

Supporting Information

Fluorinated Alcohol Mediated *N,N'*-Dialkylation of Amino Acid Derivatives via Cascade [1,5]-Hydride Transfer/Cyclization for Concise Synthesis of Tetrahydroquinazoline

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Li^{* a, b}

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#700, Qingdao 266109, P. R. China

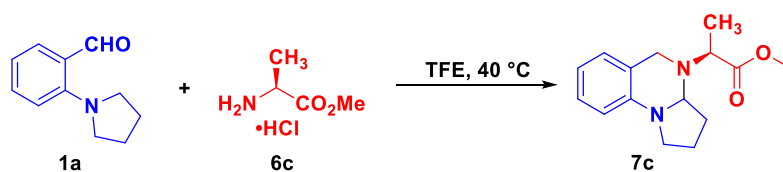
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Qingdao University of Science and Technology, Zhengzhou Rd. #53, Qingdao 266042, P. R. China

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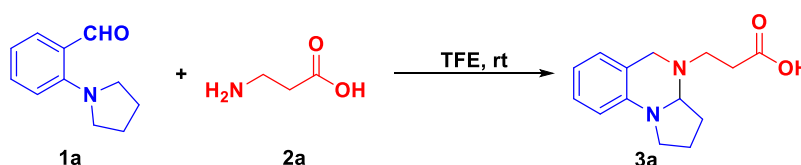
1. General Procedure

1.1 Chiral induction with chiral amino acids Experiments



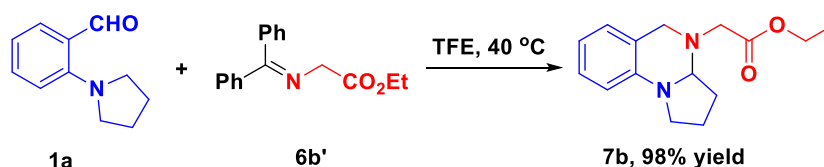
An oven-dried reaction tube was charged with 2-aminobenzaldehyde **1a** (1.0 equiv, 0.1 mmol), trifluoroethanol (TFE) (2 mL) and amino acid ester **6c** (1.5 equiv., 0.15 mmol). The reaction mixture was stirred vigorously at 40 °C and monitored by TLC. After consumption of **1a**, the reaction mixture was concentrated in vacuo and the residue was subjected to flash column chromatography for purification to afford product **7c** in 91% yield with dr = 5:1, and er = 74:26.

1.2 Gram-Scale Syntheses

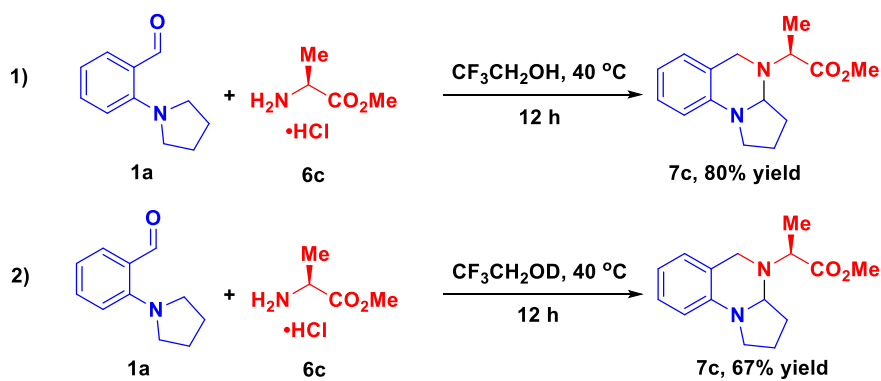


An oven-dried reaction tube was charged with 2-aminobenzaldehyde **1a** (1.0 equiv, 5 mmol), trifluoroethanol (TFE) (100 mL) and β-amino acids **2a** (1.5 equiv., 6.5 mmol). The reaction mixture was stirred vigorously at room temperature and monitored by TLC. After consumption of **1a**, the reaction mixture was concentrated in vacuo and the residue was subjected to flash column chromatography for purification to afford product **3a** in 76% yield (1.16 g).

1.3 Control experiments



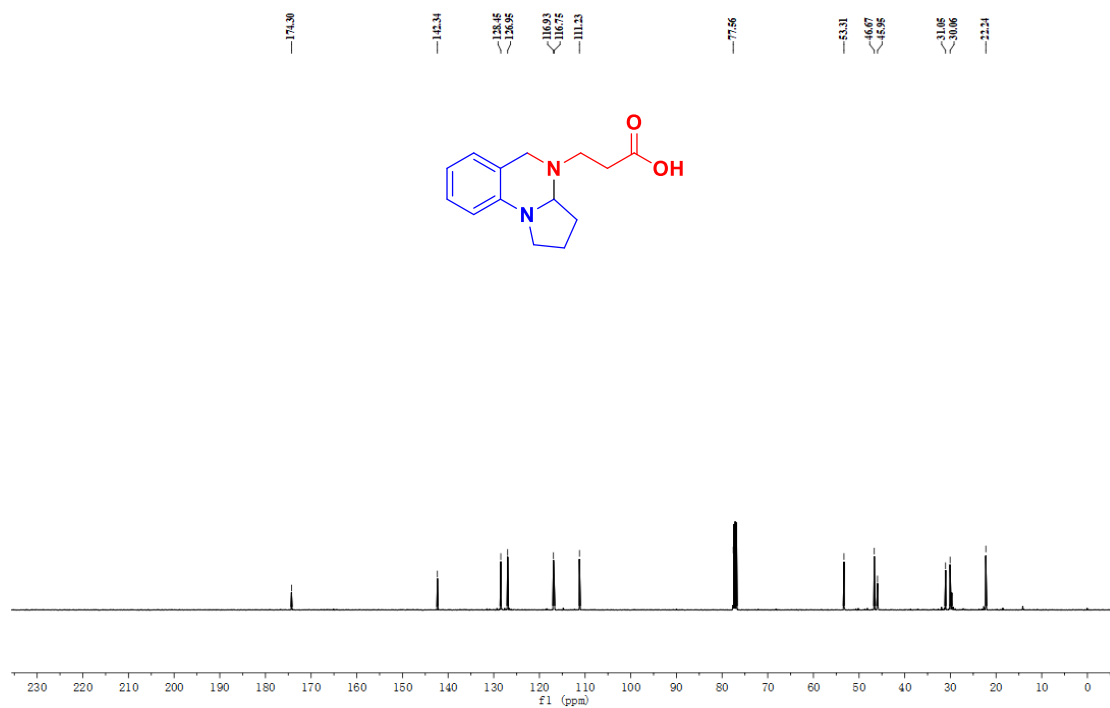
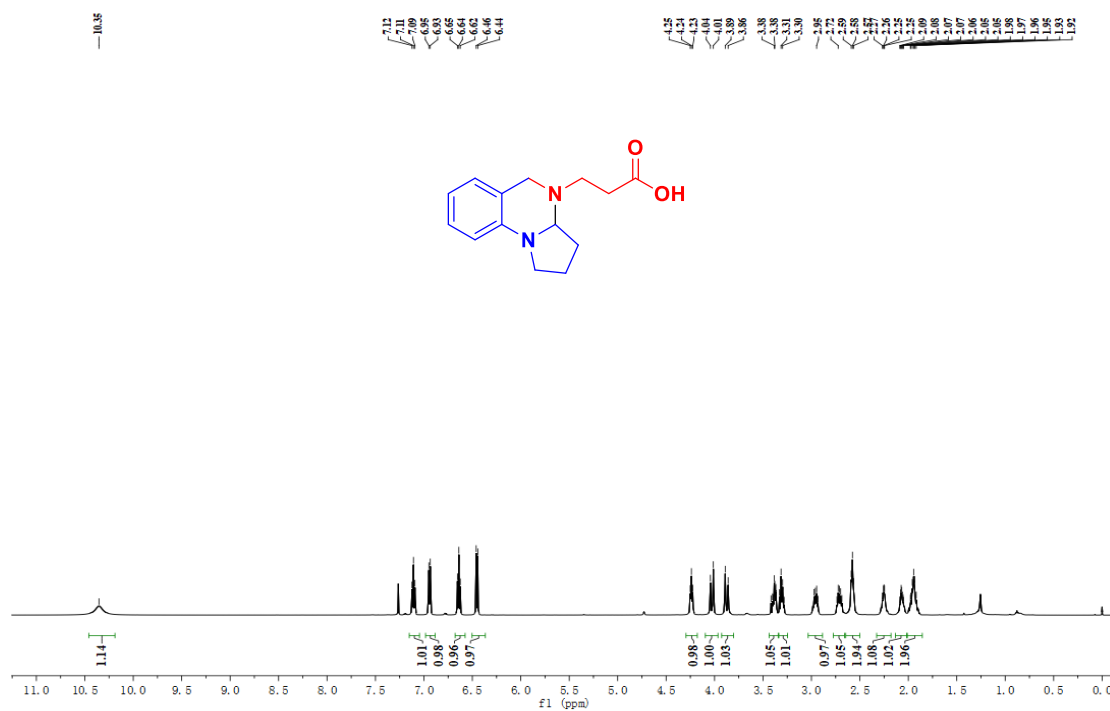
An oven-dried reaction tube was charged with 2-aminobenzaldehyde **1a** (1.0 equiv, 0.1 mmol), trifluoroethanol (TFE) (2 mL) and ethyl 2-((diphenylmethylene)amino)acetate **6b'** (1.5 equiv., 0.15 mmol). The reaction mixture was stirred vigorously at 40 °C and monitored by TLC. After consumption of **1a**, the reaction mixture was concentrated in vacuo and the residue was subjected to by flash column chromatography for purification to afford product **7b** in 98% yield.



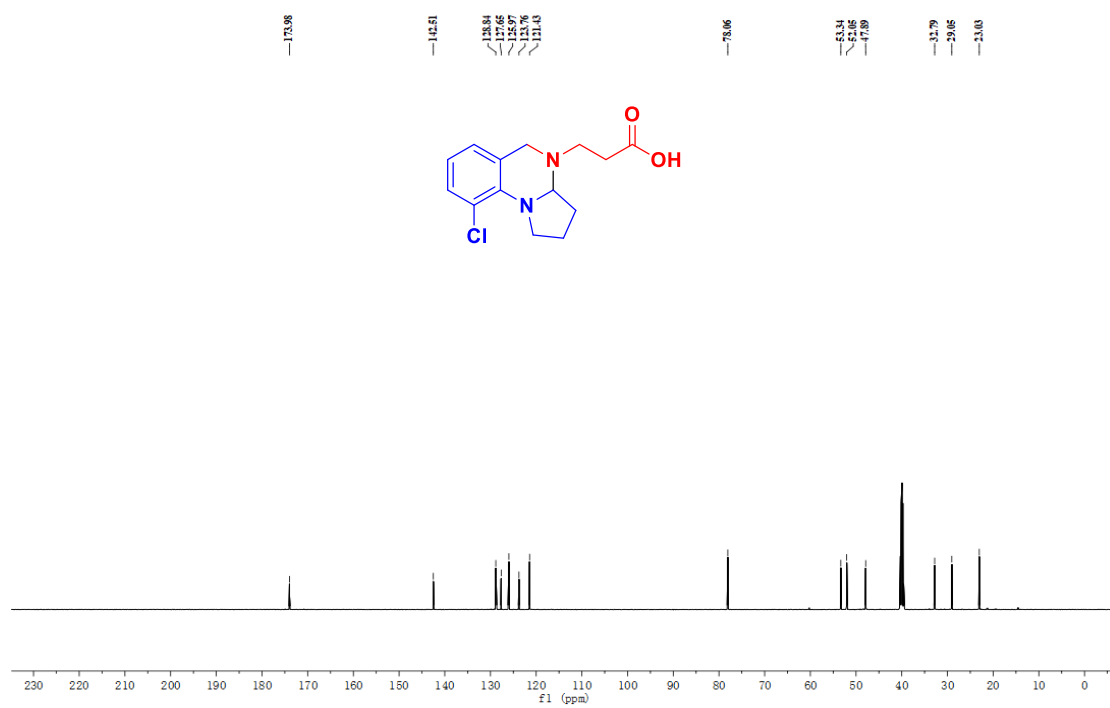
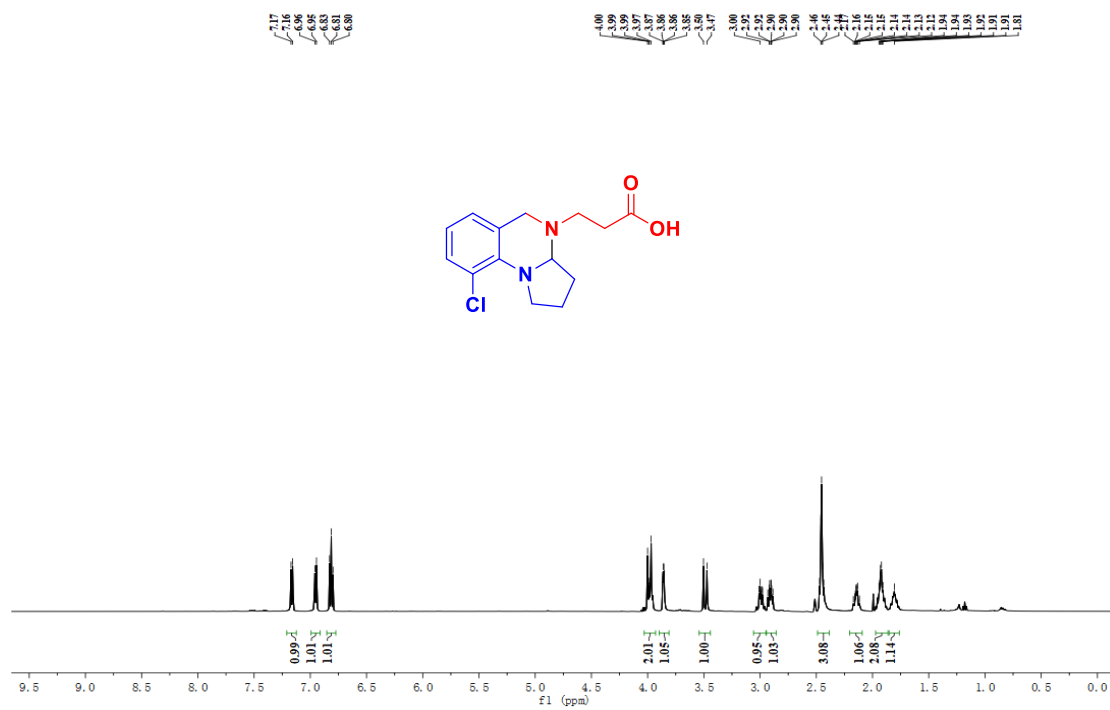
- 1) An oven-dried reaction tube was charged with 2-aminobenzaldehyde **1a** (1.0 equiv., 0.1 mmol), trifluoroethanol (TFE) (2 mL) and amino acid ester **6c** (1.5 equiv., 0.15 mmol). The reaction mixture was stirred vigorously at 40 °C with 12 h. The reaction mixture was concentrated in vacuo and the residue was subjected to by flash column chromatography for purification to afford product **7c** in 80% yield.
- 2) An oven-dried reaction tube was charged with 2-aminobenzaldehyde **1a** (1.0 equiv., 0.1 mmol), trifluoroethane (ol-D) (2 mL) and amino acid ester **6c** (1.5 equiv., 0.15 mmol). The reaction mixture was stirred vigorously at 40 °C with 12 h. The reaction mixture was concentrated in vacuo and the residue was subjected to by flash column chromatography for purification to afford product **7c** in 67% yield.

2. ¹H and ¹³C NMR Spectra

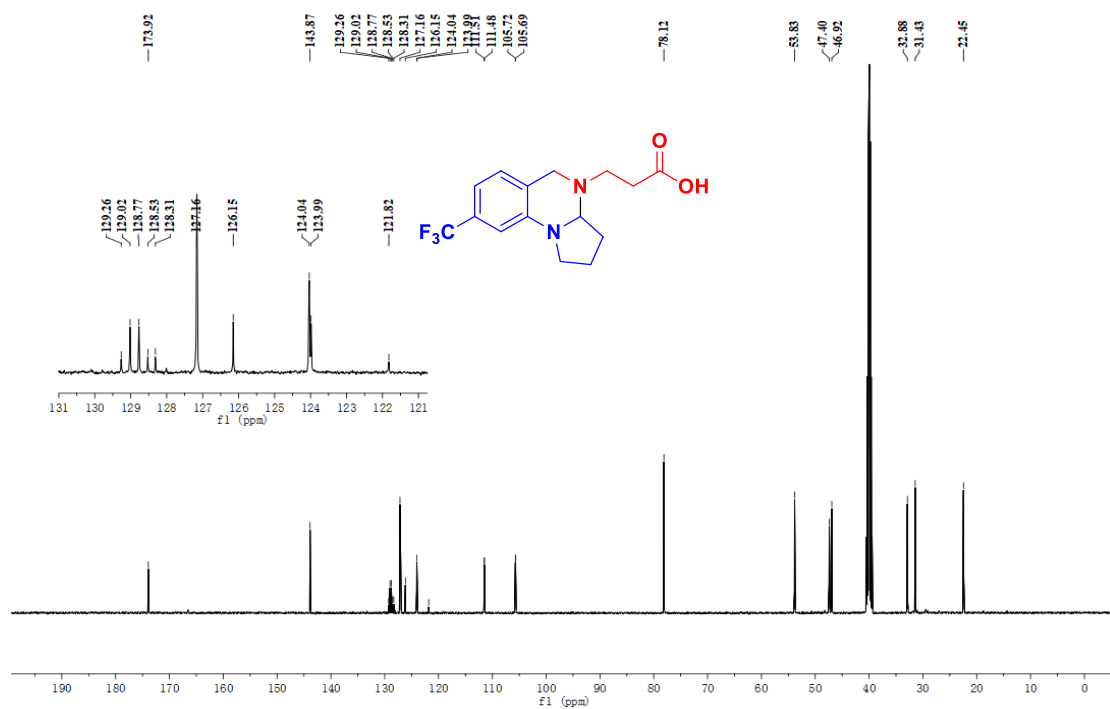
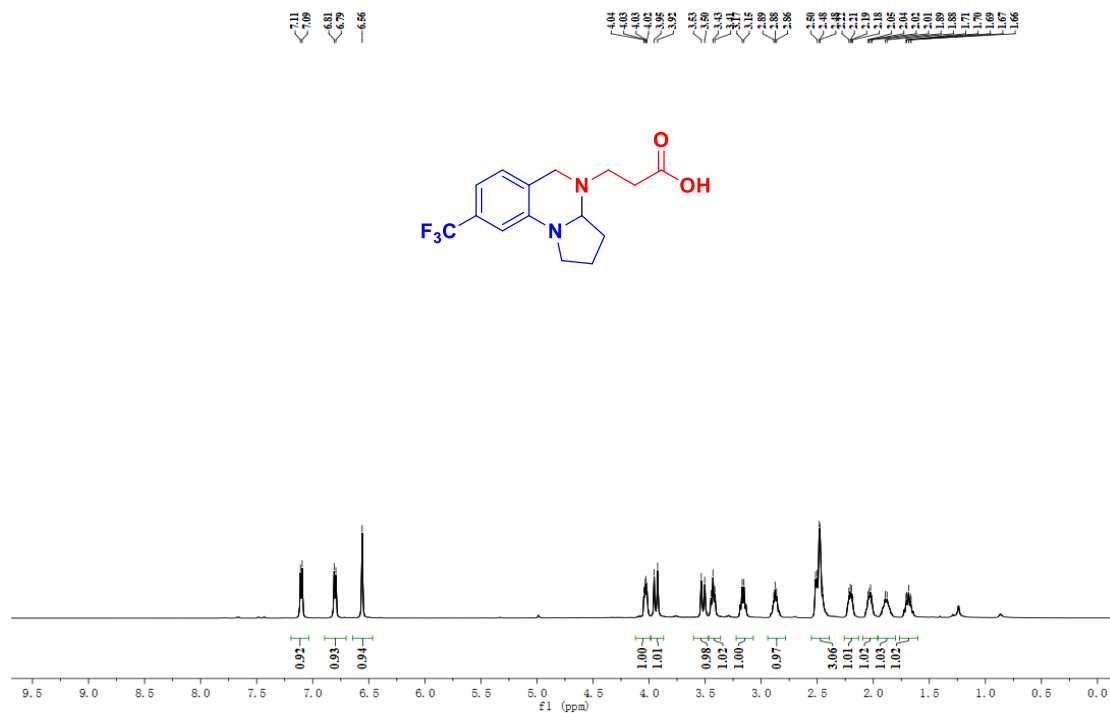
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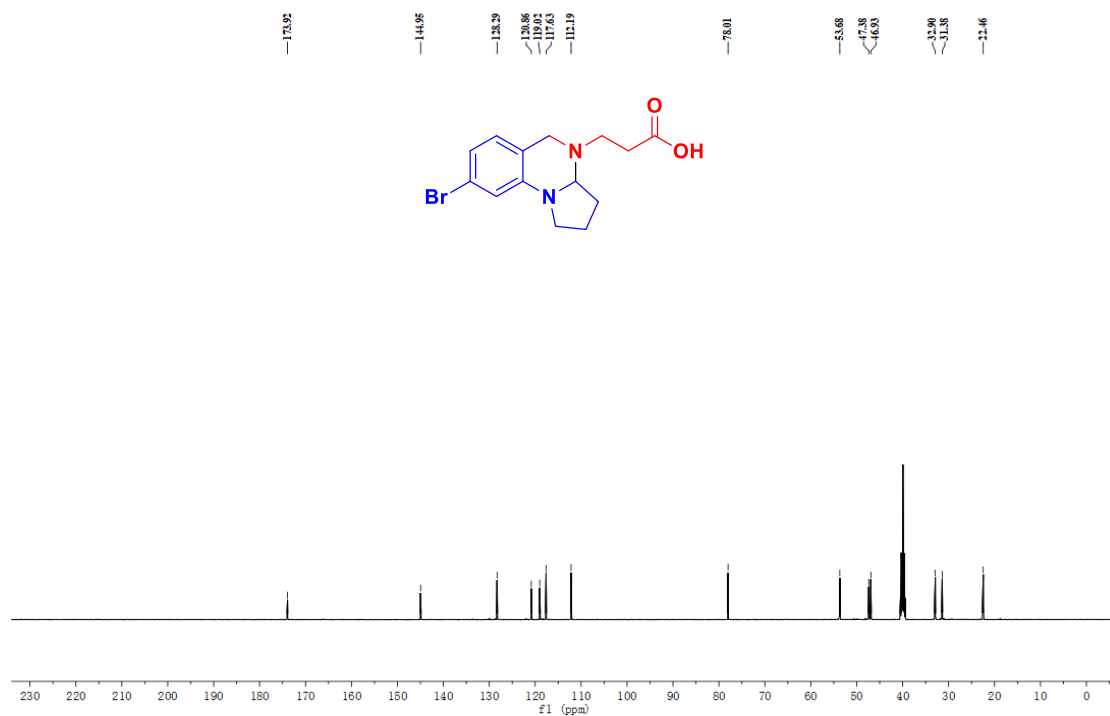
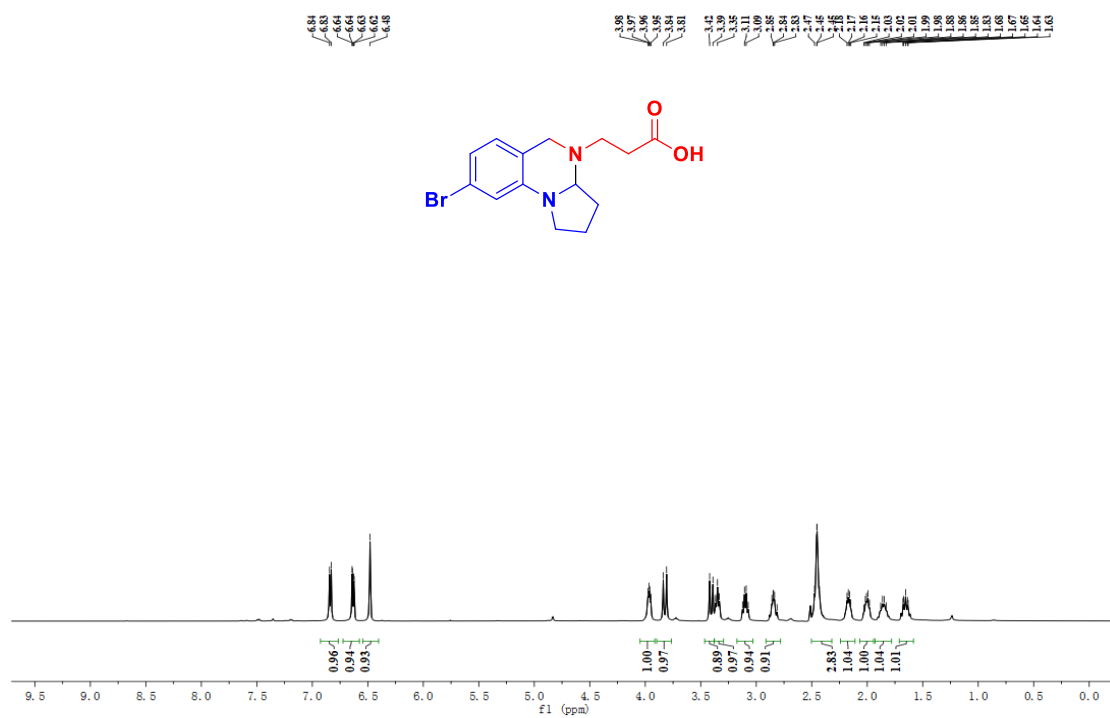
3-(9-chloro-1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)propanoic acid (3b)



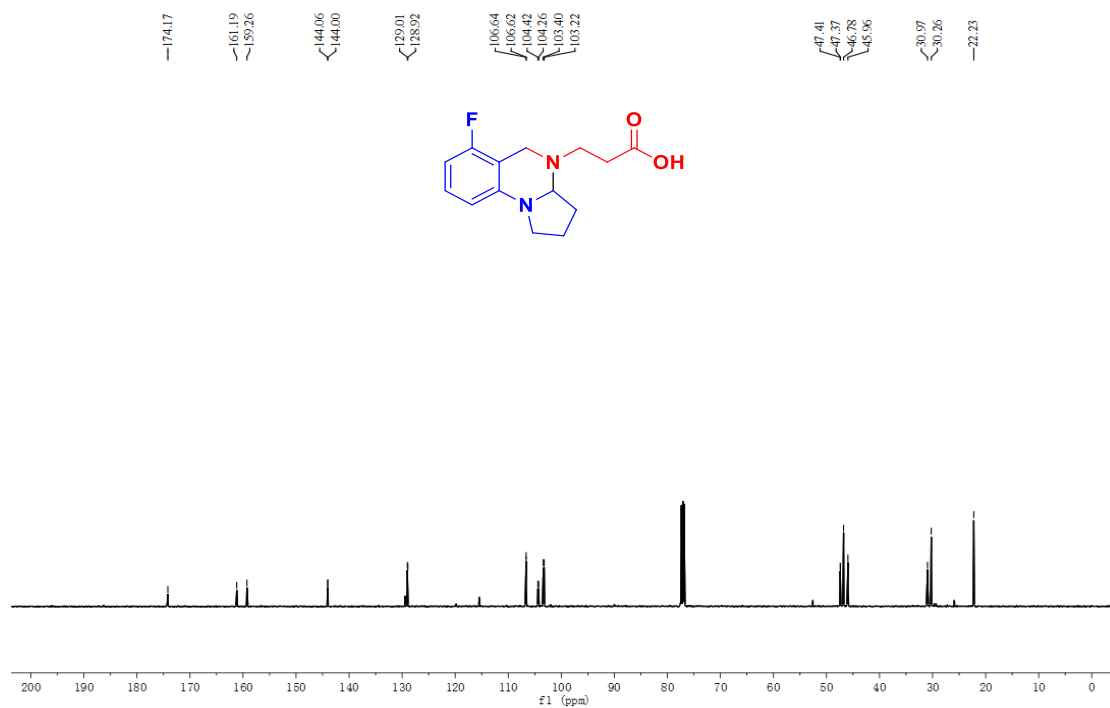
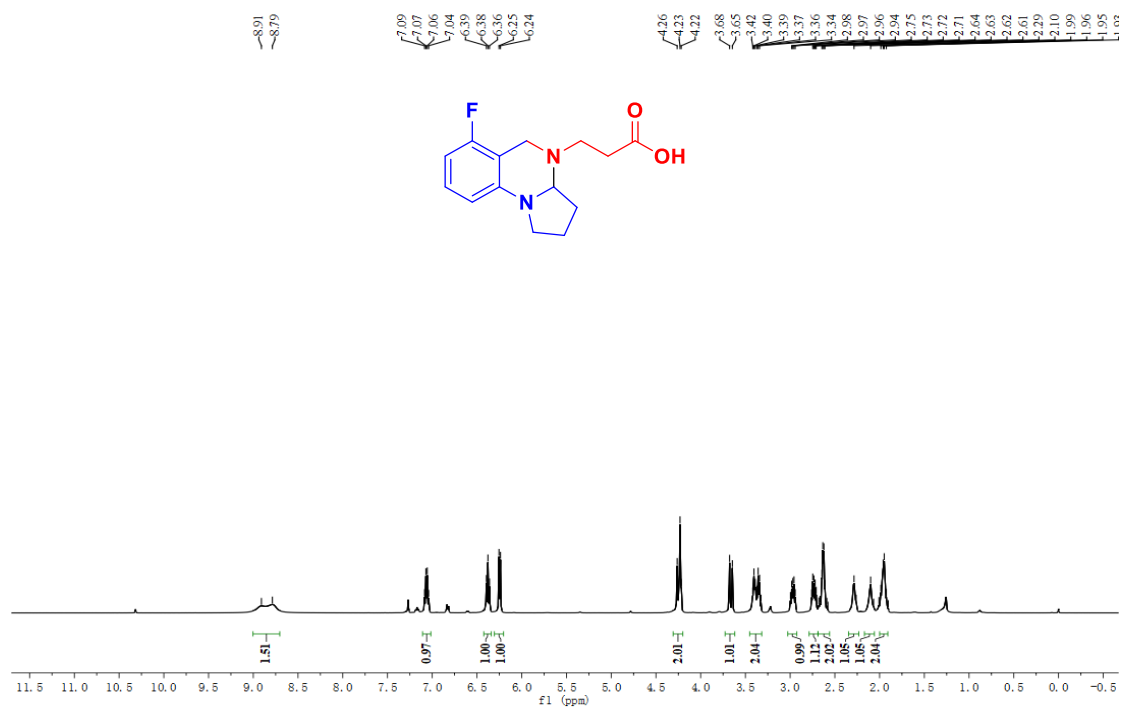
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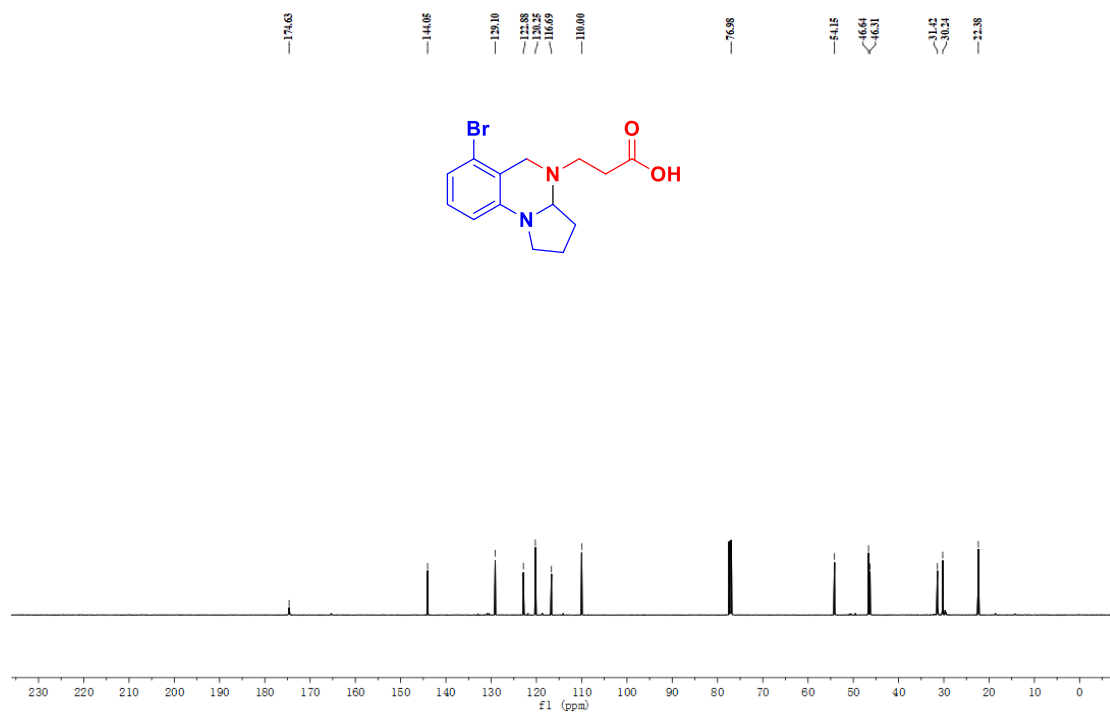
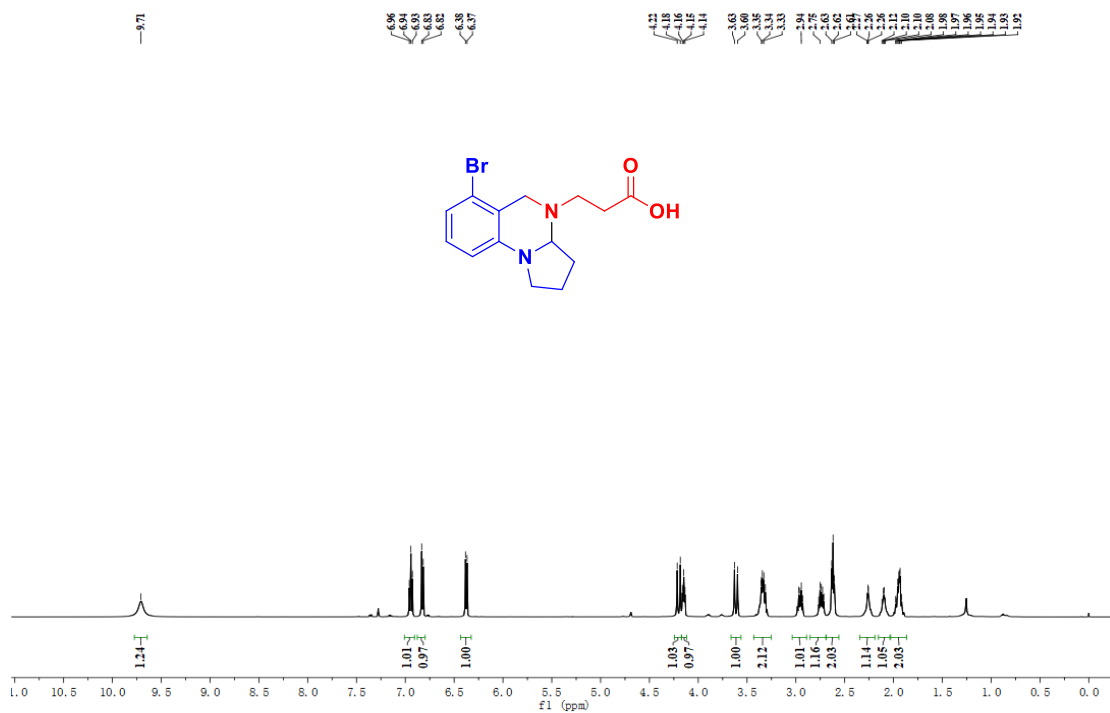
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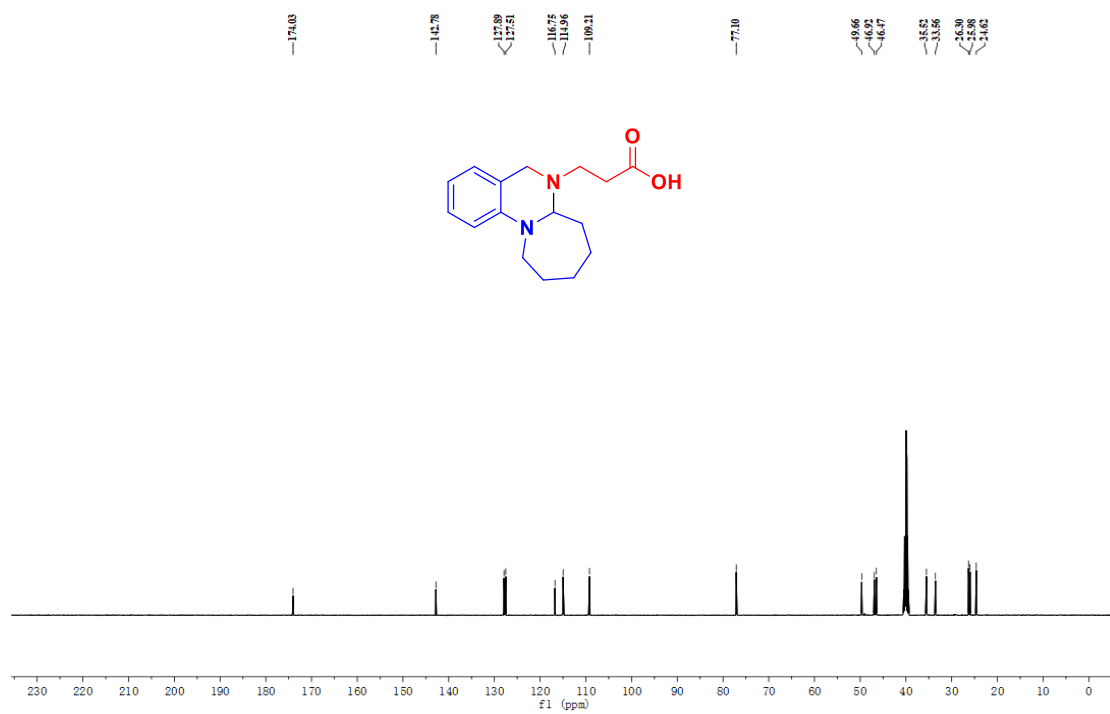
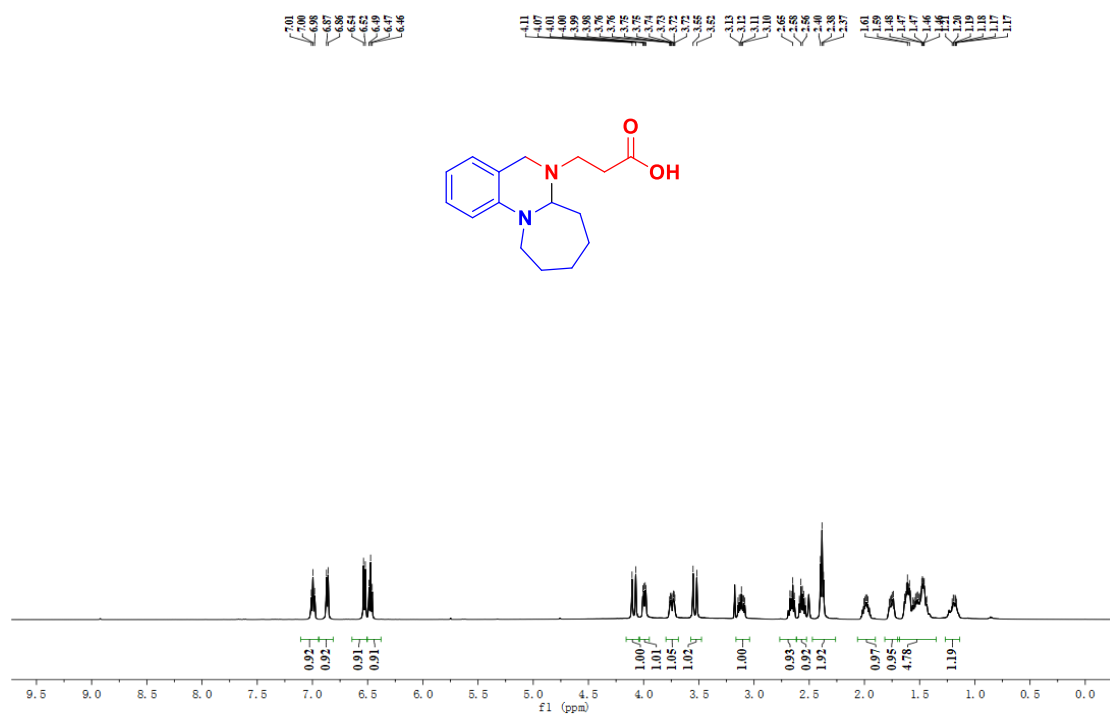
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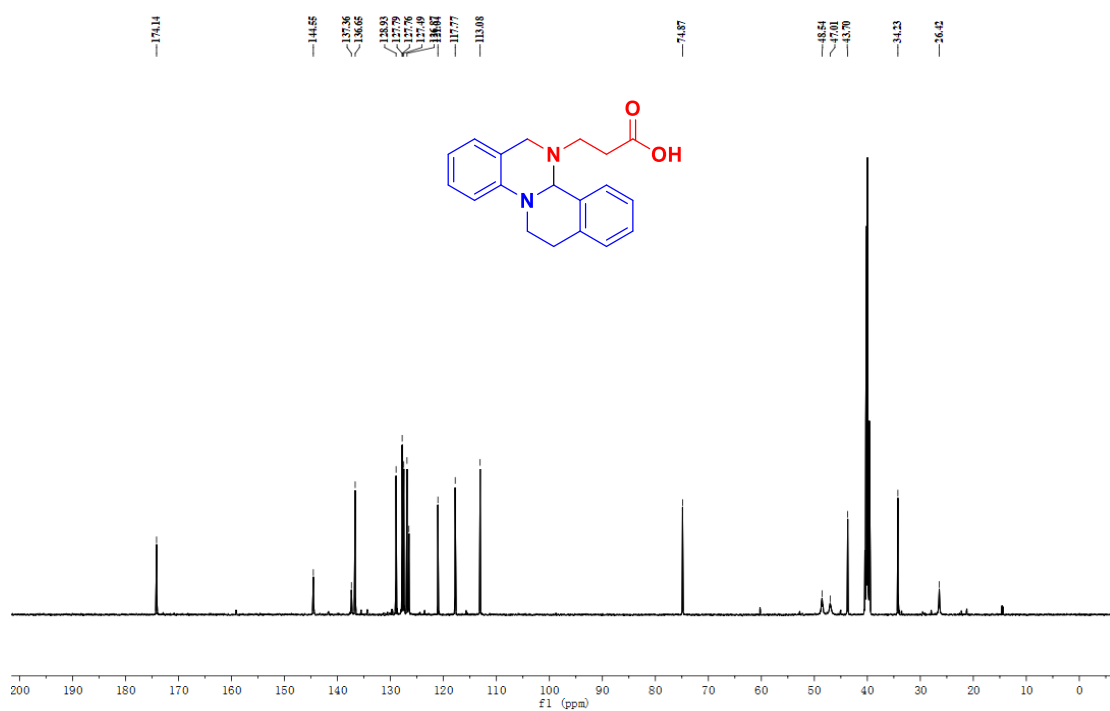
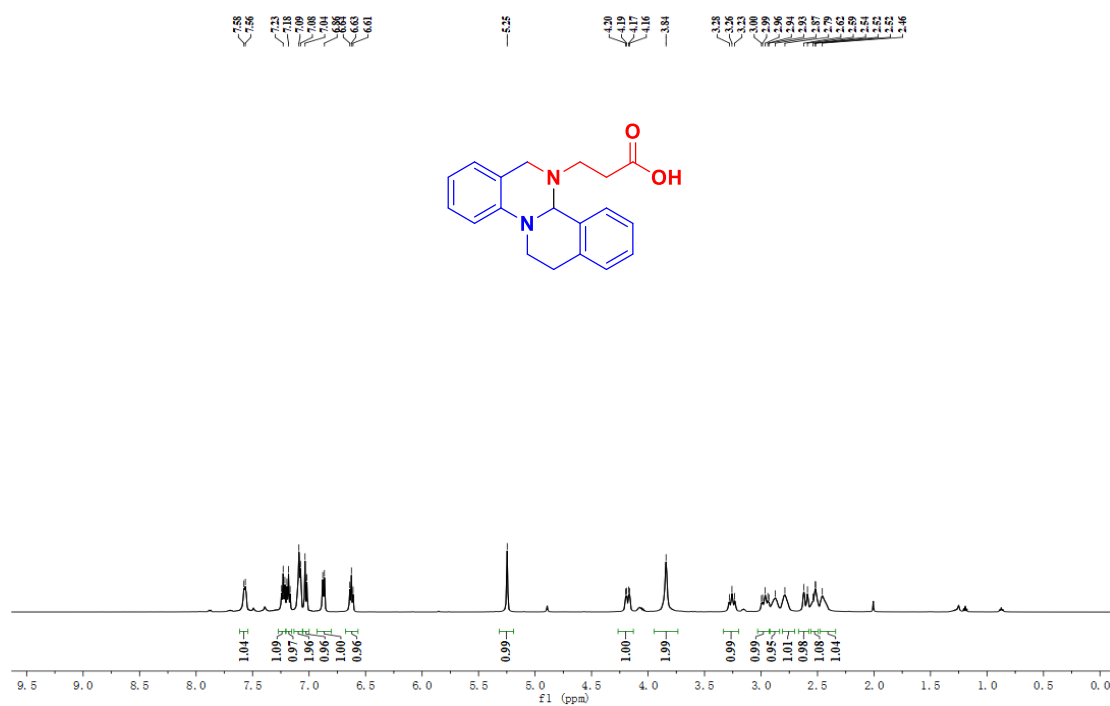
3-(6-bromo-1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)propanoic acid (3f)



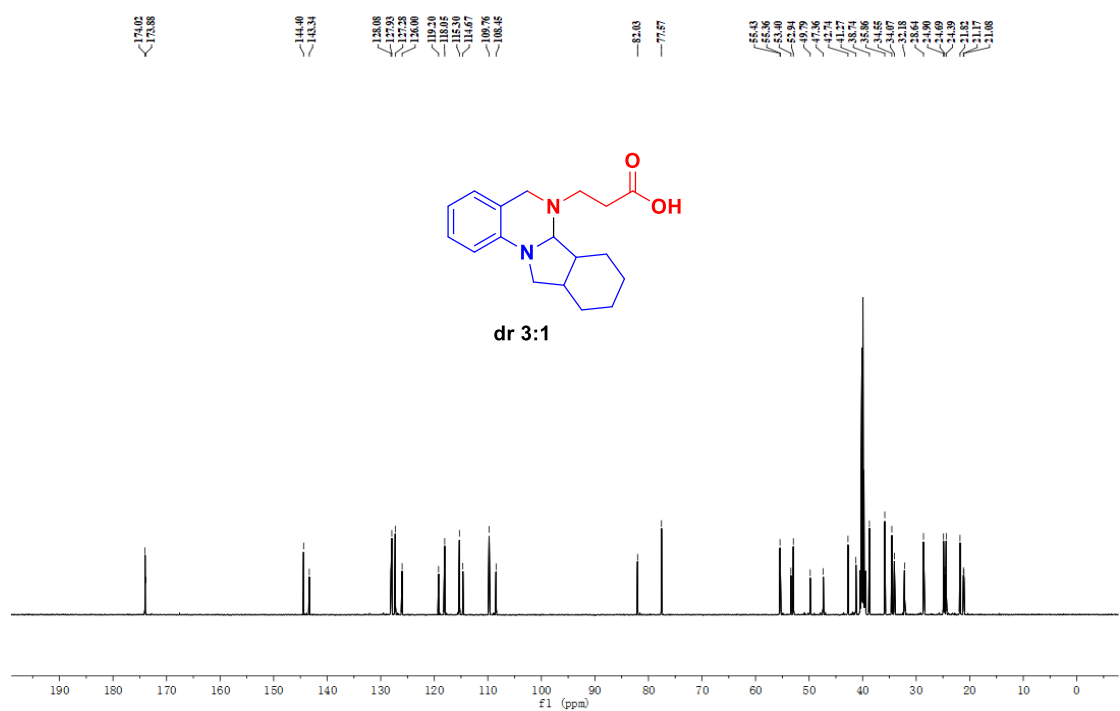
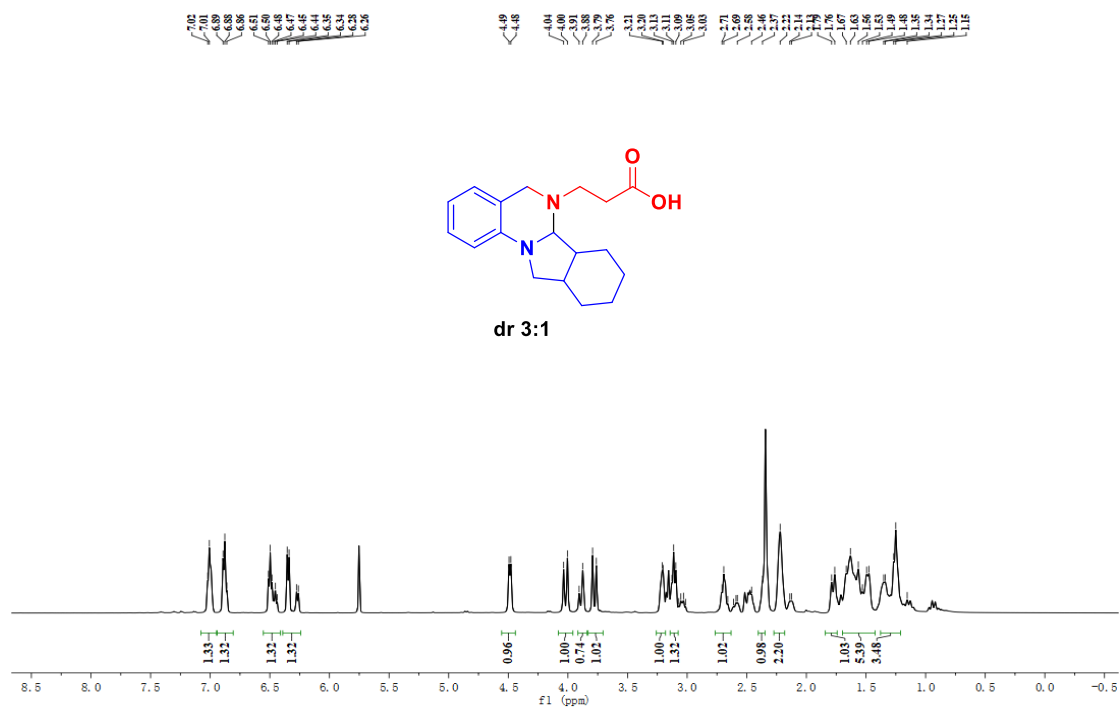
3-(6a,7,8,9,10,11-hexahydroazepino[1,2-a]quinazolin-6(5H)-yl)propanoic acid (3g)



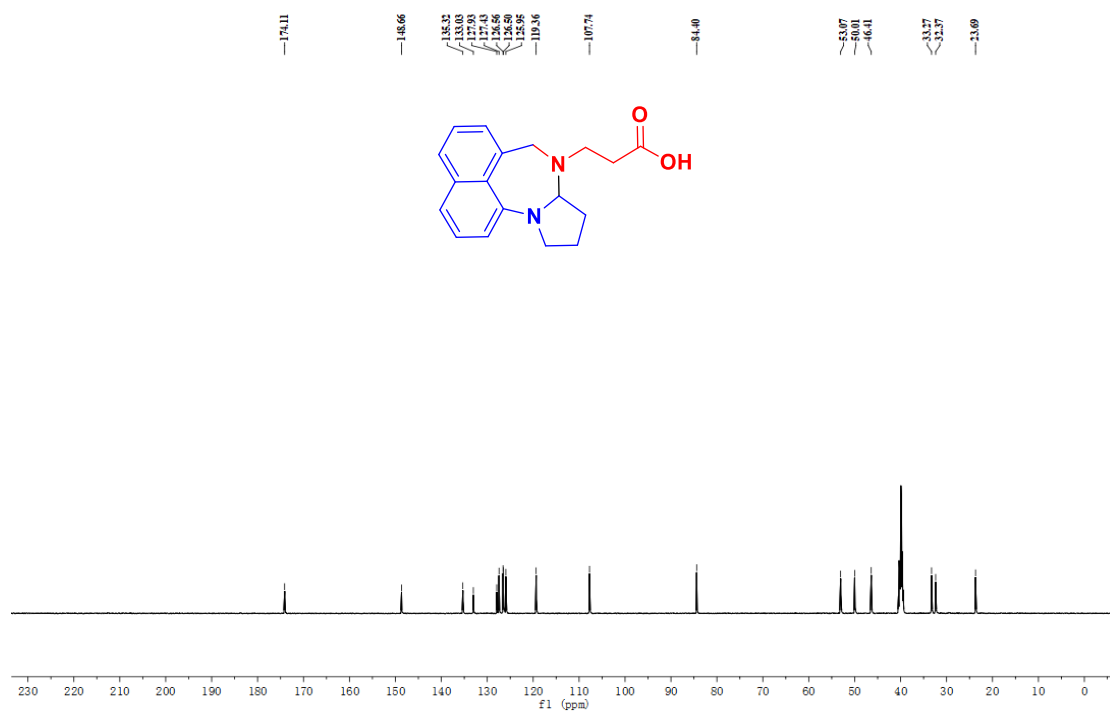
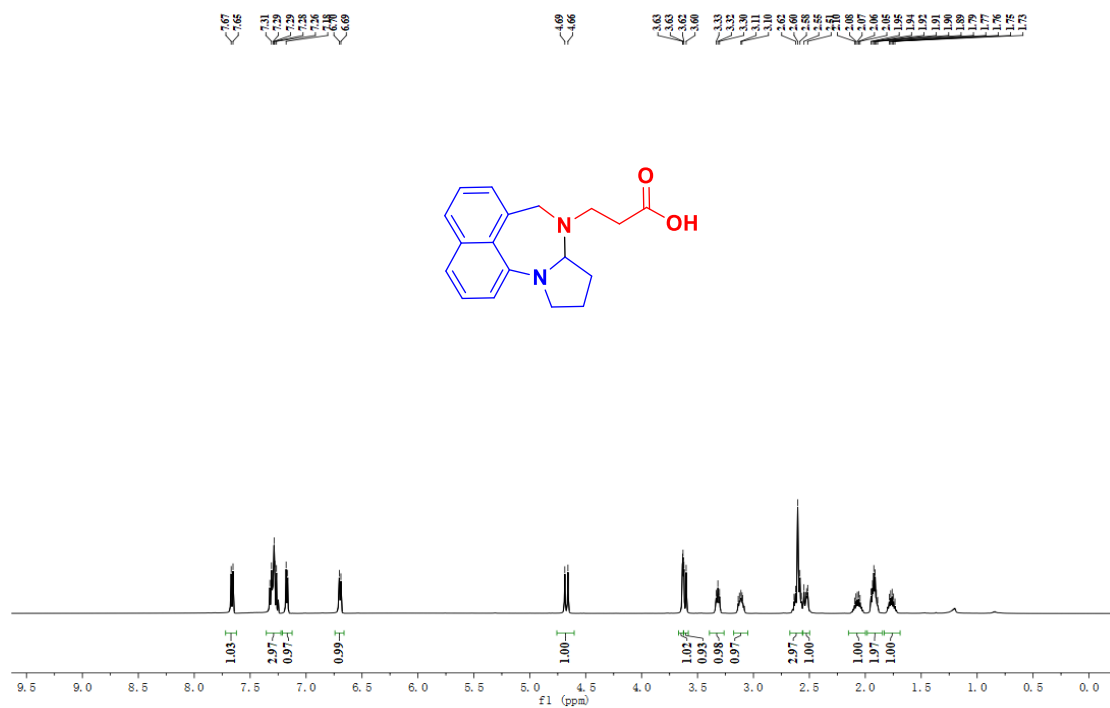
3-(12,13-dihydro-6H-isoquinolino[2,1-a]quinazolin-5(4bH)-yl)propanoic acid (3h)



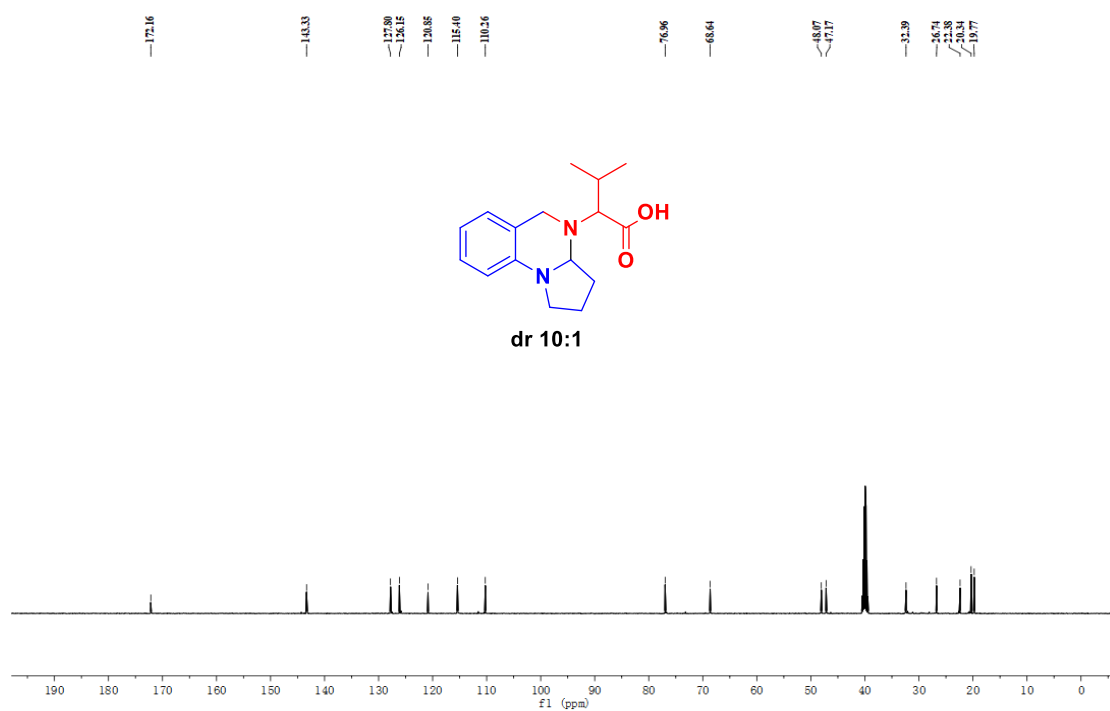
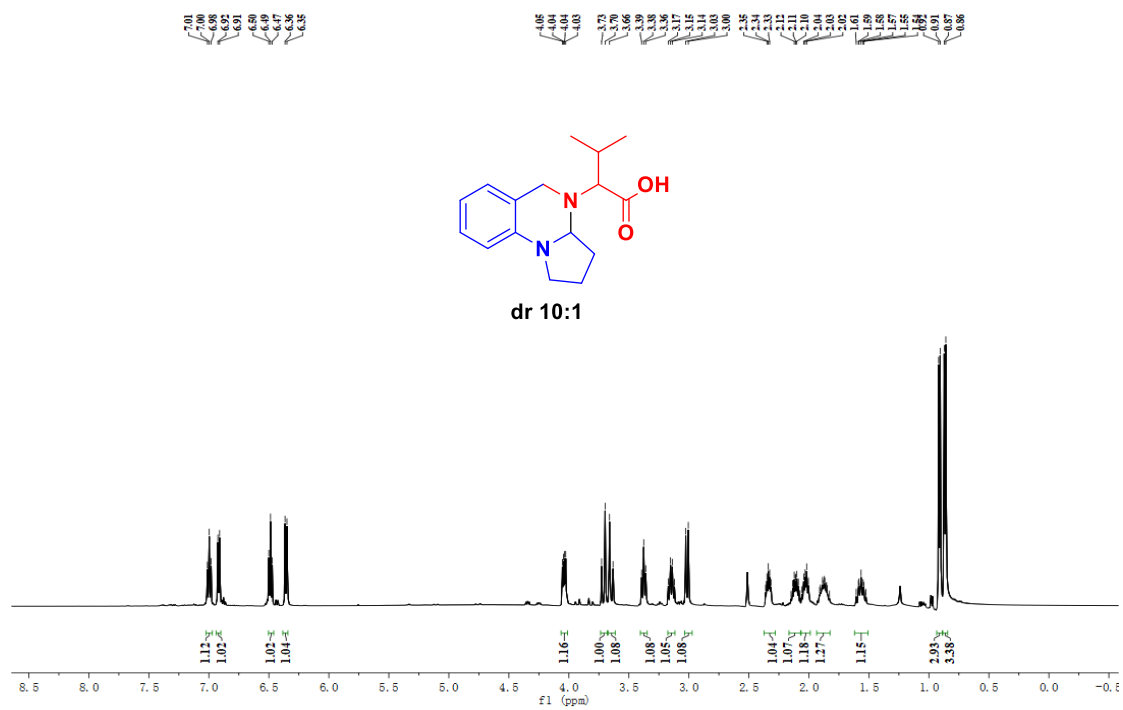
3-(6a,6b,7,8,9,10,10a,11-octahydroisindolo[2,1-a]quinazolin-6(5H)-yl)propanoic acid (3i)



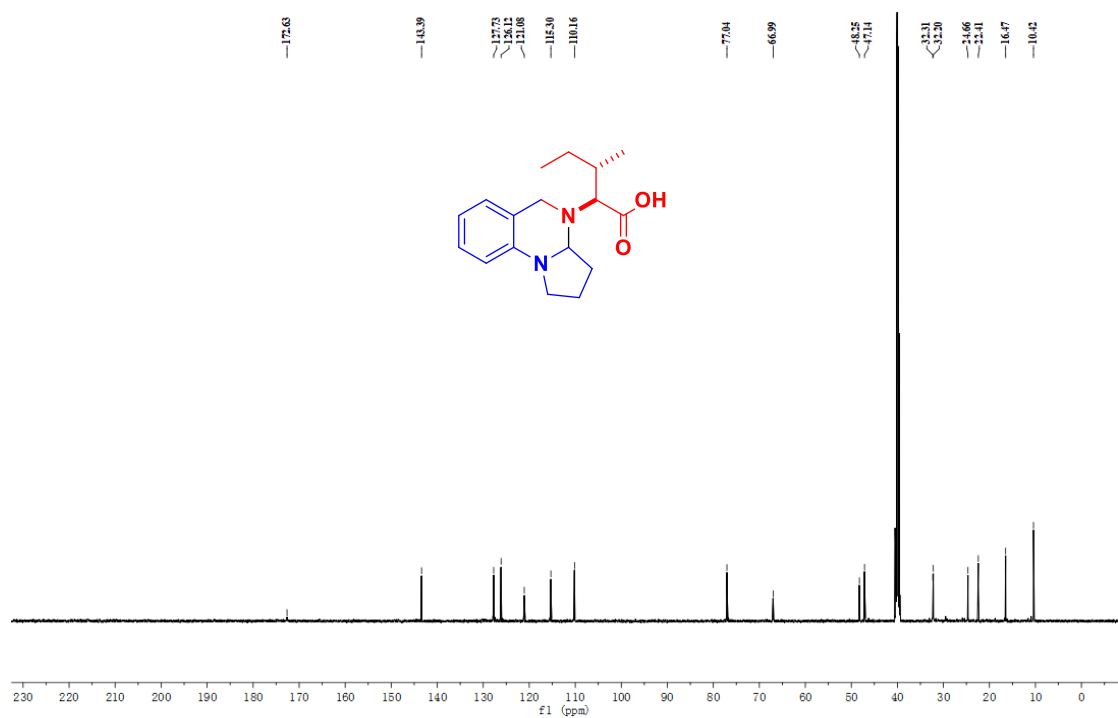
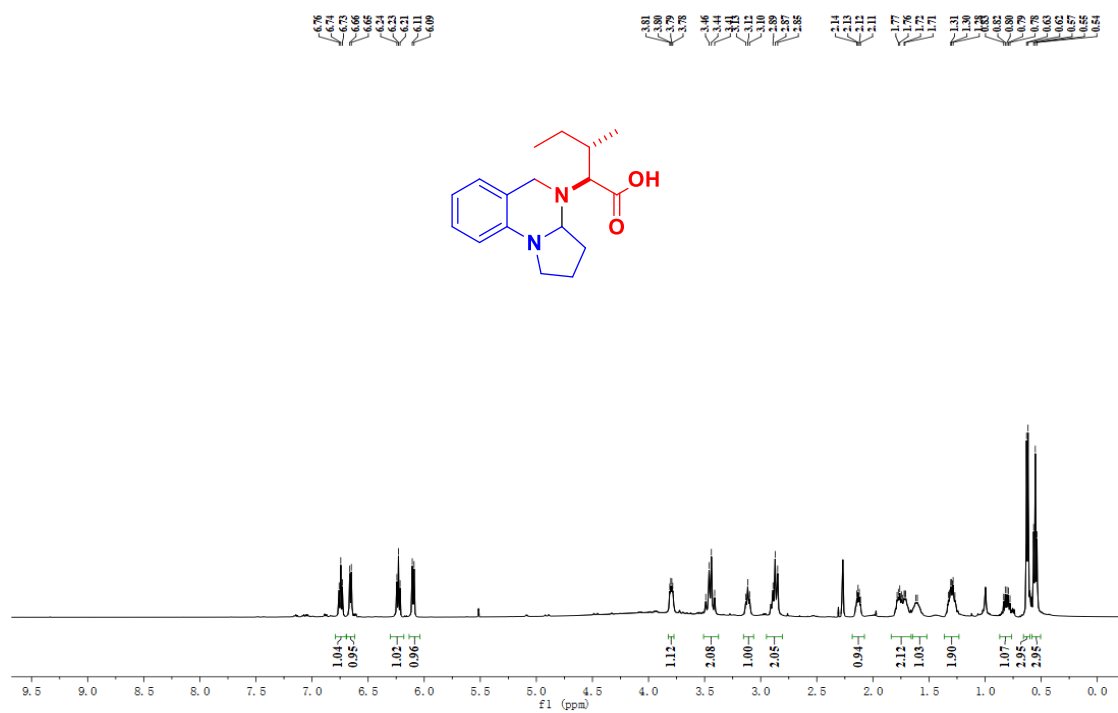
3-(1,2,3,3a-tetrahydronaphtho[1,8-ef]pyrrolo[1,2-a][1,3]diazepin-4(5H)-yl)propanoic acid (3j)



3-methyl-2-(1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)butanoic acid (5a)



3-methyl-2-(1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)pentanoic acid (5b)

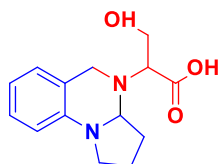


3-hydroxy-2-(1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)propanoic acid (5c)

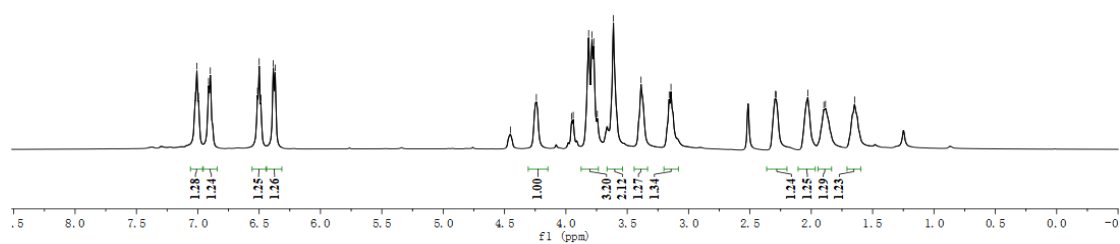
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3.77
3.74
3.39
3.16
3.14

2.29
2.03
1.90
1.88
1.66



dr 4:1



172.01

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126.31

120.62

115.48

110.49

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63.73

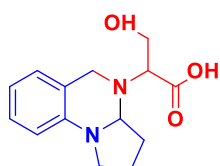
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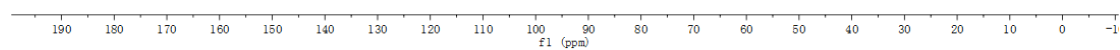
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22.42

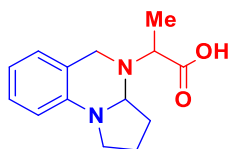


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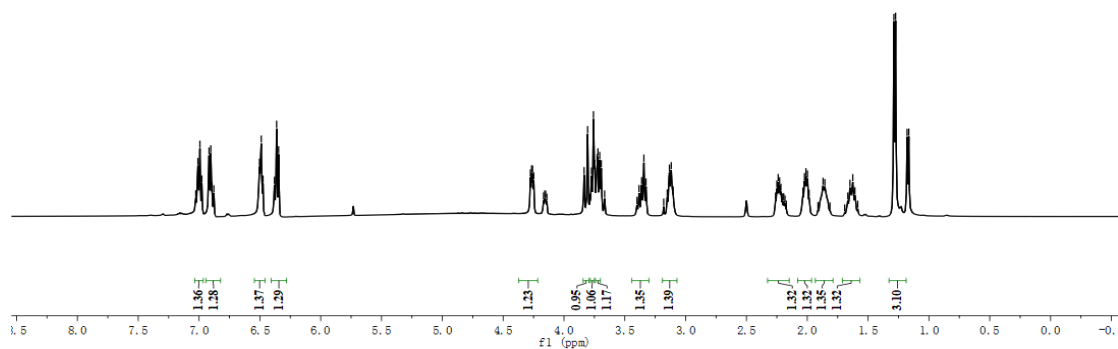


2-(1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)propanoic acid (5d)

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7.01
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6.98
6.92
6.90
6.89
6.88
6.50
6.30
6.49
6.47
6.38
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6.28
6.27
6.25
6.25
6.17
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6.15
6.14
5.83
5.81
5.79
5.77
5.76
5.75
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5.71
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5.12
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2.21
2.19
2.18
2.17
2.04
2.02
2.01
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1.65
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1.61
1.59
1.29
1.27
1.18
1.17



dr 3:1



174.09
173.67

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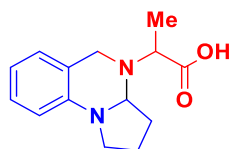
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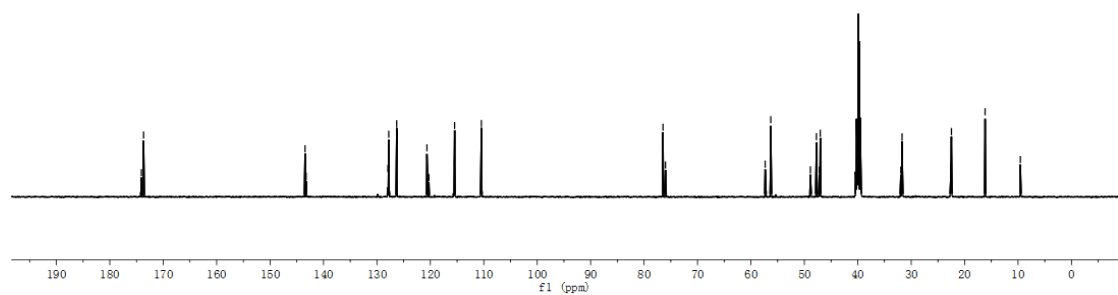
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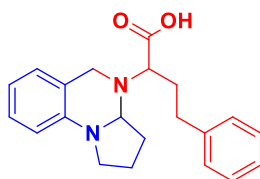


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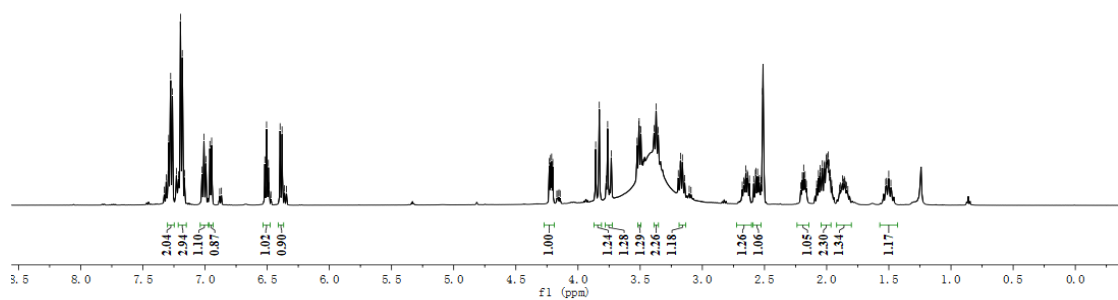


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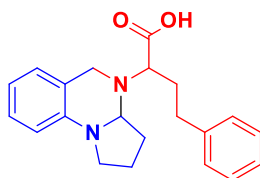
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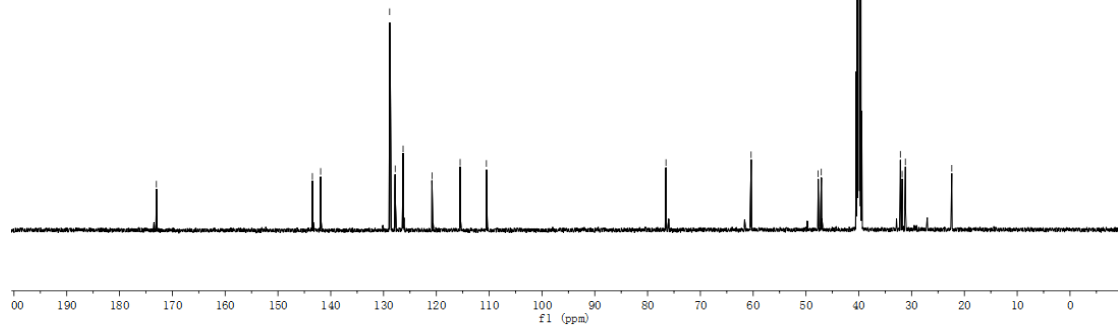
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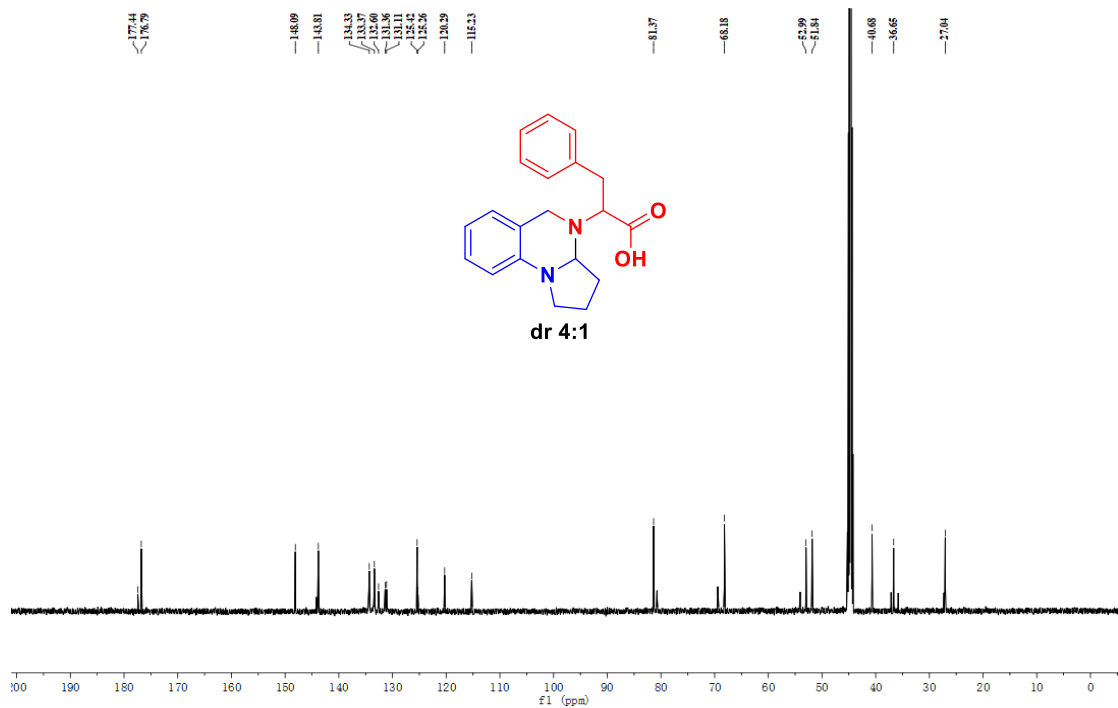
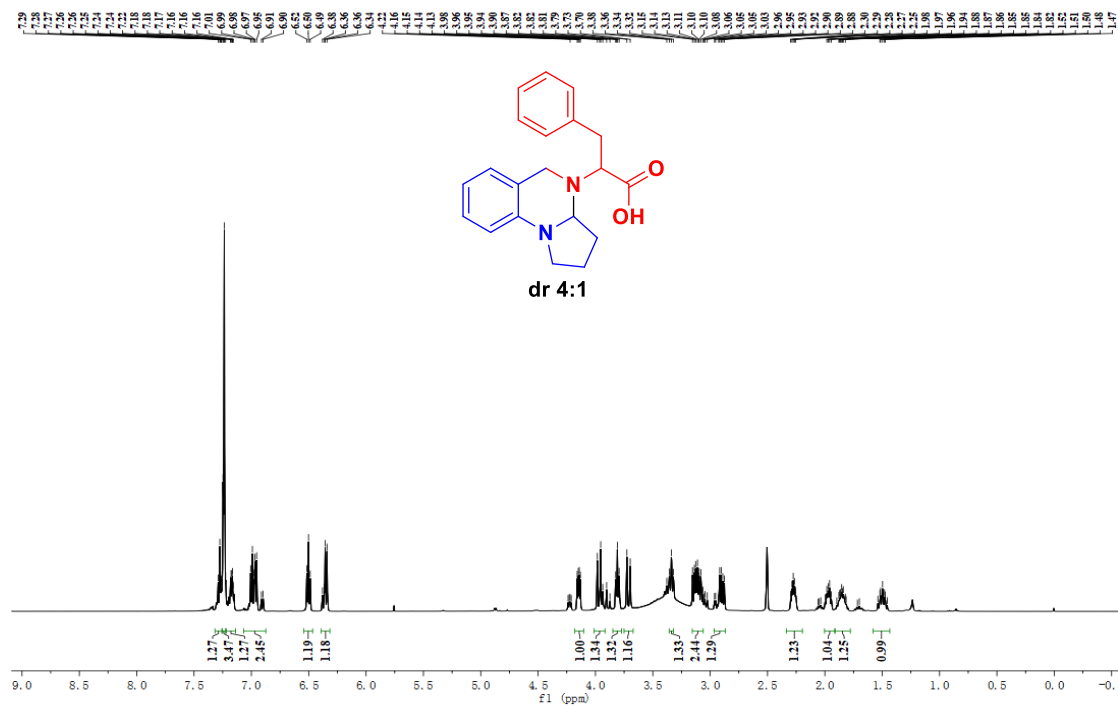
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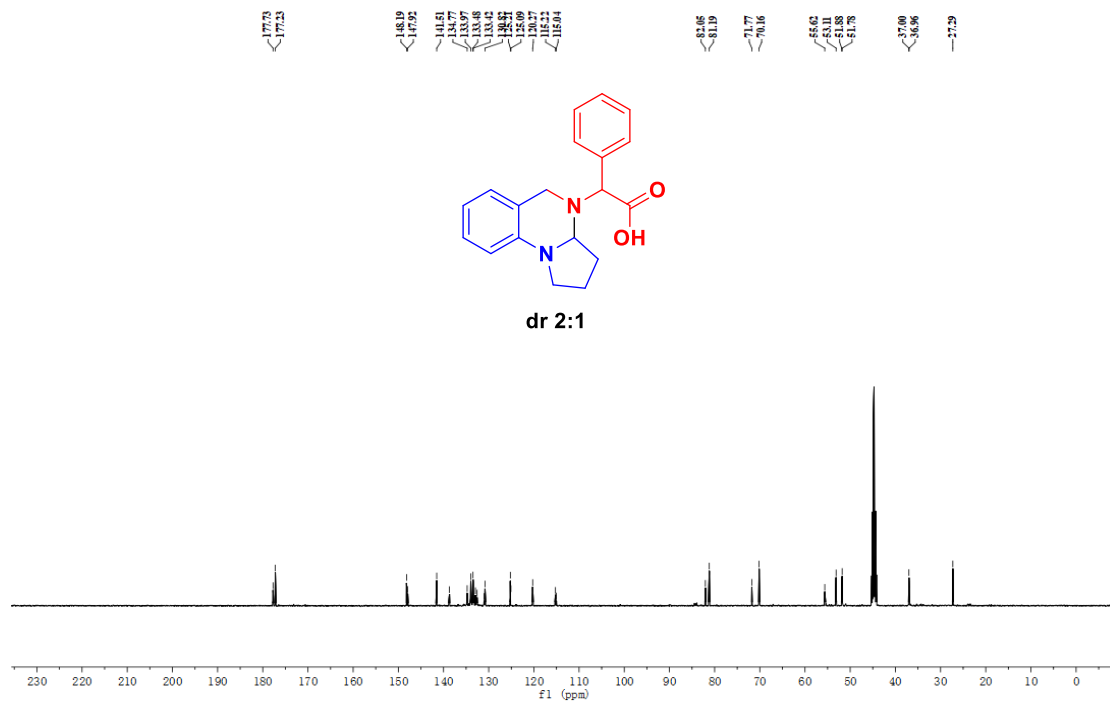
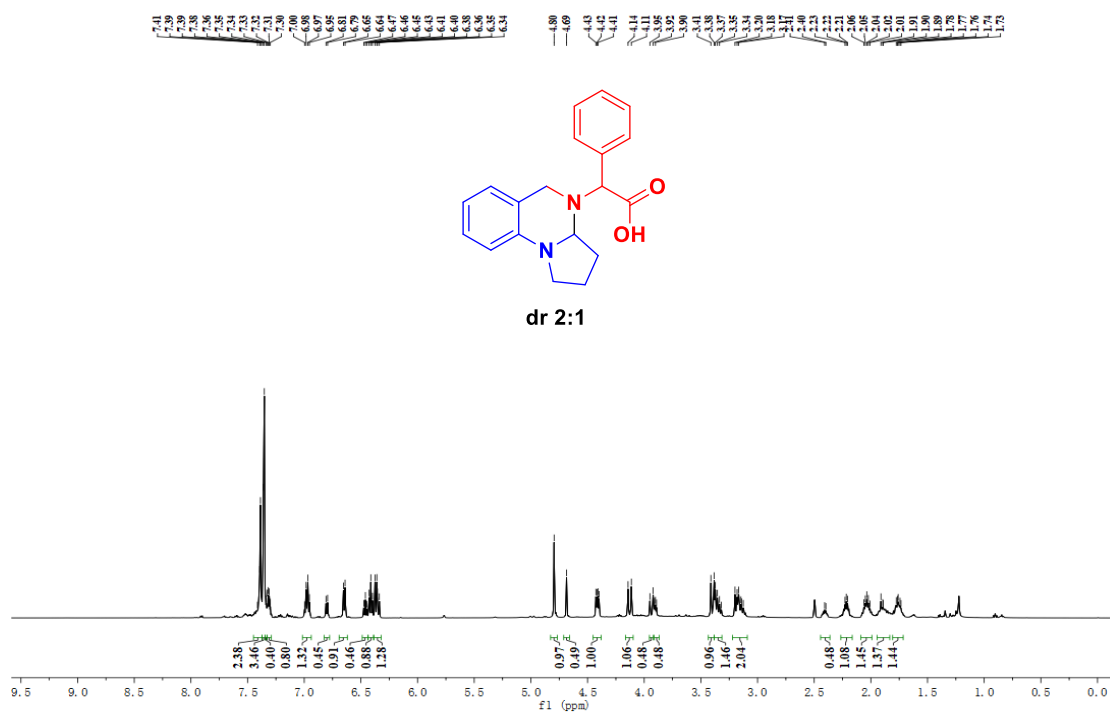
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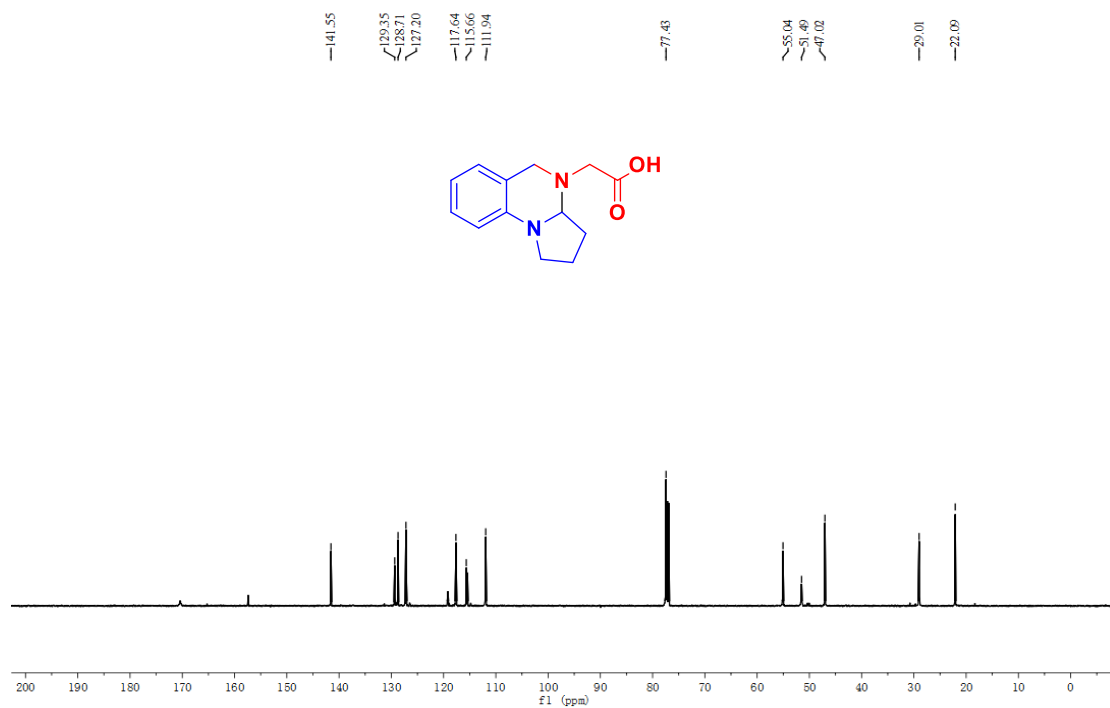
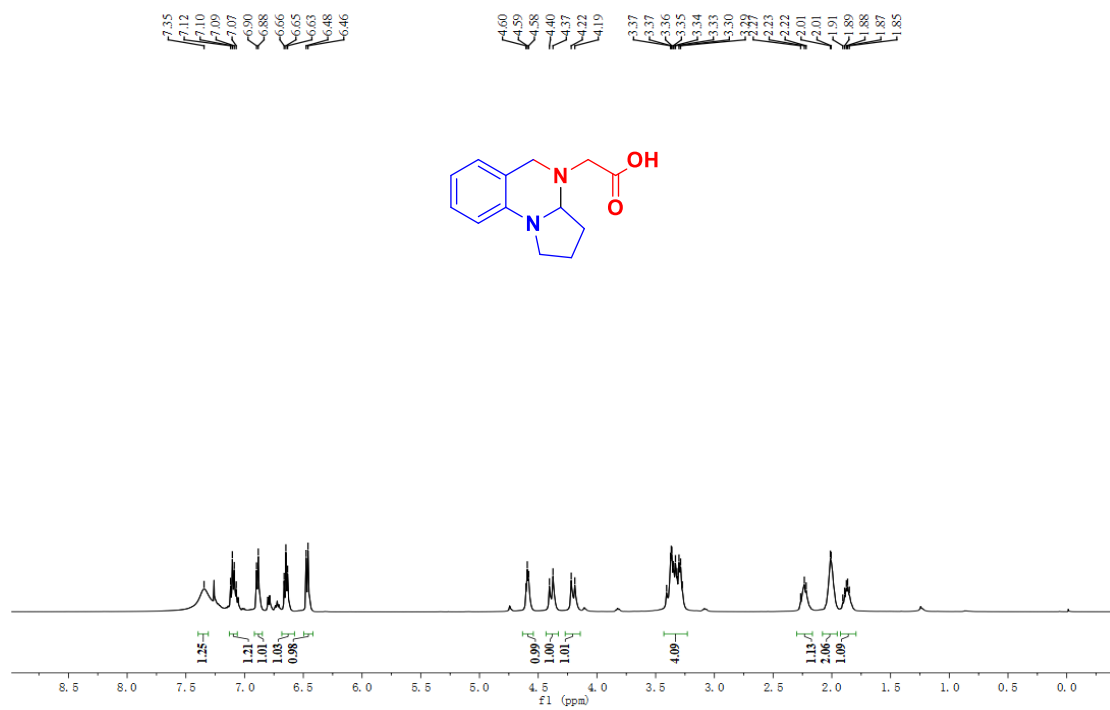
3-phenyl-2-(1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)propanoic acid (5f)



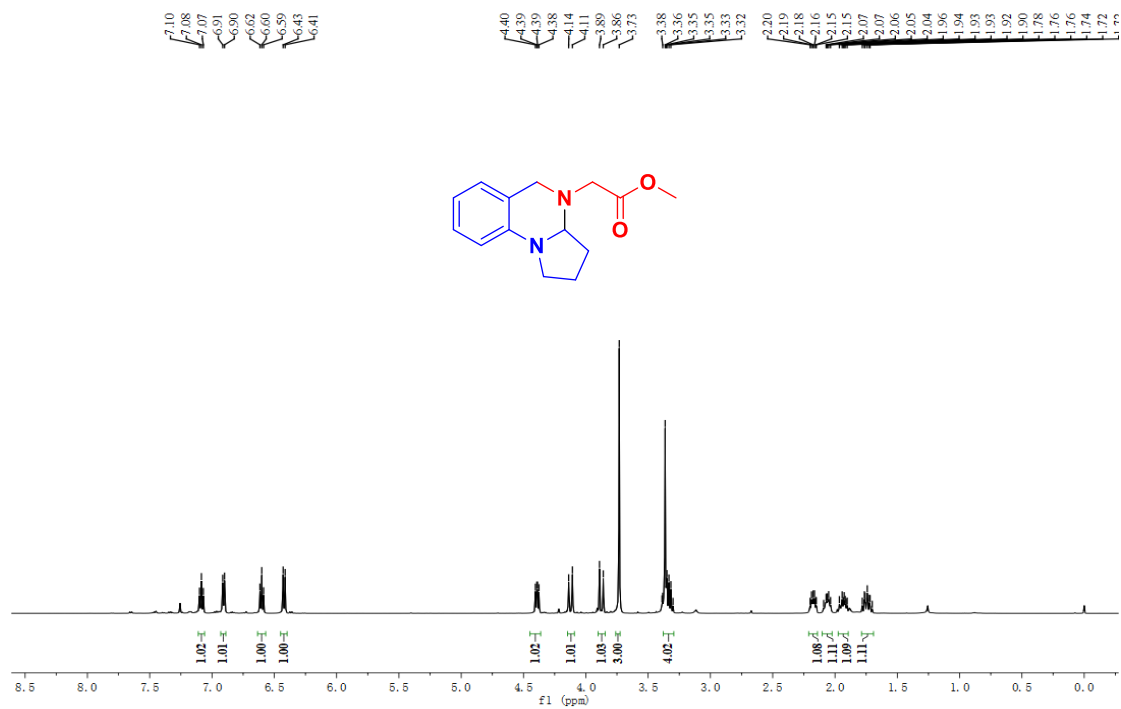
2-phenyl-2-(1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)acetic acid (5g)



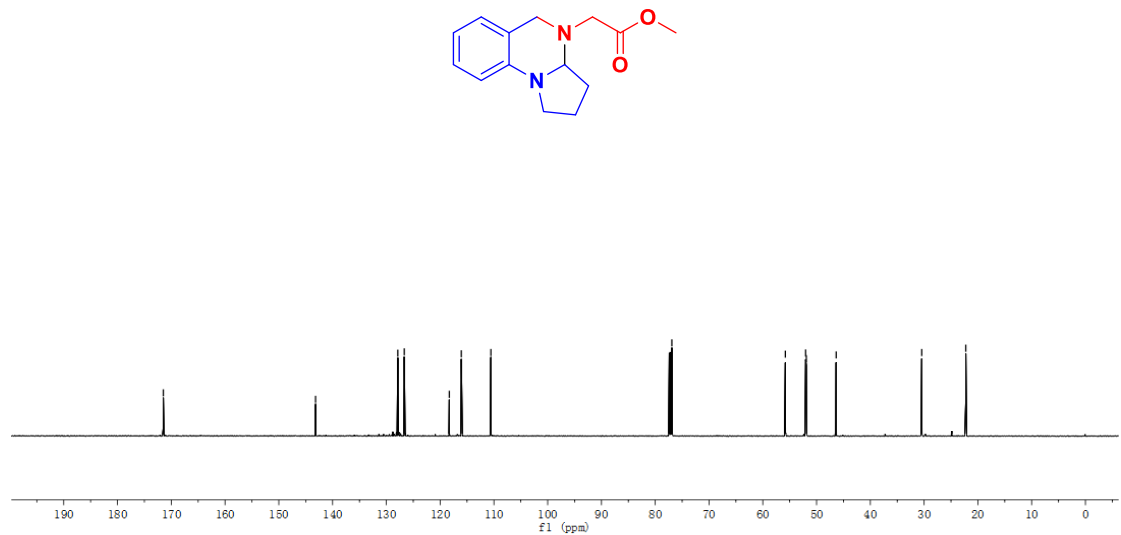
2-(1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)acetic acid (5h)



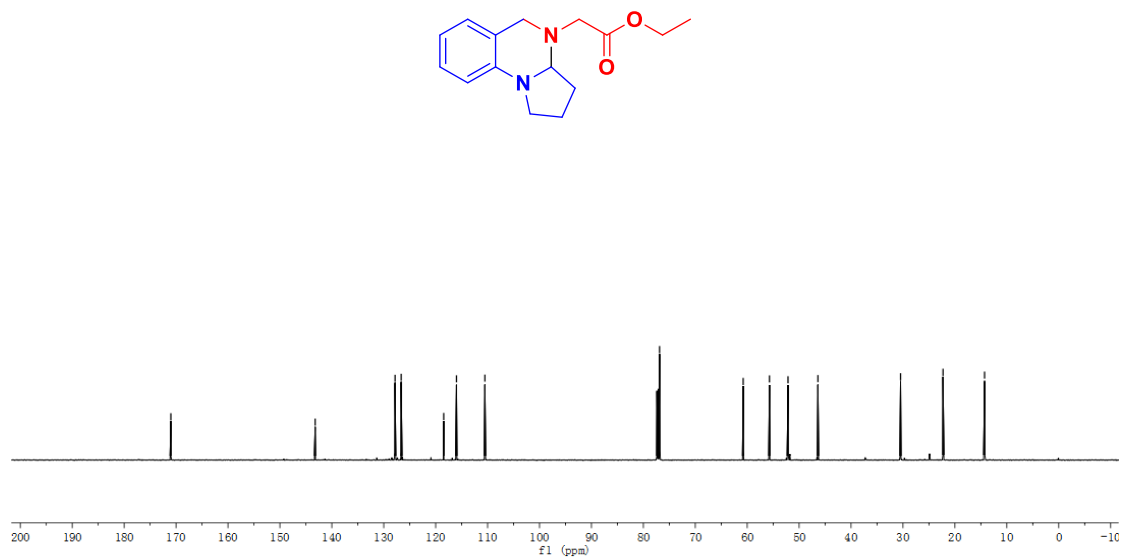
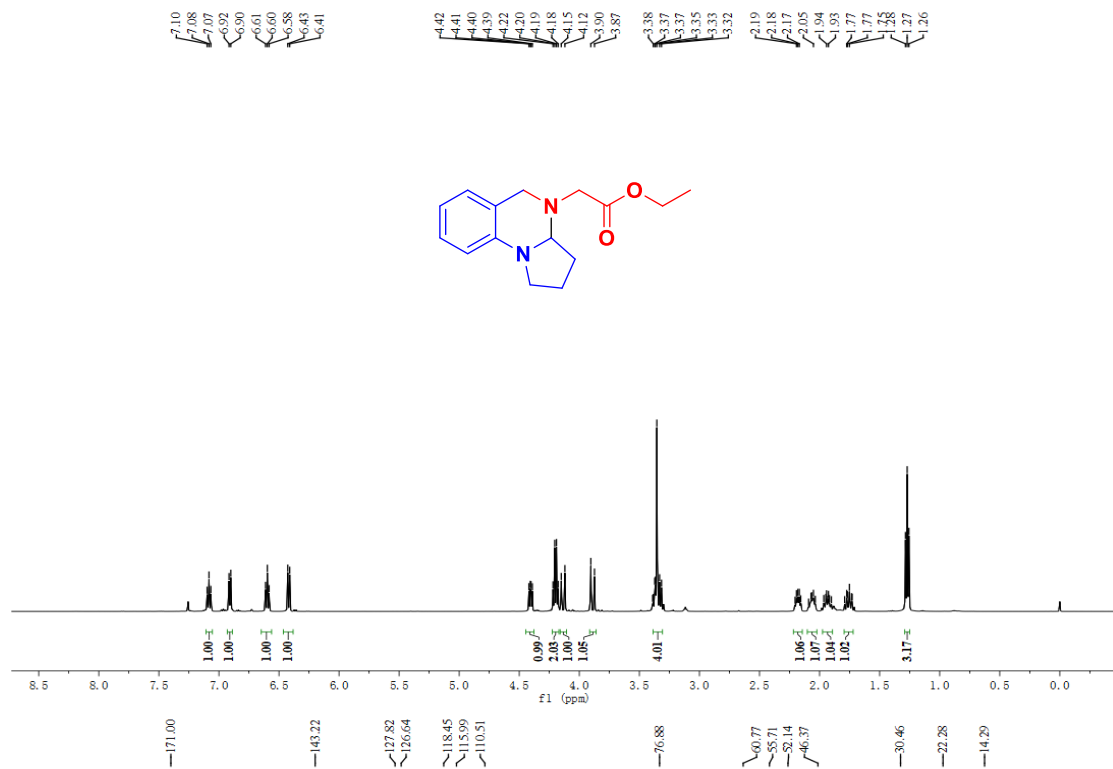
methyl 2-(1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)acetate (7a)



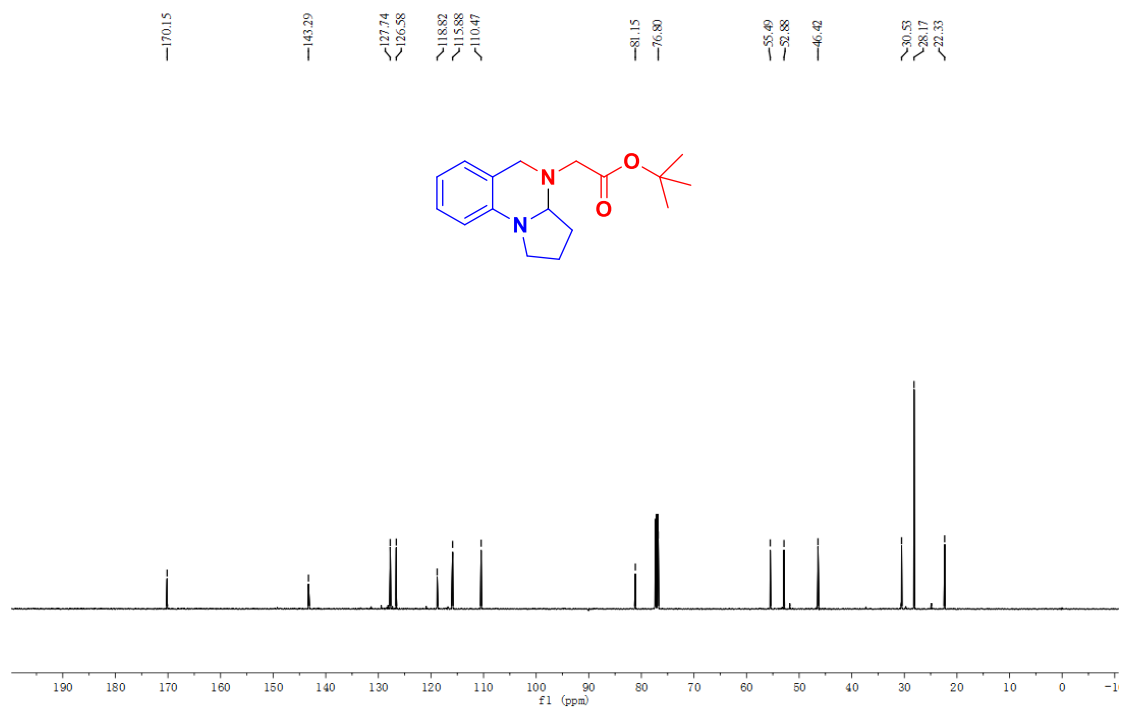
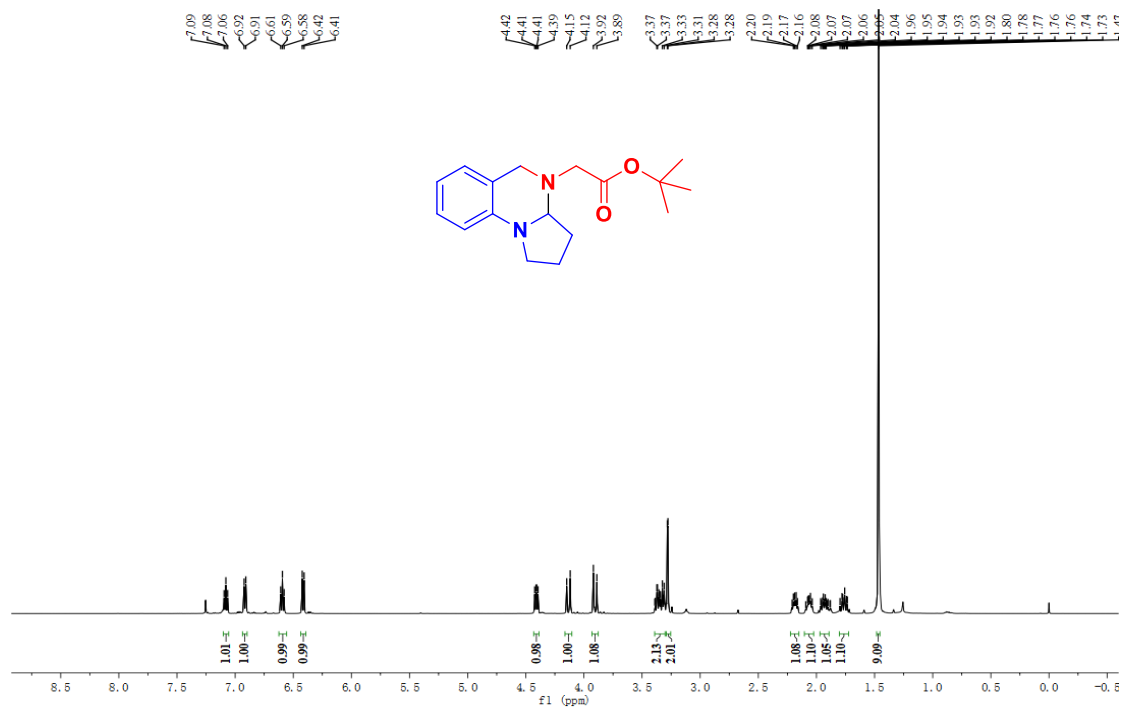
171.48, 143.17, 127.87, 126.67, 118.29, 116.06, 110.56, 76.91, 55.81, 51.03, 51.90, 46.35, 30.43, 22.25



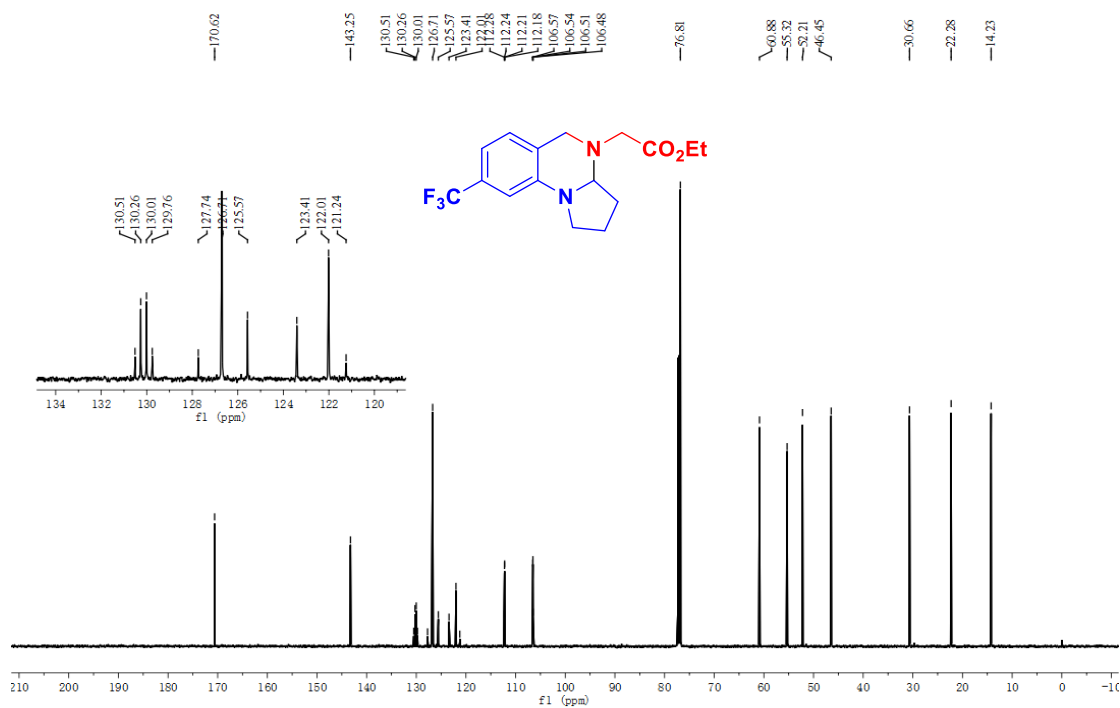
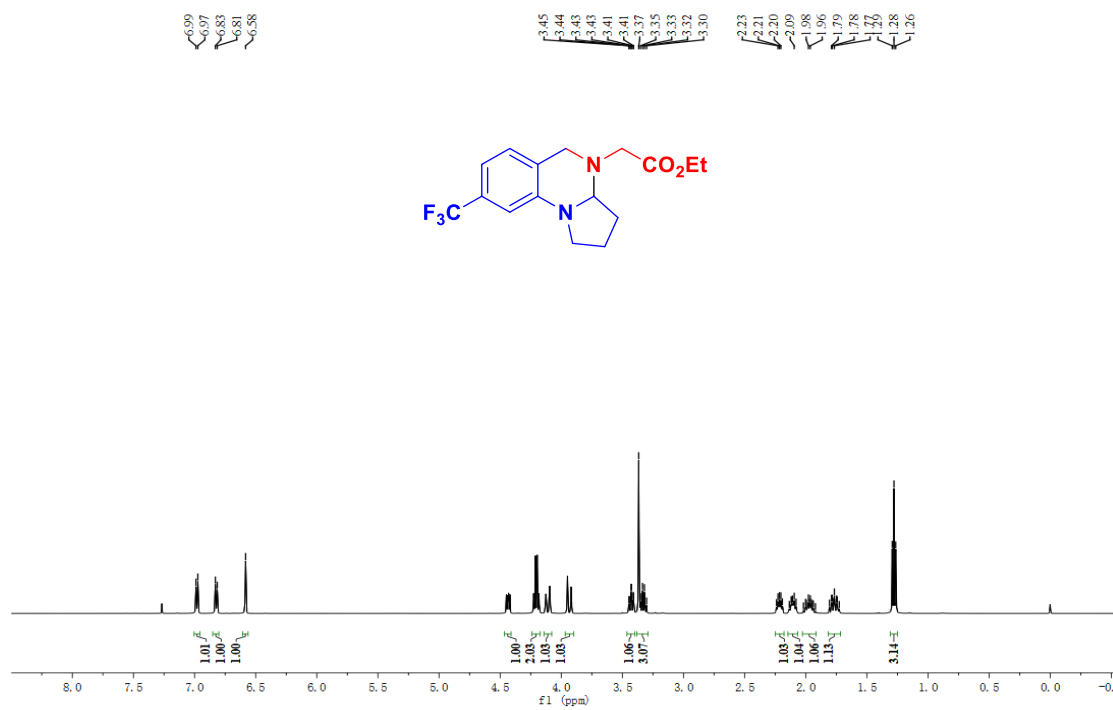
ethyl-(1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)acetate (7b)



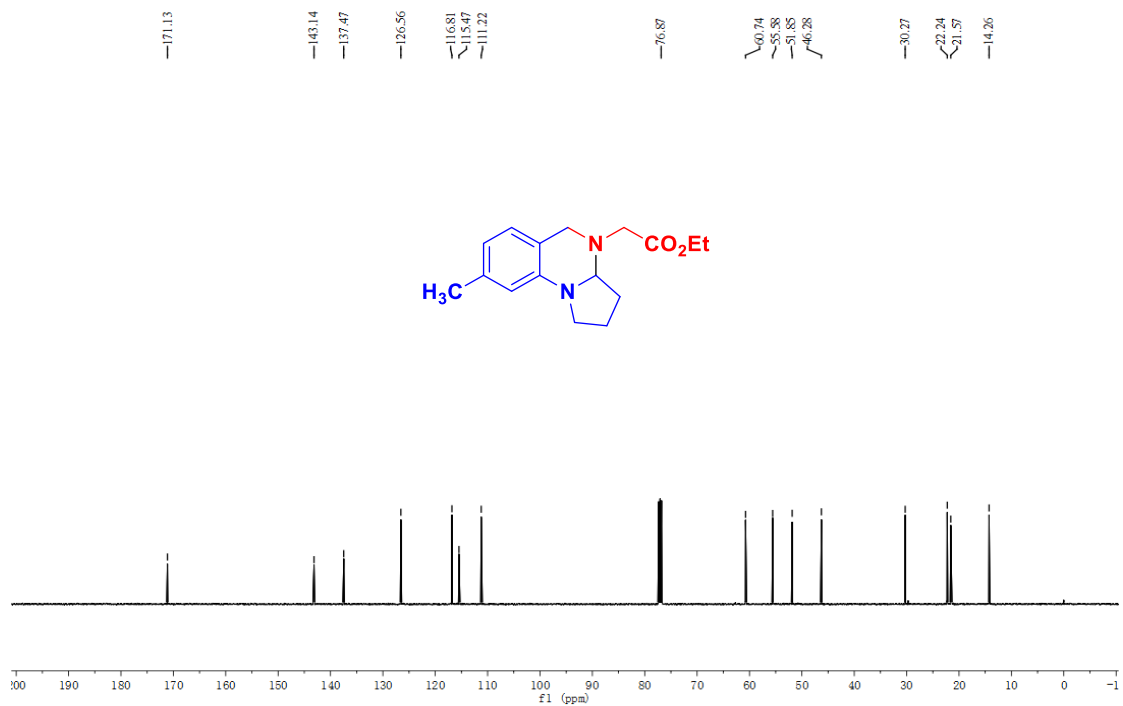
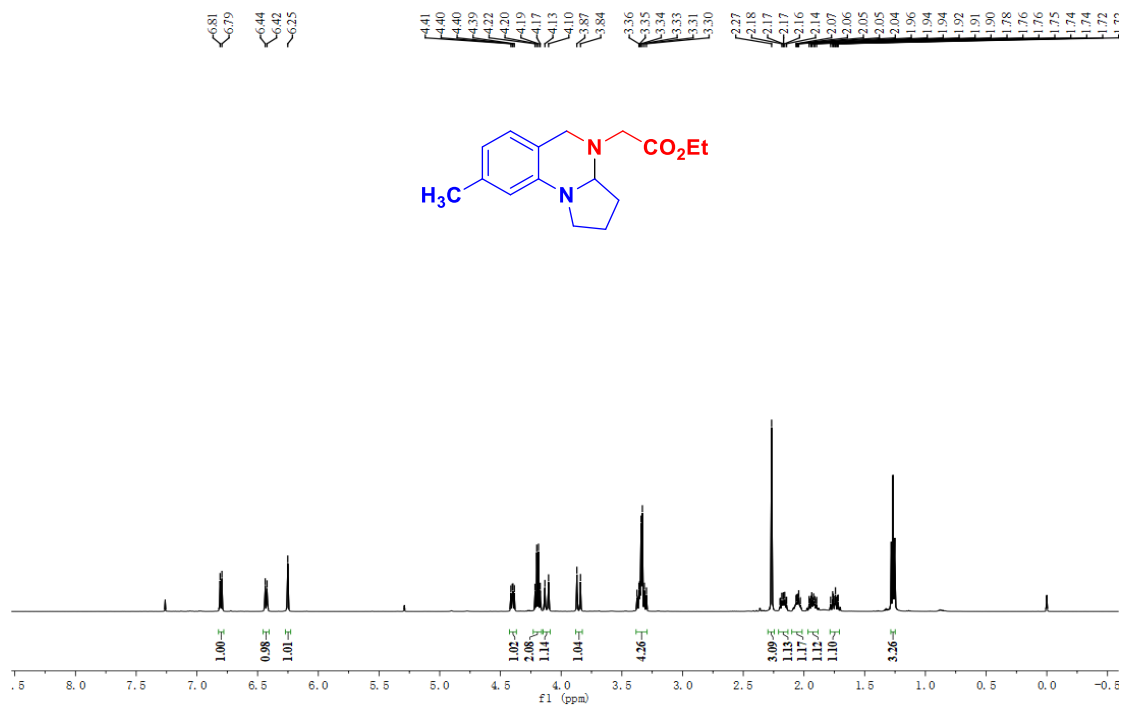
tert-butyl 2-(1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)acetate (7d)



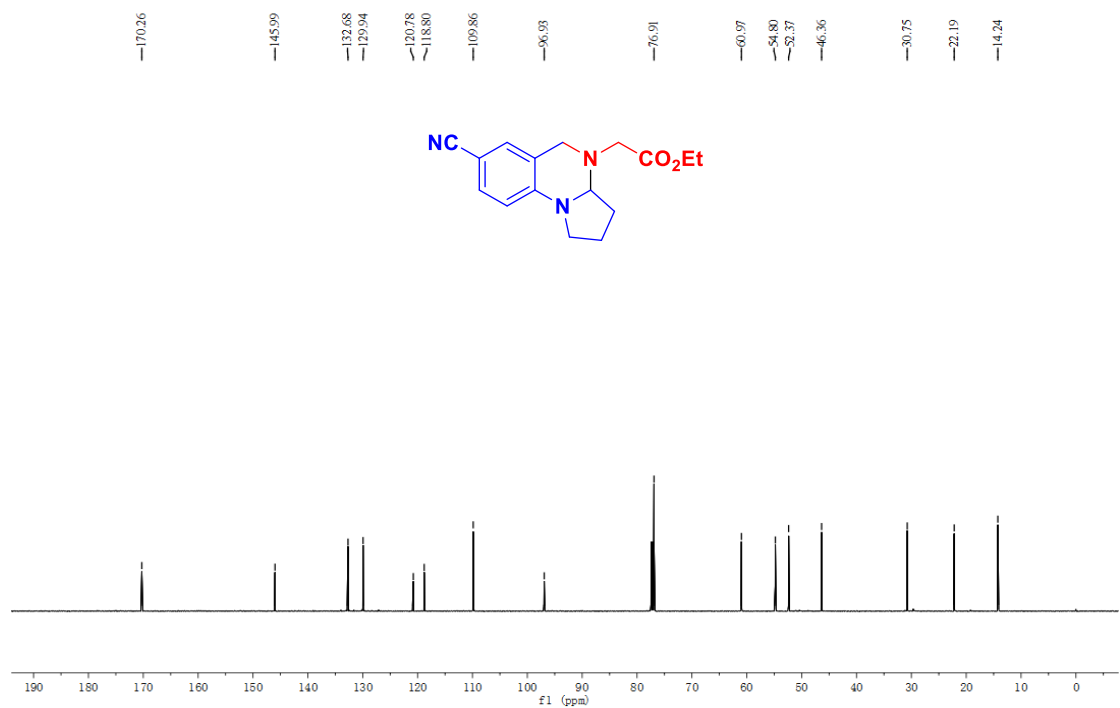
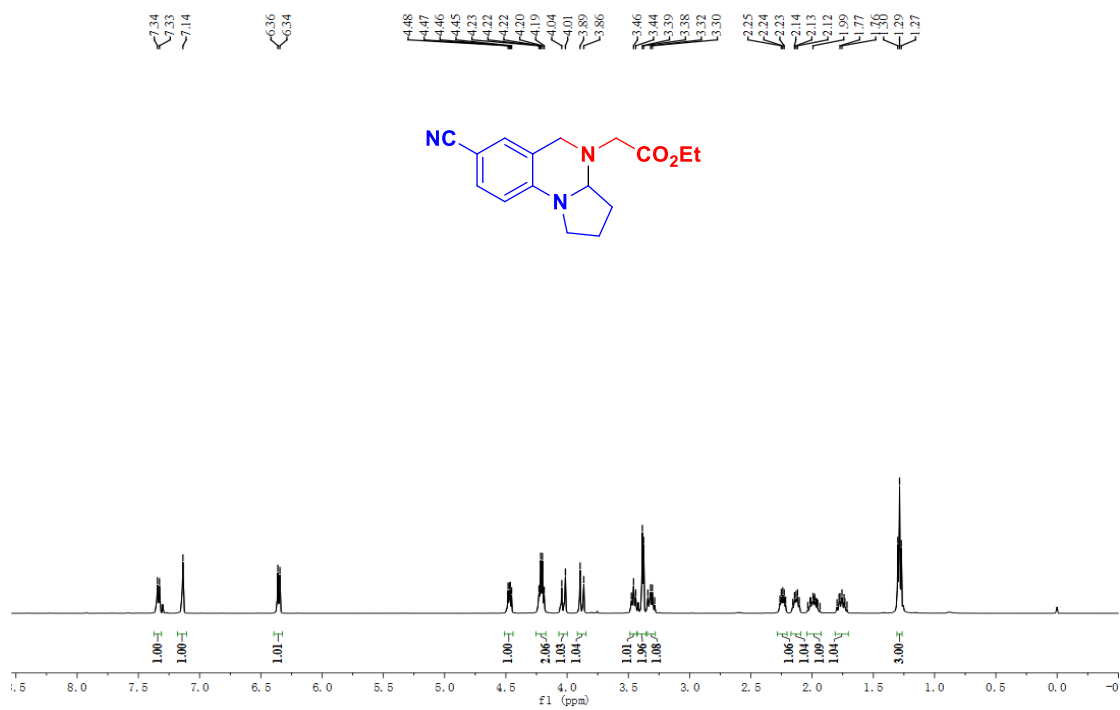
ethyl 2-(8-(trifluoromethyl)-1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)acetate (7e)



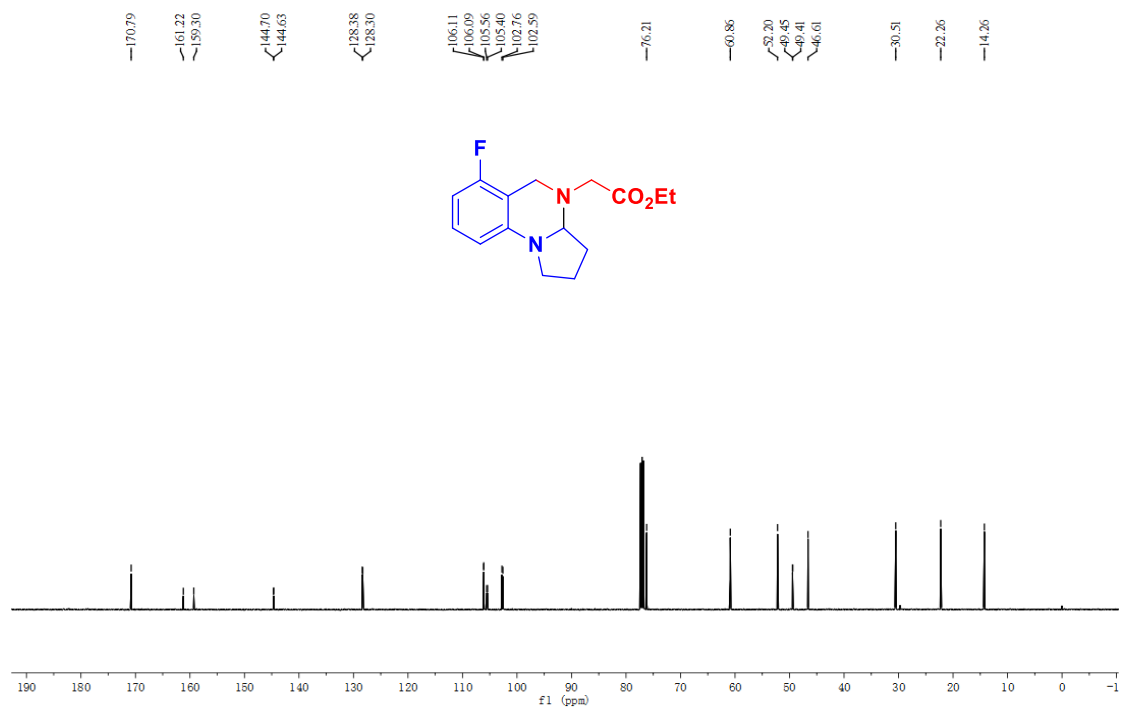
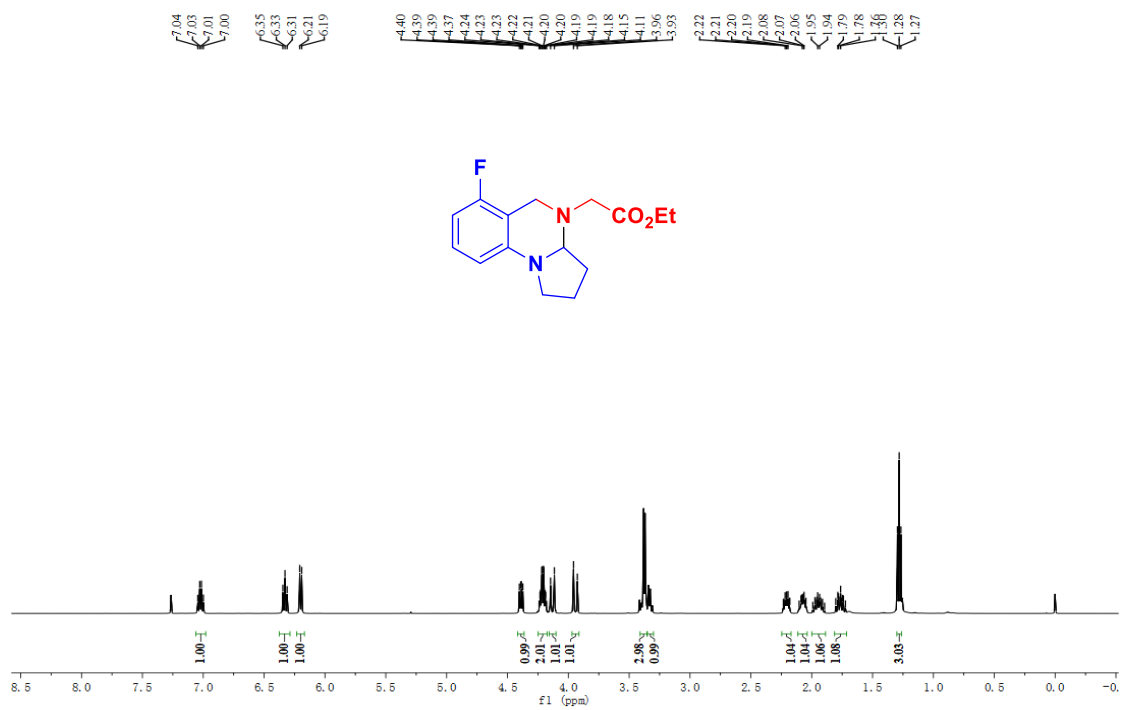
ethyl 2-(8-methyl-1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)acetate (7f)



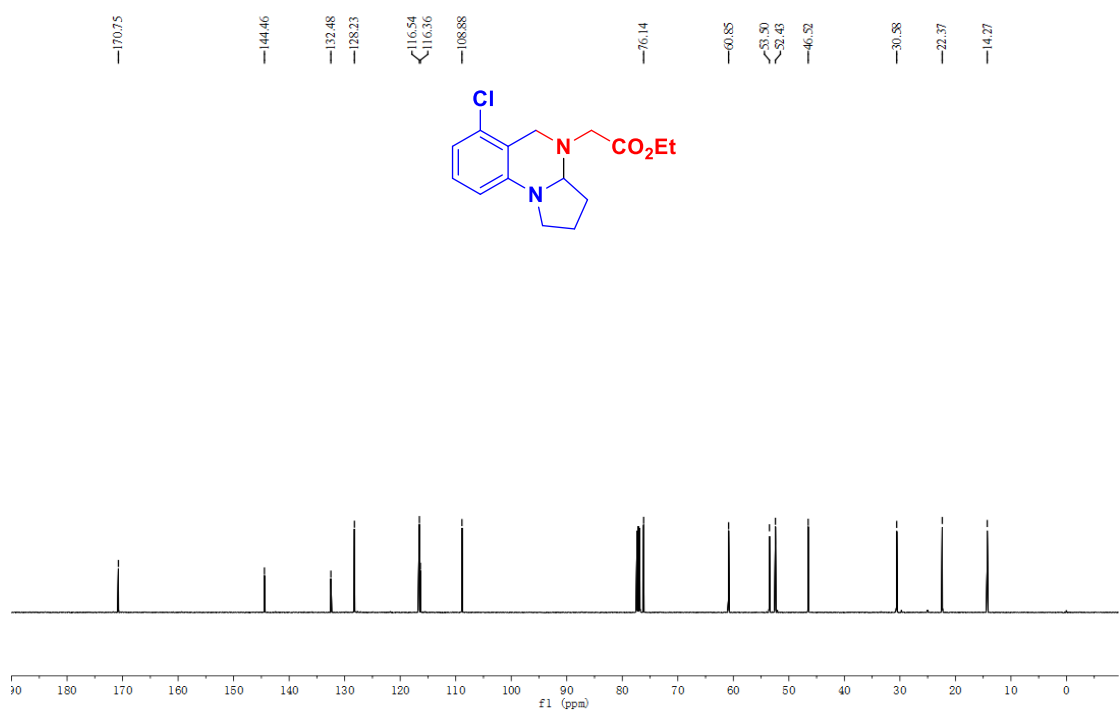
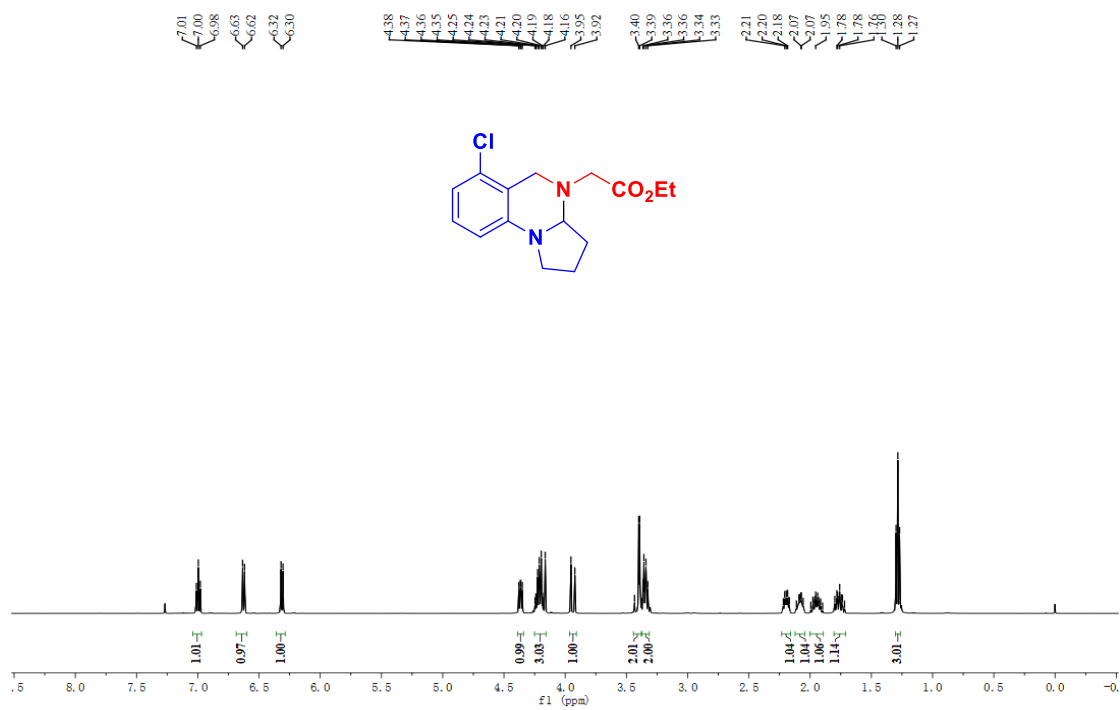
ethyl 2-(7-cyano-1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)acetate (7g)



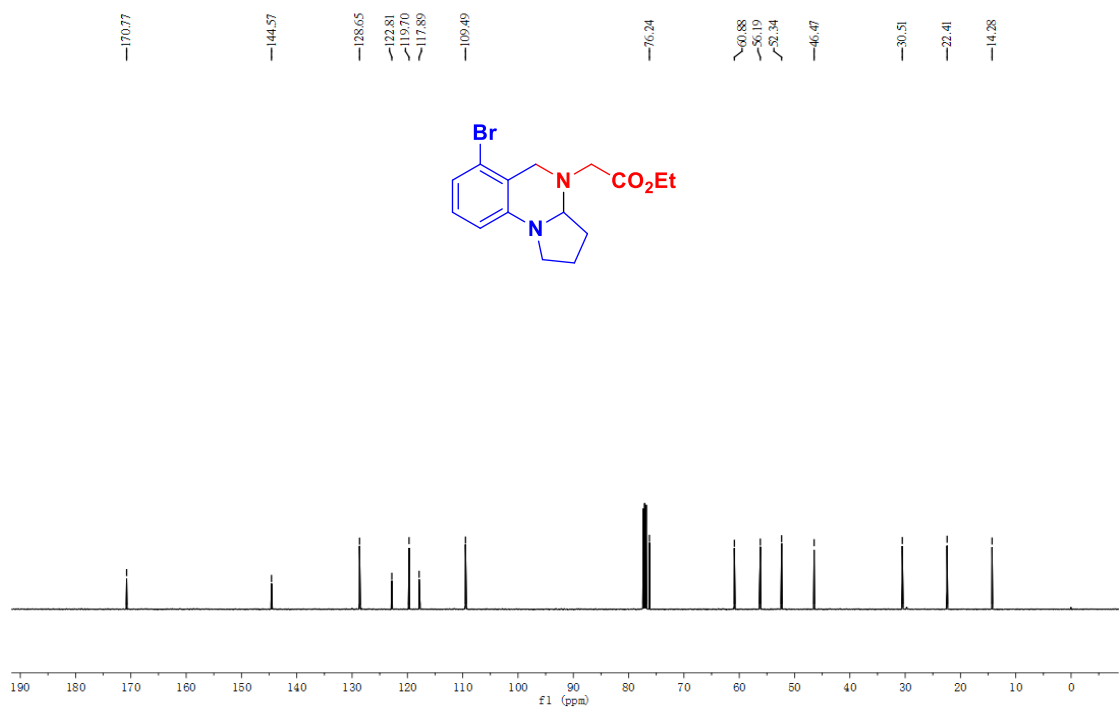
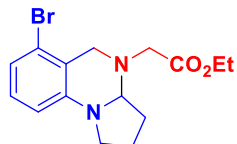
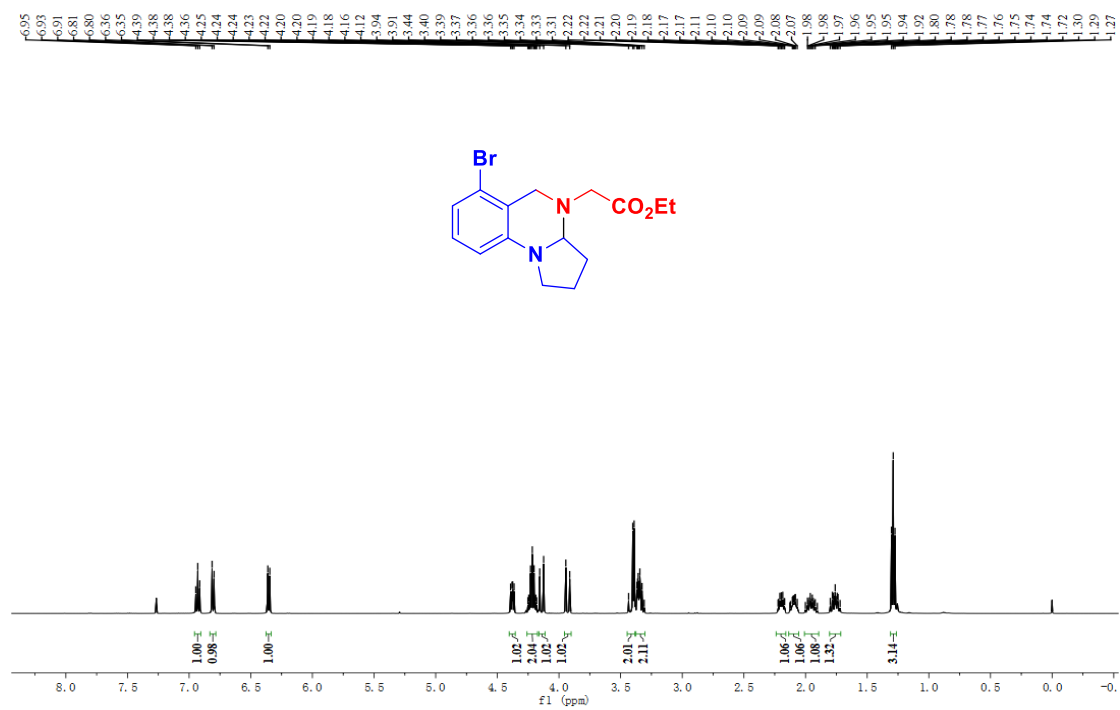
ethyl 2-(6-fluoro-1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)acetate (7i)



ethyl 2-(6-chloro-1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)acetate (7j)

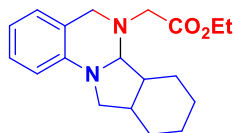


ethyl 2-(6-bromo-1,2,3,3a-tetrahydropyrrolo[1,2-a]quinazolin-4(5H)-yl)acetate (7k)

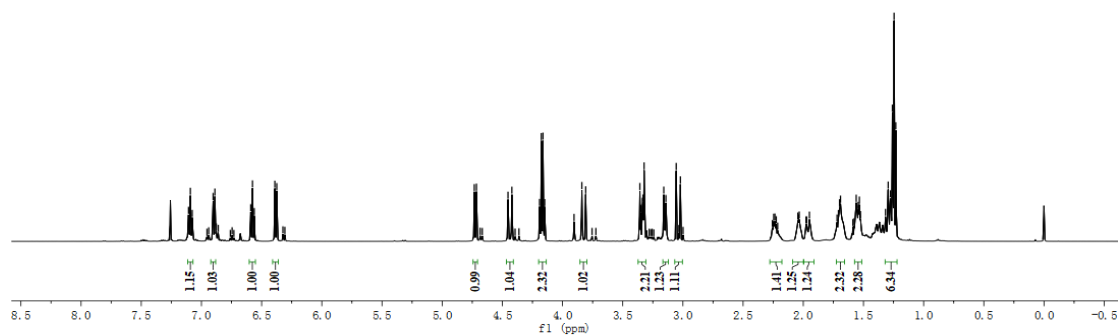


ethyl 6-amino-5,6,6a,6b,7,8,9,10,10a,11-decahydroisoindolo[2,1-a]quinoline-6-carboxylate (7l)

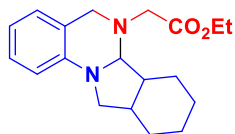
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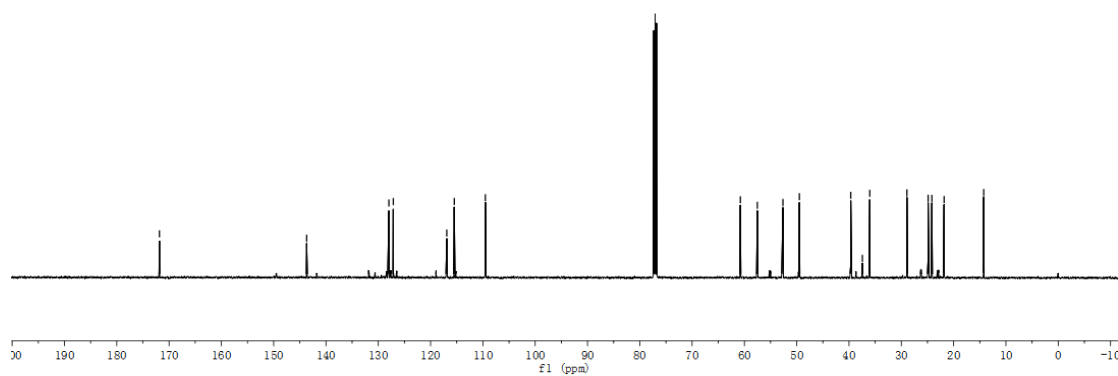
dr 7:1



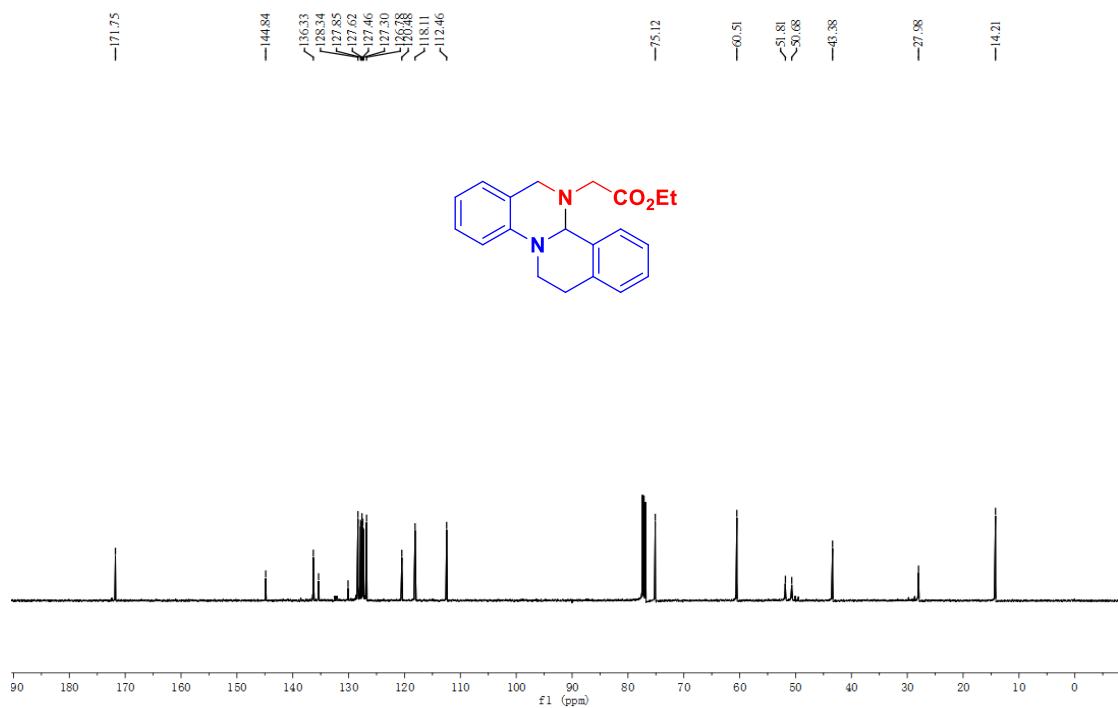
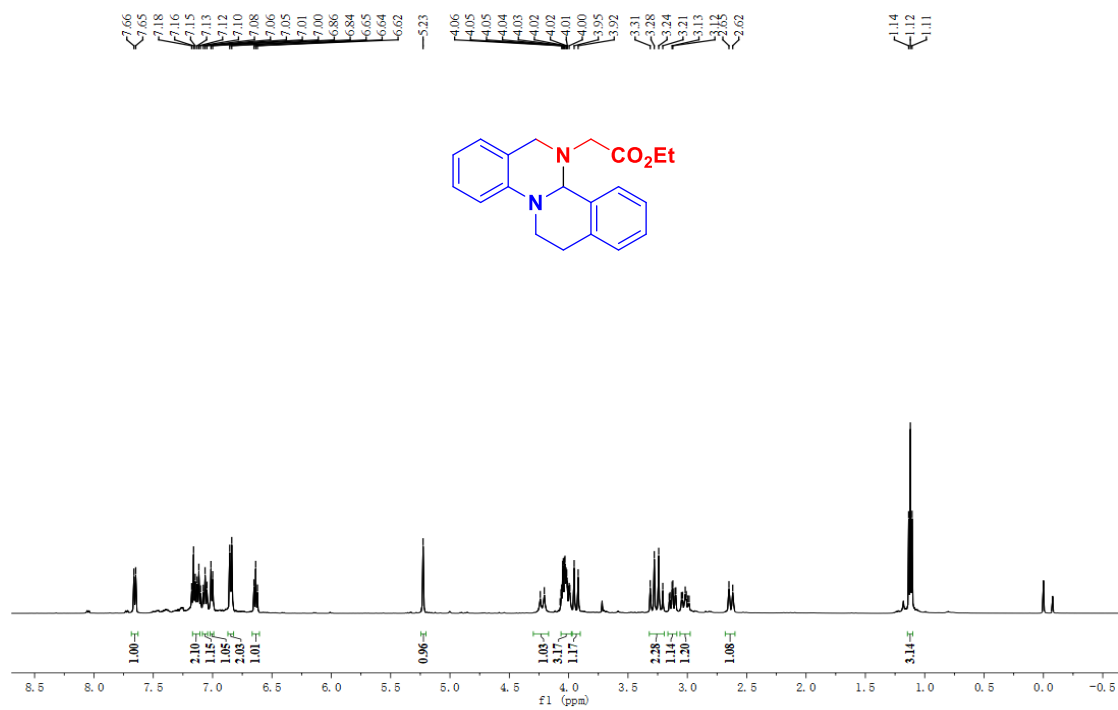
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21.82
14.25



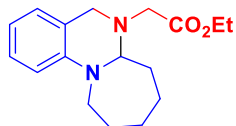
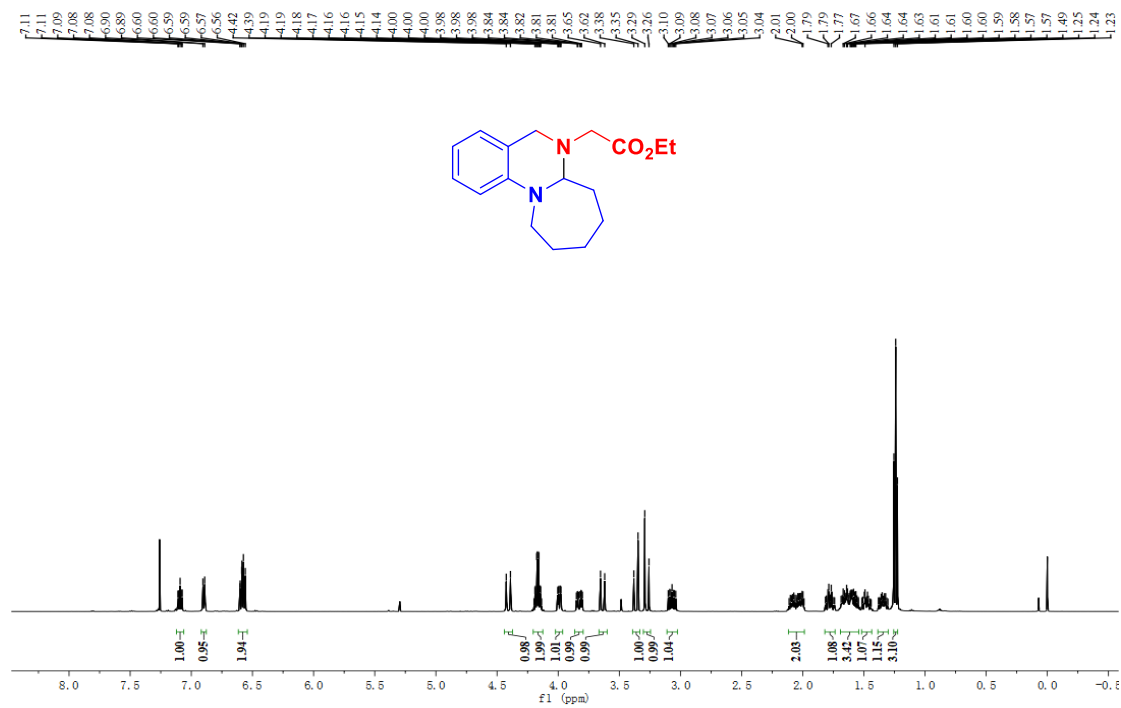
dr 7:1



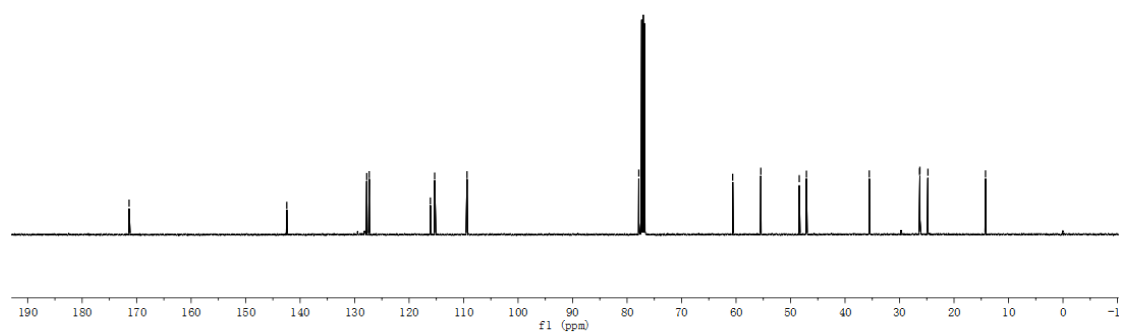
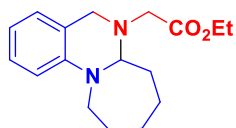
ethyl 2-(7,11b,12,13-tetrahydro-6H-isoquinolino[2,1-a]quinolin-12-yl)acetate (7m)



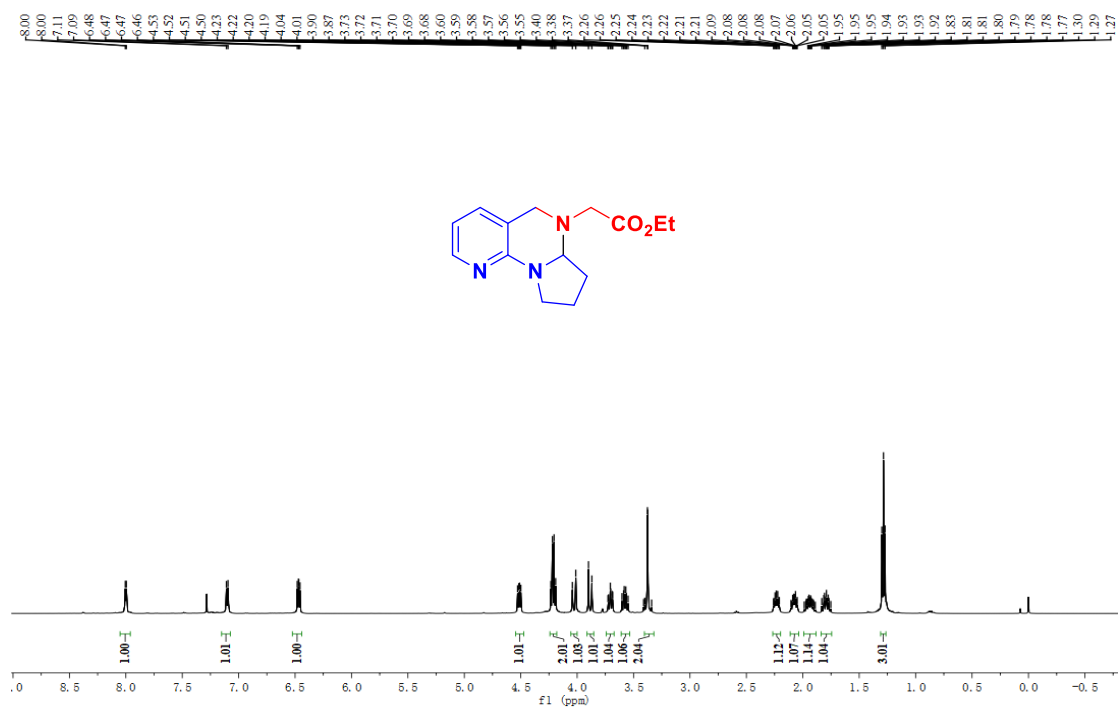
ethyl 2-(6a,7,8,9,10,11-hexahydroazepino[1,2-a]quinazolin-6(5H)-yl)acetate (7n)



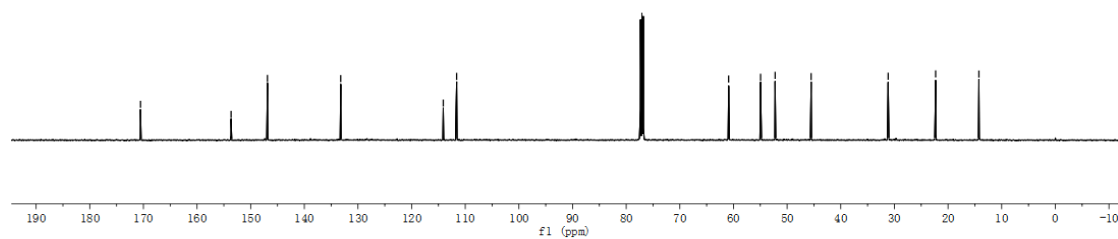
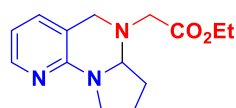
Chemical shifts (ppm): 171.39, 142.45, 127.77, 127.32, 116.10, 115.31, 109.37, 77.85, 60.62, 55.45, 48.40, 47.08, 35.56, 26.31, 26.26, 24.79, 14.22.



ethyl 6-amino-5,6,6a,7,8,9-hexahydropyrrolo[1,2-a][1,8]naphthyridine-6-carboxylate (7o)



Chemical shifts (ppm): 170.53, 153.65, 146.88, 133.22, 114.10, 111.61, 76.94, 60.50, 54.94, 52.23, 45.53, 31.16, 22.31, 14.24.



3. Determination of the Stereochemistry

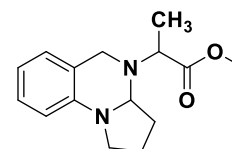
Chromaster 系统管理器报告

分析时间与日期: 2018/11/16 00:56:37
 处理日期与时间: 2019/08/21 20:56:57
 数据路径: E:\李帅帅\4\DATA\0063\
 数据处理用方法文件: 200-300氙灯模式-60/40

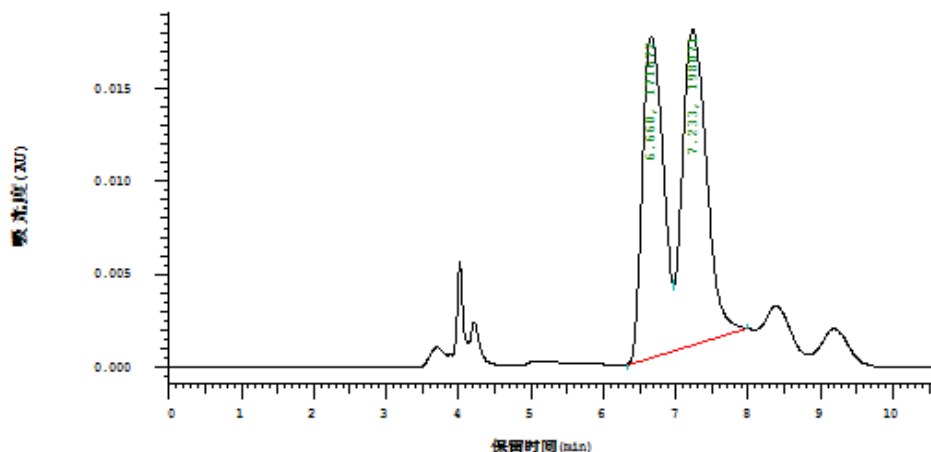
报告日期与时间: 2019/08/21 21:17:46

系统 (数据采集): Chromaster
 应用程序 (数据): 4-羟基吗啉
 样品名: CCQ-7088
 进样次数: 1 of 1
 样品注释:

系列: 0063
 样品瓶编号: 1
 样品瓶类型: UNK
 进样量: 10.0 ul



色谱类型: 固定波长色谱, 254 nm



数据处理用方法文件: 200-300氙灯模式-60/40

方法文件的创建者:

泵1: 5110+活塞清洗

泵1溶剂A: 正己烷

泵1溶剂C:

方法文件的注释:

泵1溶剂B: 异丙醇

泵1溶剂D:

色谱类型: 固定波长色谱, 254 nm

峰的定量: 面积
 计算方法: 面积%

No.	RT	面积	面积%
1	6.660	171672	46.430
2	7.233	198071	53.570
		369743	100.000

判定峰的基准: 0

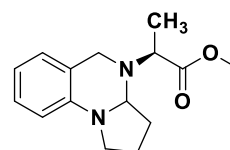
Chromaster 系统管理器报告

分析时间与日期:2018/11/16 00:43:13
 处理日期与时间:2019/08/21 21:00:26
 数据路径: E:\李帅帅\4\DATA\0062\
 数据处理用方法文件:200-300氙灯模式-60/40

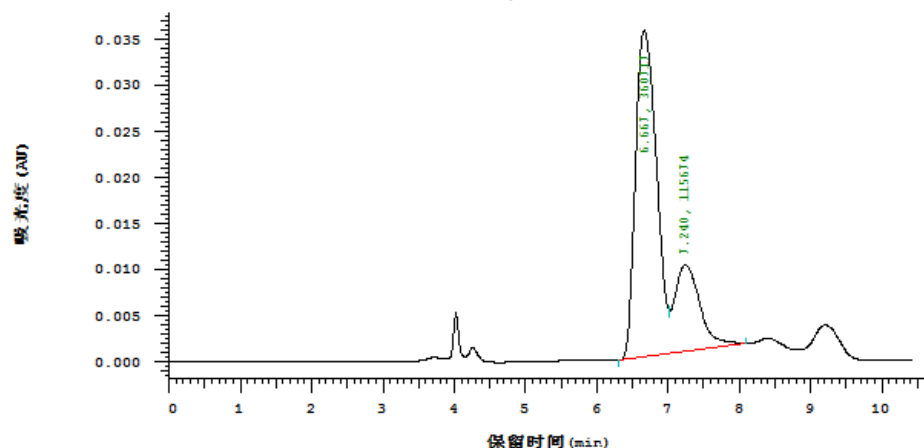
报告日期与时间:2019/08/21 21:24:18

系统(数据采集): Chromaster
 应用程序(数据): 4-羟基吡啶
 样品名: CCQ-7088
 进样次数: 1 of 1
 样品注释:

系列: 0062
 样品瓶编号: 1
 样品瓶类型: UNK
 进样量: 10.0 ul



色谱类型: 固定波长色谱, 254 nm



数据处理用方法文件:200-300氙灯模式-60/40
 方法文件的创建者:
 泵1: 5110+活塞清洗
 泵1溶剂A: 正己烷
 泵1溶剂C:
 方法文件的注释:

泵1溶剂B: 异丙醇
 泵1溶剂D:

色谱类型: 固定波长色谱, 254 nm

峰的定量: 面积
 计算方法: 面积%

No.	RT	面积	面积%
1	6.667	360717	75.719
2	7.240	115674	24.281
		476391	100.000

判定峰的基准: 0