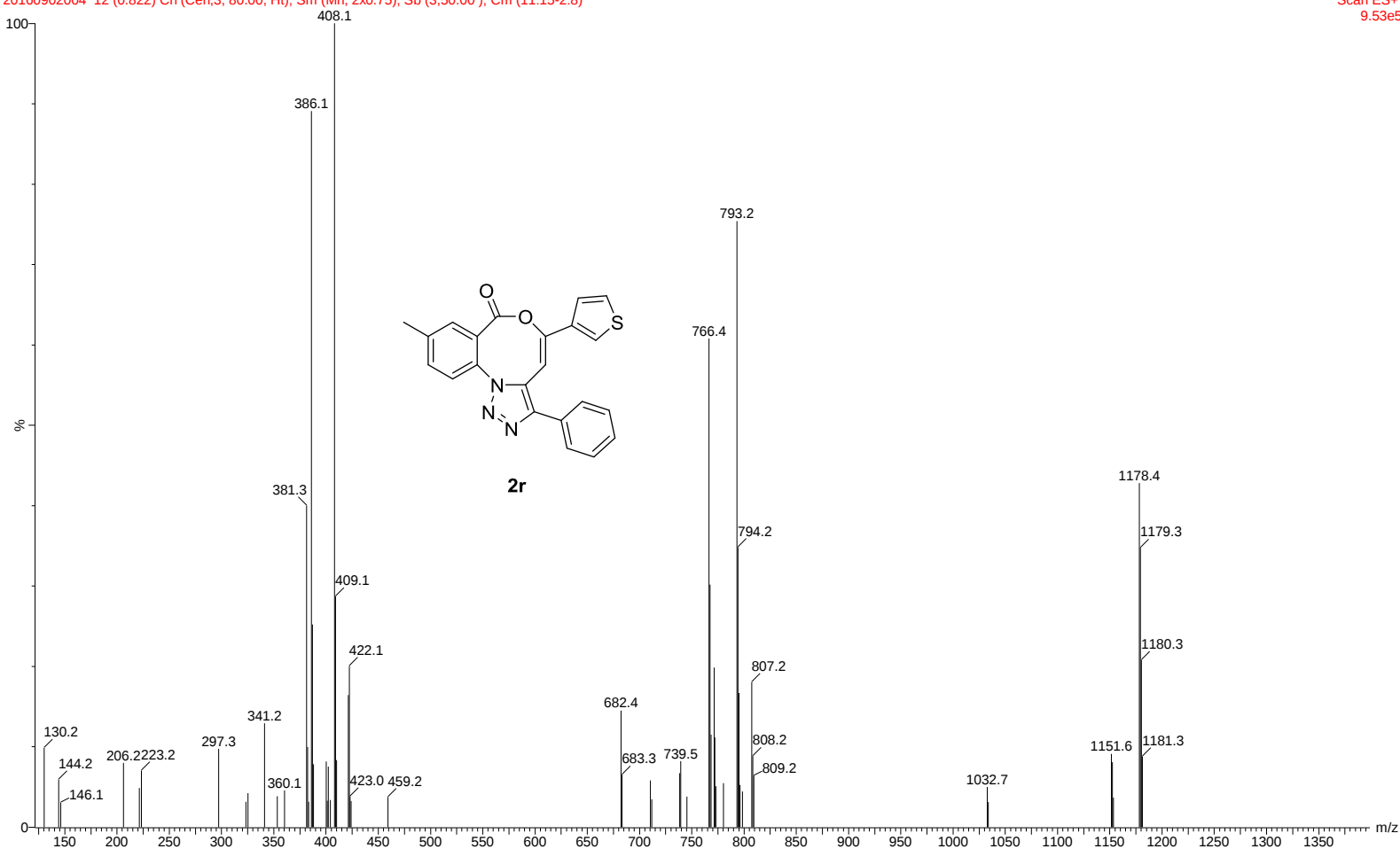


Expansion of ^{13}C NMR Spectrum (101 MHz) of compound **2r** in CDCl_3

TT-LNS-80

20160902004 12 (0.822) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (11:15-2:8)

Scan ES+
9.53e5

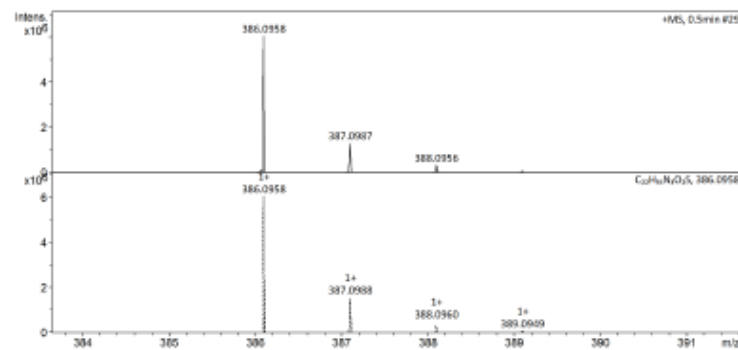
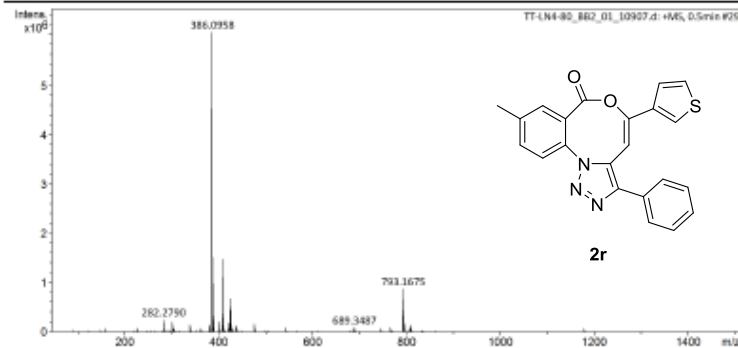


LRMS of compound **2r**

Display Report

Analysis Info		Acquisition Date 9/2/2016 10:58:12 AM	
Analysis Name D:\Data\ctu service\data\2016\20160902\TT-LN4-80_BB2_01_10907.d		Operator NCTU	
Method Small molecule.m		Instrument Impact HD	
Sample Name TT-LN4-80		1819696.00164	
Comment			

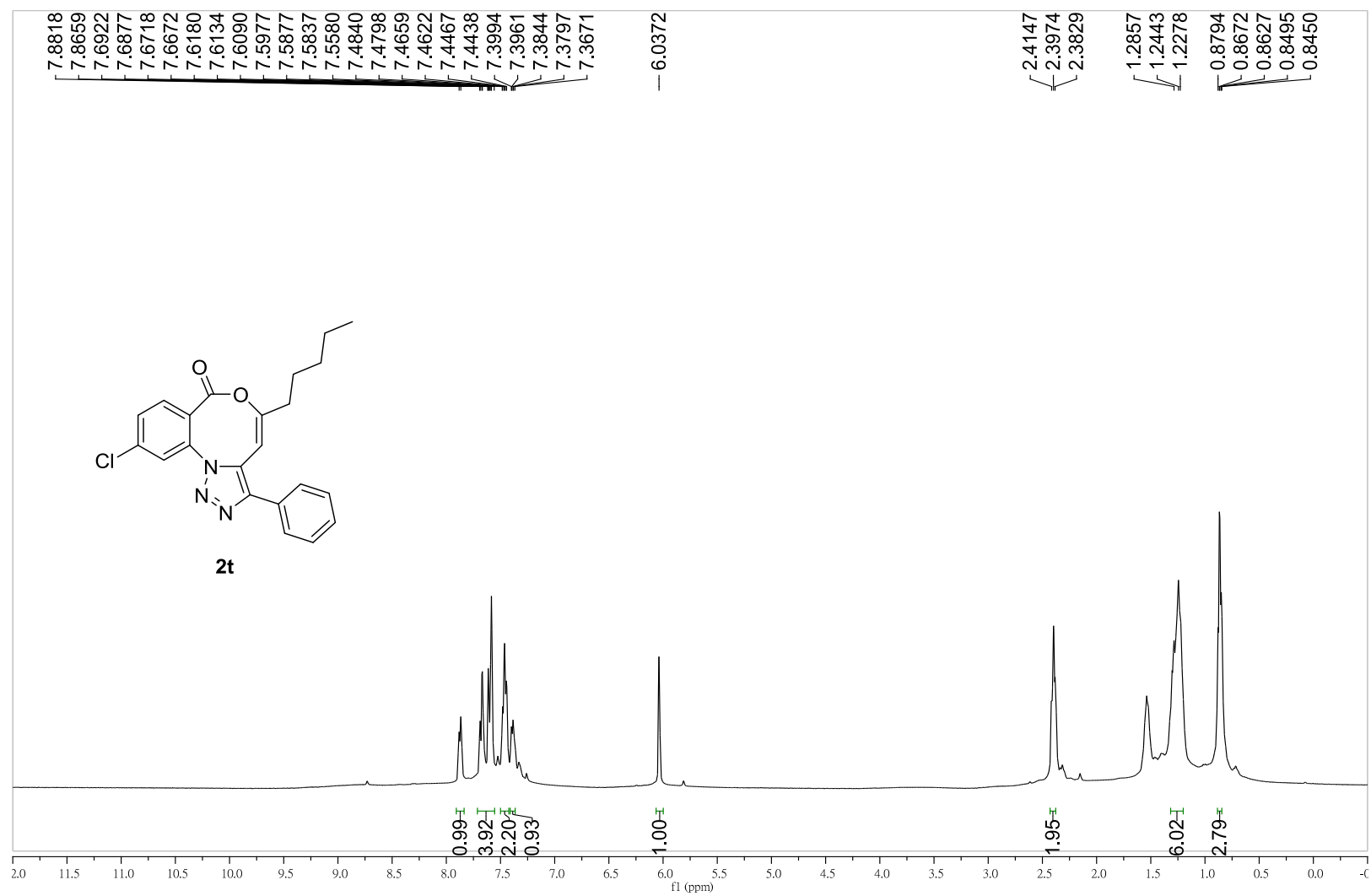
Acquisition Parameter			
Source Type	ESI	Ion Polarity	Positive
Focus	Active	Set Capillary	4500 V
Scan Begin	50 m/z	Set End Plate Offset	-500 V
Scan End	1500 m/z	Set Charging Voltage	2000 V
		Set Corona	0 nA
		Set Nebulizer	1.0 Bar
		Set Dry Heater	200 °C
		Set Dry Gas	8.0 l/min
		Set Divert Valve	Waste
		Set AP/CI Heater	0 °C



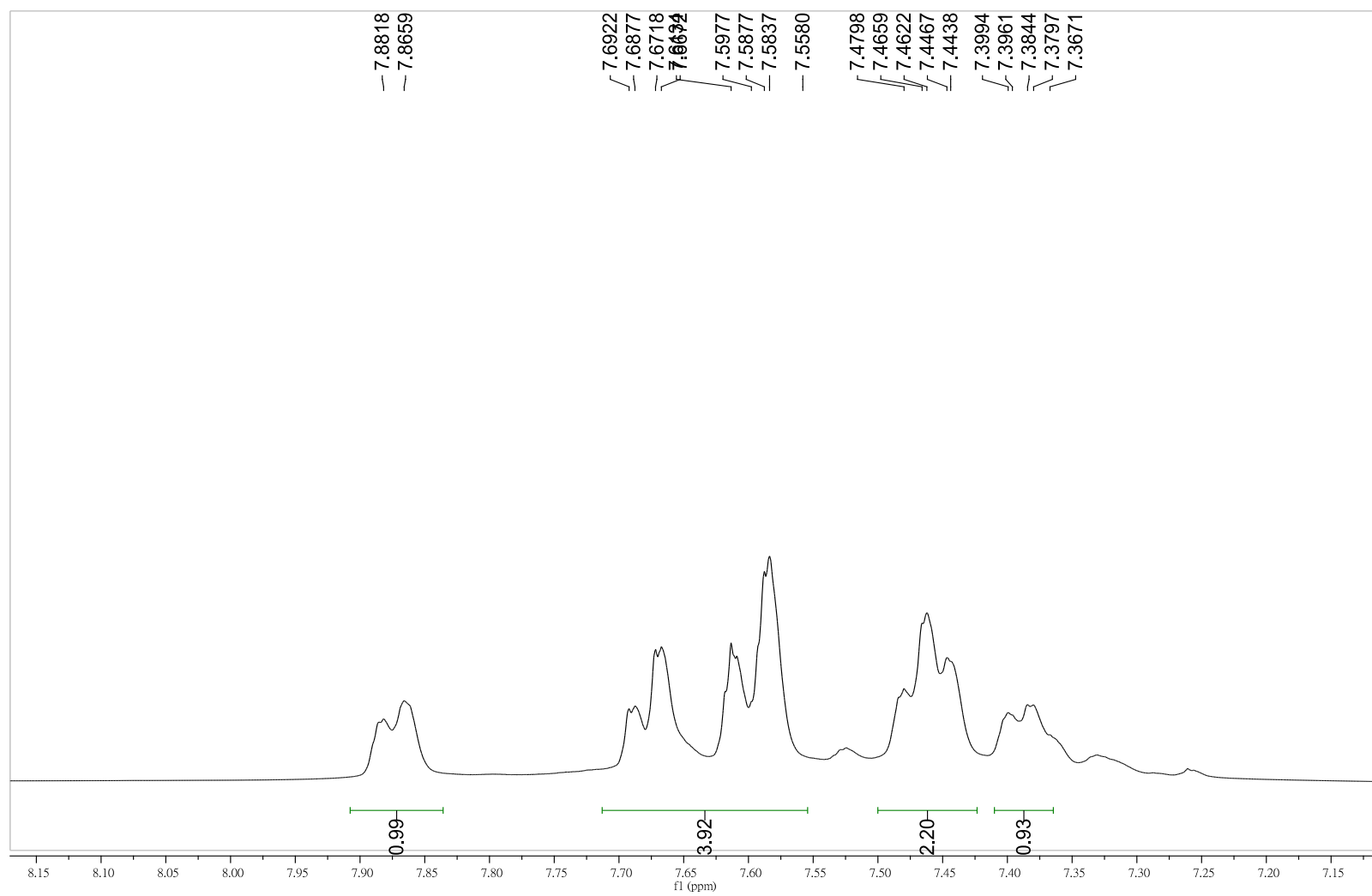
Display Report

Mass. m/z	#	Ion Formula	m/z	em [ppm]	mSigma	# Sigma	Score	rd	e ⁻ Conf	N-Rule	Adduct
386.0958	1	C ₂₂ H ₁₈ N ₃ O ₂ S	386.0958	0.0	25.7	1	100.00	18.5	even	ok	M+H

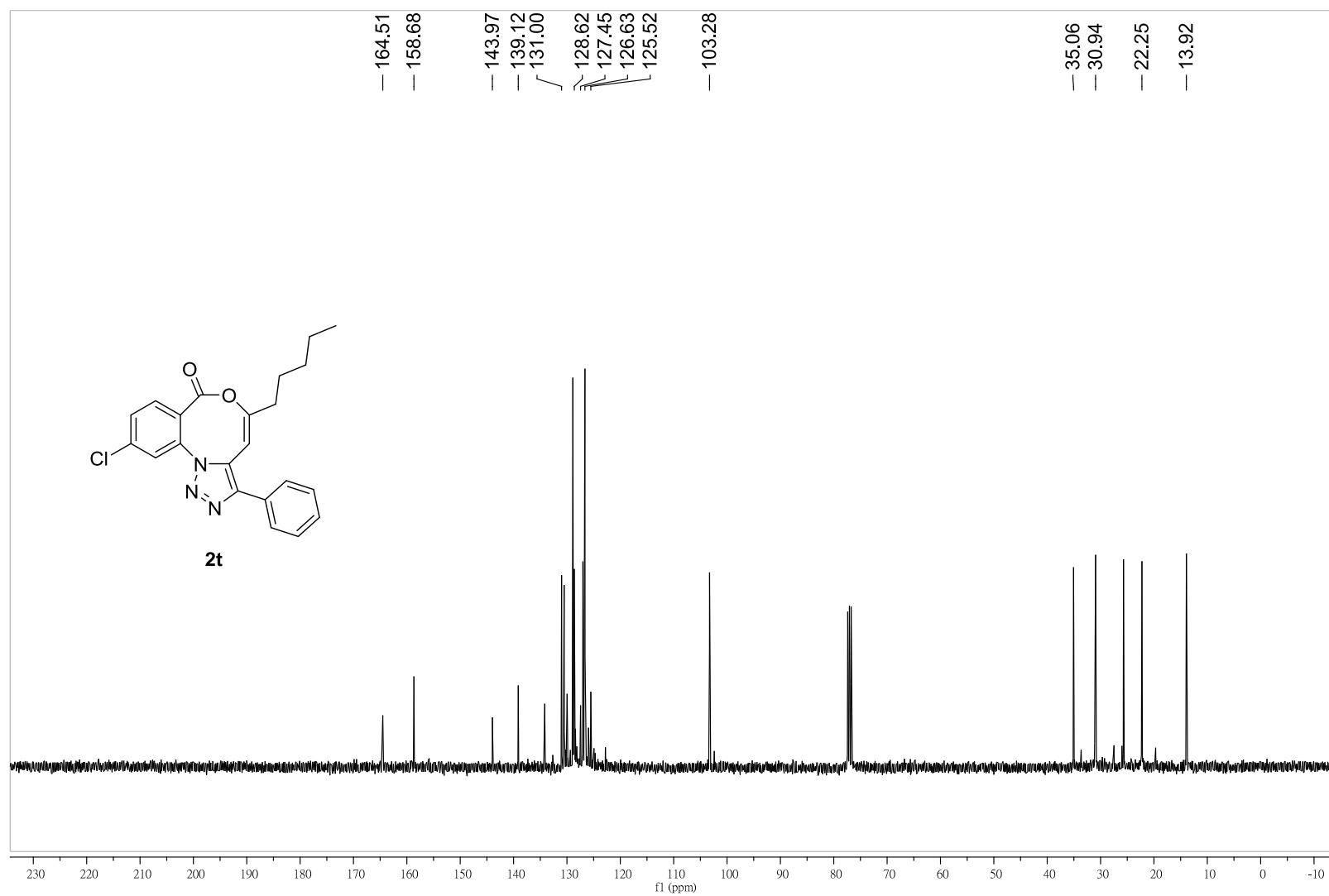
HRMS of compound 2r



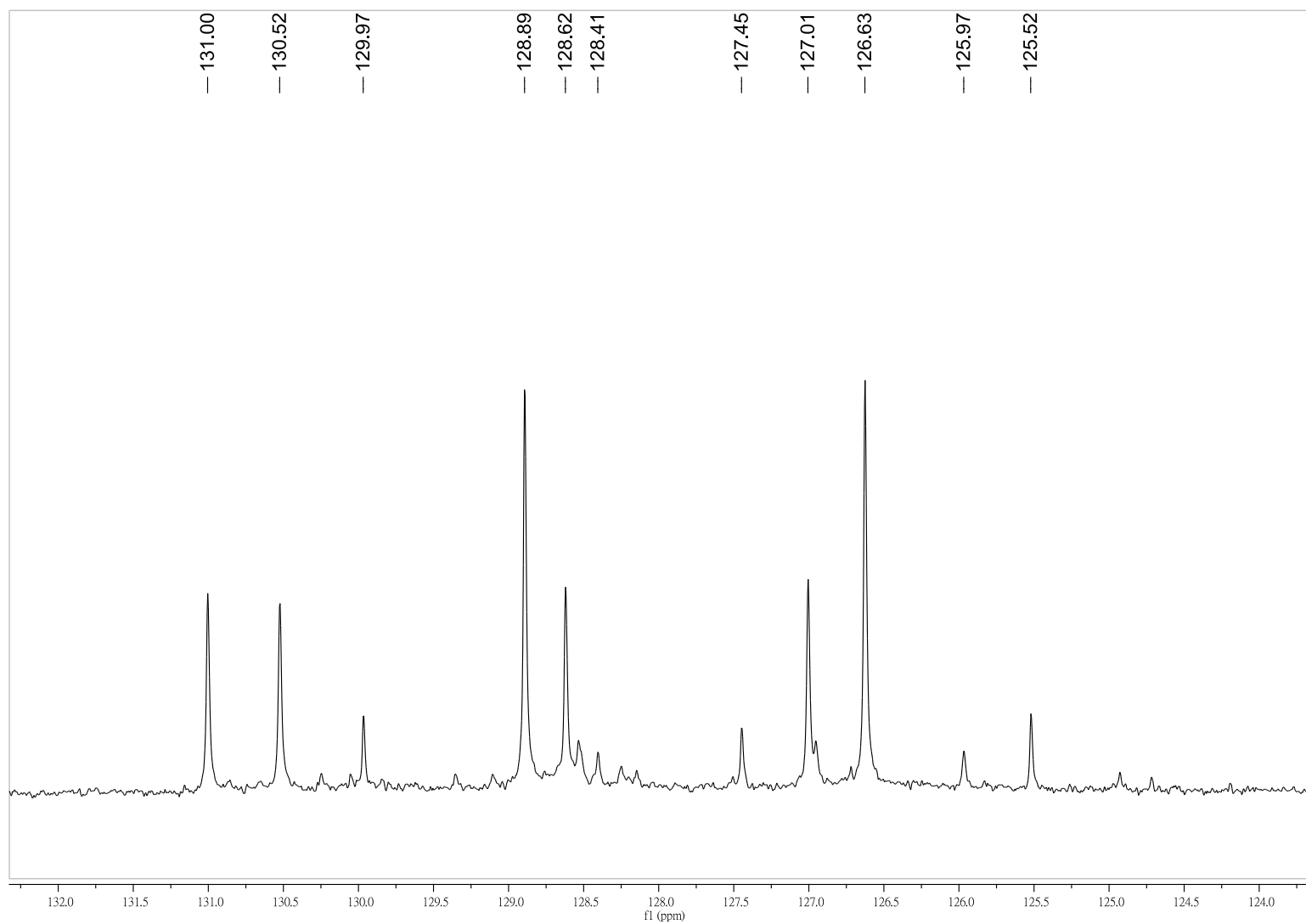
¹H NMR Spectrum (400MHz) of compound **2t** in CDCl₃



Expansion of ^1H NMR Spectrum (400MHz) of compound **2t** in CDCl_3



¹³C NMR Spectrum (75 MHz) of compound **2t** in CDCl₃

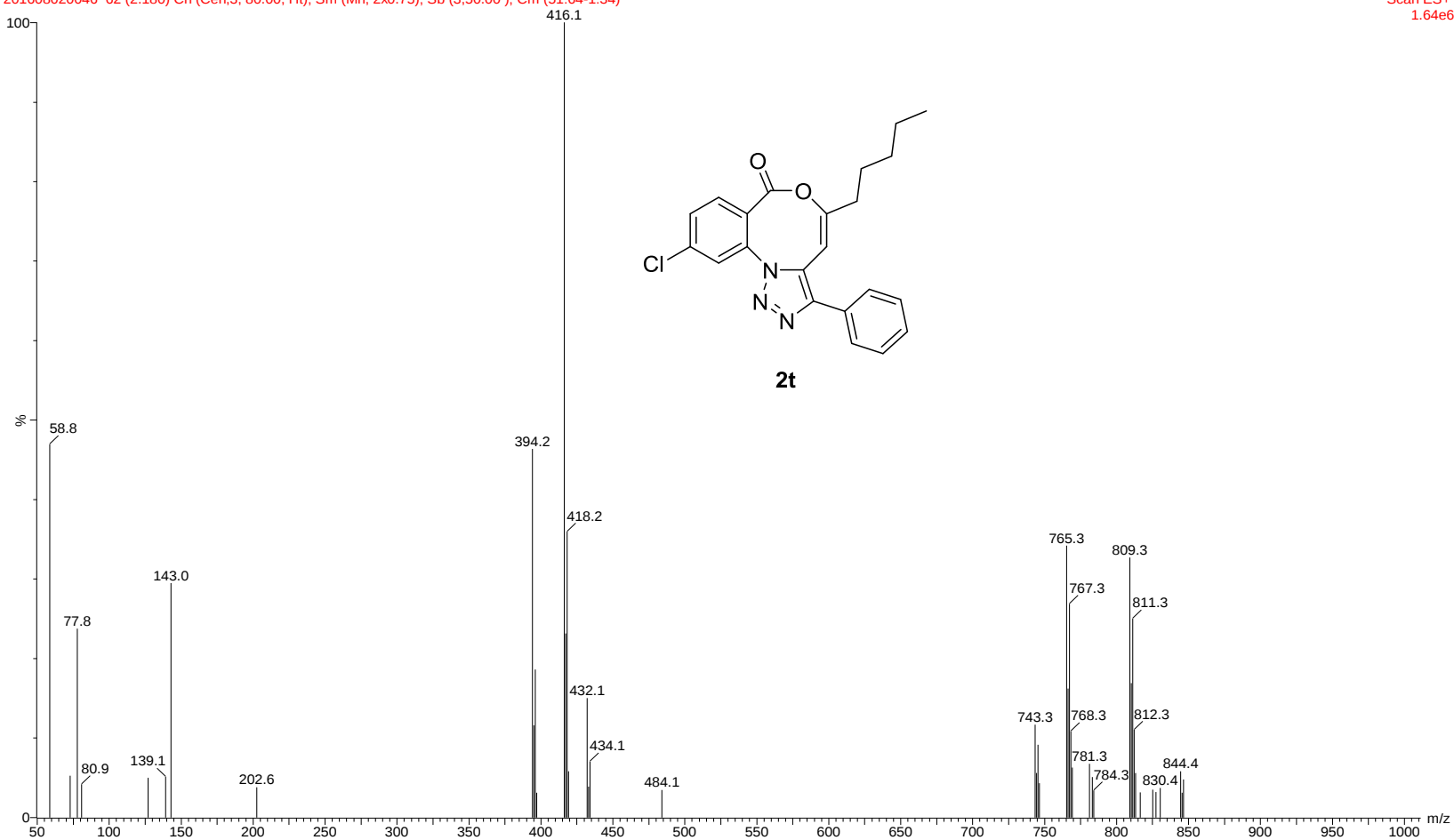


Expansion of ^{13}C NMR Spectrum (75 MHz) of compound **2t** in CDCl_3

3p45

201608020046 62 (2.180) Cn (Cen,3, 80.00, Ht); Sm (Mn, 2x0.75); Sb (3,50.00); Cm (51.64-1:34)

Scan ES+
1.64e6

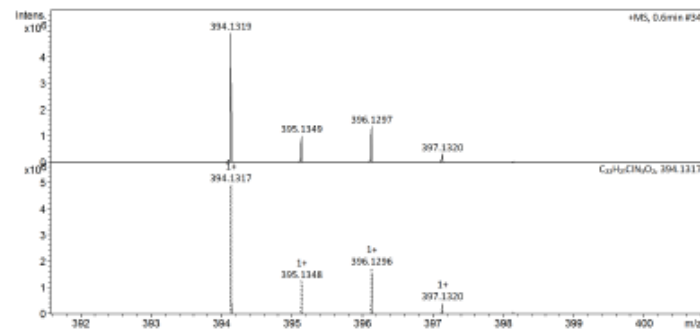
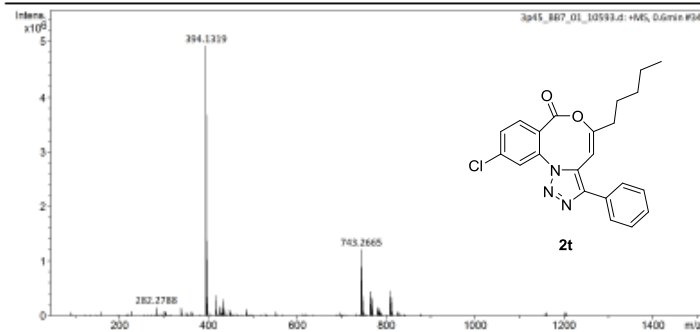


LRMS of compound 2t

Display Report

Analysis Info
 Analysis Name: D:\Data\inctu service\data\2016\20160805\3p45_BB7_01_10593.d Acquisition Date: 8/5/2016 12:13:26 PM
 Method: Small molecule.m Operator: NCTU
 Sample Name: 3p45 Instrument: Impact HD 1819696.D0164
 Comment:

Acquisition Parameter
 Source Type: ESI Ion Polarity: Positive Set Nebulizer: 1.0 Bar
 Focus: Active Set Capillary: 4500 V Set Dry Heater: 200 °C
 Scan Begin: 50 m/z Set End Plate Offset: -500 V Set Dry Gas: 6.0 l/min
 Scan End: 1500 m/z Set Charging Voltage: 2000 V Set Divert Valve: Waste
 Set Corona: 0 nA Set APCI Heater: 0 °C

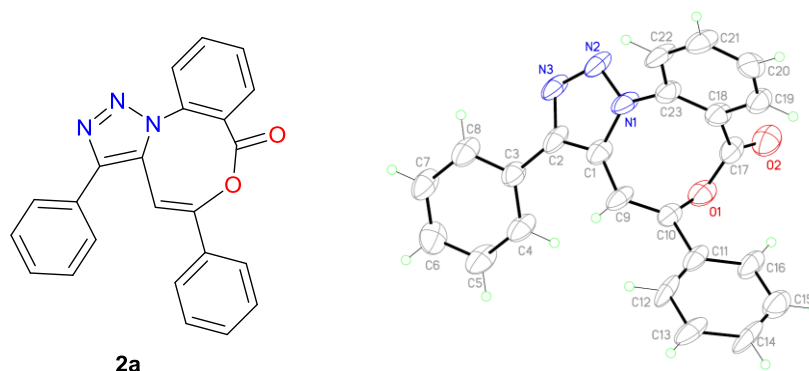


Display Report

Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rd	e ⁻ Conf	N-Rule	Adduct
394.1319	1	C ₂₂ H ₂₁ ClN ₃ O ₂	394.1317	0.5	40.8	1	100.00	13.5	even	ok	M+H

HRMS of compound 2t

X-ray crystal data of compound 2a



ORTEP diagram of compound 2a. Atomic displacement ellipsoids are drawn at the 50% probability level

CCDC no. of **2a**: 1429500

Table 1. Crystal data and structure refinement for 150925LTS.

Identification code	150925lts	
Empirical formula	C ₂₃ H ₁₅ N ₃ O ₂	
Formula weight	365.38	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	P -4 21 c	
Unit cell dimensions	a = 21.6276(14) Å	$\alpha = 90^\circ$.
	b = 21.6276(14) Å	$\beta = 90^\circ$.
	c = 8.1936(6) Å	$\gamma = 90^\circ$.
Volume	3832.6(6) Å ³	
Z	8	
Density (calculated)	1.266 Mg/m ³	
Absorption coefficient	0.083 mm ⁻¹	
F(000)	1520	

Crystal size	0.15 x 0.07 x 0.07 mm ³
Theta range for data collection	1.332 to 26.411°.
Index ranges	-26<=h<=26, -26<=k<=26, -9<=l<=9
Reflections collected	24797
Independent reflections	3828 [R(int) = 0.0787]
Completeness to theta = 25.242°	98.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9485 and 0.7895
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3828 / 0 / 255
Goodness-of-fit on F ²	1.053
Final R indices [I>2sigma(I)]	R1 = 0.0981, wR2 = 0.2457
R indices (all data)	R1 = 0.1274, wR2 = 0.2641
Absolute structure parameter	0(5)
Extinction coefficient	0.116(14)
Largest diff. peak and hole	0.703 and -0.548 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 150925LTS. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
C(1)	5670(3)	1670(3)	3509(10)	43(2)
C(2)	6261(3)	1432(3)	3417(11)	49(2)
C(3)	6829(3)	1645(3)	4236(10)	44(2)
C(4)	6873(3)	2237(3)	4863(11)	52(2)
C(5)	7404(3)	2421(3)	5642(12)	60(2)
C(6)	7910(3)	2026(4)	5766(12)	61(2)
C(7)	7872(3)	1436(3)	5137(12)	60(2)
C(8)	7335(3)	1244(3)	4338(12)	55(2)
C(9)	5361(3)	2147(3)	4427(12)	48(2)
C(10)	5006(3)	2577(3)	3751(10)	41(2)
C(11)	4619(3)	3042(3)	4584(13)	48(2)
C(12)	4645(3)	3118(3)	6264(13)	53(2)
C(13)	4263(3)	3538(3)	7109(13)	61(3)
C(14)	3849(3)	3900(3)	6118(17)	70(3)
C(15)	3818(3)	3822(3)	4445(14)	60(2)
C(16)	4198(3)	3395(3)	3677(13)	58(2)
C(17)	4651(3)	2341(3)	1028(12)	44(2)
C(18)	4342(3)	1756(3)	1664(10)	44(2)
C(19)	3713(3)	1705(3)	1462(11)	50(2)
C(20)	3415(3)	1173(3)	1858(11)	51(2)
C(21)	3736(3)	668(3)	2459(11)	56(2)
C(22)	4376(3)	719(3)	2674(11)	56(2)
C(23)	4674(3)	1258(3)	2301(10)	44(2)
N(1)	5328(2)	1279(2)	2468(9)	49(2)
N(2)	5683(2)	831(2)	1853(9)	48(2)
N(3)	6257(2)	930(2)	2457(9)	49(2)
O(1)	5025(2)	2657(2)	2027(7)	50(2)
O(2)	4583(2)	2517(2)	-323(9)	61(2)

Table 3. Bond lengths [Å] and angles [°] for 150925LTS.

C(1)-C(2)	1.381(8)
C(1)-N(1)	1.410(9)
C(1)-C(9)	1.441(10)
C(2)-N(3)	1.341(10)
C(2)-C(3)	1.473(9)
C(3)-C(4)	1.382(9)
C(3)-C(8)	1.399(9)
C(4)-C(5)	1.374(10)
C(4)-H(4)	0.9500
C(5)-C(6)	1.392(10)
C(5)-H(5)	0.9500
C(6)-C(7)	1.379(11)
C(6)-H(6)	0.9500
C(7)-C(8)	1.397(11)
C(7)-H(7)	0.9500
C(8)-H(8)	0.9500
C(9)-C(10)	1.327(9)
C(9)-H(9)	0.9500
C(10)-O(1)	1.423(9)
C(10)-C(11)	1.476(10)
C(11)-C(12)	1.388(12)
C(11)-C(16)	1.402(10)
C(12)-C(13)	1.409(11)
C(12)-H(12)	0.9500
C(13)-C(14)	1.440(12)
C(13)-H(13)	0.9500
C(14)-C(15)	1.382(14)
C(14)-H(14)	0.9500
C(15)-C(16)	1.388(11)
C(15)-H(15)	0.9500
C(16)-H(16)	0.9500
C(17)-O(2)	1.179(10)
C(17)-O(1)	1.338(9)
C(17)-C(18)	1.523(9)

C(18)-C(19)	1.377(9)
C(18)-C(23)	1.395(9)
C(19)-C(20)	1.357(9)
C(19)-H(19)	0.9500
C(20)-C(21)	1.385(11)
C(20)-H(20)	0.9500
C(21)-C(22)	1.401(10)
C(21)-H(21)	0.9500
C(22)-C(23)	1.366(9)
C(22)-H(22)	0.9500
C(23)-N(1)	1.423(8)
N(1)-N(2)	1.335(7)
N(2)-N(3)	1.353(7)

C(2)-C(1)-N(1)	103.2(6)
C(2)-C(1)-C(9)	136.5(7)
N(1)-C(1)-C(9)	120.1(5)
N(3)-C(2)-C(1)	109.2(6)
N(3)-C(2)-C(3)	121.8(5)
C(1)-C(2)-C(3)	129.0(7)
C(4)-C(3)-C(8)	119.9(6)
C(4)-C(3)-C(2)	121.1(6)
C(8)-C(3)-C(2)	119.0(6)
C(5)-C(4)-C(3)	119.9(6)
C(5)-C(4)-H(4)	120.0
C(3)-C(4)-H(4)	120.0
C(4)-C(5)-C(6)	120.9(7)
C(4)-C(5)-H(5)	119.6
C(6)-C(5)-H(5)	119.6
C(7)-C(6)-C(5)	119.6(7)
C(7)-C(6)-H(6)	120.2
C(5)-C(6)-H(6)	120.2
C(6)-C(7)-C(8)	120.1(6)
C(6)-C(7)-H(7)	120.0
C(8)-C(7)-H(7)	120.0
C(7)-C(8)-C(3)	119.6(7)

C(7)-C(8)-H(8)	120.2
C(3)-C(8)-H(8)	120.2
C(10)-C(9)-C(1)	123.5(8)
C(10)-C(9)-H(9)	118.3
C(1)-C(9)-H(9)	118.3
C(9)-C(10)-O(1)	118.9(6)
C(9)-C(10)-C(11)	127.8(8)
O(1)-C(10)-C(11)	113.1(6)
C(12)-C(11)-C(16)	119.2(7)
C(12)-C(11)-C(10)	121.1(6)
C(16)-C(11)-C(10)	119.7(8)
C(11)-C(12)-C(13)	122.7(7)
C(11)-C(12)-H(12)	118.7
C(13)-C(12)-H(12)	118.7
C(12)-C(13)-C(14)	116.0(10)
C(12)-C(13)-H(13)	122.0
C(14)-C(13)-H(13)	122.0
C(15)-C(14)-C(13)	121.6(8)
C(15)-C(14)-H(14)	119.2
C(13)-C(14)-H(14)	119.2
C(14)-C(15)-C(16)	120.1(8)
C(14)-C(15)-H(15)	120.0
C(16)-C(15)-H(15)	120.0
C(15)-C(16)-C(11)	120.5(9)
C(15)-C(16)-H(16)	119.7
C(11)-C(16)-H(16)	119.7
O(2)-C(17)-O(1)	119.0(6)
O(2)-C(17)-C(18)	122.3(7)
O(1)-C(17)-C(18)	118.6(7)
C(19)-C(18)-C(23)	119.4(6)
C(19)-C(18)-C(17)	117.4(6)
C(23)-C(18)-C(17)	123.0(6)
C(20)-C(19)-C(18)	120.6(7)
C(20)-C(19)-H(19)	119.7
C(18)-C(19)-H(19)	119.7
C(19)-C(20)-C(21)	121.0(6)

C(19)-C(20)-H(20)	119.5
C(21)-C(20)-H(20)	119.5
C(20)-C(21)-C(22)	118.6(6)
C(20)-C(21)-H(21)	120.7
C(22)-C(21)-H(21)	120.7
C(23)-C(22)-C(21)	120.3(6)
C(23)-C(22)-H(22)	119.8
C(21)-C(22)-H(22)	119.8
C(22)-C(23)-C(18)	120.0(6)
C(22)-C(23)-N(1)	118.3(6)
C(18)-C(23)-N(1)	121.5(6)
N(2)-N(1)-C(1)	111.3(5)
N(2)-N(1)-C(23)	120.8(5)
C(1)-N(1)-C(23)	126.7(6)
N(1)-N(2)-N(3)	106.0(5)
C(2)-N(3)-N(2)	110.4(5)
C(17)-O(1)-C(10)	121.9(6)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 150925LTS. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	33(3)	29(3)	67(5)	5(3)	4(3)	-4(3)
C(2)	29(3)	21(3)	97(7)	14(3)	0(4)	-2(2)
C(3)	35(3)	31(3)	66(5)	4(3)	-4(3)	-1(3)
C(4)	34(3)	27(3)	97(7)	10(3)	6(4)	-4(3)
C(5)	35(4)	31(3)	114(7)	11(4)	7(4)	-9(3)
C(6)	36(4)	56(5)	90(7)	15(4)	-7(4)	-11(3)
C(7)	28(3)	39(4)	114(8)	11(4)	-2(4)	0(3)
C(8)	36(4)	34(3)	95(7)	11(4)	3(4)	-3(3)
C(9)	21(3)	23(3)	100(7)	-3(3)	4(3)	0(2)
C(10)	20(3)	28(3)	75(6)	-3(3)	-2(3)	-4(2)
C(11)	26(3)	19(3)	99(8)	8(3)	-4(4)	-4(2)
C(12)	22(3)	28(3)	109(8)	20(4)	8(4)	9(2)
C(13)	38(4)	22(3)	124(8)	3(4)	16(4)	-1(3)
C(14)	23(3)	27(3)	160(11)	10(5)	9(5)	12(3)
C(15)	45(4)	46(4)	89(8)	-6(5)	-3(4)	11(3)
C(16)	27(3)	35(3)	111(8)	-1(4)	-3(4)	8(3)
C(17)	21(3)	33(3)	78(6)	5(4)	-1(3)	-2(2)
C(18)	33(3)	33(3)	66(6)	-5(3)	0(3)	-4(3)
C(19)	37(3)	37(3)	77(6)	-4(4)	-4(3)	4(3)
C(20)	29(3)	52(4)	72(6)	-13(4)	-5(3)	-7(3)
C(21)	38(4)	29(3)	101(7)	-10(4)	10(4)	-6(3)
C(22)	39(4)	21(3)	108(7)	-6(4)	6(4)	4(2)
C(23)	34(3)	36(3)	63(5)	-12(3)	2(3)	8(3)
N(1)	33(3)	21(2)	92(5)	-5(3)	-2(3)	-1(2)
N(2)	28(3)	22(2)	94(5)	2(3)	6(3)	3(2)
N(3)	29(3)	20(2)	98(5)	4(3)	2(3)	1(2)
O(1)	34(2)	33(2)	82(5)	1(2)	3(3)	-5(2)
O(2)	49(3)	47(3)	87(5)	11(3)	0(3)	-4(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for 150925LTS.

	x	y	z	U(eq)
H(4)	6536	2516	4756	63
H(5)	7427	2824	6102	72
H(6)	8279	2162	6281	73
H(7)	8211	1160	5246	72
H(8)	7313	843	3866	66
H(9)	5415	2154	5578	58
H(12)	4932	2877	6870	64
H(13)	4278	3580	8262	73
H(14)	3591	4198	6626	84
H(15)	3535	4062	3821	72
H(16)	4173	3342	2528	69
H(19)	3484	2044	1041	60
H(20)	2979	1147	1722	62
H(21)	3526	295	2719	67
H(22)	4605	377	3081	67