Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

for

A Facile Synthesis of Diverse 5-Arylated Triazoles via Cu-Catalyzed Oxidative Interrupted Click Reaction with Arylboronic Acids under Air Conditions

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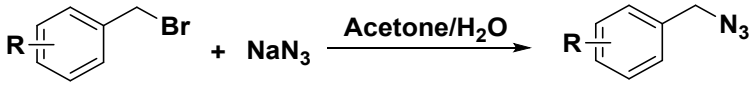
1. General Considerations

All chemicals were purchased from Adamas Reagent, energy chemical company, J&K
Scientific Ltd, Bide Pharmatech Ltd and Tansoole. The solvents were purchased from commercial suppliers and used without further purification. Unless stated otherwise, reactions were conducted in an over-dried test tube with volume of 20 ml under air atmosphere at room temperature. Azides were prepared following the known procedures. Flash column chromatography was performed over silica gel (200-300 mesh). ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker Avance 500 spectrometer (500 MHz ^{1}H, 125 MHz ^{13}C) at room temperature. Chemical shifts were reported in ppm on the scale relative to $CDCl_3$ (δ = 7.26 for ^{1}H-NMR , δ = 77.00 for ^{13}C-NMR) as an internal reference. Coupling constants (J) were reported in Hertz (Hz). The following abbreviations are used to indicate signal multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br = broad. High resolution mass spectra were recorded using a Thermo Fisher Scientific LTQ FT Ultra or Waters Micromass GCT Premier instrument.

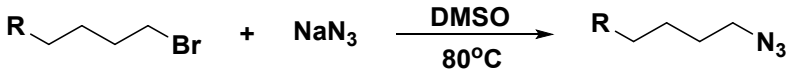
2. Experimental Procedures

Typical procedure for the synthesis of benzyl azides



In a 100 mL round-bottom flask, benzyl bromide (0.297 mL, 2.5 mmol) was dissolved in water (10 mL) and acetone (40 mL). Sodium azide (0.244 g, 3.75 mmol) was added in one portion, and the solution stirred overnight. Dichloromethane (50 mL) was added, and the organic layer separated. The aqueous layer was washed with dichloromethane (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and filtered. Solvent was removed under reduced pressure, the crude product was purified with flash chromatography (silica gel, pure petroleum ether). Spectroscopic data was consistent with previously reported results.[1]

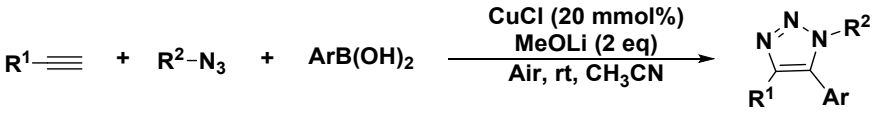
Typical procedure for the synthesis of other azide



In a 125 mL round-bottom flask, NaN_3 (0.715 g, 11 mmol) was dissolved in DMSO (20 mL) and stirred until homogeneous. Bromobutane1.072 mL, 10 mmol) was added, and the solution was stirred at 80°C overnight. Water (80 mL) was added, and the aqueous layer was extracted with ether (5 × 20 mL). The organic layers were combined, dried over anhydrous Na_2SO_4, and filtered. Solvent was removed under reduced pressure, the crude product was purified with flash chromatography (silica gel, pure petroleum ether).

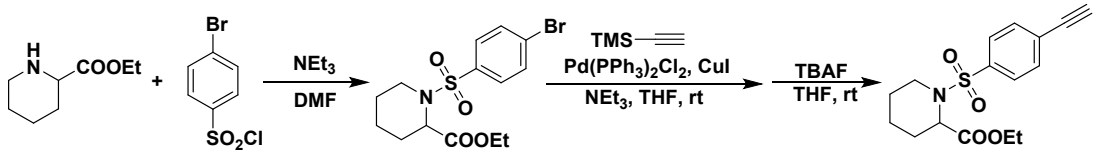
Note: Azides are explosive substances that must be carefully handled and stored.

General procedure for the copper(I)-catalyzed three-component coupling of arylboronic acid, alkynes and azides



General reaction: terminal alkynes (0.2 mmol), arylboronic acid (0.4 mmol), azides (0.3 mmol), CuCl (0.04 mmol), and MeOLi (0.4 mmol) were added in MeCN (2.5 mL) and stirred in a glass test tube for 12 h at room temperature under air. The reaction was monitored by TLC analysis. After the reaction was completed, the resulting mixture was evaporated to dryness and the crude product was purified with flash chromatography (silica gel, ethyl acetate: petroleum ether: =1:5 ~ 1:30).

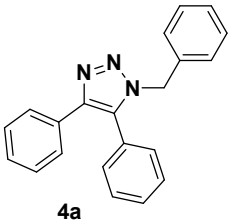
Synthesis of terminal arylalkyne 1af



Ethyl-1-((4-bromophenyl)sulfonyl)piperidine-2-carboxylate was prepared according to the literature procedure.[2] Ethyl piperidine-2-carboxylate hydrochloride (0.79 g, 5 mmol) and 4-bromobenzenesulfonyl chloride (1.28 g, 5 mmol) were dissolved in 50 mL DMF. Triethylamine (3 equiv) was then added dropwise. After stirring for 8 h at room temperature, the residue was suspended in 50 mL of ethyl acetate and washed with 2 M HCl (25 mL×3) and H_2O (25 mL). The organic layer was dried over anhydrous Na_2SO_4, and concentrated in vacuo. The crude reaction mixture was purified on silica gel. An oven-dried schlenk tube was charged with bromo-substrate (1 mmol), $Pd(PPh_3)_2Cl_2$ (14mg, 0.02 mmol) and CuI (7.6 mg, 0.04 mmol). The tube was taken to the globe box and dry THF (1 ml) was added to it. The tube was capped and taken out from the glove box. Triethylamine (216 uL, 1.55 mmol) was injected in it and trimethylsilylacetylene (176 uL, 1.25 mmol) was added to the reaction mixture slowly. The reaction mixture was stirred at room temperature for 24 hours. Upon completion, the reaction mixture was diluted with ethyl acetate, filtered through a silica gel plug, rinsed with ethyl acetate, and concentrated in vacuo. The crude reaction mixture was purified on silica gel. The internal alkyne was dissolved with anhydrous THF (1 mL), and TBAF (1.5 mL, 1M in THF) was added. The resulting mixture was stirred at room temperature overnight. Upon completion, the reaction mixture was diluted with ethyl acetate, filtered through a silica gel plug, rinsed with ethyl acetate, and concentrated in vacuo. The crude reaction mixture was purified on silica gel to give the desired product as a brown-yellow oil.

3. Characterization of Products

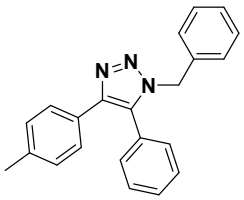
1-benzyl-4,5-diphenyl-1*H*-1,2,3-triazole (4a)



4a

white solid, 56.6 mg, 91% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 – 7.54 (m, 2H), 7.53 – 7.47 (m, 1H), 7.47 – 7.41 (m, 2H), 7.34 – 7.23 (m, 6H), 7.22 – 7.14 (m, 2H), 7.10 – 6.94 (m, 2H), 5.44 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.5, 135.4, 133.9, 130.9, 130.1, 129.7, 129.2, 128.7, 128.5, 128.2, 127.9, 127.7, 127.5, 126.7, 52.1. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{18}N_3^+$, 312.1495; Found: 312.1499.

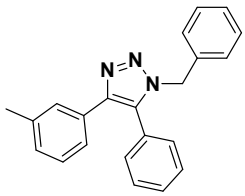
1-benzyl-5-phenyl-4-(p-tolyl)-1*H*-1,2,3-triazole (4b)



4b

shallow yellow solid, 45.5 mg, 70% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 – 7.46 (m, 3H), 7.46 – 7.41 (m, 2H), 7.29 – 7.25 (m, 3H), 7.19 – 7.14 (m, 2H), 7.09 (m, *J* = 8.0 Hz, 2H), 7.07 – 7.04 (m, 2H), 5.43 (s, 2H), 2.32 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 137.5, 135.4, 133.6, 130.1, 129.6, 129.2, 129.1, 128.7, 128.1, 128.1, 128.0, 127.5, 126.7, 52.0, 21.2. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{20}N_3^+$, 326.1652; Found: 326.1655.

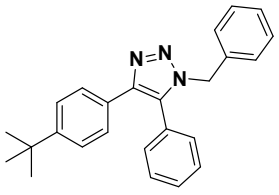
1-benzyl-5-phenyl-4-(m-tolyl)-1*H*-1,2,3-triazole (4c)



4c

shallow yellow liquid, 59.1 mg, 91% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (s, 1H), 7.53 – 7.47 (m, 1H), 7.46 – 7.38 (m, 2H), 7.32 – 7.20 (m, 4H), 7.20 – 7.15 (m, 2H), 7.15 – 7.11 (m, 1H), 7.09 – 7.01 (m, 3H), 5.44 (s, 2H), 2.30 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.7, 138.1, 135.4, 133.9, 130.8, 130.1, 129.7, 129.1, 128.7, 128.5, 128.3, 128.1, 127.9, 127.6, 127.5, 123.8, 52.1, 21.4. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{20}N_3^+$, 326.1652; Found: 326.1651.

1-benzyl-4-(4-(tert-butyl)phenyl)-5-phenyl-1*H*-1,2,3-triazole (4d)

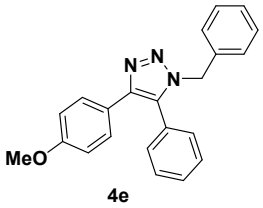


4d

white solid, 69.7 mg, 95% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 – 7.53 (m, 2H), 7.52 – 7.48 (m, 1H), 7.46 – 7.42 (m, 2H), 7.32 – 7.29 (m, 2H), 7.28 – 7.25 (m, 3H), 7.19 – 7.16 (m, 2H), 7.05 – 7.02 (m, 2H), 5.42 (s, 2H), 1.30 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.7, 144.5, 135.5, 133.6, 130.2, 129.6, 129.1, 128.7, 128.1, 128.1, 128.0, 127.5, 126.2, 125.4, 52.0, 34.6, 31.3.

HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{25}H_{25}N_4^+$, 368.2121; Found: 368.2124.

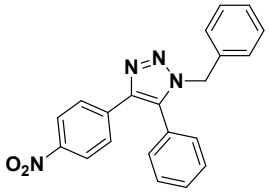
1-benzyl-4-(4-methoxyphenyl)-5-phenyl-1*H*-1,2,3-triazole (4e)



4e

white solid, 48.4 mg, 71% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 – 7.46 (m, 3H), 7.46 – 7.40 (m, 2H), 7.29 – 7.25 (m, 3H), 7.18 – 7.14 (m, 2H), 7.07 – 7.02 (m, 2H), 6.85 – 6.78 (m, 2H), 5.42 (s, 2H), 3.78 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.2, 144.4, 135.5, 133.1, 130.2, 129.6, 129.2, 128.8, 128.7, 128.1, 128.0, 127.5, 123.6, 113.9, 55.2, 52.0. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{20}N_3O^+$, 342.1601; Found: 342.1604.

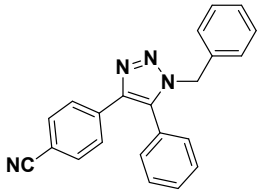
1-benzyl-4-(4-nitrophenyl)-5-phenyl-1*H*-1,2,3-triazole (4f)



4f

yellow solid, 42.8 mg, 60% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14 – 8.10 (m, 2H), 7.76 – 7.72 (m, 2H), 7.59 – 7.54 (m, 1H), 7.52 – 7.47 (m, 2H), 7.30 – 7.26 (m, 3H), 7.19 – 7.15 (m, 2H), 7.07 – 7.03 (m, 2H), 5.44 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.9, 142.4, 137.4, 135.5, 134.9, 130.4, 129.9, 129.6, 128.8, 128.4, 127.6, 127.0, 126.9, 123.9, 52.3. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{16}N_4O_2^+$, 357.1346; Found: 357.1348.

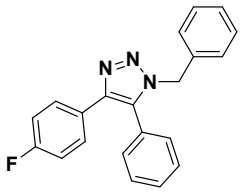
4-(1-benzyl-5-phenyl-1*H*-1,2,3-triazol-4-yl)benzonitrile (4g)



4g

white solid, 50.5 mg, 75% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 – 7.66 (m, 2H), 7.58 – 7.52 (m, 3H), 7.51 – 7.46 (m, 2H), 7.31 – 7.26 (m, 3H), 7.18 – 7.13 (m, 2H), 7.07 – 7.01 (m, 2H), 5.43 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.7, 135.5, 135.2, 134.9, 132.3, 130.3, 129.9, 129.5, 128.8, 128.4, 127.6, 127.1, 126.8, 118.8, 111.0, 52.2. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{17}N_4^+$, 337.1448; Found: 337.1447.

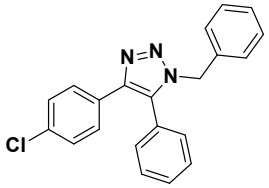
1-benzyl-4-(4-fluorophenyl)-5-phenyl-1*H*-1,2,3-triazole (4h)



4h

shallow yellow solid, 52.6 mg, 80% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 – 7.48 (m, 3H), 7.47 – 7.42 (m, 2H), 7.29 – 7.25 (m, 3H), 7.18 – 7.14 (m, 2H), 7.07 – 7.02 (m, 2H), 6.99 – 6.93 (m, 2H), 5.43 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.5 (d, *J* =b271.3 Hz), 143.8, 135.3, 133.7, 130.1, 129.8, 129.3, 128.7, 128.5 (d, *J* = 8.8 Hz), 128.2, 127.7, 127.5, 127.1 (d, *J* = 3.8 Hz), 115.4 (d, *J* = 22.5 Hz), 52.1. Known compound.[1] HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{17}FN_3^+$, 330.1401; Found: 330.1404.

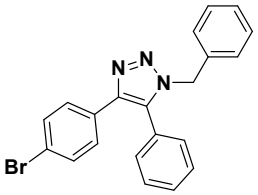
1-benzyl-4-(4-chlorophenyl)-5-phenyl-1*H*-1,2,3-triazole (4i)



4i

yellow solid, 48.3 mg, 70% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 – 7.48 (m, 3H), 7.47 – 7.43 (m, 2H), 7.30 – 7.25 (m, 3H), 7.26 – 7.22 (m, 2H), 7.17 – 7.13 (m, 2H), 7.07 – 7.02 (m, 2H), 5.43 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.6, 135.2, 134.0, 133.6, 130.0, 129.9, 129.5, 129.3, 128.7, 128.7, 128.2, 127.9, 127.5, 52.1. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{17}{}^{35}ClN_3^+$, 346.1106; Found: 346.1108.

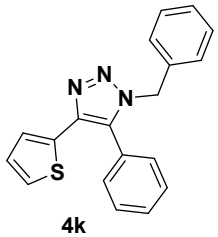
1-benzyl-4-(4-bromophenyl)-5-phenyl-1*H*-1,2,3-triazole (4j)



4j

white solid, 49.0 mg, 63% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 – 7.48 (m, 1H), 7.49 – 7.42 (m, 4H), 7.41 – 7.38 (m, 2H), 7.27 (m, 3H), 7.17 – 7.12 (m, 2H), 7.04 (m, 2H), 5.42 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.6, 135.2, 134.1, 131.6, 130.0, 129.9, 129.3, 128.7, 128.2, 128.2, 127.5, 127.5, 121.8, 52.1. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{17}{}^{79}BrN_3^+$, 390.0600; Found: 390.0604.

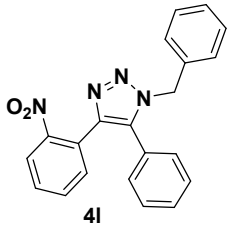
1-benzyl-5-phenyl-4-(thiophen-2-yl)-1H-1,2,3-triazole (4k)



4k

shallow grey solid, 35.5 mg, 56% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 – 7.50 (m, 1H), 7.49 – 7.44 (m, 2H), 7.30 – 7.25 (m, 3H), 7.22 – 7.19 (m, 3H), 7.06 – 7.01 (m, 3H), 6.94 – 6.90 (m, 1H), 5.41 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.6, 135.2, 133.2, 132.9, 130.2, 130.0, 129.2, 128.7, 128.2, 127.5, 127.2, 127.1, 124.9, 124.2, 52.2. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{19}H_{16}N_3S^+$, 318.1059; Found: 318.1068.

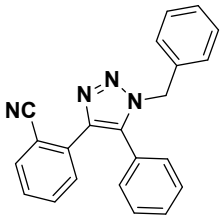
1-benzyl-4-(2-nitrophenyl)-5-phenyl-1*H*-1,2,3-triazole (4l)



4l

brown yellow solid, 46.3 mg, 65% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.87 – 7.82 (m, 1H), 7.60 – 7.55 (m, 1H), 7.55 – 7.51 (m, 1H), 7.50 – 7.45 (m, 1H), 7.44 – 7.39 (m, 1H), 7.37 – 7.29 (m, 5H), 7.14 – 7.04 (m, 4H), 5.54 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.1, 141.6, 135.7, 135.3, 132.6, 132.5, 129.7, 129.6, 129.2, 129.1, 128.9, 128.2, 127.3, 126.1, 125.8, 124.5, 52.2. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{17}N_4O_2^+$, 357.1346; Found: 357.1350.

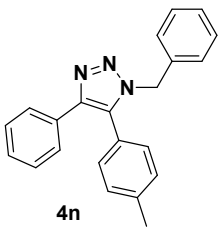
2-(1-benzyl-5-phenyl-1*H*-1,2,3-triazol-4-yl)benzonitrile (4m)



4m

white solid, 51.0 mg, 76% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 – 7.63 (m, 1H), 7.58 – 7.52 (m, 1H), 7.51 – 7.48 (m, 1H), 7.45 – 7.36 (m, 4H), 7.34 – 7.26 (m, 3H), 7.18 – 7.08 (m, 4H), 5.55 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.3, 136.2, 135.1, 134.4, 133.7, 132.6, 130.7, 129.9, 129.8, 129.1, 128.9, 128.5, 128.3, 127.4, 126.5, 117.9, 112.4, 52.3. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{17}N_4^+$, 337.1448; Found: 337.1449.

1-benzyl-4-phenyl-5-(p-tolyl)-1*H*-1,2,3-triazole (4n)

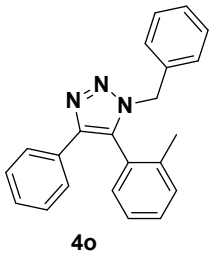


4n

shallow yeollow solid, 59.8 mg, 92% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.73 – 7.52 (m, 2H), 7.38 – 7.18 (m, 8H), 7.17 – 6.93 (m, 4H), 5.42 (s, 2H), 2.45 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.4, 139.8, 135.6, 134.0, 131.1, 129.9, 129.9, 128.7 128.4, 128.1, 127.6, 127.5, 126.7, 124.7, 51.9, 21.5. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{20}N_3^+$, 326.1652; Found:

326.1656.

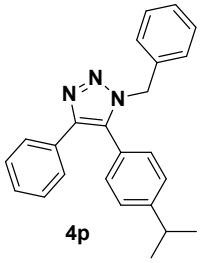
1-benzyl-4-phenyl-5-(o-tolyl)-1*H*-1,2,3-triazole (4o)



4o

yellow solid, 58.5 mg, 90% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 – 7.54 (m, 2H), 7.46 – 7.41 (m, 1H), 7.32 (s, 1H), 7.28 – 7.20 (m, 7H), 7.14 – 7.10 (m, 1H), 6.99 – 6.95 (m, 2H), 5.41 – 5.27 (m, 2H), 1.64 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.4, 138.5, 134.7, 132.9, 131.1, 130.8, 130.3, 130.1, 128.6, 128.5, 128.2, 128.1, 127.7, 127.4, 126.5, 125.7, 52.3, 19.1. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{20}N_3^+$, 326.1652; Found: 326.1654.

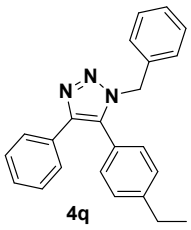
1-benzyl-5-(4-isopropylphenyl)-4-phenyl-1*H*-1,2,3-triazole (4p)



4p

white solid, 42.4 mg, 60% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 – 7.59 (m, 2H), 7.31 – 7.26 (m, 8H), 7.10 – 7.07 (m, 2H), 7.07 – 7.03 (m, 2H), 5.43 (s, 2H), 1.32 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.7, 144.3, 135.4, 134.1, 130.9, 130.0, 128.7, 128.4, 128.1, 127.7, 127.5, 127.2, 126.8, 124.9, 52.0, 34.0, 23.9. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{24}H_{24}N_4^+$, 354.1965; Found: 354.1967.

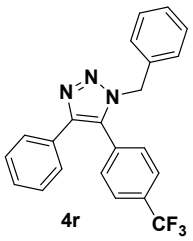
1-benzyl-5-(4-ethylphenyl)-4-phenyl-1*H*-1,2,3-triazole (4q)



4q

white solid, 40.7 mg, 60% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 – 7.58 (m, 2H), 7.32 – 7.24 (m, 8H), 7.11 – 7.05 (m, 4H), 5.42 (s, 2H), 2.75 (q, *J* = 7.6 Hz, 2H), 1.32 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.0, 144.4, 135.5, 134.1, 131.1, 130.0, 128.7, 128.7, 128.4, 128.1, 127.6, 127.5, 126.7, 124.9, 51.9, 28.7, 15.3. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{23}H_{22}N_4^+$, 340.1808; Found: 340.1812.

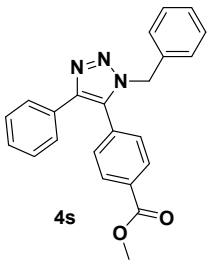
1-benzyl-4-phenyl-5-(4-(trifluoromethyl)phenyl)-1*H*-1,2,3-triazole (4x)



4r

white solid, 47.0 mg, 62% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 – 7.66 (m, 2H), 7.56 – 7.50 (m, 2H), 7.33 – 7.25 (m, 8H), 7.08 – 6.98 (m, 2H), 5.45 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.1, 135.0, 132.4, 131.8 (d, *J* = 32.5 Hz), 131.8, 130.6, 130.3, 128.9, 128.6, 128.4, 128.1, 127.3, 126.9, 126.1 (q, *J* = 3.8 Hz), 123.7 (d, *J* = 270.0 Hz), 52.4. Known compound.[3]
HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{17}F_3N_3^+$ 380.1369; Found: 380.1367.

methyl 4-(1-benzyl-4-phenyl-1*H*-1,2,3-triazol-5-yl)benzoate (4s)

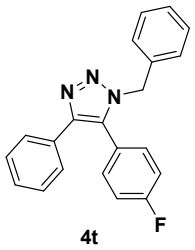


4s

shallow yellow solid, 45.0 mg, 61% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.04 – 7.98 (m, 2H), 7.55 – 7.50 (m, 2H), 7.30 – 7.27 (m, 8H), 7.07 – 7.02 (m, 2H), 5.46 (s, 2H), 2.67 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 197.3, 145.1, 137.8, 135.0, 132.9, 132.6, 130.4, 129.0, 128.8, 128.6, 128.4, 128.1, 127.4, 126.9, 115.5, 52.3, 26.7. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{23}H_{20}N_3O_2^+$,

370.1550; Found: 370.1553.

1-benzyl-5-(4-fluorophenyl)-4-phenyl-1*H*-1,2,3-triazole (4t)

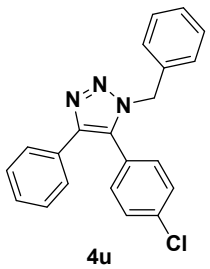


4t

yellow solid, 57.9 mg, 88% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 – 7.54 (m, 2H), 7.31 – 7.26 (m, 6H), 7.15 – 7.11 (m, 4H), 7.06 – 7.02 (m, 2H), 5.43 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.4 (d, J = 248.8 Hz), 144.8, 135.2, 132.1 (d, J = 8.8 Hz), 130.7, 128.8, 128.5, 128.3, 127.9, 127.4, 126.7, 125.8, 123.8 (d, J = 3.8 Hz), 116.5 (d, J = 21.3 Hz), 52.2. ^{19}F NMR (470 MHz, $CDCl_3$) δ -110.4 (d, J = 2.7 Hz).

HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{17}FN_3^+$, 330.1401; Found: 330.1400.

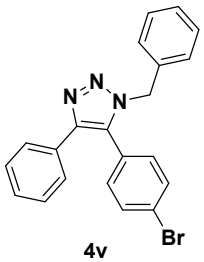
1-benzyl-5-(4-chlorophenyl)-4-phenyl-1*H*-1,2,3-triazole (4u)



4u

yellow solid, 58.7 mg, 85% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 – 7.52 (m, 2H), 7.44 – 7.40 (m, 2H), 7.32 – 7.27 (m, 6H), 7.12 – 7.07 (m, 2H), 7.07 – 7.03 (m, 2H), 5.43 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.9, 136.0, 135.2, 131.5, 130.6, 129.6, 128.8, 128.6, 128.3, 128.0, 127.4, 126.8, 126.3, 117.0, 52.2. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{17}{}^{35}ClN_3^+$, 346.1106; Found: 341.1111.

1-benzyl-5-(4-bromophenyl)-4-phenyl-1*H*-1,2,3-triazole (4v)

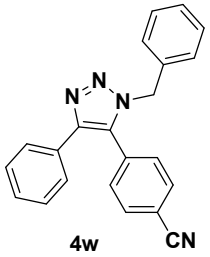


4v

yellow liquid, 58.3 mg, 75% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 – 7.51 (m, 4H), 7.33 – 7.27 (m, 6H), 7.09 – 7.04 (m, 2H), 7.04 – 6.99 (m, 2H), 5.43 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.8, 135.2, 132.7, 132.5, 131.7, 130.6, 128.8, 128.6, 128.3, 128.0, 127.4, 126.8, 124.3, 117.5, 52.2.

HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{17}{}^{79}BrN_3^+$, 390.0600; Found: 390.0601.

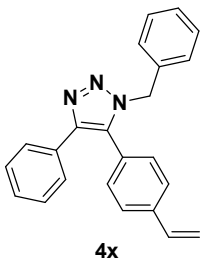
4-(1-benzyl-4-phenyl-1*H*-1,2,3-triazol-5-yl)benzonitrile (4w)



4w

white solid, 60.5 mg, 90% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 – 7.68 (m, 2H), 7.50 – 7.48 (m, 1H), 7.45 – 7.42 (m, 2H), 7.29 – 7.27 (m, 6H), 7.01 – 6.98 (m, 3H), 5.46 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 145.4, 134.1, 132.9, 132.4, 132.3, 130.9, 129.6, 129.0, 128.8, 128.7, 128.6, 127.3, 127.1, 117.9, 113.8, 52.7. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{17}N_4^+$, 337.1448; Found: 337.1449.

1-benzyl-4-phenyl-5-(4-vinylphenyl)-1*H*-1,2,3-triazole (4r)

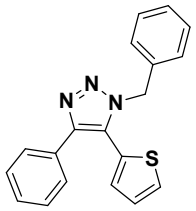


4x

shallow yellow solid, 44.5 mg, 66% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 – 7.58 (m, 2H), 7.50 – 7.46 (m, 2H), 7.31 – 7.26 (m, 6H), 7.16 – 7.12 (m, 2H), 7.11 – 7.06 (m, 2H), 6.84 – 6.73 (m, 1H), 5.87 (d, J = 17.6 Hz, 1H), 5.44 (s, 2H), 5.40 (d, J = 11.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 138.8, 135.9, 135.4, 133.7, 130.9, 130.3, 128.7, 128.5, 128.2, 127.8, 127.5, 127.0,

126.9, 126.8, 115.7, 52.0. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{23}H_{20}N_4^+$, 338.1652; Found: 338.1657.

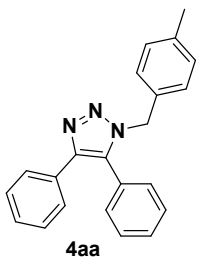
1-benzyl-4-phenyl-5-(thiophen-2-yl)-1*H*-1,2,3-triazole (4y)



4y

white solid, 33.6 mg, 53% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 – 7.62 (m, 2H), 7.57 – 7.53 (m, 1H), 7.35 – 7.29 (m, 6H), 7.17 – 7.13 (m, 1H), 7.13 – 7.09 (m, 2H), 7.04 – 6.86 (m, 1H), 5.51 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.1, 135.3, 130.7, 130.5, 129.4, 128.8, 128.5, 128.2, 128.1, 127.9, 127.4, 127.1, 126.9, 126.7, 52.2. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{19}H_{16}N_3S^+$, 318.1059; Found: 318.1065.

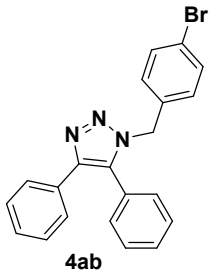
1-(4-methylbenzyl)-4,5-diphenyl-1*H*-1,2,3-triazole (4aa)



4aa

white solid, 52.1 mg, 80% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 – 7.55 (m, 2H), 7.54 – 7.48 (m, 1H), 7.47 – 7.43 (m, 2H), 7.31 – 7.25 (m, 3H), 7.21 – 7.16 (m, 2H), 7.11 – 7.05 (m, 2H), 6.97 – 6.92 (m, 2H), 5.39 (s, 2H), 2.33 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.5, 137.9, 133.8, 132.4, 131.0, 130.2, 129.6, 129.4, 129.2, 128.4, 128.0, 127.7, 127.5, 126.7, 51.8, 21.1.
HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{20}N_3^+$, 326.1652; Found: 326.1656.

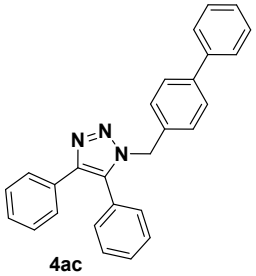
1-(4-bromobenzyl)-4,5-diphenyl-1*H*-1,2,3-triazole (4ab)



4ab

white solid, 66.1 mg, 85% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 – 7.55 (m, 2H), 7.54 – 7.49 (m, 1H), 7.48 – 7.44 (m, 2H), 7.42 – 7.38 (m, 2H), 7.33 – 7.23 (m, 3H), 7.21 – 7.13 (m, 2H), 7.00 – 6.83 (m, 2H), 5.38 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 134.3, 133.8, 131.9, 130.7, 130.0, 129.8, 129.3, 129.3, 128.5, 127.8, 127.7, 126.7, 122.3, 51.4.
HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{21}H_{17}{}^{79}BrN_3^+$, 390.0600; Found: 390.0607.

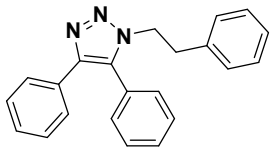
1-([1,1'-biphenyl]-4-ylmethyl)-4,5-diphenyl-1*H*-1,2,3-triazole (4ac)



4ac

white solid, 50.3 mg, 65% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 – 7.56 (m, 4H), 7.55 – 7.50 (m, 3H), 7.49 – 7.43 (m, 4H), 7.40 – 7.34 (m, 1H), 7.32 – 7.25 (m, 3H), 7.24 – 7.20 (m, 2H), 7.17 – 7.11 (m, 2H), 5.48 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 141.1, 140.4, 134.3, 133.9, 130.9, 130.2, 129.7, 129.2, 128.8, 128.5, 128.0, 127.9, 127.7, 127.5, 127.4, 127.1, 126.7, 51.8. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{27}H_{22}N_3^+$ 388.1808; Found: 388.1809.

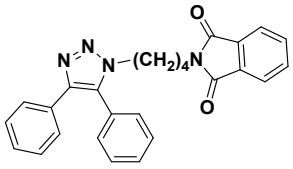
1-phenethyl-4,5-diphenyl-1*H*-1,2,3-triazole (4ad)



4ad

white solid, 39.0 mg, 60% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 – 7.53 (m, 2H), 7.52 – 7.48 (m, 1H), 7.47 – 7.42 (m, 2H), 7.30 – 7.22 (m, 6H), 7.03 – 6.99 (m, 2H), 6.98 – 6.94 (m, 2H), 4.41 (t, J = 7.4 Hz, 2H), 3.19 (t, J = 7.4 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.0, 137.2, 134.2, 131.0, 130.0, 129.6, 129.2, 128.8, 128.7, 128.4, 127.9, 127.6, 126.9, 126.7, 49.4, 36.6. HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{22}H_{20}N_3^+$, 326.1652; Found: 326.1658.

2-(4-(4,5-diphenyl-1*H*-1,2,3-triazol-1-yl)butyl)isoindoline-1,3-dione (4ae)

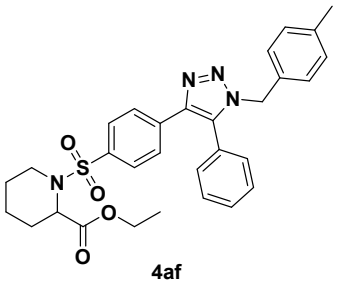


4ae

white solid, 49.8 mg, 59% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 – 7.80 (m, 2H), 7.80 – 7.67 (m, 2H), 7.59 – 7.52 (m, 2H), 7.51 – 7.46 (m, 3H), 7.36 – 7.31 (m, 2H), 7.30 – 7.23 (m, 3H), 4.28 (t, J = 7.1 Hz, 2H), 3.64 (t, J = 6.9 Hz, 2H), 1.91 – 1.78 (m, 2H), 1.75 – 1.57 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.3, 144.3, 134.0, 133.7, 132.0, 131.0, 129.9, 129.7, 129.4, 128.4, 128.0, 127.6, 126.8, 123.3, 47.6, 37.0, 27.2, 25.5.

HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{26}H_{23}N_4O_2^+$, 423.1816; Found: 423.1821.

Ethyl-1-((4-(1-(4-methylbenzyl)-5-phenyl-1*H*-1,2,3-triazol-4-yl)phenyl)sulfonyl)piperidine-2-carboxylate

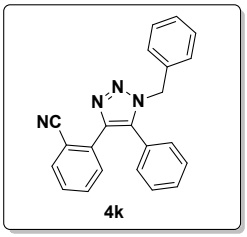


4af

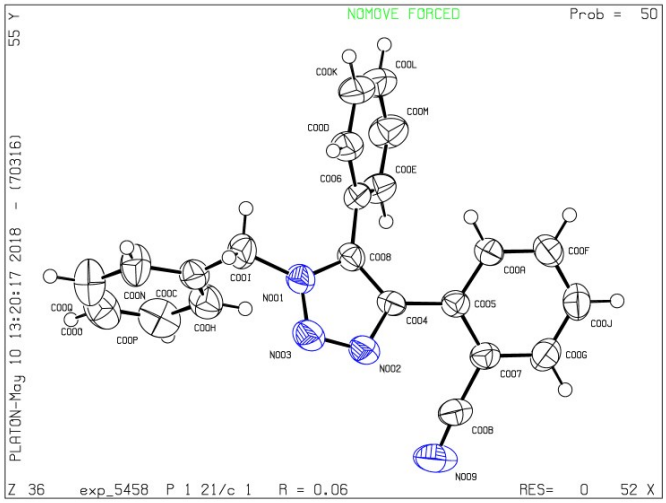
brown yellow solid, 60.0 mg, 55% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (s, 4H), 7.60 – 7.51 (m, 1H), 7.50 – 7.44 (m, 2H), 7.21 – 7.13 (m, 2H), 7.11 – 7.04 (m, 2H), 6.97 – 6.87 (m, 2H), 5.38 (s, 2H), 4.80 – 4.54 (m, 1H), 4.09 – 3.98 (m, 1H), 3.97 – 3.88 (m, 1H), 3.81 – 3.68 (m, 1H), 3.28 – 3.09 (m, 1H), 2.33 (s, 3H), 2.20 – 1.93 (m, 1H), 1.77 – 1.69 (m, 1H), 1.67 – 1.59 (m, 2H), 1.53 – 1.41 (m, 1H), 1.32 – 1.26 (m, 1H), 1.13 (t, J = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.6, 143.0, 138.8, 138.1, 135.1, 134.9, 132.0, 130.1, 130.0, 129.4, 129.4, 127.6, 127.4, 127.3, 126.7, 61.1, 55.1, 52.0, 42.6, 27.8, 24.7, 21.1, 20.0, 14.1.

HRMS (ESI) *m/z*: $[M+H]^+$ Calcd for $C_{29}H_{31}N_4O_4S^+$, 545.2217; Found: 545.2220.

4. Crystal Structure of 4k



CCDCC 1844188



Bond precision: C-C = 0.0033 A Wavelength=0.71073

Cell: a=11.8069(10) b=9.7467(7) c=15.4870(11) alpha=90 beta=102.555(7) gamma=90

Temperature: 298 K

	Calculated	Reported
Volume	1739.6(2)	1739.6(2)

Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C22 H16 N4	C22 H16 N4
Sum formula	C22 H16 N4	C22 H16 N4
Mr	336.39	336.40
Dx,g cm-3	1.284	1.284
Z	4	4
Mu (mm-1)	0.078	0.078
F000	704.0	704.2
F000'	704.23	
h,k,lmax	14,11,18	14,11,18
Nref	3065	3058
Tmin,Tmax	0.447,1.000	
Tmin'		

Correction method= # Reported T Limits: Tmin=0.447 Tmax=1.000 AbsCorr = MULTI-SCAN

Data completeness= 0.998 Theta(max)= 24.990

R(reflections)= 0.0569(2070) wR2(reflections)= 0.1572(3058)

S = 0.926 Npar= 235

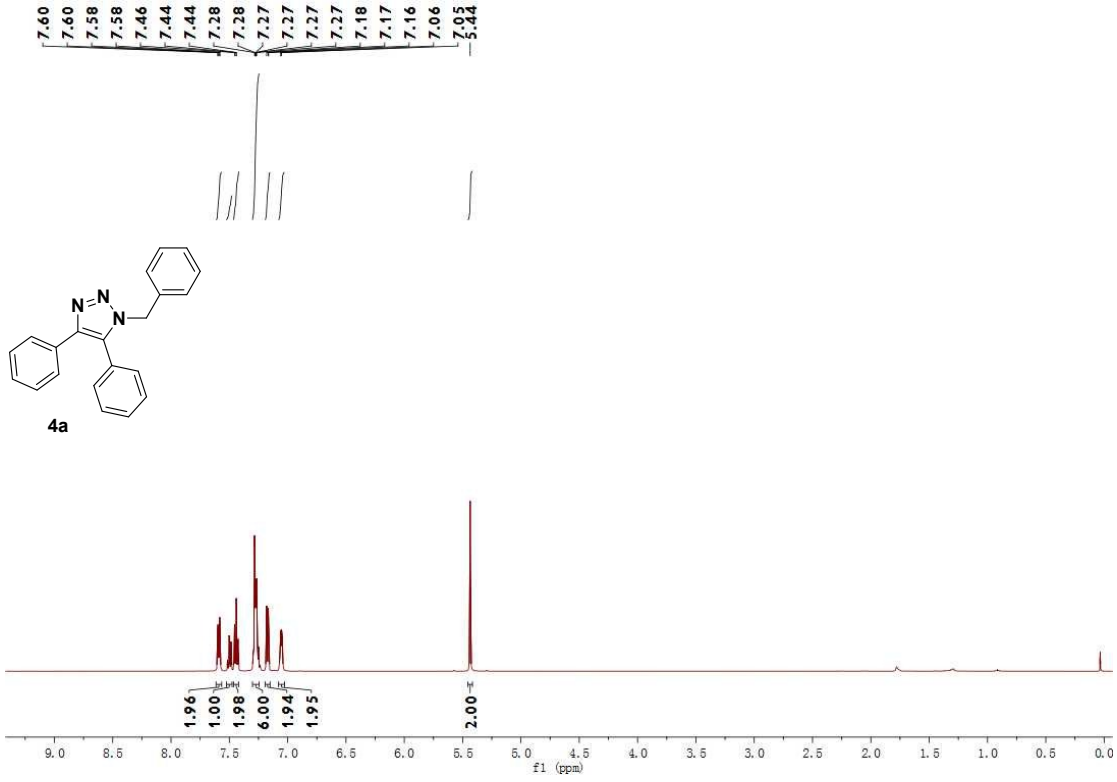
5. References

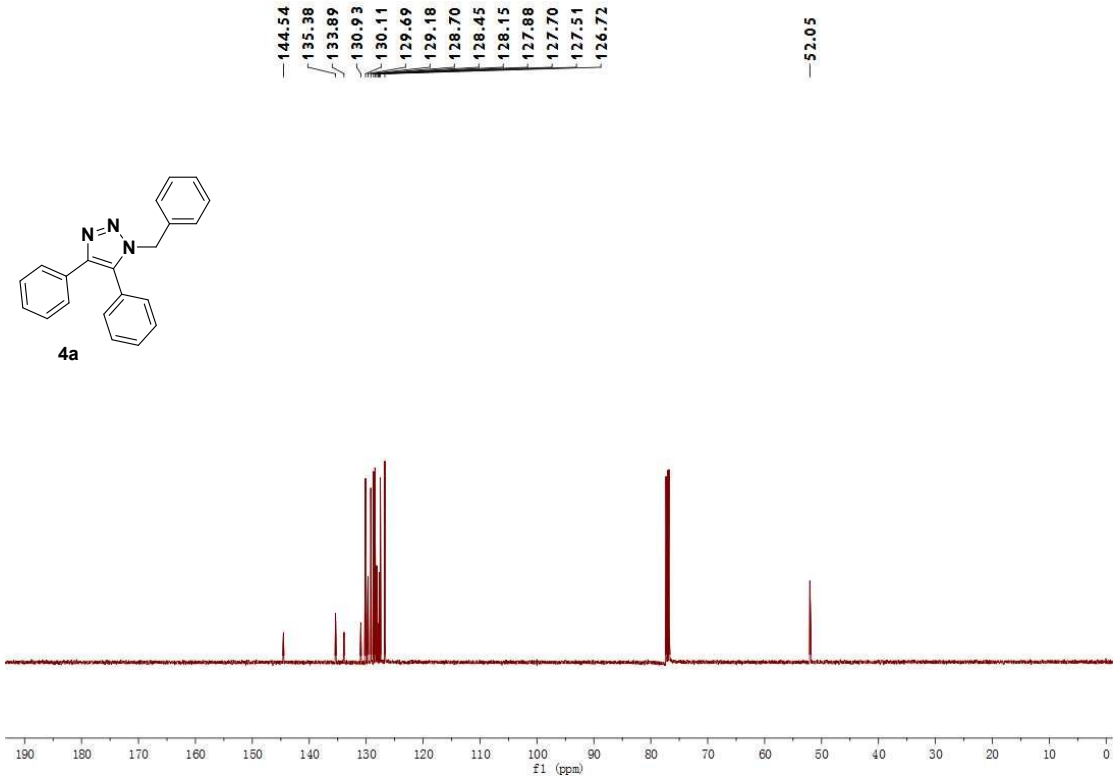
1. F. Wei, H. Li, C. Song, Y. Ma, L. Zhou, C.-H. Tung and Z. Xu, *Org. Lett.,* 2015, **17**, 2860.

2. C. Juli, M. Sippel, J. Jäger, A. Thiele, M. Weiwad, K. Schweimer, P. Rösch, M. Steinert, C. A. Sotriffer and U. Holzgrabe, *J. Med. Chem*. 2011, **54**, 277.

3. L. Ackermann, H. K. Potukuchi, D. Landsberg and R. Vicente, *Org. Lett*., 2008, **10**, 3081.

6. NMR Spectra of Products

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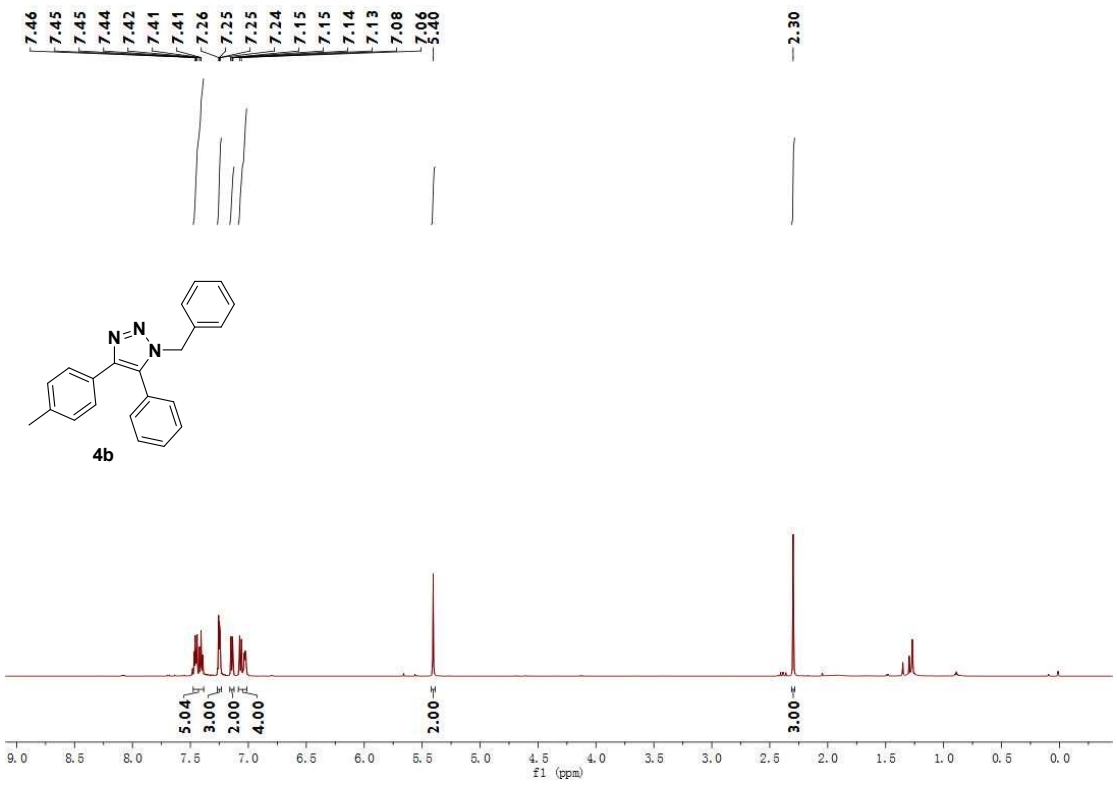
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7.13
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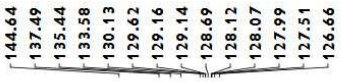
2.00
4.00
2.00
3.00
f1 (ppm)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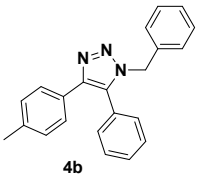

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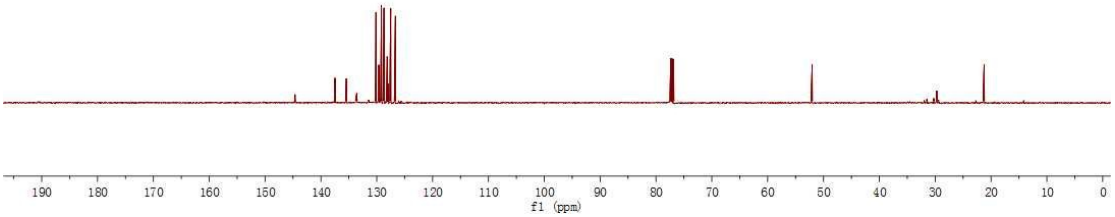
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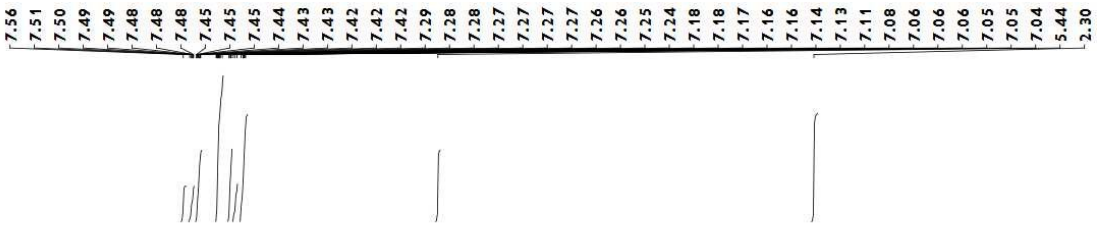
f1 (ppm)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7.43
7.42
7.42
7.42
7.29
7.28
7.28
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7.27
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7.27
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7.26
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7.18
7.18
7.17
7.16
7.16
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7.06
7.05
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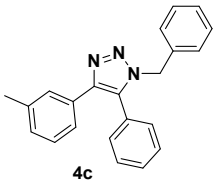
2.30



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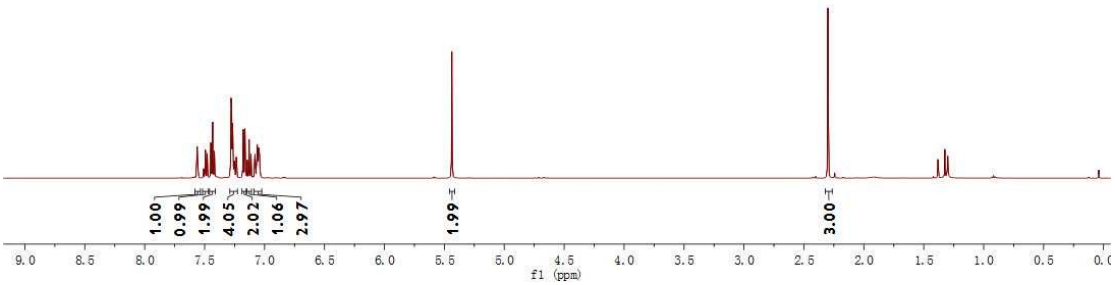
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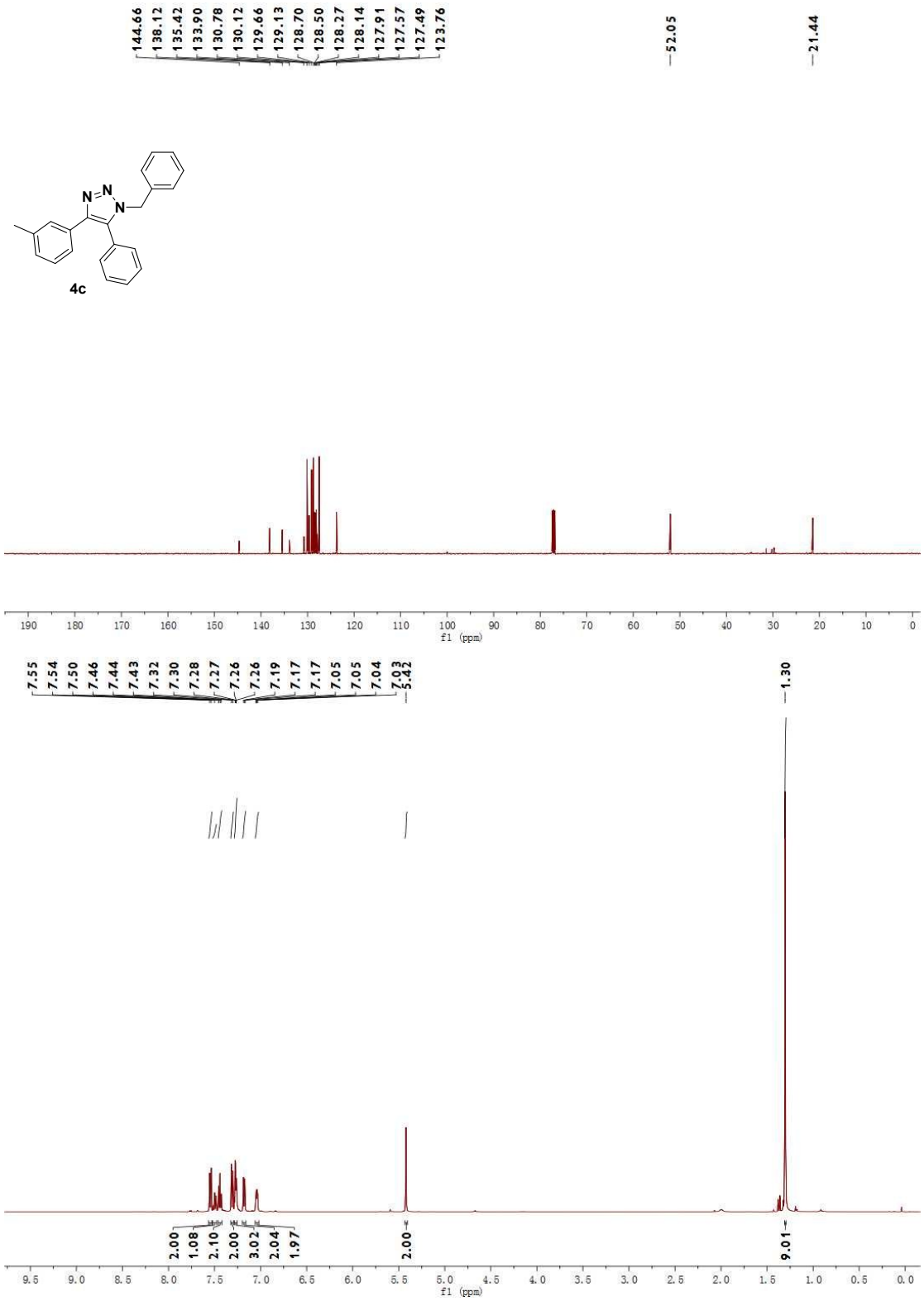
f1 (ppm)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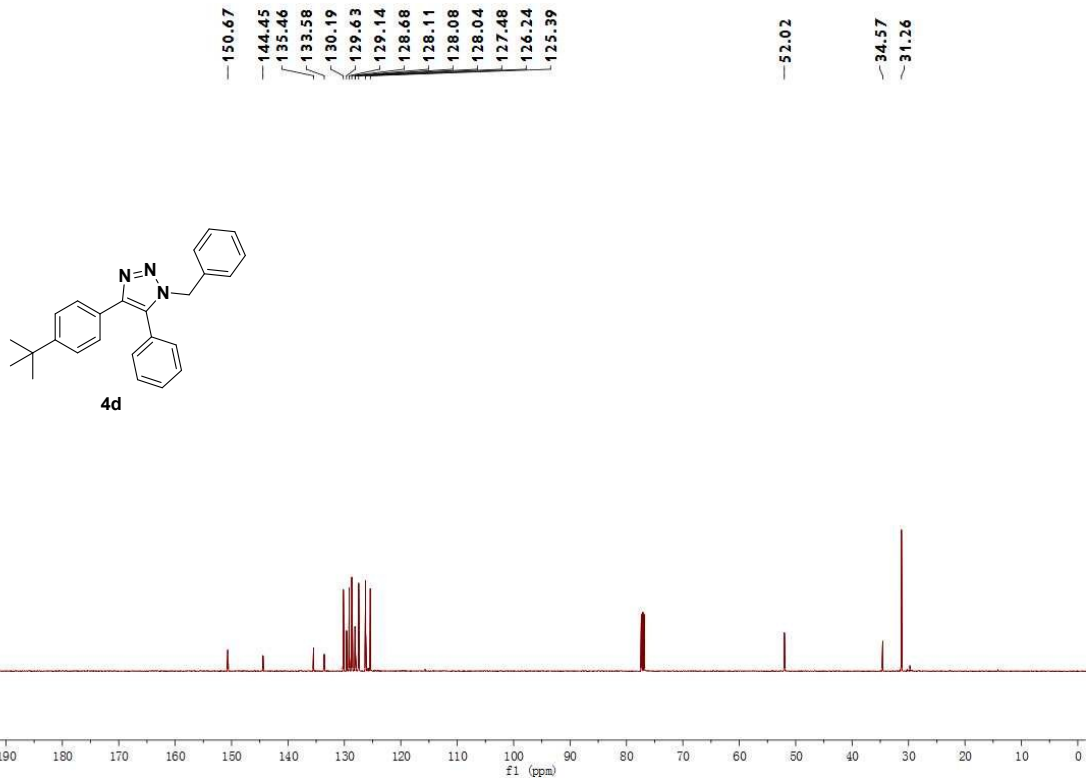
123.76
52.05
21.44
4c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
7.55
7.54
7.50
7.46
7.44
7.43
7.32
7.30
7.28
7.27
7.26
7.26
7.19
7.17
7.17
7.05
7.05
7.04
7.03
5.42
1.30
2.00
1.08
2.10
2.00
3.02
2.04
1.97
2.00
9.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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31.26
4d
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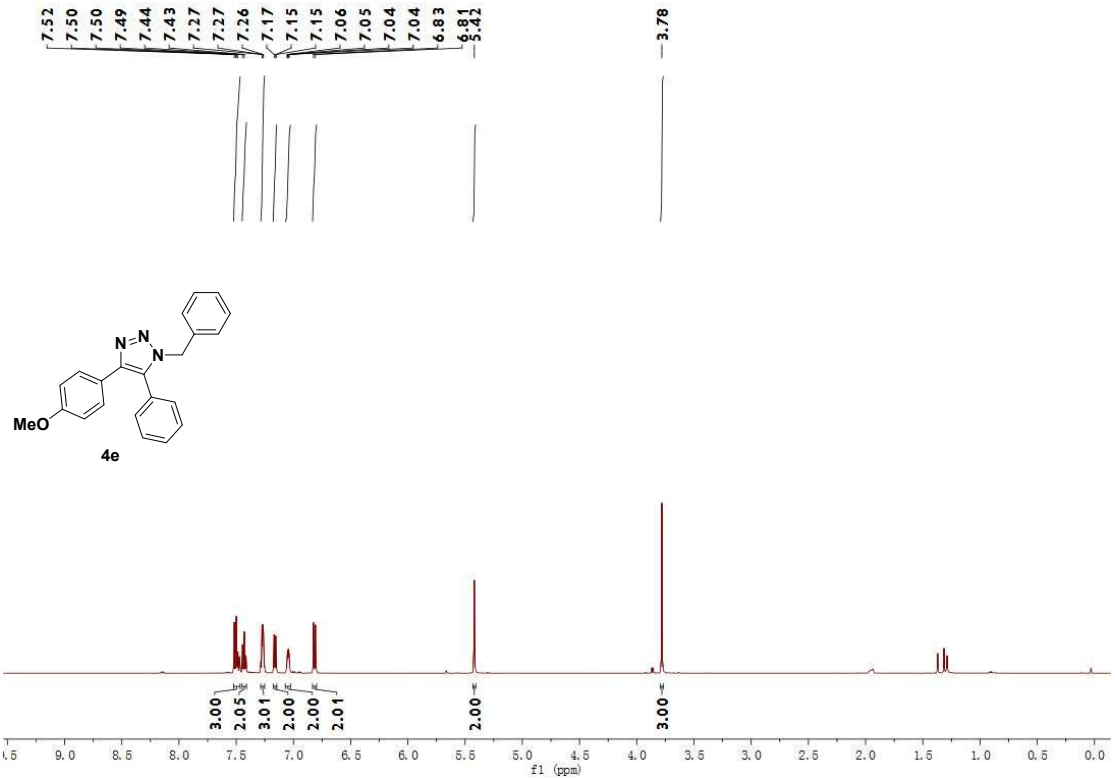
f1 (ppm)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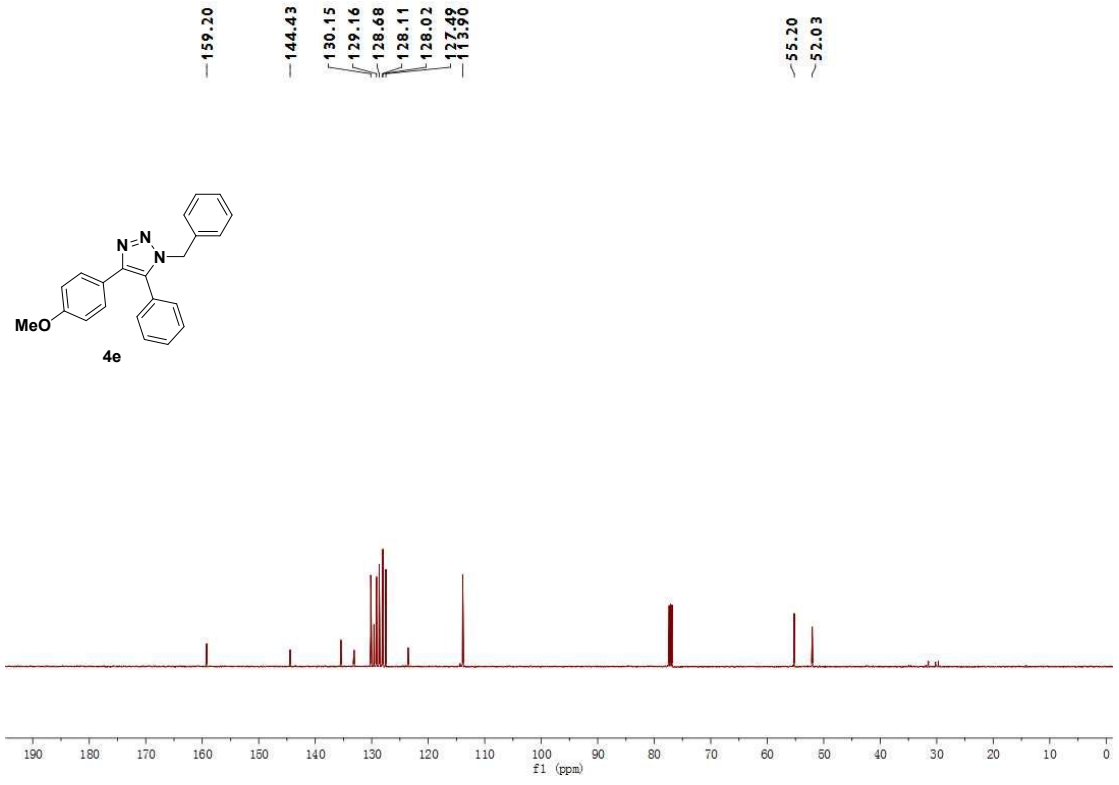
7.05
7.04
7.04
6.83
6.81
5.42
3.78
MeO
4e
3.00
2.05
3.01
2.00
2.00
2.01
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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MeO
4e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
O2N
4f
1.97
2.01
1.03
2.02
3.02
2.00
2.00
2.00
9.5
9.0
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8.0
7.5
7.0
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6.0
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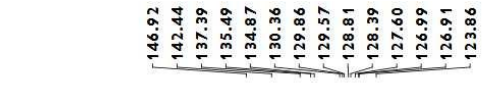
f1 (ppm)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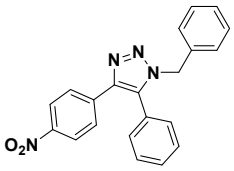
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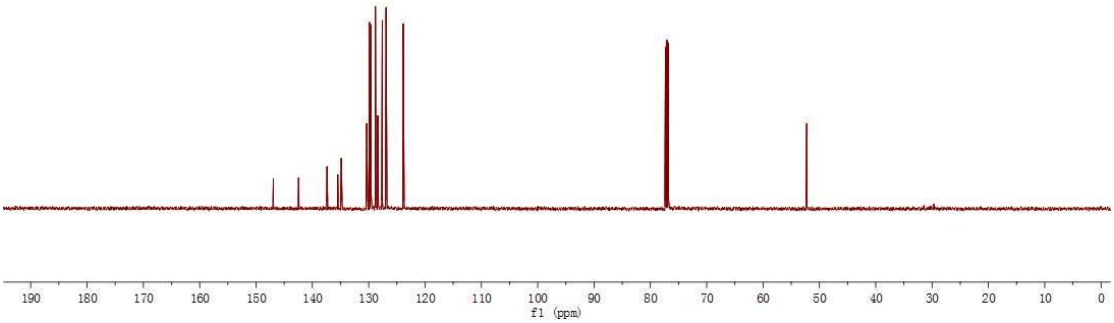
N
O2N

4f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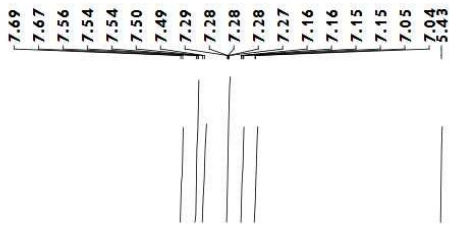
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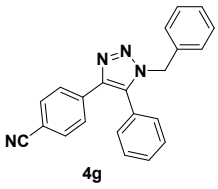
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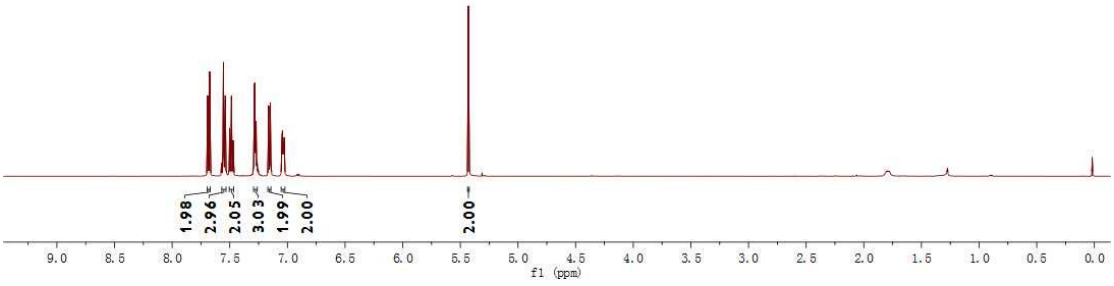
4g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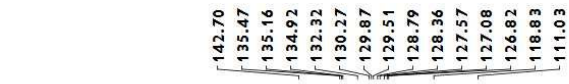
f1 (ppm)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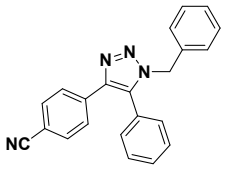
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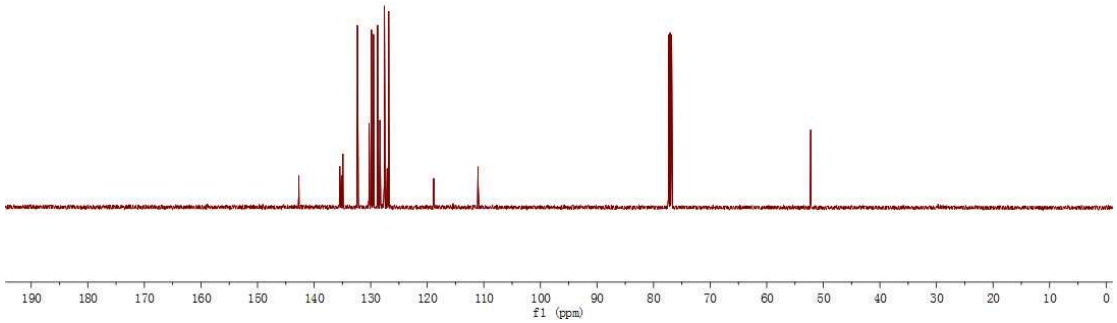
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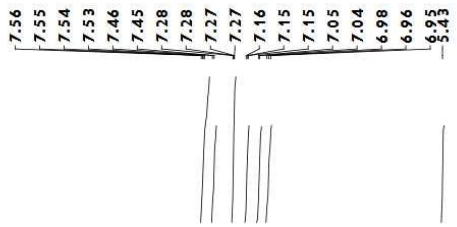
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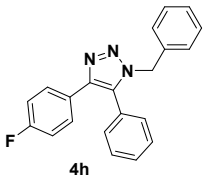
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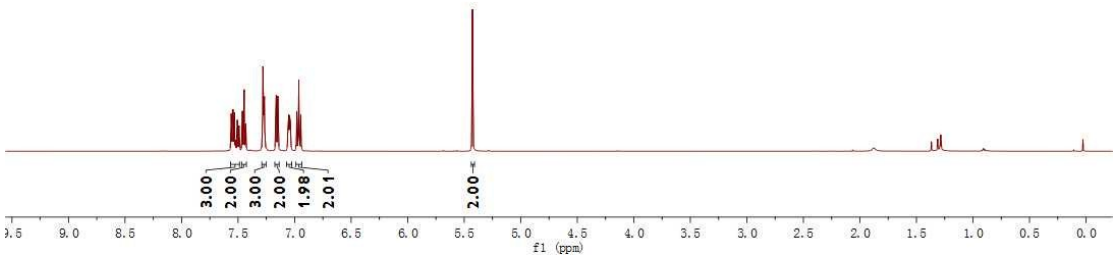
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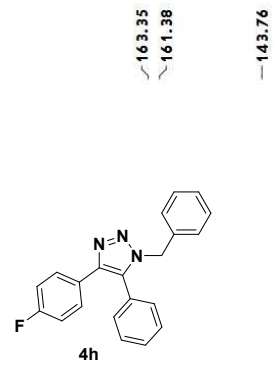
0.0
f1 (ppm)


F

4h

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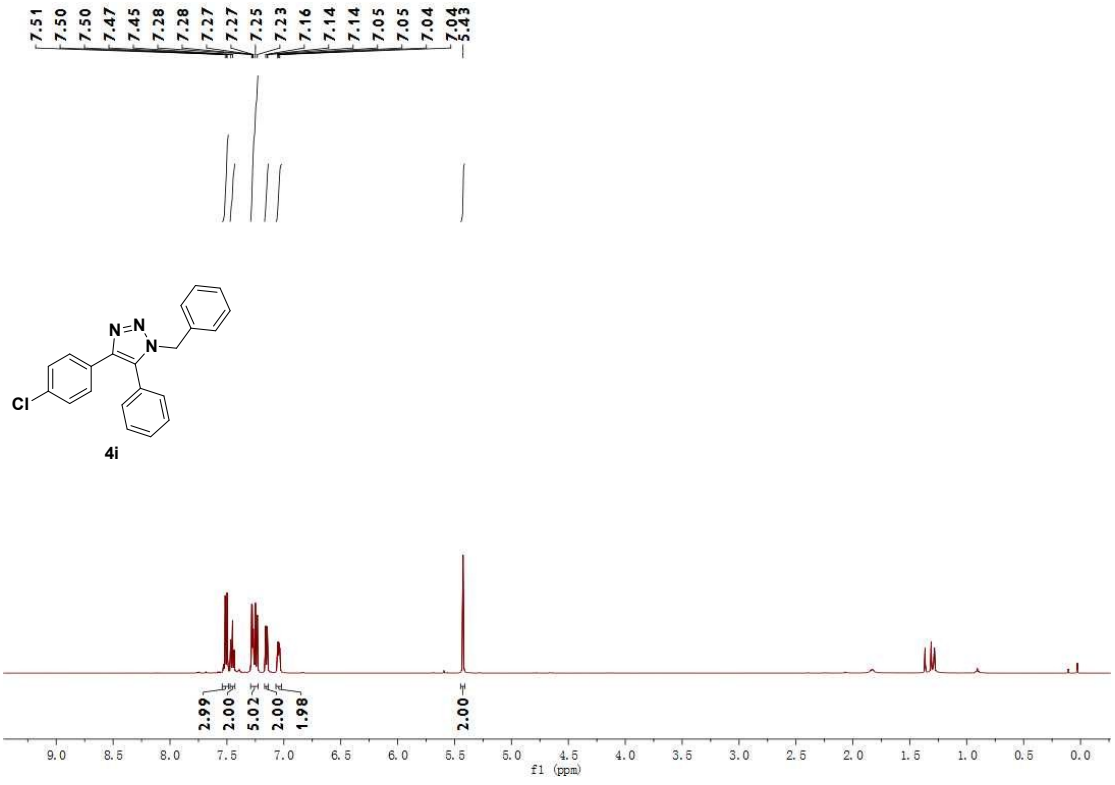
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7.05
7.04
7.04
5.43
2.99
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5.02
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8.0
7.5
7.0
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6.0
5.5
5.0
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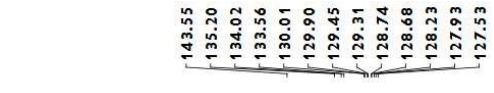
f1 (ppm)

Cl

4i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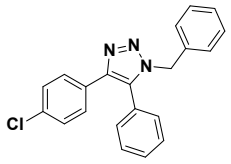
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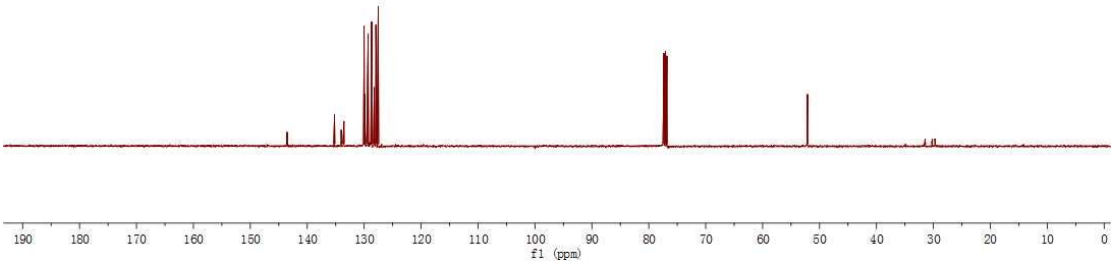
N
Cl

4i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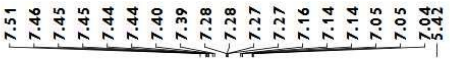
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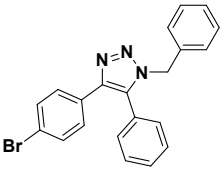
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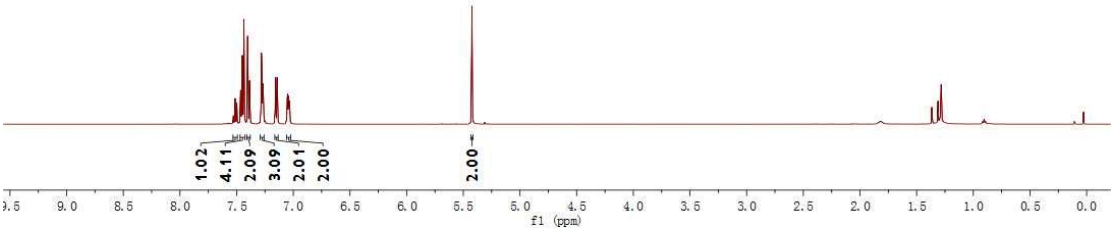
Br

4j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6.5
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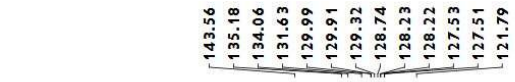
f1 (ppm)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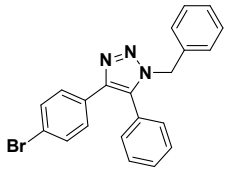
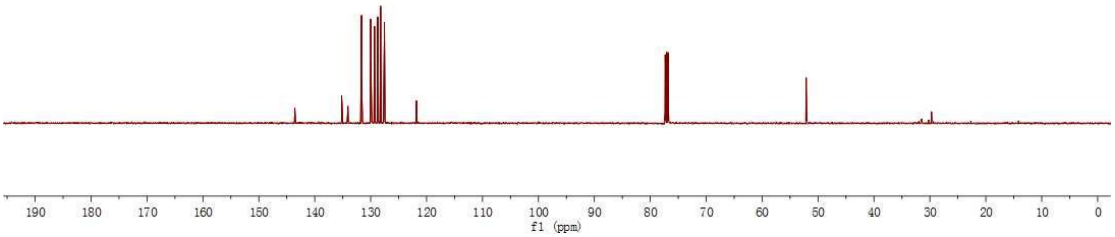

N
N
N
Br

4j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180
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100
90
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70
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50
40
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10
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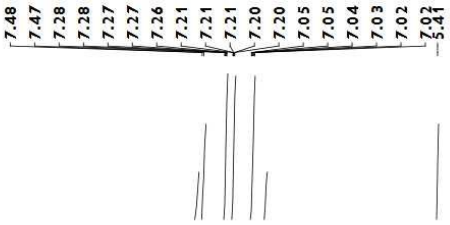
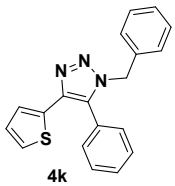
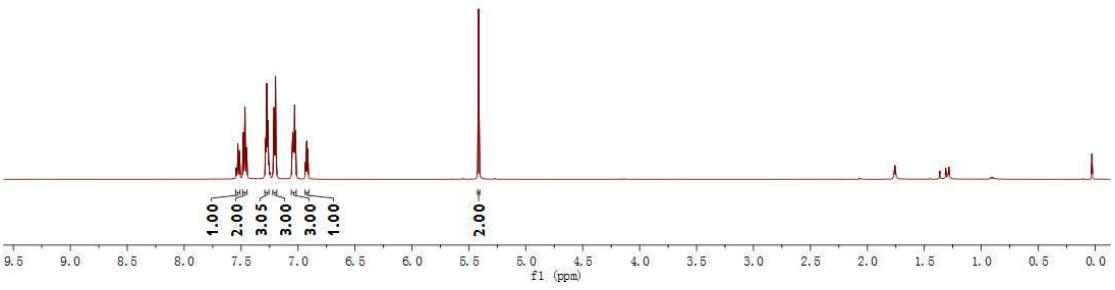
f1 (ppm)


7.48
7.47
7.28
7.28
7.27
7.27
7.26
7.21
7.21
7.21
7.20
7.20
7.05
7.05
7.04
7.03
7.02
7.02
5.41


N
N
N
S

4k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2.00
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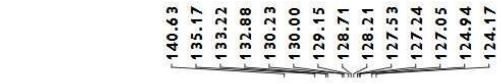
3.00
3.00
1.00
2.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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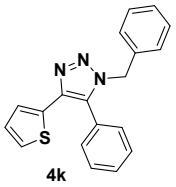
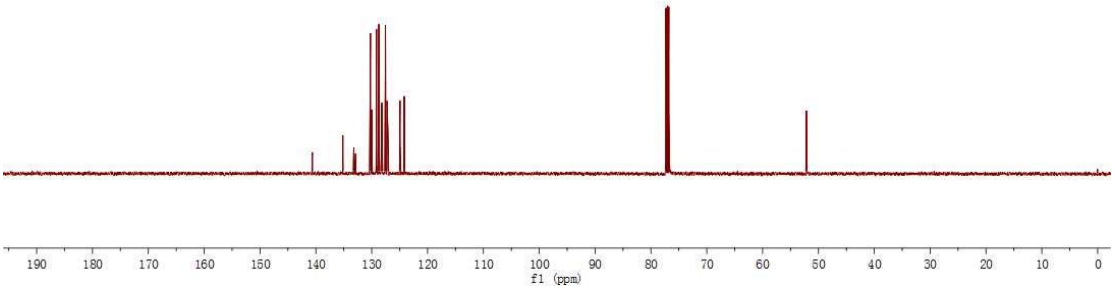
128.71
128.21
127.53
127.24
127.05
124.94
124.17
52.16


N
N
N
S

4k


190
180
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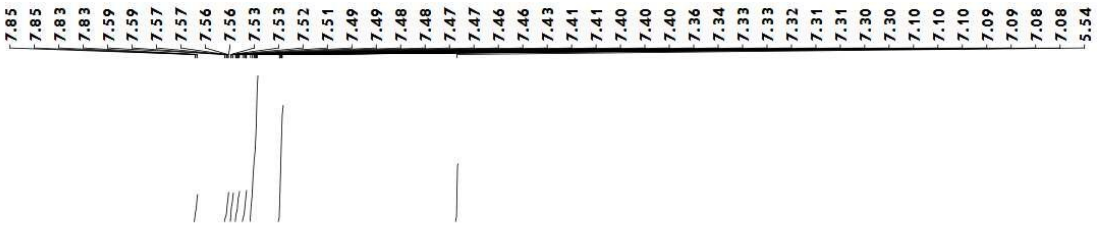
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.85
7.85
7.83
7.83
7.59
7.59
7.57
7.57
7.56
7.56
7.53
7.53
7.52
7.51
7.49
7.49
7.48
7.48
7.47
7.47
7.46
7.46
7.43
7.41
7.41
7.40
7.40
7.36
7.34
7.33
7.33
7.32
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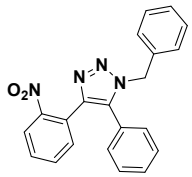
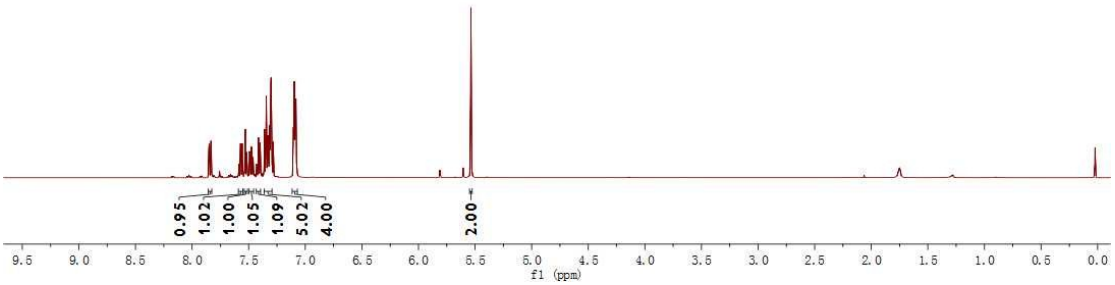
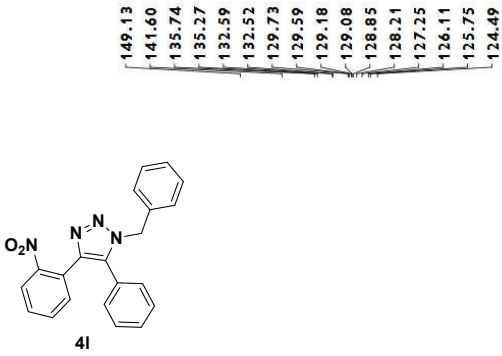
7.10
7.09
7.09
7.08
7.08
5.54

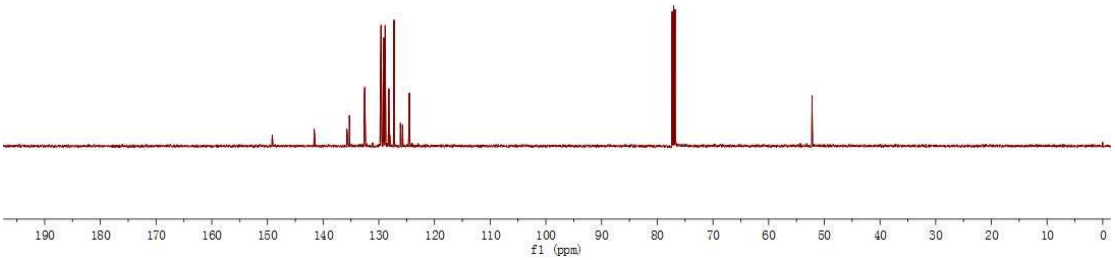

O2N
N
N
N

4l


0.95
1.02
1.00
1.05
1.09
5.02
4.00
2.00
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
f1 (ppm)


149.13
141.60
135.74
135.27
132.59
132.52
129.73
129.59
129.18
129.08
128.85
128.21
127.25
126.11
125.75
124.49
52.20
O2N
N
N
N

4l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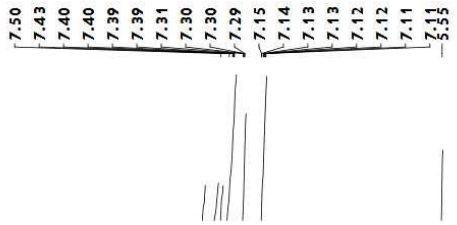
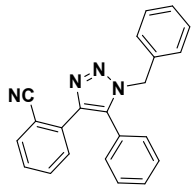
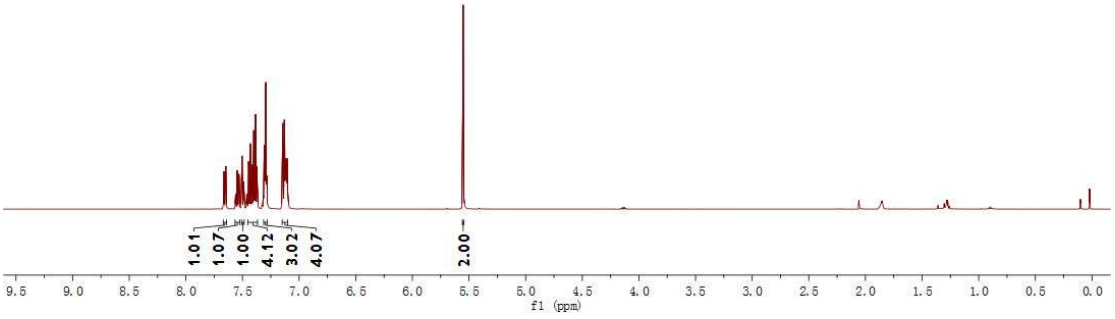
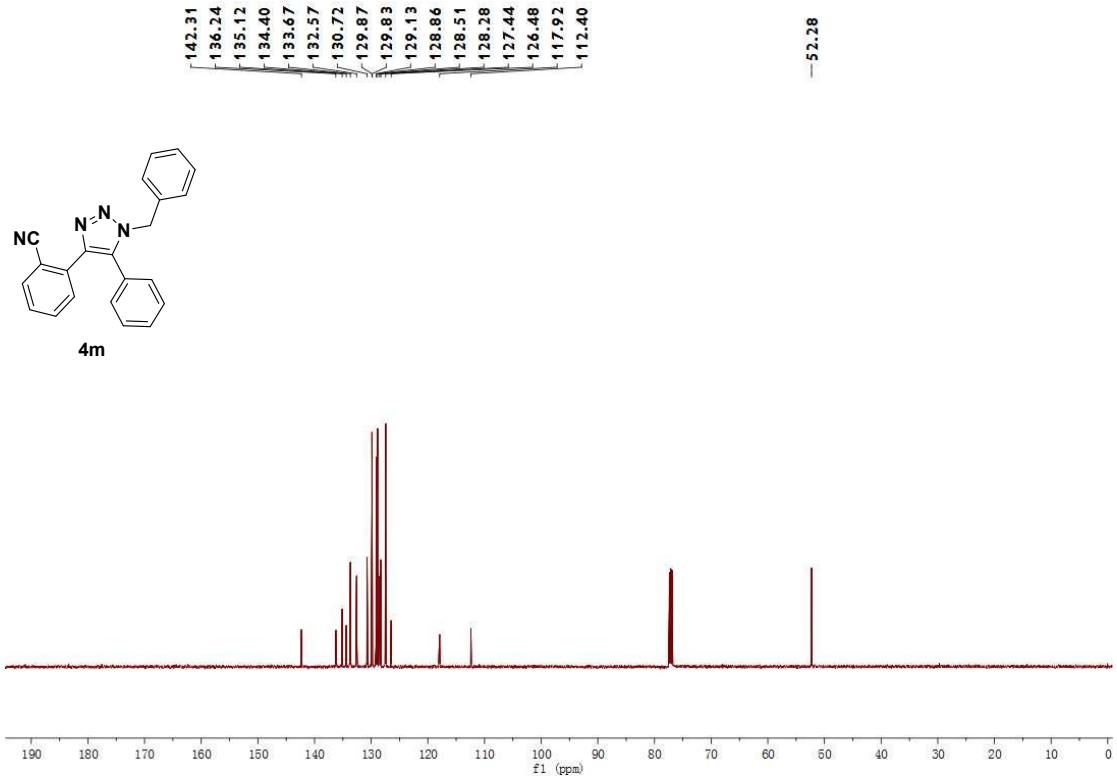
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

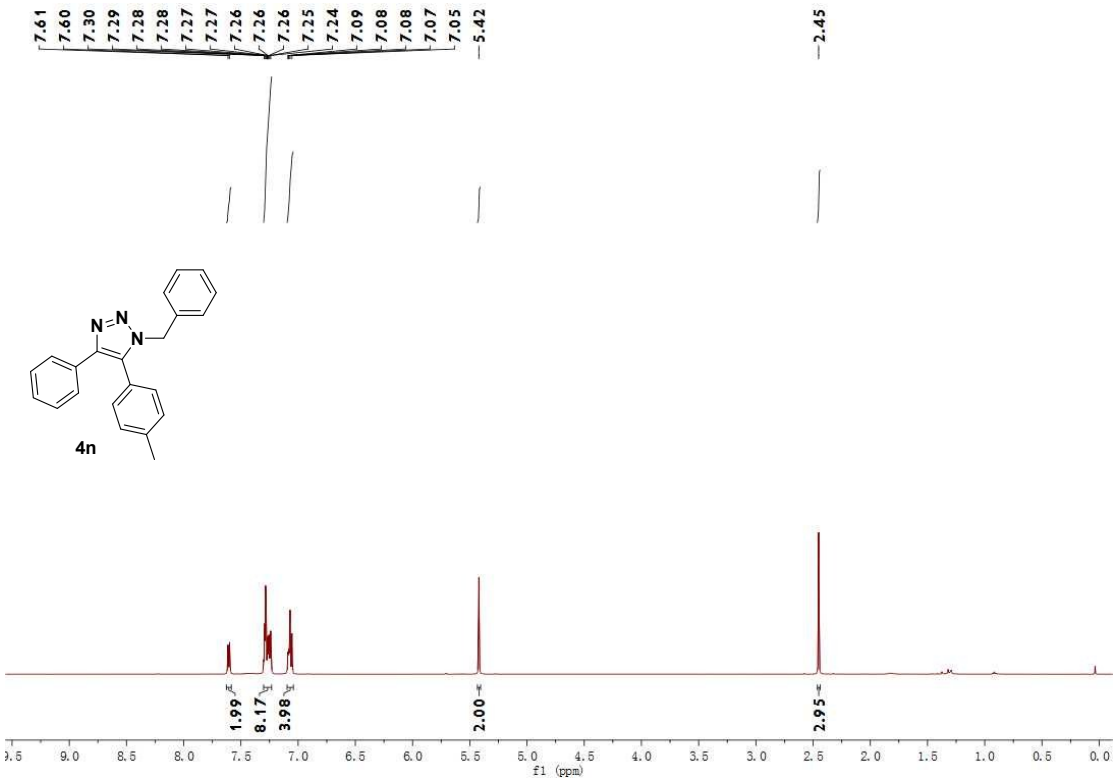

7.50
7.43
7.40
7.40
7.39
7.39
7.31
7.30
7.30
7.29
7.15
7.14
7.13
7.13
7.12
7.12
7.11
7.11
5.55

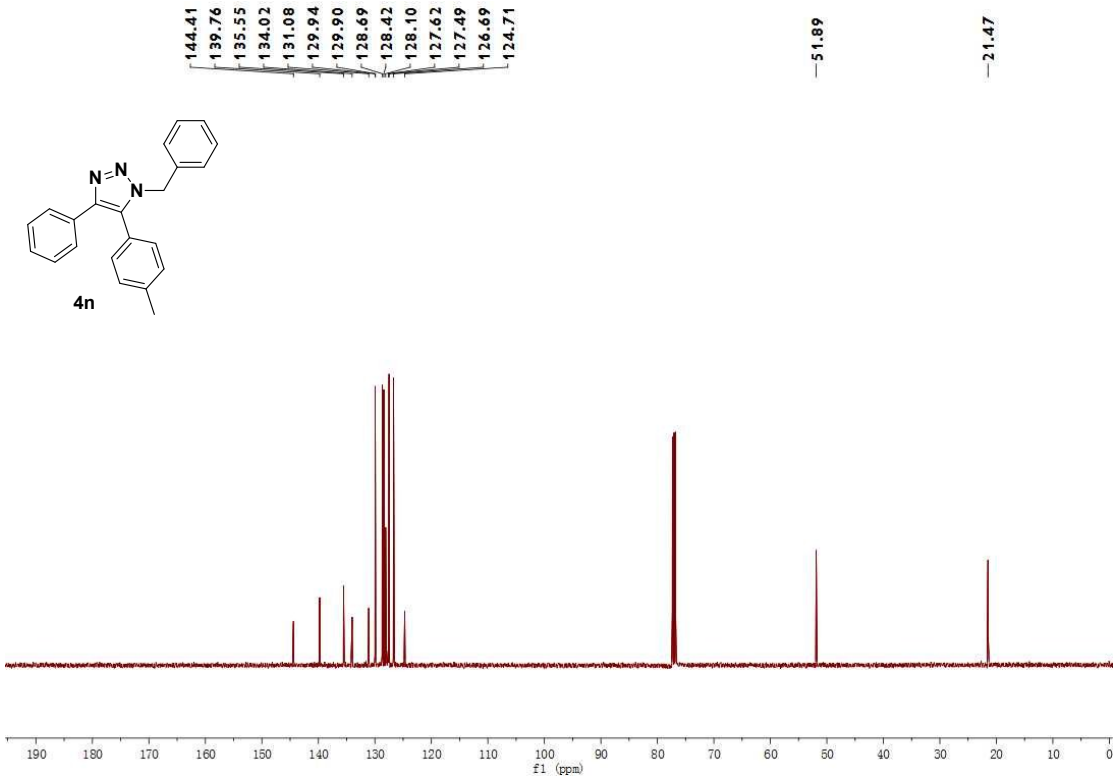

NC
N
N
N

4m


1.01
1.07
1.00
4.12
3.02
4.07
2.00
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
f1 (ppm)


142.31
136.24
135.12
134.40
133.67
132.57
130.72
129.87
129.83
129.13
128.86
128.51
128.28
127.44
126.48
117.92
112.40
52.28
NC
N
N
N
4m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.61
7.60
7.30
7.29
7.28
7.28
7.27
7.27
7.26
7.26
7.26
7.25
7.24
7.09
7.08
7.08
7.07
7.05
5.42
2.45
N
N
N
4n
1.99
8.17
3.98
2.00
2.95
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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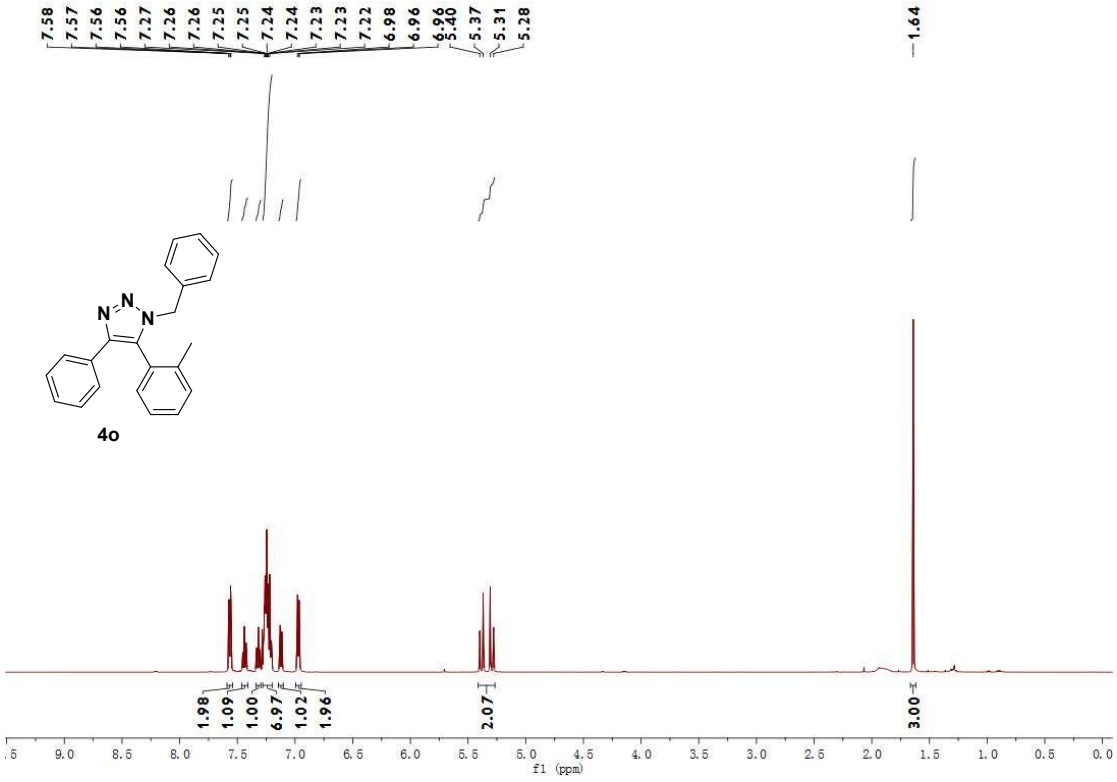
139.76
135.55
134.02
131.08
129.94
129.90
128.69
128.42
128.10
127.62
127.49
126.69
124.71
51.89
21.47
4n
f1 (ppm)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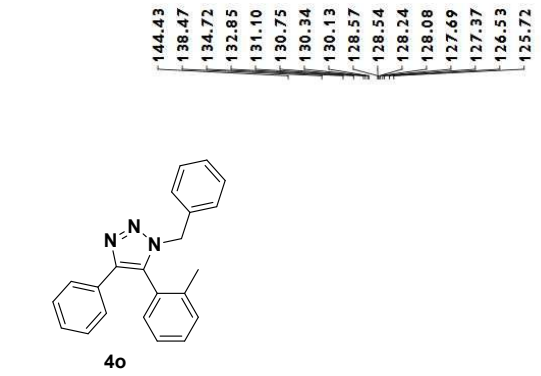
7.56
7.56
7.27
7.26
7.26
7.25
7.25
7.24
7.24
7.23
7.23
7.22
6.98
6.96
6.96
5.40
5.37
5.31
5.28
1.64
4o
1.98
1.09
1.00
6.97
1.02
1.96
2.07
3.00
f1 (ppm)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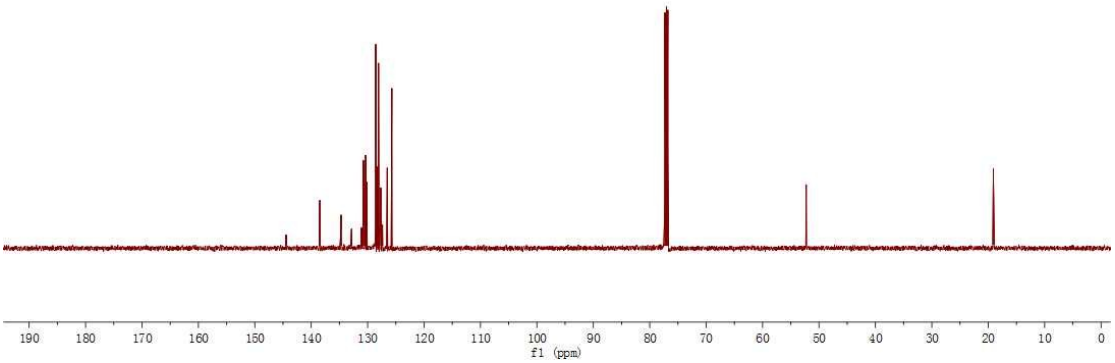
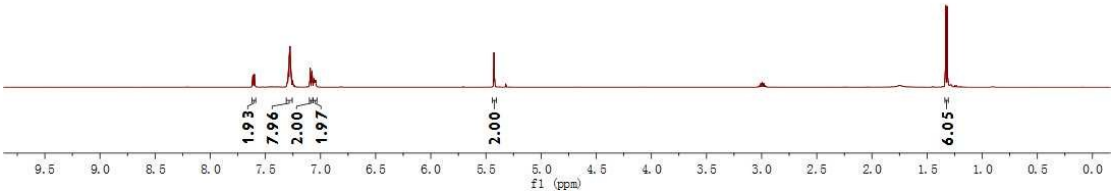
138.47
134.72
132.85
131.10
130.75
130.34
130.13
128.57
128.54
128.24
128.08
127.69
127.37
126.53
125.72
52.27
19.09

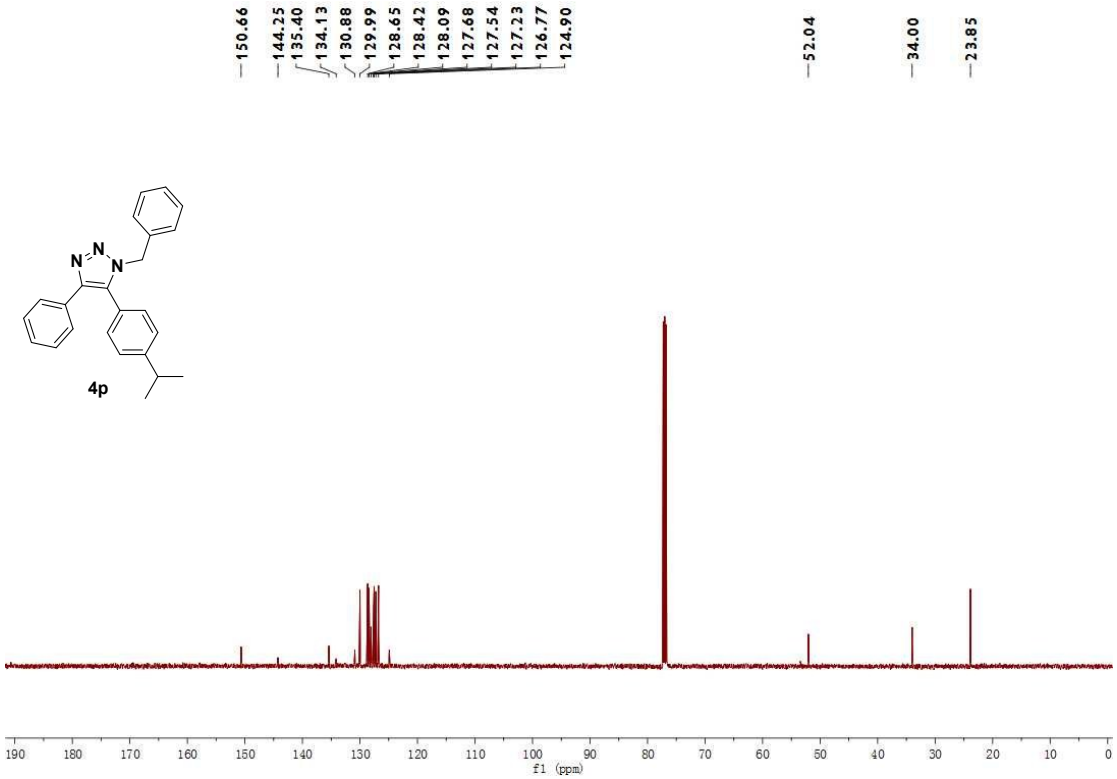
4o


190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.62
7.61
7.60
7.60
7.29
7.29
7.28
7.28
7.28
7.27
7.27
7.27
7.26
7.09
7.08
7.06
7.06
7.05
7.04
5.43
1.33
1.32

4p


1.93
7.96
2.00
1.97
2.00
6.05
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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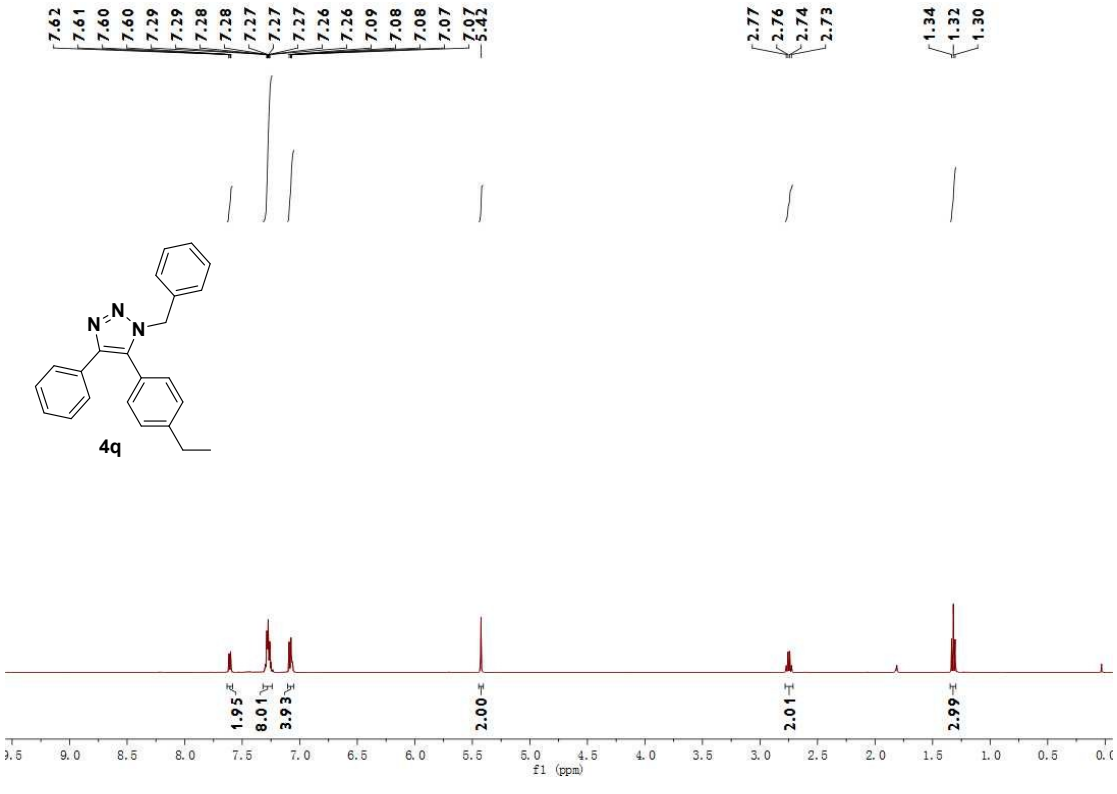
144.25
135.40
134.13
130.88
129.99
128.65
128.42
128.09
127.68
127.54
127.23
126.77
124.90
52.04
34.00
23.85
4p
f1 (ppm)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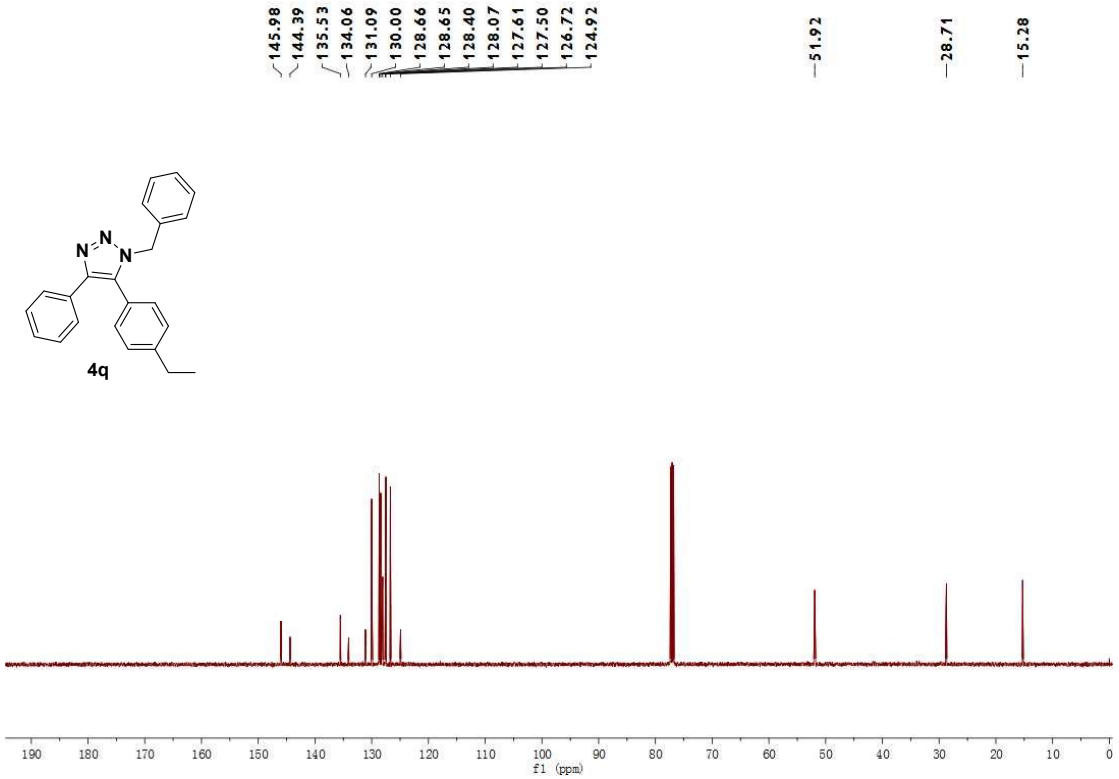
7.61
7.60
7.60
7.29
7.29
7.28
7.28
7.27
7.27
7.27
7.26
7.26
7.09
7.08
7.08
7.07
7.07
5.42
2.77
2.76
2.74
2.73
1.34
1.32
1.30
4q
1.95
8.01
3.93
2.00
2.01
2.99
f1 (ppm)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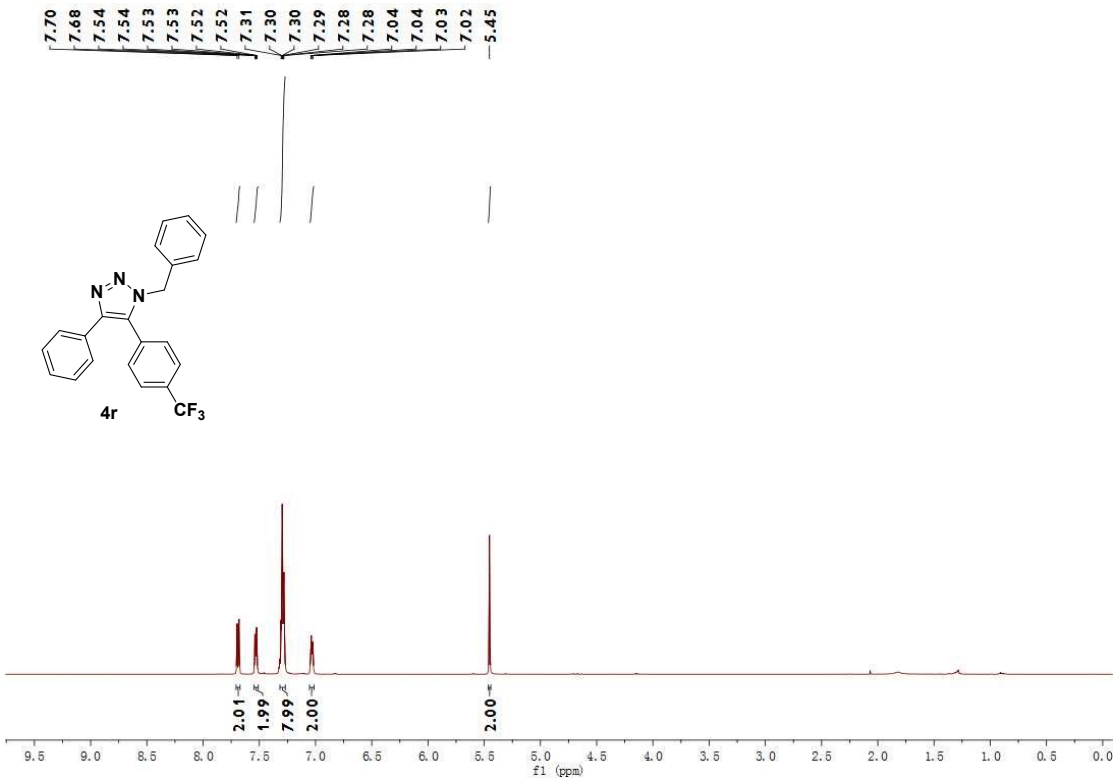
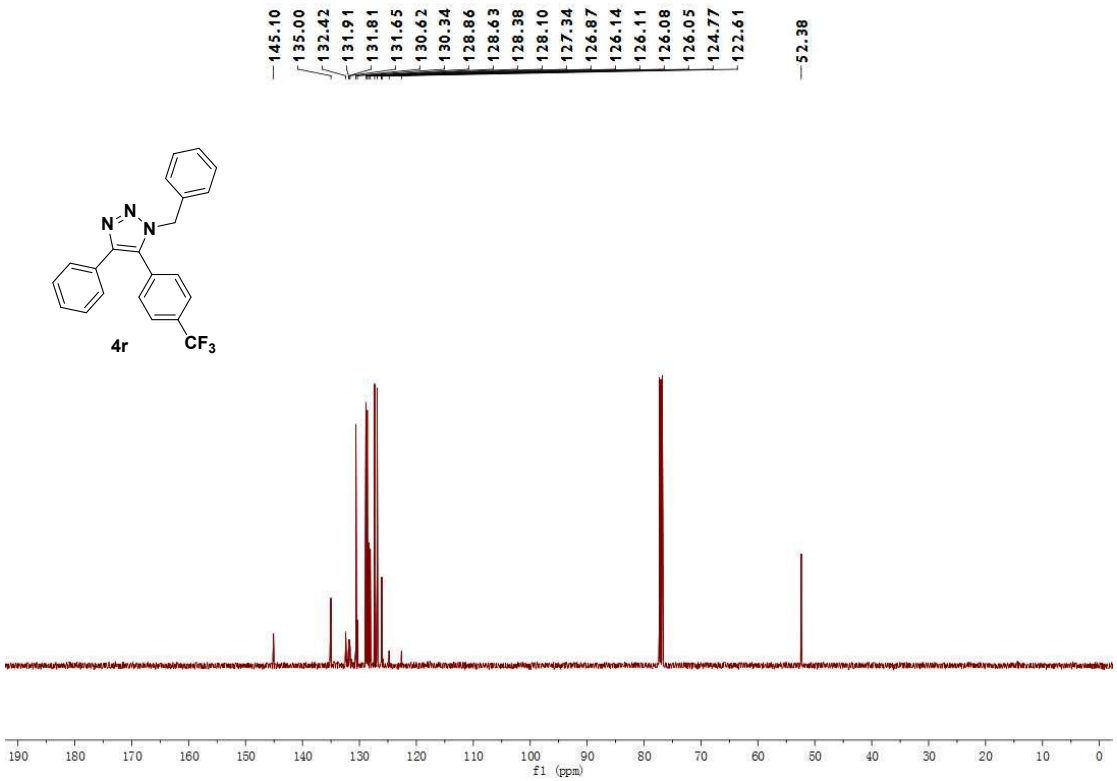
135.53
134.06
131.09
130.00
128.66
128.65
128.40
128.07
127.61
127.50
126.72
124.92
51.92
28.71
15.28
4q
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.70
7.68
7.54
7.54
7.53
7.53
7.52
7.52
7.31
7.30
7.30
7.29
7.28
7.28
7.04
7.04
7.03
7.02
5.45
4r
CF3
2.01
1.99
7.99
2.00
2.00
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
f1 (ppm)


145.10
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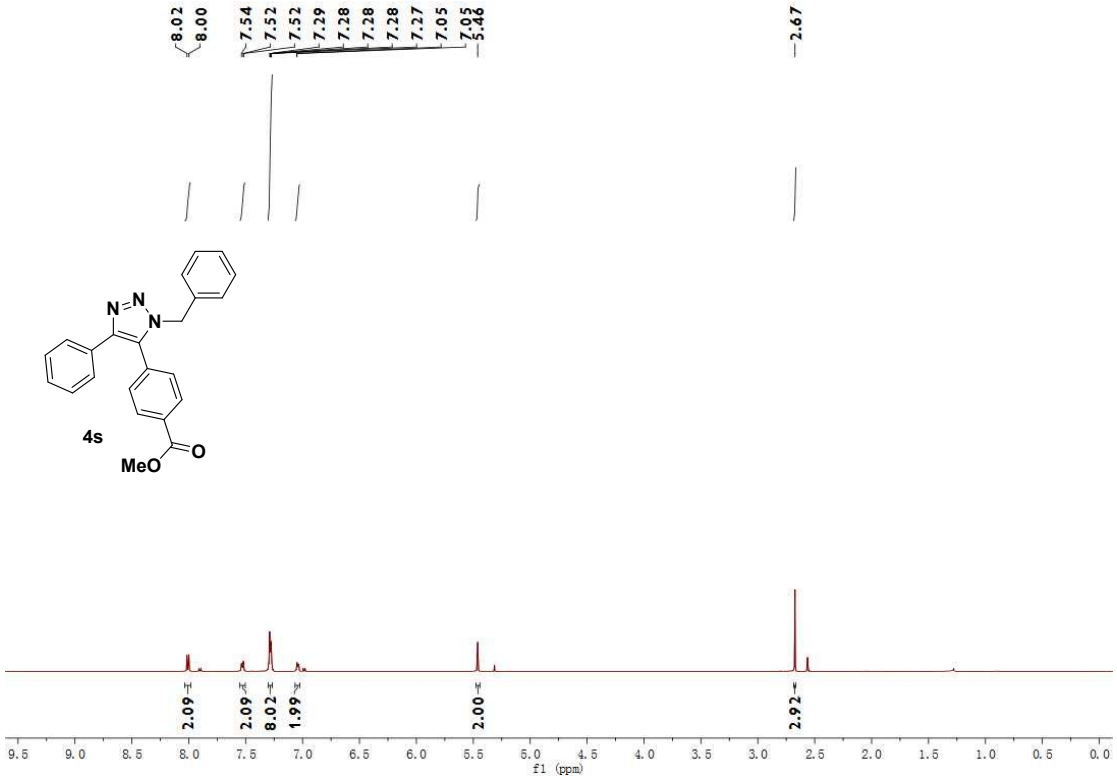
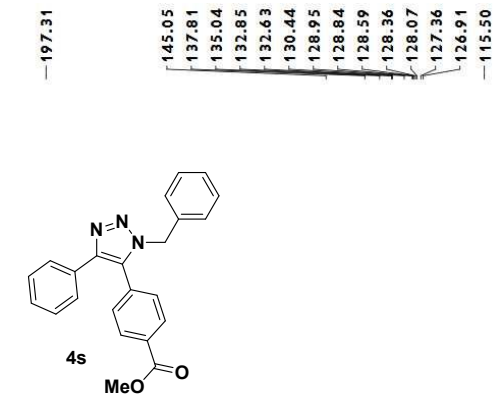
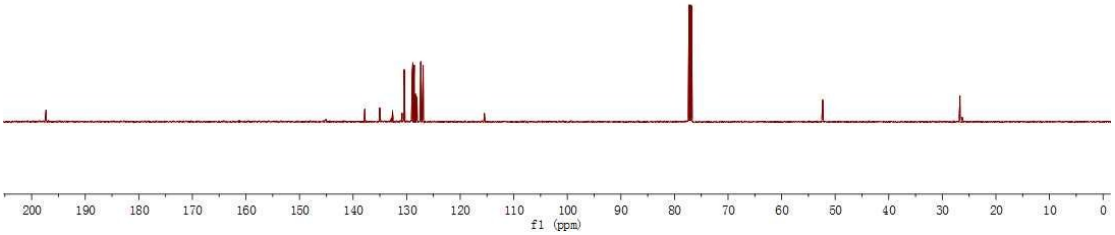
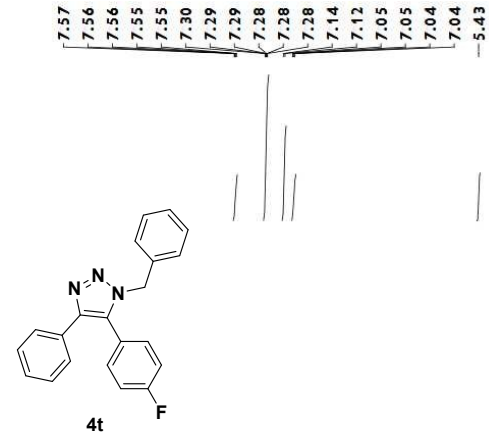
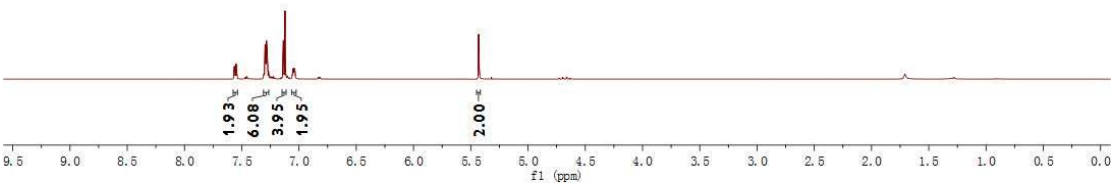
131.91
131.81
131.65
130.62
130.34
128.86
128.63
128.38
128.10
127.34
126.87
126.14
126.11
126.08
126.05
124.77
122.61
52.38
4r
CF3
f1 (ppm)

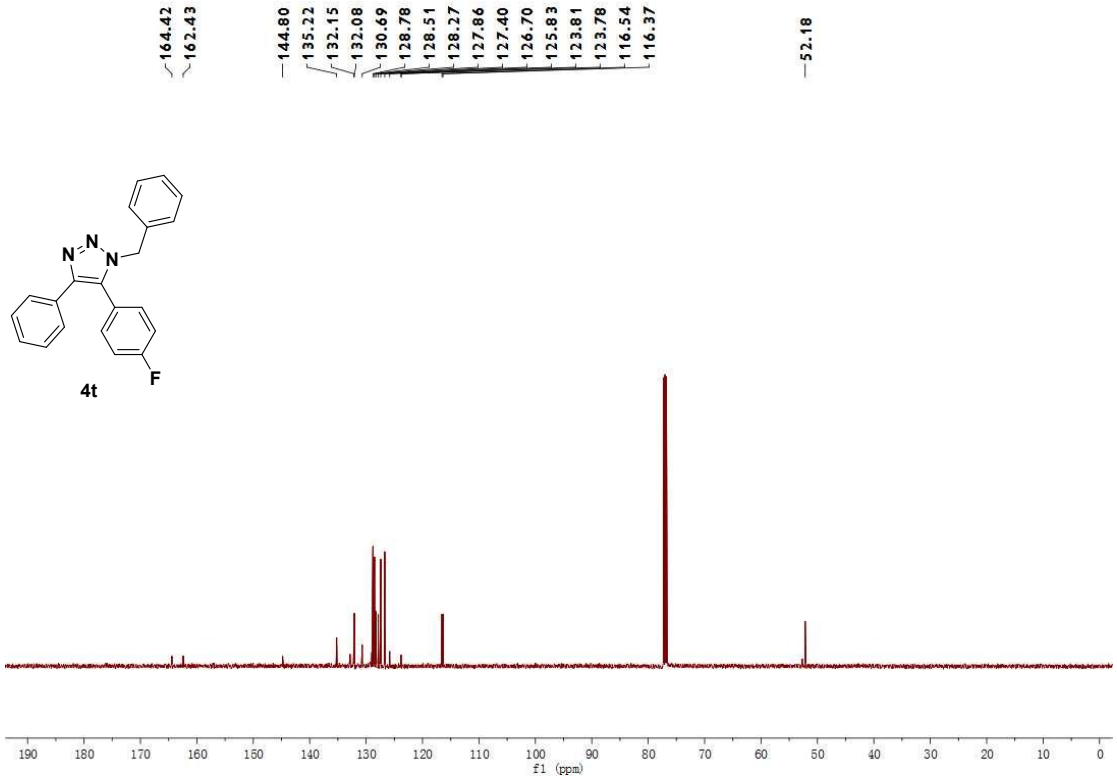

8.02
8.00
7.54
7.52
7.52
7.29
7.28
7.28
7.28
7.27
7.05
7.05
5.46
2.67
4s
MeO
O
2.09
2.09
8.02
1.99
2.00
2.92
f1 (ppm)

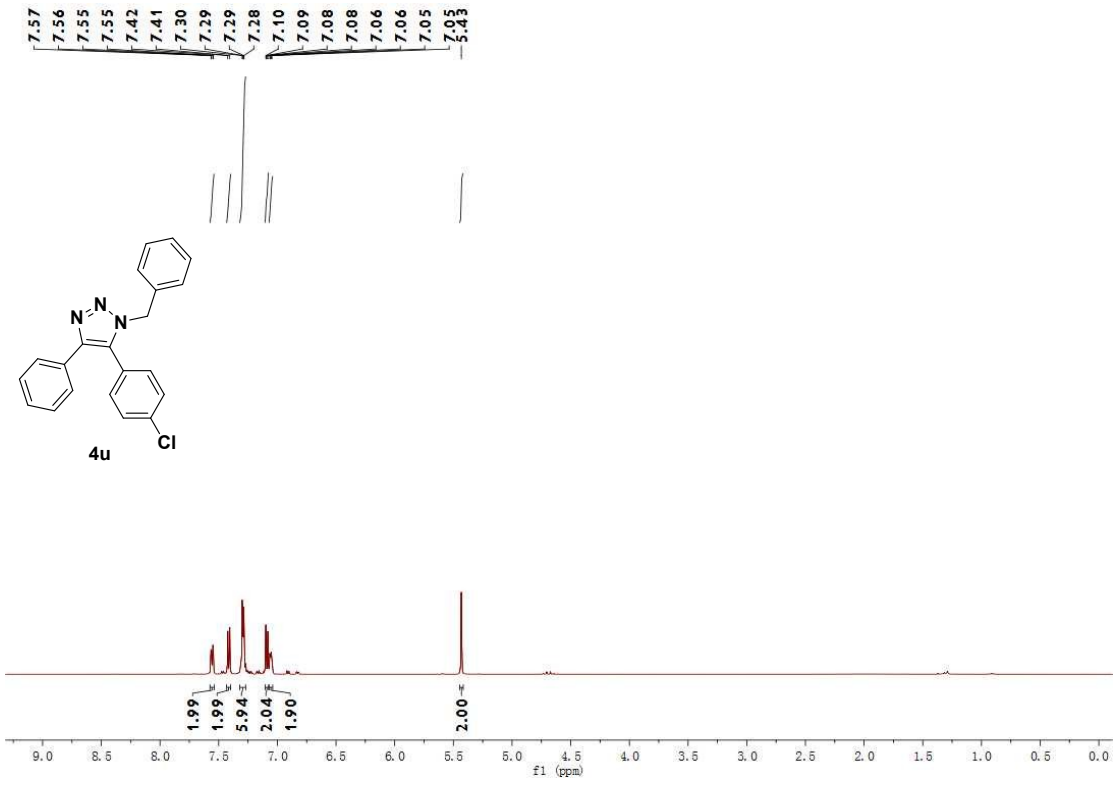

197.31
145.05
137.81
135.04
132.85
132.63
130.44
128.95
128.84
128.59
128.36
128.07
127.36
126.91
115.50
52.34
26.71
4s
MeO
O


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.57
7.56
7.56
7.55
7.55
7.30
7.29
7.29
7.28
7.28
7.28
7.14
7.12
7.05
7.05
7.04
7.04
5.43
4t
F


1.93
6.08
3.95
1.95
2.00
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
f1 (ppm)


164.42
162.43
144.80
135.22
132.15
132.08
130.69
128.78
128.51
128.27
127.86
127.40
126.70
125.83
123.81
123.78
116.54
116.37
52.18
F
4t
f1 (ppm)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7.09
7.08
7.08
7.06
7.06
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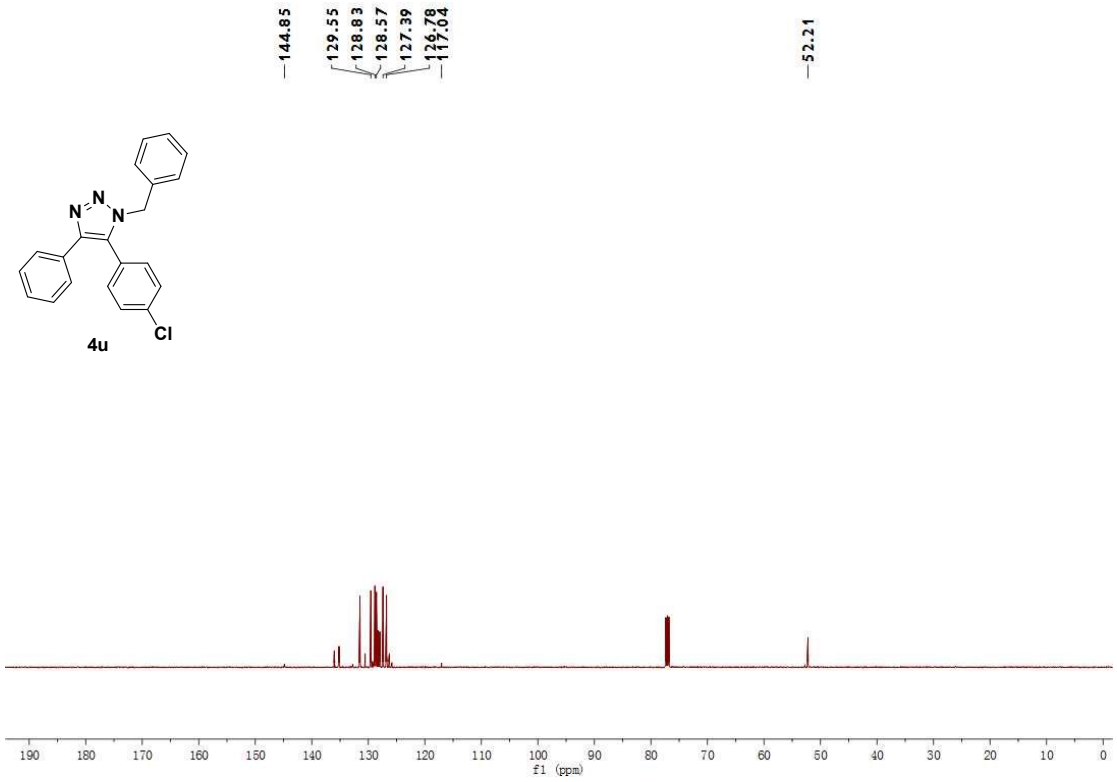
5.43
Cl
4u
1.99
1.99
5.94
2.04
1.90
2.00
f1 (ppm)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52.21
Cl
4u
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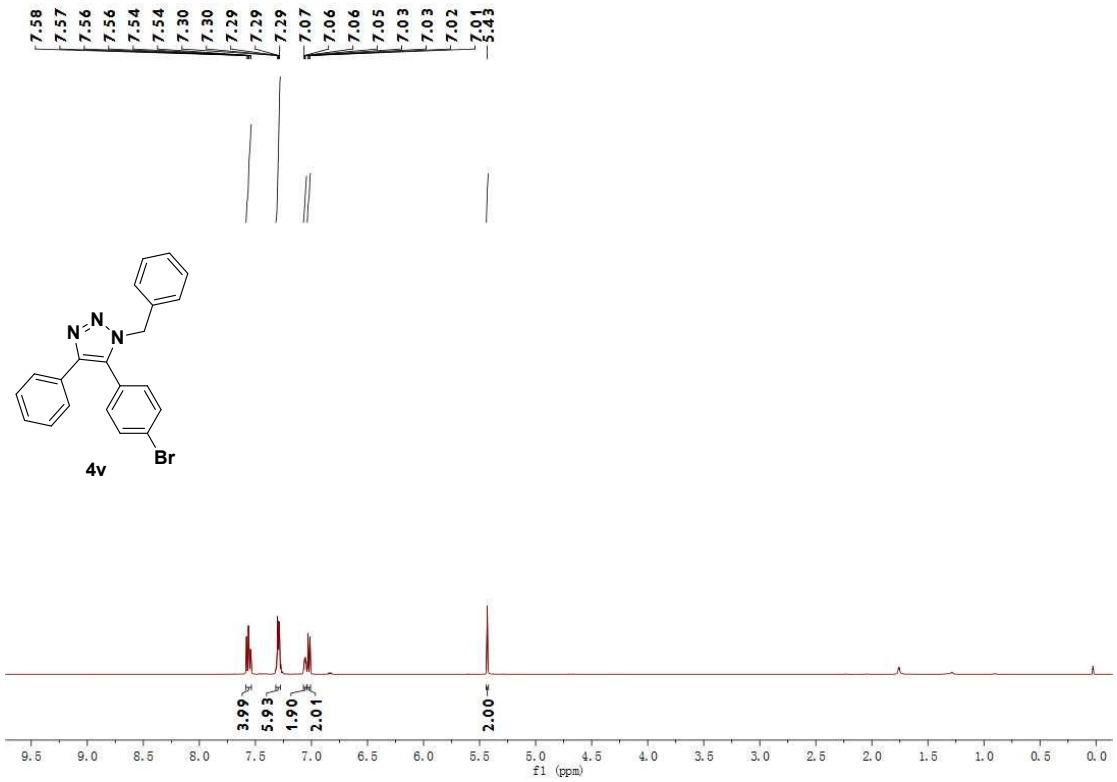
f1 (ppm)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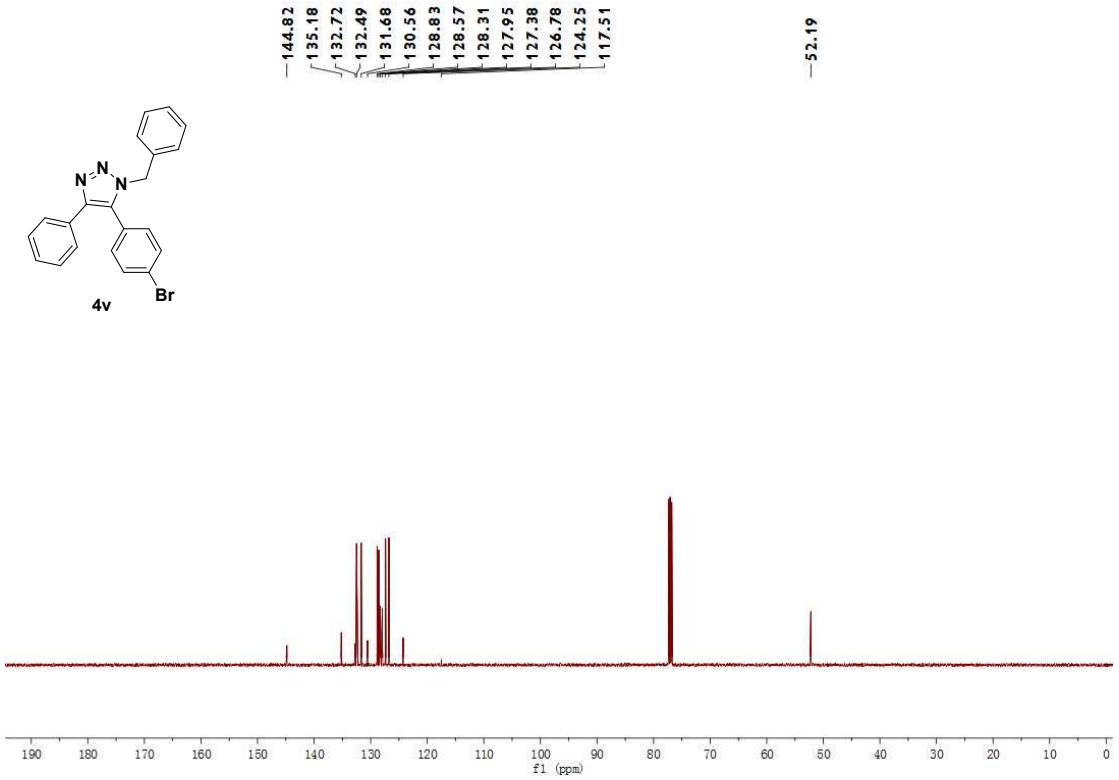
7.29
7.07
7.06
7.06
7.05
7.03
7.03
7.02
7.01
5.43
Br
4v
3.99
5.93
1.90
2.01
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.57
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127.95
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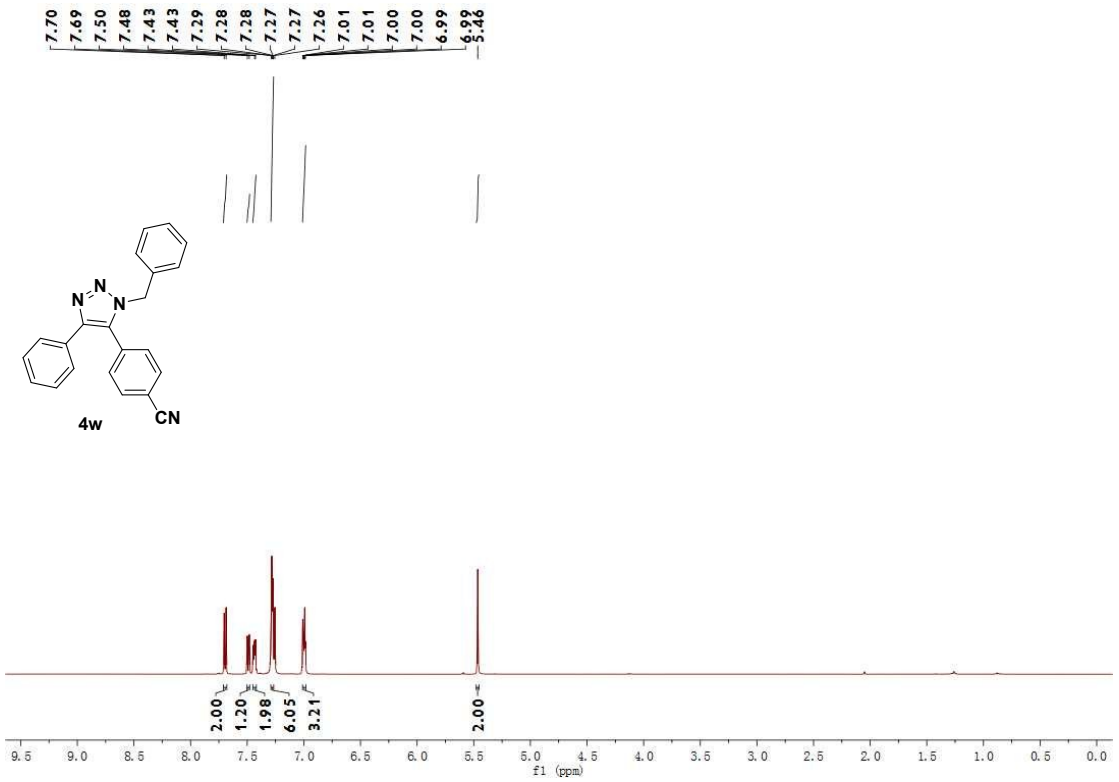
124.25
117.51
52.19
Br
4v
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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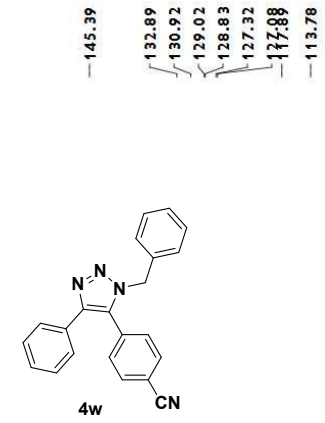
7.27
7.26
7.01
7.01
7.00
7.00
6.99
6.98
5.46
CN
4w
2.00
1.20
1.98
6.05
3.21
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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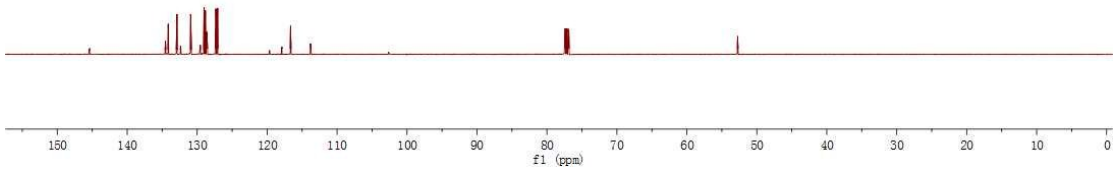
113.78
52.74
N
N
N
CN
4w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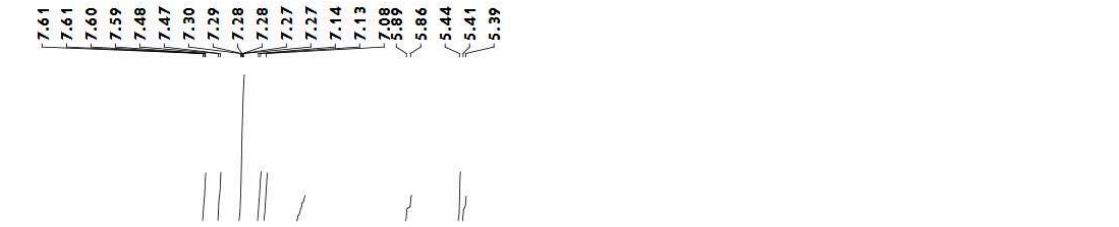
0
f1 (ppm)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7.08
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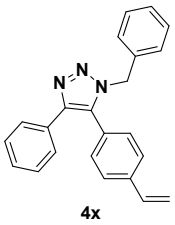
5.41
5.39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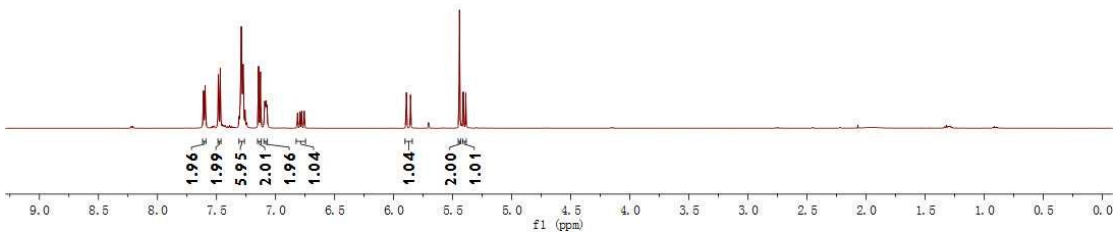
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1.96
1.04
1.04
2.00
1.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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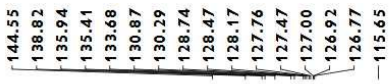
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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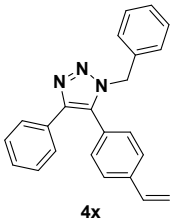
127.00
126.92
126.77
115.65
52.03



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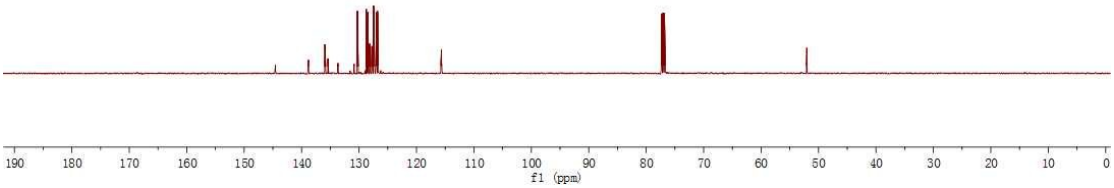
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70
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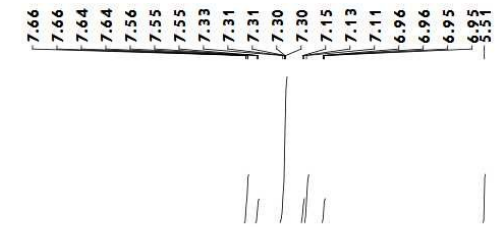
50
40
30
20
10
0
f1 (ppm)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7.11
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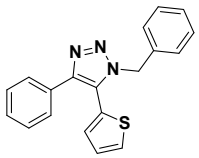
6.96
6.95
6.95
5.51



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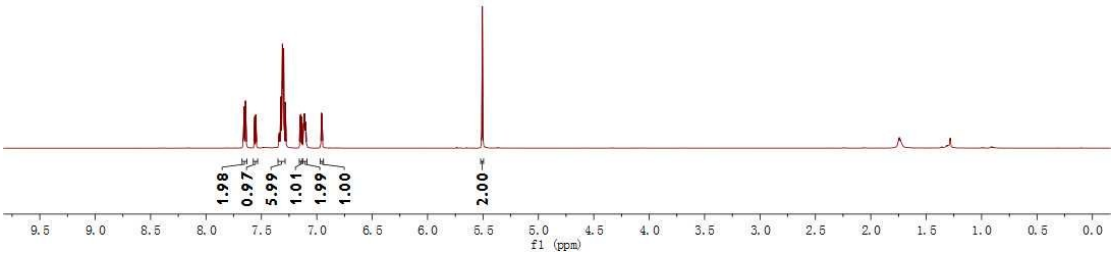
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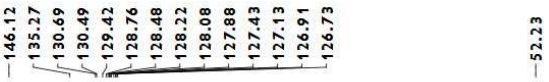
1.99
1.00
2.00
f1 (ppm)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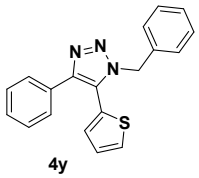
126.91
126.73
52.23



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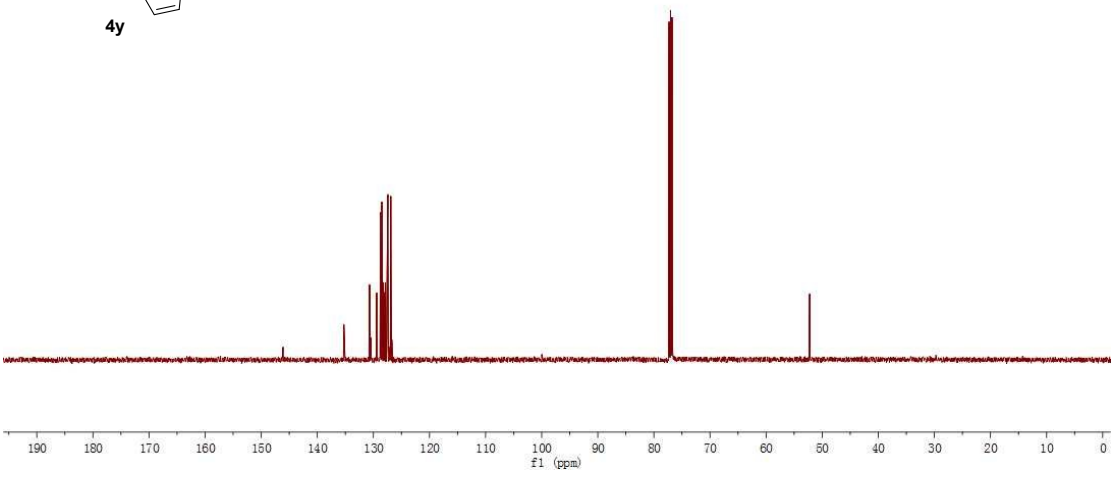
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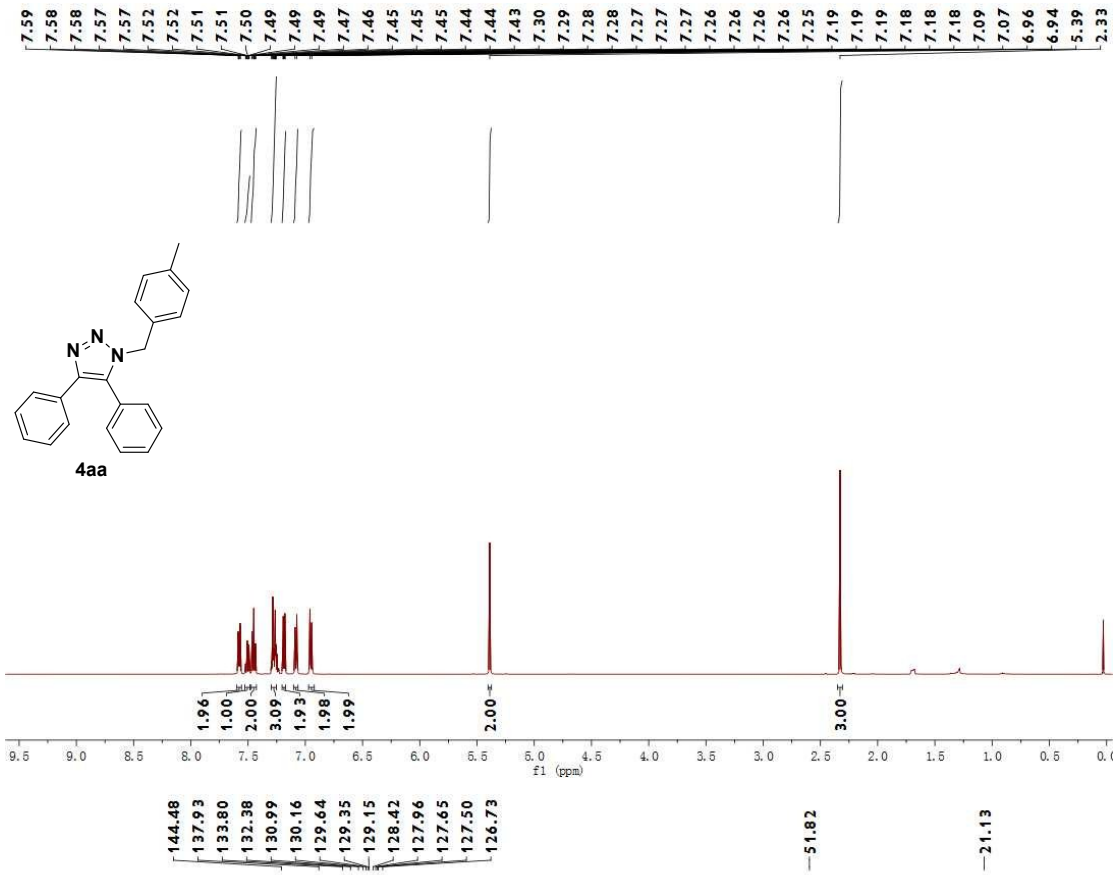
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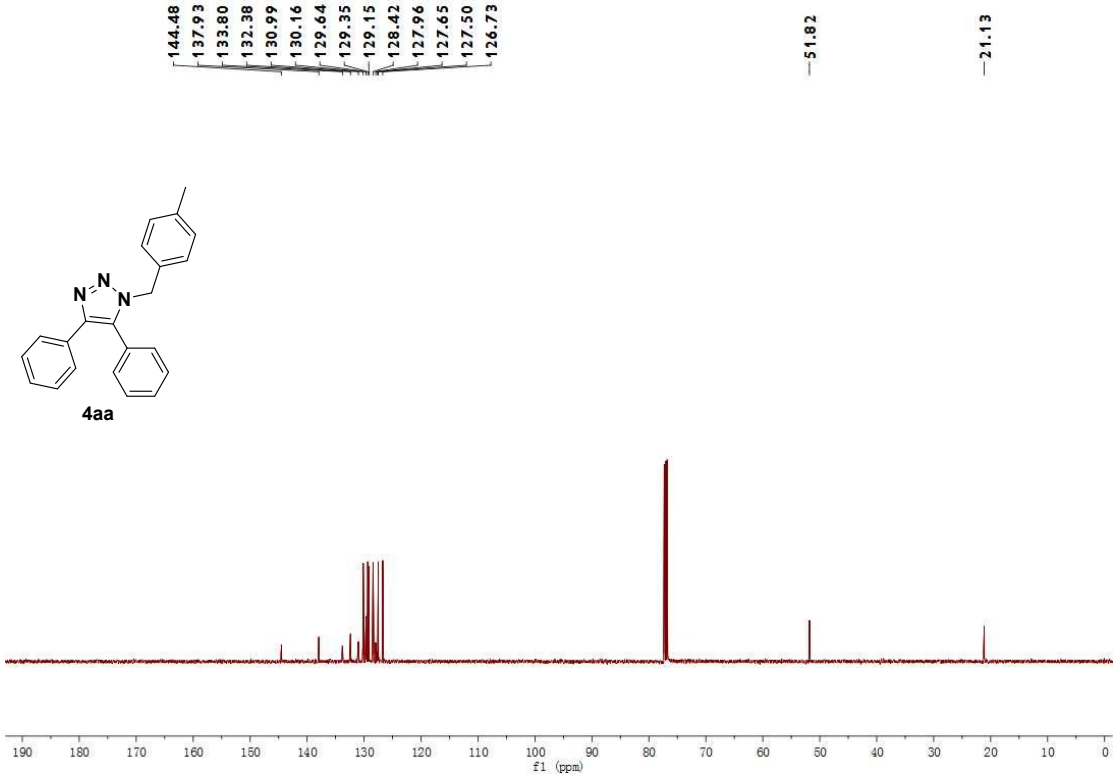
4aa
1.96 1.00 2.00 3.09 1.93 1.98 1.99 2.00 3.00
f1 (ppm)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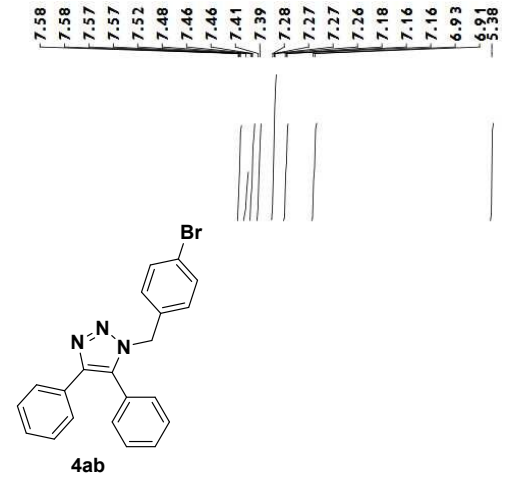
4aa
f1 (ppm)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7.18
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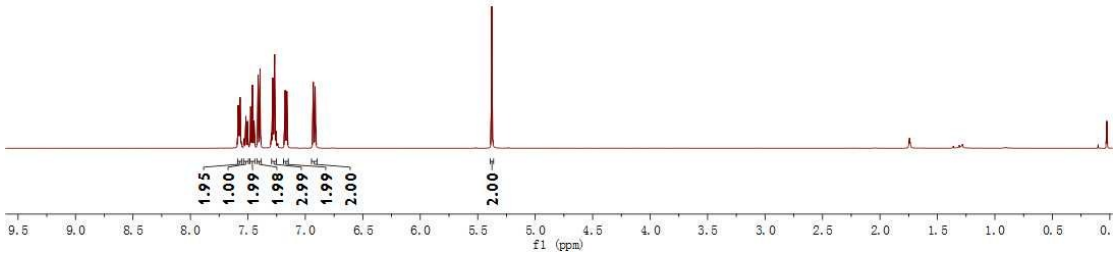
7.16
6.93
6.91
5.38
Br
N
N
N

4ab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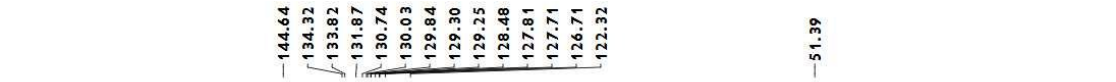
2.00
f1 (ppm)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127.81
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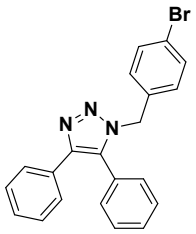
126.71
122.32
51.39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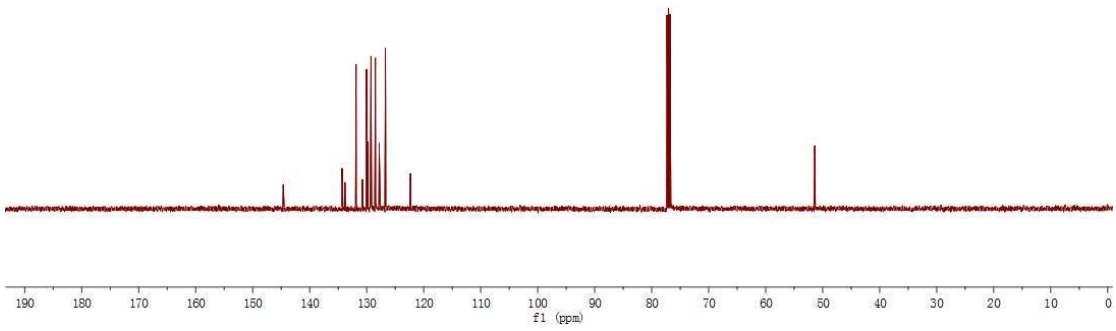
N
N
N

4ab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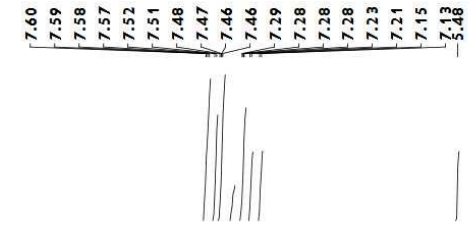
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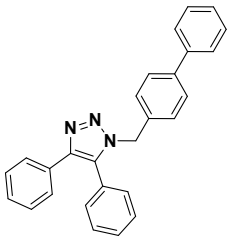
7.15
7.13
5.48



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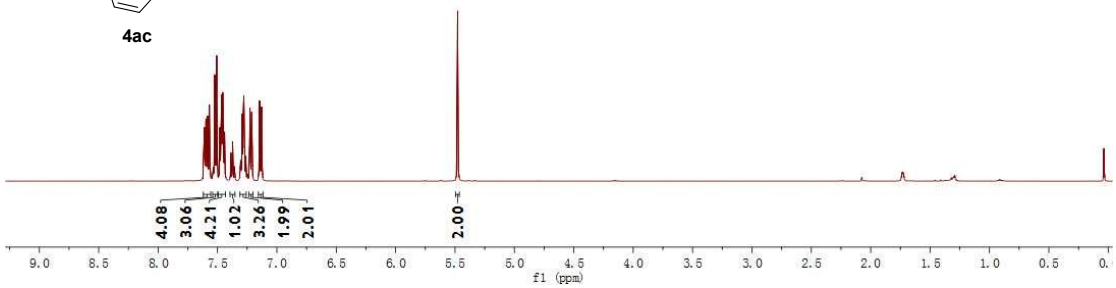
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1.02
3.26
1.99
2.01
2.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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5.0
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4.0
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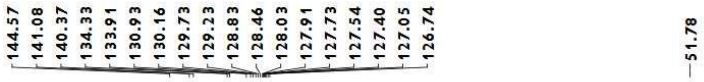
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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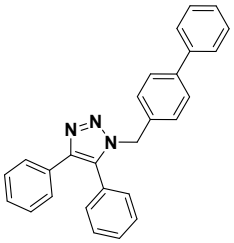
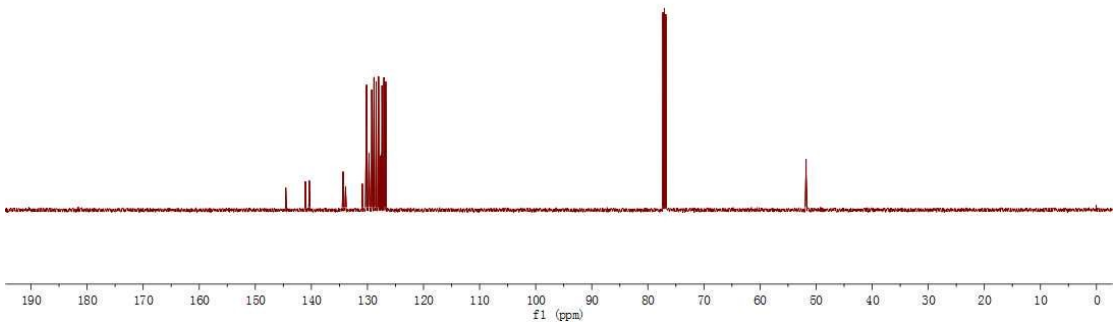
127.54
127.40
127.05
126.74
51.78



4ac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10
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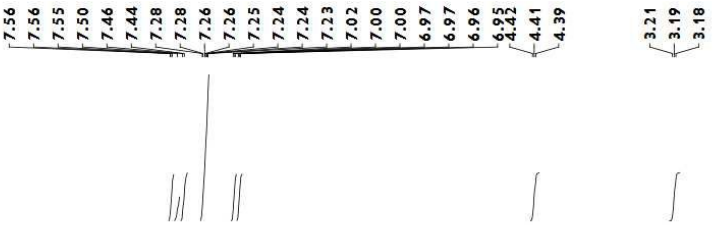
f1 (ppm)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7.23
7.02
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6.97
6.96
6.95
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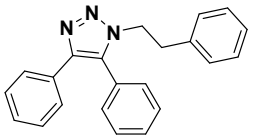
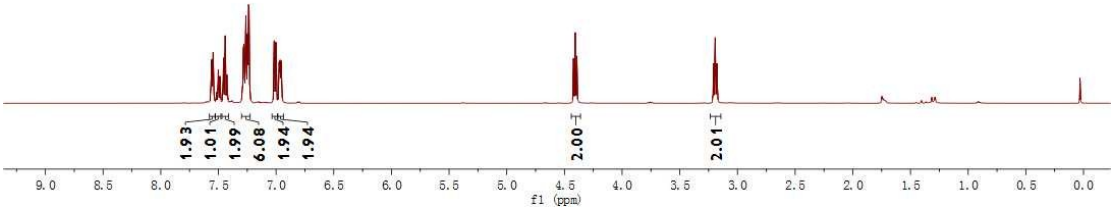
3.21
3.19
3.18



4ad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8.0
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6.5
6.0
5.5
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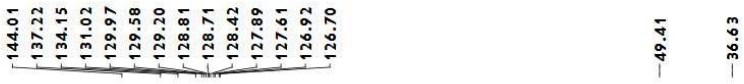
f1 (ppm)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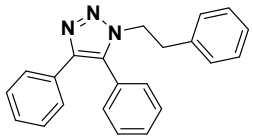
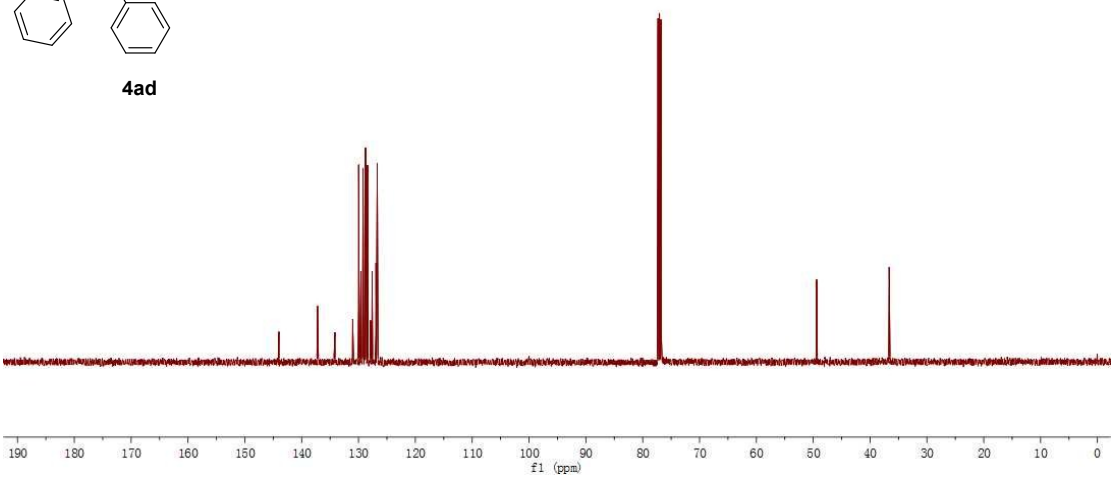
49.41
36.63



4ad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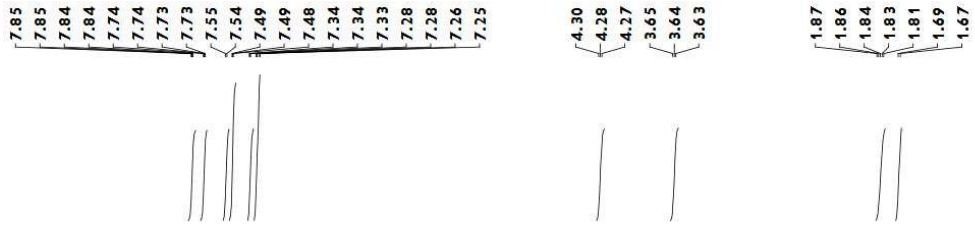
f1 (ppm)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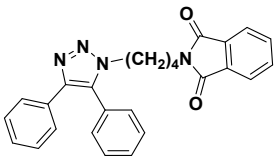
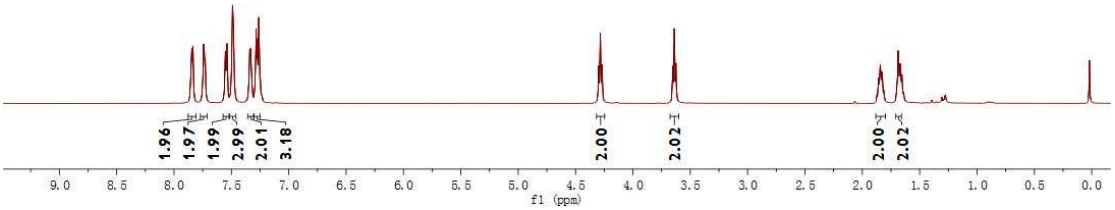
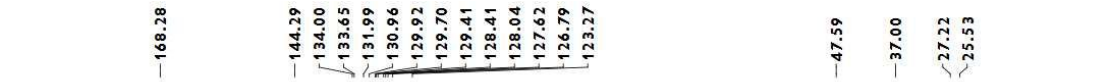
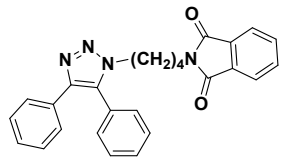
1.81
1.69
1.67


O
N
N
N
(CH2)4N
O

4ae

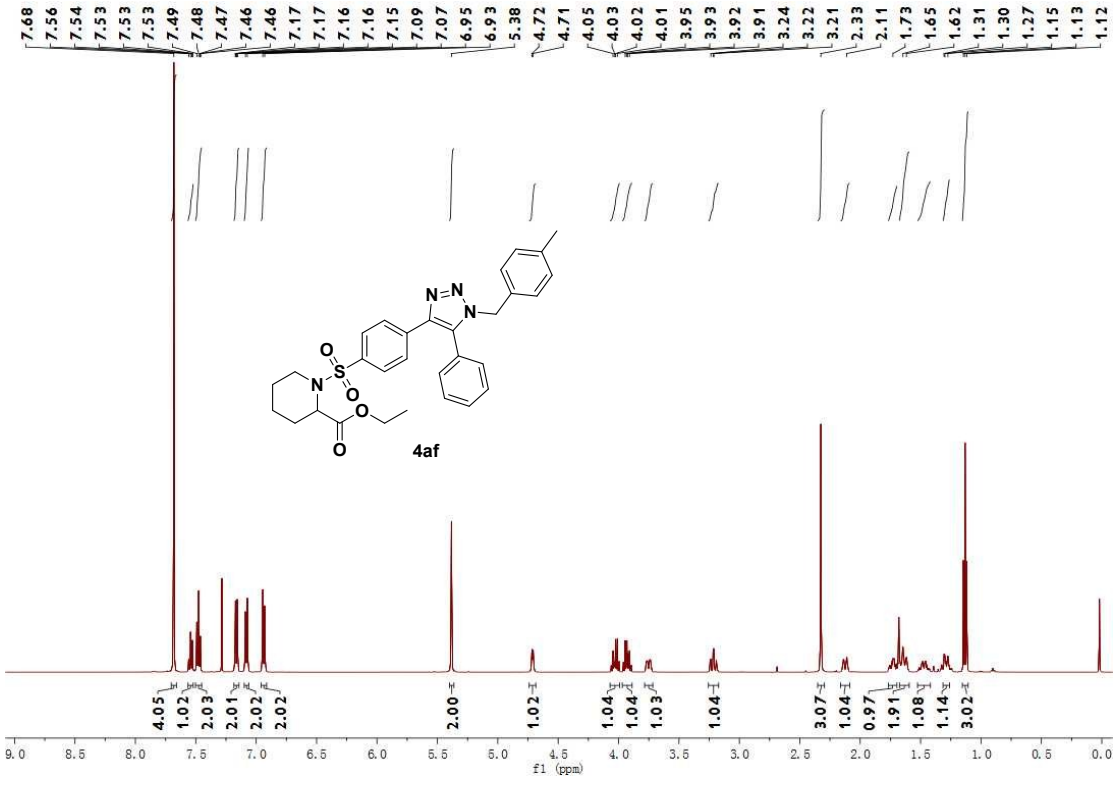

1.96
1.97
1.99
2.99
2.01
3.18
2.00
2.02
2.00
2.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

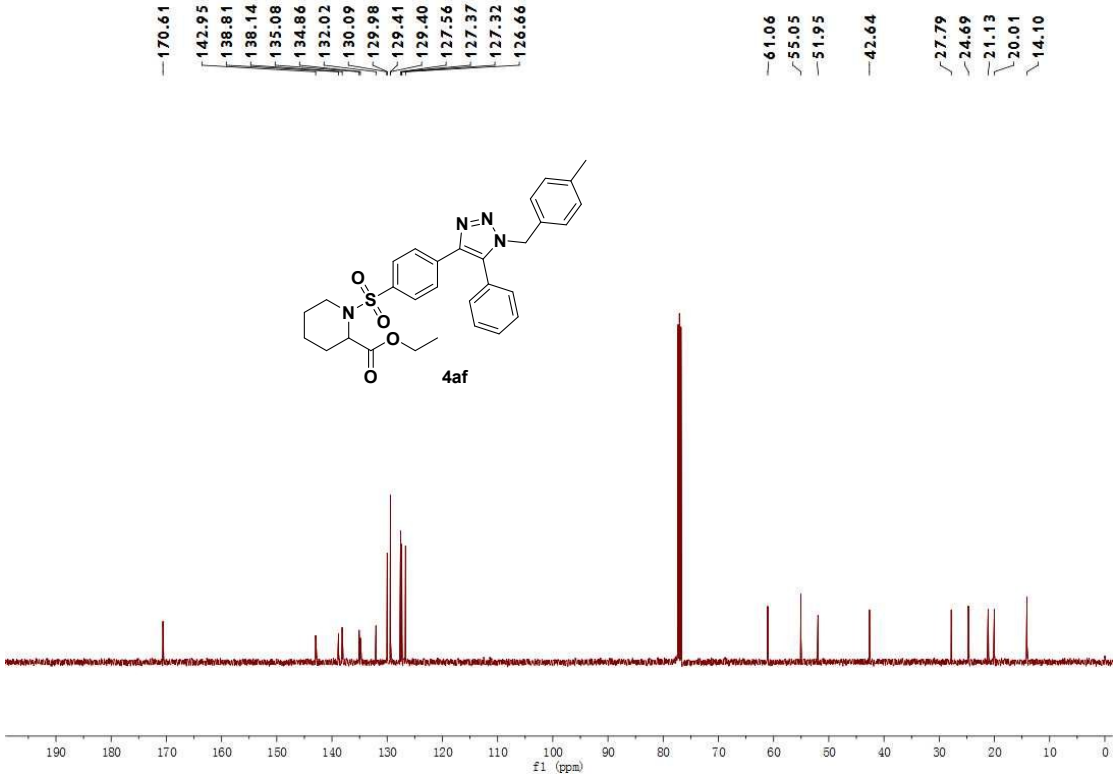

168.28
144.29
134.00
133.65
131.99
130.96
129.92
129.70
129.41
128.41
128.04
127.62
126.79
123.27
47.59
37.00
27.22
25.53


O
N
N
N
(CH2)4N
O

4ae

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.68 7.56 7.54 7.53 7.53 7.53 7.49 7.48 7.47 7.46 7.46 7.17 7.17 7.16 7.16 7.15 7.09 7.07 6.95 6.93 5.38 4.72 4.71 4.05 4.03 4.02 4.01 3.95 3.93 3.92 3.91 3.24 3.22 3.21 2.33 2.11 1.73 1.65 1.62 1.31 1.30 1.27 1.15 1.13 1.12
4af
4.05 1.02 2.03 2.01 2.02 2.02 2.00 1.02 1.04 1.04 1.03 1.04 3.07 1.04 0.97 1.91 1.08 1.14 3.02
f1 (ppm)


170.61 142.95 138.81 138.14 135.08 134.86 132.02 130.09 129.98 129.41 129.40 127.56 127.37 127.32 126.66 61.06 55.05 51.95 42.64 27.79 24.69 21.13 20.01 14.10
4af
f1 (ppm)