

Electronic Supplementary Information for

Electrochemical deoxygenative reduction of ketones

Kunhui Sun, Zhimin Xu, Velayudham Ramadoss, Lifang Tian,* and Yahui Wang*

Technical Institute of Fluorochemistry (TIF), State Key Laboratory of Materials-Oriented Chemical Engineering (MCE), 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	1
2. Experimental procedures	1
2.1 Electrode materials and dimensions.....	1
2.2 General procedure for the preparation of ketones.....	2
3. General procedure for the electrochemical reduction of ketone	3
4. Procedure for gram-scale synthesis	3
5. Optimization of the reaction conditions.....	4
6. Procedure for cyclic voltammetry (CV)	5
7. Characterization data of the starting materials.....	6
8. Detail descriptions for products	8
9. Procedure for divided-cell electrolysis	20
10. Procedure for Deuterium experiment.....	20
11. ^1H , ^{13}C and ^{19}F NMR spectra	22

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 2,4-dinitrophenylhydrazine-sulfuric acid for visualization. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (bp. 60-90 °C). ^1H , ^{13}C and ^{19}F NMR data were recorded with Bruker (400 MHz) or Jeol (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. All chemical shifts were reported relative to tetramethylsilane (0 ppm for ^1H), CDCl_3 (77.0 ppm for ^{13}C) and DMSO-d_6 (2.5 ppm for ^1H , 40.0 ppm for ^{13}C).

2. Experimental procedures

2.1 Electrode materials and dimensions

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



IKA ElectraSyn 2.0

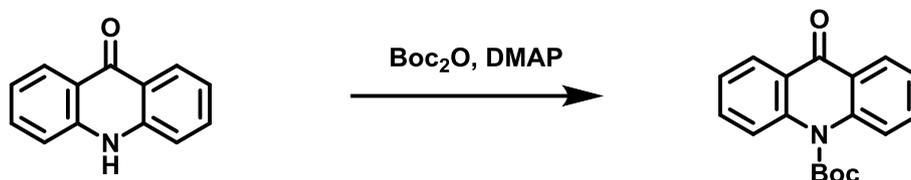


IKA Carousel complete

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.

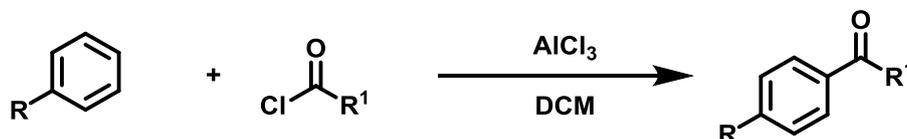
2.2 General procedure for the preparation of ketones

General procedure A¹:



To a solution of acridone (500 mg, 2.56 mmol, 1 equiv.) and Boc₂O (837 mg, 3.84 mmol, 1.5 equiv.) in MeCN (8 mL) at room temperature, DMAP (468 mg, 3.84 mmol, 1.5 equiv.) was added. The reaction mixture was stirred at rt for 8 h followed by 3 h at 60 °C. The solvent was removed under vacuum, the crude was dissolved in Et₂O. The resulting solution was washed with a diluted solution of HCl (0.1 M), brine and dried over MgSO₄. After evaporation of the solvent the crude product purified by column chromatography (pentane/Et₂O 2:1) to obtain the compound **1e** as a white solid.

General procedure B²:

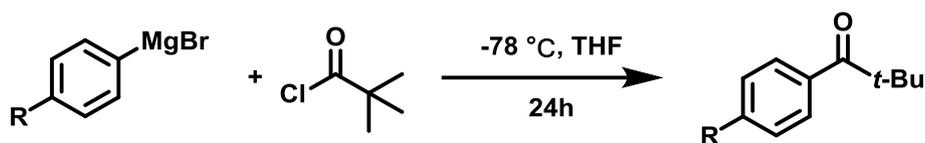


Acetyl chloride (0.46 mL) was added in a dropwise manner to a mixture of aluminium chloride (0.86 g, 21 mmol) in anhydrous DCM (100 mL). The slurry was left to stir for 30 min after which, biphenyl (0.50 g, 10.6 mmol) was added. The reaction mixture was then left to stir for 14 h. The reaction was quenched using a mixture of ice and water (150 mL) and the organic layer washed with aqueous hydrochloric acid solution (HCl) (0.5 M, 2 × 50 mL), water (100 mL), saturated sodium bicarbonate solution (NaHCO₃) (50 mL) and water (2 × 100 mL). The combined organic layer was dried over anhydrous magnesium sulfate (MgSO₄) filtered and the solvent was removed under vacuum to give the substrates **1m**, **1w** and **1x**.

[1] T. Stopka, L. Marzo, M. Zurro, S. Janich, E. Würthwein, C. G. Daniliuc, J. Alemán, and O. G. Mancheño, *Angew. Chem. Int. Ed.*, 2015, **54**, 5049–5053.

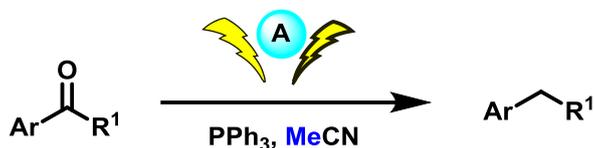
[2] R. K. Lota, S. Dhanani, C. P. Owen and S. Ahmed, *Letters in Drug Design & Discovery*, 2007, **4**, 180–184.

General procedure C³:



To a cooled (-78 °C) solution of pivaloyl chloride (3 mL) in THF (20 mL) was added a solution of freshly prepared arylmagnesium bromide (1.2 mmol in THF, 20 mL) under N₂ atmosphere. The solution was stirred for 24 h at the same temperature, then was allowed to warm to room temperature. The mixture was diluted with saturated aqueous NH₄Cl solution (30 mL) and extracted with DCM (30 mL × 3). The extract was filtered and concentrated under reduced pressure and the crude mixture was purified by silica gel column chromatography to obtain the substrate **1y**.

3. General procedure for the electrochemical reduction of ketone



The electrolysis was carried out in the electrolysis cell of IKA[®] ElectraSyn 2.0. The anodic electrode was the carbon electrode (3.0 cm × 0.8 cm × 0.2 cm) and the cathodic electrode was the nickel plate (3.0 cm × 0.8 cm × 0.2 cm). Aryl ketones (0.5 mmol), Et₄NBr (105.0 mg, 0.5 mmol), PPh₃ (655.0 mg, 2.5 mmol), MeCN (4 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). Then the reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 8 h. 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.

4. Procedure for gram-scale synthesis

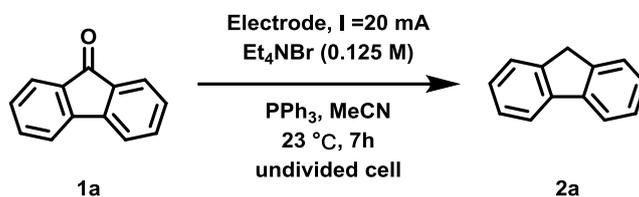
The electrolysis was carried out in the electrolysis cell of IKA[®] ElectraSyn 2.0. The anodic electrode was the carbon electrode (3.0 cm × 0.8 cm × 0.2 cm) and the cathodic electrode was the nickel plate (3.0 cm × 0.8 cm × 0.2 cm). According to General Procedure, starting 9H-fluoren-9-one (1.4 g, 8.0 mmol) was reacted with PPh₃ (10.5 g, 40 mmol) were added to an oven-dried undivided cell with MeCN (25 mL) equipped with a stirring bar. The reaction mixture was stirred and electrolyzed at a constant current of 40 mA at room

[3] J. Chen, Z. Zhang, B. Li, F. Li, Y. Wang, M. Zhao, I. D. Gridnev, T. Imamoto and W. Zhang, *Nat. Commun.*, 2018, **9**, 5000–5009.

temperature for 48 h. Solvent was evaporated under vacuum and the crude material was purified by column chromatography (petroleum ether) to afford the title compound as a white solid in 60% yield (0.8 g).

5. Optimization of the reaction conditions

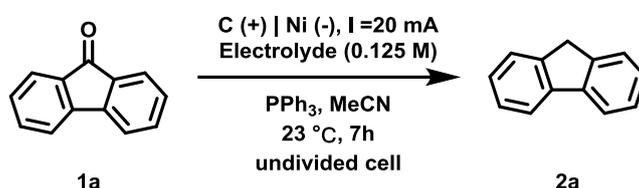
Table S1. Optimization of the electrode material



Entry	Electrode	Yield ^a
1	C (+) Ni (-)	72%
2	C (+) Pt (-)	<5%
3	C (+) C (-)	50%

^a Isolated yields after chromatography were reported.

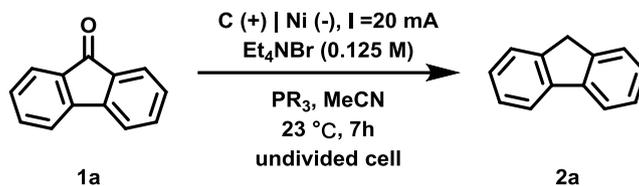
Table S2. Optimization of the electrolyte.



Entry	Electrolyte	Yield ^a
1	ⁿ Bu ₄ NBr	45%
2	Et ₄ NI	35%
3	Et ₄ NPF ₆	54%
4	Et ₄ NClO ₄	36%

^a Isolated yields after chromatography were reported.

Table S3. Optimization of the PR₃.



Entry	PR ₃	Yield ^a
1	<i>n</i> Bu ₃ P	45%
2	Cy ₃ P	14%
3	[N(Me) ₂] ₃ P	29%
4	(PhO) ₃ P	54%

^a Isolated yields after chromatography were reported.

6. Procedure for cyclic voltammetry (CV)

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 acetonitrile. 8 mL acetonitrile solvent containing (0.025 M) Et₄NBr was used as the blank. The spectrums were recorded with the scan rate of 100 mV s⁻¹ (starting from 0 V). The CV of Background, PPh₃ and Benzophenone were conducted respectively.

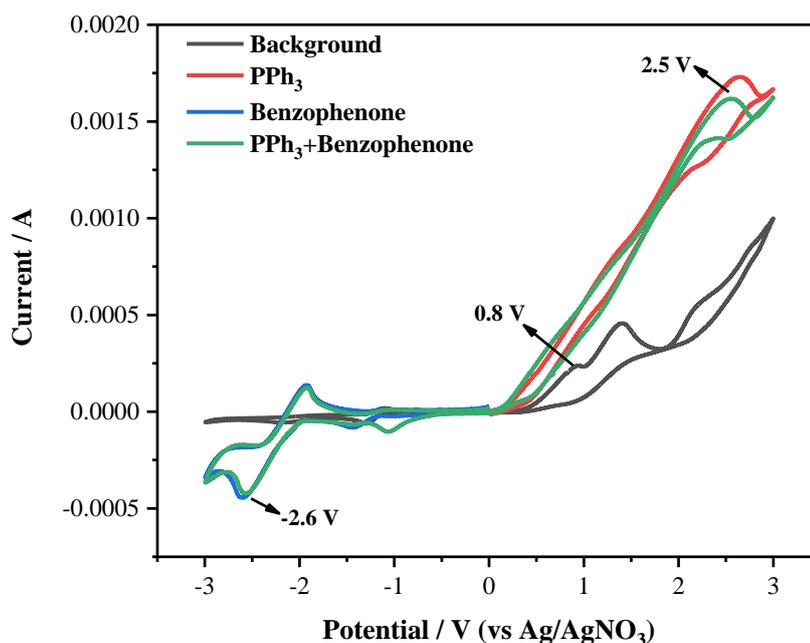
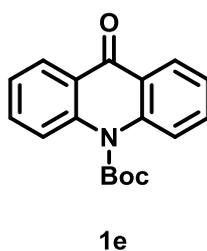


Fig 1. Cyclic voltammograms recorded in (0.025 M) Et₄NBr-MeCN solution: scan rate: 100 mV s⁻¹; starting potential: 0 V; glass carbon (3 mm diameter, Working Electrode); platinum plate (Counter Electrode); Ag/AgNO₃ (0.01 M) AgNO₃ in MeCN (Reference Electrode); Concentrations: PPh₃ (0.2 mmol / 8 ml MeCN), Benzophenone (0.2 mmol / 8 ml MeCN), PPh₃ + Benzophenone (0.2 mmol PPh₃ + 0.2 mmol Benzophenone / 8 ml MeCN).

7. Characterization data of the starting materials

tert-butyl 9-oxoacridine-10(9H)-carboxylate (1e)

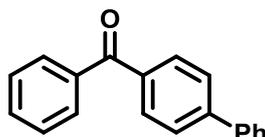


Prepared according to general procedure A from acridin-9(10H)-one on a 2.6 mmol scale. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 75% yield (566.3 mg).

R_f (petroleum ether/ethyl acetate = 20:1): 0.3; ¹H NMR (400 MHz, Chloroform-d) δ 8.45 – 8.43 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.69 – 7.65 (ddd, *J* = 8.6, 7.0, 1.7 Hz, 2H), 7.57 (dd, *J* = 8.6, 1.0 Hz, 2H), 7.39 – 7.29 (m, 2H),

1.69 (s, 9H). ^{13}C NMR (100 MHz, Chloroform-d) δ 178.84, 152.09, 139.70, 133.66, 127.49, 123.48, 123.07, 117.76, 86.61, 28.01. Spectroscopic data were in good agreement with literature⁴

[1,1'-biphenyl]-4-yl(phenyl)methanone (1m)

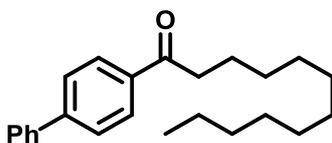


1m

Prepared according to general procedure B from 1,1'-biphenyl (2 mmol) and benzoyl chloride (2.2 mmol). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 50% yield (258.8 mg).

Rf (petroleum ether/ethyl acetate = 20:1): 0.3; ^1H NMR (400 MHz, Chloroform-d) δ 7.95 – 7.88 (m, 2H), 7.89 – 7.81 (m, 2H), 7.76 – 7.69 (m, 2H), 7.69 – 7.64 (m, 2H), 7.64 – 7.58 (m, 1H), 7.55 – 7.47 (m, 4H), 7.45 – 7.38 (m, 1H). ^{13}C NMR (100 MHz, Chloroform-d) δ 196.63, 145.50, 140.23, 138.02, 136.50, 132.67, 131.02, 130.29, 129.26, 128.60, 128.48, 127.58, 127.25. Spectroscopic data were in good agreement with literature⁵

1-([1,1'-biphenyl]-4-yl)dodecan-1-one (1w)



1w

Prepared according to general procedure B from 1,1'-biphenyl (2 mmol) and dodecanoyl chloride (2.2 mmol). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 90% yield (601.9 mg).

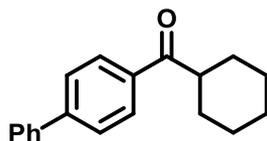
Rf (petroleum ether/ethyl acetate = 20:1): 0.3; ^1H NMR (400 MHz, Chloroform-d) δ 8.04 (d, J = 8.5 Hz, 2H), 7.68 (d, J = 8.5 Hz, 2H), 7.64 – 7.61 (m, 2H), 7.50 – 7.44 (m, 2H), 7.43 – 7.37 (m, 1H), 2.99 (t, J = 7.4, 2H), 1.87 – 1.70 (m, 2H), 1.26 (s, 16H), 0.93 – 0.82 (m, 3H). ^{13}C NMR (100 MHz, Chloroform-d) δ 200.60, 145.86, 140.27, 136.11, 129.27, 129.00, 128.50, 127.58, 127.53, 39.04, 32.26, 29.98, 29.85, 29.76, 29.69, 29.58, 24.83, 23.03, 14.47. Spectroscopic data were in good agreement with literature⁶

[4] A. A. Kulago, E. M. Mes, M. Klok, A. Meetsma, A. M. Brouwer and B. L. Feringa, *J. Org. Chem.*, 2010, **75**, 666–679.

[5] I. Sapountzis, W. Lin, C. C. Kofink, C. Despotopolou and P. Knochel, *Angew. Chem. Int. Ed.*, 2005, **44**, 1654–1657.

[6] J. Boivin and V. T. Nguyen, *Beilstein Journal of Organic Chemistry* 2007, **3**, No. 45. DOI:10.1186/1860-5397-3-45.

[1,1'-biphenyl]-4-yl(cyclohexyl)methanone (**1x**)

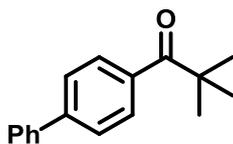


1x

Prepared according to general procedure B from 1,1'-biphenyl (2 mmol) and cyclohexanecarbonyl chloride (2.2 mmol). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 86% (456.2 mg).

Rf (petroleum ether/ethyl acetate = 20:1): 0.3; **¹H NMR** (400 MHz, Chloroform-d) δ 8.07 – 8.00 (m, 2H), 7.71 – 7.66 (m, 2H), 7.65 – 7.61 (m, 2H), 7.52 – 7.44 (m, 2H), 7.43 – 7.35 (m, 1H), 3.30 (tt, $J = 11.4, 3.3$ Hz, 1H), 1.97 – 1.83 (m, 4H), 1.66 – 1.22 (m, 6H). **¹³C NMR** (100 MHz, Chloroform-d) δ 203.72, 145.69, 140.25, 135.28, 129.23, 129.16, 128.44, 127.54, 127.53, 45.97, 29.77, 26.29, 26.19. **HRMS** m/z (ESI) calcd for C₁₉H₂₁O ([M+H]⁺) 265.1592, found 265.1592.

1-([1,1'-biphenyl]-4-yl)-2,2-dimethylpropan-1-one (**1y**)



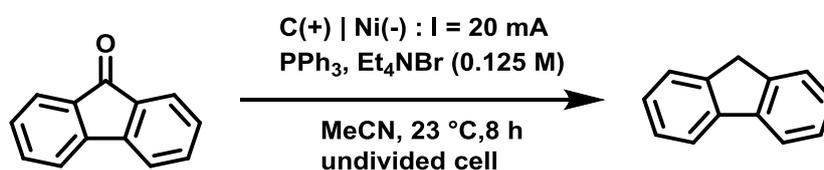
1y

Prepared according to general procedure C from [1,1'-biphenyl]-4-ylmagnesium bromide (3 mmol) and pivaloyl chloride (2 mmol). The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 94% (446.9 mg).

Rf (petroleum ether/ethyl acetate = 20:1): 0.3; **¹H NMR** (400 MHz, Chloroform-d) δ 7.88 – 7.81 (m, 2H), 7.66 – 7.60 (m, 4H), 7.47 (t, $J = 7.5$ Hz, 2H), 7.42 – 7.37 (m, 1H), 1.41 (s, 9H). **¹³C NMR** (100 MHz, Chloroform-d) δ 208.65, 144.01, 140.36, 137.20, 129.21, 129.05, 128.26, 127.48, 127.01, 44.51, 28.44. **HRMS** m/z (ESI) calcd for C₁₇H₁₉O ([M+H]⁺) 239.1436, found 239.1436.

8. Detail descriptions for products

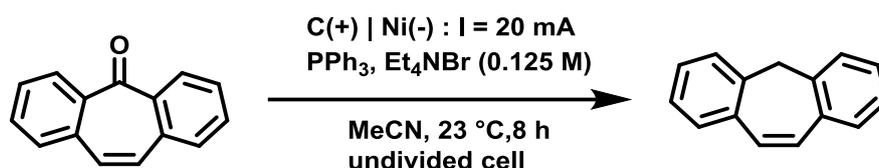
9H-fluorene (**2a**)



The starting 9H-fluoren-9-one (90.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a white solid in 72% yield (59.8 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.84 (d, *J* = 7.5 Hz, 2H), 7.59 (d, *J* = 7.4 Hz, 2H), 7.43 (t, *J* = 7.4 Hz, 2H), 7.36 (td, *J* = 7.4, 1.2 Hz, 2H), 3.94 (s, 2H); ¹³C NMR (400 MHz, Chloroform-d) δ 143.52, 142.01, 127.03, 127.00, 125.33, 120.18, 37.24. Spectroscopic data were in good agreement with literature⁷

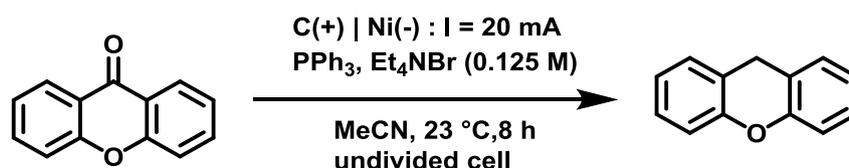
5H-dibenzo[a,d][7]annulene (2b)



The starting 5H-dibenzo[a,d][7]annulen-5-one (103.1 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a white solid in 82% yield (79.0 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.42 – 7.35 (m, 6H), 7.32 – 7.27 (m, 2H), 7.11 (s, 2H), 3.83 (s, 2H); ¹³C NMR (400 MHz, Chloroform-d) δ 138.42, 135.48, 131.83, 128.72, 128.41, 128.18, 126.37, 41.94. HRMS *m/z* (ESI) calcd for C₁₇H₁₃ ([M+H]⁺) 193.1017, found 193.1014.

9H-xanthene (2c)



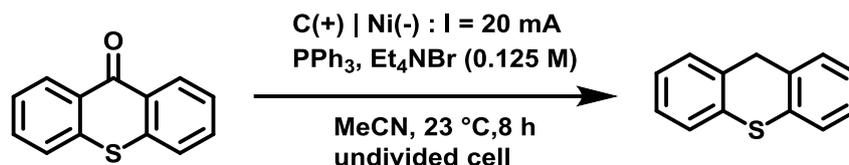
The starting 9H-xanthen-9-one (98.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a white solid in 61% yield (55.5 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.27 – 7.18 (m, 4H), 7.11 – 7.04 (m, 4H), 4.08 (s, 2H); ¹³C NMR (400 MHz, Chloroform-d) δ 152.26, 129.23, 127.95, 123.26, 120.88, 116.77, 28.19. Spectroscopic data were in good agreement with literature⁸

[7] D. L. J. Clive and R. Sunasee, *Org. Chem.*, 2007, **9**, 2677–2688.

[8] E. Böß, T. Hillringhaus, J. Nitsch and M. Klussmann, *Org. Biomol. Chem.*, 2011, **9**, 1744–1748.

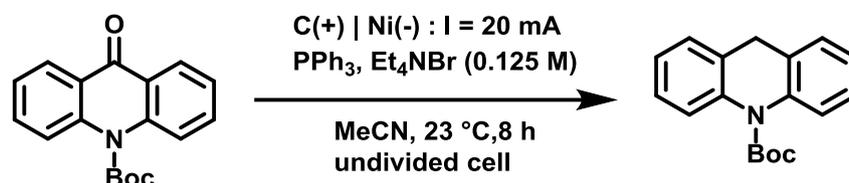
9H-thioxanthene (2d)



The starting 9H-thioxanthene-9-one (106.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a white solid in 65% yield (64.4 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.52 – 7.41 (m, 2H), 7.35 – 7.31 (m, 2H), 7.26 – 7.19 (m, 4H), 3.88 (s, 2H); ¹³C NMR (400 MHz, Chloroform-d) δ 136.51, 134.16, 128.25, 127.17, 126.92, 126.84, 39.53. Spectroscopic data were in good agreement with literature⁹

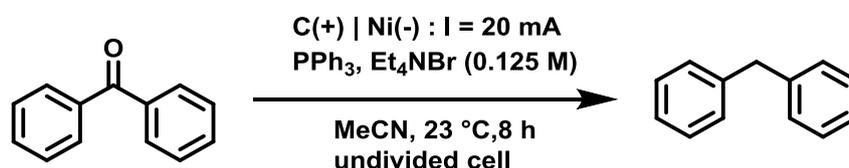
tert-butyl acridine-10(9H)-carboxylate (2e)



The starting tert-butyl 9-oxoacridine-10-carboxylate (147.6 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 49% yield (137.2 mg).

R_f (petroleum ether/ethyl acetate = 50:1): 0.3; ¹H NMR (400 MHz, Chloroform-d) δ 7.54 (d, *J* = 8.0 Hz, 2H), 7.14 – 7.08 (m, 4H), 7.01 (t, *J* = 7.4 Hz, 2H), 3.69 (s, 2H), 1.44 (s, 9H); ¹³C NMR (400 MHz, Chloroform-d) δ 152.79, 139.07, 133.30, 127.26, 126.17, 125.35, 125.25, 82.07, 34.13, 28.53. Spectroscopic data were in good agreement with literature¹⁰

diphenylmethane (2f)



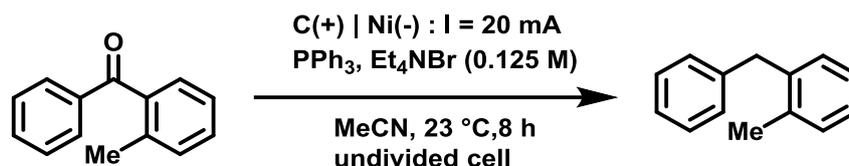
[9] X.-Q Zhu, Z. Dai, A. Yu, S. Wu and J.-P. Cheng, *J. Phys. Chem. B*, 2008, **112**, 11694–11707.

[10] T. Stopka, L. Marzo, M. Zurro, S. Janich, E.-U. Würthwein, C. G. Daniliuc, J. Alemán and O. G. Mancheño, *Angew. Chem. Int. Ed.*, 2015, **54**, 5049–5053.

The starting benzophenone (91.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a colorless oil in 92% yield (77.3 mg).

R_f (petroleum ether): 0.6; ¹H NMR (400 MHz, Chloroform-d) δ 7.34 – 7.28 (m, 4H), 7.25 – 7.19 (m, 6H), 4.01 (s, 2H); ¹³C NMR (400 MHz, Chloroform-d) δ 141.44, 129.27, 128.79, 126.39, 42.28. Spectroscopic data were in good agreement with literature¹¹

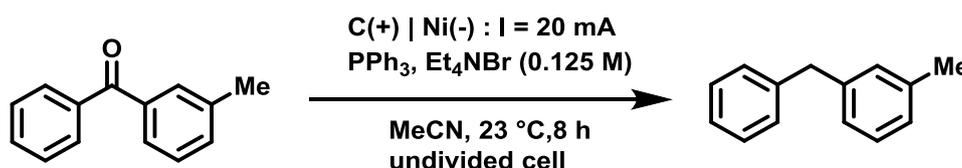
1-benzyl-2-methylbenzene (2g)



The starting phenyl(o-tolyl)methanone (98.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a colorless oil in 36% yield (32.8 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.35 – 7.29(m, 2H), 7.27 – 7.12 (m, 7H), 4.04 (s, 2H), 2.29 (s, 3H).; ¹³C NMR (400 MHz, Chloroform-d) δ 140.70, 139.24, 136.96, 130.60, 130.26, 129.06, 128.71, 126.77, 126.31, 126.24, 39.78, 20.02. Spectroscopic data were in good agreement with literature¹²

1-benzyl-3-methylbenzene (2h)



The starting phenyl(m-tolyl)methanone (98.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a colorless oil in 53% yield (48.2 mg).

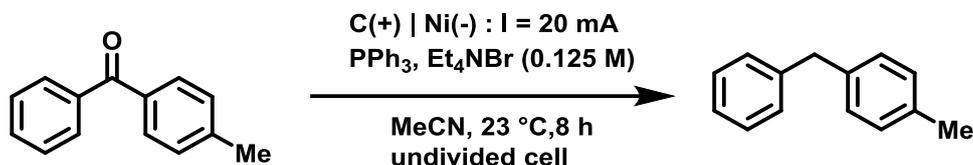
R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.41 – 7.36 (m, 2H), 7.32 – 7.28 (m, 4H), 7.17 – 7.05 (m, 3H), 4.05 (s, 2H), 2.42 (s, 3H); ¹³C NMR (400 MHz, Chloroform-d) δ 141.56, 141.34, 138.33,

[11] M. Peña-López, M. Ayán-Varela, L. A. Sarandeses and J. P. Sestelo, *Chem. Eur. J.*, 2010, **16**, 9905–9909.

[12] C.-R. Chen, S. Zhou, D. B. Biradar and H.-M. Gau, *Adv. Synth. Catal.*, 2010, **352**, 1718–1727.

130.03, 129.23, 128.75, 128.66, 127.14, 126.33, 126.30, 42.21, 21.74. Spectroscopic data were in good agreement with literature¹²

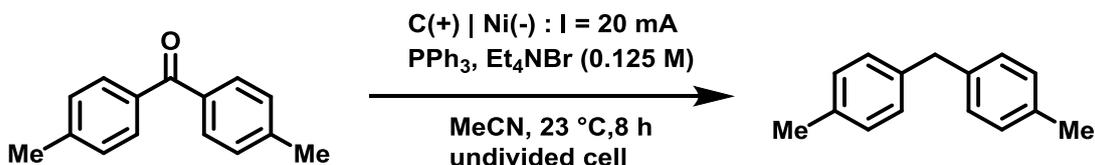
1-benzyl-4-methylbenzene (2i)



The starting phenyl(p-tolyl)methanone (98.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a colorless oil in 50% yield (45.5 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.43 – 7.34 (m, 2H), 7.31 – 7.27 (m, 3H), 7.22 – 7.17 (m, 4H), 4.05 (s, 2H), 2.42 (s, 3H); ¹³C NMR (400 MHz, Chloroform-d) δ 141.72, 138.39, 135.84, 129.47, 129.19, 129.13, 128.75, 126.29, 41.83, 21.35. Spectroscopic data were in good agreement with literature¹³

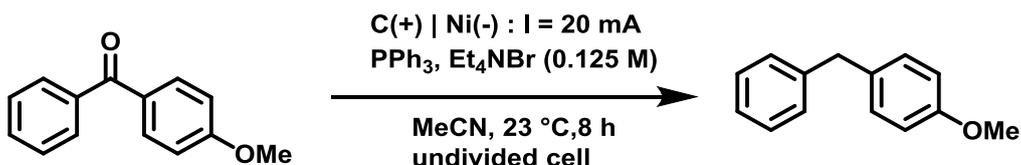
di-p-tolylmethane (2j)



The starting di-p-tolylmethanone (105.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a colorless oil in 61% yield (59.8 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.29 – 7.23 (m, 8H), 4.08 (s, 2H), 2.49 (s, 6H); ¹³C NMR (400 MHz, Chloroform-d) δ 138.69, 135.73, 129.43, 129.07, 41.41, 21.33. Spectroscopic data were in good agreement with literature¹⁴

1-benzyl-4-methoxybenzene (2k)

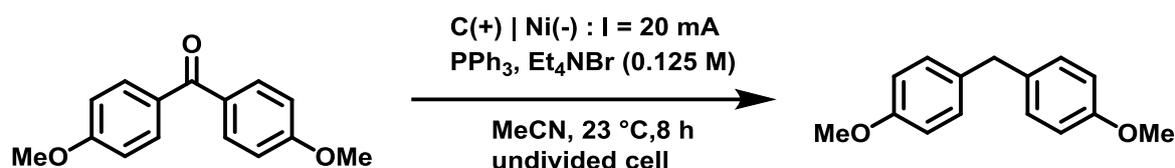


[13] G. Sun and Z. Wang, *Tetrahedron Letters*, 2008, **49**, 4929–4932.

[14] R. B. Bedford, M. Huwe and M. C. Wilkinson, *Chem. Commun.*, 2009, 600–602.

The starting (4-methoxyphenyl)(phenyl)methanone (106.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 50:1) to afford the title compound as a colorless oil in 70% yield (69.3 mg). R_f (petroleum ether/ethyl acetate = 100:1): 0.3; ¹H NMR (400 MHz, Chloroform-d) δ 7.36 (t, *J* = 7.5 Hz, 2H), 7.27 (t, *J* = 7.3 Hz, 3H), 7.19 (d, *J* = 8.5 Hz, 2H), 6.92 (d, *J* = 8.5 Hz, 2H), 4.01 (s, 2H), 3.85 (s, 3H); ¹³C NMR (400 MHz, Chloroform-d) δ 158.23, 141.88, 133.53, 130.16, 129.11, 128.73, 126.28, 114.15, 55.53, 41.33. Spectroscopic data were in good agreement with literature¹³

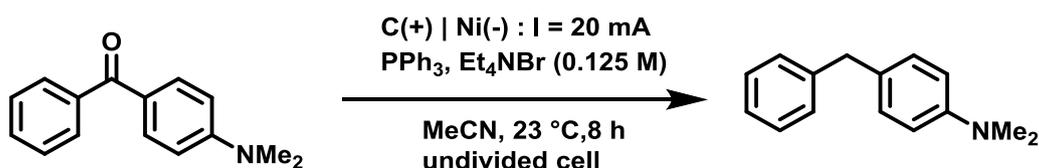
bis(4-methoxyphenyl)methane (2l)



The starting bis(4-methoxyphenyl)methanone (121.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a white solid in 56% yield (63.9 mg).

R_f (petroleum ether/ethyl acetate = 100:1): 0.3; ¹H NMR (400 MHz, Chloroform-d) δ 7.14 (d, *J* = 8.5 Hz, 4H), 6.88 (d, *J* = 8.5 Hz, 4H), 3.92 (s, 2H), 3.82 (s, 6H); ¹³C NMR (400 MHz, Chloroform-d) δ 158.15, 133.99, 130.01, 114.10, 55.51, 40.40. Spectroscopic data were in good agreement with literature¹³

4-benzyl-N,N-dimethylaniline (2m)

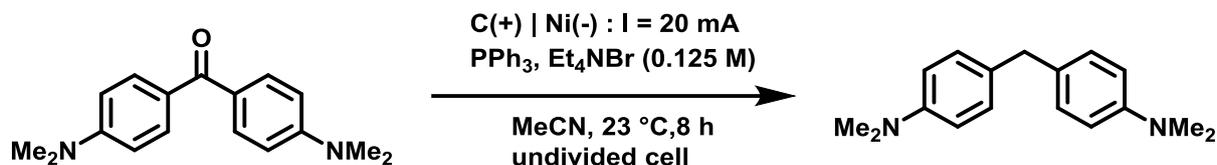


The starting (4-(dimethylamino)phenyl)(phenyl)methanone (112.5 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 20:1) to afford the title compound as a white solid in 76% yield (80.2 mg).

R_f (petroleum ether/ethyl acetate = 50:1): 0.2; ¹H NMR (400 MHz, Chloroform-d) δ 7.29 – 7.22 (m, 2H), 7.20 – 7.13 (m, 2H), 7.05 (d, *J* = 8.6 Hz, 2H), 6.68 (d, *J* = 8.6 Hz, 2H), 3.88 (s, 2H), 2.89 (s, 6H); ¹³C NMR (400

MHz, Chloroform-d) δ 149.44, 142.38, 129.85, 129.56, 129.11, 128.65, 126.09, 113.26, 41.23. Spectroscopic data were in good agreement with literature¹⁵

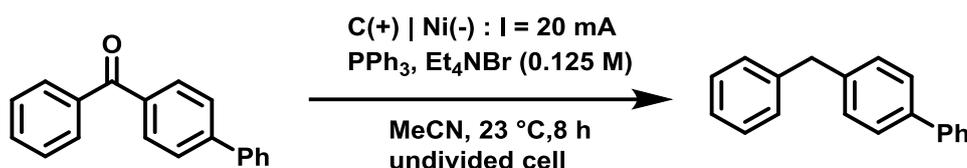
4,4'-methylenebis(N,N-dimethylaniline) (2n)



The starting bis(4-(dimethylamino)phenyl)methanone (134.1 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 10:1) to afford the title compound as a white solid in 65% yield (82.6 mg).

R_f (petroleum ether/ethyl acetate = 10:1): 0.3; ¹H NMR (400 MHz, Chloroform-d) δ 7.13 (d, *J* = 8.4 Hz, 4H), 6.75 (d, *J* = 8.5 Hz, 4H), 3.88 (s, 2H), 2.96 (s, 12H); ¹³C NMR (400 MHz, Chloroform-d) δ 149.29, 130.58, 129.68, 113.30, 41.23, 40.15. Spectroscopic data were in good agreement with literature¹⁶

4-benzyl-1,1'-biphenyl (2o)



The starting [1,1'-biphenyl]-4-yl(phenyl)methanone (129.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a white solid in 70% yield (84.5 mg).

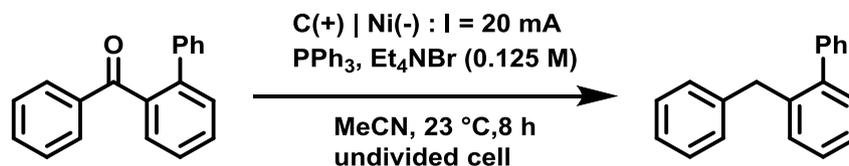
R_f (petroleum ether): 0.3; ¹H NMR (400 MHz, Chloroform-d) δ 7.71 – 7.67 (m, 2H), 7.65 – 7.60 (m, 2H), 7.55 – 7.50 (m, 2H), 7.46 – 7.39 (m, 3H), 7.39 – 7.28 (m, 5H), 4.13 (s, 2H); ¹³C NMR (400 MHz, Chloroform-d) δ 141.31, 141.28, 140.55, 139.32, 129.64, 129.28, 129.04, 128.83, 127.52, 127.39, 127.31, 126.46, 41.89. Spectroscopic data were in good agreement with literature¹⁷

2-benzyl-1,1'-biphenyl (2p)

[15] M. Amatore and C. Gosmini, *Chem. Commun.*, 2008, 5019–5021.

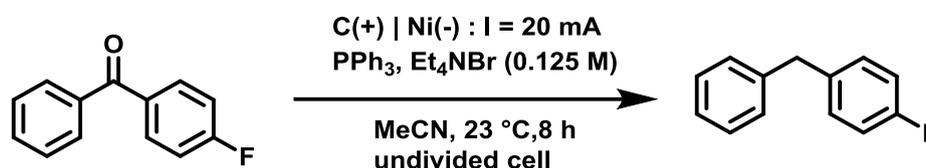
[16] Y.-Y. Lai, N.-T. Lin, Y.-H. Liu, Y. Wang and T.-Y. Luh, *Tetrahedron*, 2007, **63**, 6051–6055

[17] N. Sakai, K. Kawana, R. Ikeda, Y. Nakaike and T. Konakahara, *Eur. J. Org. Chem.*, 2011, 3178–3183.



The starting [1,1'-biphenyl]-2-yl(phenyl)methanone (129.0 mg, 0.5 mmol) was reacted with PPh_3 (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a white solid in 70% yield (84.5 mg). R_f (petroleum ether): 0.3; 1H NMR (400 MHz, Chloroform- d) δ 7.49 – 7.40 (m, 3H), 7.41 – 7.34 (m, 5H), 7.34 – 7.27 (m, 3H), 7.26 – 7.21 (m, 1H), 7.09 (d, $J = 7.5$ Hz, 2H), 4.07 (s, 2H); ^{13}C NMR (400 MHz, Chloroform- d) δ 142.57, 141.96, 141.76, 138.54, 130.63, 130.44, 129.61, 129.17, 128.55, 128.35, 127.78, 127.21, 126.49, 126.09, 39.36. Spectroscopic data were in good agreement with literature¹⁸

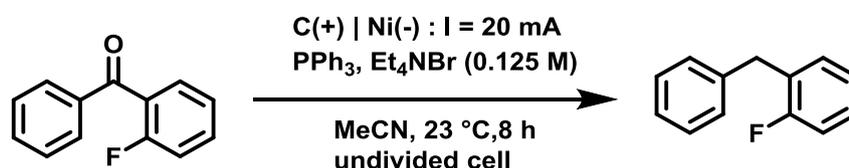
1-benzyl-4-fluorobenzene (2q)



The starting (4-fluorophenyl)(phenyl)methanone (100.0 mg, 0.5 mmol) was reacted with PPh_3 (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a colorless oil in 71% yield (66.2 mg).

R_f (petroleum ether): 0.5; 1H NMR (400 MHz, Chloroform- d) δ 7.28 (t, $J = 7.5$ Hz, 2H), 7.22 – 7.09 (m, 5H), 6.95 (t, $J = 8.7$ Hz, 2H), 3.93 (s, 2H).; ^{13}C NMR (400 MHz, Chloroform- d) δ 161.72 (d, $J = 243.9$ Hz), 141.26, 137.08 (d, $J = 3.2$ Hz), 130.60 (d, $J = 7.8$ Hz), 129.15, 128.86, 126.53, 115.53 (d, $J = 21.2$ Hz), 41.39; ^{19}F NMR (376 MHz, Chloroform- d) δ -117.30. Spectroscopic data were in good agreement with literature¹⁹

1-benzyl-2-fluorobenzene (2r)



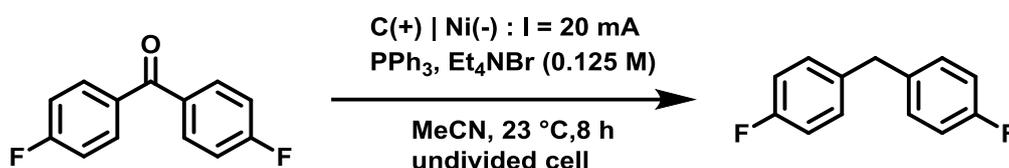
[18] R. Kuwano and M. Yokogi, *Org. Lett.*, 2005, **7**, 945–947.

[19] D. Srimani, A. Bej and A. Sarkar, *J. Org. Chem.*, 2010, **75**, 4296–4299.

The starting (2-fluorophenyl)(phenyl)methanone (100.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a colorless oil in 65% yield (60.5 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.38 – 7.33 (m, 2H), 7.31 – 7.16 (m, 5H), 7.16 – 7.02 (m, 2H), 4.07 (s, 2H); ¹³C NMR (400 MHz, Chloroform-d) δ 161.26 (d, *J* = 245.4 Hz), 140.17, 131.35 (d, *J* = 4.6 Hz), 128.97 (d, *J* = 29.3 Hz), 128.46, 128.26 (d, *J* = 8.0 Hz), 126.55, 124.38 (d, *J* = 3.6 Hz), 115.64 (d, *J* = 22.0 Hz), 35.13 (d, *J* = 3.0 Hz); ¹⁹F NMR (376 MHz, Chloroform-d) δ -117.80. Spectroscopic data were in good agreement with literature²⁰

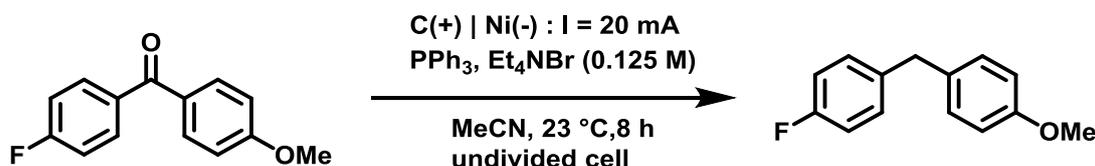
bis(4-fluorophenyl)methane (2s)



The starting bis(4-fluorophenyl)methanone (109.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a colorless oil in 87% yield (88.7 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.14 (dd, *J* = 8.3, 5.6 Hz, 4H), 7.04 – 6.97 (m, 4H), 3.94 (s, 2H); ¹³C NMR (400 MHz, Chloroform-d) δ 161.77 (d, *J* = 244.2 Hz), 136.92 (d, *J* = 3.0 Hz), 130.52 (d, *J* = 7.8 Hz), 115.62 (d, *J* = 21.2 Hz), 40.53; ¹⁹F NMR (376 MHz, Chloroform-d) δ -117.06. Spectroscopic data were in good agreement with literature²¹

1-fluoro-4-(4-methoxybenzyl)benzene (2t)



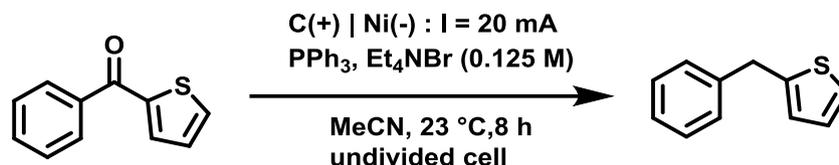
The starting (4-fluorophenyl)(4-methoxyphenyl)methanone (115.0 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 30:1) to afford the title compound as a colorless oil in 61% yield (66.1 mg).

[20] S. K. Yen, L. L. Koh, H. V. Huynh and T. S. A. Hor, *Eur. J. Inorg. Chem.*, 2009, 4288–4297.

[21] X. Qian and C. M. Kozak, *Synlett*, 2011, 6, 852–856.

Rf (petroleum ether/ethyl acetate = 50:1): 0.3; $^1\text{H NMR}$ (400 MHz, Chloroform-d) δ 7.21 – 7.11 (m, 4H), 7.06 – 6.97 (m, 2H), 6.93 – 6.86 (m, 2H), 3.94 (s, 2H), 3.82 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, Chloroform-d) δ 161.64 (d, $J = 243.7$ Hz), 158.33, 137.55 (d, $J = 3.2$ Hz), 133.34, 130.44 (d, $J = 7.8$ Hz), 130.07, 115.45 (d, $J = 21.1$ Hz), 114.22, 55.50, 40.46; $^{19}\text{F NMR}$ (376 MHz, Chloroform-d) δ -117.42. Spectroscopic data were in good agreement with literature²²

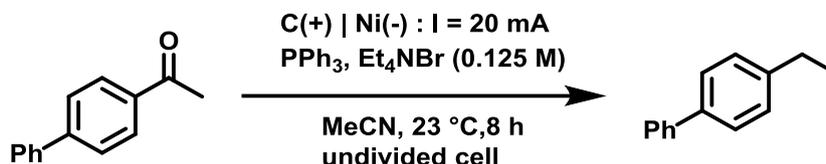
2-benzylthiophene (2u)



The starting phenyl(thiophen-2-yl)methanone (94.1 mg, 0.5 mmol) was reacted with PPh_3 (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a yellow oil in 26% yield (22.6 mg).

Rf (petroleum ether): 0.5; $^1\text{H NMR}$ (400 MHz, Chloroform-d) δ 7.40 – 7.24 (m, 5H), 7.19 (d, $J = 5.1$ Hz, 1H), 7.00 – 6.94 (m, 1H), 6.85 (d, $J = 3.3$ Hz, 1H), 4.21 (s, 2H); $^{13}\text{C NMR}$ (400 MHz, Chloroform-d) δ 144.35, 140.71, 128.91, 128.86, 127.14, 126.80, 125.47, 124.27, 36.35. Spectroscopic data were in good agreement with literature²³

4-ethyl-1,1'-biphenyl (2v)



The starting 1-([1,1'-biphenyl]-4-yl)ethan-1-one (98.0 mg, 0.5 mmol) was reacted with PPh_3 (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a white solid in 62% yield (56.5 mg).

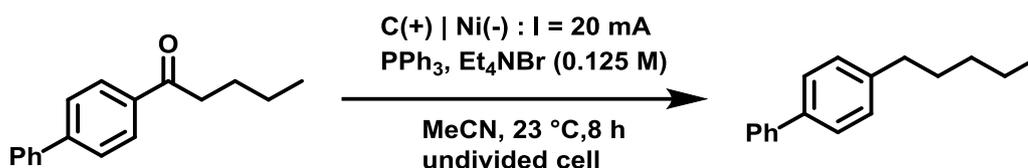
Rf (petroleum ether): 0.5; $^1\text{H NMR}$ (400 MHz, Chloroform-d) δ 7.68 (d, $J = 7.5$ Hz, 2H), 7.62 (d, $J = 8.1$ Hz, 2H), 7.51 (t, $J = 7.6$ Hz, 2H), 7.41 (t, $J = 7.4$ Hz, 1H), 7.37 (d, $J = 8.0$ Hz, 2H), 2.78 (q, $J = 7.6$ Hz, 2H), 1.37 (t, $J = 7.6$ Hz, 3H); $^{13}\text{C NMR}$ (400 MHz, Chloroform-d) δ 143.68, 141.48, 138.91, 129.03, 128.61, 127.40, 127.32, 127.28, 28.85, 15.96. Spectroscopic data were in good agreement with literature²⁴

[22] M.-B. Li, X.-L. Tang and S.-K. Tian, *Adv. Synth. Catal.*, 2011, **353**, 1980–1984.

[23] N. Henry, C. Enguehard-Gueiffier, I. Thery and A. Gueiffier, *Eur. J. Org. Chem.*, 2008, 4824–4827.

[24] B. Karimi, D. Elhamifar, J. H. Clark and A. J. Hunt, *Chem. Eur. J.*, 2010, **16**, 8047 – 8053.

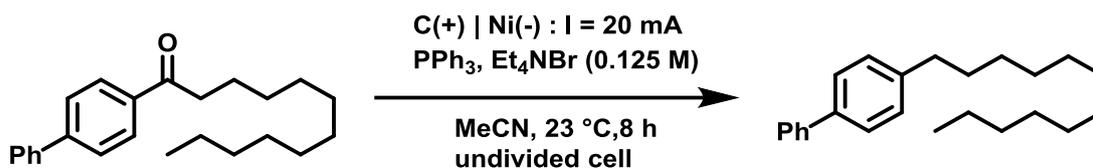
4-pentyl-1,1'-biphenyl (2w)



The starting 1-([1,1'-biphenyl]-4-yl)pentan-1-one (109.1 mg, 0.5 mmol) was reacted with PPh_3 (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 100:1) to afford the title compound as a white solid in 45% yield (50.4 mg).

Rf (petroleum ether): 0.5; $^1\text{H NMR}$ (400 MHz, Chloroform- d) δ 7.61 (d, $J = 8.3$, Hz, 2H), 7.53 (d, $J = 8.2$ Hz, 2H), 7.44 (t, $J = 7.6$ Hz, 2H), 7.36 – 7.32 (m, 1H), 7.28 (d, $J = 8.1$ Hz, 2H), 2.66 (t, $J = 7.8$, 2H), 1.72 – 1.63 (m, 2H), 1.41 – 1.34 (m, 4H), 0.92 (t, $J = 7.2$, 3H); $^{13}\text{C NMR}$ (400 MHz, Chloroform- d) δ 142.44, 141.51, 138.86, 129.15, 129.03, 127.32, 127.31, 127.28, 35.93, 31.92, 31.55, 22.92, 14.41. Spectroscopic data were in good agreement with literature²⁵

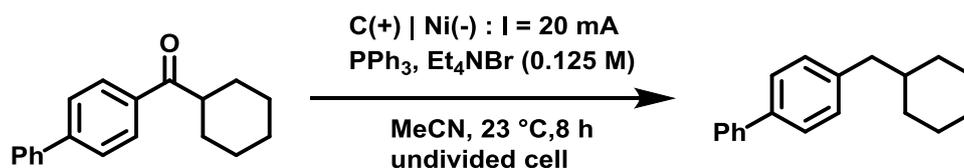
4-dodecyl-1,1'-biphenyl (2x)



The starting 1-([1,1'-biphenyl]-4-yl)dodecan-1-one (168.1 mg, 0.5 mmol) was reacted with PPh_3 (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a white solid in 50% yield (80.6 mg).

Rf (petroleum ether): 0.5; $^1\text{H NMR}$ (400 MHz, Chloroform- d) δ 7.64 – 7.59 (m, 2H), δ 7.54 (d, $J = 8.2$ Hz, 2H), 7.45 (t, $J = 7.6$ Hz, 2H), 7.37 – 7.32 (m, 1H), 7.28 (d, $J = 8.3$ Hz, 2H), 2.78 – 2.55 (m, 2H), 1.68 (p, $J = 7.6$ Hz, 2H), 1.39 – 1.24 (m, 18H), 0.94 – 0.89 (m, 3H); $^{13}\text{C NMR}$ (400 MHz, Chloroform- d) δ 142.45, 141.53, 138.86, 129.15, 129.02, 127.32, 127.31, 127.27, 35.98, 32.29, 31.88, 30.04, 30.02, 29.97, 29.90, 29.75, 29.73, 23.06, 14.49. **HRMS** m/z (ESI) calcd for $\text{C}_{24}\text{H}_{35}$ ($[\text{M}+\text{H}]^+$) 323.2739, found 323.2737.

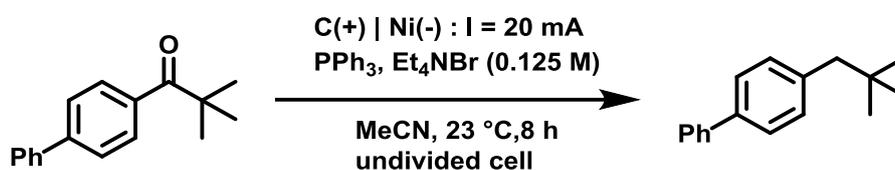
4-(cyclohexylmethyl)-1,1'-biphenyl (2y)



The starting [1,1'-biphenyl]-4-yl(cyclohexyl)methanone (132.1 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a white solid in 60% yield (60.0 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.64 – 7.59 (m, 2H), 7.55 – 7.51 (m, 2H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.38 – 7.31 (m, 1H), 7.24 (d, *J* = 8.2 Hz, 2H), 2.55 (d, *J* = 7.1 Hz, 2H), 1.79 – 1.65 (m, 5H), 1.61 – 1.53 (m, 1H), 1.30 – 1.18 (m, 3H), 1.06 – 0.95 (m, 2H); ¹³C NMR (400 MHz, Chloroform-d) δ 141.51, 140.86, 138.82, 129.92, 129.02, 127.31, 127.26, 127.10, 44.10, 40.13, 33.53, 26.92, 26.68. HRMS *m/z* (ESI) calcd for C₁₉H₂₃ ([M+H]⁺) 251.2800, found 251.1798.

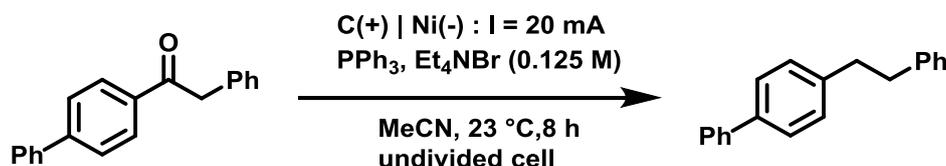
4-neopentyl-1,1'-biphenyl (2z)



The starting 1-([1,1'-biphenyl]-4-yl)-2,2-dimethylpropan-1-one (119.1 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a white solid in 50% yield (56.0 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.66 – 7.62 (m, 2H), 7.57 – 7.52 (m, 2H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.39 – 7.34 (m, 1H), 7.27 – 7.23 (m, 2H), 2.58 (s, 2H), 0.99 (s, 9H); ¹³C NMR (400 MHz, Chloroform-d) δ 141.47, 139.24, 138.91, 131.22, 129.03, 127.31, 126.66, 50.21, 32.19, 29.77. Spectroscopic data were in good agreement with literature²⁶

4-phenethyl-1,1'-biphenyl (2aa)



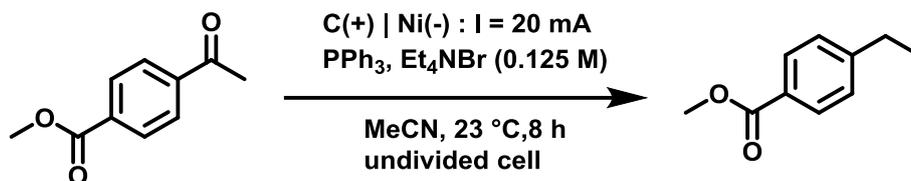
The starting 1-([1,1'-biphenyl]-4-yl)-2-phenylethan-1-one (136.1 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether) to afford the title compound as a white solid in 56% yield (72.3 mg).

R_f (petroleum ether): 0.5; ¹H NMR (400 MHz, Chloroform-d) δ 7.63 – 7.57 (m, 2H), 7.56 – 7.51 (m, 2H), 7.48 – 7.41 (m, 2H), 7.37 – 7.33 (m, 1H), 7.32 – 7.26 (m, 4H), 7.25 – 7.19 (m, 3H), 2.98 (s, 4H); ¹³C NMR

[26] C.-H. Cho, M. Sun, Y.-S. Seo, C.-B. Kim and K. Park, *J. Org. Chem.*, 2005, **70**, 1482-1485.

(400 MHz, Chloroform-d) δ 142.06, 141.38, 141.24, 139.19, 129.20, 129.06, 128.80, 128.70, 127.40, 127.37, 127.33, 126.29, 38.24, 37.91. **HRMS** m/z (ESI) calcd for $C_{20}H_{19}$ ($[M+H]^+$) 259.1487, found 259.1487.

methyl 4-ethylbenzoate (2ab)



The starting methyl 4-acetylbenzoate (89.1 mg, 0.5 mmol) was reacted with PPh₃ (655.0 mg, 2.5 mmol) according to general procedure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 70:1) to afford the title compound as a colorless oil in 53% yield (43.1 mg).

R_f (petroleum ether/ethyl acetate = 100:1): 0.3; **¹H NMR** (400 MHz, Chloroform-d) δ 7.96 (d, J = 8.3 Hz, 2H), 7.26 (d, J = 8.4 Hz, 2H), 3.90 (s, 3H), 2.70 (q, J = 7.6 Hz, 2H), 1.25 (t, J = 7.6 Hz, 3H); **¹³C NMR** (400 MHz, Chloroform-d) δ 167.52, 150.06, 130.02, 128.21, 52.29, 29.27, 15.56, 1.36. Spectroscopic data were in good agreement with literature²⁷

9. Procedure for divided-cell electrolysis

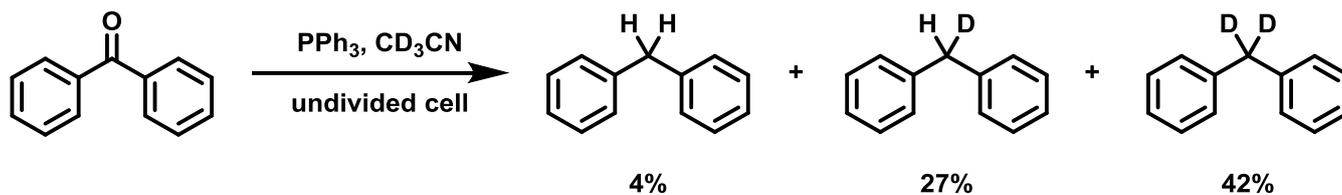
Ketone (2.0 mmol) and PPh₃ (10 mmol) were added into the anodic chamber, while the cathodic chamber was added with the same substances, ketone (2.0 mmol) and PPh₃ (10.0 mmol). Subsequently, MeCN (16 ml) containing Et₄NBr (2.0 mmol) was added to the anodic chamber and the cathodic chamber, respectively. Then the solution was stirred and electrolysed at a constant current of 20 mA at room temperature (23 °C) for 8 h. When the reaction was finished, the solution was treated by the standard procedure.

10. Procedure for Deuterium experiment

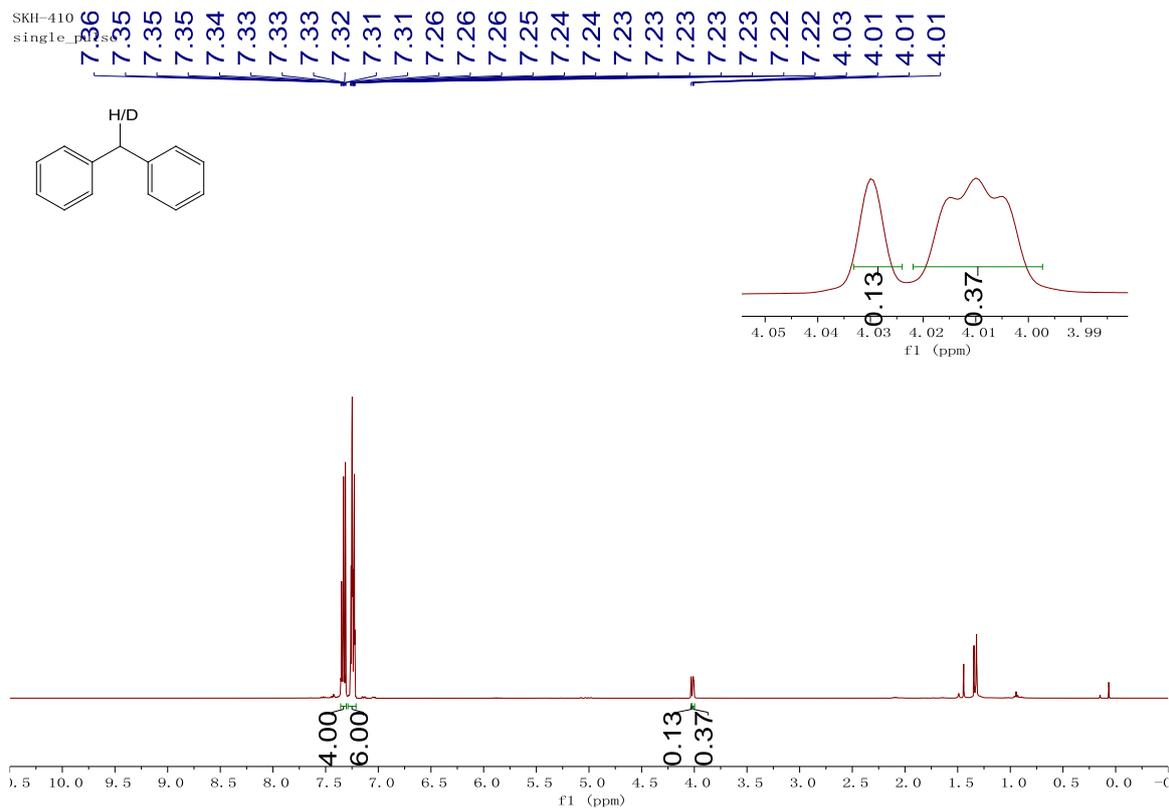
The electrolysis was carried out in the electrolysis cell of IKA® ElectraSyn 2.0. The anodic electrode was the carbon electrode (3.0 cm × 0.8 cm × 0.2 cm) and the cathodic electrode was the nickel plate (3.0 cm × 0.8 cm × 0.2 cm). Benzophenone (0.5 mmol), Et₄NBr (105.0 mg, 0.5 mmol), PPh₃ (655.0 mg, 2.5 mmol), CD₃CN (4 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). Then the reaction mixture was stirred and electrolyzed at a constant current of 20 mA at room temperature for 8 h. When the reaction was finished, the solution was treated by the standard

[27] I. Kondolff, H. Doucet and M. Santelli, *Organometallics*, 2006, **25**, 5219-5222.

procedure. It got the deuterated product with 73% yield, where H,H : H,D : D,D = 4% : 27% : 42%. $^1\text{H NMR}$ (400 MHz, Chloroform-d) δ 7.36 – 7.30 (m, 4H), 7.29 – 7.21 (m, 6H), 4.04– 4.00 (m, 0.5H).

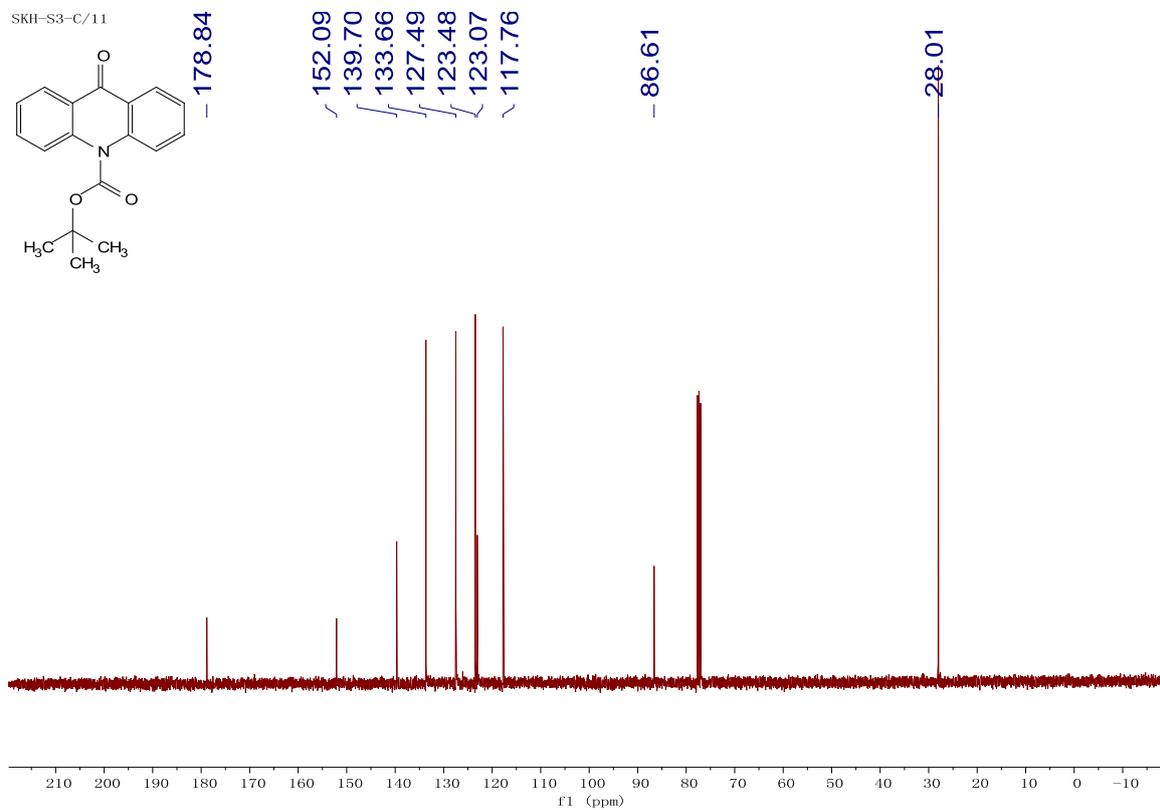
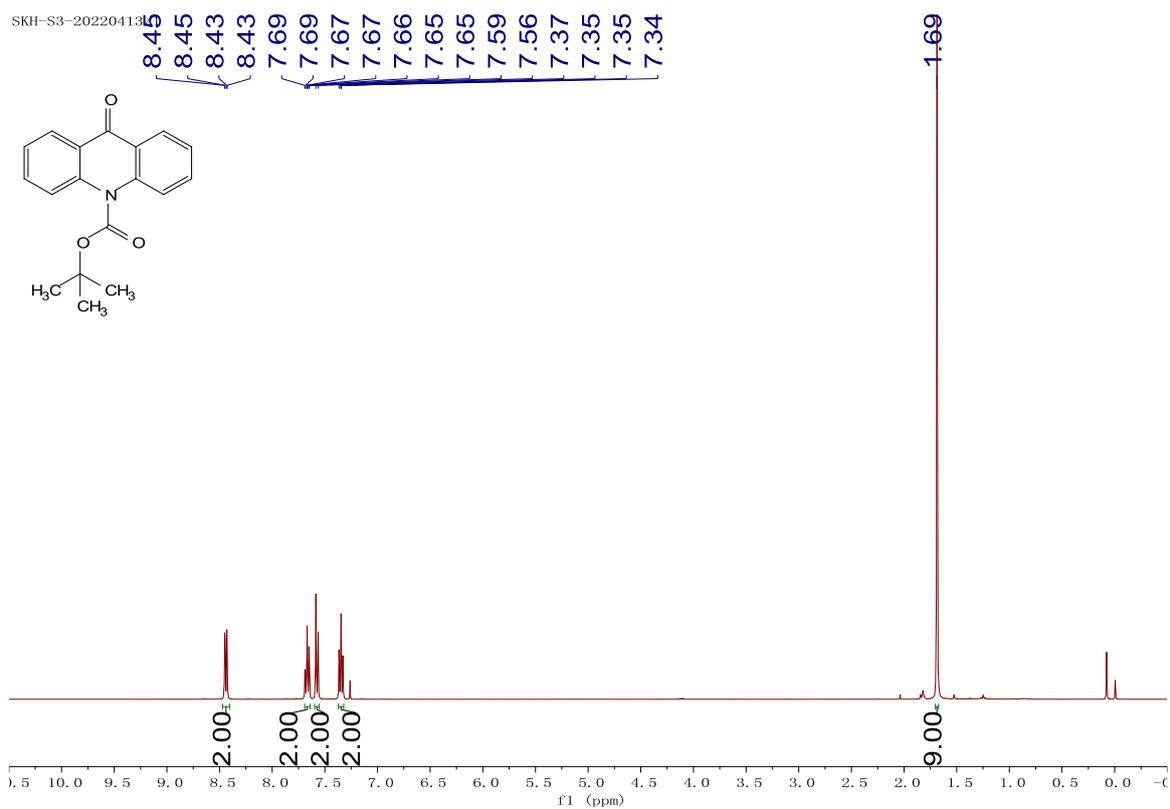


$^1\text{H NMR}$ (400 MHz, CDCl_3)

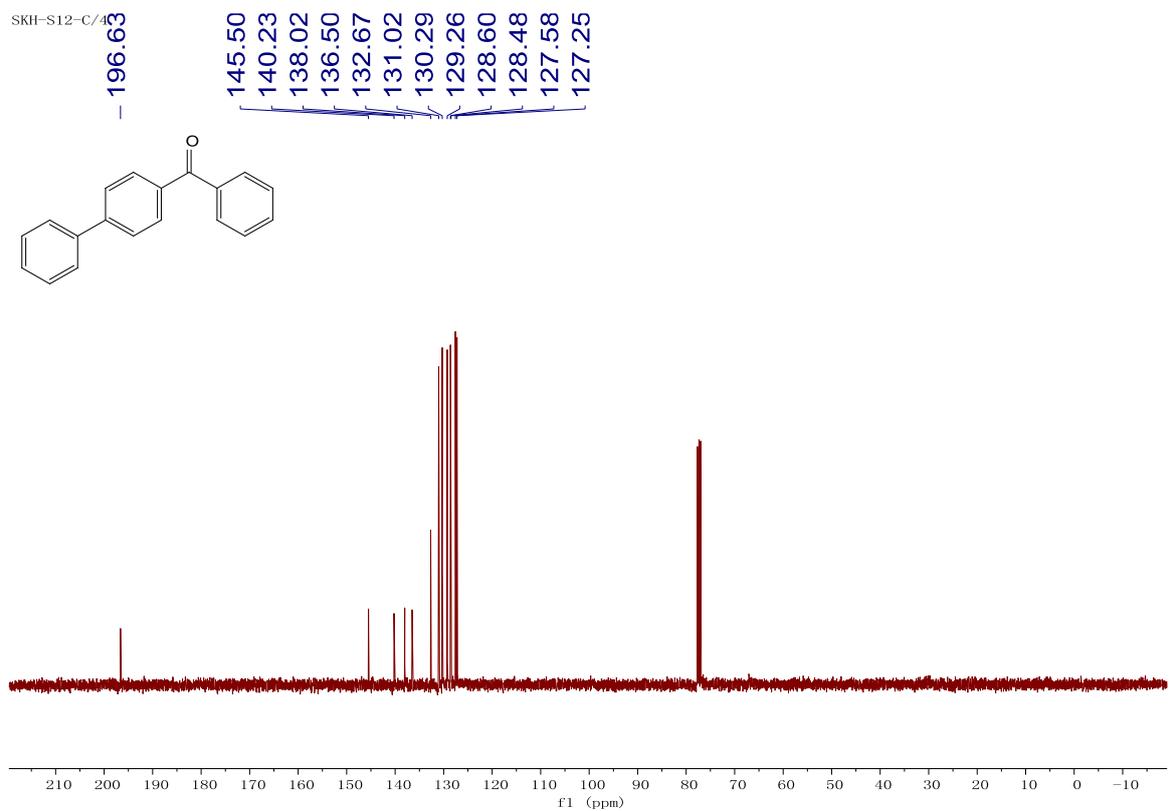
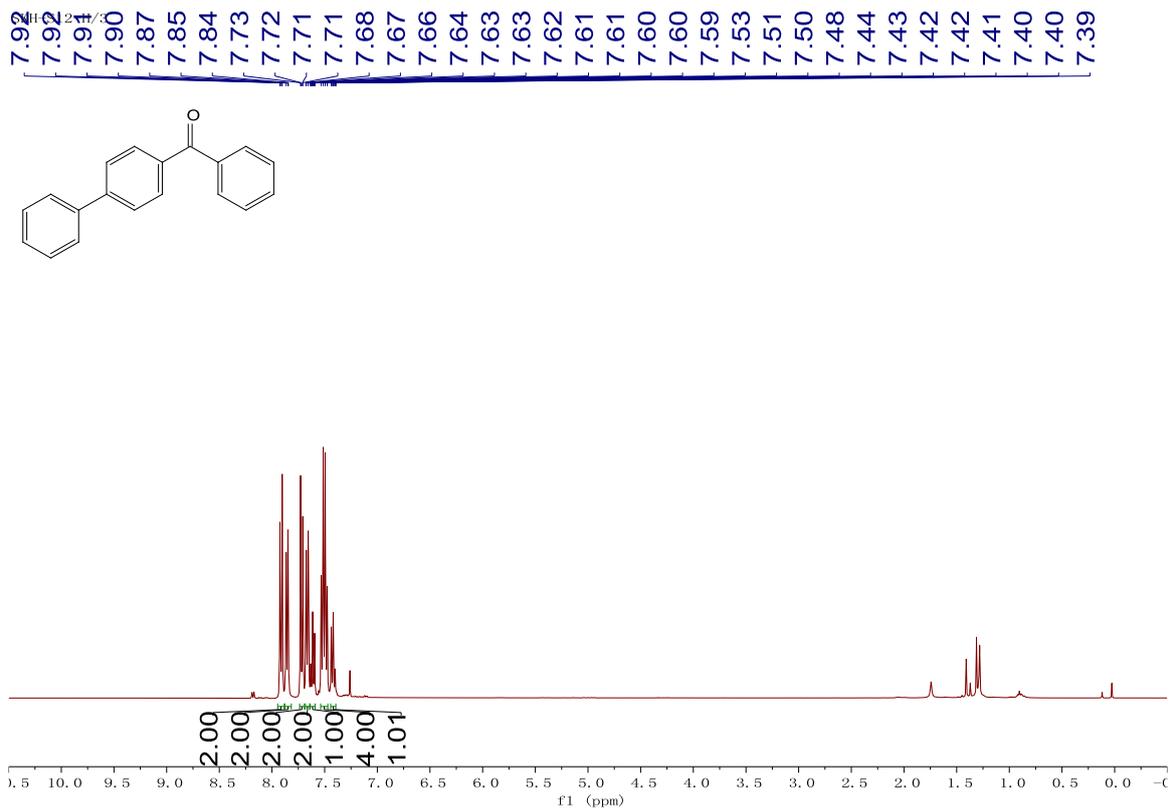


11. ^1H , ^{13}C and ^{19}F NMR spectra

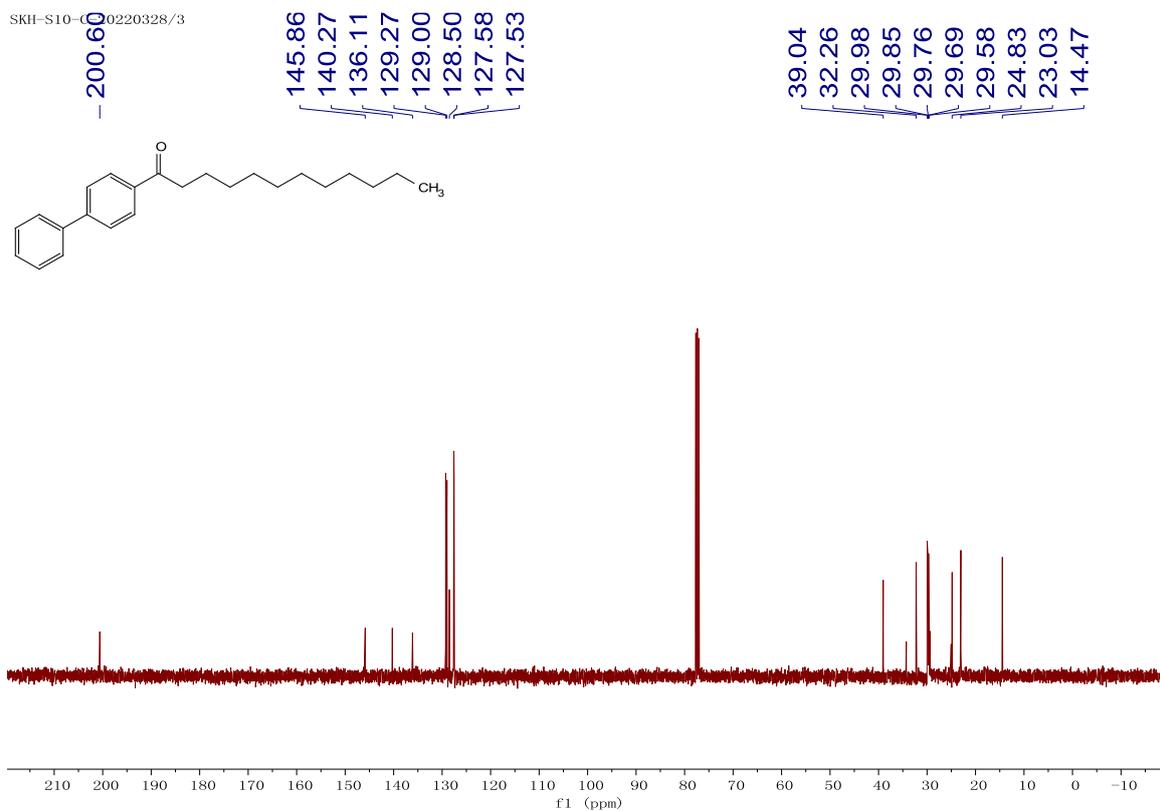
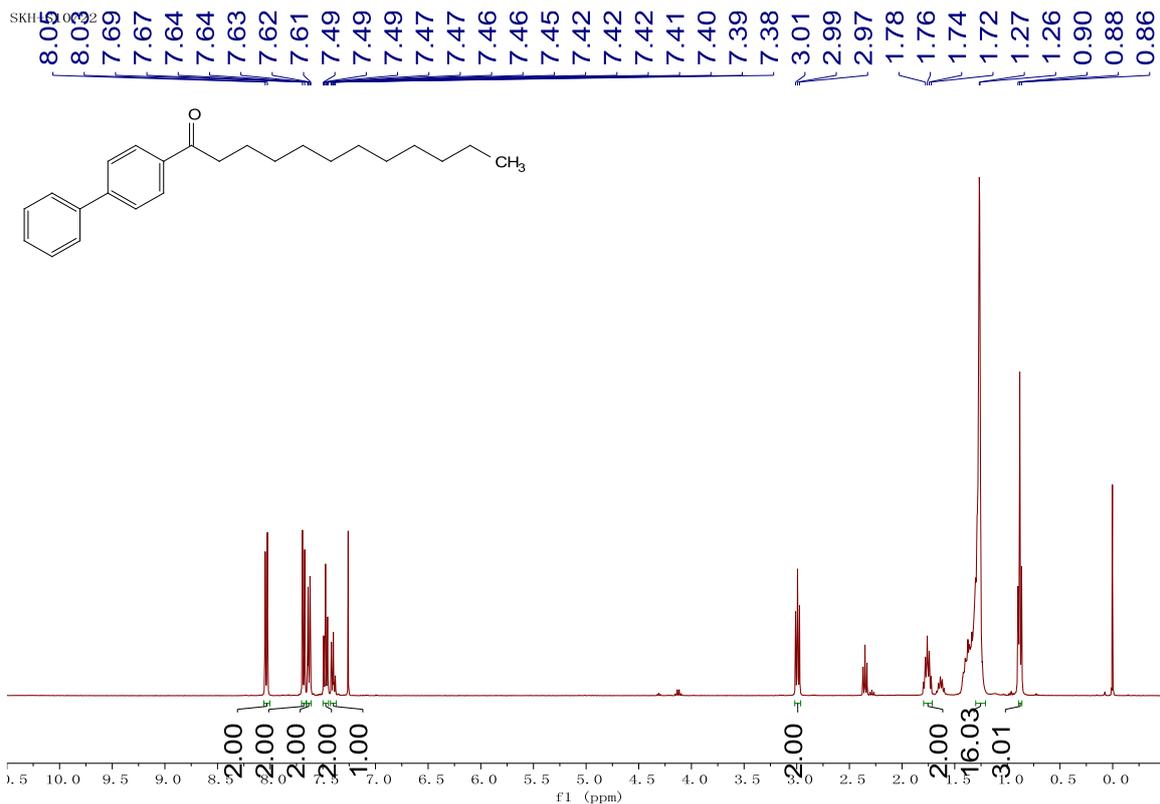
1e



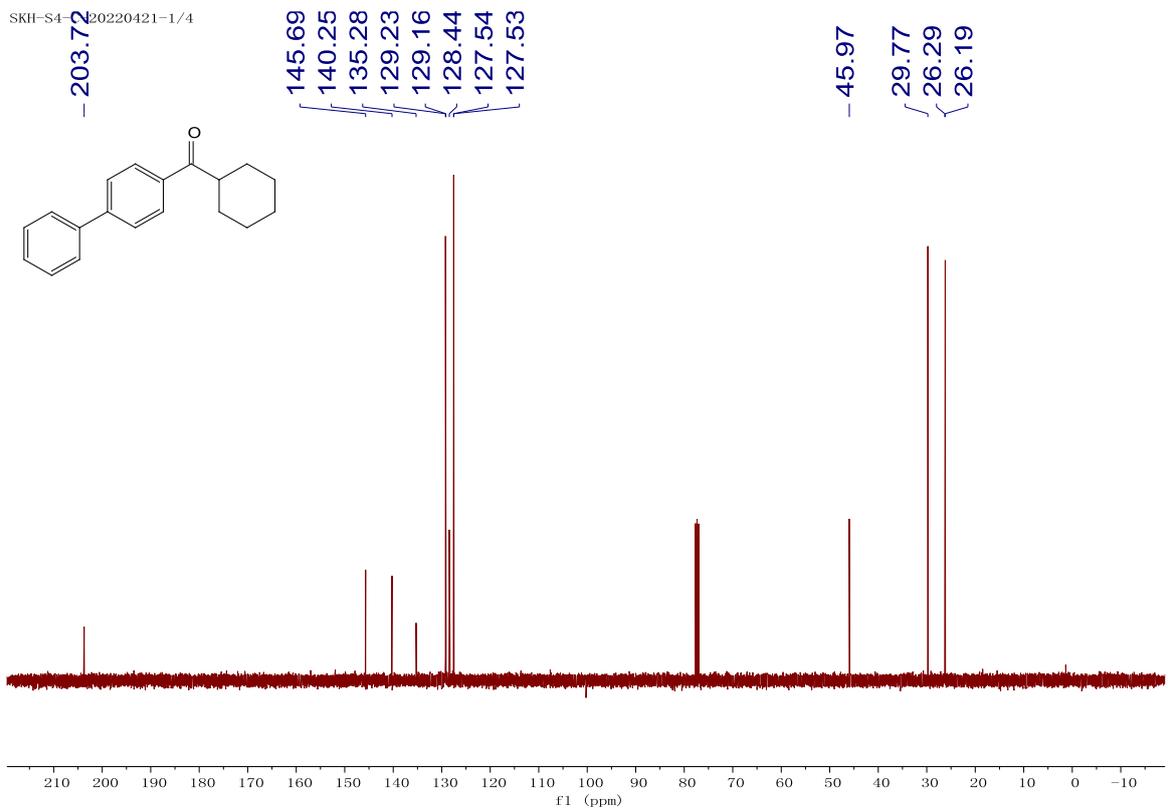
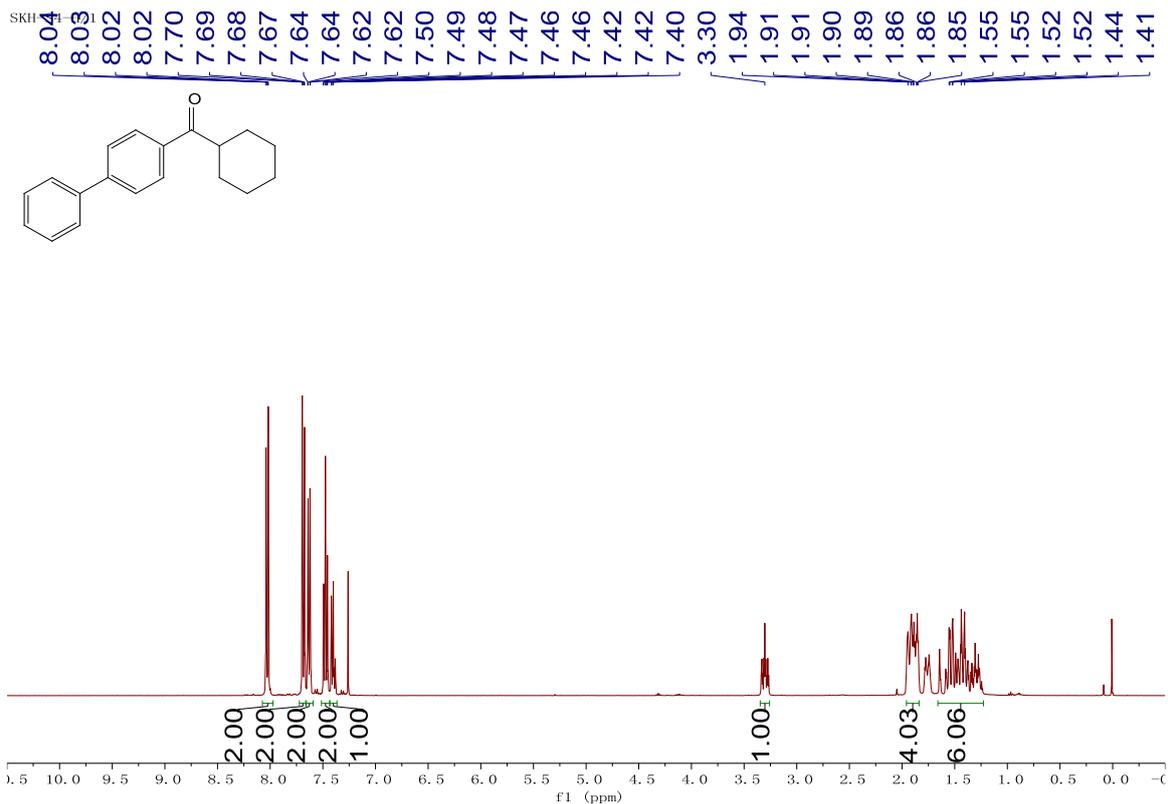
1m



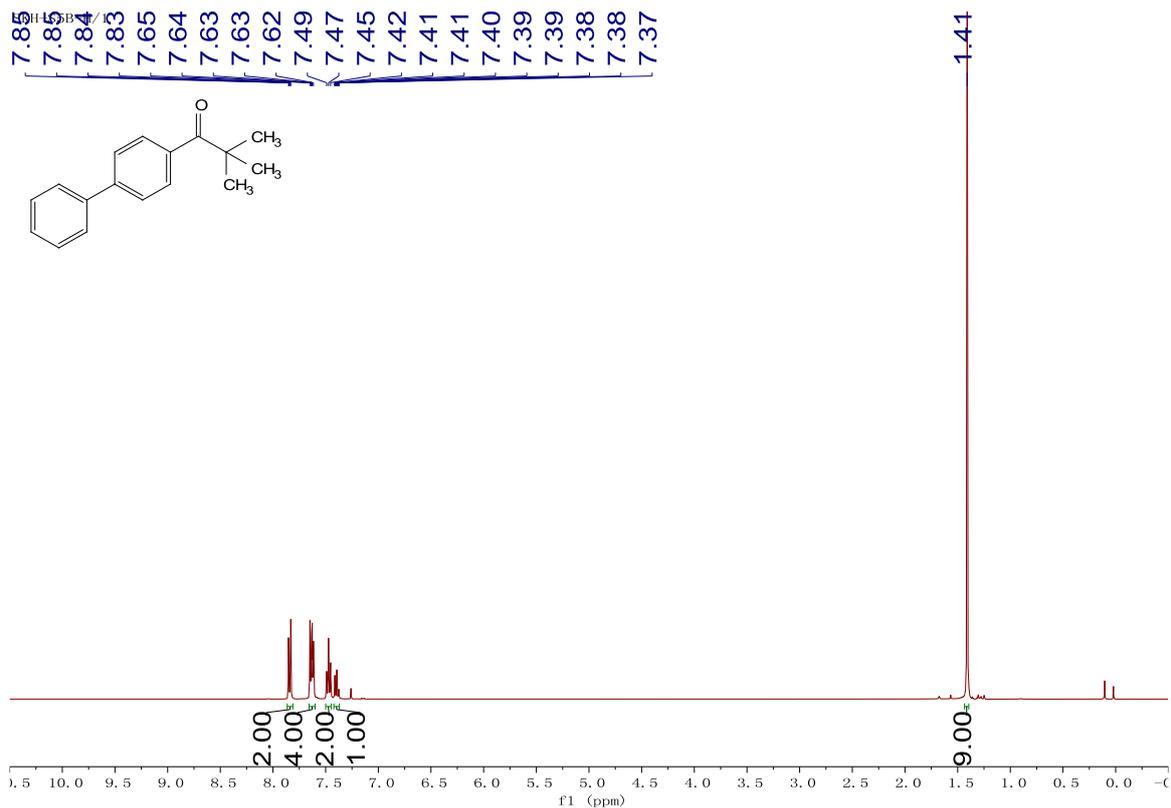
1w



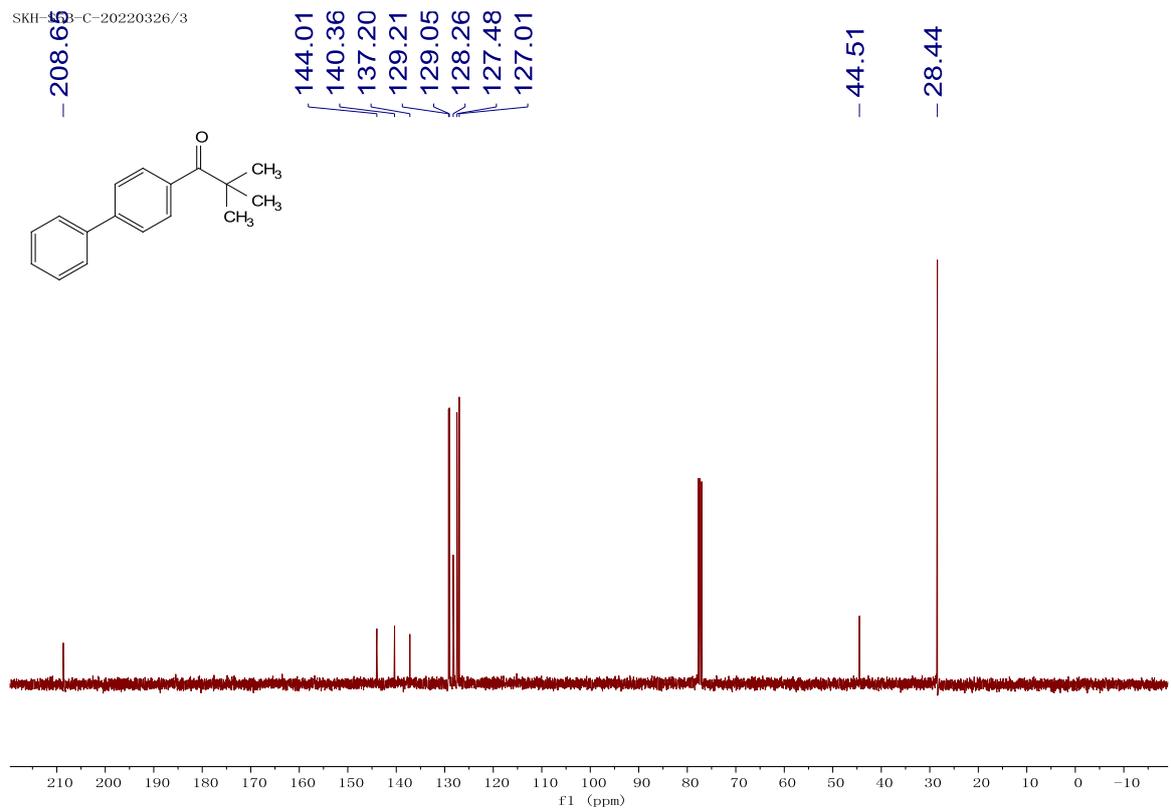
1x



1y

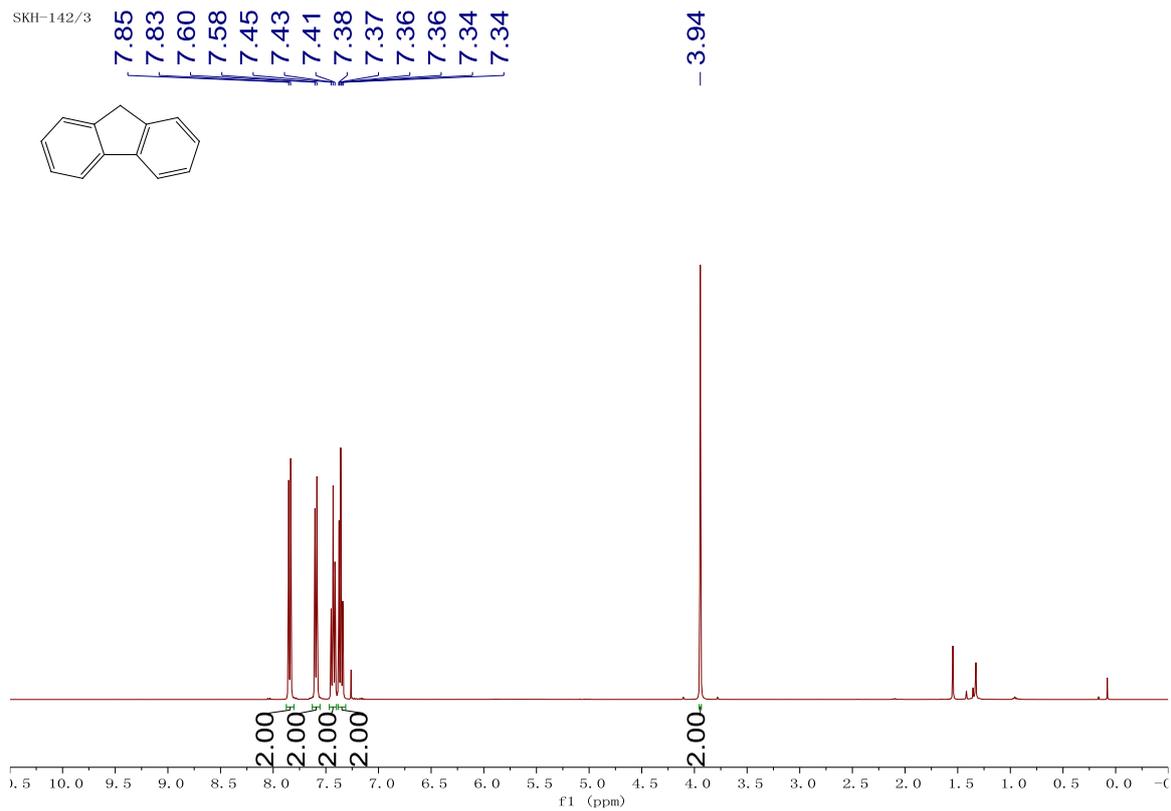
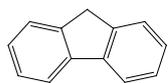


SKH-3-C-20220326/3

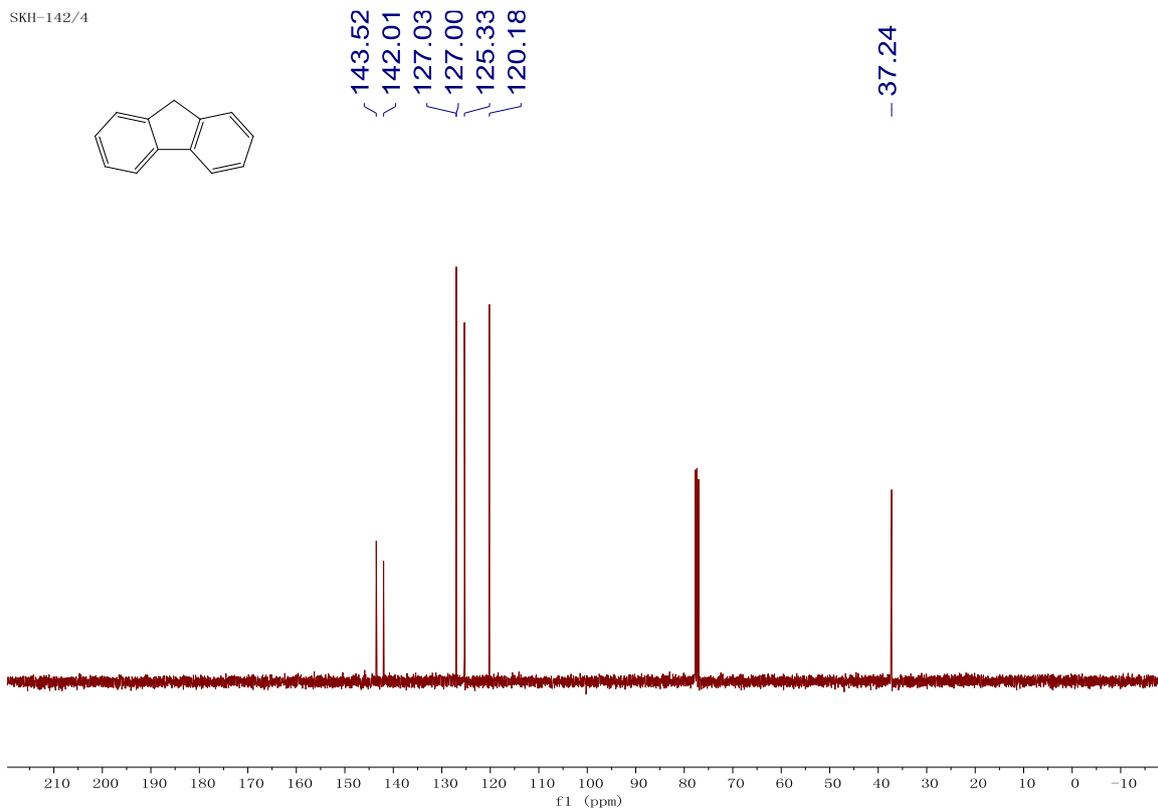
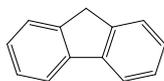


2a

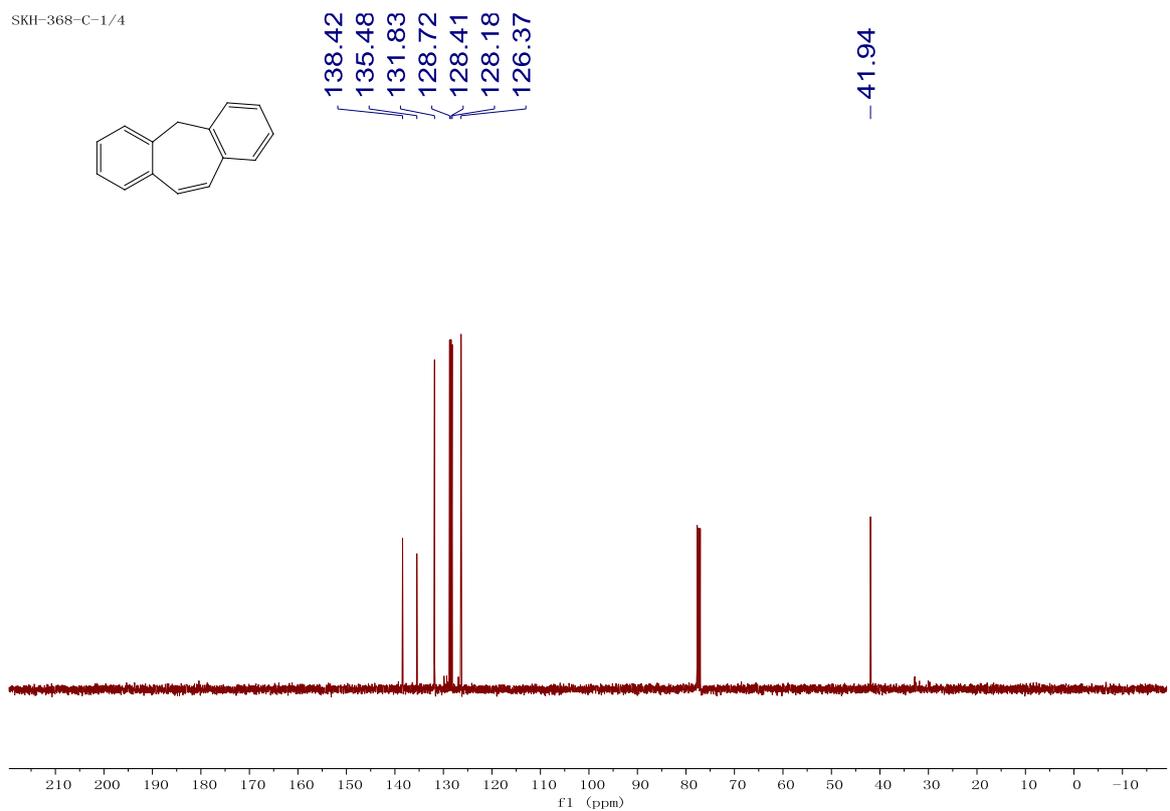
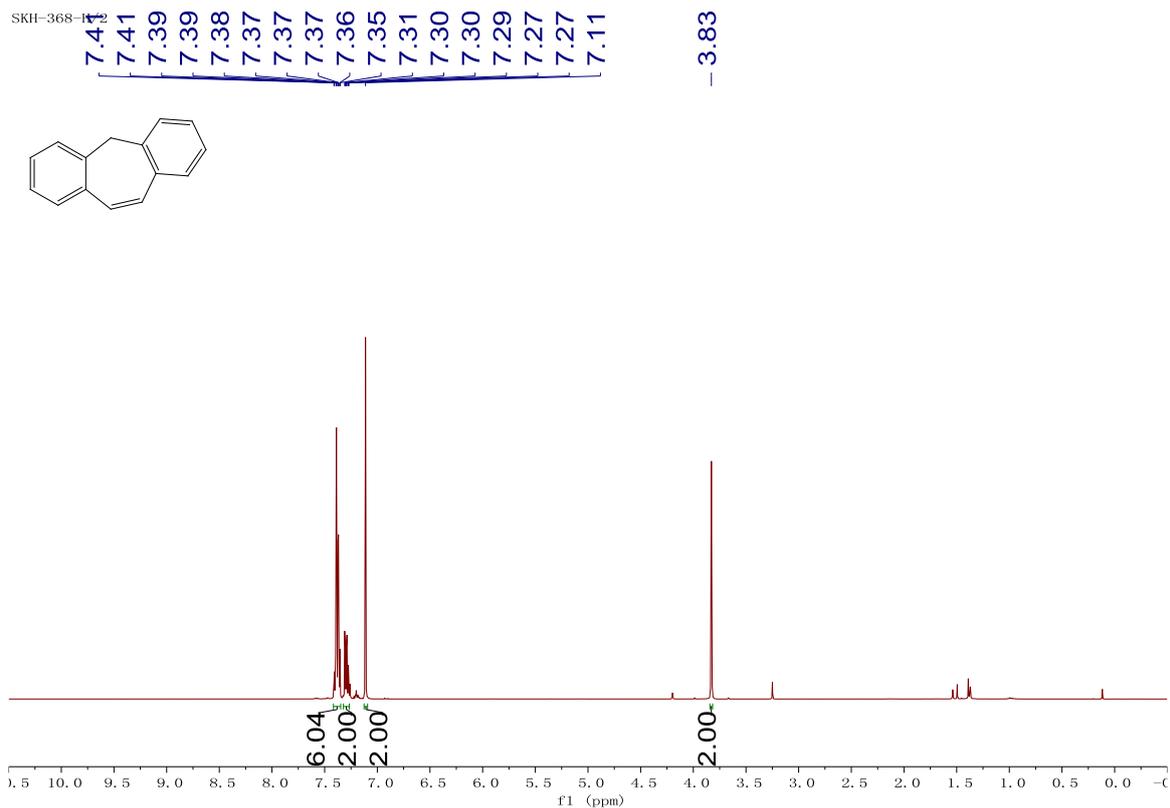
SKH-142/3



SKH-142/4

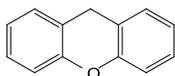


2b



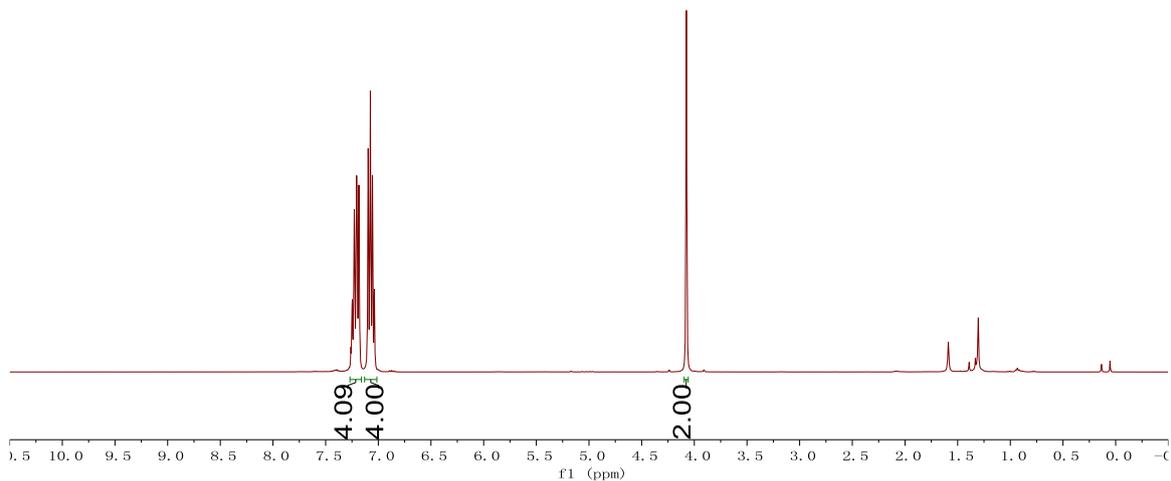
2c

skh-150/8

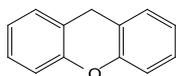


7.26
7.25
7.23
7.21
7.18
7.10
7.08
7.06
7.04

-4.08



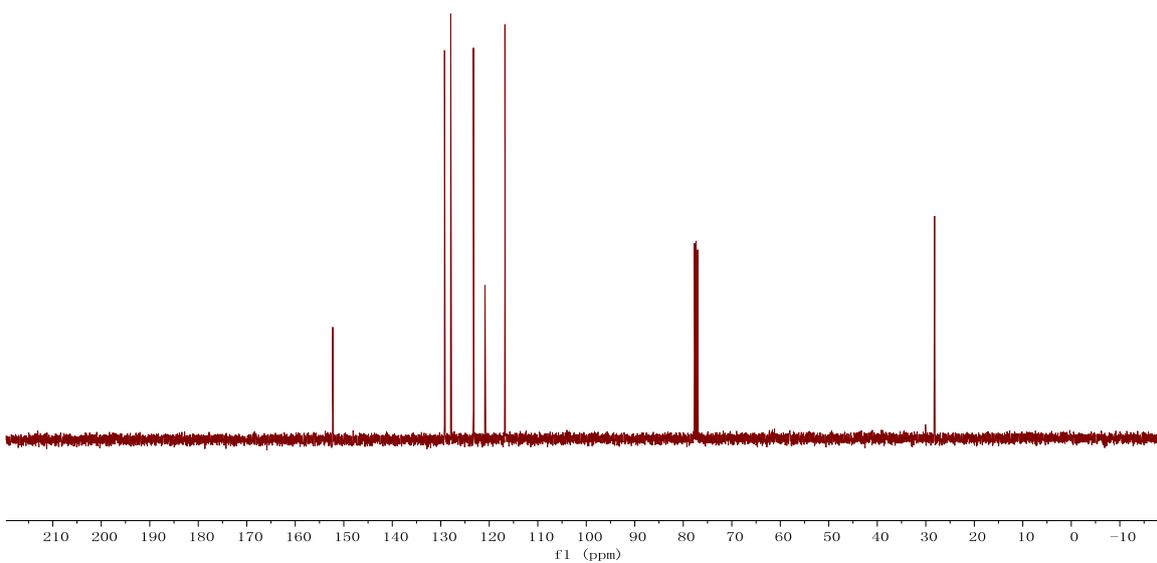
SKH-150-13C/1



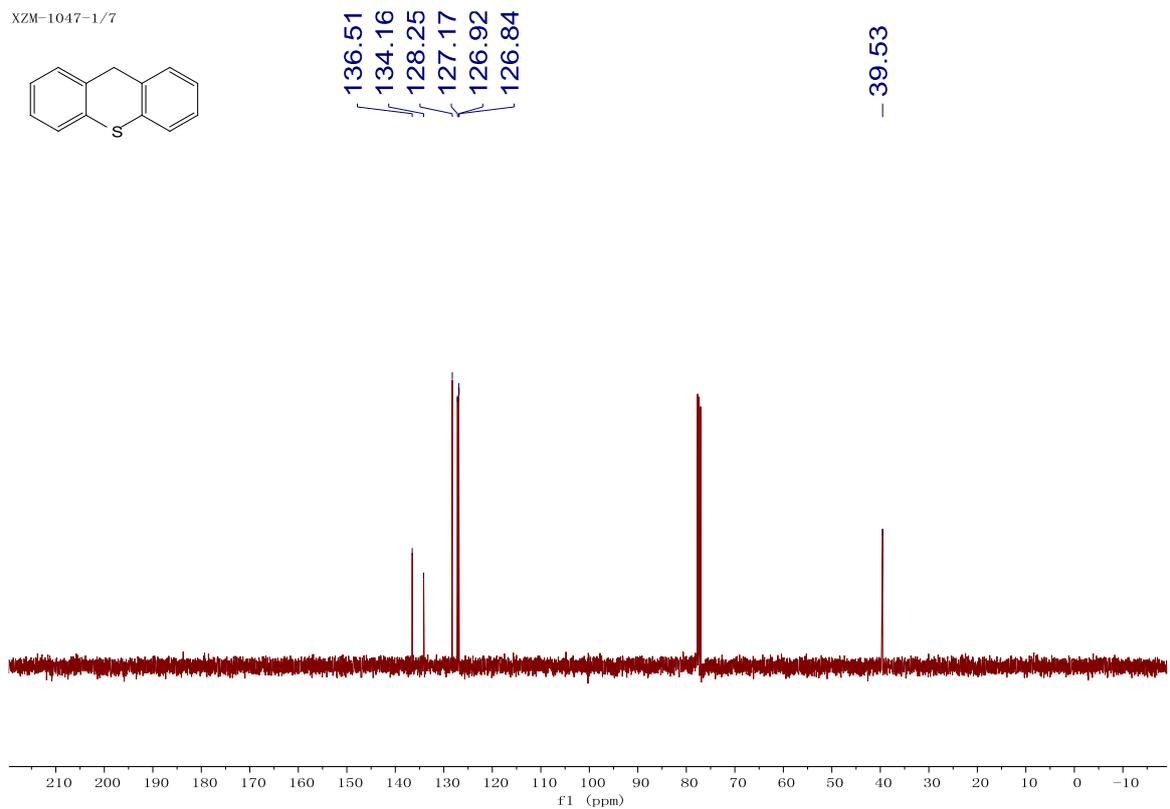
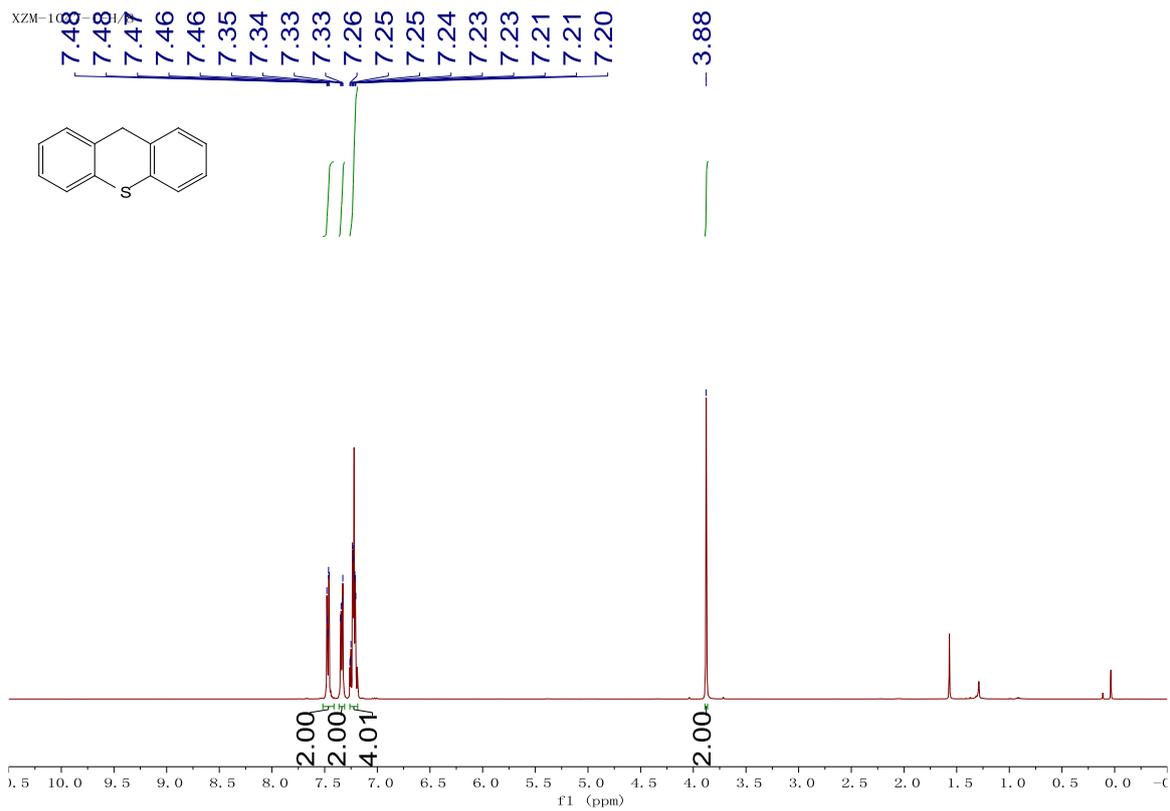
-152.26

129.23
127.95
123.26
120.88
116.77

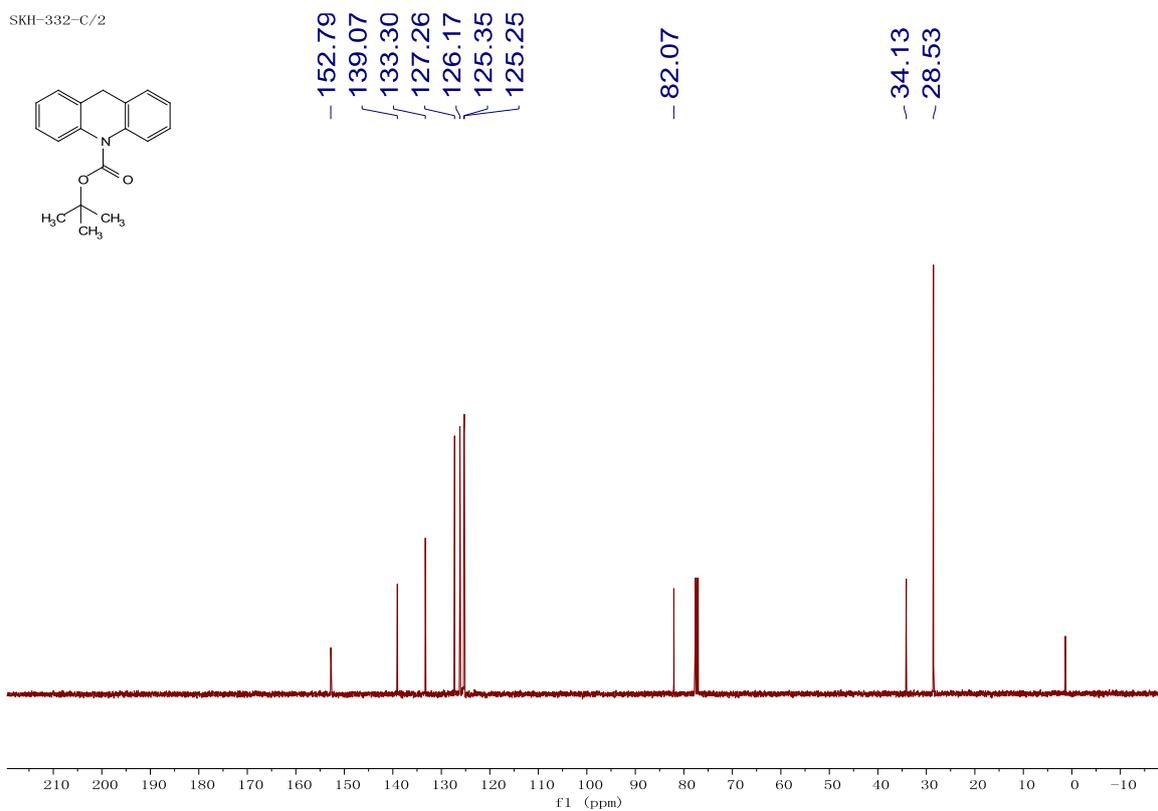
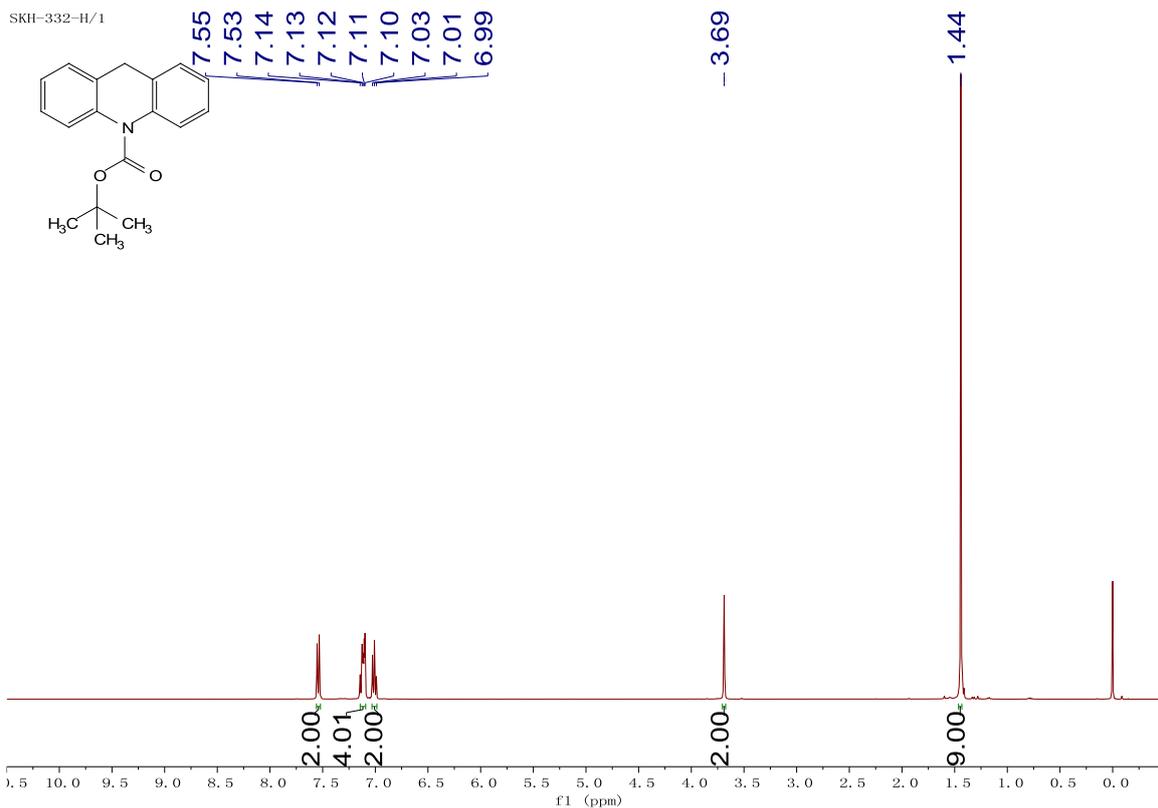
-28.19



2d

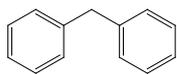


2e



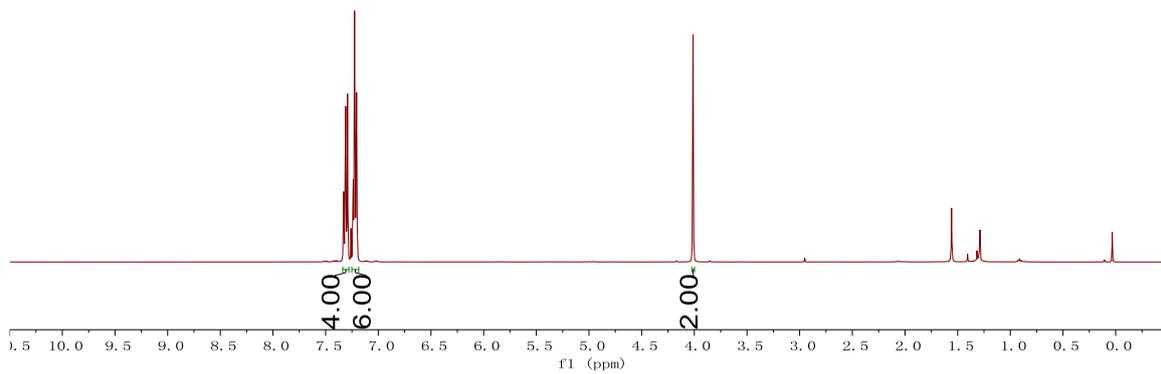
2f

XZM-1023-1/24
sw-63-S

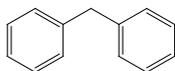


7.33
7.33
7.31
7.30
7.29
7.24
7.23
7.22
7.21

-4.01

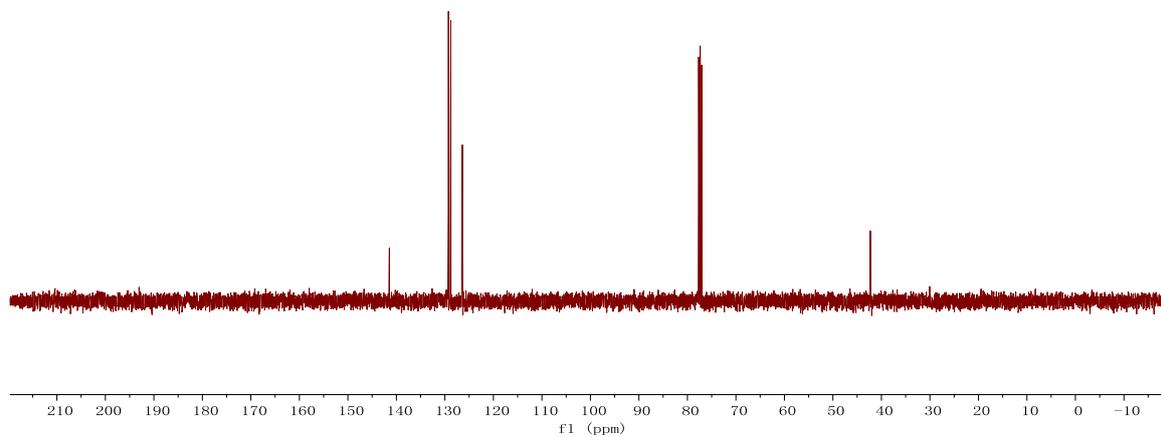


XZM-1023-1-C/25
sw-63-S



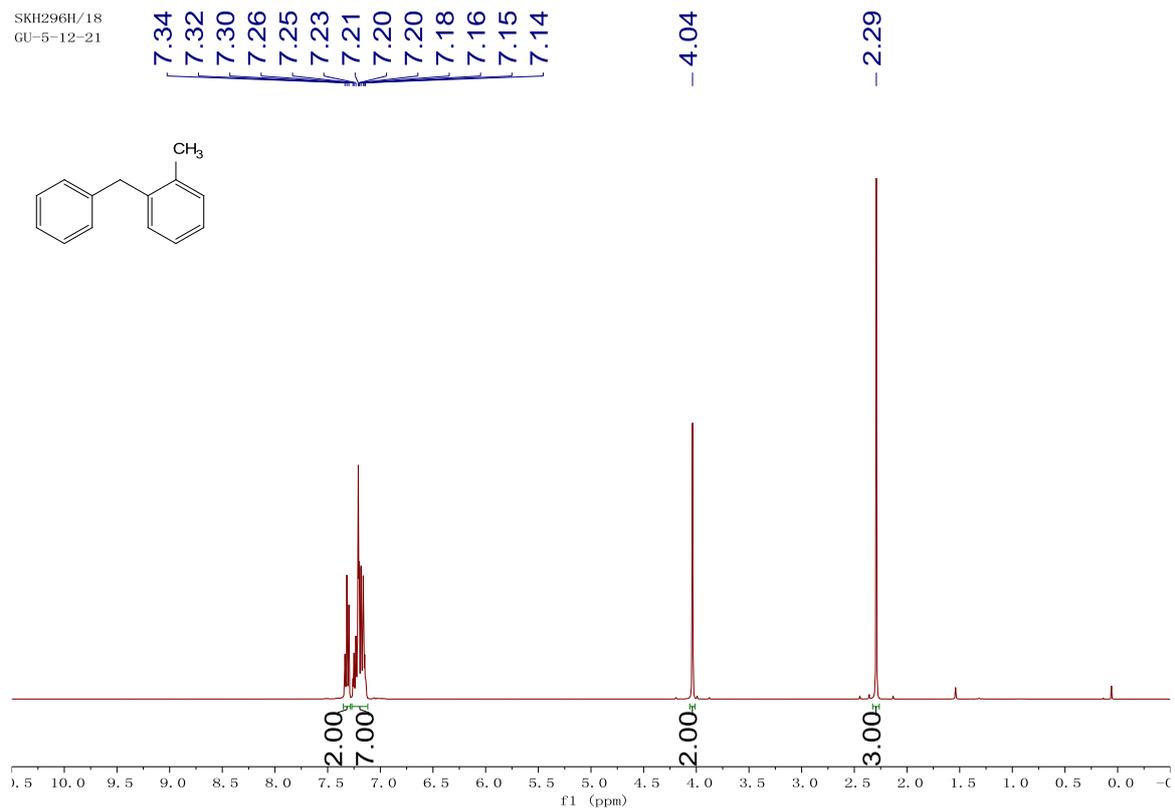
141.44
129.27
128.79
126.39

-42.28

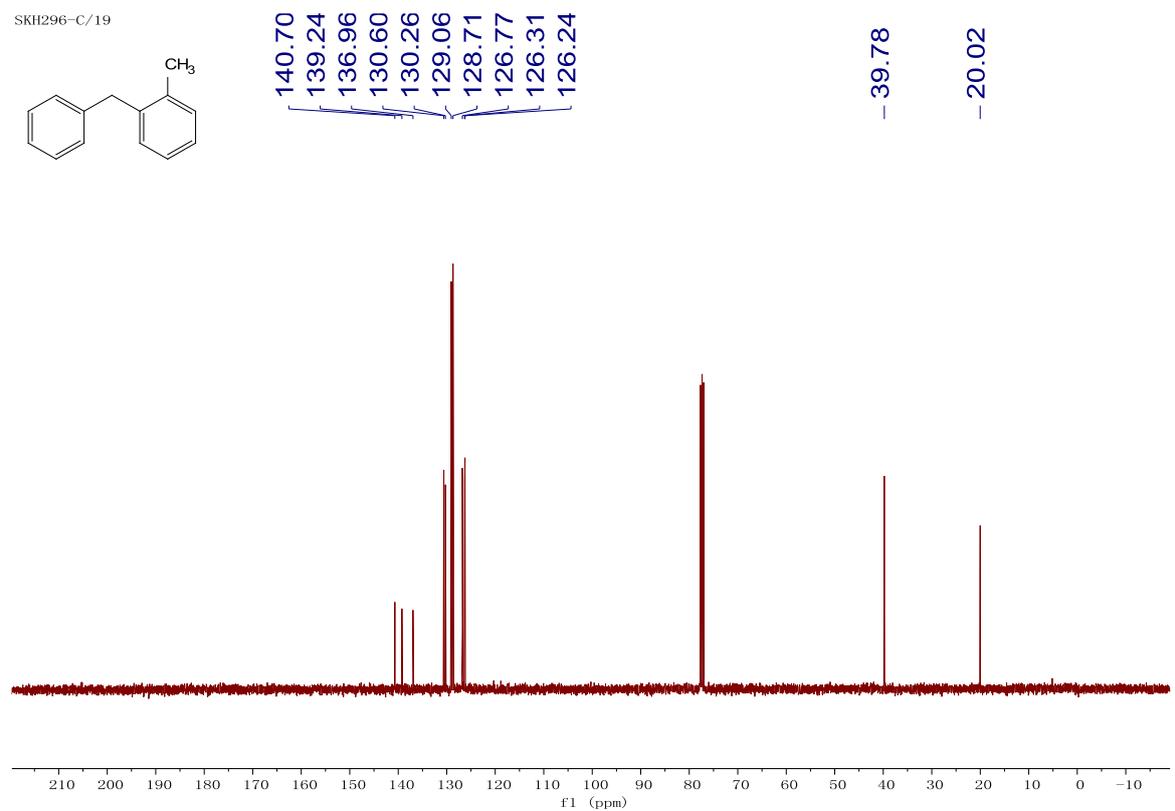


2g

SKH296H/18
GU-5-12-21

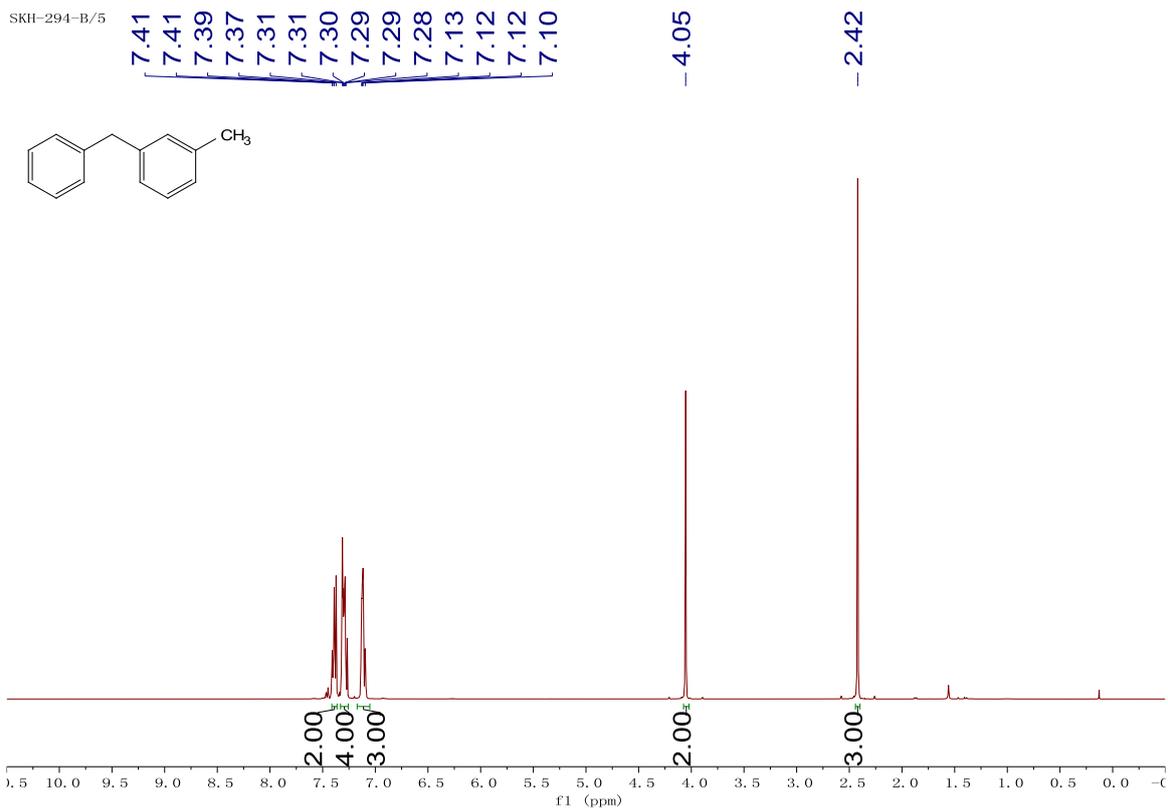


SKH296-C/19

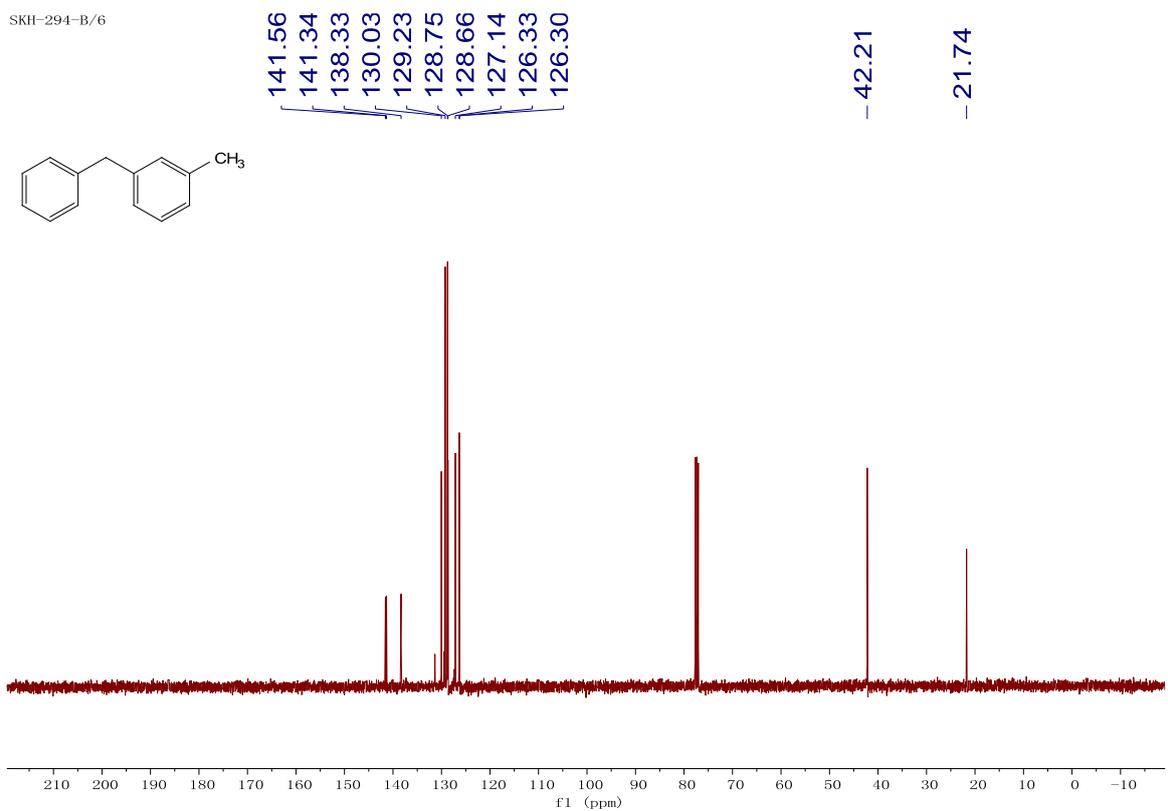


2h

SKH-294-B/5

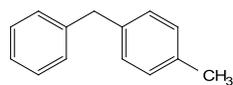


SKH-294-B/6



2i

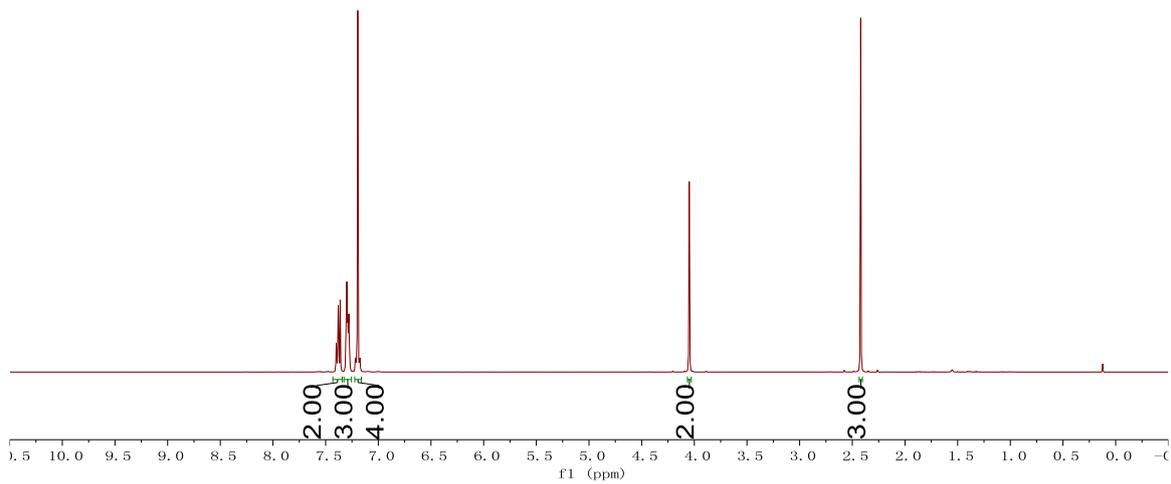
SKH-284-B/1



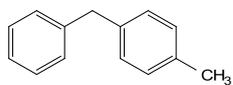
7.40
7.38
7.36
7.31
7.30
7.29
7.28
7.22
7.20
7.18
7.17

-4.05

-2.42



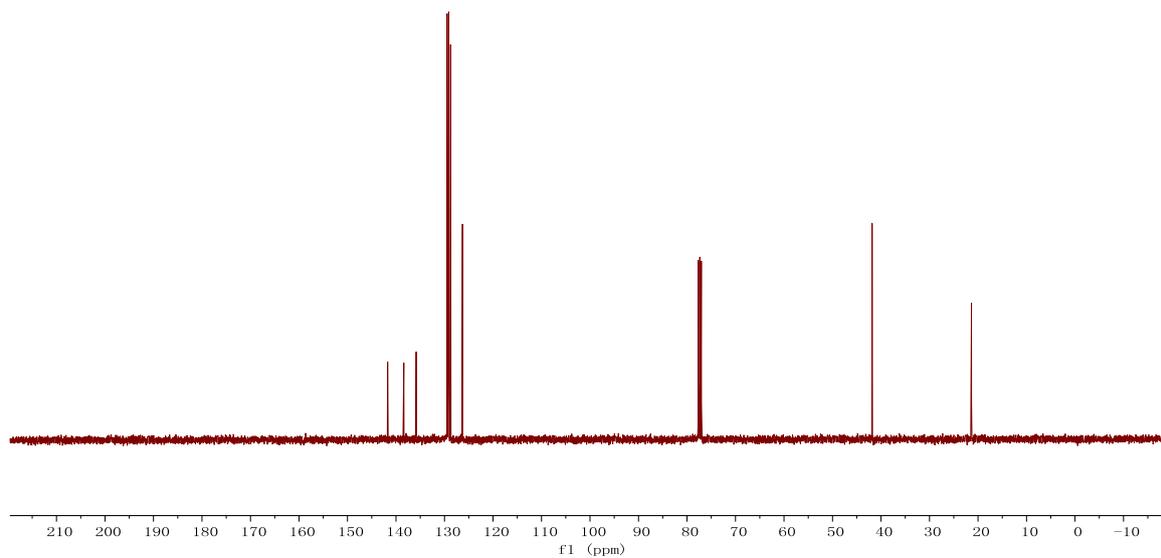
SKH-284-B-13C/3



141.72
138.39
135.84
129.47
129.19
129.13
128.75
126.29

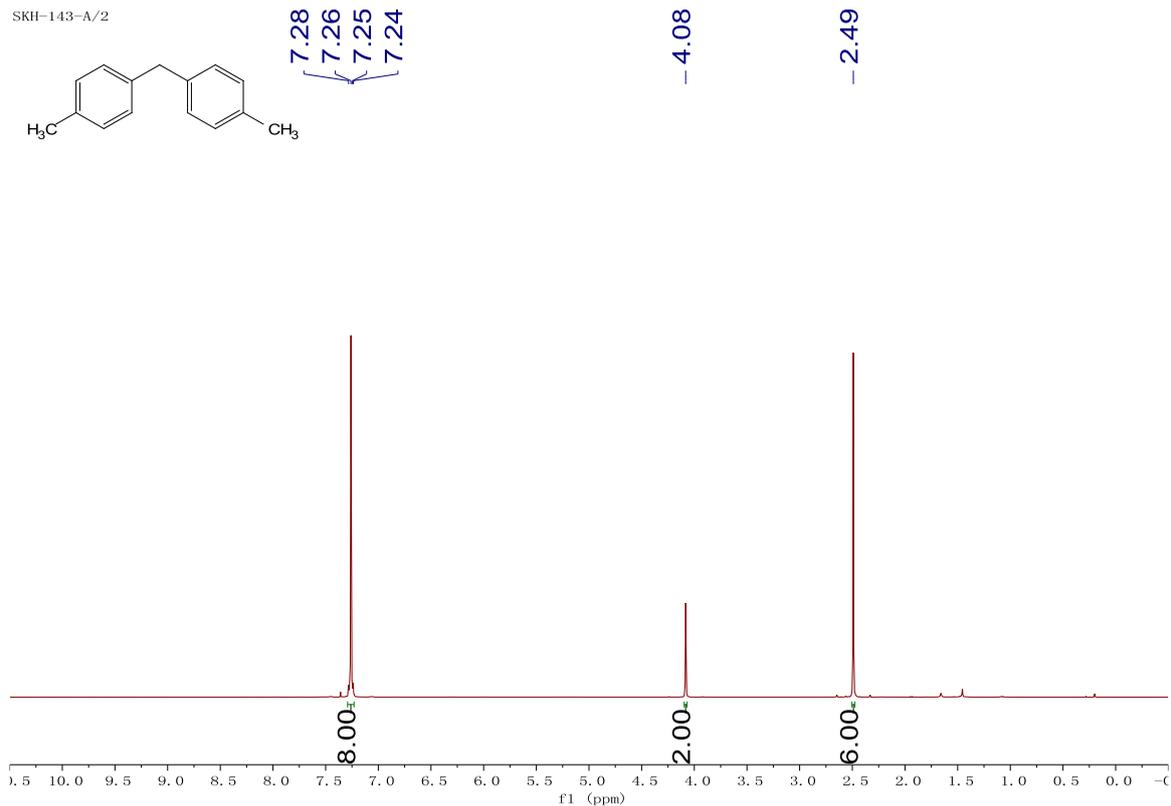
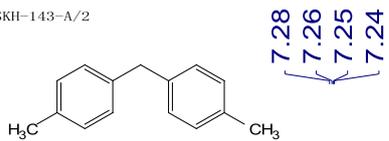
-41.83

-21.35

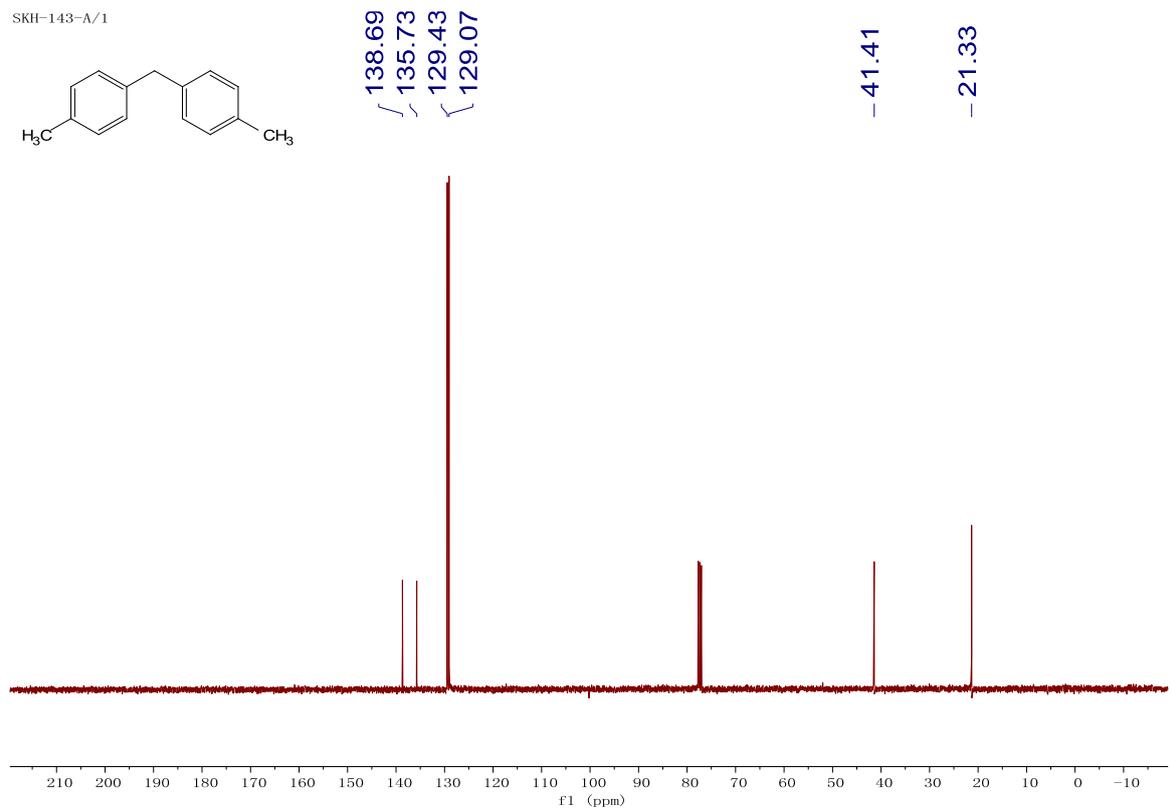
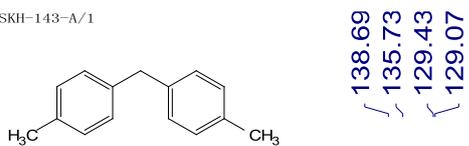


2j

SKH-143-A/2

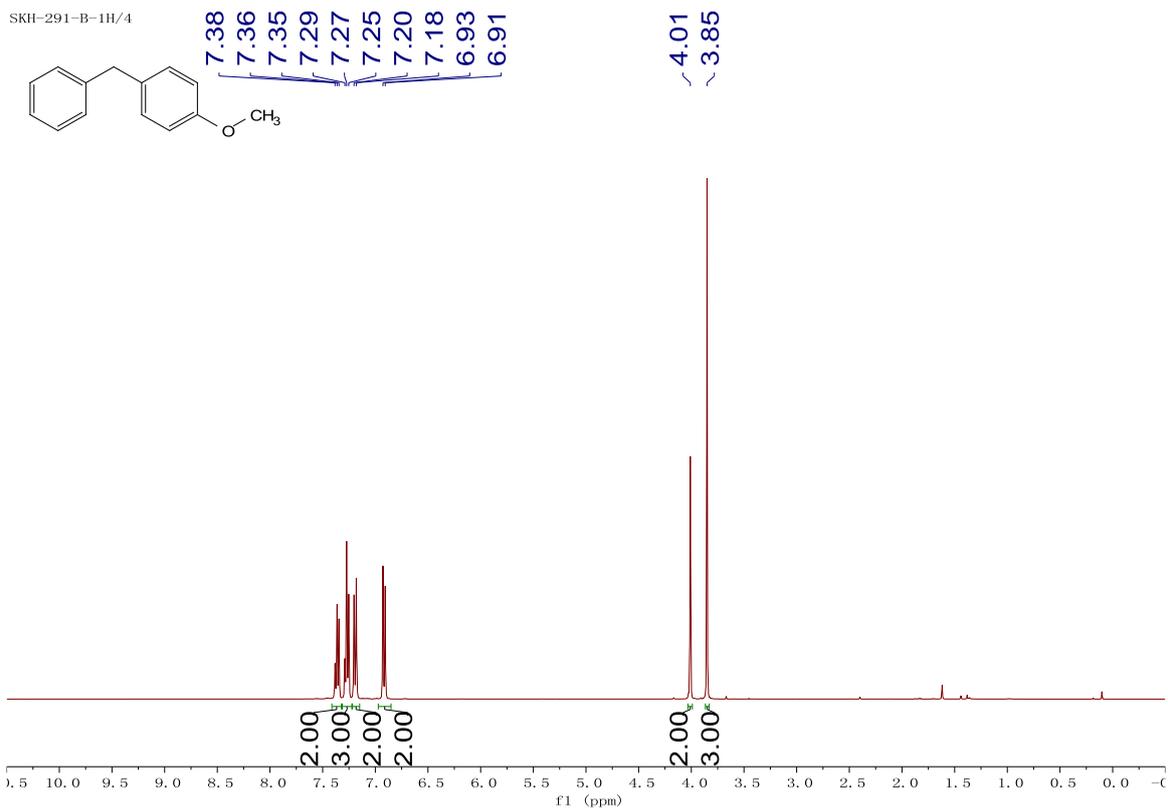


SKH-143-A/1

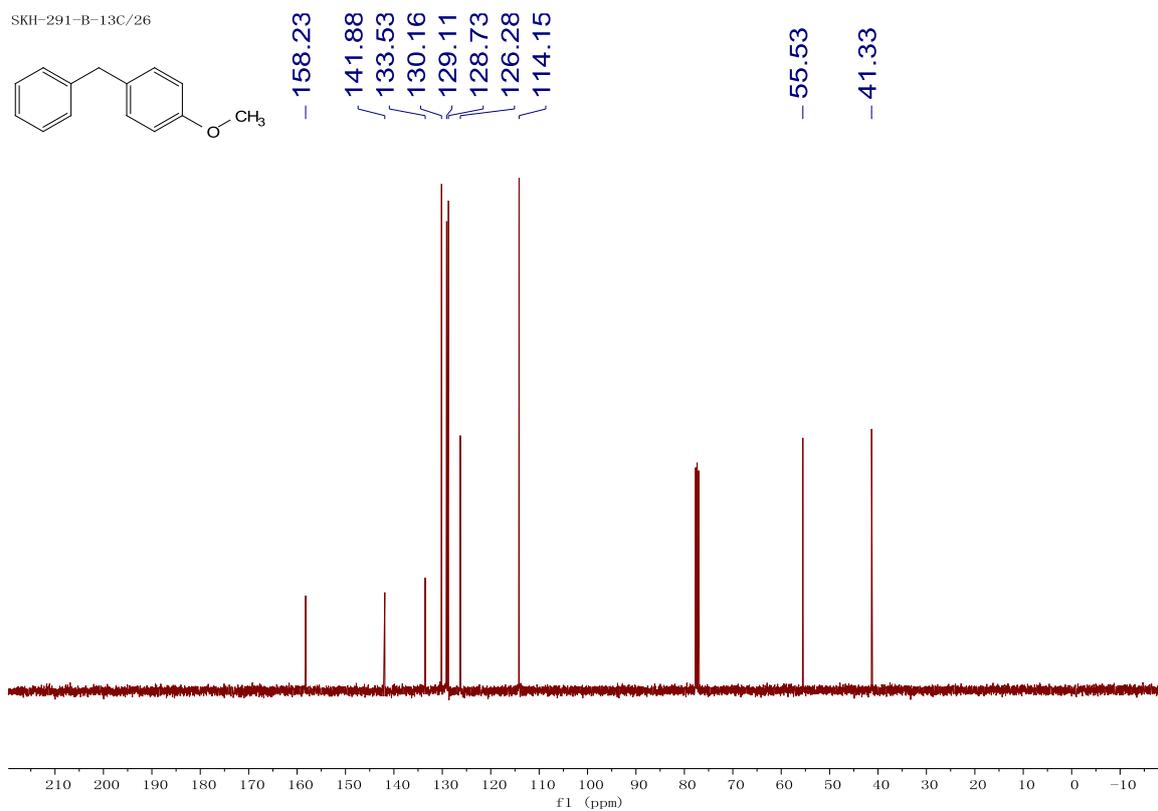


2k

SKH-291-B-1H/4

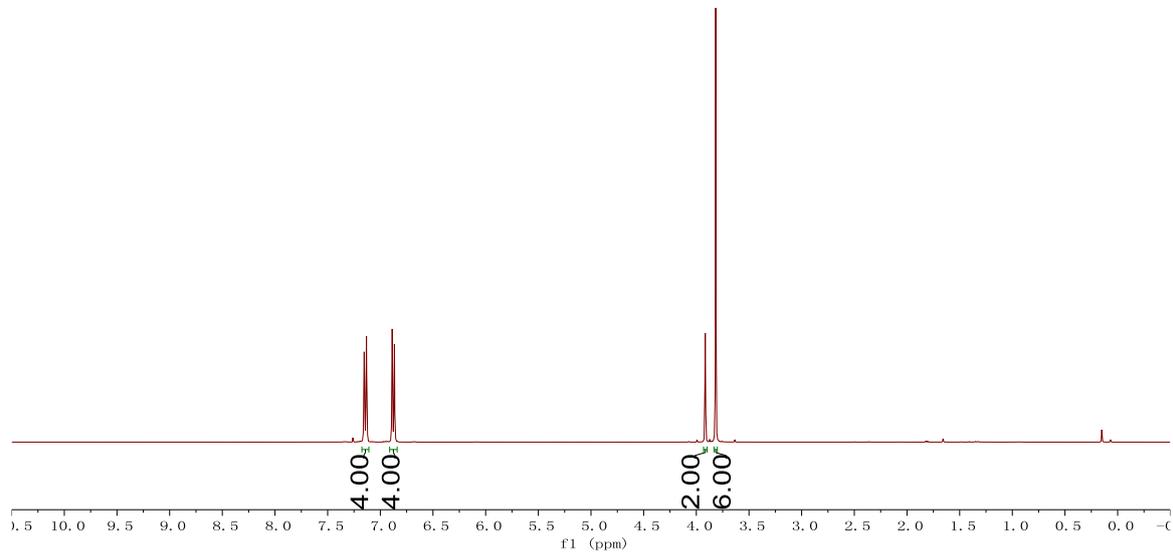
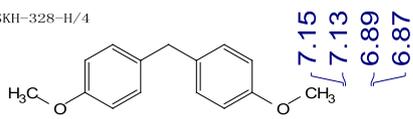


SKH-291-B-13C/26

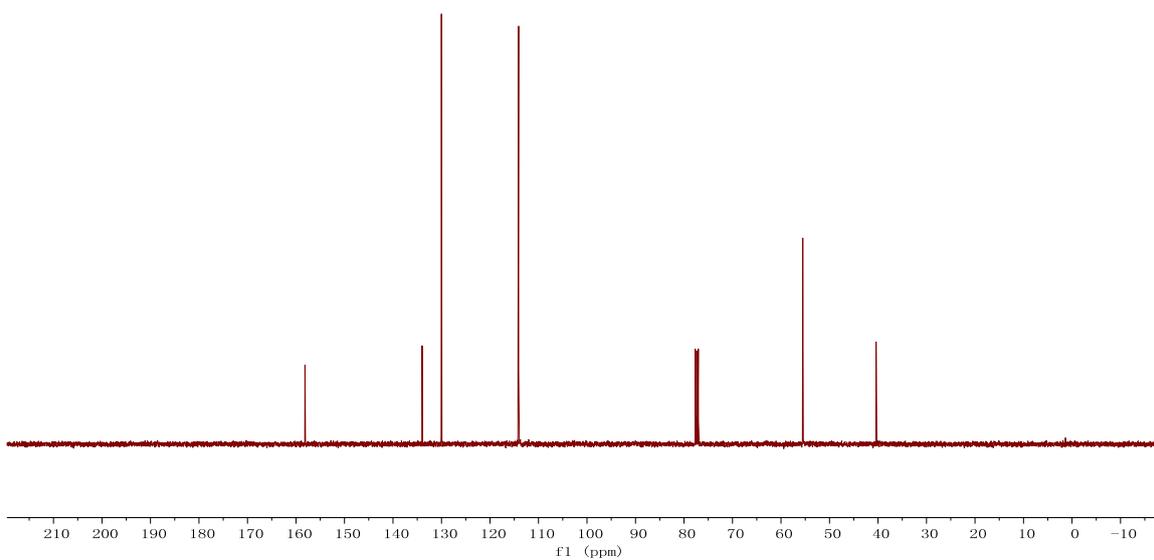
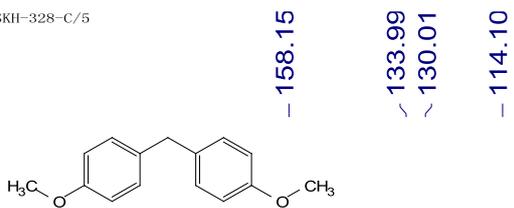


21

SKH-328-H/4

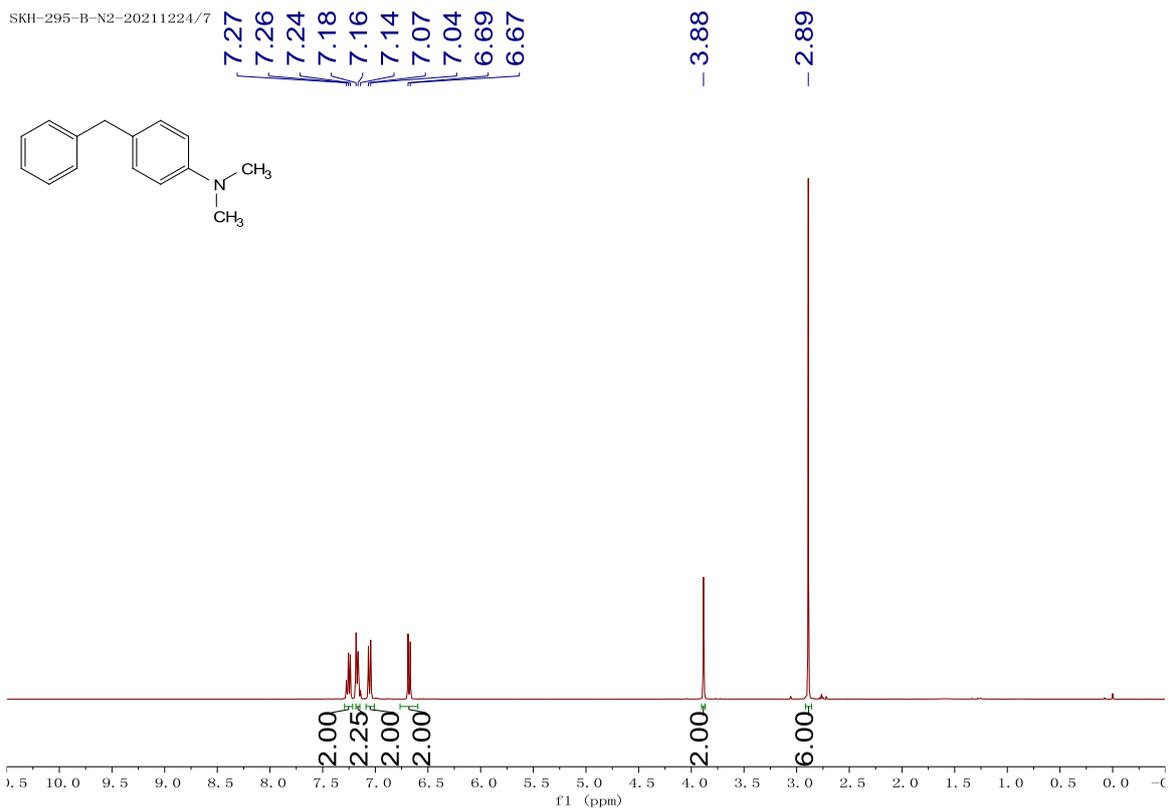


SKH-328-C/5

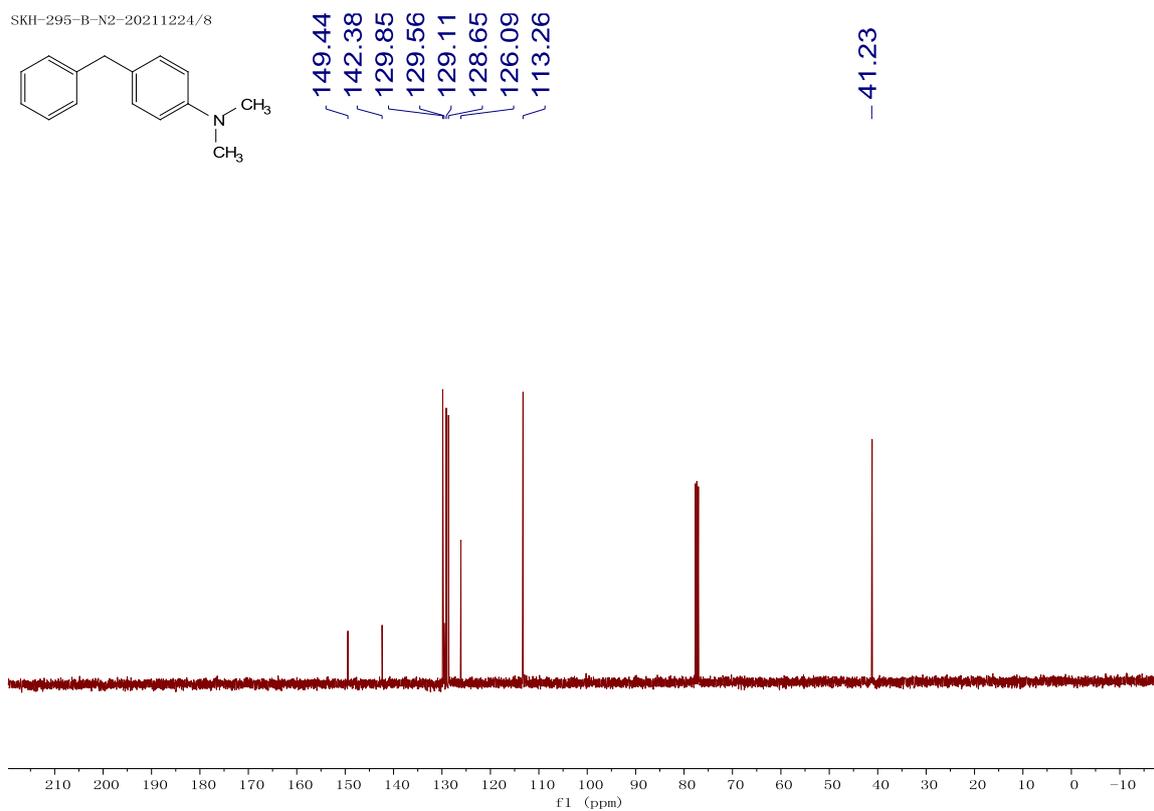


2m

SKH-295-B-N2-20211224/7

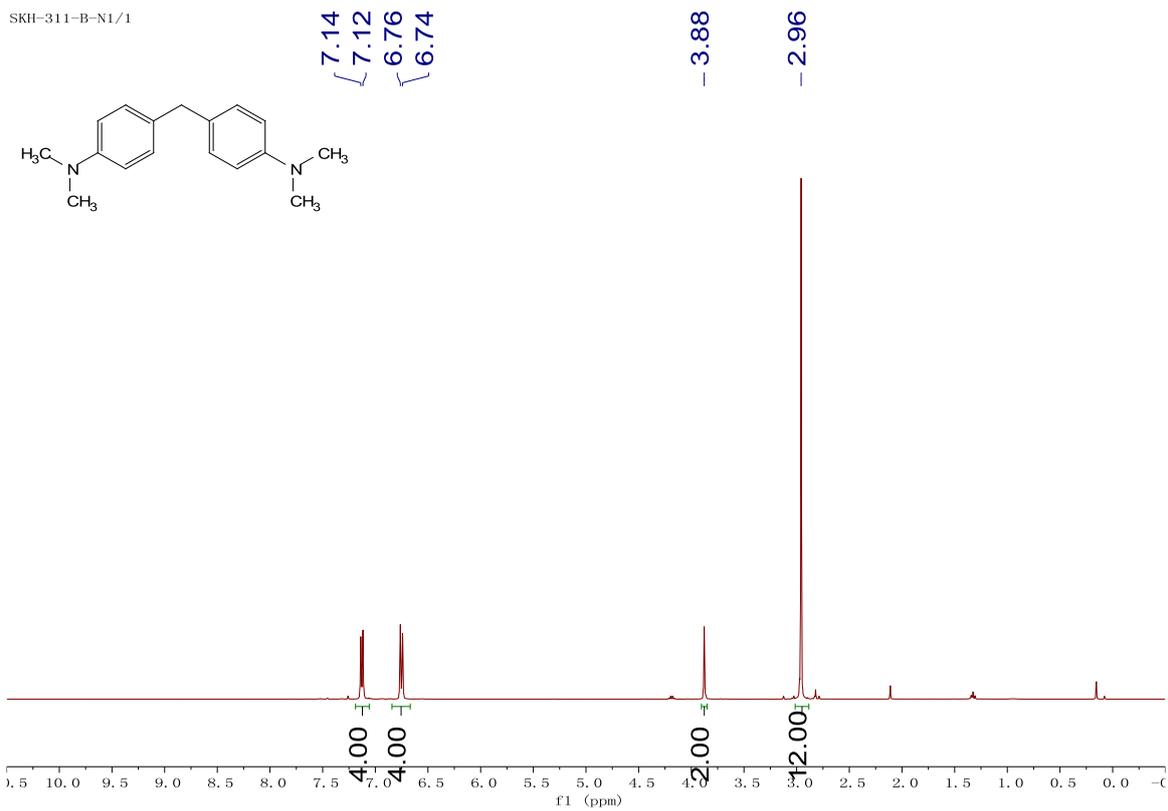


SKH-295-B-N2-20211224/8

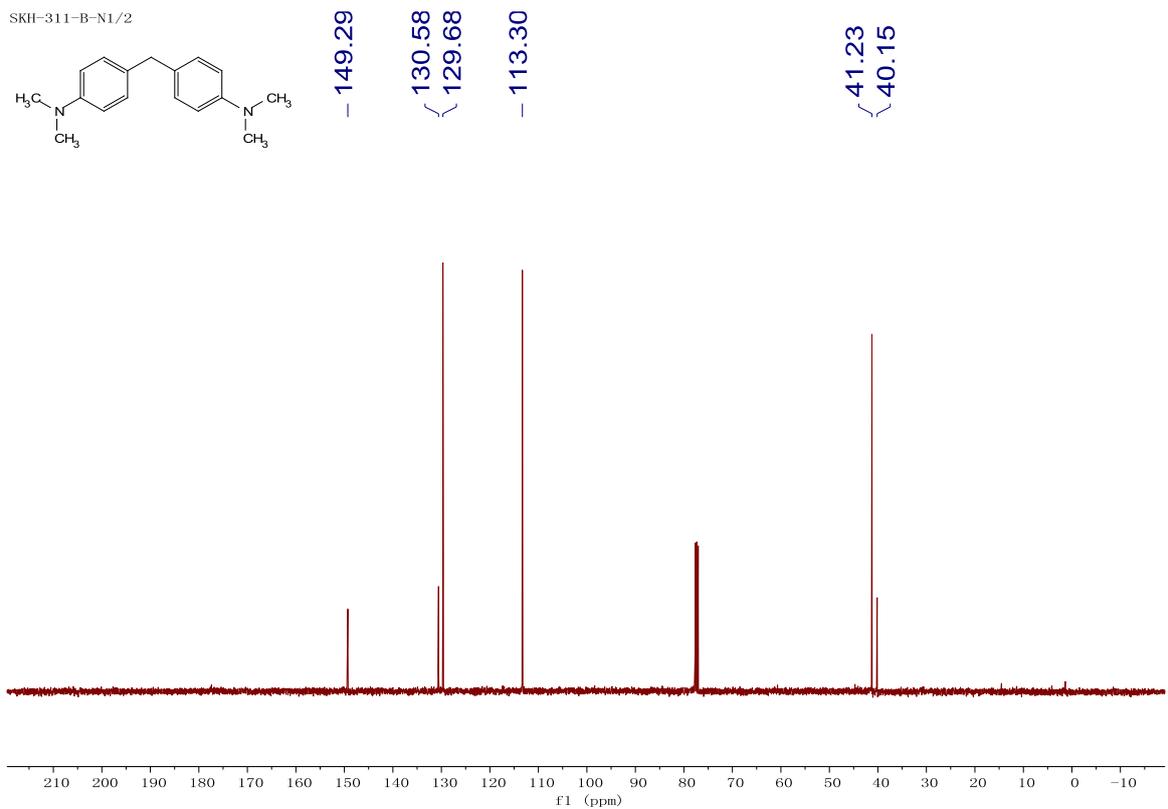


2n

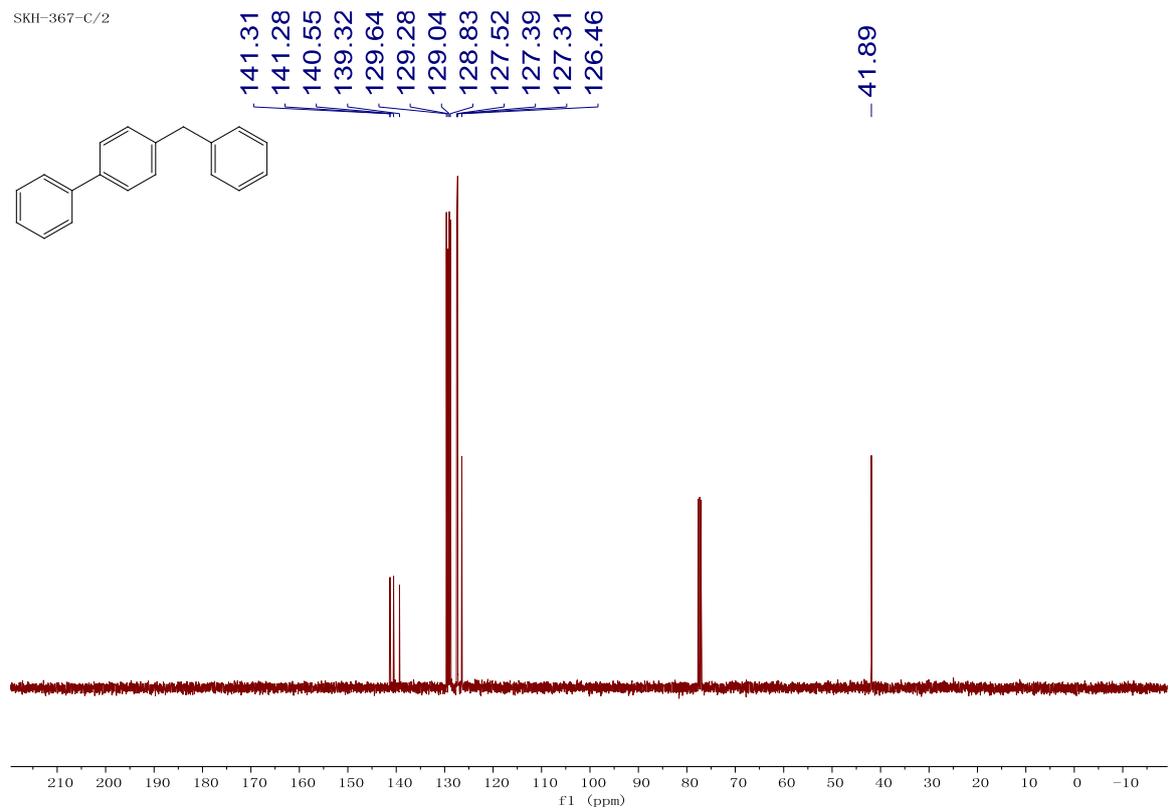
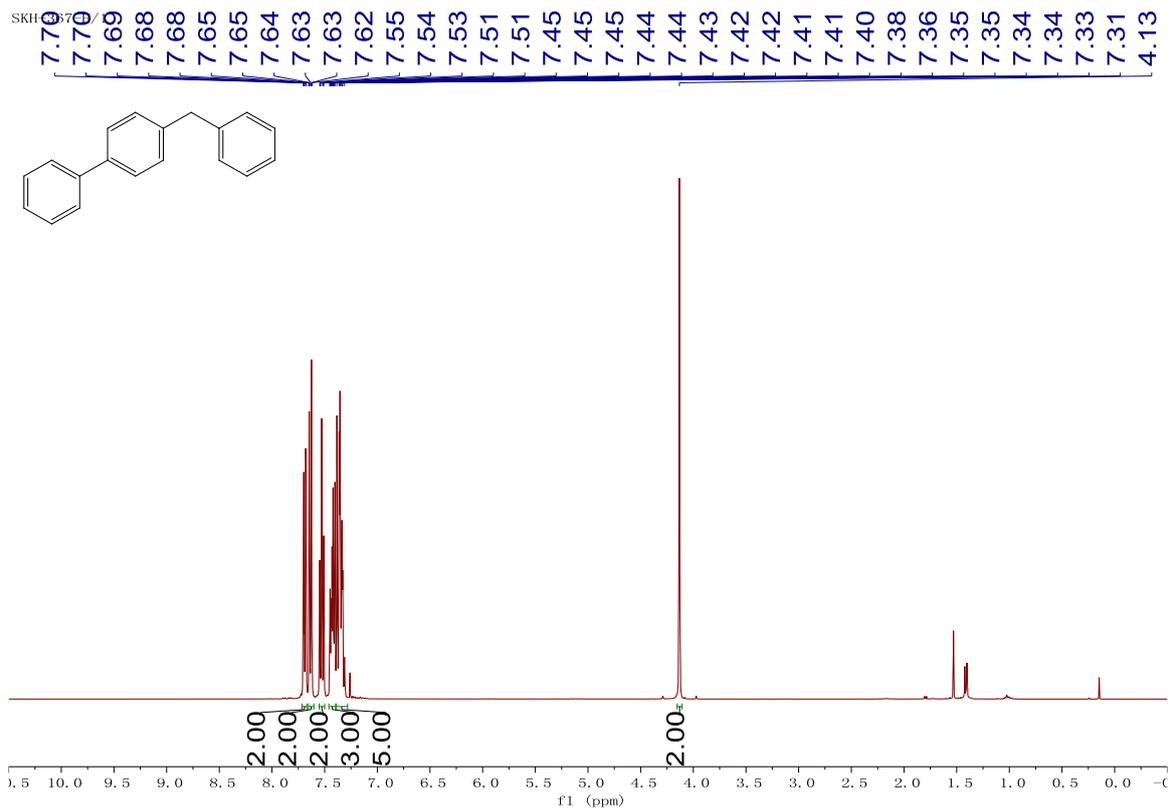
SKH-311-B-N1/1



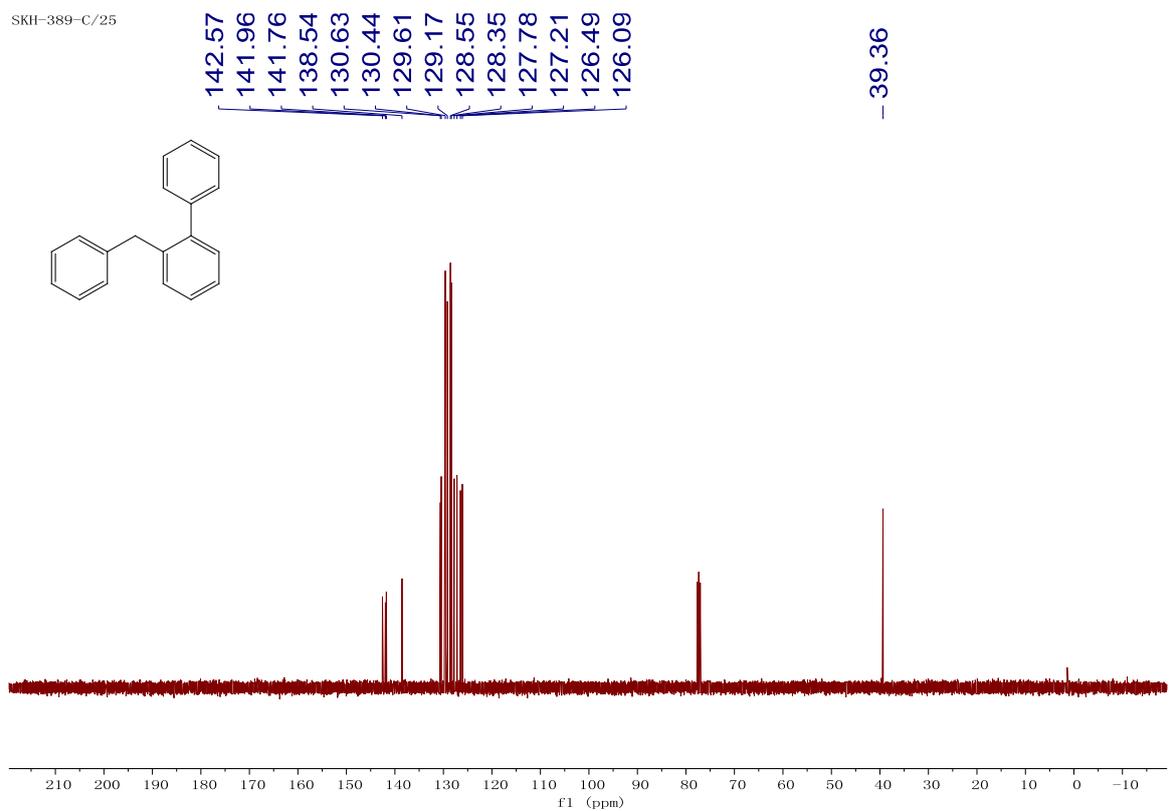
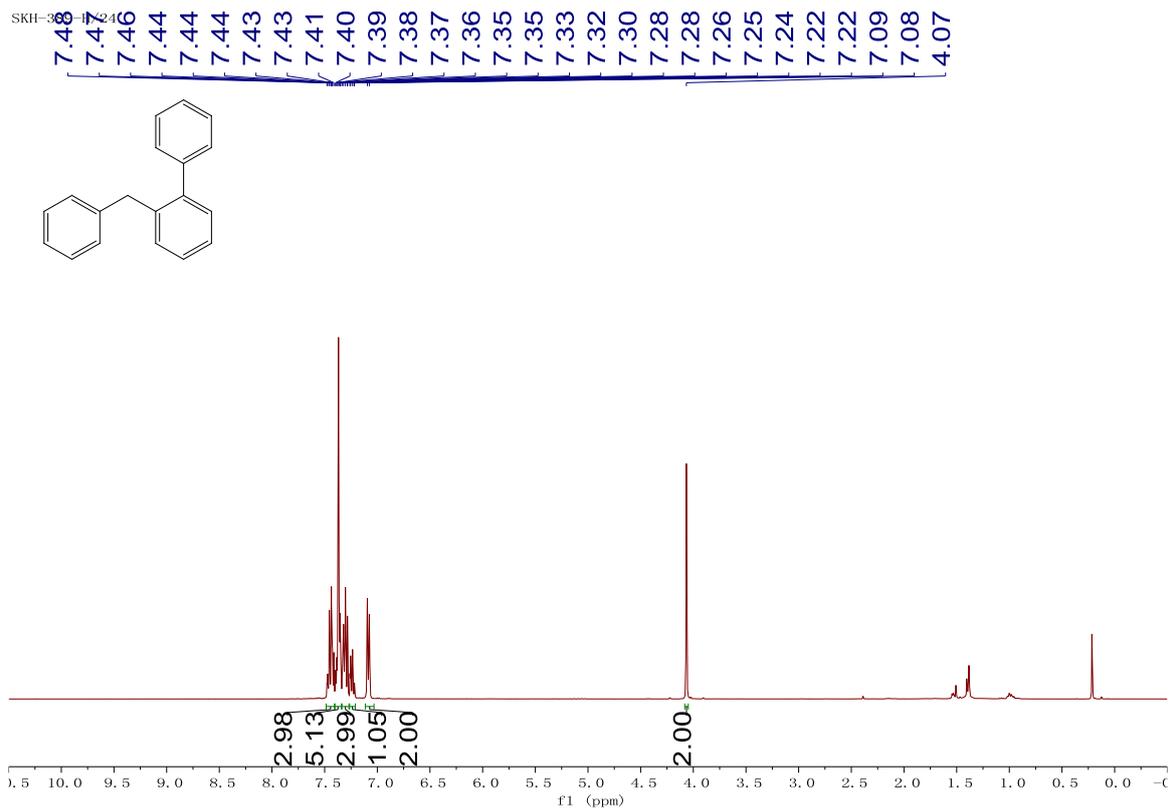
SKH-311-B-N1/2



2o

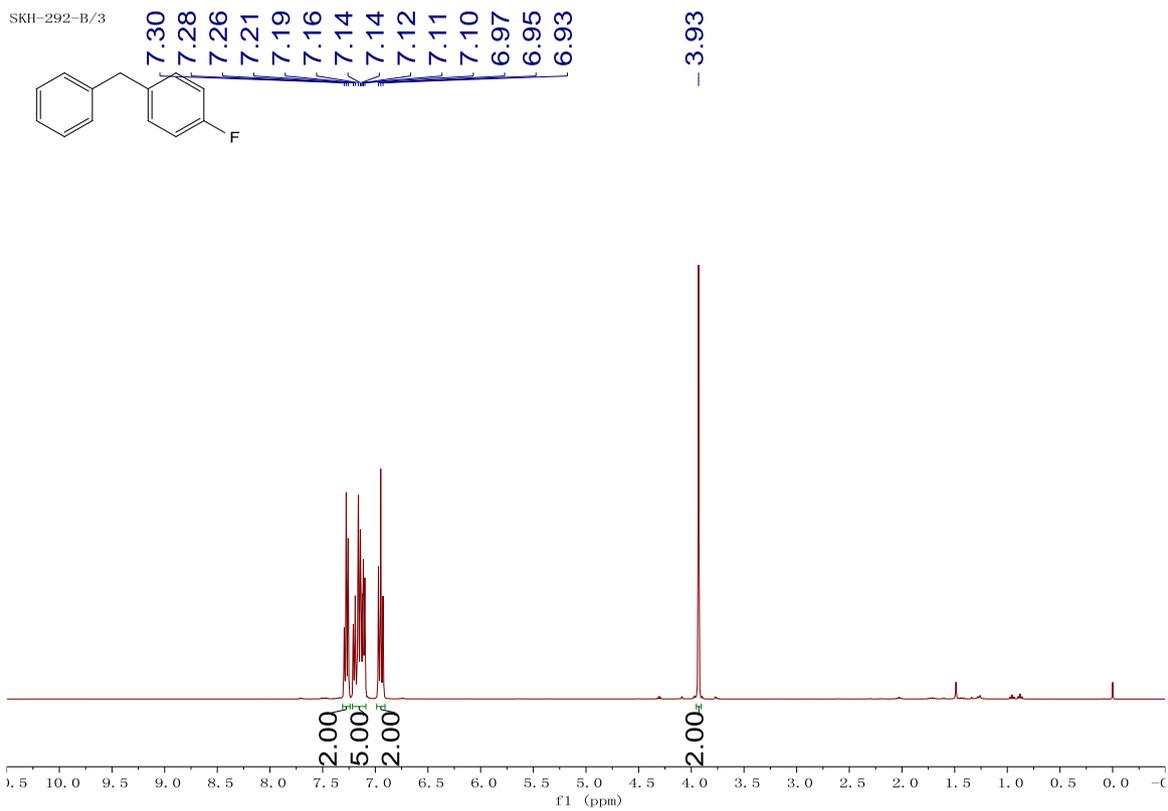


2p

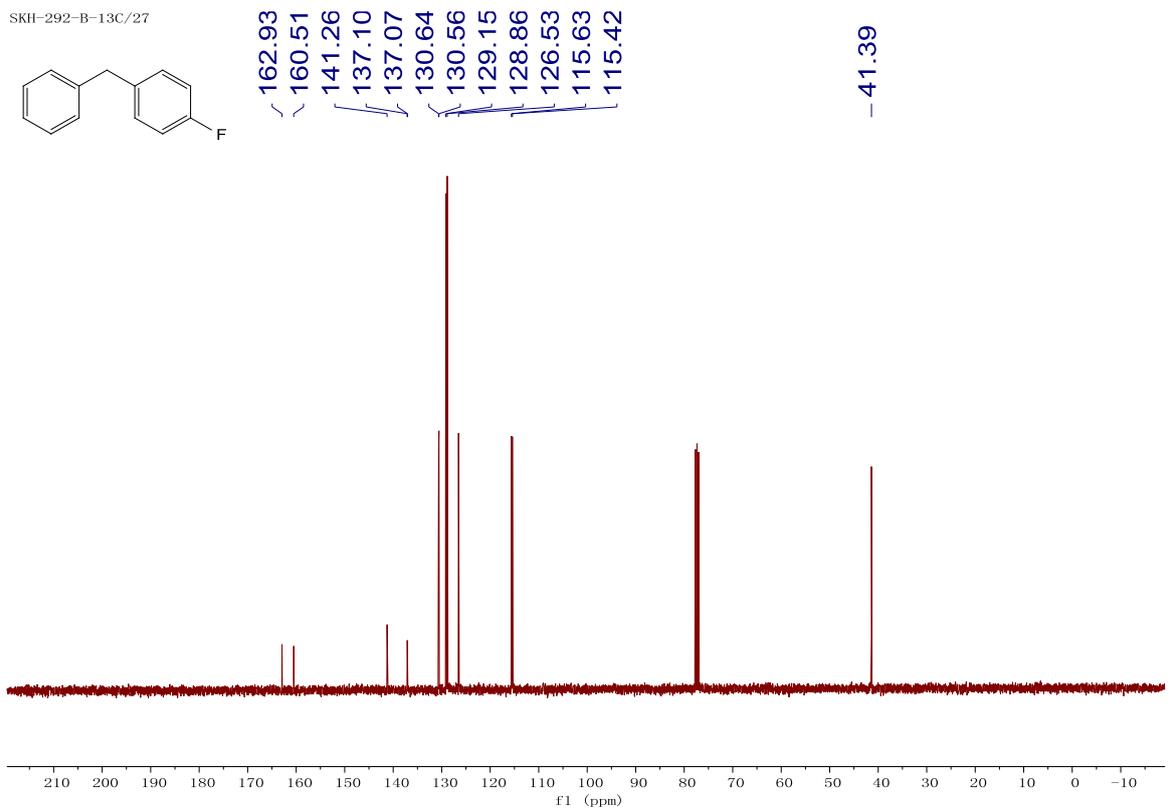


2q

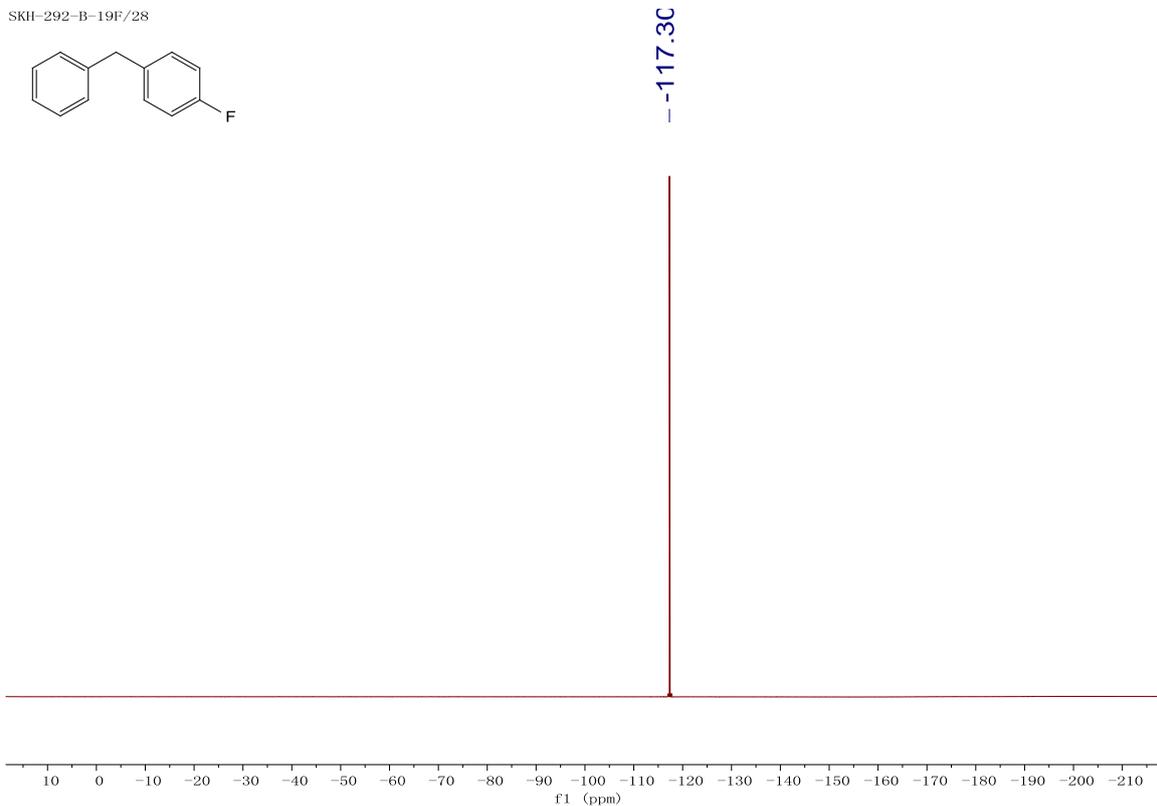
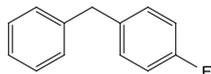
SKH-292-B/3



SKH-292-B-13C/27

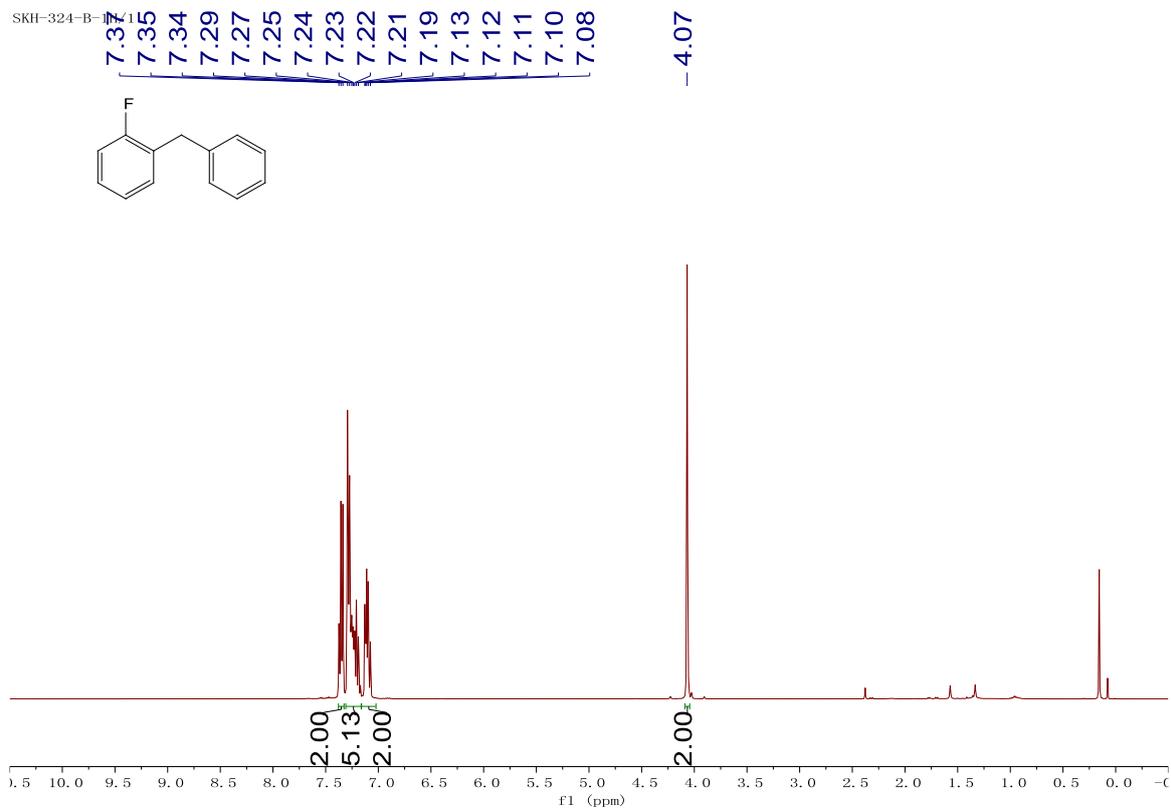
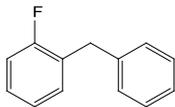


SKH-292-B-19F/28

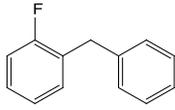


2r

SKH-324-B-1

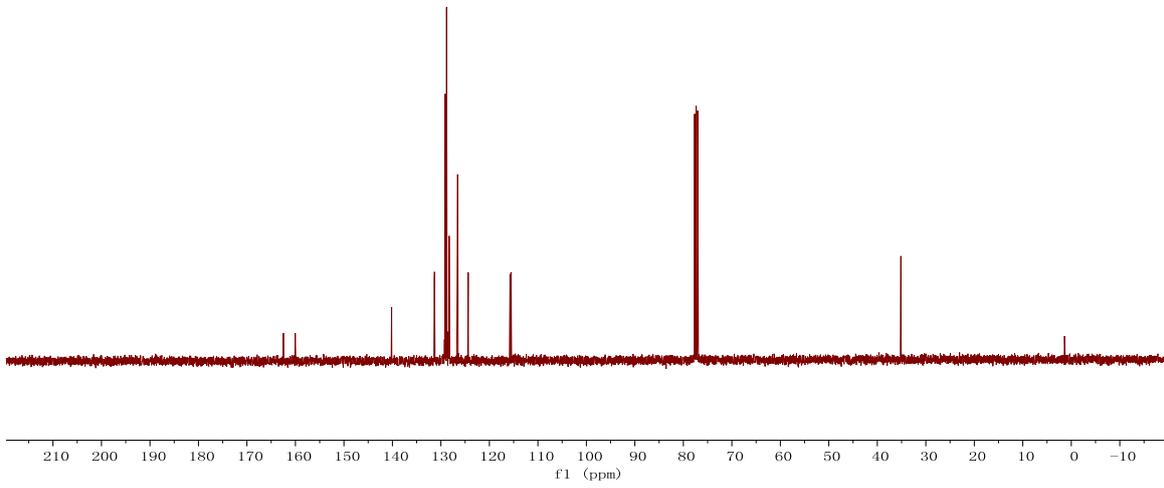


SKH-324-B-13C-20220222/4

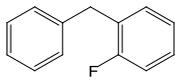


162.48
160.04
140.17
131.37
131.32
129.12
128.83
128.46
128.30
128.22
126.55
124.40
124.37
115.75
115.53

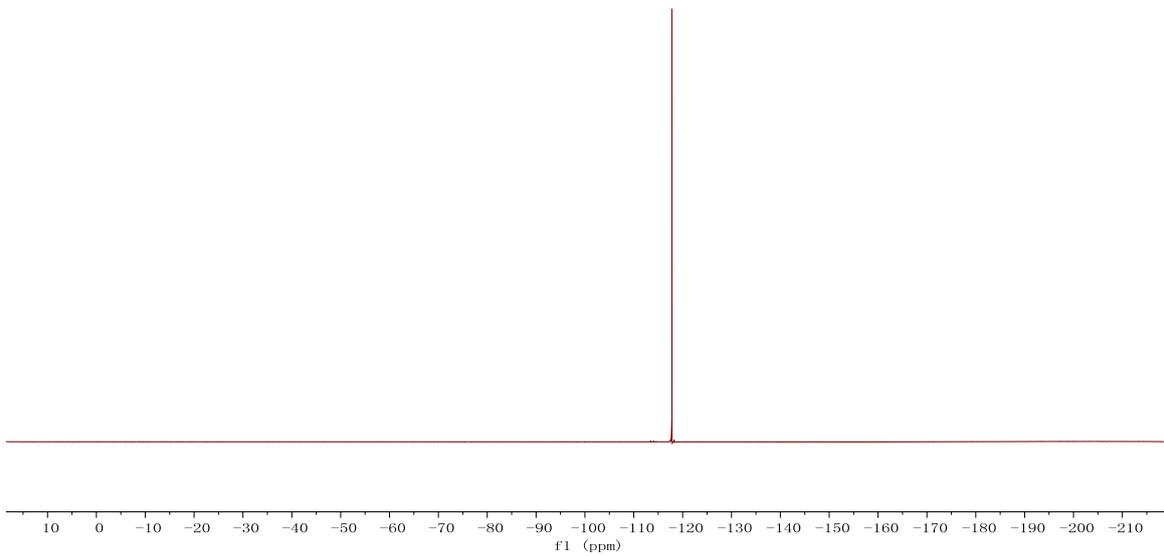
35.14
35.11



SKH-324-B-F/2

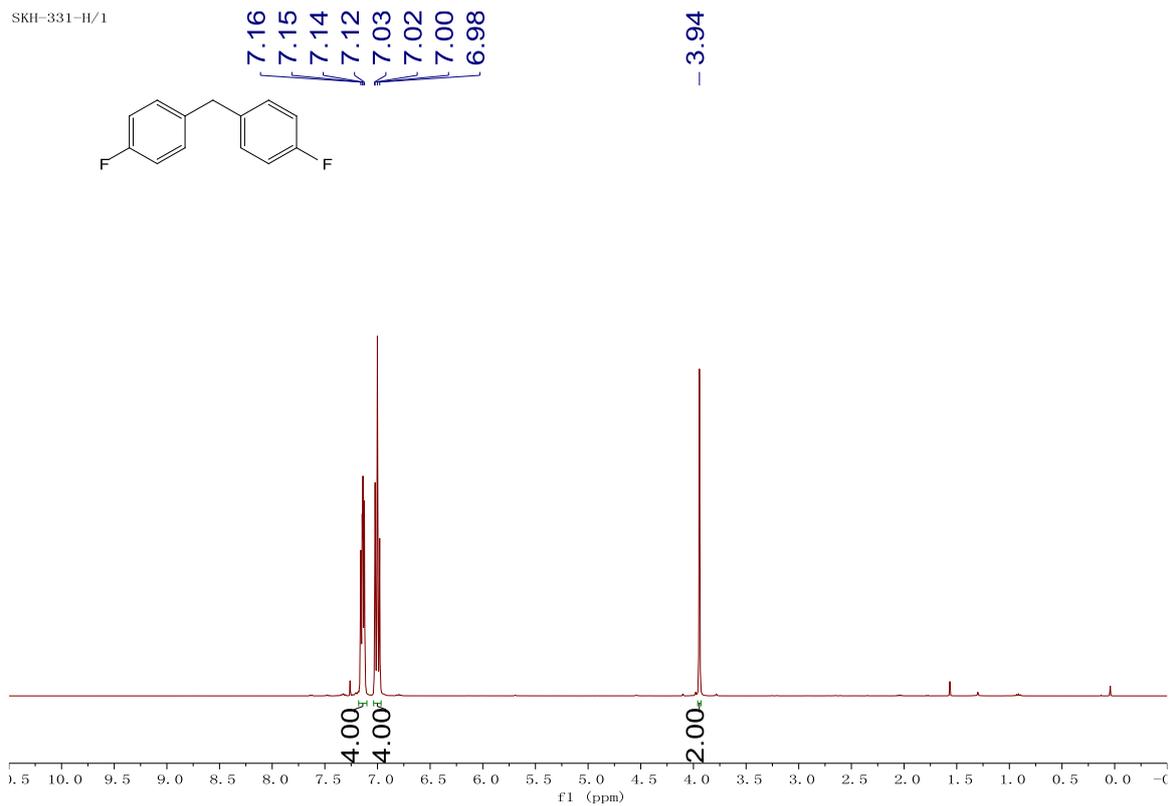


-117.8C

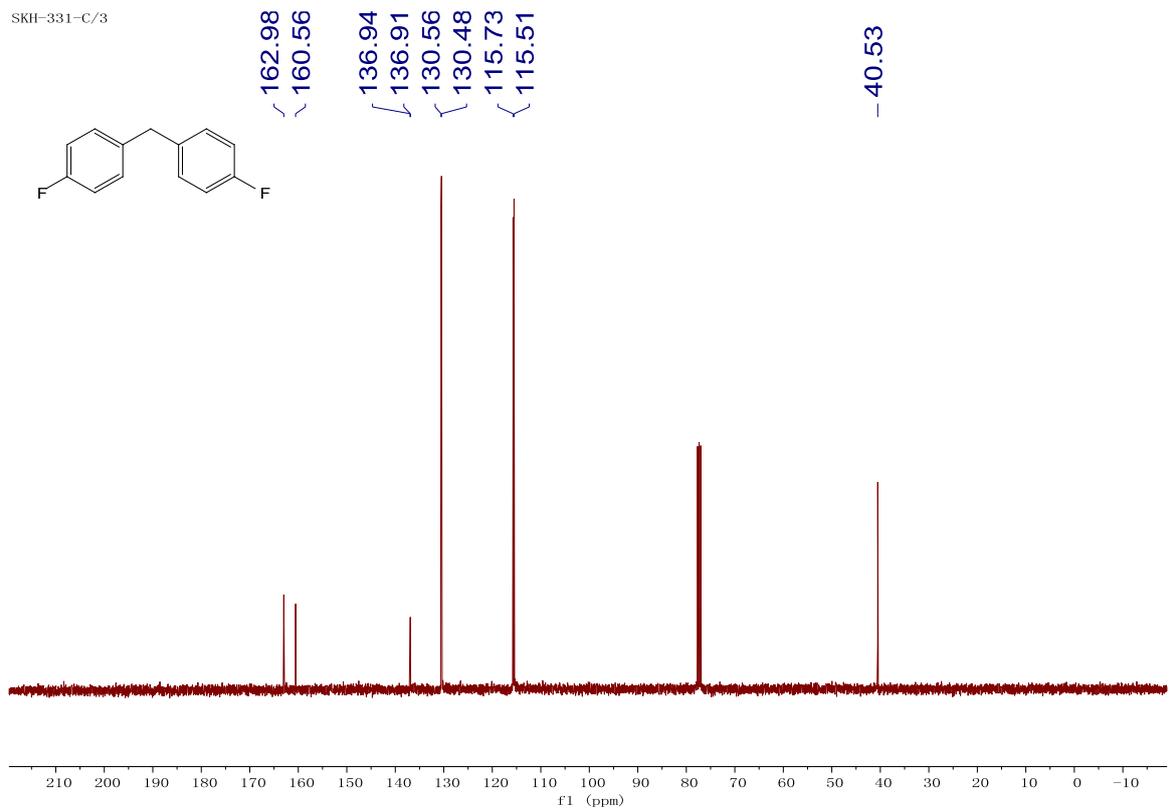


2s

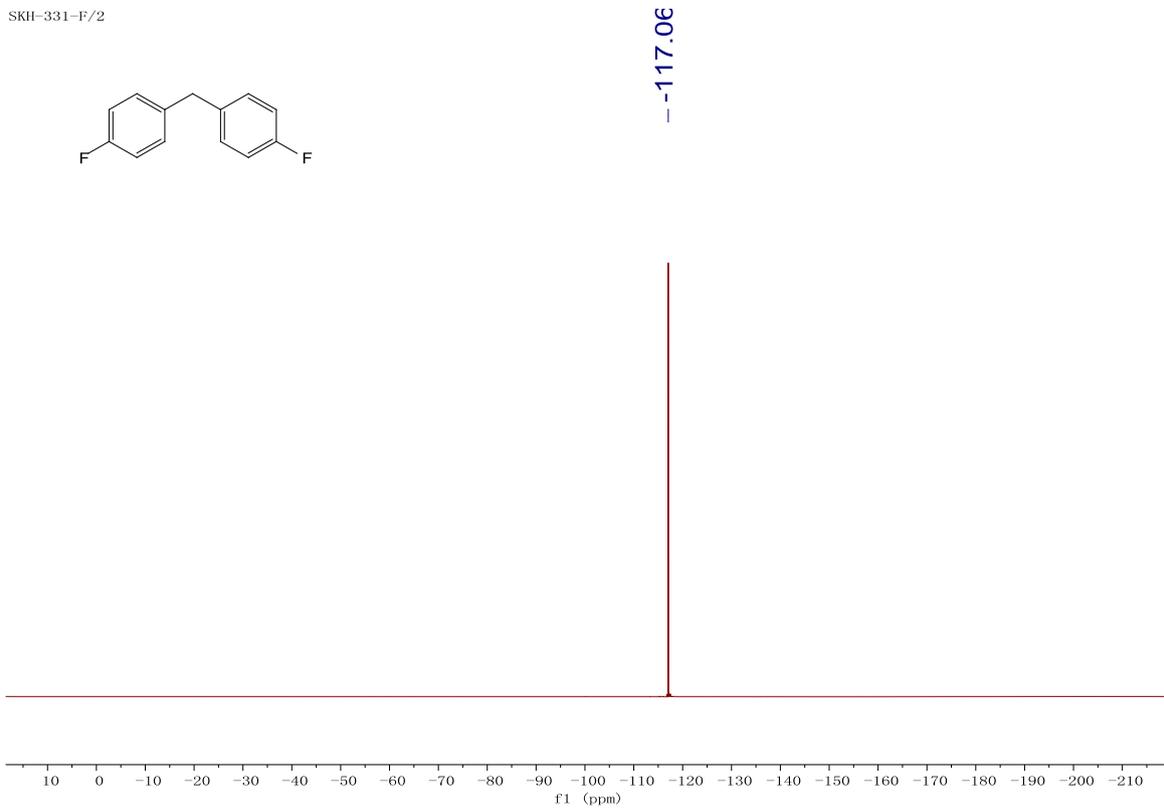
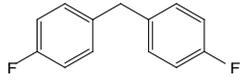
SKH-331-H/1



SKH-331-C/3

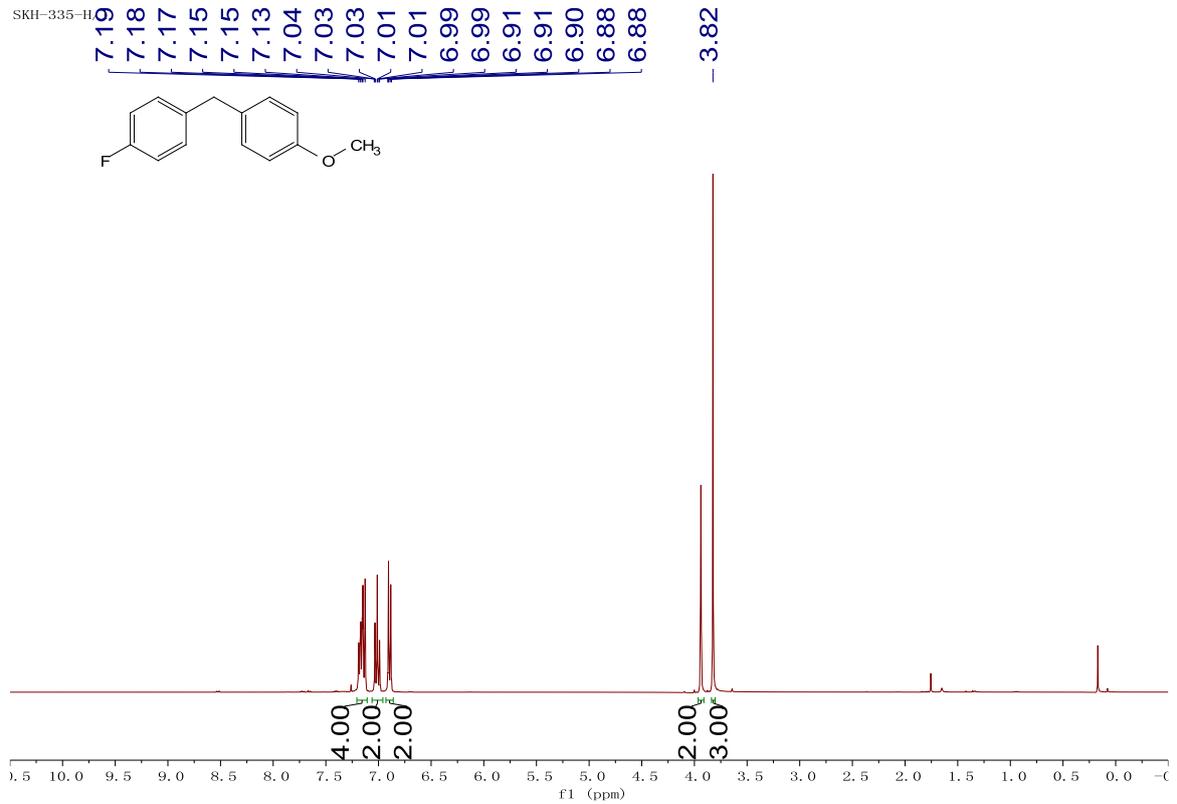
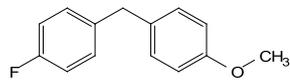


SKH-331-F/2

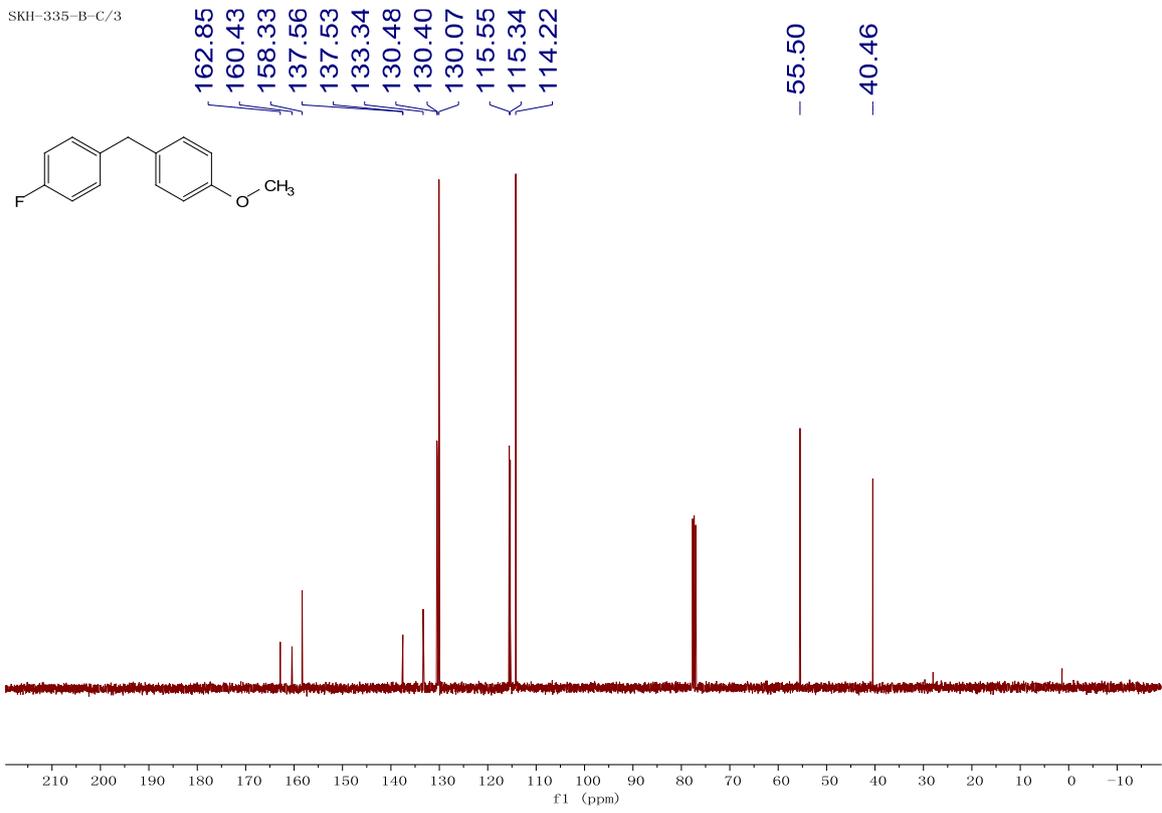


2t

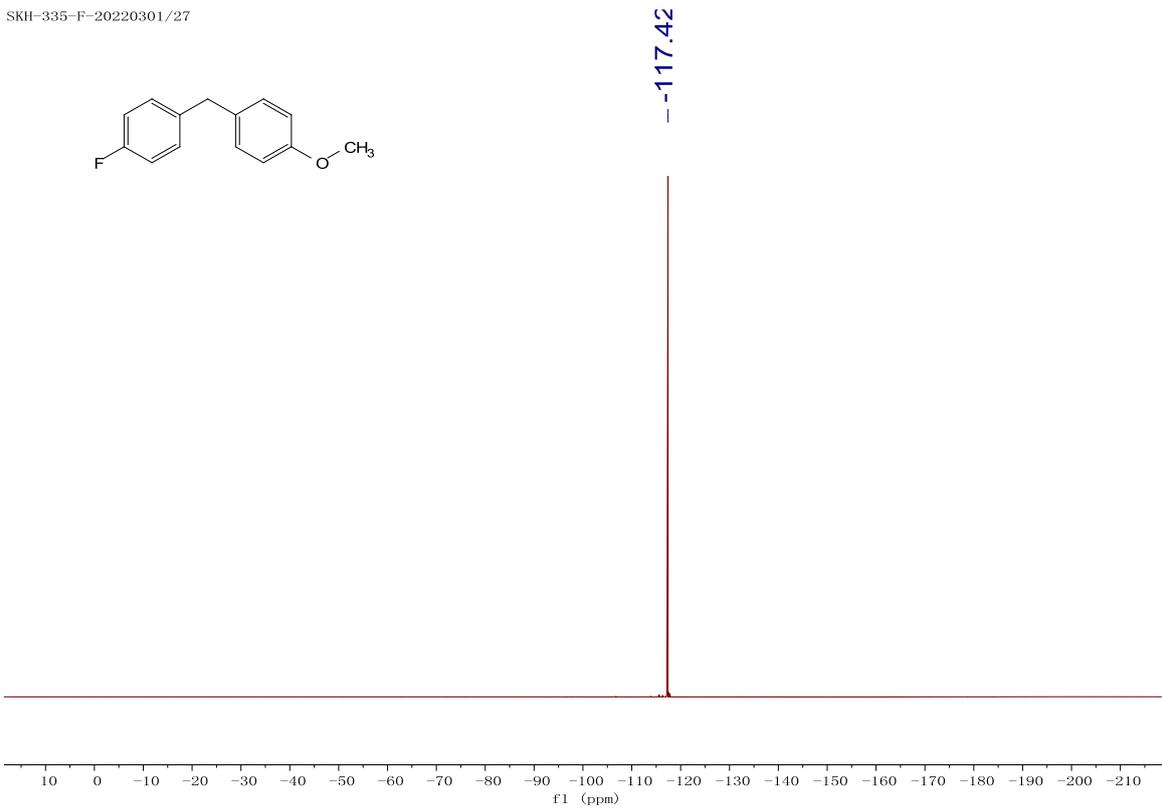
SKH-335-H



SKH-335-B-C/3

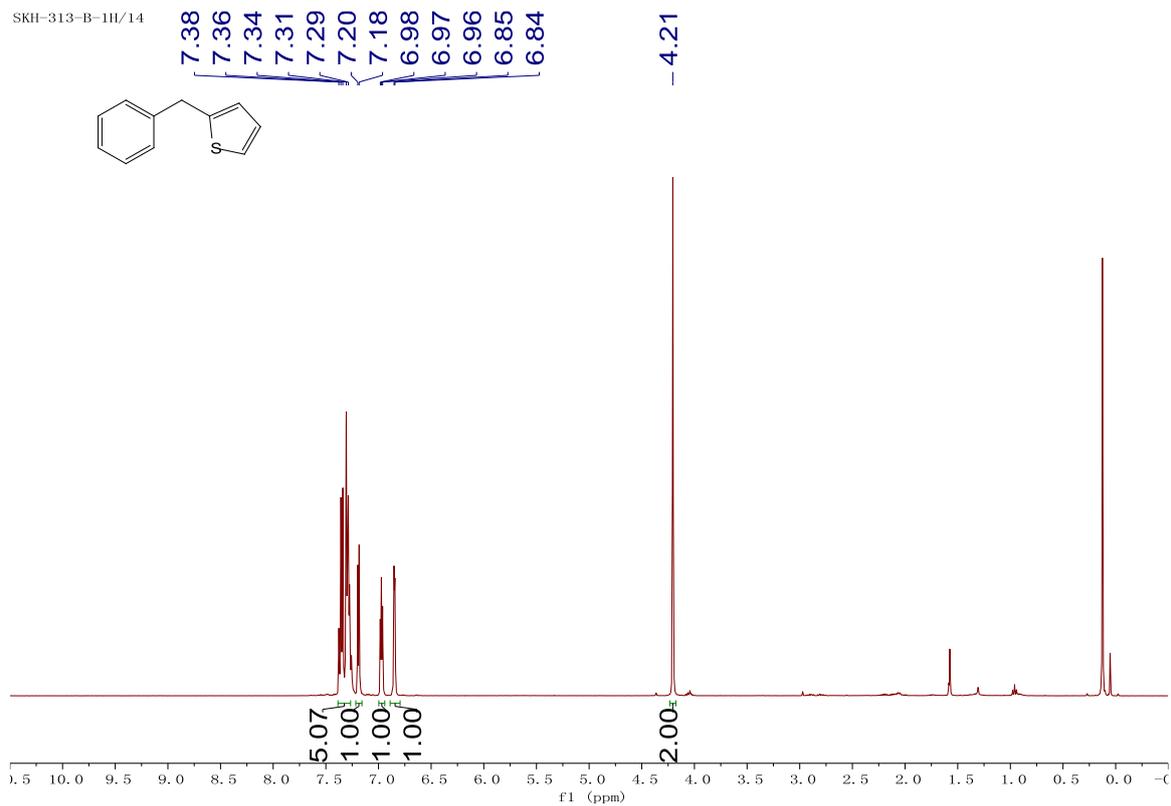
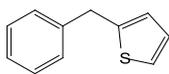


SKH-335-F-20220301/27

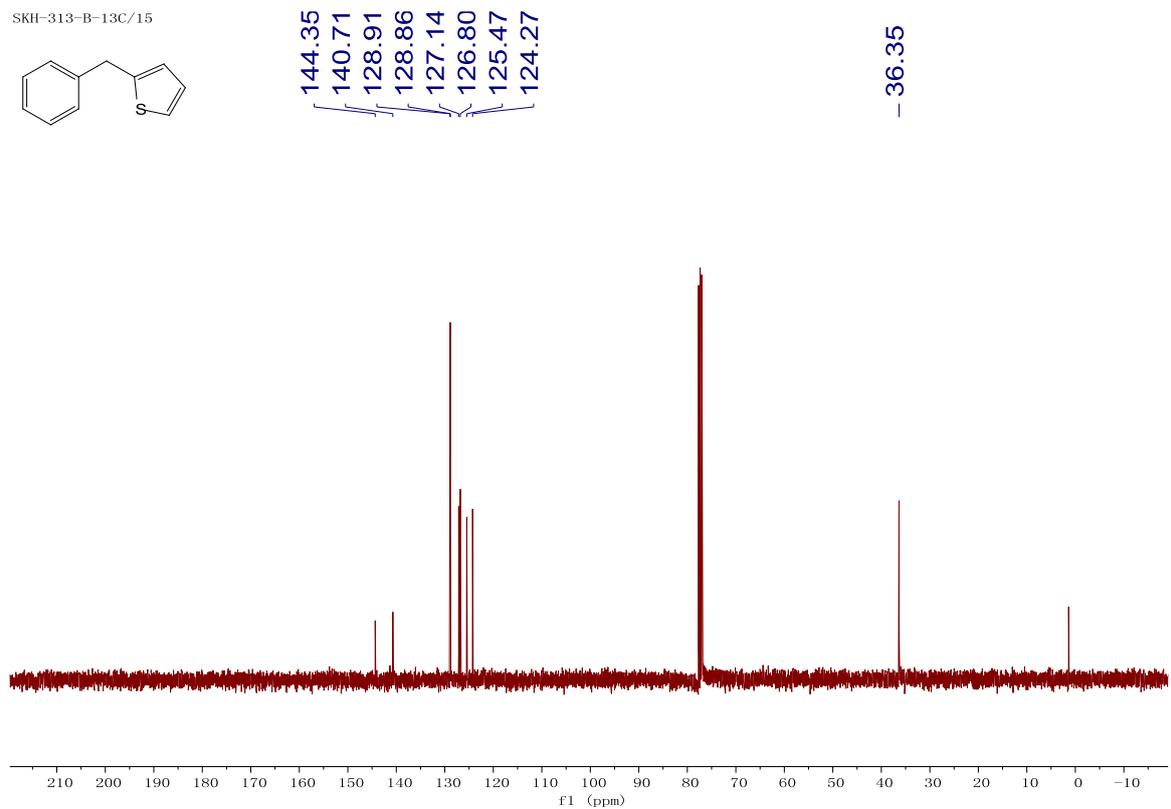
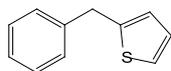


2u

SKH-313-B-1H/14

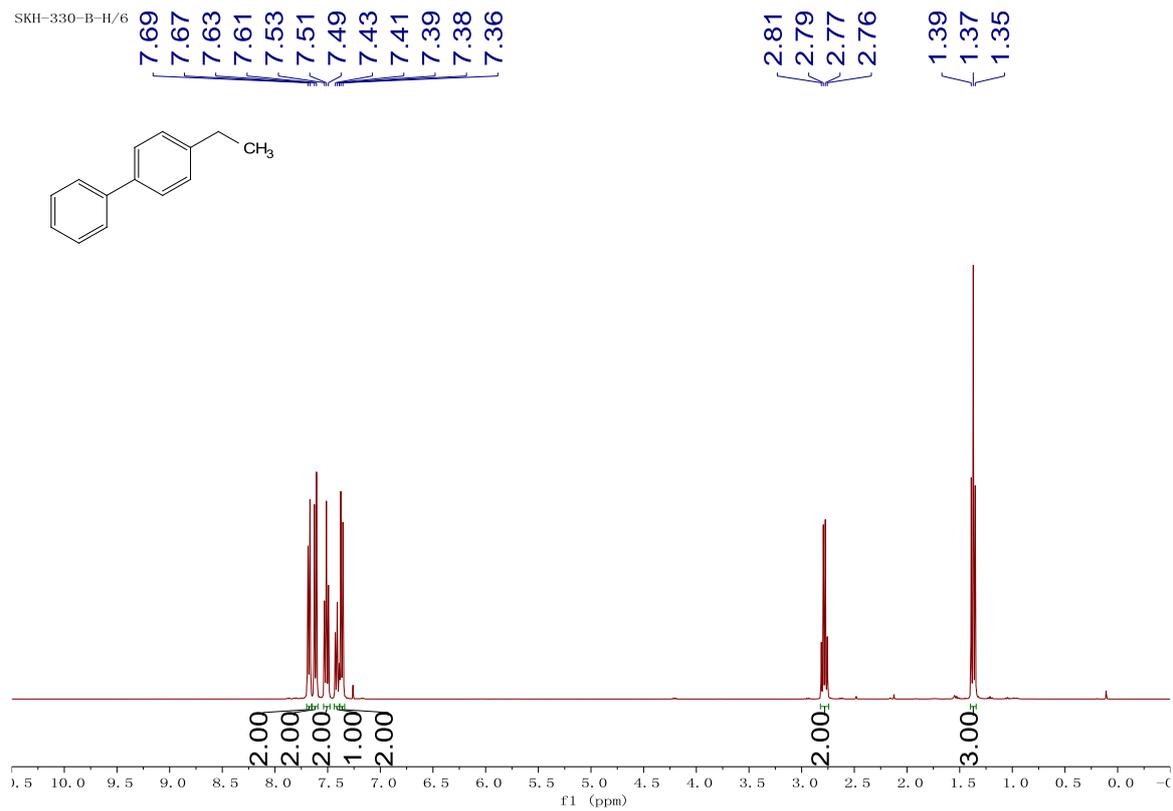
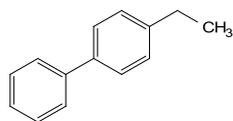


SKH-313-B-13C/15

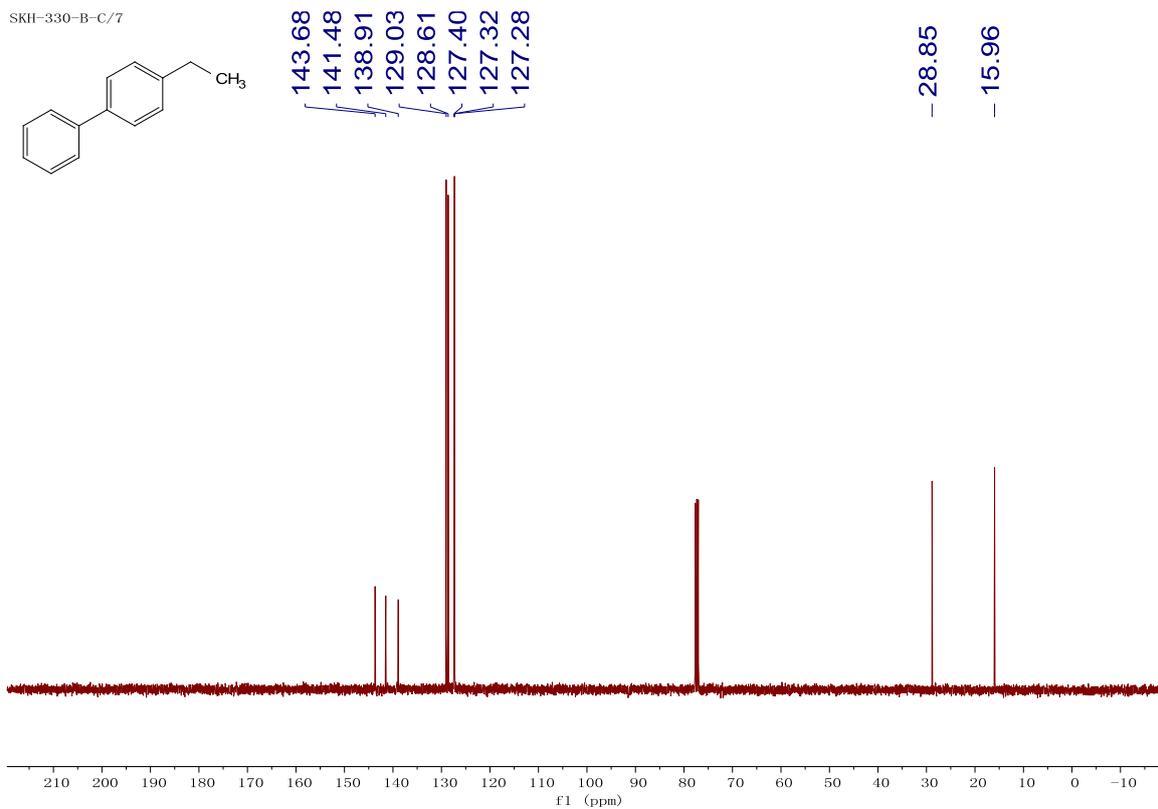
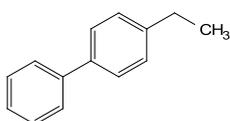


2v

SKH-330-B-H/6



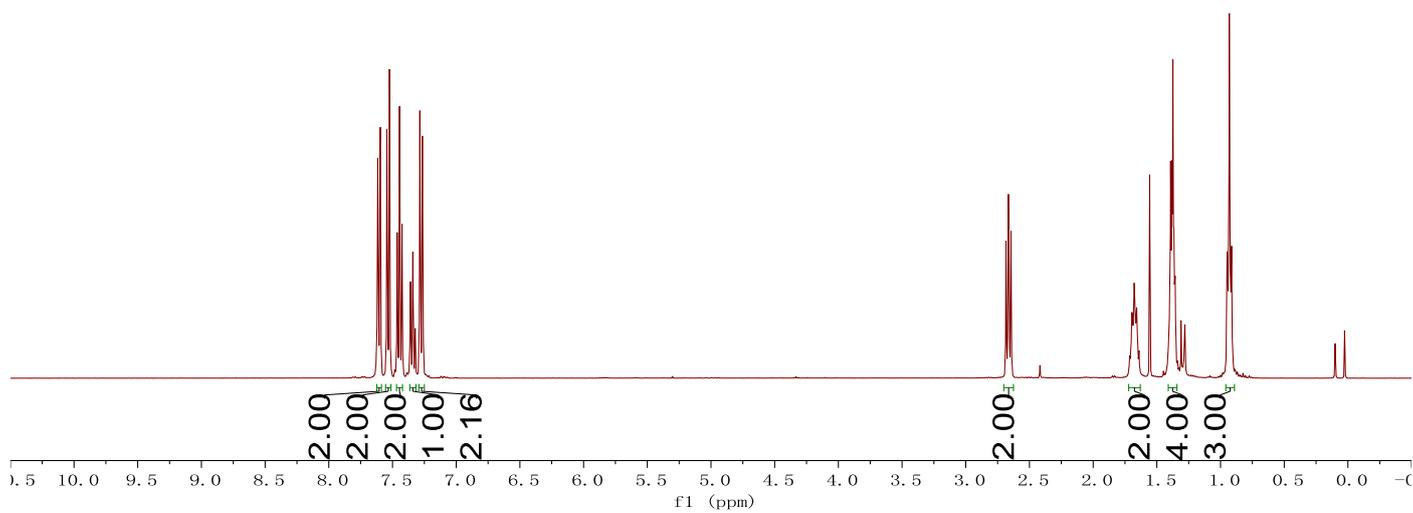
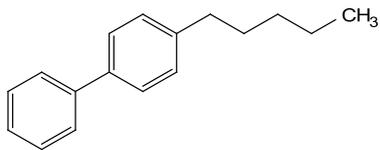
SKH-330-B-C/7



2w

桌面/10

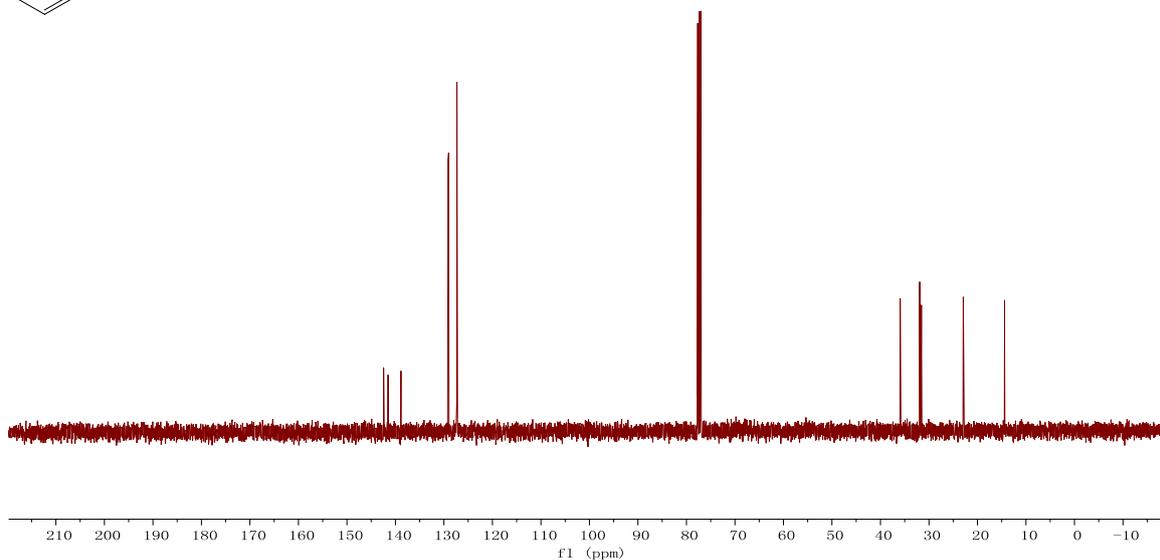
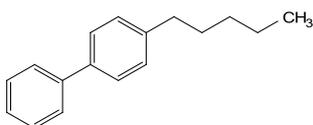
7.62 7.60 7.54 7.52 7.46 7.44 7.43 7.36 7.36 7.34 7.33 7.32 7.32 7.29 7.27 2.68 2.66 2.65 1.71 1.70 1.68 1.66 1.66 1.64 1.41 1.40 1.39 1.38 1.37 1.37 1.36 1.34 0.95 0.93 0.91



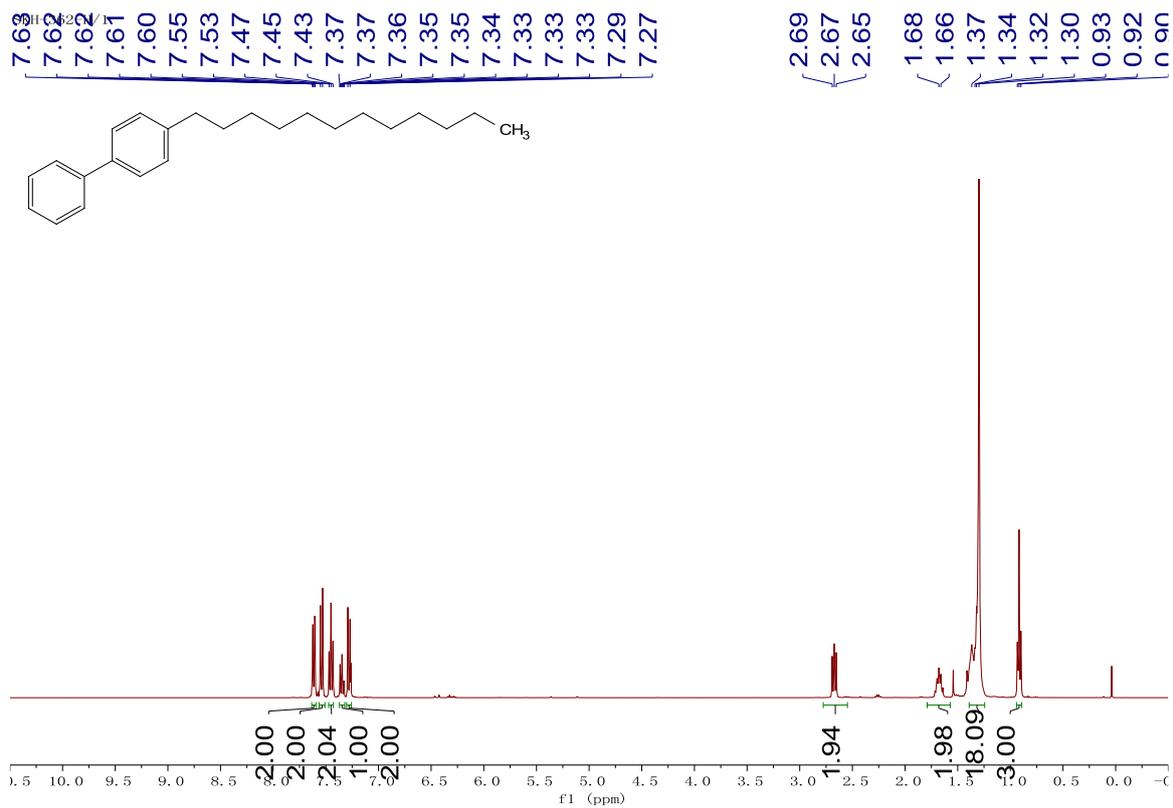
桌面/11

142.44 141.51 138.86 129.15 129.03 127.32 127.31 127.28

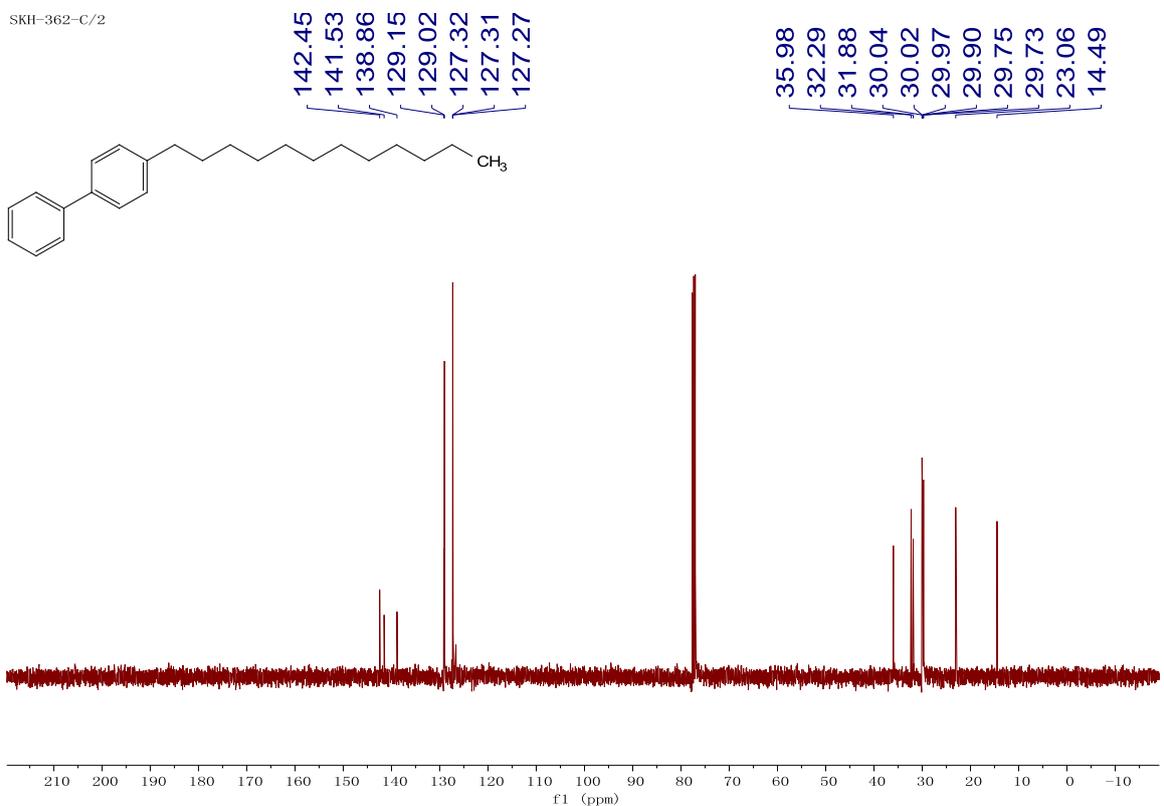
35.93 31.92 31.55 22.92 14.41



2x

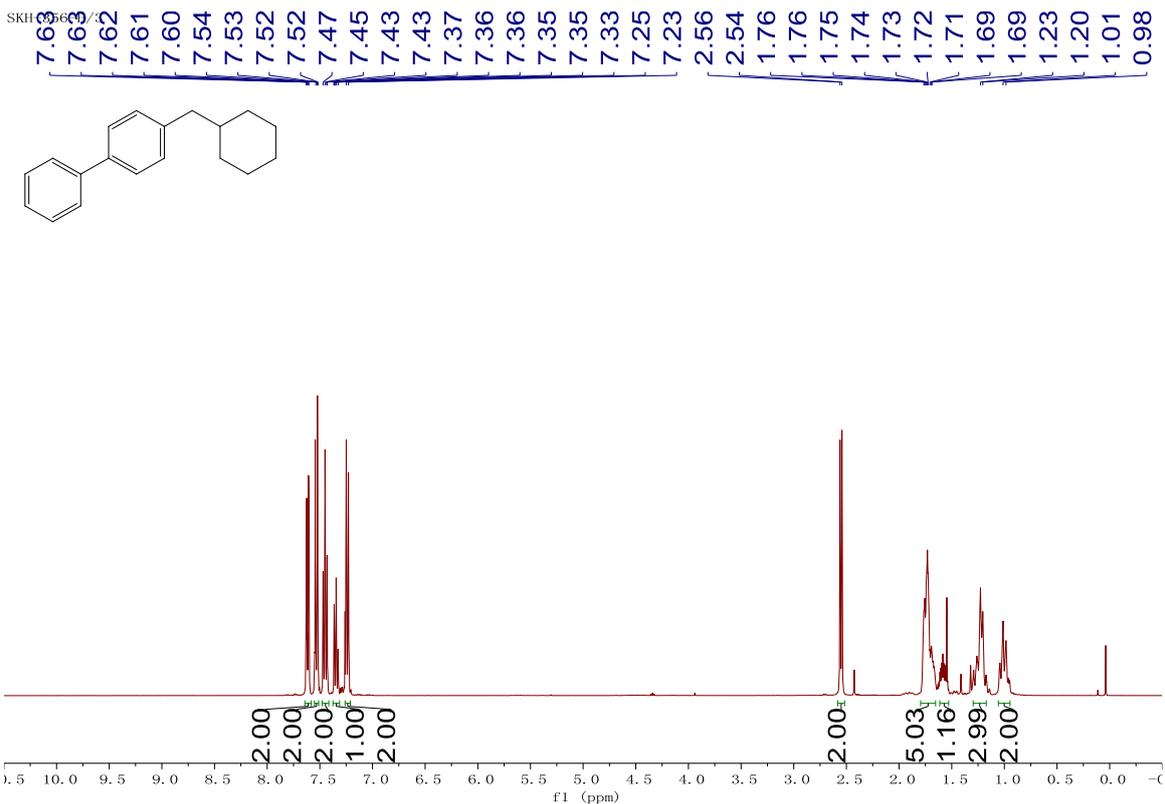


SKH-362-C/2

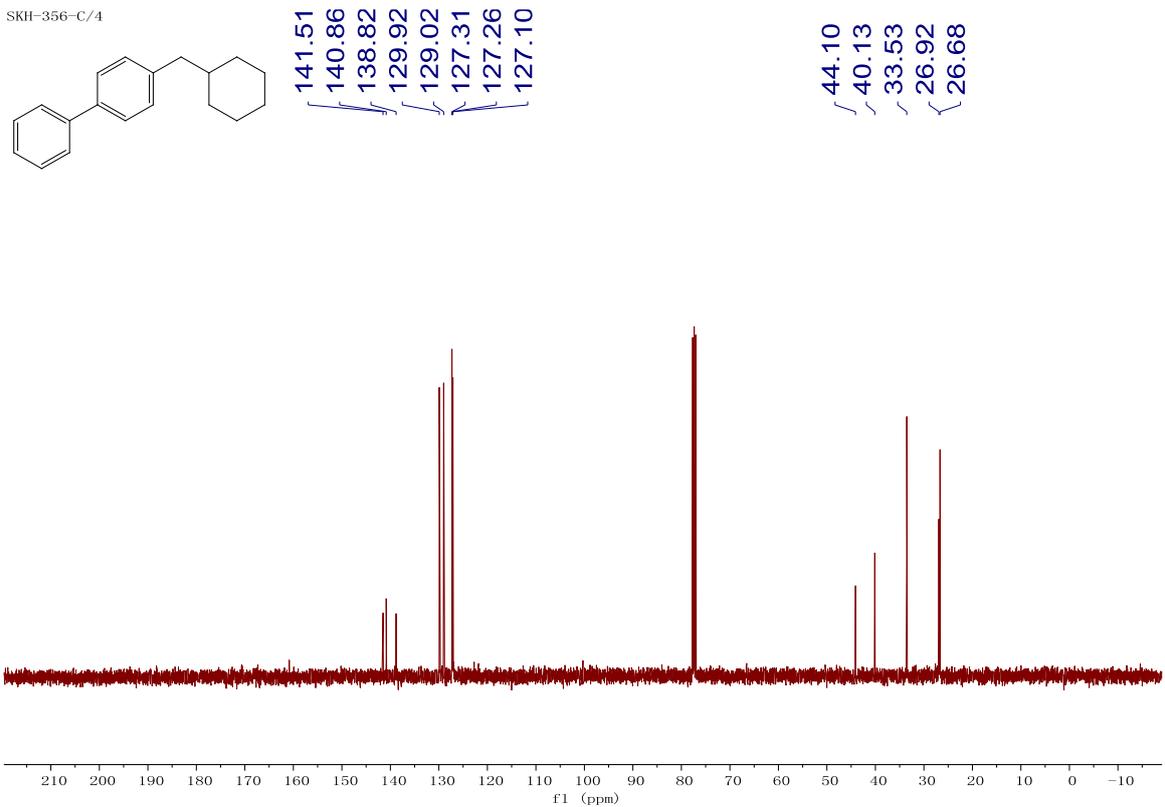


2y

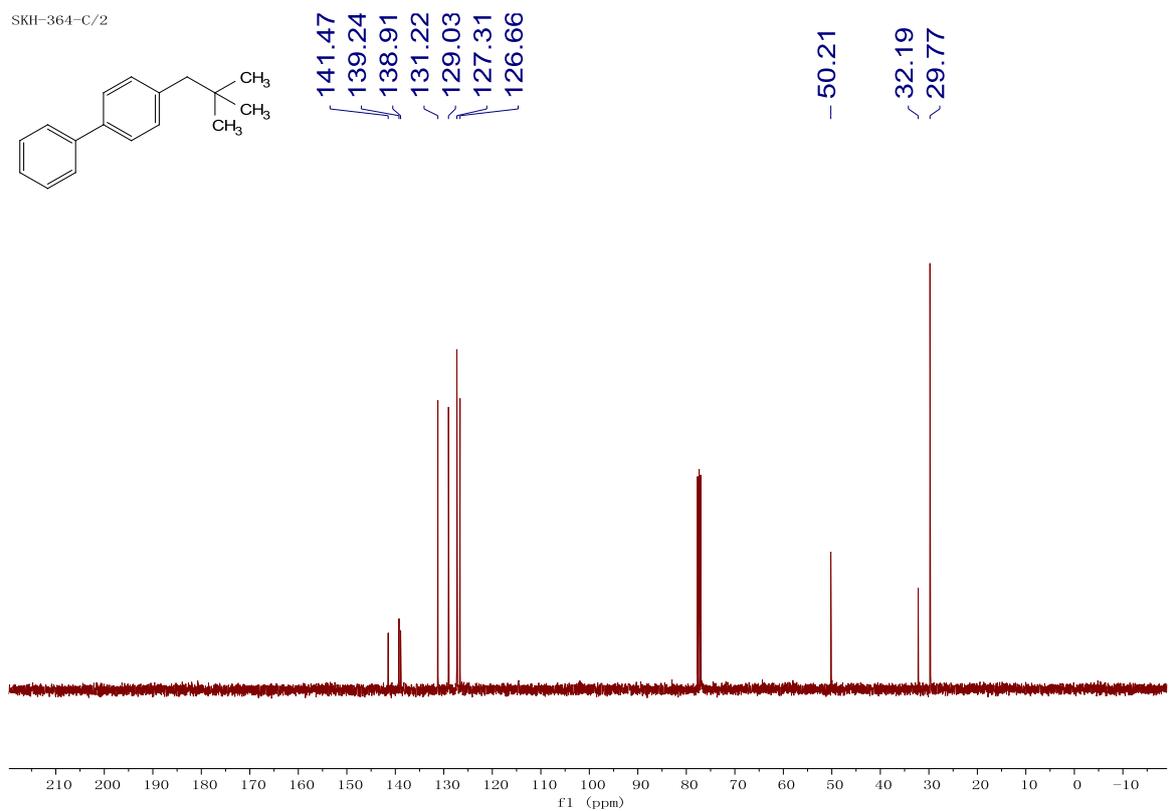
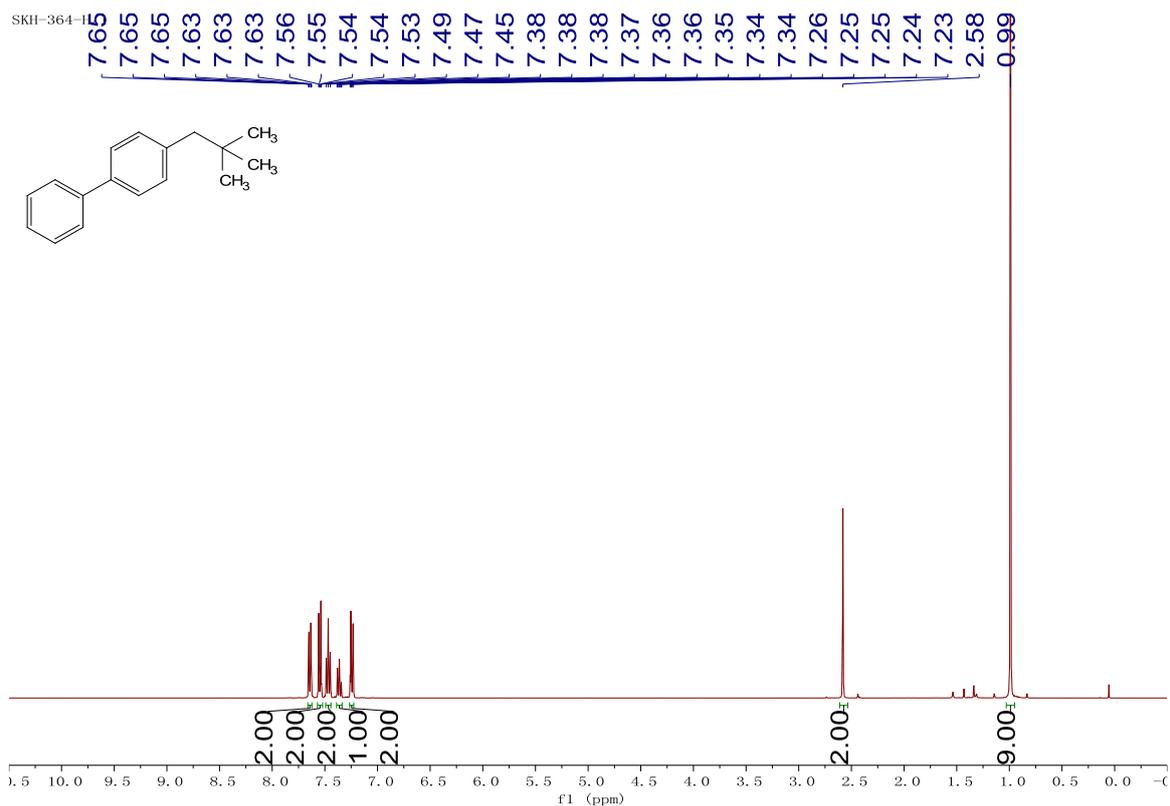
SKH-356-C/4



SKH-356-C/4

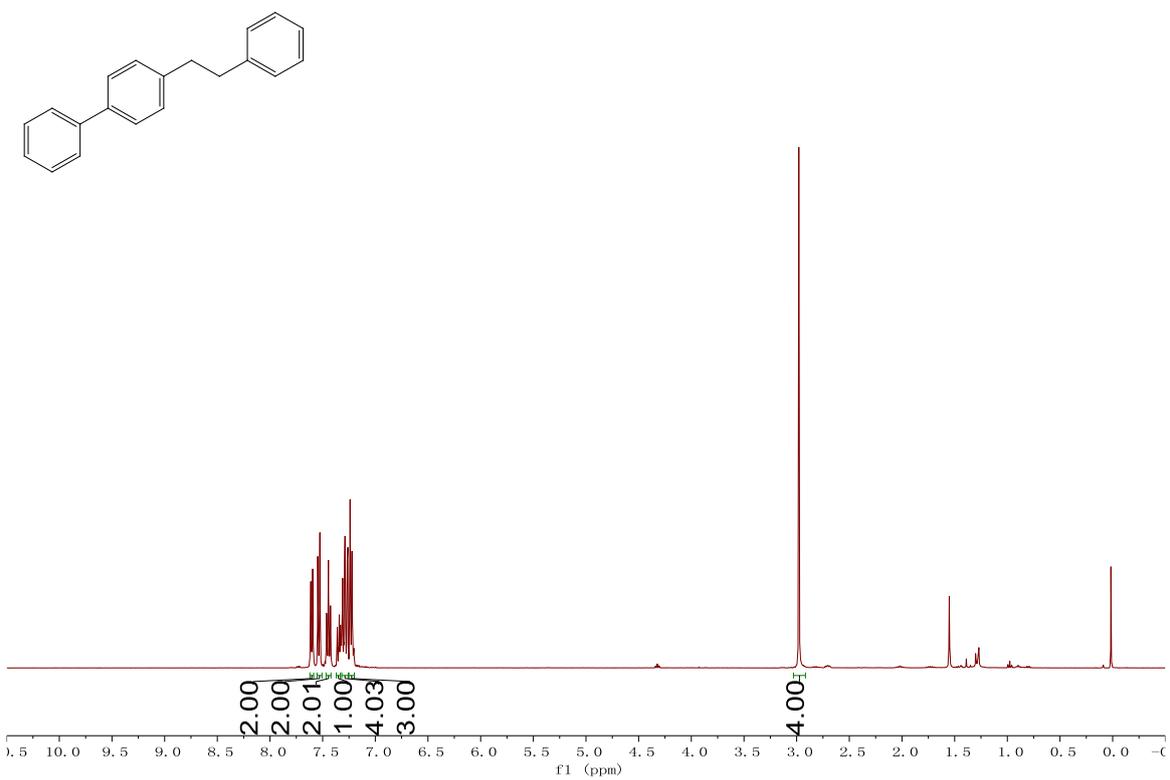


2z

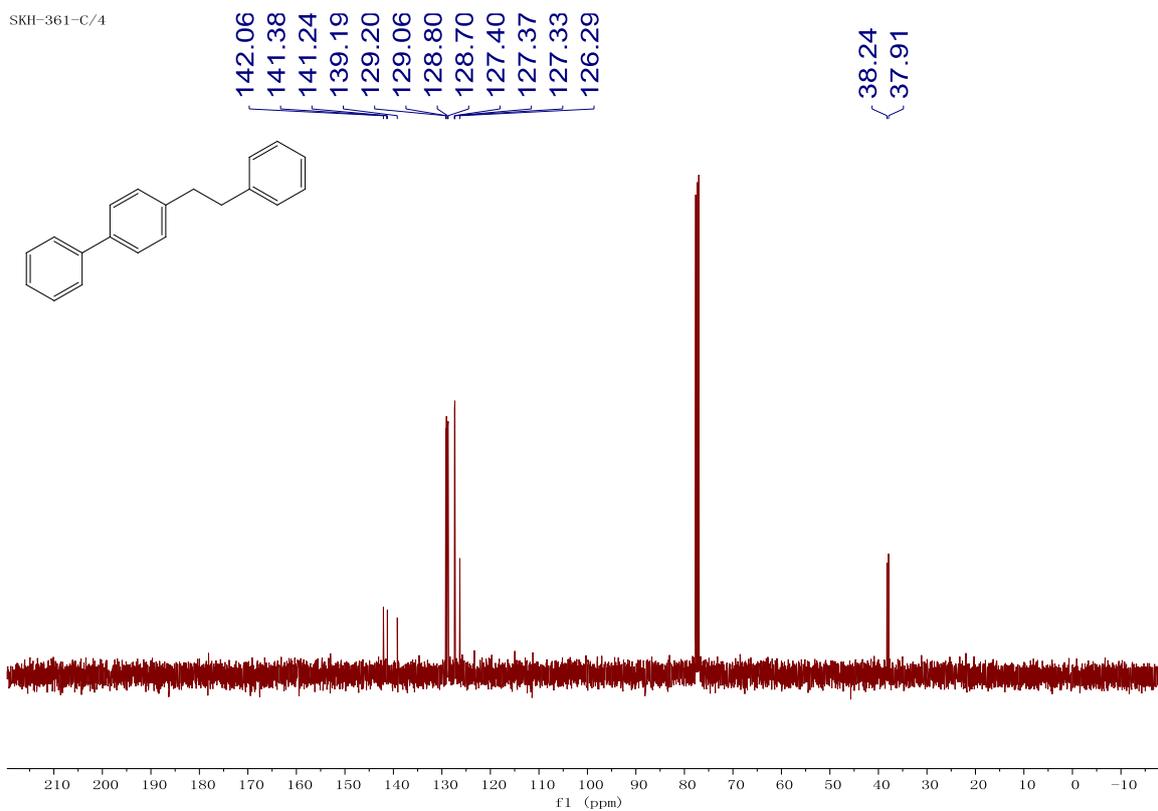


2aa

SKH-361-A-H/1

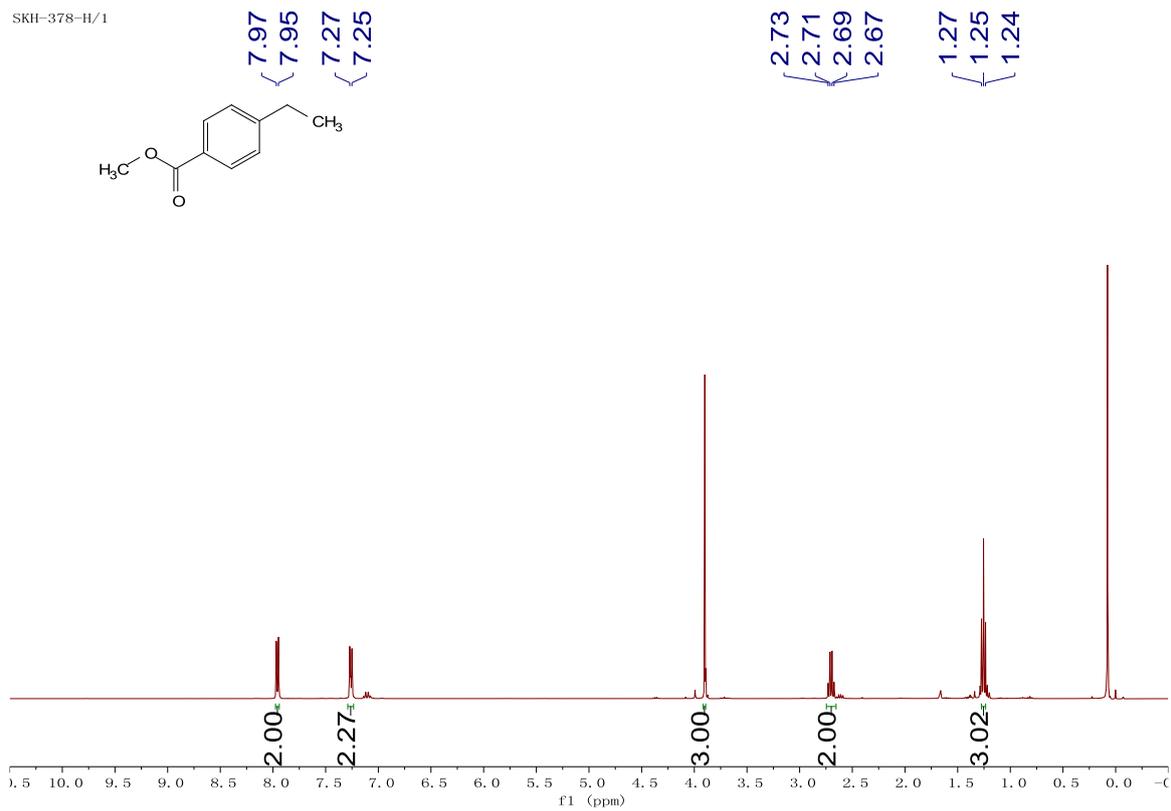
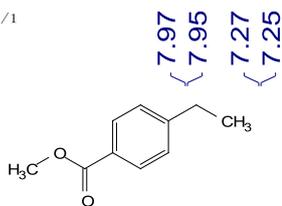


SKH-361-C/4



2ab

SKH-378-H/1



SKH-378-C/2

