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Synthesis of new surfactant-like triazine-functionalized ligands for Pd-catalyzed Heck and Sonogashira reactions in water

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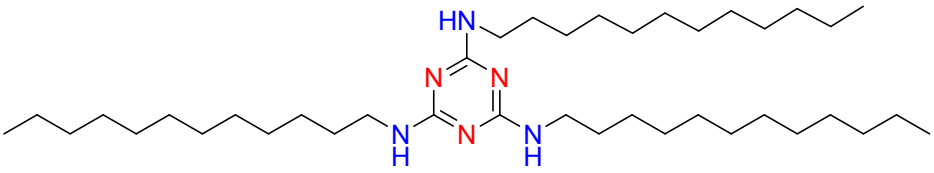
Outline

1. **Synthesis and characterization of Ligands**
2. **General procedure for the Heck reaction using the TDTAT ligand**
3. **General procedure for the Sonogashira reaction using the TDTAT ligand**
4. **General procedure for the large-scale Heck reaction using the TDTAT ligand**
5. **Spectral data for synthesized compounds**
6. **References for synthesized compounds**
7. **Copies of ^{1}HNMR and ^{13}CNMR for all synthesized compounds**

1. Synthesis and characterization of Ligands

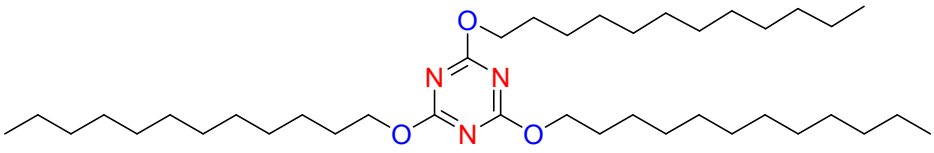
1.1 Synthesis of N^2,N^4,N^6-Tridodecyl-1,3,5-triazine-2,4,6-triamine (3a) [1]



3a

Into a canonical flask (50 mL), a mixture of dodecan-1-amine (3.1 mmol), TCT (1 mmol), and KOH (3 mmol) were stirred in THF (10 mL) at room temperature overnight. After completion of the reaction, as indicated by TLC, the mixture was filtered and washed exhaustively with CH_2Cl_2. The solvent was removed off under a vacuum. Recrystallization from ethanol gave the product. ^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.85-0.88 (m, 9H), 1.25 (brs, 54H), 1.47-1.64 (m, 6H), 3.31-3.43 (m, 6H), 5.39 (t, J = 50 Hz, 3H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 14.1, 22.7, 26.8, 29.3, 29.6, 31.9, 40.9, 163.7. Anal. Calcd. for $C_{39}H_{78}N_6$: C, 74.23; H, 12.46; N, 13.32. Found: C, 74.10; H, 12.41; N, 13.24.

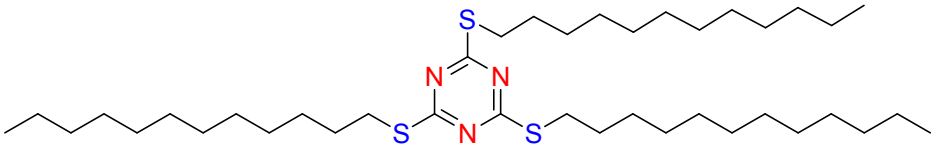
1.2 Synthesis of 2,4,6-tris(dodecyloxy)-1,3,5-triazine (3b)



3b

Into a canonical flask (50 mL), a mixture of dodecan-1-ol (3.1 mmol), TCT (1 mmol), and KOH (3 mmol) were stirred in THF (10 mL) at room temperature overnight. After completion of the reaction, as indicated by TLC, the mixture was filtered and washed exhaustively with CH_2Cl_2. The solvent was removed off under a vacuum. Recrystallization from ethanol gave the product. ^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.85-0.90 (m, 9H), 1.25 (brs, 54H), 1.72-1.83 (m, 6H), 4.37 (t, J = 7.5 Hz, 6H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 14.1, 22.6, 25.8, 28.6, 29.3, 29.6, 31.9, 68.5, 172.8. Anal. Calcd. for $C_{39}H_{75}N_3O_3$: C, 73.88; H, 11.92; N, 6.63. Found: C, 73.75; H, 11.81; N, 6.54.

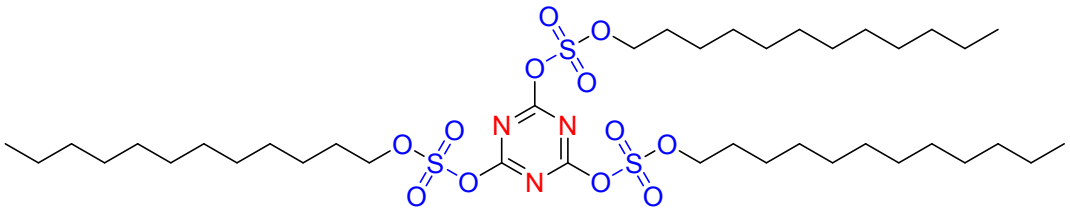
1.3 Synthesis of 2,4,6-tris(dodecylthio)-1,3,5-triazine (3c)



3c

Into a canonical flask (50 mL), a mixture of dodecane-1-thiol (3.1 mmol), TCT (1 mmol), and KOH (3 mmol) were stirred in THF (10 mL) at room temperature overnight. After completion of the reaction, as indicated by TLC, the mixture was filtered and washed exhaustively with CH_2Cl_2. The solvent was removed off under a vacuum. Recrystallization from ethanol gave the product. ^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.84-0.89 (m, 9H), 1.25 (brs, 54H), 1.73-1.82 (m, 6H), 3.07 (t, *J* = 7.5 Hz, 6H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 14.1, 22.6, 28.6, 29.3, 29.6, 31.8, 33.5, 172.7. Anal. Calcd. for $C_{39}H_{75}N_3S_3$: C, 68.66; H, 11.08; N, 6.16; S, 14.10. Found: C, 68.60; H, 11.0; N, 6.09; S, 13.97.

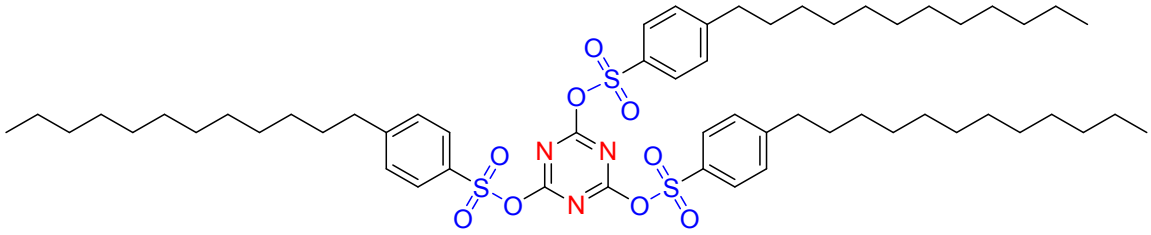
1.4 Synthesis of tridodecyl 1,3,5-triazine-2,4,6-triyl tris(sulfate) (3d)



3d

Into a canonical flask (50 mL), a mixture of sodium dodecane-1-sulfate (3.1 mmol), and TCT (1 mmol) were stirred in THF (10 mL) at room temperature overnight. After completion of the reaction, as indicated by TLC, the mixture was filtered and washed exhaustively with CH_2Cl_2. The solvent was removed off under a vacuum. Recrystallization from ethanol gave the product. ^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.85-0.89 (m, 9H), 1.25 (brs, 54H), 1.60-1.71 (m, 6H), 3.75 (t, *J* = 7.5 Hz, 6H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 14.1, 22.7, 22.9, 25.9, 28.7, 29.3, 29.6, 31.9, 70.2, 174.9. Anal. Calcd. for $C_{39}H_{75}N_3O_{12}S_3$: C, 53.58; H, 8.65; N, 4.81; S, 11.00. Found: C, 53.50; H, 8.53; N, 4.75; S, 10.90.

1.5 Synthesis of 1,3,5-triazine-2,4,6-triyl tris(4-dodecylbenzenesulfonate) (3e)



3e

Into a canonical flask (50 mL), a mixture of sodium 4-dodecylbenzenesulfonate (3.1 mmol), TCT (1 mmol) were stirred in THF (10 mL) at room temperature overnight. After completion of the reaction, as indicated by TLC, the mixture was filtered and washed exhaustively with CH_2Cl_2. The solvent was removed off under a vacuum. Recrystallization from ethanol gave the product. ^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.85-0.88 (m, 9H), 1.25 (brs, 54H), 1.57-1.68 (m, 6H), 2.71-2.76 (t, *J* = 7.5 Hz, 6H), 7.35 (d, *J* = 7.5 Hz, 6H), 7.86 (d, *J* = 7.5 Hz, 6H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 14.1, 22.7, 29.3, 29.7, 30.1, 31.8, 36.8, 129.8, 131.1, 142.4, 142.7, 163.3. Anal. Calcd. for $C_{57}H_{87}N_3O_9S_3$: C, 64.92; H, 8.32; N, 3.98; S, 9.12. Found: C, 64.85; H, 8.26; N, 3.91; S, 9.03.

2. General procedure for the Heck reaction using the TDTAT ligand

A reaction tube equipped with a magnetic stirring bar was charged with aryl halide (1.0 mmol), terminal alkene (1.2 mmol), K_2CO_3 (2 mmol), $PdCl_2$ (1.5 mol%) and TDTAT/water (3.0 mL, 3 wt.% TDTAT). The mixture was heated in an oil bath at 80 °C. After completion of the reaction monitored by GC or TLC analysis, the reaction mixture was cooled down to room temperature. The organic compound was extracted with ethyl acetate (3 x 5 mL) from the aqueous layer and dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuum. The organic mixture was then purified by silica gel column chromatography using *n*-hexane /ethyl acetate as eluent to obtain the corresponding pure coupled product. The recovered catalyst was thoroughly washed with ethyl acetate and was dried under nitrogen flow, which was subsequently reused.

3. General procedure for the Sonogashira reaction using the TDTAT ligand

Into a conical flask (10 mL) a mixture of aryl halide (1 mmol), terminal alkyne (1.1 mmol), K_2CO_3 (2 mmol), $PdCl_2$ (1.5 mol%) and TDTAT/water (3.0 mL, 3 wt.% TDTAT) were stirred at 80 °C. The reactions were monitored by TLC. Stirring was continued until the consumption of the starting materials based on reaction time in Table 5. After

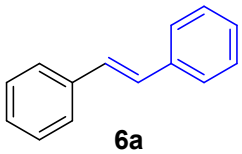
completion of the reaction, the mixture was cooled down to room temperature. The organic compound was extracted with ethyl acetate (3 x 5 mL) from the aqueous layer and dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuum. The organic mixture was then purified by silica gel column chromatography employing *n*-hexane/ethyl acetate as the eluent, affording the pure corresponding product. The recovered catalyst was thoroughly washed with ethyl acetate and was dried under nitrogen flow, which was subsequently reused.

4. General procedure for the large scale Heck reaction using the TDTAT ligand

A reaction tube equipped with a magnetic stirring bar was charged with aryl halide (10.0 mmol), terminal alkene (11 mmol), K_2CO_3 (10 mmol), $PdCl_2$ (10 mol%) and TDTAT/water (30 mL, 30 wt.% TDTAT). The mixture was heated in an oil bath at 80 °C. After completion of the reaction monitored by GC or TLC analysis, the reaction mixture was cooled down to room temperature. The organic compound was extracted with ethyl acetate from the aqueous layer and dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuum. The organic mixture was then purified by silica gel column chromatography using *n*-hexane /ethyl acetate as eluent to obtain the corresponding pure coupled product.

5. Spectral data for synthesized compounds

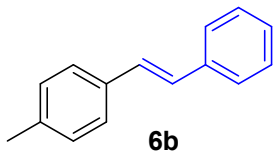
5.1. (*E*)-1,2-diphenylethene (6a)



6a

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 7.13 (s, 2H), 7.25-7.41 (m, 6H), 7.52-7.56 (m, 4H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 126.6, 127.7, 128.7, 137.4.

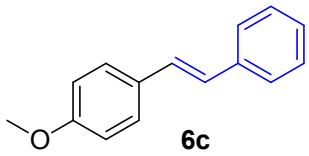
5.2. (E)-1-methyl-4-styrylbenzene (6b)



6b

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 2.27 (s, 3H), 6.98 (s, 2H), 7.02-7.18 (m, 3H), 7.23-7.29 (m, 2H), 7.32 (d, *J* = 7.5 Hz, 2H), 7.40-7.43 (m, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 21.3, 126.4, 126.5, 127.4, 127.7, 128.6, 128.7, 129.4, 134.6, 137.5.

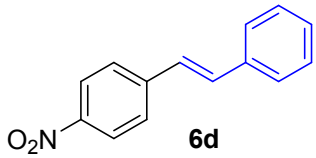
5.3. (E)-1-methoxy-4-styrylbenzene (6c)



6c

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 3.71 (s, 3H), 6.78-6.82 (m, 2H), 6.87 (d, *J* = 15 Hz, 1H), 6.98 (d, *J* = 15 Hz, 1H), 7.11-7.28 (m, 3H), 7.33-7.41 (m, 4H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 55.6, 114.2, 126.3, 126.6, 127.2, 127.8, 128.2, 128.7, 137.7, 159.2.

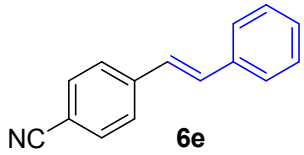
5.4. (E)-1-nitro-4-styrylbenzene (6d)



6d

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 7.06 (d, *J* = 15 Hz, 1H), 7.20 (d, J = 15 Hz, 1H), 7.25-7.36 (m, 3H), 7.45-7.50 (m, 2H), 7.54 (d, *J* = 7.5 Hz, 2H), 8.14 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 124.1, 126.3, 126.8, 127.0, 128.8, 128.9, 133.3, 136.2, 143.8, 146.7.

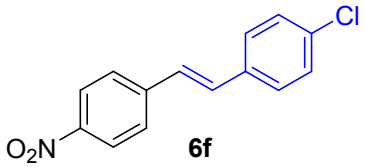
5.5. (E)-4-styrylbenzonitrile (6e)



6e

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 7.09 (d, *J* = 15 Hz, 1H), 7.22 (d, *J* = 15 Hz, 1H), 7.32-7.43 (m, 3H), 7.52-7.66 (m, 6H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 110.5, 119.0, 126.7, 126.9, 128.6, 128.8, 130.5, 132.4, 132.5, 134.2, 136.2.

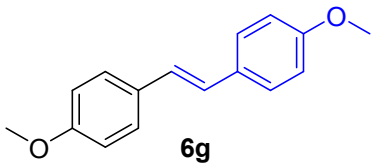
5.6. (E)-1-chloro-4-(4-nitrostyryl)benzene (6f)



6f

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 7.00-7.58 (m, 8H), 8.15 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 124.2, 126.8, 126.9, 128.2, 129.1, 134.5, 134.7, 143.4, 147.0.

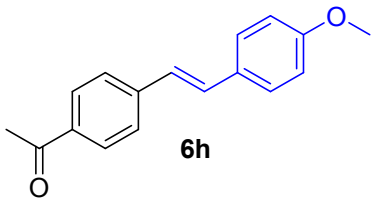
5.7. (E)-1,2-bis(4-methoxyphenyl)ethene (6g)



6g

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 3.75 (s, 6H), 6.80-6.86 (m, 6H), 7.35 (d, *J* = 10 Hz, 4H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 55.3, 114.1, 126.1, 127.0, 127.4, 159.7.

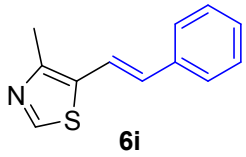
5.8. (E)-1-(4-(4-methoxystyryl)phenyl)ethan-1-one (6h)



6h

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 2.61 (s, 3H), 3.84 (s, 3H), 6.92 (d, *J* = 10 Hz, 2H), 6.99 (d, *J* = 17.5 Hz, 1H), 7.19 (d, *J* = 17.5, 1H), 7.48 (d, J = 7.5 Hz, 2H), 7.56 (d, *J* = 10 Hz, 2H), 7.94 (d, *J* = 10 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 27.3, 55.3, 114.2, 126.2, 126.8, 127.5, 128.1, 128.9, 137.2, 138.2, 160.1, 197.3.

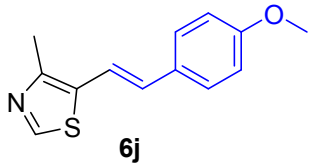
5.9. (E)-4-methyl-5-styrylthiazole (6i)



6i

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 2.54 (s, 3H), 6.82 (d, *J* = 15 Hz, 1H), 7.18 (d, *J* = 15 Hz, 1H), 7.24- 7.39 (m, 3H), 7.47 (d, *J* = 7.5 Hz, 2H), 8.56 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 16.1, 127.6, 129.5, 129.8, 130.3, 135.5, 138.4, 149.8, 150.7.

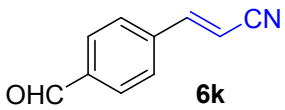
5.10. (E)-5-(4-methoxystyryl)-4-methylthiazole (6j)



6j

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 2.51 (s, 3H), 3.81 (s, 3H), 6.76 (d, *J* = 15 Hz, 1H), 6.88 (d, *J* = 7.5 Hz, 2H), 7.02 (d, *J* = 15 Hz, 1H), 7.40 (d, *J* = 7.5 Hz, 2H), 8.52 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 15.4, 55.3, 114.2, 127.6, 129.5, 130.1, 130.9, 138.0, 148.9, 149.8, 159.1.

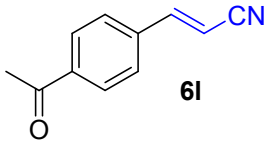
5.11. (E)-3-(4-formylphenyl)acrylonitrile (6k)



6k

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 6.03 (d, *J* = 15 Hz, 1H), 7.45 (d, *J* = 17.5 Hz, 1H), 7.62 (d, *J* = 7.5 Hz, 2H), 7.93 (d, *J* = 7.5 Hz, 2H), 10.05 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 99.7, 117.5, 127.9, 130.3, 133.4, 144.9, 148.9, 191.2.

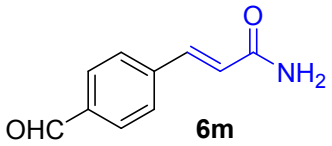
5.12. (E)-3-(4-acetylphenyl)acrylonitrile (6l)



6l

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 2.62 (s, 3H), 7.44 (d, *J* = 15 Hz, 1H), 7.55 (d, *J* = 10 Hz, 2H), 7.99 (d, *J* = 10 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 26.7, 99.0, 117.5, 127.5, 129.0, 137.5, 138.7, 149.1, 197.0.

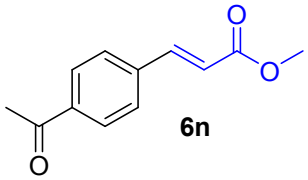
5.13. (E)-3-(4-formylphenyl)acrylamide (6m)



6m

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 5.57 (s, 2H), 6.57 (d, *J* = 17.5 Hz, 1H), 7.66-7.73 (m, 3H), 7.90 (d, *J* = 7.5 Hz, 2H), 10.03 (s, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 120.1, 128.0, 130.2, 134.2, 141.2, 143.3, 172.5, 190.2.

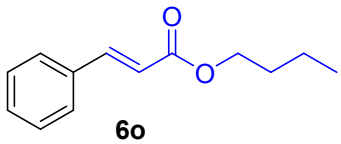
5.14. methyl (E)-3-(4-acetylphenyl)acrylate (6n)



6n

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 2.61 (s, 3H), 3.82 (s, 3H), 6.52 (d, *J* = 17.5 Hz, 1H), 7.60 (d, *J* = 7.5 Hz, 2H), 7.71 (d, *J* = 15 Hz, 1H), 7.97 (d, *J* = 10 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 26.7, 51.9, 120.3, 128.1, 128.8, 138.0, 138.6, 143.3, 166.9, 197.3.

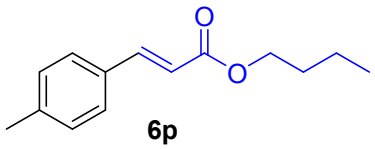
5.15. n-butyl cinnamate (6o)



6o

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.86 (t, *J* = 7.5 Hz, 3H), 1.26-1.40 (m, 2H), 1.53-1.64 (m, 2H), 4.10 (t, *J* = 7.5 Hz, 2H), 6.34 (d, *J* = 15 Hz, 1H), 7.24- 7.27 (m, 3H), 7.38-7.42 (m, 2H), 7.57 (d, *J* = 15 Hz, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 12.8, 18.2, 29.7, 63.4, 117.2, 126.9, 127.8, 129.2, 133.4, 143.5, 166.1.

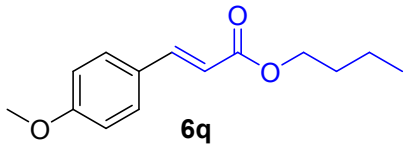
5.16. (E)-butyl 3-(p-tolyl)acrylate (6p)



6p

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.96 (t, *J* = 7.5 Hz, 3H), 1.36-1.51 (m, 2H), 1.63-1.74 (m, 2H), 2.36 (s, 3H), 4.14 (d, *J* = 7.5 Hz, 2H), 6.39 (d, *J* = 15 Hz, 1H), 7.17 (d, *J* = 7.5 Hz, 2H), 7.41 (d, *J* = 7.5 Hz, 2H), 7.66 (d, *J* = 15 Hz, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 13.8, 19.2, 21.4, 30.8, 64.3, 117.1, 128.0, 129.6, 131.7, 140.5, 144.5, 167.2.

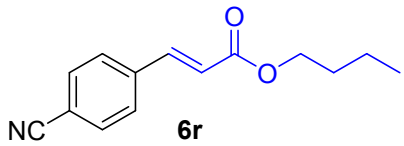
5.17. (E)-butyl 3-(4-methoxyphenyl)acrylate (6q)



6q

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.89 (t, *J* = 7.5 Hz, 3H), 1.29-1.44 (m, 2H), 1.56-1.67 (m, 2H), 3.78 (s, 3H), 4.13 (t, *J* = 7.5 Hz, 2H), 6.24 (d, *J* = 15 Hz, 1H), 6.83 (d, *J* = 7.5 Hz, 2H), 7.40 (d, *J* = 7.5 Hz, 2H), 7.57 (d, *J* = 15 Hz, 1H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 13.7, 19.2, 30.8, 55.3, 64.2, 114.3, 115.8, 129.7, 144.2, 161.3, 167.4.

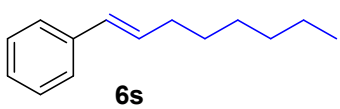
5.18. (E)-butyl 3-(4-cyanophenyl)acrylate (6r)



6r

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.97 (t, *J* = 7.5 Hz, 3H), 1.37-1.51 (m, 2H), 1.64-1.75 (m, 2H), 4.23 (t, *J* = 7.5 Hz, 2H), 6. 53 (d, *J* = 17.5 Hz, 1H), 7.61-7.69 (m, 5H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 13.7, 19.1, 30.7, 64.7, 113.3, 118.3, 121.8, 128.3, 132.6, 138.7, 142.0, 166.2.

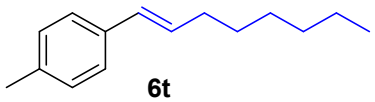
5.19. (E)-oct-1-en-1-ylbenzene (6s)



6s

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.88-0.91 (m, 3H), 1.30-1.48 (m, 8H), 2.16-2.24 (m, 2H), 6.17-6.40 (m, 2H), 7.16-7.44 (m, 5H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 14.1, 22.6, 28.9, 29.4, 31.4, 31.8, 125.9, 127.2, 128.2, 128.4, 129.7, 138.0.

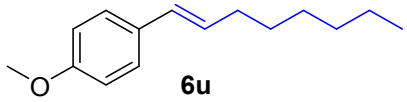
5.20. (E)-1-methyl-4-(oct-1-en-1-yl)benzene (6t)



6t

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.91-0.95 (m, 3H), 1.24-1.49 (m, 8H), 2.18-2.27 (m, 2H), 2.35 (s, 3H), 6.14-6.26 (m, 1H), 6.38 (d, *J* = 15 Hz, 1H), 7.11-7.36 (m, 4H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 14.2, 21.0, 22.6, 28.9, 29.4, 31.4, 31.7, 125.8, 128.2, 128.3, 129.5, 135.2, 137.2.

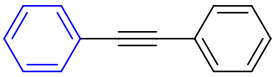
5.21. (E)-1-methoxy-4-(oct-1-en-1-yl)benzene (6u)



6u

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 0.79-0.82 (m, 3H), 1.11-1.39 (m, 8H), 2.06-2.14 (m, 2H), 3.71 (s, 3H), 5.95-6.05 (m, 1H), 6.23 (d, *J* = 15 Hz, 1H), 6.74 (d, *J* = 7.5 Hz, 2H), 7.20 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 14.2, 22.7, 29.0, 29.5, 31.5, 31.8, 55.2, 113.9, 126.6, 128.4, 128.6, 130.8, 158.6.

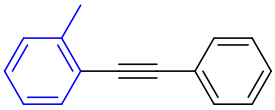
5.22. 1,2-diphenylethyne (8a)



8a

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 7.23-7.31 (m, 6H), 7.42-7.49 (m, 4H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 89.4, 123.3, 128.3, 128.4, 131.6.

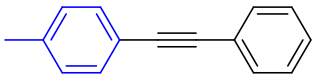
5.23. 1-methyl-2-(phenylethynyl)benzene (8b)



8b

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 2.44 (s, 3H), 7.10-7.17 (m, 3H), 7.25-7.28 (m, 3H), 7.41-7.48 (m, 3H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 20.7, 82.0, 88.3, 122.8, 123.5, 125.6, 128.2, 128.3, 128.4, 129.5, 131.5, 131.8, 140.2.

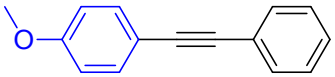
5.24. 1-methyl-4-(phenylethynyl)benzene (8c)



8c

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 2.28 (s, 3H), 8.06 (d, *J* = 7.5 Hz, 2H), 7.23-7.29 (m, 3H), 7.34 (d, *J* = 7.5 Hz, 2H), 7.41-7.46 (m, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 21.5, 88.5, 89.5, 118.0, 123.5, 128.0, 128.3, 129.1, 131.5, 132.5, 138.4.

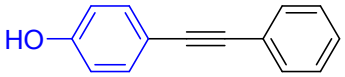
5.25. 1-methoxy-4-(phenylethynyl)benzene (8d)



8d

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 3.80 (s, 3H), 6.82-6.88 (m, 2H), 7.28-7.34 (m, 3H), 7.43-7.53 (m, 4H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 55.6, 88.2, 89.6, 114.2, 115.4, 123.5, 128.1, 128.3, 131.5, 133.1, 159.9.

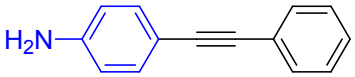
5.26. 4-(phenylethynyl)phenol (8e)



8e

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 5.04 (s, 1H), 6.73 (d, *J* = 7.5 Hz, 2H), 7.18-7.45 (m, 7H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 88.0, 89.9, 114.2, 116.4, 123.4, 128.5, 128.7, 131.5, 132.9, 158.4.

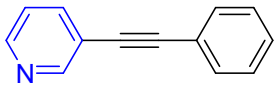
5.27. 4-(phenylethynyl)aniline (8f)



8f

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 4.18 (s, 2H), 6.63 (d, *J* = 7.5 Hz, 2H), 7.26-7.33 (m, 5H), 7.86 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 88.5, 89.4, 110.7, 114.4, 123.4, 128.4, 128.6, 131.4, 131.9, 148.0.

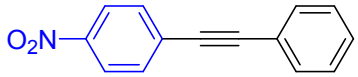
5.28. 3-(phenylethynyl)pyridine (8g)



8g

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 7.16-7.29 (m, 4H), 7.44-7.48 (m, 2H), 7.69-7.74 (m, 1H), 8.44-8.47 (m, 1H), 8.69 (s,1H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 85.9, 92.7, 120.7, 122.5, 123.1, 128.4, 128.8, 131.7, 138.0, 148.4, 152.1.

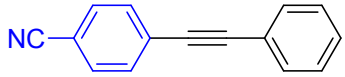
5.29. 1-nitro-4-(phenylethynyl)benzene (8h)



8h

^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 7.36-7.42 (m, 3H), 7.54-7.58 (m, 2H), 7.63-7.69 (m, 2H), 8.19-8.24 (m, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 87.4, 94.5, 122.4, 123.6, 128.5, 129.3, 130.2, 131.8, 132.3, 146.3.

5.30. 4-(phenylethynyl)benzonitrile (8i)



8i

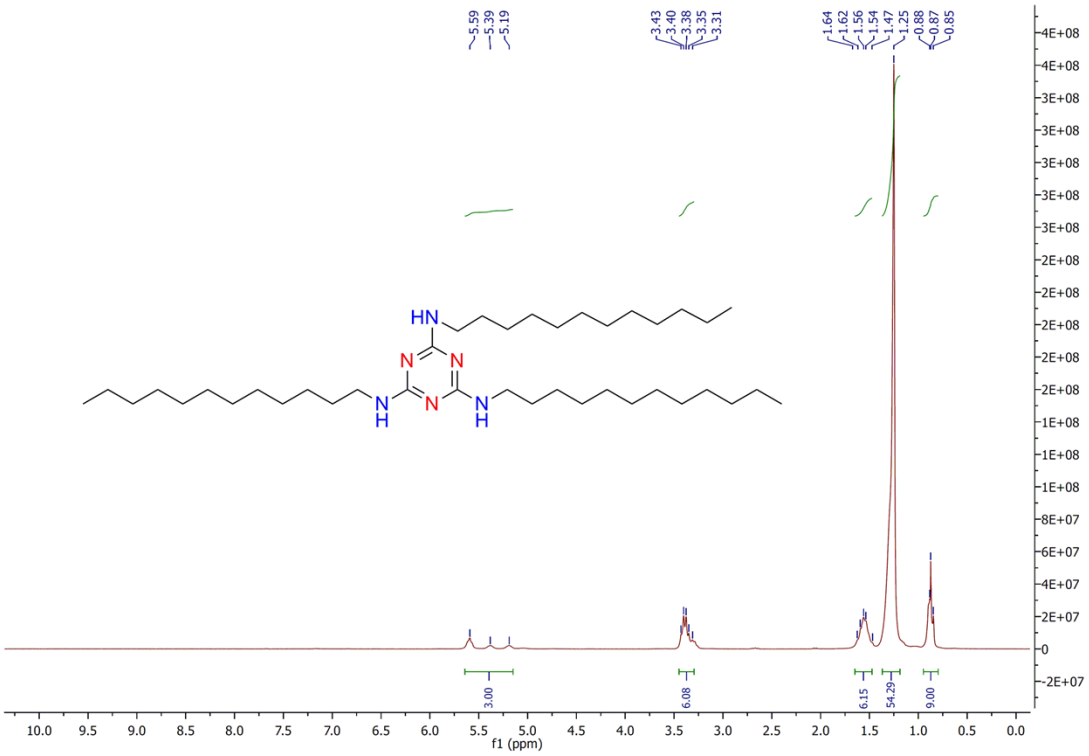
^{1}H NMR (250 MHz, $CDCl_3$): δ (ppm) = 7.23-7.32 (m, 3H), 7.55-7.58 (m, 4H), 7.74 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR (62.5 MHz, $CDCl_3$): 88.3, 94.9, 110.0, 119.1, 123.4, 123.5, 128.1, 128.4, 131.5, 132.5, 134.7.

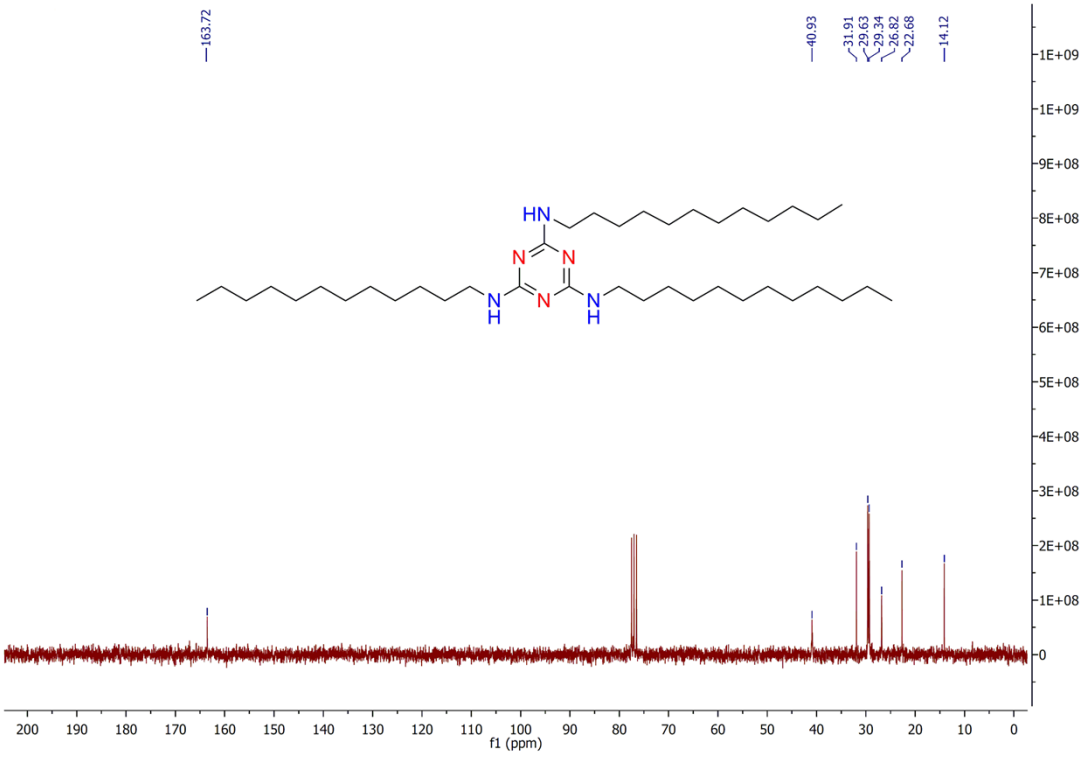
6. References for synthesized compounds

1. X.-J. Li, J.-L. Zhang, Y. Geng, Z. Jin, *J. Org. Chem.*, **2013**, 78, 5078.

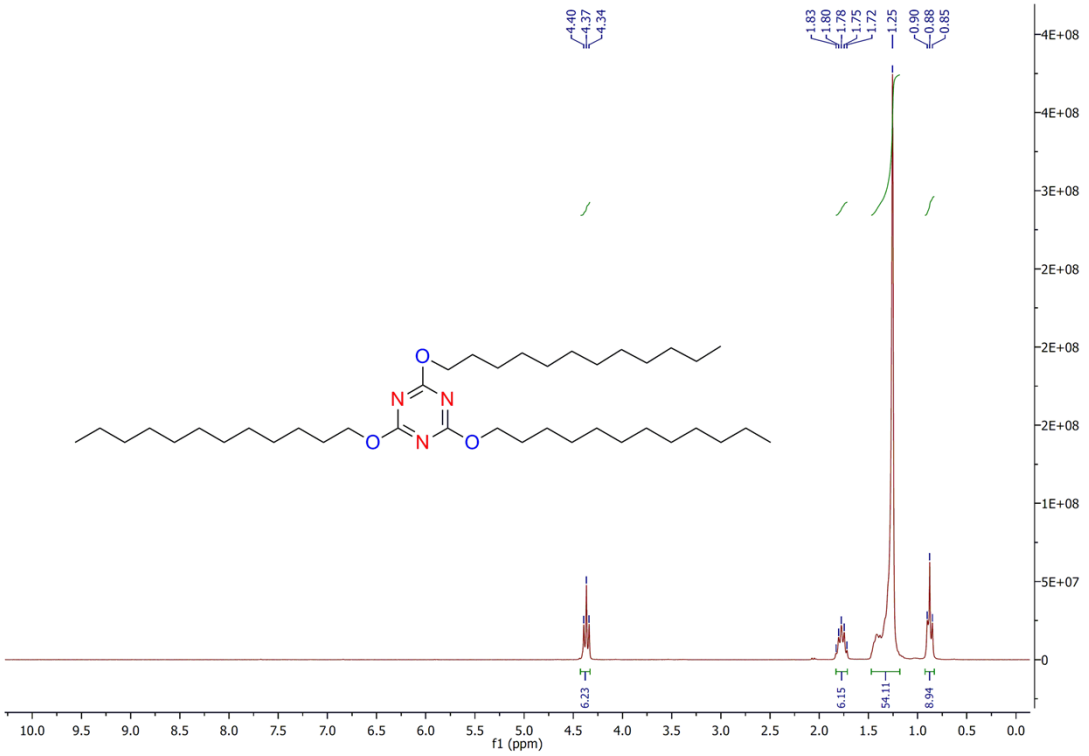
7. Copies of ^{1}H, ^{13}C NMR for all synthesized compounds

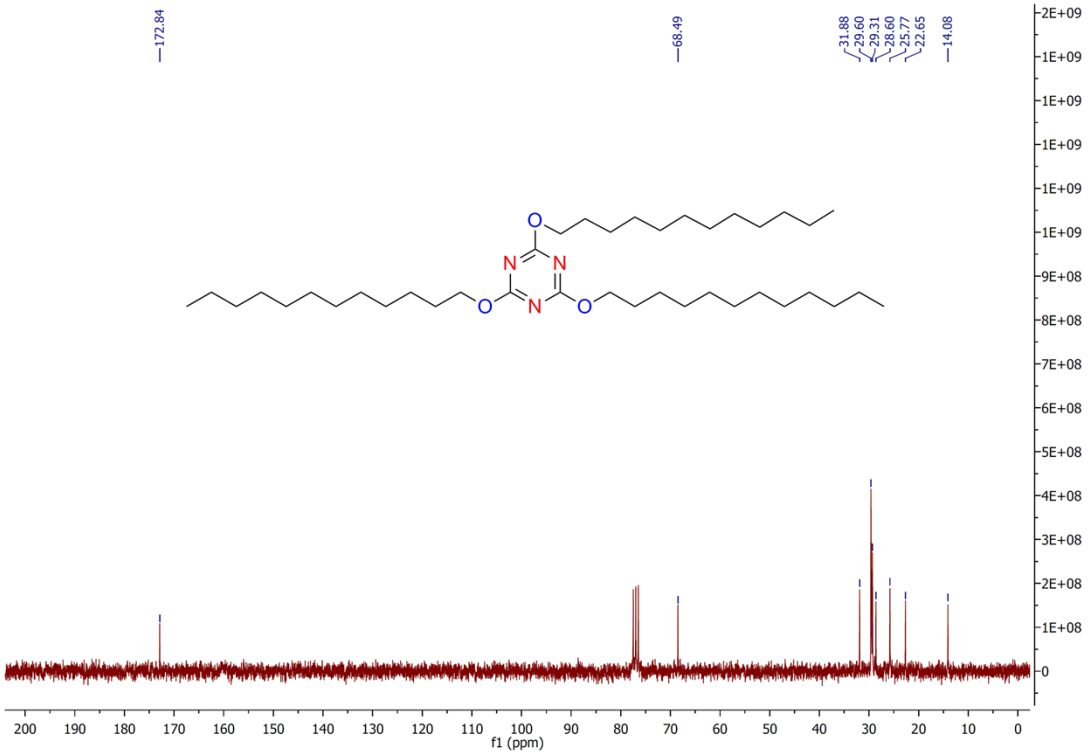
7.1. *N*^{2},*N*^{4},*N*^{6}-Tridodecyl-1,3,5-triazine-2,4,6-triamine (3a)



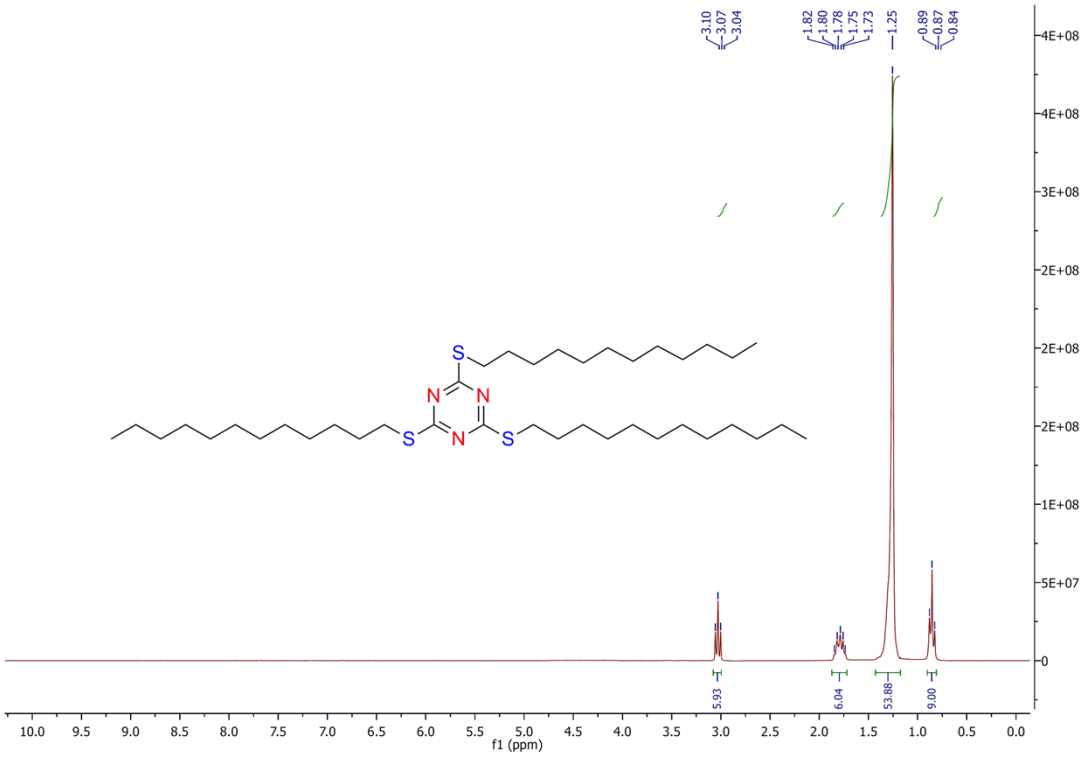


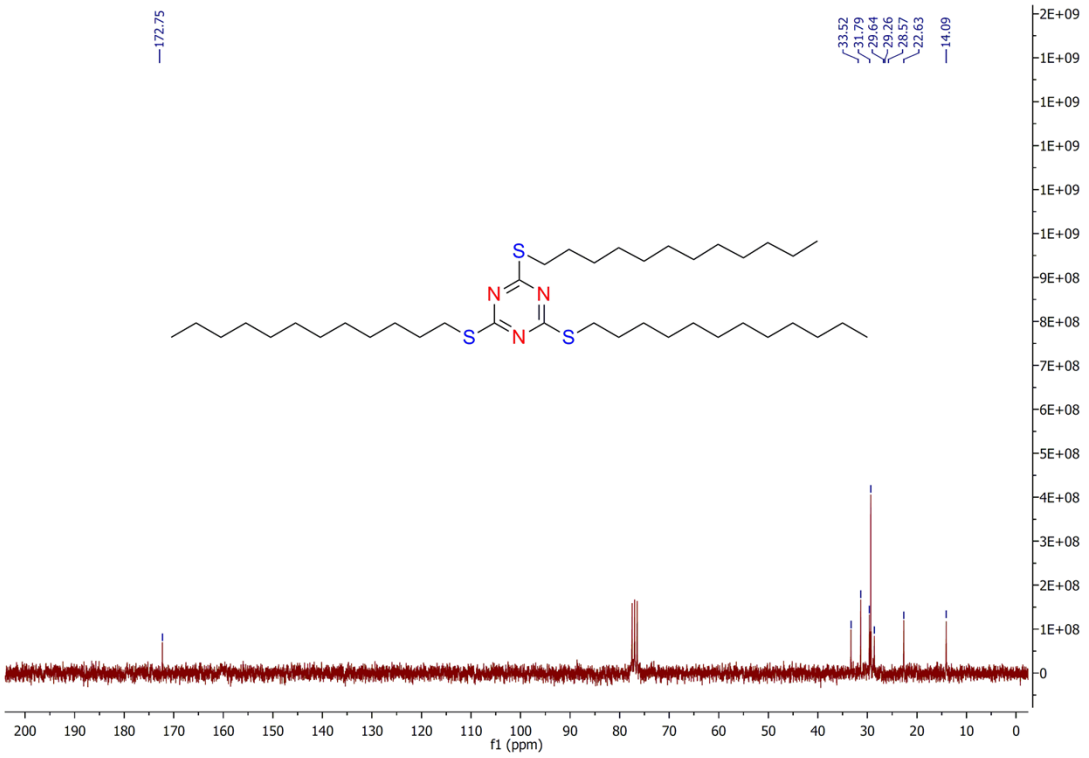
7.2. 2,4,6-tris(dodecyloxy)-1,3,5-triazine (3b)



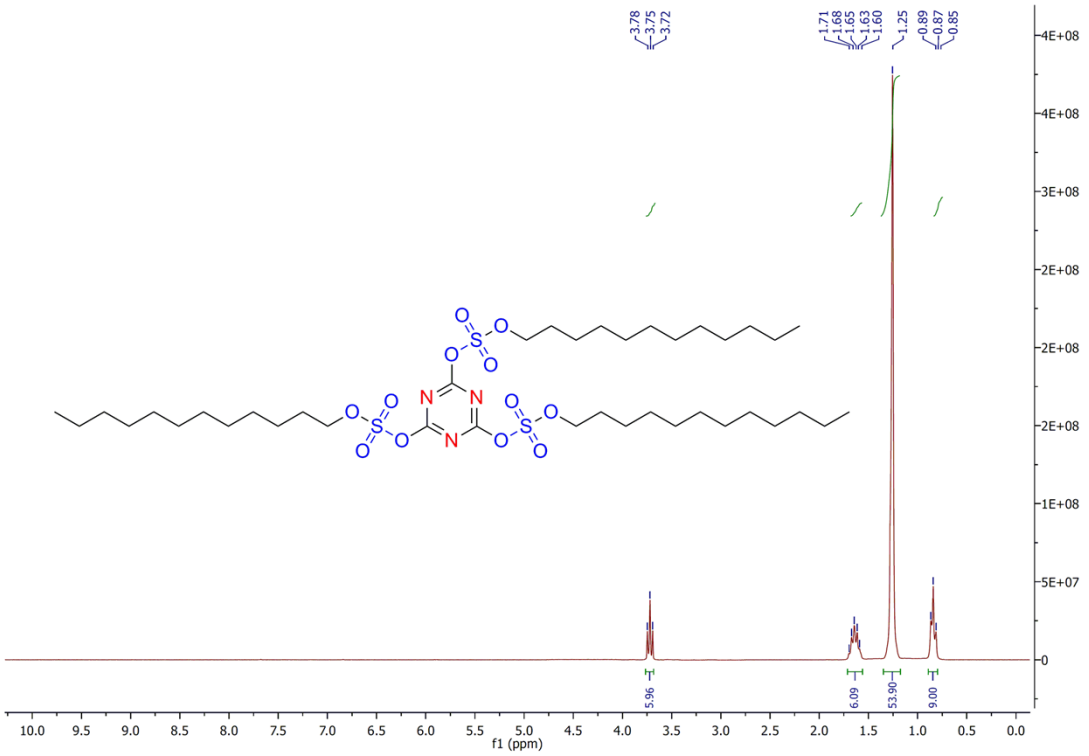


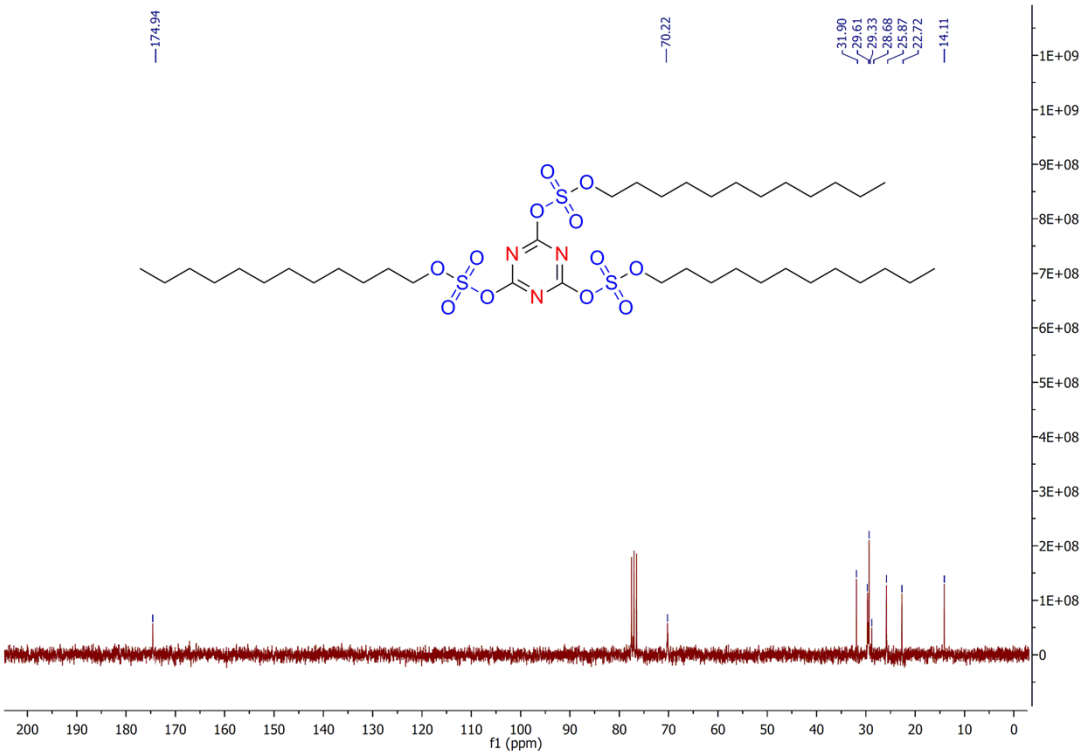
7.3. 2,4,6-tris(dodecylthio)-1,3,5-triazine (3c)



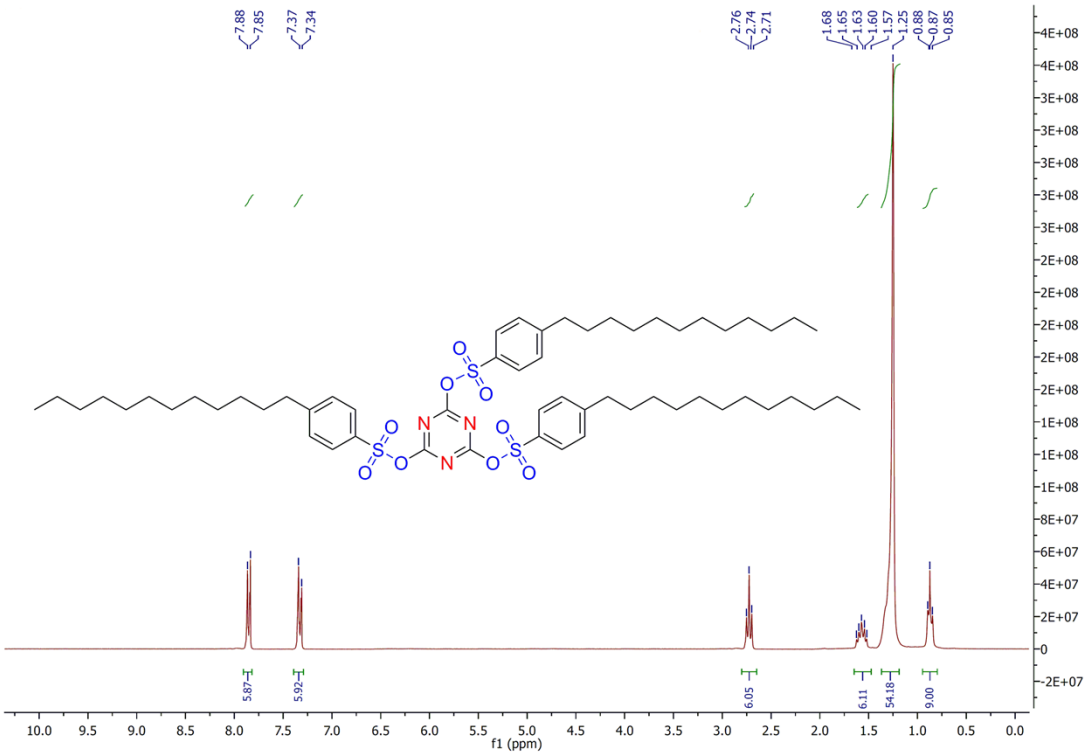


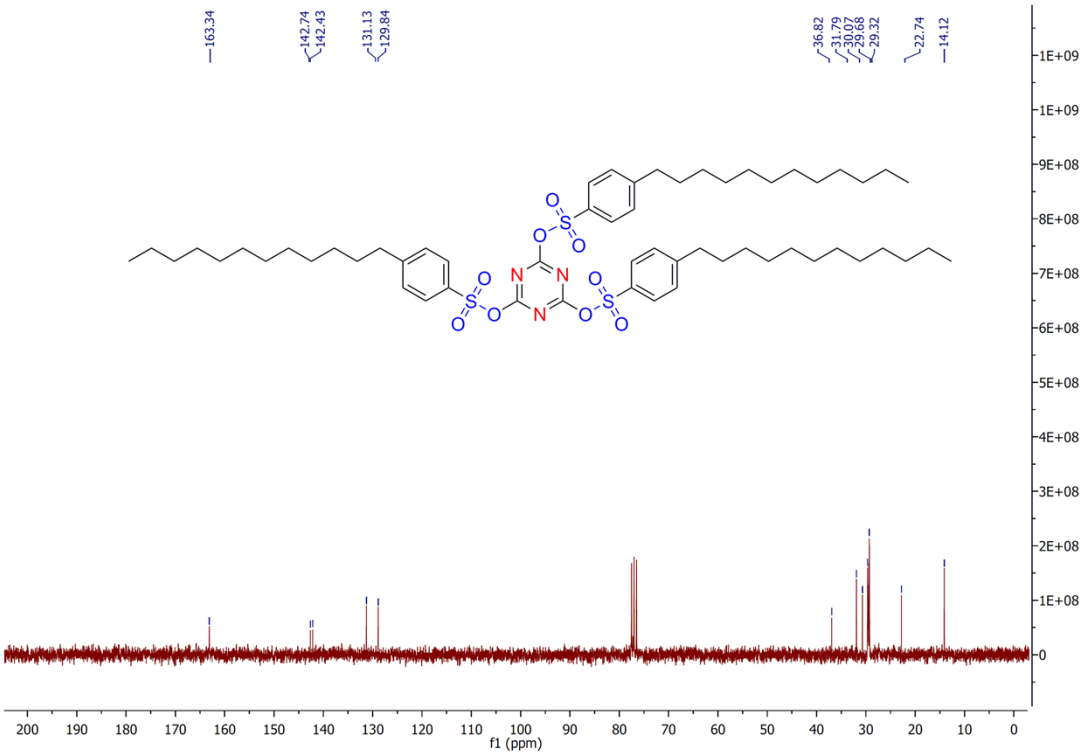
7.4. tridodecyl 1,3,5-triazine-2,4,6-triyl tris(sulfate) (3d)



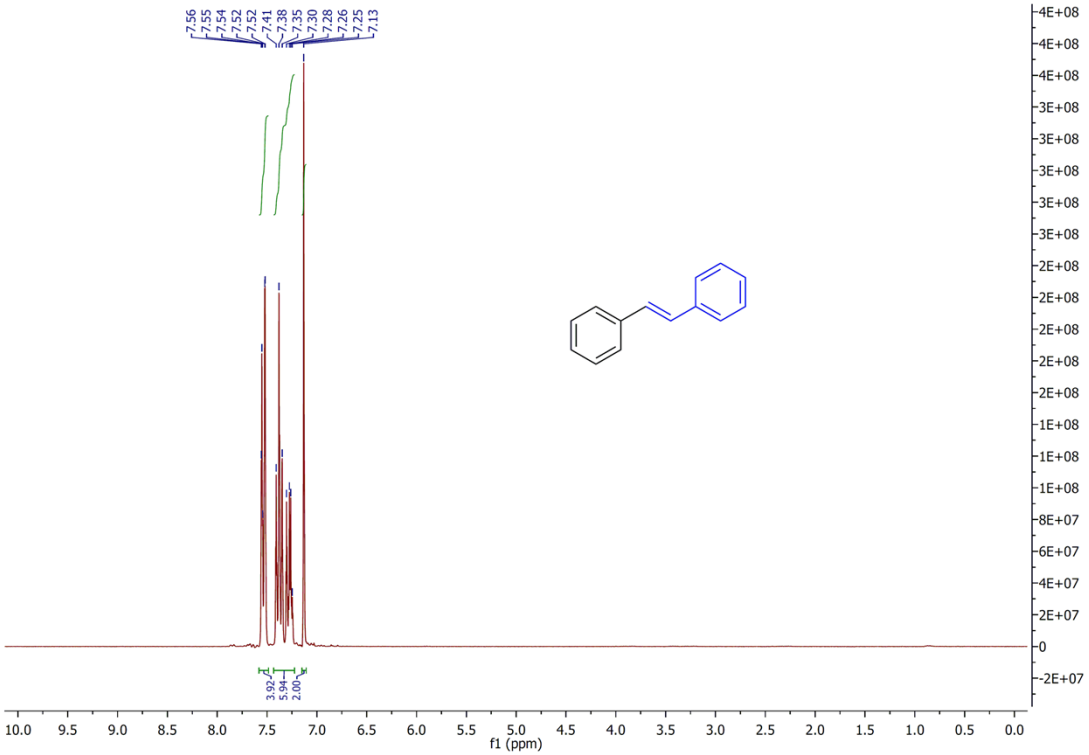


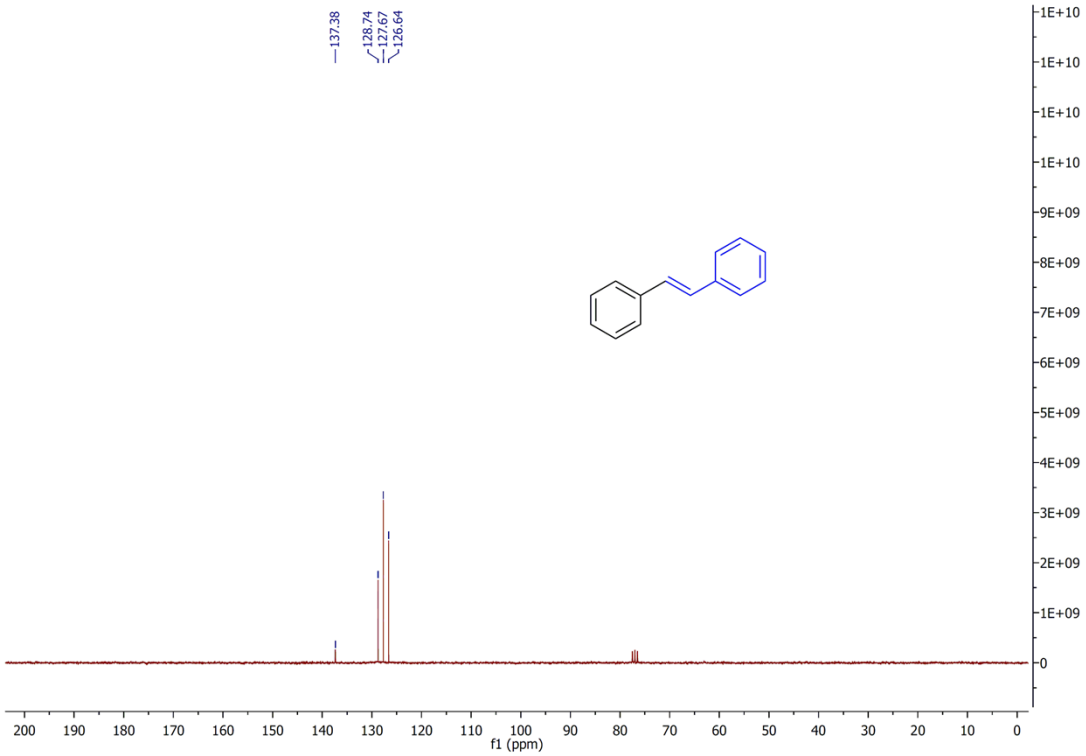
7.5. 1,3,5-triazine-2,4,6-triyl tris(4-dodecylbenzenesulfonate) (3e)



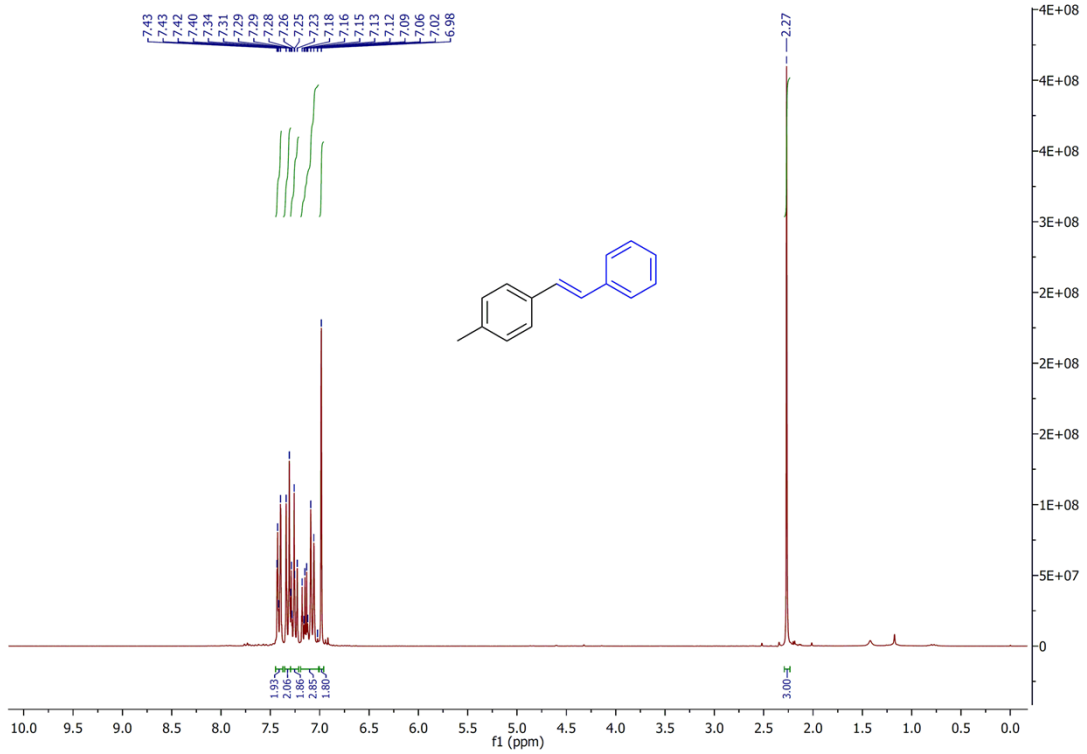


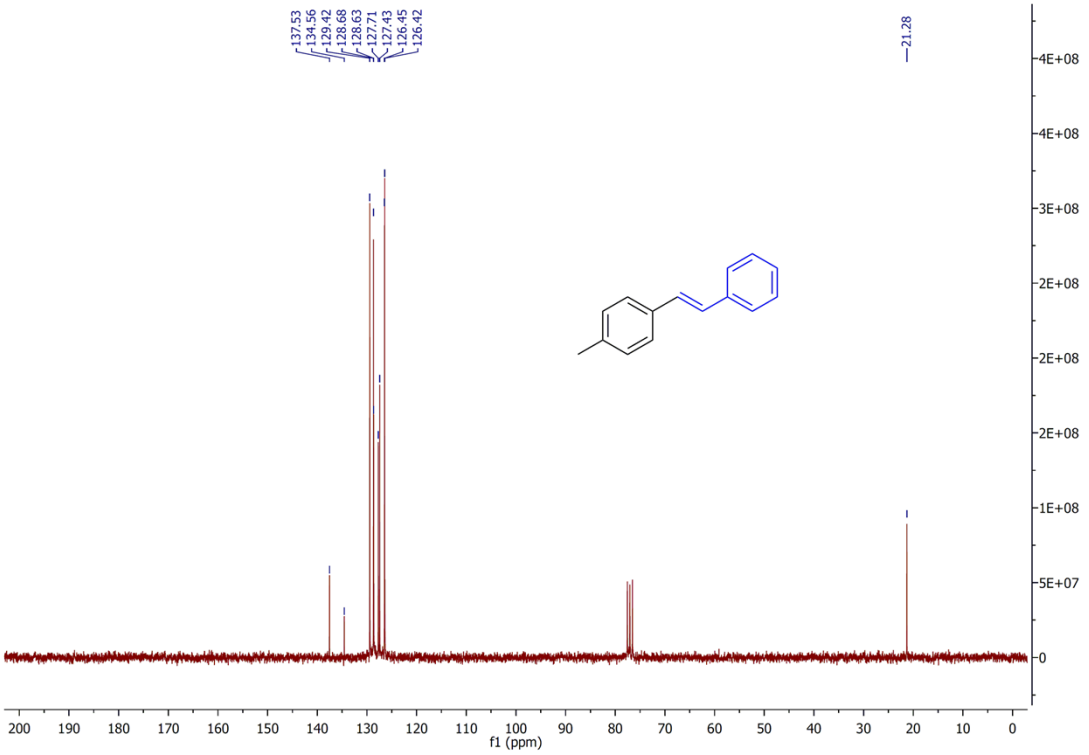
7.6. (*E*)-1,2-diphenylethene (6a)



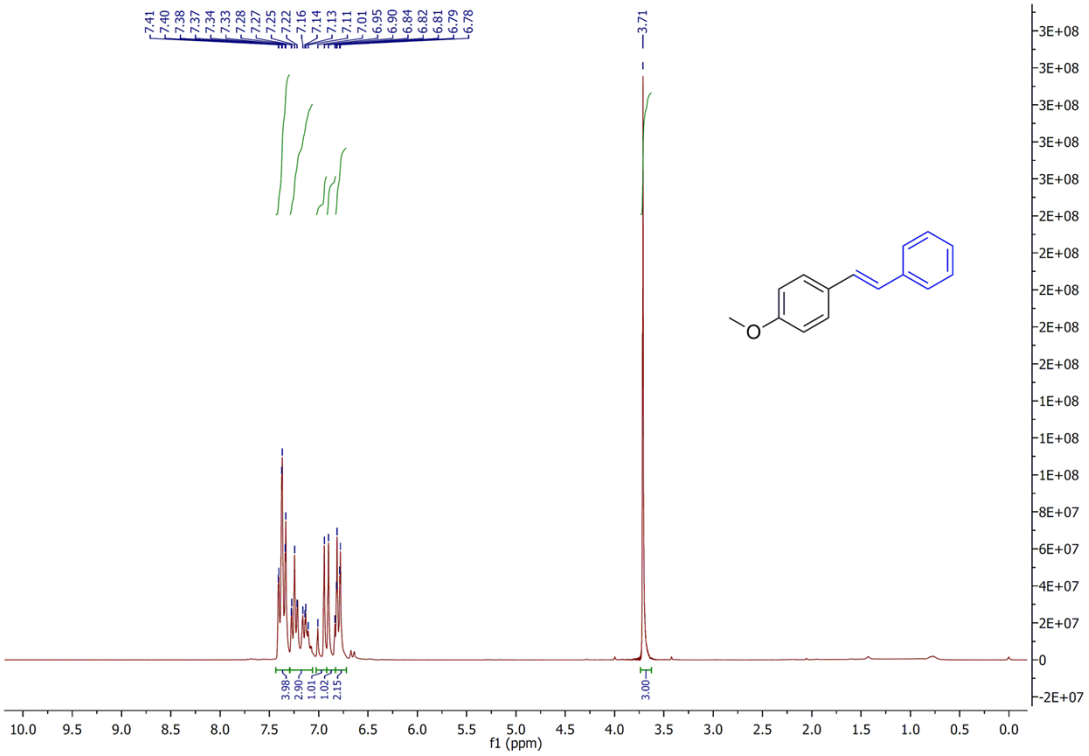


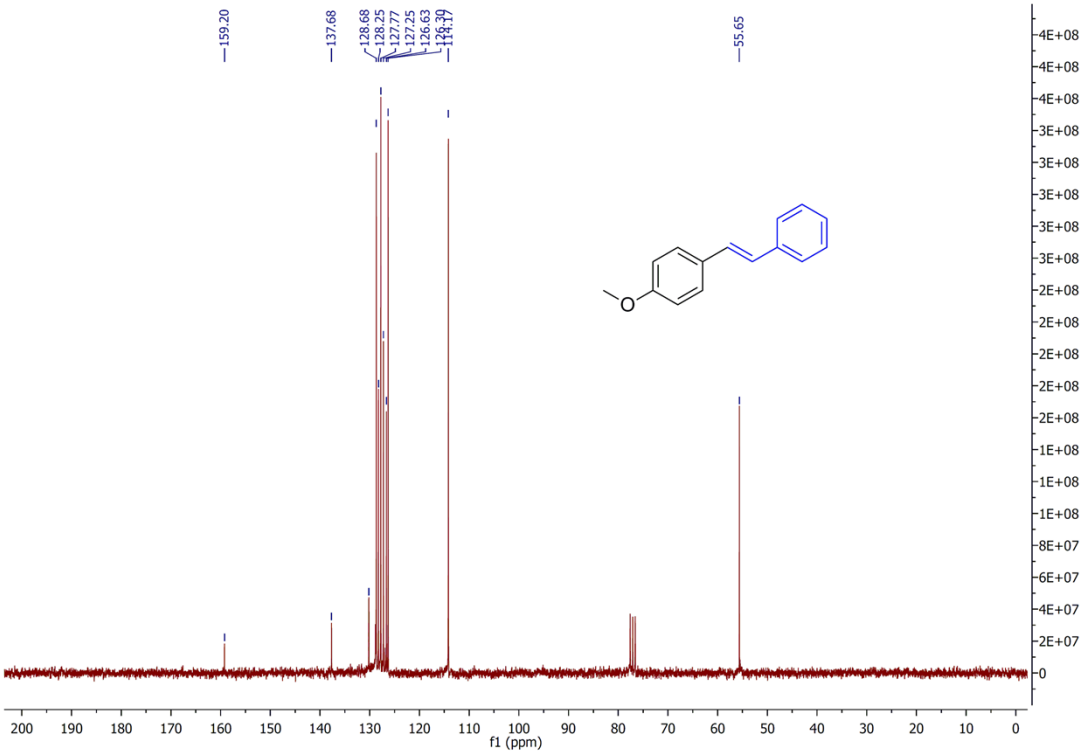
7.7. (E)-1-methyl-4-styrylbenzene (6b)



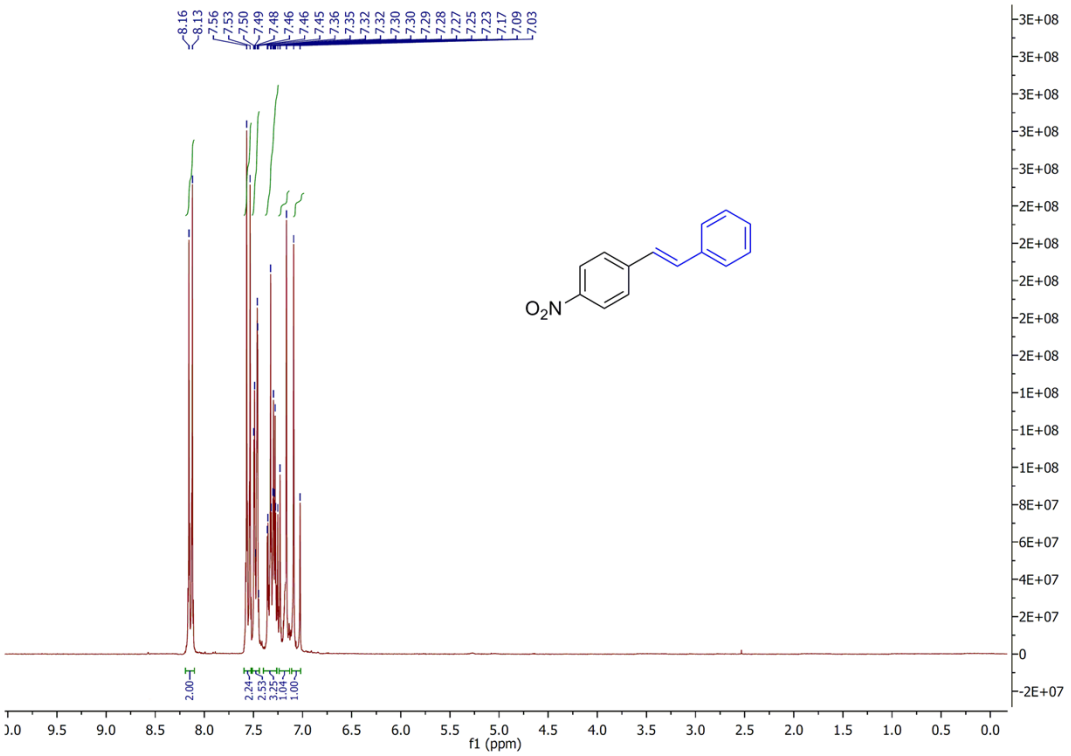


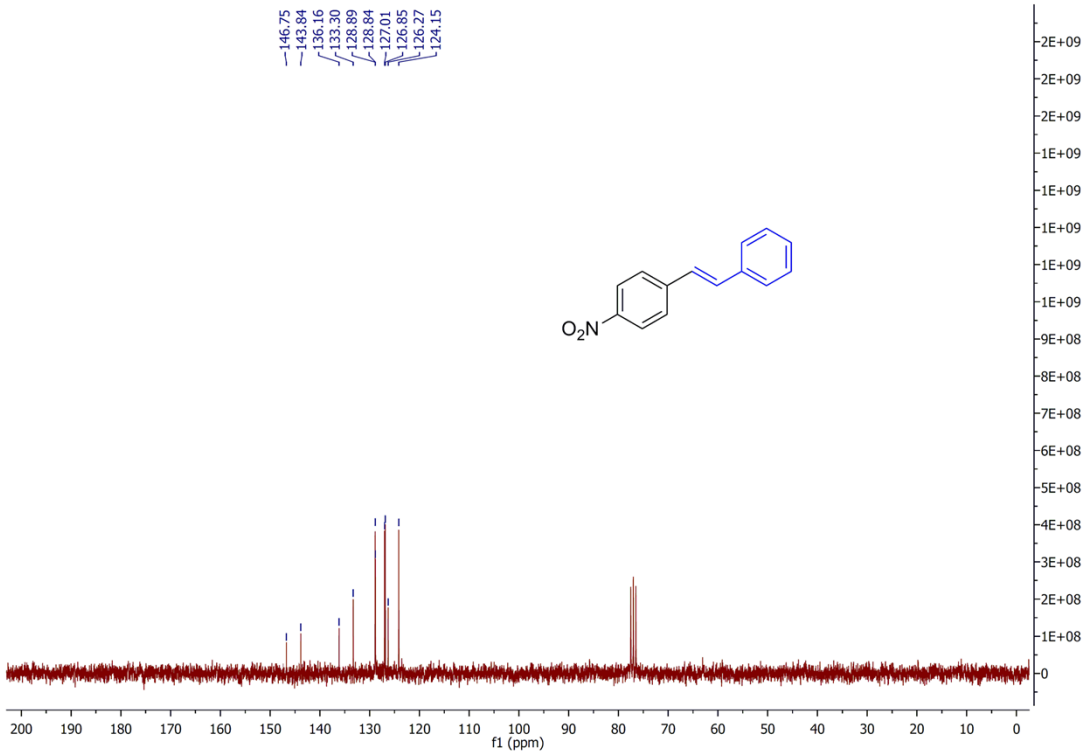
7.8. (E)-1-methoxy-4-styrylbenzene (6c)



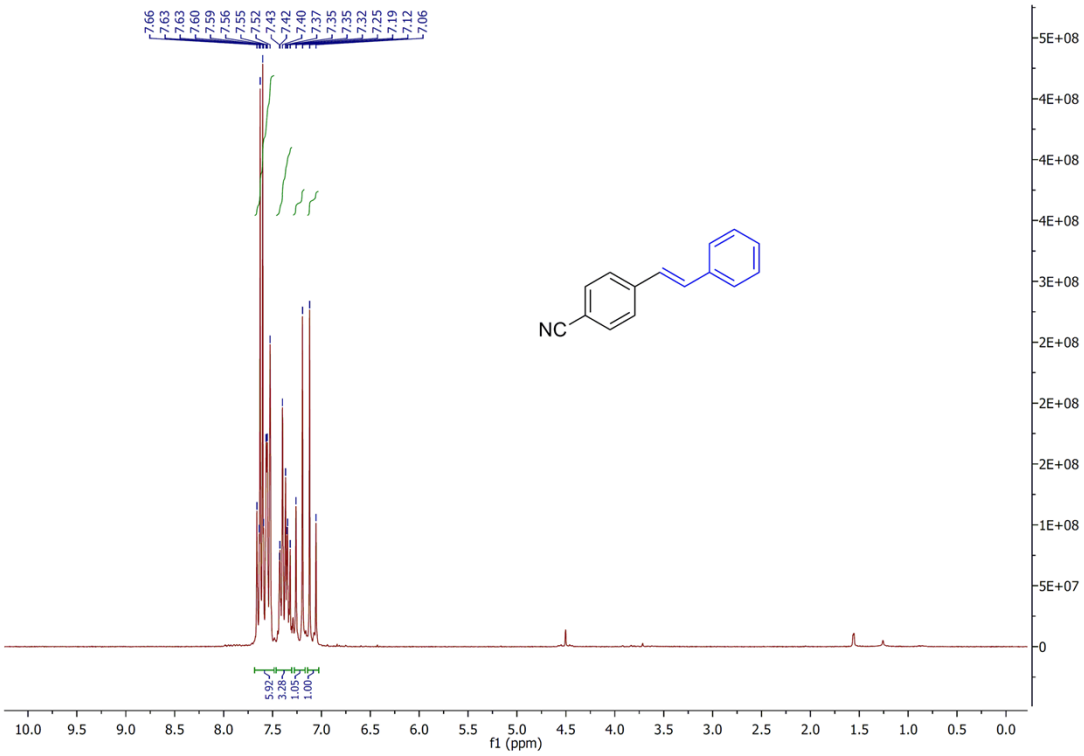


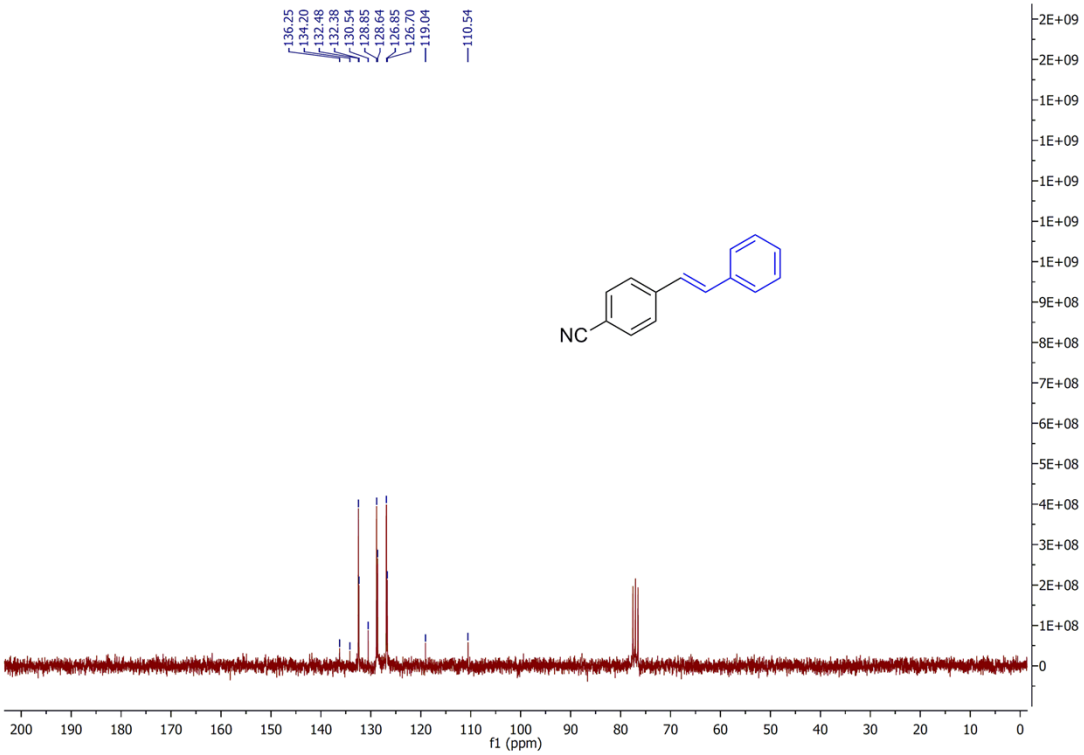
7.9. (E)-1-nitro-4-styrylbenzene (6d)



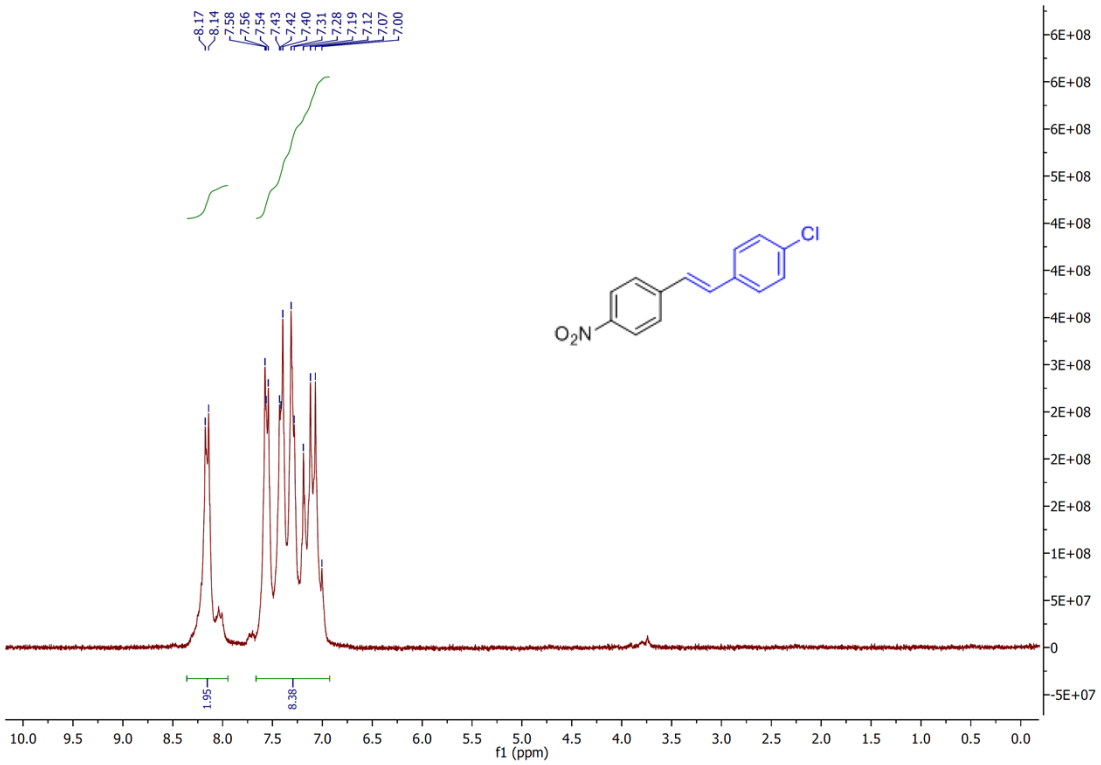


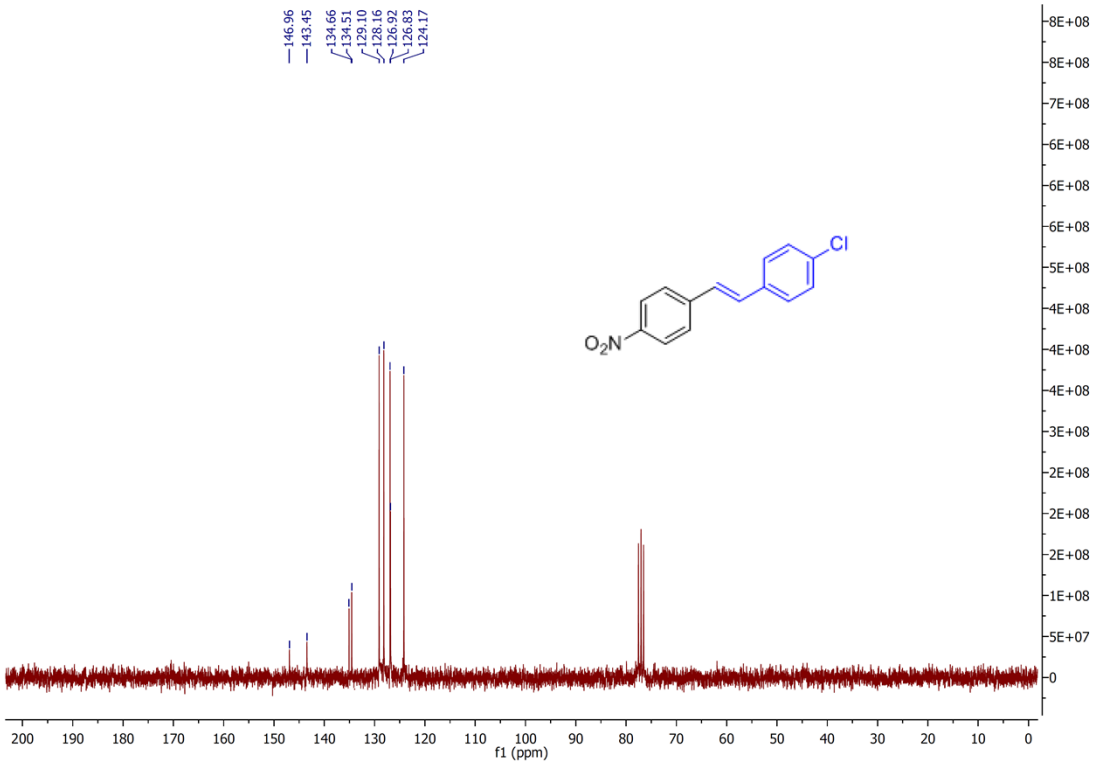
7.10. (E)-4-styrylbenzonitrile (6e)



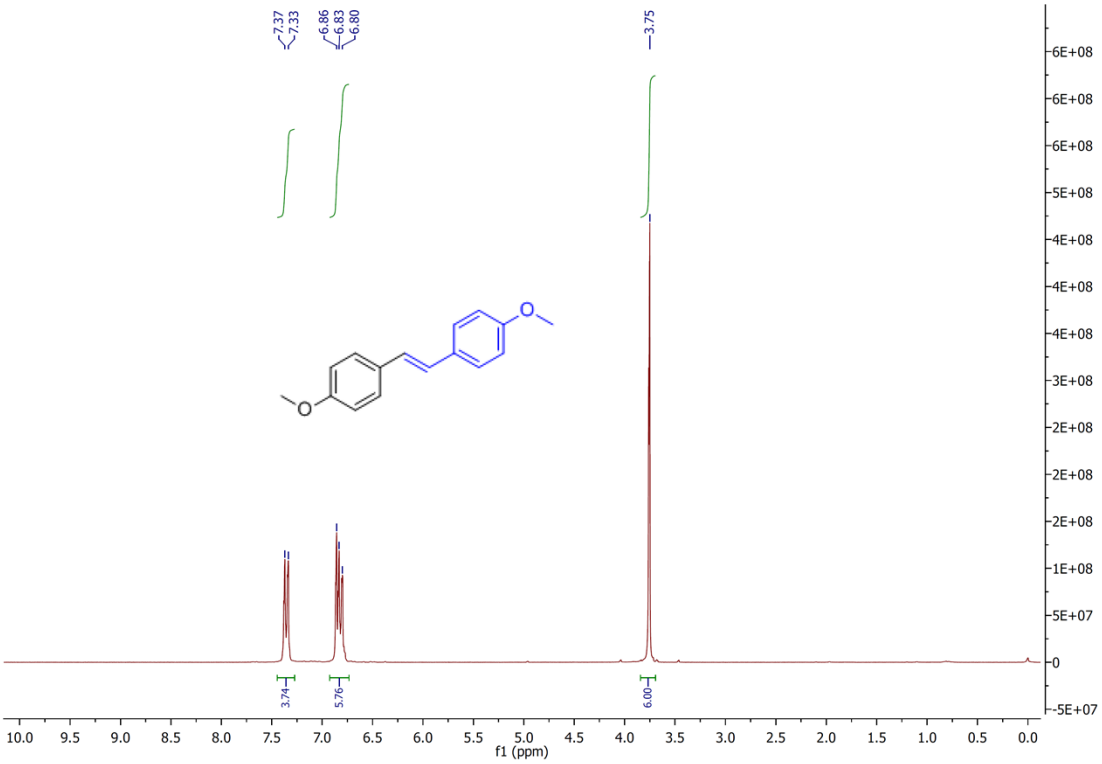


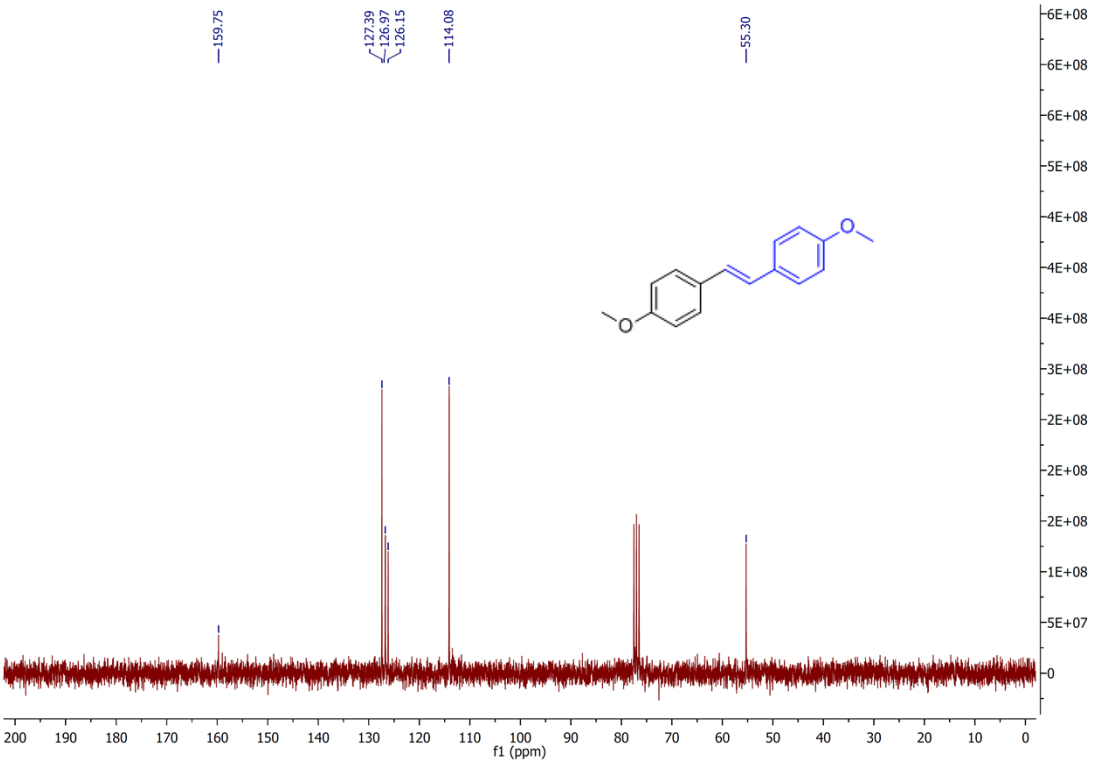
7.11. (E)-1-chloro-4-(4-nitrostyryl)benzene (6f)



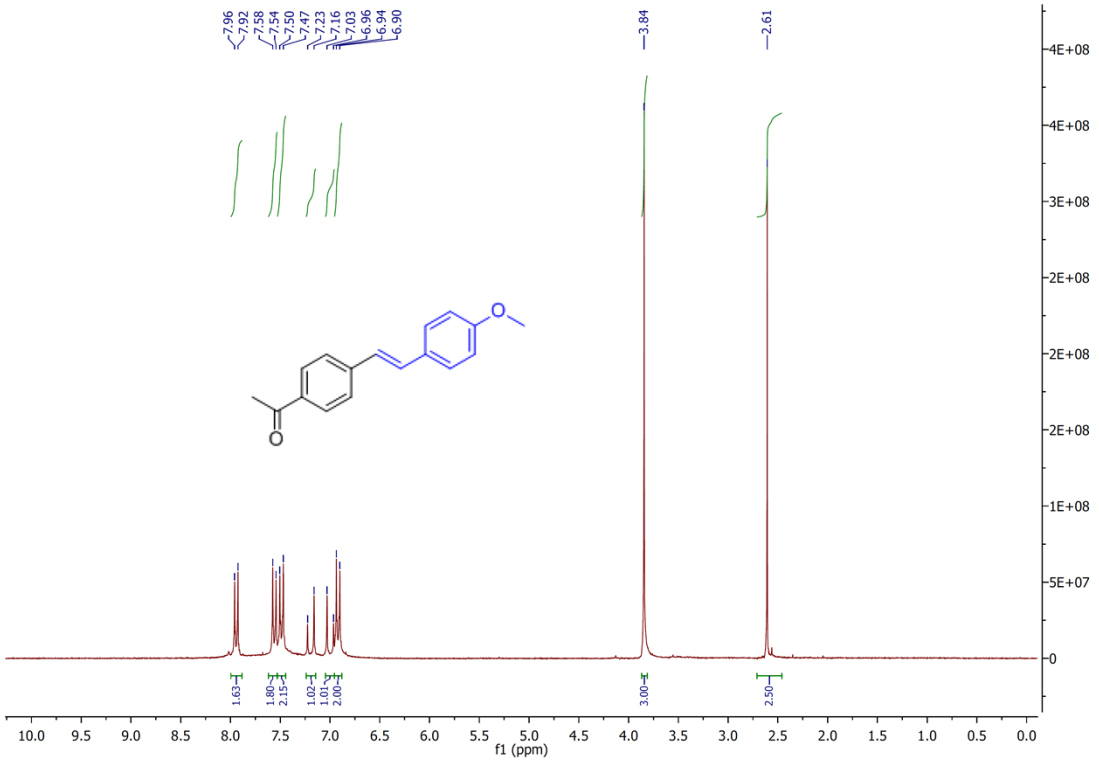


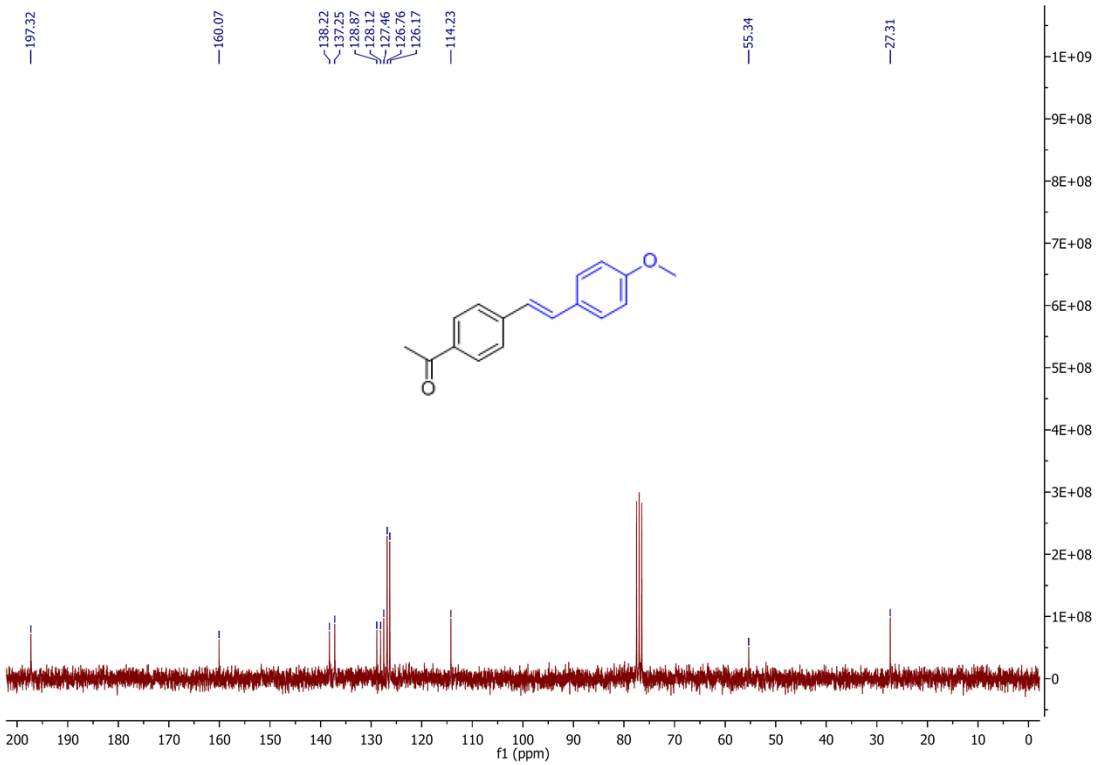
7.12. (E)-1,2-bis(4-methoxyphenyl)ethene (6g)



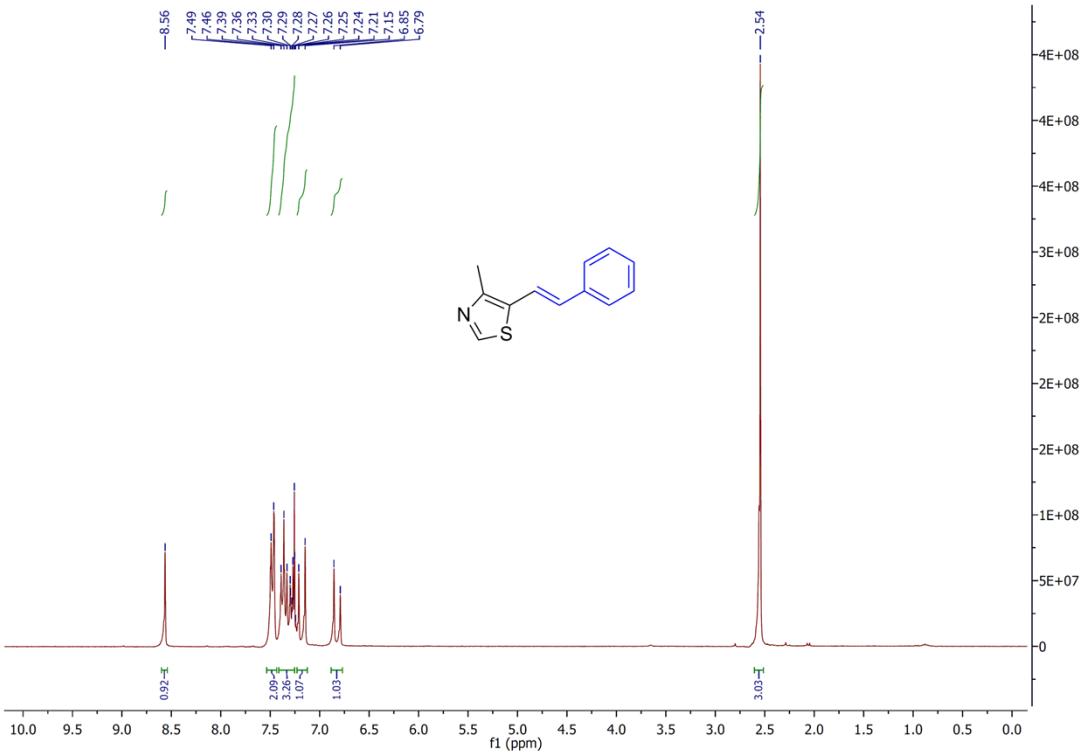


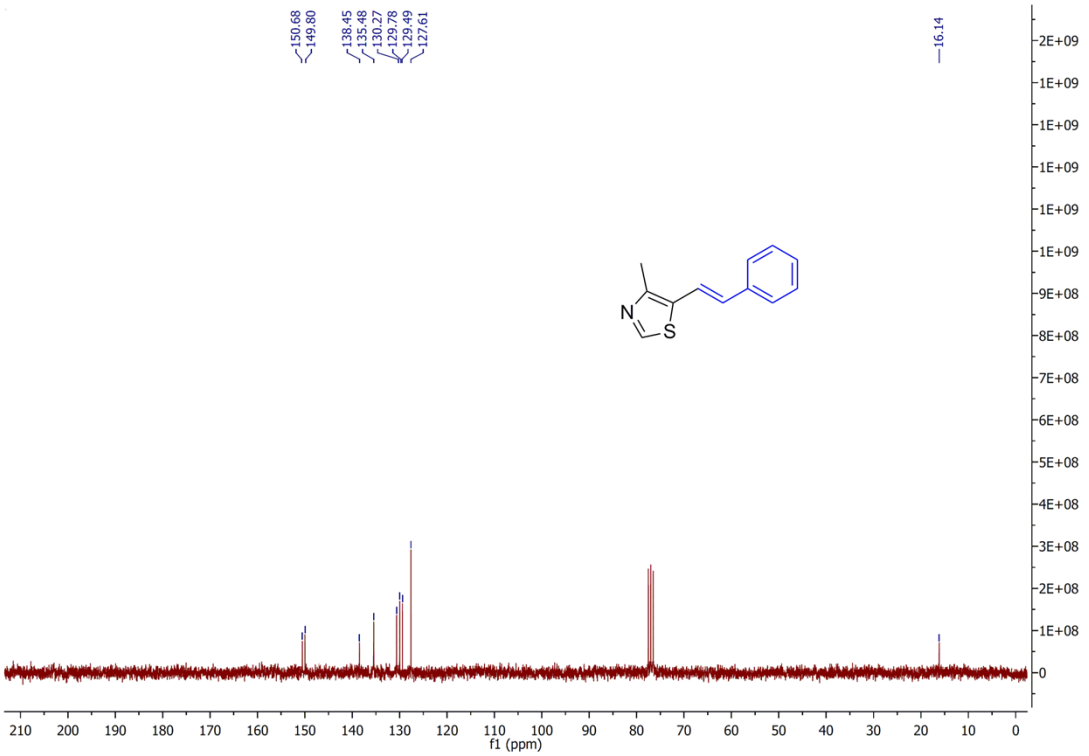
7.13. (E)-1-(4-(4-methoxystyryl)phenyl)ethan-1-one (6h)



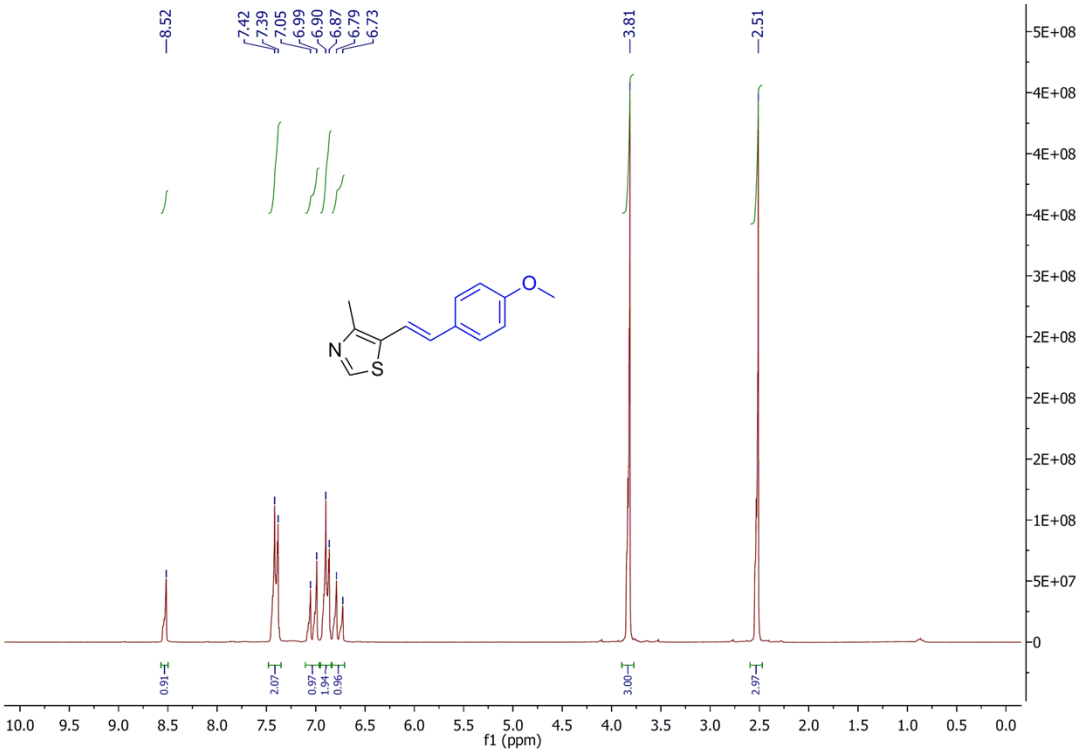


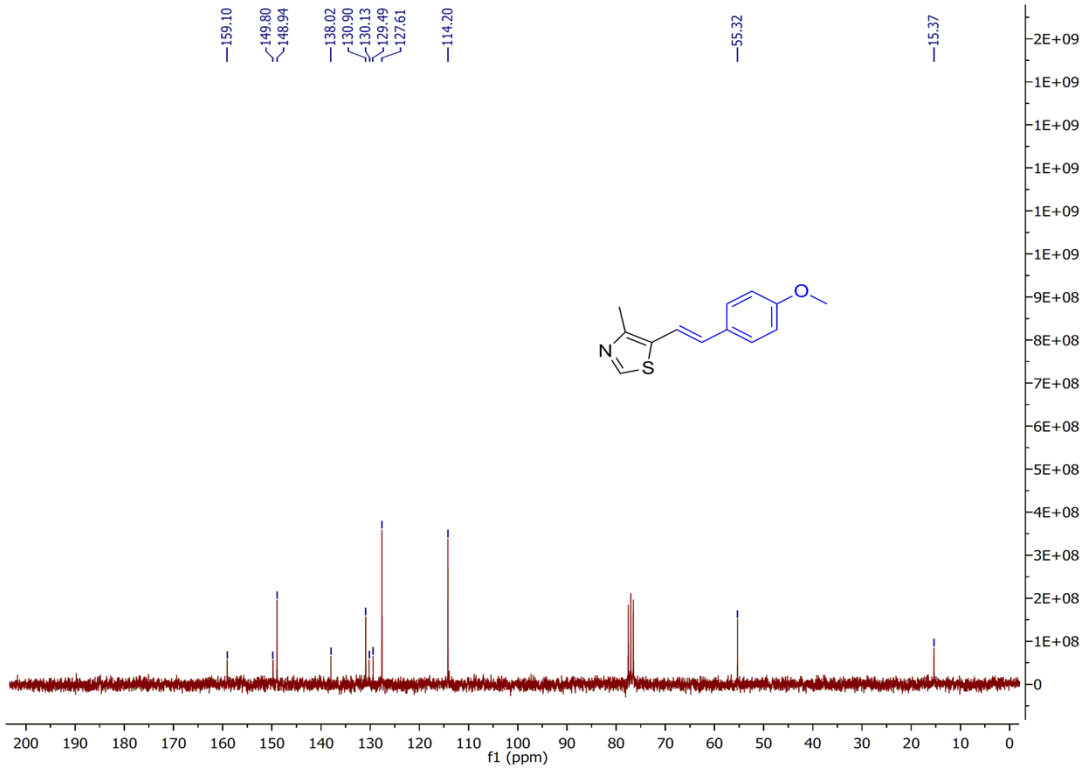
7.14. (E)-4-methyl-5-styrylthiazole (6i)



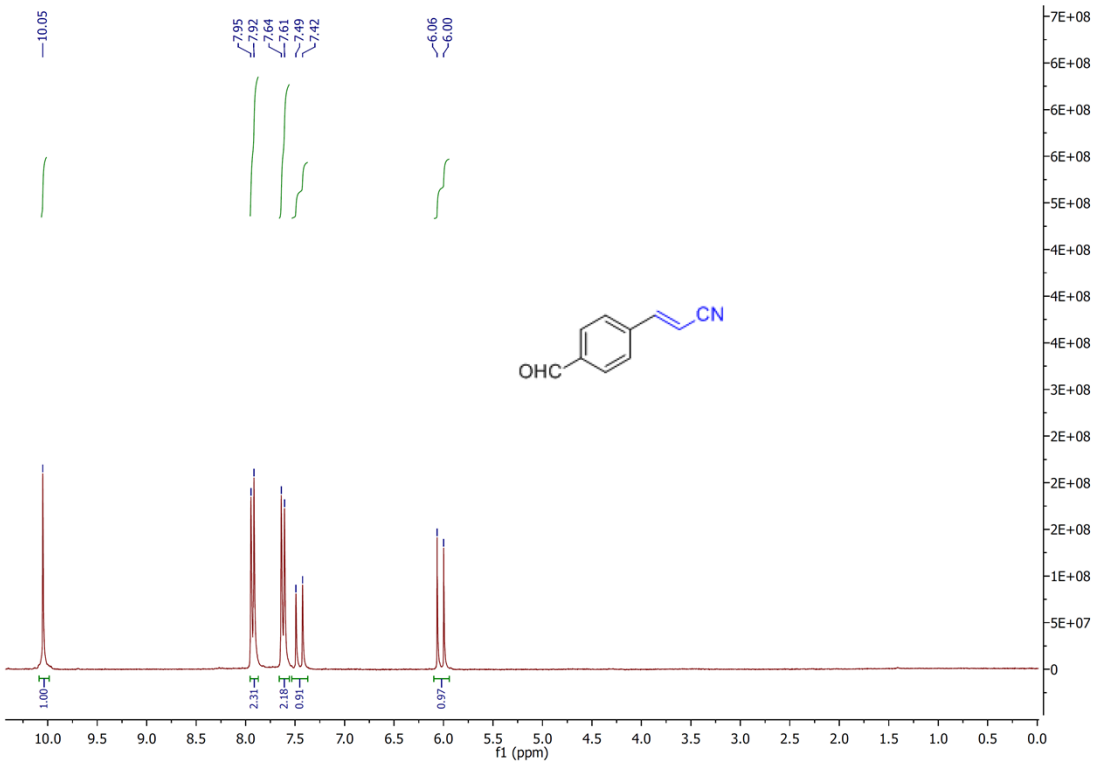


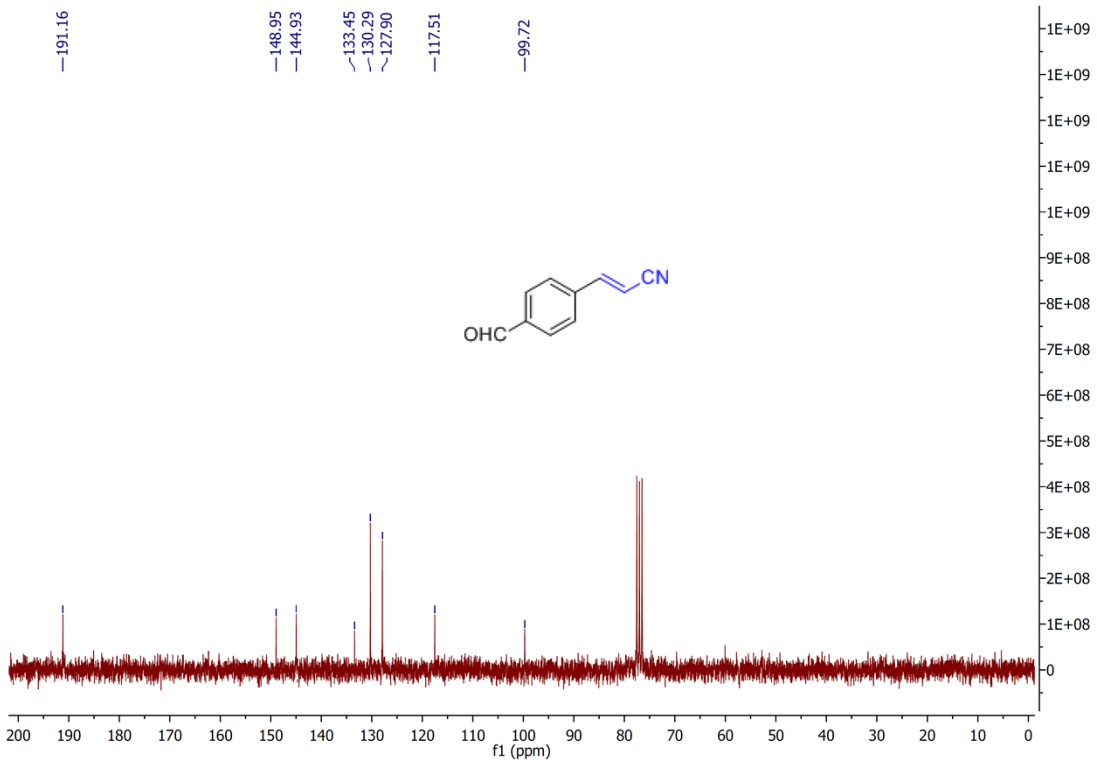
7.15. (E)-5-(4-methoxystyryl)-4-methylthiazole (6j)



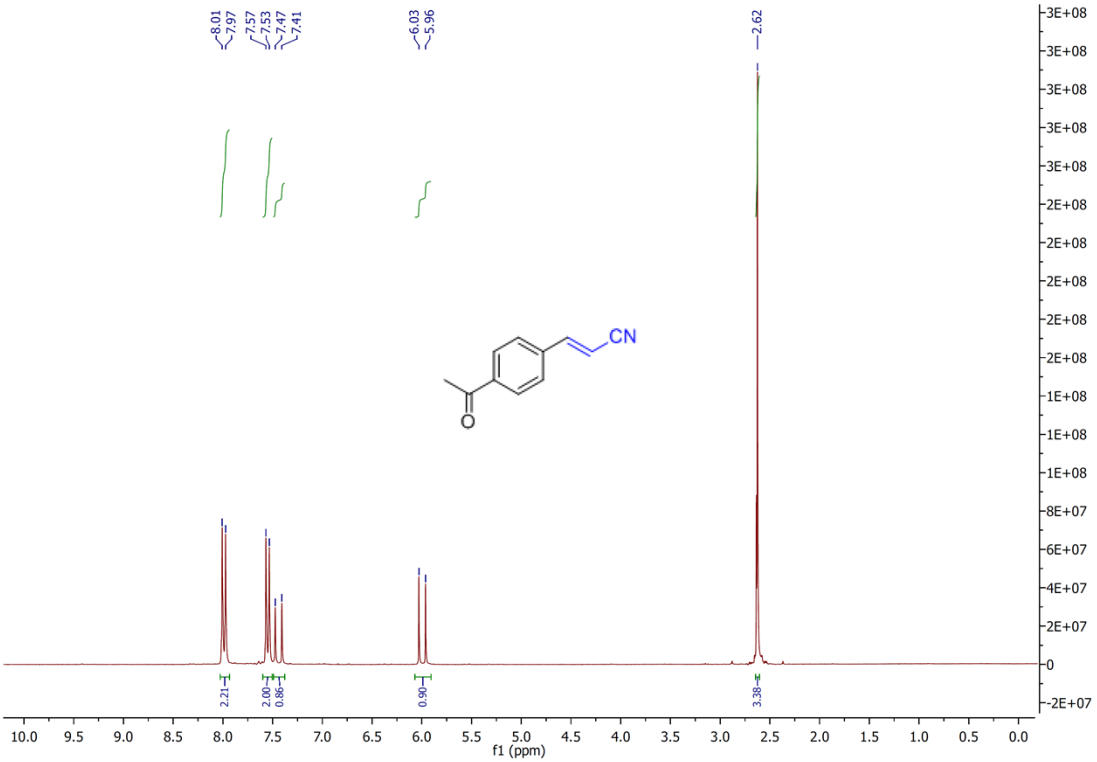


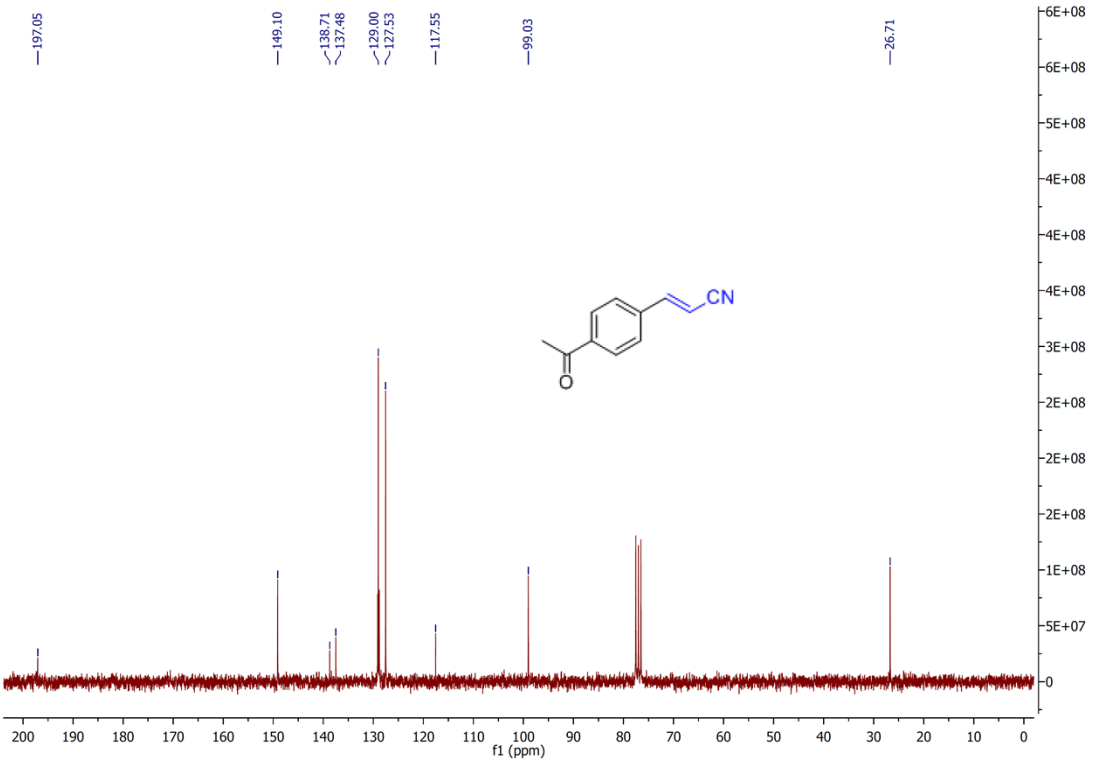
7.16. (E)-3-(4-formylphenyl)acrylonitrile (6k)



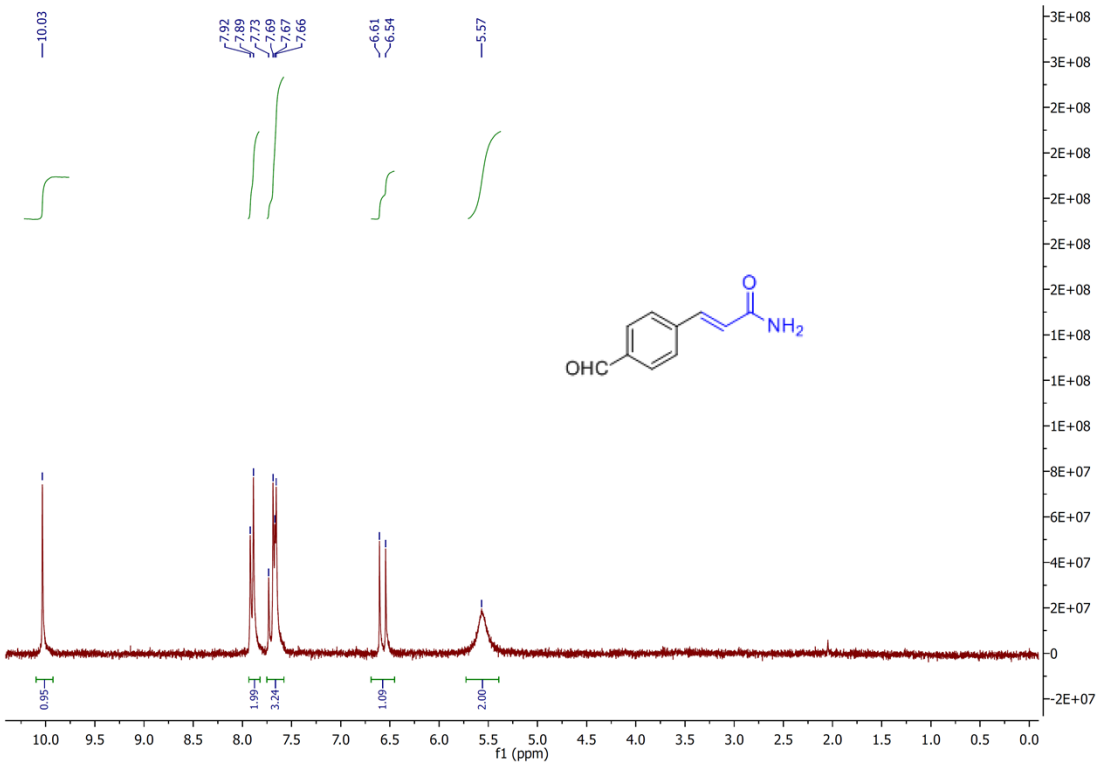


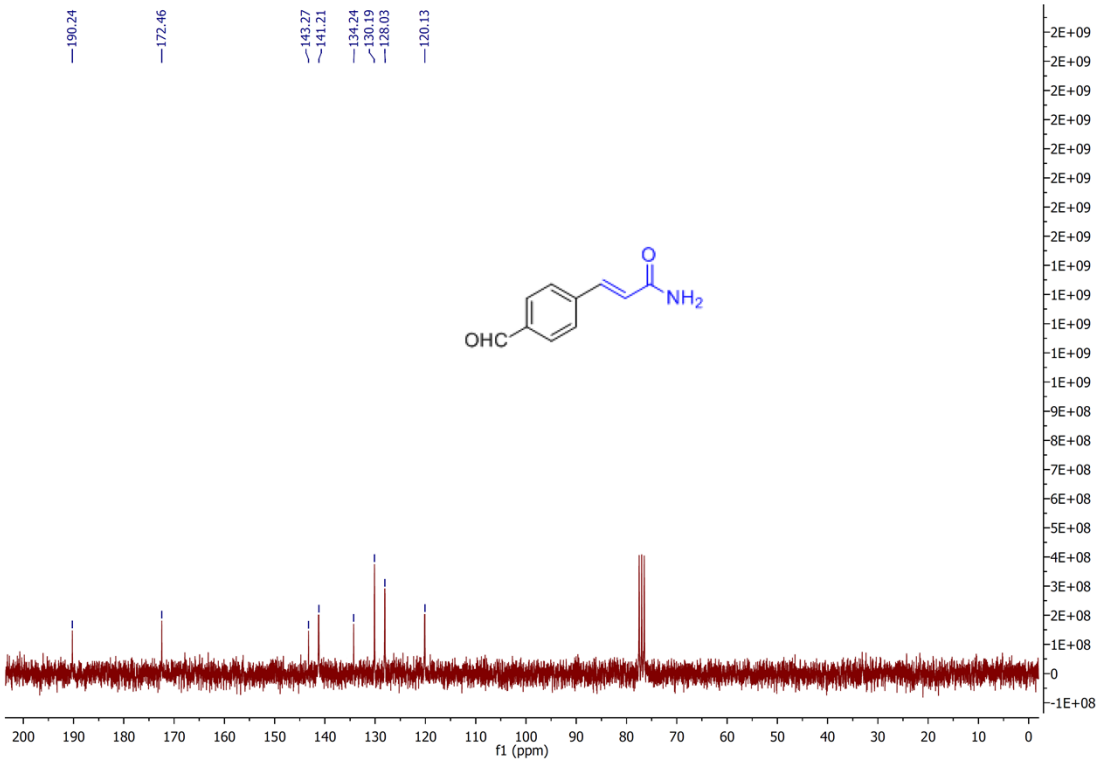
7.17. (E)-3-(4-acetylphenyl)acrylonitrile (6l)



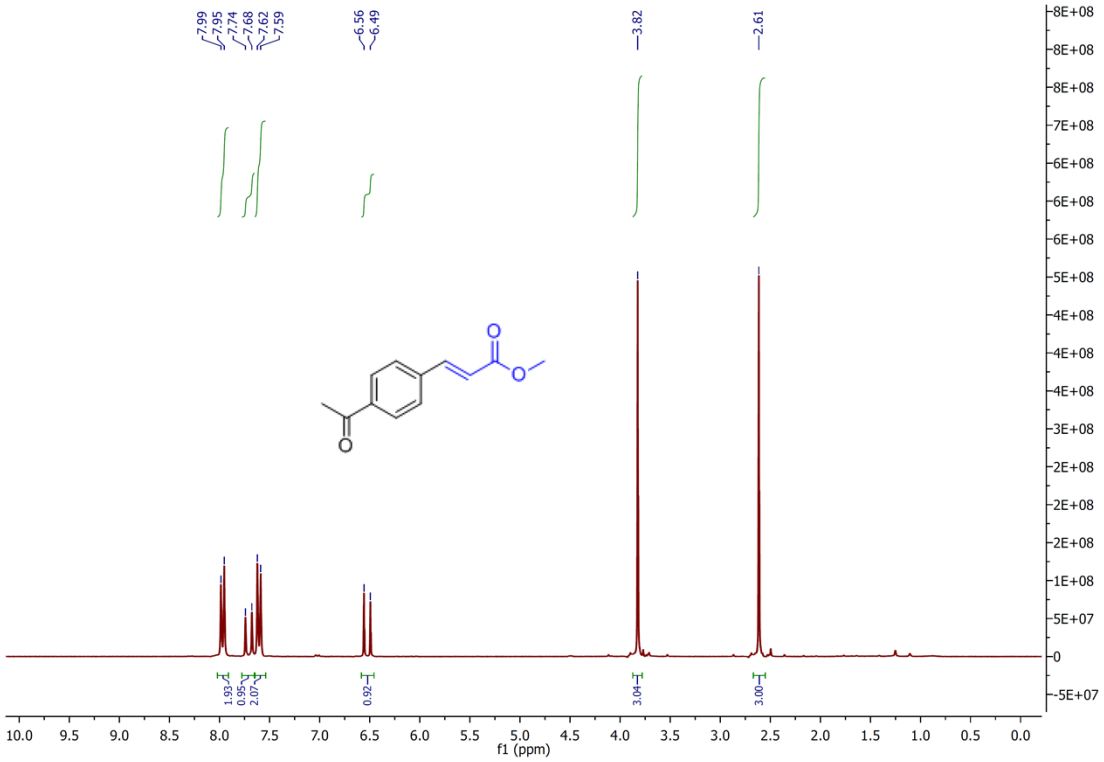


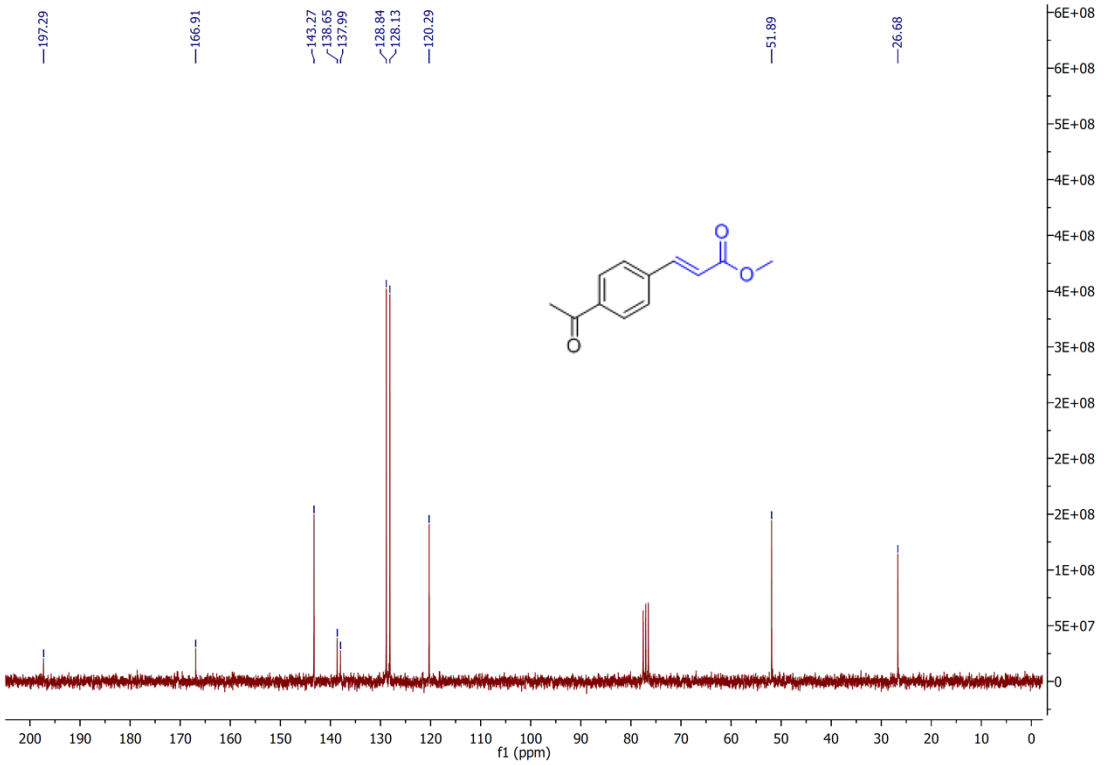
7.18. (E)-3-(4-formylphenyl)acrylamide (6m)



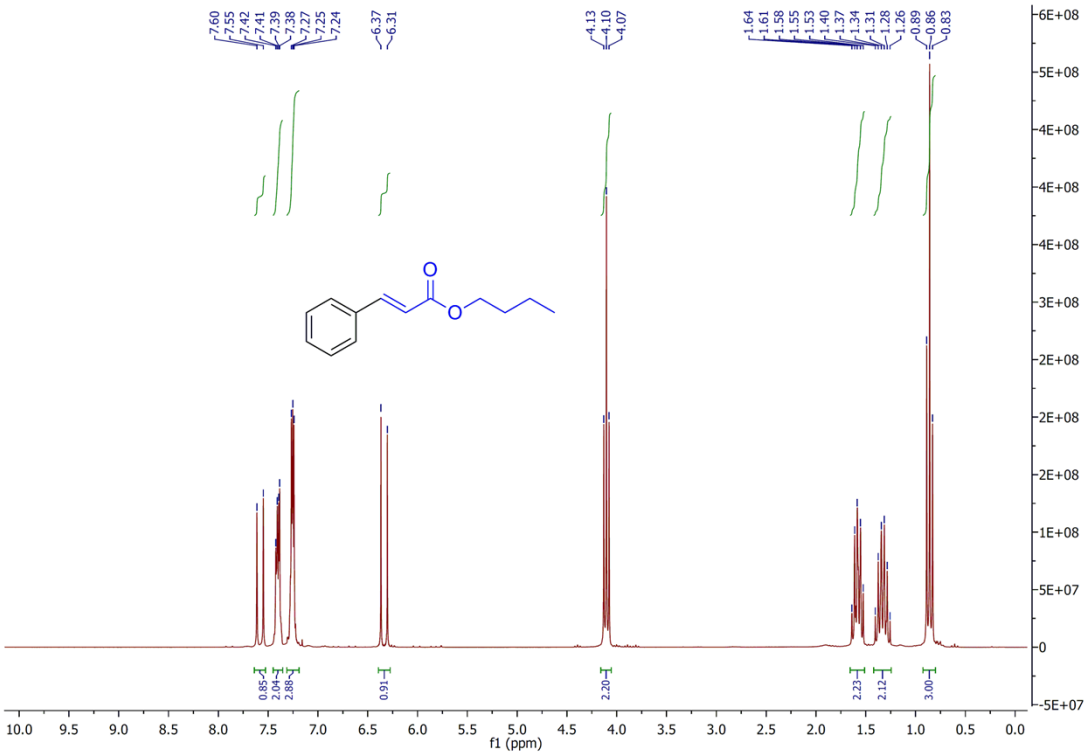


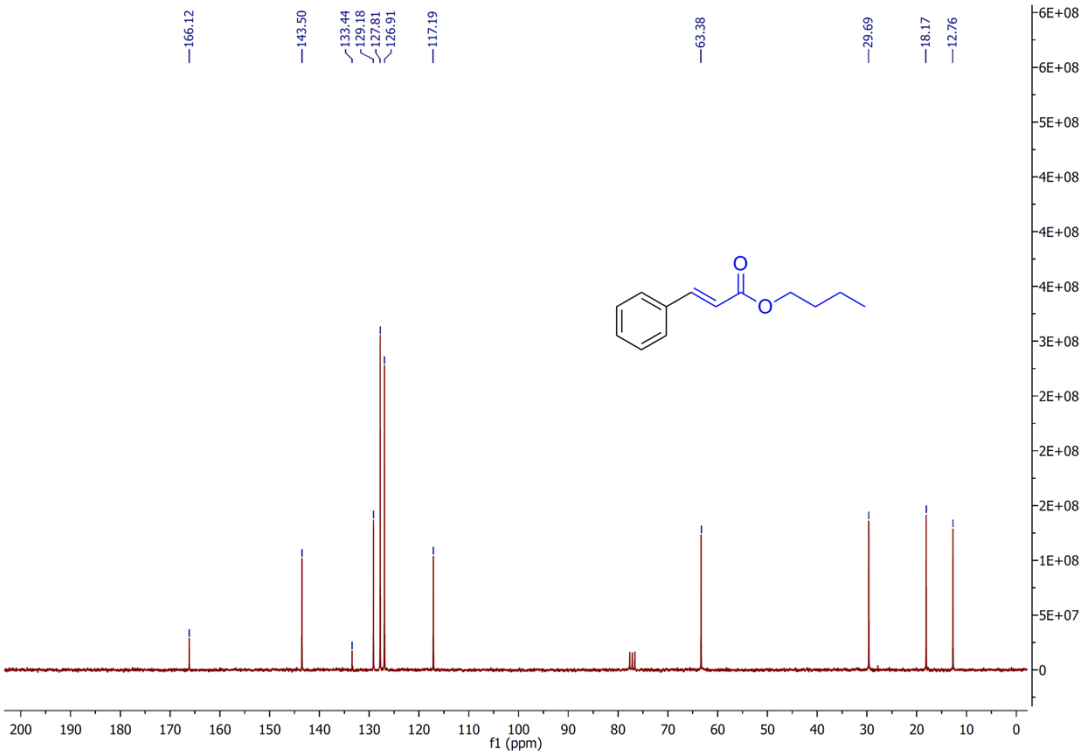
7.19. methyl (E)-3-(4-acetylphenyl)acrylate (6n)



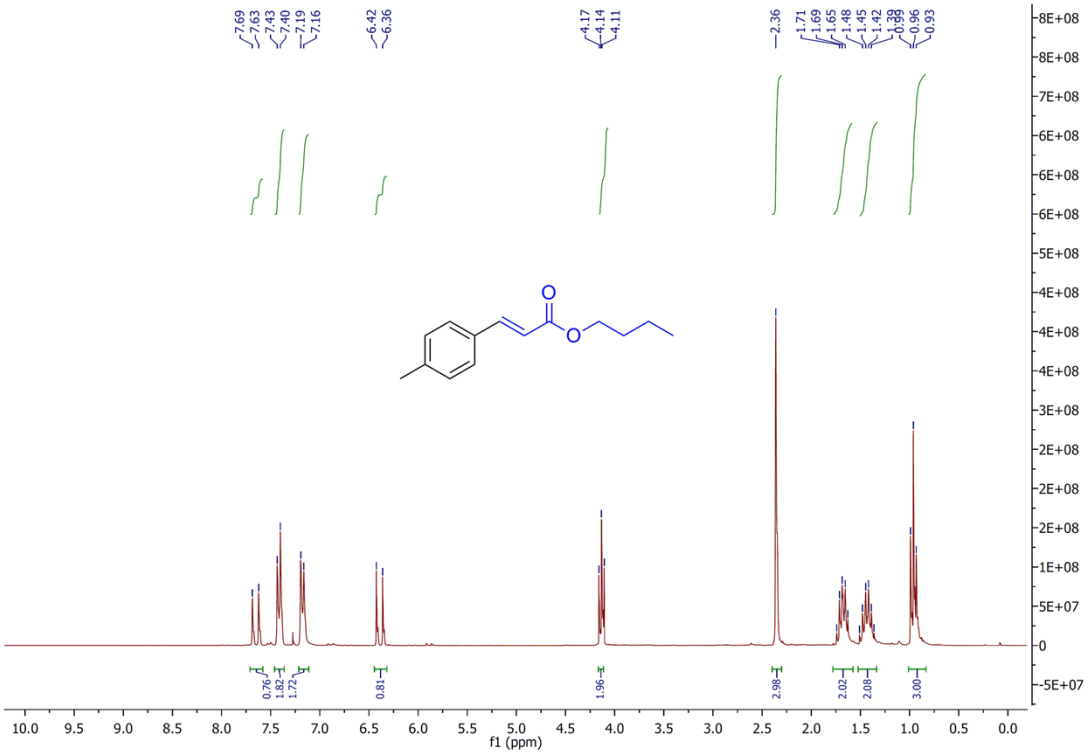


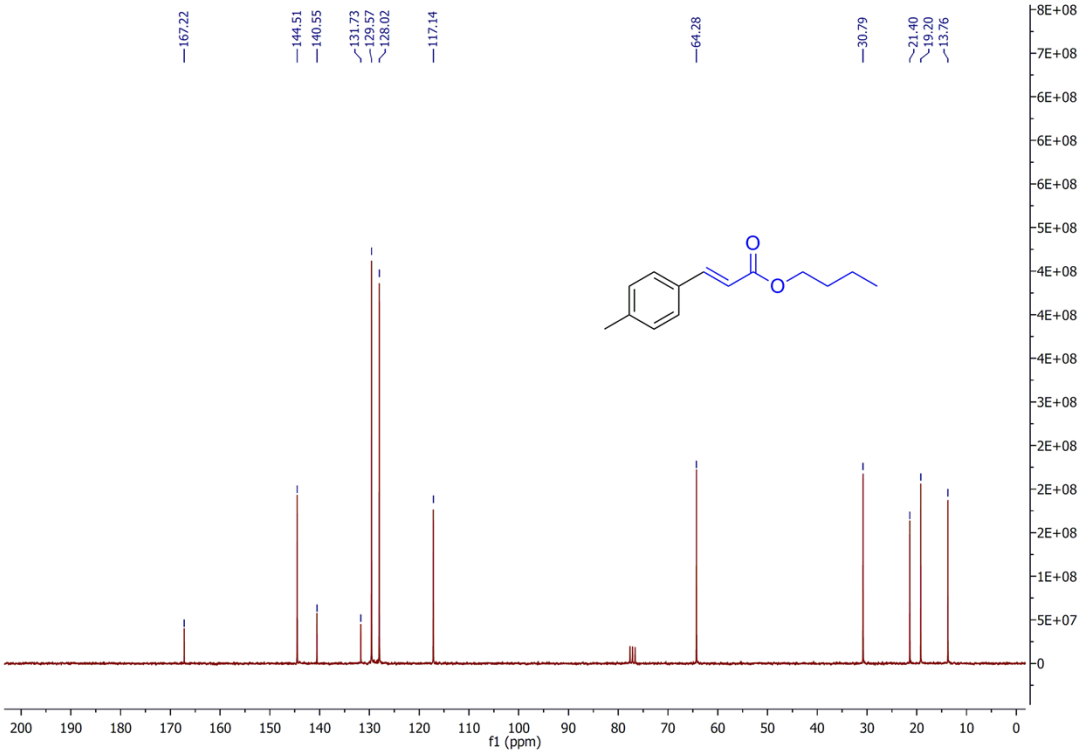
7.20. n-butyl cinnamate (6o)



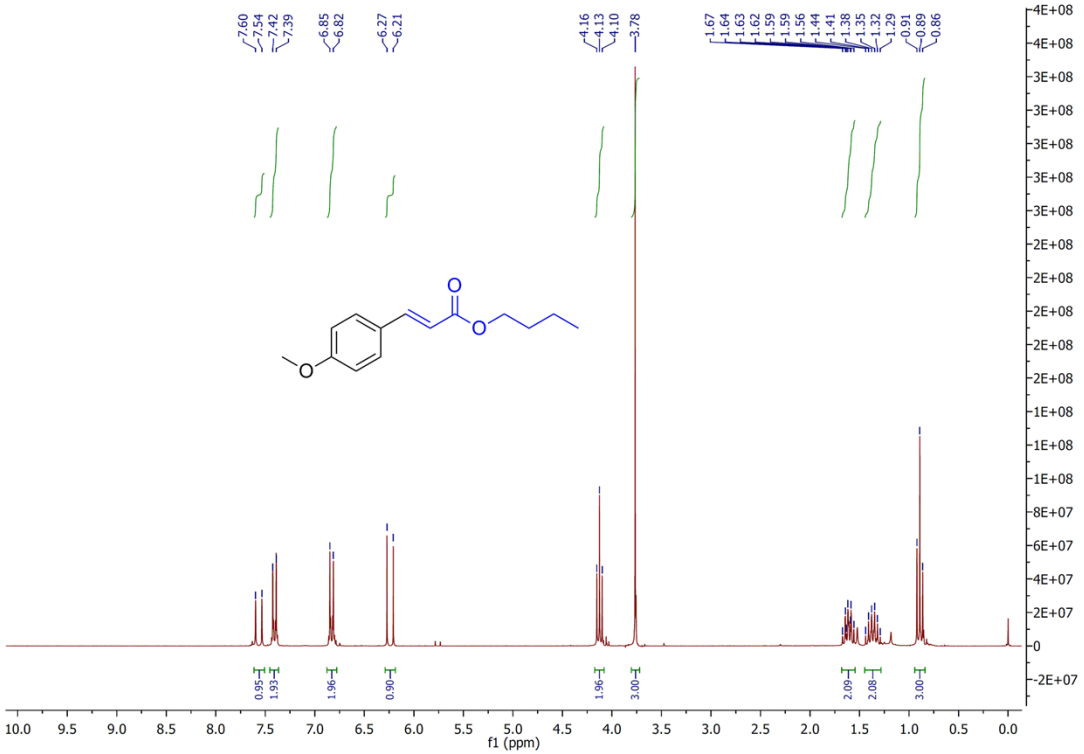


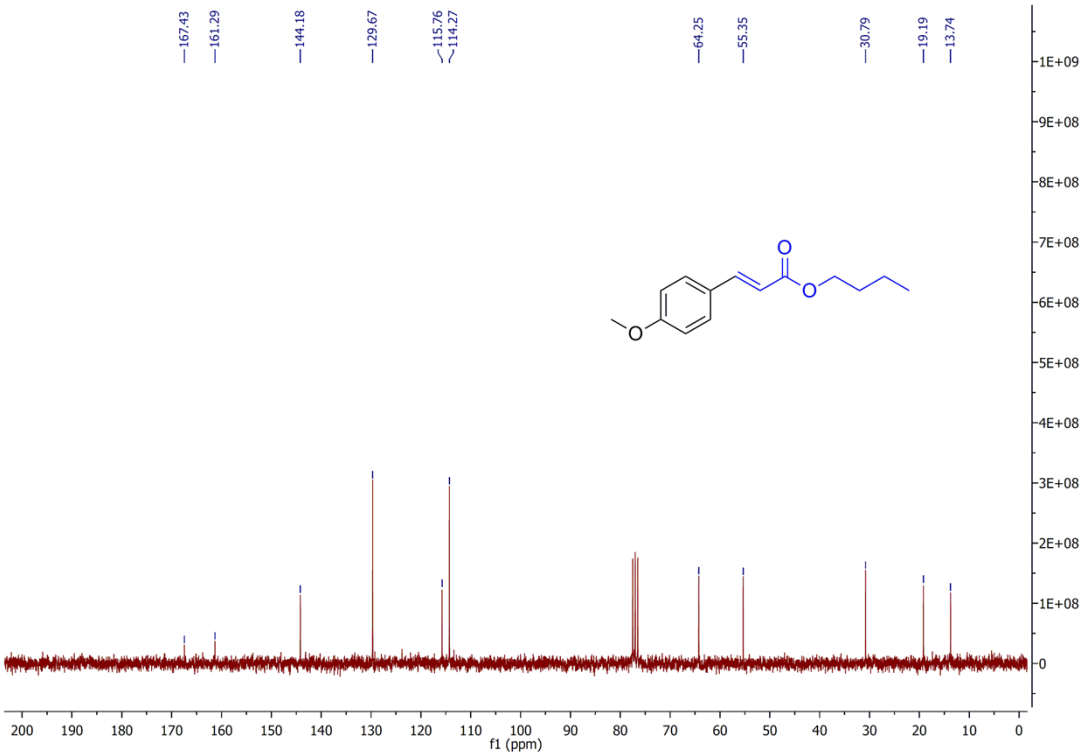
7.21. (E)-butyl 3-(p-tolyl)acrylate (6p)



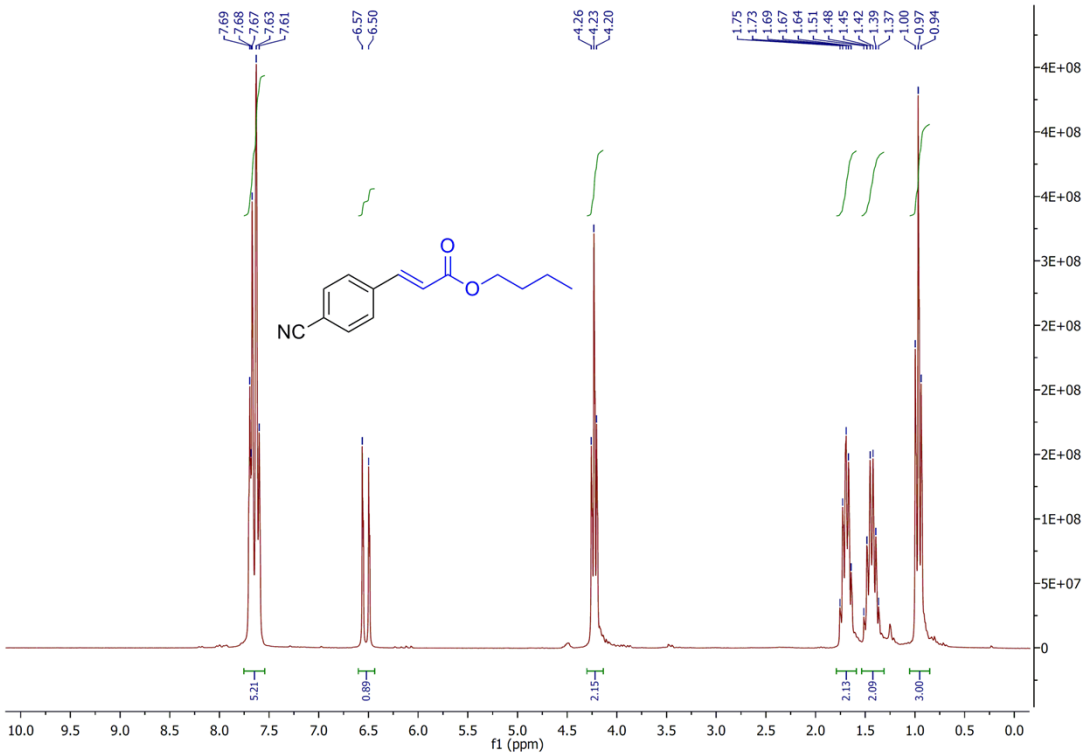


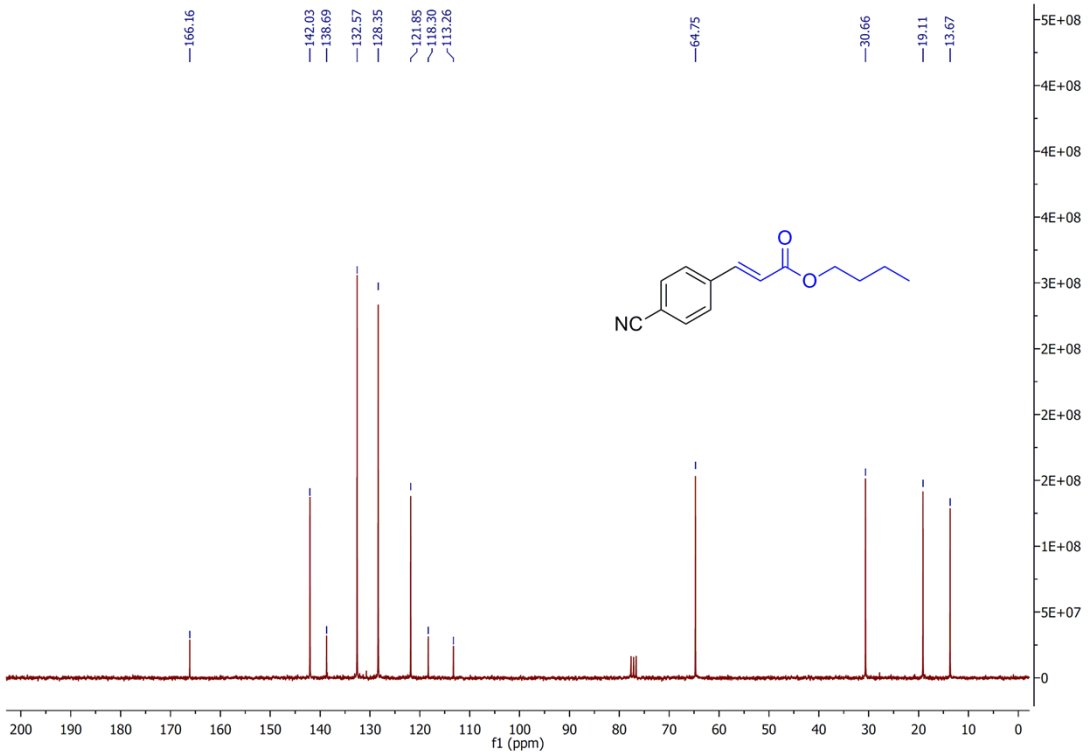
7.22. (E)-butyl 3-(4-methoxyphenyl)acrylate (6q)



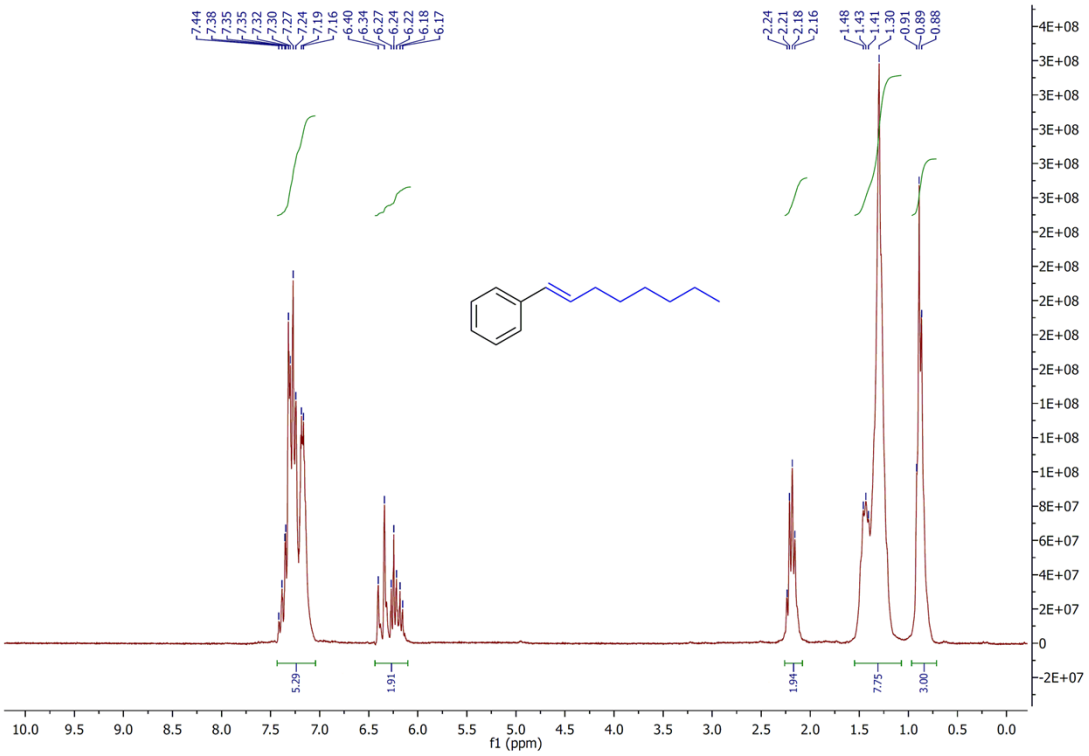


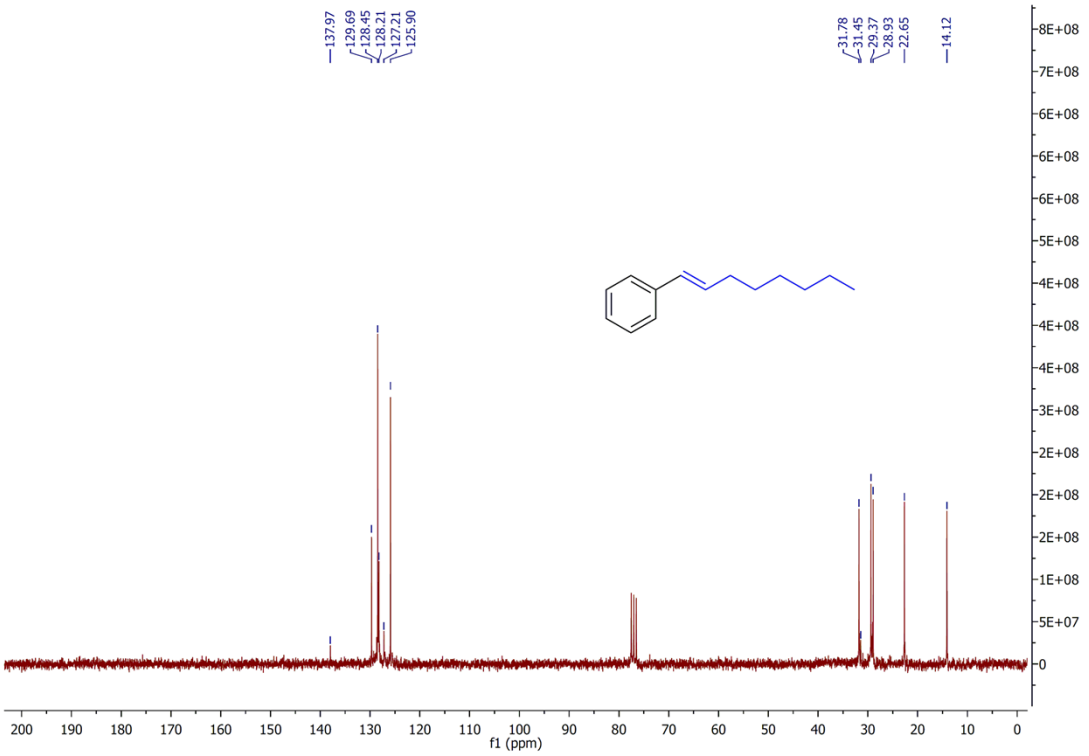
7.23. (E)-butyl 3-(4-cyanophenyl)acrylate (6r)



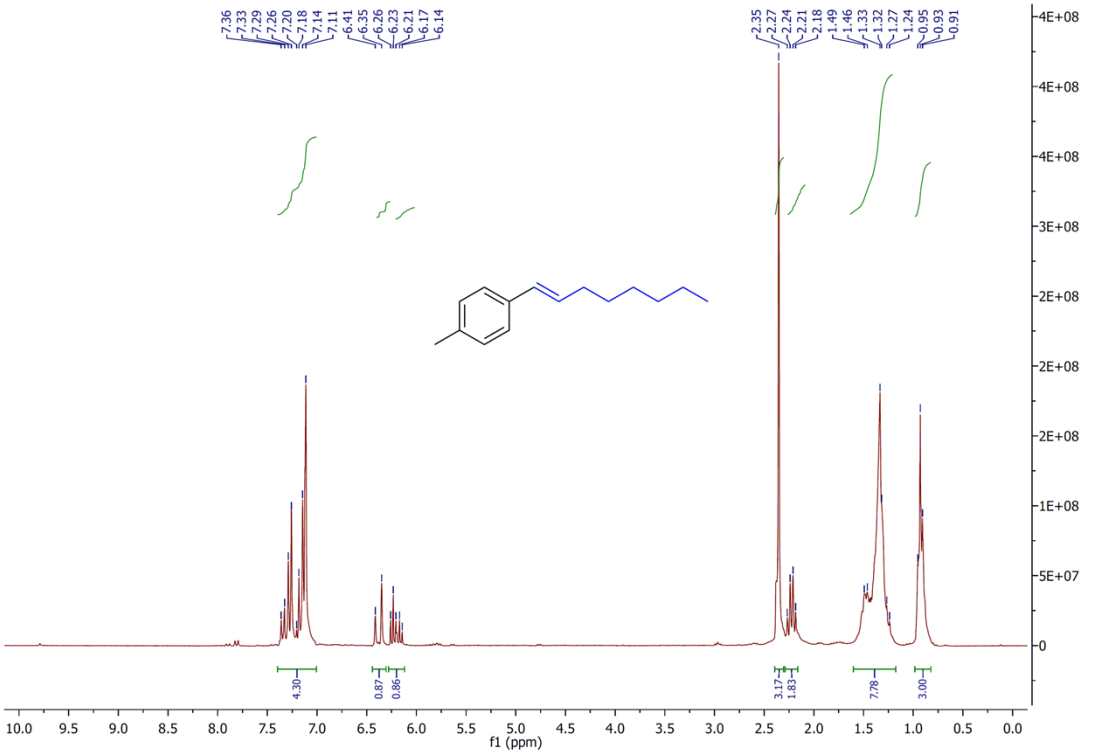


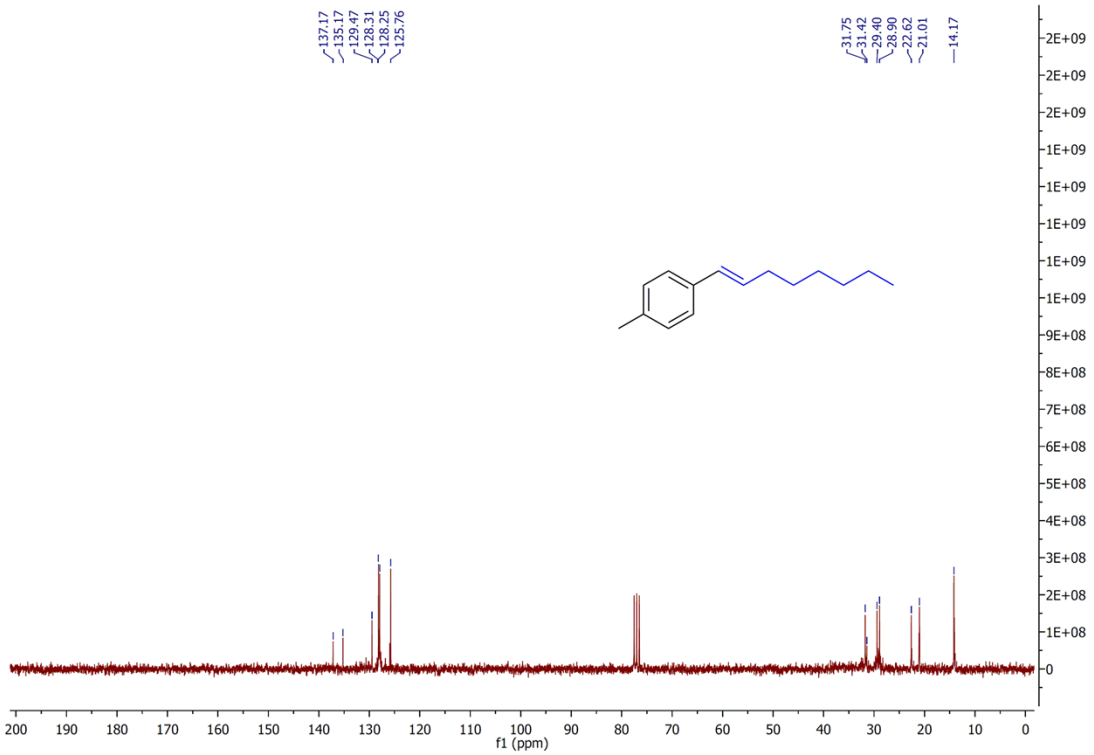
7.24. (E)-oct-1-en-1-ylbenzene (6s)



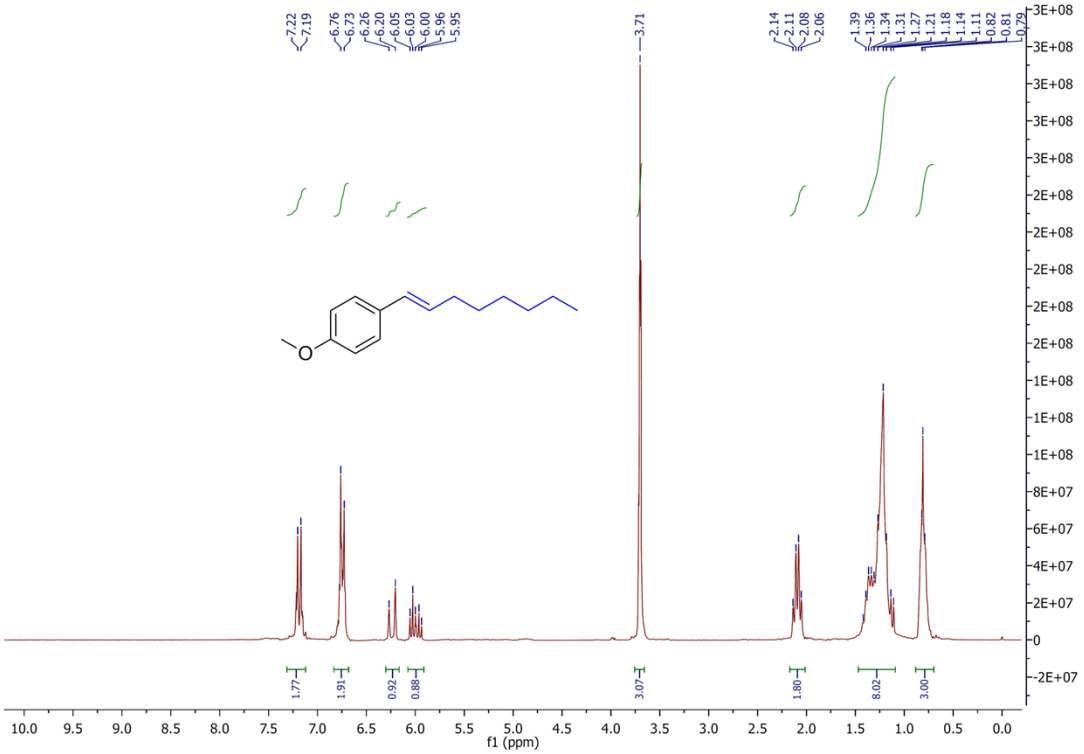


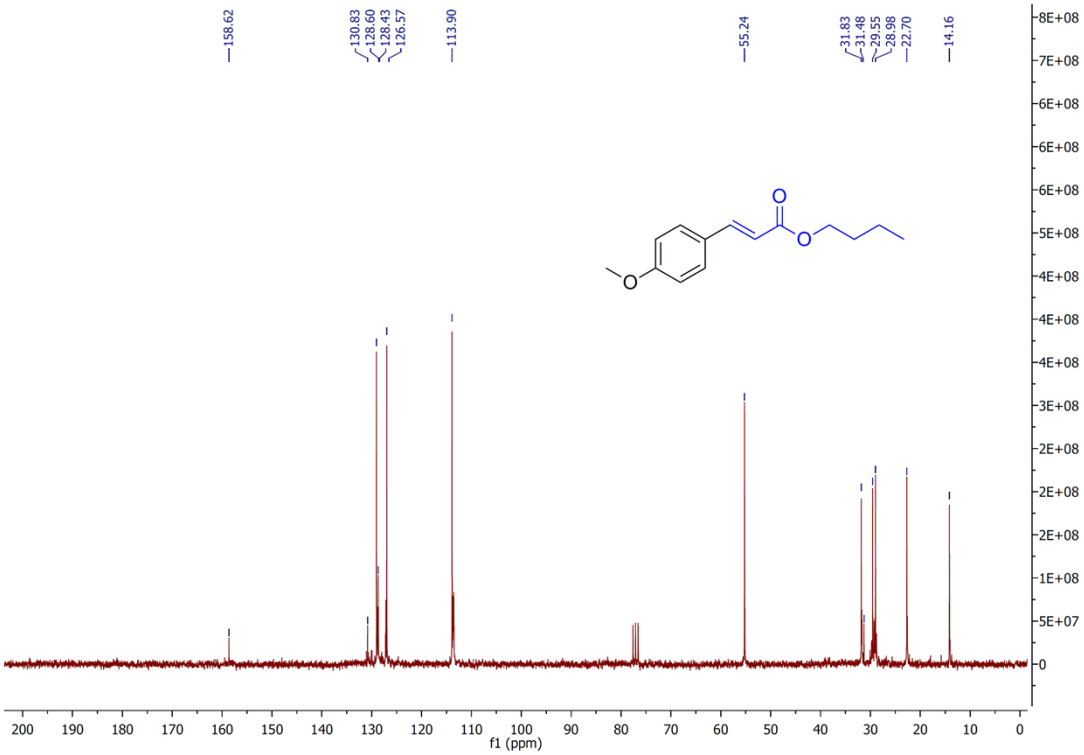
7.25. (E)-1-methyl-4-(oct-1-en-1-yl)benzene (6t)



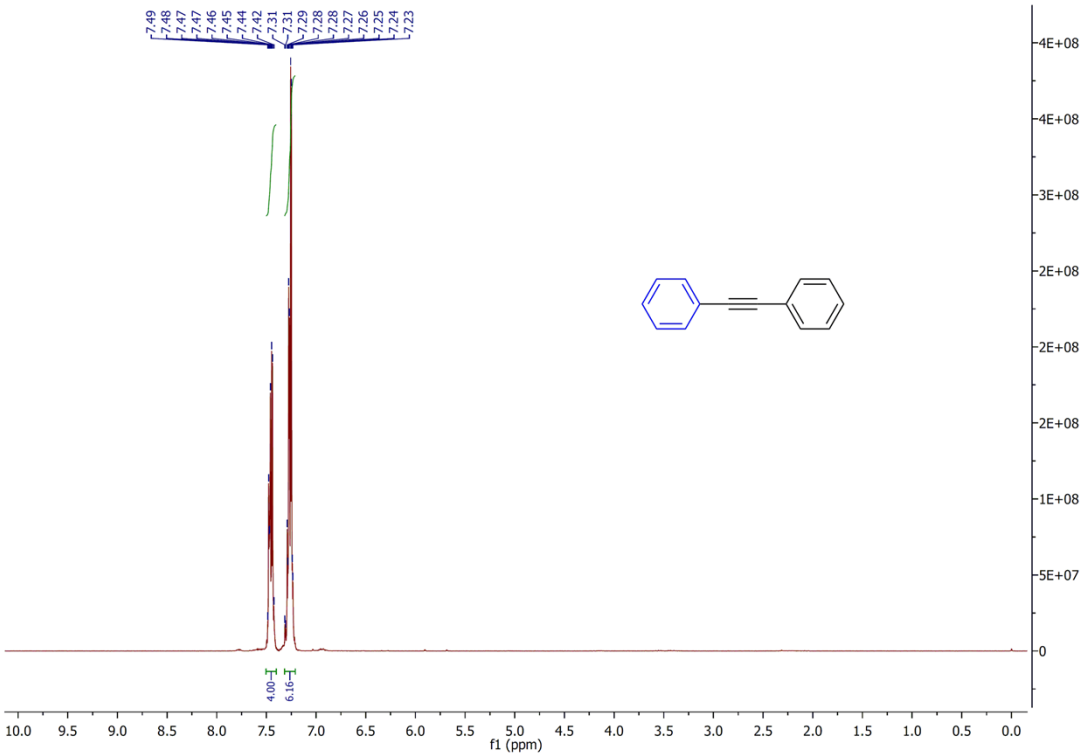


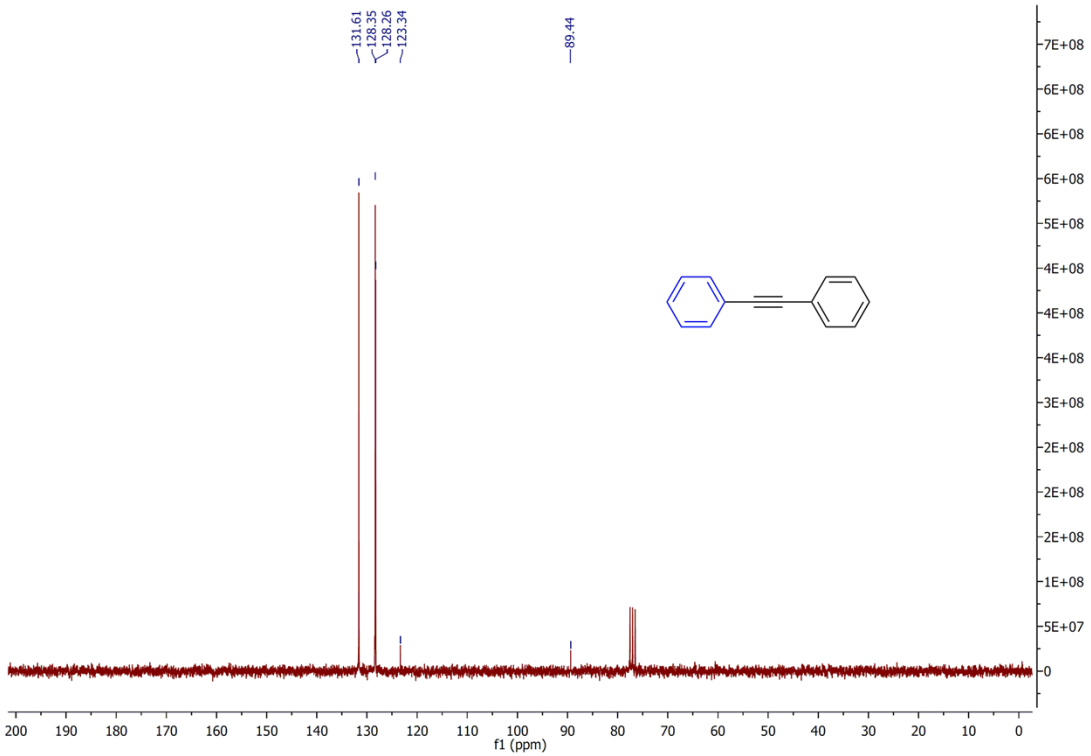
7.26. (E)-1-methoxy-4-(oct-1-en-1-yl)benzene (6u)



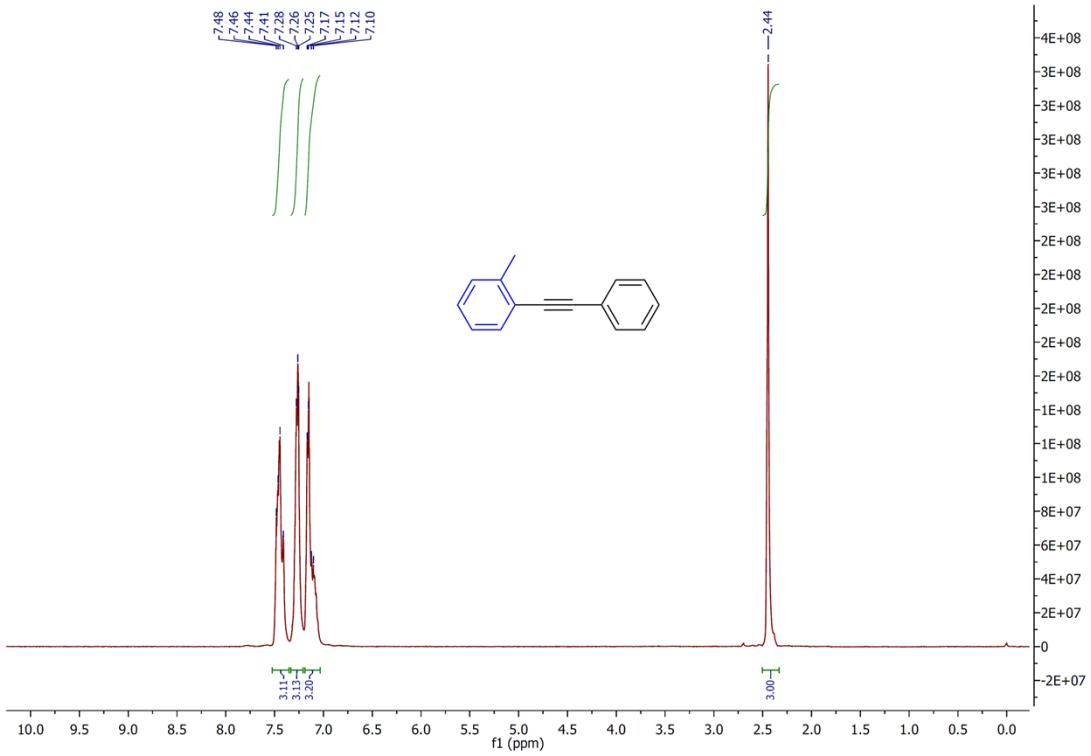


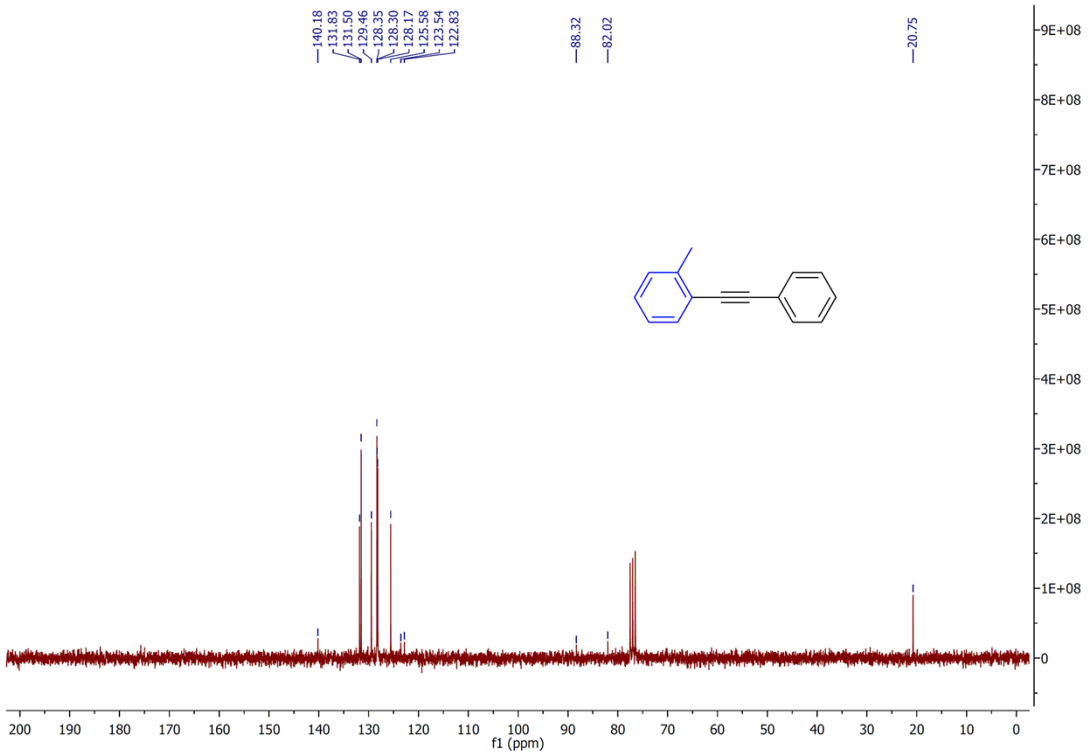
7.27. 1,2-diphenylethyne (8a)



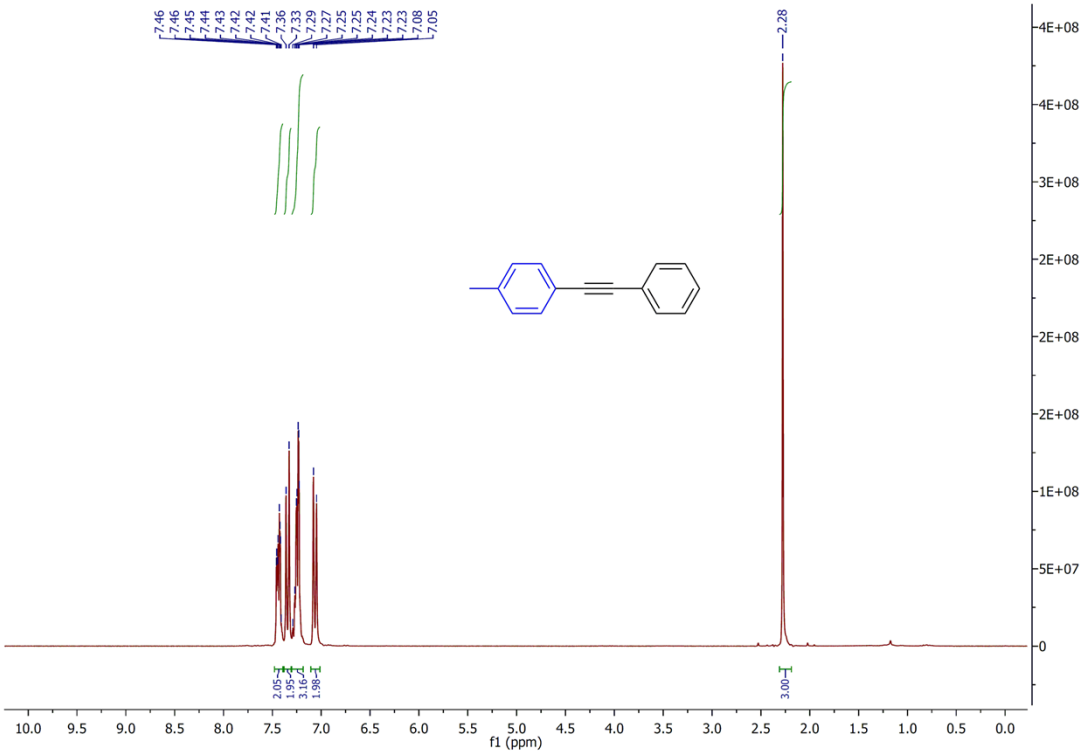


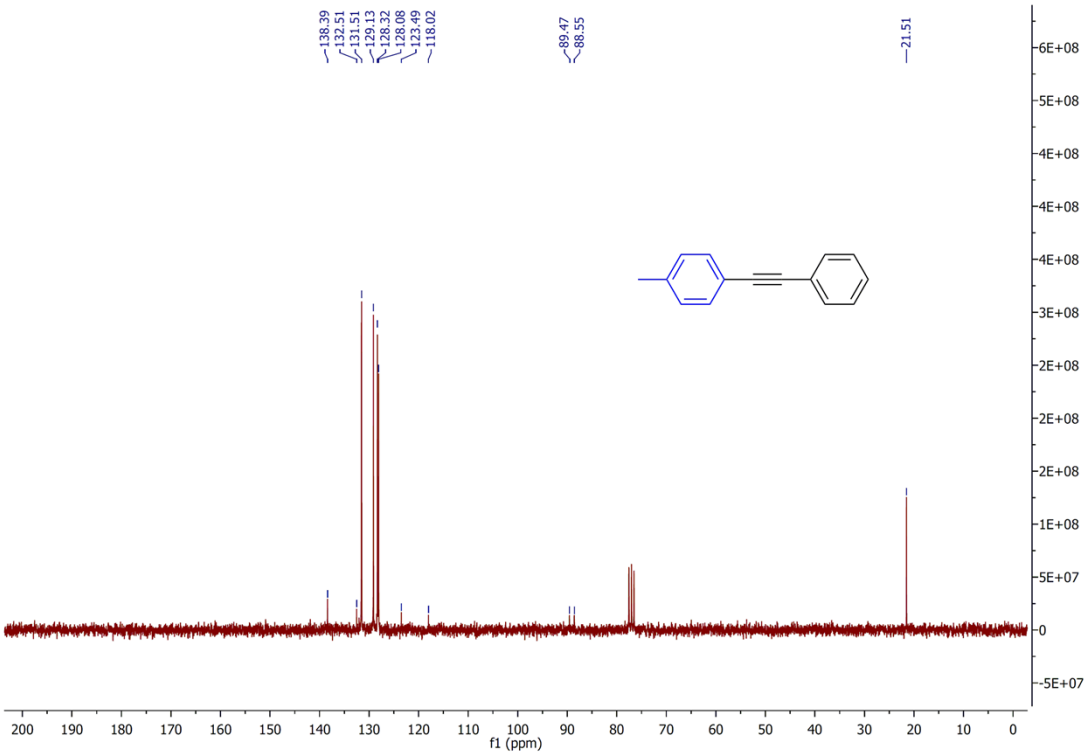
7.28. 1-methyl-2-(phenylethynyl)benzene (8b)



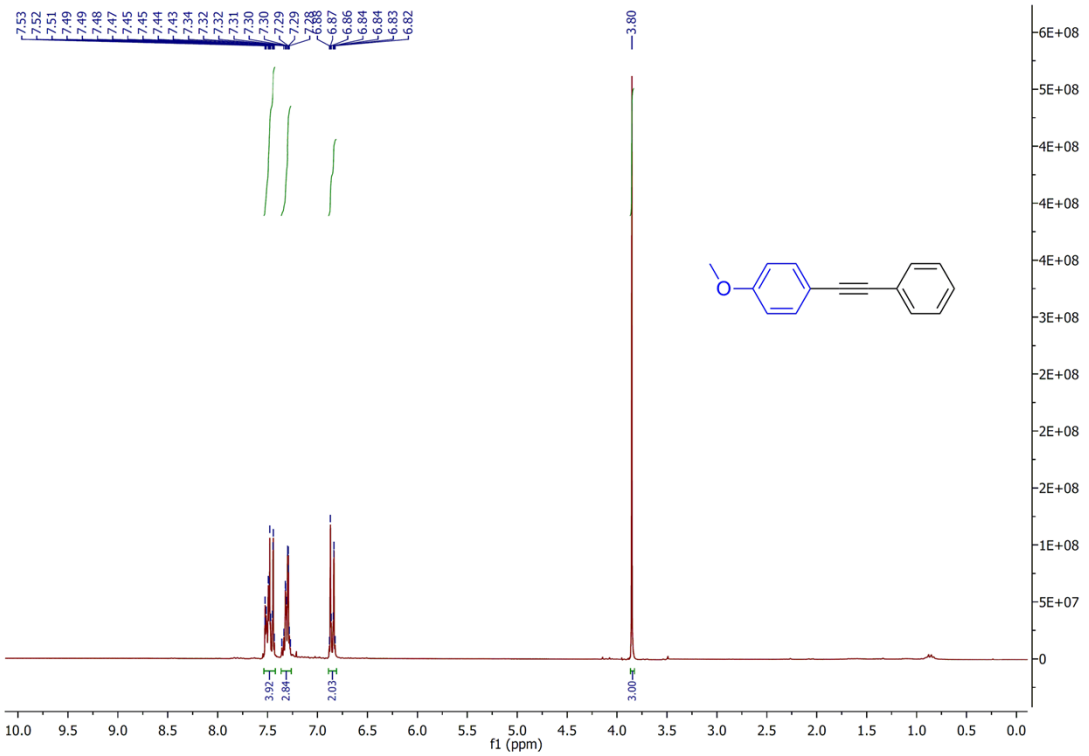


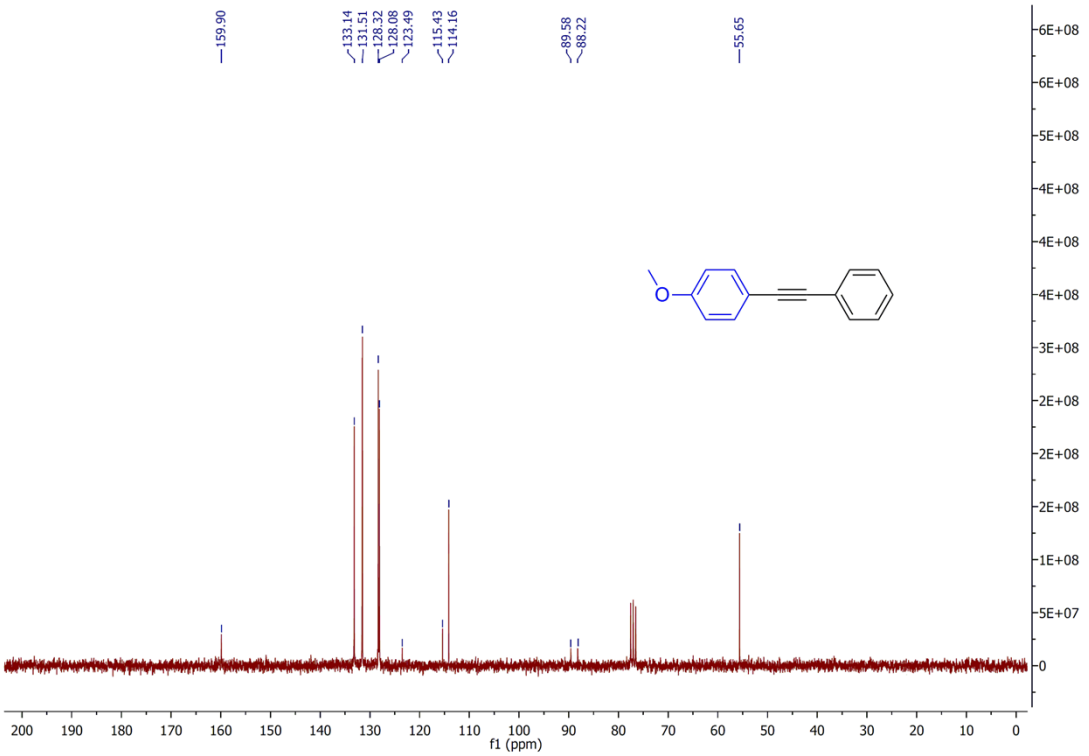
7.29. 1-methyl-4-(phenylethynyl)benzene (8c)



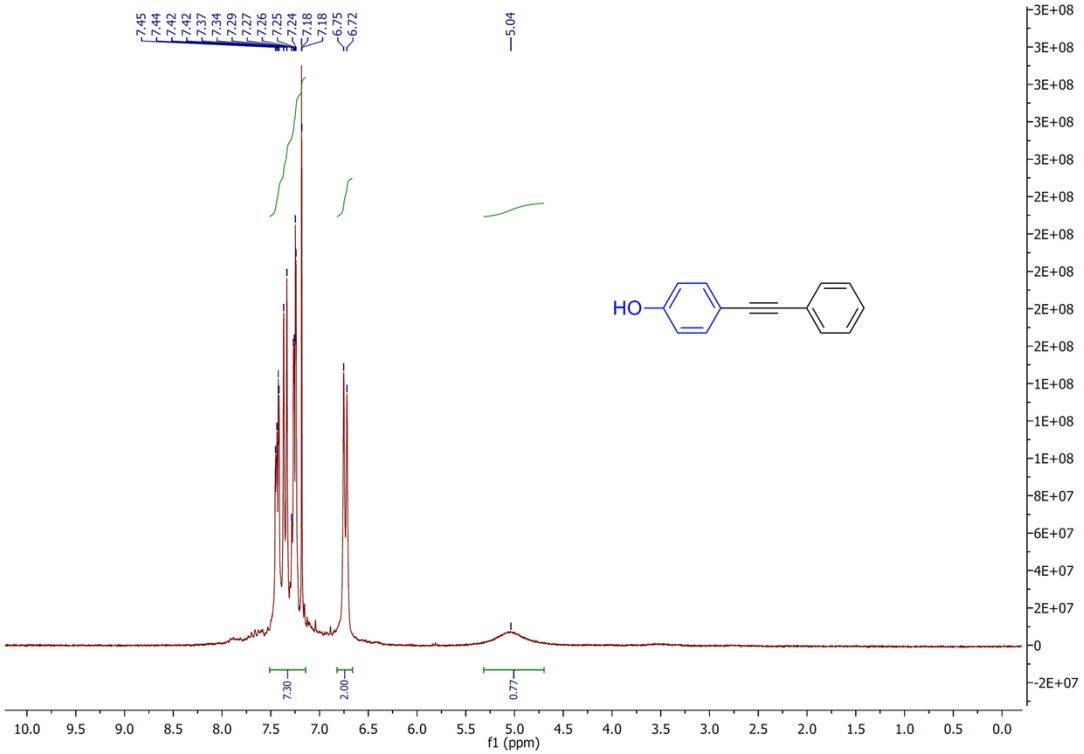


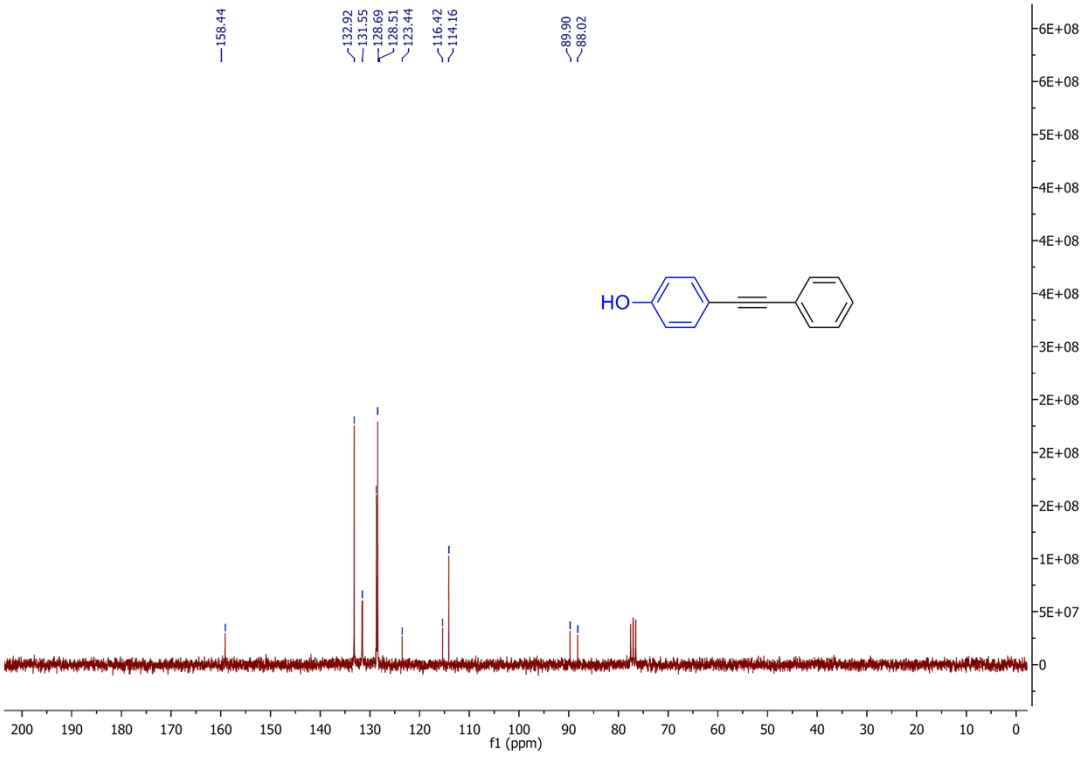
7.30. 1-methoxy-4-(phenylethynyl)benzene (8d)



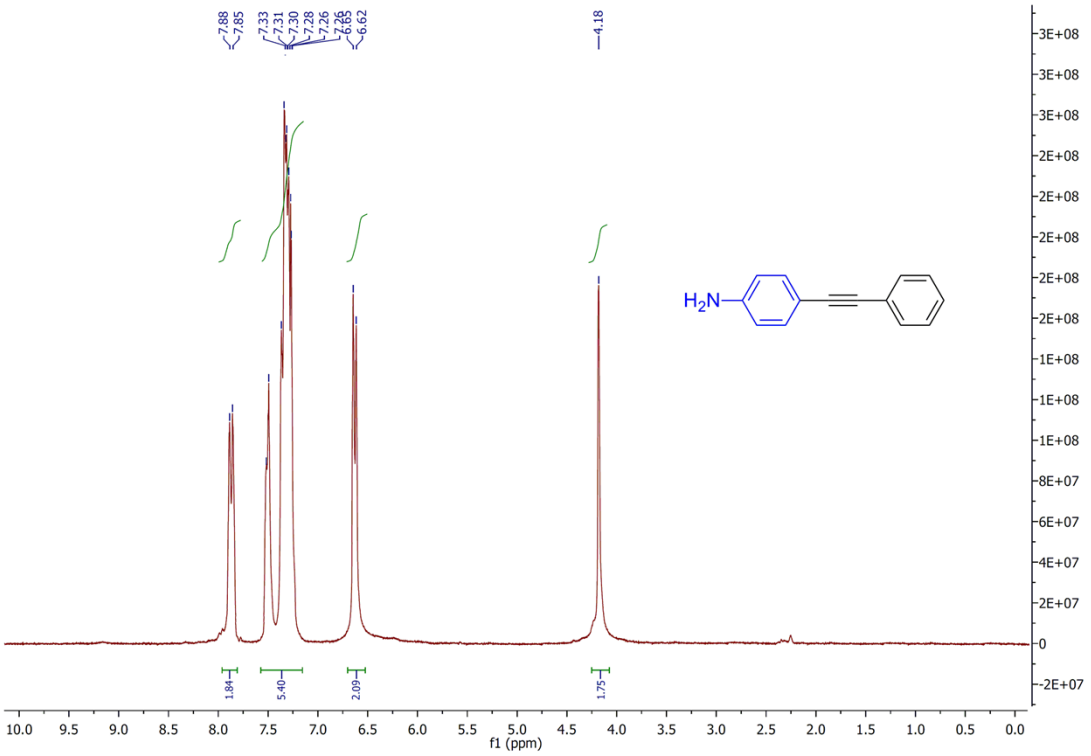


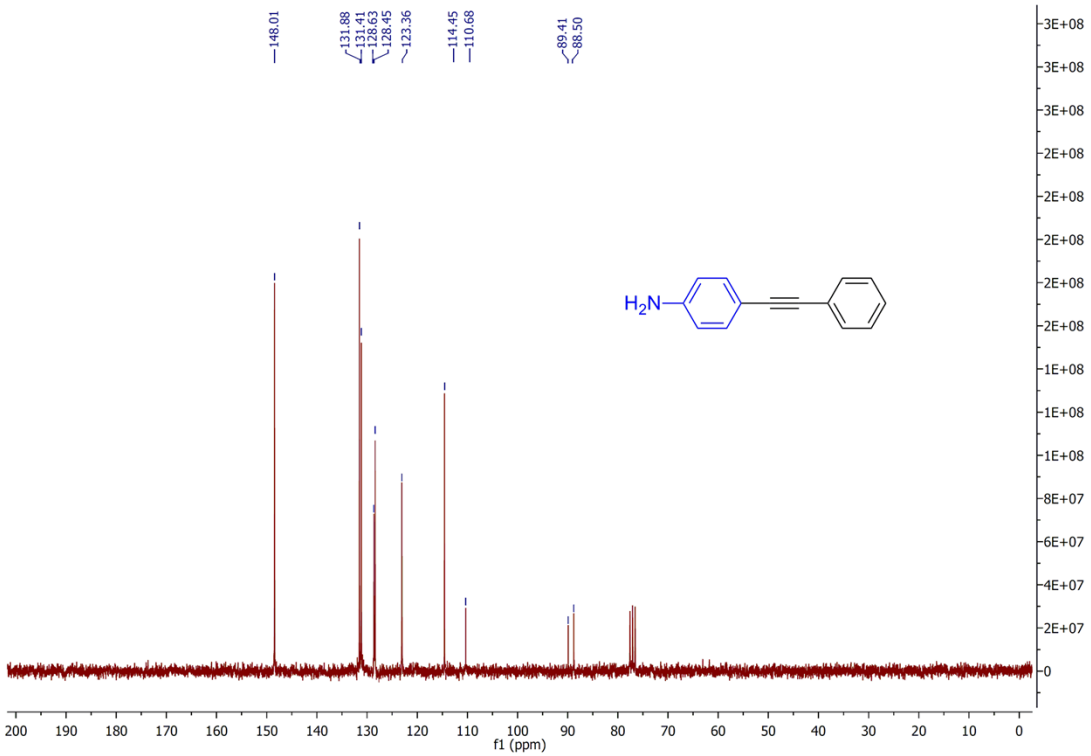
7.31. 4-(phenylethynyl)phenol (8e)



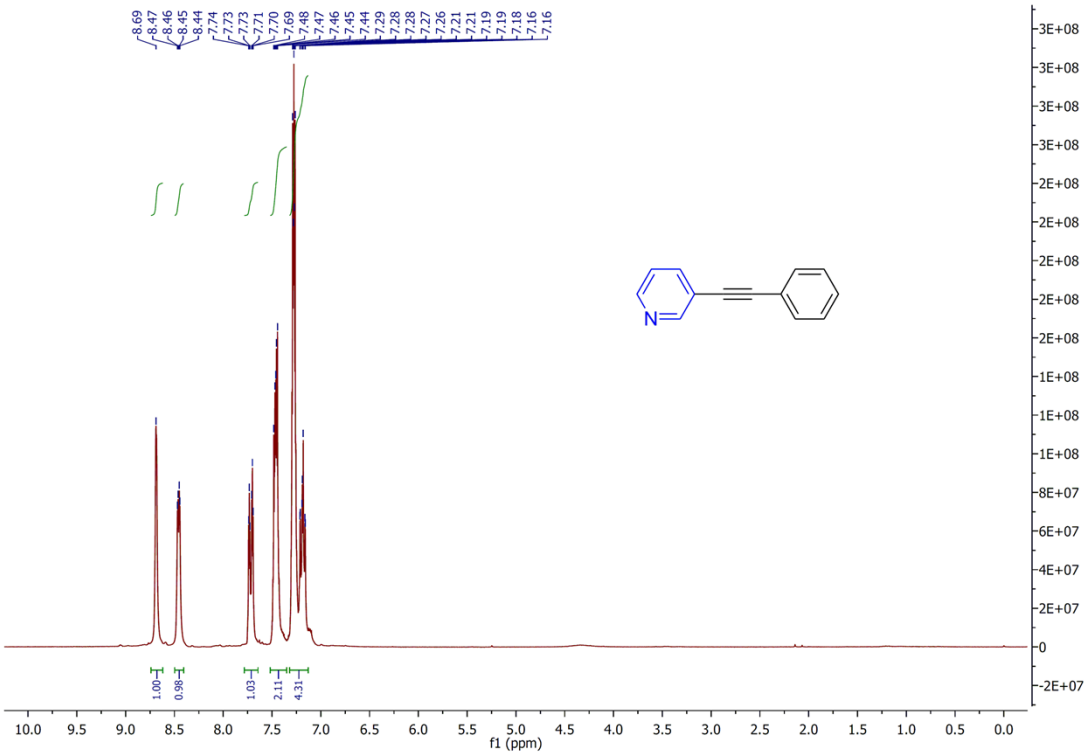


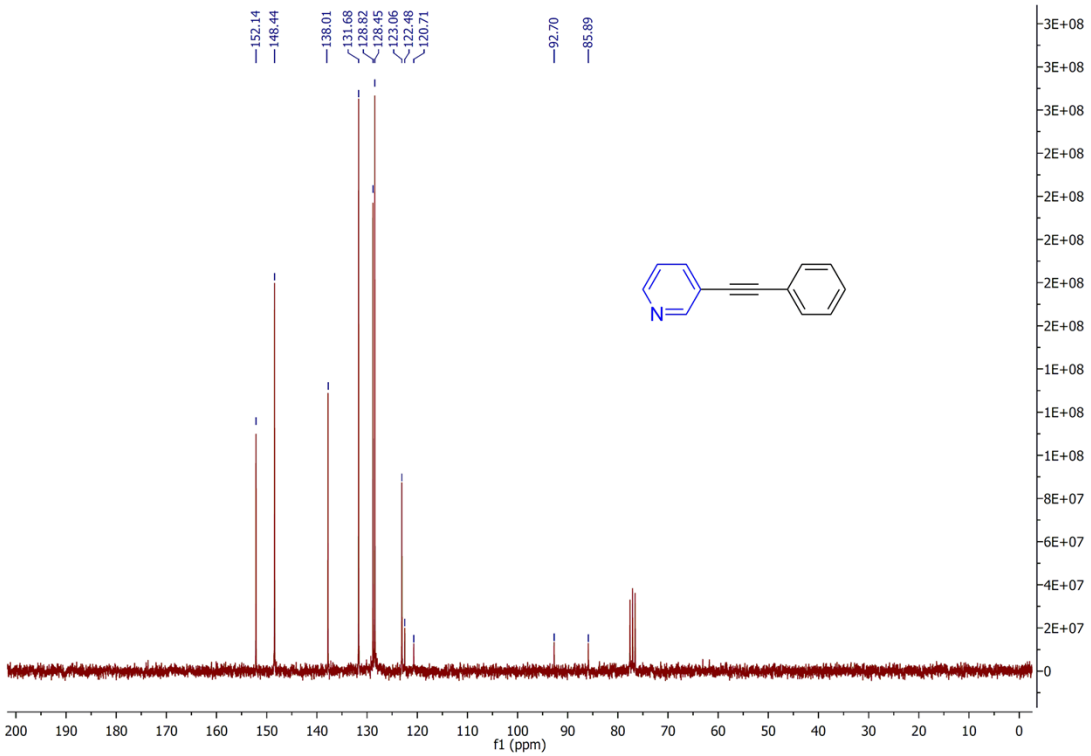
7.32. 4-(phenylethynyl)aniline (8f)



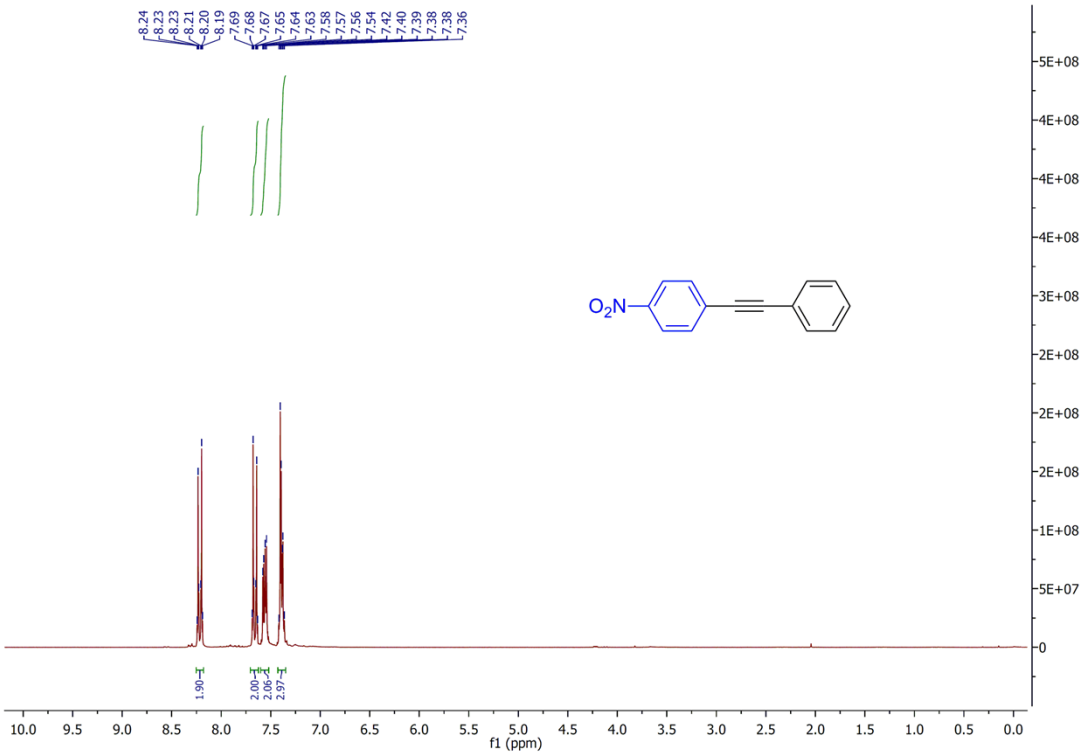


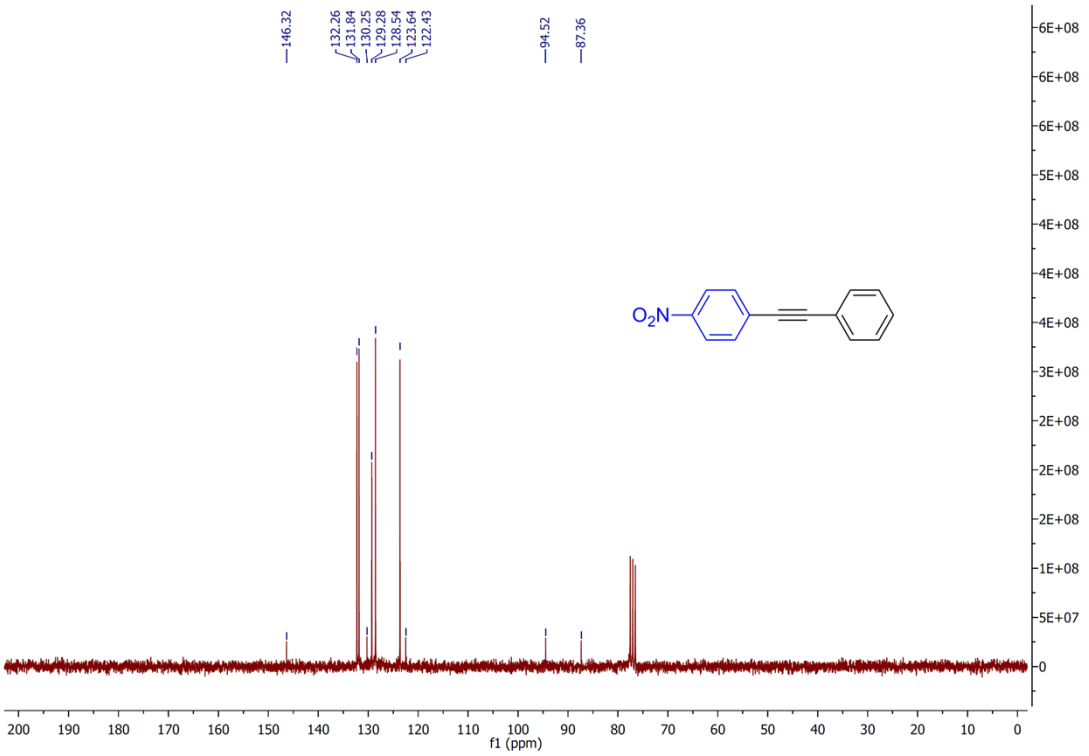
7.33. 3-(phenylethynyl)pyridine (8g)





7.34. 1-nitro-4-(phenylethynyl)benzene (8h)





7.35. 4-(phenylethynyl)benzonitrile (8i)