Electronic Supplementary Material (ESI) for ChemComm.

Synergistic Effect of Self-assembling and Visible-light Induced the Oxidative C-H Acylation of N-heterocyclic Aromatic Compounds with Aldehydes

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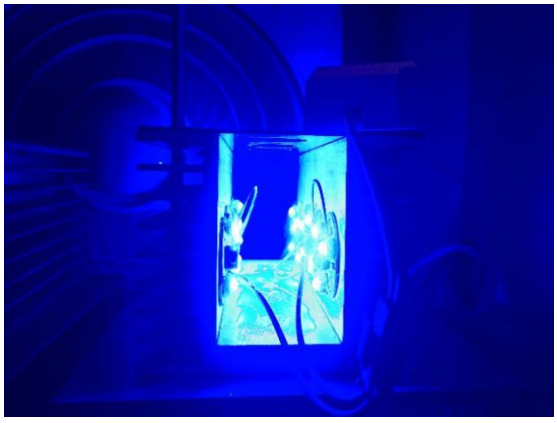
1. General Information

All manipulations were carried out by standard schlenk techniques. Unless otherwise noted, analytical grade solvents and commercially available reagents were used to conduct the reactions. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (boiling point is between 60-90 ^{o}C). Gradient flash chromatography was conducted eluting with a continuous gradient using petroleum ether and ethyl acetate. The known compounds were characterized by ^{1}H NMR and ^{13}C NMR. GC-MS spectra were recorded on a Varian GC-MS 3900-2100T. The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Advance III 400 MHz NMR spectrometer with tetramethylsilane as an internal standard. The chemical shifts (δ) were given in part per million relative to internal tetramethyl silane (TMS, 0 ppm for ^{1}H), $CDCl_3$ (77.3 ppm for ^{13}C). The source of the blue LEDs is common LED lights. The power of each light is 3W. There is 3.0 cm distance between the reactor and LEDs. This reaction could be well-performed using a round bottle (25 mL). Below we will add two pictures of our instrument.



2. General Procedures for Oxidative Coupling between N-heterocycles and Aldehydes

In a dried schlenk tube, isoquinoline **1** (0.5 mmol), aldehydes (2.5 mmol), TBHP in decane (1.0 mmol) and TFA (1.5 eq.) were stirred in 1.0 mL ethyl acetate for 24 hours at room temperature under an nitrogen atmosphere irradiated by blue LEDs. After completion of the reaction, as indicated by TLC and GC-MS, the mixture was diluted by ethyl acetate. The pure product was obtained by flash column chromatography on silica gel using petroleum ether and ethyl acetate.

3. Mechanism Studies

3.1 Radical-inhibiting experiment

In a dried schlenk tube, isoquinoline 1 (0.5 mmol), aldehydes (2.5 mmol), TBHP in decane (1.0 mmol), TFA (1.5 eq.) and 2,2,6,6-tetramethlpiperidinooxy (TEMPO, 0.6 mmol) or BHT (0.6 mmol) were stirred in 1.0 mL ethyl acetate for 24 hours at room temperature under an nitrogen atmosphere irradiated by blue LEDs. After completion of the reaction, the mixture was detected by GC-MS.

3.2 Trapping of Radical Intermediates with Radical Scavenger 1,1-Diphenylethylene

In a dried schlenk tube, isoquinoline 1 (0.5 mmol), aldehydes (2.5 mmol), TBHP in decane (1.0 mmol), TFA (1.5 eq.) and 1,1-Diphenylethylene (0.6 mmol) were stirred in 1.0 mL ethyl acetate for 24 hours at room temperature under an nitrogen atmosphere irradiated by blue LEDs. After completion of the reaction, the mixture was detected by GC-MS.

3.3 . Electron paramagnetic resonance (EPR) study

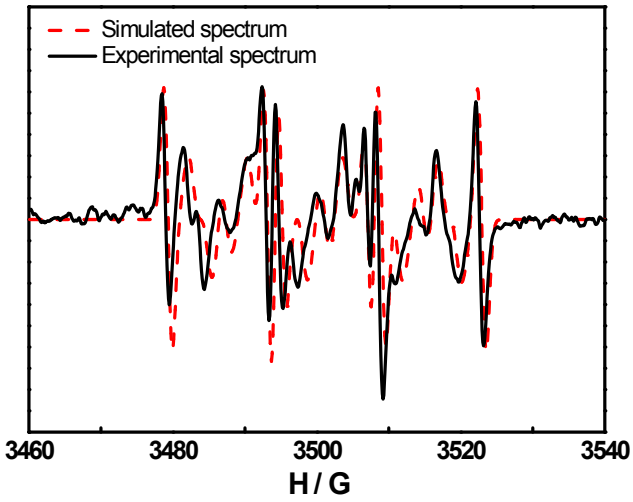


Figure S1. Experimental (full line) and simulated (dashed line) EPR spectrum of the trapped radical intermediates.

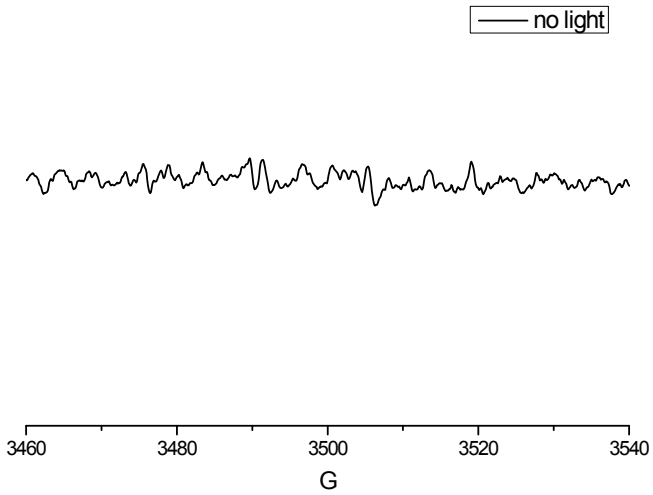


Figure S2. electron paramagnetic resonance (EPR) study.

3.4. Controle experiment

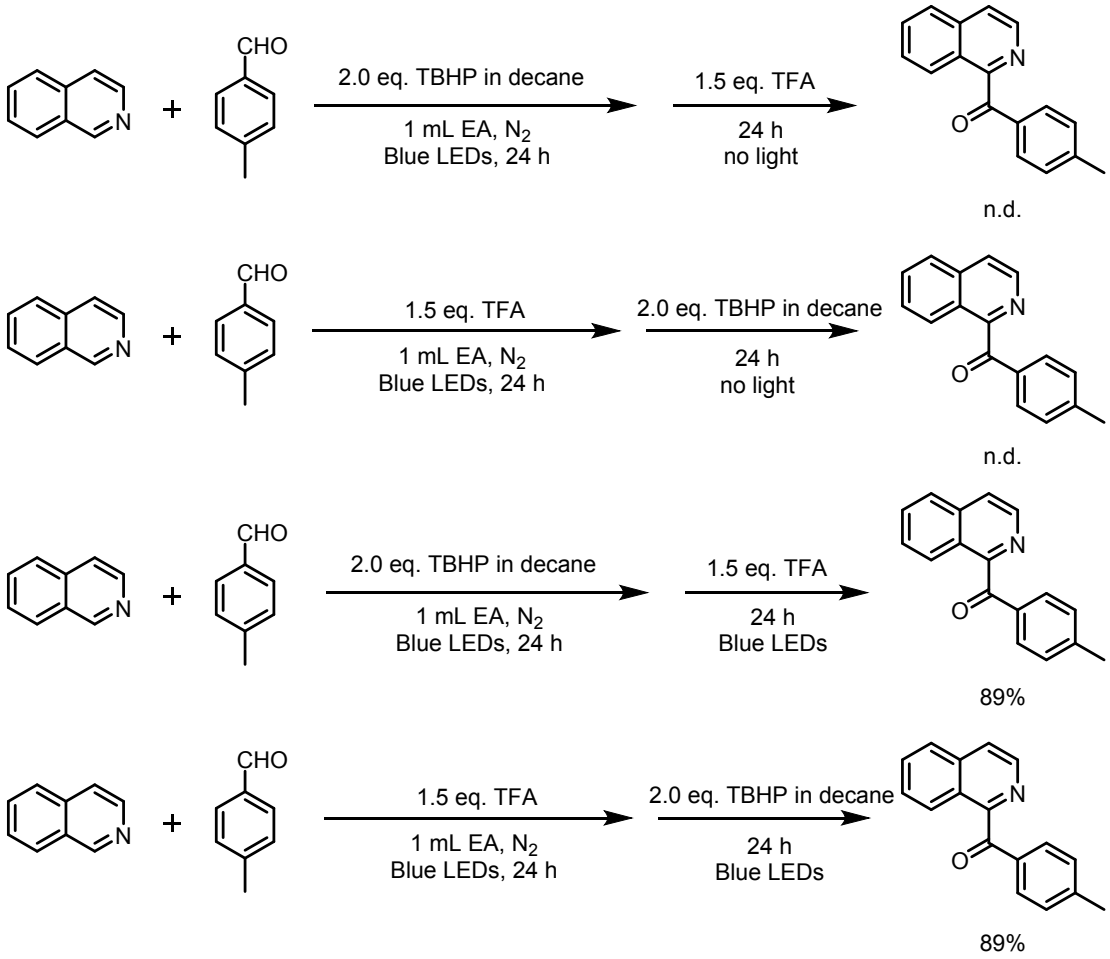


Figure S3. Controlled experiment

3.5. NMR study

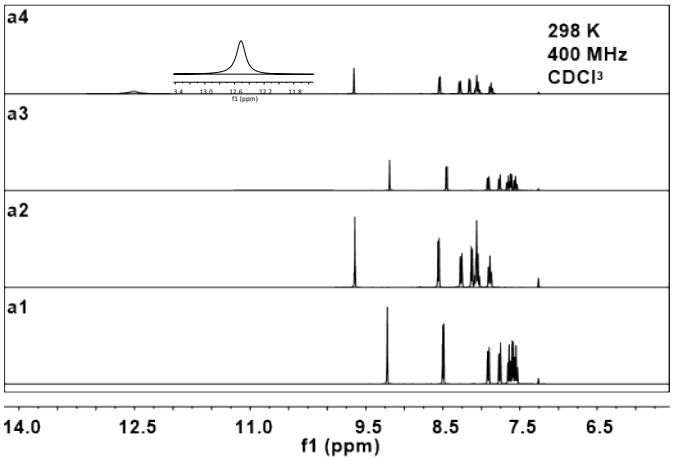


Figure S4. 1H NMR spectra of (a1) isoquinoline, (a2) isoquinoline /TFA mixture in 1 : 1 ratio, (a3) isoquinoline /TBHP mixture in 1 : 1 ratio, and (a4) isoquinoline /TFA/ tBuOOH mixture in 1 : 1 : 1 ratio in CDCl3.

3.6. UV-Visible study

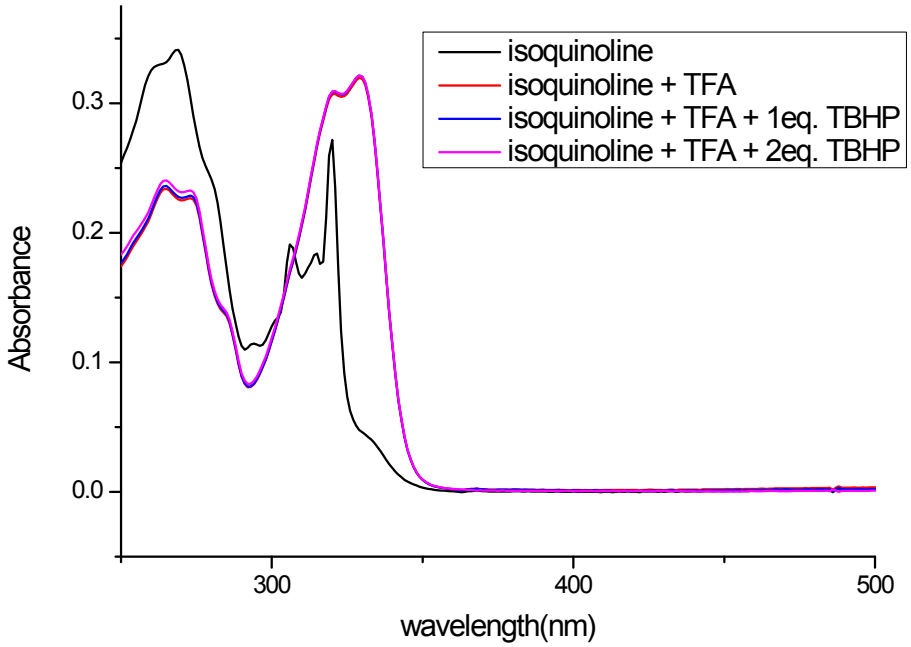
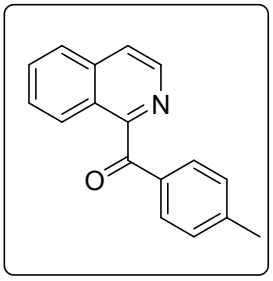
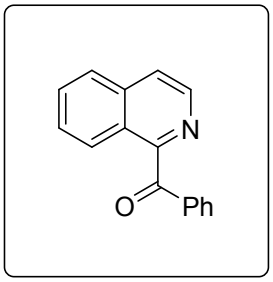


Figure S5. UV-vis absorption spectra.

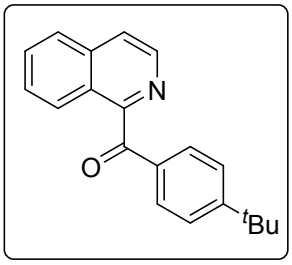
4. Characterization of Products



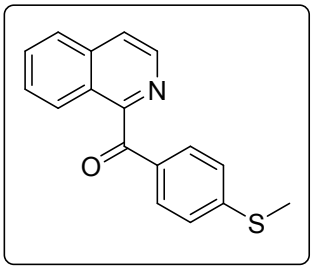
Isoquinolin-1-yl(p-tolyl)methanone [1] (**3a**): 109.9 mg (yield: 89 %). ^{1}H NMR (400 MHz, DMSO) δ 8.62 (d, *J* = 5.6 Hz, 1H), 8.14 (d, *J* = 8.4 Hz, 1H), 8.08 - 8.07 (m , 1H), 8.01 - 7.99 (m, 1H), 7.89 - 7.85 (m, 1H), 7.75 - 7.69 (m, 3H), 7.37 (d, *J* = 8 Hz, 2H), 2.41 (s, 3H). ^{13}C NMR (101 MHz, DMSO) δ 194.53, 156.88, 145.26, 141.67, 136.58, 134.05, 131.50, 130.84, 129.82, 129.08, 127.86, 125.84, 125.71, 122.94, 21.79.



Isoquinolin-1-yl(phenyl)methanone [1] (**3b**): 103.7 mg (yield: 89 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (d, *J* = 6 Hz, 1H), 8.22 (d, *J* = 8.4 Hz, 1H), 7.97 - 7.90 (m, 3H), 7.81 (d, *J* = 5.6 Hz, 1H), 7.75 - 7.71 (m, 1H), 7.63 - 7.58 (m, 2H), 7.49 - 7.45 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.81, 156.42, 141.17, 136.70, 136.61, 133.74, 130.78, 128.51, 128.37, 127.15, 126.41, 126.14, 122.67.

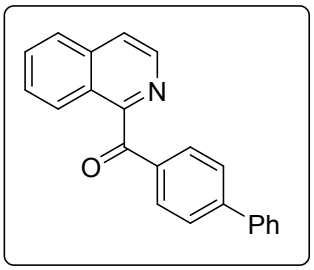


(4-(Tert-butyl)phenyl)(isoquinolin-1-yl)methanone (**3c**): 130.1 mg (yield: 90 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 (d, *J* = 5.6 Hz, 1H), 8.22 (d, *J* = 8.8 Hz, 1H), 7.93 – 7.88 (m , 3H), 7.81 (d, *J* = 5.6 Hz, 1H), 7.74 (t, *J* = 15.2 Hz, 1H), 7.63 (t, *J* = 15.6 Hz, 1H), 7.50 (d, *J* = 8.4 Hz, 2H), 1.34 (s, 9H). ^{13}C NMR (101 MHz, DMSO) δ 194.47, 157.59, 156.77, 141.18, 136.70, 133.97, 130.76, 128.27, 127.08, 126.42, 126.28, 125.55, 122.49, 35.26, 31.08. HRMS: calc. for $[M+H]^+$ $C_{20}H_{20}NO$: 290.1539 found: 292.1539.

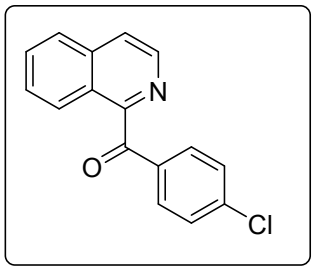


Isoquinolin-1-yl(4-(methylthio)phenyl)methanone (**3d**): 111.6 mg (yield: 80 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (d, *J* = 5,6 Hz, 1H), 8.19 (d, *J* = 8.4 Hz, 1H), 7.91 –

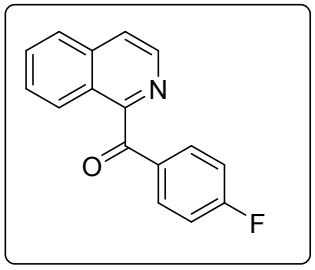
7.86 (m , 3H), 7.79 (d, *J* = 5.6 Hz, 1H), 7.72 (t, *J* = 14.8 Hz, 1H), 7.60 (t, *J* = 15.6 Hz, 1H), 7.26 (d, *J* = 8.4 Hz, 2H), 2.50 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.75, 156.58, 147.06, 141.19, 136.70, 132.82, 131.12, 130.73, 128.29, 127.11, 126.39, 126.21, 124.81, 122.53, 14.75. HRMS: calc. for $[M+H]^+$ $C_{17}H_{14}NOS$: 280.0791 found: 280.0796.



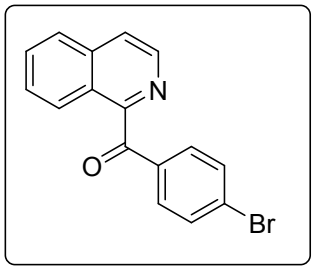
[1,1'-Biphenyl]-4-yl(isoquinolin-1-yl)methanone [1] (**3e**): 108.1 mg (yield: 70 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.61 (d, *J* = 5,6 Hz, 1H), 8.26 (d, *J* = 8.4 Hz, 1H), 8.03 (d , *J* = 8.4 Hz, 2H), 7.90 (d, *J* = 8.4 Hz, 1H), 7.80 (d, *J* = 5.6 Hz, 1H), 7.74 – 7.67 (m, 3H), 7.63 – 7.59 (m, 3H), 7.46 – 7.43 (m, 2H), 7.39 – 7.36 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.38, 156.44, 146.40, 141.22, 139.95, 136.77, 135.40, 131.43, 130.82, 129.02, 128.44, 128.37, 127.40, 127.23, 127.19, 126.49, 126.24, 122.76.



(4-Chlorophenyl)(isoquinolin-1-yl)methanone [3] (**3f**): 81.4 mg (yield: 61 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (d, *J* = 5,6 Hz, 1H), 8.25 (d, *J* = 8.0 Hz, 1H), 7.93 – 7.90 (m , 3H), 7.81 (d, *J* = 5.6 Hz, 1H), 7.76 – 7.72 (m, 1H), 7.65 – 7.61 (m, 1H), 7.46 – 7.42 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.42, 155.60, 141.08, 140.18, 136.78, 135.01, 132.21, 130.87, 128.81, 128.57, 127.20, 126.46, 126.06, 123.04.

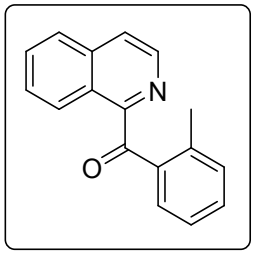


(4-Fluorophenyl)(isoquinolin-1-yl)methanone [1] (**3g**): 61.4 mg (yield: 49 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (d, *J* = 5,6 Hz, 1H), 8.23 (d, *J* = 9.2 Hz, 1H), 8.04 – 7.99 (m , 2H), 7.92 (d, *J* = 8.4 Hz, 1H), 7.81 (d, *J* = 5.6 Hz, 1H), 7.76 – 7.72 (m, 1H), 7.65 – 7.61 (m, 1H), 7.17 – 7.12 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.08, 166.12 (d, *J* = 257.0 Hz), 155.95, 141.09, 136.77, 133.56 (d, *J* = 9.6 Hz), 133.00 (d, *J* = 2.9 Hz), 130.83, 128.48, 127.17, 126.43, 126.09, 122.86, 115.69 (d, *J* = 22.1 Hz). These data are consistent with reported literature values.[4]

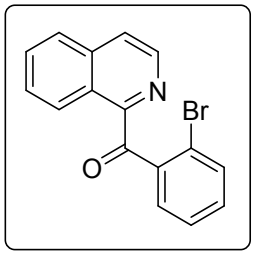


(4-Bromophenyl)(isoquinolin-1-yl)methanone [3] (**3h**): 113.3 mg (yield: 73 %). ^{1}H

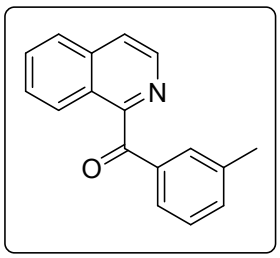
NMR (400 MHz, $CDCl_3$) δ 8.60 (d, *J* = 6 Hz, 1H), 8.26 (d, *J* = 9.2 Hz, 1H), 7.93 (d , *J* = 8.4 Hz, 1H), 7.85 – 7.82 (m, 3H), 7.77 – 7.73 (m, 1H), 7.66 – 7.60 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.62, 155.54, 141.09, 136.80, 135.45, 132.27, 131.79, 130.87, 129.04, 128.58, 127.19, 126.48, 126.07, 123.05.



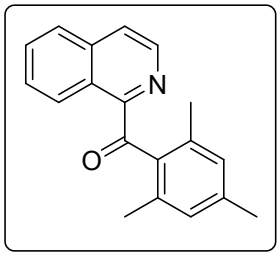
Isoquinolin-1-yl(o-tolyl)methanone [1] (**3i**): 104.9 mg (yield: 85 %). ^1H NMR (400 MHz, $CDCl_3$) δ 8.55 (d, *J* = 5.6 Hz, 1H), 8.41 (d, *J* = 8.4 Hz, 1H), 7.89 (d , *J* = 8.4 Hz, 1H), 7.77 – 7.70 (m, 2H), 7.66 – 7.62 (m, 1H), 7.43 – 7.39 (m, 2H), 7.31 (d, *J* = 7.6 Hz, 1H), 7.19 (t, *J* = 15.2 Hz, 1H), 2,53 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.76, 156.97, 141.37, 139.72, 137.31, 136.80, 132.12, 131.90, 131.82, 130.70, 128.60, 127.16, 126.41, 126.25, 125.51, 122.93, 21.37.



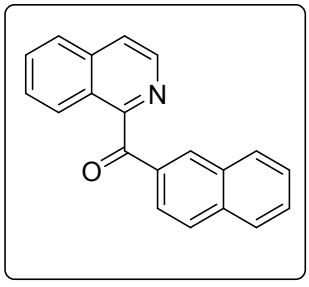
(2-Bromophenyl)(isoquinolin-1-yl)methanone [3] (**3j**): 65.2 mg (yield: 42 %). ^1H NMR (400 MHz, DMSO) δ 8.78 – 8.74 (m, 1H), 8.56 (d, *J* = 5.6 Hz, 1H), 8.17 – 8.13 (m , 2H), 7.93 – 7.85 (m, 2H), 7.71 – 7.69 (m, 1H), 7.65 – 7.63 (m, 1H), 7.58 – 7.48 (m, 2H), ^{13}C NMR (101 MHz, DMSO) δ 197.04, 153.15, 141.77, 141.71, 137.06, 133.22, 132.79, 131.43, 131.10, 130.11, 128.13, 128.05, 126.35, 126.05, 125.18, 119.88.



Isoquinolin-1-yl(m-tolyl)methanone [1] (**3k**): 111.1 mg (yield: 90 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (d, *J* = 5.6 Hz, 1H), 8.19 (d, *J* = 9.2 Hz, 1H), 7.91 (d , *J* = 8 Hz, 1H), 7.81 – 7.71 (m, 4H), 7.63 – 7.59 (m, 1H), 7.42 (d, J = 7.6 Hz, 1H), 7.35 (t, *J* = 15.2 Hz, 1H), 2,38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.11, 156.69, 141.20, 138.35, 136.68, 136.65, 134.61, 131.07, 130.75, 128.42, 128.32, 128.11, 127.12, 126.38, 126.18, 122.55, 21.37.

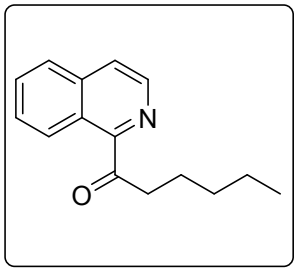


Isoquinolin-1-yl(mesityl)methanone (**3l**): 92.1 mg (yield: 67 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.01 – 9.04 (m, 1H), 8.54 (d, *J* = 5.6 Hz, 1H), 7.90 – 7.87 (m , 1H), 7.77 – 7.72 (m, 3H), 6.89 (s, 2H), 2.31 (s, 3H), 2.13 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.36, 154.24, 141.79, 139.14, 138.43, 137.11, 135.02, 130.51, 129.32, 128.71, 127.27, 126.54, 126.21, 124.29, 21.36, 19.93. HRMS: calc. for $[M+Na]^+$ $C_{19}H_{17}NNaO$: 298.1202 found: 298.1202.

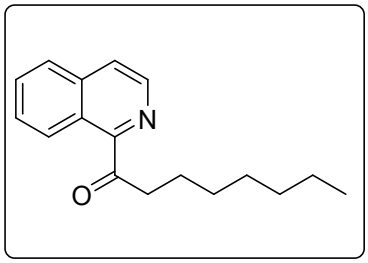


Isoquinolin-1-yl(naphthalen-2-yl)methanone [1] (**3m**): 86.3 mg (yield: 61 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 5.6 Hz, 1H), 8.34 (s, 1H), 8.22 – 8.24 (m, 1H), 8.16 – 8.13 (m, 1H), 7.93 – 7.89 (m, 2H), 7.86 – 7.80 (m, 3H), 7.73 – 7.69 (m, 1H), 7.60 –

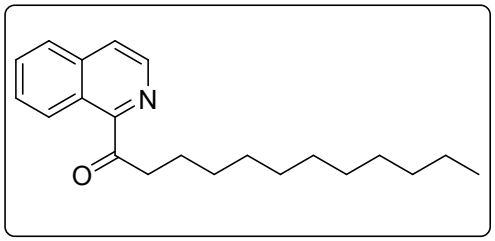
7.54 (m, 2H), 7.49 – 7.45 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.84, 156.72, 141.29, 136.76, 135.94, 134.00, 133.81, 132.38, 130.82, 129.89, 128.93, 128.52, 128.40, 127.84, 127.19, 126.78, 126.52, 126.23, 125.29, 122.68.



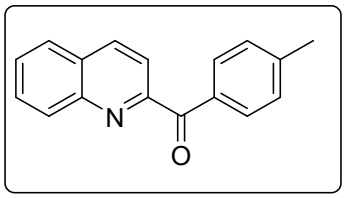
1-(Isoquinolin-1-yl)hexan-1-one (**3n**): 53.3 mg (yield: 47 %). ^{1}H NMR (400 MHz, DMSO) δ 8.69 – 8.66 (m, 1H), 8.63 (d, *J* = 5.6 Hz, 1H), 8.10 – 8.08 (m, 2H), 7.87 – 7.82 (m, 1H), 7.79 – 7.75 (m, 1H), 3.27 (t, *J* = 14.8 Hz, 2H), 1.71 – 1.65 (m, 2H), 1.36 – 1.32 (m, 4H), 0.90 – 0.86 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.04, 153.61, 141.04, 137.00, 130.34, 128.93, 126.98, 126.72, 125.70, 124.22, 40.37, 31.55, 23.86, 22.59, 14.02. HRMS: calc. for $[M+H]^+$ $C_{15}H_{18}NO$: 228.1383 found: 228.1384.



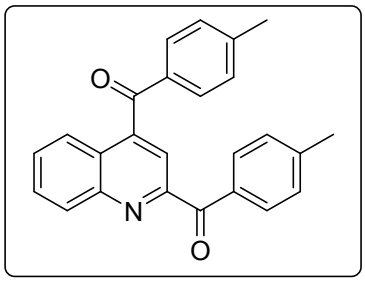
1-(Isoquinolin-1-yl)octan-1-one [2] (**3o**): 96.9 mg (yield: 76 %). ^{1}H NMR (400 MHz, DMSO) δ 8.67 – 8.65 (m, 1H), 8.62 (d, *J* = 5.6 Hz 1H), 8.09 – 8.07 (m, 2H), 7.85 - 7.81 (m, 1H), 7.77 – 7.74 (m, 1H), 3.27 – 3.24 (m, 2H), 1.68 – 1.64 (m, 2H), 1.30 – 1.24 (m, 8H), 0.84 – 0.83 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.06, 153.63, 141.05, 137.00, 130.35, 128.93, 126.98, 126.73, 125.70, 124.22, 40.42, 31.77, 29.34, 29.21, 24.19, 22.67, 14.14.



1-(Isoquinolin-1-yl)dodecan-1-one (**3p**): 139.9 mg (yield: 90 %). ^{1}H NMR (400 MHz, DMSO) δ 8.68 – 8.66 (m, 1H), 8.63 (d, *J* = 5.6 Hz, 1H), 8.10 – 8.07 (m, 2H), 7.86 – 7.82 (m, 1H), 7.78 – 7.74 (m, 1H), 3.26 (t, *J* = 21.6 Hz, 2H), 1.70 – 1.63 (m, 2H), 1.35 – 1.22 (m, 16H), 0.85 (t, *J* = 13.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.07, 153.64, 141.05, 137.00, 130.35, 128.93, 126.98, 126.73, 125.70, 124.21, 40.43, 31.94, 29.65, 29.55, 29.54, 29.38, 24.19, 22.72, 14.16. HRMS: calc. for $[M+H]^+$ $C_{21}H_{30}NO$: 312.2322 found: 213.2323.

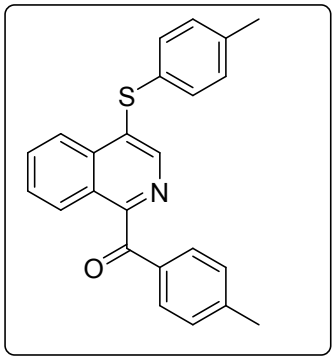


Quinolin-2-yl(p-tolyl)methanone [2] (**3q**): 33.3 mg (yield: 27 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (d, *J* = 8.4 Hz, 1H), 8.20 (d, *J* = 8.4 Hz, 1H), 8.14 (d, *J* = 8.4 Hz, 2H), 8.07 (d, *J* = 8.4 Hz, 1H), 7.89 (d, *J* = 8 Hz, 1H), 7.80 - 7.75 (m, 1H), 7.66 – 7.62 (m, 1H), 7.31 (d, *J* = 8 Hz, 2H), 2.45 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.56, 155.03, 146.72, 144.06, 137.08, 133.51, 131.63, 130.51, 130.08, 128.96, 128.86, 128.33, 127.67, 120.86, 21.83.

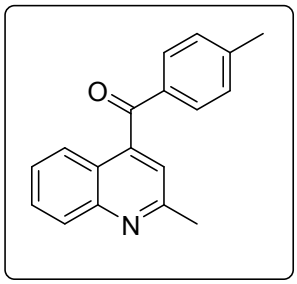


Quinoline-2,4-diylbis(p-tolylmethanone) [2] (**3r**): 120.5 mg (yield: 66 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (d, *J* = 8 Hz, 1H), 8.18 (d, *J* = 8.4 Hz, 2H), 8.09 (s, 1H), 7.95 (d, *J* = 8.8 Hz, 1H), 7.83 – 7.78 (m, 3H), 7.65 - 7.60 (m, 1H), 7.34 – 7.26 (m, 4H), 2.45 (d,

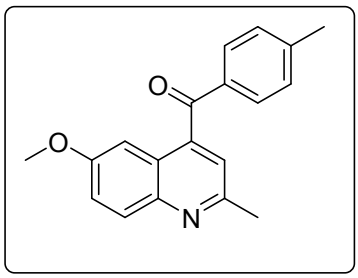
J = 8 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.35, 192.75, 154.21, 147.19, 145.78, 145.65, 144.31, 133.95, 133.23, 131.66, 131.03, 130.57, 129.64, 129.37, 129.04, 125.71, 125.43, 119.37, 21.90, 21.87.



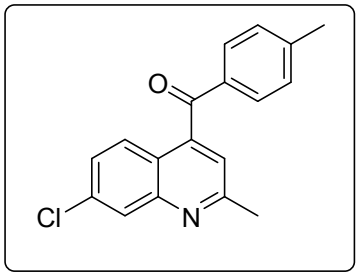
p-Tolyl(4-(p-tolylthio)isoquinolin-1-yl)methanone (**3s**): 110.7 mg (yield: 60 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (s, 1H), 8.34 (d, *J* = 8.4 Hz, 1H), 8.25 (d, *J* = 8.4 Hz, 1H), 7.84 (d, *J* = 8.4 Hz, 2H), 7.76 – 7.72 (m, 1H), 7.63 - 7.59 (m, 1H), 7.33 – 7.31 (m, 2H), 2.24 (d, *J* = 8 Hz, 2H), 7.14 (d, *J* = 8 Hz, 2H), 2.39 (s, 3H), 2.32 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 194.21, 155.36, 144.73, 142.56, 138.30, 135.67, 134.20, 132.12, 131.70, 131.16, 130.91, 130.48, 129.22, 129.18, 128.68, 126.87, 126.36, 124.47, 21.87, 21.21. HRMS: calc. for $[M+Na]^+$ $C_{24}H_{19}NNaOS$: 392.1080 found: 392.1084.



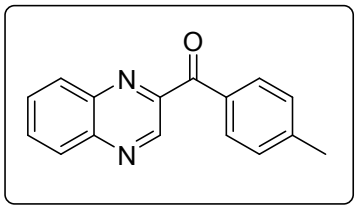
(2-Methylquinolin-4-yl)(p-tolyl)methanone [3] (**3t**): 96.6 mg (yield: 74 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.4 Hz, 1H), 7.77 – 7.69 (m, 4H), 7.46 – 7.42 (m, 1H), 7.28 – 7.25 (m, 3H), 2.79 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.94, 158.28, 148.21, 145.36, 145.12, 134.19, 130.43, 129.97, 129.50, 129.13, 126.60, 125.24, 123.34, 120.27, 25.43, 21.84.



(6-Methoxy-2-methylquinolin-4-yl)(p-tolyl)methanone (**3u**): 116.4 mg (yield: 80 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 9.2 Hz, 1H), 7.77 – 7.75 (m, 2H), 7.38 – 7.35 (m, 1H), 7.30 – 7.27 (m, 2H), 7.23 (s, 1H), 7.11 (d, *J* = 2.8 Hz, 1H), 3.76 (s, 3H), 2.74 (s, 3H), 2.44 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.37, 157.87, 155.35, 145.25, 144.60, 143.24, 134.31, 130.50, 129.48, 124.27, 122.71, 120.96, 102.97, 55.49, 25.06, 21.86. HRMS: calc. for $[M+H]^+$ $C_{19}H_{18}NO_2$: 292.1332 found: 292.1339.



(7-Chloro-2-methylquinolin-4-yl)(p-tolyl)methanone (**3v**): 95.9 mg (yield: 65 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 2 Hz, 1H), 7.74 – 7.71 (m, 3H), 7.40 – 7.38 (m, 1H), 7.28 (t, *J* = 8 Hz, 3H), 2.78 (s, 3H), 2.44 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 195.37, 159.63, 148.72, 145.60, 144.86, 135.85, 133.96, 130.43, 129.58, 128.19, 127.57, 126.57, 121.77, 120.56, 25.47, 21.87. HRMS: calc. for $[M+H]^+$ $C_{18}H_{15}ClNO$: 296.0837 found: 296.0836.



Quinoxalin-2-yl(p-tolyl)methanone [2] (**3w**): 59.5 mg (yield: 48 %). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.46 (s, 1H), 8.20 – 8.14 (m, 4H), 7.91 – 7.82 (m, 2H), 7.33 (d, *J* = 8 Hz, 2H),

2.46 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 191.95, 148.96, 145.34, 144.77, 143.08, 140.42, 132.91, 131.93, 131.42, 130.79, 130.43, 129.38, 129.17, 21.88.

References

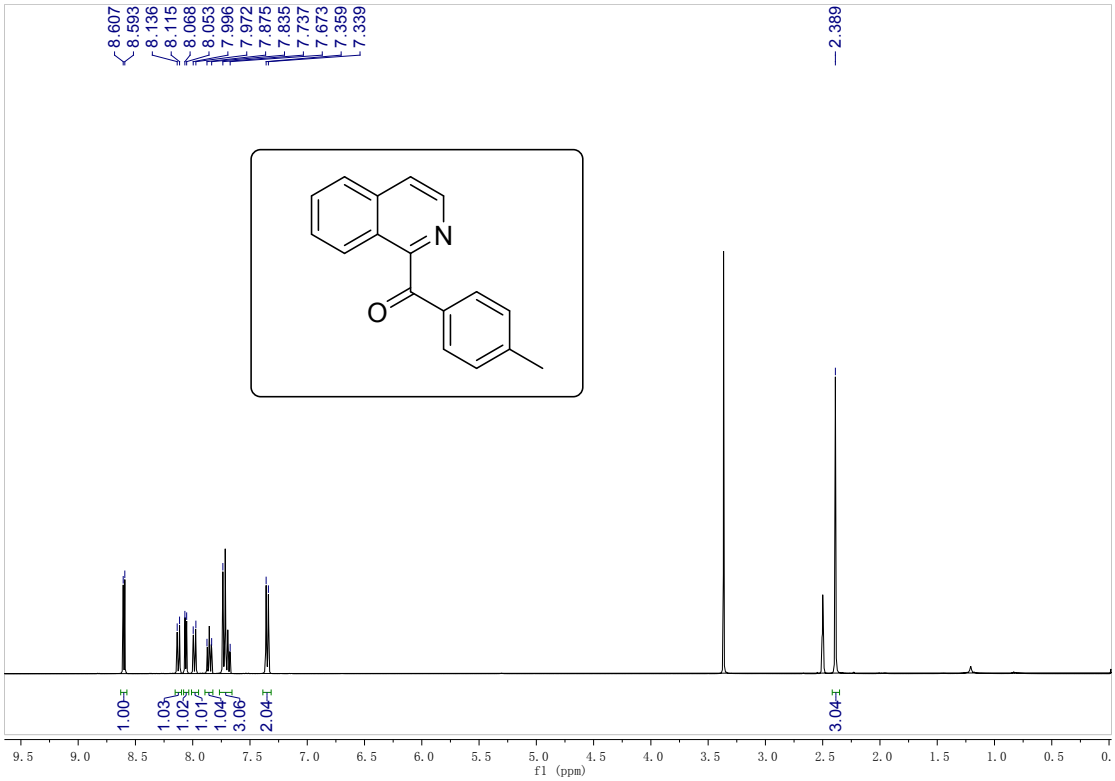
[1] Kiran Matcha and Andrey P. Antonchick, *Angew. Chem. Int. Ed.,* 2013*, **52,*** 2082 – 2086.

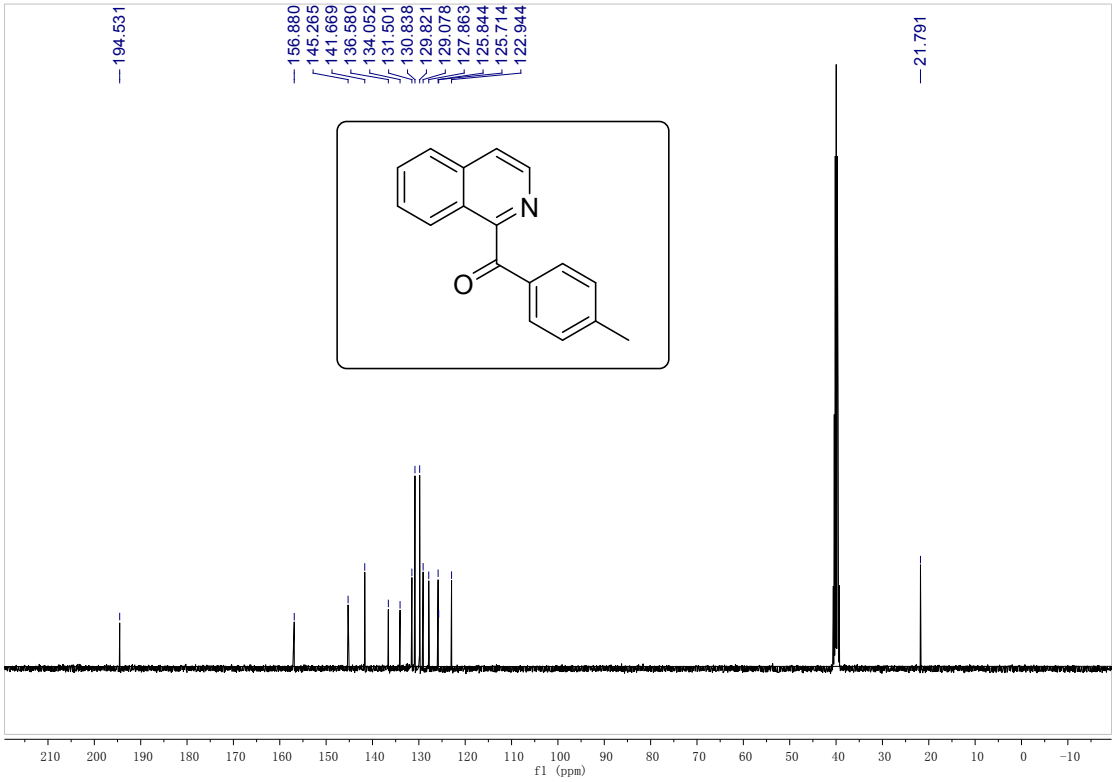
[2] Yogesh Siddaraju, Kandikere Ramaiah Prabhu, *Tetrahedron*, 2016, ***72***, 959 – 967.

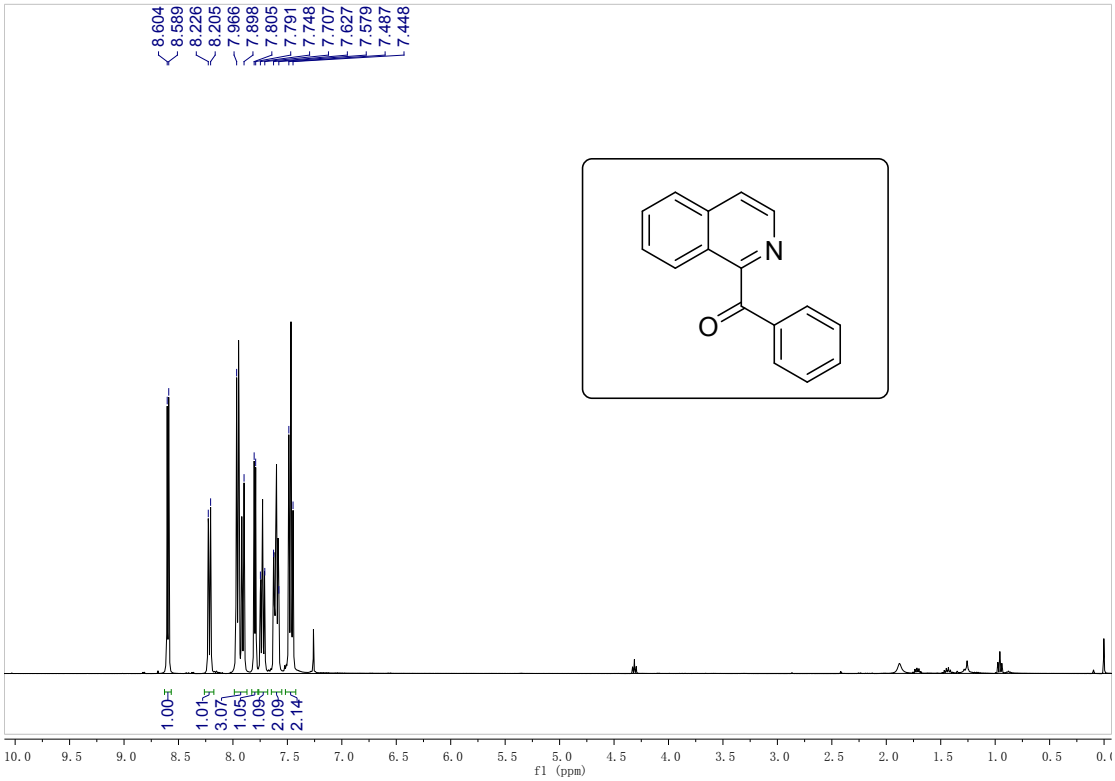
[3] Jiayu Chen, Miao Wan, Jing Hua, Yi Sun, Zheng Lv, Wei Li and Lei Liu, *Org. Biomol. Chem.,* 2015, ***13***, 11561 – 11566.

[4] B. Reux, T. Nevalainen, K. H. Raitio and A. M. P. Koskinen, *Bioorg. Med. Chem.*, 2009, ***17***, 4441.

6. NMR Spectra of Products



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7.579
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O
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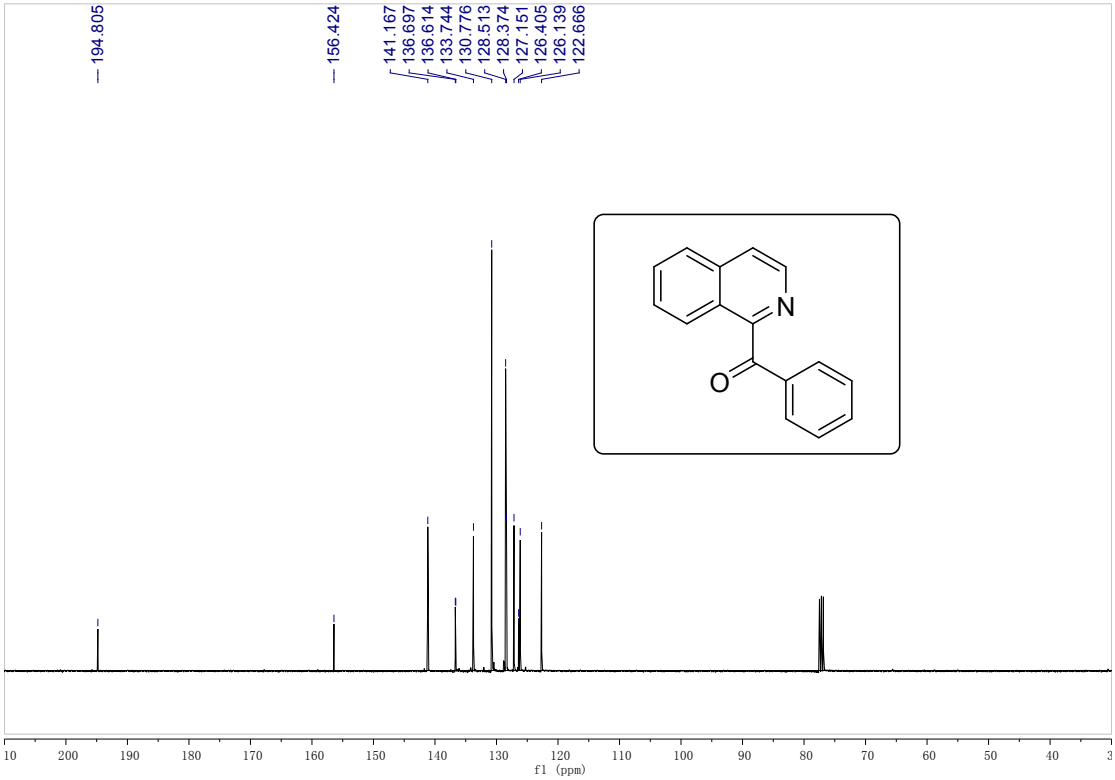
1.05
1.09
2.09
2.14
f1 (ppm)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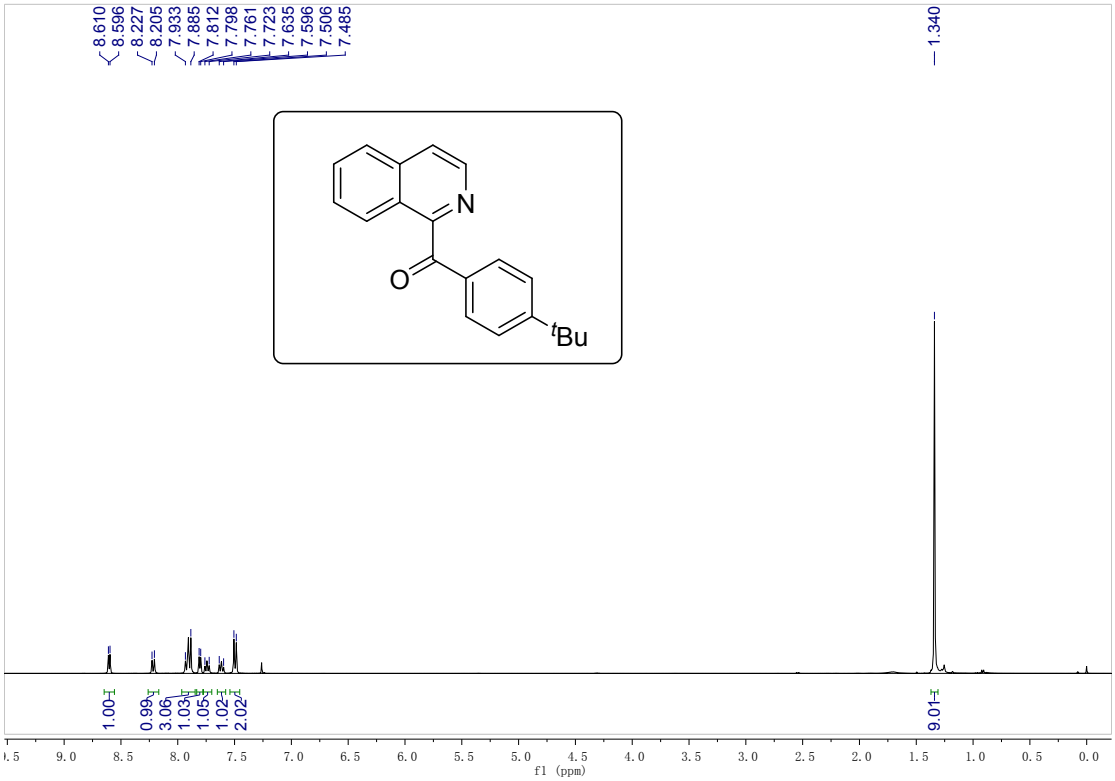
N
O
f1 (ppm)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7.723
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7.596
7.506
7.485
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O
tBu
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3.06
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1.05
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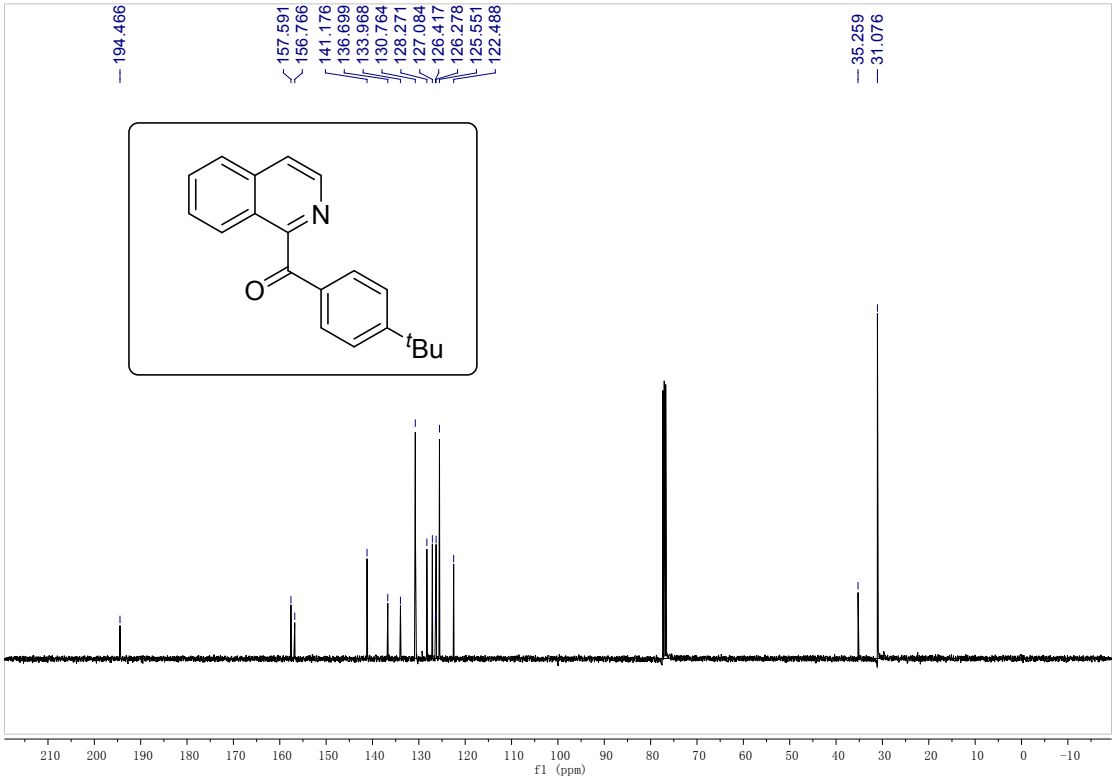
9.01
f1 (ppm)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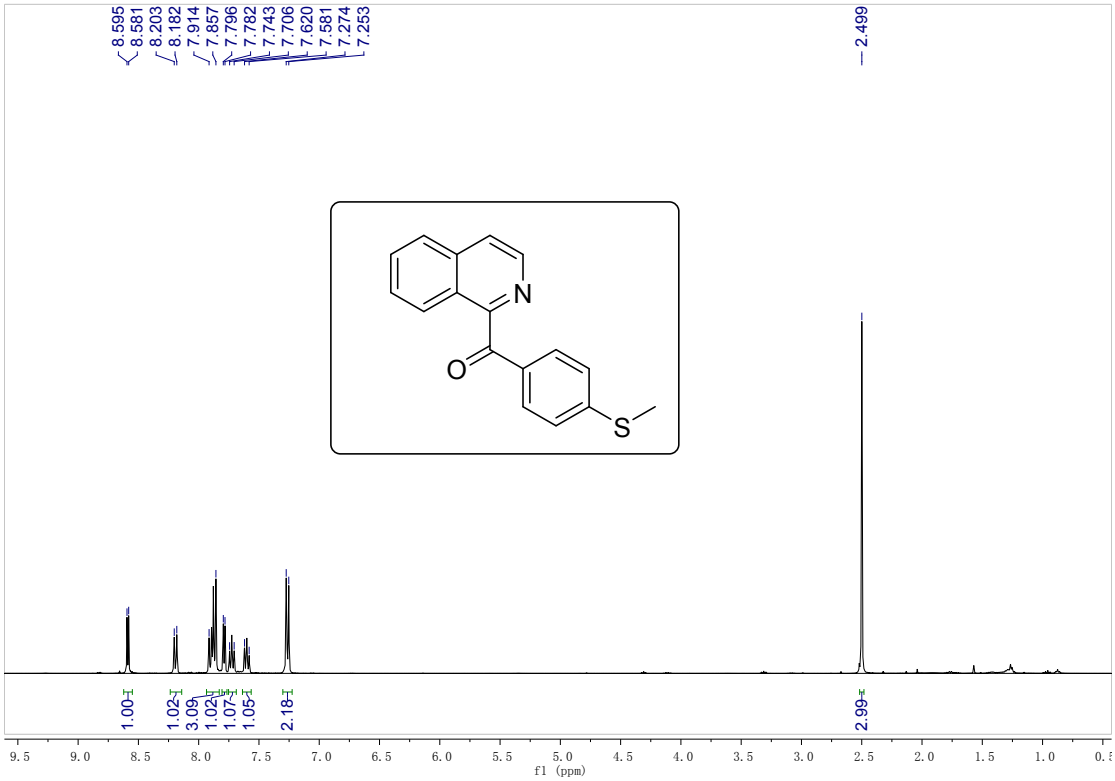
O
tBu
f1 (ppm)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7.706
7.620
7.581
7.274
7.253
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O
S
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1.05
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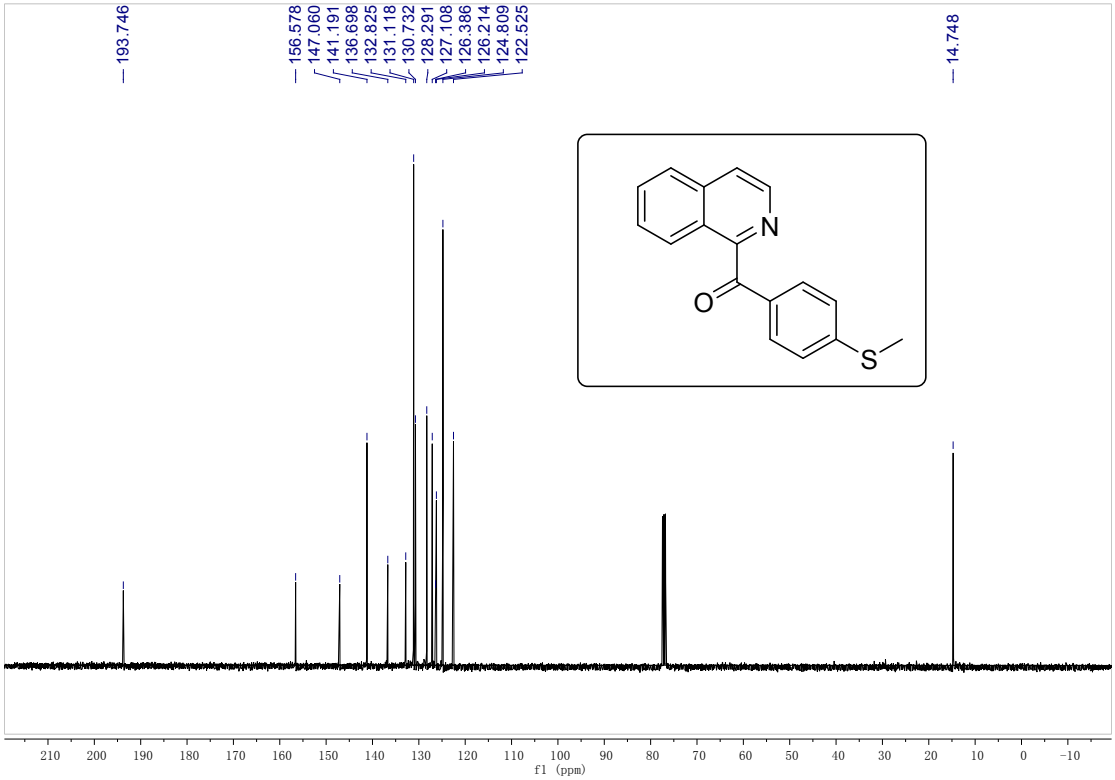
2.99
f1 (ppm)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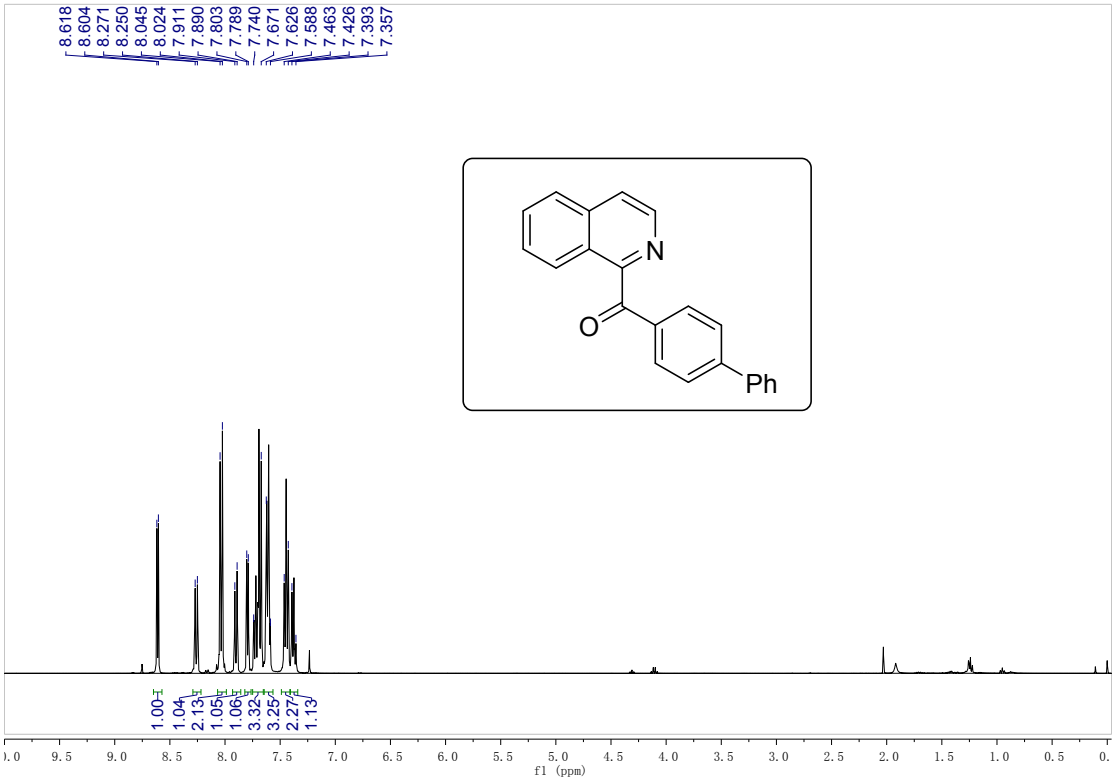
N
O
S
f1 (ppm)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7.588
7.463
7.426
7.393
7.357
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O
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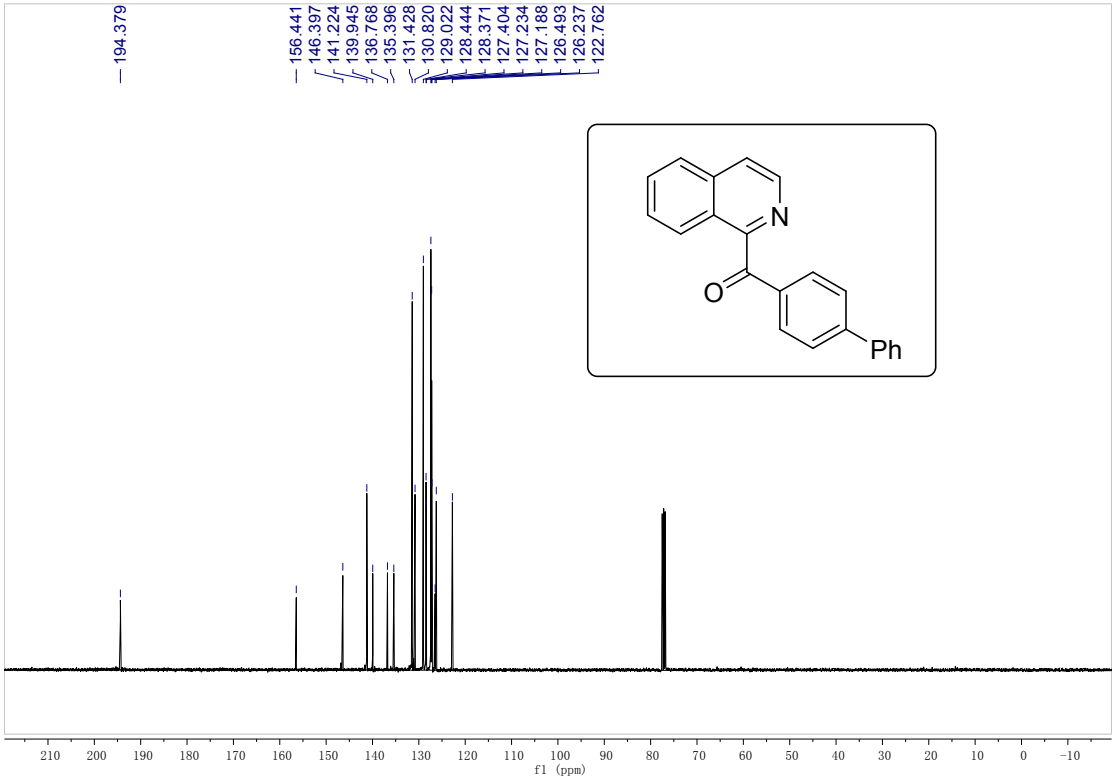
1.13
f1 (ppm)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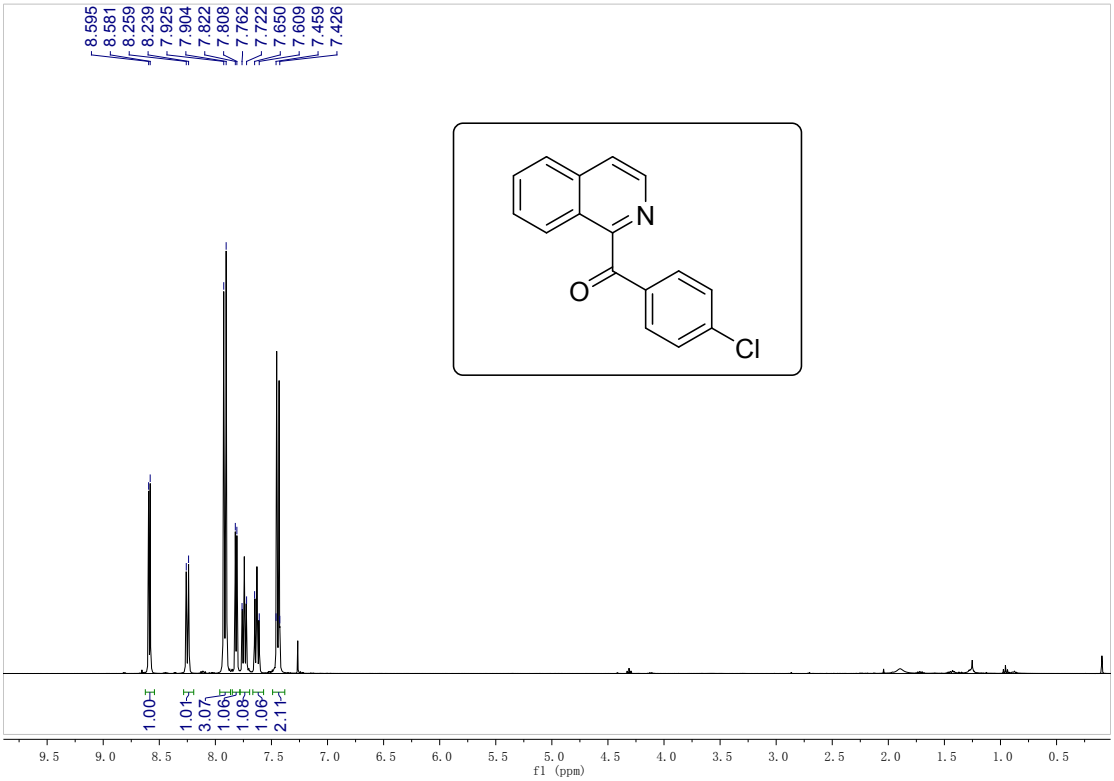
O
Ph
f1 (ppm)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7.609
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O
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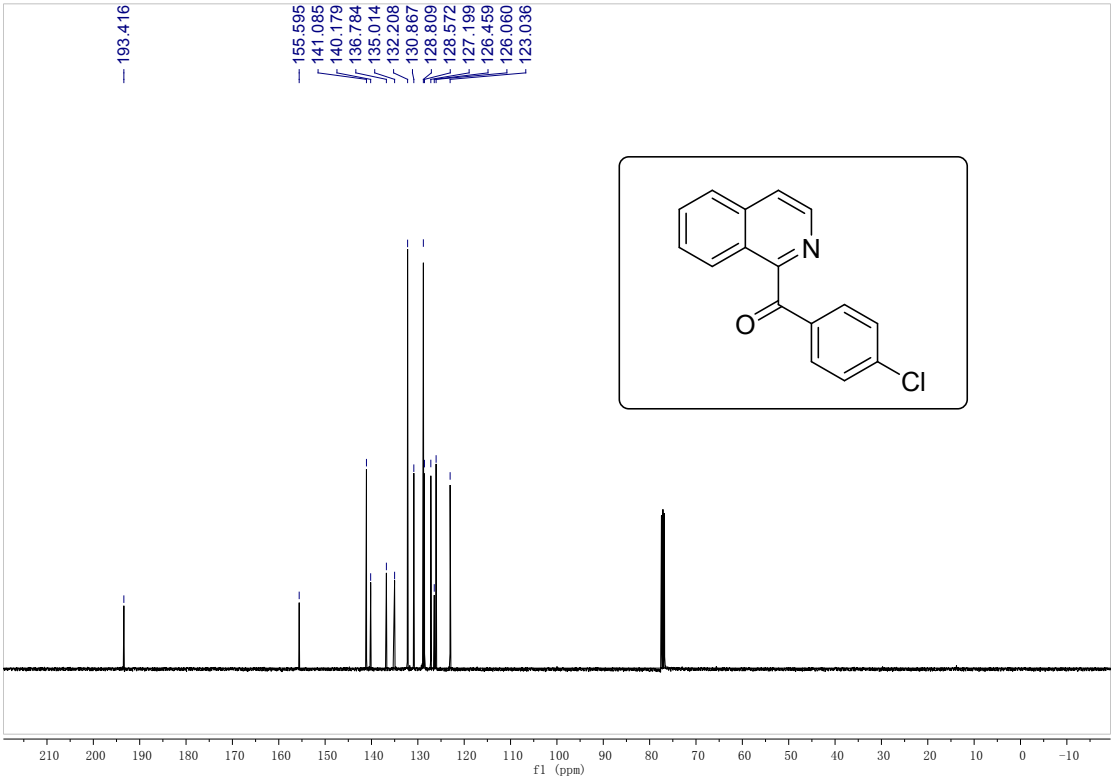
f1 (ppm)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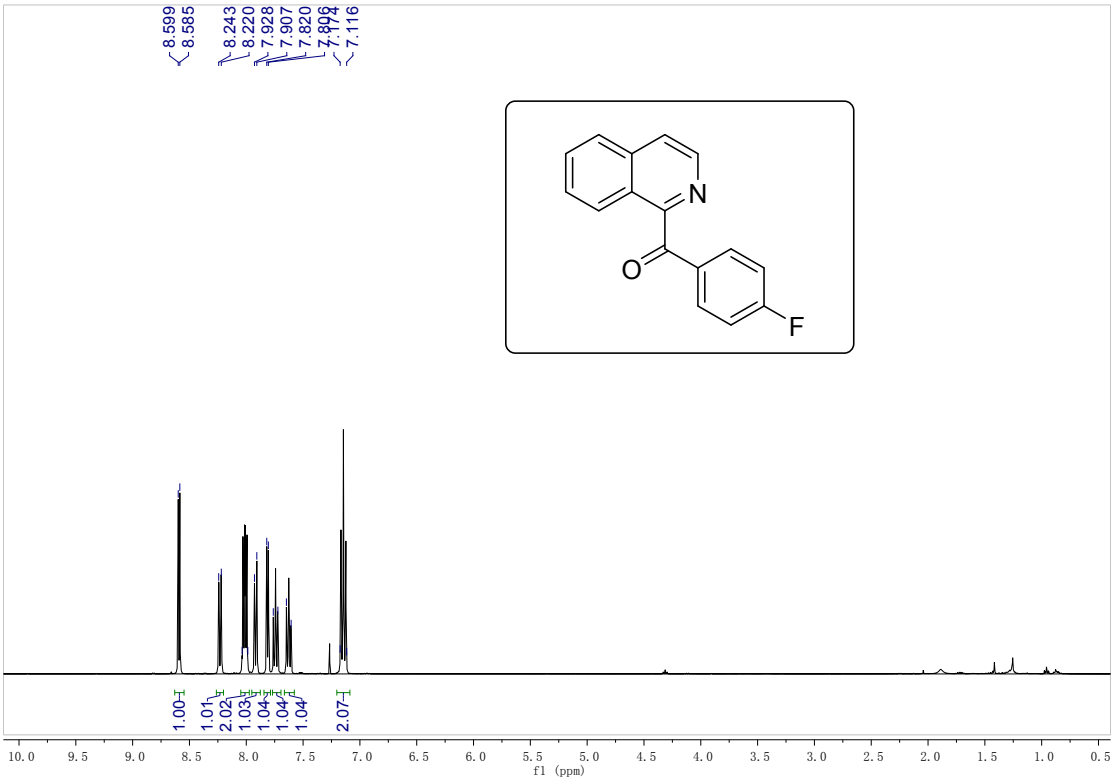
f1 (ppm)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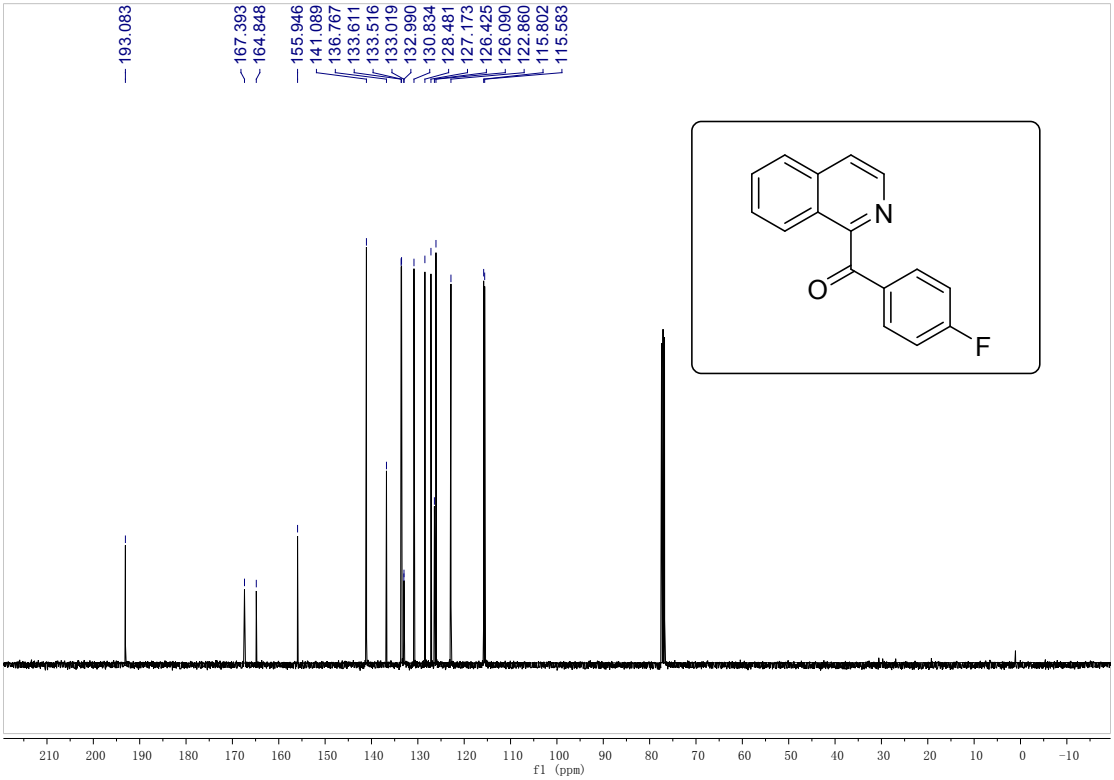
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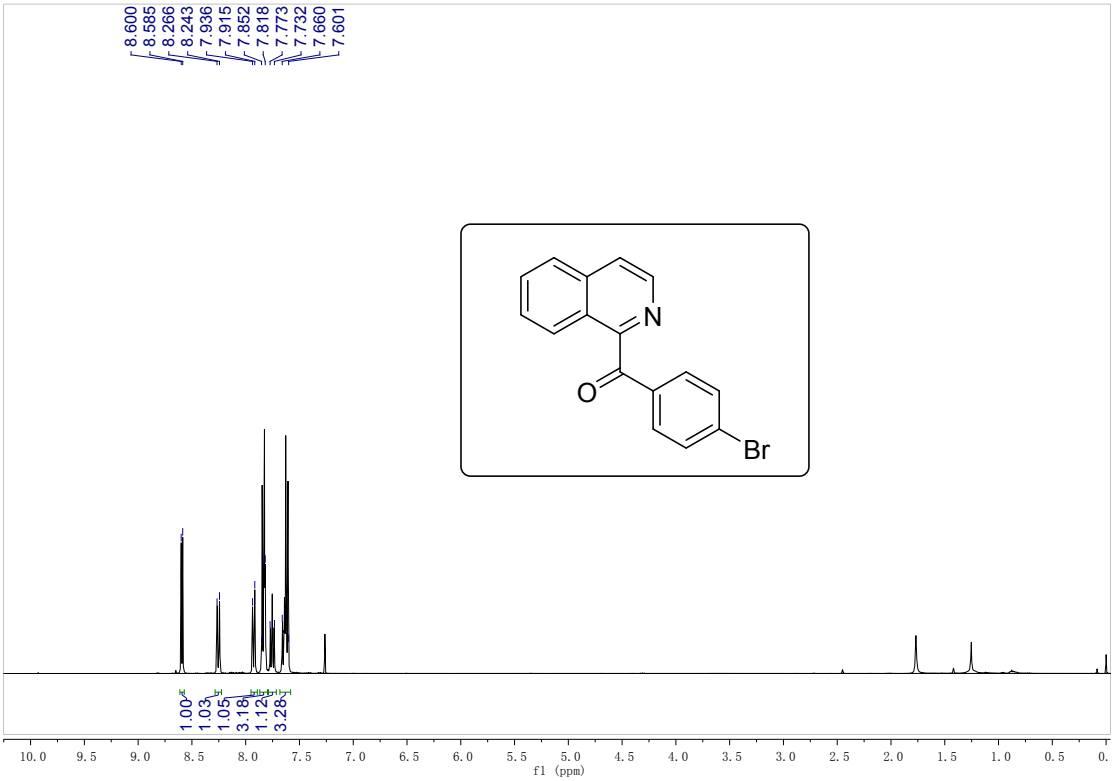
f1 (ppm)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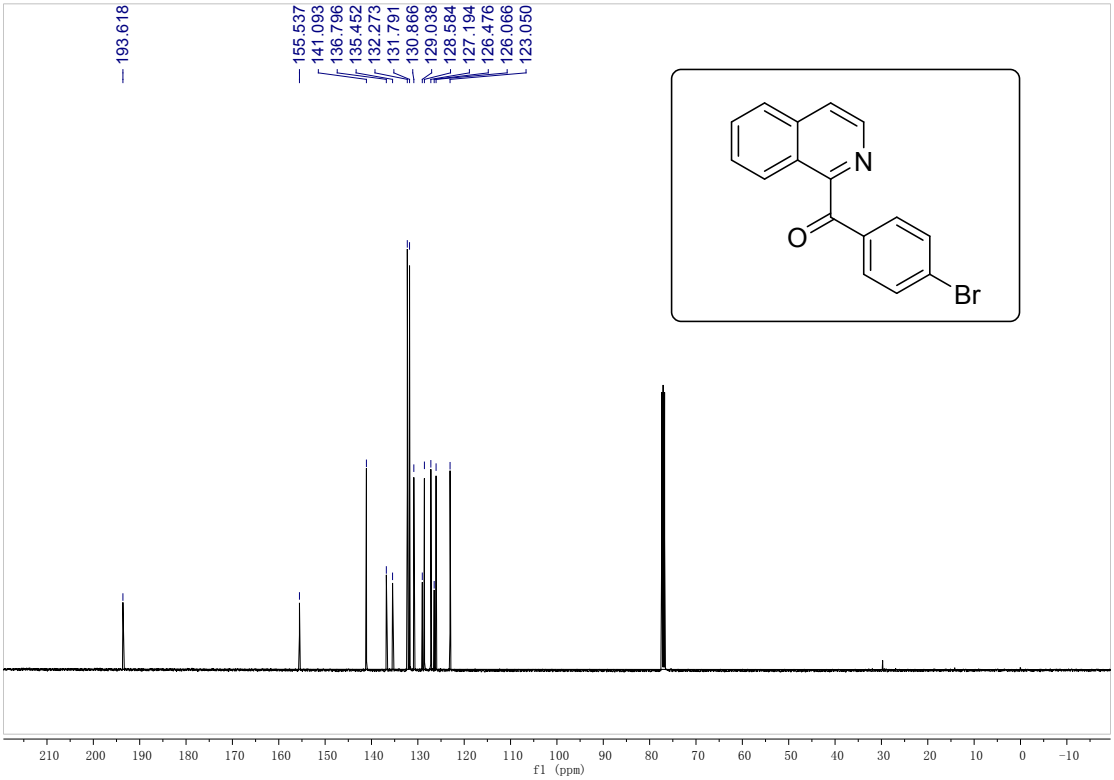
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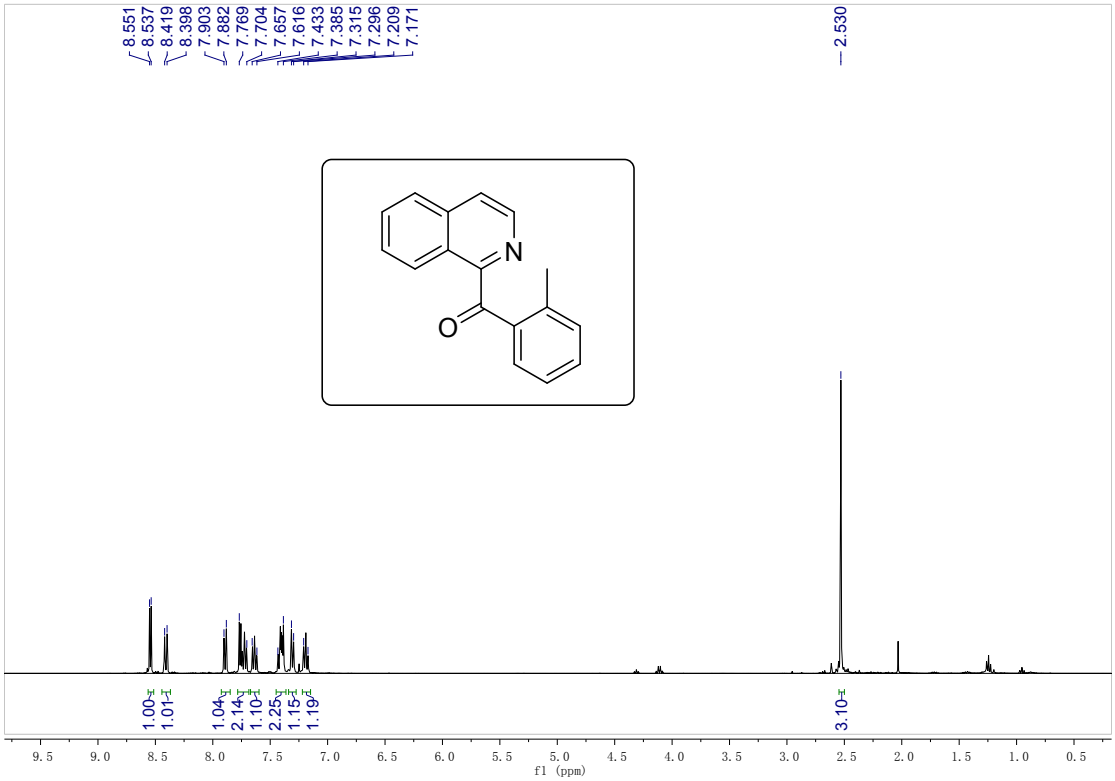
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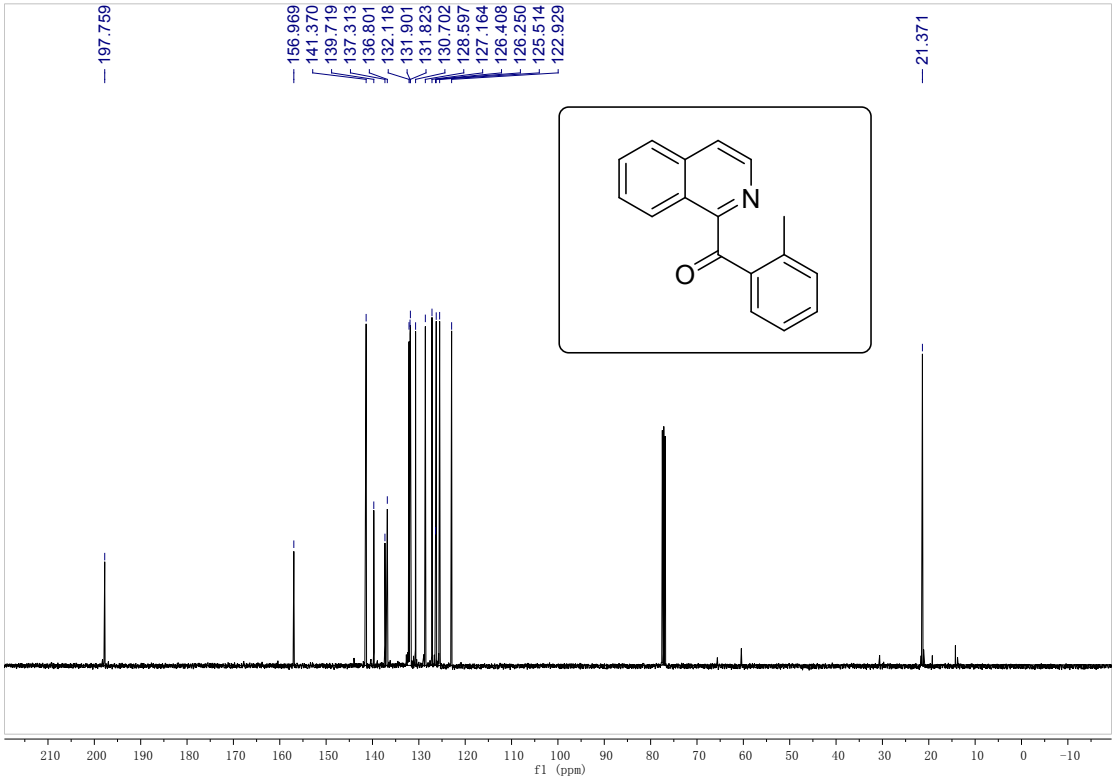
f1 (ppm)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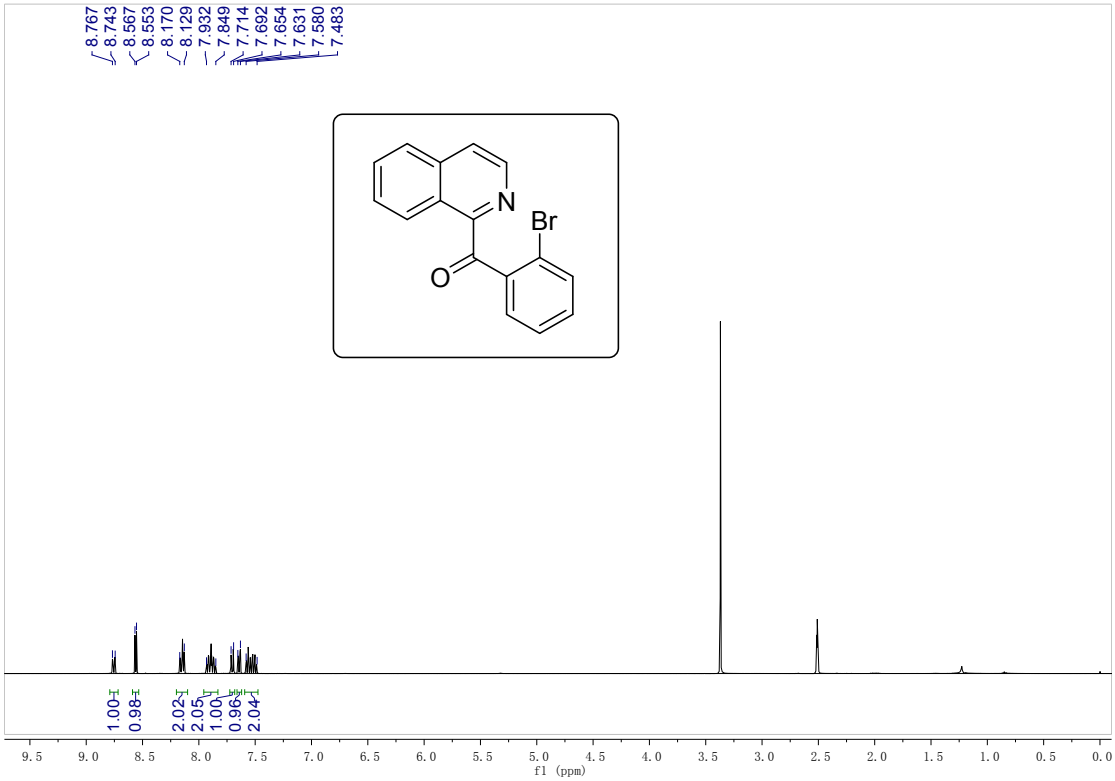
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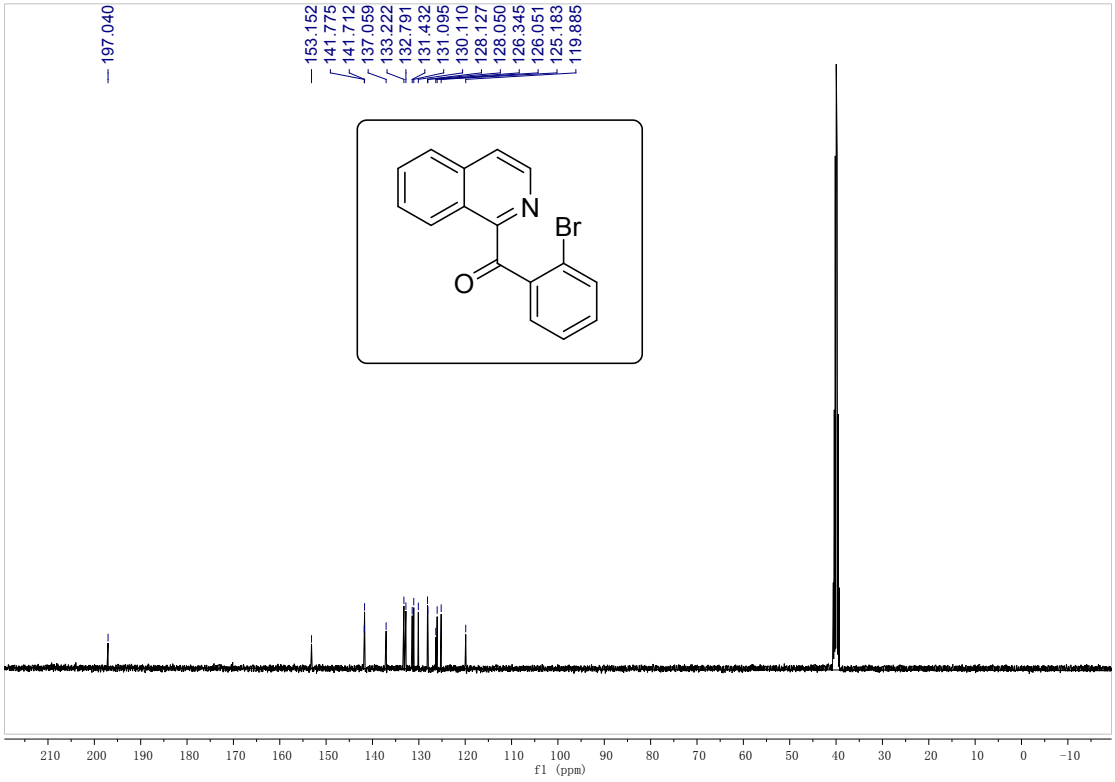
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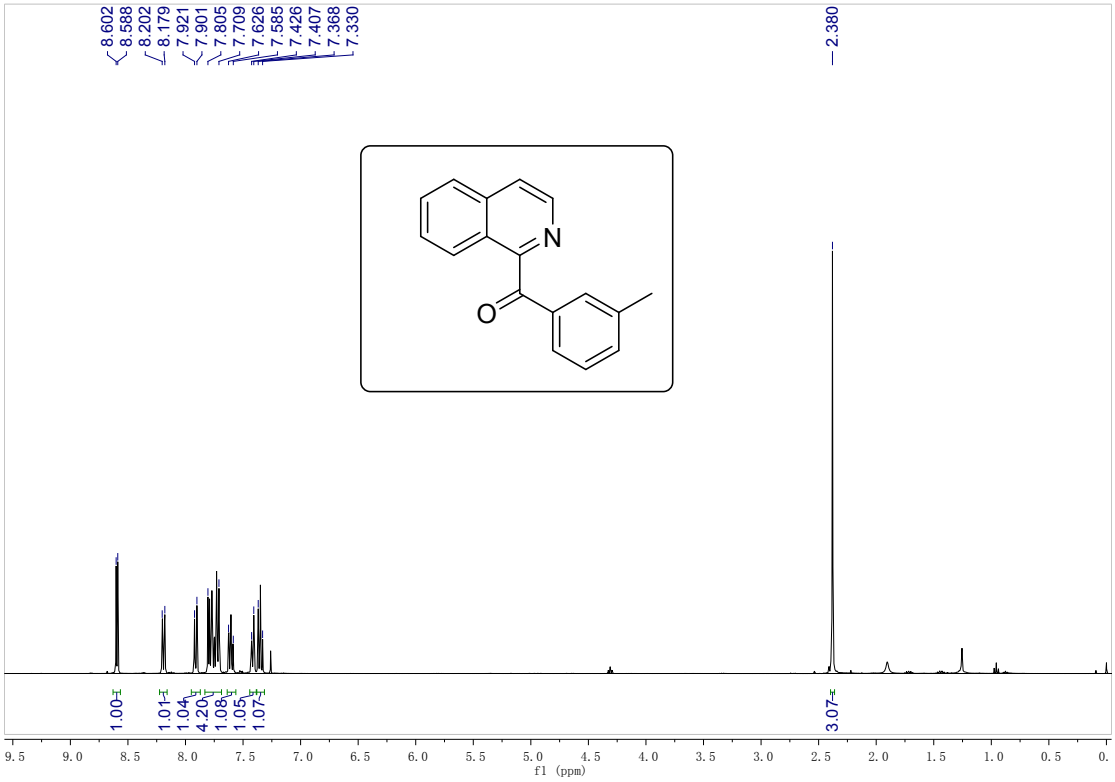
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7.368
7.330
2.380
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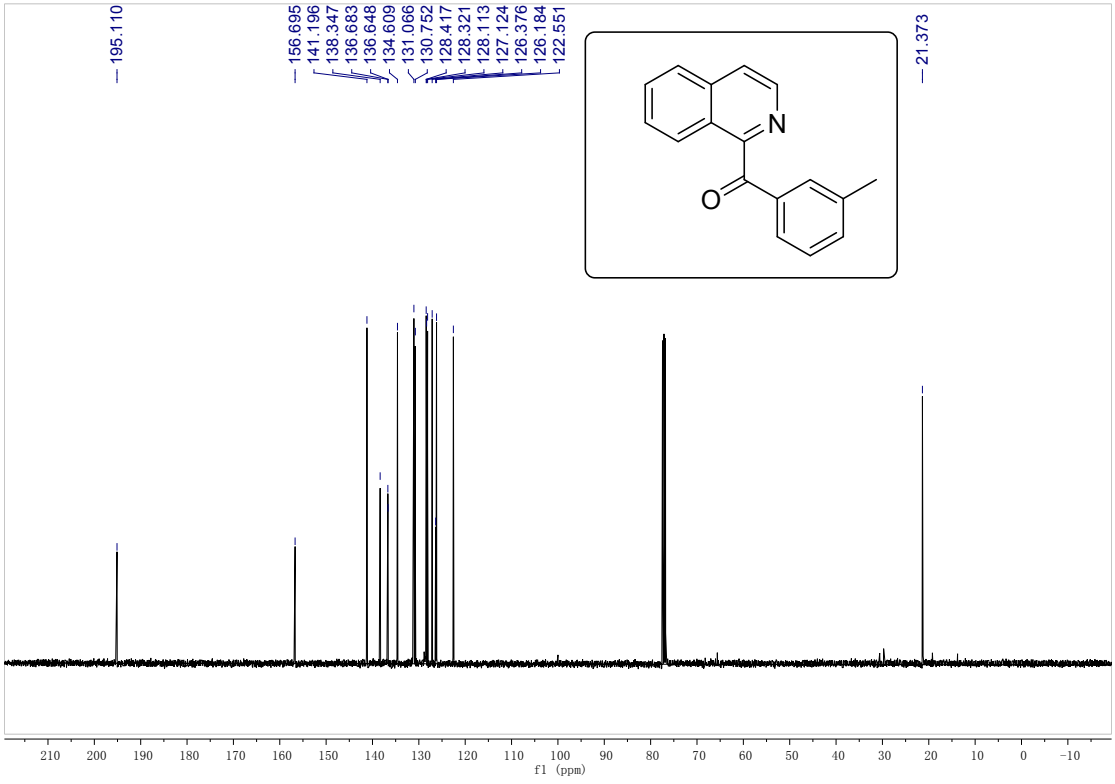
O
1.00
1.01
1.04
4.20
1.08
1.05
1.07
3.07
f1 (ppm)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156.695
141.196
138.347
136.683
136.648
134.609
131.066
130.752
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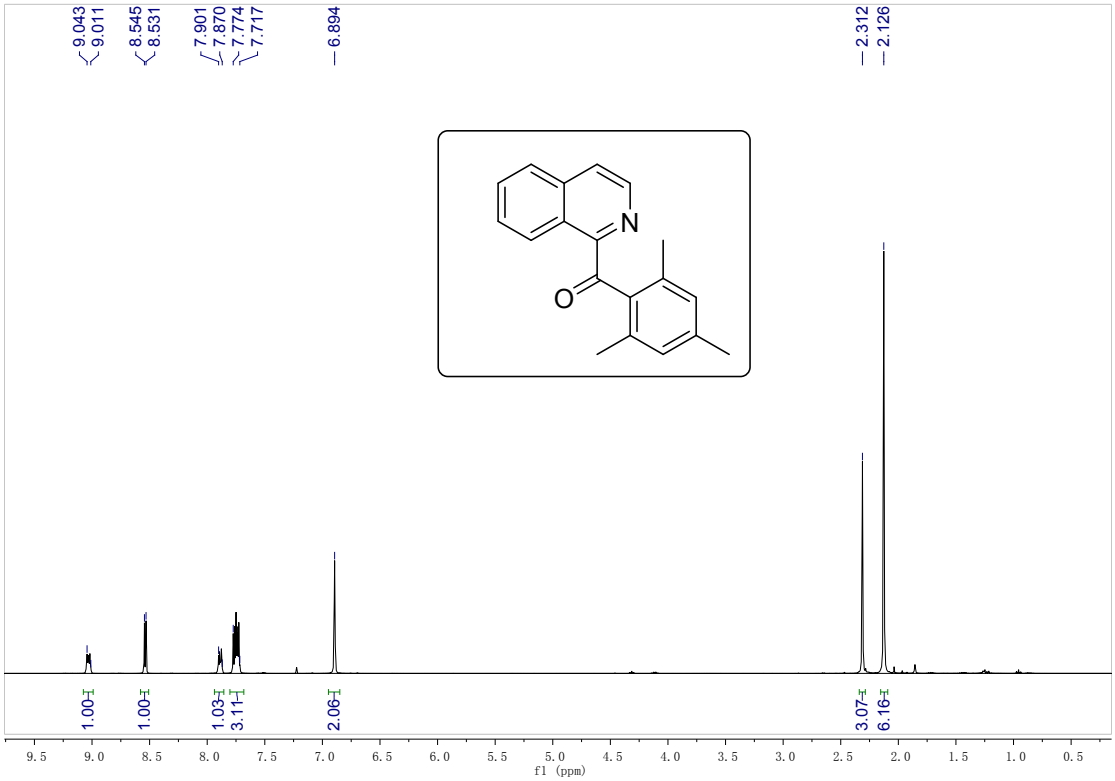
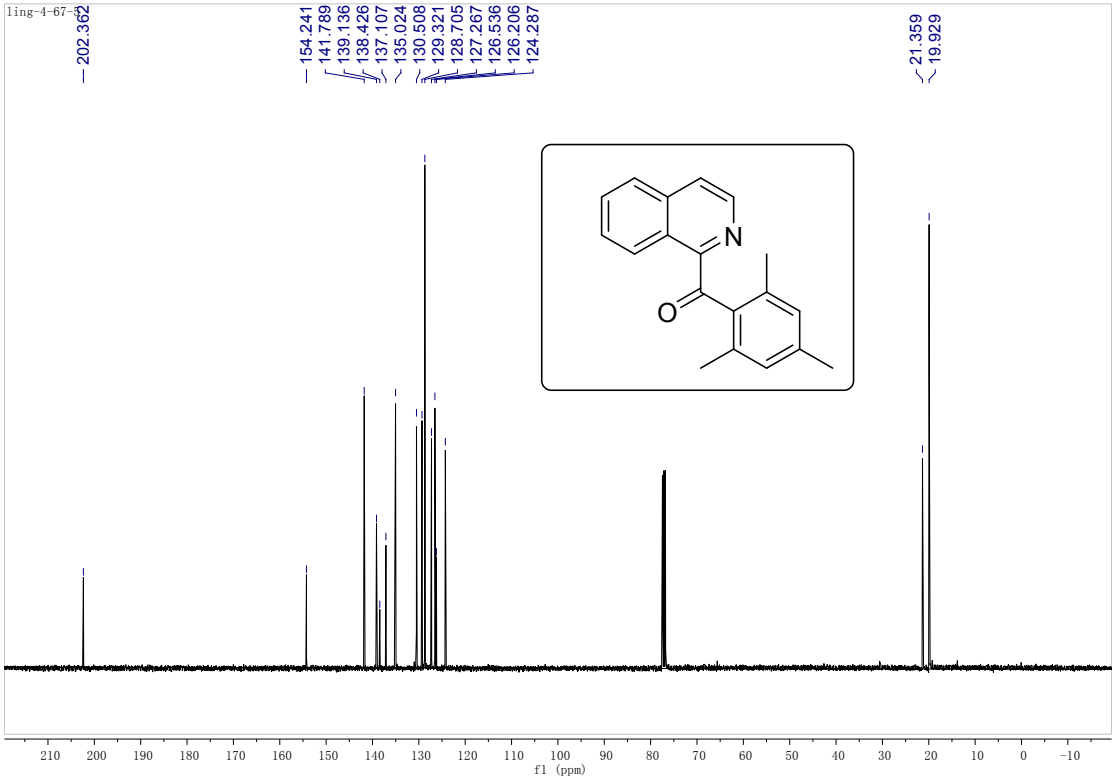
128.321
128.113
127.124
126.376
126.184
122.551
21.373
N
O
f1 (ppm)


9.043
9.011
8.545
8.531
7.901
7.870
7.774
7.717
6.894
2.312
2.126
N
O
1.00
1.00
1.03
3.11
2.06
3.07
6.16
f1 (ppm)


ling-4-67-c2
202.362
154.241
141.789
139.136
138.426
137.107
135.024
130.508
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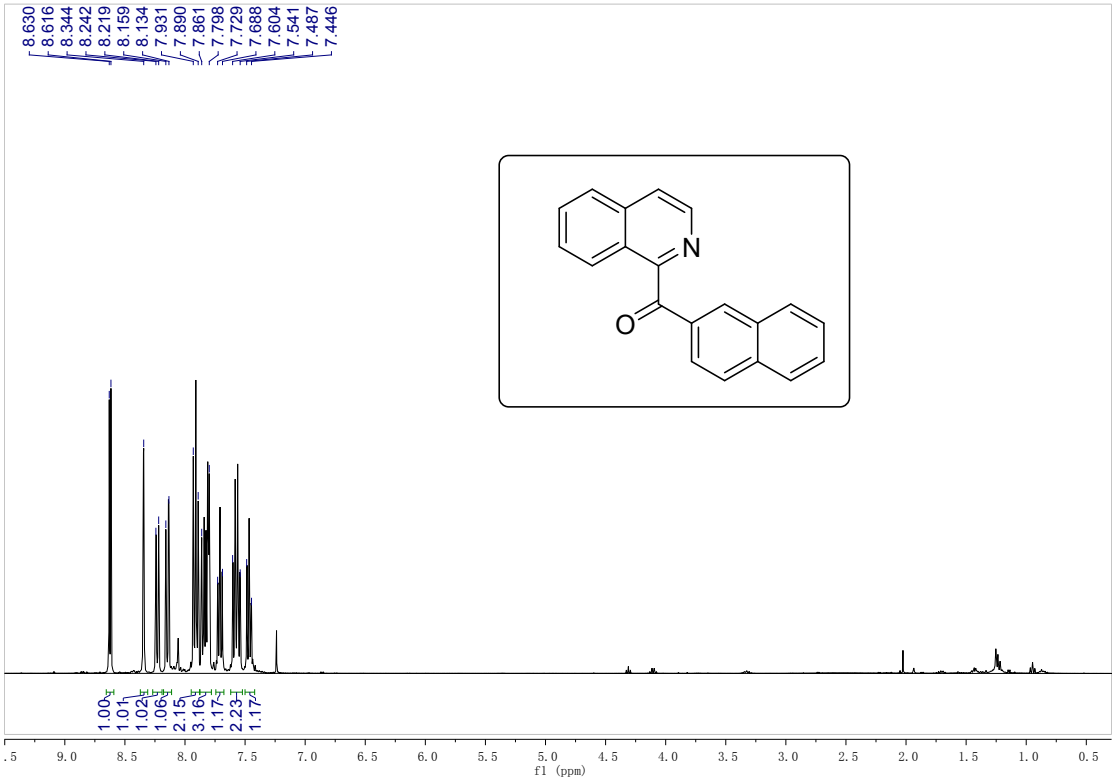
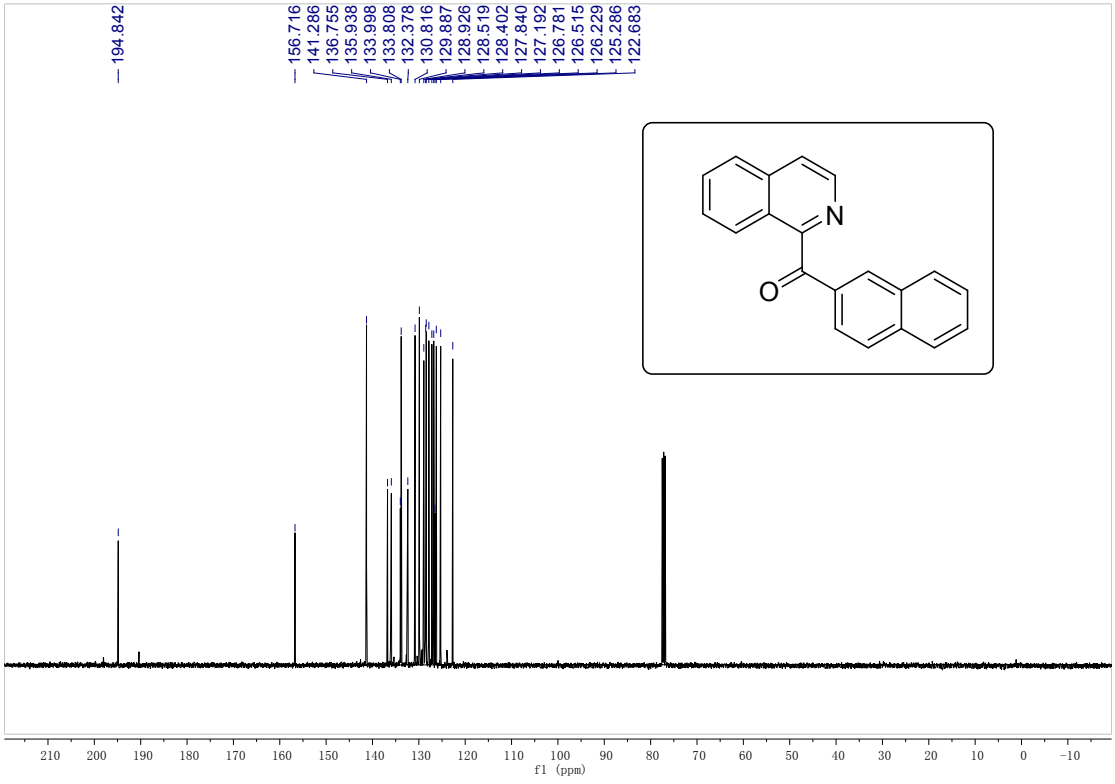
128.705
127.267
126.536
126.206
124.287
21.359
19.929
N
O
f1 (ppm)


8.630
8.616
8.344
8.242
8.219
8.159
8.134
7.931
7.890
7.861
7.798
7.729
7.688
7.604
7.541
7.487
7.446
N
O
1.00
1.01
1.02
1.06
2.15
3.16
1.17
2.23
1.17
f1 (ppm)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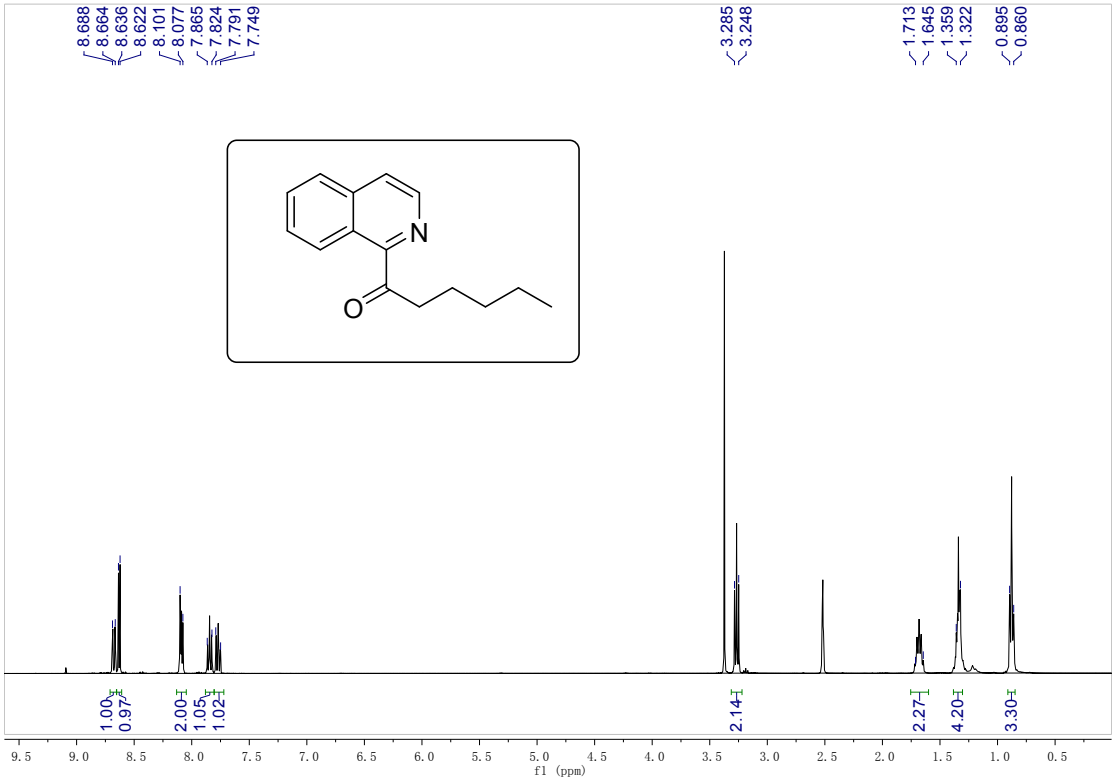
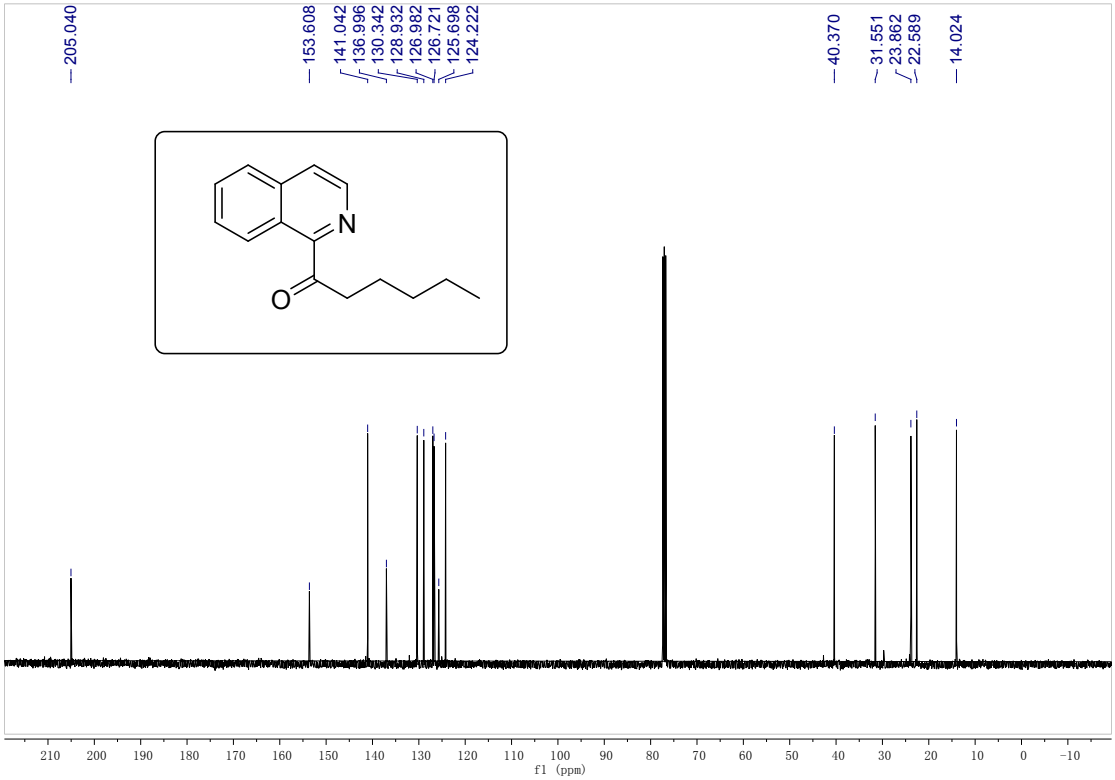
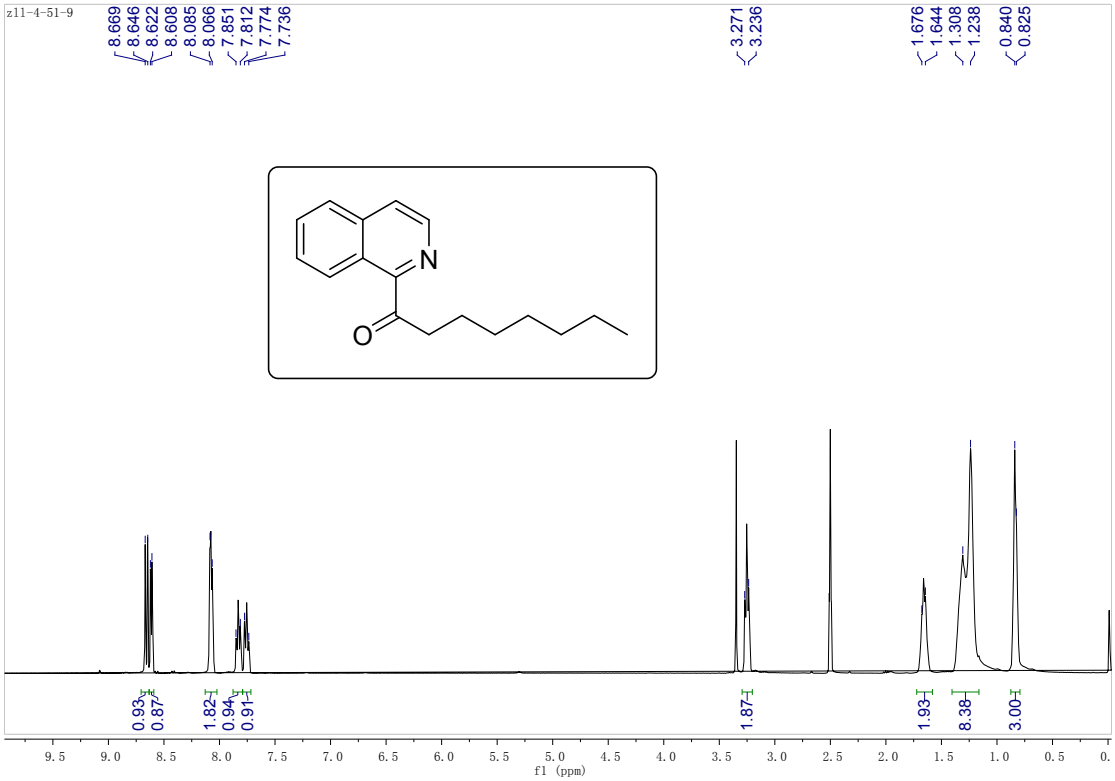
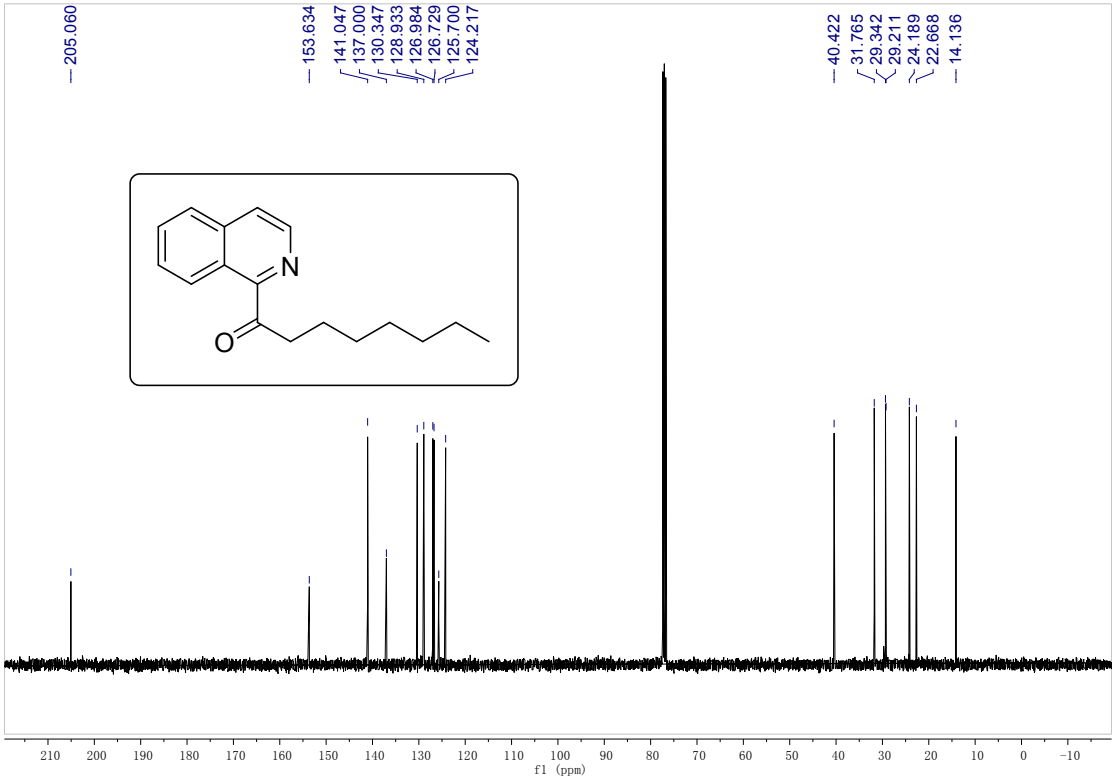
156.716
141.286
136.755
135.938
133.998
133.808
132.378
130.816
129.887
128.926
128.519
128.402
127.840
127.192
126.781
126.515
126.229
125.286
122.683
N
O
f1 (ppm)


8.688
8.664
8.636
8.622
8.101
8.077
7.865
7.824
7.791
7.749
3.285
3.248
1.713
1.645
1.359
1.322
0.895
0.860
N
O
1.00
0.97
2.00
1.05
1.02
2.14
2.27
4.20
3.30
f1 (ppm)


205.040
153.608
141.042
136.996
130.342
128.932
126.982
126.721
125.698
124.222
40.370
31.551
23.862
22.589
14.024
N
O
f1 (ppm)


z11-4-51-9
8.669
8.646
8.622
8.608
8.085
8.066
7.851
7.812
7.774
7.736
3.271
3.236
1.676
1.644
1.308
1.238
0.840
0.825
N
O
0.93
0.87
1.82
0.94
0.91
1.87
1.93
8.38
3.00
f1 (ppm)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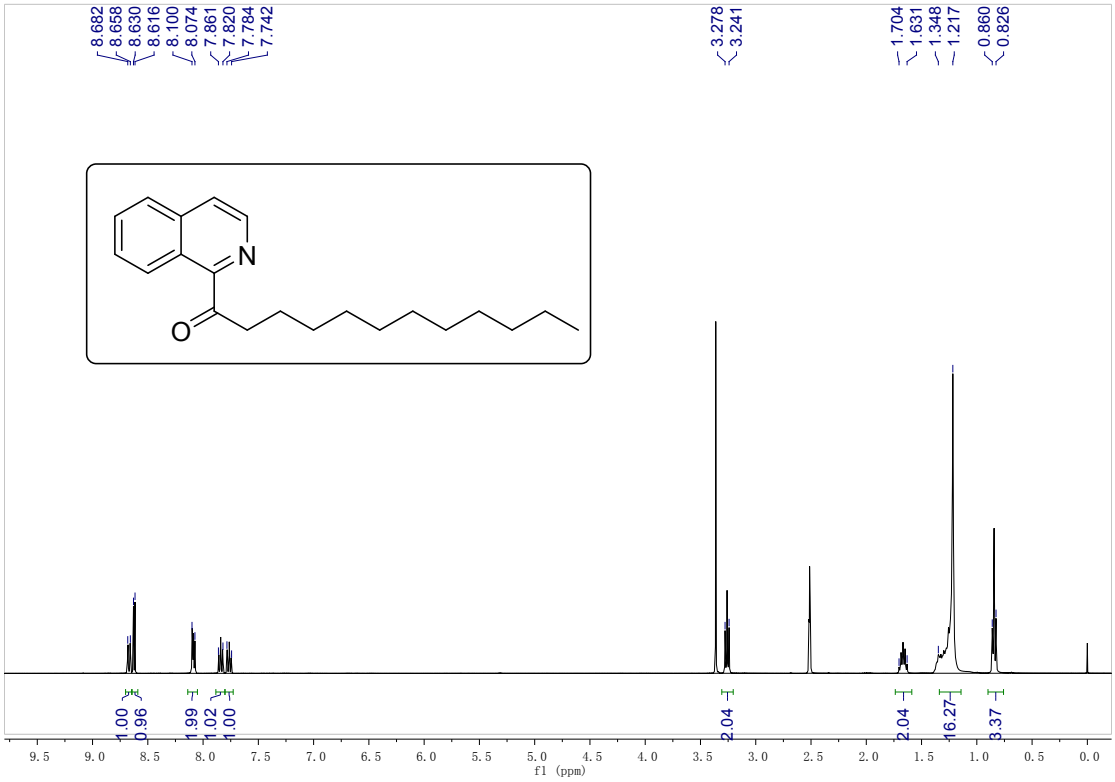
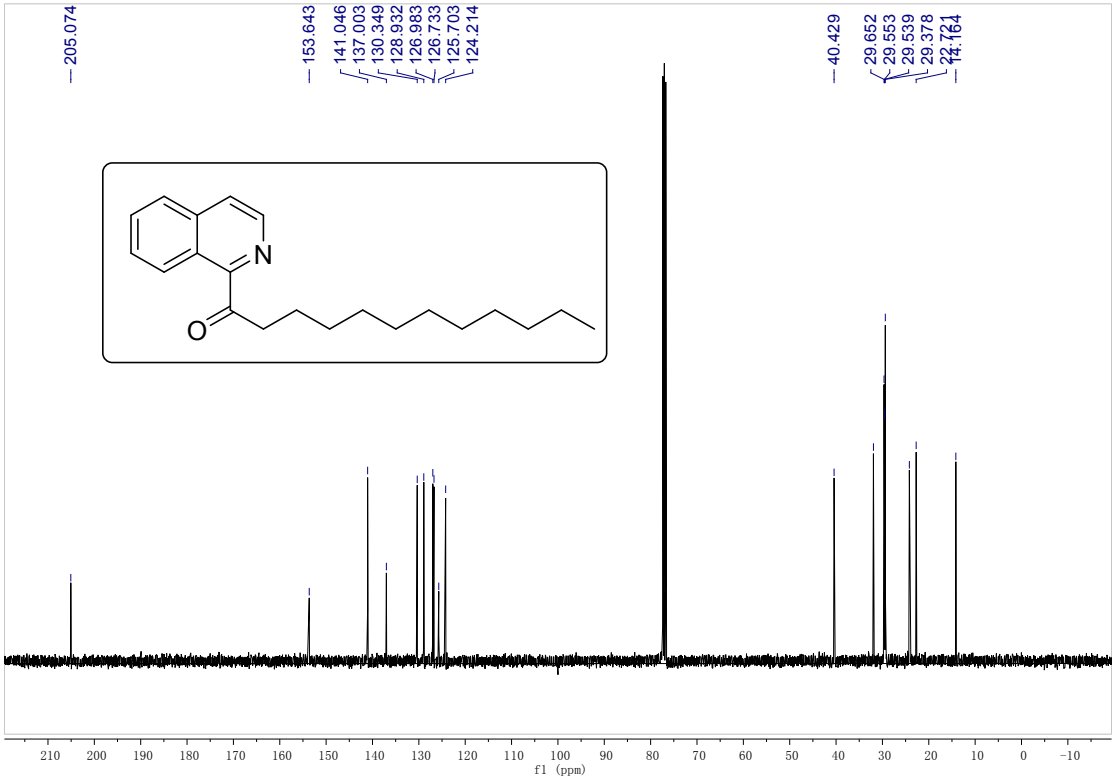
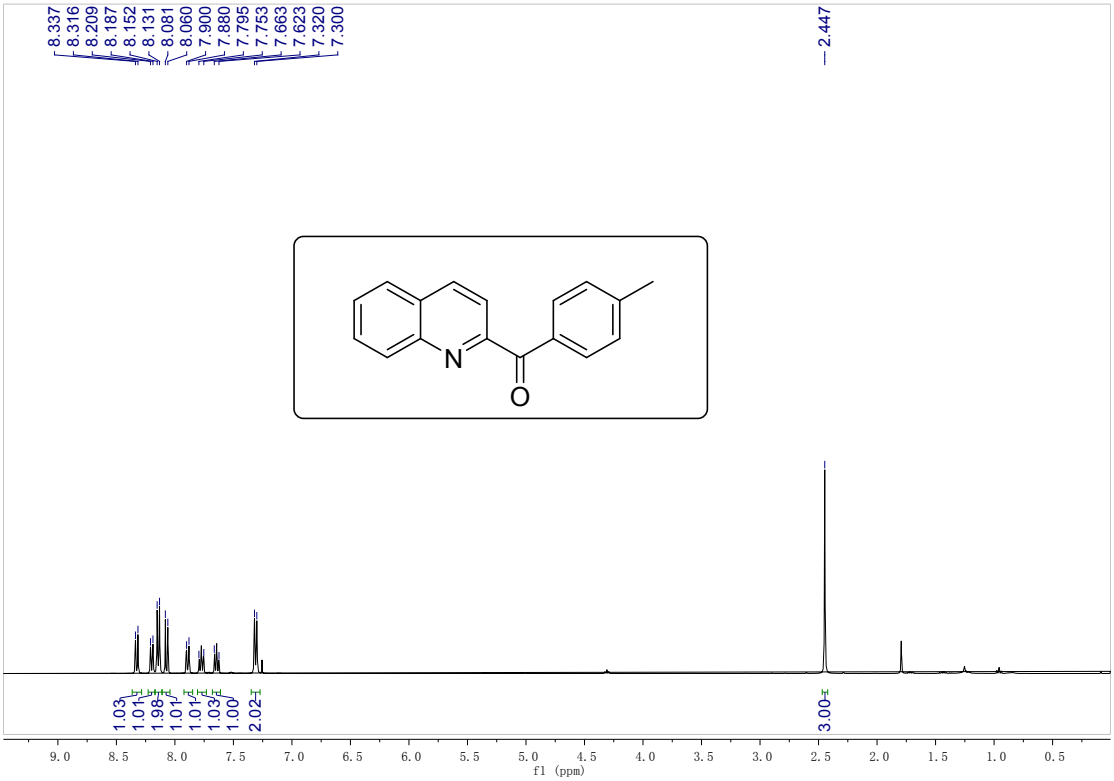
153.634
141.047
137.000
130.347
128.933
126.984
126.729
125.700
124.217
40.422
31.765
29.342
29.211
24.189
22.668
14.136
N
O
f1 (ppm)


8.682
8.658
8.630
8.616
8.100
8.074
7.861
7.820
7.784
7.742
3.278
3.241
1.704
1.631
1.348
1.217
0.860
0.826
N
O
1.00
0.96
1.99
1.02
1.00
2.04
2.04
16.27
3.37
f1 (ppm)


205.074
153.643
141.046
137.003
130.349
128.932
126.983
126.733
125.703
124.214
40.429
29.652
29.553
29.539
29.378
22.721
14.164
N
O
f1 (ppm)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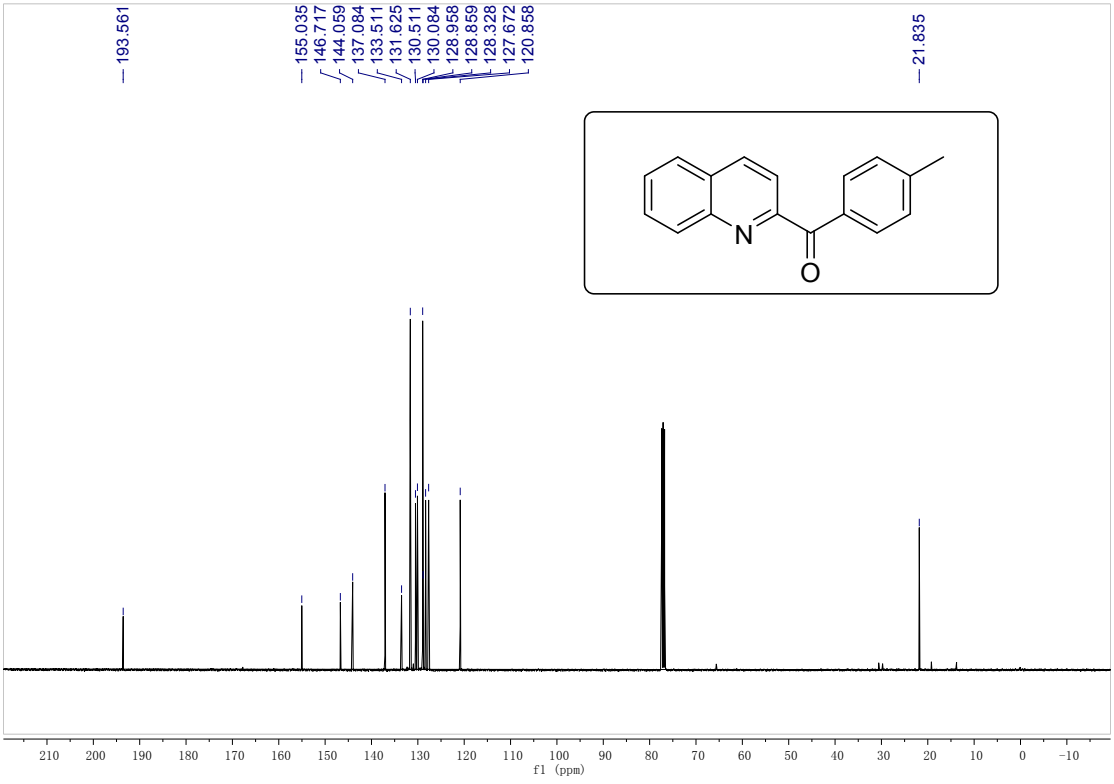
8.316
8.209
8.187
8.152
8.131
8.081
8.060
7.900
7.880
7.795
7.753
7.663
7.623
7.320
7.300
2.447
N
O
1.03
1.01
1.98
1.01
1.01
1.03
1.00
2.02
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


193.561
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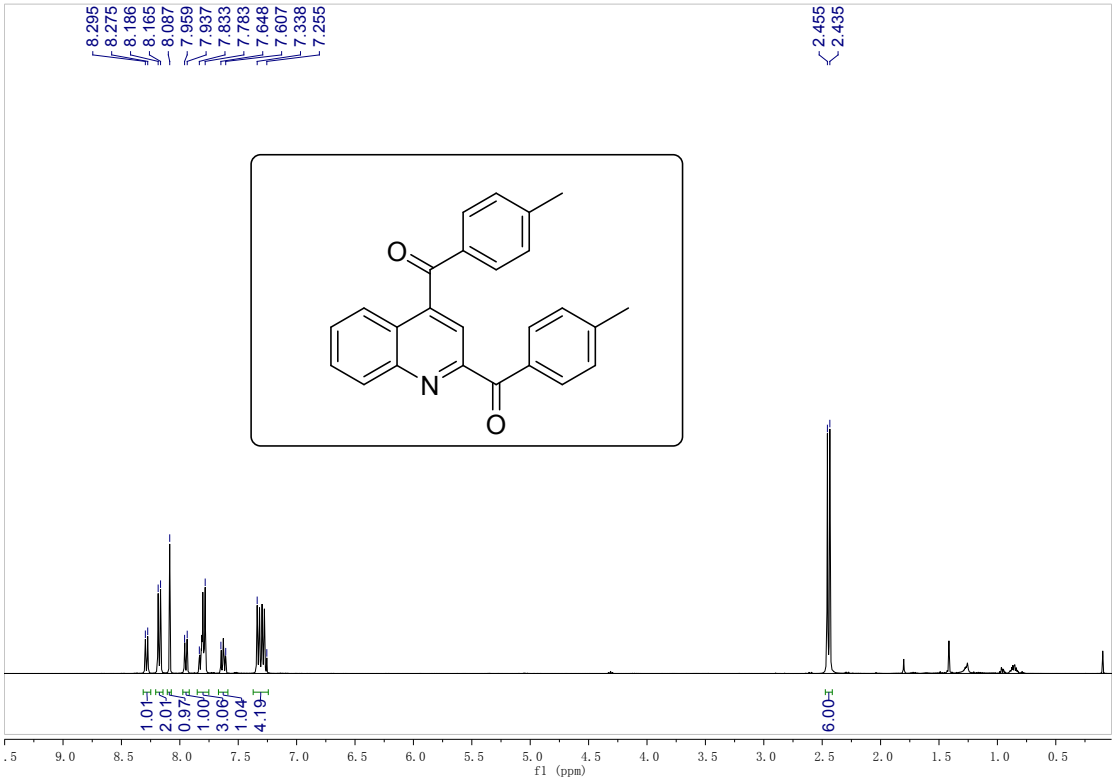
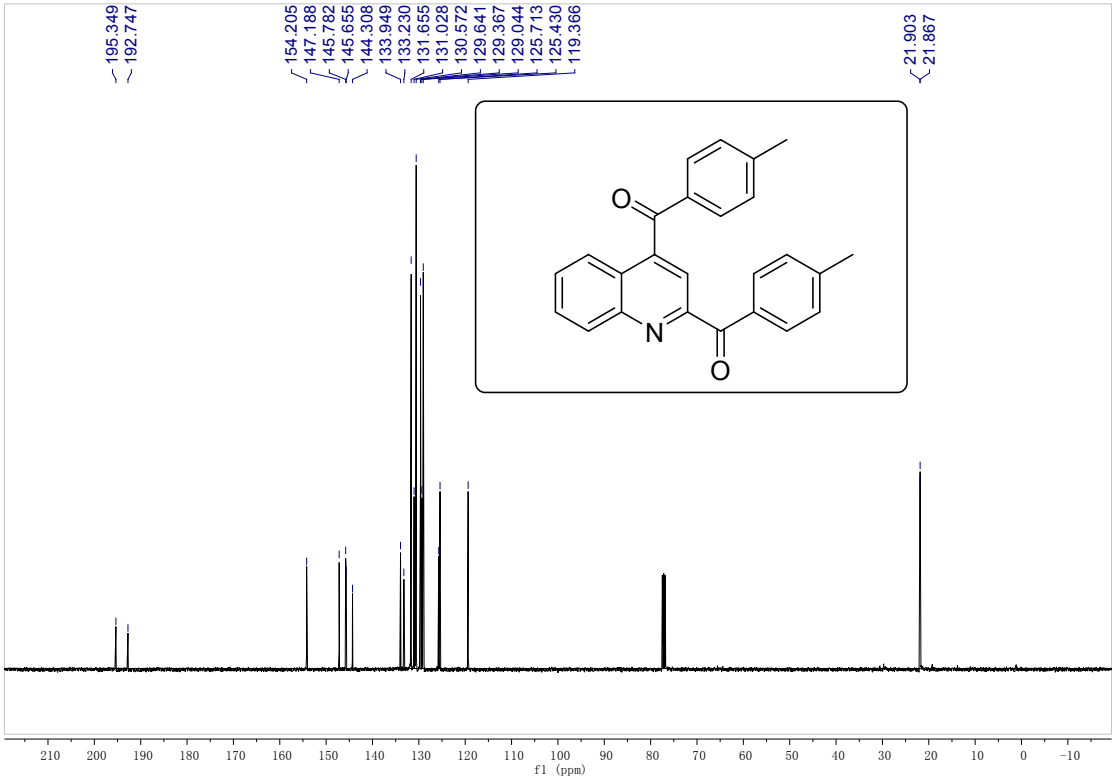
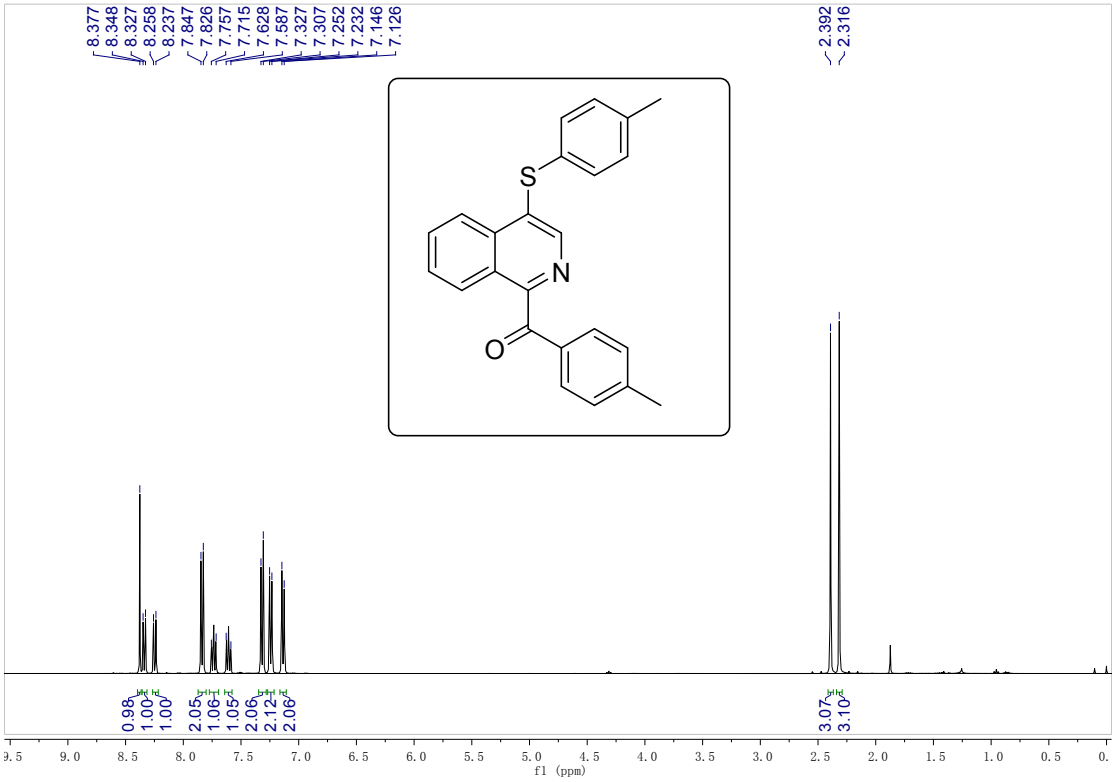
146.717
144.059
137.084
133.511
131.625
130.511
130.084
128.958
128.859
128.328
127.672
120.858
21.835
N
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


8.295
8.275
8.186
8.165
8.087
7.959
7.937
7.833
7.783
7.648
7.607
7.338
7.255
2.455
2.435
O
N
O
1.01
2.01
0.97
1.00
3.06
1.04
4.19
6.00
f1 (ppm)


195.349
192.747
154.205
147.188
145.782
145.655
144.308
133.949
133.230
131.655
131.028
130.572
129.641
129.367
129.044
125.713
125.430
119.366
21.903
21.867
O
N
O
f1 (ppm)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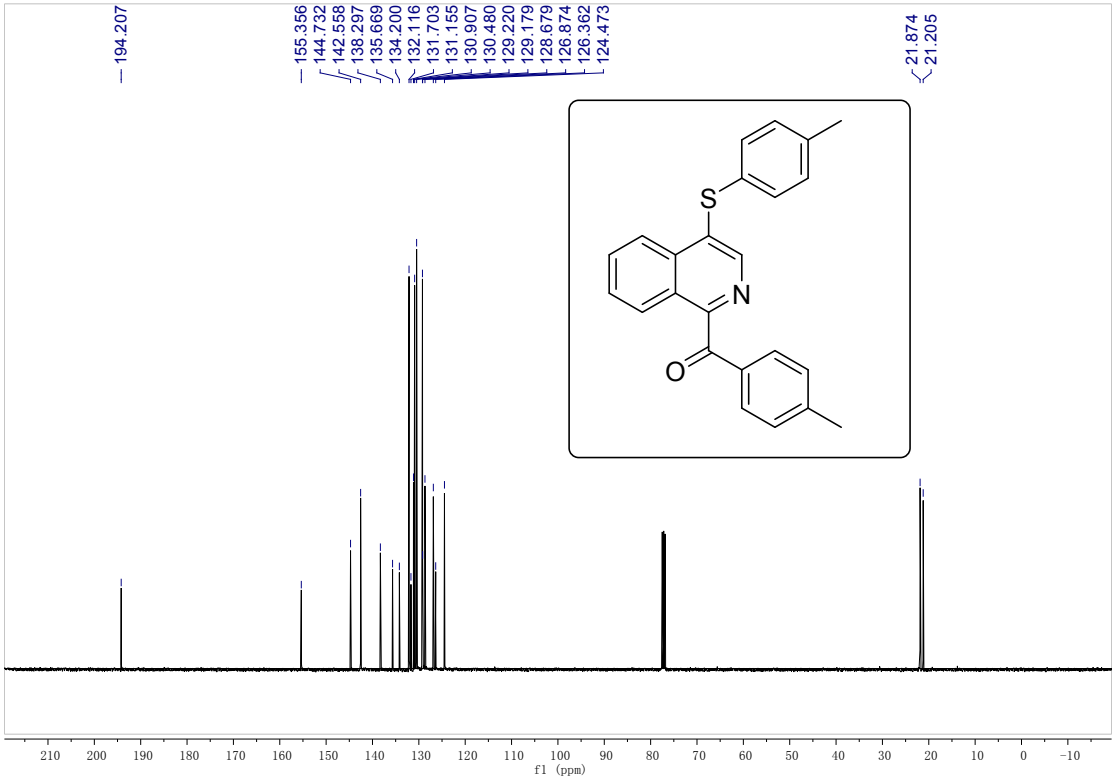
8.348
8.327
8.258
8.237
7.847
7.826
7.757
7.715
7.628
7.587
7.327
7.307
7.252
7.232
7.146
7.126
2.392
2.316
S
N
O
0.98
1.00
1.00
2.05
1.06
1.05
2.06
2.12
2.06
3.07
3.10
f1 (ppm)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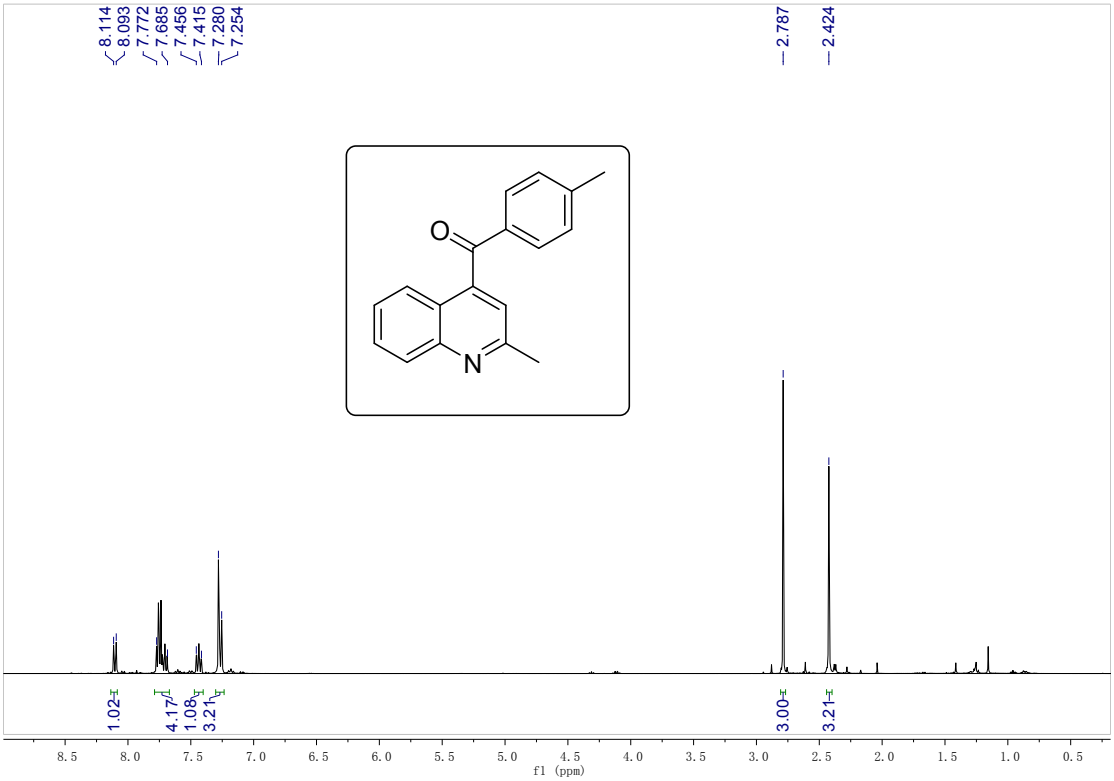
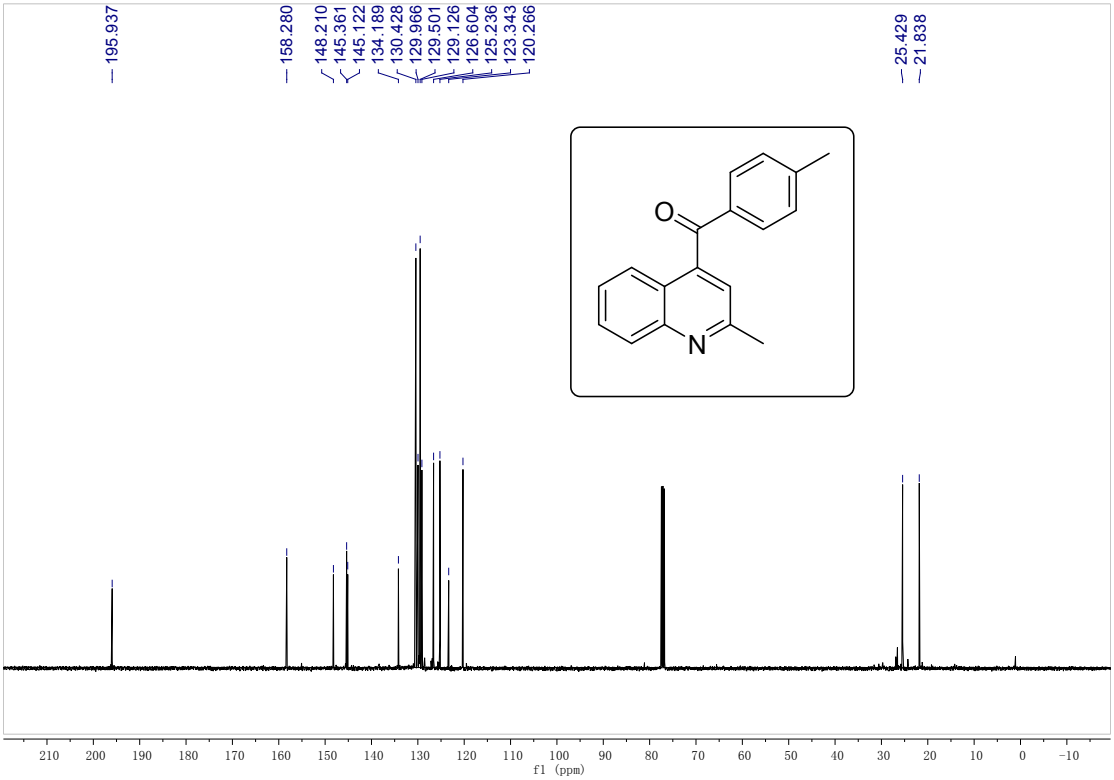
142.558
138.297
135.669
134.200
132.116
131.703
131.155
130.907
130.480
129.220
129.179
128.679
126.874
126.362
124.473
21.874
21.205
S
N
O
f1 (ppm)


8.114
8.093
7.772
7.685
7.456
7.415
7.280
7.254
2.787
2.424
O
N
1.02
4.17
1.08
3.21
3.00
3.21
f1 (ppm)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145.361
145.122
134.189
130.428
129.966
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129.126
126.604
125.236
123.343
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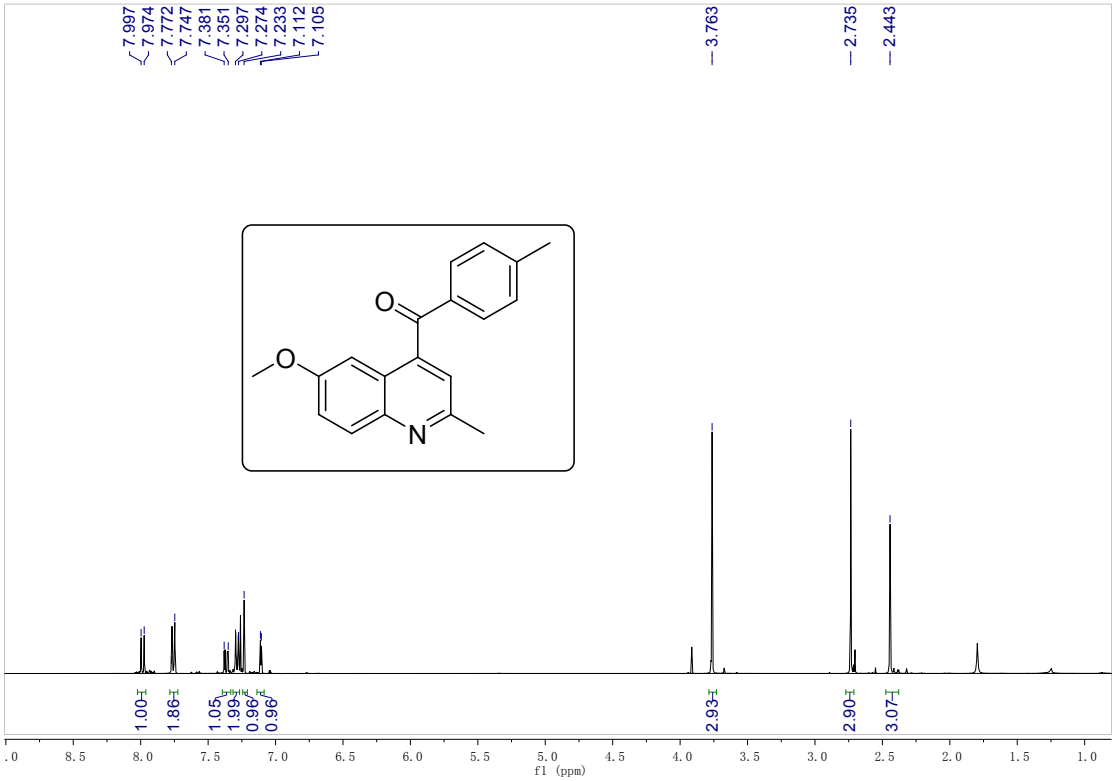
25.429
21.838
O
N
f1 (ppm)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7.974
7.772
7.747
7.381
7.351
7.297
7.274
7.233
7.112
7.105
3.763
2.735
2.443
O
O
N
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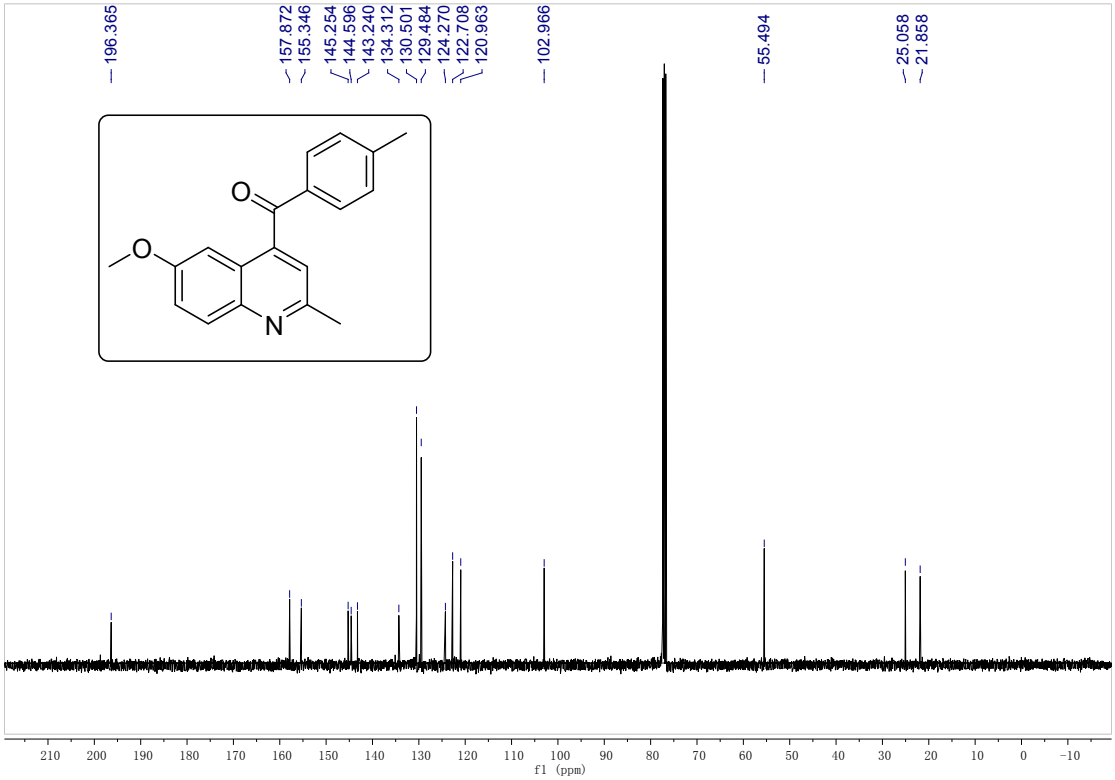
1.86
1.05
1.99
0.96
0.96
2.93
2.90
3.07
f1 (ppm)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157.872
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144.596
143.240
134.312
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129.484
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122.708
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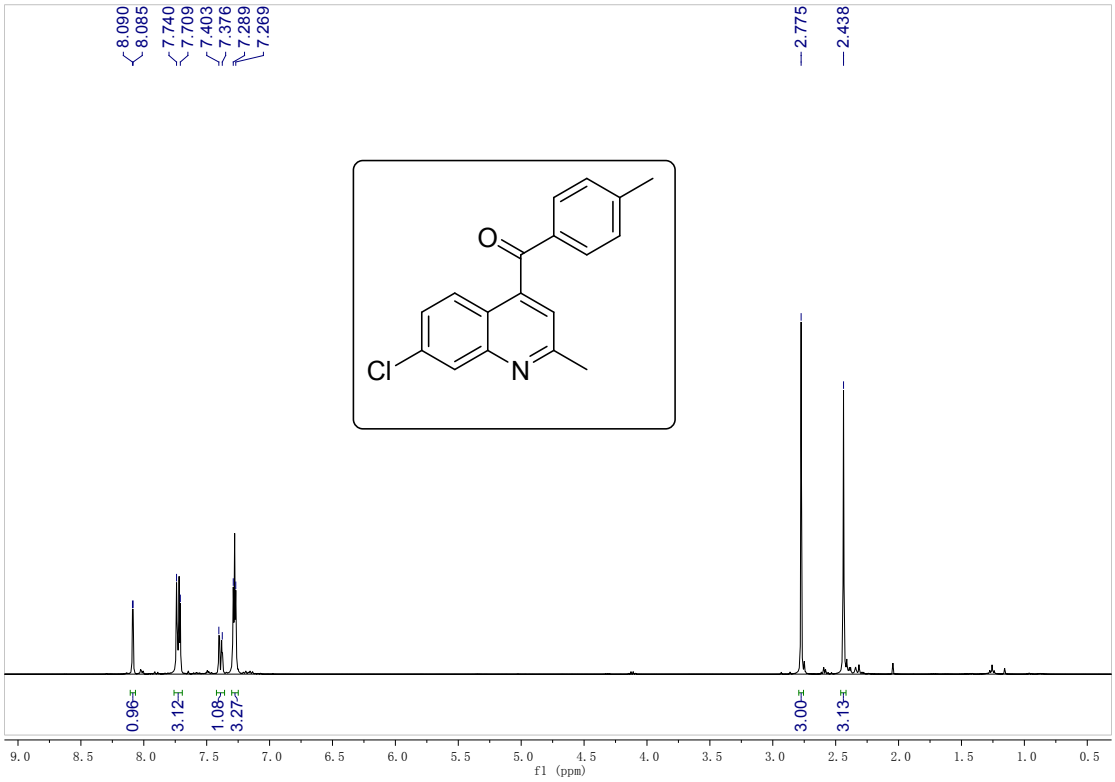
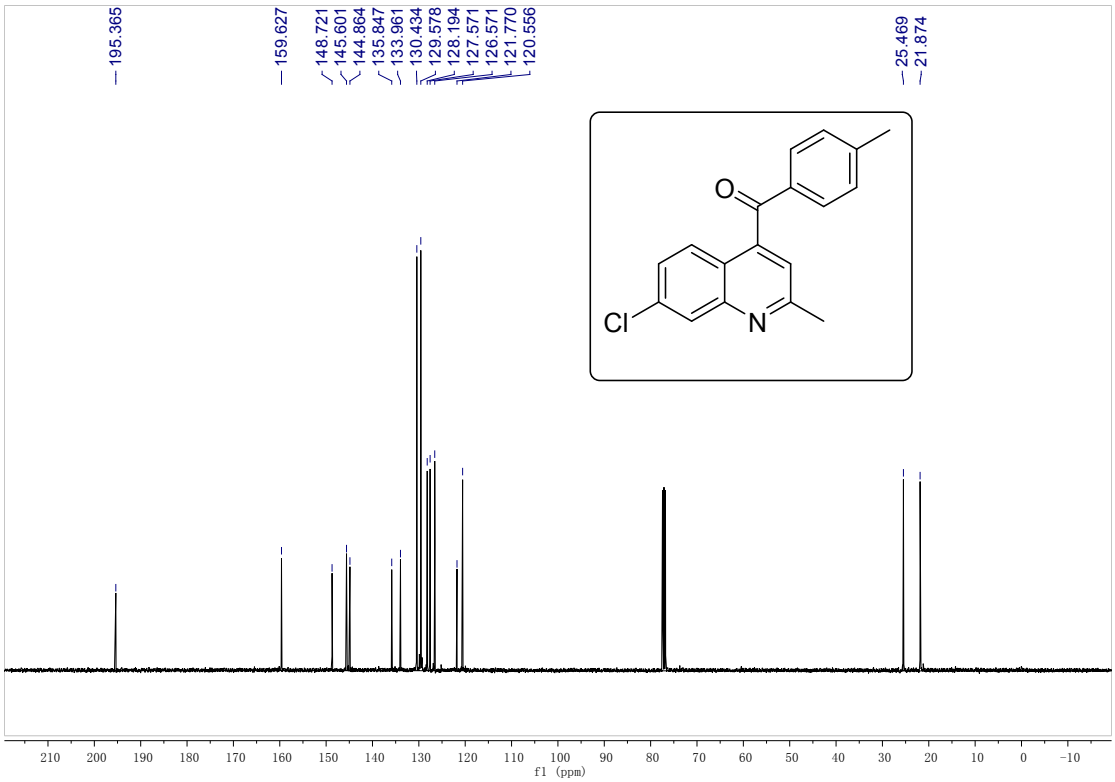
O
N
f1 (ppm)


8.090
8.085
7.740
7.709
7.403
7.376
7.289
7.269
2.775
2.438
O
Cl
N
0.96
3.12
1.08
3.27
3.00
3.13
f1 (ppm)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159.627
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145.601
144.864
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133.961
130.434
129.578
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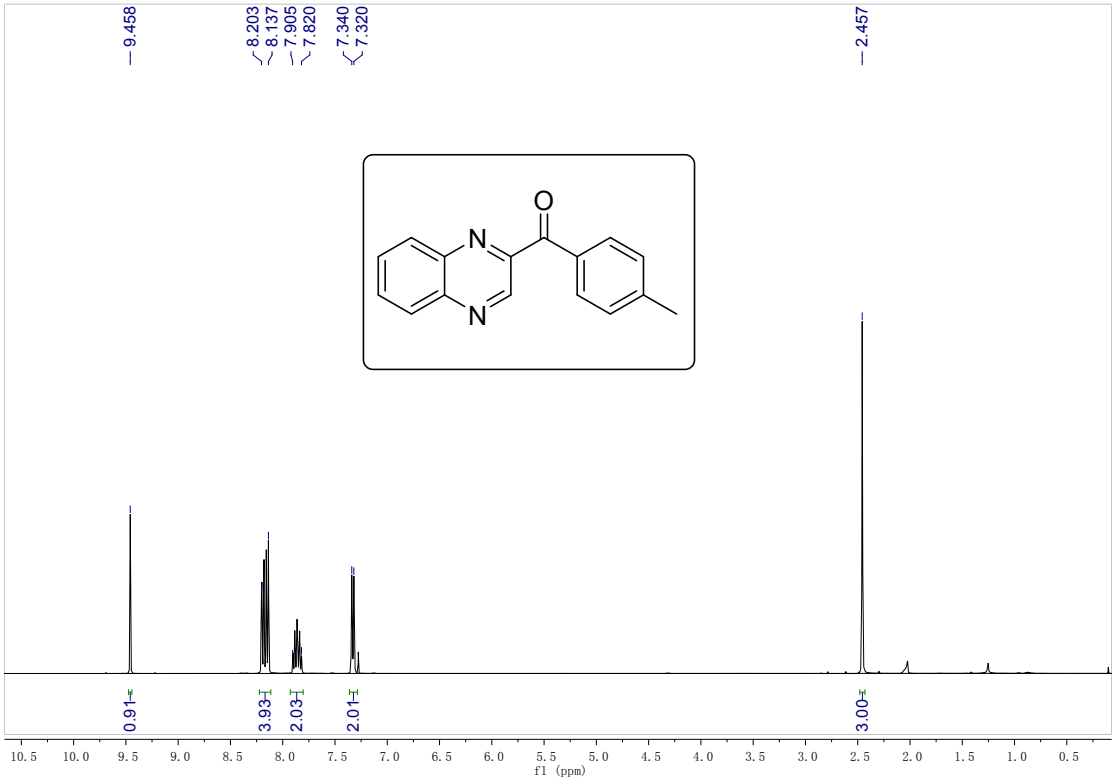
121.770
120.556
25.469
21.874
O
Cl
N
f1 (ppm)


9.458
8.203
8.137
7.905
7.820
7.340
7.320
2.457
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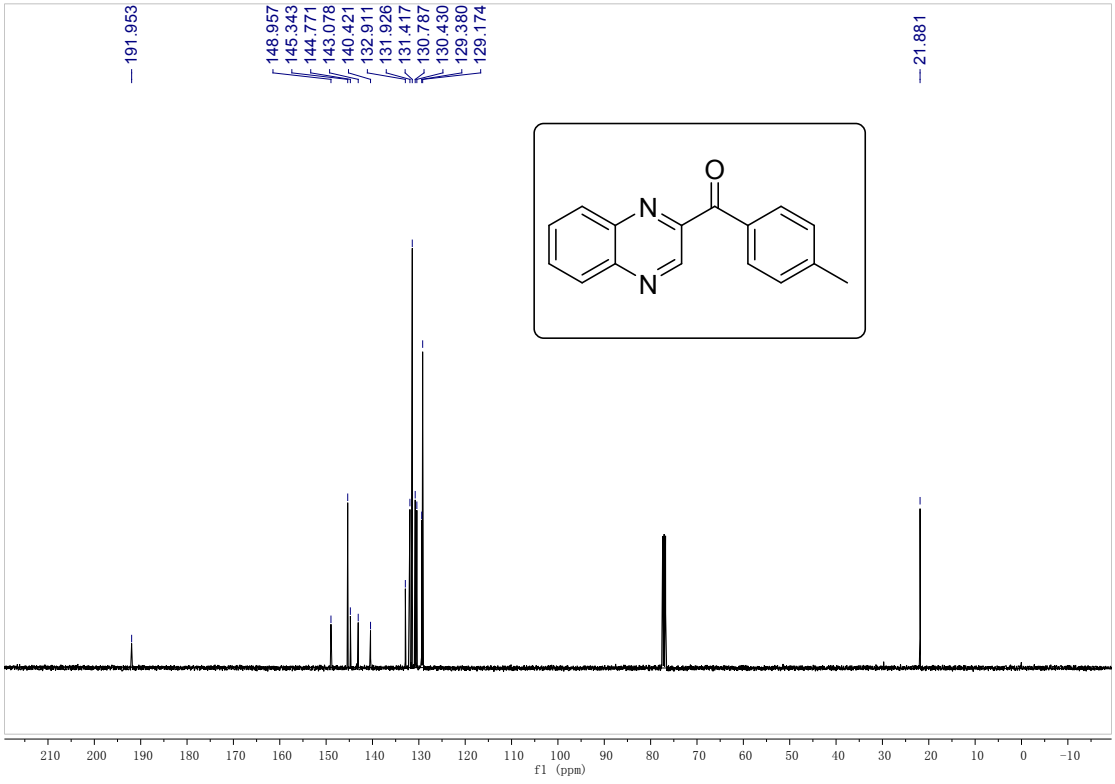
N
N
0.91
3.93
2.03
2.01
3.00
f1 (ppm)


191.953
148.957
145.343
144.771
143.078
140.421
132.911
131.926
131.417
130.787
130.430
129.380
129.174
21.881
O
N
N
f1 (ppm)