

Electronic Supplementary Information for

**Electrochemical deoxygenative arylation of aldehydes and
ketones**

Meng Li, Yue Tian, Kunhui Sun, Zhimin Xu, Lifang Tian,* and Yahui Wang*

School of Chemistry and Molecular Engineering, Nanjing Tech University, Nanjing
211816 China

E-mail: tianlifang@njtech.edu.cn, ias_yhwang@njtech.edu.cn.

Contents

1. General information	2
2. Experimental procedures	2
2.1 Electrode materials and dimensions	2
2.2 General procedure for electrochemical deoxygenative arylation of aldehydes/ketones	3
2.3. Procedure for gram-scale synthesis	4
2.4. Optimization of reaction conditions	5
3.Cyclic voltammetry (CV) graphs	7
4. Mechanistic experiment	9
5. Detail descriptions for products	12
6. ^1H NMR, ^{13}C NMR and ^{19}F NMR spectra	32

1. General information

Reagents were purchased at the highest commercial quality grade and used without further purification, unless otherwise stated. Yields refer to chromatographically and spectroscopically (^1H NMR) homogeneous material, unless otherwise stated. Reactions were monitored by thin layer chromatography (TLC) carried out on 0.25 mm E. Merck silica plates (60F-254), using UV light (254 nm) and TLC stain with anisaldehyde-sulfuric acid for visualization. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (b.p. 60-90 °C). ^1H and ^{13}C NMR data were recorded with Bruker (400 MHz) or Jeol (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ (ppm):) were reported in ppm and coupling constants (J) in Hz. And they were recorded relative to tetramethylsilane (0 ppm for ^1H), CDCl_3 (77.0 ppm for ^{13}C). High resolution mass spectra (HRMS) were measured on a TOF mass spectrometer.

2. Experimental procedures

2.1 Electrode materials and dimensions

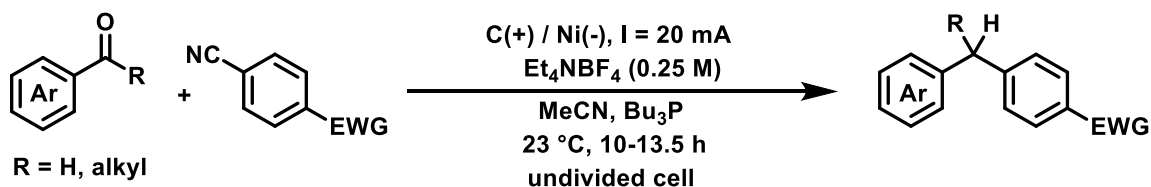
The instrument for undivided electrolysis was IKA[®] ElectraSyn 2.0 with carousel. The electrodes used in IKA[®] ElectraSyn 2.0 were purchased from IKA Company. The anodic electrode was the stainless steel electrode (3.0 cm \times 0.8 cm \times 0.2 cm) (3.0 cm was the height of the electrode immersed in the solution) and the cathodic electrode was the carbon plate (3.0 cm \times 0.8 cm \times 0.2 cm).



Figure S1. IKA[®] ElectraSyn 2.0

Cyclic voltammograms were recorded on an electrochemical workstation CS150H (CorrTest[®]). A steady glassy carbon disk electrode (3 mm in diameter) was used as the working electrode; a platinum plate was used as the counter electrode; the reference was an Ag/AgNO₃ electrode with (0.01 M) AgNO₃ in acetonitrile.

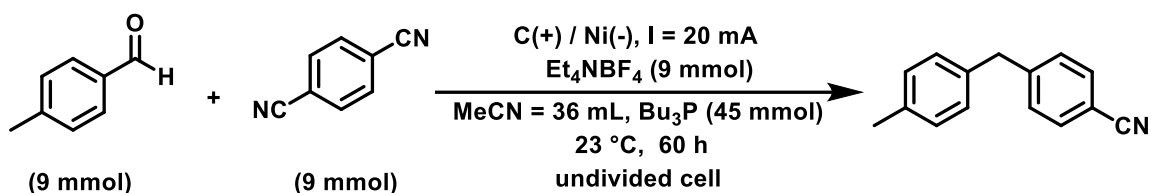
2.2 General procedure for electrochemical deoxygenative arylation of aldehydes/ketones



The electrolysis was carried out in the electrolysis cell of IKA[®] ElectraSyn 2.0. The anodic electrode was the carbon plate electrode (3.0 cm × 0.8 cm × 0.2 cm) and the cathodic electrode was the stainless steel plate (3.0 cm × 0.8 cm × 0.2 cm). Aldehydes or ketones derivatives (1.0 mmol), 1,4-dicyanobenzene (128 mg, 1.0 mmol), Et₄NBF₄ (217 mg, 1.0 mmol), tributylphosphine (1011.6 mg, 5.0 mmol)

and MeCN = 4.0 mL were added to an oven-dried undivided cell (6 mL) equipped with a stirring bar (the order of the addition did not affect the result). The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10-13.5 h. (The specific reaction time depends on the complete consumption of the substrates.). When the reaction was finished, the solvent was evaporated under vacuum and the crude material was purified by column chromatography or preparative TLC to furnish the desired product.

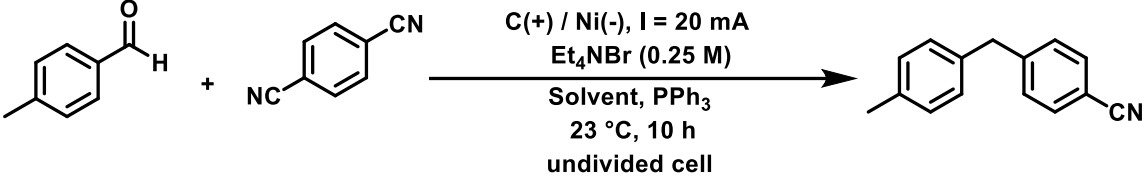
2.3. Procedure for gram-scale synthesis



The electrolysis was carried out in the electrochemical workstation CS150H (CorrTest[®]). The anodic electrode was the carbon plate electrode (3.0 cm × 0.8 cm × 0.2 cm) and the cathodic electrode was the stainless steel plate (3.0 cm × 0.8 cm × 0.2 cm). According to General Procedure, starting p-tolualdehyde (1.08 g, 9.0 mmol) and 1,4-dicyanobenzene (1.15 g, 9 mmol), Et₄NBF₄ (1.95 g, 9 mmol), Tributylphosphine (9.09 g, 45 mmol) were added to an oven-dried undivided cell with MeCN (36 mL) equipped with a stirring bar. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 60 h. Then the reaction mixture was evaporated under vacuum, and the crude material was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 42% yield (782 mg).

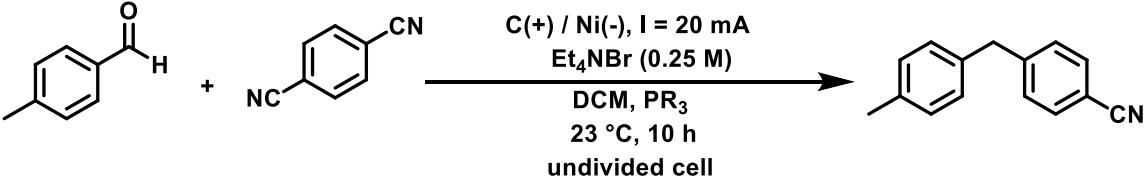
2.4. Optimization of reaction conditions

Table S1. Optimization of the solvent.

		
Entry	Solvent	yield ^a
1	DMSO = 4 mL	54%
2	DMA = 4 mL	<5%
3	MeOH = 4 mL	No
4	THF:DMA = 3:1 mL	50%
5	DCM = 4 mL	50%

^aIsolated yields after chromatography

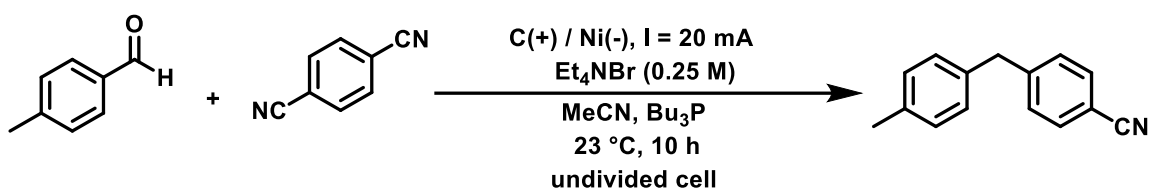
Table S2. Optimization of the PR₃

		
Entry	PR ₃	yield ^a
1	Bu ₃ P	60%
2	PCy ₃	<5%

3	$[\text{N}(\text{CH}_3)_2]\text{P}$	13%
4	$(\text{PhO})_3\text{P}$	32%
5	$(4\text{-F-Ph})_3\text{P}$	44%
6	PPh_3	50%

^aIsolated yields after chromatography

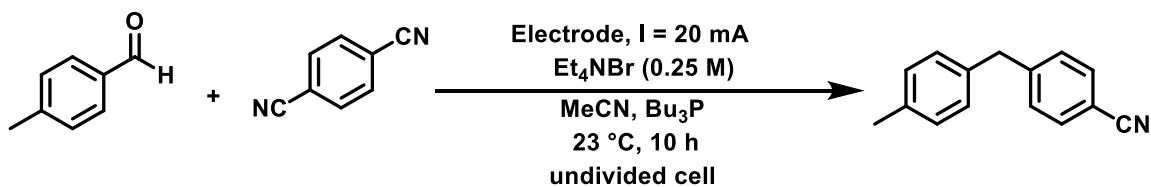
Table S3. Optimization of the equivalent of Bu_3P



Entry	Bu_3P (X equiv)	yield ^a
1	2.0 equiv	60%
2	3.0 equiv	63%
3	4.0 equiv	54%
4	5.0 equiv	71%

^aIsolated yields after chromatography

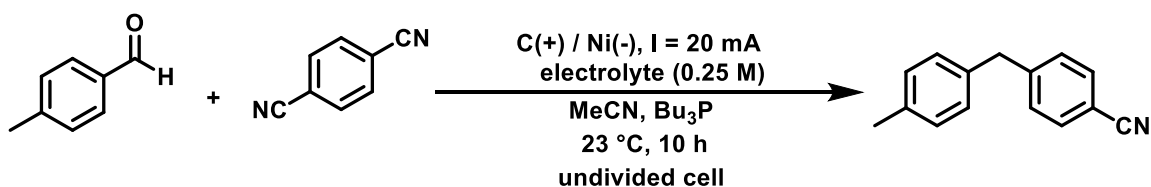
Table S4. Optimization of the electrode material



Entry	Electrode	yield ^a
1	C(+) / Ni(-)	71%
2	C(+) / C(-)	67%
3	C(+) / Pt(-)	69%
4	C(+) / SST(-)	68%

^aIsolated yields after chromatography

Table S5. Optimization of the electrolyte



Entry	Electrolyte	Yield ^a
1	Et ₄ NI	59%
2	Et ₄ NCl	62%
3	Et ₄ NPF ₆	52%
4	Et ₄ NBF ₄	75%

^aIsolated yields after chromatography

3.Cyclic voltammetry (CV) graphs

Cyclic voltammetry was performed in a three-electrode cell under air at room temperature. A steady glassy carbon disk electrode (3 mm in diameter) was used

as the working electrode; a platinum plate was used as the counter electrode; the reference was an Ag/AgNO₃ electrode with (0.01 M) AgNO₃ in MeCN.

Cyclic voltammograms recorded in (0.1 M) Et₄NBF₄ - MeCN solution (Figure S2.): scan rate: 100 mV s⁻¹; starting potential: 0 V; glass carbon (3 mm diameter, Working Electrode); platinum plate (Counter Electrode); Ag/AgNO₃ (0.01 M) AgNO₃ in MeCN, Reference Electrode); Concentrations: p-tolualdehyde (1a) (0.8mmol / 8 mL MeCN), 1,4-dicyanobenzene (2a) (0.8 mmol / 8 mL MeCN).

Cyclic voltammograms recorded in (0.1 M) Et₄NBF₄ - MeCN solution (Figure S3.): scan rate: 100 mV s⁻¹; starting potential: 0 V; glass carbon (3 mm diameter, Working Electrode); platinum plate (Counter Electrode); Ag/AgNO₃ (0.01 M) AgNO₃ in MeCN, Reference Electrode); Concentrations: tributylphosphine (0.8 mmol / 8 mL MeCN).

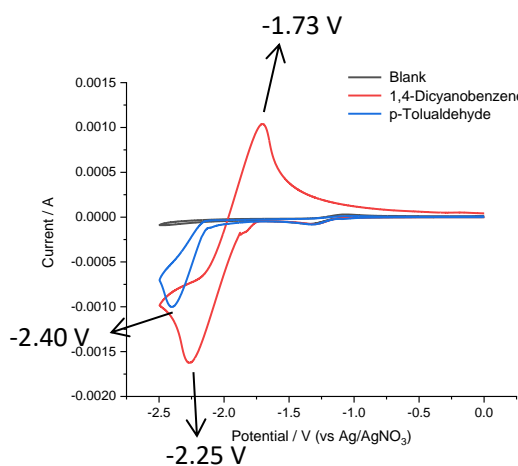


Figure S2.

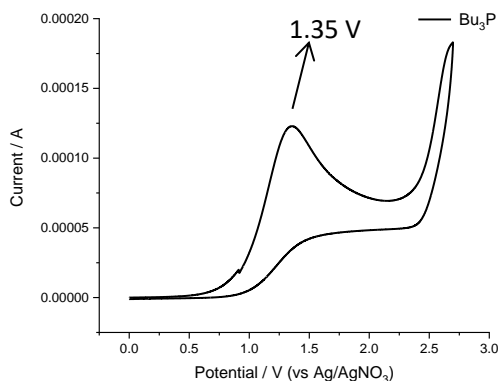
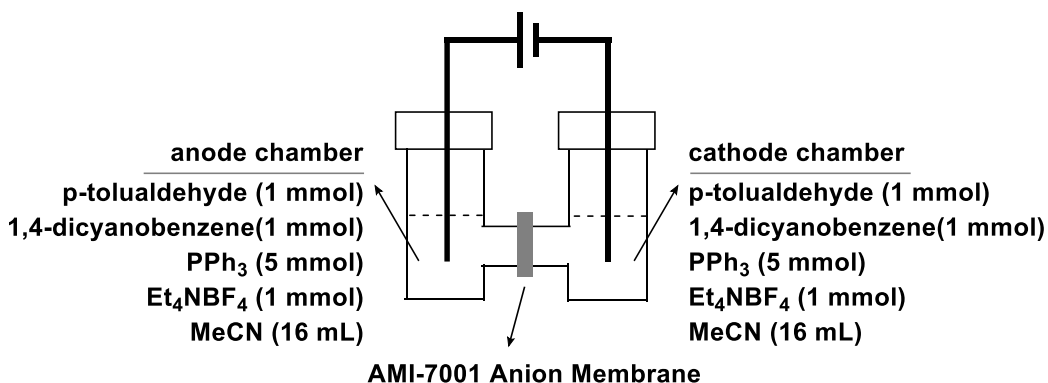
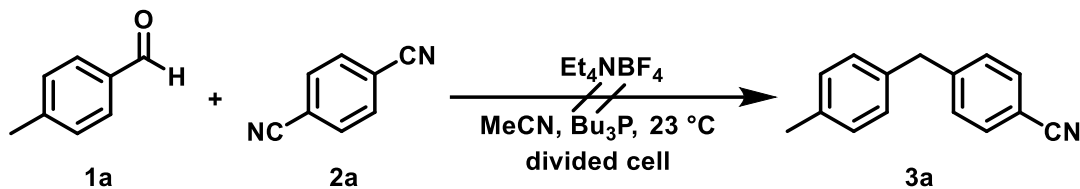


Figure S3.

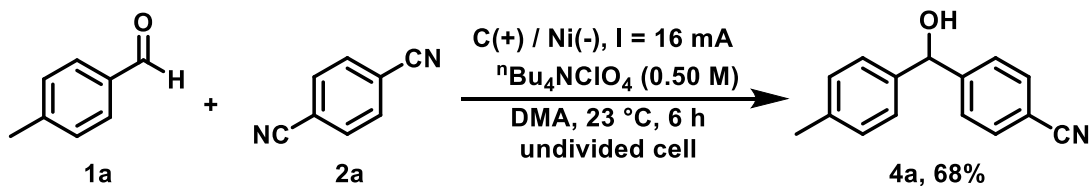
4. Mechanistic experiment

4.1. Experiment in the divided cell



p-tolualdehyde (1.0 mmol), 1,4-dicyanobenzene (1.0 mmol) and PPh_3 (5 mmol) were added into the anodic chamber, while the cathodic chamber was added with the same substances. Subsequently, MeCN (16 ml) containing Et_4NBF_4 (1.0 mmol) was added to the anodic chamber and the cathodic chamber, respectively. Then the solution was stirred and electrolysed at a constant current of 5 mA at room temperature ($23\text{ }^\circ\text{C}$) for 4 h. When the reaction was finished, the solution was treated by the standard procedure.

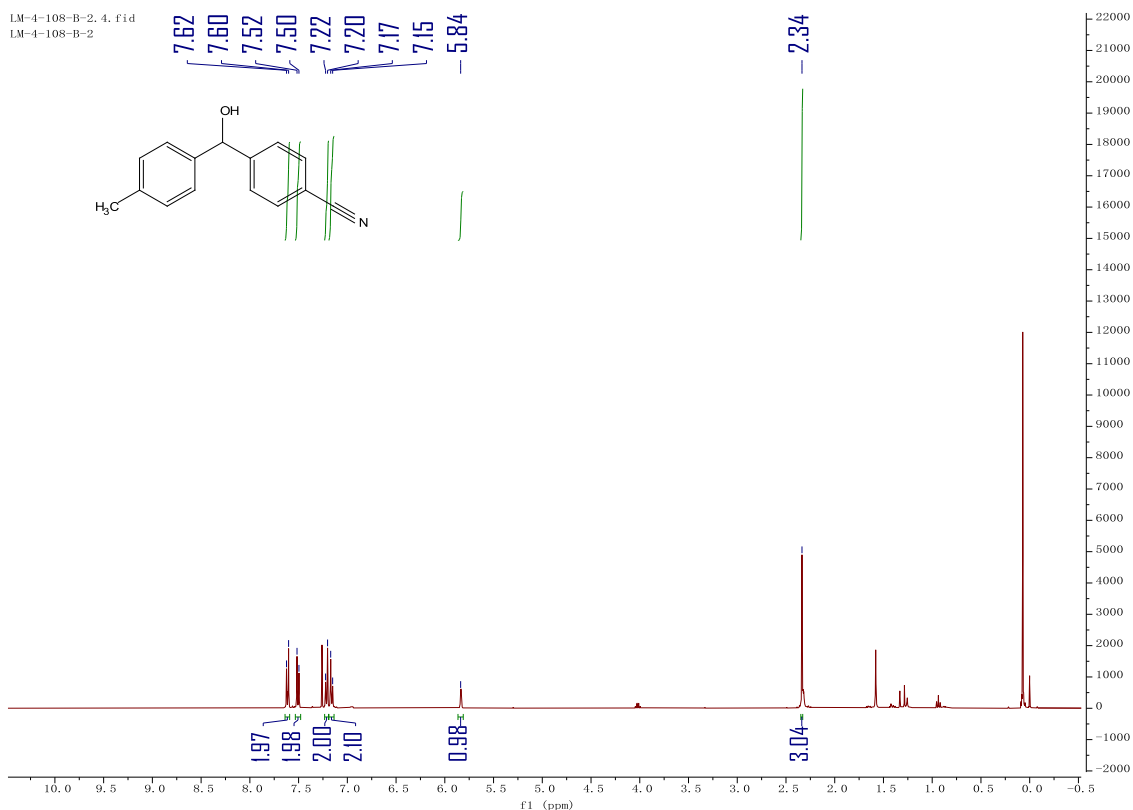
4.2. Step-by-step response



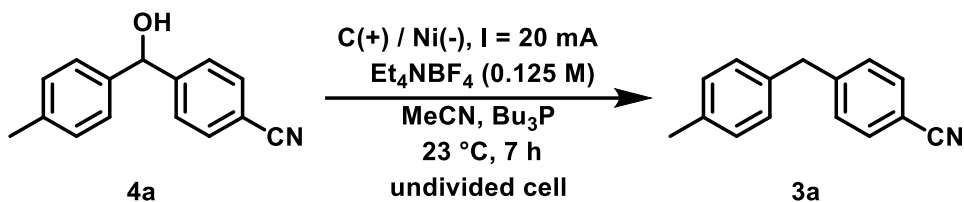
The starting p-tolualdehyde (240mg, 2.0 mmol), 1,4-dicyanobenzene (256mg, 2.0 mmol) and $n\text{Bu}_4\text{NClO}_4$ (684 mg, 2.0 mmol) were added to an oven-dried undivided cell with DMA (4 mL) equipped with a stirring bar. The reaction mixture was stirred and electrolyzed at a constant current of 16 mA at room temperature for 6 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 3:1) to afford the reaction intermediate 4-(hydroxy(p-tolyl)methyl)benzonitrile as a colorless oil in 68% yield (303 mg).

^1H NMR (400 MHz, Chloroform- d) δ 7.61 (d, J = 8.5 Hz, 2H), 7.51 (d, J = 7.9 Hz, 2H), 7.21 (d, J = 8.2 Hz, 2H), 7.16 (d, J = 8.0 Hz, 2H), 5.84 (s, 1H), 2.34 (s, 3H).

NMR data matched those reported in the literature¹

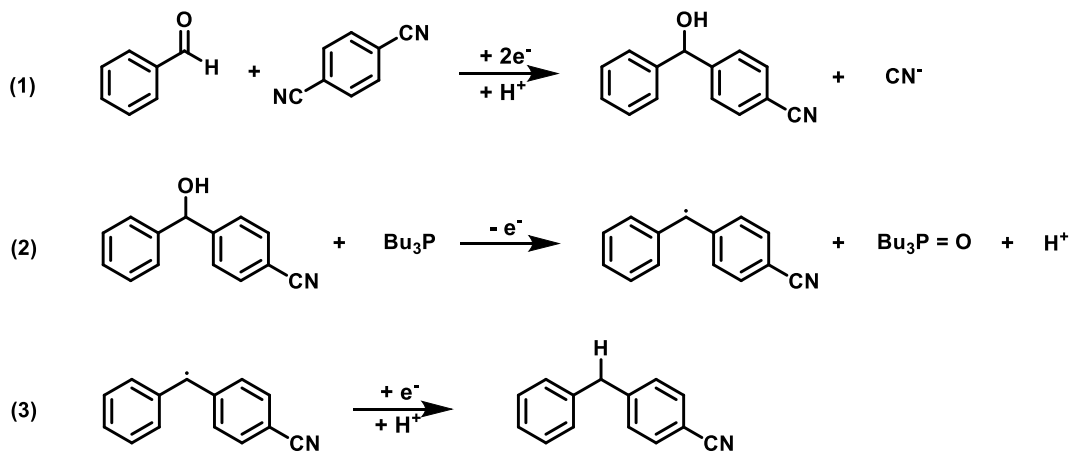


¹ S. Zhang, L. Li, J. Li, J. Shi, K. Xu, W. Gao, L. Zong, G. Li and M. Findlater, *Angew. Chem. Int. Ed.*, 2021, **60**, 7275–7282.

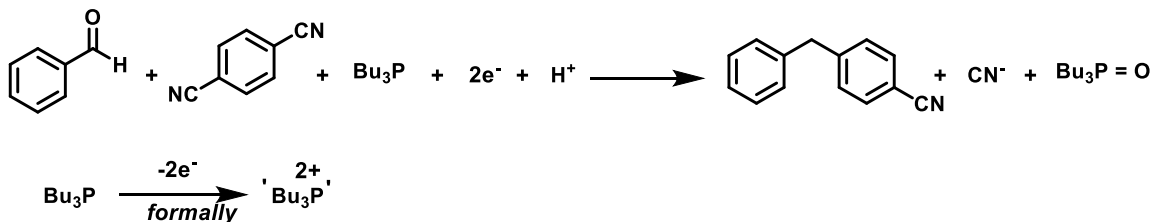


Then 4-(hydroxy(p-tolyl)methyl)benzonitrile (111.5 mg, 0.5 mmol), tributylphosphine (303 mg, 3.0 mmol) and Et₄NBF₄ (108.5 mg, 0.5 mmol) were added to an oven-dried undivided cell with MeCN (4 mL) equipped with a stirring bar. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 7 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 62% yield (64 mg).

4.3. Speculation about the reaction process

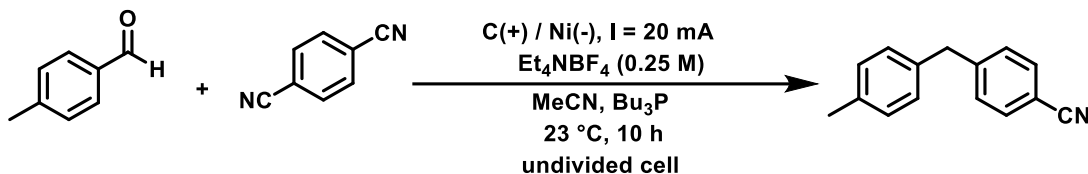


(1) + (2) + (3) = Total:



5. Detail descriptions for products

4-(4-methylbenzyl)benzonitrile (3a)



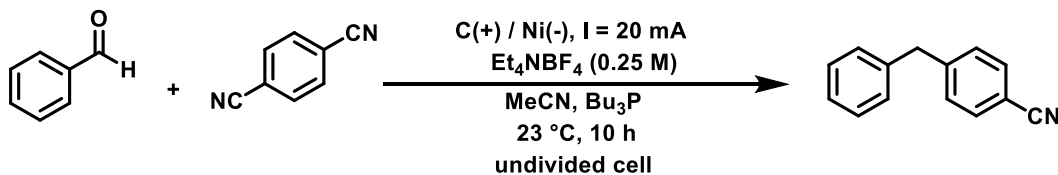
The starting p-tolualdehyde (120 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 75% yield (155 mg).

¹H NMR (400 MHz, Chloroform-d) δ 7.57 (d, J = 8.2 Hz, 2H), 7.30 (d, J = 8.1 Hz, 2H), 7.16 (d, J = 7.8 Hz, 2H), 7.09 (d, J = 7.6 Hz, 2H), 4.01 (s, 2H), 2.36 (s, 3H).

¹³C NMR (101 MHz, Chloroform-d) δ 147.23, 136.45, 136.36, 132.39, 129.71, 129.59, 128.99, 119.20, 110.00, 41.68, 21.18.

NMR data matched those reported in the literature²

4-benzylbenzonitrile (3b)

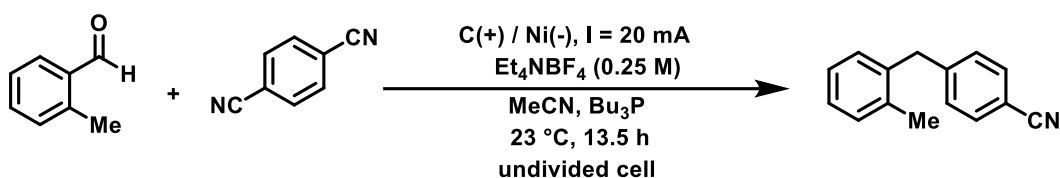


The starting benzaldehyde (106 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0

² D. Zhang, Z. Xu, T. Tang, L. Le, C. Wang, N. Kambe and R. Qiu, *Org. Lett.*, 2022, **24**, 3155–3160.

mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 75% yield (145 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.57 (d, J = 8.2 Hz, 2H), 7.37 – 7.23 (m, 5H), 7.19 (d, J = 7.3 Hz, 2H), 4.05 (s, 2H). **¹³C NMR** (101 MHz, Chloroform-d) δ 146.95, 139.56, 132.48, 129.84, 129.17, 128.97, 126.87, 119.22, 110.18, 42.16. NMR data matched those reported in the literature³

4-(2-methylbenzyl)benzonitrile (3c)

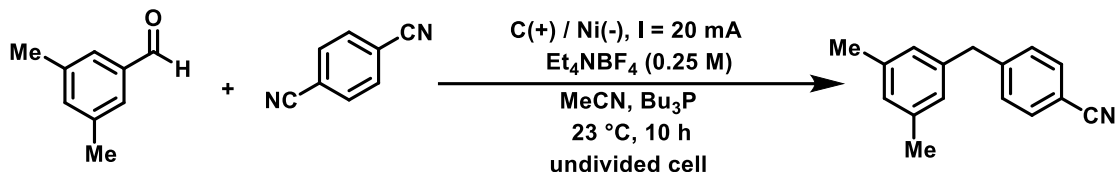


The starting 2-methylbenzaldehyde (120 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 13.5 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 57% yield (118 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.56 (d, J = 8.2 Hz, 2H), 7.27 – 7.17 (m, 5H), 7.13 – 7.08 (m, 1H), 4.05 (s, 2H), 2.22 (s, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 146.48, 137.47, 136.84, 132.49, 130.87, 130.32, 129.67, 127.37, 126.56, 119.30, 110.15, 39.86, 19.90.

NMR data matched those reported in the literature²

³ T. Wang, S. Yang, S. Xu, C. Han, G. Guo and J. Zhao, *RSC Adv.*, 2017, 7, 15805-15808.

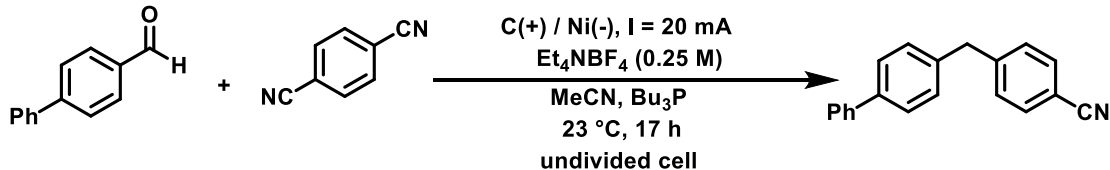
4-(3,5-dimethylbenzyl)benzonitrile (3d)



The starting 3,5-dimethylbenzaldehyde (134 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 72% yield (160 mg). **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 6.90 (s, 1H), 6.80 (s, 2H), 3.96 (s, 2H), 2.30 (s, 6H). **¹³C NMR** (101 MHz, Chloroform-*d*) δ 147.26, 139.47, 138.52, 132.48, 129.84, 128.51, 127.02, 119.32, 110.09, 42.09, 21.50.

NMR data matched those reported in the literature⁴

4-([1,1'-biphenyl]-4-ylmethyl)benzonitrile (3e)



The starting 4-biphenylcarboxaldehyde (182 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and

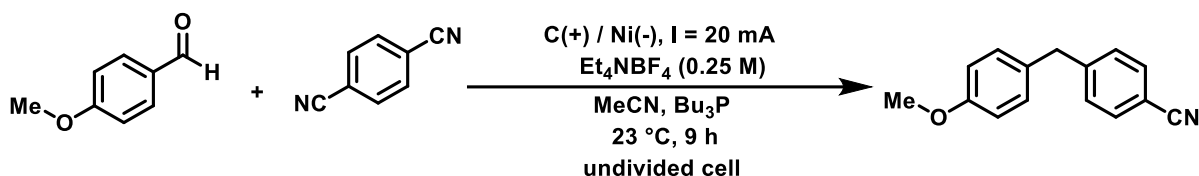
⁴ Q.-L. Wang, Z. Sun, H. Huang, G. Mao and G. Deng, *Green Chem.*, 2022, **24**, 3293-3299.

electrolyzed at a constant current of 20 mA at room temperature for 17 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 41% yield (111 mg).

¹H NMR (400 MHz, Chloroform-d) δ 7.63 – 7.52 (m, 6H), 7.44 (m, 2H), 7.39 – 7.30 (m, 3H), 7.24 (d, J = 8.2 Hz, 2H), 4.08 (s, 2H). **¹³C NMR** (101 MHz, Chloroform-d) δ 146.95, 140.98, 139.98, 138.71, 132.68, 130.00, 129.69, 129.12, 127.80, 127.63, 127.33, 119.32, 110.43, 41.93.

NMR data matched those reported in the literature²

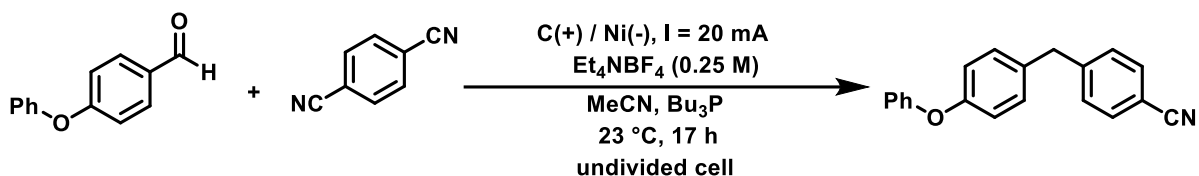
4-(4-methoxybenzyl)benzonitrile (3f)



The starting 4-methoxybenzaldehyde (136 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 9 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 58% yield (128 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.58 (d, J = 8.2 Hz, 2H), 7.30 (d, J = 8.3 Hz, 2H), 7.11 (d, J = 8.6 Hz, 2H), 6.96 – 6.84 (m, 2H), 4.00 (s, 2H), 3.82 (s, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 158.59, 147.51, 132.51, 131.65, 130.20, 129.77, 119.31, 114.39, 110.12, 55.51, 41.33.

NMR data matched those reported in the literature³

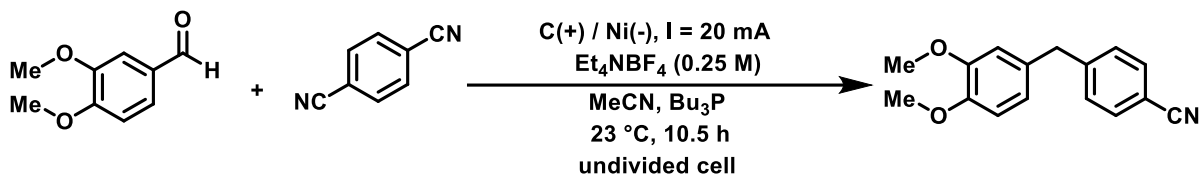
4-(4-phenoxybenzyl)benzonitrile (3g)



The starting 4-phenoxybenzaldehyde (198 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 17 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 49% yield (140 mg). **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 8.3 Hz, 2H), 7.40 – 7.27 (m, 4H), 7.16 – 7.08 (m, 3H), 7.04 – 6.98 (m, 2H), 6.99 – 6.93 (m, 2H), 4.02 (s, 2H). **¹³C NMR** (101 MHz, Chloroform-*d*) δ 157.41, 156.27, 147.06, 134.42, 132.62, 130.49, 130.04, 129.87, 123.59, 119.38, 119.27, 119.11, 110.34, 41.50.

NMR data matched those reported in the literature⁵

4-(3,4-dimethoxybenzyl)benzonitrile (3h)



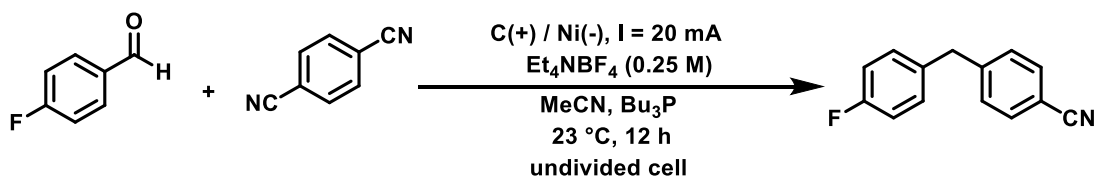
The starting 3,4-dimethoxybenzaldehyde (166 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0

⁵ M. Miao, L.-L. Liao, G.-M. Cao, W.-J. Zhou and D.-G. Yu, *Sci China Chem.*, 2019, **62**, 1519–1524.

mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10.5 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 44% yield (111 mg). **¹H NMR** (401 MHz, Chloroform-d) δ 7.51 (d, J = 8.3 Hz, 2H), 7.27 (d, J = 8.5 Hz, 2H), 6.98 (d, J = 8.1 Hz, 1H), 6.52 – 6.38 (m, 2H), 3.93 (s, 2H), 3.77 (d, J = 13.8 Hz, 6H). **¹³C NMR** (101 MHz, Chloroform-d) δ 160.16, 158.41, 147.66, 132.24, 130.94, 129.62, 120.49, 119.50, 109.65, 104.37, 98.92, 55.56, 35.96.

NMR data matched those reported in the literature⁶

4-(4-fluorobenzyl)benzonitrile (3i)



The starting 4-fluorobenzaldehyde (124 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 12 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 66% yield (139 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.57 (d, J = 8.1 Hz, 2H), 7.27 (d, J = 8.0 Hz, 2H), 7.13 (dd, J = 8.3, 5.5 Hz, 2H), 6.99 (t, J = 8.6 Hz, 2H), 4.01 (s, 2H). **¹³C NMR** (101 MHz, Chloroform-d) δ 161.88 (d, J = 245.0 Hz), 146.77, 135.30 (d, J = 3.3

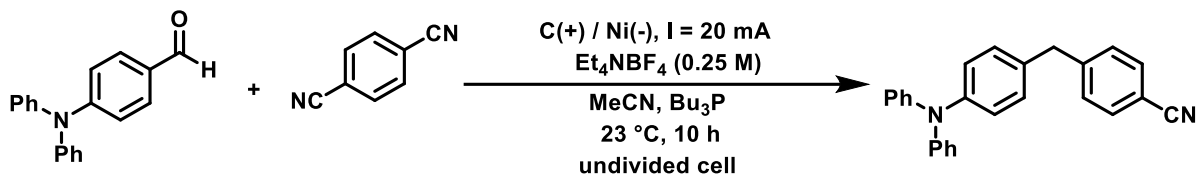
⁶ Y. Mo, Z. Lu, G. Rughoobur, P. Patil, N. Gershenfeld, A. I. Akinwande, S. L. Buchwald and K. F. Jensen, *Science*, 2020, **368**, 1352-1357.

Hz), 132.58, 130.69, 129.79, 119.17, 115.80 (d, $J = 21.3$ Hz), 110.38, 41.31. ^{19}F

NMR (376 MHz, Chloroform- d) δ -116.08.

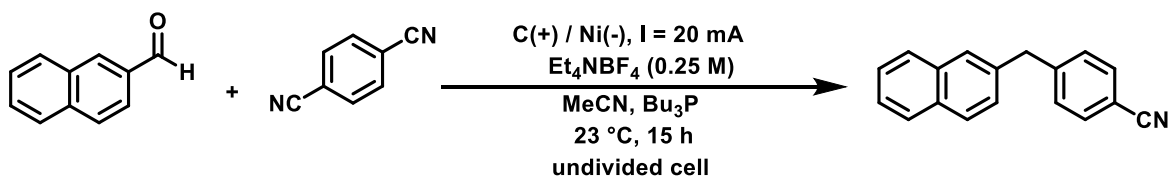
NMR data matched those reported in the literature¹

4-(4-(diphenylamino)benzyl)benzonitrile (3j)



The starting 4-(diphenylamino)benzaldehyde (273 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a pale yellow liquid in 38% yield (135 mg). ^1H **NMR** (400 MHz, Chloroform- d) δ 7.61 (d, $J = 8.1$ Hz, 2H), 7.34 (d, $J = 8.1$ Hz, 3H), 7.26 (t, $J = 7.8$ Hz, 4H), 7.10 (d, $J = 7.8$ Hz, 3H), 7.07 - 6.96(m, 6H), 4.00 (s, 2H). ^{13}C **NMR** (101 MHz, Chloroform- d) δ 147.97, 147.16, 146.69, 133.74, 132.57, 129.96, 129.89, 129.50, 124.48, 124.39, 123.03, 119.29, 110.24, 41.62. **HRMS** m/z (ESI) calcd for $\text{C}_{26}\text{H}_{21}\text{N}_2$ ($[\text{M}+\text{H}]^+$) 361.1700, found 361.1700.

4-(naphthalen-2-ylmethyl)benzonitrile (3k)

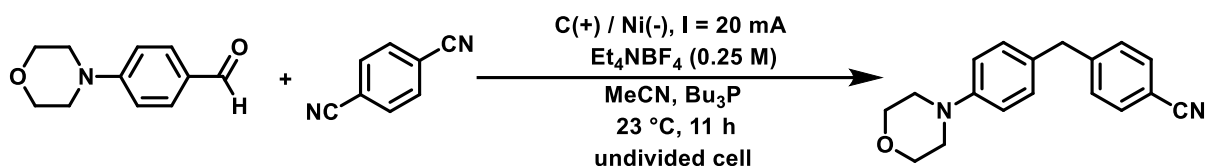


The starting 2-naphthaldehyde (156 mg, 1.0 mmol) was reacted with

1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 15 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 37% yield (90 mg). **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.89 – 7.79 (m, 3H), 7.66 (s, 1H), 7.61 (d, *J* = 8.1 Hz, 2H), 7.56 – 7.46 (m, 2H), 7.38 – 7.28 (m, 3H), 4.22 (s, 2H). **¹³C NMR** (101 MHz, Chloroform-*d*) δ 146.86, 137.06, 133.82, 132.59, 132.50, 130.00, 128.78, 127.97, 127.82, 127.68, 127.53, 126.59, 126.05, 119.28, 110.36, 42.38.

NMR data matched those reported in the literature⁷

4-(4-morpholinobenzyl)benzonitrile (3l)

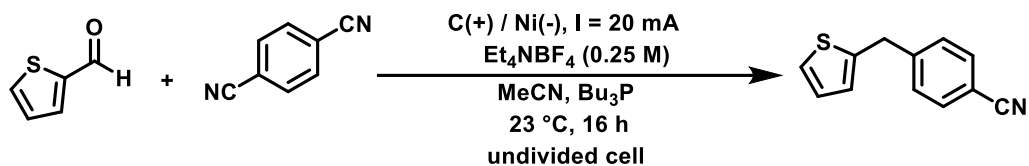


The starting 4-morpholinobenzaldehyde (191 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 11 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 5:1) to afford the title compound as a pale yellow solid in 35% yield (88 mg). **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 8.1 Hz, 2H), 7.29 (d, *J* = 7.9 Hz, 2H), 7.09 (d, *J* = 8.5 Hz, 2H), 6.89 (d, *J* = 8.5 Hz, 2H), 3.98 (s, 2H), 3.91 – 3.85 (m, 4H), 3.19 – 3.11 (m, 4H). **¹³C NMR** (101 MHz, Chloroform-*d*) δ 150.28, 147.61, 132.54, 131.00, 130.01, 129.82, 119.36, 116.23, 110.13, 67.19, 49.66, 41.36.

⁷ C. Han, Z. Zhang, S. Xu, K. Wang, K. Chen and J. Zhao, *J. Org. Chem.*, 2019, **84**, 16308–16313.

HRMS m/z (ESI) calcd for $C_{18}H_{19}N_2O$ ($[M+H]^+$) 279.1492, found 279.1490.

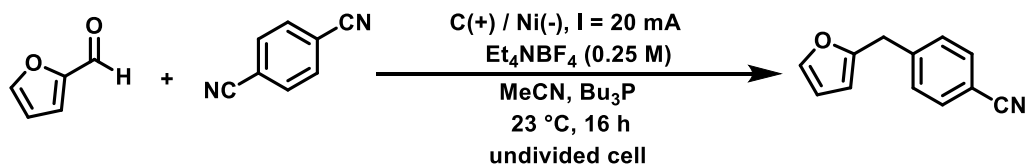
4-(thiophen-2-ylmethyl)benzonitrile (3m)



The starting 2-thienaldehyde (112 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 16 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a pale yellow liquid in 30% yield (60 mg). **^1H NMR** (400 MHz, Chloroform- d) δ 7.61 (d, J = 8.2 Hz, 2H), 7.37 (d, J = 8.1 Hz, 2H), 7.21 (d, J = 5.2 Hz, 1H), 7.01 – 6.95 (m, 1H), 6.88 – 6.80 (m, 1H), 4.24 (s, 2H). **^{13}C NMR** (101 MHz, Chloroform- d) δ 146.11, 141.95, 132.65, 129.58, 127.34, 126.15, 124.92, 119.17, 110.69, 36.24.

NMR data matched those reported in the literature⁶

4-(furan-2-ylmethyl)benzonitrile (3n)

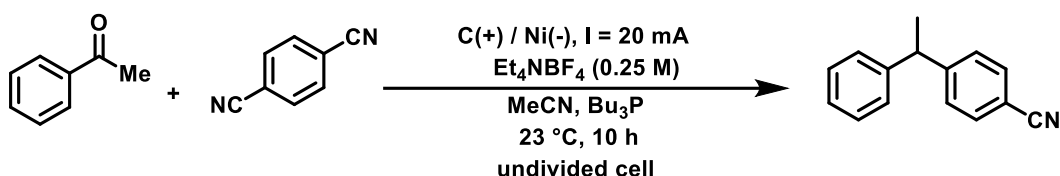


The starting furfural (96 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a

constant current of 20 mA at room temperature for 16 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 34% yield (63 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.61 (d, J = 8.2 Hz, 2H), 7.35 (d, J = 8.4 Hz, 3H), 6.37 – 6.32 (m, 1H), 6.10 (d, J = 3.1 Hz, 1H), 4.05 (s, 2H). **¹³C NMR** (101 MHz, Chloroform-d) δ 152.81, 144.01, 142.30, 132.60, 129.70, 119.18, 110.70, 107.31, 34.75.

NMR data matched those reported in the literature⁶

4-(1-phenylethyl)benzonitrile (3o)

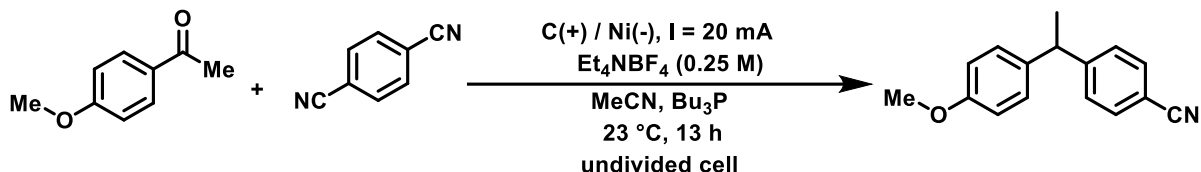


The starting acetophenone (120 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 71% yield (147 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.58 (d, J = 8.3 Hz, 2H), 7.34 – 7.27 (m, 4H), 7.25 (d, J = 8.8 Hz, 1H), 7.22 – 7.18 (m, 2H), 4.22 (q, J = 7.2 Hz, 1H), 1.66 (d, J = 7.2 Hz, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 152.17, 144.92, 132.51, 128.92, 128.69, 127.82, 126.88, 119.31, 110.12, 45.13, 21.70.

NMR data matched those reported in the literature⁸

⁸ K.-J. Jiao, D. Liu, H.-X. Ma, H. Qiu, P. Fang and T.-S. Mei, *Angew. Chem. Int. Ed.*, 2020, **59**, 6520–6524.

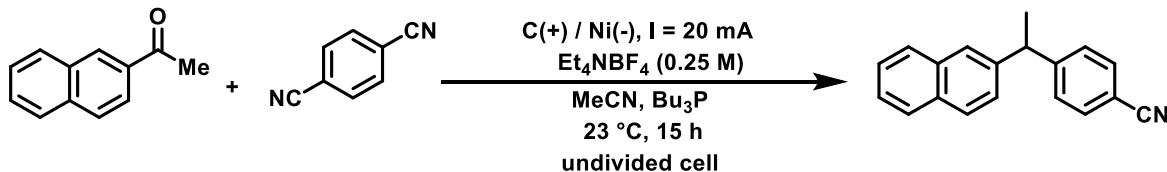
4-(1-(4-methoxyphenyl)ethyl)benzonitrile (3p)



The starting 4'-methoxyacetophenone (150 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 13 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 50% yield (118 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.56 (d, J = 8.2 Hz, 2H), 7.31 (d, J = 8.1 Hz, 2H), 7.11 (d, J = 8.6 Hz, 2H), 6.86 (d, J = 8.6 Hz, 2H), 4.20 – 4.12 (m, 1H), 3.79 (s, 3H), 1.63 (d, J = 7.2 Hz, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 158.49, 152.65, 137.05, 132.51, 128.78, 128.61, 119.35, 114.27, 110.05, 55.54, 44.36, 21.91.

NMR data matched those reported in the literature⁹

4-(1-(naphthalen-2-yl)ethyl)benzonitrile (3q)

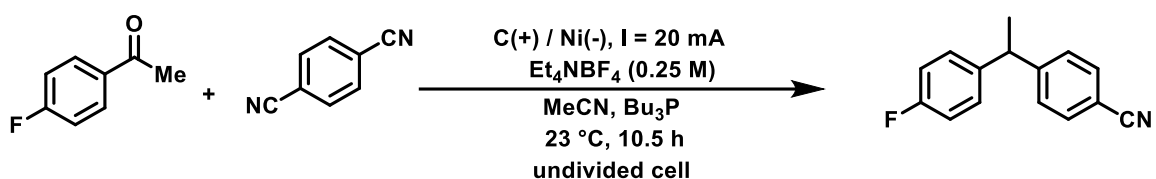


The starting 2-acetonaphthone (170 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and

⁹ H. Tanaka, K. Sakai, A. Kawamura, K. Oisaki and M. Kanai, *Chem. Commun.*, 2018, **54**, 3215-3218.

electrolyzed at a constant current of 20 mA at room temperature for 15 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 67% yield (172 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.88 – 7.77 (m, 3H), 7.72 (s, 1H), 7.58 (d, J = 8.2 Hz, 2H), 7.54 – 7.46 (m, 2H), 7.36 (d, J = 8.2 Hz, 2H), 7.27 (d, J = 7.4 Hz, 1H), 4.37 (q, J = 7.1 Hz, 1H), 1.76 (d, J = 7.2 Hz, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 151.96, 142.27, 133.69, 132.49, 128.76, 128.59, 127.96, 127.86, 126.60, 126.50, 126.02, 125.81, 119.26, 110.18, 45.17, 21.58. **HRMS** m/z (ESI) calcd for C₁₉H₁₆N ([M+H]⁺) 258.1278, found 258.1275.

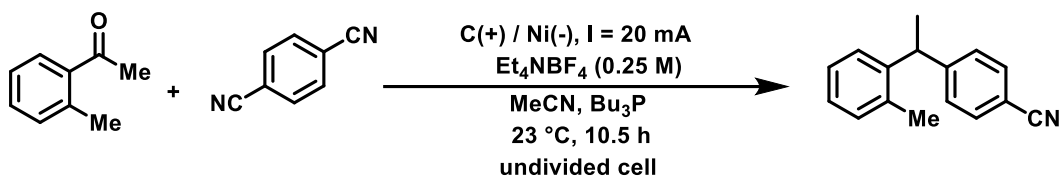
4-(1-(4-fluorophenyl)ethyl)benzonitrile (3r)



The starting 1-(4-fluorophenyl)ethan-1-one (138 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10.5 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 5:1) to afford the title compound as a pale yellow liquid in 72% yield (161 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.59 (d, J = 8.3 Hz, 2H), 7.33 (d, J = 8.1 Hz, 2H), 7.18 (dd, J = 8.6, 5.5 Hz, 2H), 7.01 (d, J = 17.3 Hz, 2H), 4.22 (q, J = 7.2 Hz, 1H), 1.66 (d, J = 7.3 Hz, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 161.54 (d, J = 245.0 Hz), 151.72, 140.44 (d, J = 3.2 Hz), 132.35, 129.05 (d, J = 7.9 Hz), 128.38, 118.99, 115.46 (d, J = 21.2 Hz), 110.08, 44.17, 21.61. **¹⁹F NMR** (376 MHz,

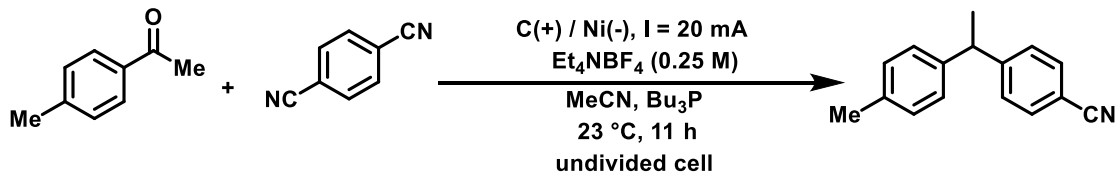
Chloroform-d) δ -116.14. **HRMS** m/z (ESI) calcd for $C_{15}H_{13}FN$ ($[M+H]^+$) 226.1027, found 226.1027.

4-(1-(*o*-tolyl)ethyl)benzonitrile (3s)



The starting 2'-methylacetophenone (134 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10.5 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 57% yield (125 mg). **1H NMR** (400 MHz, Chloroform-d) δ 7.58 (d, J = 8.2 Hz, 2H), 7.32 – 7.25 (m, 4H), 7.22 – 7.15 (m, 2H), 4.40 (q, J = 7.1 Hz, 1H), 2.23 (s, 3H), 1.66 (d, J = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-d) δ 152.23, 142.57, 136.31, 132.53, 130.97, 128.74, 126.99, 126.88, 126.60, 119.34, 110.02, 41.51, 22.05, 20.03. **HRMS** m/z (ESI) calcd for $C_{16}H_{16}N$ ($[M+H]^+$) 222.1278, found 222.1278.

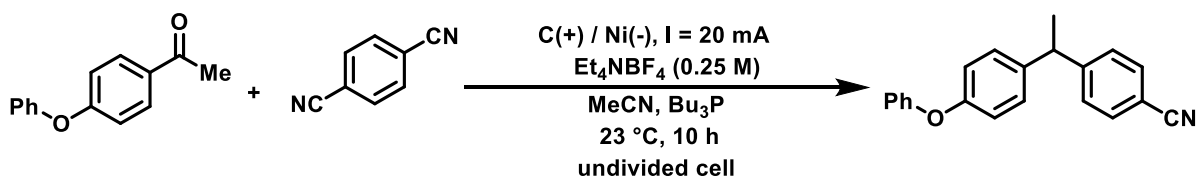
4-(1-(*p*-tolyl)ethyl)benzonitrile (3t)



The starting 4'-methylacetophenone (134 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0

mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 11 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 83% yield (183 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.57 (d, J = 8.2 Hz, 2H), 7.32 (d, J = 8.1 Hz, 2H), 7.15 – 7.07 (m, 4H), 4.16 (q, J = 7.1 Hz, 1H), 2.33 (s, 3H), 1.64 (d, J = 7.2 Hz, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 152.49, 142.00, 136.50, 132.53, 129.63, 128.67, 127.71, 119.37, 110.10, 44.80, 21.79, 21.29. **HRMS** m/z (ESI) calcd for C₁₆H₁₆N ([M+H]⁺) 222.1278, found 222.1277.

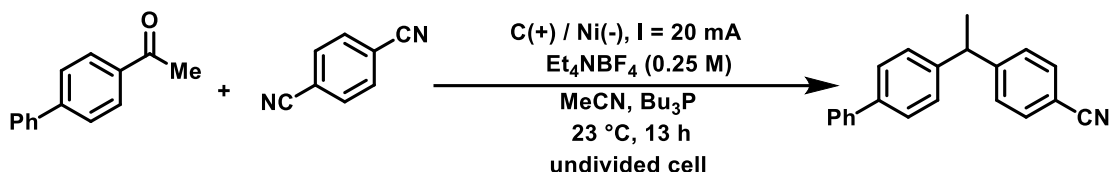
4-(1-(4-phenoxyphenyl)ethyl)benzonitrile (3u)



The starting 4'-phenoxyacetophenone (212 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 5:1) to afford the title compound as a pale yellow liquid in 64% yield (190 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.58 (d, J = 8.2 Hz, 2H), 7.34 (d, J = 13.8 Hz, 4H), 7.18 – 7.09 (m, 3H), 7.00 (d, J = 8.1 Hz, 2H), 6.95 (d, J = 8.5 Hz, 2H), 4.19 (q, J = 7.1 Hz, 1H), 1.65 (d, J = 7.2 Hz, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 157.35, 156.10, 152.20, 139.71, 132.53, 130.00, 129.03, 128.62, 123.55, 119.25, 119.14, 119.11, 110.18, 44.48, 21.85. **HRMS** m/z (ESI) calcd for

C₂₁H₁₈NO ([M+H]⁺) 300.1383, found 300.1382.

4-(1-([1,1'-biphenyl]-4-yl)ethyl)benzonitrile (3v)

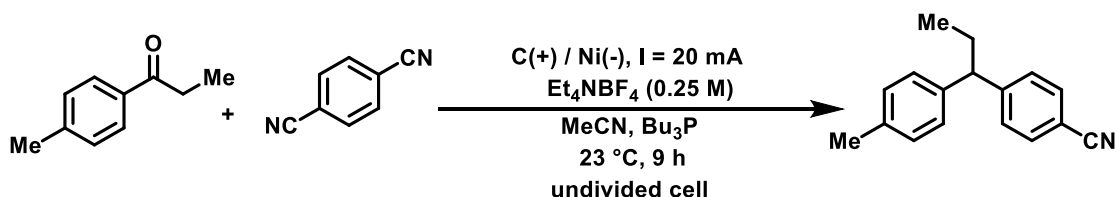


The starting 4-acetylbiphenyl (196 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 13 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 54% yield (154 mg).

¹H NMR (400 MHz, Chloroform-d) δ 7.66 – 7.55 (m, 6H), 7.47 (t, J = 7.6 Hz, 2H), 7.39 (d, J = 8.2 Hz, 3H), 7.29 (d, J = 8.0 Hz, 2H), 4.27 (q, J = 7.1 Hz, 1H), 1.72 (d, J = 7.2 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 152.11, 144.04, 140.96, 139.87, 132.62, 129.08, 128.75, 128.27, 127.67, 127.58, 127.31, 119.33, 110.29, 44.89, 21.77.

NMR data matched those reported in the literature¹⁰

4-(1-(p-tolyl)propyl)benzonitrile (3w)

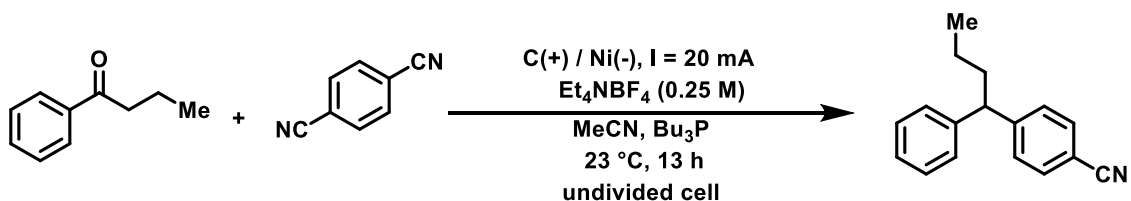


The starting 4'-methylpropiophenone (148 mg, 1.0 mmol) was reacted with

¹⁰ M. Miao, L.-L. Liao, G.-M. Cao, W.-J. Zhou and D.-G. Yu, *Sci. China Chem.*, **62**, 1519–1524 (2019).

1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 9 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 58% yield (136 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.56 (d, J = 8.1 Hz, 2H), 7.33 (d, J = 8.2 Hz, 2H), 7.15 – 7.06 (m, 4H), 3.82 (t, J = 7.7 Hz, 1H), 2.32 (s, 3H), 2.12 – 2.01 (m, 2H), 0.90 (t, J = 7.3 Hz, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 151.38, 140.75, 136.49, 132.53, 129.64, 128.93, 127.98, 119.37, 110.08, 53.17, 28.53, 21.29, 12.92. **HRMS** m/z (ESI) calcd for C₁₆H₁₆N ([M+H]⁺) 236.1434, found 236.1435.

4-(1-phenylbutyl)benzonitrile (3x)

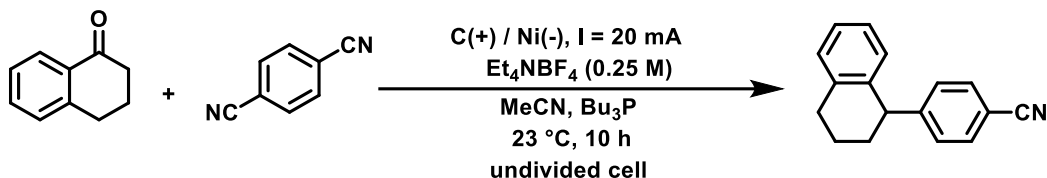


The starting butyrophenone (148 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 13 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a pale yellow liquid in 66% yield (154 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.56 (d, J = 8.2 Hz, 2H), 7.40 – 7.24 (m, 4H), 7.20 (t, J = 6.7 Hz, 3H), 3.96 (t, J = 7.8 Hz, 1H), 2.06 – 1.98 (m, 2H), 1.25 (m, 2H), 0.93 (t, J = 7.3 Hz, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 151.26,

143.92, 132.57, 128.97, 128.12, 126.92, 119.34, 110.18, 51.45, 37.74, 21.32, 14.30.

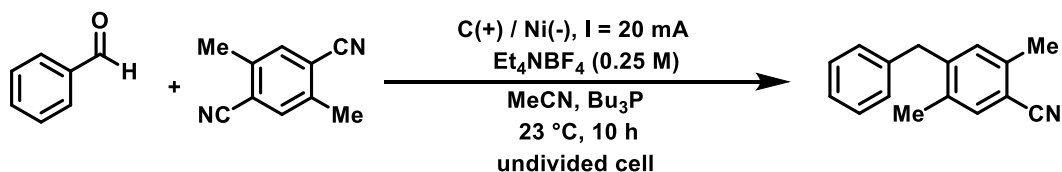
NMR data matched those reported in the literature⁴

4-(1,2,3,4-tetrahydronaphthalen-1-yl)benzonitrile (3y)



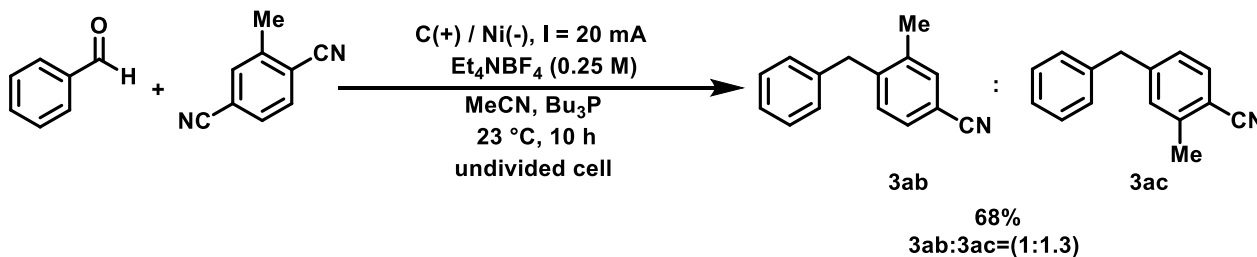
The starting 1-tetralone (146 mg, 1.0 mmol) was reacted with 1,4-dicyanobenzene (128 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 48% yield (112 mg). **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.56 (d, J = 7.6 Hz, 2H), 7.20 (d, J = 7.8 Hz, 2H), 7.16 (d, J = 4.0 Hz, 2H), 7.09 – 7.02 (m, 1H), 6.76 (d, J = 7.7 Hz, 1H), 4.20 (t, J = 6.4 Hz, 1H), 2.98 - 2.81 (m, 2H), 2.25 – 2.14 (m, 1H), 1.91 – 1.72 (m, 3H). **¹³C NMR** (101 MHz, Chloroform-*d*) δ 153.47, 137.93, 132.39, 130.26, 129.87, 129.56, 126.75, 126.20, 119.37, 110.07, 45.94, 33.28, 29.82, 20.88. **HRMS** m/z (ESI) calcd for $\text{C}_{17}\text{H}_{16}\text{N}$ ($[\text{M}+\text{H}]^+$) 234.1278, found 234.1277.

4-benzyl-2,5-dimethylbenzonitrile (3aa)



The starting benzaldehyde (106 mg, 1.0 mmol) was reacted with 2,5-dimethylterephthalonitrile (156 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 57% yield (127 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.41 (s, 1H), 7.33 (t, J = 7.3 Hz, 2H), 7.26 (q, J = 7.3, 5.9 Hz, 1H), 7.12 (d, J = 7.3 Hz, 2H), 7.07 (s, 1H), 4.01 (s, 2H), 2.50 (s, 3H), 2.26 (s, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 144.63, 139.62, 139.04, 135.16, 134.01, 131.87, 128.87, 128.82, 126.61, 118.64, 110.70, 39.66, 20.16, 19.19. **HRMS** m/z (ESI) calcd for C₁₆H₁₆N ([M+H]⁺) 222.1278, found 222.1277.

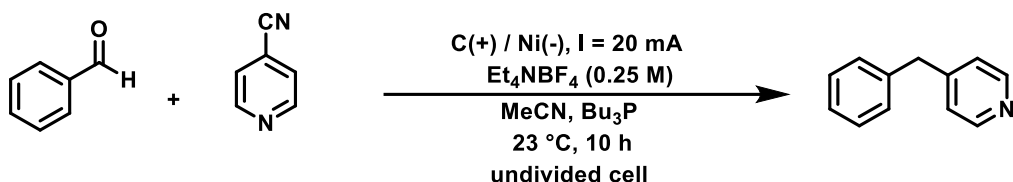
4-benzyl-3-methylbenzonitrile : 4-benzyl-2-methylbenzonitrile (3ab:3ac)



The starting benzaldehyde (106 mg, 1.0 mmol) was reacted with methyl 2-methylterephthalonitrile (142 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a pale yellow liquid in 68% yield

(140 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.51 (d, J = 7.9 Hz, 1H), 7.44 (d, J = 7.9 Hz, 2H), 7.34 – 7.29 (m, 3H), 7.28 – 7.22 (m, 2H), 7.22 – 7.14 (m, 4H), 7.11 (d, J = 7.0 Hz, 2H), 4.03 (s, 1H), 4.00 (s, 2H), 2.51 (s, 3H), 2.30 (s, 2H). **¹³C NMR** (101 MHz, Chloroform-d) δ 146.76, 144.98, 142.32, 139.79, 138.87, 138.29, 133.79, 132.87, 130.99, 130.72, 130.05, 129.17, 128.98, 128.96, 128.92, 127.11, 126.83, 126.74, 119.39, 118.56, 110.65, 110.40, 42.16, 39.74, 20.69, 19.76. **HRMS** m/z (ESI) calcd for C₁₅H₁₄N ([M+H]⁺) 208.1121, found 208.1119.

4-benzylpyridine (3ad)

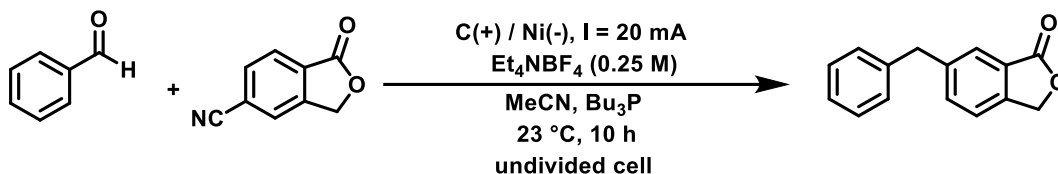


The starting benzaldehyde (106 mg, 1.0 mmol) was reacted with 4-cyanopyridine (104 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 4:1) to afford the title compound as a dark brown liquid in 74% yield (125 mg). **¹H NMR** (400 MHz, Chloroform-d) δ 8.49 (d, J = 6.0 Hz, 2H), 7.32 (t, J = 7.3 Hz, 2H), 7.25 (d, J = 7.2 Hz, 1H), 7.17 (d, J = 7.1 Hz, 2H), 7.10 (d, J = 5.8 Hz, 2H), 3.96 (s, 2H). **¹³C NMR** (101 MHz, Chloroform-d) δ 150.32, 150.12, 139.15, 129.34, 129.03, 126.98, 124.49, 41.54.

NMR data matched those reported in the literature¹¹

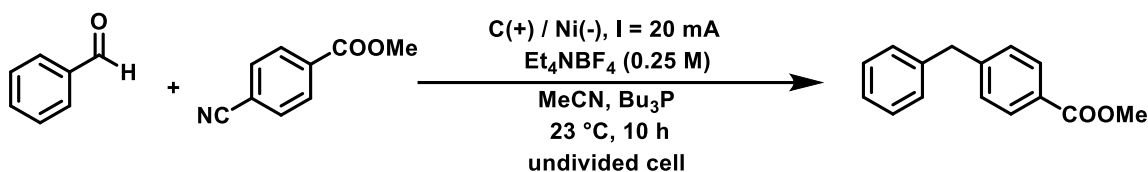
6-benzylisobenzofuran-1(3H)-one (3ae)

¹¹ T. W. Reidl and J. S. Bandar, *J. Am. Chem. Soc.*, 2021, **143**, 11939–11945.



The starting benzaldehyde (106 mg, 1.0 mmol) was reacted with 5-cyanophthalide (159 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a white solid in 24% yield (53 mg). **^1H NMR** (400 MHz, Chloroform- d) δ 7.86 (d, J = 7.9 Hz, 1H), 7.41 (d, J = 7.9 Hz, 1H), 7.35 (t, J = 7.4 Hz, 2H), 7.28 (d, J = 8.2 Hz, 2H), 7.21 (d, J = 7.1 Hz, 2H), 5.28 (s, 2H), 4.13 (s, 2H). **^{13}C NMR** (101 MHz, Chloroform- d) δ 148.69, 147.54, 139.84, 130.40, 129.30, 129.10, 127.00, 126.07, 124.10, 122.56, 69.81, 42.48. **HRMS** m/z (ESI) calcd for $\text{C}_{15}\text{H}_{13}\text{O}_2$ ($[\text{M}+\text{H}]^+$) 225.0911, found 225.0910.

methyl 4-benzylbenzoate (3af)



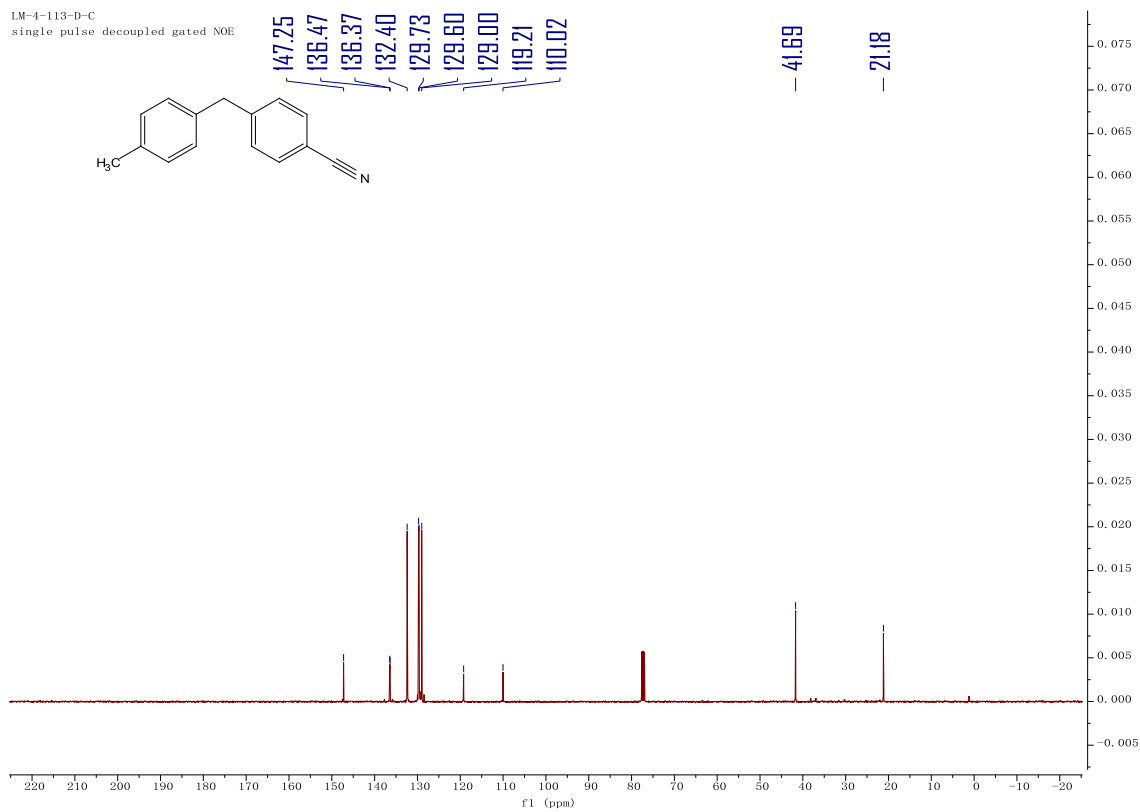
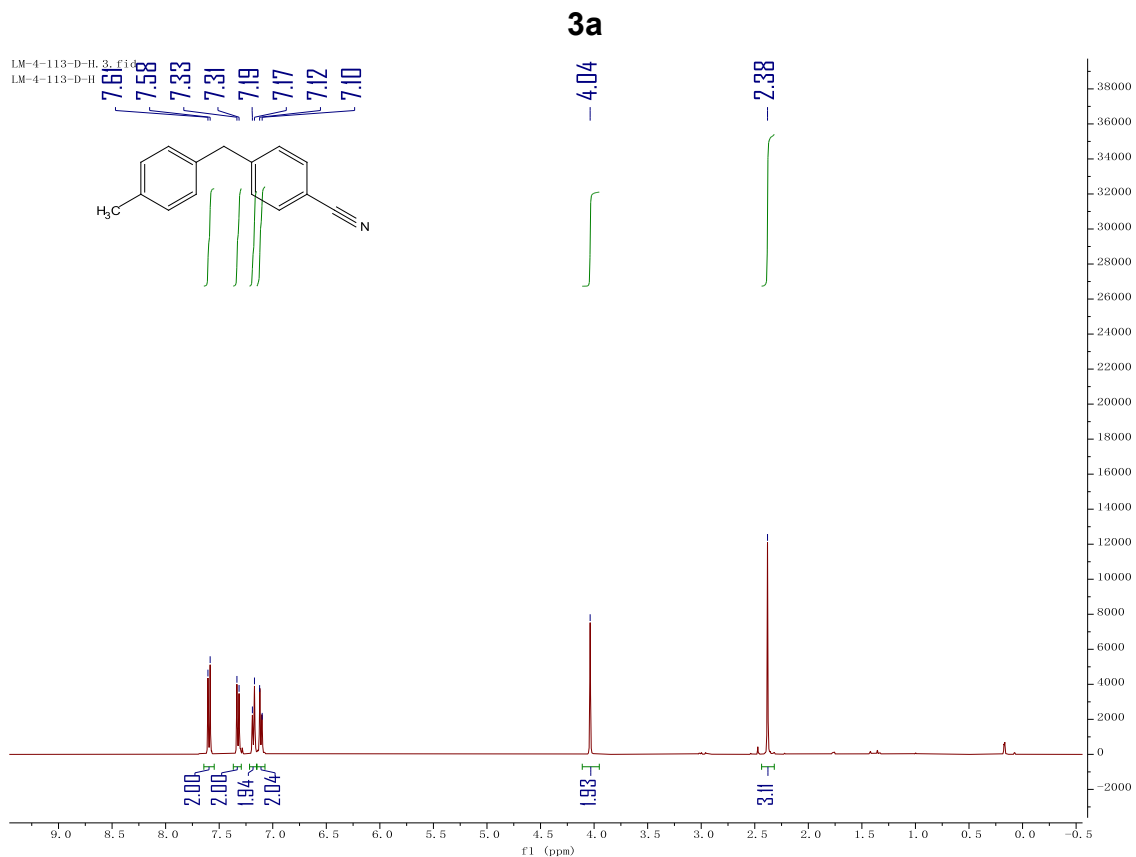
The starting benzaldehyde (106 mg, 1.0 mmol) was reacted with methyl 4-cyanobenzoate (161 mg, 1.0 mmol) and tributylphosphine (1011 mg, 5.0 mmol) according to General Procedure. The reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 10 h. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a colorless liquid in 58% yield (131

mg). **¹H NMR** (400 MHz, Chloroform-d) δ 7.98 (d, J = 8.1 Hz, 2H), 7.34 – 7.23 (m, 5H), 7.19 (d, J = 7.3 Hz, 2H), 4.04 (s, 2H), 3.91 (s, 3H). **¹³C NMR** (101 MHz, Chloroform-d) δ 167.36, 146.83, 140.42, 130.12, 129.25, 128.91, 128.38, 126.68, 52.32, 42.22.

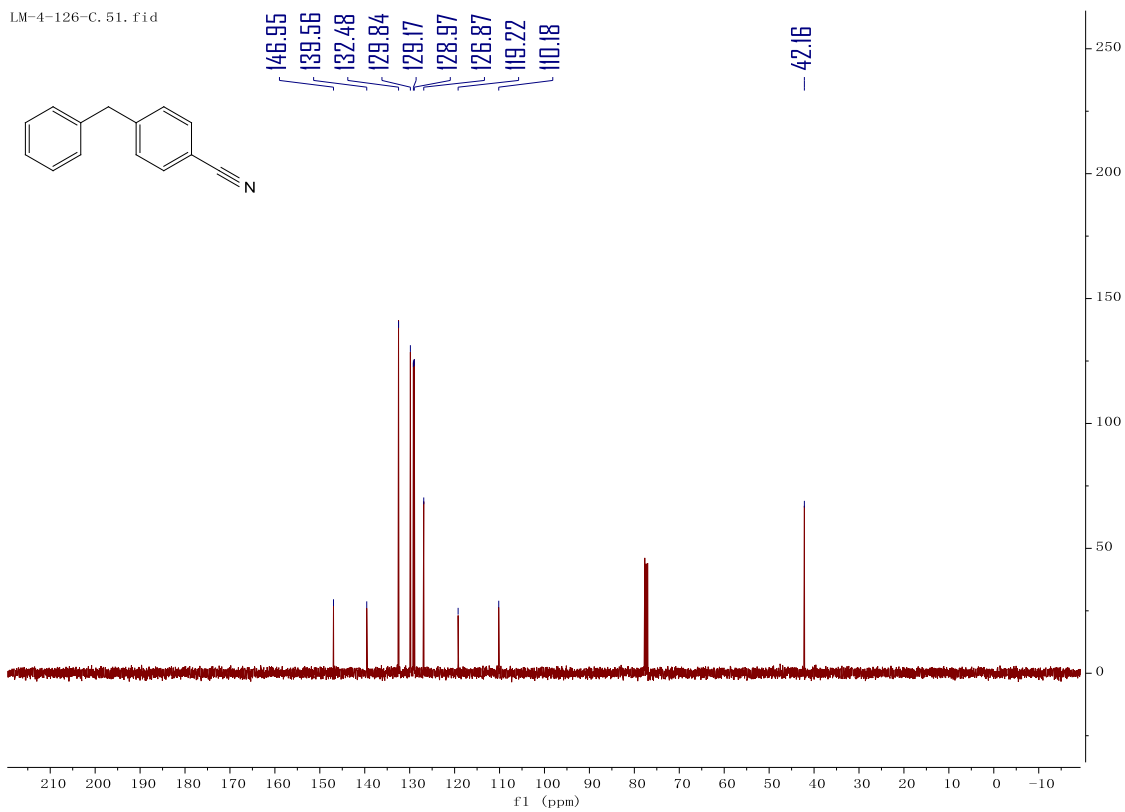
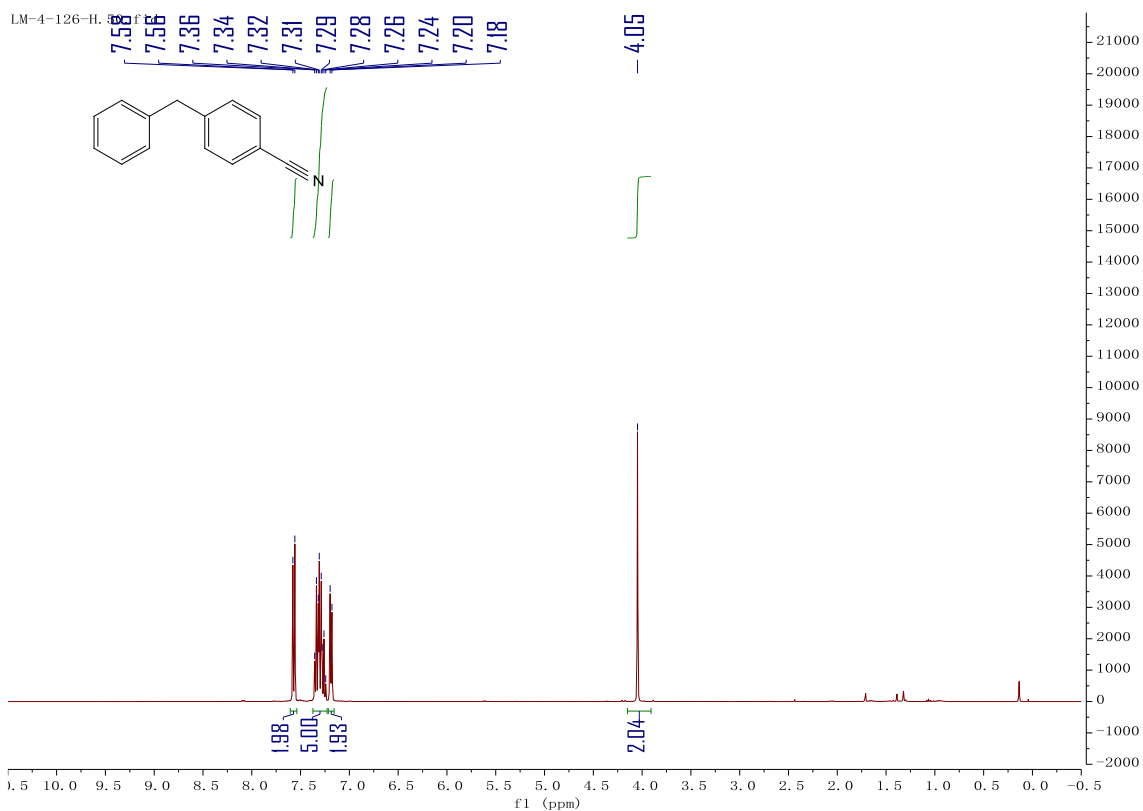
NMR data matched those reported in the literature¹²

6. ¹H NMR, ¹³C NMR and ¹⁹F NMR spectra

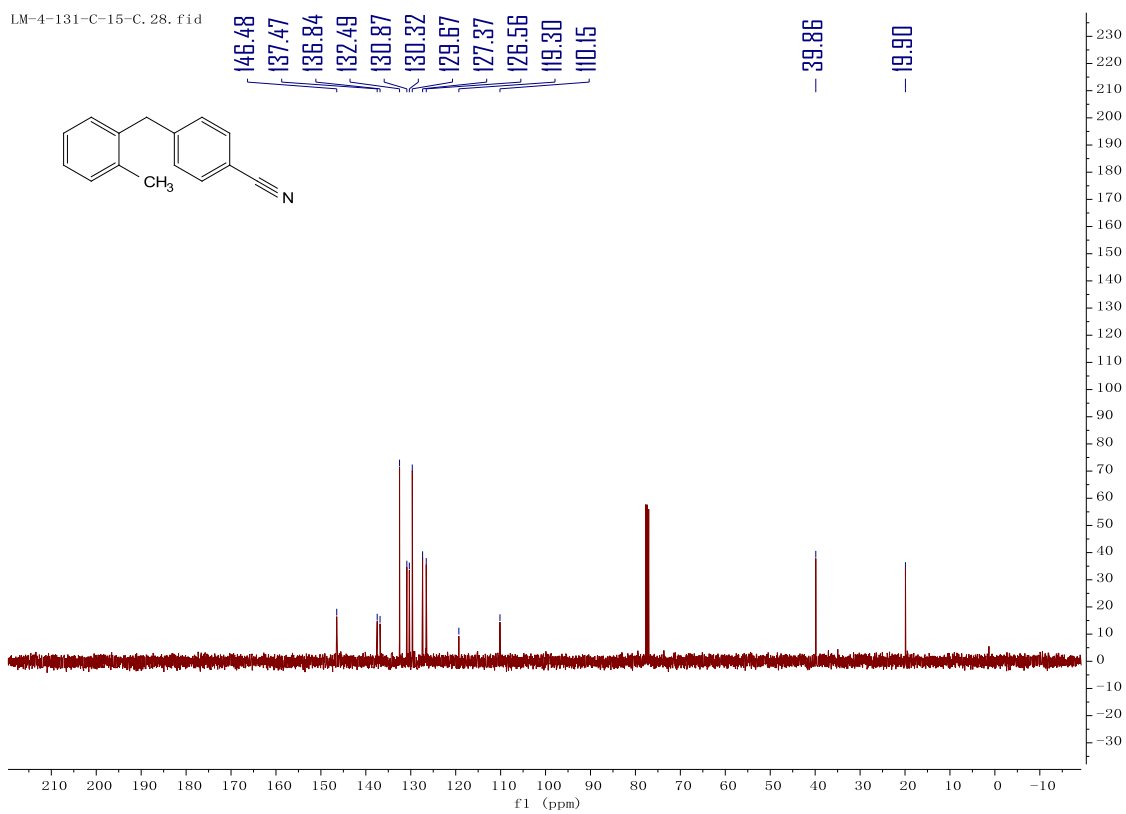
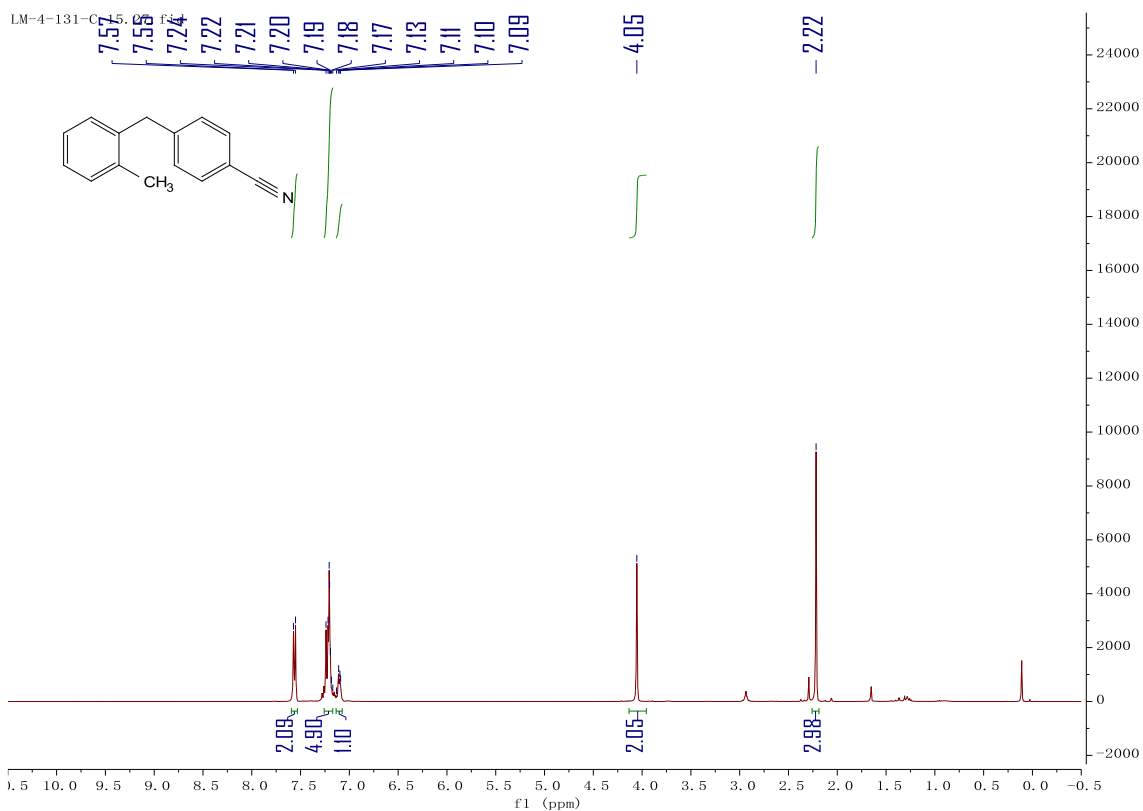
¹² T. Suga, and Y. Ukaji, *Org. Lett.*, 2018, **20**, 7846–7850.



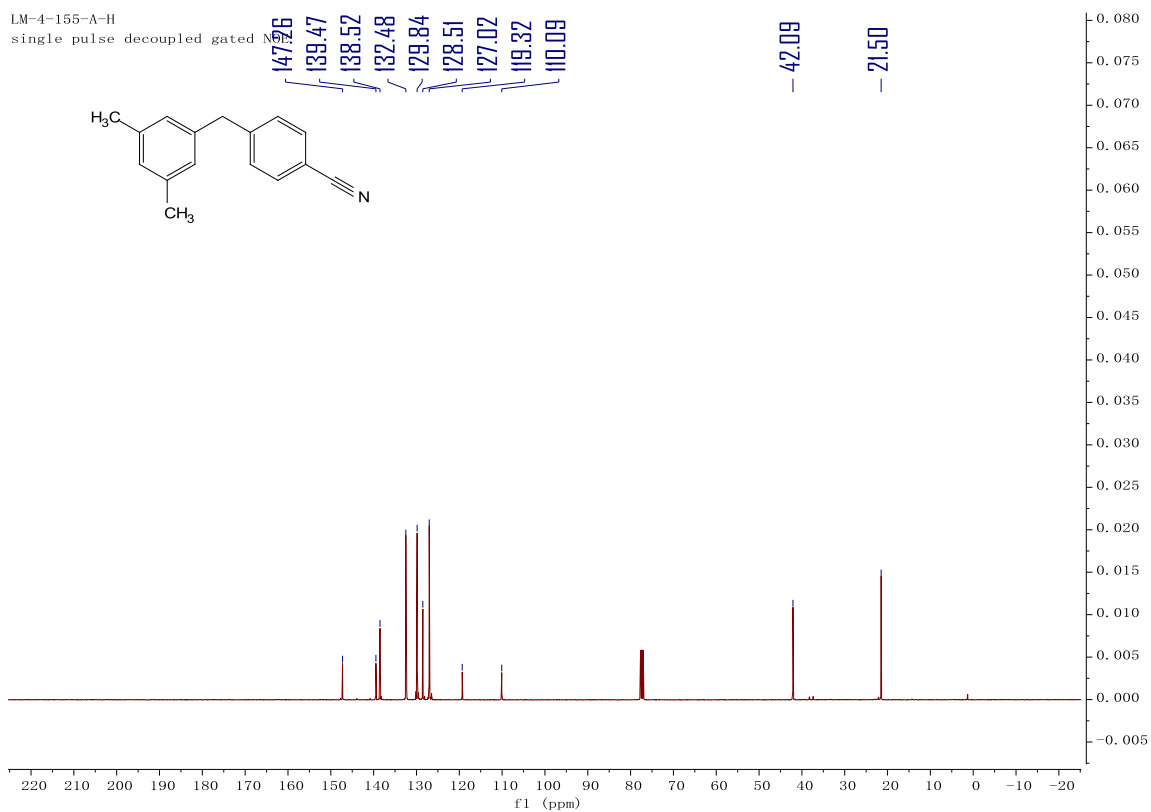
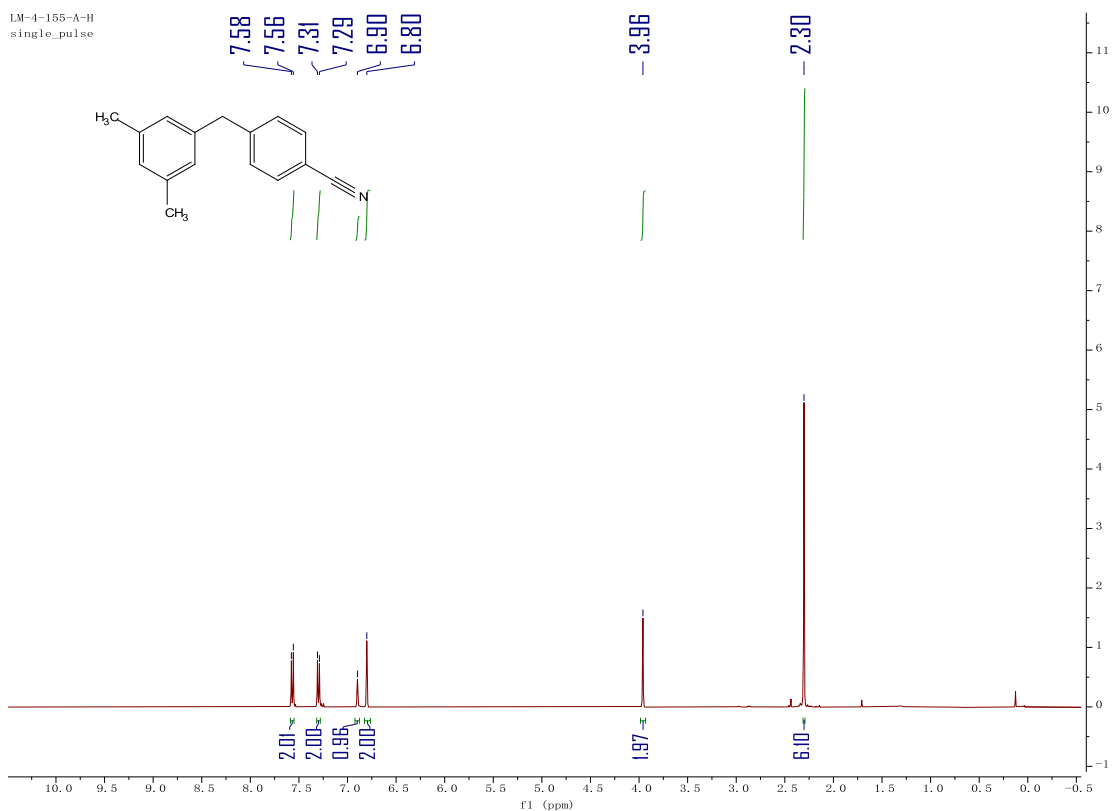
3b

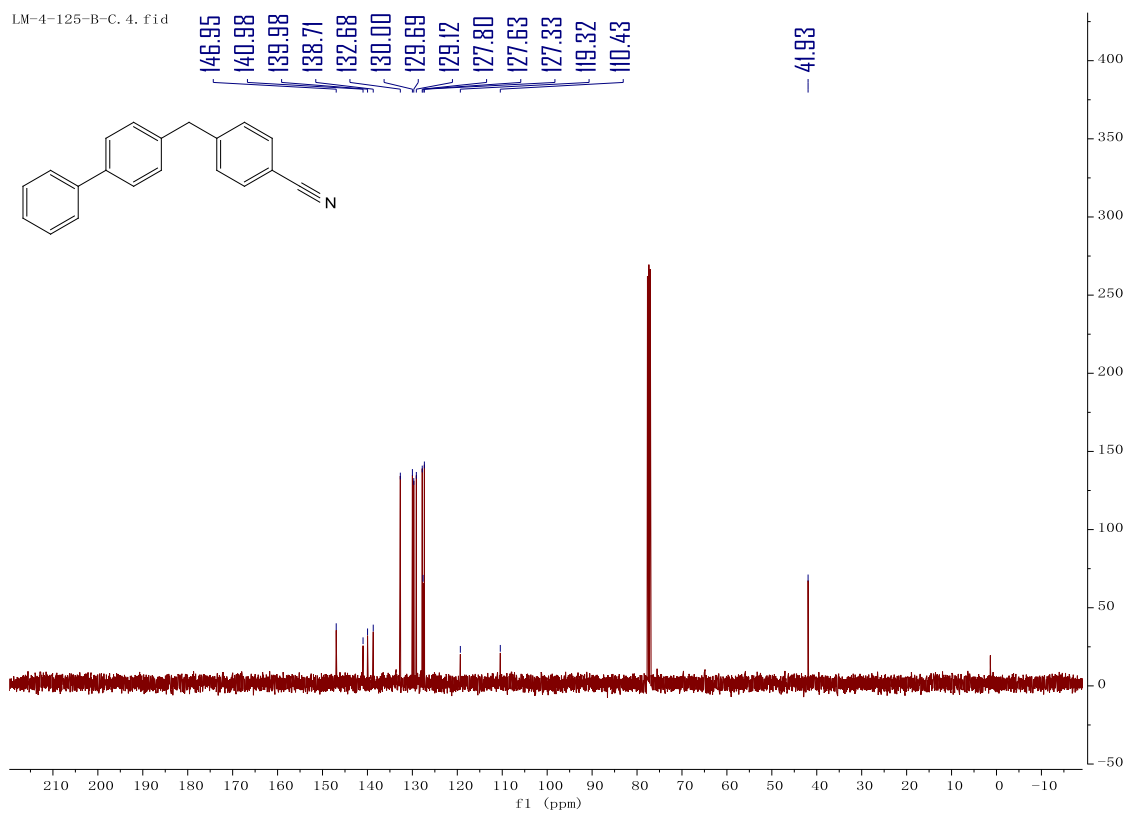
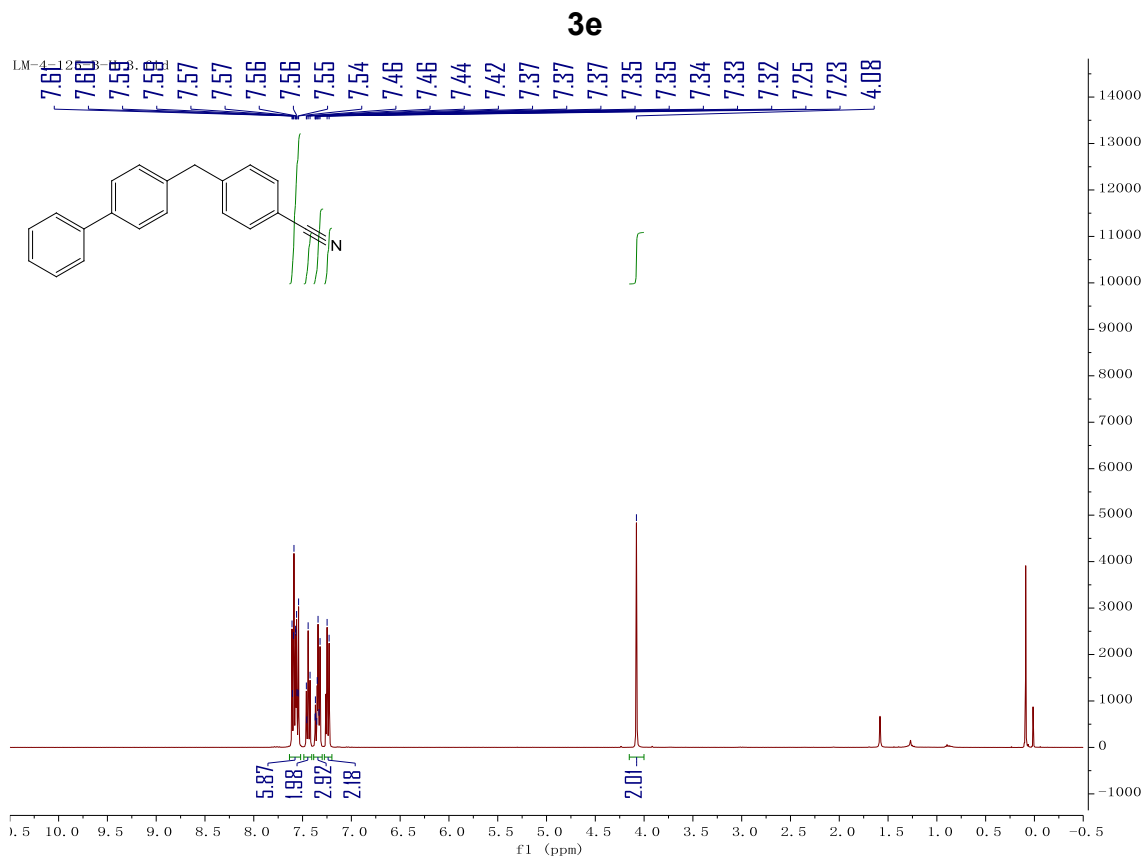


3c

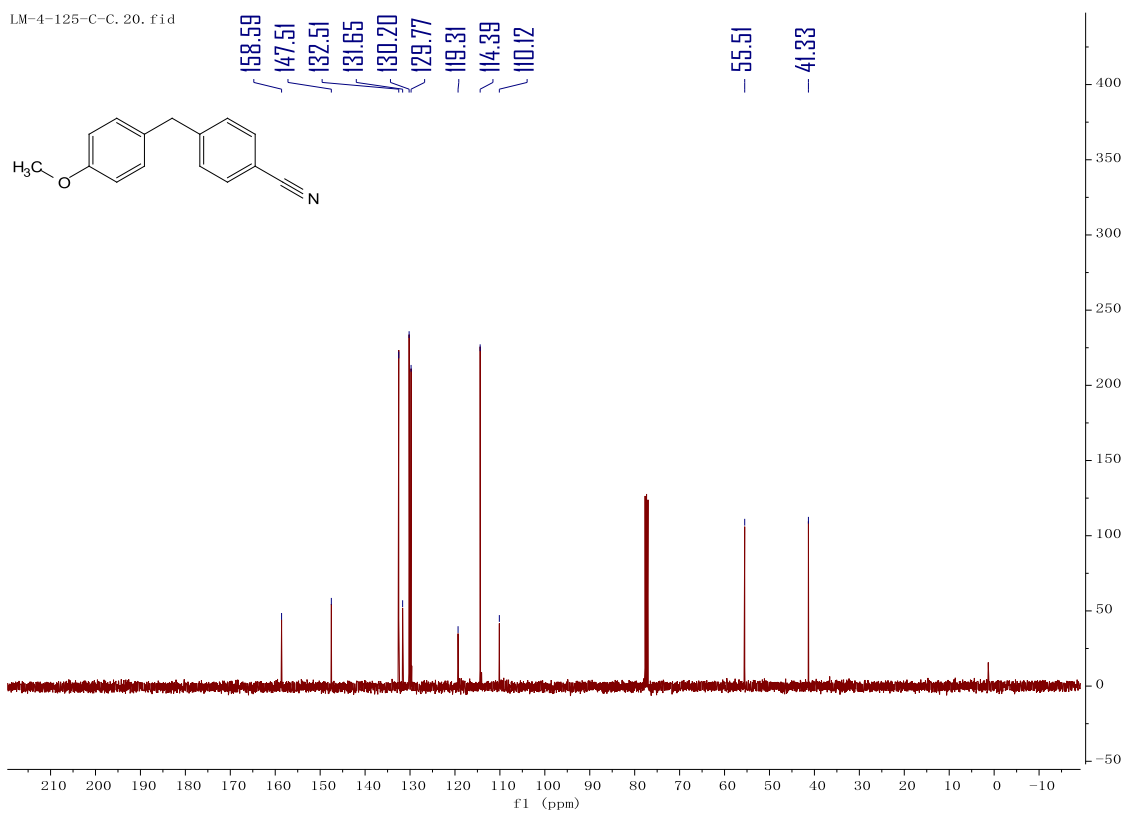
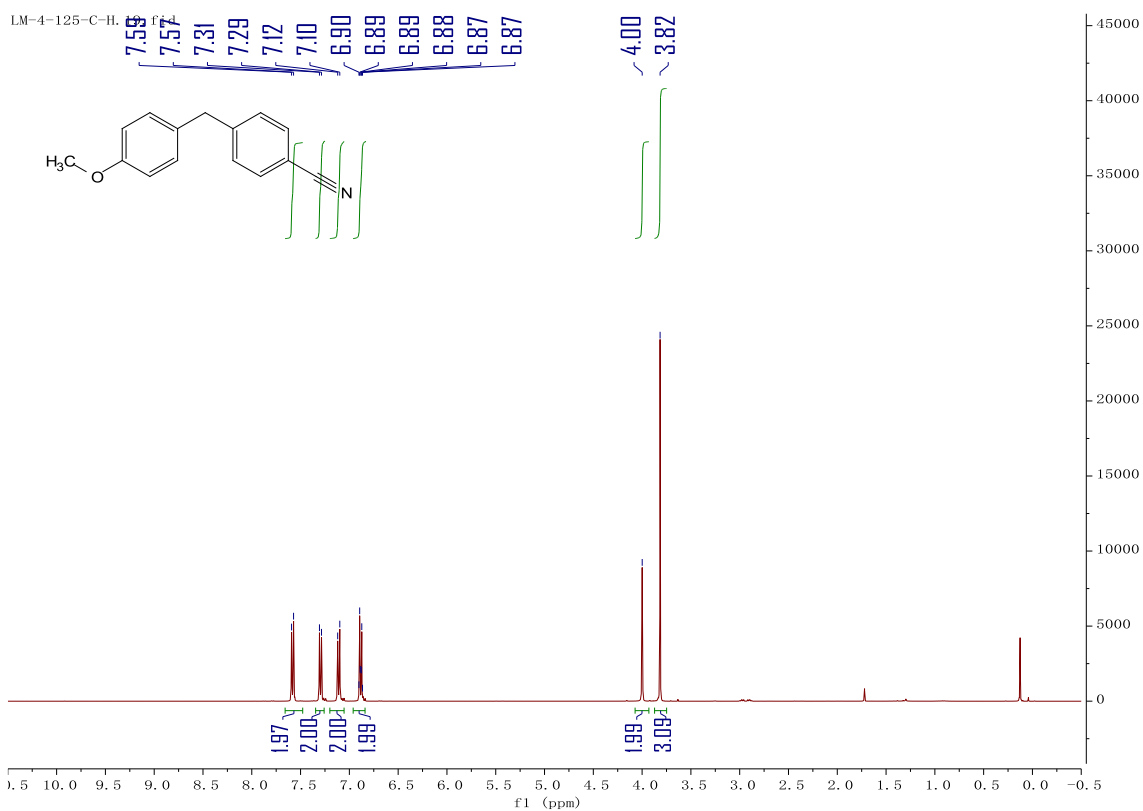


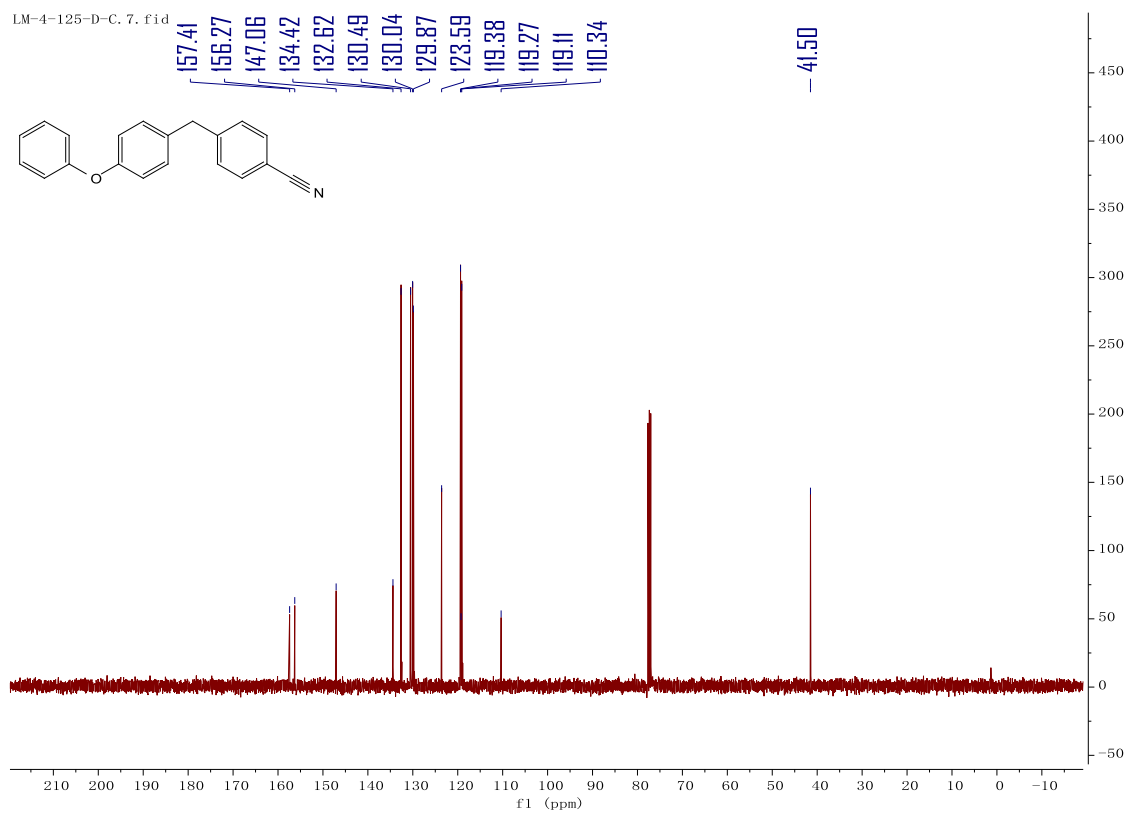
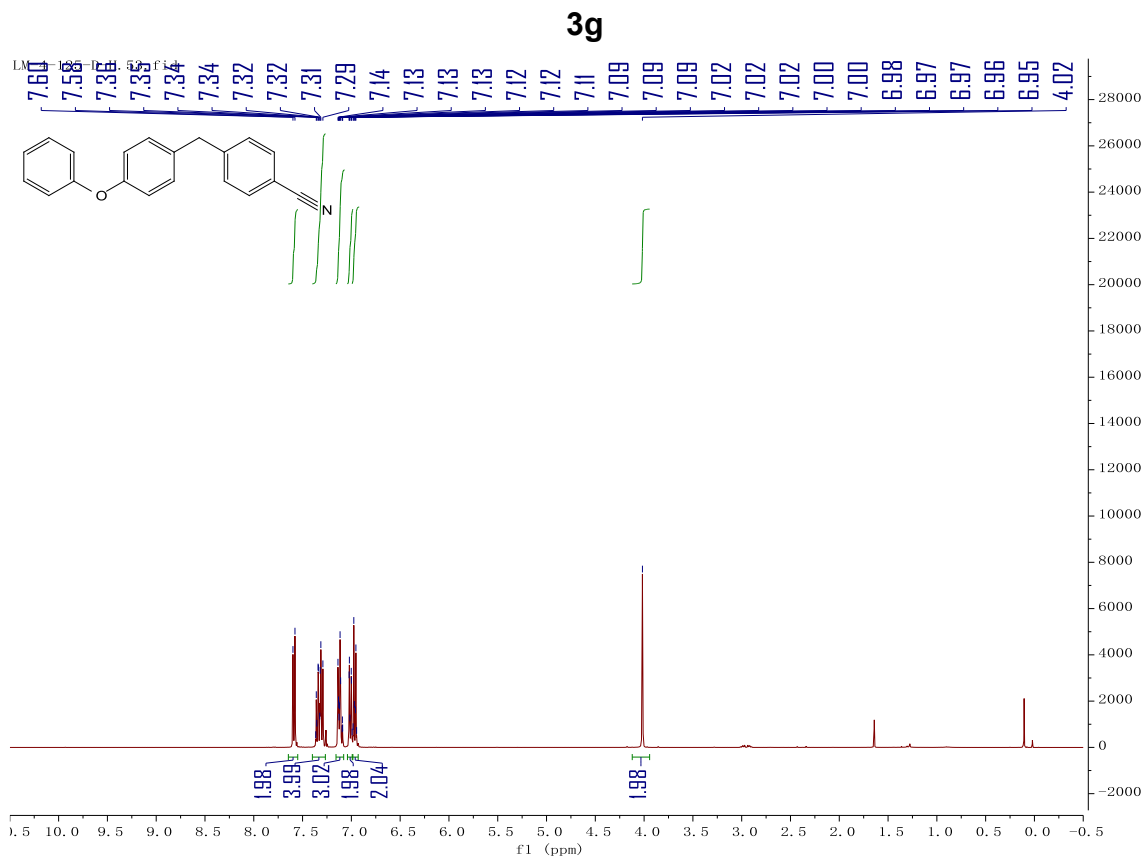
3d



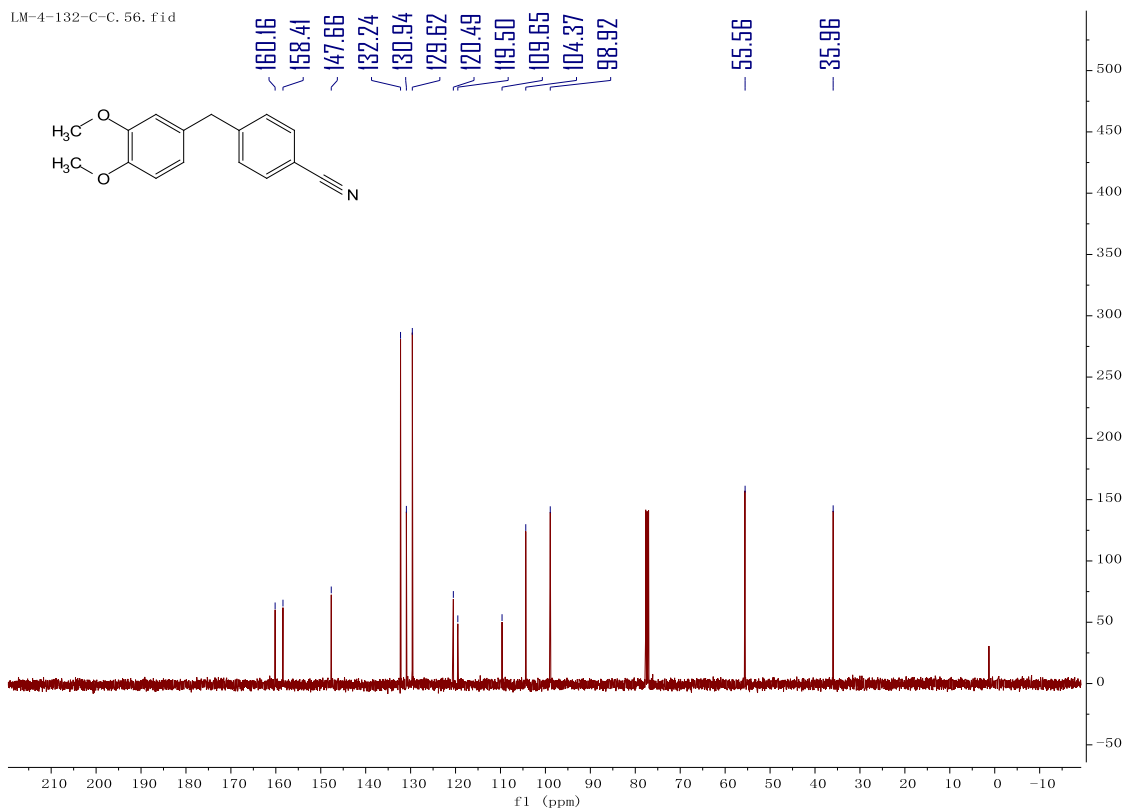
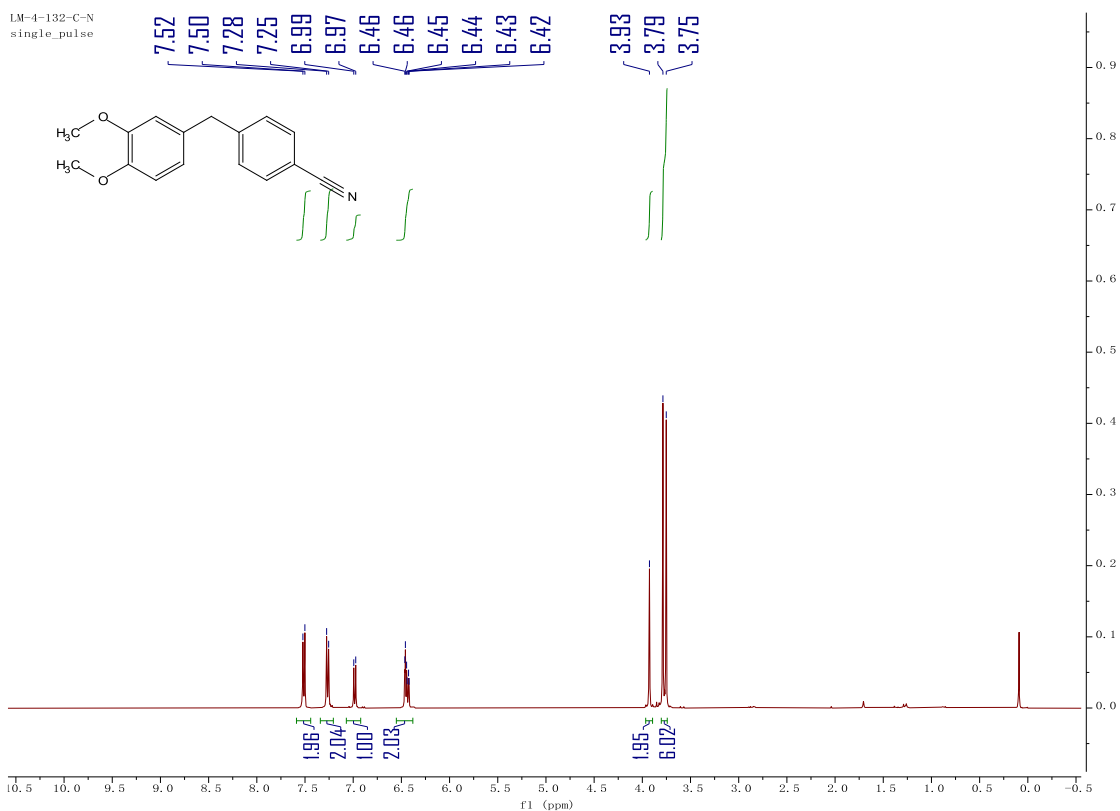


3f

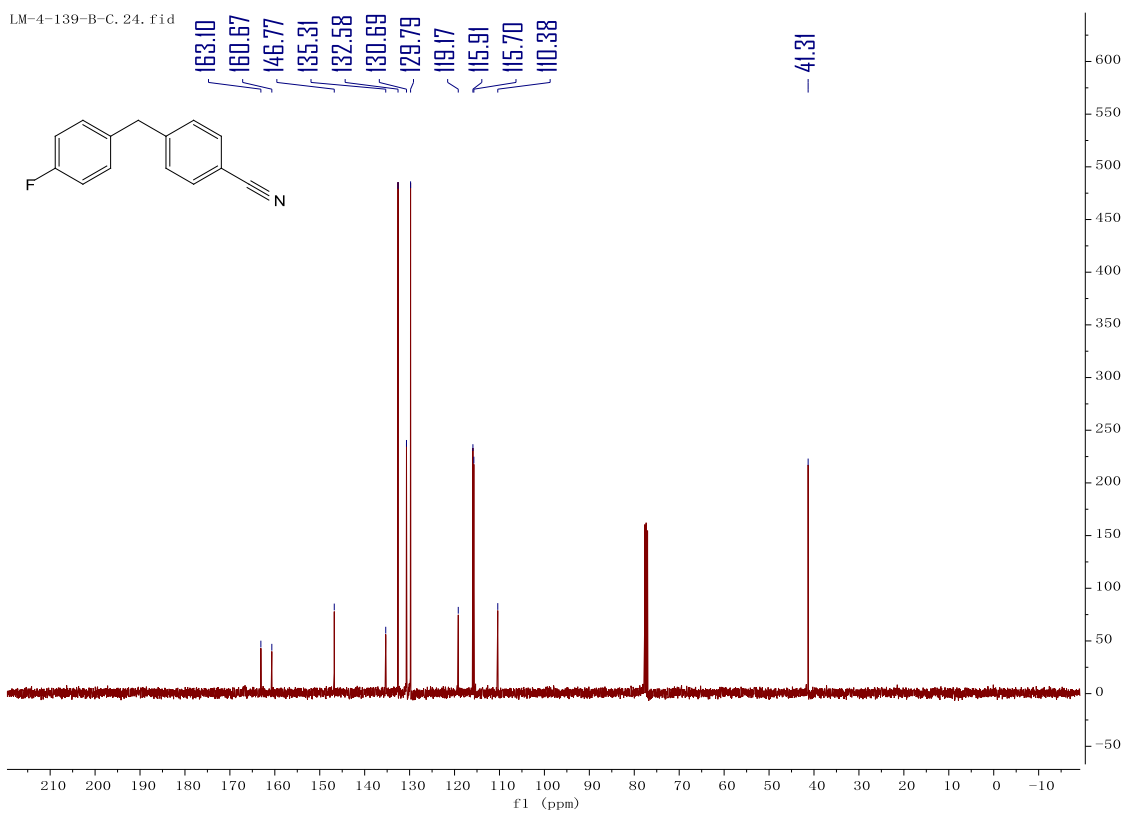
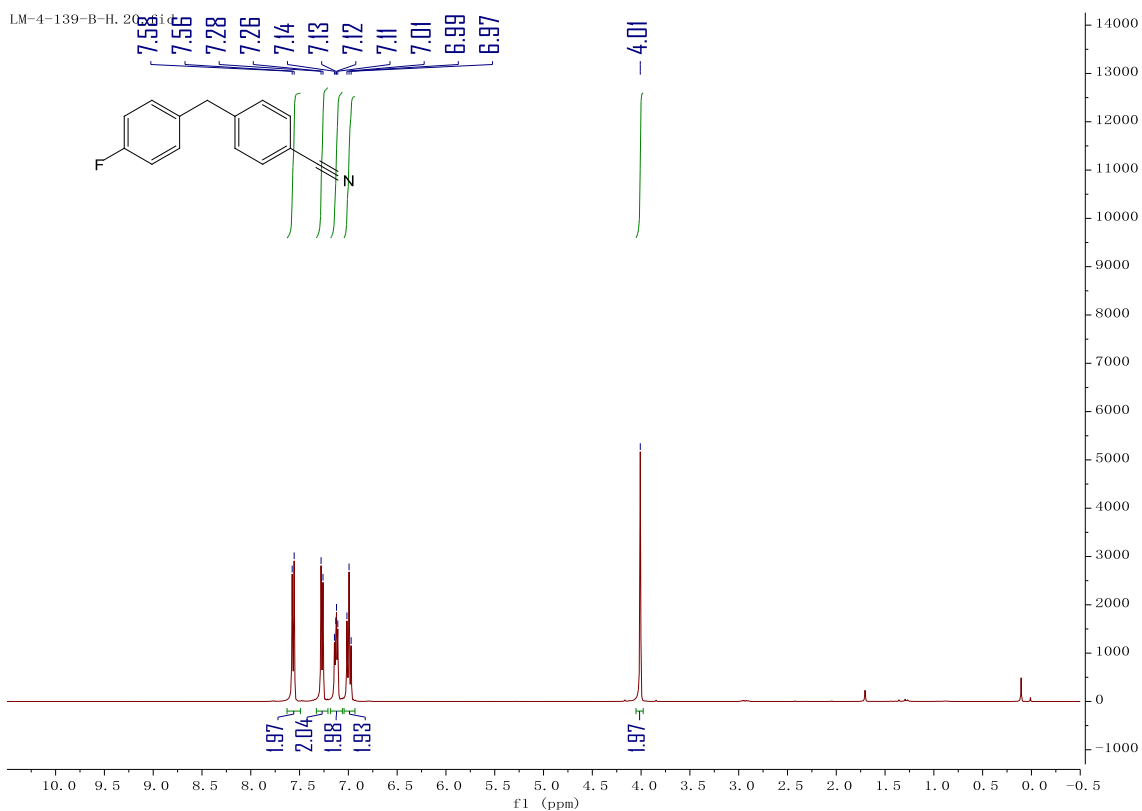




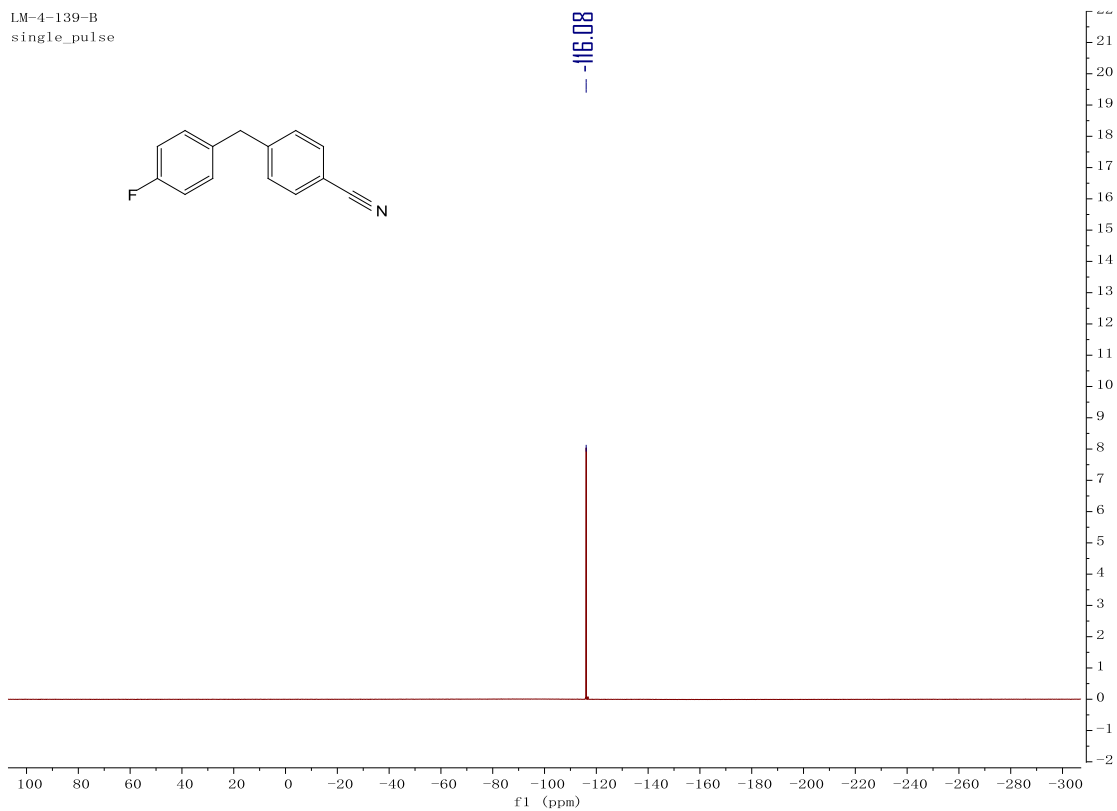
3h



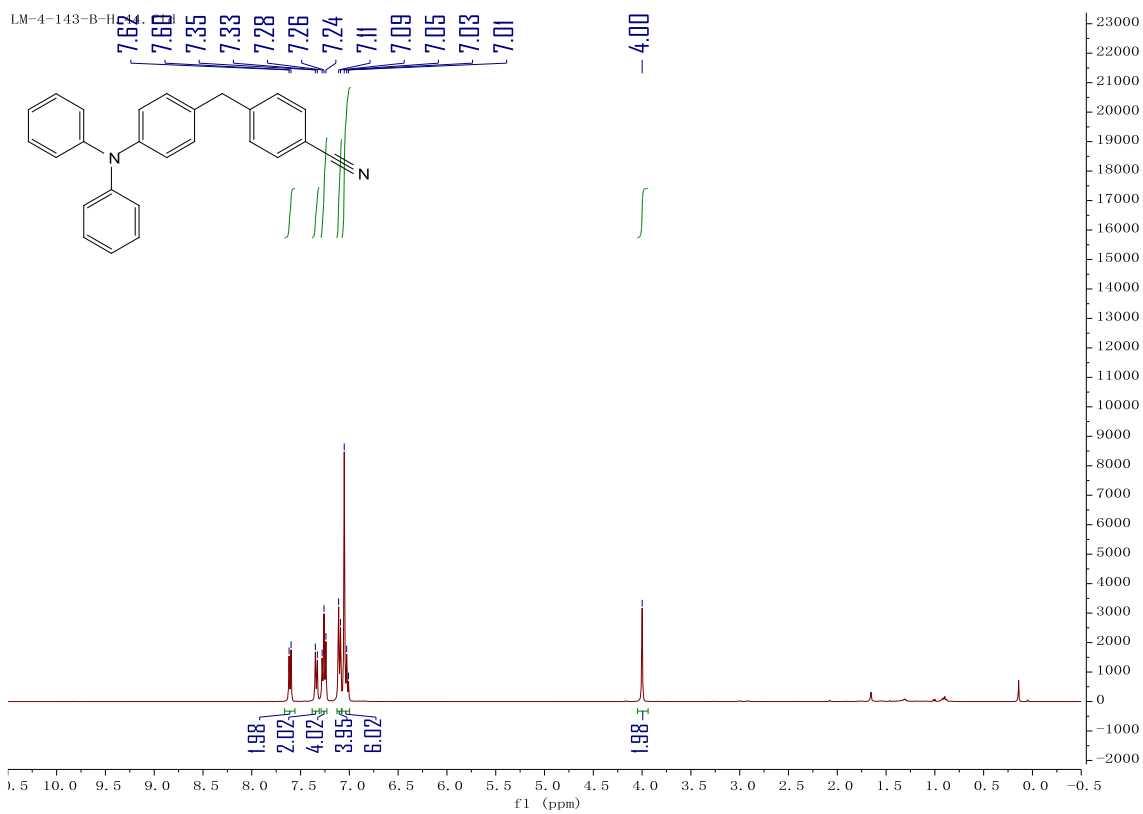
3i

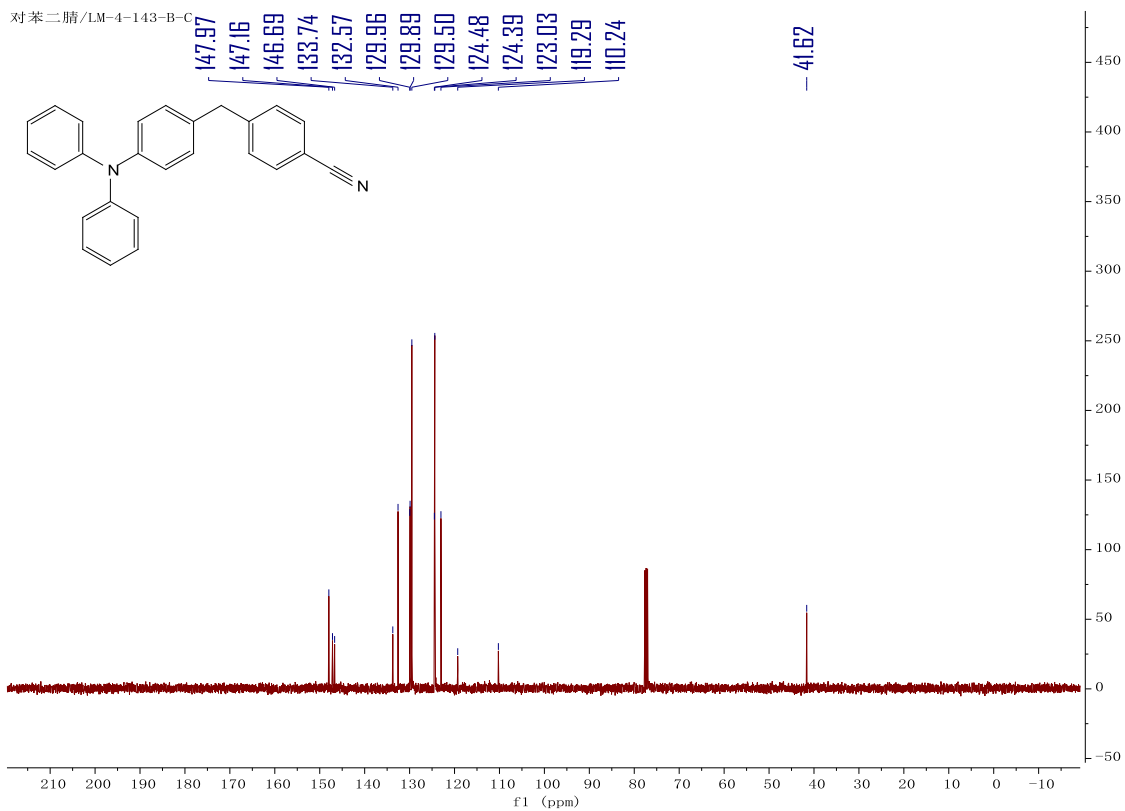


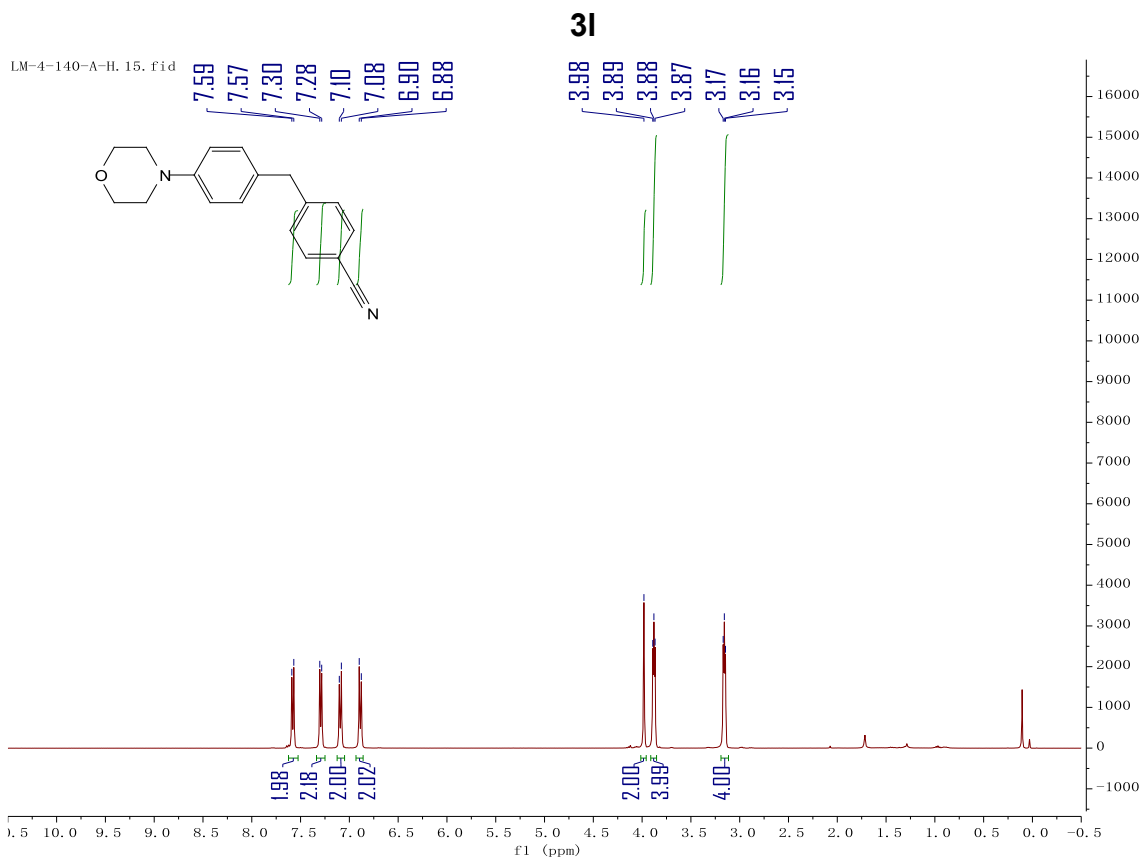
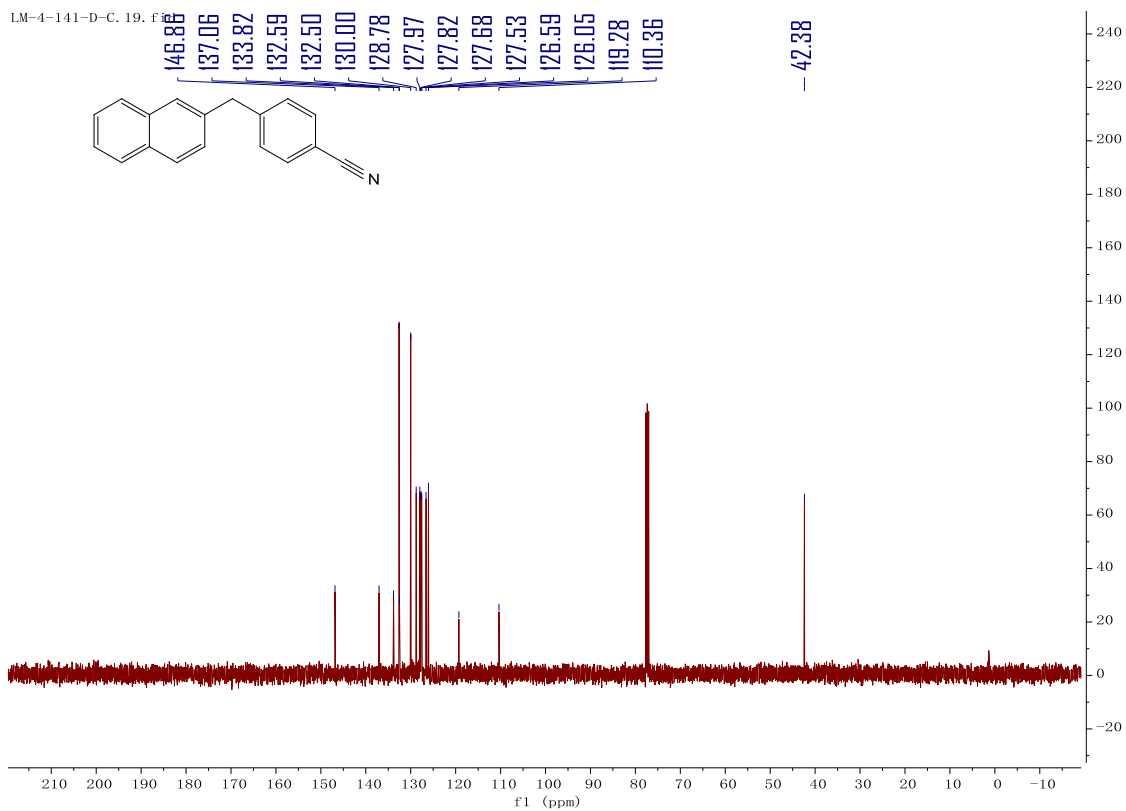
LM-4-139-B
single_pulse



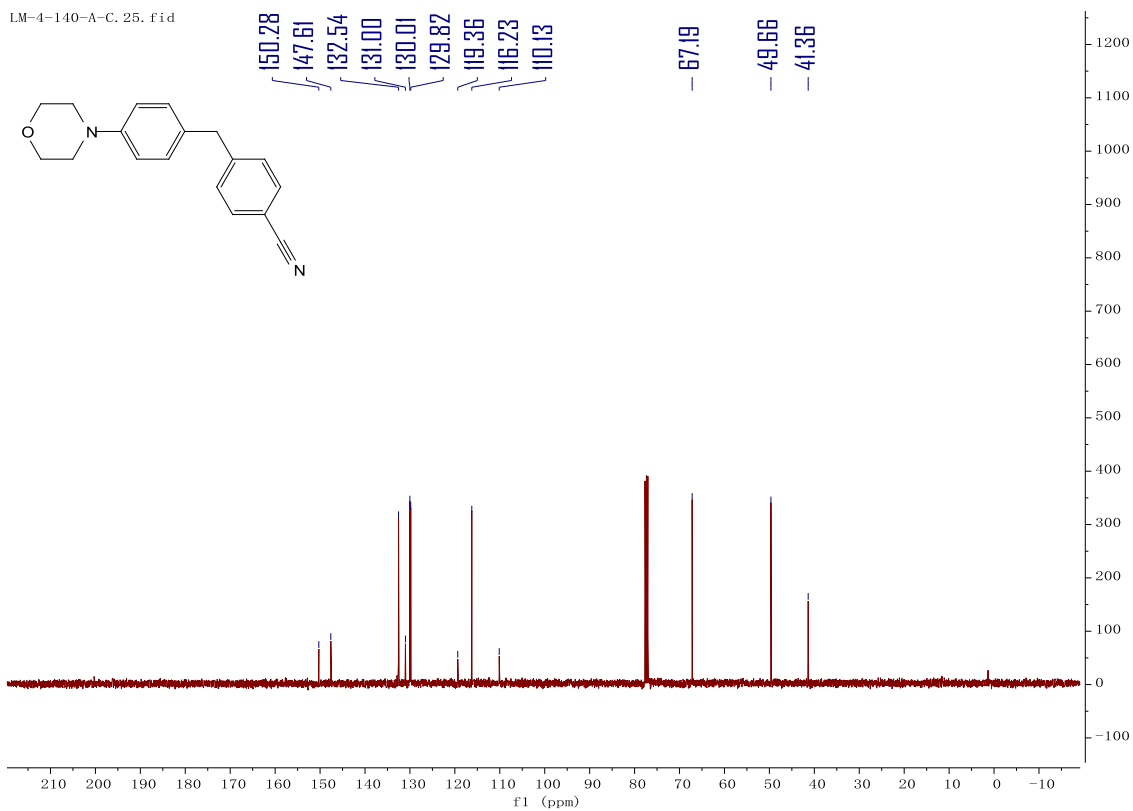
3j





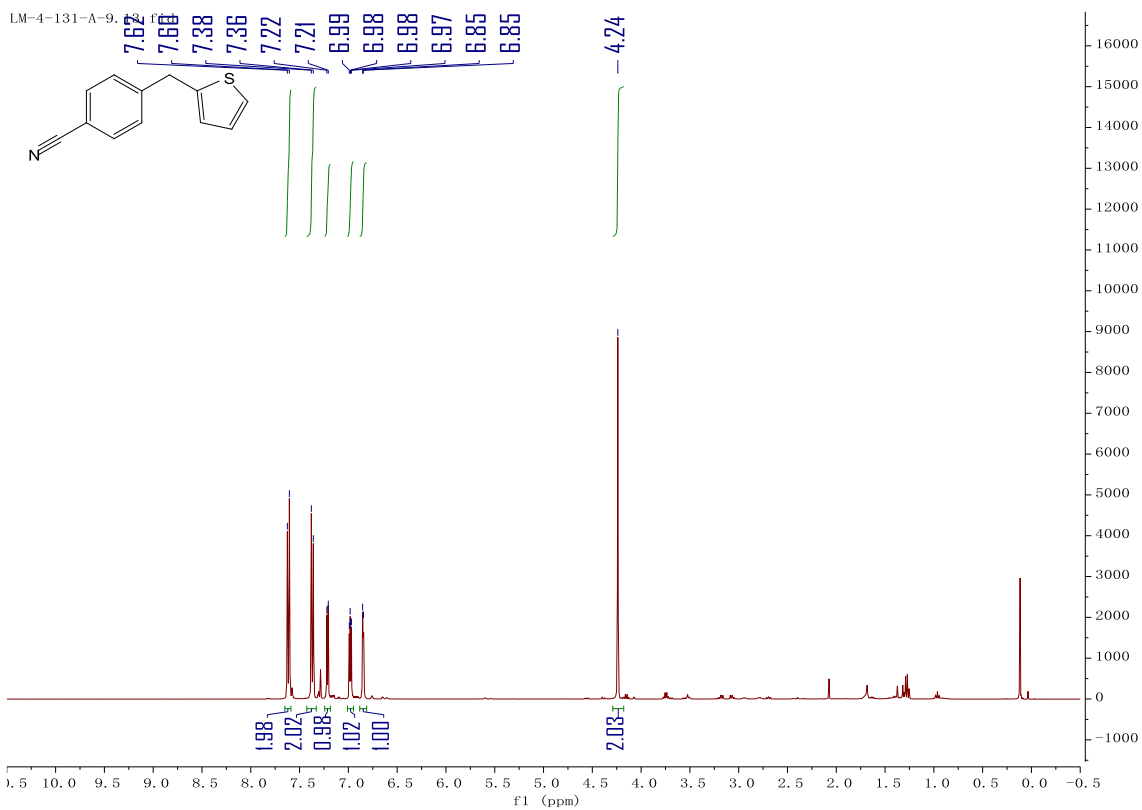


LM-4-140-A-C, 25, fid

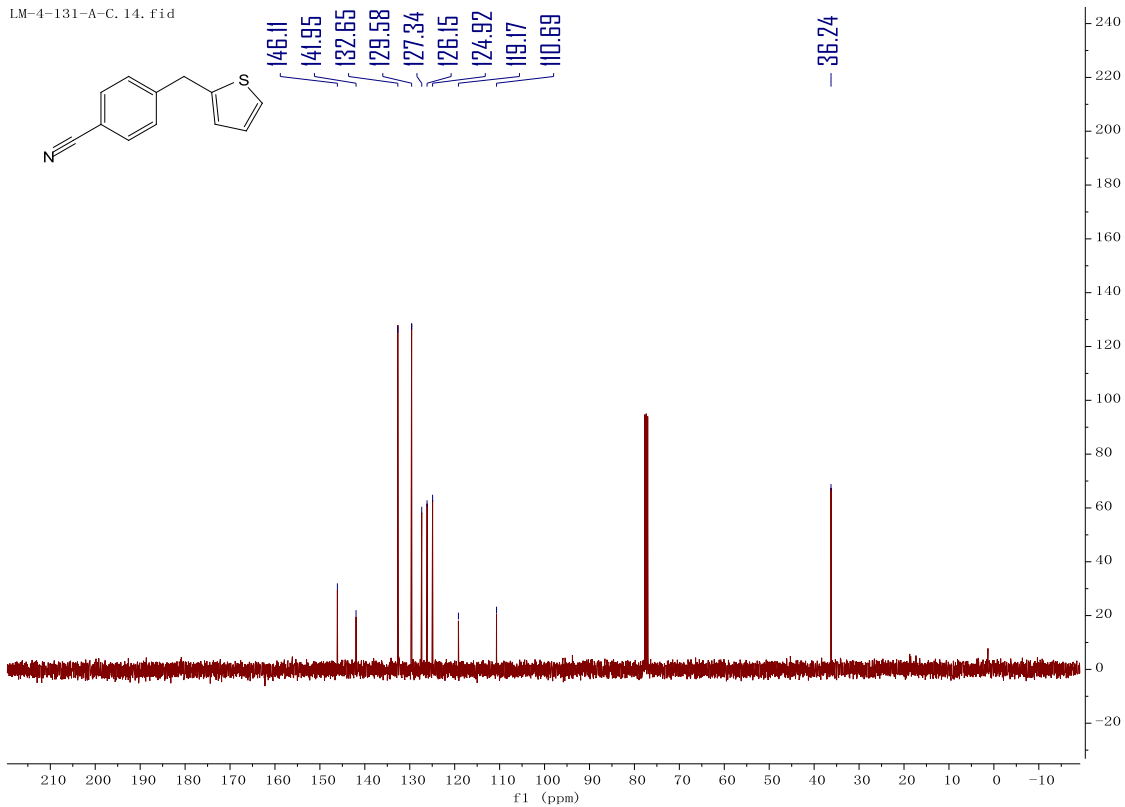


3m

LM-4-131-A-9, 25, fid

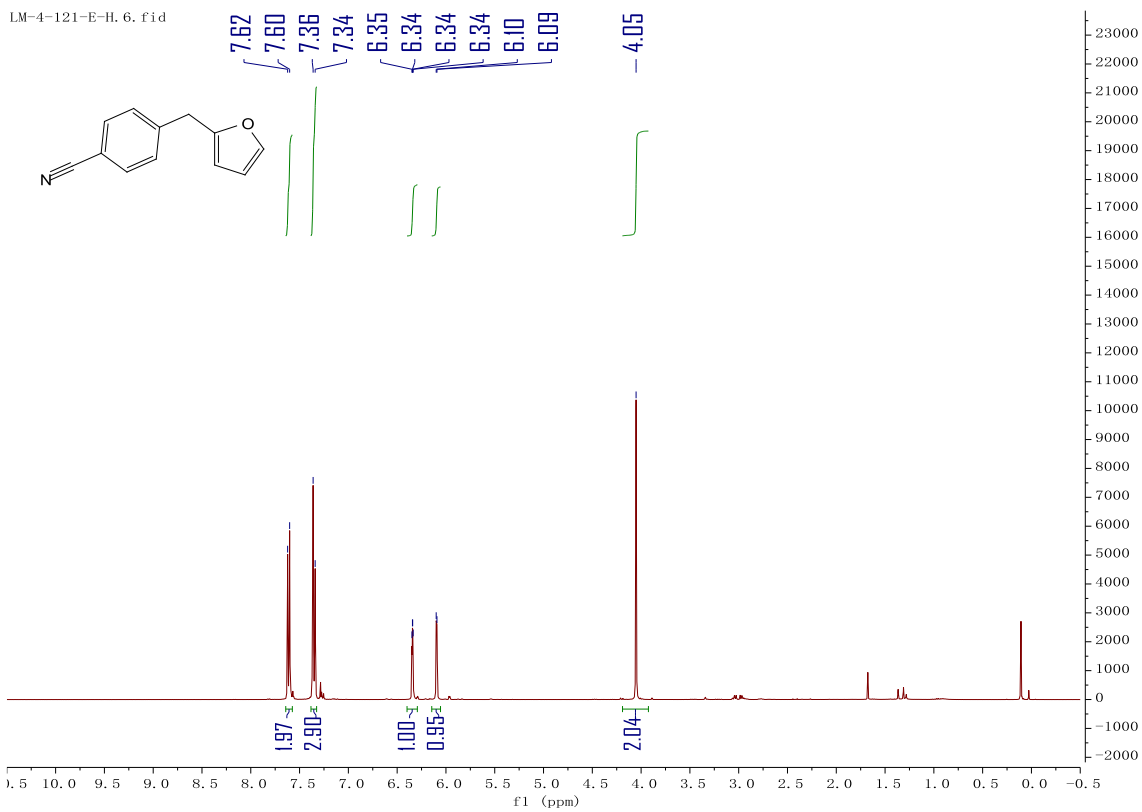


LM-4-131-A-C. 14. fid

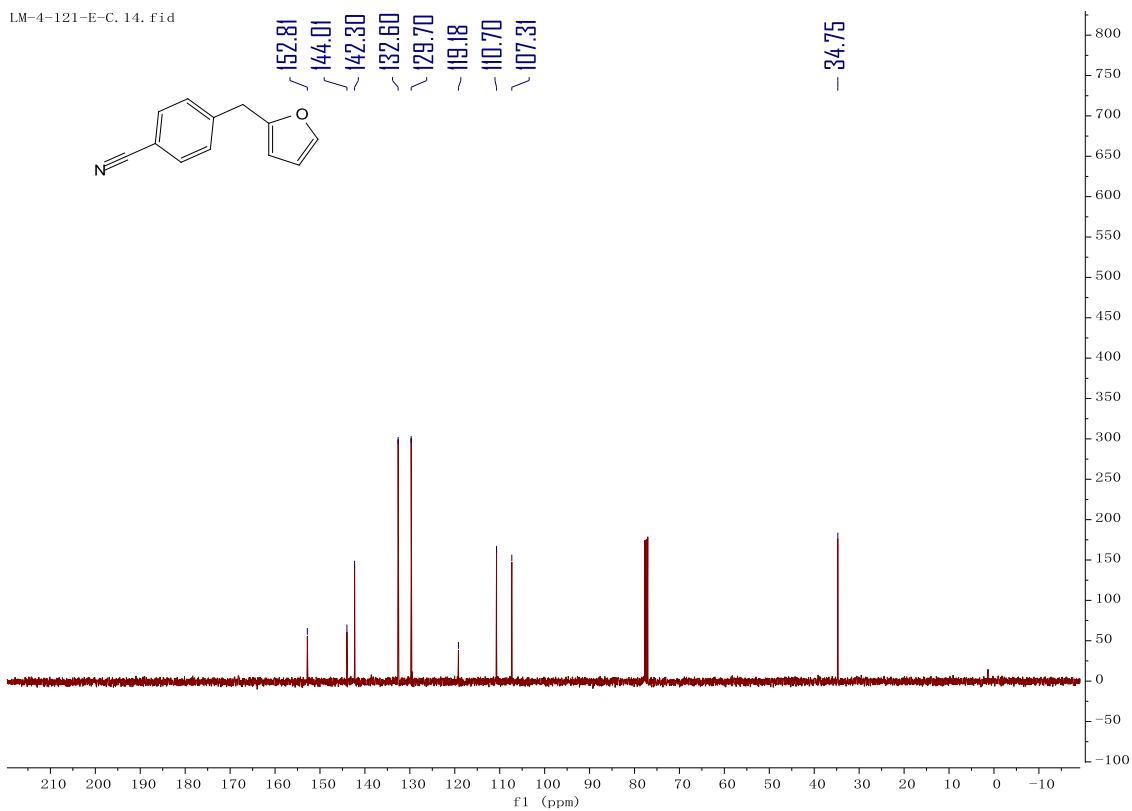


3n

LM-4-121-E-H. 6. fid

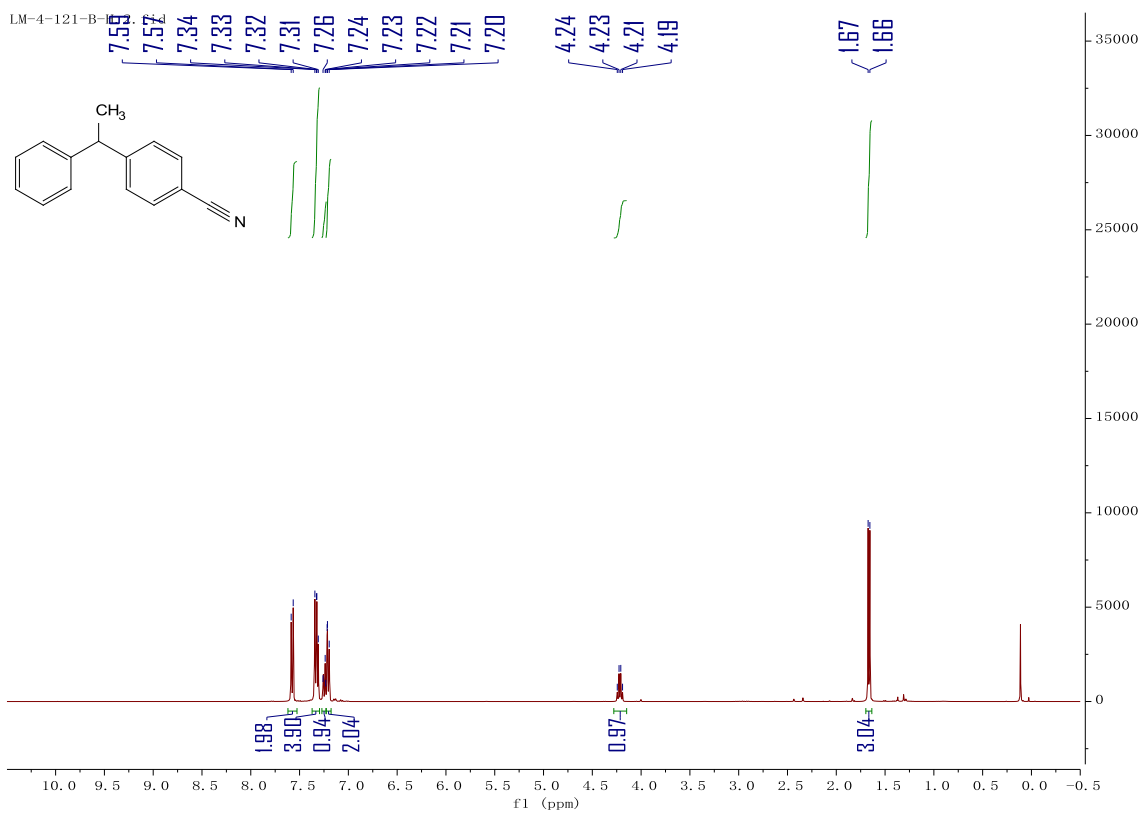


LM-4-121-E-C, 14. fid

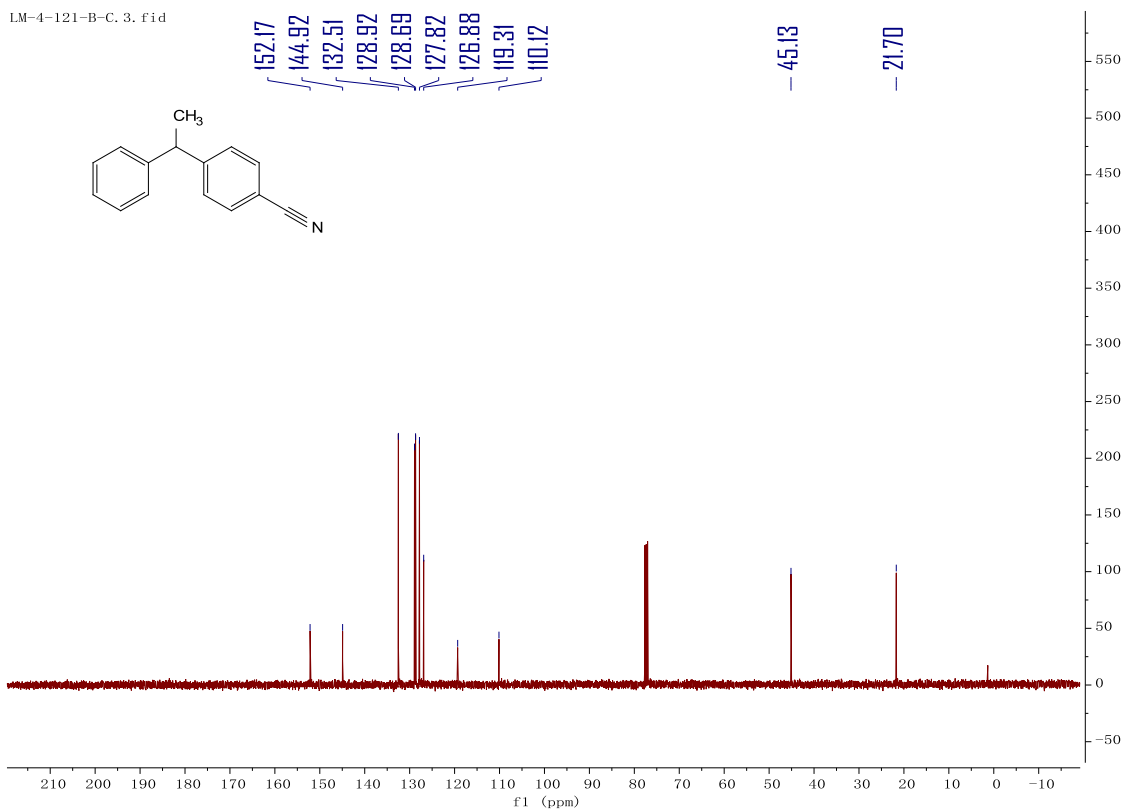


3o

LM-4-121-B-1

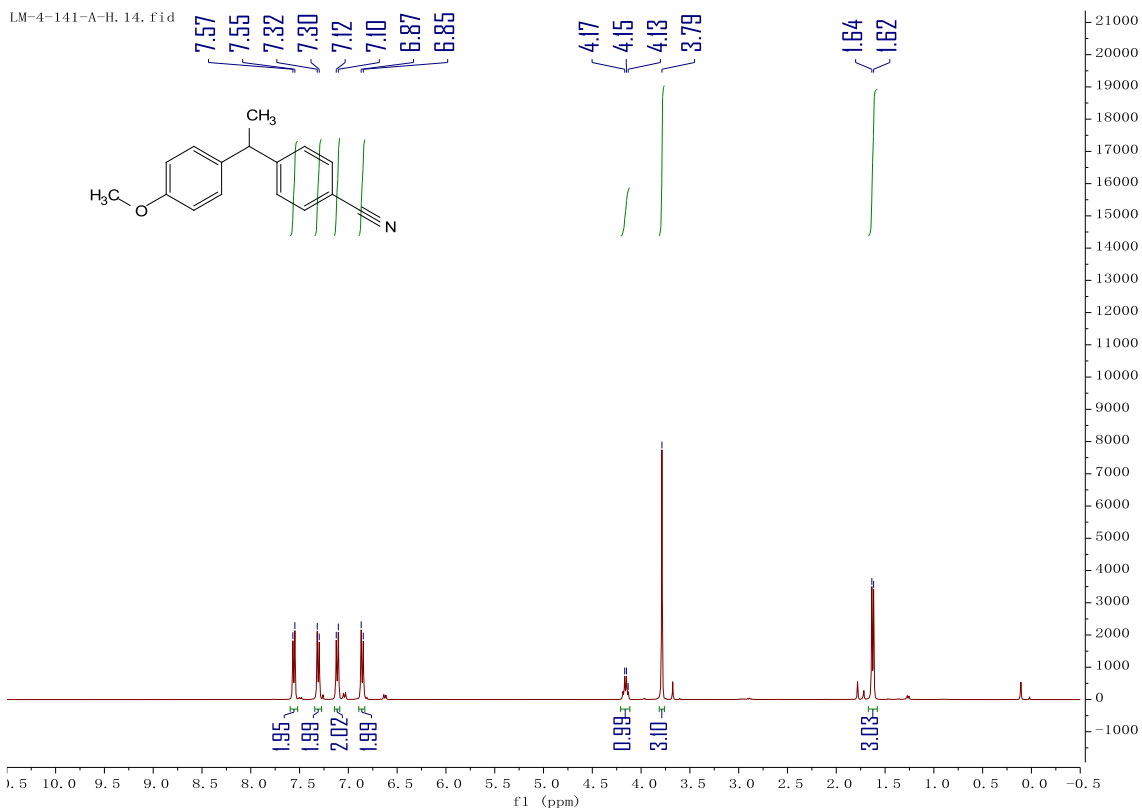


LM-4-121-B-C. 3. fid

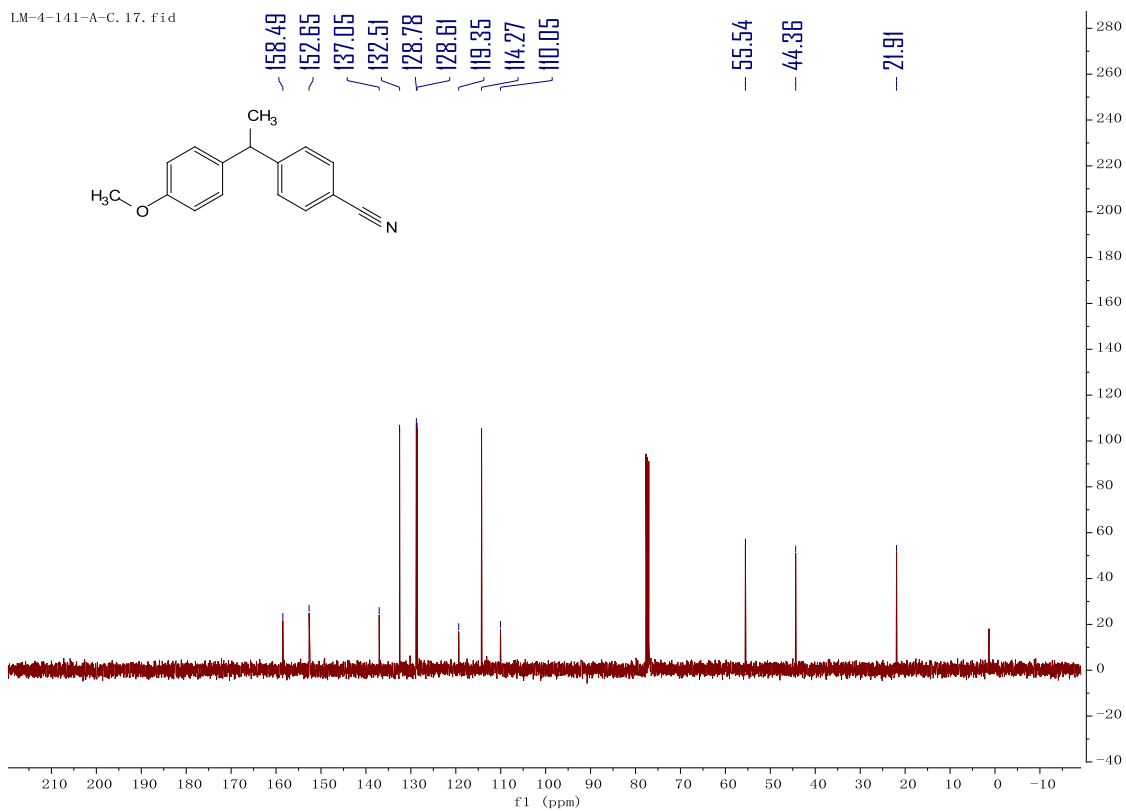


3p

LM-4-141-A-H. 14. fid

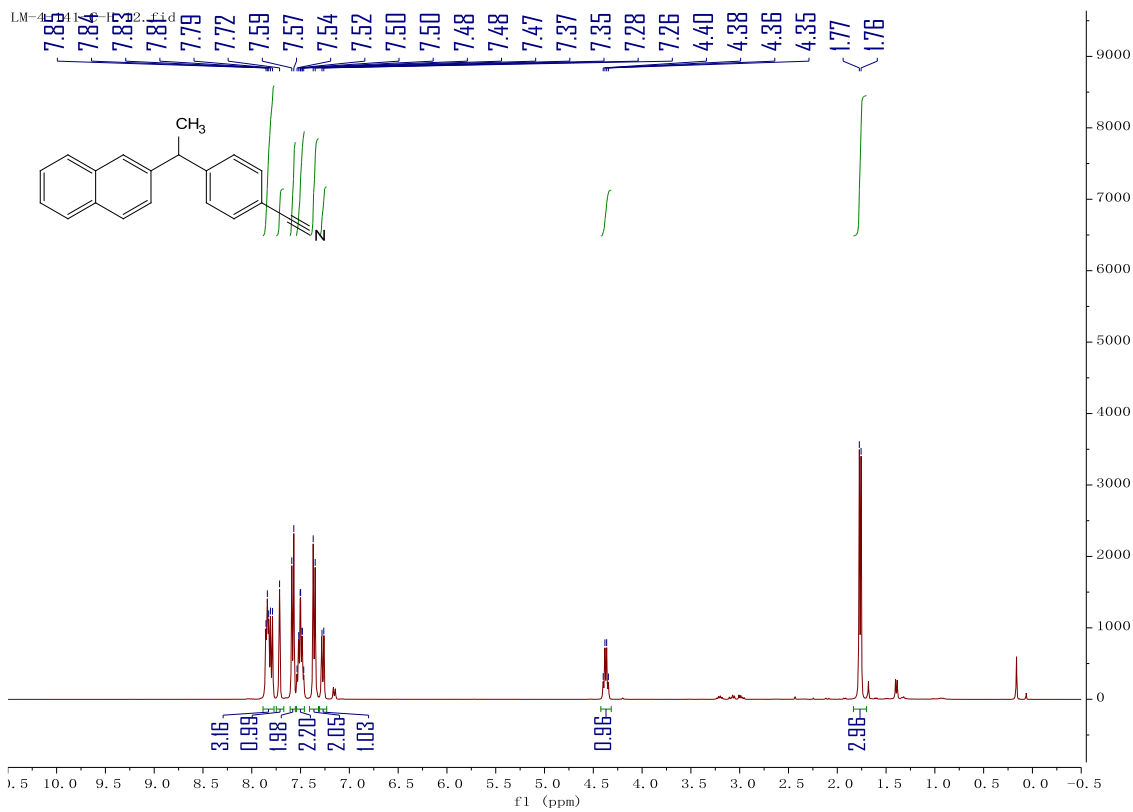


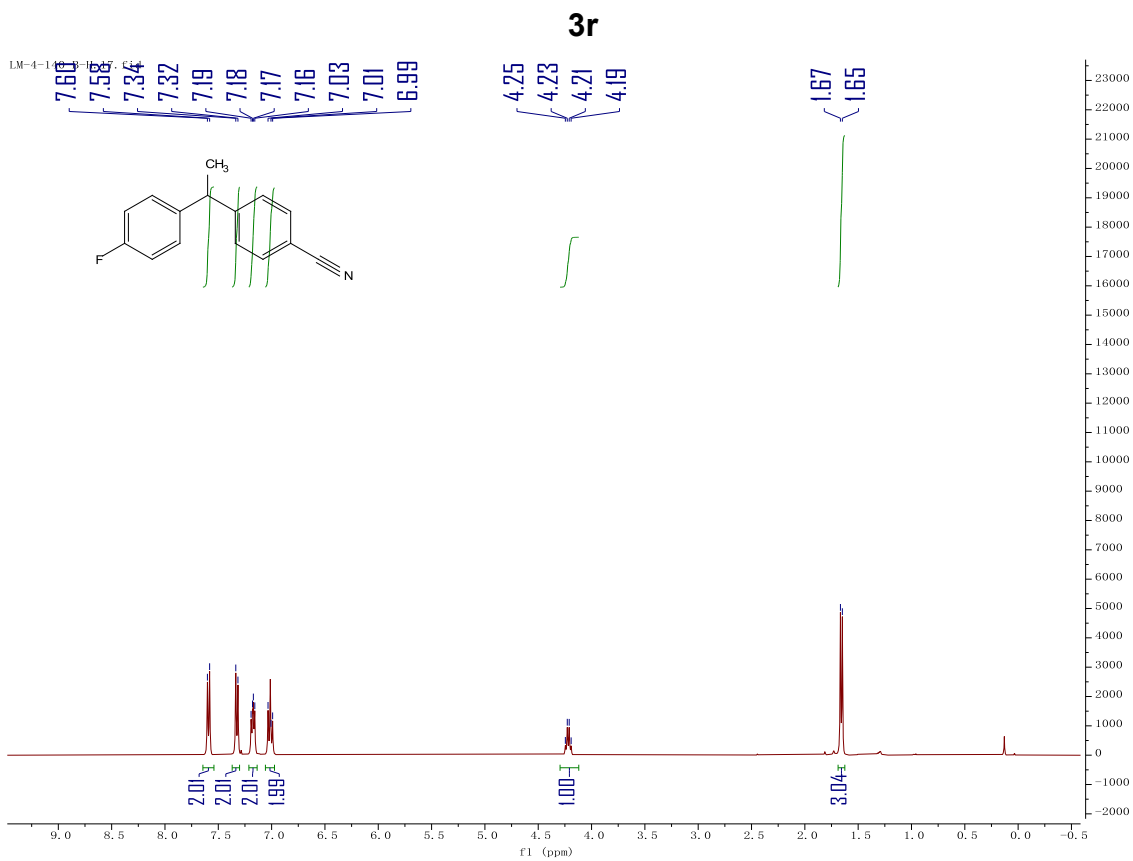
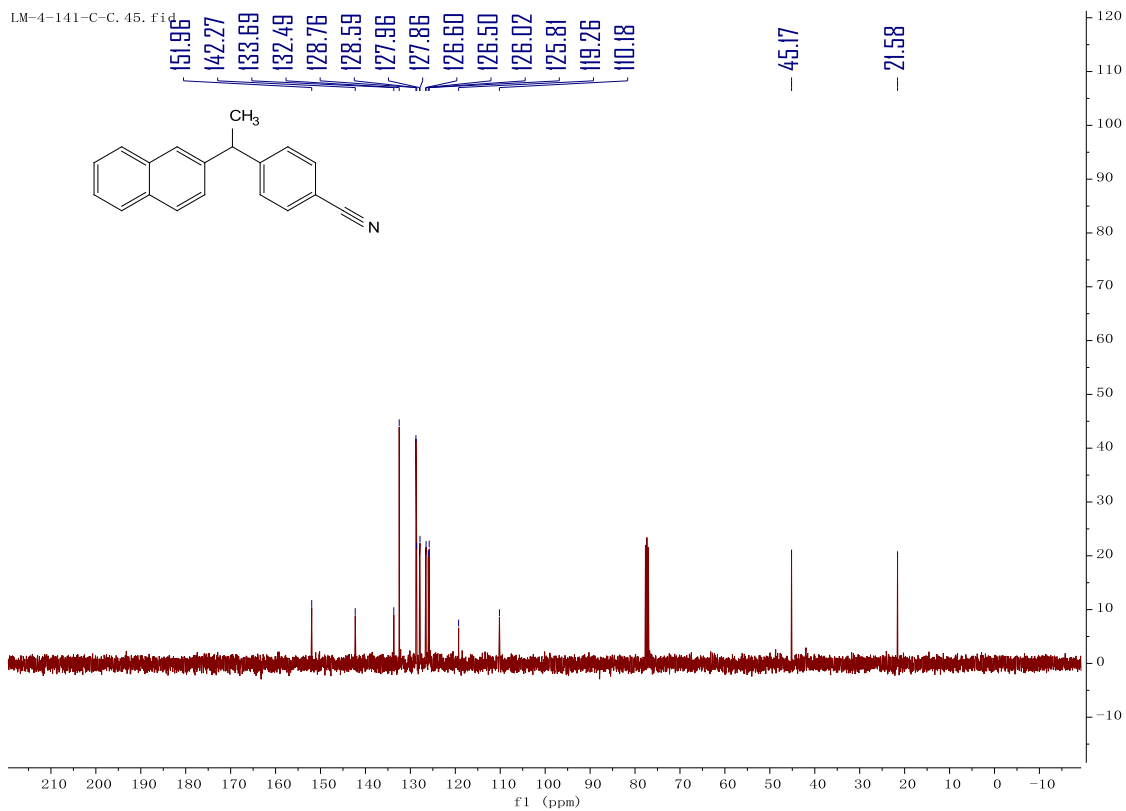
LM-4-141-A-C, 17, fid



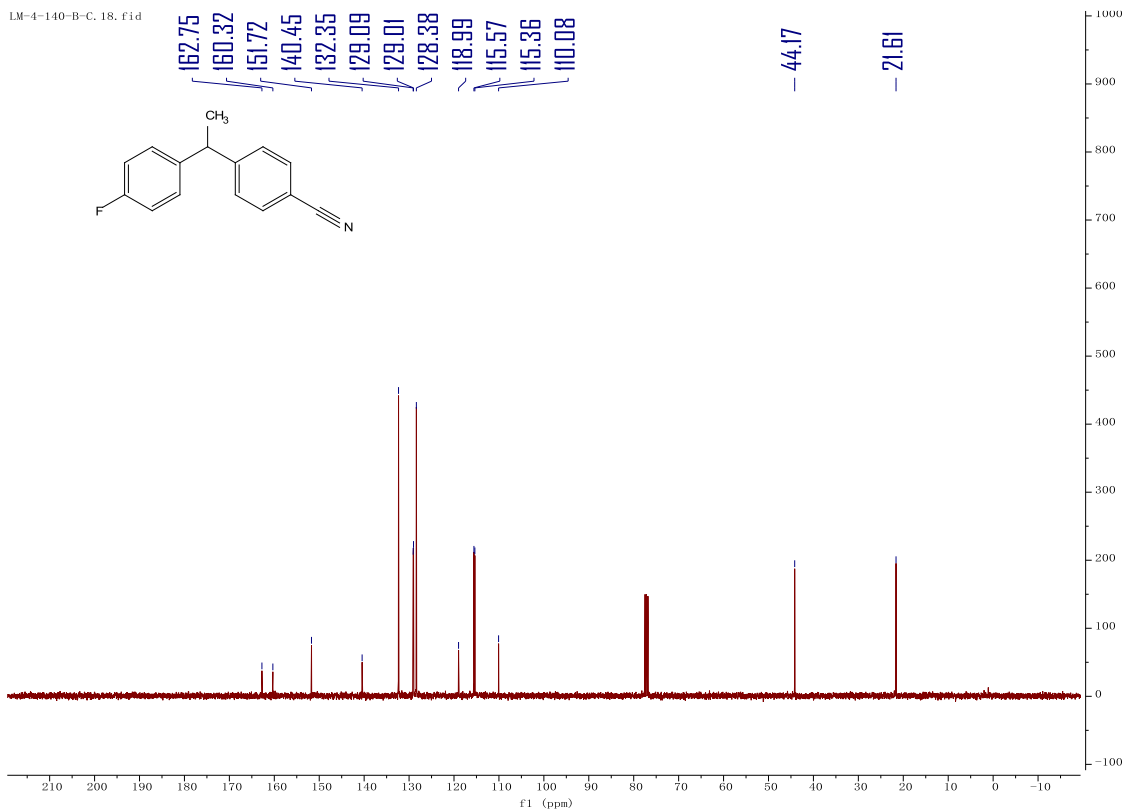
3q

LM-4-141-A-C, 17, fid

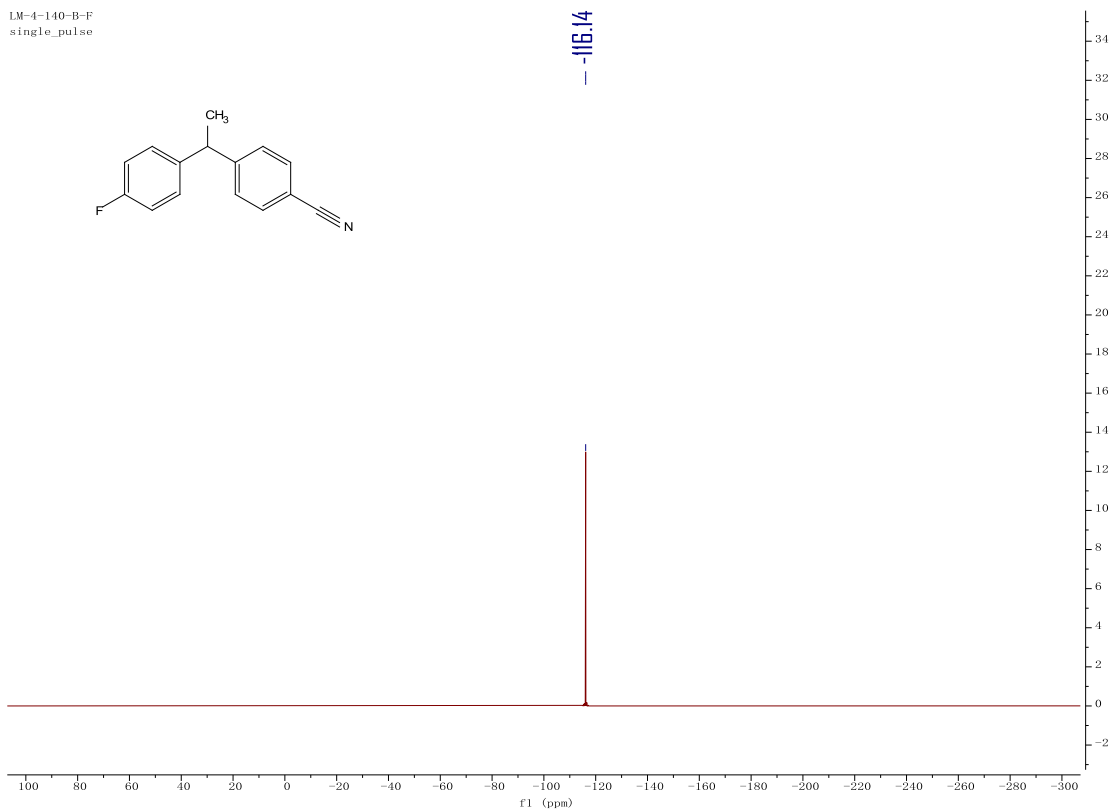


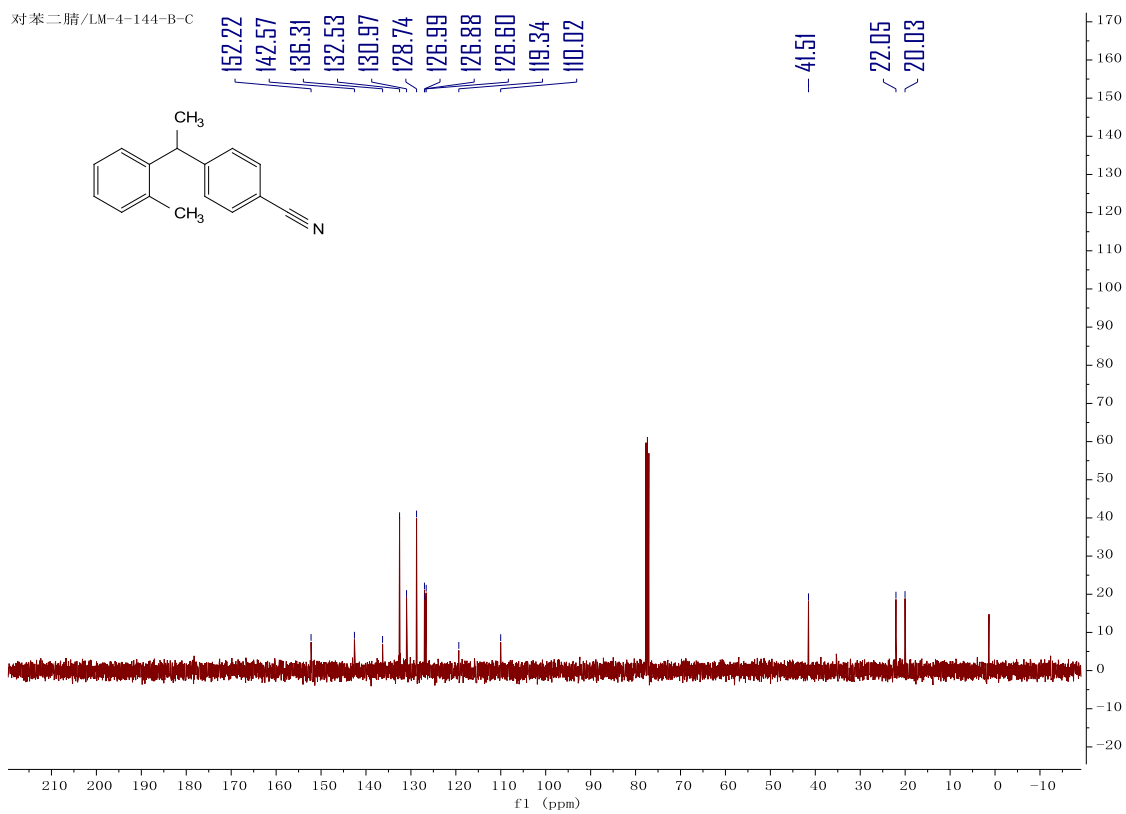
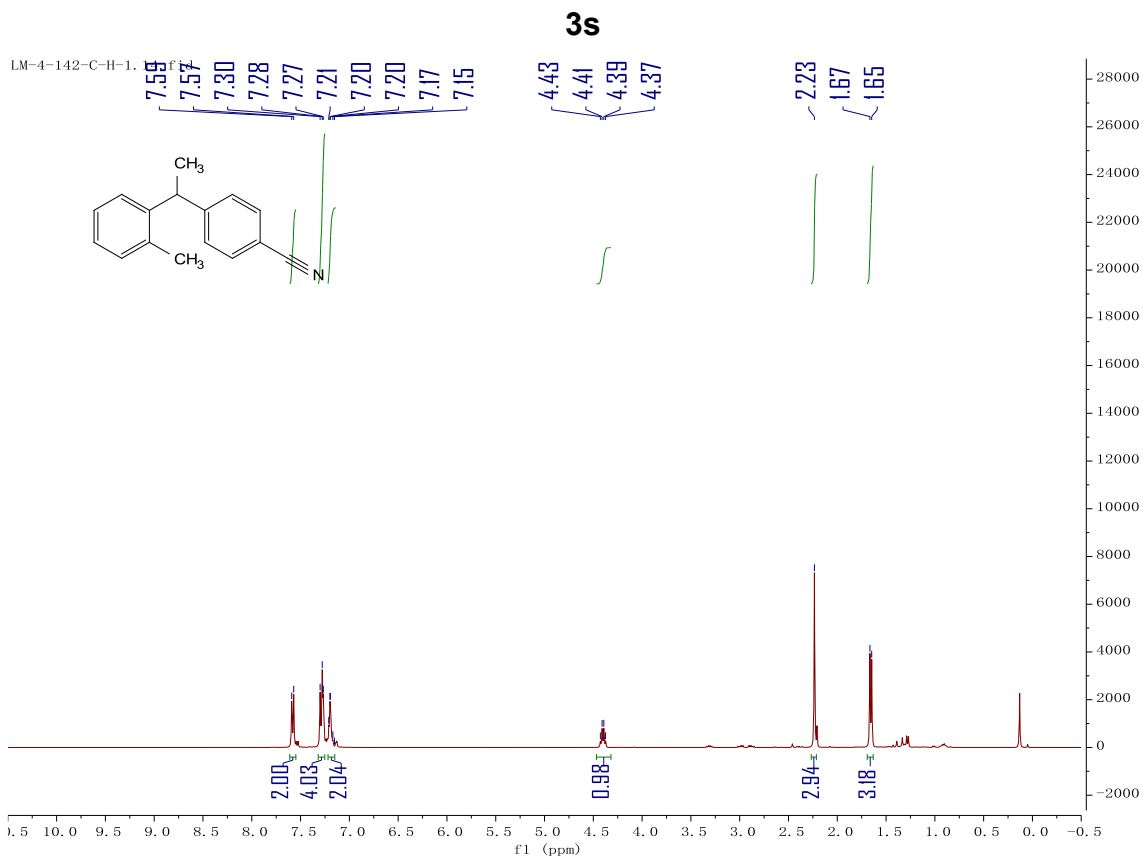


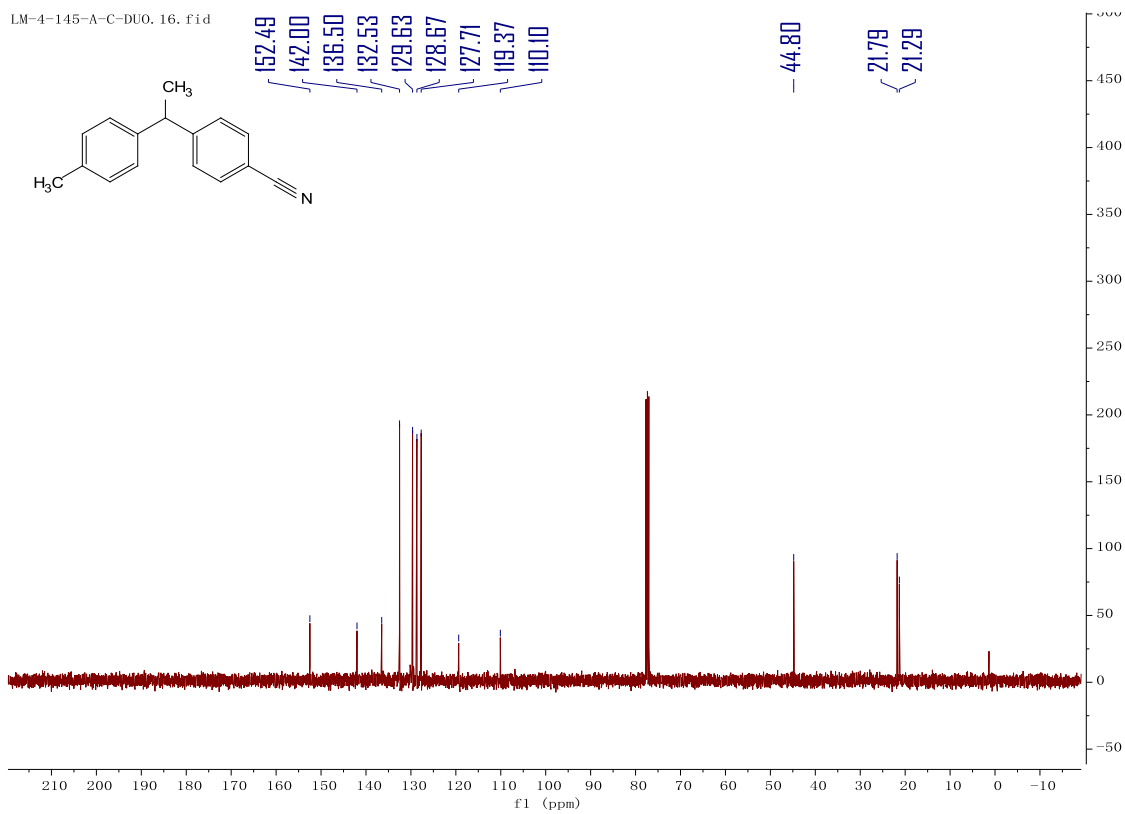
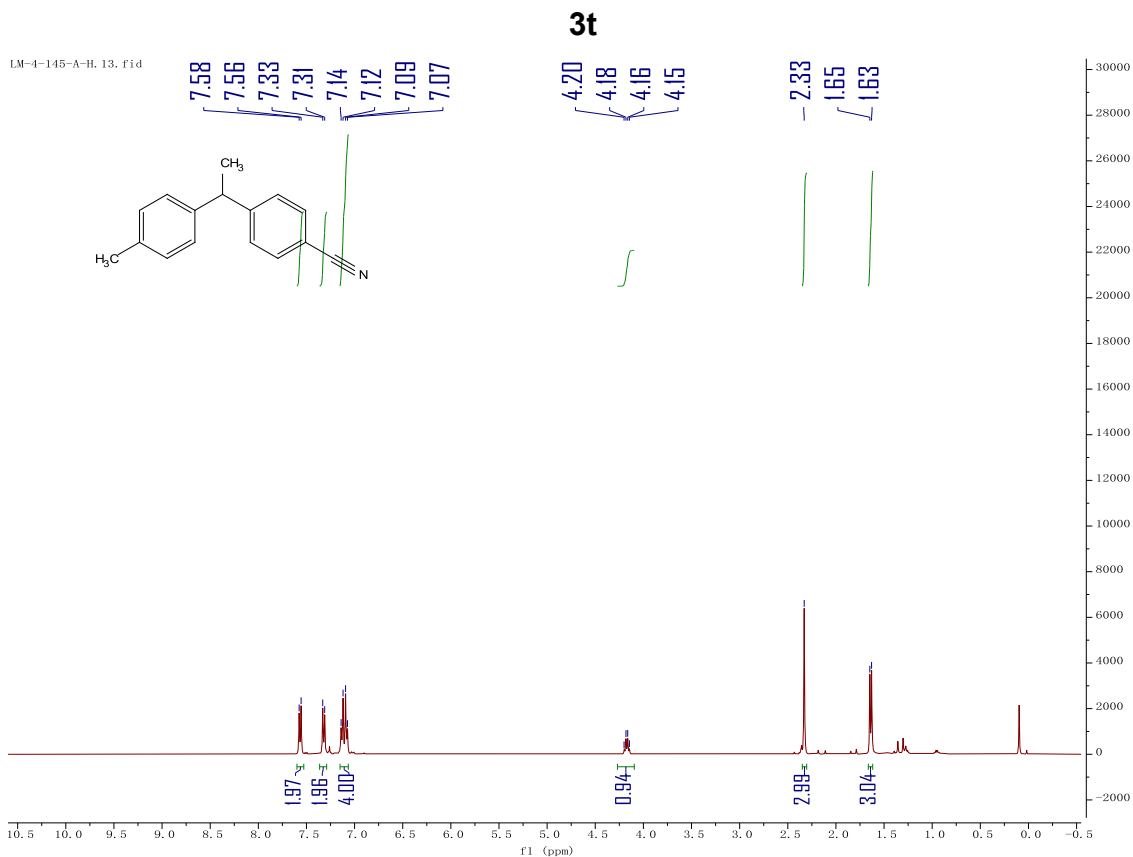
LM-4-140-B-C, 18, f1d



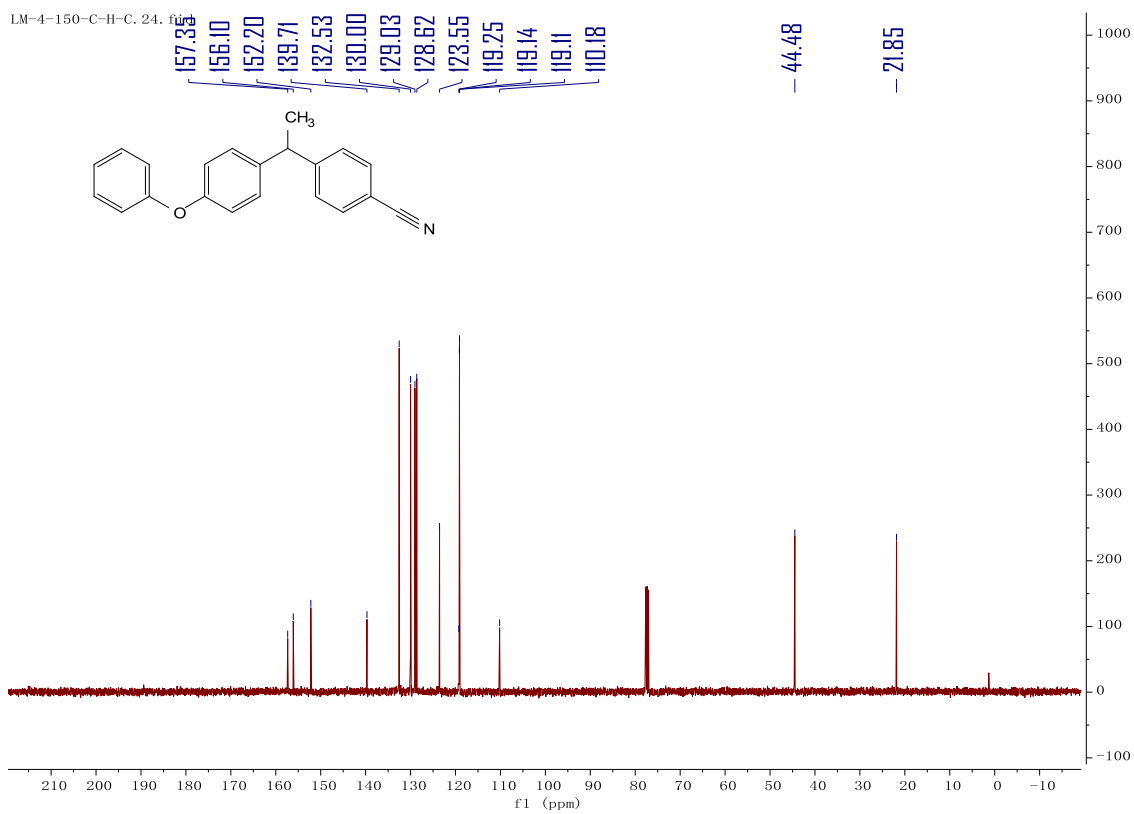
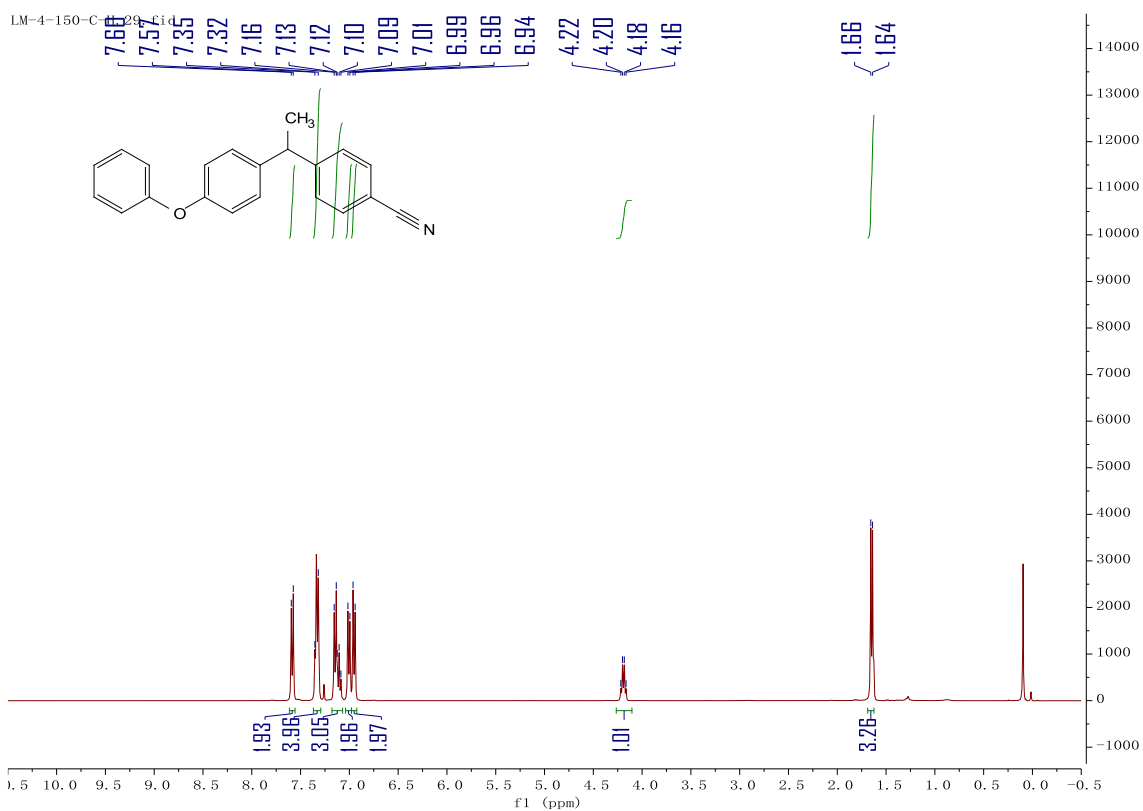
LM-4-140-B-F
single_pulse

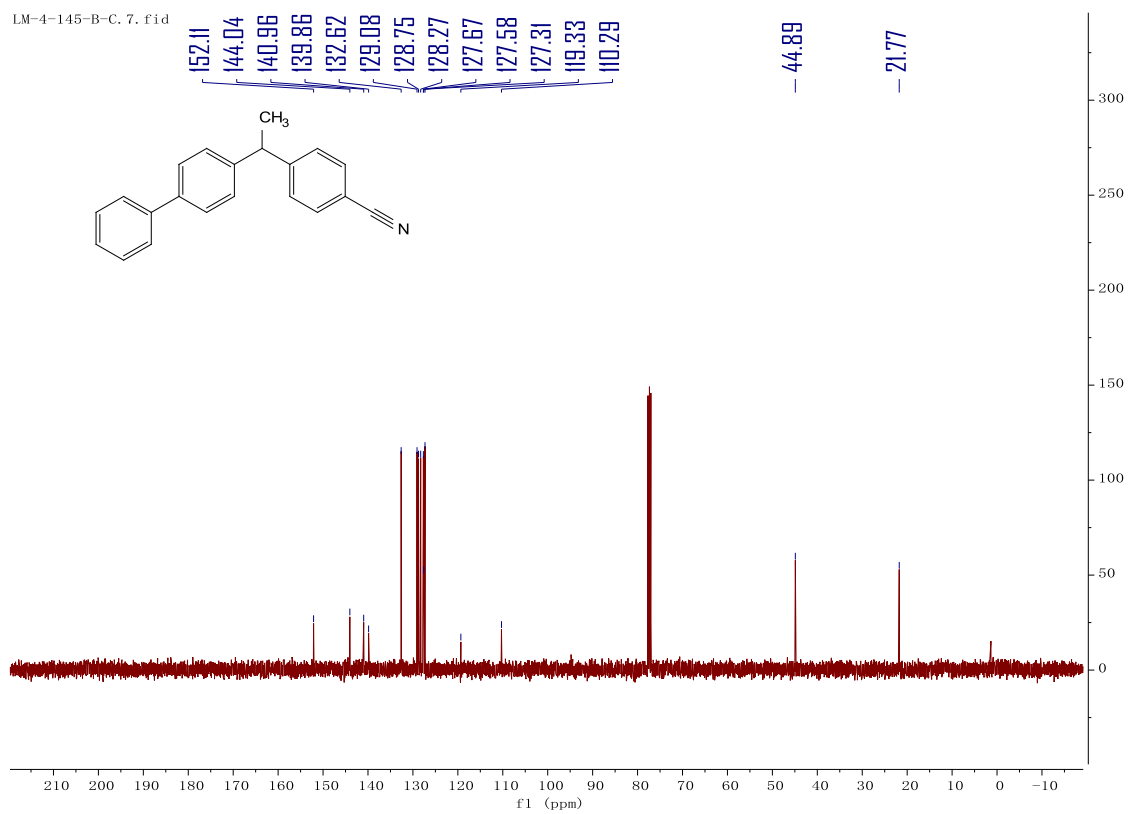
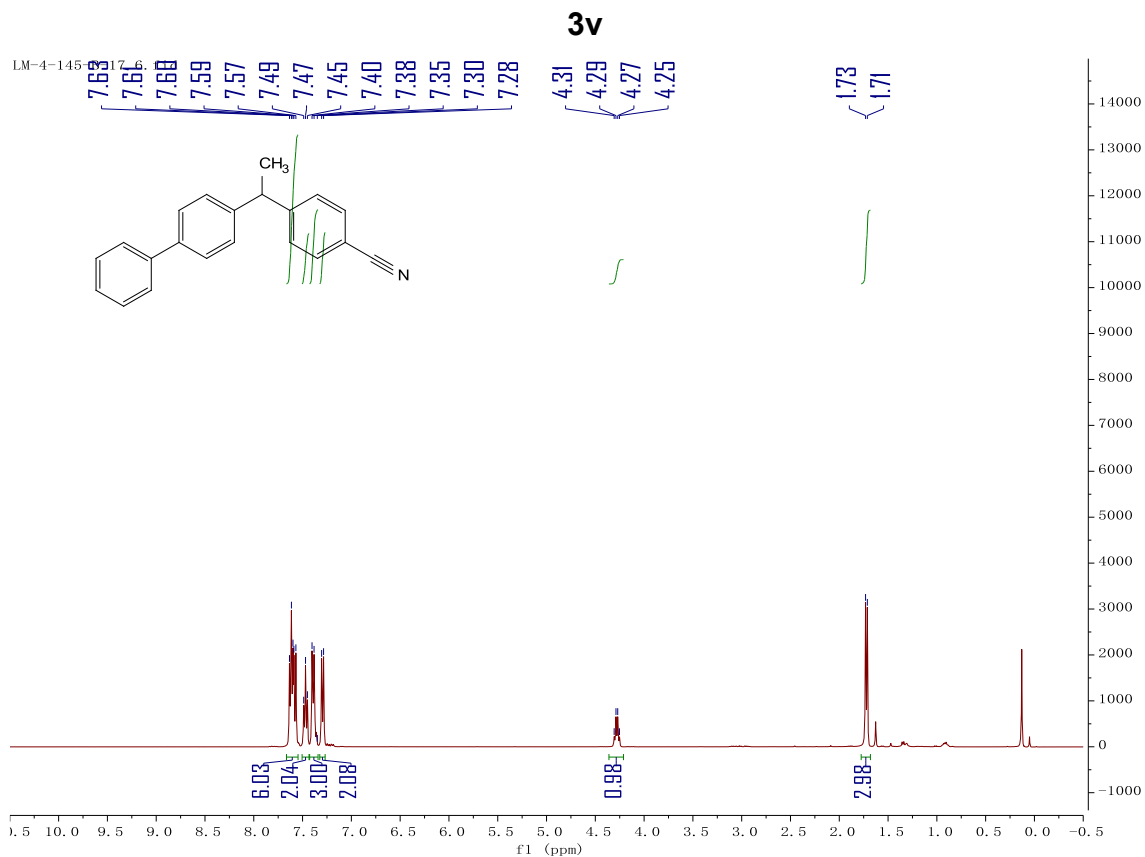


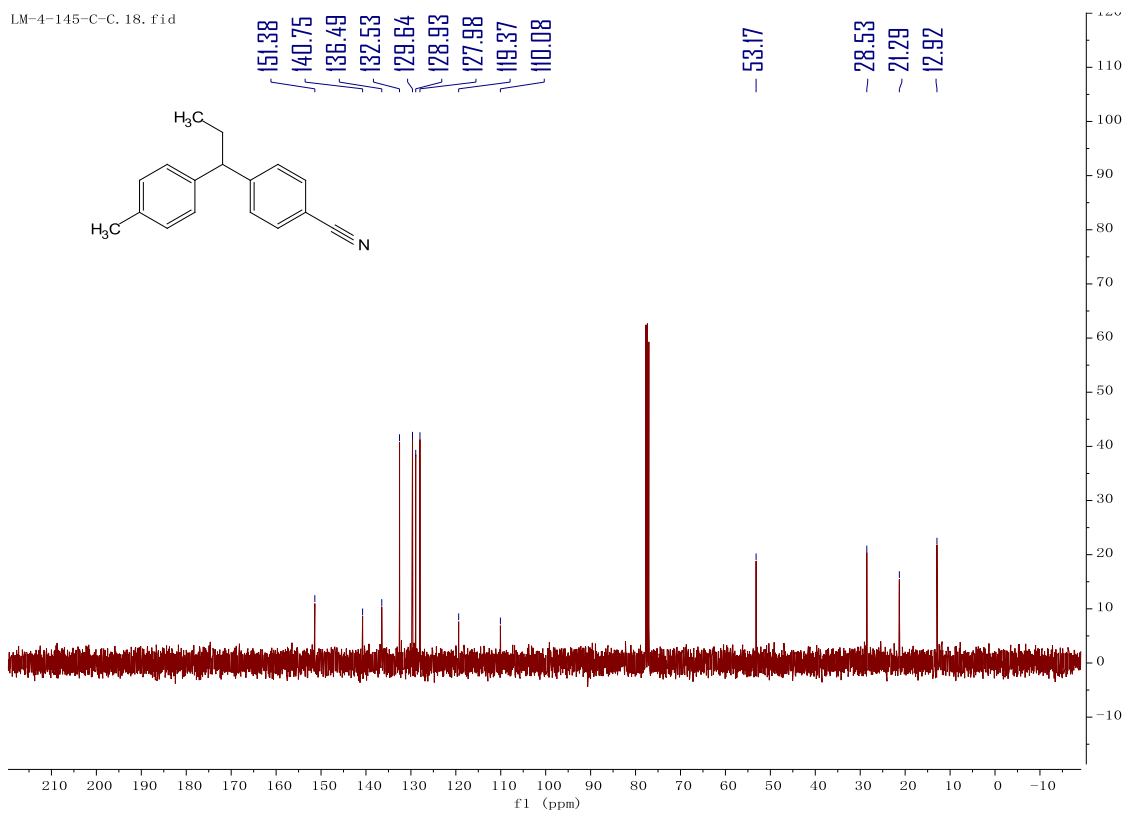
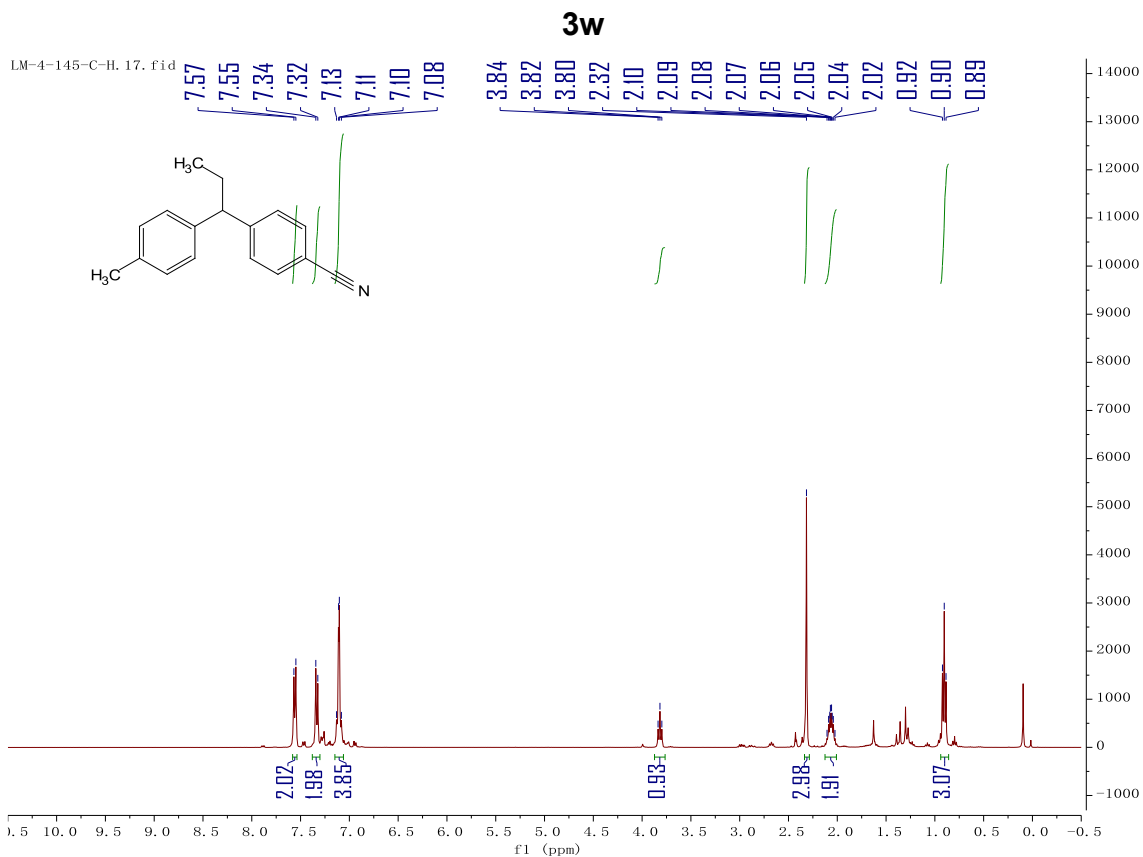


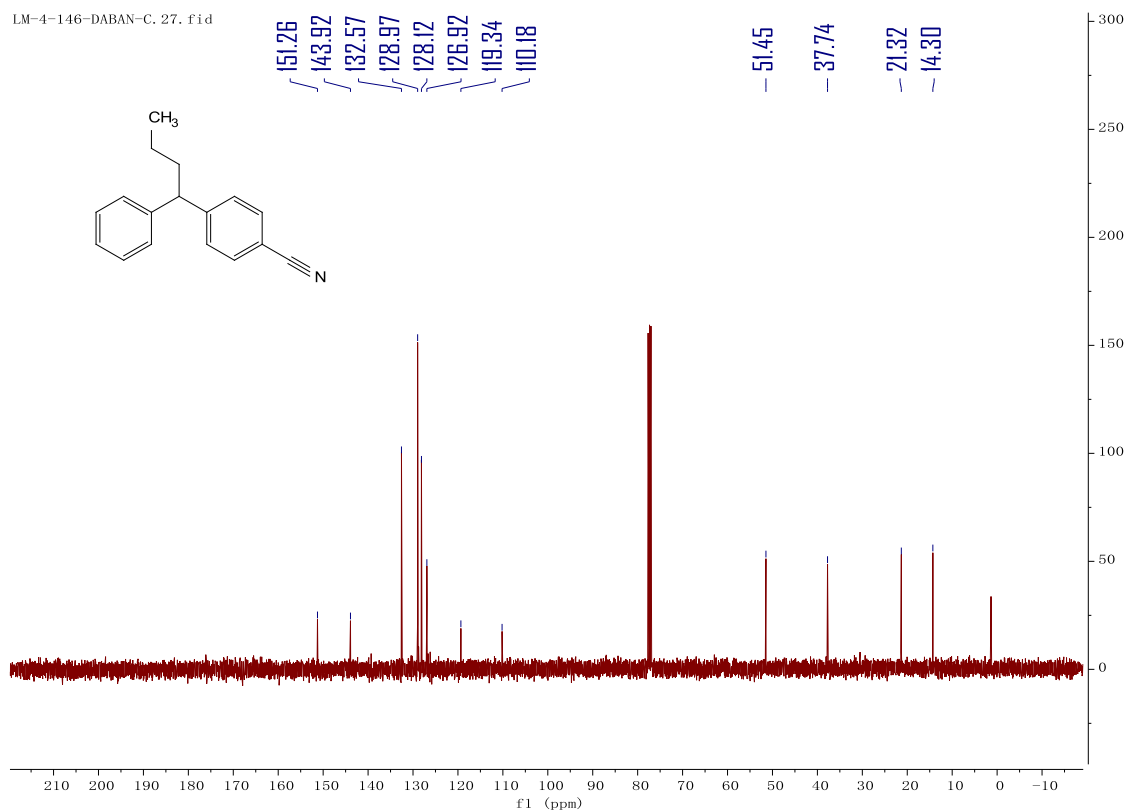
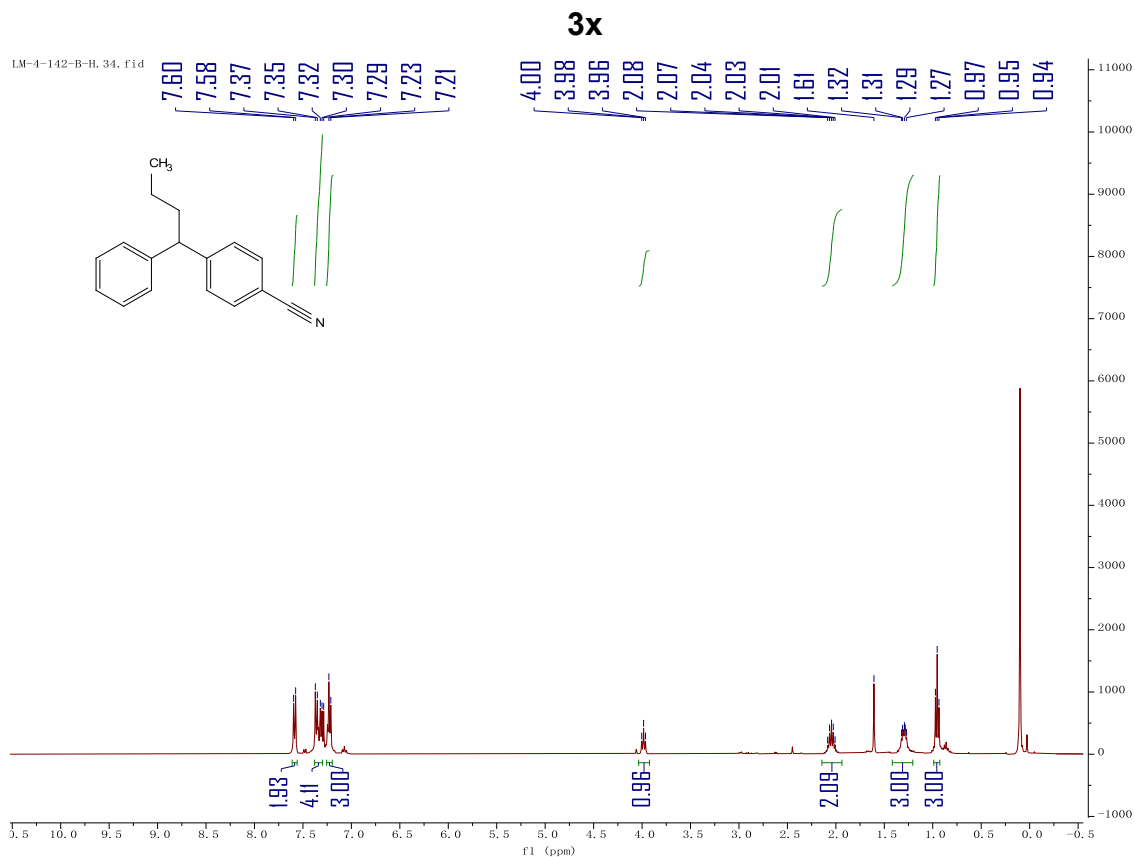


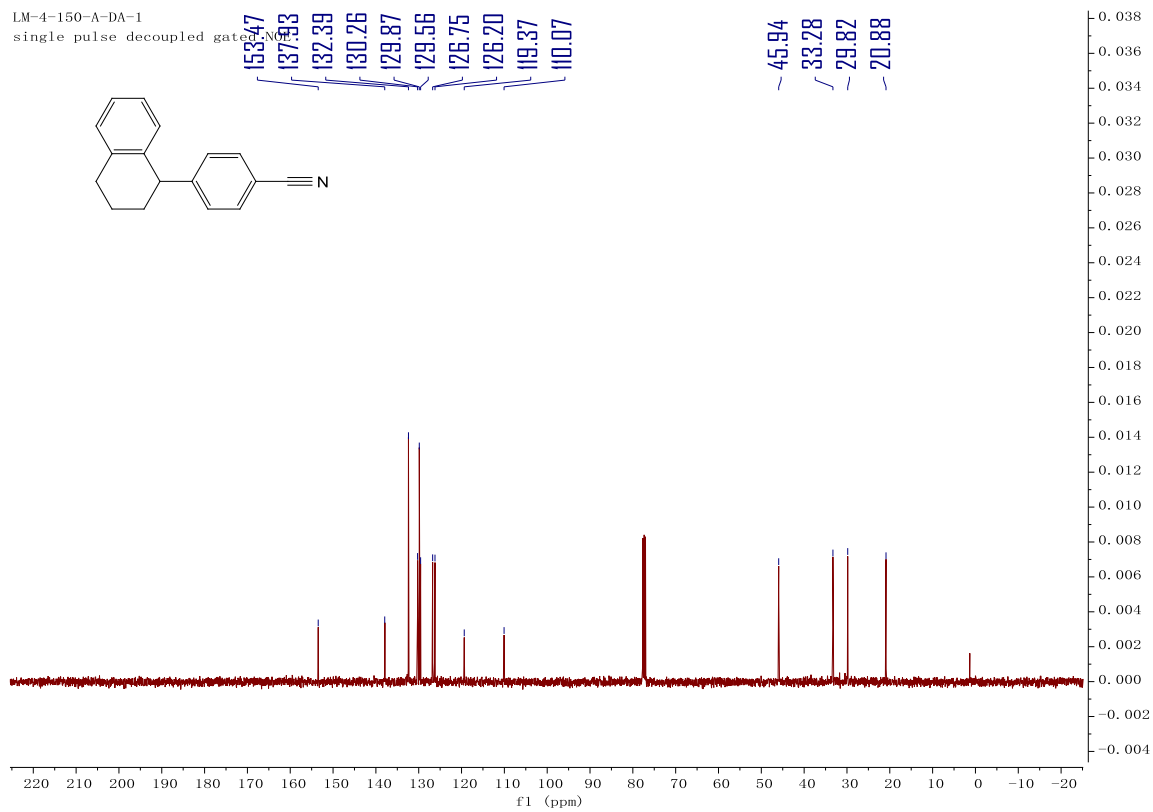
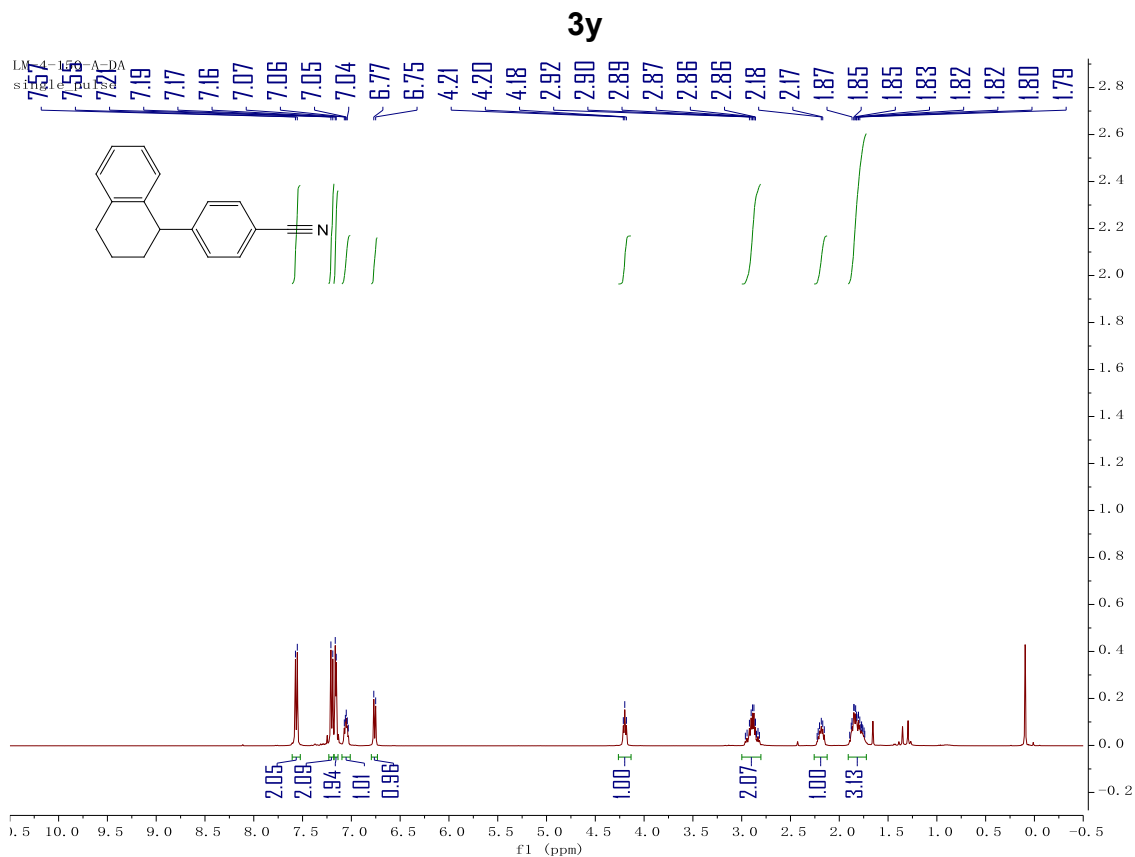
3u



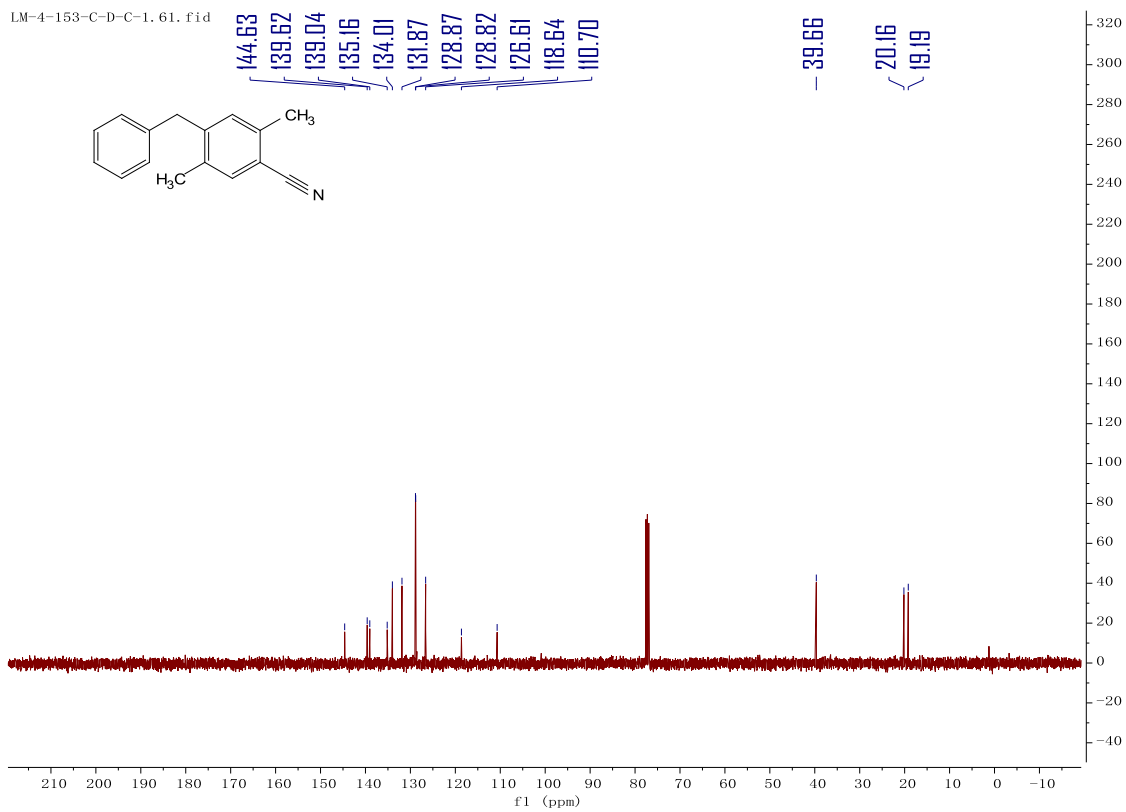
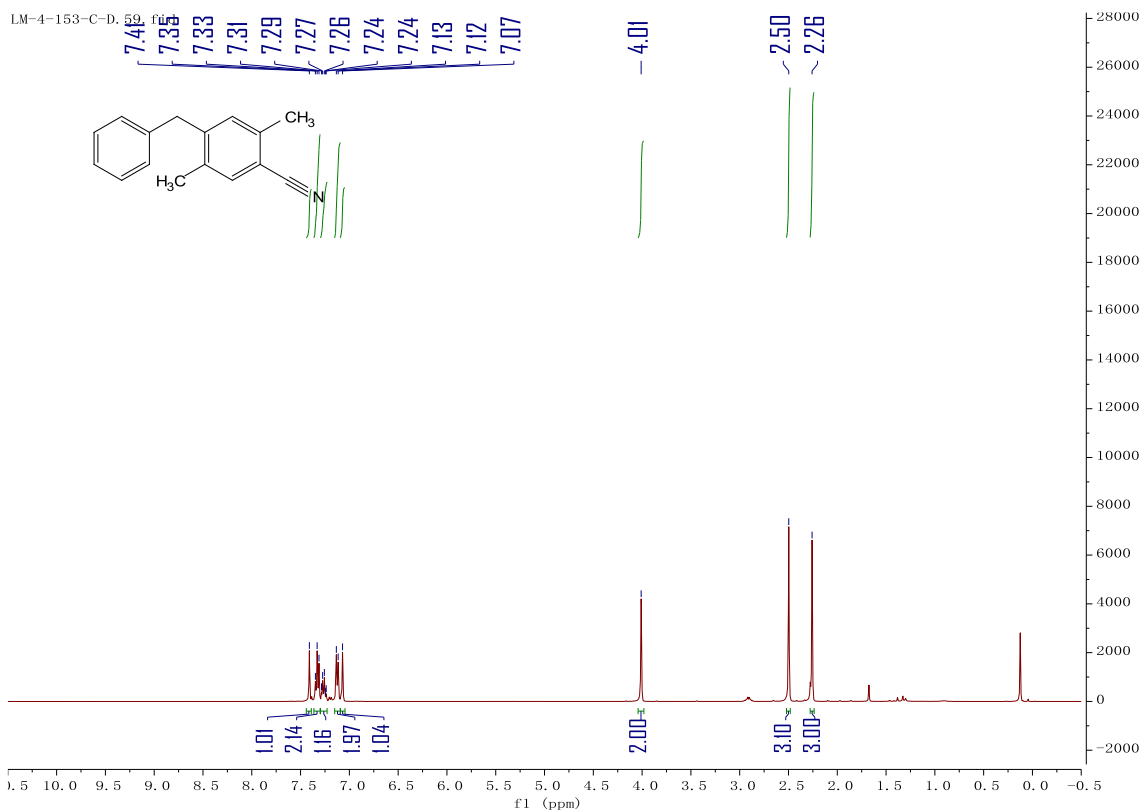




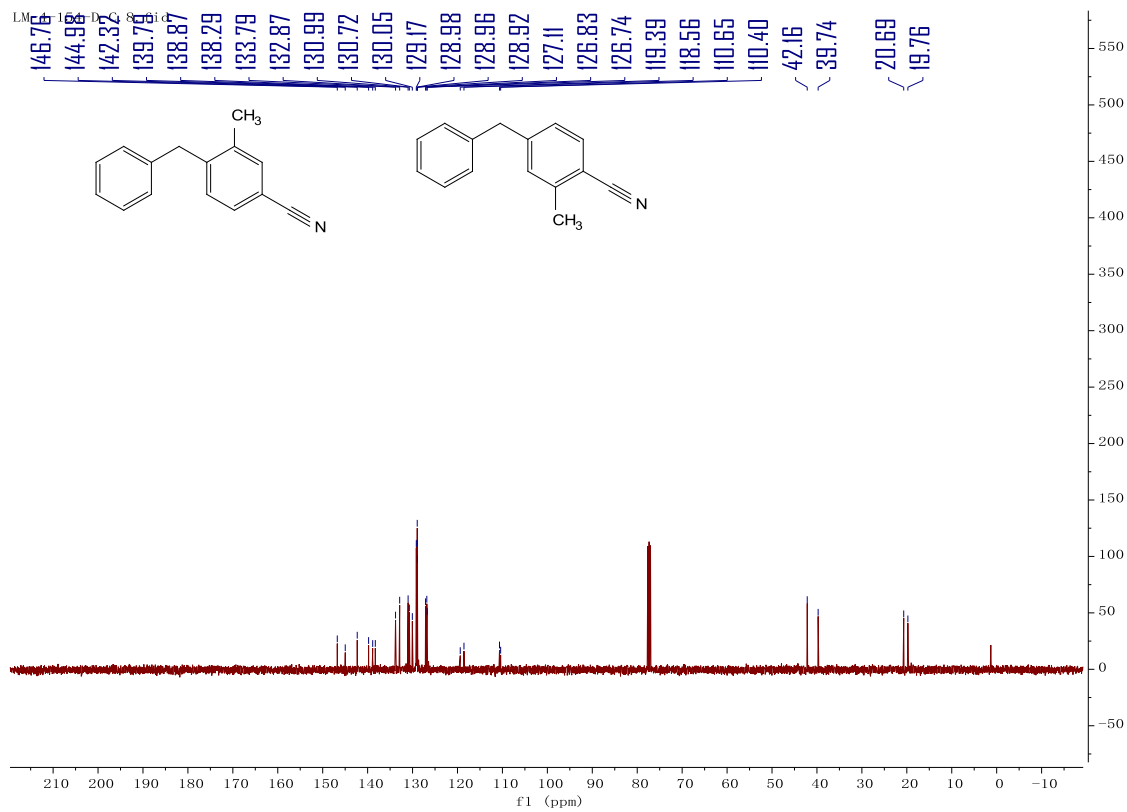
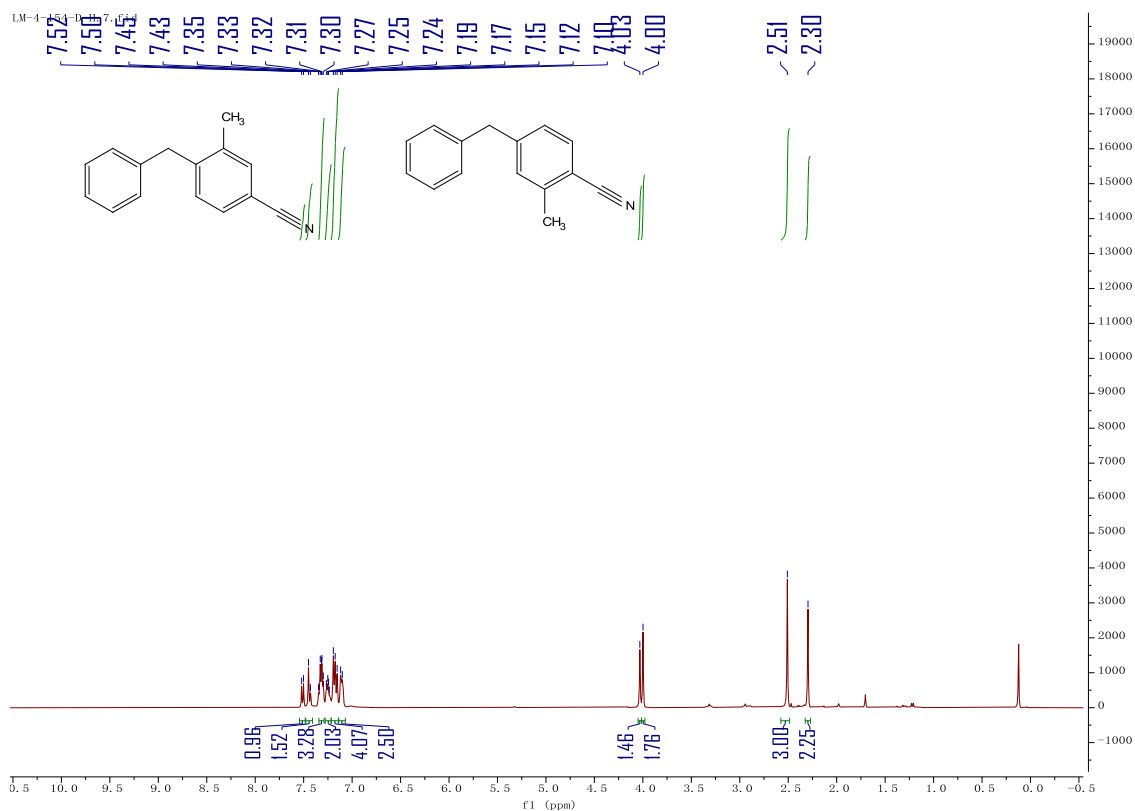




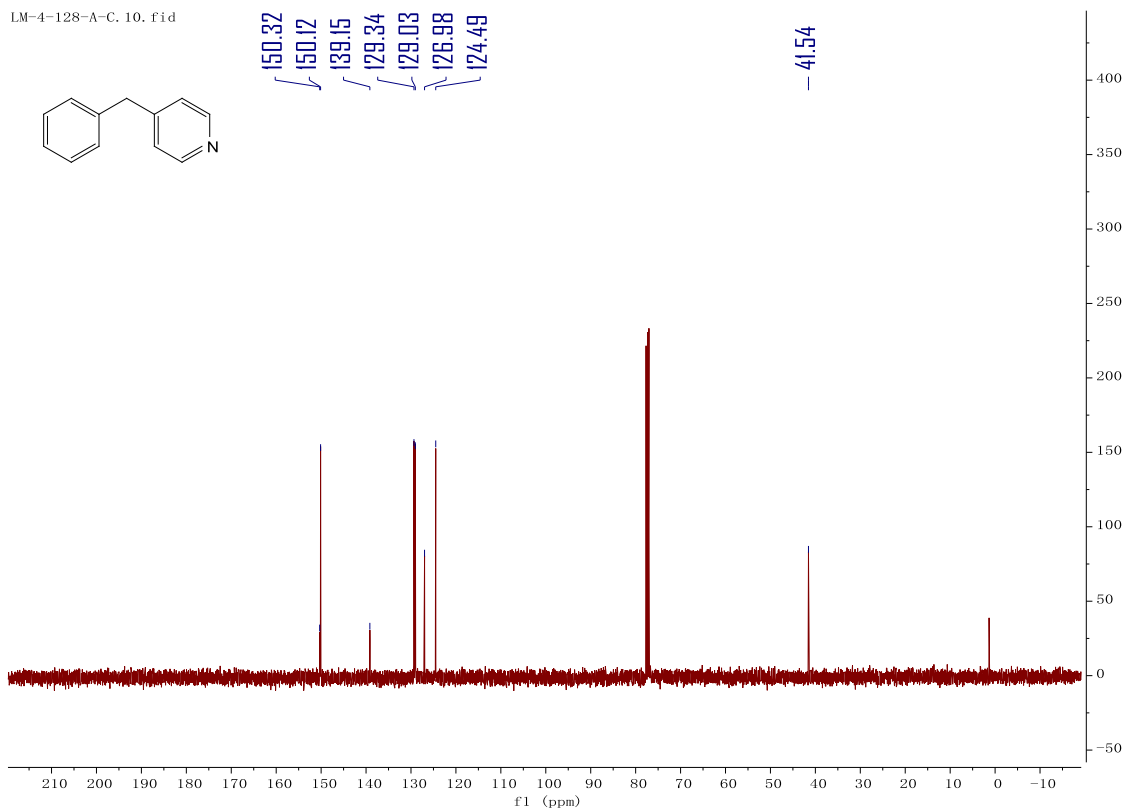
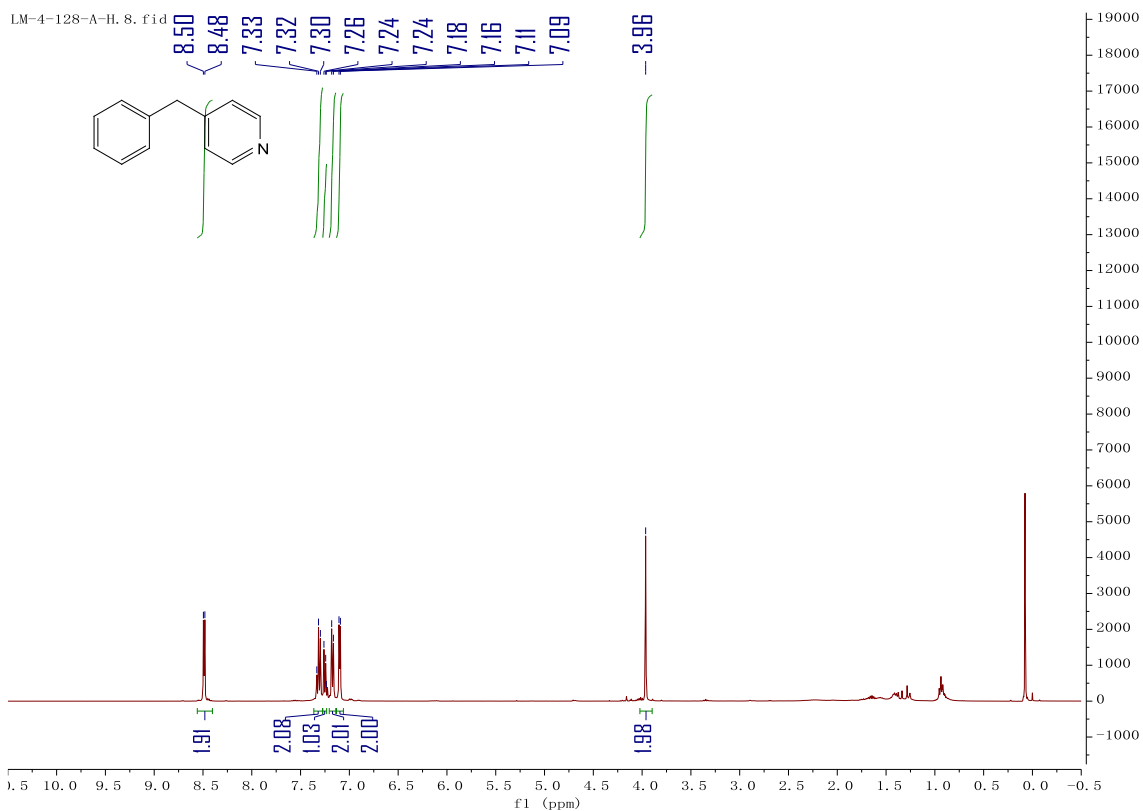
3aa

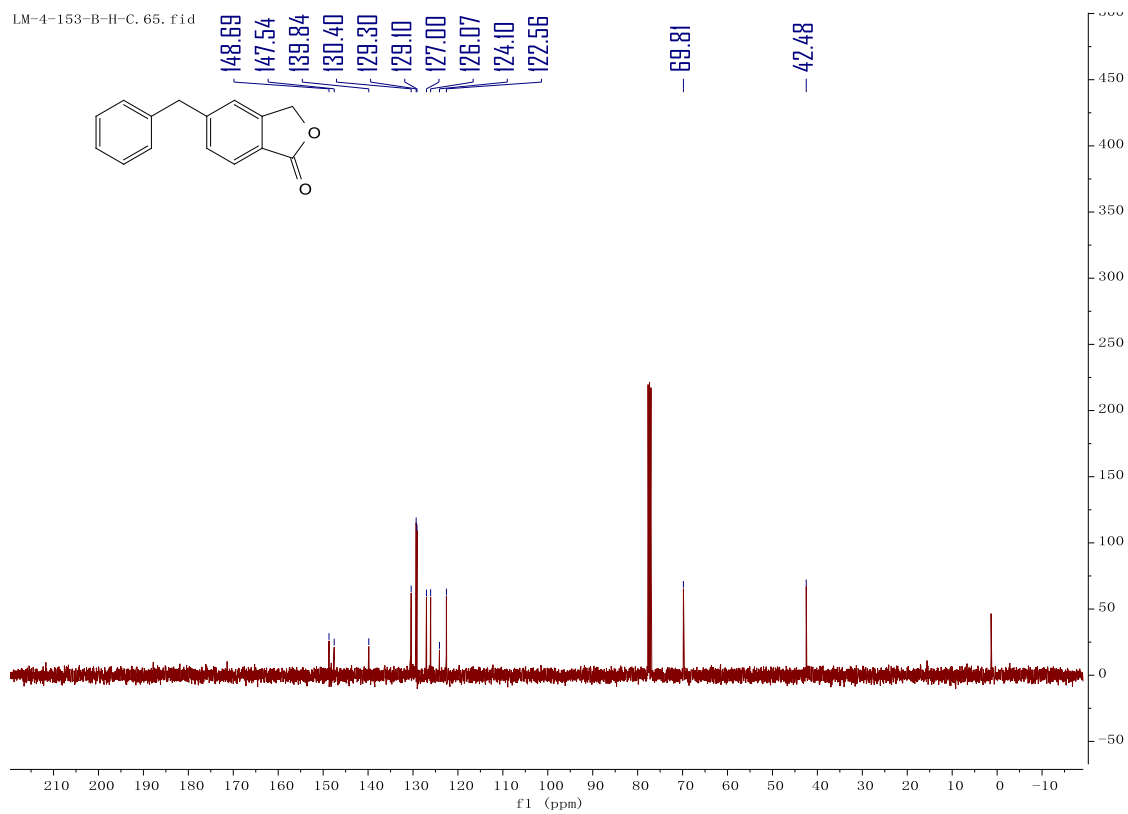
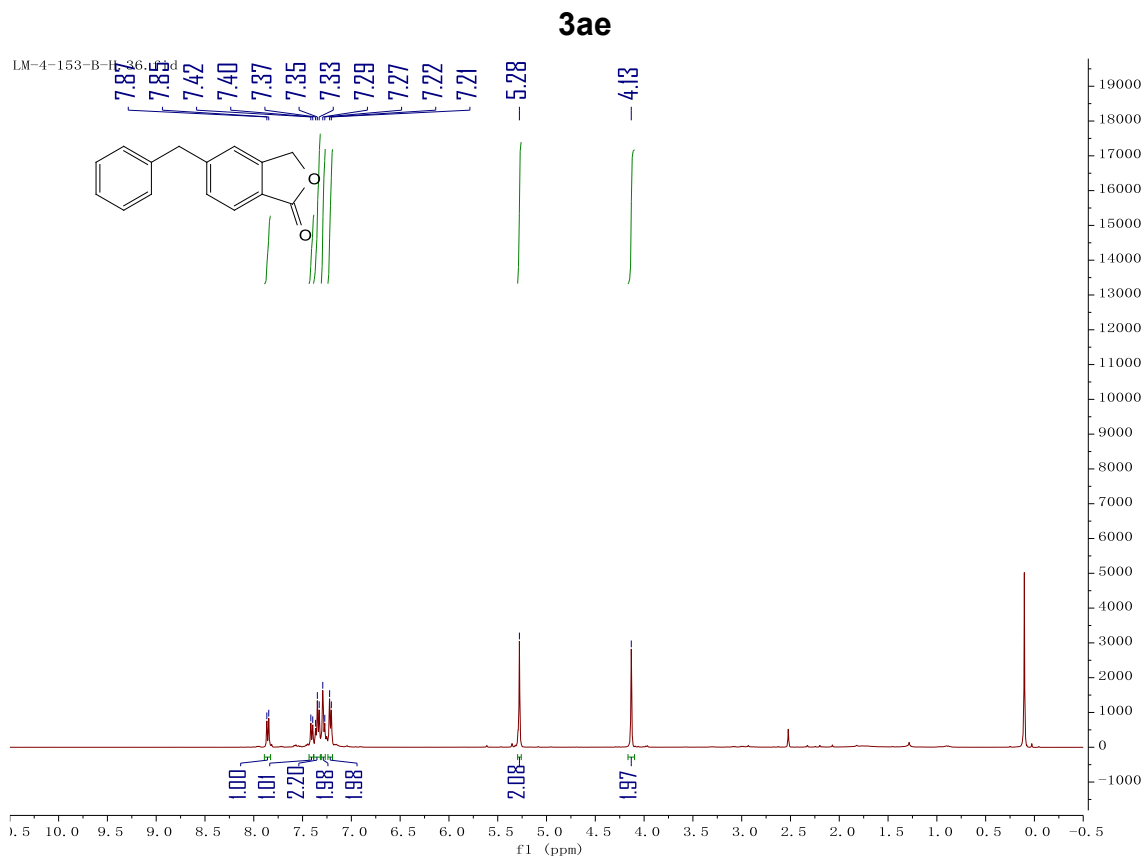


3ab: 3ac



3ad





3af

