Electronic supplementary materials *Mendeleev Commun.*, 2025, **35**, 530–532

One-pot synthesis of acetyl(iso)quinolines/pyridines employing the sodium-promoted Claisen condensation of the corresponding carboxylates

Xin He, Tian Yuan, Jie Sui, Shuhan Dong, Ziyi Wang, Di Liu and Yi Zou

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1. General

Information

All reactions were carried out under air. Melting points were measured with the YRT-3 melting point apparatus. NMR spectra were recorded on a Bruker Avance spectrometer operating at 400 MHz (^{1}H NMR) and 101 MHz (^{13}C NMR) in $CDCl_3$. All ^{1}H NMR chemical shifts were reported in ppm and were referenced to the residual peaks of $CDCl_3$ at 7.26, coupling constants *J* were given in Hz. The following abbreviations are used to describe peak patterns where appropriate: singlet (s), doubet (d), doublet of doublets (dd), triplet (t), multiplet (m), and broad resonances (br). Mass spectra (MS) were obtained on a Waters Q-TOF micro TM apparatus. The thin layer chromatography (TLC) was performed on pre-coated, glass-backed silica gel plates. Visualization of the developed chromatogram was performed by UV absorbance (254 nm).

Chemicals

Unless noted otherwise, the materials obtained from commercial suppliers were used without further purification, substrates **1b, 1c, 1d, 1e, 1f, 1g, 1i, 1j, 1k, 1l, 1m** were prepared according to reference [G. P. Shen, J. J. Jiang, F. Sun, X. Shen, D. R. Zhu, and X. Q. Liu, *J. Heterocyclic Chem.,* 2013, **50**, 1152]. All solvents were analytical purity.

2. Experimental procedure

2.1 General procedure for the synthesis of 2.

To a 25 mL flask equipped with a mechanical agitator and condenser, compound **1** (5.0 mmol) and dry ethyl acetate (0.89 g, 10.0 mmol) were added. Metallic sodium (0.14 g, 6.0 mmol) was then sliced and carefully introduced into the flask. A noticeable increase in the temperature was observed, after which the reaction mixture was allowed to cool naturally to room temperature and stirred for 4 hours. Once the reaction was complete, as confirmed by TLC, 0.1 mL of absolute ethanol was added, and stirring continued for an additional 10 minutes.

Subsequently, 25% sulfuric acid (3 mL) was added to the reaction flask. The mixture was heated to reflux for 3 hours. After cooling, the solvent was evaporated under reduced pressure. The residue was treated with 1 mL of water, and the pH was adjusted to 8-9 using a 30% sodium hydroxide solution. The aqueous layer was extracted with ethyl acetate (3 × 2 mL), and the combined organic phases were dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure, and the crude product was then purified *via* column chromatography to afford the desired product **2**.

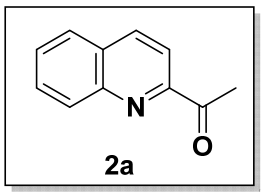
2.2 Gram-scale experiment for industrial production of 2a

To a 250 mL flask equipped with a mechanical agitator and condenser, compound **1a** (10 g, 50 mmol) and ethyl acetate after dehydration treatment (10 mL, 100 mmol) were added. Metallic sodium (1.4 g, 60 mmol) was then sliced and carefully introduced into the flask. A noticeable increase in the temperature was observed, after which the reaction mixture was allowed to cool naturally to room temperature and stirred for 4 hours. Once the reaction was complete, as confirmed by TLC, 1 mL of absolute ethanol was added, and stirring continued for an additional 10 minutes.

Subsequently, 25% sulfuric acid (30 mL) was added to the reaction flask. The mixture was heated to reflux for 3 hours. After cooling, the solvent was evaporated under reduced pressure. The residue was treated with 10 mL of water, and the pH was adjusted to 8-9 using a 30% sodium hydroxide solution. The aqueous layer was extracted with ethyl acetate (3 × 20 mL), and the combined organic phases were dried over anhydrous sodium sulfate. Following solvent removal under reduced pressure, the crude product was obtained, which was further purified by recrystallization in 95% ethanol to give the desired product **2a** as a yellow needle crystal, achieving a yield of 92.3%.

3. Characterization of all products

1-(Quinolin-2-yl)ethan-1-one **2a**



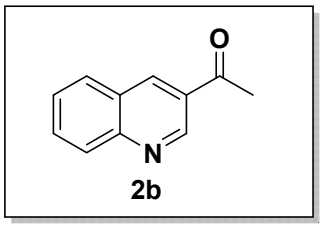
Obtained as a yellow solid (786 mg, 92%); m.p. 51.7-52.3°C.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.29 (d, J = 8.5 Hz, 1H), 8.22 (d, J = 8.5 Hz, 1H), 8.15 (d, J = 8.5 Hz, 1H), 7.92 – 7.87 (m, 1H), 7.81 (ddd, J = 8.4, 6.8, 1.5 Hz, 1H), 7.70 – 7.65 (m, 1H), 2.90 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 200.75, 153.23, 147.24, 136.91, 130.58, 130.02, 129.59, 128.59, 127.68, 117.99, 25.63.

MS (ESI): $[M+H]^+$ calcd. for $C_{11}H_{10}NO$,172.1; found.172.0.

1-(Quinolin-3-yl)ethan-1-one **2b**



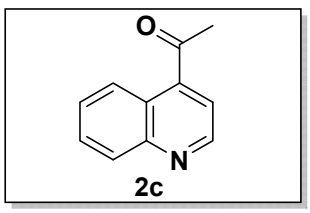
Obtained as a white solid (717 mg, 84%); m.p. 85.9-87.2°C.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.31 (t, J = 1.0 Hz, 1H), 8.51 (d, J = 1.1 Hz, 1H), 8.08 (dq, J = 7.6, 0.8 Hz, 1H), 8.04 – 7.99 (m, 1H), 7.82 – 7.74 (m, 2H), 2.86 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 196.84, 149.85, 149.26, 137.43, 132.10, 129.52, 129.41, 129.27, 127.64, 126.86, 26.91.

MS (ESI): $[M+H]^+$ calcd. for $C_{11}H_{10}NO$,172.1; found.172.5

1-(Quinolin-4-yl)ethan-1-one **2c**



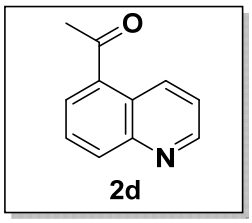
Obtained as a slight yellow oil (715 mg, 84%).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.01 (d, J = 4.4 Hz, 1H), 8.45 (dd, J = 8.5, 1.4 Hz, 1H), 8.19 – 8.13 (m, 1H), 7.76 (ddd, J = 8.4, 6.9, 1.4 Hz, 1H), 7.66-7.59 (m, 2H), 2.74 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 201.36, 149.84, 149.15, 142.55, 129.94, 128.38, 128.15, 125.51, 123.63, 119.91, 30.10.

MS (ESI): $[M+H]^+$ calcd. for $C_{11}H_{10}NO$,172.1; found. 172.0.

1-(Quinolin-5-yl)ethan-1-one **2d**



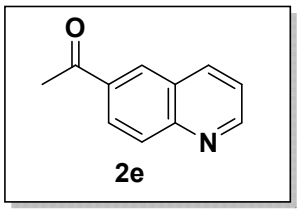
Obtained as a colorless oil (687 mg, 80%).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.21 (ddd, J = 8.8, 1.7, 0.9 Hz, 1H), 8.95 (dd, J = 4.2, 1.7 Hz, 1H), 8.28 (dt, J = 8.4, 1.1 Hz, 1H), 8.07 (dd, J = 7.2, 1.2 Hz, 1H), 7.75 (dd, J = 8.5, 7.3 Hz, 1H), 7.52 (dd, J = 8.8, 4.2 Hz, 1H), 2.77 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 200.67, 150.80, 148.38, 134.82, 134.71, 129.70, 127.89, 125.94, 122.91, 29.63.

MS (ESI): $[M+H]^+$ calcd. for $C_{11}H_{10}NO$,172.1; found. 172.4.

1-(Quinolin-6-yl)ethan-1-one **2e**



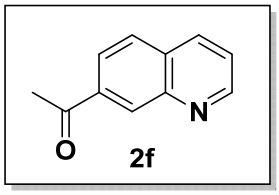
Obtained as a slight yellow solid (724 mg, 85%). m.p. 71.9-73.2°C.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.03 (dd, J = 4.2, 1.7 Hz, 1H), 8.47 (d, J = 2.0 Hz, 1H), 8.29 (td, J = 8.8, 8.3, 1.9 Hz, 2H), 8.17 (dd, J = 8.8, 0.8 Hz, 1H), 7.51 (dd, J = 8.3, 4.2 Hz, 1H), 2.76 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.67, 150.80, 148.38, 134.82, 134.71, 129.70, 127.89, 125.94, 122.91, 29.63.

MS (ESI): $[M+H]^+$ calcd. for $C_{11}H_{10}NO$,172.1; found. 172.4.

1-(Quinolin-7-yl)ethan-1-one **2f**



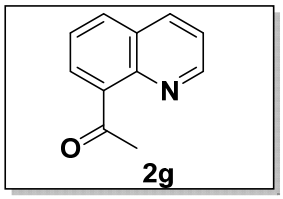
Obtained as a slight yellow solid (713 mg, 83%).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.01 (dd, J = 4.2, 1.7 Hz, 1H), 8.70 – 8.68 (m, 1H), 8.21 (ddd, J = 8.3, 1.7, 0.9 Hz, 1H), 8.13 (dd, J = 8.6, 1.8 Hz, 1H), 7.89 (d, J = 8.5 Hz, 1H), 7.52 (dd, J = 8.3, 4.2 Hz, 1H), 2.77 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 197.50, 152.69, 150.14, 137.60, 134.89, 130.09, 129.91, 127.75, 127.47, 122.01, 26.85.

MS (ESI): $[M+H]^+$ calcd. for $C_{11}H_{10}NO$,172.1; found. 172.1.

1-(Quinolin-8-yl)ethan-1-one **2g**



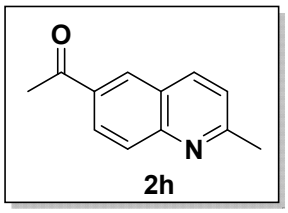
Obtained as a yellow solid (689 mg, 81%); m.p. 38.9-39.2°C.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.00 (dd, J = 4.2, 1.8 Hz, 1H), 8.22 (dd, J = 8.3, 1.8 Hz, 1H), 7.96 (d, J = 7.6 Hz, 2H), 7.61 (t, J = 7.7 Hz, 1H), 7.48 (dd, J = 8.3, 4.2 Hz, 1H), 2.97 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 198.00, 151.46, 147.79, 137.52, 135.88, 131.55, 130.84, 128.36, 124.40, 123.03, 26.79.

MS (ESI): $[M+H]^+$ calcd. for $C_{11}H_{10}NO$,172.1; found. 172.6.

1-(2-Methylquinolin-6-yl)ethan-1-one **2h**



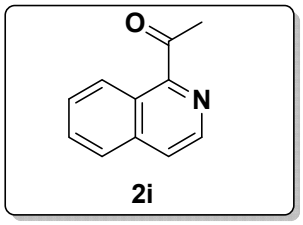
Obtained as a white solid (732 mg, 79%). m.p. 60.3-61.7°C.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.42 (d, J = 2.0 Hz, 1H), 8.25 (dd, J = 8.8, 2.0 Hz, 1H), 8.18 (d, J = 8.4 Hz, 1H), 8.07 (d, J = 8.8 Hz, 1H), 7.38 (d, J = 8.4 Hz, 1H), 2.79 (s, 3H), 2.74 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 197.60, 161.85, 149.82, 137.60, 134.17, 129.63, 129.15, 127.87, 125.65, 123.01, 26.81, 25.63.

MS (ESI): $[M+H]^+$ calcd. for $C_{12}H_{12}NO$,186.1; found 186.0.

1-(Isoquinolin-1-yl)ethan-1-one **2i**



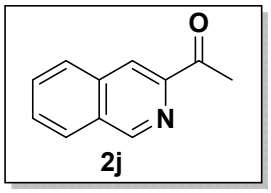
Obtained as a slightly yellow oil (745 mg, 87%).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.98 (ddd, J = 7.7, 1.9, 0.8 Hz, 1H), 8.60 (d, J = 5.5 Hz, 1H), 7.89 – 7.86 (m, 1H), 7.83 (dd, J = 5.5, 0.9 Hz, 1H), 7.76 – 7.67 (m, 2H), 2.89 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 202.70, 152.80, 141.05, 137.05, 130.34, 129.13, 126.97, 126.87, 125.74, 124.63, 28.59.

MS (ESI): $[M+H]^+$ calcd. for $C_{11}H_{10}NO$,172.1; found. 172.1.

1-(Isoquinolin-3-yl)ethan-1-one **2j**



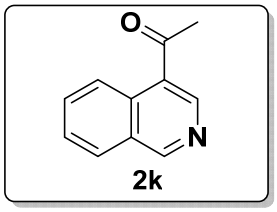
Obtained as an off-white solid (647 mg, 76%). m.p. 85.5-86.7°C.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.31 (t, J = 1.0 Hz, 1H), 8.51 (d, J = 1.1 Hz, 1H), 8.08 (dq, J = 7.6, 0.8 Hz, 1H), 8.04 – 7.99 (m, 1H), 7.82 – 7.74 (m, 2H), 2.86 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 200.37, 151.97, 147.69, 135.50, 131.04, 130.16, 129.49, 128.67, 127.60, 120.33, 26.63.

MS (ESI): $[M+H]^+$ calcd. for $C_{11}H_{10}NO$,172.1; found. 172.4.

1-(Isoquinolin-4-yl)ethan-1-one **2k**



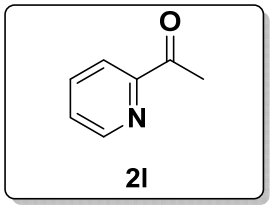
Obtained as a yellow solid (750 mg, 88%). m.p. 71.5-72.7°C.

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.34 (s, 1H), 9.04 (s, 1H), 8.87 (d, J = 8.7 Hz, 1H), 8.01 (d, J = 8.2 Hz, 1H), 7.86 – 7.79 (m, 1H), 7.67 (t, J = 7.6 Hz, 1H), 2.78 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ (ppm): 200.13, 156.85, 145.86, 132.77, 132.73, 128.71, 128.20, 127.91, 127.75, 125.43, 29.59.

MS (ESI): $[M+H]^+$ calcd. for $C_{11}H_{10}NO$,172.1; found. 172.4.

1-(Pyridin-2-yl)ethan-1-one **2l**



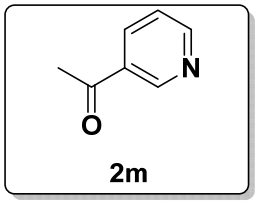
Obtained as a colorless oil (540 mg, 89%).

^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 8.73 (ddd, J = 4.8, 1.7, 1.0 Hz, 1H), 8.03 – 7.93 (m, 2H), 7.66 (ddd, J = 7.3, 4.7, 1.5 Hz, 1H), 2.64 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ (ppm): 199.93, 153.43, 149.67, 137.93, 128.14, 121.59, 26.09.

MS (ESI): $[M+H]^+$ calcd. for C_7H_8NO,122.1; found. 122.5.

1-(Pyridin-3-yl)ethan-1-one **2m**



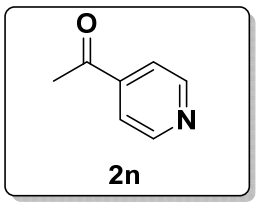
Obtained as a slightly yellow oil (558 mg, 92%).

^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 9.11 (dd, J = 2.3, 0.9 Hz, 1H), 8.78 (dd, J = 4.8, 1.7 Hz, 1H), 8.26 (ddd, J = 8.0, 2.3, 1.7 Hz, 1H), 7.54 (ddd, J = 7.9, 4.8, 0.9 Hz, 1H), 2.62 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ (ppm): 197.79, 153.82, 149.95, 135.95, 132.41, 124.25, 27.30.

MS (ESI): $[M+H]^+$ calcd. for C_7H_8NO, 122.1; found.122.3.

1-(Pyridin-4-yl)ethan-1-one **2n**



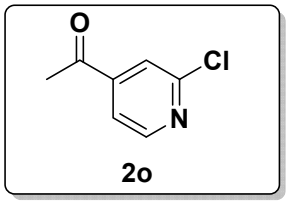
Obtained as a colorless oil (496 mg, 82%).

^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 8.84 – 8.75 (m, 2H), 7.82 – 7.76 (m, 2H), 2.62 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ (ppm): 198.48, 151.23, 142.95, 121.71, 27.24.

MS (ESI): $[M+H]^+$ calcd. for C_7H_8NO, 122.1; found.122.6.

***1-(2-Chloropyridin-4-yl)ethan-1-one* 2o**



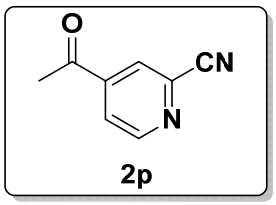
Obtained as a white solid (611 mg, 79%). m.p.36.1-36.6C.

^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 8.98 (dd, J = 5.0, 0.9 Hz, 1H), 8.47 (dd, J = 1.7, 0.9 Hz, 1H), 8.13 (dd, J = 5.1, 1.7 Hz, 1H), 2.67 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ (ppm): 197.08, 151.91, 151.56, 146.39, 122.79, 121.13, 27.48.

MS (ESI): $[M+H]^+$ calcd. for C_7H_7ClNO,156.0; found.155.4.

***4-Acetylpicolinonitrile* 2p**



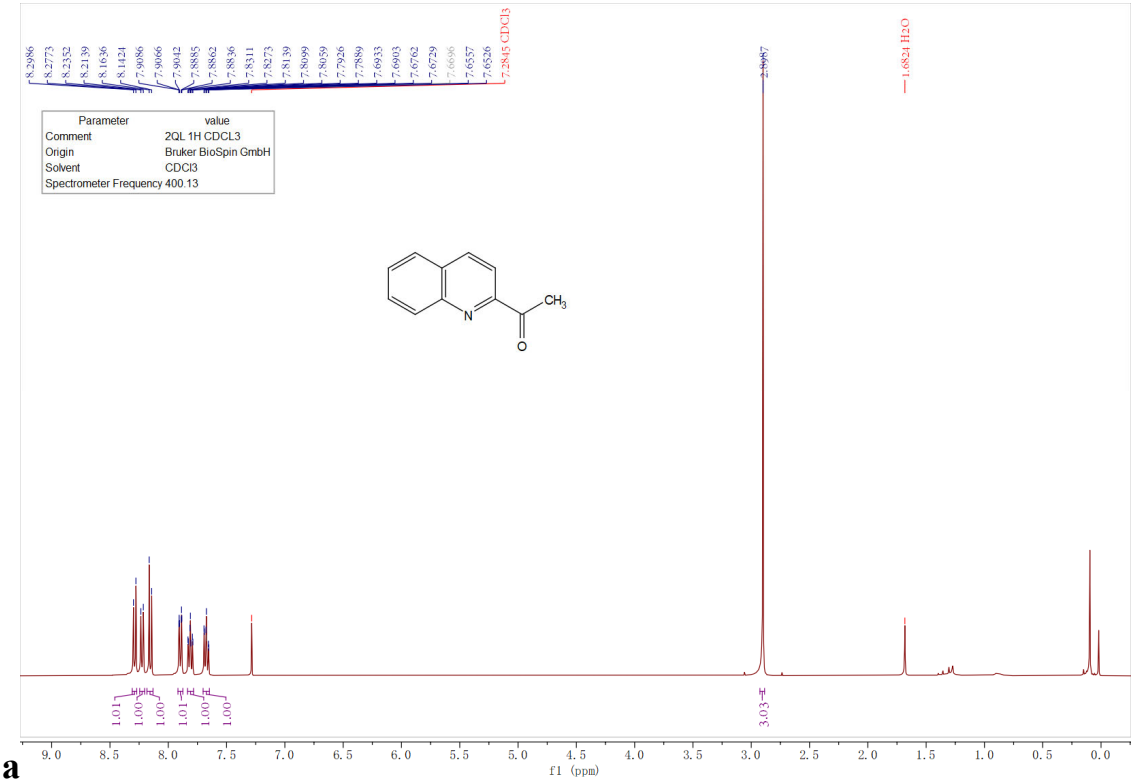
Obtained as a white solid (585 mg, 80%). m.p.100.9-101.1C.

^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 9.00-8.96 (m, 1H), 8.46 (dt, J = 1.6, 0.7 Hz, 1H), 8.12 (dd, J = 5.0, 1.7 Hz, 1H), 2.67 (s, 3H).

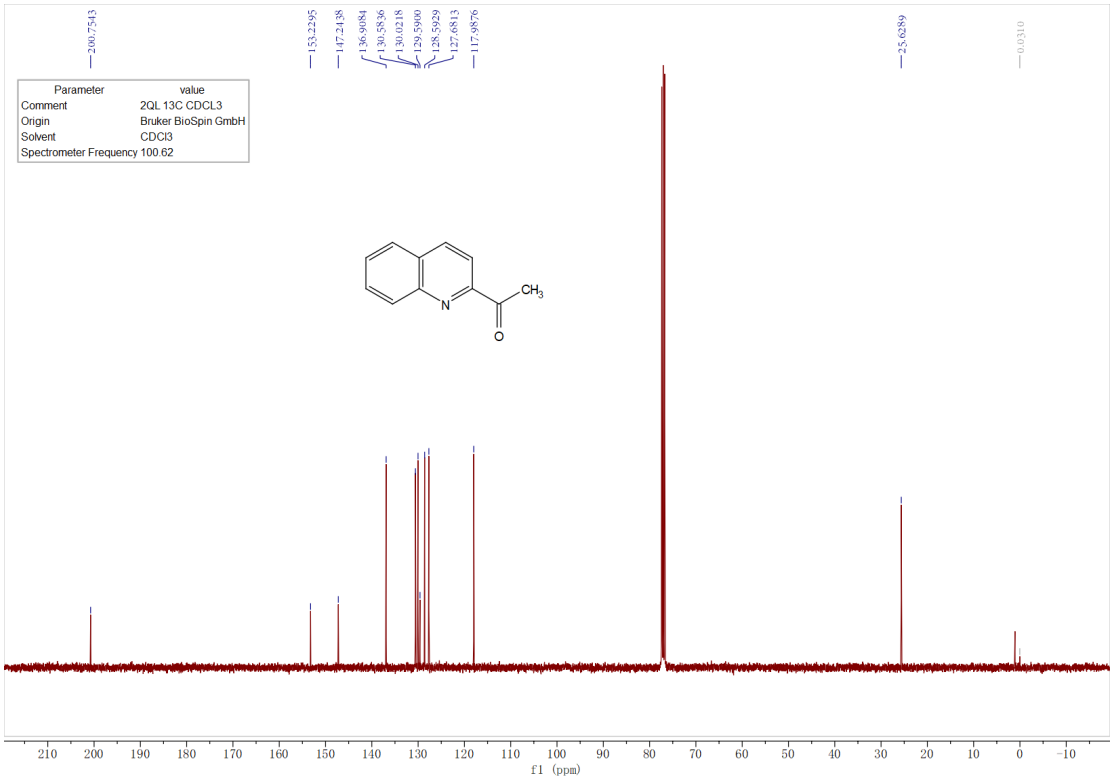
^{13}C NMR (101 MHz, DMSO-d_6) δ (ppm): 196.90, 152.94, 144.27, 134.19, 127.27, 125.63, 117.57, 27.43.

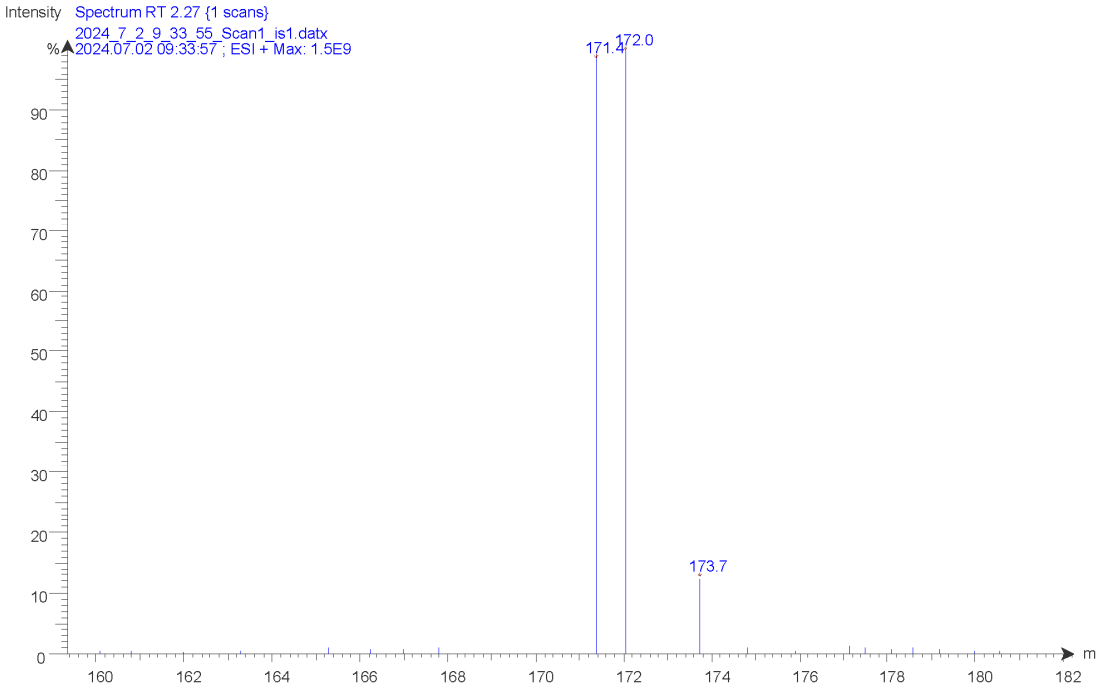
MS (ESI): $[M+NH_4]^+$ calcd. for $C_8H_9N_3O$,164.1; found.164.4.

4. ^{1}H NMR, 13C NMR and MS spectra of all products



2a




Intensity
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%
171.4
172.0
173.7
m
0
10
20
30
40
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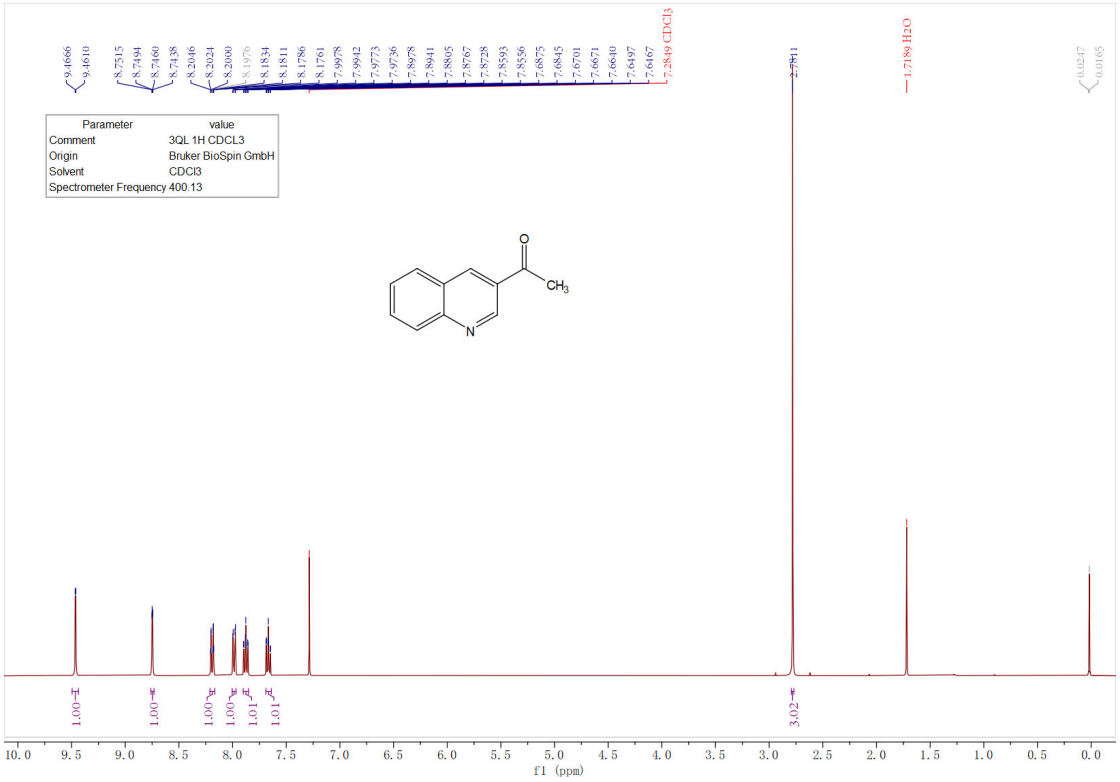
60
70
80
90
160
162
164
166
168
170
172
174
176
178
180
182

2b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8.1786
8.1761
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7.8941
7.8805
7.8767
7.8728
7.8593
7.8556
7.6875
7.6845
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7.6640
7.6497
7.6467
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2.7841
1.7189 H2O
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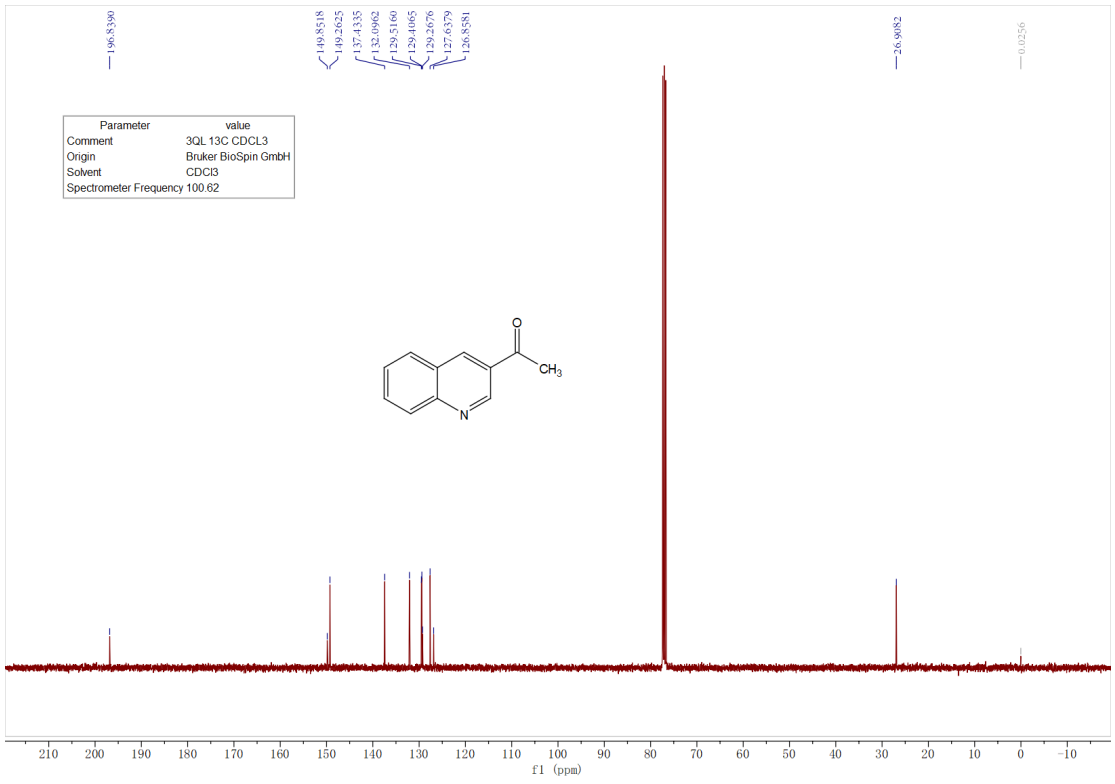
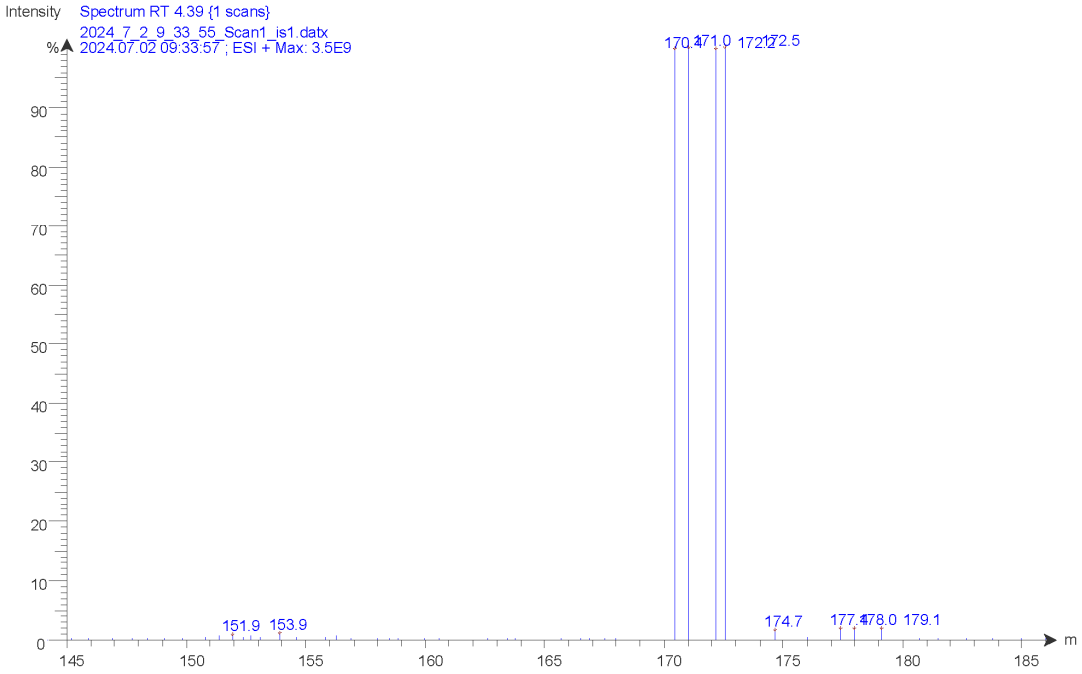
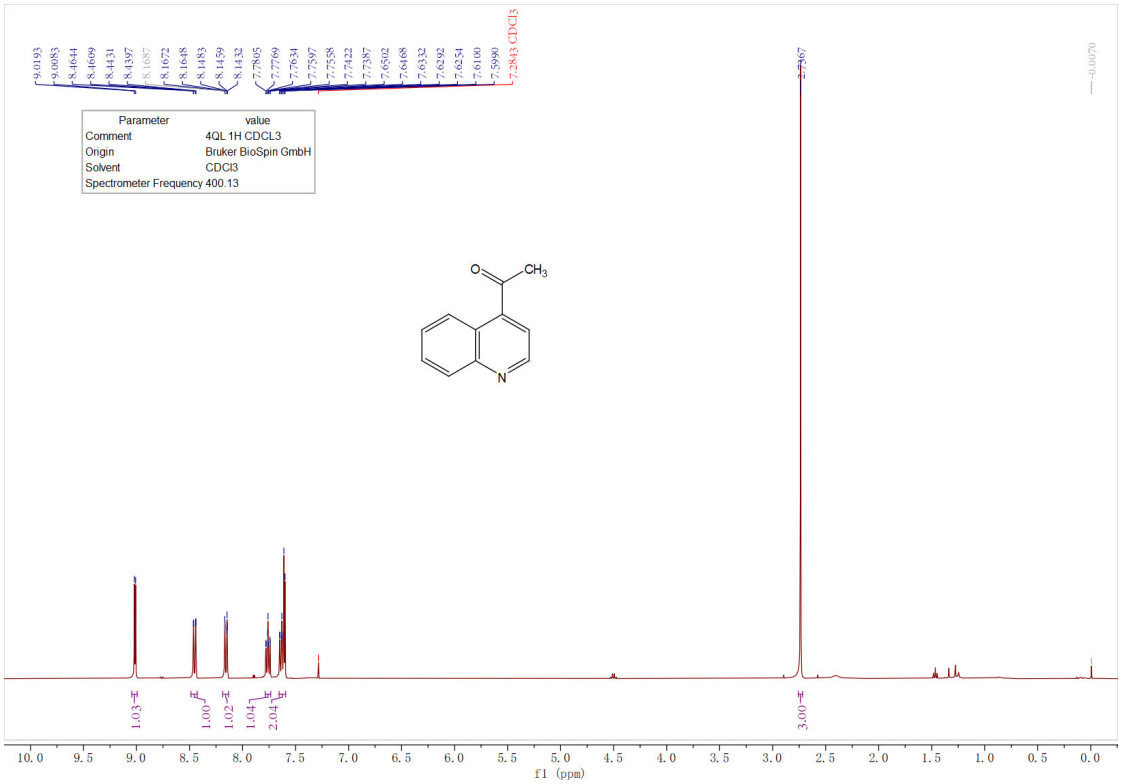
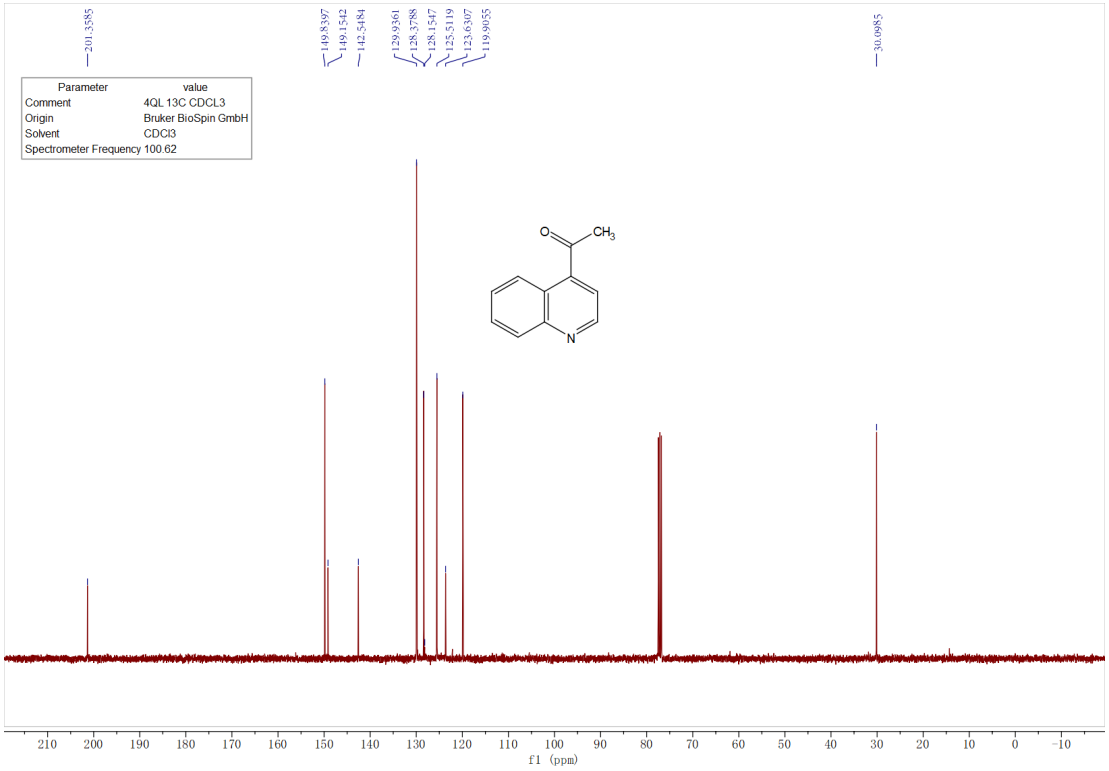
0.0165
Parameter value
Comment 3QL 1H CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 400.13
O
CH3
N
1.00
1.00
1.00
1.00
1.01
1.01
3.02
f1 (ppm)

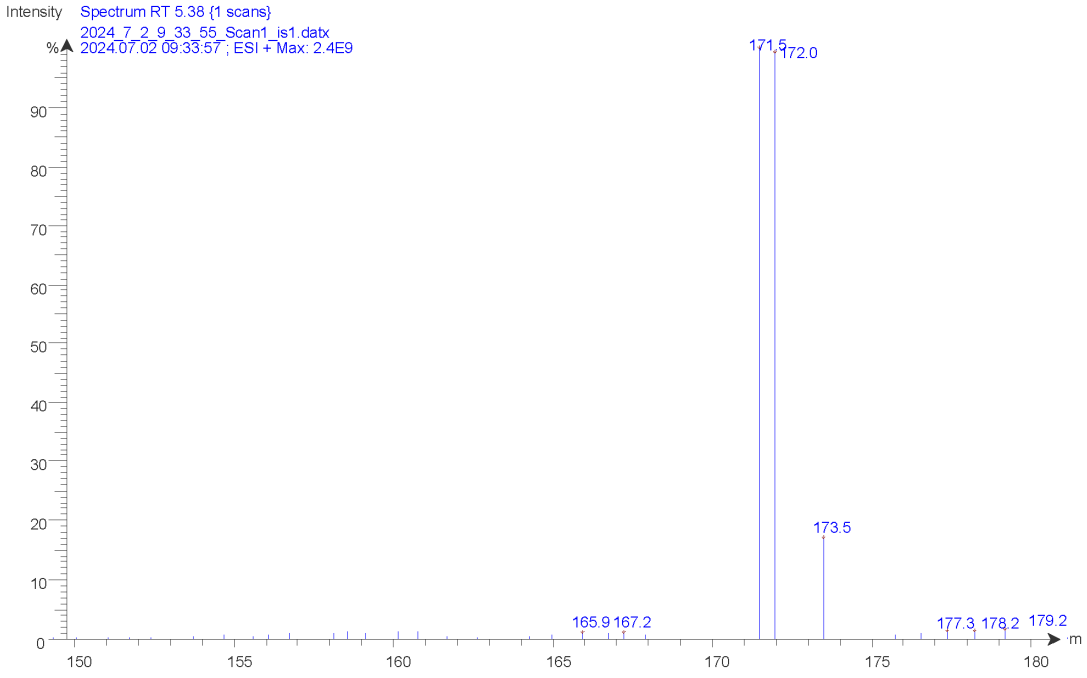

196.8390
149.8518
149.2625
137.4335
132.0962
129.5160
129.4065
129.2676
127.6379
126.8581
26.9082
0.0256
Parameter value
Comment 3QL 13C CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 100.62
O
CH3
N
f1 (ppm)


Intensity
Spectrum RT 4.39 {1 scans}
2024_7_2_9_33_55_Scan1_is1.datx
2024.07.02 09:33:57 ; ESI + Max: 3.5E9
%
90
80
70
60
50
40
30
20
10
0
145
150
155
160
165
170
175
180
185
m
151.9
153.9
174.7
179.1

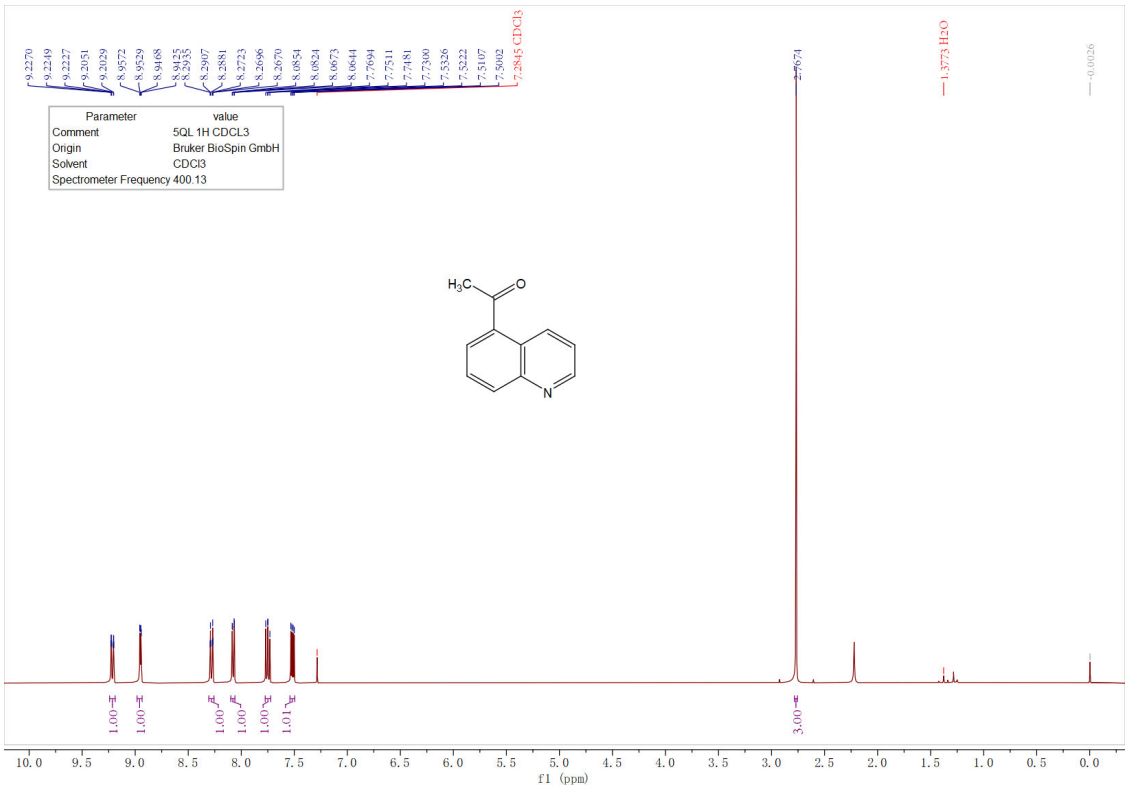
2c

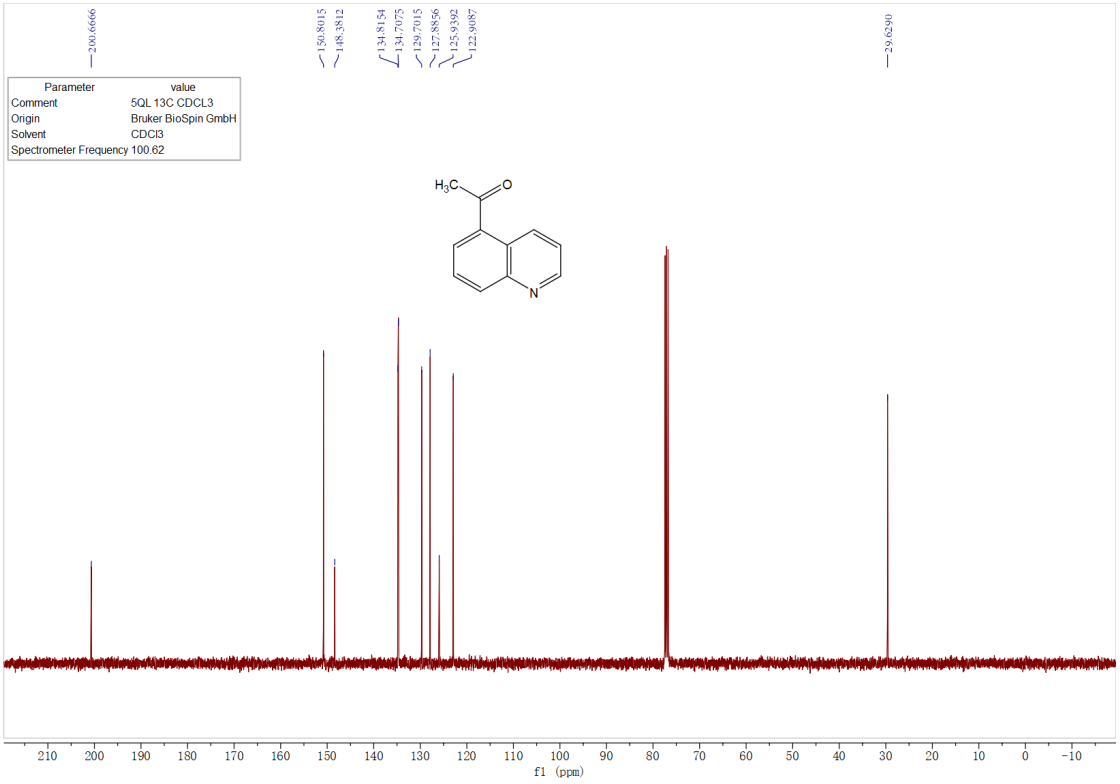

Parameter value
Comment 4QL 1H CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 400.13
f1 (ppm)

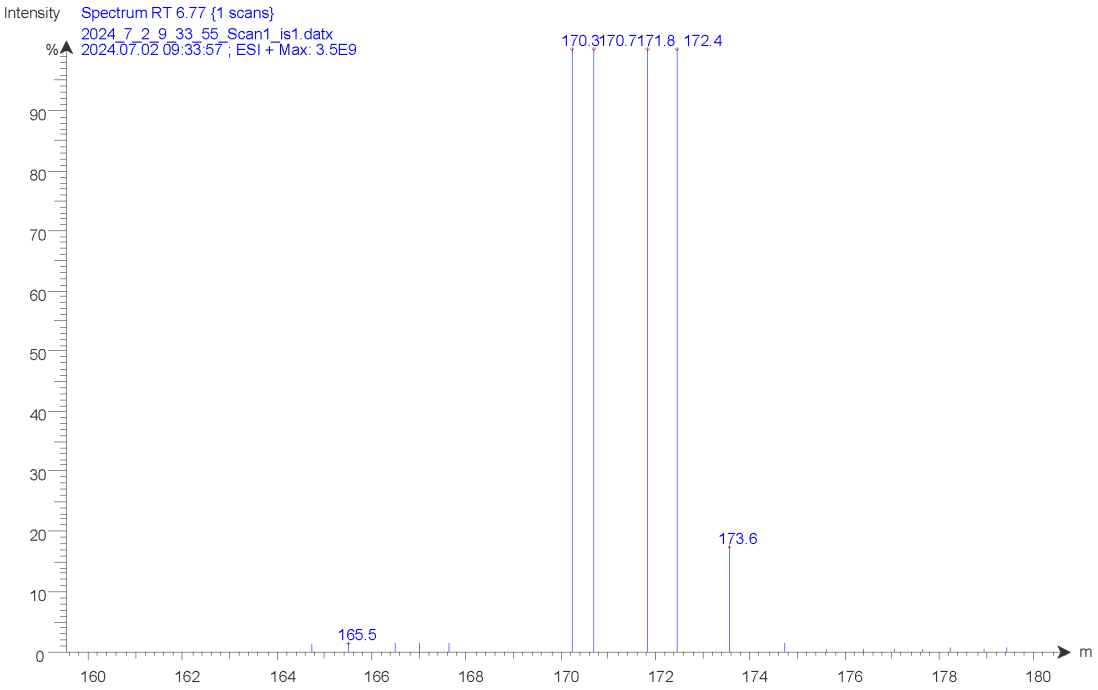

Parameter value
Comment 4QL 13C CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 100.62
f1 (ppm)


Intensity
Spectrum RT 5.38 {1 scans}
2024_7_2_9_33_55_Scan1_is1.datx
2024.07.02 09:33:57 ; ESI + Max: 2.4E9
%
171.5
172.0
173.5
165.9
167.2
177.3
178.2
179.2
m
150
155
160
165
170
175
180

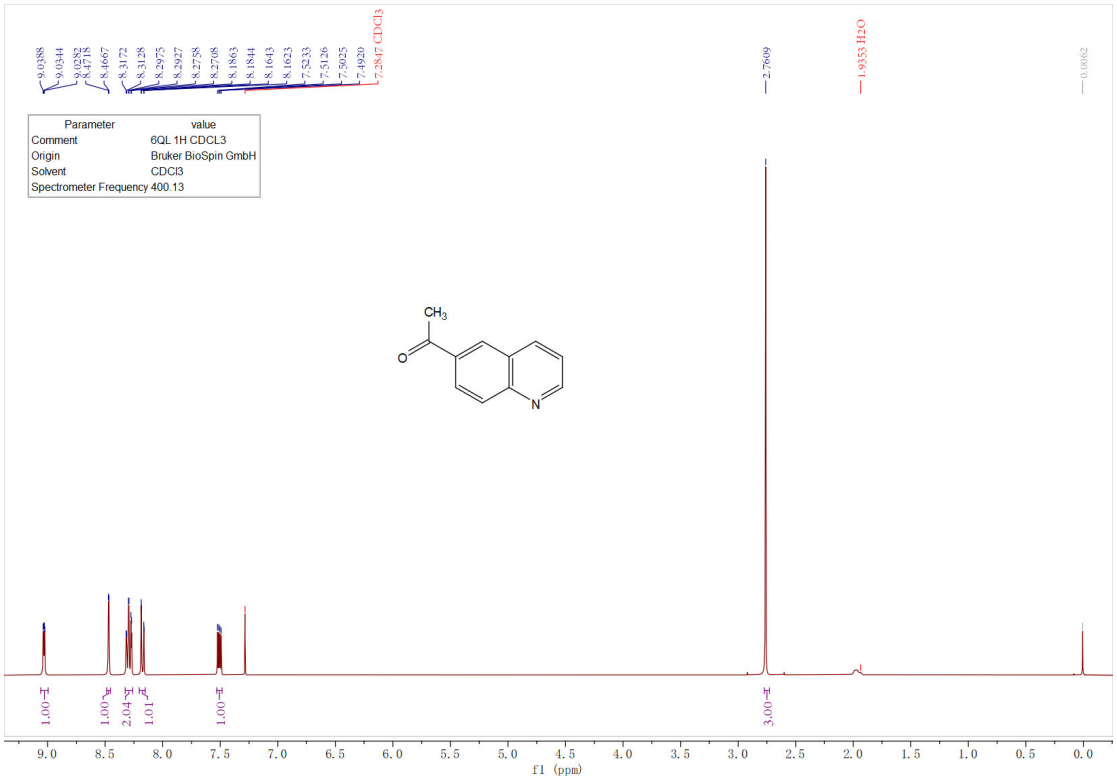
2d

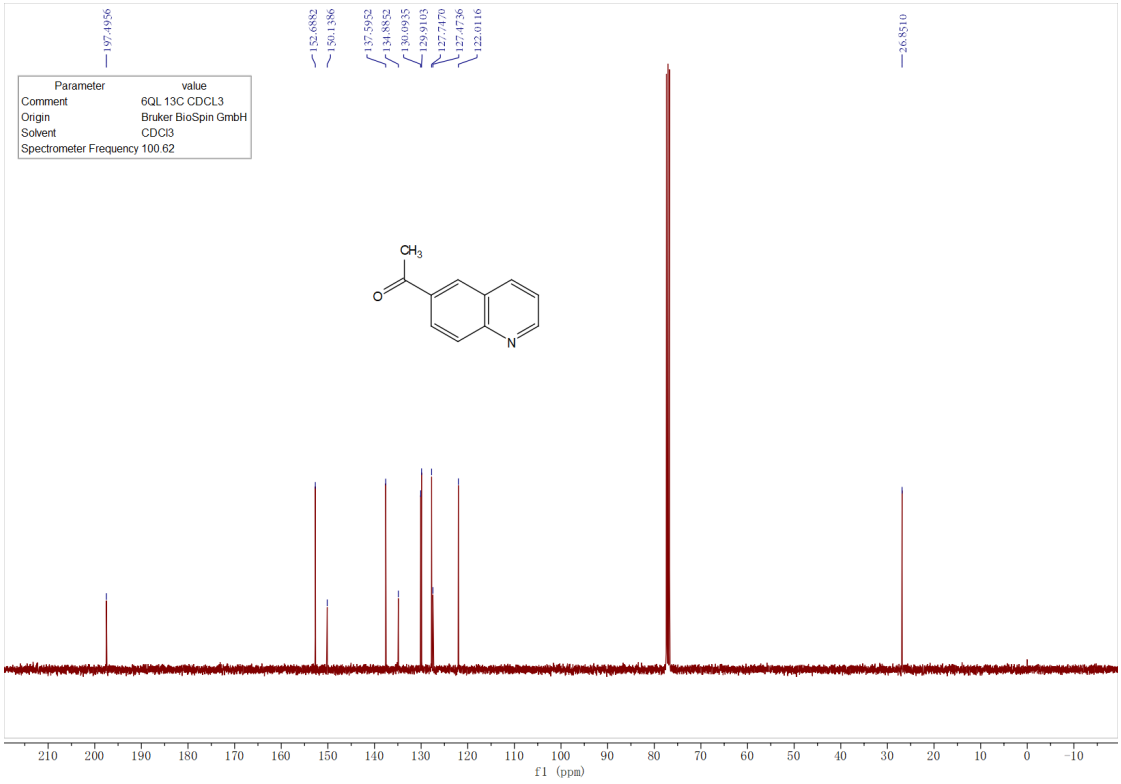

9.2270
9.2249
9.2227
9.2051
9.2029
8.9572
8.9539
8.9468
8.9425
8.9335
8.2907
8.2881
8.2723
8.2696
8.2670
8.0854
8.0824
8.0673
8.0644
7.7694
7.7511
7.7481
7.7300
7.5326
7.5322
7.5107
7.5002
7.2845 CDCl3
2.7674
1.3773 H2O
0.0026
Parameter value
Comment 5QL 1H CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 400.13
H3C
O
N
1.00
1.00
1.00
1.00
1.00
1.01
3.00
10.0 9.5 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 0.0
f1 (ppm)

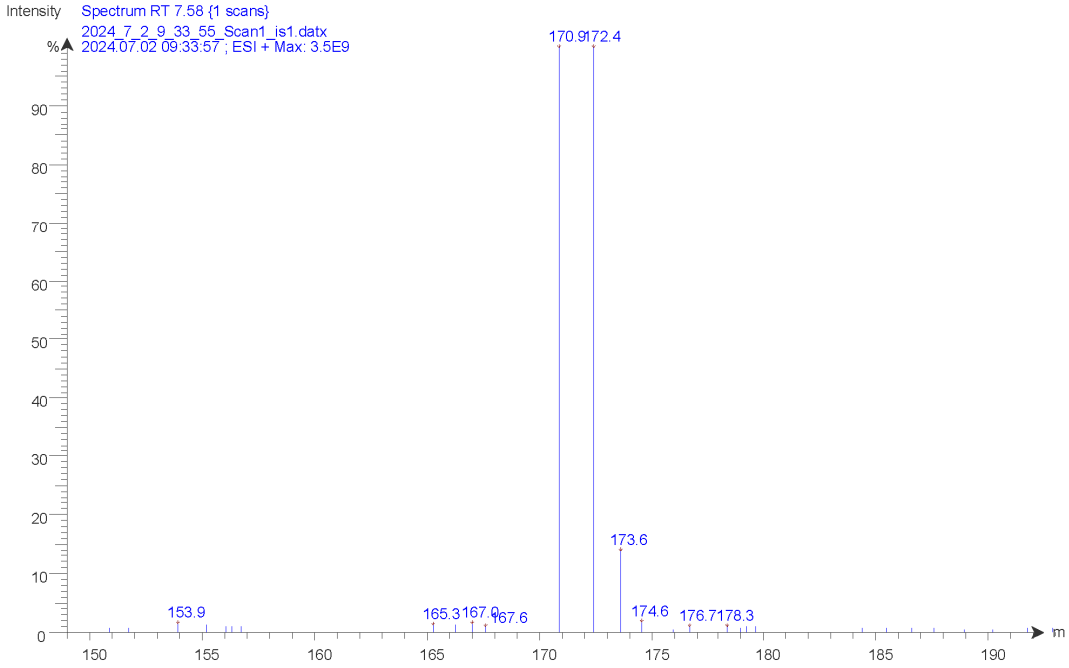

200.6666
150.8015
148.3812
134.8154
134.7075
129.7015
127.8856
125.9392
122.9087
28.6290
Parameter value
Comment 5QL 13C CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 100.62
H3C
O
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Intensity
Spectrum RT 6.77 {1 scans}
2024_7_2_9_33_55_Scan1_is1.datx
2024.07.02 09:33:57 ; ESI + Max: 3.5E9
%
170.3
170.7
171.8
172.4
173.6
165.5
m
0
10
20
30
40
50
60
70
80
90
160
162
164
166
168
170
172
174
176
178
180

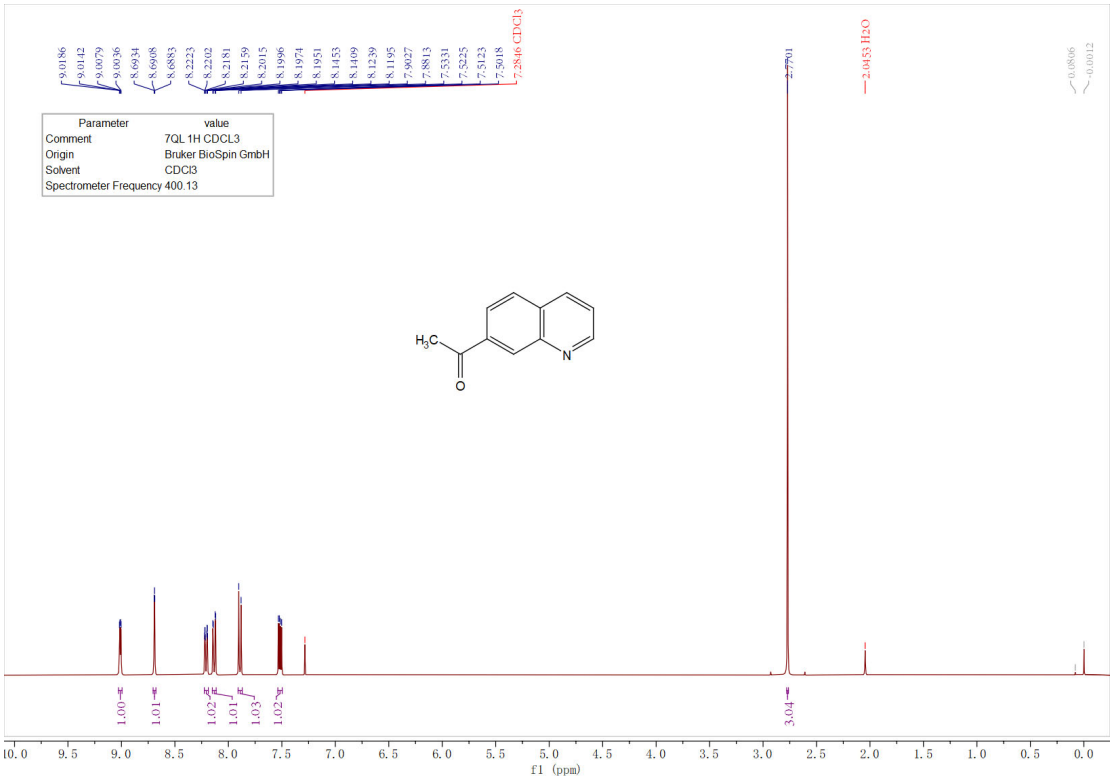
2e

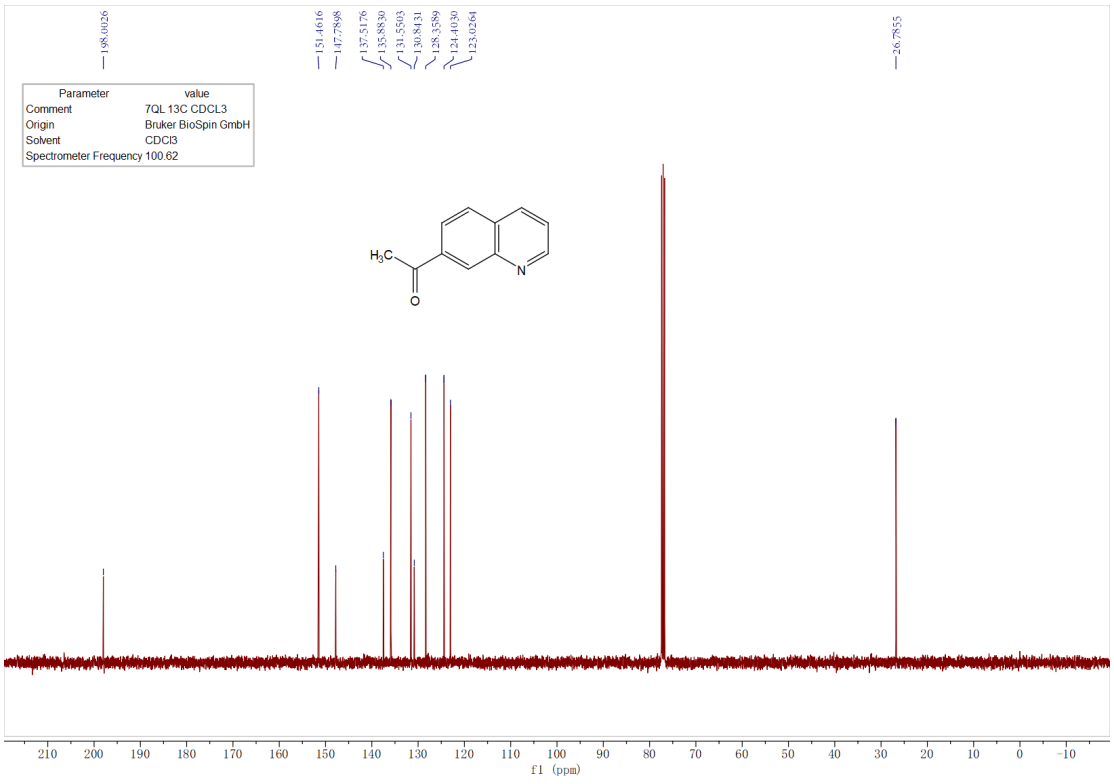

9.0388
9.0344
9.0282
8.4718
8.4667
8.3172
8.3128
8.2975
8.2927
8.2758
8.2708
8.1863
8.1844
8.1643
8.1623
7.5233
7.5126
7.5025
7.4920
7.2847 CDCl3
2.7609
1.9353 H2O
0.0062
Parameter value
Comment 6QL 1H CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 400.13
CH3
O
N
1.00
1.00
2.04
1.01
1.00
3.00
f1 (ppm)

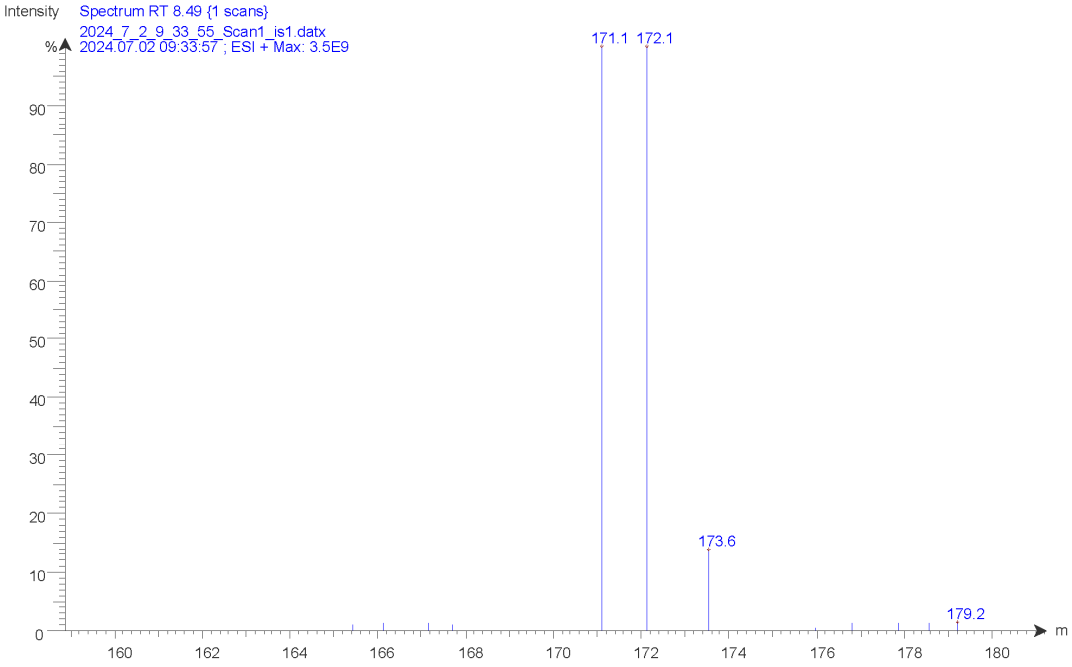

197.4956
152.6882
150.1386
137.5952
134.8852
130.0935
129.9103
127.7470
127.4736
122.0116
26.8510
Parameter value
Comment 6QL 13C CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 100.62
CH3
O
N
f1 (ppm)


Intensity
Spectrum RT 7.58 {1 scans}
2024_7_2_9_33_55_Scan1_is1.datx
2024.07.02 09:33:57 ; ESI + Max: 3.5E9
%
170.9
172.4
173.6
153.9
165.3
167.0
167.6
174.6
176.7
178.3
m
150
155
160
165
170
175
180
185
190

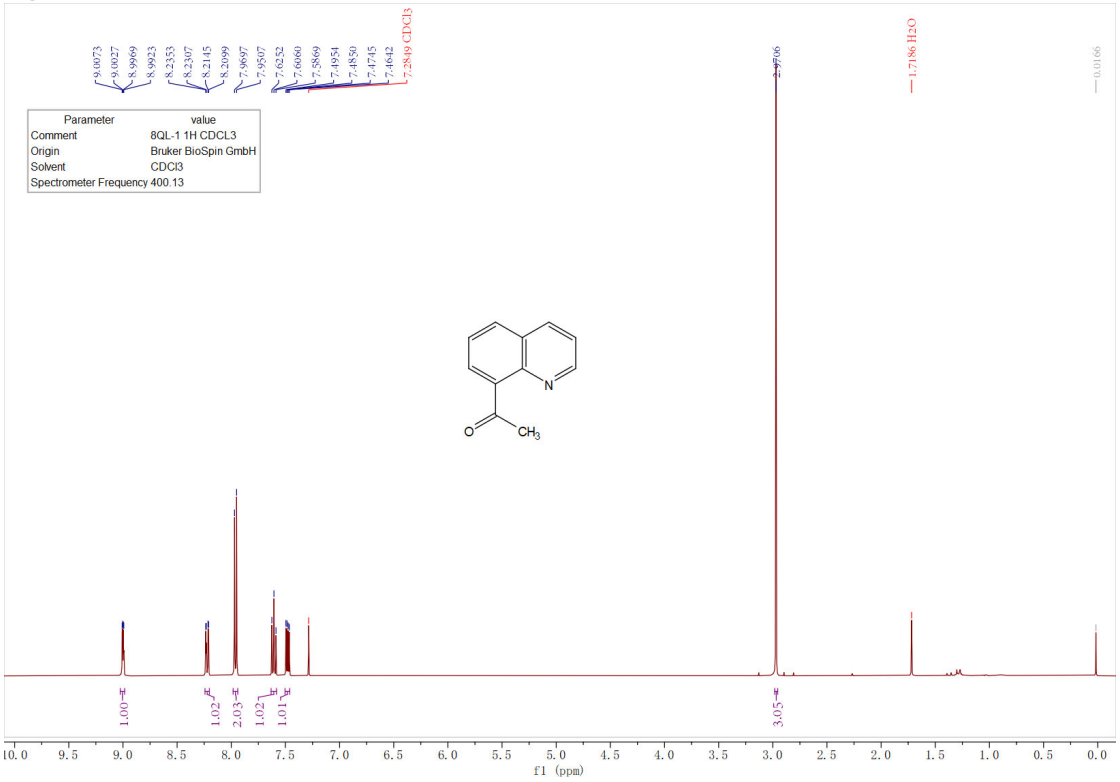
2f

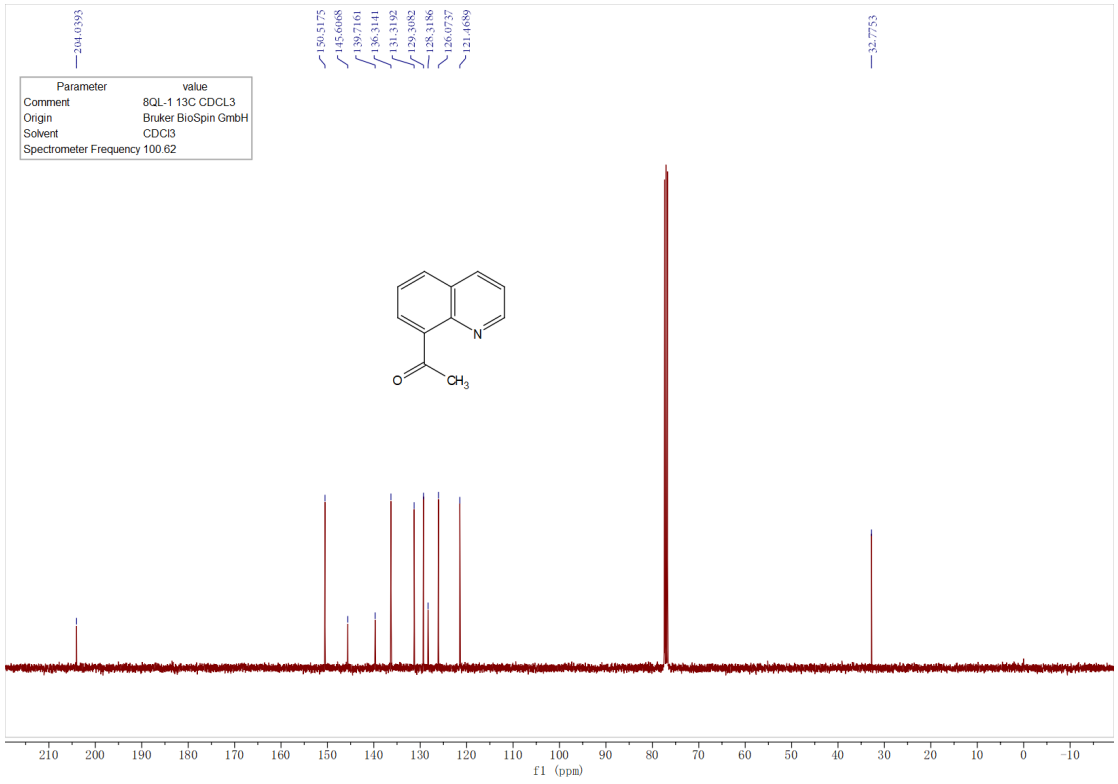

9.0186
9.0142
9.0079
9.0036
8.6934
8.6908
8.6883
8.2223
8.2202
8.2181
8.2159
8.2015
8.1996
8.1974
8.1951
8.1453
8.1409
8.1239
8.1195
7.9027
7.8813
7.5331
7.5225
7.5123
7.5018
7.2846 CDCl3
2.7701
2.0453 H2O
0.0806
-0.0012
Parameter value
Comment 7QL 1H CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 400.13
H3C
O
N
1.00
1.01
1.02
1.01
1.03
1.02
3.04
f1 (ppm)

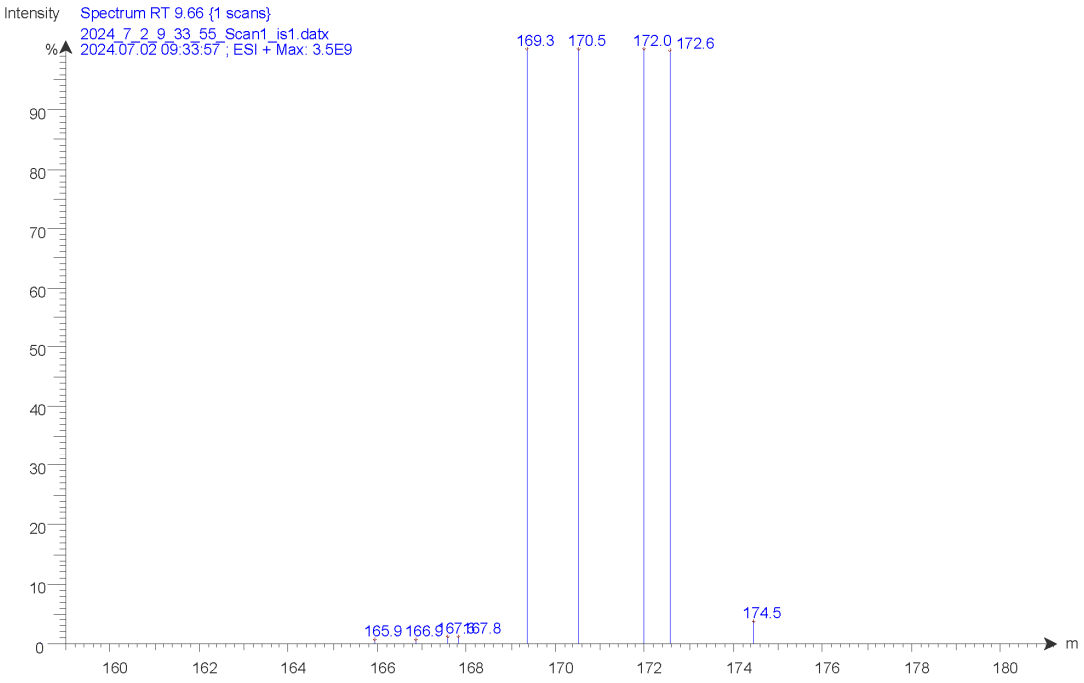

198.0026
151.4616
147.7898
137.5176
135.8830
131.5503
130.8431
128.3589
124.4030
123.0264
26.7855
Parameter value
Comment 7QL 13C CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 100.62
H3C
O
N
f1 (ppm)


Intensity
Spectrum RT 8.49 {1 scans}
2024_7_2_9_33_55_Scan1_is1.datx
2024.07.02 09:33:57 ; ESI + Max: 3.5E9
%
171.1
172.1
173.6
179.2
m
0
10
20
30
40
50
60
70
80
90
160
162
164
166
168
170
172
174
176
178
180

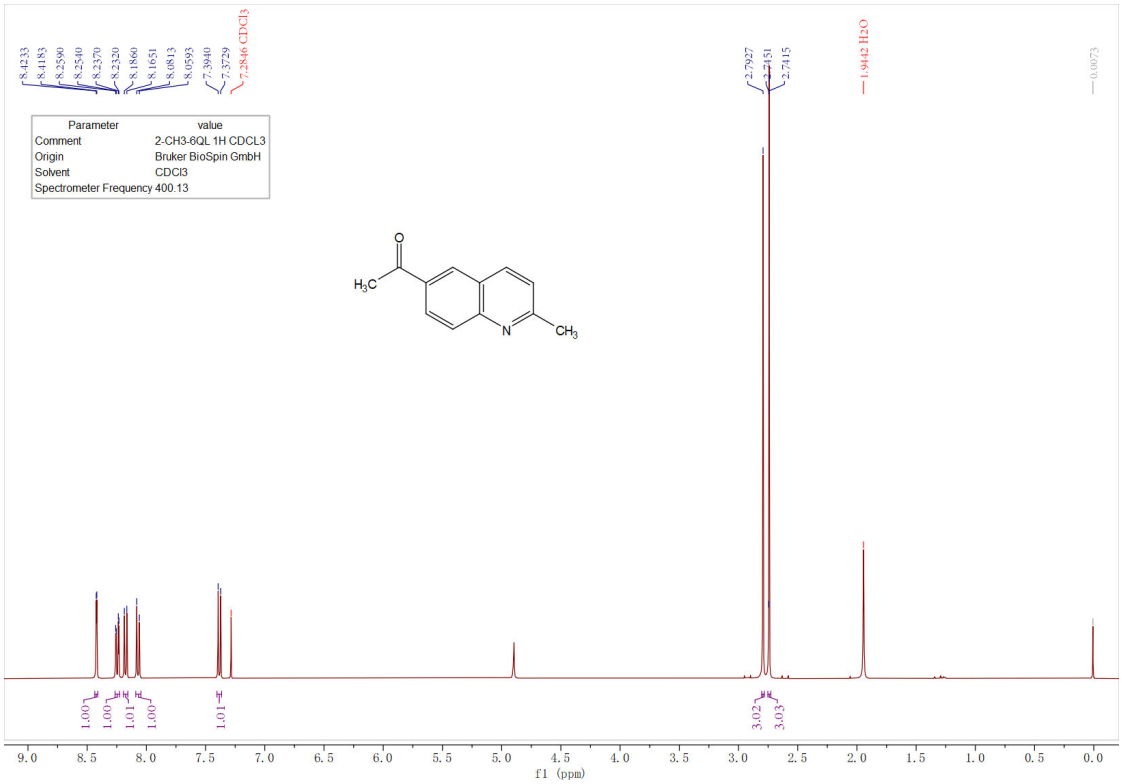
2g

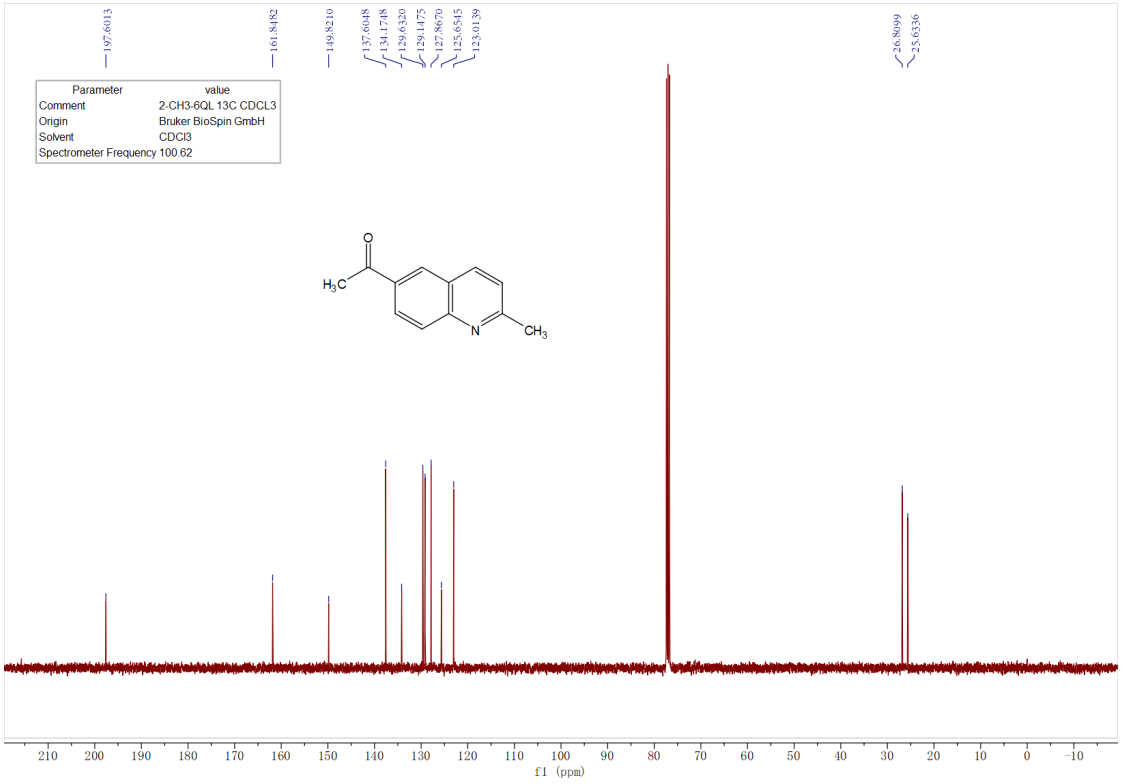

9.0073
9.0027
8.9969
8.9923
8.2353
8.2307
8.2145
8.2099
7.9697
7.9507
7.6252
7.6060
7.5869
7.4954
7.4850
7.4745
7.4642
7.2849 CDCl3
2.9706
1.7186 H2O
0.0166
Parameter value
Comment 8QL-1 1H CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 400.13
N
O
CH3
1.00
1.02
2.03
1.02
1.01
3.05
f1 (ppm)

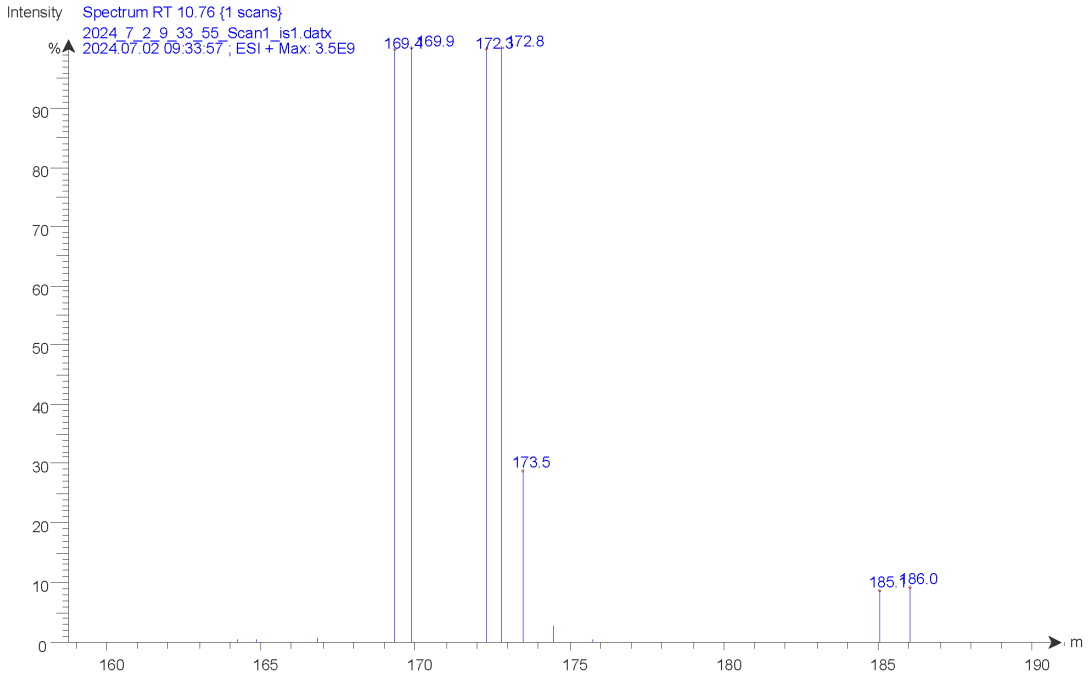

204.0393
150.5175
145.6068
139.7161
136.3141
131.3192
129.3082
128.3186
126.0737
121.4689
32.7753
Parameter value
Comment 8QL-1 13C CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 100.62
N
O
CH3
f1 (ppm)


Intensity
Spectrum RT 9.66 {1 scans}
2024_7_2_9_33_55_Scan1_is1.datx
2024.07.02 09:33:57 ; ESI + Max: 3.5E9
%
169.3
170.5
172.0
172.6
174.5
165.9
166.9
167.8
m
160
162
164
166
168
170
172
174
176
178
180
0
10
20
30
40
50
60
70
80
90

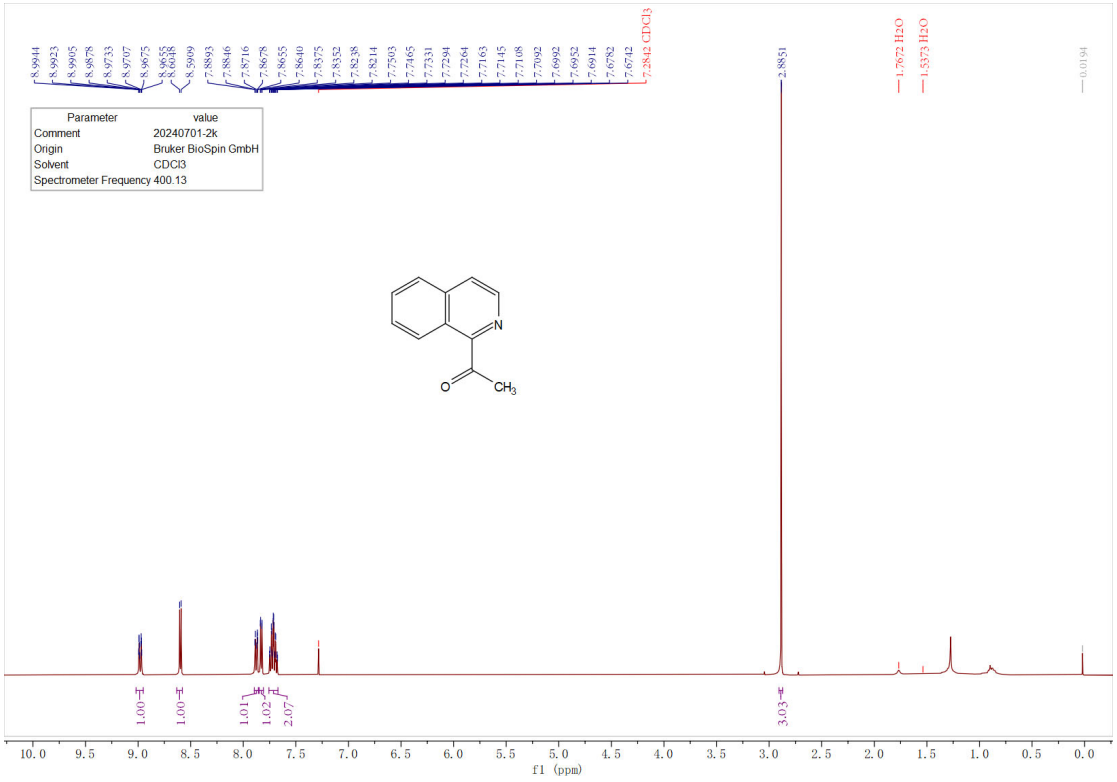
2h

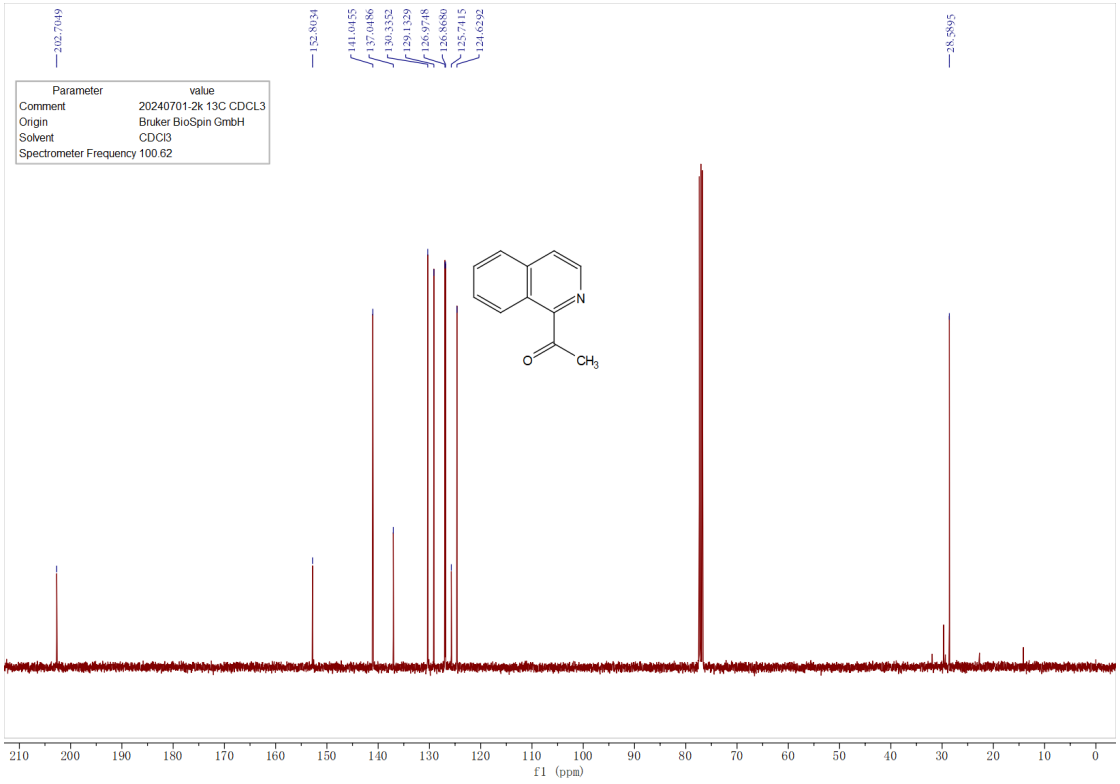

8.4233
8.4183
8.2590
8.2540
8.2370
8.2320
8.1860
8.1651
8.0813
8.0593
7.3940
7.3729
7.2846 CDCl3
2.7927
2.7151
2.7415
1.9442 H2O
0.0073
Parameter value
Comment 2-CH3-6QL 1H CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 400.13
O
H3C
N
CH3
1.00
1.00
1.01
1.00
1.01
3.02
3.03
f1 (ppm)

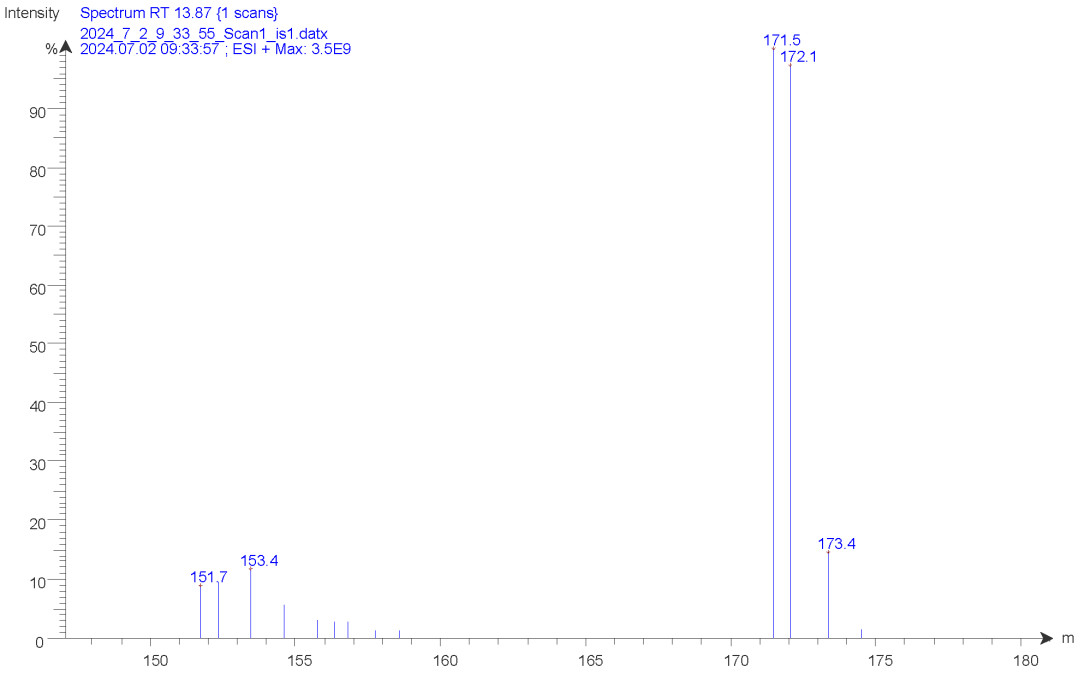

197.6013
161.8482
149.8210
137.6448
134.1748
129.6320
129.1475
127.8670
125.6545
123.0139
28.8099
25.6336
Parameter value
Comment 2-CH3-6QL 13C CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 100.62
O
H3C
N
CH3
f1 (ppm)


Intensity
Spectrum RT 10.76 {1 scans}
2024_7_2_9_33_55_Scan1_is1.datx
2024.07.02 09:33:57 ; ESI + Max: 3.5E9
%
169.4
169.9
172.1
172.8
173.5
185.1
186.0
m
0
10
20
30
40
50
60
70
80
90
160
165
170
175
180
185
190

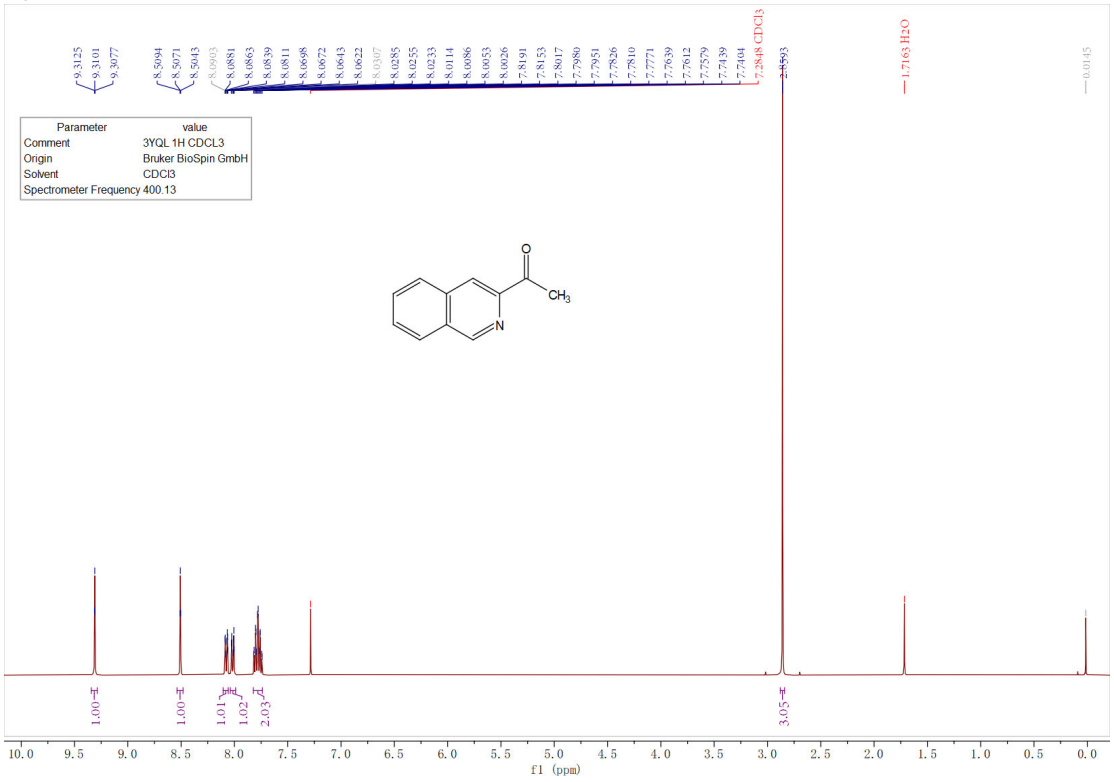
2i

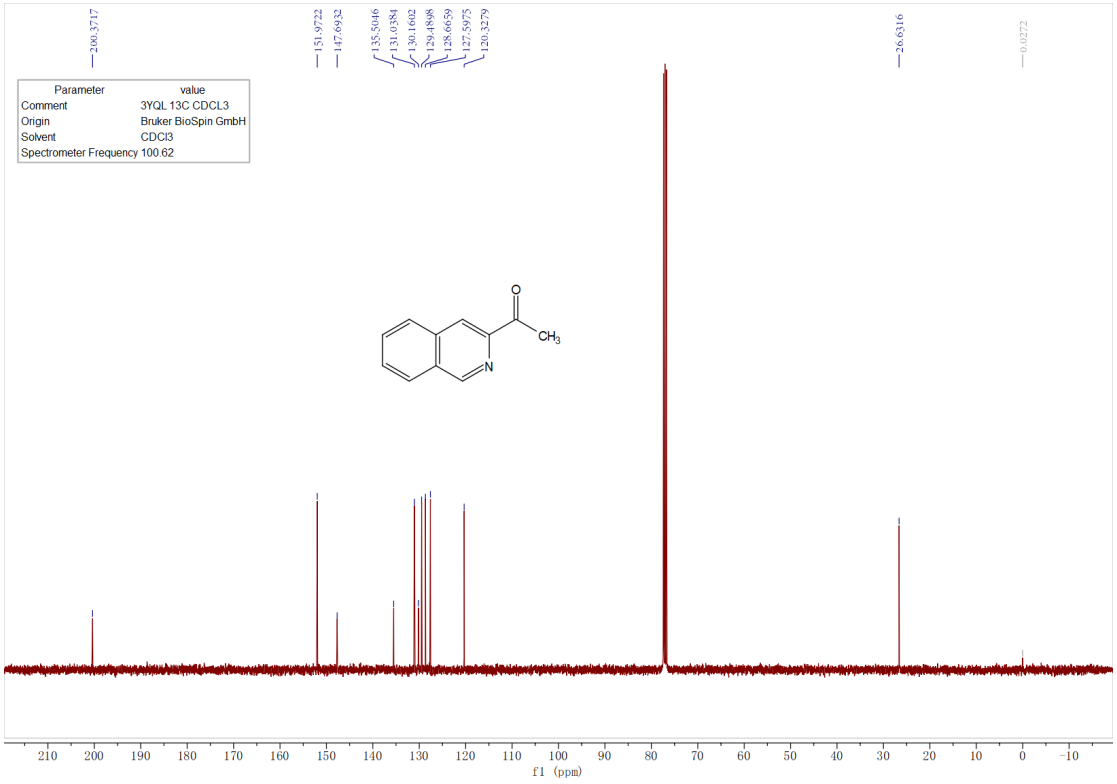

Parameter value
Comment 20240701-2k
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 400.13
8.9944
8.9923
8.9905
8.9878
8.9733
8.9707
8.9675
8.9655
8.6048
8.5969
7.8893
7.8846
7.8716
7.8678
7.8655
7.8640
7.8375
7.8352
7.8238
7.8214
7.7503
7.7465
7.7331
7.7294
7.7264
7.7163
7.7145
7.7108
7.7092
7.6992
7.6952
7.6914
7.6782
7.6742
7.2842 CDCl3
2.8851
1.7672 H2O
1.5373 H2O
0.0194
1.00
1.00
1.01
1.02
2.07
3.03
f1 (ppm)

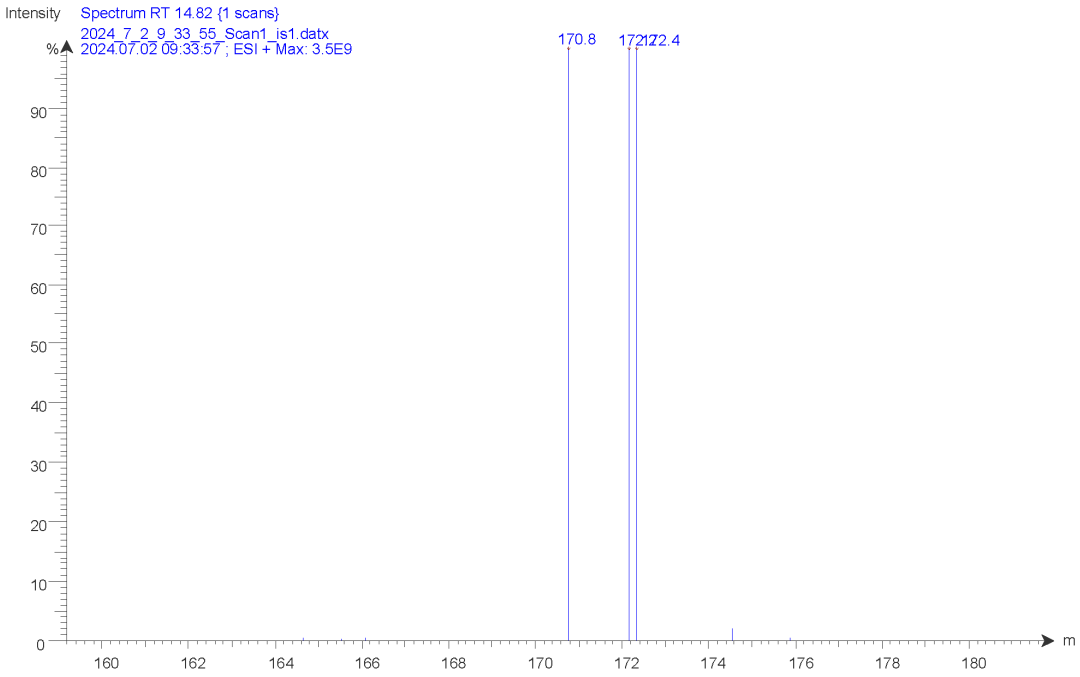

Parameter value
Comment 20240701-2k 13C CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 100.62
202.7049
152.8034
141.0455
137.0486
130.3352
129.1329
126.9748
126.8680
125.7415
124.6292
28.5895
f1 (ppm)


Intensity
Spectrum RT 13.87 {1 scans}
2024_7_2_9_33_55_Scan1_is1.datx
2024.07.02 09:33:57 ; ESI + Max: 3.5E9
%
90
80
70
60
50
40
30
20
10
0
150
155
160
165
170
175
180
m
171.5
172.1
173.4
153.4
151.7

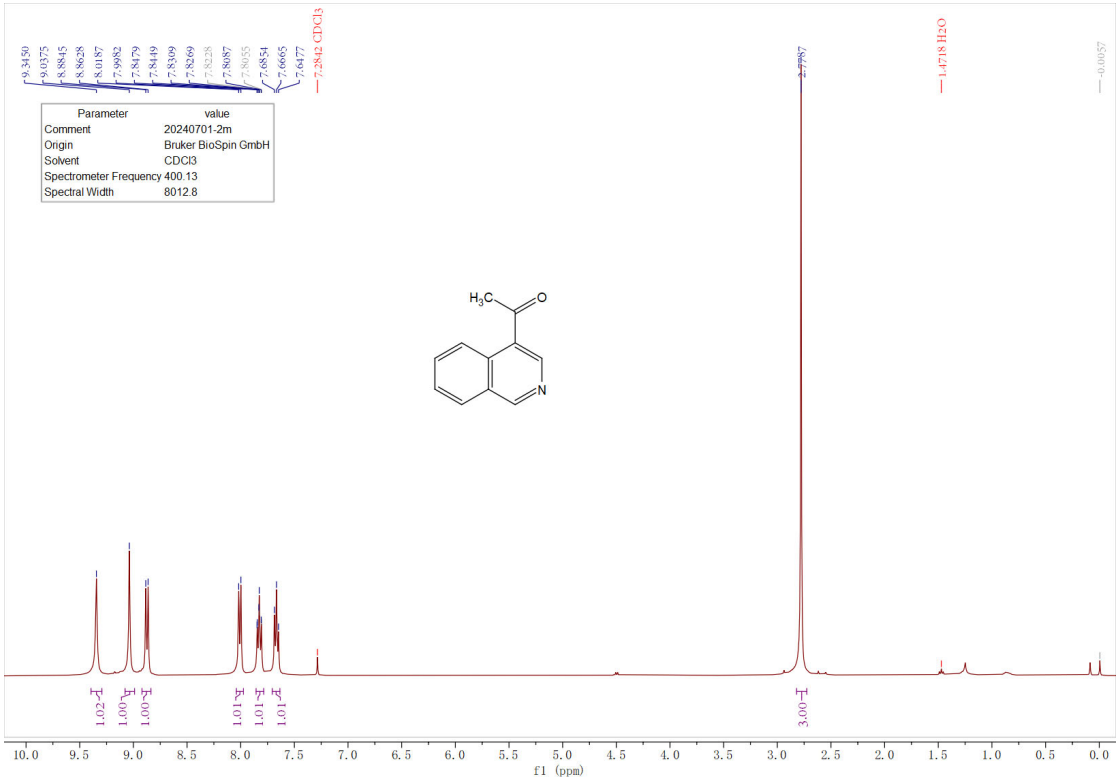
2j

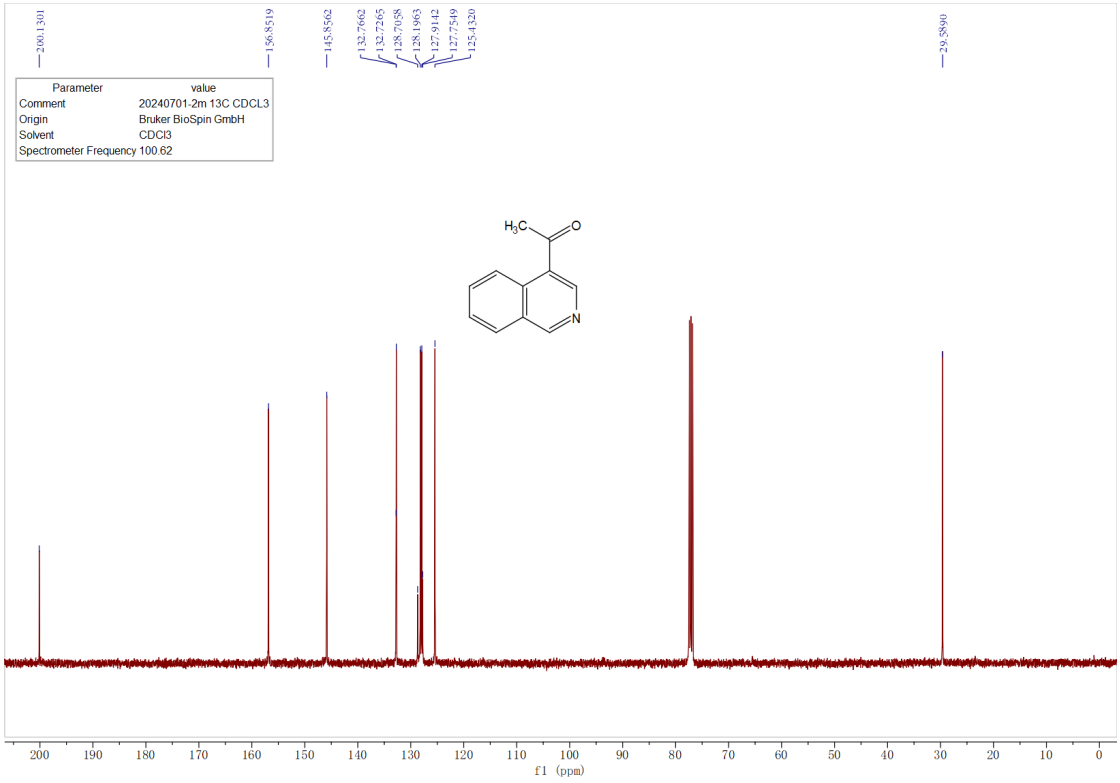

Parameter value
Comment 3YQL 1H CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 400.13
9.3125
9.3101
9.3077
8.5094
8.5071
8.5043
8.0903
8.0881
8.0863
8.0839
8.0811
8.0698
8.0672
8.0643
8.0622
8.0307
8.0285
8.0255
8.0233
8.0114
8.0086
8.0053
8.0026
7.8191
7.8153
7.8017
7.7980
7.7951
7.7826
7.7810
7.7771
7.7639
7.7612
7.7579
7.7439
7.7404
7.2848 CDCl3
2.0593
1.7163 H2O
0.0145
1.00
1.00
1.01
1.02
2.03
3.05
f1 (ppm)

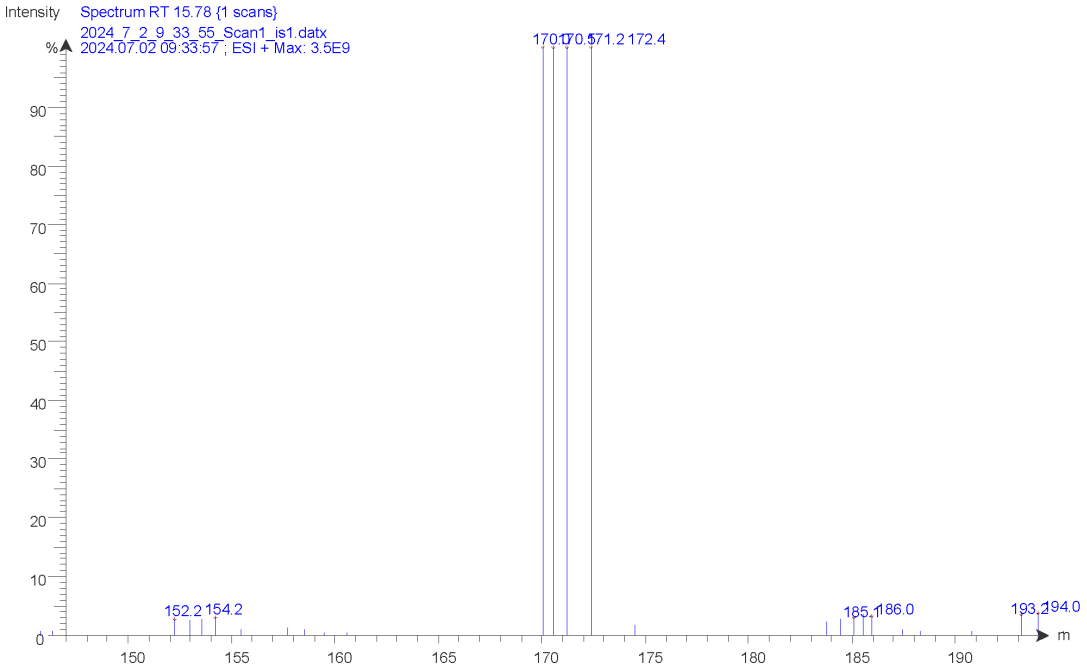

Parameter value
Comment 3YQL 13C CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 100.62
200.3717
151.9722
147.6932
135.5046
131.0384
130.1602
129.4898
128.6659
127.5975
120.3279
26.6316
0.0272
f1 (ppm)


Intensity
Spectrum RT 14.82 {1 scans}
2024_7_2_9_33_55_Scan1_is1.datx
2024.07.02 09:33:57 ; ESI + Max: 3.5E9
%
170.8
172.4
m
160
162
164
166
168
170
172
174
176
178
180
0
10
20
30
40
50
60
70
80
90

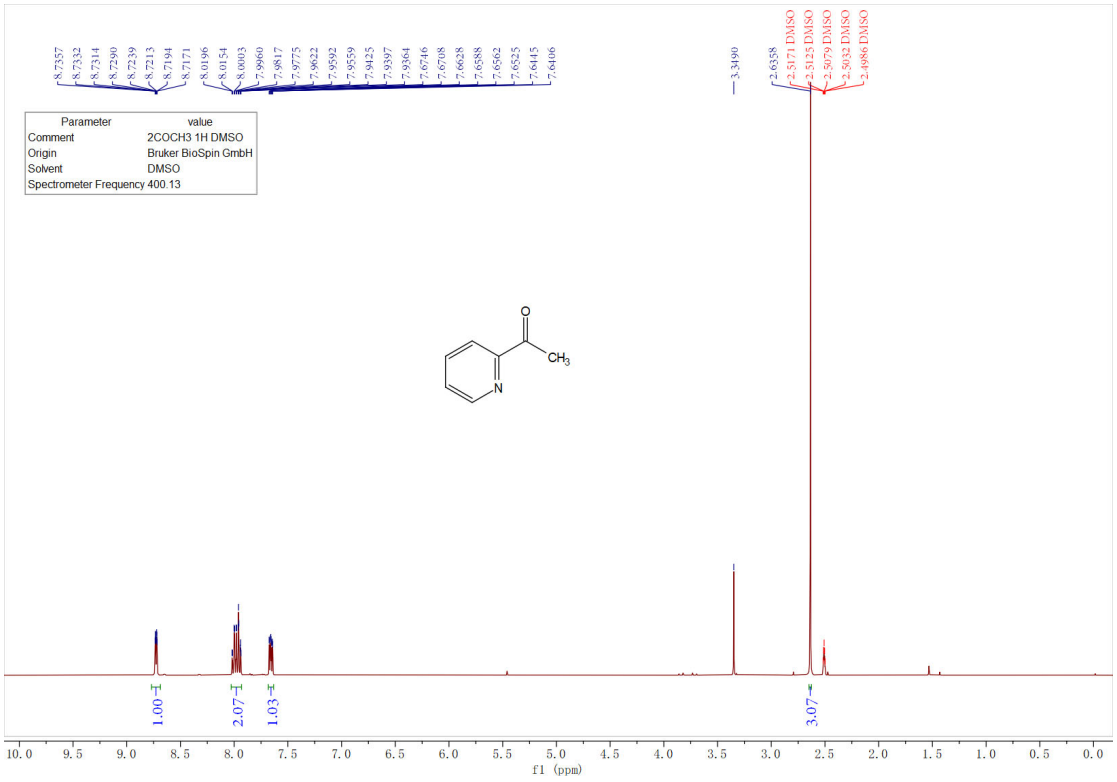
2k

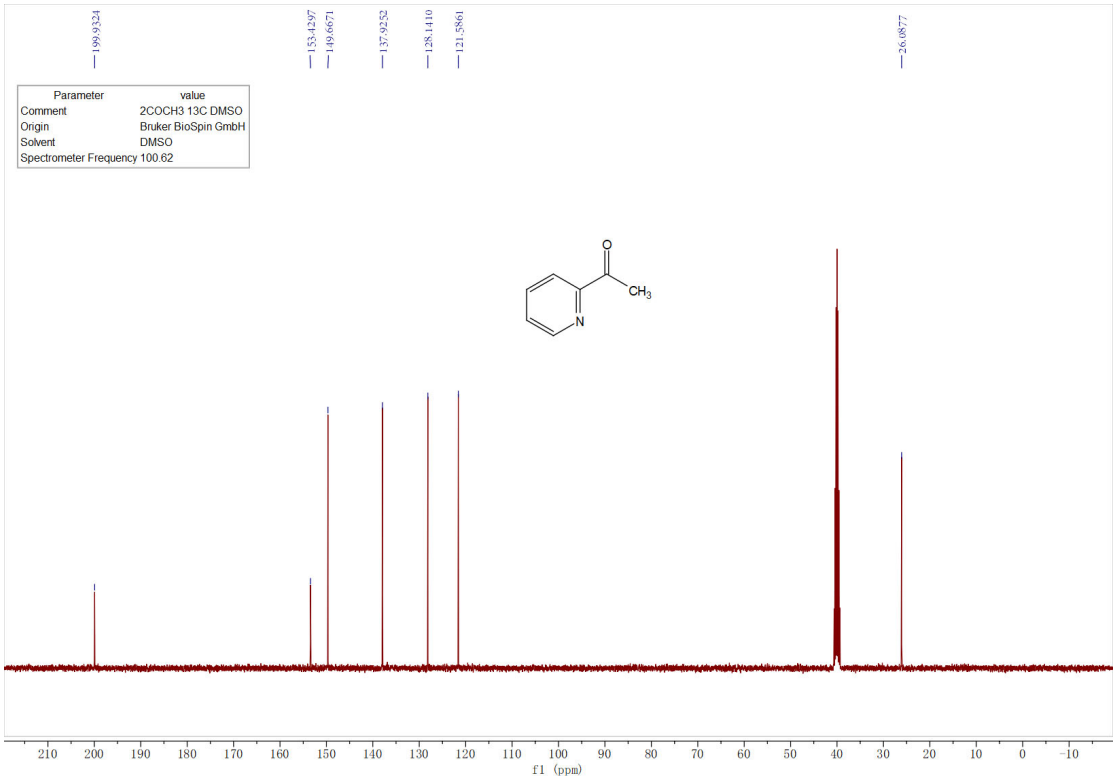

9.3450
9.0375
8.8845
8.8628
8.0187
7.9982
7.8479
7.8449
7.8309
7.8269
7.8087
7.6854
7.6665
7.6477
7.2842 CDCl3
2.7787
1.4718 H2O
0.0057
Parameter value
Comment 20240701-2m
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 400.13
Spectral Width 8012.8
H3C
O
N
1.02
1.00
1.00
1.01
1.01
1.01
3.00
f1 (ppm)

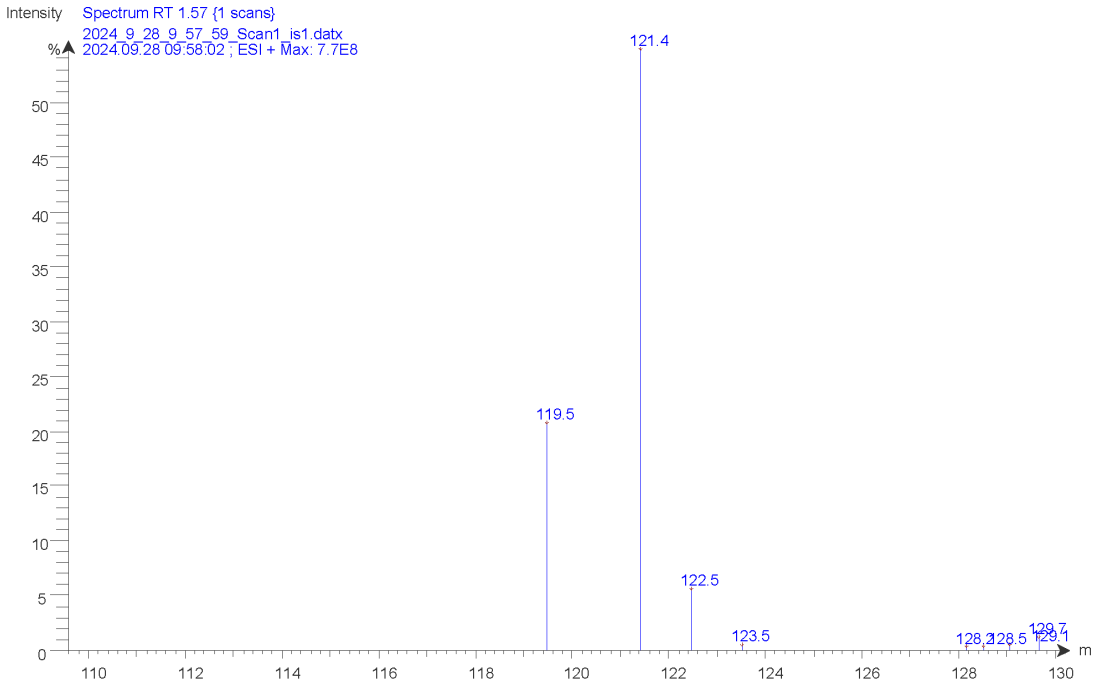

200.1301
156.8519
145.8562
132.7662
132.7265
128.7058
128.1963
127.9142
127.7549
125.4320
29.5890
Parameter value
Comment 20240701-2m 13C CDCL3
Origin Bruker BioSpin GmbH
Solvent CDCl3
Spectrometer Frequency 100.62
H3C
O
N
f1 (ppm)


Intensity
Spectrum RT 15.78 {1 scans}
2024_7_2_9_33_55_Scan1_is1.datx
2024.07.02 09:33:57 ; ESI + Max: 3.5E9
%
170.0
170.5
171.2
172.4
152.2
154.2
185.1
186.0
193.2
194.0
m
0
10
20
30
40
50
60
70
80
90
150
155
160
165
170
175
180
185
190

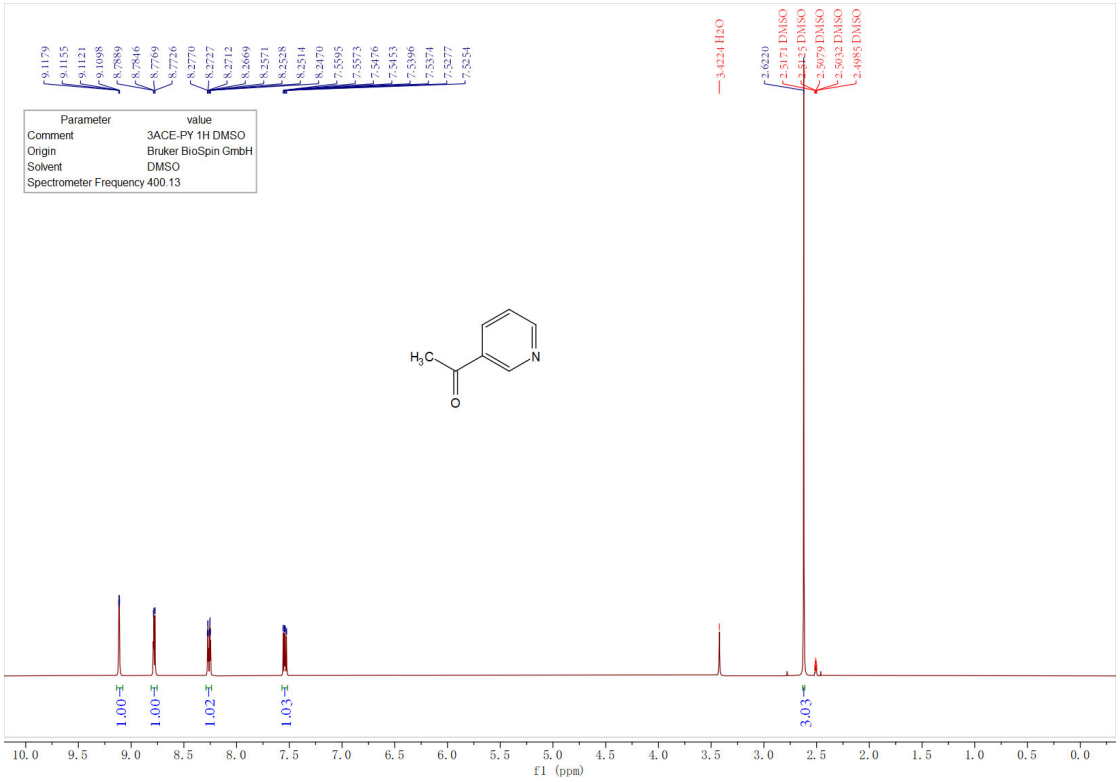
21

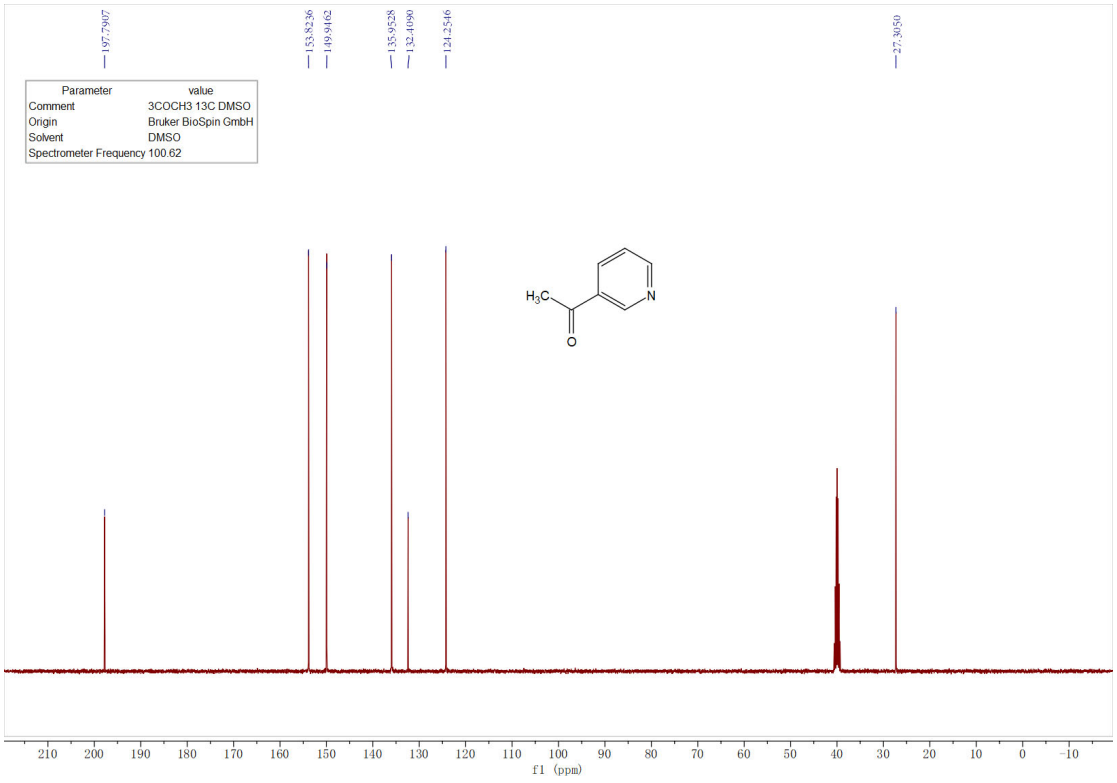

8.7357
8.7332
8.7314
8.7290
8.7239
8.7213
8.7194
8.7171
8.0196
8.0154
8.0003
7.9960
7.9817
7.9775
7.9622
7.9592
7.9559
7.9425
7.9397
7.9364
7.6746
7.6708
7.6628
7.6588
7.6562
7.6525
7.6445
7.6406
3.3490
2.6358
2.5171 DMSO
2.5125 DMSO
2.5079 DMSO
2.5032 DMSO
2.4986 DMSO
Parameter value
Comment 2COCH3 1H DMSO
Origin Bruker BioSpin GmbH
Solvent DMSO
Spectrometer Frequency 400.13
O
CH3
N
1.00
2.07
1.03
3.07
f1 (ppm)

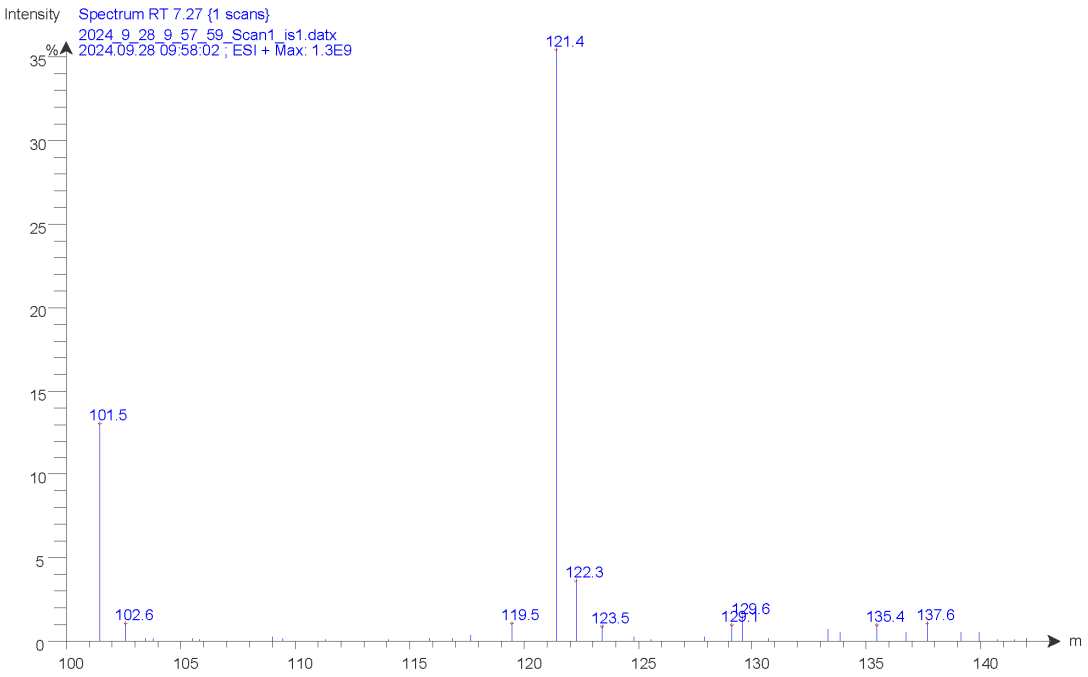

199.9324
153.4297
149.6671
137.9252
128.1410
121.5861
26.0877
Parameter value
Comment 2COCH3 13C DMSO
Origin Bruker BioSpin GmbH
Solvent DMSO
Spectrometer Frequency 100.62
O
CH3
N
f1 (ppm)


Intensity
Spectrum RT 1.57 {1 scans}
2024_9_28_9_57_59_Scan1_is1.datx
2024.09.28 09:58:02 ; ESI + Max: 7.7E8
%
121.4
119.5
122.5
123.5
128.1
128.5
129.1
129.7
m
0
5
10
15
20
25
30
35
40
45
50
110
112
114
116
118
120
122
124
126
128
130

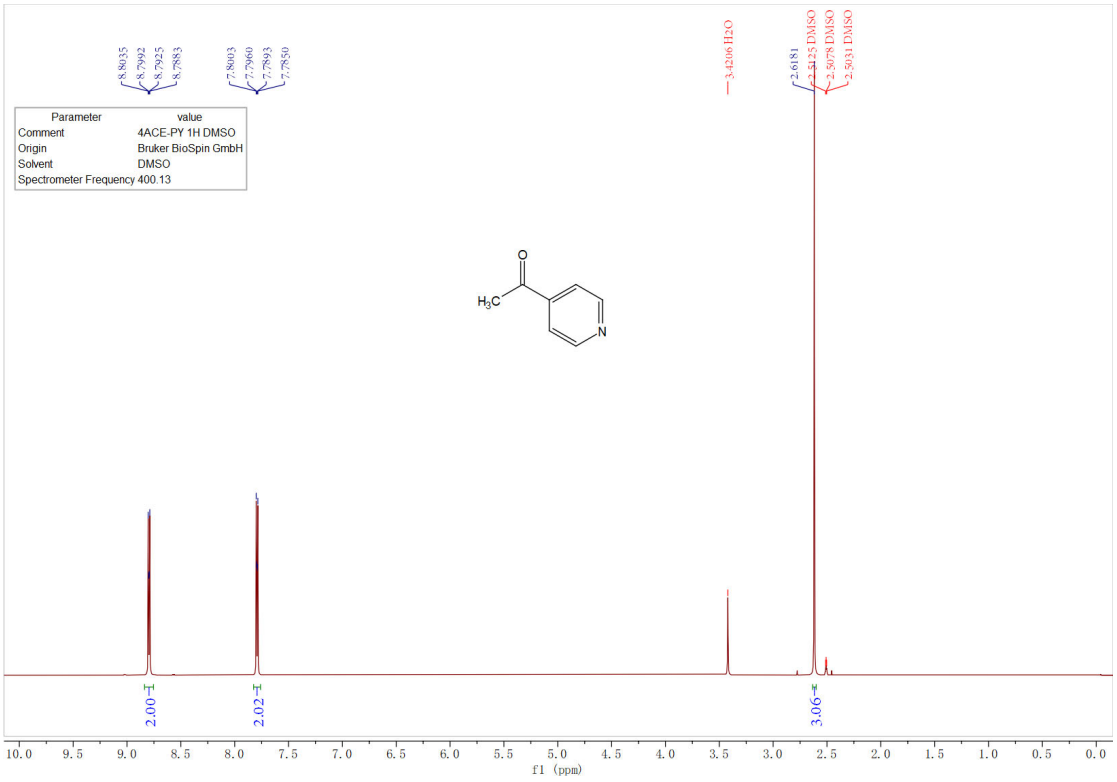
2m

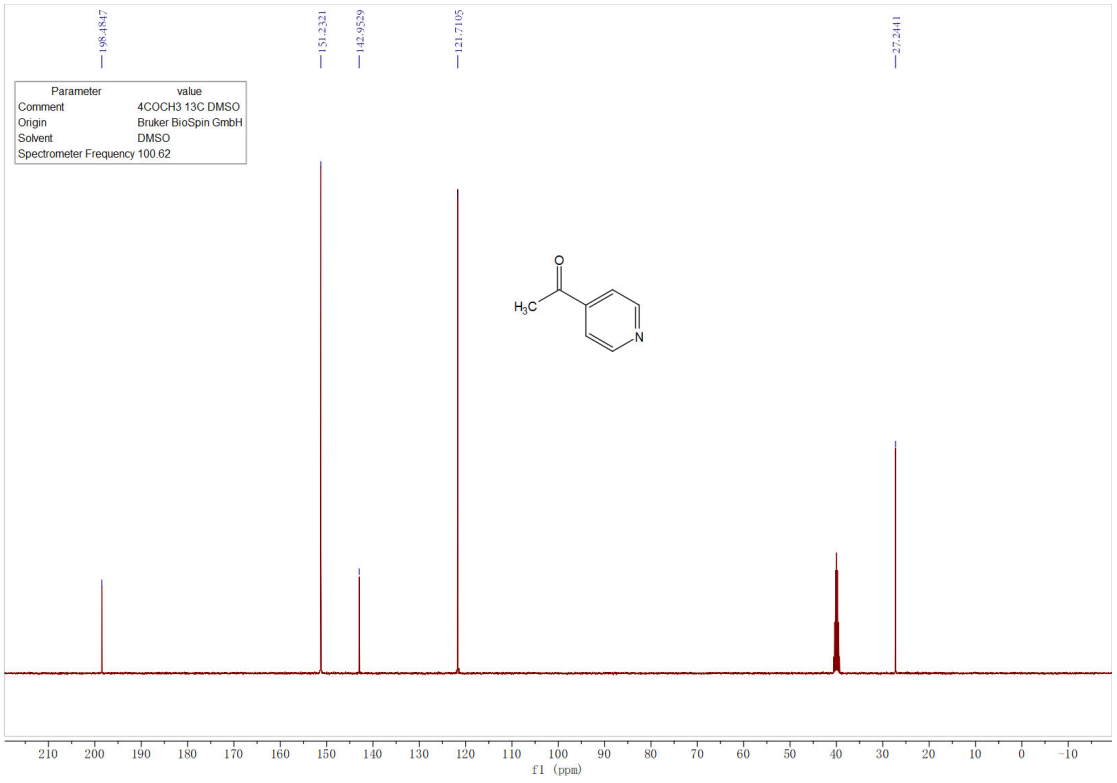

9.1179
9.1155
9.1121
9.1098
8.7889
8.7846
8.7769
8.7726
8.2770
8.2727
8.2712
8.2669
8.2571
8.2528
8.2514
8.2470
7.5595
7.5573
7.5476
7.5453
7.5396
7.5374
7.5277
7.5254
3.4224 H2O
2.6220
2.5171 DMSO
2.5125 DMSO
2.5079 DMSO
2.5032 DMSO
2.4985 DMSO
Parameter value
Comment 3ACE-PY 1H DMSO
Origin Bruker BioSpin GmbH
Solvent DMSO
Spectrometer Frequency 400.13
1.00
1.00
1.02
1.03
3.03
f1 (ppm)

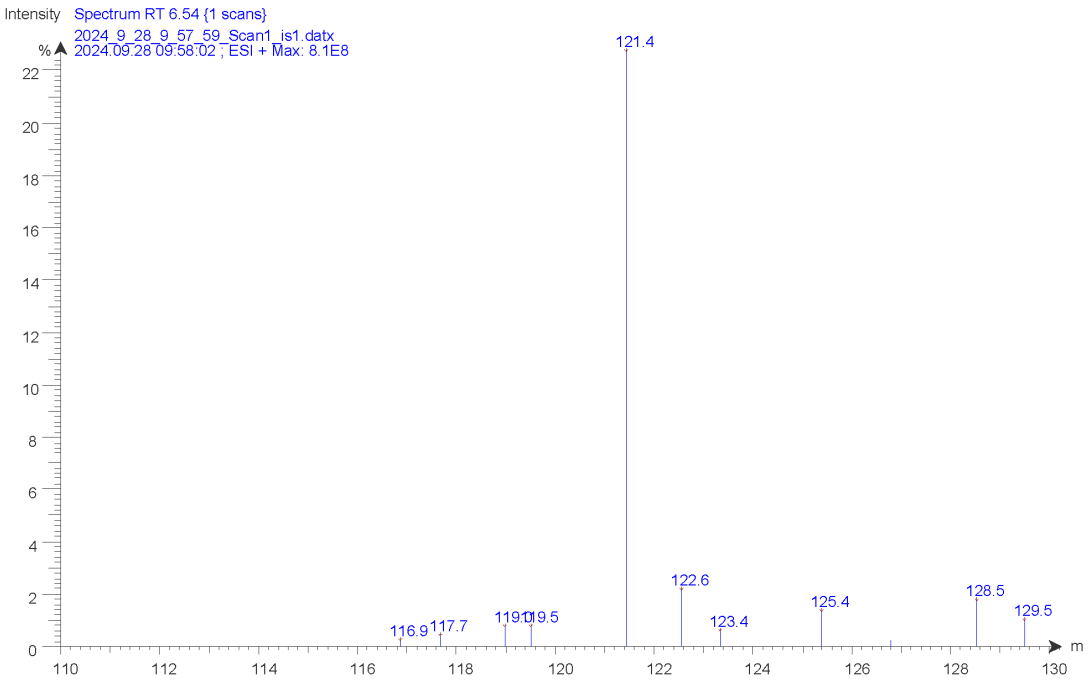

197.7907
153.8236
149.9462
135.9528
132.4090
124.2546
27.3050
Parameter value
Comment 3COCH3 13C DMSO
Origin Bruker BioSpin GmbH
Solvent DMSO
Spectrometer Frequency 100.62
f1 (ppm)


Intensity
Spectrum RT 7.27 {1 scans}
2024_9_28_9_57_59_Scan1_is1.datx
2024.09.28 09:58:02 ; ESI + Max: 1.3E9
%
35
30
25
20
15
10
5
0
121.4
101.5
122.3
102.6
119.5
123.5
129.6
135.4
137.6
100
105
110
115
120
125
130
135
140
m

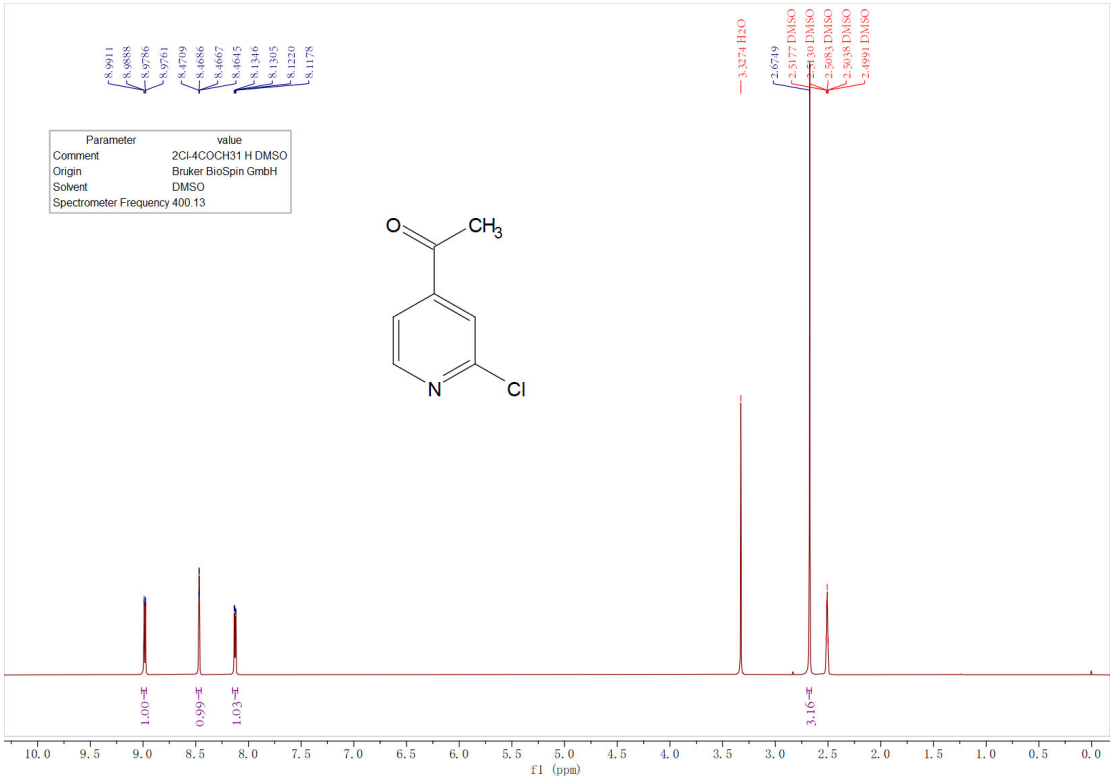
2n

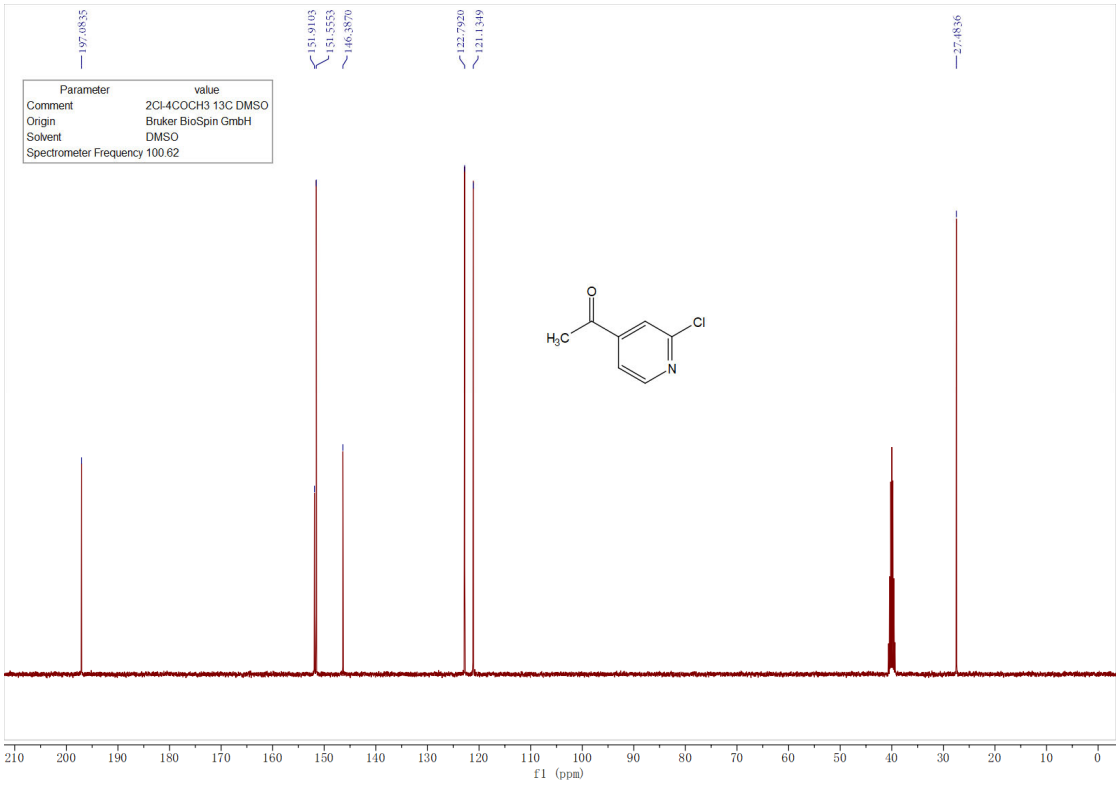

8.8035
8.7992
8.7925
8.7883
7.8803
7.7960
7.7893
7.7850
3.4206 H2O
2.6181
2.5125 DMSO
2.5078 DMSO
2.5031 DMSO
Parameter value
Comment 4ACE-PY 1H DMSO
Origin Bruker BioSpin GmbH
Solvent DMSO
Spectrometer Frequency 400.13
O
H3C
N
2.00
2.02
3.06
f1 (ppm)

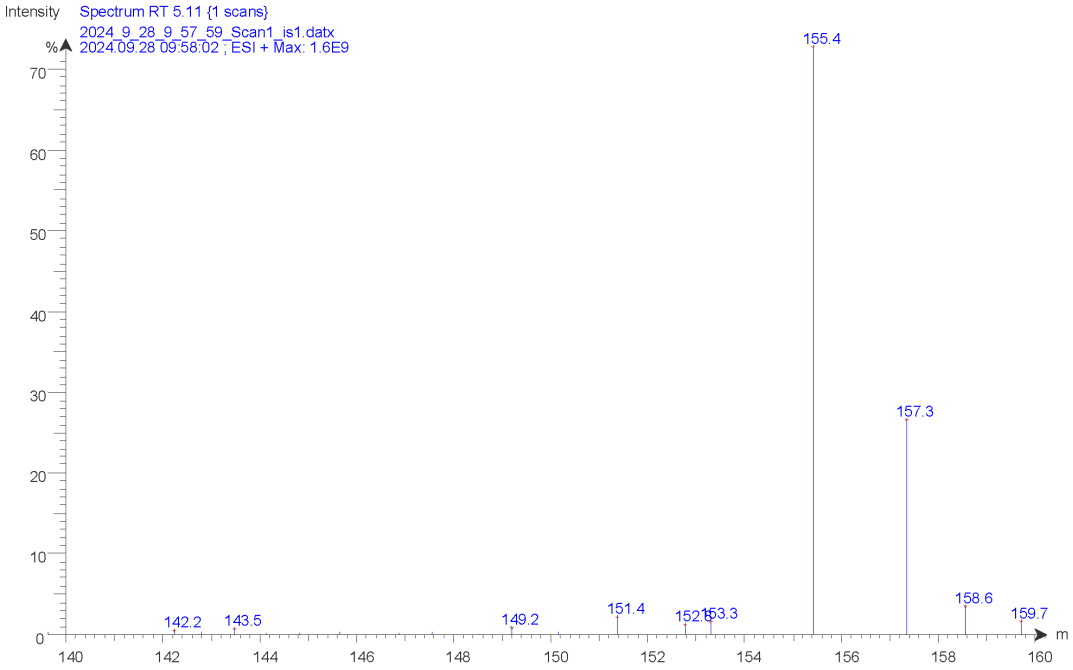

198.4847
151.2321
142.9529
121.7105
27.2441
Parameter value
Comment 4COCH3 13C DMSO
Origin Bruker BioSpin GmbH
Solvent DMSO
Spectrometer Frequency 100.62
O
H3C
N
f1 (ppm)


Intensity
Spectrum RT 6.54 {1 scans}
2024_9_28_9_57_59_Scan1_is1.datx
2024.09.28 09:58:02 ; ESI + Max: 8.1E8
%
22
20
18
16
14
12
10
8
6
4
2
0
110
112
114
116
118
120
122
124
126
128
130
m
121.4
122.6
123.4
125.4
128.5
129.5
116.9
117.7
119.0
119.5

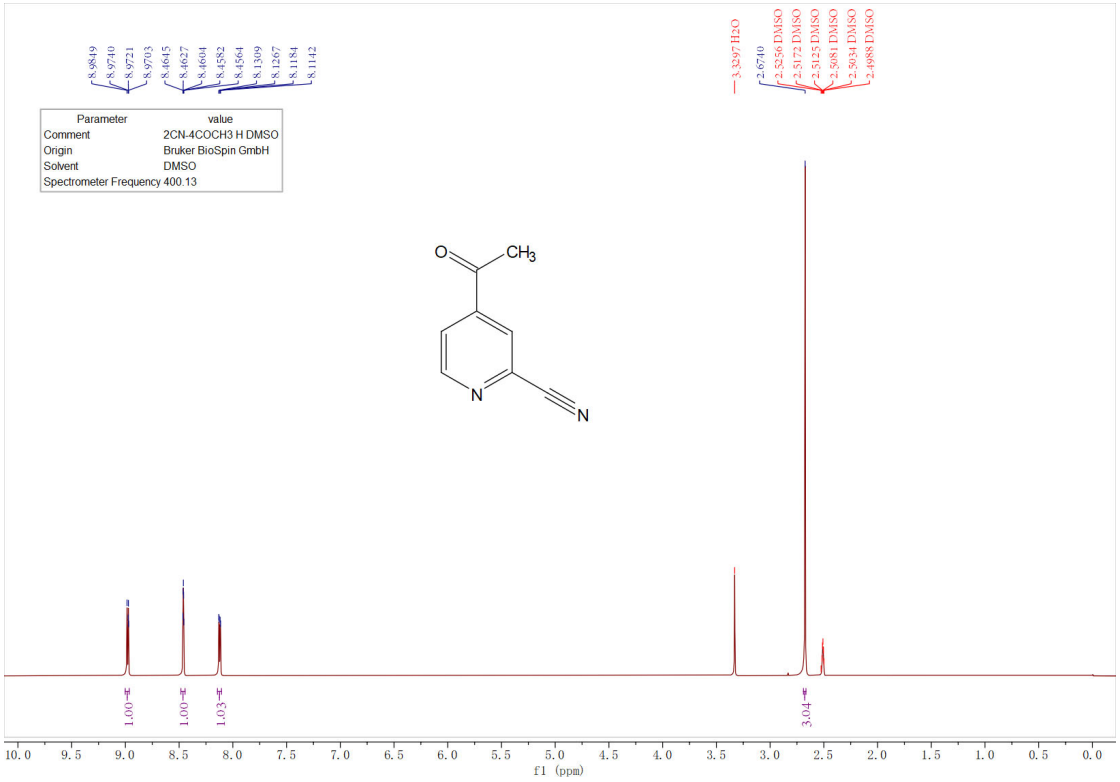
2o

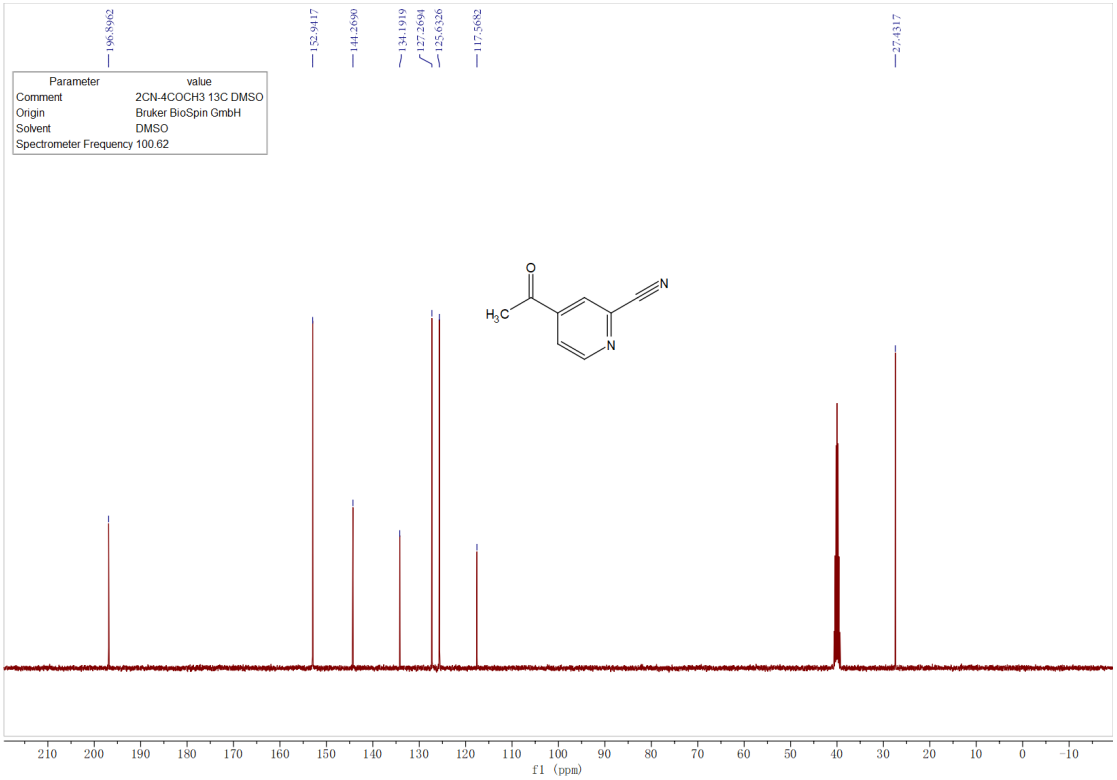

8.9911
8.9888
8.9786
8.9761
8.4709
8.4686
8.4667
8.4645
8.1346
8.1305
8.1220
8.1178
3.3274 H2O
2.6749
2.5177 DMSO
2.5130 DMSO
2.5083 DMSO
2.5038 DMSO
2.4991 DMSO
Parameter value
Comment 2Cl-4COCH31 H DMSO
Origin Bruker BioSpin GmbH
Solvent DMSO
Spectrometer Frequency 400.13
O
CH3
N
Cl
1.00
0.99
1.03
3.16
f1 (ppm)

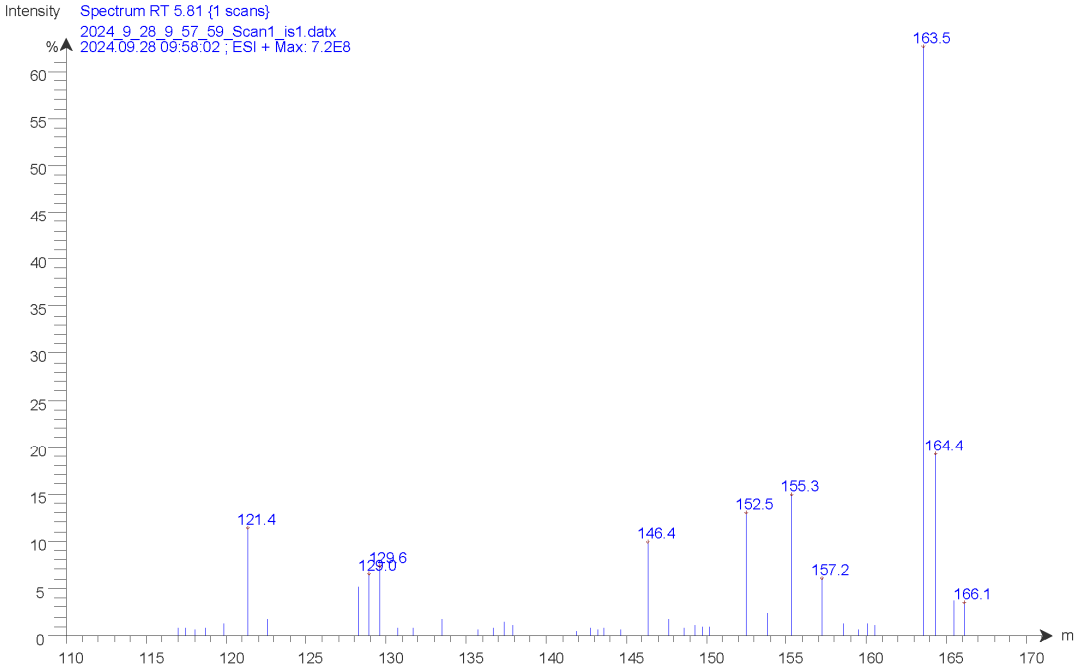

197.0835
151.9103
151.5553
146.3870
122.7920
121.1349
27.4836
Parameter value
Comment 2Cl-4COCH3 13C DMSO
Origin Bruker BioSpin GmbH
Solvent DMSO
Spectrometer Frequency 100.62
O
H3C
Cl
N
f1 (ppm)


Intensity
Spectrum RT 5.11 {1 scans}
2024_9_28_9_57_59_Scan1_is1.datx
2024.09.28 09:58:02 ; ESI + Max: 1.6E9
%
70
60
50
40
30
20
10
0
140
142
144
146
148
150
152
154
156
158
160
m
155.4
157.3
158.6
159.7
142.2
143.5
149.2
151.4
153.3

2p


8.9849
8.9740
8.9721
8.9703
8.4645
8.4627
8.4604
8.4582
8.4564
8.1309
8.1267
8.1184
8.1142
3.3297 H2O
2.6749
2.5256 DMSO
2.5172 DMSO
2.5125 DMSO
2.5081 DMSO
2.5034 DMSO
2.4988 DMSO
Parameter value
Comment 2CN-4COCH3 H DMSO
Origin Bruker BioSpin GmbH
Solvent DMSO
Spectrometer Frequency 400.13
O
CH3
N
N
1.00
1.00
1.03
3.04
10.0 9.5 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 0.0
f1 (ppm)


196.8962
152.9417
144.2690
134.1919
127.2694
125.6326
117.5682
27.4317
Parameter value
Comment 2CN-4COCH3 13C DMSO
Origin Bruker BioSpin GmbH
Solvent DMSO
Spectrometer Frequency 100.62
O
H3C
N
N
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)


Intensity
Spectrum RT 5.81 {1 scans}
2024_9_28_9_57_59_Scan1_is1.datx
2024.09.28 09:58:02 ; ESI + Max: 7.2E8
%
163.5
164.4
155.3
152.5
121.4
146.4
129.6
129.0
157.2
166.1
m