

A General Method for the Preparation of Active Esters by Palladium Catalyzed Alkoxy carbonylation of Aryl Bromides.

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Supporting information

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General Methods

All purchased chemicals were used as received without further purification. Solvents were dried according to standard procedures and flash chromatography was carried out on silica gel 60 (230-400 mesh). The chemical shifts are reported in ppm relative to solvent residual peak. The ^1H NMR spectra were recorded at 400 MHz, ^{13}C NMR spectra were recorded at 100 MHz and ^{19}F NMR spectra were recorded at 367 MHz. The chemical shifts of the NMR spectra are reported in parts per million (ppm) relative to the solvent residual peak. NMR spectra are reported as follows (s = singlet, d = doublet, t = triplet, q = quartet, quin = quintuplet, sext = sextet, spt = septet, m = multiplet, b = broad, dd = doublet of doublets, dt = doublet of triplets). MS spectra were recorded on a LC TOF (ES) apparatus.

COware

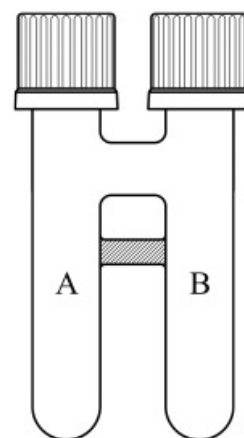
Two glass vials (Chamber A and B) connected with a glass tube to allow gas-transfer.

Total Volume = 20 mL.

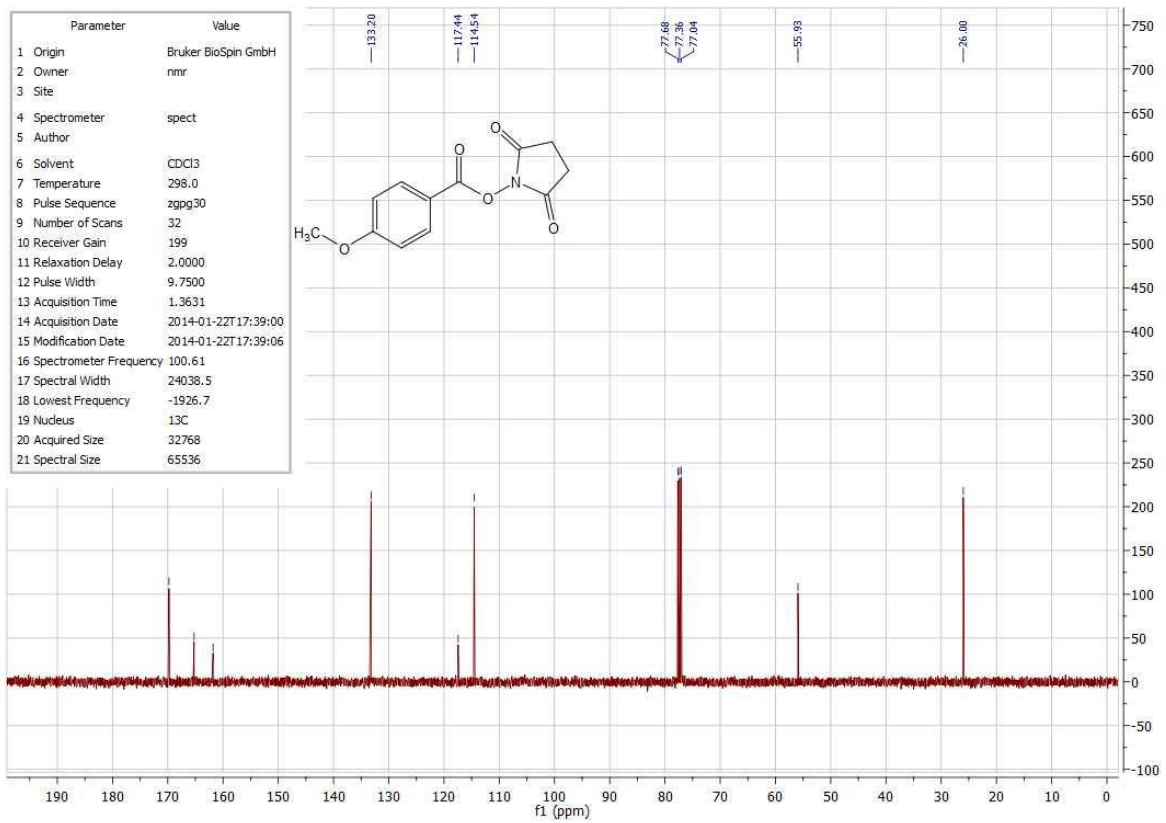
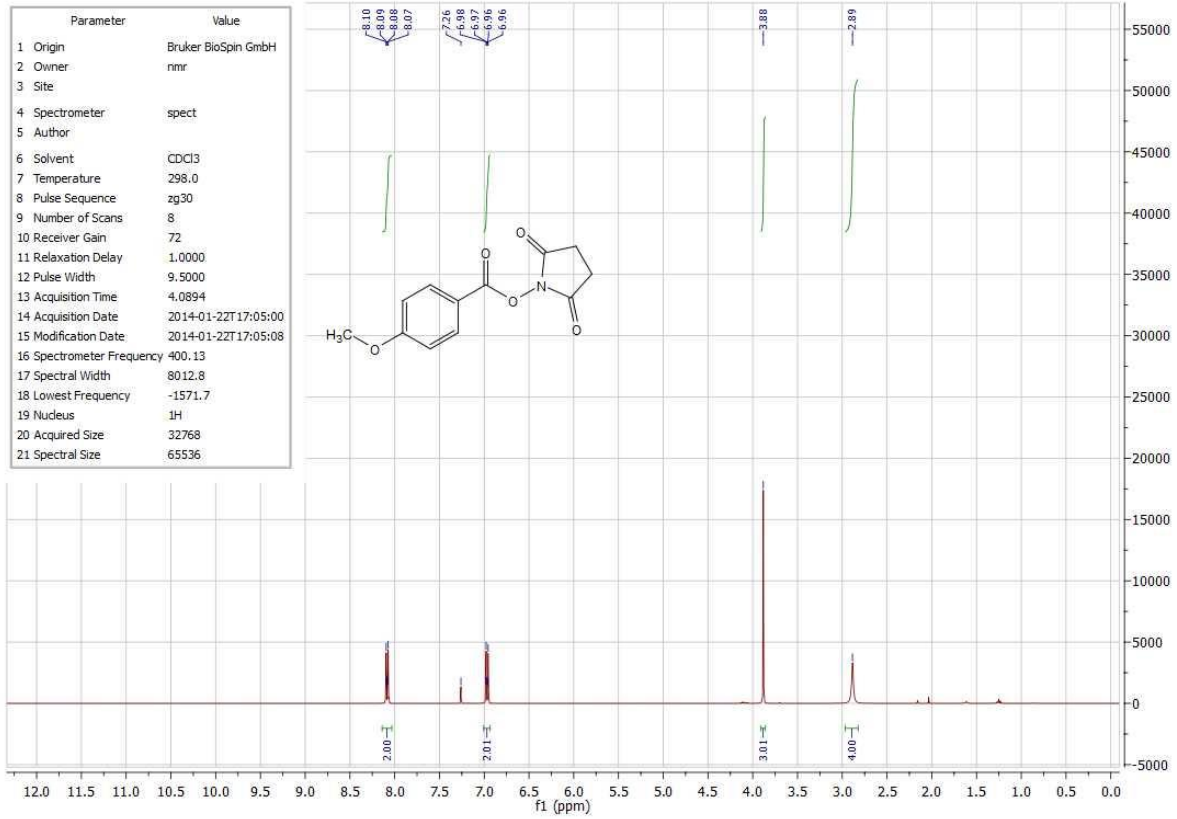
Closed with a screw cap and a Teflon[®] coated silicone seal.

Glassware under pressure - Warning !

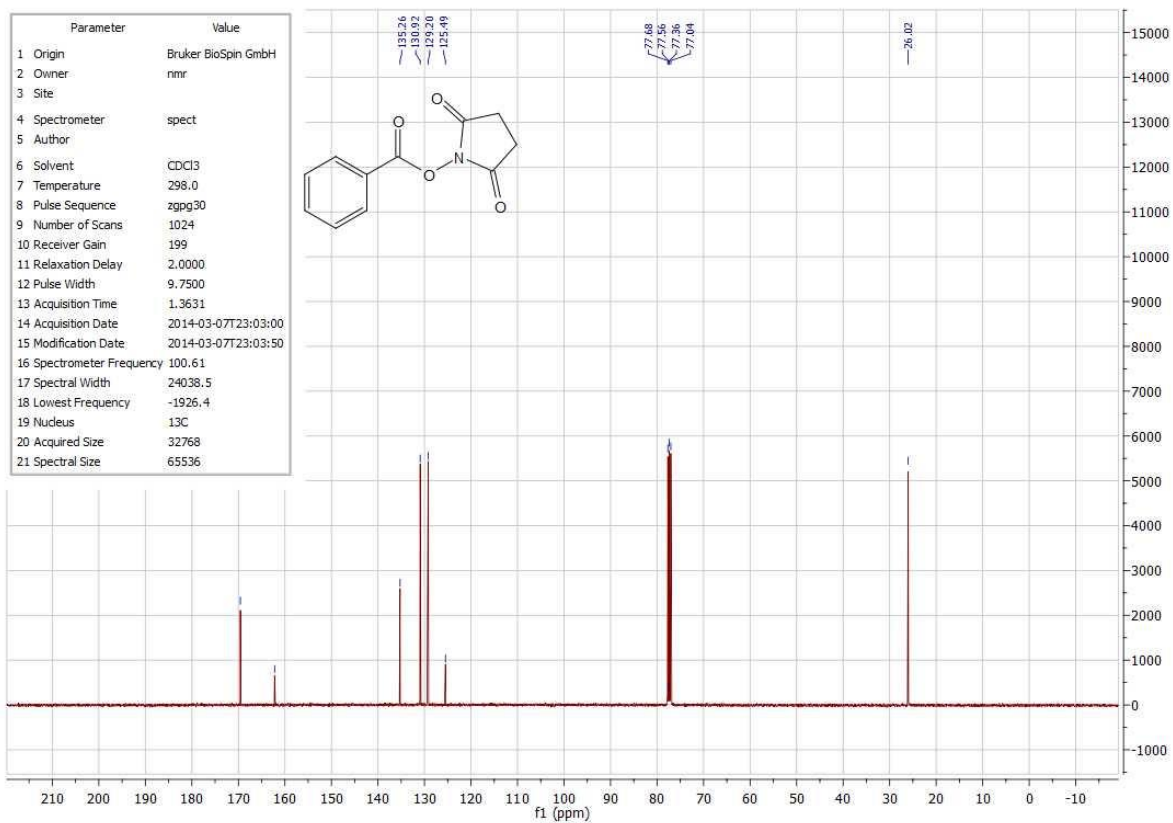
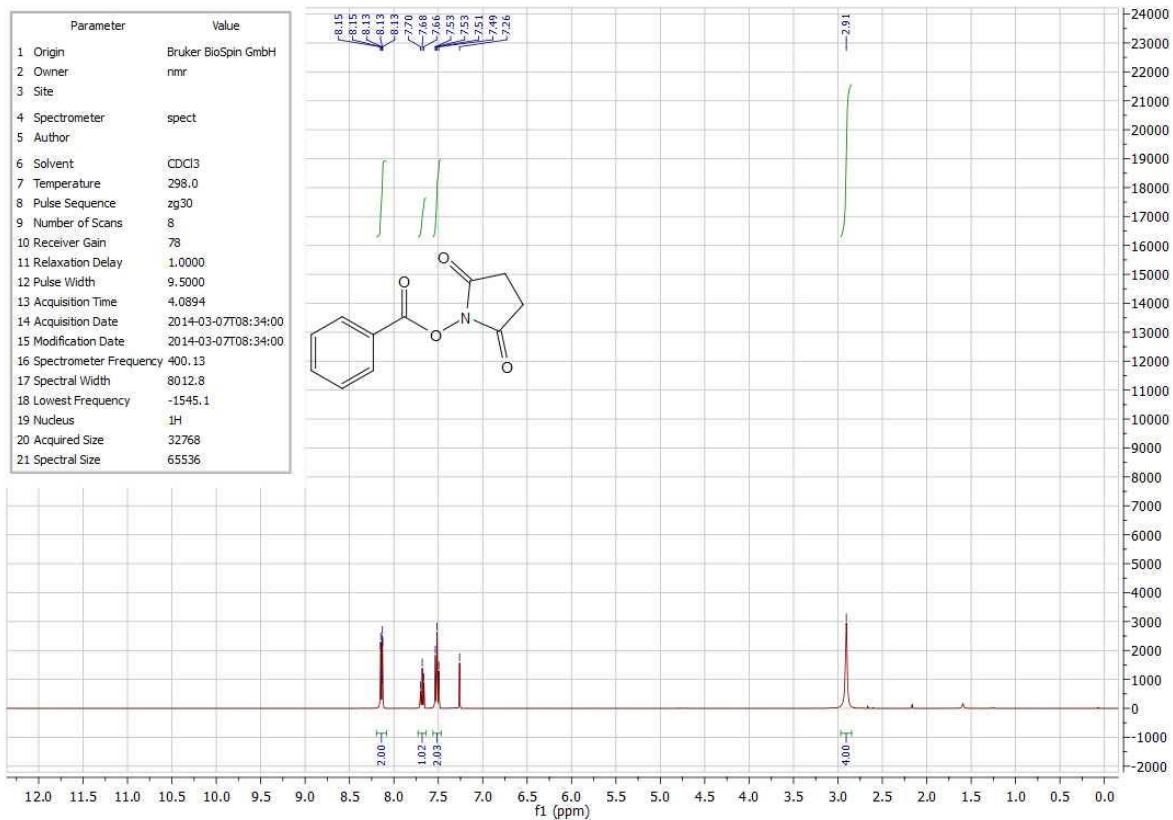
- Glass equipment should always be examined for damages to its surface, which may weaken its strength. S-3
- One must abide to all laboratory safety procedures and always work behind a shield when working with glass equipment under pressure.
- COware is pressure tested to 224 psi, but should under no circumstances be operated above 60 psi (5 bar).



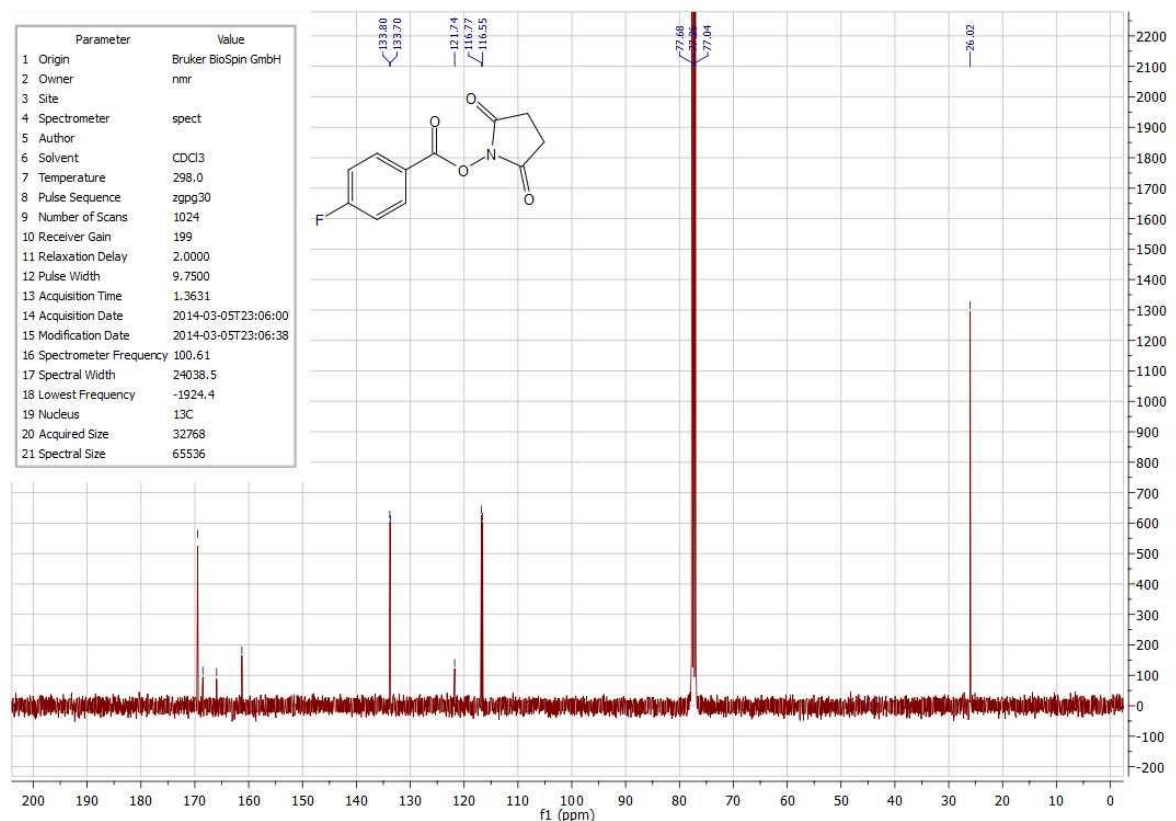
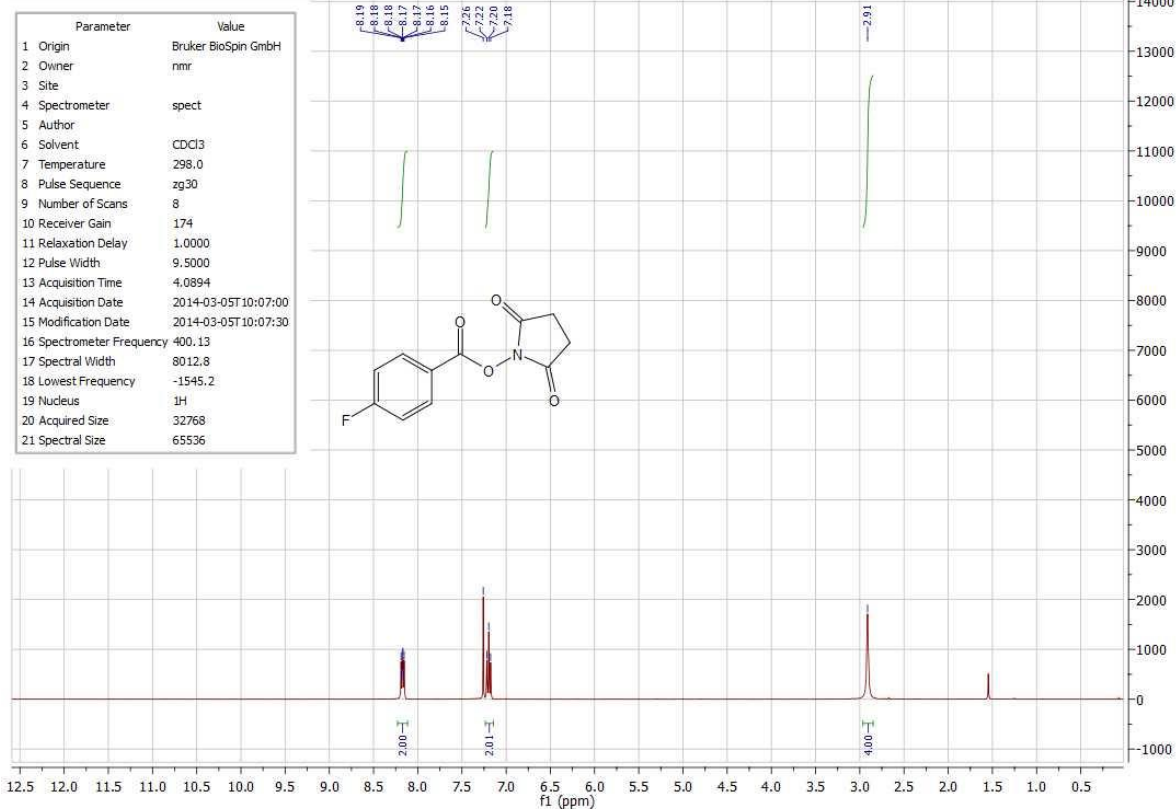
NMR-spectra
(2a) 2,5-dioxopyrrolidin-1-yl 4-methoxybenzoate

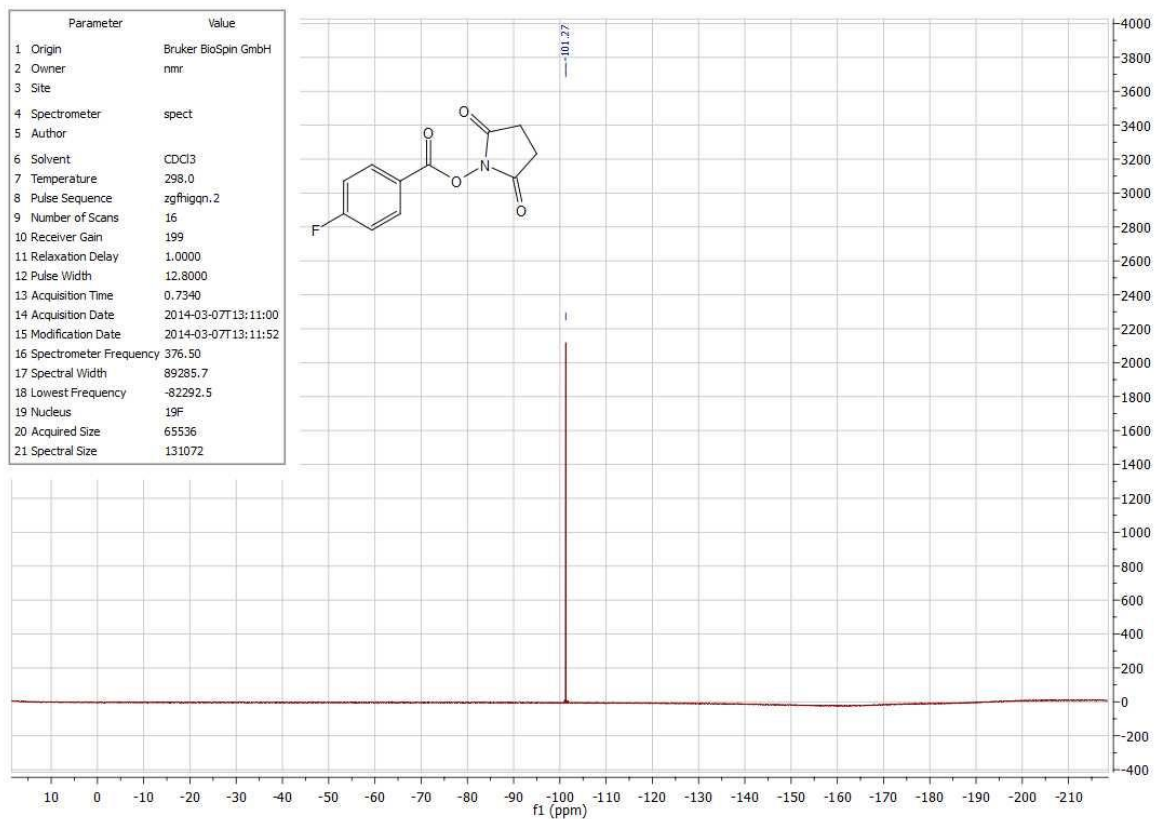


(3a) 2,5-dioxopyrrolidin-1-yl benzoate

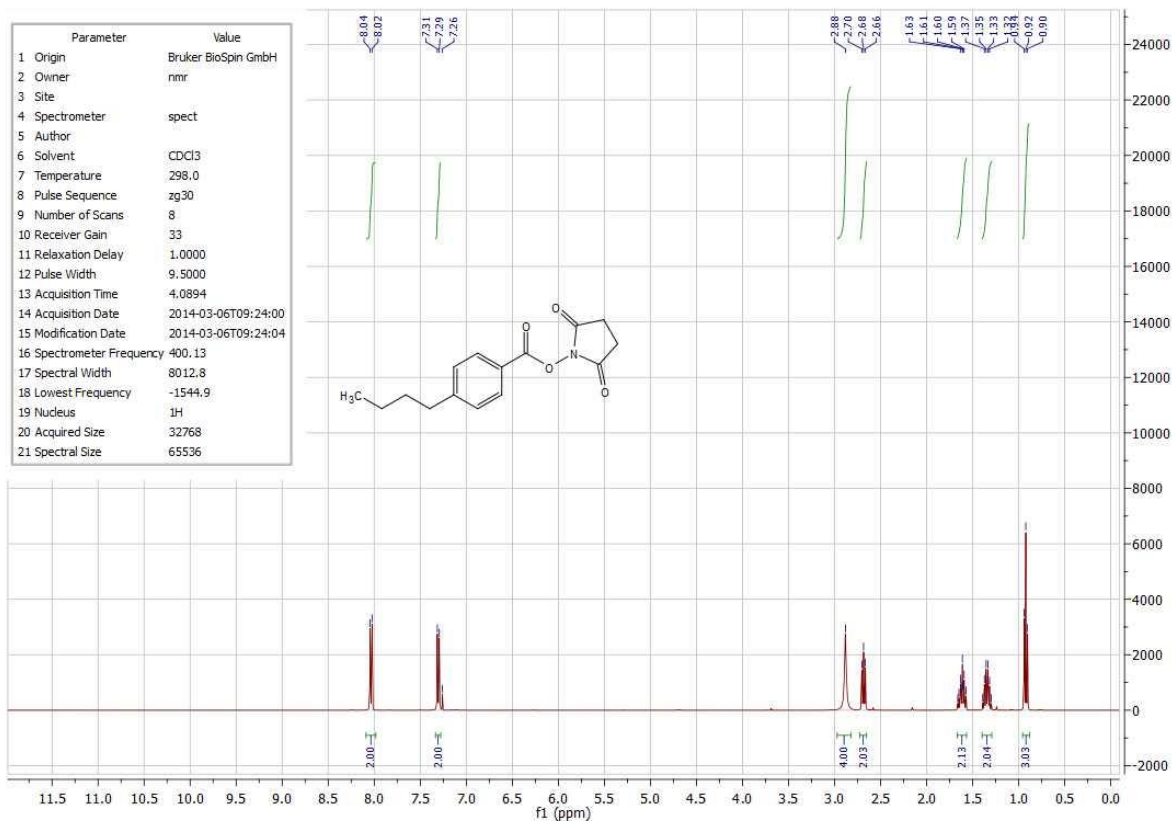


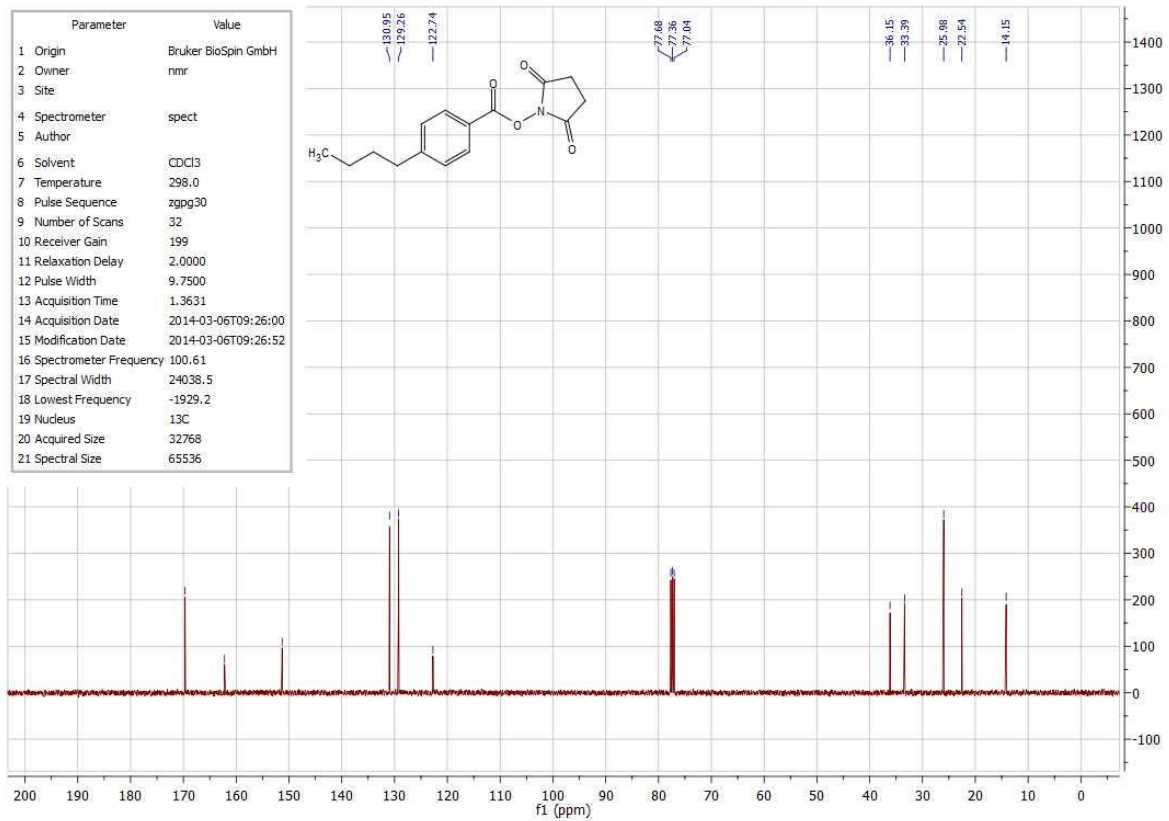
(4a) 2,5-dioxypyrrolidin-1-yl 4-fluorobenzoate



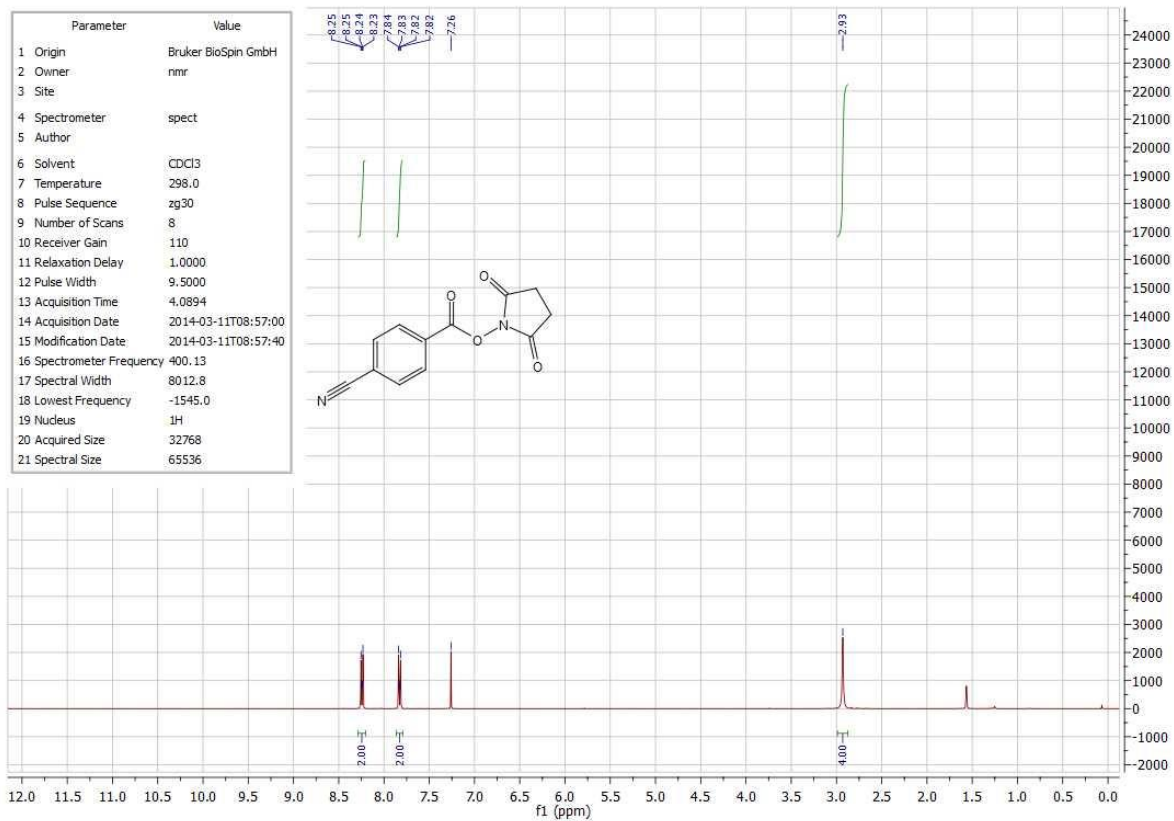


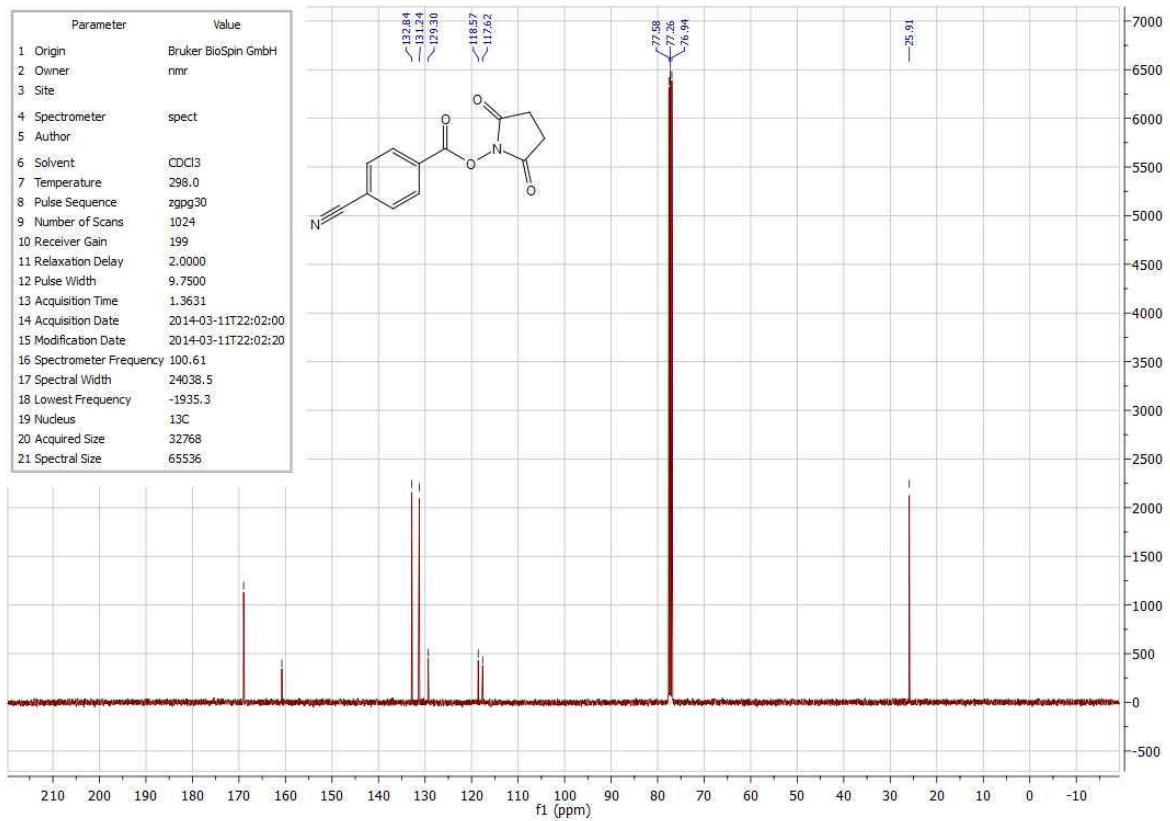
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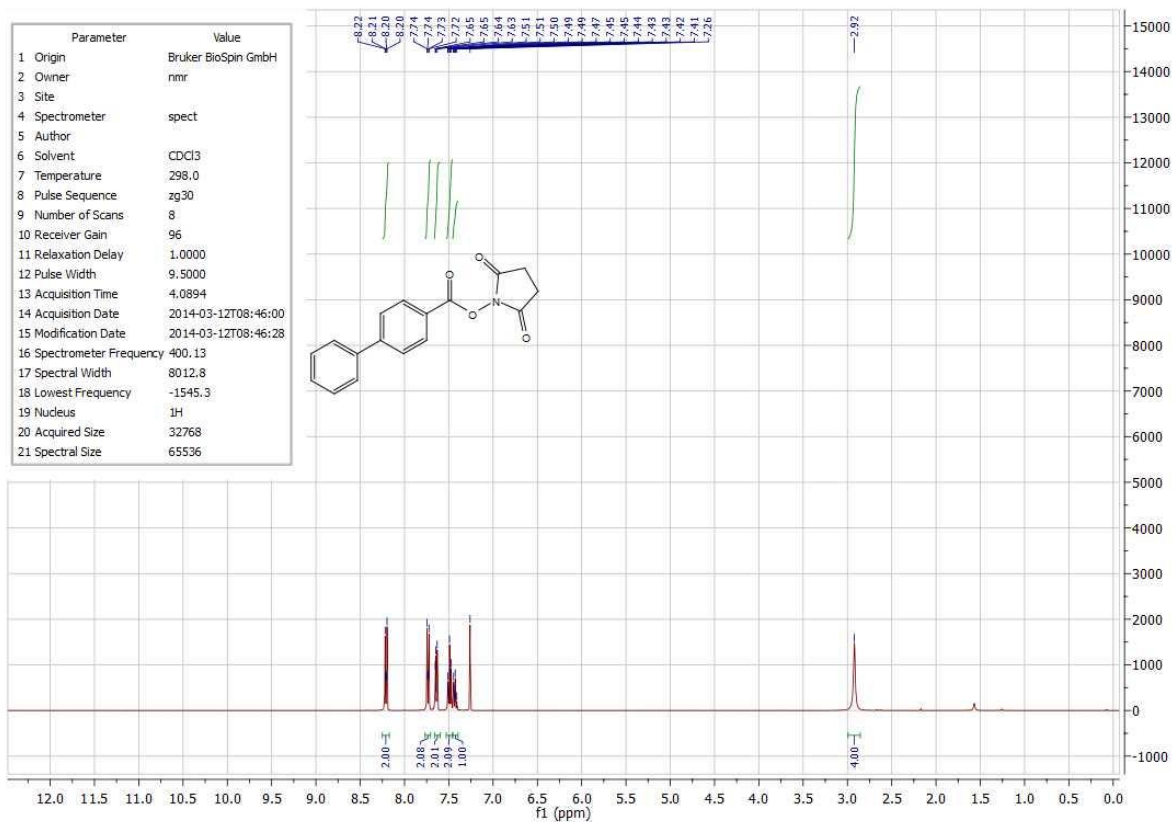


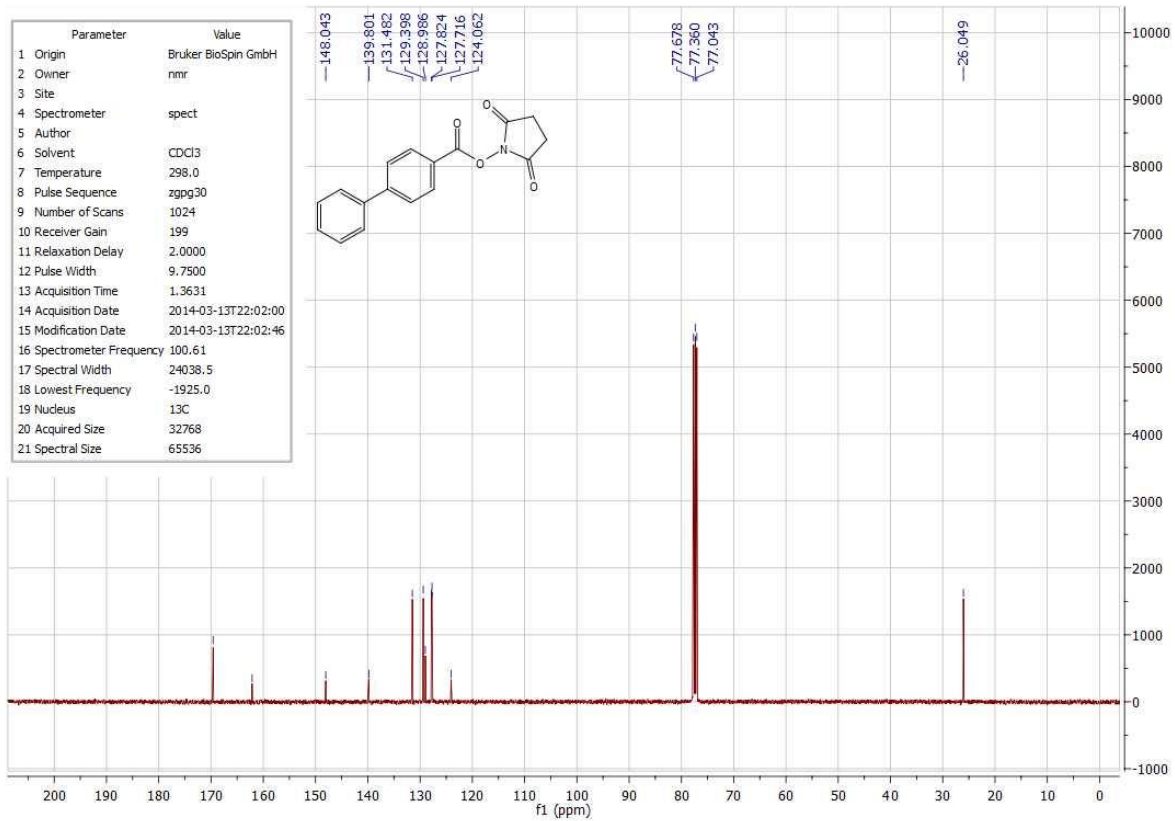
(6a) 2,5-dioxopyrrolidin-1-yl 4-cyanobenzoate



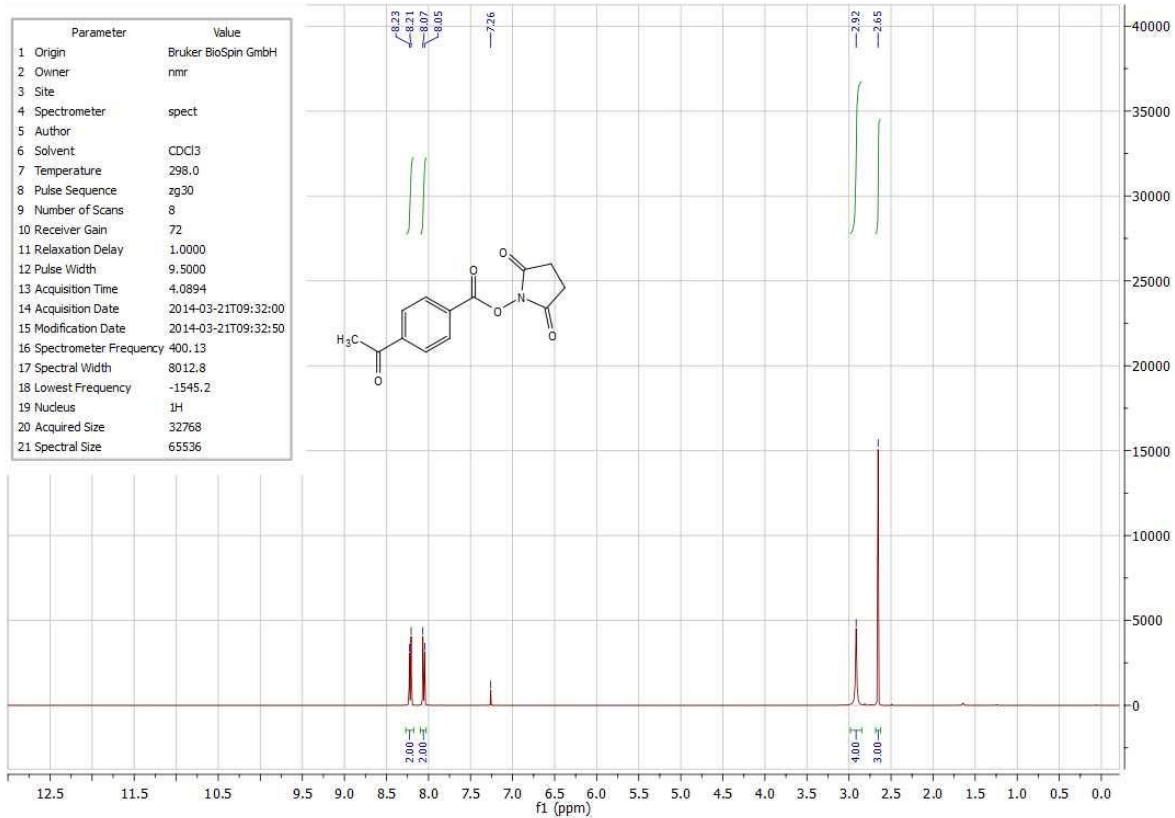


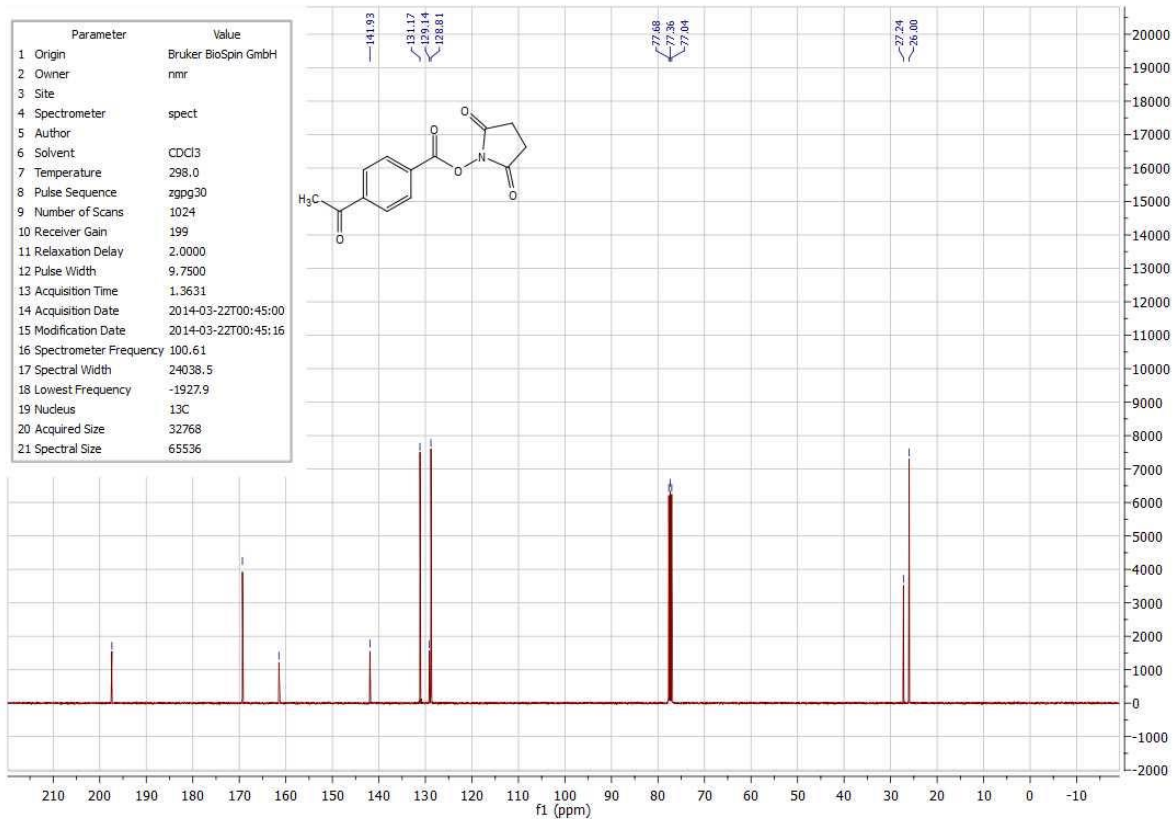
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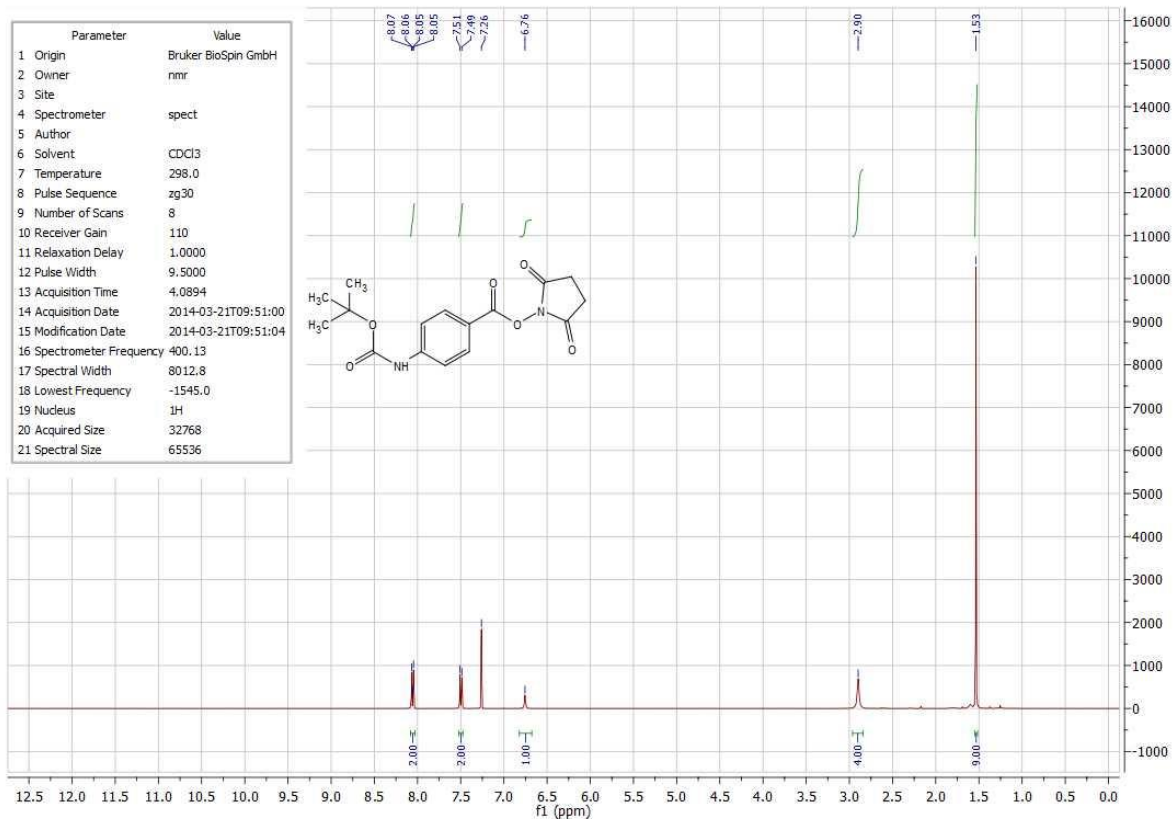


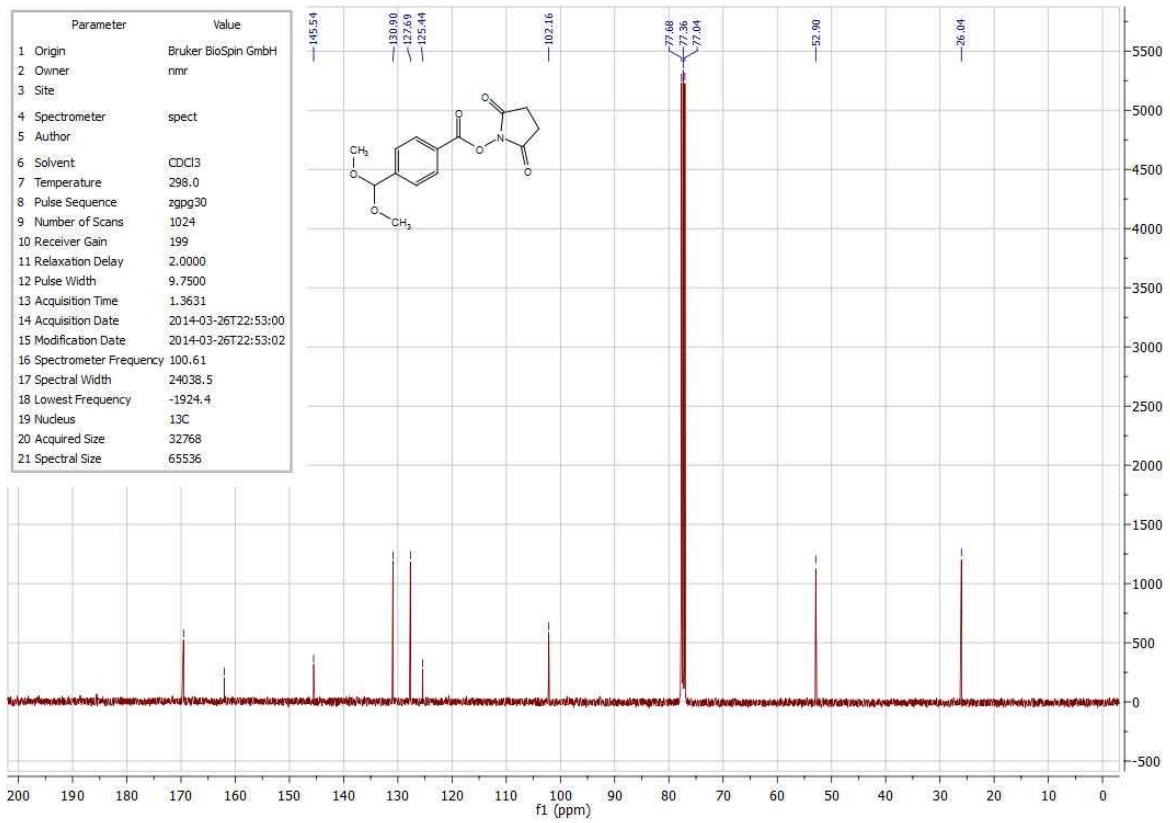
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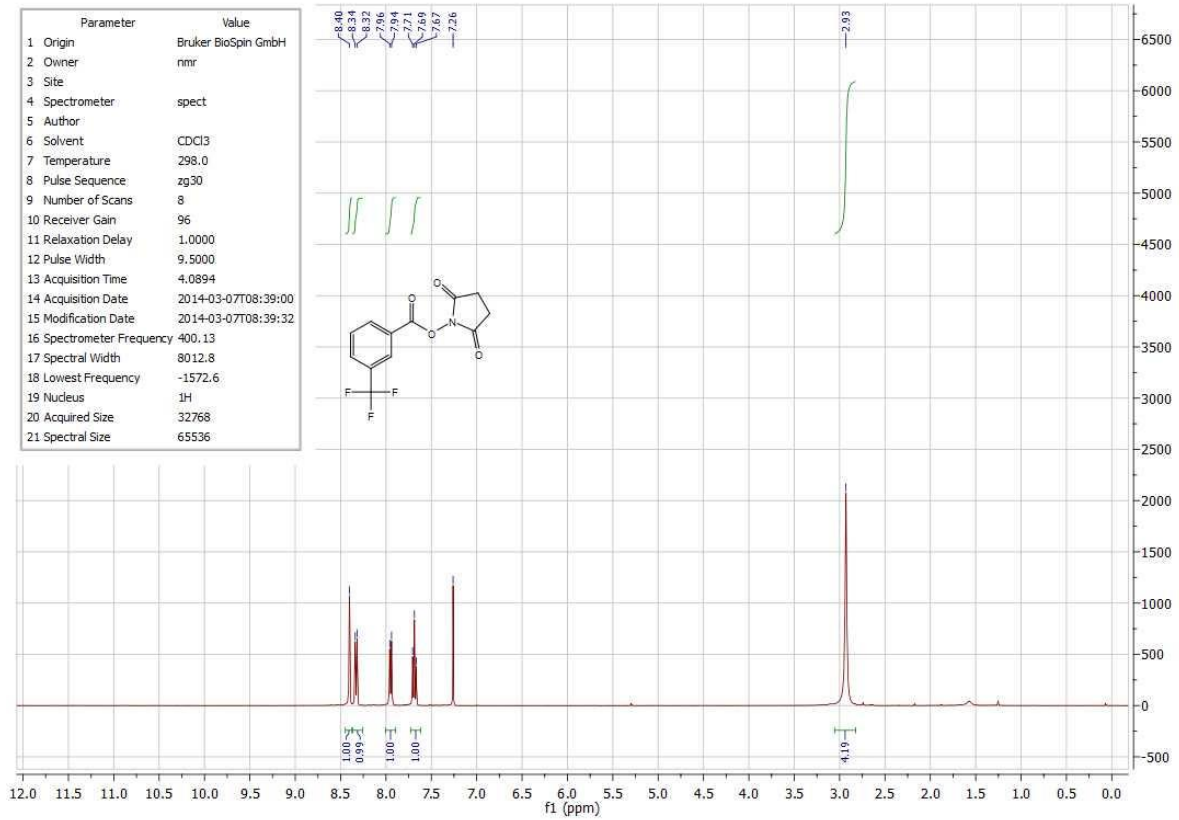


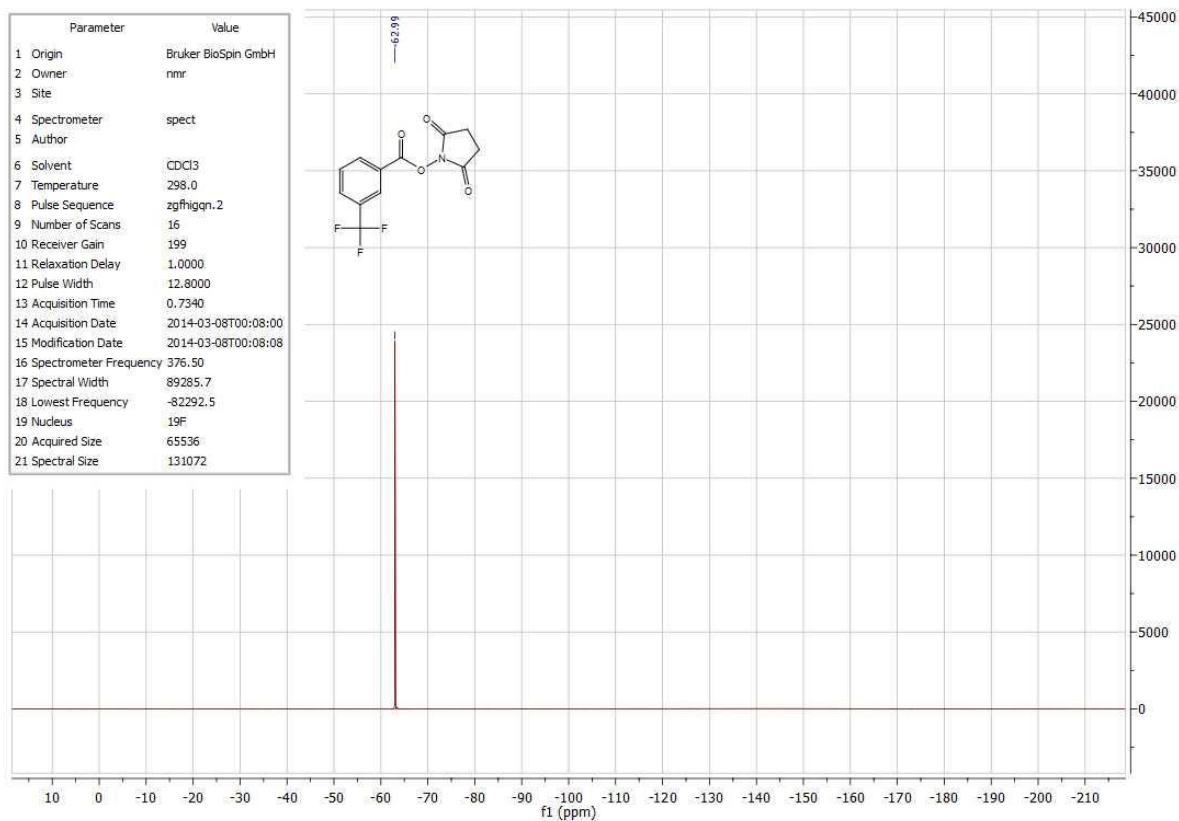
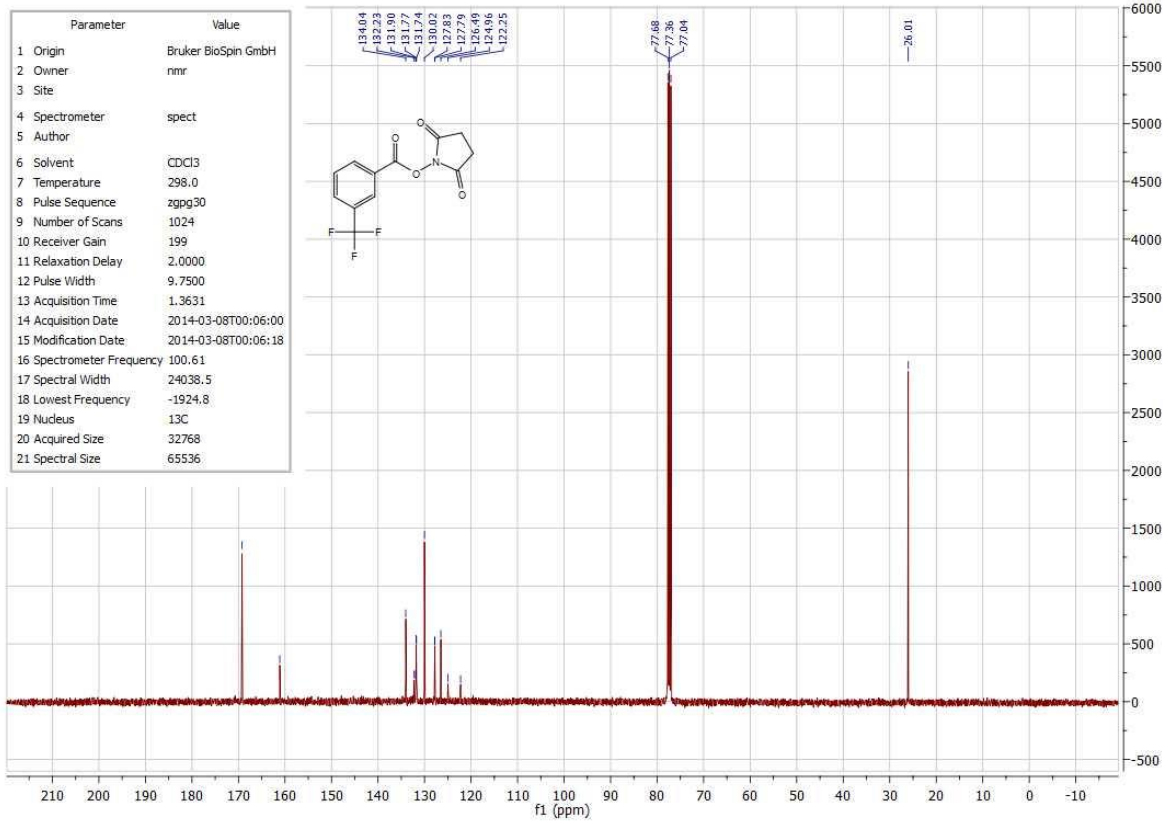
(9a) 2,5-dioxopyrrolidin-1-yl 4-(*tert*-butoxycarbonylamino)benzoate



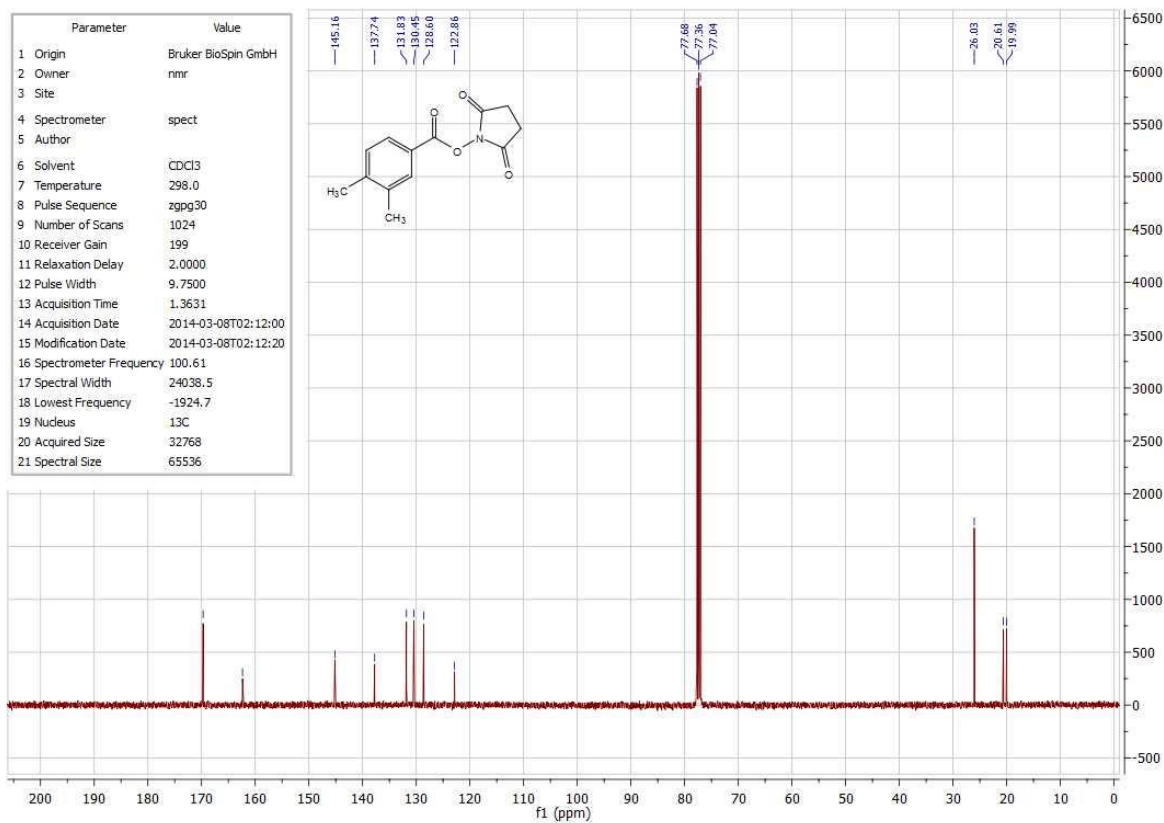
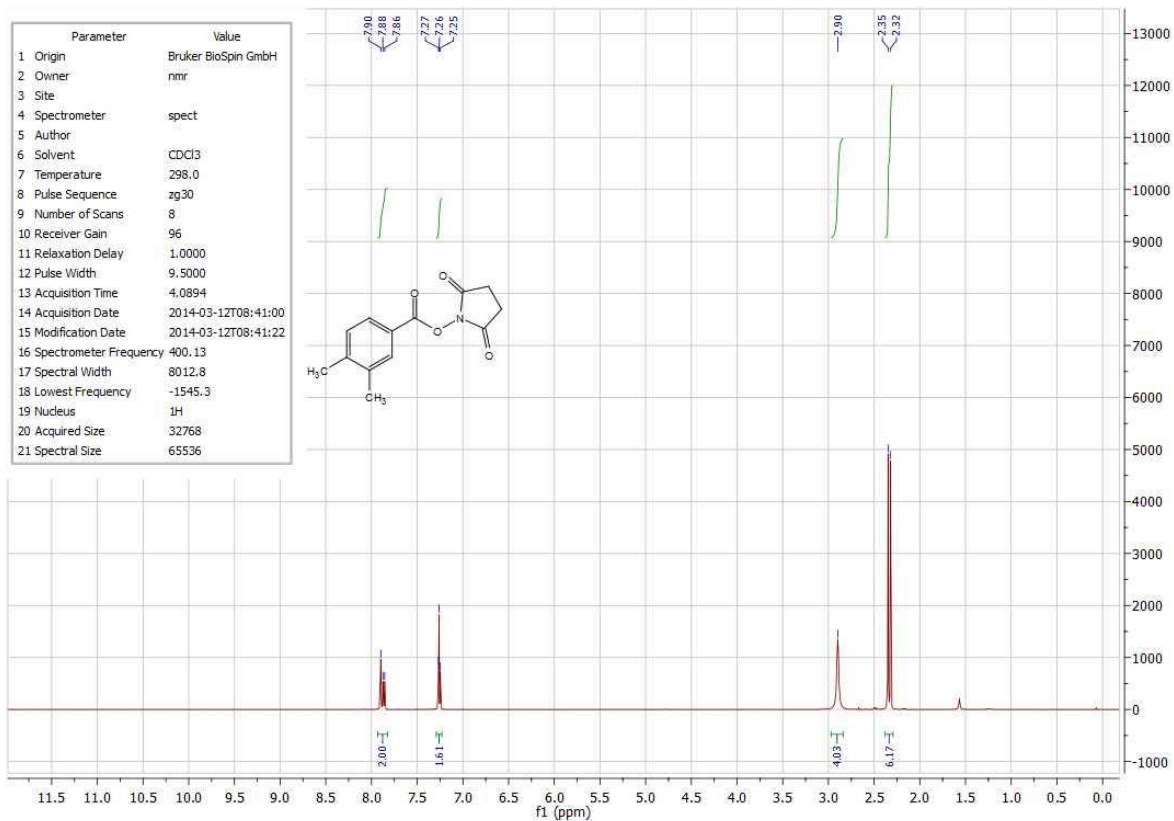


(11a) 2,5-dioxopyrrolidin-1-yl 3-(trifluoromethyl)benzoate

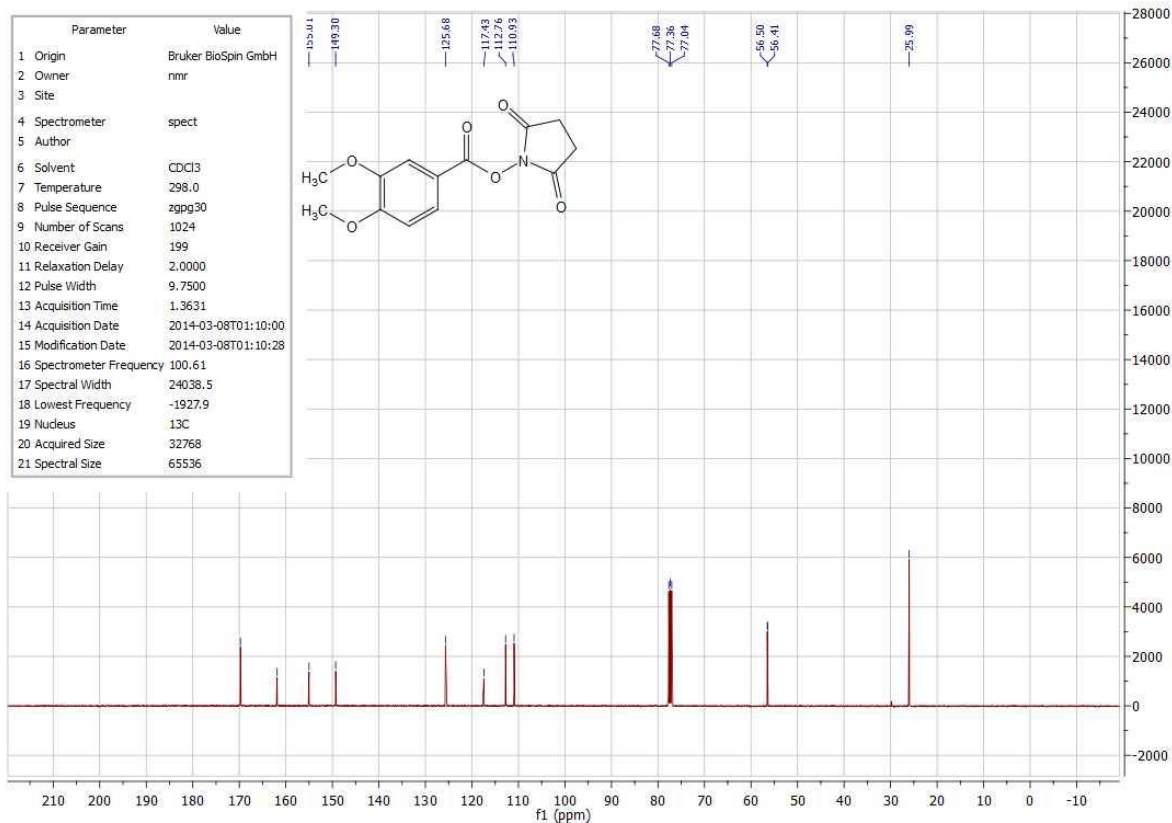
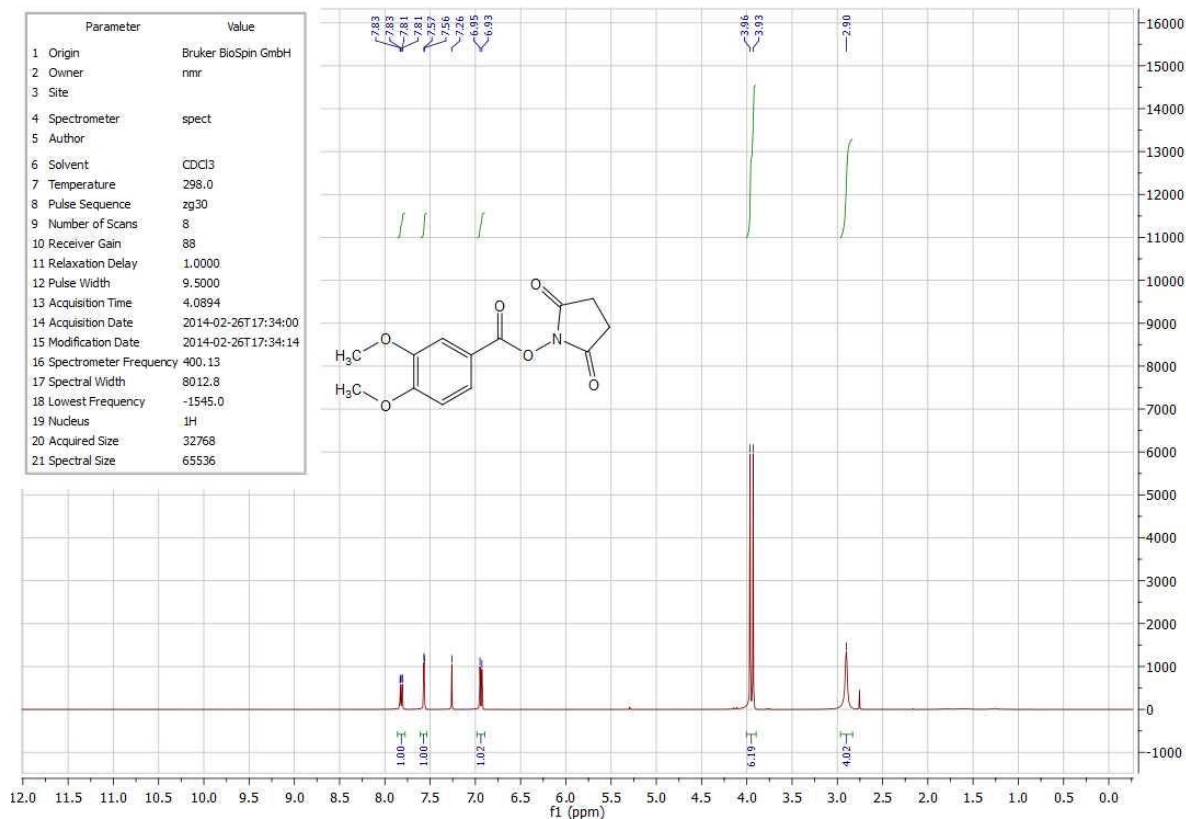




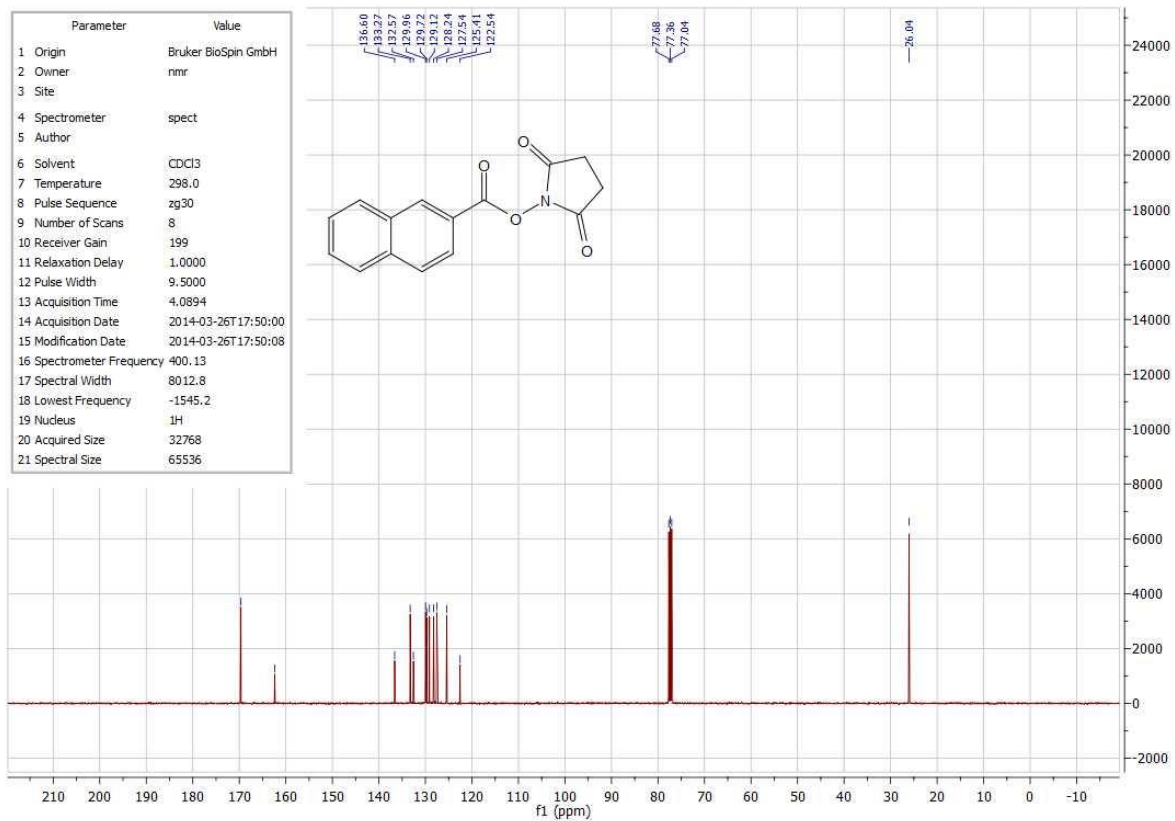
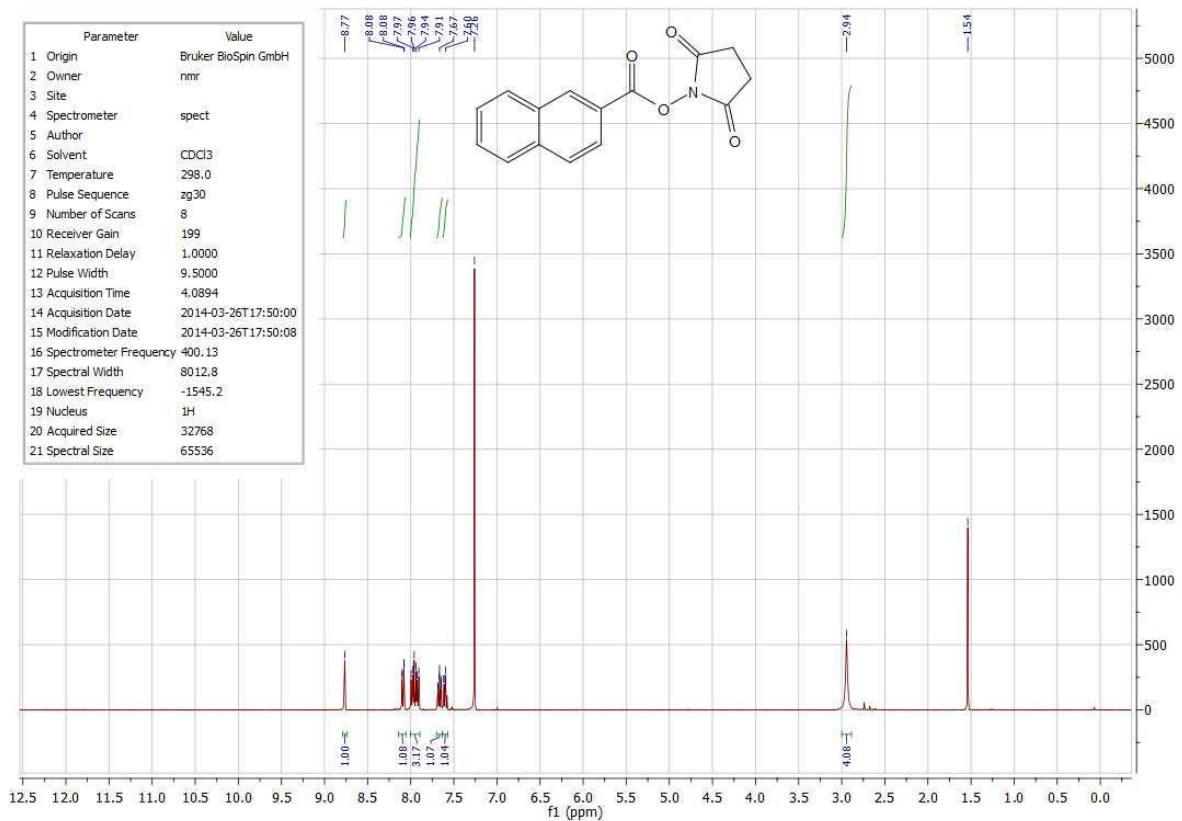
(12a) 2,5-dioxopyrrolidin-1-yl 3,4-dimethylbenzoate



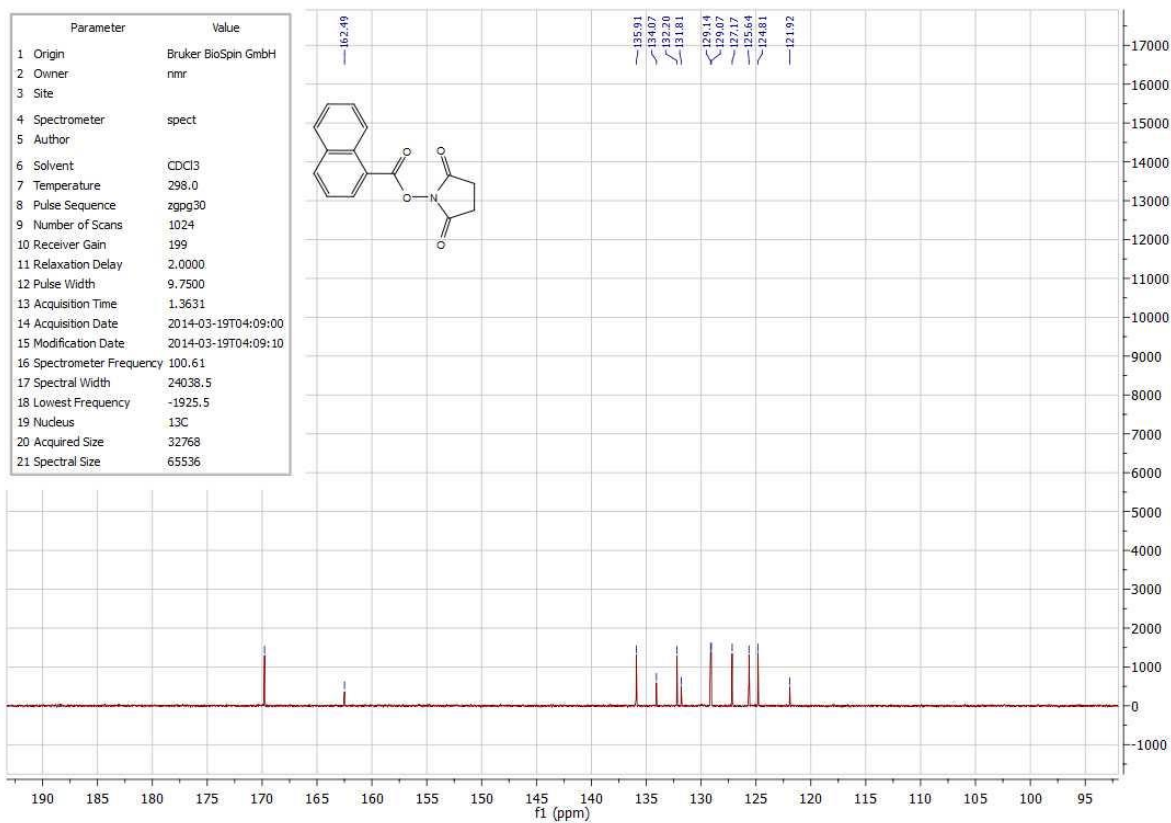
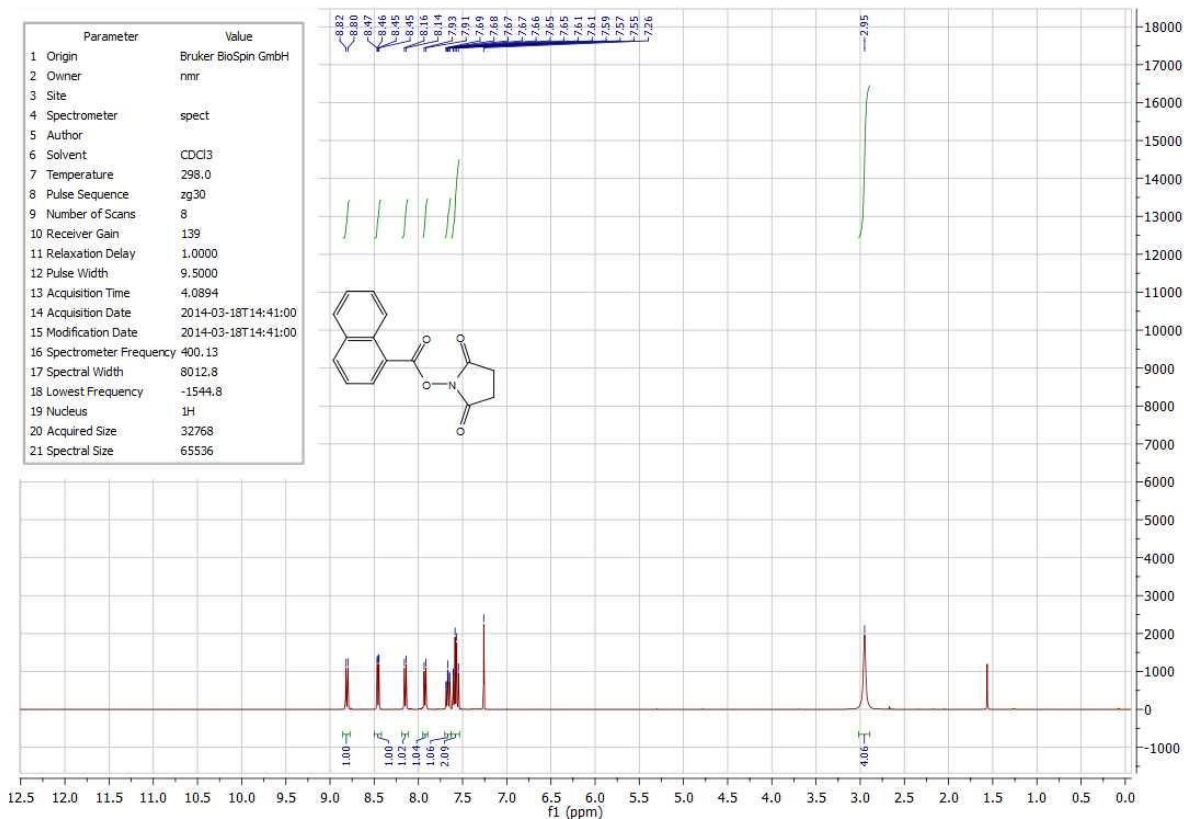
(13a) 2,5-dioxopyrrolidin-1-yl 3,4-dimethoxybenzoate



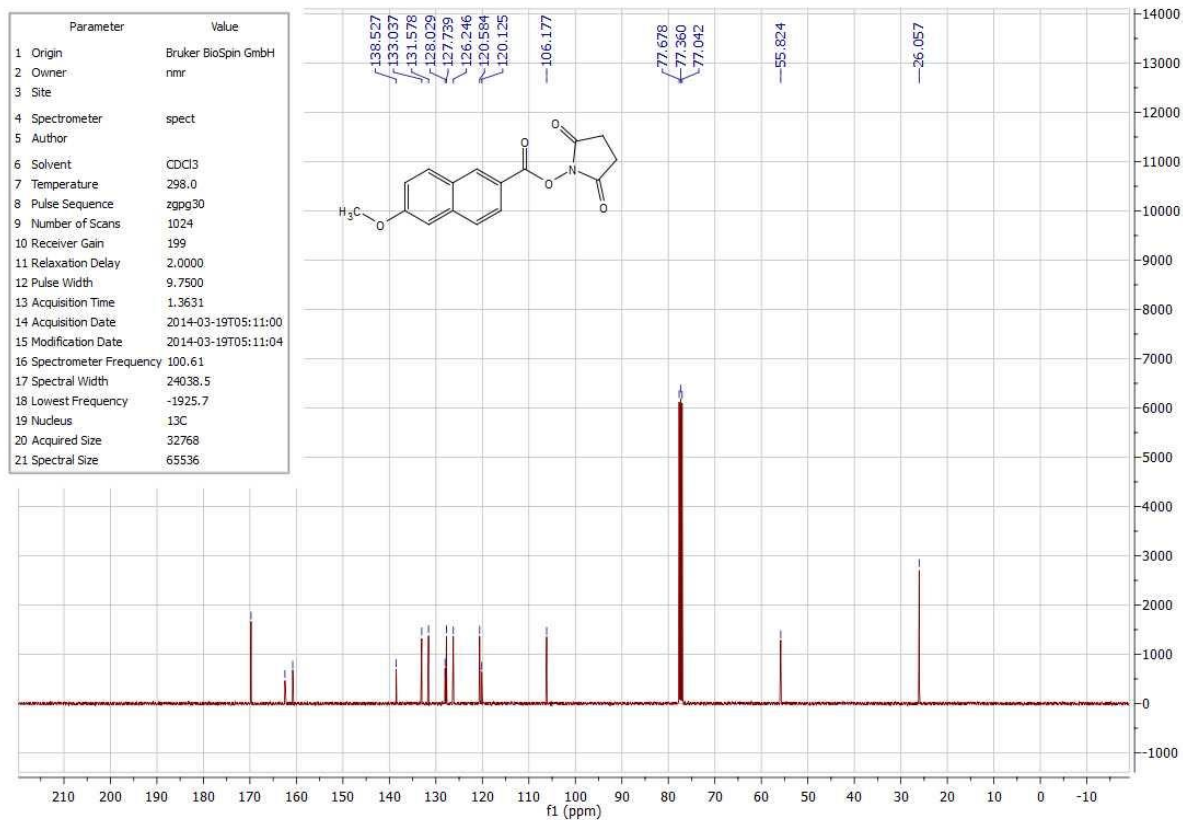
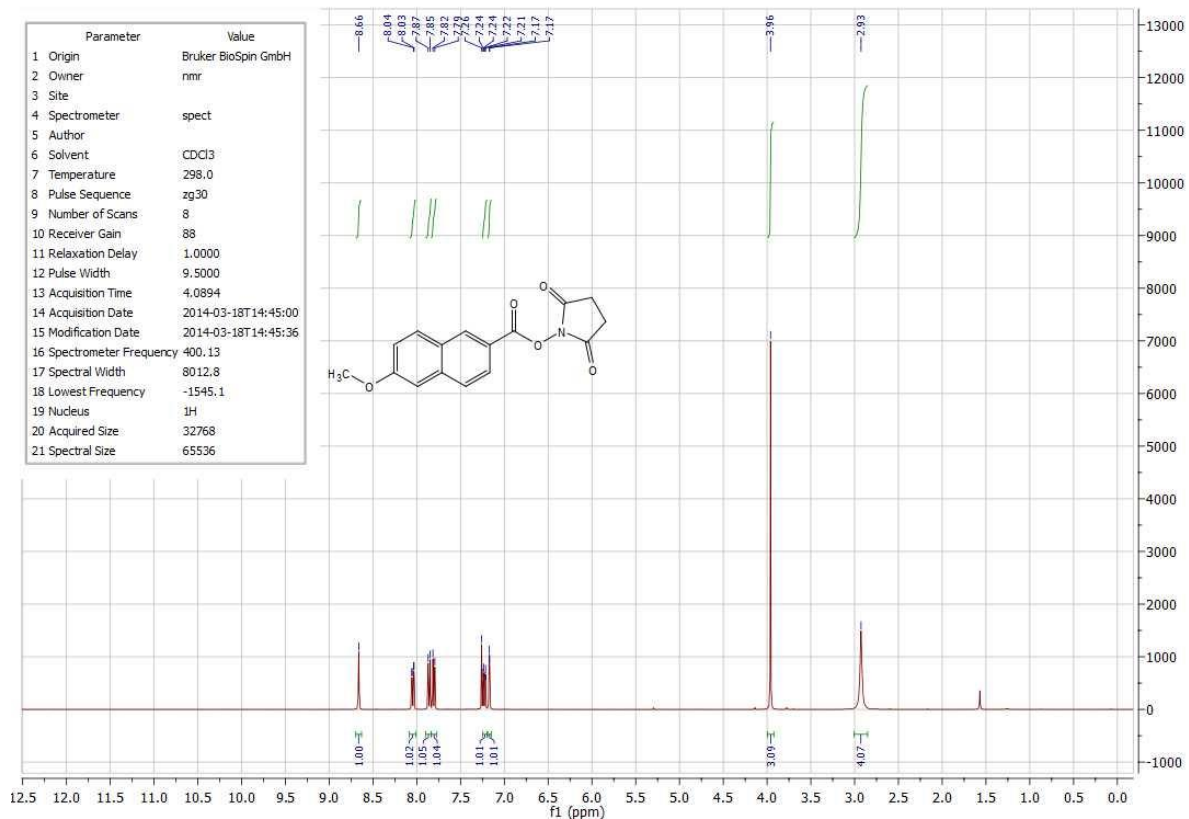
(14a) 2,5-dioxopyrrolidin-1-yl 2-naphthoate



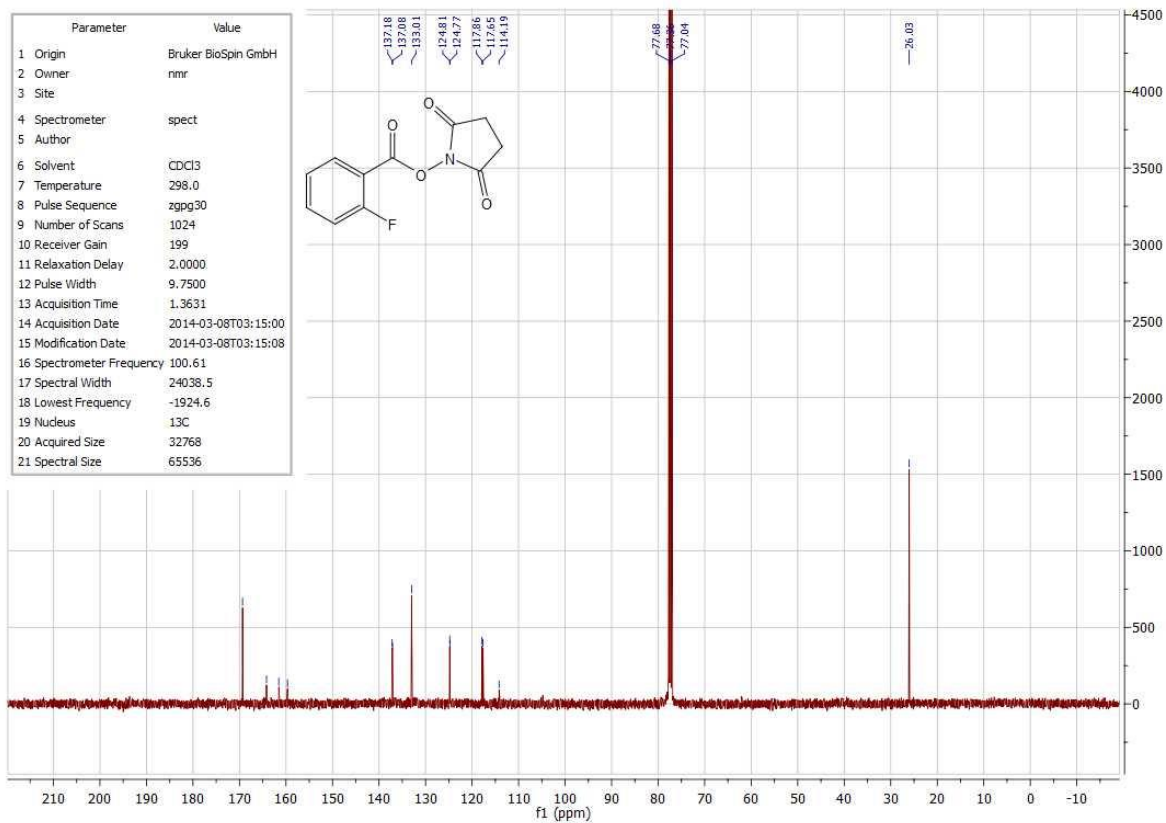
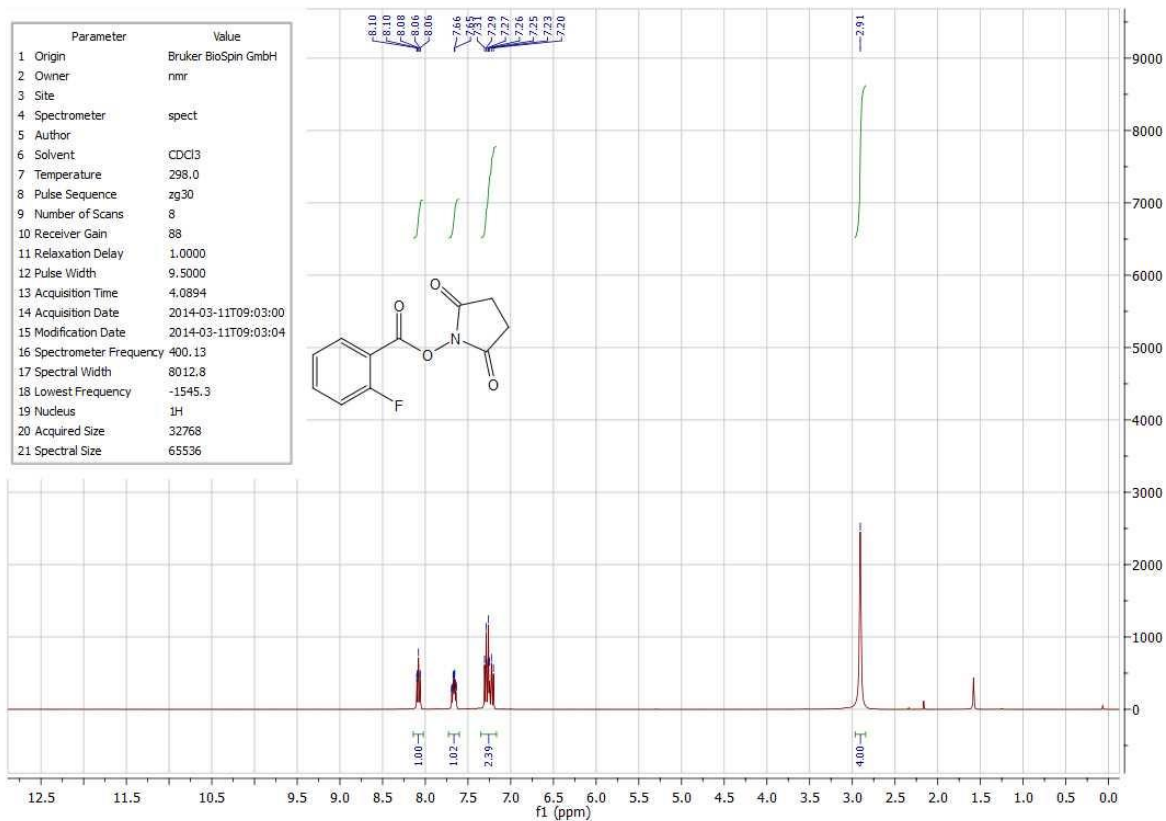
(15a) 2,5-dioxopyrrolidin-1-yl 1-naphthoate

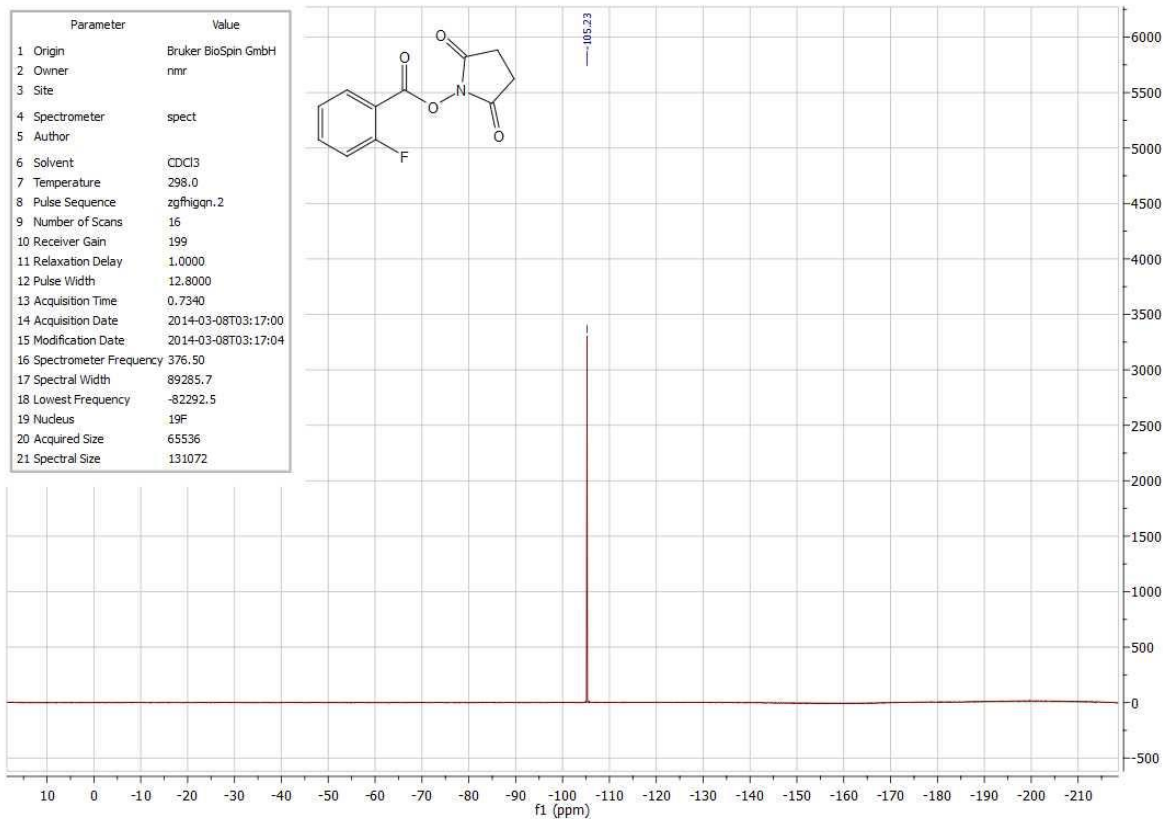


(16a) 2,5-dioxopyrrolidin-1-yl 6-methoxy-2-naphthoate

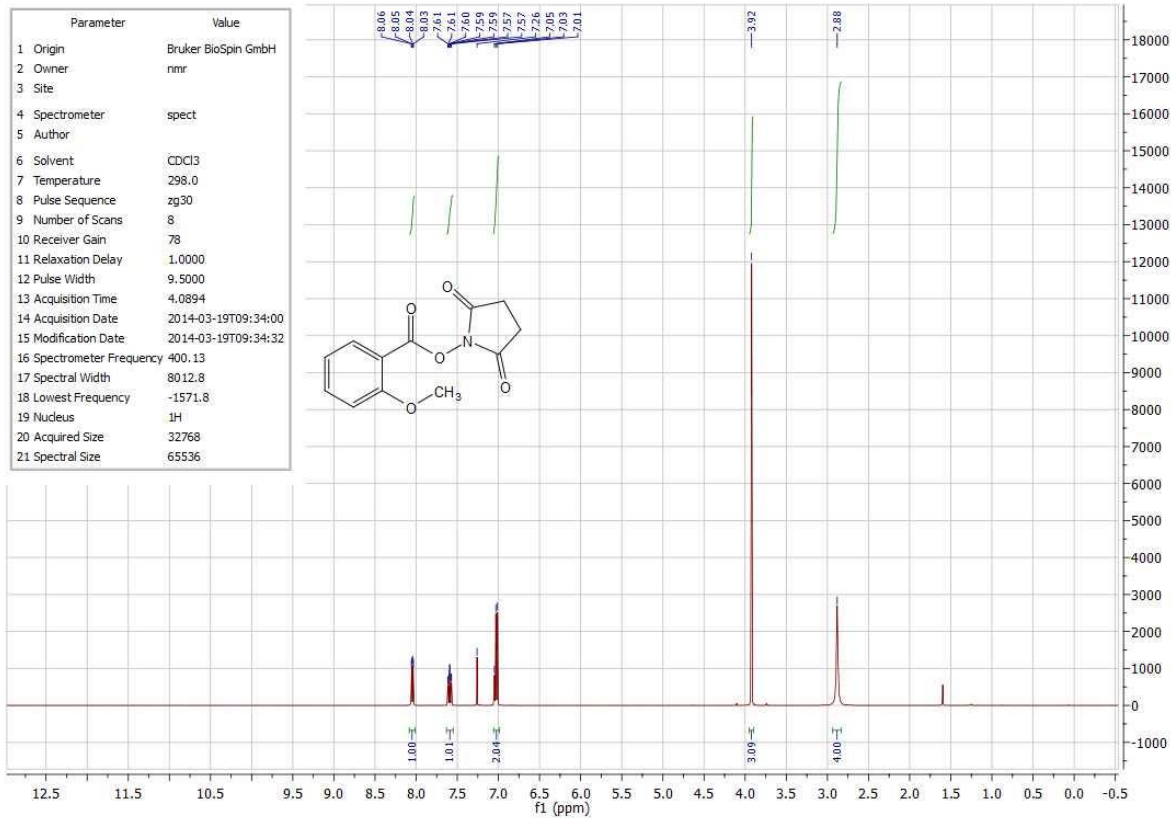


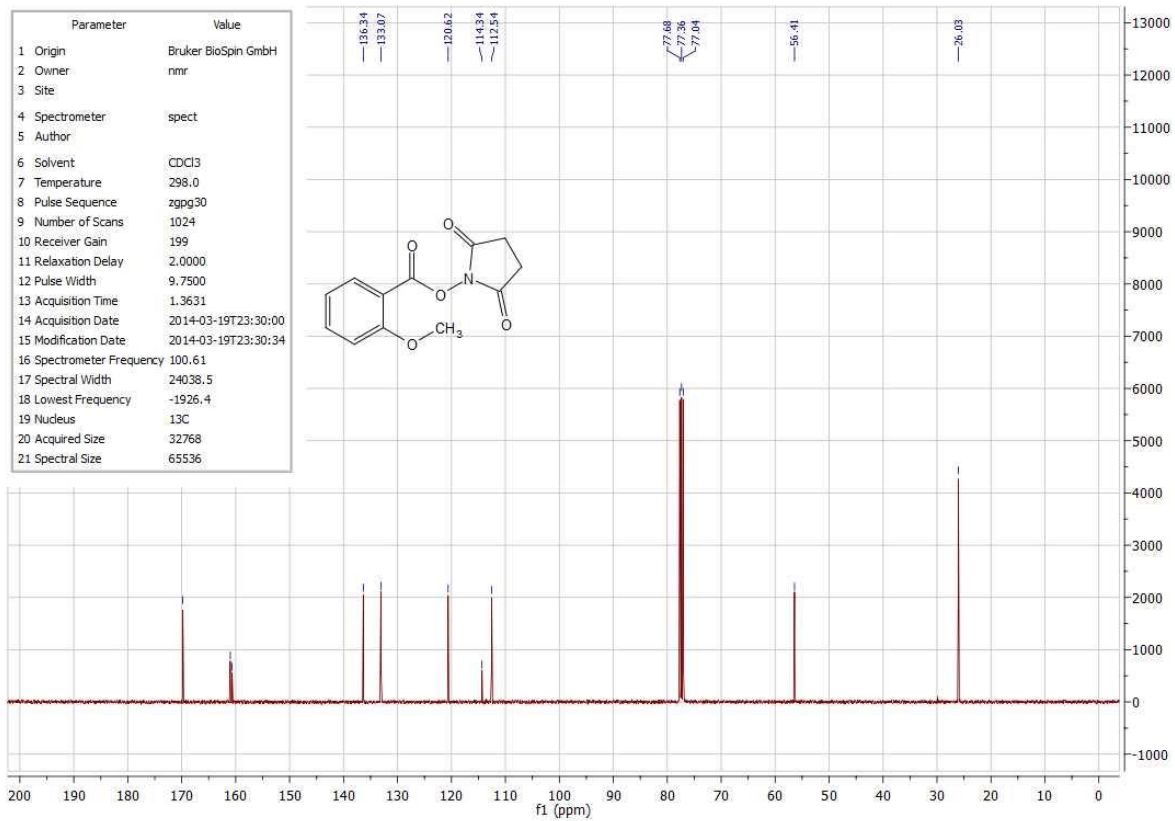
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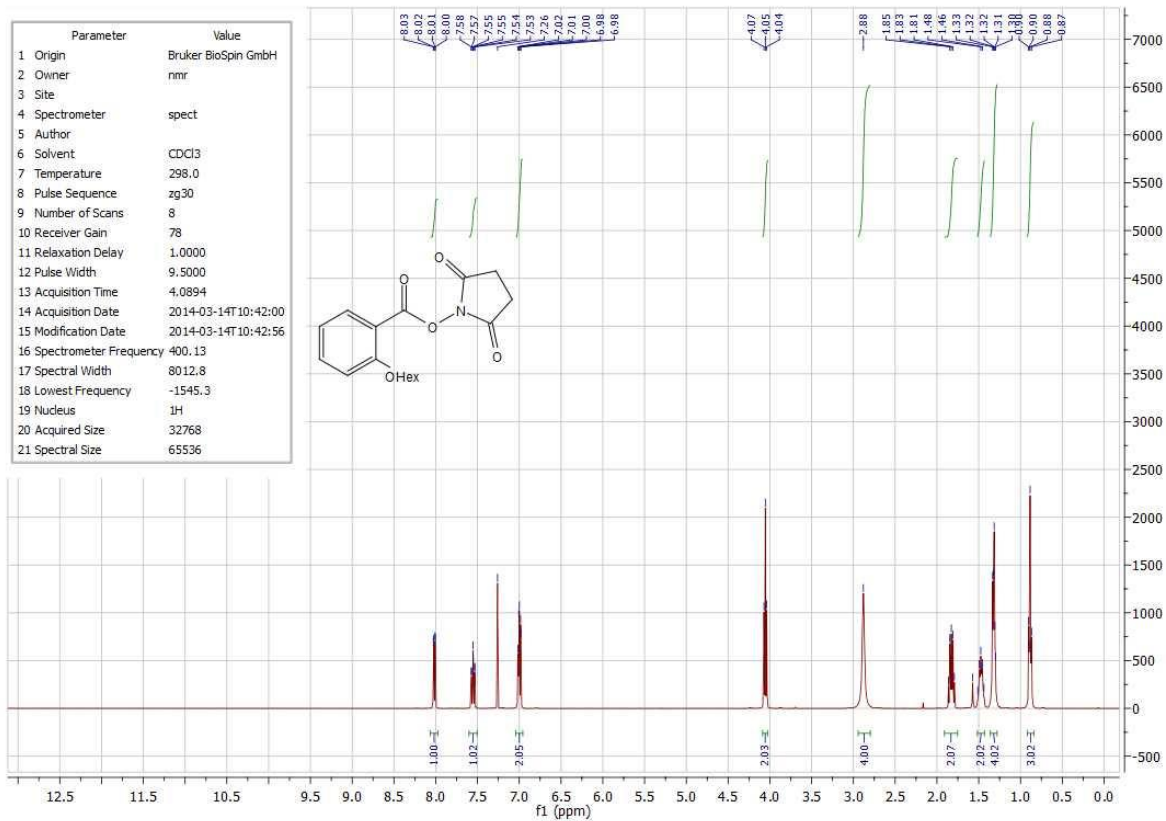


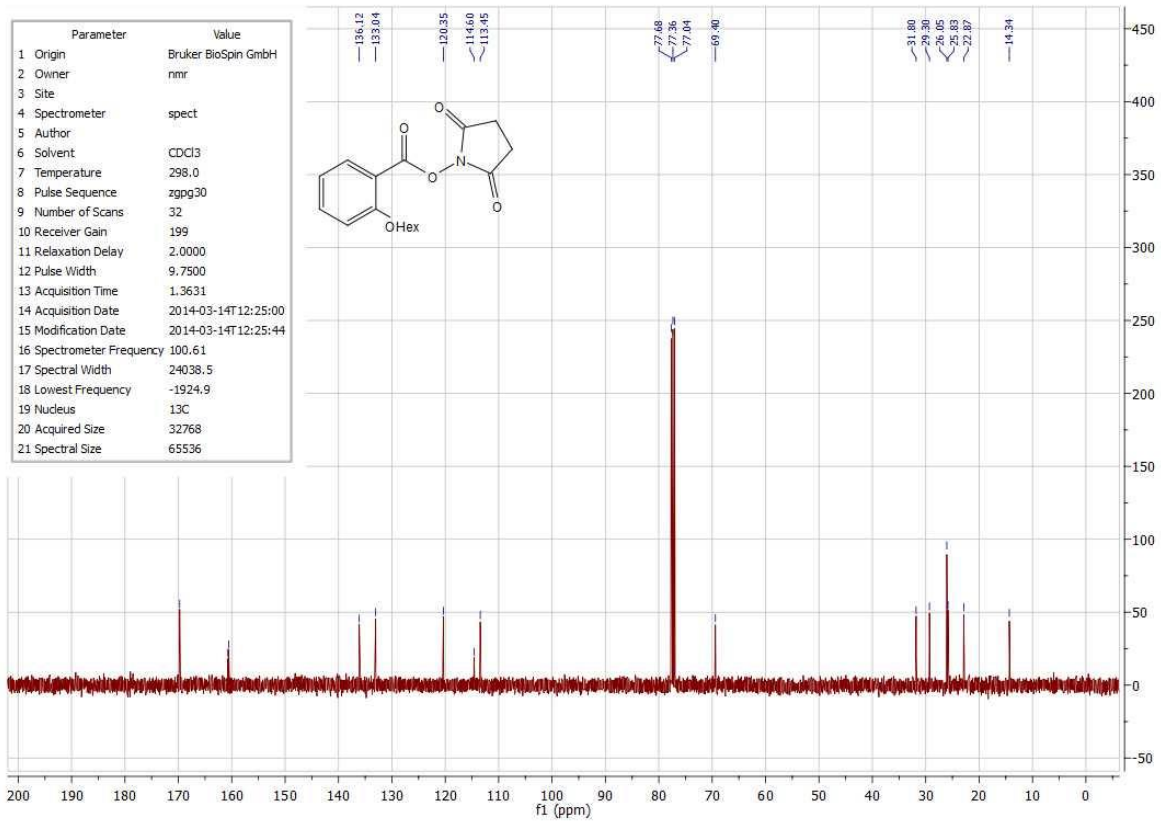
(18a) 2,5-dioxopyrrolidin-1-yl 2-methoxybenzoate



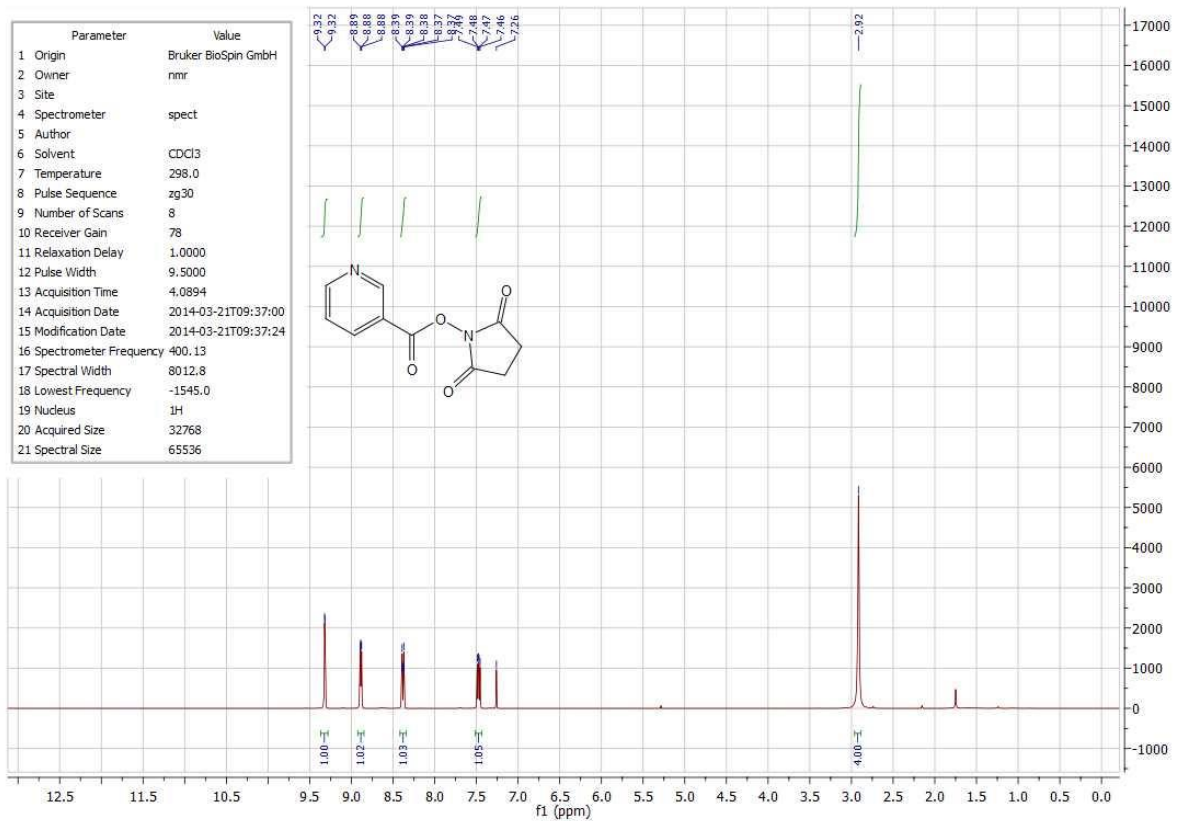


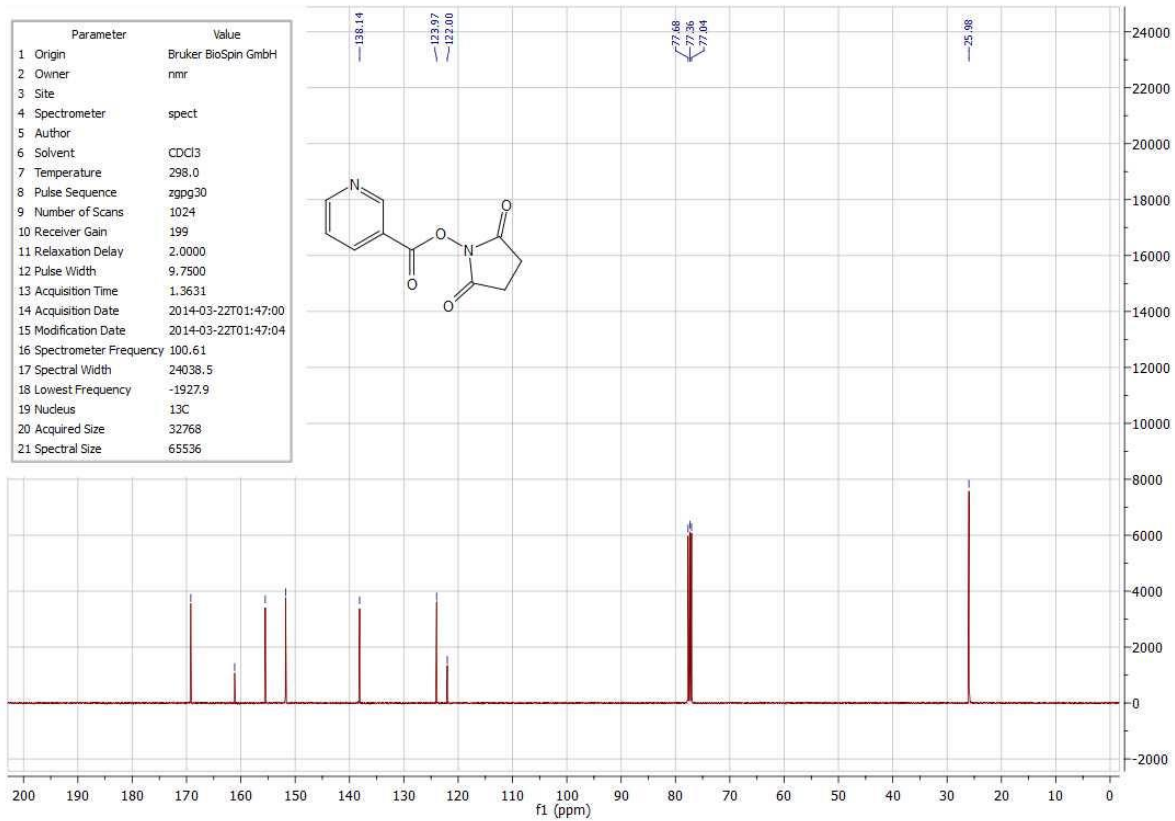
(19a) 2,5-dioxopyrrolidin-1-yl 2-(hexyloxy)benzoate



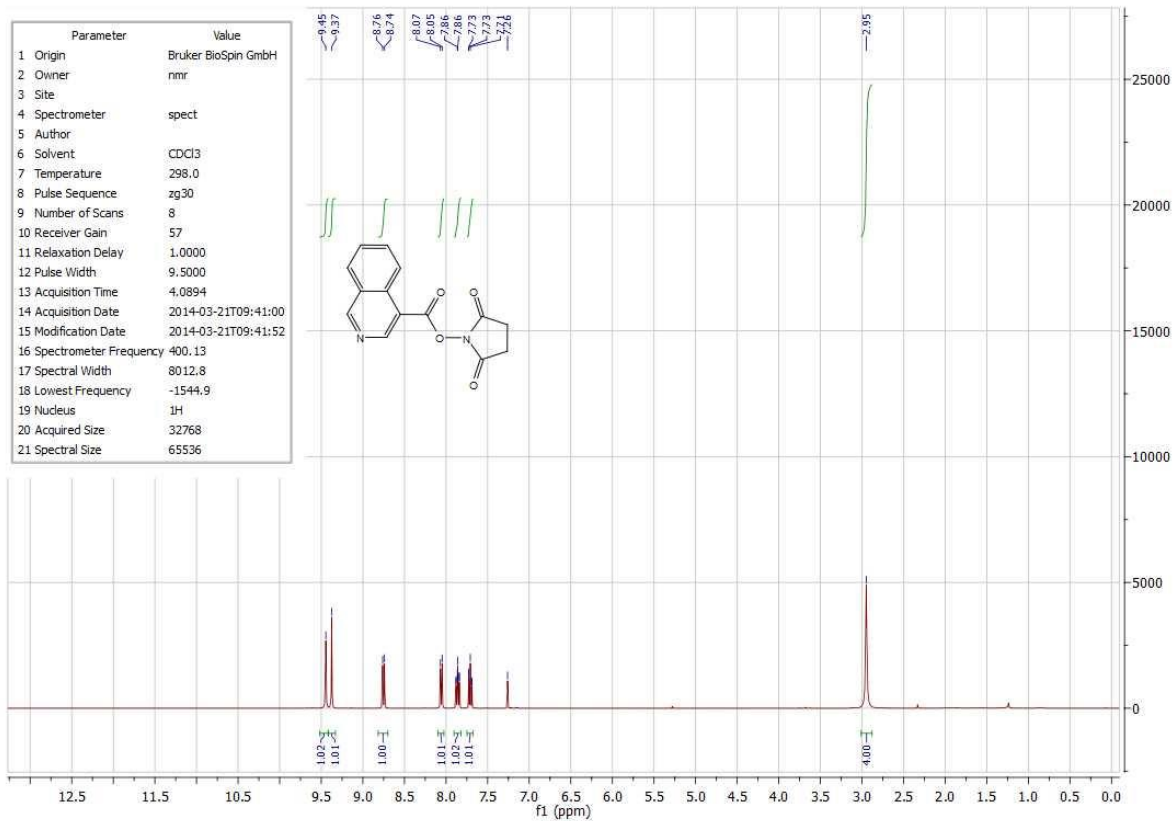


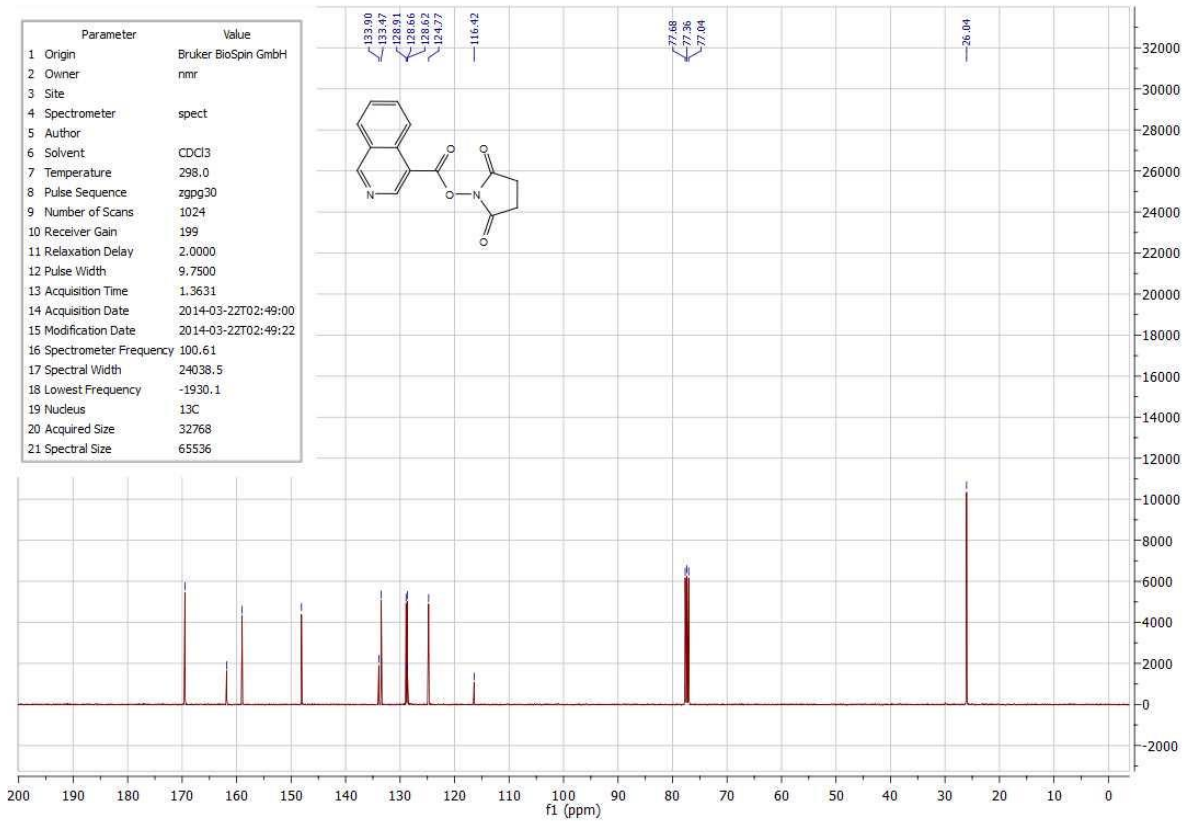
(20a) 2,5-dioxopyrrolidin-1-yl nicotinate



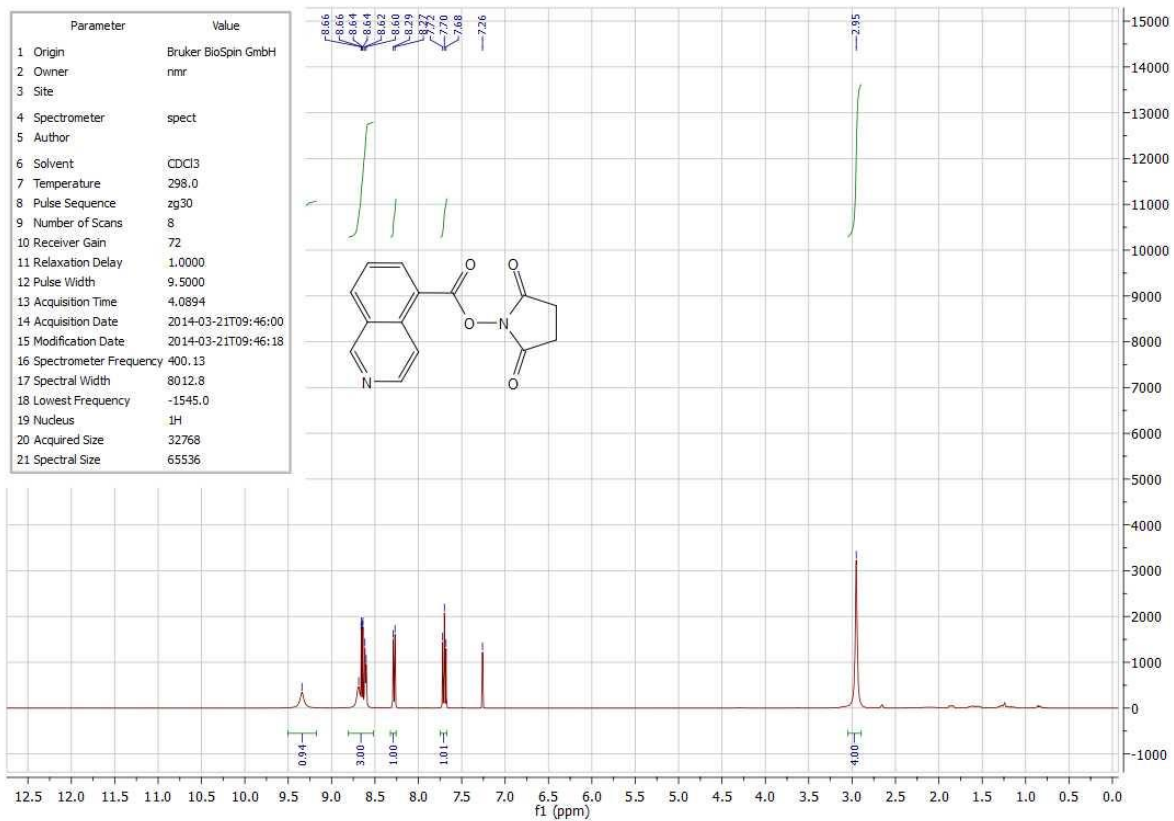


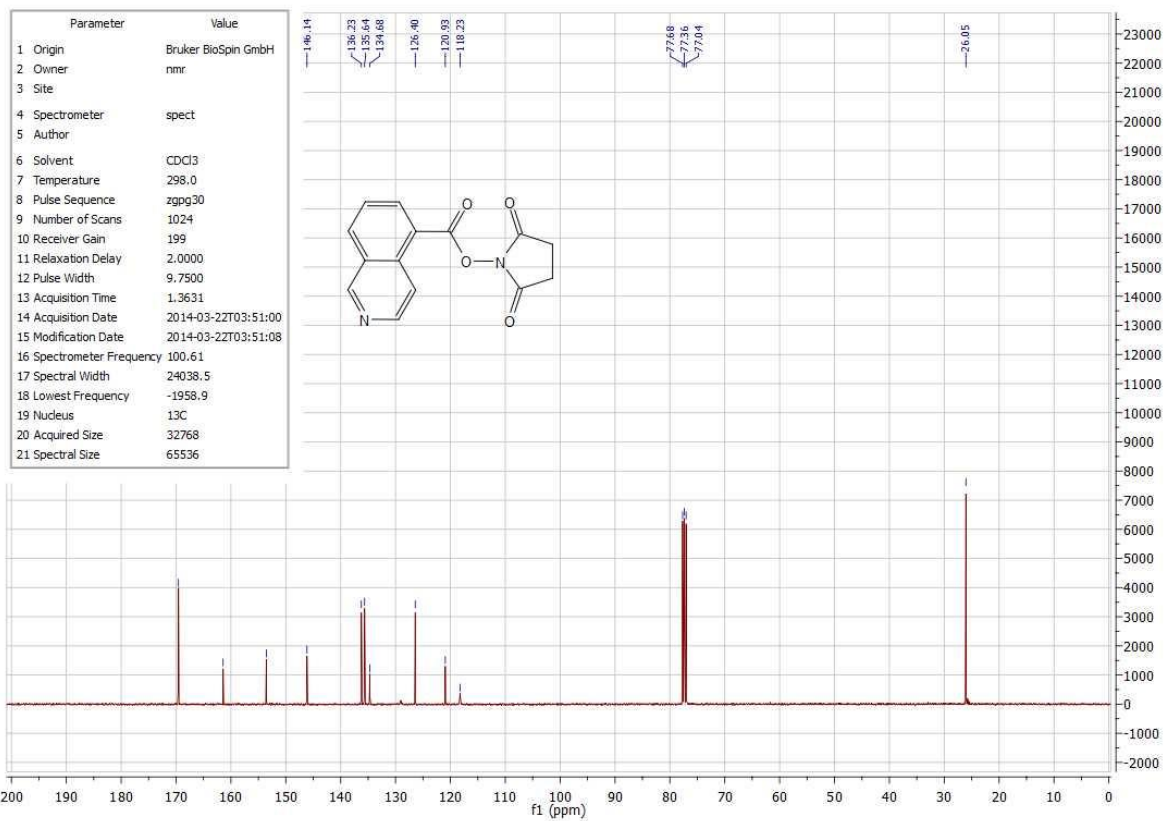
(21a) 2,5-dioxopyrrolidin-1-yl isoquinoline-4-carboxylate



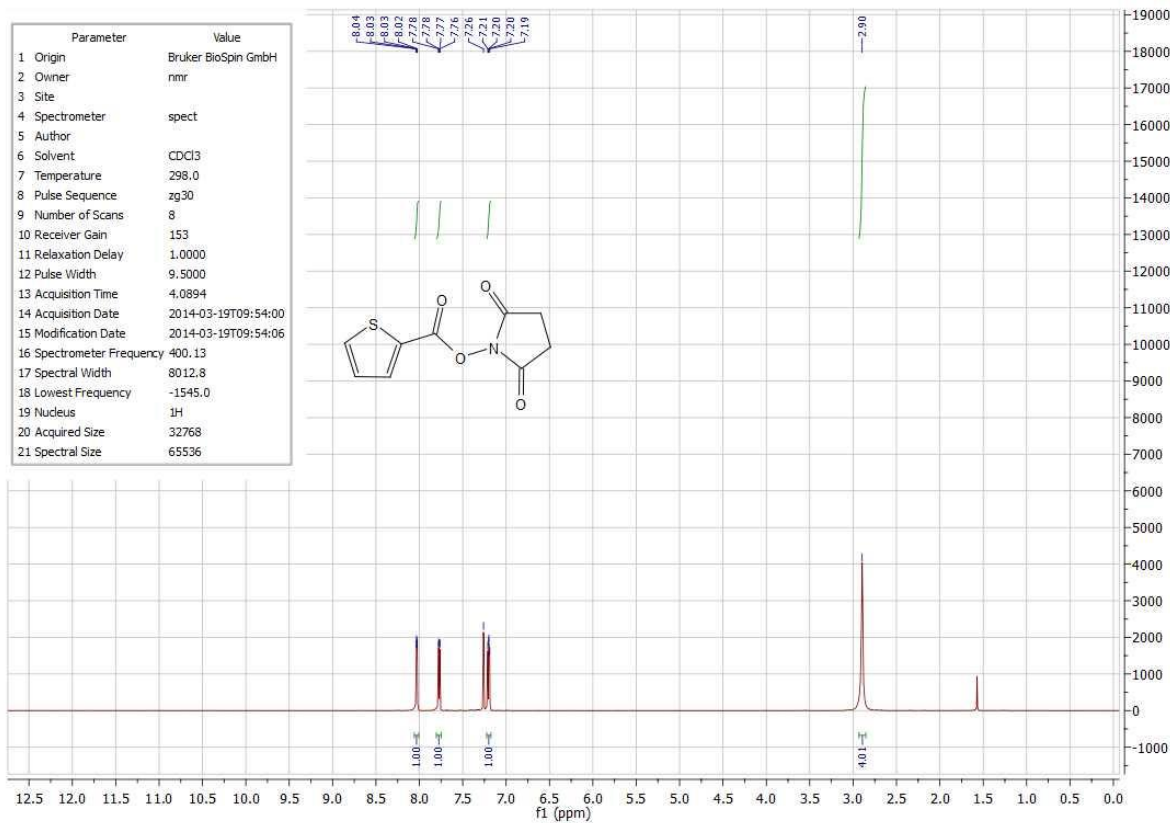


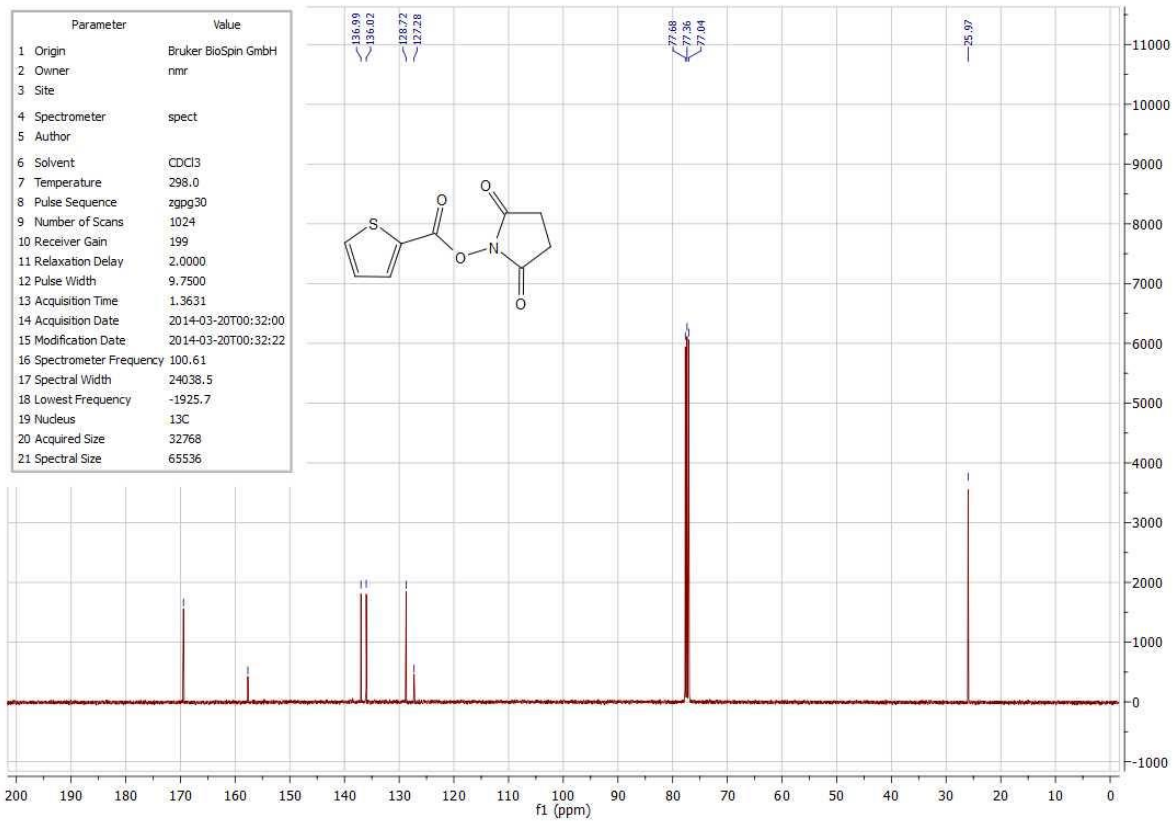
(22a) 2,5-dioxopyrrolidin-1-yl isoquinoline-5-carboxylate



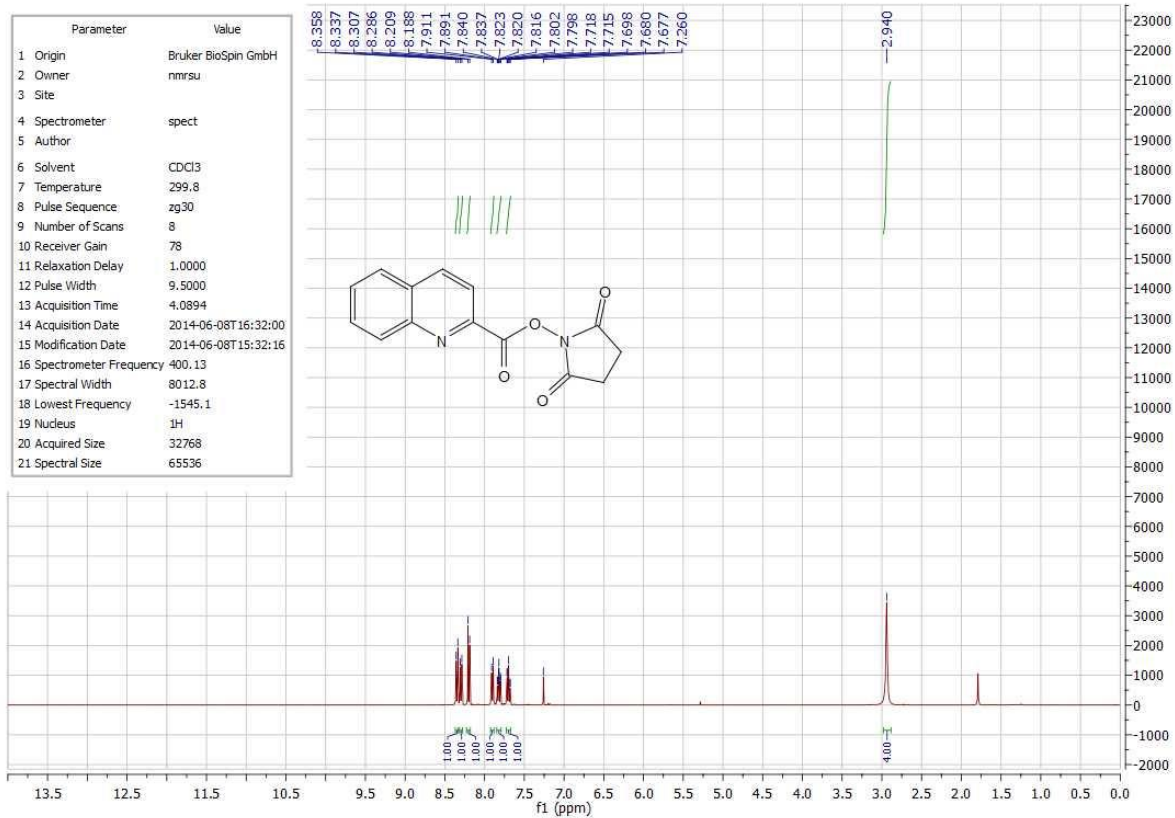


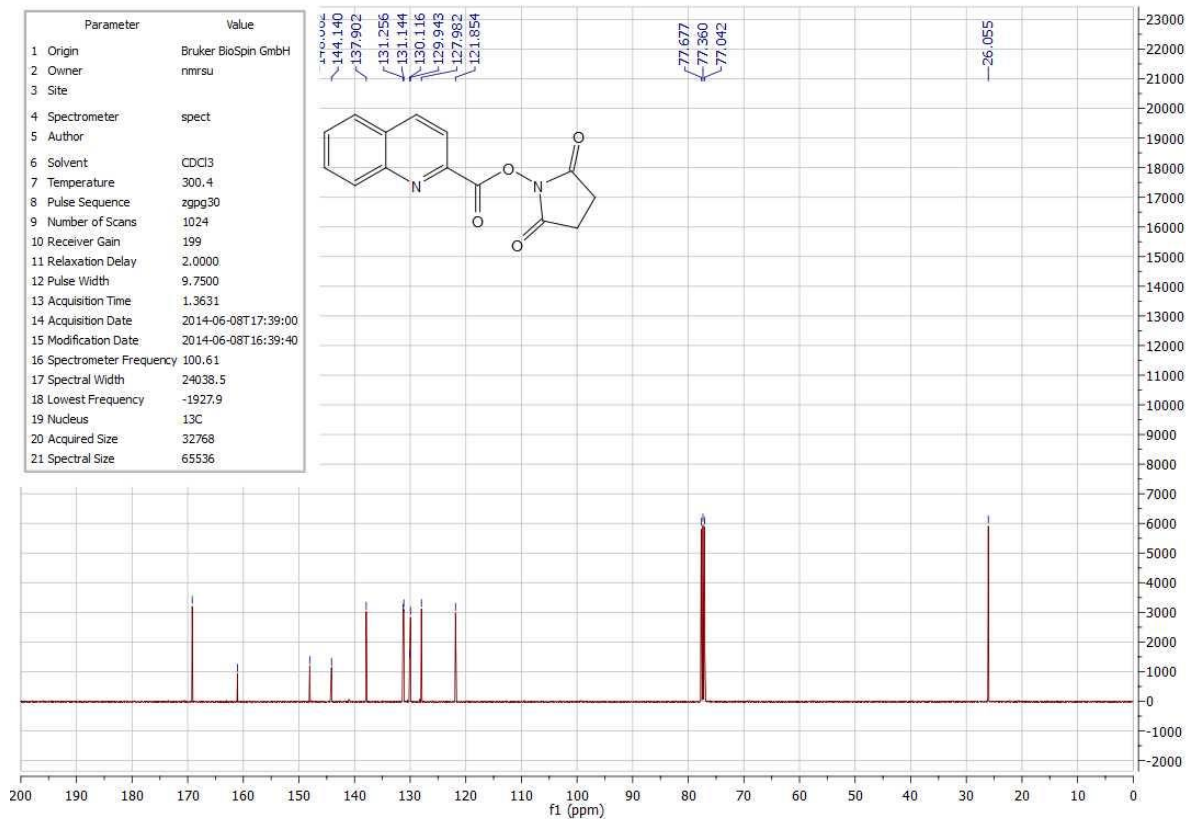
(23a) 2,5-dioxopyrrolidin-1-yl thiophene-2-carboxylate



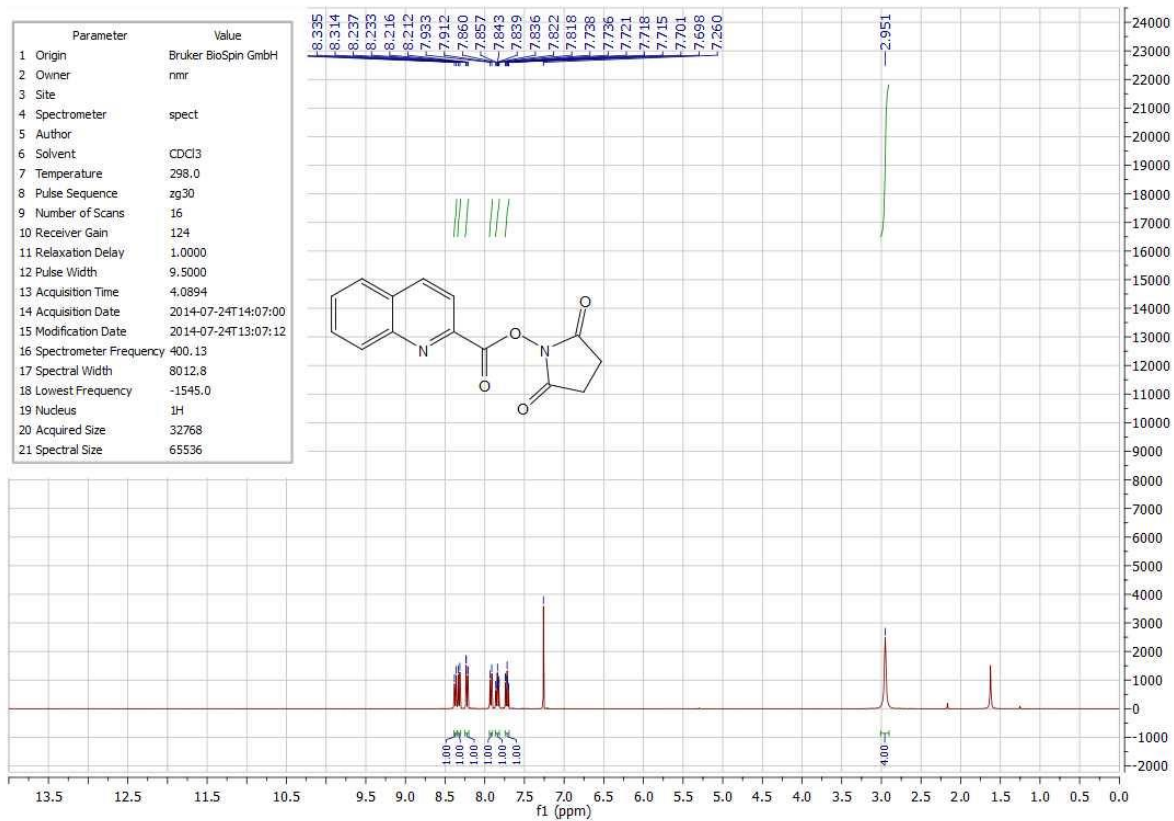


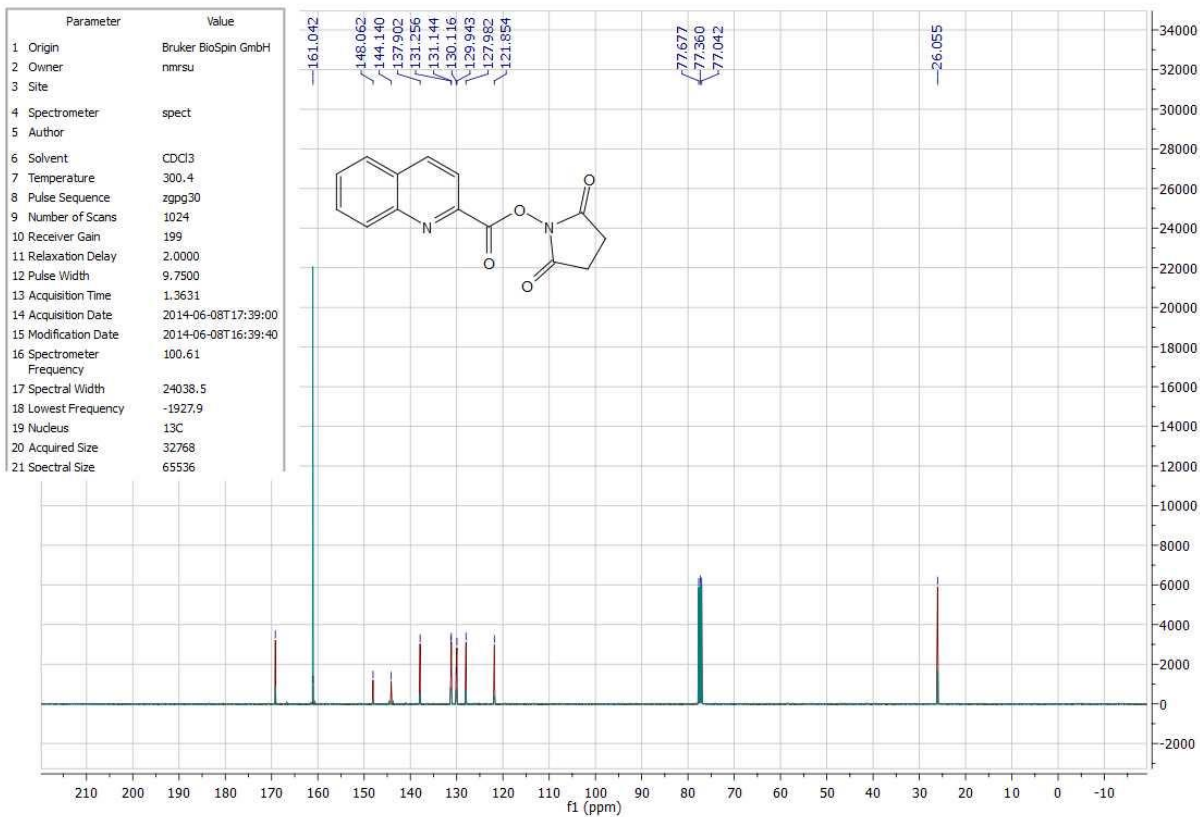
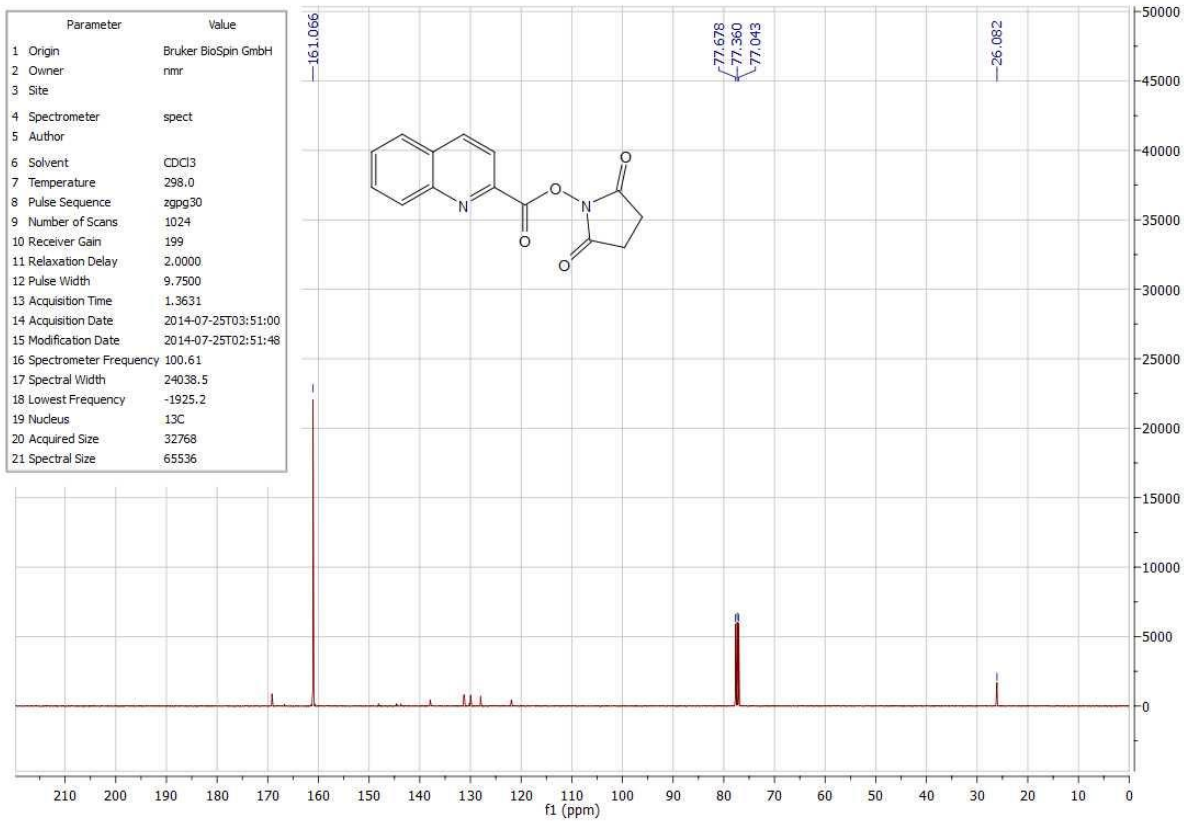
(24a) 2,5-dioxopyrrolidin-1-yl quinoline-2-carboxylate



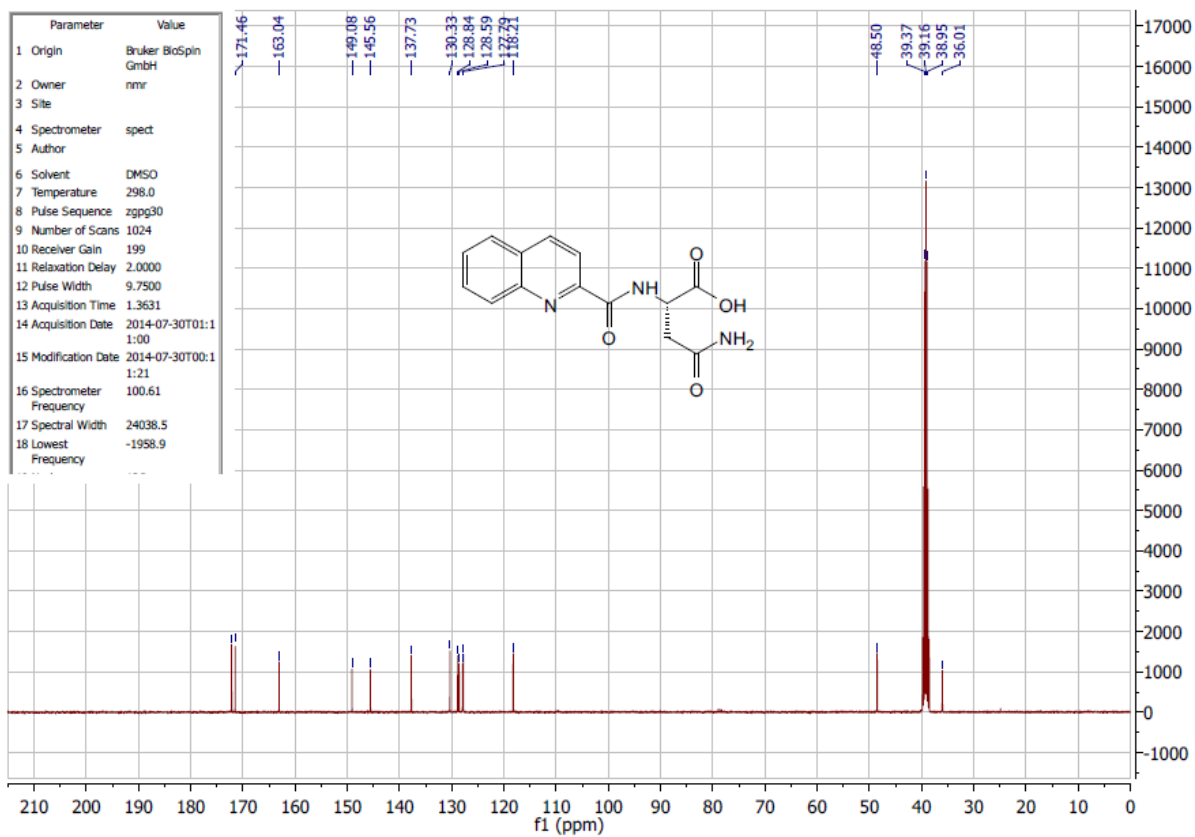
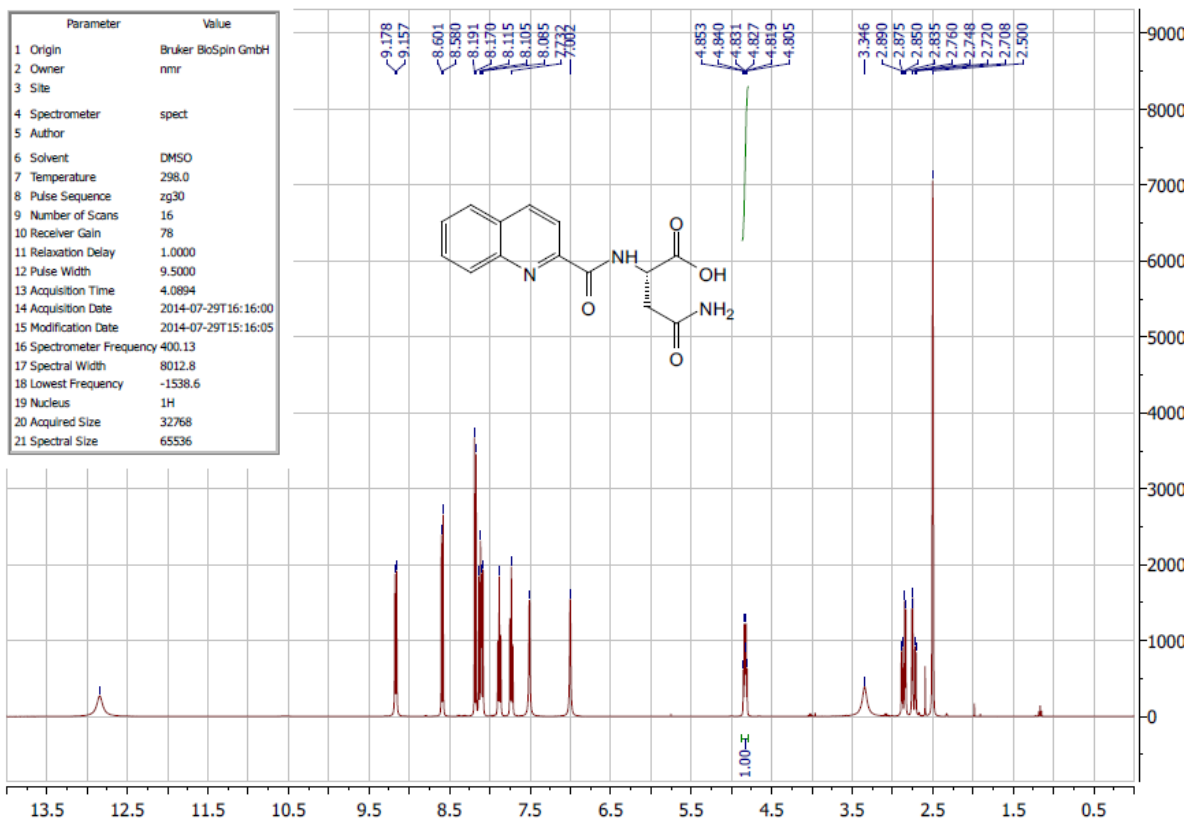


(24a) [C¹³]-2,5-dioxopyrrolidin-1-yl quinoline-2-carboxylate

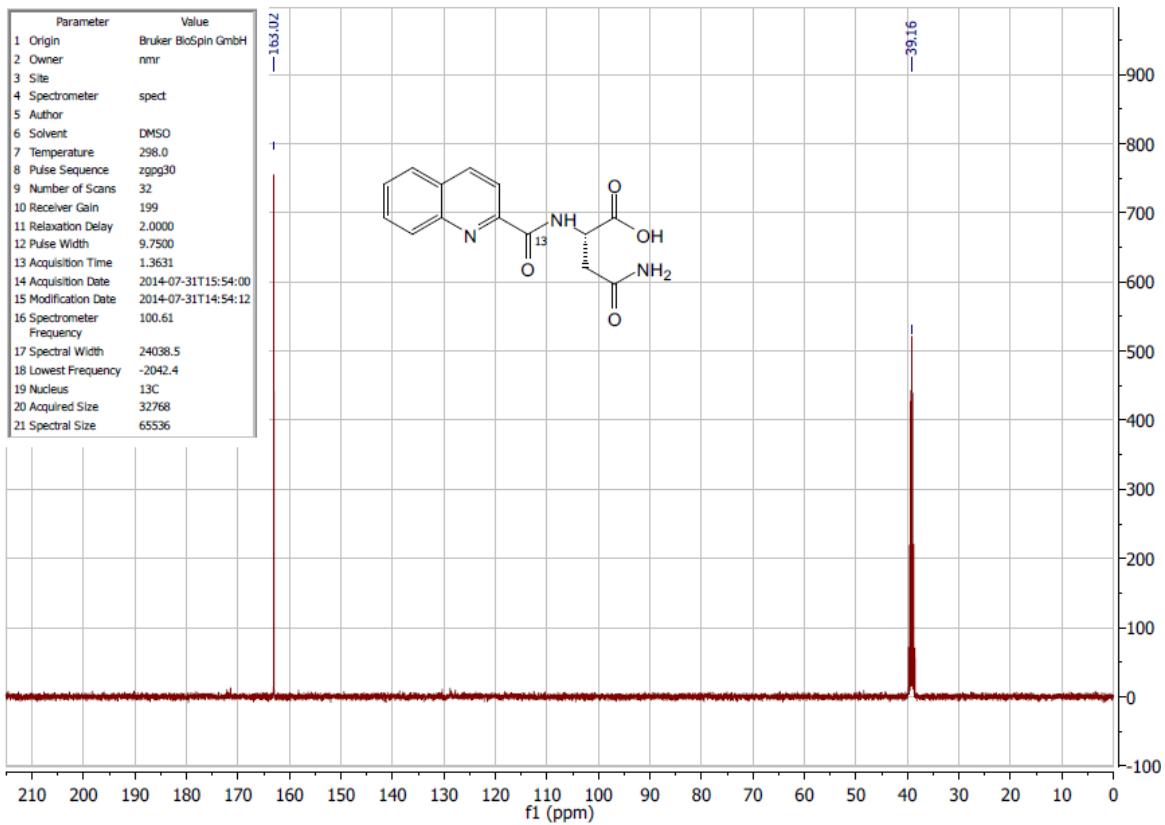
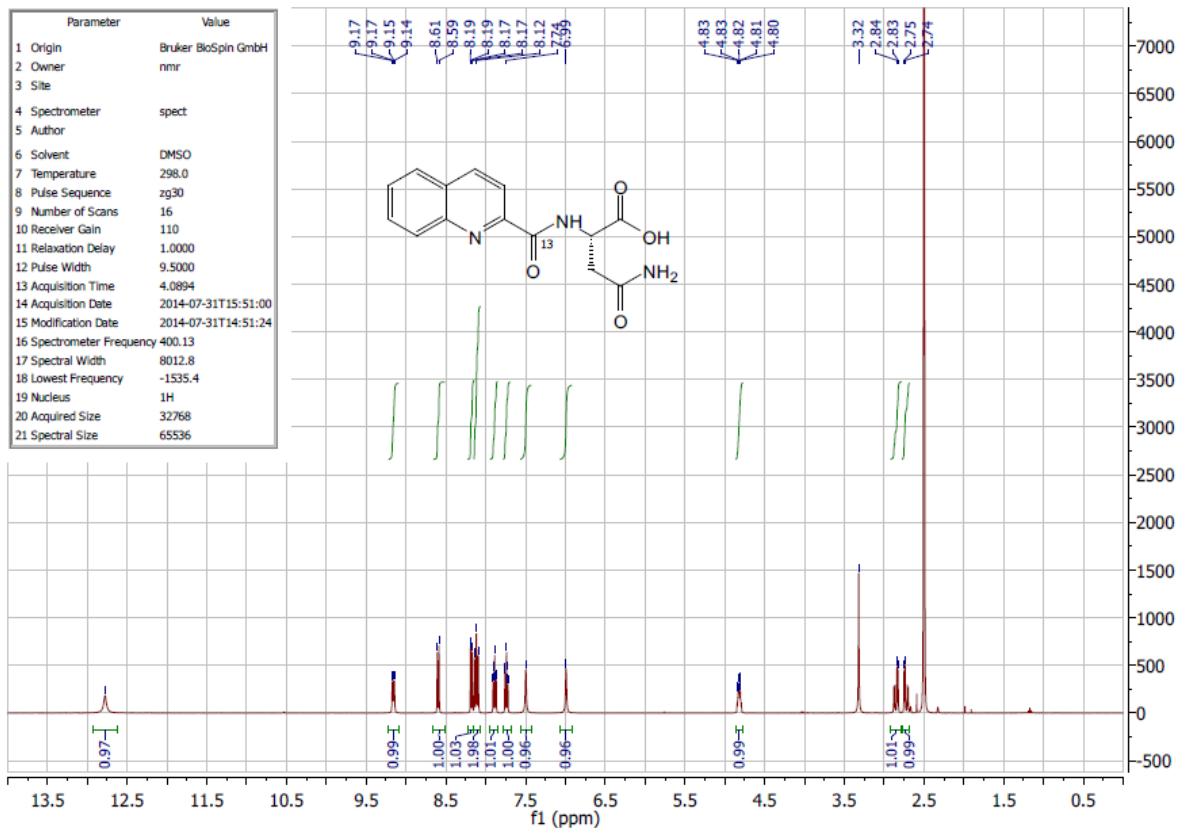


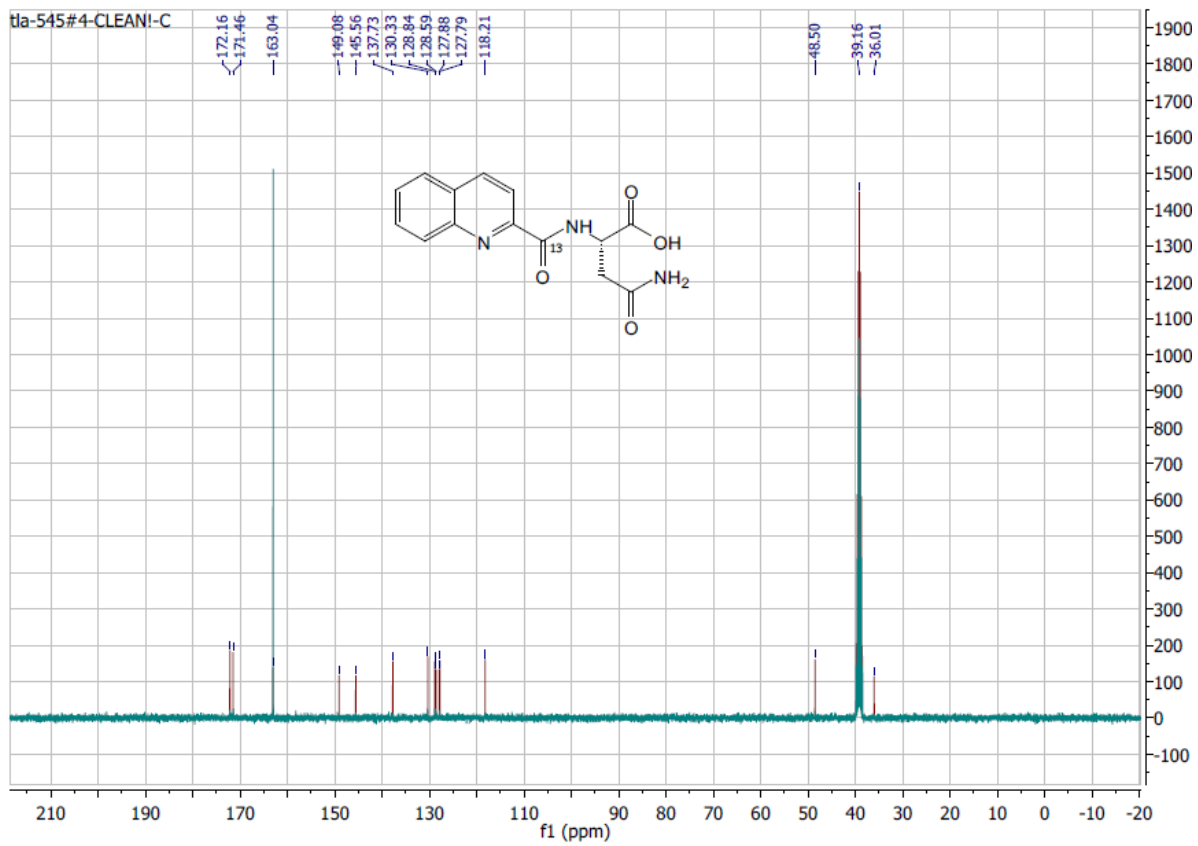


(25) (quinoline-2-carbonyl)-L-asparagine

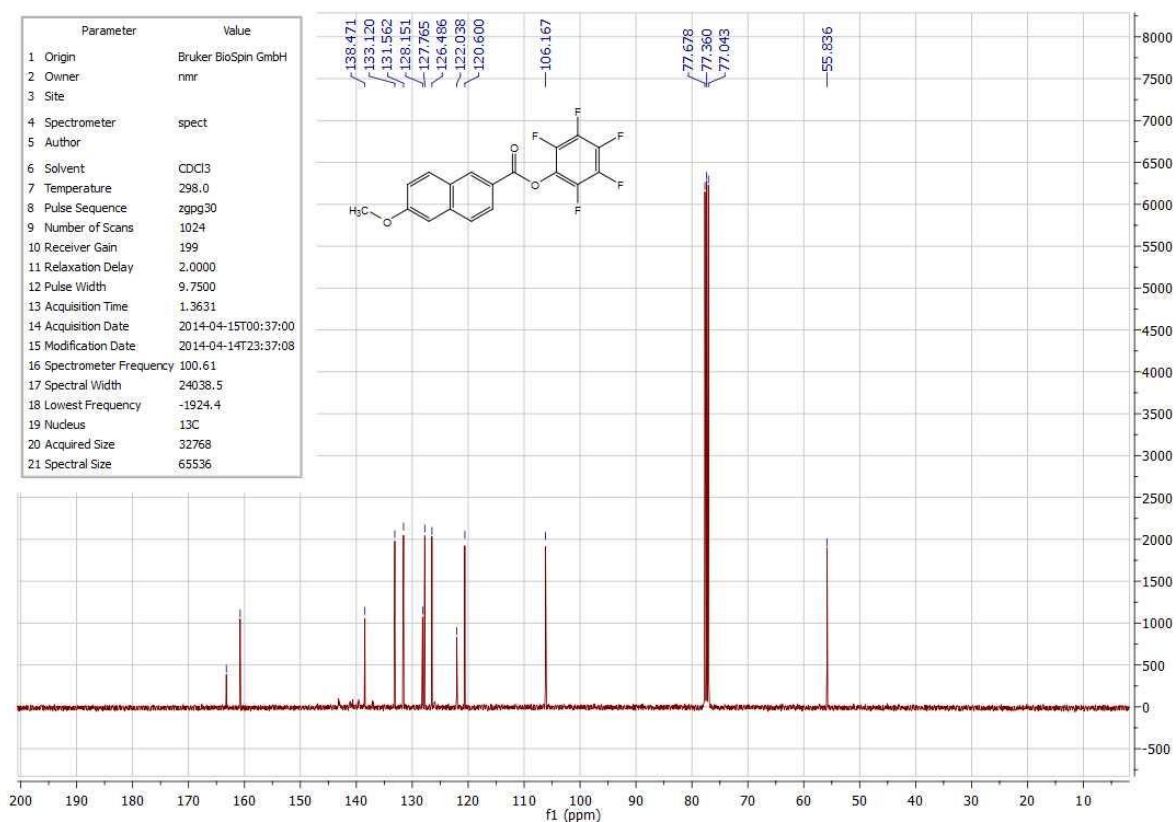
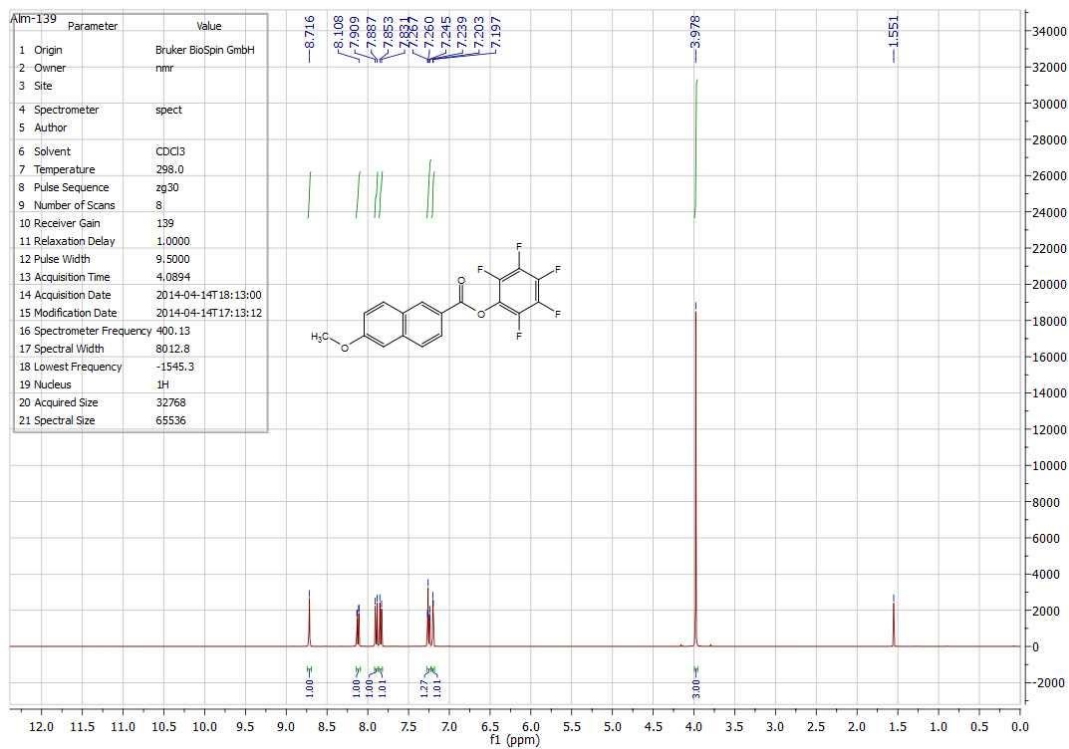


(25) [¹³C]-L-asparagine

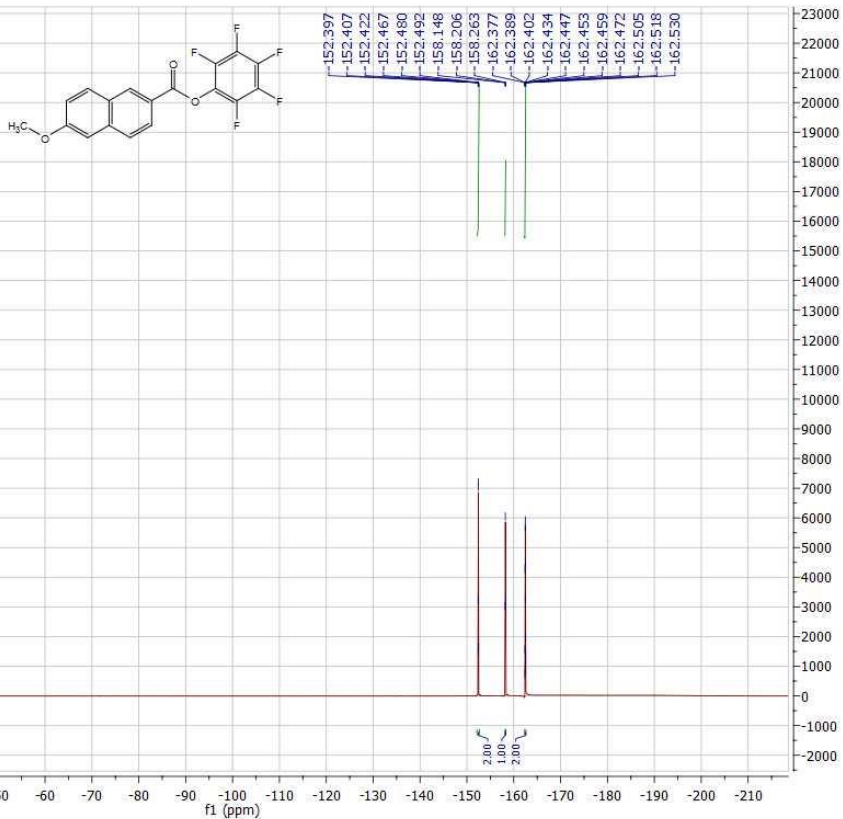




(2b) Perfluorophenyl 6-methoxy-2-naphthoate

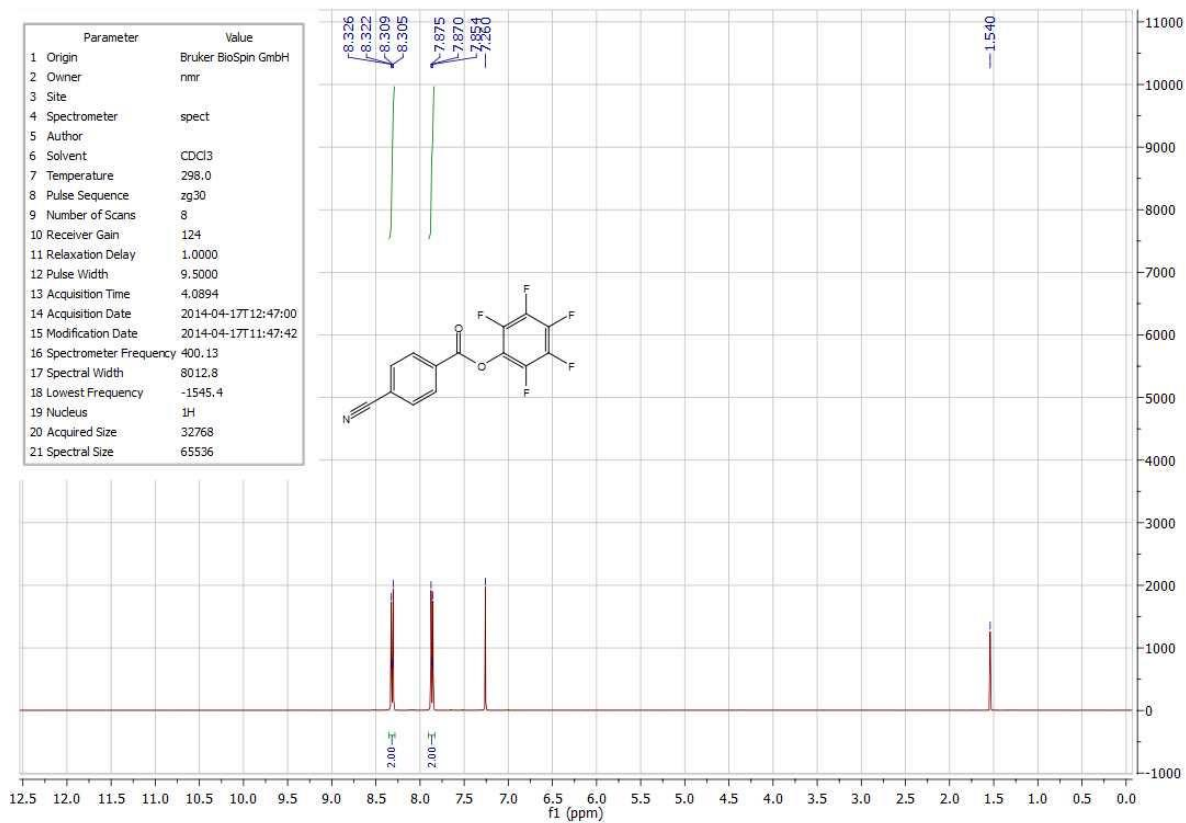


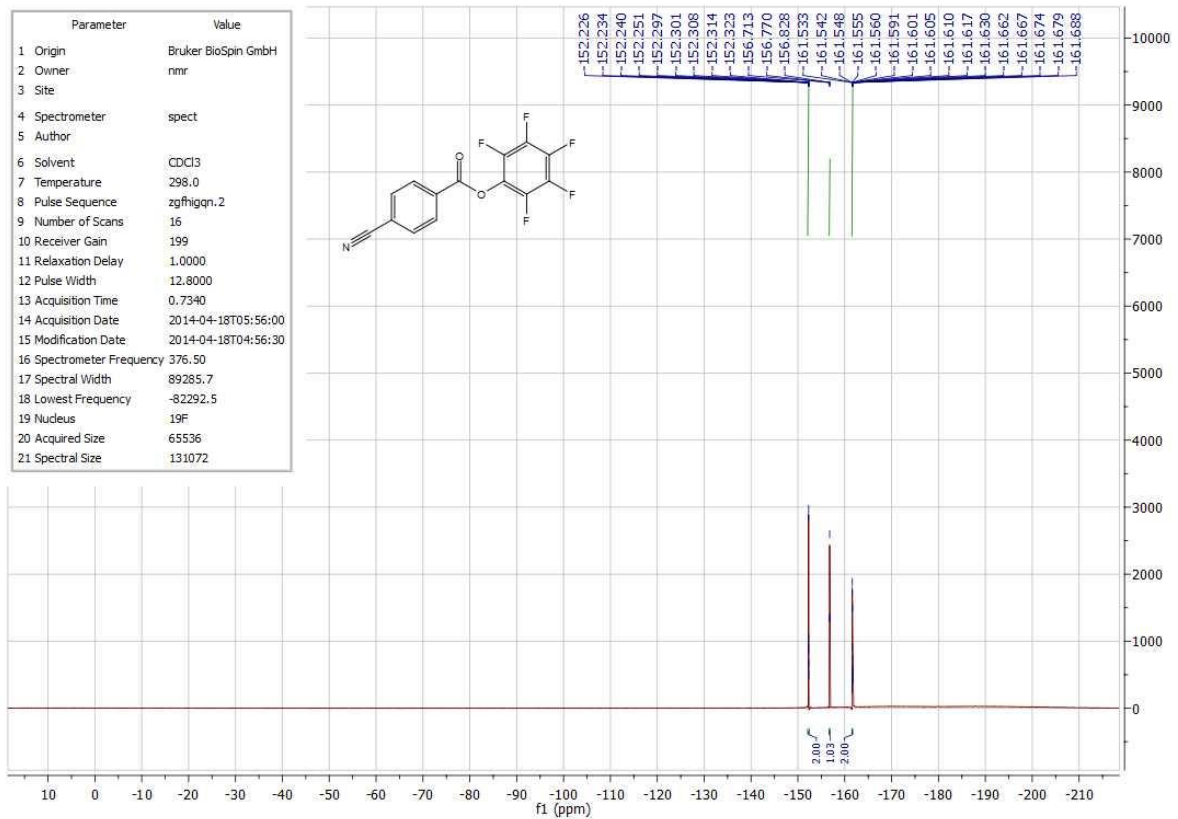
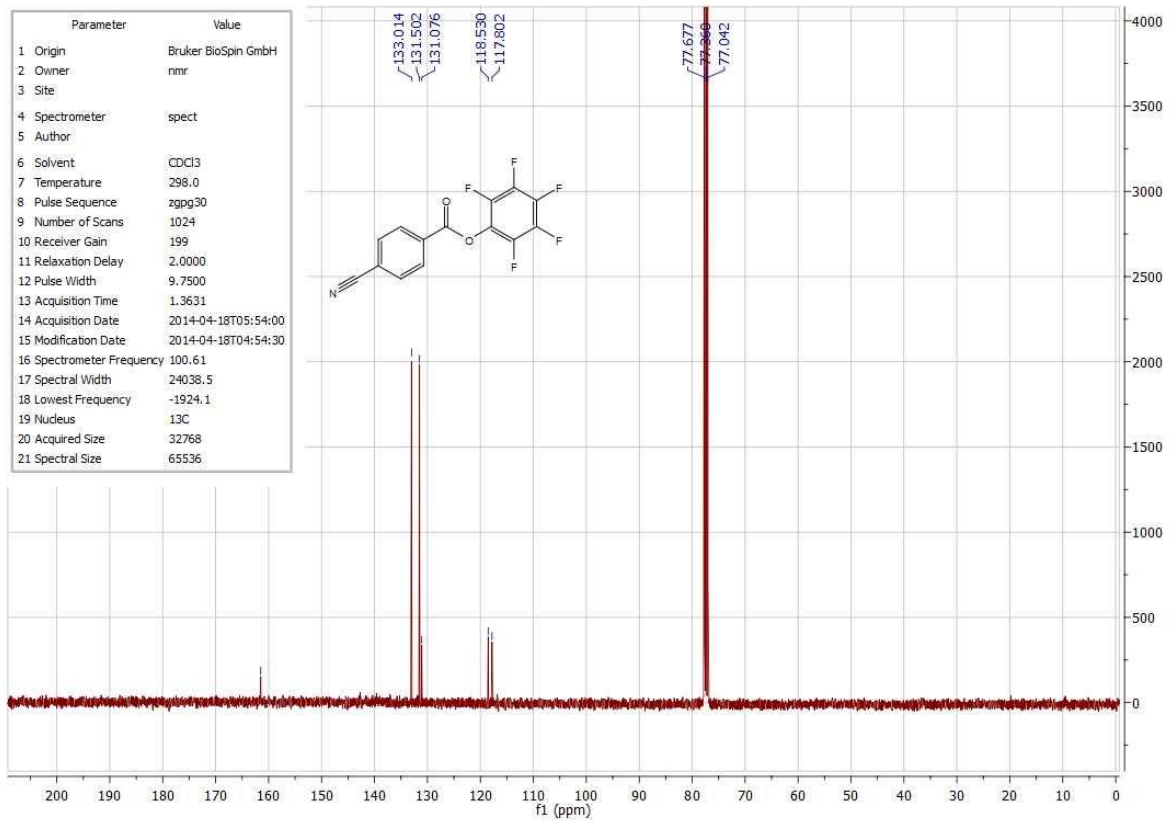
Parameter	Value
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2 Owner	nmr
3 Site	
4 Spectrometer	spect
5 Author	
6 Solvent	CDCl3
7 Temperature	298.0
8 Pulse Sequence	zgfgqn
9 Number of Scans	16
10 Receiver Gain	199
11 Relaxation Delay	1.0000
12 Pulse Width	12.8000
13 Acquisition Time	0.7340
14 Acquisition Date	2014-04-12T13:54:00
15 Modification Date	2014-04-12T12:54:14
16 Spectrometer Frequency	376.50
17 Spectral Width	89285.7
18 Lowest Frequency	-82292.5
19 Nucleus	19F
20 Acquired Size	65536
21 Spectral Size	131072



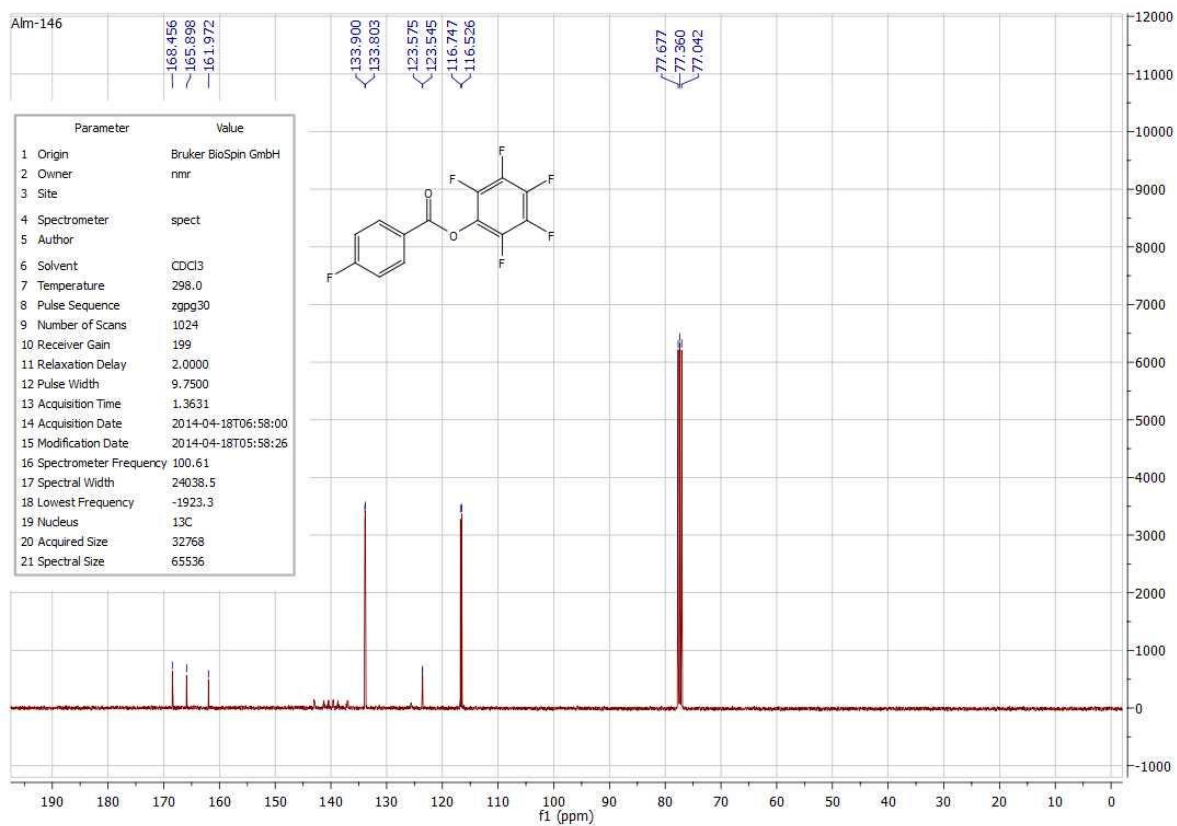
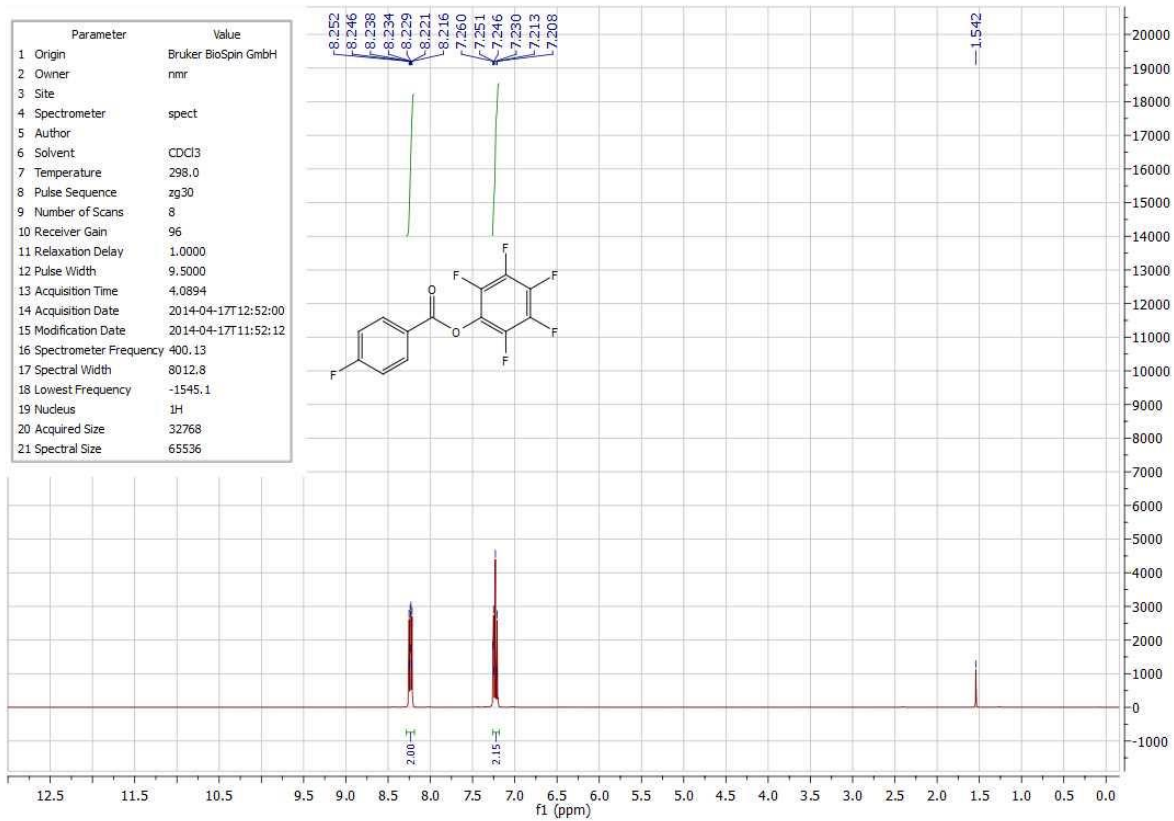
(3b) Perfluorophenyl 4-cyanobenzoate

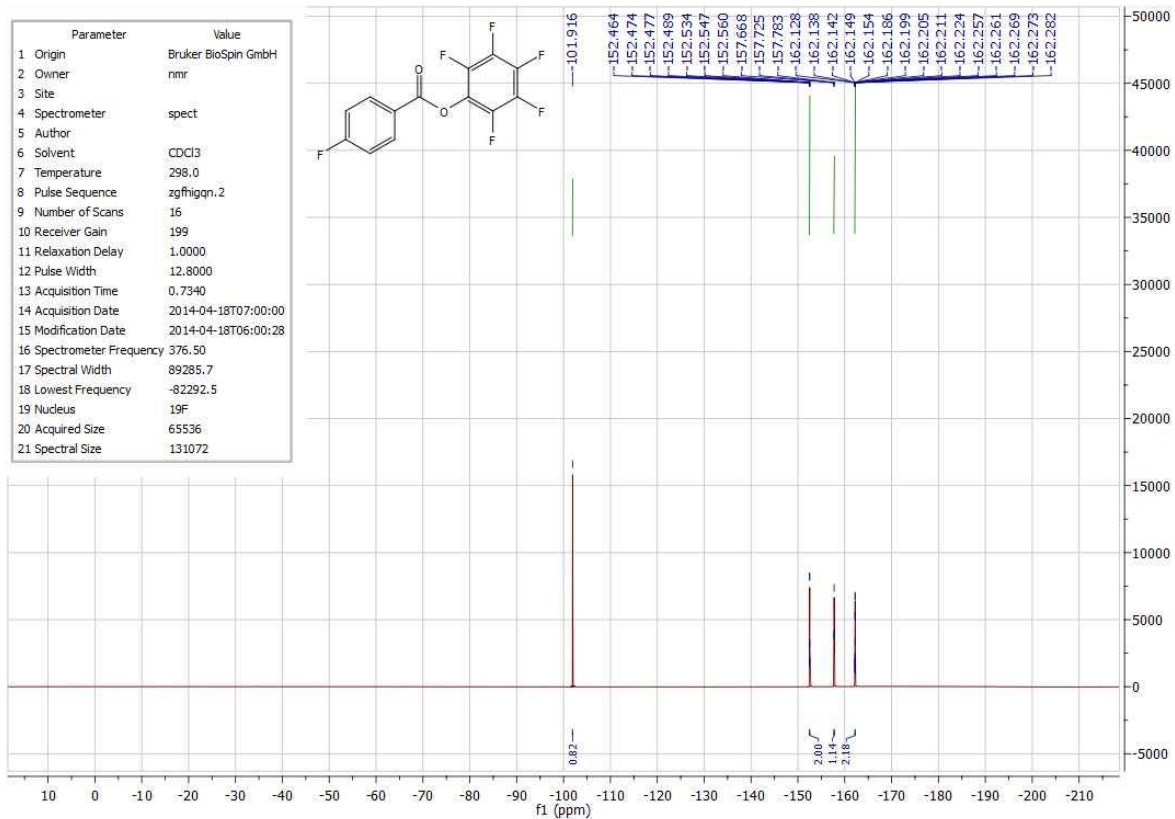
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2 Owner	nmr
3 Site	
4 Spectrometer	spect
5 Author	
6 Solvent	CDCl3
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11 Relaxation Delay	1.0000
12 Pulse Width	9.5000
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15 Modification Date	2014-04-17T11:47:42
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17 Spectral Width	8012.8
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19 Nucleus	1H
20 Acquired Size	32768
21 Spectral Size	65536



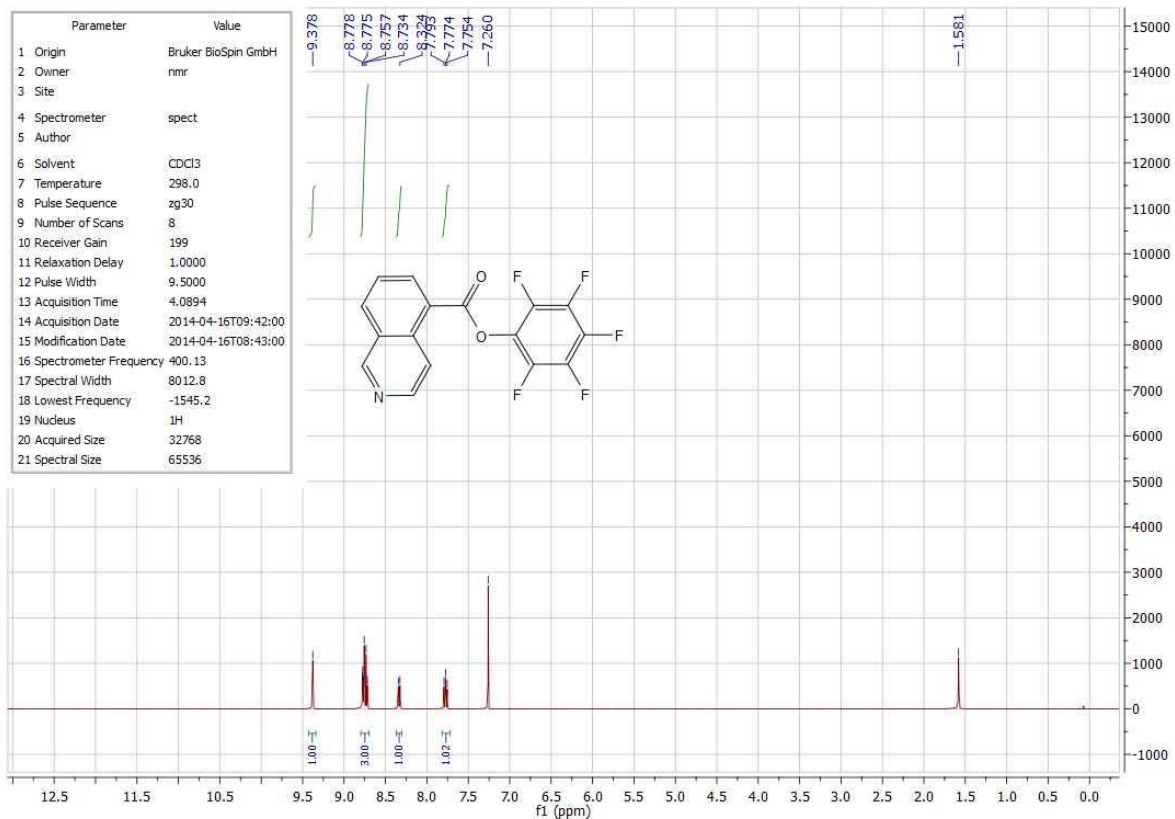


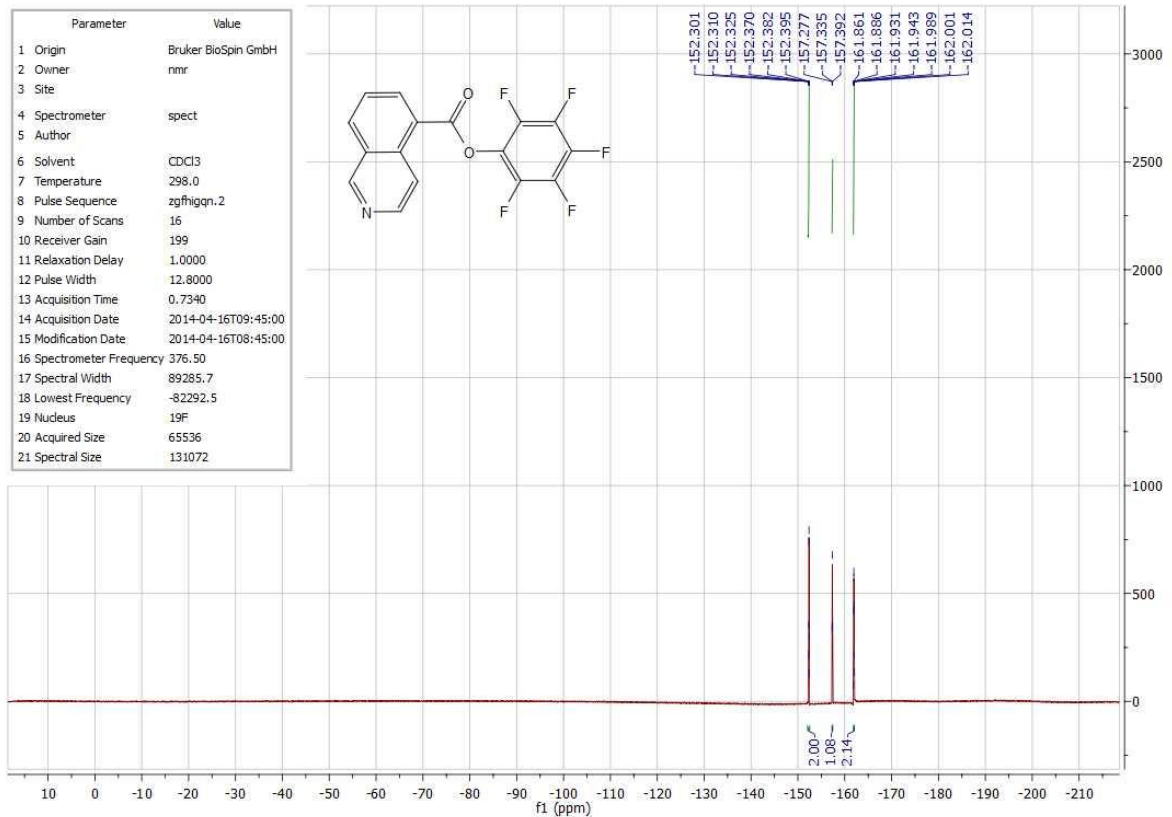
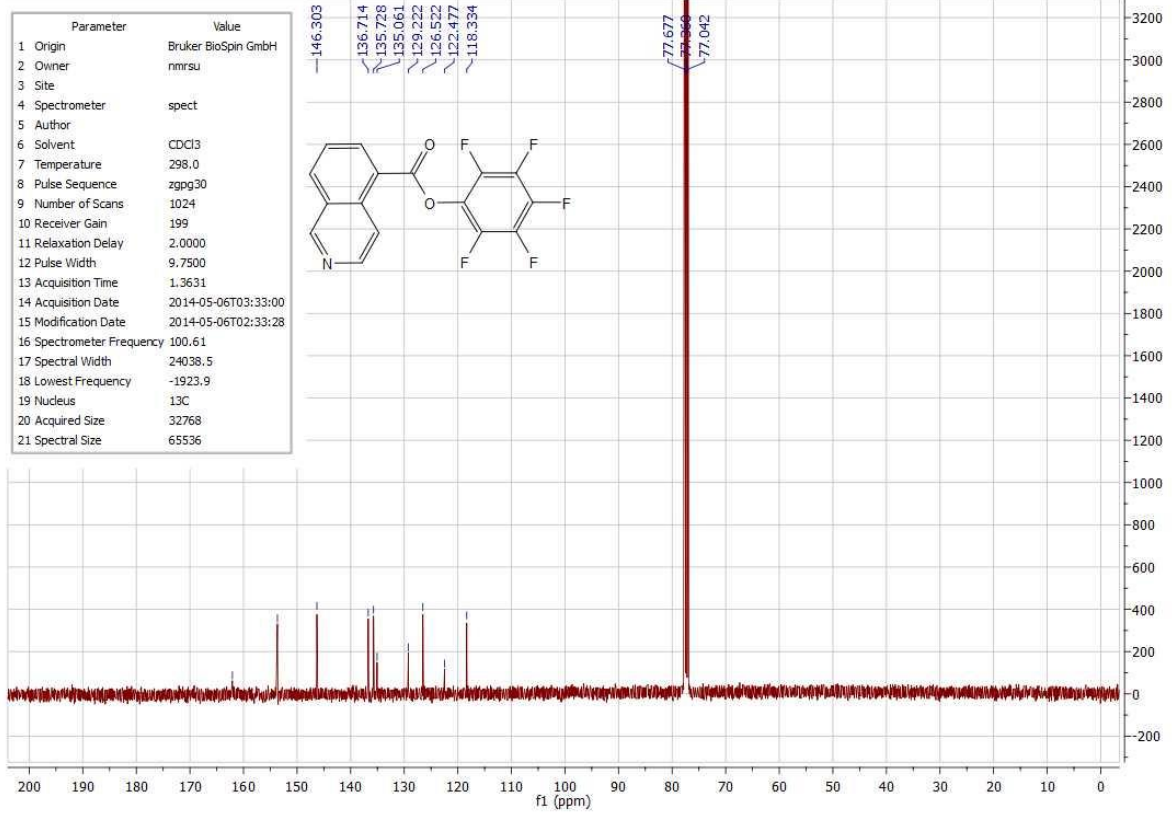
(4b) Perfluorophenyl 4-fluorobenzoate



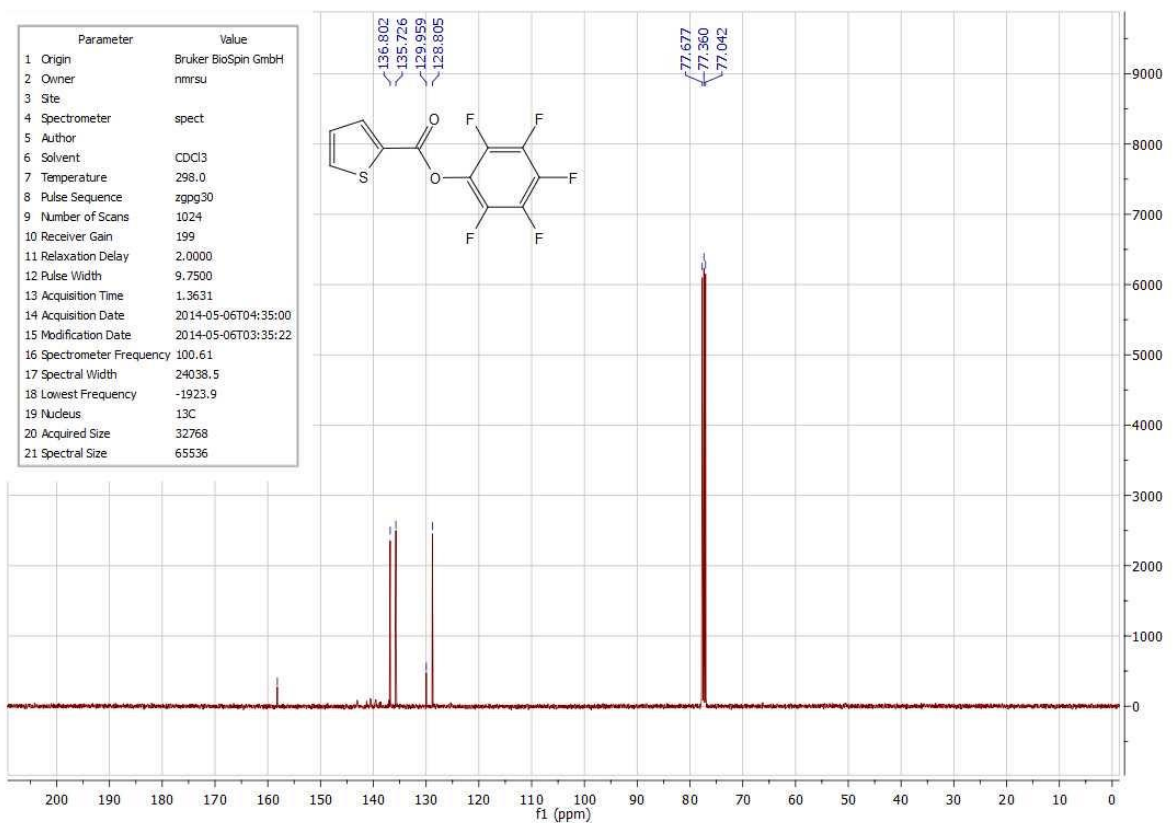
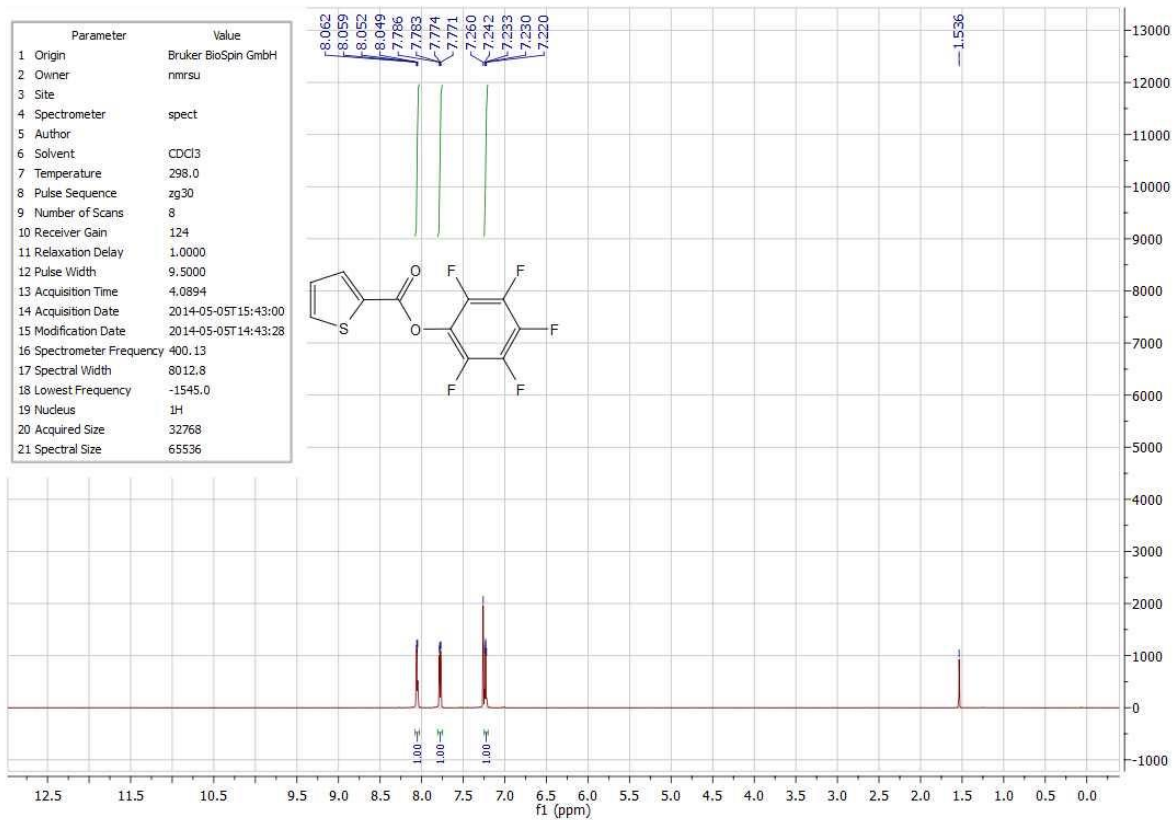


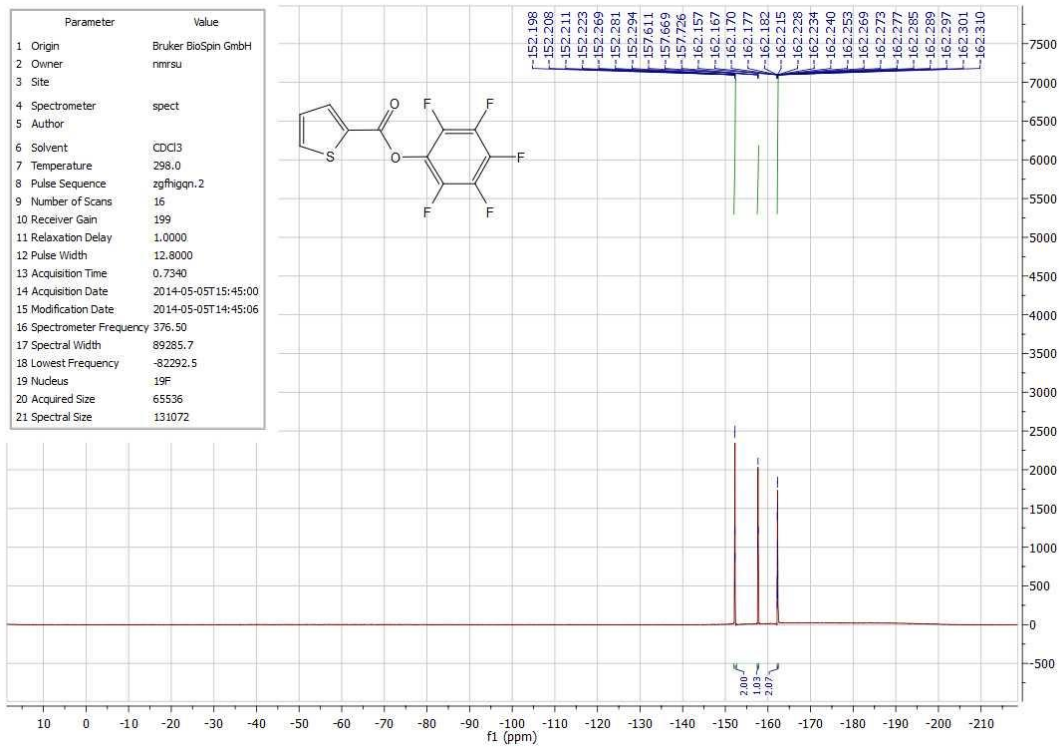
(5b) Perfluorophenyl isoquinoline-5-carboxylate



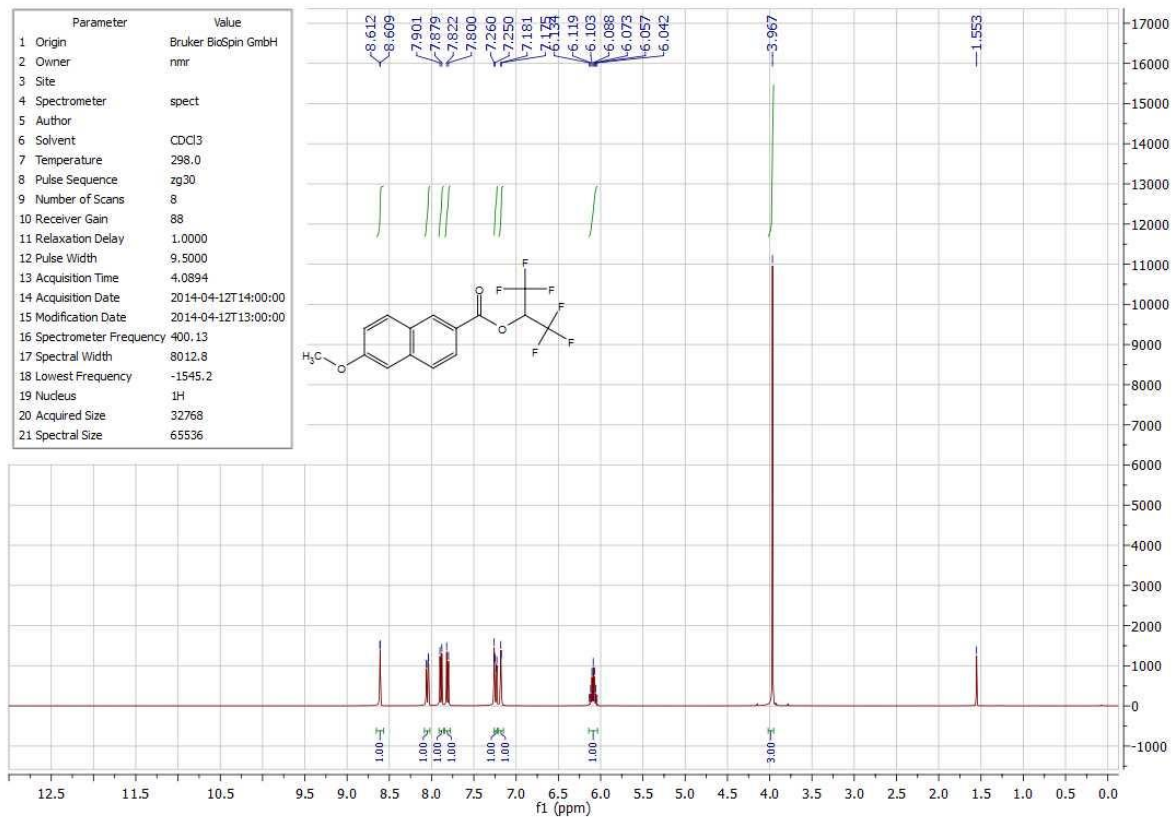


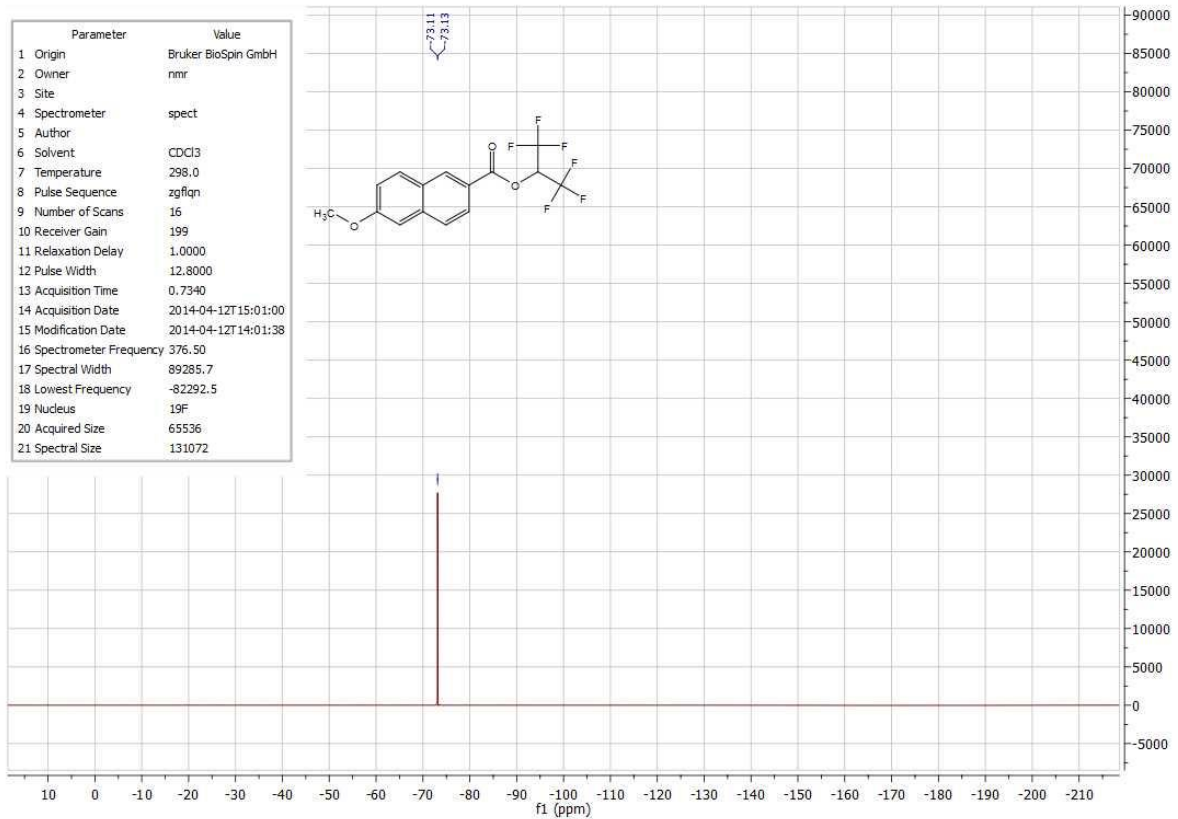
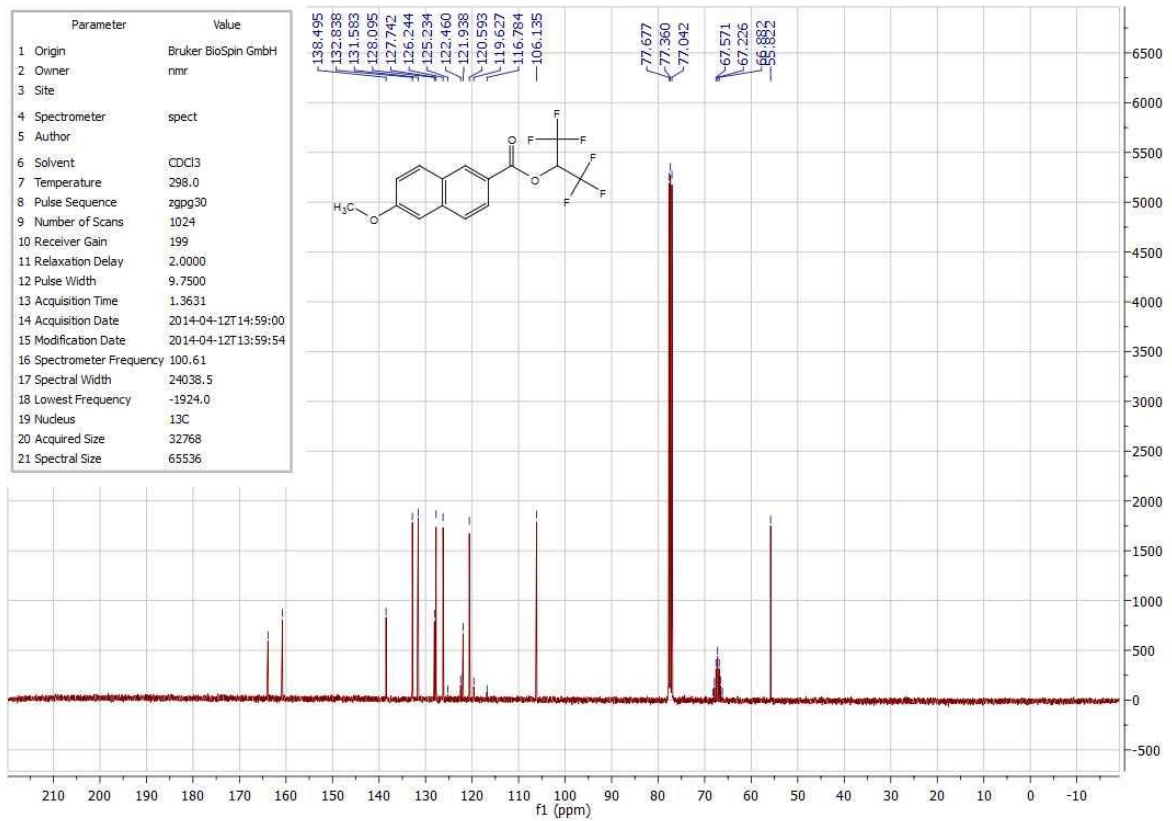
(6b) Perfluorophenyl thiophene-2-carboxylate



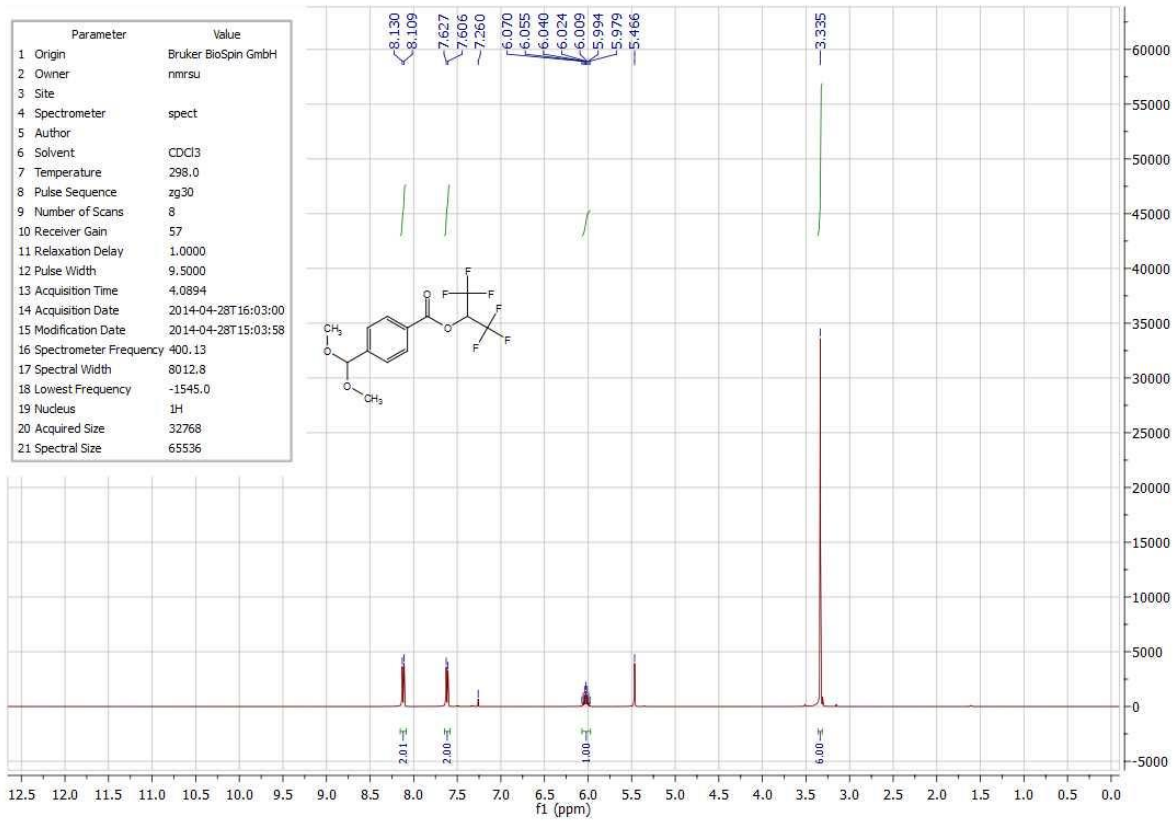
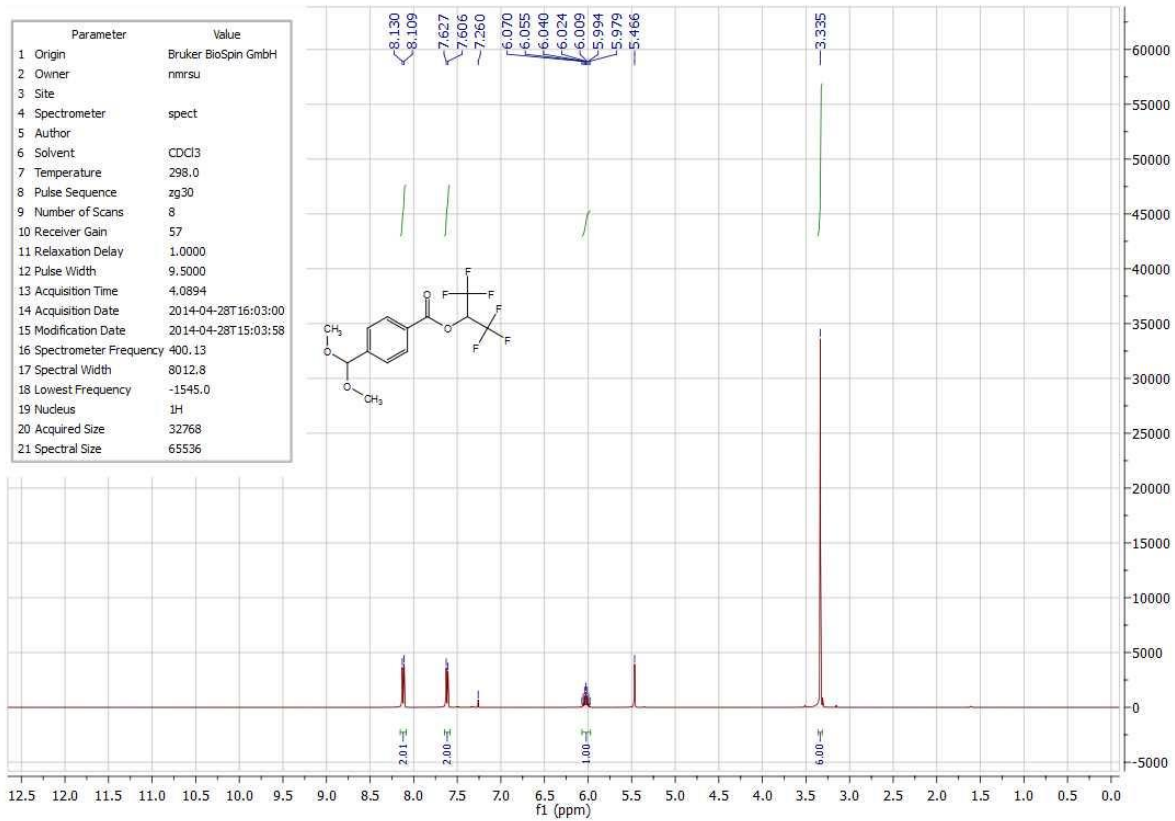


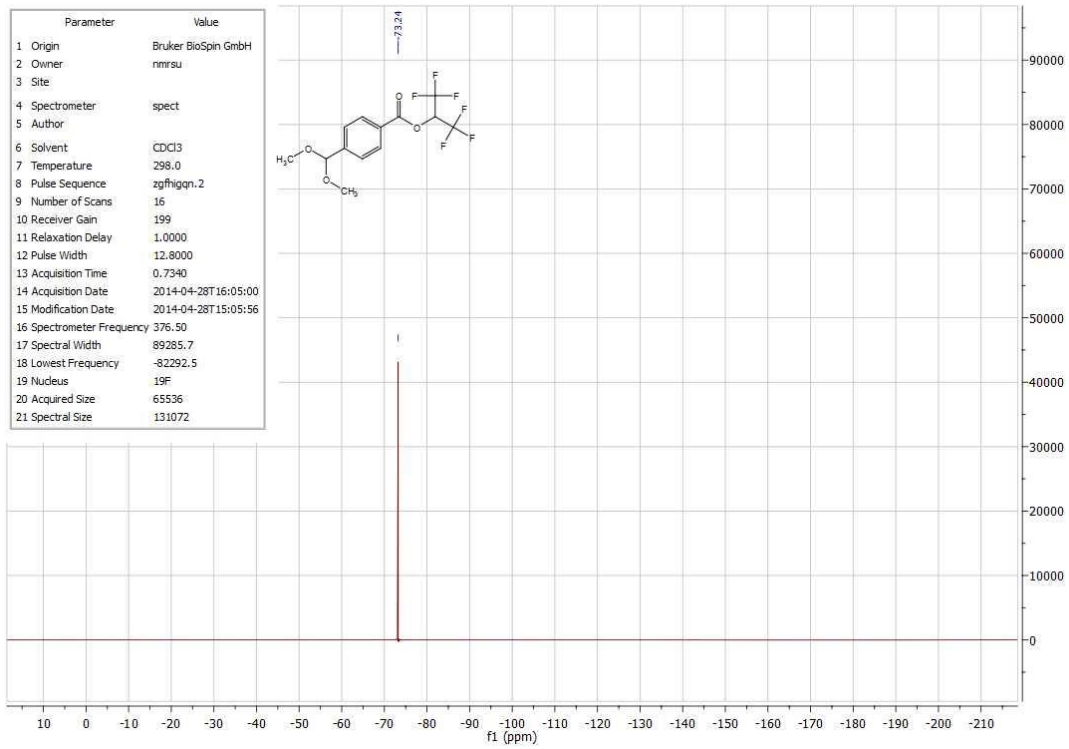
(2c) 1,1,1,3,3,3-hexafluoropropan-2-yl 6-methoxy-2-naphthoate



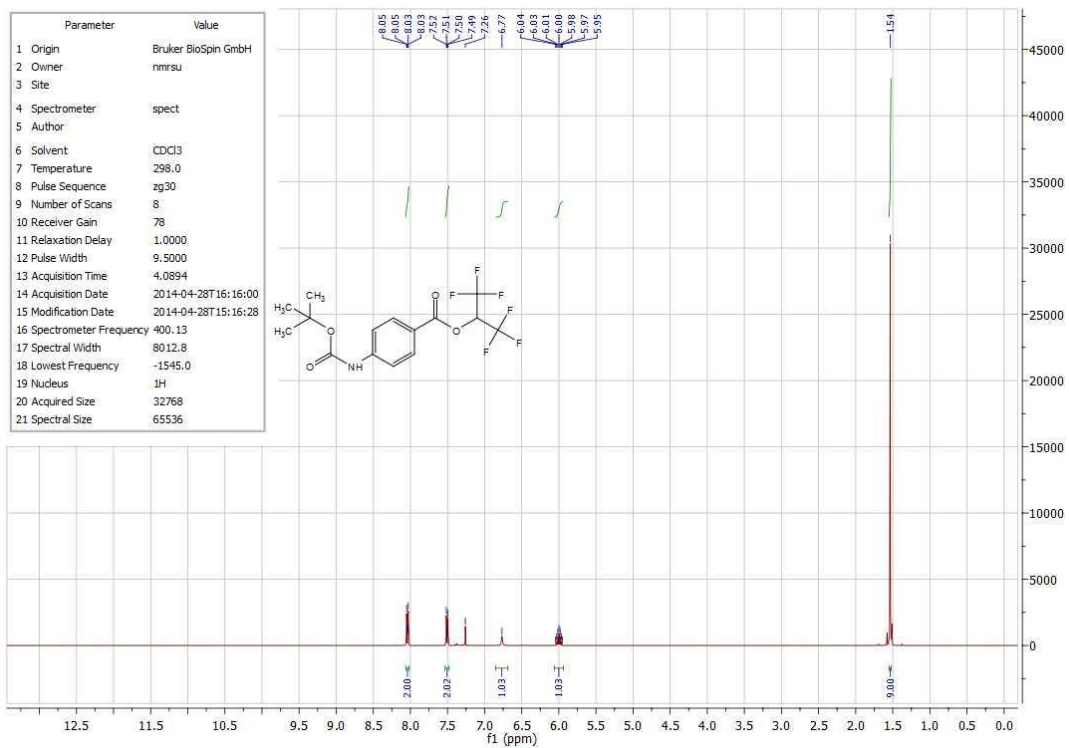


(3c) 1,1,1,3,3,3-hexafluoropropan-2-yl 4-(dimethoxymethyl)benzoate

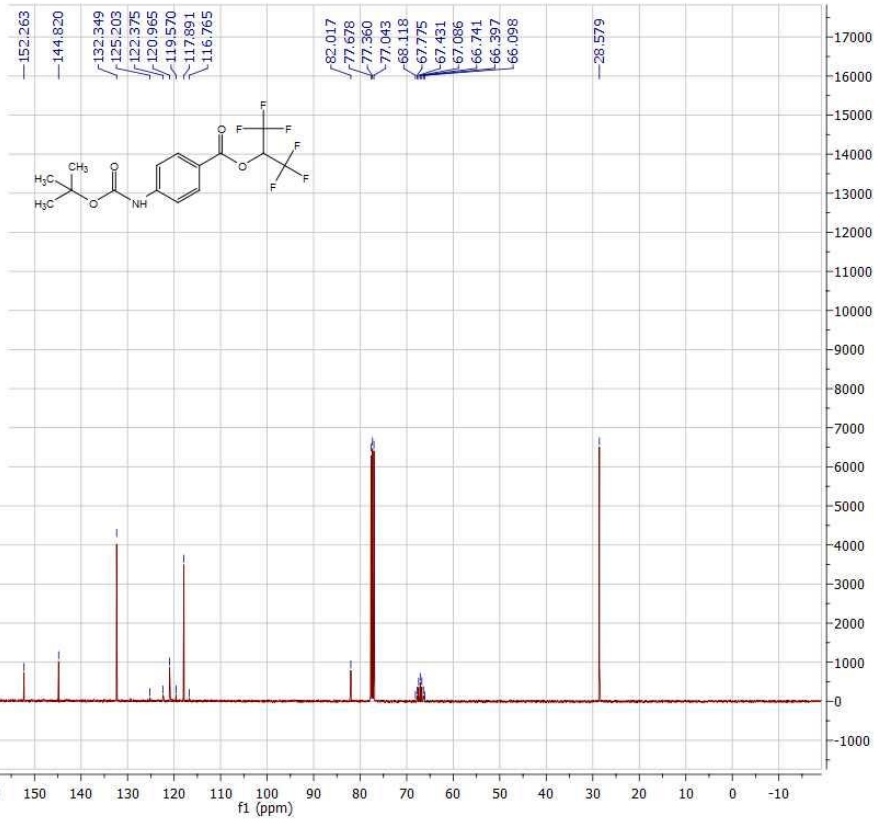




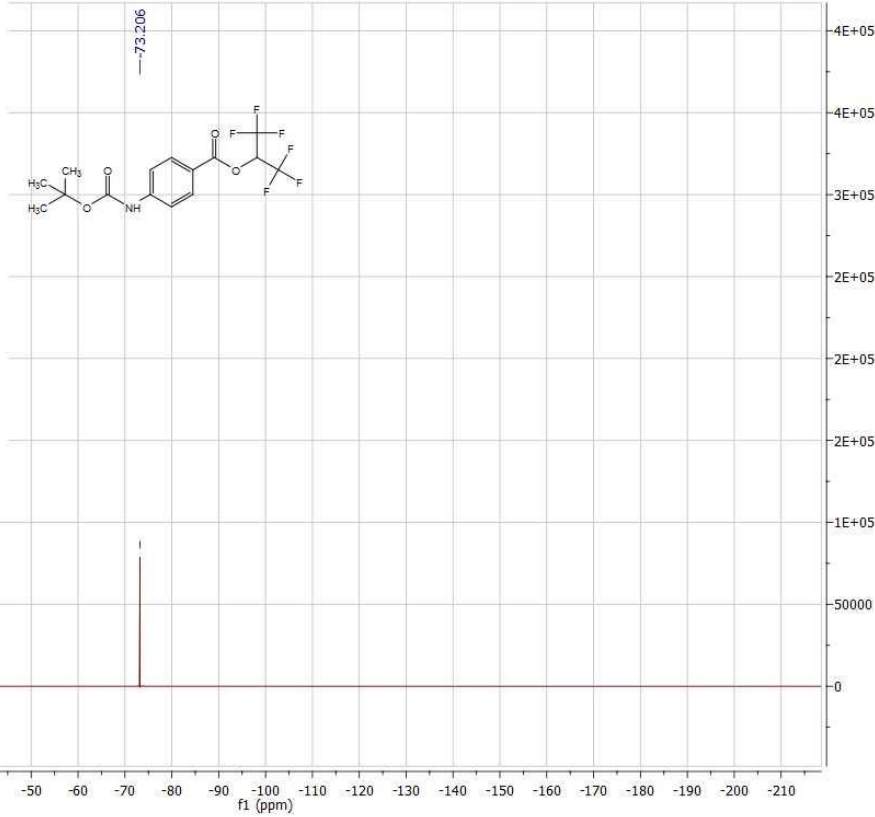
(4c) 1,1,1,3,3,3-hexafluoropropan-2-yl 4-(tert-butoxycarbonylamino)benzoate



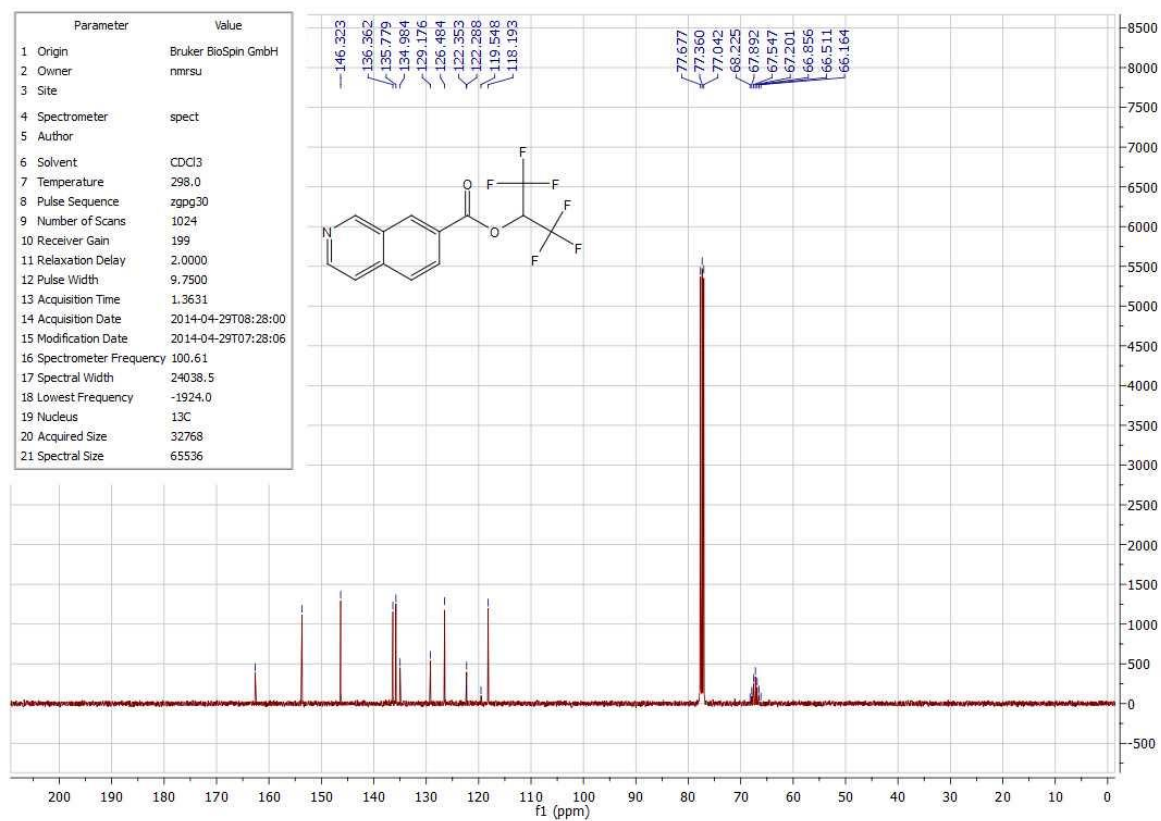
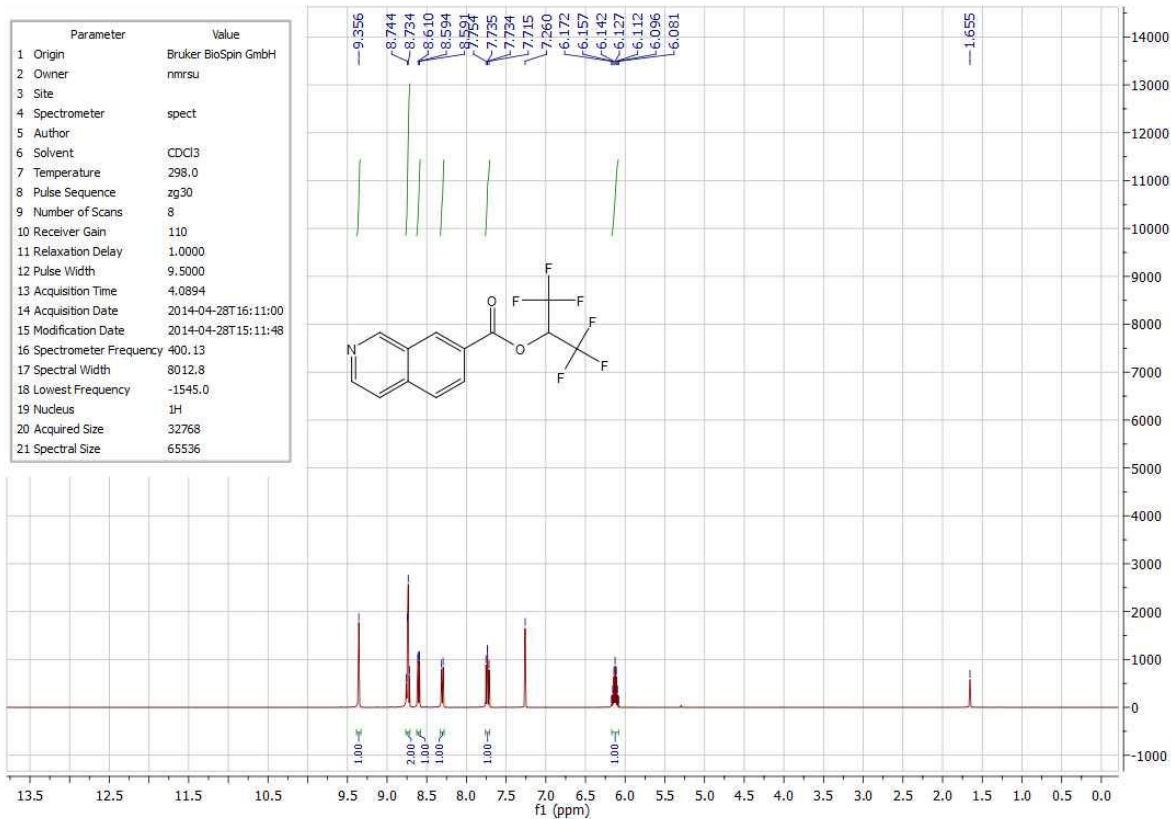
Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Owner	nmrsu
3 Site	
4 Spectrometer	spect
5 Author	
6 Solvent	CDCl3
7 Temperature	298.0
8 Pulse Sequence	zgpg30
9 Number of Scans	1024
10 Receiver Gain	199
11 Relaxation Delay	2.0000
12 Pulse Width	9.7500
13 Acquisition Time	1.3631
14 Acquisition Date	2014-04-30T06:37:00
15 Modification Date	2014-04-30T05:37:42
16 Spectrometer Frequency	100.61
17 Spectral Width	24038.5
18 Lowest Frequency	-1923.8
19 Nucleus	13C
20 Acquired Size	32768
21 Spectral Size	65536

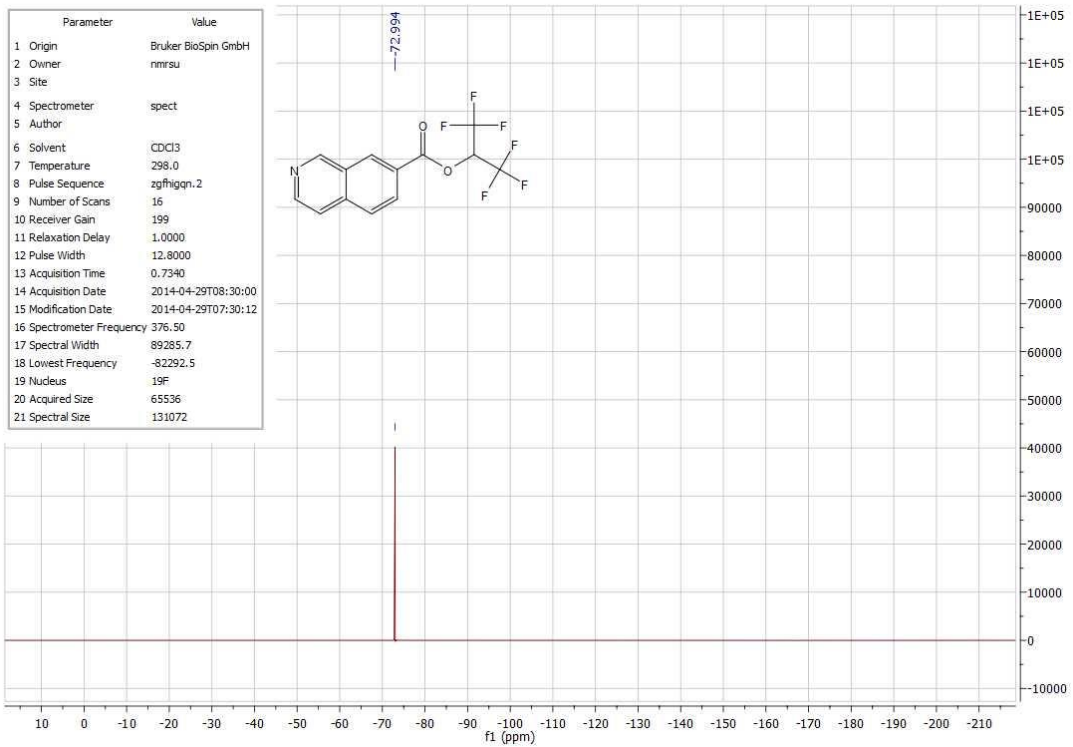


Parameter	Value
1 Origin	Bruker BioSpin GmbH
2 Owner	nmrsu
3 Site	
4 Spectrometer	spect
5 Author	
6 Solvent	CDCl3
7 Temperature	298.0
8 Pulse Sequence	zgfhgqn.2
9 Number of Scans	16
10 Receiver Gain	199
11 Relaxation Delay	1.0000
12 Pulse Width	12.8000
13 Acquisition Time	0.7340
14 Acquisition Date	2014-04-29T18:59:00
15 Modification Date	2014-04-29T17:59:12
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18 Lowest Frequency	-82292.5
19 Nucleus	19F
20 Acquired Size	65536
21 Spectral Size	131072

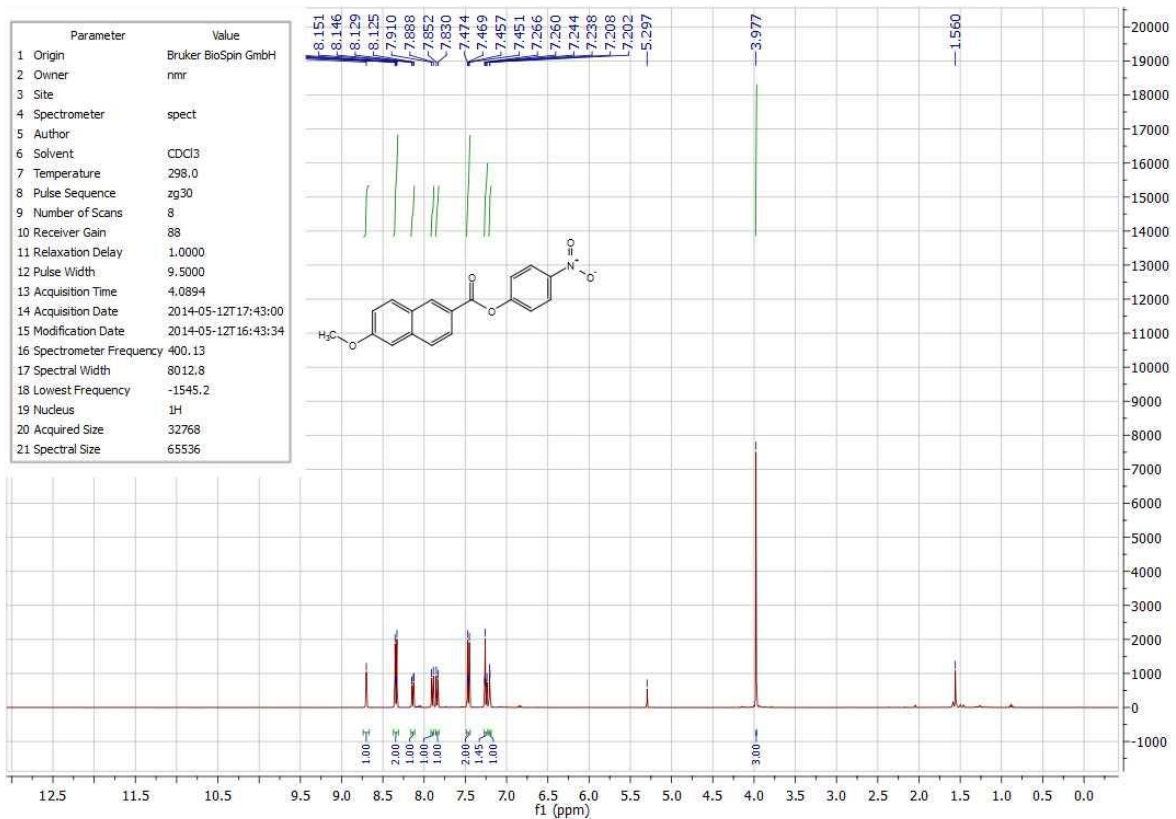


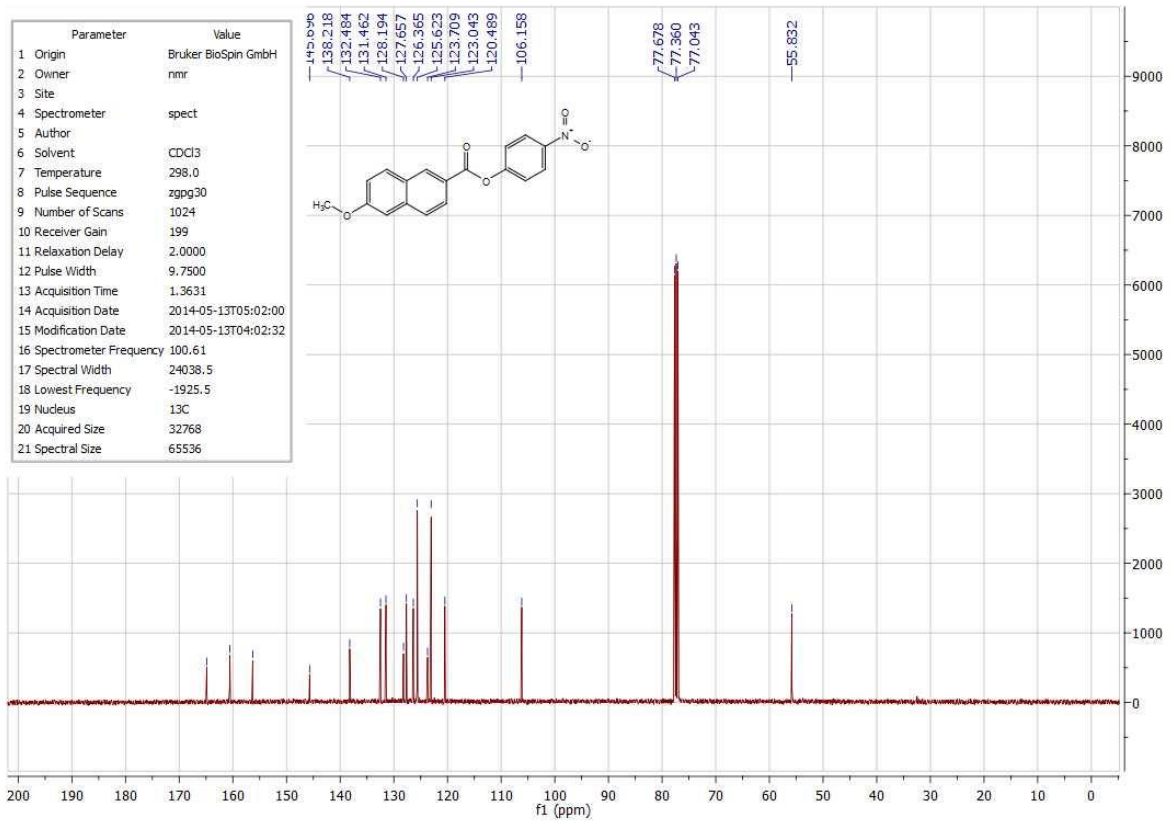
(5c) 1,1,1,3,3,3-hexafluoropropan-2-yl isoquinoline-7-carboxylate



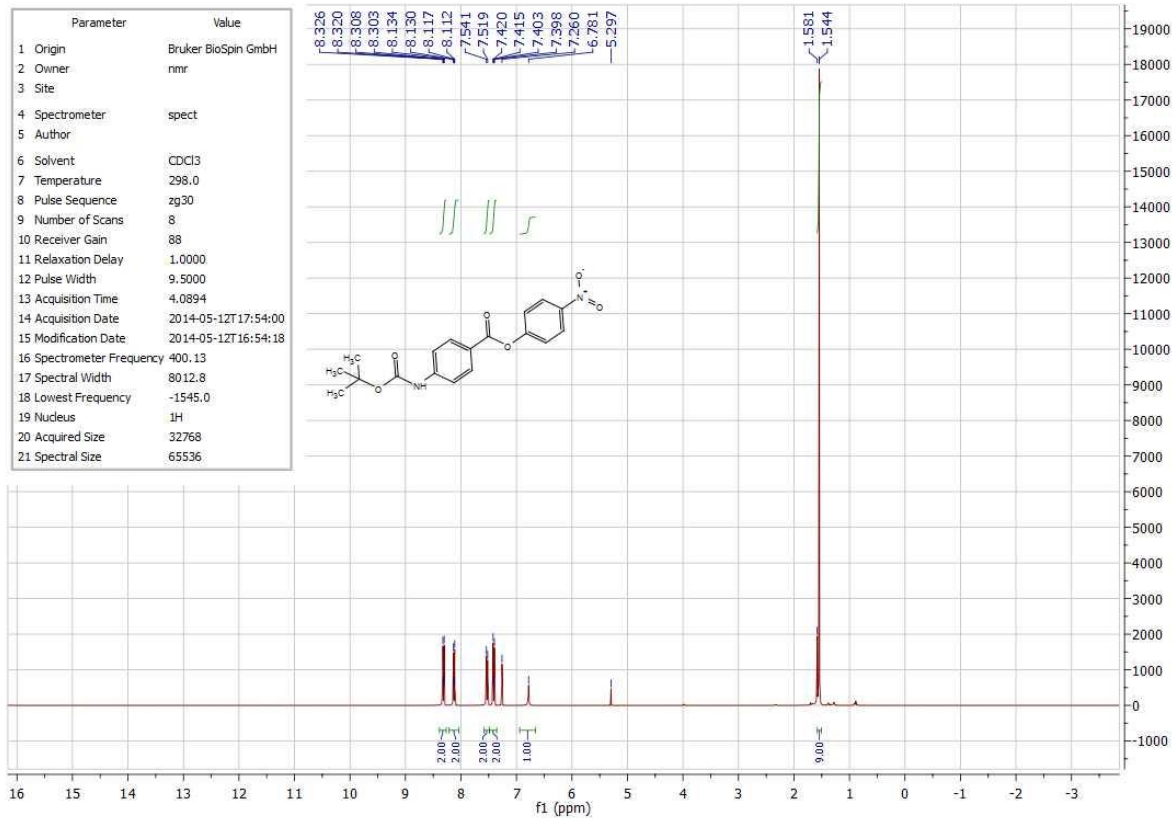


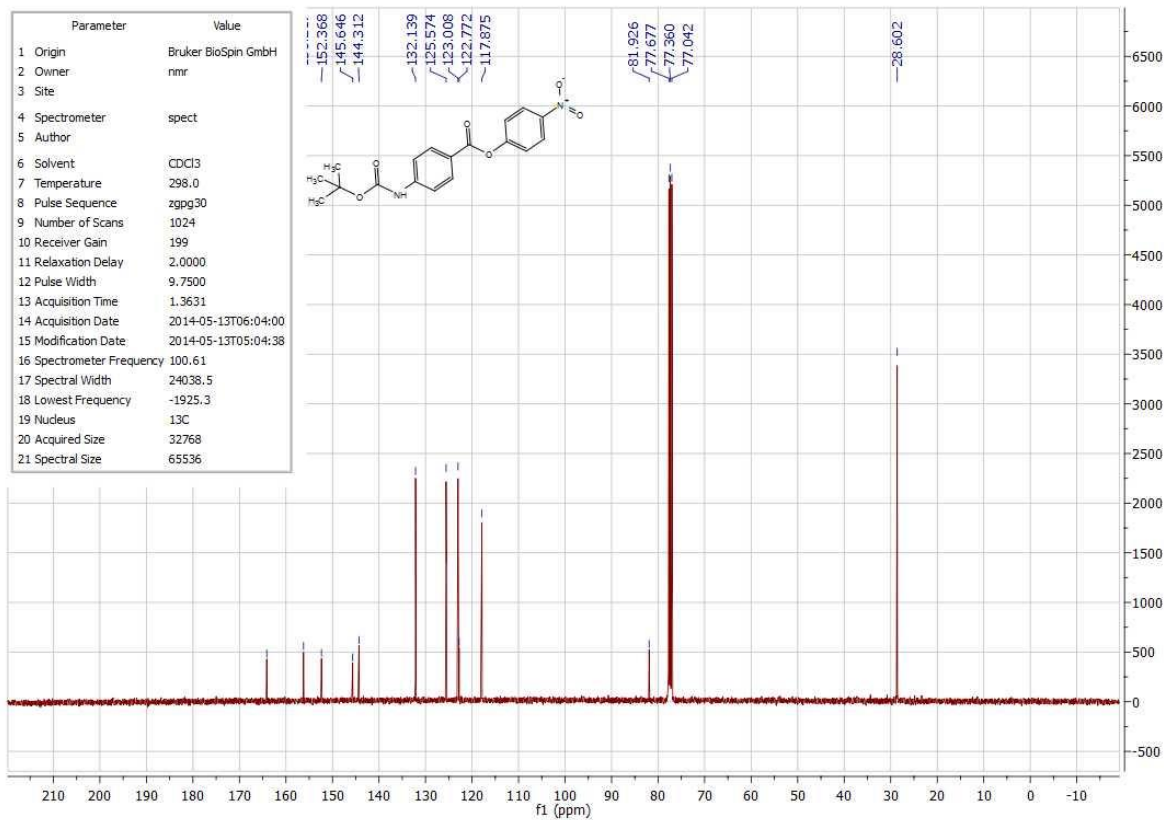
(2d) 4-nitrophenyl 6-methoxy-2-naphthoate





(3d) 4-nitrophenyl 4-(tert-butoxycarbonylamino)benzoate





(2e) 1,3-dioxoisoindolin-2-yl 6-methoxy-2-naphthoate

