Supporting Information

for

Recyclable heterogeneous nanocrystal promoted cascade reaction in water: An access to green synthesis of highly functionalized 4H-pyrans containing phosphonate motif

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[#] This author contributed equally to this work

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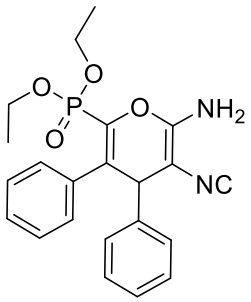
1. General procedure for the synthesis of 2-amino-3-cyano-4H-pyrans 3.

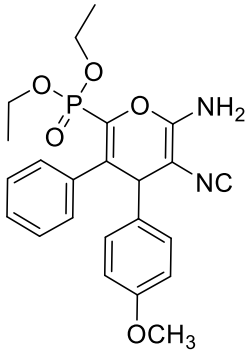
A solution of ZCS-NH_2 (80 mg), diethyl (2-phenylacetyl) phosphonates **1** (0.225 mmol) and 2-benzylidenemalononitriles **2** (0.15 mmol) in H_2O (1.5 mL) was stirred at 40 ^oC for the time indicated in Table 2. After the 2-benzylidenemalononitriles was consumed as indicated (monitored by TLC), the reaction solution was filtered, concentrated in vacuo. The crude product was purified by column chromatography on silica gel (eluent PE:EtOAc =10:1) to afford pure products 2-amino-3-cyano-4H-pyrans **3**.

Diethyl (6-amino-5-isocyano-3,4-diphenyl-4H-pyran-2-yl)phosphonate (3a)



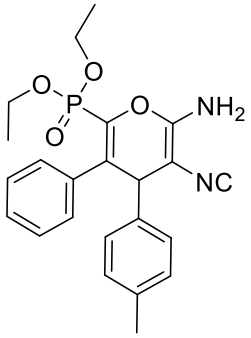
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.28 – 7.17 (m, 6H), 7.07 (d, *J* = 6.6 Hz, 2H), 6.93 (d, *J* = 6.6 Hz, 2H), 4.88 (s, 2H), 4.16 (s, 1H), 4.01 – 3.90 (m, 2H), 3.87 – 3.72 (m, 2H), 1.12 (q, *J* = 7.0, 4.3 Hz, 6H) ppm.

Diethyl (6-amino-5-isocyano-4-(4-methoxyphenyl)-3-phenyl-4H-pyran-2-yl)phosphonate (3b)



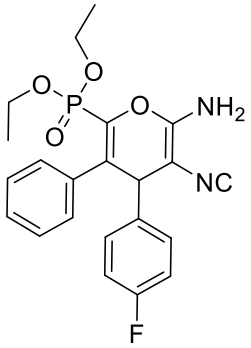
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.21 (d, *J* = 7.0 Hz, 3H), 7.03 – 6.88 (m, 4H), 6.79 (d, *J* = 8.3 Hz, 2H), 4.67 (s, 2H), 4.11 (s, 1H), 4.03 – 3.89 (m, 2H), 3.87 – 3.69 (m, 5H), 1.12 (t, *J* = 6.9 Hz, 6H).

Diethyl (6-amino-5-isocyano-3-phenyl-4-(p-tolyl)-4H-pyran-2-yl)phosphonate (3c)



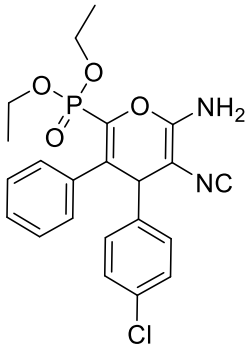
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.23 – 7.14 (m, 3H), 7.04 (d, *J* = 8.1 Hz, 2H), 6.97 – 6.91 (m, 4H), 5.09 – 4.87 (m, 2H), 4.10 (s, 1H), 3.98 – 3.89 (m, 2H), 3.83 – 3.74 (m, 2H), 2.28 (s, 3H), 1.16 – 1.05 (m, 3H) ppm.

Diethyl (6-amino-4-(4-fluorophenyl)-5-isocyano-3-phenyl-4H-pyran-2-yl)phosphonate (3d)



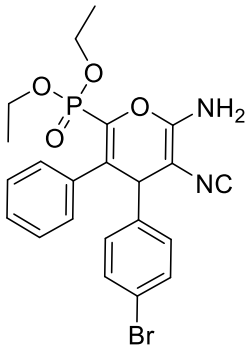
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.11 (m, 4H), 7.02 – 6.87 (m, 3H), 6.87 – 6.70 (m, 2H), 5.10 – 4.71 (m, 2H), 4.18 (s, 1H), 4.02 – 3.88 (m, 2H), 3.89 – 3.67 (m, 2H), 1.11 (q, *J* = 6.8 Hz, 6H) ppm.

Diethyl (6-amino-4-(4-chlorophenyl)-5-isocyano-3-phenyl-4H-pyran-2-yl)phosphonate (3e)



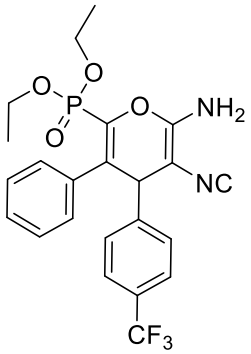
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 – 7.17 (m, 3H), 7.04 – 6.99 (m, 2H), 6.96 – 6.89 (m, 4H), 4.97 (s, 2H), 4.17 (s, 1H), 3.98 – 3.87 (m, 2H), 3.84 – 3.72 (m, 2H), 1.13 – 1.06 (m, 6H) ppm.

Diethyl (6-amino-4-(4-bromophenyl)-5-isocyano-3-phenyl-4H-pyran-2-yl)phosphonate (3f)



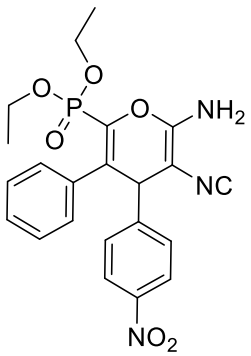
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 (d, *J* = 8.3 Hz, 2H), 7.25 – 7.14 (m, 3H), 6.92 (d, *J* = 8.2 Hz, 4H), 5.03 (s, 2H), 4.15 (s, 1H), 4.01 – 3.85 (m, 2H), 3.85 – 3.66 (m, 2H), 1.09 (q, *J* = 7.0, 4.1 Hz, 6H) ppm.

Diethyl(6-amino-5-isocyano-3-phenyl-4-(4-(trifluoromethyl)phenyl)-4H-pyran-2-yl)phosphonate (3g)



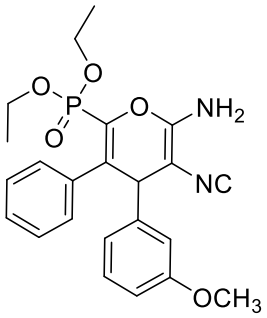
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.50 (d, *J* = 8.0 Hz, 2H), 7.30 – 7.05 (m, 5H), 6.93 (d, *J* = 6.7 Hz, 5H), 5.06 (s, 2H), 4.27 (s, 1H), 4.02 – 3.85 (m, 2H), 3.85 – 3.61 (m, 2H), 1.10 (q, *J* = 7.0, 3.3 Hz, 6H) ppm.

Diethyl (6-amino-5-isocyano-4-(4-nitrophenyl)-3-phenyl-4H-pyran-2-yl)phosphonate (3h)



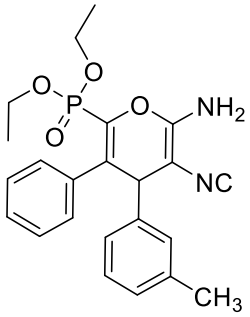
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.11 (d, *J* = 8.7 Hz, 2H), 7.27 – 7.14 (m, 5H), 6.95 (d, *J* = 6.5 Hz, 2H), 5.21 (s, 2H), 4.38 (s, 1H), 4.05 – 3.89 (m, 2H), 3.88 – 3.71 (m, 2H), 1.11 (t, *J* = 7.0 Hz, 6H) ppm.

Diethyl (6-amino-5-isocyano-4-(3-methoxyphenyl)-3-phenyl-4H-pyran-2-yl)phosphonate (3i)



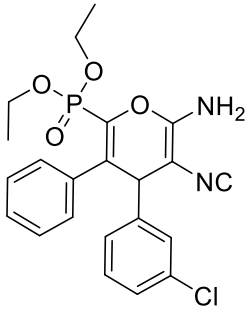
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.29 – 7.12 (m, 5H), 7.07 (s, 1H), 6.94 (d, *J* = 5.4 Hz, 3H), 4.99 (s, 2H), 4.17 (s, 1H), 4.02 – 3.72 (m, 4H), 1.12 (q, *J* = 9.7, 7.1 Hz, 1H) ppm.

Diethyl (6-amino-5-isocyano-3-phenyl-4-(m-tolyl)-4H-pyran-2-yl) phosphonate (3j)



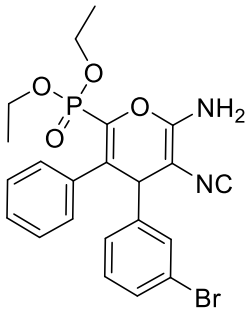
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 – 7.10 (m, 4H), 7.02 (d, *J* = 7.5 Hz, 1H), 6.95 – 6.90 (m, 2H), 6.85 (d, *J* = 5.7 Hz, 2H), 4.80 (s, 2H), 4.10 (s, 1H), 4.02 – 3.89 (m, 2H), 3.87 – 3.74 (m, 2H), 2.27 (s, 3H), 1.11 (q, *J* = 7.0 Hz, 6H) ppm.

Diethyl (6-amino-4-(3-chlorophenyl)-5-isocyano-3-phenyl-4H-pyran-2-yl)phosphonate (3k)



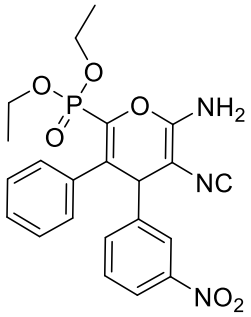
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.05 – 7.98 (m, 1H), 7.86 (t, *J* = 2.0 Hz, 1H), 7.36 (t, *J* = 7.8 Hz, 1H), 7.33 – 7.27 (m, 1H), 7.23 – 7.11 (m, 3H), 6.91 – 6.83 (m, 2H), 5.07 (s, 2H), 4.33 (s, 1H), 3.96 – 3.85 (m, 2H), 3.84 – 3.66 (m, 2H), 1.06 (q, *J* = 17.1, 7.1 Hz, 6H) ppm.

Diethyl (6-amino-4-(3-bromophenyl)-5-isocyano-3-phenyl-4H-pyran-2-yl)phosphonate (3l)



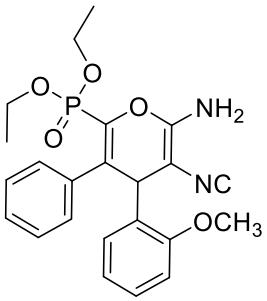
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.35 (d, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 7.2 Hz, 4H), 7.12 (t, *J* = 7.8 Hz, 1H), 6.95 (t, *J* = 8.2 Hz, 3H), 5.03 (s, 2H), 4.16 (s, 1H), 4.00 – 3.89 (m, 2H), 3.88 – 3.73 (m, 2H), 1.12 (q, *J* = 7.2 Hz, 6H) ppm.

Diethyl (6-amino-5-isocyano-4-(3-nitrophenyl)-3-phenyl-4H-pyran-2-yl)phosphonate (3m)



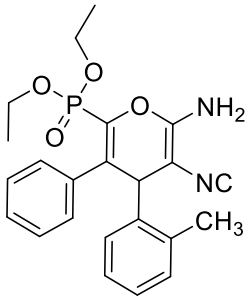
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.10 – 8.04 (m, 1H), 7.97 – 7.85 (m, 1H), 7.45 – 7.34 (m, 2H), 7.21 (q, *J* = 6.8, 6.1 Hz, 3H), 7.00 – 6.87 (m, 2H), 5.05 (s, 2H), 4.38 (s, 1H), 4.03 – 3.90 (m, 2H), 3.89 – 3.74 (m, 2H), 1.18 – 1.05 (m, 6H) ppm.

Diethyl (6-amino-5-isocyano-4-(2-methoxyphenyl)-3-phenyl-4H-pyran-2-yl)phosphonate (3n)



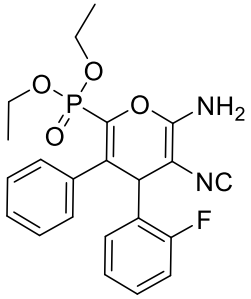
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.20 – 7.11 (m, 5H), 7.01 – 6.94 (m, 2H), 6.89 (t, *J* = 7.4 Hz, 1H), 6.74 (d, *J* = 8.1 Hz, 1H), 4.77 (s, 2H), 4.71 (s, 1H), 4.00 – 3.90 (m, 2H), 3.86 – 3.73 (m, 2H), 3.57 (s, 3H), 1.11 (q, *J* = 7.3 Hz, 6H) ppm.

Diethyl (6-amino-5-isocyano-3-phenyl-4-(o-tolyl)-4H-pyran-2-yl)phosphonate (3o)



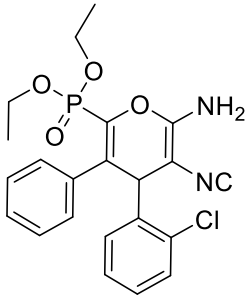
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.27 – 7.14 (m, 5H), 7.10 (t, *J* = 7.3 Hz, 1H), 6.98 (d, *J* = 7.5 Hz, 1H), 6.88 (d, *J* = 7.0 Hz, 2H), 4.76 (s, 2H), 4.52 (s, 1H), 4.04 – 3.89 (m, 2H), 3.87 – 3.72 (m, 2H), 1.96 (s, 3H), 1.18 – 1.08 (m, 6H) ppm.

Diethyl (6-amino-4-(2-fluorophenyl)-5-isocyano-3-phenyl-4H-pyran-2-yl)phosphonate (3p)



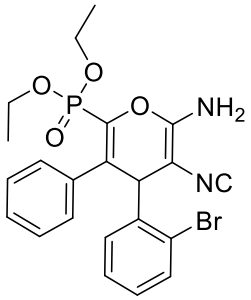
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.25 – 7.12 (m, 5H), 7.06 (t, *J* = 7.4 Hz, 1H), 7.01 – 6.83 (m, 3H), 4.88 (s, 2H), 4.56 (s, 1H), 4.03 – 3.89 (m, 2H), 3.87 – 3.72 (m, 2H), 1.12 (q, *J* = 6.9, 3.5 Hz, 6H) ppm.

Diethyl (6-amino-4-(2-chlorophenyl)-5-isocyano-3-phenyl-4H-pyran-2-yl)phosphonate (3q)



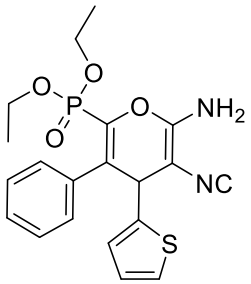
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.26 – 7.10 (m, 7H), 6.95 (d, *J* = 6.6 Hz, 2H), 4.89 (s, 1H), 4.86 (s, 2H), 4.00 – 3.88 (m, 2H), 3.85 – 3.74 (m, 2H), 1.12 (q, *J* = 6.9 Hz, 6H) ppm.

Diethyl (6-amino-4-(2-bromophenyl)-5-isocyano-3-phenyl-4H-pyran-2-yl)phosphonate (3r)



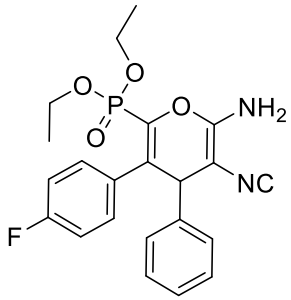
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40 (d, *J* = 8.0 Hz, 1H), 7.32 – 7.26 (m, 2H), 7.24 – 7.15 (m, 3H), 7.08 – 7.02 (m, 1H), 6.96 (d, *J* = 6.7 Hz, 2H), 4.96 (s, 1H), 4.80 (s, 2H), 4.04 – 3.89 (m, 2H), 3.87 – 3.74 (m, 2H), 1.17 – 1.07 (m, 6H) ppm.

Diethyl (6-amino-5-isocyano-3-phenyl-4-(thiophen-2-yl)-4H-pyran-2-yl) phosphonate (3t)



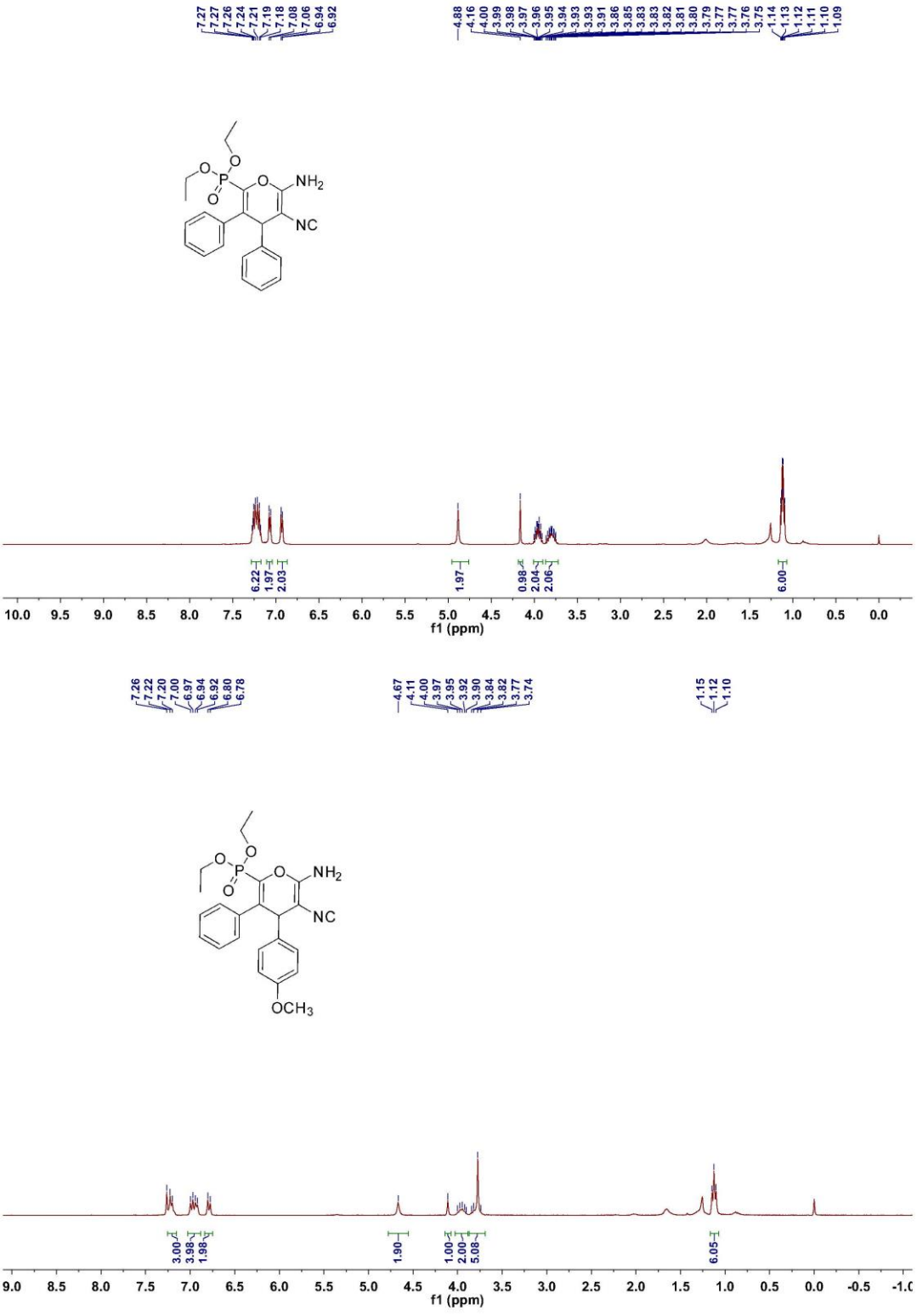
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 – 7.23 (m, 3H), 7.20 (d, *J* = 5.1 Hz, 1H), 7.08 – 7.00 (m, 2H), 6.87 – 6.80 (m, 1H), 6.66 (d, *J* = 2.9 Hz, 1H), 5.01 (s, 2H), 4.49 (s, 1H), 4.02 – 3.84 (m, 3H), 3.80 – 3.71 (m, 1H), 1.16 (t, *J* = 7.1 Hz, 3H), 1.09 (t, *J* = 7.1 Hz, 3H) ppm.

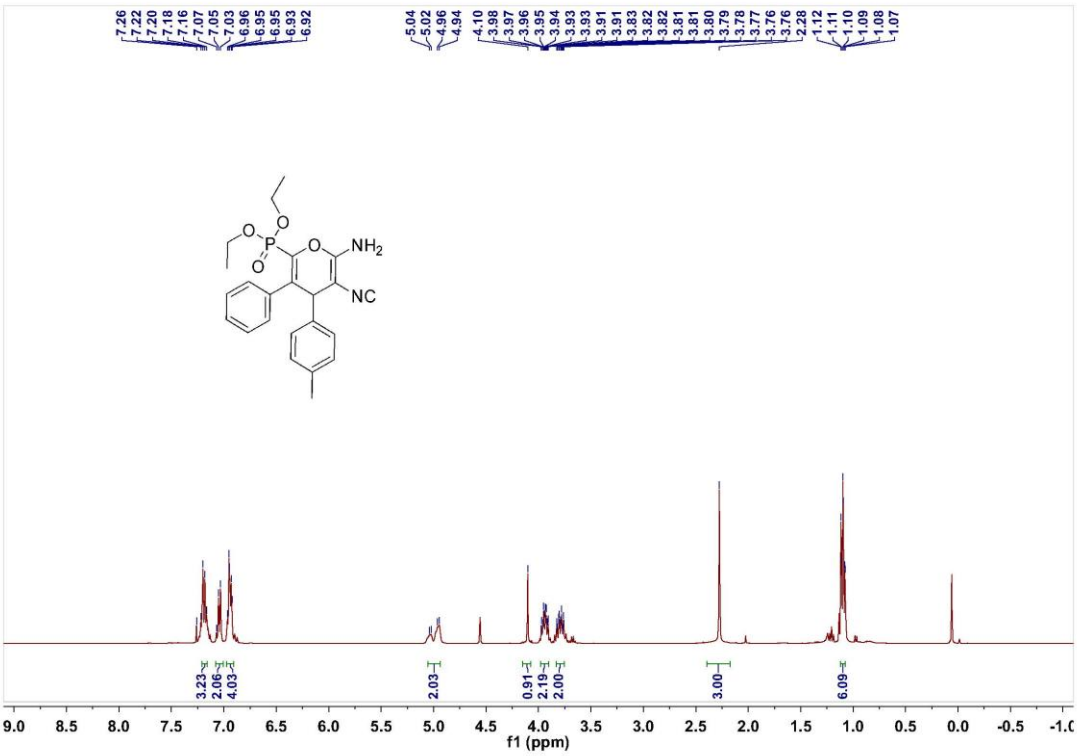
Diethyl (6-amino-3-(4-fluorophenyl)-5-isocyano-4-phenyl-4H-pyran-2-yl)phosphonate (**3v**)



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.21 – 7.15 (m, 3H), 7.01 – 6.96 (m, 2H), 6.82 (d, *J* = 6.9 Hz, 4H), 5.00 – 4.72 (m, 2H), 4.05 (s, 1H), 3.95 – 3.86 (m, 2H), 3.83 – 3.72 (m, 2H), 1.07 (q, *J* = 7.1, 3.1 Hz, 6H) ppm.

2. NMR spectra for compounds 3




7.26
7.22
7.20
7.18
7.16
7.07
7.05
7.03
6.96
6.95
6.95
6.93
6.92
5.04
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3.81
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3.80
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3.77
3.76
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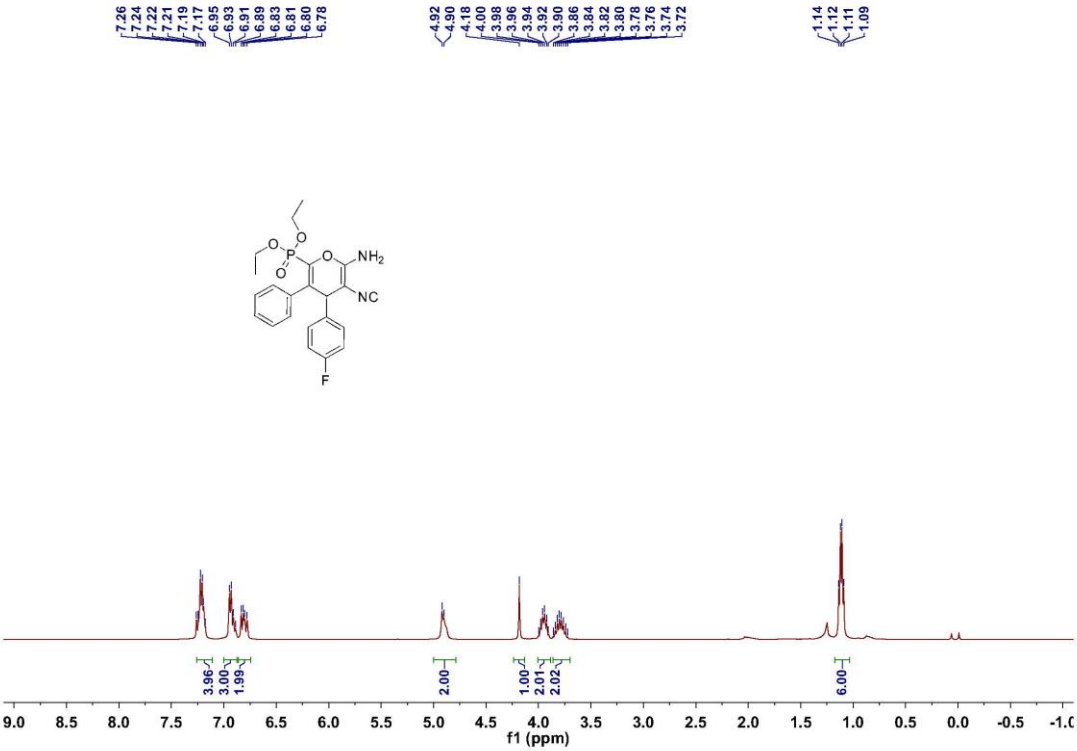
2.28
1.12
1.11
1.10
1.09
1.08
1.07
NH2
NC
3.23
2.06
4.03
2.03
0.91
2.19
2.00
3.00
6.09
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


7.26
7.24
7.22
7.21
7.19
7.17
6.95
6.93
6.91
6.89
6.83
6.81
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3.74
3.72
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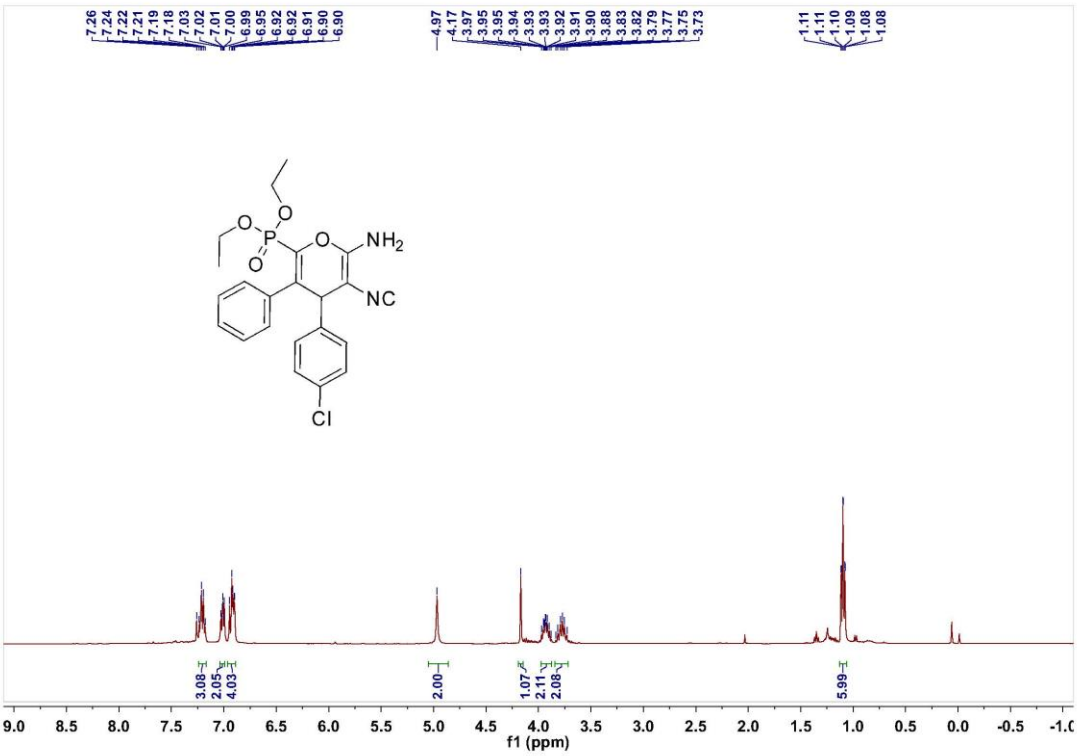
1.11
1.09
NH2
NC
F
3.96
3.00
1.99
2.00
1.00
2.01
2.02
6.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


7.26
7.24
7.22
7.21
7.19
7.18
7.03
7.02
7.01
7.00
6.99
6.95
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3.83
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3.73
1.11
1.11
1.10
1.09
1.08
1.08
NH2
NC
Cl
3.08
2.05
4.03
2.00
1.07
2.11
2.08
5.99
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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2.5
2.0
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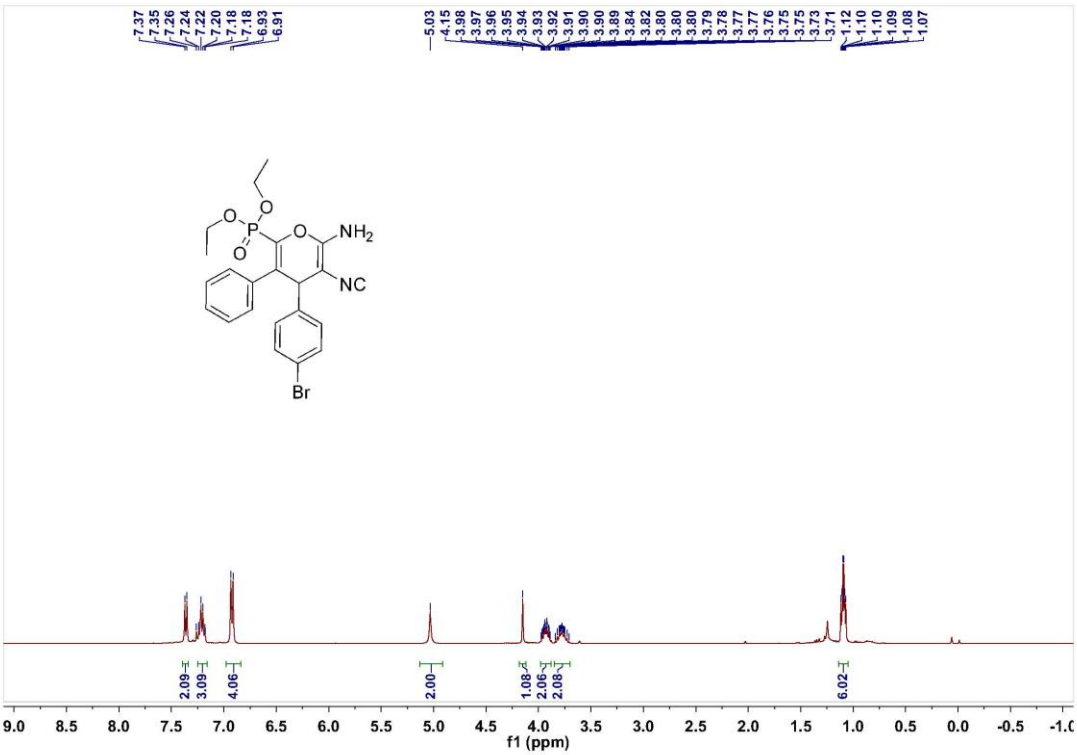
f1 (ppm)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7.35
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7.18
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6.91
5.03
4.15
3.98
3.97
3.96
3.95
3.94
3.93
3.92
3.91
3.90
3.90
3.89
3.84
3.82
3.80
3.80
3.80
3.79
3.78
3.77
3.77
3.76
3.75
3.75
3.73
3.71
1.12
1.10
1.10
1.09
1.08
1.07
NH2
NC
Br
2.09
3.09
4.06
2.00
1.08
2.06
2.08
6.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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2.5
2.0
1.5
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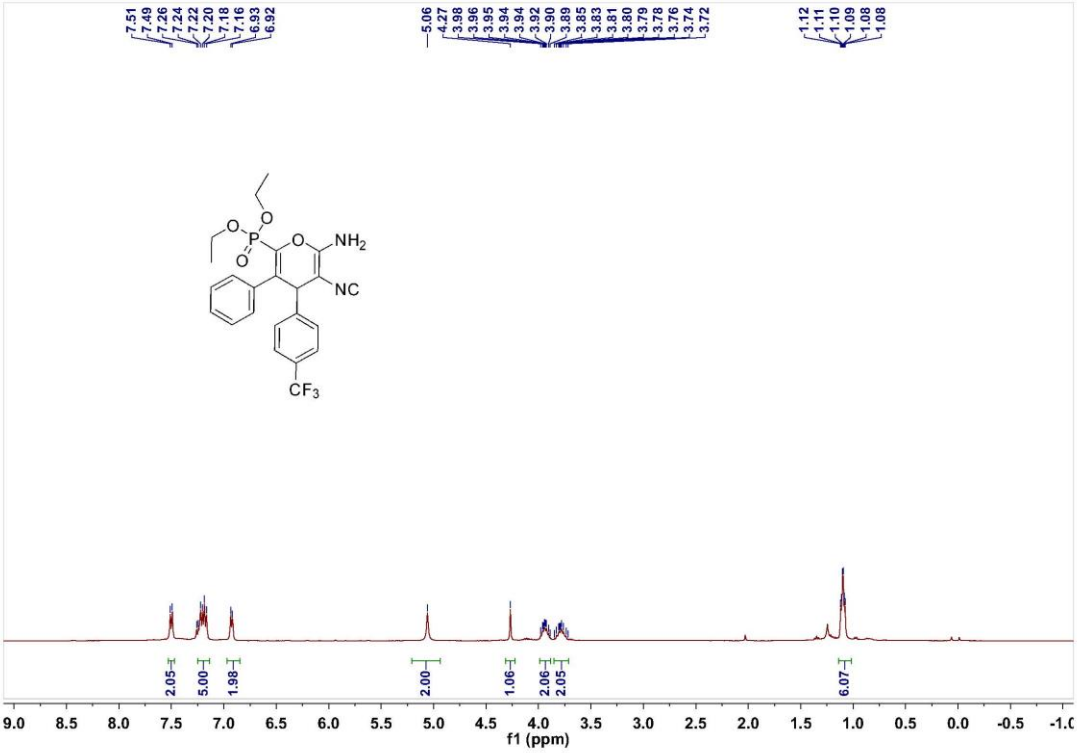
-0.5
f1 (ppm)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7.16
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6.92
5.06
4.27
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3.96
3.95
3.94
3.94
3.92
3.90
3.89
3.85
3.83
3.81
3.80
3.79
3.78
3.76
3.74
3.72
1.12
1.11
1.10
1.09
1.08
1.08
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CF3
2.05
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1.98
2.00
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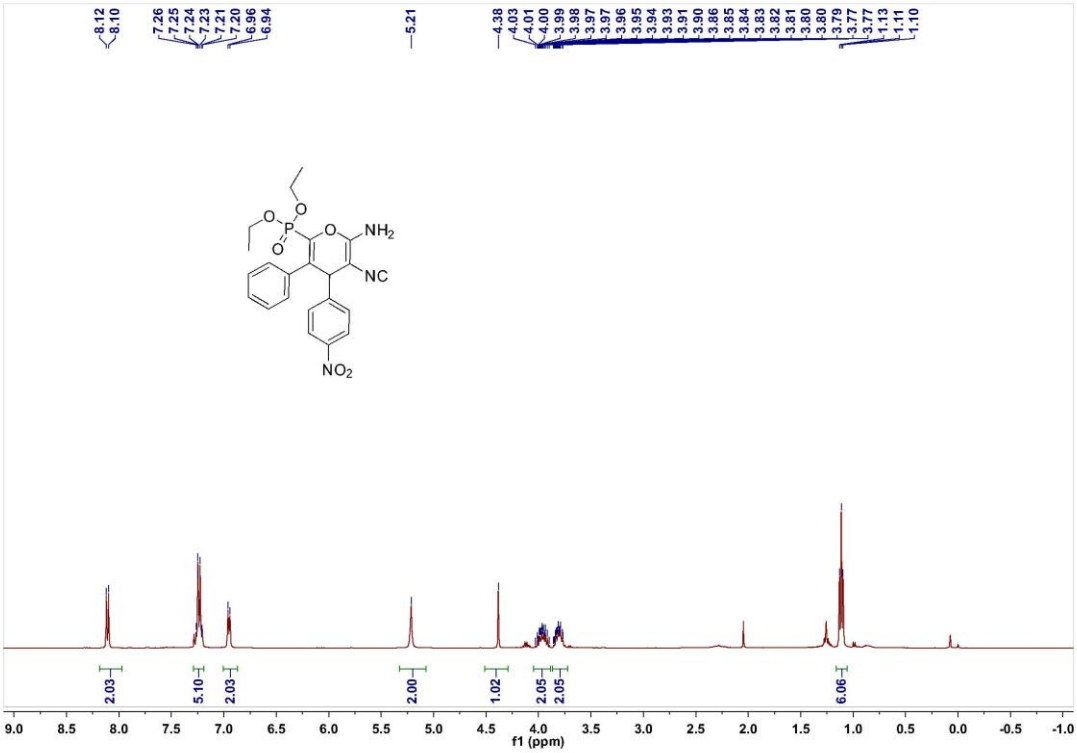
2.06
2.05
6.07
f1 (ppm)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8.10
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7.23
7.21
7.20
6.96
6.94
5.21
4.38
4.03
4.01
4.00
3.99
3.98
3.97
3.97
3.96
3.95
3.94
3.93
3.91
3.90
3.86
3.85
3.84
3.83
3.82
3.81
3.80
3.80
3.79
3.77
3.77
1.13
1.11
1.10
NH2
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NO2
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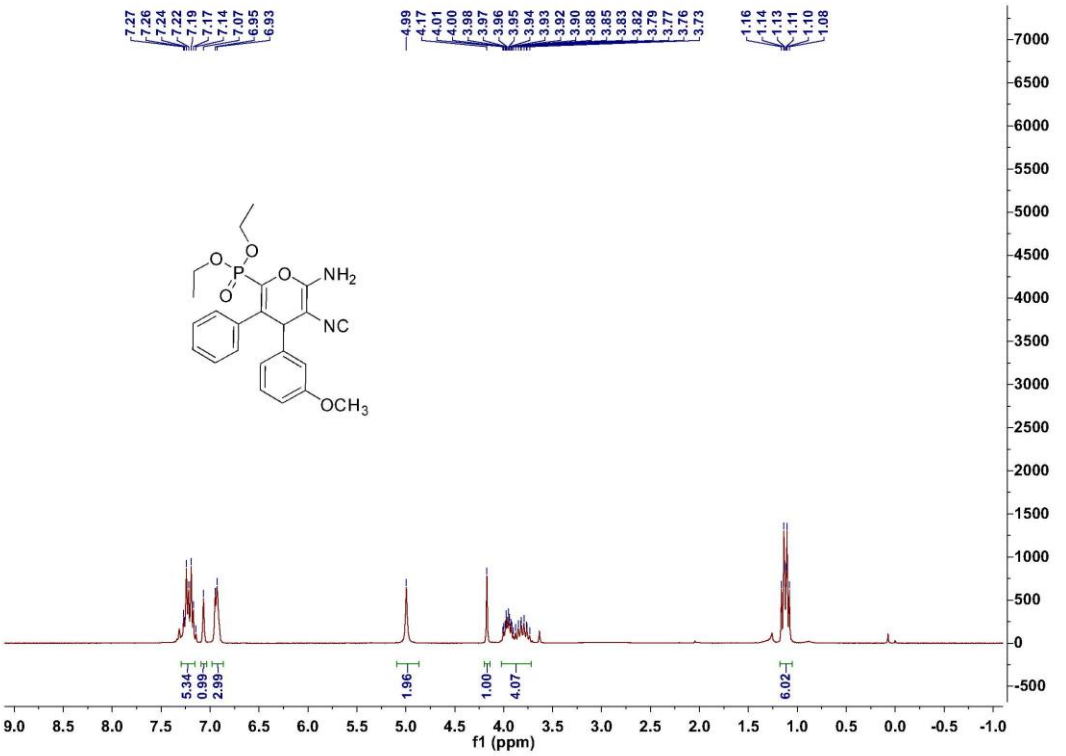
5.10
2.03
2.00
1.02
2.05
2.05
6.06
f1 (ppm)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7.17
7.14
7.07
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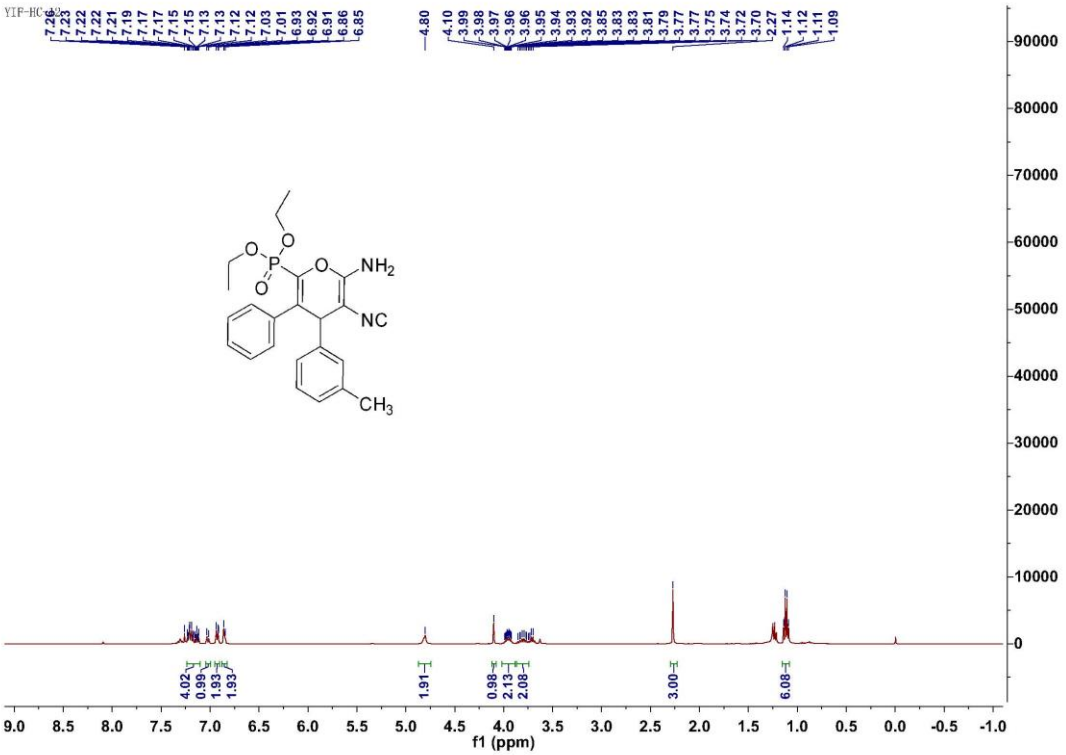
6.93
4.99
4.17
4.01
4.00
3.98
3.97
3.96
3.95
3.94
3.93
3.92
3.90
3.88
3.85
3.83
3.82
3.79
3.77
3.76
3.73
1.16
1.14
1.13
1.11
1.10
1.08
NH2
NC
OCH3
5.34
0.99
2.99
1.96
1.00
4.07
6.02
f1 (ppm)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7.15
7.13
7.13
7.12
7.12
7.03
7.01
6.93
6.92
6.91
6.86
6.85
4.80
4.10
3.99
3.98
3.97
3.96
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3.93
3.92
3.85
3.83
3.83
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3.77
3.75
3.74
3.72
3.70
2.27
1.14
1.12
1.11
1.09
NH2
NC
CH3
4.02
0.99
1.93
1.93
1.91
0.98
2.13
2.08
3.00
6.08
f1 (ppm)


7.37
7.34
7.27
7.26
7.25
7.22
7.14
7.12
7.09
6.98
6.95
6.92
5.03
4.16
4.00
3.97
3.97
3.94
3.92
3.90
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3.79
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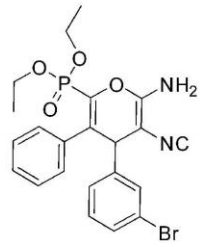
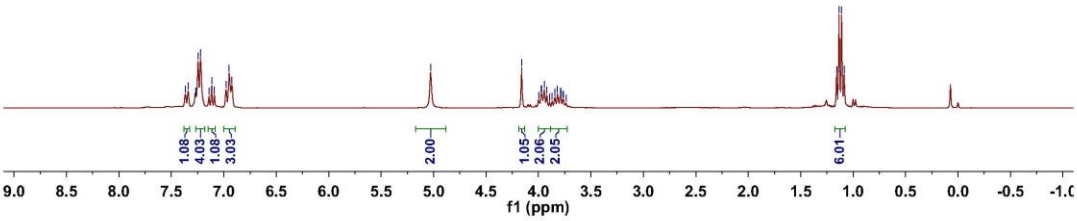
3.77
3.76
3.74
1.16
1.13
1.11
1.09


O
O
P
O
O
O
NH2
NC
Br


1.08
4.03
1.08
3.03
2.00
1.05
2.06
2.05
6.01
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
-0.5
f1 (ppm)


8.08
8.07
8.06
7.93
7.92
7.43
7.42
7.40
7.37
7.35
7.26
7.24
7.22
7.20
7.19
7.18
6.94
6.92
6.92
5.05
4.38
4.01
3.99
3.99
3.98
3.97
3.96
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3.92
3.89
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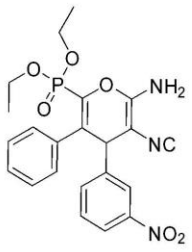
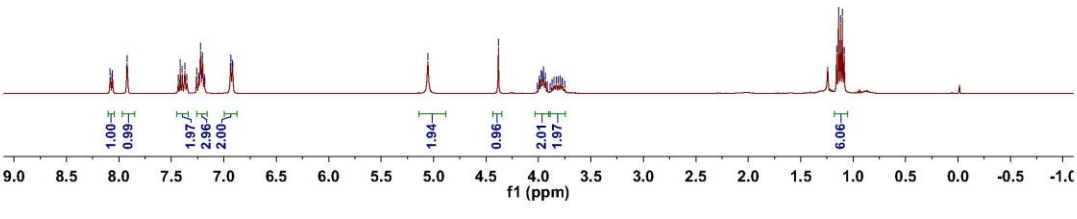
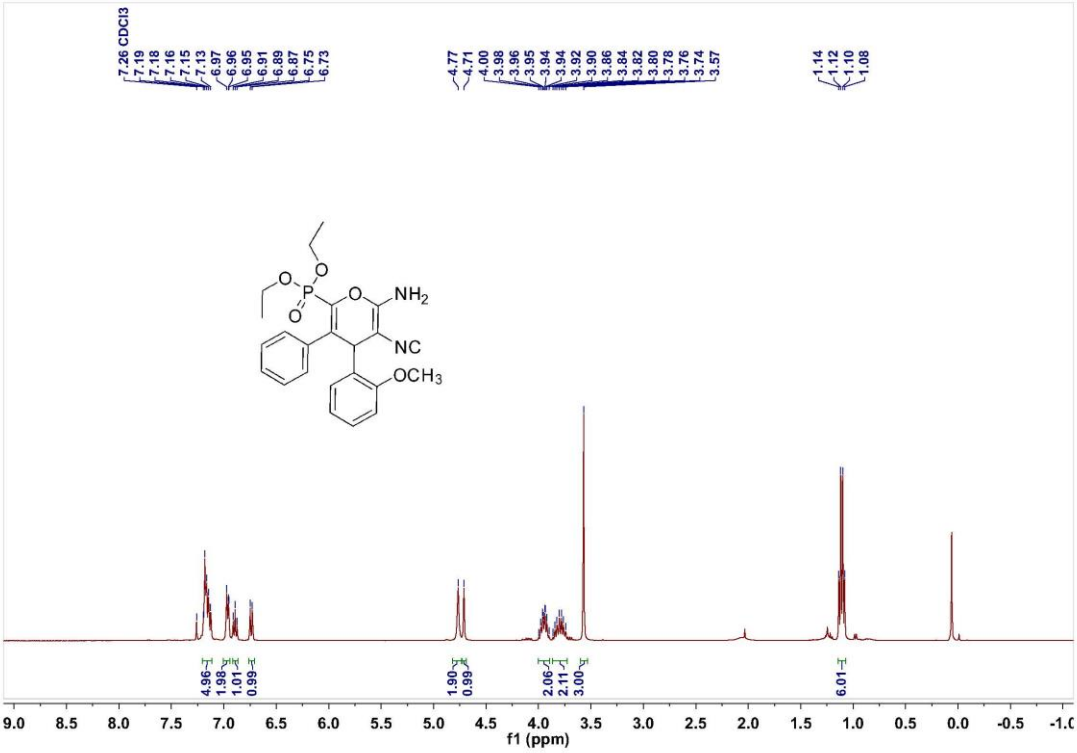
3.75
1.16
1.14
1.12
1.10
1.09


O
O
P
O
O
O
NH2
NC
NO2


1.00
0.99
1.97
2.96
2.00
1.94
0.96
2.01
1.97
6.06
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
-0.5
f1 (ppm)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7.18
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6.97
6.96
6.95
6.91
6.89
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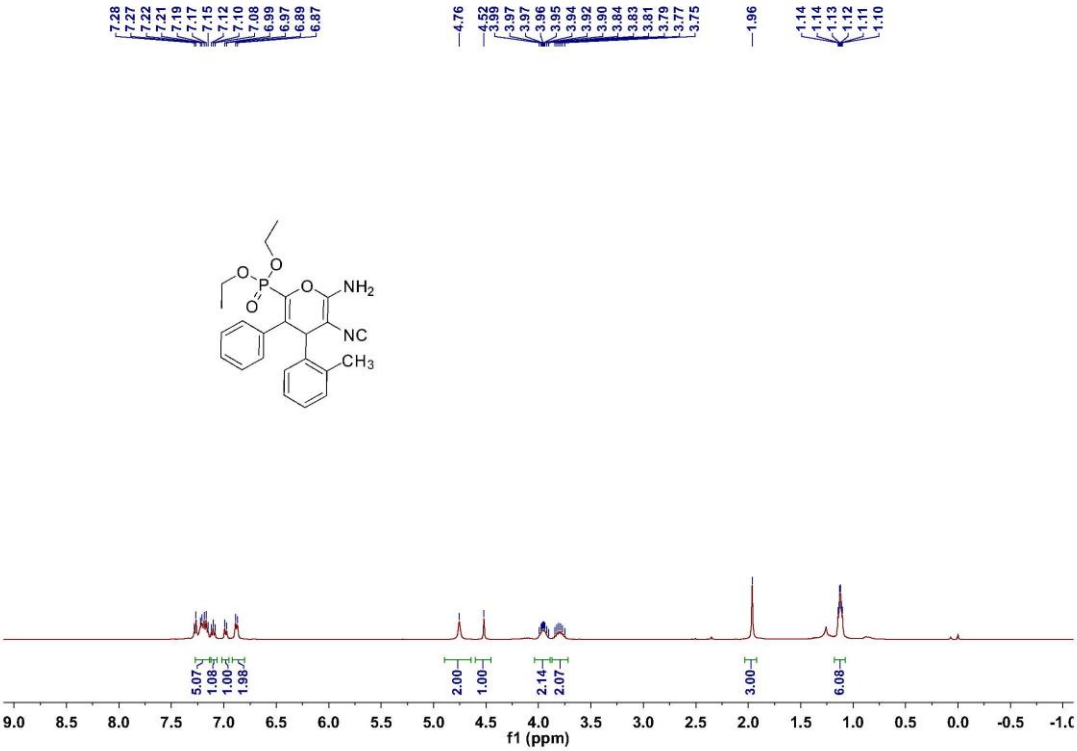
6.75
6.73
4.77
4.71
4.00
3.98
3.96
3.95
3.94
3.94
3.92
3.90
3.86
3.84
3.82
3.80
3.78
3.76
3.74
3.57
1.14
1.12
1.10
1.08
NH2
NC
OCH3
4.96
1.98
1.01
0.99
1.90
0.99
2.06
2.11
3.00
6.01
f1 (ppm)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7.21
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7.12
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7.08
6.99
6.97
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6.87
4.76
4.52
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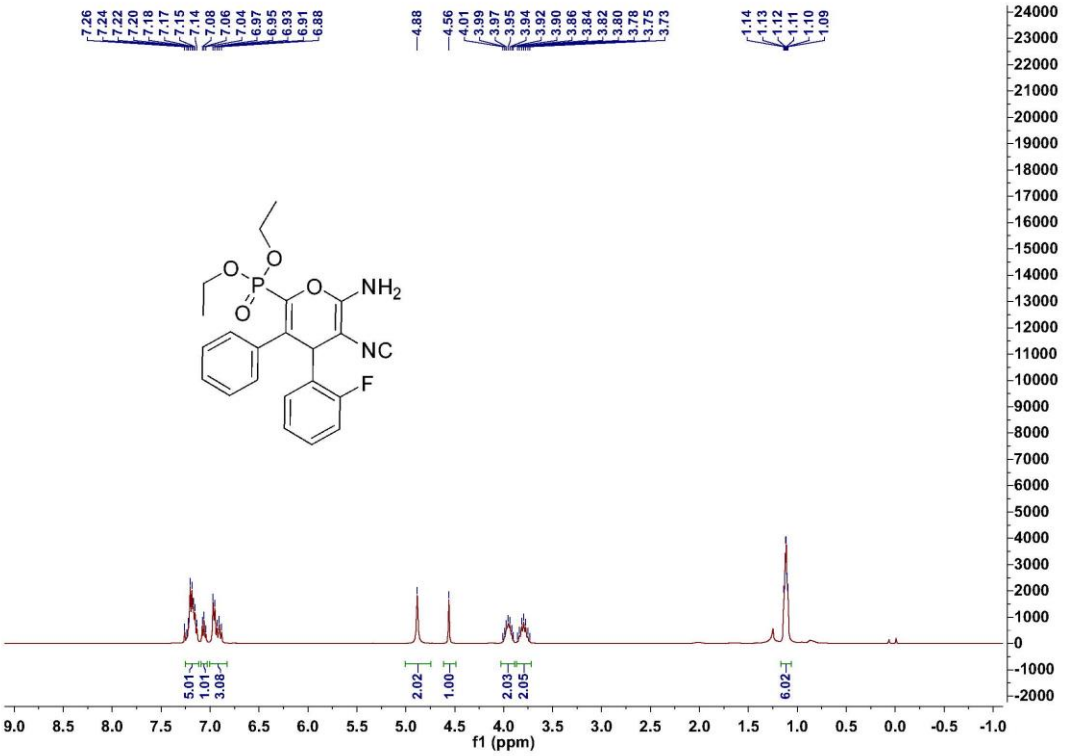
3.97
3.97
3.96
3.95
3.94
3.92
3.90
3.84
3.83
3.81
3.79
3.77
3.75
1.96
1.14
1.14
1.13
1.12
1.11
1.10
NH2
NC
CH3
5.07
1.08
1.00
1.98
2.00
1.00
2.14
2.07
3.00
6.08
f1 (ppm)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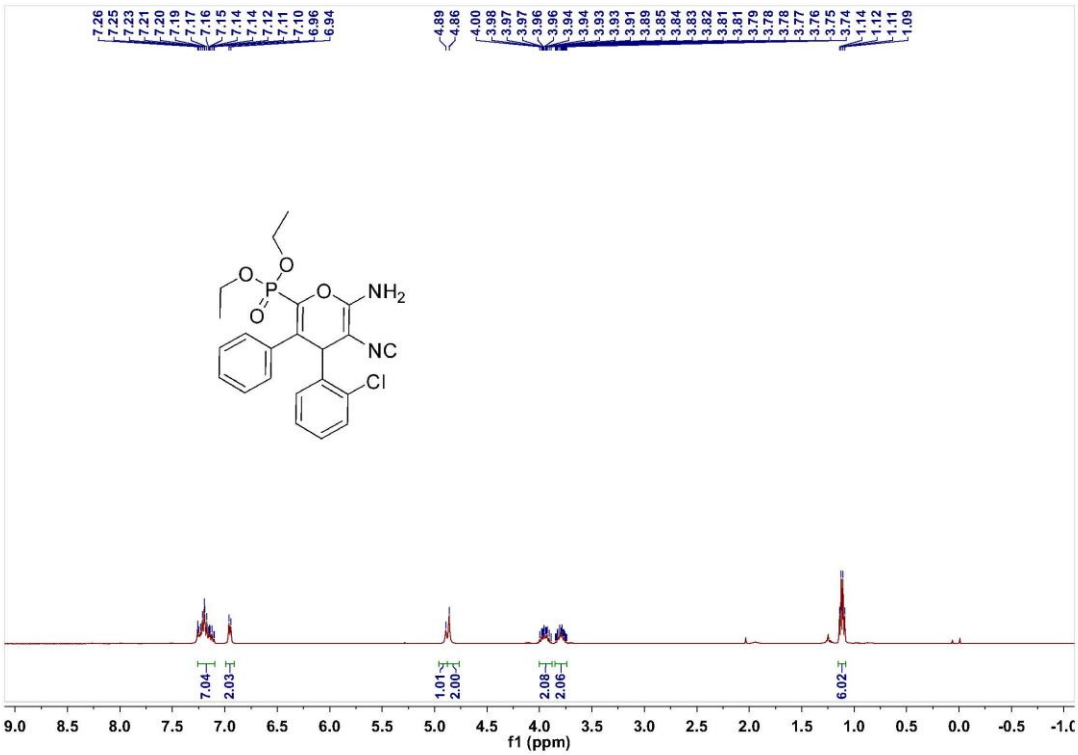
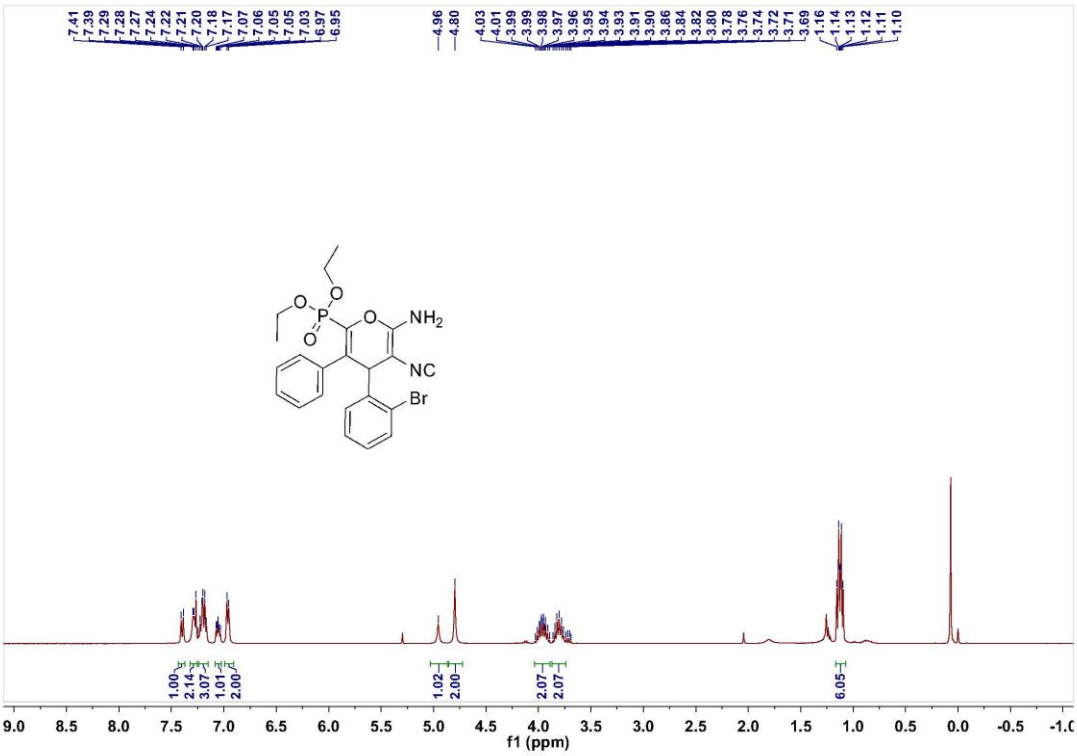
4.88 4.56 4.01 3.99 3.97 3.95 3.94 3.92 3.90 3.86 3.84 3.82 3.80 3.78 3.75 3.73
1.14 1.13 1.12 1.11 1.10 1.09
NH2
NC
F
5.01 1.01 3.08 2.02 1.00 2.03 2.05 6.02
f1 (ppm)


7.26 7.25 7.23 7.21 7.20 7.19 7.17 7.16 7.15 7.14 7.14 7.12 7.11 7.10 6.96 6.94
4.89 4.86 4.00 3.98 3.97 3.97 3.96 3.96 3.94 3.94 3.93 3.93 3.91 3.89 3.85 3.84 3.83 3.82 3.81 3.81 3.79 3.78 3.78 3.77 3.76 3.75 3.74 1.14 1.12 1.11 1.09
NH2
NC
Cl
7.04 2.03 1.01 2.00 2.08 2.06 6.02
f1 (ppm)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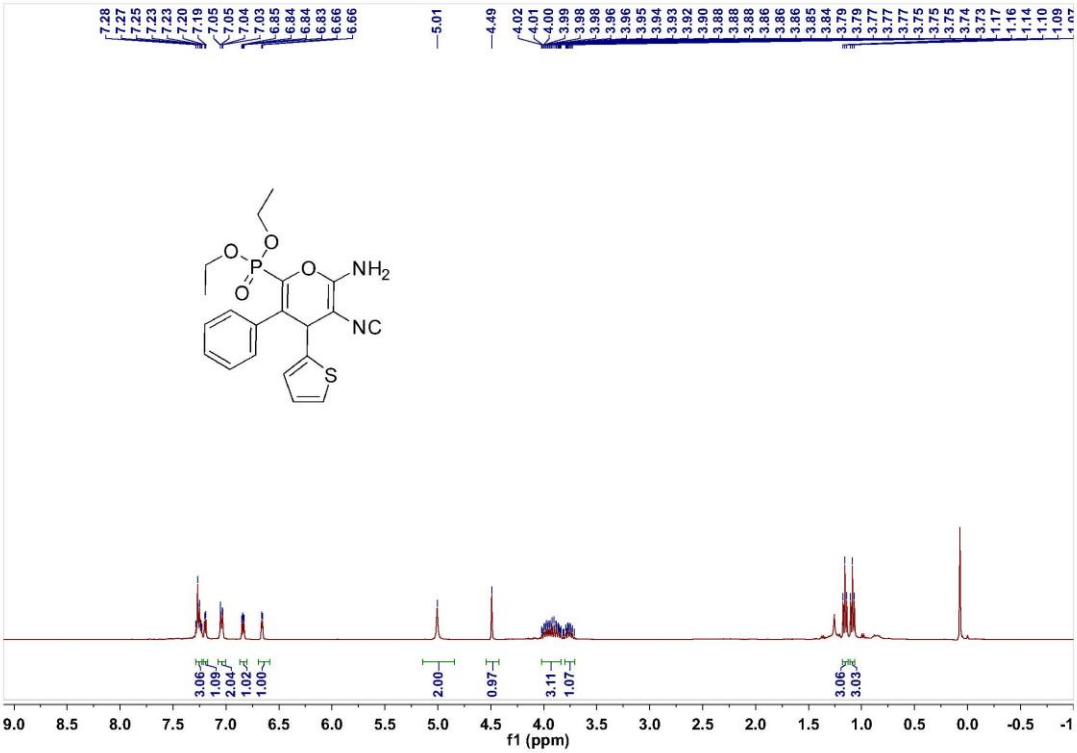
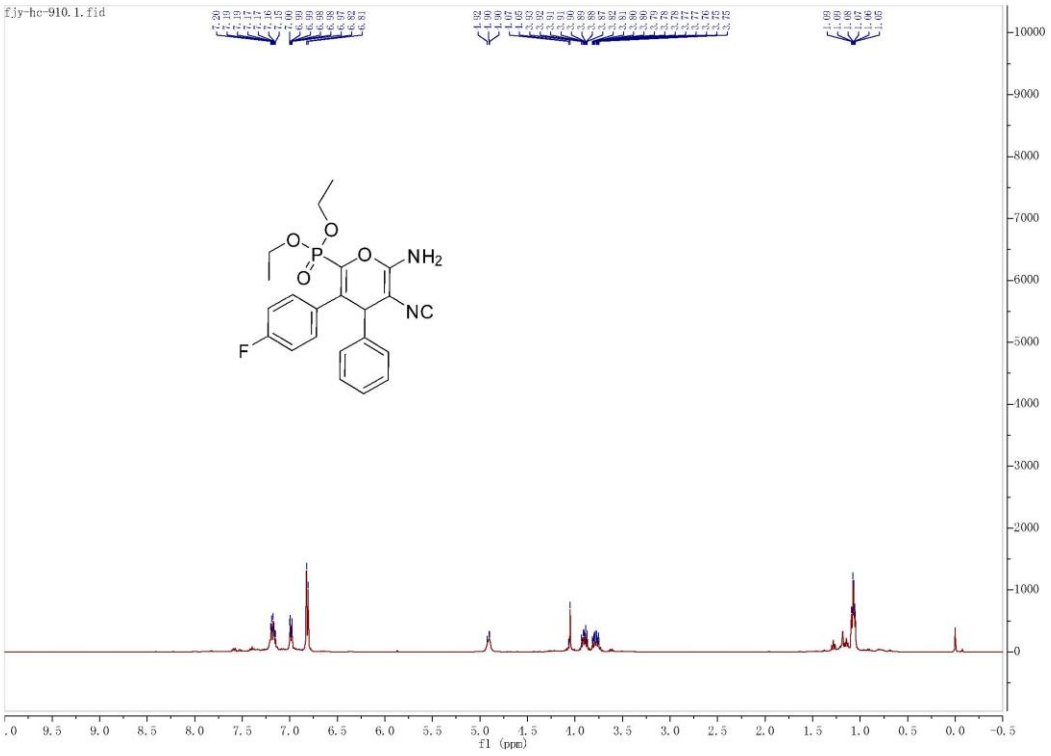
4.96 4.80
4.03 4.01 3.99 3.99 3.98 3.97 3.96 3.95 3.94 3.93 3.91 3.90 3.86 3.84 3.82 3.80 3.78 3.76 3.74 3.72 3.71 3.69
1.16 1.14 1.13 1.12 1.11 1.10
NH2
NC
Br
1.00 2.14 3.07 1.01 2.00
1.02 2.00
2.07 2.07
6.05
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


7.28 7.27 7.25 7.23 7.23 7.20 7.19 7.05 7.05 7.04 7.03 6.85 6.84 6.84 6.83 6.66 6.66
5.01
4.49
4.02 4.01 4.00 3.99 3.98 3.98 3.96 3.96 3.95 3.94 3.93 3.92 3.90 3.88 3.88 3.88 3.86 3.86 3.86 3.85 3.84 3.79 3.79 3.77 3.77 3.77 3.75 3.75 3.75 3.74 3.73 1.17 1.16 1.14 1.10 1.09
NH2
NC
S
3.06 1.09 2.04 1.02 1.00
2.00
0.97
3.11 1.07
3.06 3.03
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 -0.5
f1 (ppm)


fjy-hc-910.1.fid
NH2
NC
F
fl (ppm)