Supporting Information for:

Metal- and oxidant-free C−H Bond Halogenation/Imidation of N-heterocycle

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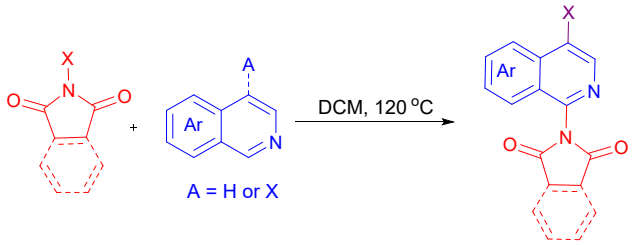
Part 1. Experimental Procedures and Analytical Data

General Methods:

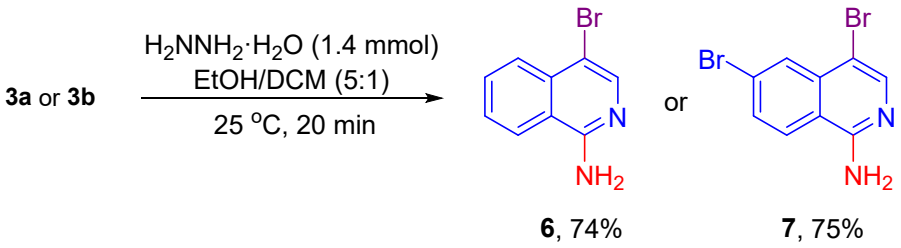
All experiments were conducted in air, utilizing commercially available analytical reagents and solvents that do not require further purification. The ^{1}H NMR and ^{13}C NMR spectra were recorded at 400 MHz and 101 MHz, using a Bruker Ascend 400 MHz NMR spectrometer. TMS was employed as the reference for chemical shifts. High-resolution mass spectrometry (HRMS) was performed on an Agilent Technologies LC-TOF instrument. X-ray crystallography of compound **3a** was carried out using a Bruker Smart Apex CCD area detector diffractometer with graphite-monochromated Mo Kα radiation at a temperature of 23(2) °C.

General procedure for the synthesis of product 3 and 5:



NBS or NPS (2 mmol) were added to a solution of substrate (1 mmol) in dichloromethane (4 mL), and the solution was stirred at 120°C for 12 hours. After the reaction completed, extract the mixture with saturated sodium bicarbonate solution (30 mL × 3 times). Dry the combined organic layers over anhydrous sodium sulfate. Remove the solvent under reduced pressure to obtain the crude product. Purify the crude material by flash silica gel column chromatography to isolate product **3** (PE:EA = 30:1 → 10:1) and Product 5 (PE:EA = 10:1 → 2:1).

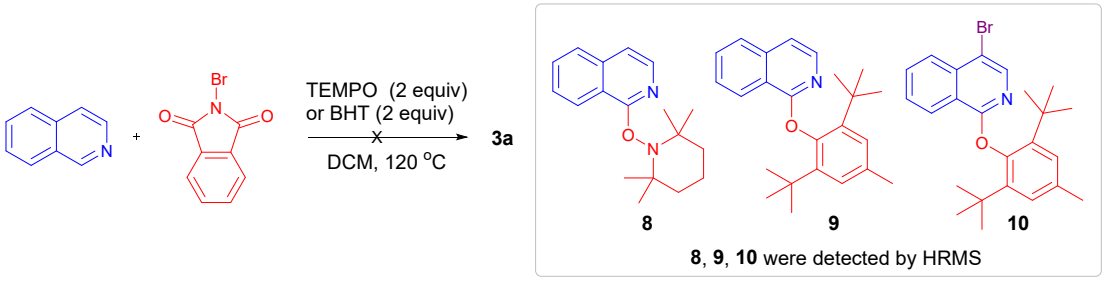
Procedure for the synthesis of compound 6 and 7:



6, 74% **7**, 75%

To the solution of product **3a** or **3b** in ethanol/dichloromethane (2 mL / 0.4 mL), add hydrazine hydrate (65 wt%, 104 μL, 1.4 mmol). Stir the resulting mixture at 25 °C for 20 minutes. A white precipitate is observed upon reaction completion. After the reaction is complete, add 1 mL of H_2O and extract with diethyl ether (30 mL × 3). Dry the combined organic layers over anhydrous sodium sulfate and concentrate under reduced pressure. Purify the target compound by flash silica gel column chromatography using ethyl acetate/petroleum ether (10:1 → 3:1) to obtain product **6** or **7**.

Radical trapping experiments

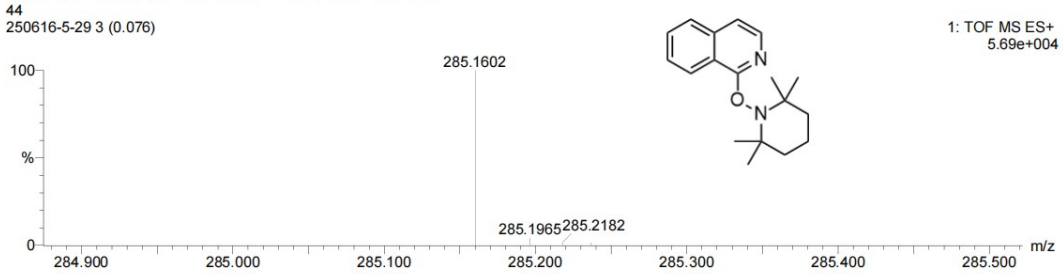


Isoquinoline (1 mmol, 1 equiv), 2-bromoisoindoline-1,3-dione (2 mmol, 2 equiv), 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO) (2 mmol, 2 equiv) or 2,6-di-tert-butyl-4-methylphenol (BHT) (2 mmol, 2 equiv) and DCM (4 mL), were added in a Schlenk tube, and the mixture was stirred for 12 hours at 120 °C using a heating mantle under nitrogen atmosphere. The target compound was detected by HMRS.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
912 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 18-18 H: 25-25 N: 0-100 O: 0-100 Na: 0-8
44
250616-5-29 3 (0.076)



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
285.1965	285.1967	-0.2	-0.7	7.5	52.7	n/a	n/a	C18 H25 N2 O

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1527 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
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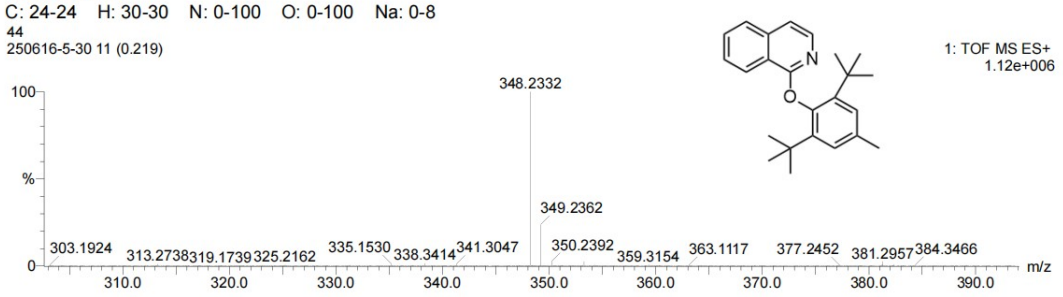
44
250616-5-30 11 (0.219)



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
348.2332	348.2327	0.5	1.4	10.5	1044.7	n/a	n/a	C24 H30 N O

Elemental Composition Report **Page 1**

Single Mass Analysis

Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
2728 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
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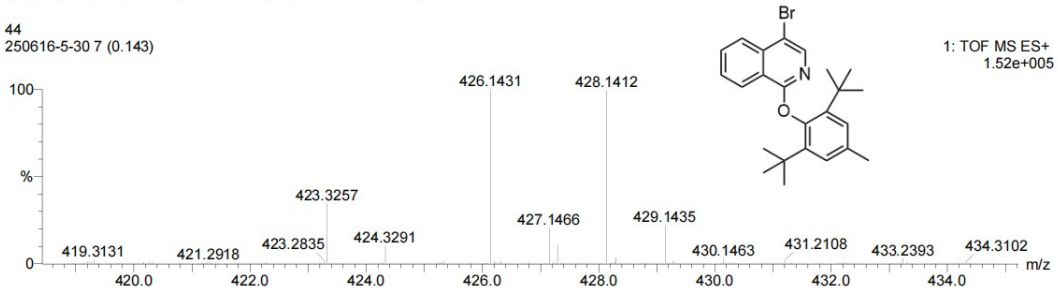
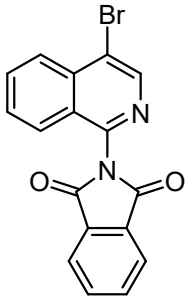


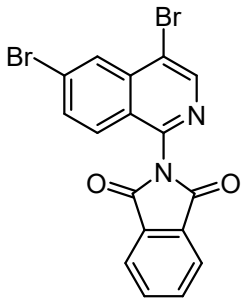
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
426.1431	426.1433	-0.2	-0.5	10.5	835.0	n/a	n/a	C24 H29 N O Br

Part 2. NMR Spectra of New Compounds and Selected Known Compounds

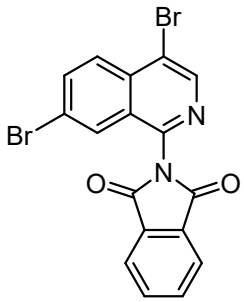
^{1}H and ^{13}C NMR data



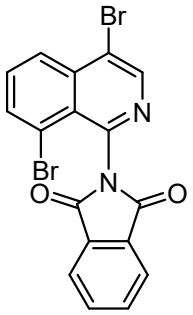
2-(4-Bromoisoquinolin-1-yl)isoindoline-1,3-dione (**3a**). Product **3a** was synthesized according to General procedure, yellow solid (316 mg, 0.90 mmol, 90% yield), mp 186–187 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.79 (s, 1H), 8.28 (d, J = 8.5 Hz, 1H), 8.03 (dd, J = 5.5, 3.1 Hz, 2H), 7.90–7.83 (m, 4H), 7.72–7.66 (m, 1H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 167.1, 144.9, 143.7, 136.8, 134.8, 132.3, 132.0, 129.3, 127.1, 126.8, 125.1, 124.2, 121.3. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{17}H_{10}BrN_2O_2$: 352.9926; found: 352.9925.



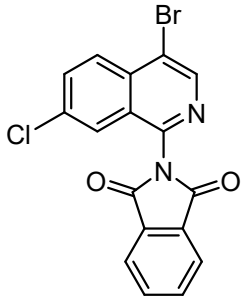
2-(4,6-Dibromoisoquinolin-1-yl)isoindoline-1,3-dione (**3b**). Product **3b** was synthesized according to General procedure, yellow solid (350 mg, 0.81 mmol, 81% yield), mp 168–169 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.80 (s, 1H), 8.45 (d, J = 1.8 Hz, 1H), 8.02 (dd, J = 5.5, 3.0 Hz, 2H), 7.86 (dd, J = 5.5, 3.0 Hz, 2H), 7.75 (dd, J = 9.1, 1.6 Hz, 1H), 7.70 (d, J = 8.9 Hz, 1H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 166.9, 145.0, 144.8, 137.8, 135.0, 134.3, 133.0, 131.9, 129.2, 127.9, 126.9, 125.6, 124.3, 123.6, 119.6. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{17}H_9Br_2N_2O_2$: 432.9010; found: 432.9016.



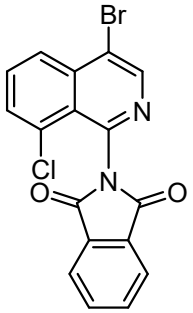
2-(4,7-Dibromoisoquinolin-1-yl)isoindoline-1,3-dione (**3c**). Product **3c** was synthesized according to General procedure, yellow solid (260 mg, 0.60 mmol, 60% yield), mp 250–251 °C. **^{1}H NMR** (400 MHz, CDCl3) δ 8.82 (s, 1H), 8.17 (d, J = 9.0 Hz, 1H), 8.06 (dd, J = 5.5, 3.1 Hz, 2H), 8.00 (d, J = 1.9 Hz, 1H), 7.96 (dd, J = 9.0, 1.9 Hz, 1H), 7.90 (dd, J = 5.6, 3.1 Hz, 2H). **^{13}C{^{1}H} NMR** (101 MHz, CDCl3) δ 166.9, 144.1, 144.0, 135.9, 135.4, 135.0, 134.3, 131.9, 128.6, 128.0, 127.3, 124.4, 123.9, 123.6, 121.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{17}H_9Br_2N_2O_2$: 432.9010; found: 432.9020.



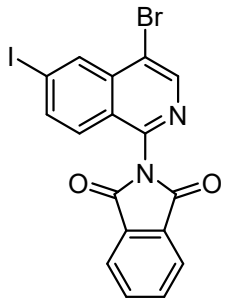
2-(4,8-Dibromoisoquinolin-1-yl)isoindoline-1,3-dione (**3d**). Product **3d** was synthesized according to General procedure, yellow solid (324 mg, 0.75 mmol, 75% yield), mp 237–238 °C. **^{1}H NMR** (400 MHz, CDCl3) δ 8.85 (s, 1H), 8.34 (d, J = 8.6 Hz, 1H), 8.05–7.99 (m, 3H), 7.83 (dd, J = 5.5, 3.1 Hz, 2H), 7.65 (t, J = 8.0 Hz, 1H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 167.0, 144.3, 142.9, 138.8, 136.4, 134.6, 132.7, 132.0, 127.5, 125.9, 124.3, 121.8, 118.5. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{17}H_9Br_2N_2O_2$: 432.9010; found: 432.9019.



2-(4-Bromo-7-chloroisoquinolin-1-yl)isoindoline-1,3-dione (**3e**). Product **3e** was synthesized according to General procedure, yellow solid (297 mg, 0.77 mmol, 77% yield), mp 220–221 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.86 (s, 1H), 8.30 (dd, J = 6.3, 3.5 Hz, 1H), 8.02 (dd, J = 5.5, 3.1 Hz, 2H), 7.84 (dd, J = 5.5, 3.1 Hz, 2H), 7.77 (q, J = 3.5 Hz, 2H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 167.0, 144.4, 138.8, 134.6, 132.6, 132.3, 131.7, 130.3, 126.8, 125.0, 124.2, 121.7. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{17}H_9BrClN_2O_2$: 386.9536; found: 386.9535.

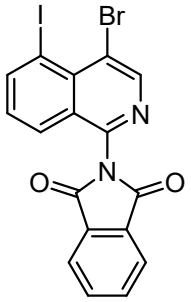


2-(4-Bromo-8-chloroisoquinolin-1-yl)isoindoline-1,3-dione (**3f**). Product **3f** was synthesized according to General procedure, yellow solid (278 mg, 0.72 mmol, 72% yield), mp 156–157 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.85 (s, 1H), 8.30 (dt, J = 7.6, 3.8 Hz, 1H), 8.01 (dd, J = 5.5, 3.1 Hz, 2H), 7.84 (dd, J = 5.5, 3.1 Hz, 2H), 7.77 – 7.74 (m, 2H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 167.0, 144.4, 142.3, 138.8, 134.6, 133.0, 132.3, 131.7, 130.3, 126.8, 125.0, 124.2, 121.7. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{17}H_9BrClN_2O_2$: 386.9536; found: 386.9538.

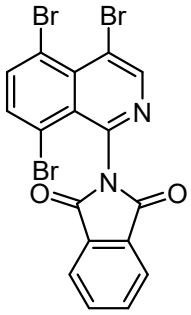


2-(4-Bromo-6-iodoisoquinolin-1-yl)isoindoline-1,3-dione (**3g**). Product **3g** was synthesized according to General procedure, yellow solid (272 mg, 0.57 mmol, 57% yield), mp 216–217 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.80 (s, 1H), 8.68 (d, J = 1.6 Hz, 1H), 8.03 (dd, J = 5.5, 3.0 Hz, 2H), 7.94 (dd, J = 8.8, 1.6 Hz, 1H), 7.90–7.86 (m, 2H), 7.53 (d, J = 8.8 Hz, 1H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 166.9, 144.6, 138.2, 137.8, 135.7, 135.0, 131.9, 126.3, 125.9, 124.3, 119.3, 100.4. HRMS (ESI) m/z $[M+H]^+$

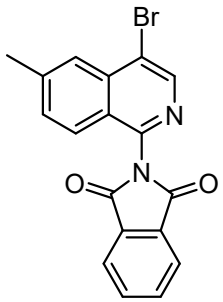
calcd for $C_{17}H_9BrIN_2O_2$: 478.8892; found: 478.8896.



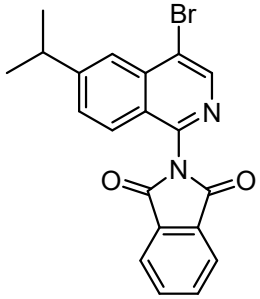
2-(4-Bromo-5-iodoisoquinolin-1-yl)isoindoline-1,3-dione (**3h**). Product **3h** was synthesized according to General procedure, yellow solid (210 mg, 0.44 mmol, 44% yield), mp 225–226 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.90 (s, 1H), 8.20 (dd, J = 7.5, 1.2 Hz, 1H), 8.06–7.99 (m, 2H), 7.91–7.85 (m, 3H), 7.44 (dd, J = 8.5, 7.5 Hz, 1H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 166.9, 148.1, 139.8, 135.0, 133.7, 131.9, 129.3, 129.0, 125.8, 124.3, 119.9, 118.4. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{17}H_9BrIN_2O_2$: 478.8892; found: 478.8895.



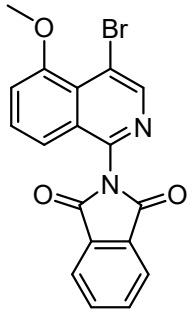
2-(4,5,8-Tribromoisoquinolin-1-yl)isoindoline-1,3-dione (**3i**). Product **3i** was synthesized according to General procedure, yellow solid (153 mg, 0.30 mmol, 30% yield), mp 203–204 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.96 (s, 1H), 8.04 (dd, J = 5.5, 3.1 Hz, 2H), 8.00 (d, J = 8.1 Hz, 1H), 7.87 (dd, J = 5.5, 3.1 Hz, 2H), 7.77 (d, J = 8.2 Hz, 1H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 166.5, 148.5, 143.6, 139.2, 135.9, 135.7, 134.8, 132.4, 127.7, 124.4, 120.0, 118.5, 118.3. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{17}H_8Br_3N_2O_2$: 510.8115; found: 510.8125.



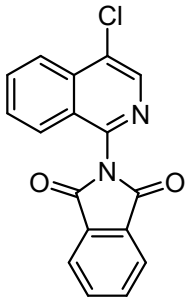
2-(4-Bromo-6-methylisoquinolin-1-yl)isoindoline-1,3-dione (**3j**). Product **3j** was synthesized according to General procedure, yellow solid (132 mg, 0.36 mmol, 36% yield), mp 217–218 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.74 (s, 1H), 8.05–8.00 (m, 3H), 7.88–7.84 (m, 2H), 7.71 (d, J = 8.6 Hz, 1H), 7.50 (dd, J = 8.7, 1.6 Hz, 1H), 2.62 (s, 3H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 167.1, 144.6, 143.8, 143.4, 137.0, 134.8, 132.0, 131.6, 125.7, 125.5, 125.0, 124.2, 120.6, 22.3. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{18}H_{12}BrN_2O_2$: 367.0082; found: 367.0086.



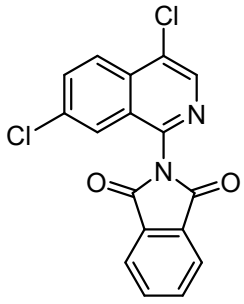
2-(4-Bromo-6-isopropylisoquinolin-1-yl)isoindoline-1,3-dione (**3k**). Product **3k** was synthesized according to General procedure, yellow solid (173 mg, 0.44 mmol, 44% yield), mp 119–120 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.75 (s, 1H), 8.08–8.00 (m, 3H), 7.90–7.83 (m, 2H), 7.75 (d, J = 8.7 Hz, 1H), 7.58 (dd, J = 8.7, 1.7 Hz, 1H), 3.19 (h, J = 6.9 Hz, 1H), 1.37 (d, J = 6.9 Hz, 6H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 167.1, 153.9, 144.5, 143.7, 137.1, 134.8, 132.0, 129.3, 125.8, 125.1, 124.2, 123.2, 121.1, 34.8, 23.6. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{20}H_{16}BrN_2O_2$: 395.0395; found: 395.0399.



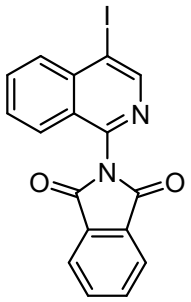
2-(4-Bromo-5-methoxyisoquinolin-1-yl)isoindoline-1,3-dione (**3l**). Product **3l** was synthesized according to General procedure, brown solid (96 mg, 0.25 mmol, 25% yield), mp 215–216 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.74 (s, 1H), 8.01 (dd, J = 5.5, 3.1 Hz, 2H), 7.85 (dd, J = 5.5, 3.1 Hz, 2H), 7.54 (t, J = 8.2 Hz, 1H), 7.40 (d, J = 8.5 Hz, 1H), 7.16 (d, J = 7.8 Hz, 1H), 4.01 (s, 3H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 167.0, 155.6, 146.0, 134.8, 132.0, 129.4, 128.9, 128.6, 124.2, 116.9, 115.8, 111.0, 55.9. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{18}H_{12}BrN_2O_3$: 383.0031; found: 383.0031.



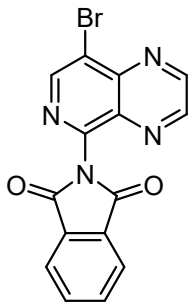
2-(4-Chloroisoquinolin-1-yl)isoindoline-1,3-dione (**3m**). Product **3m** was synthesized according to General procedure, yellow solid (262 mg, 0.85 mmol, 85% yield), mp 198–199 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.65 (s, 1H), 8.32 (d, J = 8.5 Hz, 1H), 8.03 (dd, J = 5.5, 3.0 Hz, 2H), 7.91–7.84 (m, 4H), 7.72–7.67 (m, 1H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 167.1, 140.9, 135.5, 134.9, 132.1, 132.0, 130.0, 129.3, 126.7, 125.0, 124.2, 124.2. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{17}H_{10}ClN_2O_2$: 309.0431; found: 309.0435.



2-(4,7-Dichloroisoquinolin-1-yl)isoindoline-1,3-dione (**3n**). Product **3n** was synthesized according to General procedure, yellow solid (171 mg, 0.50 mmol, 50% yield), mp 252–253 ℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.65 (d, J = 0.8 Hz, 1H), 8.30–8.24 (m, 1H), 8.07–8.01 (m, 2H), 7.91–7.86 (m, 2H), 7.85–7.79 (m, 2H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 167.0, 141.2, 135.6, 135.0, 133.9, 133.2, 131.9, 129.9, 127.5, 126.1, 124.4, 124.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{17}H_9Cl_2N_2O_2$: 343.0041; found: 343.0043.

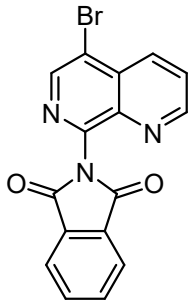


2-(4-Iodoisoquinolin-1-yl)isoindoline-1,3-dione (**3o**). Product **3o** was synthesized according to General procedure, yellow solid (260 mg, 0.65 mmol, 65% yield), mp 195–196℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.02 (s, 1H), 8.14 (d, J = 8.5 Hz, 1H), 8.02 (dt, J = 7.0, 3.5 Hz, 2H), 7.89–7.84 (m, 3H), 7.80–7.76 (m, 1H), 7.71–7.65 (m, 1H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 167.1, 149.88, 146.0, 139.2, 134.9, 132.7, 131.98, 131.6, 129.5, 127.1, 125.3, 124.3, 98.6. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{17}H_{10}IN_2O_2$: 400.9787; found: 400.9791.

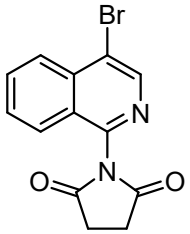


2-(8-Bromopyrido[3,4-b]pyrazin-5-yl)isoindoline-1,3-dione (**3p**). Product **3q** was synthesized according to General procedure, yellow solid (177 mg, 0.50 mmol, 50% yield), mp 204–205℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.18 (d, J = 1.7 Hz, 1H), 9.14 (s, 1H), 8.96 (d, J = 1.7 Hz, 1H), 8.02 (dd, J = 5.5, 3.0 Hz, 2H), 7.85 (dd, J = 5.5, 3.1 Hz, 2H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 166.6, 150.1, 147.8, 147.4, 146.7, 145.1, 135.9, 134.8, 132.0, 124.3, 121.8. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{15}H_8BrN_4O_2$:

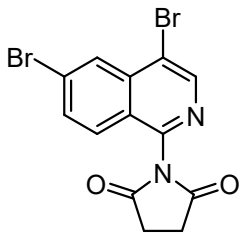
354.9831; found: 354.9836.



2-(5-Bromo-1,7-naphthyridin-8-yl)isoindoline-1,3-dione (**3q**). Product **3r** was synthesized according to General procedure, yellow solid (176 mg, 0.50 mmol, 50% yield), mp 172–173℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.00 (dd, J = 4.1, 1.6 Hz, 1H), 8.89 (s, 1H), 8.55 (dd, J = 8.6, 1.6 Hz, 1H), 8.02 (dd, J = 5.5, 3.0 Hz, 2H), 7.84 (dd, J = 5.5, 3.1 Hz, 2H), 7.76 (dd, J = 8.6, 4.1 Hz, 1H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 167.0, 153.1, 146.3, 144.2, 134.7, 134.6, 133.0, 132.3, 126.8, 124.2, 120.0. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{16}H_9BrN_3O_2$: 353.9878; found: 353.9879.

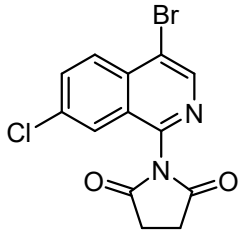


1-(4-Bromoisoquinolin-1-yl)pyrrolidine-2,5-dione (**5a**). Product **5a** was synthesized according to General procedure, yellow solid (213 mg, 0.70 mmol, 70% yield), mp 159–160℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.75 (s, 1H), 8.25 (d, J = 8.5 Hz, 1H), 7.91–7.83 (m, 1H), 7.73–7.67 (m, 2H), 3.12–2.99 (m, 4H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 175.9, 145.1, 143.7, 136.8, 132.4, 129.5, 126.9, 126.2, 124.5, 121.5, 29.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{13}H_{10}BrN_2O_2$: 304.9926; found: 304.9927.

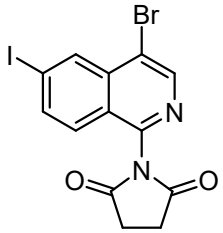


1-(4,6-Dibromoisoquinolin-1-yl)pyrrolidine-2,5-dione (**5b**). Product **5b** was synthesized according to

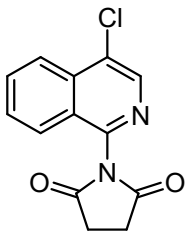
General procedure, yellow solid (184 mg, 0.48 mmol, 48% yield), mp 209–210°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.77 (s, 1H), 8.44 (d, J = 1.9 Hz, 1H), 7.77 (dd, J = 8.9, 1.9 Hz, 1H), 7.56 (d, J = 8.9 Hz, 1H), 3.07 (d, J = 3.3 Hz, 4H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 175.7, 144.8, 137.8, 133.2, 129.3, 128.0, 126.2, 124.8, 120.0, 29.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{13}H_9Br_2N_2O_2$: 384.9010; found:384.9026.



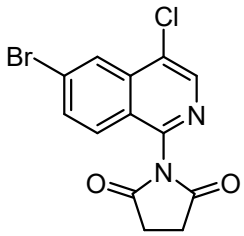
1-(4-Bromo-7-chloroisoquinolin-1-yl)pyrrolidine-2,5-dione (**5c**). Product **5c** was synthesized according to General procedure, yellow solid (169 mg, 0.50 mmol, 50% yield), mp 181–182°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.75 (s, 1H), 8.20 (d, J = 9.0 Hz, 1H), 7.79 (dd, J = 9.0, 2.0 Hz, 1H), 7.66 (d, J = 2.0 Hz, 1H), 3.15–3.03 (m, 4H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 175.8, 144.2, 144.1, 135.8, 135.2, 133.5, 128.8, 126.9, 123.4, 121.2, 29.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{13}H_9BrClN_2O_2$: 338.9536; found: 338.9542.



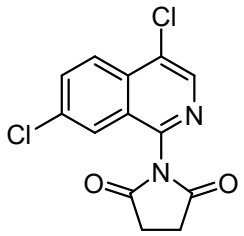
1-(4-Bromo-6-iodoisoquinolin-1-yl)pyrrolidine-2,5-dione (**5d**). Product **5d** was synthesized according to General procedure, yellow solid (245 mg, 0.57 mmol, 57% yield), mp 222–223°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.76 (s, 1H), 8.66 (d, J = 1.6 Hz, 1H), 7.95 (dd, J = 8.8, 1.6 Hz, 1H), 7.39 (d, J = 8.8 Hz, 1H), 3.11–3.03 (m, 4H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 175.7, 145.3, 144.6, 138.4, 137.7, 135.9, 125.6, 125.1, 119.6, 100.6, 29.6, 29.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{13}H_9BrIN_2O_2$: 430.8892; found: 430.8894.



1-(4-Chloroisoquinolin-1-yl)pyrrolidine-2,5-dione (**5e**). Product **5e** was synthesized according to General procedure, yellow solid (148 mg, 0.57 mmol, 57% yield), mp 123–124°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.61 (s, 1H), 8.30 (d, J = 8.5 Hz, 1H), 7.92–7.85 (m, 1H), 7.71 (dd, J = 4.3, 1.5 Hz, 2H), 3.12–3.02 (m, 4H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 175.9, 144.3, 140.8, 135.5, 132.2, 130.3, 129.4, 125.9, 124.4, 124.4, 29.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{13}H_{10}ClN_2O_2$: 261.0431; found: 261.0436.

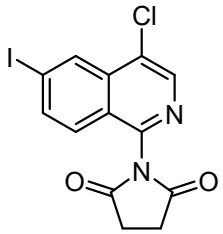


1-(6-Bromo-4-chloroisoquinolin-1-yl)pyrrolidine-2,5-dione (**5f**). Product **5f** was synthesized according to General procedure, yellow solid (240 mg, 0.71 mmol, 71% yield), mp 169–170°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.61 (s, 1H), 8.44 (d, J = 1.9 Hz, 1H), 7.72 (dd, J = 8.9, 1.9 Hz, 1H), 7.56 (d, J = 8.9 Hz, 1H), 3.04 (q, J = 1.2 Hz, 4H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 175.9, 144.5, 141.9, 136.4, 133.0, 129.0, 127.7, 126.7, 126.2, 124.4, 29.7, 29.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{13}H_9BrClN_2O_2$: 338.9536; found: 338.9538.

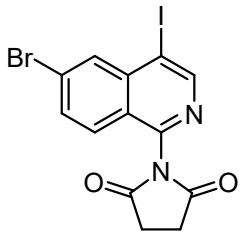


1-(4,7-Dichloroisoquinolin-1-yl)pyrrolidine-2,5-dione (**5g**). Product **5g** was synthesized according to General procedure, yellow solid (229 mg, 0.78 mmol, 78% yield), mp 197–198°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.61 (s, 1H), 8.24 (d, J = 9.0 Hz, 1H), 7.80 (dd, J = 9.0, 2.0 Hz, 1H), 7.67 (d, J = 2.0 Hz, 1H), 3.16–3.03 (m, 4H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 175.8, 143.5, 141.2, 135.8, 133.9, 133.2, 130.2, 126.6, 126.2, 123.4, 29.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{13}H_9Cl_2N_2O_2$: 295.0041; found:

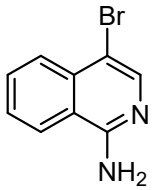
295.0045.



1-(4-Chloro-6-iodoisoquinolin-1-yl)pyrrolidine-2,5-dione (**5h**). Product **5h** was synthesized according to General procedure, yellow solid (247 mg, 0.64 mmol, 64% yield), mp 255–256°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.70 (d, J = 1.6 Hz, 1H), 8.62 (s, 1H), 7.97 (dd, J = 8.8, 1.7 Hz, 1H), 7.40 (dd, J = 8.8, 0.5 Hz, 1H), 3.10–3.05 (m, 4H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 175.7, 141.8, 138.3, 136.4, 133.4, 128.6, 125.6, 124.7, 100.2, 29.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{13}H_9ClIN_2O_2$: 386.9397; found: 386.9395.

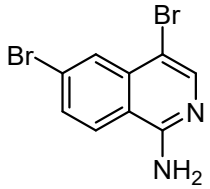


1-(6-Bromo-4-iodoisoquinolin-1-yl)pyrrolidine-2,5-dione (**5i**). Product **5i** was synthesized according to General procedure, brown solid (215 mg, 0.50 mmol, 50% yield), mp 206–207°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.98 (s, 1H), 8.31 (d, J = 1.8 Hz, 1H), 7.75 (dd, J = 8.9, 1.9 Hz, 1H), 7.51 (d, J = 8.9 Hz, 1H), 3.07 (d, J = 1.4 Hz, 4H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 177.2, 175.8, 150.9, 146.3, 140.3, 134.0, 133.3, 128.4, 126.4, 124.7, 96.9, 29.6, 29.1. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_{13}H_9BrIN_2O_2$: 430.8892; found: 430.8896.



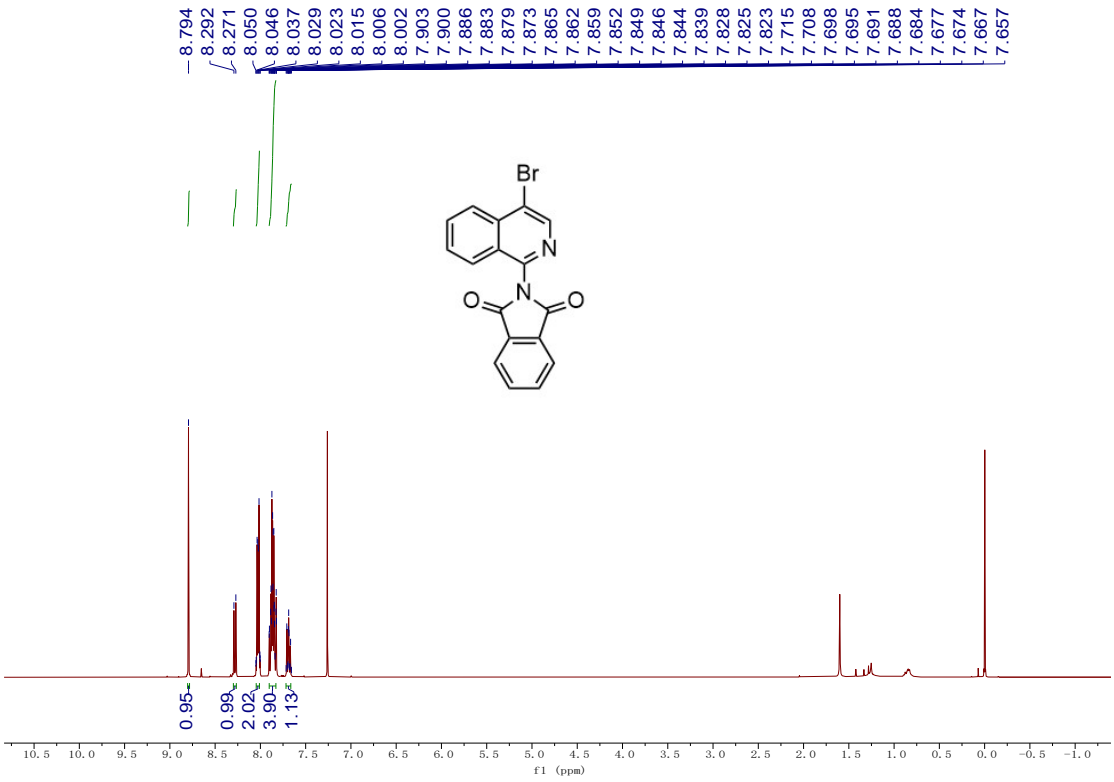
4-Bromoisoquinolin-1-amine (**6**). Product **6** was synthesized according to General procedure, yellow solid (164 mg, 0.74 mmol, 74% yield), mp 125–126°C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.12 (s, 1H), 8.08

(d, J = 8.4 Hz, 1H), 7.80–7.72 (m, 2H), 7.60–7.55 (m, 1H), 5.17 (s, 2H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 155.6, 142.6, 135.6, 131.3, 127.1, 126.7, 122.9, 119.0, 108.5. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_9H_8BrN_2$: 222.9871; found: 222.9875.

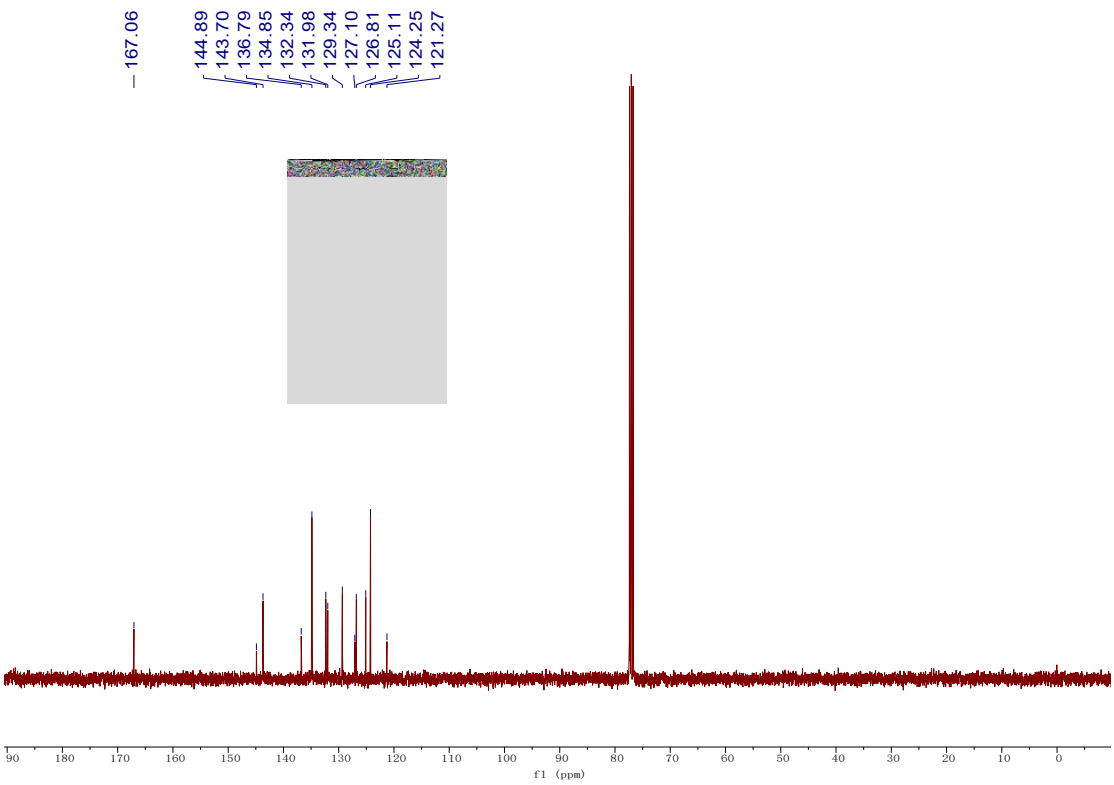


4,6-dibromoisoquinolin-1-amine (**7**). Product **7** was synthesized according to General procedure, yellow solid (227 mg, 0.75 mmol, 75% yield), mp 200–201℃. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.23 (s, 1H), 8.12 (s, 1H), 7.63 (d, J = 1.4 Hz, 2H), 5.21 (s, 2H). **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ 155.6, 143.9, 136.9, 130.4, 129.1, 126.4, 124.7, 117.4, 106.9. HRMS (ESI) m/z $[M+H]^+$ calcd for $C_9H_7Br_2N_2$: 302.8955; found: 302.8962.

^{1}H NMR spectra of **3a** (400 MHz, $CDCl_3$)



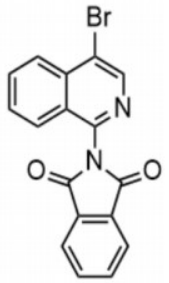
^{13}C NMR spectra of **3a** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

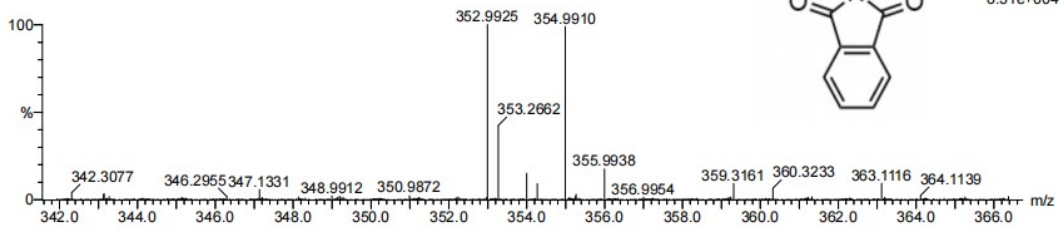
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1278 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 17-17 H: 10-10 N: 0-100 O: 0-100 Na: 0-8 Br: 1-4



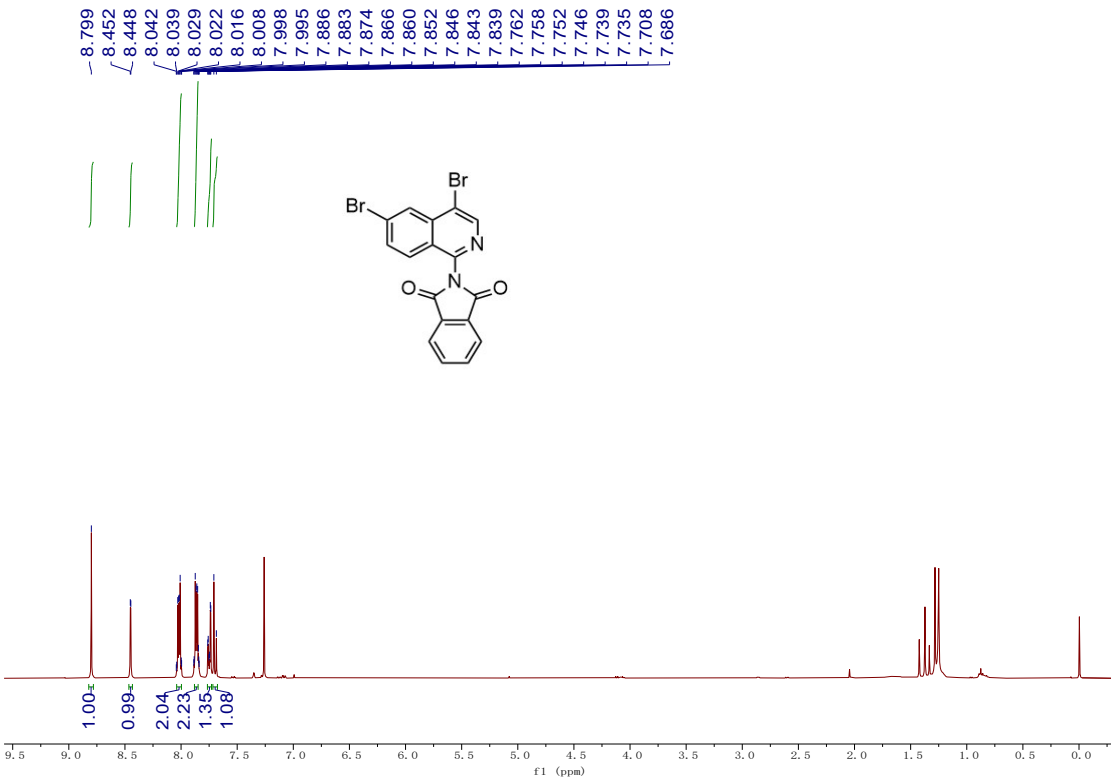
44
250616-5-1 10 (0.202)

1: TOF MS ES+
6.31e+004

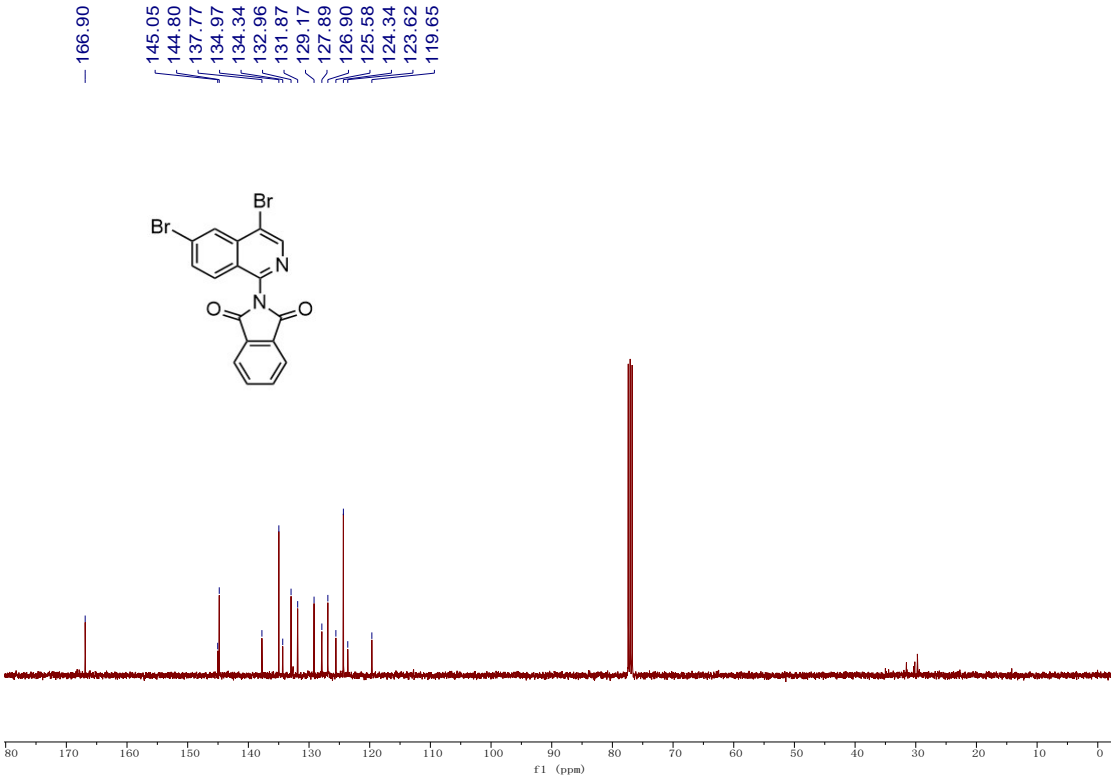


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
352.9925	352.9926	-0.1	-0.3	13.5	828.7	n/a	n/a	C17 H10 N2 O2 Br

^{1}H NMR spectra of **3b** (400 MHz, $CDCl_3$)



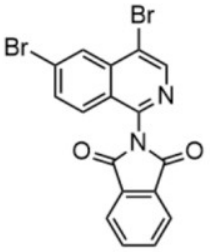
^{13}C NMR spectra of **3b** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

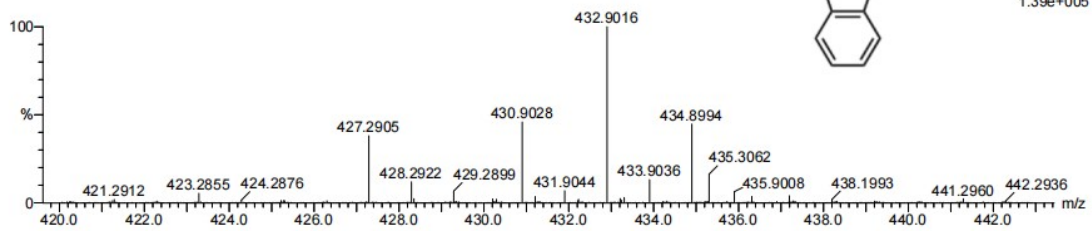
Multiple Mass Analysis: 2 mass(es) processed
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Odd and Even Electron Ions
24 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 17-17 H: 0-100 N: 2-2 O: 2-2 Na: 0-1 Se: 0-1 Br: 1-3



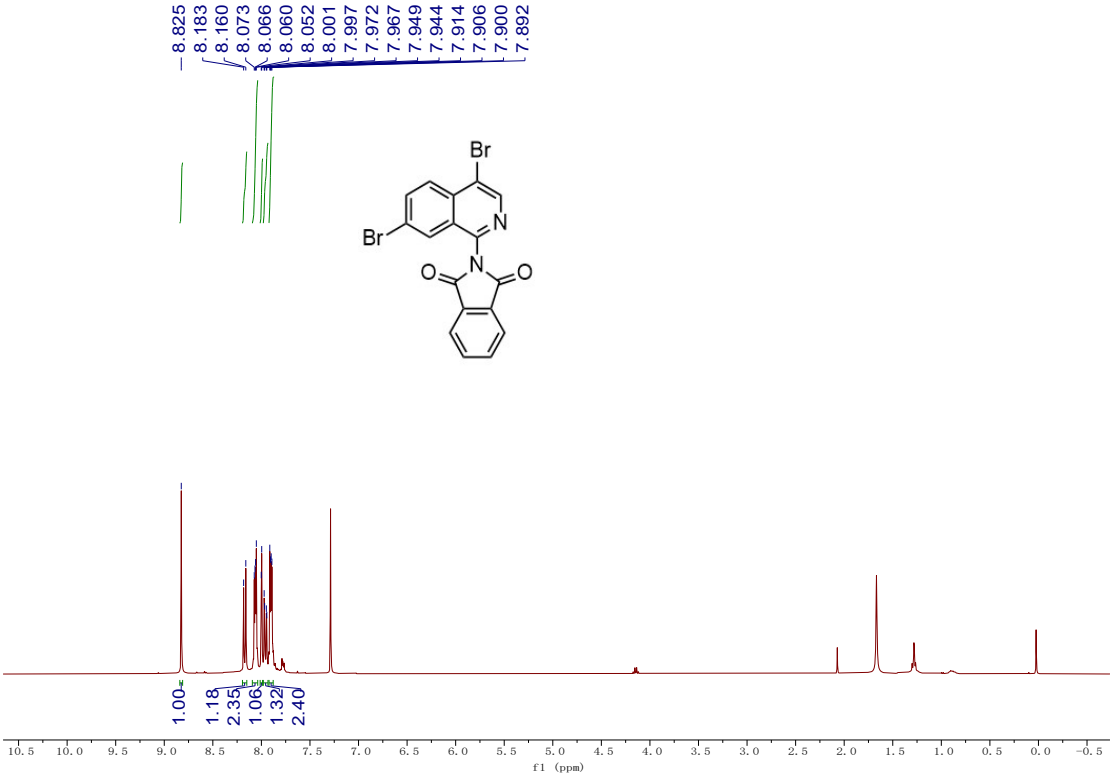
44
250616-5-2 7 (0.143)

1: TOF MS ES+
1.39e+005

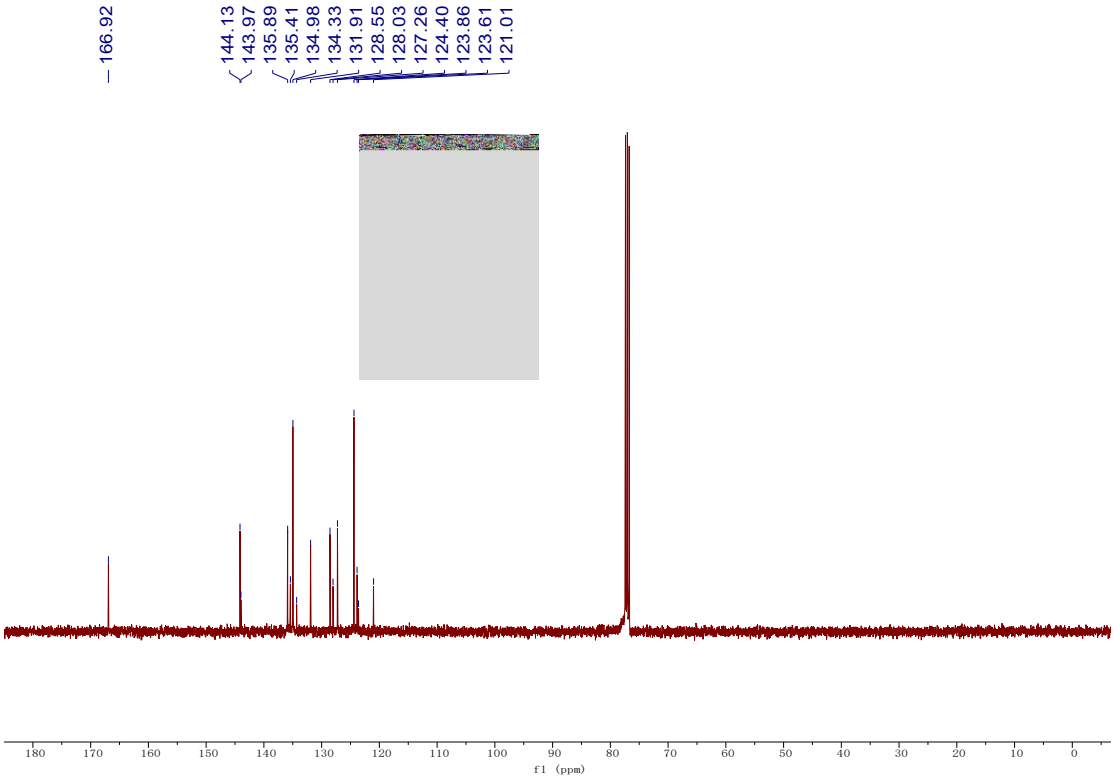


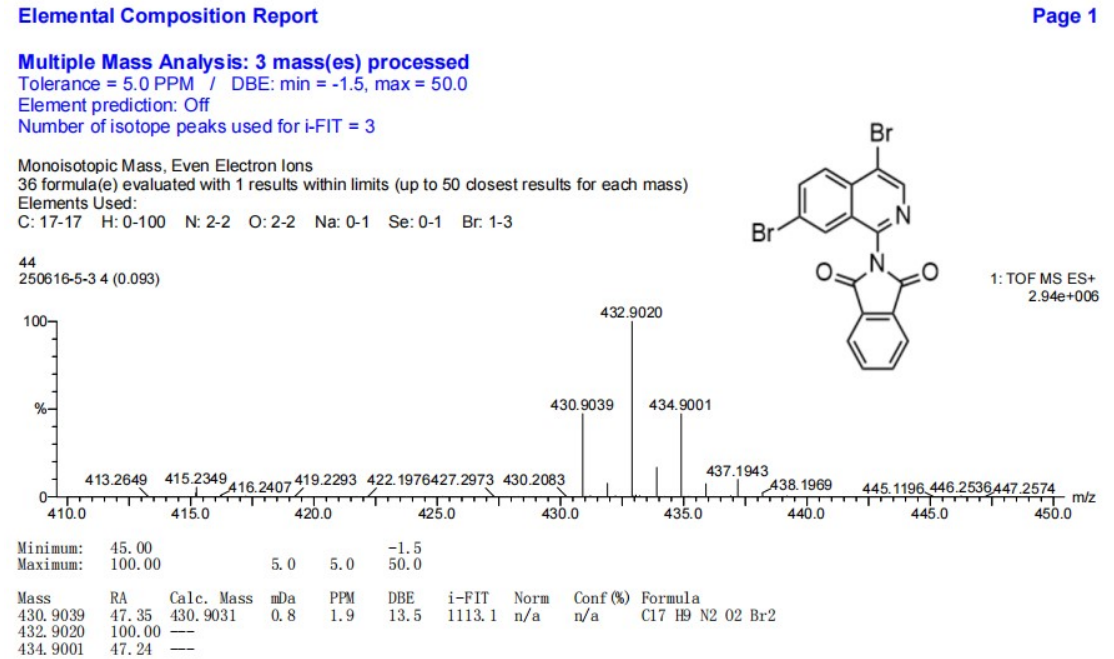
Minimum:	45.00				-1.5				
Maximum:	100.00		5.0	5.0	50.0				
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
430.9028	45.88	430.9031	-0.3	-0.7	13.5	835.5	n/a	n/a	C17 H9 N2 O2 Br2
432.9016	100.00	---							

^{1}H NMR spectra of **3c** (400 MHz, $CDCl_3$)

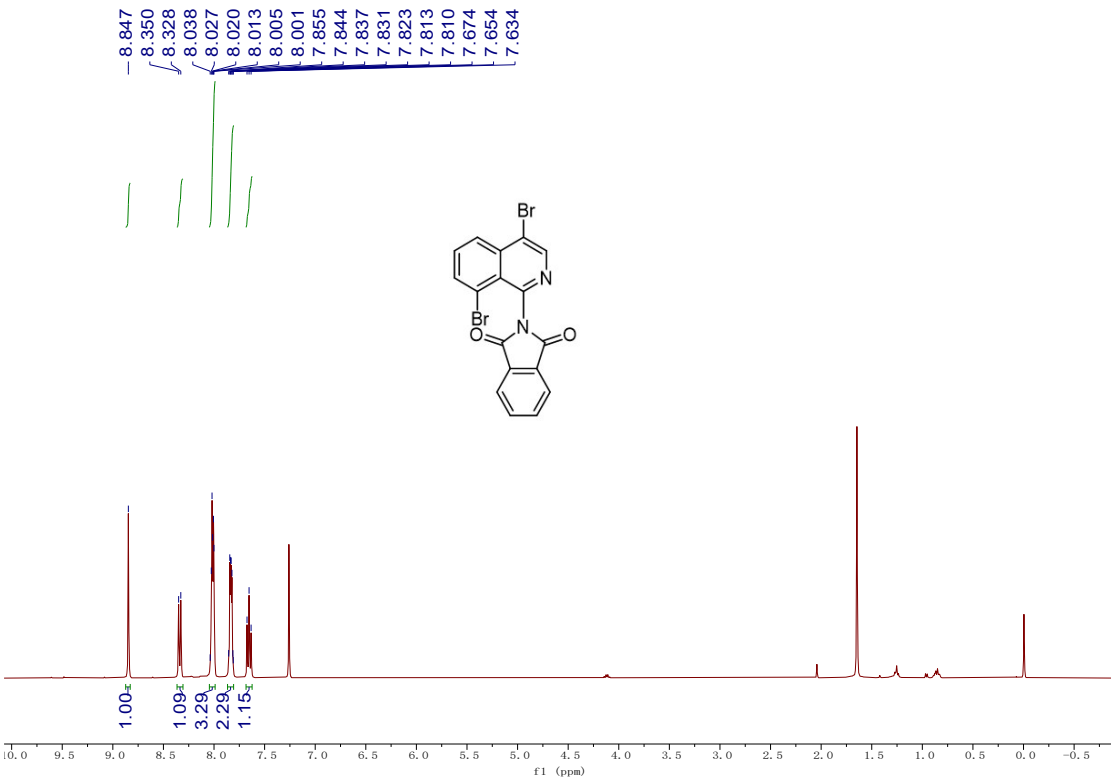


^{13}C NMR spectra of **3c** (101 MHz, $CDCl_3$)

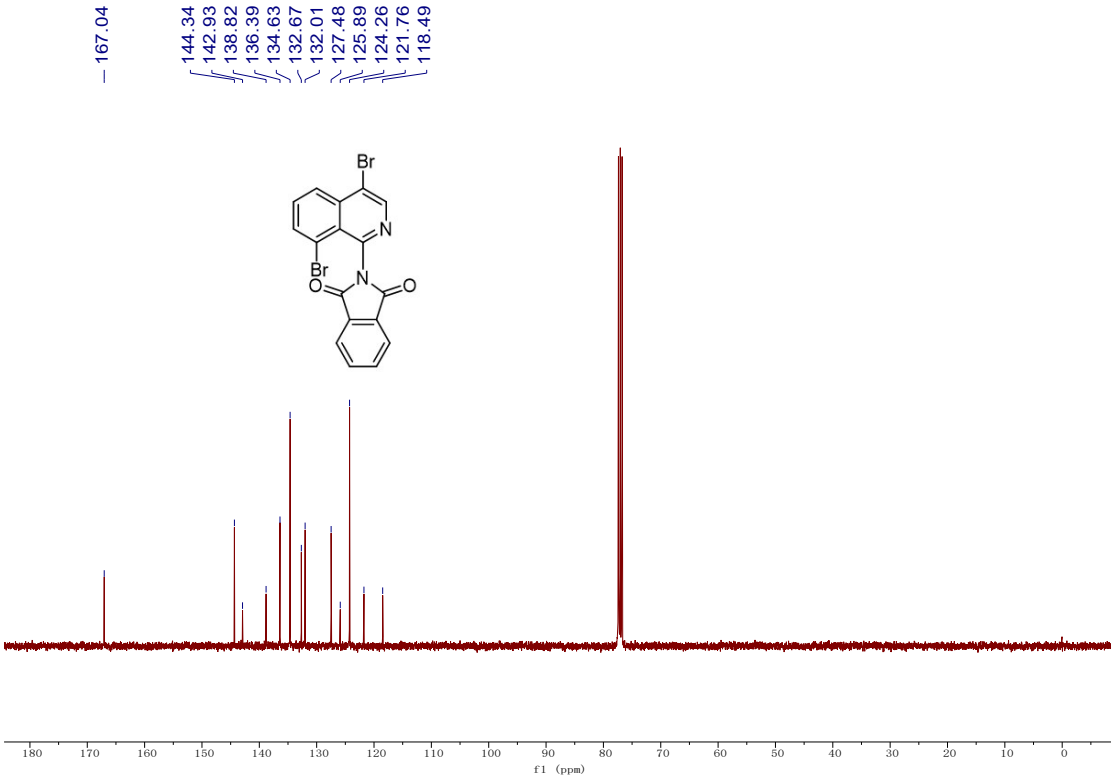




^{1}H NMR spectra of **3d** (400 MHz, $CDCl_3$)



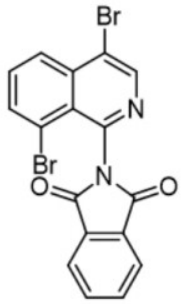
^{13}C NMR spectra of **3d** (101 MHz, $CDCl_3$)



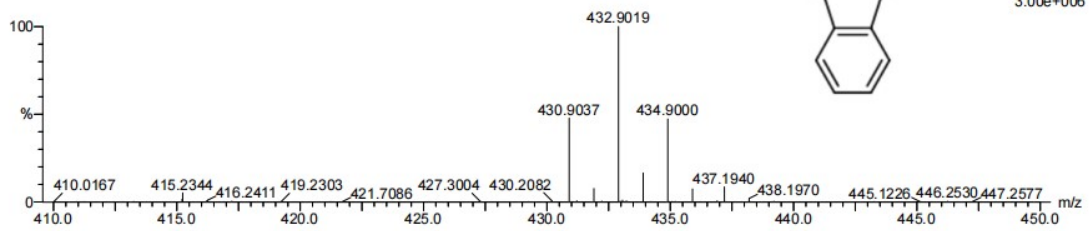
Elemental Composition Report **Page 1**

Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
36 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 17-17 H: 0-100 N: 2-2 O: 2-2 Na: 0-1 Se: 0-1 Br: 1-3

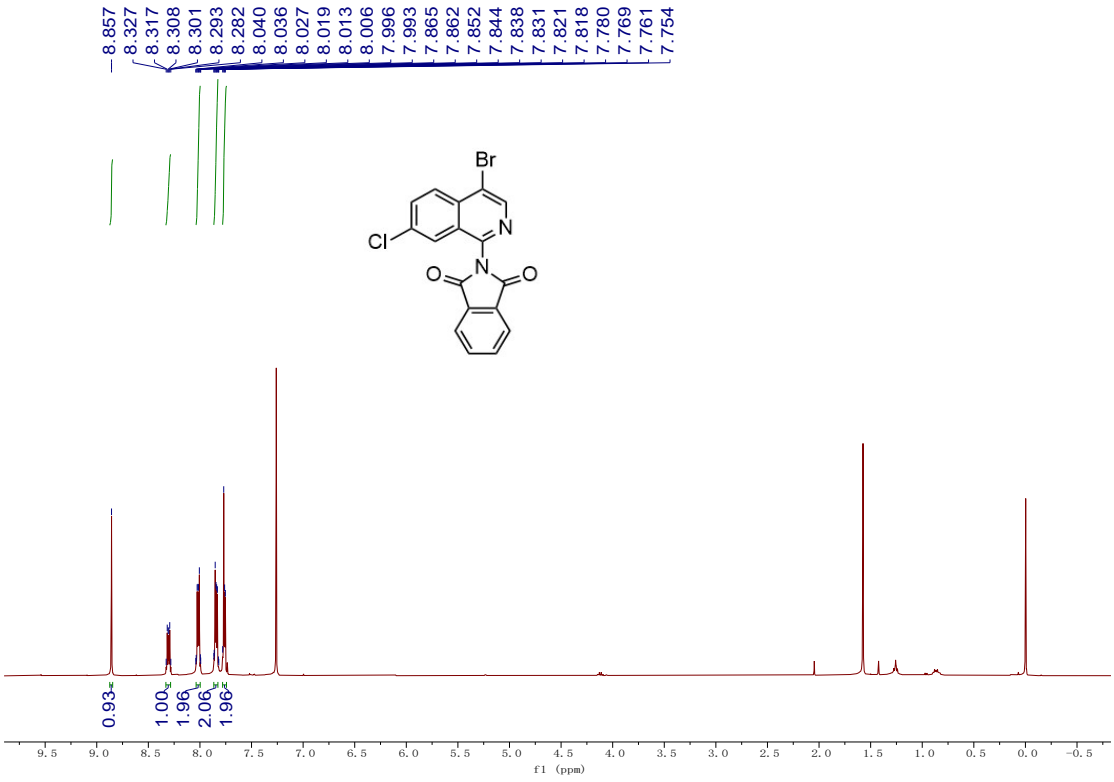


44
250616-5-4 4 (0.093)

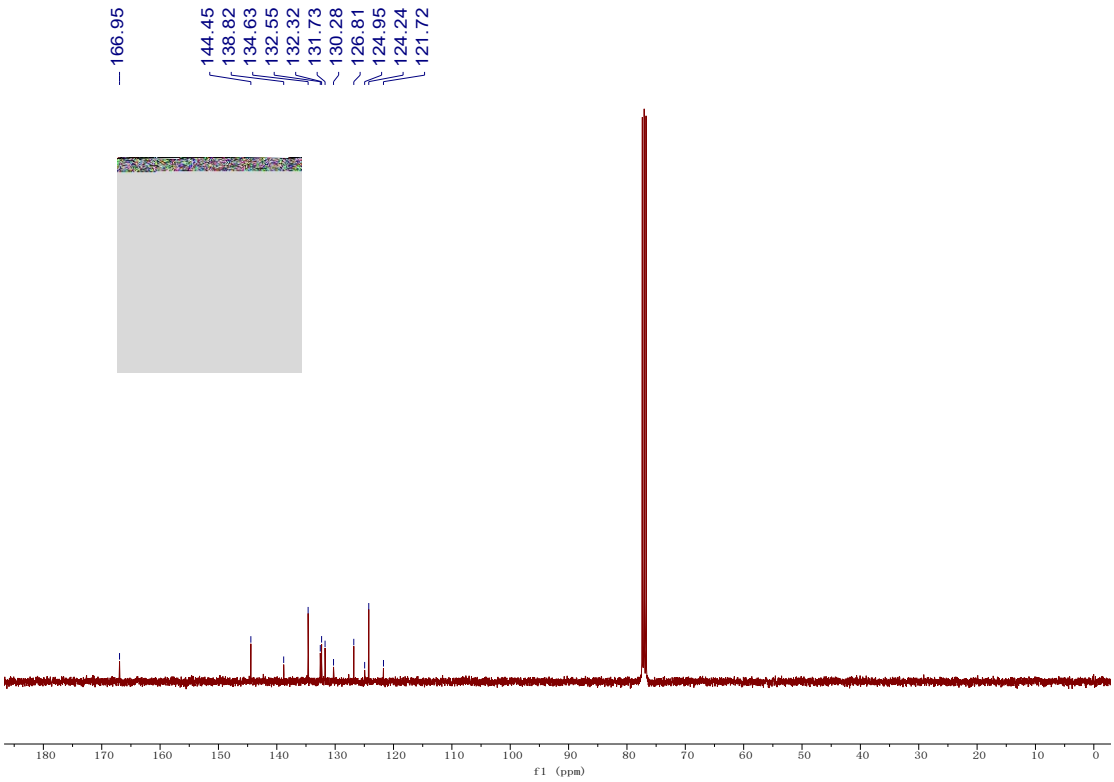


Minimum:	45.00				-1.5				
Maximum:	100.00		5.0	5.0	50.0				
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
430.9037	47.76	430.9031	0.6	1.4	13.5	1153.8	n/a	n/a	C17 H9 N2 O2 Br2
432.9019	100.00	---							
434.9000	47.26	---							

^{1}H NMR spectra of **3e** (400 MHz, $CDCl_3$)



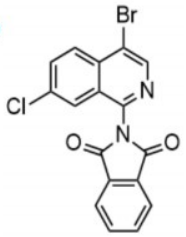
^{13}C NMR spectra of **3e** (101 MHz, $CDCl_3$)



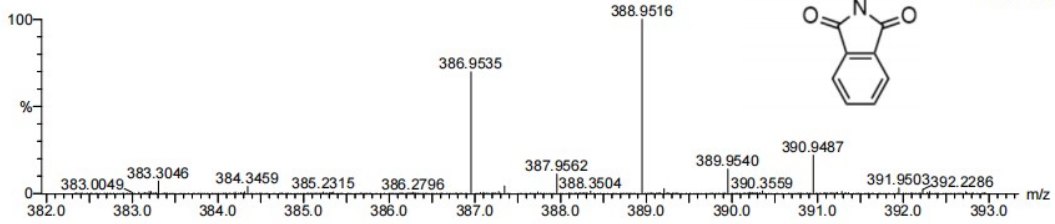
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
889 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 17-17 H: 9-9 N: 0-100 O: 0-100 Cl: 1-6 Br: 1-4
44
250616-5-10 8 (0.160)

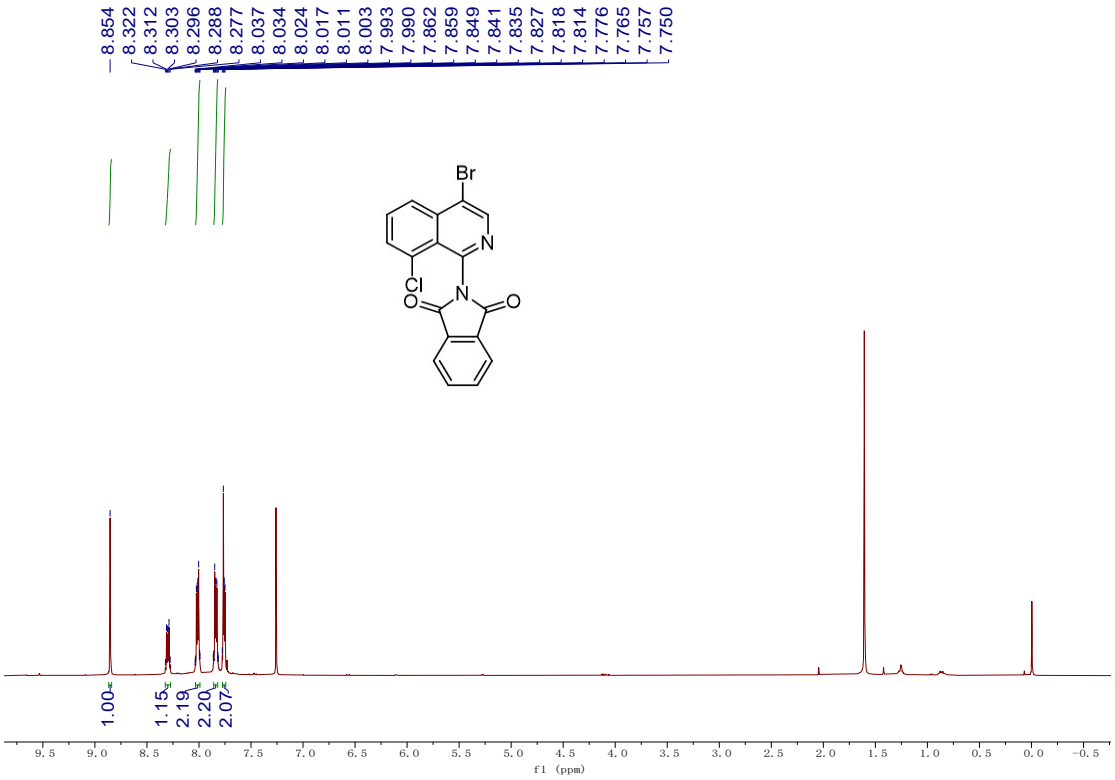


1: TOF MS ES+
1.03e+005

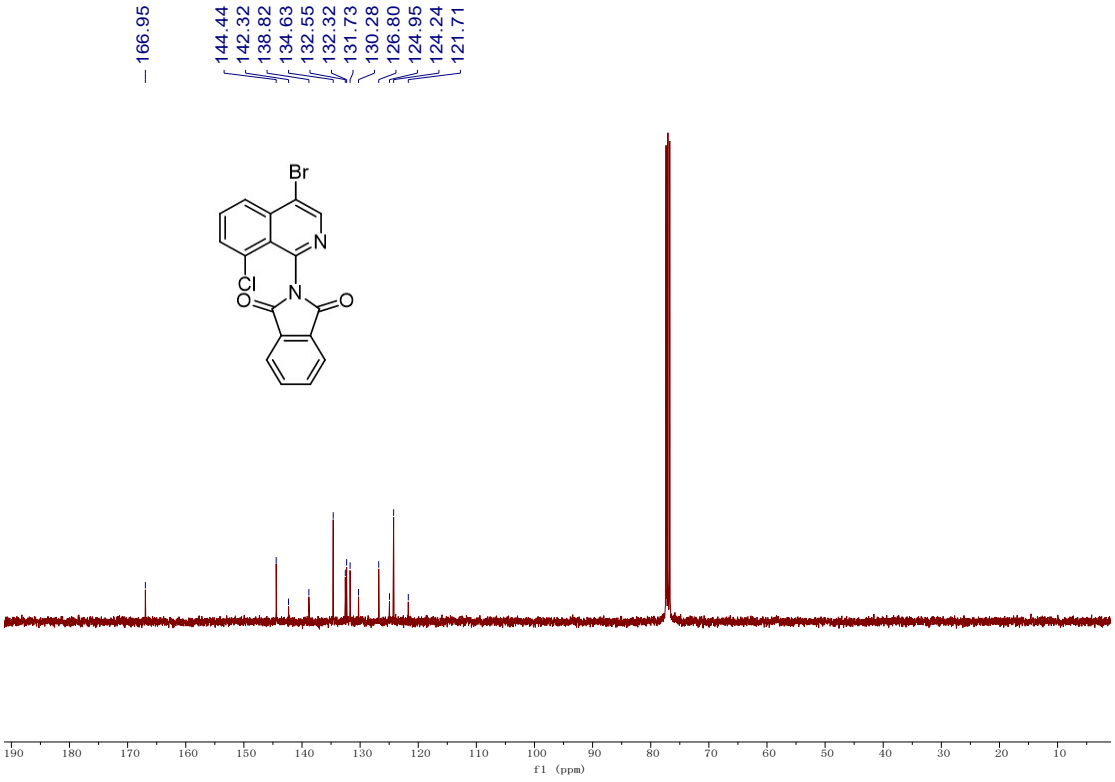


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
386.9535	386.9536	-0.1	-0.3	13.5	831.7	n/a	n/a	C17 H9 N2 O2 Cl Br

^{1}H NMR spectra of **3f** (400 MHz, $CDCl_3$)



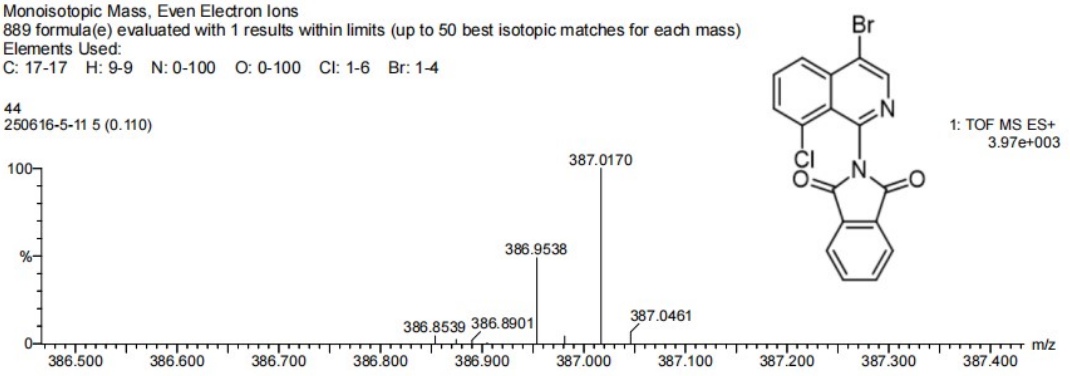
^{13}C NMR spectra of **3f** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

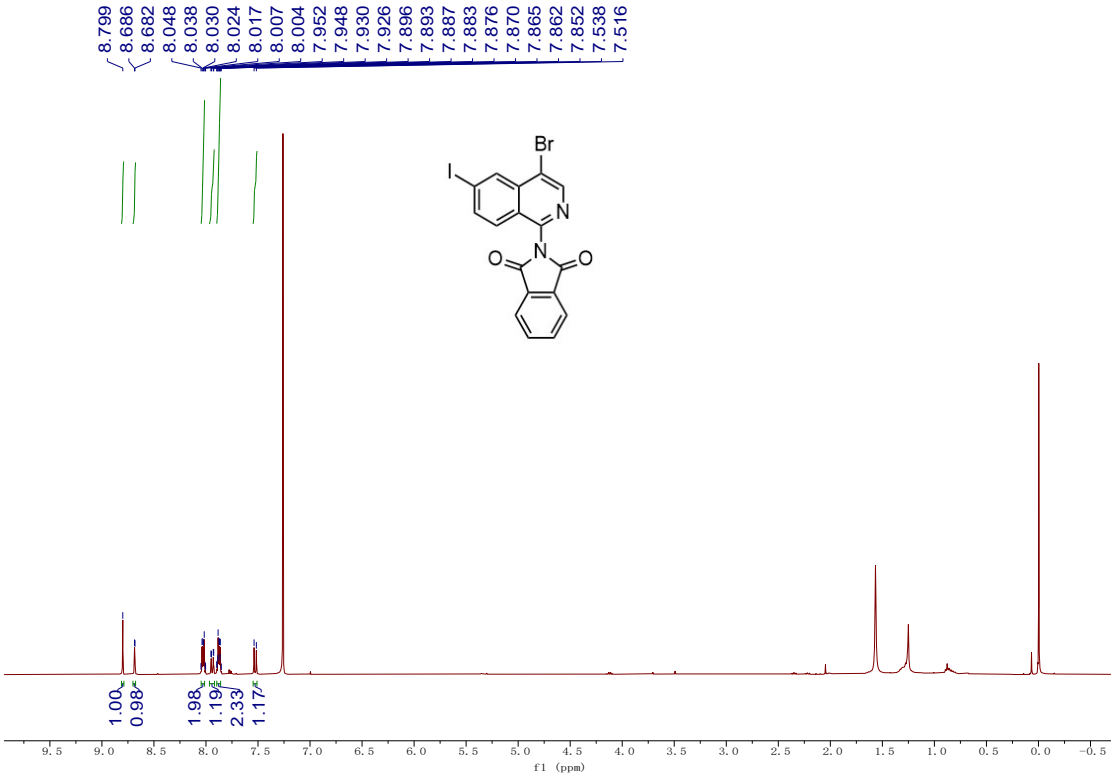
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
889 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 17-17 H: 9-9 N: 0-100 O: 0-100 Cl: 1-6 Br: 1-4

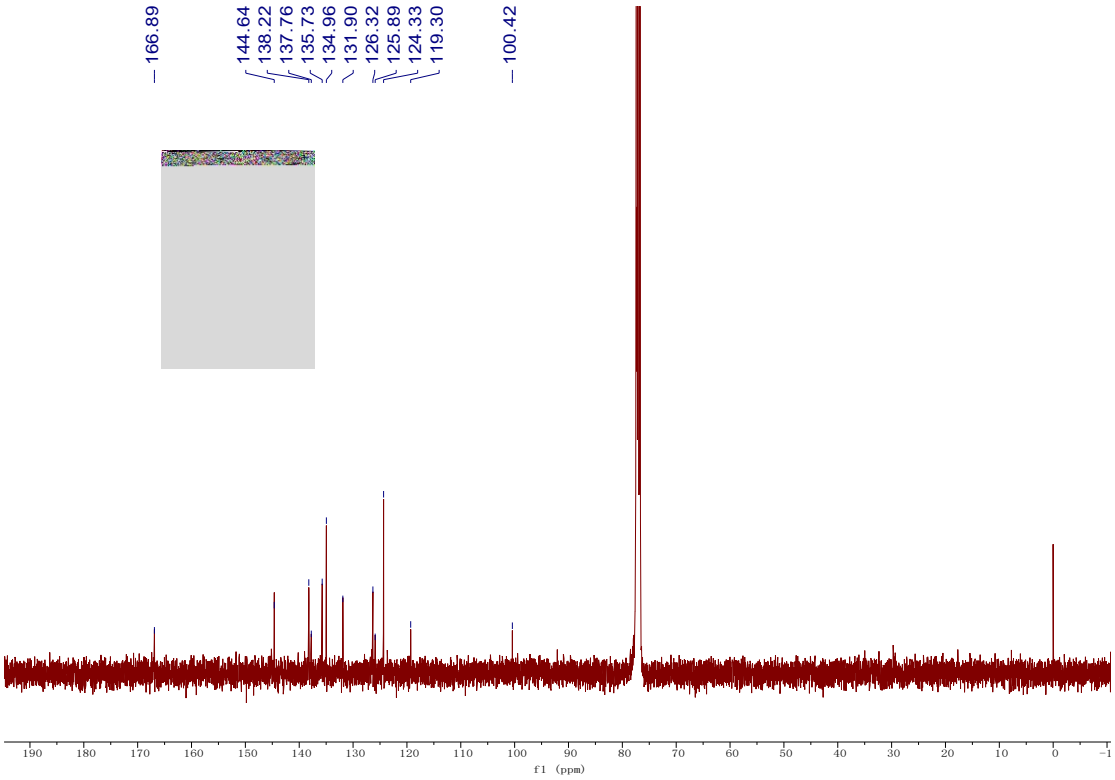


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
386.9538	386.9536	0.2	0.5	13.5	64.5	n/a	n/a	C17 H9 N2 O2 Cl Br

^{1}H NMR spectra of **3g** (400 MHz, $CDCl_3$)



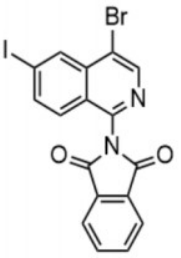
^{13}C NMR spectra of **3g** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

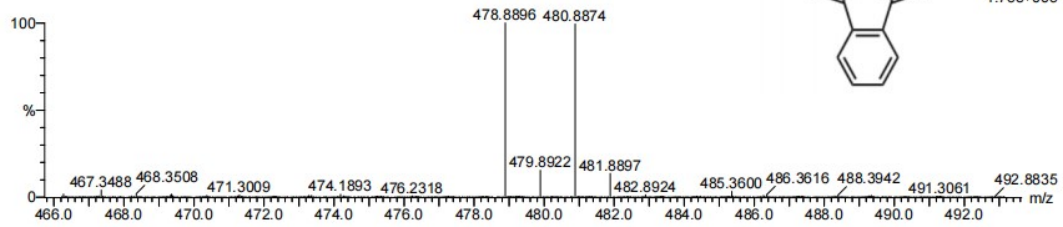
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
391 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 17-17 H: 9-9 N: 0-100 O: 0-100 Br: 1-4 I: 1-4



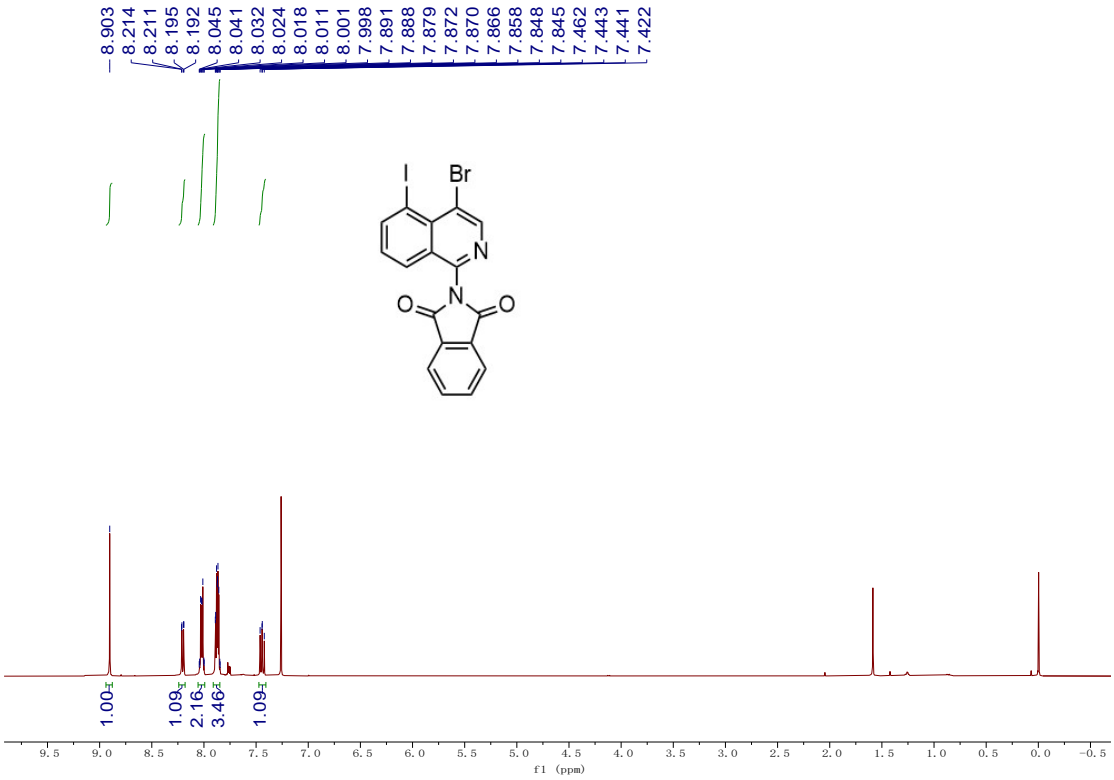
44
250616-5-12 7 (0.143)

1: TOF MS ES+
1.75e+005

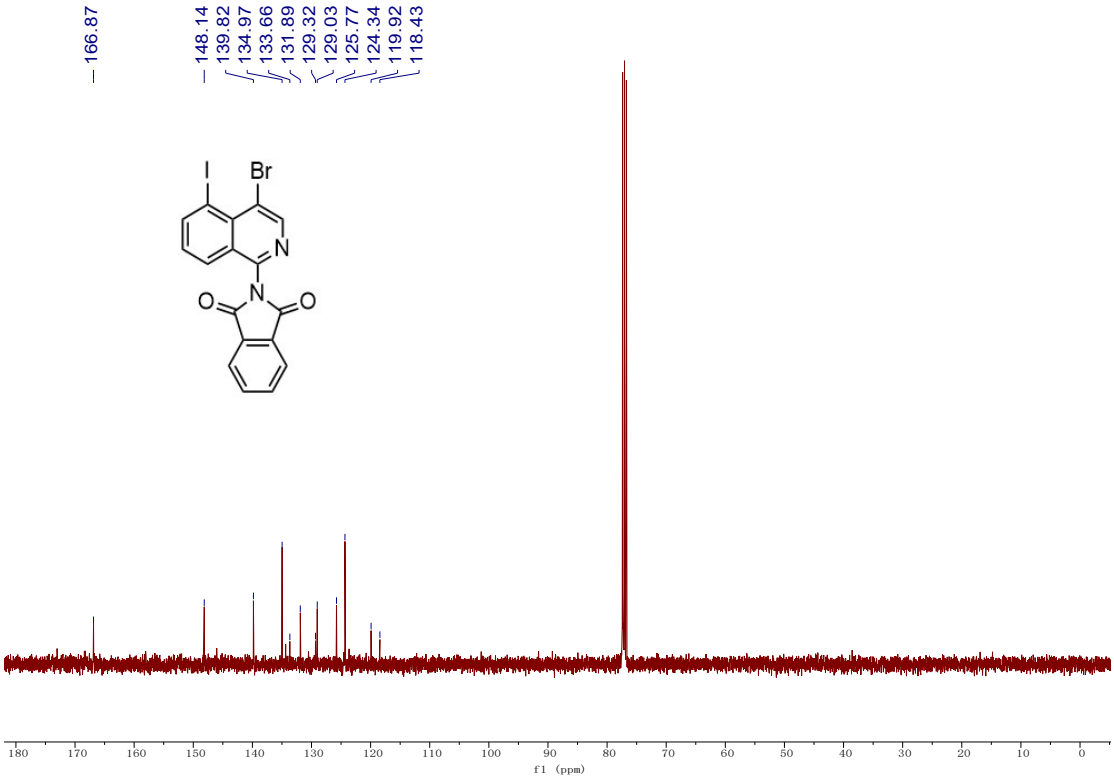


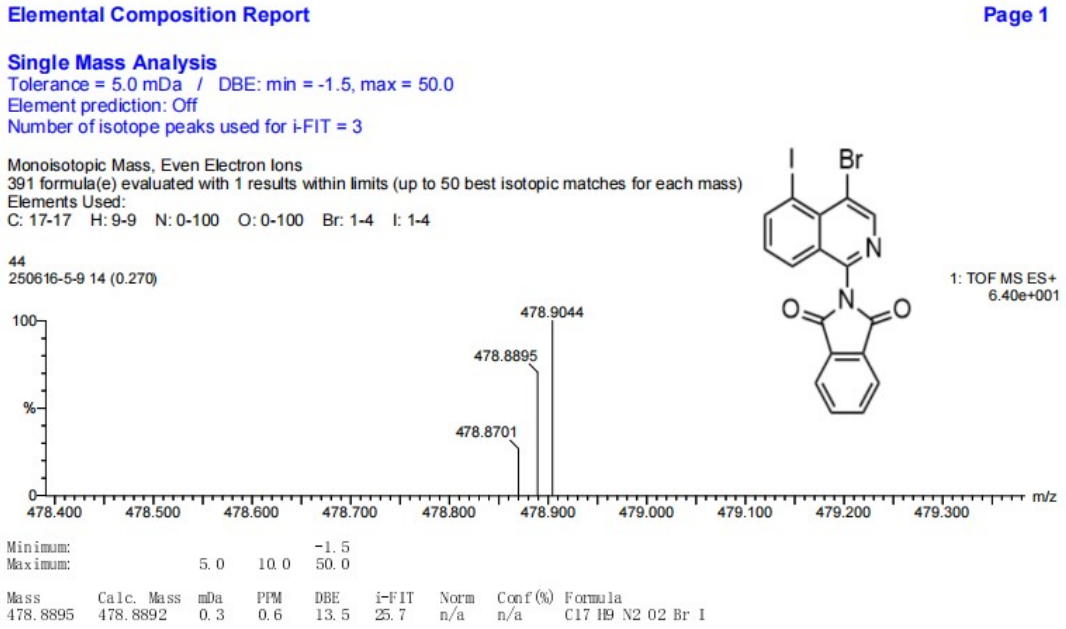
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
478.8896	478.8892	0.4	0.8	13.5	919.7	n/a	n/a	C17 H9 N2 O2 Br I

^{1}H NMR spectra of **3h** (400 MHz, $CDCl_3$)

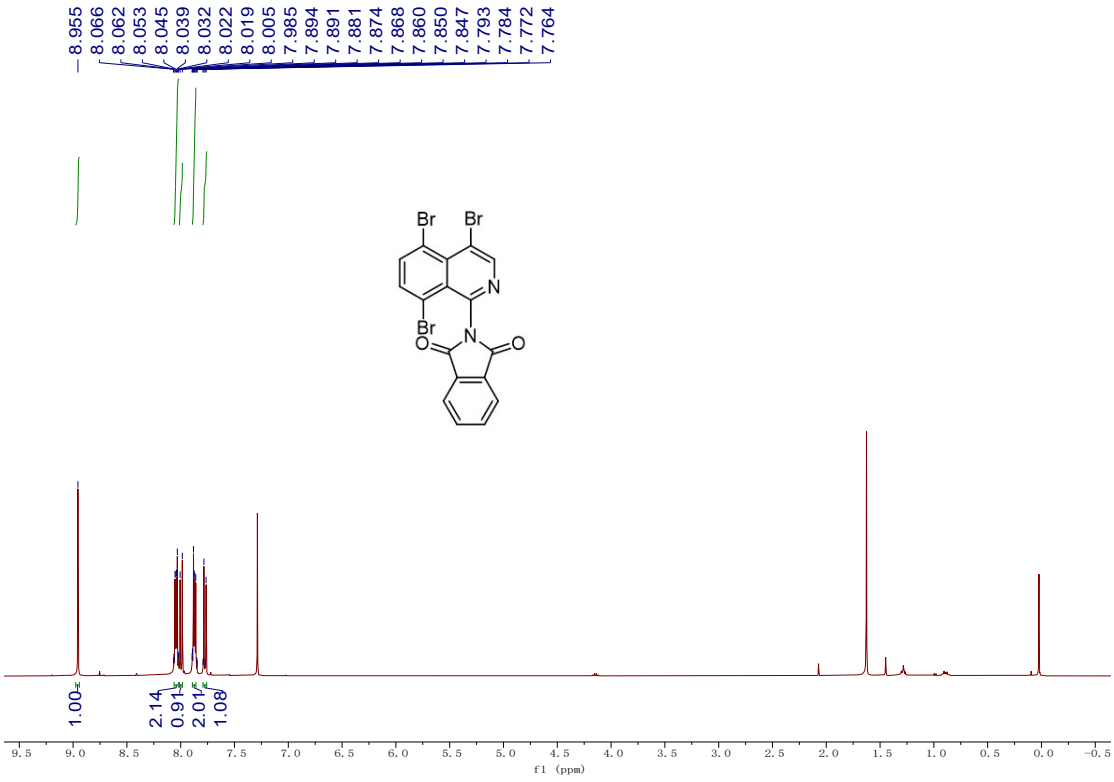


^{13}C NMR spectra of **3h** (101 MHz, $CDCl_3$)

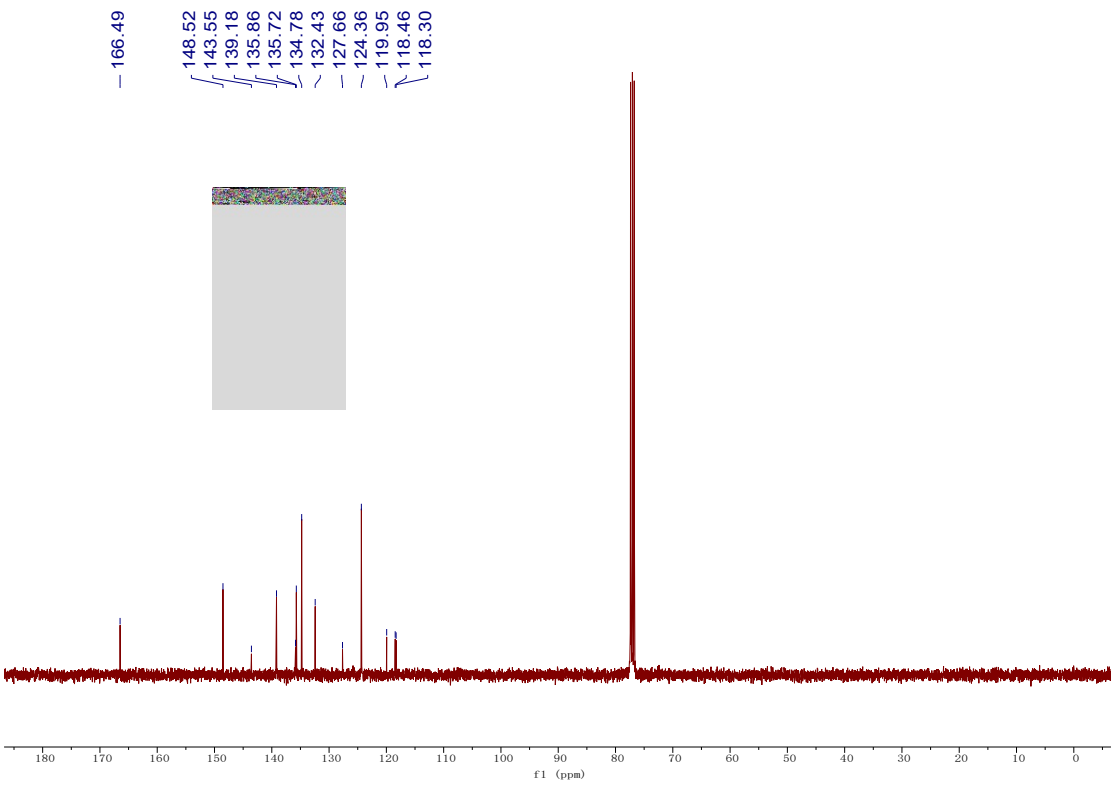




^{1}H NMR spectra of **3i** (400 MHz, $CDCl_3$)



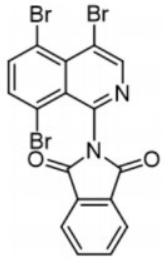
^{13}C NMR spectra of **3i** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

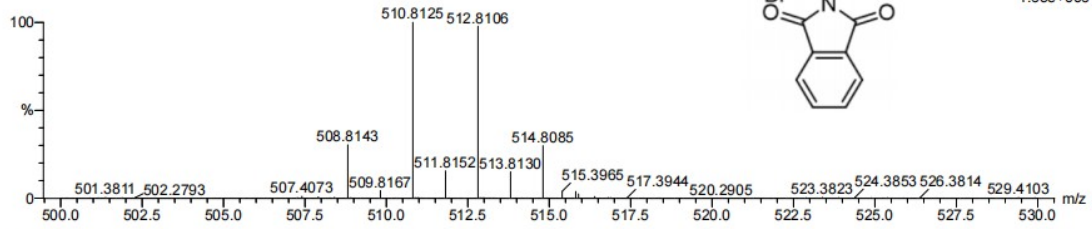
Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
36 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 17-17 H: 0-100 N: 2-2 O: 2-2 Na: 0-1 Se: 0-1 Br: 1-3



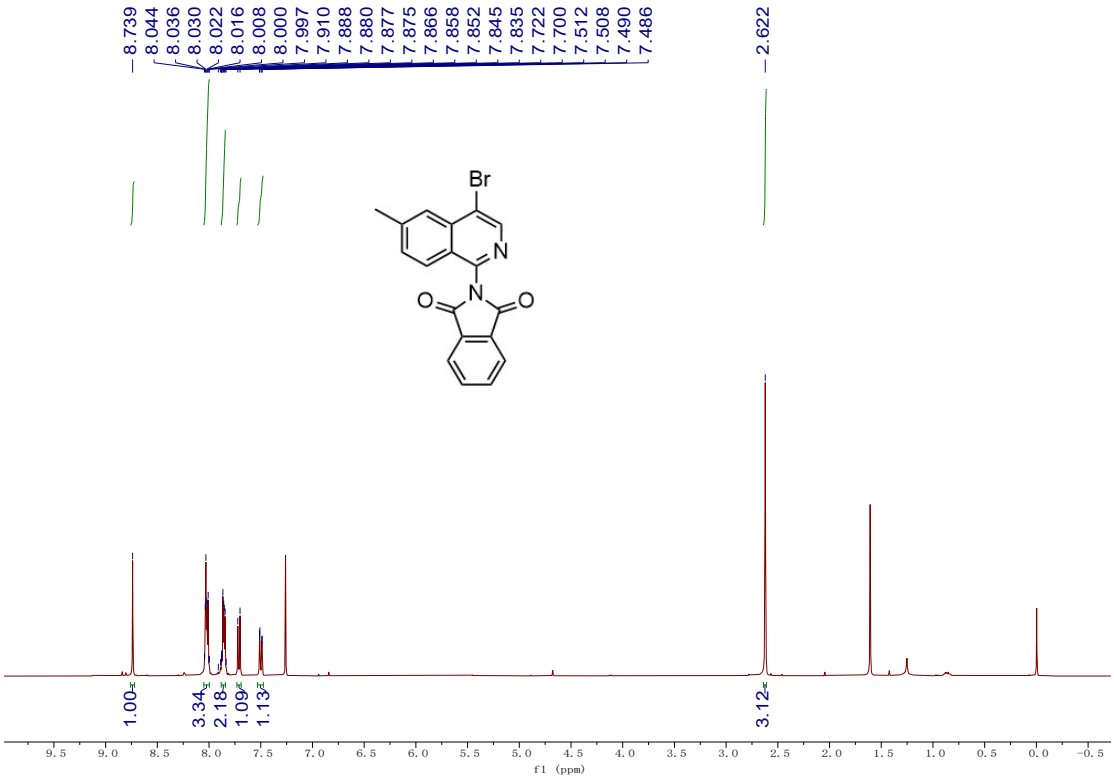
44
250616-5-5 4 (0.093)

1: TOF MS ES+
1.63e+006

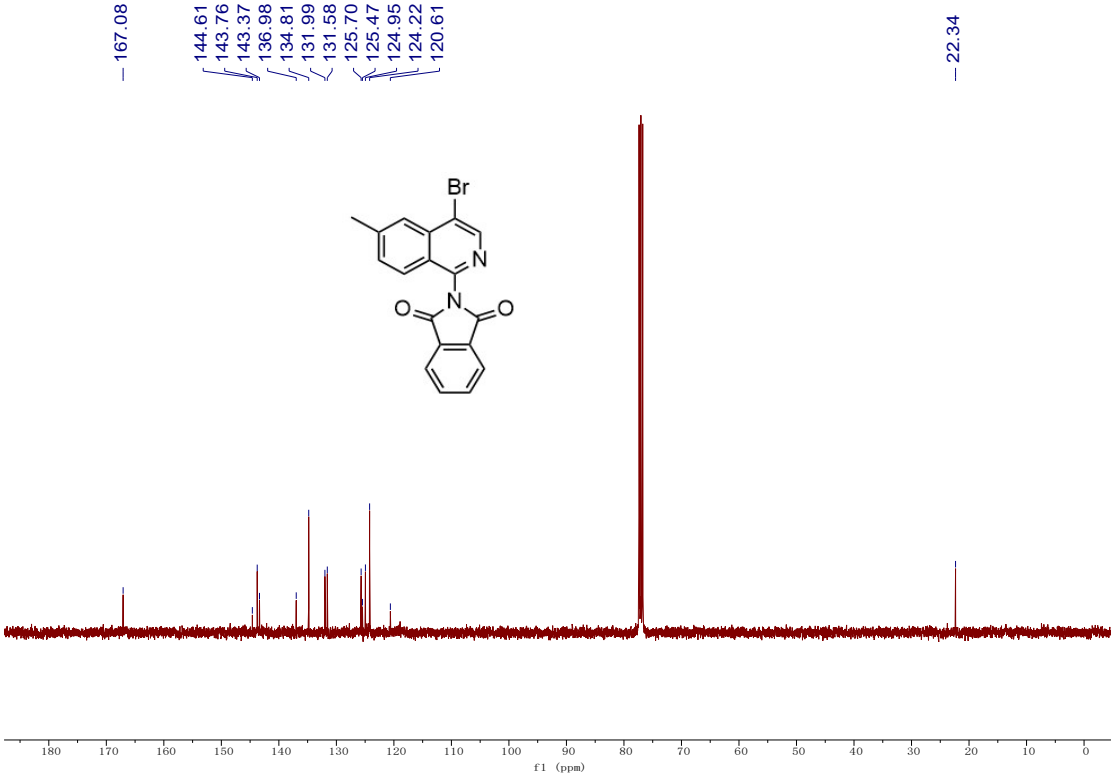


Minimum:	30.00				-1.5				
Maximum:	100.00		5.0	5.0	50.0				
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
508.8143	30.33	508.8136	0.7	1.4	13.5	917.3	n/a	n/a	C17 H8 N2 O2 Br3
510.8125	100.00	---							
512.8106	97.79	---							

^{1}H NMR spectra of **3j** (400 MHz, $CDCl_3$)



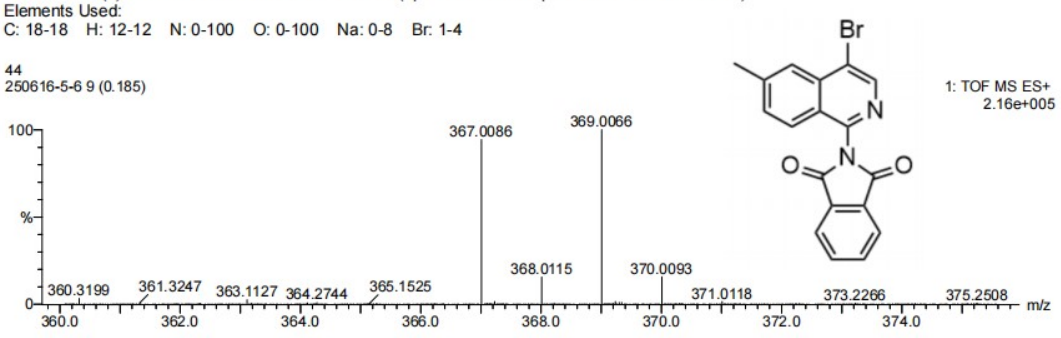
^{13}C NMR spectra of **3j** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

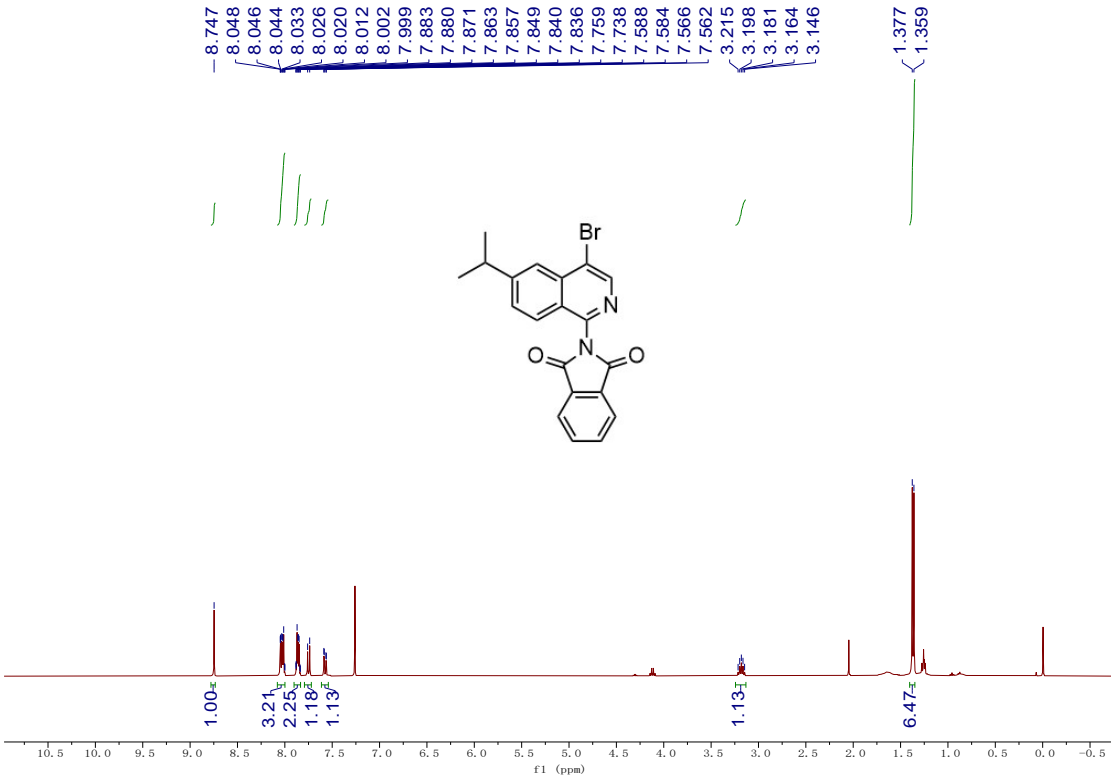
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1499 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 18-18 H: 12-12 N: 0-100 O: 0-100 Na: 0-8 Br: 1-4

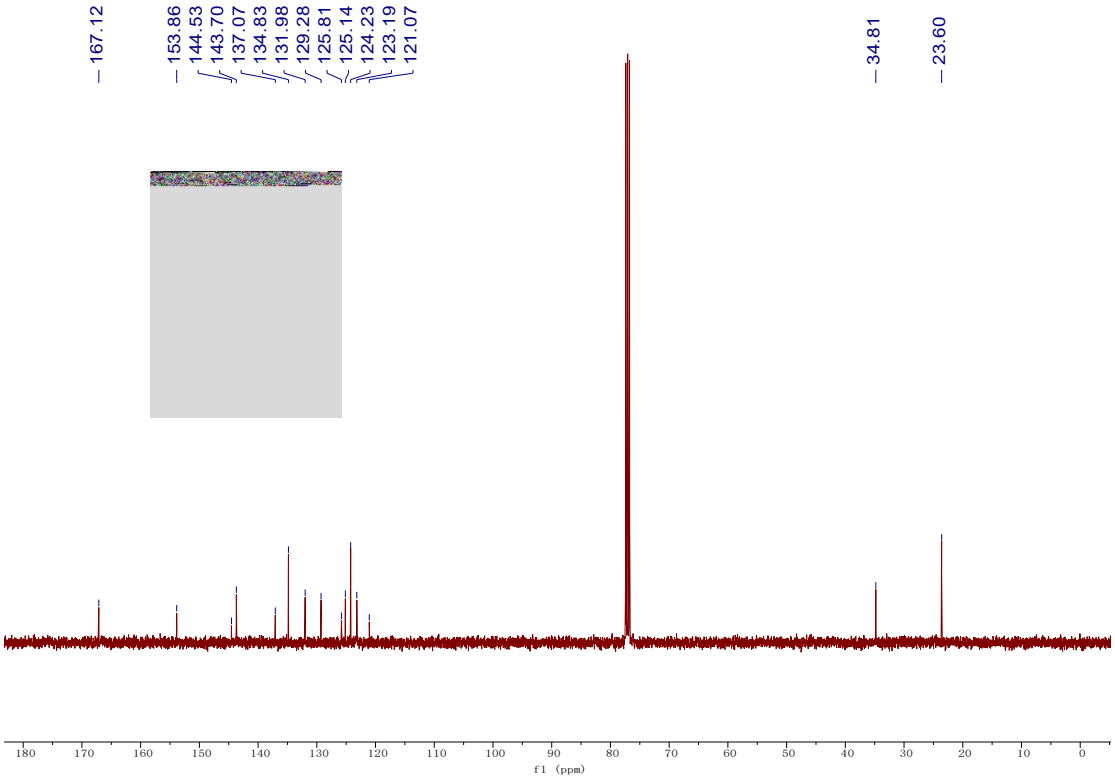


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
367.0086	367.0082	0.4	1.1	13.5	916.2	n/a	n/a	C18 H12 N2 O2 Br

^{1}H NMR spectra of **3k** (400 MHz, $CDCl_3$)



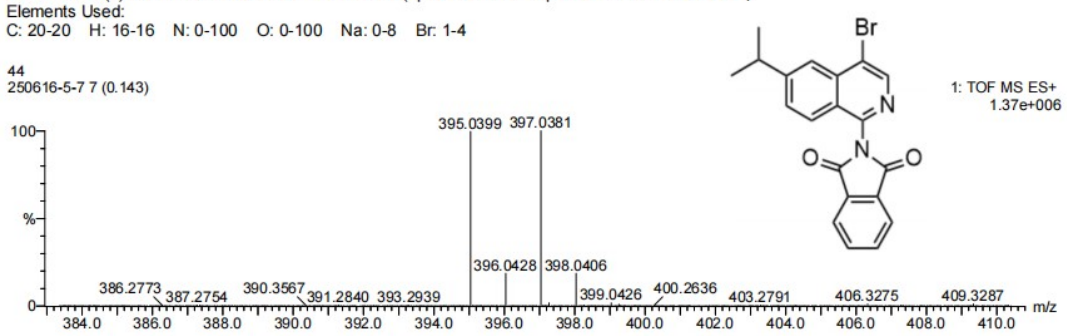
^{13}C NMR spectra of **3k** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

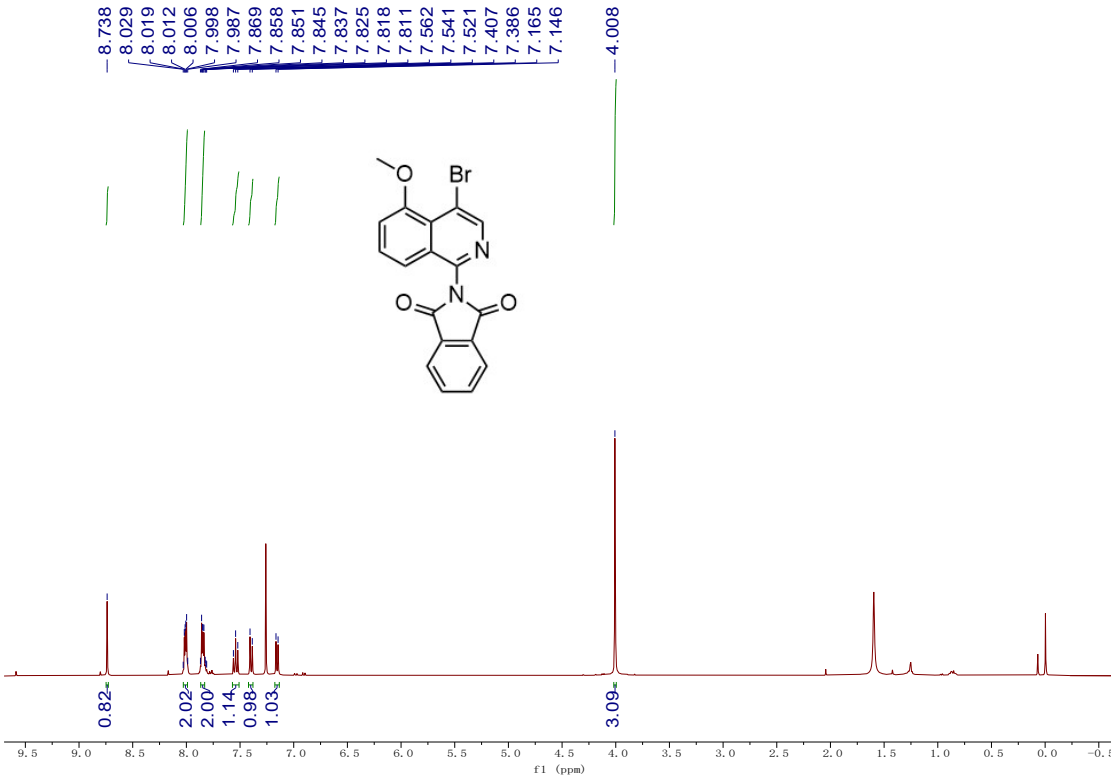
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
2014 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 20-20 H: 16-16 N: 0-100 O: 0-100 Na: 0-8 Br: 1-4

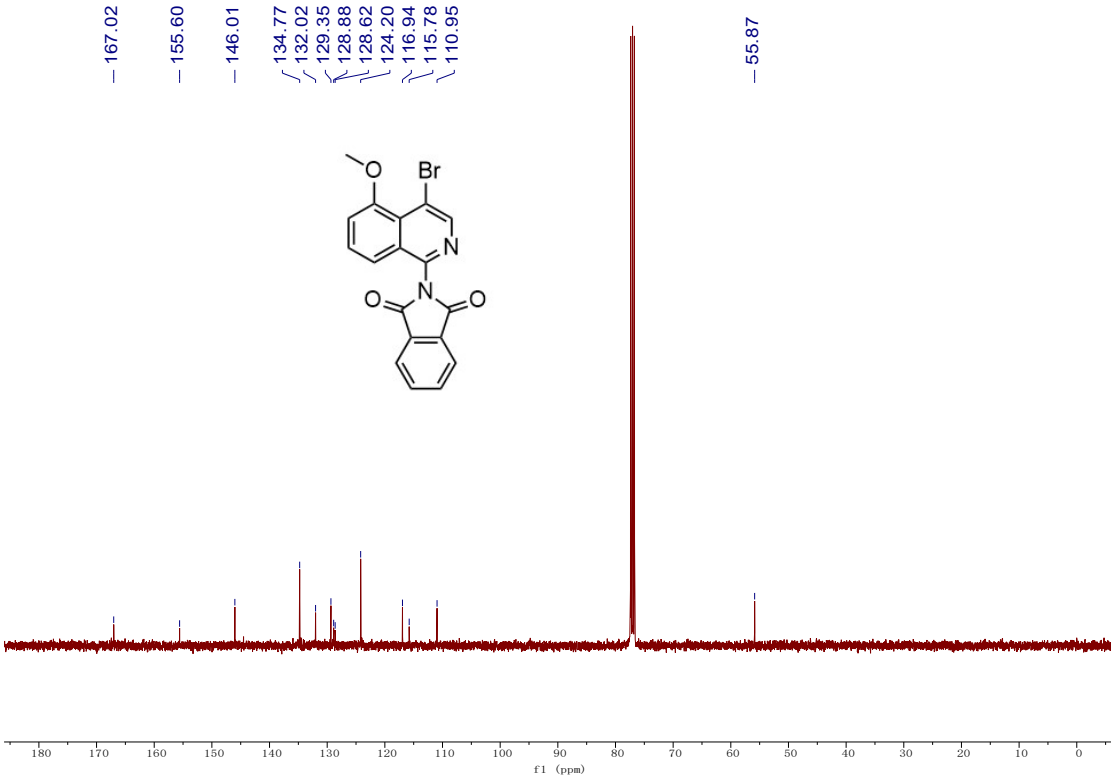


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
395.0399	395.0395	0.4	1.0	13.5	1052.3	n/a	n/a	C20 H16 N2 O2 Br

^{1}H NMR spectra of **3l** (400 MHz, $CDCl_3$)



^{13}C NMR spectra of **3l** (101 MHz, $CDCl_3$)

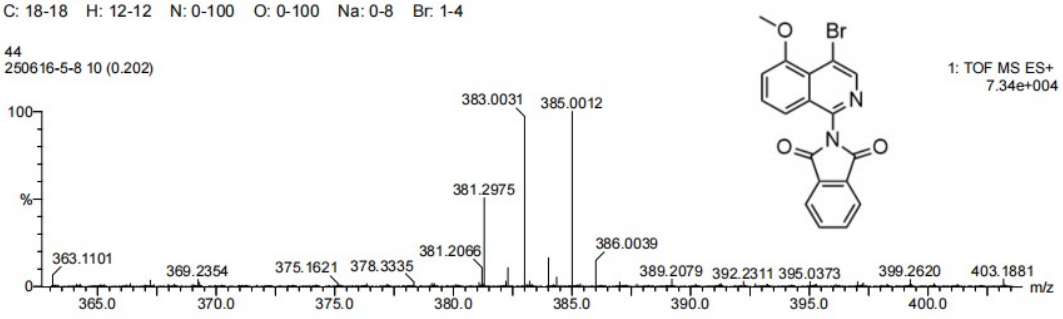


Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

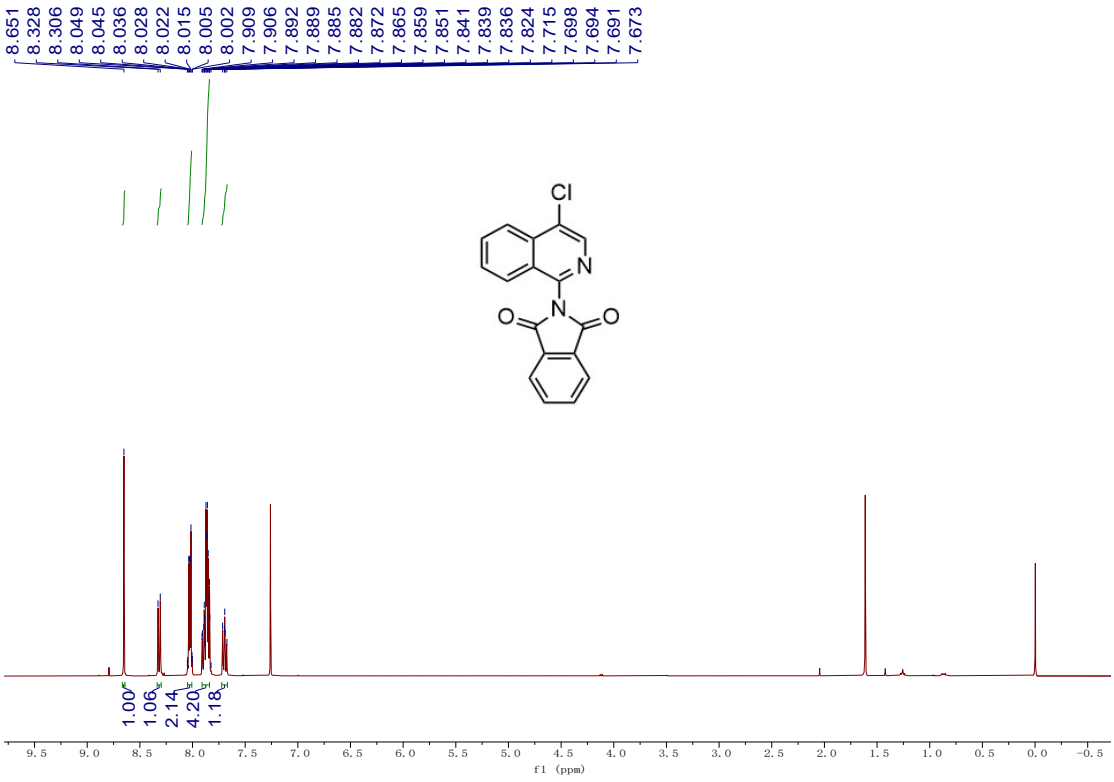
Monoisotopic Mass, Even Electron Ions
1765 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 18-18 H: 12-12 N: 0-100 O: 0-100 Na: 0-8 Br: 1-4

44
250616-5-8 10 (0.202)

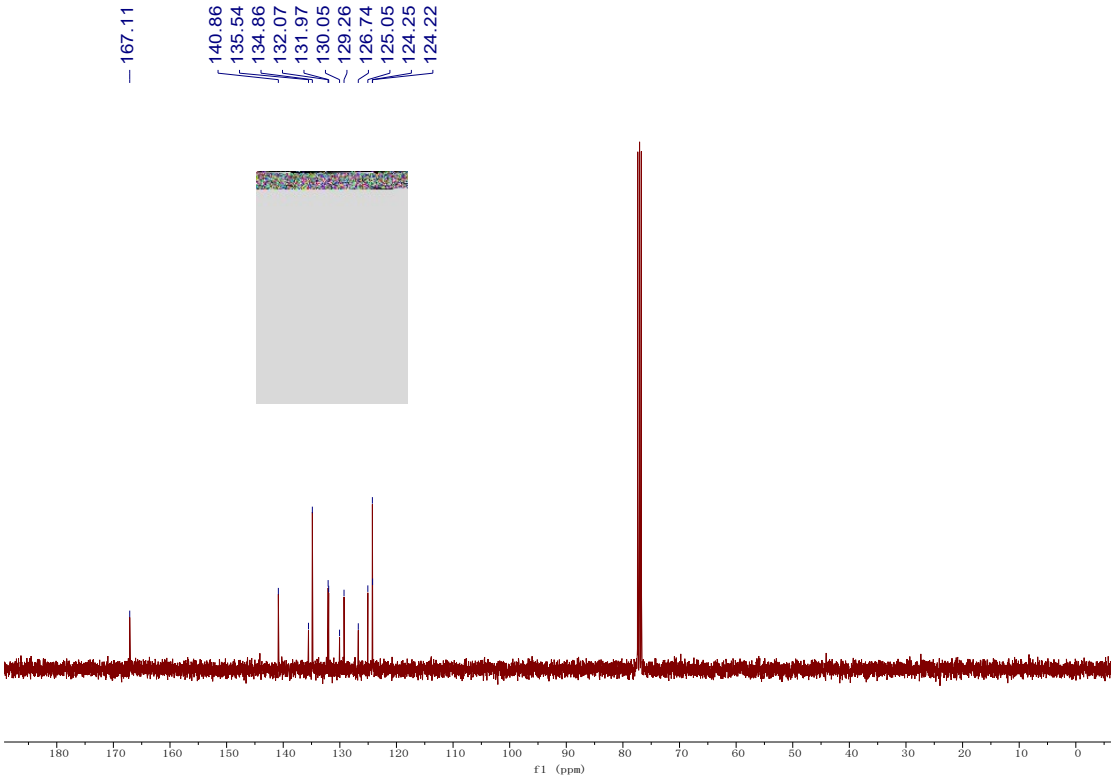


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
383.0031	383.0031	0.0	0.0	13.5	840.9	n/a	n/a	C18 H12 N2 O3 Br

^{1}H NMR spectra of **3m** (400 MHz, $CDCl_3$)



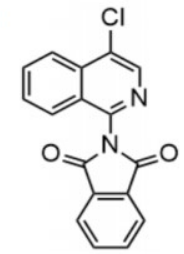
^{13}C NMR spectra of **3m** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

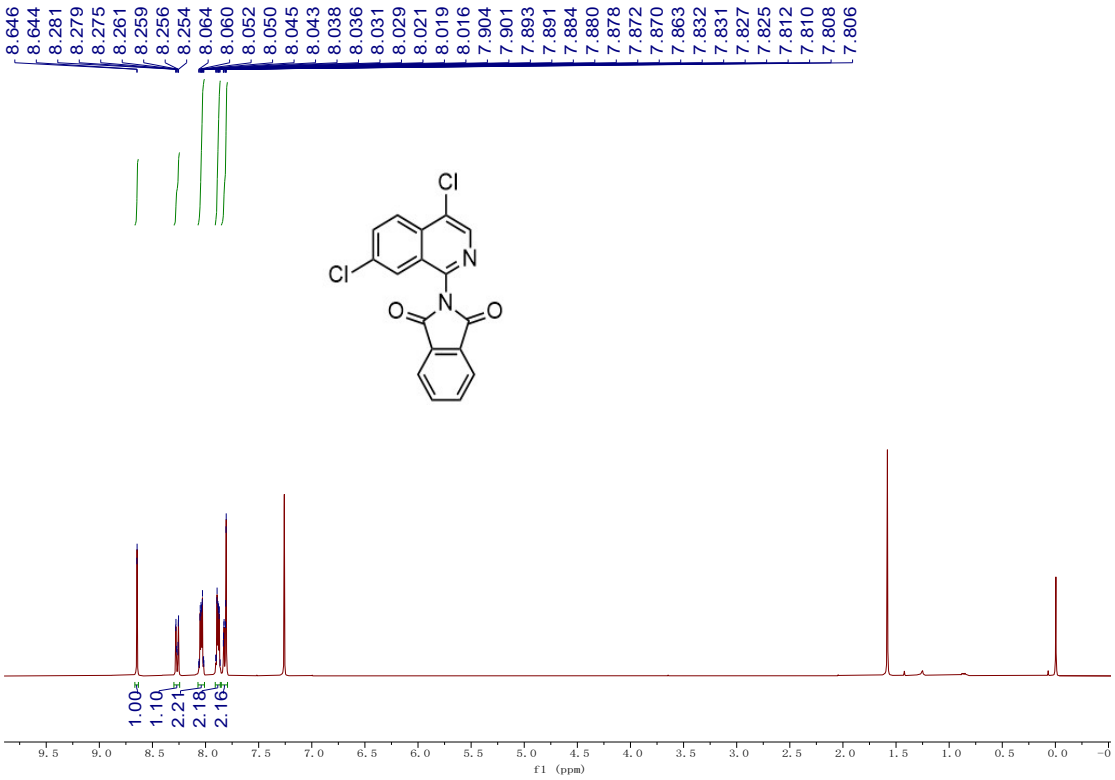
Monoisotopic Mass, Even Electron Ions
572 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 17-17 H: 10-10 N: 0-100 O: 0-100 Cl: 1-6
44
250616-5-14 11 (0.219)



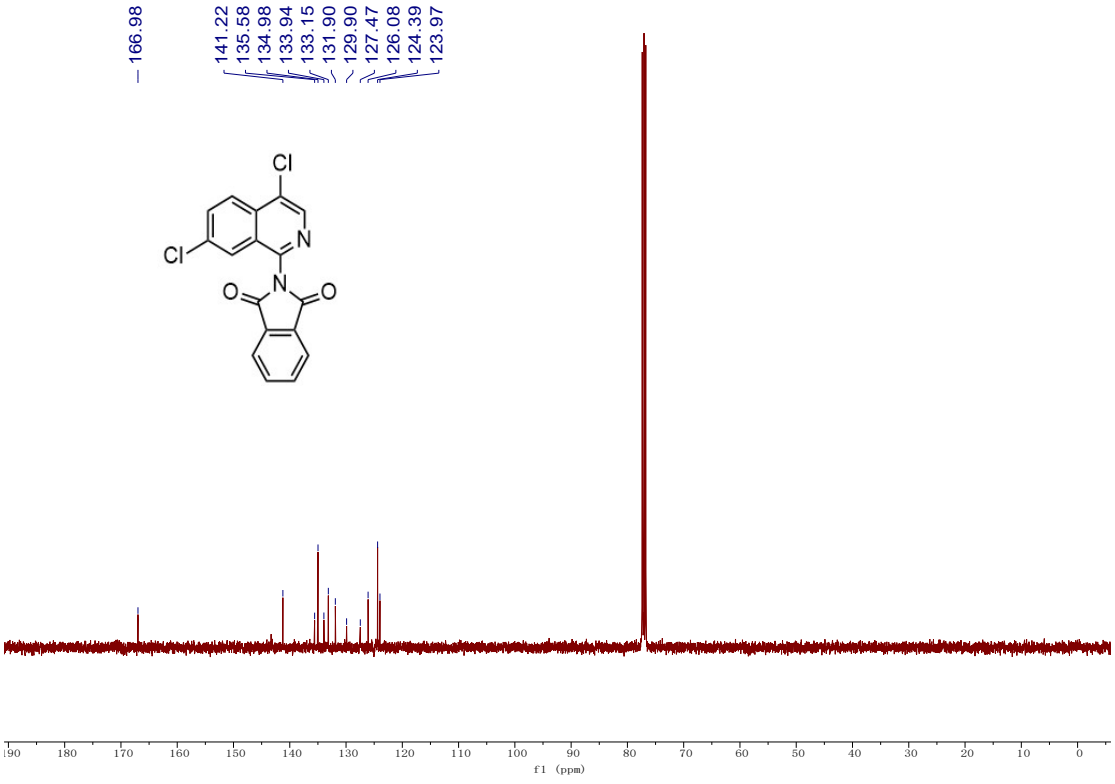
1: TOF MS ES+
1.26e+005

Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
309.0435	309.0431	0.4	1.3	13.5	834.9	n/a	n/a	C17 H10 N2 O2 Cl

^{1}H NMR spectra of **3n** (400 MHz, $CDCl_3$)



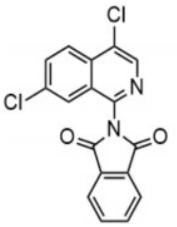
^{13}C NMR spectra of **3n** (101 MHz, $CDCl_3$)



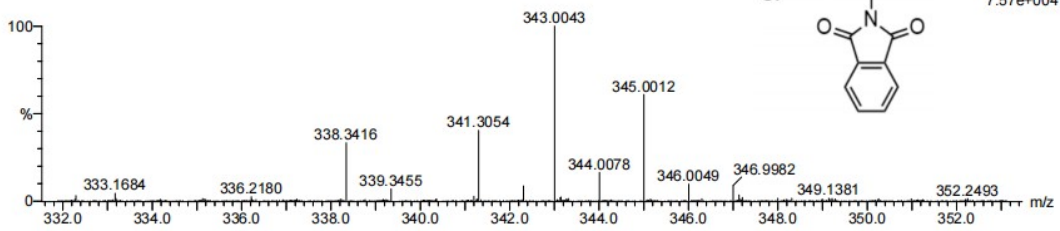
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
747 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 17-17 H: 9-9 N: 0-100 O: 0-100 Cl: 1-6
44
250616-5-15 9 (0.185)

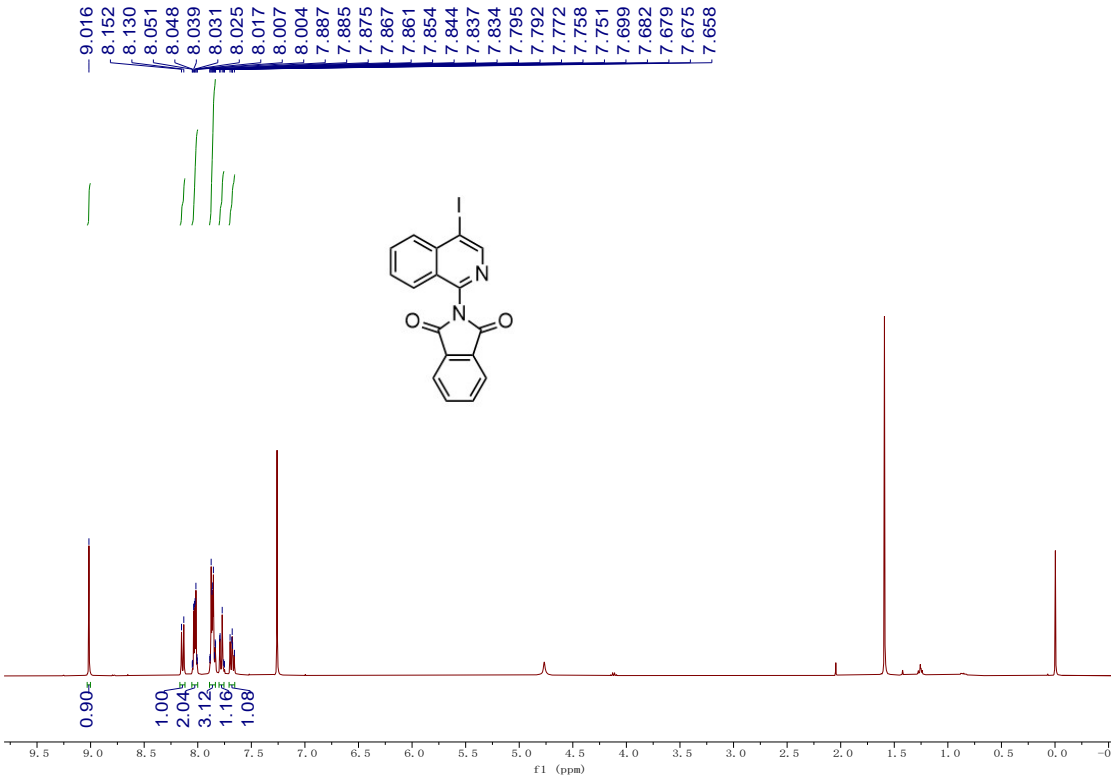


1: TOF MS ES+
7.57e+004

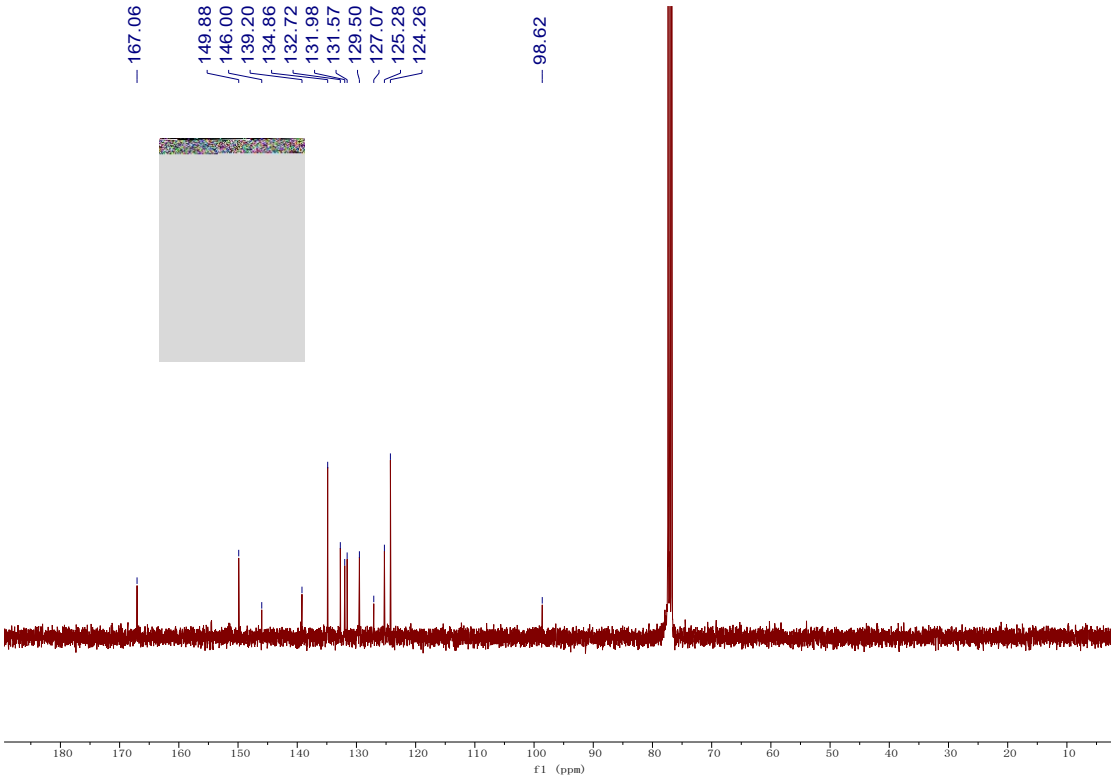


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
343.0043	343.0041	0.2	0.6	13.5	745.8	n/a	n/a	C17 H9 N2 O2 Cl2

^{1}H NMR spectra of **3o** (400 MHz, $CDCl_3$)



^{13}C NMR spectra of **3o** (101 MHz, $CDCl_3$)

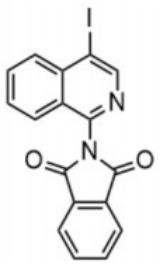


Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
235 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 17-17 H: 10-10 N: 0-100 O: 0-100 I: 1-4

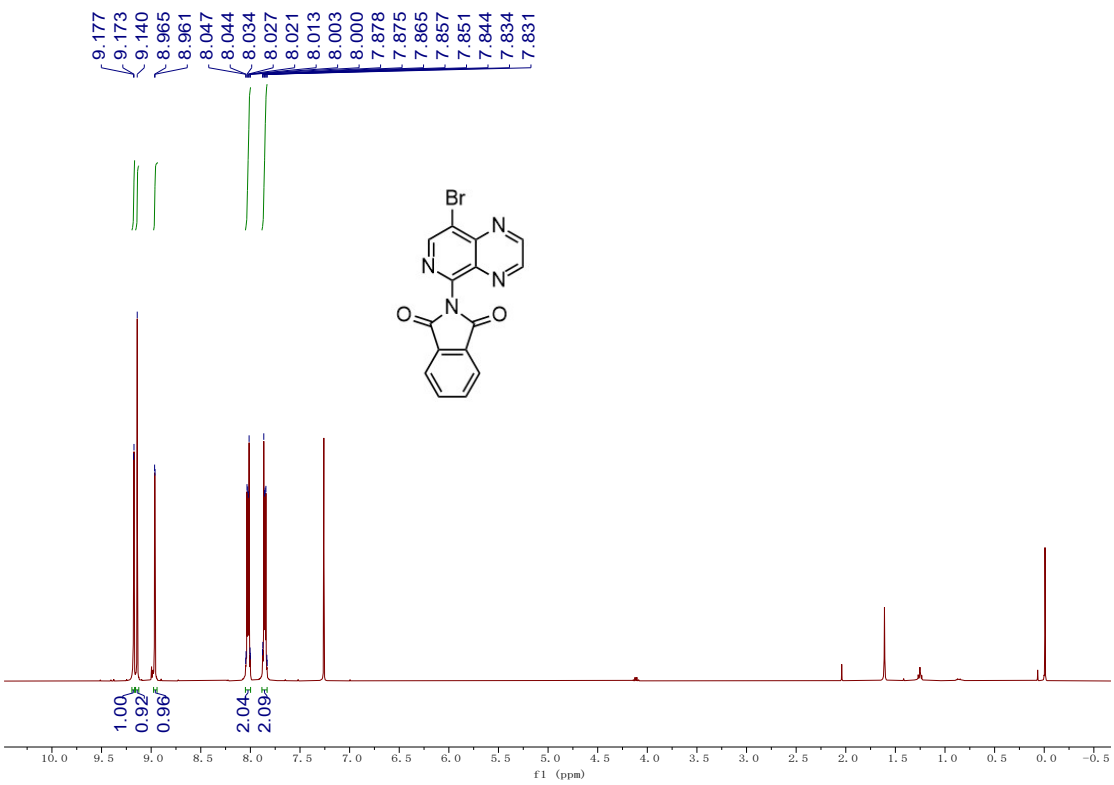
44
250616-5-16 8 (0.160)



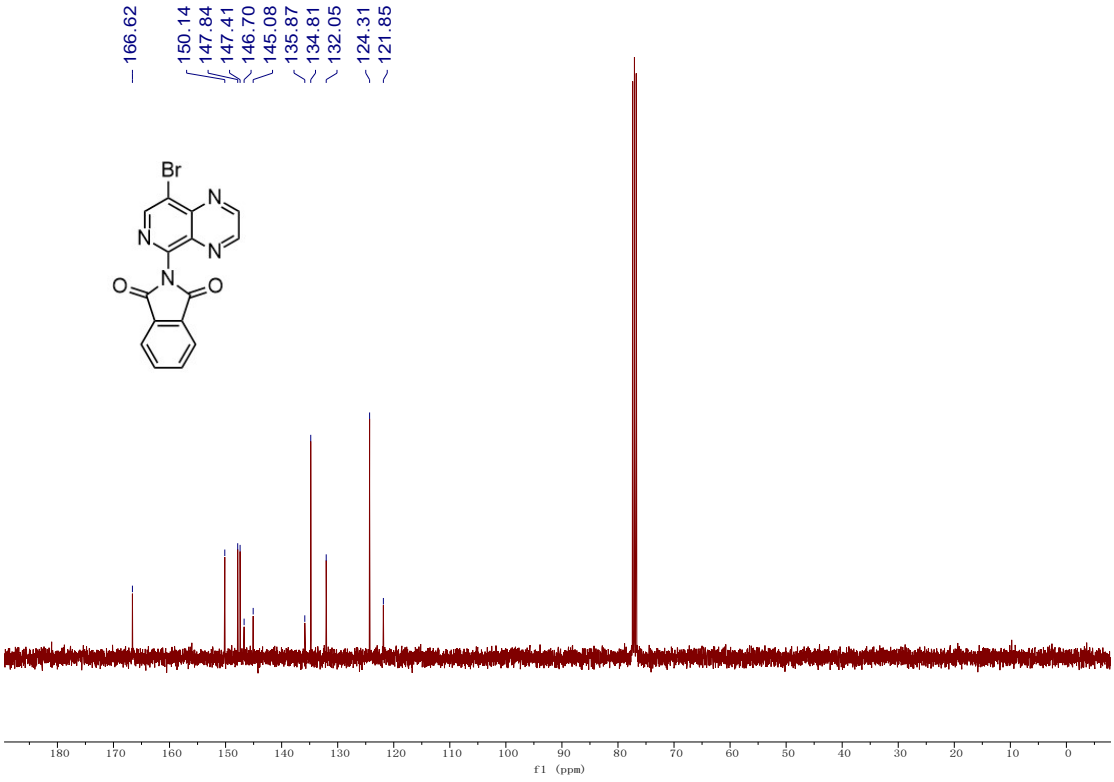
1: TOF MS ES+
5.34e+005

Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
400.9791	400.9787	0.4	1.0	13.5	966.8	n/a	n/a	C17 H10 N2 O2 I

^{1}H NMR spectra of **3p** (400 MHz, $CDCl_3$)



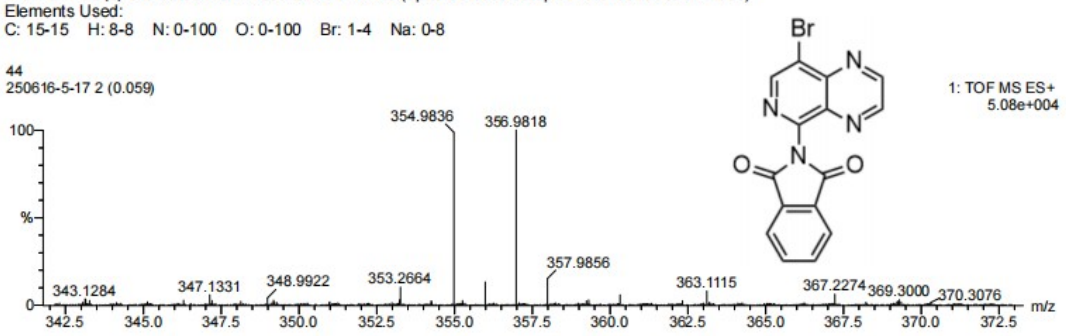
^{13}C NMR spectra of **3p** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

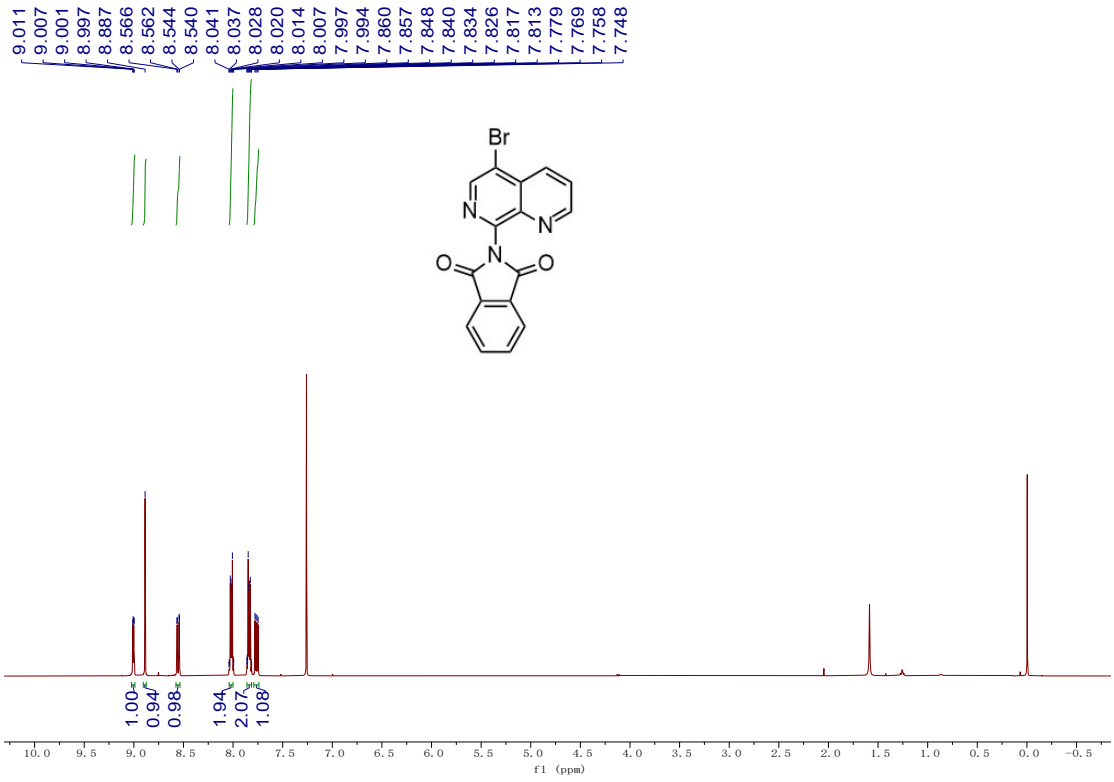
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1281 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 15-15 H: 8-8 N: 0-100 O: 0-100 Br: 1-4 Na: 0-8

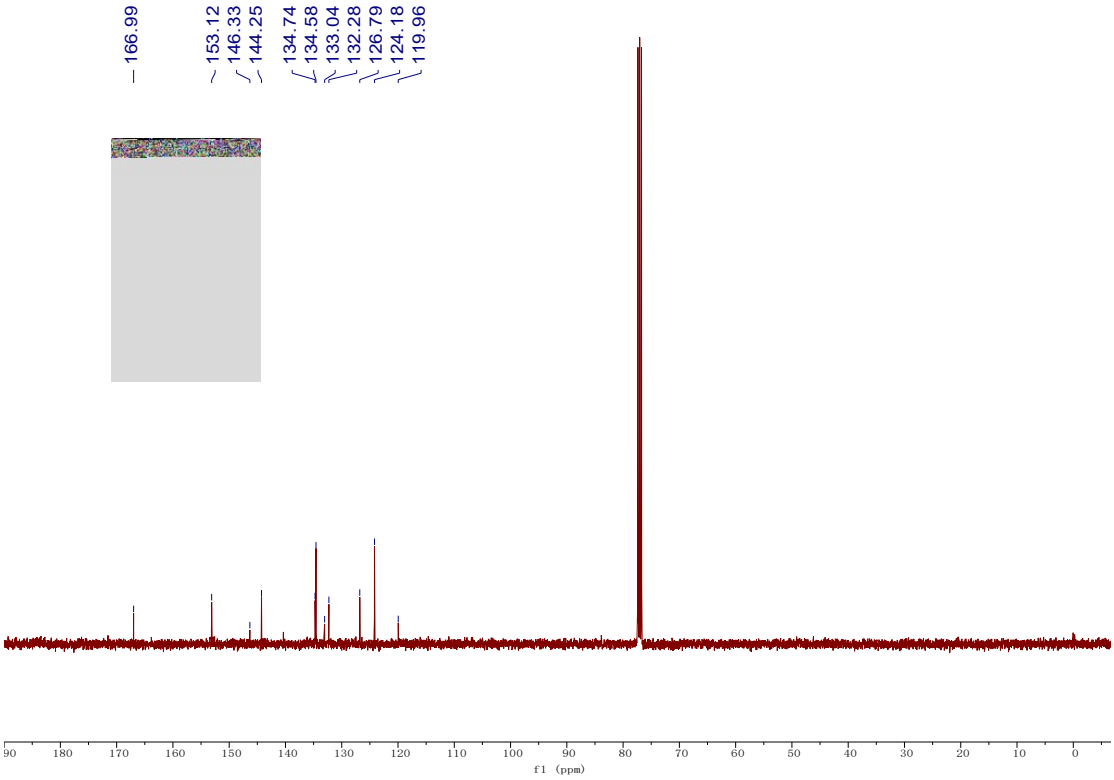


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
354.9836	354.9831	0.5	1.4	13.5	772.0	n/a	n/a	C15 H8 N4 O2 Br

^{1}H NMR spectra of **3q** (400 MHz, $CDCl_3$)



^{13}C NMR spectra of **3q** (101 MHz, $CDCl_3$)

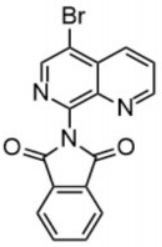


Elemental Composition Report **Page 1**

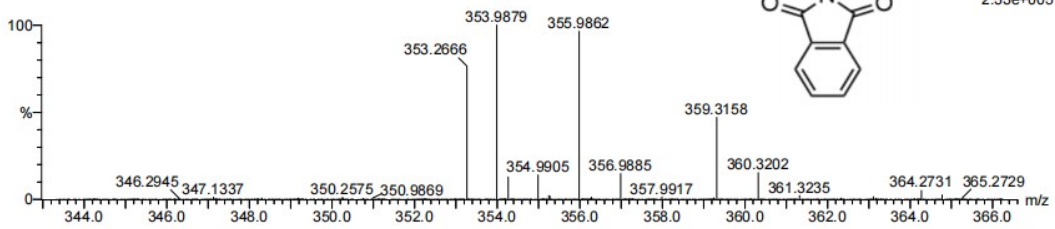
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1282 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 16-16 H: 9-9 N: 0-100 O: 0-100 Na: 0-8 Br: 1-4

44
250616-5-18 8 (0.160)

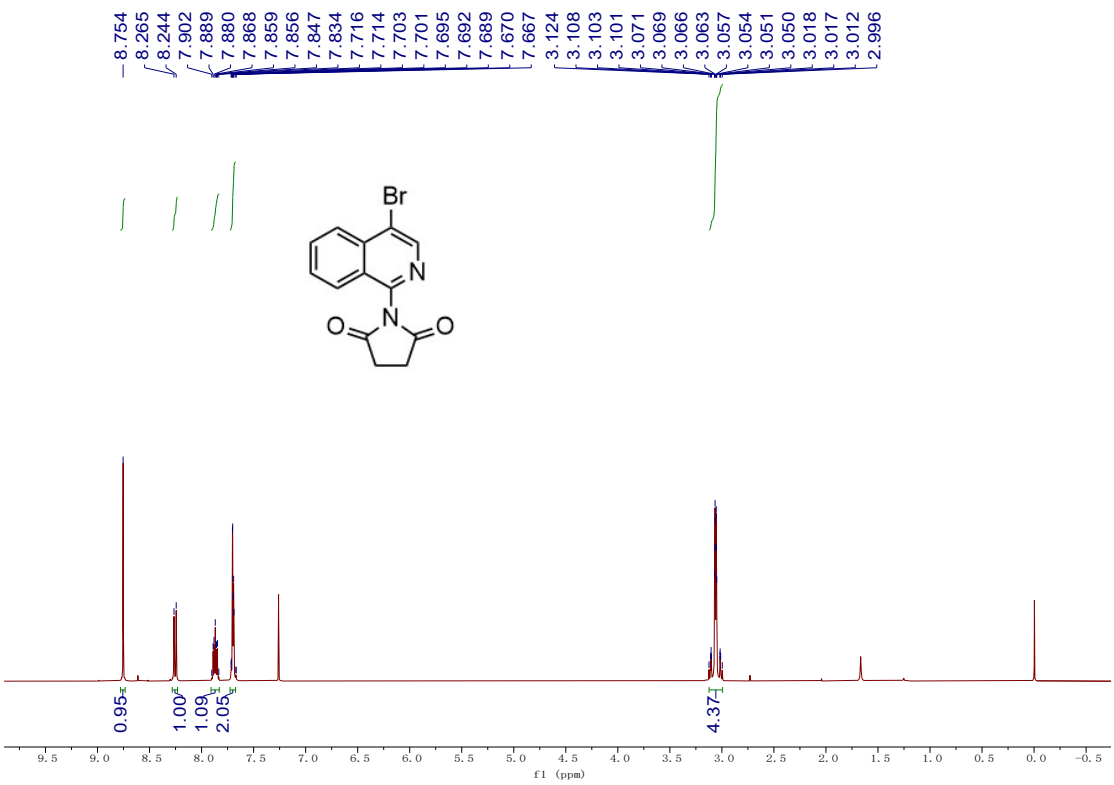


1: TOF MS ES+
2.33e+005

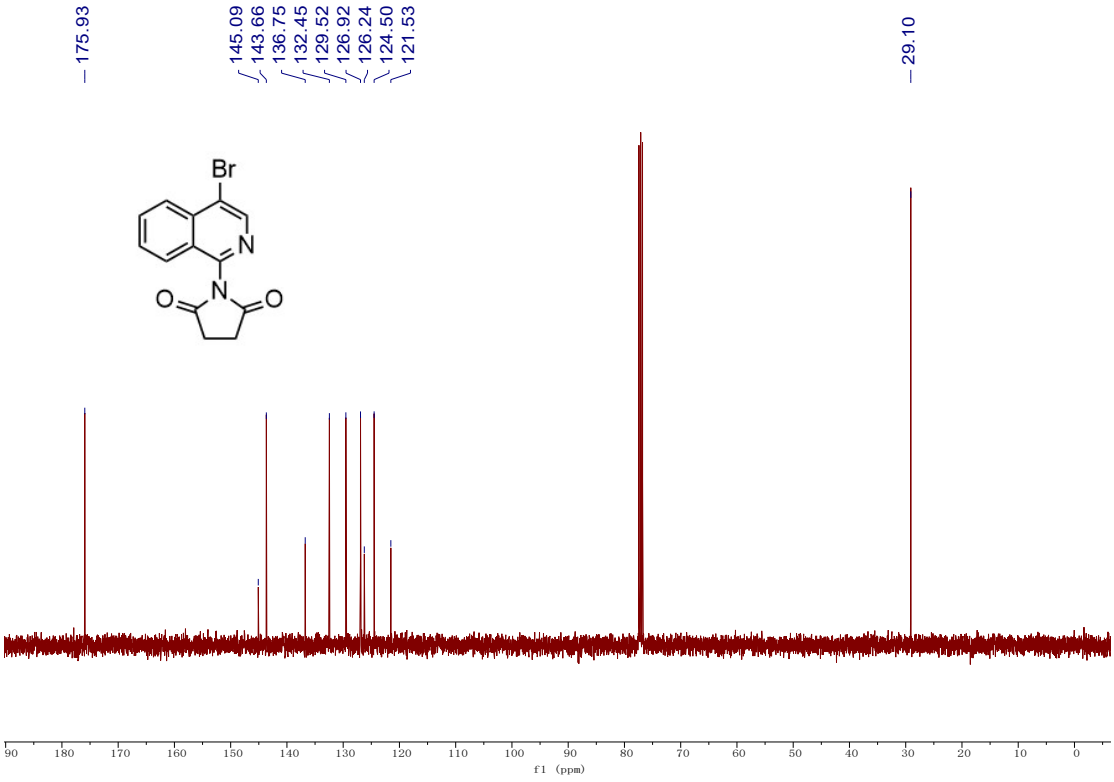


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
353.9879	353.9878	0.1	0.3	13.5	973.3	n/a	n/a	C16 H9 N3 O2 Br

^{1}H NMR spectra of **5a** (400 MHz, $CDCl_3$)



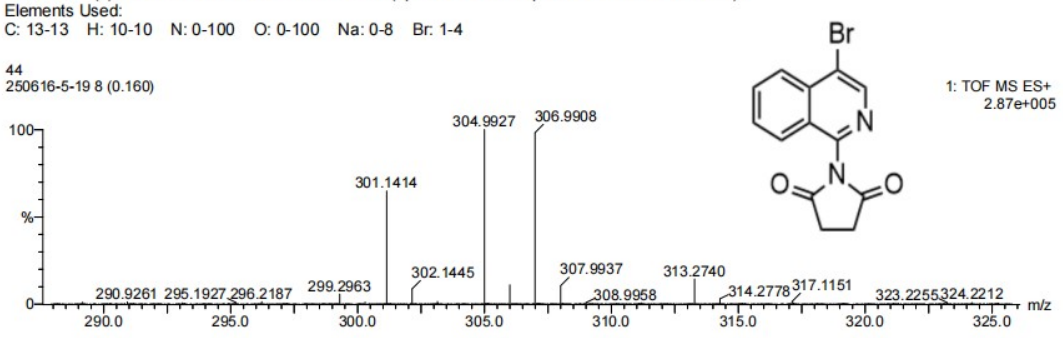
^{13}C NMR spectra of **5a** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

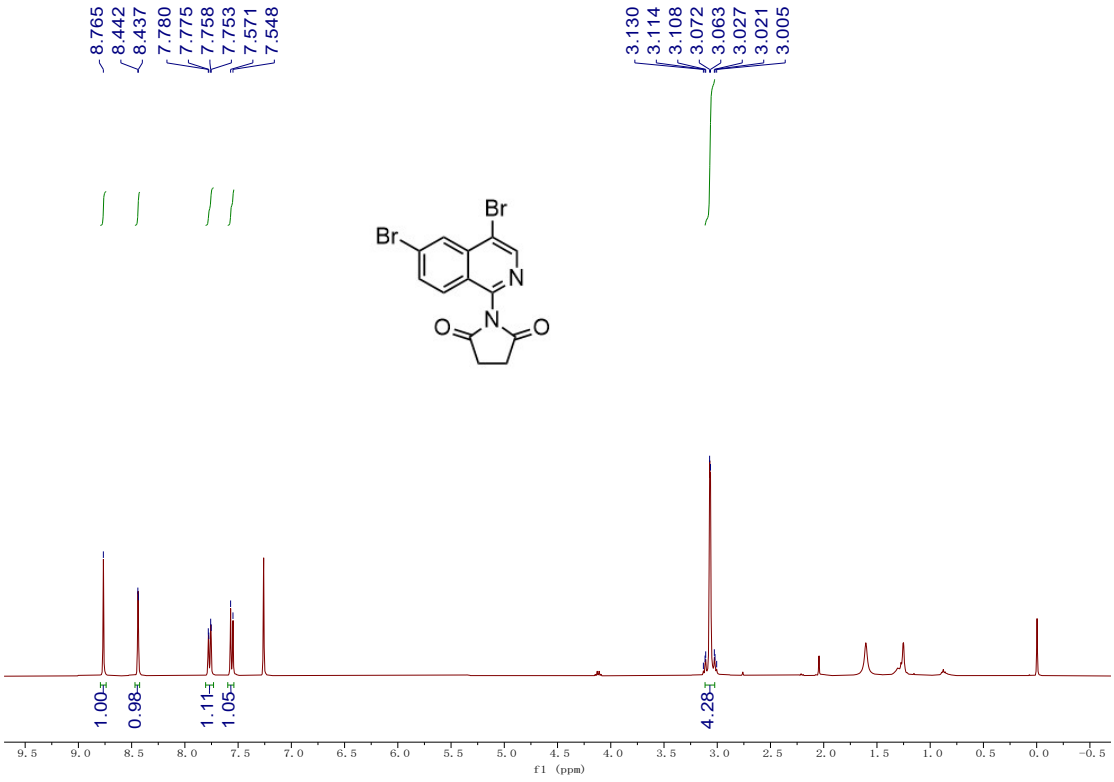
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
685 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-13 H: 10-10 N: 0-100 O: 0-100 Na: 0-8 Br: 1-4

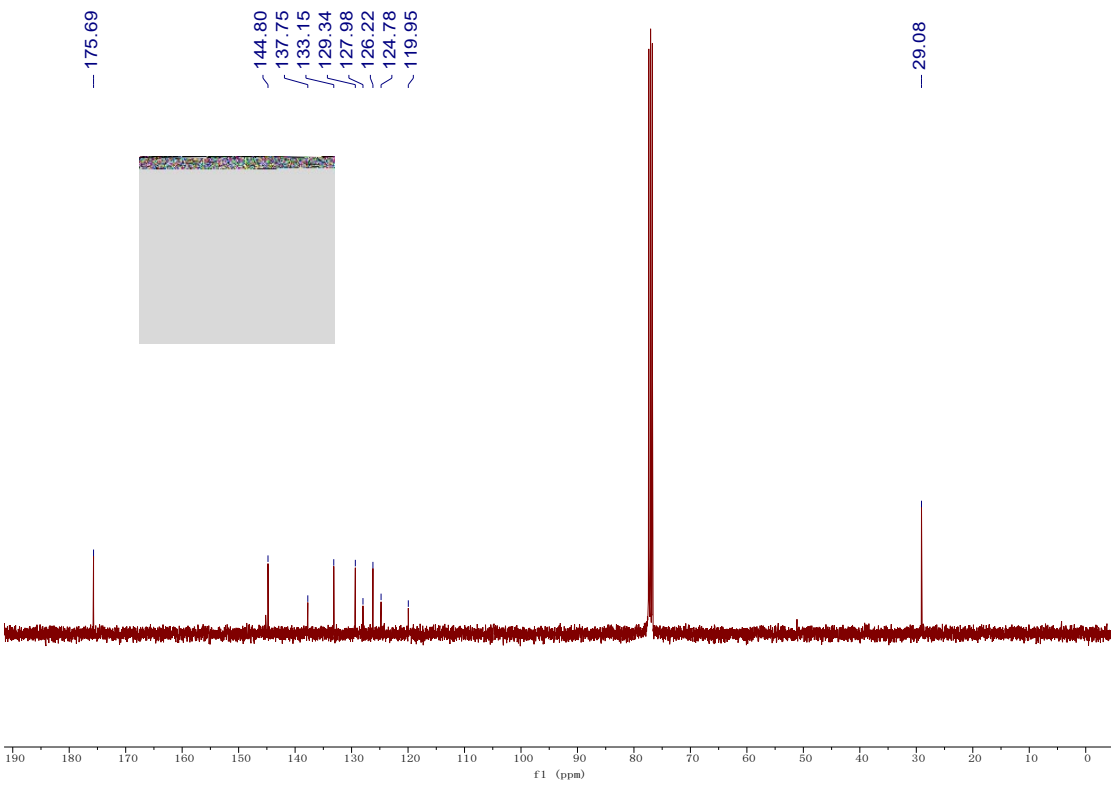


Minimum:				-1.5			
Maximum:		5.0	10.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%) Formula
304.9927	304.9926	0.1	0.3	9.5	1002.7	n/a	n/a C13 H10 N2 O2 Br

^{1}H NMR spectra of **5b** (400 MHz, $CDCl_3$)



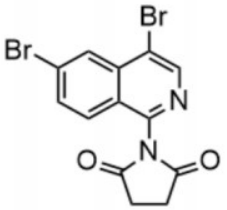
^{13}C NMR spectra of **5b** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

Multiple Mass Analysis: 3 mass(es) processed
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

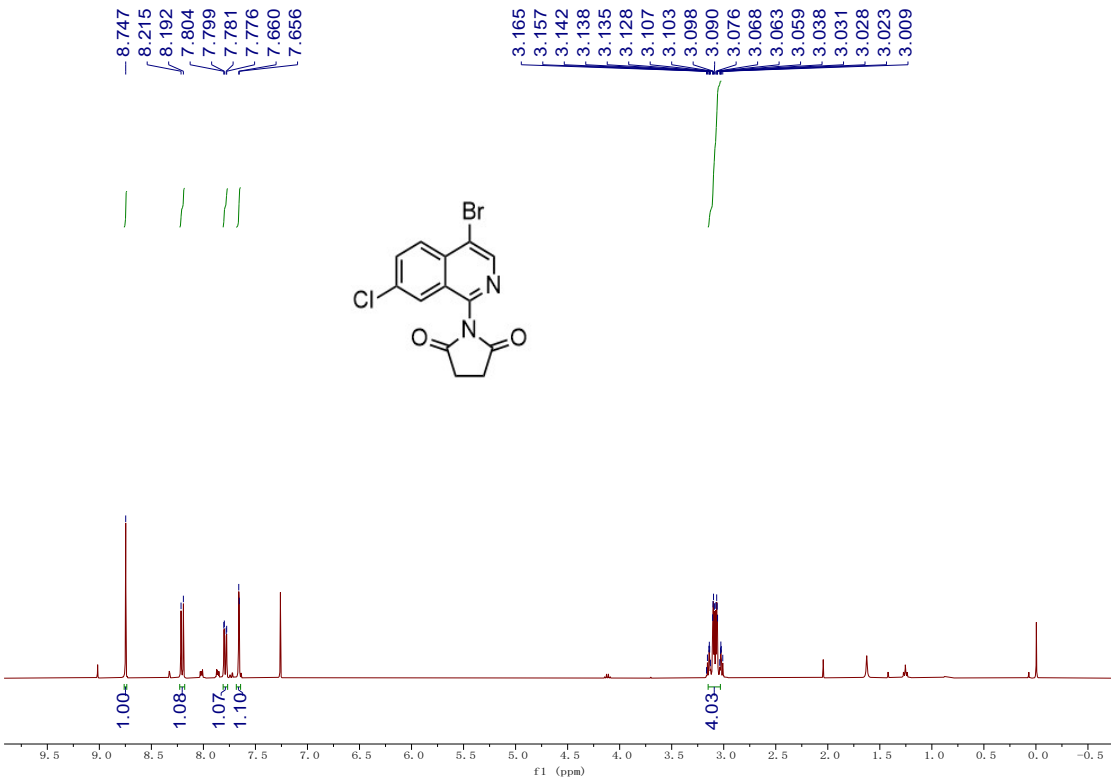
Monoisotopic Mass, Even Electron Ions
36 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 13-13 H: 0-100 N: 2-2 O: 2-2 Na: 0-1 Se: 0-1 Br: 0-2
44
250616-5-21 4 (0.093)



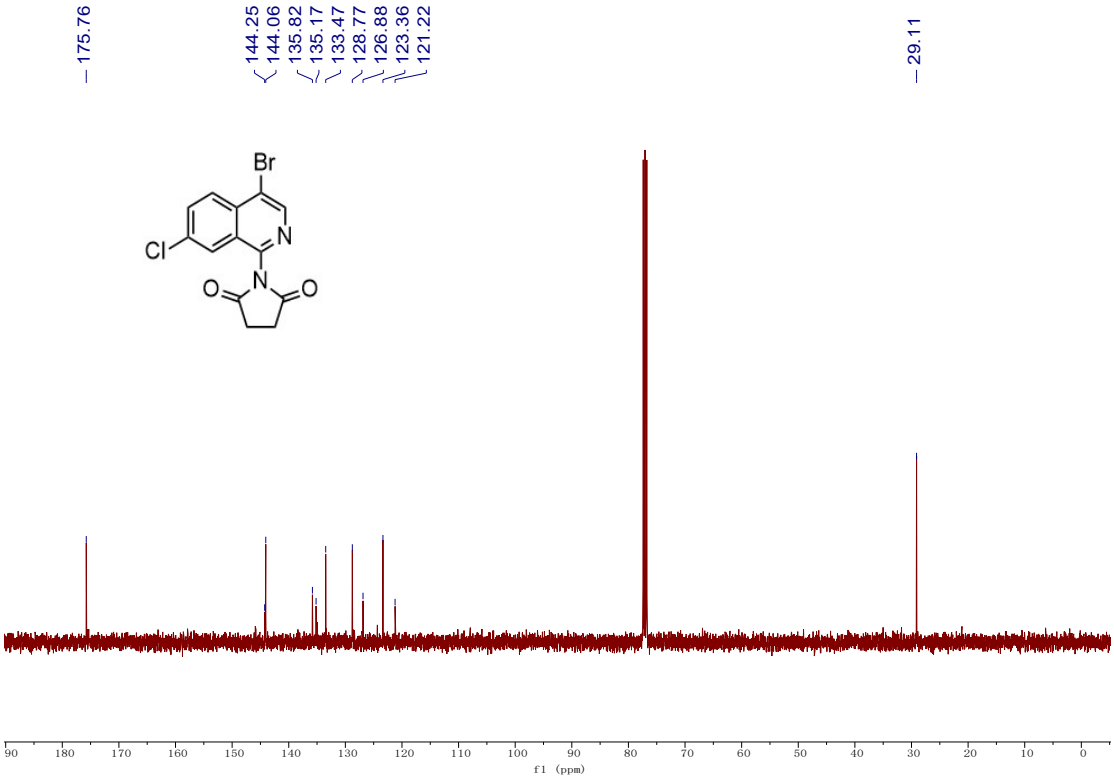
1: TOF MS ES+
6.56e+006

Minimum:	40.00				-1.5				
Maximum:	100.00		5.0	5.0	50.0				
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
382.9041	49.67	382.9031	1.0	2.6	9.5	1200.3	n/a	n/a	C13 H9 N2 O2 Br2
384.9026	100.00	---							
386.9003	48.73	---							

^{1}H NMR spectra of **5c** (400 MHz, $CDCl_3$)



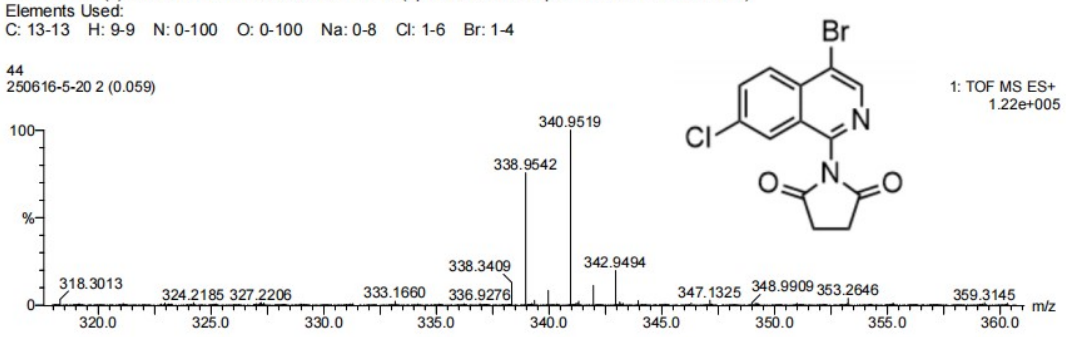
^{13}C NMR spectra of **5c** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

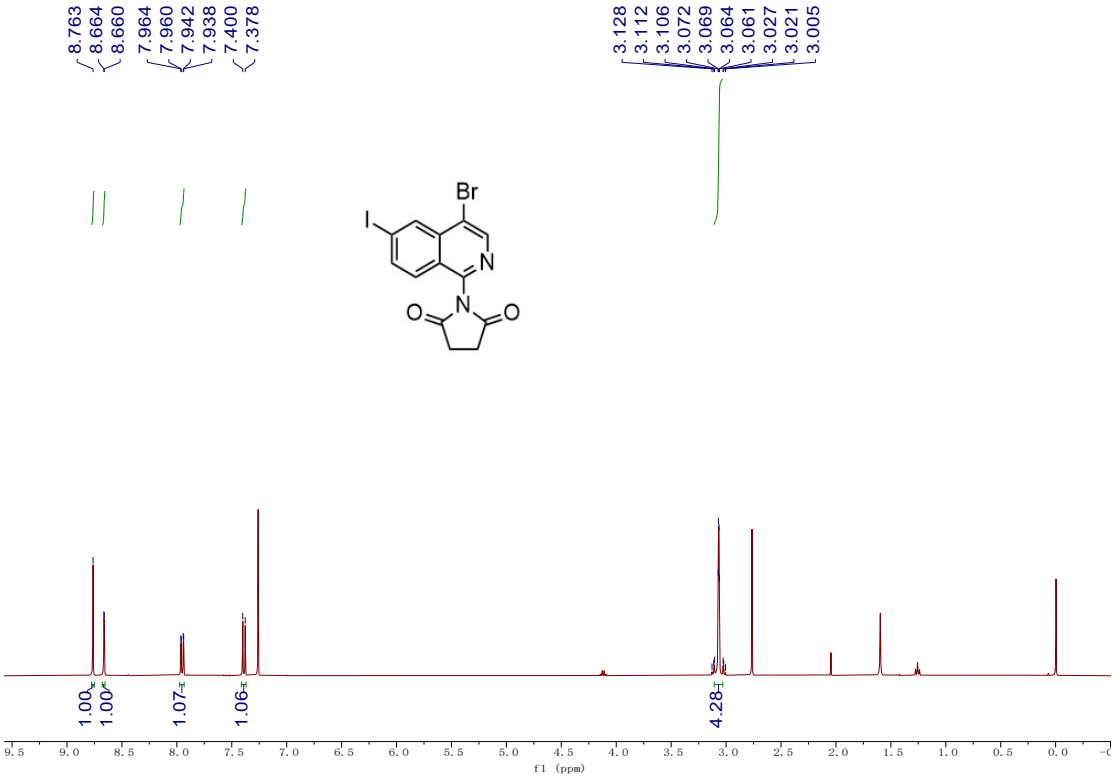
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1487 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-13 H: 9-9 N: 0-100 O: 0-100 Na: 0-8 Cl: 1-6 Br: 1-4

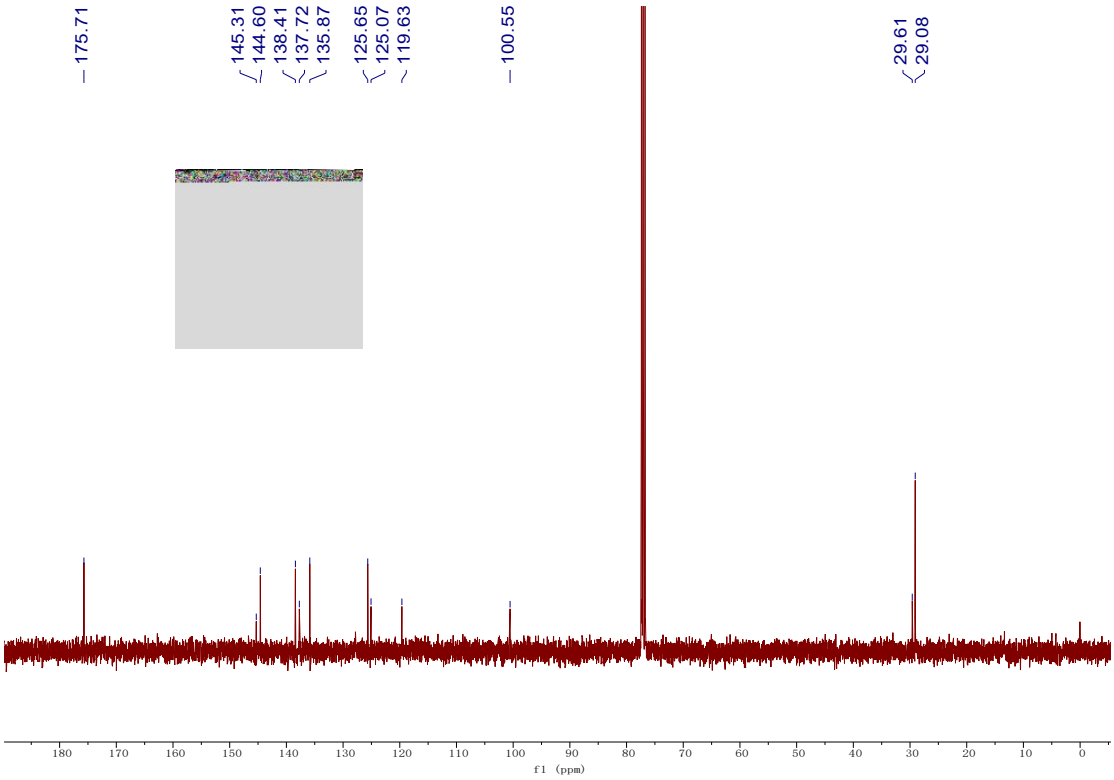


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
338.9542	338.9536	0.6	1.8	9.5	886.2	n/a	n/a	C13 H9 N2 O2 Cl Br

^{1}H NMR spectra of **5d** (400 MHz, $CDCl_3$)



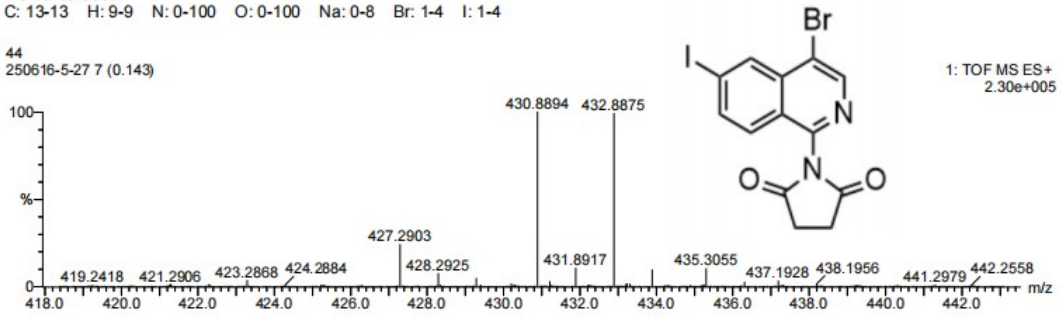
^{13}C NMR spectra of **5d** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

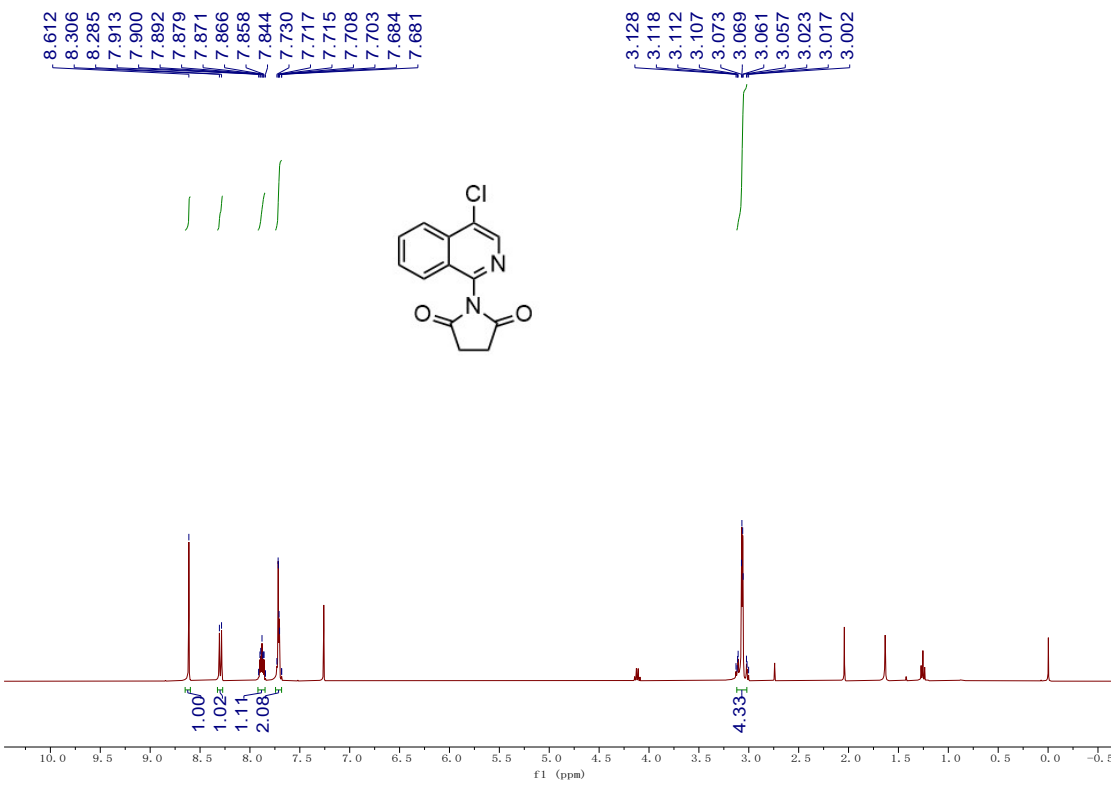
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
743 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-13 H: 9-9 N: 0-100 O: 0-100 Na: 0-8 Br: 1-4 I: 1-4

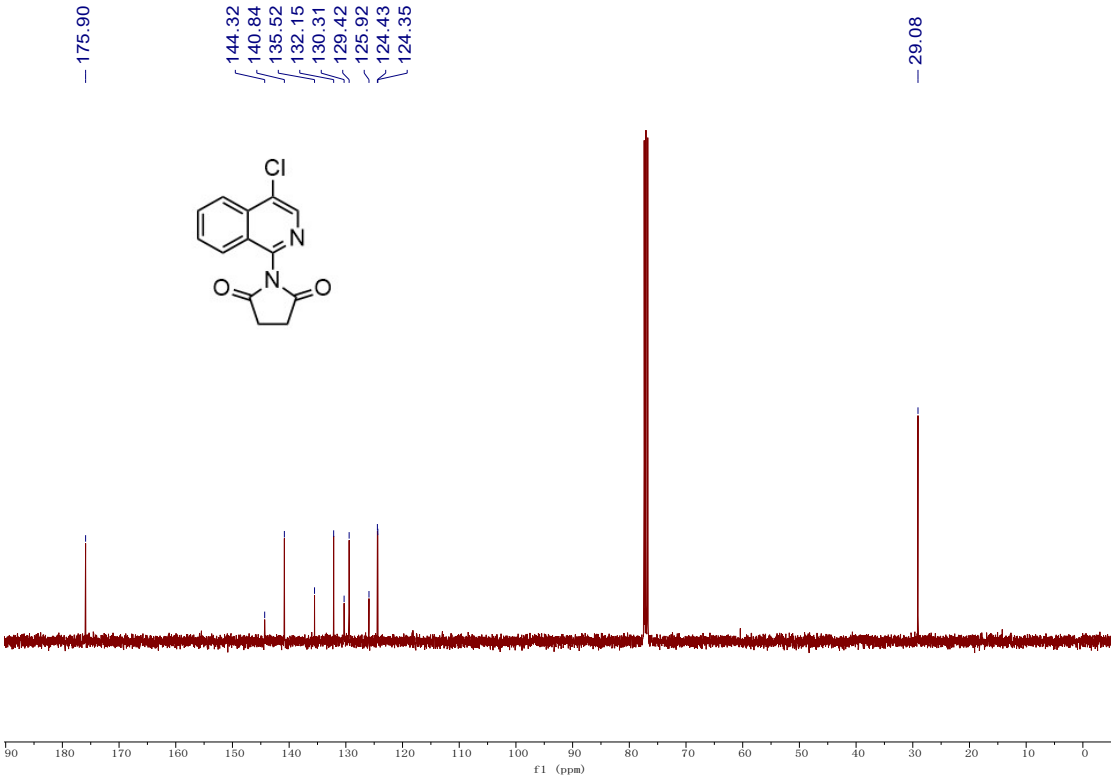


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
430.8894	430.8892	0.2	0.5	9.5	951.1	n/a	n/a	C13 H9 N2 O2 Br I

^{1}H NMR spectra of **5e** (400 MHz, $CDCl_3$)



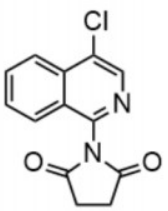
^{13}C NMR spectra of **5e** (101 MHz, $CDCl_3$)



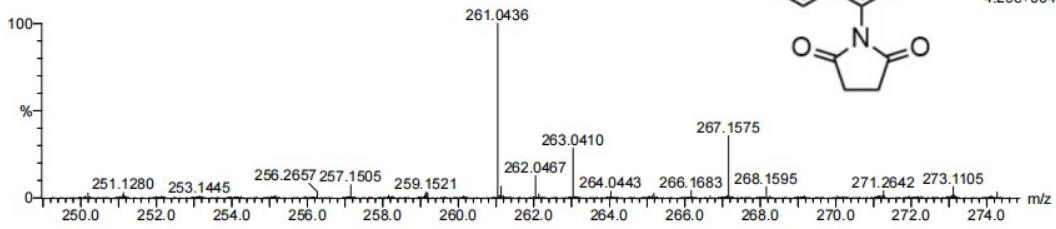
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1175 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-13 H: 10-10 N: 0-100 O: 0-100 Na: 0-8 Cl: 1-6
44
250616-5-24 12 (0.236)

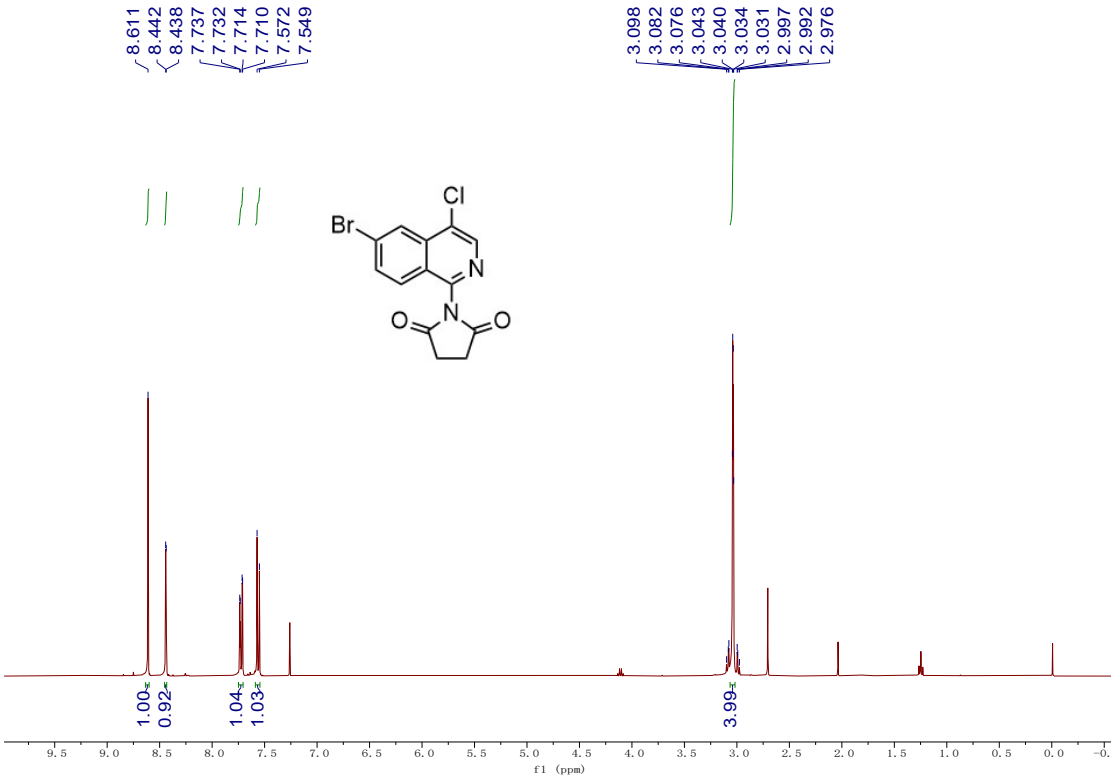


1: TOF MS ES+
4.29e+004

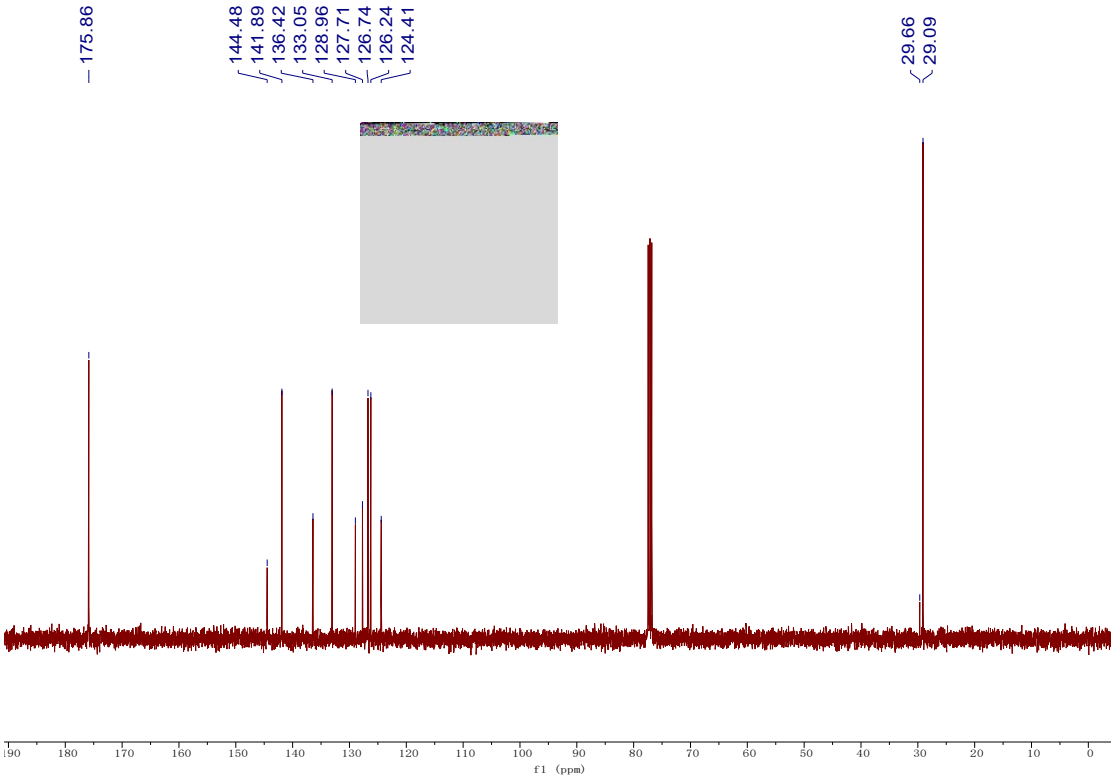


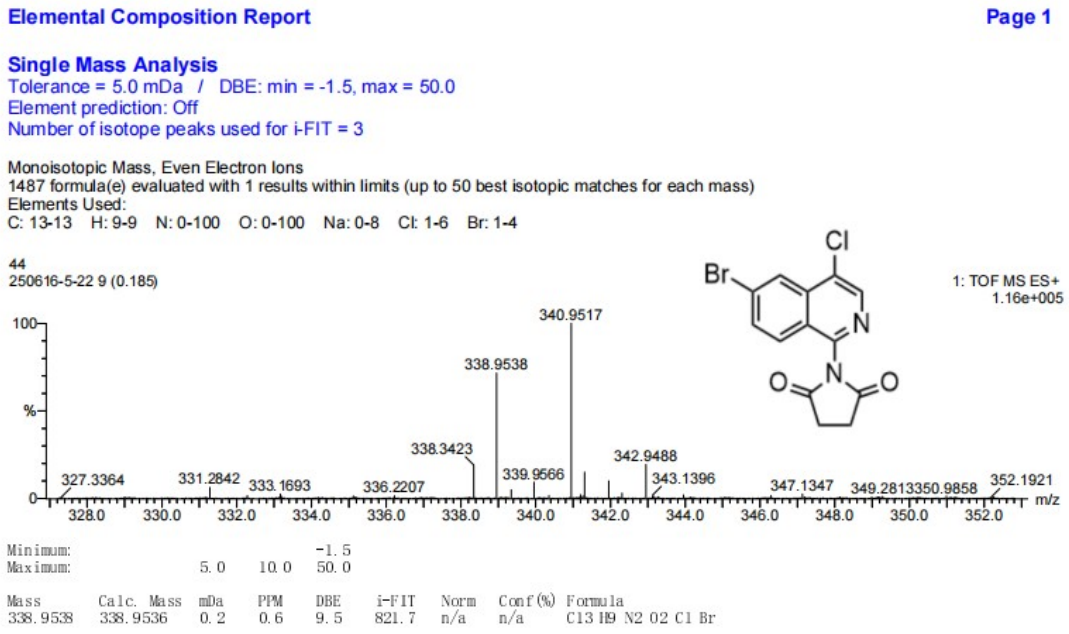
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
261.0436	261.0431	0.5	1.9	9.5	718.5	n/a	n/a	C13 H10 N2 O2 Cl

^{1}H NMR spectra of **5f** (400 MHz, $CDCl_3$)

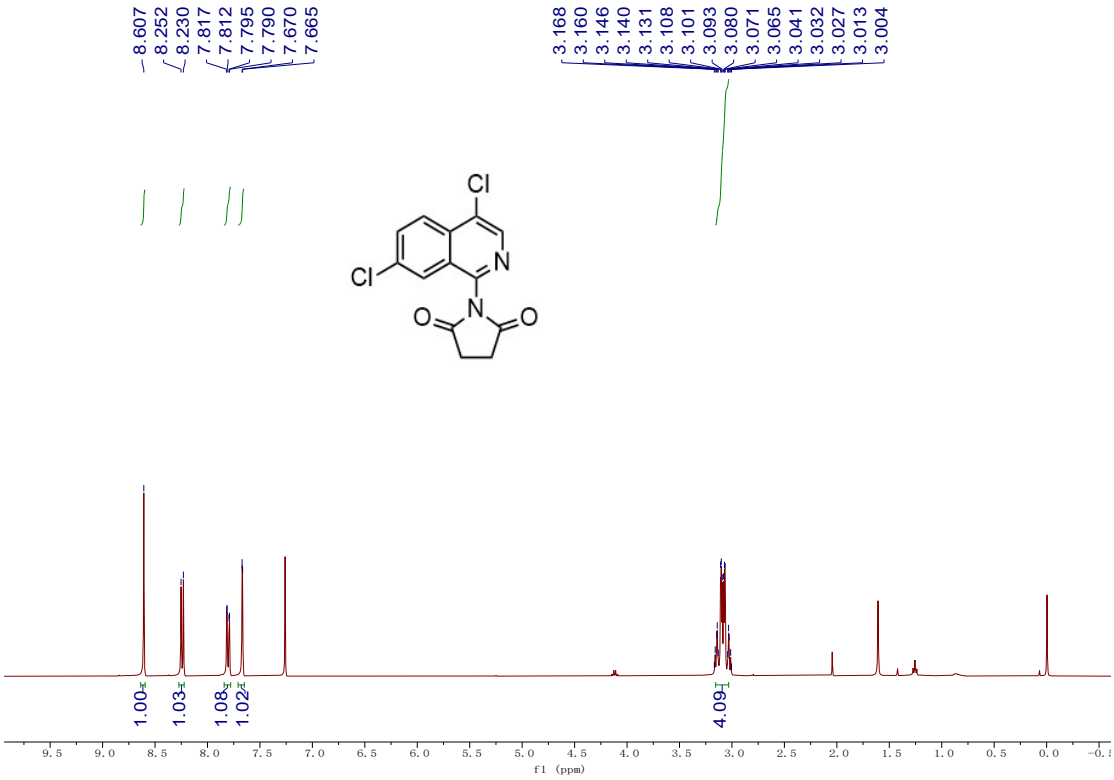


^{13}C NMR spectra of **5f** (101 MHz, $CDCl_3$)

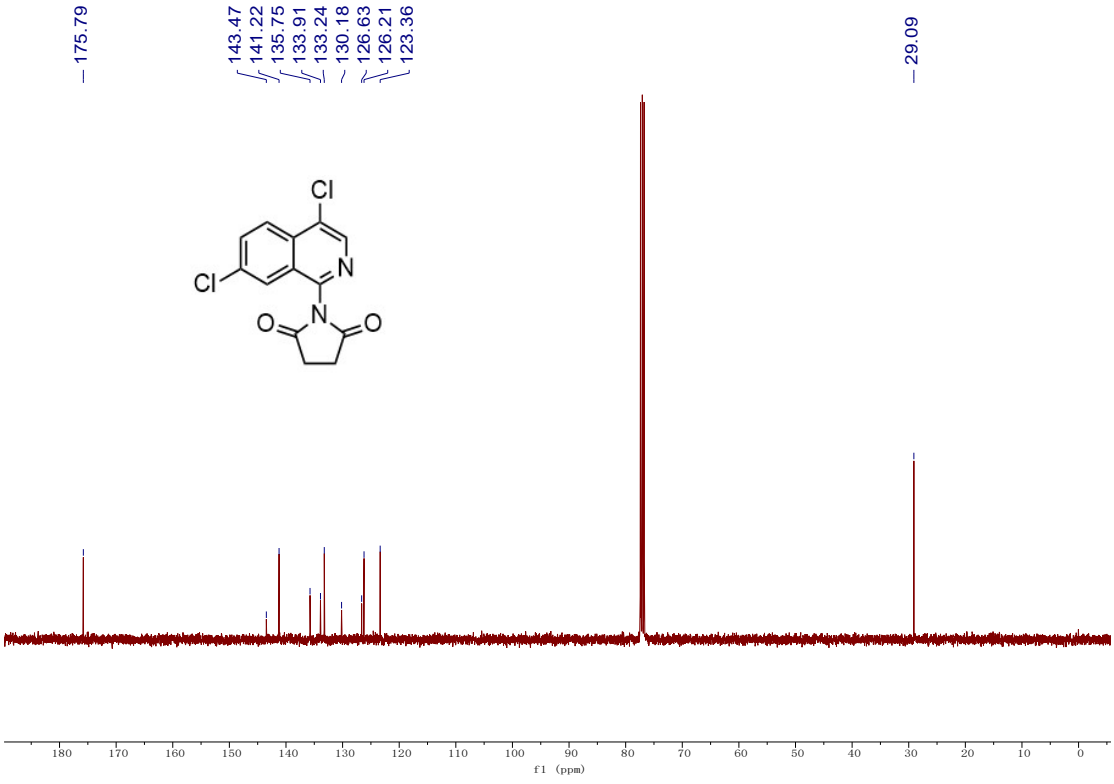




^{1}H NMR spectra of **5g** (400 MHz, $CDCl_3$)



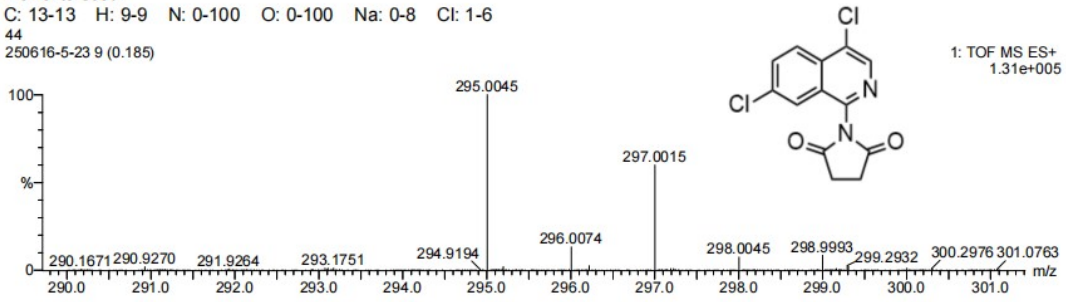
^{13}C NMR spectra of **5g** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

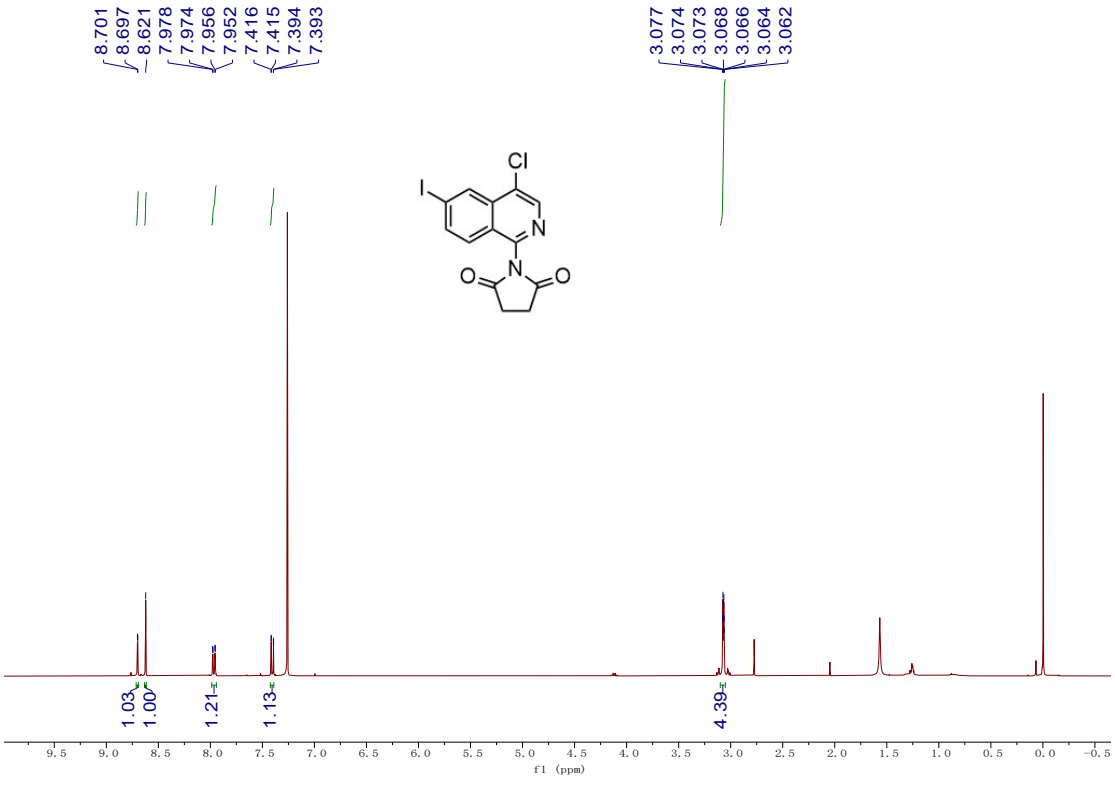
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1832 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-13 H: 9-9 N: 0-100 O: 0-100 Na: 0-8 Cl: 1-6
44
250616-5-23 9 (0.185)

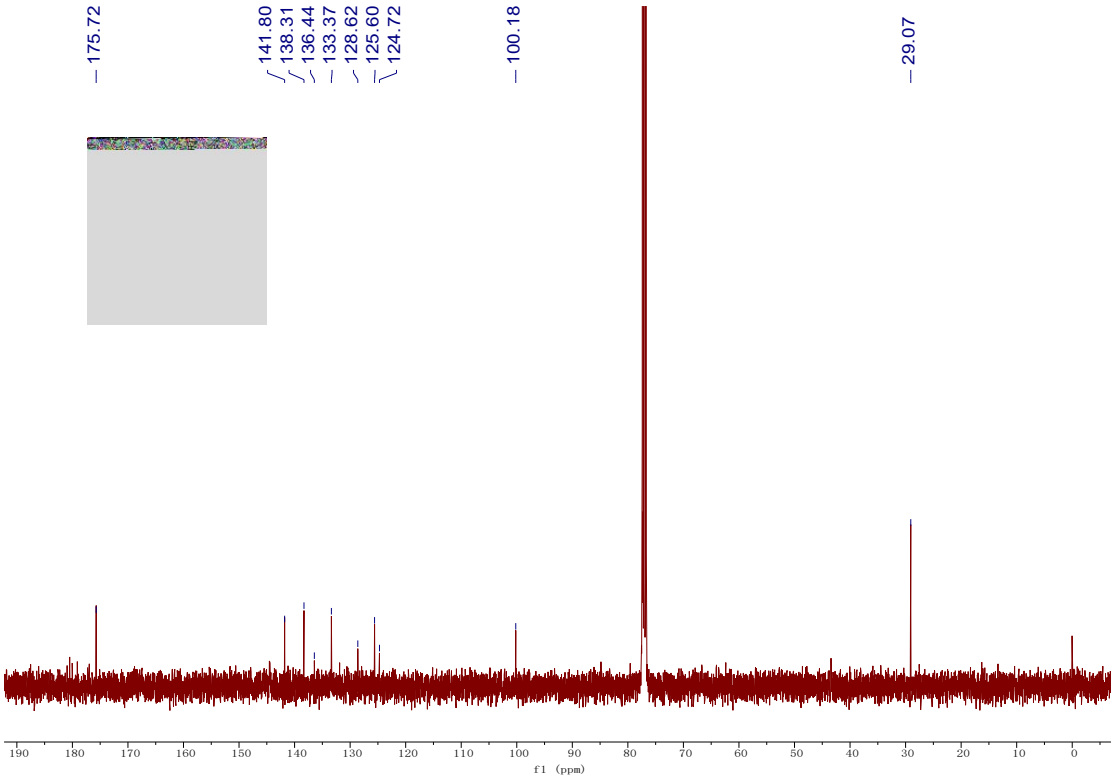


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
295.0045	295.0041	0.4	1.4	9.5	853.1	n/a	n/a	C13 H9 N2 O2 Cl2

^{1}H NMR spectra of **5h** (400 MHz, $CDCl_3$)



^{13}C NMR spectra of **5h** (101 MHz, $CDCl_3$)

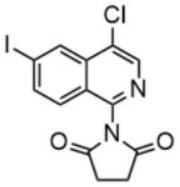


Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1249 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-13 H: 9-9 N: 0-100 O: 0-100 Na: 0-8 Cl: 1-6 I: 1-4

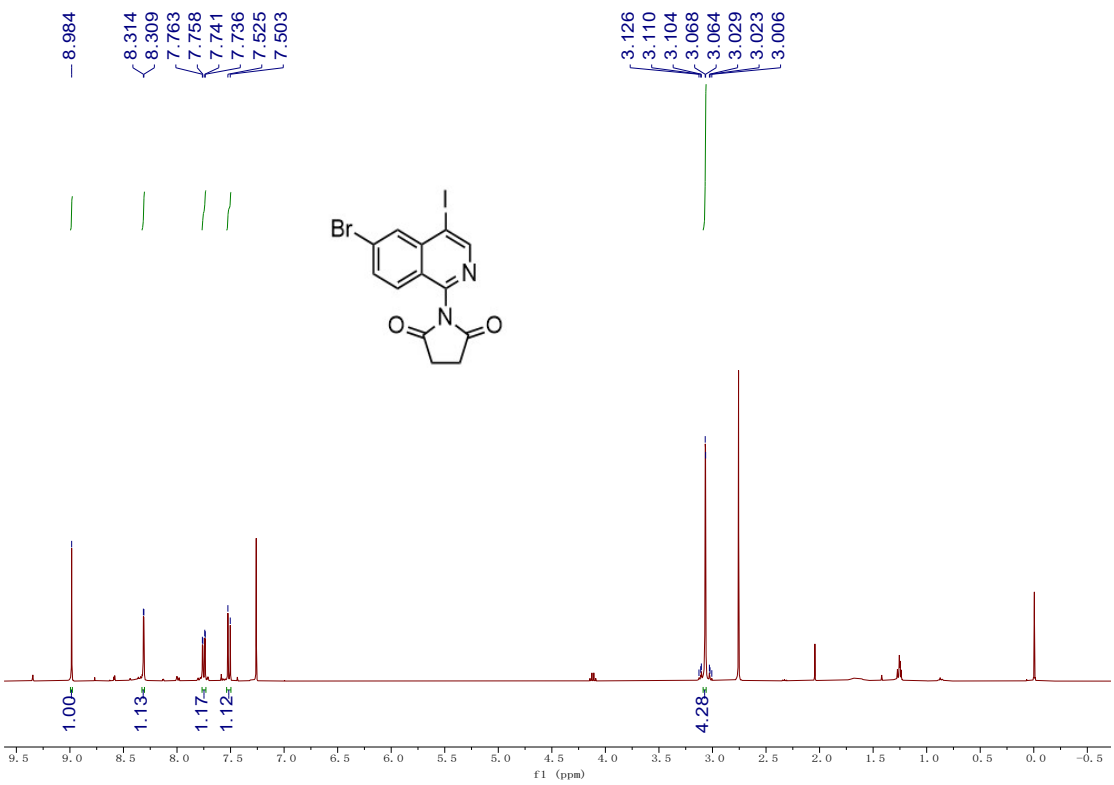
44
250616-5-28 10 (0.202)



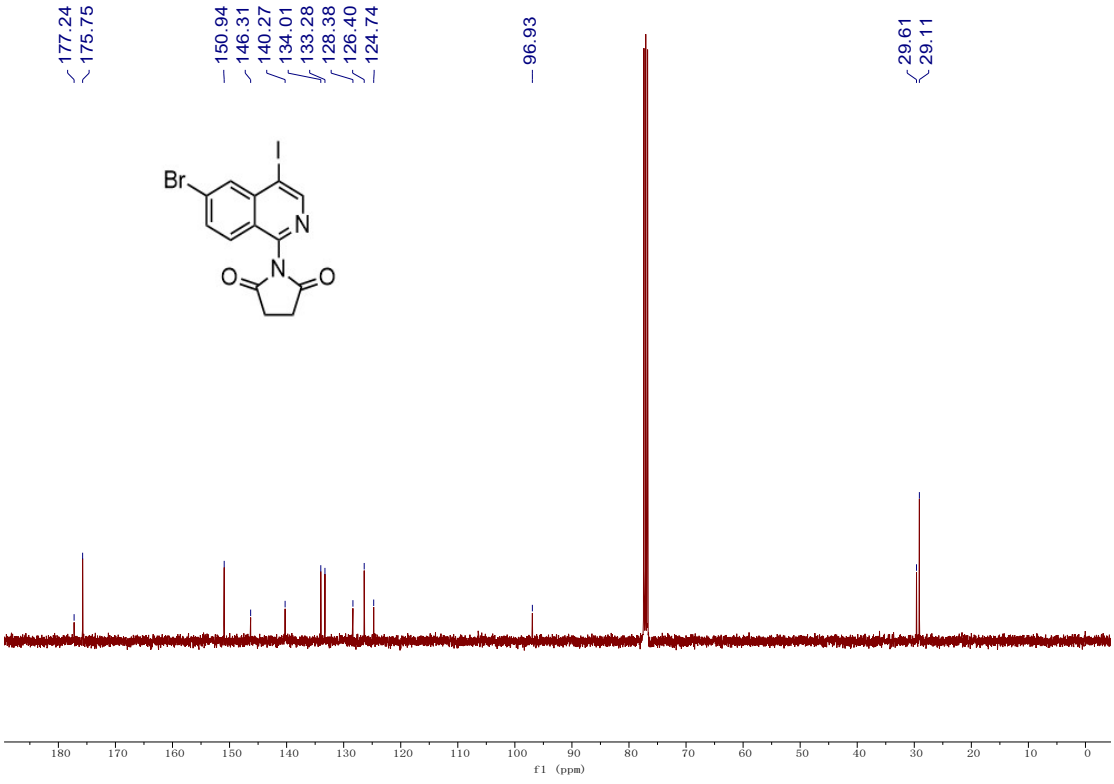
1: TOF MS ES+
3.55e+004

Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
386.9395	386.9397	-0.2	-0.5	9.5	761.8	n/a	n/a	C13 H9 N2 O2 Cl I

^{1}H NMR spectra of **5i** (400 MHz, $CDCl_3$)



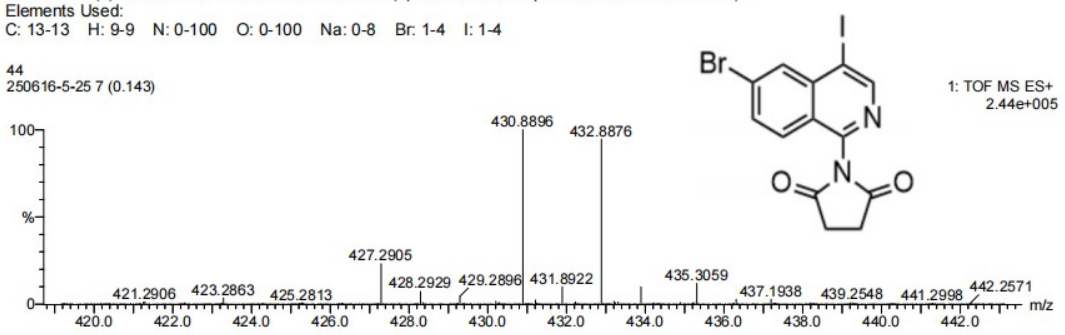
^{13}C NMR spectra of **5i** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

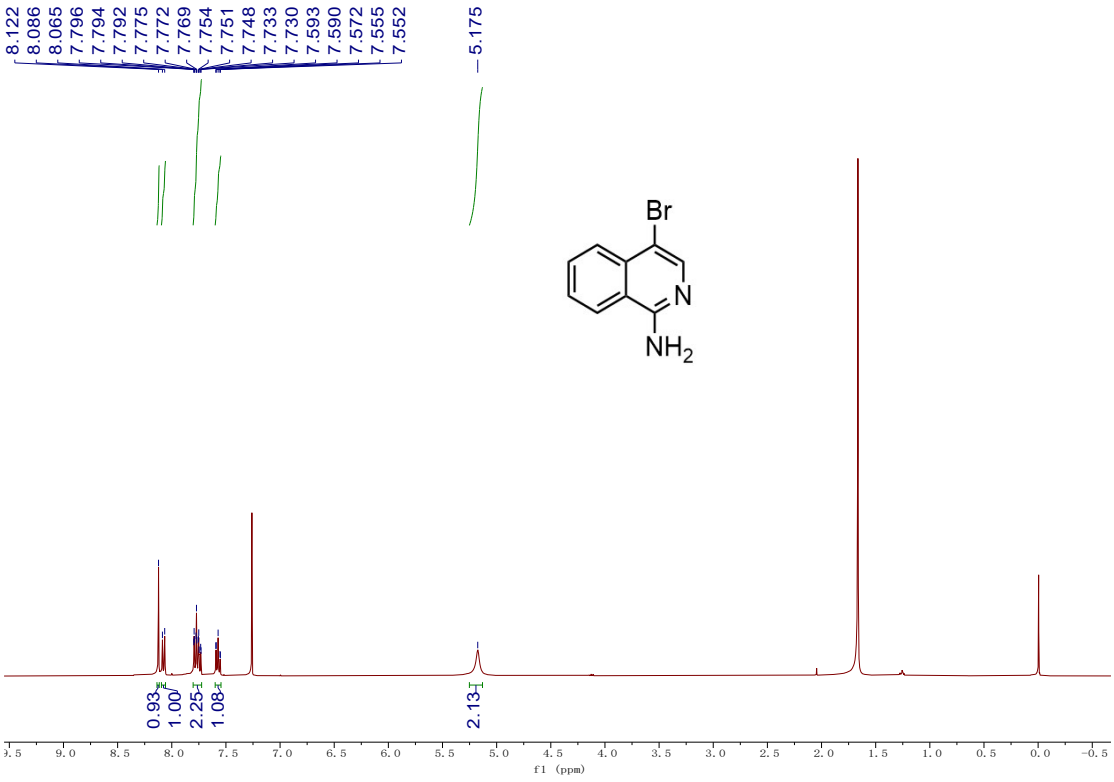
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
743 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 13-13 H: 9-9 N: 0-100 O: 0-100 Na: 0-8 Br: 1-4 I: 1-4

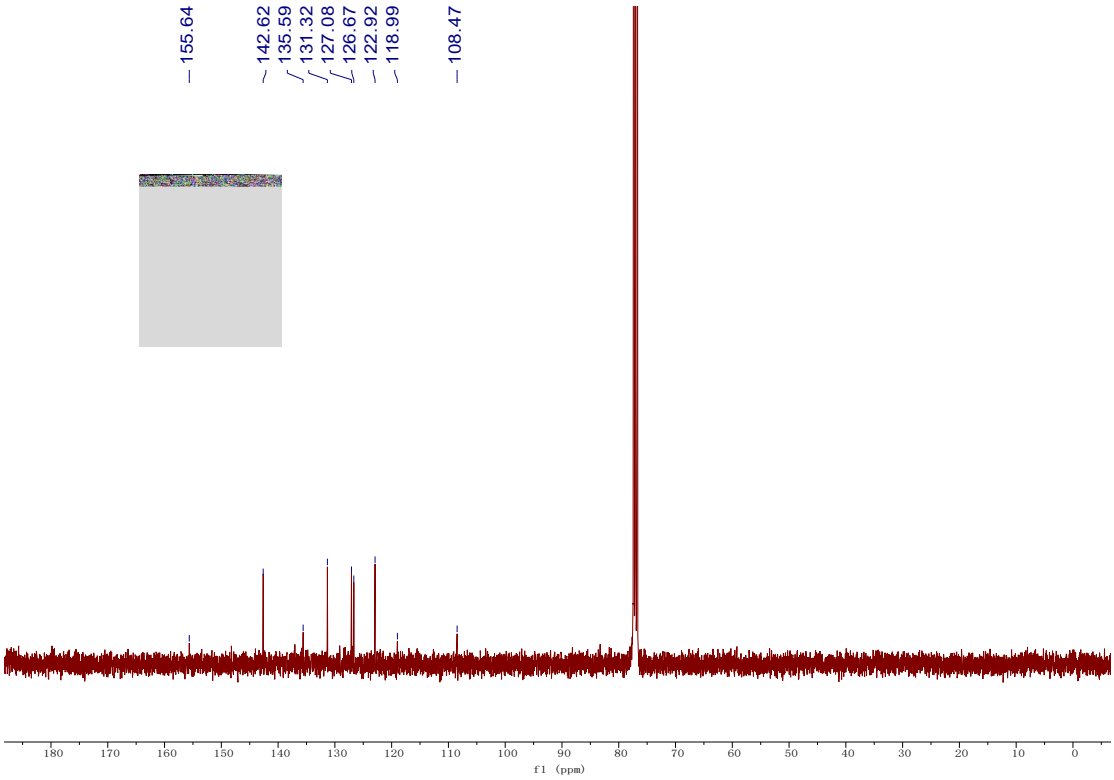


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
430.8896	430.8892	0.4	0.9	9.5	911.0	n/a	n/a	C13 H9 N2 O2 Br I

^{1}H NMR spectra of **6** (400 MHz, $CDCl_3$)



^{13}C NMR spectra of **6** (101 MHz, $CDCl_3$)

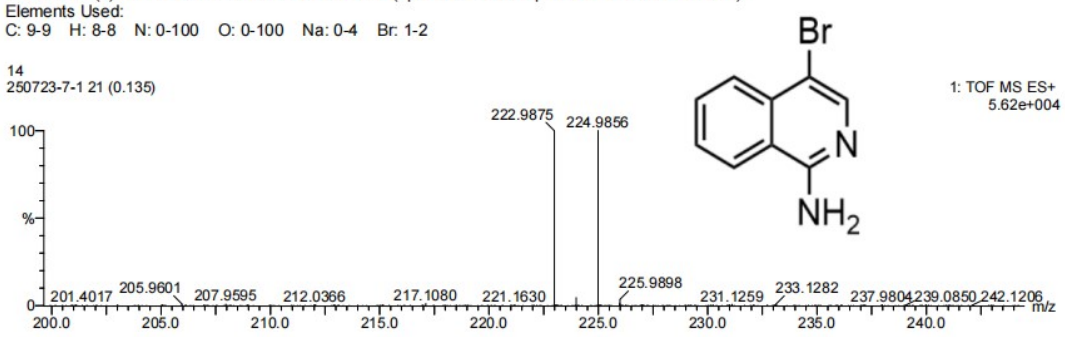


Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

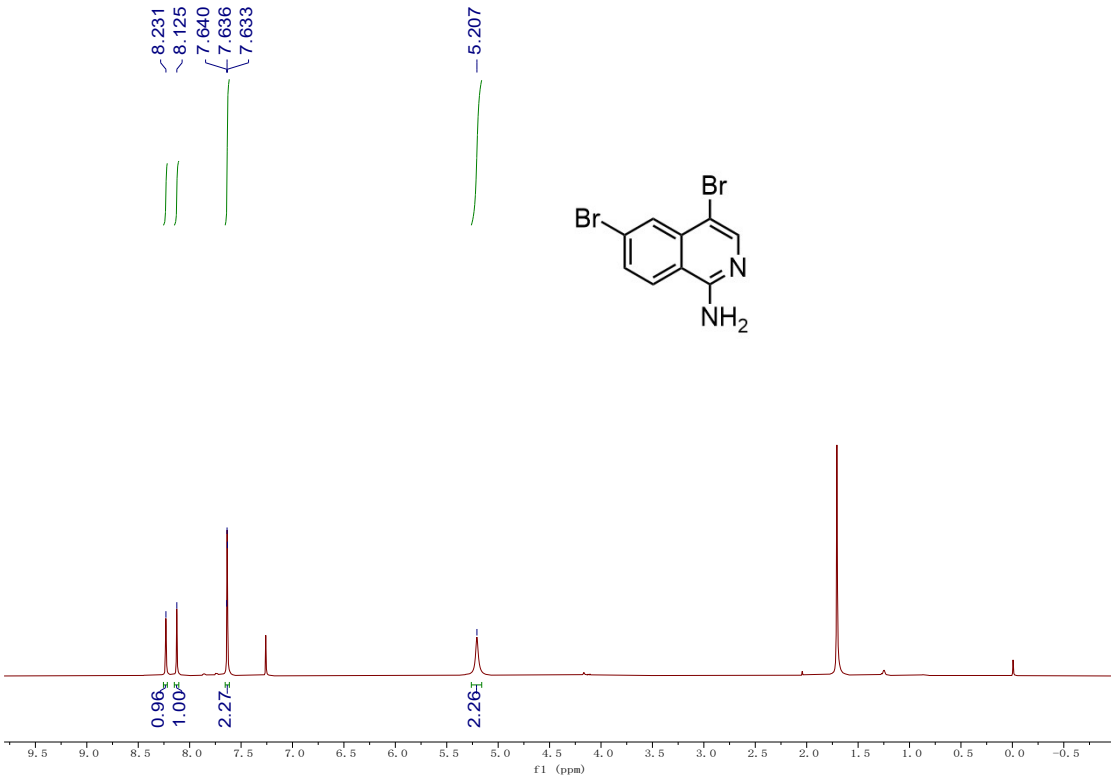
Monoisotopic Mass, Even Electron Ions
173 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 9-9 H: 8-8 N: 0-100 O: 0-100 Na: 0-4 Br: 1-2

14
250723-7-1 21 (0.135)

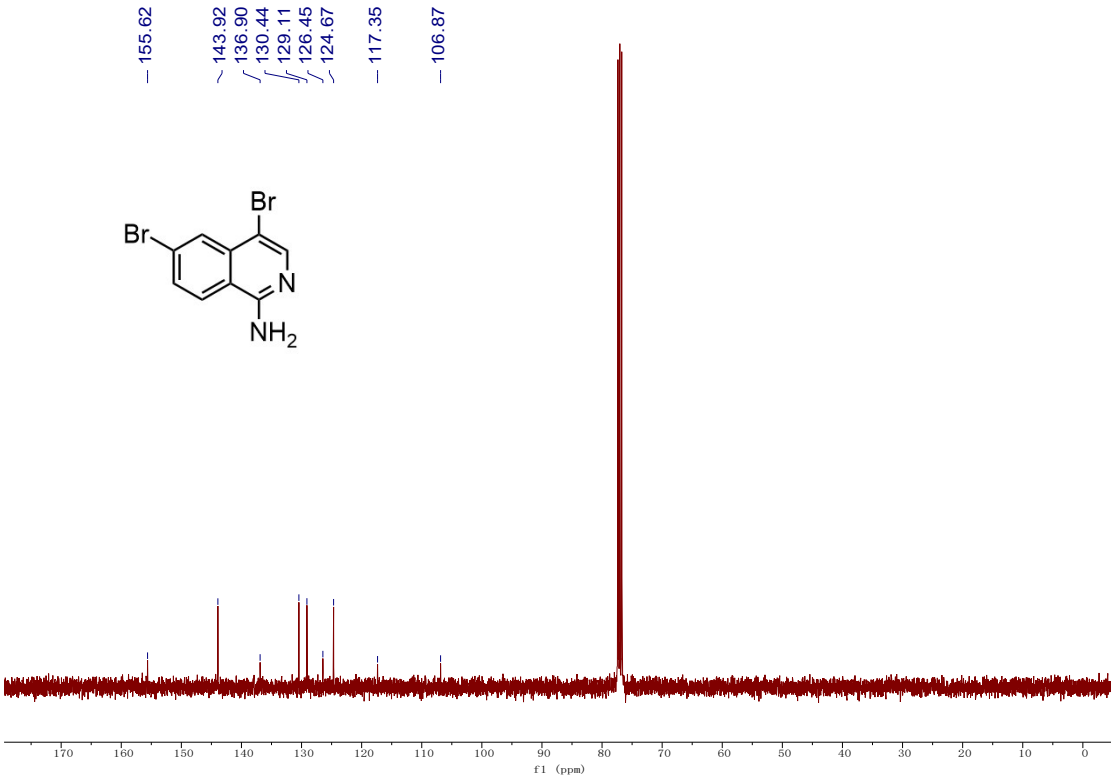


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
222.9875	222.9871	0.4	1.8	6.5	276.1	n/a	n/a	C9 H8 N2 Br

^{1}H NMR spectra of **7** (400 MHz, $CDCl_3$)



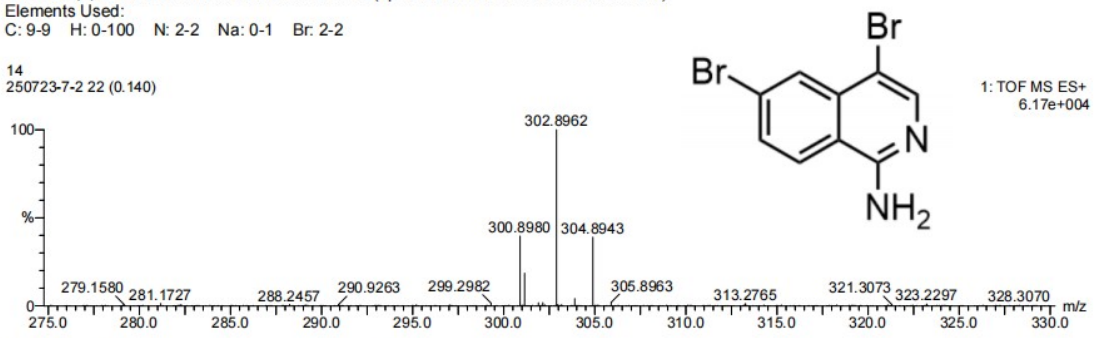
^{13}C NMR spectra of **7** (101 MHz, $CDCl_3$)



Elemental Composition Report **Page 1**

Multiple Mass Analysis: 2 mass(es) processed
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

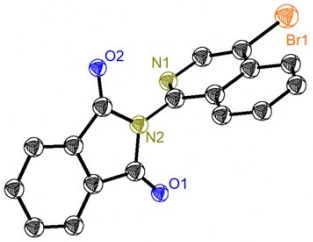
Monoisotopic Mass, Even Electron Ions
4 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 9-9 H: 0-100 N: 2-2 Na: 0-1 Br: 2-2



Minimum:	39.00				-1.5				
Maximum:	100.00		5.0	5.0	50.0				
Mass	RA	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
300.8980	39.58	300.8976	0.4	1.3	6.5	417.2	n/a	n/a	C9 H7 N2 Br2
302.8962	100.00	---							

Part 3. Crystal Data and Structural Refinement of Compound 3a (2454358)

Crystal Data and Structural Refinement of Compounds **3a**



X-ray of 3a

Sample preparation: The product **3a** was separated by silica gel column chromatography with the eluent of petroleum ether/ethyl acetate = 10:1, then solution was slowly volatilized into crystal **3a**.

Experimental

Single crystals of $C_{17}H_9BrN_2O_2$ [**3a**] were [The crystals were growth by means of evaporation of a mixture of n-hexane and toluene at RT.]. A suitable crystal was selected and [] on a XtaLAB Synergy, Single source at offset/far, HyPix diffractometer. The crystal was kept at 298(2) K during data collection. Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program using Intrinsic Phasing and refined with the SHELXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [**3a**]

Crystal Data for $C_{17}H_9BrN_2O_2$ (M =353.17 g/mol): monoclinic, space group C2/c (no. 15), a = 18.9318(14) Å, b = 11.3071(6) Å, c = 15.6112(10) Å, β = 121.523(7)°, V = 2848.6(4) Å^3, Z = 8, T = 298(2) K, μ(Mo Kα) = 2.894 mm^{-1}, *Dcalc* = 1.647 g/cm^3, 10889 reflections measured (4.398° ≤ 2Θ ≤ 59.36°), 3529 unique (R_{int} = 0.0411, R_{sigma} = 0.0477) which were used in all calculations. The final R_1 was 0.0522 (I > 2σ(I)) and wR_2 was 0.1200 (all data).

Refinement model description

Number of restraints - 19, number of constraints 0.

Details:

1. Fixed Uiso

At 1.2 times of:

All C(H) groups

2. Restrained distances

O1A-C10 ≈ O1-C10

with sigma of 0.02

3. Uiso/Uaniso restraints and constraints

C10 ≈ O1 ≈ O1A: within 2A with sigma of 0.01 and sigma for terminal atoms of 0.02 within 2A

4. Others

Sof(O1A)=1-FVAR(1)

Sof(O1)=FVAR(1)

5.a Aromatic/amide H refined with riding coordinates:

C1(H1), C7(H7), C4(H4), C15(H15), C12(H12), C5(H5), C13(H13), C6(H6), C14(H14)

This report has been created with Olex2, compiled on 2022.04.07 svn.rca3783a0 for OlexSys. Please let us know if there are any errors or if you would like to have additional features.

Table 1 Crystal data and structure refinement for 3a.

Identification code	**3a**
Empirical formula	$C_{17}H_9BrN_2O_2$
Formula weight	353.17
Temperature/K	298(2)
Crystal system	monoclinic
Space group	C2/c
a/Å	18.9318(14)
b/Å	11.3071(6)
c/Å	15.6112(10)
α/°	90
β/°	121.523(7)
γ/°	90
Volume/Å^3	2848.6(4)
Z	8
ρ_{calc}g/cm^3	1.647
μ/mm^{-1}	2.894

F(000)	1408.0
Crystal size/mm^3	0.15 × 0.13 × 0.12
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.398 to 59.36
Index ranges	-24 ≤ h ≤ 25, -14 ≤ k ≤ 15, -11 ≤ l ≤ 20
Reflections collected	10889
Independent reflections	3529 [R_{int} = 0.0411, R_{sigma} = 0.0477]
Data/restraints/parameters	3529/19/209
Goodness-of-fit on F^2	1.035
Final R indexes [I>=2σ (I)]	R_1 = 0.0522, wR_2 = 0.1120
Final R indexes [all data]	R_1 = 0.0803, wR_2 = 0.1200
Largest diff. peak/hole / e $Å^{-3}$	0.38/-0.63