Electronic Supplementary Material (ESI) for Chemical Communications.

Supporting Information for

Biphosphine copolymer encapsulated single-atom Rh catalyst for regioselective hydroaminomethylation of alkenes

Kang Zhao,[a,b] Hongli Wang,[a,b] Xinzhi Wang,[a,b] Xinjiang Cui,*[a] and Feng Shi*[a]

[a.] State Key Laboratory for Oxo Synthesis and Selective Oxidation, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, No.18, Tianshui Middle Road, Lanzhou, 730000, People's Republic of China

[b.] University of Chinese Academy of Sciences, No. 19A, Yuquanlu, Beijing, 100049, People's Republic of China

E-mail: xinjiangcui@licp.cas.cn, fshi@licp.cas.cn

Contents

1. General Information

Chemicals and materials

All solvents and chemicals, unless otherwise noted, were obtained commercially and were used as received without further purification. All glassware was dried before using. Analytical thin layer chromatography (TLC) was performed using pre-coated Jiangyou silica gel HSGF254 (0.2mm±0.03mm). Flash chromatography was performed using silica gel 60, 0.063-0.2 mm, 200-300 mesh (Jiangyou, Yantai) with the indicated solvent system.

Instrumental measurements and physical characterization

Gas chromatography analysis was performed on Agilent 7890A GC equipped with a HP-5 capillary column and FID detector. GC-MS analysis was in general recorded on an Agilent 5977A MSD GC-MS.

High resolution mass spectra were recorded on a Bruker Q-TOF II MSD.

The contents of Rh in the catalysts were measured by inductively coupled plasma-atomic emission spectrometry (ICP-AES), using Iris advantage Thermo Jarrel Ash device.

Fourier transform infrared (FT-IR) spectrum were recorded with a Bruker VERTEX 70FTIR spectrometer.

The liquid nuclear magnetic resonance spectra (NMR) were recorded on a Bruker AvanceTM III 400 MHz in deuterated chloroform unless otherwise noted. Data are reported in parts per million (ppm) as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet, dd = doublet of doublet and br = broad signal), coupling constant in Hz and integration.

Solid-state nuclear magnetic resonance (SSNMR, ^{13}C and ^{31}P) were carried out on a Bruker NEO WB 400M spectrometer, a sample spinning rate of 5.0 kHz was used for ^{13}C, a sample spinning rate of 10.0 kHz was used for ^{31}P.

Powder X-ray diffraction (PXRD) measurements were conducted were conducted by a STADIP automated transmission diffractometer (STOE) equipped with an incident beam curved germanium monochromator selecting CuKα1 radiation and a 6° position sensitive detector (PSD) (step size: 0.014°, step time: 25.05 s). The XRD patterns were scanned in the 2θ range of 10-90°.

Nitrogen adsorption-desorption isotherms were measured at 77 K using an American Quantachrome iQ_2 automated gas sorption analyzer. The samples were outgassed at 120 °C for 12 h before the measurements. Surface areas were calculated from the adsorption data using Langmuir and Brunauer-Emmett-Teller (BET) methods. The pore-size-distribution curves were obtained from the adsorption branches using non-local density functional theory (NLDFT) method.

X-ray photoelectron spectroscopy (XPS) measurements were carried out by a VG ESCALAB 210 instrument equipped with a dual Mg/Al anode X-ray source, a hemispherical capacitor analyzer, and a 5 keV Ar^+ ion gun. All spectra were recorded by using AlKa (1361 eV) radiation. The electron binding energy was referenced to the C1s peak at 284.8 eV.

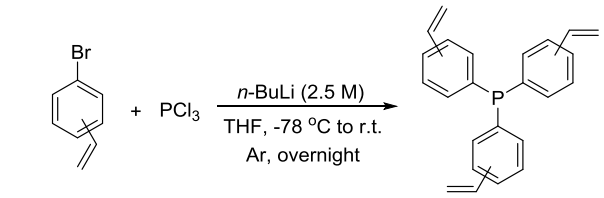
The thermal properties of Rh@POPs catalysts were evaluated using a METTLER TOLEDO simultaneous thermal analyzer over the temperature range from 30 to 800 °C under nitrogen atmosphere (20 mL/min) with a heating rate of 5 °C/min.

Field emission scanning electron microscopy (SEM) observations were performed on a Hitachi S-4800 microscope operated at an accelerating voltage of 5.0 kV.

High-resolution transmission electron microscope (HR-TEM) analysis was carried out on a Talos F200S operating at 200 kV.

The abreaction-corrected HAADF-STEM images were obtained on a Titan FEI Titan Themis 60-300 equipped with a probe corrector, with a guaranteed resolution of 80 pm and accelerating voltage of 200 kV.

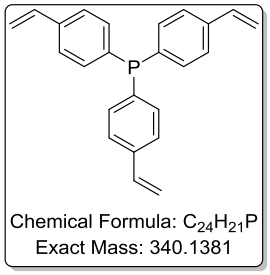
2. Synthetic procedure of the ligands



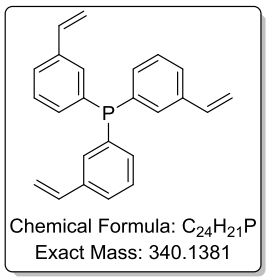
p or *m*-bromovinylbenzene

p-3vPPh$_3$ or *m*-3vPPh$_3$

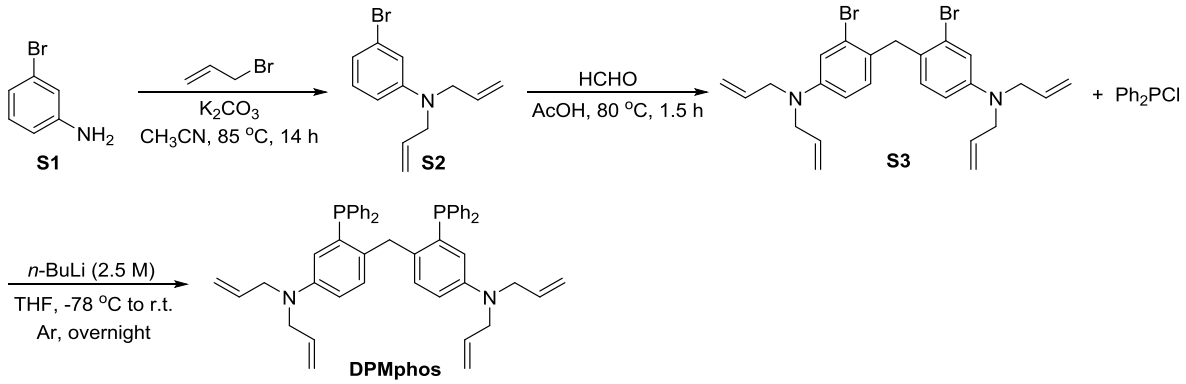
A solution of *n*-butyllithium in hexane (2.5 M, 12.6 ml, 31.5 mmol) was added dropwise over a period of 20 min to a solution of *p* or *m*-bromovinylbenzene (6.04 g, 33 mmol) in anhydrous tetrahydrofuran (40 ml) at -78 °C under argon atmosphere. The solution was stirred for 1 h and then phosphorus trichloride (1.37 g, 10 mmol) dissolved in anhydrous tetrahydrofuran (5 ml) was added dropwise over a period of 5 min. The mixture was continued to stir at -78 °C for 1 h and the system was recovered to room temperature, and allowed to react overnight. The reaction was quenched with 2N HCl solution. The mixture was extracted with ethyl acetate and water for 3 times, the combined organic phases were dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using EtOAc–petroleum ether mixture as an eluent to afford the desired compound *p*-3vPPh$_3$ or *m*-3vPPh$_3$ as a white solid.



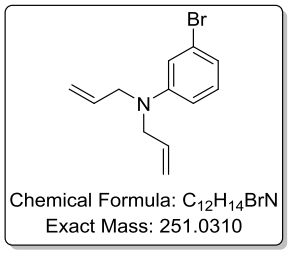
Tris(4-vinylphenyl)phosphane (*p*-3vPPh$_3$)[1]: white solid, 2.4 g, 70% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.0 Hz, 6H), 7.27 (t, *J* = 7.7 Hz, 6H), 6.69 (dd, *J* = 17.6, 10.9 Hz, 3H), 5.76 (d, *J* = 17.6 Hz, 3H), 5.26 (d, *J* = 10.9 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.09, 136.81, 136.70, 136.47, 134.09, 133.90, 126.47, 126.40, 114.82. **^{31}P NMR** (162 MHz, $CDCl_3$) δ -6.85.



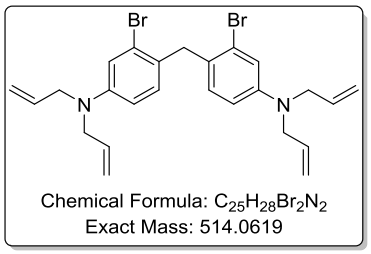
Tris(3-vinylphenyl)phosphane (*m*-3vPPh$_3$)[1]: white solid, 2.4 g, 70% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.1 Hz, 6H), 7.34 – 7.24 (m, 3H), 7.17 (t, *J* = 7.2 Hz, 3H), 6.64 (dd, *J* = 17.6, 10.9 Hz, 3H), 5.67 (d, *J* = 17.6 Hz, 3H), 5.21 (d, *J* = 11.0 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 137.80 (d, *J* = 7.7 Hz), 137.33 (d, *J* = 11.1 Hz), 136.59, 133.13 (d, *J* = 16.5 Hz), 132.01 (d, *J* = 23.0 Hz), 128.85 (d, *J* = 6.3 Hz), 126.63, 114.55. **^{31}P NMR** (162 MHz, $CDCl_3$) δ -5.15.



Compound S2 and S3 were prepared according to literature precedent.[2]

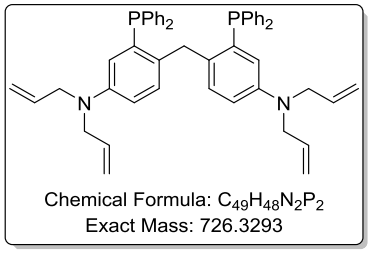


***N*, *N*-Diallyl-3-bromoaniline:** yellow viscous liquid, 17.1 g, 85% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.01 (t, *J* = 8.1 Hz, 1H), 6.78 (dd, *J* = 7.6, 5.3 Hz, 2H), 6.58 (dd, *J* = 8.4, 1.9 Hz, 1H), 5.81 (ddd, *J* = 15.5, 9.7, 4.7 Hz, 2H), 5.37 – 4.81 (m, 4H), 4.19 – 3.59 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 150.00, 133.33, 130.36, 123.45, 119.13, 116.39, 115.08, 110.94, 52.79.



4,4'-Methylenebis(*N*, *N*-diallyl-3-bromoaniline): light yellow viscous liquid, 14 g, 80% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.89 (d, *J* = 2.1 Hz, 1H), 6.81 (d, *J* = 8.6 Hz, 1H), 6.53 (dd, *J* = 8.6, 2.4 Hz, 1H), 5.81 (ddt, *J* = 17.6, 9.7, 4.7 Hz, 2H), 5.39 – 4.93 (m, 5H), 3.95 (s, 1H), 3.86 (d, *J* = 4.4 Hz, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.26, 133.63, 130.91, 127.01, 125.67, 116.38, 116.07, 111.76, 52.85, 39.88.

A solution of *n*-butyllithium in hexane (2.5 M, 8.4 ml, 21 mmol) was added dropwise over a period of 10 min to a solution of S3 (5.16 g, 10 mmol) in anhydrous tetrahydrofuran (60 ml) at -78 °C under argon atmosphere. The solution was stirred for 1 h and then chlorodiphenylphosphine (4.84 g, 22 mmol) dissolved in anhydrous tetrahydrofuran (5 ml) was added dropwise. The mixture was continued to stir at -78 °C for 1 h and the system was recovered to room temperature, and allowed to react overnight. The reaction was quenched with 2N HCl solution. The mixture was extracted with ethyl acetate and water for 3 times, the combined organic phases were dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using EtOAc–petroleum ether mixture as an eluent to afford the desired compound DPMphos as a colorless viscous liquid, 2.4 g, 33% yield.



4,4'-Methylenebis(*N,N*-diallyl-3-(diphenylphosphaneyl)aniline) (DPMphos): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (s, 20H), 6.78 (dd, *J* = 7.5, 4.3 Hz, 2H), 6.48 (d, *J* = 6.9 Hz, 2H), 6.18 (s, 2H), 5.82 – 5.44 (m, 4H), 4.94 (dd, *J* = 21.7, 13.8 Hz, 8H), 4.19 (s, 2H), 3.62 (s, 8H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.63, 137.56, 137.45, 136.17, 136.05, 134.21, 134.10, 134.01, 133.74, 130.74, 130.70, 130.66, 128.31, 128.26, 117.89, 116.07, 113.16, 52.99, 36.18. **^{31}P NMR** (162 MHz, $CDCl_3$) δ -12.84. HRMS (ESI): Calcd. for $C_{49}H_{49}N_2P_2$ $[M+H]^+$: 727.3371, found: 727.3365.

3. Synthetic procedure of the porous organic polymer catalysts

Preparation of POL-*p*-3vPPh$_3$

Under argon atmosphere, 0.48 g of *p*-3vPPh$_3$ was dissolved in 5 mL of THF, followed by the addition of 16 mg of AIBN. The mixture was transferred into an autoclave, stirred at room temperature for 0.5 h, and then heated to 100 ^{o}C for 24 h without stirring. After the polymerization was finished, the resulted white solid was filtered, washed with THF (20 ml×3), and dried under vacuum at 60 ^{o}C for 12 h. Finally, POL-*p*-3vPPh$_3$ was obtained.

Preparation of CPOL-DPMphos&*p*-3vPPh$_3$

Under argon atmosphere, 0.48 g of *p*-3vPPh$_3$ and 0.12 g of DPMphos was dissolved in 6 mL of THF, followed by the addition of 16 mg of AIBN. The mixture was transferred into an autoclave, stirred at room temperature for 0.5 h, and then heated to 100 ^{o}C for 24 h without stirring. After the polymerization was finished, the resulted yellowish solid was filtered, washed with THF (20 ml×3), and dried under vacuum at 60 ^{o}C for 12 h. Finally, CPOL-DPMphos&*p*-3vPPh$_3$ was obtained.

Preparation of Rh/POL-*p*-3vPPh$_3$

Under argon atmosphere, 7.7 mg of Rh(CO)$_2$(acac) was dissolved in 10 mL of THF, followed by the addition of 0.43 g of CPOL-DPMphos&*p*-3vPPh$_3$. After stirring for 24 h under argon atmosphere at room temperature, the resulted yellowish solid was filtered, washed with THF (20 ml×3), and dried under vacuum at 60 ^{o}C for 12 h, quantitative yield of the Rh/POL-*p*-3vPPh$_3$ was obtained. 0.426 wt.% of Rh contents was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES).

Preparation of Rh/CPOL-DPMphos&*p*-3vPPh$_3$

Under argon atmosphere, 10.32 mg of Rh(CO)$_2$(acac) was dissolved in 10 mL of THF, followed by the addition of 0.5 g of CPOL-DPMphos&*p*-3vPPh$_3$. After stirring for 24 h under argon atmosphere at room temperature, the resulted yellowish solid was filtered, washed with THF (20 ml×3), and dried under vacuum at 60 ^{o}C for 12 h, quantitative yield of the Rh/CPOL-DPMphos&*p*-3vPPh$_3$ was obtained. 0.520 wt.% of Rh contents was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES).

Preparation of Rh@POL-*p*-3vPPh$_3$ or Rh@POL-*m*-3vPPh$_3$

Under argon atmosphere, 0.48 g of *p*-3vPPh$_3$ and 7.7 mg of Rh(CO)$_2$(acac) was dissolved in 10 mL of THF, the mixture was stirred at room temperature for 3 h, and evaporated to dryness under vacuum. Then, the dried mixture and 16 mg of AIBN were dissolved in 5 mL of THF under argon atmosphere. The obtained solution was transferred into an autoclave, stirred at room temperature for 0.5 h, and then heated to 100 ^{o}C for 24 h without stirring. After the polymerization was finished, the resulted yellowish solid was filtered, washed with THF (20 mL×3), and dried under vacuum at 60 ^{o}C for 12 h. Finally, Rh@POL-*p*-3vPPh$_3$ was obtained. 0.517 wt.% of Rh contents was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES).

Rh@POL-*m*-3vPPh$_3$ was obtained as a yellowish solid according to the above procedure just by replacing 0.48 g of *p*-3vPPh$_3$ with 0.48 g of *m*-3vPPh$_3$. 0.507 wt.% of Rh contents was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES).

Preparation of Rh@CPOL-DPMphos&*p*-3vPPh$_3$ or Rh@CPOL-DPMphos&*m*-3vPPh$_3$

Under argon atmosphere, 0.12 g of DPMphos and 10.32 mg of Rh(CO)$_2$(acac) was dissolved in 10 mL of THF, the mixture was stirred at room temperature for 3 h, and evaporated to dryness under vacuum. Then, the dried mixture, 0.48 g of *p*-3vPPh$_3$, and 16 mg of AIBN were dissolved in 6 mL of THF under argon atmosphere. The obtained solution was transferred into an autoclave, stirred at room temperature for 0.5 h, and then heated to 100 ^{o}C for 24 h without stirring. After the polymerization was finished, the resulted yellowish solid was filtered, washed with THF (20 mL×3), and dried under vacuum at 60 ^{o}C for 12 h. Finally, Rh@CPOL-DPMphos&*p*-3vPPh$_3$ was obtained. 0.434 wt.% of Rh contents was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES).

Rh@CPOL-DPMphos&*m*-3vPPh$_3$ was obtained as a yellowish solid according to the above procedure just by replacing 0.48 g of *p*-3vPPh$_3$ with 0.48 g of *m*-3vPPh$_3$. 0.428 wt.% of Rh contents was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES).

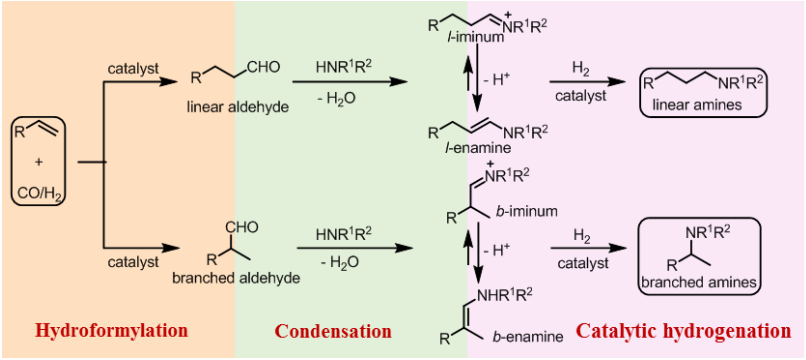
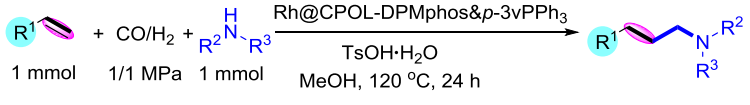
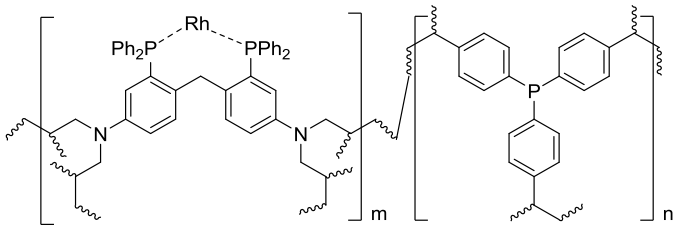


Figure S1. General mechanism of hydroaminomethylation of alkenes and amines.

4. General procedure for the hydroaminomethylation

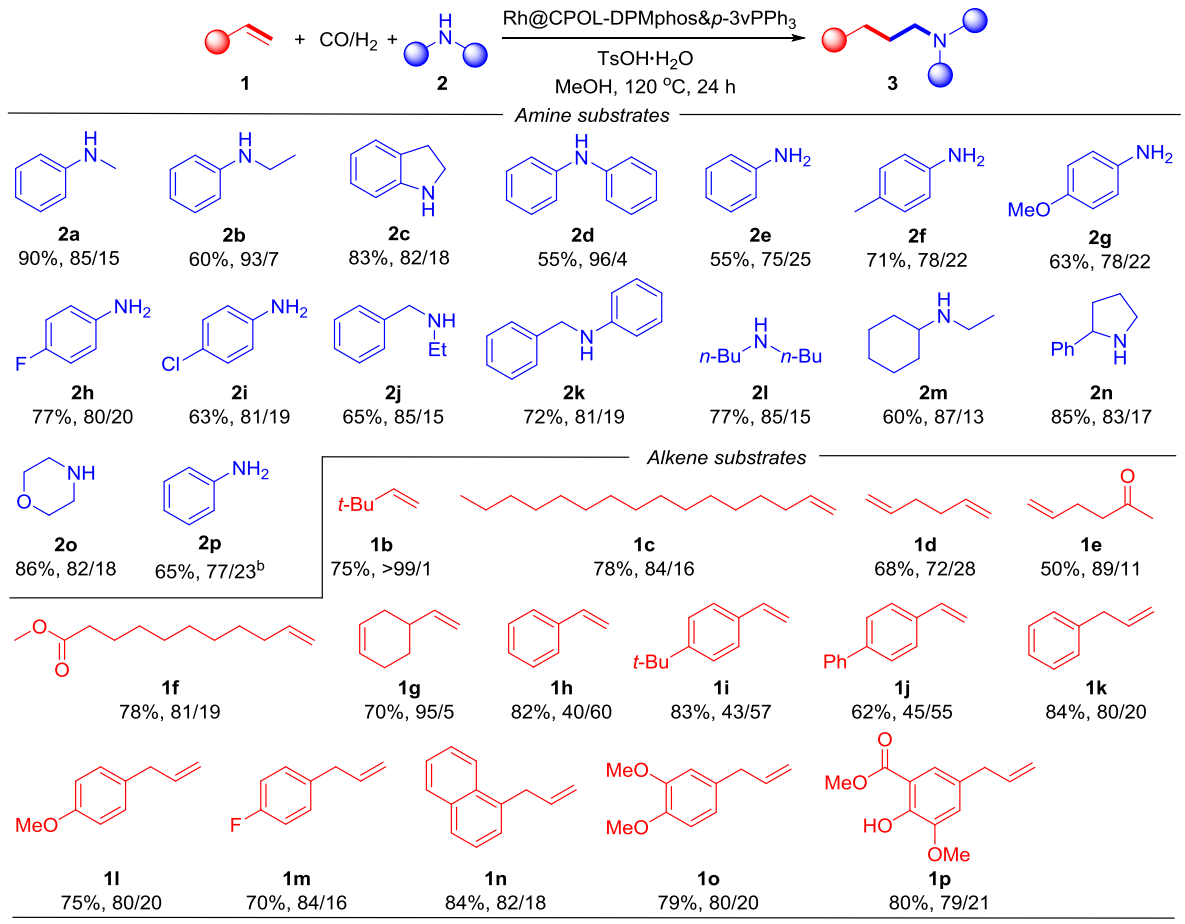




Rh@CPOL-DPMphos&*p*-3vPPh$_3$

As a typical hydroaminomethylation recipe, the as-prepared Rh@CPOL-DPMphos&*p*-3vPPh$_3$ catalyst (25 mg containing 1.0 μmol of Rh), 1-octene (112 mg, 1.0 mmol), *N*-Methylaniline (108 mg, 1.0 mmol), PTSA.H_2O (15.2 mg, 0.08 mol) and MeOH (2.0 ml) were added into a 80 ml stainless-steel autoclave with a magnetic stir bar. After the autoclave was sealed and purged with CO for four times, the pressure of syngas (CO/H_2 = 1:1) was adjusted to 2 MPa and the autoclave was put into a preheated reactor, then stirring at 120 °C for 24 h. After the reaction, the autoclave was cooled to room temperature and the pressure was carefully released. Subsequently, the reaction mixture was diluted with ethyl acetate (6 ml), and the catalyst was removed from the system by centrifugation and analyzed by gas chromatography (Agilent 7890A GC equipped with a HP-5 capillary column with 5 wt.% phenyl groups and the FID detector) to give the ratio of l/b of the products. GC yield was obtained by GC analysis using *n*-decane as the internal standard, isolated yield was obtained by flash column chromatography on silica gel using EtOAc–petroleum ether mixture as an eluent.

For recycling, the Rh@CPOL-DPMphos&*p*-3vPPh$_3$ catalyst was separated by centrifugation, washed with MeOH (8.0 ml×3), and used directly for the next run. The Rh contents of the used catalyst and filtrate after each run were determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES).



Scheme S1 Substrate scope of amines and alkenes. [a] Reaction conditions: alkenes (1 mmol), amines (1 mmol), Rh@CPOL-DPMphos&*p*-3vPPh$_3$ (25 mg, containing 1 μmol of Rh), TsOH·H$_2$O (0.08 mmol), total pressure (2 MPa) of syngas (CO : H$_2$ = 1 : 1), methanol (2 ml), 120 °C, 24 h. Isolated yields of the products of *n*- and *iso*-amines. The ratio of *n*-/*iso*-amines was determined by GC analysis. [b] aniline (0.5 mmol), product of double hydroaminomethylation was obtained.

5. Optimization of the reaction conditions

Table S1. Screen of the reaction solvents.[a]

Entry	Sol.	Conv. (%)	Yield (%)	Sel. (%)	
				Amine (l/b)	Others
1	Toluene	96	47	49 (73/27)	51
2	1,4-Dioxane	95	32	34 (63/37)	66
3	THF	95	74	78 (74/26)	22
4	MeOH	95	94	99 (82/18)	1
5	EtOH	95	69	73 (73/27)	27
6	DME	95	69	73 (38/62)	27
7	CH_3CN	95	36	38 (82/18)	62
8	Anisole	61	27	44 (70/30)	56
9	DMF	66	38	58 (75/25)	42
10	*n*-Hexane	97	91	94 (79/21)	6
11	DCE	-	-	-	-

[a] Reaction conditions: 1-octene (1 mmol), *N*-methylaniline (1 mmol), Rh@CPOL-DPMphos&*p*-3vPPh$_3$ (25 mg, containing 1 μmol of Rh), TsOH·H_2O (0.08 mmol), total pressure (6 MPa) of syngas (CO : H_2 = 1 : 5), Solvent (2 ml), 120 °C, 12 h. Conversion, yield and, ratio of *n*-/*iso*-amines were determined by GC analysis using *n*-decane as the internal standard.

Table S2. Screen of CO/H_2 pressure.[a]

Entry	Total pressure (X MPa) of syngas ($CO : H_2 = Y : Z$)	Conv. (%)	Yield (%)	Sel. (%)	
				Amine (l/b)	Others
1	1/5	98	97	99 (82/18)	1
2	1/4	93	91	98 (82/18)	2
3	1/3	96	95	99 (83/17)	1
4	1/2	95	93	98 (84/16)	2
5	1/1	94	86	91 (85/15)	9
6	0.5/0.5	96	50	52(88/12)	48
7	2/2	95	95	>99(78/22)	<1
8	3/3	95	93	98(83/17)	2

[a] Reaction conditions: 1-octene (1 mmol), *N*-methylaniline (1 mmol), Rh@CPOL-DPMphos&*p*-3vPPh$_3$ (25 mg, containing 1 μmol of Rh), $TsOH \cdot H_2O$ (0.08 mmol), total pressure (X MPa) of syngas ($CO : H_2 = Y : Z$), MeOH (2 ml), 120 °C, 12 h. Conversion, yield and, ratio of *n*-/*iso*-amines were determined by GC analysis using *n*-decane as the internal standard.

Table S3. Screen of the reaction temperature.[a]

Entry	T (°C)	Conv. (%)	Yield (%)	Sel. (%)	
				Amine (l/b)	Others
1	80	95	45	47(87/13)	53
2	100	95	70	83(86/14)	17
3	120	96	80	84(87/13)	16
4	140	92	74	83(86/14)	17
5	160	91	20	22(88/12)	78

[a] Reaction conditions: 1-octene (1 mmol), *N*-methylaniline (1 mmol), Rh@CPOL-DPMphos&*p*-3vPPh$_3$ (25 mg, containing 1 μmol of Rh), TsOH·H_2O (0.08 mmol), total pressure (2 MPa) of syngas (CO : H_2 = 1 : 1), MeOH (2 ml), T, 12 h. Conversion, yield and, ratio of *n*-/*iso*-amines were determined by GC analysis using *n*-decane as the internal standard.

Table S4. Screen of PTSA·H_2O dosage.[a]

Entry	PTSA·H_2O (X mmol)	Conv. (%)	Yield (%)	Sel. (%)	
				Amine (l/b)	Others
1	0	95	12	13(59/41)	87
2	0.02	96	35	36(88/12)	64
3	0.04	94	58	62(86/14)	38
4	0.08	90	80	89(85/15)	11
5	0.16	96	78	81(84/16)	19

[a] Reaction conditions: 1-octene (1 mmol), *N*-methylaniline (1 mmol), Rh@CPOL-DPMphos&*p*-3vPPh_3 (25 mg, containing 1 μmol of Rh), TsOH·H_2O (X mmol), total pressure (2 MPa) of syngas (CO : H_2 = 1 : 1), MeOH (2 ml), 120 °C, 12 h. Conversion, yield and, ratio of *n*-/*iso*-amines were determined by GC analysis using *n*-decane as the internal standard.

Table S5. Screen of the Rh dosage.[a]

Entry	Rh content (X mol%)	Conv. (%)	Yield (%)	Sel. (%)	
				Amine (l/b)	Others
1	0.05	95	60	63(85/15)	37
2	0.1	90	80	89(85/15)	11
3	0.15	94	78	82(86/14)	18
4	0.2	95	79	83(85/15)	17
5	0.25	94	79	84(85/15)	16
6	0.3	95	81	84(85/15)	16

[a] Reaction conditions: 1-octene (1 mmol), *N*-methylaniline (1 mmol), Rh@CPOL-DPMphos&*p*-3vPPh$_3$ (Rh, X mol%), TsOH·H_2O (0.08 mmol), total pressure (2 MPa) of syngas (CO : H_2 = 1 : 1), MeOH (2 ml), 120 °C, 12 h. Conversion, yield and, ratio of *n*-/*iso*-amines were determined by GC analysis using *n*-decane as the internal standard.

Table S6. Screen of the reaction time.[a]

Entry	Time (h)	Conv. (%)	Yield (%)	Sel. (%)	
				Amine (l/b)	Others
1	1	95	13	14(86/14)	86
2	3	88	34	39(88/12)	61
3	6	94	49	52(86/14)	48
4	9	94	60	64(86/14)	36
5	12	90	80	89(85/15)	11
6	18	94	87	93(84/16)	7
7	24	96	95 (90)[b]	99(85/15)	1

[a] Reaction conditions: 1-octene (1 mmol), *N*-methylaniline (1 mmol), Rh@CPOL-DPMphos&*p*-3vPPh$_3$ (25 mg, containing 1 μmol of Rh), TsOH·H_2O (0.08 mmol), total pressure (2 MPa) of syngas (CO : H_2 = 1 : 1), MeOH (2 ml), 120 °C, t. Conversion, yield and, ratio of *n*-/*iso*-amines were determined by GC analysis using *n*-decane as the internal standard. [b] Isolated yield.

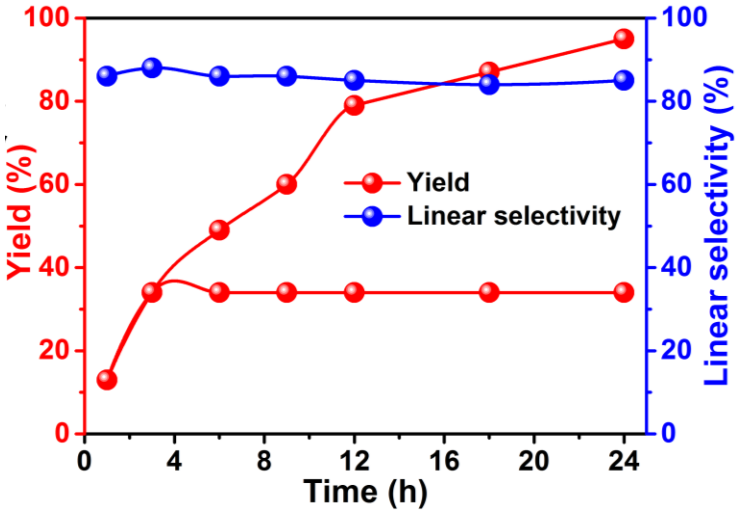


Figure S2. Kinetic curve and hot filtration experiments for hydroaminomethylation of 1-octene.

Table S7. Comparison of the reaction performance of Rh@CPOL-DPMphos&*p*-3vPPh$_3$ reported in this work with other heterogeneous catalysts.

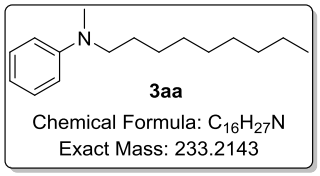
Alkene/Amine (M/N)	Cat.	CO/H$_2$ (X/Y MPa)	T (°C)	Conv. (%)	Yield (%)	Amine (l/b)	Ref.
1-octene/*N*-methylaniline (1/1)	Rh@CPOL-DPMphos&*p*-3vPPh$_3$	1/1	120	96	95	85/15	This work
1-octene/di-*n*-propylamine (1/1.5)	$Ph_2P(CH_2CH_2O)_nCH_3$ (n=16)-stabilized Rh nanoparticle catalyst	3/3	120	99	96	79/21	3
1-hexene/*N*-methylaniline (1/8.8)	Au/Co_3O_4	2/2	120	100	39	41/59	4
1-hexene/morpholine (1/1)	Rh-HMS-F	1.35/5.4	120	100	73	64/36	5
1-hexene/pyrrolidine (1/1)	Rh-ETS-10	1.35/5.4	100	100	81	56/44	6

Table S8. Rh contents in the catalysts and the filtration after each cycle, and the corresponding reaction performance. [a]

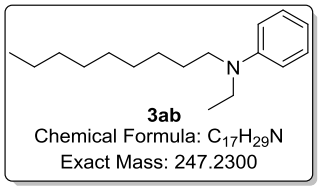
Entry	Recycling	Rh contents in the catalysts (wt.%)	Rh contents in the filtration (ppm)	Conv. (%) [b]	Yield (%) [b]	Amine (l/b) [b]
1	The 1st run	0.434	<0.1 ppm	90	80	85/15
2	The 2nd run	0.426	<0.1 ppm	89	76	84/16
3	The 3rd run	0.432	<0.1 ppm	86	77	82/18

[a] Rh content was determined by ICP-AES. [b] Reaction conditions: 1-octene (1 mmol), *N*-methylaniline (1 mmol), Rh dosage (1 μmol), TsOH·H$_2$O (0.08 mmol), total pressure (2 MPa) of syngas (CO : H$_2$ = 1 : 1), methanol (2 ml), 120 °C, 12 h. Conversion, yield, and ratio of *n*-/*iso*-amines were determined by GC analysis using *n*-decane as the internal standard.

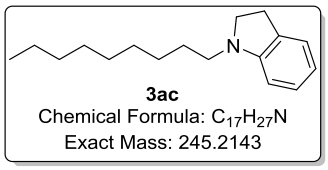
6. NMR data of the products



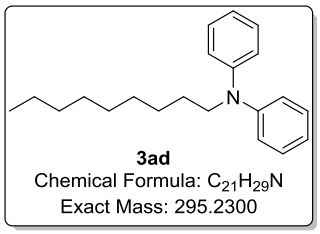
***N*-Methyl-*N*-nonylaniline:** yellow oil liquid, 210 mg, 90% yield, 85% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 – 7.00 (m, 2H), 6.99 – 6.40 (m, 3H), 3.29 (t, *J* = 7.5 Hz, 2H), 2.91 (s, 3H), 1.56 (d, *J* = 6.3 Hz, 2H), 1.28 (d, *J* = 12.9 Hz, 12H), 0.88 (t, *J* = 5.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.47, 129.25, 115.87, 112.16, 52.95, 38.39, 32.02, 29.77, 29.71, 29.43, 27.34, 26.76, 22.82, 14.26.



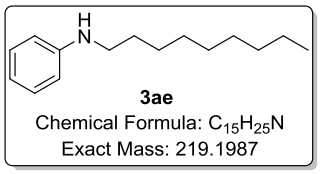
***N*-Ethyl-*N*-nonylaniline:** colorless oil liquid, 146 mg, 60% yield, 93% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.21 (dd, *J* = 15.8, 7.8 Hz, 2H), 6.63 (dd, *J* = 16.8, 7.8 Hz, 3H), 3.35 (q, *J* = 7.0 Hz, 2H), 3.29 – 3.16 (m, 2H), 1.57 (s, 2H), 1.29 (d, *J* = 14.8 Hz, 12H), 1.14 (t, *J* = 7.0 Hz, 3H), 0.88 (t, *J* = 6.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.13, 129.35, 115.29, 111.85, 50.59, 45.01, 32.03, 29.79, 29.72, 29.44, 27.65, 27.37, 22.82, 14.26, 12.44.



1-Nonylindoline: colorless oil liquid, 208 mg, 83% yield, 82% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.10 – 6.96 (m, 2H), 6.61 (t, *J* = 7.2 Hz, 1H), 6.52 – 6.35 (m, 1H), 3.31 (t, *J* = 8.3 Hz, 2H), 3.08 – 2.80 (m, 4H), 1.67 – 1.51 (m, 2H), 1.29 (t, *J* = 19.2 Hz, 12H), 0.88 (t, *J* = 6.0 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.49, 152.89, 130.09, 129.69, 128.68, 128.56, 127.86, 127.37, 124.43, 124.39, 121.37, 121.02, 120.99, 119.23, 119.20, 117.31, 116.98, 109.68, 109.47, 106.94, 106.46, 100.92, 100.81, 56.99, 54.17, 53.16, 49.43, 46.51, 34.95, 34.67, 34.39, 32.64, 32.03, 31.96, 30.38, 29.81, 29.75, 29.68, 29.63, 29.60, 29.45, 29.39, 29.37, 28.74, 28.69, 27.48, 27.42, 27.17, 27.14, 26.99, 22.82, 22.78, 18.14, 17.90, 14.25, 14.23.

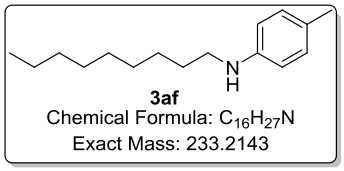


***N*-Nonyl-*N*-phenylaniline:** colorless oil liquid, 161 mg, 55% yield, 96% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.25 (t, *J* = 7.8 Hz, 4H), 6.98 (d, *J* = 7.9 Hz, 4H), 6.92 (t, *J* = 7.2 Hz, 2H), 3.76 – 3.57 (m, 2H), 1.64 (d, *J* = 6.8 Hz, 2H), 1.27 (d, *J* = 16.5 Hz, 12H), 0.87 (t, *J* = 6.5 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.24, 129.35, 121.13, 121.01, 52.49, 32.00, 29.74, 29.59, 29.39, 27.57, 27.23, 22.80, 14.25.



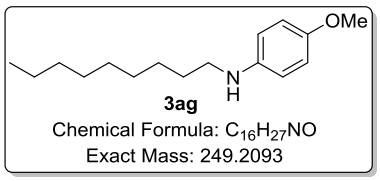
3ae
Chemical Formula: $C_{15}H_{25}N$
Exact Mass: 219.1987

***N*-Nonylaniline:** yellow oil liquid, 120 mg, 55% yield, 75% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.16 (t, *J* = 7.7 Hz, 2H), 6.67 (dd, *J* = 8.7, 5.0 Hz, 1H), 6.58 (d, *J* = 7.8 Hz, 2H), 3.59 (s, 0.89H), 3.08 (t, *J* = 7.1 Hz, 1.63H), 2.95 – 2.72 (m, 0.33H), 1.80 – 1.65 (m, 0.32H), 1.59 (dt, *J* = 14.3, 7.0 Hz, 1.60H), 1.46 – 1.18 (m, 12H), 0.95 (d, *J* = 6.6 Hz, 0.77H), 0.88 (t, *J* = 6.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.66, 129.31, 117.15, 117.01, 112.78, 112.72, 50.44, 44.10, 34.93, 33.04, 32.01, 29.75, 29.70, 29.60, 29.41, 27.31, 27.08, 22.80, 18.18, 14.24.



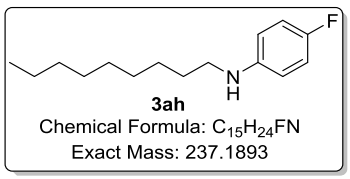
3af
Chemical Formula: $C_{16}H_{27}N$
Exact Mass: 233.2143

4-Methyl-*N*-nonylaniline: yellow oil liquid, 166 mg, 71% yield, 78% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.96 (d, *J* = 7.9 Hz, 2H), 6.50 (d, *J* = 8.1 Hz, 2H), 3.39 (s, 1H), 3.04 (t, *J* = 7.1 Hz, 1.74H), 2.82 (dd, *J* = 12.1, 7.4 Hz, 0.26H), 2.22 (s, 3H), 1.74 – 1.64 (m, 0.27H), 1.55 (dd, *J* = 14.0, 7.0 Hz, 1.74H), 1.27 (s, 12H), 0.93 (d, *J* = 6.6 Hz, 0.68H), 0.88 (t, *J* = 6.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.40, 129.75, 126.21, 126.05, 112.94, 112.86, 50.79, 44.44, 34.92, 32.99, 32.00, 29.73, 29.69, 29.60, 29.40, 27.31, 27.06, 22.78, 20.44, 18.12, 14.20.



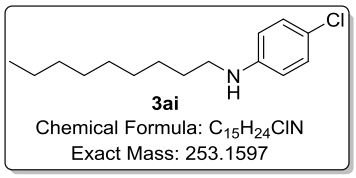
3ag
Chemical Formula: $C_{16}H_{27}NO$
Exact Mass: 249.2093

4-Methoxy-*N*-nonylaniline: yellow oil liquid, 156 mg, 63% yield, 78% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.76 (d, *J* = 8.7 Hz, 2H), 6.55 (d, *J* = 8.7 Hz, 2H), 3.72 (s, 3H), 3.29 (s, 1H), 3.03 (t, *J* = 7.1 Hz, 1.68H), 2.81 (dd, *J* = 11.9, 7.3 Hz, 0.29H), 1.74 – 1.63 (m, 0.26H), 1.57 (dt, *J* = 14.3, 7.0 Hz, 1.68H), 1.39 – 1.19 (m, 12H), 0.94 (d, *J* = 6.6 Hz, 0.55H), 0.88 (t, *J* = 6.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 151.98, 142.96, 114.92, 114.02, 55.83, 55.81, 51.43, 45.07, 34.91, 33.02, 31.97, 29.77, 29.71, 29.66, 29.58, 29.37, 27.30, 27.04, 22.76, 18.14, 14.18.



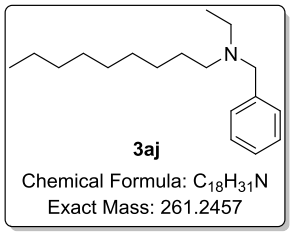
3ah
Chemical Formula: $C_{15}H_{24}FN$
Exact Mass: 237.1893

4-Fluoro-*N*-nonylaniline: yellow oil liquid, 182 mg, 77% yield, 80% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.87 (t, *J* = 8.7 Hz, 2H), 6.51 (dd, *J* = 8.8, 4.4 Hz, 2H), 3.45 (s, 0.78H), 3.28 – 3.12 (m, 0.18H), 3.04 (t, *J* = 7.1 Hz, 1.56H), 2.97 (dd, *J* = 12.0, 6.1 Hz, 0.22H), 2.88 – 2.79 (m, 0.26H), 1.70 (td, *J* = 12.8, 6.6 Hz, 0.22H), 1.64 – 1.51 (m, 1.72H), 1.27 (s, 12H), 0.95 (d, *J* = 6.6 Hz, 0.62H), 0.88 (t, *J* = 6.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.95, 156.85, 154.62, 154.53, 145.15, 145.06, 145.05, 115.81, 115.59, 113.58, 113.50, 113.43, 51.17, 44.82, 34.92, 33.04, 32.02, 29.74, 29.69, 29.60, 29.41, 27.30, 27.07, 22.81, 18.17, 14.24.



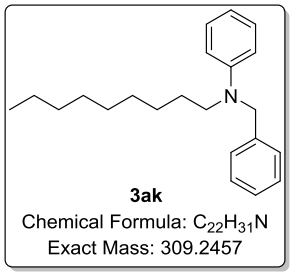
3ai
Chemical Formula: $C_{15}H_{24}ClN$
Exact Mass: 253.1597

4-Chloro-*N*-nonylaniline: yellow oil liquid, 160 mg, 63% yield, 81% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.08 (d, *J* = 8.6 Hz, 2H), 6.48 (d, *J* = 8.6 Hz, 2H), 3.59 (s, 0.86H), 3.03 (t, *J* = 7.1 Hz, 1.75H), 2.89 – 2.77 (m, 0.28H), 1.68 (dd, *J* = 12.4, 6.3 Hz, 0.28H), 1.56 (dd, *J* = 14.1, 7.1 Hz, 1.77H), 1.31 (d, *J* = 34.7 Hz, 12H), 0.94 (d, *J* = 6.6 Hz, 0.70H), 0.88 (t, *J* = 6.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 147.18, 129.08, 113.76, 50.52, 44.19, 34.86, 32.96, 32.00, 31.98, 29.71, 29.67, 29.56, 29.54, 29.39, 27.25, 27.04, 22.79, 18.10, 14.23.



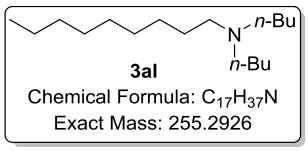
3aj
Chemical Formula: $C_{18}H_{31}N$
Exact Mass: 261.2457

***N*-Benzyl-*N*-ethylnonan-1-amine:** yellow oil liquid, 170 mg, 65% yield, 85% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.46 – 7.01 (m, 5H), 3.55 (s, 2H), 2.50 (dd, *J* = 14.1, 7.0 Hz, 2H), 2.45 – 2.33 (m, 2H), 1.45 (d, *J* = 6.4 Hz, 2H), 1.25 (s, 12H), 1.03 (t, *J* = 7.1 Hz, 3H), 0.88 (t, *J* = 6.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.27, 129.00, 128.21, 126.74, 58.16, 53.38, 47.37, 32.05, 29.75, 29.46, 27.65, 27.12, 22.84, 14.28, 11.86.



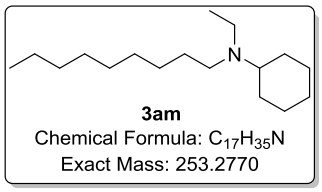
3ak
Chemical Formula: $C_{22}H_{31}N$
Exact Mass: 309.2457

***N*-Benzyl-*N*-nonylaniline:** colorless oil liquid, 222 mg, 72% yield, 81% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.26 (q, *J* = 7.4 Hz, 2H), 7.22 – 7.10 (m, 5H), 6.64 (t, *J* = 9.1 Hz, 3H), 4.51 (s, 2H), 3.35 (dd, *J* = 15.7, 8.0 Hz, 1.81H), 3.28 – 3.07 (m, 0.34H), 1.64 (s, 1.95H), 1.28 (d, *J* = 14.6 Hz, 12H), 0.92 (d, *J* = 6.6 Hz, 0.47H), 0.88 (t, *J* = 6.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.89, 148.70, 139.26, 138.94, 129.28, 129.18, 128.63, 128.60, 126.79, 126.61, 116.00, 112.49, 112.16, 58.70, 55.43, 54.56, 51.44, 34.91, 32.35, 32.01, 29.80, 29.75, 29.65, 29.40, 27.30, 27.23, 27.18, 22.80, 18.00, 14.25.



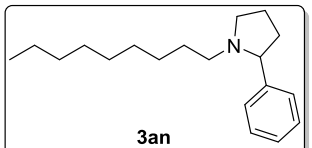
3al
Chemical Formula: $C_{17}H_{37}N$
Exact Mass: 255.2926

***N*,*N*-Dibutylnonan-1-amine:** yellow oil liquid, 196 mg, 77% yield, 85% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.38 (dd, *J* = 13.0, 5.6 Hz, 6H), 1.48 – 1.36 (m, 6H), 1.30 (dd, *J* = 16.9, 9.6 Hz, 16H), 0.98 – 0.80 (m, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 54.39, 54.08, 32.05, 29.81, 29.78, 29.46, 29.36, 27.82, 27.18, 22.83, 20.96, 14.26.



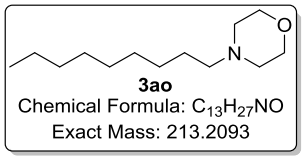
3am
Chemical Formula: $C_{17}H_{35}N$
Exact Mass: 253.2770

***N*-Ethyl-*N*-nonylcyclohexanamine:** colorless oil liquid, 152 mg, 60% yield, 87% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.53 (q, *J* = 7.1 Hz, 2H), 2.45 – 2.36 (m, 2H), 1.77 (d, *J* = 9.8 Hz, 4H), 1.62 (d, *J* = 11.9 Hz, 1H), 1.42 (dd, *J* = 14.1, 7.4 Hz, 2H), 1.31 – 1.16 (m, 18H), 1.02 (t, *J* = 7.1 Hz, 3H), 0.88 (t, *J* = 6.6 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 60.00, 50.35, 44.40, 32.05, 29.81, 29.79, 29.46, 29.35, 27.84, 26.64, 26.40, 22.82, 14.26.



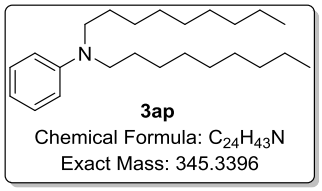
3an
Chemical Formula: $C_{19}H_{31}N$
Exact Mass: 273.2457

1-Nonyl-2-phenylpyrrolidine: colorless oil liquid, 232 mg, 85% yield, 83% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37 – 7.27 (m, 4H), 7.27 – 7.19 (m, 1H), 3.34 (td, *J* = 8.7, 2.3 Hz, 1H), 3.23 – 3.10 (m, 1H), 2.51 (dt, *J* = 11.7, 8.2 Hz, 1H), 2.22 – 2.07 (m, 2H), 1.93 (ddt, *J* = 13.1, 8.5, 5.8 Hz, 2H), 1.80 (dddd, *J* = 12.2, 9.1, 6.1, 2.8 Hz, 1H), 1.68 (dddd, *J* = 12.2, 10.8, 9.2, 6.2 Hz, 1H), 1.41 (dd, *J* = 13.7, 7.7 Hz, 2H), 1.30 – 1.16 (m, 12H), 0.87 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.35, 139.00, 128.38, 127.61, 126.90, 70.59, 54.72, 53.80, 35.29, 32.03, 29.71, 29.70, 29.44, 28.93, 27.62, 22.81, 22.58, 14.26.



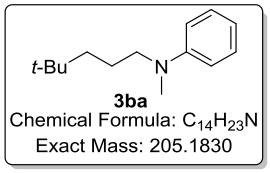
3ao
Chemical Formula: $C_{13}H_{27}NO$
Exact Mass: 213.2093

4-Nonylmorpholine: yellow oil liquid, 183 mg, 86% yield, 82% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.81 – 3.62 (m, 4H), 2.44 (s, 4H), 2.32 (dd, *J* = 8.7, 6.8 Hz, 2H), 1.57 – 1.40 (m, 2H), 1.37 – 1.18 (m, 12H), 0.88 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 66.96, 59.25, 53.81, 31.90, 29.59, 29.56, 29.30, 27.53, 26.56, 22.68, 14.11.



3ap
Chemical Formula: $C_{24}H_{43}N$
Exact Mass: 345.3396

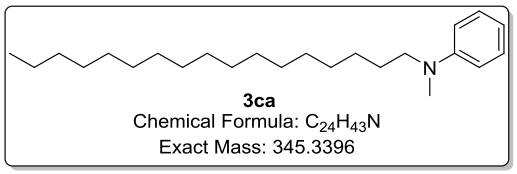
***N*,*N*-Dinonylaniline:** colorless oil liquid, 112 mg, 65% yield, 77% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.19 (t, *J* = 7.9 Hz, 2H), 6.61 (dd, *J* = 10.6, 8.0 Hz, 3H), 3.40 – 2.91 (m, 4H), 1.56 (s, 4H), 1.29 (d, *J* = 12.1 Hz, 24H), 0.87 (d, *J* = 6.9 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 148.48, 148.19, 129.23, 129.13, 115.06, 112.01, 111.66, 77.08, 58.18, 52.14, 51.11, 34.81, 31.97, 29.78, 29.73, 29.64, 29.38, 27.29, 27.28, 27.15, 26.43, 22.76, 17.83, 14.19.



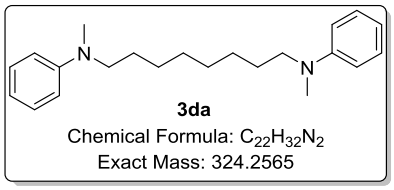
3ba
Chemical Formula: $C_{14}H_{23}N$
Exact Mass: 205.1830

***N*-(4,4-Dimethylpentyl)-*N*-methylaniline:** colorless oil liquid, 154 mg, 75% yield, >99% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.22 (dd, *J* = 8.3, 7.5 Hz, 2H), 6.67 (t, *J* = 9.0 Hz, 3H), 3.25 (t, *J* = 7.6 Hz, 2H), 2.91 (s, 3H), 1.63 –

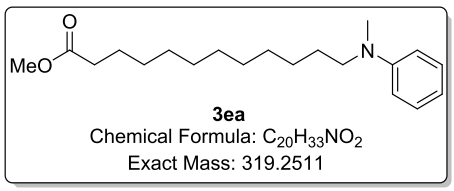
1.40 (m, 2H), 1.18 (dd, *J* = 10.8, 6.1 Hz, 2H), 0.88 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.47, 129.27, 115.89, 112.12, 53.72, 41.44, 38.31, 30.41, 29.53, 21.88.



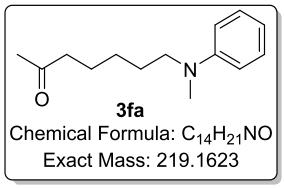
***N*-Heptadecyl-*N*-methylaniline:** white solid, 269 mg, 78% yield, 84% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.22 (t, *J* = 7.9 Hz, 2H), 6.67 (dd, *J* = 13.2, 7.4 Hz, 3H), 3.38 – 3.18 (m, 2H), 2.91 (s, 3H), 1.55 (d, *J* = 6.0 Hz, 2H), 1.28 (d, *J* = 16.7 Hz, 28H), 0.88 (t, *J* = 6.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.47, 129.26, 129.19, 115.88, 112.17, 111.87, 60.02, 52.97, 39.63, 38.41, 34.86, 32.37, 32.09, 30.17, 29.86, 29.82, 29.78, 29.72, 29.53, 27.35, 27.22, 26.77, 22.86, 17.93, 14.29.



N1,N8-Dimethyl-N1,N8-diphenyloctane-1,8-diamine: colorless oil liquid, 110 mg, 68% yield, 72% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.21 (t, *J* = 7.8 Hz, 4H), 6.66 (t, *J* = 8.6 Hz, 6H), 3.31 – 3.23 (m, 3.33H), 3.21 – 3.13 (m, 0.44H), 3.07 – 2.96 (m, 0.42H), 2.91 (s, 1H), 2.89 (s, 5H), 1.88 (s, 0.42H), 1.54 (s, 3.71H), 1.39 (d, *J* = 7.9 Hz, 0.69H), 1.30 (s, 6.84H), 1.16 – 1.00 (m, 0.39H), 0.88 (d, *J* = 6.5 Hz, 1.3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.71, 149.40, 129.22, 129.16, 115.87, 115.67, 112.13, 111.85, 59.91, 52.85, 39.58, 38.37, 34.72, 32.26, 29.65, 27.60, 27.22, 27.09, 26.73, 17.86.

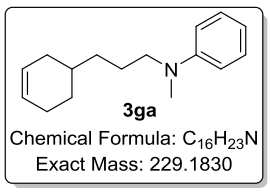


Methyl 12-(methyl(phenyl)amino)dodecanoate: yellow oil liquid, 249 mg, 78% yield, 81% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.20 (t, *J* = 7.9 Hz, 2H), 6.66 (dd, *J* = 13.4, 7.5 Hz, 3H), 3.65 (s, 3H), 3.34 – 3.23 (m, 1.64H), 3.18 (dt, *J* = 16.6, 8.3 Hz, 0.21H), 3.09 – 2.95 (m, 0.20H), 2.93 (s, 0.49H), 2.90 (s, 2.46H), 2.29 (t, *J* = 7.5 Hz, 2H), 1.89 (s, 0.14H), 1.66 – 1.49 (m, 3.81H), 1.27 (d, *J* = 2.4 Hz, 14H), 1.08 (dd, *J* = 17.3, 9.2 Hz, 0.26H), 0.88 (d, *J* = 6.6 Hz, 0.59H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 174.30, 149.69, 149.36, 129.14, 129.07, 115.79, 115.56, 112.07, 111.78, 59.89, 52.83, 51.45, 39.50, 38.29, 34.71, 34.12, 32.25, 29.96, 29.66, 29.59, 29.57, 29.47, 29.29, 29.18, 27.23, 27.07, 26.68, 24.99, 17.81.

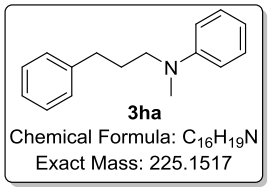


7-(Methyl(phenyl)amino)heptan-2-one: colorless oil liquid, 110 mg, 50% yield, 89% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.21 (t, *J* = 7.8 Hz, 2H), 6.66 (t, *J* = 7.6 Hz, 3H), 3.36 – 3.22 (m, 2H), 2.94 (s, 0.33H), 2.90 (s, 2.70H), 2.41 (t, *J* = 7.3 Hz, 2H), 2.11 (s, 3H), 1.66 – 1.48 (m, 3.79H), 1.36 – 1.20 (m, 2.74H), 0.89 (d, *J* = 6.6 Hz,

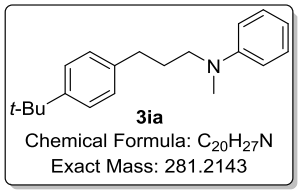
0.51H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 209.01, 149.28, 129.17, 129.13, 115.91, 112.10, 111.91, 59.68, 52.57, 43.62, 41.42, 39.58, 38.32, 31.84, 29.96, 29.90, 29.74, 28.40, 26.72, 26.54, 23.66, 17.54.



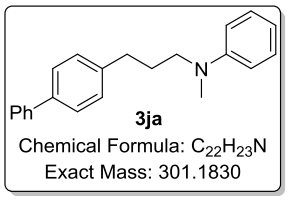
***N*-(3-(Cyclohex-3-en-1-yl)propyl)-*N*-methylaniline:** yellow oil liquid, 160 mg, 70% yield, 95% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.14 (ddd, *J* = 7.7, 5.8, 2.3 Hz, 2H), 6.76 – 6.45 (m, 3H), 5.58 (s, 2H), 3.35 – 3.06 (m, 2H), 2.84 (s, 3H), 2.10 – 1.84 (m, 3H), 1.77 – 1.36 (m, 5H), 1.34 – 1.05 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.47, 129.26, 127.16, 126.63, 115.96, 112.21, 53.20, 38.39, 34.17, 33.63, 32.06, 29.09, 25.37, 24.10.



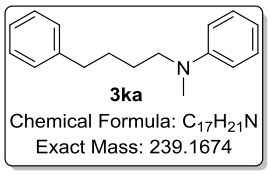
***N*-Methyl-*N*-(3-phenylpropyl)aniline:** colorless oil liquid, 186 mg, 82% yield, 40% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 – 7.13 (m, 7H), 6.67 (t, *J* = 9.7 Hz, 3H), 3.48 (dd, *J* = 14.6, 7.6 Hz, 0.63H), 3.41 – 3.28 (m, 1.45H), 3.20 (dt, *J* = 14.2, 7.1 Hz, 0.68H), 2.90 (s, 1.21H), 2.73 (s, 1.89H), 2.64 (t, *J* = 7.7 Hz, 0.81H), 1.97 – 1.79 (m, 0.79H), 1.29 (d, *J* = 6.9 Hz, 2.12H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.39, 149.08, 145.25, 141.90, 129.27, 128.57, 128.49, 128.45, 127.40, 126.48, 125.99, 115.84, 112.29, 111.83, 61.09, 52.34, 39.64, 38.40, 38.38, 33.44, 28.27, 18.92.



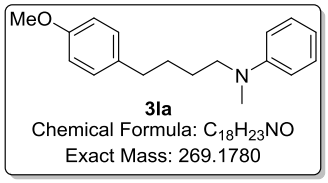
***N*-(3-(4-(Tert-butyl)phenyl)propyl)-*N*-methylaniline:** colorless oil liquid, 232 mg, 83% yield, 43% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30 (t, *J* = 7.0 Hz, 2H), 7.25 – 7.16 (m, 2H), 7.12 (dd, *J* = 10.2, 8.4 Hz, 2H), 6.65 (t, *J* = 7.1 Hz, 3H), 3.46 – 3.36 (m, 1.10H), 3.32 (dd, *J* = 15.6, 8.2 Hz, 0.90H), 3.16 (dd, *J* = 14.3, 7.1 Hz, 0.66H), 2.88 (s, 1.31H), 2.76 (s, 1.70H), 2.60 (t, *J* = 7.7 Hz, 0.85H), 1.94 – 1.77 (m, 0.87H), 1.30 (s, 9H), 1.27 (d, *J* = 7.0 Hz, 2.13H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.40, 149.23, 149.14, 148.72, 142.06, 138.80, 129.24, 128.10, 126.96, 125.39, 125.34, 116.09, 115.80, 112.29, 111.85, 61.06, 52.40, 39.62, 38.39, 37.86, 34.48, 34.45, 32.86, 31.54, 28.26, 18.95.



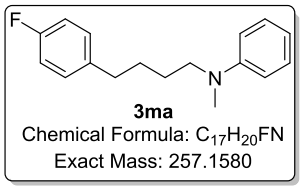
***N*-(3-([1,1'-Biphenyl]-4-yl)propyl)-*N*-methylaniline:** colorless oil liquid, 186 mg, 62% yield, 45% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.6 Hz, 2H), 7.52 (t, *J* = 7.5 Hz, 2H), 7.42 (t, *J* = 7.5 Hz, 2H), 7.36 – 7.18 (m, 5H), 6.69 (t, *J* = 6.9 Hz, 3H), 3.52 (dd, *J* = 14.6, 7.7 Hz, 0.55H), 3.47 – 3.32 (m, 1.39H), 3.30 – 3.18 (m, 0.69H), 2.92 (s, 1.39H), 2.78 (s, 0.64H), 2.68 (t, *J* = 7.7 Hz, 0.87H), 2.01 – 1.87 (m, 0.86H), 1.33 (d, *J* = 6.9 Hz, 1.72H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.42, 149.10, 144.38, 141.15, 141.07, 141.05, 139.40, 138.96, 129.31, 128.89, 128.87, 127.83, 127.28, 127.23, 127.18, 127.12, 116.16, 115.91, 112.33, 111.90, 61.10, 52.37, 39.75, 38.45, 38.08, 33.08, 28.29, 18.96.



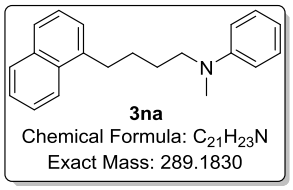
***N*-Methyl-*N*-(4-phenylbutyl)aniline:** colorless oil liquid, 200 mg, 84% yield, 80% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.18 (ddt, *J* = 22.6, 15.7, 7.7 Hz, 7H), 6.63 (dd, *J* = 19.3, 8.5 Hz, 3H), 3.26 (dd, *J* = 14.5, 8.2 Hz, 1.81H), 3.12 – 2.97 (m, 0.27H), 2.90 (s, 0.74H), 2.84 (s, 2.34H), 2.77 – 2.66 (m, 0.26H), 2.60 (t, *J* = 6.9 Hz, 1.66H), 2.32 (dd, *J* = 13.1, 8.8 Hz, 0.20H), 2.19 (dt, *J* = 13.1, 6.6 Hz, 0.19H), 1.71 – 1.50 (m, 3.23H), 0.85 (d, *J* = 6.6 Hz, 0.64H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.34, 142.35, 129.21, 128.44, 128.38, 125.83, 115.95, 112.14, 59.38, 52.66, 41.39, 39.53, 38.34, 35.88, 34.57, 29.09, 26.48, 17.87.



***N*-(4-(4-Methoxyphenyl)butyl)-*N*-methylaniline:** colorless oil liquid, 202 mg, 75% yield, 80% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.20 (dd, *J* = 14.4, 6.9 Hz, 2H), 7.06 (t, *J* = 8.1 Hz, 2H), 6.81 (d, *J* = 8.4 Hz, 2H), 6.64 (dd, *J* = 21.1, 7.9 Hz, 3H), 3.76 (s, 3H), 3.30 (s, 1.81H), 3.05 (dd, *J* = 14.5, 8.2 Hz, 0.25H), 2.94 (s, 0.66H), 2.88 (s, 2.36H), 2.73 – 2.63 (m, 0.29H), 2.57 (s, 1.58H), 2.30 (dd, *J* = 13.3, 8.7 Hz, 0.19H), 2.22 – 2.08 (m, 0.19H), 1.60 (s, 2.32H), 0.87 (d, *J* = 6.6 Hz, 0.67H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.89, 157.81, 149.68, 149.40, 134.51, 132.88, 130.06, 129.35, 129.25, 129.17, 115.95, 115.80, 113.81, 113.71, 112.17, 111.96, 59.40, 55.33, 52.75, 40.52, 39.60, 38.41, 34.99, 34.72, 29.38, 26.46, 17.91.

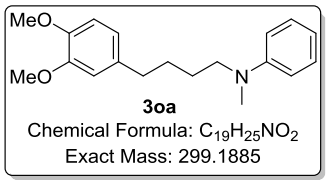


***N*-(4-(4-Fluorophenyl)butyl)-*N*-methylaniline:** colorless oil liquid, 180 mg, 70% yield, 84% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27 – 7.15 (m, 2H), 7.07 (dt, *J* = 11.8, 6.1 Hz, 2H), 6.93 (t, *J* = 8.6 Hz, 2H), 6.72 – 6.58 (m, 3H), 3.30 (d, *J* = 6.7 Hz, 1.85H), 3.11 – 2.98 (m, 0.21H), 2.93 (s, 0.5H), 2.87 (s, 2.53H), 2.75 – 2.65 (m, 0.22H), 2.57 (d, *J* = 6.6 Hz, 1.68H), 2.30 (dd, *J* = 13.3, 8.9 Hz, 0.16H), 2.15 (dd, *J* = 13.6, 6.9 Hz, 0.16H), 1.66 – 1.51 (m, 3.37H), 0.85 (d, *J* = 6.6 Hz, 0.56H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.51, 160.09, 149.63, 149.37, 138.00, 137.97, 136.48, 136.45, 130.49, 130.41, 129.80, 129.72, 129.26, 129.20, 116.03, 115.21, 115.00, 112.19, 111.99, 59.37, 52.67, 40.54, 39.59, 38.39, 35.07, 34.69, 29.23, 26.42, 17.75.



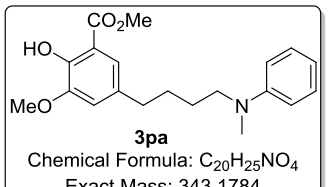
***N*-Methyl-*N*-(4-(naphthalen-1-yl)butyl)aniline:** yellow oil liquid, 243 mg, 84% yield, 82% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.1 Hz, 1H), 7.86 – 7.75 (m, 1H), 7.67 (d, *J* = 8.1 Hz, 1H), 7.49 – 7.38 (m, 2H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.29 – 7.13 (m, 3H), 6.64 (dd, *J* = 16.5, 8.4 Hz, 3H), 3.32 – 3.24 (m, 2H), 3.18 (dd, *J* = 14.6, 7.4 Hz, 0.28H), 3.04 (t, *J* = 7.3 Hz, 1.79H), 2.95 (s, 0.61H), 2.84 (s, 2.65H), 2.63 (dd, *J* = 13.7, 9.2 Hz, 0.25H), 2.37 (dq, *J* =

13.6, 6.8 Hz, 0.19H), 1.81 – 1.57 (m, 3.65H), 0.86 (d, *J* = 6.6 Hz, 0.66H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.63, 149.39, 138.46, 136.98, 134.03, 133.97, 132.06, 131.89, 129.26, 129.19, 128.88, 128.80, 127.26, 126.84, 126.68, 126.03, 125.82, 125.78, 125.62, 125.50, 125.47, 125.32, 124.03, 123.86, 116.02, 112.22, 112.16, 59.94, 52.69, 39.82, 38.46, 38.37, 33.67, 33.05, 28.41, 26.99, 18.19.



3oa
Chemical Formula: $C_{19}H_{25}NO_2$
Exact Mass: 299.1885

***N*-(4-(3,4-Dimethoxyphenyl)butyl)-*N*-methylaniline:** colorless oil liquid, 235 mg, 79% yield, 80% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.21 (t, *J* = 7.8 Hz, 2H), 6.77 (d, *J* = 7.9 Hz, 1H), 6.73 – 6.58 (m, 5H), 3.97 – 3.66 (m, 6H), 3.30 (t, *J* = 11.5 Hz, 1.81H), 3.05 (dd, *J* = 14.6, 8.0 Hz, 0.24H), 2.95 (s, 0.58H), 2.89 (s, 2.39H), 2.66 (dd, *J* = 12.3, 7.0 Hz, 0.29H), 2.57 (d, *J* = 6.4 Hz, 1.58H), 2.34 (dd, *J* = 13.3, 8.4 Hz, 0.18H), 2.24 – 2.12 (m, 0.18H), 1.61 (d, *J* = 3.2 Hz, 3.33H), 0.89 (d, *J* = 6.5 Hz, 0.59H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.58, 149.33, 148.79, 148.72, 147.21, 147.12, 135.02, 133.34, 129.19, 129.12, 121.05, 120.17, 115.91, 115.77, 112.18, 112.11, 111.92, 111.66, 111.17, 111.01, 59.28, 55.93, 55.82, 52.66, 40.99, 39.63, 38.36, 35.45, 34.49, 29.28, 26.44, 18.04.



3pa
Chemical Formula: $C_{20}H_{25}NO_4$
Exact Mass: 343.1784

Methyl 2-hydroxy-3-methoxy-5-(4-(methyl(phenyl)amino)butyl)benzoate: colorless viscous liquid, 274 mg, 80% yield, 79% linear selectivity. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 10.87 (s, 0.19H), 10.85 (s, 0.72H), 7.26 – 7.15 (m, 3H), 6.84 (s, 1H), 6.73 – 6.57 (m, 3H), 3.92 (s, 3.07H), 3.87 (s, 2.26H), 3.84 (s, 0.77H), 3.32 (d, *J* = 6.5 Hz, 1.78H), 3.07 (dd, *J* = 14.6, 7.7 Hz, 0.26H), 2.94 (s, 0.65H), 2.89 (s, 2.35H), 2.74 – 2.62 (m, 0.26H), 2.56 (d, *J* = 6.6 Hz, 1.57H), 2.33 (dd, *J* = 13.5, 8.4 Hz, 0.20H), 2.25 – 2.12 (m, 0.20H), 1.68 – 1.55 (m, 3.26H), 0.89 (d, *J* = 6.5 Hz, 0.73H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.87, 150.28, 150.20, 149.53, 149.29, 148.34, 148.29, 132.59, 130.97, 129.18, 129.12, 120.76, 119.99, 117.59, 117.10, 115.95, 115.88, 112.10, 111.95, 59.19, 56.18, 52.58, 52.33, 40.86, 39.63, 38.33, 35.36, 34.24, 29.06, 26.29, 17.90.

7. Catalyst characterization

FT-IR spectra

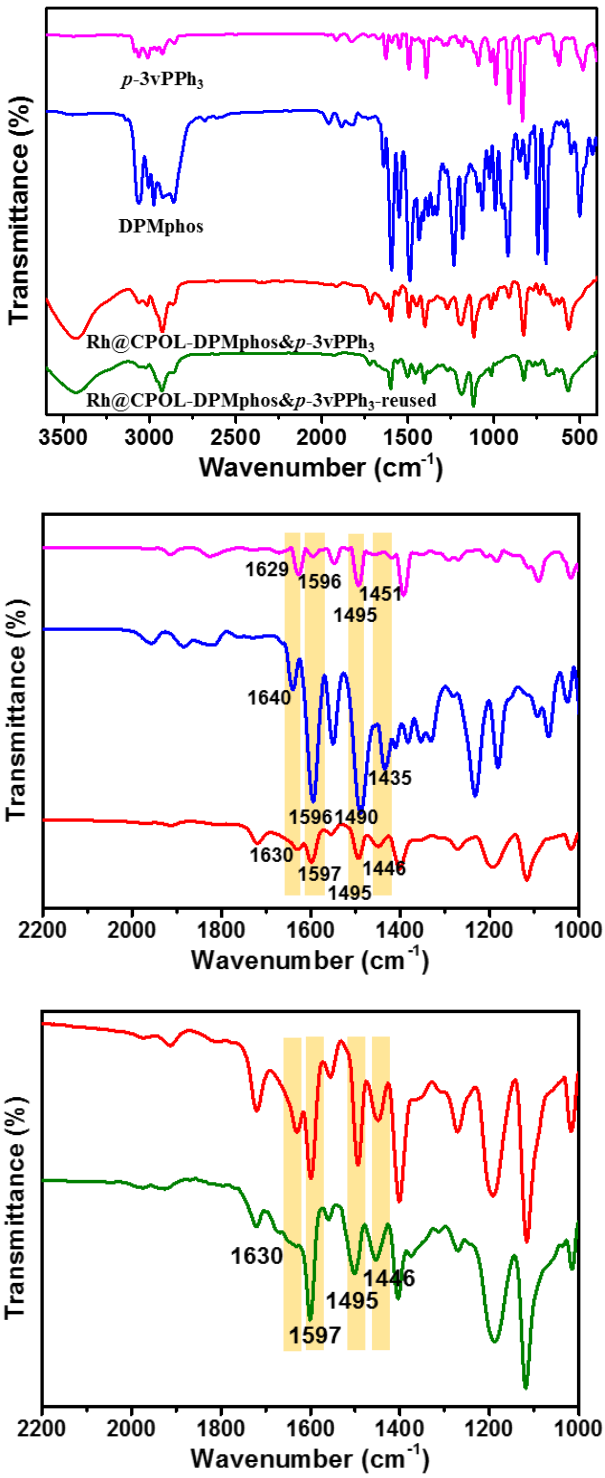


Figure S3. FT-IR spectra of *p*-3vPPh$_3$, DPMphos, Rh@CPOL-DPMphos&*p*-3vPPh$_3$ and Rh@CPOL-DPMphos&*p*-3vPPh$_3$-reused (magenta line for *p*-3vPPh$_3$, blue line for DPMphos, red line for Rh@CPOL-DPMphos&*p*-3vPPh$_3$, olive line for Rh@CPOL-DPMphos&*p*-3vPPh$_3$-reused).

N_2 adsorption-desorption analysis

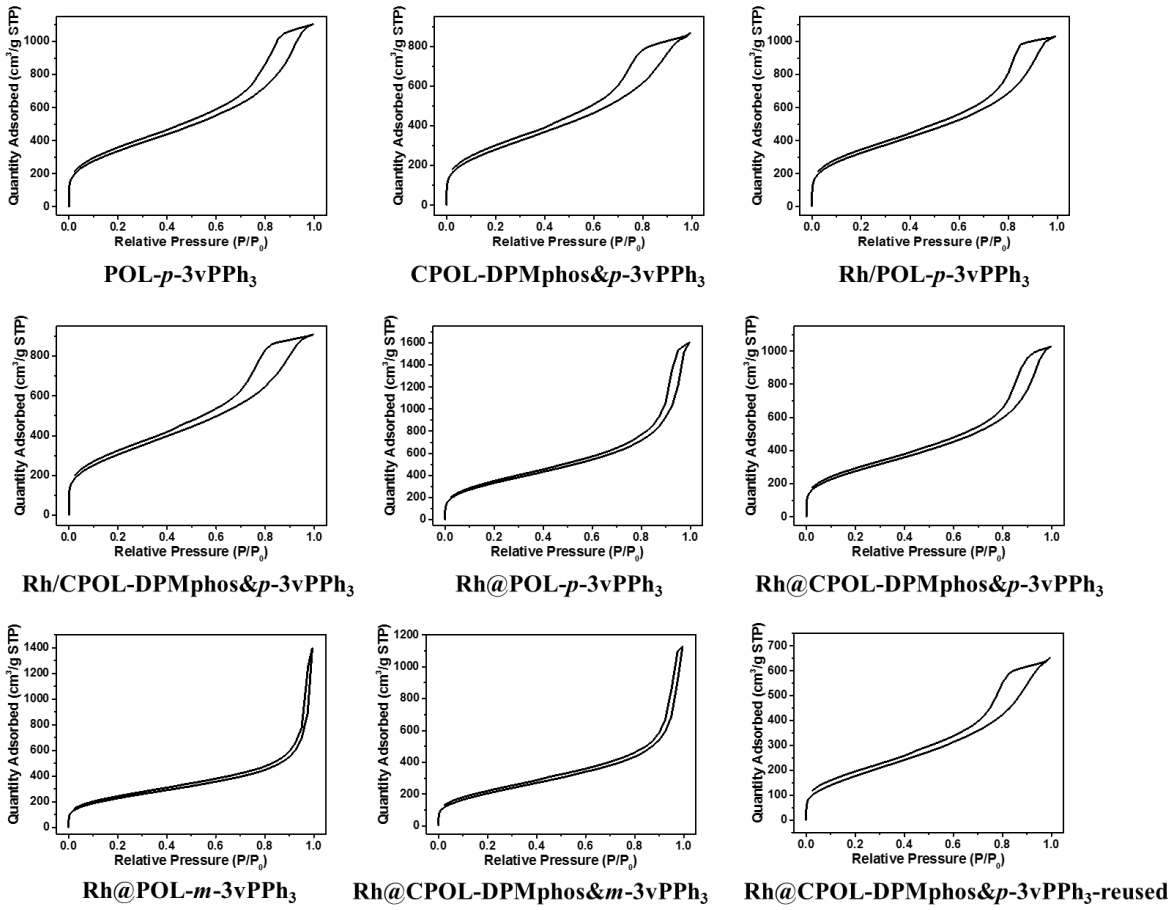


Figure S4. N_2 adsorption-desorption isotherm of the POPs catalysts.

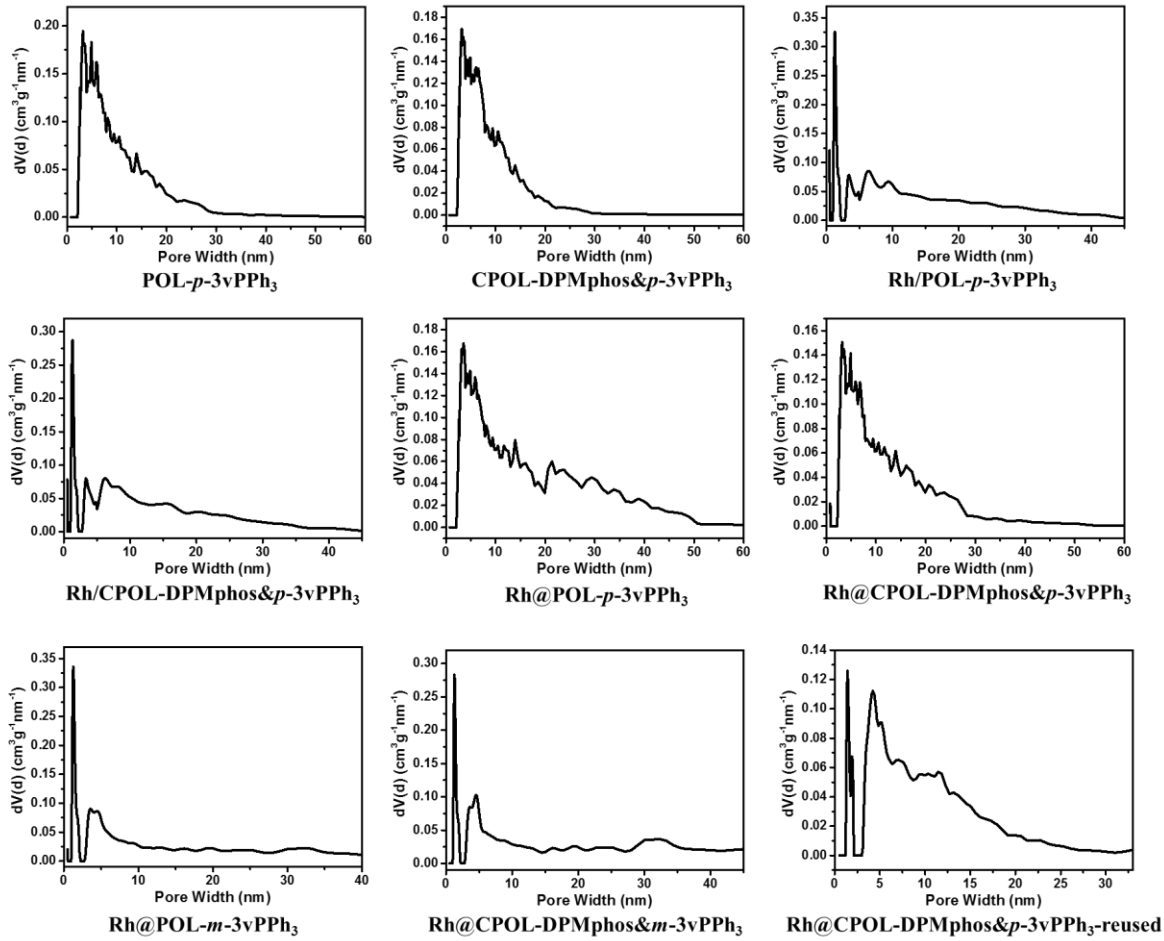


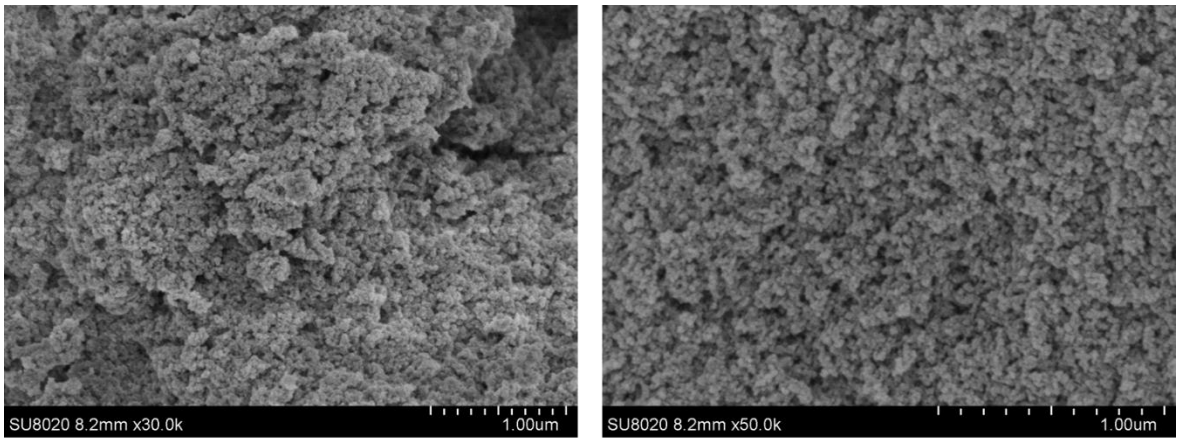
Figure S5. Pore size distribution of the POPs catalysts.

Table S9. The physical properties of the POPs catalysts.

Entry	Catalyst	SA (m^2g^{-1})[a]	APW (nm)[a]	PV ($cm^{-3}g^{-1}$)[a]
1	POL-*p*-3vPPh$_3$	1251.820	5.473	1.713
2	CPOL-DPMphos&*p*-3vPPh$_3$	1037.315	5.815	1.345
3	Rh/POL-*p*-3vPPh$_3$	1197.569	5.329	1.596
4	Rh/CPOL-DPMphos&*p*-3vPPh$_3$	1126.451	4.992	1.406
5	Rh@POL-*p*-3vPPh$_3$	1220.837	8.142	2.485
6	Rh@CPOL-DPMphos&*p*-3vPPh$_3$	1013.211	6.291	1.593
7	Rh@POL-*m*-3vPPh$_3$	835.971	1.034	2.160
8	Rh@CPOL-DPMphos&*m*-3vPPh$_3$	767.364	9.084	1.743
9	Rh@CPOL-DPMphos&*p*-3vPPh$_3$-reused	679.511	5.938	1.009

[a] Determined by an IQ_2 automated gas sorption analyzer. SA: BET surface area; APW: average pore width; PV: pore volume.

Scanning electron micrographs



CPOL-DPMphos&*p*-3vPPh$_3$ **Rh@CPOL-DPMphos&*p*-3vPPh$_3$**

Figure S6. SEM images of the CPOL-DPMphos&*p*-3vPPh$_3$ and Rh@CPOL-DPMphos&*p*-3vPPh$_3$.

TGA analysis

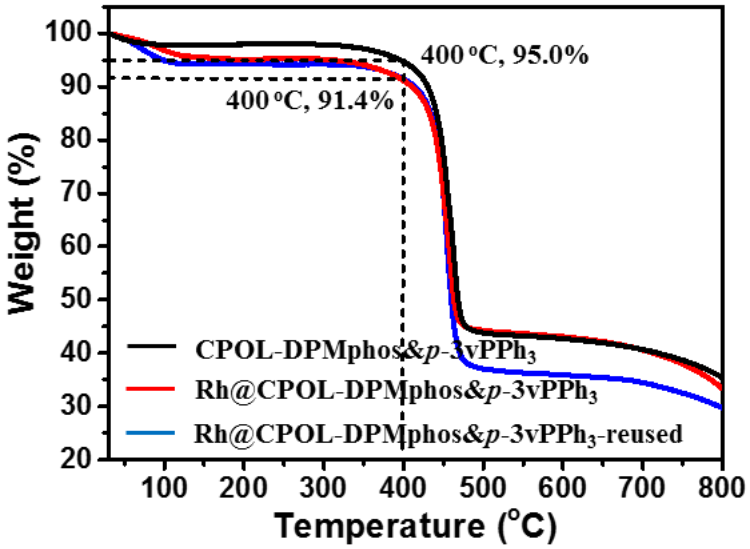


Figure S7. TGA analysis of the CPOL-DPMphos&*p*-3vPPh$_3$, Rh@CPOL-DPMphos&*p*-3vPPh$_3$, and Rh@CPOL-DPMphos&*p*-3vPPh$_3$-reused.

Experimental PXRD profiles

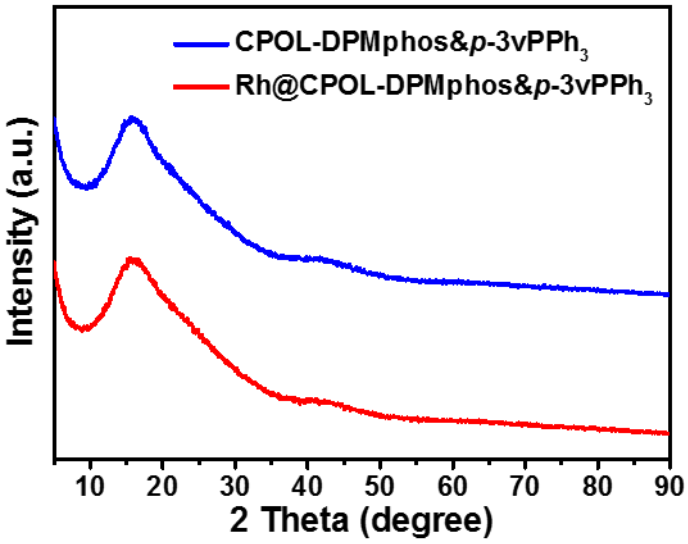


Figure S8. PXRD profiles of CPOL-DPMphos&*p*-3vPPh$_3$ and Rh@CPOL-DPMphos&*p*-3vPPh$_3$.

Transmission electron micrographs

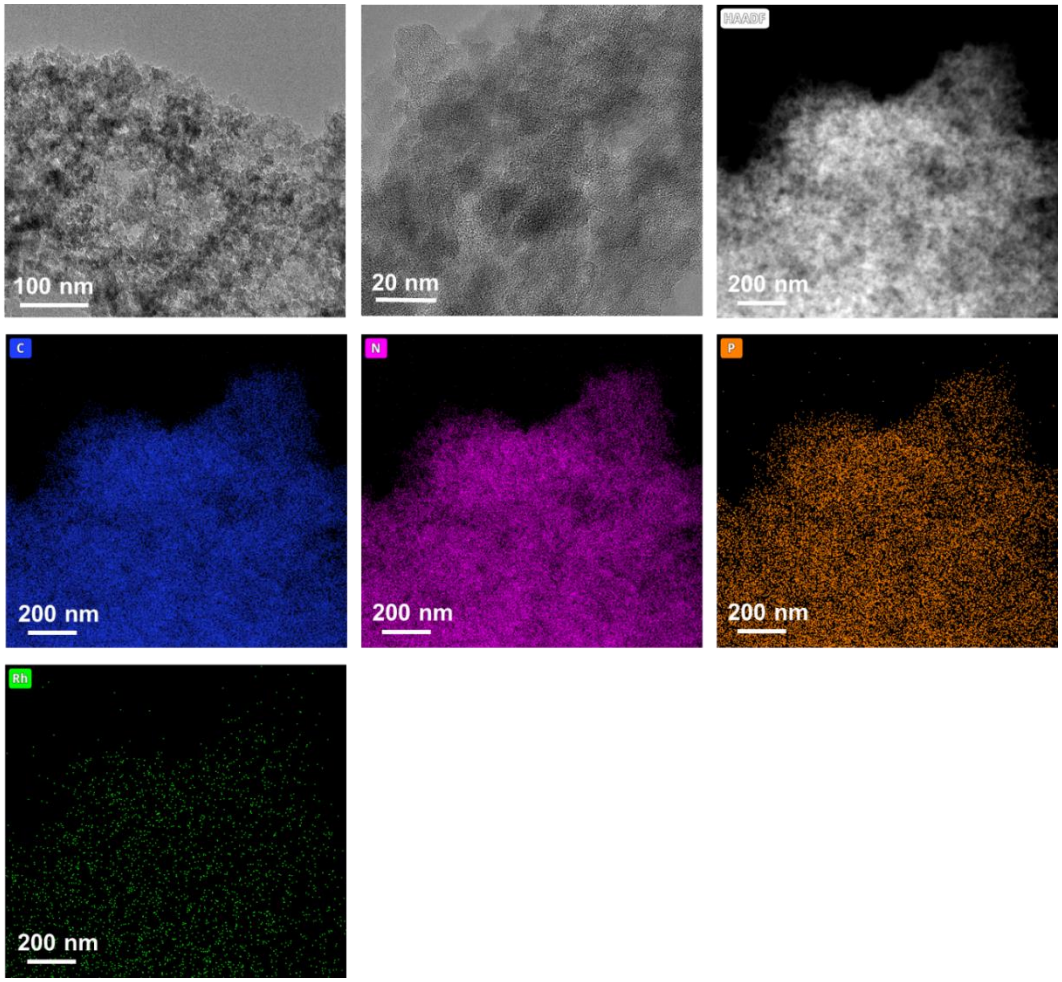


Figure S9. TEM images of the Rh@CPOL-DPMphos&*p*-3vPPh$_3$.

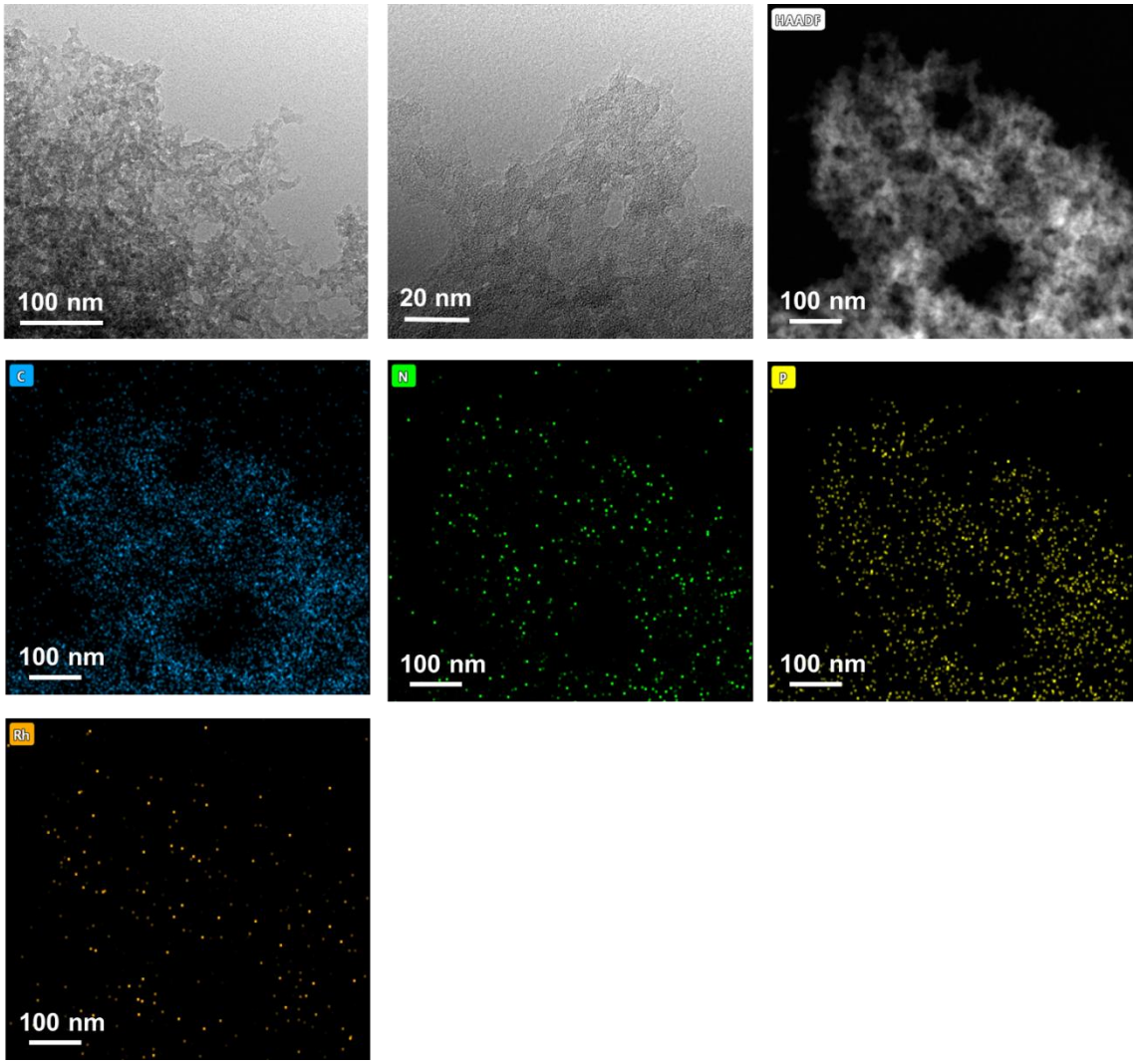


Figure S10. TEM images of the Rh@CPOL-DPMphos&*p*-3vPPh$_3$-reused.

X-ray photoelectron spectroscopy analysis

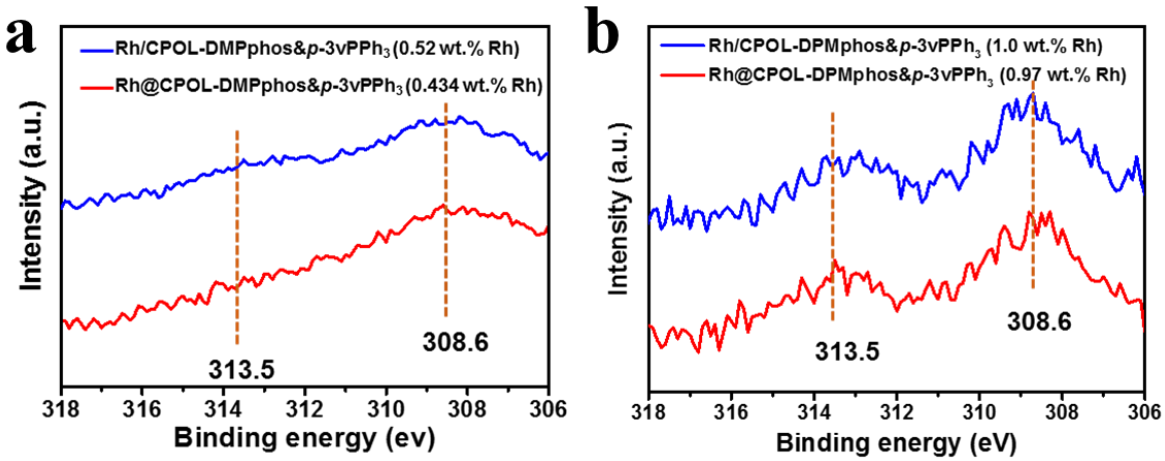


Figure S11. (a) Rh3d-XPS analysis of Rh/CPOL-DPMphos&*p*-3vPPh$_3$ (0.52 wt.% Rh) and Rh@CPOL-DPMphos&*p*-3vPPh$_3$ (0.434 wt.% Rh), (b) Rh3d-XPS analysis of Rh/CPOL-DPMphos&*p*-3vPPh$_3$ (1.0 wt.% Rh) and Rh@CPOL-DPMphos&*p*-3vPPh$_3$ (0.97 wt.% Rh).

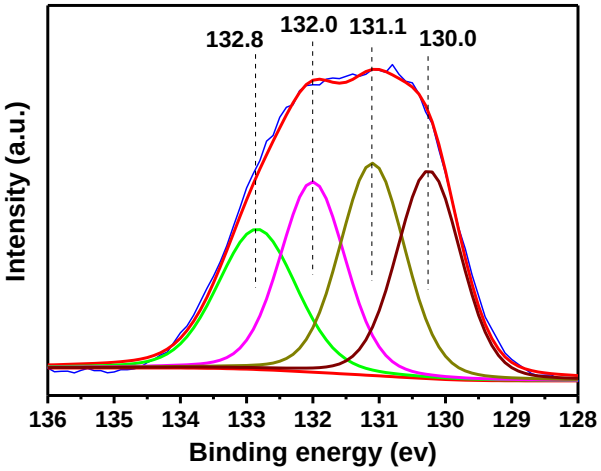


Figure S12. P2p-XPS analysis of CPOL-DPMphos&*p*-3vPPh$_3$.

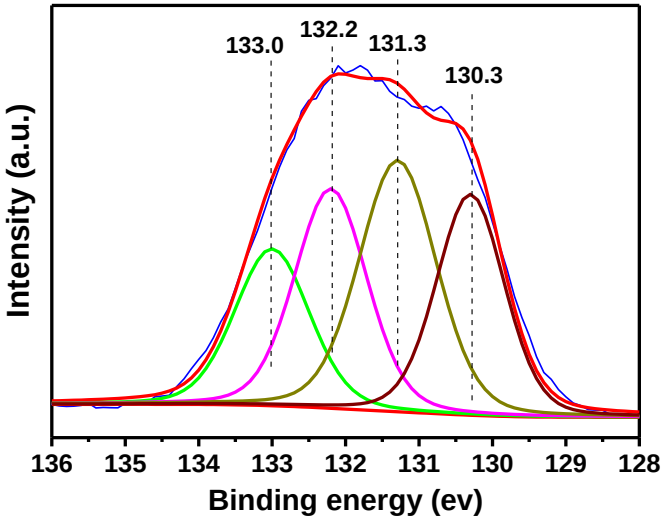
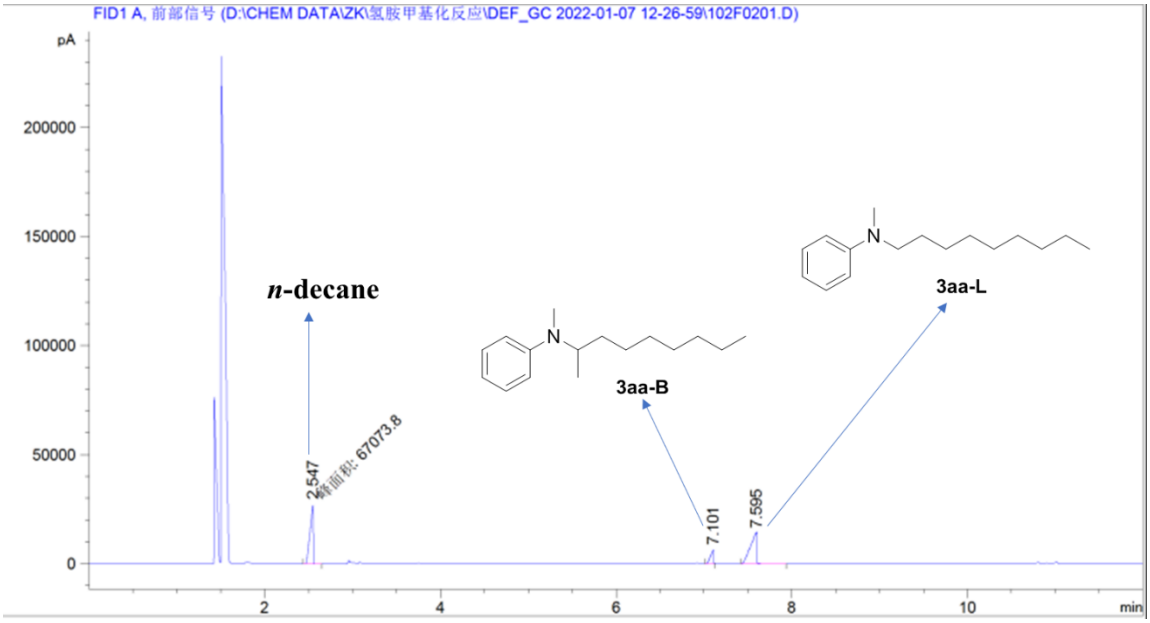


Figure S13. P2p-XPS analysis of Rh@CPOL-DPMphos&*p*-3vPPh$_3$.

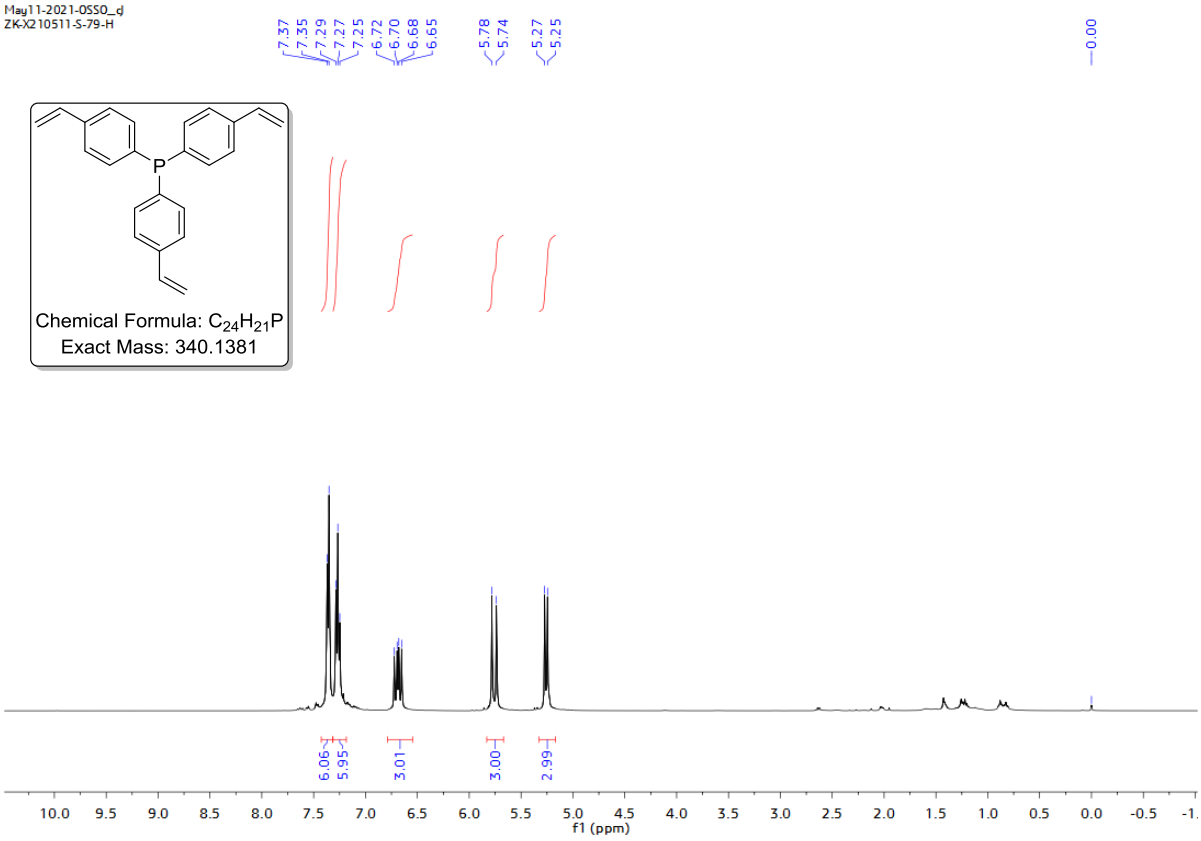
8. Copies of quantitative GC and NMR spectra

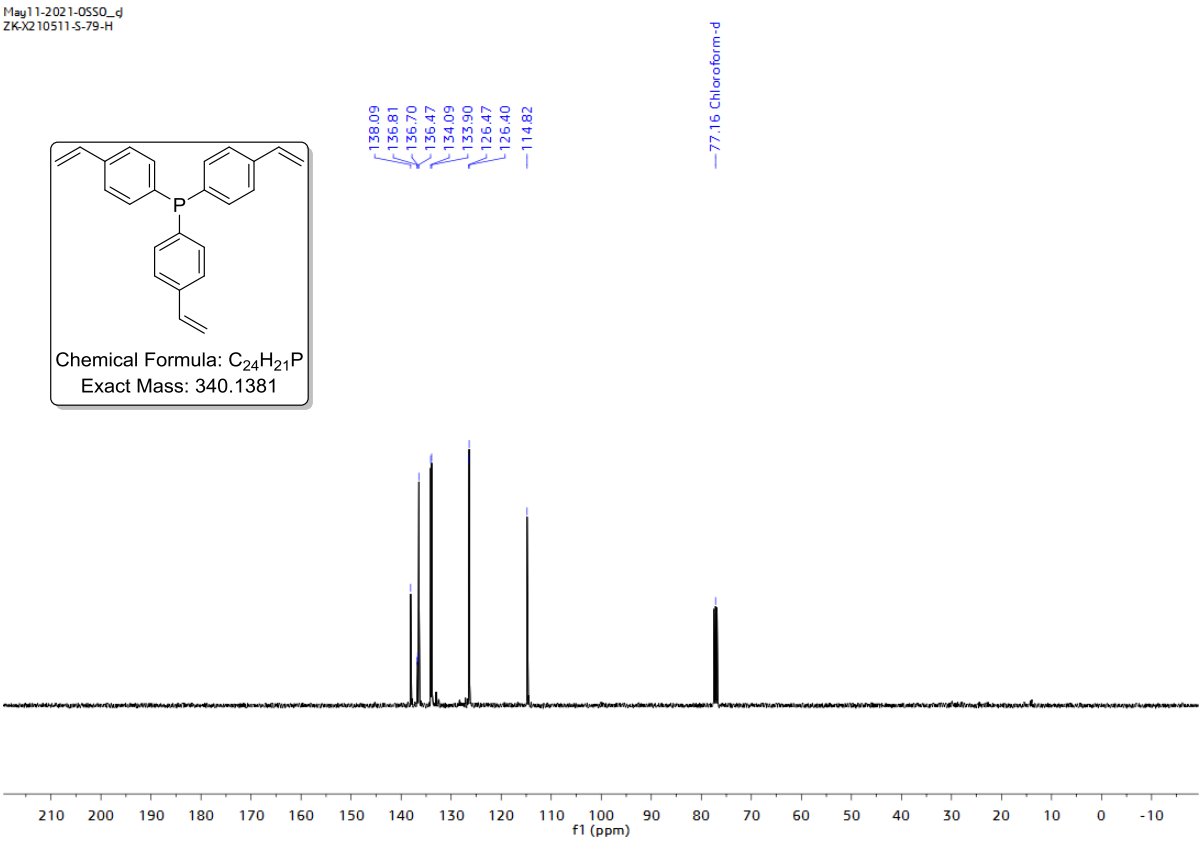
Quantitative GC spectra of 1-octene hydroaminomethylation

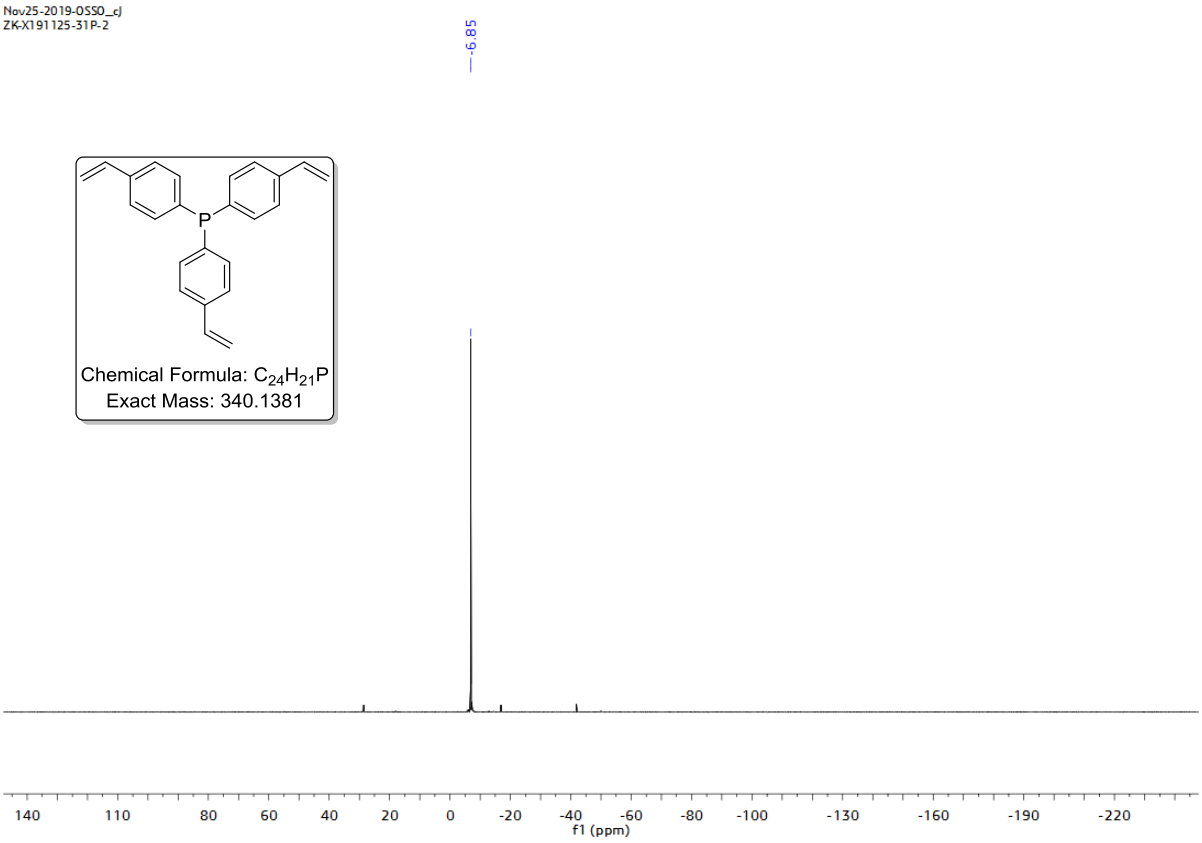


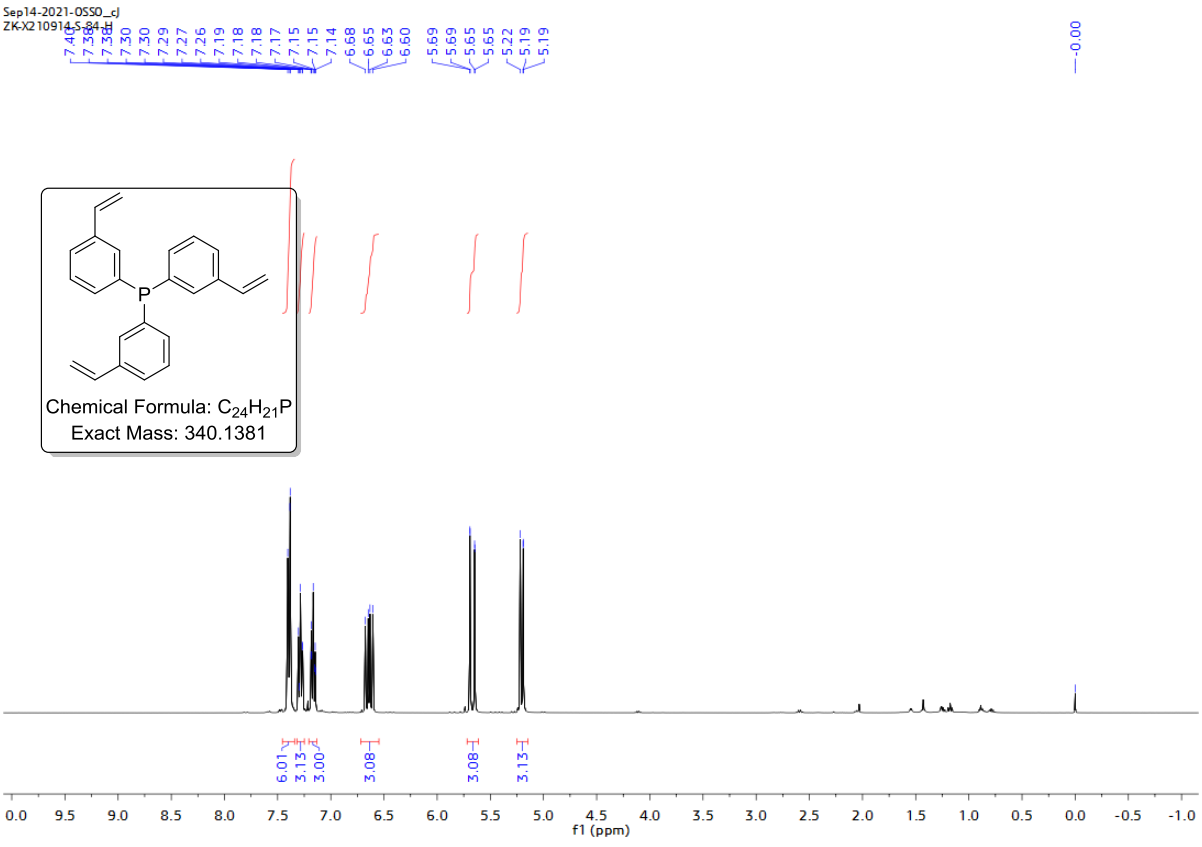
Peak No.	Ret. time	Area	Area%
1	2.547	67074	43.4885
2	7.101	13074	8.47673
3	7.595	74085	48.03472
Total		15423	100.0000

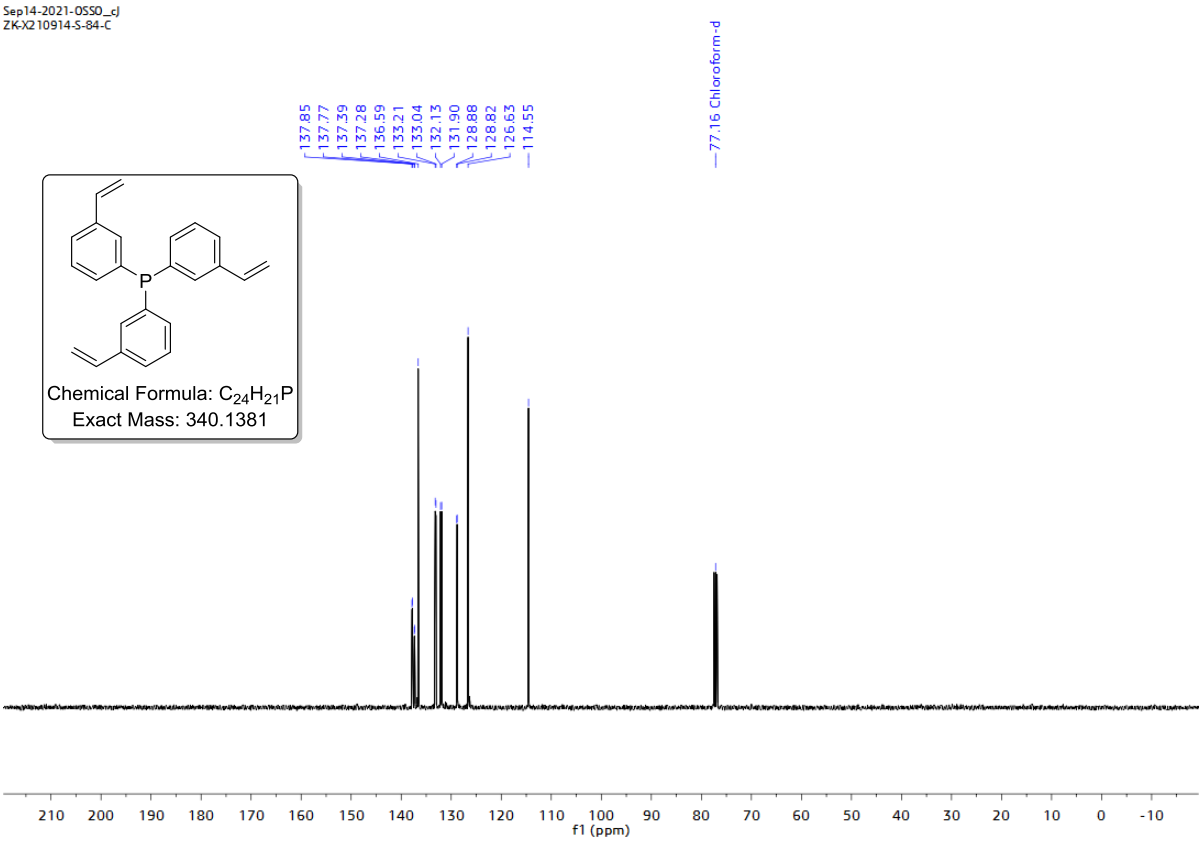
NMR spectra of the ligands

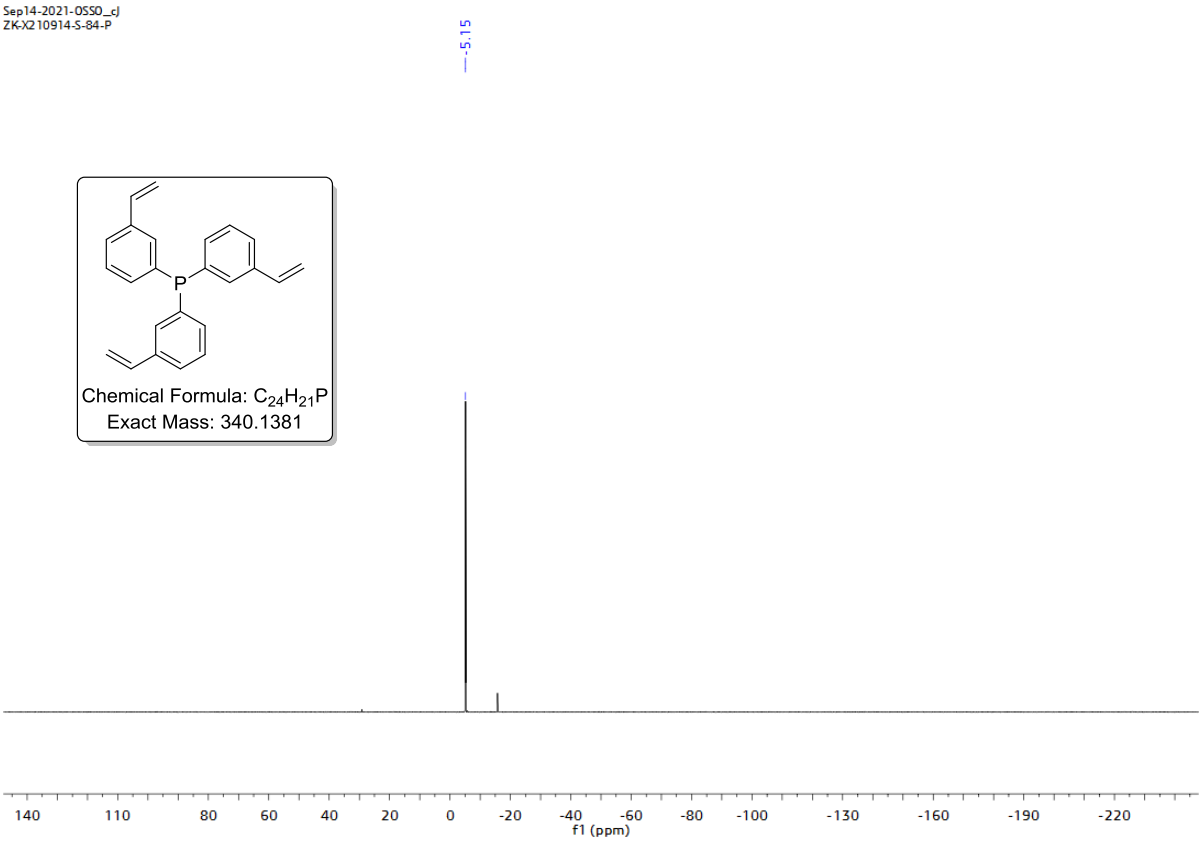


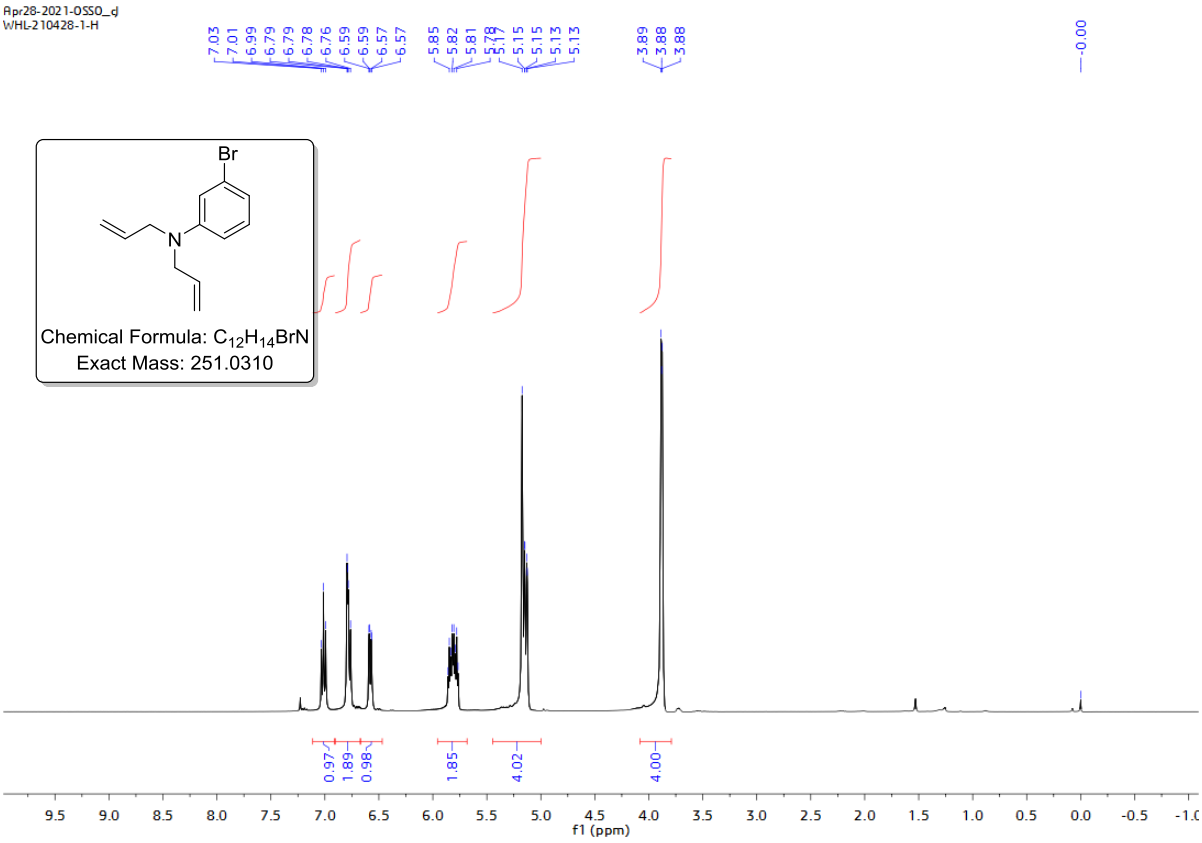


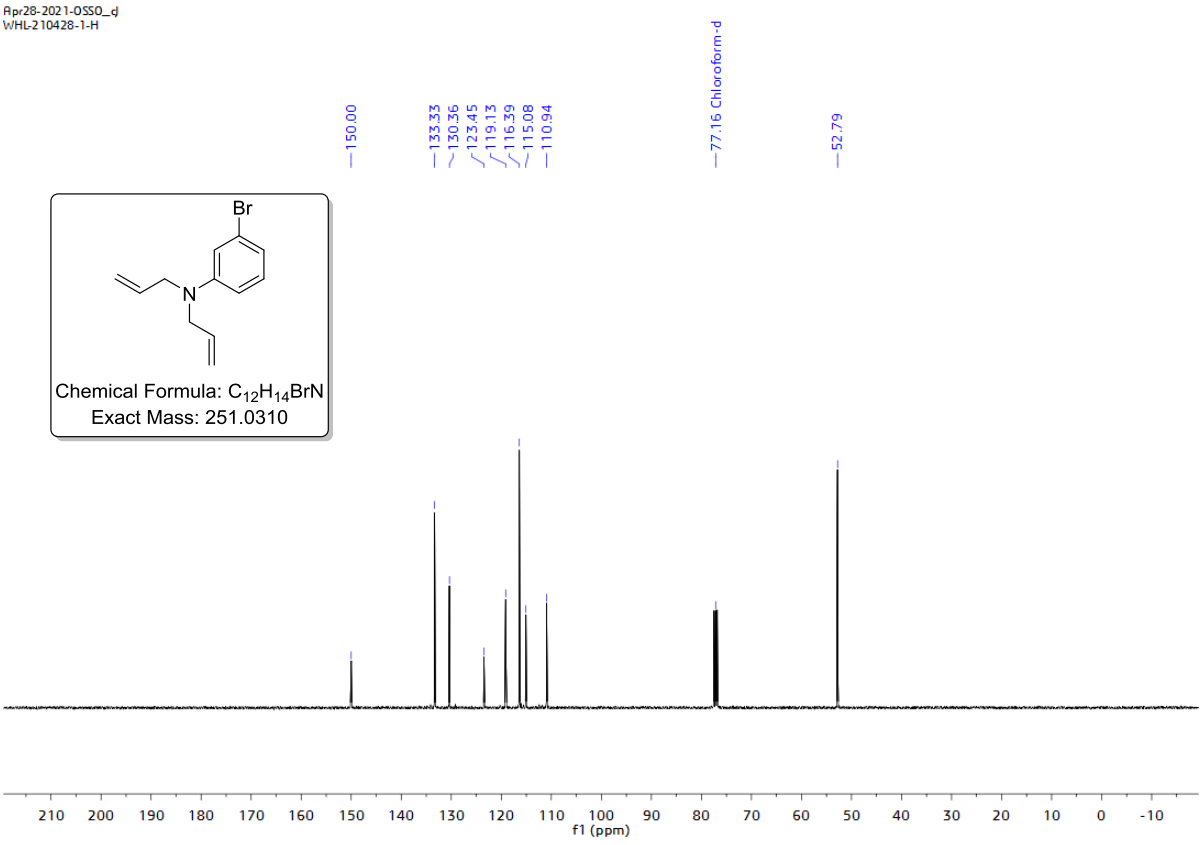

Nov25-2019-OSSO_cj
ZK-X191125-31P-2
-6.85
Chemical Formula: C24H21P
Exact Mass: 340.1381
140 110 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220
f1 (ppm)

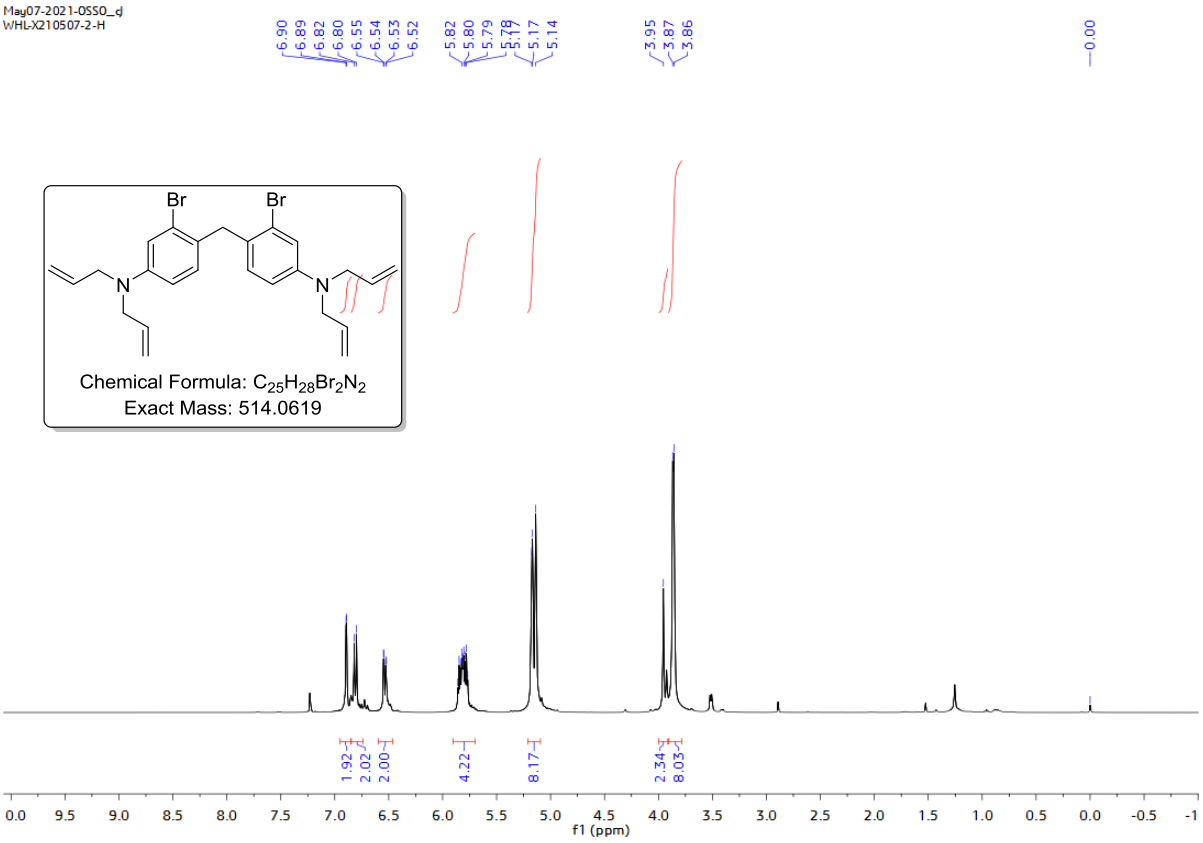

Sep14-2021-OSSO_cj
ZK-X210914-5-84-H
7.40 7.36 7.35 7.30 7.30 7.29 7.27 7.26 7.19 7.18 7.18 7.17 7.15 7.15 7.14 6.68 6.65 6.63 6.60 5.69 5.69 5.65 5.65 5.22 5.19 5.19
-0.00
Chemical Formula: C24H21P
Exact Mass: 340.1381
6.01 3.13 3.00 3.08 3.08 3.13
0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

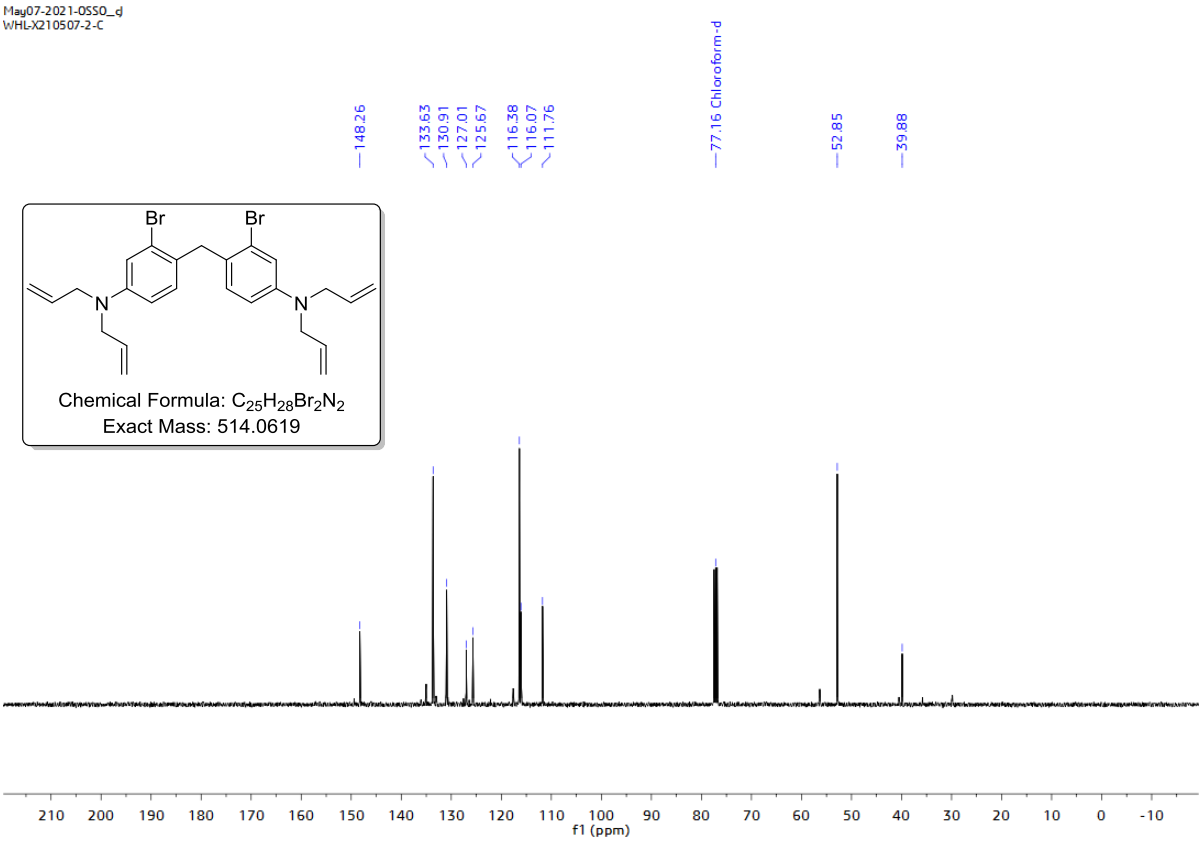

Sep14-2021-OSSO_cj
ZK-X210914-S-84-C
137.85
137.77
137.39
137.28
136.59
135.21
133.04
132.13
131.90
128.88
128.82
126.63
114.55
77.16 Chloroform-d
Chemical Formula: C24H21P
Exact Mass: 340.1381
f1 (ppm)

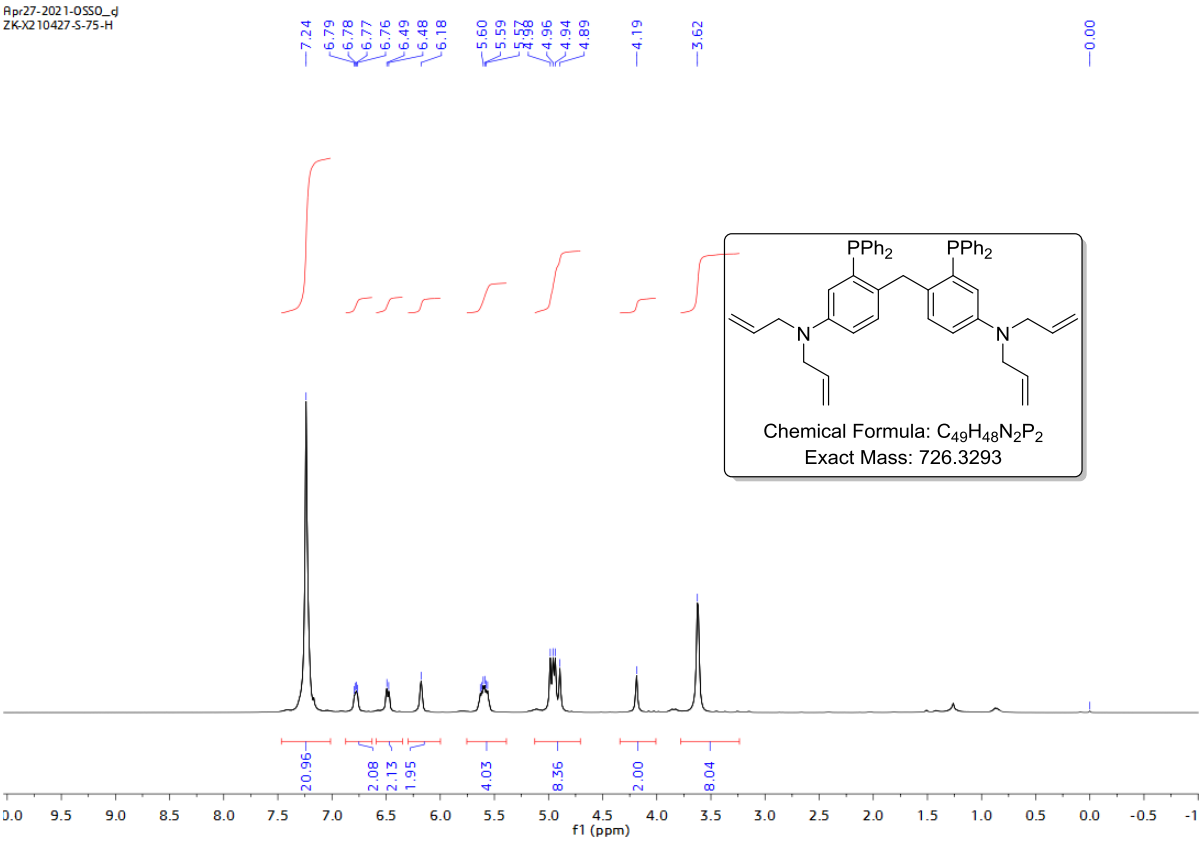

Sep14-2021-OSSO_cj
ZK-X210914-S-84-P
-5.15
Chemical Formula: C24H21P
Exact Mass: 340.1381
f1 (ppm)

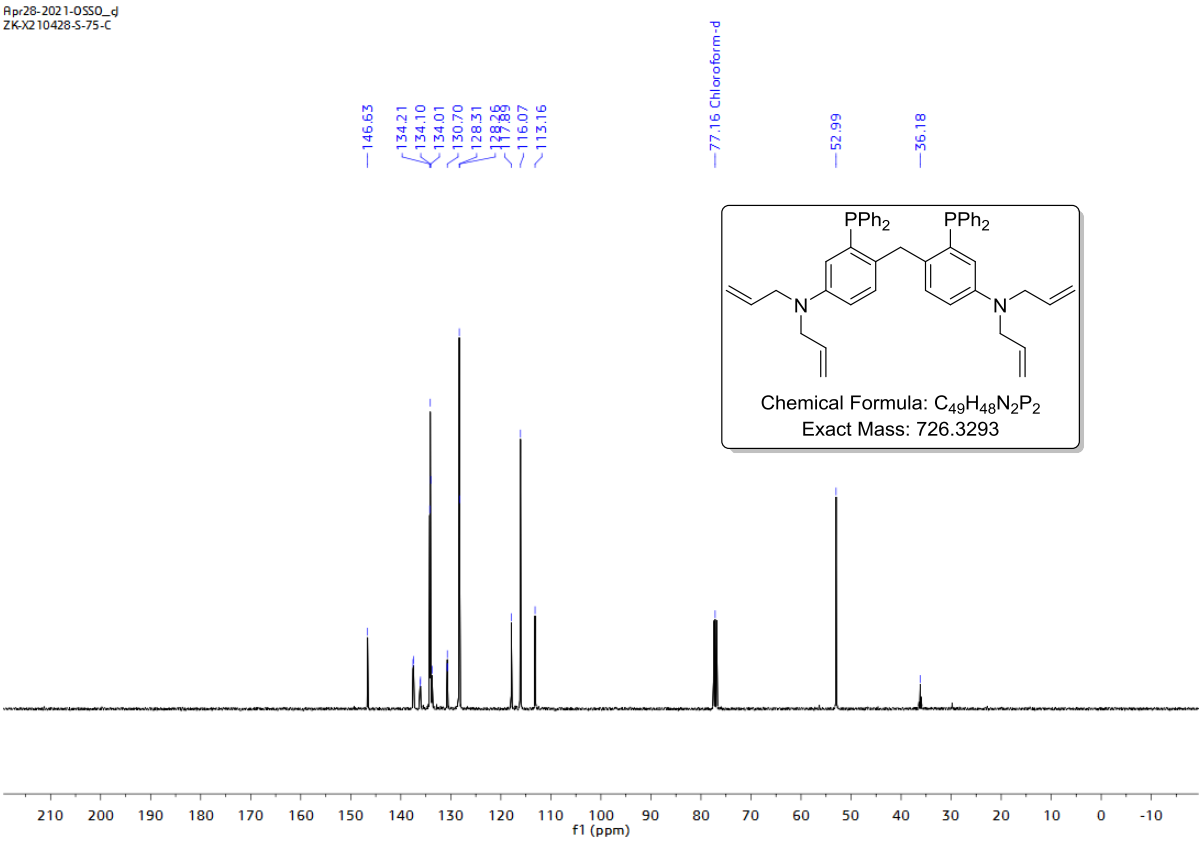

Apr28-2021-OSS0_d
WHL-210428-1-H
7.03
7.01
6.99
6.79
6.79
6.78
6.76
6.59
6.59
6.57
6.57
5.85
5.82
5.81
5.15
5.15
5.13
5.13
3.89
3.88
3.88
0.00
Br
N
Chemical Formula: C12H14BrN
Exact Mass: 251.0310
0.97
1.89
0.98
1.85
4.02
4.00
f1 (ppm)


Apr28-2021-OSS0_d
WHL-210428-1-H
150.00
133.33
130.36
123.45
119.13
116.39
115.08
110.94
77.16 Chloroform-d
52.79
Br
N
Chemical Formula: C12H14BrN
Exact Mass: 251.0310
f1 (ppm)

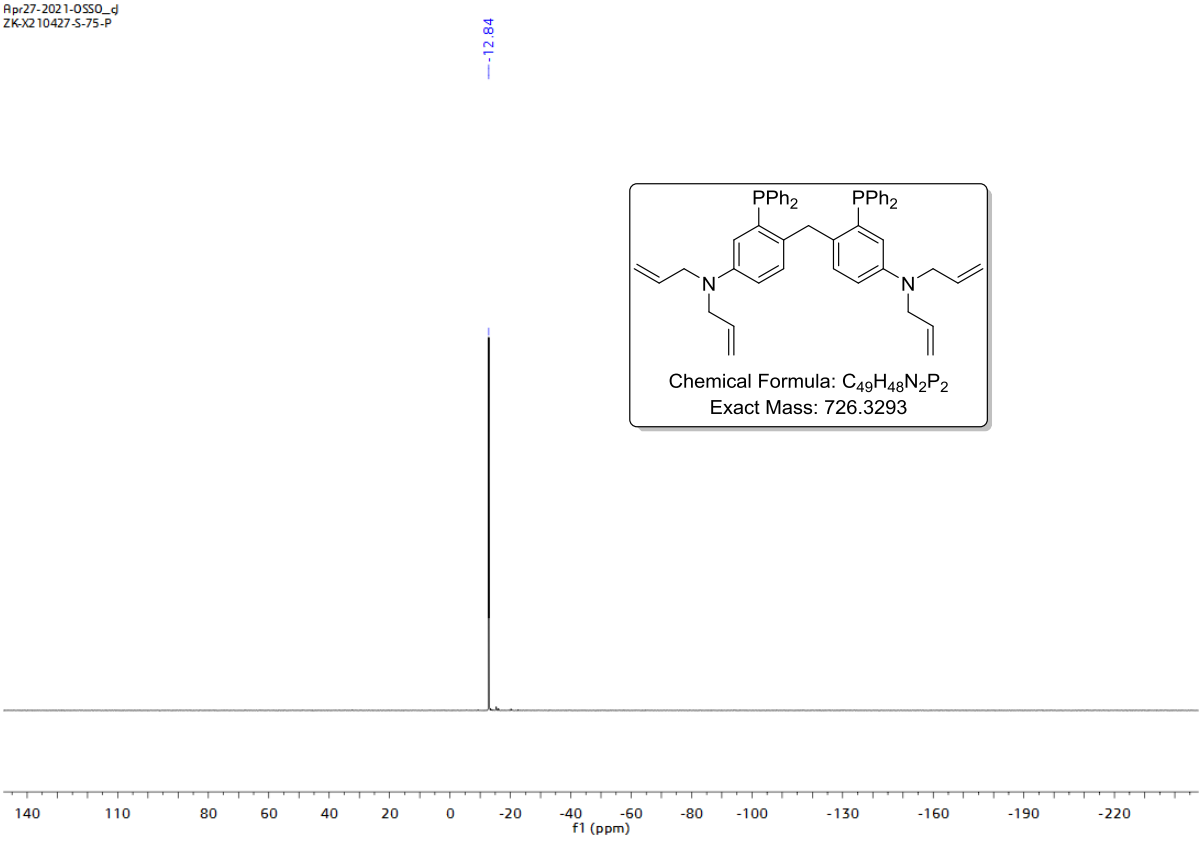

May07-2021-0SS0_d
WHL-X210507-2-H
6.90
6.89
6.82
6.80
6.55
6.54
6.53
6.52
5.82
5.80
5.79
5.78
5.17
5.14
3.95
3.87
3.86
0.00
Br
Br
N
N
Chemical Formula: C25H28Br2N2
Exact Mass: 514.0619
1.92
2.02
2.00
4.22
8.17
2.34
8.03
f1 (ppm)


May07-2021-0SS0_d
WHL-X210507-2-C
148.26
133.63
130.91
127.01
125.67
116.38
116.07
111.76
77.16 Chloroform-d
52.85
39.88
Br
Br
N
N
Chemical Formula: C25H28Br2N2
Exact Mass: 514.0619
f1 (ppm)

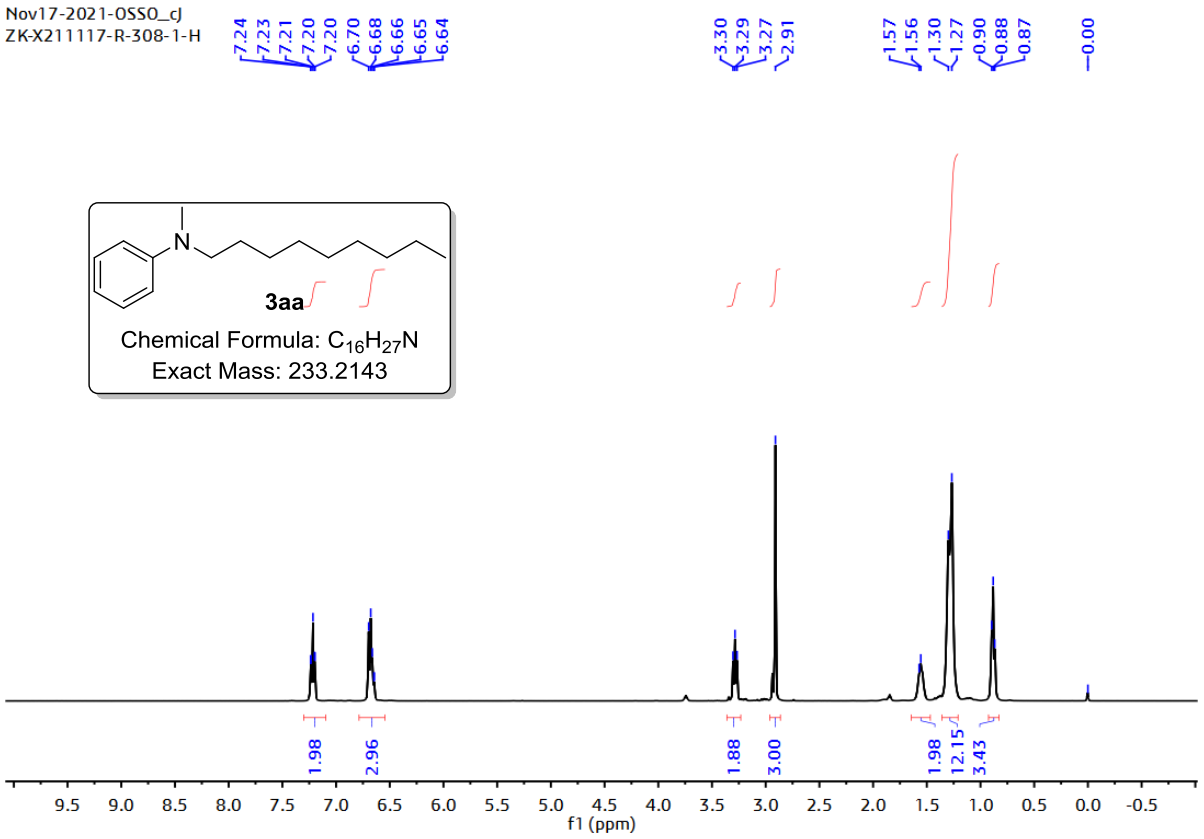

Apr27-2021-OSSO_d
ZK-X210427-S-75-H
7.24
6.79
6.78
6.77
6.76
6.49
6.48
6.18
5.60
5.59
5.57
4.98
4.96
4.94
4.89
4.19
3.62
0.00
PPh2
PPh2
N
N
Chemical Formula: C49H48N2P2
Exact Mass: 726.3293
20.96
2.08
2.13
1.95
4.03
8.36
2.00
8.04
f1 (ppm)

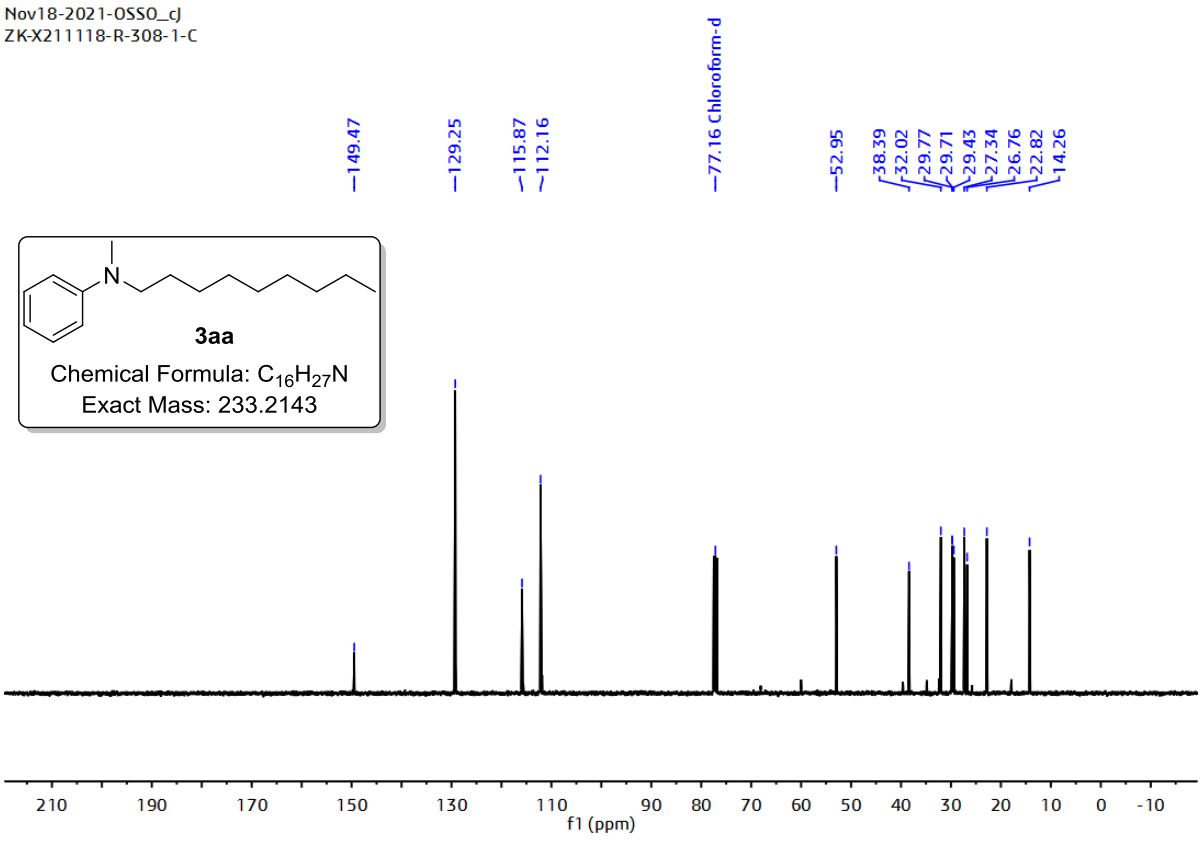

Apr28-2021-OSSO_d
ZK-X210428-S-75-C
146.63
134.21
134.10
134.01
130.70
128.31
116.07
113.16
77.16 Chloroform-d
52.99
36.18
PPh2
PPh2
N
N
Chemical Formula: C49H48N2P2
Exact Mass: 726.3293
f1 (ppm)

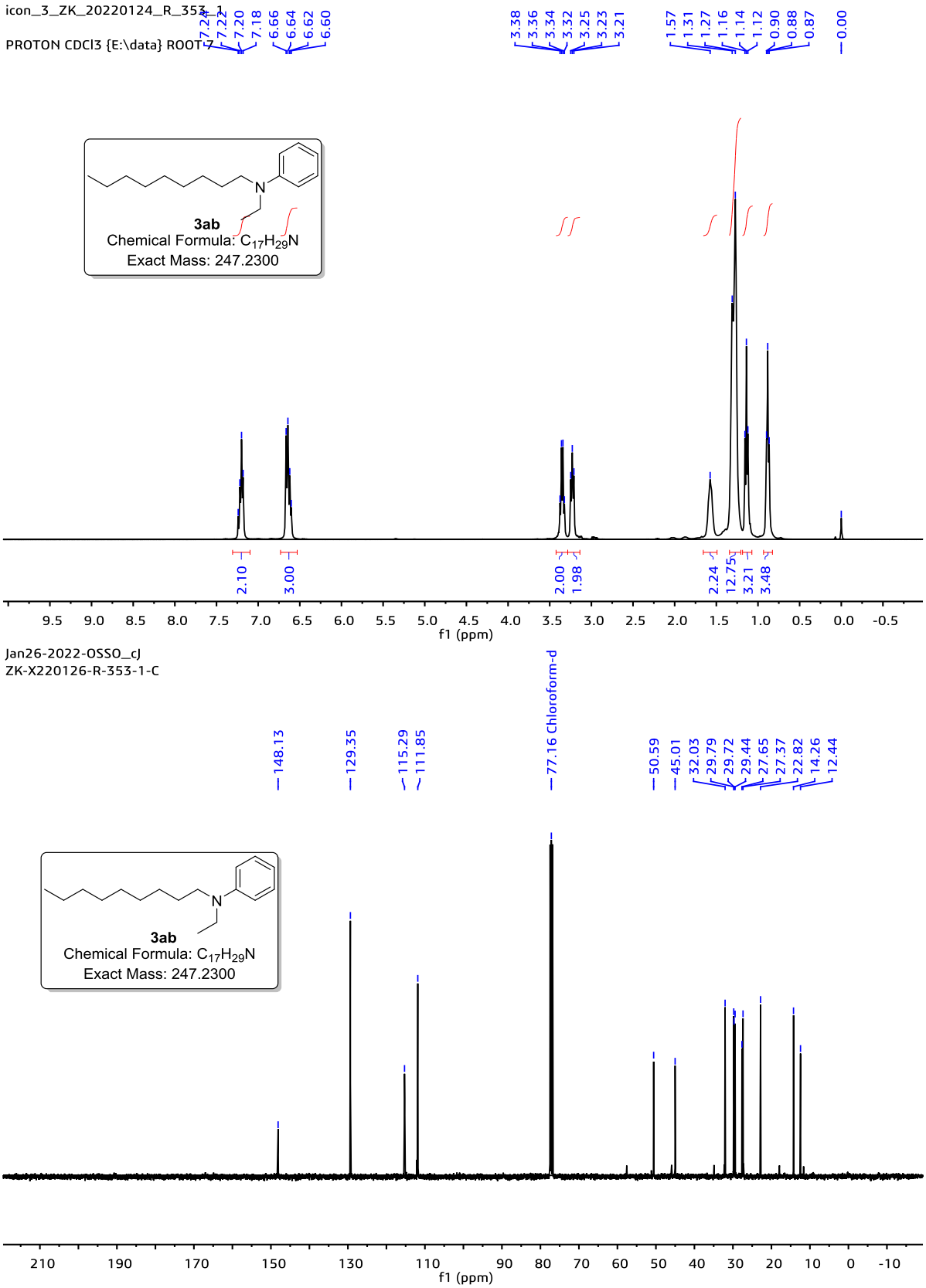
Apr27-2021-OSS0_cj
ZK-X210427-S-75-P

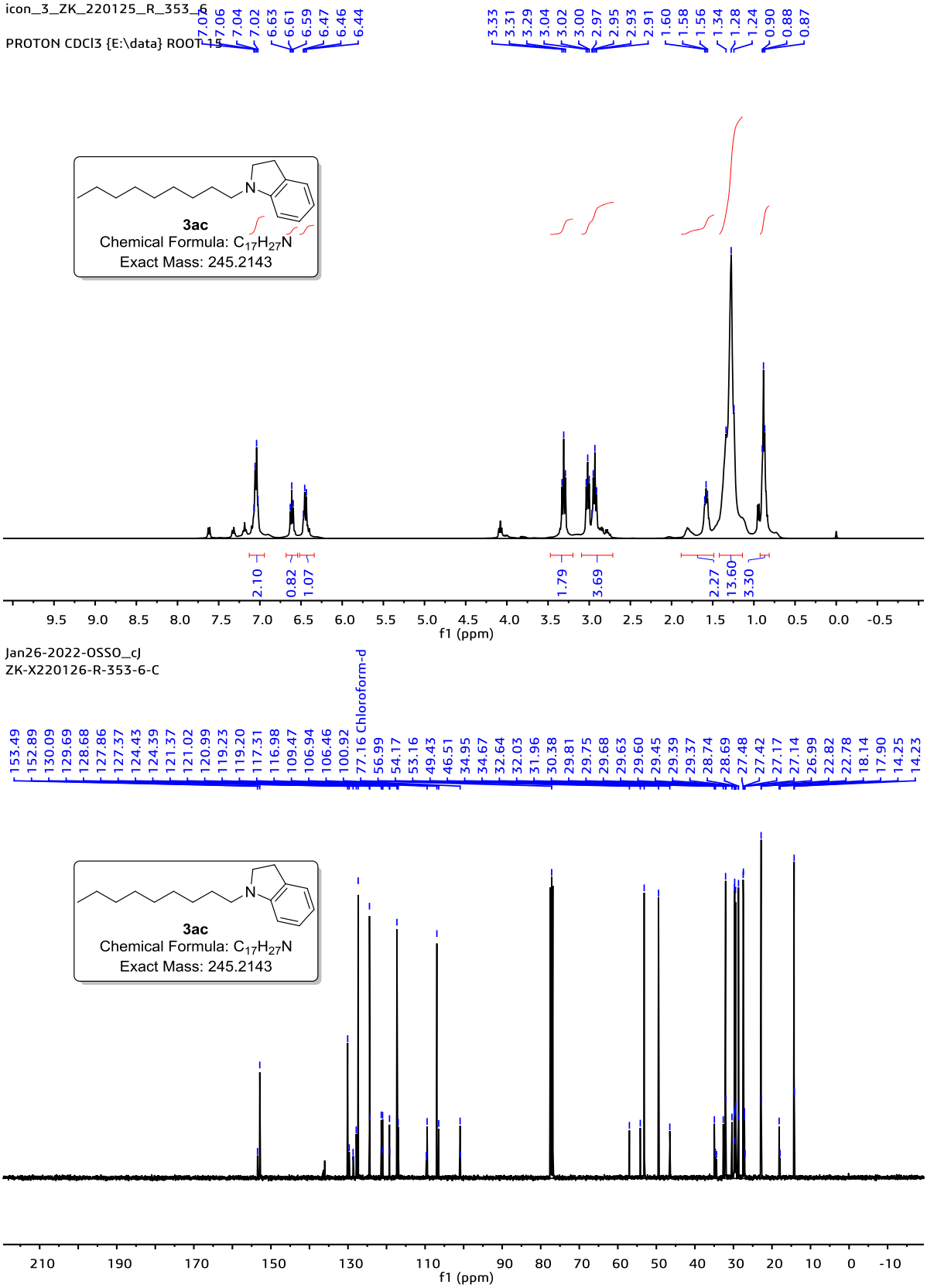


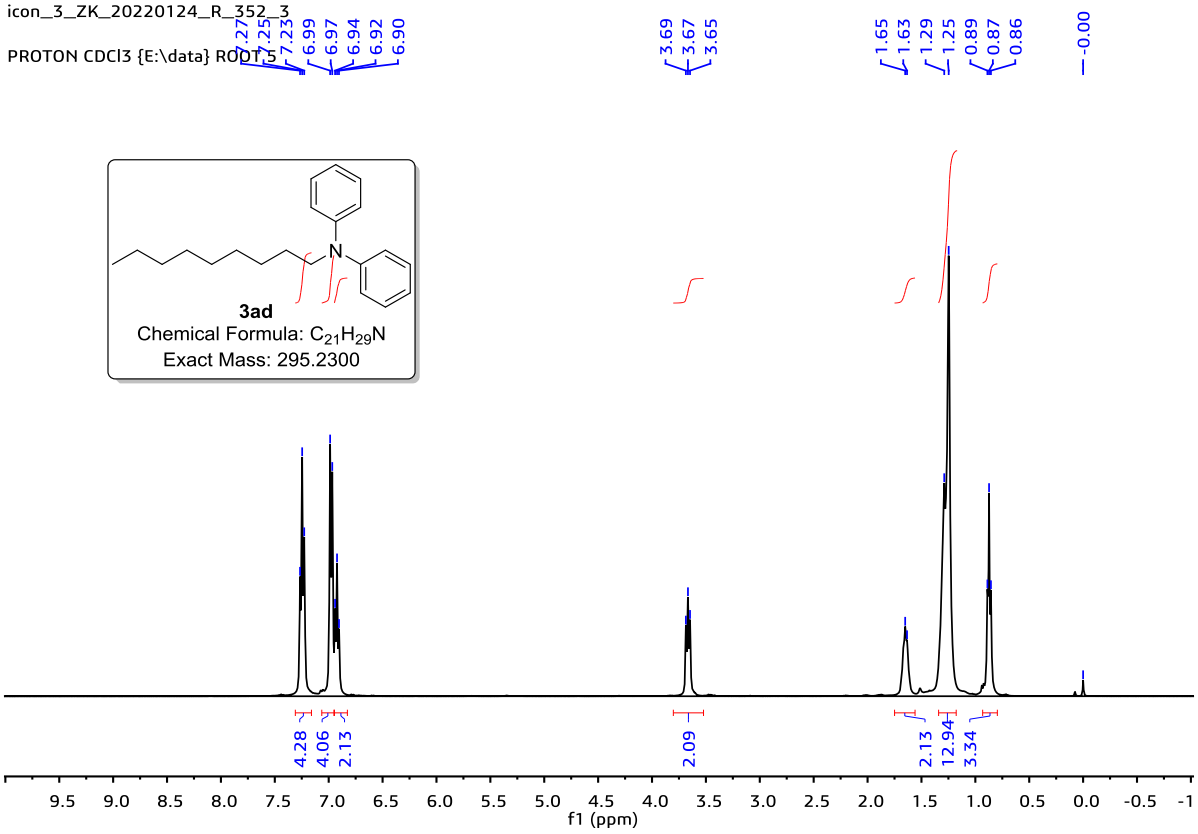
NMR spectra of the products

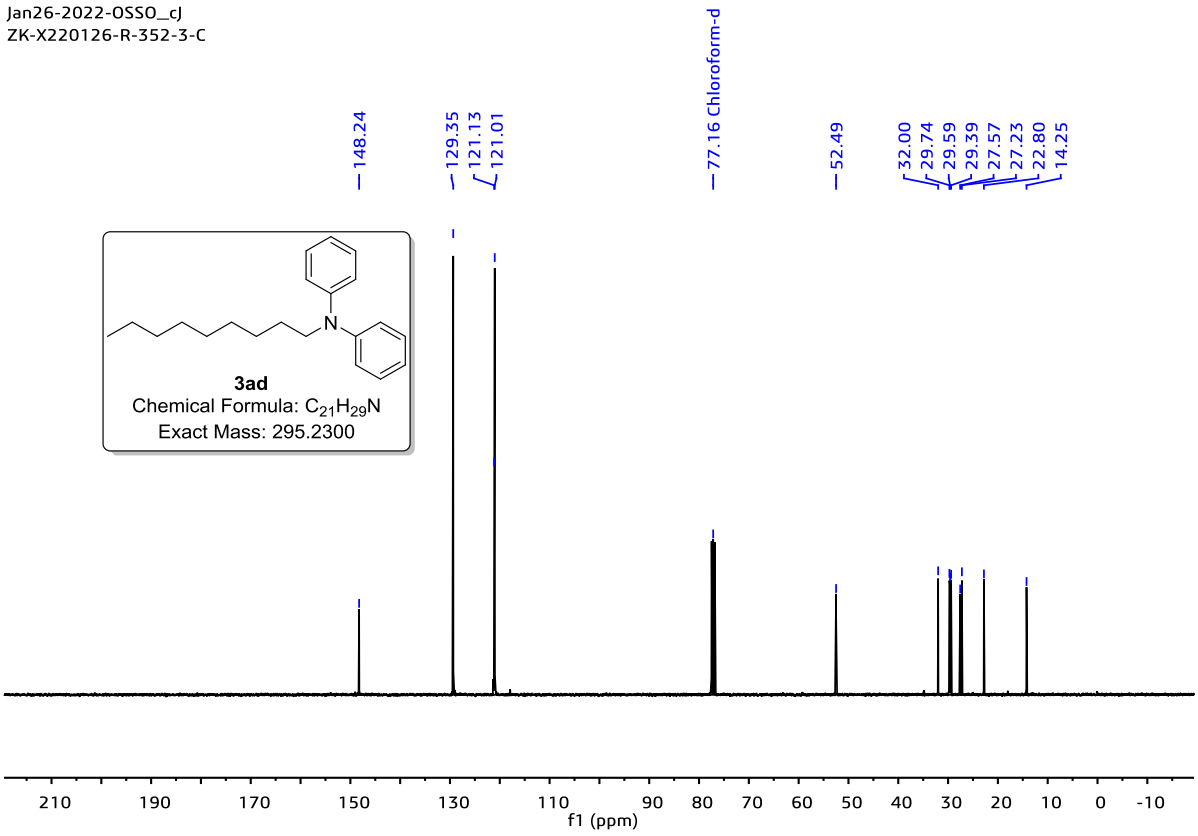


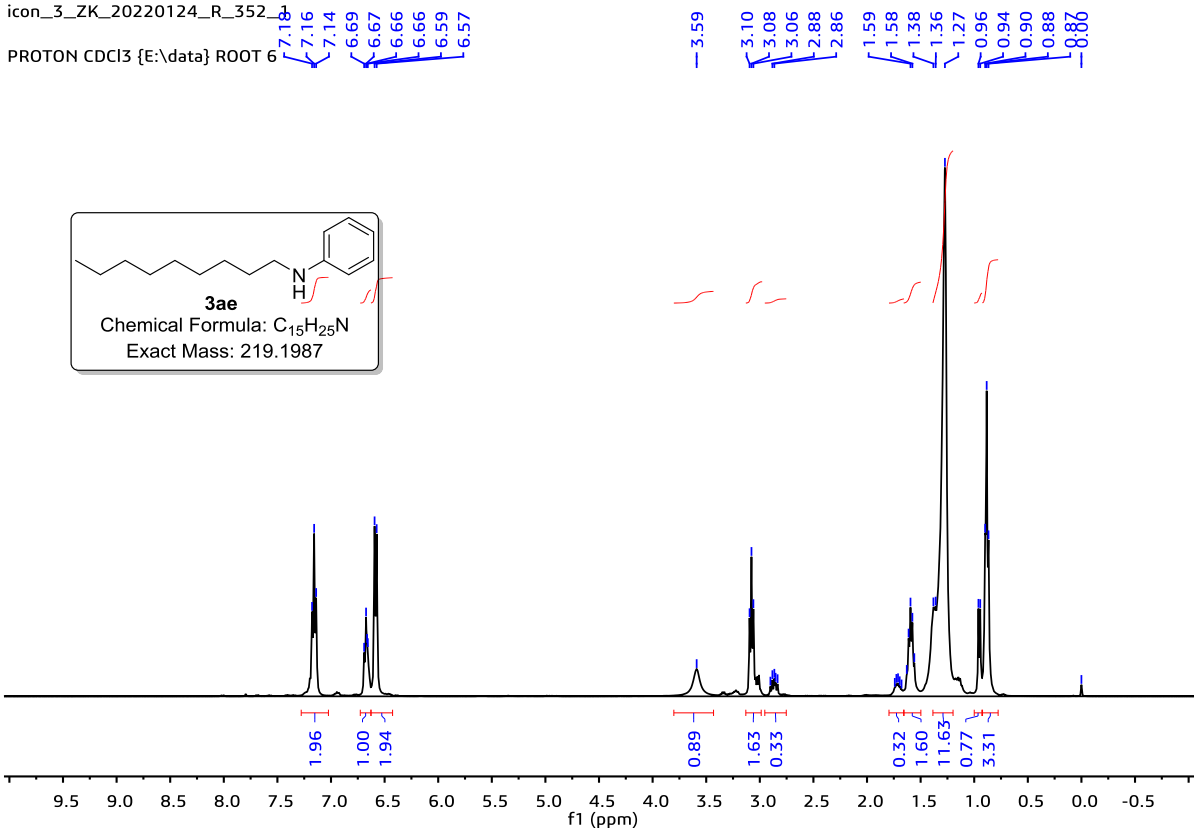


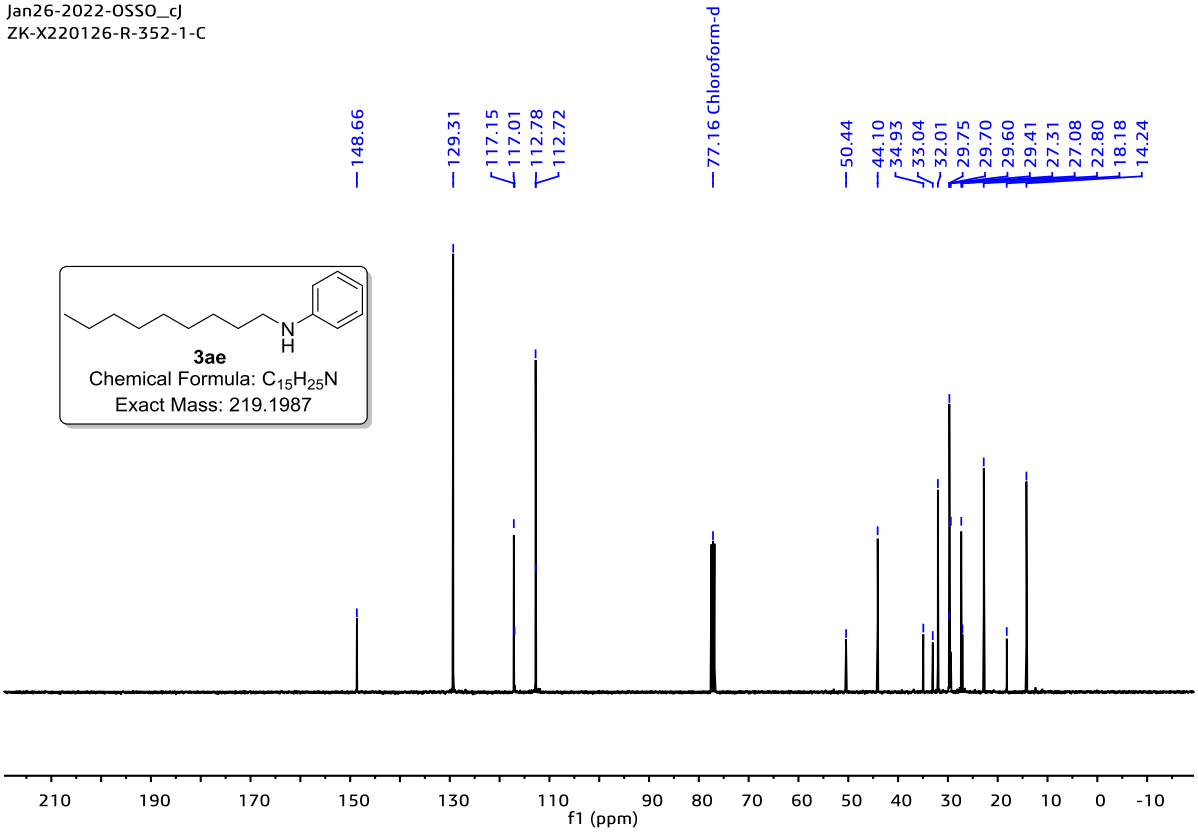

icon_3_ZK_20220124_R_353_1
PROTON CDCl3 {E:\data} ROOT 7
7.24
7.22
7.20
7.18
6.66
6.64
6.62
6.60
3.38
3.36
3.34
3.32
3.25
3.23
3.21
1.57
1.31
1.27
1.16
1.14
1.12
0.90
0.88
0.87
0.00
3ab
Chemical Formula: C17H29N
Exact Mass: 247.2300
2.10
3.00
2.00
1.98
2.24
12.75
3.21
3.48
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
Jan26-2022-OSSO_cJ
ZK-X220126-R-353-1-C
148.13
129.35
115.29
111.85
77.16 Chloroform-d
50.59
45.01
32.03
29.79
29.72
29.44
27.65
27.37
22.82
14.26
12.44
3ab
Chemical Formula: C17H29N
Exact Mass: 247.2300
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

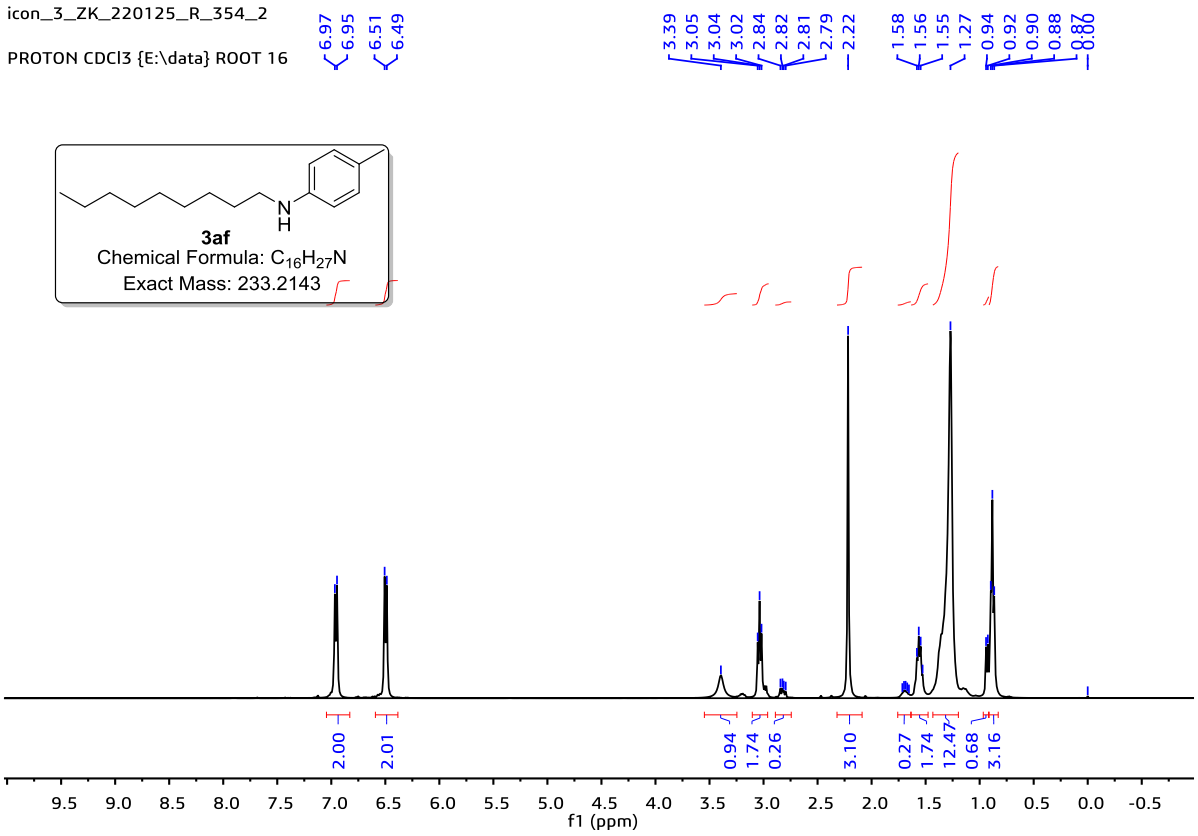

icon_3_ZK_220125_R_353_6
PROTON CDCl3 {E:\data} ROOT 15
7.08
7.06
7.04
7.02
6.63
6.61
6.59
6.47
6.46
6.44
3.33
3.31
3.29
3.04
3.02
3.00
2.97
2.95
2.93
2.91
1.60
1.58
1.56
1.34
1.28
1.24
0.90
0.88
0.87
3ac
Chemical Formula: C17H27N
Exact Mass: 245.2143
2.10
0.82
1.07
1.79
3.69
2.27
13.60
3.30
f1 (ppm)
Jan26-2022-OSSO_cJ
ZK-X220126-R-353-6-C
153.49
152.89
130.09
129.69
128.68
127.86
127.37
124.43
124.39
121.37
121.02
120.99
119.23
119.20
117.31
116.98
109.47
106.94
106.46
100.92
77.16 Chloroform-d
56.99
54.17
53.16
49.43
46.51
34.95
34.67
32.64
32.03
31.96
30.38
29.81
29.75
29.68
29.63
29.60
29.45
29.39
29.37
28.74
28.69
27.48
27.42
27.17
27.14
26.99
22.82
22.78
18.14
17.90
14.25
14.23
3ac
Chemical Formula: C17H27N
Exact Mass: 245.2143
f1 (ppm)

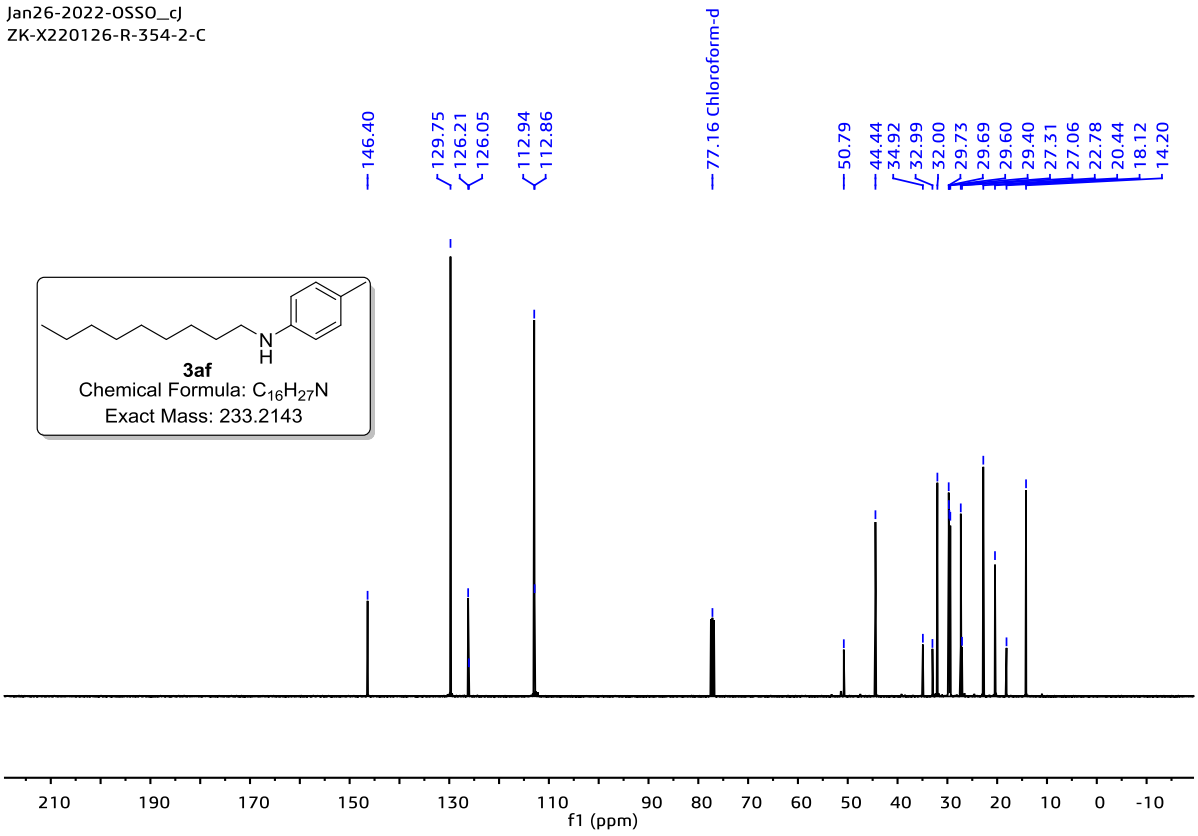

icon_3_ZK_20220124_R_352_3
PROTON CDCl3 {E:\data} ROUT 5
7.27
7.25
7.23
6.99
6.97
6.94
6.92
6.90
3.69
3.67
3.65
1.65
1.63
1.29
1.25
0.89
0.87
0.86
-0.00
3ad
Chemical Formula: C21H29N
Exact Mass: 295.2300
4.28
4.06
2.13
2.09
2.13
12.94
3.34
f1 (ppm)


Jan26-2022-OSSO_cJ
ZK-X220126-R-352-3-C
148.24
129.35
121.13
121.01
77.16 Chloroform-d
52.49
32.00
29.74
29.59
29.39
27.57
27.23
22.80
14.25
3ad
Chemical Formula: C21H29N
Exact Mass: 295.2300
f1 (ppm)


icon_3_ZK_20220124_R_352_1
PROTON CDCl3 {E:\data} ROOT 6
7.18
7.16
7.14
6.69
6.67
6.66
6.66
6.59
6.57
3.59
3.10
3.08
3.06
2.88
2.86
1.59
1.58
1.38
1.36
1.27
0.96
0.94
0.90
0.88
0.87
0.00
3ae
Chemical Formula: C15H25N
Exact Mass: 219.1987
1.96
1.00
1.94
0.89
1.63
0.33
0.32
1.60
11.63
0.77
3.31
f1 (ppm)

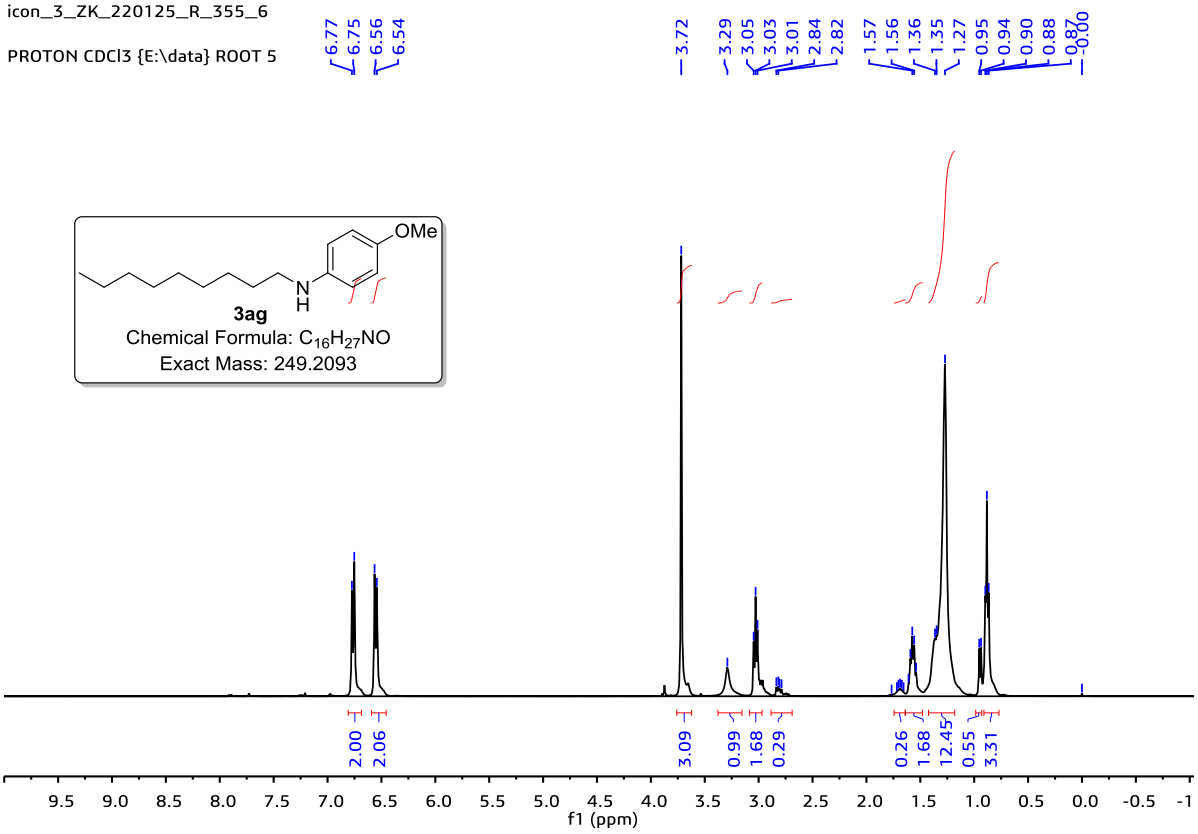

Jan26-2022-OSSO_cJ
ZK-X220126-R-352-1-C
148.66
129.31
117.15
117.01
112.78
112.72
77.16 Chloroform-d
50.44
44.10
34.93
33.04
32.01
29.75
29.70
29.60
29.41
27.31
27.08
22.80
18.18
14.24
3ae
Chemical Formula: C15H25N
Exact Mass: 219.1987
f1 (ppm)


icon_3_ZK_220125_R_354_2
PROTON CDCl3 {E:\data} ROOT 16
6.97
6.95
6.51
6.49
3.39
3.05
3.04
3.02
2.84
2.82
2.81
2.79
2.22
1.58
1.56
1.55
1.27
0.94
0.92
0.90
0.88
0.87
0.86
3af
Chemical Formula: C16H27N
Exact Mass: 233.2143
2.00
2.01
0.94
1.74
0.26
3.10
0.27
1.74
12.47
0.68
3.16
f1 (ppm)


Jan26-2022-OSSO_cJ
ZK-X220126-R-354-2-C
146.40
129.75
126.21
126.05
112.94
112.86
77.16 Chloroform-d
50.79
44.44
34.92
32.99
32.00
29.73
29.69
29.60
29.40
27.31
27.06
22.78
20.44
18.12
14.20
3af
Chemical Formula: C16H27N
Exact Mass: 233.2143
f1 (ppm)

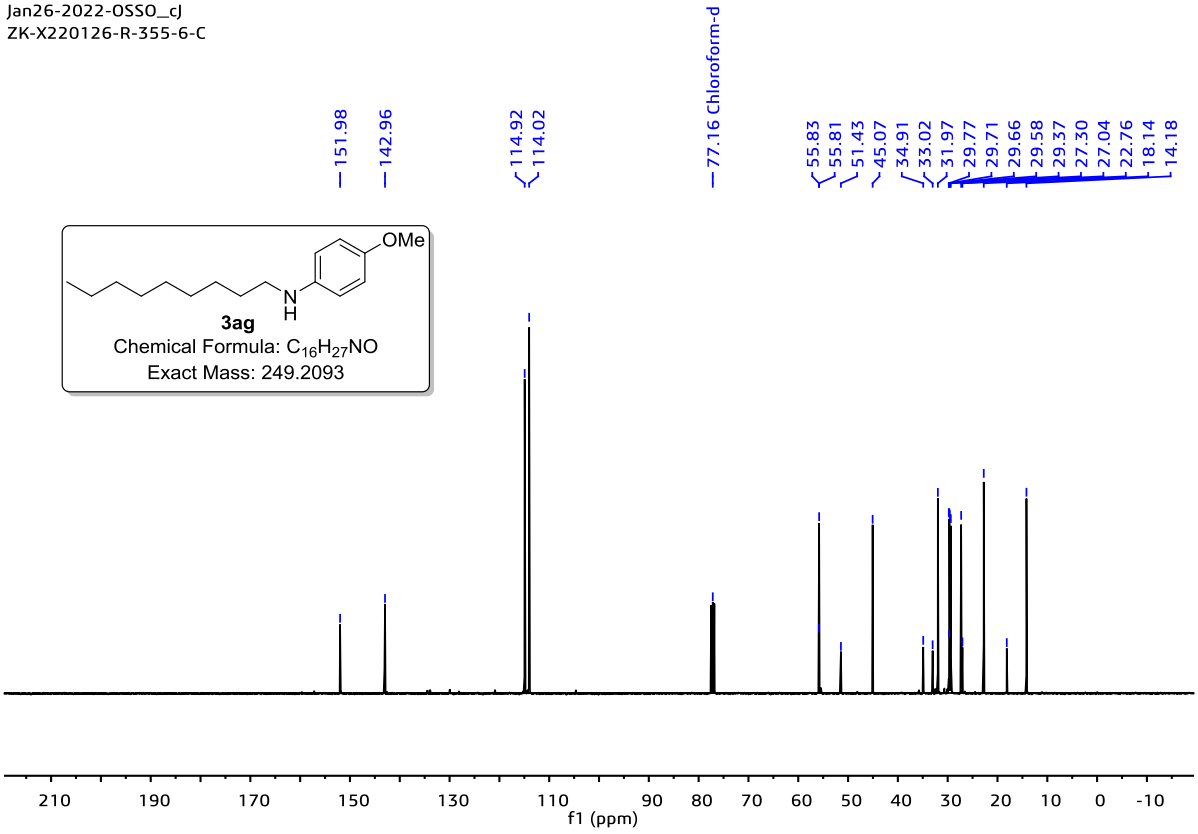
icon_3_ZK_220125_R_355_6

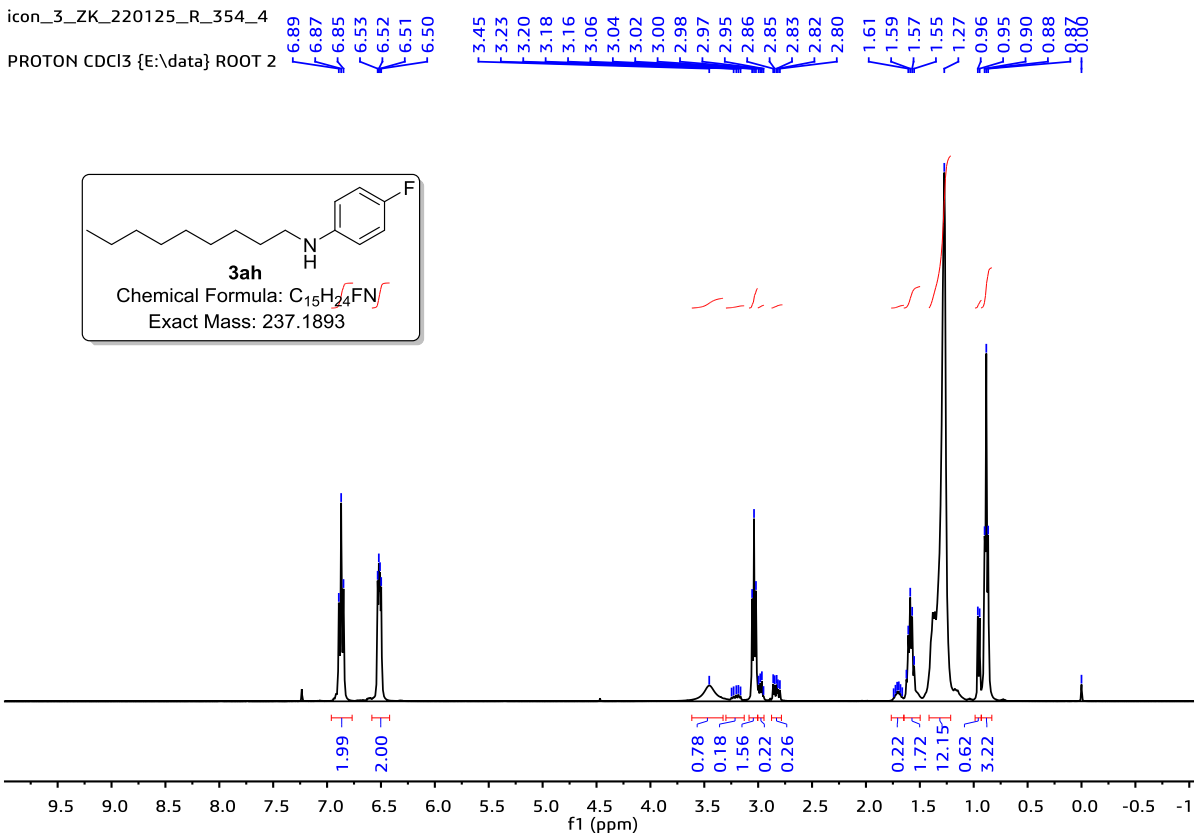
PROTON CDCl3 {E:\data} ROOT 5

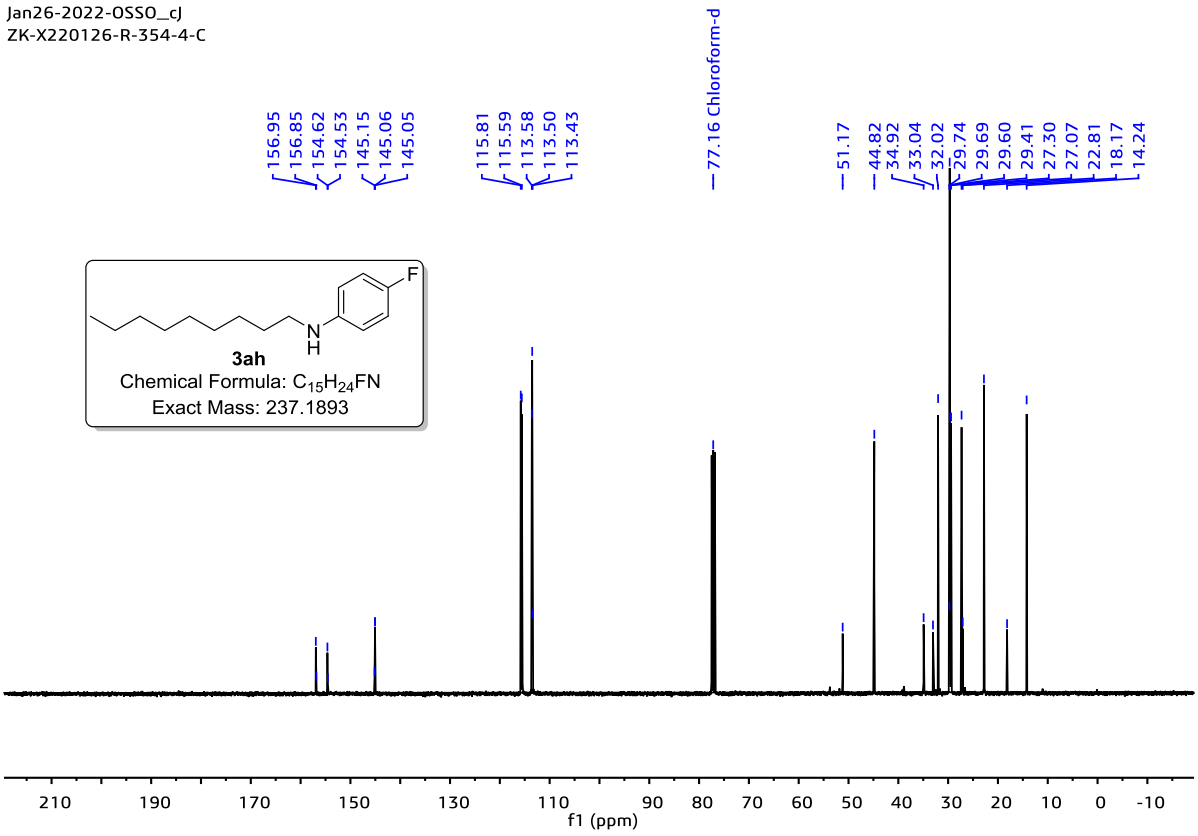


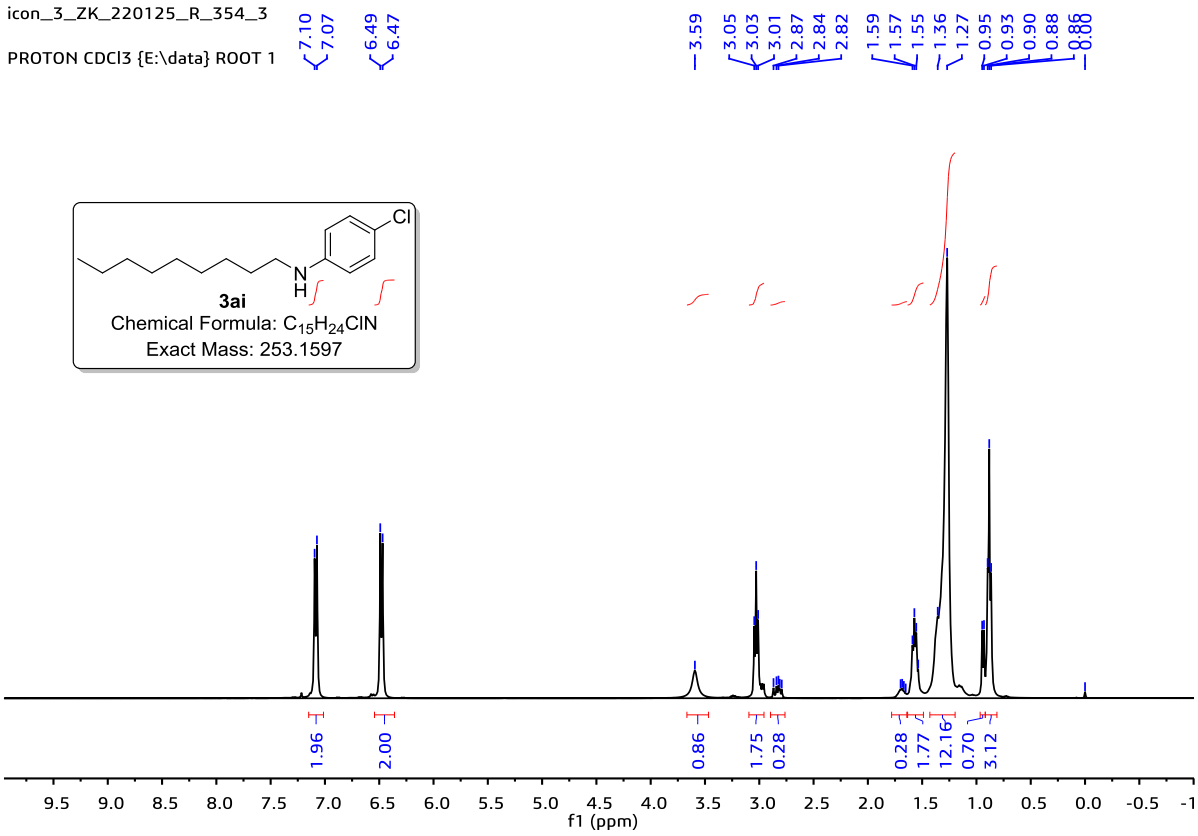
Jan26-2022-OSSO_cJ

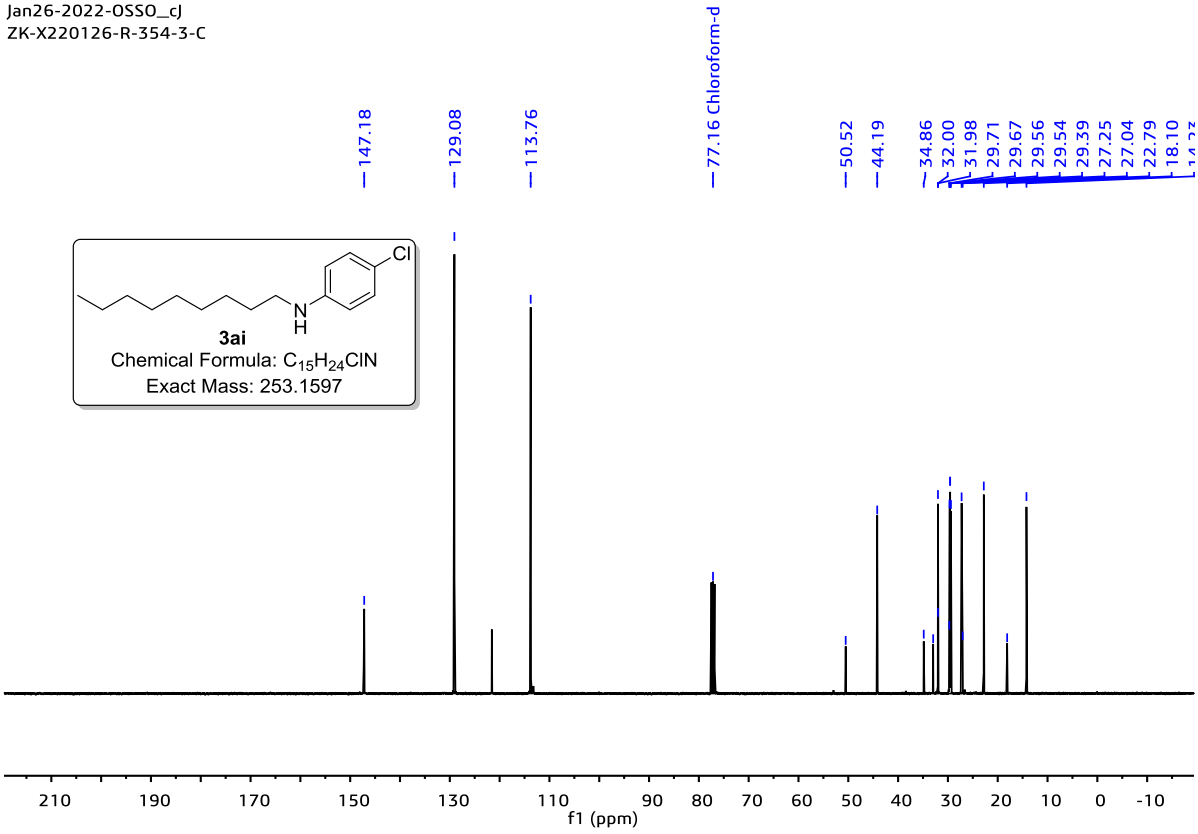
ZK-X220126-R-355-6-C

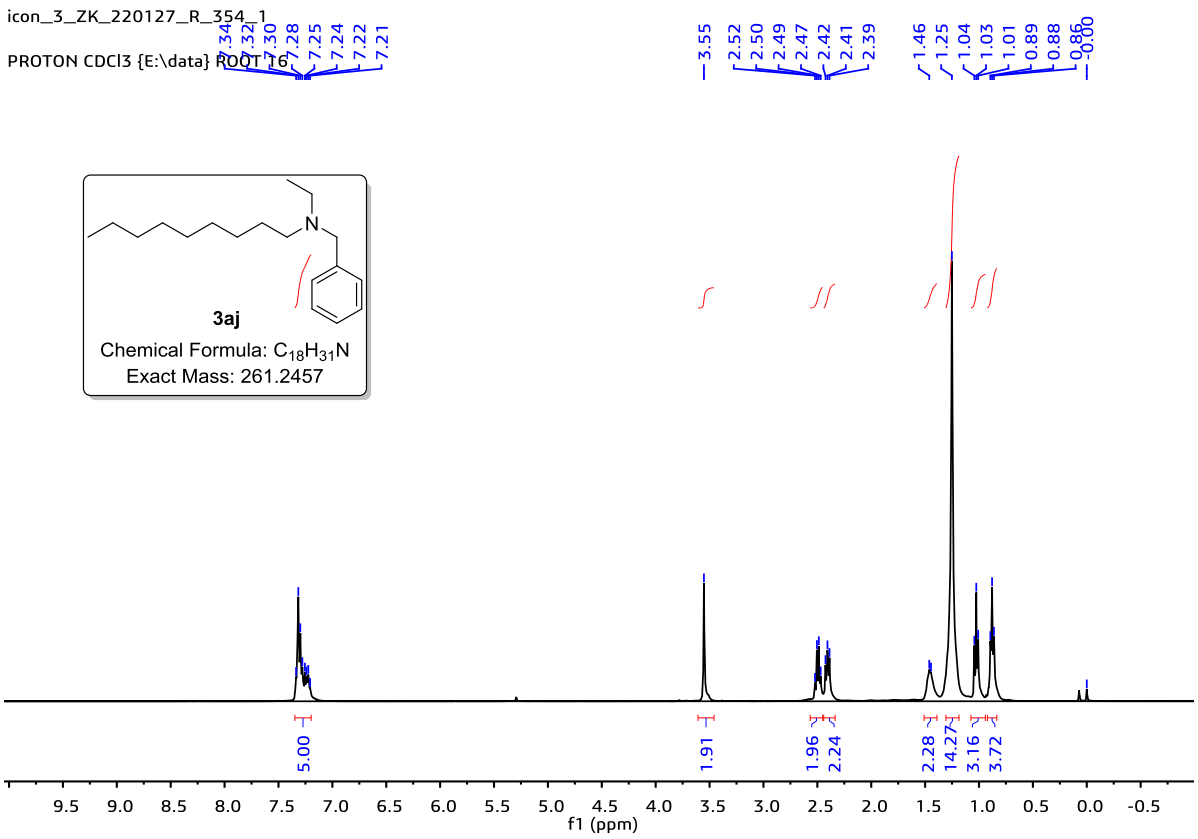


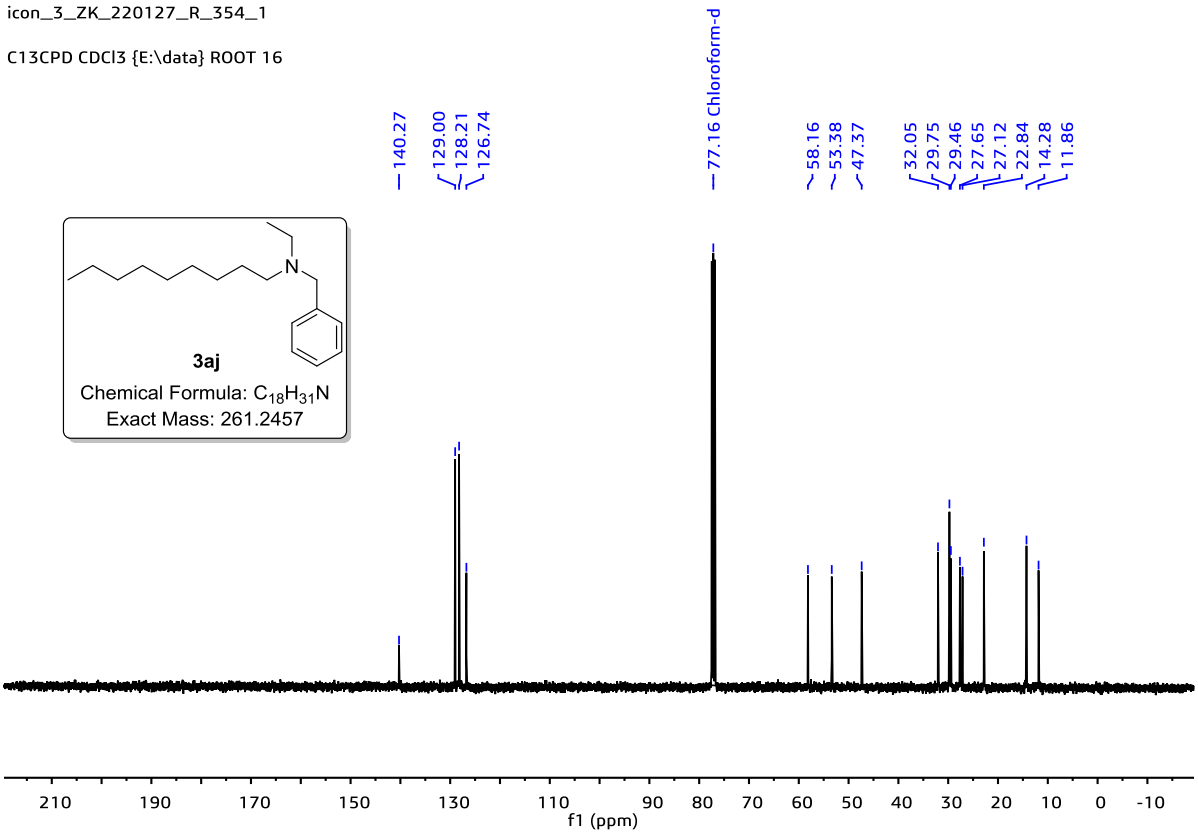

icon_3_ZK_220125_R_354_4
PROTON CDCl3 {E:\data} ROOT 2
6.89 6.87 6.85 6.53 6.52 6.51 6.50
3.45 3.23 3.20 3.18 3.16 3.06 3.04 3.02 3.00 2.98 2.97 2.95 2.86 2.85 2.83 2.82 2.80
1.61 1.59 1.57 1.55 1.27 0.96 0.95 0.90 0.88 0.00
F
N
H
3ah
Chemical Formula: C15H24FN
Exact Mass: 237.1893
1.99 2.00 0.78 0.18 1.56 0.22 0.26 0.22 1.72 12.15 0.62 3.22
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

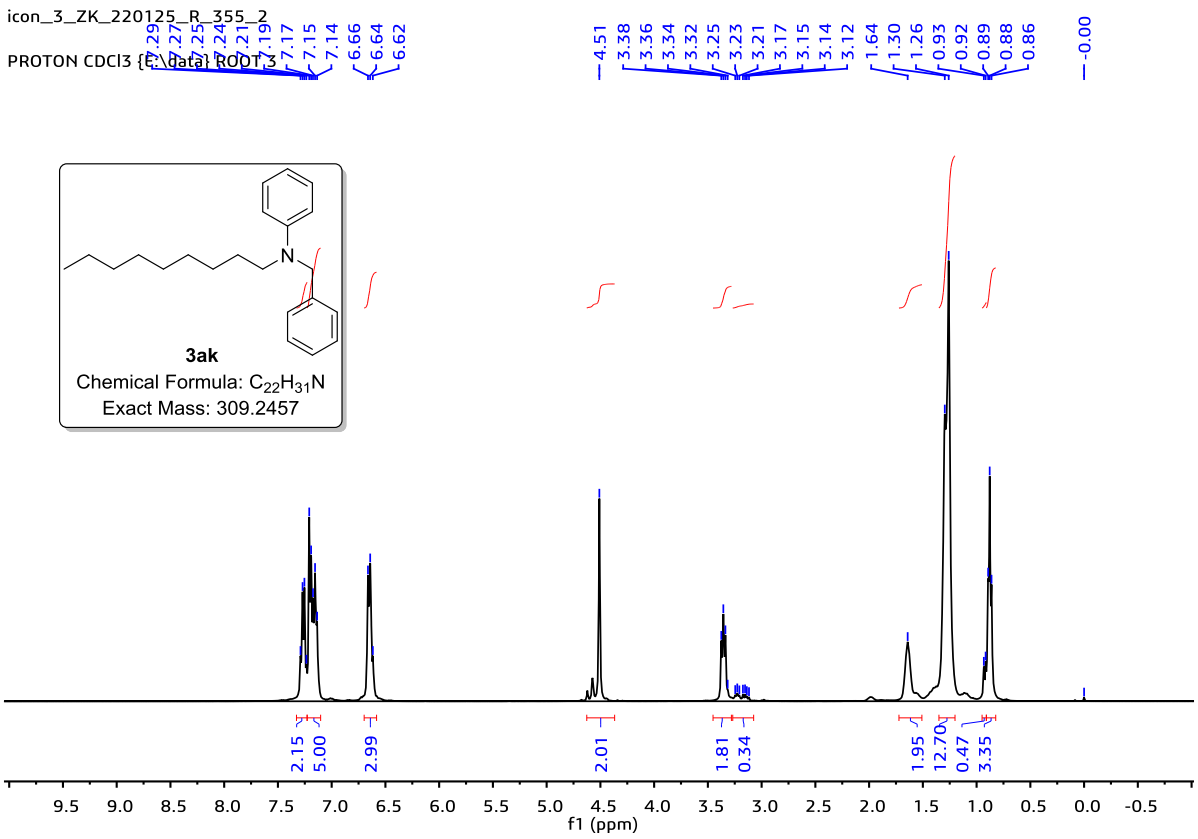

Jan26-2022-OSSO_cJ
ZK-X220126-R-354-4-C
156.95 156.85 154.62 154.53 145.15 145.06 145.05
115.81 115.59 113.58 113.50 113.43
77.16 Chloroform-d
51.17 44.82 34.92 33.04 32.02 29.74 29.69 29.60 29.41 27.30 27.07 22.81 18.17 14.24
F
N
H
3ah
Chemical Formula: C15H24FN
Exact Mass: 237.1893
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

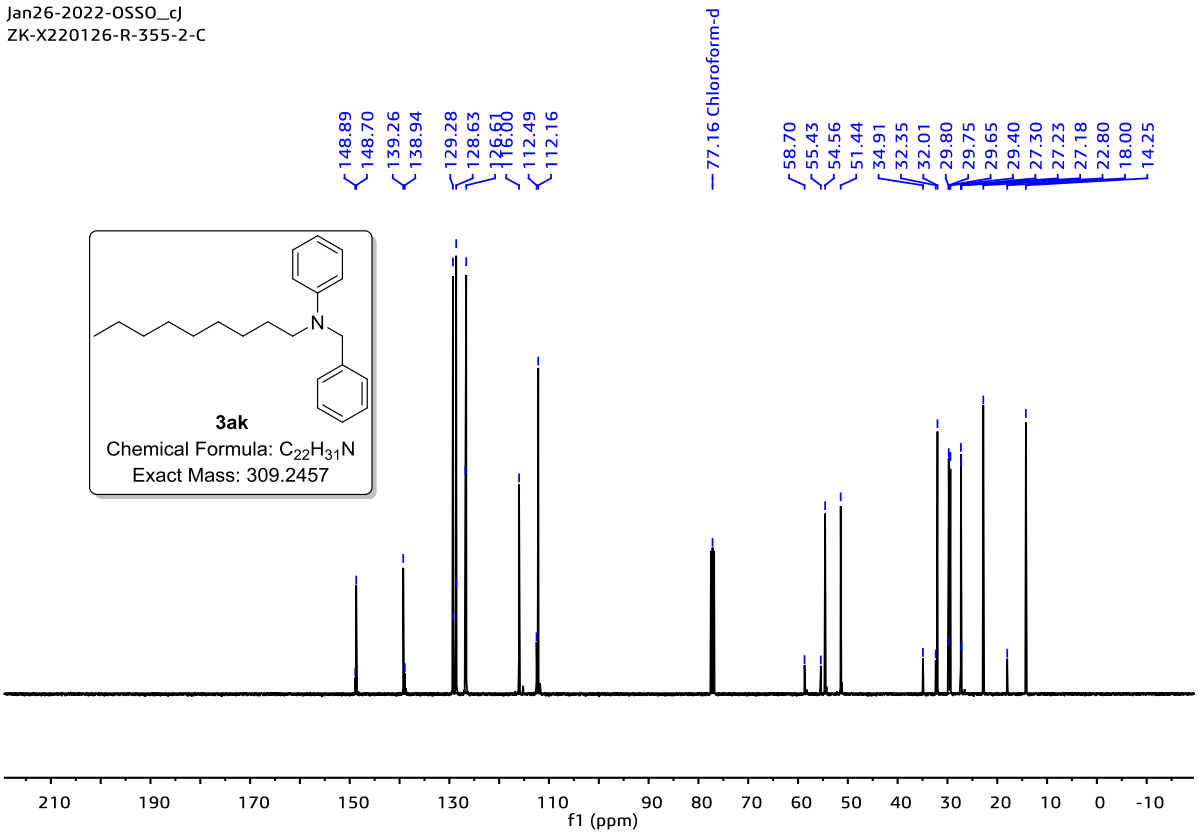

icon_3_ZK_220125_R_354_3
PROTON CDCl3 {E:\data} ROOT 1
7.10
7.07
6.49
6.47
3.59
3.05
3.03
3.01
2.87
2.84
2.82
1.59
1.57
1.55
1.36
1.27
0.95
0.93
0.90
0.88
0.86
0.86
Cl
N
H
3ai
Chemical Formula: C15H24ClN
Exact Mass: 253.1597
1.96
2.00
0.86
1.75
0.28
0.28
1.77
12.16
0.70
3.12
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


Jan26-2022-OSSO_cJ
ZK-X220126-R-354-3-C
147.18
129.08
113.76
77.16 Chloroform-d
50.52
44.19
34.86
32.00
31.98
29.71
29.67
29.56
29.54
29.39
27.25
27.04
22.79
18.10
Cl
N
H
3ai
Chemical Formula: C15H24ClN
Exact Mass: 253.1597
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


icon_3_ZK_220127_R_354_1
PROTON CDCl3 {E:\data} ROOT 16
7.34
7.32
7.30
7.28
7.25
7.24
7.22
7.21
3.55
2.52
2.50
2.49
2.47
2.42
2.41
2.39
1.46
1.25
1.04
1.03
1.01
0.89
0.88
0.86
0.00
3aj
Chemical Formula: C18H31N
Exact Mass: 261.2457
5.00
1.91
1.96
2.24
2.28
14.27
3.16
3.72
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

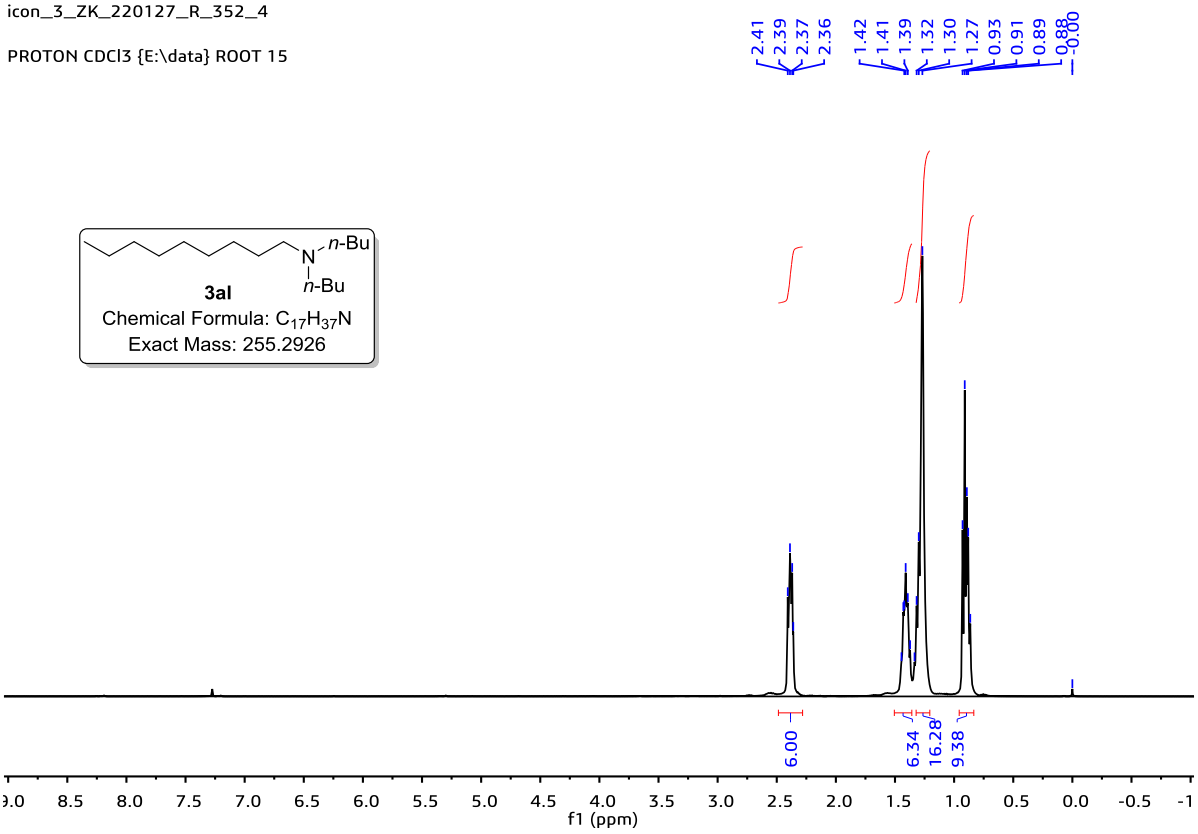

icon_3_ZK_220127_R_354_1
C13CPD CDCl3 {E:\data} ROOT 16
140.27
129.00
128.21
126.74
77.16 Chloroform-d
58.16
53.38
47.37
32.05
29.75
29.46
27.65
27.12
22.84
14.28
11.86
3aj
Chemical Formula: C18H31N
Exact Mass: 261.2457
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


icon_3_ZK_220125_R_355_2
PROTON CDCl3 {E:\data} ROOT 3
7.29
7.27
7.25
7.24
7.21
7.19
7.17
7.15
7.14
6.66
6.64
6.62
4.51
3.38
3.36
3.34
3.32
3.25
3.23
3.21
3.17
3.15
3.14
3.12
1.64
1.30
1.26
0.93
0.92
0.89
0.88
0.86
-0.00
3ak
Chemical Formula: C22H31N
Exact Mass: 309.2457
2.15
5.00
2.99
2.01
1.81
0.34
1.95
12.70
0.47
3.35
f1 (ppm)


Jan26-2022-OSSO_cJ
ZK-X220126-R-355-2-C
148.89
148.70
139.26
138.94
129.28
128.63
116.61
116.00
112.49
112.16
77.16 Chloroform-d
58.70
55.43
54.56
51.44
34.91
32.35
32.01
29.80
29.75
29.65
29.40
27.30
27.23
27.18
22.80
18.00
14.25
3ak
Chemical Formula: C22H31N
Exact Mass: 309.2457
f1 (ppm)

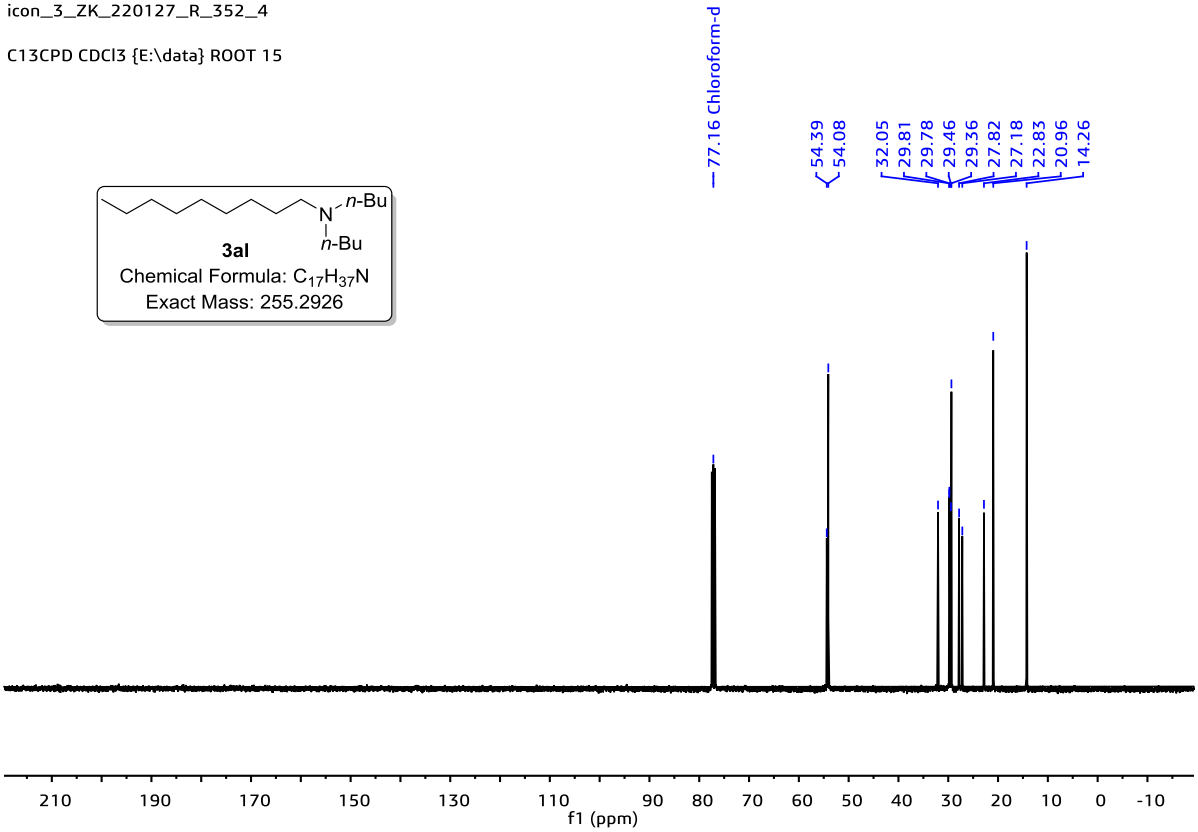
icon_3_ZK_220127_R_352_4

PROTON CDCl3 {E:\data} ROOT 15



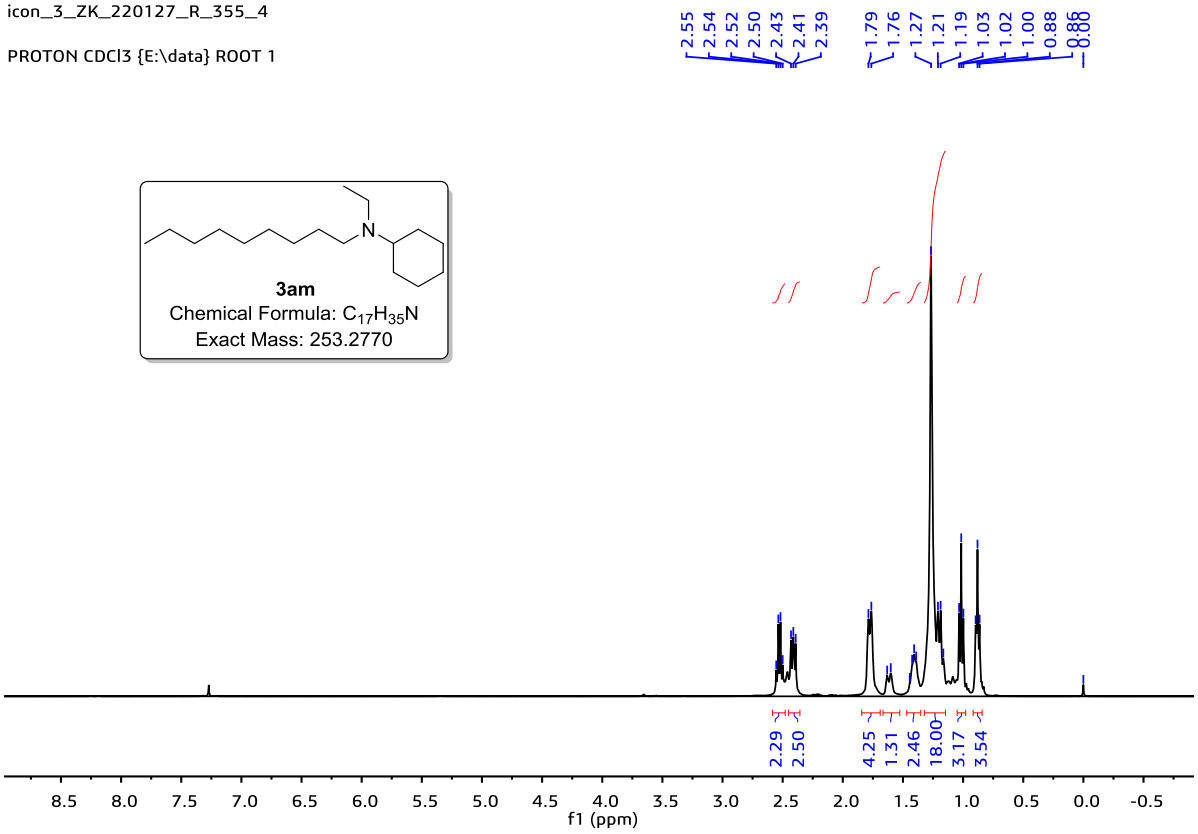
icon_3_ZK_220127_R_352_4

C13CPD CDCl3 {E:\data} ROOT 15



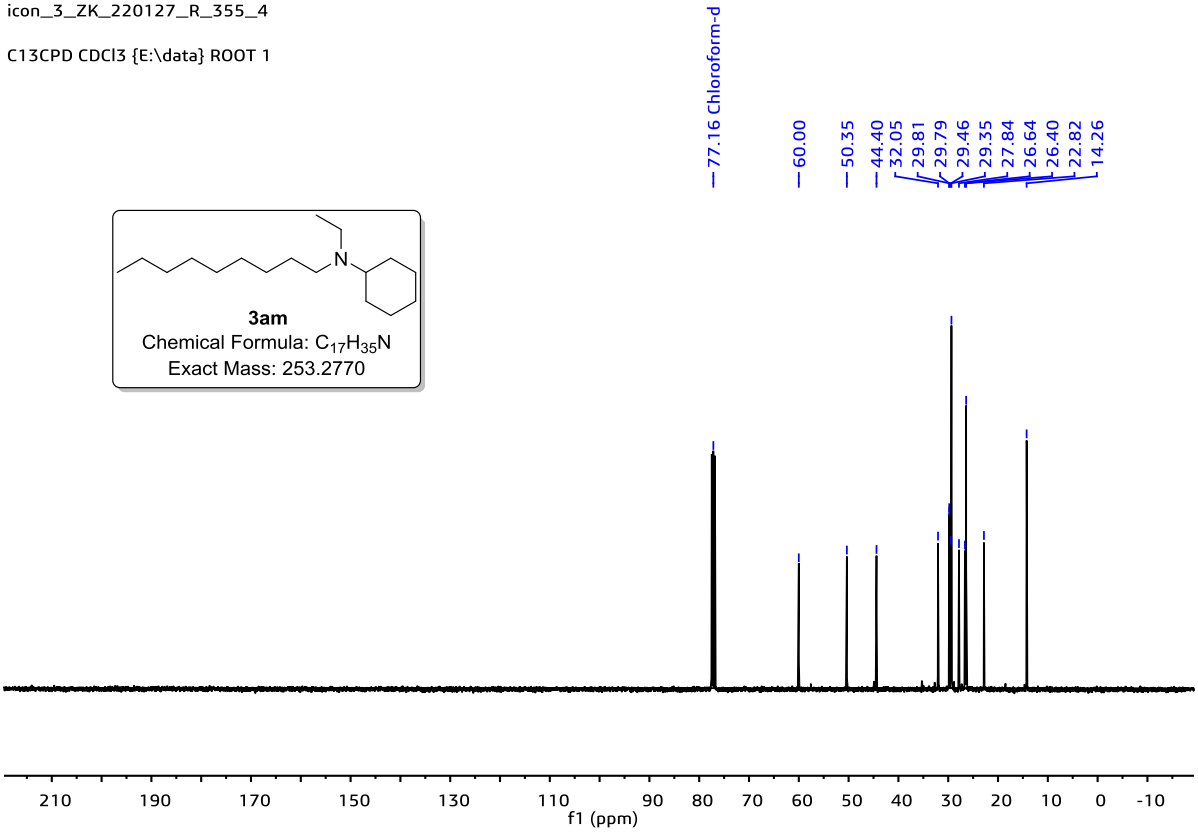
icon_3_ZK_220127_R_355_4

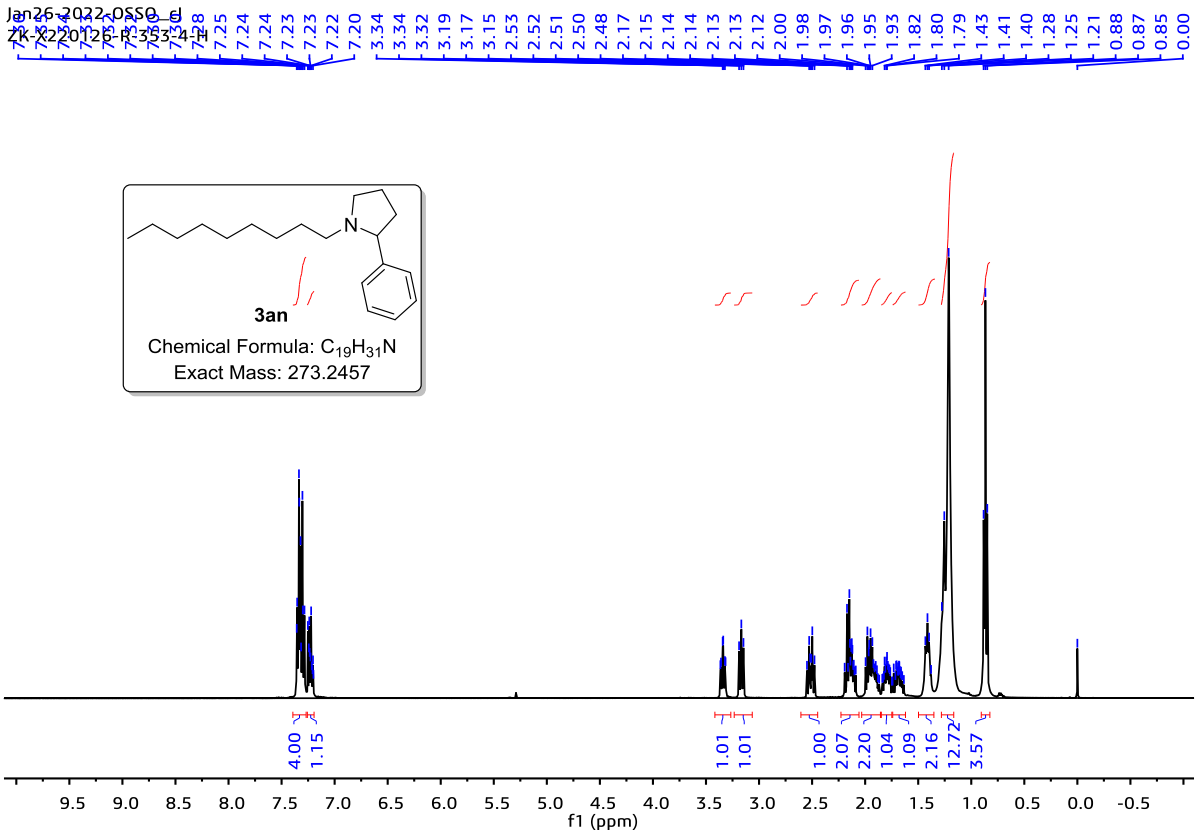
PROTON CDCl3 {E:\data} ROOT 1

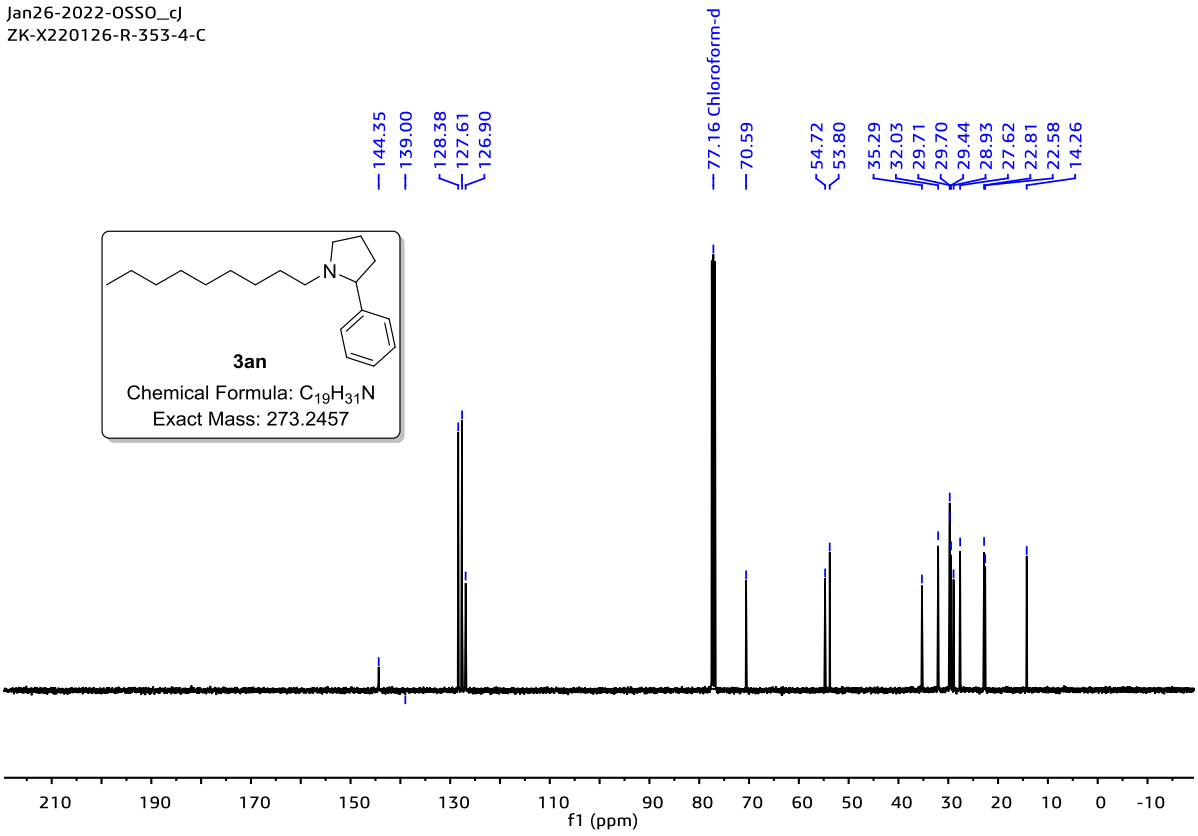


icon_3_ZK_220127_R_355_4

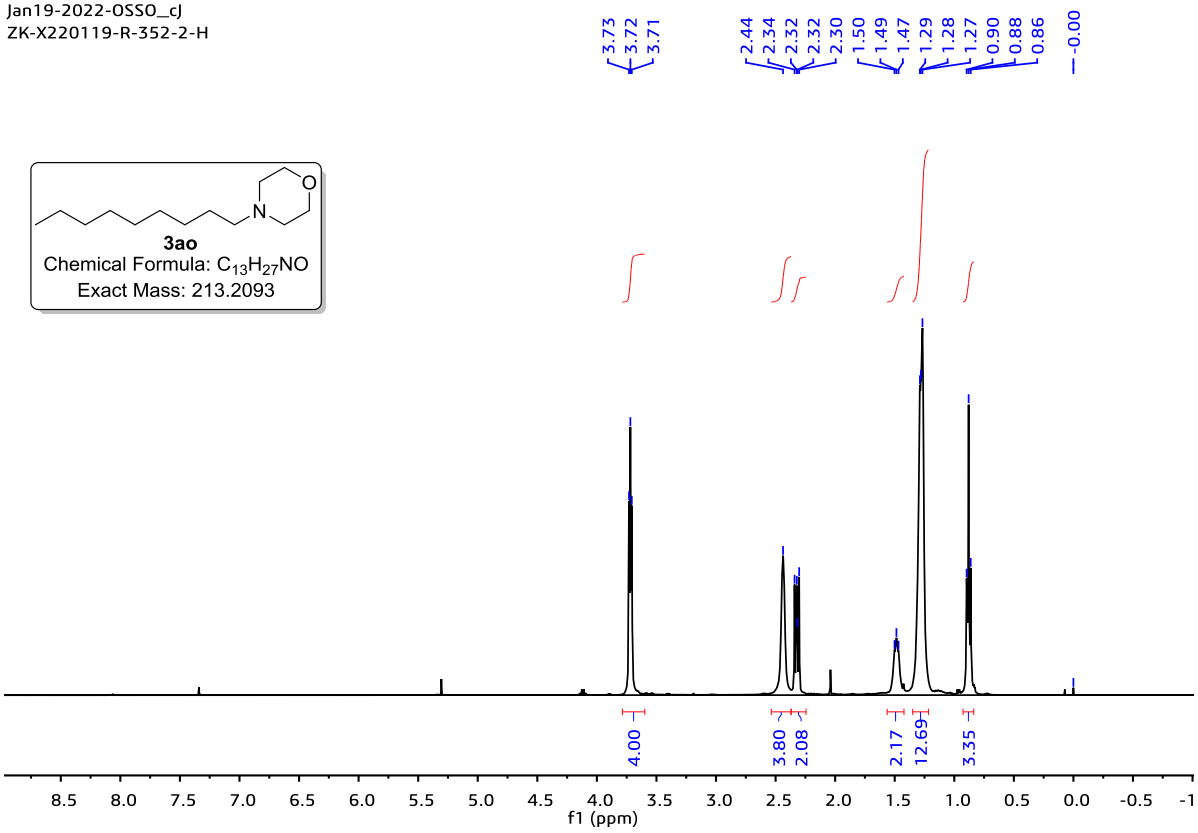
C13CPD CDCl3 {E:\data} ROOT 1




Jan26-2022-OSSO_cJ
ZK-X220126-R-353-4-H
3an
Chemical Formula: C19H31N
Exact Mass: 273.2457
f1 (ppm)

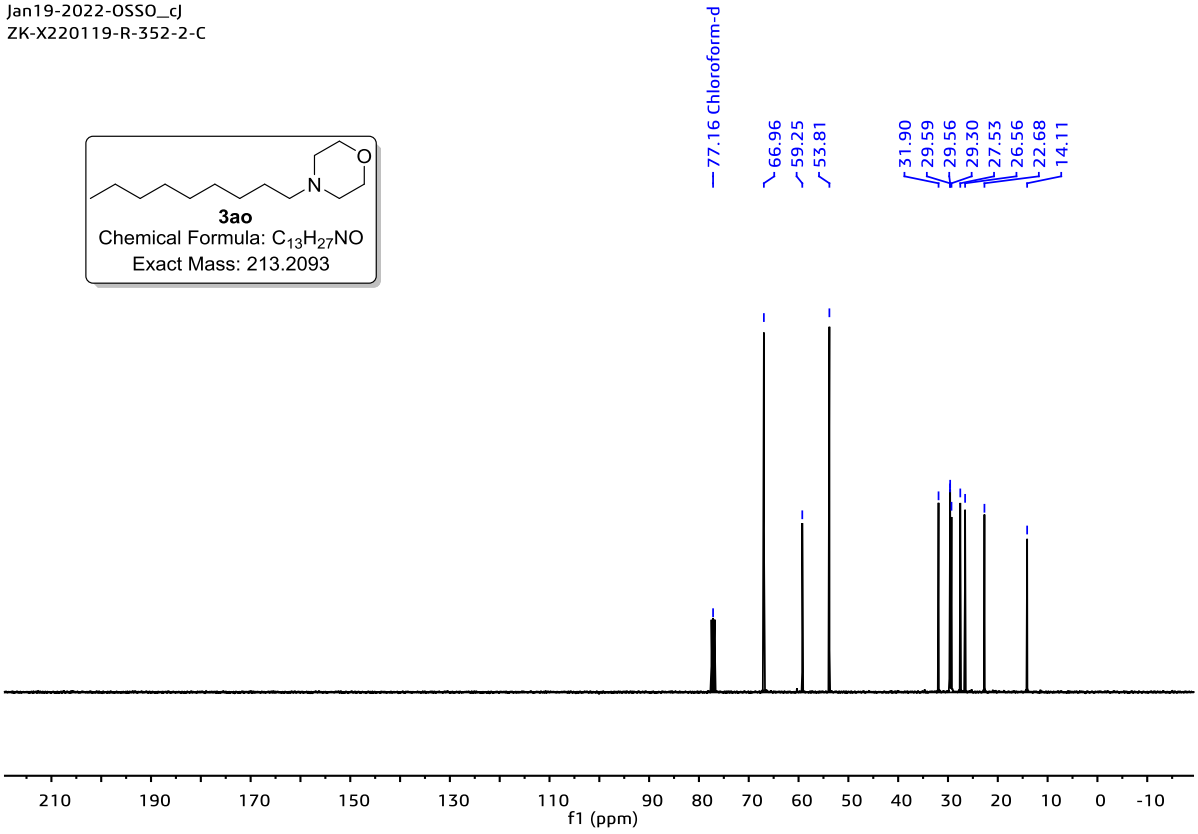

Jan26-2022-OSSO_cJ
ZK-X220126-R-353-4-C
77.16 Chloroform-d
3an
Chemical Formula: C19H31N
Exact Mass: 273.2457
f1 (ppm)

Jan19-2022-OSSO_cJ
ZK-X220119-R-352-2-H

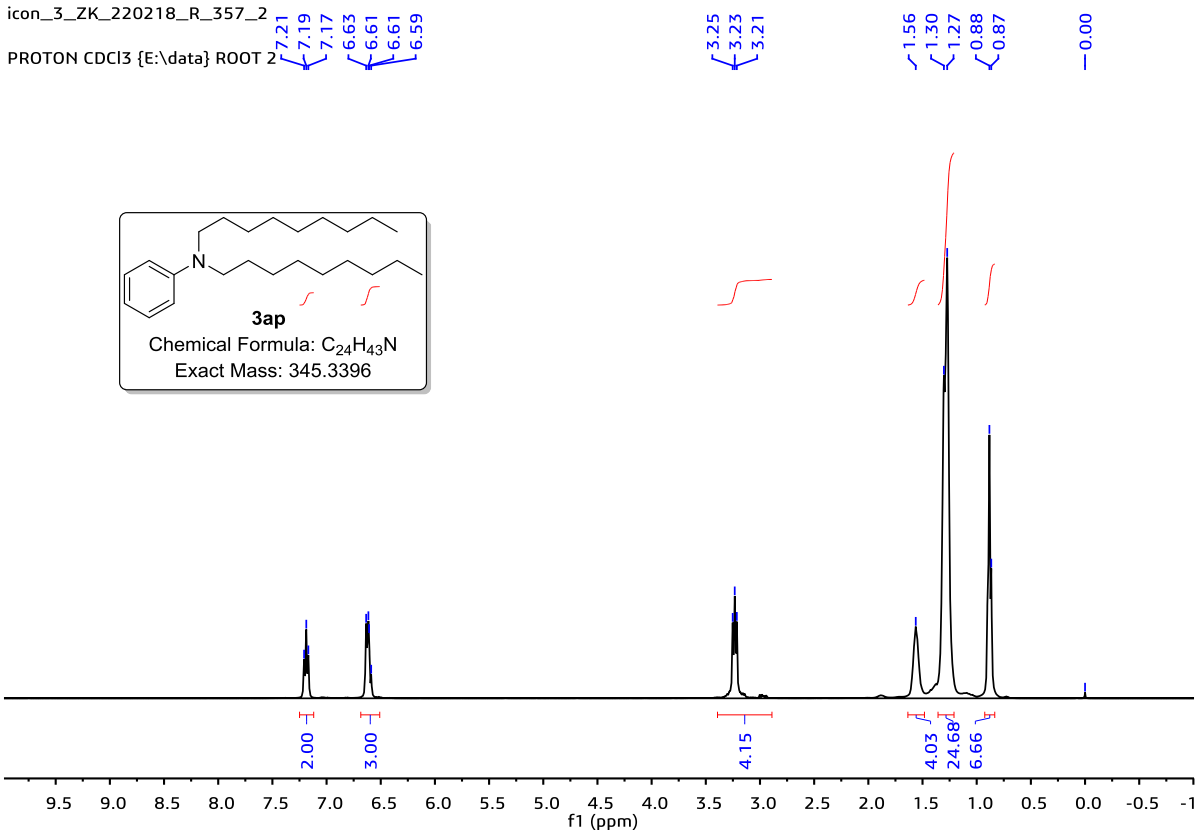


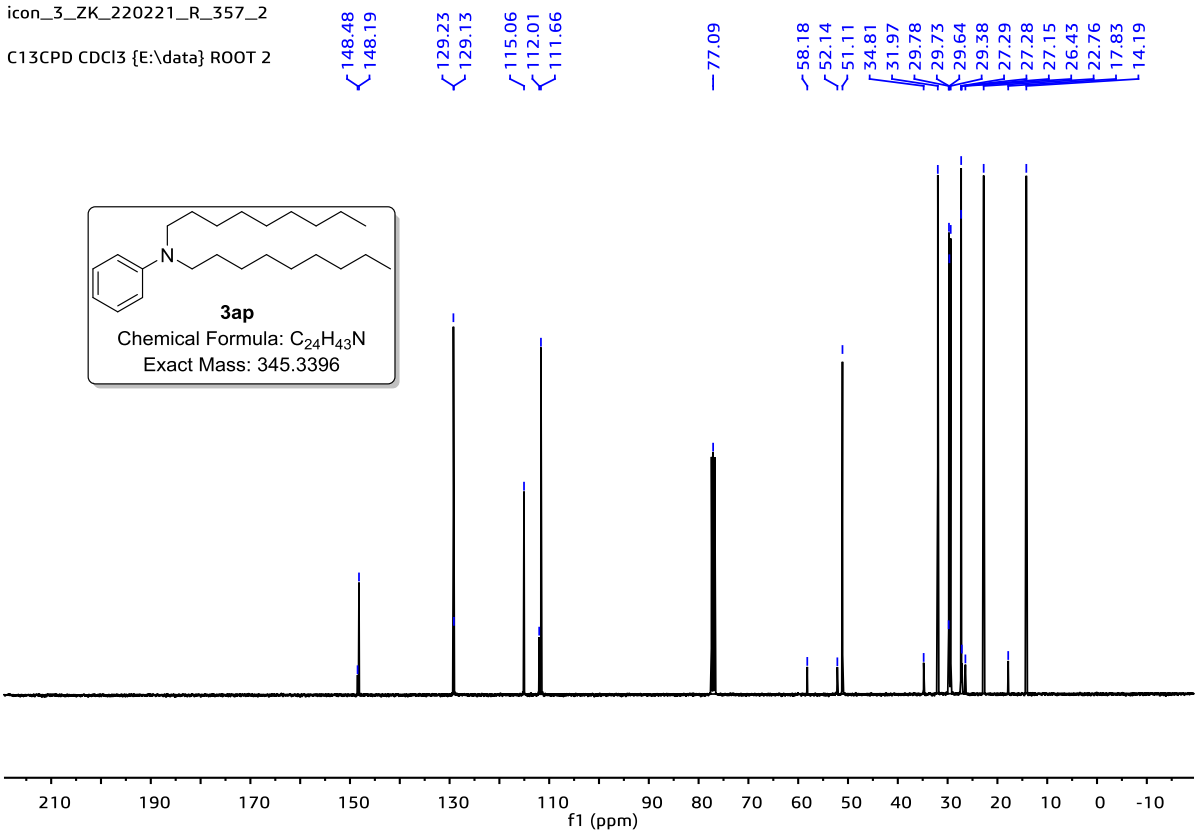
3ao
Chemical Formula: $C_{13}H_{27}NO$
Exact Mass: 213.2093

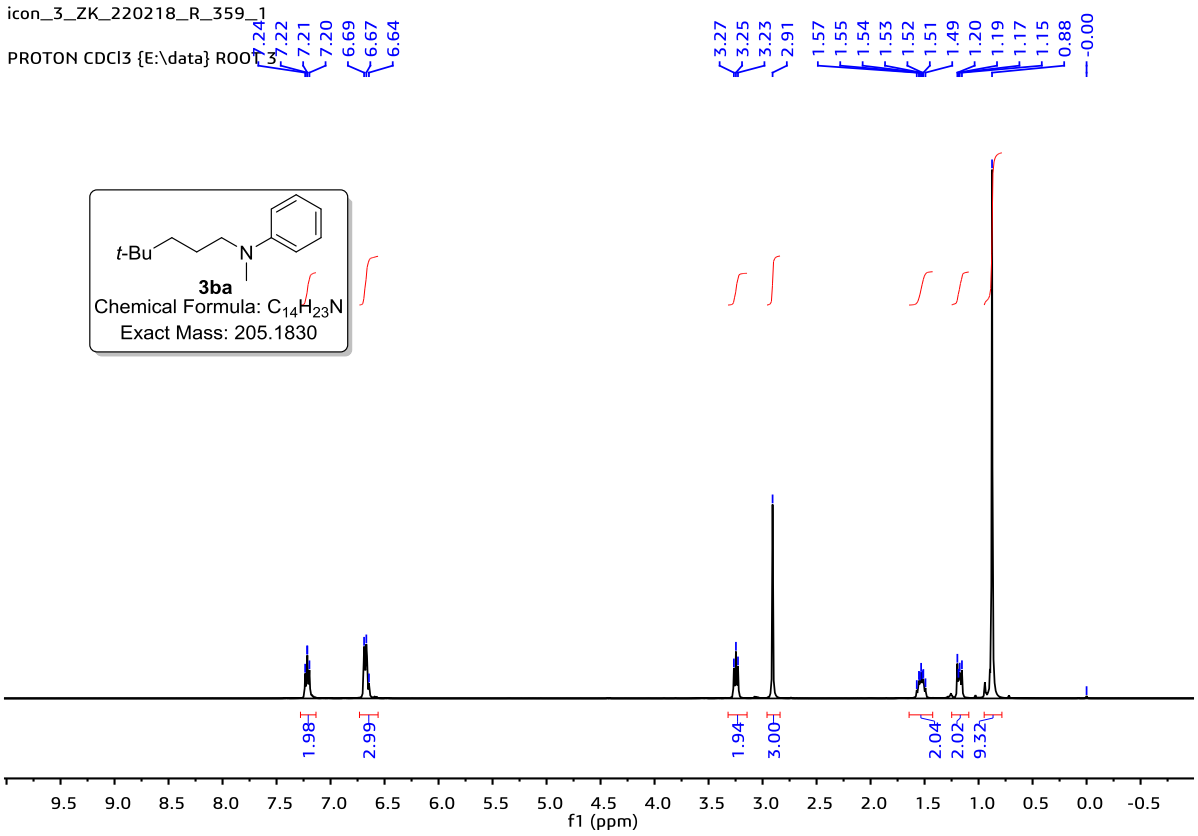
Jan19-2022-OSSO_cJ
ZK-X220119-R-352-2-C

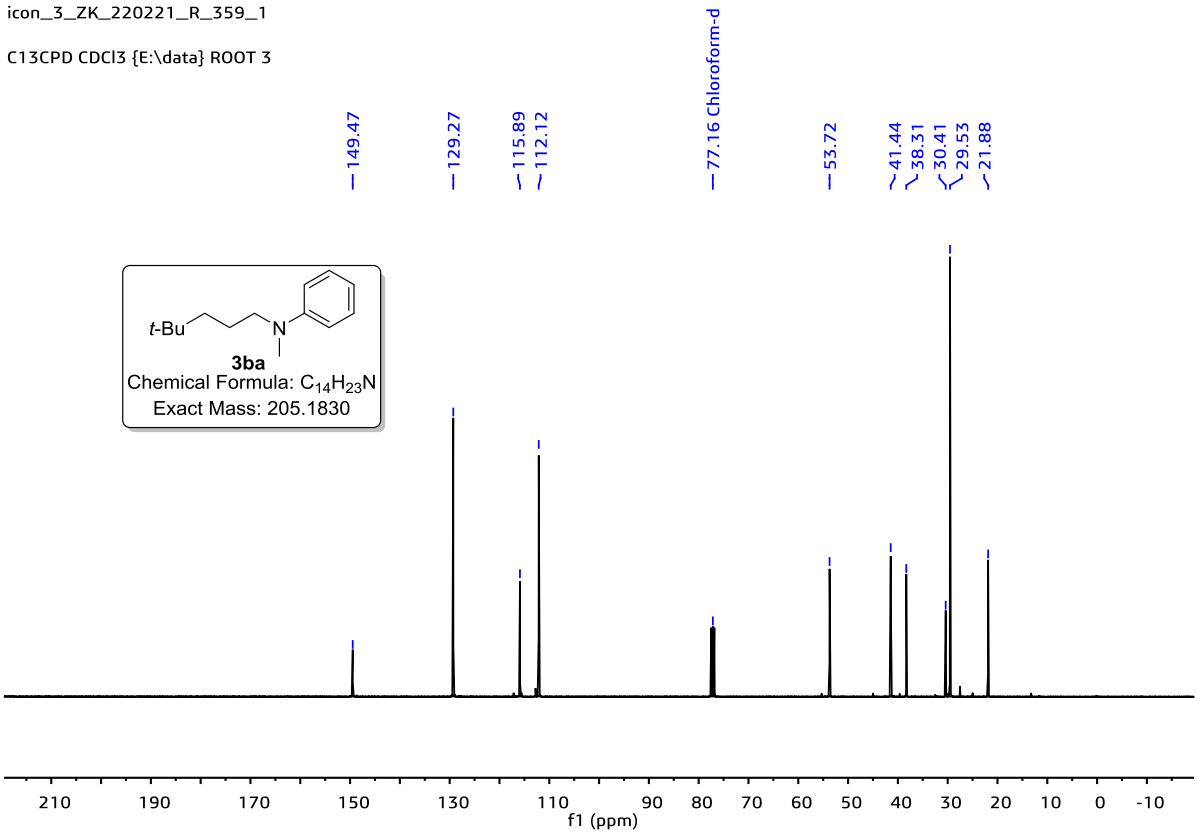


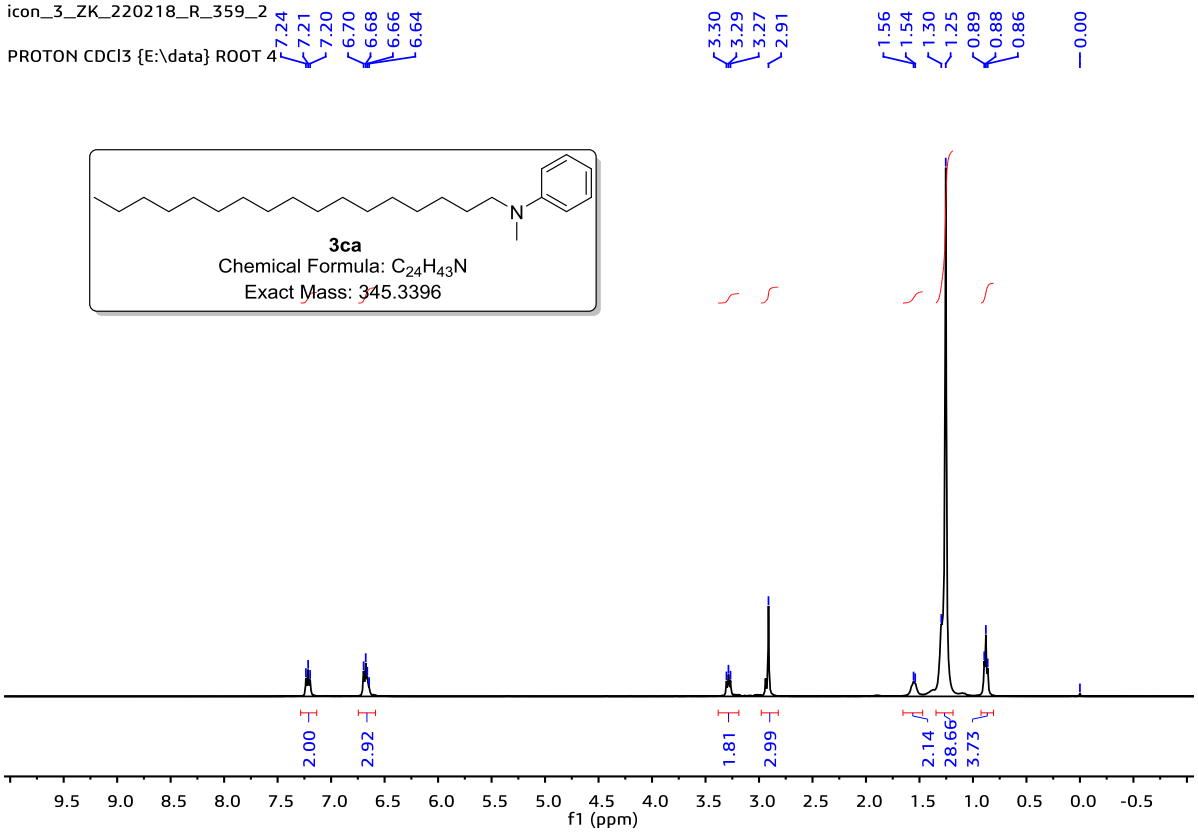
3ao
Chemical Formula: $C_{13}H_{27}NO$
Exact Mass: 213.2093

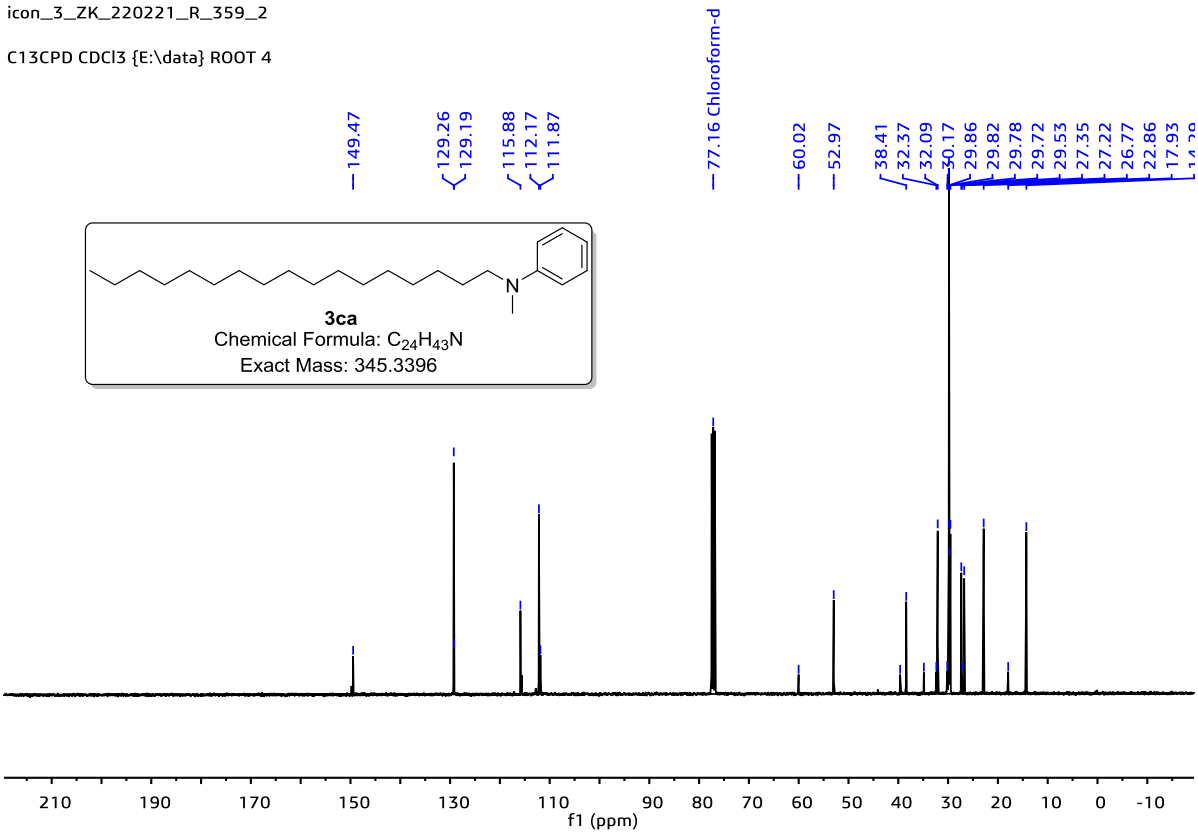

icon_3_ZK_220218_R_357_2
PROTON CDCl3 {E:\data} ROOT 2
7.21
7.19
7.17
6.63
6.61
6.61
6.59
3.25
3.23
3.21
1.56
1.30
1.27
0.88
0.87
0.00
3ap
Chemical Formula: C24H43N
Exact Mass: 345.3396
2.00
3.00
4.15
4.03
24.68
6.66
f1 (ppm)

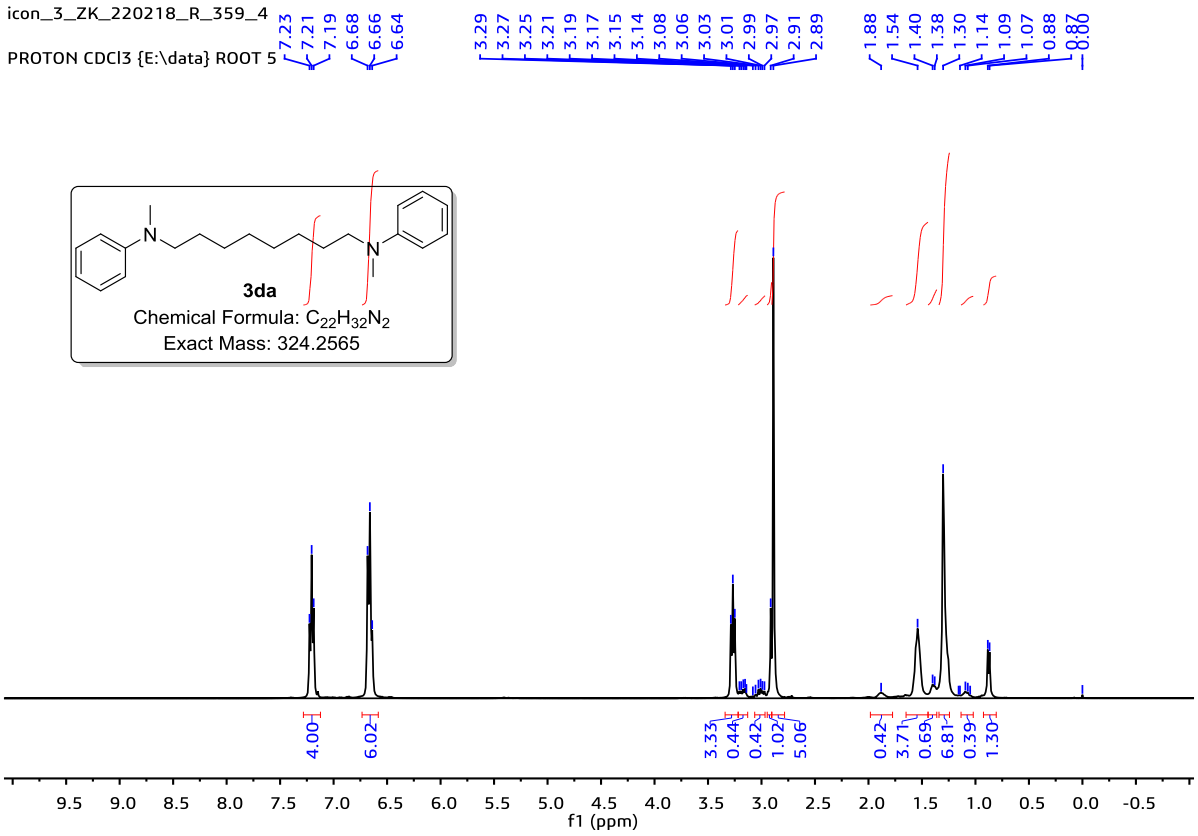

icon_3_ZK_220221_R_357_2
C13CPD CDCl3 {E:\data} ROOT 2
148.48
148.19
129.23
129.13
115.06
112.01
111.66
77.09
58.18
52.14
51.11
34.81
31.97
29.78
29.73
29.64
29.38
27.29
27.28
27.15
26.43
22.76
17.83
14.19
3ap
Chemical Formula: C24H43N
Exact Mass: 345.3396
f1 (ppm)

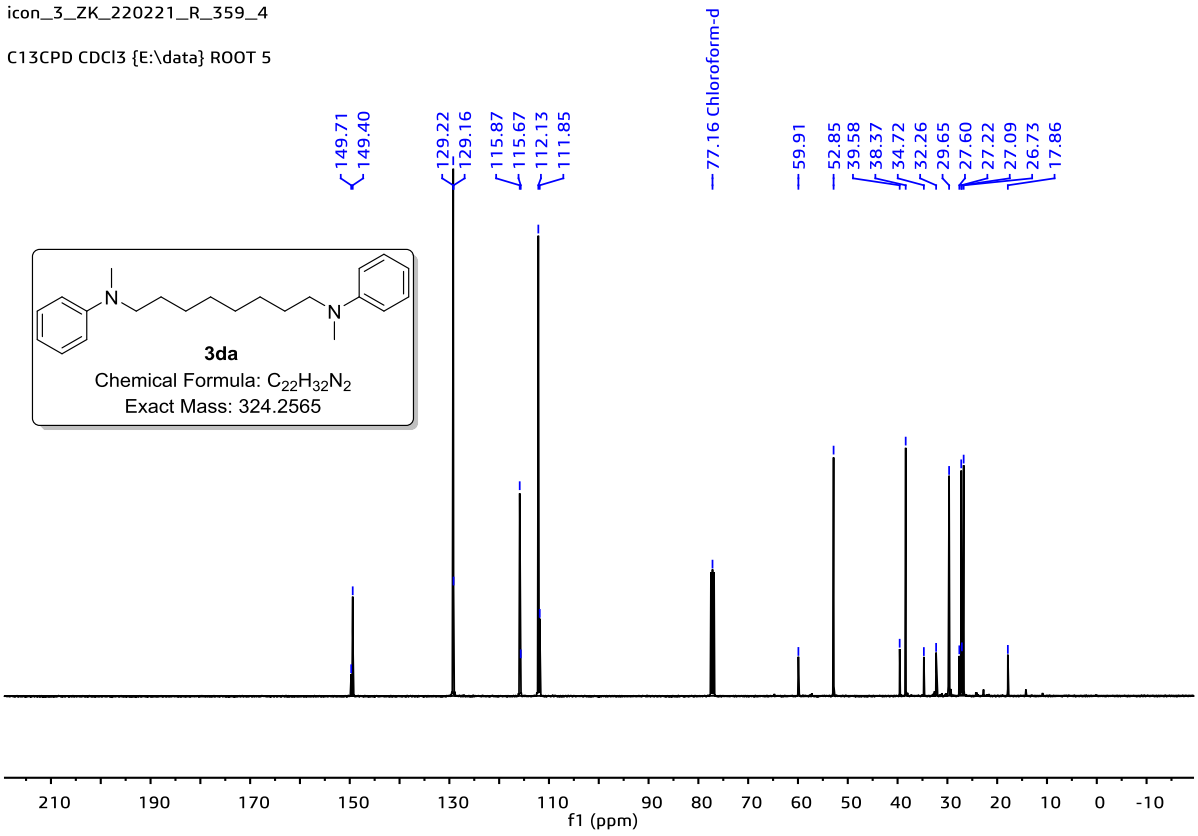

icon_3_ZK_220218_R_359_1
PROTON CDCl3 {E:\data} ROOT 3
7.24
7.22
7.21
7.20
6.69
6.67
6.64
3.27
3.25
3.23
2.91
1.57
1.55
1.54
1.53
1.52
1.51
1.49
1.20
1.19
1.17
1.15
0.88
-0.00
t-Bu
N
3ba
Chemical Formula: C14H23N
Exact Mass: 205.1830
1.98
2.99
1.94
3.00
2.04
2.02
9.32
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

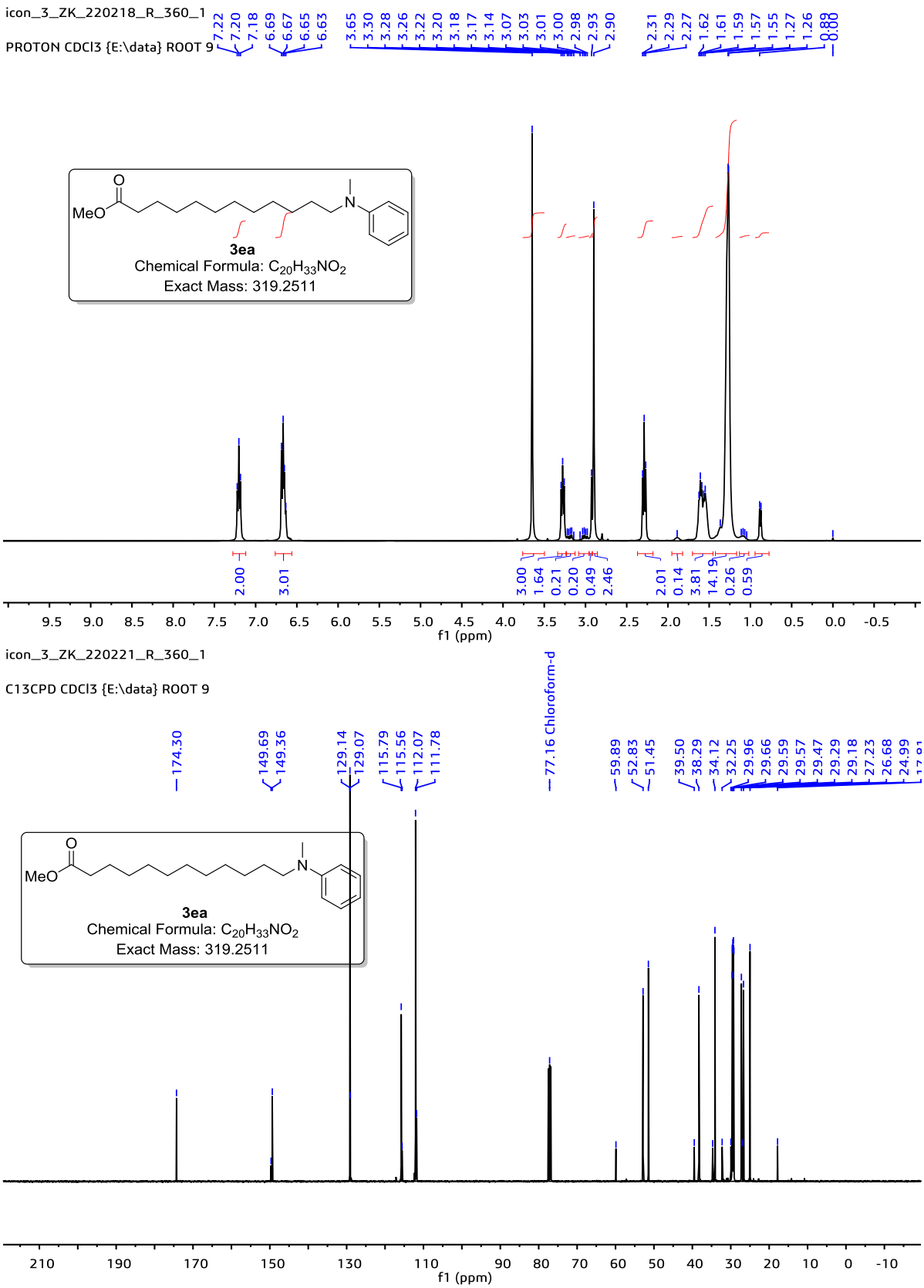

icon_3_ZK_220221_R_359_1
C13CPD CDCl3 {E:\data} ROOT 3
149.47
129.27
115.89
112.12
77.16 Chloroform-d
53.72
41.44
38.31
30.41
29.53
21.88
t-Bu
N
3ba
Chemical Formula: C14H23N
Exact Mass: 205.1830
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

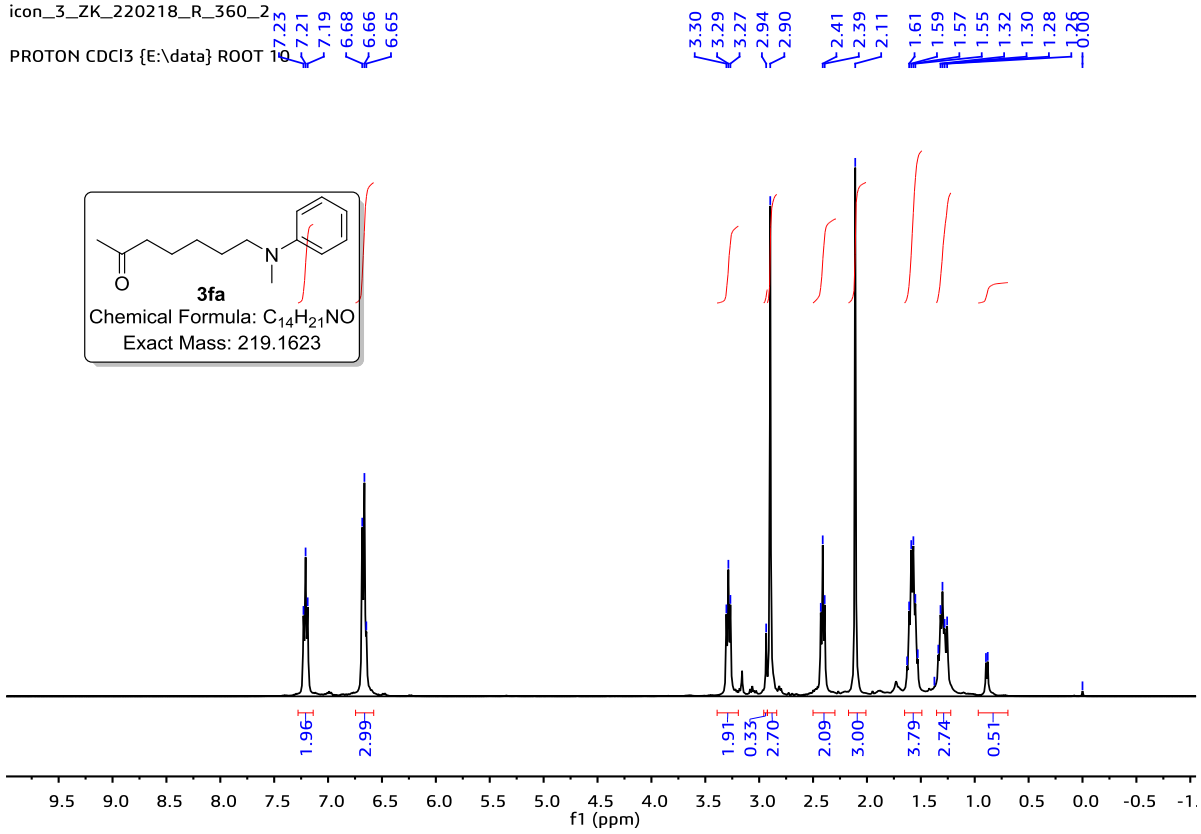

icon_3_ZK_220218_R_359_2
PROTON CDCl3 {E:\data} ROOT 4
7.24
7.21
7.20
6.70
6.68
6.66
6.64
3.30
3.29
3.27
2.91
1.56
1.54
1.30
1.25
0.89
0.88
0.86
0.00
3ca
Chemical Formula: C24H43N
Exact Mass: 345.3396
2.00
2.92
1.81
2.99
2.14
28.66
3.73
f1 (ppm)

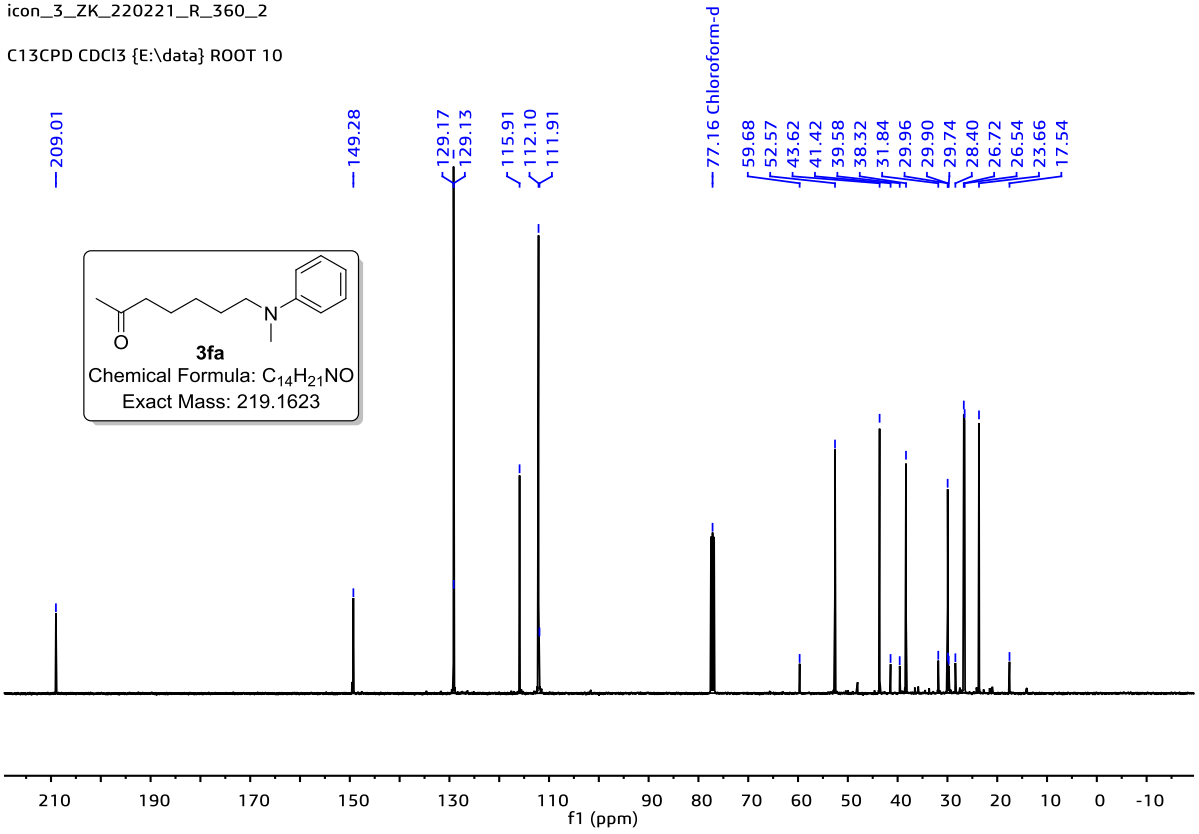

icon_3_ZK_220221_R_359_2
C13CPD CDCl3 {E:\data} ROOT 4
149.47
129.26
129.19
115.88
112.17
111.87
77.16 Chloroform-d
60.02
52.97
38.41
32.37
32.09
30.17
29.86
29.82
29.78
29.72
29.53
27.35
27.22
26.77
22.86
17.93
3ca
Chemical Formula: C24H43N
Exact Mass: 345.3396
f1 (ppm)

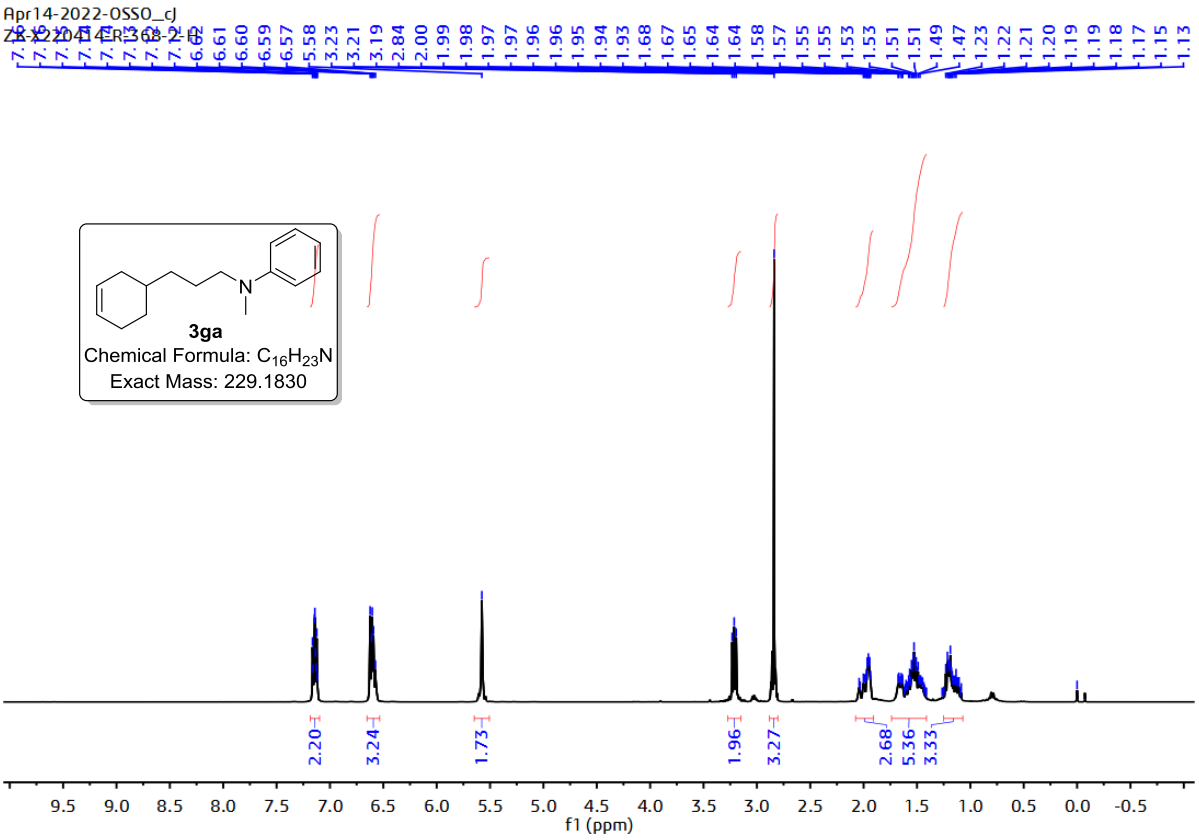

icon_3_ZK_220218_R_359_4
PROTON CDCl3 {E:\data} ROOT 5
7.23
7.21
7.19
6.68
6.66
6.64
3.29
3.27
3.25
3.21
3.19
3.17
3.15
3.14
3.08
3.06
3.03
3.01
2.99
2.97
2.91
2.89
1.88
1.54
1.40
1.38
1.30
1.14
1.09
1.07
0.88
0.87
0.00
3da
Chemical Formula: C22H32N2
Exact Mass: 324.2565
4.00
6.02
3.33
0.44
0.42
1.02
5.06
0.42
3.71
0.69
6.81
0.39
1.30
f1 (ppm)

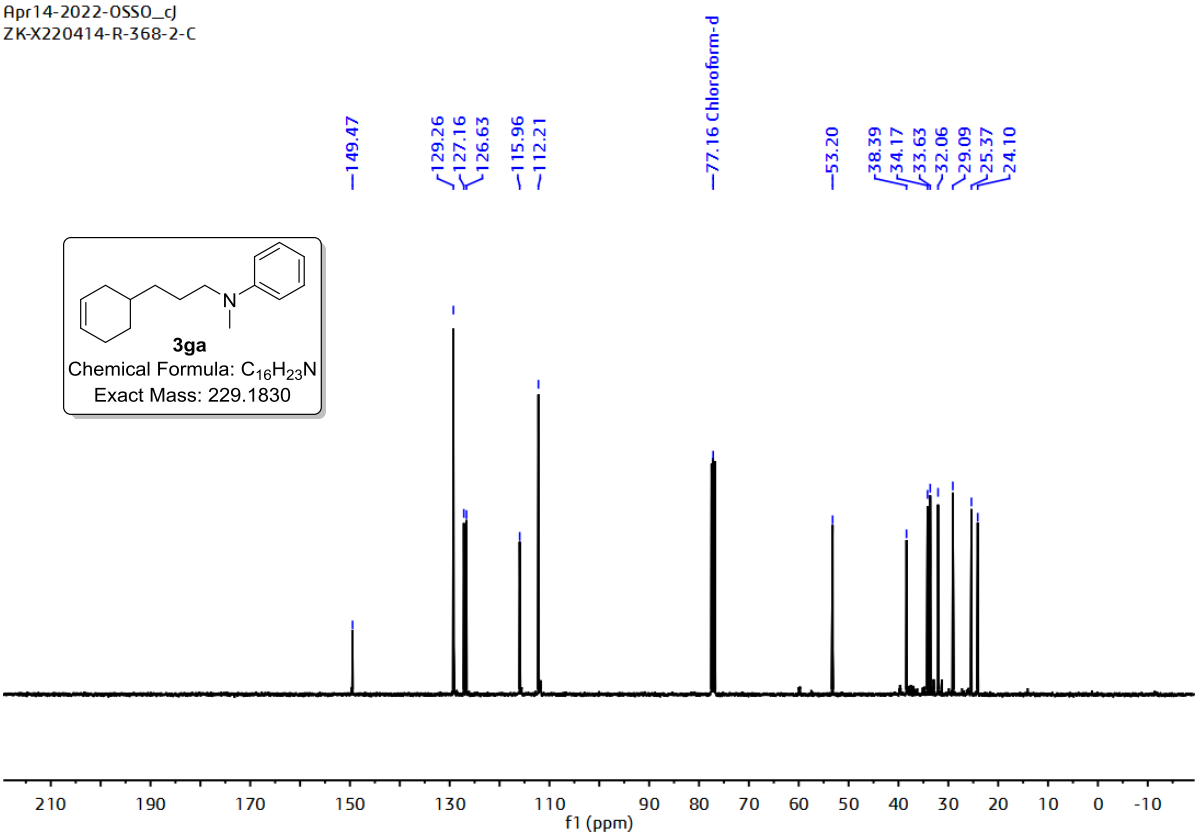

icon_3_ZK_220221_R_359_4
C13CPD CDCl3 {E:\data} ROOT 5
149.71
149.40
129.22
129.16
115.87
115.67
112.13
111.85
77.16 Chloroform-d
59.91
52.85
39.58
38.37
34.72
32.26
29.65
27.60
27.22
27.09
26.73
17.86
3da
Chemical Formula: C22H32N2
Exact Mass: 324.2565
f1 (ppm)

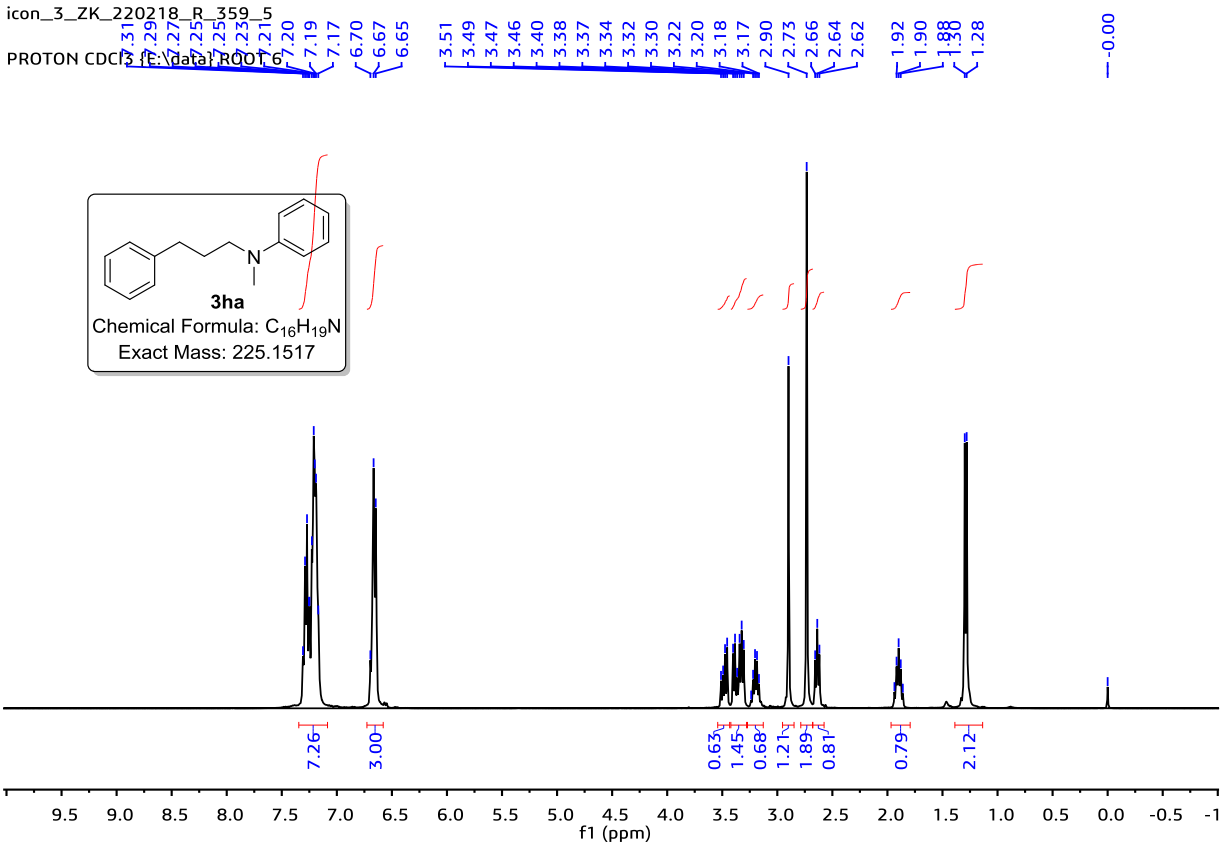

icon_3_ZK_220218_R_360_1
PROTON CDCl3 {E:\data} ROOT 9
7.22 7.20 7.18 6.69 6.67 6.65 6.63
3.65 3.30 3.28 3.26 3.22 3.20 3.18 3.17 3.14 3.07 3.03 3.01 3.00 2.98 2.93 2.90
2.31 2.29 2.27 1.62 1.61 1.59 1.57 1.55 1.27 1.26 0.88 0.00
MeO
O
N
3ea
Chemical Formula: C20H33NO2
Exact Mass: 319.2511
2.00 3.01 3.00 1.64 0.21 0.20 0.49 2.46 2.01 0.14 3.81 14.19 0.26 0.59
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
icon_3_ZK_220221_R_360_1
C13CPD CDCl3 {E:\data} ROOT 9
174.30 149.69 149.36 129.14 129.07 115.79 115.56 112.07 111.78
77.16 Chloroform-d
59.89 52.83 51.45 39.50 38.29 34.12 32.25 29.96 29.66 29.59 29.57 29.47 29.29 29.18 27.23 26.68 24.99
MeO
O
N
3ea
Chemical Formula: C20H33NO2
Exact Mass: 319.2511
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

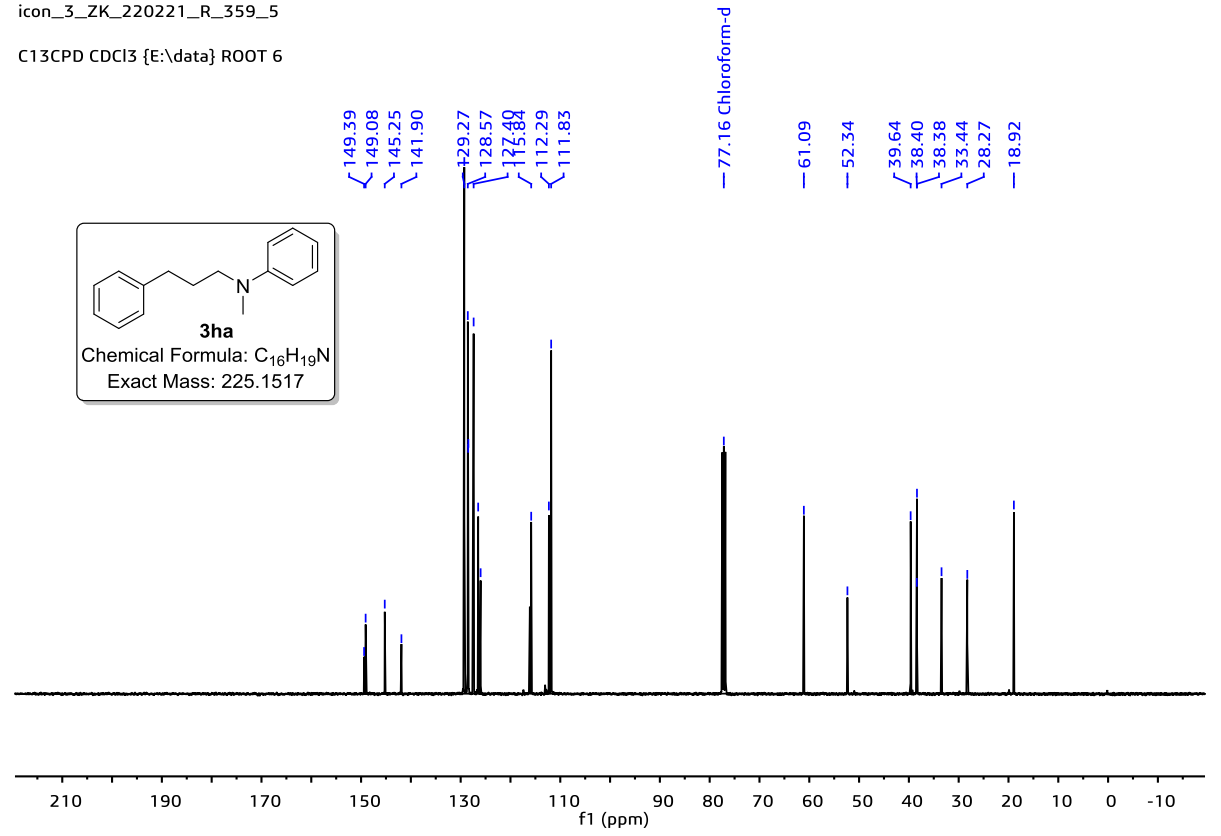

icon_3_ZK_220218_R_360_2
PROTON CDCl3 {E:\data} ROOT 10
7.23
7.21
7.19
6.68
6.66
6.65
3.30
3.29
3.27
2.94
2.90
2.41
2.39
2.11
1.61
1.59
1.57
1.55
1.32
1.30
1.28
1.26
0.00
3fa
Chemical Formula: C14H21NO
Exact Mass: 219.1623
1.96
2.99
1.91
0.33
2.70
2.09
3.00
3.79
2.74
0.51
f1 (ppm)

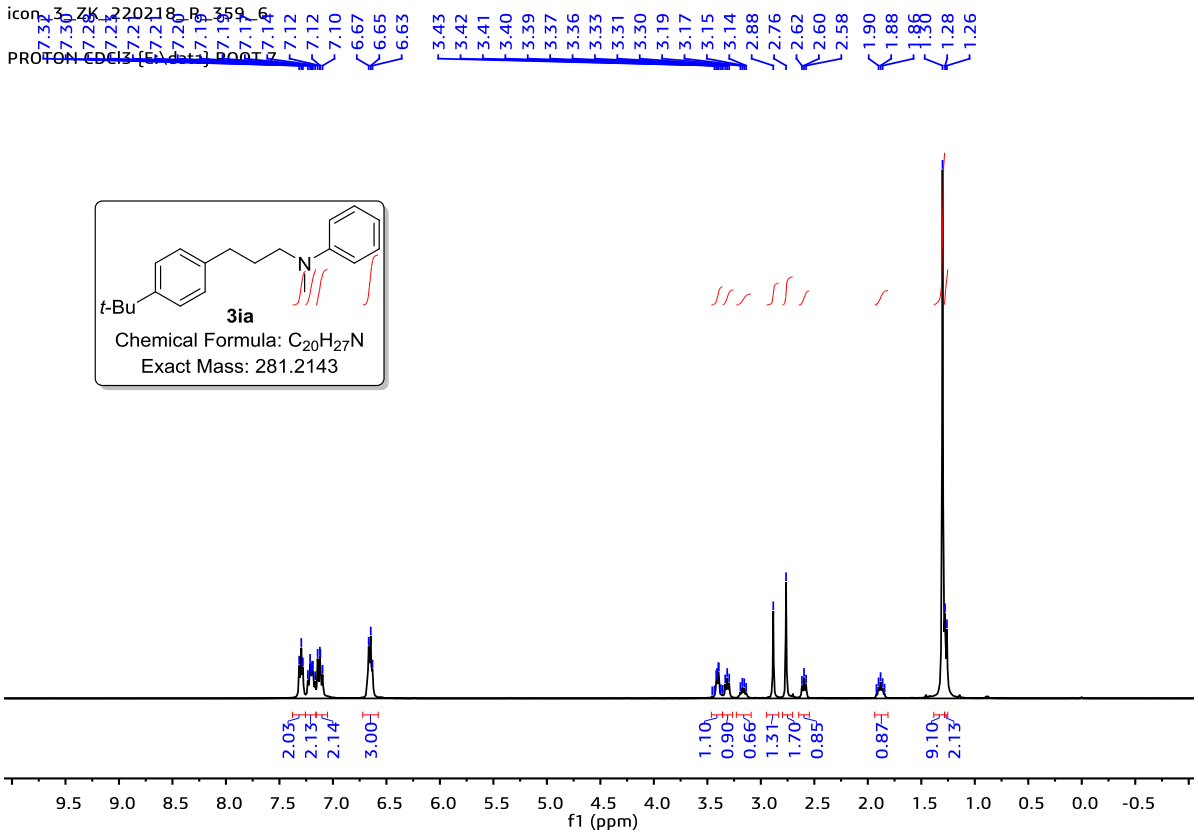

icon_3_ZK_220221_R_360_2
C13CPD CDCl3 {E:\data} ROOT 10
209.01
149.28
129.17
129.13
115.91
112.10
111.91
77.16 Chloroform-d
59.68
52.57
43.62
41.42
39.58
38.32
31.84
29.96
29.90
29.74
28.40
26.72
26.54
23.66
17.54
3fa
Chemical Formula: C14H21NO
Exact Mass: 219.1623
f1 (ppm)

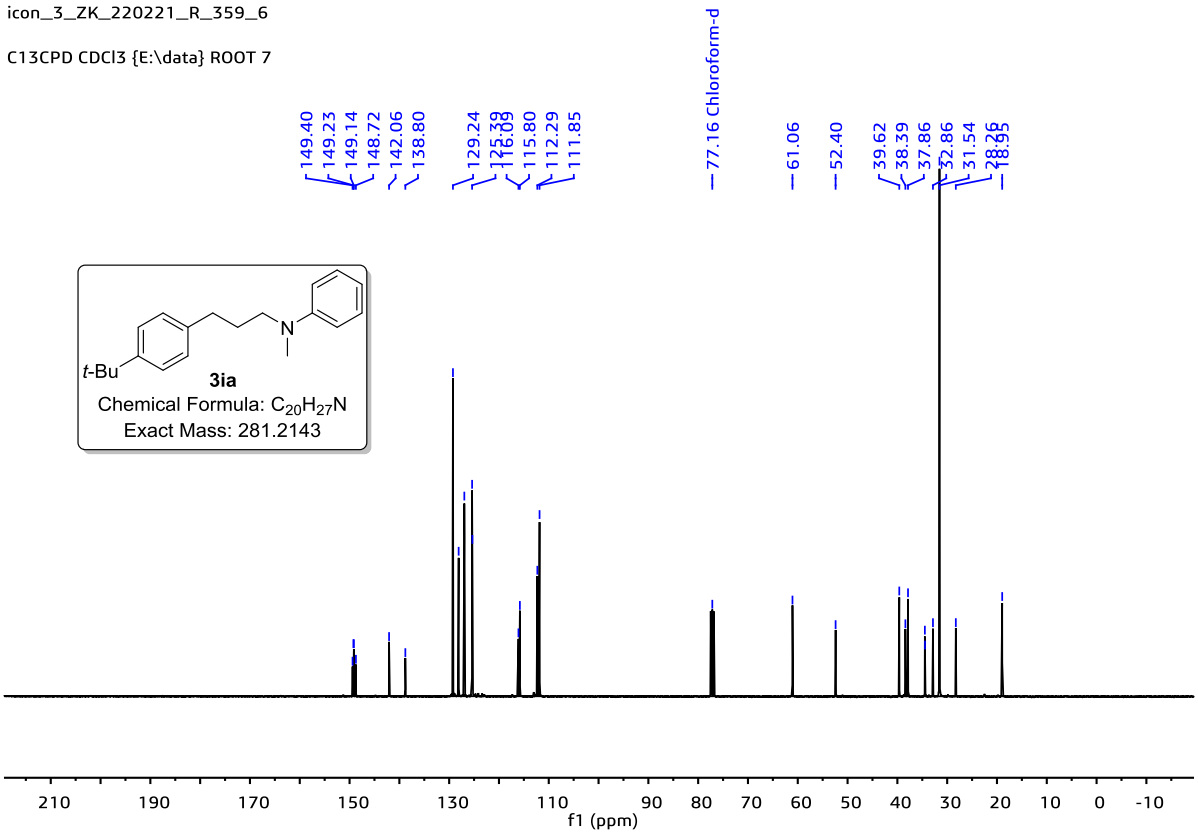

Apr14-2022-OSSO_cJ
ZK-X220414-R-368-2-H
3ga
Chemical Formula: C16H23N
Exact Mass: 229.1830
2.20
3.24
1.73
1.96
3.27
2.68
5.36
3.33
f1 (ppm)

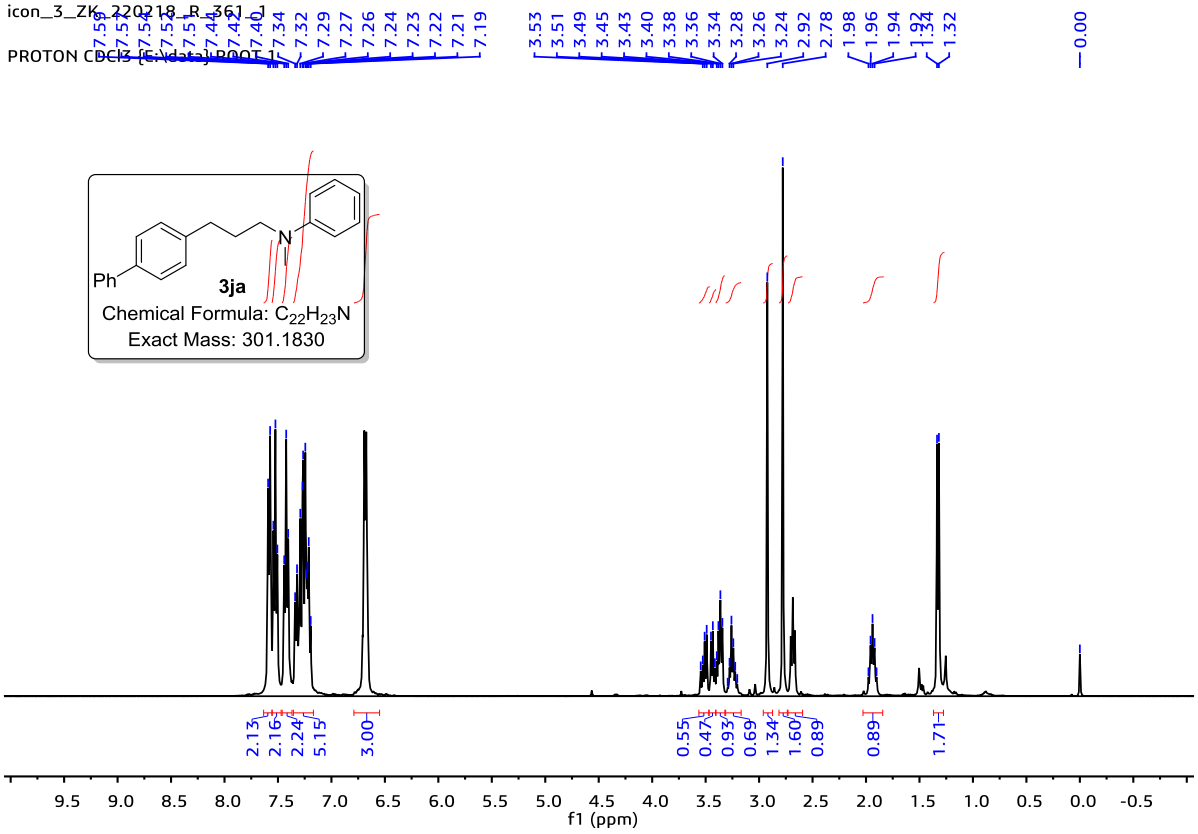

Apr14-2022-OSSO_cJ
ZK-X220414-R-368-2-C
149.47
129.26
127.16
126.63
115.96
112.21
77.16 Chloroform-d
53.20
38.39
34.17
33.63
32.06
29.09
25.37
24.10
3ga
Chemical Formula: C16H23N
Exact Mass: 229.1830
f1 (ppm)

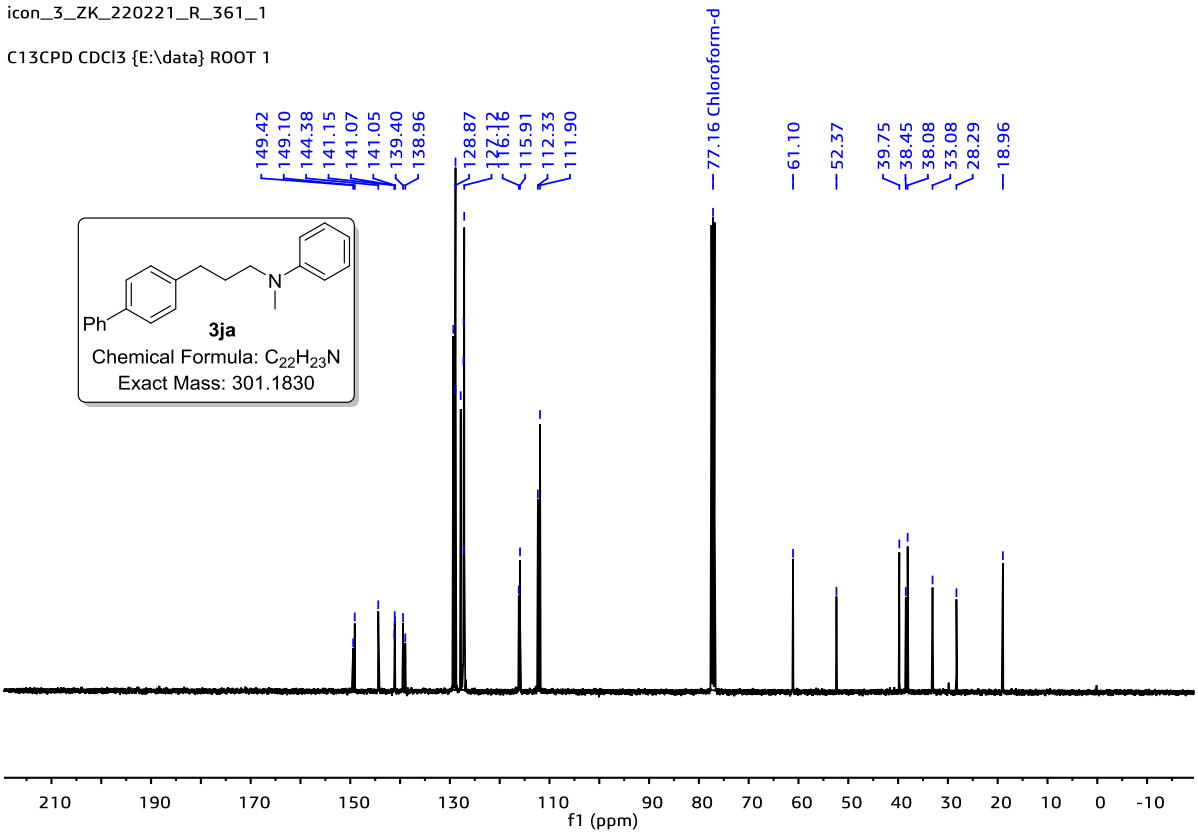

icon_3_ZK_220218_R_359_5
PROTON CDCl3 {E:\data} ROOT 6
3ha
Chemical Formula: C16H19N
Exact Mass: 225.1517
f1 (ppm)

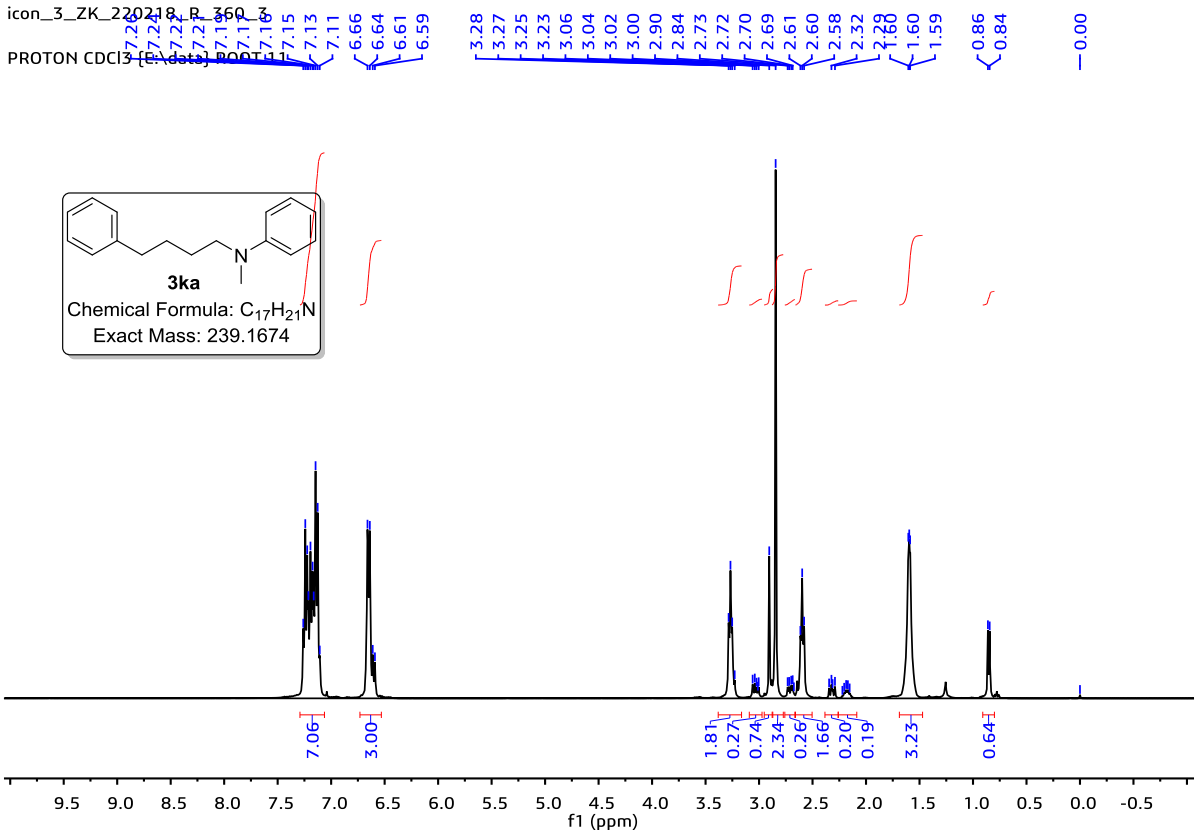

icon_3_ZK_220221_R_359_5
C13CPD CDCl3 {E:\data} ROOT 6
77.16 Chloroform-d
3ha
Chemical Formula: C16H19N
Exact Mass: 225.1517
f1 (ppm)

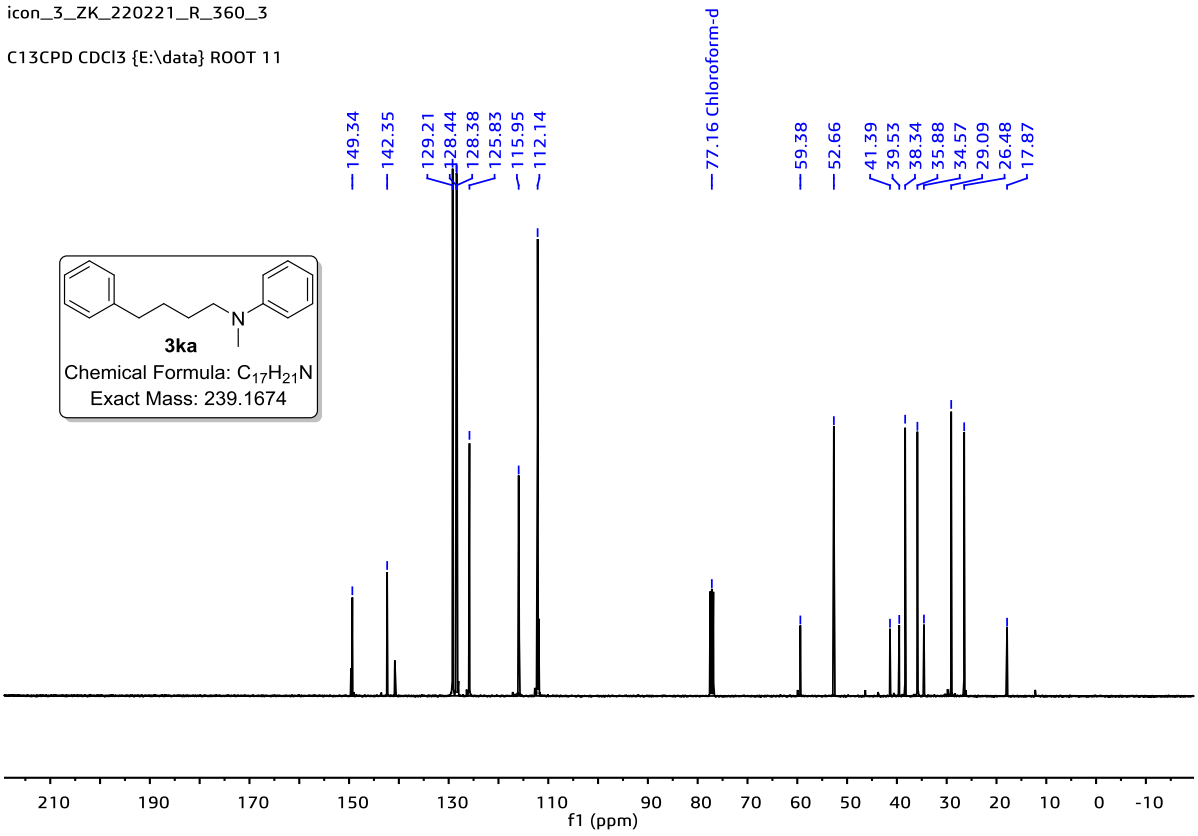

icon_3_ZK_220218_R_359_6
PROTON CDCl3 {E:\data} ROOT 7
7.32 7.30 7.29 7.23 7.22 7.21 7.20 7.19 7.18 7.17 7.14 7.12 7.12 7.10 6.67 6.65 6.63
3.43 3.42 3.41 3.40 3.39 3.37 3.36 3.33 3.31 3.30 3.19 3.17 3.15 3.14 2.88 2.76 2.62 2.60 2.58 1.90 1.88 1.30 1.28 1.26
t-Bu
N
3ia
Chemical Formula: C20H27N
Exact Mass: 281.2143
2.03 2.13 2.14 3.00 1.10 0.90 0.66 1.31 1.70 0.85 0.87 9.10 2.13
f1 (ppm)

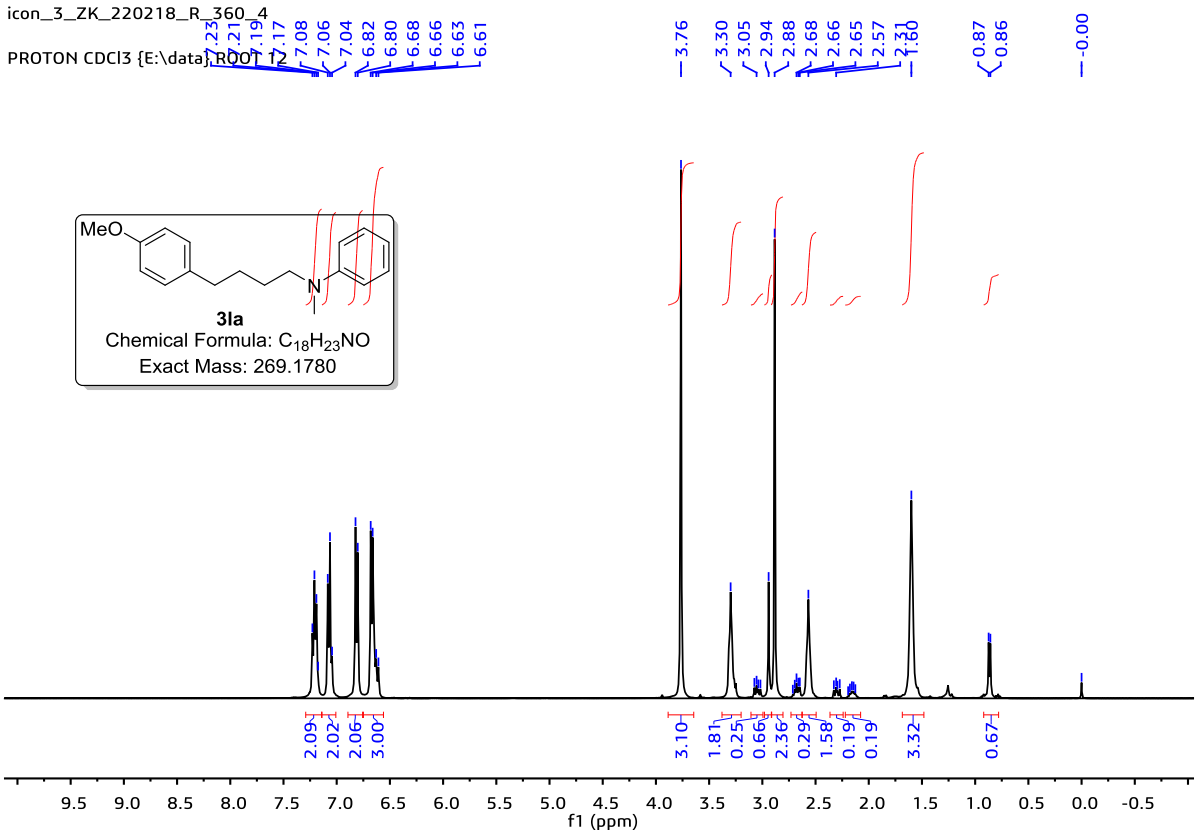

icon_3_ZK_220221_R_359_6
C13CPD CDCl3 {E:\data} ROOT 7
149.40 149.23 149.14 148.72 142.06 138.80 129.24 125.39 116.09 115.80 112.29 111.85 77.16 Chloroform-d 61.06 52.40 39.62 38.39 37.86 32.86 31.54 28.26 18.95
t-Bu
N
3ia
Chemical Formula: C20H27N
Exact Mass: 281.2143
f1 (ppm)

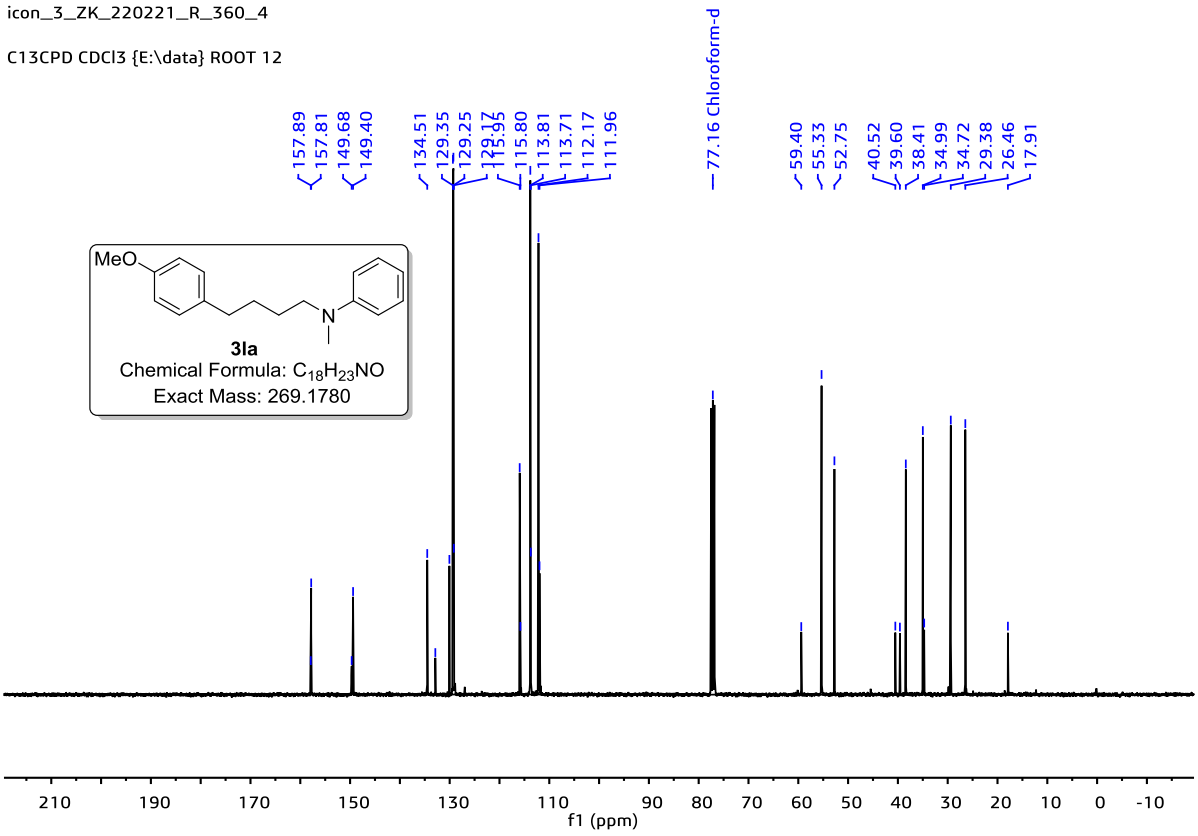

icon_3_ZK_220218_R_361_1
PROTON CDCl3 {E:\data} ROOT 1
7.58 7.57 7.54 7.52 7.51 7.44 7.42 7.40 7.34 7.32 7.29 7.27 7.26 7.24 7.23 7.22 7.21 7.19
3.53 3.51 3.49 3.45 3.43 3.40 3.38 3.36 3.34 3.28 3.26 3.24 2.92 2.78 1.98 1.96 1.94 1.92 1.34 1.32
0.00
Ph
N
3ja
Chemical Formula: C22H23N
Exact Mass: 301.1830
2.13 2.16 2.24 5.15 3.00 0.55 0.47 0.93 0.69 1.34 1.60 0.89 0.89 1.71
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

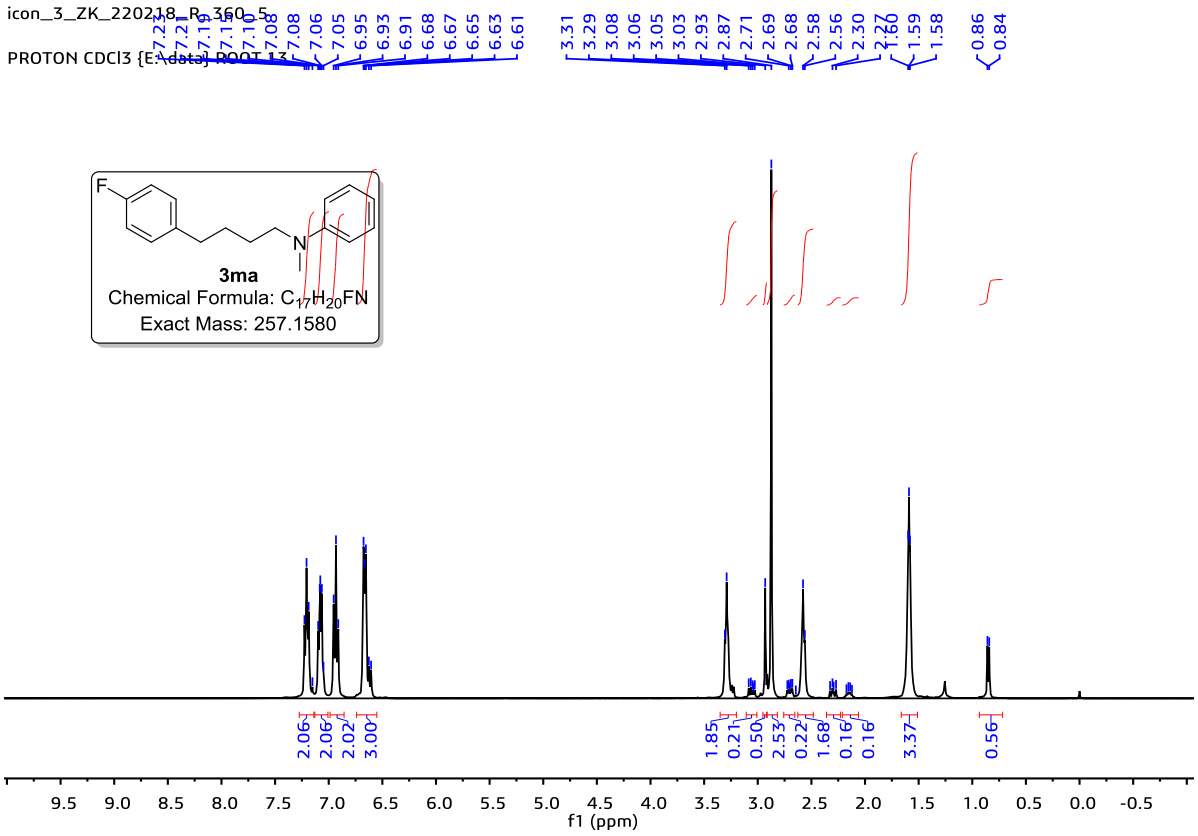

icon_3_ZK_220221_R_361_1
C13CPD CDCl3 {E:\data} ROOT 1
149.42 149.10 144.38 141.15 141.07 141.05 139.40 138.96 128.87 127.12 116.16 115.91 112.33 111.90
77.16 Chloroform-d
61.10 52.37 39.75 38.45 38.08 33.08 28.29 18.96
Ph
N
3ja
Chemical Formula: C22H23N
Exact Mass: 301.1830
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

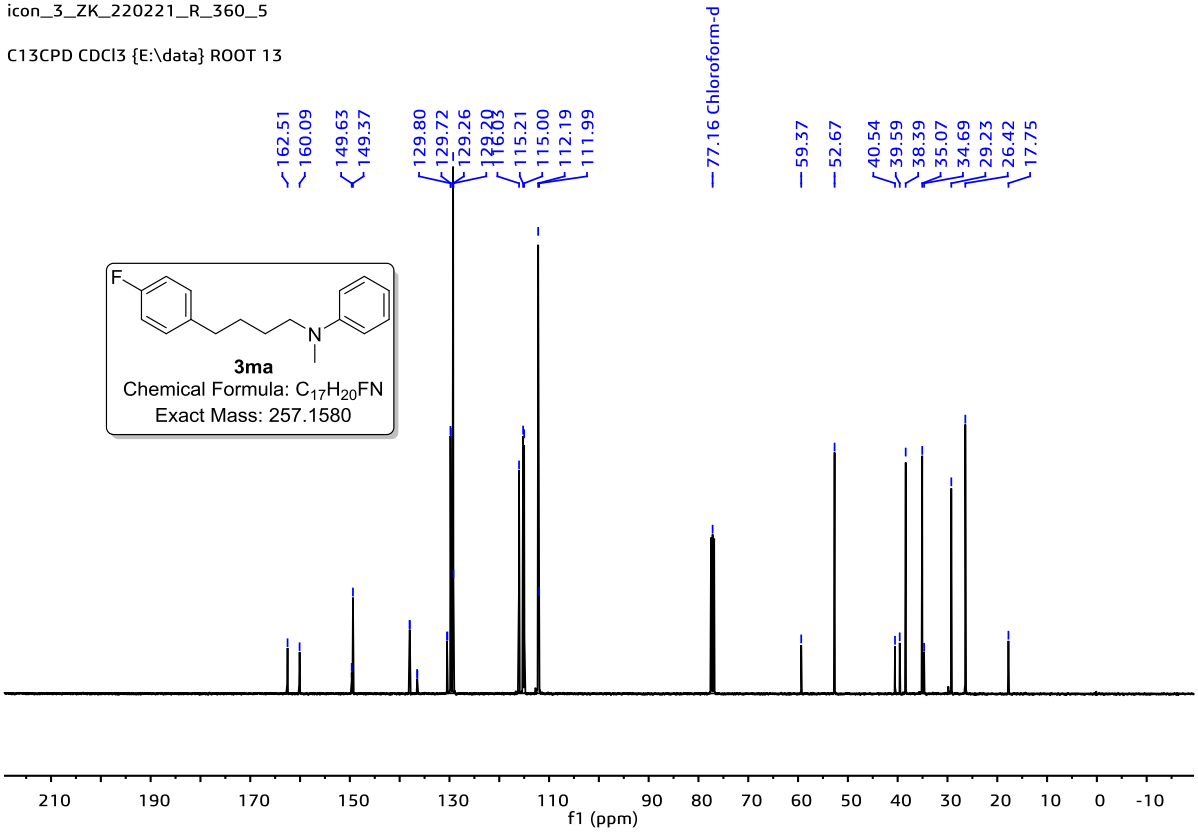

icon_3_ZK_220218_R_360_3
PROTON CDCl3 {E:\data} ROOT 11
7.26
7.24
7.22
7.21
7.18
7.17
7.16
7.15
7.13
7.11
6.66
6.64
6.61
6.59
3.28
3.27
3.25
3.23
3.06
3.04
3.02
3.00
2.90
2.84
2.73
2.72
2.70
2.69
2.61
2.60
2.58
2.32
2.29
1.60
1.60
1.59
0.86
0.84
0.00
3ka
Chemical Formula: C17H21N
Exact Mass: 239.1674
7.06
3.00
1.81
0.27
0.74
2.34
0.26
1.66
0.20
0.19
3.23
0.64
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

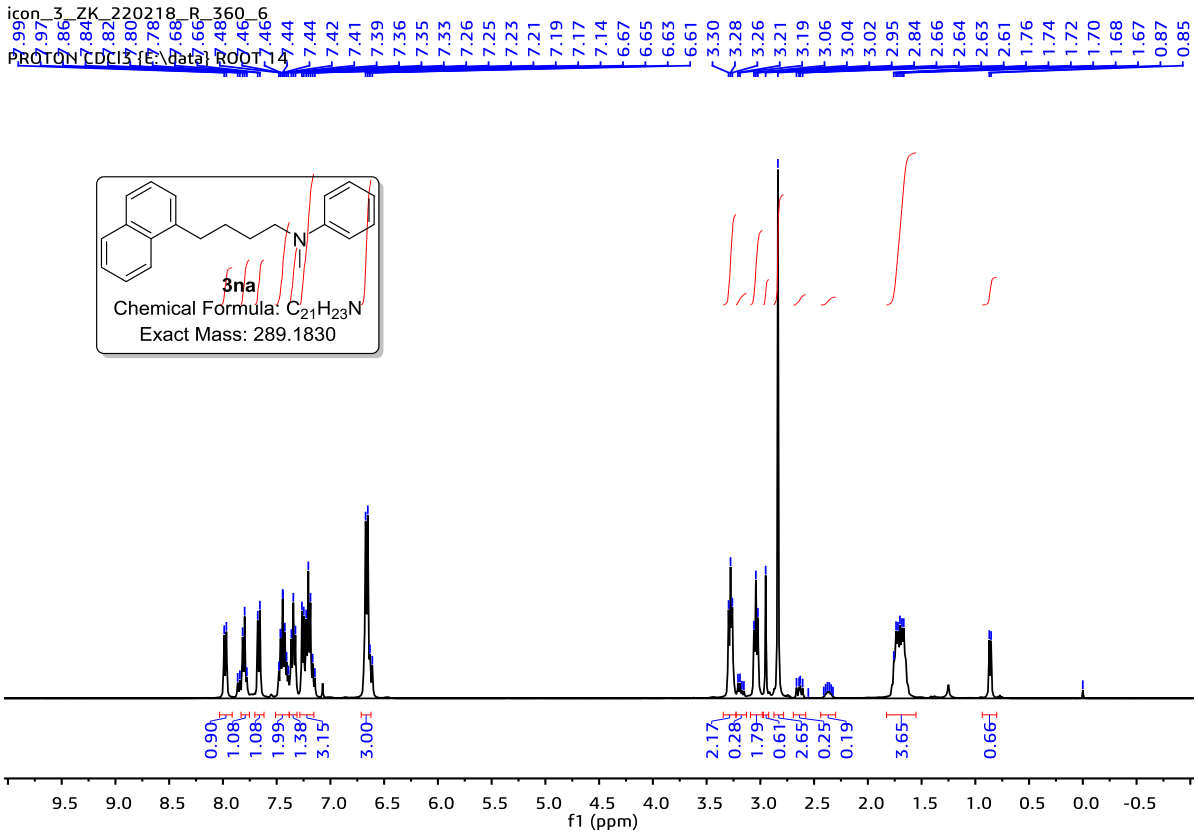

icon_3_ZK_220221_R_360_3
C13CPD CDCl3 {E:\data} ROOT 11
149.34
142.35
129.21
128.44
128.38
125.83
115.95
112.14
77.16 Chloroform-d
59.38
52.66
41.39
39.53
38.34
35.88
34.57
29.09
26.48
17.87
3ka
Chemical Formula: C17H21N
Exact Mass: 239.1674
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

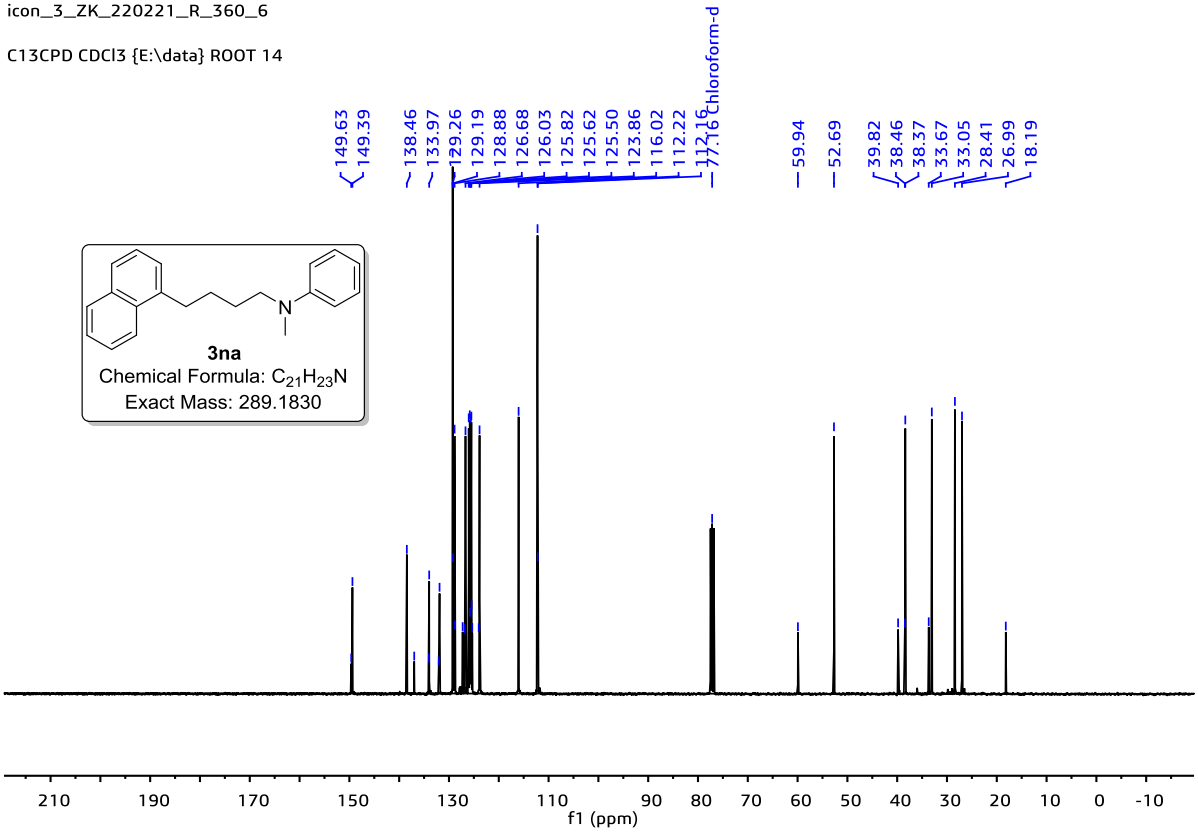

icon_3_ZK_220218_R_360_4
PROTON CDCl3 {E:\data} ROOT 12
7.23
7.21
7.19
7.17
7.08
7.06
7.04
6.82
6.80
6.68
6.66
6.63
6.61
3.76
3.30
3.05
2.94
2.88
2.68
2.66
2.65
2.57
1.31
1.60
0.87
0.86
-0.00
MeO
N
3la
Chemical Formula: C18H23NO
Exact Mass: 269.1780
2.09
2.02
2.06
3.00
3.10
1.81
0.25
0.66
2.36
0.29
1.58
0.19
0.19
3.32
0.67
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

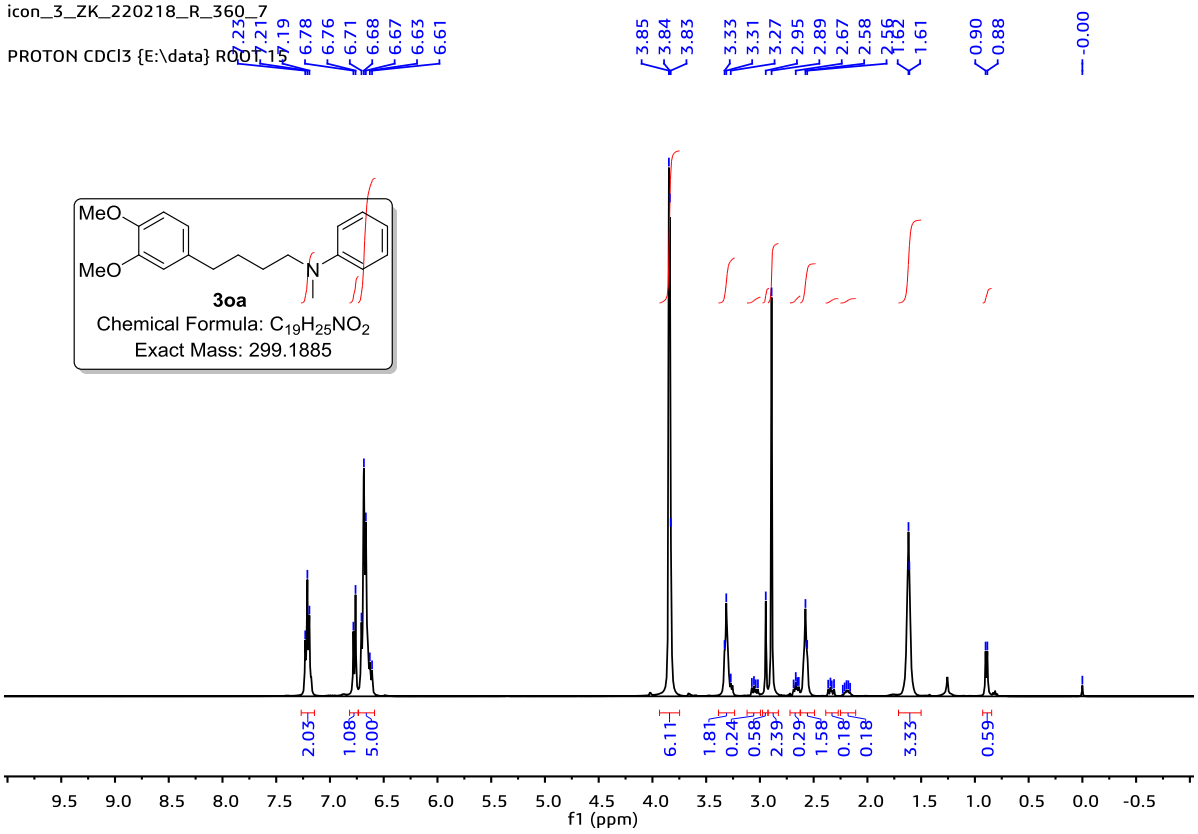

icon_3_ZK_220221_R_360_4
C13CPD CDCl3 {E:\data} ROOT 12
157.89
157.81
149.68
149.40
134.51
129.35
129.25
115.80
113.81
113.71
112.17
111.96
77.16 Chloroform-d
59.40
55.33
52.75
40.52
39.60
38.41
34.99
34.72
29.38
26.46
17.91
MeO
N
3la
Chemical Formula: C18H23NO
Exact Mass: 269.1780
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

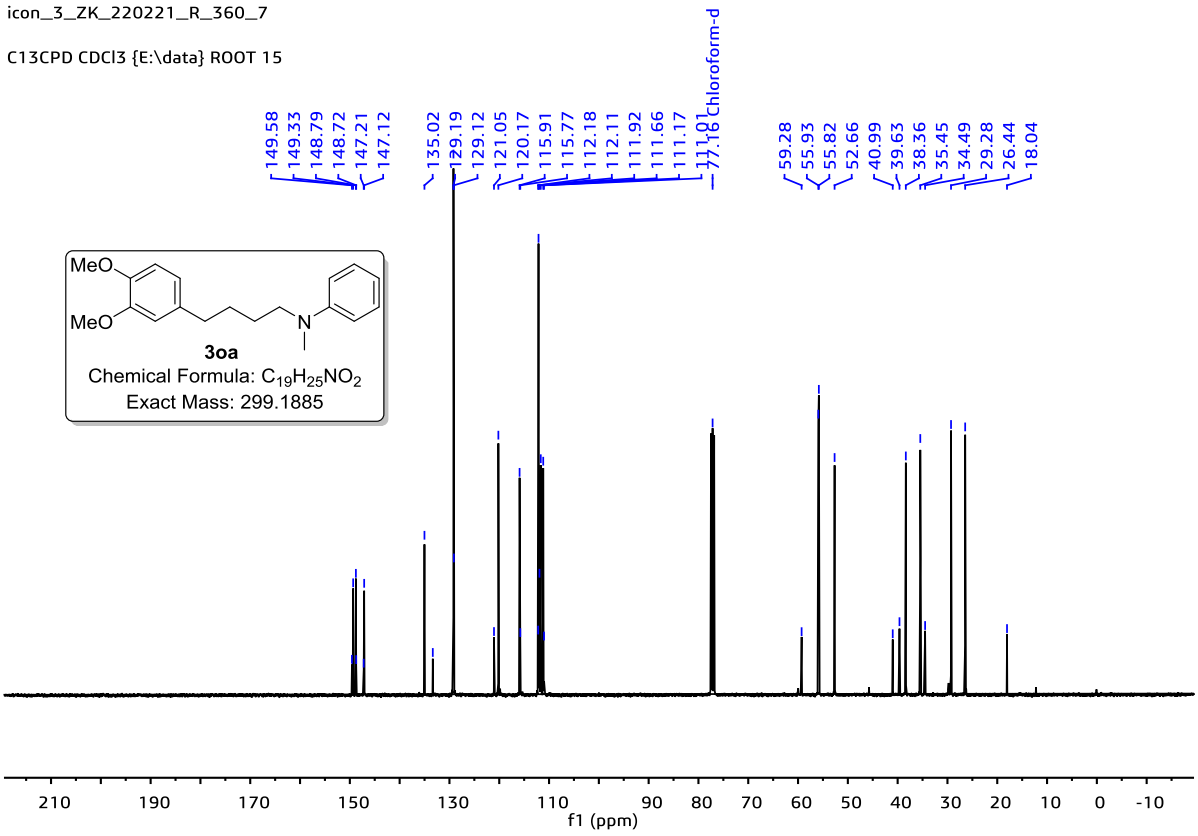

icon_3_ZK_220218_R_360_5
PROTON CDCl3 {E:\data} ROOT 13
7.25 7.21 7.19 7.15 7.10 7.08 7.08 7.06 7.05 6.95 6.93 6.91 6.68 6.67 6.65 6.63 6.61
3.31 3.29 3.08 3.06 3.05 3.03 2.93 2.87 2.71 2.69 2.68 2.58 2.56 2.30 2.27 1.60 1.59 1.58 0.86 0.84
F
N
3ma
Chemical Formula: C17H20FN
Exact Mass: 257.1580
2.06 2.06 2.02 3.00 1.85 0.21 0.50 2.53 0.22 1.68 0.16 0.16 3.37 0.56
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

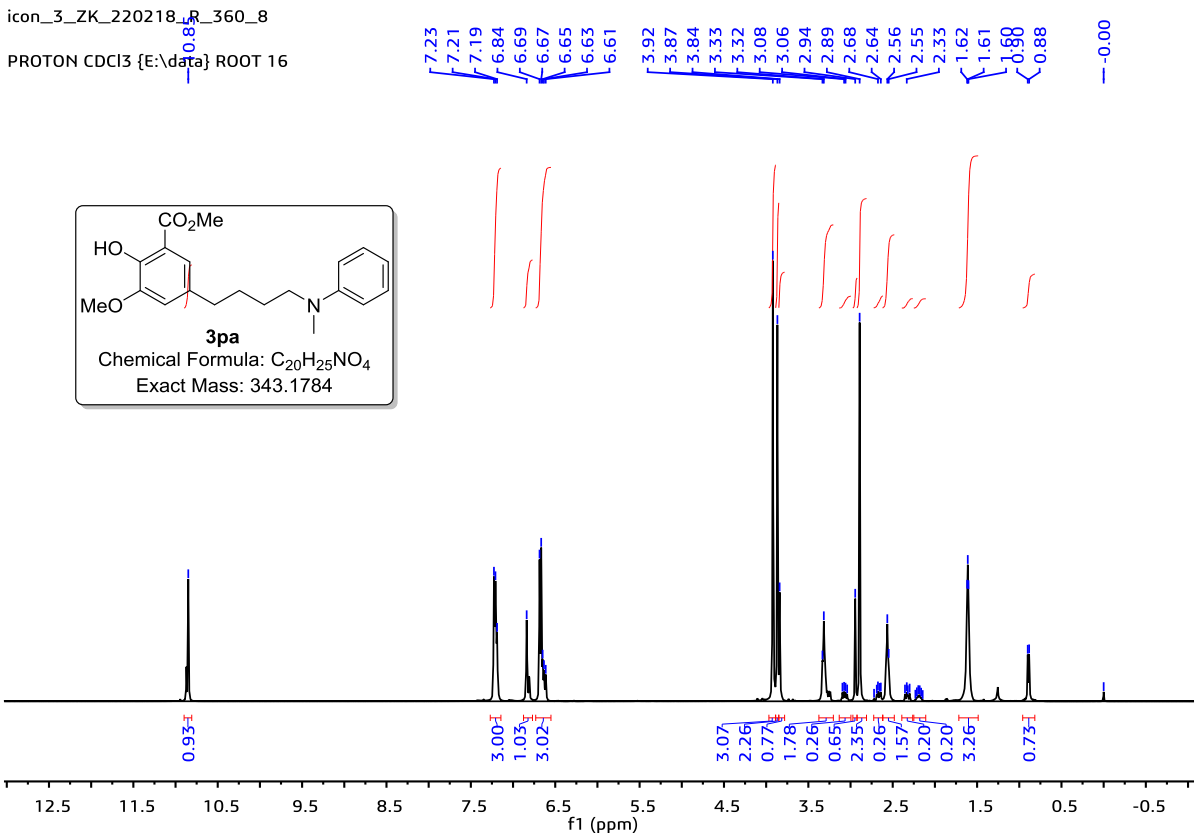

icon_3_ZK_220221_R_360_5
C13CPD CDCl3 {E:\data} ROOT 13
162.51 160.09 149.63 149.37 129.80 129.72 129.26 115.21 115.00 112.19 111.99
77.16 Chloroform-d
59.37 52.67 40.54 39.59 38.39 35.07 34.69 29.23 26.42 17.75
F
N
3ma
Chemical Formula: C17H20FN
Exact Mass: 257.1580
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

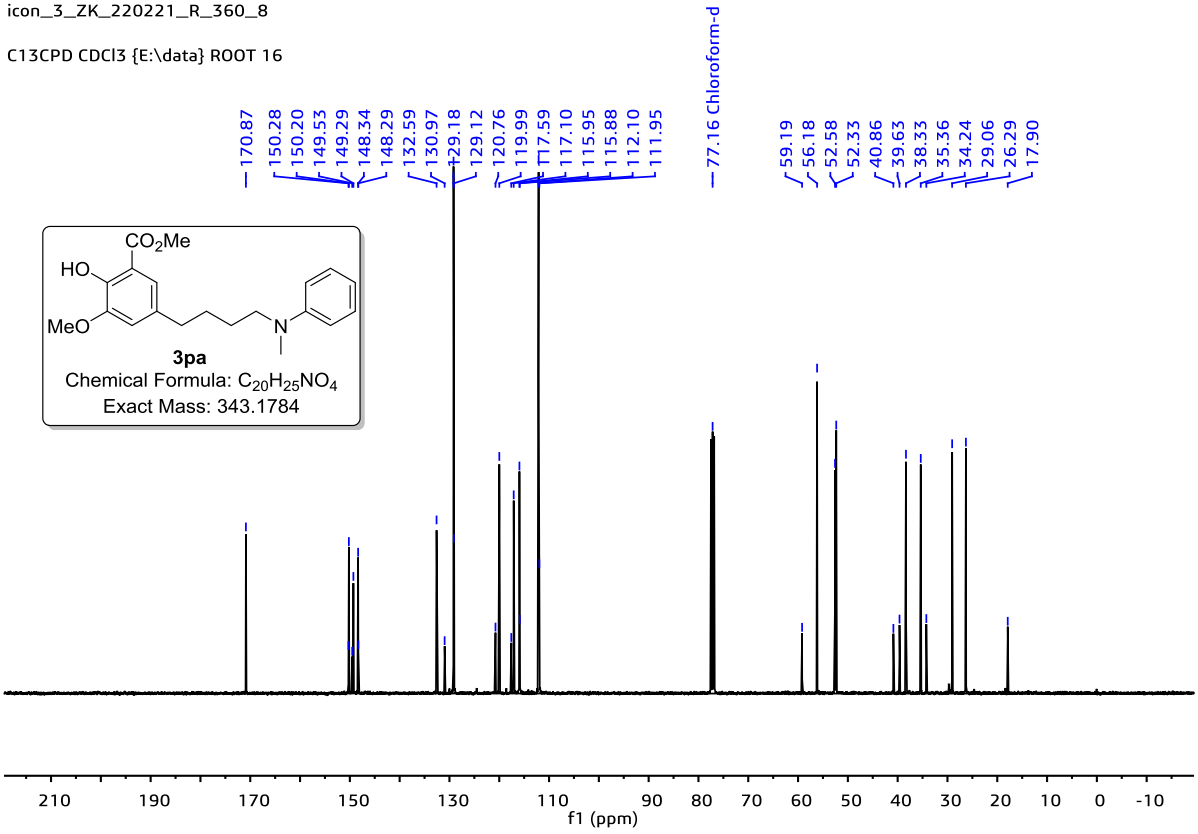

icon_3_ZK_220218_R_360_6
PROTON CDCl3 {E:\data} ROOT 14
3na
Chemical Formula: C21H23N
Exact Mass: 289.1830
f1 (ppm)


icon_3_ZK_220221_R_360_6
C13CPD CDCl3 {E:\data} ROOT 14
Chloroform-d
3na
Chemical Formula: C21H23N
Exact Mass: 289.1830
f1 (ppm)


icon_3_ZK_220218_R_360_7
PROTON CDCl3 {E:\data} ROOT 15
MeO
MeO
N
3oa
Chemical Formula: C19H25NO2
Exact Mass: 299.1885
f1 (ppm)


icon_3_ZK_220221_R_360_7
C13CPD CDCl3 {E:\data} ROOT 15
Chloroform-d
MeO
MeO
N
3oa
Chemical Formula: C19H25NO2
Exact Mass: 299.1885
f1 (ppm)


icon_3_ZK_220218_R_360_8
PROTON CDCl3 {E:\data} ROOT 16
10.85
7.23
7.21
7.19
6.84
6.69
6.67
6.65
6.63
6.61
3.92
3.87
3.84
3.33
3.32
3.08
3.06
2.94
2.89
2.68
2.64
2.56
2.55
2.33
1.62
1.61
1.60
0.90
0.88
-0.00
CO2Me
HO
MeO
N
3pa
Chemical Formula: C20H25NO4
Exact Mass: 343.1784
0.93
3.00
1.03
3.02
3.07
2.26
0.77
1.78
0.26
0.65
2.35
0.26
1.57
0.20
0.20
3.26
0.73
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


icon_3_ZK_220221_R_360_8
C13CPD CDCl3 {E:\data} ROOT 16
170.87
150.28
150.20
149.53
149.29
148.34
148.29
132.59
130.97
129.18
129.12
120.76
119.99
117.59
117.10
115.95
115.88
112.10
111.95
77.16 Chloroform-d
59.19
56.18
52.58
52.33
40.86
39.63
38.33
35.36
34.24
29.06
26.29
17.90
CO2Me
HO
MeO
N
3pa
Chemical Formula: C20H25NO4
Exact Mass: 343.1784
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

9. References

1. G. Ji, C. Li, D. Xiao, G. Wang, Z. Sun, M. Jiang, G. Hou, L. Yan and Y Ding, *J. Mater. Chem. A*, 2021, **9**, 9165-9174.
2. T. Egawa, Y. Koide, K. Hanaoka, T. Komatsu, T. Terai and T. Nagano, *Chem. Commun.*, 2011, **47**, 4162-4164.
3. K. Li, Y. Wang, Y. Xu, W. Li, M. Niu, J. Jiang and Z. Jin, *Catal. Commun.*, 2013, **34**, 73-77.
4. X. Liu, A. Hamasaki, Y. Yamane, S. Aikawa, T. Ishida, M. Haruta and M. Tokunaga, *Catal. Sci. Technol.*, 2013, **3**, 3000-3006.
5. N. Sudheesh and R. S. Shukla, *Appl. Catal., A*, 2013, **453**, 159-166.
6. N. Sudheesh and R. S. Shukla, *Appl. Catal., A*, 2014, **473**, 116-124.