Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Expedient synthesis of 3-phenylbicyclo[1.1.1]pentan-1-amine via metal-free homolytic aromatic alkylation of benzene.

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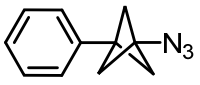
General Information

Materials. All chemicals were purchased from Sigma-Aldrich, Alfa Aesar, Merck and TCI and were used as received. Benzene was taken from Sure/Seal™ bottle (Sigma-Aldrich). Reaction progress was monitored using Merck 60 F254, 0.25 µm silica gel plates (TLC plates) and spots were visualized by UV and/or ceric ammonium molybdate stain. Flash column chromatography was carried out using Merck 60 F254, 0.040-0.063 µm silica gel on Biotage, SP1 HPFC Flash Purification system with 25+M column cartridge, and a flow rate of 25ml/min. Preparative TLC chromatography was carried out using Merck 60 F254, 0.25 µm silica gel plates. Names of structures were generated using ChemBioDraw Ultra 13.0.2.

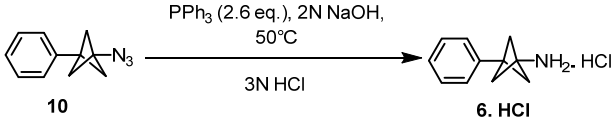
Instrumentation. Proton nuclear magnetic resonance (^{1}H NMR) spectra and carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on Bruker Ultrashield 600 MHz Plus or on Bruker 400 MHz with CryoProbe. Chemical shifts for protons were reported in parts per million (ppm) that are referenced to residual protium in the NMR solvent ($CDCl_3$: 7.26 ppm; CD_3OD: 3.31 ppm; CD_3CN: 1.94 ppm). Chemical shifts for carbon were reported in ppm referenced to the carbon resonances of the NMR solvent ($CDCl_3$: 77.16 ppm; CD_3OD: 49.0 ppm). NMR spectra were processed using MestReNova 9.0.0. and ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, br s = broad singlet, d = doublet, dd = doublet of doublets, br d = broad doublet, br dd = broad doublet of doublets, ddd = doublet of doublet of doublet, t = triplet, td = triplet of doublets, q = quartet, m = multiplet), coupling constants (Hz), and integration. High resolution electrospray ionization (HRMS ESI) mass spectra were recorded using Agilent 6210 Time-of-Flight LC/MS. Infrared (IR) spectra were measured on a PerkinElmer Spectrum100 FT-IR spectrophotometer. UV reactions were carried out using a Rayonet Reactor, RMR-600, at 254 nm. Yield refers to isolated yield of analytically pure material unless otherwise noted.



1-azido-3-phenylbicyclo[1.1.1]pentane, 10: To a solution of 1-azido-3-iodobicyclo[1.1.1]pentane (100mg, 0.425 mmol) in benzene (6 ml) was added tetrabutylammonium cyanoborohydride (119.9mg, 0.425mmol) followed by AIBN (76.8mg, 0.468mmol). A steady stream of air was bubbled through the reaction mixture and the solution was refluxed at 100 °C. Three periodic additions of tetrabutylammonium cyanoborohydride (119.9mg, 0.425mmol) were performed at 0.5 h intervals. The reaction was monitored by ^{1}H NMR and complete consumption of 1-azido-3-iodobicyclo[1.1.1]pentane was observed after 2.5h. The reaction mixture was allowed to cool to room temperature and concentrated *in vacuo*. The crude product was purified by column chromatography on silica gel (gradient elution from 0:100 to 7:93 ethyl acetate - hexanes over 30 column volumes) to afford the desired product **10** as a yellow oil (50.6mg, 65%).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.33-7.30 (m, 2H), 7.27-7.21 (m, 3H), 2.28 (s, 6H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 138.1, 128.5, 127.1, 126.5, 54.6, 51.1, 37.6; **IR** (thin film, cm^{-1}) 2923, 2109, 1208, 749, 697; **TLC** (5:95 Ethyl acetate/Hexane): R_f = 0.62

3-phenylbicyclo[1.1.1]pentan-1-amine hydrochloride, 6

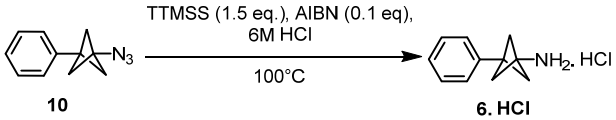


Staudinger reaction

To a solution of 1-azido-3-phenylbicyclo[1.1.1]pentane **10** (27.2mg, 0.147mmol) in chloroform (2.5 ml) was added triphenylphosphine (100.2mg, 0.382mmol) and the reaction mixture was then heated to 50 °C. The reaction was monitored by TLC which showed complete consumption of the starting material, 1-azido-3-phenylbicyclo[1.1.1]pentane **10** after 2.5h. The reaction mixture was cooled to room temperature and chloroform was evaporated thoroughly *in vacuo*. The resulting residue was reconstituted in 2N NaOH (1 ml) and MeOH (1 ml) and stirred at 50 °C for 15 h. The reaction mixture was then diluted with water (2 ml) and the organics were

extracted with EtOAc (3×3 ml). The organic layer was acidified with 3N HCl (3 ml) and the aqueous layer was concentrated *in vacuo* to afford the desired product **6** as an off-white solid (22.5mg, 78.4%).

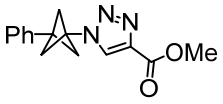
TTMSS Reduction



10 **6. HCl**

To 1-azido-3-phenylbicyclo[1.1.1]pentane (100mg, 0.540mmol) was added tris(trimethylsilyl)silane (0.25ml, 0.810mmol), AIBN (8.86mg, 0.054mmol) and 6M HCl (1.2ml). The reaction mixture was stirred under atmospheric conditions and heated at 100°C for 5h. After 1/2h of heating, the reaction mixture turned from colourless to dark brown. The reaction was monitored by ^{1}H NMR for the disappearance of the starting material, 1-azido-3-phenylbicyclo[1.1.1]pentane and complete consumption of starting material was observed at 5h. Hence, the reaction was quenched with water (1ml) and extracted with ethyl acetate (2ml). The aqueous layer was separated and the organics were extracted with 2M HCl solution (2ml). The aqueous layers were combined and washed with ethyl acetate (4ml). The aqueous layer was concentrated *in vacuo* to afford 3-phenylbicyclo[1.1.1]pentan-1-amine hydrochloride (83mg, 79%) as a brown solid.

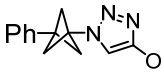
^{1}H NMR (600 MHz, MeOD): δ 7.33-7.30 (m, 2H), 7.26-7.24 (m, 3H), 2.36 (s, 6H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 138.3, 129.4, 128.3, 127.3, 54.4, 44.4, 39.8; **HRMS (ESI^{+})** Calcd for C11H13N +H, 160.1048; Found, 160.1113; **IR** (thin film, cm^{-1}): 2923, 2733, 1988, 1251, 765, 698.



methyl-1-(3-phenylbicyclo[1.1.1]pentan-1-yl)-1*H*-1,2,3-triazole-4-carboxylate, 12a: To a solution of methyl 1-(3-iodobicyclo[1.1.1]pentan-1-yl)-1*H*-1,2,3-triazole-4-carboxylate (50mg, 0.157mmol) in benzene (3 ml) was added tetrabutylammonium cyanoborohydride (22.2 mg, 0.079mmol) followed by AIBN (28.3 mg, 0.173mmol). A steady stream of air was bubbled through the reaction mixture and the solution was refluxed at 100 °C. Tetrabutylammonium cyanoborohydride (22.2mg, 0.079mmol) was added to the reaction

mixture three additional times at 0.5 h intervals. The reaction was monitored by ^{1}H NMR and complete consumption of the starting material was observed after 4h. The reaction mixture was then cooled to room temperature and concentrated *in vacuo*. The resulting residue was purified by column chromatography on silica gel (gradient elution from 0:100 to 40:60 ethyl acetate - hexanes over 30 column volumes) to afford the desired product **12a** as a white solid (28.2 mg, 67%).

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.16 (s, 1H), 7.38-7.35 (m, 2H), 7.32-7.28 (m, 3H), 3.96 (s, 3H), 2.70 (s,6H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 161.3, 139.9, 136.9, 128.6, 127.6, 126.5, 126.4, 55.5, 52.4, 49.7, 38.7; **HRMS (ESI^{+})** Calcd for C15H15N3O2+Na, 292.1164; Found, 292.1058; **IR** (thin film, cm^{-1}): 3111, 1731, 1447, 1201, 747, 700; **TLC** (50:50 Ethyl acetate/Hexane): R_f = 0.67



4-ethoxy-1-(3-phenylbicyclo[1.1.1]pentan-1-yl)-1*H*-1,2,3-triazole, 12b: To a solution of 4-ethoxy-1-(3-iodobicyclo[1.1.1]pentan-1-yl)-1*H*-1,2,3-triazole (50mg, 0.164mmol) in benzene (3 ml) was added tetrabutylammonium cyanoborohydride (23.1 mg, 0.082mmol) followed by AIBN (29.6 mg, 0.1803 mmol). A steady stream of air was bubbled through the reaction mixture and the solution was refluxed at 100 °C. Tetrabutylammonium cyanoborohydride (23.1 mg, 0.082mmol) was added to the reaction mixture three additional times at 0.5 h intervals. The reaction was monitored by ^{1}H NMR and complete consumption of the starting material was observed after 2.5 h. The reaction mixture was then allowed to cool to room temperature and concentrated *in vacuo*. The crude product obtained was purified by column chromatography on silica gel (gradient elution from 0:100 to 40:60 ethyl acetate - hexanes over 30 column volumes) to afford the desired product **12b** as a white solid (30.1mg, 72%).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.37-7.35 (m, 2H), 7.30-7.28 (m, 3H), 7.04 (s, 1H), 4.30 (q, *J* = 6.6 Hz, 2H), 2.63 (s, 6H), 1.42 (t, *J* = 7.2Hz, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 161.0, 137.5, 128.6, 127.4, 126.6, 104.7, 66.5, 55.2, 49.8, 38.5, 15.0; **HRMS (ESI^{+})** Calcd for C15H17N3O+H,

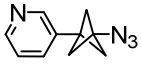
256.1372; Found 256.1447; **IR** (thin film, cm^{-1}): 2979, 1562, 1341, 1220, 887, 741, 696; **TLC** (50:50 Ethyl acetate/Hexane): R_f = 0.63

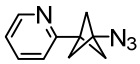
 **2-(3-azidobicyclo[1.1.1]pentan-1-yl)pyridine (2/6 - regioisomer),**

3-(3-azidobicyclo[1.1.1]pentan-1-yl)pyridine (3/5 - regioisomer),

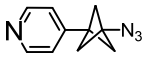
4-(3-azidobicyclo[1.1.1]pentan-1-yl)pyridine (4 substituted regioisomer), 12c

To 1-azido-3-iodobicyclo[1.1.1]pentane (50mg, 0.213mmol) was added pyridine (3ml), tetrabutylammonium cyanoborohydride (90mg, 0.319mmol) and AIBN (38.4mg, 0.234mmol). A steady stream of air was bubbled through the reaction mixture and the solution was refluxed at 100 °C. After 50 minutes, ^{1}H NMR showed complete consumption of 1-azido-3-iodobicyclo[1.1.1]pentane. The reaction mixture was evaporated and the crude product was purified by silica gel flash chromatography (gradient elution 0:100 to 60:40 ethyl acetate - hexanes over 30 column volumes) to afford **12c** as a mixture of isomers. The product was further purified by preparative TLC (80:20 ethyl acetate - hexanes) to afford **3-(3-azidobicyclo[1.1.1]pentan-1-yl)pyridine** (2.2mg), **2-(3-azidobicyclo[1.1.1]pentan-1-yl)pyridine** (10.6mg) and **4-(3-azidobicyclo[1.1.1]pentan-1-yl)pyridine** (2.4mg) as separate isomers with a combined total yield of 38%.

 **3-(3-azidobicyclo[1.1.1]pentan-1-yl)pyridine (3/5 - regioisomer), ^{1}H NMR** (600 MHz, CD_3CN): δ 8.48-8.47 (m, 1H), 8.47-8.46 (m, 1H), 7.69 (ddd, *J* = 7.8, 2.3, 1.6Hz, 1H), 7.35 (ddd, *J* = 7.8, 4.9, 0.9 Hz, 1H), 2.34 (s, 6H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 147.9, 147.6, 134.6, 133.8, 123.5, 54.6, 51.2, 35.7; **HRMS (ESI$^+$)** Calcd for C10H10N4+H, 187.0905; Found, 187.0983; **IR** (thin film, cm^{-1}) 2988, 2112, 1267, 736; **TLC** (70:30 Ethyl acetate/Hexane): R_f = 0.61

 **2-(3-azidobicyclo[1.1.1]pentan-1-yl)pyridine (2/6 - regioisomer), ^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.57-8.56 (m, 1H), 7.65 (td, J = 7.7, 1.8 Hz, 1H), 7.21 (dd, J = 7.8 , 1.1 Hz, 1H), 7.18 (ddd, J = 7.7 , 4.9, 1.1 Hz, 1H), 2.38 (s, 6H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 157.2, 149.3, 136.9,

122.3, 121.4, 54.4, 51.4, 38.3; **HRMS (ESI$^+$)** Calcd for C10H10N4+H, 187.0905; Found, 187.0985; **IR** (thin film, cm^{-1}) 2988, 2112, 1275, 741, 577; **TLC** (70:30 Ethyl acetate/Hexane): R_f = 0.74


4-(3-azidobicyclo[1.1.1]pentan-1-yl)pyridine (4 substituted regioisomer), ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.55 (d, *J* = 4.8 Hz, 2H), 7.16 (d, *J* = 4.8 Hz, 2H), 2.32 (s, 6H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 149.3, 147.6, 121.9, 54.5, 51.2, 36.8; **HRMS (ESI$^+$)** Calcd for C10H10N4+H, 187.0905; Found 187.0985; **IR** (thin film, cm^{-1}): 2989, 2112, 1602, 1275, 739, 674; **TLC** (70:30 Ethyl acetate/Hexane): R_f = 0.50

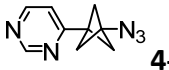

5-(3-azidobicyclo[1.1.1]pentan-1-yl)pyrimidine, 4-(3-azidobicyclo[1.1.1]pentan-1-yl)pyrimidine, 2-(3-azidobicyclo[1.1.1]pentan-1-yl)pyrimidine, 12d

To 1-azido-3-iodobicyclo[1.1.1]pentane (50mg, 0.213mmol) was added pyrimidine (3ml), tetrabutylammonium cyanoborohydride (60mg, 0.213mmol) and AIBN (38.4mg, 0.234mmol). A steady stream of air was bubbled through the reaction mixture and the solution was refluxed at 100 °C. After 35 minutes, ^{1}H NMR showed consumption of 1-azido-3-iodobicyclo[1.1.1]pentane. The reaction mixture was evaporated, quenched with water and extracted with ethyl acetate thrice. The organics were dried over anhydrous sodium sulfate and concentrated *in vacuo.* The crude obtained was purified by silica gel flash chromatography (gradient elution 0:100 to 75:25 ethyl acetate - hexanes over 30 column volumes) to afford **12d** (18.7mg, 47%) as a mixture of isomers. This was then further purified by preparative TLC with 2:98 methanol: dichloromethane (with ammonium hydroxide) to afford the 2-substituted regioisomer of 2-(3-azidobicyclo[1.1.1]pentan-1-yl)pyrimidine (2.9mg, 7.3%) (TLC Rf 0.47) and a mixture of 4- and 5-substituted regioisomers (6.7mg, 16.8%) (TLC Rf 0.35). The total yield of all isomers was 24%.

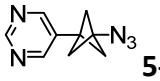

2-(3-azidobicyclo[1.1.1]pentan-1-yl)pyrimidine ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.71 (d, *J* = 4.8Hz, 2H), 7.19 (t, *J* = 4.8Hz, 1H), 2.45 (s, 6H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 166.4, 157.3,

119.4, 54.6, 51.6, 38.9; **HRMS (ESI$^+$)** Calcd for C9H9N5+H, 188.0858; Found, 188.0929; **IR** (thin film, cm^{-1}): 2982, 2112, 1265, 737, 651; **TLC** (75:25 Ethyl acetate/Hexane): R_f = 0.44

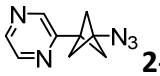
Visible peaks for the ^{1}H NMR of the mixture



4-(3-azidobicyclo[1.1.1]pentan-1-yl)pyrimidine major , ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.15 (br s , 1H), 8.67 (d, *J* = 4.8 Hz, 1H), 7.21 (dd, *J* = 4.8Hz, 1.2 Hz, 1H), 2.39 (s, 6H); **HRMS (ESI$^+$)** Calcd for C9H9N5+H, 188.0858; Found, 188.0937; **TLC** (75:25 Ethyl acetate/Hexane): R_f = 0.35



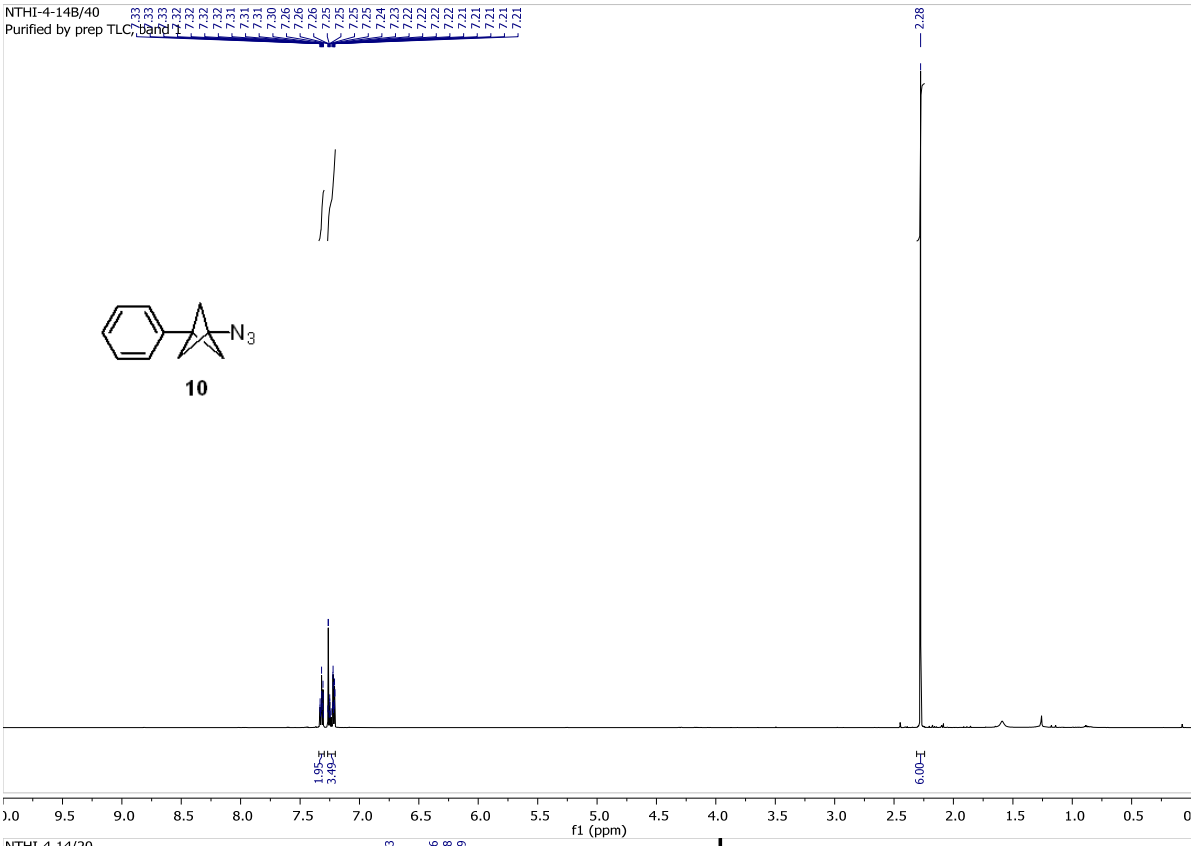
5-(3-azidobicyclo[1.1.1]pentan-1-yl)pyrimidine minor, ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.12 (s, 1H), 8.59 (s, 2H), 2.38 (s, 6H); **HRMS (ESI$^+$)** Calcd for C9H9N5+H, 188.0858; Found, 188.0937; **TLC** (75:25 Ethyl acetate/Hexane): R_f = 0.35



2-(3-azidobicyclo[1.1.1]pentan-1-yl)pyrazine, 12e: Pyrazine (5g) was melted at 100°C to form a colourless liquid. 1-azido-3-iodobicyclo[1.1.1]pentane (100mg, 0.425mmol), tetrabutylammonium cyanoborohydride (120mg, 0.425mmol) and AIBN (76.7mg, 0.468mmol) was added to the hot pyrazine and the reaction mixture was refluxed at 100°C. Subsequently addition of 1 equivalent of the reducing agent was performed periodically, after every 30 min, for the first 2.5 hours followed by an additional equivalent of AIBN after the 3 hours. Heating was stopped after 3.5 hours and the un-reacted pyrazine was distilled out. The residue was subjected to purification by silica gel flash chromatography (gradient elution with 0 to 50% ethyl acetate - hexanes over 45 column volumes) to afford **12e** (19 mg, 24 %) as a yellow oil.

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.52 – 8.51 (m, 2H), 8.47 (br d, *J* = 2.4 Hz, 1H), 2.43 (s, 6H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 153, 144.2, 143.2, 143, 54.5, 51.6, 36.7; **HRMS (ESI$^+$)** Calcd for C9H9N5+H, 188.0858; Found, 188.0939; IR (thin film, cm^{-1}): 2992, 2110, 1406, 1274, 769, 589; **TLC** (25:75 Ethyl acetate/Hexane): R_f = 0.34

^{1}H, ^{13}C NMR Spectra:



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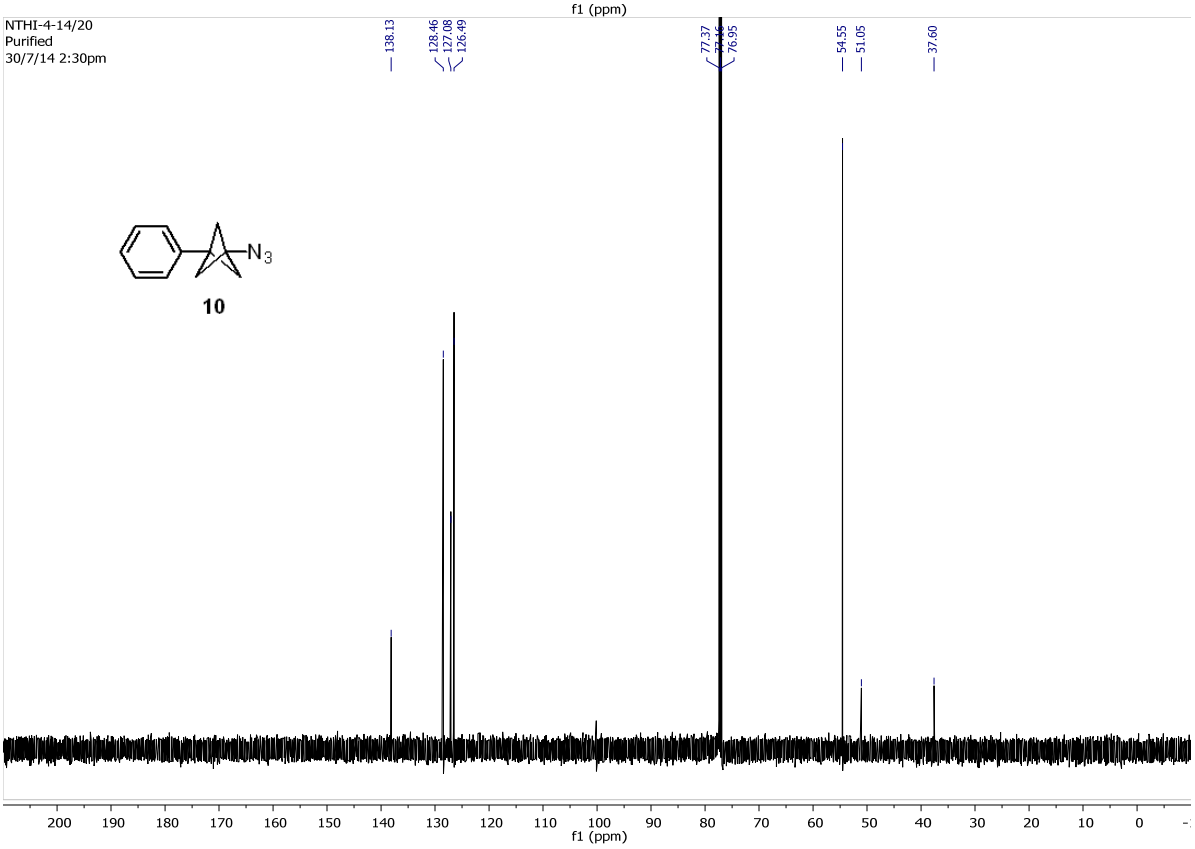

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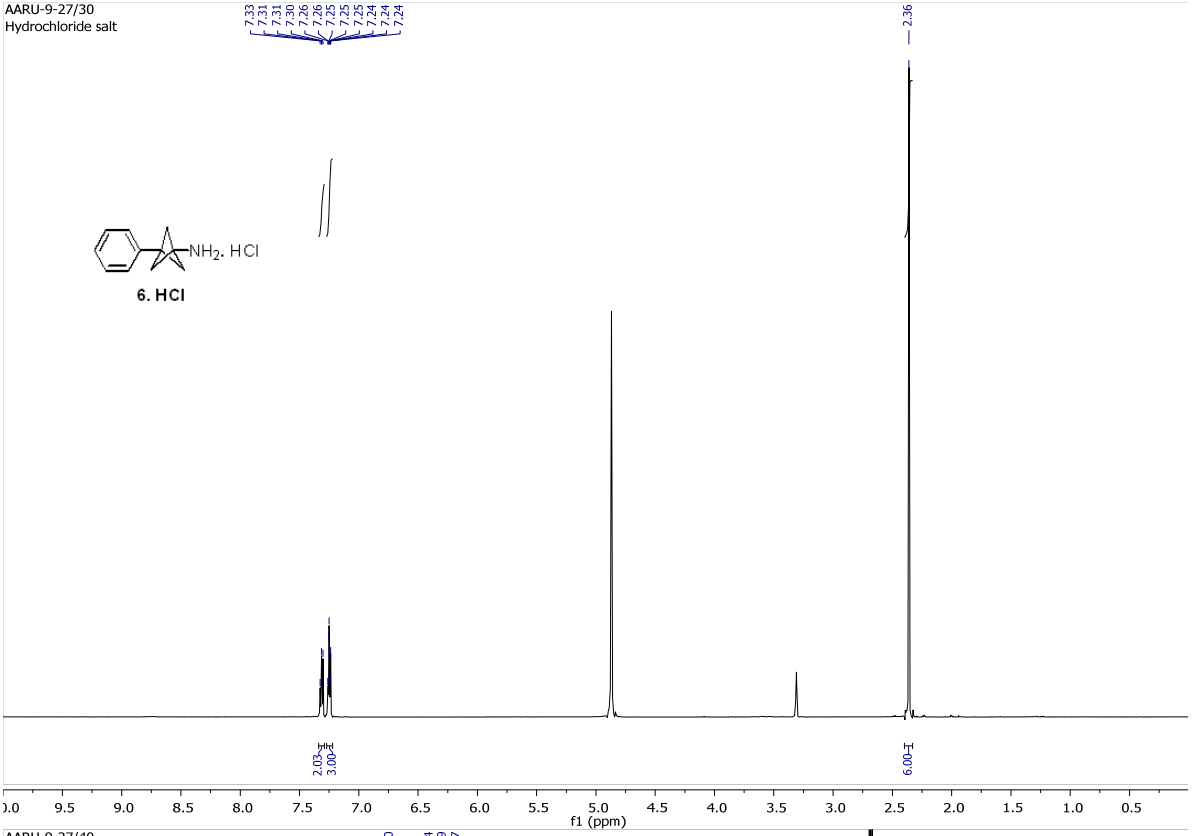
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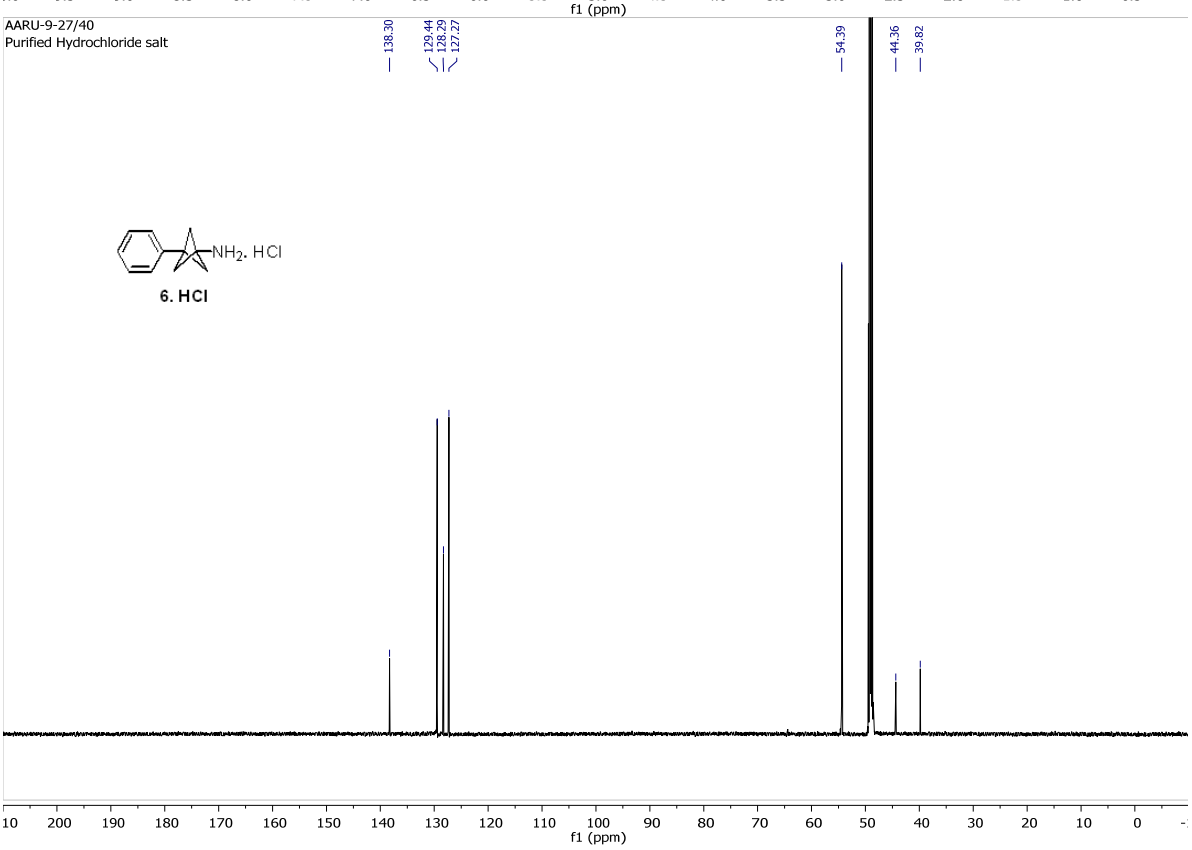
6.00
f1 (ppm)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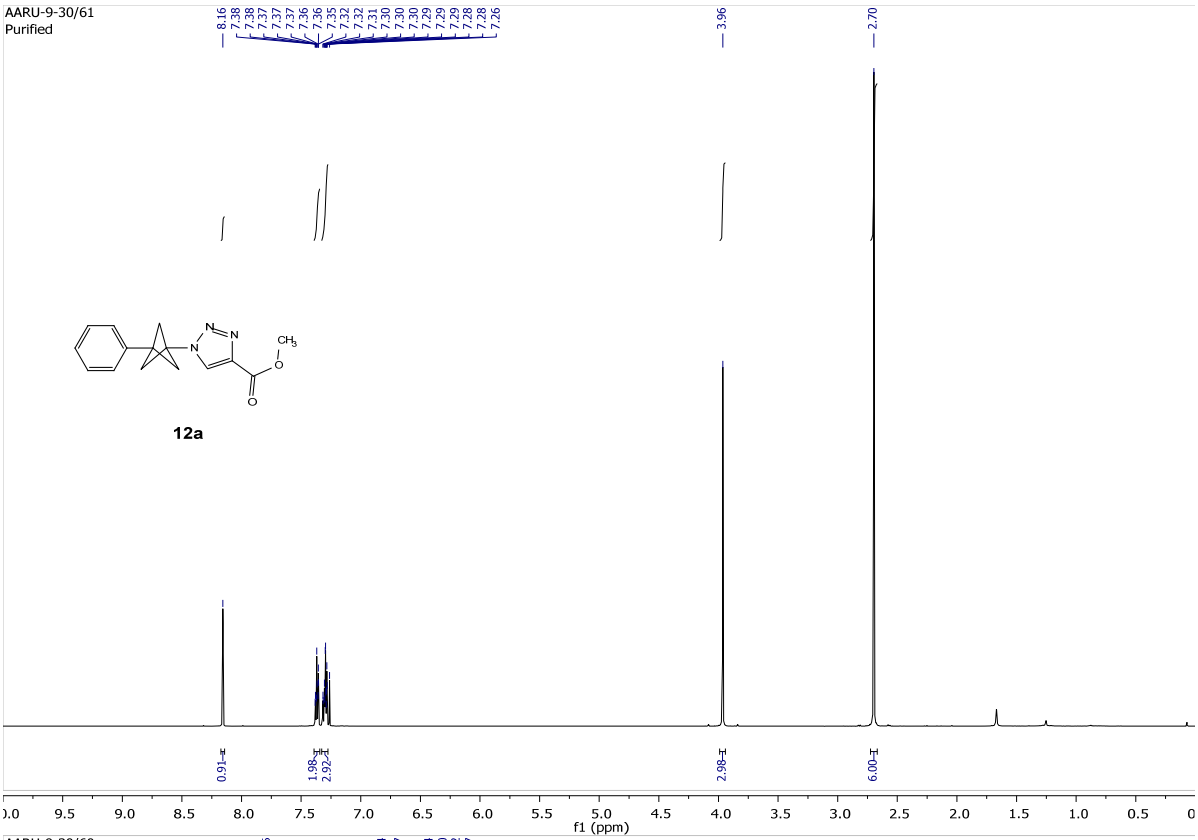
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7.29
7.29
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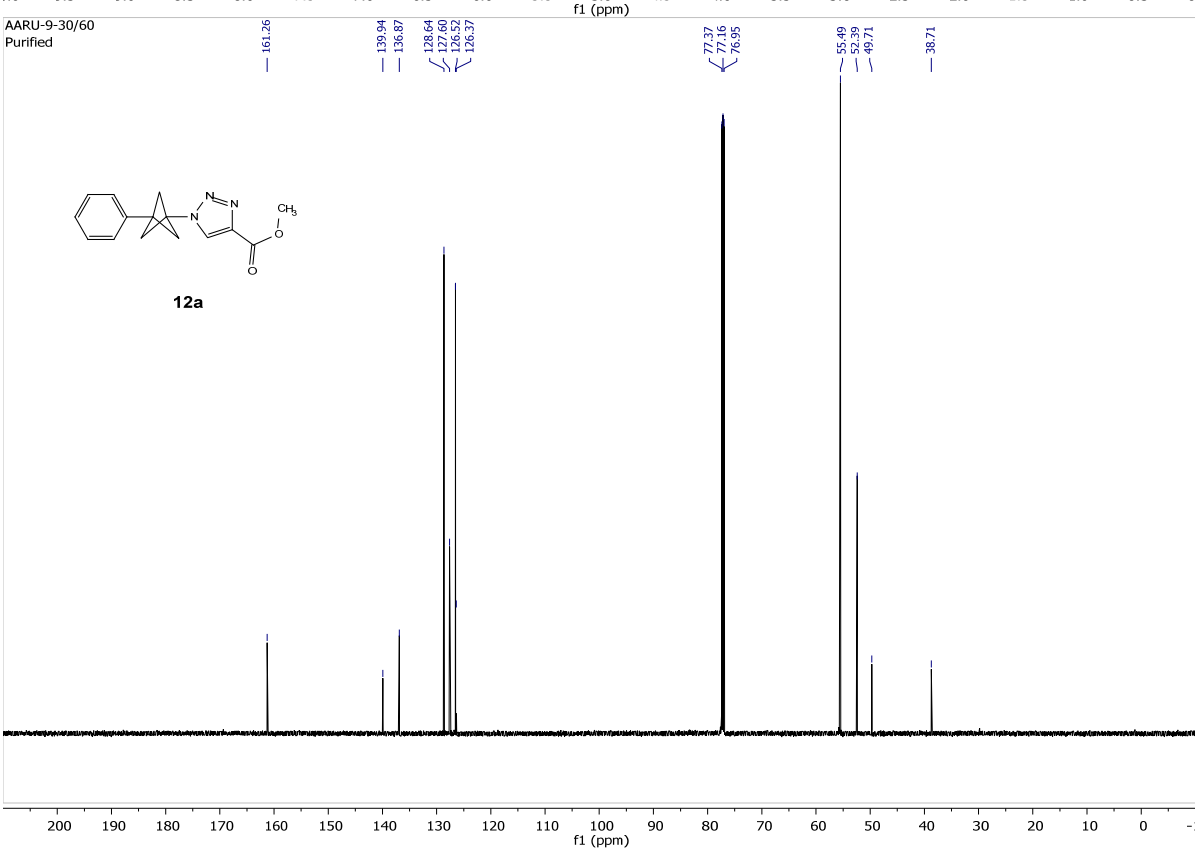
f1 (ppm)

12a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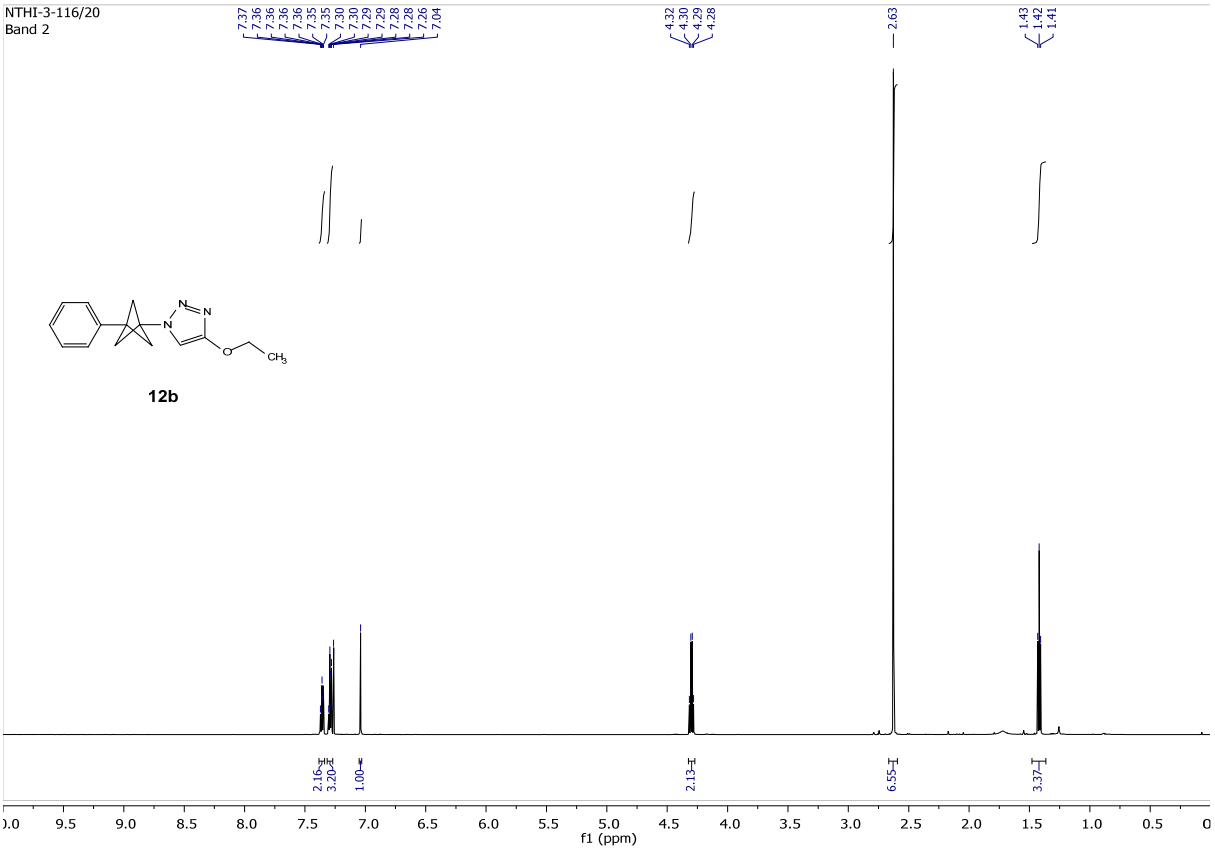
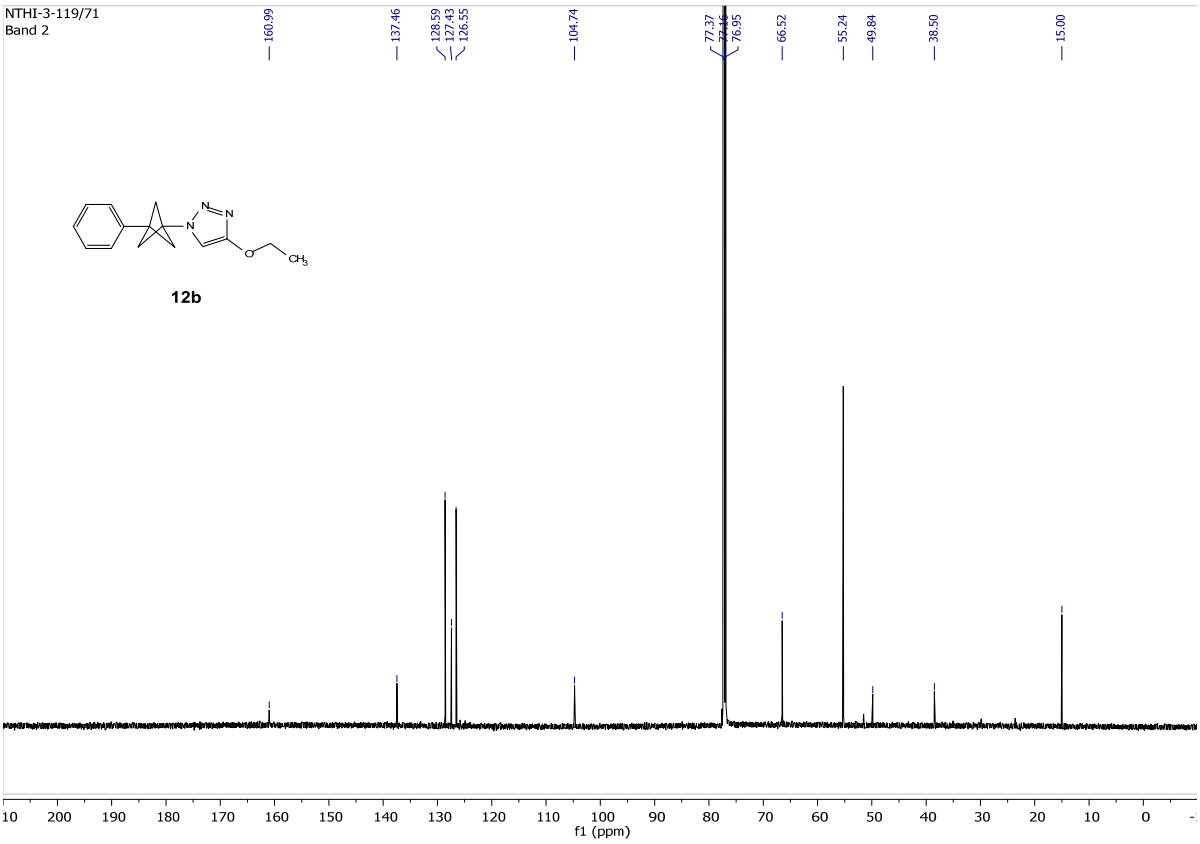
139.94
136.87
128.64
127.60
126.52
126.37
77.37
77.16
76.95
55.49
52.39
49.71
38.71
f1 (ppm)

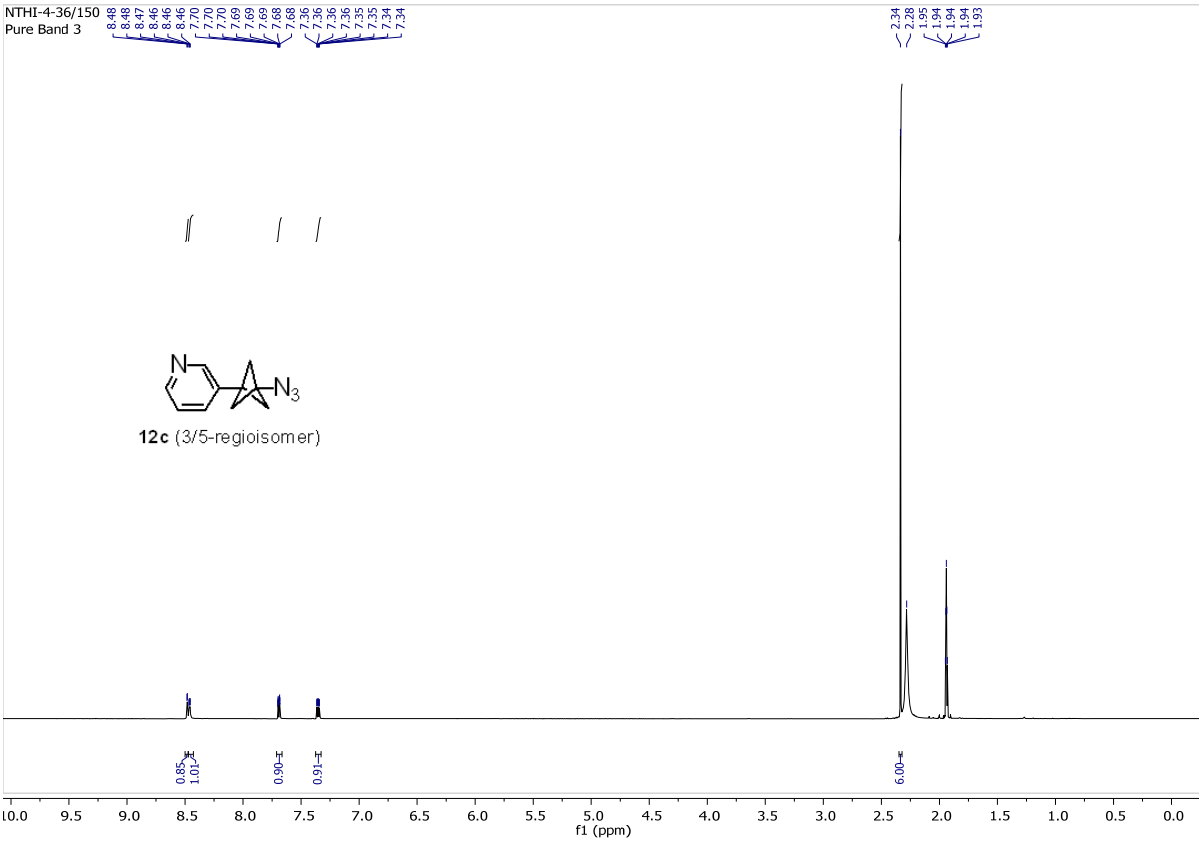
12a

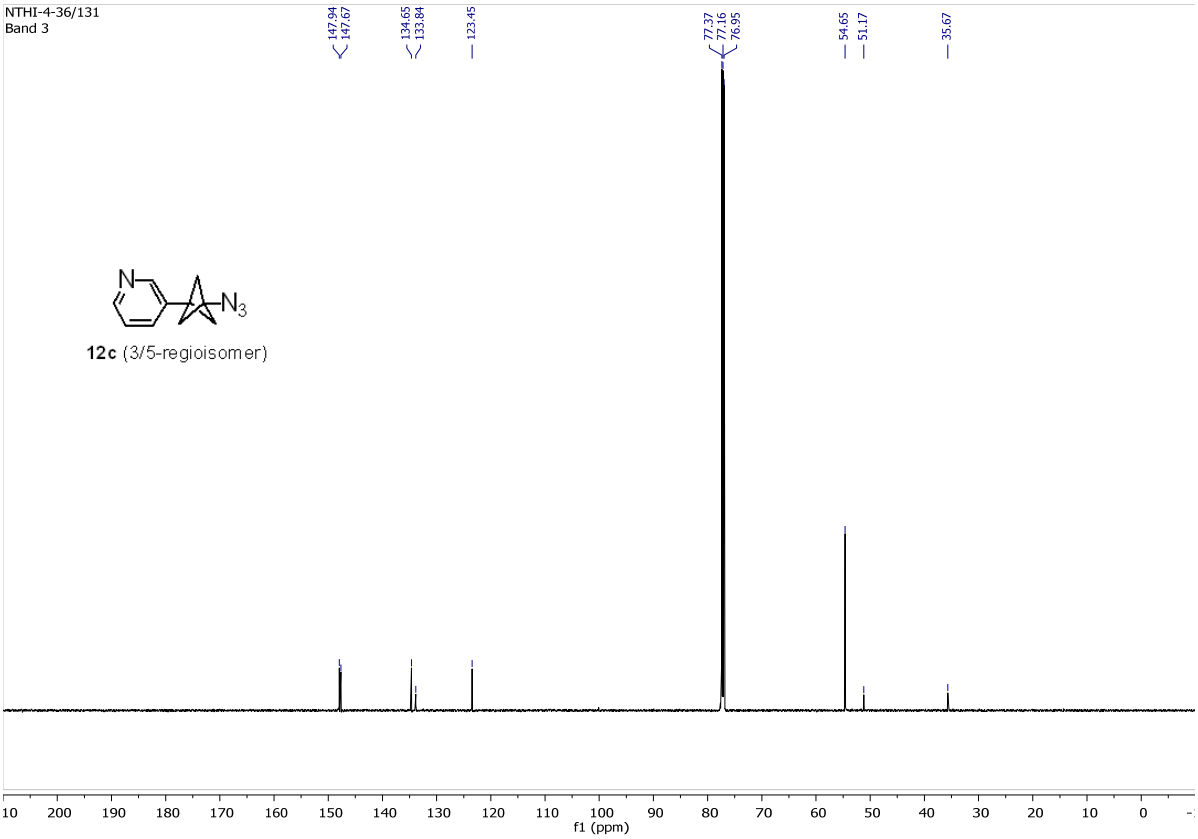

NTHI-3-116/20
Band 2
7.37
7.36
7.36
7.36
7.36
7.35
7.35
7.30
7.30
7.29
7.29
7.28
7.28
7.26
7.04
4.32
4.30
4.29
4.28
2.63
1.43
1.42
1.41
2.16
3.20
1.00
2.13
6.55
3.37
f1 (ppm)

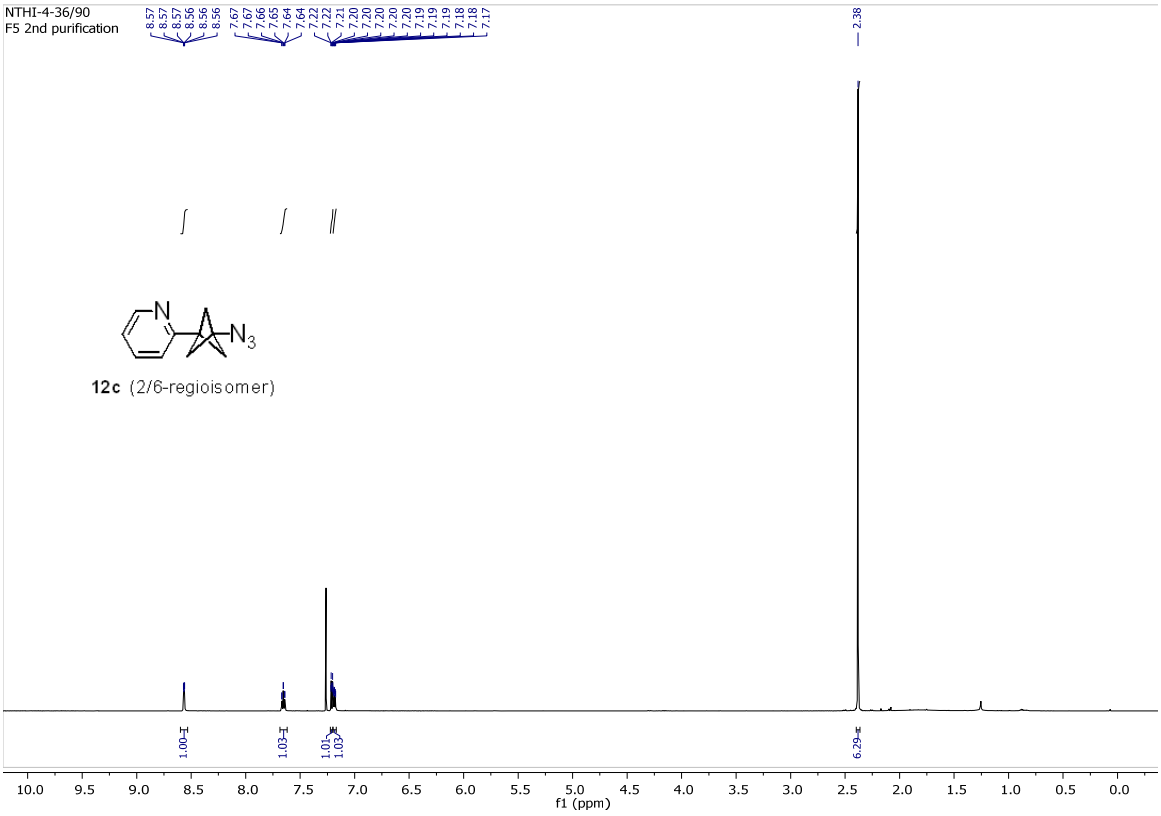
12b

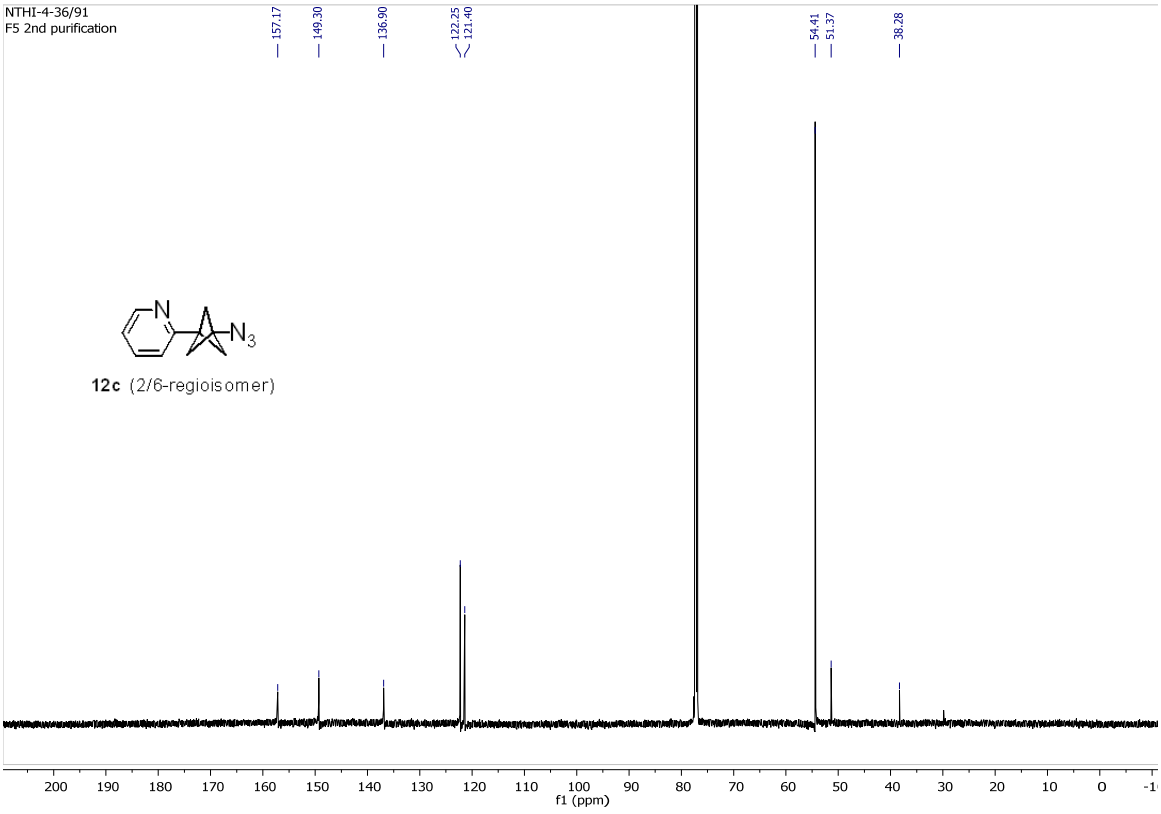

NTHI-3-119/71
Band 2
160.99
137.46
128.59
127.43
126.55
104.74
77.37
77.16
76.95
66.52
55.24
49.84
38.50
15.00
f1 (ppm)

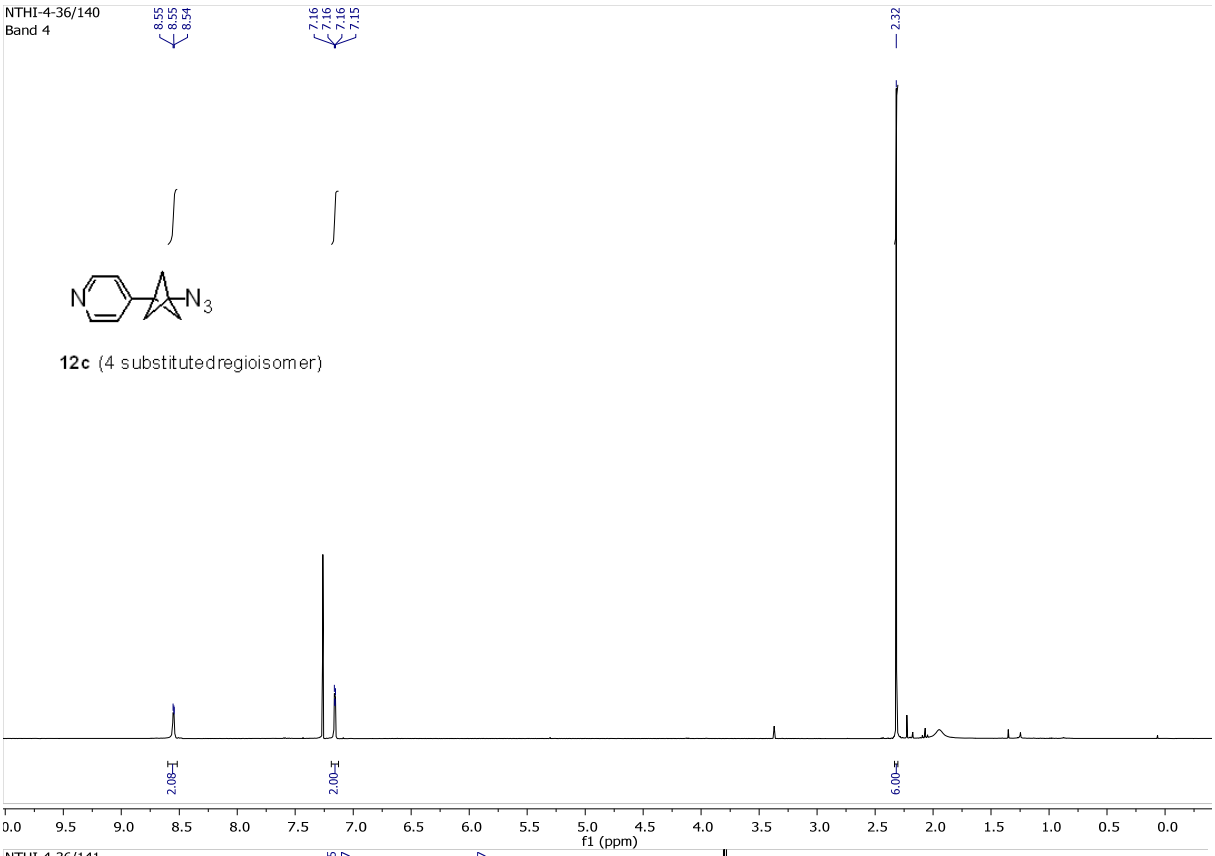
12b


NTHI-4-36/150
Pure Band 3
8.48
8.48
8.47
8.46
8.46
8.46
7.70
7.70
7.70
7.69
7.69
7.69
7.68
7.68
7.36
7.36
7.36
7.35
7.35
7.34
7.34
2.34
2.28
1.95
1.94
1.94
1.94
1.93
N
N3
12c (3/5-regioisomer)
0.85
1.01
0.90
0.91
6.00
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)

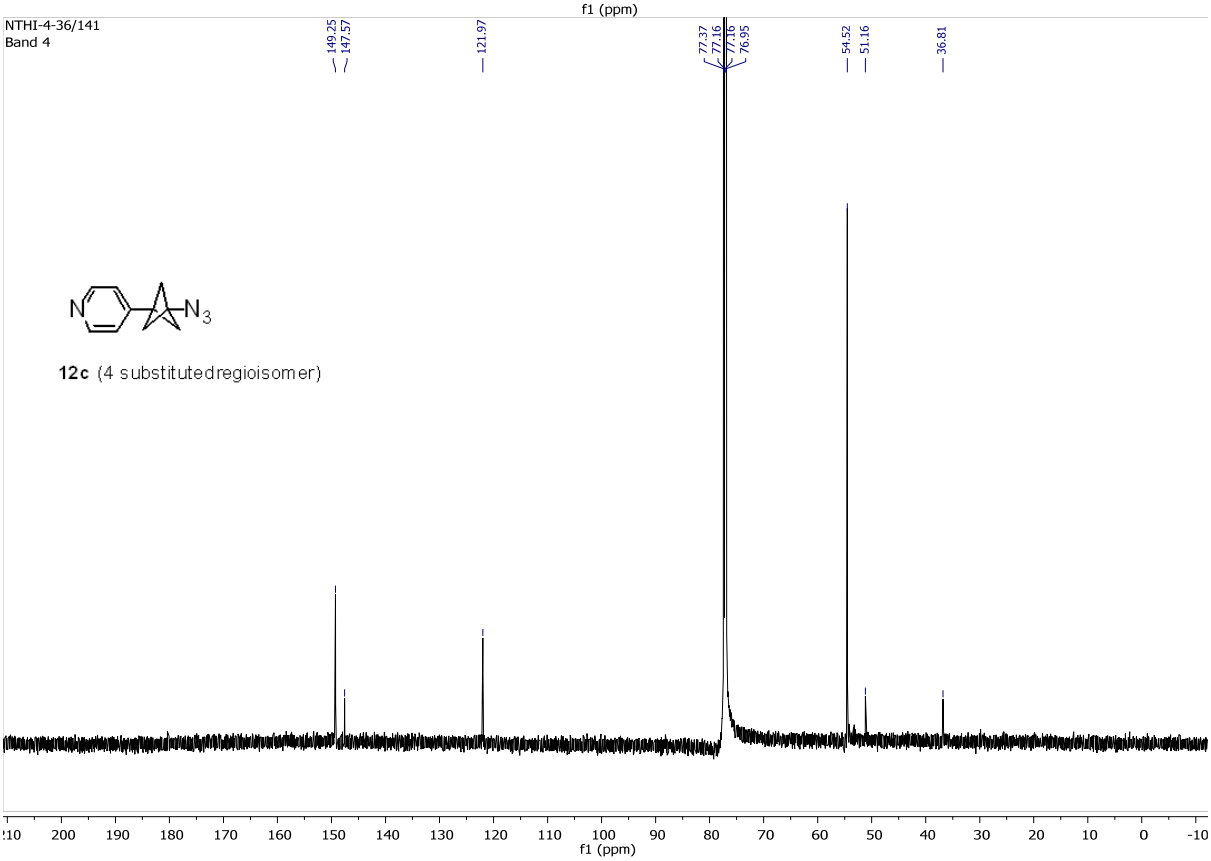

NTHI-4-36/131
Band 3
147.94
147.67
134.65
133.84
123.45
77.37
77.16
76.95
54.65
51.17
35.67
N
N3
12c (3/5-regioisomer)
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


NTHI-4-36/90
F5 2nd purification
8.57
8.57
8.57
8.56
8.56
8.56
7.67
7.67
7.66
7.65
7.64
7.64
7.22
7.21
7.20
7.20
7.20
7.20
7.20
7.19
7.19
7.19
7.18
7.18
7.17
2.38
N
N3
12c (2/6-regioisomer)
1.00
1.03
1.01
1.03
6.29
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)

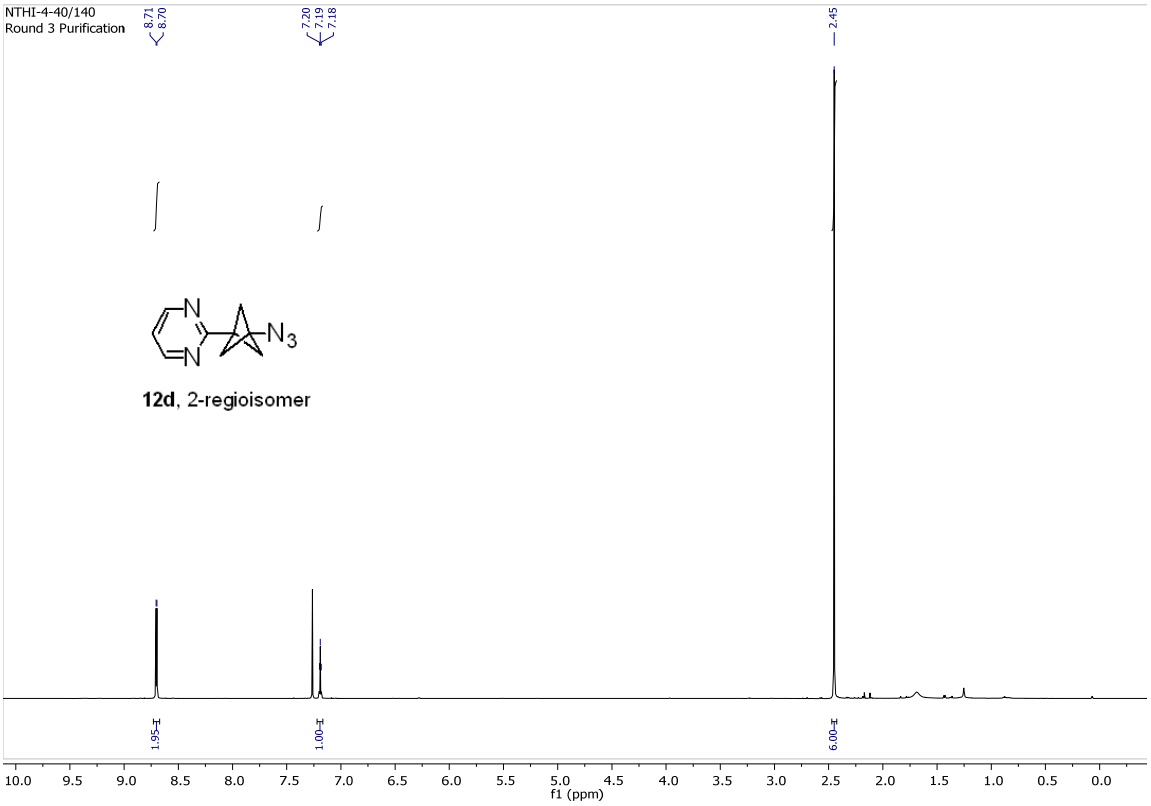

NTHI-4-36/91
F5 2nd purification
157.17
149.30
136.90
122.25
121.40
54.41
51.37
38.28
N
N3
12c (2/6-regioisomer)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

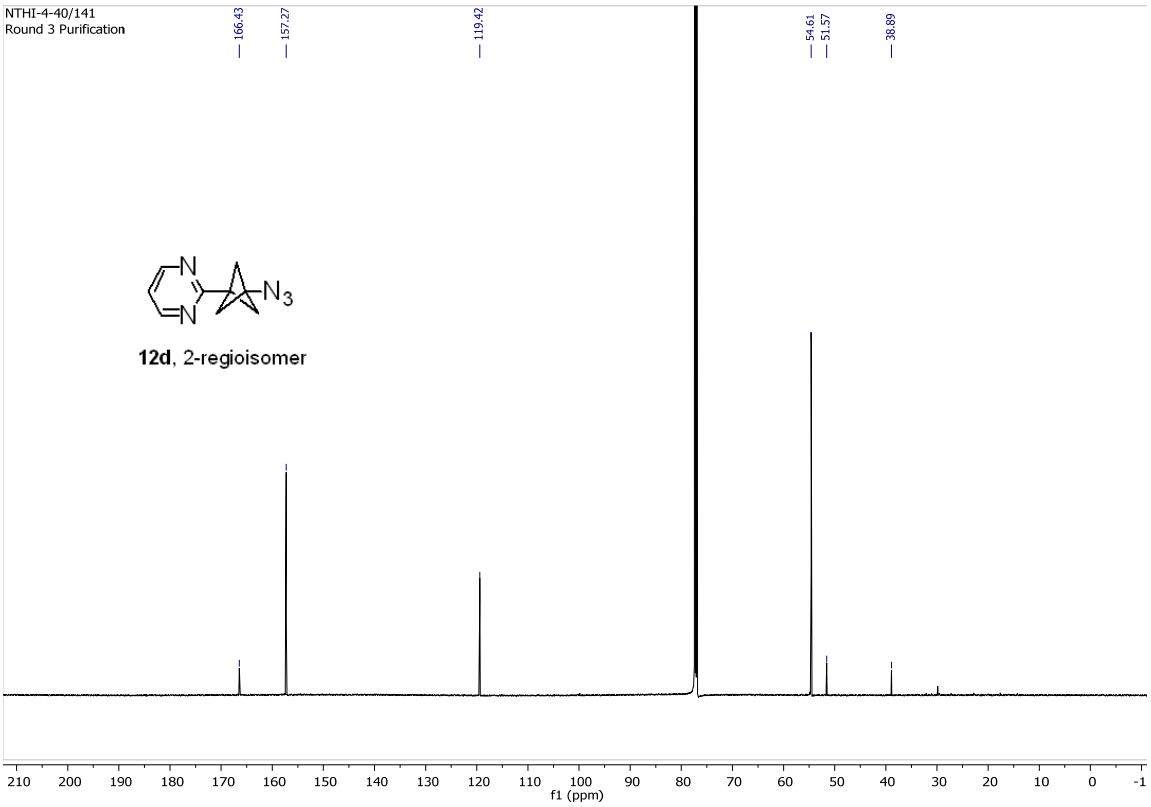


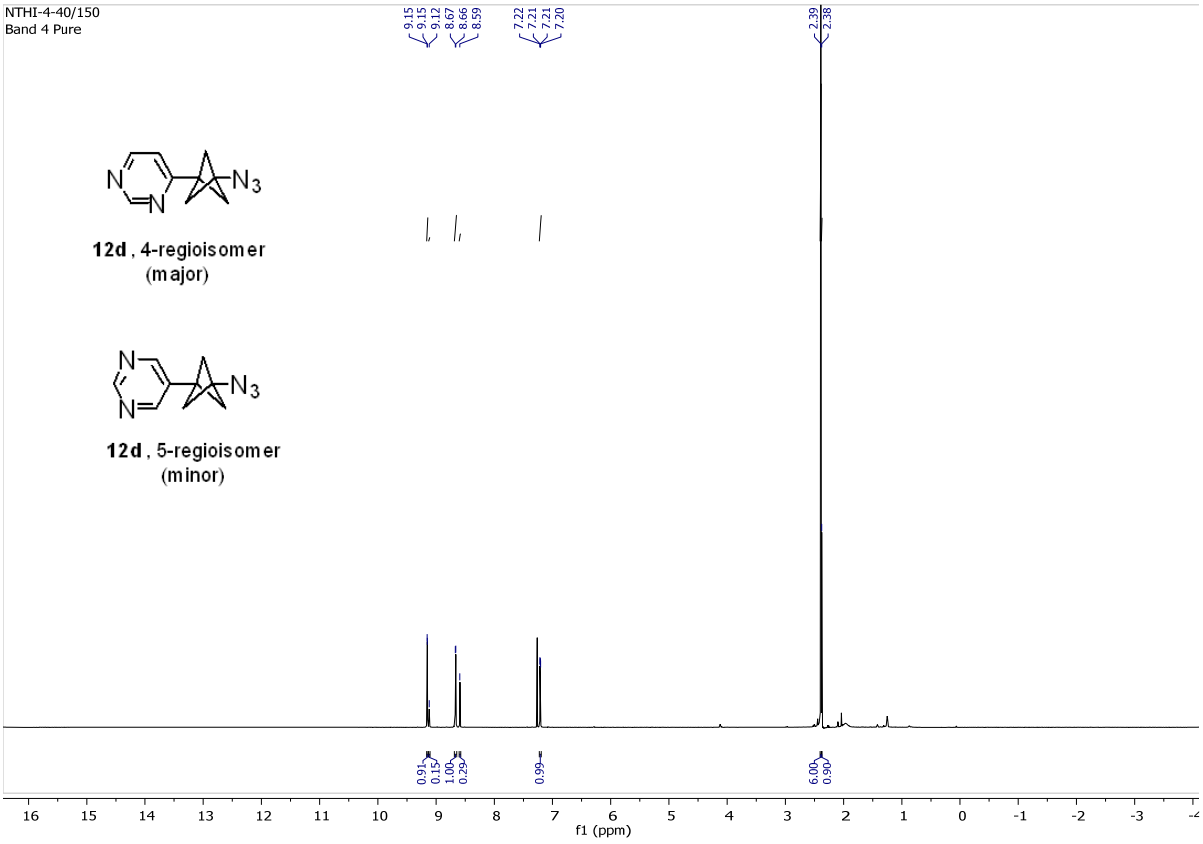
12c (4 substitutedregioisomer)

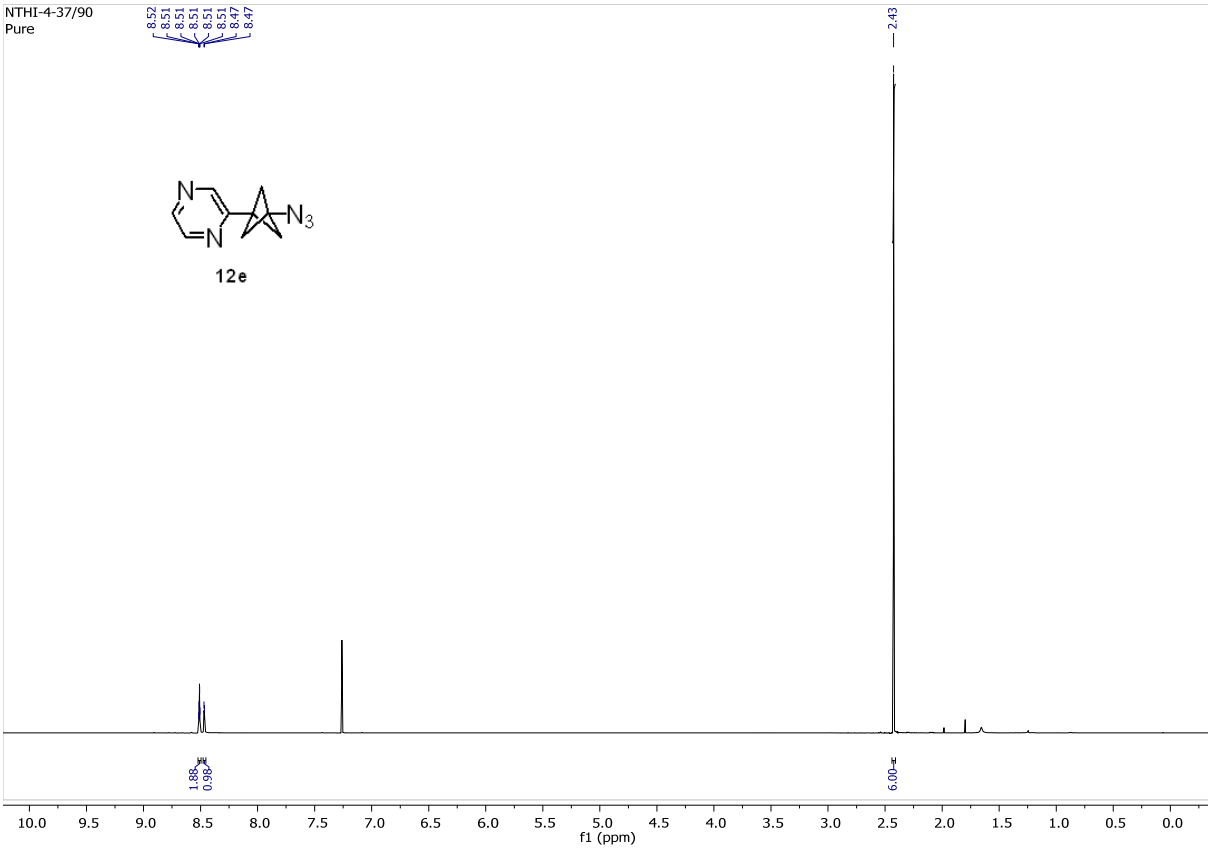


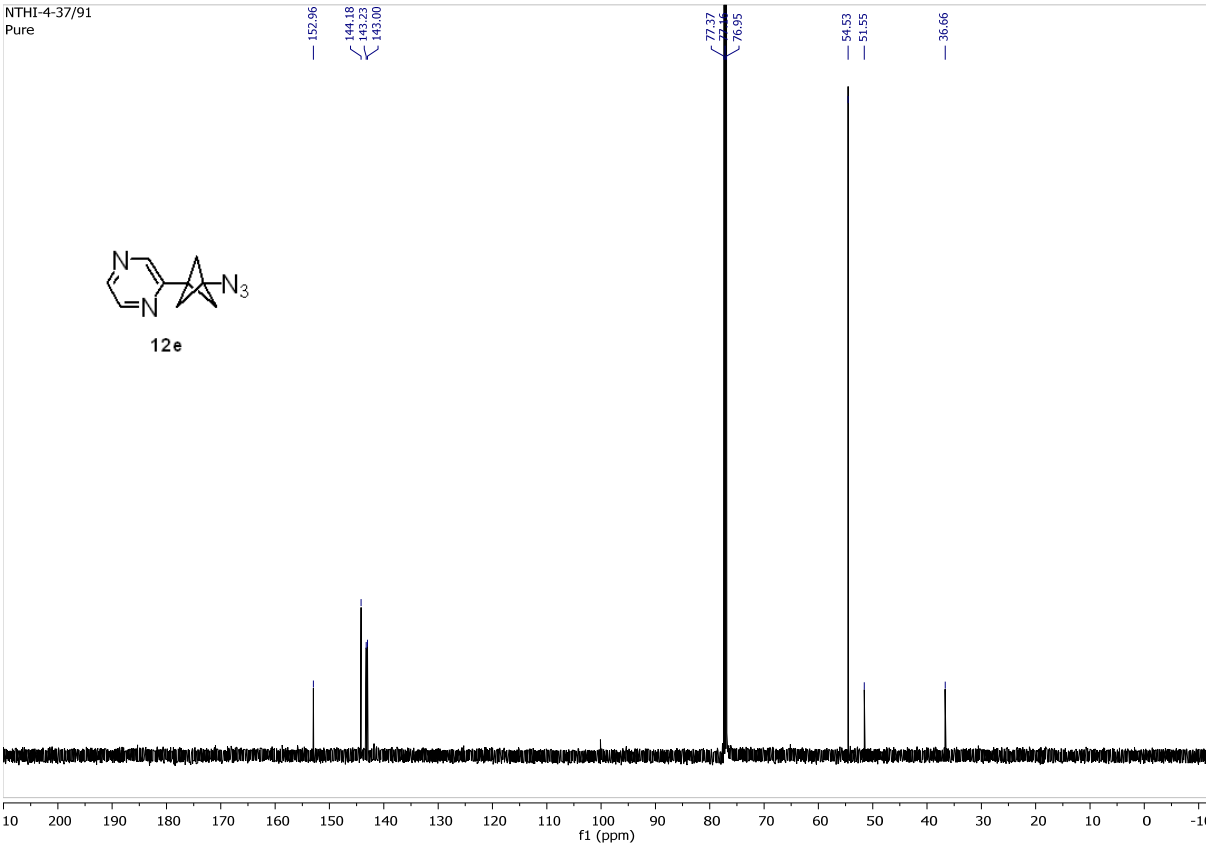
12c (4 substitutedregioisomer)


NTHI-4-40/140
Round 3 Purification
8.71
8.70
7.20
7.19
7.18
2.45
N
N
N3
12d, 2-regioisomer
1.95
1.00
6.00
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)


NTHI-4-40/141
Round 3 Purification
166.43
157.27
119.42
54.61
51.57
38.89
N
N
N3
12d, 2-regioisomer
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


NTHI-4-40/150
Band 4 Pure
9.15
9.15
9.12
8.67
8.66
8.59
7.22
7.21
7.21
7.20
2.39
2.38
N3
12d , 4-regioisomer
(major)
N3
12d , 5-regioisomer
(minor)
0.91
0.15
1.00
0.29
0.99
6.00
0.90
f1 (ppm)


NTHI-4-37/90
Pure
8.52
8.51
8.51
8.51
8.51
8.51
8.47
8.47
2.43
N3
12e
1.88
0.98
6.00
f1 (ppm)


NTHI-4-37/91
Pure
152.96
144.18
143.23
143.00
77.37
76.95
54.53
51.55
36.66
N
N
N3
12e
f1 (ppm)