Supplementary Information

Synthesis of ^{15}N-Labeled Heterocycles via the Cleavage of C-N Bonds of Anilines and Glycine-^{15}N

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Supplementary Data

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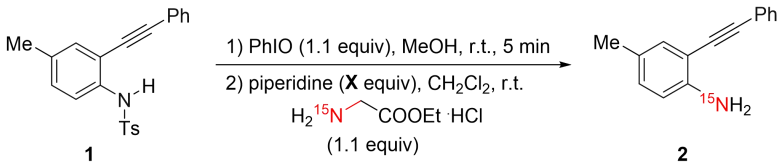
1. General Information

All reactions were performed in Schlenk tubes. Flash column chromatography was performed using silica gel (60-A pore size, 32–63 μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr (house vacuum) at 25–35 °C. Commercial reagents and solvents were used as received. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale.

2. Evaluation of Conditions

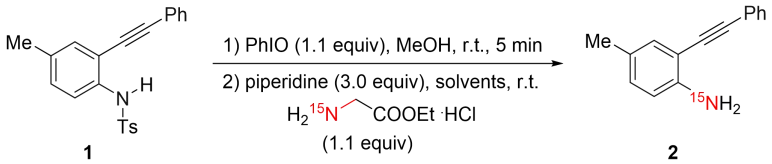
Table 1 Evaluation of ratio of secondary amine



entry	Secondary amine (equiv)	solvent	**2** yield[a] (%)
1	Piperidine (0.2)	DCM	<5
2	Piperidine (0.5)	DCM	12
3	Piperidine (1.0)	DCM	18
4	Piperidine (1.5)	DCM	52
5	Piperidine (2.0)	DCM	72
6	Piperidine (2.5)	DCM	82
7	Piperidine (3.0)	DCM	94
8	Piperidine (3.5)	DCM	88

[a]isolated yield.

Table 2 Evaluation of solvents



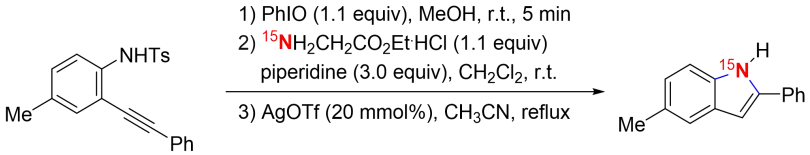
entry	Secondary amine (equiv)	solvent	**2** yield[a] (%)
1	Piperidine (3.0)	CH_3CN	88
2	Piperidine (3.0)	DCE	86
3	Piperidine (3.0)	1,4-Dioxane	81
4	Piperidine (3.0)	EA	80

5	Piperidine（3.0）	THF	77
6	Piperidine（3.0）	$CHCl_3$	80
7	Piperidine（3.0）	DMF	87
8	Piperidine（3.0）	DCM	94

[a]isolated yield.

3. Experimental Procedures

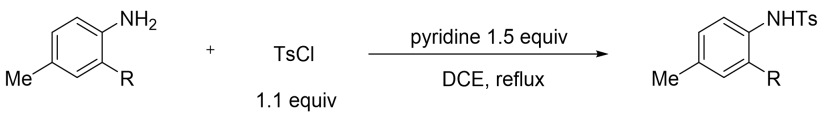
Representative Procedure



1 **4**

PhIO (0.22 mmol) was added to a solution of N-Ts *p*-toluidine **1** (0.2 mmol) in MeOH (2.0 mL) at 25 °C. After 5 min, the reaction mixture was concentrated in vacuo. The resulting mixture was mixed with the Ethyl glycinate-^{15}N(1.1 equiv) and piperidine(3.0 equiv) in DCM (2.0 mL) at 25 °C under air atmosphere for 4 h. Then, the reaction mixture was passed through a short silica gel column to remove piperidine with DCM as flushing agent and then concentrated in vacuo. The resulting crude product was mixed with the AgOTf (0.04 mmol) in MeCN (2 mL). The reaction was stirred at 80 °C for 12 h. After the intermediate was consumed completely (monitored by TLC analysis). The mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (petroleum ether/DCM = 2/1) to furnish the desired compound **4**.

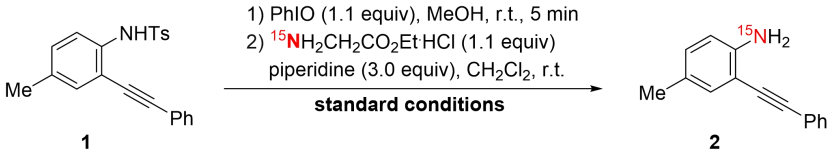
The procedure for synthesis of a series of starting material 1



To a stirred solution of arylamine (1.0 mmol) in DCE (10 mL) was added TsCl (1.1 equiv) and pyridine (1.5 equiv). The resulted mixture was stirred at 80 °C for 4 h. Then the reaction was cooled down to room

temperature and quenched by saturated $CuSO_4$ solution and the reaction mixture was extracted with CH_2Cl_2 (3 x 5 mL). The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated. The residue was purified by flash column chromatography on silica gel (petroleum ether /ethyl acetate = 5/1) to furnish the desired compound .

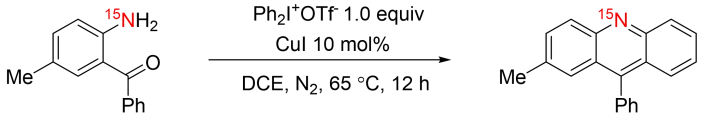
The procedure of standard conditions for synthesis of 2



1 **2**

PhIO (0.22 mmol) was added to a solution of N-Ts *p*-toluidine **1** (0.2 mmol) in MeOH (2.0 mL) at 25 °C. After 5 min, the reaction mixture was concentrated in vacuo. The resulting mixture was mixed with the Ethyl glycinate-^{15}N(1.1 equiv) and piperidine(3.0 equiv) in DCM (2.0 mL) at 25 °C under air atmosphere for 4 h. Then, the reaction mixture was passed through a short silica gel column to remove piperidine with DCM as flushing agent and then concentrated in vacuo to furnish the crude compound **2**.

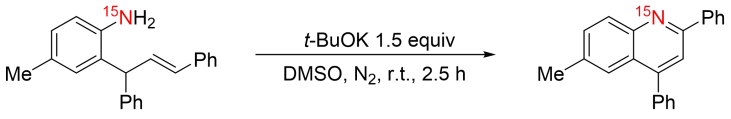
The procedure[1] for synthesis of compound 33



Pre.33 **33**

After standard conditions, the crude precursor of **33**(**Pre.33**) and CuI(0.02 mmol), and diaryliodonium salt(0.20 mmol) in a Schlenk tube. The tube was evacuated and recharged with N_2 for 3 times. After dichloroethane (2.0 mL) was added, the tube was sealed and the mixture was allowed to stir at 65 °C for 12 h until the complete consumption of starting material observed by TLC. 2N K_2CO_3 aq. was added and the mixture was extracted with EA (5 mL x 3). Combined the organic layer and dried over anhydrous Na_2SO_4. Evaporation of the solvent followed by purification on silica gel (petroleum ether/ethyl acetate = 5/1) provided the corresponding product **33**.

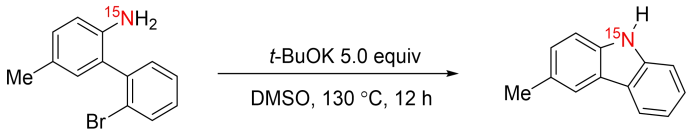
The procedure[2] for synthesis of compound 34



Pre.34 **34**

After standard conditions, the crude precursor of **34**(**Pre.34**) and *t*-BuOK (1.5 equiv) in DMSO (2.0 mL) were taken in a Schlenk tube. The tube containing the reaction mixture was purged with nitrogen for 5 min and allowed to stir at room temperature until the complete consumption of starting material observed by TLC. The reaction mixture was diluted with $NaHCO_3$ (5 mL) followed by washing with ethyl acetate (3 x 10 mL). The organic extract was dried over anhydrous Na_2SO_4. The solvents were removed under reduced pressure to provide the crude product **34** which was purified by flash column chromatography on silica gel using petroleum ether /ethyl acetate as eluent.

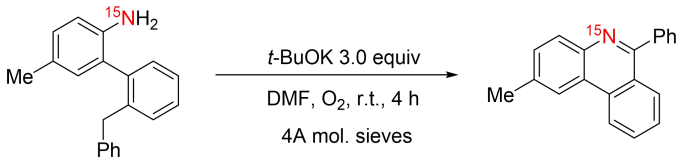
The procedure[3] for synthesis of compound 35



Pre.35 **35**

After standard conditions[a], the crude precursor of **35**(**Pre.35**) and potassium tert-butoxide (1.0 mmol) were taken in a Schlenk tube. The tube was first evacuated and then filled with nitrogen gas. Then 3 mL of DMSO solvent was added and heated at 130 °C for 12 h. After completion of the reaction, the reaction mixture was cooled to room temperature and then diluted with water and extracted with ethyl acetate (3 x 20 mL). The combined organic layer was washed with brine, dried over anhydrous Na_2SO_4, evaporated under reduced pressure. Then the crude product was purified by column chromatography using silica gel and petroleum ether/DCM = 2/1 as eluent to afford the pure product **35**.([a]The step 2 of standard conditions demanded 80 °C in MeCN (2.0 mL) under air atmosphere for 6 h to desire higher yield. Then the MeCN was evaporated in vacuo and the residue was passed through a short silica gel column to remove piperidine with DCM as flushing agent.)

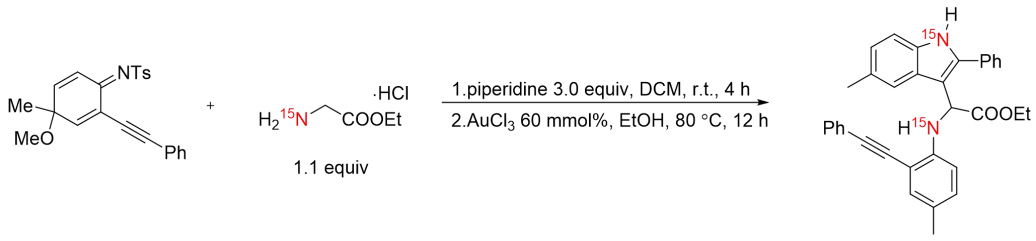
The procedure[4] for synthesis of compound 36



Pre.36 **36**

After standard conditions[a], the crude precursor of **36**(**Pre.36**) and *t*-BuOK (3.0 equiv) in a 1-neck round bottom flask equipped with a magnetic stir bar and 4A-molecular sieves. The flask was then evacuated and backfilled with O_2 three times and the vessel was equipped with an inlet of O_2 for the remainder of the reaction. To this mixture was then added DMF (1.0 mL per 100 mg of aniline) dropwise at room temperature and the mixture was allowed to stir vigorously. Upon completion of the reaction (monitored by TLC), the mixture was directly purified by silica gel chromatography (petroleum ether/ethyl acetate) to afford the desired product **36**. ([a]The step 2 of standard conditions demanded 80 °C in MeCN (2.0 mL) under air atmosphere for 6 h to desire higher yield. Then the MeCN was evaporated in vacuo and the residue was passed through a short silica gel column to remove piperidine with DCM as flushing agent.)

The procedure for synthesis of compound 3



3

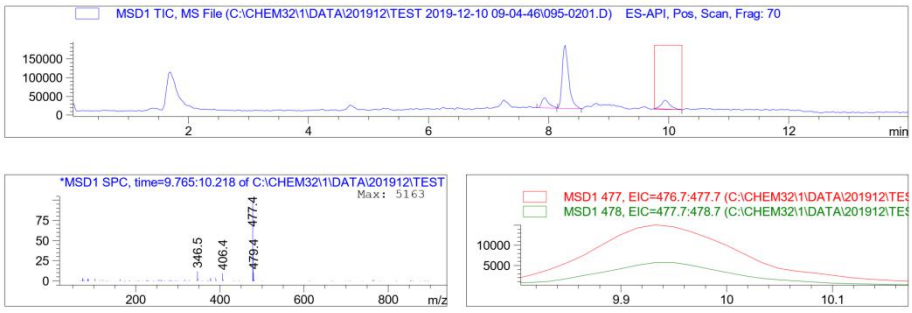
Ethyl glycinate-^{15}N (1.1 equiv) and piperidine (3.0 equiv) were added to a solution of 2-Alynycyclohexadienimines (0.4 mmol) in DCM (4.0 mL) at 25 °C under air atmosphere for 4 h. Then,

the reaction mixture was concentrated in vacuo to remove the solvent DCM. The resulting crude product was mixed with the $AuCl_3$(60 mmol%) in EtOH (4.0 mL). The reaction was stirred at 80 °C for 12 h. After the intermediate was consumed completely (monitored by TLC analysis). The mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate = 5/1) to furnish the desired compound **3**.

Supplementary References

1. X. Pang, Z. Lou, M. Li, L. Wen and C. Chen, *Eur. J. Org. Chem.*, 2015, **2015**, 3361-3369.
2. M. Rehan, G. Hazra and P. Ghorai, *Org. Lett.*, 2015, **17**, 1668-1671.
3. R. Singha, A. Ahmed, Y. Nuree, M. Ghosh and J. K. Ray, *RSC Advances*, 2015, **5**, 50174-50177.
4. C. J. Evoniuk, G. D. P. Gomes, S. P. Hill, S. Fujita, K. Hanson and I. V. Alabugin, *J. Am. Chem. Soc.*, 2017, **139**, 16210-16221.

4. LC-MS analysis of intermediates II and VI[a]




```
Peak #3 at  9.943 min ( 9.759 to 10.218 min)
-> The analysis found only one component, indicating a pure peak. <-

Component 1:  Peak at Scan 589.3. Top ions are  477 478
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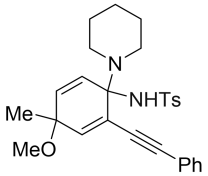
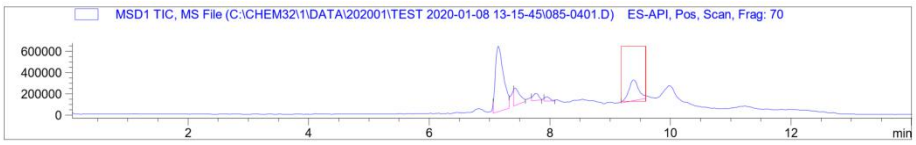
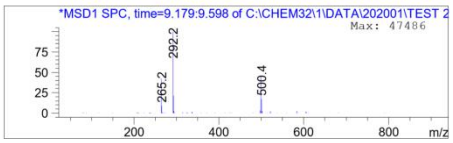


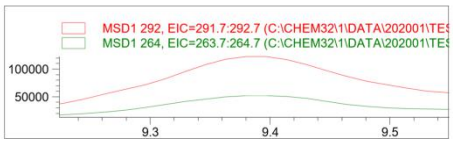
II

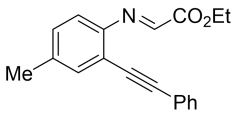
confirmed by LC-MS

Exact Mass: 476.2134









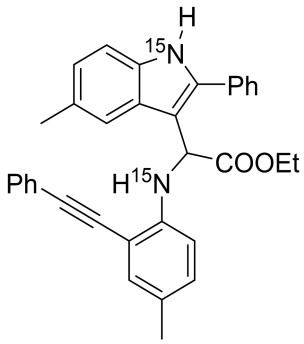
VI[a]

confirmed by LC-MS

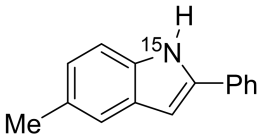
Exact Mass: 291.1259

[a]The reaction was analyzed by LC-MS using unlabeled glycine ethyl ester hydrochloride.

5. Characterization of Products

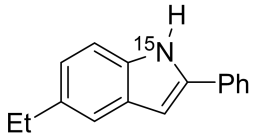


Ethyl 2-((4-methyl-2-(phenylethynyl)phenyl)amino-^{15}N)-2-(5-methyl-2-phenyl-1*H*-indol-3-yl-^{15}N)acetate 3: 150.1 mg, 30% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.07 (d, *J* = 96.2 Hz, 1H), 7.83 (s, 1H), 7.77 – 7.68 (m, 2H), 7.50 (t, *J* = 7.5 Hz, 2H), 7.44 (d, *J* = 7.3 Hz, 1H), 7.40 – 7.35 (m, 2H), 7.28 – 7.22 (m, 4H), 7.12 (d, *J* = 2.0 Hz, 1H), 7.01 (d, *J* = 8.3 Hz, 1H), 6.80 (dd, *J* = 8.4, 2.1 Hz, 1H), 6.16 (d, *J* = 8.3 Hz, 1H), 5.91 (d, *J* = 86.1 Hz, 1H), 5.49 (s, 1H), 4.30 (dq, *J* = 10.6, 7.1 Hz, 1H), 4.21 – 4.10 (m, 1H), 2.29 (s, 3H), 2.14 (s, 3H), 1.26 – 1.22 (m, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.2, 145.0 (d, *J* = 14.6 Hz), 137.4 (d, *J* = 14.5 Hz), 134.3 (d, *J* = 15.5 Hz), 132.2, 132.0, 131.3, 130.3, 129.8, 128.9, 128.8, 128.5, 128.2, 127.8, 126.8, 125.9, 124.2 (d, *J* = 2.0 Hz), 123.4, 120.0, 110.7, 110.1 (d, *J* = 1.8 Hz), 108.2 (d, *J* = 2.3 Hz), 108.0 (d, *J* = 4.4 Hz), 95.1, 86.1, 61.5, 53.8 (d, *J* = 10.7 Hz), 21.5, 20.2, 14.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{34}H_{31}{}^{15}N_2O_2$, 501.2321; found, 501.2311.

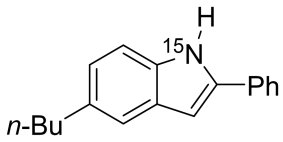


5-methyl-2-phenyl-1*H*-indole-^{15}N 4: 38.3 mg, 92% yield. Colorless crystal, mp:215-216 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.15 (dd, *J* = 95.3, 2.1 Hz, 1H), 7.62 – 7.57 (m, 2H), 7.43 – 7.36 (m, 3H), 7.31 – 7.26 (m, 1H), 7.24 (d, *J* = 8.3 Hz, 1H), 7.00 (dd, *J* = 8.3, 1.6 Hz, 1H), 6.72 (ddd, *J* = 4.3, 2.1, 1.0

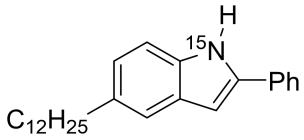
Hz, 1H), 2.43 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 137.9 (d, *J* = 14.4 Hz), 135.1 (d, *J* = 15.8 Hz), 132.4 (d, *J* = 2.2 Hz), 129.5 (d, *J* = 4.6 Hz), 129.4, 128.9, 127.5, 125.0 (d, *J* = 1.4 Hz), 123.9 (d, *J* = 2.1 Hz), 120.3, 110.5 (d, *J* = 1.8 Hz), 99.5 (d, *J* = 4.0 Hz), 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{15}H_{14}{}^{15}N$, 209.1091; found, 209.1084.



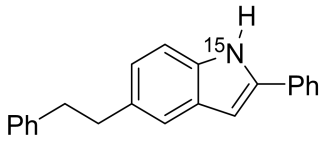
5-ethyl-2-phenyl-1*H*-indole-^{15}*N* 5: 41.8 mg, 94% yield. White solid, mp:149-150 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 95.4, 2.1 Hz, 1H), 7.65 – 7.60 (m, 2H), 7.45 – 7.38 (m, 3H), 7.33 – 7.26 (m, 2H), 7.05 (dd, *J* = 8.3, 1.6 Hz, 1H), 6.76 (ddd, *J* = 4.4, 2.2, 0.9 Hz, 1H), 2.74 (q, *J* = 7.6 Hz, 2H), 1.29 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 137.9 (d, *J* = 14.2 Hz), 136.2, 135.3 (d, *J* = 15.9 Hz), 132.5, 129.5 (d, *J* = 4.4 Hz), 129.0, 127.5, 125.0 (d, *J* = 1.4 Hz), 123.0 (d, *J* = 2.1 Hz), 119.0, 110.6 (d, *J* = 1.7 Hz), 99.7 (d, *J* = 3.9 Hz), 29.0, 16.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{16}H_{16}{}^{15}N$, 223.1248; found, 223.1252.



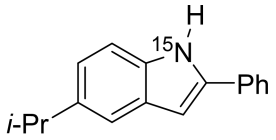
5-butyl-2-phenyl-1*H*-indole-^{15}*N* 6: 46.5 mg, 93% yield. Pale yellow solid, mp:132-133 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.22 (ddd, *J* = 95.4, 2.2, 0.8 Hz, 1H), 7.65 – 7.61 (m, 2H), 7.44 – 7.39 (m, 3H), 7.32 – 7.27 (m, 2H), 7.02 (dd, *J* = 8.2, 1.6 Hz, 1H), 6.75 (ddd, *J* = 4.4, 2.2, 0.9 Hz, 1H), 2.74 – 2.66 (m, 2H), 1.70 – 1.60 (m, 2H), 1.38 (dq, *J* = 14.7, 7.4 Hz, 2H), 0.94 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 137.9 (d, *J* = 14.3 Hz), 135.3 (d, *J* = 15.9 Hz), 134.8, 132.5 (d, *J* = 2.2 Hz), 129.4 (d, *J* = 4.3 Hz), 128.9, 127.5, 125.0 (d, *J* = 1.3 Hz), 123.4 (d, *J* = 2.1 Hz), 119.7, 110.5 (d, *J* = 1.7 Hz), 99.6 (d, *J* = 3.9 Hz), 35.8, 34.4, 22.4, 14.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{18}H_{20}{}^{15}N$, 251.1561; found, 251.1568.



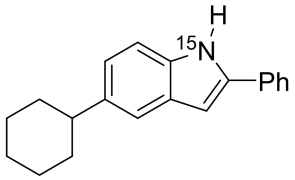
5-dodecyl-2-phenyl-1*H*-indole-^{15}*N* 7: 65.2 mg, 90% yield. Colorless crystal, mp:134-135 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 (dd, *J* = 95.4, 2.1 Hz, 1H), 7.65 – 7.60 (m, 2H), 7.44 – 7.38 (m, 3H), 7.32 – 7.26 (m, 2H), 7.02 (dd, *J* = 8.3, 1.6 Hz, 1H), 6.75 (dd, *J* = 4.5, 2.1 Hz, 1H), 2.72 – 2.65 (m, 2H), 1.70 – 1.61 (m, 2H), 1.25 (s, 18H), 0.88 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 137.9 (d, *J* = 14.4 Hz), 135.3 (d, *J* = 15.9 Hz), 134.8, 132.5, 129.4 (d, *J* = 4.3 Hz), 128.9, 127.5, 125.0 (d, *J* = 1.4 Hz), 123.4 (d, *J* = 2.1 Hz), 119.7, 110.5 (d, *J* = 1.6 Hz), 99.6 (d, *J* = 4.0 Hz), 36.1, 32.3, 31.9, 29.7, 29.7, 29.6, 29.4, 29.4, 22.7, 14.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{26}H_{36}{}^{15}N$, 363.2813; found, 363.2815.



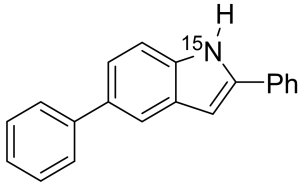
5-phenethyl-2-phenyl-1*H*-indole-^{15}N 8: 49.5 mg, 83% yield. Pale yellow solid, mp:178-179 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.22 (dd, *J* = 95.4, 2.1 Hz, 1H), 7.65 – 7.60 (m, 2H), 7.45 – 7.38 (m, 3H), 7.33 – 7.25 (m, 4H), 7.24 – 7.17 (m, 3H), 7.03 (dd, *J* = 8.3, 1.6 Hz, 1H), 6.75 (dd, *J* = 4.5, 2.1 Hz, 1H), 3.04 – 2.94 (m, 4H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 142.2, 138.0 (d, *J* = 14.3 Hz), 135.4 (d, *J* = 15.8 Hz), 133.7, 132.4 (d, *J* = 2.2 Hz), 129.5 (d, *J* = 4.4 Hz), 129.0, 128.5, 128.3, 127.6, 125.8, 125.1 (d, *J* = 1.3 Hz), 123.4 (d, *J* = 2.1 Hz), 119.8, 110.6 (d, *J* = 1.7 Hz), 99.7 (d, *J* = 4.0 Hz), 38.8, 38.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{22}H_{20}{}^{15}N$, 299.1561; found, 299.1564.



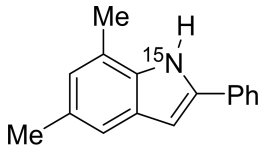
5-isopropyl-2-phenyl-1*H*-indole-^{15}N 9: 43.4 mg, 92% yield. Colorless crystal, mp:170-171 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.22 (dd, *J* = 95.3, 2.1 Hz, 1H), 7.66 – 7.61 (m, 2H), 7.47 (d, *J* = 1.6 Hz, 1H), 7.42 (t, *J* = 7.7 Hz, 2H), 7.33 – 7.27 (m, 2H), 7.09 (dd, *J* = 8.4, 1.6 Hz, 1H), 6.77 (ddd, *J* = 4.4, 2.2, 0.9 Hz, 1H), 3.01 (hept, *J* = 6.9 Hz, 1H), 1.31 (d, *J* = 6.9 Hz, 6H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 141.0, 138.0 (d, *J* = 14.3 Hz), 135.4 (d, *J* = 15.8 Hz), 132.6 (d, *J* = 2.3 Hz), 129.4 (d, *J* = 4.4 Hz), 129.0, 127.5, 125.1 (d, *J* = 1.4 Hz), 121.7 (d, *J* = 2.1 Hz), 117.5, 110.6 (d, *J* = 1.7 Hz), 99.8 (d, *J* = 4.0 Hz), 34.2, 24.6; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{18}{}^{15}N$, 237.1404; found, 237.1407.



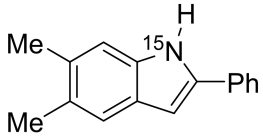
5-cyclohexyl-2-phenyl-1*H*-indole-^{15}N 10: 52.5 mg, 95% yield. Pale yellow solid, mp:178-179 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.21 (dd, *J* = 95.4, 2.1 Hz, 1H), 7.66 – 7.60 (m, 2H), 7.47 – 7.38 (m, 3H), 7.32 – 7.27 (m, 2H), 7.06 (dd, *J* = 8.4, 1.6 Hz, 1H), 6.76 (dd, *J* = 4.6, 2.1 Hz, 1H), 2.62 – 2.54 (m, 1H), 1.97 – 1.73 (m, 5H), 1.55 – 1.42 (m, 3H), 1.41 – 1.26 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 140.3, 137.9 (d, *J* = 14.3 Hz), 135.4 (d, *J* = 16.0 Hz), 132.5, 129.4 (d, *J* = 4.4 Hz), 128.9, 127.5, 125.1 (d, *J* = 1.4 Hz), 122.1 (d, *J* = 2.1 Hz), 117.9, 110.6 (d, *J* = 1.7 Hz), 99.8 (d, *J* = 4.0 Hz), 44.7, 35.1, 27.1, 26.3; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{22}{}^{15}N$, 277.1717; found, 277.1721.



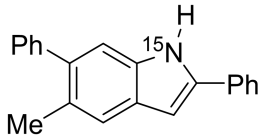
2,5-diphenyl-1*H*-indole-^{15}N 11: 48.6 mg, 90% yield. Pale yellow solid, mp:189-190 ℃. ^{1}H NMR (400 MHz, Acetone-d_6) δ 10.74 (dd, *J* = 97.1, 2.2 Hz, 1H), 7.90 – 7.83 (m, 3H), 7.70 – 7.65 (m, 2H), 7.53 – 7.39 (m, 7H), 7.34 – 7.26 (m, 2H), 6.96 (dd, *J* = 4.4, 2.2 Hz, 1H); ^{13}C NMR (101 MHz, Acetone-d_6) δ 143.2, 139.4 (d, *J* = 14.2 Hz), 137.8 (d, *J* = 15.7 Hz), 133.7, 133.3 (d, *J* = 2.3 Hz), 130.7 (d, *J* = 4.1 Hz), 129.6, 129.4, 128.2, 127.6, 126.9, 125.8 (d, *J* = 1.4 Hz), 122.2 (d, *J* = 2.1 Hz), 119.3, 112.2 (d, *J* = 1.8 Hz), 100.2 (d, *J* = 3.7 Hz); HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{16}{}^{15}N$, 271.1248; found, 271.1241.



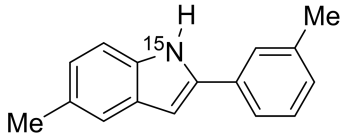
5,7-dimethyl-2-phenyl-1*H*-indole-^{15}N 12: 32.9 mg, 74% yield. Pale yellow solid, mp:76-77 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.1 (ddd, *J* = 94.9, 2.2, 0.8 Hz, 1H), 7.7 (dd, *J* = 8.3, 1.3 Hz, 2H), 7.4 (dd, *J* = 8.5, 7.0 Hz, 2H), 7.3 – 7.3 (m, 1H), 7.3 – 7.2 (m, 1H), 6.8 – 6.8 (m, 1H), 6.7 (dd, *J* = 4.4, 2.2 Hz, 1H), 2.5 (s, 3H), 2.4 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 137.6 (d, *J* = 14.2 Hz), 134.7 (d, *J* = 15.5 Hz), 132.7 (d, *J* = 2.2 Hz), 129.7, 129.1 (d, *J* = 4.5 Hz), 128.9, 127.5, 125.1 (d, *J* = 1.4 Hz), 124.7 (d, *J* = 1.8 Hz), 119.7 (d, *J* = 1.5 Hz), 117.9, 100.1 (d, *J* = 4.0 Hz), 21.4, 16.7; HRMS (m/z): $[M+H]^+$ calcd. for $C_{16}H_{16}{}^{15}N$, 223.1248; found, 223.1252.



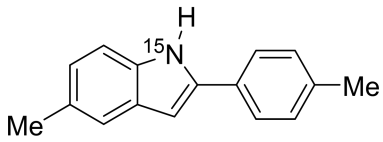
5,6-dimethyl-2-phenyl-1*H*-indole-^{15}N 13: 26.7 mg, 60% yield. Colorless crystal, mp:211-212 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.09 (dd, *J* = 95.2, 2.1 Hz, 1H), 7.60 (d, *J* = 7.7 Hz, 2H), 7.42 – 7.36 (m, 3H), 7.30 – 7.25 (m, 1H), 7.13 (s, 1H), 6.71 (dd, *J* = 4.6, 2.1 Hz, 1H), 2.35 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 137.0 (d, *J* = 14.0 Hz), 135.8 (d, *J* = 15.7 Hz), 132.6, 131.5, 128.9, 128.9, 127.6 (d, *J* = 3.9 Hz), 127.3, 124.9 (d, *J* = 1.4 Hz), 120.7, 111.3, 99.3 (d, *J* = 4.0 Hz); HRMS (m/z): $[M+H]^+$ calcd. for $C_{16}H_{16}{}^{15}N$, 223.1248; found, 223.1252.



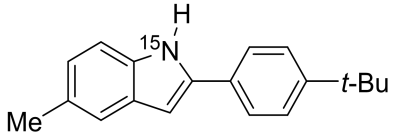
5-methyl-2,6-diphenyl-1*H*-indole-^{15}N 14: 37.5 mg, 66% yield. Colorless crystal, mp:194-195 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.2 (dd, *J* = 95.5, 2.1 Hz, 1H), 7.7 – 7.6 (m, 2H), 7.5 (s, 1H), 7.4 – 7.4 (m, 6H), 7.4 – 7.3 (m, 2H), 7.2 (s, 1H), 6.8 – 6.8 (m, 1H), 2.3 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.0, 138.3 (d, *J* = 14.2 Hz), 137.1, 135.5 (d, *J* = 15.8 Hz), 132.4 (d, *J* = 2.2 Hz), 129.5, 129.0, 128.7 (d, *J* = 4.3 Hz), 127.9, 127.6, 127.5, 126.4, 125.1 (d, *J* = 1.5 Hz), 121.3, 111.8 (d, *J* = 1.7 Hz), 99.3 (d, *J* = 3.9 Hz), 20.9; HRMS (m/z): $[M+H]^+$ calcd. for $C_{21}H_{18}{}^{15}N$, 285.1404; found, 285.1405.



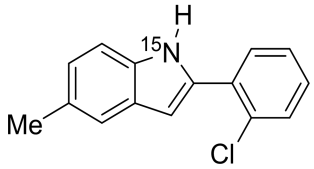
5-methyl-2-(m-tolyl)-1*H*-indole-^{15}N 15: 40.0 mg, 90% yield. Colorless crystal, mp:196-197 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 (dd, *J* = 95.4, 2.2 Hz, 1H), 7.47 – 7.41 (m, 2H), 7.41 – 7.39 (m, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.26 (d, *J* = 8.2 Hz, 1H), 7.12 (d, *J* = 7.5 Hz, 1H), 7.00 (dd, *J* = 8.2, 1.6 Hz, 1H), 6.72 (ddd, *J* = 4.4, 2.2, 0.9 Hz, 1H), 2.44 (s, 3H), 2.41 (s, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 138.6, 138.0 (d, *J* = 14.3 Hz), 135.0 (d, *J* = 15.8 Hz), 132.4 (d, *J* = 2.1 Hz), 129.5 (d, *J* = 4.4 Hz), 129.4, 128.9, 128.4, 125.8 (d, *J* = 1.4 Hz), 123.8 (d, *J* = 2.0 Hz), 122.2 (d, *J* = 1.3 Hz), 120.2, 110.5 (d, *J* = 1.6 Hz), 99.4 (d, *J* = 4.0 Hz), 21.5, 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{16}H_{16}{}^{15}N$, 223.1248; found, 223.1247.



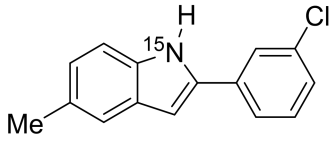
5-methyl-2-(p-tolyl)-1*H*-indole-^{15}N 16: 32.4 mg, 73% yield. Colorless crystal, mp:201-202 ℃. ^{1}H NMR (400 MHz, Acetone-d_6) δ 10.47 (dd, J = 96.8, 2.2 Hz, 1H), 7.71 (d, J = 8.2 Hz, 2H), 7.32 (s, 1H), 7.28 (d, J = 8.3 Hz, 1H), 7.23 (d, J = 8.2 Hz, 2H), 6.92 (dd, J = 8.3, 1.6 Hz, 1H), 6.76 – 6.71 (m, 1H), 2.38 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (101 MHz, Acetone-d_6) δ 138.9 (d, J = 14.3 Hz), 137.6, 136.5 (d, J = 15.6 Hz), 130.8 (d, J = 2.3 Hz), 130.4 (d, J = 4.0 Hz), 130.2, 129.0, 125.6 (d, J = 1.3 Hz), 123.9 (d, J = 2.0 Hz), 120.5, 111.5 (d, J = 1.8 Hz), 98.8 (d, J = 3.7 Hz), 21.4, 21.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{16}H_{16}{}^{15}N$, 223.1248; found, 223.1247.



2-(4-(tert-butyl)phenyl)-5-methyl-1*H*-indole-^{15}N 17: 49.7 mg, 94% yield. Colorless crystal, mp:219-220 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.19 (dd, J = 95.3, 2.1 Hz, 1H), 7.57 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8.4 Hz, 2H), 7.39 (d, J = 1.6 Hz, 1H), 7.26 (d, J = 8.3 Hz, 1H), 7.00 (d, J = 8.3 Hz 1H), 6.73 – 6.68 (m, 1H), 2.44 (s, 3H), 1.35 (s, 9H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 150.7, 138.0 (d, J = 14.4 Hz), 135.0 (d, J = 15.9 Hz), 129.7 (d, J = 2.1 Hz), 129.6 (d, J = 4.6 Hz), 129.3, 125.9, 124.8 (d, J = 1.3 Hz), 123.7 (d, J = 2.1 Hz), 120.2, 110.4 (d, J = 1.7 Hz), 99.0 (d, J = 3.9 Hz), 34.6, 31.3, 21.5; HRMS (m/z): $[M+H]^+$ calcd. for $C_{19}H_{22}{}^{15}N$, 265.1717; found, 265.1718.

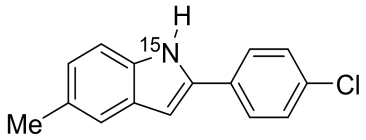


2-(2-chlorophenyl)-5-methyl-1*H*-indole-^{15}N 18: 32.9 mg, 68% yield. Colorless crystal, mp:172-173 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.62 (dd, J = 96.5, 2.2 Hz, 1H), 7.62 (dd, J = 7.7, 1.7 Hz, 1H), 7.47 – 7.41 (m, 2H), 7.32 – 7.25 (m, 2H), 7.22 (td, J = 7.7, 1.7 Hz, 1H), 7.04 (dd, J = 8.3, 1.6 Hz, 1H), 6.79 – 6.74 (m, 1H), 2.44 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 135.1 (d, J = 14.6 Hz), 134.7 (d, J = 15.7 Hz), 131.2 (d, J = 1.8 Hz), 131.2, 130.8, 130.6 (d, J = 1.3 Hz), 129.3, 128.6, 128.4 (d, J = 4.2 Hz), 127.1, 124.3 (d, J = 2.1 Hz), 120.3, 110.7 (d, J = 1.8 Hz), 103.0 (d, J = 3.9 Hz), 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{15}H_{13}Cl{}^{15}N$, 243.0701; found, 243.0702.

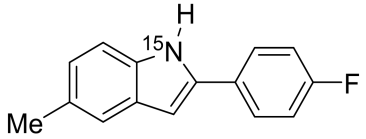


2-(3-chlorophenyl)-5-methyl-1*H*-indole-^{15}N 19: 32.9 mg, 68% yield. Colorless crystal, mp:208-209 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.12 (dd, J = 95.4, 2.1 Hz, 1H), 7.57 (d, J = 1.4 Hz, 1H), 7.45 (dt, J = 7.8, 1.4 Hz, 1H), 7.39 (s, 1H), 7.31 (t, J = 7.8 Hz, 1H), 7.27 – 7.22 (m, 2H), 7.02 (d, J = 8.3 Hz, 1H), 6.72 (dd, J = 4.5, 2.1 Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 136.3 (d, J = 14.5 Hz), 135.3 (d, J = 15.7 Hz), 134.9, 134.2, 130.2, 129.7, 129.3 (d, J = 4.2 Hz), 127.4,

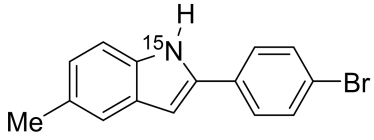
125.0, 124.5, 123.0, 120.4, 110.7, 100.5 (d, J = 4.0 Hz), 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{15}H_{13}Cl^{15}N$, 243.0701; found, 243.0701.



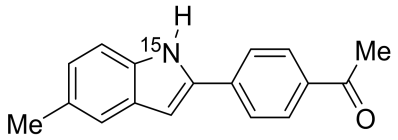
2-(4-chlorophenyl)-5-methyl-1*H*-indole-^{15}N 20: 37.8 mg, 78% yield. Colorless crystal, mp:214-215 ℃. ^{1}H NMR (400 MHz, Acetone-d_6) δ 10.57 (ddd, J = 96.8, 2.3, 0.8 Hz, 1H), 7.86 – 7.80 (m, 2H), 7.48 – 7.42 (m, 2H), 7.34 (s, 1H), 7.29 (dd, J = 8.2, 0.8 Hz, 1H), 6.96 (dd, J = 8.3, 1.6 Hz, 1H), 6.82 (ddd, J = 4.1, 2.3, 0.8 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (101 MHz, Acetone-d_6) δ 137.4 (d, J = 14.4 Hz), 136.7 (d, J = 15.6 Hz), 133.0, 132.4 (d, J = 2.4 Hz), 130.3 (d, J = 4.0 Hz), 129.6, 129.3, 127.2 (d, J = 1.8 Hz), 124.5 (d, J = 2.1 Hz), 120.7, 111.6 (d, J = 1.7 Hz), 100.0 (d, J = 3.6 Hz), 21.4. HRMS (m/z): $[M+H]^+$ calcd. for $C_{15}H_{13}Cl^{15}N$, 243.0701; found, 243.0701.



2-(4-fluorophenyl)-5-methyl-1*H*-indole-^{15}N 21: 32.6 mg, 72% yield. White solid, mp:169-170 ℃. ^{1}H NMR (400 MHz, Acetone-d_6) δ 10.51 (ddd, J = 96.8, 2.2, 0.8 Hz, 1H), 7.88 – 7.82 (m, 2H), 7.34 – 7.32 (m, 1H), 7.31 – 7.27 (m, 1H), 7.23 – 7.16 (m, 2H), 6.95 (dd, J = 8.3, 1.6 Hz, 1H), 6.75 (ddd, J = 4.1, 2.2, 0.8 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (101 MHz, Acetone-d_6) δ 162.8 (d, J (C,F) = 244.9 Hz), 137.7 (d, J = 14.3 Hz), 136.6 (d, J = 15.6 Hz), 130.3 (d, J = 4.1 Hz), 130.2 – 130.1 (m), 129.2, 127.6 (dd, J = 8.0, 1.4 Hz), 124.2 (d, J = 2.1 Hz), 120.6, 116.4 (d, J = 21.8 Hz), 111.6 (d, J = 1.9 Hz), 99.4 (dd, J = 3.8, 1.4 Hz), 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{15}H_{13}F^{15}N$, 227.0997; found, 227.0999.

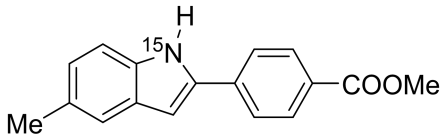


2-(4-bromophenyl)-5-methyl-1*H*-indole-^{15}N 22: 38.9 mg, 68% yield. Pale yellow solid, mp:184-185 ℃. ^{1}H NMR (400 MHz, Acetone-d_6) δ 10.59 (dd, J = 96.9, 2.3 Hz, 1H), 7.78 (d, J = 8.6 Hz, 2H), 7.60 (d, J = 8.6 Hz, 2H), 7.35 (s, 1H), 7.29 (d, J = 8.3 Hz, 1H), 6.96 (dd, J = 8.3, 1.6 Hz, 1H), 6.86 – 6.82 (m, 1H), 2.38 (s, 3H); ^{13}C NMR (101 MHz, Acetone-d_6) δ 137.4 (d, J = 14.6 Hz), 136.7 (d, J = 15.6 Hz), 132.8 (d, J = 2.4 Hz), 132.6, 130.2 (d, J = 4.0 Hz), 129.3, 127.4 (d, J = 1.8 Hz), 124.5 (d, J = 2.2 Hz), 121.1, 120.7, 111.7 (d, J = 1.7 Hz), 100.1 (d, J = 3.7 Hz), 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{15}H_{13}Br^{15}N$, 287.0196; found, 287.0197.

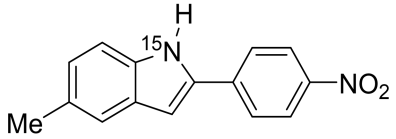


1-(4-(5-methyl-1*H*-indol-2-yl-^{15}N)phenyl)ethan-1-one 23: 23.0 mg, 46% yield. Colorless crystal, mp:197-198 ℃. ^{1}H NMR (400 MHz, Acetone-d_6) δ 10.74 (dd, J = 97.0, 2.3 Hz, 1H), 8.04 (d, J = 8.5 Hz, 2H), 7.96 (d, J = 8.5 Hz, 2H), 7.38 (s, 1H), 7.33 (d, J = 8.3 Hz, 1H), 7.02 – 6.95 (m, 2H), 2.59 (s, 3H), 2.39 (s, 3H); ^{13}C NMR (101 MHz, Acetone-d_6) δ 197.0, 137.6 (d, J = 2.3 Hz), 137.3 (d, J = 14.3 Hz), 137.1 (d, J = 15.6 Hz), 136.3, 130.1 (d, J = 4.0 Hz), 129.7, 129.5, 125.3 (d, J = 1.4 Hz), 125.0 (d,

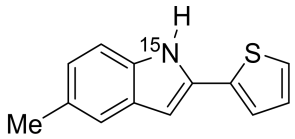
J = 2.1 Hz), 120.9, 111.8 (d, *J* = 1.7 Hz), 101.4 (d, *J* = 3.7 Hz), 26.5, 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{16}{}^{15}NO$, 251.1197; found, 251.1198.



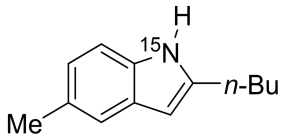
methyl 4-(5-methyl-1*H*-indol-2-yl-^{15}*N*)benzoate 24: 36.7 mg, 69% yield. Colorless crystal, mp:216-217 ℃. ^{1}H NMR (400 MHz, Acetone-d_6) δ 10.72 (d, *J* = 97.0 Hz, 1H), 8.05 (dd, *J* = 8.0, 1.0 Hz, 2H), 7.97 – 7.93 (m, 2H), 7.39 – 7.36 (m, 1H), 7.33 (d, *J* = 8.3 Hz, 1H), 7.02 – 6.96 (m, 2H), 3.89 (d, *J* = 1.0 Hz, 3H), 2.39 (s, 3H); ^{13}C NMR (101 MHz, Acetone-d_6) δ 166.8, 137.8, 137.3 (d, *J* = 14.6 Hz), 137.1 (d, *J* = 15.6 Hz), 130.7, 130.2 (d, *J* = 3.8 Hz), 129.5, 129.2, 125.4 (d, *J* = 1.4 Hz), 125.1 (d, *J* = 2.0 Hz), 121.0, 111.8 (d, *J* = 1.7 Hz), 101.4 (d, *J* = 3.7 Hz), 52.2, 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{17}H_{16}{}^{15}NO_2$, 267.1146; found, 267.1147.



5-methyl-2-(4-nitrophenyl)-1*H*-indole-^{15}*N* 25: 29.4 mg, 58% yield. Colorless crystal, mp:214-215 ℃. ^{1}H NMR (400 MHz, Acetone-d_6) δ 10.84 (dd, *J* = 97.1, 2.2 Hz, 1H), 8.28 (d, *J* = 8.9 Hz, 2H), 8.06 (d, *J* = 8.9 Hz, 2H), 7.40 (s, 1H), 7.34 (d, *J* = 8.3 Hz, 1H), 7.10 – 7.07 (m, 1H), 7.03 (dd, *J* = 8.3, 1.5 Hz, 1H), 2.40 (s, 3H); ^{13}C NMR (101 MHz, Acetone-d_6) δ 147.0, 139.7 (d, *J* = 2.4 Hz), 137.4 (d, *J* = 15.5 Hz), 136.1 (d, *J* = 14.7 Hz), 130.1 (d, *J* = 3.9 Hz), 129.9, 125.9 (d, *J* = 1.4 Hz), 125.8 (d, *J* = 2.2 Hz), 124.9, 121.2, 112.0 (d, *J* = 1.9 Hz), 103.0 (d, *J* = 3.7 Hz), 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{15}H_{13}N^{15}NO_2$, 254.0942; found, 254.0939.

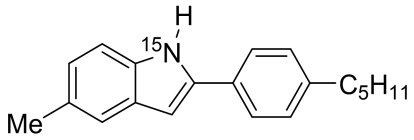


5-methyl-2-(thiophen-2-yl)-1*H*-indole-^{15}*N* 26: 30.8 mg, 72% yield. Colorless crystal, mp:185-186 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.10 (ddd, *J* = 95.8, 2.2, 0.8 Hz, 1H), 7.38 – 7.35 (m, 1H), 7.27 – 7.21 (m, 3H), 7.07 (dd, *J* = 5.1, 3.6 Hz, 1H), 7.03 – 6.98 (m, 1H), 6.64 (ddd, *J* = 4.3, 2.2, 0.8 Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 135.8 (d, *J* = 2.8 Hz), 134.8 (d, *J* = 15.8 Hz), 132.4 (d, *J* = 14.9 Hz), 129.6, 129.3 (d, *J* = 4.4 Hz), 127.8, 124.4, 124.1 (d, *J* = 2.1 Hz), 122.7, 120.2, 110.4 (d, *J* = 1.7 Hz), 100.0 (d, *J* = 3.9 Hz), 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{13}H_{12}{}^{15}NS$, 215.0655; found, 215.0655.

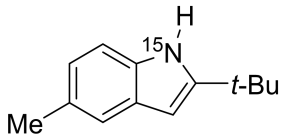


2-butyl-5-methyl-1*H*-indole-^{15}*N* 27: 27.1 mg, 72% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 (d, *J* = 95.2 Hz, 1H), 7.30 (s, 1H), 7.16 (d, *J* = 8.2 Hz, 1H), 6.93 (d, *J* = 8.2 Hz, 1H), 6.17 – 6.10 (m, 1H), 2.72 (td, *J* = 7.7, 2.4 Hz, 2H), 1.73 – 1.63 (m, 2H), 1.46 – 1.34 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 140.0 (d, *J* = 13.2 Hz), 134.0 (d, *J* = 15.8

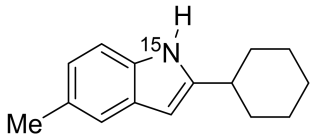
Hz), 129.1 (d, *J* = 4.8 Hz), 128.7, 122.3, 119.4, 109.9, 98.9, 31.3, 28.0 (d, *J* = 2.1 Hz), 22.4, 21.4, 13.9; HRMS (m/z): $[M]^+$ calcd. for $C_{13}H_{17}{}^{15}N$, 188.1331; found, 188.1326.



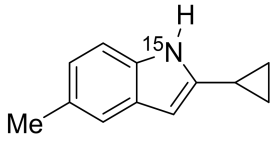
5-methyl-2-(4-pentylphenyl)-1*H*-indole-^{15}N 28: 46.2 mg, 83% yield. Colorless crystal, mp:204-205 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.17 (dd, *J* = 95.3, 2.1 Hz, 1H), 7.54 (d, *J* = 8.2 Hz, 2H), 7.39 (s, 1H), 7.27 – 7.21 (m, 2H), 6.99 (dd, *J* = 8.2, 1.6 Hz, 1H), 6.69 (ddd, *J* = 4.4, 2.1, 0.9 Hz, 1H), 2.66 – 2.59 (m, 2H), 2.44 (s, 3H), 1.68 – 1.59 (m, 2H), 1.38 – 1.30 (m, 4H), 0.94 – 0.86 (m, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 142.6, 138.1 (d, *J* = 14.3 Hz), 135.0 (d, *J* = 15.9 Hz), 129.9 (d, *J* = 2.3 Hz), 129.6 (d, *J* = 4.4 Hz), 129.3, 129.0, 125.0 (d, *J* = 1.4 Hz), 123.7 (d, *J* = 2.0 Hz), 120.1, 110.4 (d, *J* = 1.7 Hz), 99.0 (d, *J* = 4.0 Hz), 35.7, 31.5, 31.1, 22.5, 21.5, 14.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{24}{}^{15}N$, 279.1874; found, 279.1877.



2-(tert-butyl)-5-methyl-1*H*-indole-^{15}N 29: 36.1 mg, 96% yield. Pale yellow solid, mp:94-95 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (dd, *J* = 94.9, 2.1 Hz, 1H), 7.32 (d, *J* = 1.6 Hz, 1H), 7.19 (d, *J* = 8.2 Hz, 1H), 6.94 (dd, *J* = 8.2, 1.6 Hz, 1H), 6.16 (dd, *J* = 4.9, 2.1 Hz, 1H), 2.42 (s, 3H), 1.37 (s, 9H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 148.8 (d, *J* = 12.9 Hz), 134.0 (d, *J* = 15.7 Hz), 128.8 (d, *J* = 4.8 Hz), 128.7, 122.5 (d, *J* = 2.0 Hz), 119.7, 109.9 (d, *J* = 1.6 Hz), 96.4 (d, *J* = 4.1 Hz), 30.3, 21.4; HRMS (m/z): $[M]^+$ calcd. for $C_{13}H_{17}{}^{15}N$, 188.1331; found, 188.1326.

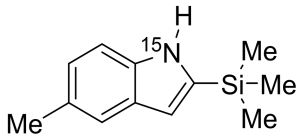


2-cyclohexyl-5-methyl-1*H*-indole-^{15}N 30: 38.5 mg, 90% yield. Pale yellow solid, mp:124-125 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.74 (dd, *J* = 95.1, 2.2 Hz, 1H), 7.31 (s, 1H), 7.15 (d, *J* = 8.2 Hz, 1H), 6.92 (d, *J* = 8.2 Hz, 1H), 6.15 – 6.11 (m, 1H), 2.64 (tt, *J* = 11.0, 2.8 Hz, 1H), 2.41 (s, 3H), 2.07 – 2.01 (m, 2H), 1.86 – 1.80 (m, 2H), 1.77 – 1.70 (m, 1H), 1.50 – 1.29 (m, 5H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.2 (d, *J* = 12.7 Hz), 133.7 (d, *J* = 15.7 Hz), 128.8 (d, *J* = 4.7 Hz), 128.6, 122.3 (d, *J* = 1.9 Hz), 119.6, 109.9 (d, *J* = 1.6 Hz), 96.9 (d, *J* = 4.0 Hz), 37.3 (d, *J* = 1.9 Hz), 32.9, 26.2, 26.1, 21.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{15}H_{20}{}^{15}N$, 215.1561; found, 215.1563.

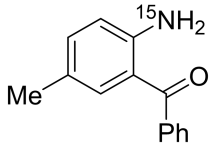


2-cyclopropyl-5-methyl-1*H*-indole-^{15}N 31: 32.4 mg, 94% yield. Pale yellow solid, mp:87-88 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.76 (dd, *J* = 95.4, 2.1 Hz, 1H), 7.27 (s, 1H), 7.12 (d, *J* = 8.2 Hz, 1H), 6.92 (d, *J* = 8.2 Hz, 1H), 6.05 (dd, *J* = 4.5, 2.1 Hz, 1H), 2.41 (s, 3H), 1.90 (tt, *J* = 9.0, 5.4 Hz, 1H), 0.96 – 0.89 (m, 2H), 0.77 – 0.71 (m, 2H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 141.7 (d, *J* = 13.6 Hz), 134.0 (d, *J* = 15.7 Hz), 128.9 (d, *J* = 4.6 Hz), 128.7, 122.4 (d, *J* = 2.0 Hz), 119.4, 109.8 (d, *J* = 1.6 Hz),

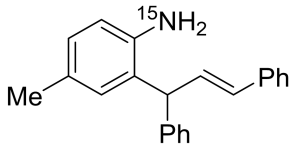
97.1 (d, J = 4.2 Hz), 21.4, 8.9 (d, J = 2.9 Hz), 7.3; HRMS (m/z): $[M+H]^+$ calcd. for $C_{12}H_{14}{}^{15}N$, 173.1091; found, 173.1085.



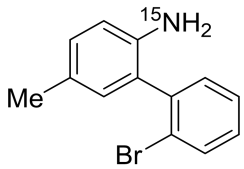
5-methyl-2-(trimethylsilyl)-1*H*-indole-^{15}*N* 32: 28.6 mg, 70% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 (dd, J = 96.0, 2.1 Hz, 1H), 7.39 (s, 1H), 7.27 (d, J = 8.3 Hz, 1H), 7.00 (d, J = 8.2 Hz, 1H), 6.65 – 6.61 (m, 1H), 2.43 (s, 3H), 0.32 (s, 9H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 138.2 (d, J = 7.4 Hz), 136.9 (d, J = 15.0 Hz), 128.9 (d, J = 5.9 Hz), 128.8, 123.9 (d, J = 2.0 Hz), 120.0 (d, J = 1.4 Hz), 110.7 (d, J = 2.2 Hz), 110.4 (d, J = 1.5 Hz), 21.4, -1.1; HRMS (m/z): $[M+H]^+$ calcd. for $C_{12}H_{18}{}^{15}NSi$, 205.1173; found, 205.1174.



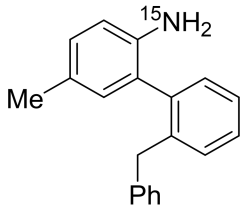
(2-(amino-^{15}*N*)-5-methylphenyl)(phenyl)methanone Pre.33: 195.1 mg, 92% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.66 – 7.61 (m, 2H), 7.55 – 7.49 (m, 1H), 7.45 (dd, J = 8.1, 6.6 Hz, 2H), 7.22 (d, J = 2.0 Hz, 1H), 7.11 (dd, J = 8.4, 2.0 Hz, 1H), 6.66 (dd, J = 8.4, 2.3 Hz, 1H), 5.83 (s, 2H), 2.16 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 199.0, 148.7 (d, J = 13.3 Hz), 140.1, 135.3 (d, J = 1.6 Hz), 134.0, 130.9, 129.0, 128.0, 124.5, 118.1, 117.1 (d, J = 2.2 Hz), 20.3; HRMS (m/z): $[M+H]^+$ calcd. for $C_{14}H_{14}{}^{15}NO$, 213.1040; found, 213.1041.



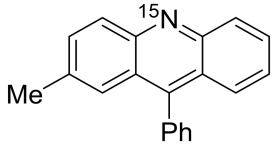
(E)-2-(1,3-diphenylallyl)-4-methylaniline-^{15}*N* Pre.34: 255.1 mg, 85% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 – 7.17 (m, 10H), 6.89 (d, J = 6.3 Hz, 2H), 6.66 (dd, J = 15.9, 7.0 Hz, 1H), 6.60 – 6.54 (m, 1H), 6.26 (d, J = 15.9 Hz, 1H), 4.86 (d, J = 7.0 Hz, 1H), 3.32 (s, 2H), 2.22 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 141.8, 141.6 (d, J = 10.2 Hz), 137.1, 131.5, 131.3, 129.6, 128.7, 128.6, 128.4, 128.2 (d, J = 2.2 Hz), 128.0, 128.0, 127.3, 126.7, 126.3, 116.6 (d, J = 2.2 Hz), 49.5, 20.7; HRMS (m/z): $[M]^+$ calcd. for $C_{22}H_{21}{}^{15}N$, 300.1644; found, 300.1645.



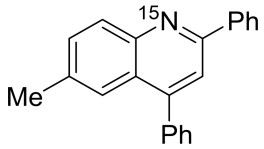
2'-bromo-5-methyl-[1,1'-biphenyl]-2-amine-^{15}*N* Pre.35: 183.4 mg, 70% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.68 (dd, J = 8.0, 1.2 Hz, 1H), 7.41 – 7.35 (m, 1H), 7.33 – 7.29 (m, 1H), 7.26 – 7.20 (m, 1H), 7.03 (dd, J = 8.1, 2.0 Hz, 1H), 6.85 (d, J = 2.0 Hz, 1H), 6.71 (dd, J = 8.1, 1.8 Hz, 1H), 3.31 (s, 2H), 2.28 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 140.8 (d, J = 11.6 Hz), 140.1, 133.0, 131.8, 130.6, 129.6, 129.1, 127.8, 127.7, 127.6, 124.1, 115.7 (d, J = 2.9 Hz), 20.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{13}H_{13}Br{}^{15}N$, 263.0196; found, 263.0198.



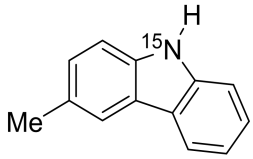
2'-benzyl-5-methyl-[1,1'-biphenyl]-2-amine-^{15}N Pre.36: 227.5 mg, 83% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.28 – 7.10 (m, 7H), 6.98 – 6.93 (m, 3H), 6.72 (d, *J* = 2.0 Hz, 1H), 6.64 (dd, *J* = 8.0, 2.0 Hz, 1H), 3.90 – 3.76 (m, 2H), 3.13 (s, 2H), 2.21 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 141.2 (d, *J* = 11.3 Hz), 141.0, 140.2, 138.7, 130.9, 130.4, 130.1, 129.0, 128.9 (d, *J* = 1.3 Hz), 128.1, 127.8, 127.3, 127.1 (d, *J* = 2.1 Hz), 126.6, 125.7, 115.3 (d, *J* = 3.0 Hz), 39.2, 20.4; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{20}{}^{15}N$, 275.1561; found, 275.1563.



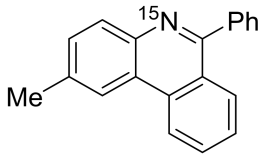
2-methyl-9-phenylacridine-^{15}N 33: 224.2 mg, 76% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.26 (d, *J* = 8.8 Hz, 1H), 8.19 (d, *J* = 8.8 Hz, 1H), 7.73 (dd, *J* = 8.8, 6.6 Hz, 1H), 7.66 (d, *J* = 8.8 Hz, 1H), 7.64 – 7.56 (m, 4H), 7.45 – 7.37 (m, 4H), 2.45 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 148.1, 147.6, 146.1, 136.1, 135.4, 132.9 (d, *J* = 4.1 Hz), 130.4, 129.5 (d, *J* = 3.7 Hz), 129.4, 129.2 (d, *J* = 9.1 Hz), 128.4, 128.2, 126.7, 125.5, 125.3 (d, *J* = 2.3 Hz), 125.1 (d, *J* = 2.3 Hz), 124.7, 22.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{16}{}^{15}N$, 271.1248; found, 271.1249.



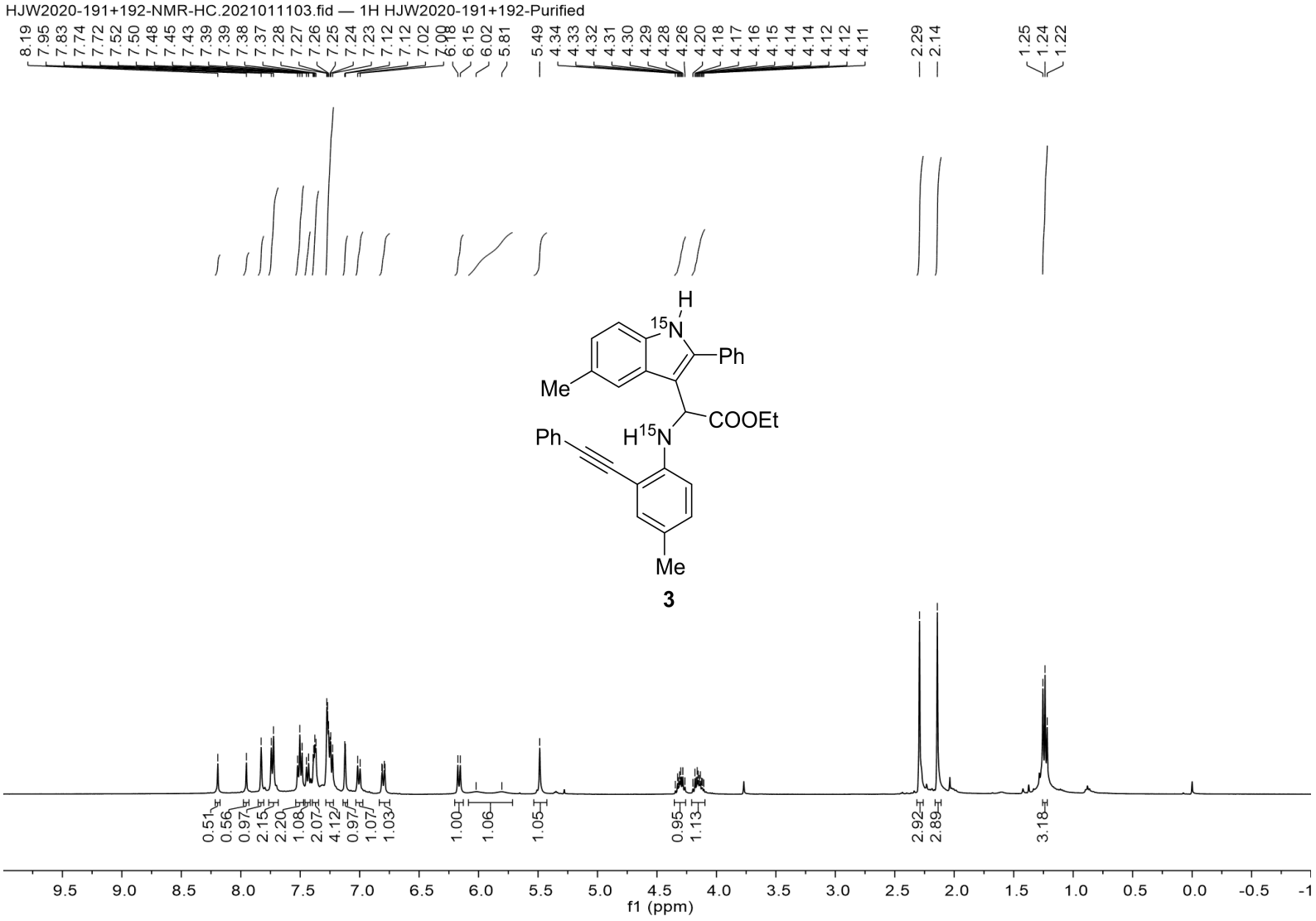
6-methyl-2,4-diphenylquinoline-^{15}N 34: 245.8 mg, 71% yield. Yellow oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 – 8.15 (m, 3H), 7.80 – 7.76 (m, 1H), 7.65 (s, 1H), 7.59 – 7.50 (m, 8H), 7.48 – 7.44 (m, 1H), 2.48 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.9, 147.1, 144.2, 139.5, 138.5, 136.4, 131.9, 129.7 (d, *J* = 9.6 Hz), 129.5, 129.2, 128.8, 128.6, 128.3, 127.5 (d, *J* = 2.4 Hz), 125.7, 124.4, 119.5 (d, *J* = 2.5 Hz), 21.8; HRMS (m/z): $[M+H]^+$ calcd. for $C_{22}H_{18}{}^{15}N$, 297.1404; found, 297.1402.



3-methyl-9*H*-carbazole-^{15}N 35: 158.4 mg, 61% yield. Yellow solid, mp:172-173 ℃. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.06 – 8.00 (m, 1H), 7.87 (s, 1H), 7.77 (s, 1H), 7.42 – 7.34 (m, 2H), 7.31 – 7.18 (m, 3H), 2.52 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 139.7 (d, *J* = 15.6 Hz), 137.6 (d, *J* = 15.8 Hz), 128.7, 127.1 (d, *J* = 1.7 Hz), 125.6, 123.5 (d, *J* = 3.8 Hz), 123.2, 123.2 (d, *J* = 3.6 Hz), 120.2, 119.2, 110.5 (d, *J* = 1.8 Hz), 110.2 (d, *J* = 2.0 Hz), 21.4; HRMS (m/z): $[M]^+$ calcd. for $C_{13}H_{11}{}^{15}N$, 182.0862; found, 182.0858.

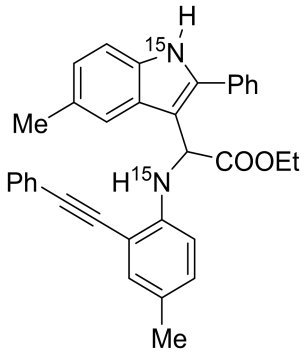


2-methyl-6-phenylphenanthridine-^{15}N 36: 208.0 mg, 64% yield. Colorless oil. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.69 (d, *J* = 8.3 Hz, 1H), 8.40 (s, 1H), 8.15 (dd, *J* = 8.3, 1.9 Hz, 1H), 8.09 (d, *J* = 8.2 Hz, 1H), 7.84 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 1H), 7.76 – 7.71 (m, 2H), 7.62 – 7.51 (m, 5H), 2.65 (s, 3H); ^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.2 (d, *J* = 4.4 Hz), 142.0 (d, *J* = 7.1 Hz), 136.8, 133.2, 130.6 (d, *J* = 3.6 Hz), 130.4, 130.0 (d, *J* = 9.2 Hz), 129.7 (d, *J* = 1.7 Hz), 128.8, 128.6, 128.4, 127.0, 125.2 (d, *J* = 1.8 Hz), 123.5 (d, *J* = 1.6 Hz), 122.1, 121.7 (d, *J* = 5.9 Hz), 121.5, 22.0; HRMS (m/z): $[M+H]^+$ calcd. for $C_{20}H_{16}{}^{15}N$, 271.1248; found, 271.1246.

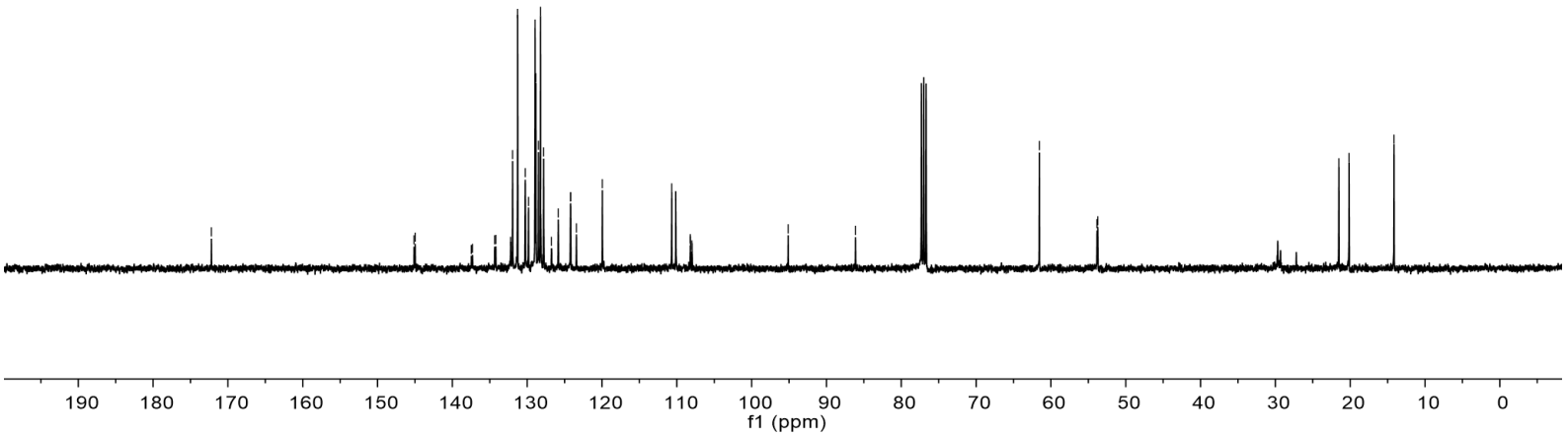

HJW2020-191+192-NMR-HC.2021011103.fid — 1H HJW2020-191+192-Purified
8.19 7.95 7.83 7.74 7.72 7.52 7.50 7.48 7.45 7.43 7.39 7.39 7.38 7.37 7.28 7.27 7.26 7.25 7.24 7.23 7.12 7.12 7.02 7.00 6.18 6.15 6.02 5.81 5.49 4.34 4.33 4.32 4.31 4.30 4.29 4.28 4.26 4.20 4.18 4.17 4.16 4.15 4.14 4.14 4.12 4.12 4.11 2.29 2.14 1.25 1.24 1.22
H
15N
Ph
Me
COOEt
Ph
H15N
Me
3
0.51 0.56 0.97 2.15 2.20 1.08 2.07 4.12 0.97 1.07 1.03 1.00 1.06 1.05 0.95 1.13 2.92 2.89 3.18
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 -0.5 -1
f1 (ppm)

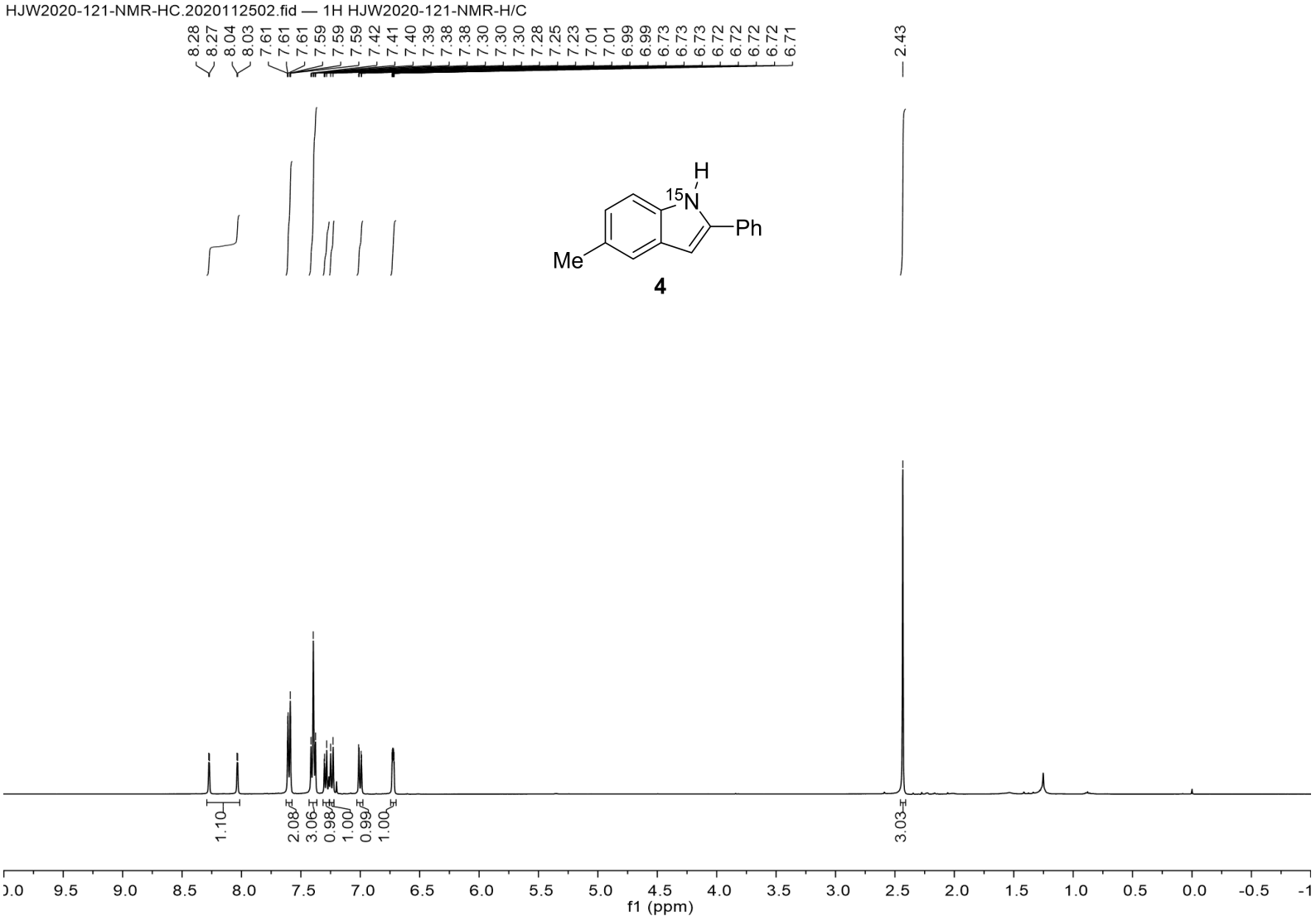
HJW2020-191+192-NMR-HC.2021011104.fid — 13C HJW2020-191+192-Purified

172.21
145.12
144.98
137.47
137.32
134.35
134.20
132.21
131.95
131.28
130.26
129.83
128.95
128.85
128.50
128.21
127.79
126.77
125.85
124.21
124.19
123.42
119.95
110.69
110.14
110.13
108.20
108.17
107.98
107.94
95.12
86.11
61.55
53.84
53.74
21.51
20.15
14.15



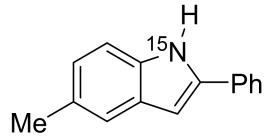
3



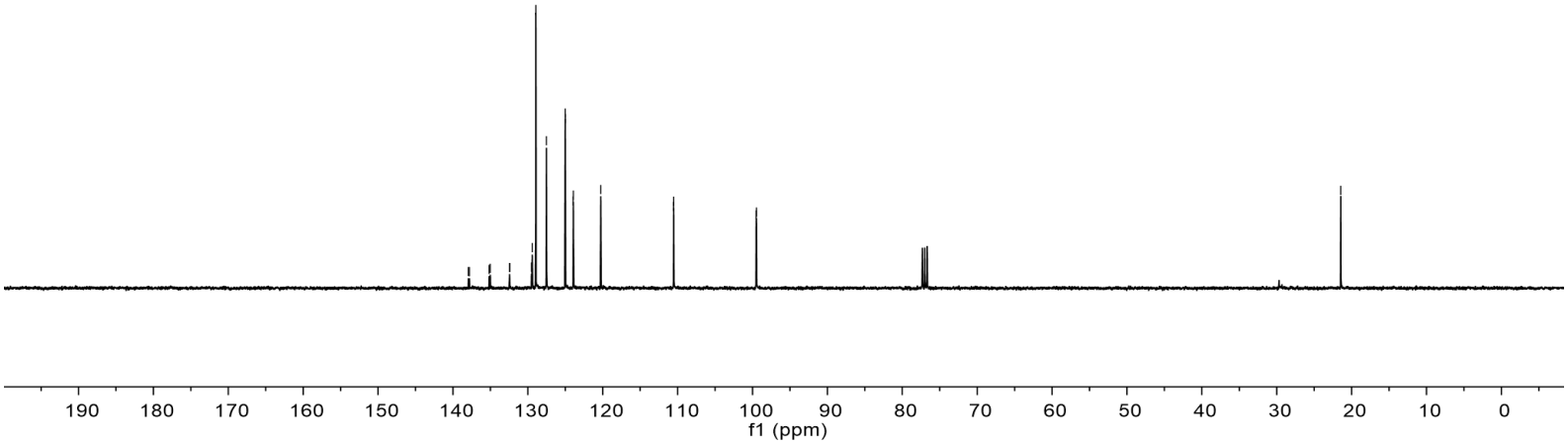

HJW2020-121-NMR-HC.2020112502.fid — 1H HJW2020-121-NMR-H/C
8.28
8.27
8.04
8.03
7.61
7.61
7.61
7.59
7.59
7.59
7.42
7.41
7.40
7.39
7.38
7.38
7.30
7.30
7.30
7.28
7.25
7.23
7.01
7.01
6.99
6.99
6.73
6.73
6.73
6.72
6.72
6.72
6.72
6.71
2.43
H
15N
Ph
Me
4
1.10
2.08
3.06
0.98
1.00
0.99
1.00
3.03
f1 (ppm)

HJW2020-121-NMR-HC.2020112507.fid — 13C HJW2020-121-NMR-H/C

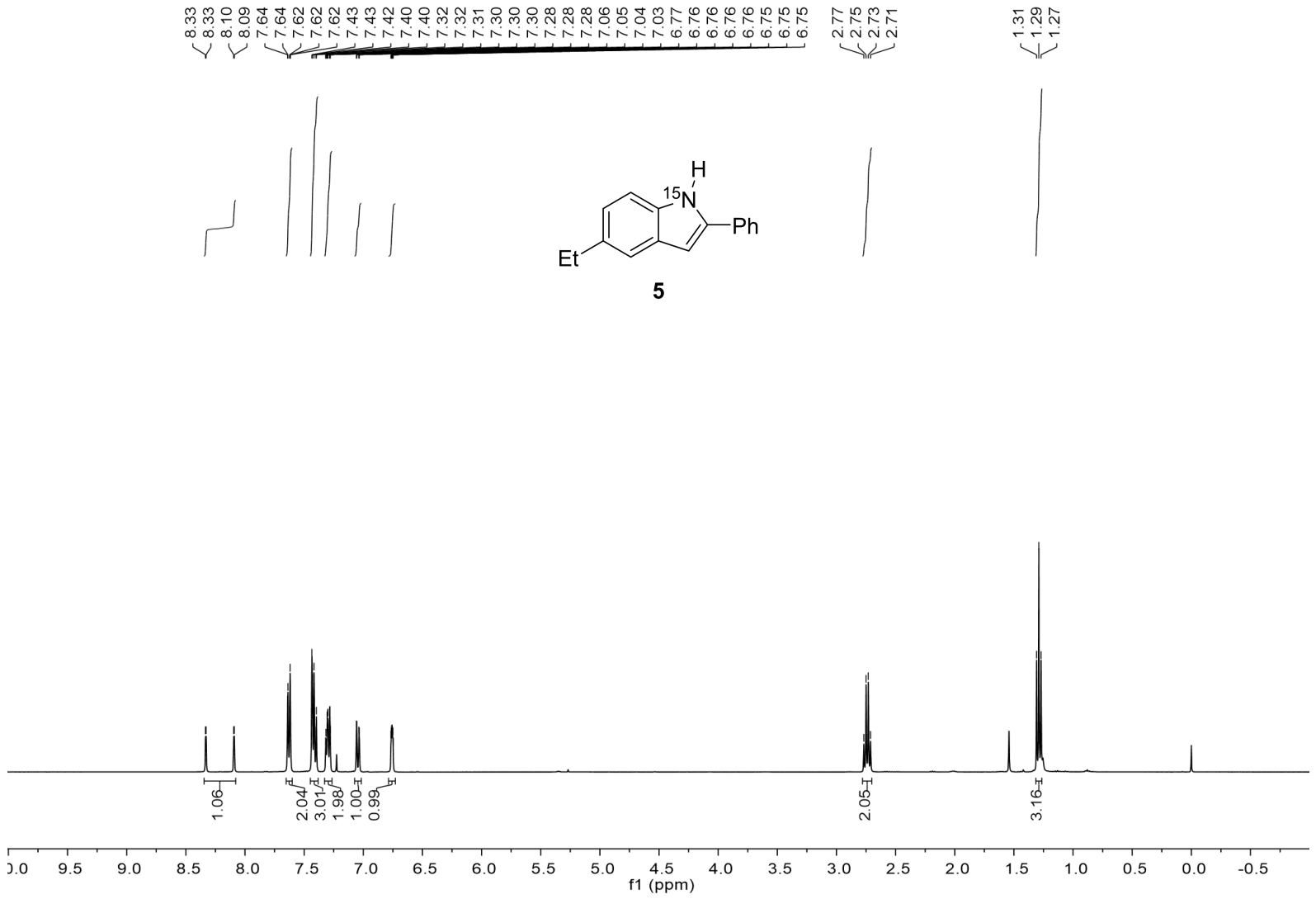
137.95
137.80
135.17
135.02
132.45
132.43
129.52
129.47
129.42
128.93
127.52
125.02
125.00
123.94
123.92
120.26
110.54
110.52
99.51
99.47
21.45



4



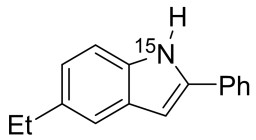
HJW2020-80-NMR-HC.2020111301.fid — 1H HJW2020-80-NMR-H/C-2



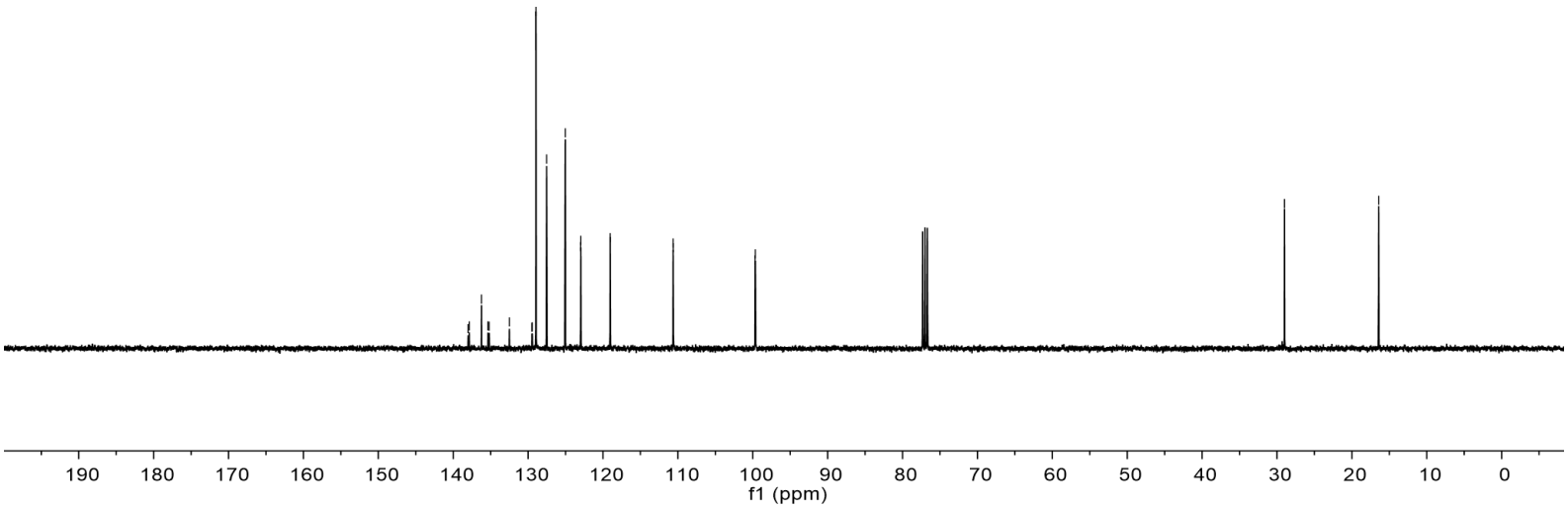
5

HJW2020-80-NMR-HC.2020111302.fid — 13C HJW2020-80-NMR-H/C-2

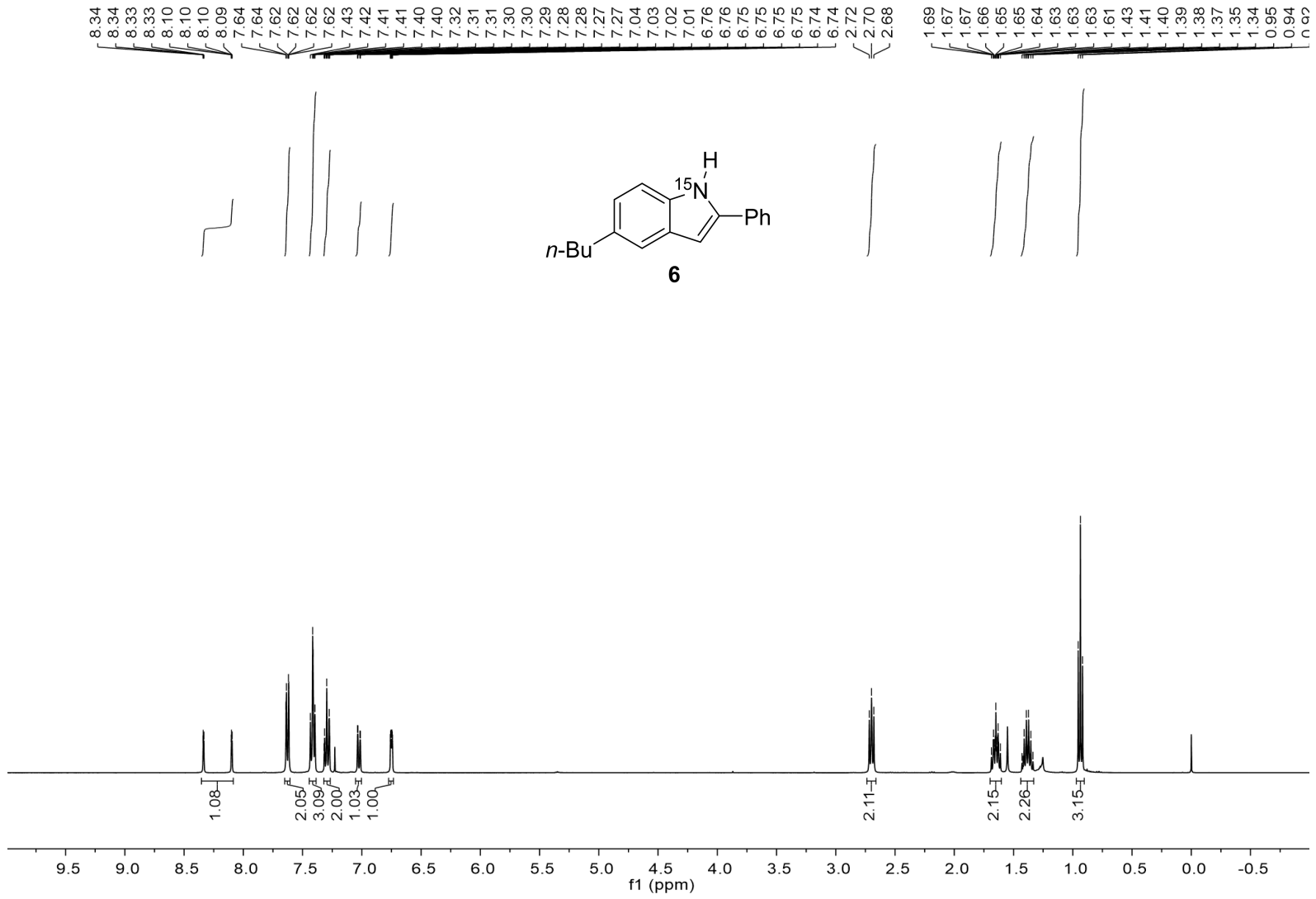
138.01
137.87
136.22
135.36
135.20
132.50
129.49
129.45
128.95
127.53
125.05
125.04
122.98
122.96
119.04
110.63
110.61
99.69
99.65
28.99
16.43



5

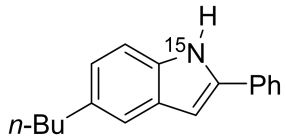


HJW2020-95-NMR-HC.2020111701.fid — 1H HJW2020-95-NMR-H/C

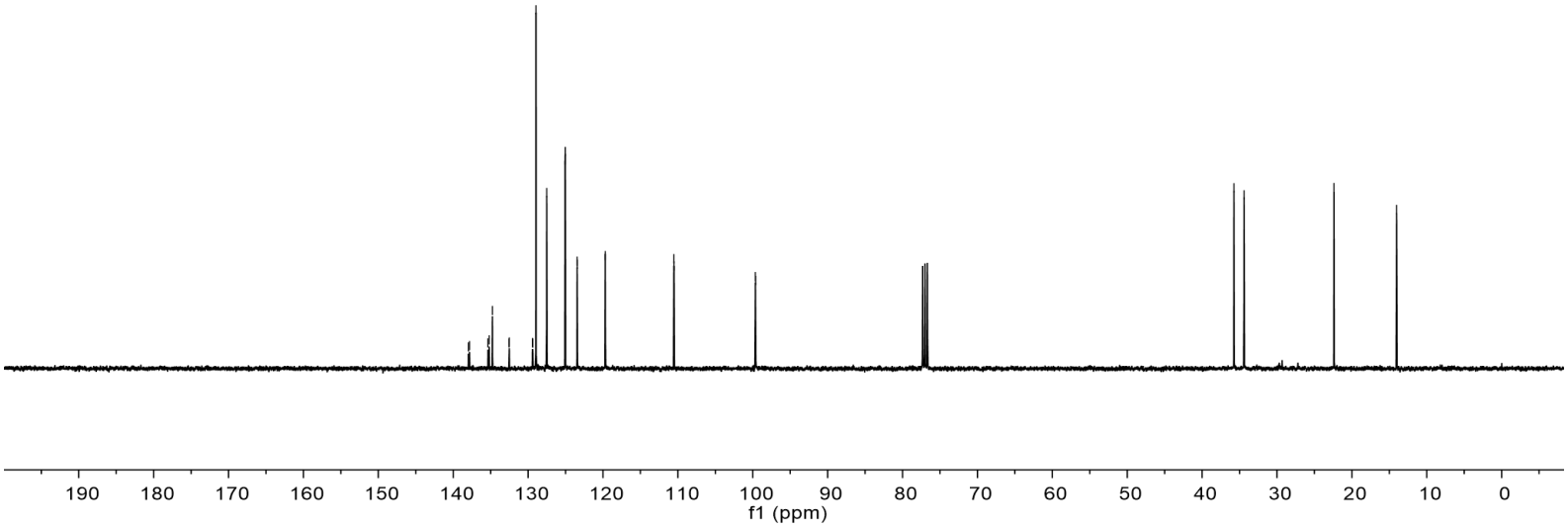


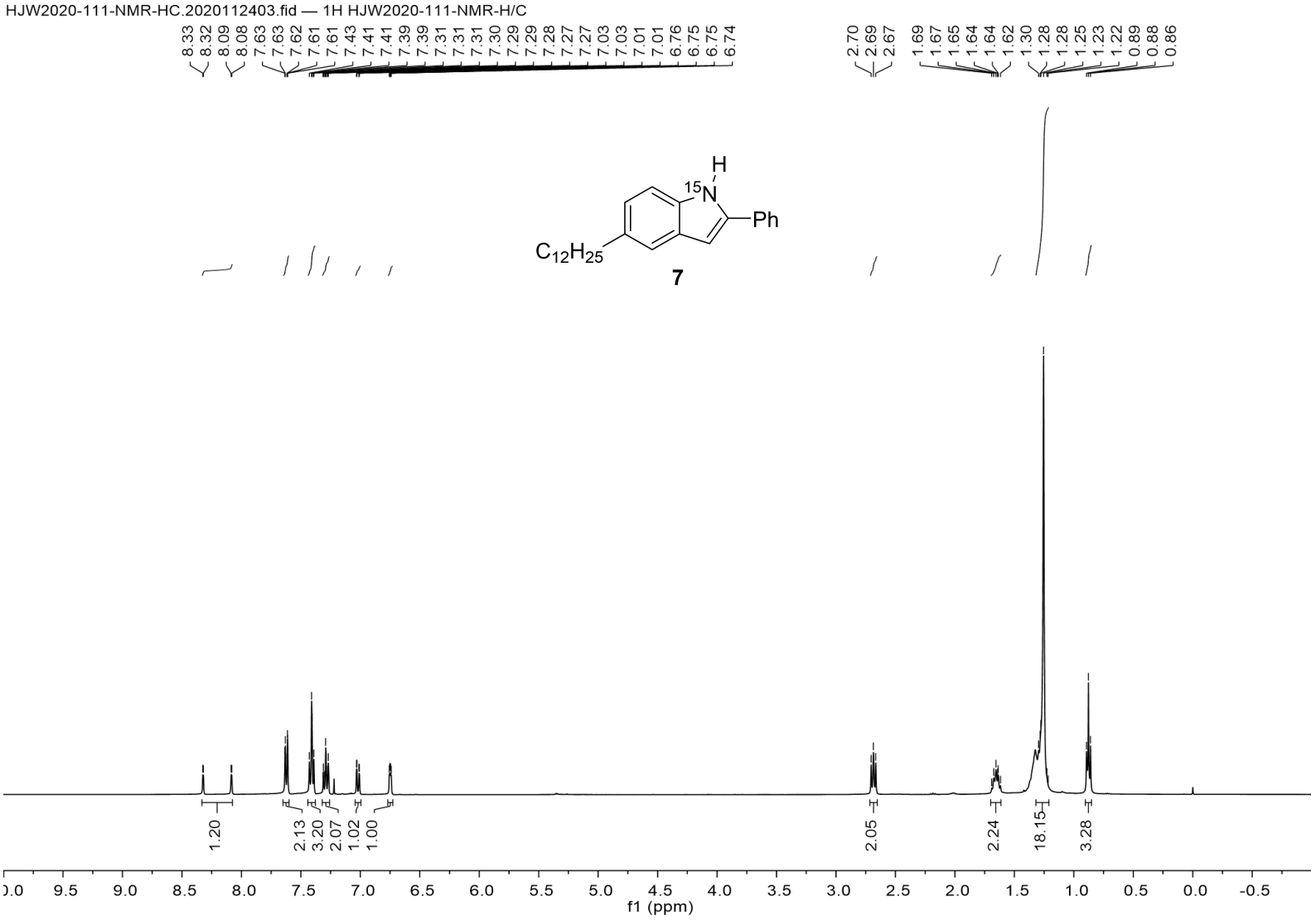
HJW2020-95-NMR-HC.2020111801.fid — 13C HJW2020-95-NMR-H/C

137.97
137.83
135.36
135.20
134.76
132.54
132.51
129.43
129.38
128.95
127.52
125.05
125.04
123.45
123.43
119.70
110.53
110.51
99.65
99.61
35.75
34.40
22.39
14.03



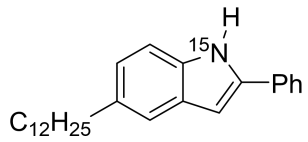
6



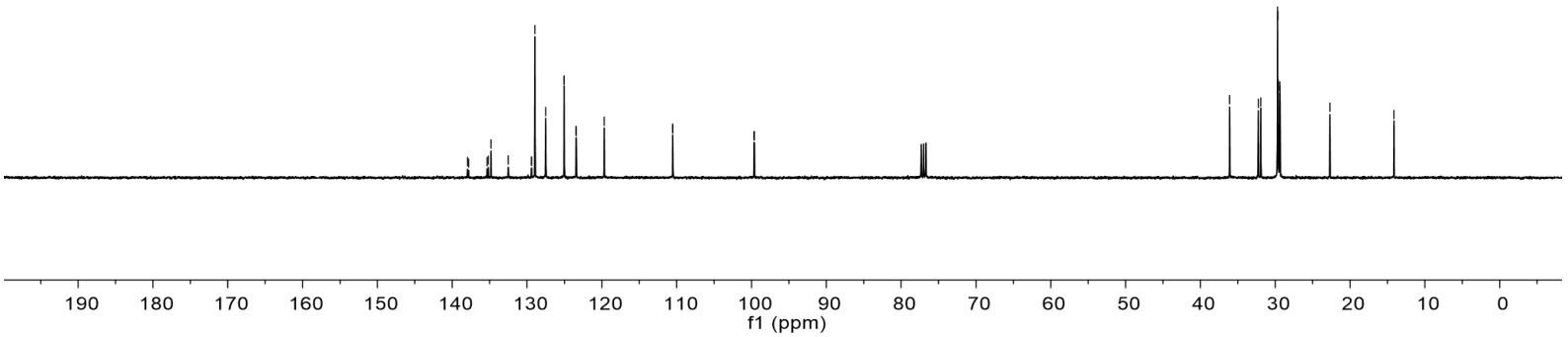

HJW2020-111-NMR-HC.2020112403.fid — 1H HJW2020-111-NMR-H/C
8.33 8.32 8.09 8.08 7.63 7.63 7.62 7.61 7.61 7.43 7.41 7.41 7.39 7.39 7.31 7.31 7.31 7.30 7.29 7.29 7.28 7.27 7.27 7.03 7.03 7.01 7.01 6.76 6.75 6.75 6.74
2.70 2.69 2.67 1.69 1.67 1.65 1.64 1.64 1.62 1.30 1.28 1.28 1.25 1.23 1.22 0.89 0.88 0.86
H
15N
Ph
C12H25
7
1.20 2.13 3.20 2.07 1.02 1.00 2.05 2.24 18.15 3.28
f1 (ppm)

HJW2020-111-NMR-HC.2020112407.fid — 13C HJW2020-111-NMR-H/C

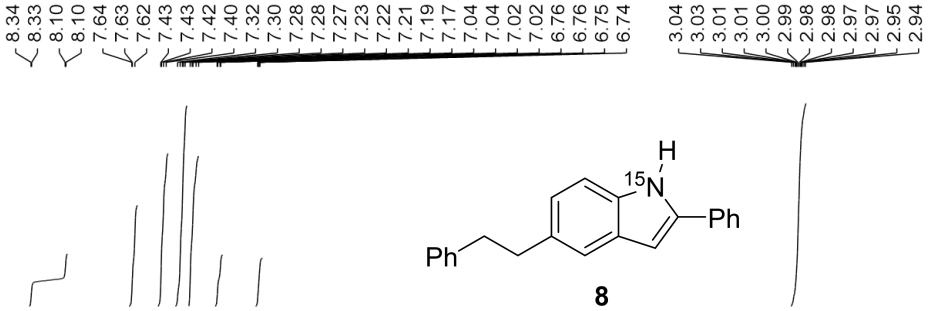
137.95
137.81
135.35
135.19
134.83
132.53
129.43
129.38
128.94
127.51
125.05
125.03
123.44
123.42
119.69
110.53
110.51
99.66
99.62
36.09
32.27
31.93
29.70
29.65
29.61
29.39
29.37
22.70
14.13



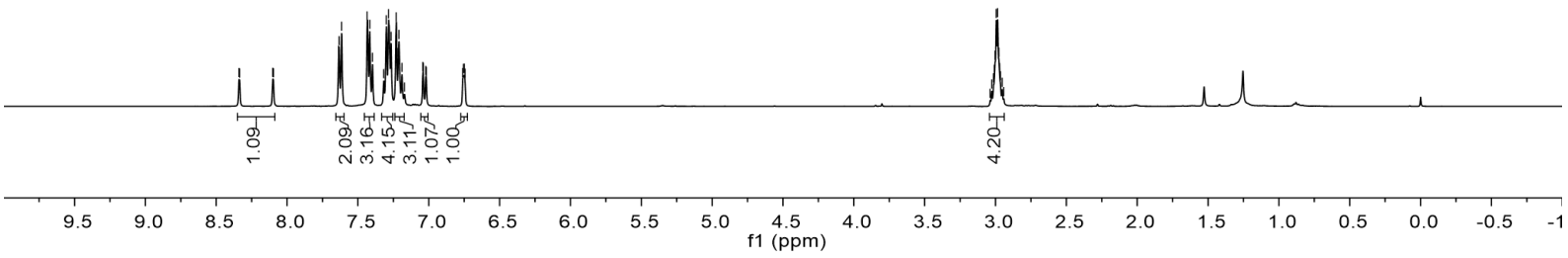
7

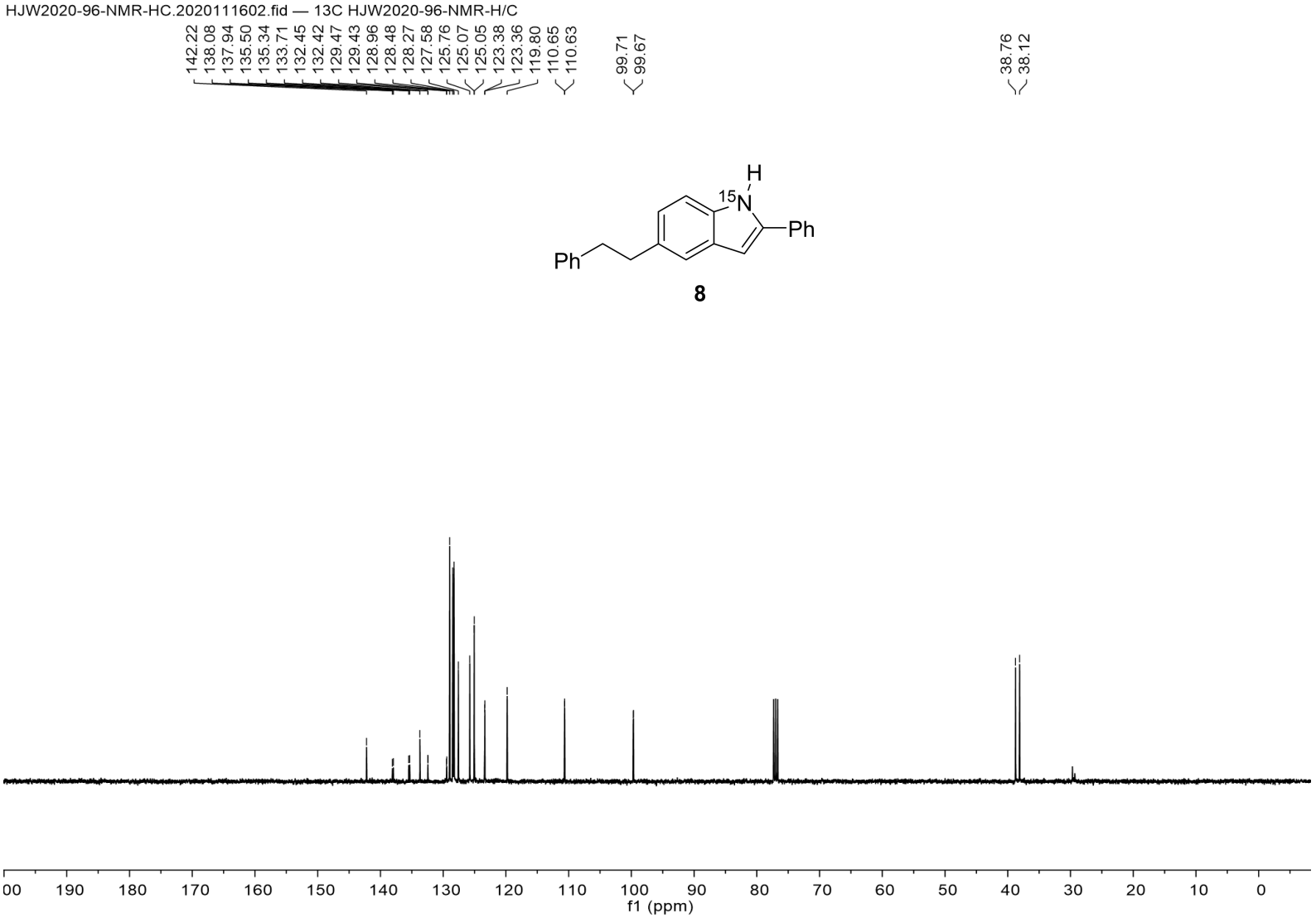


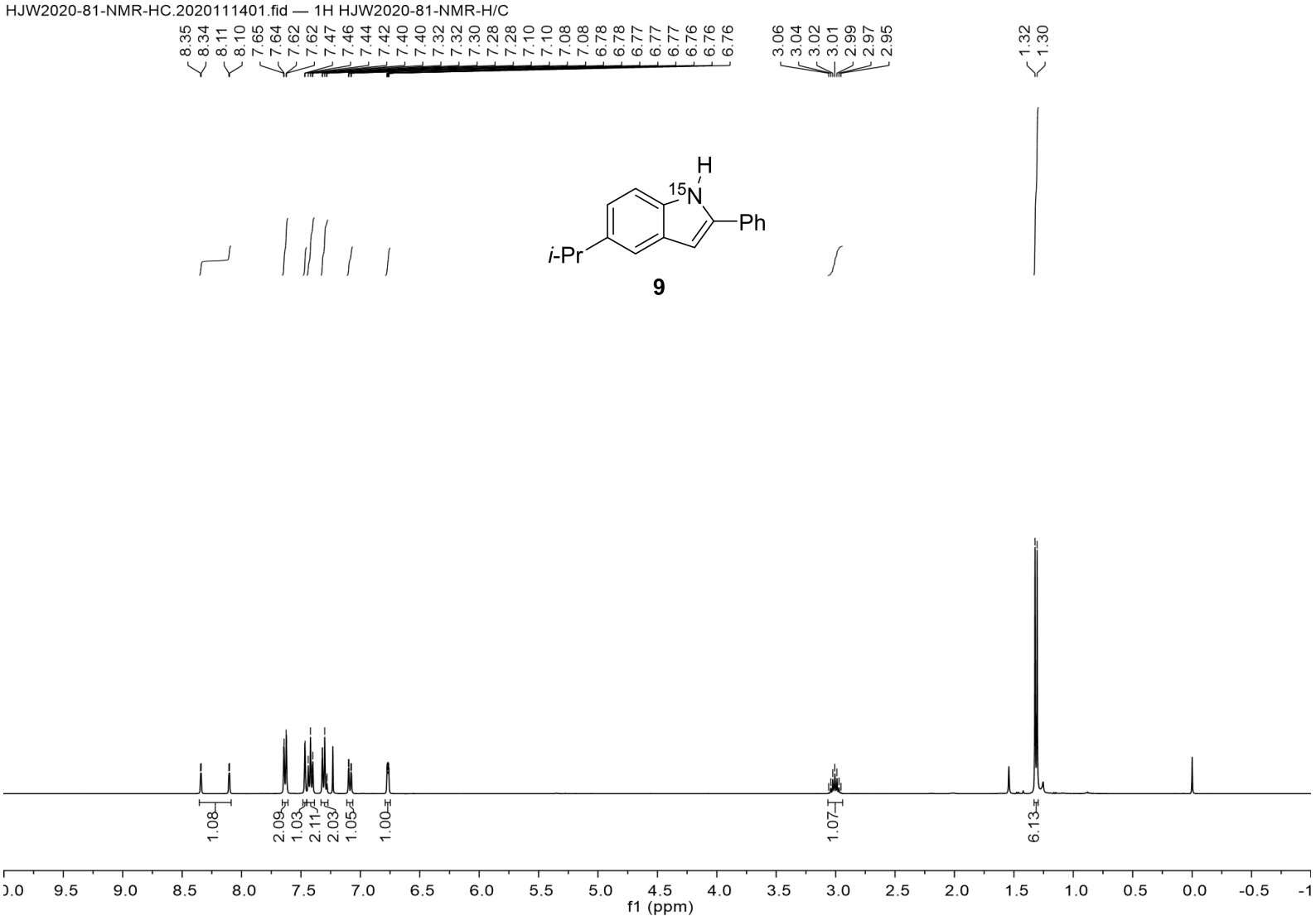
HJW2020-96-NMR-HC.2020111601.fid — 1H HJW2020-96-NMR-H/C



8

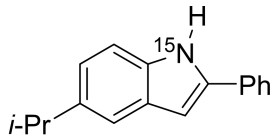



HJW2020-96-NMR-HC.2020111602.fid — 13C HJW2020-96-NMR-H/C
142.22
138.08
137.94
135.50
135.34
133.71
132.45
132.42
129.47
129.43
128.96
128.48
128.27
127.58
125.76
125.07
125.05
123.38
123.36
119.80
110.65
110.63
99.71
99.67
38.76
38.12
H
15N
Ph
Ph
8
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

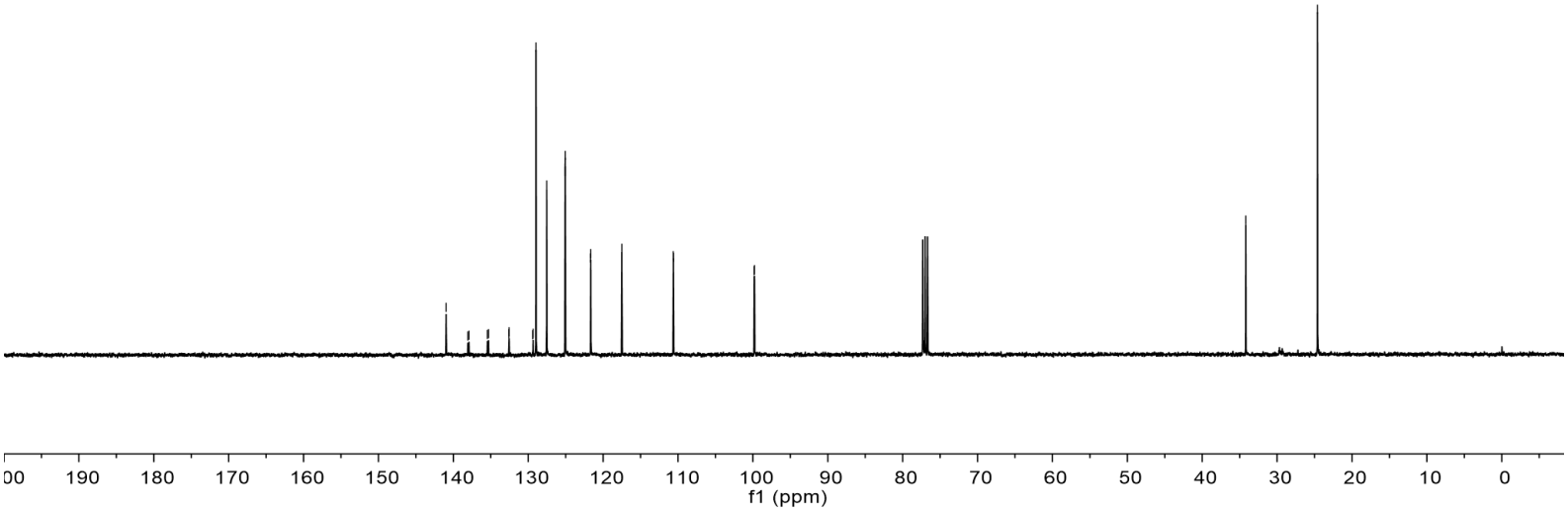

HJW2020-81-NMR-HC.2020111401.fid — 1H HJW2020-81-NMR-H/C
8.35
8.34
8.11
8.10
7.65
7.64
7.62
7.62
7.47
7.46
7.44
7.42
7.40
7.40
7.32
7.32
7.30
7.28
7.28
7.10
7.10
7.08
7.08
6.78
6.78
6.77
6.77
6.77
6.76
6.76
6.76
3.06
3.04
3.02
3.01
2.99
2.97
2.95
1.32
1.30
H
15N
Ph
i-Pr
9
1.08
2.09
1.03
2.11
2.03
1.05
1.00
1.07
6.13
f1 (ppm)

HJW2020-81-NMR-HC.2020111407.fid — 13C HJW2020-81-NMR-H/C

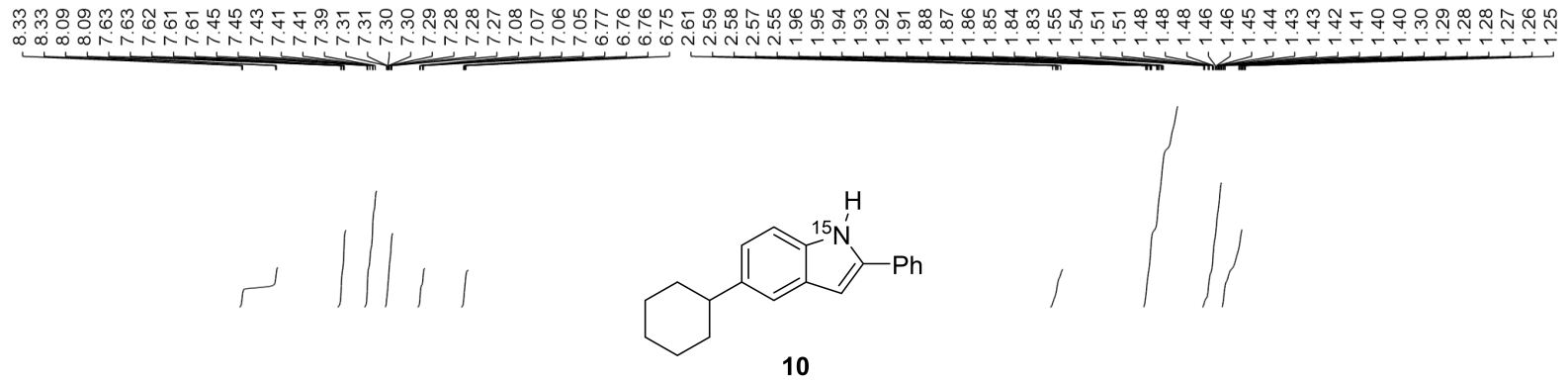
140.97
138.06
137.92
135.46
135.30
132.57
132.55
129.39
129.35
128.96
127.53
125.08
125.07
121.67
121.65
117.50
110.63
110.61
99.83
99.79
34.19
24.61



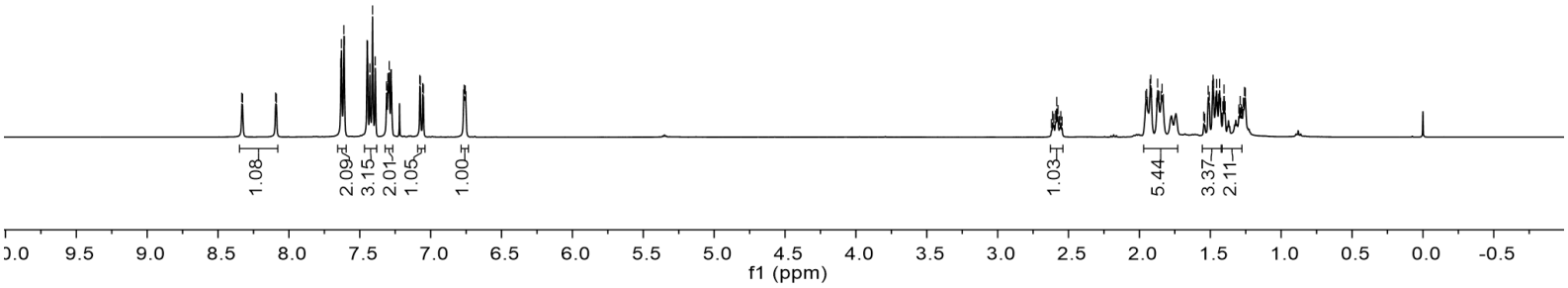
9



HJW2020-110-NMR-HC.2020112402.fid — 1H HJW2020-110-NMR-H/C

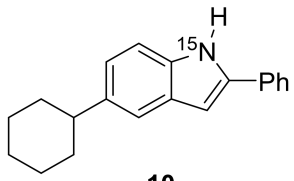


10

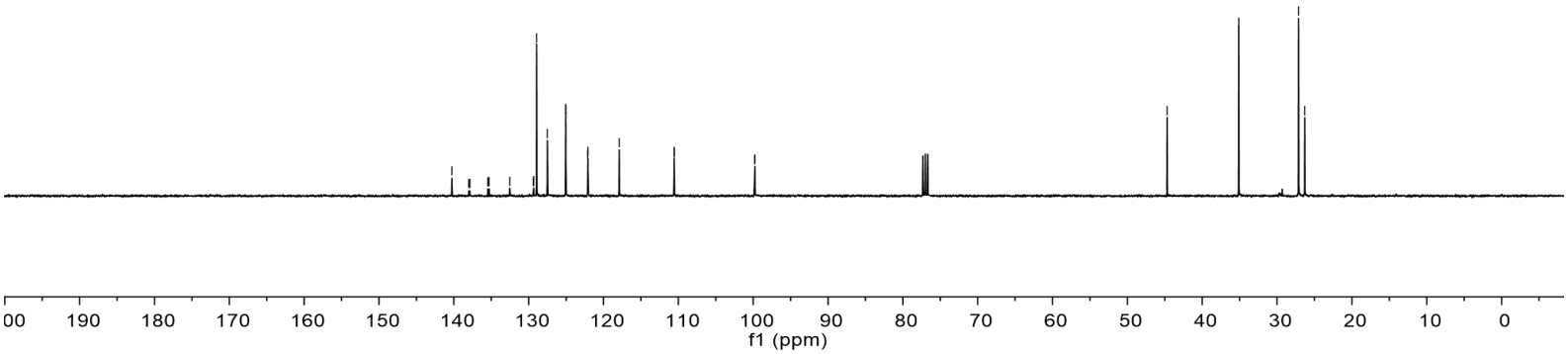


HJW2020-110-NMR-HC.2020112406.fid — 13C HJW2020-110-NMR-H/C

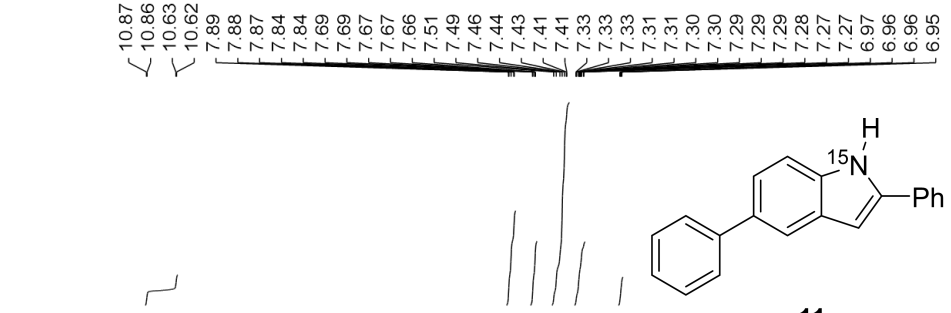
140.26
138.01
137.86
135.46
135.30
132.53
129.37
129.33
128.94
127.50
125.06
125.05
122.10
122.08
117.91
110.56
110.55
99.82
99.78
44.69
35.12
27.13
26.30



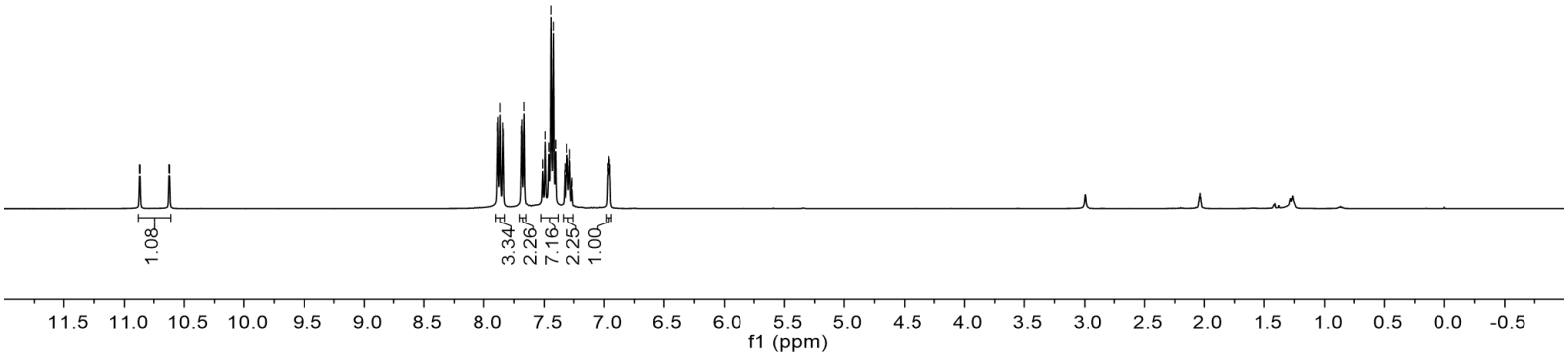
10



HJW2020-87-NMR-HC.2020122603.fid — 1H HJW2020-87-NMR-H/C-acetone

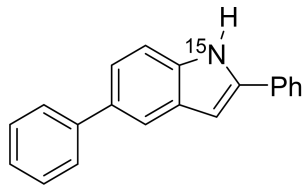


11

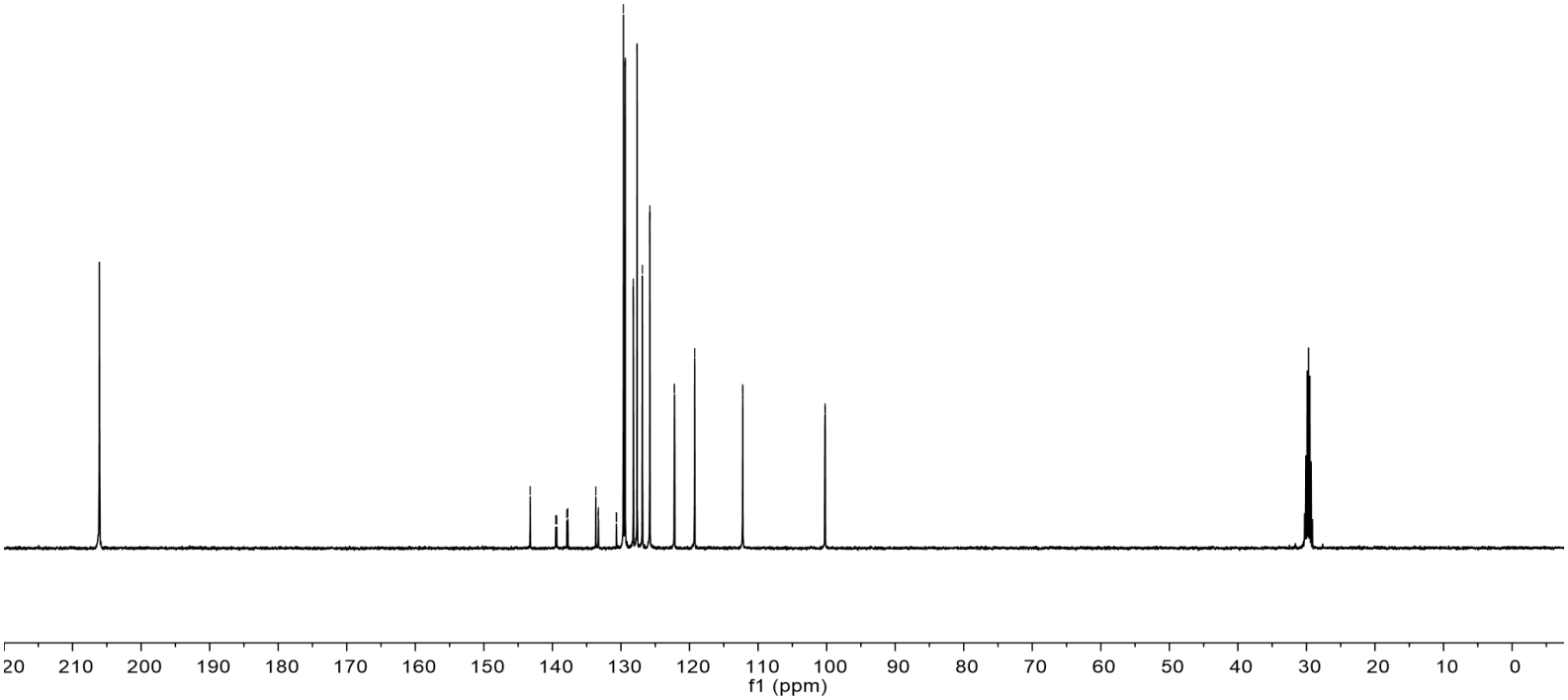


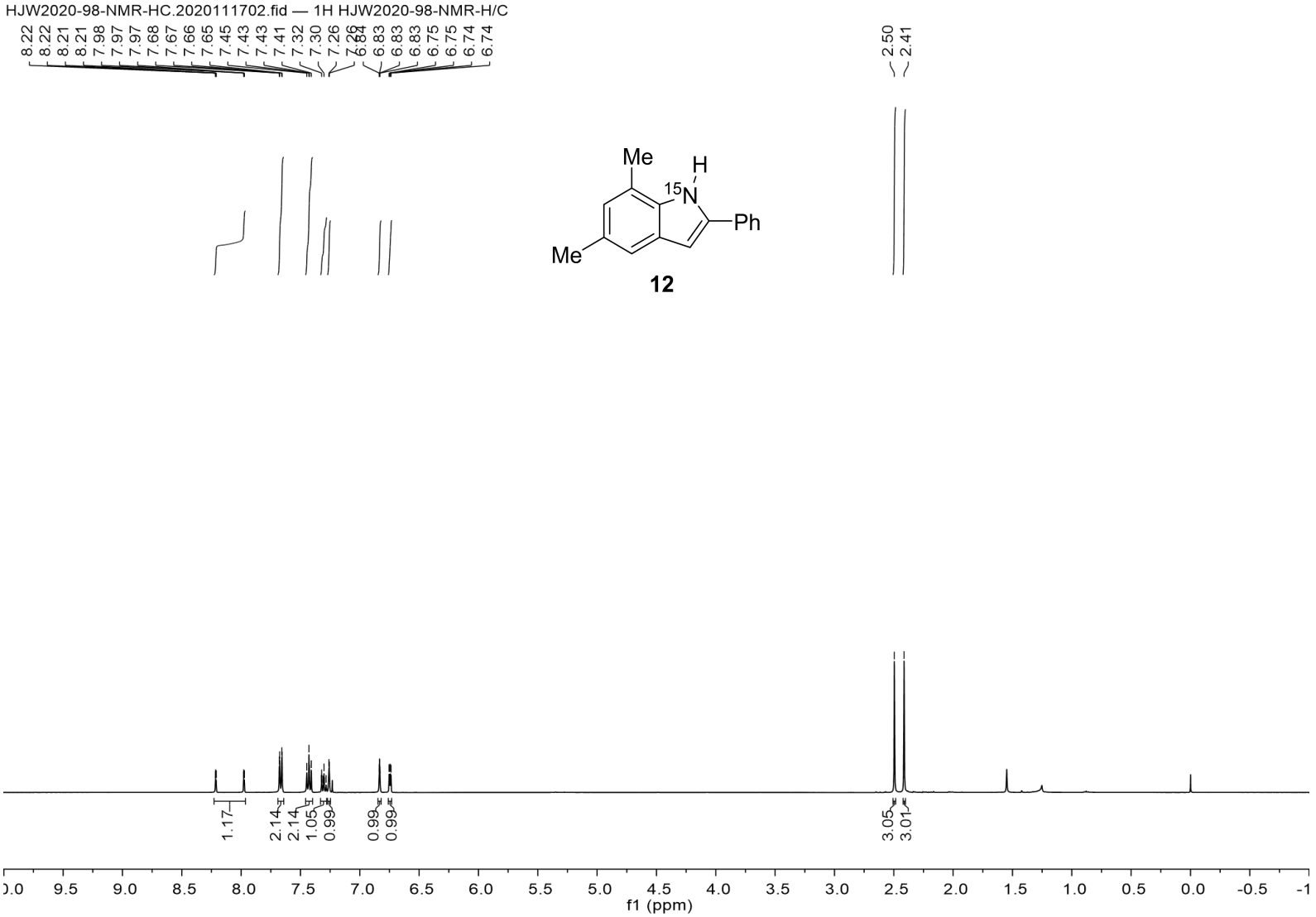
HJW2020-87-NMR-HC.2020122604.fid — 13C HJW2020-87-NMR-H/C-acetone

143.24
139.51
139.37
137.90
137.74
133.68
133.31
133.29
130.67
130.63
129.63
129.38
128.19
127.64
126.87
125.79
125.78
122.20
122.18
119.25
112.25
112.23
100.24
100.20



11

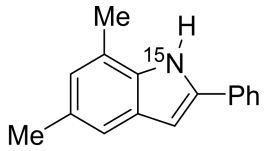



HJW2020-98-NMR-HC.2020111702.fid — 1H HJW2020-98-NMR-H/C
8.22
8.22
8.21
8.21
7.98
7.97
7.97
7.68
7.67
7.66
7.65
7.45
7.43
7.43
7.41
7.32
7.30
7.26
6.84
6.83
6.83
6.75
6.74
6.74
2.50
2.41
Me
H
15N
Ph
Me
12
1.17
2.14
2.14
1.05
0.99
0.99
0.99
3.05
3.01
f1 (ppm)

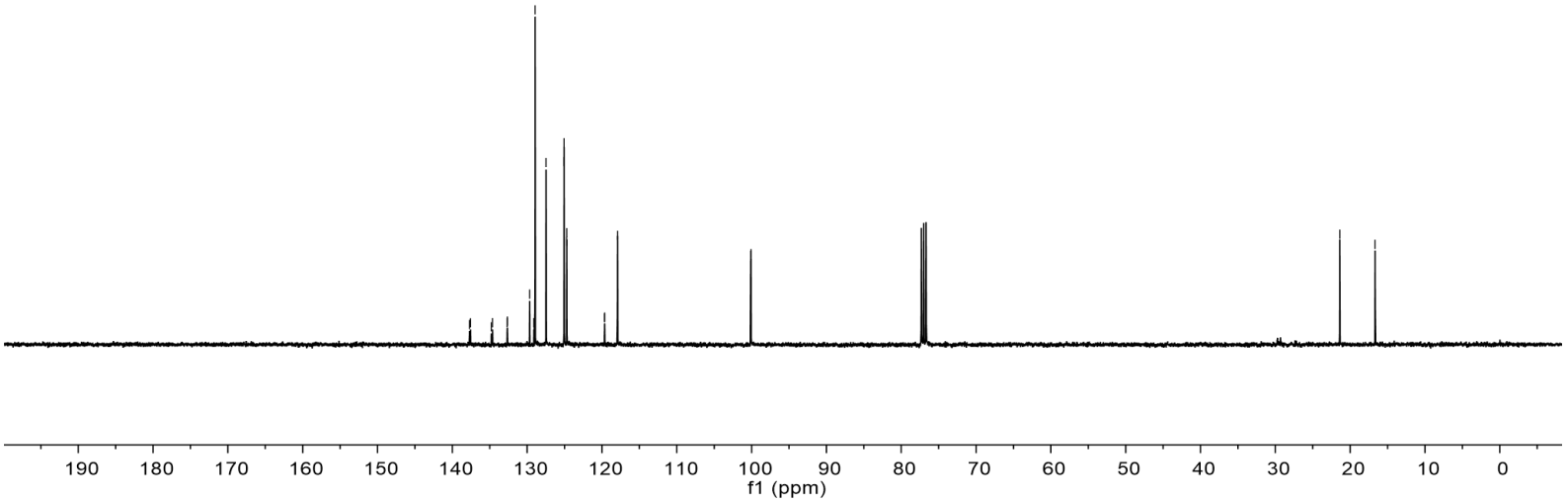
HJW2020-98-NMR-HC.2020111705.fid — 13C HJW2020-98-NMR-H/C

137.71
137.57
134.79
134.63
132.66
132.64
129.66
129.10
129.06
128.94
127.48
125.07
125.06
124.72
124.70
119.66
119.65
117.92
100.12
100.08

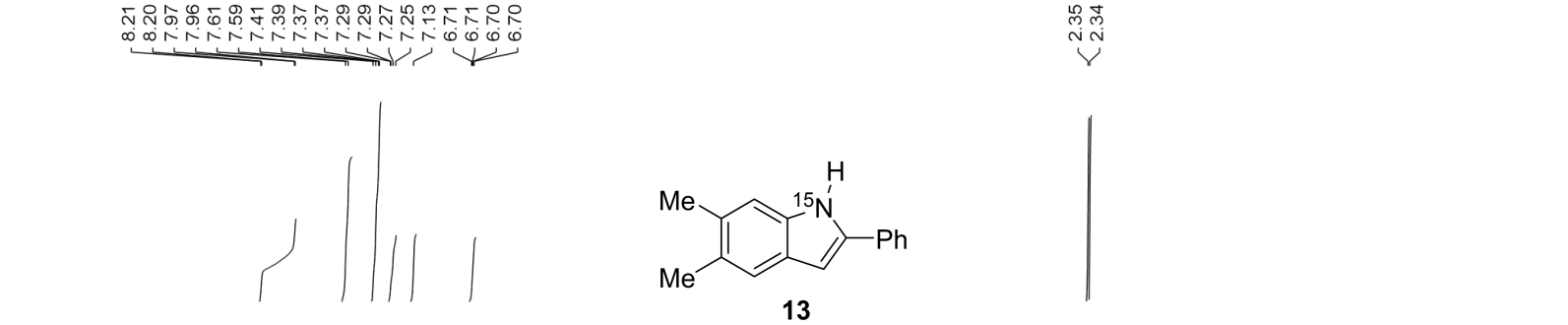
21.39
16.66



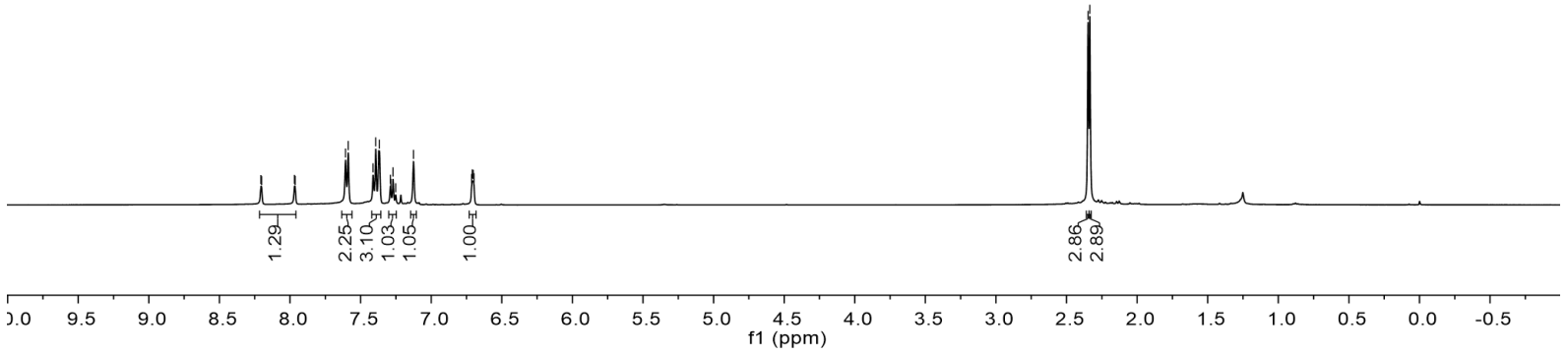
12



HJW2020-97-NMR-HC.2020112301.fid — 1H HJW2020-97-NMR-H/C



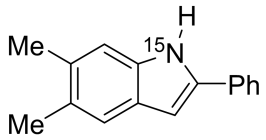
13



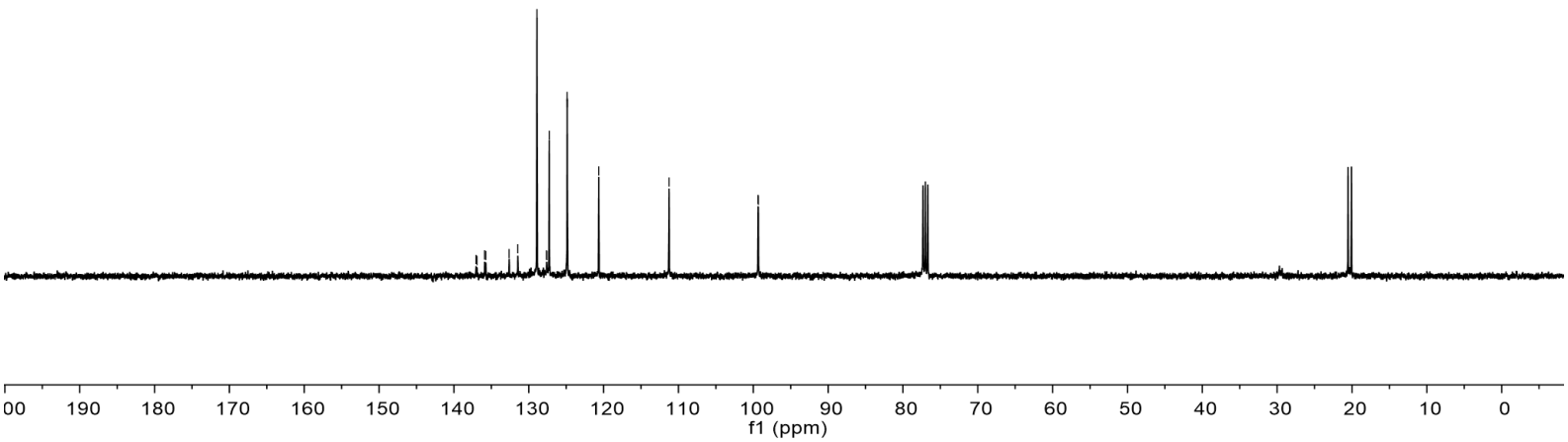
HJW2020-97-NMR-HC.2020112401.fid — 13C HJW2020-97-NMR-H/C

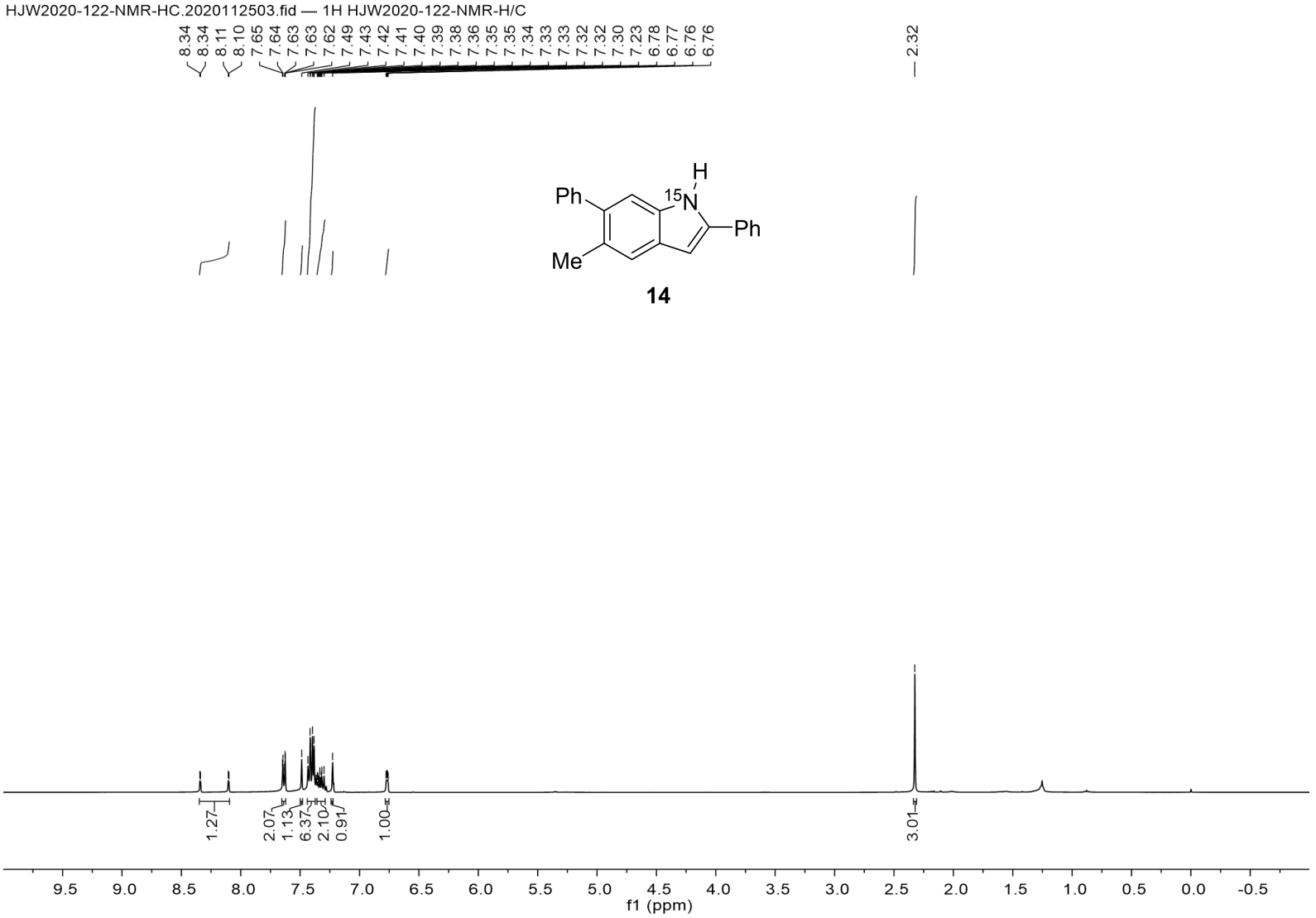
137.05
136.91
135.90
135.74
132.62
131.47
128.91
128.85
127.62
127.58
127.27
124.87
124.85
120.65
111.26

99.36
99.32



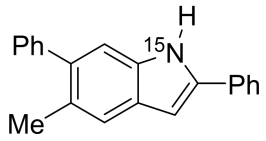
13



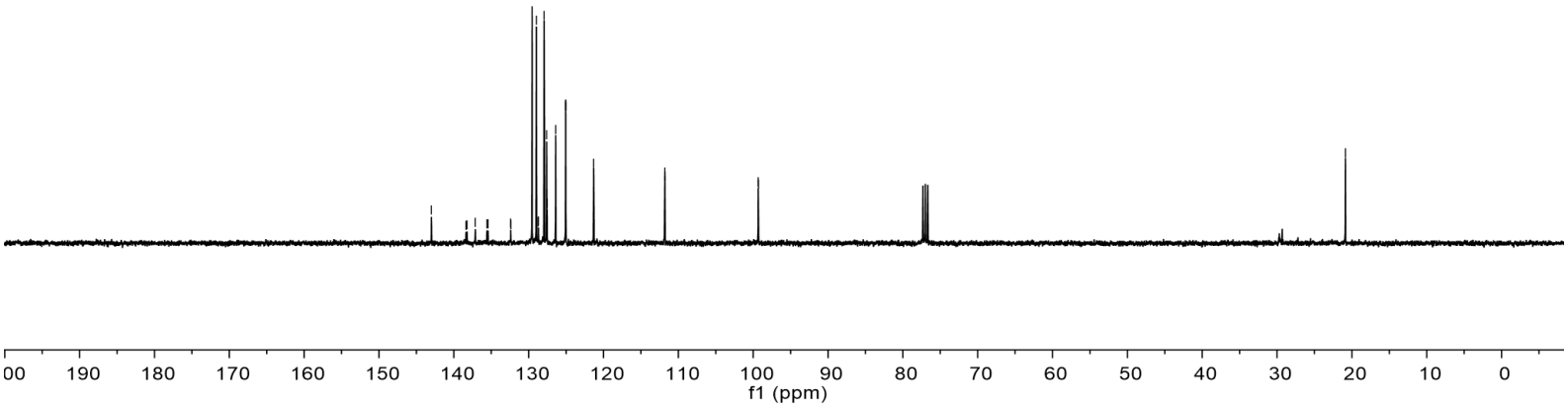

HJW2020-122-NMR-HC.2020112503.fid — 1H HJW2020-122-NMR-H/C
8.34
8.34
8.11
8.10
7.65
7.64
7.63
7.63
7.62
7.49
7.43
7.42
7.41
7.40
7.39
7.38
7.36
7.35
7.35
7.34
7.33
7.33
7.32
7.32
7.30
7.23
6.78
6.77
6.76
6.76
2.32
Ph
15N
H
Ph
Me
14
1.27
2.07
1.13
6.37
2.10
0.91
1.00
3.01
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

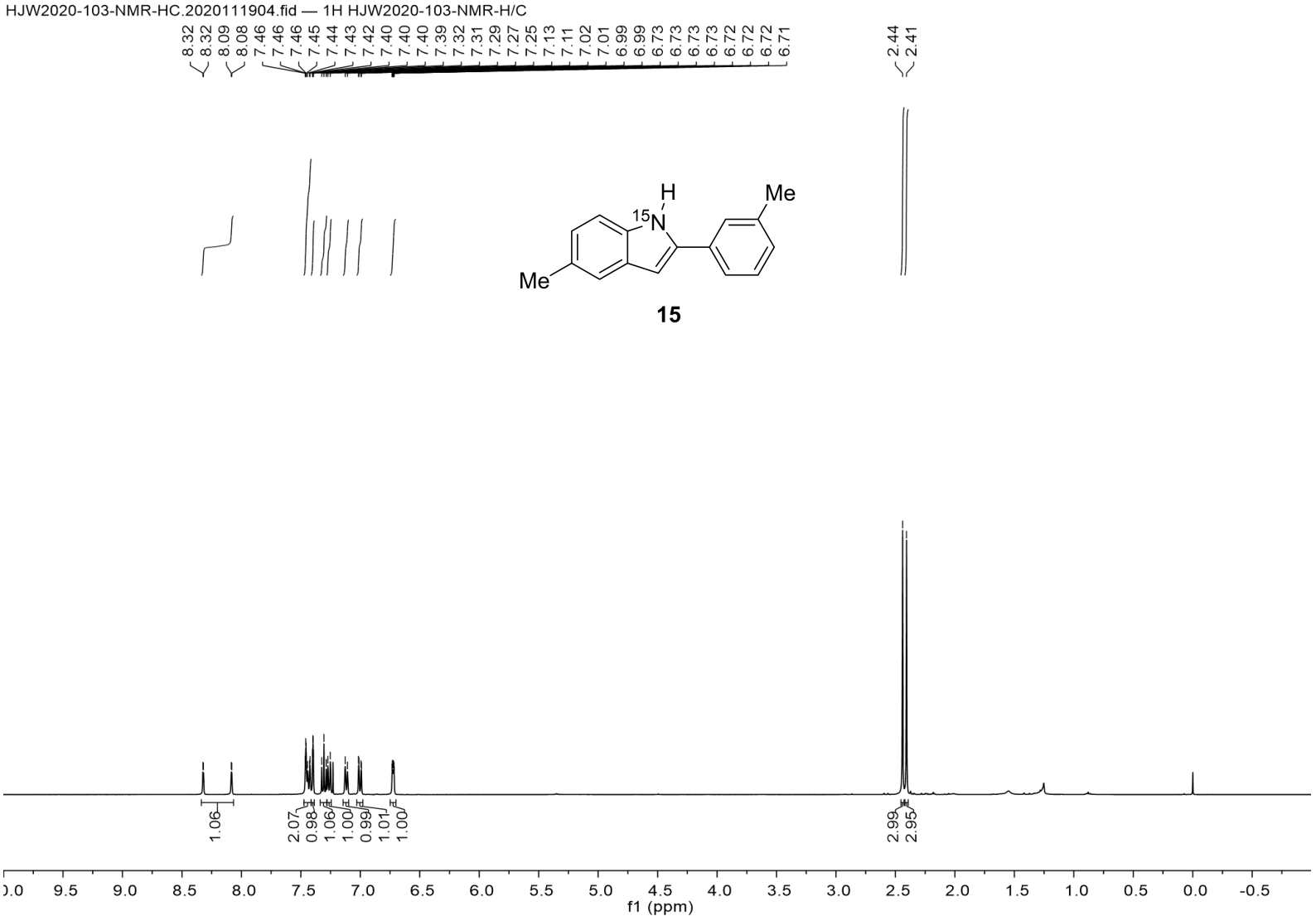
HJW2020-122-NMR-HC.2020112508.fid — 13C HJW2020-122-NMR-H/C

143.00
138.38
138.24
137.14
135.58
135.43
132.40
132.38
129.54
128.97
128.73
128.69
127.93
127.60
127.53
126.40
125.06
125.05
121.31
111.81
111.79
99.33
99.29
20.87



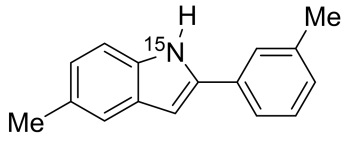
14



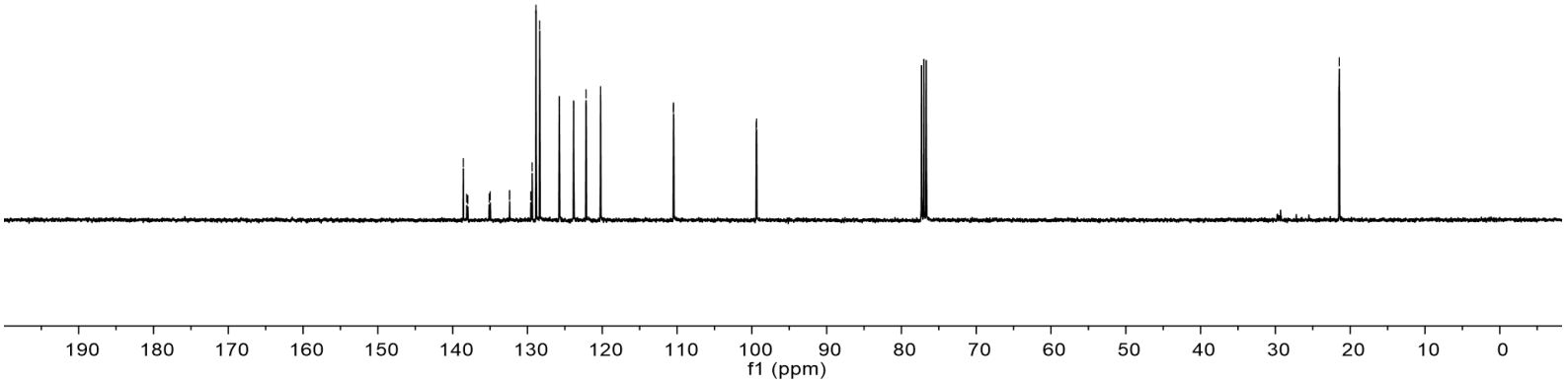

HJW2020-103-NMR-HC.2020111904.fid — 1H HJW2020-103-NMR-H/C
8.32 8.32 8.09 8.08 7.46 7.46 7.46 7.45 7.44 7.43 7.42 7.40 7.40 7.40 7.39 7.32 7.31 7.29 7.27 7.25 7.13 7.11 7.02 7.01 6.99 6.99 6.73 6.73 6.73 6.73 6.72 6.72 6.71
2.44 2.41
H
15N
Me
Me
15
1.06 2.07 0.98 1.06 1.00 0.99 1.01 1.00 2.99 2.95
f1 (ppm)

HJW2020-103-NMR-HC.2020111905.fid — 13C HJW2020-103-NMR-H/C

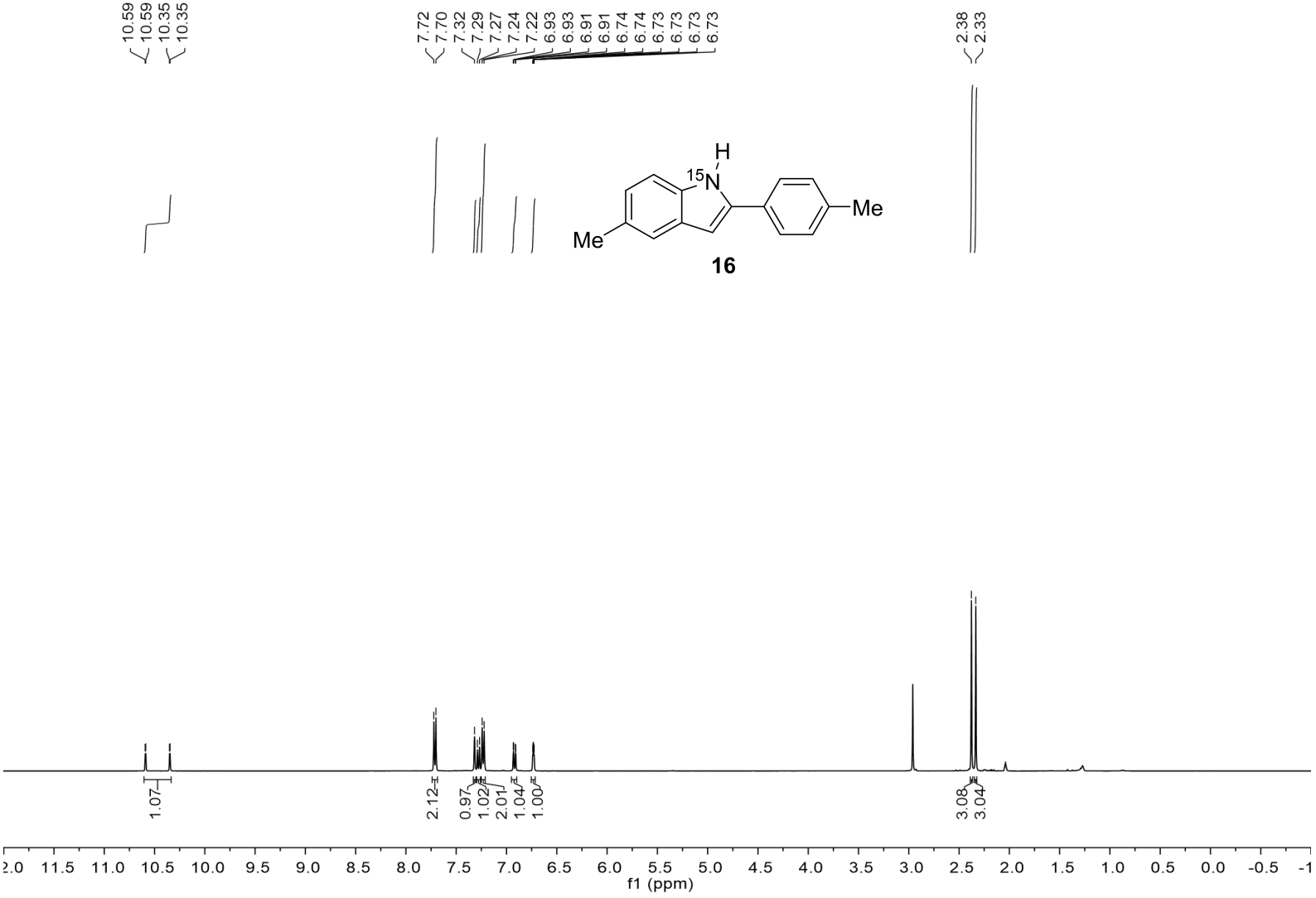
138.58
138.12
137.98
135.13
134.97
132.41
132.38
129.55
129.51
129.37
128.85
128.36
125.76
125.74
123.84
123.82
122.17
122.16
120.22
110.48
110.46
99.41
99.37
21.51
21.45



15

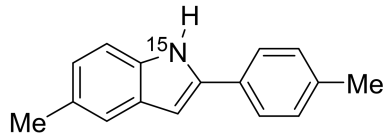


HJW2020-82-NMR-HC.2020111101.fid — 1H HJW2020-82-NMR-H/C-acetone

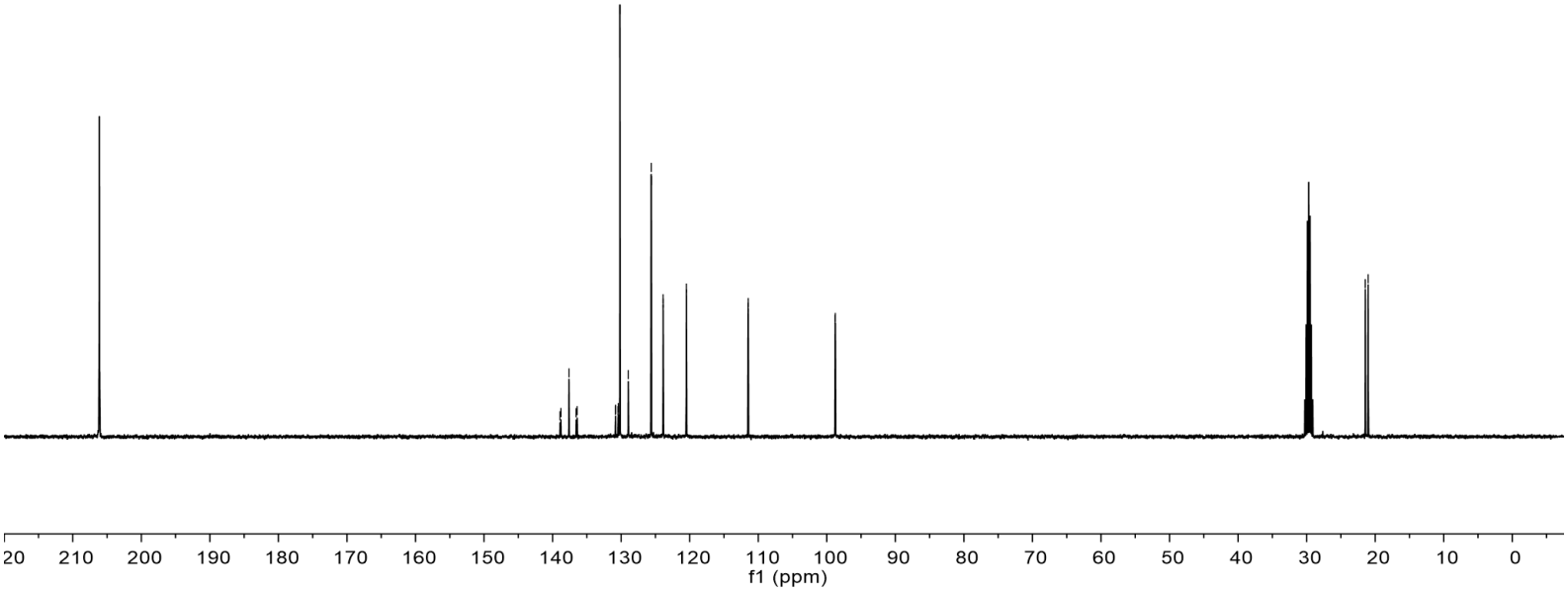


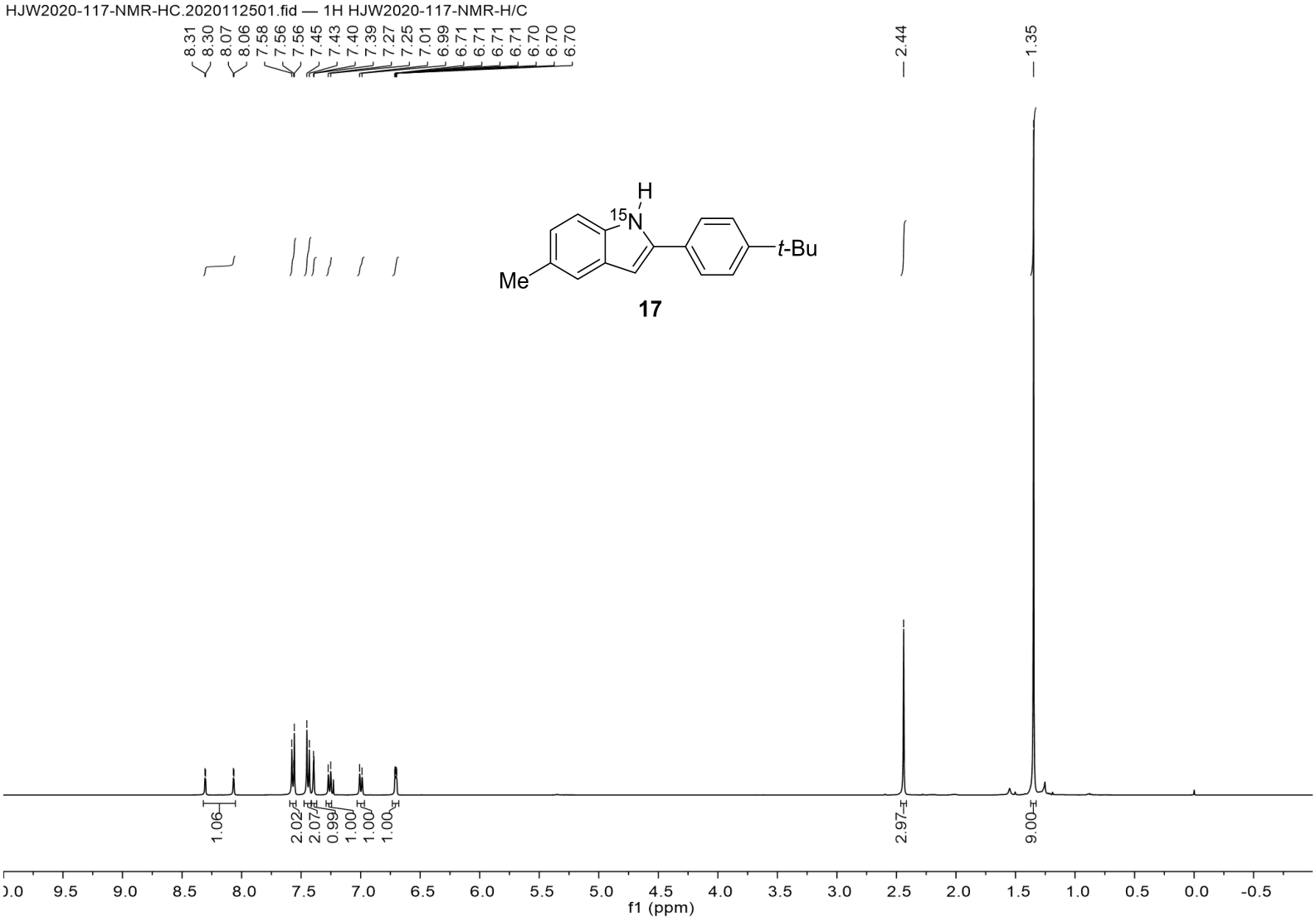
HJW2020-82-NMR-HC.2020111203.fid — 13C HJW2020-82-NMR-H/C

138.93
138.78
137.63
136.58
136.42
130.85
130.83
130.46
130.42
130.18
128.97
125.62
125.60
123.88
123.86
120.49
111.49
111.48
98.79
98.75
21.45
21.03



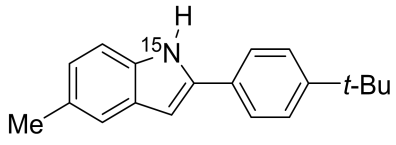
16



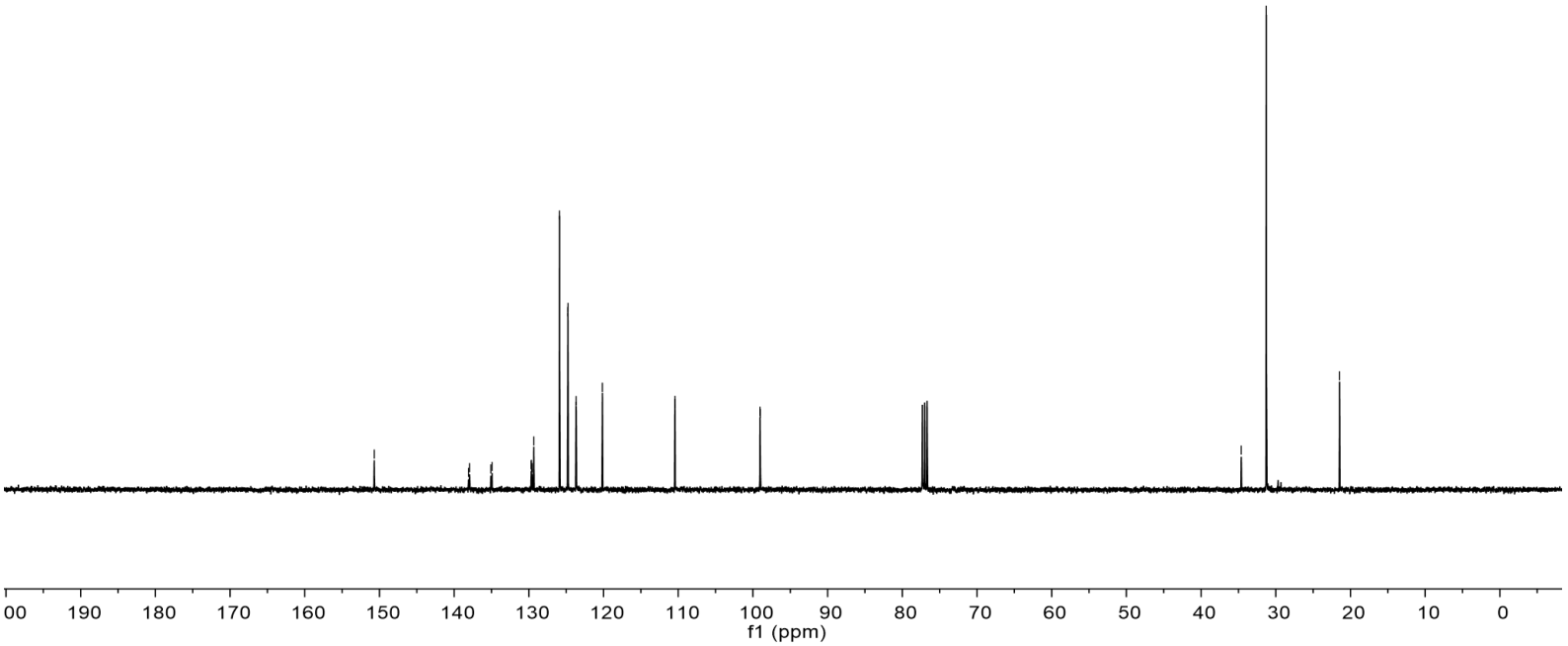

HJW2020-117-NMR-HC.2020112501.fid — 1H HJW2020-117-NMR-H/C
8.31
8.30
8.07
8.06
7.58
7.56
7.56
7.45
7.43
7.40
7.39
7.27
7.25
7.01
6.99
6.71
6.71
6.71
6.70
6.70
6.70
2.44
1.35
H
15N
Me
t-Bu
17
1.06
2.02
2.07
0.99
1.00
1.00
1.00
2.97
9.00
f1 (ppm)

HJW2020-117-NMR-HC.2020112506.fid — 13C HJW2020-117-NMR-H/C

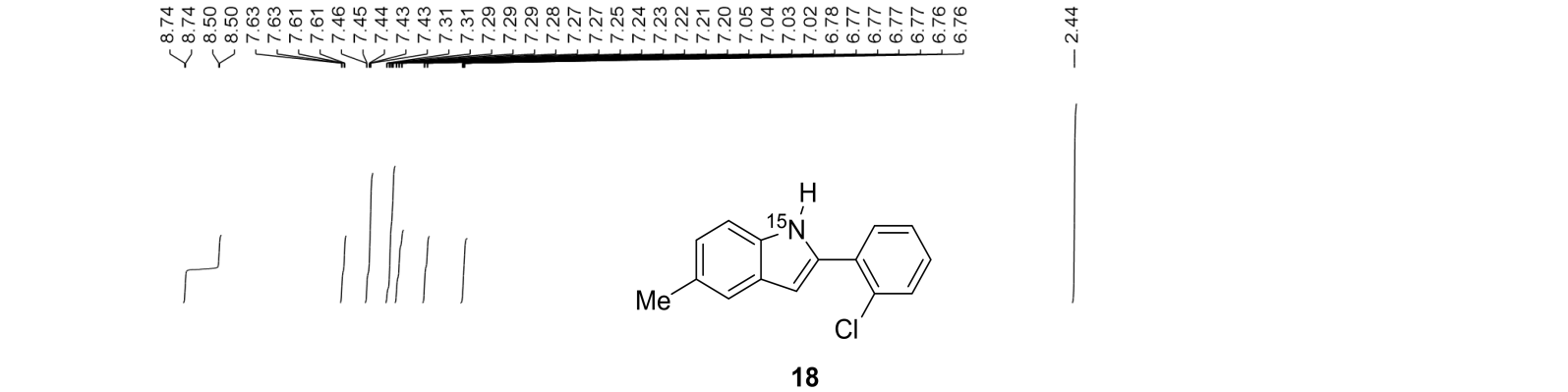
150.70
138.08
137.94
135.08
134.93
129.68
129.66
129.62
129.57
129.33
125.89
124.78
124.76
123.69
123.67
120.16
110.45
110.44
99.06
99.02
34.63
31.26
21.46



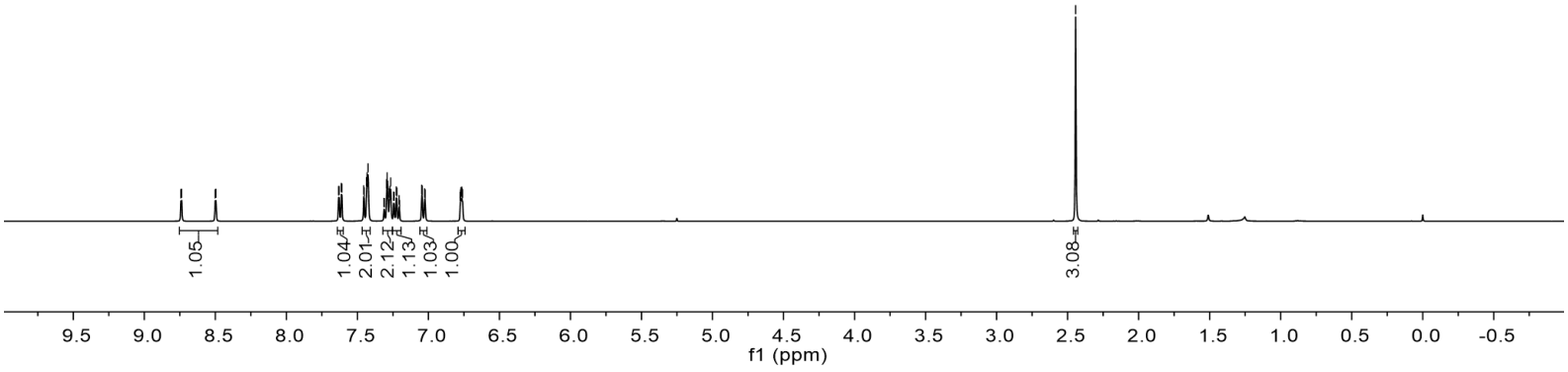
17



HJW2020-94-NMR-HC.2020111004.fid — 1H HJW2020-94-NMR-H/C



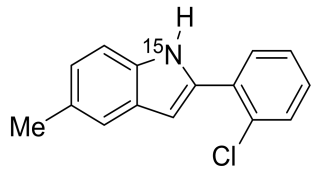
18



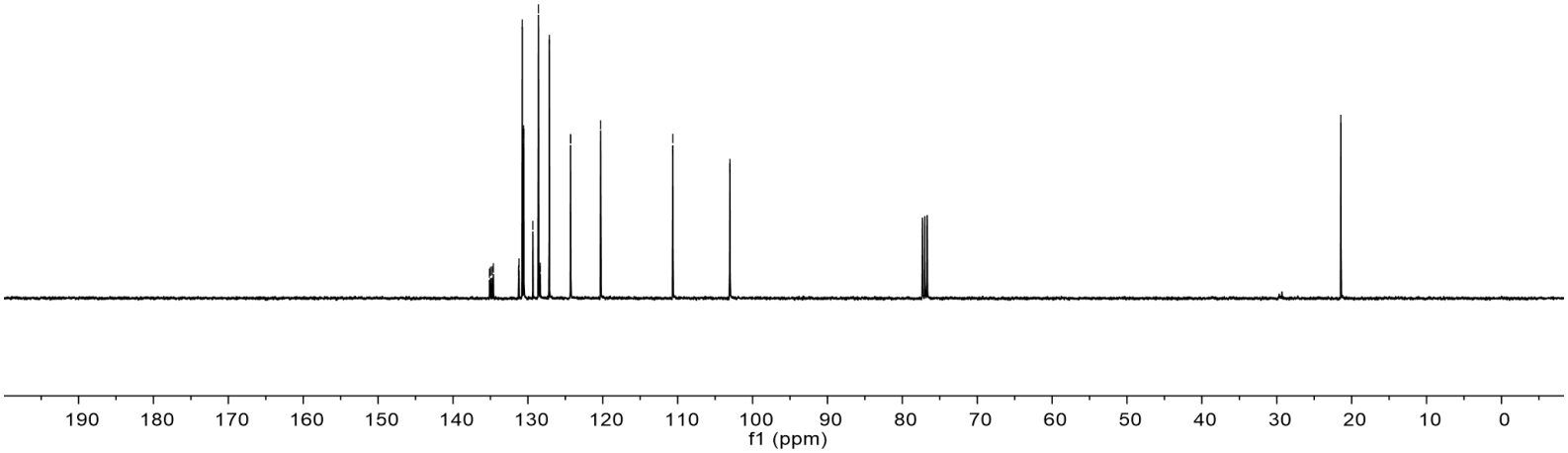
HJW2020-94-NMR-HC.2020111106.fid — 13C HJW2020-94-NMR-H/C

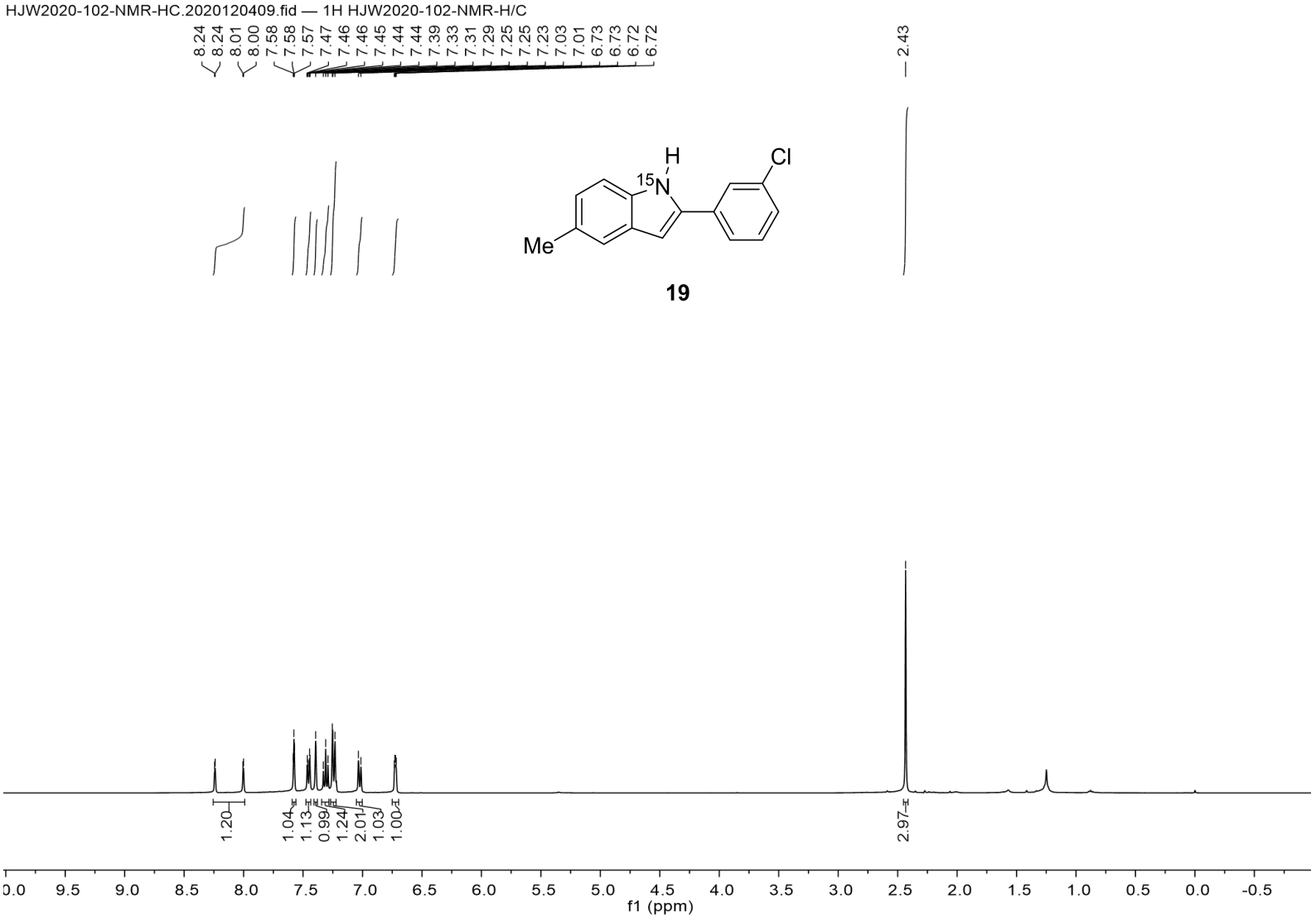
135.12
134.98
134.76
134.60
131.25
131.23
131.19
130.75
130.57
130.55
129.35
128.60
128.39
128.35
127.15
124.31
124.29
120.28
110.66
110.65
103.06
103.02

— 21.44



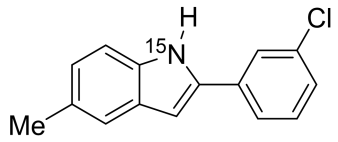
18



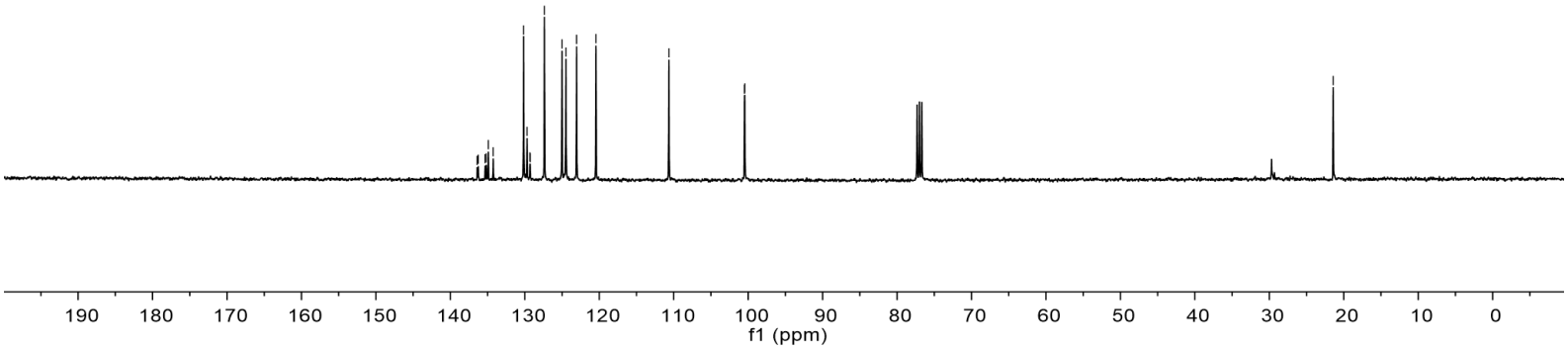

HJW2020-102-NMR-HC.2020120409.fid — 1H HJW2020-102-NMR-H/C
8.24
8.24
8.01
8.00
7.58
7.58
7.57
7.47
7.46
7.46
7.45
7.44
7.44
7.39
7.33
7.31
7.29
7.25
7.25
7.23
7.03
7.01
6.73
6.73
6.72
6.72
2.43
H
15N
Cl
Me
19
1.20
1.04
1.13
0.99
1.24
2.01
1.03
1.00
2.97
f1 (ppm)

18210860001.2020120404.fid — 13C HJW2020-102-NMR-H/C

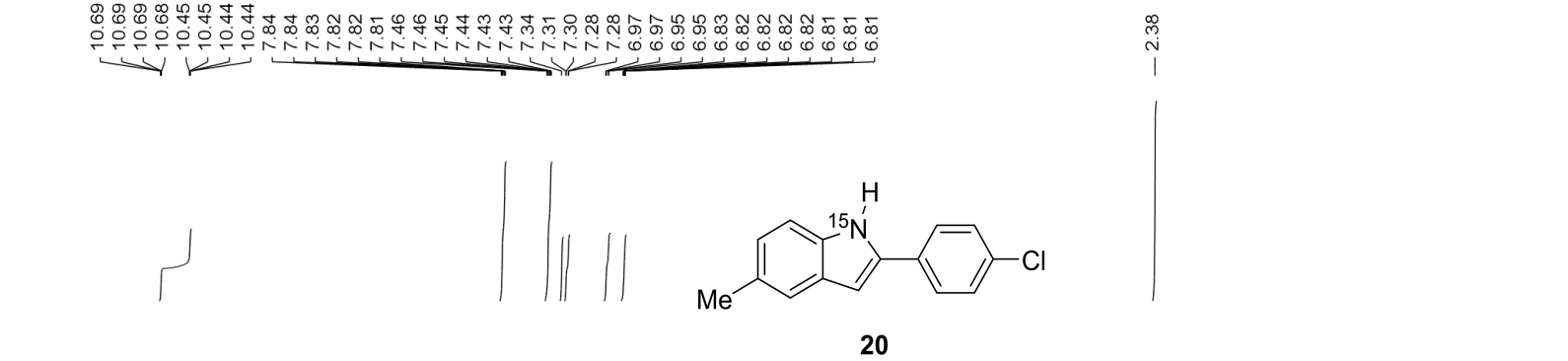
136.40
136.26
135.33
135.17
134.90
134.25
130.16
129.70
129.32
129.28
127.37
125.01
124.49
123.05
120.45
110.65
100.50
100.46
21.43



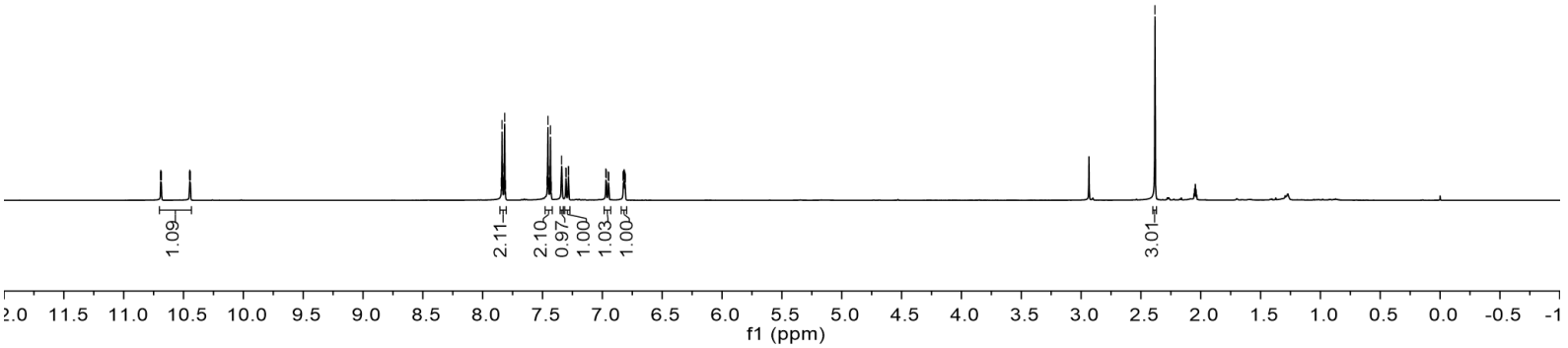
19



HJW2020-99-NMR-HC.2020111703.fid — 1H HJW2020-99-NMR-H/C

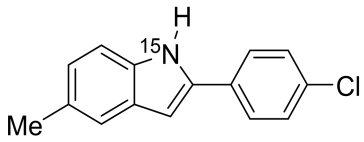


20

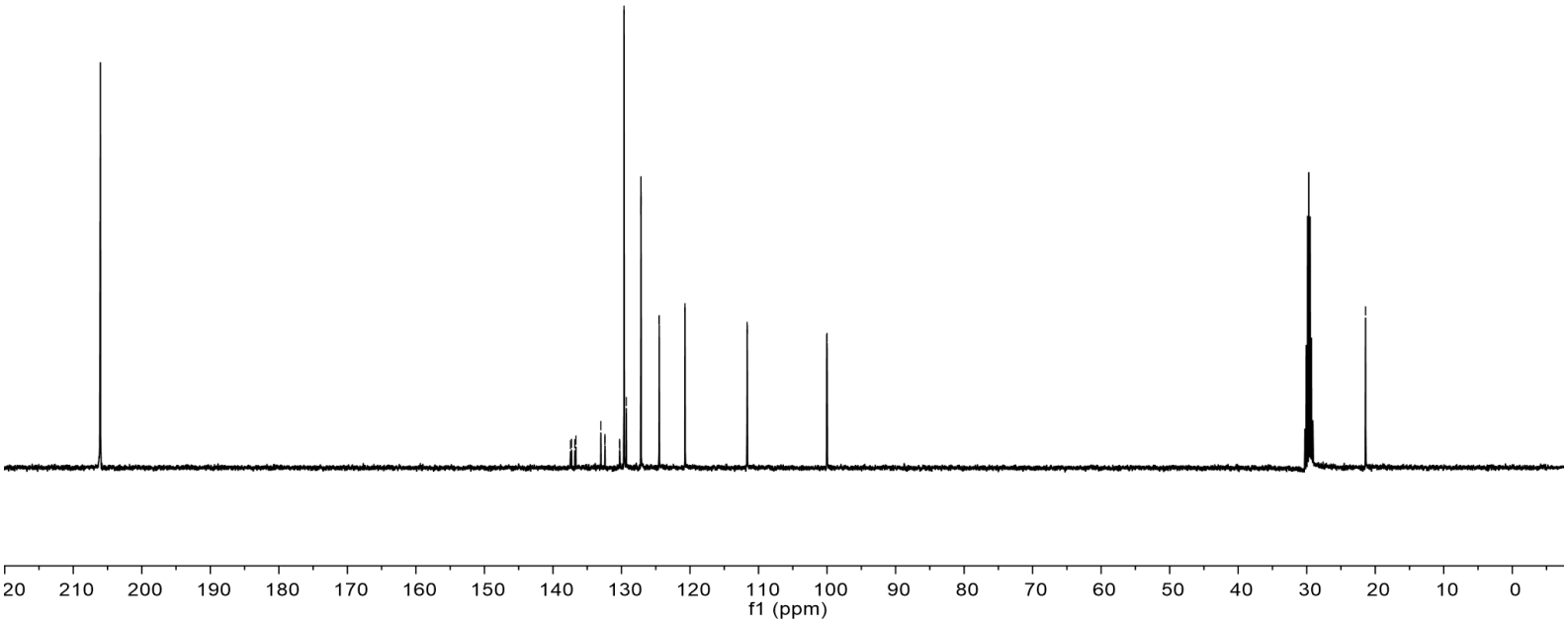


HJW2020-99-NMR-HC.2020111704.fid — 13C HJW2020-99-NMR-H/C

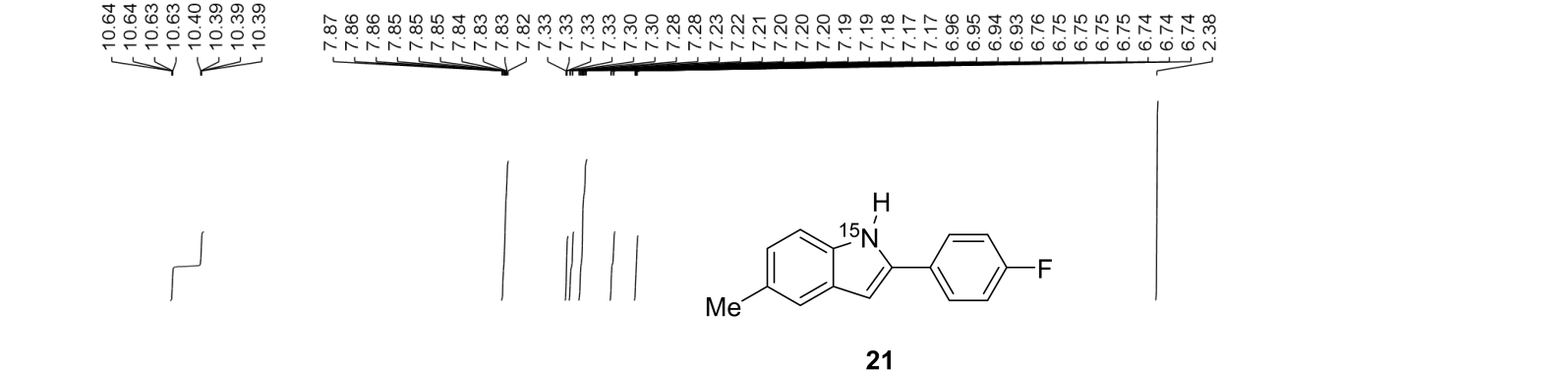
137.43
137.29
136.81
136.66
133.01
132.43
132.40
130.27
130.23
129.63
129.30
127.16
127.14
124.51
124.49
120.73
111.66
111.64
100.04
100.01
21.42



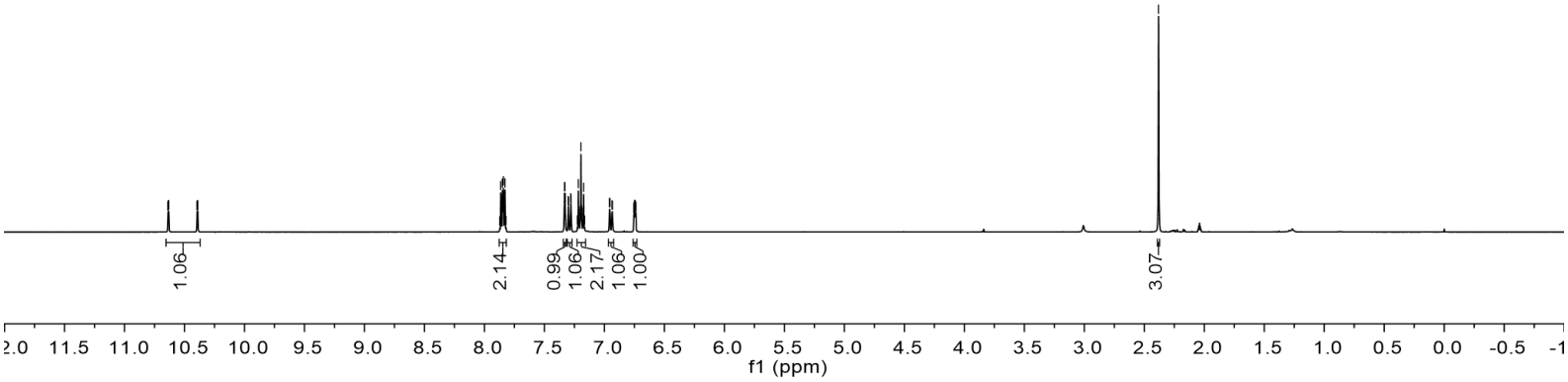
20



HJW2020-91-NMR-HC.2020112304.fid — 1H HJW2020-91-NMR-H/C



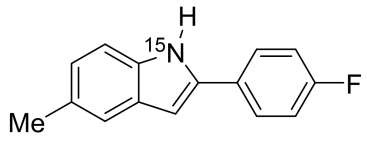
21



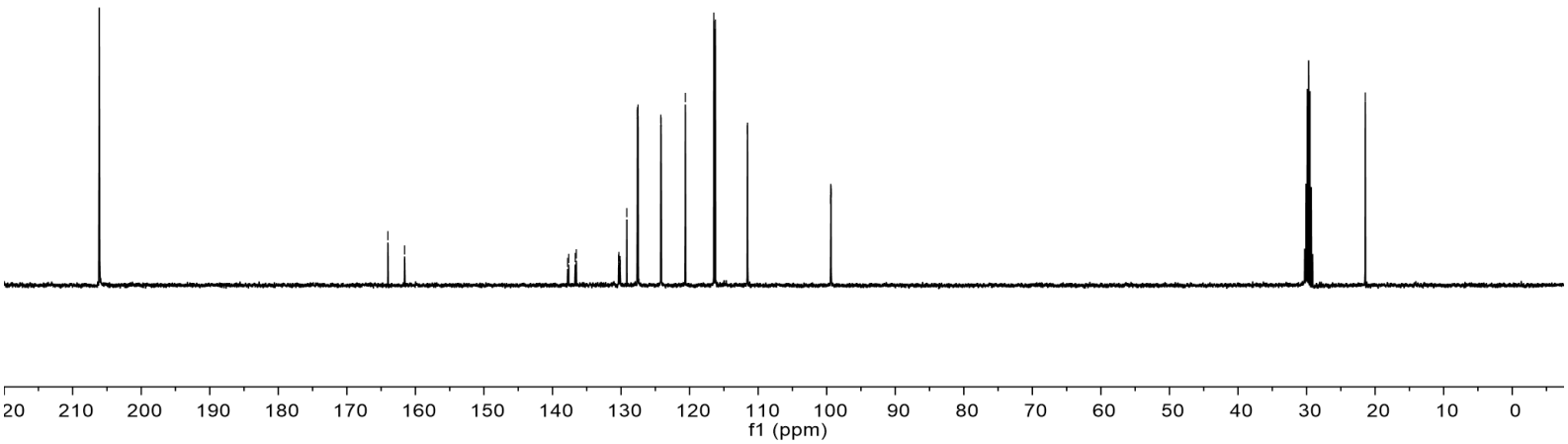
HJW2020-91-NMR-HC.2020112306.fid — 13C HJW2020-91-NMR-H/C

164.01
161.57
137.79
137.65
136.69
136.53
130.36
130.32
130.18
130.16
130.12
129.17
127.62
127.61
127.54
127.53
124.19
124.17
120.63
116.47
116.25
111.58
111.56
99.43
99.41
99.39
99.37

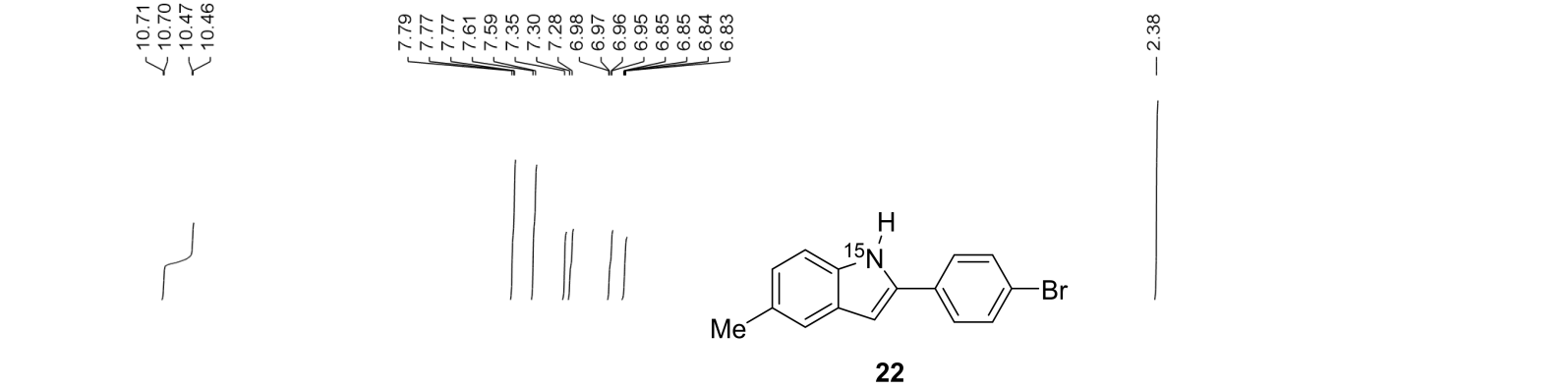
21.43



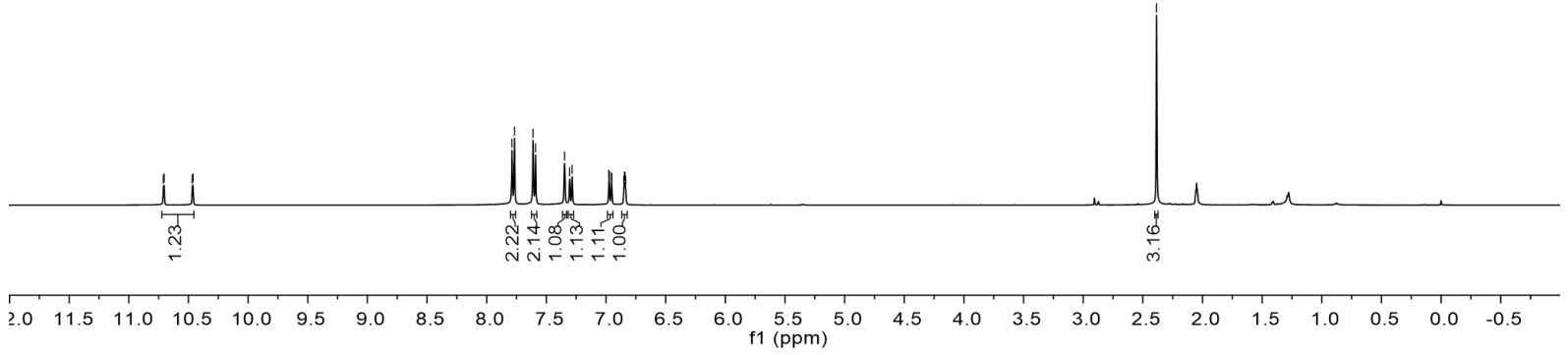
21



HJW2020-83-NMR-HC.2021010802.fid — 1H HJW2020-83

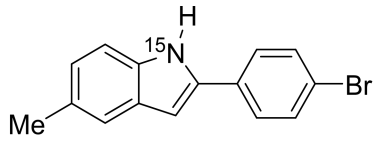


22

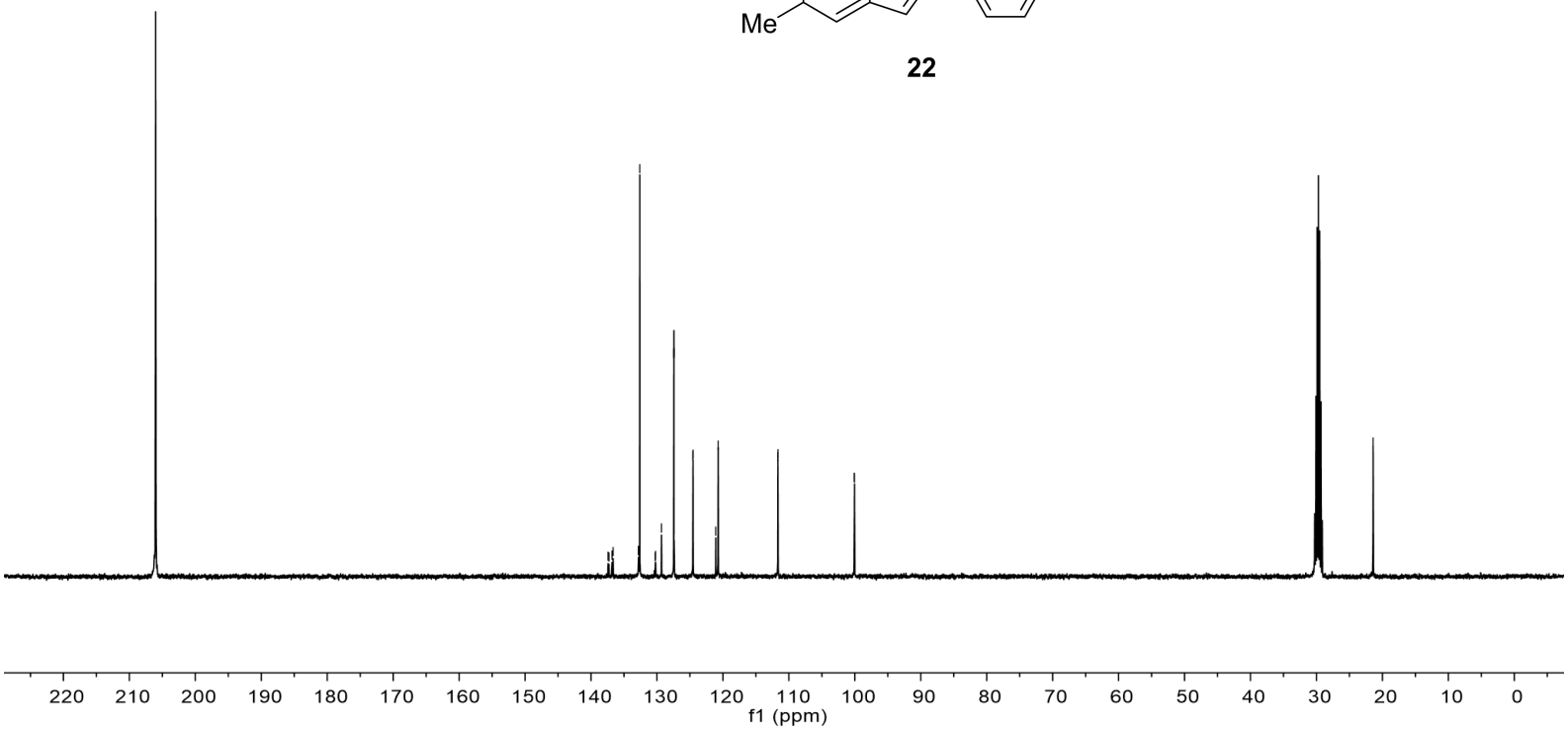


HJW2020-83-NMR-HC.2021010804.fid — 13C HJW2020-83

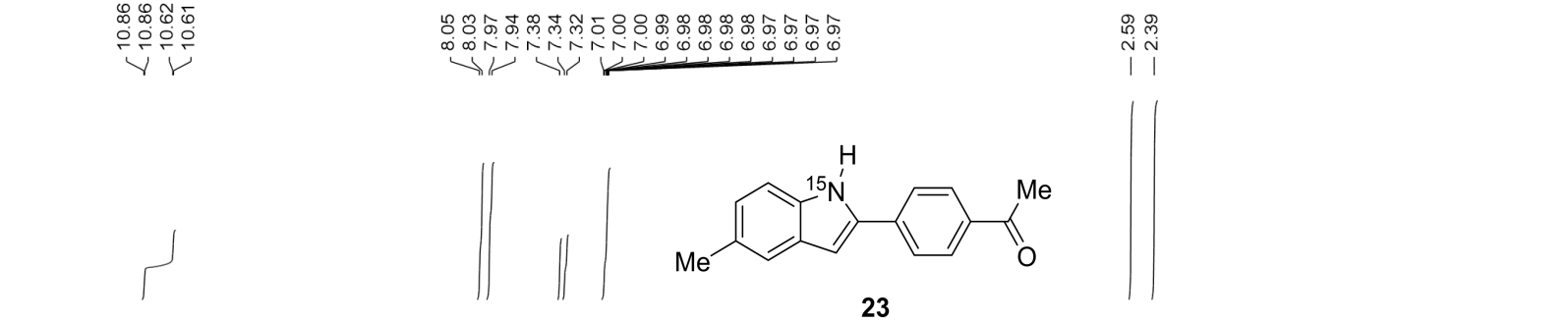
137.43
137.28
136.81
136.66
132.80
132.78
132.61
130.25
130.22
129.32
127.44
127.42
124.56
124.53
121.09
120.73
111.67
111.65
100.09
100.05
21.41



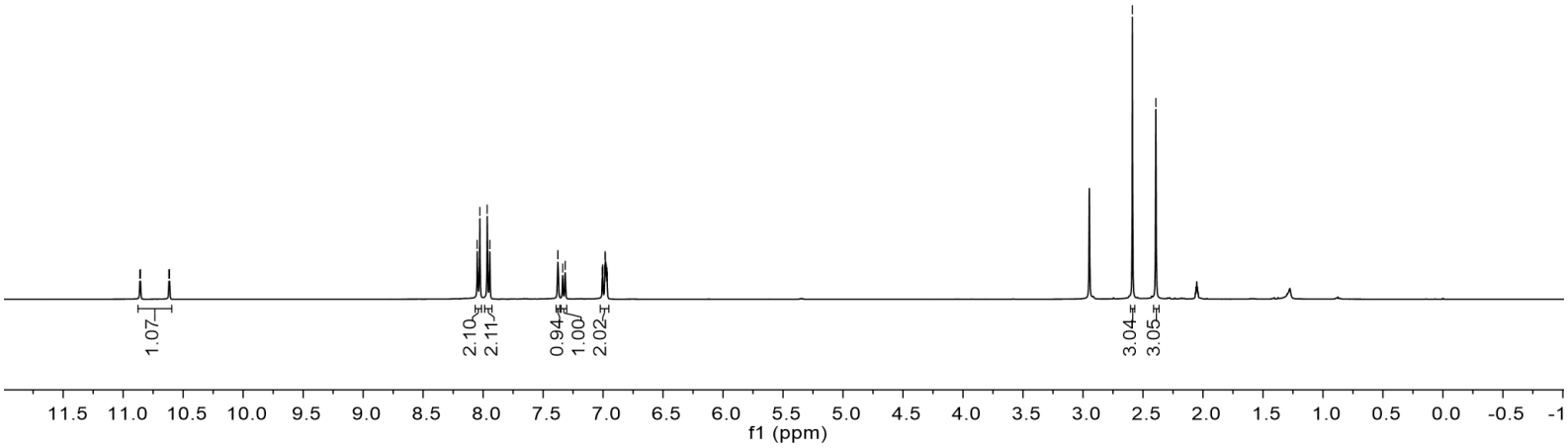
22



HJW2020-101-NMR-HC.2020120410.fid — 1H HJW2020-101-NMR-H/C

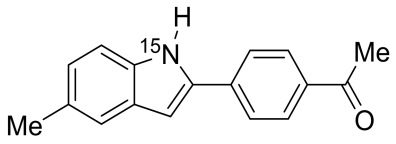


23

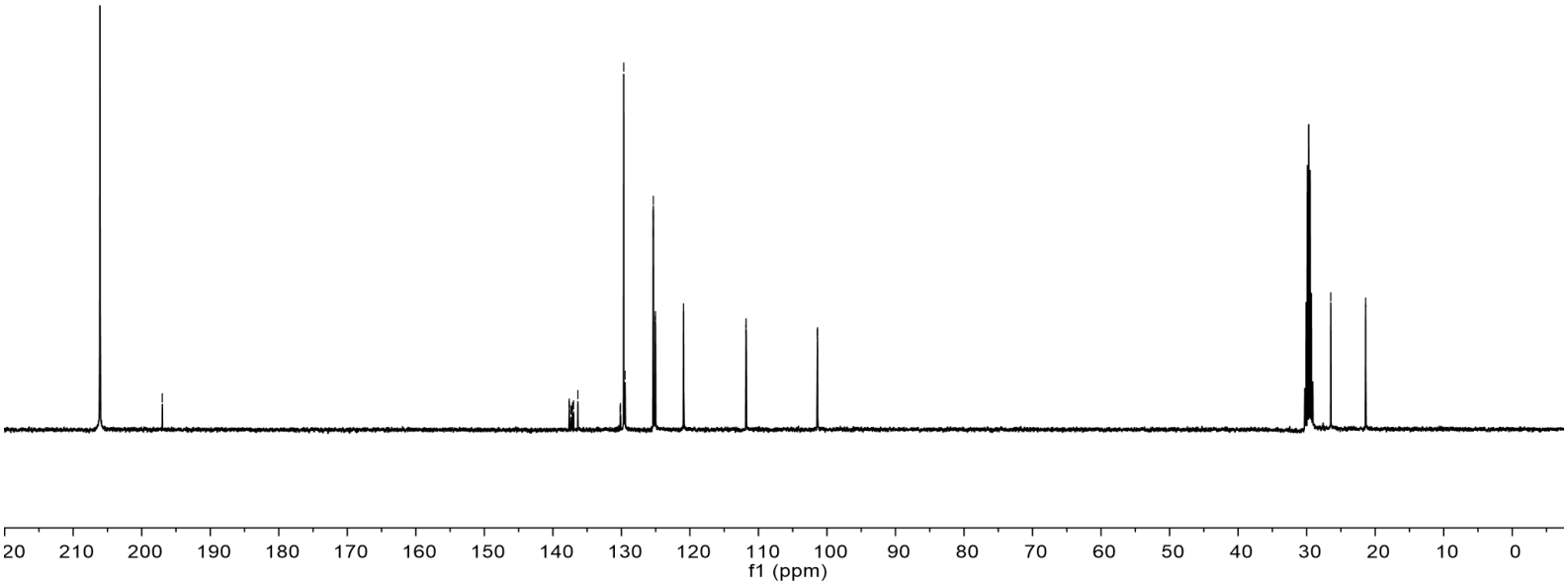


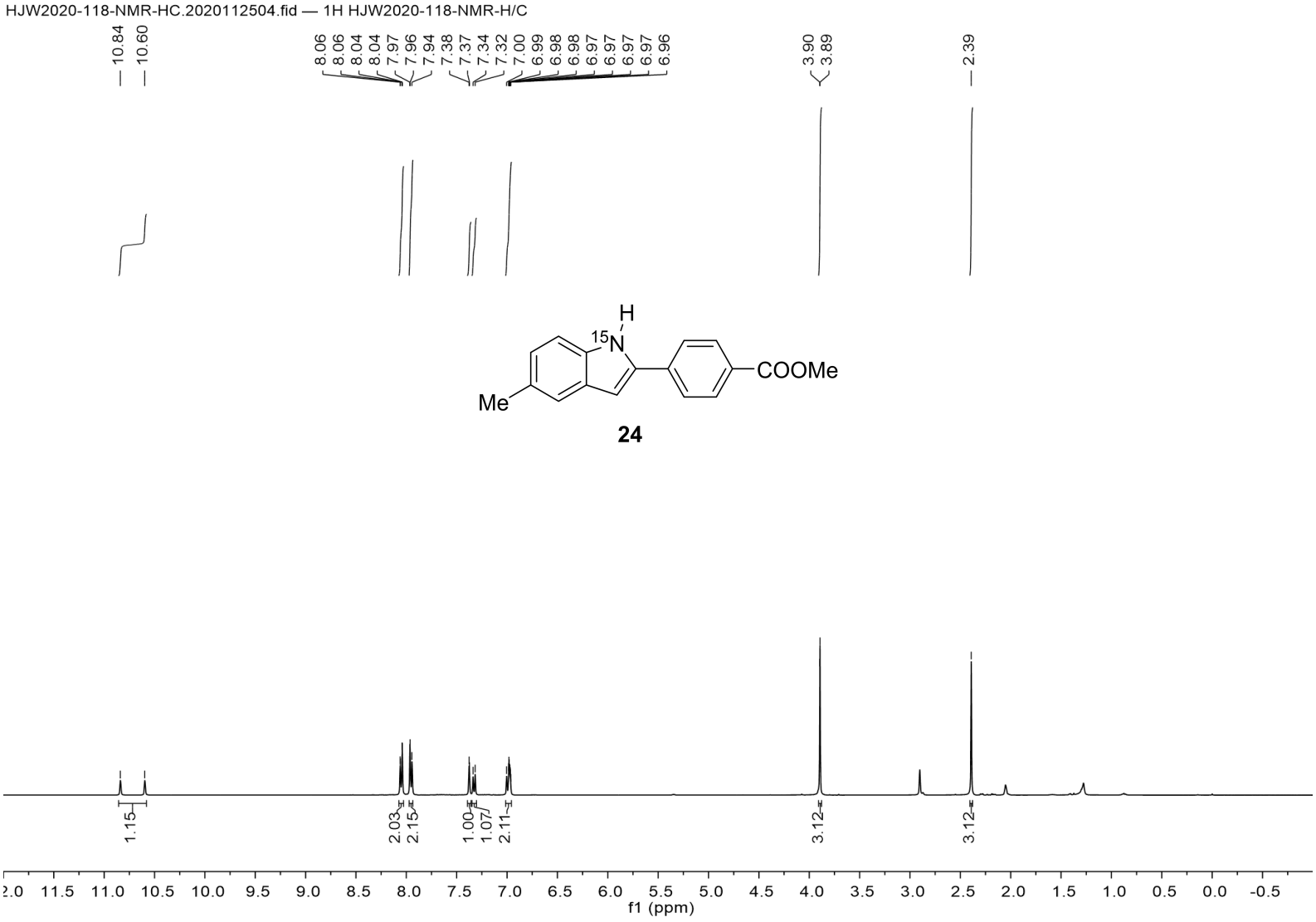
HJW2020-101-NMR-HC.2020120411.fid — 13C HJW2020-101-NMR-H/C

196.99
137.64
137.61
137.36
137.22
137.13
136.98
136.35
130.17
130.13
129.66
129.47
125.35
125.35
125.34
125.05
125.03
120.92
111.81
111.79
101.42
101.39
26.49
21.40



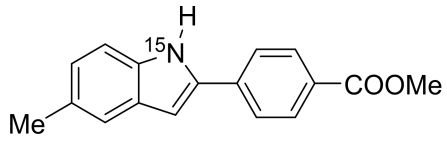
23



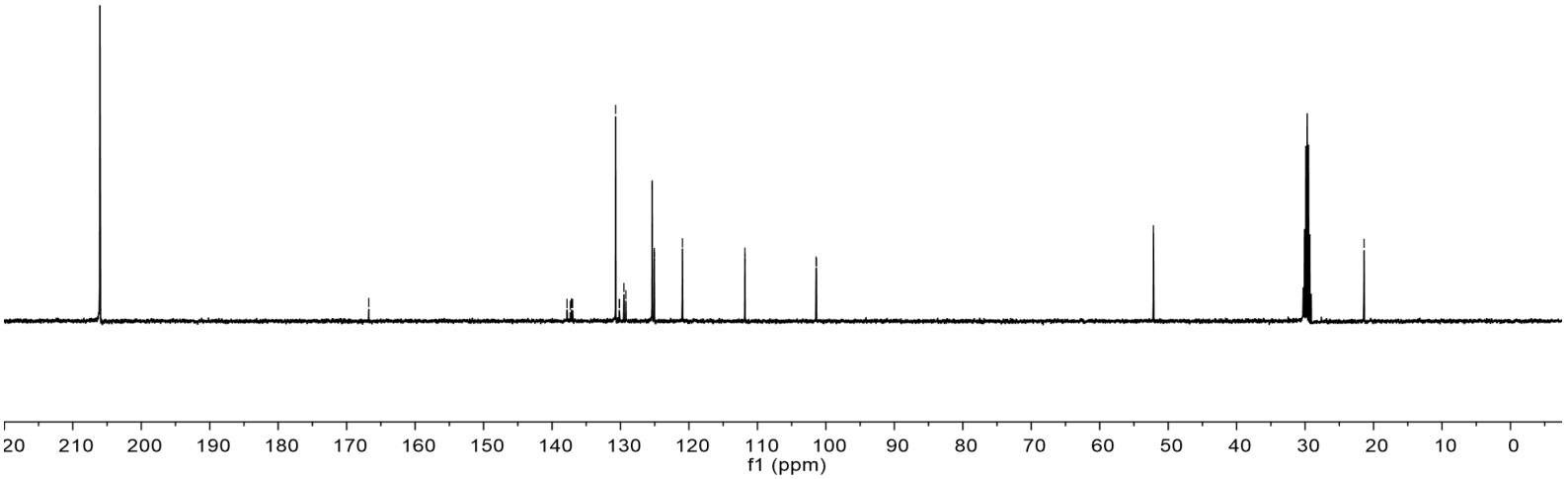

HJW2020-118-NMR-HC.2020112504.fid — 1H HJW2020-118-NMR-H/C
10.84
10.60
8.06
8.06
8.04
8.04
7.97
7.96
7.94
7.38
7.37
7.34
7.32
7.00
6.99
6.98
6.98
6.97
6.97
6.97
6.96
3.90
3.89
2.39
H
15N
COOMe
Me
24
1.15
2.03
2.15
1.00
1.07
2.11
3.12
3.12
f1 (ppm)

HJW2020-118-NMR-HC.2020112505.fid — 13C HJW2020-118-NMR-H/C

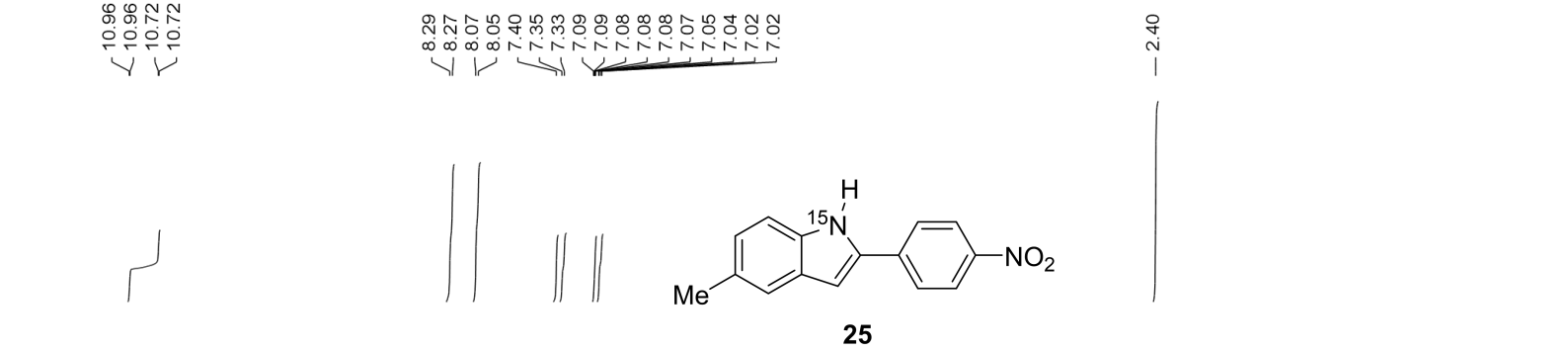
166.77
137.81
137.33
137.18
137.14
136.99
130.71
130.18
130.14
129.50
129.21
125.38
125.36
125.07
125.05
120.95
111.83
111.81
101.42
101.39
52.15
21.40



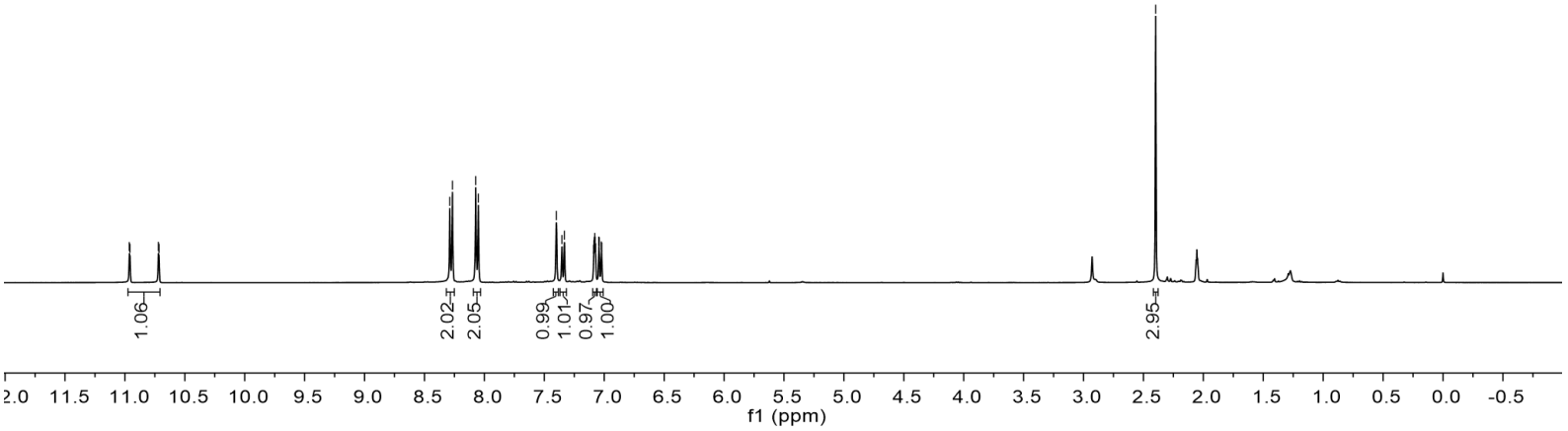
24

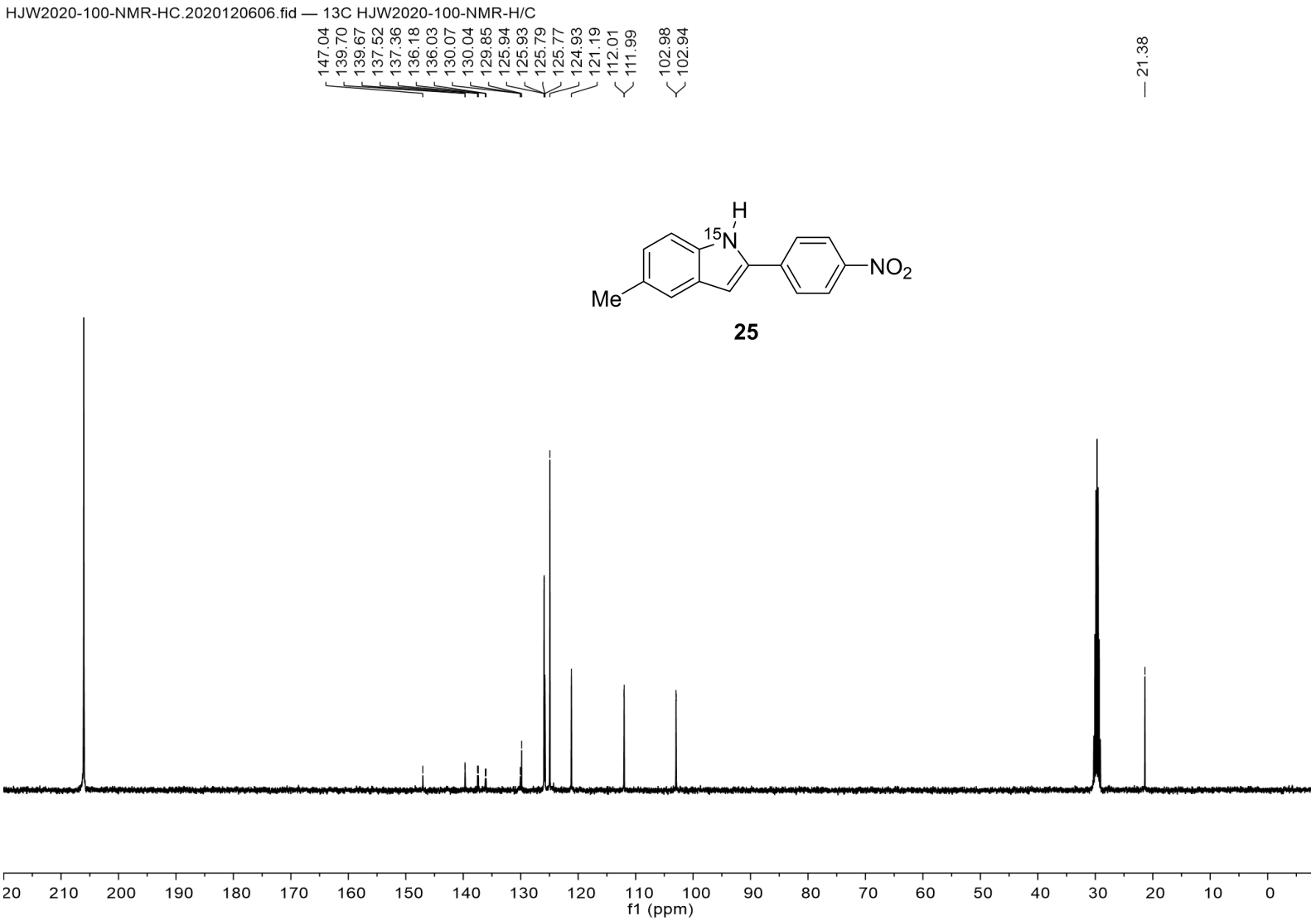


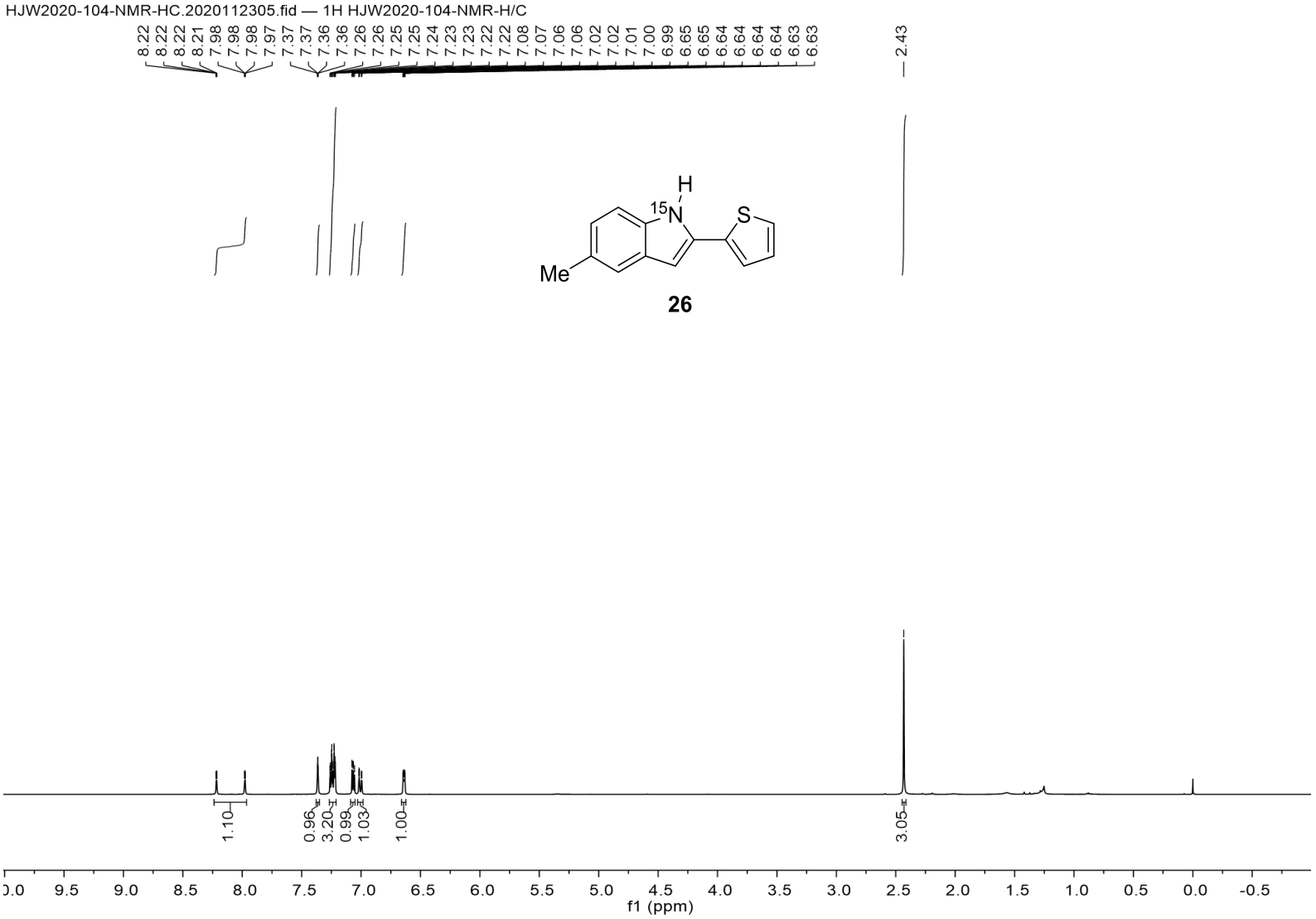
HJW2020-100-NMR-HC.2020120605.fid — 1H HJW2020-100-NMR-H/C



25

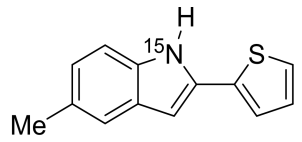



HJW2020-100-NMR-HC.2020120606.fid — 13C HJW2020-100-NMR-H/C
147.04
139.70
139.67
137.52
137.36
136.18
136.03
130.07
130.04
129.85
125.94
125.93
125.79
125.77
124.93
121.19
112.01
111.99
102.98
102.94
21.38
H
15N
NO2
Me
25
f1 (ppm)

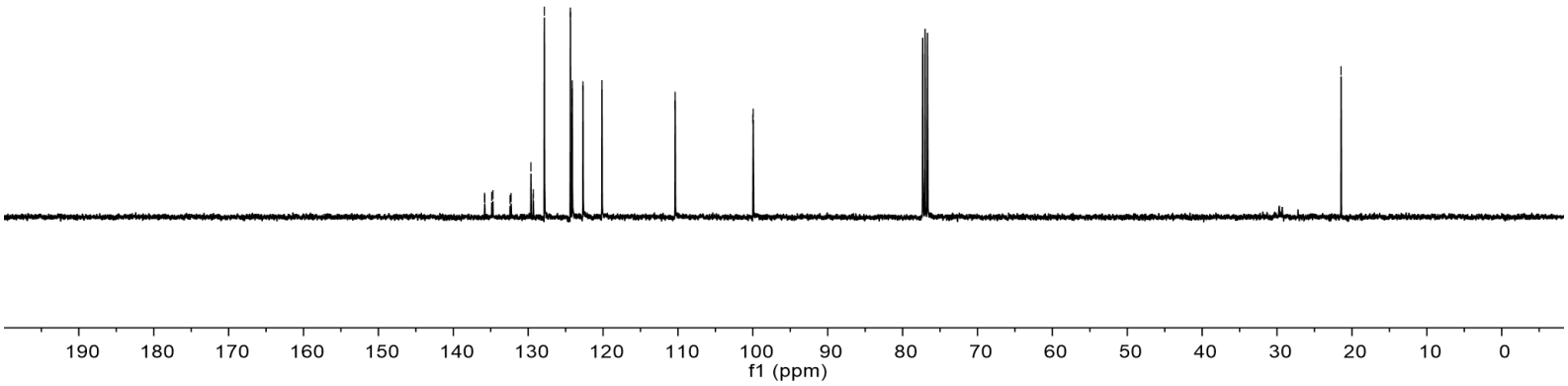

HJW2020-104-NMR-HC.2020112305.fid — 1H HJW2020-104-NMR-H/C
8.22 8.22 8.22 8.21 7.98 7.98 7.98 7.97 7.37 7.37 7.36 7.36 7.26 7.26 7.25 7.25 7.24 7.23 7.23 7.22 7.22 7.08 7.07 7.06 7.06 7.02 7.02 7.01 7.00 6.99 6.65 6.65 6.64 6.64 6.64 6.64 6.63 6.63
2.43
H
15N
S
Me
26
1.10
0.96
3.20
0.99
1.03
1.00
3.05
f1 (ppm)

HJW2020-104-NMR-HC.2020112307.fid — 13C HJW2020-104-NMR-H/C

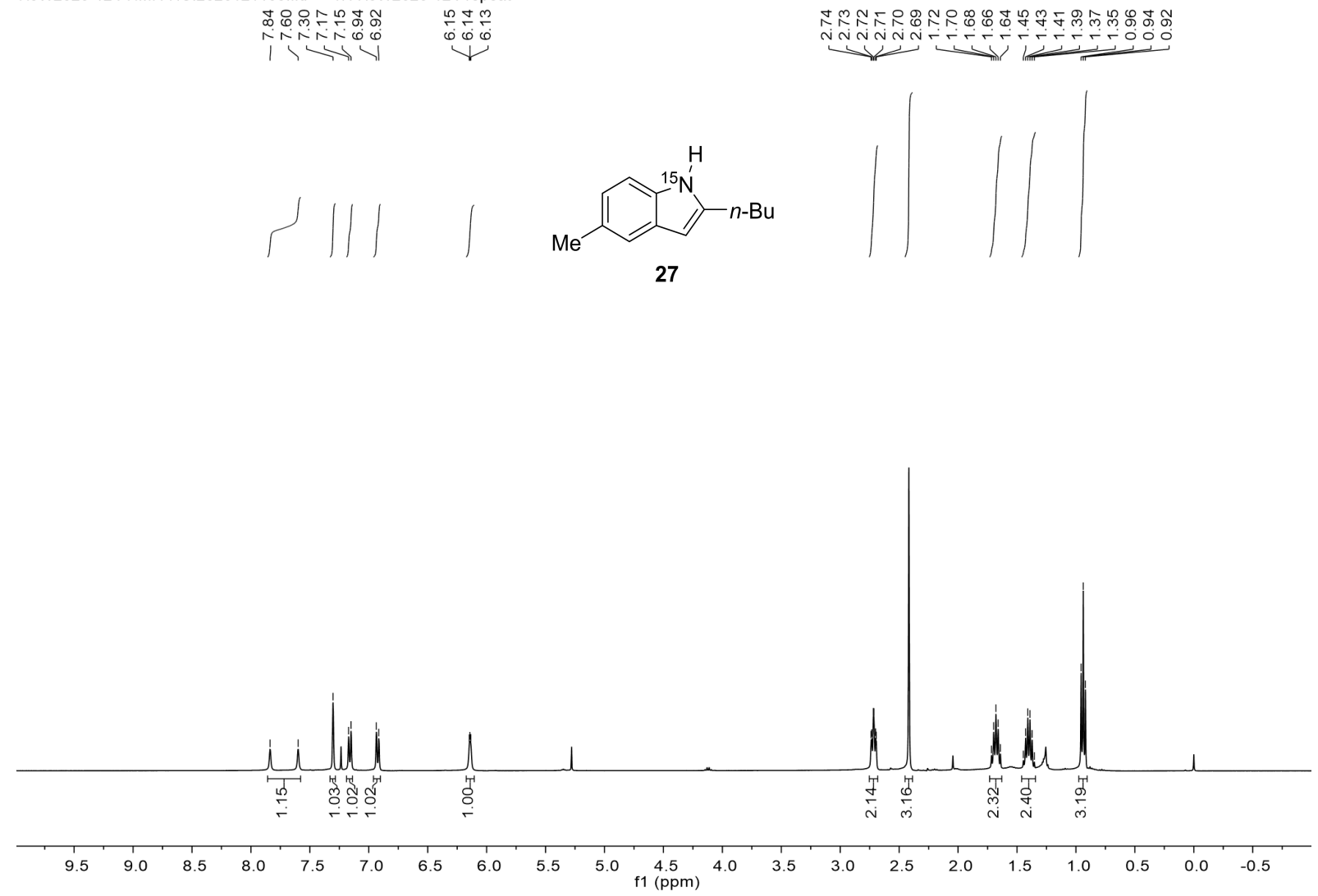
135.81
135.79
134.87
134.71
132.42
132.28
129.63
129.36
129.31
127.84
124.37
124.13
124.11
122.68
120.15
110.38
110.37
99.98
99.94
21.44



26



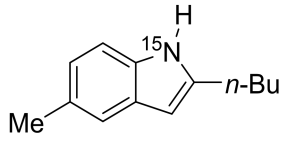
HJW2020-124-NMR-HC.2020121105.fid — 1H HJW2020-124-repeat



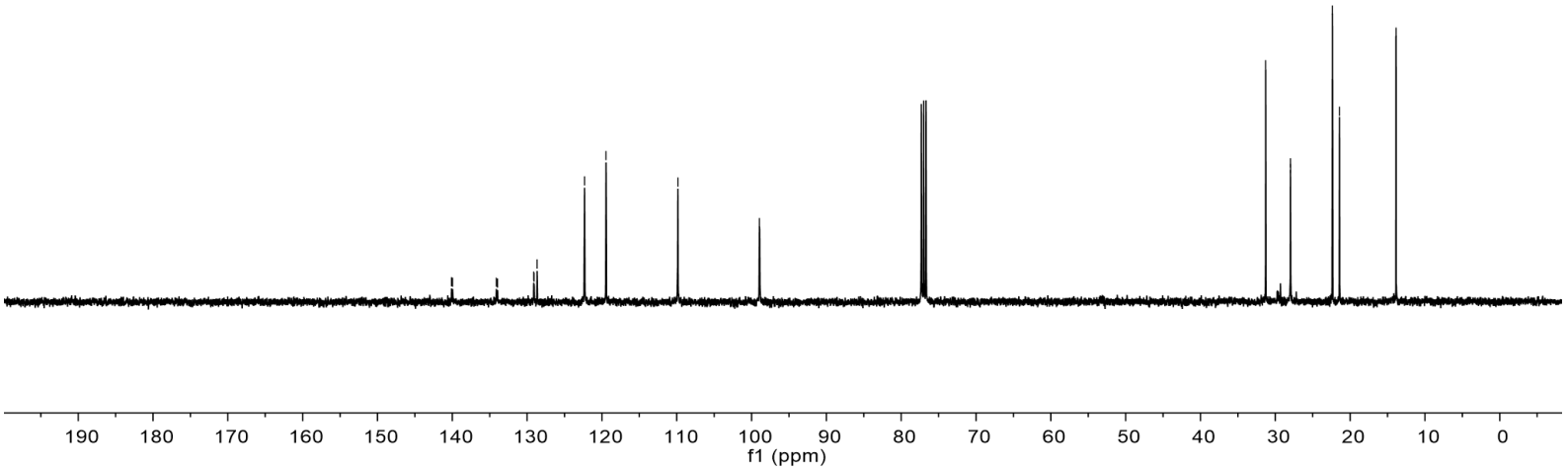
27

HJW2020-124-NMR-HC.2020121108.fid — HJW 124 13C

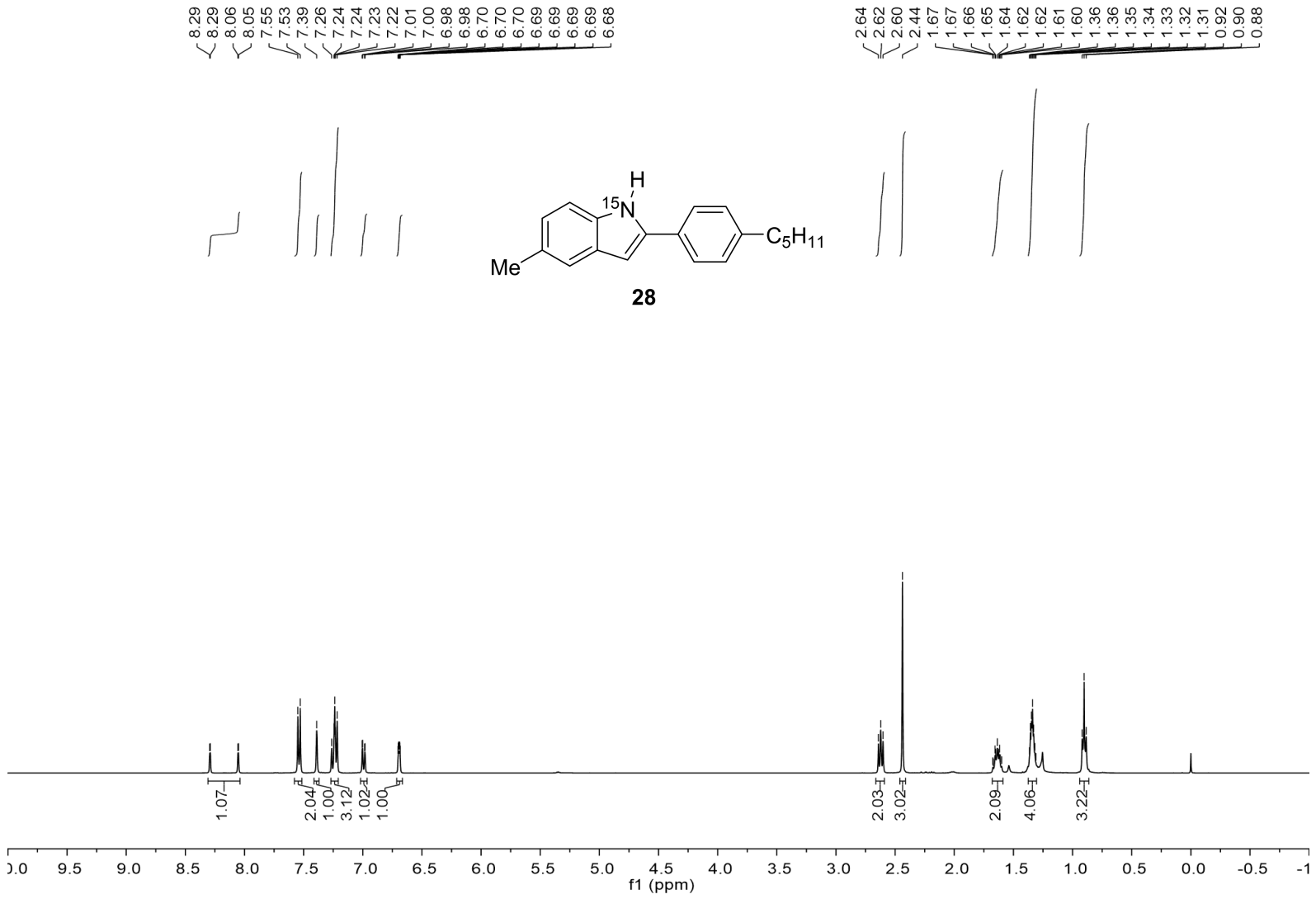
140.10
139.97
134.11
133.95
129.12
129.07
128.66
122.33
119.45
109.86
98.95
31.29
27.98
27.96
22.36
21.42
13.86



27



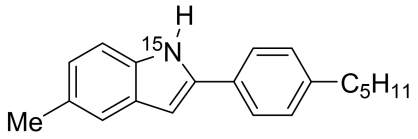
HJW2020-90-NMR-HC.2020111003.fid — 1H HJW2020-90-NMR-H/C



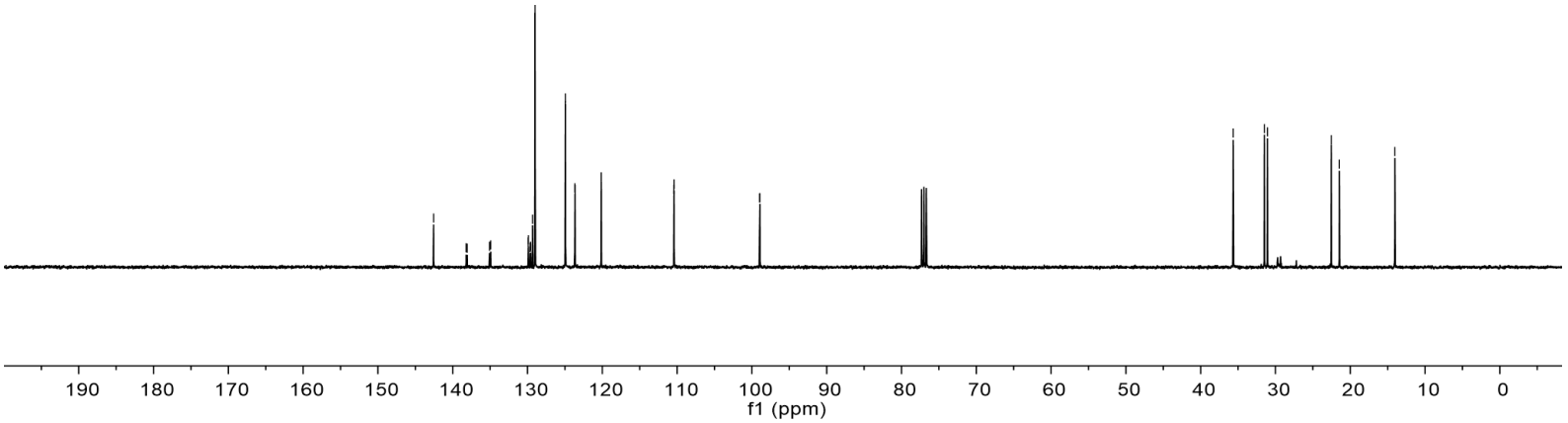
28

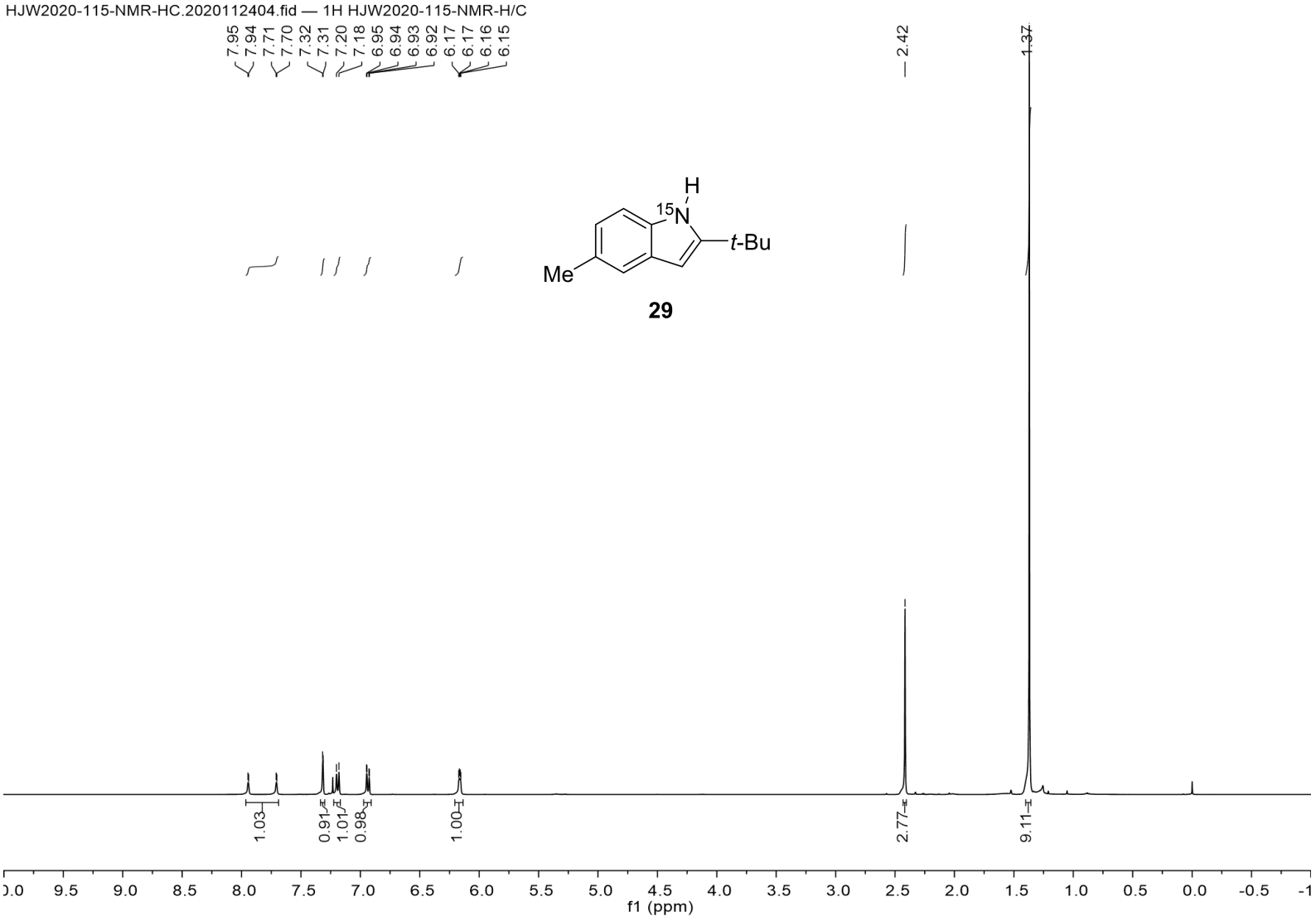
HJW2020-90-NMR-HC.2020111105.fid — 13C HJW2020-90-NMR-H/C

142.56
138.20
138.06
135.08
134.92
129.90
129.88
129.64
129.59
129.33
128.99
124.96
124.95
123.68
123.65
120.15
110.43
110.41
98.97
98.93
35.65
31.49
31.08
22.54
21.45
14.03



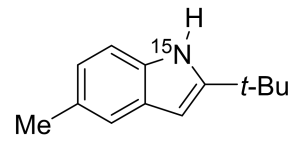
28



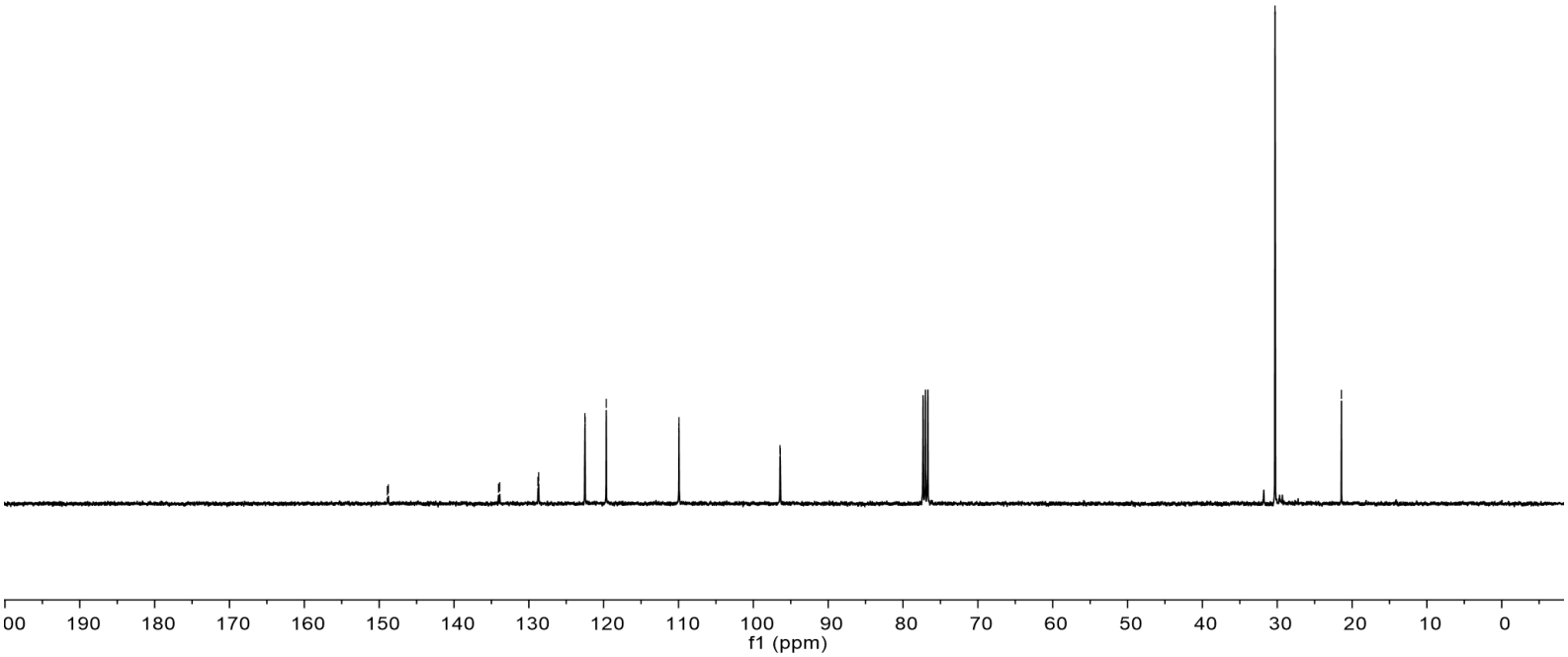

HJW2020-115-NMR-HC.2020112404.fid — 1H HJW2020-115-NMR-H/C
7.95
7.94
7.71
7.70
7.32
7.31
7.20
7.18
6.95
6.94
6.93
6.92
6.17
6.16
6.15
2.42
1.37
H
15N
t-Bu
Me
29
1.03
0.91
1.01
0.98
1.00
2.77
9.11
f1 (ppm)

HJW2020-115-NMR-HC.2020112408.fid — 13C HJW2020-115-NMR-H/C

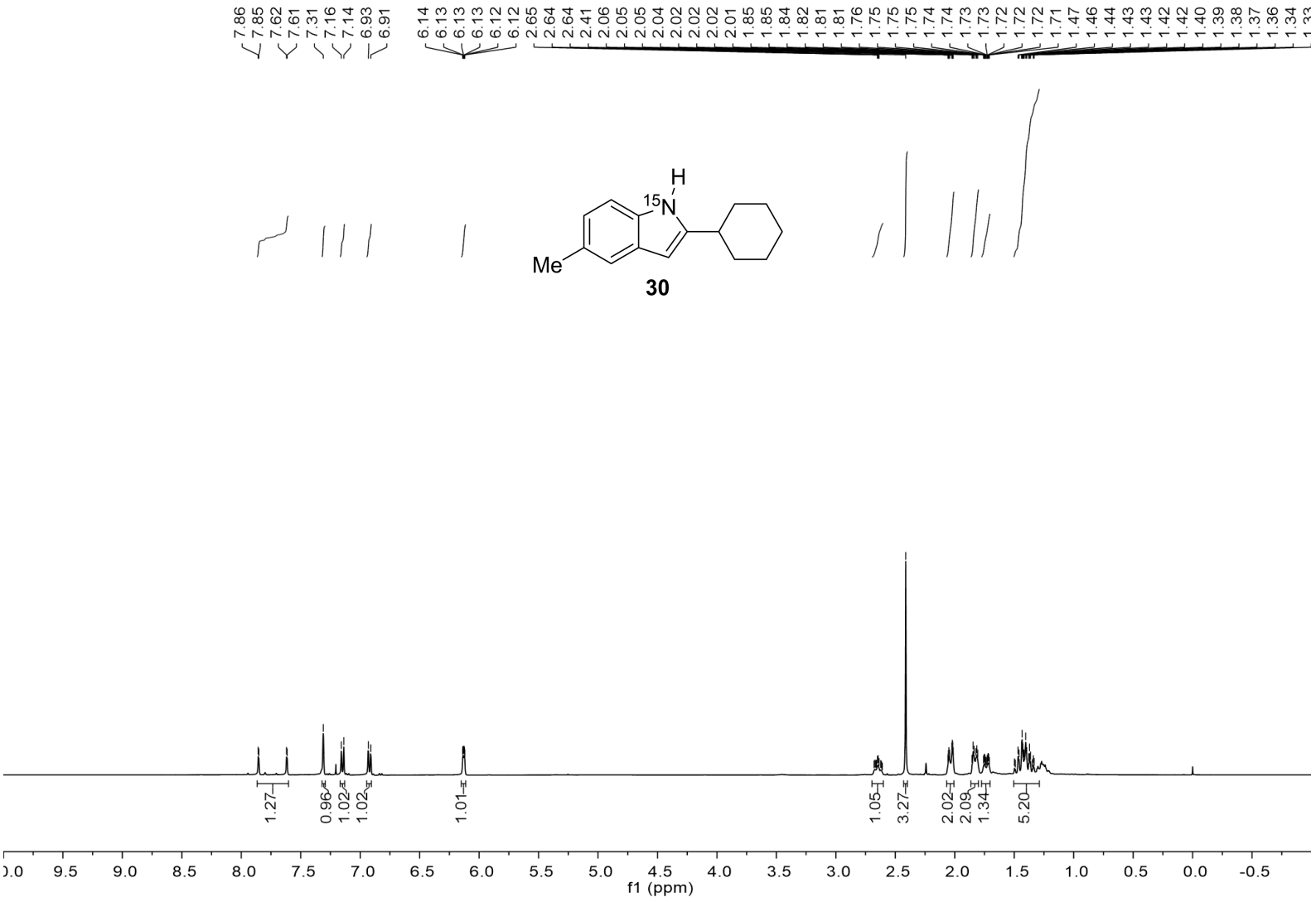
148.90
148.77
134.05
133.90
128.78
128.73
128.70
122.51
122.49
119.66
109.94
109.93
96.44
96.40
30.29
21.42



29

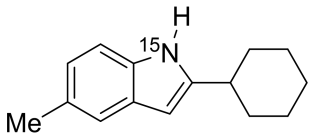


HJW2020-88-NMR-HC.2020120206.fid — 1H HJW2020-88-NMR-H/C

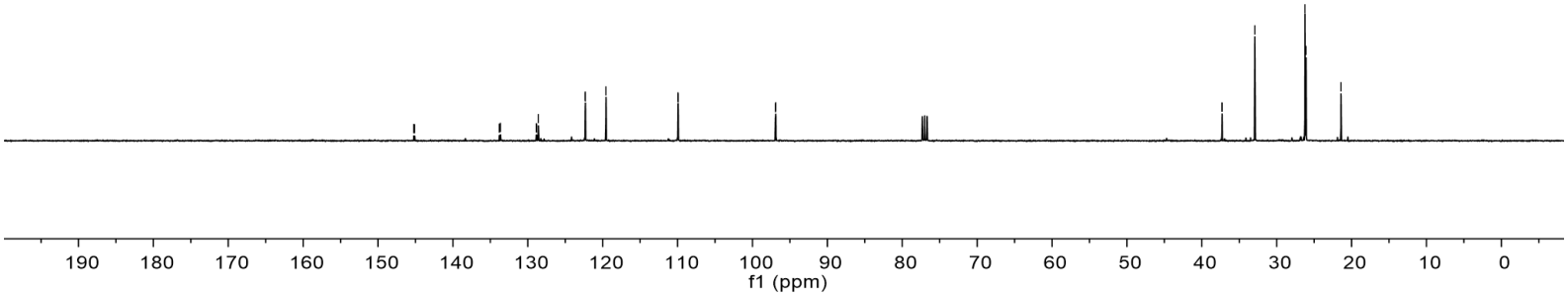


HJW2020-88-NMR-HC.2020120209.fid — 13C HJW2020-88-NMR-H/C

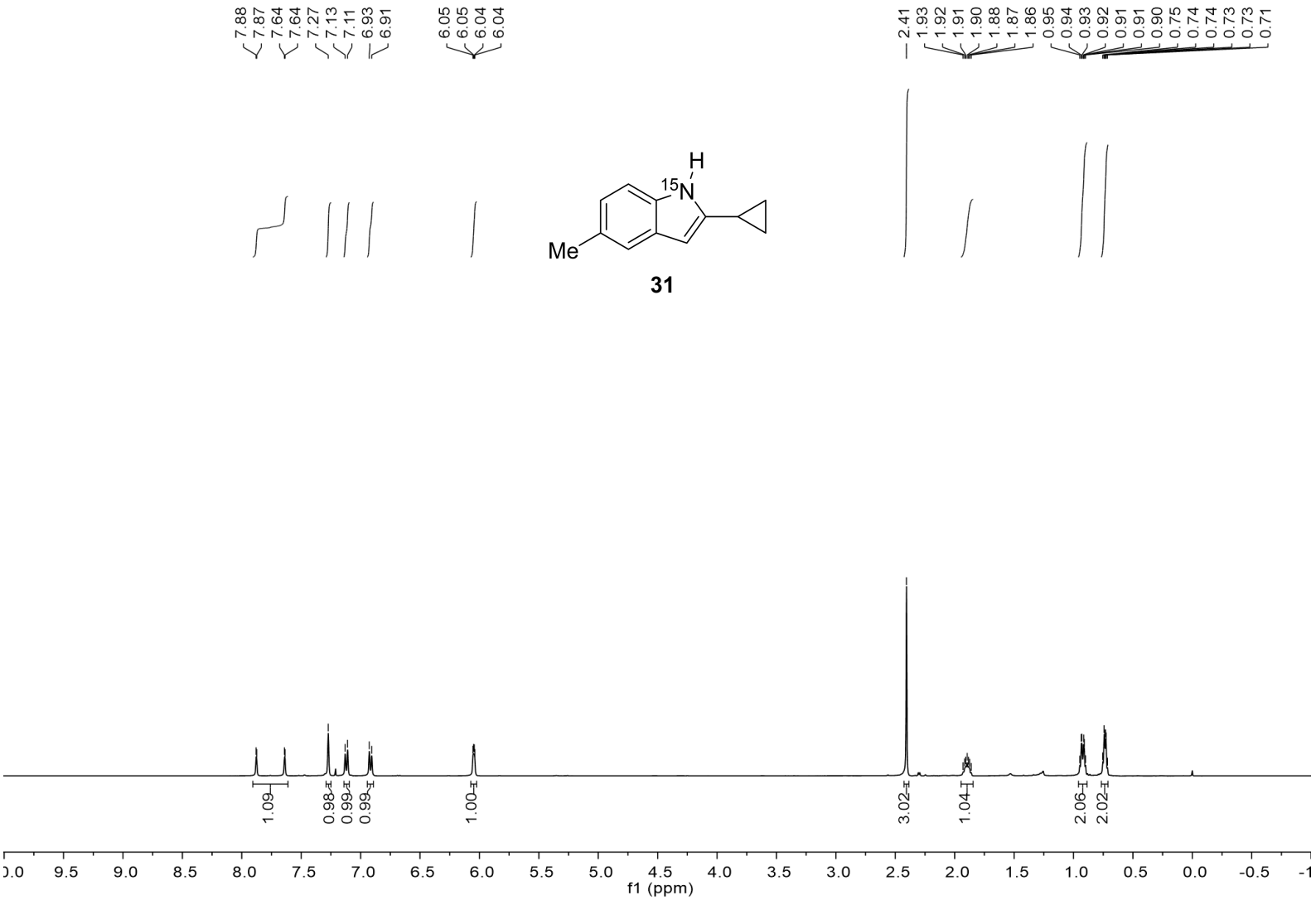
145.23
145.11
133.81
133.65
128.87
128.82
128.59
122.33
122.31
119.56
109.94
109.93
96.93
96.89
37.31
37.29
32.92
26.22
26.08
21.41



30



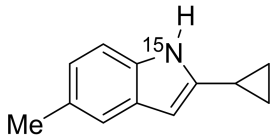
HJW2020-116-NMR-HC.2020120602.fid — 1H HJW2020-116-NMR-H/C



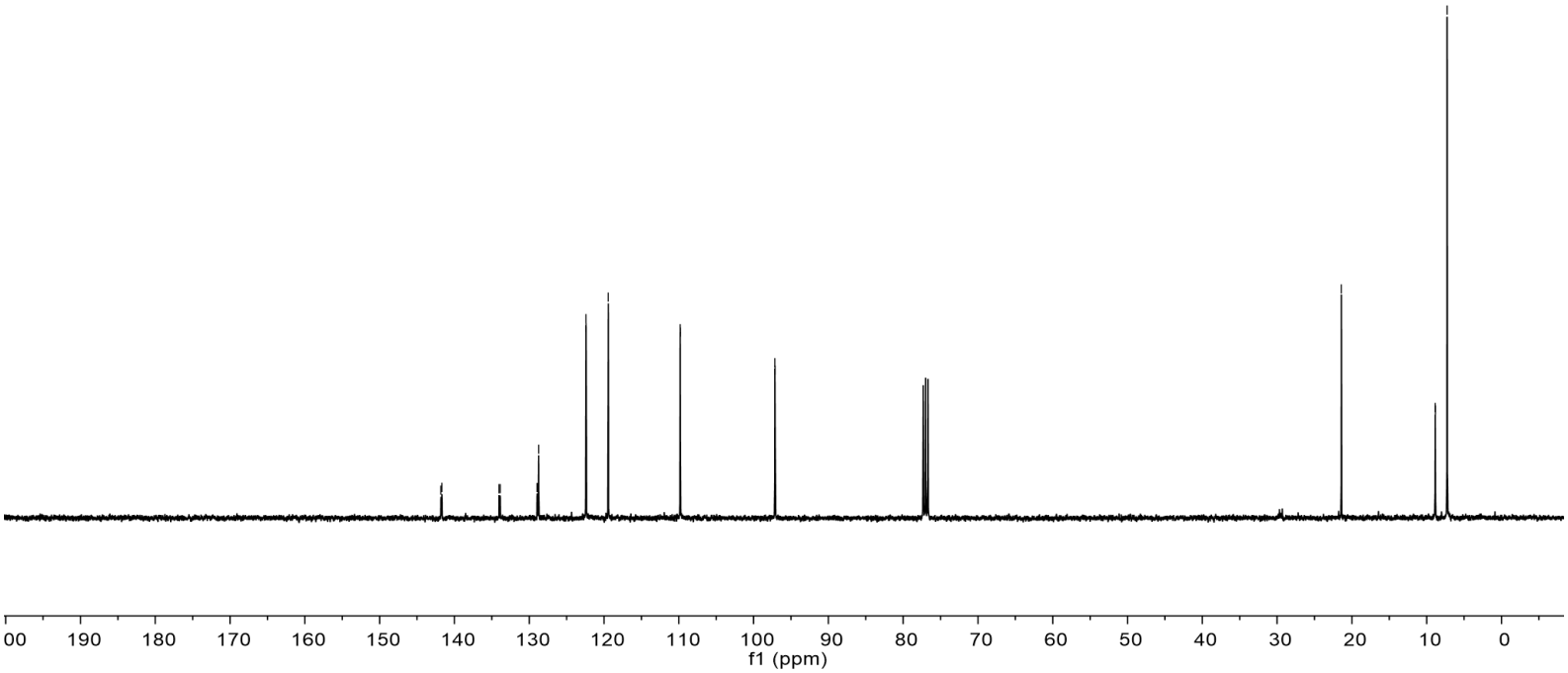
31

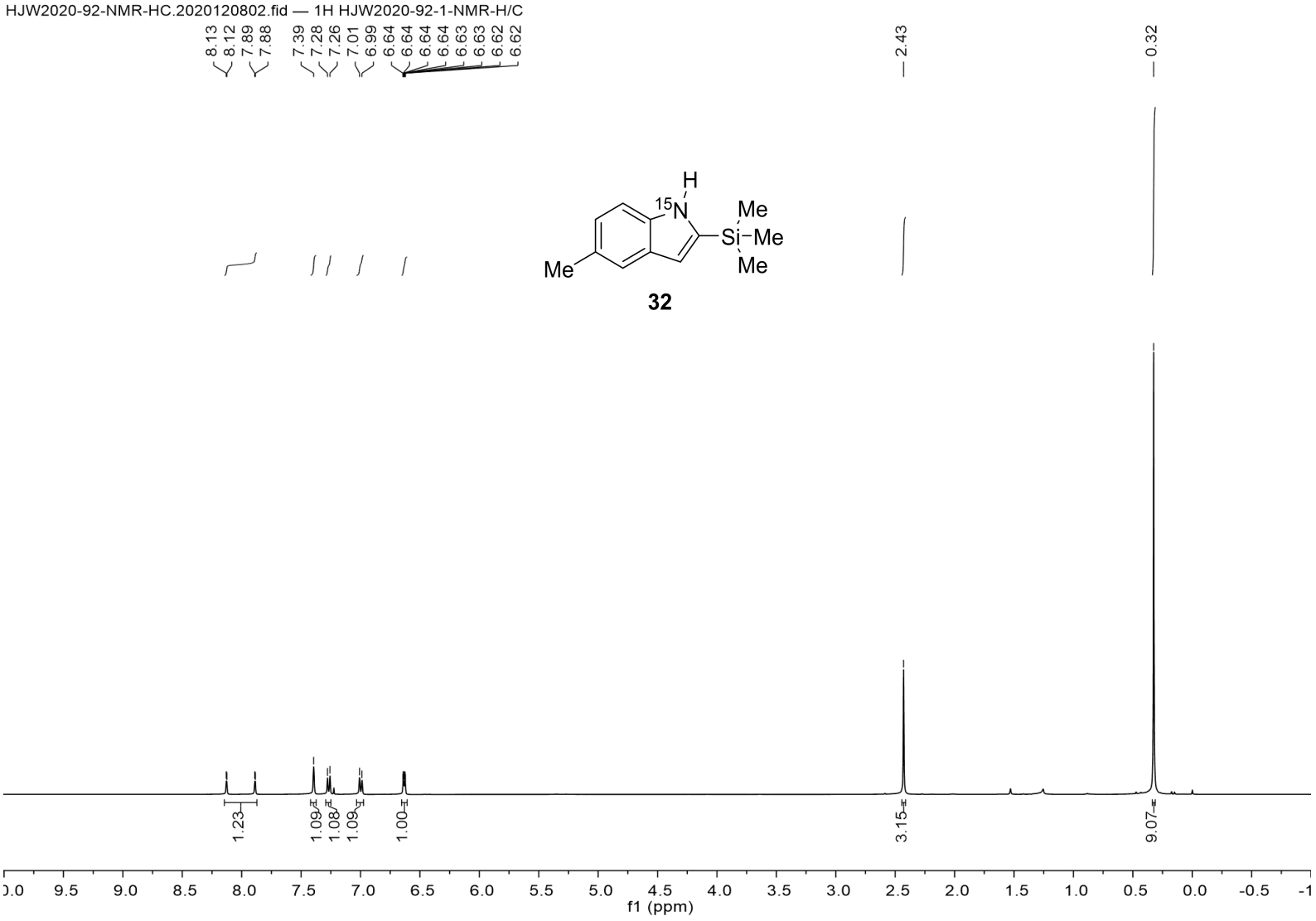
HJW2020-116-NMR-HC.2020120701.fid — 13C HJW2020-116-NMR-H/C

141.81
141.68
134.03
133.88
128.95
128.90
128.75
122.41
122.40
119.43
109.81
109.80
97.15
97.11
21.41
8.87
8.84
7.26



31



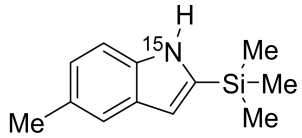

HJW2020-92-NMR-HC.2020120802.fid — 1H HJW2020-92-1-NMR-H/C
8.13
8.12
7.89
7.88
7.39
7.28
7.26
7.01
6.99
6.64
6.64
6.64
6.64
6.63
6.63
6.62
6.62
2.43
0.32
H
15N
Me
Si
Me
Me
Me
32
1.23
1.09
1.08
1.09
1.00
3.15
9.07
f1 (ppm)

HJW2020-92-NMR-HC.2020120806.fid — 13C HJW2020-92-1-repeat-NMR-H/C

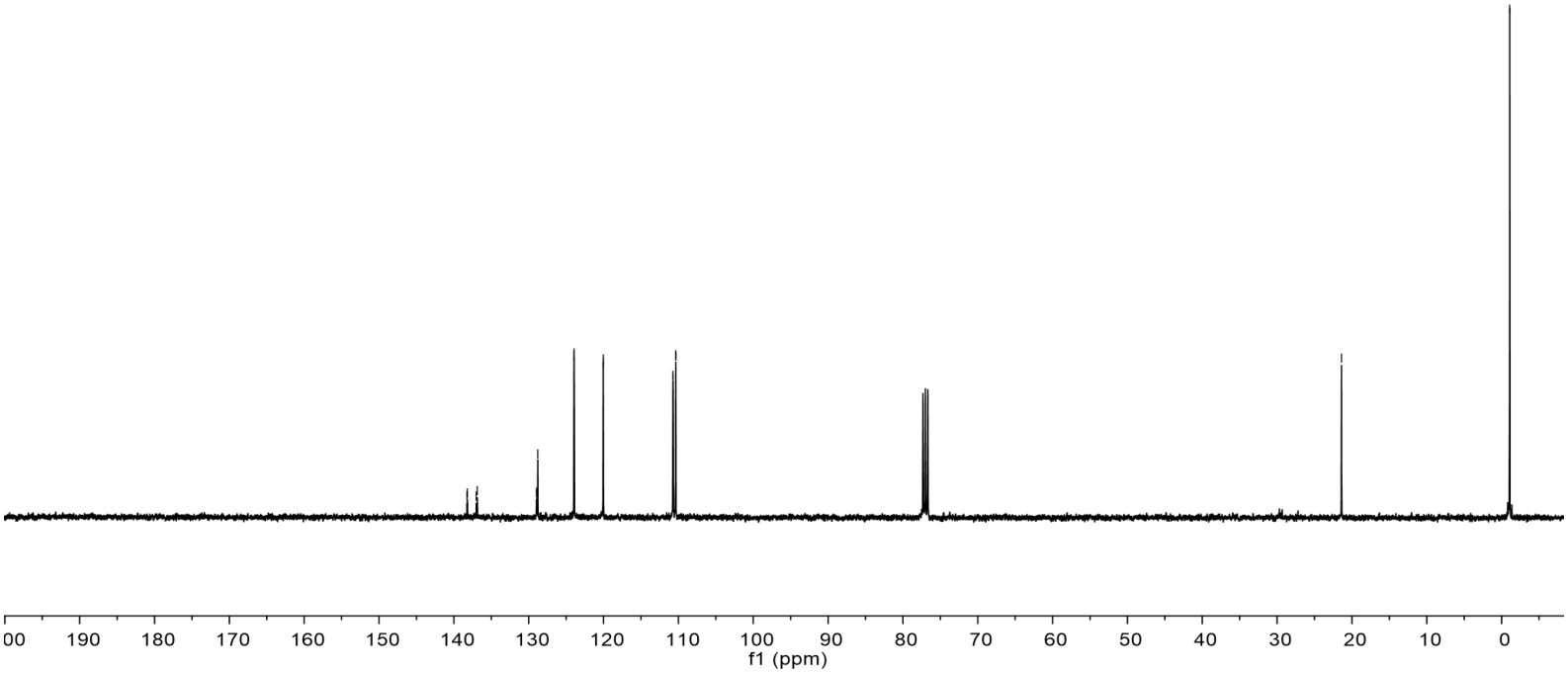
138.26
138.19
137.02
136.87
128.97
128.91
128.79
123.95
123.93
120.05
120.04
110.73
110.70
110.36
110.34

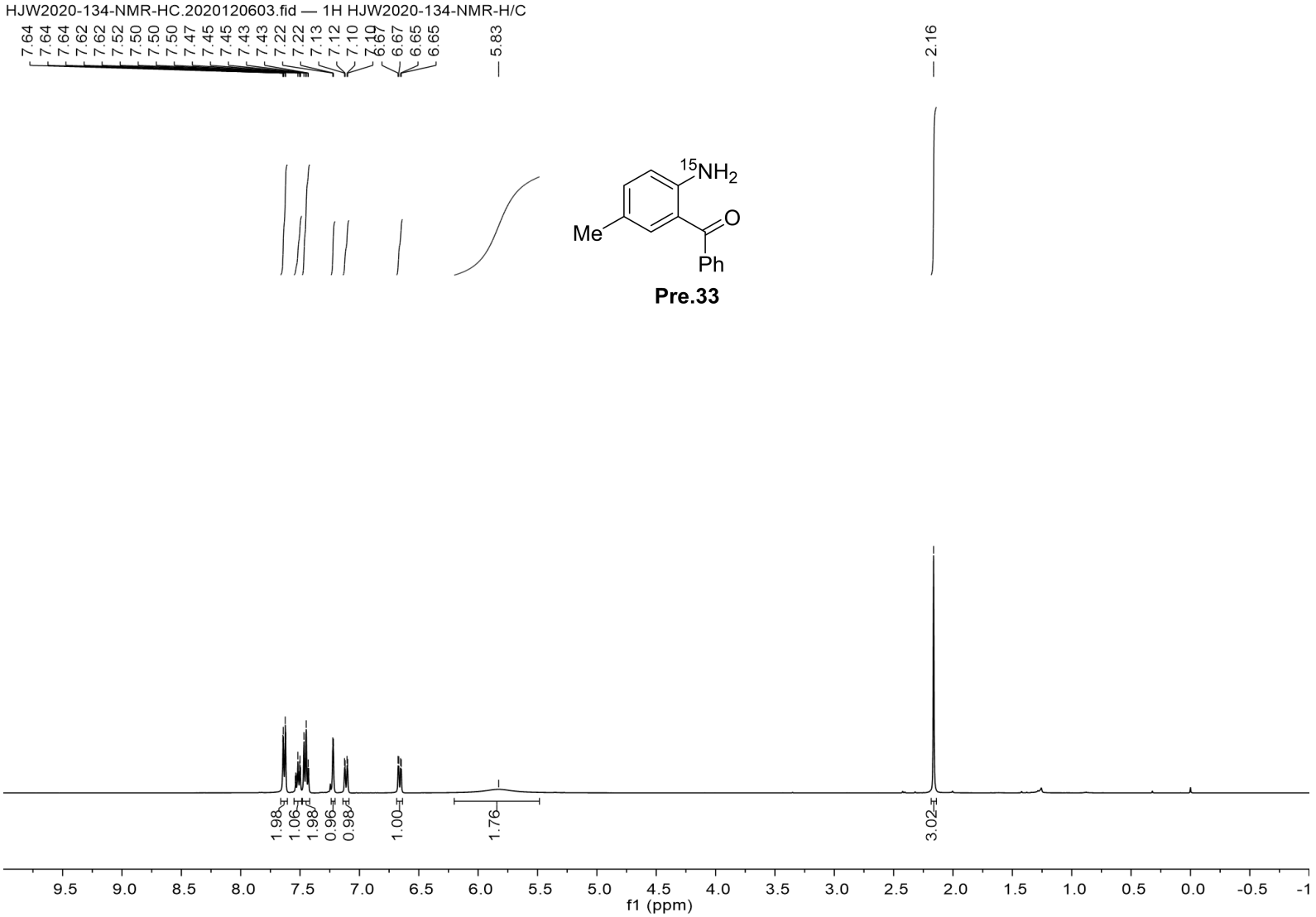
21.39

-1.08



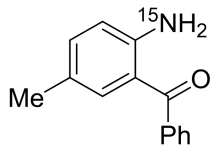
32



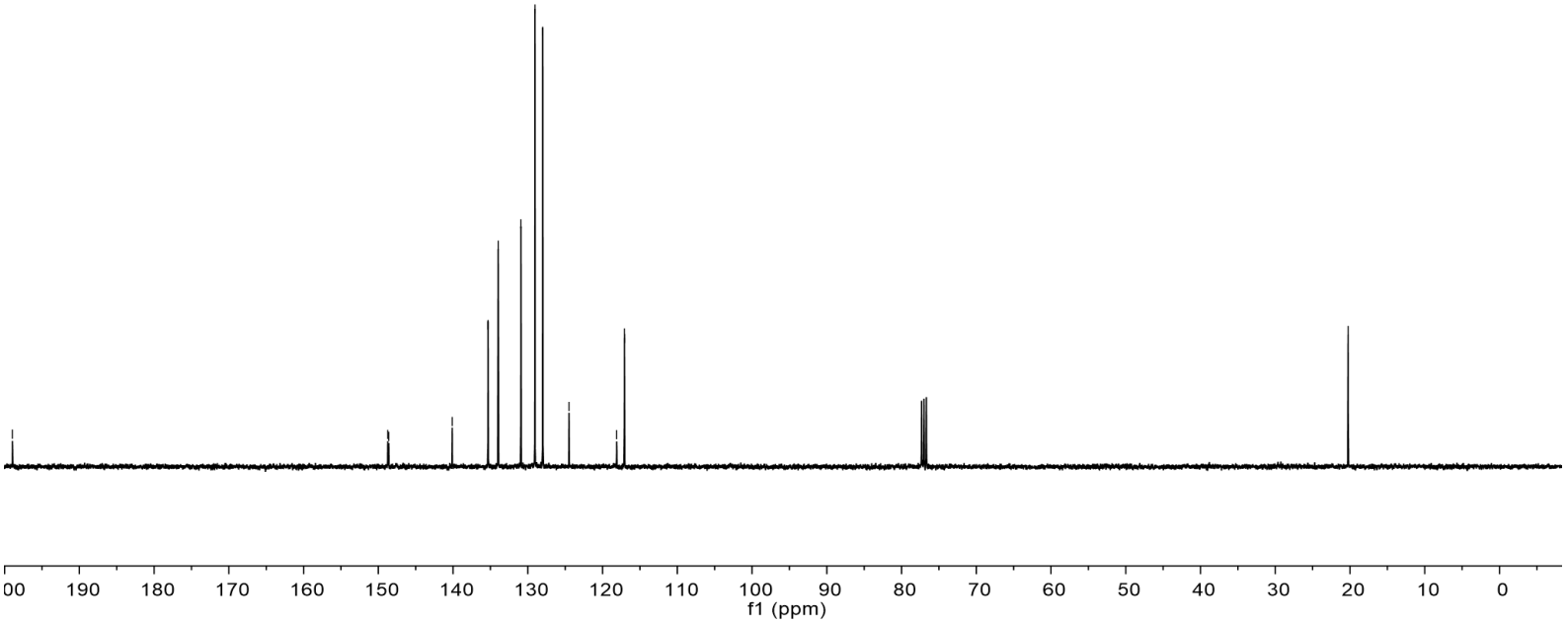

HJW2020-134-NMR-HC.2020120603.fid — 1H HJW2020-134-NMR-H/C
7.64
7.64
7.64
7.62
7.62
7.52
7.50
7.50
7.50
7.47
7.45
7.45
7.43
7.43
7.22
7.22
7.13
7.12
7.10
7.10
6.67
6.67
6.65
6.65
5.83
2.16
15NH2
O
Me
Ph
Pre.33
1.98
1.06
1.98
0.96
0.98
1.00
1.76
3.02
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)

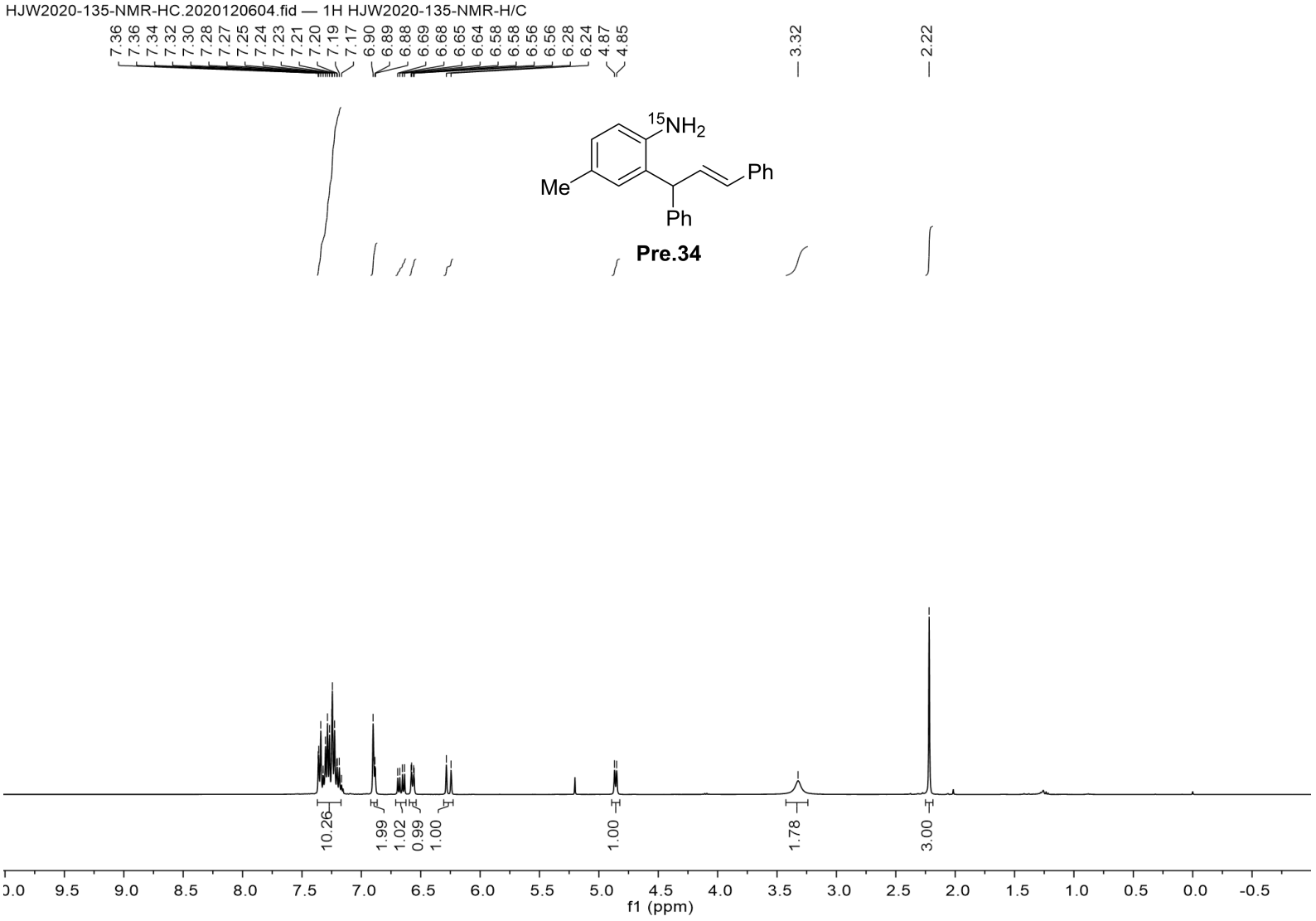
HJW2020-134-NMR-HC.2020120607.fid — 13C HJW2020-134-NMR-H/C

198.95
148.74
148.60
140.11
135.33
135.31
133.97
130.92
129.04
128.00
124.49
118.11
117.08
117.06
20.26



Pre.33



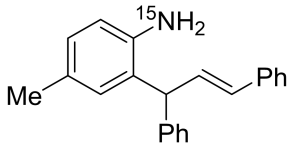

HJW2020-135-NMR-HC.2020120604.fid — 1H HJW2020-135-NMR-H/C
7.36
7.36
7.34
7.32
7.30
7.28
7.27
7.25
7.24
7.23
7.21
7.20
7.19
7.17
6.90
6.89
6.88
6.69
6.68
6.65
6.64
6.58
6.58
6.56
6.56
6.28
6.24
4.87
4.85
3.32
2.22
15NH2
Me
Ph
Ph
Pre.34
10.26
1.99
1.02
0.99
1.00
1.00
1.78
3.00
f1 (ppm)

HJW2020-135-NMR-HC.2020120608.fid — 13C HJW2020-135-NMR-H/C

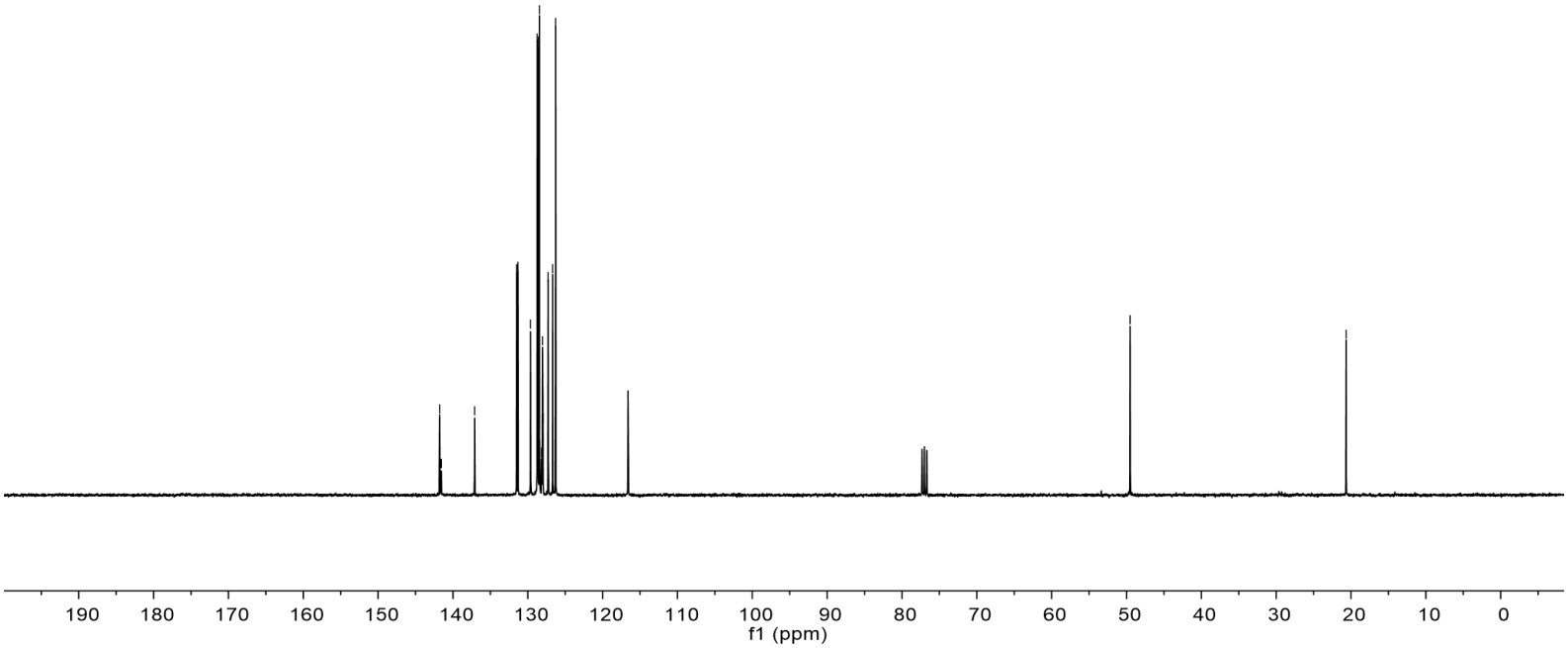
141.79
141.65
141.55
137.10
131.49
131.31
129.63
128.74
128.63
128.45
128.16
128.14
128.04
127.97
127.28
126.68
126.29
116.61
116.59

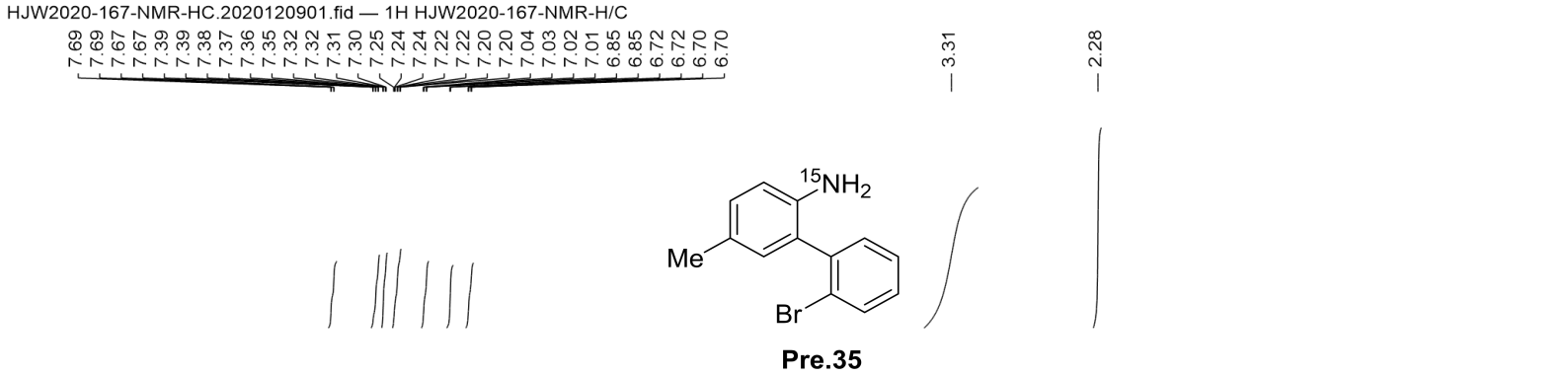
49.52

20.66



Pre.34



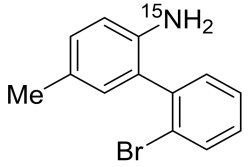

HJW2020-167-NMR-HC.2020120901.fid — 1H HJW2020-167-NMR-H/C
7.69
7.69
7.67
7.67
7.39
7.39
7.38
7.37
7.36
7.35
7.32
7.32
7.31
7.30
7.25
7.24
7.24
7.22
7.22
7.20
7.20
7.04
7.03
7.02
7.01
6.85
6.85
6.72
6.72
6.70
6.70
3.31
2.28
15NH2
Me
Br
1.03
1.12
1.16
1.21
1.03
0.97
1.00
2.16
3.07
9.5
9.0
8.5
8.0
7.5
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
-0.5
f1 (ppm)

Pre.35

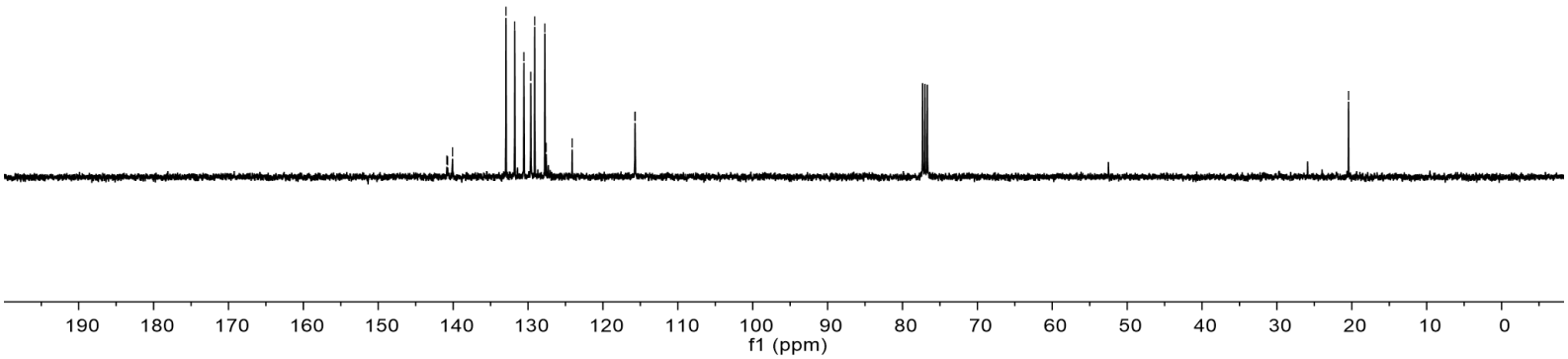
HJW2020-167-NMR-HC.2020120902.fid — 13C HJW2020-167

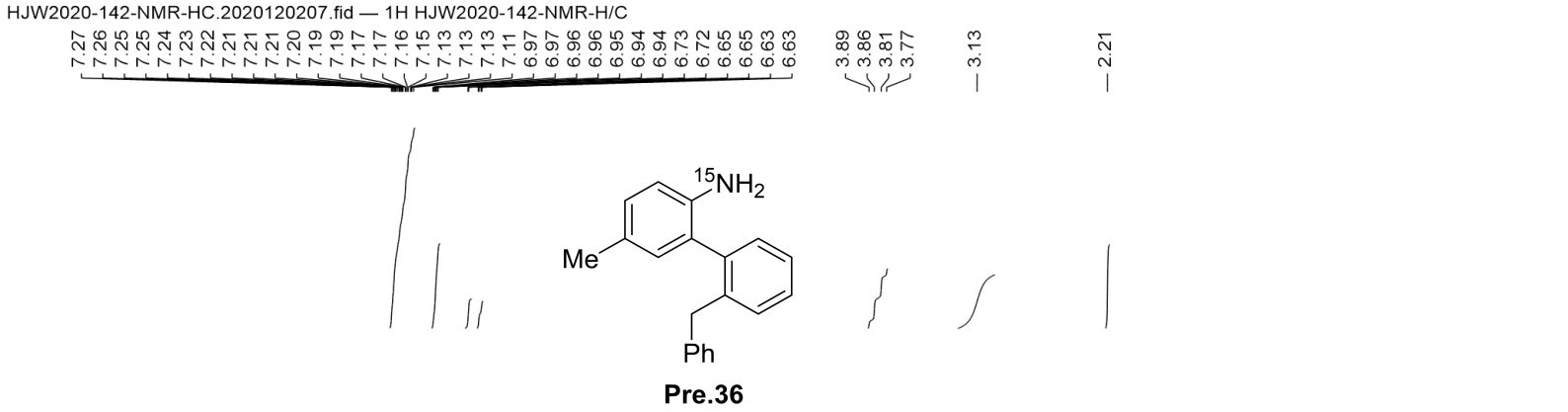
140.85
140.73
140.08
132.95
131.78
130.55
129.64
129.11
127.76
127.71
127.58
124.11
115.73
115.70

— 20.44

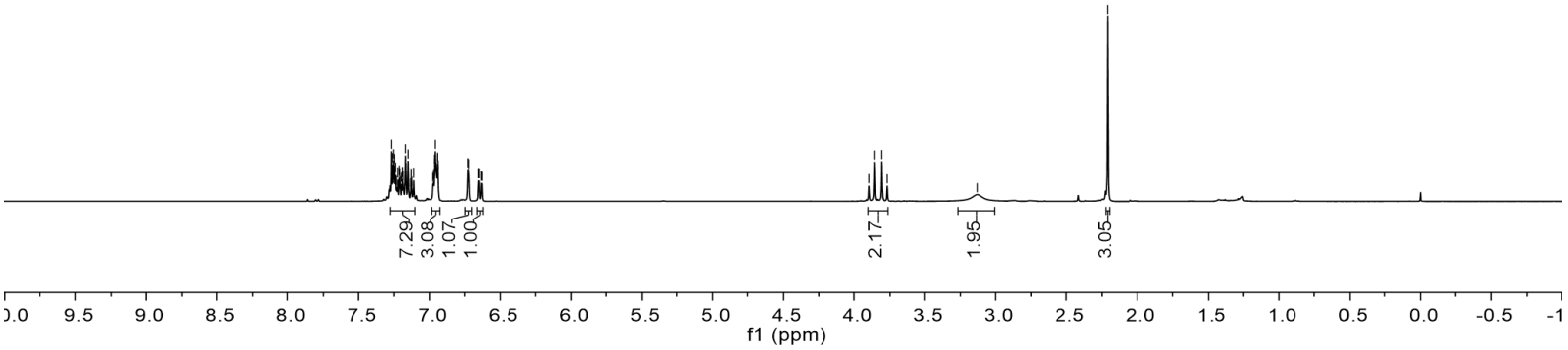


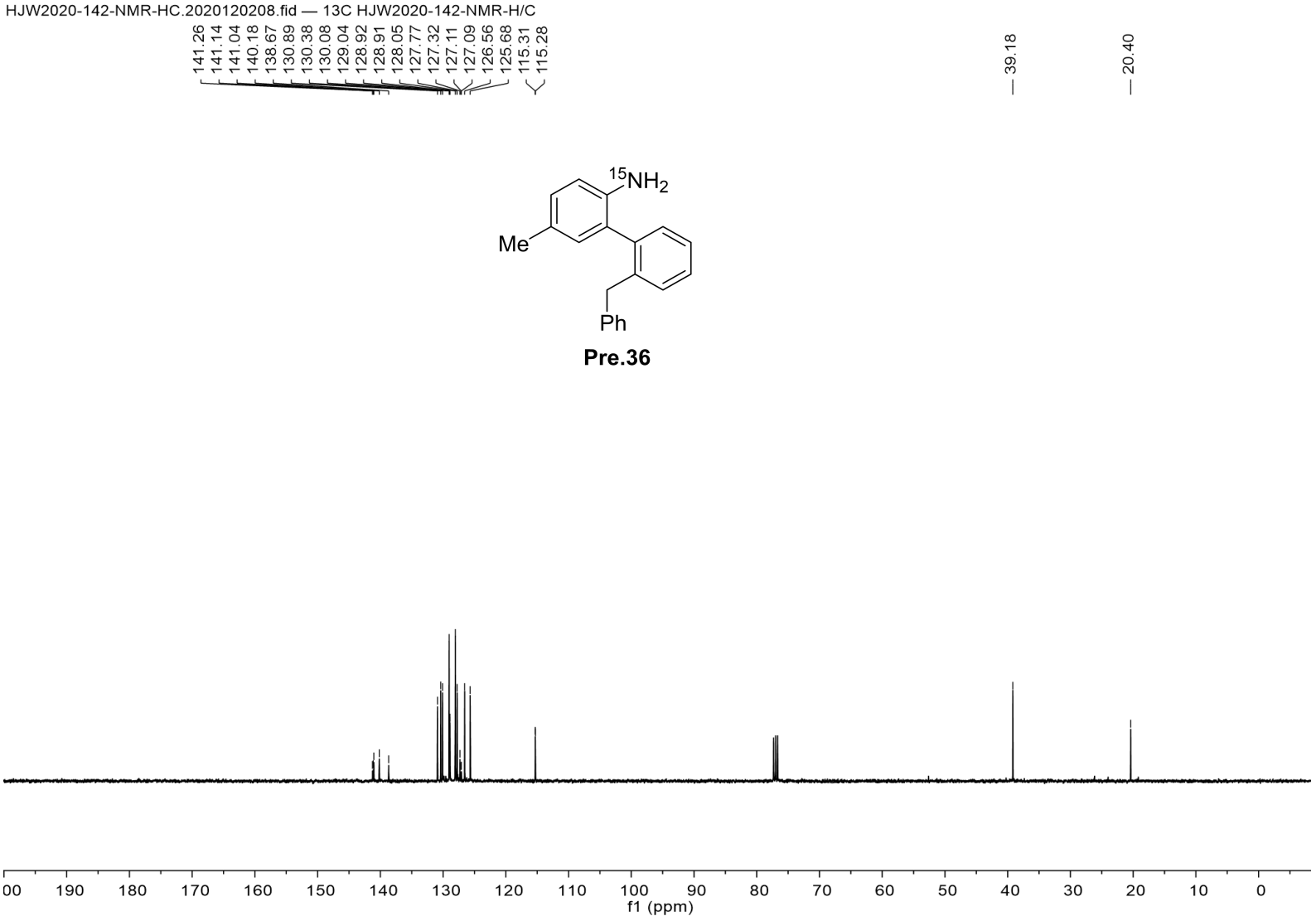
Pre.35




HJW2020-142-NMR-HC.2020120207.fid — 1H HJW2020-142-NMR-H/C
7.27 7.26 7.25 7.25 7.24 7.23 7.22 7.21 7.21 7.21 7.20 7.19 7.19 7.17 7.17 7.16 7.15 7.13 7.13 7.13 7.11 6.97 6.97 6.96 6.96 6.95 6.94 6.94 6.73 6.72 6.65 6.65 6.63 6.63
3.89 3.86 3.81 3.77
3.13
2.21
15NH2
Me
Ph

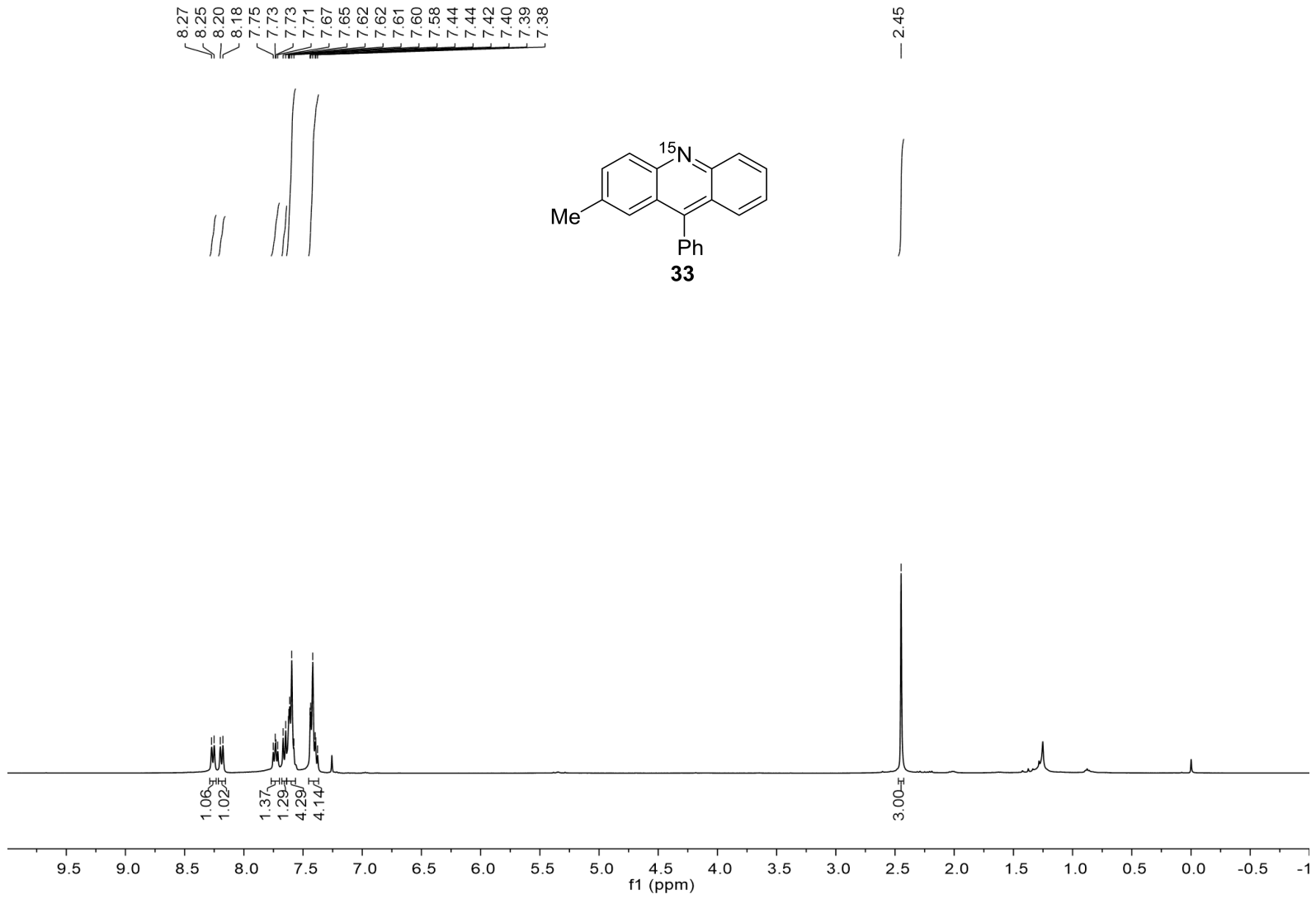
Pre.36


7.29
3.08
1.07
1.00
2.17
1.95
3.05
f1 (ppm)


HJW2020-142-NMR-HC.2020120208.fid — 13C HJW2020-142-NMR-H/C
141.26
141.14
141.04
140.18
138.67
130.89
130.38
130.08
129.04
128.92
128.91
128.05
127.77
127.32
127.11
127.09
126.56
125.68
115.31
115.28
39.18
20.40
15NH2
Me
Ph
f1 (ppm)

Pre.36

HJW2020-139-NMR-HC.2021010701.fid — 1H HJW2020-139-Purified



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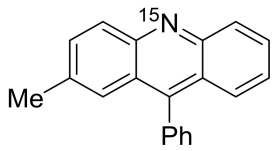
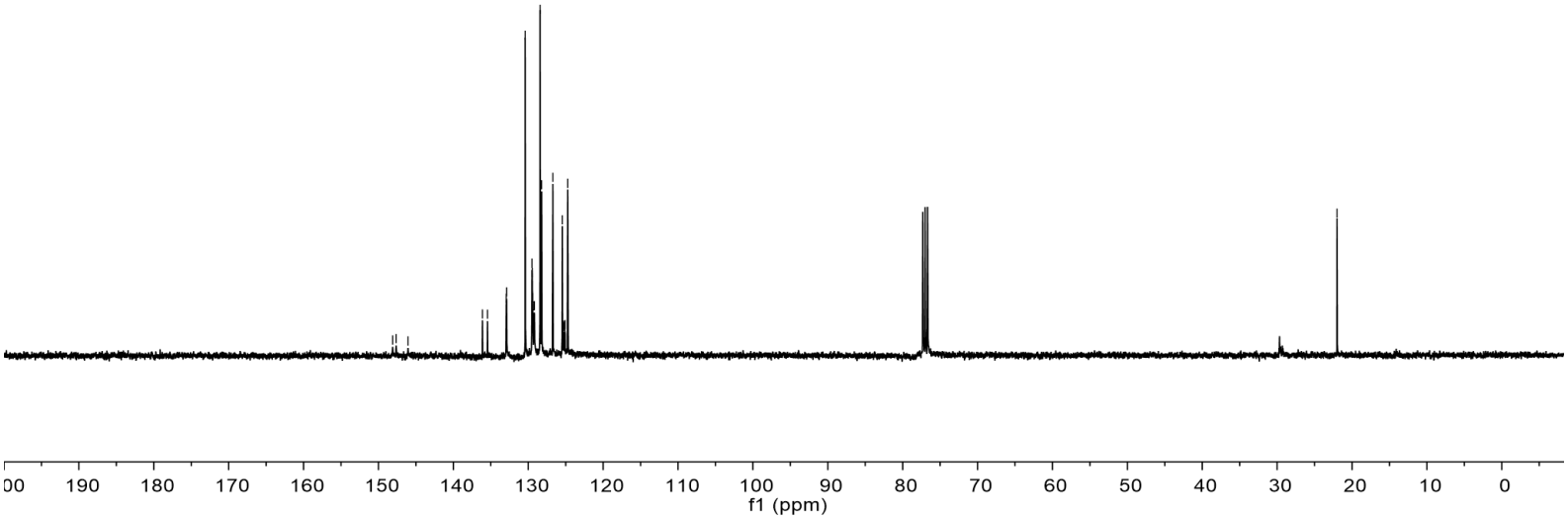
HJW2020-139-NMR-HC.2021010707.fid — 13C HJW2020-139-purified

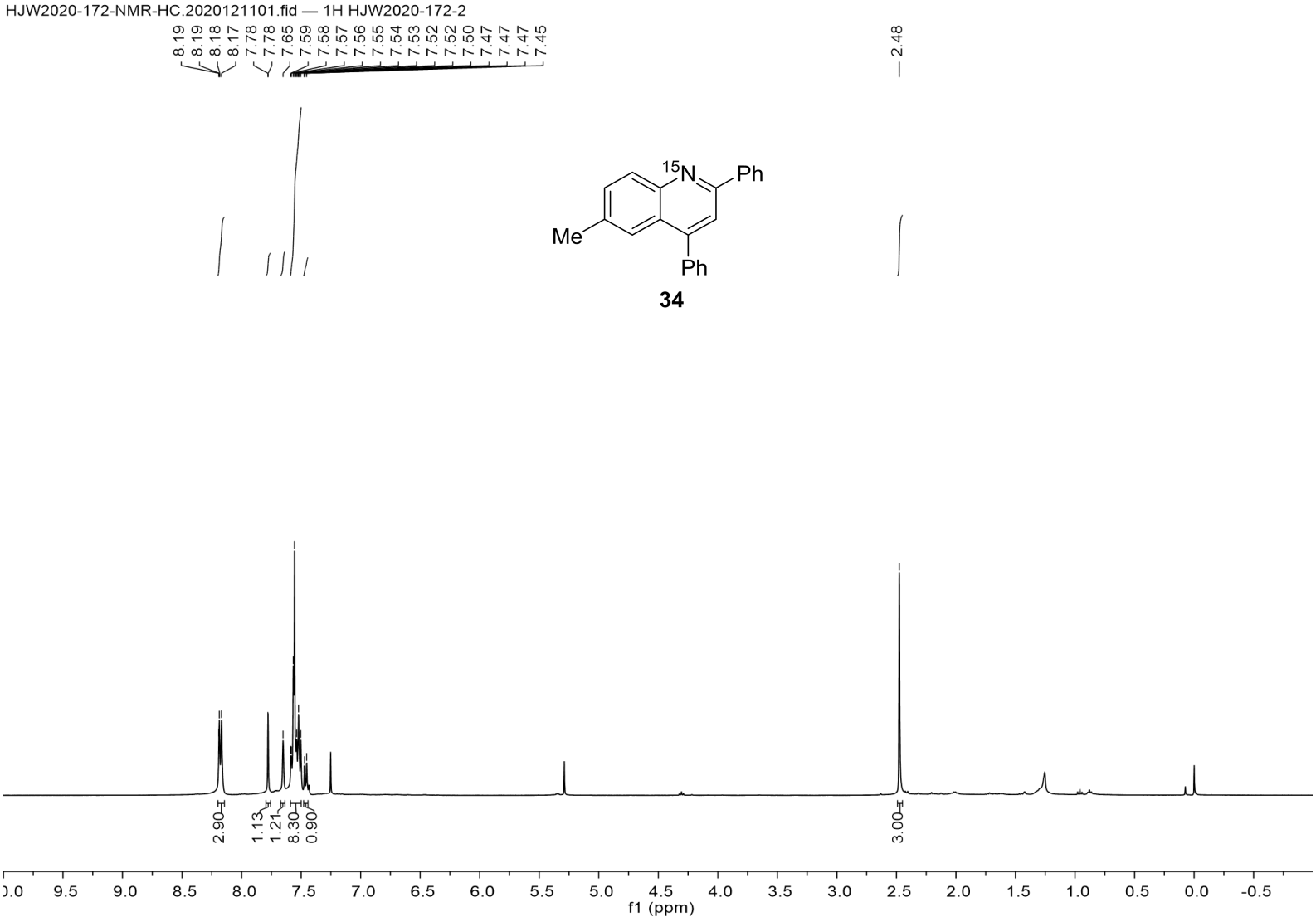
148.11
147.65
146.05
136.12
135.45
132.93
132.89
130.41
129.51
129.47
129.41
129.27
129.18
128.42
128.22
126.72
125.46
125.27
125.25
125.11
125.09
124.73

22.01



33

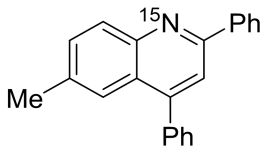



HJW2020-172-NMR-HC.2020121101.fid — 1H HJW2020-172-2
8.19
8.19
8.18
8.17
7.78
7.78
7.65
7.59
7.58
7.57
7.56
7.55
7.54
7.53
7.52
7.52
7.50
7.47
7.47
7.47
7.45
2.48
15N
Ph
Me
Ph
34
2.90
1.13
1.21
8.30
0.90
3.00
f1 (ppm)

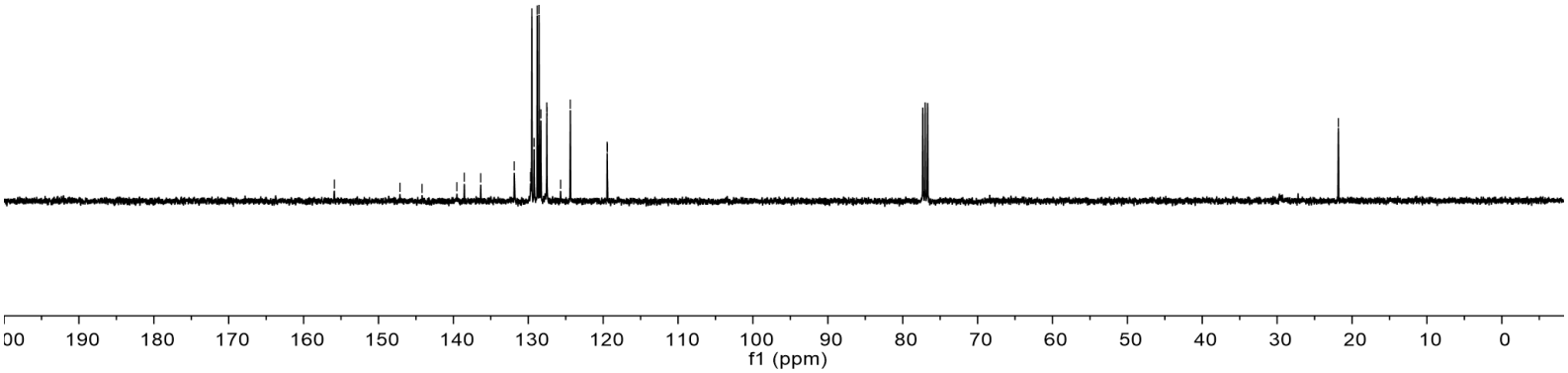
HJW2020-172-NMR-HC.2020121103.fid — 13C HJW2020-172-2

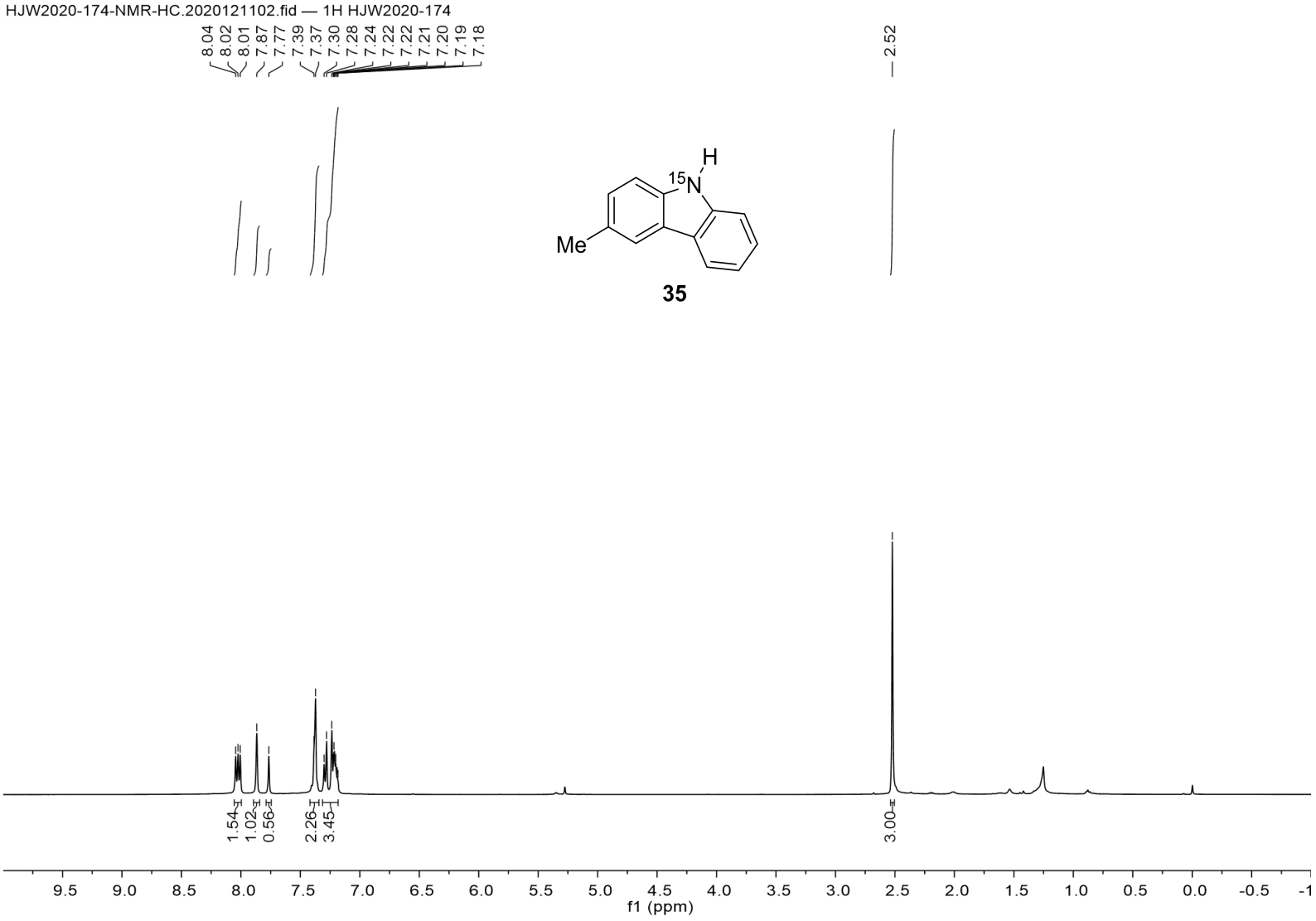
155.89
147.15
144.19
139.54
138.54
136.35
131.88
129.70
129.61
129.53
129.22
128.81
128.58
128.32
127.53
127.51
125.68
124.38
119.47
119.44

21.83



34

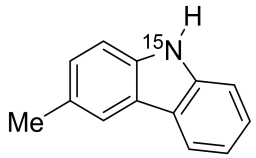



HJW2020-174-NMR-HC.2020121102.fid — 1H HJW2020-174
8.04
8.02
8.01
7.87
7.77
7.39
7.37
7.30
7.28
7.24
7.22
7.22
7.21
7.20
7.19
7.18
2.52
H
15N
Me
35
1.54
1.02
0.56
2.26
3.45
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

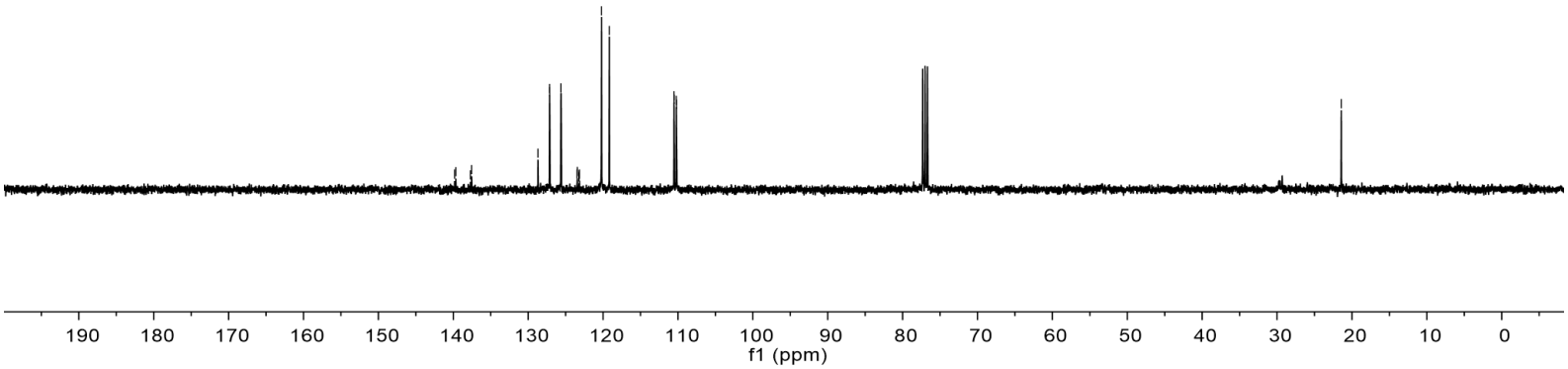
HJW2020-174-NMR-HC.2020121104.fid — 13C HJW2020-174

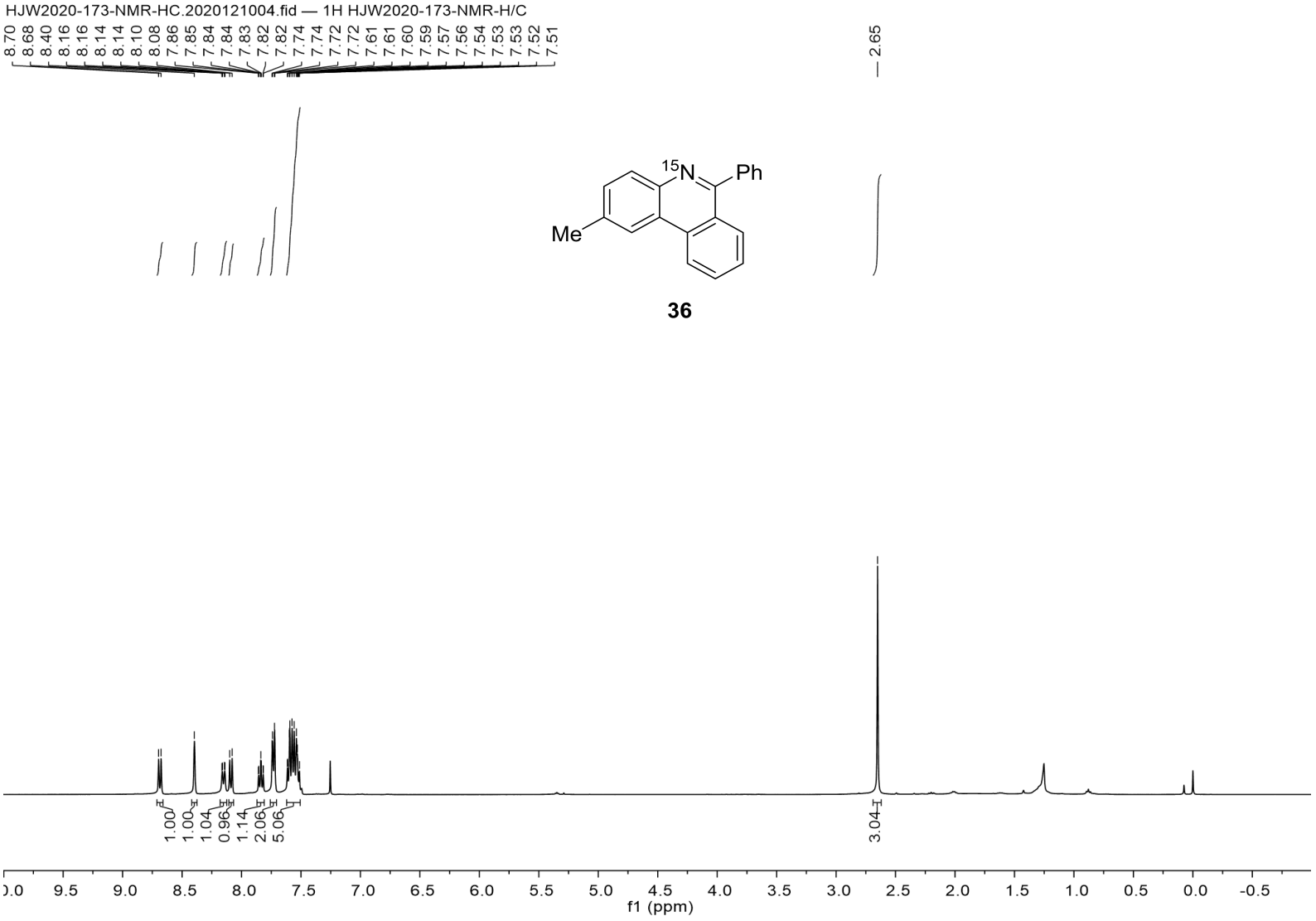
139.82
139.67
137.71
137.56
128.70
127.15
127.13
125.62
123.47
123.44
123.22
123.19
123.15
120.21
119.17
110.53
110.51
110.22
110.20

— 21.42



35




HJW2020-173-NMR-HC.2020121004.fid — 1H HJW2020-173-NMR-H/C
8.70
8.68
8.40
8.16
8.16
8.14
8.14
8.10
8.08
7.86
7.85
7.84
7.84
7.83
7.82
7.82
7.74
7.74
7.72
7.72
7.61
7.61
7.60
7.59
7.57
7.56
7.54
7.53
7.53
7.52
7.51
2.65
15N
Ph
Me
1.00
1.00
1.04
0.96
1.14
2.06
5.06
3.04
f1 (ppm)

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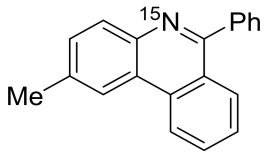
HJW2020-173-NMR-HC.2020121005.fid — 13C HJW2020-173-NMR-H/C

160.25
160.20
142.00
141.93
136.84
133.18
130.59
130.56
130.36
130.00
129.91
129.75
129.73
128.84
128.61
128.38
126.95
125.25
125.23
123.55
123.53
122.13
121.75
121.69
121.52

22.02



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