Supplementary Information

Nickel-Catalyzed Barbier-Type Reaction of Carbonyl Derivatives with Unactivated Alkyl Halides

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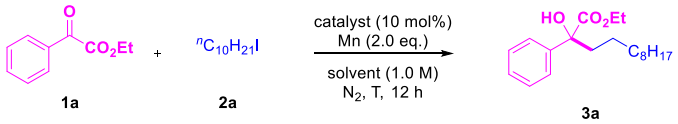
1. General Information

All reactions were carried out under nitrogen atmosphere in oven-dried (100 °C) glassware with magnetic stirring. All commercial reagents were used without additional purification, unless otherwise stated. Anhydrous solvents including DMF, DCM, MeCN, toluene, etc. were commercially available and used directly. Starting compounds (1 and 2) were purchased from Bide Chemical Co. Ltd., J&K Chemical Co. Ltd., Energy Chemical Co. Ltd., Adamas Chemical Co. Ltd. or Aladdin Chemical Co. Ltd. unless otherwise indicated. All new compounds were characterized by NMR spectroscopy, IR spectroscopy, high-resolution mass spectroscopy, and melting point (if solids). NMR spectra were recorded on an Agilent 400 MHz, Varian 400 MHz or Bruker 400 MHz spectrometers and were calibrated using residual solvent as an internal reference (CDCl3: 7.26 ppm for ^{1}H NMR and 77.16 ppm for ^{13}C NMR). Melting points were measured on a SGW X-4 apparatus. All IR spectra were taken on a BRUKER TENSOR 27 FT-IR spectrometer. ESI-HRMS spectra were obtained on a Thermo Fisher Scientific LTQ FT Ultra or an Agilent Technologies 6224 TOF LC/MS.

2. Reaction Optimization

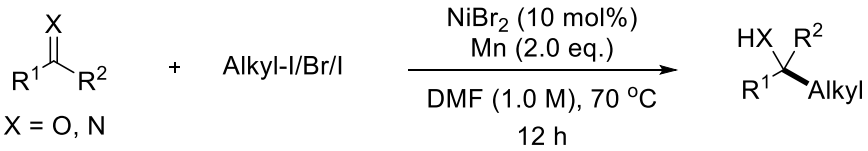


1a (0.2 mmol) **2a** (2.0 eq.) **3a**

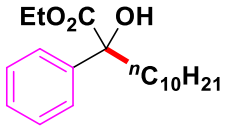
entry	catalyst	T (°C)	solvent	yield[a] (%)	entry	catalyst	T (°C)	solvent	yield[a] (%)
1	$NiBr_2$	70	DMF	76	14	$NiBr_2$	70	NMP	45
2	$Ni(cod)_2$	70	DMF	56	15	$NiBr_2$	70	MeCN	11
3	$Ni(acac)_2$	70	DMF	37	16	$NiBr_2$	70	DCM	<5
4	$Ni(OAc)_2{\cdot}4H_2O$	70	DMF	35	17	$NiBr_2$	70	toluene	<5
5	$NiBr_2{\cdot}bpy$	70	DMF	30	18	$NiBr_2$	60	DMF	64
6	CuCl	70	DMF	59	19	$NiBr_2$	50	DMF	58
7	$CoCl_2$	70	DMF	63	20	$NiBr_2$	40	DMF	<5
8	$Fe(OAc)_2$	70	DMF	65	21	$NiBr_2$	30	DMF	<5
9[b]	$NiBr_2$	70	DMF	60	22[e]	$NiBr_2$	70	DMF	56
10	-	70	DMF	38	23[f]	$NiBr_2$	70	DMF	60
11[c]	$NiBr_2$	70	DMF	62	24[g]	$NiBr_2$	70	DMF	64
12[d]	$NiBr_2$	70	DMF	<20	25[h]	$NiBr_2$	70	DMF	64
13	$NiBr_2$	70	DMA	62					

[a]Isolated yield; [b]Using Zn instead of Mn; [c]6 h; [d]1 h; [e]0.67 M in DMF; [f]0.4 M in DMF; [g]0.2 M in DMF; [h]4.0 eq. of **2a** and Mn, 0.25 M in DMF

3. General Procedure

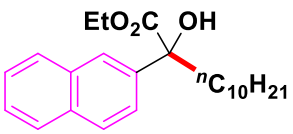


General Procedure (GP): In a nitrogen-filled glove box, $NiBr_2$ (8.7 mg, 0.04 mmol), ketone or imine (if solid) (0.4 mmol), manganese powder (43.9 mg, 0.8 mmol) and DMF (0.4 mL) were added to a 4 mL vial equipped with a magnetic stirrer bar. Ketone or imine (if liquid) (0.4 mmol) and alkyl halide (0.8 mmol) were then charged to the vial. The sealed reaction vial was then removed from the glove box, and the reaction mixture was stirred at 70 °C for 12 h. The resulting mixture was quenched with saturated NH_4Cl, extracted three times with diethyl ether and filtered through a short pad of silica gel, eluted with ethyl acetate, and concentrated in vacuo. The crude product was purified by flash column chromatography (hexanes/EtOAc) on silica gel to give the corresponding product.



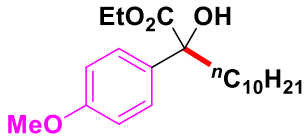
ethyl 2-hydroxy-2-phenyldodecanoate (3a)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 76%. Colorless oil (97.3 mg). The product was obtained in 58% yield (74.2 mg) when using 1-bromodecane as substrate. **IR (neat, cm^{-1})** 3514, 2922, 2855, 1724, 1455, 1238, 657. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.68 – 7.59 (m, 2H), 7.43 – 7.34 (m, 2H), 7.33 – 7.27 (m, 1H), 4.37 – 4.18 (m, 2H), 3.83 (s, 1H), 2.30 – 2.15 (m, 1H), 2.10 – 1.95 (m, 1H), 1.55 – 1.40 (m, 1H), 1.38 – 1.22 (m, 18H), 0.91 (t, J = 6.8 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.5, 142.2, 128.2, 127.6, 125.6, 78.4, 62.4, 39.8, 32.0, 29.7, 29.7, 29.6, 29.5, 29.4, 23.7, 22.8, 14.2, 14.1. **HRMS (ESI)** calculated for $C_{20}H_{32}O_3Na$ $[M+Na]^+$ *m/z* 343.2244, found 343.2237.



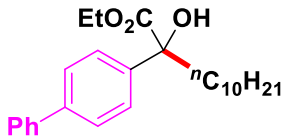
ethyl 2-hydroxy-2-(naphthalen-2-yl)dodecanoate (3b)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 82%. Yellow oil (121.3 mg). **IR (neat, cm^{-1})** 3510, 2921, 2854, 1723, 1458, 1232, 748. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.12 (d, *J* = 1.9 Hz, 1H), 7.91 – 7.80 (m, 3H), 7.72 (dd, *J* = 8.7, 1.9 Hz, 1H), 7.52 – 7.45 (m, 2H), 4.37 – 4.18 (m, 2H), 3.94 (s, 1H), 2.38 – 2.23 (m, 1H), 2.19 – 2.07 (m, 1H), 1.52 – 1.41 (m, 1H), 1.39 – 1.21 (m, 18H), 0.89 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 175.5, 139.5, 133.1, 132.8, 128.4, 127.9, 127.5, 126.2, 124.6, 123.9, 78.5, 62.6, 39.7, 32.0, 29.8, 29.7, 29.6, 29.5, 29.4, 23.7, 22.8, 14.2. **HRMS (ESI)** calculated for $C_{24}H_{34}O_3Na$ $[M+Na]^+$ *m/z* 393.2400, found 393.2407.



ethyl 2-hydroxy-2-(4-methoxyphenyl)dodecanoate (3c)

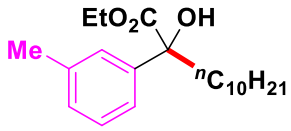
The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 72%. Light yellow oil (100.8 mg). **IR (neat, cm^{-1})** 3513, 2922, 2853, 1723, 1508, 1241, 1029, 830. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.60 – 7.45 (m, 2H), 6.97 – 6.81 (m, 2H), 4.36 – 4.12 (m, 2H), 3.79 (s, 3H), 3.76 (s, 1H), 2.22 – 2.08 (m, 1H), 2.03 – 1.87 (m, 1H), 1.51 – 1.37 (m, 1H), 1.34 – 1.19 (m, 18H), 0.87 (t, *J* = 6.6 Hz, 3H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 175.7, 159.1, 134.3, 126.8, 113.5, 78.0, 62.3, 55.3, 39.8, 32.0, 29.7, 29.6, 29.6, 29.5, 29.4, 23.7, 22.7, 14.2, 14.2. **HRMS (ESI)** calculated for $C_{21}H_{34}O_4Na$ $[M+Na]^+$ *m/z* 373.2349, found 373.2345.



ethyl 2-([1,1'-biphenyl]-4-yl)-2-hydroxydodecanoate (3d)

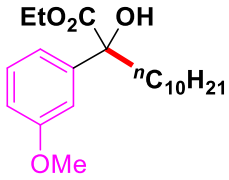
The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 67%. White solid (53.3 mg). **IR (neat, cm^{-1})** 3539, 2915, 2851, 1716, 1478, 1241, 1014, 689, 646. **Melting point**: 35-37 ℃. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.73 – 7.68 (m, 2H), 7.65 – 7.57 (m, 4H), 7.49 – 7.42 (m, 2H), 7.39 – 7.33 (m, 1H), 4.39 – 4.18 (m, 2H), 3.86 (s, 1H), 2.31 – 2.16 (m, 1H), 2.12 – 1.98 (m, 1H), 1.56 – 1.43 (m, 1H), 1.40 – 1.23 (m, 18H), 0.90 (t, *J* = 6.7 Hz, 3H). **^{13}C NMR (101 MHz,**

$CDCl_3$) δ 175.5, 141.3, 140.7, 140.5, 128.8, 127.4, 127.1, 127.0, 126.1, 78.3, 62.5, 39.9, 32.0, 29.8, 29.7, 29.6, 29.5, 29.4, 23.7, 22.8, 14.2. **HRMS (ESI)** calculated for $C_{26}H_{36}O_3Na$ $[M+Na]^+$ *m/z* 419.2557, found 419.2554.



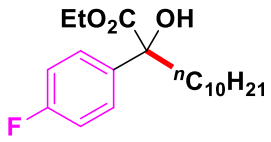
ethyl 2-hydroxy-2-(m-tolyl)dodecanoate (3e)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 73%. Colorless oil (94 mg). **IR (neat, cm^{-1})** 3515, 2922, 2855, 1723, 1457, 1227, 1091, 699. **^1H NMR (400 MHz, $CDCl_3$)** δ 7.50 – 7.41 (m, 2H), 7.27 (t, *J* = 7.6 Hz, 1H), 7.13 (d, *J* = 7.5 Hz, 1H), 4.40 – 4.17 (m, 2H), 3.84 (s, 1H), 2.40 (s, 3H), 2.28 – 2.15 (m, 1H), 2.09 – 1.96 (m, 1H), 1.58 – 1.43 (m, 1H), 1.41 – 1.26 (m, 18H), 0.93 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.5, 142.2, 137.8, 128.3, 128.1, 126.1, 122.6, 78.3, 62.3, 39.8, 32.0, 29.7, 29.6, 29.6, 29.5, 29.4, 23.7, 22.7, 21.6, 14.1, 14.1. **HRMS (ESI)** calculated for $C_{21}H_{34}O_3Na$ $[M+Na]^+$ *m/z* 357.2400, found 357.2394.



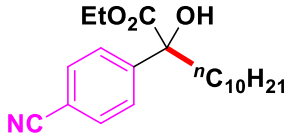
ethyl 2-hydroxy-2-(3-methoxyphenyl)dodecanoate (3f)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 68%. Light yellow oil (95.2 mg). **IR (neat, cm^{-1})** 3511, 2922, 2854, 1724, 1595, 1459, 1246, 1039, 694. **^1H NMR (400 MHz, $CDCl_3$)** δ 7.32 – 7.25 (m, 1H), 7.24 – 7.19 (m, 2H), 6.88 – 6.81 (m, 1H), 4.37 – 4.17 (m, 2H), 3.83 (s, 4H), 2.27 – 2.11 (m, 1H), 2.07 – 1.92 (m, 1H), 1.54 – 1.39 (m, 1H), 1.39 – 1.20 (m, 18H), 0.90 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.3, 159.6, 143.9, 129.2, 117.9, 113.0, 111.4, 78.3, 62.4, 55.2, 39.9, 32.0, 29.7, 29.6, 29.6, 29.5, 29.4, 23.7, 22.7, 14.2, 14.1. **HRMS (ESI)** calculated for $C_{21}H_{34}O_4Na$ $[M+Na]^+$ *m/z* 373.2349, found 373.2354.



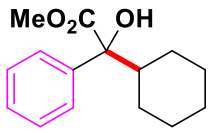
ethyl 2-(4-fluorophenyl)-2-hydroxydodecanoate (3g)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 64%. Colorless oil (86 mg). **IR (neat, cm^{-1})** 3512, 2923, 2856, 1725, 1506, 1229, 833. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.65 – 7.52 (m, 2H), 7.12 – 6.88 (m, 2H), 4.33 – 4.11 (m, 2H), 3.85 (s, 1H), 2.22 – 2.08 (m, 1H), 2.03 – 1.89 (m, 1H), 1.51 – 1.36 (m, 1H), 1.32 – 1.19 (m, 18H), 0.87 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 175.38, 162.37 (d, *J* = 246.4 Hz), 137.95 (d, *J* = 3.0 Hz), 127.50 (d, *J* = 8.1 Hz), 114.99 (d, *J* = 22.2 Hz), 78.02, 62.60, 40.05, 32.01, 29.73, 29.69, 29.65, 29.55, 29.43, 23.67, 22.78, 14.20, 14.16. **^{19}F NMR (377 MHz, CDCl$_3$)** δ -115.47. **HRMS (ESI)** calculated for $C_{20}H_{31}O_3FNa$ $[M+Na]^+$ *m/z* 361.2149, found 361.2157.



ethyl 2-(4-cyanophenyl)-2-hydroxydodecanoate (3h)

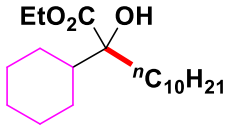
The crude product was purified by flash column chromatography (hexanes/EtOAc= 10/1) on silica gel to give the title compound. Yield: 62%. Colorless oil (85.3 mg). **IR (neat, cm^{-1})** 3502, 2922, 2855, 1726, 1459, 1240, 843. **^{1}H NMR (400 MHz, CDCl$_3$)** δ7.74 (d, *J* = 8.1 Hz, 2H), 7.62 (d, *J* = 8.1 Hz, 2H), 4.39 – 4.15 (m, 2H), 3.91 (s, 1H), 2.18 – 2.07 (m, 1H), 2.02 – 1.86 (m, 1H), 1.39 – 1.14 (m, 19H), 0.85 (t, *J* = 6.7 Hz, 3H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 174.4, 147.2, 132.0, 126.6, 118.7, 111.56, 78.1, 63.0, 40.0, 31.9, 29.6, 29.5, 29.4, 29.3, 23.5, 22.7, 14.1, 14.1. **HRMS (ESI)** calculated for $C_{21}H_{30}NO_2$ $[M-OH]^+$ *m/z* 328.2271, found 328.2270.



methyl 2-cyclohexyl-2-hydroxy-2-phenylacetate (3i)

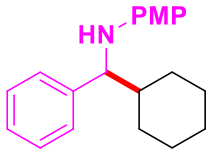
The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 67%. Colorless oil (66.5 mg). **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.68 – 7.59 (m, 2H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.30 – 7.22 (m, 1H), 3.77 (d, *J* = 0.9 Hz, 3H), 3.67 (s,

1H), 2.29 – 2.17 (m, 1H), 1.87 – 1.74 (m, 1H), 1.70 – 1.60 (m, 2H), 1.48 – 1.39 (m, 2H), 1.37 – 1.24 (m, 1H), 1.22 – 1.03 (m, 4H). The analytical data are in accordance with the literature[1].



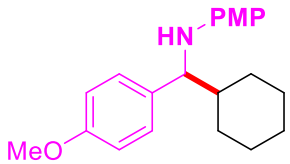
ethyl 2-cyclohexyl-2-hydroxydodecanoate (3j)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 50%. Colorless oil (65.2 mg). **IR (neat, cm^{-1})** 3530, 2922, 2854, 1723, 1455, 1223, 713. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 4.30 – 4.16 (m, 2H), 3.14 (s, 1H), 1.88 – 1.51 (m, 7H), 1.47 – 1.37 (m, 1H), 1.35 – 1.05 (m, 24H), 0.86 (t, J = 6.7 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 177.0, 79.8, 61.5, 45.2, 36.1, 31.8, 29.7, 29.5, 29.5, 29.5, 29.4, 29.3, 27.4, 26.3, 26.2, 25.8, 23.6, 22.6, 14.2, 14.0. **HRMS (ESI)** calculated for $C_{20}H_{38}O_3Na$ $[M+Na]^+$ *m/z* 349.2713, found 349.2713.



N-(cyclohexyl(phenyl)methyl)-4-methoxyaniline (3k)

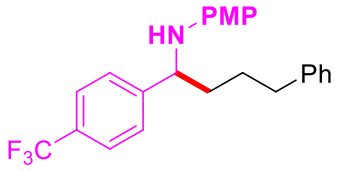
The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 53%. Yellow oil (34.8 mg). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.33 – 7.27 (m, 4H), 7.24 – 7.18 (m, 1H), 6.71 – 6.64 (m, 2H), 6.51 – 6.43 (m, 2H), 4.05 (d, J = 6.2 Hz, 1H), 3.91 (s, 1H), 3.68 (s, 3H), 1.96 – 1.86 (m, 1H), 1.81 – 1.49 (m, 5H), 1.25 – 0.94 (m, 5H). The analytical data are in accordance with the literature[2].



N-(cyclohexyl(4-methoxyphenyl)methyl)-4-methoxyaniline (3l)

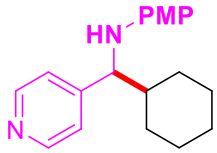
The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 45%. Yellow oil (29.3 mg). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.23 – 7.12 (m, 2H), 6.90 – 6.80 (m, 2H), 6.75

– 6.62 (m, 2H), 6.51 – 6.39 (m, 2H), 4.00 (d, J = 6.1 Hz, 1H), 3.78 (s, 3H), 3.68 (s, 3H), 1.95 – 1.85 (m, 1H), 1.81 – 1.55 (m, 5H), 1.31 – 0.95 (m, 5H). The analytical data are in accordance with the literature[3].



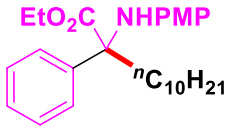
4-methoxy-N-(4-phenyl-1-(4-(trifluoromethyl)phenyl)butyl)aniline (3m)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 58%. Yellow oil (92.6 mg). **IR (neat, cm^{-1})** 3404, 2935, 1508, 1319, 1236, 1113, 821. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.63 (d, J = 8.1 Hz, 2H), 7.50 (d, J = 8.1 Hz, 2H), 7.41 – 7.34 (m, 2H), 7.32 – 7.26 (m, 1H), 7.25 – 7.17 (m, 2H), 6.82 – 6.68 (m, 2H), 6.58 – 6.45 (m, 2H), 4.38 (t, J = 6.3 Hz, 1H), 3.90 (s, 1H), 3.76 (s, 3H), 2.87 – 2.64 (m, 2H), 2.00 – 1.67 (m, 4H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 152.1, 148.74 (q, J = 1.5 Hz), 141.8, 141.2, 129.2 (q, J = 32.2 Hz), 128.5, 128.5, 126.8, 126.0, 125.6 (q, J = 3.8 Hz), 124.3 (q, J = 272.7 Hz), 114.8, 114.5, 58.7, 55.7, 38.3, 35.6, 27.9. **^{19}F NMR (377 MHz, CDCl$_3$)** δ -62.15. **HRMS (ESI)** calculated for $C_{24}H_{25}NOF_3$ $[M+H]^+$ *m/z* 400.1883, found 400.1881.



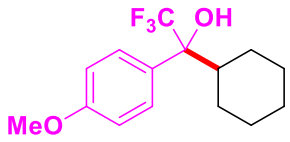
N-(cyclohexyl(pyridin-4-yl)methyl)-4-methoxyaniline (3n)

The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 2/1) on silica gel to give the title compound. Yield: 51%. Yellow oil (30.1 mg). **IR (neat, cm^{-1})** 2920, 2847, 1507, 1232, 1096, 1030, 807. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.53 (s, 2H), 7.26 – 7.09 (m, 2H), 6.85 – 6.56 (m, 2H), 6.51 – 6.21 (m, 2H), 4.05 (d, J = 5.7 Hz, 1H), 3.68 (s, 3H), 1.94 – 1.51 (m, 6H), 1.34 – 0.98 (m, 5H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 152.7, 152.1, 149.7, 141.4, 122.8, 114.9, 114.4, 63.5, 55.8, 44.5, 30.2, 29.1, 26.3, 26.3. **HRMS (ESI)** calculated for $C_{19}H_{25}N_2O$ $[M+H]^+$ *m/z* 297.1961, found 397.1955.



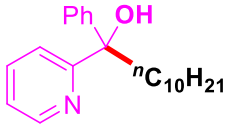
ethyl 2-((4-methoxyphenyl)amino)-2-phenyldodecanoate (3o)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 56%. Yellow oil (95.6 mg). **IR (neat, cm^{-1})** 3405, 2922, 2854, 1727, 1509, 1238, 1177, 1033, 819, 697. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.60 (d, *J* = 7.6 Hz, 2H), 7.36 – 7.30 (m, 2H), 7.29 – 7.23 (m, 1H), 6.70 – 6.53 (m, 2H), 6.43 – 6.22 (m, 2H), 4.97 (s, 1H), 4.24 – 4.14 (m, 1H), 4.10 – 4.00 (m, 1H), 3.66 (s, 3H), 2.56 – 2.34 (m, 2H), 1.32 – 1.16 (m, 16H), 1.13 (t, *J* = 7.1, 1.3 Hz, 3H), 0.87 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 174.1, 152.0, 141.5, 138.5, 128.5, 127.4, 127.2, 116.5, 114.4, 66.6, 61.8, 55.6, 33.3, 32.0, 29.6, 29.5, 29.5, 29.4, 29.4, 23.8, 22.7, 14.2, 14.0. **HRMS (ESI)** calculated for $C_{27}H_{40}NO_3$ $[M+H]^+$ *m/z* 426.3002, found 426.2998.



1-cyclohexyl-2,2,2-trifluoro-1-(4-methoxyphenyl)ethan-1-ol (3p)

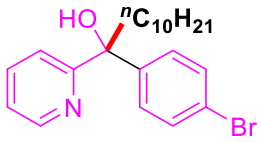
The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 41%. Yellow oil (47.2 mg). **IR (neat, cm^{-1})** 2928, 2853, 1609, 1512, 1250, 1147, 833. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.45 (d, *J* = 8.8 Hz, 2H), 6.95 – 6.83 (m, 2H), 3.82 (s, 3H), 2.61 – 2.36 (m, 1H), 2.19 – 1.97 (m, 2H), 1.90 – 1.77 (m, 1H), 1.75 – 1.58 (m, 2H), 1.37 – 1.25 (m, 2H), 1.20 – 0.96 (m, 4H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 159.35, 129.9, 127.0 (q, *J* = 1.7 Hz), 126.2 (q, *J* = 288.8 Hz), 113.6, 80.2, 79.9, 79.6, 79.4, 55.3, 44.0, 26.7 (q, *J* = 2.2 Hz), 26.5, 26.4, 26.3, 26.1. **^{19}F NMR (376 MHz, CDCl$_3$)** δ -73.28. **HRMS (ESI)** calculated for $C_{15}H_{19}O_2F_3$ $[M]^+$ *m/z* 288.1332, found 288.1331.



1-phenyl-1-(pyridin-2-yl)undecan-1-ol (3q)

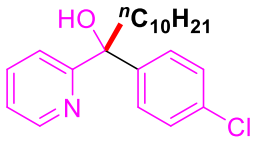
The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 58%. Yellow oil (75.4 mg). **IR (neat, cm^{-1})** 3361, 2921, 2853,

1584, 1458, 1387, 998, 698. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.50 (d, *J* = 4.9 Hz, 1H), 7.69 – 7.59 (m, 1H), 7.58 – 7.52 (m, 2H), 7.37 – 7.29 (m, 3H), 7.25 – 7.18 (m, 1H), 7.18 – 7.12 (m, 1H), 5.99 (s, 1H), 2.40 – 2.16 (m, 2H), 1.53 – 1.40 (m, 1H), 1.37 – 1.21 (m, 14H), 1.20 – 1.09 (m, 1H), 0.89 (t, *J* = 6.7 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.9, 147.2, 146.7, 137.0, 128.2, 126.8, 126.0, 121.9, 120.5, 77.2, 41.4, 32.0, 30.1, 29.7, 29.6, 29.6, 29.4, 23.6, 22.7, 14.22. **HRMS (ESI)** calculated for $C_{22}H_{31}NONa$ $[M+Na]^+$ *m/z* 348.2298, found 348.2290.



1-(4-bromophenyl)-1-(pyridin-2-yl)undecan-1-ol (3r)

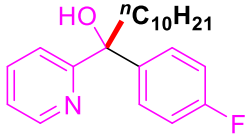
The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 55%. Light yellow oil (44.3 mg). **IR (neat, cm^{-1})** 3353, 2920, 2853, 1583, 1469, 1388, 1074, 1003, 744. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.51 (d, *J* = 4.9 Hz, 1H), 7.72 – 7.63 (m, 1H), 7.46 – 7.36 (m, 4H), 7.31 (d, *J* = 8.0 Hz, 1H), 7.23 – 7.16 (m, 1H), 5.91 (s, 1H), 2.43 – 2.01 (m, 2H), 1.45 – 1.34 (m, 1H), 1.33 – 1.17 (m, 14H), 1.17 – 1.10 (m, 1H), 0.87 (t, *J* = 6.7 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.1, 147.1, 145.5, 137.3, 131.2, 127.8, 122.1, 120.8, 120.3, 76.9, 41.1, 31.8, 29.9, 29.5, 29.5, 29.3, 23.4, 22.6, 14.1. **HRMS (ESI)** calculated for $C_{22}H_{31}NOBr$ $[M+H]^+$ *m/z* 404.1583, found 404.1582.



1-(4-chlorophenyl)-1-(pyridin-2-yl)undecan-1-ol (3s)

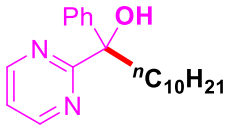
The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 58%. Light yellow oil (41.6 mg). **IR (neat, cm^{-1})** 3356, 2921, 2853, 1574, 1390, 1085, 1006, 824, 747. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.48 (d, *J* = 4.8 Hz, 1H), 7.82 – 7.56 (m, 1H), 7.50 – 7.39 (m, 2H), 7.31 – 7.23 (m, 3H), 7.21 – 7.10 (m, 1H), 5.94 (s, 1H), 2.38 – 2.00 (m, 2H), 1.47 – 1.04 (m, 17H), 0.85 (t, *J* = 6.8 Hz, 3H). ^{13}C **NMR (101 MHz, $CDCl_3$)** δ 163.4, 147.4, 145.3, 137.2, 132.7, 128.4, 127.6, 122.2,

120.4, 76.9, 41.3, 32.0, 30.0, 29.7, 29.6, 29.4, 23.6, 22.8, 14.2. **HRMS (ESI)** calculated for $C_{22}H_{31}NOCl$ $[M+H]^+$ *m/z* 360.2088, found 360.2081.



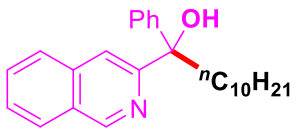
1-(4-fluorophenyl)-1-(pyridin-2-yl)undecan-1-ol (3t)

The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 56%. Light yellow oil (38.6 mg). **IR (neat, cm^{-1})** 3362, 2922, 2854, 1595, 1505, 1389, 1225, 1077, 827. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.50 (d, *J* = 4.9 Hz, 1H), 7.65 (t, *J* = 7.8 Hz, 1H), 7.57 – 7.44 (m, 2H), 7.35 – 7.26 (m, 1H), 7.22 – 7.11 (m, 1H), 6.98 (t, *J* = 8.5 Hz, 2H), 5.96 (s, 1H), 2.37 – 2.08 (m, 2H), 1.47 – 1.34 (m, 1H), 1.32 – 1.17 (m, 14H), 1.16 – 1.07 (m, 1H), 0.87 (t, *J* = 6.7 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.6, 161.8 (d, *J* = 245.4 Hz), 147.3, 142.5 (d, *J* = 3.0 Hz), 137.2, 127.8 (d, *J* = 8.0 Hz), 122.1, 120.4, 115.0 (d, *J* = 21.0 Hz), 76.9, 41.5, 32.2, 30.1, 29.7, 29.6, 29.4, 23.6, 22.8, 14.2. **HRMS (ESI)** calculated for $C_{22}H_{31}NOF$ $[M+H]^+$ *m/z* 344.2384, found 344.2377.



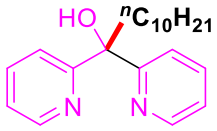
1-phenyl-1-(pyrimidin-2-yl)undecan-1-ol (3u)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 1/1) on silica gel to give the title compound. Yield: 55%. Colorless oil (77.7 mg). **IR (neat, cm^{-1})** 3440, 2921, 2853, 1565, 1372, 1064, 807, 698. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 9.04 – 8.60 (m, 2H), 7.96 – 7.64 (m, 2H), 7.31 (t, *J* = 7.7 Hz, 2H), 7.24 – 7.18 (m, 1H), 7.16 – 7.10 (m, 1H), 5.49 (s, 1H), 2.56 – 2.30 (m, 2H), 1.52 – 1.05 (m, 16H), 0.88 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 172.5, 156.7, 145.7, 128.0, 126.8, 125.9, 119.0, 78.5, 41.6, 31.9, 29.9, 29.6, 29.6, 29.4, 23.7, 22.7, 14.2. **HRMS (ESI)** calculated for $C_{21}H_{30}N_2ONa$ $[M+Na]^+$ *m/z* 349.2250, found 349.2248.



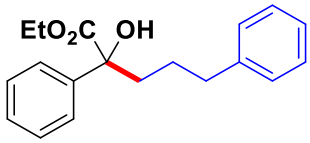
1-(isoquinolin-3-yl)-1-phenylundecan-1-ol (3v)

The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 75%. Yellow oil (56.2 mg). **IR (neat, cm^{-1})** 3354, 2920, 2853, 1603, 1387, 1025, 823, 756, 700. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.21 – 7.98 (m, 2H), 7.83 – 7.68 (m, 2H), 7.66 – 7.60 (m, 2H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.39 (d, *J* = 8.6 Hz, 1H), 7.33 (t, *J* = 7.8 Hz, 2H), 7.28 – 7.19 (m, 1H), 6.73 (s, 1H), 2.59 – 2.26 (m, 2H), 1.64 – 1.50 (m, 1H), 1.44 – 1.19 (m, 16H), 1.17 – 1.04 (m, 1H), 0.89 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.4, 146.2, 145.5, 137.3, 129.9, 128.9, 128.3, 127.5, 127.1, 127.0, 126.6, 126.4, 118.5, 77.3, 40.5, 31.9, 30.1, 29.7, 29.6, 29.6, 29.4, 23.6, 22.7, 14.2. **HRMS (ESI)** calculated for $C_{26}H_{34}NO$ $[M+H]^+$ *m/z* 376.2635, found 376.2627.



1,1-di(pyridin-2-yl)undecan-1-ol (3w)

The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 10/1) on silica gel to give the title compound. Yield: 41%. Colorless oil (26.7 mg). **IR (neat, cm^{-1})** 3349, 2920, 2853, 1582, 1431, 1390, 1144, 798, 619. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.55 – 8.41 (m, 2H), 7.94 – 7.76 (m, 2H), 7.74 – 7.53 (m, 2H), 7.18 – 7.01 (m, 2H), 6.49 (s, 1H), 2.44 – 2.25 (m, 2H), 1.31 – 1.12 (m, 16H), 0.85 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.8, 147.5, 136.8, 121.9, 121.1, 78.3, 41.9, 32.0, 30.0, 29.7, 29.6, 29.4, 23.6, 22.8, 14.2. **HRMS (ESI)** calculated for $C_{21}H_{31}N_2O$ $[M+H]^+$ *m/z* 327.2431, found 327.2428.

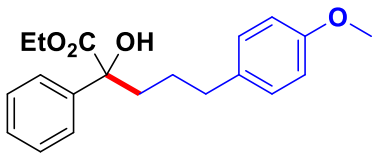


ethyl 2-hydroxy-2,5-diphenylpentanoate (4a)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 71%. Colorless oil (87.0 mg). The product was obtained in 47% yield (56.0 mg) when using (3-bromopropyl)benzene as substrate. **IR (neat, cm^{-1})** 3507, 1721, 1449, 1239, 1018, 737, 695. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.67 – 7.51 (m, 2H), 7.45 – 7.24 (m, 5H), 7.25 – 7.12 (m, 3H), 4.42 – 4.11 (m, 2H), 3.85 (s, 1H), 2.67 (t, *J* = 7.6 Hz, 2H), 2.34 – 2.02 (m,

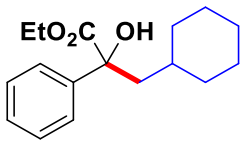
2H), 1.89 – 1.58 (m, 2H), 1.27 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.3, 142.1, 142.0, 128.4, 128.3, 128.2, 127.7, 125.8, 125.5, 78.2, 62.5, 39.3, 35.8, 25.5, 14.1.

HRMS (ESI) calculated for $C_{19}H_{22}O_3Na$ $[M+Na]^+$ *m/z* 342.1461, found 342.1462.



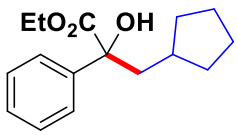
ethyl 2-hydroxy-5-(4-methoxyphenyl)-2-phenylpentanoate (4b)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 10/1) on silica gel to give the title compound. Yield: 62%. Colorless oil (81.3 mg). **IR (neat, cm^{-1})** 3505, 2926, 1722, 1507, 1239, 1026, 697. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.71 – 7.50 (m, 2H), 7.44 – 7.33 (m, 2H), 7.33 – 7.27 (m, 1H), 7.17 – 7.00 (m, 2H), 6.90 – 6.70 (m, 2H), 4.40 – 4.15 (m, 2H), 3.84 (s, 1H), 3.79 (s, 3H), 2.60 (t, *J* = 7.6 Hz, 2H), 2.31 – 2.17 (m, 1H), 2.13 – 1.99 (m, 1H), 1.88 – 1.72 (m, 1H), 1.66 – 1.53 (m, 1H), 1.27 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.3, 157.8, 142.0, 134.2, 129.3, 128.2, 127.6, 125.5, 113.8, 78.3, 62.5, 55.3, 39.2, 34.9, 25.7, 14.1. **HRMS (ESI)** calculated for $C_{20}H_{24}O_4Na$ $[M+Na]^+$ *m/z* 351.1567, found 351.1564.



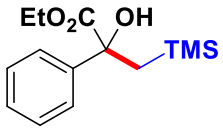
ethyl 3-cyclohexyl-2-hydroxy-2-phenylpropanoate (4c)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 10/1) on silica gel to give the title compound. Yield: 71%. Colorless oil (78.1 mg). **IR (neat, cm^{-1})** 3509, 2919, 2849, 1720, 1446, 1240, 1024, 696. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.71 – 7.57 (m, 2H), 7.36 (t, *J* = 7.5 Hz, 2H), 7.33 – 7.26 (m, 1H), 4.36 – 4.13 (m, 2H), 3.85 (s, 1H), 2.18 (dd, *J* = 14.3, 6.6 Hz, 1H), 1.97 (dd, *J* = 14.3, 5.2 Hz, 1H), 1.84 – 1.51 (m, 6H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.28 – 1.12 (m, 2H), 1.07 – 0.94 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.9, 143.0, 128.2, 127.5, 125.5, 78.1, 62.4, 46.8, 34.9, 33.9, 33.8, 26.4, 26.4, 14.1. **HRMS (ESI)** calculated for $C_{17}H_{24}O_3Na$ $[M+Na]^+$ *m/z* 299.1618, found 299.1612.



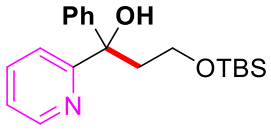
ethyl 3-cyclopentyl-2-hydroxy-2-phenylpropanoate (4d)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 10/1) on silica gel to give the title compound. Yield: 68%. Colorless oil (71.3 mg). **IR (neat, cm^{-1})** 3509, 2947, 2864, 1720, 1447, 1231, 1025, 697. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.69 – 7.55 (m, 2H), 7.38 – 7.30 (m, 2H), 7.29 – 7.23 (m, 1H), 4.29 – 4.08 (m, 2H), 3.83 (s, 1H), 2.35 – 2.23 (m, 1H), 2.13 (dd, *J* = 14.1, 5.8 Hz, 1H), 1.96 – 1.84 (m, 1H), 1.82 – 1.64 (m, 2H), 1.63 – 1.39 (m, 4H), 1.26 (t, *J* = 7.1 Hz, 3H), 1.19 – 1.03 (m, 2H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 175.8, 142.7, 128.1, 127.5, 125.6, 78.3, 62.4, 45.5, 36.3, 34.1, 33.2, 25.0, 24.7, 14.0. **HRMS (ESI)** calculated for $C_{16}H_{22}O_3Na$ $[M+Na]^+$ *m/z* 285.1461 found 285.1456.



ethyl 2-hydroxy-2-phenyl-3-(trimethylsilyl)propanoate (4e)

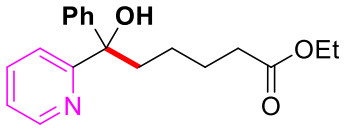
The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 53%. Light yellow oil (56.4 mg). **IR (neat, cm^{-1})** 3509, 2954, 1719, 1243, 1098, 1024, 844, 728. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 7.68 – 7.59 (m, 2H), 7.43 – 7.32 (m, 2H), 7.31 – 7.26 (m, 1H), 4.28 – 4.18 (m, 2H), 3.87 (d, *J* = 1.0 Hz, 1H), 1.73 – 1.56 (m, 1H), 1.29 (t, *J* = 7.1 Hz, 3H), -0.01 (s, 9H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 176.4, 144.4, 128.1, 127.4, 125.4, 77.8, 62.4, 29.3, 14.1, -0.1. **HRMS (ESI)** calculated for $C_{14}H_{22}O_3NaSi$ $[M+Na]^+$ *m/z* 289.1230, found 289.1222.



3-((tert-butyldimethylsilyl)oxy)-1-phenyl-1-(pyridin-2-yl)propan-1-ol (4f)

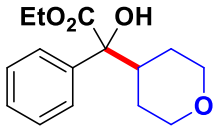
The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 3/1) on silica gel to give the title compound. Yield: 49%. Yellow oil (33.3 mg). **IR (neat, cm^{-1})** 3445, 2937, 1581, 1461, 1251, 1072, 833, 659. **^{1}H NMR (400 MHz, CDCl$_3$)** δ 8.51 (d, *J* = 4.8 Hz, 1H), 7.80 – 7.55 (m, 4H), 7.34 – 7.23 (m, 2H), 7.21 – 7.14 (m, 1H), 7.13 – 7.06 (m, 1H), 5.82 (s, 1H), 3.86 – 3.57 (m, 2H), 3.01 – 2.53 (m, 2H), 0.85 (s, 9H), -0.01 – -0.11 (m, 6H). **^{13}C NMR (101 MHz, CDCl$_3$)** δ 165.0, 147.8, 146.4, 136.8, 128.1, 126.7, 125.7, 121.7,

120.9, 78.7, 61.1, 41.8, 25.9, 18.1, -5.6, -5.6. **HRMS (ESI)** calculated for $C_{20}H_{30}NO_2Si$ $[M+H]^+$ *m/z* 344.2040, found 344.2038.



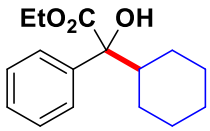
ethyl 6-hydroxy-6-phenyl-6-(pyridin-2-yl)hexanoate (4g)

The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 3/1) on silica gel to give the title compound. Yield: 46%. Yellow solid (28.5 mg). **Melting point**: 38-40 °C. **IR (neat, cm^{-1})** 3329, 2942, 1716, 1391, 1166, 696. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.47 (d, *J* = 4.9 Hz, 1H), 7.71 – 7.58 (m, 1H), 7.51 (d, *J* = 7.7 Hz, 2H), 7.34 – 7.26 (m, 3H), 7.22 – 7.10 (m, 2H), 5.97 (s, 1H), 4.06 (q, *J* = 7.1 Hz, 2H), 2.68 – 2.10 (m, 4H), 1.86 – 1.56 (m, 2H), 1.51 – 1.34 (m, 1H), 1.19 (t, *J* = 7.1 Hz, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 173.7, 163.6, 147.2, 146.3, 137.1, 128.3, 126.9, 125.9, 122.0, 120.4, 77.0, 60.2, 40.9, 34.3, 25.4, 23.3, 14.2. **HRMS (ESI)** calculated for $C_{19}H_{24}NO_3$ $[M+H]^+$ *m/z* 314.1751, found 314.1747.



ethyl 2-hydroxy-2-phenyl-2-(tetrahydro-2H-pyran-4-yl)acetate (4h)

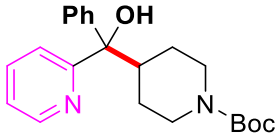
The crude product was purified by flash column chromatography (hexanes/EtOAc= 3/1) on silica gel to give the title compound. Yield: 73%. Colorless oil (76.7 mg). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.72 – 7.64 (m, 2H), 7.42 – 7.34 (m, 2H), 7.34 – 7.27 (m, 1H), 4.40 – 4.19 (m, 2H), 4.10 – 3.89 (m, 2H), 3.75 (s, 1H), 3.50 – 3.25 (m, 2H), 2.56 – 2.42 (m, 1H), 2.01 – 1.79 (m, 1H), 1.62 – 1.45 (m, 1H), 1.42 – 1.28 (m, 4H), 1.10 – 0.98 (m, 1H). The analytical data are in accordance with the literature[4].



ethyl 2-cyclohexyl-2-hydroxy-2-phenylacetate (4i)

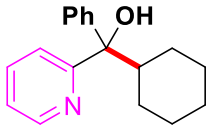
The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 82%. Colorless oil (85.9 mg). **^{1}H NMR (400 MHz, $CDCl_3$)** δ

7.76 – 7.66 (m, 2H), 7.41 – 7.33 (m, 2H), 7.33 – 7.26 (m, 1H), 4.36 – 4.15 (m, 2H), 3.75 (s, 1H), 2.34 – 2.16 (m, 1H), 1.90 – 1.80 (m, 1H), 1.72 – 1.63 (m, 2H), 1.53 – 1.42 (m, 2H), 1.38 – 1.28 (m, 4H), 1.26 – 1.10 (m, 4H). The analytical data are in accordance with the literature[4].



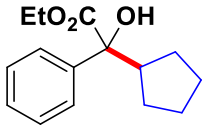
tert-butyl 4-(hydroxy(phenyl)(pyridin-2-yl)methyl)piperidine-1-carboxylate (4j)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 5/1) on silica gel to give the title compound. Yield: 60%. White solid (88.3 mg). **Melting point**: 144-146 ℃. **IR (neat, cm^{-1})** 3472, 2840, 1661, 1458, 1150, 976, 761, 532. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.45 (d, *J* = 4.9 Hz, 1H), 7.71 – 7.58 (m, 3H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.24 – 7.08 (m, 2H), 6.13 (s, 1H), 4.11 (s, 2H), 3.03 – 2.42 (m, 3H), 1.57 – 1.47 (m, 3H), 1.42 (s, 9H), 0.97 (d, *J* = 13.2 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.4, 154.7, 147.0, 145.1, 137.2, 128.3, 126.8, 125.9, 122.1, 120.3, 79.2, 78.6, 44.8, 43.7, 28.4, 26.2, 25.8. **HRMS (ESI)** calculated for $C_{22}H_{29}N_2O_3$ $[M+H]^+$ *m/z* 369.2173 found 369.2164.



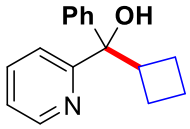
cyclohexyl(phenyl)(pyridin-2-yl)methanol (4k)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 75%. White solid (80.7 mg). **Melting point**: 51-53 ℃. **IR (neat, cm^{-1})** 3339, 2922, 2852, 1583, 1385, 937, 750, 703. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.53 – 8.35 (m, 1H), 7.70 – 7.58 (m, 3H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.15 – 7.08 (m, 1H), 6.15 (s, 1H), 2.50 – 2.35 (m, 1H), 1.80 – 1.56 (m, 4H), 1.41 – 1.15 (m, 5H), 1.11 – 1.02 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.3, 146.9, 146.0, 137.1, 128.2, 126.5, 126.0, 121.8, 120.5, 79.5, 46.3, 26.9, 26.8, 26.7, 26.5. **HRMS (ESI)** calculated for $C_{18}H_{22}NO$ $[M+H]^+$ *m/z* 268.1596, found 268.1695.



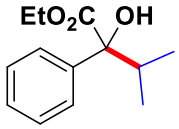
ethyl 2-cyclopentyl-2-hydroxy-2-phenylacetate (4l)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 71%. Colorless oil (70.2 mg). The product was obtained in 50% yield (49.6 mg) when using bromocyclopentane as substrate. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.72 – 7.66 (m, 2H), 7.40 – 7.34 (m, 2H), 7.32 – 7.26 (m, 1H), 4.38 – 4.14 (m, 2H), 3.81 (s, 1H), 3.03 – 2.88 (m, 1H), 1.75 – 1.55 (m, 5H), 1.53 – 1.44 (m, 1H), 1.41 – 1.34 (m, 2H), 1.31 (t, J = 7.1 Hz, 3H). The analytical data are in accordance with the literature[4].



cyclobutyl(phenyl)(pyridin-2-yl)methanol (4m)

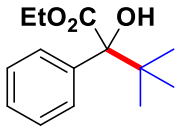
The Reaction was run on a 0.2 mmol scale. The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 68%. Yellow oil (32.5 mg). **IR (neat, cm^{-1})** 3345, 2936, 1584, 1434, 1384, 999, 756, 693. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.66 – 8.34 (m, 1H), 7.64 – 7.55 (m, 1H), 7.53 – 7.45 (m, 2H), 7.35 – 7.08 (m, 5H), 6.12 (s, 1H), 3.53 – 3.40 (m, 1H), 2.31 – 2.17 (m, 1H), 2.07 – 1.91 (m, 2H), 1.88 – 1.69 (m, 2H), 1.62 – 1.49 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 162.8, 147.0, 145.5, 136.9, 128.2, 126.9, 126.5, 122.0, 120.7, 77.4, 43.7, 23.0, 21.5, 17.4. **HRMS (ESI)** calculated for $C_{16}H_{18}NO$ $[M+H]^+$ *m/z* 240.1383, found 240.1380.



ethyl 2-hydroxy-3-methyl-2-phenylbutanoate (4n)

Following the **GP** using 2-bromopropane and the crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 42%. Colorless oil (37.5 mg). **IR (neat, cm^{-1})** 3509, 2972, 1720, 1455, 1243, 1158, 1026, 756, 697. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.73 – 7.64 (m, 2H), 7.45 – 7.34 (m, 2H), 7.33 – 7.25 (m, 1H), 4.38 –

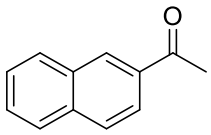
4.18 (m, 2H), 3.72 (s, 1H), 2.79 – 2.49 (m, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.02 (d, *J* = 6.6 Hz, 3H), 0.73 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.8, 141.4, 128.1, 127.4, 126.0, 80.9, 62.5, 35.9, 17.2, 15.9, 14.1. **HRMS (ESI)** calculated for $C_{13}H_{18}O_3$ $[M]^+$ *m/z* 222.1250, found 222.1247.



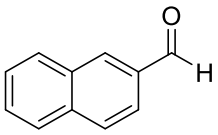
ethyl 2-hydroxy-3,3-dimethyl-2-phenylbutanoate (4o)

The crude product was purified by flash column chromatography (hexanes/EtOAc= 20/1) on silica gel to give the title compound. Yield: 42%. Colorless oil (39.6 mg). The product was obtained in 36% yield (33.9 mg) when using 2-bromo-2-methylpropane as substrate. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.95 – 7.54 (m, 2H), 7.46 – 7.15 (m, 3H), 4.34 (q, *J* = 7.2 Hz, 2H), 3.77 (s, 1H), 1.50 – 1.25 (m, 3H), 1.15 – 0.95 (m, 9H). The analytical data are in accordance with the literature[4].

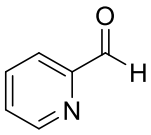
Reactions using common ketone, aldehydes and 1-bromodecane as substrates



25% yield

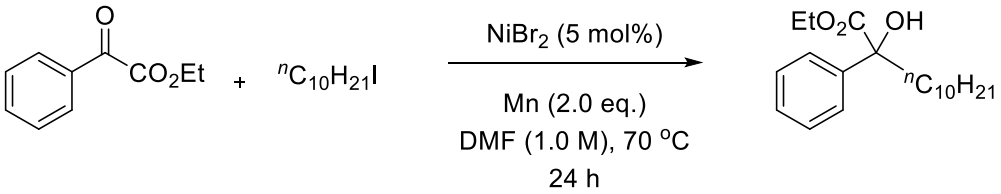


7% yield



5% yield

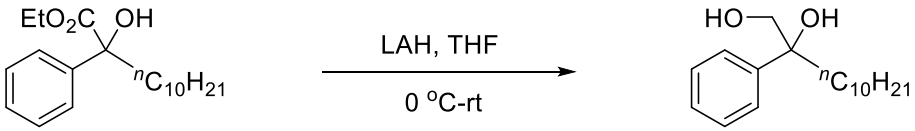
4. Gram-scale Synthesis



In a nitrogen-filled glove box, $NiBr_2$ (87.2 mg, 0.4 mmol), ethyl 2-oxo-2-phenylacetate (1.42 g, 8.0 mmol), manganese powder (864 mg, 16 mmol), 1-iododecane (4.28 g, 16 mmol) and DMF (8 mL) were charged to Schlenk tube equipped with a magnetic stirring bar. The reaction mixture was stirred at 70 °C for 24 h. The resulting mixture was filtered through a short pad of silica gel, eluted with ethyl acetate, and concentrated in vacuo. The crude product was purified by flash column chromatography (hexanes/EtOAc) on silica gel to give the corresponding product as a colorless oil in 57% yield (1.46 g).

5. Synthetic Applications

5.1 Synthesis of vicinal diol

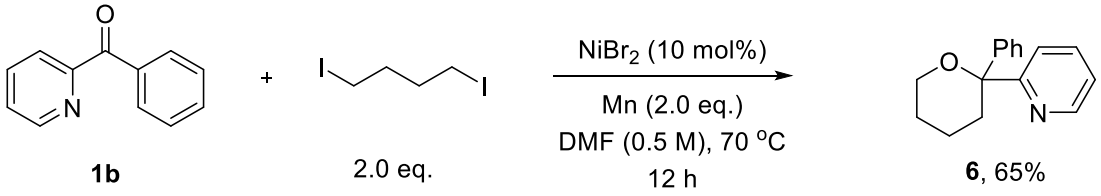


3a **5**, 85%

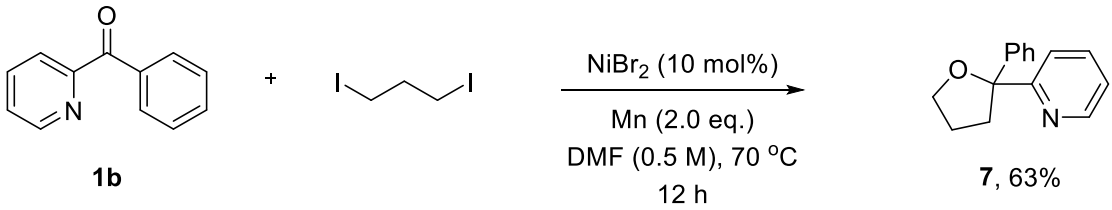
To a solution of **3a** (0.1 mmol, 1.0 equiv.) in dried THF (2.0 mL) was added $LiAlH_4$ (0.2 mmol, 2.0 eq.) at 0 °C, then the mixture was slowly warmed to room temperature. The reaction mixture was stirred at room temperature overnight. Then the mixture was quenched with H_2O, filtered through a pad of diatomite and the filter cake was washed with EtOAc. The organic layer was separated, and the aqueous phase was washed with EtOAc three times. The combined organic layer was washed with brine, dried over Na_2SO_4, filtered and concentrated in vacuo to afford **5** as a white solid (23.6 mg, 85%). **Melting point**: 51-53 °C. **IR (neat, cm^{-1})** 3385, 2914, 2849, 1456, 1303, 1041, 599. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.45 – 7.35 (m, 4H), 7.33 – 7.26 (m, 1H), 3.95 – 3.64 (m, 2H), 2.72 (s, 1H), 2.02 – 1.67 (m, 4H), 1.41 – 1.16 (m, 16H), 1.12 – 0.99 (m, 1H), 0.89 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 143.70 128.5, 127.0, 125.6, 77.4, 70.7, 38.6, 32.0,

30.1, 29.6, 29.5, 29.4, 23.1, 22.8, 14.2. **HRMS (ESI)** calculated for $C_{18}H_{30}O_2$ $[M]^+$ *m/z* 278.2240, found 278.2245.

5.2 Construction of heterocycles



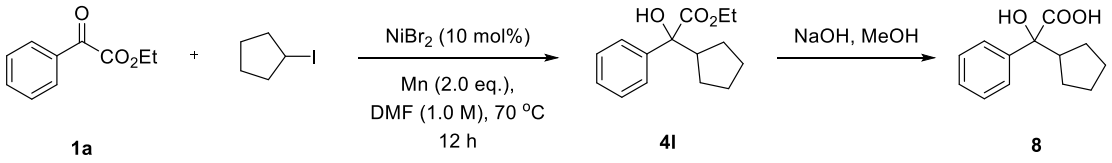
In a nitrogen-filled glove box, $NiBr_2$ (8.72 mg, 0.04 mmol), phenyl(pyridin-2-yl)methanone (73.2 mg, 0.4 mmol), manganese powder (43.2 mg, 0.8 mmol), 1,4-diiodobutane (247 mg, 0.8 mmol) and DMF (0.8 mL) were charged to a 4 mL vial equipped with a magnetic stirring bar. The reaction mixture was stirred at 70 °C for 12 h. The resulting mixture was filtered through a short pad of silica gel, eluting with ethyl acetate and concentrated in vacuo. The crude product was purified by flash column chromatography (hexanes/EtOAc = 3:1) on silica gel to give the corresponding product as a yellow oil in 65% yield (62.1 mg). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.61 – 8.55 (m, 1H), 7.64 – 7.55 (m, 2H), 7.53 – 7.45 (m, 2H), 7.35 – 7.25 (m, 2H), 7.22 – 7.14 (m, 1H), 7.12 – 7.04 (m, 1H), 4.01 – 3.81 (m, 1H), 3.80 – 3.63 (m, 1H), 2.85 – 2.66 (m, 1H), 2.42 – 2.19 (m, 1H), 1.87 – 1.48 (m, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 164.2, 148.6, 145.8, 136.6, 128.2, 126.5, 125.8, 121.5, 121.2, 80.8, 63.4, 34.3, 25.9, 20.5. The analytical data are in accordance with the literature[5].



In a nitrogen-filled glove box, $NiBr_2$ (8.72 mg, 0.04 mmol), phenyl(pyridin-2-yl)methanone (73.2 mg, 0.4 mmol), manganese powder (43.2 mg, 0.8 mmol), 1,3-diiodopropane (236 mg, 0.8 mmol) and DMF (0.8 mL) were charged to a 4 mL vial equipped with a magnetic stirring bar. The reaction mixture was stirred at 70 °C for 12 h. The resulting mixture was filtered through a short pad of silica gel, eluting with ethyl acetate and concentrated in vacuo. The crude product was purified by flash column chromatography (hexanes/EtOAc = 10:1) on silica gel to give the corresponding product as a yellow oil in 63% yield (56.7 mg). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.54 (d, *J* = 4.8 Hz, 1H), 7.61 – 7.48 (m, 4H), 7.27 (t, *J* = 7.6 Hz, 2H), 7.17 (t, *J* = 7.3 Hz, 1H), 7.11 – 7.04 (m, 1H), 4.23 – 3.94

(m, 2H), 3.35 – 2.92 (m, 1H), 2.54 – 2.24 (m, 1H), 2.09 – 1.75 (m, 2H). The analytical data are in accordance with the literature[5].

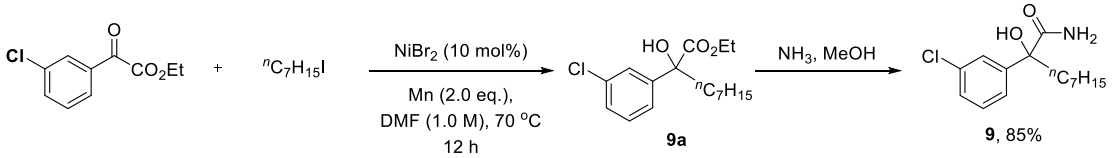
5.3 Synthesis of bioactive compounds



1a **4l** **8**

In a nitrogen-filled glove box, $NiBr_2$ (8.72 mg, 0.04 mmol), phenyl(pyridin-2-yl)methanone (73.2 mg, 0.4 mmol), manganese powder (43.2 mg, 0.8 mmol), iodocyclopentane (156 mg, 0.8 mmol) and DMF (0.4 mL) were charged to a 4 mL vial equipped with a magnetic stirring bar. The reaction mixture was stirred at 70 °C for 12 h. The resulting mixture was filtered through a short pad of silica gel, eluted with ethyl acetate, and concentrated in vacuo. The crude product was purified by flash column chromatography (hexanes/EtOAc = 20:1) on silica gel to give the corresponding product as a colorless oil in 71% yield (70.2 mg).

To a solution of **4l** (24.8 mg, 0.1mmol) in methanol (0.56 mL) was added 1 N NaOH (0.18 mL). The reaction mixture was allowed to warm to 70 ℃ and stirred for 6.0 hours. After cooling to room temperature, the mixture was extracted with ethyl ether. The combined organic solution was washed with brine, dried over sodium sulfate, and concentrated in vacuo. The reaction mixture was purified by PTLC (silica gel, PE/EtOAc = 3/1) to afford **8** as a white solid (18.7 mg, 85%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.78 – 7.51 (m, 2H), 7.45 – 7.16 (m, 3H), 3.18 – 2.76 (m, 1H), 1.75 – 1.50 (m, 4H), 1.48 – 1.25 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 180.5, 141.0, 128.3, 127.9, 126.0, 79.3, 47.3, 27.0, 26.5, 26.4, 26.0. The analytical data are in accordance with the literature[5].

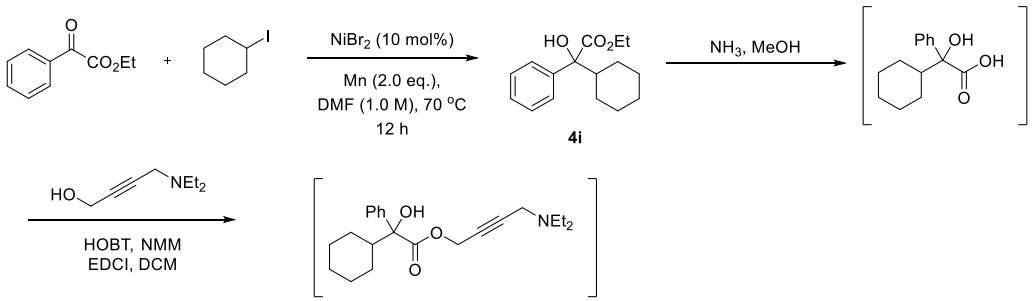


9a **9**, 85%

In a nitrogen-filled glove box, $NiBr_2$ (8.72 mg, 0.04 mmol), phenyl(pyridin-2-yl)methanone (73.2 mg, 0.4 mmol), manganese powder (43.2 mg, 0.8 mmol), 1-iodoheptane (180.8 mg, 0.8 mmol) and DMF (0.4 mL) were charged to a 4 mL vial equipped with a magnetic stirring bar. The reaction mixture was stirred at 70 °C for 12 h. The resulting mixture was filtered through a short pad of silica gel, eluted with ethyl acetate, and concentrated in vacuo. The crude product was purified by flash column chromatography (hexanes/EtOAc = 20:1) on silica gel to give the corresponding product as

a colorless oil in 57% yield (71.1 mg). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.61 (s, 1H), 7.50 – 7.41 (m, 1H), 7.29 – 7.18 (m, 2H), 4.34 – 4.09 (m, 2H), 3.83 (s, 1H), 2.19 – 2.05 (m, 1H), 2.02 – 1.87 (m, 1H), 1.46 – 1.33 (m, 1H), 1.33 – 1.16 (m, 12H), 0.85 (t, *J* = 6.5 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 175.0, 144.2, 134.3, 129.5, 127.8, 126.0, 123.9, 78.0, 62.8, 39.9, 31.8, 29.6, 29.2, 23.6, 22.7, 14.1. The analytical data are in accordance with the literature[5].

To a flame-dried round bottom flask was added **9a** (9.0 mg, 0.03 mmol) and was added 0.5 mL of 7 N NH_3 in MeOH dropwise. This was allowed to stir over 12 hours while monitoring by TLC. Then the reaction mixture was quenched with H_2O, and washed with EtOAc. The organic layer was separated, and the aqueous phase was washed with EtOAc three times. The combined organic solution was washed with brine, dried over sodium sulfate, and concentrated in vacuo. The reaction mixture was purified by flash column chromatography (silica gel, PE/EtOAc = 3/1) to afford **9** as a yellow oil in 85% yield (38.6 mg). The analytical data are in accordance with the literature[5].

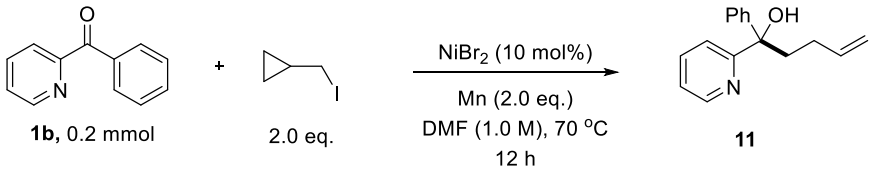


In a nitrogen-filled glove box, $NiBr_2$ (8.72 mg, 0.04 mmol), phenyl(pyridin-2-yl)methanone (73.2 mg, 0.4 mmol), manganese powder (43.2 mg, 0.8 mmol), iodocyclohexane (167.2 mg, 0.8 mmol) and DMF (0.4 mL) were charged to a 4 mL vial equipped with a magnetic stirring bar. The reaction mixture was stirred at 70 °C for 12 h. The resulting mixture was filtered through a short pad of silica gel, eluted with ethyl acetate, and concentrated in vacuo. The crude product was purified by flash column chromatography (hexanes/EtOAc = 20:1) on silica gel to give the corresponding product **4i** as a colorless oil in 82% yield (85.9 mg).

To a solution of **4i** (52.4 mg, 0.2 mmol) in methanol (1.2 mL) was added 1 N NaOH (0.2 mL). The reaction mixture was allowed to warm to 70 °C and stirred for 12.0 hours. After cooling to room temperature, the mixture was extracted with ethyl ether. The combined organic solution was washed with brine, dried over sodium sulfate, and concentrated in vacuo to give a crude product as a white

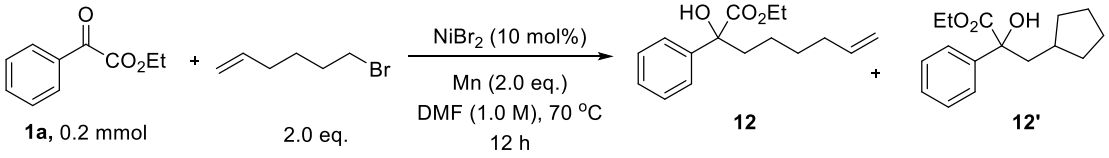
solid. Then the 1-hydroxybenzotriazole hydrate (HOBT) (27 mg, 0.2 mmol, 1.0 equiv) and N-methylmorpholine (66 uL, 0.6 mmol, 3.0 equiv) were added sequentially to a mixture of 2-cyclohexyl-2-hydroxy-2-phenylacetic acid, 4-(diethylamino)but-2-yn-1-ol (28.2 mg, 0.2 mmol, 1.0 equiv) and DCM (1.2 mL) at 0° C. After 1 h, 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCI•HCl) (68.7 mg, 0.36 mmol, 1.8 equiv) was added and the reaction mixture was stirred at room temperature for 12 hours. Then the reaction mixture was quenched with H_2O, and washed with EtOAc. The organic layer was separated, and the aqueous phase was washed with EtOAc three times. The combined organic layer was washed with brine and dried over Na_2SO_4. The reaction mixture was purified by flash column chromatography (silica gel, PE/EtOAc = 3/1) to afford **10** as a yellow oil in 65% yield (46.4 mg). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.75 – 7.50 (m, 2H), 7.41 – 7.20 (m, 3H), 4.90 – 4.56 (m, 2H), 3.64 (s, 1H), 3.41 (d, *J* = 2.1 Hz, 2H), 2.45 (q, *J* = 7.2 Hz, 4H), 2.31 – 2.19 (m, 1H), 1.84 – 1.71 (m, 1H), 1.68 – 1.59 (m, 2H), 1.57 – 1.49 (m, 1H), 1.47 – 1.36 (m, 1H), 1.35 – 1.23 (m, 1H), 1.21 – 1.06 (m, 4H), 1.02 (t, *J* = 7.0 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 174.9, 140.3, 128.0, 127.4, 126.0, 82.4, 80.9, 77.8, 54.0, 47.1, 45.7, 40.6, 27.1, 26.3, 26.3, 26.1, 25.4, 12.5. The analytical data are in accordance with the literature[5].

6. Mechanistic experiments

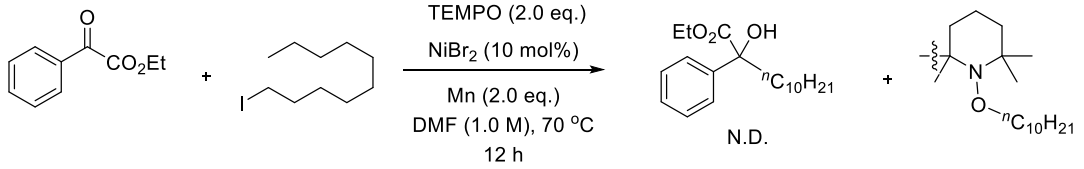


In a nitrogen-filled glove box, $NiBr_2$ (8.72 mg, 0.04 mmol), phenyl(pyridin-2-yl)methanone (73.2 mg, 0.4 mmol), manganese powder (43.2 mg, 0.8 mmol), (iodomethyl)cyclopropane (144 mg, 0.8 mmol) and DMF (0.4 mL) were charged to a 4 mL vial equipped with a magnetic stirring bar. The reaction mixture was stirred at 70 °C for 12 h. The resulting mixture was filtered through a short pad of silica gel, eluted with ethyl acetate, and concentrated in vacuo. The crude product was purified by flash column chromatography (hexanes/EtOAc = 10:1) on silica gel to give the corresponding product as a yellow oil in 45% yield (43.2 mg). **IR (neat, cm^{-1})** 3351, 2851, 1584, 1387, 1060, 909, 751, 696. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.64 – 8.39 (m, 1H), 7.70 – 7.61 (m,

1H), 7.59 – 7.50 (m, 2H), 7.38 – 7.29 (m, 3H), 7.27 – 7.19 (m, 1H), 7.19 – 7.14 (m, 1H), 6.04 (s, 1H), 5.94 – 5.77 (m, 1H), 5.06 – 4.86 (m, 2H), 2.58 – 2.28 (m, 2H), 2.27 – 2.15 (m, 1H), 2.01 – 1.87 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 163.5, 147.3, 146.3, 138.8, 137.1, 128.3, 126.9, 126.1, 126.0, 122.1, 120.5, 114.4, 77.0, 40.5, 28.1. **HRMS (ESI)** calculated for $C_{16}H_{17}NONa$ $[M+Na]^+$ *m/z* 262.1202, found 262.1208.



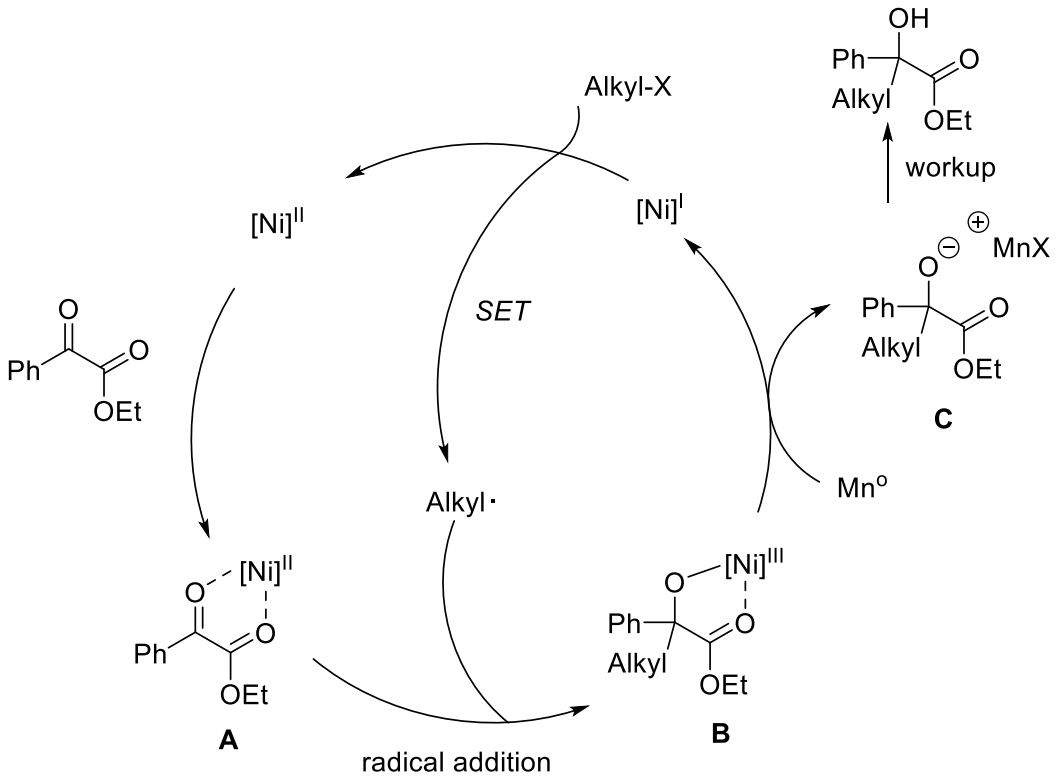
In a nitrogen-filled glove box, $NiBr_2$ (4.36 mg, 0.02 mmol), ethyl 2-oxo-2-phenylacetate (35.6 mg, 0.2 mmol), manganese powder (21.6 mg, 0.4 mmol), 6-bromohex-1-ene (65.2 mg, 0.4 mmol) and DMF (0.4 mL) were charged to a 4 mL vial equipped with a magnetic stirring bar. The reaction mixture was stirred at 70 °C for 12 h. The resulting mixture was filtered through a short pad of silica gel, eluted with ethyl acetate, and concentrated in vacuo. The crude product was purified by flash column chromatography (hexanes/EtOAc = 20:1) on silica gel to give the corresponding product as a yellow oil in 45% yield (43.2 mg, **12**:**12'** = 1.25:1).



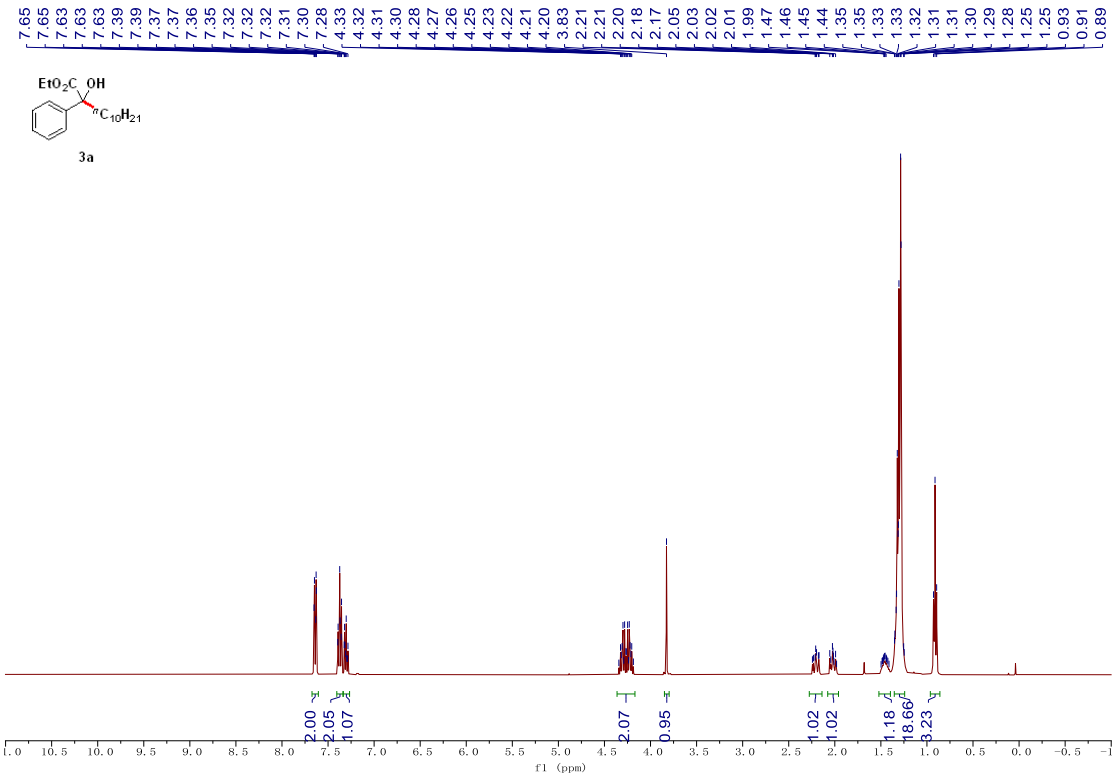
Chemical Formula: $C_{18}H_{36}NO^{\bullet}$
Exact Mass: 282.28
GC-MS found: 282.3

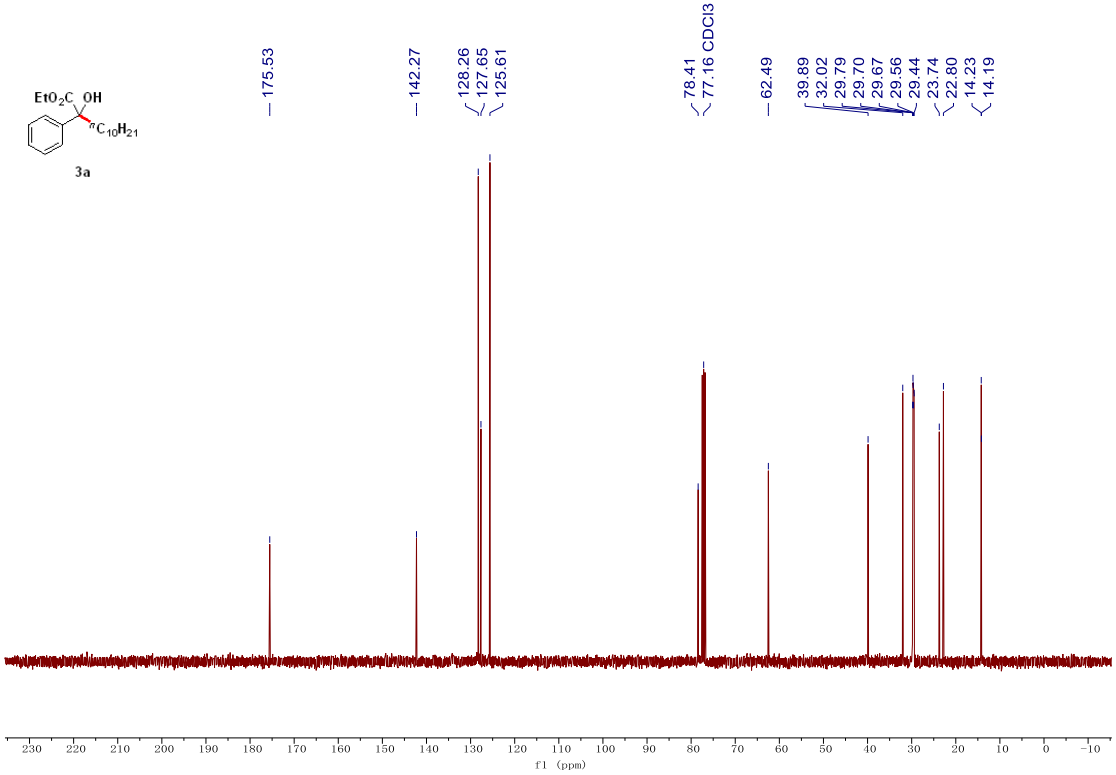
In a nitrogen-filled glove box, $NiBr_2$ (4.36 mg, 0.02 mmol), ethyl 2-oxo-2-phenylacetate (35.6mg, 0.2 mmol), manganese powder (21.6 mg, 0.4 mmol), 1-iododecane (107 mg, 0.4 mmol), 2,2,6,6 -tetramethylpiperidinyl-1-oxide (TEMPO, 62.4 mg, 0.4 mmol) and DMF (0.4 mL) were charged to a 4 mL vial equipped with a magnetic stirring bar. The reaction mixture was stirred at 70 °C for 12 h. The resulting mixture was filtered through a short pad of silica gel and the filter cake was washed with EtOAc. The TEMPO adduct was detected by GC-MS analysis (calculated 282.5, found 282.3).

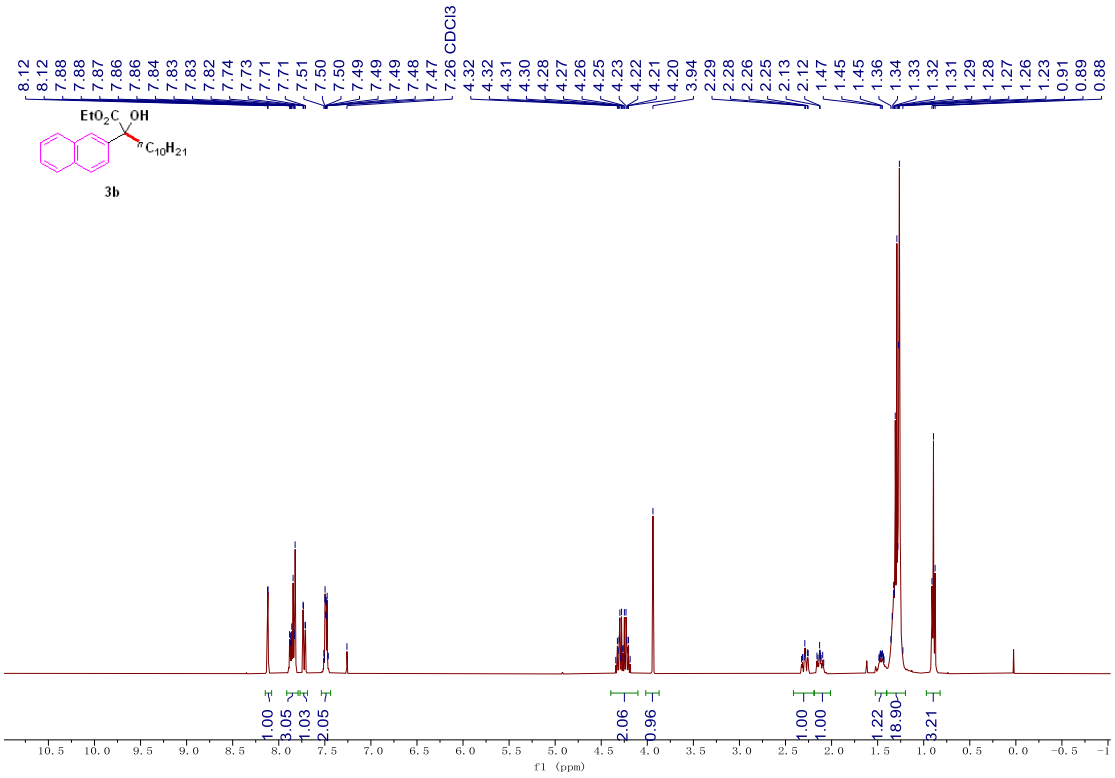
7. Proposed Mechanism



8. NMR Spectra



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7.51
7.50
7.50
7.49
7.49
7.49
7.48
7.47
7.26 CDCl3
4.32
4.32
4.31
4.30
4.28
4.27
4.26
4.25
4.23
4.22
4.21
4.20
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2.28
2.26
2.25
2.13
2.12
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1.32
1.31
1.29
1.28
1.27
1.26
1.23
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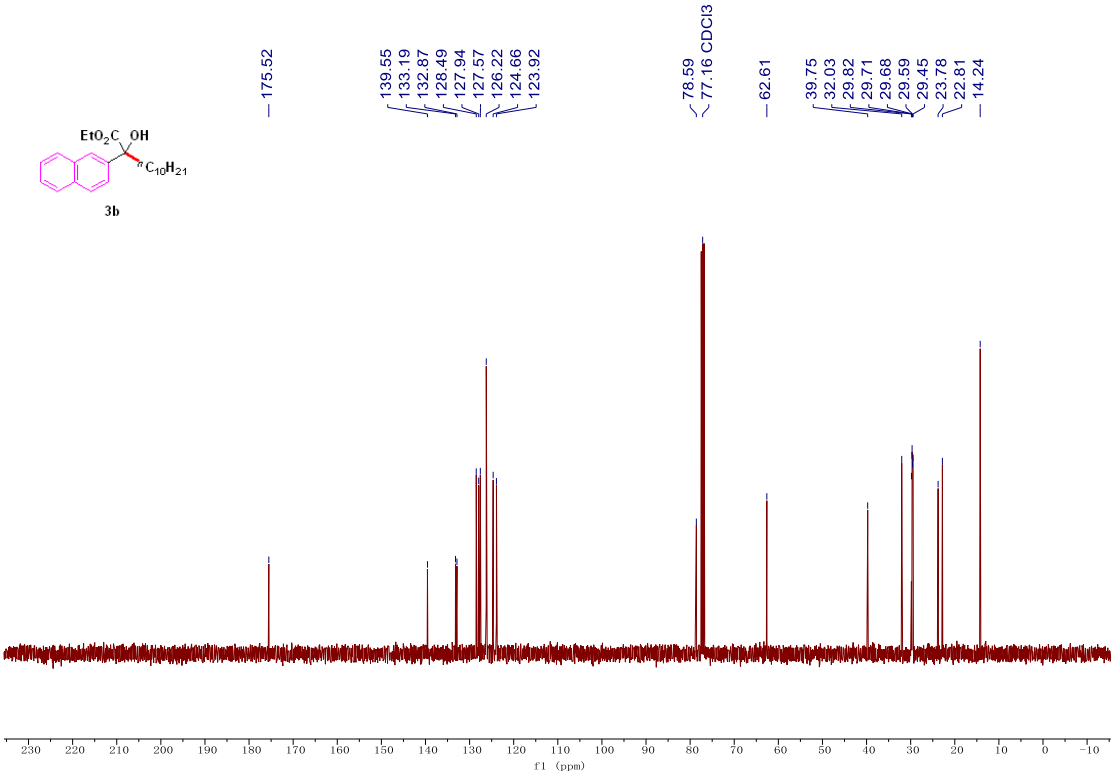
3.21
f1 (ppm)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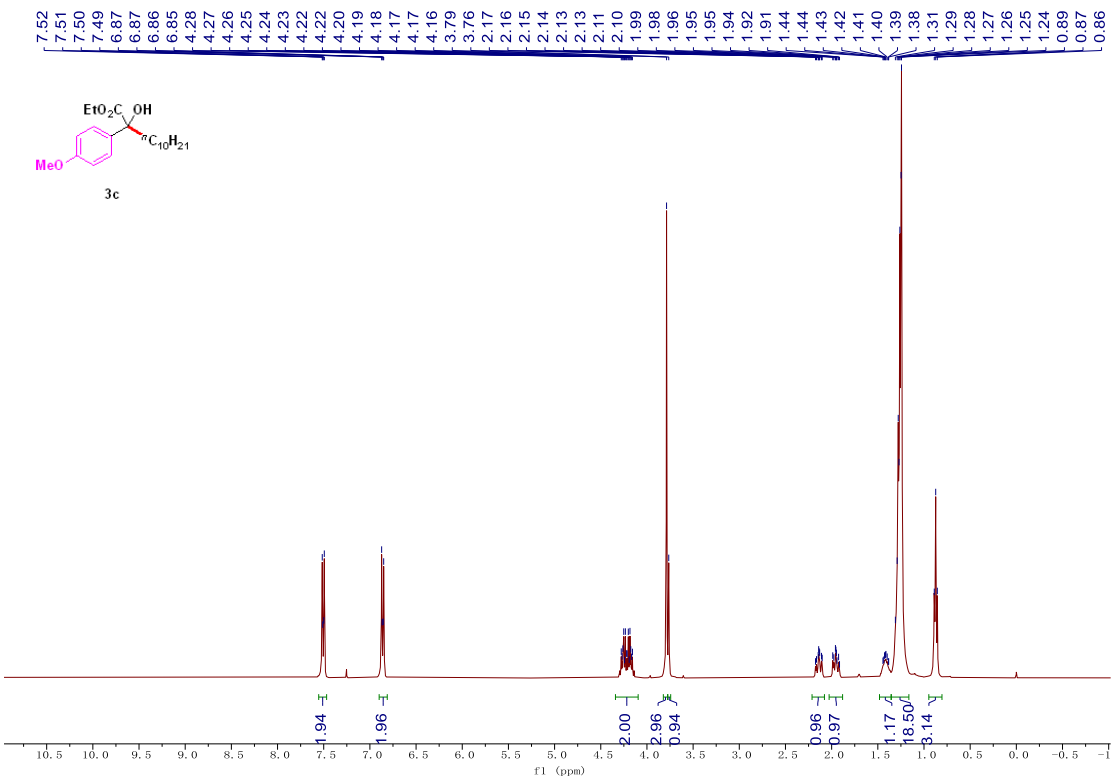
3b
f1 (ppm)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3c
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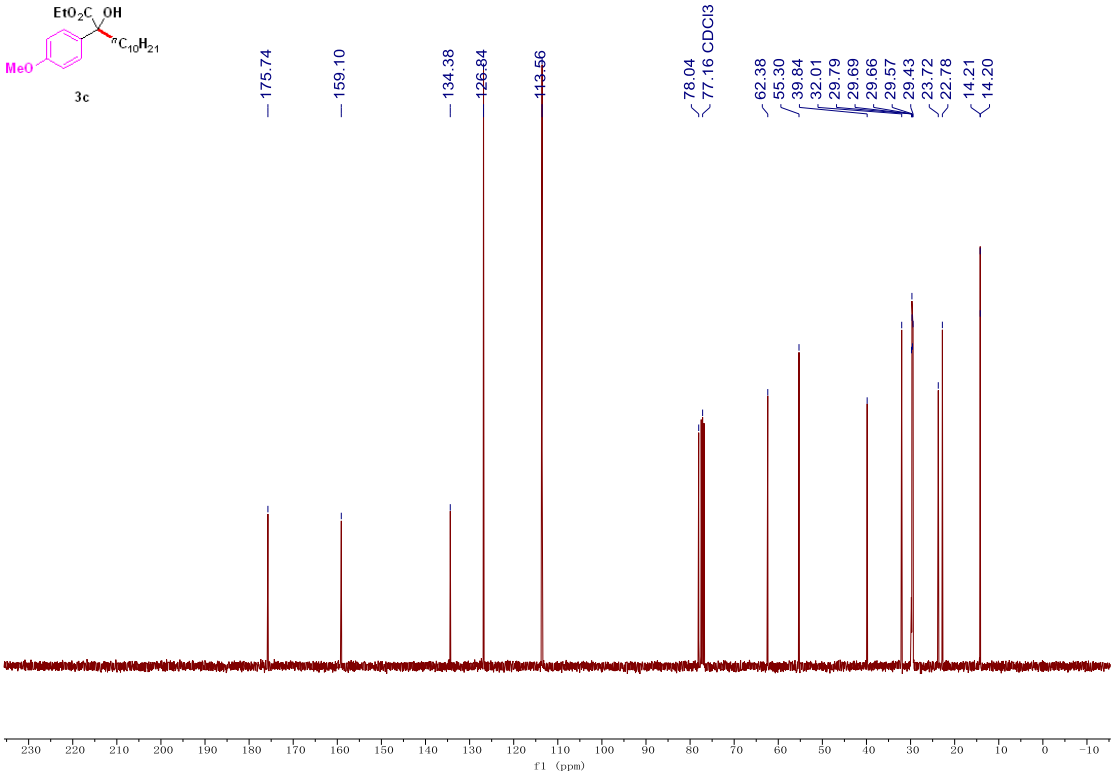
f1 (ppm)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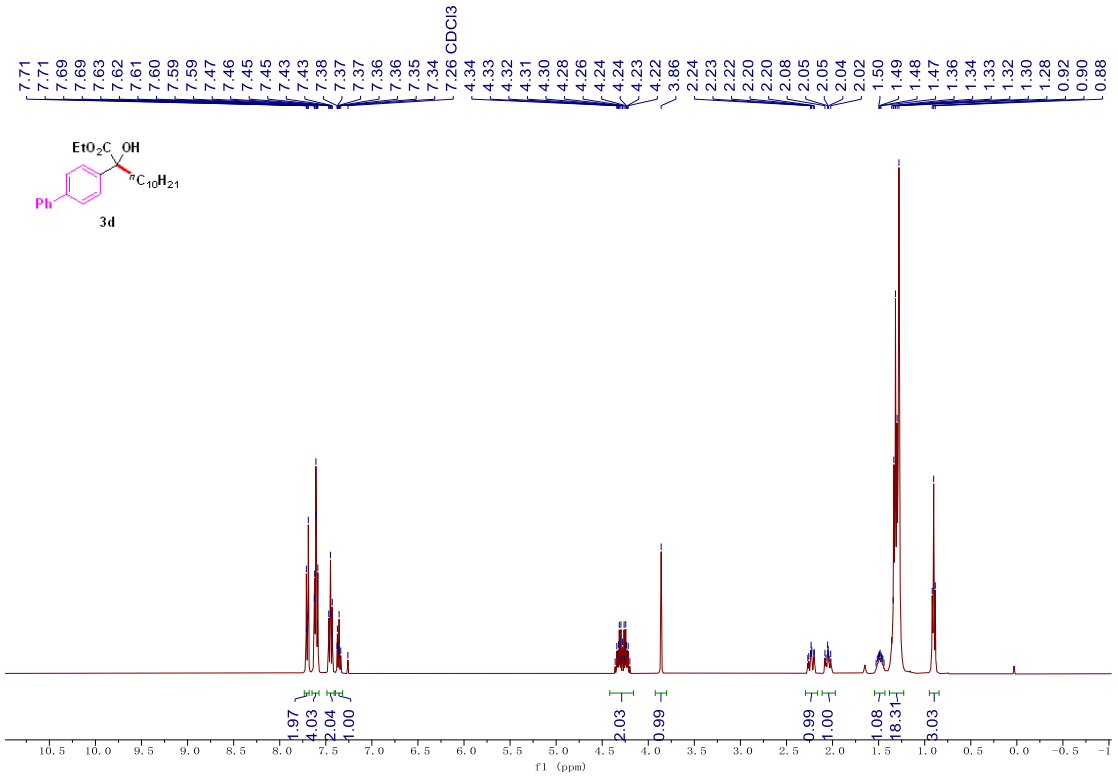
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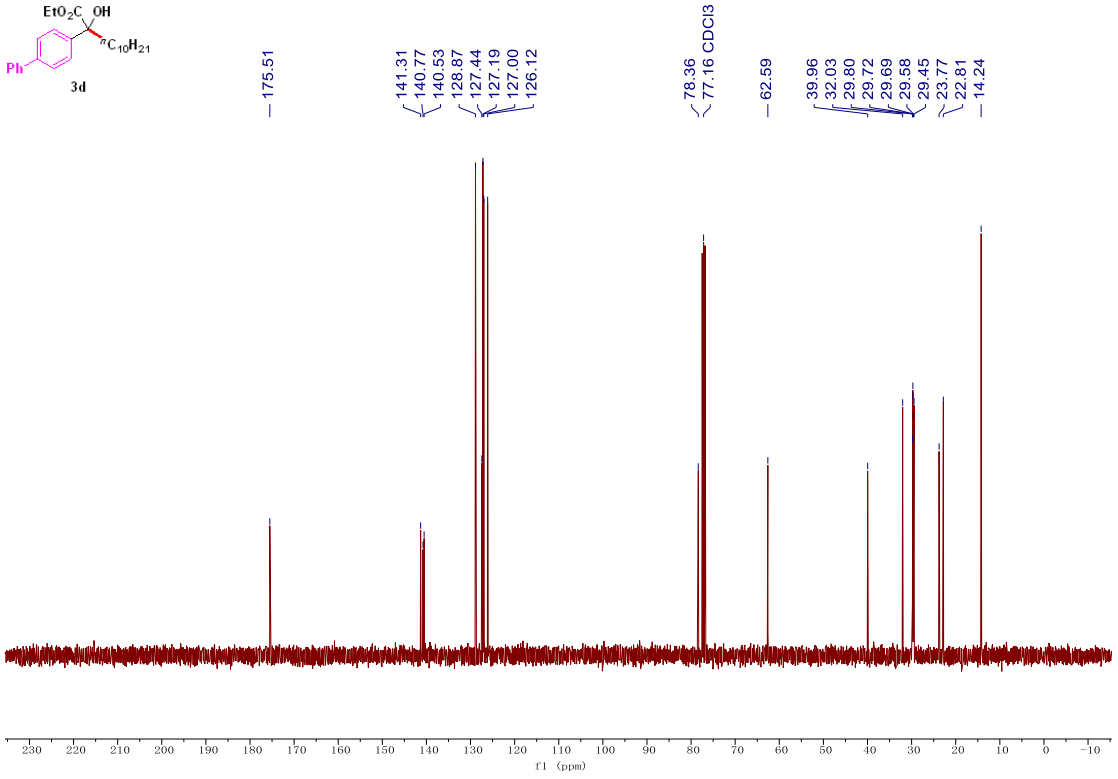
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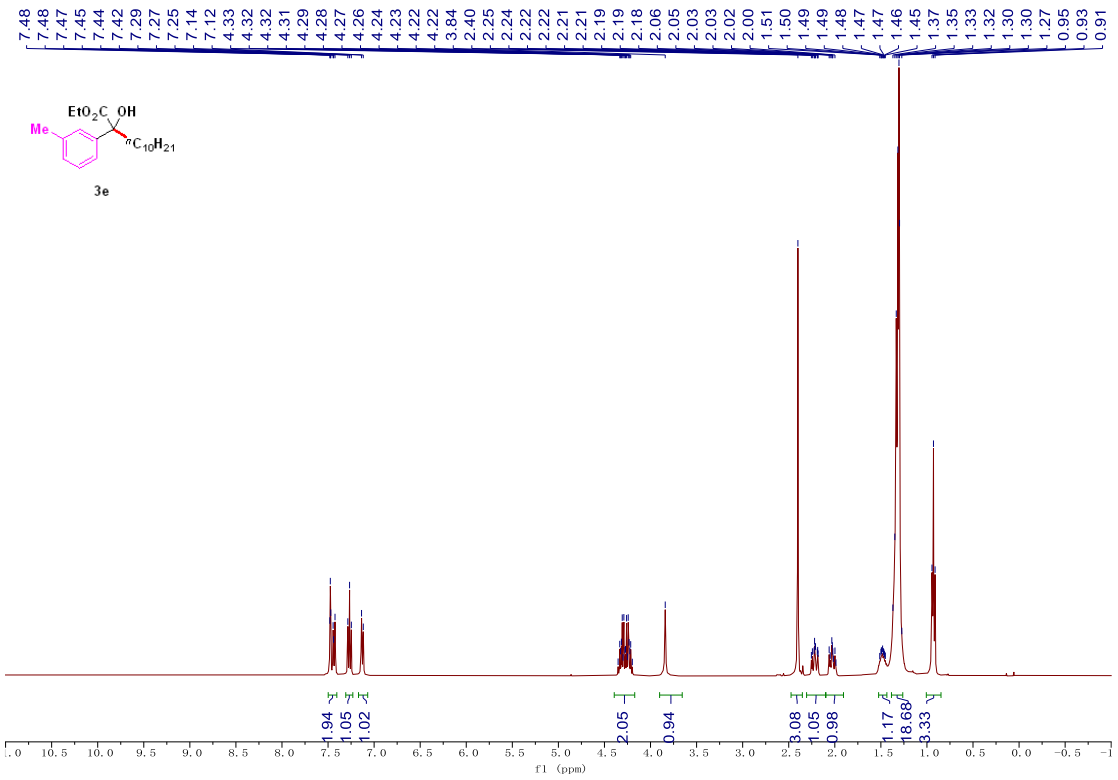
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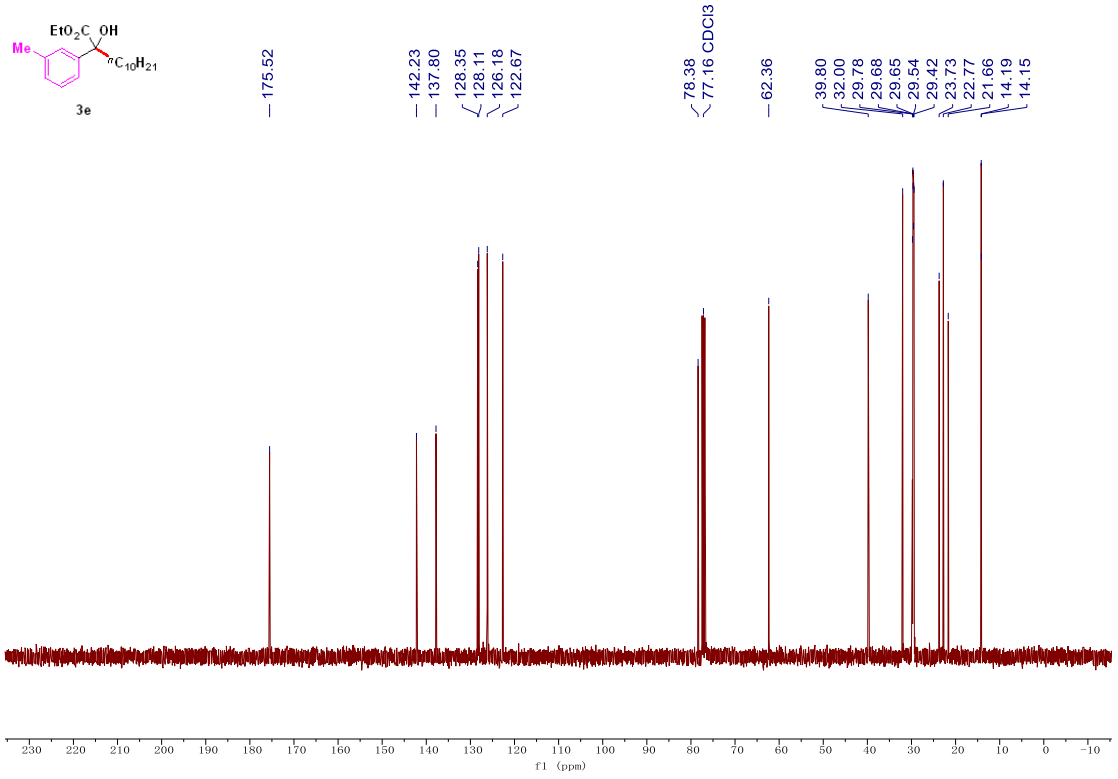
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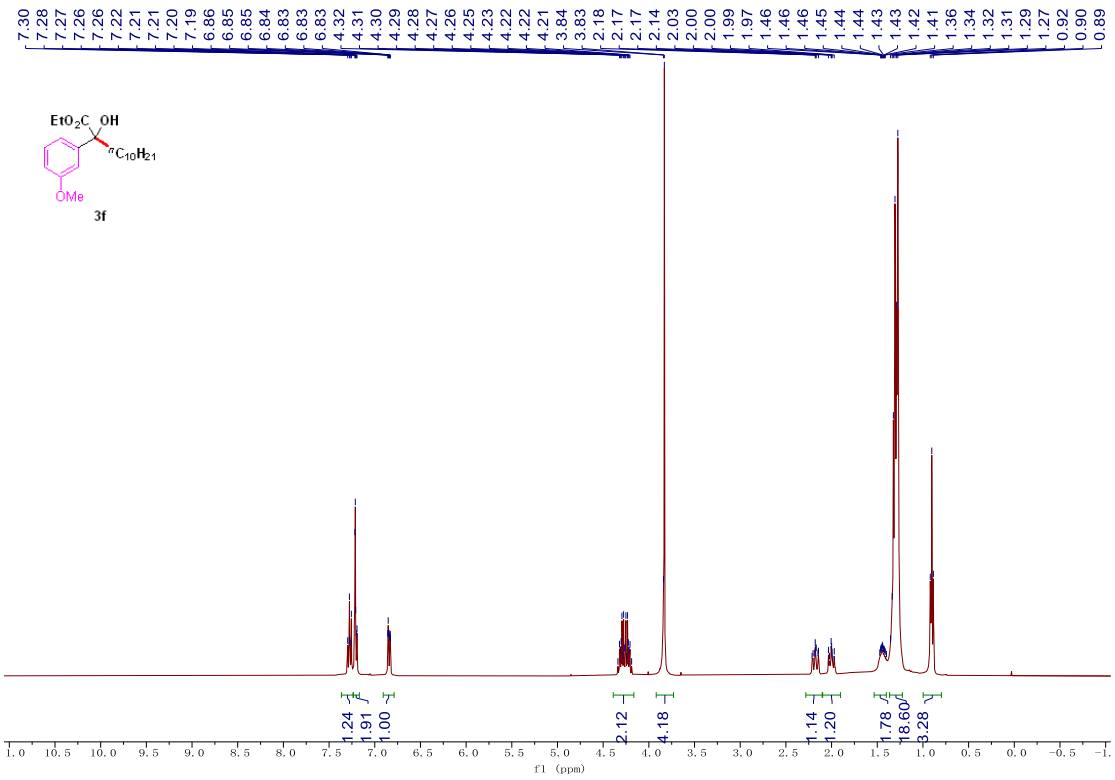
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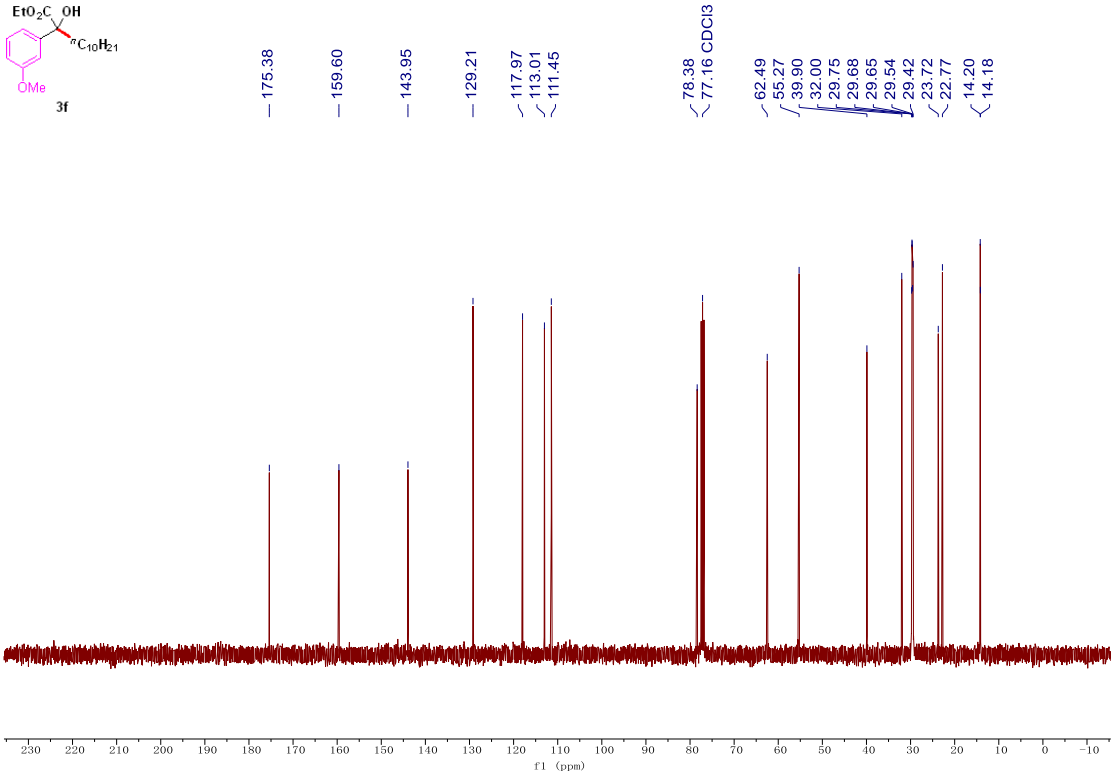
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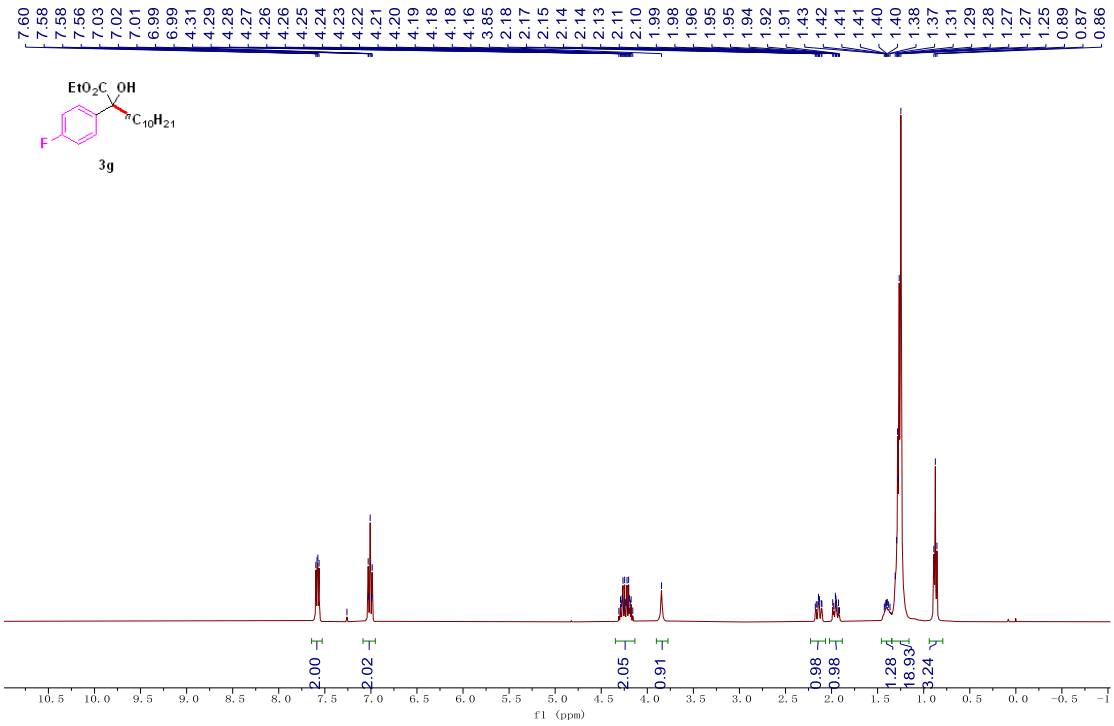
f1 (ppm)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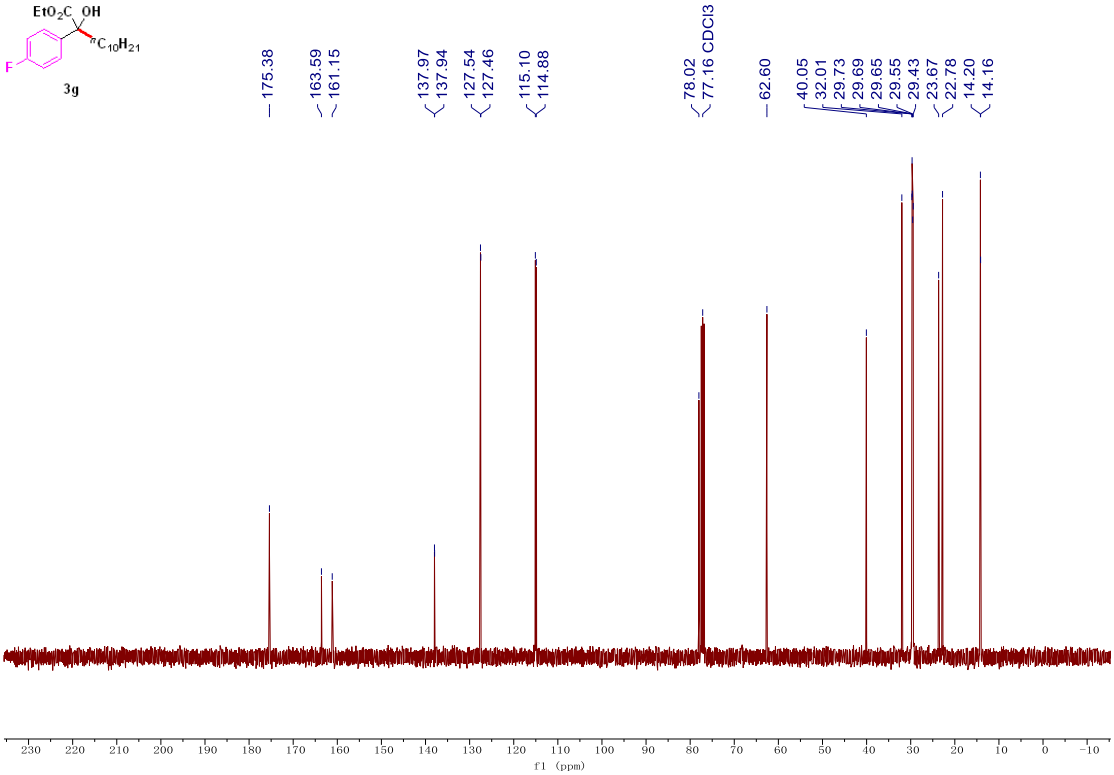
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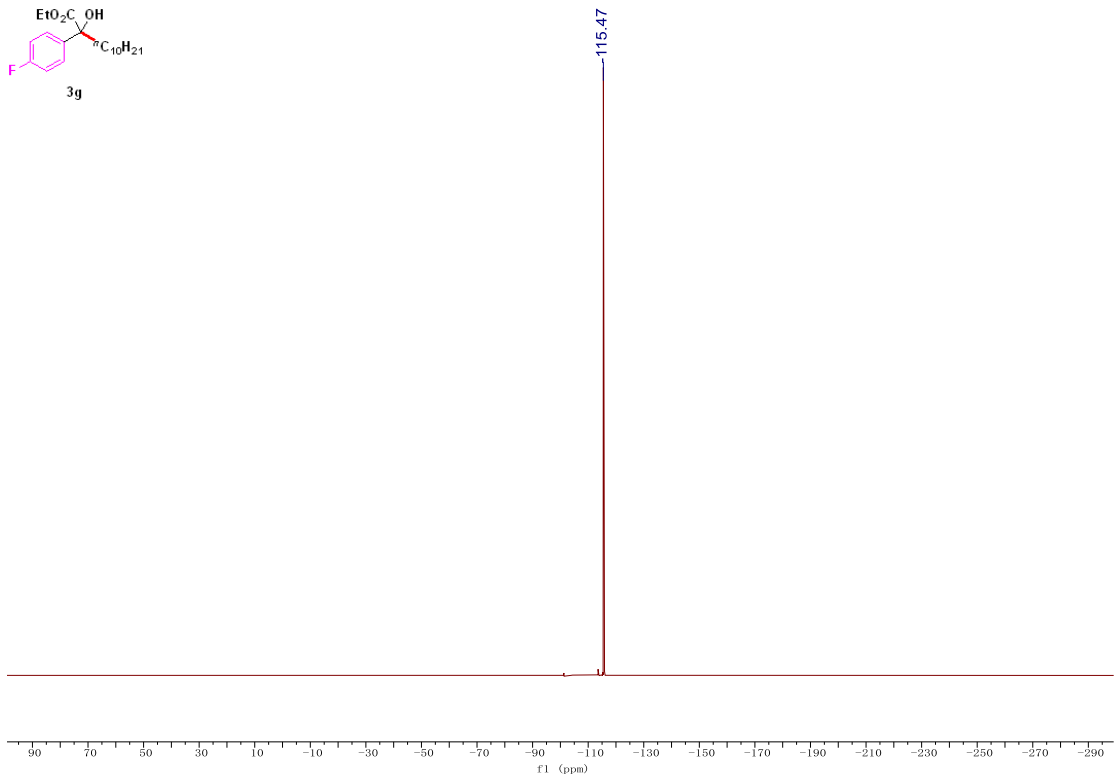
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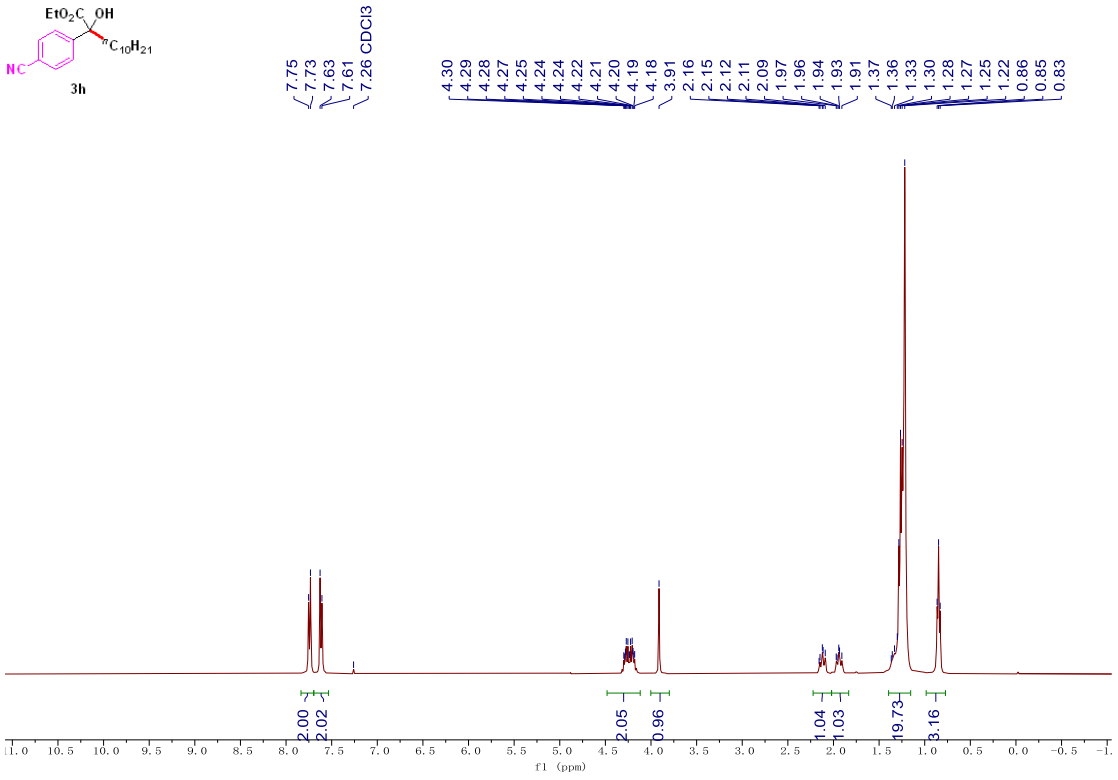
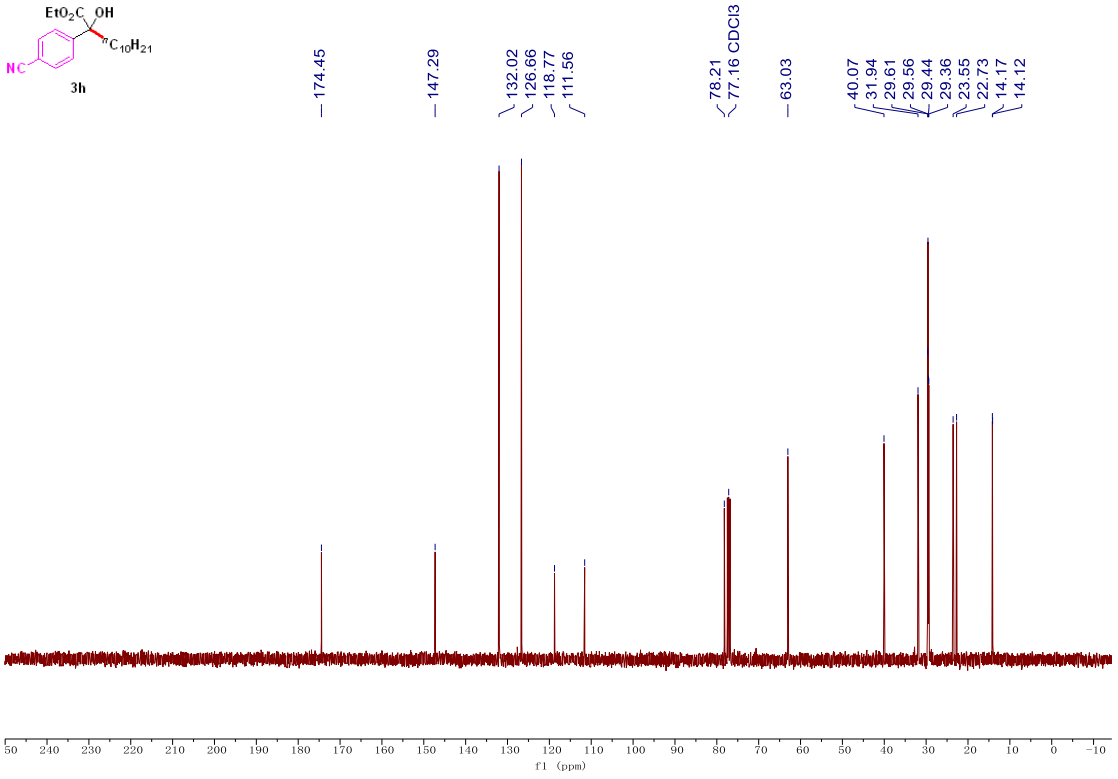
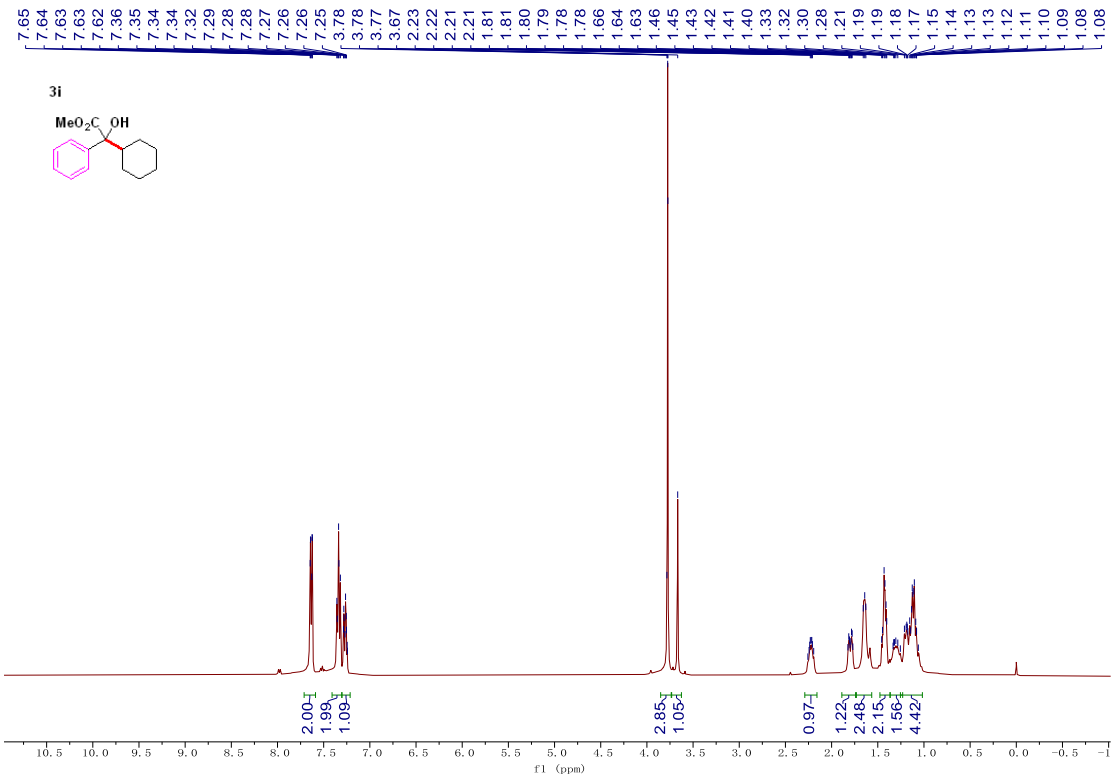
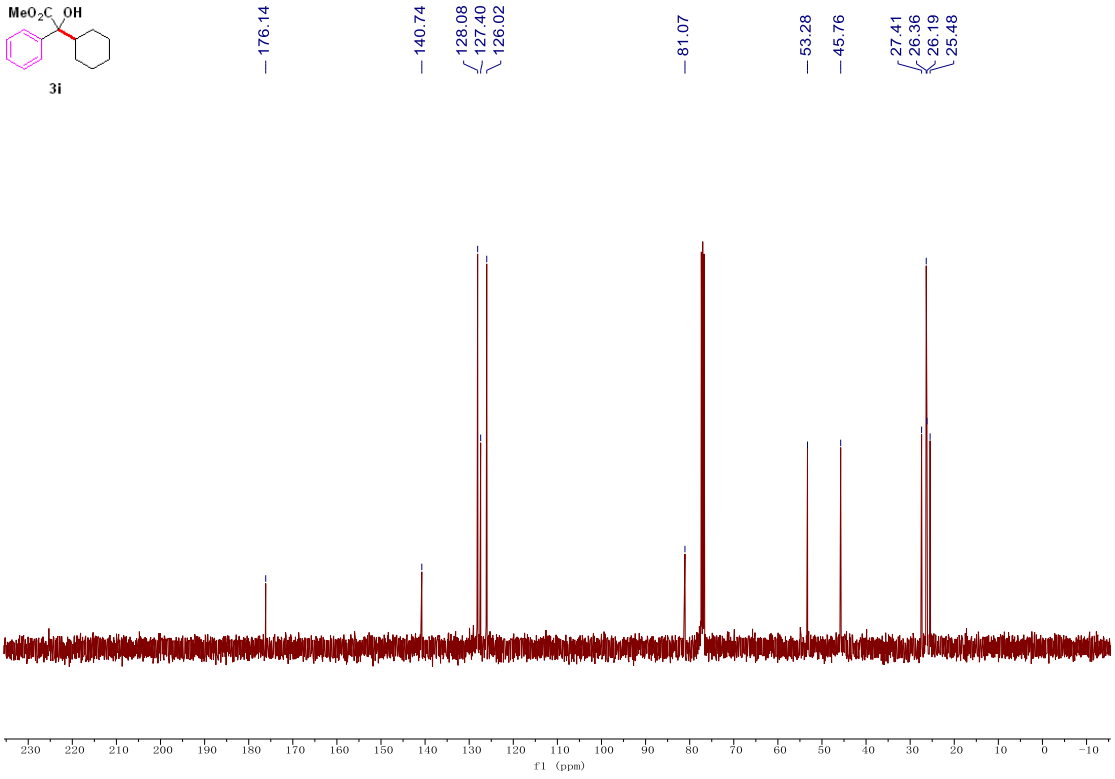
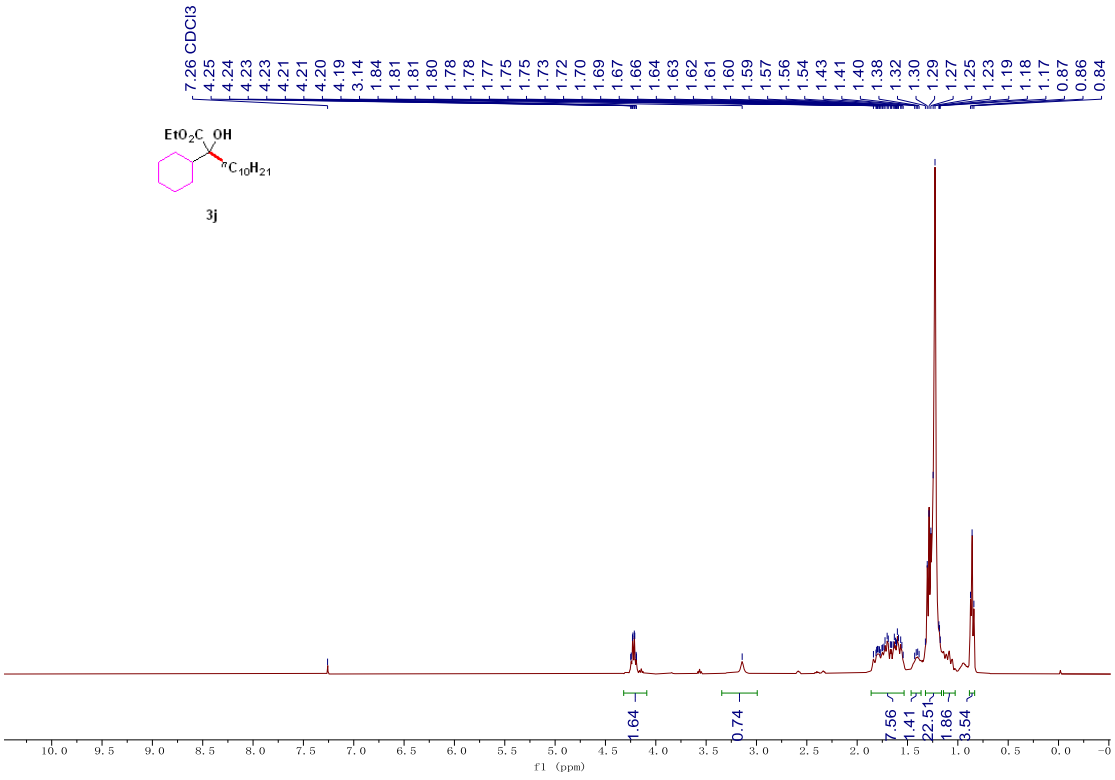
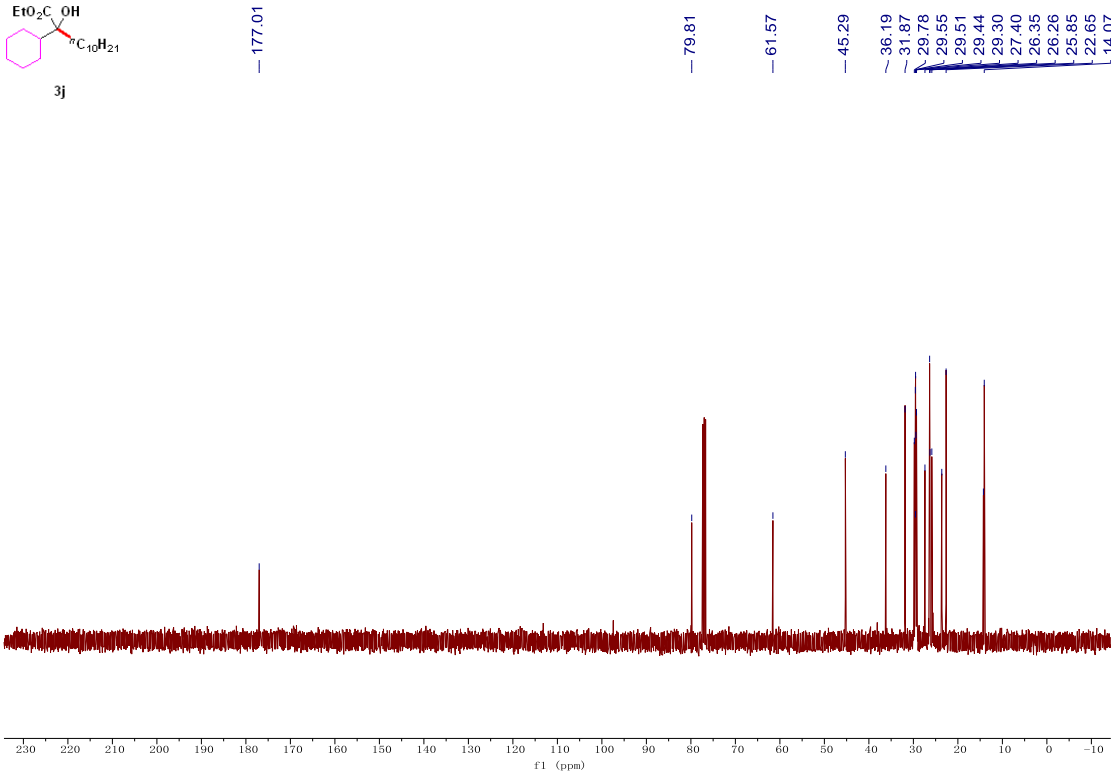

EtO2C OH
nC10H21
NC
3h
7.75
7.73
7.63
7.61
7.26 CDCl3
4.30
4.29
4.28
4.27
4.25
4.24
4.24
4.22
4.21
4.20
4.19
4.18
3.91
2.16
2.15
2.12
2.11
2.09
1.97
1.96
1.94
1.93
1.91
1.37
1.36
1.33
1.30
1.28
1.27
1.25
1.22
0.86
0.85
0.83
2.00
2.02
2.05
0.96
1.04
1.03
19.73
3.16
f1 (ppm)

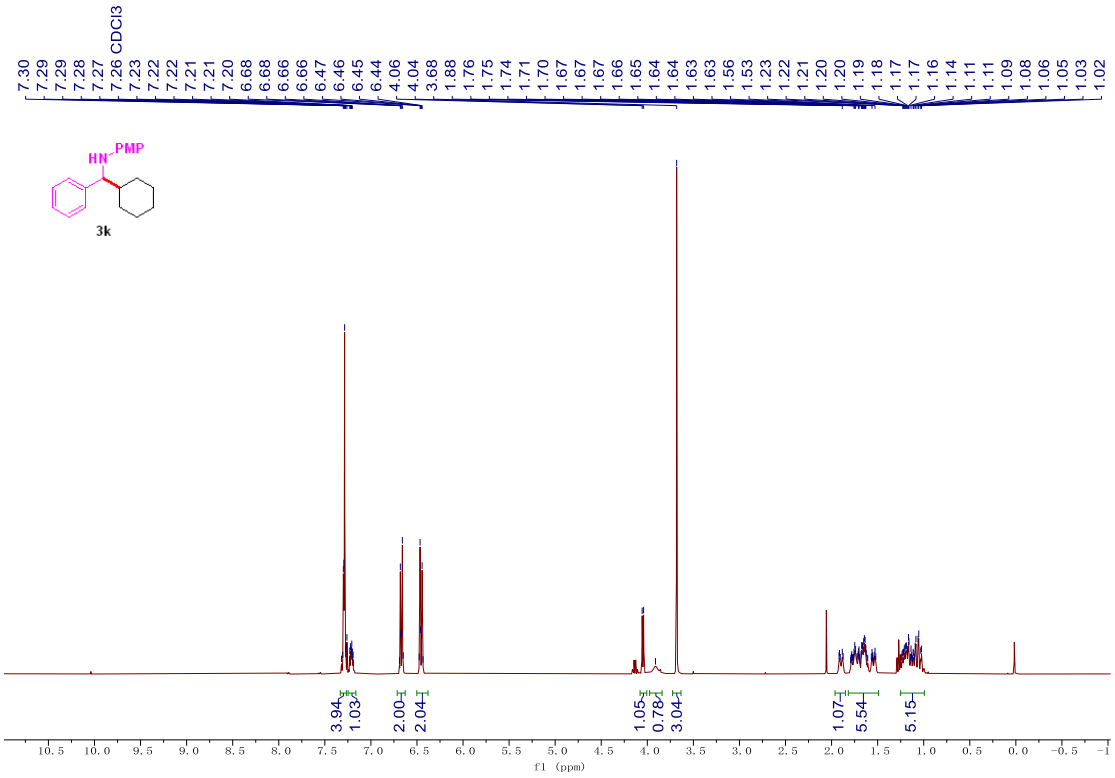

EtO2C OH
nC10H21
NC
3h
174.45
147.29
132.02
126.66
118.77
111.56
78.21
77.16 CDCl3
63.03
40.07
31.94
29.61
29.56
29.44
29.36
23.55
22.73
14.17
14.12
f1 (ppm)

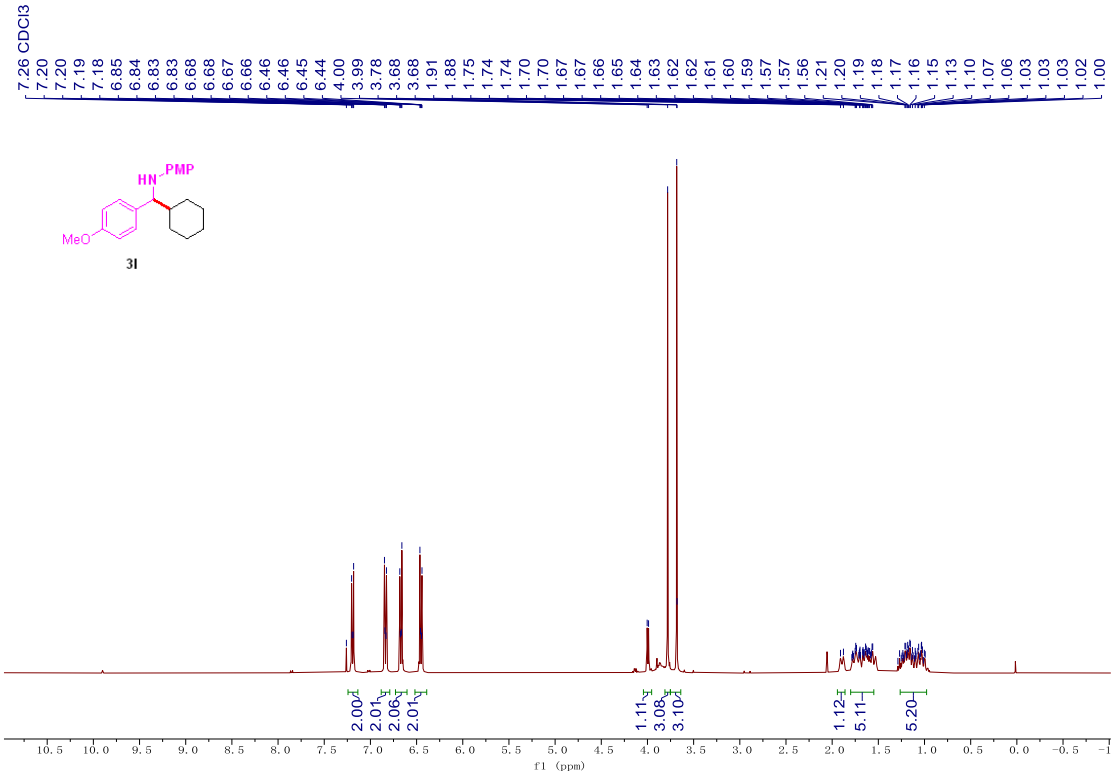

7.65 7.64 7.63 7.63 7.62 7.36 7.35 7.34 7.34 7.32 7.29 7.28 7.28 7.27 7.26 7.26 7.25 3.78 3.78 3.77 3.67 2.23 2.22 2.21 2.21 1.81 1.81 1.80 1.79 1.78 1.78 1.66 1.64 1.63 1.46 1.45 1.43 1.42 1.41 1.40 1.33 1.32 1.30 1.28 1.21 1.19 1.19 1.18 1.17 1.15 1.14 1.13 1.13 1.12 1.11 1.10 1.09 1.08 1.08
3i
MeO2C OH
2.00 1.99 1.09 2.85 1.05 0.97 1.22 2.48 2.15 1.56 4.42
f1 (ppm)

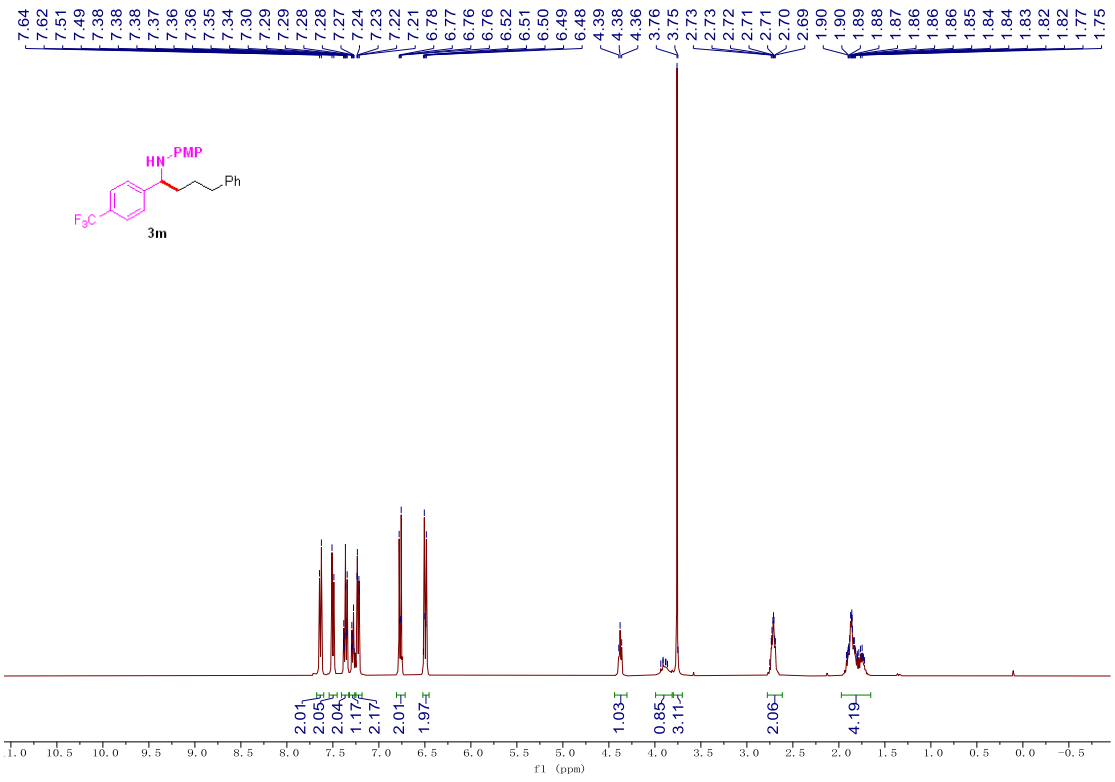

MeO2C OH
3i
176.14
140.74
128.08
127.40
126.02
81.07
53.28
45.76
27.41
26.36
26.19
25.48
f1 (ppm)

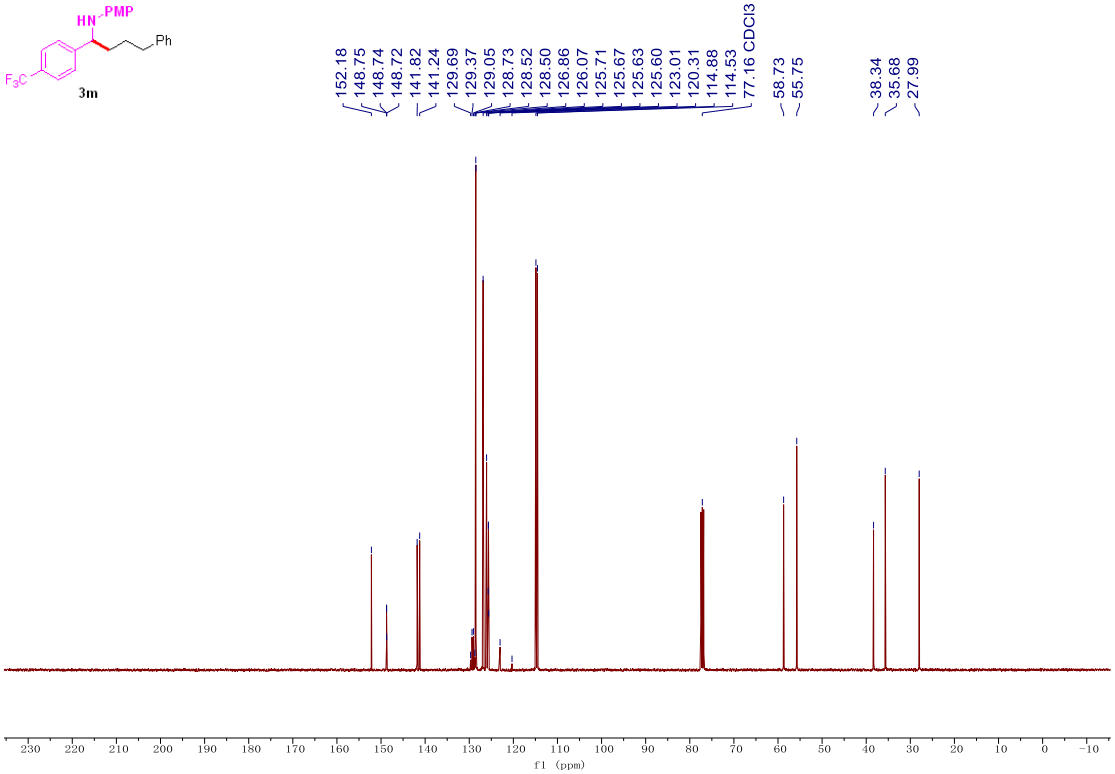

7.26 CDCl3
4.25
4.24
4.23
4.23
4.21
4.21
4.20
4.19
3.14
1.84
1.81
1.81
1.80
1.78
1.78
1.77
1.75
1.75
1.73
1.72
1.70
1.69
1.67
1.66
1.64
1.63
1.62
1.61
1.60
1.59
1.57
1.56
1.54
1.43
1.41
1.40
1.38
1.32
1.30
1.29
1.27
1.25
1.23
1.19
1.18
1.17
0.87
0.86
0.84
EtO2C OH
nC10H21
3j
1.64
0.74
7.56
1.41
22.51
1.86
3.54
f1 (ppm)

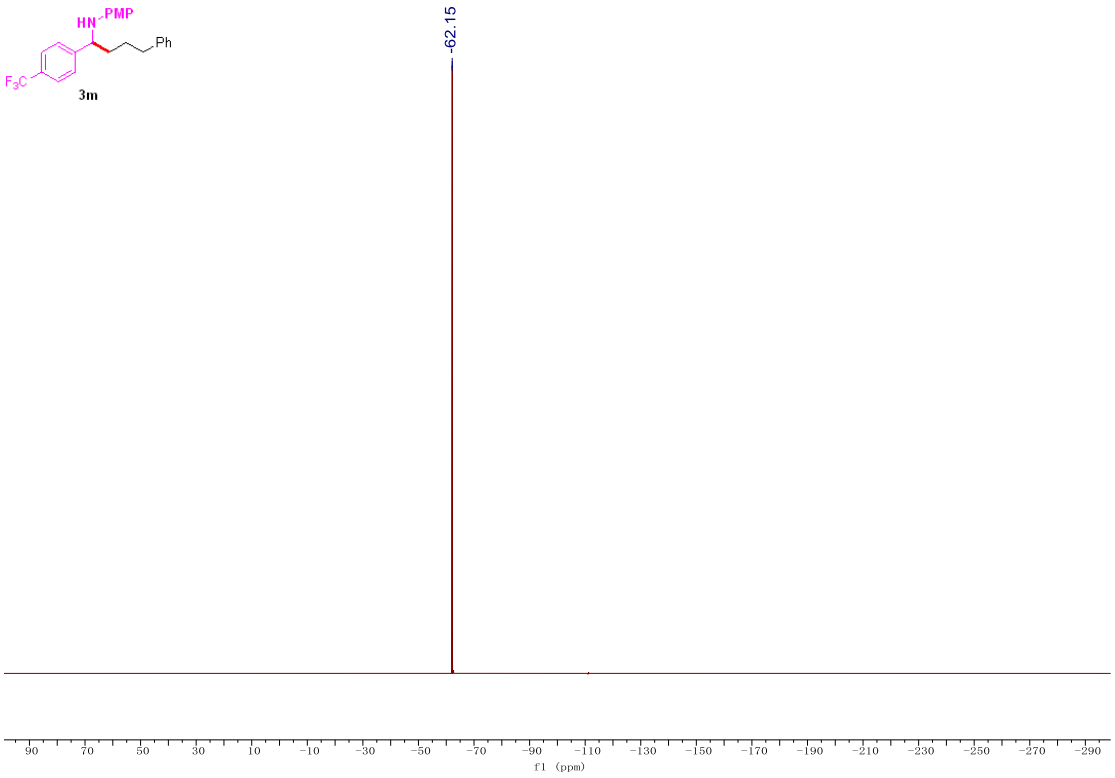

EtO2C OH
nC10H21
3j
177.01
79.81
61.57
45.29
36.19
31.87
29.78
29.55
29.51
29.44
29.30
27.40
26.35
26.26
25.85
22.65
14.07
f1 (ppm)

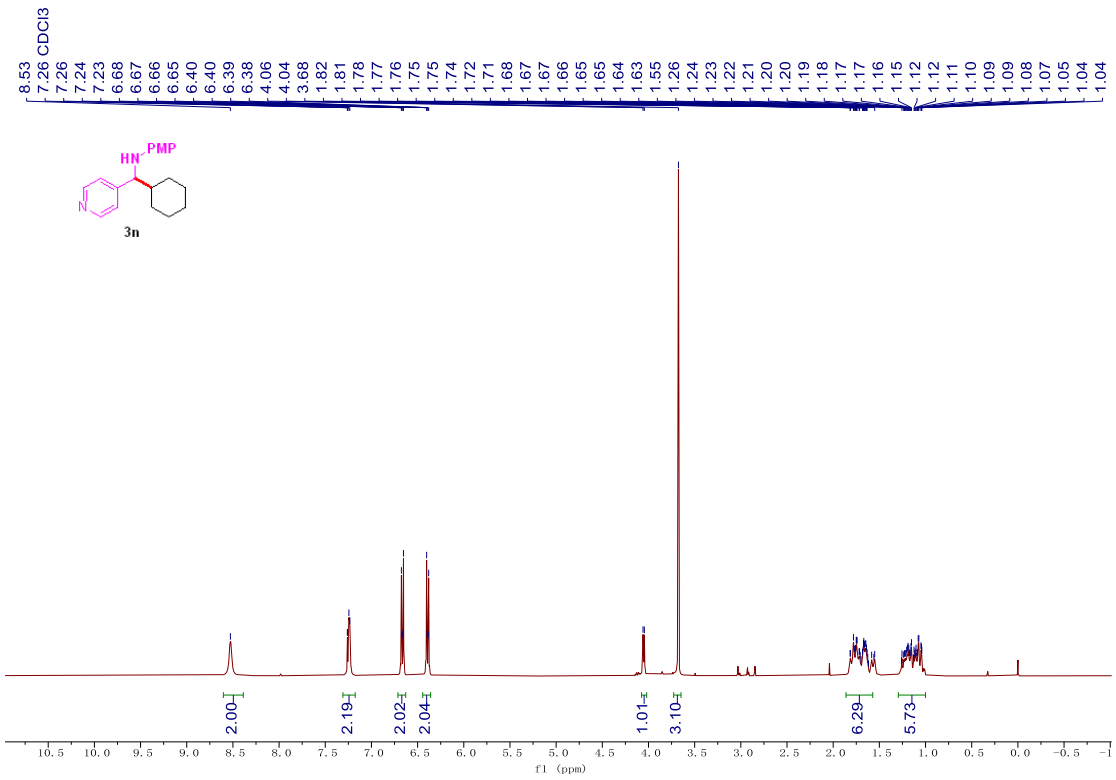

7.30 7.29 7.29 7.28 7.27 7.26 CDCl3 7.23 7.22 7.22 7.21 7.21 7.20 6.68 6.68 6.66 6.66 6.47 6.46 6.45 6.44 4.06 4.04 3.68 1.88 1.76 1.75 1.74 1.71 1.70 1.67 1.67 1.67 1.66 1.65 1.64 1.64 1.63 1.63 1.56 1.53 1.23 1.22 1.21 1.20 1.20 1.19 1.18 1.17 1.16 1.14 1.11 1.11 1.09 1.08 1.06 1.05 1.03 1.02
HN PMP
3k
3.94 1.03 2.00 2.04 1.05 0.78 3.04 1.07 5.54 5.15
f1 (ppm)

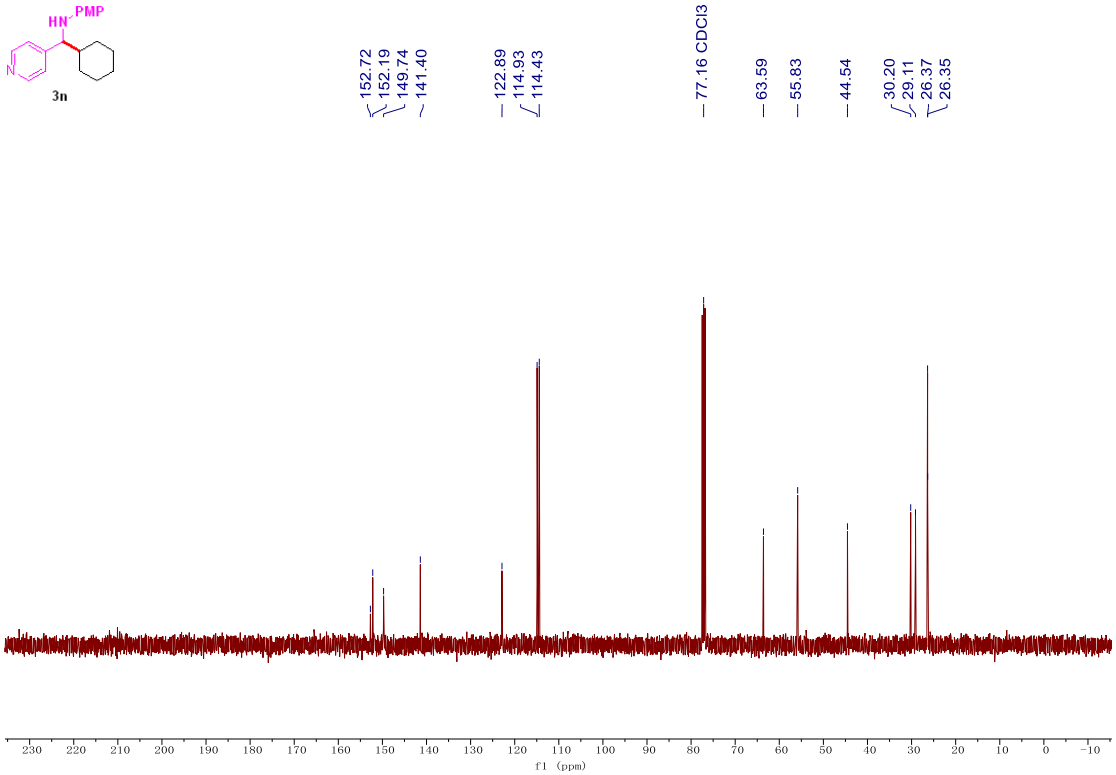

7.26 CDCl3 7.20 7.20 7.19 7.18 6.85 6.84 6.83 6.83 6.68 6.68 6.67 6.66 6.46 6.46 6.45 6.44 4.00 3.99 3.78 3.68 3.68 1.91 1.88 1.75 1.74 1.74 1.70 1.70 1.67 1.67 1.66 1.65 1.64 1.63 1.62 1.62 1.61 1.60 1.59 1.57 1.57 1.56 1.21 1.20 1.19 1.18 1.17 1.16 1.15 1.13 1.10 1.07 1.06 1.03 1.03 1.03 1.02 1.00
HN PMP
MeO
3l
2.00 2.01 2.06 2.01 1.11 3.08 3.10 1.12 5.11 5.20
f1 (ppm)

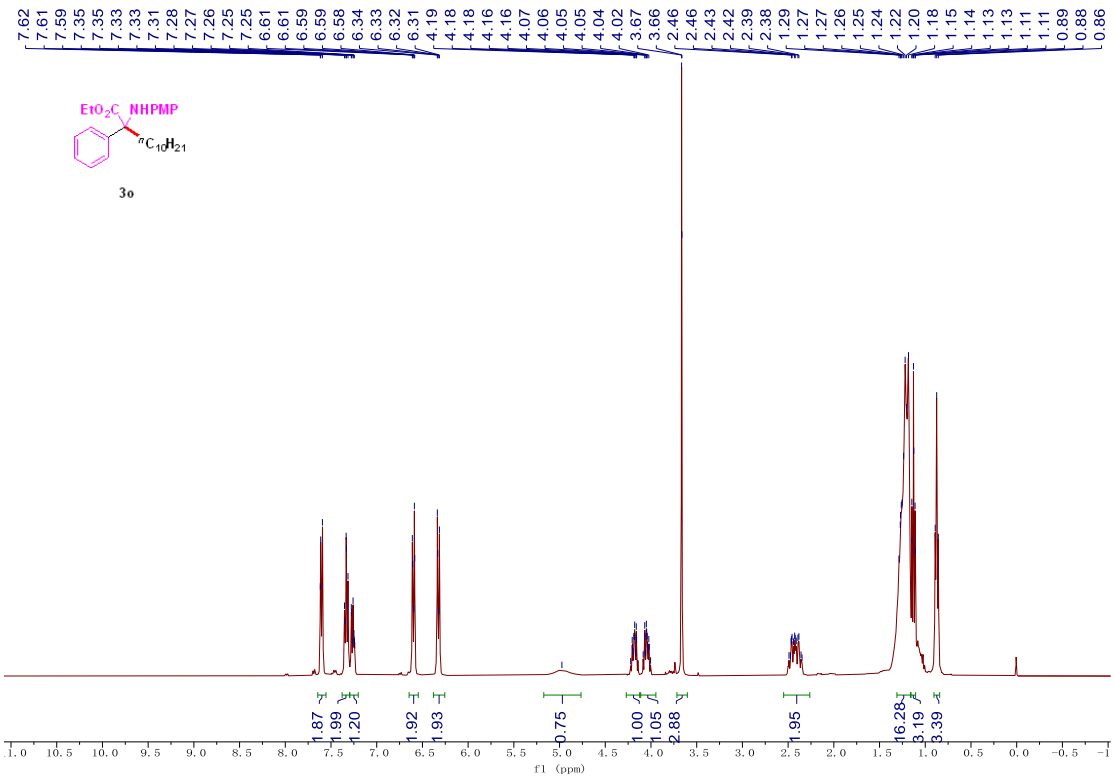

7.64 7.62 7.51 7.49 7.38 7.38 7.38 7.37 7.36 7.36 7.35 7.34 7.30 7.29 7.29 7.28 7.28 7.27 7.24 7.23 7.22 7.21 6.78 6.77 6.76 6.76 6.52 6.51 6.50 6.49 6.48 4.39 4.38 4.36 3.76 3.75 2.73 2.73 2.72 2.71 2.71 2.70 2.69 1.90 1.90 1.89 1.88 1.87 1.86 1.86 1.86 1.85 1.84 1.84 1.83 1.82 1.82 1.77 1.75
HN PMP
Ph
F3C
3m
2.01 2.05 2.04 1.17 2.17 2.01 1.97 1.03 0.85 3.11 2.06 4.19
f1 (ppm)

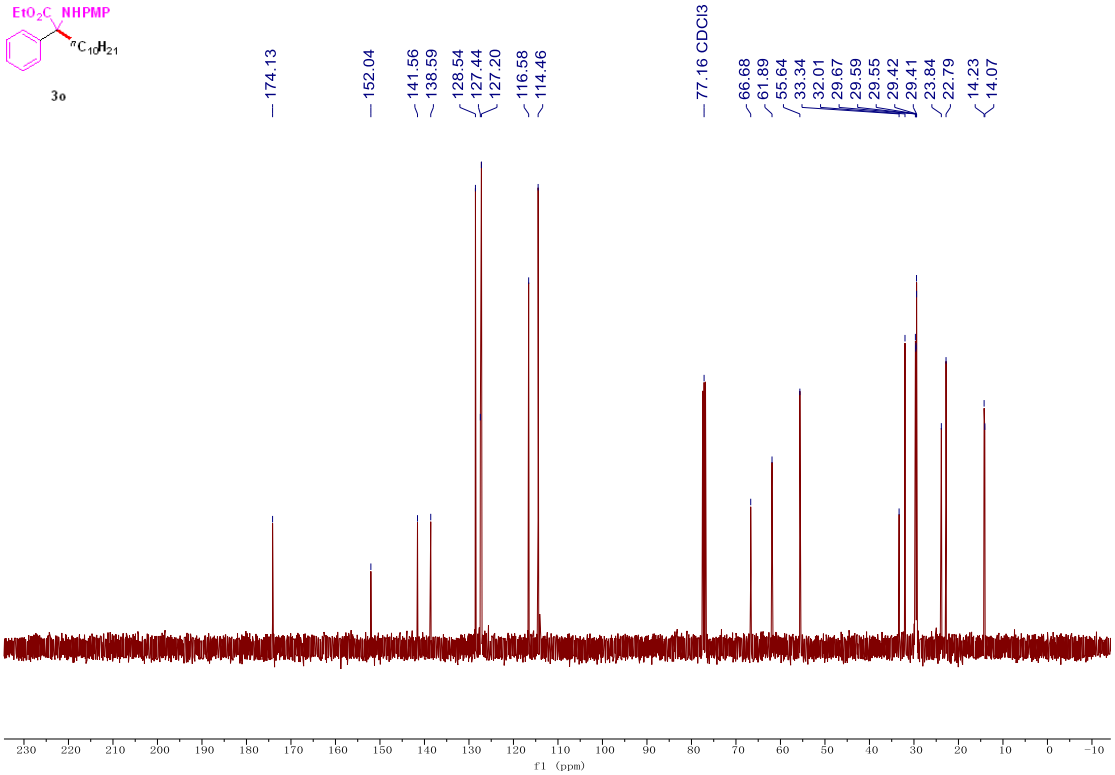

HN PMP
Ph
F3C
3m
152.18 148.75 148.74 148.72 141.82 141.24 129.69 129.37 129.05 128.73 128.52 128.50 126.86 126.07 125.71 125.67 125.63 125.60 123.01 120.31 114.88 114.53 77.16 CDCl3 58.73 55.75 38.34 35.68 27.99
f1 (ppm)

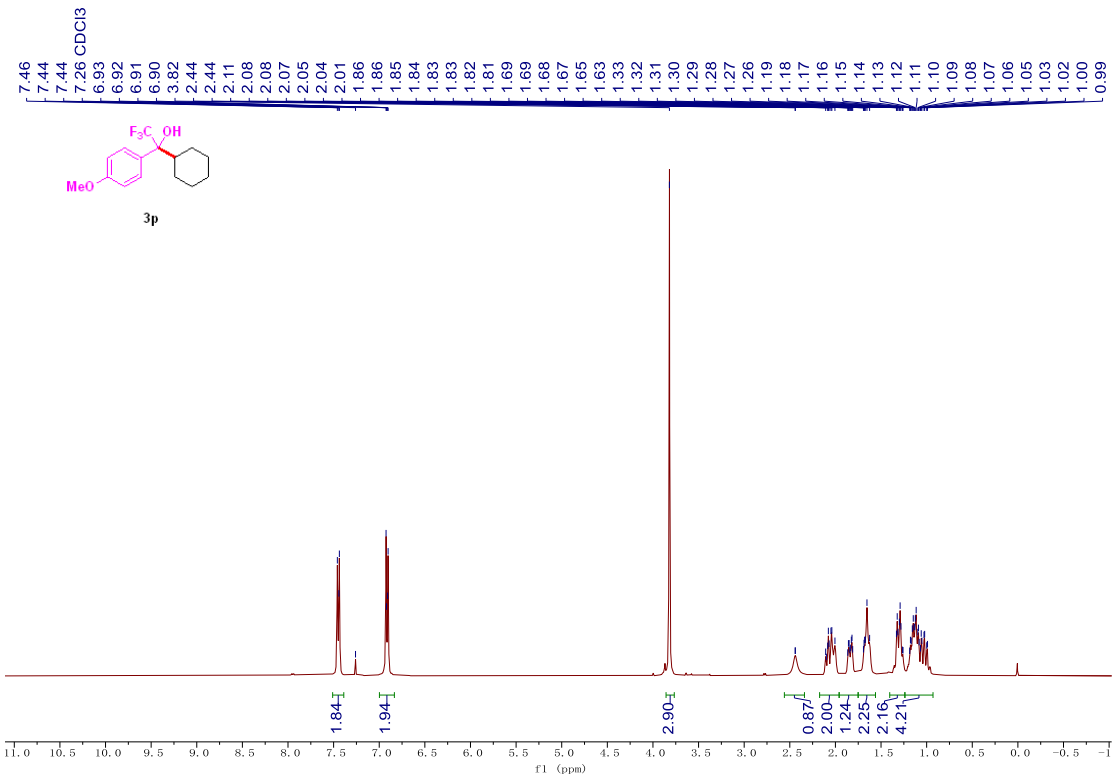

HN
PMP
Ph
F3C
3m
-62.15
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

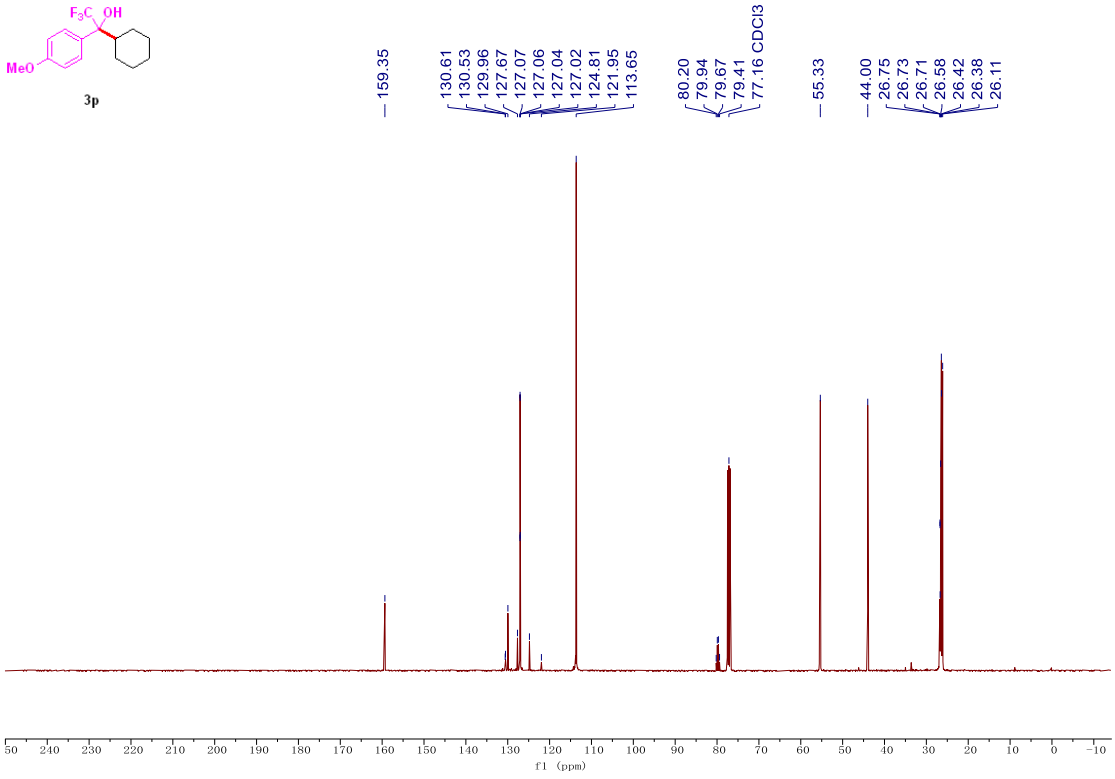

8.53
7.26 CDCl3
7.26
7.24
7.23
6.68
6.67
6.66
6.65
6.40
6.40
6.39
6.38
4.06
4.04
3.68
1.82
1.81
1.78
1.77
1.76
1.75
1.75
1.74
1.72
1.71
1.68
1.67
1.67
1.66
1.65
1.65
1.64
1.63
1.55
1.26
1.24
1.23
1.22
1.21
1.20
1.20
1.19
1.18
1.17
1.17
1.16
1.15
1.12
1.12
1.11
1.10
1.09
1.09
1.08
1.07
1.05
1.04
1.04
HN
PMP
N
3n
2.00
2.19
2.02
2.04
1.01
3.10
6.29
5.73
f1 (ppm)

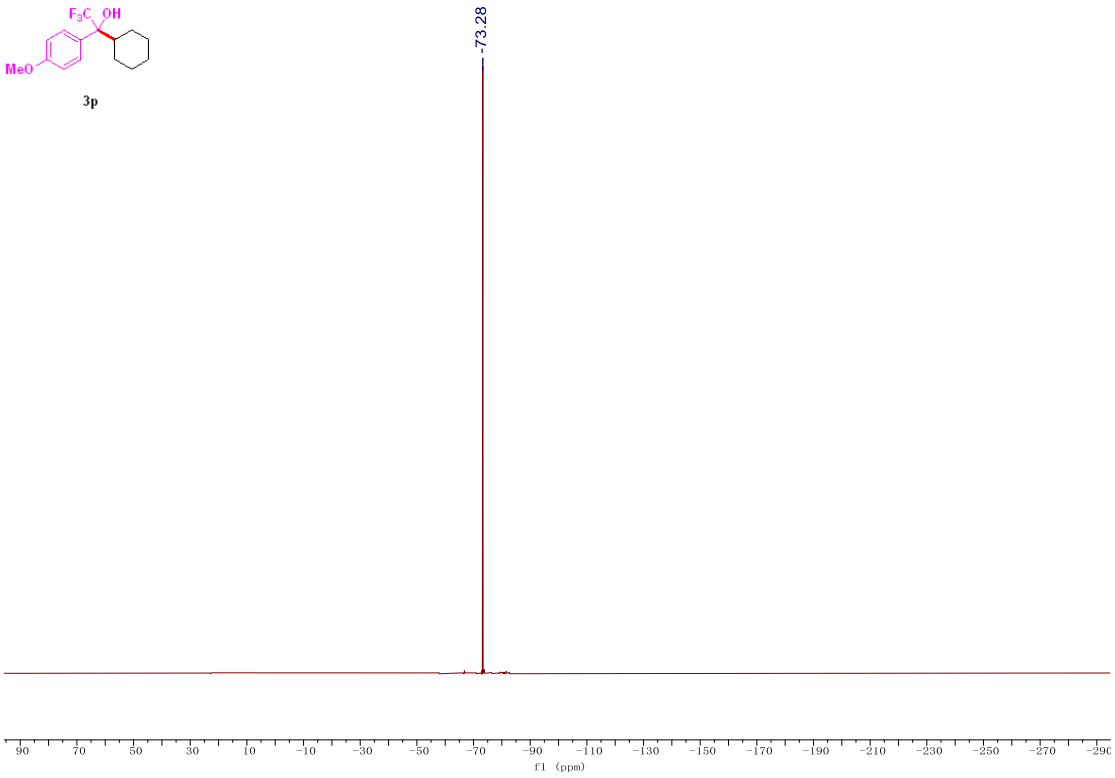

HN
PMP
N
3n
152.72
152.19
149.74
141.40
122.89
114.93
114.43
77.16 CDCl3
63.59
55.83
44.54
30.20
29.11
26.37
26.35
f1 (ppm)

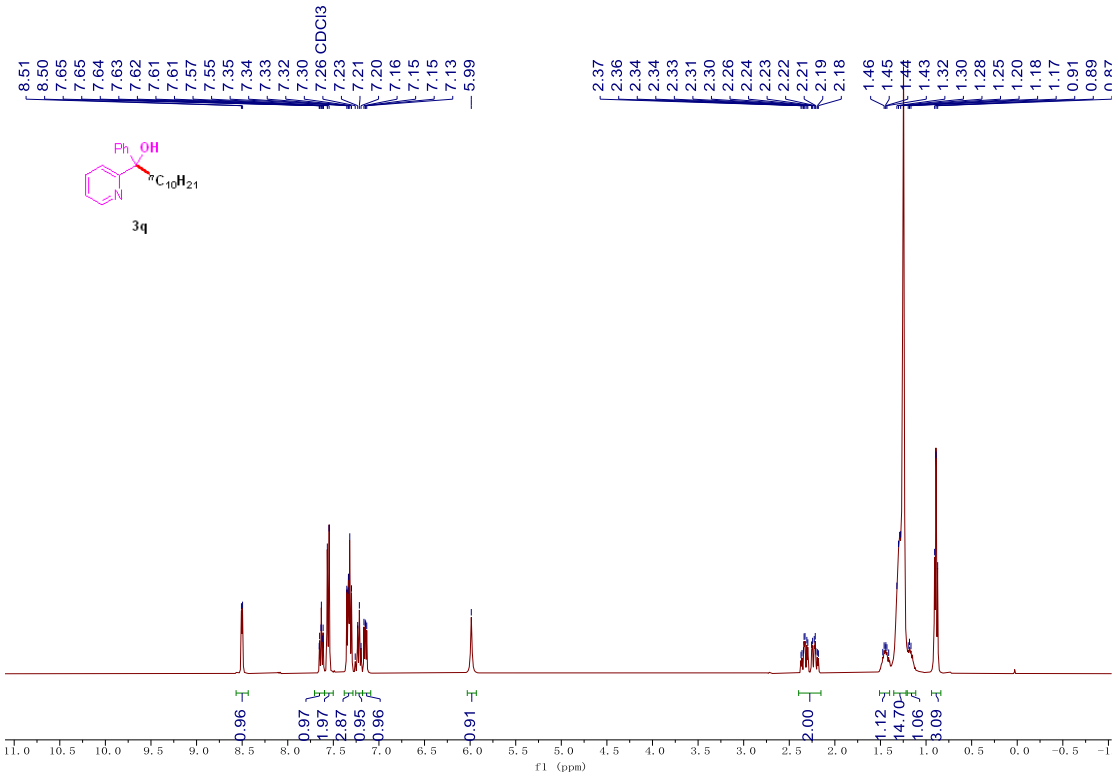

7.62 7.61 7.59 7.35 7.35 7.33 7.33 7.31 7.28 7.27 7.26 7.25 7.25 6.61 6.61 6.59 6.59 6.58 6.34 6.33 6.32 6.31 4.19 4.18 4.18 4.16 4.16 4.07 4.06 4.05 4.05 4.04 4.02 3.67 3.66 2.46 2.46 2.43 2.42 2.39 2.38 1.29 1.27 1.27 1.26 1.25 1.24 1.22 1.20 1.18 1.15 1.14 1.13 1.13 1.11 1.11 0.89 0.88 0.86
EtO2C NHPMP
nC10H21
3o
1.87 1.99 1.20 1.92 1.93 0.75 1.00 1.05 2.88 1.95 16.28 3.19 3.39
f1 (ppm)


EtO2C NHPMP
nC10H21
3o
174.13 152.04 141.56 138.59 128.54 127.44 127.20 116.58 114.46 77.16 CDCl3 66.68 61.89 55.64 33.34 32.01 29.67 29.59 29.55 29.42 29.41 23.84 22.79 14.23 14.07
f1 (ppm)


7.46
7.44
7.44
7.26 CDCl3
6.93
6.92
6.91
6.90
3.82
2.44
2.44
2.11
2.08
2.08
2.07
2.05
2.04
2.01
1.86
1.86
1.85
1.84
1.83
1.83
1.82
1.81
1.69
1.69
1.68
1.67
1.65
1.63
1.33
1.32
1.31
1.30
1.29
1.28
1.27
1.26
1.19
1.18
1.17
1.16
1.15
1.14
1.13
1.12
1.11
1.10
1.09
1.08
1.07
1.06
1.05
1.03
1.02
1.00
0.99
F3C
OH
MeO
3p
1.84
1.94
2.90
0.87
2.00
1.24
2.25
2.16
4.21
f1 (ppm)


F3C
OH
MeO
3p
159.35
130.61
130.53
129.96
127.67
127.07
127.06
127.04
127.02
124.81
121.95
113.65
80.20
79.94
79.67
79.41
77.16 CDCl3
55.33
44.00
26.75
26.73
26.71
26.58
26.42
26.38
26.11
f1 (ppm)


F3C
OH
MeO
3p
-73.28
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)


8.51
8.50
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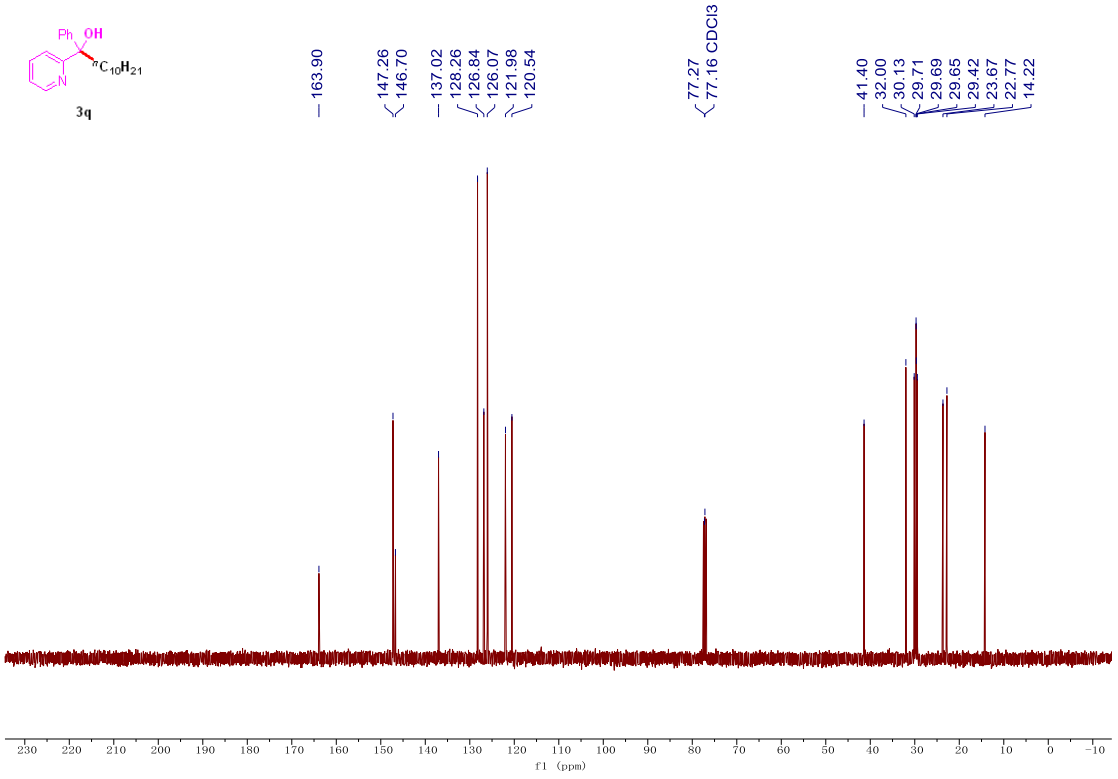
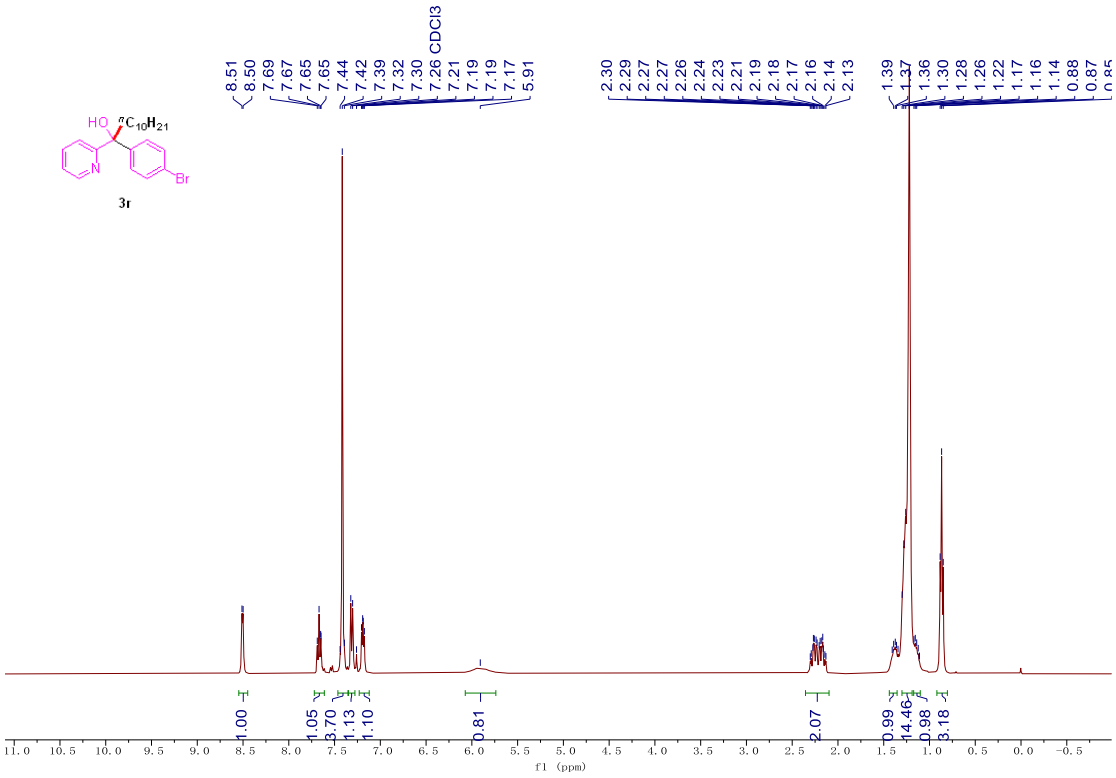
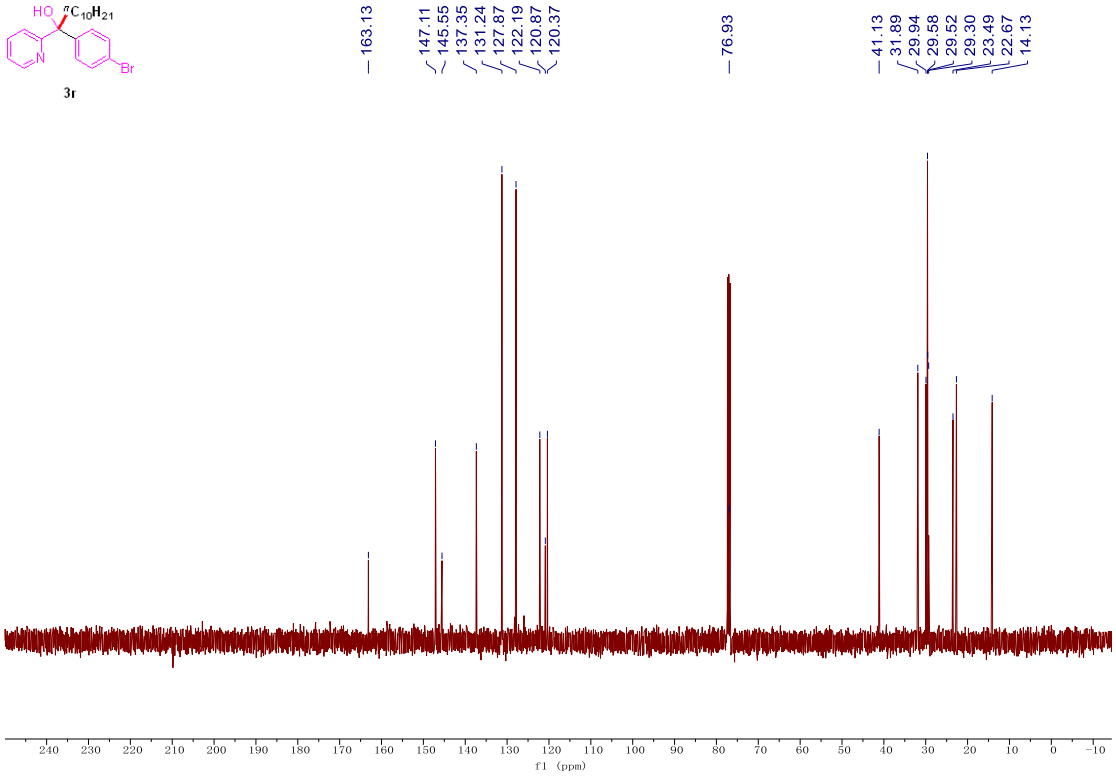
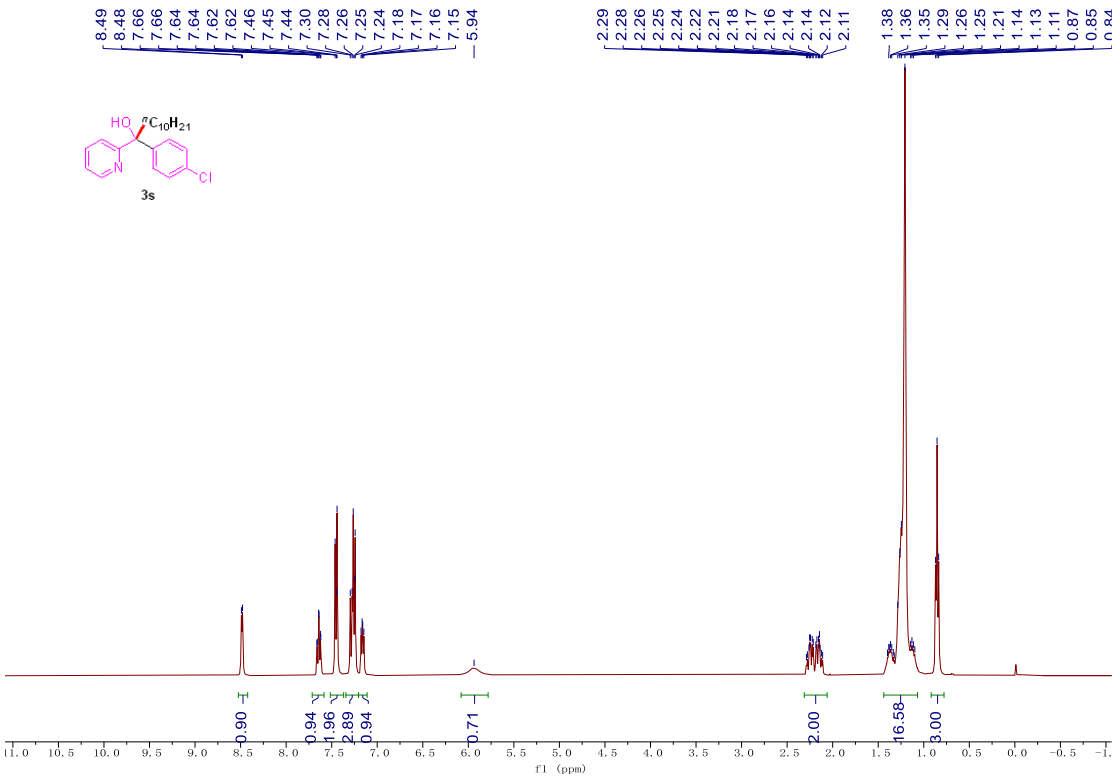
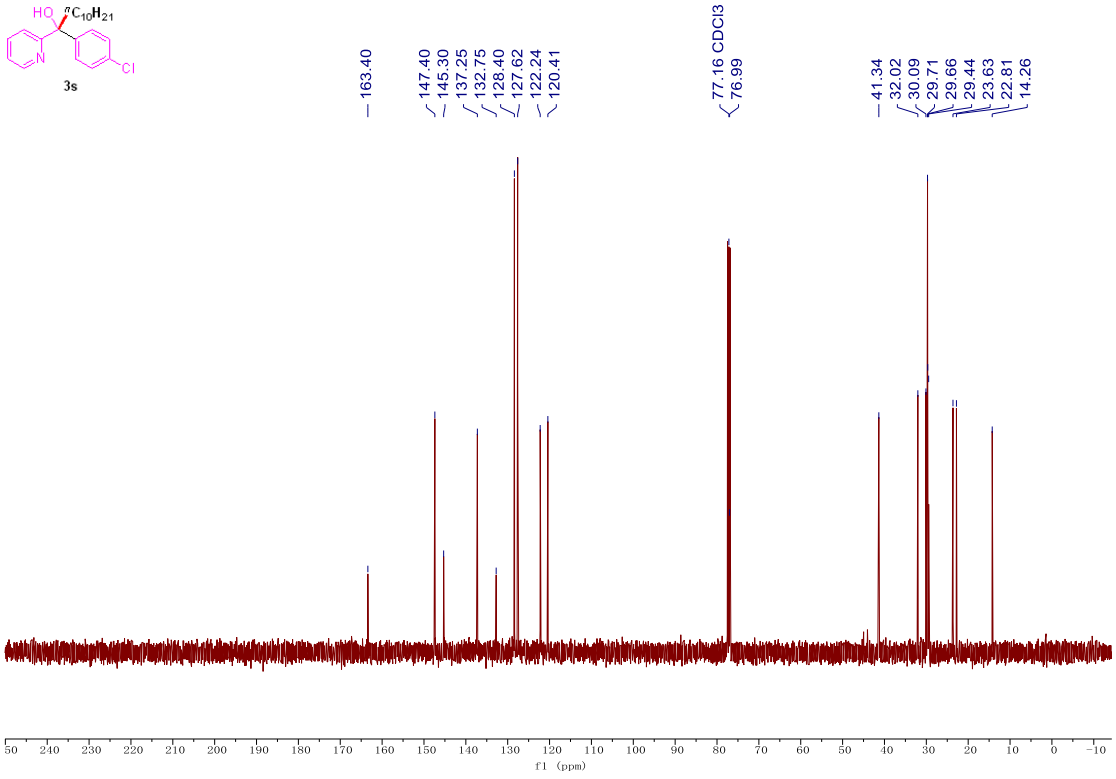
7.65
7.64
7.63
7.62
7.61
7.61
7.57
7.55
7.35
7.34
7.33
7.32
7.30
7.26 CDCl3
7.23
7.21
7.20
7.16
7.15
7.15
7.13
5.99
2.37
2.36
2.34
2.34
2.33
2.31
2.30
2.26
2.24
2.23
2.22
2.21
2.19
2.18
1.46
1.45
1.44
1.43
1.32
1.30
1.28
1.25
1.20
1.18
1.17
0.91
0.89
Ph
OH
$^{n}C_{10}H_{21}$
N
3q
0.96
0.97
1.97
2.87
0.95
0.96
0.91
2.00
1.12
14.70
1.06
3.09
f1 (ppm)

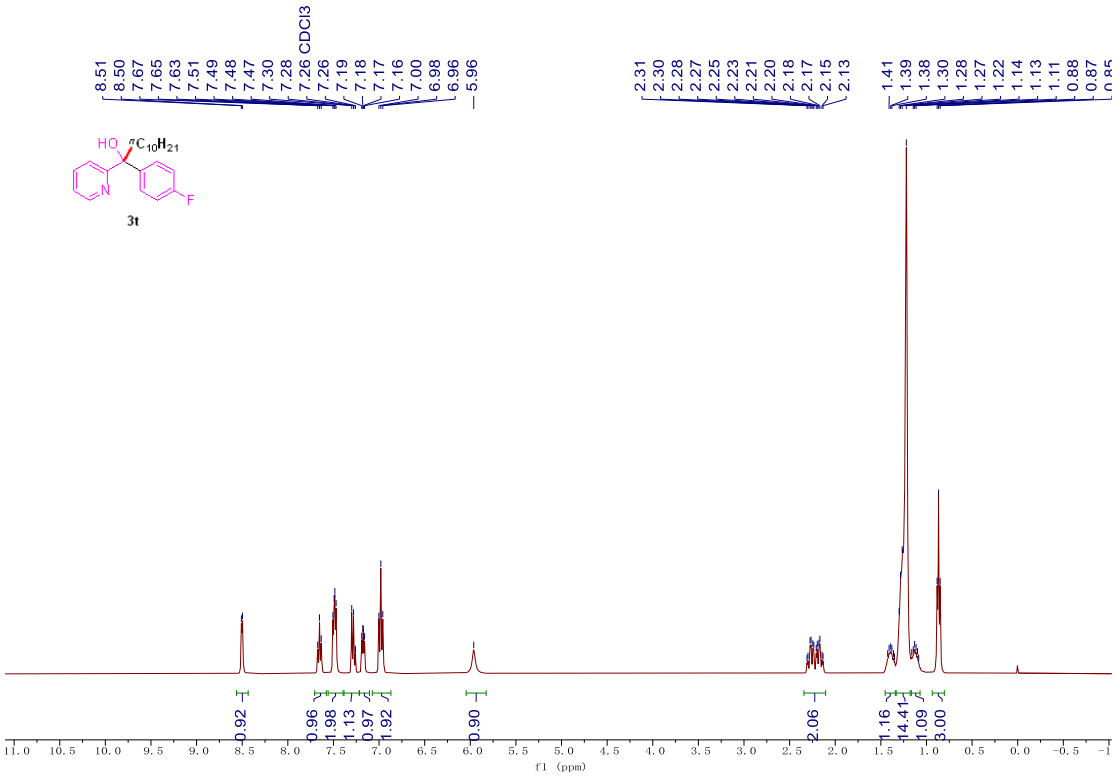

Ph
OH
$^{n}C_{10}H_{21}$
N
3q
163.90
147.26
146.70
137.02
128.26
126.84
126.07
121.98
120.54
77.27
77.16 CDCl3
41.40
32.00
30.13
29.71
29.69
29.65
29.42
23.67
22.77
14.22
f1 (ppm)

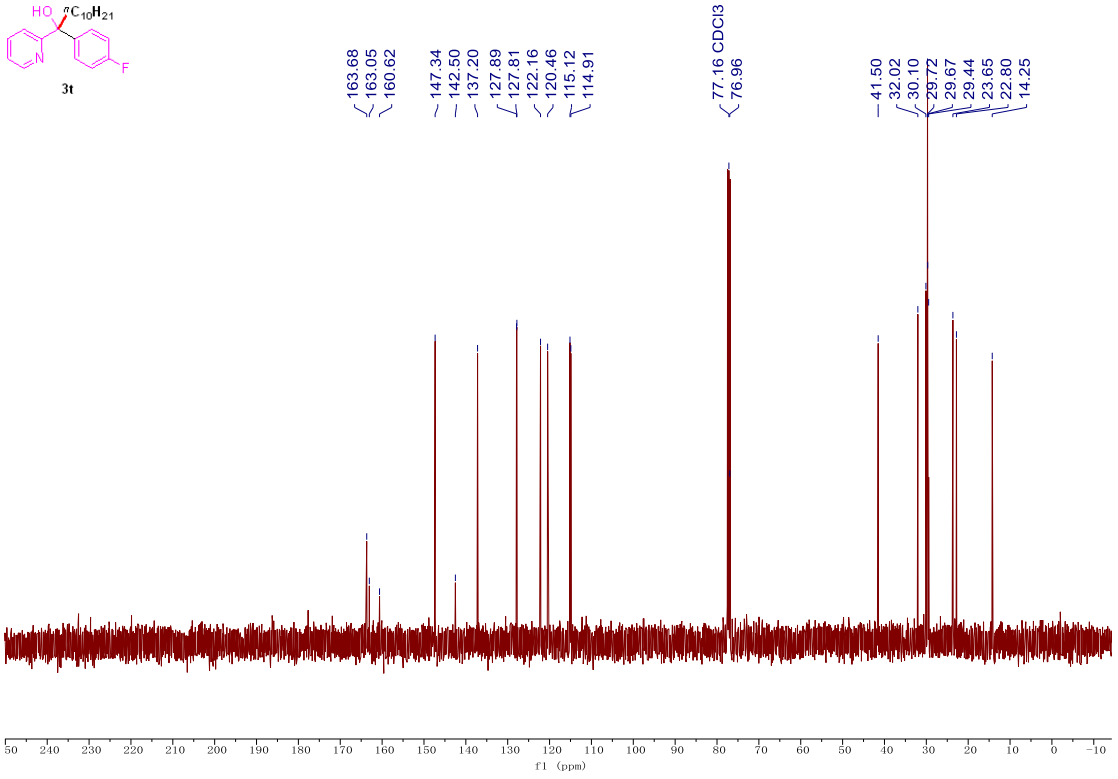

8.51
8.50
7.69
7.67
7.65
7.65
7.44
7.42
7.39
7.32
7.30
7.26 CDCl3
7.21
7.19
7.19
7.17
5.91
2.30
2.29
2.27
2.27
2.26
2.24
2.23
2.21
2.19
2.18
2.17
2.16
2.14
2.13
1.39
1.37
1.36
1.30
1.28
1.26
1.22
1.17
1.16
1.14
0.88
0.87
HO
$^{n}C_{10}H_{21}$
N
Br
3r
1.00
1.05
3.70
1.13
1.10
0.81
2.07
0.99
14.46
0.98
3.18
f1 (ppm)

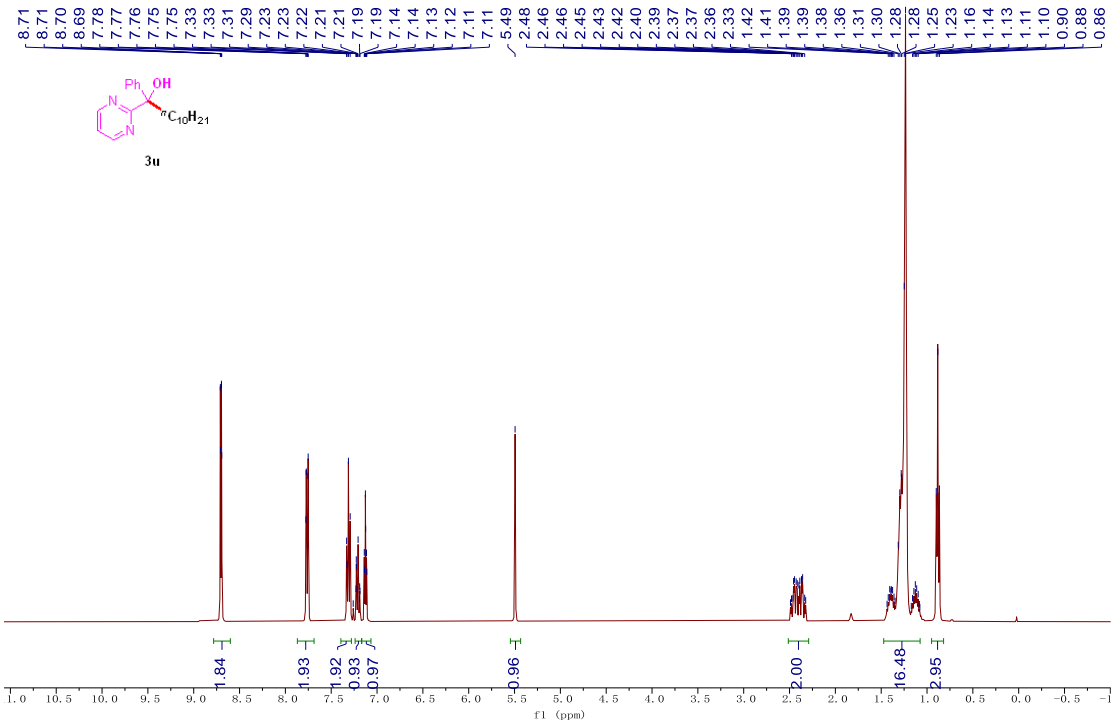

HO
$^{n}C_{10}H_{21}$
N
Br
3r
163.13
147.11
145.55
137.35
131.24
127.87
122.19
120.87
120.37
76.93
41.13
31.89
29.94
29.58
29.52
29.30
23.49
22.67
14.13
f1 (ppm)


8.49
8.48
7.66
7.66
7.64
7.64
7.62
7.62
7.46
7.45
7.44
7.30
7.28
7.26
7.25
7.24
7.18
7.17
7.16
7.15
5.94
2.29
2.28
2.26
2.25
2.24
2.22
2.21
2.18
2.17
2.16
2.14
2.14
2.12
2.11
1.38
1.36
1.35
1.29
1.26
1.25
1.21
1.14
1.13
1.11
0.87
0.85
HO
$^{n}C_{10}H_{21}$
N
Cl
3s
0.90
0.94
1.96
2.89
0.94
0.71
2.00
16.58
3.00
f1 (ppm)


HO
$^{n}C_{10}H_{21}$
N
Cl
3s
163.40
147.40
145.30
137.25
132.75
128.40
127.62
122.24
120.41
77.16 CDCl3
76.99
41.34
32.02
30.09
29.71
29.66
29.44
23.63
22.81
14.26
f1 (ppm)


8.51
8.50
7.67
7.65
7.63
7.51
7.49
7.48
7.47
7.30
7.28
7.26 CDCl3
7.26
7.19
7.18
7.17
7.16
7.00
6.98
6.96
5.96
2.31
2.30
2.28
2.27
2.25
2.23
2.21
2.20
2.18
2.17
2.15
2.13
1.41
1.39
1.38
1.30
1.28
1.27
1.22
1.14
1.13
1.11
0.88
0.87
HO
nC10H21
N
F
3t
0.92
0.96
1.98
1.13
0.97
1.92
0.90
2.06
1.16
14.41
1.09
3.00
f1 (ppm)


HO
nC10H21
N
F
3t
163.68
163.05
160.62
147.34
142.50
137.20
127.89
127.81
122.16
120.46
115.12
114.91
77.16 CDCl3
76.96
41.50
32.02
30.10
29.72
29.67
29.44
23.65
22.80
14.25
f1 (ppm)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8.71
8.70
8.69
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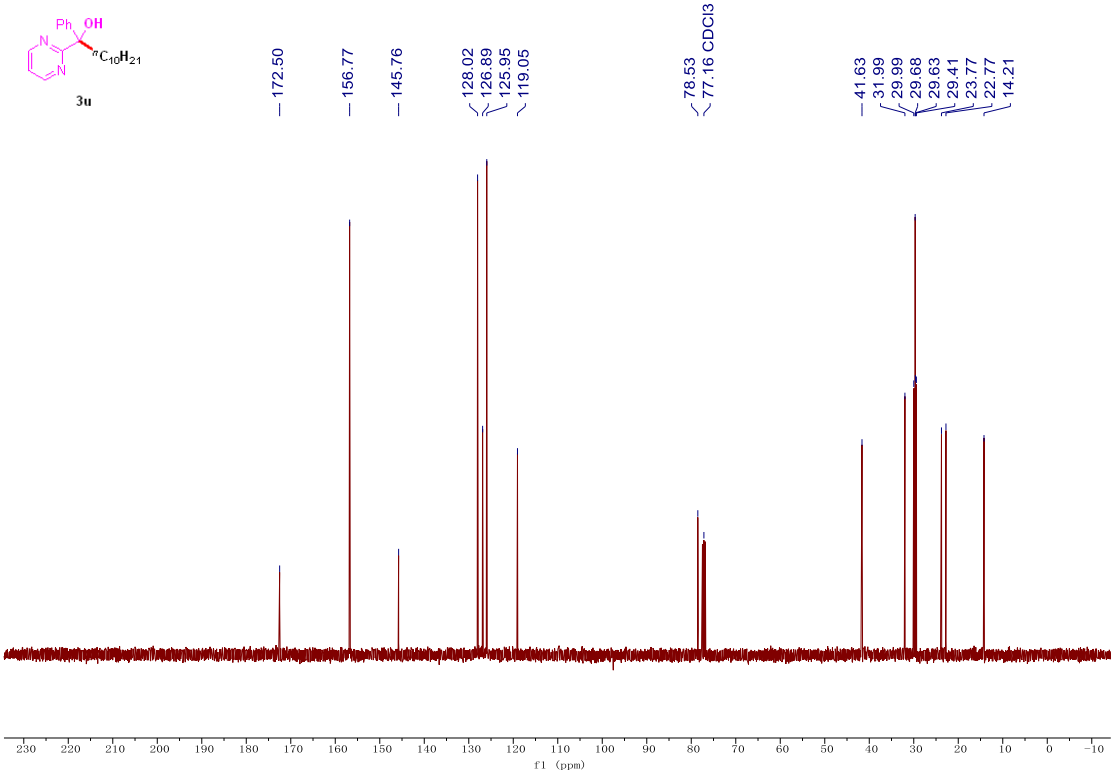
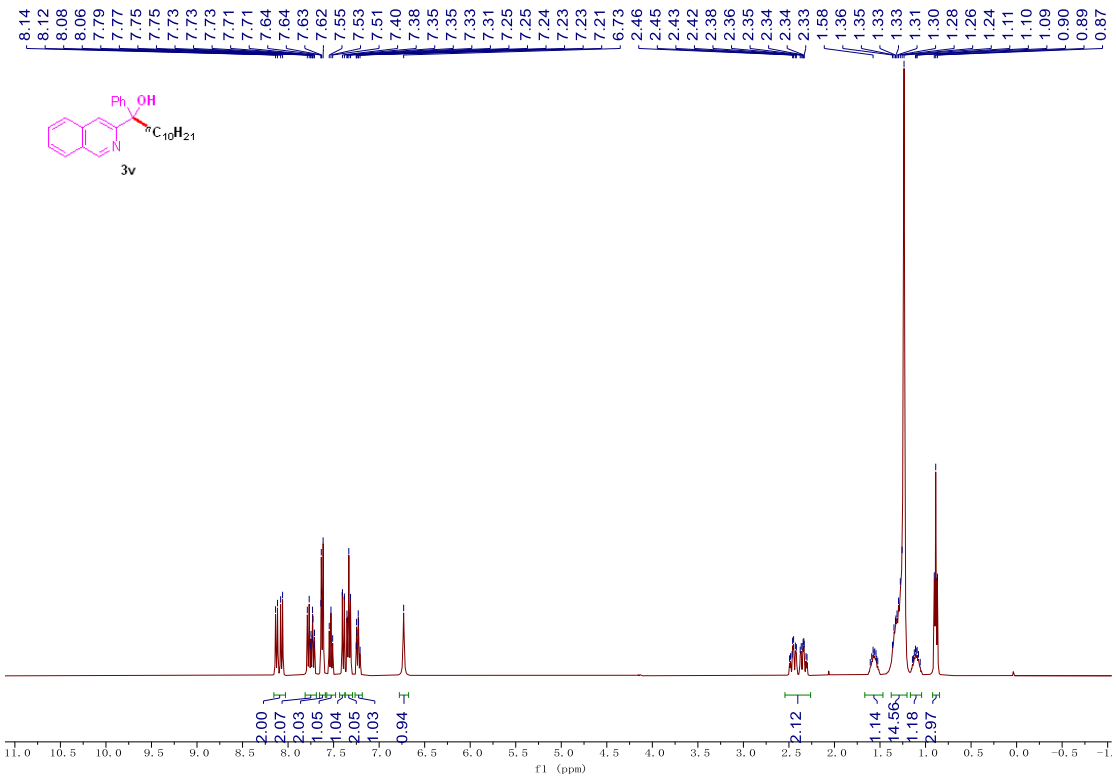
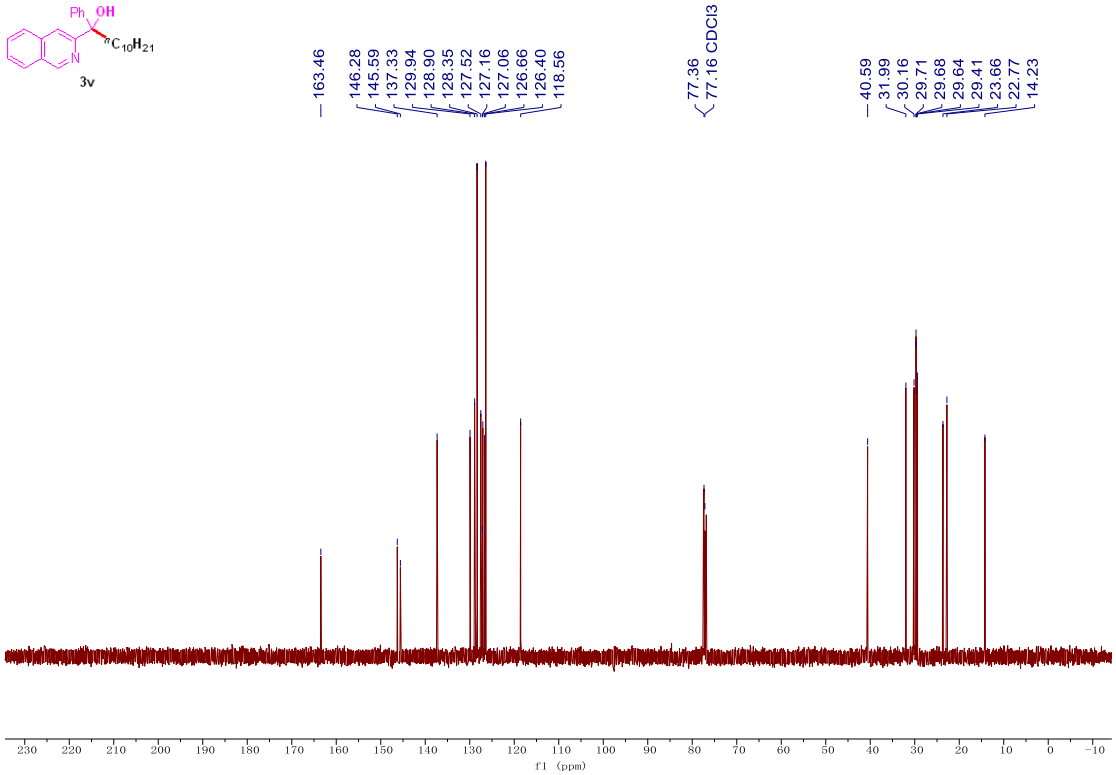
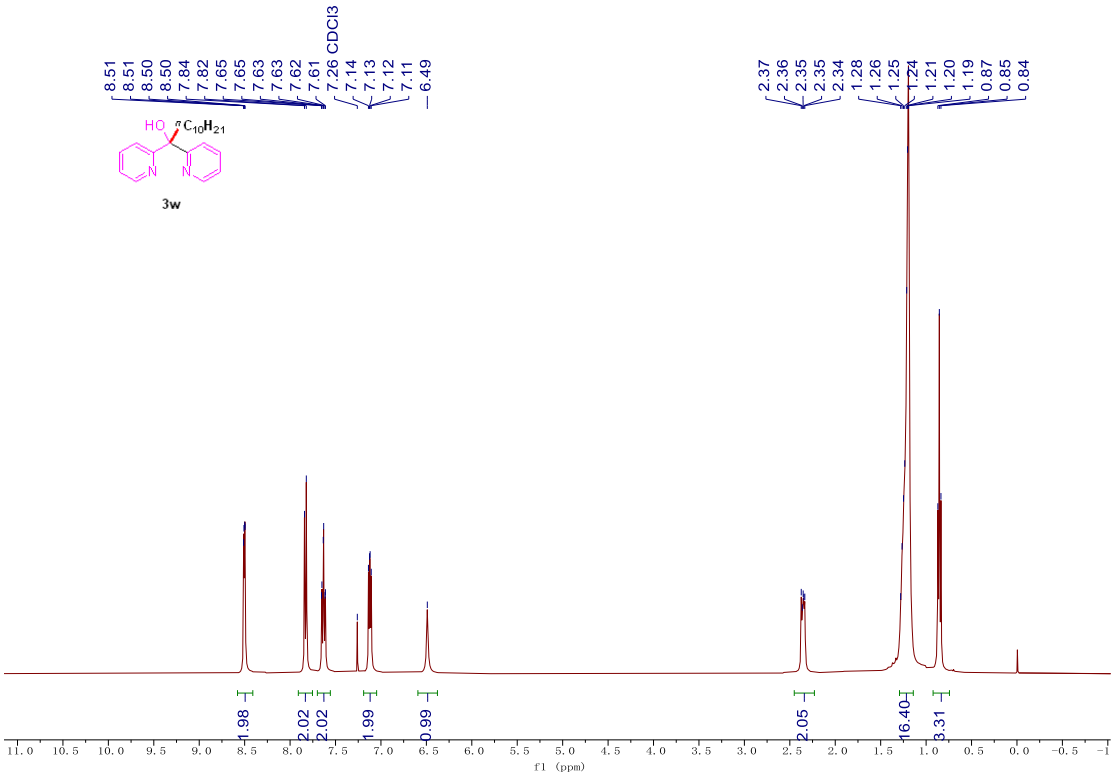
7.77
7.76
7.75
7.75
7.33
7.33
7.31
7.29
7.23
7.23
7.22
7.21
7.21
7.19
7.19
7.14
7.14
7.13
7.12
7.11
7.11
5.49
2.48
2.46
2.46
2.45
2.43
2.42
2.40
2.39
2.37
2.37
2.36
2.33
1.42
1.41
1.39
1.39
1.38
1.36
1.31
1.30
1.28
1.28
1.25
1.23
1.16
1.14
1.13
1.11
1.10
0.90
0.88
0.86
Ph
OH
$^{n}C_{10}H_{21}$
N
N
3u
1.84
1.93
1.92
0.93
0.97
0.96
2.00
16.48
2.95
f1 (ppm)


Ph
OH
$^{n}C_{10}H_{21}$
N
N
3u
172.50
156.77
145.76
128.02
126.89
125.95
119.05
78.53
77.16 CDCl3
41.63
31.99
29.99
29.68
29.63
29.41
23.77
22.77
14.21
f1 (ppm)


8.14 8.12 8.08 8.06 7.79 7.77 7.75 7.75 7.73 7.73 7.73 7.71 7.71 7.64 7.64 7.63 7.62 7.55 7.53 7.51 7.40 7.38 7.35 7.35 7.33 7.31 7.25 7.25 7.24 7.23 7.23 7.21 6.73 2.46 2.45 2.43 2.42 2.38 2.36 2.35 2.34 2.34 2.33 1.58 1.36 1.35 1.33 1.33 1.31 1.30 1.28 1.26 1.24 1.11 1.10 1.09 0.90 0.89 0.87
Ph OH
n C10H21
N
3v
2.00 2.07 2.03 1.05 1.04 2.05 1.03 0.94 2.12 1.14 14.56 1.18 2.97
f1 (ppm)


Ph OH
n C10H21
N
3v
163.46 146.28 145.59 137.33 129.94 128.90 128.35 127.52 127.16 127.06 126.66 126.40 118.56
77.36 77.16 CDCl3
40.59 31.99 30.16 29.71 29.68 29.64 29.41 23.66 22.77 14.23
f1 (ppm)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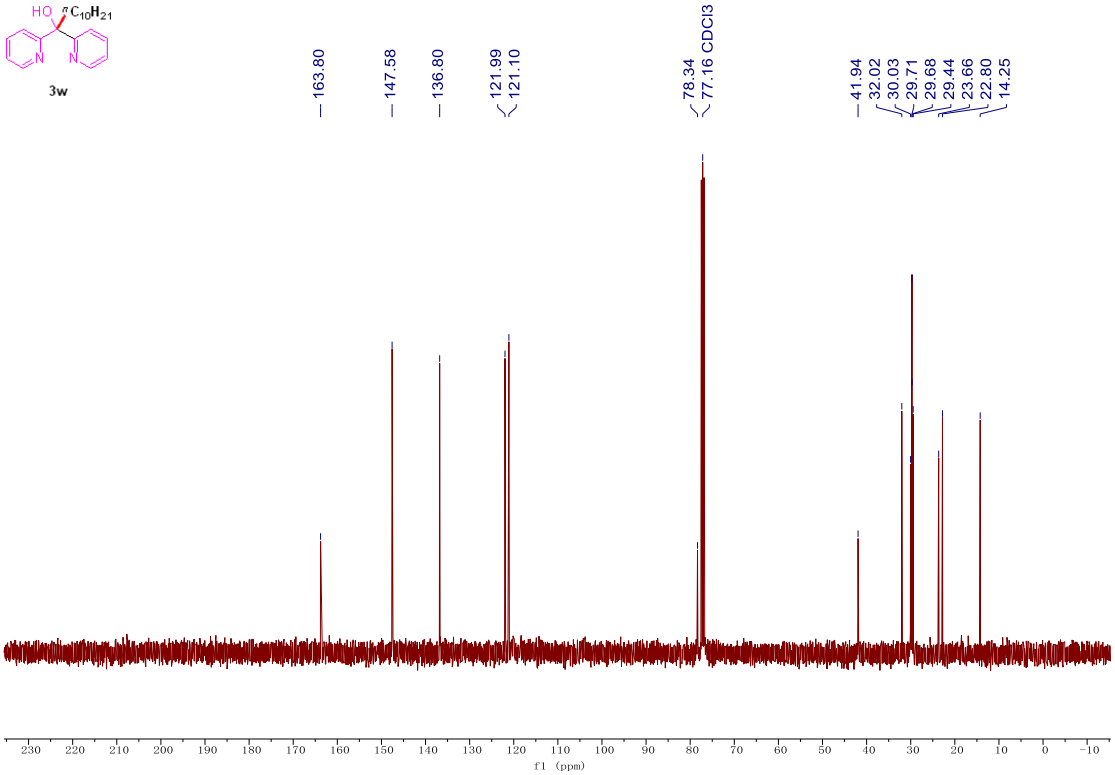
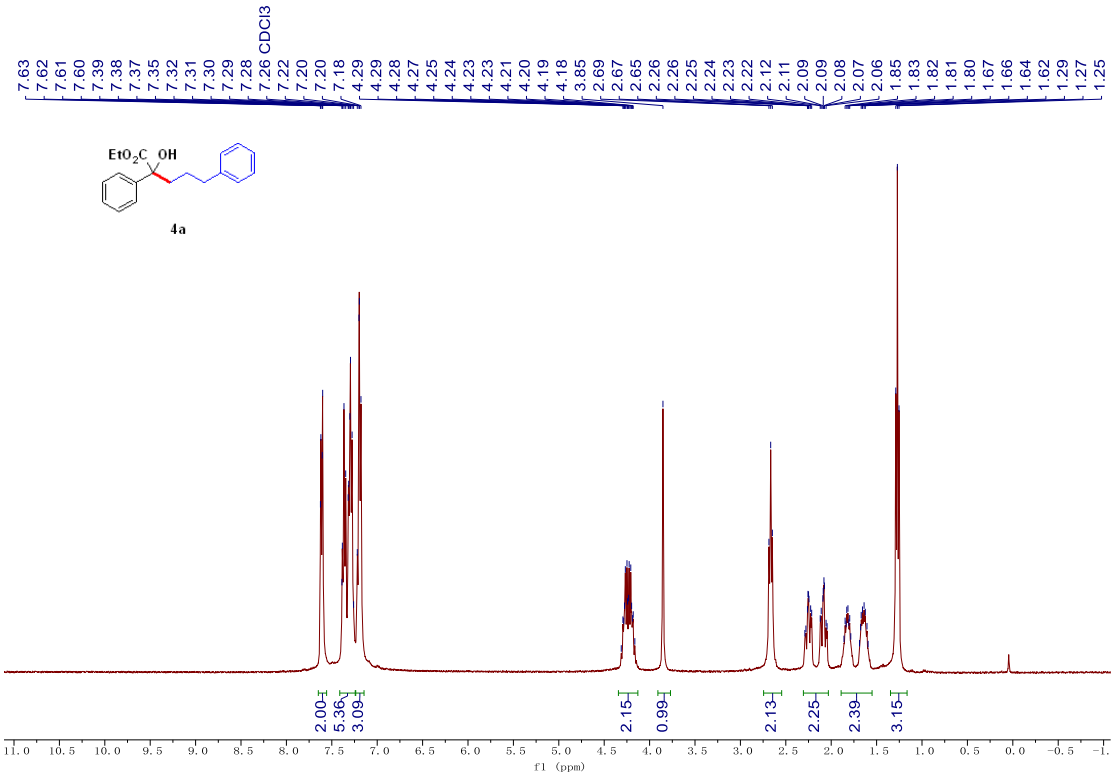
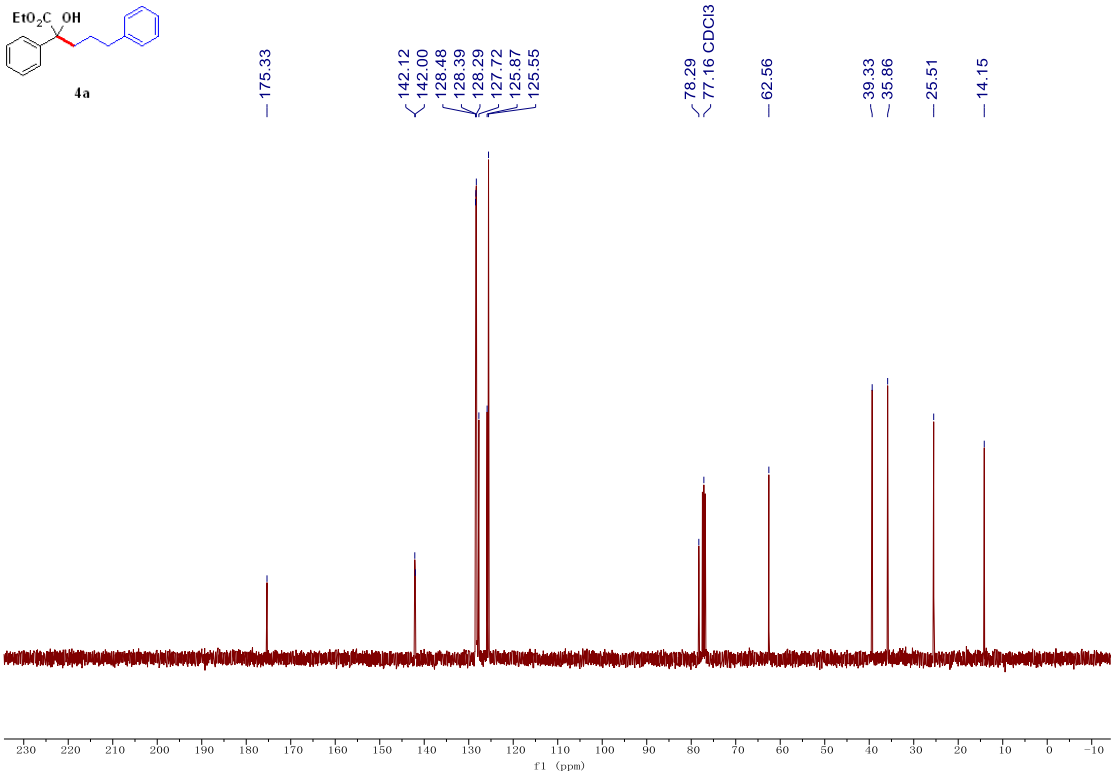
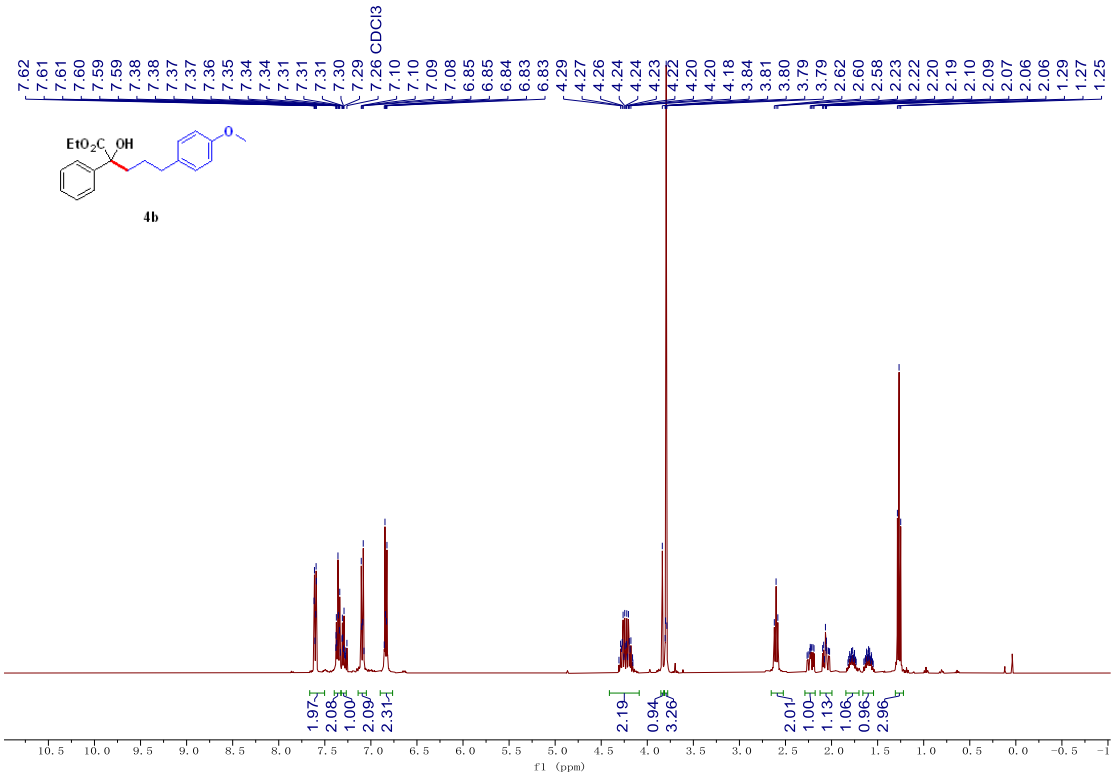
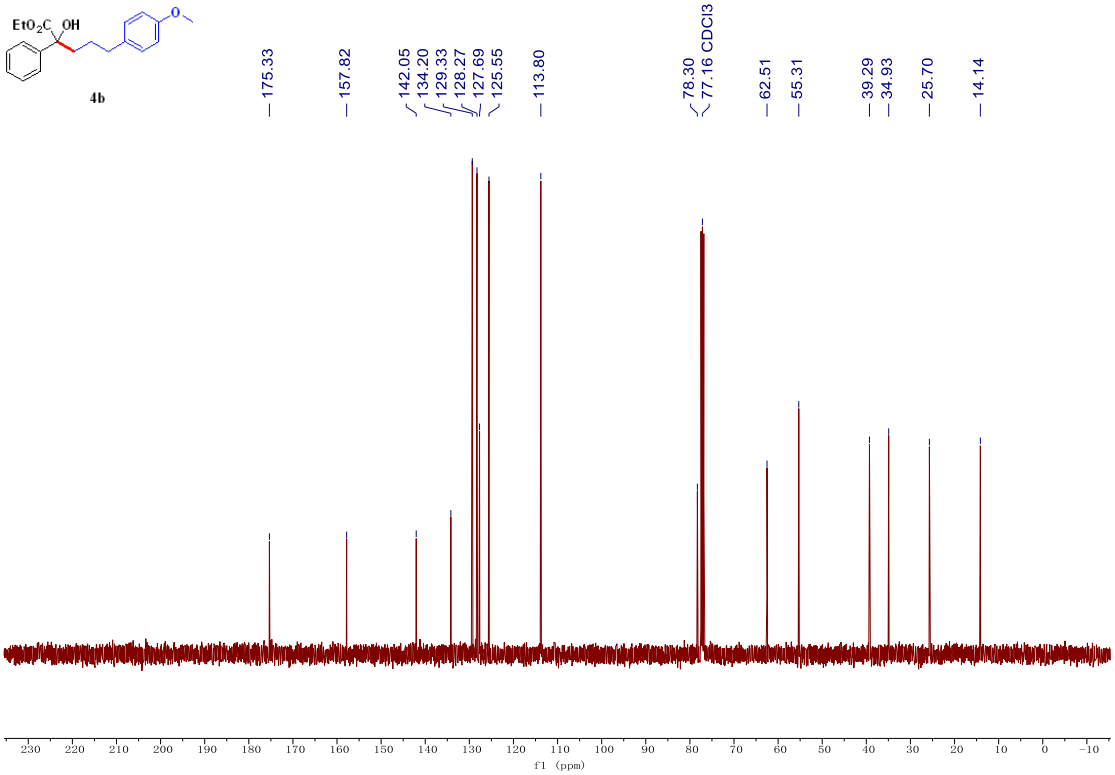
8.51
8.50
8.50
7.84
7.82
7.65
7.63
7.63
7.62
7.61
7.26 CDCl3
7.14
7.13
7.12
7.11
6.49
2.37
2.36
2.35
2.35
2.34
1.28
1.26
1.25
1.24
1.21
1.20
1.19
0.87
0.85
0.84
HO
nC10H21
3w
1.98
2.02
2.02
1.99
0.99
2.05
16.40
3.31
f1 (ppm)

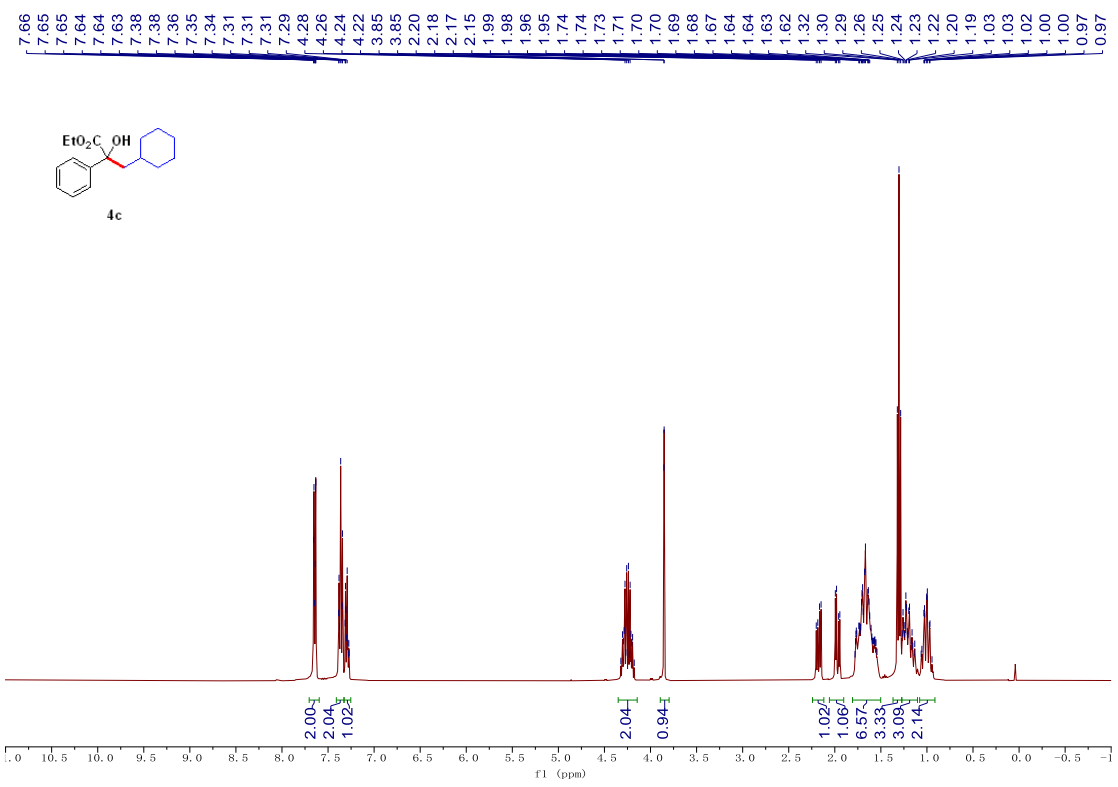

HO
nC10H21
3w
163.80
147.58
136.80
121.99
121.10
78.34
77.16 CDCl3
41.94
32.02
30.03
29.71
29.68
29.44
23.66
22.80
14.25
f1 (ppm)

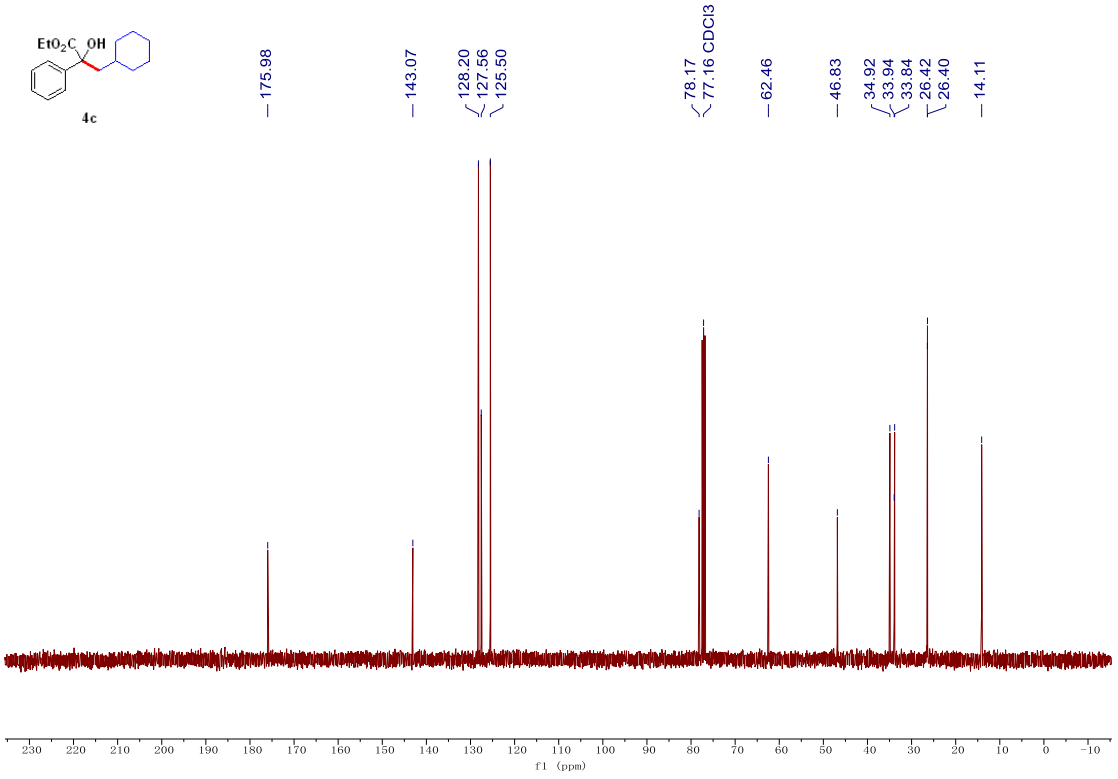

7.63 7.62 7.61 7.60 7.39 7.38 7.37 7.35 7.32 7.31 7.30 7.29 7.28 7.26 CDCl3 7.22 7.20 7.20 7.18 4.29 4.29 4.28 4.27 4.25 4.24 4.23 4.23 4.21 4.20 4.19 4.18 3.85 2.69 2.67 2.65 2.26 2.26 2.25 2.24 2.23 2.22 2.12 2.11 2.09 2.09 2.08 2.07 2.06 1.85 1.83 1.82 1.81 1.80 1.67 1.66 1.64 1.62 1.29 1.27 1.25
EtO2C OH
4a
2.00 5.36 3.09 2.15 0.99 2.13 2.25 2.39 3.15
f1 (ppm)

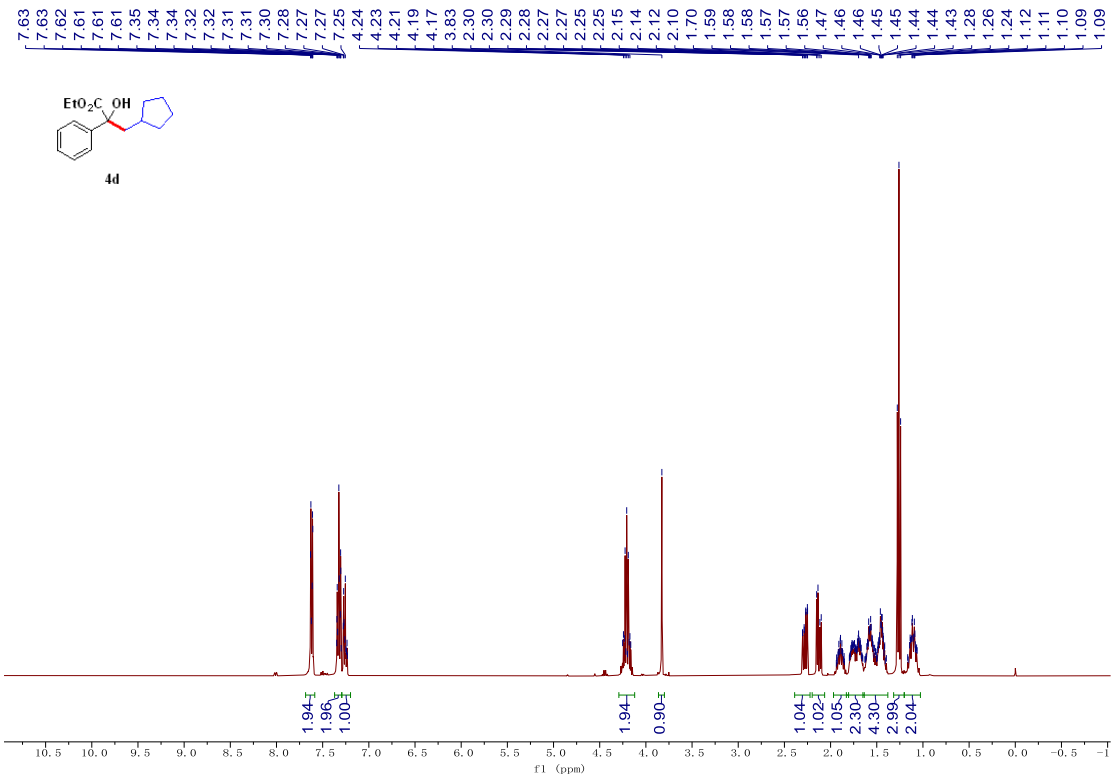

EtO2C OH
4a
175.33
142.12 142.00 128.48 128.39 128.29 127.72 125.87 125.55
78.29 77.16 CDCl3
62.56
39.33 35.86
25.51
14.15
f1 (ppm)

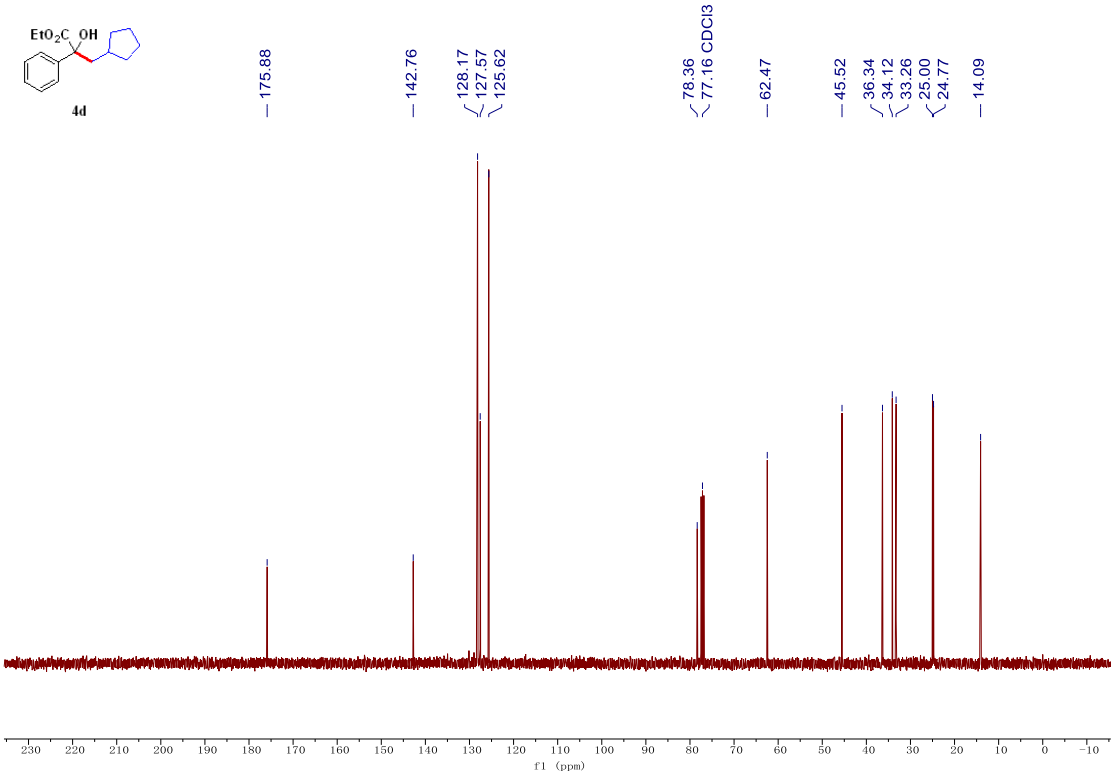

7.62 7.61 7.61 7.60 7.59 7.59 7.38 7.38 7.37 7.37 7.36 7.35 7.34 7.34 7.31 7.31 7.31 7.30 7.29 7.26 CDCl3 7.10 7.09 7.08 6.85 6.85 6.84 6.83 6.83
4.29 4.27 4.26 4.24 4.24 4.23 4.22 4.20 4.20 4.18 3.84 3.81 3.80 3.79 3.79 2.62 2.60 2.58 2.23 2.22 2.20 2.19 2.10 2.09 2.07 2.06 2.06 1.29 1.27 1.25
EtO2C OH
O
4b
1.97 2.08 1.00 2.09 2.31
2.19 0.94 3.26
2.01 1.00 1.13 1.06 0.96 2.96
f1 (ppm)


EtO2C OH
O
4b
175.33
157.82
142.05
134.20
129.33
128.27
127.69
125.55
113.80
78.30
77.16 CDCl3
62.51
55.31
39.29
34.93
25.70
14.14
f1 (ppm)


7.66 7.65 7.65 7.64 7.64 7.63 7.38 7.38 7.36 7.35 7.34 7.31 7.31 7.31 7.29 4.28 4.26 4.24 4.22 3.85 3.85 2.20 2.18 2.17 2.15 1.99 1.98 1.96 1.95 1.74 1.74 1.73 1.71 1.70 1.70 1.69 1.68 1.67 1.64 1.64 1.63 1.62 1.32 1.30 1.29 1.26 1.25 1.24 1.23 1.22 1.20 1.19 1.03 1.03 1.02 1.00 1.00 0.97 0.97
EtO2C OH
4c
2.00 2.04 1.02 2.04 0.94 1.02 1.06 6.57 3.33 3.09 2.14
f1 (ppm)


EtO2C OH
4c
175.98
143.07
128.20
127.56
125.50
78.17
77.16 CDCl3
62.46
46.83
34.92
33.94
33.84
26.42
26.40
14.11
f1 (ppm)


7.63 7.63 7.62 7.61 7.61 7.61 7.35 7.34 7.34 7.32 7.32 7.31 7.31 7.30 7.28 7.27 7.27 7.25 4.24 4.23 4.21 4.19 4.17 3.83 2.30 2.30 2.29 2.28 2.27 2.27 2.25 2.25 2.15 2.14 2.12 2.10 1.70 1.59 1.58 1.58 1.57 1.57 1.56 1.47 1.46 1.46 1.45 1.45 1.44 1.44 1.43 1.28 1.26 1.24 1.12 1.11 1.10 1.09 1.09
EtO2C OH
4d
1.94 1.96 1.00 1.94 0.90 1.04 1.02 1.05 2.30 4.30 2.99 2.04
f1 (ppm)


EtO2C OH
4d
175.88
142.76
128.17
127.57
125.62
78.36
77.16 CDCl3
62.47
45.52
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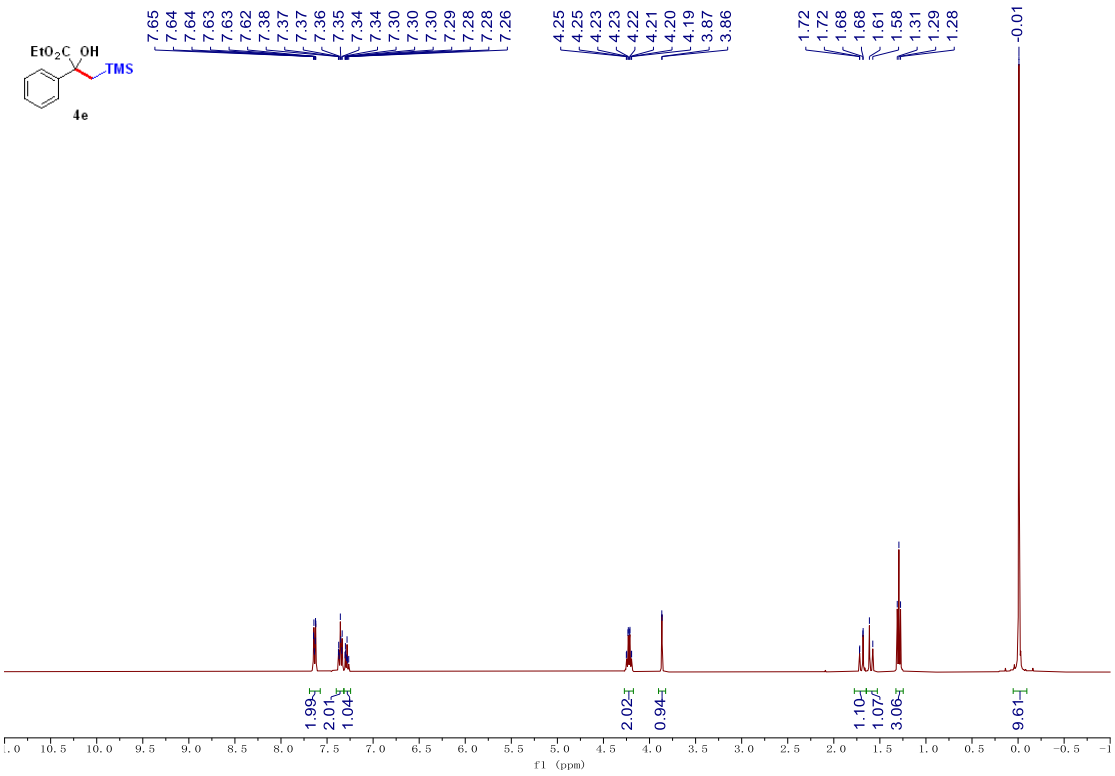
33.26
25.00
24.77
14.09
f1 (ppm)


EtO2C OH
TMS
4e
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7.64
7.63
7.63
7.62
7.38
7.37
7.37
7.36
7.35
7.34
7.34
7.30
7.30
7.30
7.29
7.28
7.28
7.26
4.25
4.25
4.23
4.23
4.22
4.21
4.20
4.19
3.87
3.86
1.72
1.72
1.68
1.68
1.61
1.58
1.31
1.29
1.28
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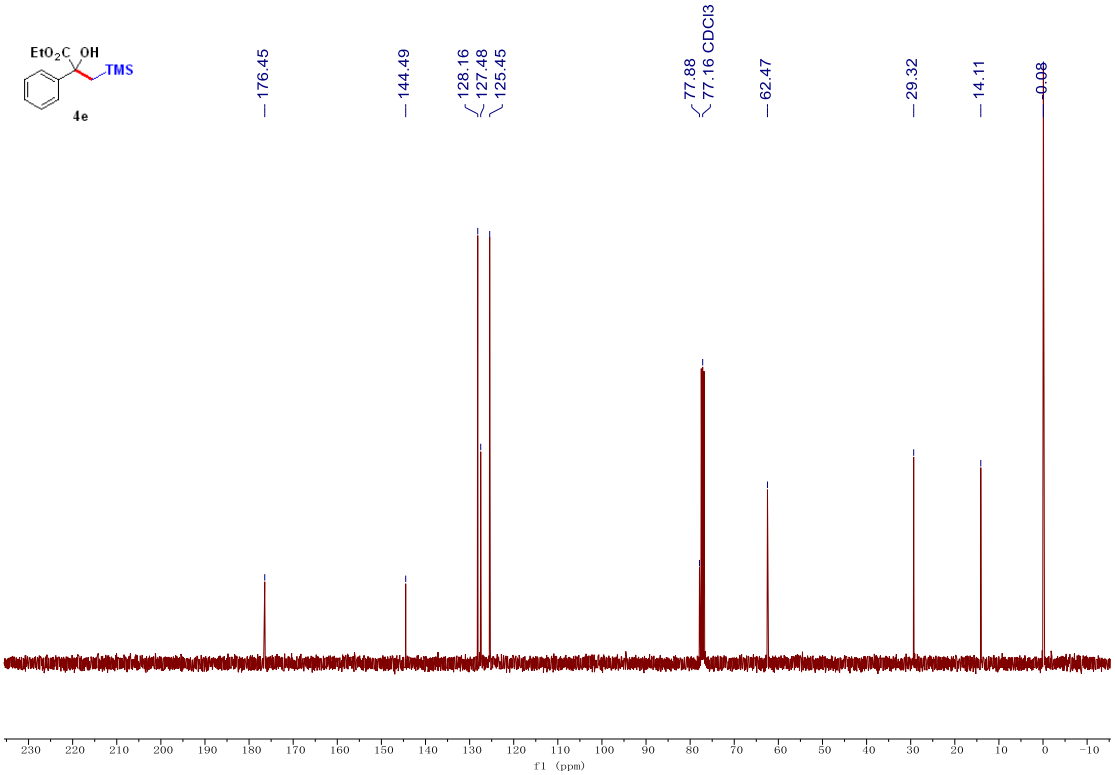
1.99
2.01
1.04
2.02
0.94
1.10
1.07
3.06
9.61
f1 (ppm)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TMS
4e
176.45
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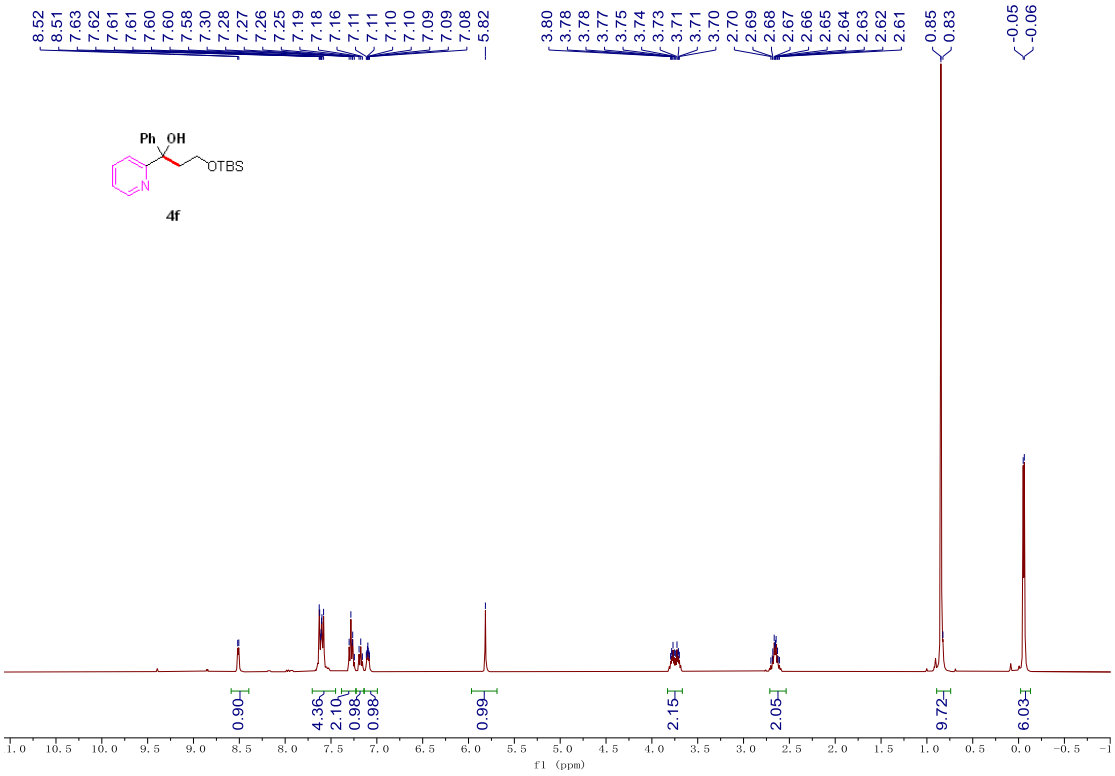
128.16
127.48
125.45
77.88
77.16 CDCl3
62.47
29.32
14.11
0.08
f1 (ppm)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7.60
7.58
7.30
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7.27
7.26
7.25
7.19
7.18
7.16
7.11
7.11
7.10
7.10
7.09
7.09
7.08
5.82
3.80
3.78
3.78
3.77
3.75
3.74
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3.71
3.71
3.70
2.70
2.69
2.68
2.67
2.66
2.65
2.64
2.63
2.62
2.61
0.85
0.83
-0.05
-0.06
Ph
OH
OTBS
N
4f
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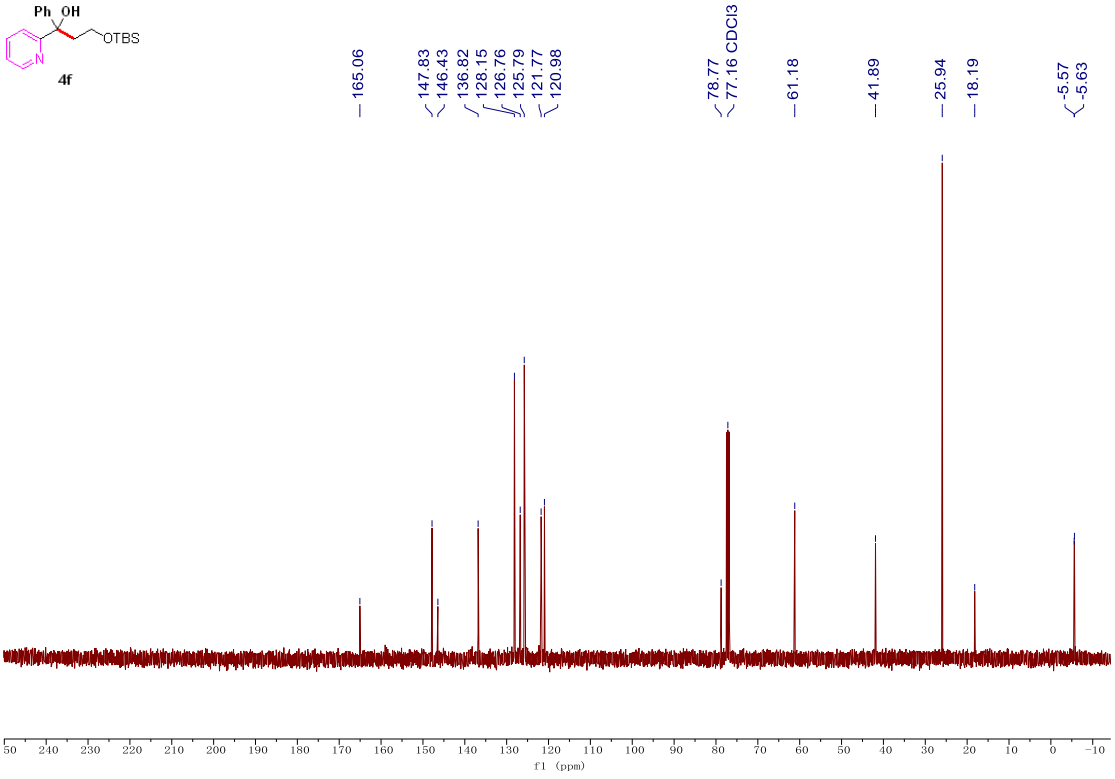
2.10
0.98
0.98
0.99
2.15
2.05
9.72
6.03
f1 (ppm)


Ph
OH
OTBS
N
4f
165.06
147.83
146.43
136.82
128.15
126.76
125.79
121.77
120.98
78.77
77.16 CDCl3
61.18
41.89
25.94
18.19
-5.57
-5.63
f1 (ppm)


8.48
8.47
7.64
7.63
7.62
7.61
7.60
7.59
7.52
7.50
7.32
7.31
7.30
7.29
7.27
7.20
7.19
7.17
7.15
7.14
7.13
7.12
5.97
4.09
4.07
4.05
4.03
2.36
2.35
2.33
2.33
2.32
2.30
2.28
2.26
2.24
2.22
2.21
2.20
2.20
2.19
2.17
2.16
1.67
1.65
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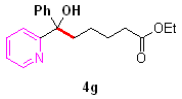
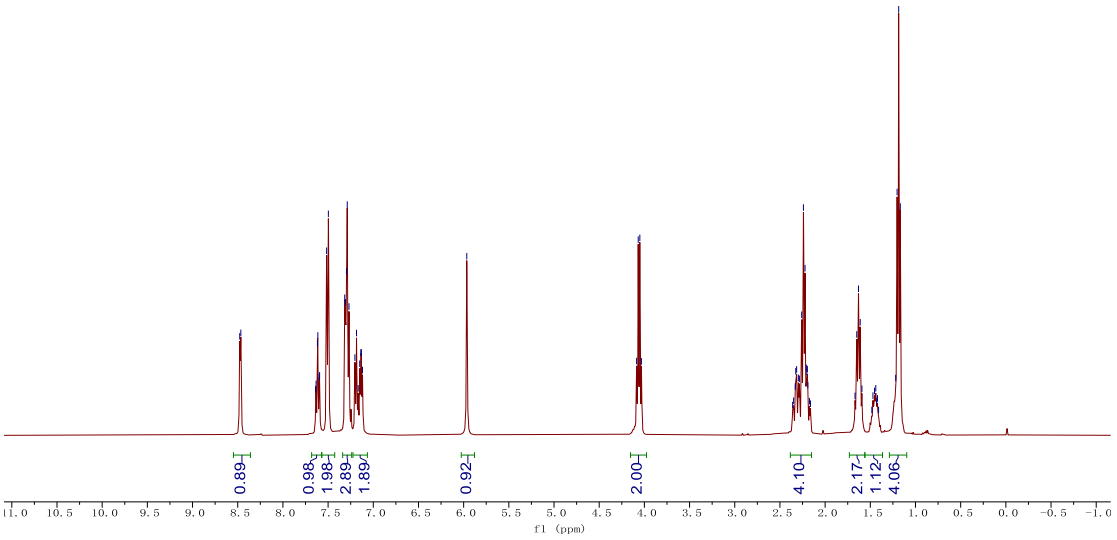
1.44
1.43
1.42
1.22
1.20
1.19
1.17


Ph
OH
OEt
N
O

4g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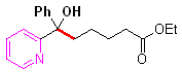
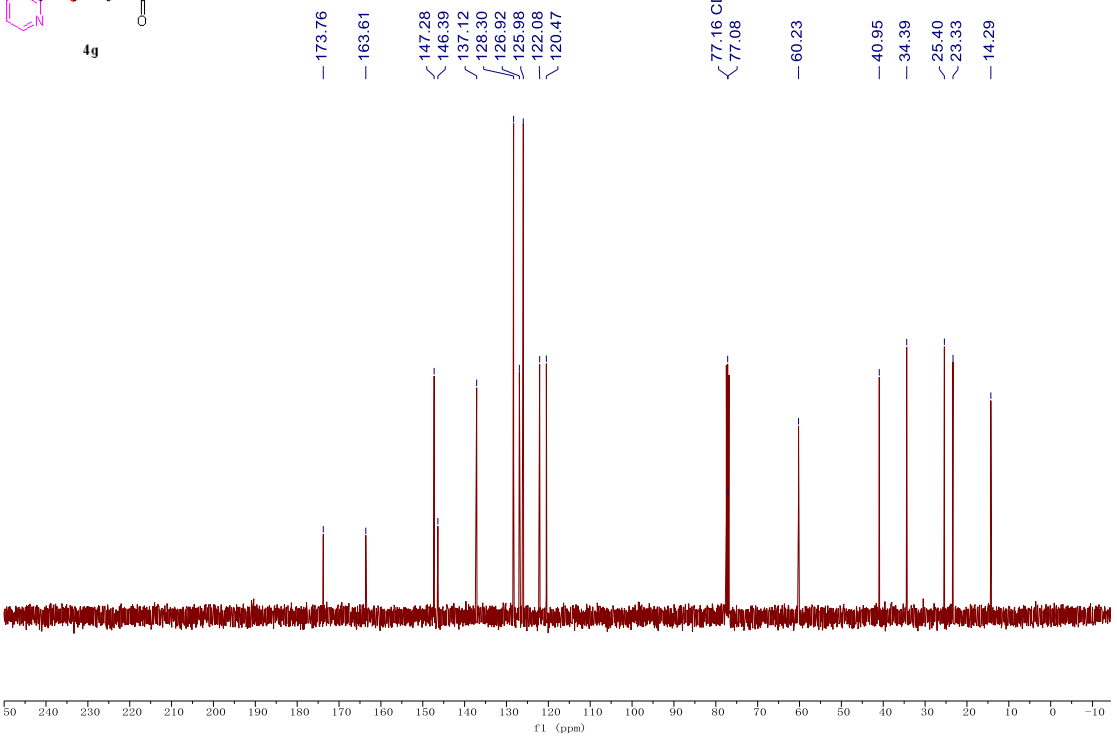
0.92
2.00
4.10
2.17
1.12
4.06
f1 (ppm)


Ph
OH
OEt
N
O

4g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163.61
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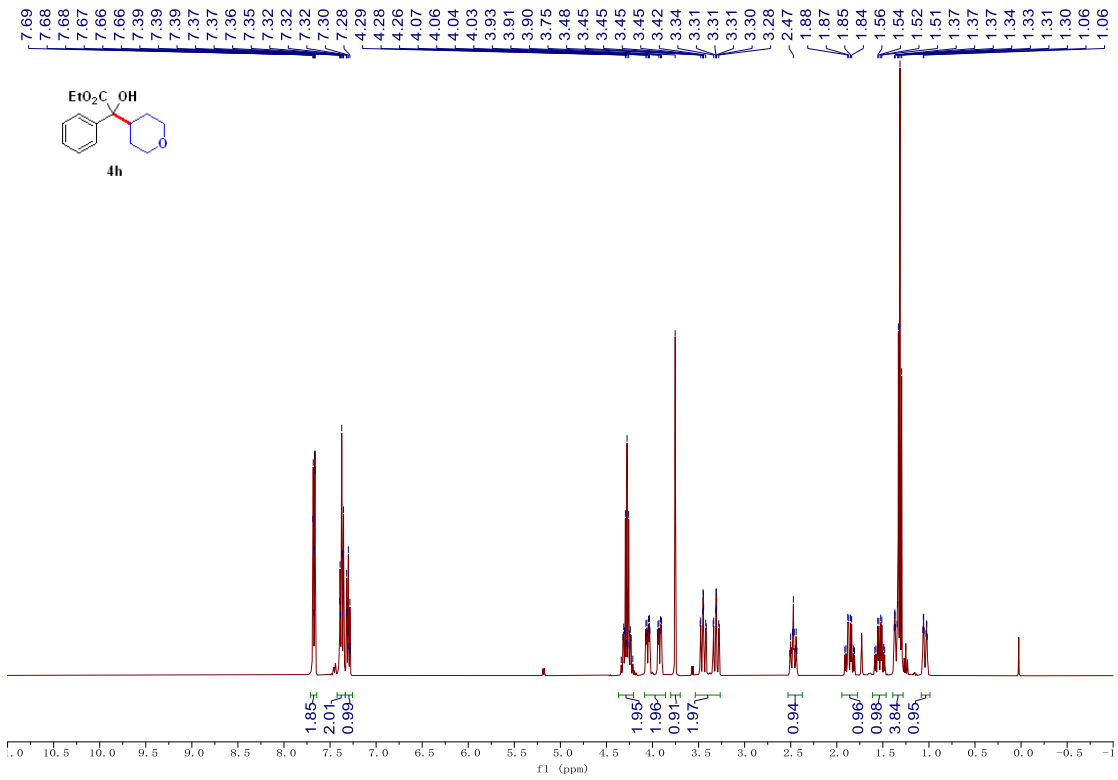
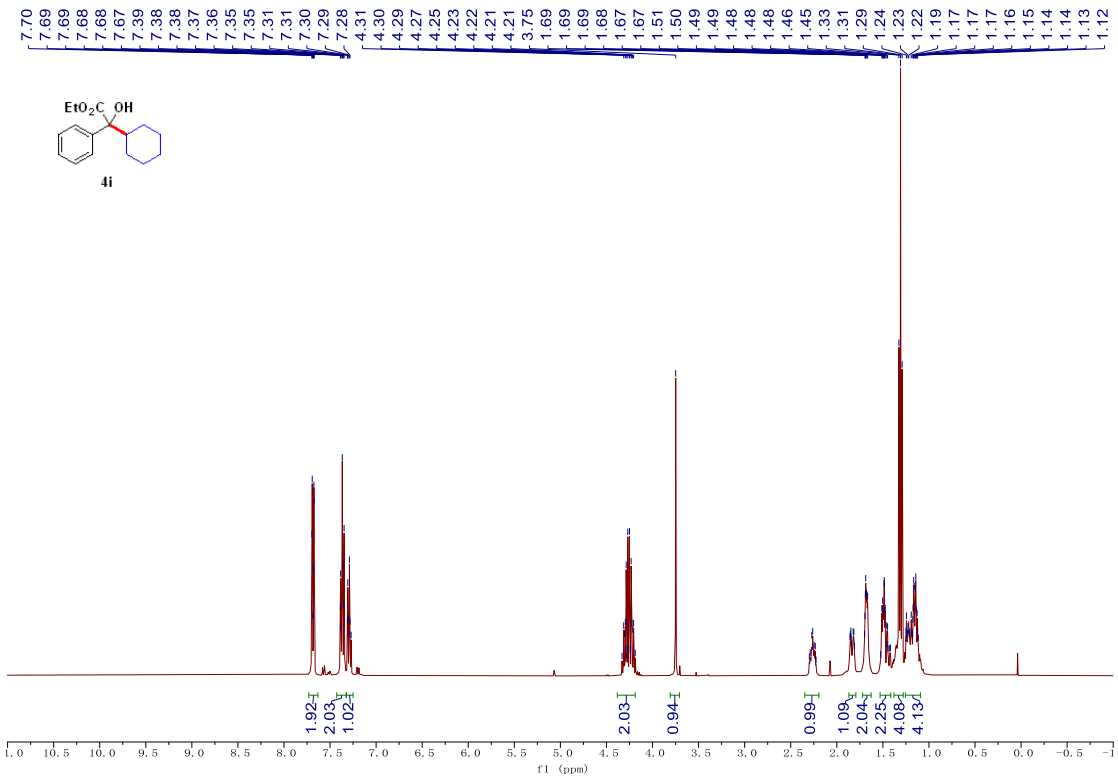
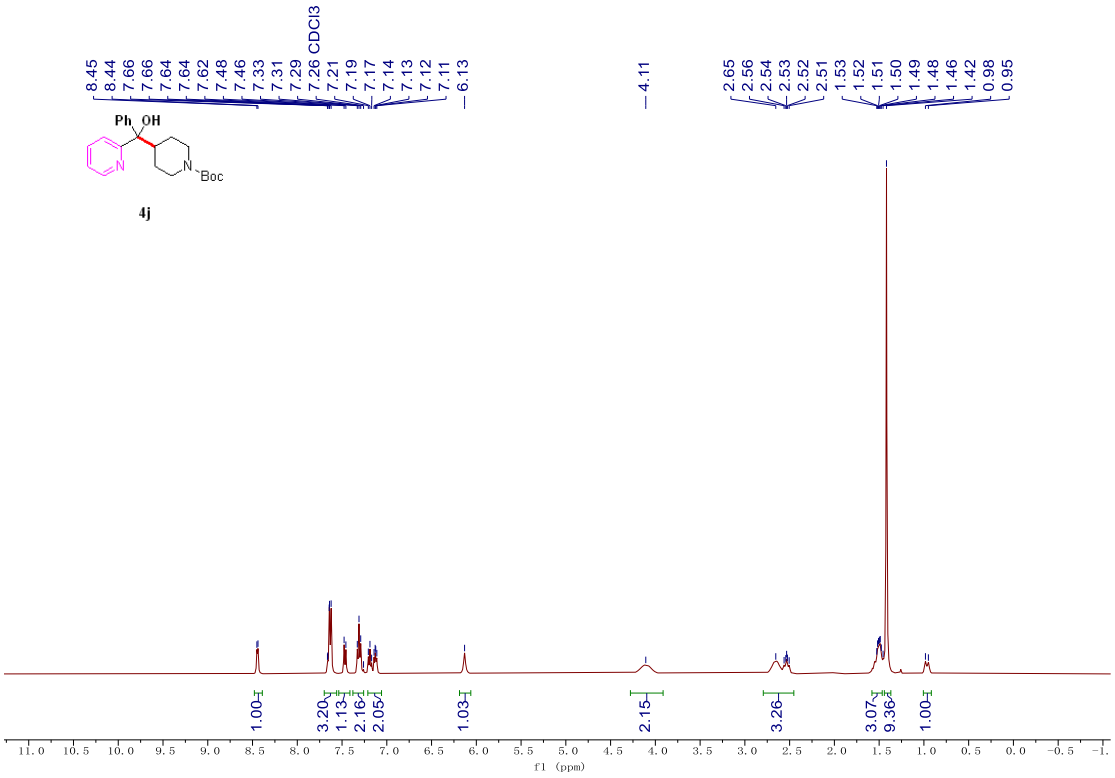
146.39
137.12
128.30
126.92
125.98
122.08
120.47
77.16 CDCl3
77.08
60.23
40.95
34.39
25.40
23.33
14.29
f1 (ppm)


EtO2C OH
O
4h
f1 (ppm)


EtO2C OH
4i
f1 (ppm)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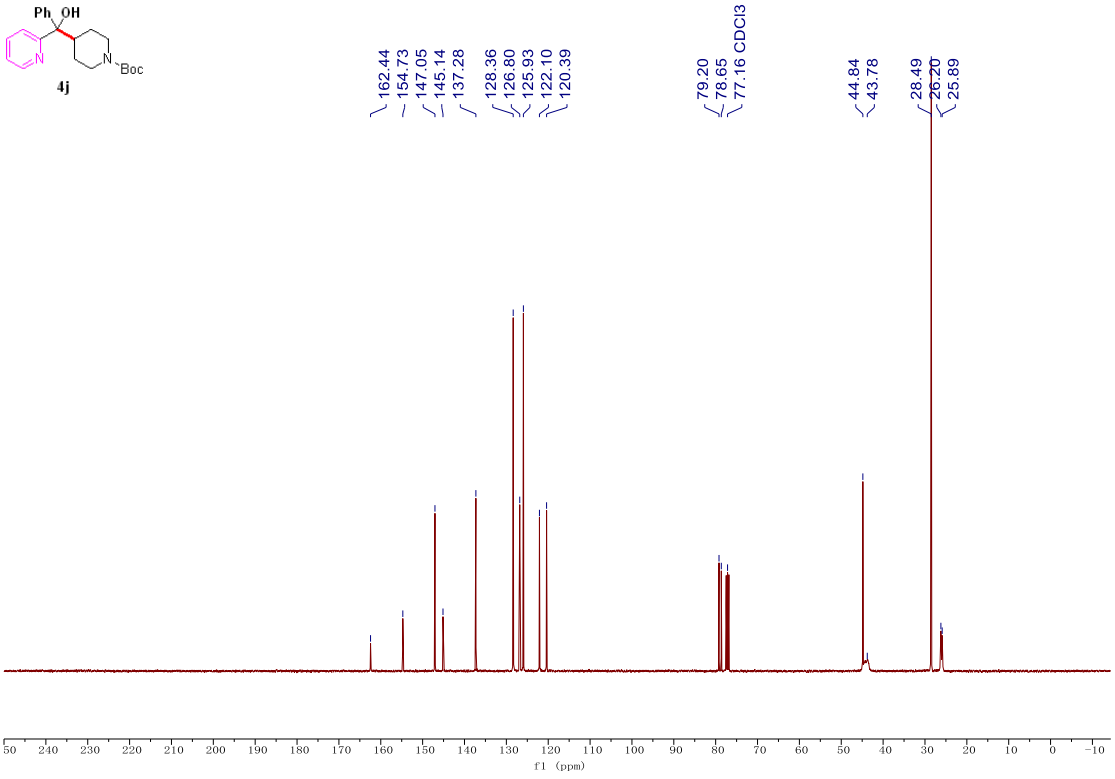
7.33
7.31
7.29
7.26 CDCl3
7.21
7.19
7.17
7.14
7.13
7.12
7.11
6.13
4.11
2.65
2.56
2.54
2.53
2.52
2.51
1.53
1.52
1.51
1.50
1.49
1.48
1.46
1.42
0.98
0.95
Ph
OH
N
Boc
4j
1.00
3.20
1.13
2.16
2.05
1.03
2.15
3.26
3.07
9.36
1.00
f1 (ppm)


Ph
OH
N
Boc
4j
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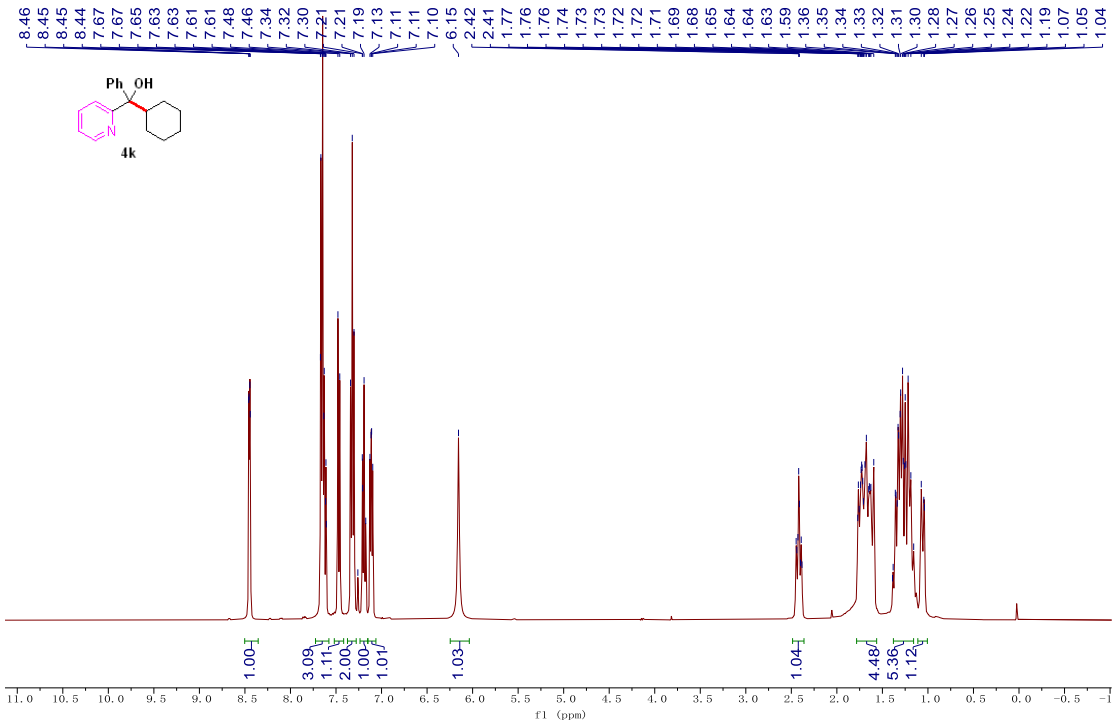
147.05
145.14
137.28
128.36
126.80
125.93
122.10
120.39
79.20
78.65
77.16 CDCl3
44.84
43.78
28.49
26.20
25.89
f1 (ppm)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7.63
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7.61
7.61
7.48
7.46
7.34
7.32
7.30
7.21
7.21
7.19
7.13
7.11
7.11
7.10
6.15
2.42
2.41
1.77
1.76
1.76
1.74
1.73
1.73
1.72
1.72
1.71
1.69
1.68
1.65
1.64
1.64
1.63
1.59
1.36
1.35
1.34
1.33
1.32
1.31
1.30
1.28
1.27
1.26
1.25
1.24
1.22
1.19
1.07
1.05
1.04
Ph OH
N
4k
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3.09
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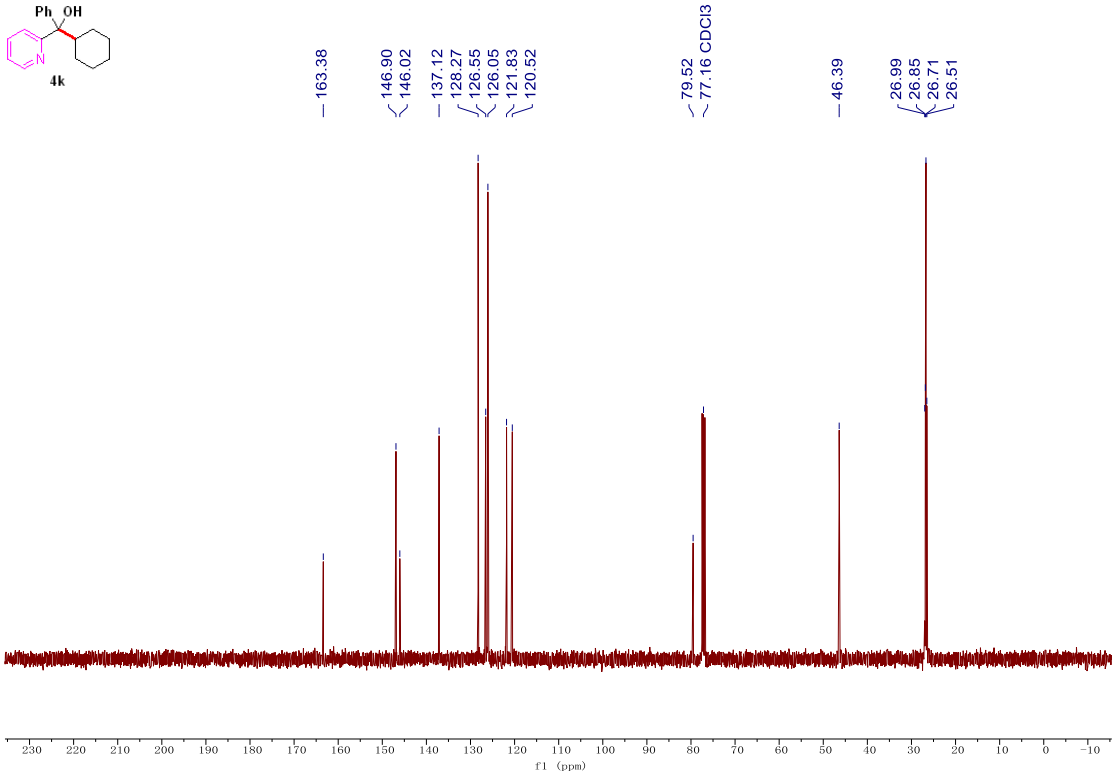
2.00
1.00
1.01
1.03
1.04
4.48
5.36
1.12
f1 (ppm)


Ph OH
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4k
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137.12
128.27
126.55
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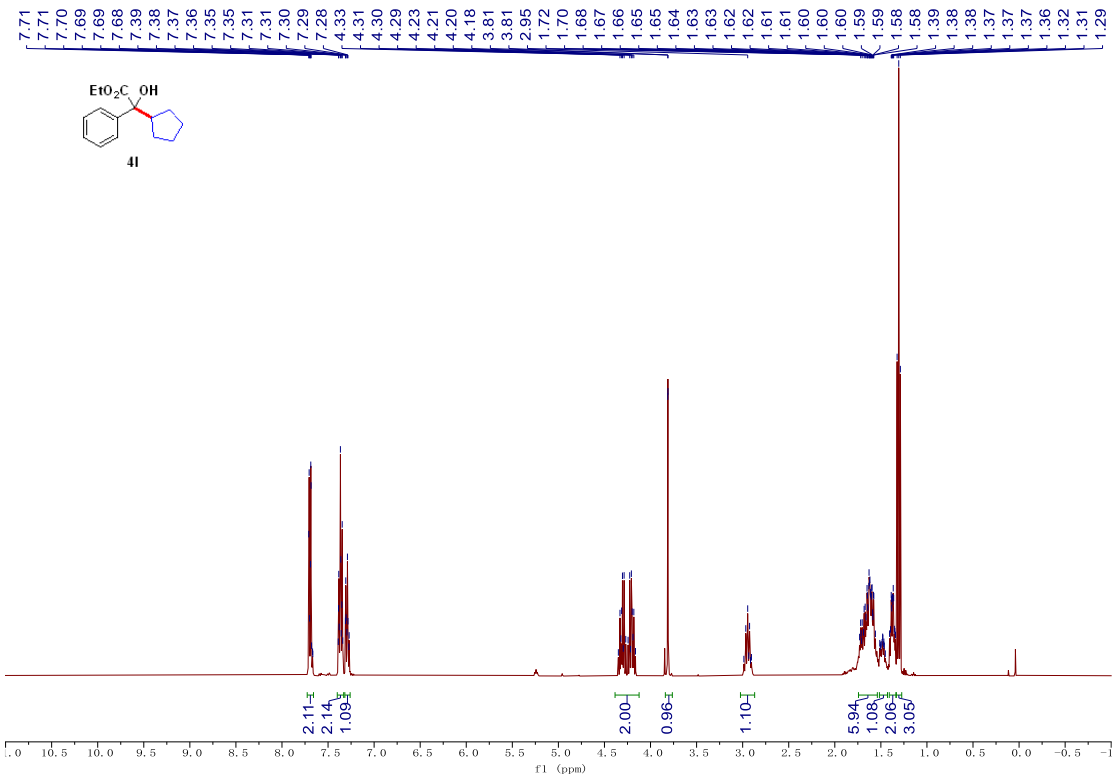
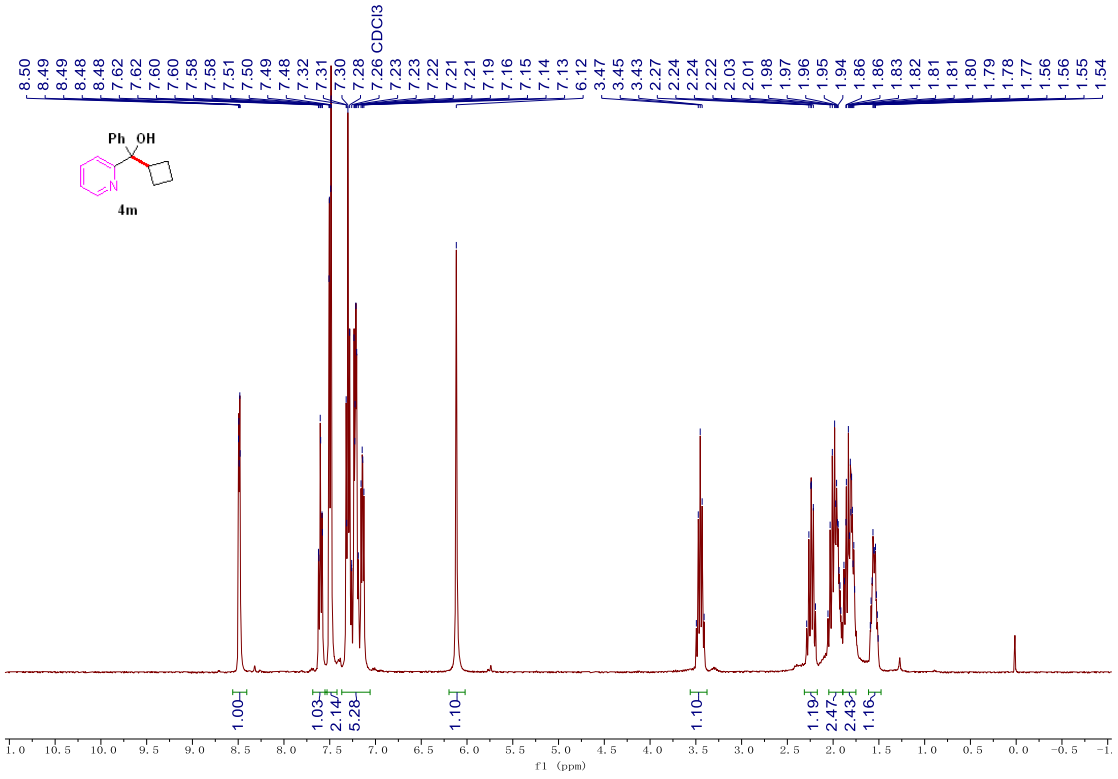
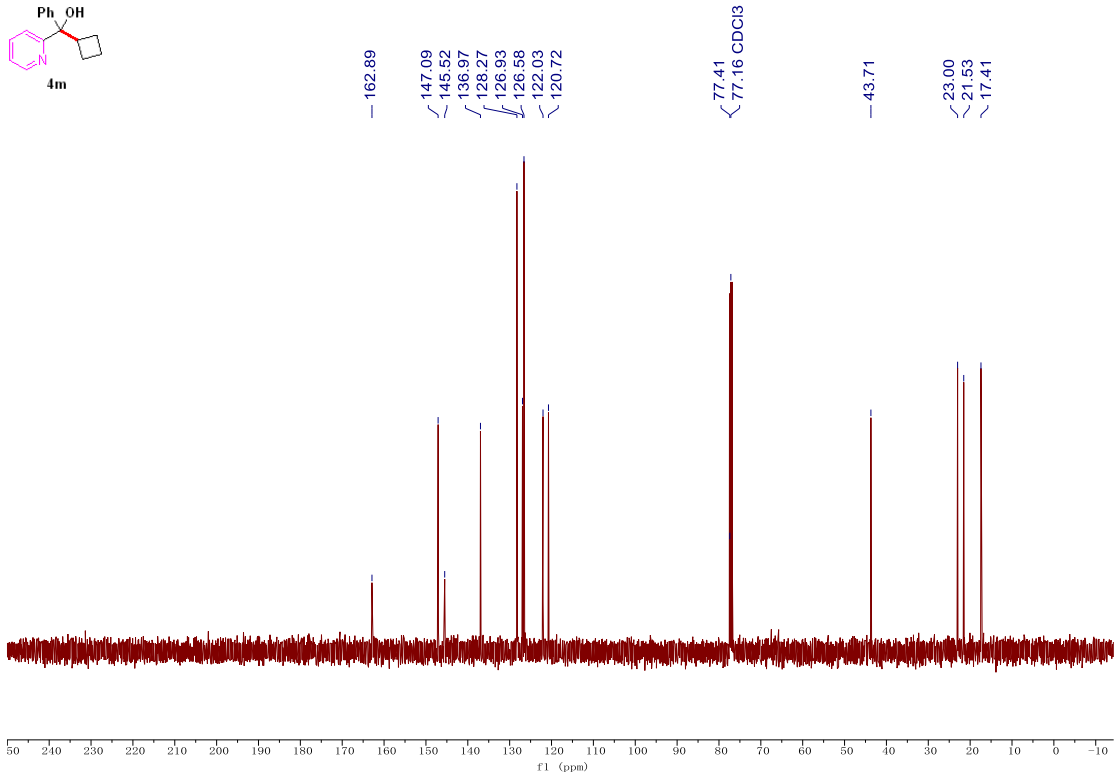
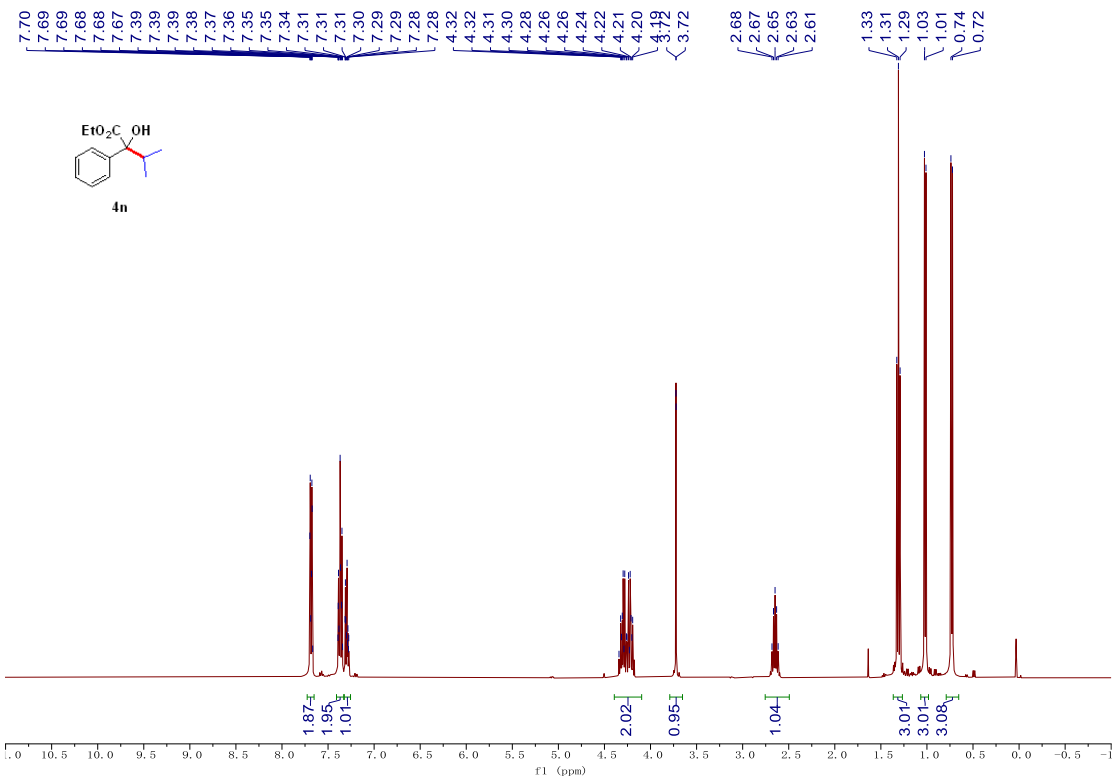
121.83
120.52
79.52
77.16 CDCl3
46.39
26.99
26.85
26.71
26.51
f1 (ppm)


EtO2C OH
4l
f1 (ppm)


Ph OH
N
4m
CDCl3
f1 (ppm)


Ph OH
N
4m
162.89
147.09
145.52
136.97
128.27
126.93
126.58
122.03
120.72
77.41
77.16 CDCl3
43.71
23.00
21.53
17.41
f1 (ppm)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7.69
7.69
7.68
7.68
7.67
7.39
7.39
7.39
7.38
7.37
7.36
7.35
7.35
7.34
7.31
7.31
7.31
7.30
7.29
7.29
7.28
7.28
4.32
4.32
4.31
4.30
4.28
4.26
4.26
4.24
4.22
4.21
4.20
4.19
3.72
2.68
2.67
2.65
2.63
2.61
1.33
1.31
1.29
1.03
1.01
0.74
0.72
EtO2C OH
4n
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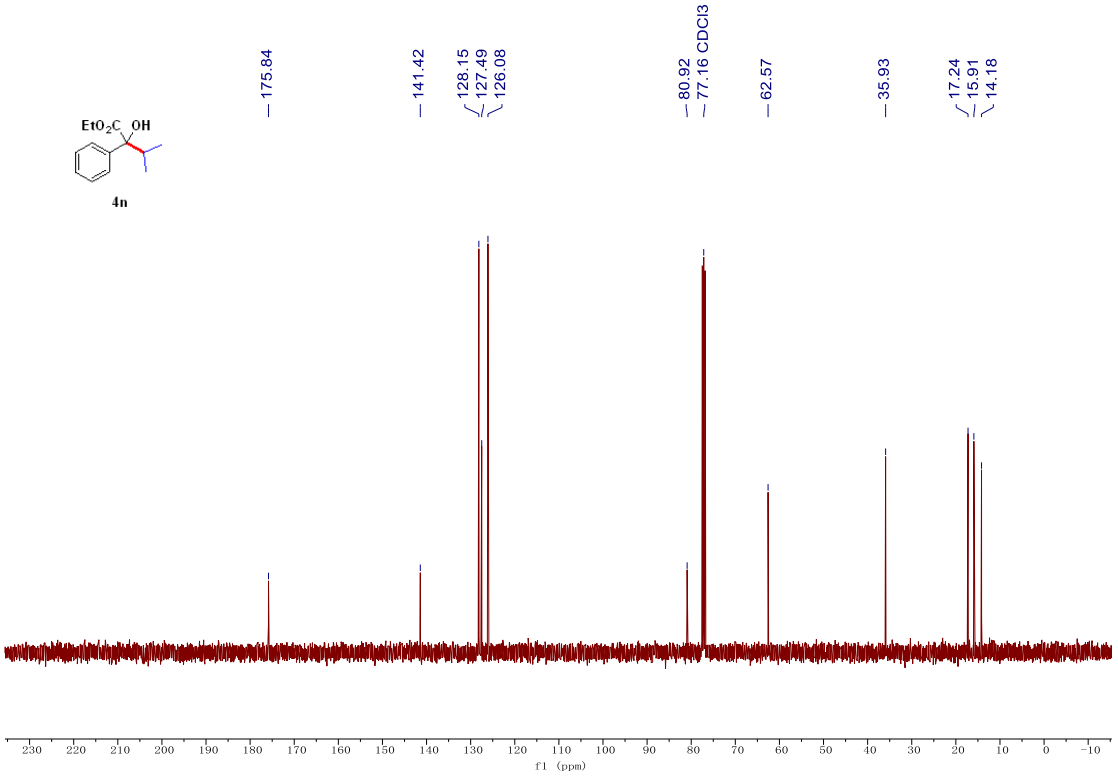
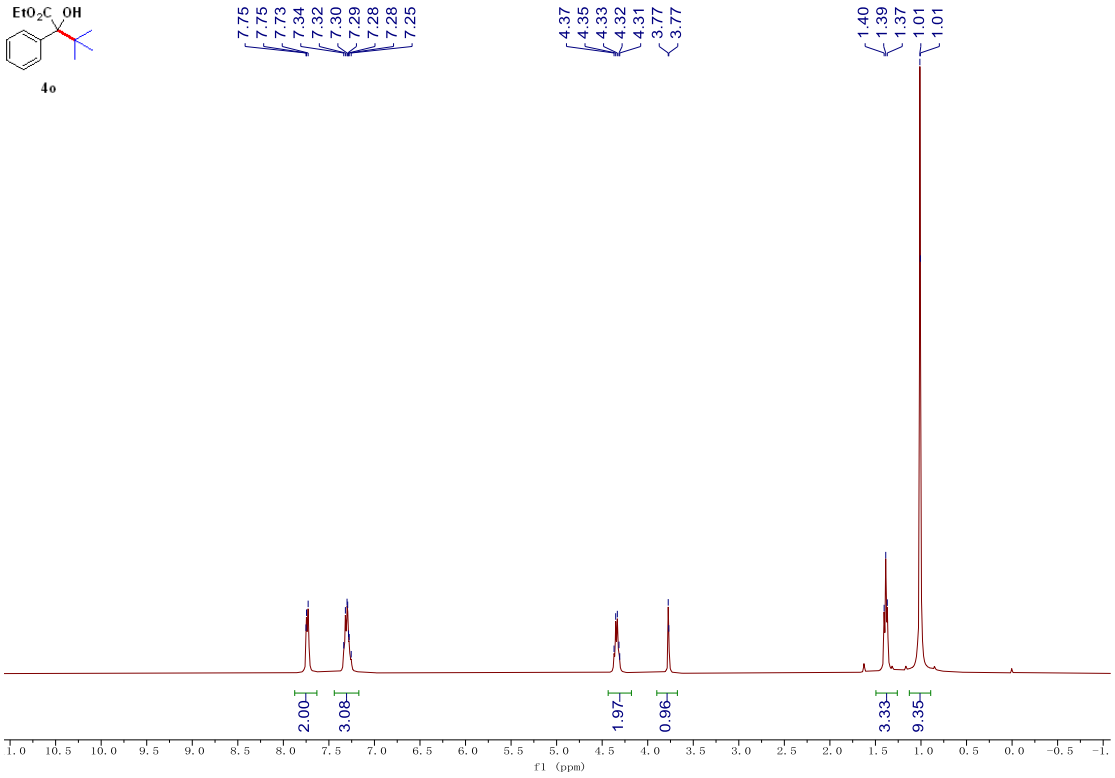
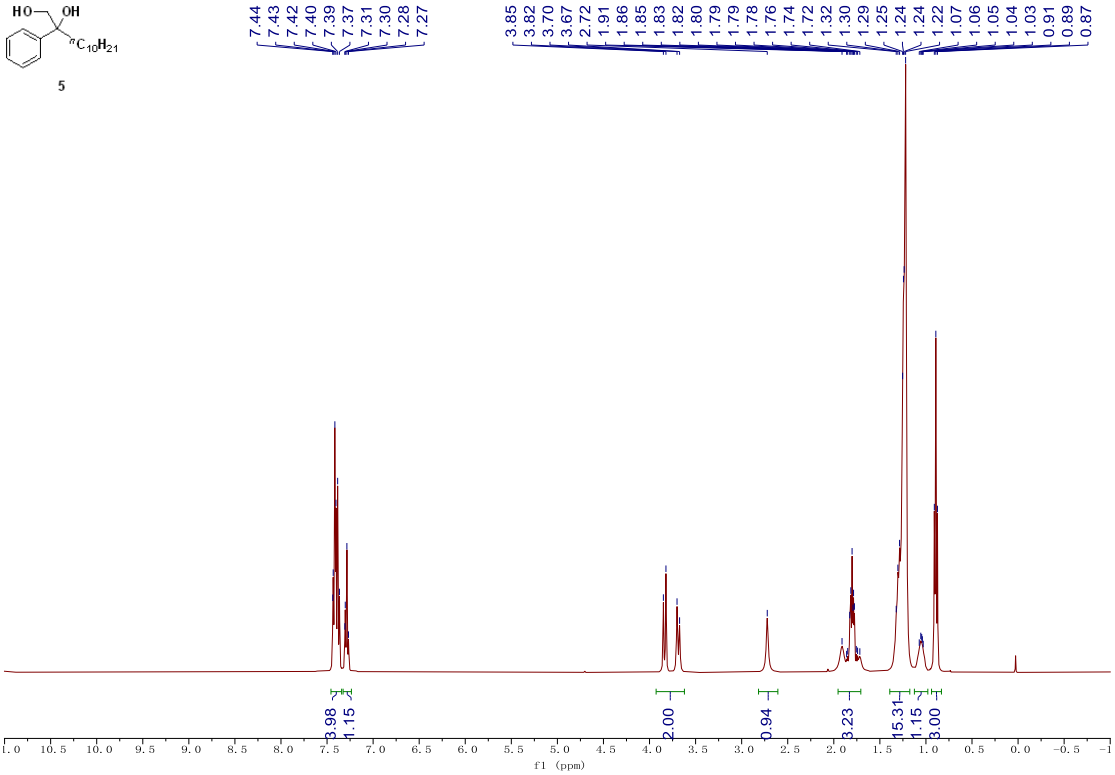
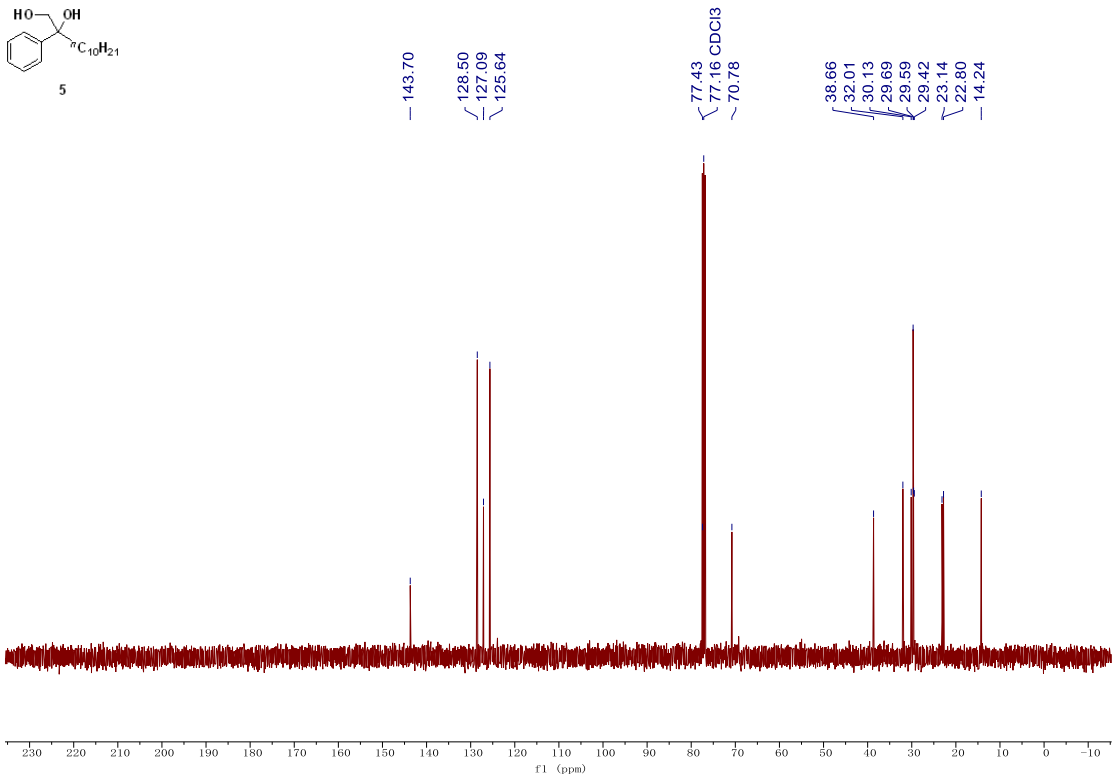
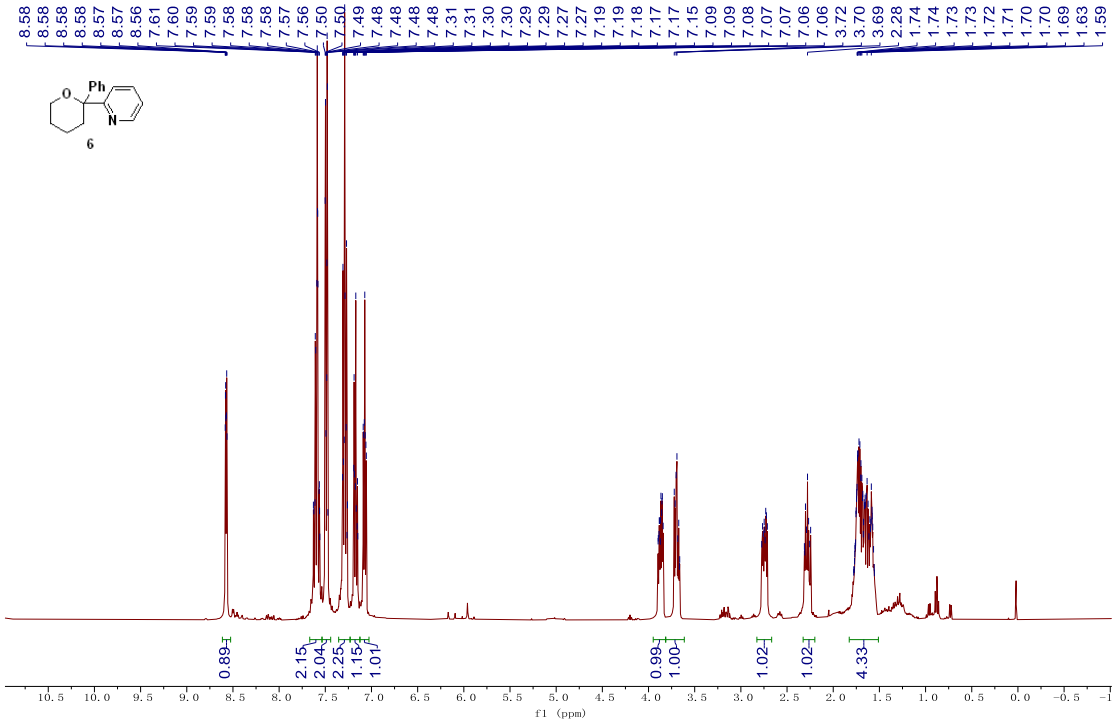
1.95
1.01
2.02
0.95
1.04
3.01
3.01
3.08
f1 (ppm)

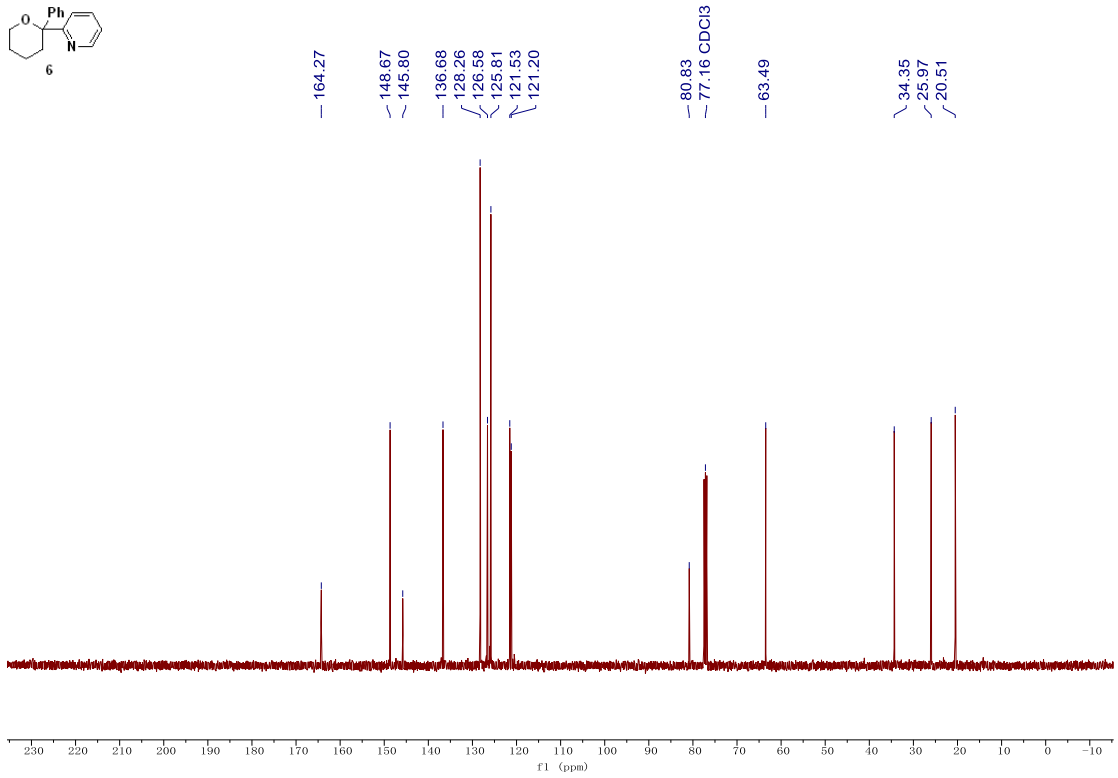

175.84
141.42
128.15
127.49
126.08
80.92
77.16 CDCl3
62.57
35.93
17.24
15.91
14.18
EtO2C OH
4n
f1 (ppm)


EtO2C OH
4o
7.75
7.75
7.73
7.34
7.32
7.30
7.29
7.28
7.28
7.25
4.37
4.35
4.33
4.32
4.31
3.77
3.77
1.40
1.39
1.37
1.01
1.01
2.00
3.08
1.97
0.96
3.33
9.35
f1 (ppm)


HO OH
C10H21
5
7.44
7.43
7.42
7.40
7.39
7.37
7.31
7.30
7.28
7.27
3.85
3.82
3.70
3.67
2.72
1.91
1.86
1.85
1.83
1.82
1.80
1.79
1.79
1.78
1.76
1.74
1.72
1.32
1.30
1.29
1.25
1.24
1.24
1.22
1.07
1.06
1.05
1.04
1.03
0.91
0.89
0.87
3.98
1.15
2.00
0.94
3.23
15.31
1.15
3.00
f1 (ppm)


HO
OH
$^{n}C_{10}H_{21}$
5
143.70
128.50
127.09
125.64
77.43
77.16 CDCl3
70.78
38.66
32.01
30.13
29.69
29.59
29.42
23.14
22.80
14.24
f1 (ppm)


Ph
O
N
6
0.89
2.15
2.04
2.25
1.15
1.01
0.99
1.00
1.02
1.02
4.33
f1 (ppm)


Ph
O
N
6
164.27
148.67
145.80
136.68
128.26
126.58
125.81
121.53
121.20
80.83
77.16 CDCl3
63.49
34.35
25.97
20.51
f1 (ppm)


8.54
8.53
7.58
7.57
7.56
7.55
7.54
7.29
7.27
7.26
7.25
7.19
7.17
7.15
7.09
7.08
7.07
7.05
4.14
4.12
4.10
4.09
4.04
4.02
4.02
4.00
4.00
3.98
3.14
3.13
3.12
3.11
3.10
3.09
3.08
2.46
2.44
2.43
2.42
2.41
2.39
2.00
1.99
1.98
1.97
1.97
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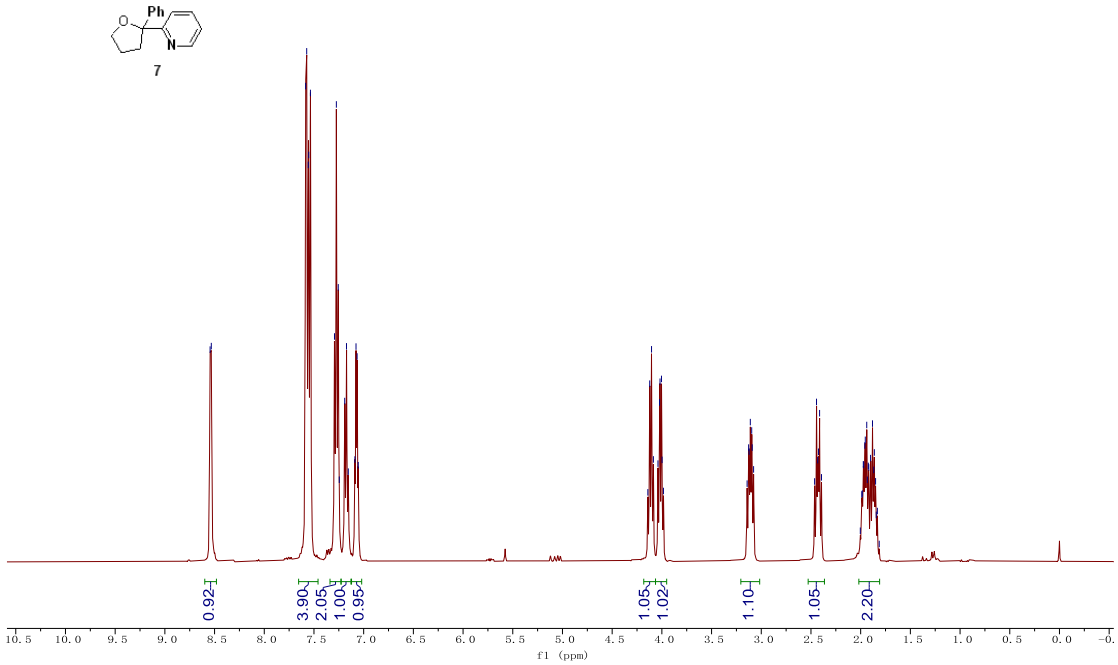
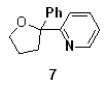
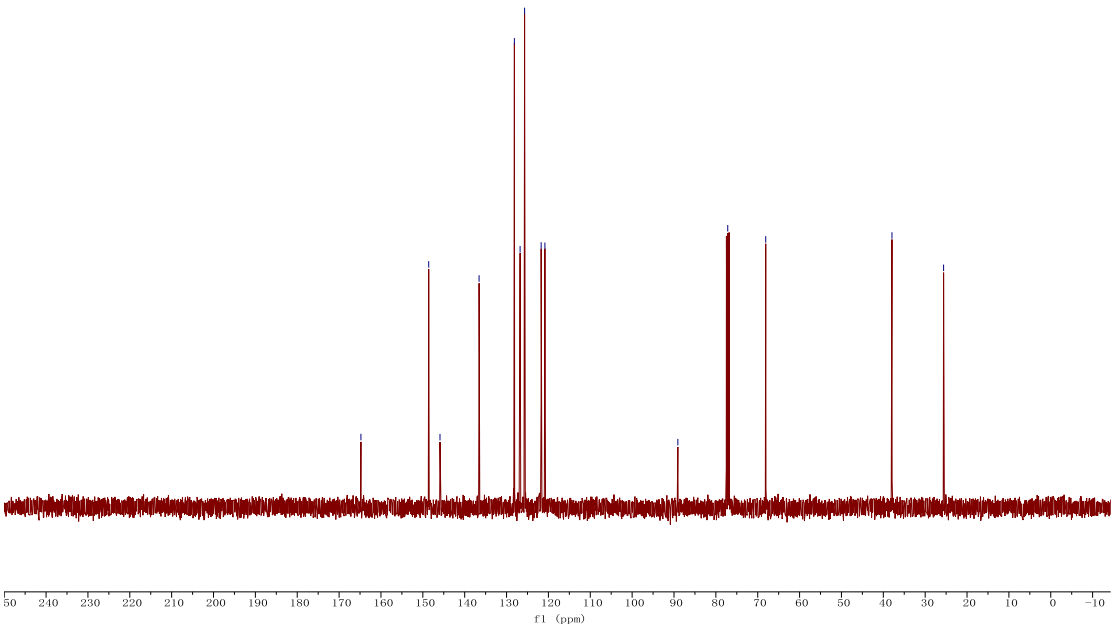
1.88
1.87
1.86
1.85
1.84
1.83


Ph
O
N
7
0.92
3.90
2.05
1.00
0.95
1.05
1.02
1.10
1.05
2.20
f1 (ppm)



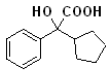
7

164.79
148.59
145.90
136.56
128.13
126.77
125.69
121.73
120.84
89.08
77.16 CDCl3
68.09
37.93
25.60



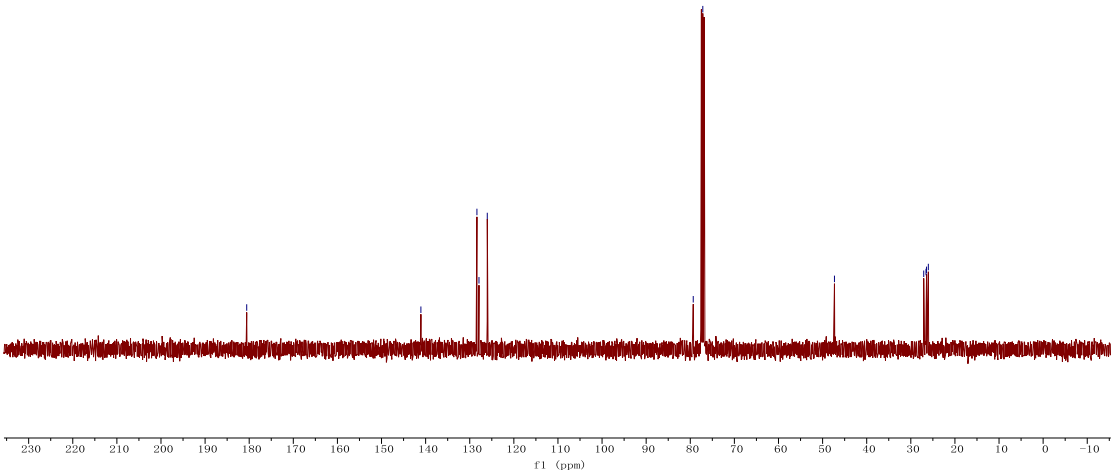
50 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10

f1 (ppm)



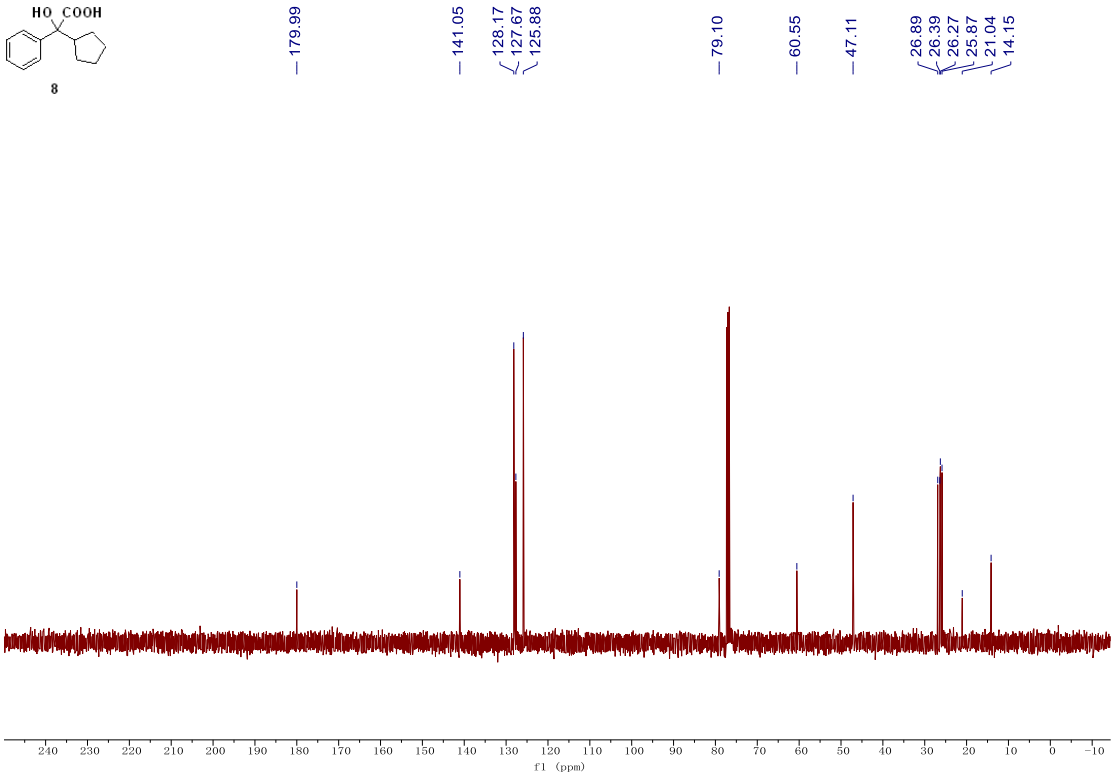
8

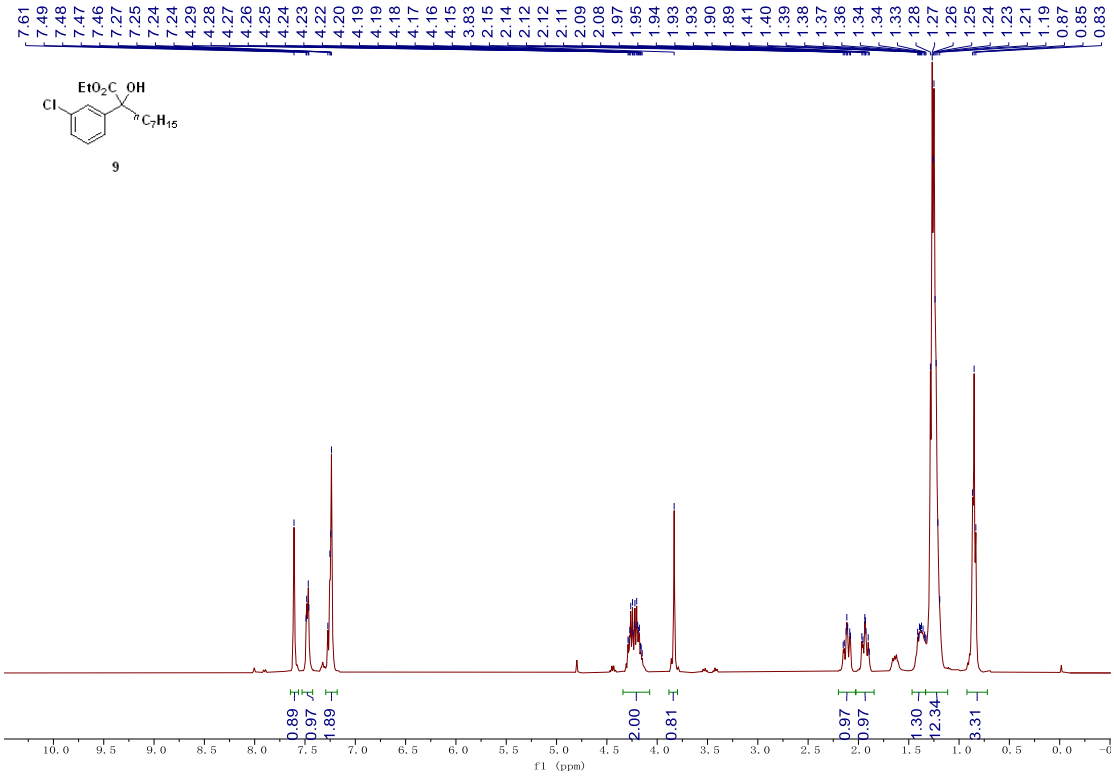
180.56
141.07
128.37
127.90
126.02
79.33
77.16 CDCl3
47.31
27.07
26.56
26.43
26.03



230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10

f1 (ppm)


HO COOH
8
179.99
141.05
128.17
127.67
125.88
79.10
60.55
47.11
26.89
26.39
26.27
25.87
21.04
14.15
f1 (ppm)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7.48
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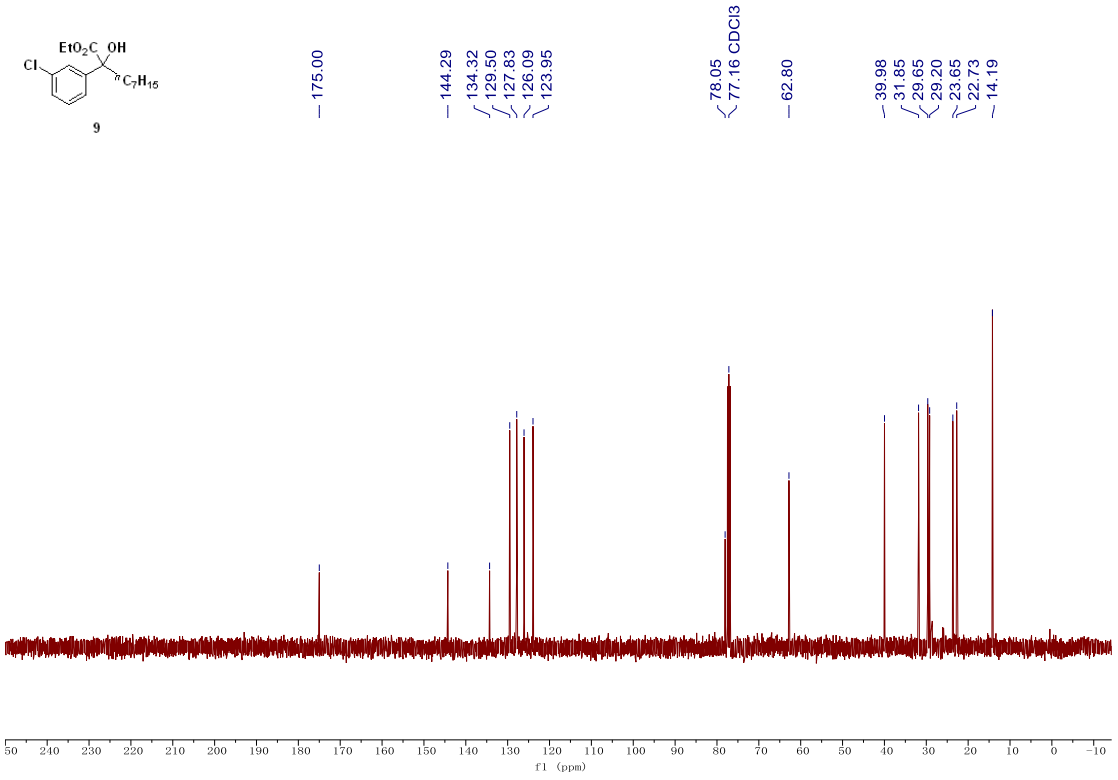
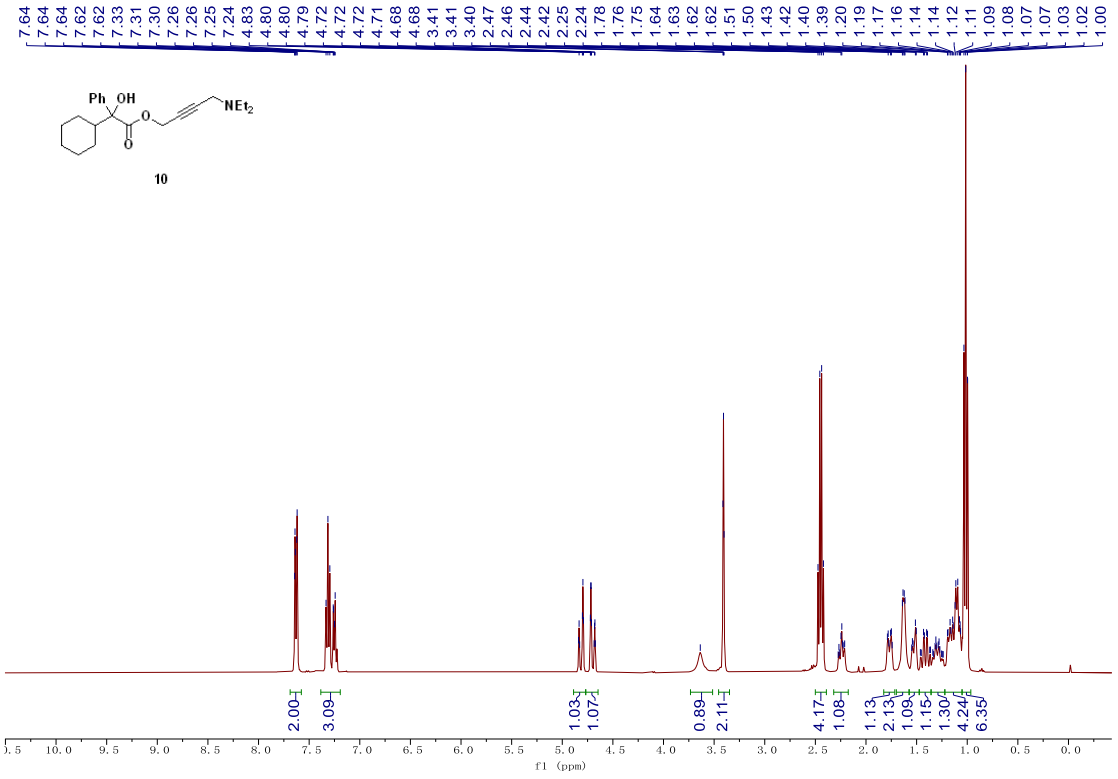
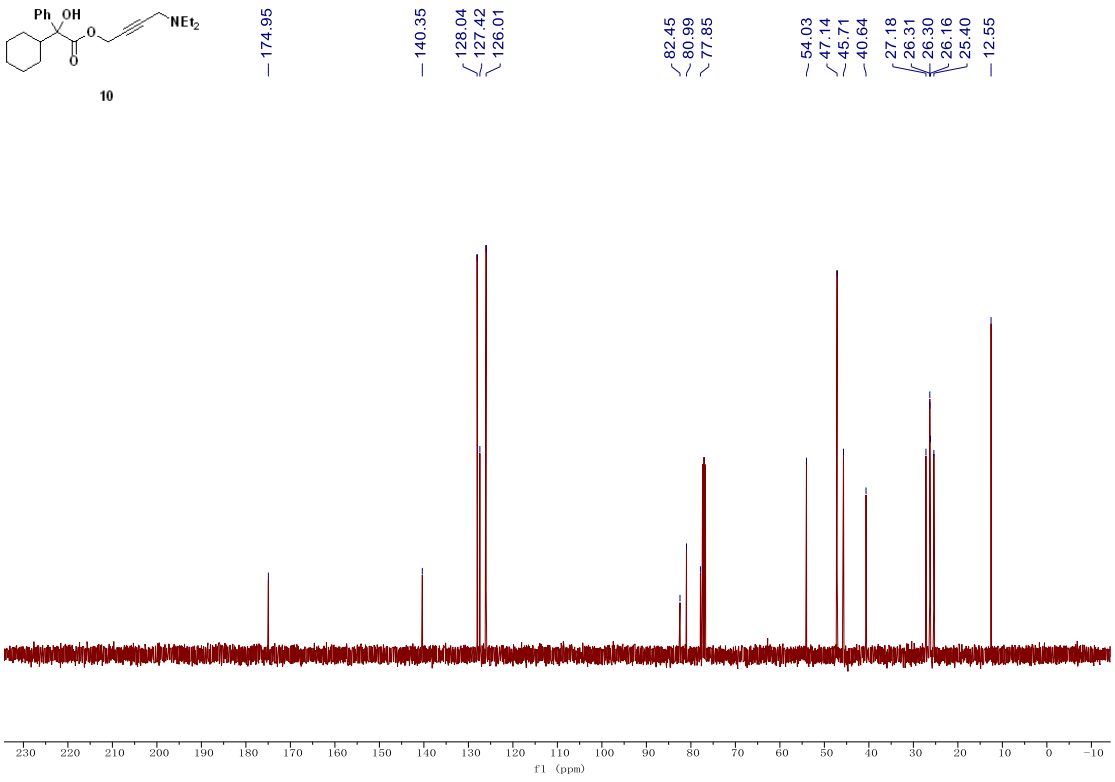
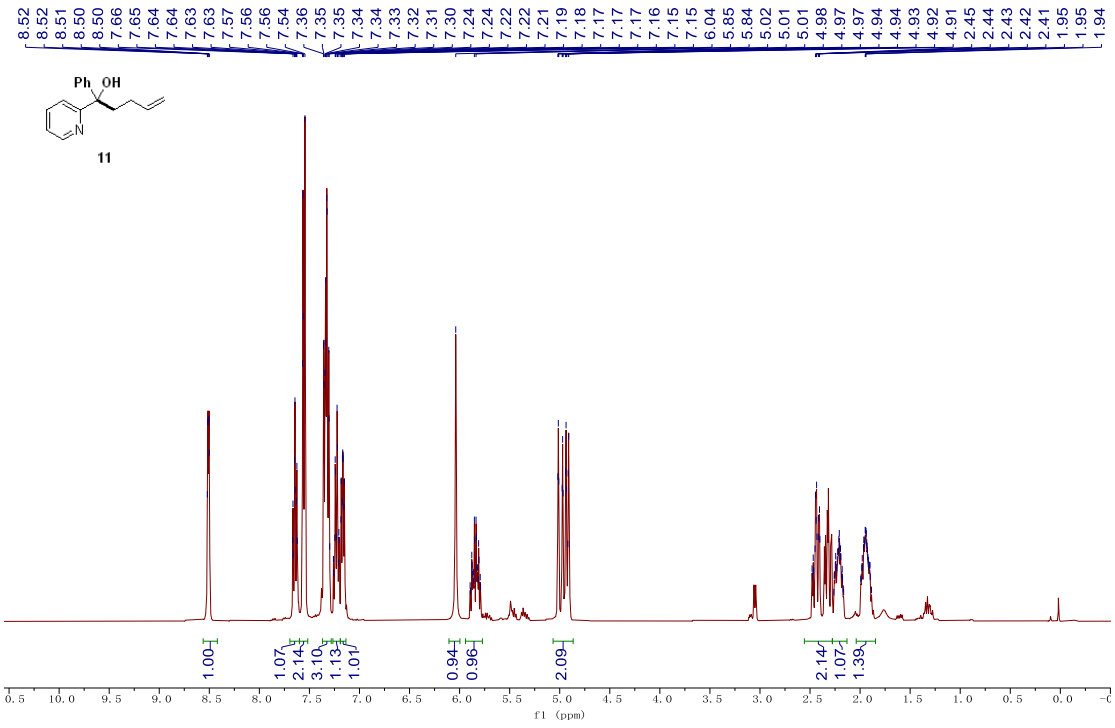
7.27
7.25
7.24
7.24
4.29
4.28
4.27
4.26
4.25
4.24
4.23
4.22
4.20
4.19
4.19
4.18
4.17
4.16
4.15
3.83
2.15
2.14
2.12
2.12
2.11
2.09
2.08
1.97
1.95
1.94
1.93
1.90
1.89
1.41
1.40
1.39
1.38
1.37
1.36
1.34
1.34
1.33
1.28
1.27
1.26
1.25
1.24
1.23
1.21
1.19
0.87
0.85
0.83
EtO2C OH
Cl
nC7H15
9
0.89
0.97
1.89
2.00
0.81
0.97
0.97
1.30
12.34
3.31
f1 (ppm)


EtO2C OH
Cl
nC7H15
9
175.00
144.29
134.32
129.50
127.83
126.09
123.95
78.05
77.16 CDCl3
62.80
39.98
31.85
29.65
29.20
23.65
22.73
14.19
f1 (ppm)


Ph OH
NEt2
O
O
10
2.00
3.09
1.03
1.07
0.89
2.11
4.17
1.08
1.13
2.13
1.09
1.15
1.30
4.24
6.35
f1 (ppm)


Ph OH
NEt2
O
O
10
174.95
140.35
128.04
127.42
126.01
82.45
80.99
77.85
54.03
47.14
45.71
40.64
27.18
26.31
26.30
26.16
25.40
12.55
f1 (ppm)


8.52
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8.50
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7.64
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7.31
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7.22
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7.17
7.17
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7.15
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4.94
4.94
4.93
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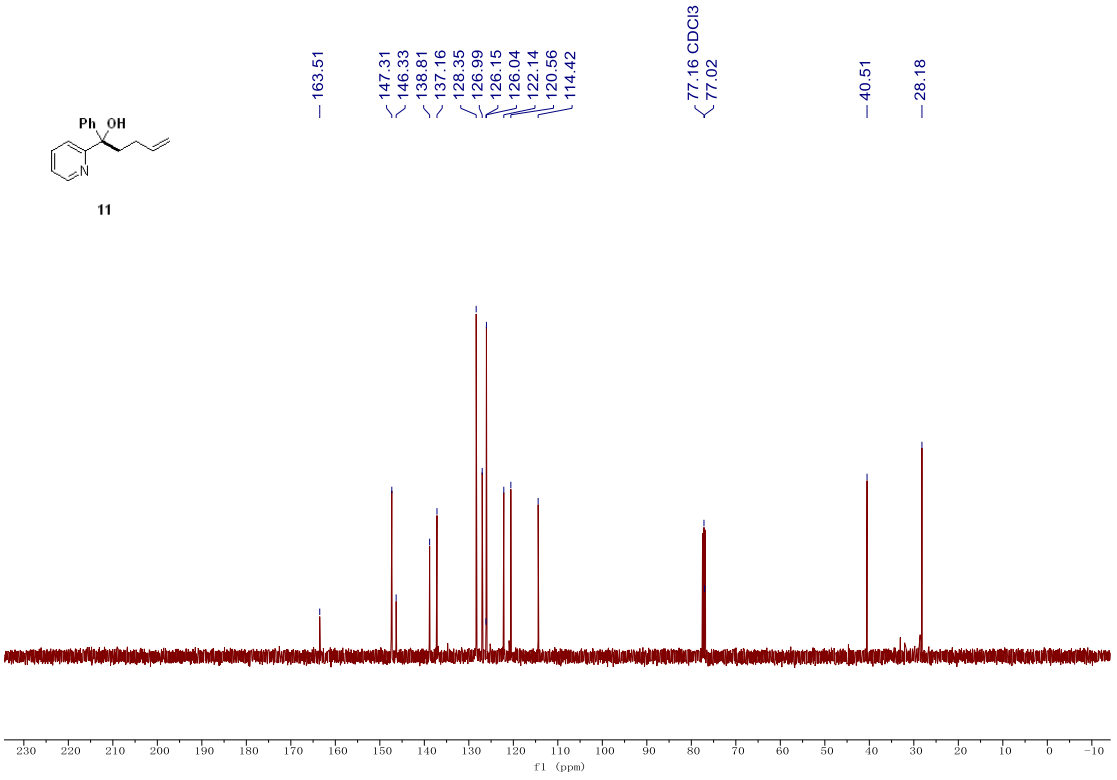
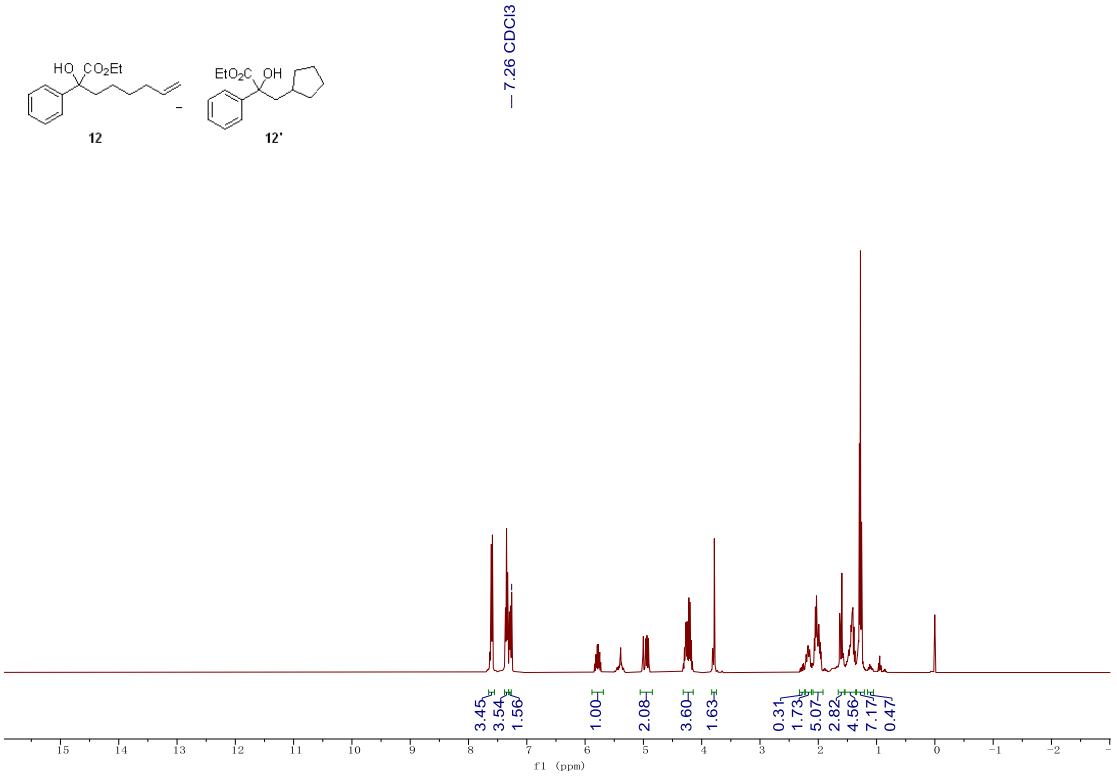
4.91
2.45
2.44
2.43
2.42
2.41
1.95
1.95
1.94
Ph OH
N
11
1.00
1.07
2.14
3.10
1.13
1.01
0.94
0.96
2.09
2.14
1.07
1.39
f1 (ppm)


Ph OH
N
11
163.51
147.31
146.33
138.81
137.16
128.35
126.99
126.15
126.04
122.14
120.56
114.42
77.16 CDCl3
77.02
40.51
28.18
f1 (ppm)


HO CO2Et
12
EtO2C OH
12'
7.26 CDCl3
3.45
3.54
1.56
1.00
2.08
3.60
1.63
0.31
1.73
5.07
2.82
4.56
7.17
0.47
f1 (ppm)

9. References

1. S. Xie, D. Li, H. Huang, F. Zhang and Y. Chen, *J. Am. Chem. Soc.*, 2019, **141**, 16237-16242.
2. Z. M. Rubanov, V. V. Levin and A. D. Dilman, *Org. Lett.*, 2024, **26**, 3174-3178.
3. X. Wang, B. Zhu, J. Dong, H. Tian, Y. Liu, H. Song and Q. Wang, *Chem. Commun.*, 2021, **57**, 5028-5031.
4. K. Ota, K. Nagao and H. Ohmiya, *Org. Lett.*, 2021, **23**, 4420-4425.
5. H. Xia, X. Jiang, D. Lin, S. Zhang, Z. Yu, X. Wu, J. Qu and Y. Chen, *J. Am. Chem. Soc.*, 2024, **146**, 28468-28481.