

Supporting Information for:

**Secondary Phosphine Oxides Stabilized Au/Pd Nanoalloys:
Metal Components-Controlled Regioselective Hydrogenation
toward Phosphinyl (Z)-[3]Dendralenes**

Yun-Tao Xia,^a Xiao-Yu Xie,^a Su-Hang Cui,^a Yi-Gang Ji,^c and Lei Wu,^{*,a,b}

^aJiangsu Key Laboratory of Pesticide Science and Department of Chemistry, College of Sciences, Nanjing Agricultural University, Nanjing 210095, P. R. China. Tel/Fax: +862584395351; Email: rickywu@njau.edu.cn.

^bBeijing National Laboratory for Molecular Sciences and Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, P. R. China.

^cJiangsu Key Laboratory of Biofunctional Molecules, Department of Life Sciences and Chemistry, Jiangsu Second Normal University, Nanjing 210013, P. R. China

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1. General Information

Solvents and reagents were reagent grade and used without purification unless otherwise noted. Column chromatography was performed using silica gel (200–300 mesh). All ¹H-NMR (400 MHz) spectra were recorded on a Bruker-DMX 400 using CDCl₃ solution in the presence of tetramethylsilane (TMS) as an internal standard and are reported in ppm (δ). Coupling constants are reported in Hertz (Hz). Spectral splitting patterns are designated as s, singlet; d, doublet; t, triplet; q, quartet; p, pentet; m, multiplet; and br, broad. High resolution mass spectroscopic data of the products were collected on a Waters Micromass GCT instrument using EI (70 eV) or an Agilent Technologies 6540 UHD Accurate-Mass Q-TOF LC/MS using ESI. Transmission Electron Microscopy (TEM): the tetrahydrofuran dispersion of catalyst was drop-cast onto a 300 mesh carbon coated copper grid, and TEM pictures were taken on a FEI-Tecnai G2 F30 S-TWIN at an accelerating voltage of 75 kV. Fourier Transform Infrared Spectroscopy (FT-IR): FT-IR measurements were carried out on a Bruker Optics FT-IR Alpha spectrometer equipped with a DTGS detector, KBr beamsplitter at 4 cm⁻¹ resolution in the range 4000–500 cm⁻¹. X-ray Photoelectron Spectroscopy (XPS): These experiments were carried out at the testing center of Yangzhou University, The instrument model: ESCALAB250Xi. Energy Dispersive X-ray Spectroscopy (EDX): EDX analyses were performed on a FEI-Tecnai G2 F30 equipped with an EDS Detector employing copper as internal standard. Samples were prepared drop casting (from various THF solutions). Inductively Coupled Plasma (ICP): Inductively coupled plasma-mass spectra (ICP-MS) were taken from Optima 7300 DV (PerkinElmer). Phase Analysis of X-ray Diffraction (XRD): These experiments were carried out at the testing center of Nanjing University of Science and Technology, XRD data were collected with CuKa radiation on Bruker C8 ADVANCE. UV Spectrophotometer (UV): These experiments were carried out at the testing center of Nanjing University of Science and Technology, using Cary 5000, and the spectral range: 200 ~ 3300 nm, absorbance linear range: 0 ~ 8.0 Abs, wavelength accuracy: ± 0.08 nm (UV/Vis); ± 0.4 nm (NIR). The surface area of the catalysts was determined by the multi-point BET method using N₂ adsorption (V-Sorb 2800P analyzer, Jinaipu, China).

2. Synthesis of the Au(I)-SPO and Pd(II)-SPO Precursors

Au(I)-SPO: $\{[\text{Ph}_2(\text{O})\text{H}]\text{AuCl}\}^{[1]}$

To a 25 mL vial was added HAuCl₄•3H₂O (500 mg, 1.27 mmol) and ethanol (10 ml), after tetrahydrothiophene (2.24 mL, 25.4 mmol, 20 eq.) was dropped until the yellow color had completely dissipated. The white precipitation [(tht)AuCl] was collected by centrifugation and washed with cold ethanol for twice before drying in vacuum, giving the desired gold complex in a quantitative yield. Then diphenylphosphine oxide (101 mg, 0.5 mmol) and [(tht)AuCl] (160 mg, 0.5 mmol) were dissolved in dichloromethane (5 mL). The resulting solution was stirred for three hours, then concentrated under vacuum and diluted with hexane (10 mL). The mixture was kept overnight at -30 °C. After filtration, a white solid of [Ph₂(O)H]AuCl (155 mg, 0.36 mmol) was obtained.

[1] I. Cano, A. M. Chapman, A. Urakawa, and P. W. N. M. van Leeuwen, *J. Am. Chem. Soc.*, **2014**, *136*, 2520.

Pd(II)-SPO: $\{\text{PdCl}_2[\text{Ph}_2(\text{O})\text{H}]_2\}^{[2]}$

To a 25 mL vial was added allylpalladium chloride dimer [Pd(Cl)(C₃H₅)₂] (182.9 mg, 0.5 mmol), diphenylphosphine oxide (202 mg, 1mmol) and CH₂Cl₂ (5 mL), the solution was stirred for three hours. The resulting solution was concentrated under vacuum and precipitated with 10 mL hexanes. A yellow solid (235 mg, 0.4 mmol) was obtained after filtration.

[2] Robin B. B., Samantha L. H., and Michael E. L.; *Organometallics*; **2003**, *22*, 1364.

3. Synthetic Procedures for SPOs-Au/PdNAs, SPOs-PdNPs and SPOs-AuNPs

3.1 Procedure for Synthesizing Au/PdNAs-2

The gold complex $[\text{Ph}_2(\text{O})\text{H}]\text{AuCl}$ (0.1 mmol, 43 mg) and palladium complex $\text{PdCl}_2[\text{Ph}_2(\text{O})\text{H}]_2$ (0.05 mmol, 29 mg) were dissolved in $\text{THF}/\text{CH}_2\text{Cl}_2$ (3 ml/10 ml) and cooled to 0 °C in an ice bath. A solution of NaBH_4 (60 mg, 1.6 mmol, 16 eq.) in degassed methanol (2 mL) was prepared immediately prior to use and then added via syringe to the rapidly stirred solution resulting in immediate formation of a deep purple-brown, homogenous solution. The reaction mixture was extracted with dichloromethane (3 x 30 mL). The deep brown dichloromethane fractions were combined and washed with water (3 x 50 mL) and then concentrated under vacuum to give **Au/PdNAs-2** as black powder: 35.5 mg, 60 % (based on Au). The purified nanoalloys were obtained from precipitation with ice-cold methanol to remove unanchored ligands.

Au/PdNAs-1 and **Au/PdNAs-3**: The procedures for **Au/PdNAs-1** and **Au/PdNAs-3** were similar to the descriptions of **Au/PdNAs-2**, with differed precursor loadings as follows:

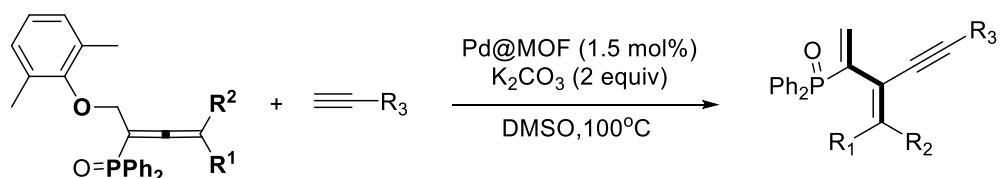
nanoalloys	Au/PdNAs-1	Au/PdNAs-3
amounts of metal precursors	$[\text{Ph}_2(\text{O})\text{H}]\text{AuCl}$: 0.1 mmol, 43 mg $\text{PdCl}_2[\text{Ph}_2(\text{O})\text{H}]_2$: 0.1 mmol, 58 mg	$[\text{Ph}_2(\text{O})\text{H}]\text{AuCl}$: 0.1 mmol, 43 mg $\text{PdCl}_2[\text{Ph}_2(\text{O})\text{H}]_2$: 0.033 mmol, 19 mg
yield	38.7 mg, 40% (based on Au)	37.2 mg, 49% (based on Au)

3.2 Procedure for Synthesizing SPOs-PdNPs

The palladium complex $\text{PdCl}_2[\text{Ph}_2(\text{O})\text{H}]_2$ (0.1 mmol, 58.8 mg) was dissolved in a mixture of $\text{THF}/\text{CH}_2\text{Cl}_2$ (3 mL/10 ml) and cooled to 0 °C in an ice bath. A solution of NaBH_4 (60 mg, 1.6 mmol, 16 eq.) in degassed methanol (2 mL) was prepared immediately prior to use and then added in one portion via syringe to the rapidly stirred solution resulting in the immediate formation of homogenous dark-brown solution. The fractions were washed with water (3 x 50 mL) and then concentrated under vacuum to afford the palladium nanoparticles as black powder: 36.7 mg, 42 % (based on Pd).

The synthetic procedure for **SPOs-AuNPs** refers to: [3] Cano, I., Huertos, M. A., Chapman, A. M., and P. W. N. M. van Leeuwen, *J. Am. Chem. Soc.*, **2015**, 137, 7718.

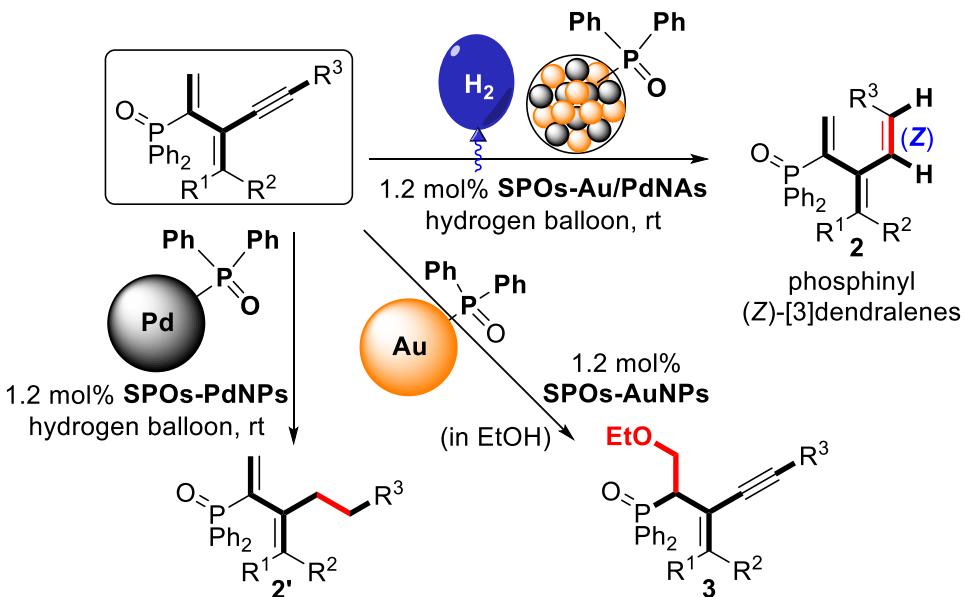
4. General Procedure for the Preparation of Phosphinyl Conjugated Enynes^[4]



To a 25 mL vial was added allenylphosphine oxides (1 mmol, 526 mg), alkynes (2 mmol, 204 mg), potassium carbonate (K₂CO₃) (2 mmol, 276 mg), DMSO (10 mL) and Pd@MOF (1.5 mol%) respectively. The reaction was then allowed to react at 100 °C for a certain time until the complete consuming of starting materials monitored by TLC. The reaction mixture was extracted with EtOAc (10 mL×3). The combined organic extract was washed with brine and dried over anhydrous Na₂SO₄. The solvent was evaporated under reduced pressure and the residue was purified by column chromatography on silica gel using petroleum ether/ethylacetate (1.5/1) as the eluent to afford the enynes.

Note: please also refer to our previous work on synthesizing phosphinyl conjugated enynes: [4]
X. D. Wang, J. J. Wu, X. Sun, W. C. Yang, Zhu. J, Wu. L, *Adv. Synth. Catal.*, **2018**, 360, 3518.

5. General Procedures for the Catalytic Reactions in This Work.



Catalytic hydrogenations: To a 10 mL vial equipped with hydrogen balloon was added **1a** (0.2 mmol, 101 mg), DMAc (2 mL) and **Au/PdNAs-2** (0.0024 mmol, 4 mg). The reaction was then allowed to react at room temperature until the complete consuming of starting materials monitored by TLC. The reaction was quenched by adding 2 mL water, and then extracted with diethyl ether (2 mL×3). Purification on silica chromatography with ethyl acetate and petroleum ether (eluent: 1:2 (v/v) of ethyl acetate/petroleum ether) afforded the target product **2a** (96 mg, 94% yield) as a white solid.

Conditions for SPOs-AuNPs catalyzed Michael addition: **1a** (0.2 mmol), SPOs-AuNPs (1.2 mol%, 0.94 mg), NaOH (0.4 mmol) and ethanol (2 mL), 50 °C, 24 h;

Conditions for SPOs-PdNPs catalyzed hydrogenation: **1a** (0.2 mmol), SPOs-PdNPs (1.2 mol%, 2.1 mg), H₂(balloon) and DMF (2 mL), 6 h;

6. ICP Data for All of the Nanocatalysts

Detecting element	Pd-NPs	Au/PdNAs-1	Au/PdNAs-2	Au/PdNAs-3
Au (wt%)	-	20.33 %	33.43 %	25.82 %
Pd (wt%)	12.12 %	7.33 %	6.61%	3.94 %

7. XPS Spectra for Au/PdNAS-2

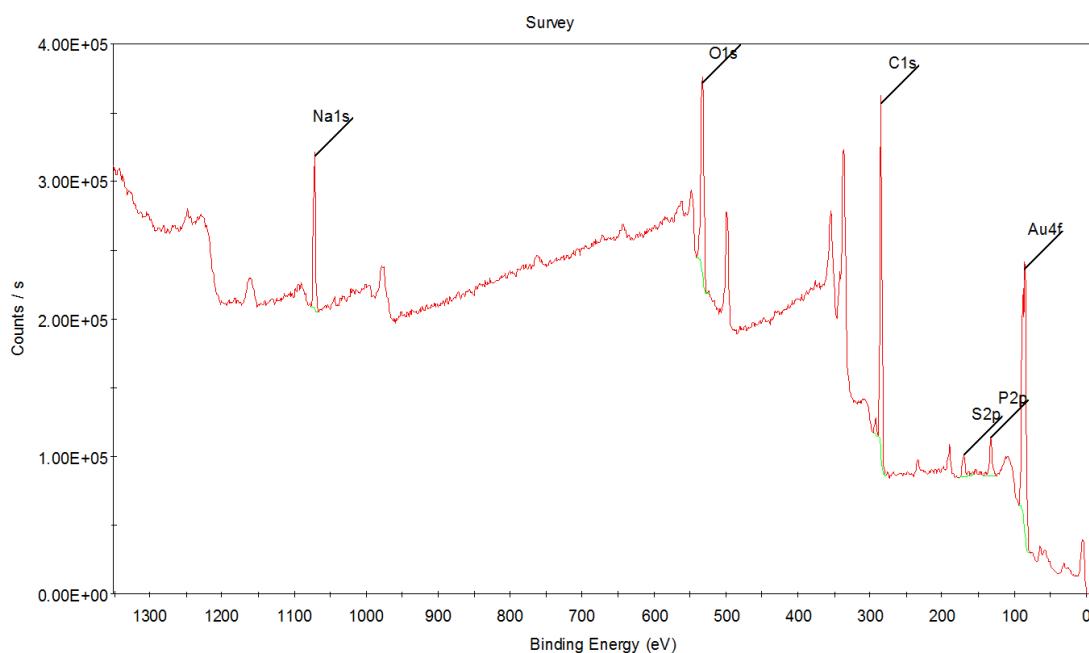


Figure S1. XPS spectrum of Au/PdNAS-2.

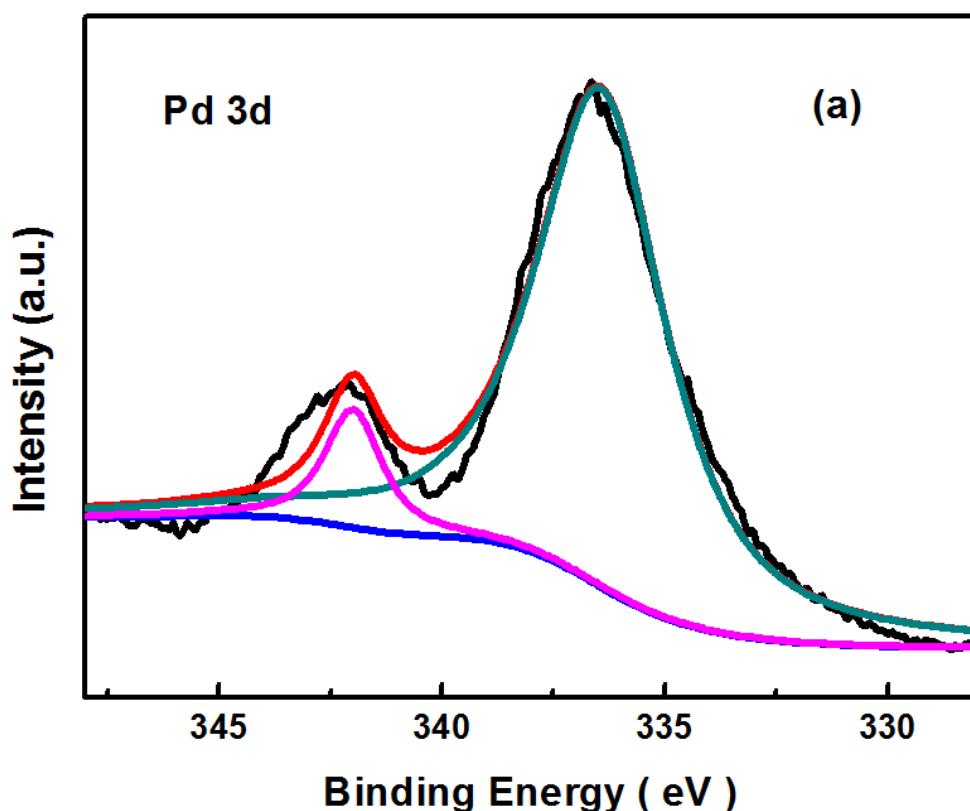


Figure S2. XPS spectrum of Au/PdNAS-2, which shows the presence of Pd 3d.

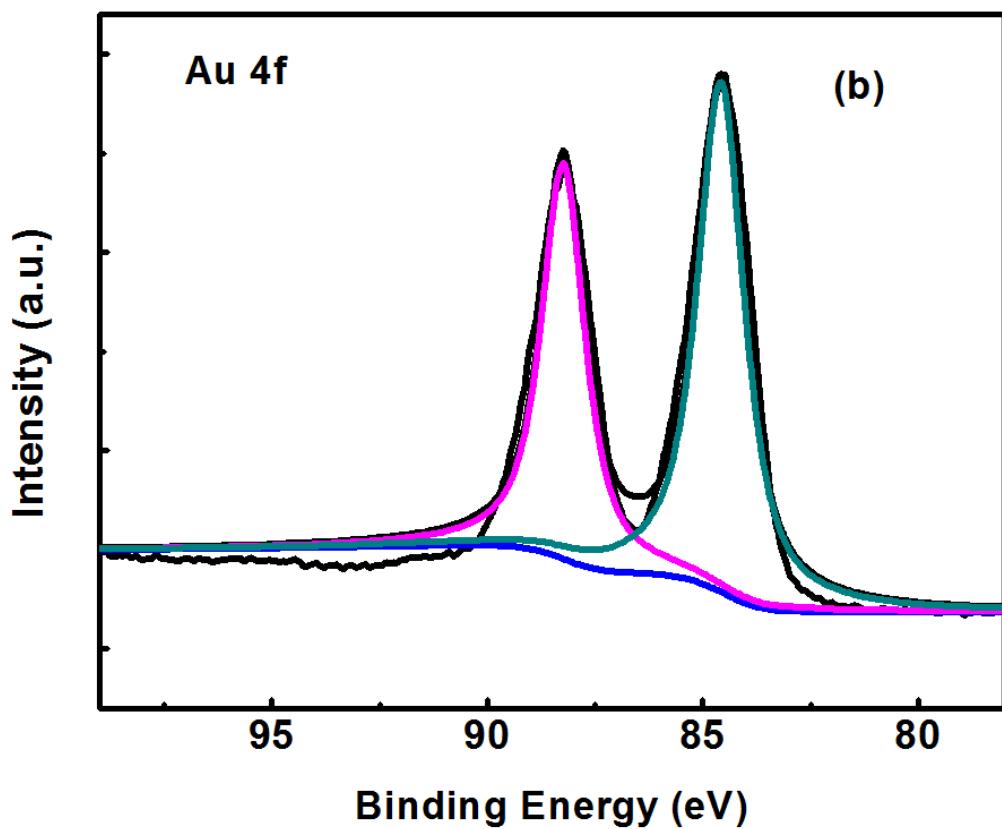


Figure S3. XPS spectrum of Au/PdNAs-2, which shows the presence of Au 4f.

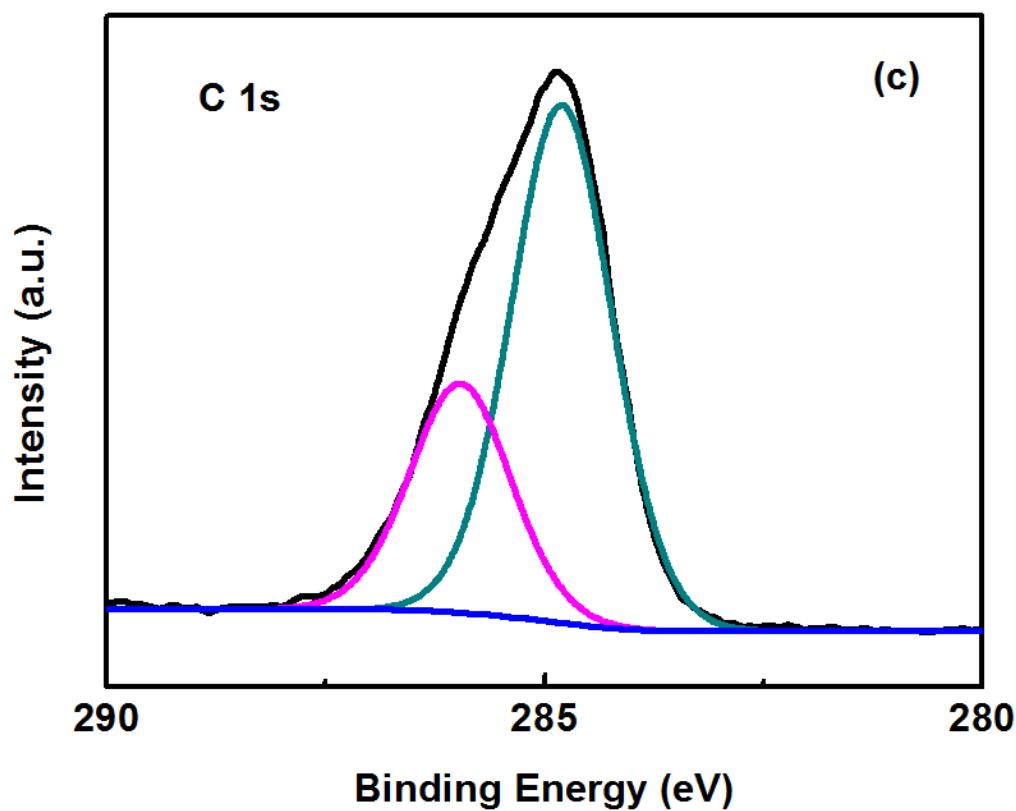


Figure S4. XPS spectrum of Au/PdNAs-2, which shows the presence of C 1s.

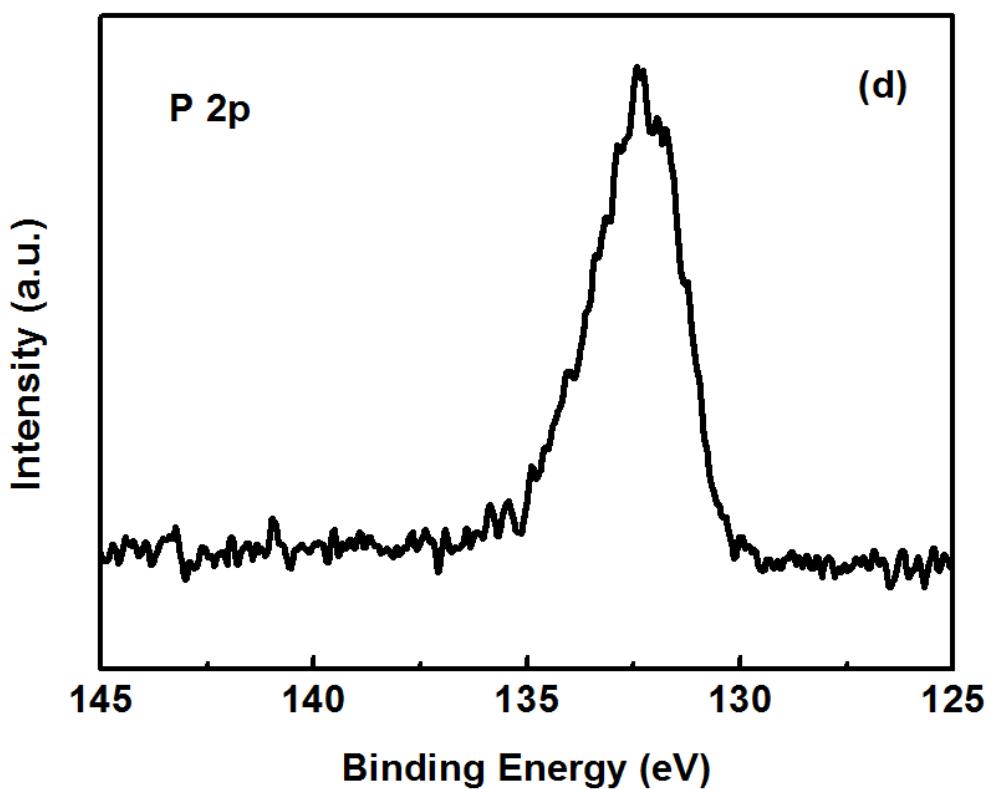


Figure S5. XPS spectrum of Au/PdNAs-2, which shows the presence of P 2p.

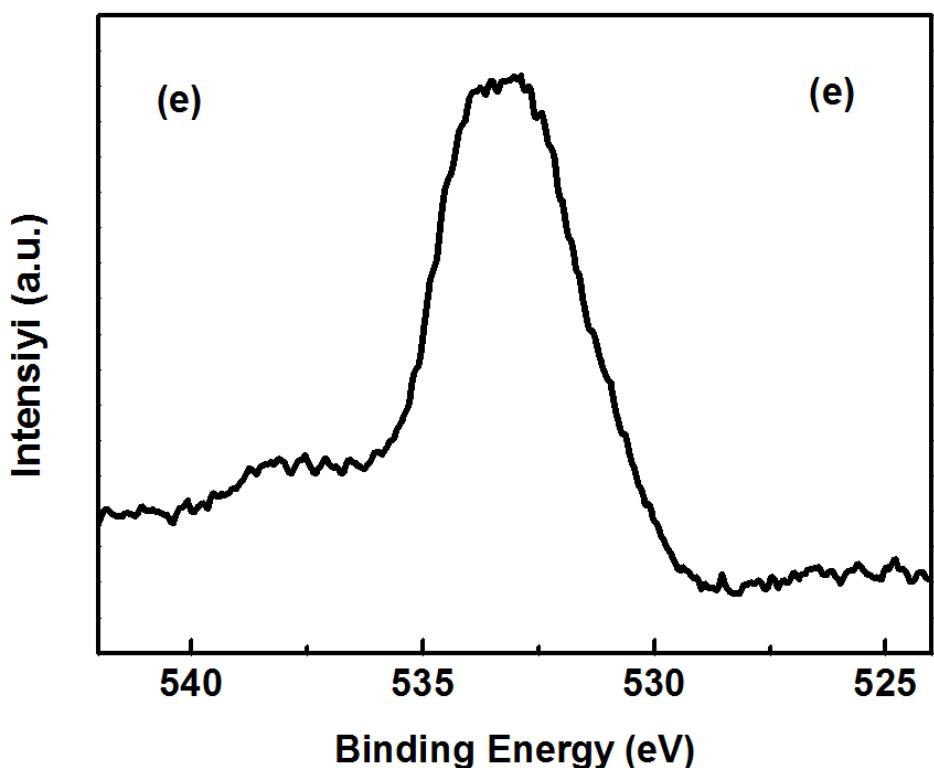


Figure S6. XPS spectrum of Au/PdNAs-2, which shows the presence of O1s.

8. EDX Spectra for SPOs-PdNPs, Au/PdNAS-1, Au/PdNAS-2 and Au/PdNAS-3

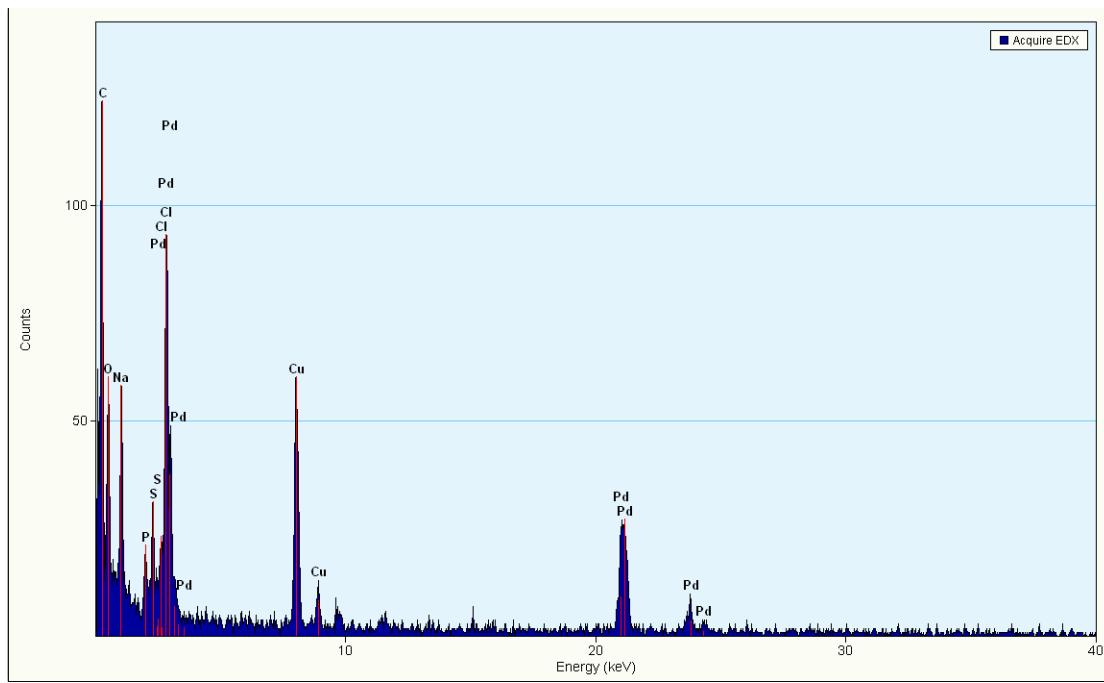


Figure S7. EDX spectrum for SPOs-PdNPs

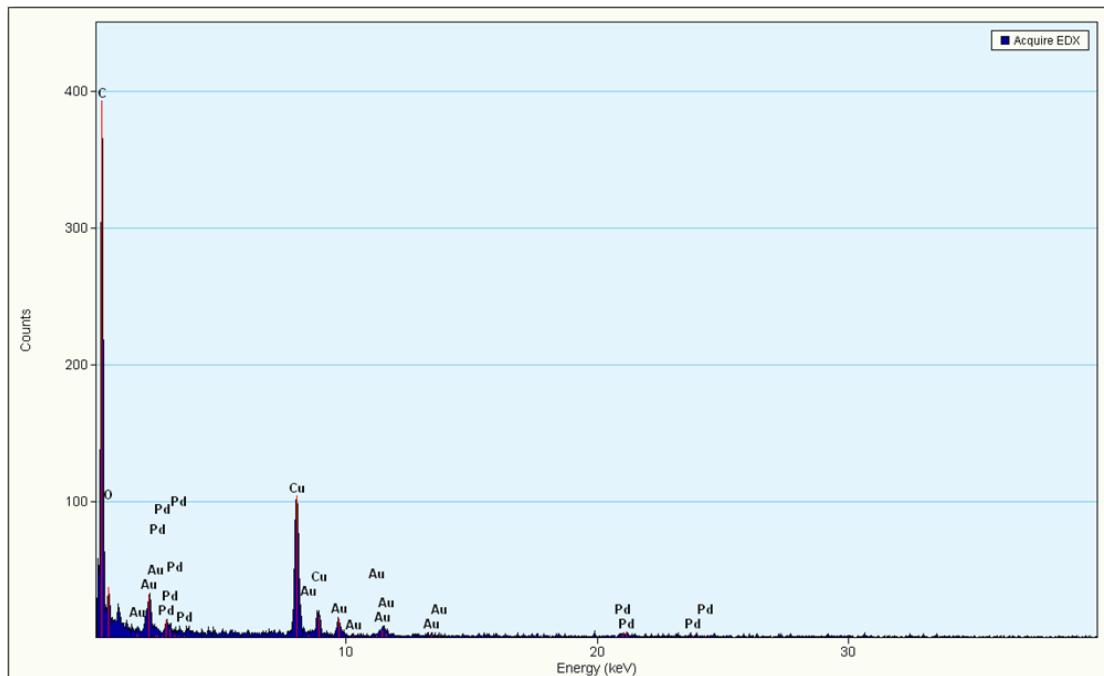


Figure S8. EDX spectrum for Au/PdNAS-1

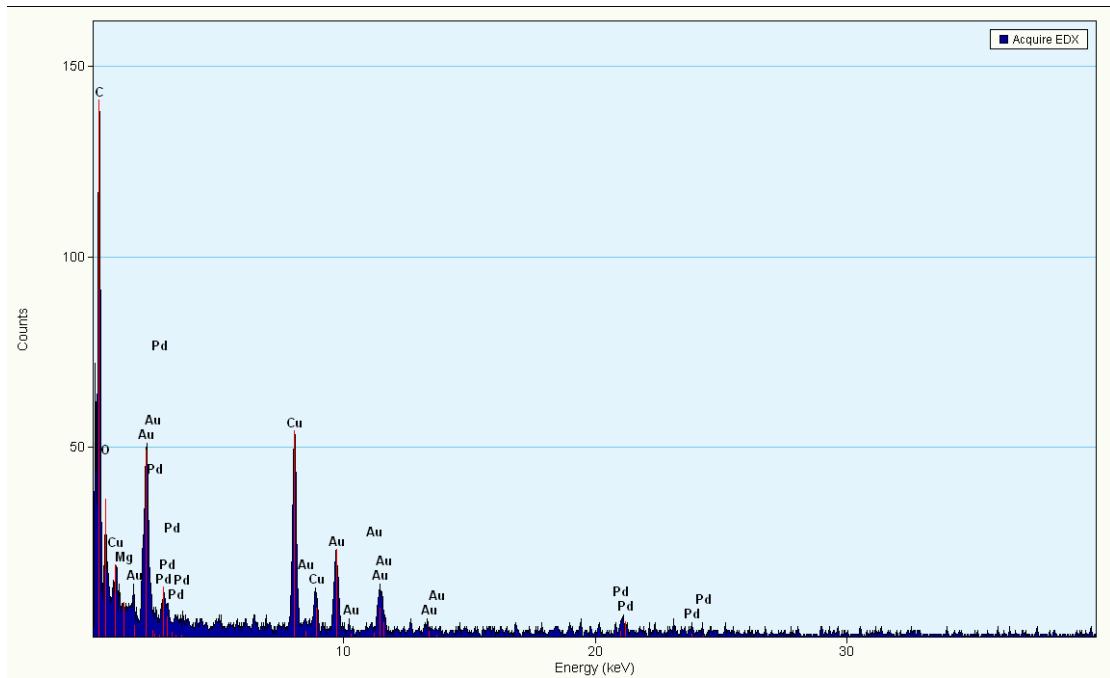


Figure S9. EDX spectrum for Au/PdNAs-2

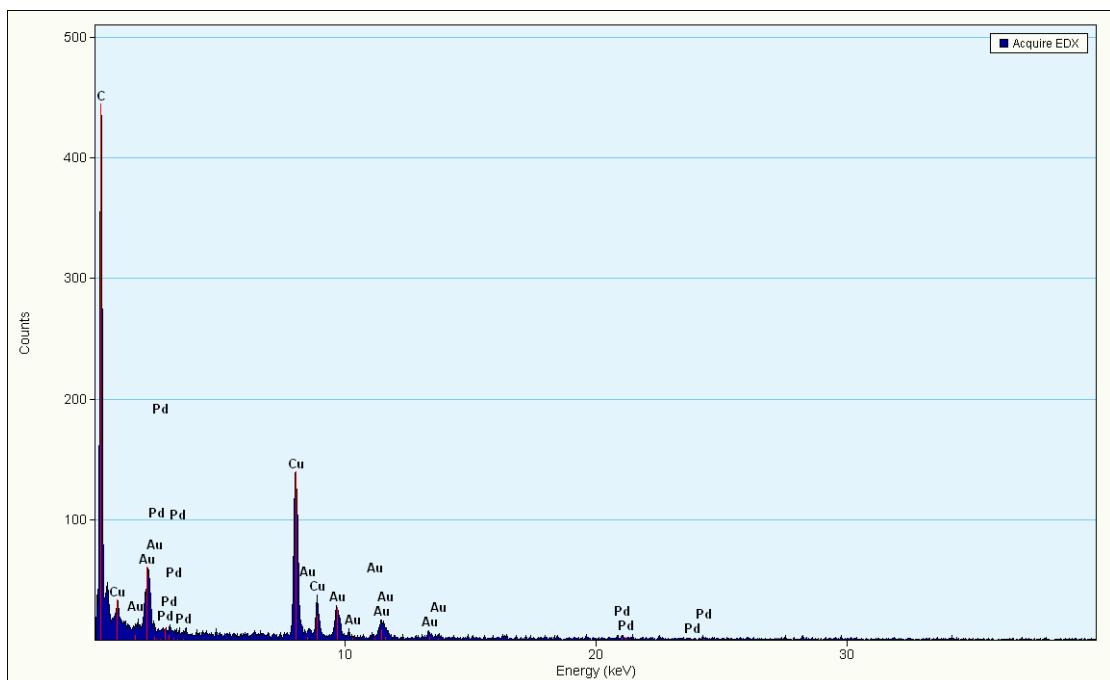


Figure S10. EDX spectrum for Au/PdNAs-3

Detecting element	Au/PdNAs-1 (Au/Pd)	Au/PdNAs-2 (Au/Pd)	Au/PdNAs-3 (Au/Pd)
EDX (TEM)	1.6:1	3.0:1	4.4:1

9. TEM Images for All of the Nanocatalysts and STEM-mappings for Nanoalloys

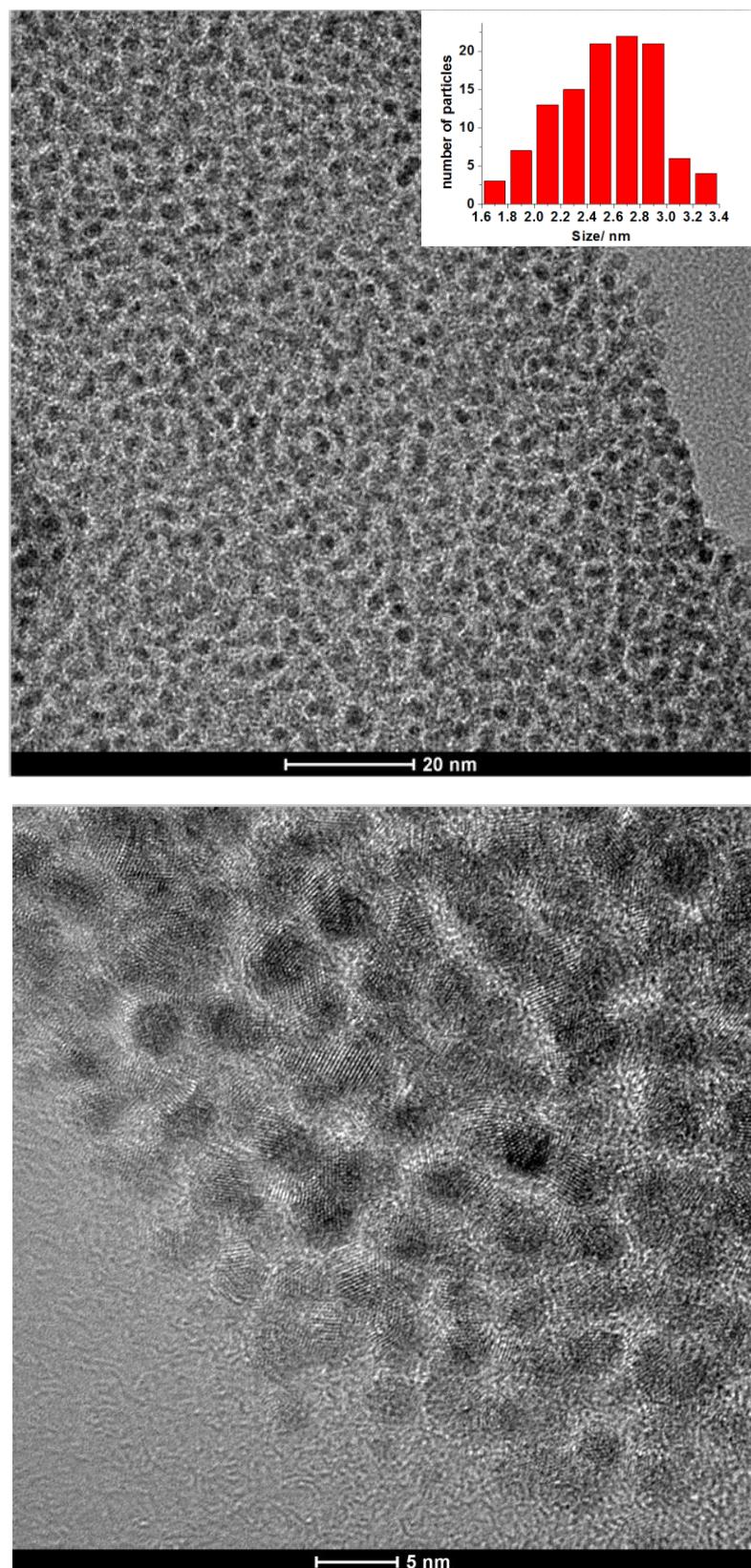


Figure S11. TEM images of SPOs-PdNPs

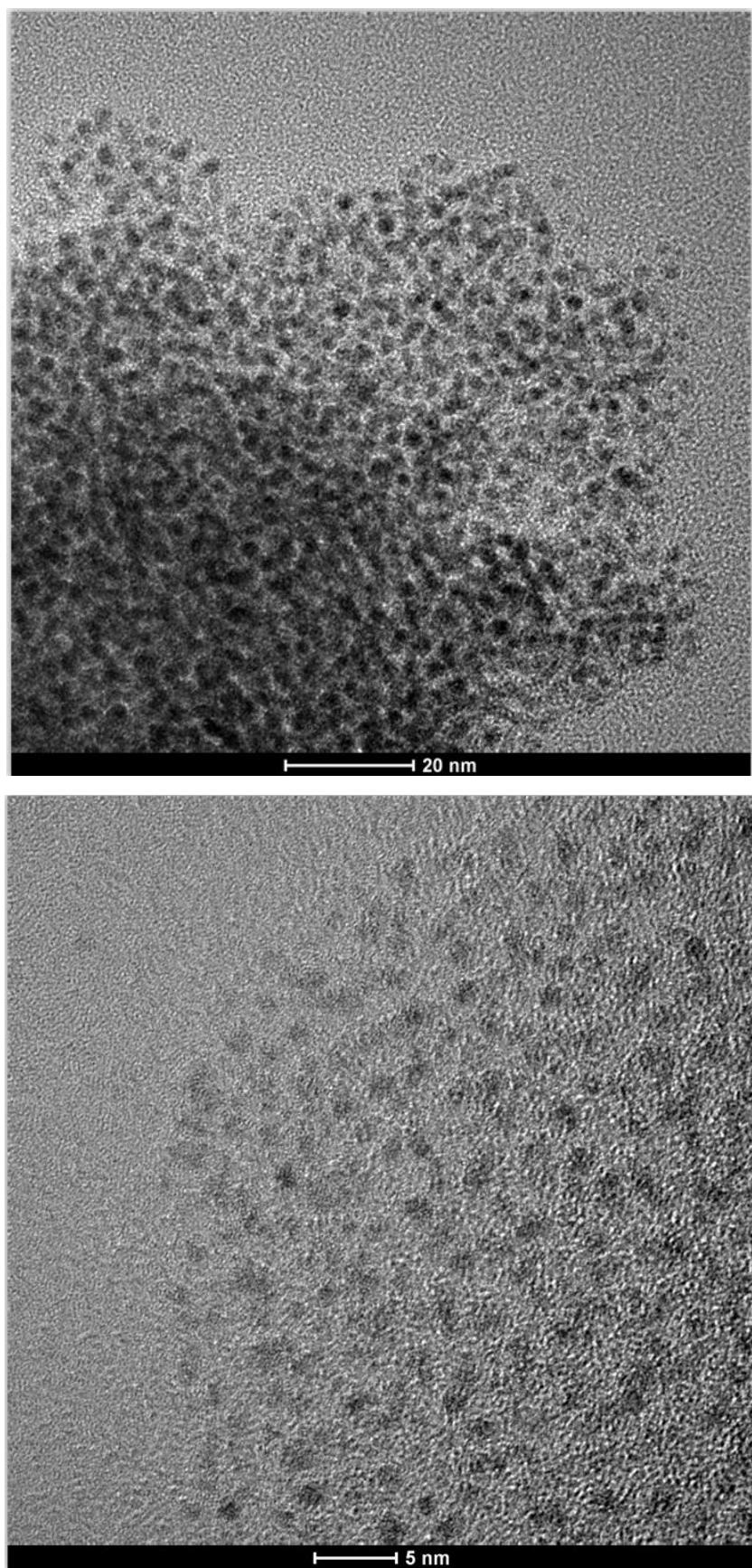


Figure S12. TEM images of Au/PdNAs-1

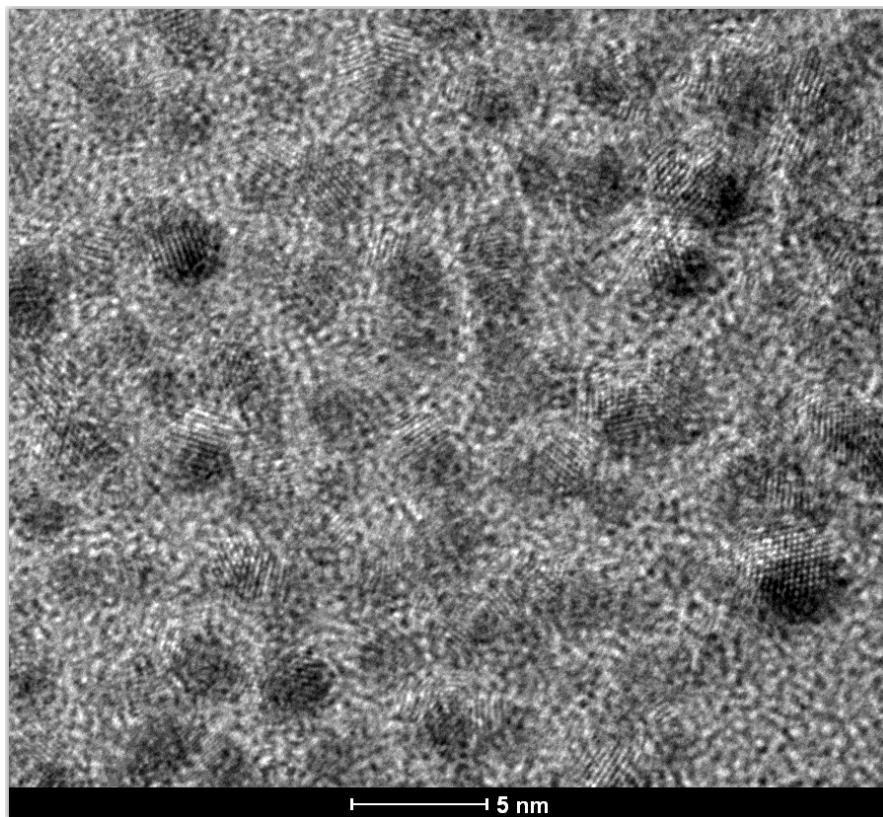
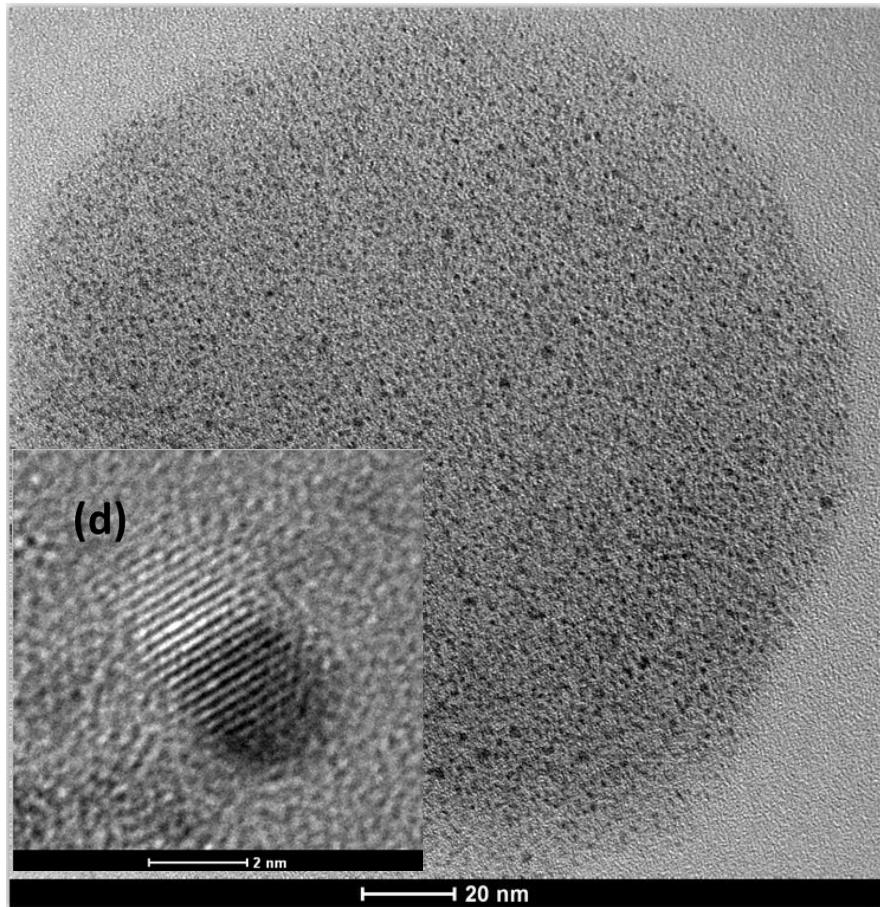


Figure S13. TEM images of Au/PdNAS-2

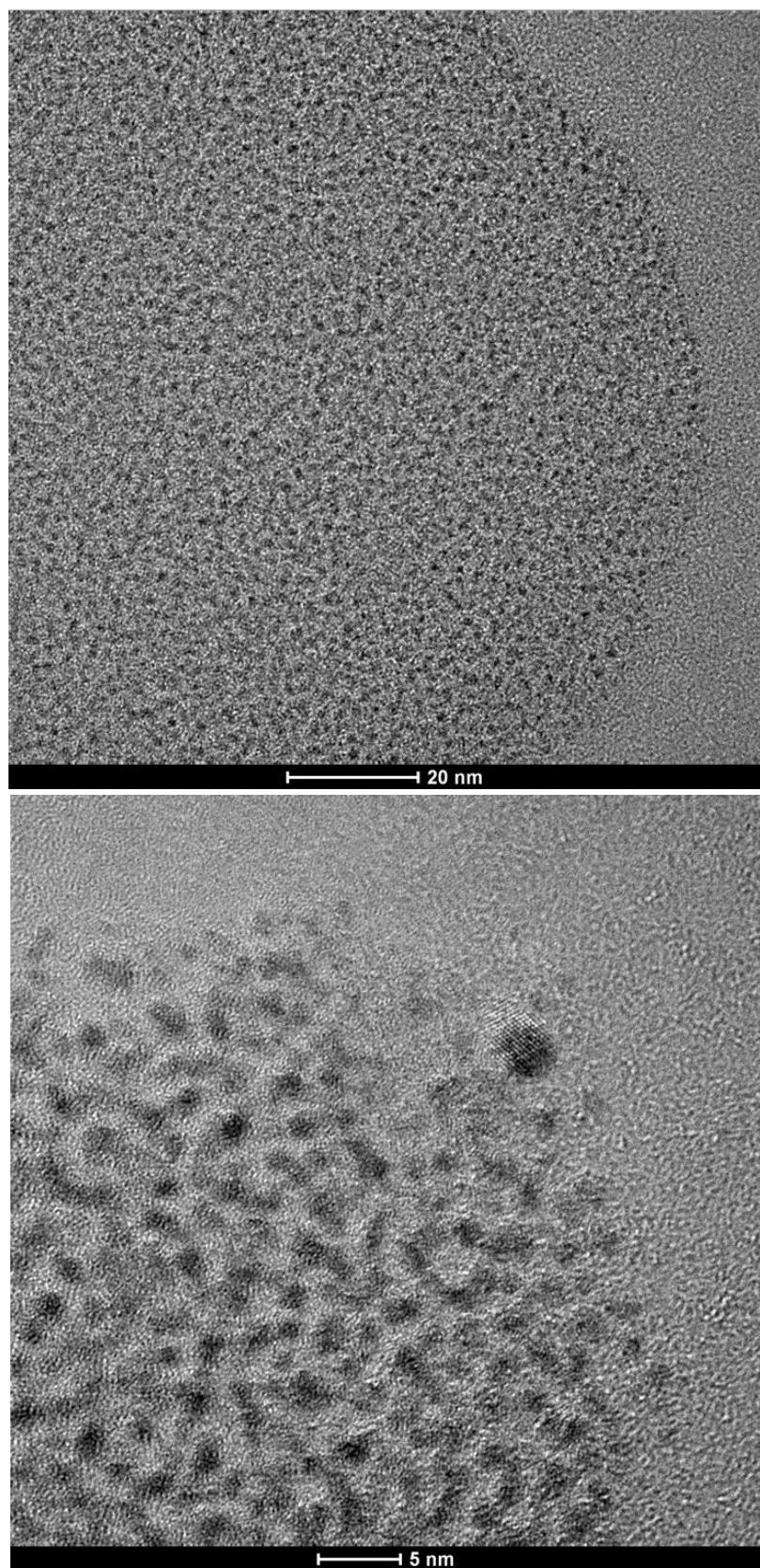


Figure S14. TEM images of Au/PdNAs-3

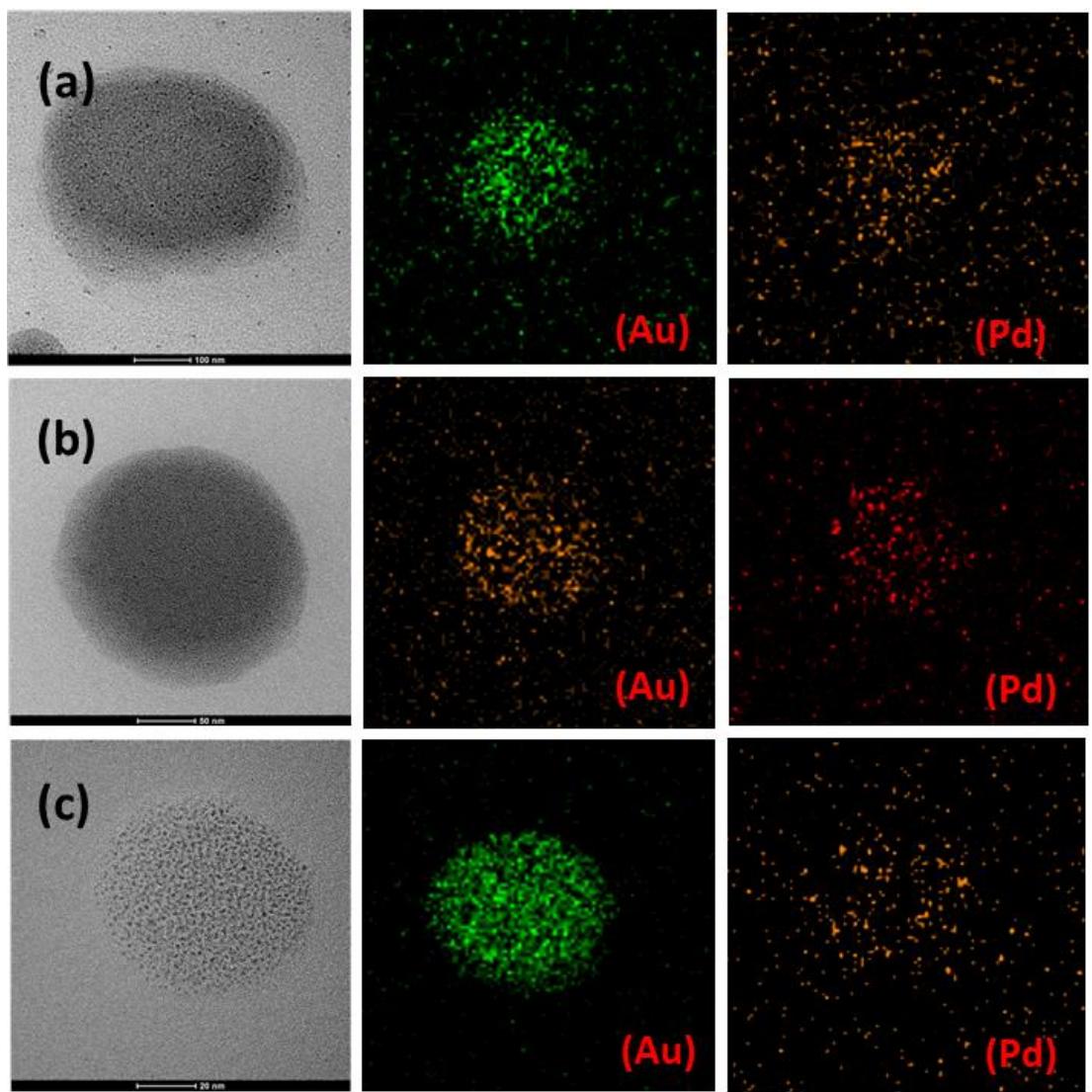


Figure S15. The STEM element-mappings of (a) Au/Pd NAs-1, (b) Au/Pd NAs-2, (c) Au/Pd NAs-3

10. ATR FT-IR Spectra

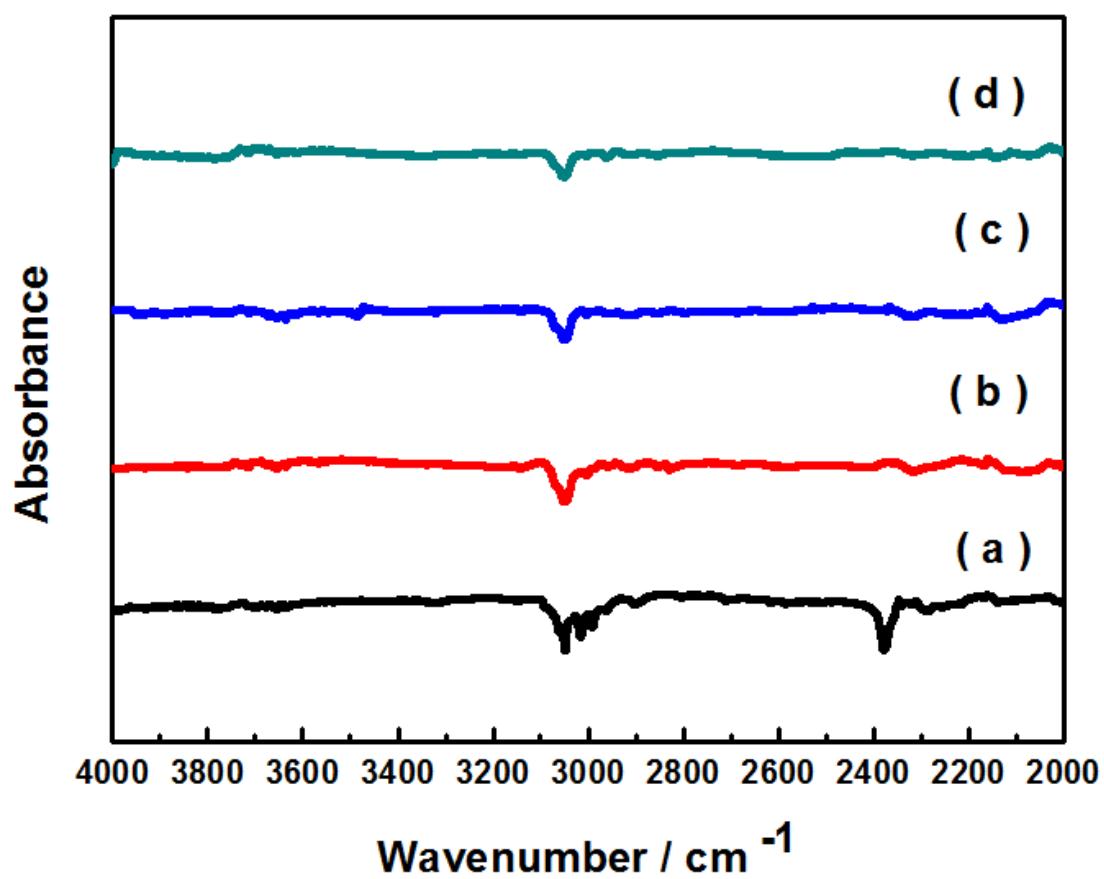


Figure S16. ATR FT-IR spectra: **a** (Diphenylphosphine Oxides), **b** (SPOs-PdNPs), **c** (SPOs-AuNPs), **d** (Au/PdNAs-2).

11. UV-Vis Spectra

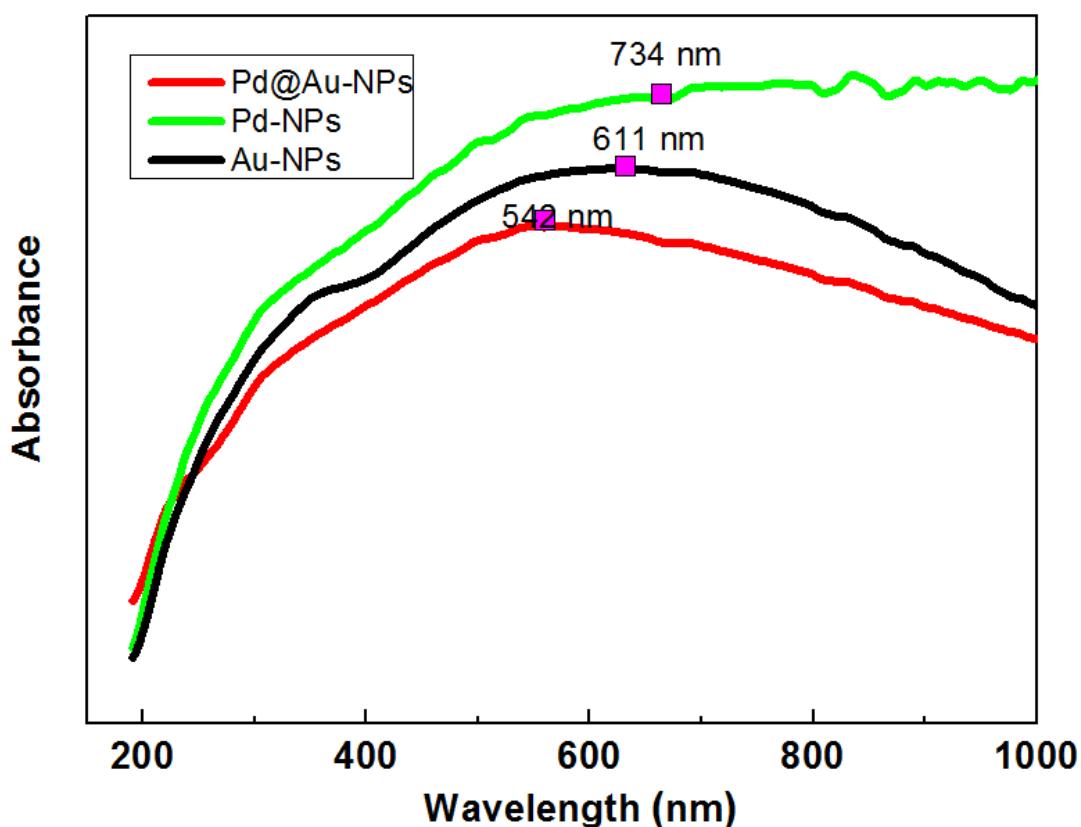


Figure S17. UV spectra of SPOs-PdNPs, SPOs-AuNPs and Au/PdNAs-2.

12. XRD Spectra for SPOs-PdNPs, SPOs-AuNPs and Au/PdNAS-2

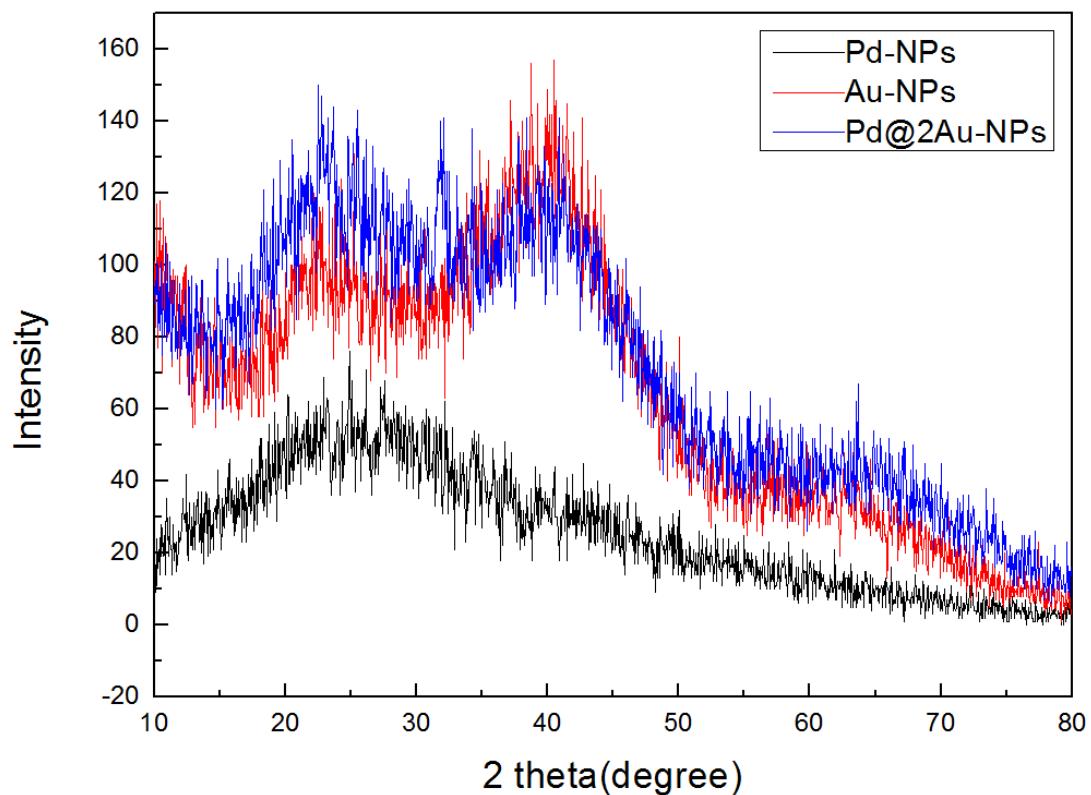
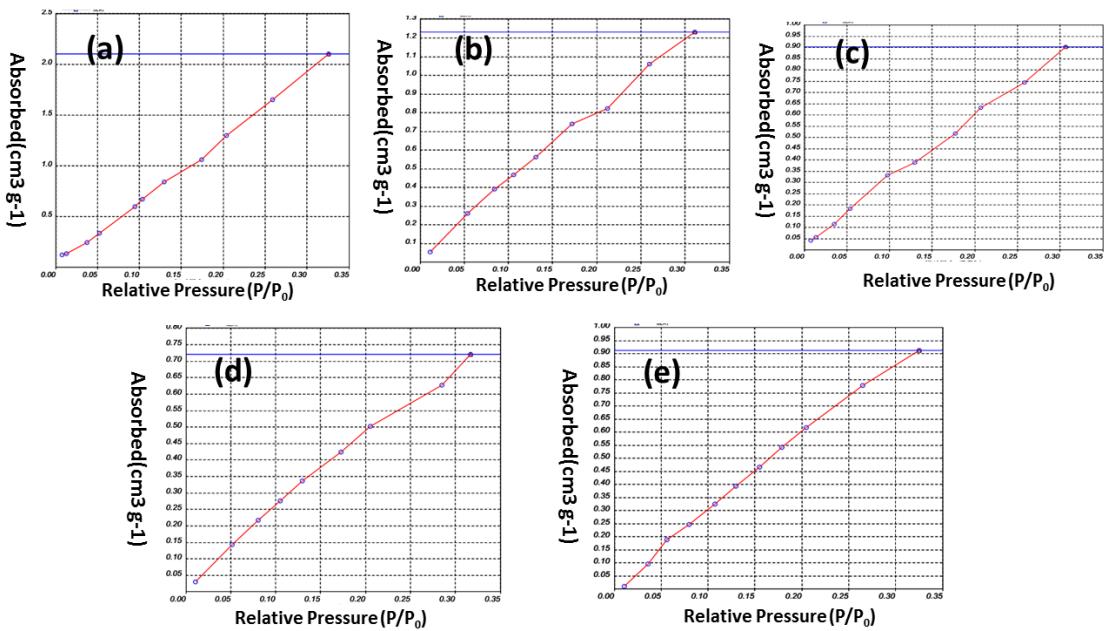


Figure S18. XRD spectra of SPOs-PdNPs, SPOs-AuNPs and Au/PdNAS-2.

13. BET Measurements



samples	temperature (°C)/time (h)	BET (m ² /g)
SPO-PdNPs	50/2	11.3
SPO-AuNPs	50/2	5.2
Au/PdNAs-1	50/2	4.2
Au/PdNAs-2	50/2	3.5
Au/PdNAs-3	50/2	4.8

Figure S19. BET surface area of (a) SPO-PdNPs, (b) SPO-AuNPs, (c) Au/PdNAs-1, (d) Au/PdNAs-2, and (e) Au/PdNAs-3

14. X-ray Crystal Structure of 2b

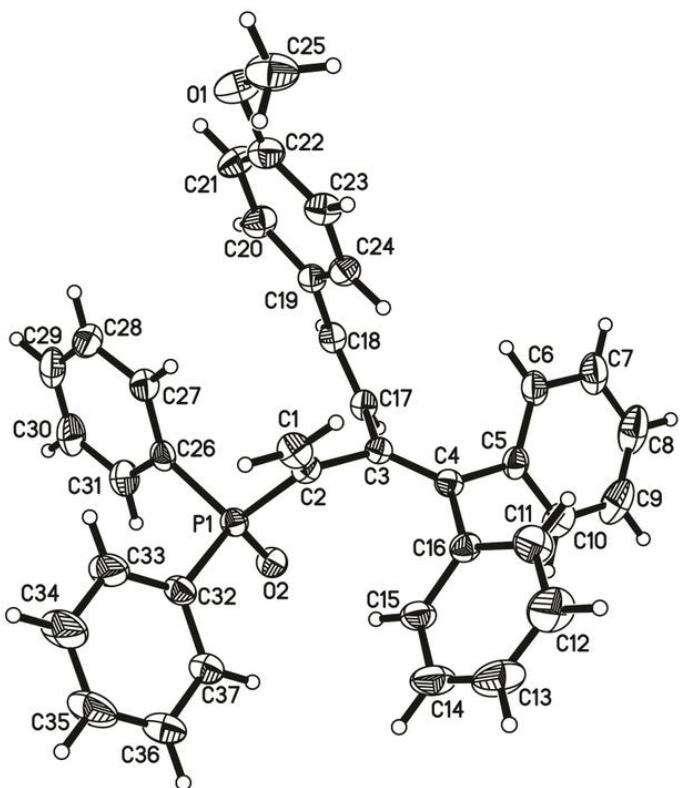


Table 1. Crystal data and structure refinement for $C_{37} H_{31} O_2 P$ (**2b**).

Empirical formula	$C_{37} H_{31} O_2 P$	
Formula weight	538.59	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	$a = 10.2210(6)$ Å	$\alpha = 82.845(2)^\circ$.
	$b = 11.6331(6)$ Å	$\beta = 74.362(2)^\circ$.
	$c = 13.5866(7)$ Å	$\gamma = 72.425(2)^\circ$.
Volume	$1481.42(14)$ Å ³	
Z	2	
Density (calculated)	1.207 Mg/m ³	
Absorption coefficient	0.124 mm ⁻¹	
F(000)	568	
Crystal size	0.200 x 0.160 x 0.120 mm ³	
Theta range for data collection	2.156 to 27.535°.	
Index ranges	-13 ≤ h ≤ 13, -15 ≤ k ≤ 15, -17 ≤ l ≤ 17	

Reflections collected	22063
Independent reflections	6765 [$R(\text{int}) = 0.0366$]
Completeness to theta = 25.242°	98.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7456 and 0.6492
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6765 / 0 / 362
Goodness-of-fit on F^2	1.012
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0536$, $wR_2 = 0.1209$
R indices (all data)	$R_1 = 0.0988$, $wR_2 = 0.1402$
Extinction coefficient	n/a
Largest diff. peak and hole	0.182 and -0.275 e. \AA^{-3}

Table 2. Bond lengths [Å] and angles [°] for C₃₇ H₃₁ O₂ P (**2b**).

C(1)-C(2)	1.321(2)
C(1)-H(1A)	0.9300
C(1)-H(1B)	0.9300
C(2)-C(3)	1.496(2)
C(2)-P(1)	1.8177(19)
C(3)-C(4)	1.341(2)
C(3)-C(17)	1.492(2)
C(4)-C(5)	1.493(3)
C(4)-C(16)	1.496(2)
C(5)-C(10)	1.384(3)
C(5)-C(6)	1.387(3)
C(6)-C(7)	1.383(3)
C(6)-H(6)	0.9300
C(7)-C(8)	1.355(4)
C(7)-H(7)	0.9300
C(8)-C(9)	1.354(4)
C(8)-H(8)	0.9300
C(9)-C(10)	1.392(4)
C(9)-H(9)	0.9300
C(10)-H(10)	0.9300
C(11)-C(16)	1.376(3)
C(11)-C(12)	1.392(3)
C(11)-H(11)	0.9300
C(12)-C(13)	1.365(4)
C(12)-H(12)	0.9300
C(13)-C(14)	1.349(4)
C(13)-H(13)	0.9300
C(14)-C(15)	1.371(3)
C(14)-H(14)	0.9300
C(15)-C(16)	1.379(3)
C(15)-H(15)	0.9300
C(17)-C(18)	1.328(3)
C(17)-H(17)	0.9300
C(18)-C(19)	1.457(3)
C(18)-H(18)	0.9300
C(19)-C(24)	1.389(3)

C(19)-C(20)	1.397(3)
C(20)-C(21)	1.374(3)
C(20)-H(20)	0.9300
C(21)-C(22)	1.380(3)
C(21)-H(21)	0.9300
C(22)-O(1)	1.370(3)
C(22)-C(23)	1.377(3)
C(23)-C(24)	1.378(3)
C(23)-H(23)	0.9300
C(24)-H(24)	0.9300
C(25)-O(1)	1.434(3)
C(25)-H(25A)	0.9600
C(25)-H(25B)	0.9600
C(25)-H(25C)	0.9600
C(26)-C(27)	1.375(3)
C(26)-C(31)	1.392(3)
C(26)-P(1)	1.801(2)
C(27)-C(28)	1.395(3)
C(27)-H(27)	0.9300
C(28)-C(29)	1.365(4)
C(28)-H(28)	0.9300
C(29)-C(30)	1.358(4)
C(29)-H(29)	0.9300
C(30)-C(31)	1.364(3)
C(30)-H(30)	0.9300
C(31)-H(31)	0.9300
C(32)-C(37)	1.376(3)
C(32)-C(33)	1.377(3)
C(32)-P(1)	1.8108(19)
C(33)-C(34)	1.381(3)
C(33)-H(33)	0.9300
C(34)-C(35)	1.360(4)
C(34)-H(34)	0.9300
C(35)-C(36)	1.360(4)
C(35)-H(35)	0.9300
C(36)-C(37)	1.379(3)
C(36)-H(36)	0.9300
C(37)-H(37)	0.9300

O(2)-P(1)	1.4771(13)
C(2)-C(1)-H(1A)	120.0
C(2)-C(1)-H(1B)	120.0
H(1A)-C(1)-H(1B)	120.0
C(1)-C(2)-C(3)	124.41(17)
C(1)-C(2)-P(1)	121.96(15)
C(3)-C(2)-P(1)	113.62(12)
C(4)-C(3)-C(17)	121.60(16)
C(4)-C(3)-C(2)	122.91(15)
C(17)-C(3)-C(2)	114.73(14)
C(3)-C(4)-C(5)	120.99(16)
C(3)-C(4)-C(16)	123.84(17)
C(5)-C(4)-C(16)	115.05(16)
C(10)-C(5)-C(6)	118.0(2)
C(10)-C(5)-C(4)	119.94(19)
C(6)-C(5)-C(4)	122.05(19)
C(7)-C(6)-C(5)	120.5(2)
C(7)-C(6)-H(6)	119.7
C(5)-C(6)-H(6)	119.7
C(8)-C(7)-C(6)	120.6(3)
C(8)-C(7)-H(7)	119.7
C(6)-C(7)-H(7)	119.7
C(9)-C(8)-C(7)	119.9(3)
C(9)-C(8)-H(8)	120.0
C(7)-C(8)-H(8)	120.0
C(8)-C(9)-C(10)	120.7(3)
C(8)-C(9)-H(9)	119.7
C(10)-C(9)-H(9)	119.7
C(5)-C(10)-C(9)	120.2(3)
C(5)-C(10)-H(10)	119.9
C(9)-C(10)-H(10)	119.9
C(16)-C(11)-C(12)	120.5(2)
C(16)-C(11)-H(11)	119.7
C(12)-C(11)-H(11)	119.7
C(13)-C(12)-C(11)	120.0(2)
C(13)-C(12)-H(12)	120.0
C(11)-C(12)-H(12)	120.0
C(14)-C(13)-C(12)	120.0(2)

C(14)-C(13)-H(13)	120.0
C(12)-C(13)-H(13)	120.0
C(13)-C(14)-C(15)	120.2(3)
C(13)-C(14)-H(14)	119.9
C(15)-C(14)-H(14)	119.9
C(14)-C(15)-C(16)	121.7(2)
C(14)-C(15)-H(15)	119.2
C(16)-C(15)-H(15)	119.2
C(11)-C(16)-C(15)	117.54(19)
C(11)-C(16)-C(4)	121.17(19)
C(15)-C(16)-C(4)	121.19(17)
C(18)-C(17)-C(3)	129.36(17)
C(18)-C(17)-H(17)	115.3
C(3)-C(17)-H(17)	115.3
C(17)-C(18)-C(19)	130.02(17)
C(17)-C(18)-H(18)	115.0
C(19)-C(18)-H(18)	115.0
C(24)-C(19)-C(20)	116.36(18)
C(24)-C(19)-C(18)	123.76(17)
C(20)-C(19)-C(18)	119.83(17)
C(21)-C(20)-C(19)	121.4(2)
C(21)-C(20)-H(20)	119.3
C(19)-C(20)-H(20)	119.3
C(20)-C(21)-C(22)	121.0(2)
C(20)-C(21)-H(21)	119.5
C(22)-C(21)-H(21)	119.5
O(1)-C(22)-C(23)	124.8(2)
O(1)-C(22)-C(21)	116.4(2)
C(23)-C(22)-C(21)	118.8(2)
C(22)-C(23)-C(24)	120.0(2)
C(22)-C(23)-H(23)	120.0
C(24)-C(23)-H(23)	120.0
C(23)-C(24)-C(19)	122.44(19)
C(23)-C(24)-H(24)	118.8
C(19)-C(24)-H(24)	118.8
O(1)-C(25)-H(25A)	109.5
O(1)-C(25)-H(25B)	109.5
H(25A)-C(25)-H(25B)	109.5

O(1)-C(25)-H(25C)	109.5
H(25A)-C(25)-H(25C)	109.5
H(25B)-C(25)-H(25C)	109.5
C(27)-C(26)-C(31)	118.52(19)
C(27)-C(26)-P(1)	125.07(16)
C(31)-C(26)-P(1)	116.40(16)
C(26)-C(27)-C(28)	119.7(2)
C(26)-C(27)-H(27)	120.2
C(28)-C(27)-H(27)	120.2
C(29)-C(28)-C(27)	120.4(2)
C(29)-C(28)-H(28)	119.8
C(27)-C(28)-H(28)	119.8
C(28)-C(29)-C(30)	120.1(2)
C(28)-C(29)-H(29)	119.9
C(30)-C(29)-H(29)	119.9
C(31)-C(30)-C(29)	120.3(2)
C(31)-C(30)-H(30)	119.9
C(29)-C(30)-H(30)	119.9
C(30)-C(31)-C(26)	121.0(2)
C(30)-C(31)-H(31)	119.5
C(26)-C(31)-H(31)	119.5
C(37)-C(32)-C(33)	118.99(19)
C(37)-C(32)-P(1)	118.03(15)
C(33)-C(32)-P(1)	122.88(17)
C(34)-C(33)-C(32)	120.1(2)
C(34)-C(33)-H(33)	120.0
C(32)-C(33)-H(33)	120.0
C(35)-C(34)-C(33)	120.1(3)
C(35)-C(34)-H(34)	119.9
C(33)-C(34)-H(34)	119.9
C(34)-C(35)-C(36)	120.5(2)
C(34)-C(35)-H(35)	119.8
C(36)-C(35)-H(35)	119.8
C(35)-C(36)-C(37)	119.9(2)
C(35)-C(36)-H(36)	120.1
C(37)-C(36)-H(36)	120.1
C(32)-C(37)-C(36)	120.5(2)
C(32)-C(37)-H(37)	119.8

C(36)-C(37)-H(37)	119.8
C(22)-O(1)-C(25)	116.9(2)
O(2)-P(1)-C(26)	112.50(9)
O(2)-P(1)-C(32)	111.66(8)
C(26)-P(1)-C(32)	104.66(9)
O(2)-P(1)-C(2)	110.98(8)
C(26)-P(1)-C(2)	108.99(9)
C(32)-P(1)-C(2)	107.76(9)

Symmetry transformations used to generate equivalent atoms:

Table 3. Hydrogen bonds for C₃₇H₃₁O₂P (**2b**).

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(37)-H(37)...O(2) ^{#1}	0.93	2.45	3.223(3)	140.0

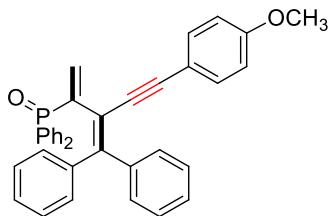
Symmetry transformations used to generate equivalent atoms: #1 -x,-y+1,-z+1

15. Characterizations of Substrates and Catalytic Products

15.1 Substrates

These substrates (**1b-1m**) are new compounds, for known compounds, see our previous work: *Adv. Synth. Catal.*, **2018**, *360*, 3518.

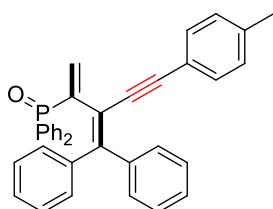
(3-(diphenylmethylene)-5-(4-methoxyphenyl)pent-1-en-4-yn-2yl)diphenylphosphine oxide (1b)



1b

A white solid (472 mg, 88 % yield), *m.p.*: 193.2 – 194.7 °C. **¹H NMR** (400 MHz, CDCl₃) δ 7.90 – 7.73 (m, 4H), 7.58 – 7.38 (m, 8H), 7.37 – 7.15 (m, 6H), 7.12 – 7.03 (m, 2H), 6.77 – 6.51 (m, 4H), 6.24 – 5.88 (m, 2H), 3.77 (s, 3H). **¹³C NMR** (101 MHz, CDCl₃) δ 159.5, 151.3 (d, *J* = 7.1 Hz), 142.9, 141.9, 141.7, 140.7, 136.0 (d, *J* = 7.7 Hz), 132.7, 132.3, 132.2, 131.9, 131.8, 131.6, 130.4, 130.2, 128.4, 128.3, 127.9, 127.5, 127.4, 118.1, 118.0, 115.2, 113.5, 96.2, 88.7, 55.3. **³¹P NMR** (162 MHz, CDCl₃) δ 27.94 (s). **HRMS (ESI):** ([M+H]⁺) Calcd for C₃₇H₃₀O₂P⁺: 537.1978, Found: 537.1975. **IR (film) v** 3064, 3010, 2921, 2839, 2192, 1912, 1604, 1508, 1464, 1435, 1249, 1209, 1190, 1158, 1022, 832, 693, 562 cm⁻¹.

(3-(diphenylmethylene)-5-(p-tolyl)pent-1-en-4-yn-2yl)diphenylphosphine oxide (1c)

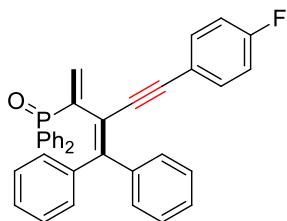


1c

A white solid (447 mg, 86% yield), *m.p.*: 181.6 – 183.4 °C. **¹H NMR** (400 MHz, CDCl₃) δ 7.90 – 7.73 (m, 4H), 7.60 – 7.40 (m, 8H), 7.37 – 7.15 (m, 6H), 7.17 – 7.02 (m, 2H), 6.96 (d, *J* = 8.0 Hz, 2H), 6.60 (d, *J* = 7.9 Hz, 2H), 6.07 (t, *J* = 28.5 Hz, 2H), 2.30 (s, 3H). **¹³C NMR** (101 MHz, CDCl₃) δ 151.8 (d, *J* = 7.0 Hz), 142.9, 142.0, 141.6, 140.6, 138.2, 136.0 (d, *J* = 7.7 Hz), 132.6, 132.2, 132.1,

131.9, 131.8, 131.6, 131.1, 130.3, 130.2, 128.7, 128.4, 128.3, 127.7 (d, $J = 51.5$ Hz), 127.8, 127.5, 118.0, 117.9, 96.2, 89.3, 21.5. **^{31}P NMR** (162 MHz, CDCl_3) δ 27.77 (s). **HRMS (ESI)**: ([$\text{M}+\text{H}]^+$) Calcd for $\text{C}_{37}\text{H}_{30}\text{OP}^+$: 521.2029, Found: 521.2026. **IR** (film) ν 3317, 3055, 2219, 1911, 1597, 1509, 1436, 1180, 705, 557 cm^{-1} .

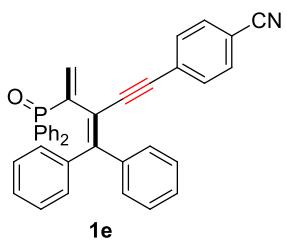
(3-(diphenylmethylene)-5-(4-fluorophenyl)pent-1-en-4-yn-2-yl)diphenylphosphine oxide (1d)



1d

A white solid (456 mg, 87% yield), m.p.: 173.8 – 175.0 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.87 – 7.70 (m, 4H), 7.60 – 7.50 (m, 2H), 7.47 – 7.35 (m, 6H), 7.35 – 7.28 (m, 3H), 7.28 – 7.20 (m, 3H), 7.20 – 7.09 (m, 2H), 6.83 (t, $J = 8.7$ Hz, 2H), 6.67 – 6.55 (m, 2H), 6.06 (d, $J = 40$, 1H), 5.93 (d, $J = 18.3$ Hz, 1H). **^{13}C NMR** (101 MHz, CDCl_3) δ 162.3 (d, $J = 250.5$ Hz), 152.5, 142.9, 141.9, 141.5, 140.6, 136.0 (d, $J = 8.5$ Hz), 133.0 (d, $J = 8.1$ Hz), 132.2, 132.1, 132.0 (d, $J = 104.0$ Hz), 131.9 (d, $J = 12.1$ Hz), 131.6, 130.3, 130.2, 128.4, 128.3, 128.1, 127.9, 127.6, 119.1 (d, $J = 3.4$ Hz), 117.8 (d, $J = 8.8$ Hz), 115.3, 115.0, 94.8, 89.5. **^{31}P NMR** (162 MHz, CDCl_3) δ 27.81 (s). **HRMS (ESI)**: ([$\text{M}+\text{H}]^+$) Calcd for $\text{C}_{36}\text{H}_{27}\text{FOP}^+$: 525.1778, Found: 525.1767. **IR** (film) ν 3052, 1896, 1597, 1503, 1435, 1209, 1115, 829, 711, 617 cm^{-1} .

4-(3-(diphenylmethylene)-4-(diphenylphosphoryl)pent-4-en-1-yn-1-yl)benzonitrile(1e)

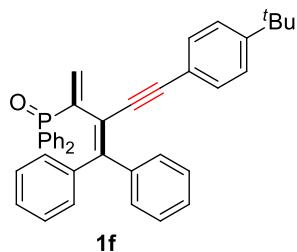


1e

A white solid (419 mg, 79% yield), m.p.: 191.0 – 193.0 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.91 – 7.69 (m, 4H), 7.54 (t, $J = 7.3$ Hz, 2H), 7.50 – 7.35 (m, 8H), 7.35 – 7.13 (m, 8H), 6.65 (d, $J = 8.3$ Hz, 2H), 6.03 (d, $J = 38.8$ Hz, 1H), 5.82 (d, $J = 18.3$ Hz, 1H). **^{13}C NMR** (101 MHz, CDCl_3) δ 154.68 (d, $J = 6.8$ Hz), 142.6, 141.7, 141.3, 140.3, 136.19 (d, $J = 8.8$ Hz), 132.5, 132.1, 132.0, 131.9 (d, $J = 2.0$

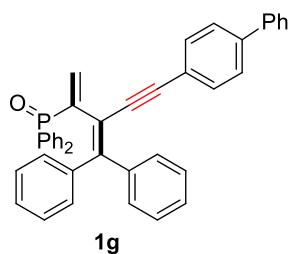
Hz), 131.6, 131.5, 131.4, 130.3, 130.2, 128.5, 128.4, 128.3, 128.0, 127.9, 127.8, 127.7, 118.6, 117.3 (d, J = 8.6 Hz), 111.0, 94.1, 93.8. ^{31}P NMR (162 MHz, CDCl_3) δ 27.98 (s). HRMS (ESI): ([M+H] $^+$) Calcd for $\text{C}_{37}\text{H}_{27}\text{NOP}^+$: 532.1825, Found: 532.1823. IR (film) ν 3055, 2225, 2187, 1679, 1603, 1445, 1435, 1208, 1177, 946, 725, 635 cm^{-1} .

(5-(4-(tert-butyl)phenyl)-3-(diphenylmethylene)pent-1-en-4-yn-2-yl)diphenylphosphine oxide (1f)



A white solid (461 mg, 82% yield), m.p.: 168.2 – 170.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.96 – 7.75 (m, 4H), 7.60 – 7.40 (m, 8H), 7.38 – 7.15 (m, 8H), 7.14 – 7.00 (m, 2H), 6.68 (d, J = 8.4 Hz, 2H), 6.10 (dd, J = 28.0, 20.6 Hz, 2H), 1.29 (s, 9H). ^{13}C NMR (101 MHz, CDCl_3) δ 151.9 (d, J = 7.0 Hz), 151.4, 142.9, 142.0, 141.6, 140.6, 136.1 (d, J = 7.9 Hz), 132.6, 132.3, 132.1, 131.9, 131.8, 131.6, 131.0, 130.4, 130.2, 128.4, 128.3, 127.9, 127.7 (d, J = 50.5 Hz), 127.6, 124.9, 120.1, 118.0 (d, J = 9.0 Hz), 96.3, 89.4, 34.8, 31.2. ^{31}P NMR (162 MHz, CDCl_3) δ 27.74 (s). HRMS (ESI): ([M+H] $^+$) Calcd for $\text{C}_{40}\text{H}_{36}\text{OP}^+$: 563.2498, Found: 563.2498. IR (film) ν 3055, 2958, 2899, 2194, 1887, 1461, 1436, 1213, 1190, 1148, 946, 713, 693, 573 cm^{-1} .

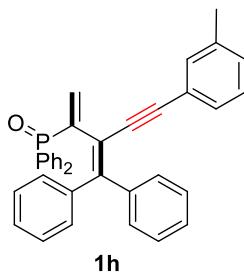
(5-([1,1'-biphenyl]-4-yl)-3-(diphenylmethylene)pent-1-en-4-yn-2-yl)diphenylphosphine oxide (1g)



A white solid (512 mg, 88% yield), m.p.: 172.4 – 174.2 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.99 – 7.80 (m, 4H), 7.65 – 7.54 (m, 4H), 7.53 – 7.40 (m, 10H), 7.39 – 7.31 (m, 4H), 7.31 – 7.22 (m, 3H), 7.20 – 7.10 (m, 2H), 6.79 (d, J = 8.3 Hz, 2H), 6.13 (d, J = 38.2 Hz, 1H), 6.04 (d, J = 18.3 Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 152.45 (d, J = 6.9 Hz), 142.9, 142.0, 141.6, 140.8, 140.6, 140.3, 136.1 (d, J = 8.4 Hz), 132.7, 132.3, 132.2, 132.0, 131.9, 131.6, 130.4, 130.3, 128.9, 128.5, 128.4,

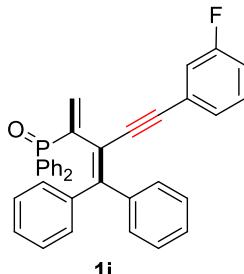
128.1, 128.0, 127.7, 127.6, 127.5, 127.0, 126.6, 122.0, 117.92 (d, $J = 8.8$ Hz), 96.0, 90.7. **^{31}P NMR** (162 MHz, CDCl_3) δ 27.87 (s). **HRMS (ESI)**: ([M+H] $^+$) Calcd for $\text{C}_{42}\text{H}_{32}\text{OP}^+$: 583.2185, Found: 583.2183. **IR** (film) ν 3052, 2222, 1902, 1597, 1484, 1437, 1210, 1178, 1117, 968, 840, 715, 617, 556 cm^{-1} .

(3-(diphenylmethylene)-5-(m-tolyl)pent-1-en-4-yn-2-yl)diphenylphosphine oxide (1h)



A white solid (452 mg, 87% yield), m.p.: 184.8 – 186.5 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.91 – 7.67 (m, 6H), 7.61 – 7.37 (m, 8H), 7.37 – 7.11 (m, 8H), 6.67 (d, $J = 8.3$ Hz, 2H), 5.98 (dd, $J = 59.8$, 28.3 Hz, 2H), 3.88 (s, 3H). **^{13}C NMR** (101 MHz, CDCl_3) δ 166.5, 153.8 (d, $J = 6.9$ Hz), 142.7, 141.8, 141.3, 140.4, 136.1 (d, $J = 8.0$ Hz), 132.5, 132.2, 132.1, 131.9 (d, $J = 2.0$ Hz), 131.5, 131.0, 130.3, 130.2, 129.1, 129.0, 128.5, 128.4, 128.3, 127.9, 127.7 (d, $J = 8.1$ Hz), 127.6, 117.51 (d, $J = 8.7$ Hz), 94.9, 92.8, 52.2. **^{31}P NMR** (162 MHz, CDCl_3) δ 27.93 (s). **HRMS (ESI)**: ([M+H] $^+$) Calcd for $\text{C}_{37}\text{H}_{30}\text{OP}^+$: 521.2029, Found: 521.2027. **IR** (film) ν 3639, 3422, 3053, 2953, 1909, 1718, 1604, 1436, 1309, 1188, 1113, 791, 961, 643 cm^{-1} .

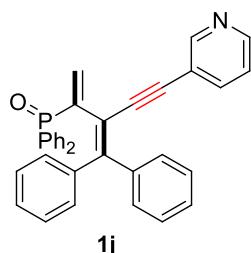
(3-(diphenylmethylene)-5-(3-fluorophenyl)pent-1-en-4-yn-2-yl)diphenylphosphine oxide (1i)



A white solid (393 mg, 75 % yield), m.p.: 116.1 – 118.2 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.89 – 7.71 (m, 4H), 7.60 – 7.50 (m, 2H), 7.50 – 7.37 (m, 6H), 7.35 – 7.20 (m, 6H), 7.23 – 7.12 (m, 2H), 7.11 – 7.01 (m, 1H), 6.95 – 6.80 (m, 1H), 6.48 (d, $J = 7.7$ Hz, 1H), 6.27 (d, $J = 8.0$ Hz, 1H), 5.99 (dd, $J = 54.0$, 28.4 Hz, 2H). **^{13}C NMR** (101 MHz, CDCl_3) δ 163.2, 160.8, 153.3 (d, $J = 7.0$ Hz), 142.3 (d, $J = 94.9$ Hz), 141.4, 140.5, 136.1 (d, $J = 8.2$ Hz), 132.5, 132.2, 132.1, 132.0 (d, $J = 6.1$

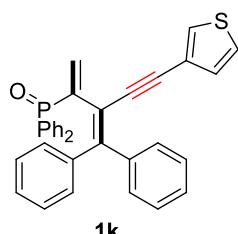
Hz), 131.5, 130.3, 130.2, 129.4 (d, J = 8.7 Hz), 128.5, 128.4, 128.0 (d, J = 56.6 Hz), 127.9, 127.6, , 127.0 (d, J = 6.1 Hz), 124.8 (d, J = 9.7 Hz), 117.8 (d, J = 23.2 Hz), 117.5 (d, J = 9.1 Hz), 115.3 (d, J = 21 Hz), 94.5, 90.7. ^{31}P NMR (162 MHz, CDCl_3) δ 27.87 (s). HRMS (ESI): ([M+H] $^+$) Calcd for $\text{C}_{36}\text{H}_{27}\text{FOP}^+$: 525.1778, Found: 525.1774. IR (film) ν 3054, 2198, 1605, 1574, 1484, 1435, 1310, 1214, 1188, 1116, 998, 874, 755, 711, 680, 617 cm^{-1} .

(3-(diphenylmethylene)-5-(pyridin-3-yl)pent-1-en-4-yn-2-yl)diphenylphosphine oxide (1j)



A white solid (426 mg, 84% yield), m.p.: 186.4 – 187.1 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.43 – 8.29 (d, J = 4.0 Hz, 1H), 7.95 – 7.71 (m, 5H), 7.59 – 7.37 (m, 8H), 7.35 – 7.14 (m, 8H), 7.10 – 6.95 (m, 1H), 6.96 – 6.81 (m, 1H), 6.02 (d, J = 38.6 Hz, 1H), 5.86 (d, J = 18.3 Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 153.8 (d, J = 6.9 Hz), 151.7, 148.2, 142.7, 141.8, 141.3, 140.4, 137.9, 136.1 (d, J = 8.6 Hz), 131.9 (d, J = 104 Hz), 132.2, 132.1, 132.0 (d, J = 4.4 Hz), 131.4, 130.3, 130.1, 128.5, 128.4, 128.3, 128.0, 127.7, 127.6, 122.6, 120.1, 117.41 (d, J = 8.7 Hz), 92.9, 92.3. ^{31}P NMR (162 MHz, CDCl_3) δ 27.83 (s). HRMS (ESI): ([M+H] $^+$) Calcd for $\text{C}_{35}\text{H}_{27}\text{NOP}^+$: 508.1825, Found: 508.1822. IR (film) ν 3053, 2922, 2219, 1588, 1475, 1436, 1403, 1209, 1180, 1116, 1022, 996.44, 900, 725, 694, 655, 630 cm^{-1} .

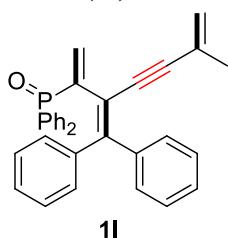
(3-(diphenylmethylene)-5-(thiophen-3-yl)pent-1-en-4-yn-2-yl)diphenylphosphine oxide (1k)



A white solid (425 mg, 83% yield), m.p.: 190.6 – 192.1 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.98 – 7.70 (m, 4H), 7.65 – 7.37 (m, 8H), 7.35 – 7.15 (m, 5H), 7.16 – 7.00 (m, 3H), 6.81 – 6.33 (m, 2H), 6.02 (t, J = 29.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 152.02 (d, J = 7.0 Hz), 142.8, 141.9, 141.5,

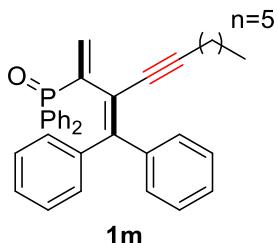
140.6, 136.0 (d, $J = 8.1$ Hz), 132.6, 132.2, 132.1, 131.9, 131.8, 131.3, 130.1, 129.4, 128.5, 128.4, 128.3, 128.0, 127.9, 127.6, 127.5, 124.8, 122.0, 117.8 (d, $J = 8.9$ Hz), 91.4, 89.28 (d, $J = 2.3$ Hz). ^{31}P NMR (162 MHz, CDCl₃) δ 27.76 (s). HRMS (ESI): ([M+H]⁺) Calcd for C₃₄H₂₆OPS⁺: 513.1436, Found: 513.1437. IR (film) ν 3055, 2195, 1824, 1589, 1489, 1435, 1386, 1213, 1188, 1148, 958, 906, 726, 692, 620 cm⁻¹.

(3-(diphenylmethylene)-6-methylhepta-1,6-dien-4-yn-2-yl)diphenylphosphine oxide (1l)



A white solid (348 mg, 74 % yield), m.p.: 113.2 – 114.7 °C. ^1H NMR (400 MHz, CDCl₃) δ 7.83 – 7.69 (m, 4H), 7.58 – 7.50 (m, 2H), 7.49 – 7.40 (m, 4H), 7.40 – 7.30 (m, 2H), 7.28 – 7.18 (m, 6H), 7.14 – 6.98 (m, 2H), 6.04 (d, $J = 38.2$, 1H), 5.93 (d, $J = 18.3$, 1H), 5.04 – 4.87 (m, 1H), 4.56 (d, $J = 0.9$ Hz, 1H), 1.44 (s, 3H). ^{13}C NMR (101 MHz, CDCl₃) δ 152.30 (d, $J = 6.9$ Hz), 142.9, 141.9, 141.5, 140.6, 135.9 (d, $J = 7.9$ Hz), 132.6, 132.2, 132.1, 131.9, 131.8, 131.5, 130.3, 130.0, 128.3, 128.2, 127.8, 127.6 (d, $J = 45.5$ Hz), 127.5, 126.6, 121.8, 117.8, 97.0, 88.8, 22.4. ^{31}P NMR (162 MHz, CDCl₃) δ 27.93 (s). HRMS (ESI): ([M+H]⁺) Calcd for C₃₃H₂₈OP⁺: 471.1872, Found: 471.1869. IR (film) ν 3054, 2919, 2847, 2223, 1810, 1600, 1490, 1436, 1182, 1116, 1097, 898, 791, 773, 691, 618, 574 cm⁻¹.

(3-(diphenylmethylene)undec-1-en-4-yn-2-yl)diphenylphosphine oxide (1m)

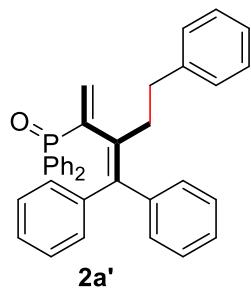


A white solid (432 mg, 84 % yield), m.p.: 99.9 – 101.7 °C. ^1H NMR (400 MHz, CDCl₃) δ 7.77 (dd, $J = 11.9, 7.2$ Hz, 4H), 7.53 – 7.45 (m, 2H), 7.44 – 7.40 (m, 4H), 7.37 – 7.32 (m, 2H), 7.20 (d, $J = 7.4$ Hz, 3H), 7.17 – 7.12 (m, 3H), 7.02 – 6.96 (m, 2H), 5.95 (d, $J = 26.0$ Hz, 1H), 5.88 (d, $J = 5.5$ Hz, 1H), 1.70 – 1.65 (m, 2H), 1.29 – 1.18 (m, 2H), 1.12 (d, $J = 19.3$ Hz, 6H), 0.87 (t, $J = 7.2$ Hz,

3H). **¹³C NMR** (101 MHz, CDCl₃) δ 150.3 (d, J = 7.1 Hz), 143.5, 142.6, 141.6, 140.8, 135.2 (d, J = 8.0 Hz), 132.7, 132.2, 132.1, 131.7, 130.2, 129.9, 128.2, 128.1, 127.8, 127.6, 127.4, 127.1, 118.39 (d, J = 8.8 Hz), 98.6, 80.6, 31.3, 28.4, 27.8, 22.5, 19.3, 14.2. **³¹P NMR** (162 MHz, CDCl₃) δ 27.62 (s). **HRMS (ESI)**: ([M+H]⁺) Calcd for: C₃₆H₃₆OP⁺: 515.2498, Found: 515.2501. **IR** (film) ν 3053, 2928, 2855, 2199, 1590, 1439, 1189, 1100, 948, 727, 691 cm⁻¹.

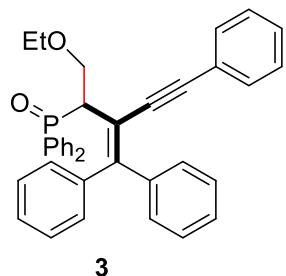
15.2 Catalytic Products

(3-(diphenylmethylene)-5-phenylpent-1-en-2-yl)diphenylphosphine oxide (2a')



A white solid (81.6 mg, 80% yield), *m.p.*: 93.4 – 95.1 °C. **¹H NMR** (400 MHz, CDCl₃) δ 7.73 – 7.65 (m, 4H), 7.60 – 7.52 (m, 2H), 7.50 – 7.44 (m, 4H), 7.30 – 7.20 (m, 8H), 7.17 – 7.05 (m, 5H), 6.61 (dd, J = 7.3, 1.8 Hz, 2H), 5.87 (dd, J = 41.9, 1.1 Hz, 1H), 5.66 (dd, J = 19.5, 1.1 Hz, 1H), 2.65 – 2.50 (m, 2H), 2.45 – 2.32 (m, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 145.4, 144.6, 143.4, 142.5, 142.0, 141.3, 137.3 (d, J = 2.5 Hz), 134.31 (d, J = 9.9 Hz), 132.4, 132.0, 131.9, 131.4, 130.0, 128.9, 128.5, 128.4, 128.2, 128.1, 128.0, 127.8, 127.7, 126.9, 126.4, 125.7, 35.2, 34.7. **³¹P NMR** (162 MHz, CDCl₃) δ 28.25 (s). **HRMS (ESI)**: ([M+H]⁺) Calcd for C₃₆H₃₂OP⁺: 511.2185, Found: 511.2175. **IR** (film) ν 3679, 2989, 2900, 2241, 1598, 1574, 1491, 1436, 1178, 1074, 917, 716, 695, cm⁻¹.

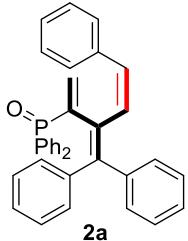
(3-(diphenylmethylene)-1-ethoxy-5-phenylpent-4-yn-2-yl)diphenylphosphine oxide (3)



A white solid (84 mg, 76% yield), *m.p.*: 96.4 – 97.1 °C. **¹H NMR** (400 MHz, CDCl₃) δ 7.76 – 7.65 (m, 2H), 7.63 – 7.54 (m, 2H), 7.53 – 7.47 (m, 2H), 7.46 – 7.35 (m, 9H), 7.35 – 7.29 (m, 3H), 7.26 – 7.18

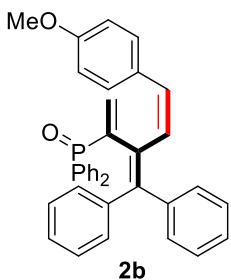
(m, 3H), 7.14 – 7.02 (m, 4H), 4.45 – 4.30 (m, 1H), 4.20 – 4.10 (m, 1H), 4.08 – 3.94 (m, 1H), 3.68 – 3.38 (m, 2H), 1.16 (t, J = 7.0 Hz, 3H). **^{13}C NMR** (101 MHz, CDCl_3) δ 151 (d, J = 6.1 Hz), 141.7, 140.5, 132.7, 131.7, 131.6, 131.1 (d, J = 8.1 Hz), 130.2, 130.0, 128.6 (d, J = 11.1 Hz), 128.2, 128.1, 128.0, 127.7, 127.4, 123.58, 116.1, 88.7, 67.8, 66.9, 46.0, 45.3, 15.1. **^{31}P NMR** (162 MHz, CDCl_3) δ 31.05 (s). **IR** (film) ν 3679, 2976, 2964, 2222, 1590, 1574, 1491, 1431, 1178, 1073, 916, 719, 696, cm^{-1} .

(Z)-(3-(diphenylmethylene)-5-phenylpenta-1,4-dien-2-yl)diphenylphosphine oxide (2a)



A white solid (96 mg, 94% yield), *m.p.*: 166.4 – 167.1 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.52 – 7.37 (m, 8H), 7.35 – 7.15 (m, 12H), 7.10 (s, 5H), 6.05 (d, J = 12.0 Hz, 1H), 5.95 (d, J = 12.1 Hz, 1H), 5.76 (d, J = 42.0 Hz, 1H), 5.35 (d, J = 19.4 Hz, 1H). **^{13}C NMR** (101 MHz, CDCl_3) δ 145.0 (d, J = 7.5 Hz), 143.5, 142.6, 142.3, 142.0, 136.8, 135.5 (d, J = 10.4 Hz), 133.6 (d, J = 7.1 Hz), 133.1, 132.4 (d, J = 103.0 Hz), 131.9, 131.8, 131.5 (d, J = 2.6 Hz), 130.4 (d, J = 85.8 Hz), 129.5, 129.3, 128.3, 128.2, 127.8, 127.7, 127.5, 127.3, 127.1, 126.9. **^{31}P NMR** (162 MHz, CDCl_3) δ 28.62 (s). **HRMS (ESI):** ([M+H] $^+$) Calcd for $\text{C}_{36}\text{H}_{30}\text{OP}^+$: 509.2029, Found: 509.2022. **IR** (film) ν 3675, 2988, 2901, 2221, 1590, 1574, 1491, 1436, 1178, 1072, 916, 719, 694, 596 cm^{-1} .

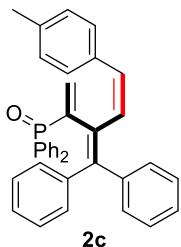
(Z)-(3-(diphenylmethylene)-5-(4-methoxyphenyl)penta-1,4-dien-2-yl)diphenylphosphine oxide (2b)



A white solid (103 mg, 96% yield) *m.p.*: 164.7 – 168.5 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.55 – 7.44 (m, 8H), 7.38 – 7.24 (m, 9H), 7.20 – 7.07 (m, 5H), 6.82 (d, J = 8.7 Hz, 2H), 5.99 (dd, J = 12.0, 1.7 Hz, 1H), 5.91 (d, J = 12.0 Hz, 1H), 5.79 (d, J = 42.1 Hz, 1H), 5.38 (d, J = 19.5 Hz, 1H), 3.82 (s, 3H). **^{13}C NMR** (101 MHz, CDCl_3) δ 158.8, 144.8 (d, J = 8.1 Hz), 143.0 (d, J = 94.9 Hz), 142.3, 142.1, 135.3

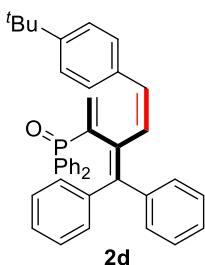
(d, $J = 10.3$ Hz), 133.9 (d, $J = 7.0$ Hz), 132.7, 132.4 (d, $J = 103.0$ Hz), 131.9, 131.8, 131.4, 130.6, 130.3 (d, $J = 89.9$ Hz), 129.7, 128.2, 128.1, 127.8, 127.6, 127.5, 127.0 (d, $J = 40.4$ Hz), 113.1, 55.3. ^{31}P NMR (162 MHz, CDCl₃) δ 28.72 (s). HRMS (ESI): ([M+H]⁺) Calcd for C₃₇H₃₂O₂P⁺: 539.2134, Found: 539.2128. IR (film) ν 3675, 2987, 2900, 1889, 1605, 1571, 1454, 1026, 945, 825, 696, 634 cm⁻¹.

(Z)-(3-(diphenylmethylene)-5-(p-tolyl)penta-1,4-dien-2-yl)diphenylphosphine oxide (2c)



A white solid (97 mg, 93% yield) *m.p.*: 119.4 – 121.1 °C. ^1H NMR (400 MHz, CDCl₃) δ 7.53 – 7.43 (m, 8H), 7.36 – 7.27 (m, 6H), 7.26 – 7.12 (m, 8H), 7.08 (d, $J = 7.9$ Hz, 2H), 6.04 (dd, $J = 12.1, 1.6$ Hz, 1H), 5.94 (d, $J = 12.1$ Hz, 1H), 5.78 (d, $J = 42.1$ Hz, 1H), 5.38 (d, $J = 19.5$ Hz, 1H), 2.36 (s, 3H). ^{13}C NMR (101 MHz, CDCl₃) δ 144.8, 143.2, 142.3, 142.0, 137.0, 135.4, 134.0, 133.8, 133.1, 133.0, 131.9, 131.8, 131.4, 130.3 (d, $J = 88.9$ Hz), 129.3, 128.6, 128.4, 128.2, 128.1, 127.8, 127.5, 127.0 (d, $J = 44.4$ Hz), 21.3. ^{31}P NMR (162 MHz, CDCl₃) δ 28.75 (s). HRMS (ESI): ([M+H]⁺) Calcd for C₃₇H₃₂OP⁺ : 523.2185, Found: 523.2183. IR (film) ν 3674, 2987, 2900, 2216, 1903, 1730, 1435, 1178, 1073, 923, 735, 692, 574 cm⁻¹.

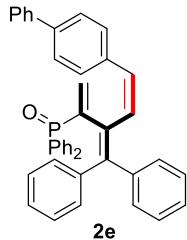
(Z)-(5-(4-(tert-butyl)phenyl)-3-(diphenylmethylene)penta-1,4-dien-2-yl)diphenylphosphine oxide (2d)



A white solid (106 mg, 94% yield) *m.p.*: 115.5 – 117.1 °C. ^1H NMR (400 MHz, CDCl₃) δ 7.55 – 7.43 (m, 8H), 7.39 – 7.30 (m, 5H), 7.25 – 7.13 (m, 6H), 7.12 (s, 5H), 6.02 (dd, $J = 12.1, 1.6$ Hz, 1H), 5.95 (d, $J = 12.1$ Hz, 1H), 5.83 (d, $J = 42.0$ Hz, 1H), 5.42 (d, $J = 19.4$ Hz, 1H), 1.40 – 1.32 (m, 9H). ^{13}C NMR (101 MHz, CDCl₃) δ 150.2, 144.8 (d, $J = 7.5$ Hz), 143.1 (d, $J = 93.9$ Hz), 142.3, 142.1, 135.5 (d, $J = 10.7$ Hz), 134.0, 133.8 (d, $J = 8.1$ Hz), 133.0 (d, $J = 2.9$ Hz), 132.0, 131.9, 131.8, 131.4 (d, $J =$

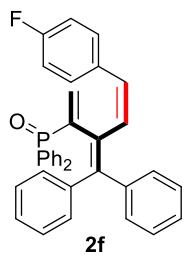
2.7 Hz), 130.4 (d, J = 88.9 Hz), 129.0, 128.7, 128.2, 128.18, 127.8, 127.4, 127.0 (d, J = 15.2 Hz), 124.5, 34.6, 31.4. **^{31}P NMR** (162 MHz, CDCl_3) δ 28.74 (s). **HRMS (ESI):** ([M+H] $^+$) Calcd for $\text{C}_{40}\text{H}_{38}\text{OP}^+$: 565.2655, Found: 565.2652. **IR** (film) ν 3675, 2987, 2971, 2900, 2205, 1907, 1406, 1250, 1056, 892, 765 cm^{-1} .

(Z)-(5-([1,1'-biphenyl]-4-yl)-3-(diphenylmethylene)penta-1,4-dien-2-yl)diphenylphosphine oxide (2e)



A white solid (78 mg, 67% yield) *m.p.*: 177.3 – 179.0 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.67 (d, J = 7.5 Hz, 2H), 7.60 – 7.45 (m, 12H), 7.44 – 7.25 (m, 10H), 7.19 (d, J = 13.1 Hz, 5H), 6.14 (d, J = 12.1 Hz, 1H), 6.05 (d, J = 12.1 Hz, 1H), 5.89 (d, J = 42.0 Hz, 1H), 5.46 (d, J = 19.4 Hz, 1H). **^{13}C NMR** (101 MHz, CDCl_3) δ 145.2 (d, J = 7.5 Hz), 143.4 (d, J = 94.9 Hz), 142.4, 142.1, 140.9, 139.7, 136.0, 135.5 (d, J = 10.1 Hz), 133.7 (d, J = 7.2 Hz), 133.0, 132.7, 131.9, 131.8, 131.5 (d, J = 2.6 Hz), 130.9, 130.0, 129.8, 128.9, 128.3, 128.2, 127.9, 127.6, 127.3 (d, J = 8.1 Hz), 127.0, 126.9, 126.3. **^{31}P NMR** (162 MHz, CDCl_3) δ 28.67 (s). **HRMS (ESI):** ([M+H] $^+$) Calcd for $\text{C}_{42}\text{H}_{34}\text{OP}^+$: 585.2342, Found: 585.2341. **IR** (film) ν 3061, 3015, 1888, 1486, 1408, 1196, 1111, 943, 860, 790, 690, 639 cm^{-1} .

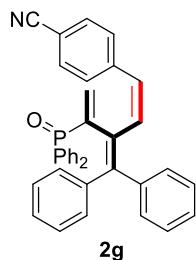
(Z)-(3-(diphenylmethylene)-5-(3-fluorophenyl)penta-1,4-dien-2-yl)diphenylphosphine oxide (2f)



A white solid (86 mg, 82% yield) *m.p.*: 116.1 – 118.2 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.57 – 7.52 (m, 4H), 7.49 – 7.42 (m, 4H), 7.39 – 7.33 (m, 4H), 7.32 – 7.23 (m, 5H) 7.17 – 7.10 (m, 3H), 7.10 – 7.04 (m, 2H), 6.94 – 6.84 (m, 2H), 6.11 (dd, J = 12.0, 1.3 Hz, 1H), 5.92 (d, J = 12.1 Hz, 1H), 5.81 (d, J = 41.9 Hz, 1H), 5.42 (d, J = 19.3 Hz, 1H). **^{13}C NMR** (101 MHz, CDCl_3) δ 163.1, 160.6, 145.2, 144.2 (d, J = 34.3 Hz), 142.3, 142.0, 135.2 (d, J = 11.0 Hz), 133.2 (d, J = 44.4 Hz), 132.8, 131.9, 131.8, 131.6 (d,

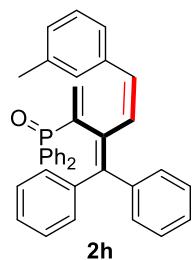
$J = 2.7$ Hz), 130.7, 130.7, 129.9, 129.2, 128.3, 128.2, 127.8, 127.4, 127.1 (d, $J = 16.3$ Hz), 114.5 (d, $J = 22.2$ Hz). **^{31}P NMR** (162 MHz, CDCl_3) δ 28.68 (s). **HRMS (ESI)**: ([M+H] $^+$) Calcd for $\text{C}_{36}\text{H}_{29}\text{FOP}^+$: 527.1935, Found: 527.1933. **IR** (film) ν 3658, 3379, 2988, 2213, 1921, 1681, 1610, 1437, 1312, 1170, 906, 821, 792, 694 cm^{-1} .

(Z)-4-(3-(diphenylmethylene)-4-(diphenylphosphoryl)penta-1,4-dien-1-yl)benzonitrile (2g)



A white solid (92 mg, 86% yield) *m.p.*: 177.3 – 178.8 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.66 – 7.57 (m, 4H), 7.56 – 7.50 (m, 1.1 Hz, 2H), 7.48 – 7.33 (m, 10H), 7.33 – 7.22 (m, 3H), 7.12 – 6.95 (m, 3H), 6.92 – 6.71 (m, 2H), 5.99 (dd, $J = 12.0, 1.2$ Hz, 1H), 5.82 (dd, $J = 27.1, 18.1$ Hz, 2H), 5.39 (d, $J = 19.1$ Hz, 1H). **^{13}C NMR** (101 MHz, CDCl_3) δ 145.9 (d, $J = 8.1$ Hz), 145.3, 144.3, 142.3, 141.7 (d, $J = 12.7$ Hz), 135.2 (d, $J = 9.1$ Hz), 132.9 (d, $J = 7.1$ Hz), 132.5, 132.2, 131.9, 131.8, 131.5, 131.1, 130.7, 123.0, 129.4, 128.4, 128.3, 127.9, 127.5, 127.2 (d, $J = 18.2$ Hz), 119.3, 109.7. **^{31}P NMR** (162 MHz, CDCl_3) δ 28.78 (s). **HRMS (ESI)**: ([M+H] $^+$) Calcd for $\text{C}_{37}\text{H}_{29}\text{NOP}^+$: 534.1981, Found: 534.1979. **IR** (film) ν 3674, 3057, 2961, 2900, 2223, 1906, 1603, 1438, 1177, 1116, 971, 862, 729, 574 cm^{-1} .

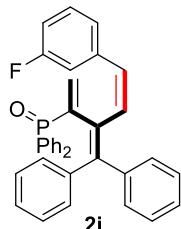
(Z)-(3-(diphenylmethylene)-5-(m-tolyl)penta-1,4-dien-2-yl)diphenylphosphine oxide (2h)



A white solid (96 mg, 92% yield) *m.p.*: 102.0 – 103.4 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.55 – 7.42 (m, 8H), 7.37 – 7.24 (m, 7H), 7.16 (d, $J = 3.1$ Hz, 7H), 7.03 (d, $J = 7.8$ Hz, 2H), 6.10 (dd, $J = 12.0, 1.8$ Hz, 1H), 5.98 (d, $J = 12.1$ Hz, 1H), 5.80 (d, $J = 41.9$ Hz, 1H), 5.41 (d, $J = 19.4$ Hz, 1H), 2.34 (s, 3H). **^{13}C NMR** (101 MHz, CDCl_3) δ 144.9 (d, $J = 7.5$ Hz), 143.0 (d, $J = 47.0$ Hz), 142.3, 142.0, 136.8 (d, $J = 25.2$ Hz), 135.4 (d, $J = 10.2$ Hz), 133.7 (d, $J = 7.2$ Hz), 133.2, 132.9, 131.9, 131.8, 131.4 (d, $J =$

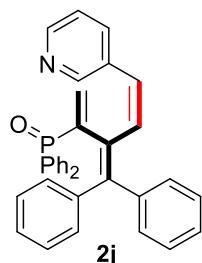
2.7 Hz), 130.4 (d, J = 82.8 Hz), 130.0, 129.4, 128.2, 128.1, 127.8, 127.6, 127.5, 127.2, 126.7 (d, J = 42.4 Hz), 21.5. **^{31}P NMR** (162 MHz, CDCl_3) δ 28.57 (s). **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{37}\text{H}_{32}\text{OP}^+$: 523.2185, Found: 523.2185. **IR** (film) ν 3675, 2987, 2900, 2216, 1920, 1406, 1066, 1056, 891, 789, 736, 696 cm^{-1} .

(Z)-(3-(diphenylmethylene)-5-(4-fluorophenyl)penta-1,4-dien-2-yl)diphenylphosphine oxide (2i)



A white solid (89 mg, 85% yield) *m.p.*: 169.5 – 171.9 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.60 – 7.51 (m, 4H), 7.50 – 7.42 (m, 4H), 7.40 – 7.23 (m, 8H), 7.16 – 7.07 (m, 4H), 7.03 (dd, J = 6.6, 3.1 Hz, 2H), 6.90 (t, J = 8.7 Hz, 2H), 5.97 (d, J = 12.0, 1H), 5.87 (d, J = 12.0 Hz, 1H), 5.77 (d, J = 41.9 Hz, 1H), 5.38 (d, J = 19.6 Hz, 1H). **^{13}C NMR** (101 MHz, CDCl_3) δ 163.6, 161.2, 145.5 (d, J = 7.5 Hz), 143.5 (d, J = 93.9 Hz), 142.2, 141.8, 138.9 (d, J = 8.0 Hz), 135.4 (d, J = 10.5 Hz), 133.2 (d, J = 7.0 Hz), 132.8, 131.9, 131.8, 131.5 (d, J = 3.0 Hz), 130.3 (d, J = 83.8 Hz), 129.0 (d, J = 8.3 Hz), 128.3, 128.2, 127.9, 127.5, 127.2 (d, J = 29.3 Hz), 125.0, 115.6 (d, J = 22.2 Hz), 113.7 (d, J = 21.2 Hz). **^{31}P NMR** (162 MHz, CDCl_3) δ 28.51 (s). **HRMS (ESI):** ($[\text{M}+\text{H}]^+$) Calcd for $\text{C}_{36}\text{H}_{29}\text{FOP}^+$: 527.1935, Found: 527.1933. **IR** (film) ν 3641, 3408, 2987, 2919, 1922, 1504.56, 1435, 1194, 1092, 931, 618, 572 cm^{-1} .

(Z)-(3-(diphenylmethylene)-5-(pyridin-3-yl)penta-1,4-dien-2-yl)diphenylphosphine oxide (2j)

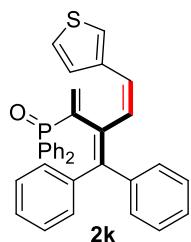


A white solid (72 mg, 71% yield). *m.p.*: 173.2 – 174.7 °C. **^1H NMR** (400 MHz, CDCl_3) δ 8.35 (d, J = 3.7 Hz, 1H), 8.17 (s, 1H), 7.89 (d, J = 7.9 Hz, 1H), 7.58 – 6.90 (m, 21H), 6.10 (d, J = 12.0, 1H), 5.85 (dd, J = 27.0, 14.8 Hz, 2H), 5.41 (d, J = 19.2 Hz, 1H). **^{13}C NMR** (101 MHz, CDCl_3) δ 149.8, 147.7,

145.7 (d, $J = 7.4$ Hz), 144.0 (d, $J = 93.9$ Hz), 142.1, 141.8, 136.0, 135.3 (d, $J = 10.5$ Hz), 133.0 (d, $J = 7.2$ Hz), 132.6 (d, $J = 5.1$ Hz), 131.8, 131.7, 131.7, 131.6, 131.6, 130.3 (d, $J = 7.5$ Hz), 129.3, 128.4, 128.2, 127.9, 127.5, 127.3 (d, $J = 26.3$ Hz), 122.8. ^{31}P NMR (162 MHz, CDCl_3) δ 28.55 (s).

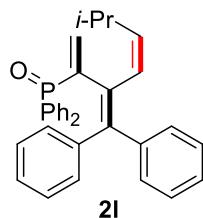
HRMS (ESI): ([M+H]⁺) Calcd for $\text{C}_{35}\text{H}_{29}\text{NOP}^+$: 510.1981, Found: 510.1980. **IR** (film) ν 3672, 3052, 2219, 1673, 1588, 1436, 1311, 1264, 1179, 1114, 907, 842 cm^{-1} .

(Z)-(3-(diphenylmethylene)-5-(thiophen-3-yl)penta-1,4-dien-2-yl)diphenylphosphine oxide (2k)



A white solid (71 mg, 69% yield). *m.p.*: 190.6 – 192.1 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.58 (dd, $J = 11.7, 7.9$ Hz, 4H), 7.48 (dd, $J = 9.6, 8.1$ Hz, 4H), 7.45 – 7.35 (m, 4H), 7.33 – 7.24 (m, 3H), 7.24 – 7.18 (m, 2H), 7.16 – 7.05 (m, 6H), 5.98 – 5.87 (m, 2H), 5.81 (d, $J = 42.0$ Hz, 1H), 5.40 (d, $J = 19.4$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 145.1, 144.0 (d, $J = 93.9$ Hz), 142.3, 142.0, 138.4, 134.9 (d, $J = 10.5$ Hz), 133.8 (d, $J = 7.2$ Hz), 132.9, 131.9, 131.8, 131.5 (d, $J = 2.8$ Hz), 130.7, 129.9, 128.9, 128.6, 128.2, 128.1, 127.8, 127.4, 127.0 (d, $J = 16.4$ Hz), 124.3, 124.1. ^{31}P NMR (162 MHz, CDCl_3) δ 28.95 (s). **HRMS (ESI):** ([M+H]⁺) Calcd for $\text{C}_{34}\text{H}_{28}\text{OPS}^+$: 515.1593, Found: 515.1590. **IR** (film) ν 2921, 2747, 2158, 1510, 1435, 1226, 1193, 964, 754, 696 cm^{-1} .

(Z)-(3-(diphenylmethylene)-6-methylhepta-1,4-dien-2-yl)diphenylphosphine oxide (2l)

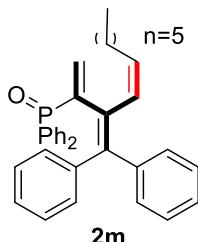


A white solid (60 mg, 63% yield). *m.p.*: 91.6 – 92.7 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.64 – 7.56 (m, 4H), 7.50 (td, $J = 7.3, 1.3$ Hz, 2H), 7.45 – 7.30 (m, 4H), 7.28 – 7.11 (m, 10H), 5.93 (dd, $J = 41.2, 1.1$ Hz, 1H), 5.58 (dd, $J = 15.3, 12.5$, 2H), 4.76 – 4.61 (m, 1H), 2.67 – 2.47 (m, 1H), 0.46 (d, $J = 6.5$ Hz, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 146.7, 145.8, 143.4, 142.6 (d, $J = 31.3$ Hz), 140.0, 134.0 (d, $J = 12.8$ Hz), 133.0, 132.0, 131.9, 131.5 (d, $J = 2.8$ Hz), 130.8 (d, $J = 48.5$ Hz), 128.2, 128.1, 127.6,

127.5, 126.8 (d, $J = 2.4$ Hz), 125.5, 100.0, 27.6, 21.6. **^{31}P NMR** (162 MHz, CDCl_3) δ 27.88 (s).

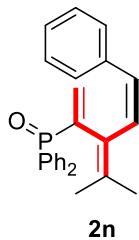
HRMS (ESI): ([M+H] $^+$) Calcd for $\text{C}_{33}\text{H}_{32}\text{OP}^+$: 475.2185, Found: 475.2185. **IR** (film) ν 3052, 2958, 2921, 2222, 1597, 1465, 1188, 1113, 1072, 1028, 945, 905, 759 cm^{-1} .

(Z)-(3-(diphenylmethylene)undeca-1,4-dien-2-yl)diphenylphosphine oxide (2m)



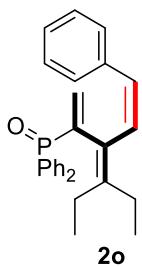
A white solid (62 mg, 60% yield). *m.p.*: 116.6 – 117.7 °C. **^1H NMR** (400 MHz, CDCl_3) δ 7.70 – 7.58 (m, 4H), 7.55 – 7.45 (m, 2H), 7.45 – 7.36 (m, 4H), 7.28 – 7.20 (m, 5H), 7.20 – 7.08 (m, 5H), 5.90 (d, $J = 41.1$, 1H), 5.75 – 5.55 (m, 2H), 5.01 – 4.85 (m, 1H), 1.80 – 1.65 (m, 2H), 1.28 – 1.22 (m, 2H), 1.20 – 1.15 (m, 4H), 0.91 – 0.84 (m, 5H). **^{13}C NMR** (101 MHz, CDCl_3) δ 146.6, 145.7, 143.8, 142.8, 142.4, 134.1, 133.9, 133.8, 132.9, 132.0, 131.9, 131.51 (d, $J = 2.8$ Hz), 130.9, 130.3, 128.2, 128.1, 127.7, 127.5, 126.8, 126.7, 31.9, 29.3, 29.0, 28.5, 22.7, 14.2. **^{31}P NMR** (162 MHz, CDCl_3) δ 27.64 (s). **HRMS (ESI):** ([M+H] $^+$) Calcd for $\text{C}_{36}\text{H}_{38}\text{OP}^+$: 517.2655, Found: 517.2650. **IR** (film) ν 3057, 2924, 2850, 2323, 1596, 1491, 1437, 1313, 1069, 812, 755, 693 cm^{-1} .

(Z)-(4-methyl-3-styrylpenta-1,3-dien-2-yl)diphenylphosphine oxide (2n)



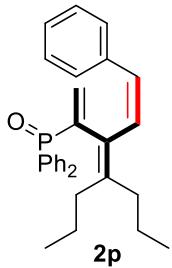
A pale yellow oil (71 mg, 93% yield). **^1H NMR** (400 MHz, CDCl_3) δ 7.72 – 7.64 (m, 4H), 7.52 – 7.46 (m, 2H), 7.43 – 7.35 (m, 4H), 7.30 – 7.14 (m, 5H), 6.14 (d, $J = 12.1$ Hz, 1H), 5.97 – 5.84 (m, 2H), 5.78 (dd, $J = 19.5$, 1.3 Hz, 1H), 1.66 (t, $J = 2.0$ Hz, 3H), 1.58 (t, $J = 3.9$ Hz, 3H). **^{13}C NMR** (101 MHz, CDCl_3) δ 144.6, 143.7, 137.5, 135.7 (d, $J = 7.4$ Hz), 132.5, 132.4, 132.1, 132.0, 131.7 (d, $J = 2.2$ Hz), 131.5, 130.7, 129.3, 128.6, 128.2, 128.1, 128.0, 127.0, 21.7. **^{31}P NMR** (162 MHz, CDCl_3) δ 28.56 (s). **HRMS (ESI):** ([M+H] $^+$) Calcd for $\text{C}_{26}\text{H}_{26}\text{OP}^+$: 385.1716, Found: 385.1715. **IR** (film) ν 3674, 3397, 2987, 2900, 2216, 1908, 1589, 1436, 1175, 1099, 1027, 907, 692 cm^{-1} .

(Z)-(4-ethyl-3-styrylhexa-1,3-dien-2-yl)diphenylphosphine oxide (2o)



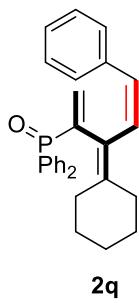
A pale yellow oil (76 mg, 92% yield). **¹H NMR** (400 MHz, CDCl₃) δ 7.83 – 7.56 (m, 4H), 7.55 – 7.46 (m, 2H), 7.45 – 7.35 (m, 6H), 7.30 – 7.14 (m, 3H), 6.16 (d, *J* = 12.1 Hz, 1H), 6.01 – 5.74 (m, 3H), 2.24 – 1.96 (m, 4H), 0.93 (t, *J* = 7.5 Hz, 3H), 0.80 (t, *J* = 7.6 Hz, 3H). **¹³C NMR** (101 MHz, CDCl₃) δ 146.2, 146.1, 143.7, 142.8, 137.3, 132.6, 132.2, 132.1, 131.8 (d, *J* = 10.1 Hz), 131.6, 131.6, 130.9, 129.0, 128.8, 128.2, 128.0, 127.8, 127.2, 24.6, 24.4, 13.1, 11.7. **³¹P NMR** (162 MHz, CDCl₃) δ 28.16 (s). **HRMS (ESI):** ([M+H]⁺) Calcd for C₂₈H₃₀OP⁺: 413.2029, Found: 413.2028. **IR (film) v** 3675, 2967, 2931, 2220, 1590, 1436, 1347, 1171, 1027, 907, 836, 725, 692 cm⁻¹.

(Z)-diphenyl(4-propyl-3-styrylhepta-1,3-dien-2-yl)phosphine oxide (2p)



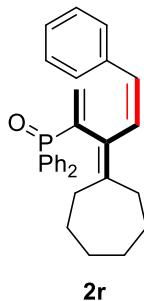
A pale yellow oil (80 mg, 91% yield). **¹H NMR** (400 MHz, CDCl₃) δ 7.76 – 7.56 (m, 4H), 7.55 – 7.45 (m, 2H), 7.44 – 7.38 (m, 4H), 7.34 (d, *J* = 7.2 Hz, 2H), 7.29 – 7.18 (m, 3H), 6.16 (d, *J* = 12.1 Hz, 1H), 6.04 – 5.68 (m, 3H), 2.02 (dd, *J* = 11.3, 4.6 Hz, 3H), 1.44 – 1.32 (m, 2H), 1.32 – 1.18 (m, 3H), 0.87 (t, *J* = 7.3 Hz, 3H), 0.79 (t, *J* = 7.3 Hz, 3H). **¹³C NMR** (101 MHz, CDCl₃) δ 144.0 (d, *J* = 7.1 Hz) 143.7, 142.8, 137.3, 132.7, 132.1, 132.0, 131.9 (d, *J* = 10.1 Hz), 131.7, 131.6 (d, *J* = 3.0 Hz), 130.9, 129.2, 128.9, 128.7 (d, *J* = 7.1 Hz), 128.2, 128.1, 127.8, 127.2, 34.2, 34.1, 21.9 (d, *J* = 2.0 Hz), 20.7 (d, *J* = 2.4 Hz), 14.5, 14.4. **³¹P NMR** (162 MHz, CDCl₃) δ 28.12 (s). **HRMS (ESI):** ([M+H]⁺) Calcd for C₃₀H₃₄OP⁺: 441.2342, Found: 441.2341. **IR (film) v** 3674, 2958, 2869, 2220, 1436, 1377, 1191, 1116, 1099, 906, 787, 724, 692, 643 cm⁻¹.

(Z)-(3-cyclohexylidene-5-phenylpenta-1,4-dien-2-yl)diphenylphosphine oxide (2q)



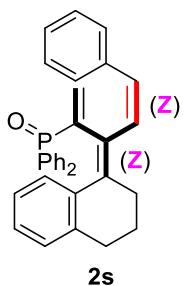
A pale yellow oil (80 mg, 94% yield). **¹H NMR** (400 MHz, CDCl₃) δ 7.70 (dd, *J* = 11.5, 7.6 Hz, 4H), 7.55 – 7.49 (m, 2H), 7.43 (t, *J* = 7.7 Hz, 4H), 7.34 – 7.17 (m, 5H), 6.19 (d, *J* = 12.1 Hz, 1H), 6.01 – 5.77 (m, 3H), 2.11 (dd, *J* = 22.7, 17.5 Hz, 4H), 1.47 – 1.28 (m, 6H). **¹³C NMR** (101 MHz, CDCl₃) δ 144.1, 143.2, 142.4 (d, *J* = 8.1 Hz), 137.5, 132.6, 132.2, 132.1, 131.6 (d, *J* = 2.0 Hz), 131.5, 130.8, 129.1, 128.8, 128.2, 128.1, 127.9, 127.0, 125.6 (d, *J* = 7.1 Hz), 31.6, 31.6, 27.6, 26.8, 26.4. **³¹P NMR** (162 MHz, CDCl₃) δ 28.12 (s). **HRMS (ESI):** ([M+H]⁺) Calcd for C₂₉H₃₀OP⁺: 425.2029, Found: 425.2027. **IR (film)** ν 3673, 2978, 2924, 1719, 1597, 1432, 1391, 1112, 1068, 996, 944, 721, 690 cm⁻¹.

(Z)-(3-cycloheptylidene-5-phenylpenta-1,4-dien-2-yl)diphenylphosphine oxide (2r)



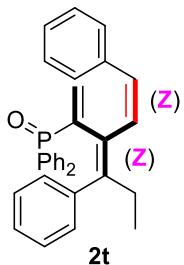
A pale yellow oil (76 mg, 87% yield). **¹H NMR** (400 MHz, CDCl₃) δ 7.70 – 7.54 (m, 4H), 7.53 – 7.45 (m, 2H), 7.44 – 7.35 (m, 4H), 7.35 – 7.11 (m, 5H), 6.13 (d, *J* = 12.1 Hz, 1H), 6.07 – 5.65 (m, 3H), 2.29 (s, 4H), 1.47 (s, 8H). **¹³C NMR** (101 MHz, CDCl₃) δ 145.3, 145.2, 143.5, 142.6, 137.3, 132.7, 132.3 (d, *J* = 10.1 Hz), 132.1, 132.0, 131.7, 131.6 (d, *J* = 2.8 Hz), 130.1, 129.2, 128.9, 128.2, 128.0, 127.8, 127.2, 33.2, 32.6, 29.6, 29.3, 28.3, 26.4. **³¹P NMR** (162 MHz, CDCl₃) δ 28.75 (s). **HRMS (ESI):** ([M+H]⁺) Calcd for C₃₀H₃₂OP⁺: 439.2185, Found: 439.2176. **IR (film)** ν 3675, 2977, 2921, 1713, 1590, 1436, 1393, 1116, 1066, 998, 949, 725, 691 cm⁻¹.

((Z)-3-((Z)-3,4-dihydronaphthalen-1(2H)-ylidene)-5-phenylpenta-1,4-dien-2-yl)diphenylphosphine oxide (2s)



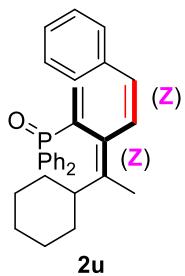
A pale yellow solid (82 mg, 87% yield). *m.p.*: 131.5–133.4 °C. **1H NMR** (400 MHz, CDCl₃) δ 7.98 – 7.89 (m, 1H), 7.71 – 7.61 (m, 4H), 7.58 – 7.45 (m, 4H), 7.44 – 7.35 (m, 4H), 7.28 (dd, *J* = 9.1, 6.0 Hz, 2H), 7.20 – 7.11 (m, 3H), 7.06 – 7.00 (m, 1H), 6.21 (d, *J* = 12.1 Hz, 1H), 5.93 (t, *J* = 27.1 Hz, 2H), 5.59 (d, *J* = 19.2, 1H), 2.54 (t, *J* = 6.5 Hz, 2H), 2.30 (dd, *J* = 5.8, 4.1 Hz, 2H), 1.68 – 1.57 (m, 2H). **13C NMR** (101 MHz, CDCl₃) δ 145.5, 144.6, 140.0, 139.1 (d, *J* = 7.0 Hz), 137.3, 136.5, 134.0 (d, *J* = 10.4 Hz), 132.3 (d, *J* = 102.0 Hz), 131.9, 131.8, 131.7, 131.5 (d, *J* = 3.0 Hz), 130.0 (d, *J* = 77.8 Hz), 129.4 (d, *J* = 7.1 Hz), 128.7, 128.3, 128.2, 128.1, 127.4 (d, *J* = 31.3 Hz), 127.3, 125.0, 29.5, 29.1, 22.5. **31P NMR** (162 MHz, CDCl₃) δ 28.76 (s). **HRMS (ESI):** ([M+H]⁺) Calcd for C₃₃H₃₀OP⁺: 473.2029, Found: 473.2028. **IR (film) v** 3050, 2968, 2927, 2228, 1598, 1475, 1182, 1118, 1073, 1022, 944, 905, 786 cm⁻¹.

Diphenyl((Z)-4-phenyl-3-((Z)-styryl)hexa-1,3-dien-2-yl)phosphine oxide (2t)



A pale yellow oil (77 mg, 84% yield). **1H NMR** (400 MHz, CDCl₃) δ 7.51 – 7.45 (m, 6H), 7.42 (d, *J* = 7.3 Hz, 2H), 7.38 – 7.31 (m, 6H), 7.29 (d, *J* = 4.3 Hz, 4H), 7.25 – 7.20 (m, 2H), 6.29 – 6.15 (m, 2H), 5.55 (dd, *J* = 42.6, 0.8 Hz, 1H), 5.17 (dd, *J* = 19.9, 0.8 Hz, 1H), 2.49 (t, *J* = 7.3 Hz, 2H), 0.79 (t, *J* = 7.5 Hz, 3H). **13C NMR** (101 MHz, CDCl₃) δ 145.4, 145.3, 143.2, 142.3, 141.3, 137.1, 133.9 (d, *J* = 10.4 Hz), 132.9, 132.0, 131.9, 131.5 (d, *J* = 3.0 Hz), 130.8 (d, *J* = 6.6 Hz), 129.2, 129.1, 128.4, 128.2, 128.1, 127.9, 127.7, 127.3, 126.4, 28.7, 11.5 (d, *J* = 2.1 Hz). **31P NMR** (162 MHz, CDCl₃) δ 29.36 (s). **HRMS (ESI):** ([M+H]⁺) Calcd for C₃₂H₃₀OP⁺: 461.2029, Found: 461.2030. **IR (film) v** 3052, 2971, 2931, 2872, 2217, 1907, 1591, 1435, 1185, 949, 917, 759, 691 cm⁻¹.

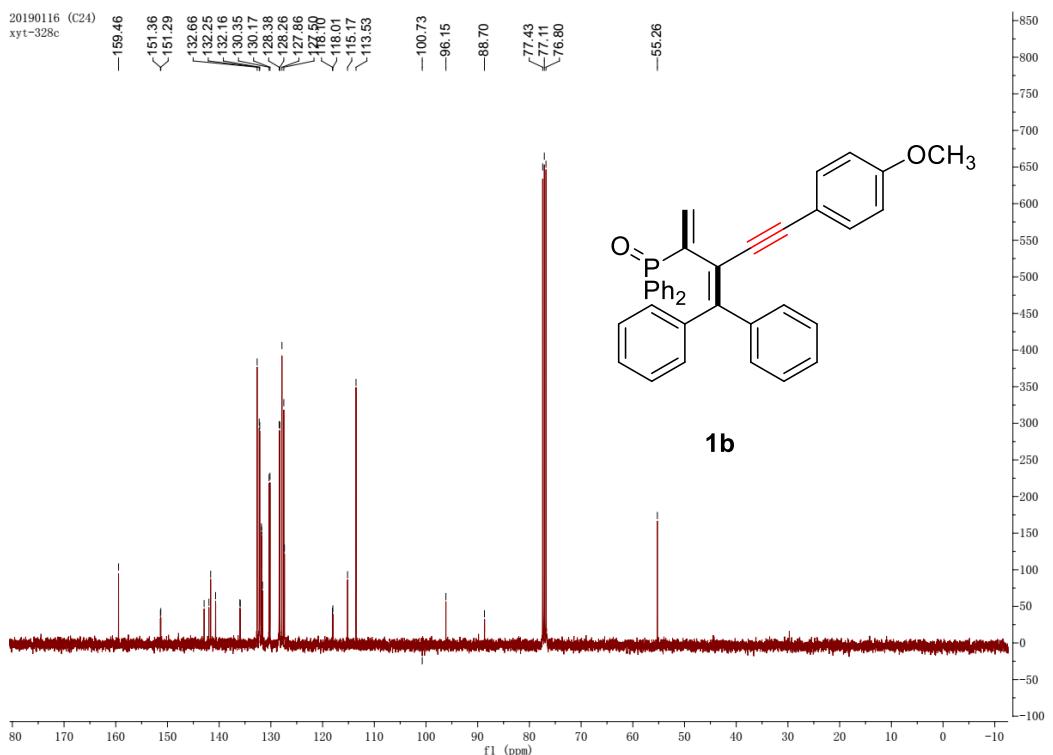
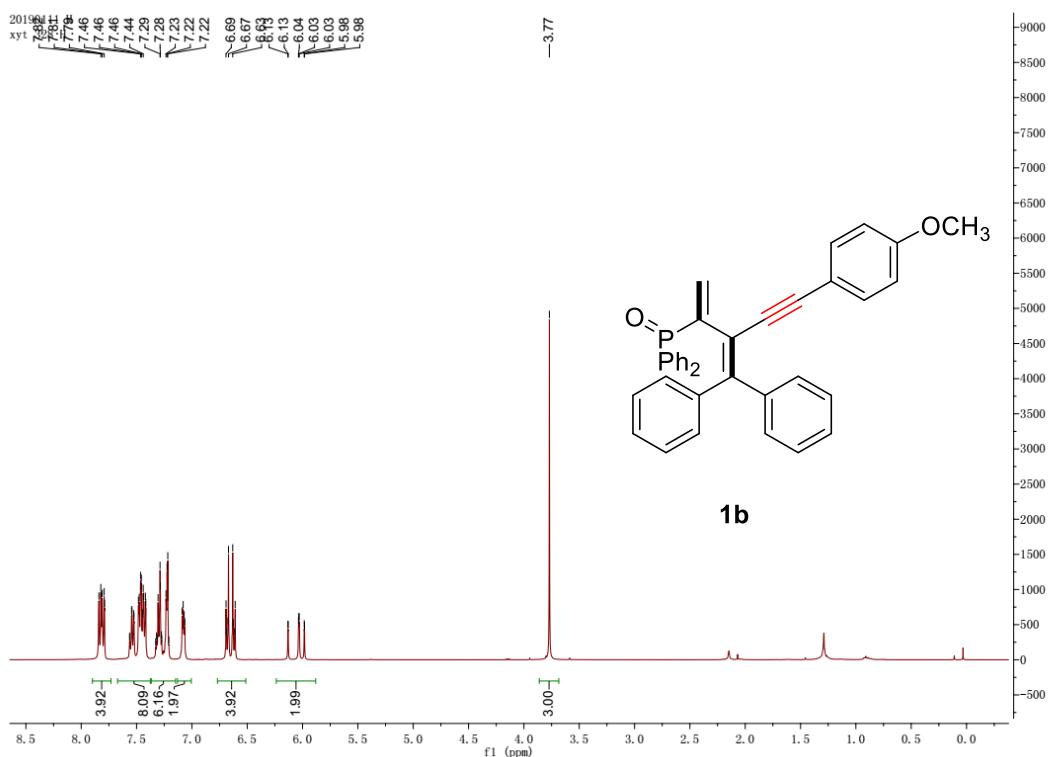
((Z)-4-cyclohexyl-3-((Z)-styryl)penta-1,3-dien-2-yl)diphenylphosphine oxide (2u)

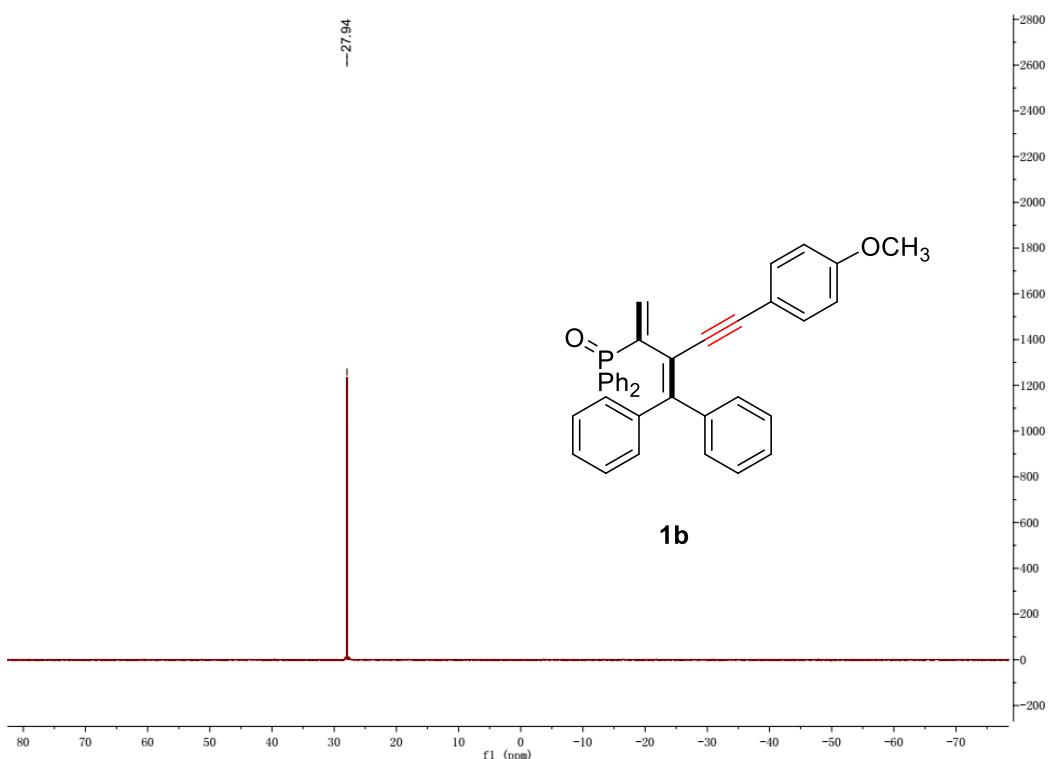


A pale yellow oil (70mg, 77% yield). **¹H NMR** (400 MHz, CDCl₃) δ 7.75 – 7.65 (m, 4H), 7.54 – 7.49 (m, 2H), 7.46 – 7.40 (m, 4H), 7.37 (d, *J* = 7.3 Hz, 2H), 7.29 – 7.23 (m, 2H), 7.21 – 7.16 (m, 1H), 6.21 (d, *J* = 12.1 Hz, 1H), 5.95 (d, *J* = 12.1 Hz, 1H), 5.87 (dd, *J* = 41.3, 1.4 Hz, 1H), 5.78 (dd, *J* = 19.5, 1.4 Hz, 1H), 2.42 (s, 1H), 1.68 – 1.59 (m, 3H), 1.53 – 1.47 (m, 3H), 1.20 (s, 3H), 1.09 (s, 3H). **¹³C NMR** (101 MHz, CDCl₃) δ 144.4, 143.5, 143.3 (d, *J* = 8.1 Hz), 137.6, 132.2, 132.6, 131.6, 132.1, 131.7 (d, *J* = 2.7 Hz), 131.3 (d, *J* = 9.1 Hz), 130.5, 129.9, 128.6, 128.2, 128.1, 127.9, 127.1 (d, *J* = 5.1 Hz), 42.6, 30.6, 26.3, 26.2, 14.7. **³¹P NMR** (162 MHz, CDCl₃) δ 28.11 (s). **HRMS (ESI):** ([M+H]⁺) Calcd for C₃₁H₃₃OP: 453.2342, Found: 453.2343. **IR** (film) ν 3675, 2924, 2850, 2219, 1447, 1436, 1374, 1176, 1115, 907, 771, 725, 692, 620 cm⁻¹.

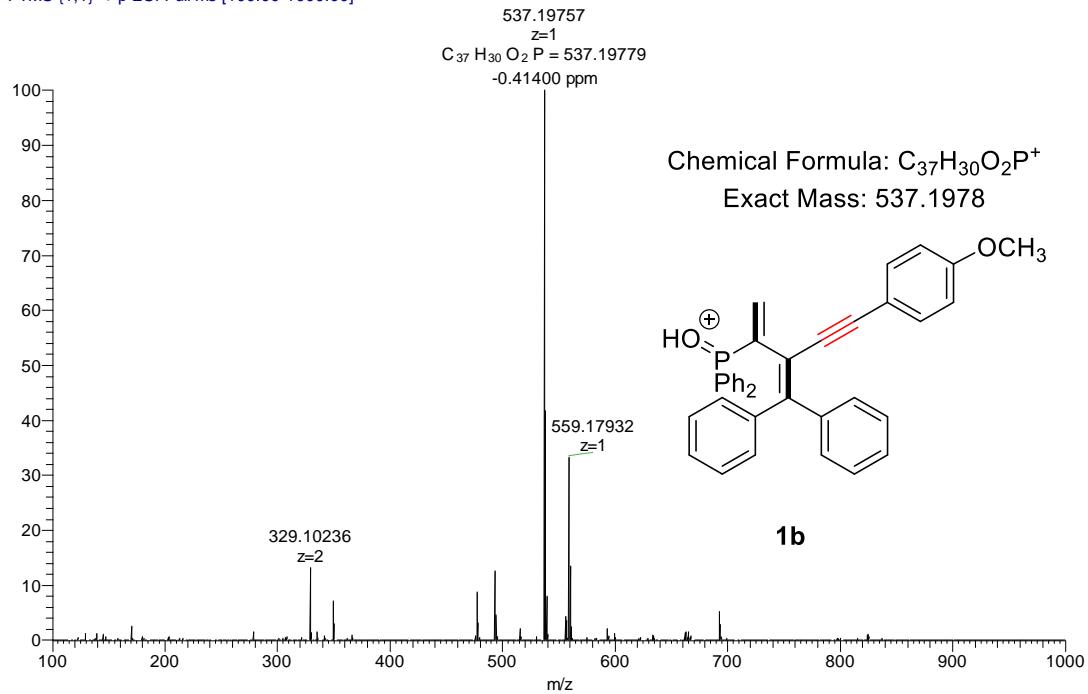
16. ^1H -NMR, ^{13}C -NMR, ^{31}P -NMR and HR-MS Spectra

1b

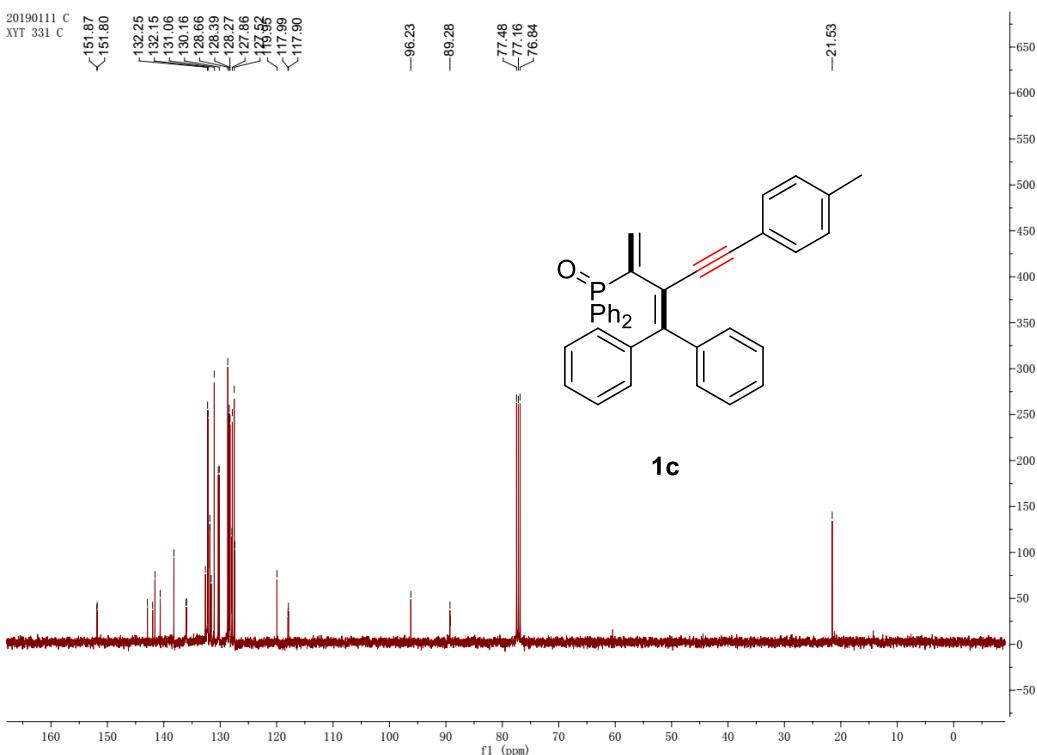
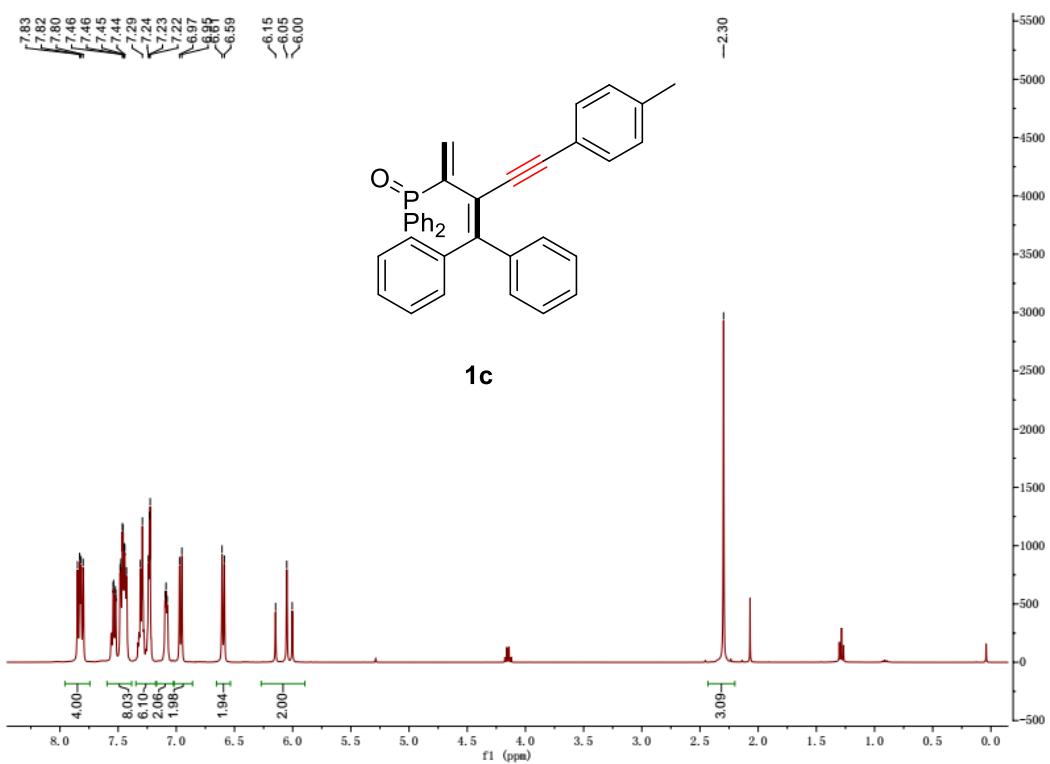


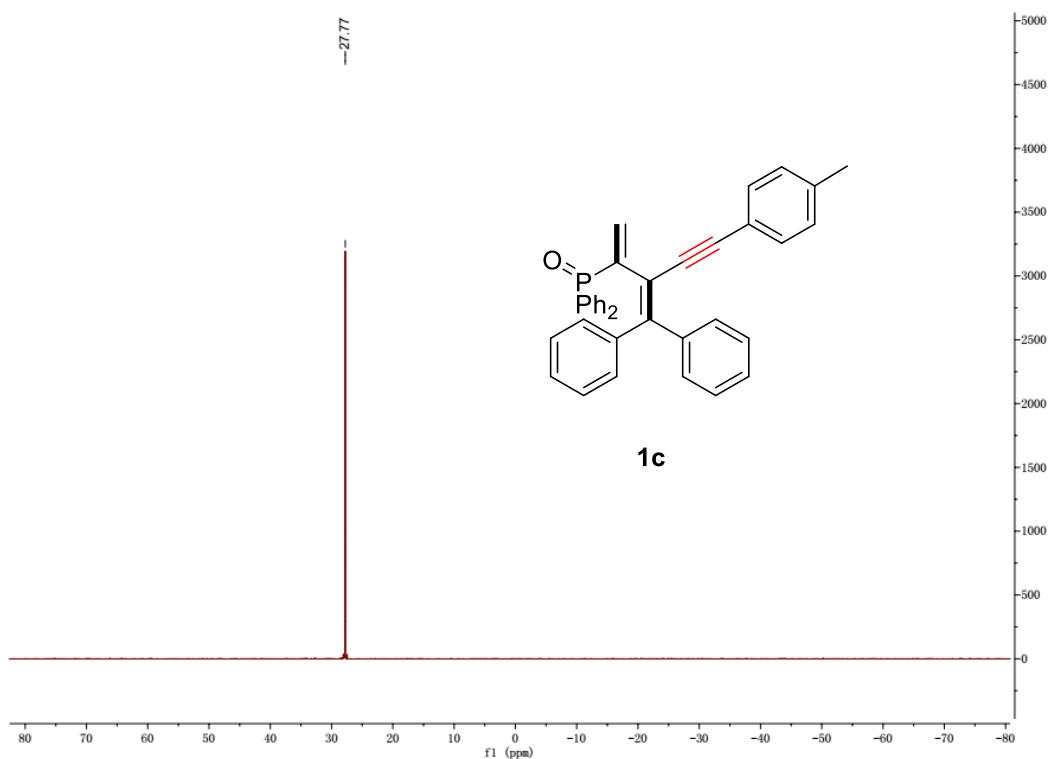


20190109-1 #33 RT: 0.49 AV: 1 NL: 7.27E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

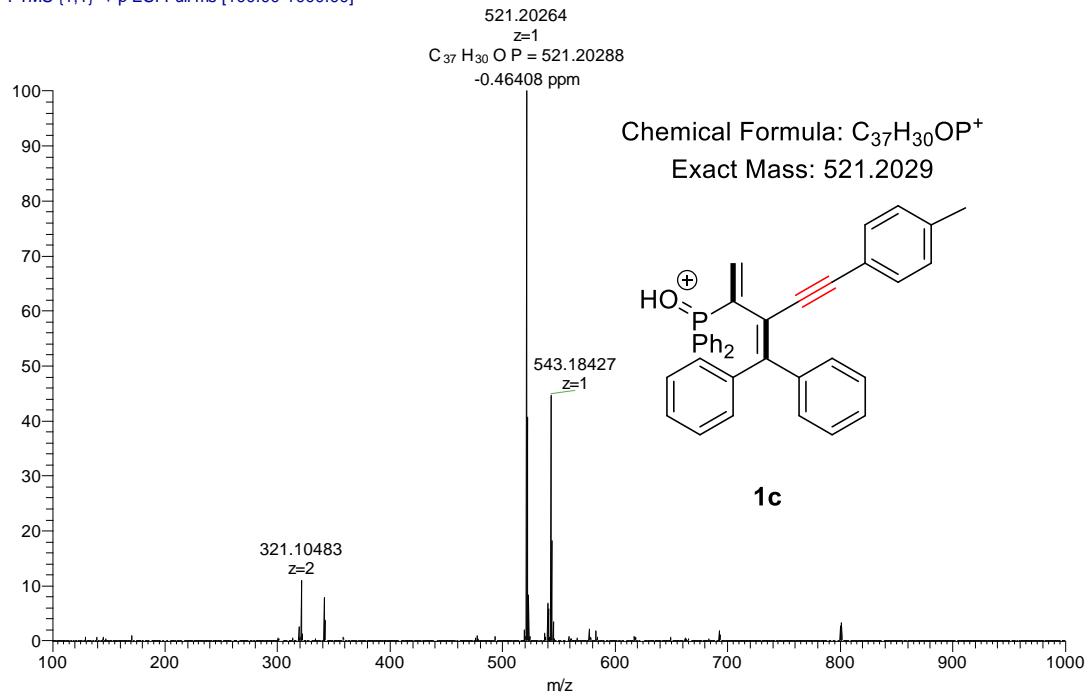


1c

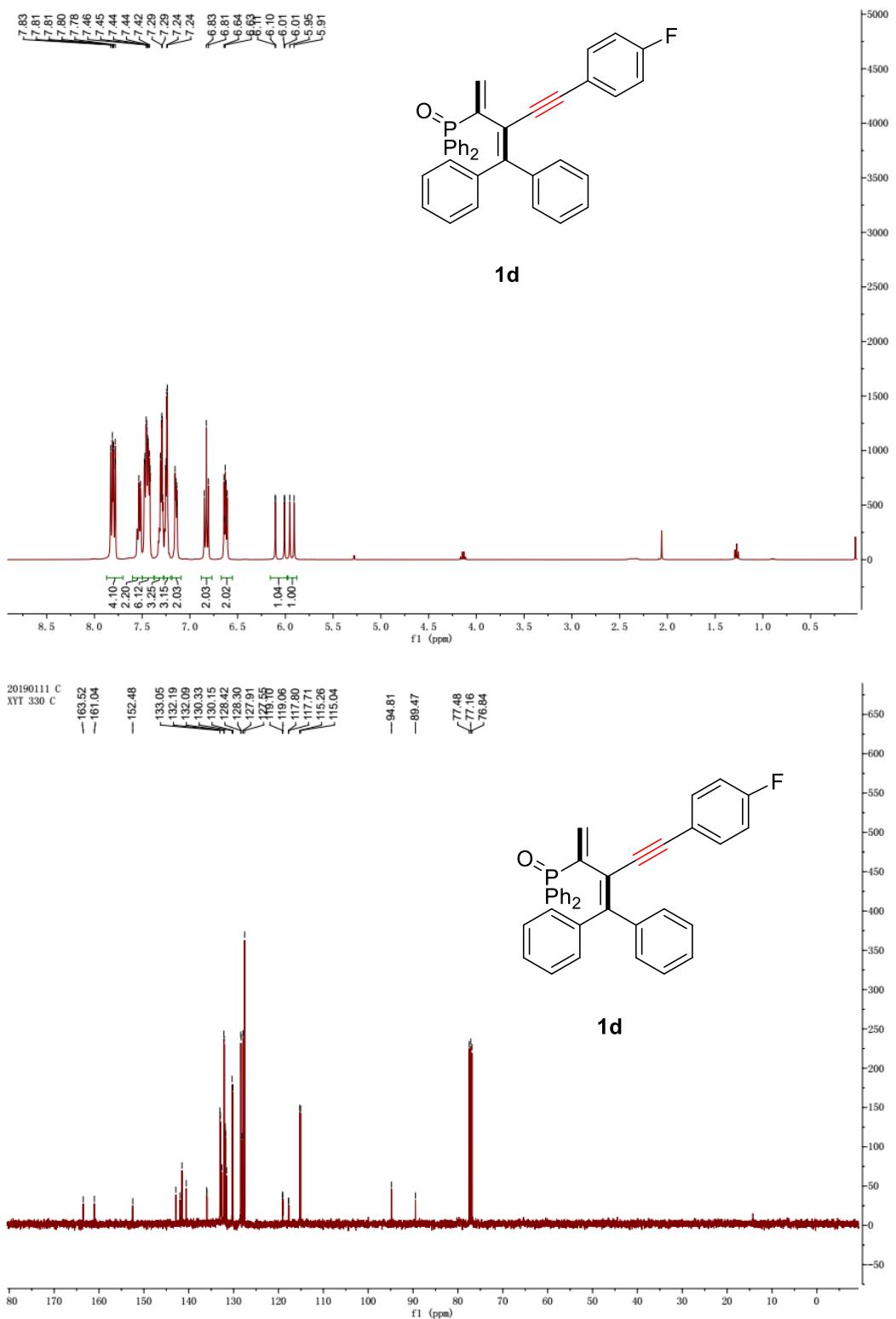




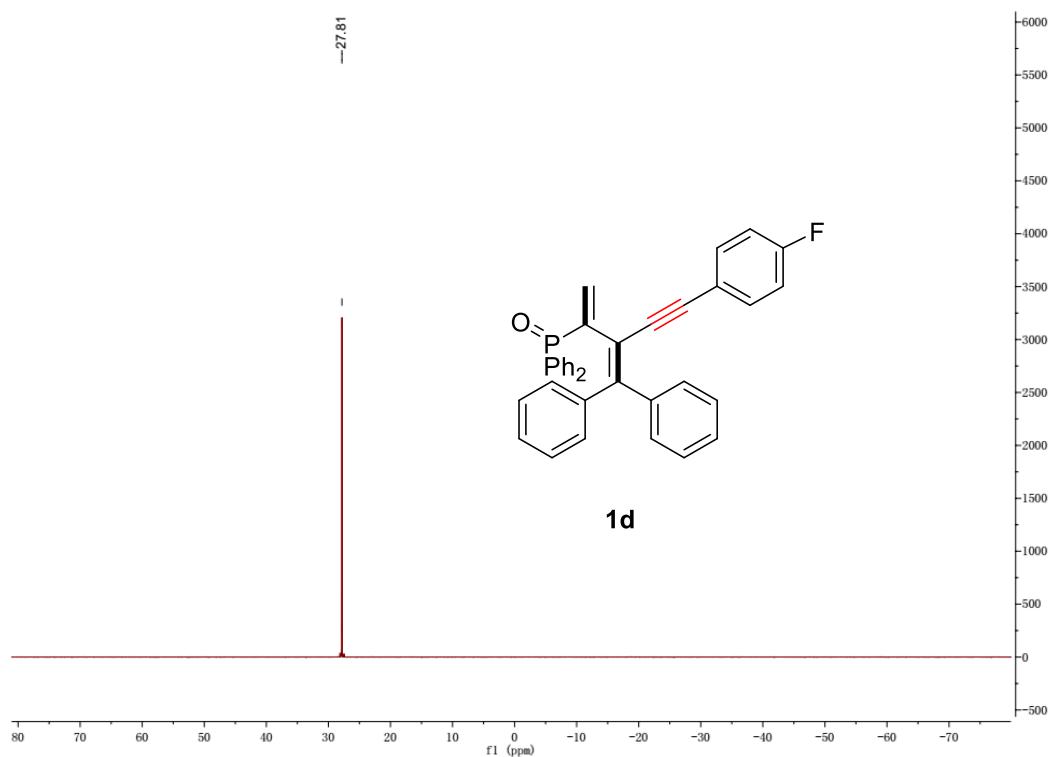
20190109-2 #33 RT: 0.49 AV: 1 NL: 1.49E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



1d



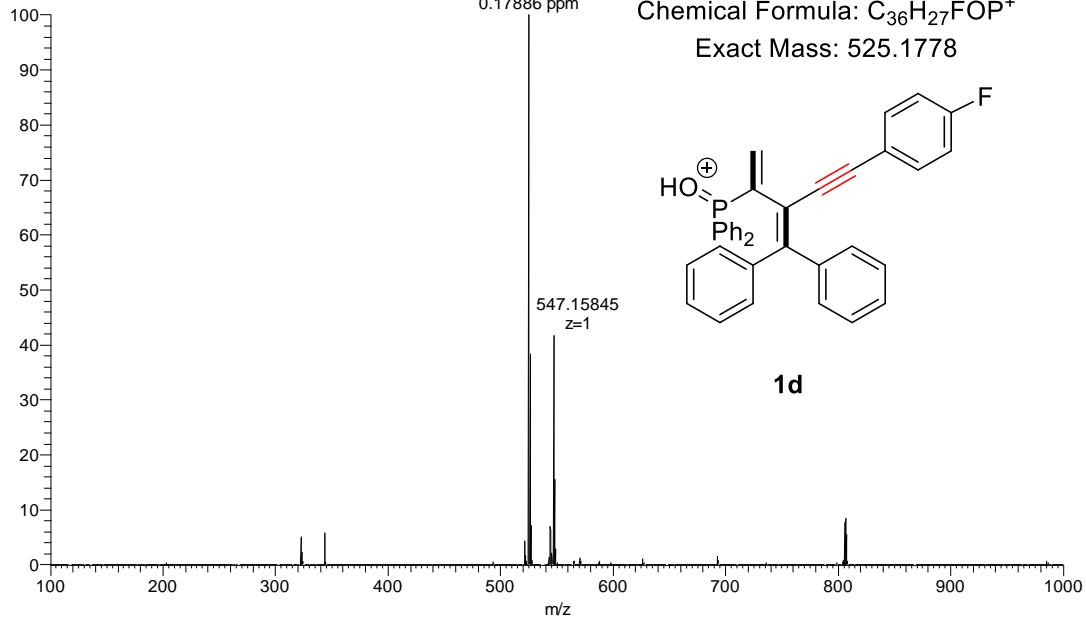
51



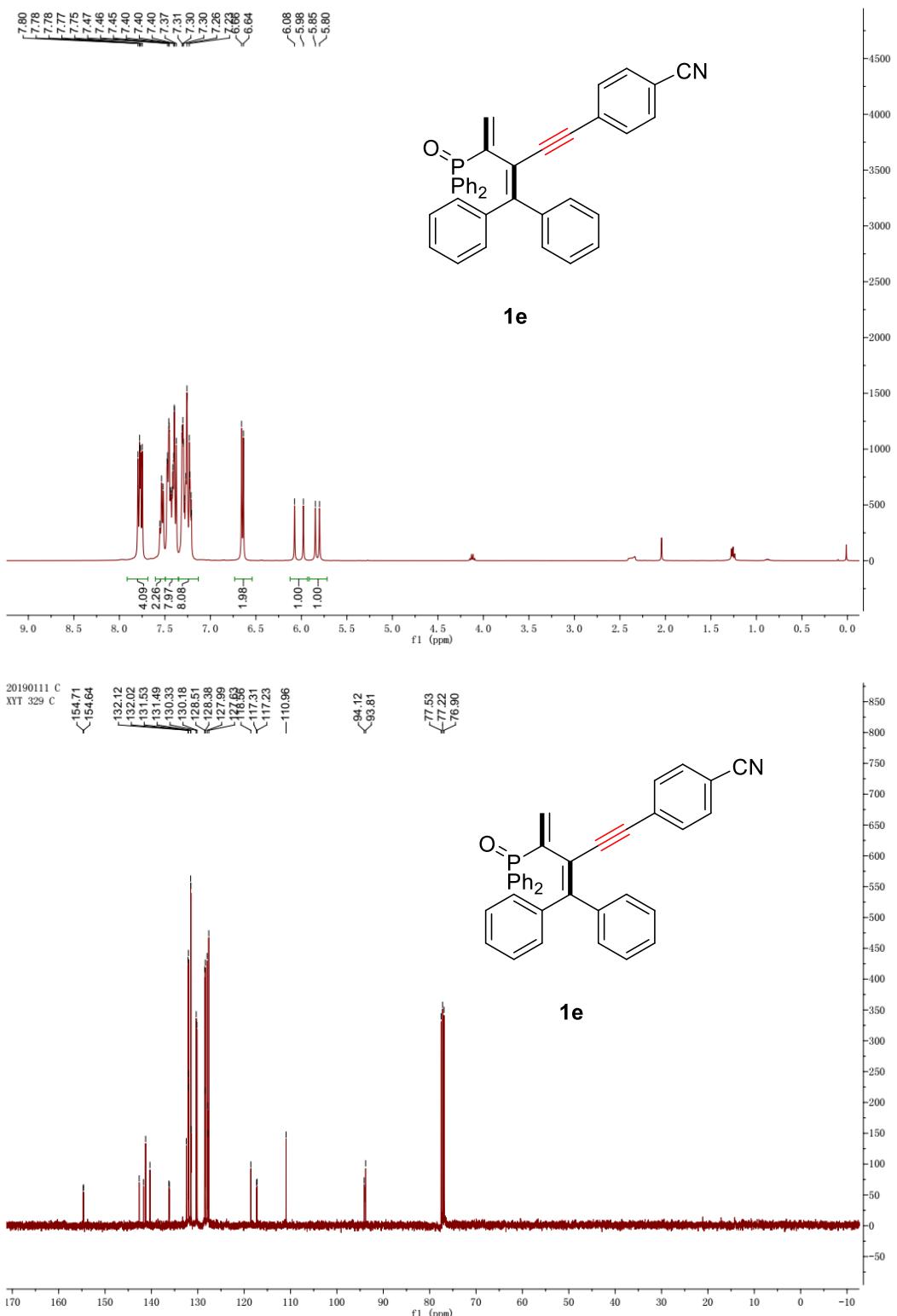
20190109-3 #25 RT: 0.37 AV: 1 NL: 5.23E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

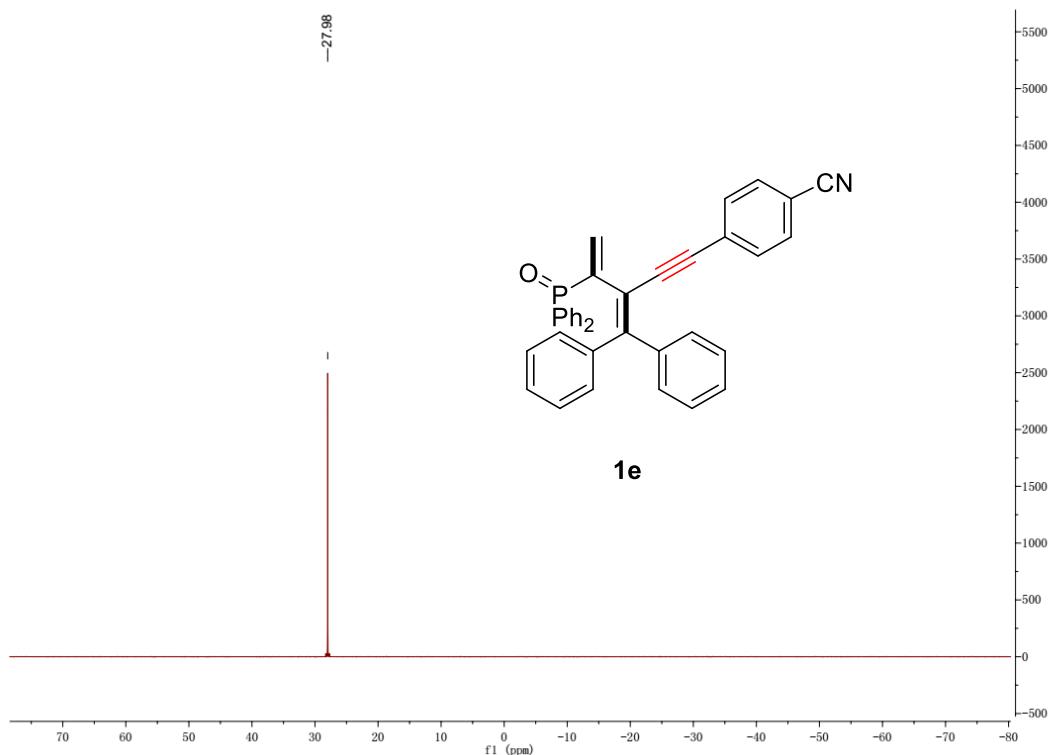
525.17676
 $z=1$
 $C_{39} H_{26} P = 525.17666$
0.17886 ppm

Chemical Formula: $C_{36}H_{27}FOP^+$
Exact Mass: 525.1778

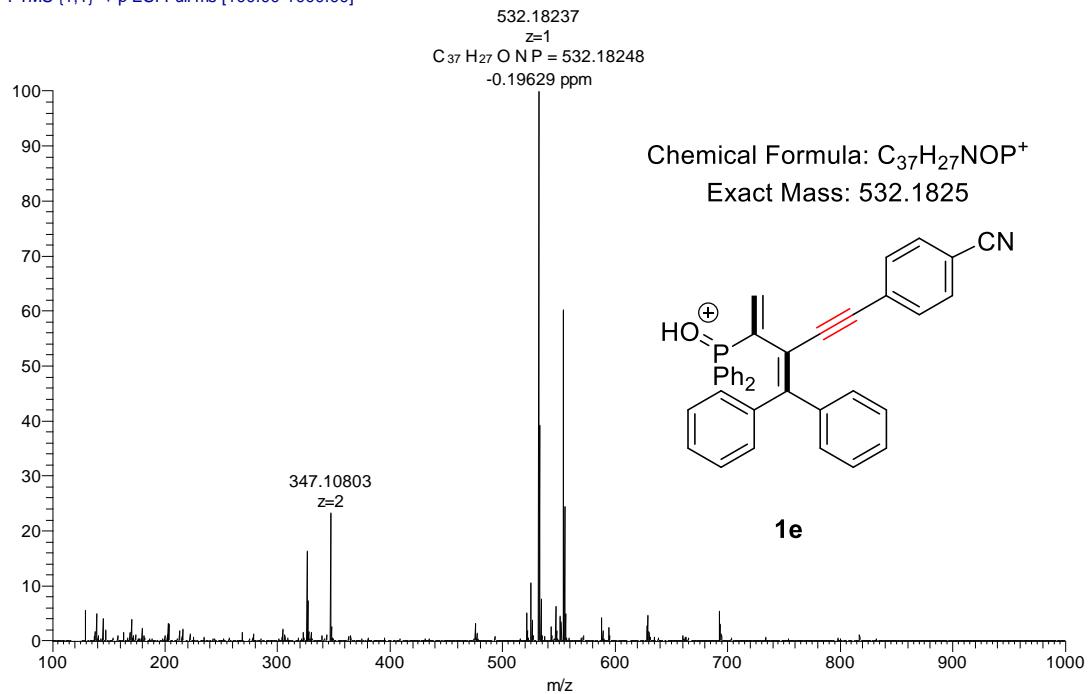


1e

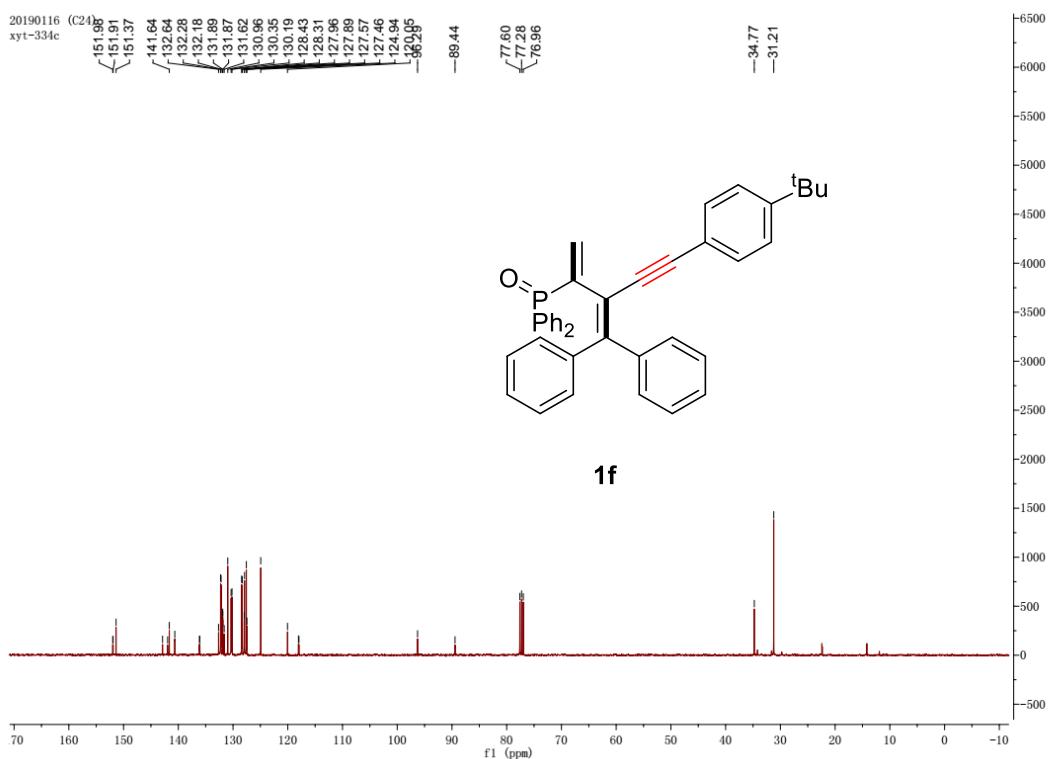
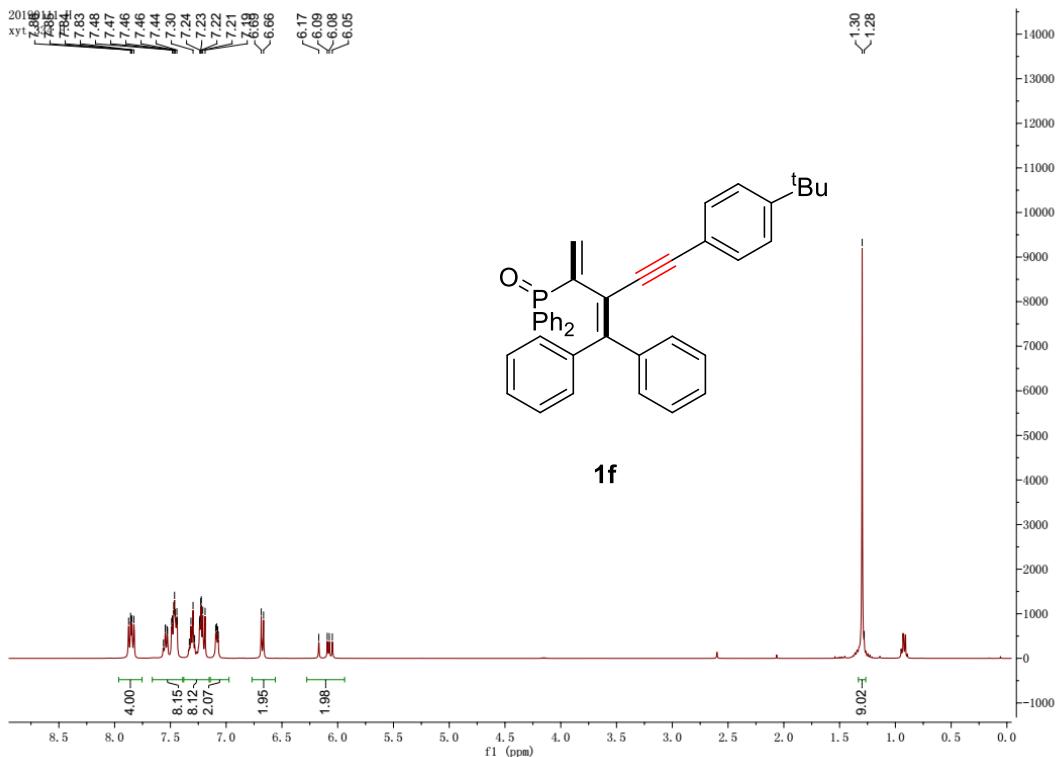




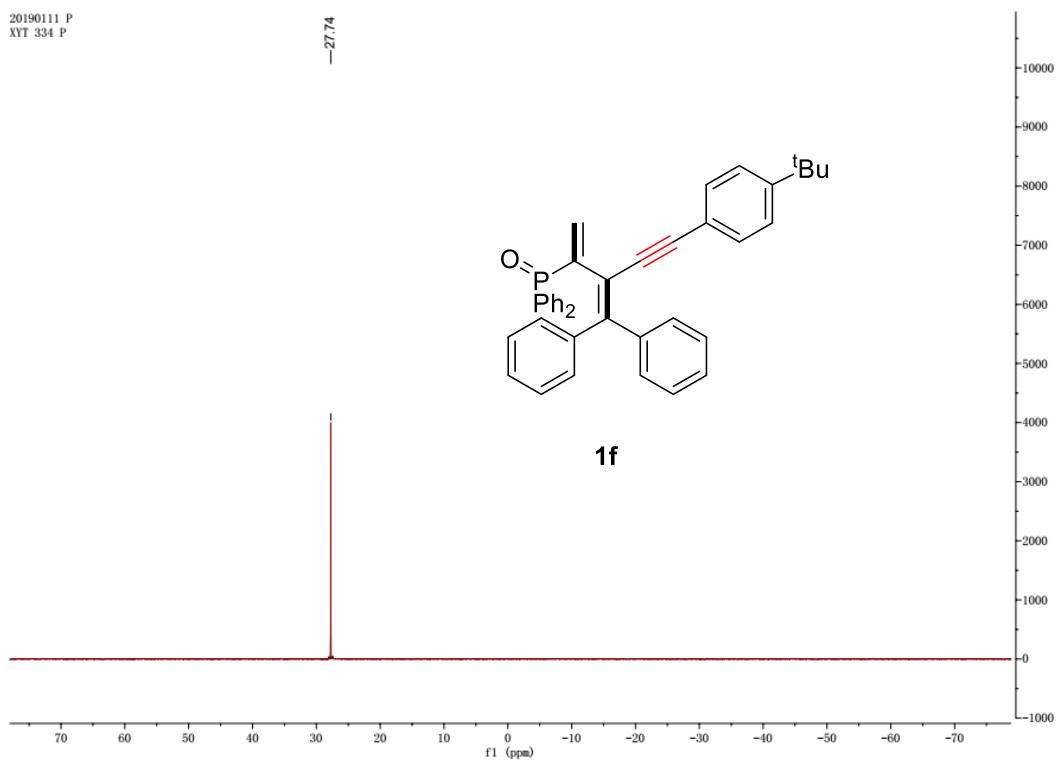
20190109-4 #37 RT: 0.57 AV: 1 NL: 2.34E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



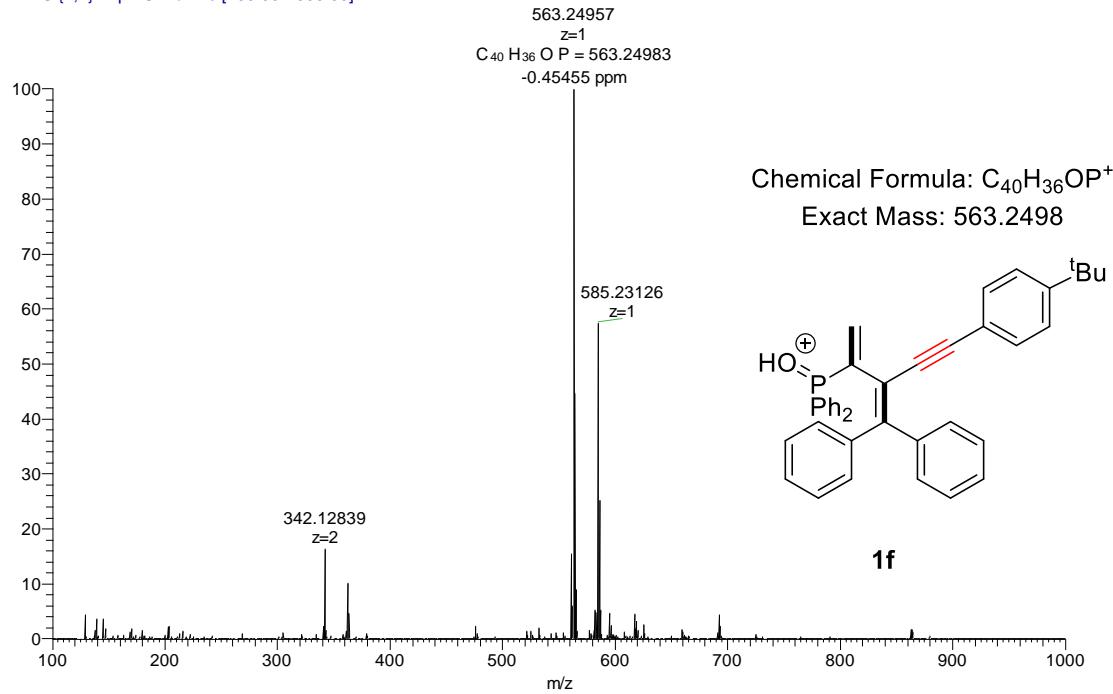
1f



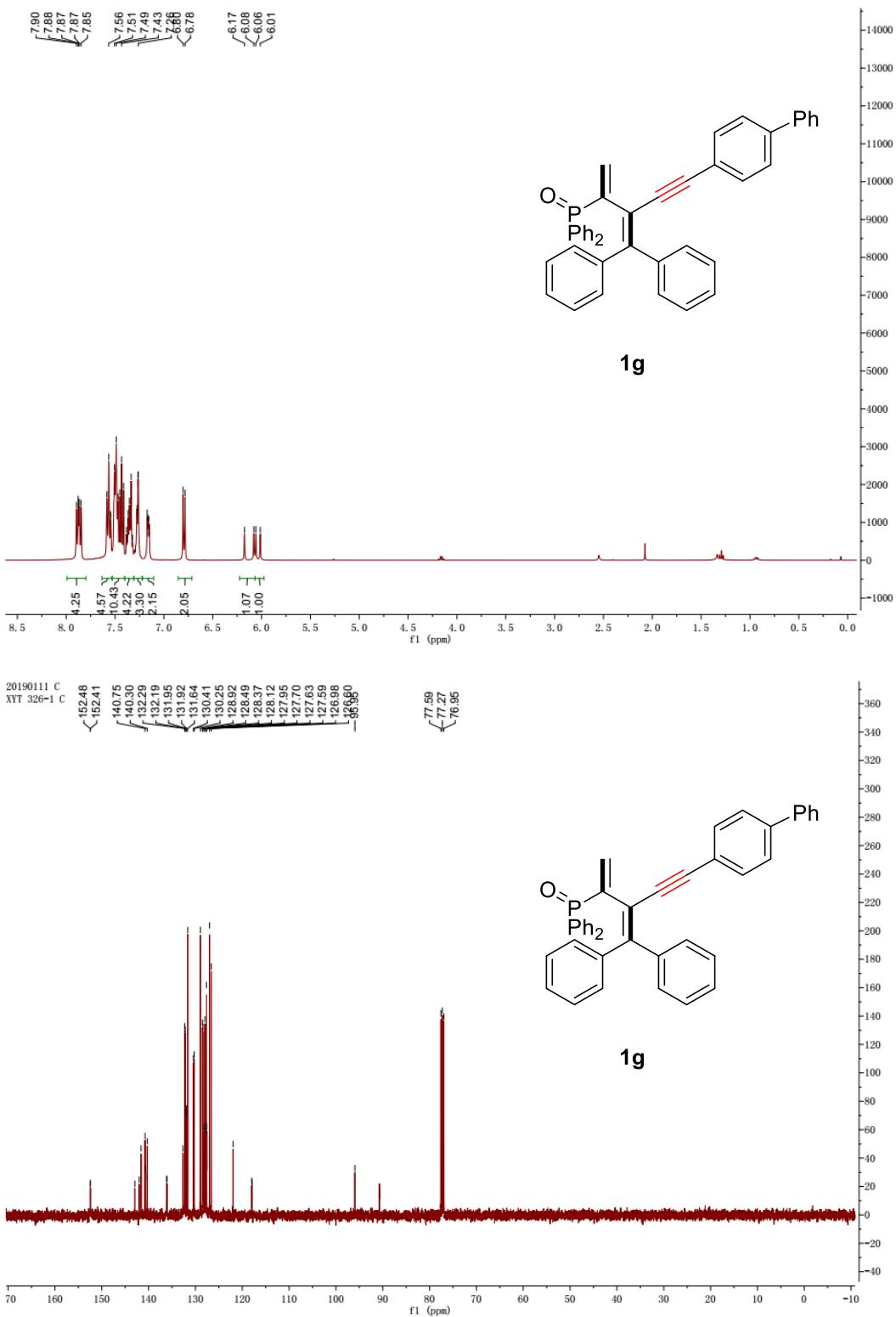
20190111 P
XYT 334 P

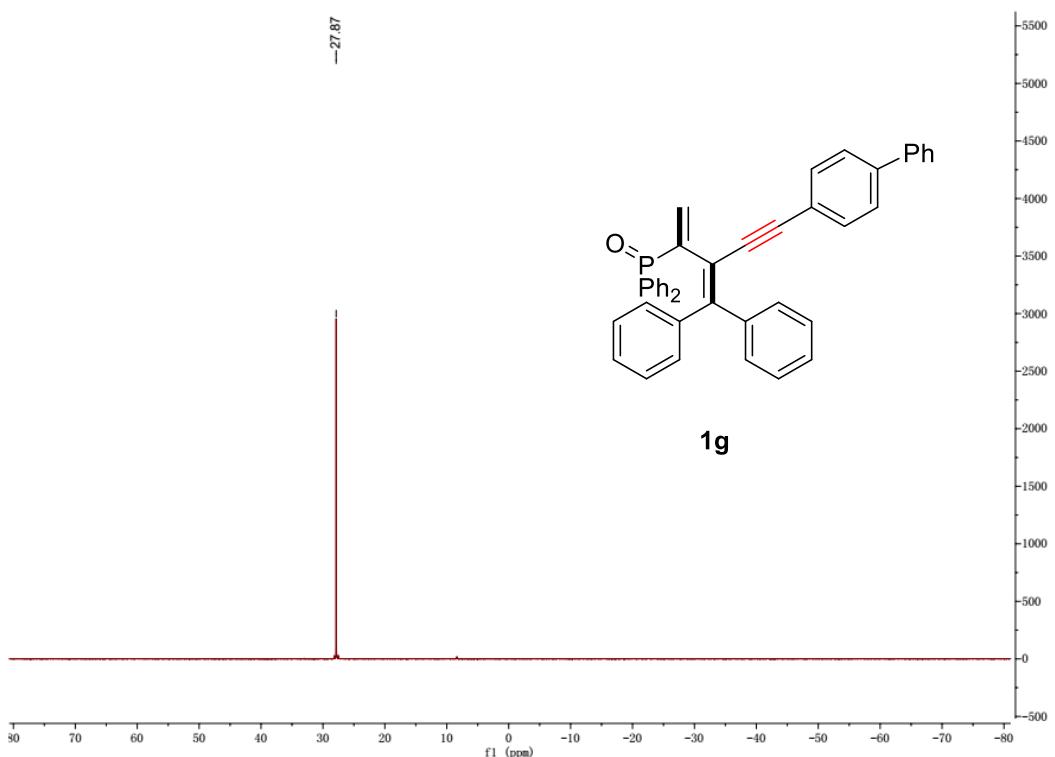


20190109-5 #37 RT: 0.56 AV: 1 NL: 4.14E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

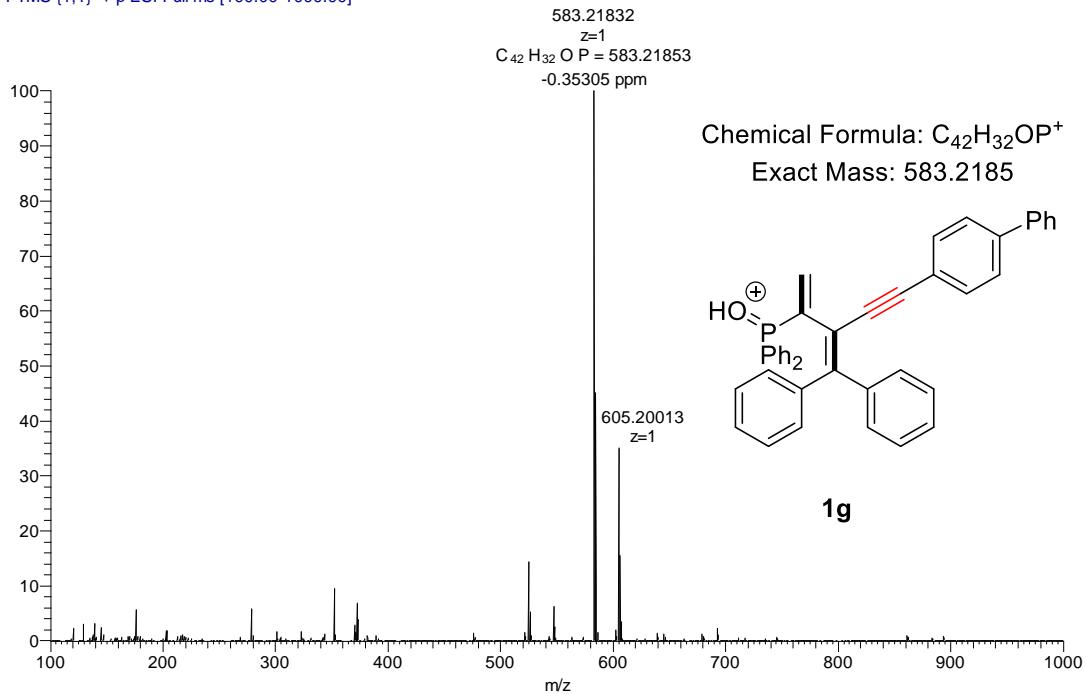


1g

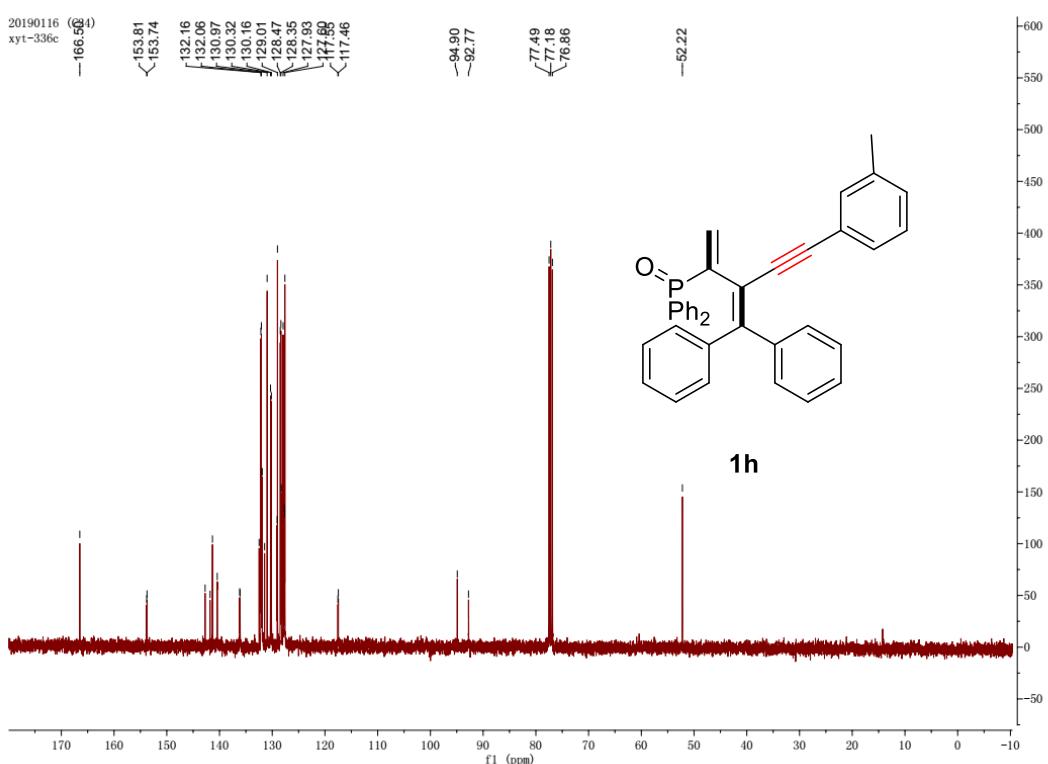
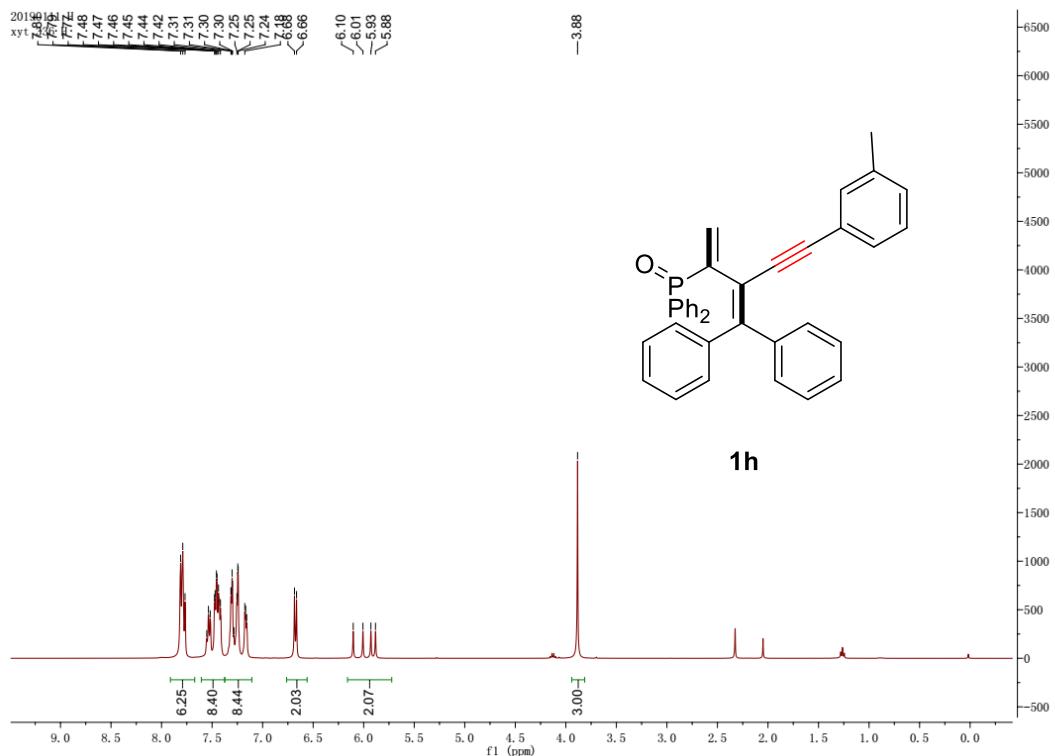




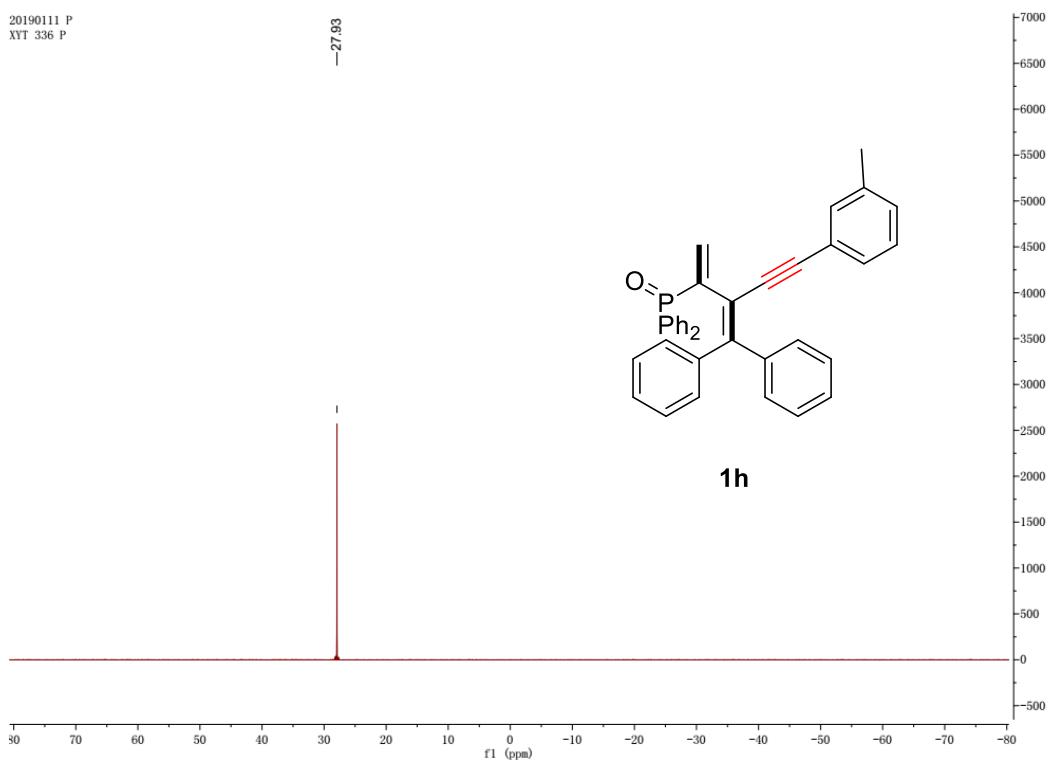
20190109-8 #29 RT: 0.44 AV: 1 NL: 4.96E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



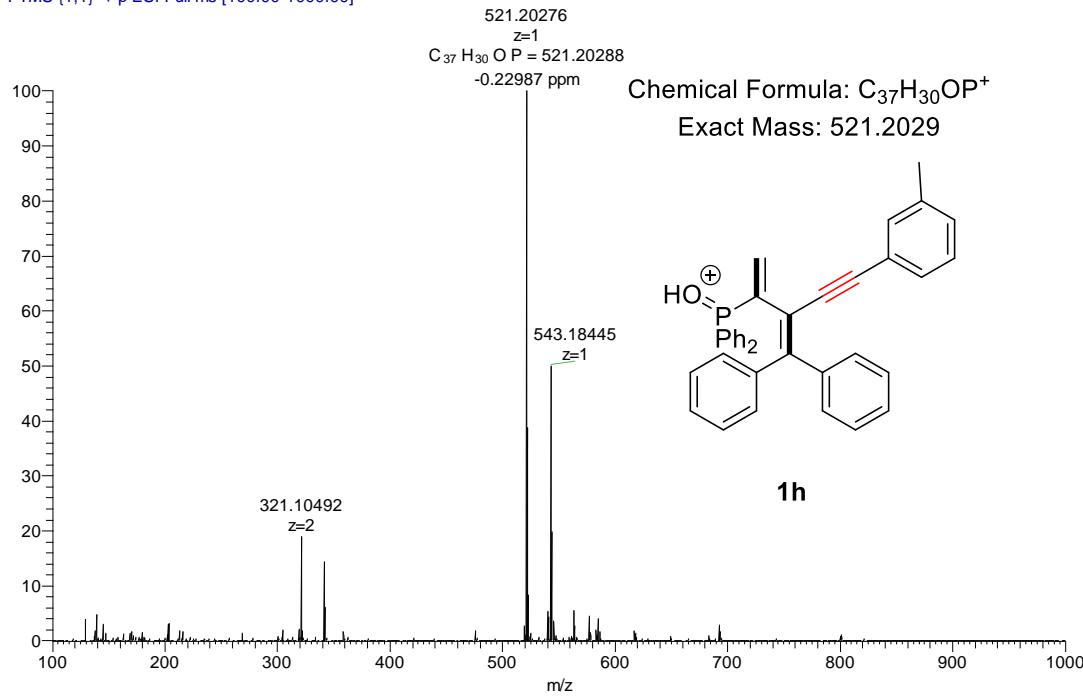
1h



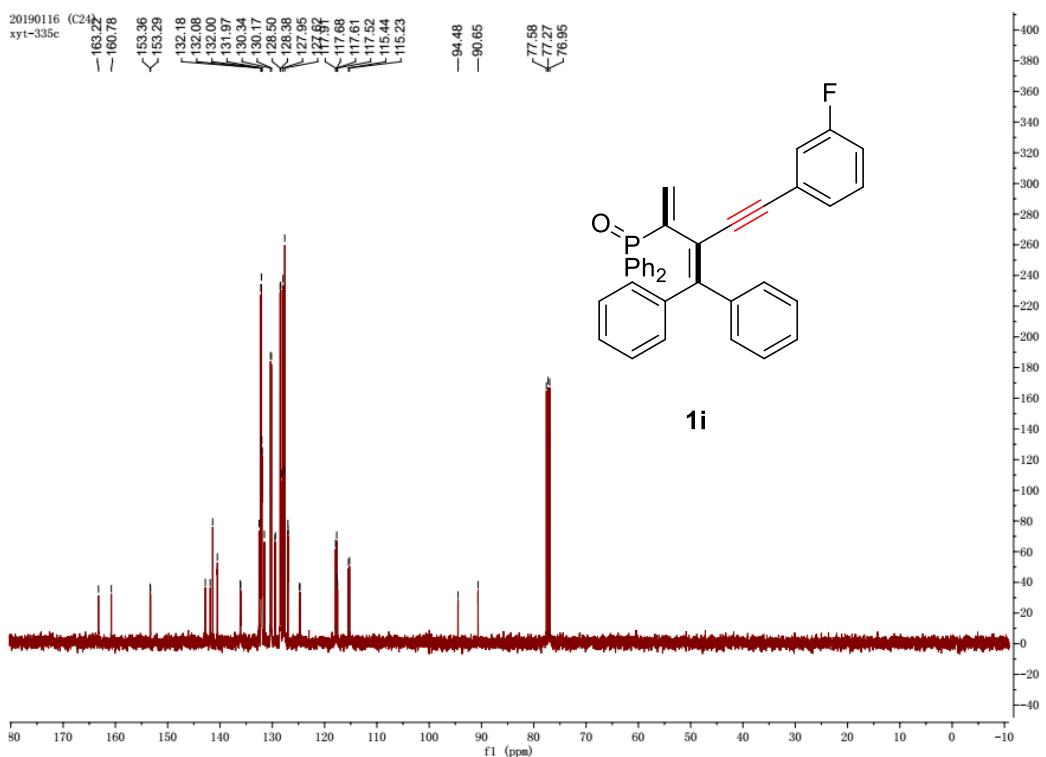
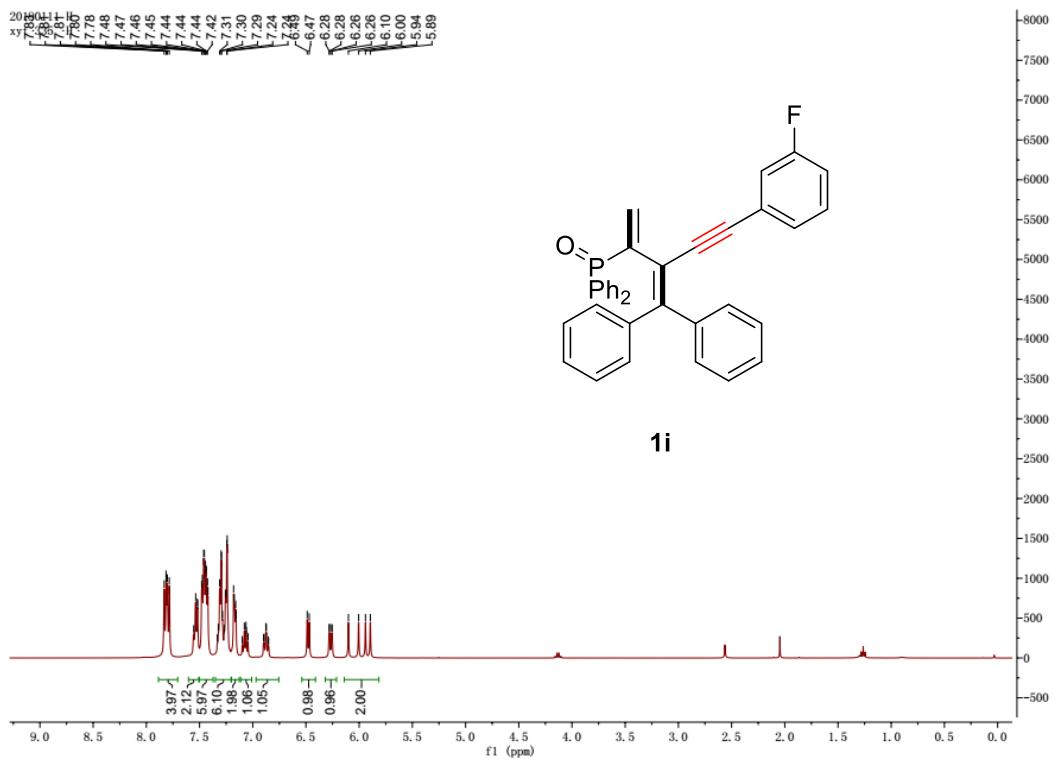
20190111 P
XYT 336 P



20190109-6 #37 RT: 0.56 AV: 1 NL: 2.71E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

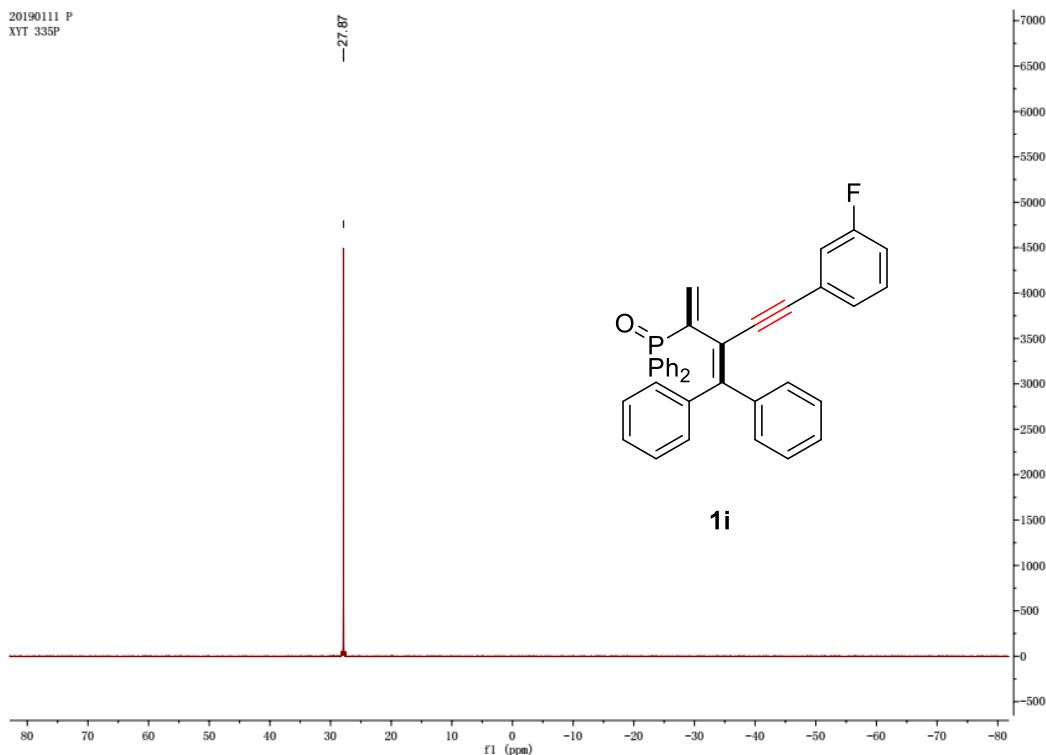


1i



61

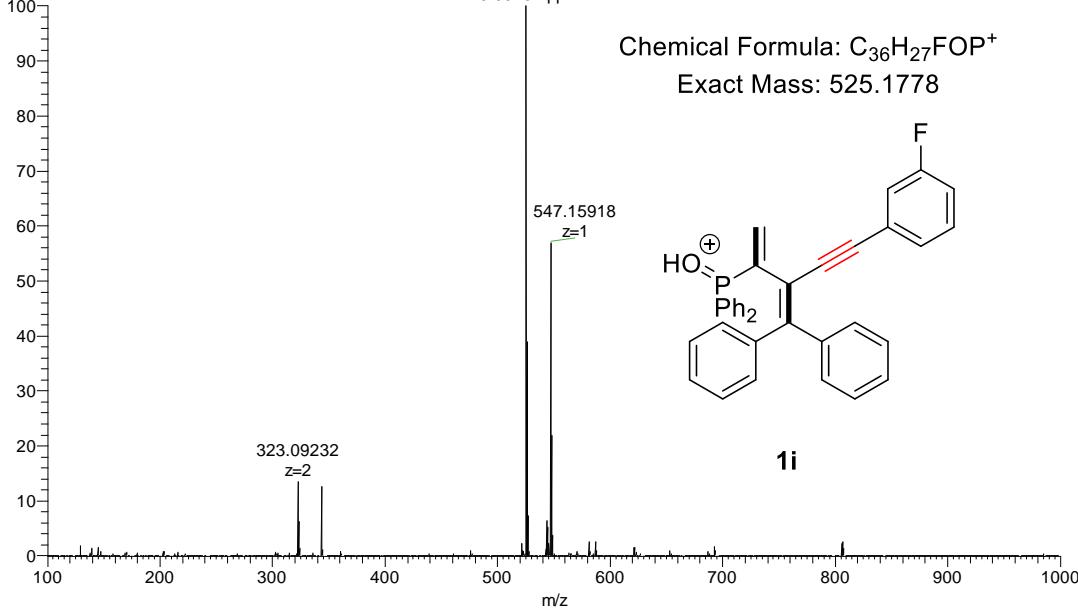
20190111 P
XYT 335P



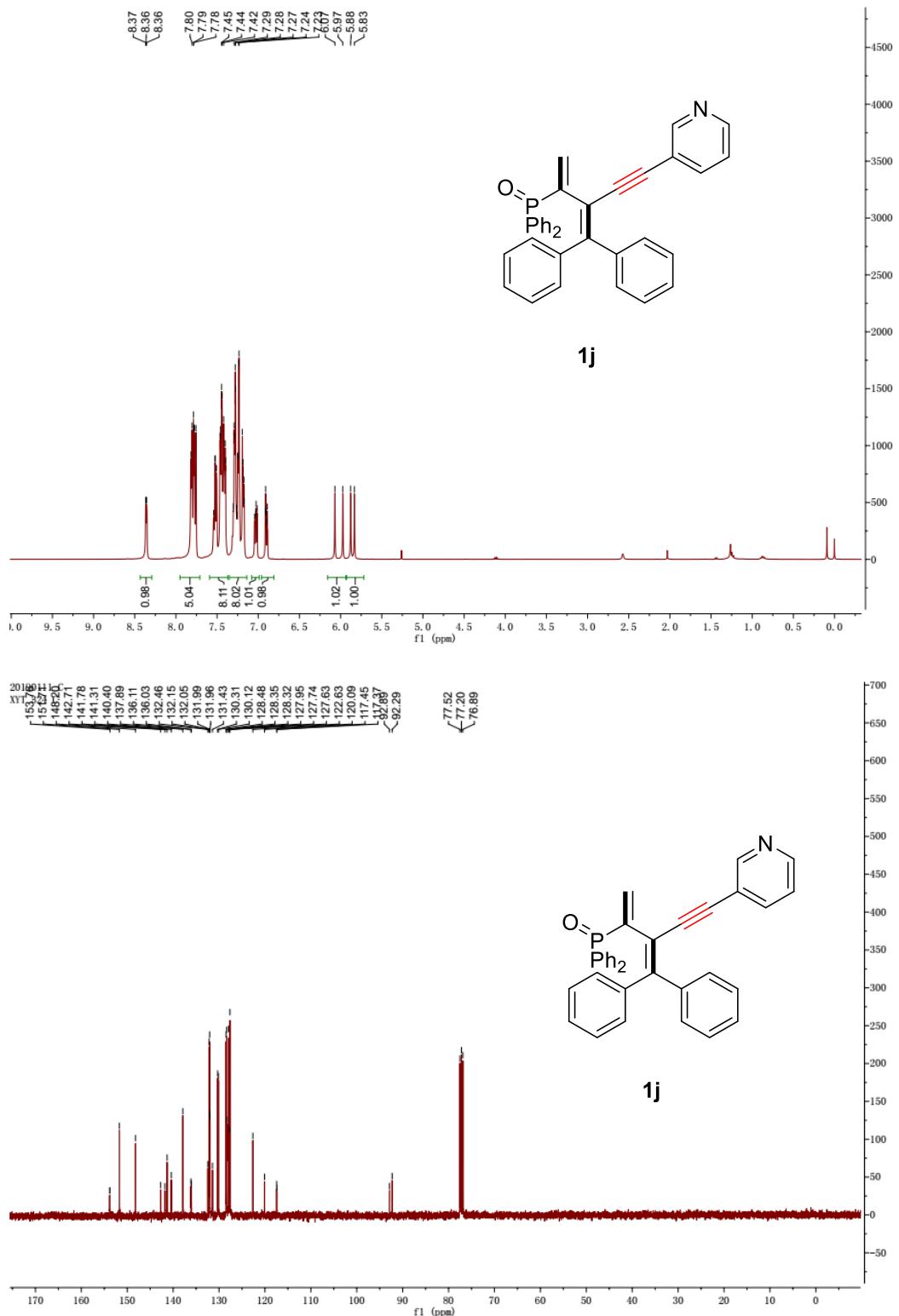
20190109-7 #37 RT: 0.55 AV: 1 NL: 7.98E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

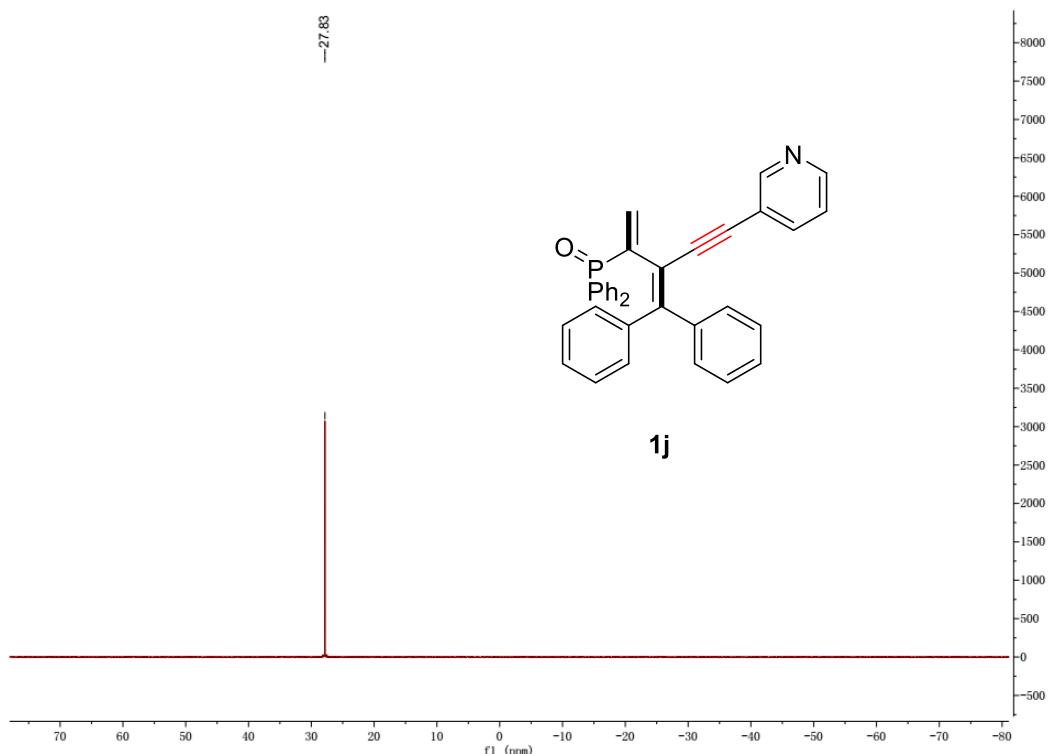
525.17749
 $\text{z}=1$
 $\text{C}_{36}\text{H}_{27}\text{OFOP} = 525.17781$
-0.60261 ppm

Chemical Formula: $\text{C}_{36}\text{H}_{27}\text{FOP}^+$
Exact Mass: 525.1778

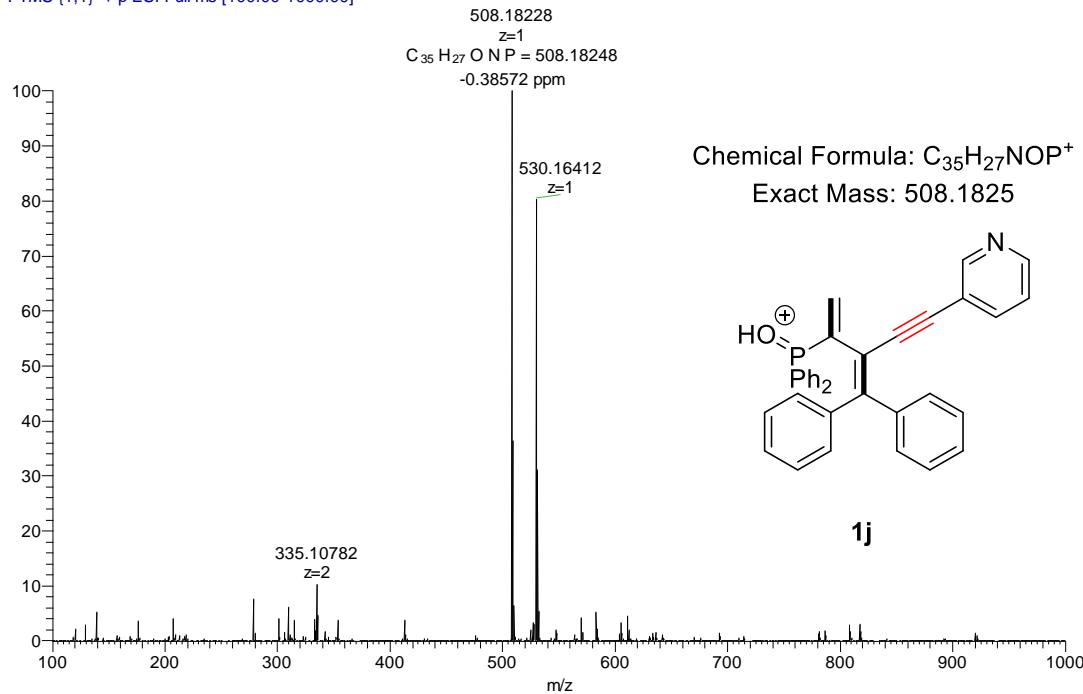


1j

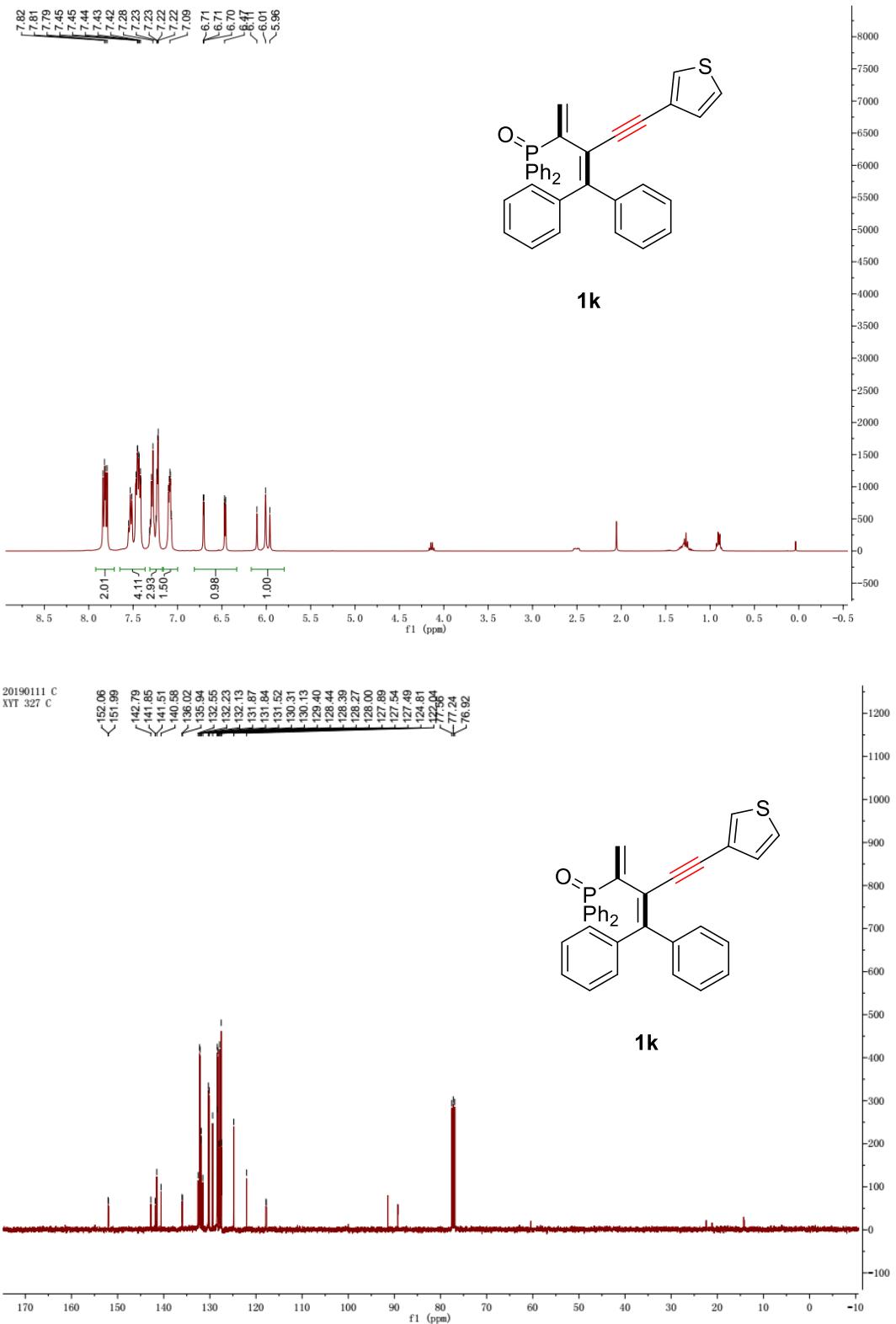


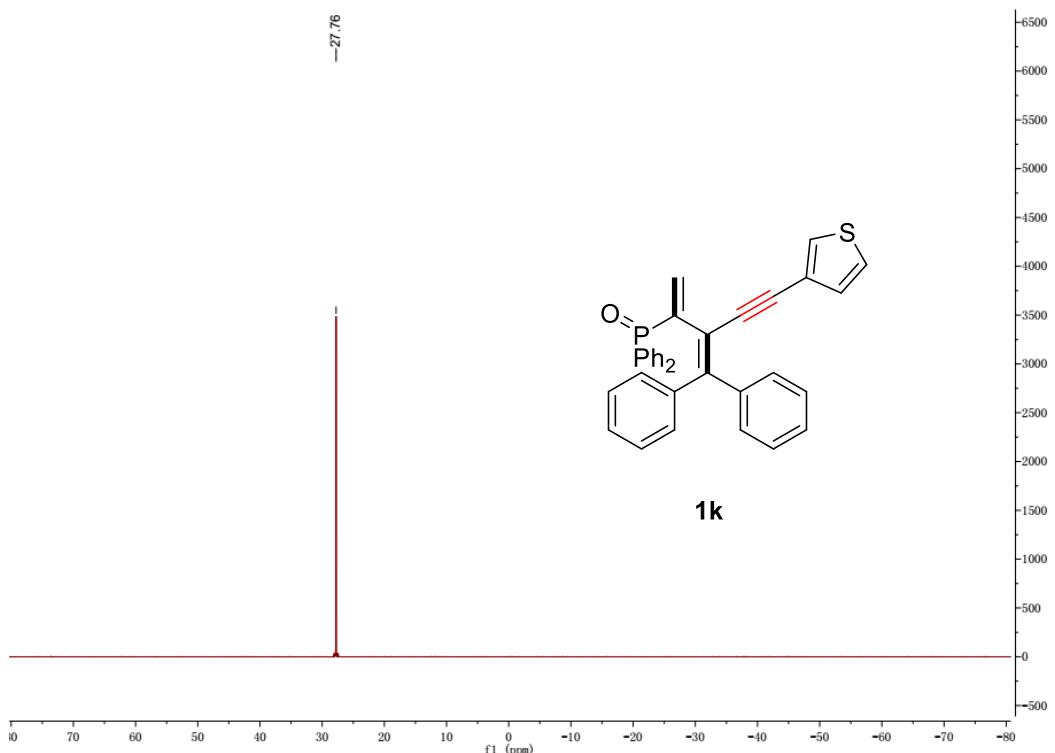


20190109-9 #29 RT: 0.43 AV: 1 NL: 5.56E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



1k

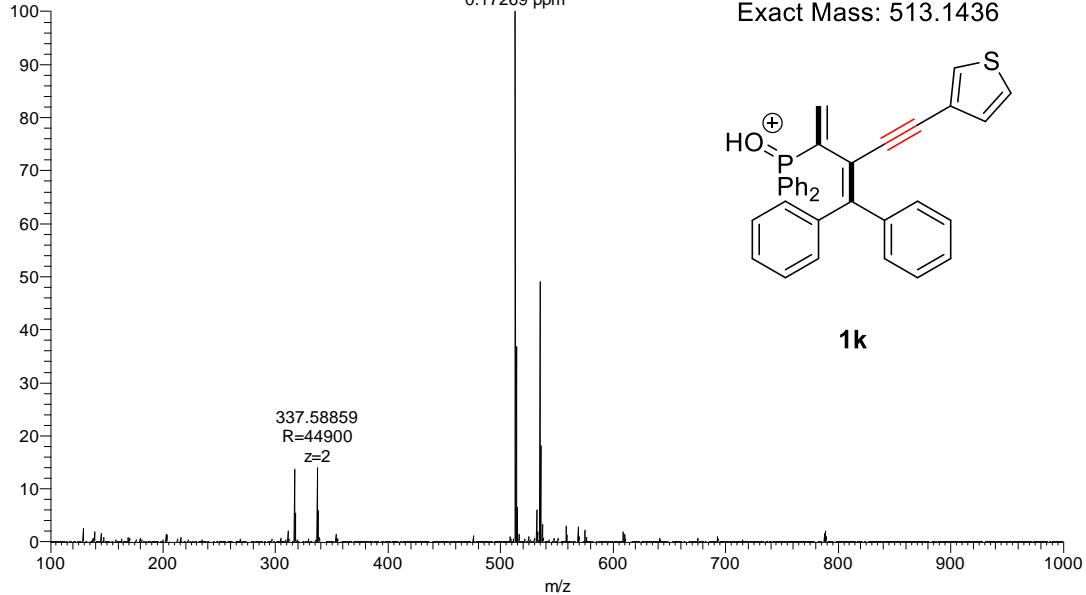




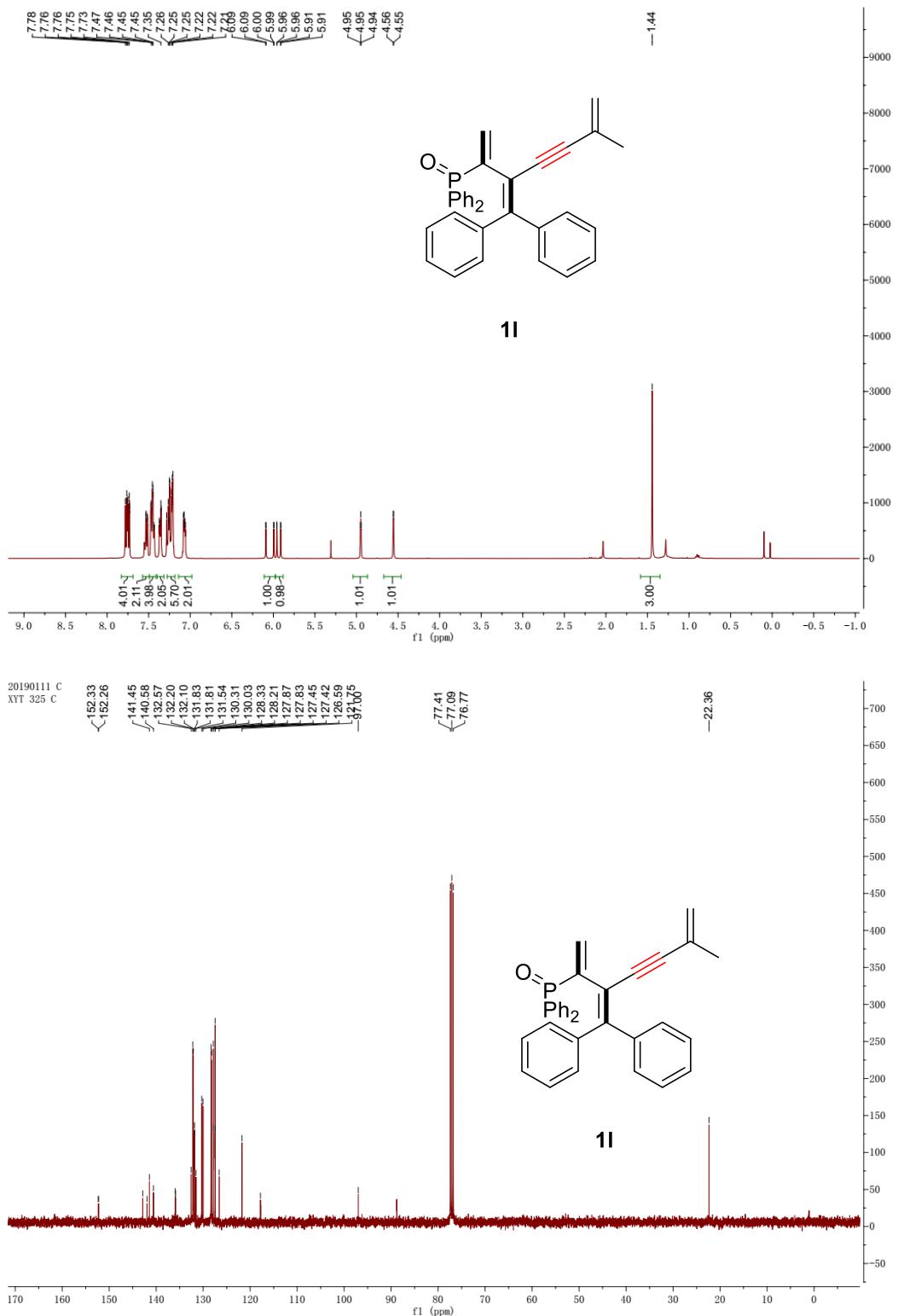
20190109-10 #37 RT: 0.55 AV: 1 NL: 4.84E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

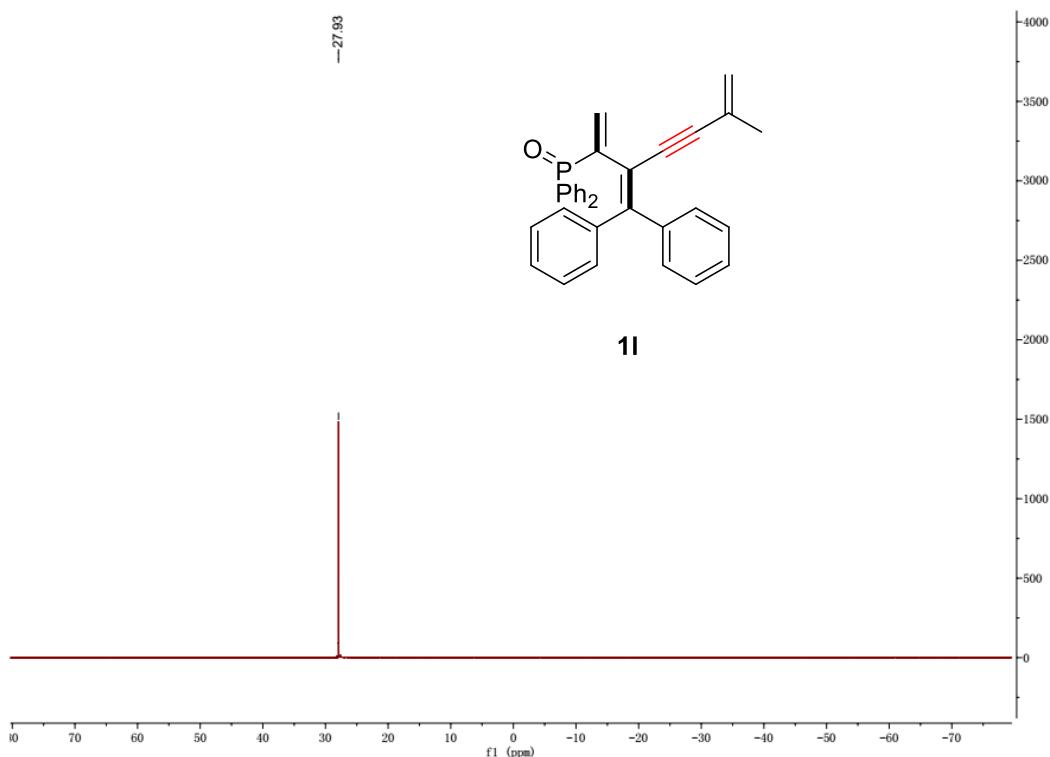
513.14374
R=35700
 $z=1$
 $C_{34}H_{26}OPS = 513.14365$
0.17269 ppm

Chemical Formula: $C_{34}H_{26}OPS^+$
Exact Mass: 513.1436

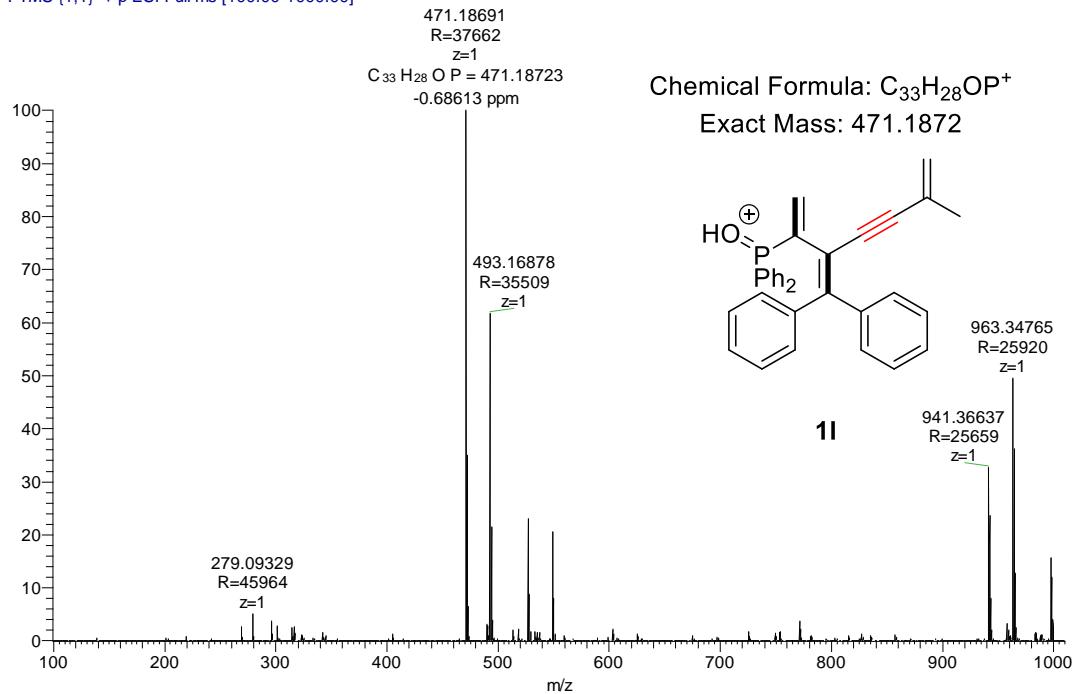


11

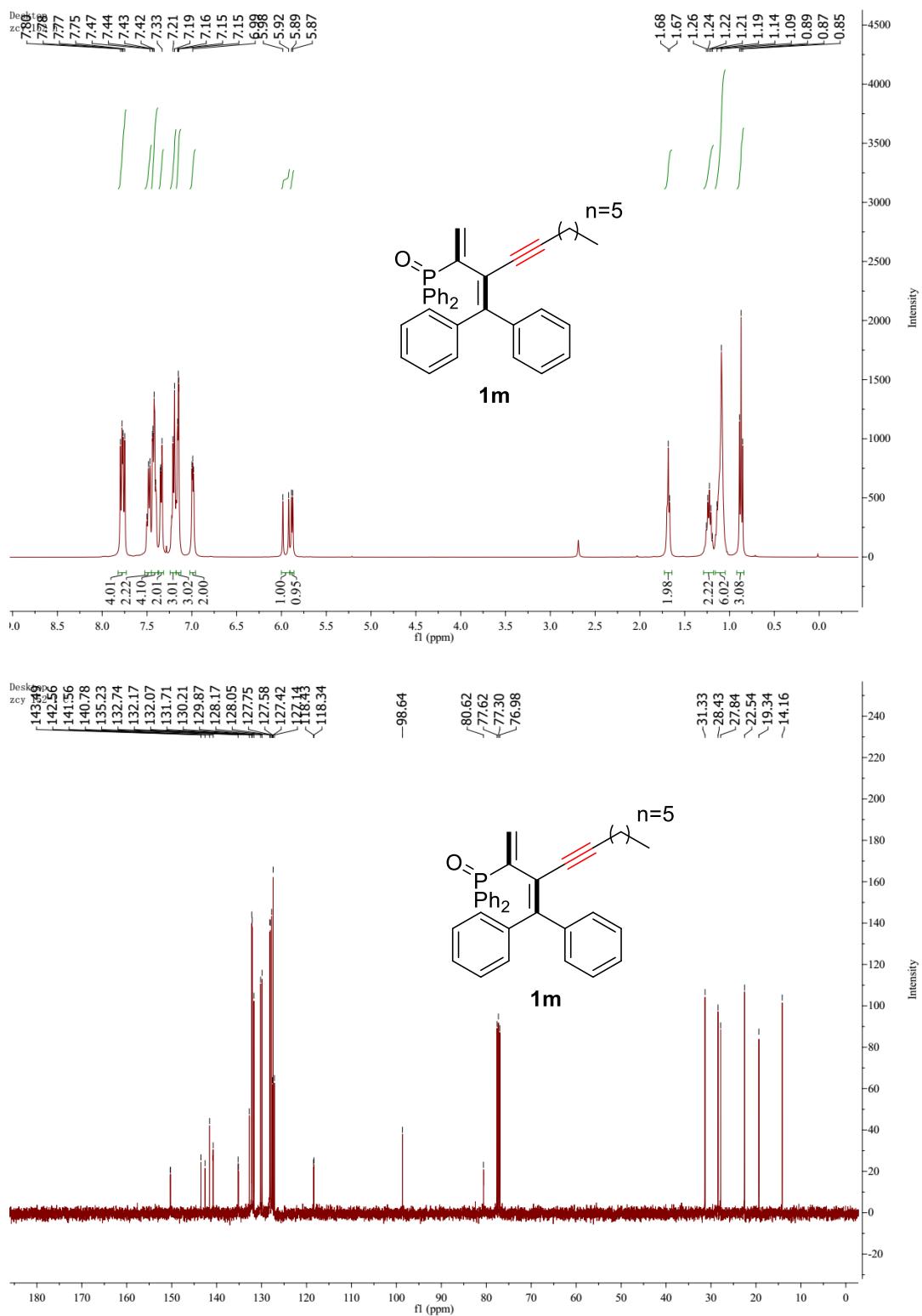




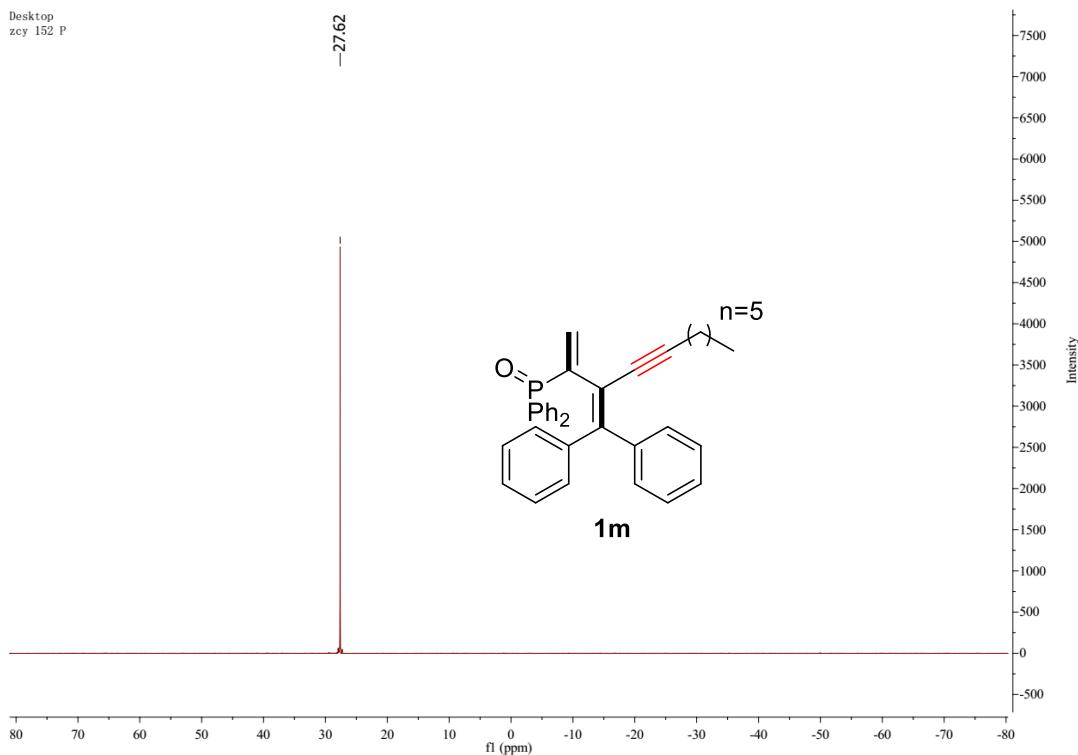
20190109-11 #27 RT: 0.39 AV: 1 SB: 24 0.01-0.06 , 0.70-1.03 NL: 1.01E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



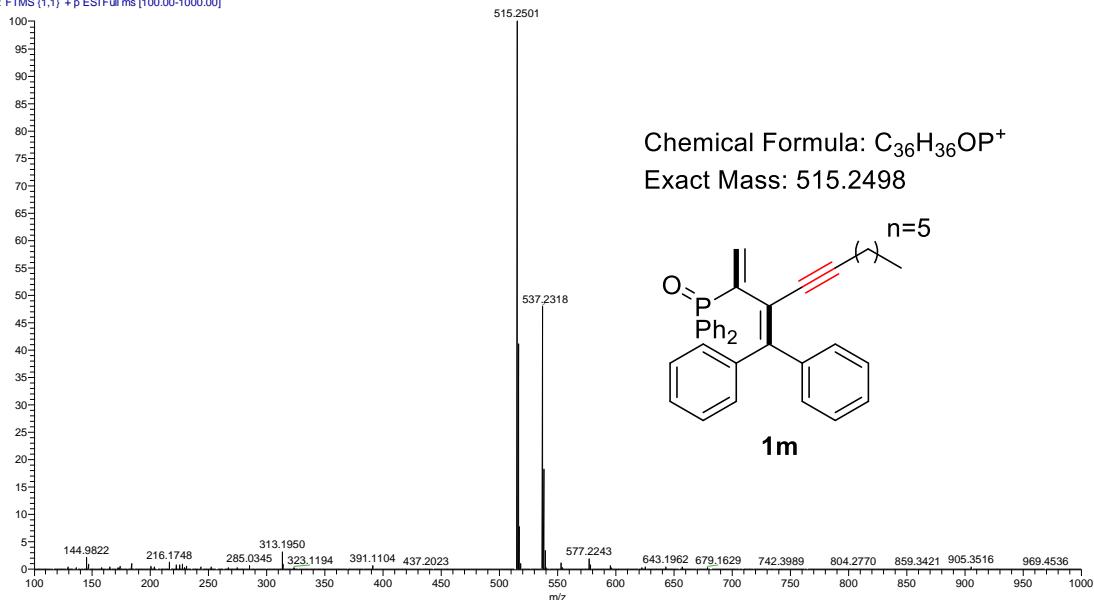
1m



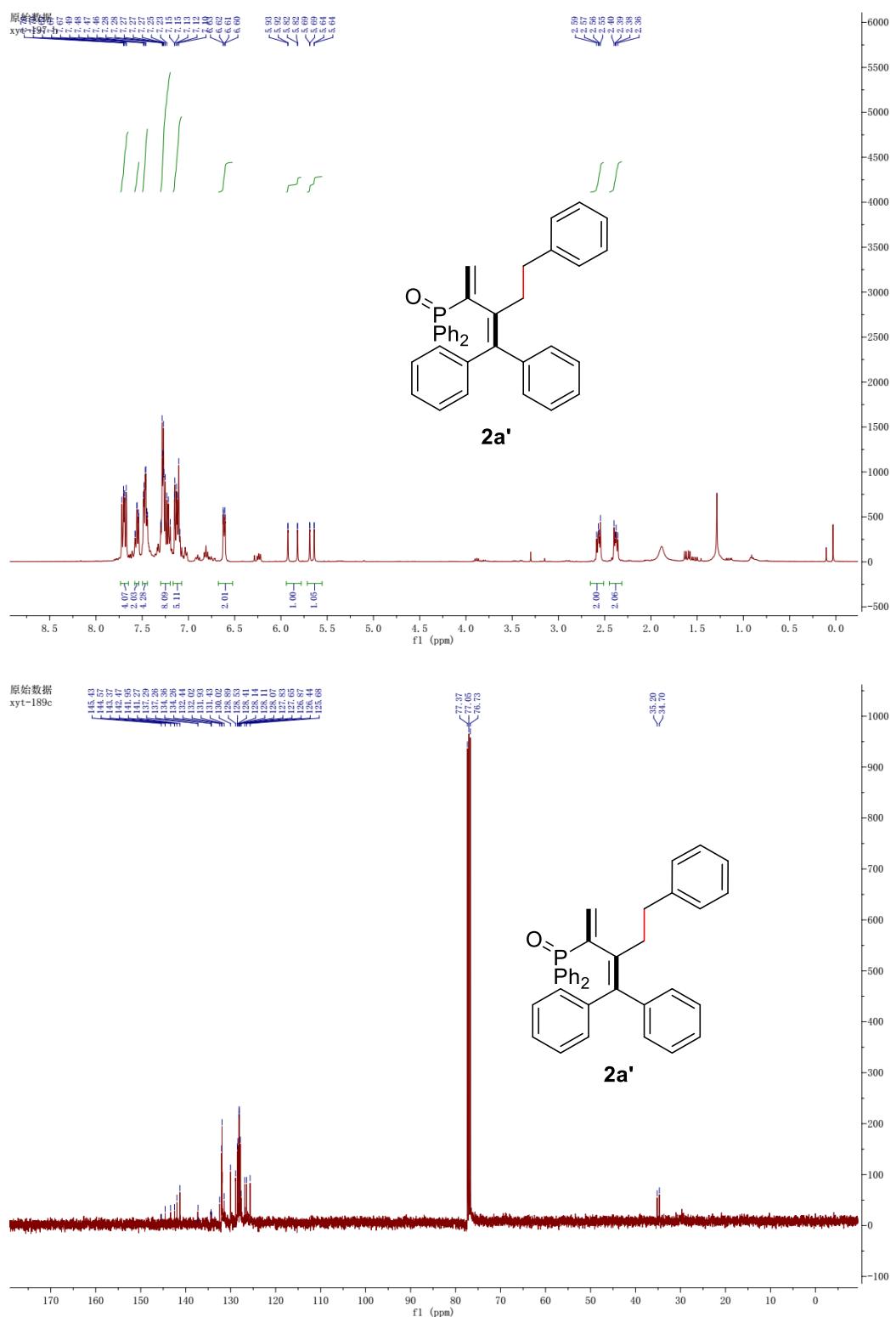
Desktop
zcy 152 P

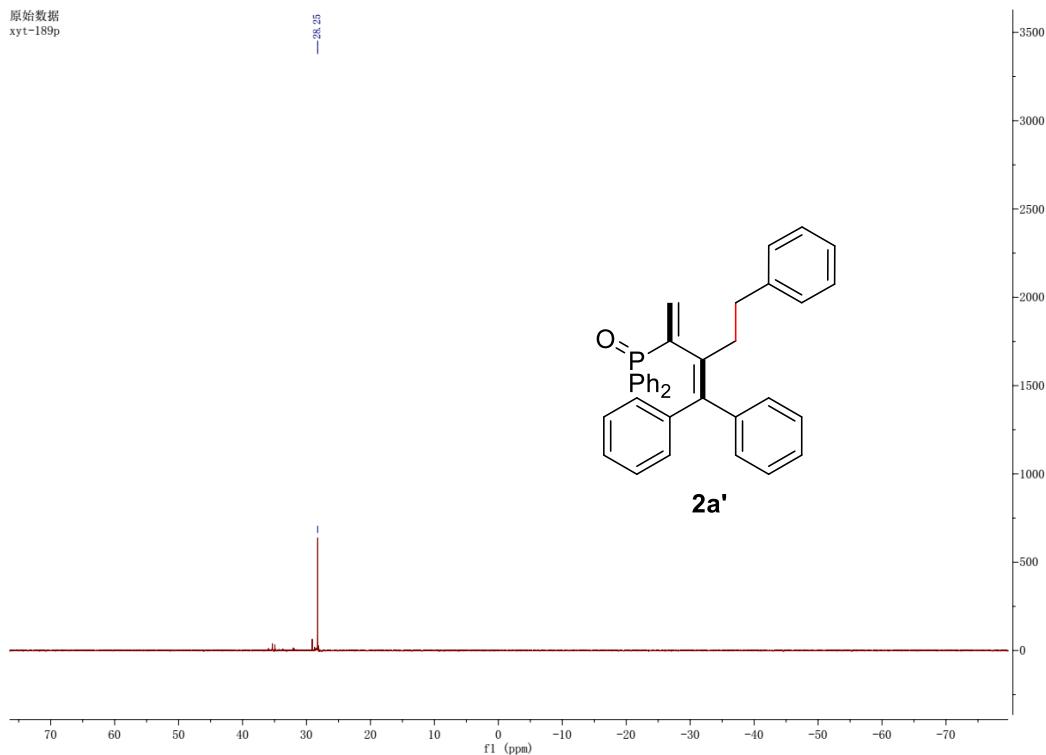


20190708-16 #7 RT: 0.11 AV: 1 NL: 3.70E5
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]

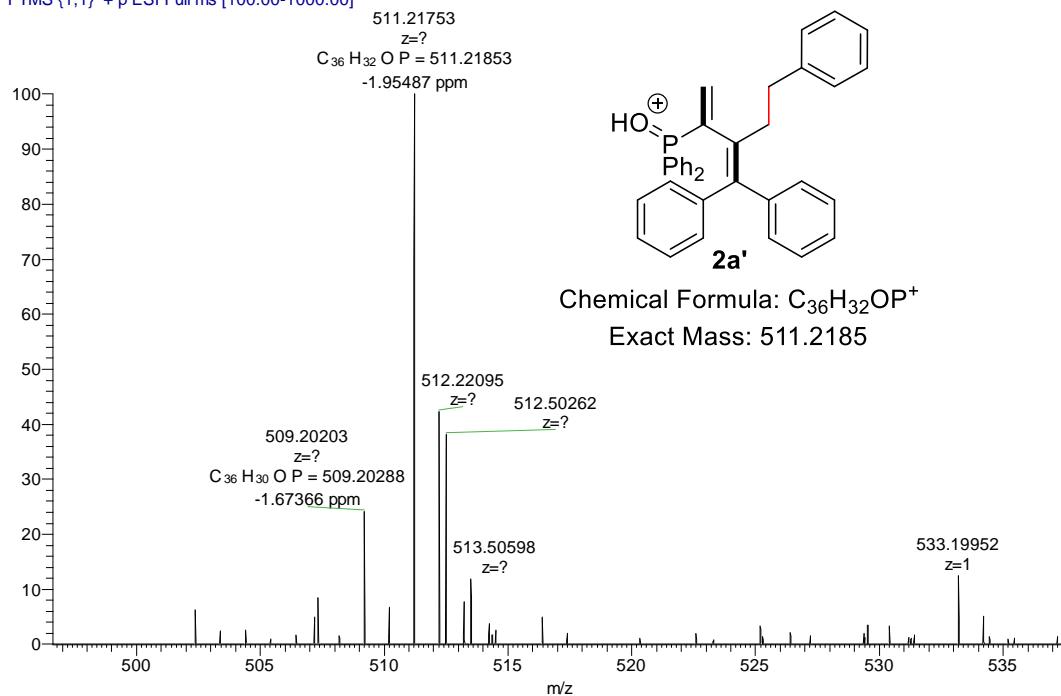


2a'

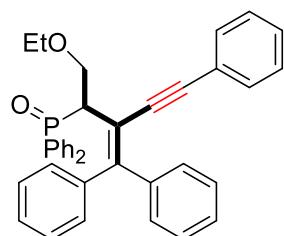
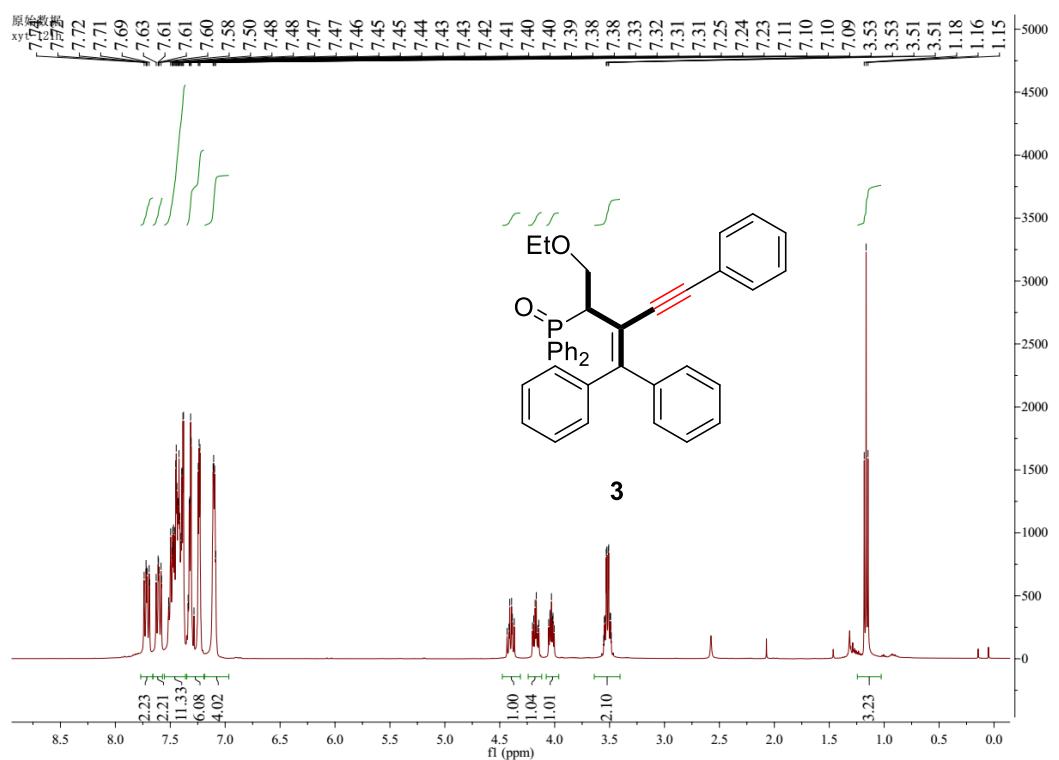




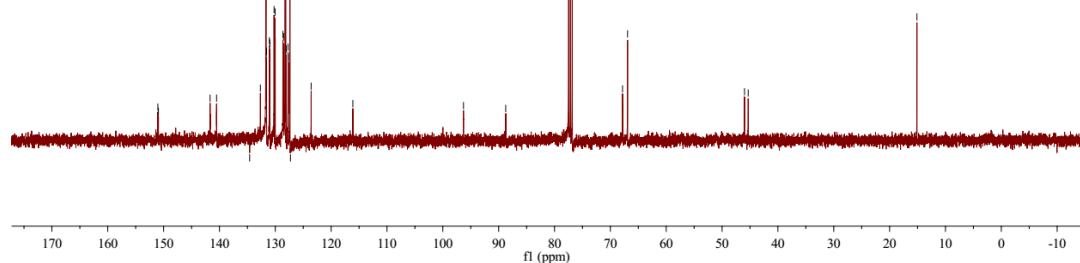
NJNY-20180812-2 #29 RT: 0.40 AV: 1 NL: 1.18E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



3

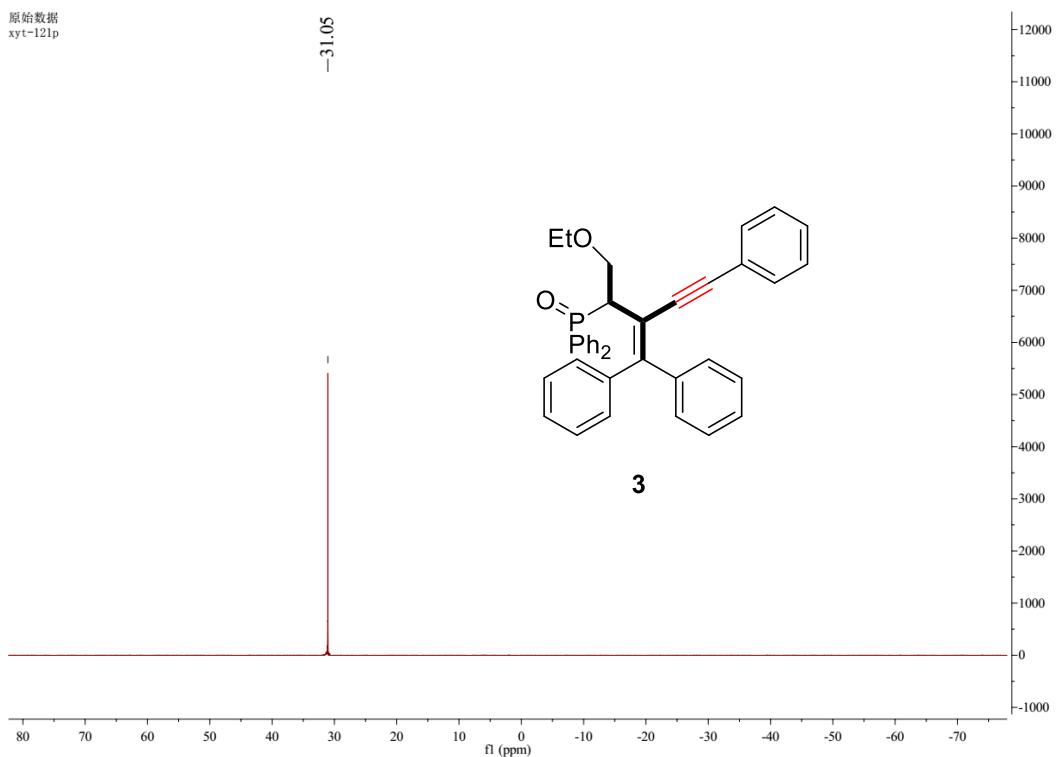


3



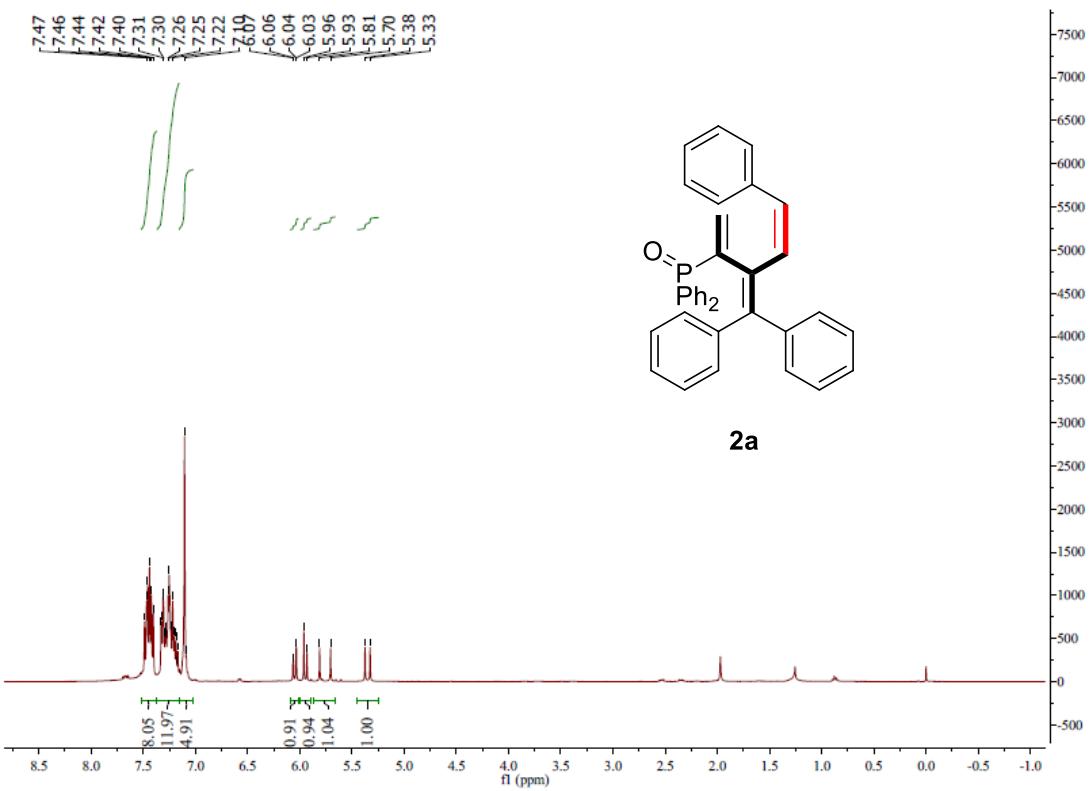
73

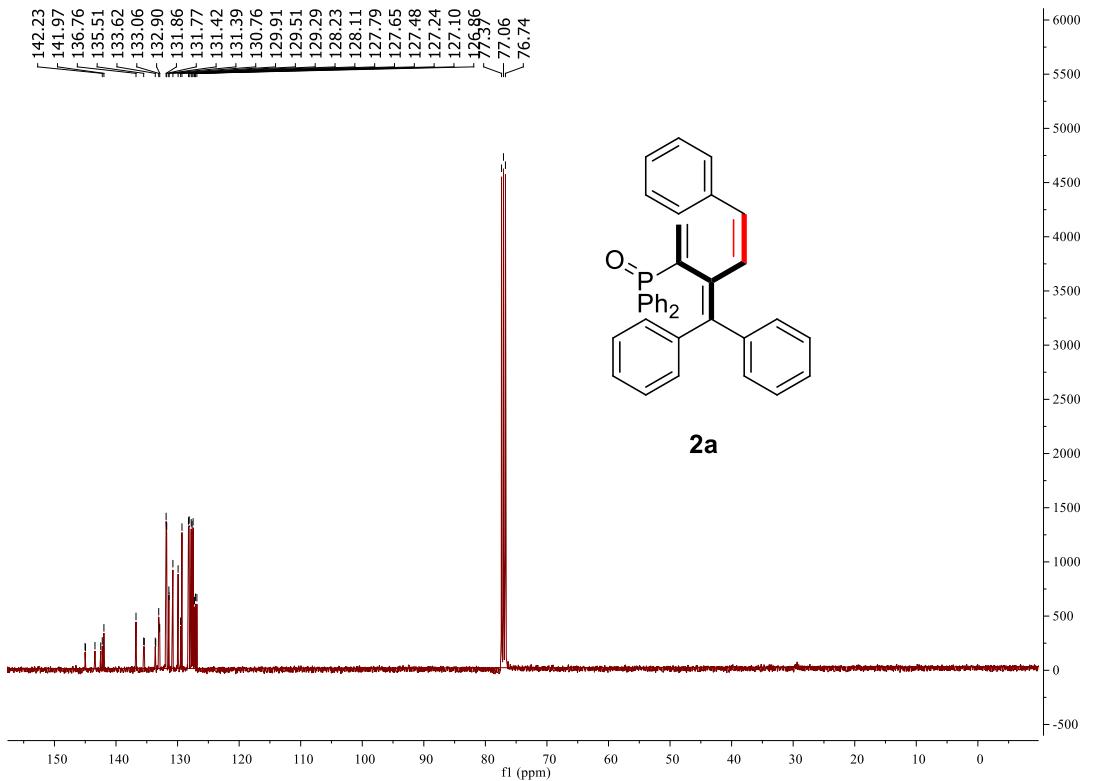
原始数据
xyt-121p



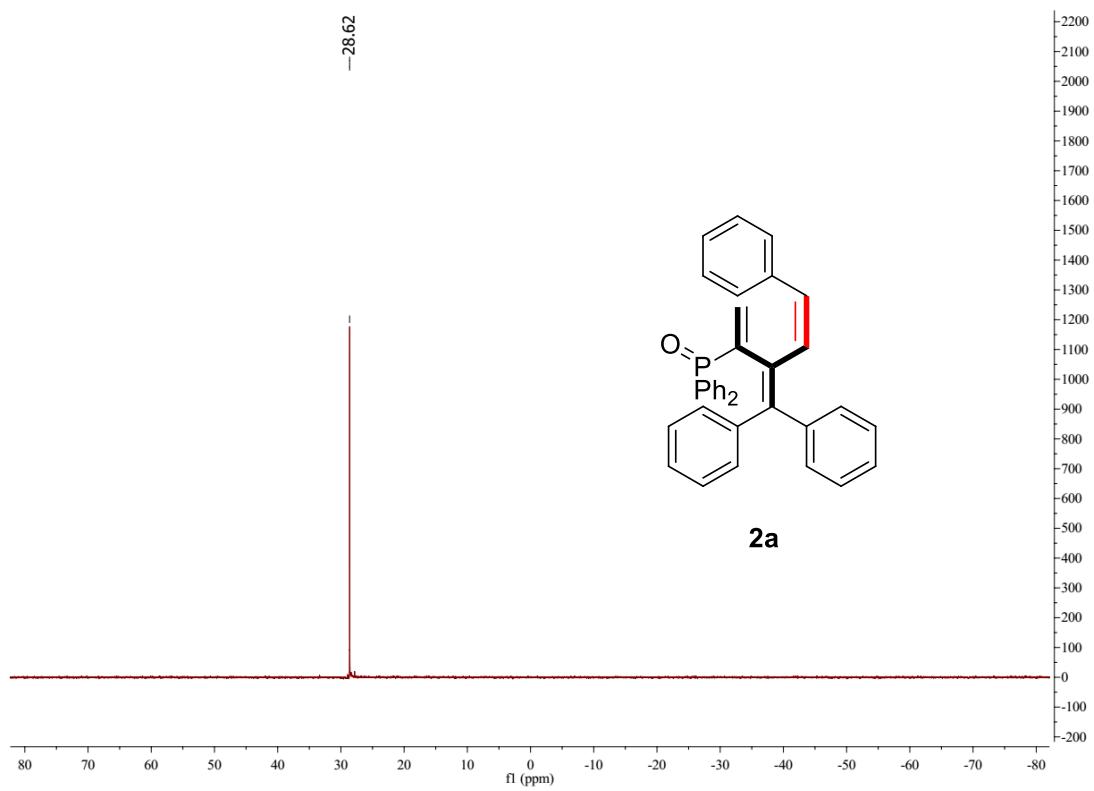
3

2a





2a

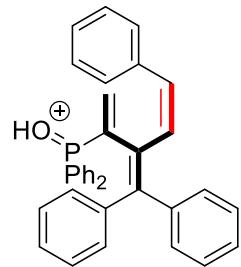
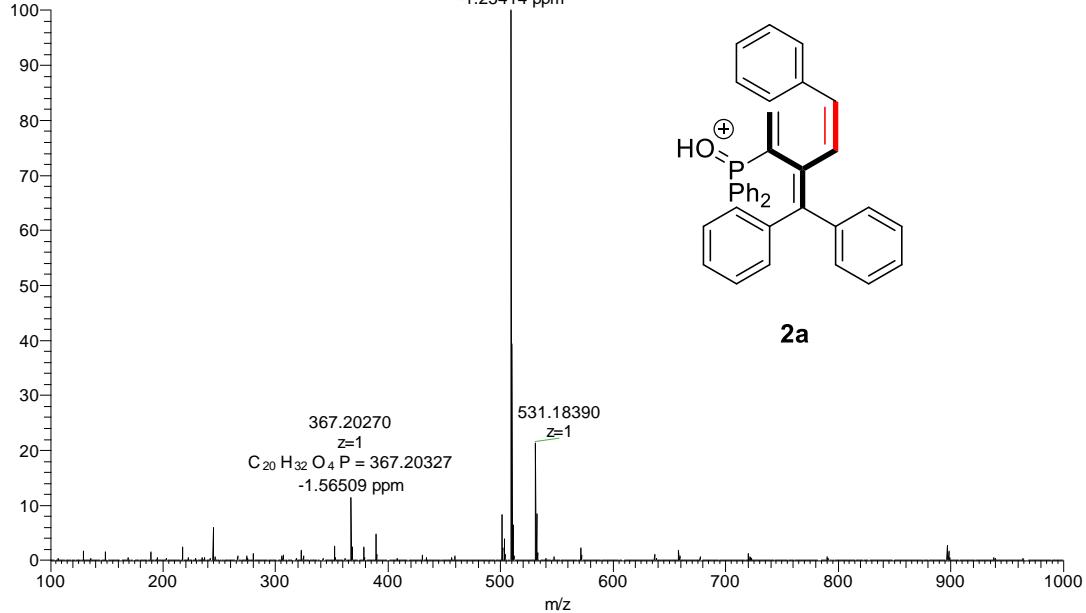


2a

NJNY-20180817-27 #31 RT: 0.45 AV: 1 NL: 1.86E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

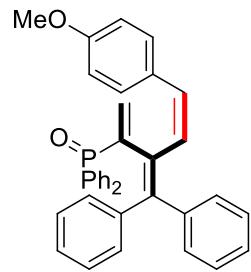
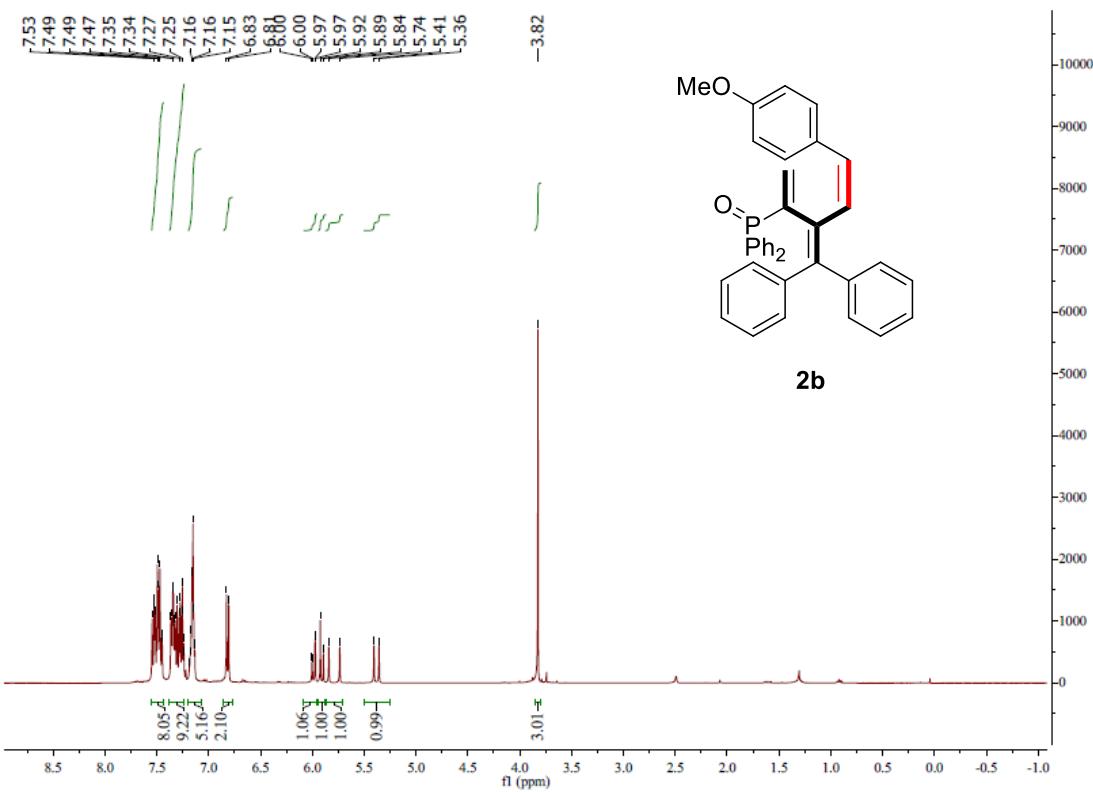
509.20224
 $z=1$
 $C_{36}H_{30}OP = 509.20288$
 -1.25414 ppm

Chemical Formula: $C_{36}H_{30}OP^+$
Exact Mass: 509.2029

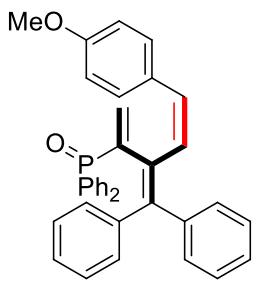
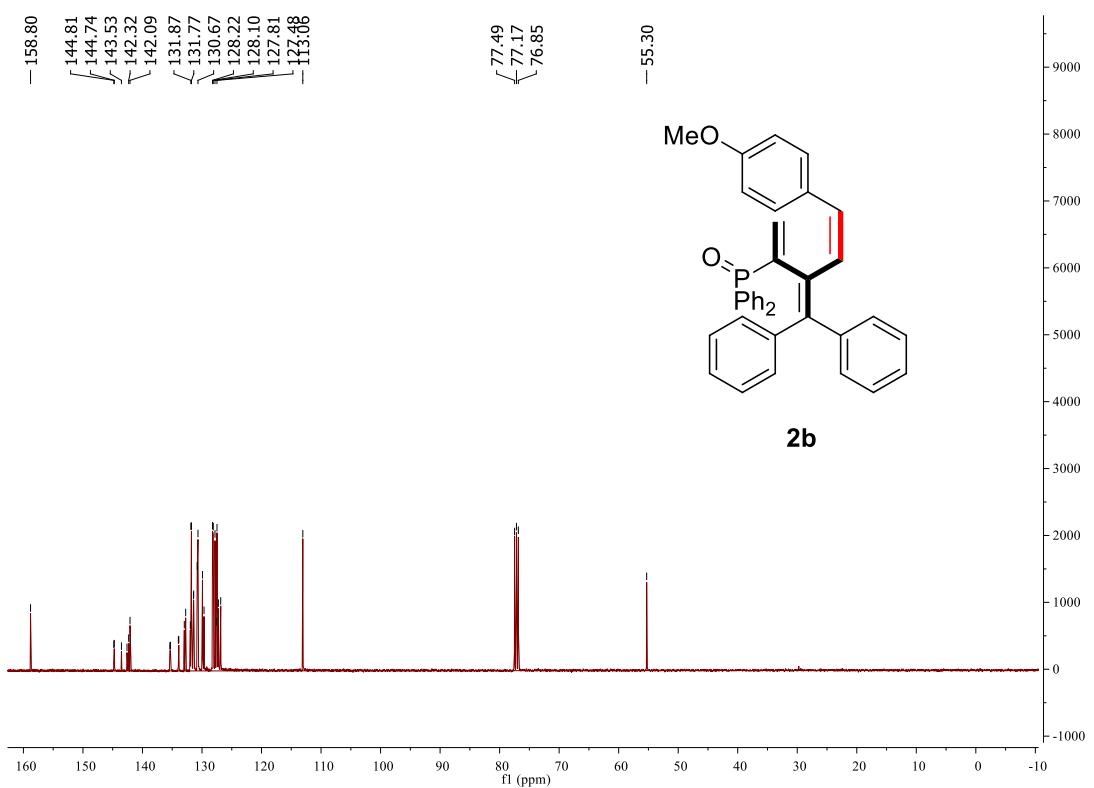


2a

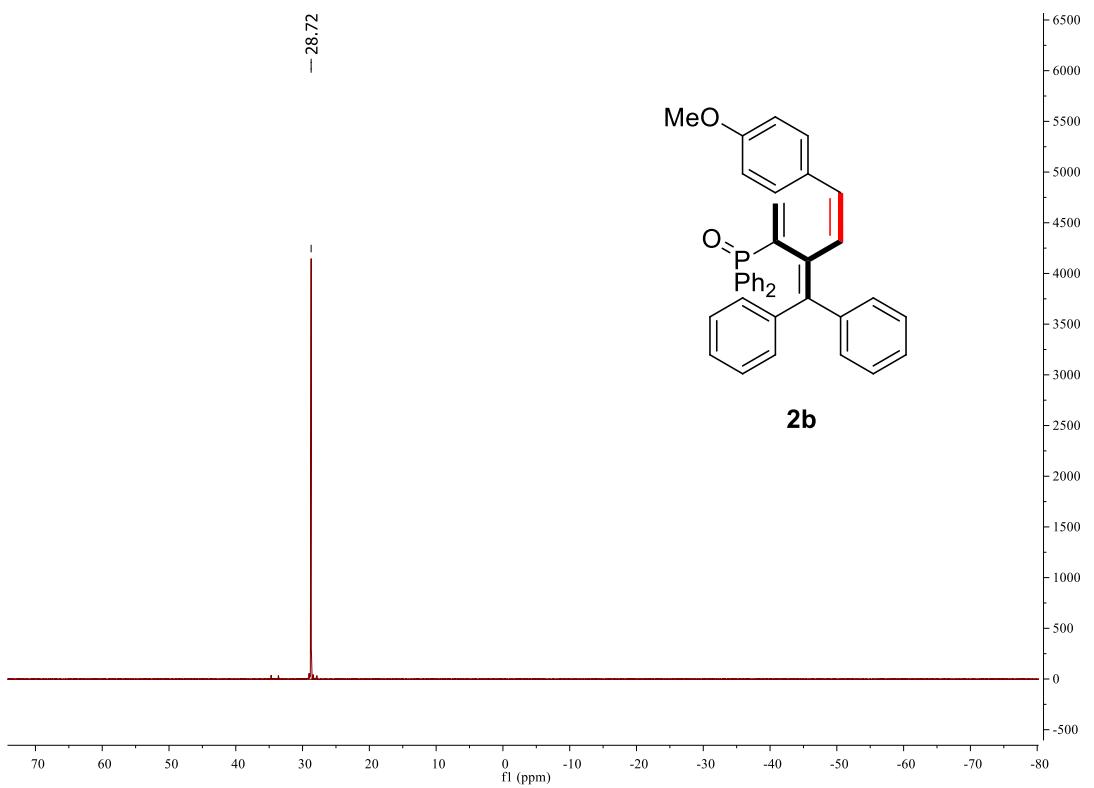
2b



2b

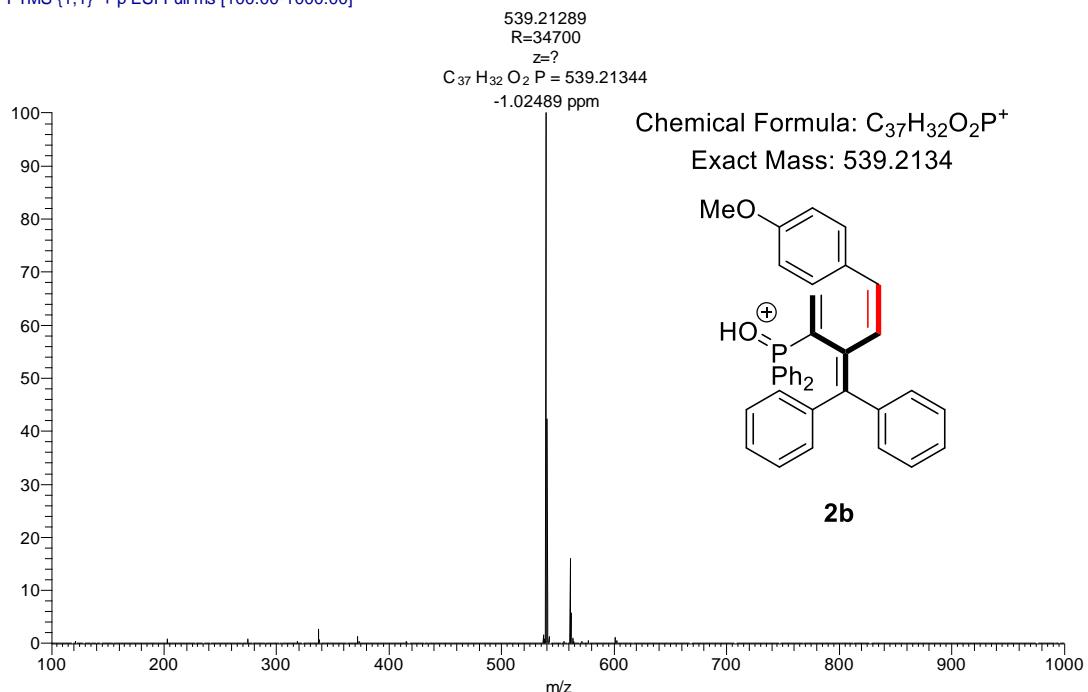


2b

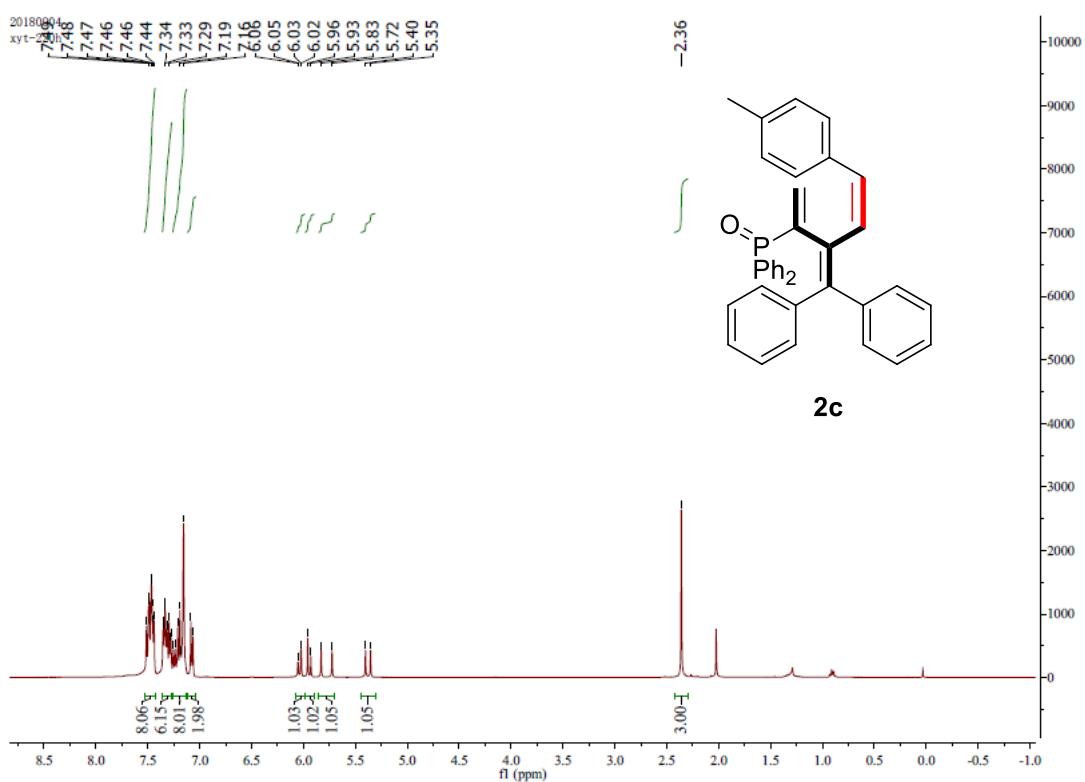


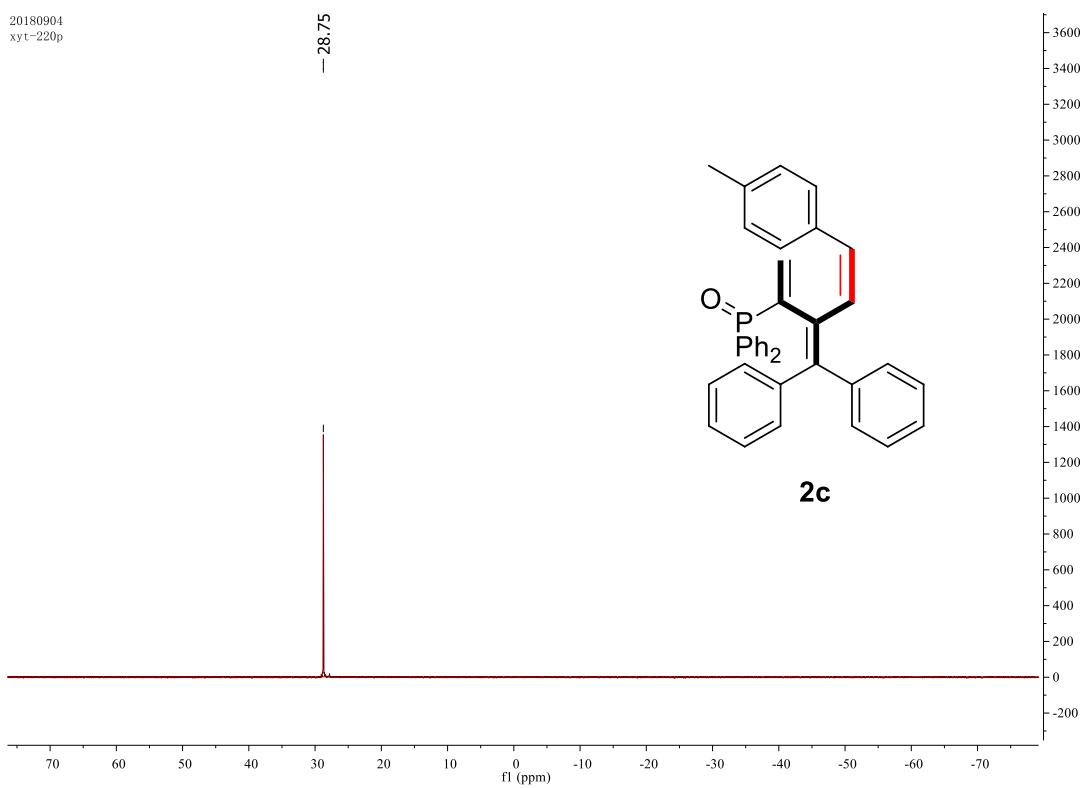
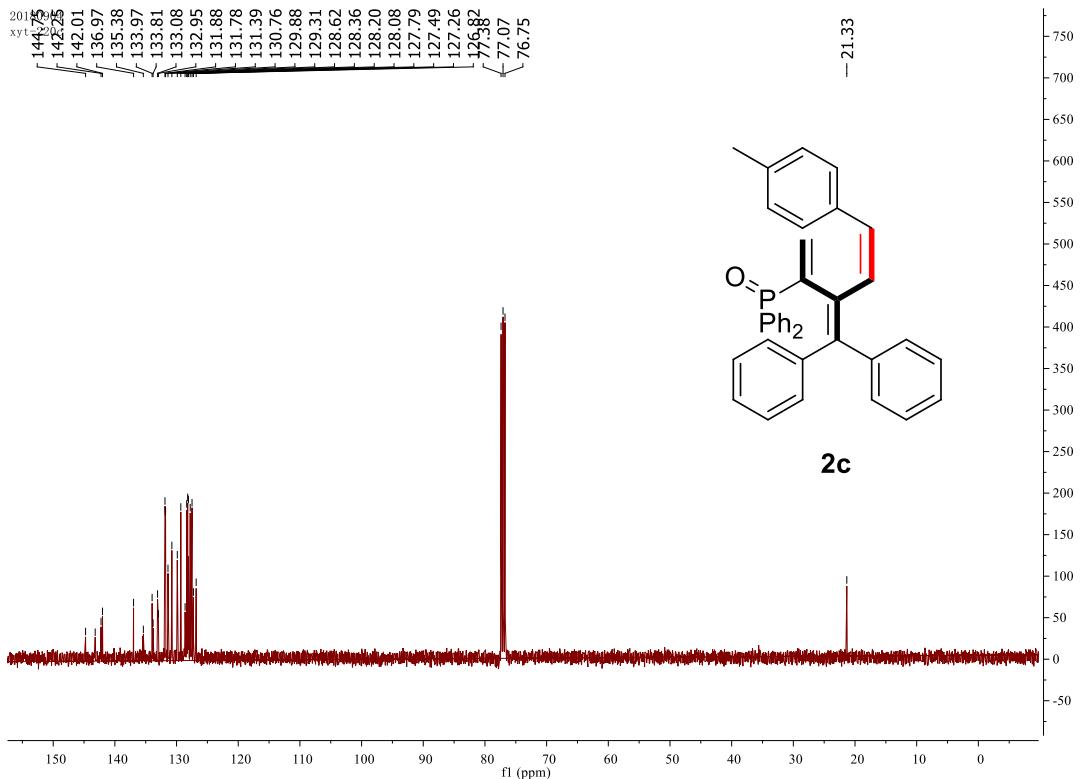
2b

njny-20180903-7 #31 RT: 0.46 AV: 1 NL: 2.76E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

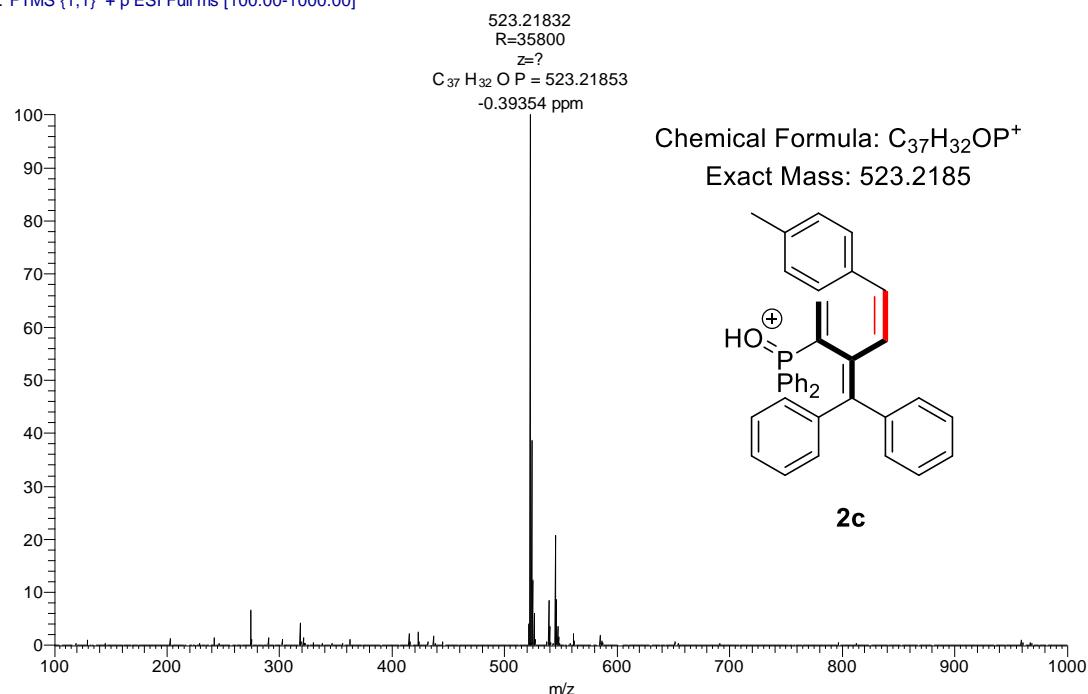


2c

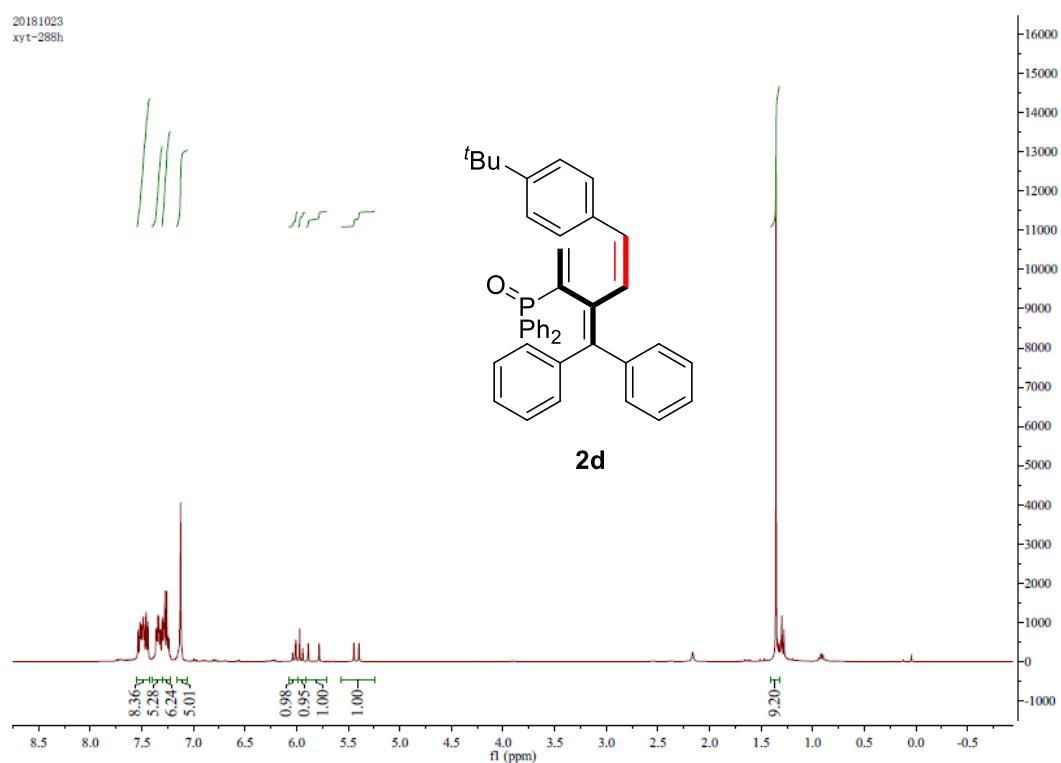


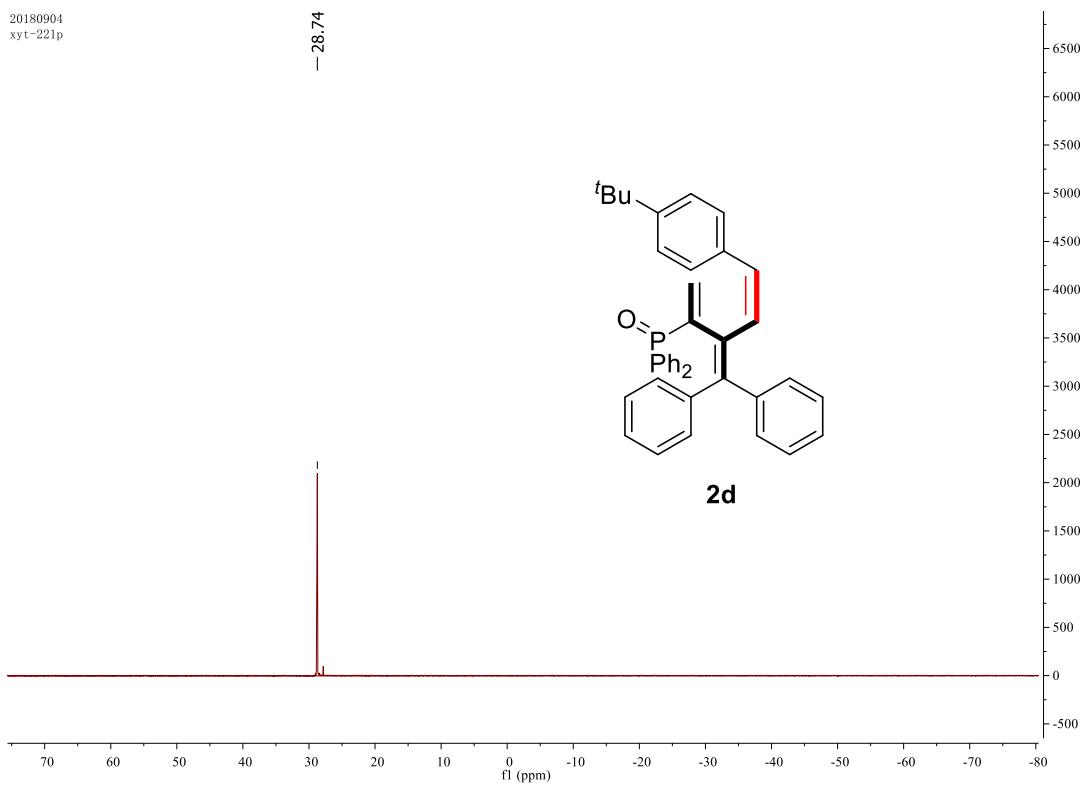
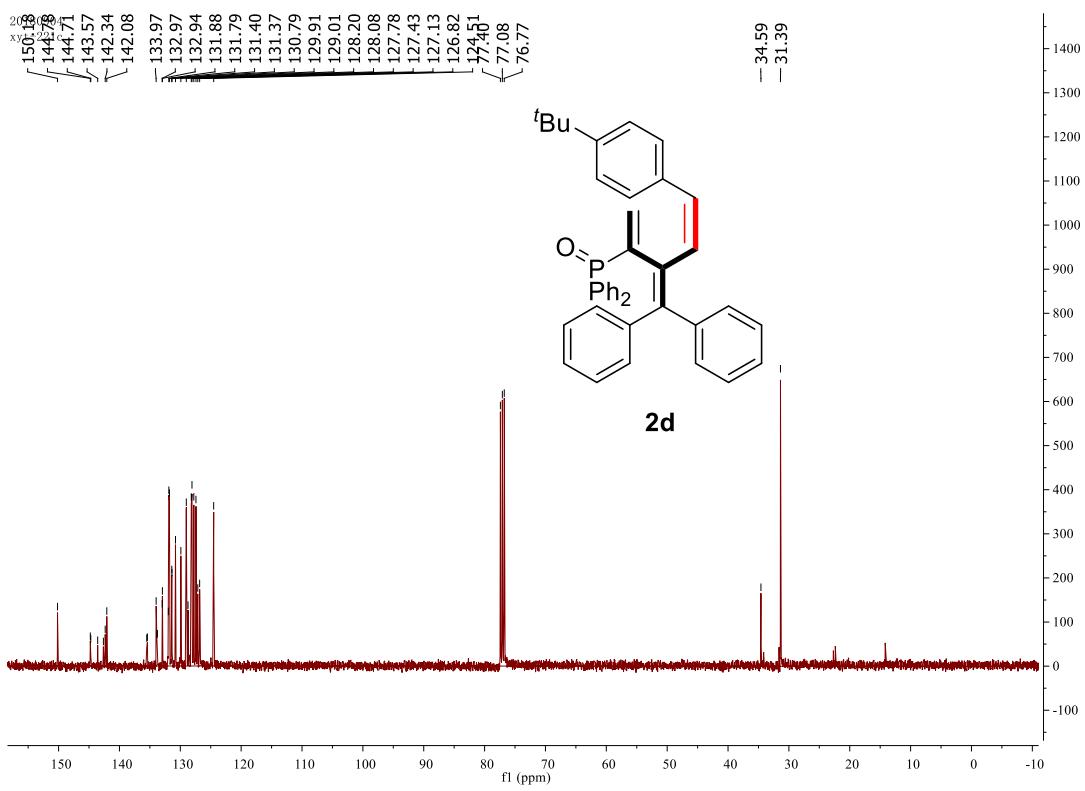


njny-20180903-8 #39 RT: 0.59 AV: 1 NL: 8.89E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

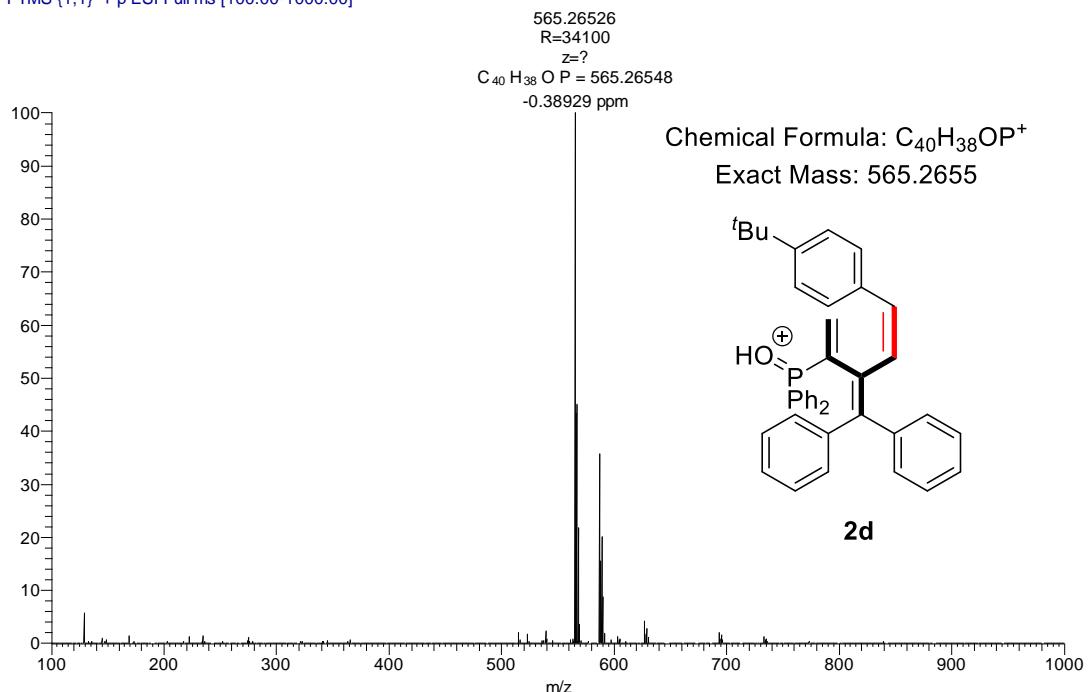


2d



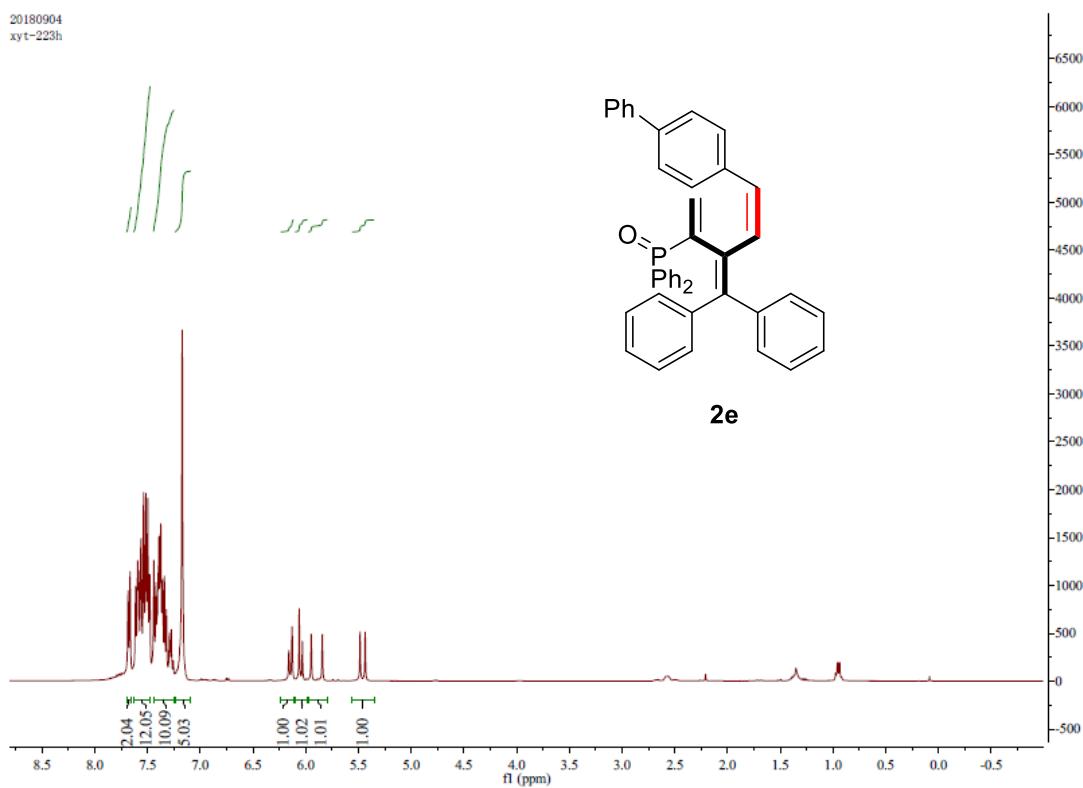


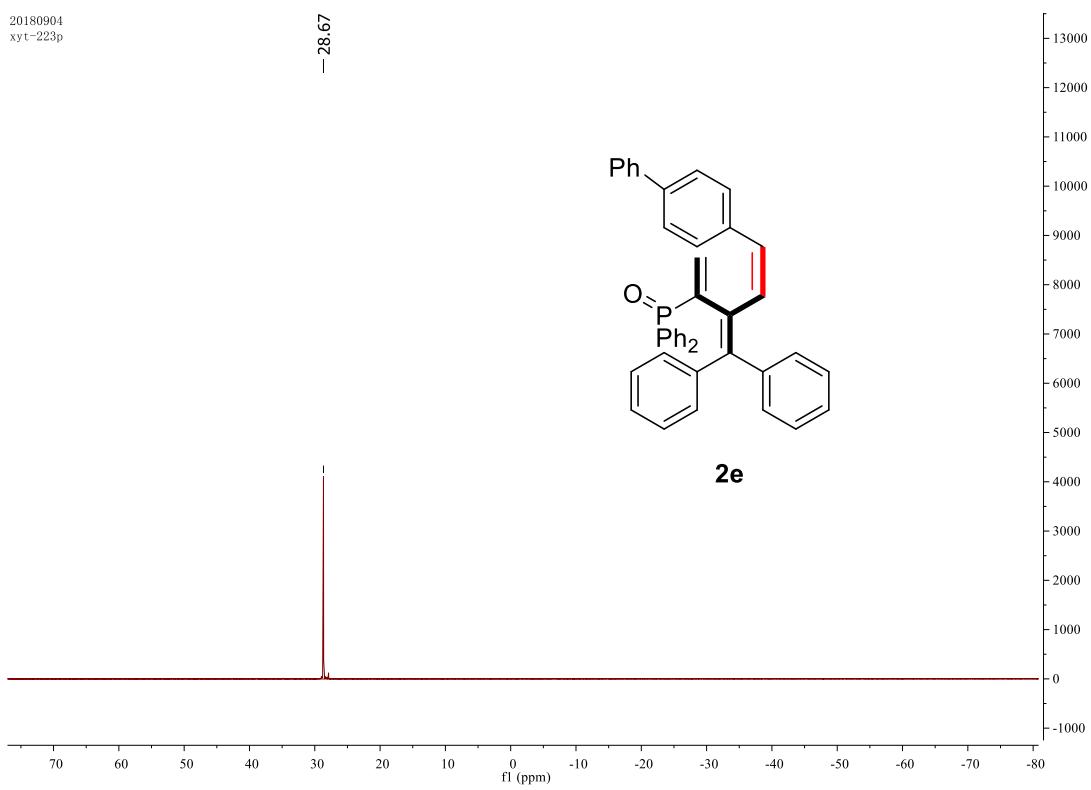
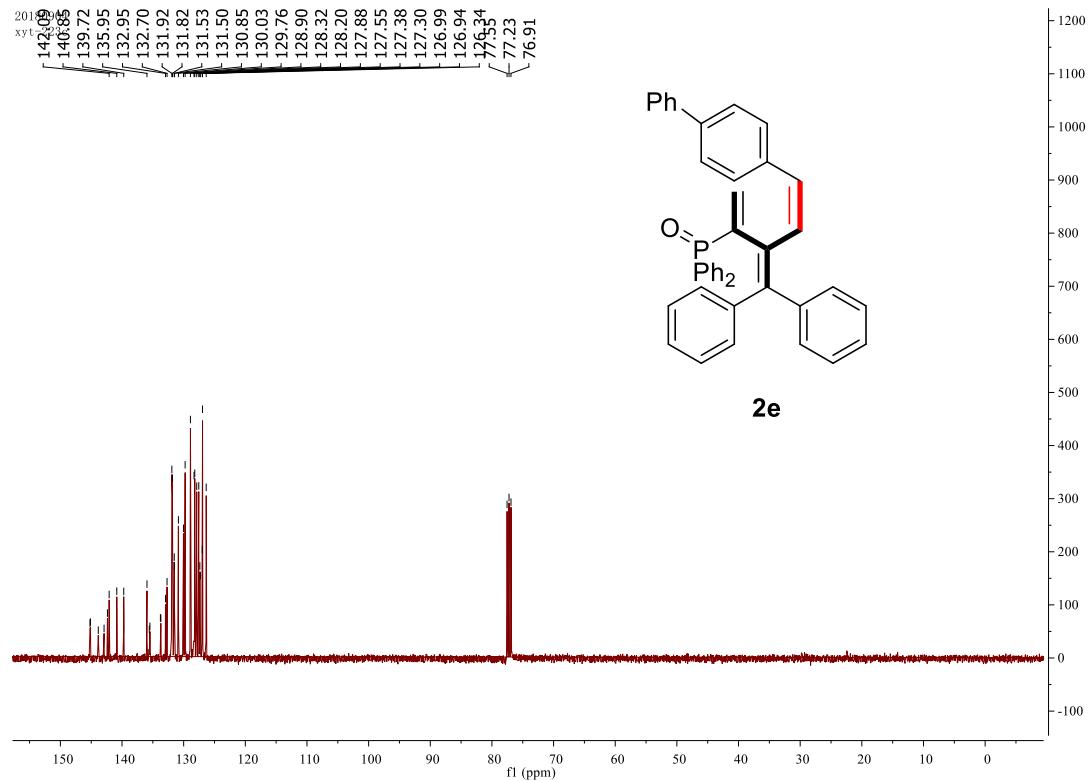
njny-20180903-10 #31 RT: 0.48 AV: 1 NL: 3.41E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



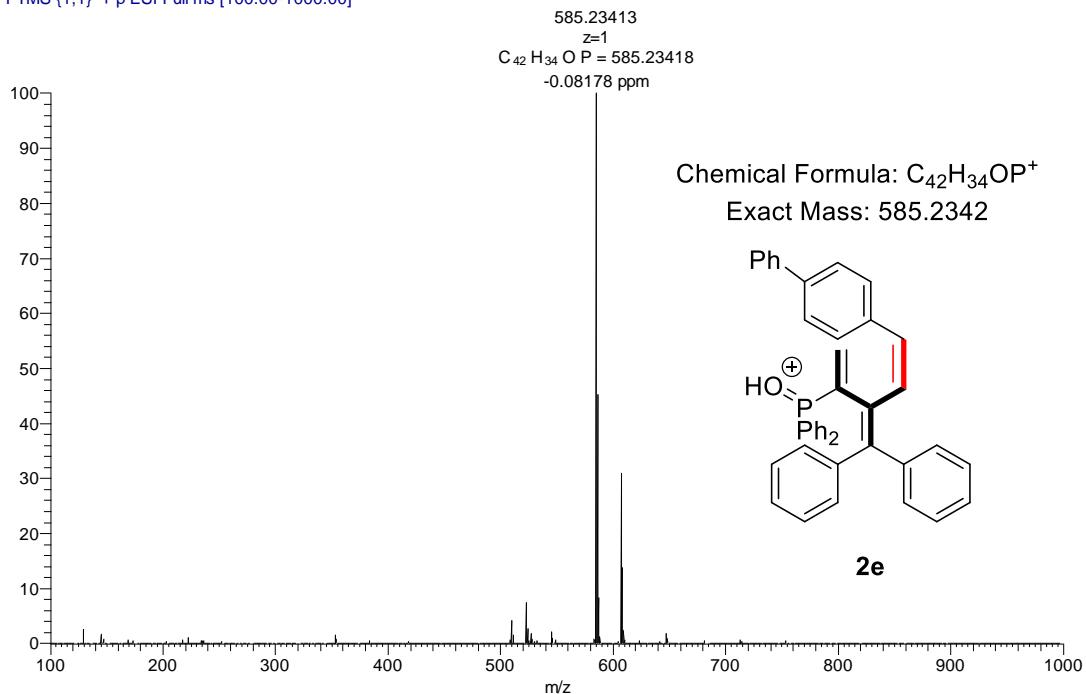
2e

20180904
xyt-223h

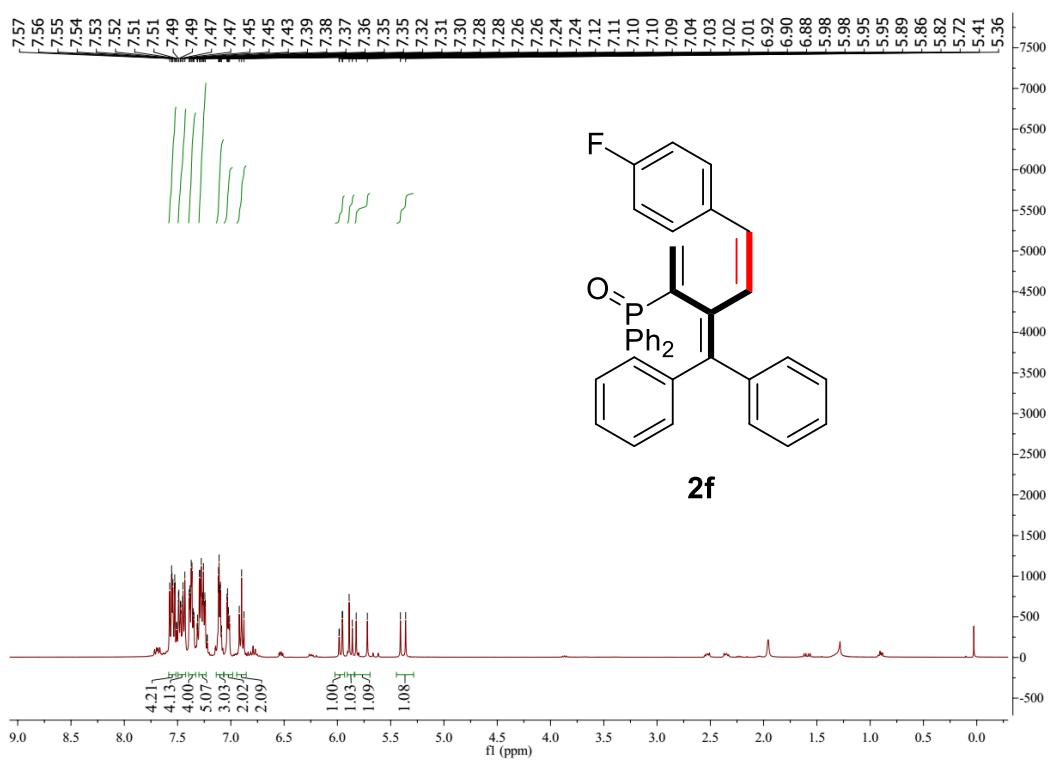


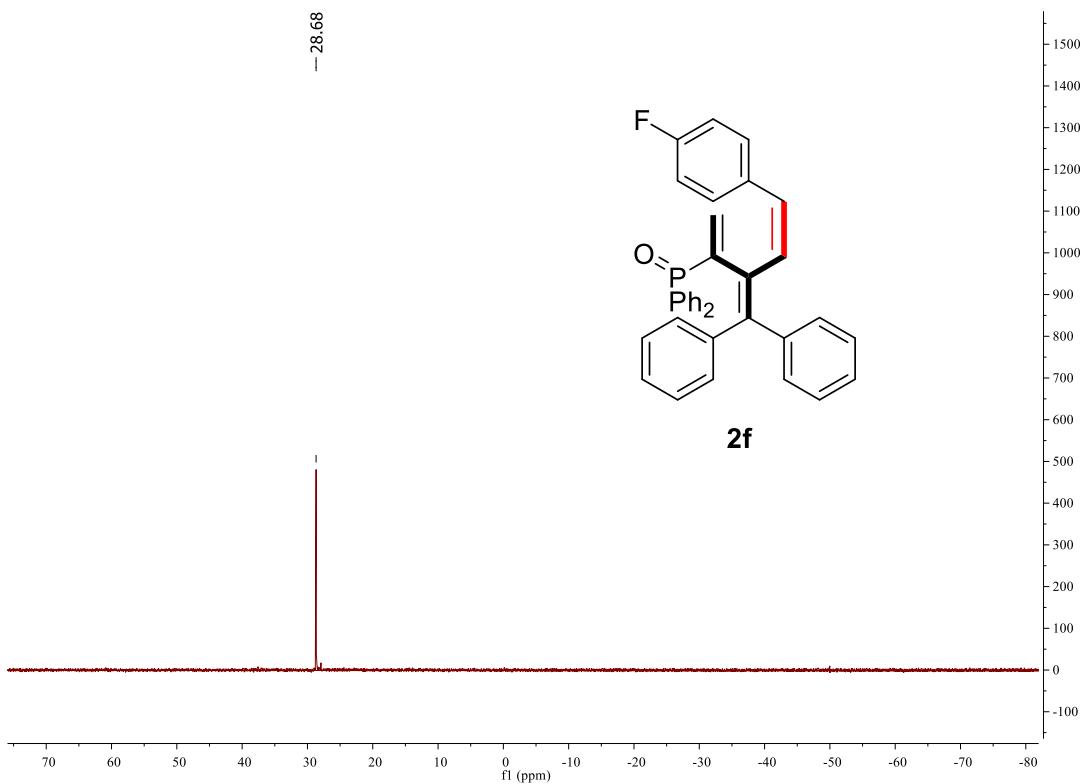
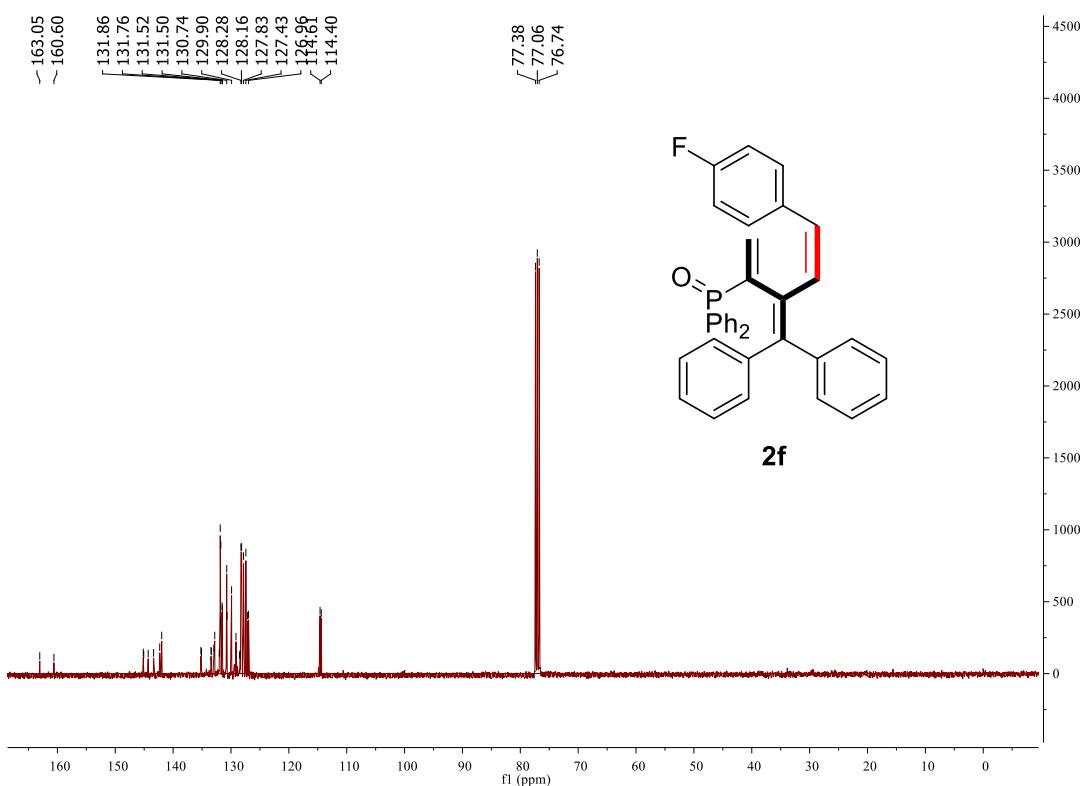


20181014-4 #31 RT: 0.46 AV: 1 NL: 1.81E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

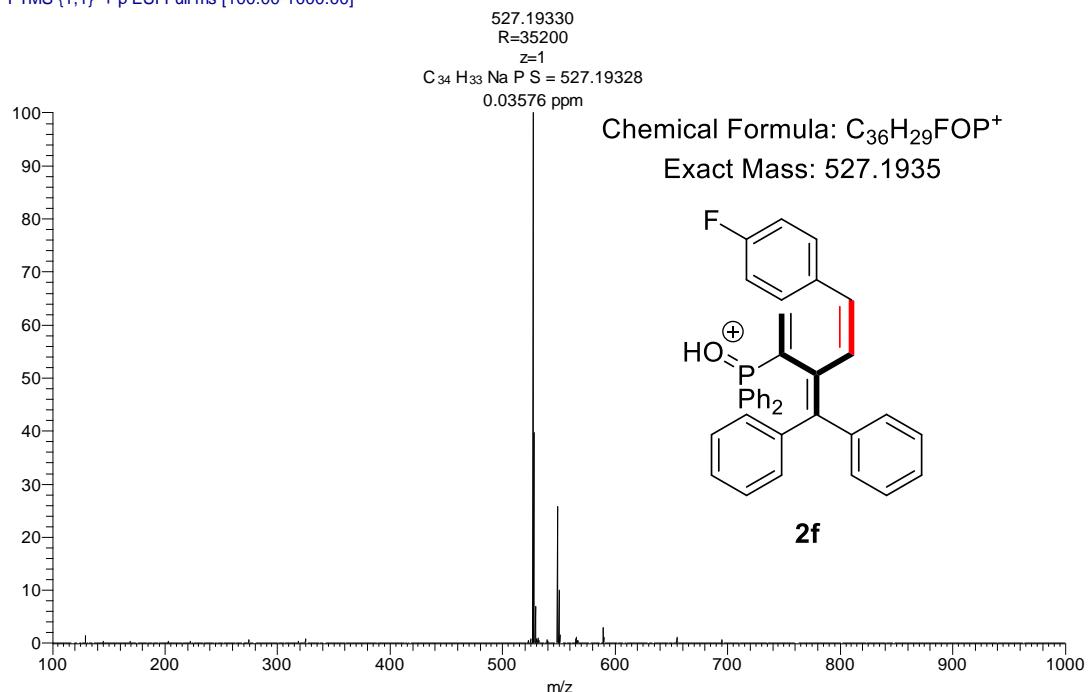


2f

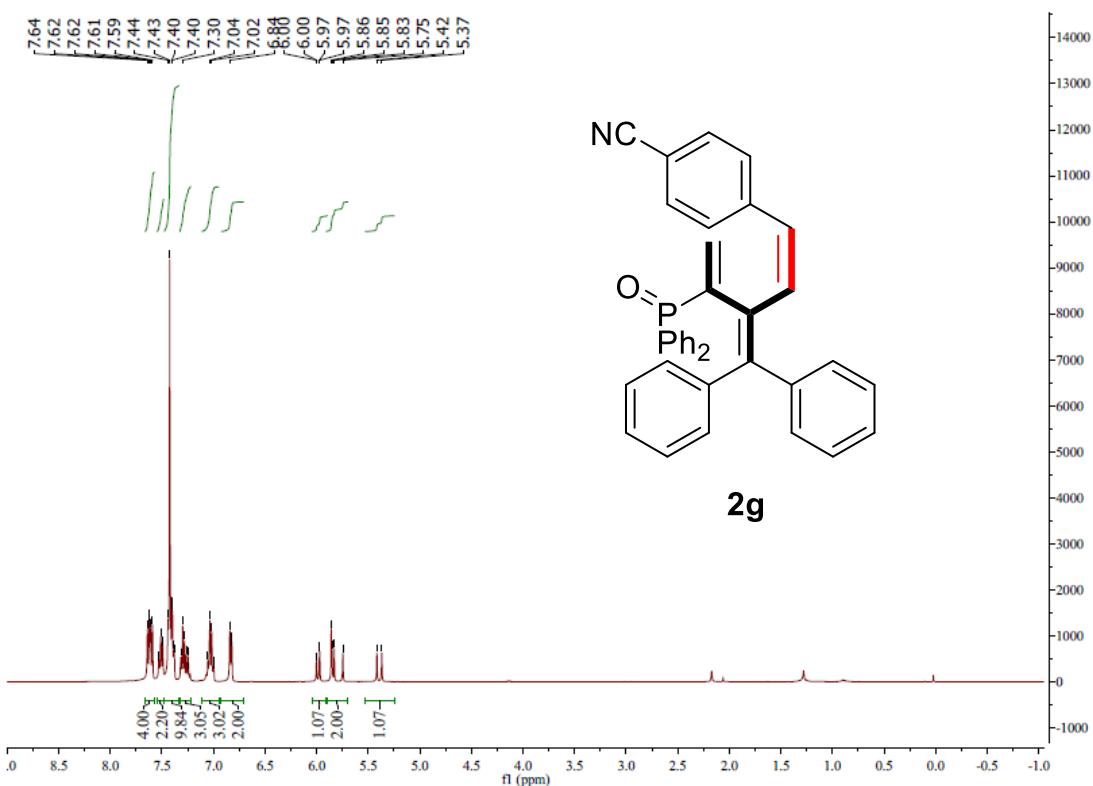


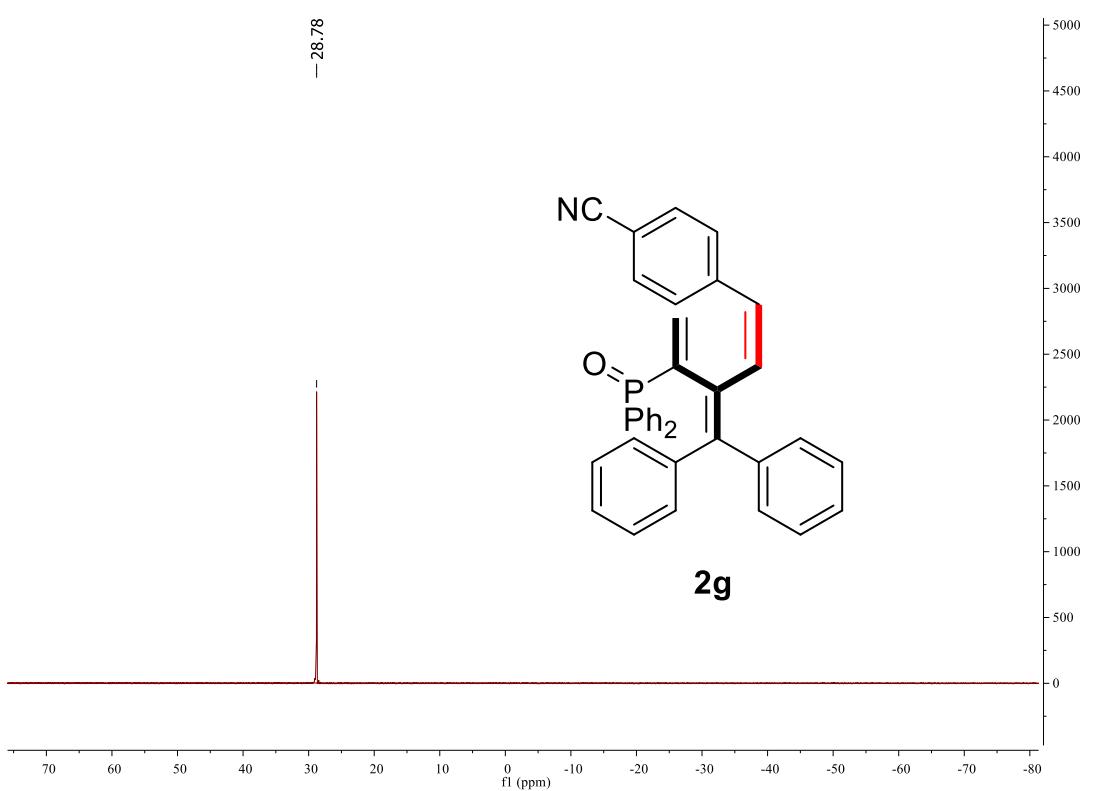
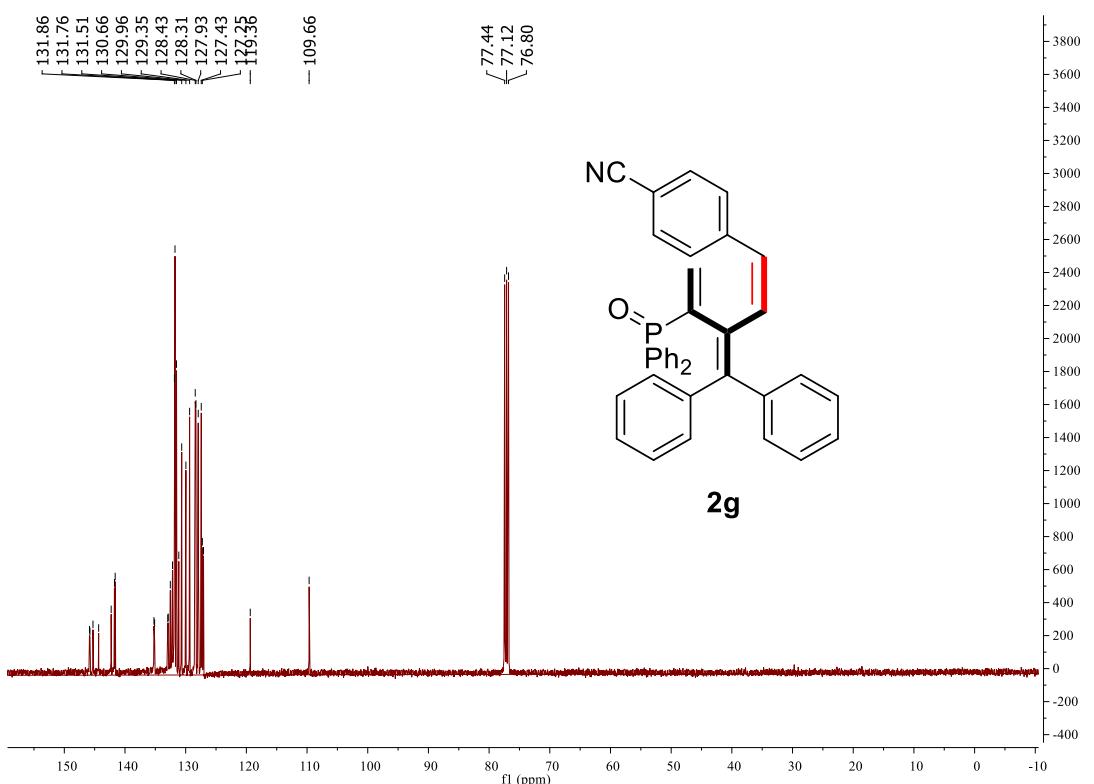


niny-20180903-11 #31 RT: 0.47 AV: 1 NL: 7.92E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

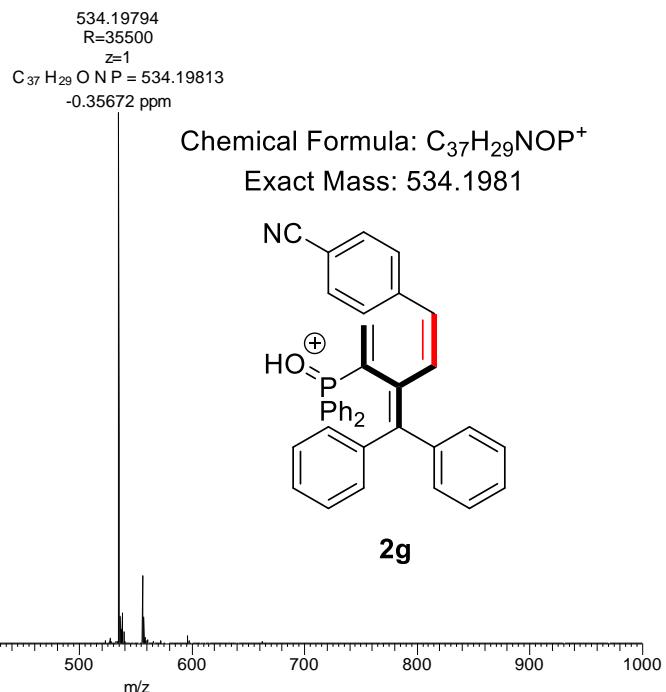


2g

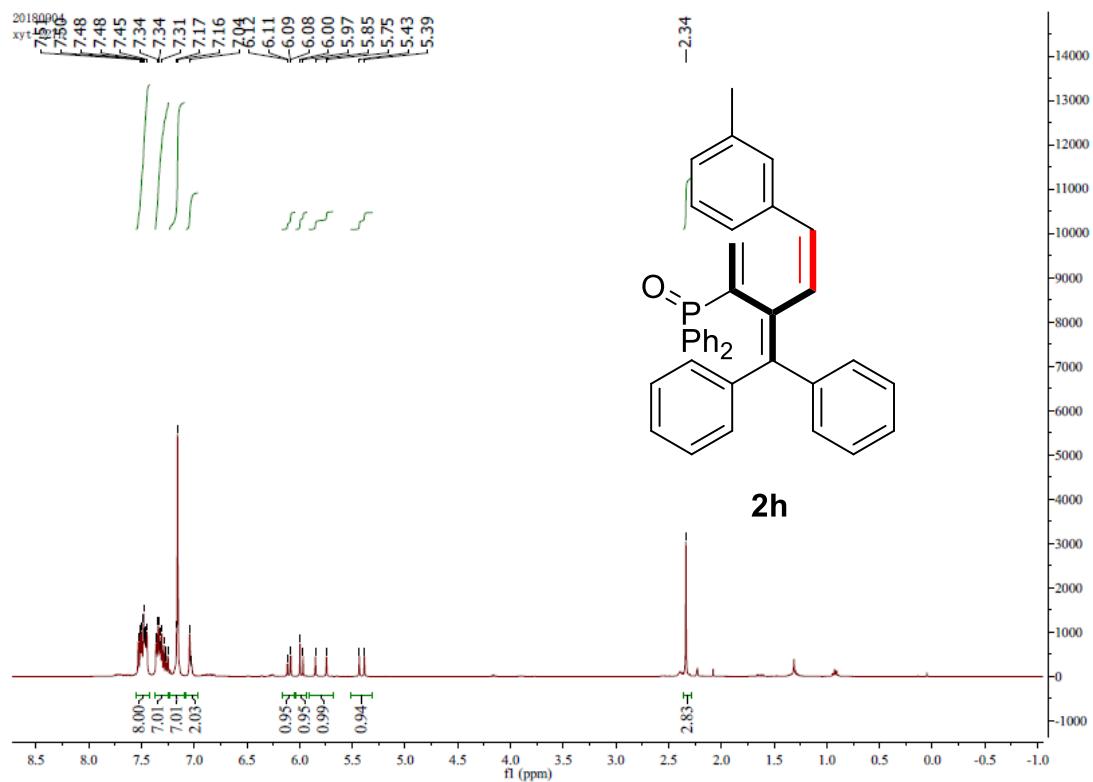


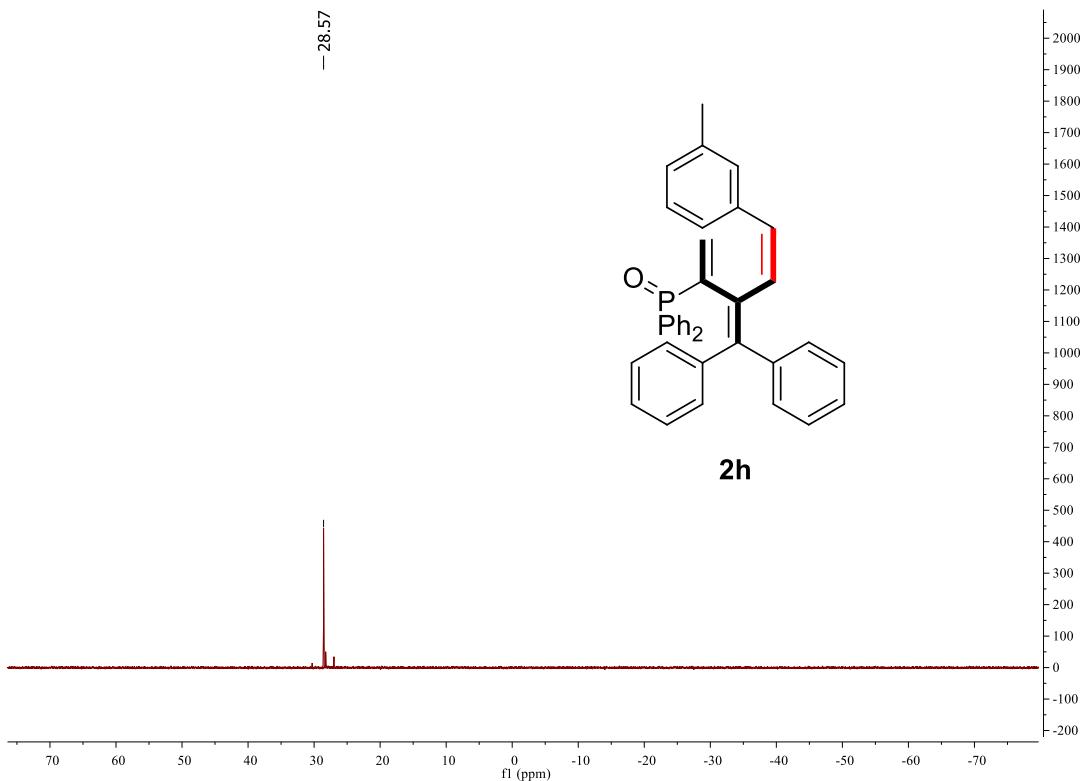
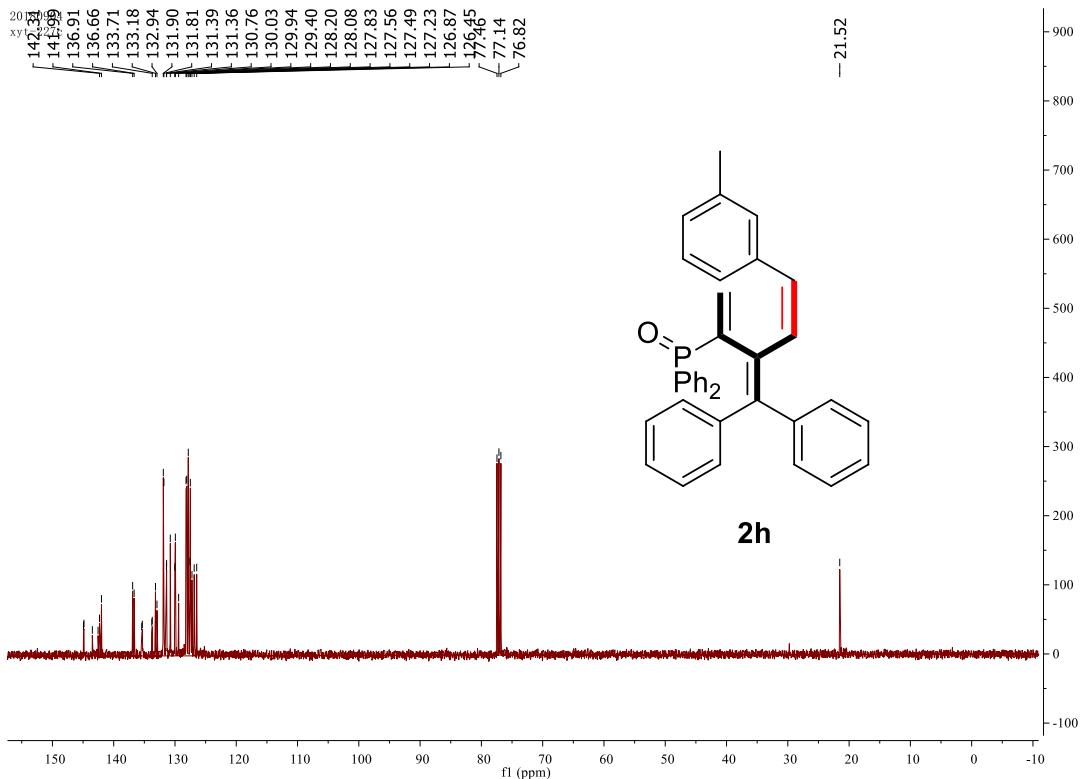


njny-20180903-12 #33 RT: 0.50 AV: 1 NL: 1.99E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

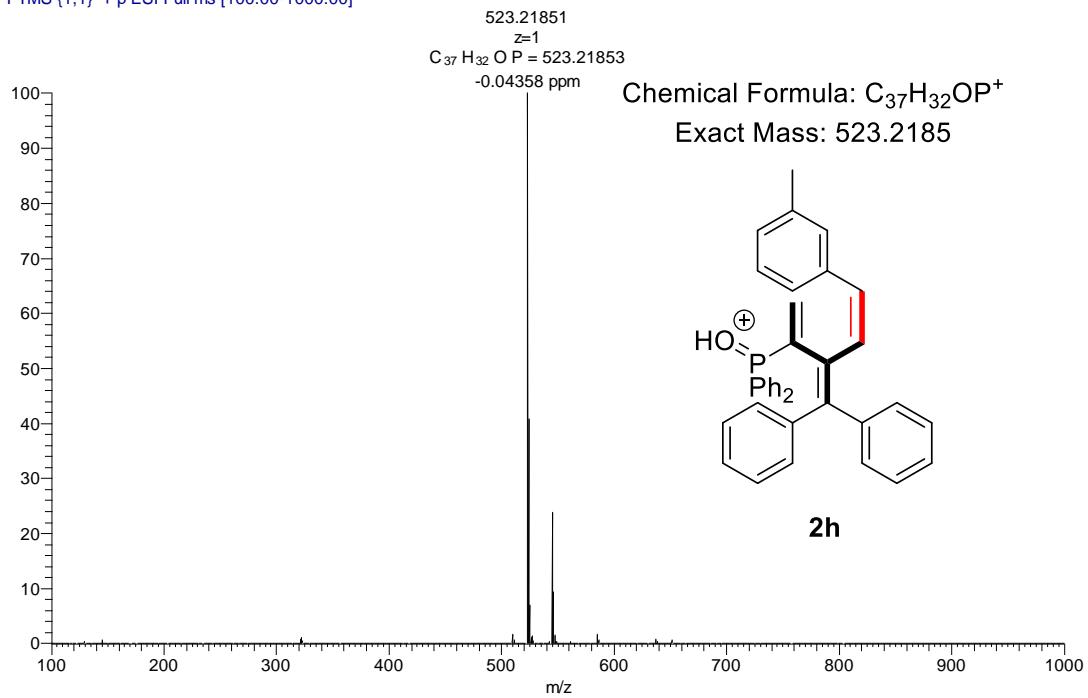


2h

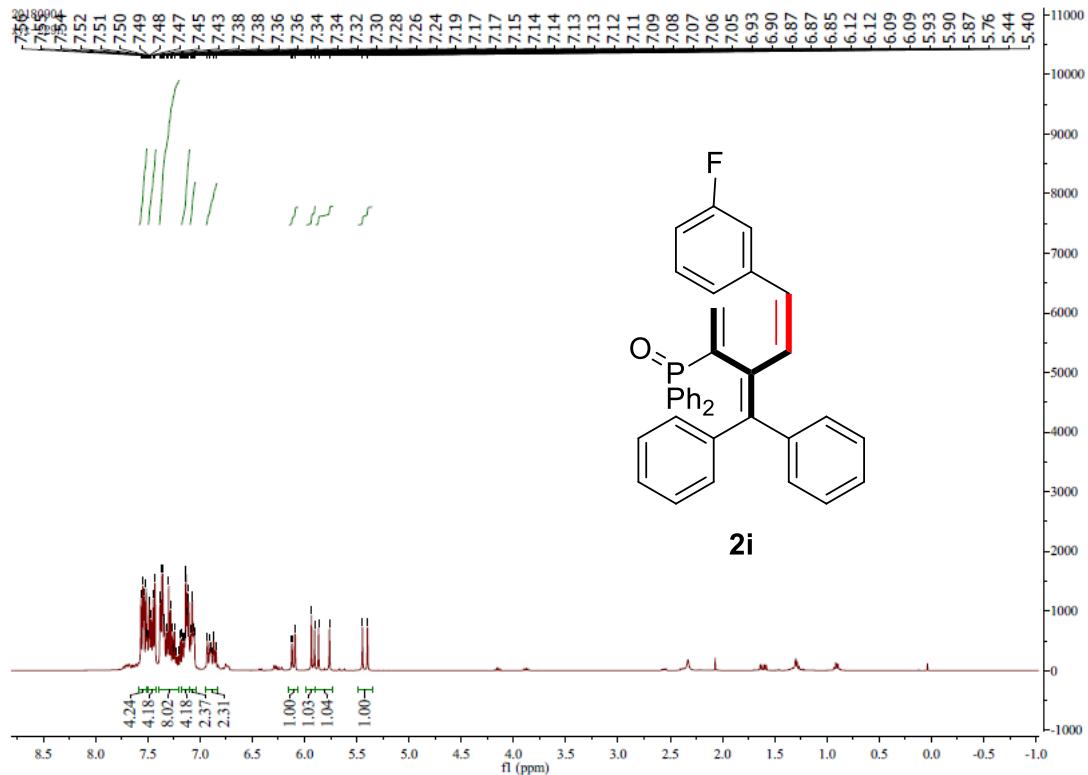


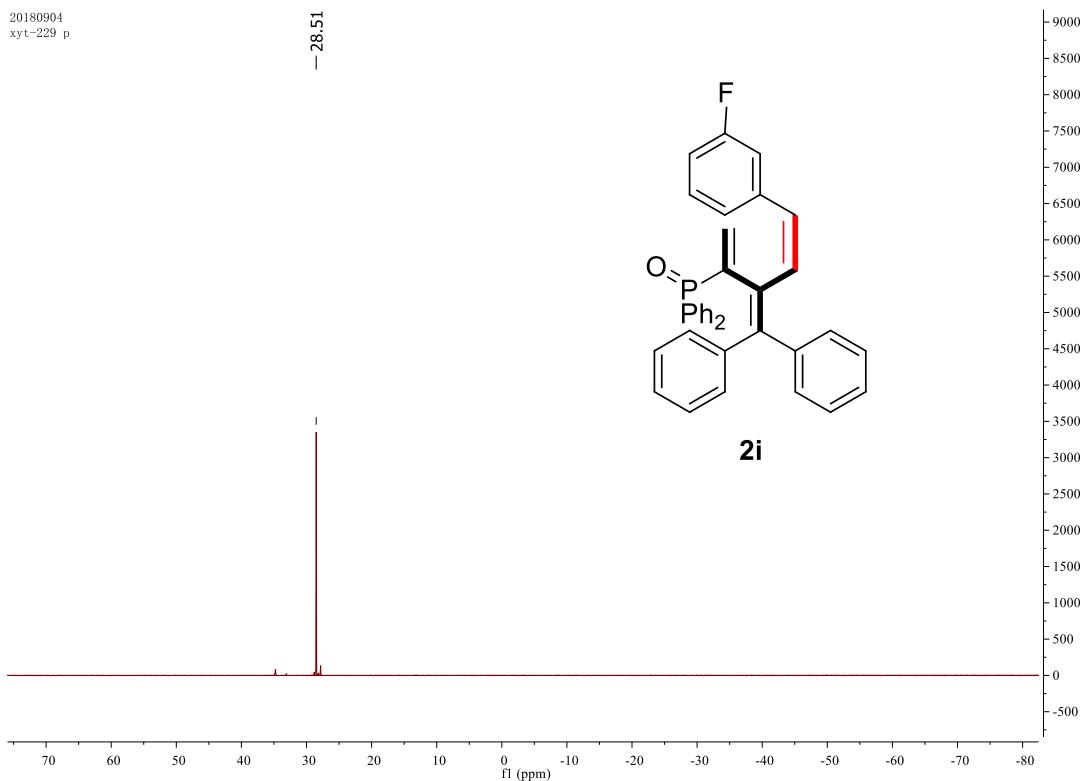
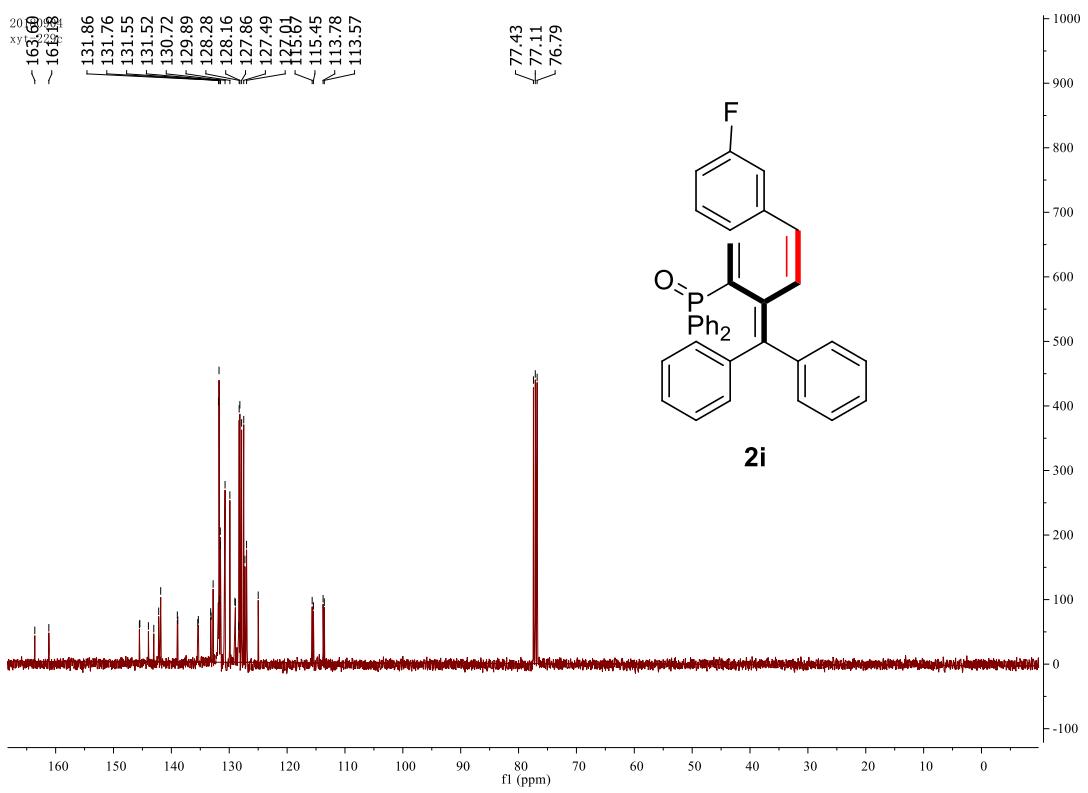


20181014-3 #53 RT: 0.77 AV: 1 NL: 5.13E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

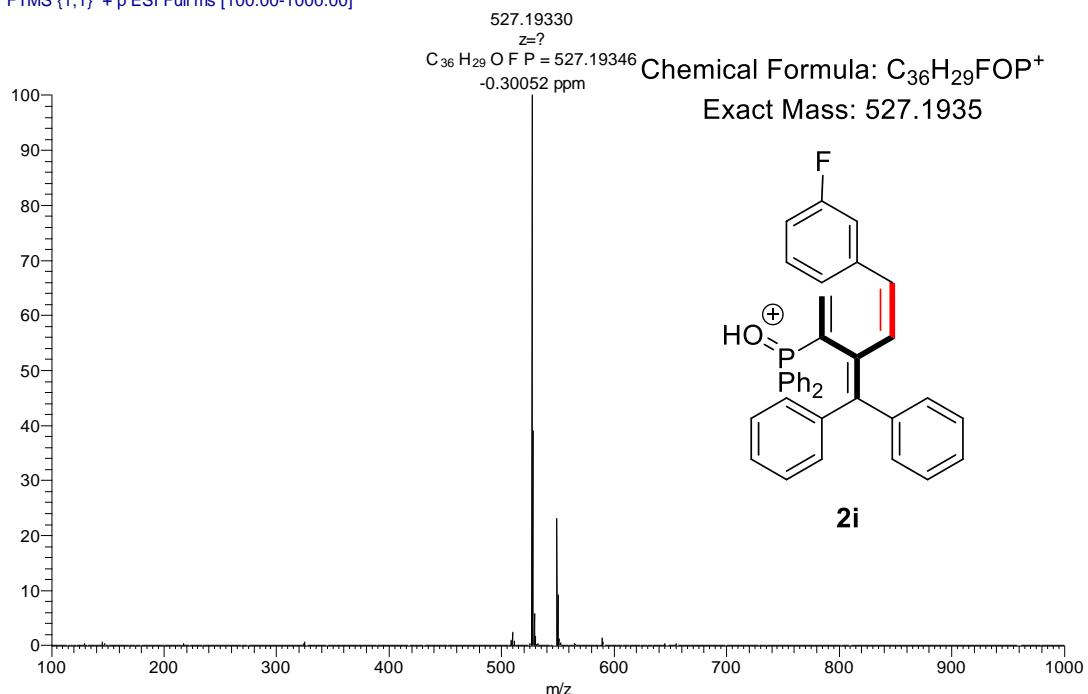


2i

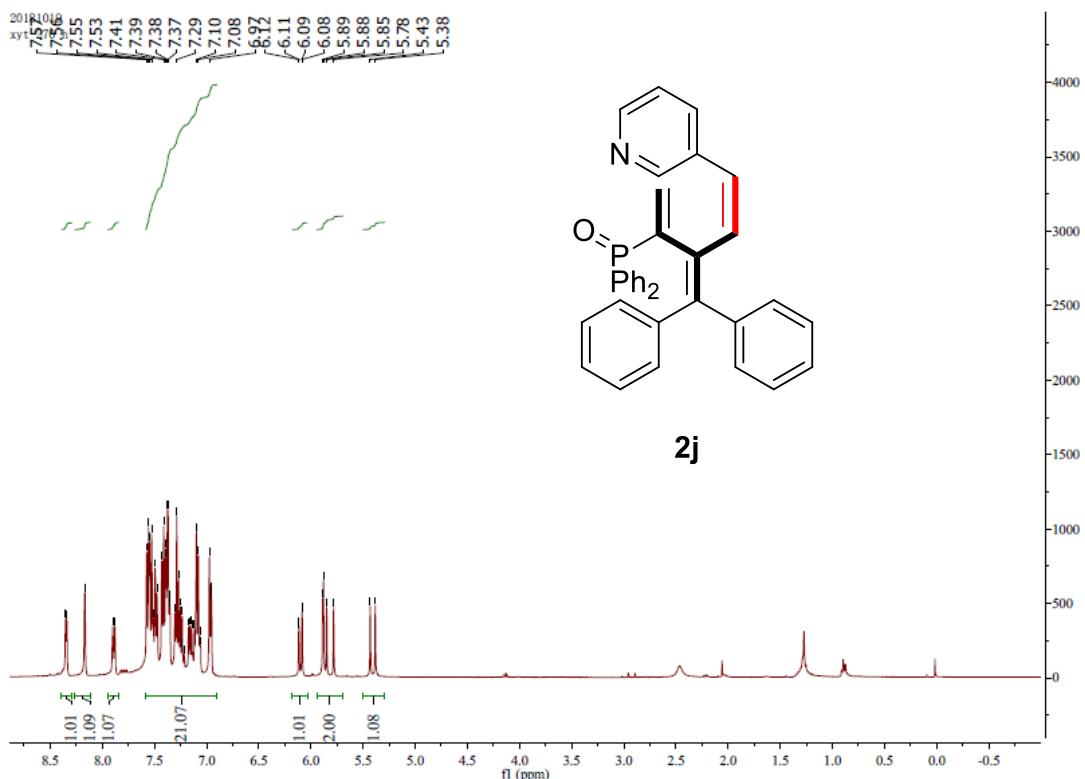


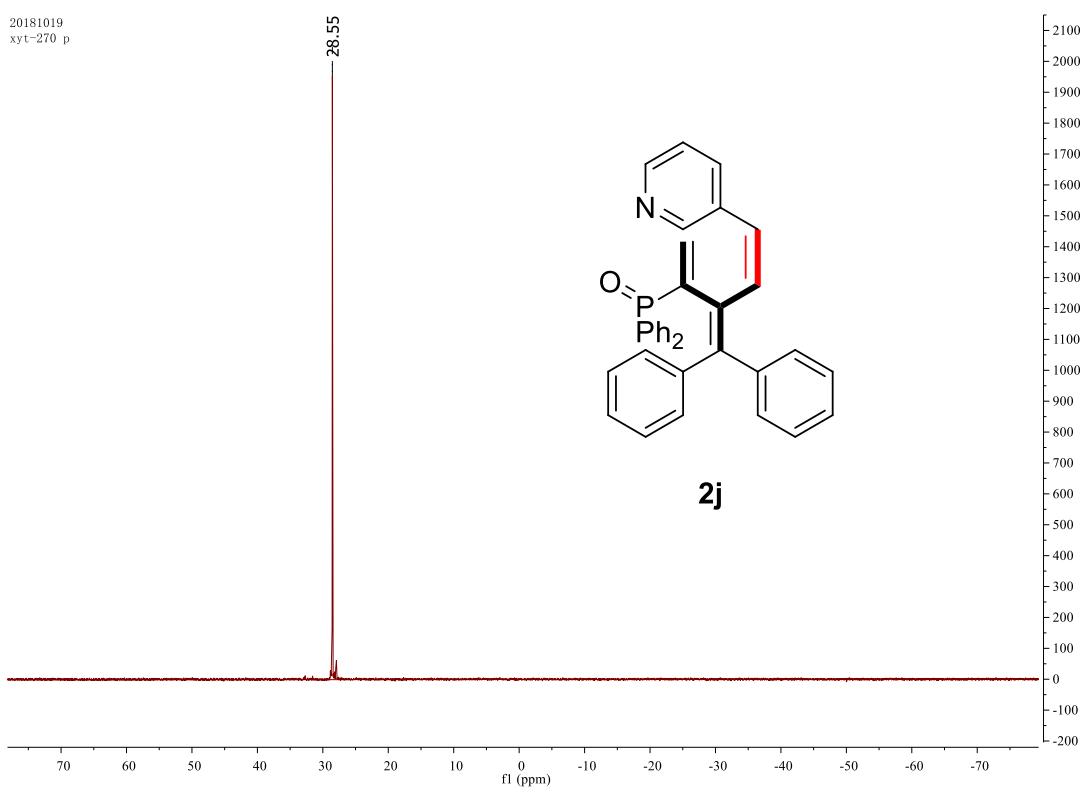
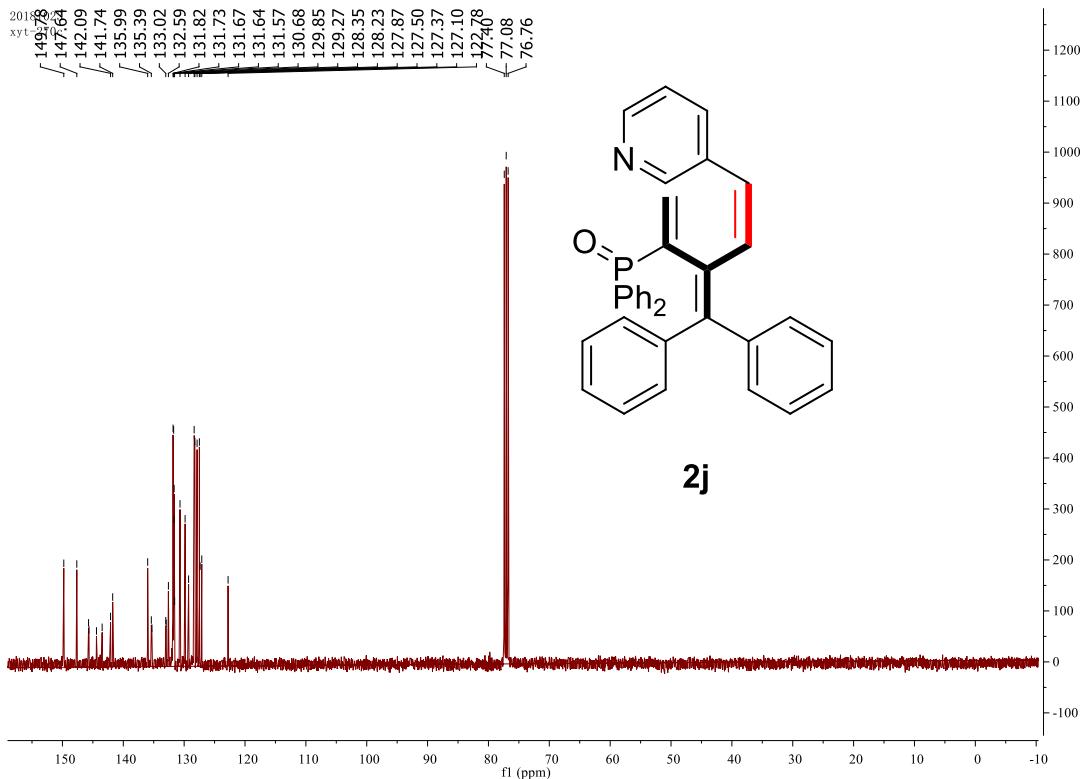


20181014-2 #53 RT: 0.78 AV: 1 NL: 4.30E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



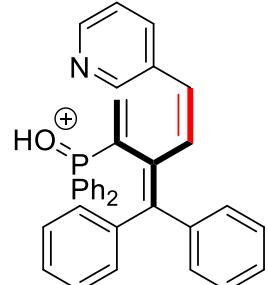
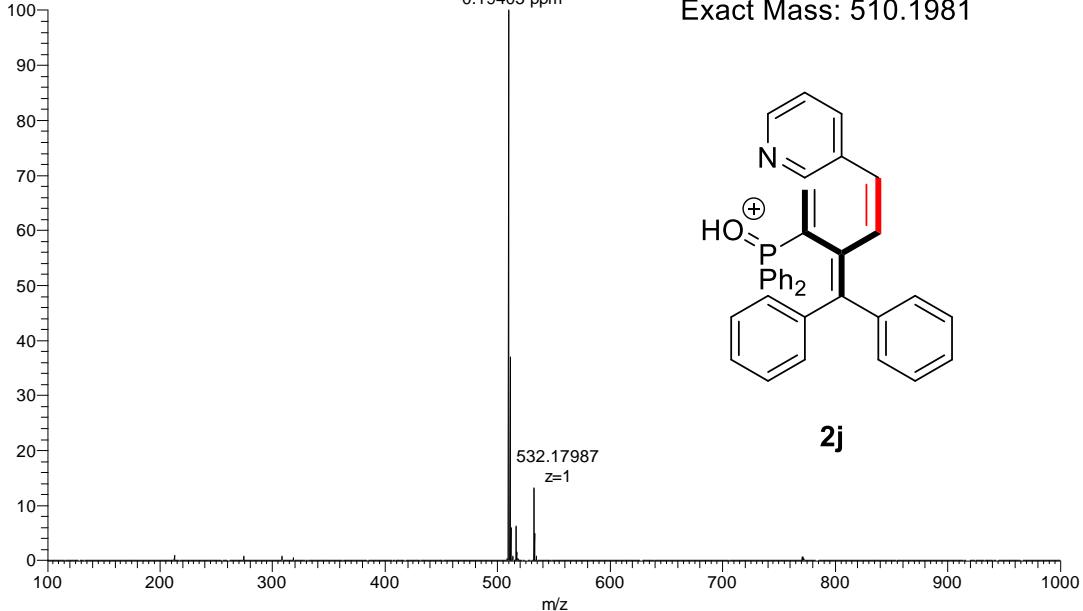
2j





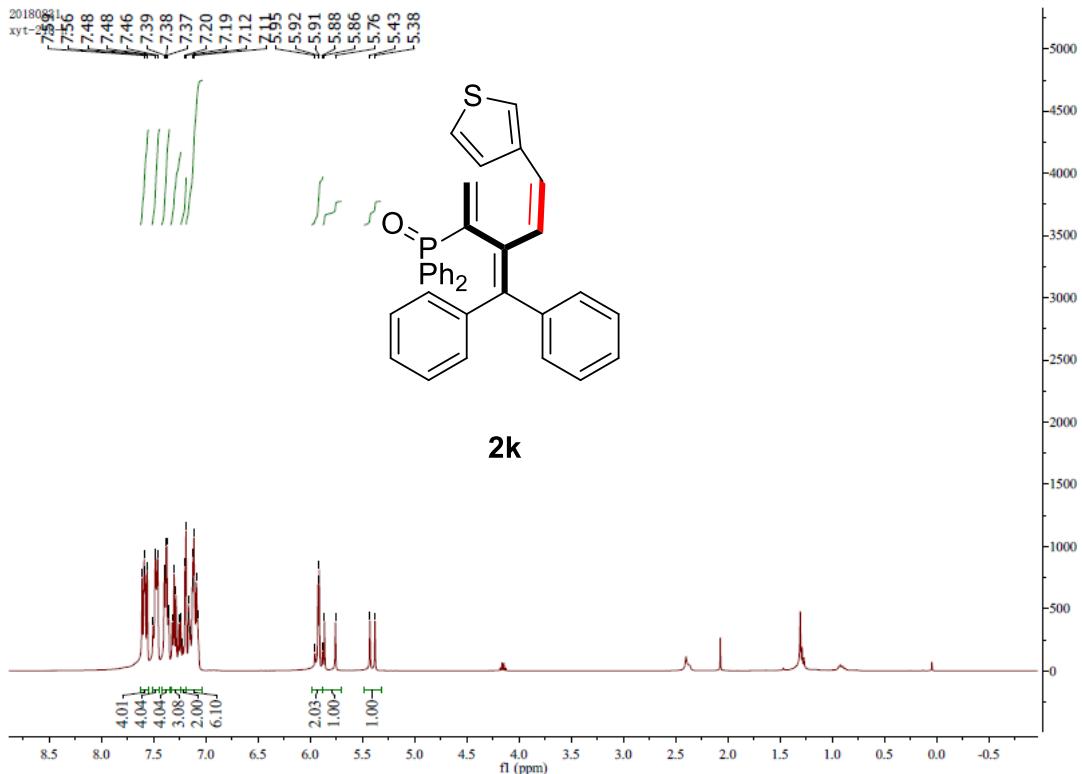
20181014-1 #29 RT: 0.42 AV: 1 NL: 6.09E7
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

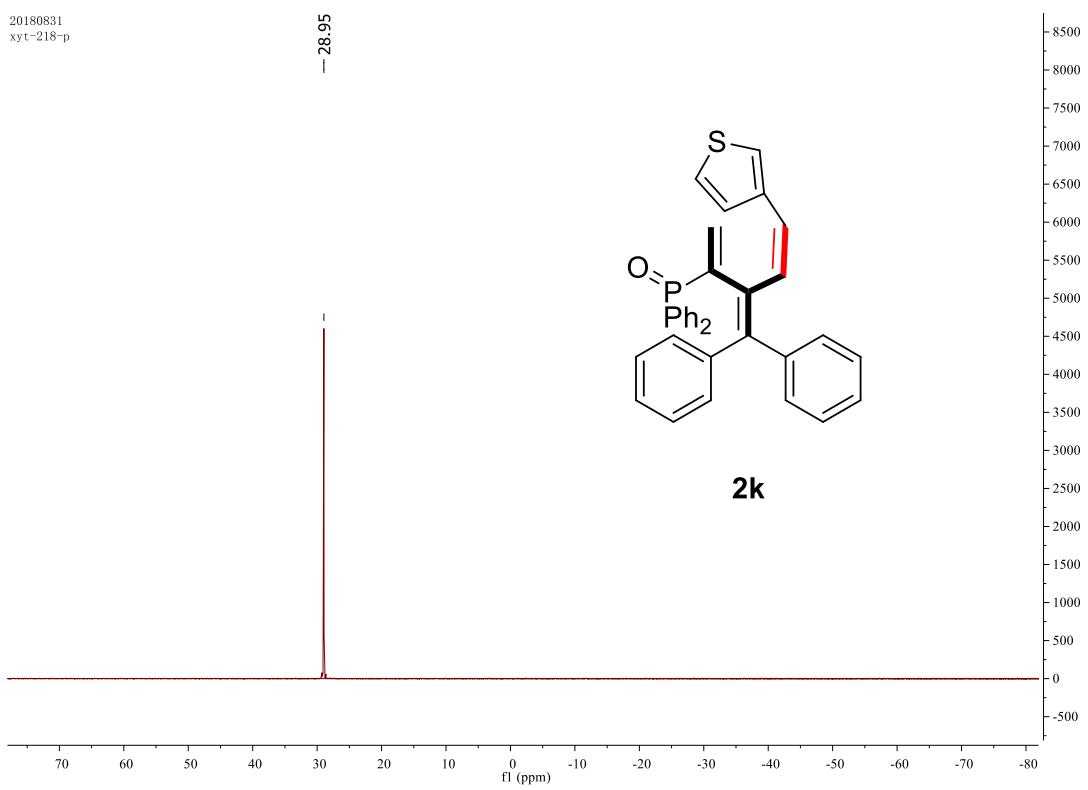
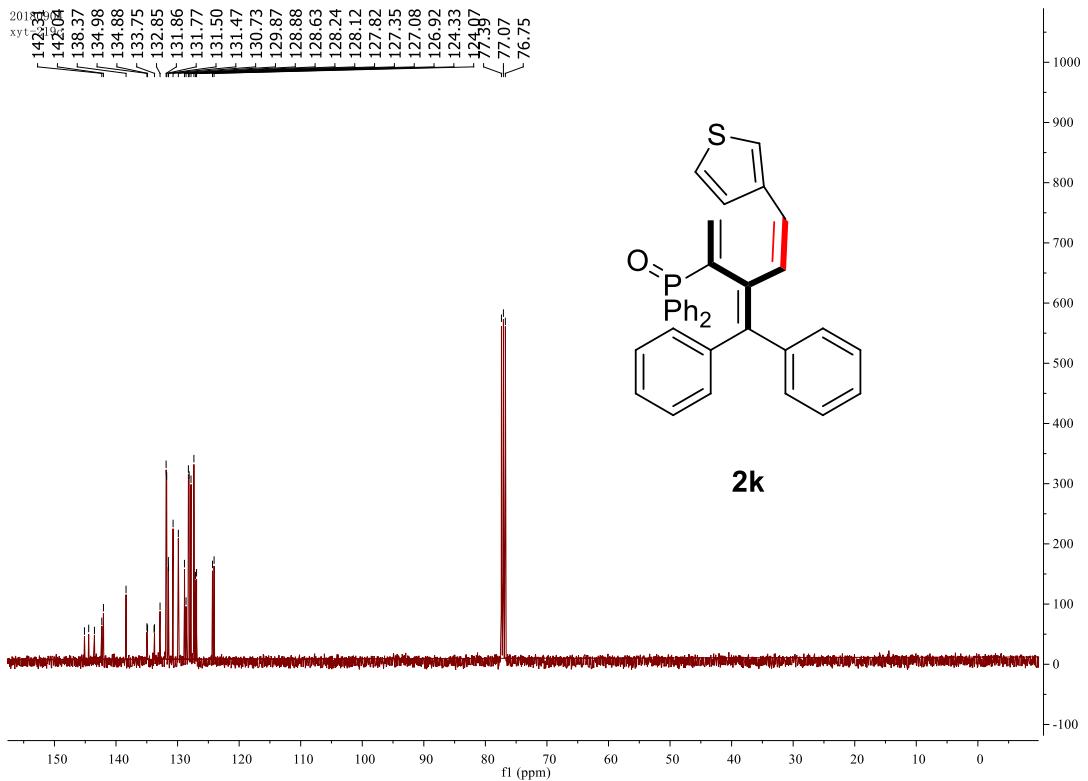
510.19803
 $z=1$
 $C_{35}H_{29}ONP = 510.19813$
Chemical Formula: $C_{35}H_{29}NOP^+$
Exact Mass: 510.1981



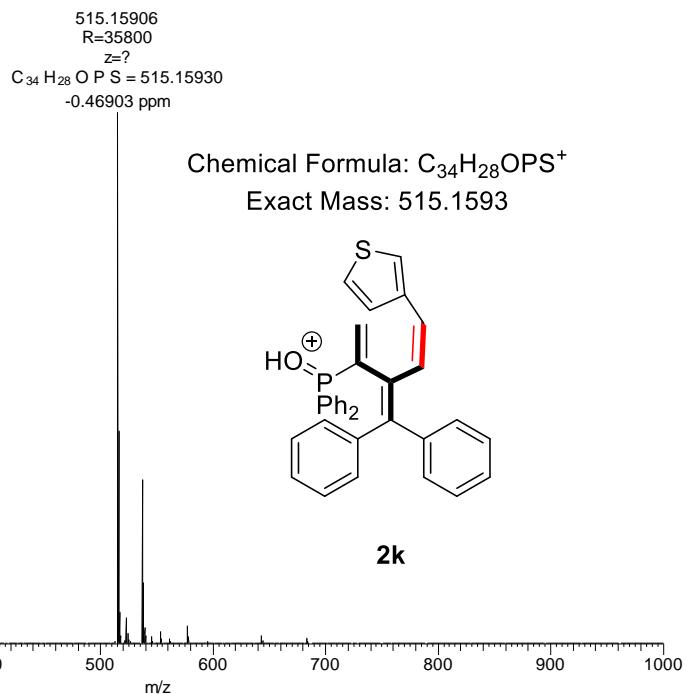
2j

2k

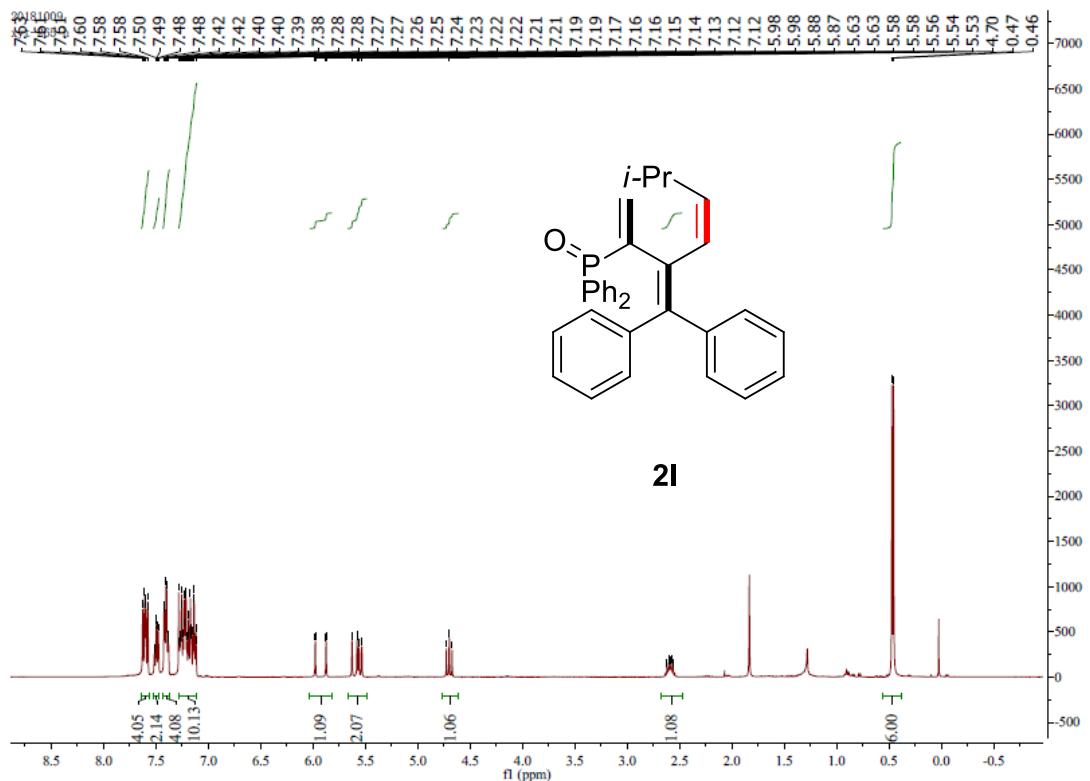


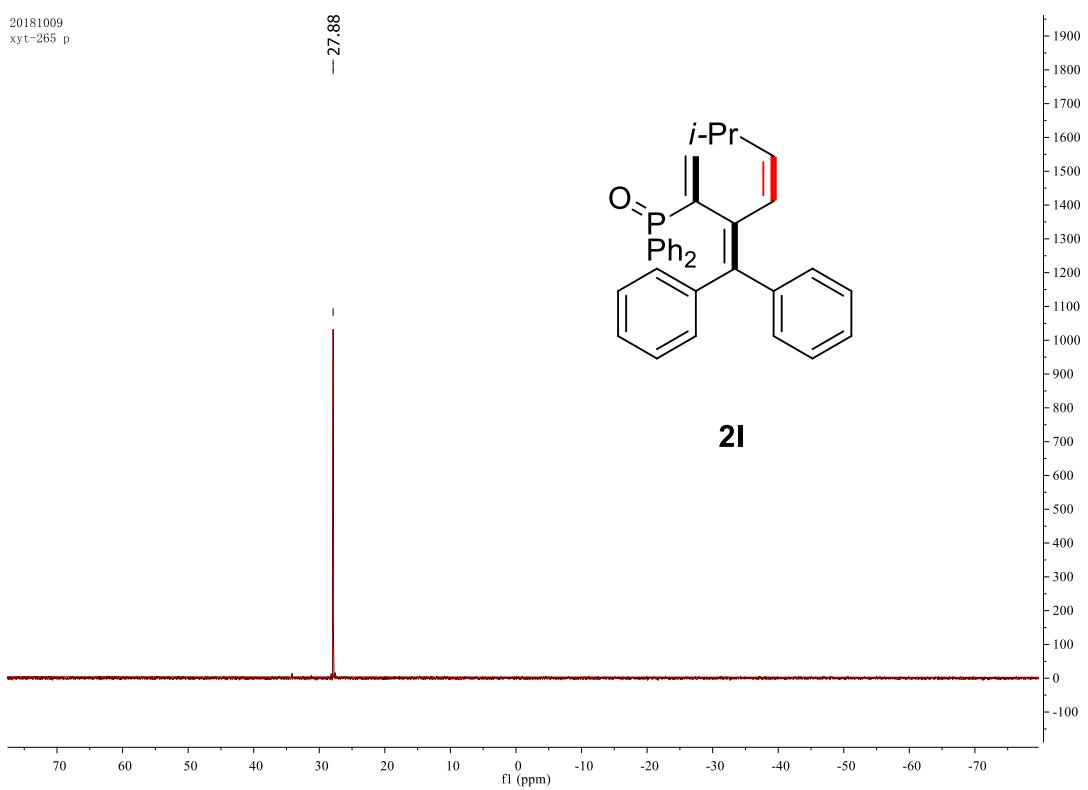
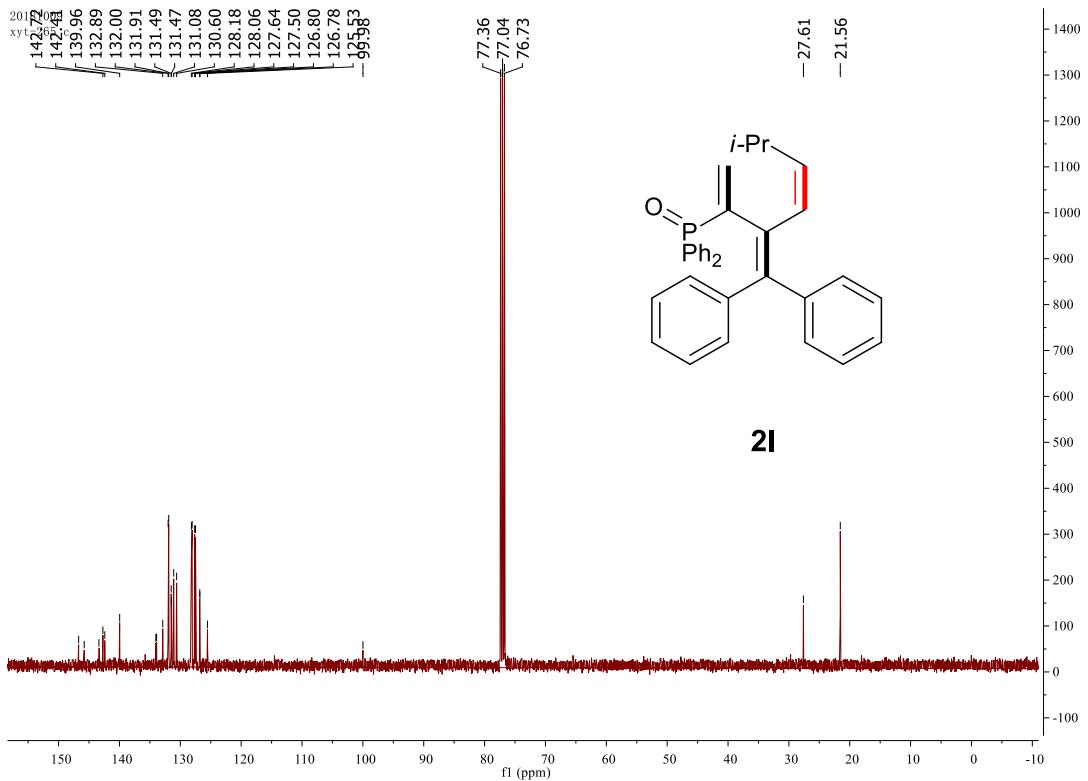


njny-20180903-9 #39 RT: 0.60 AV: 1 NL: 4.39E5
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

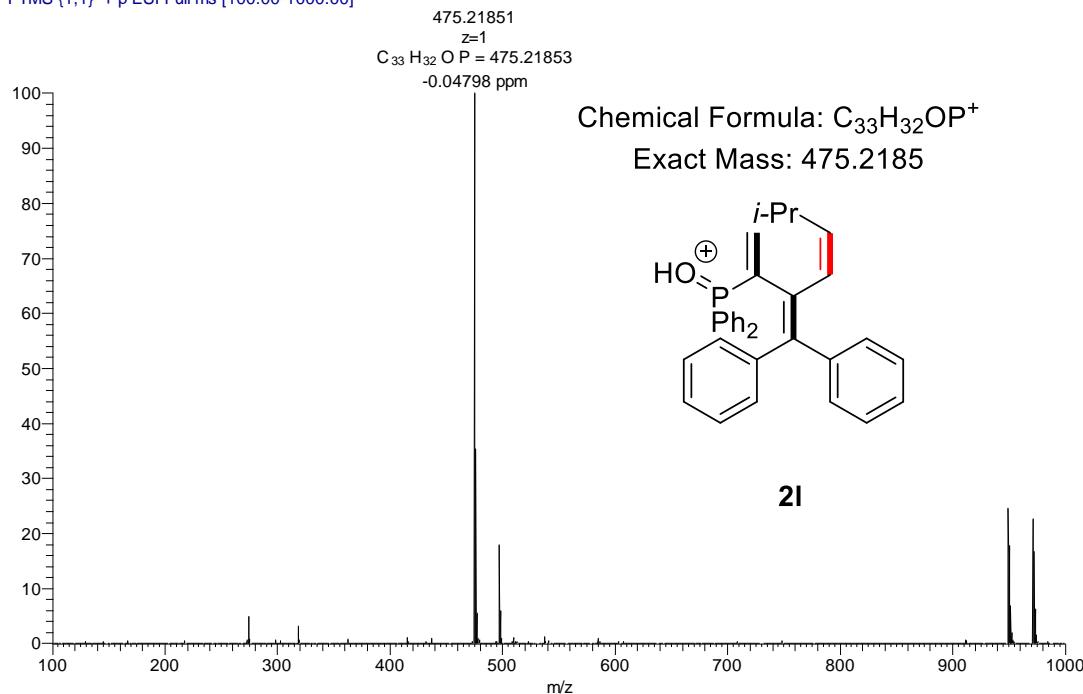


2l

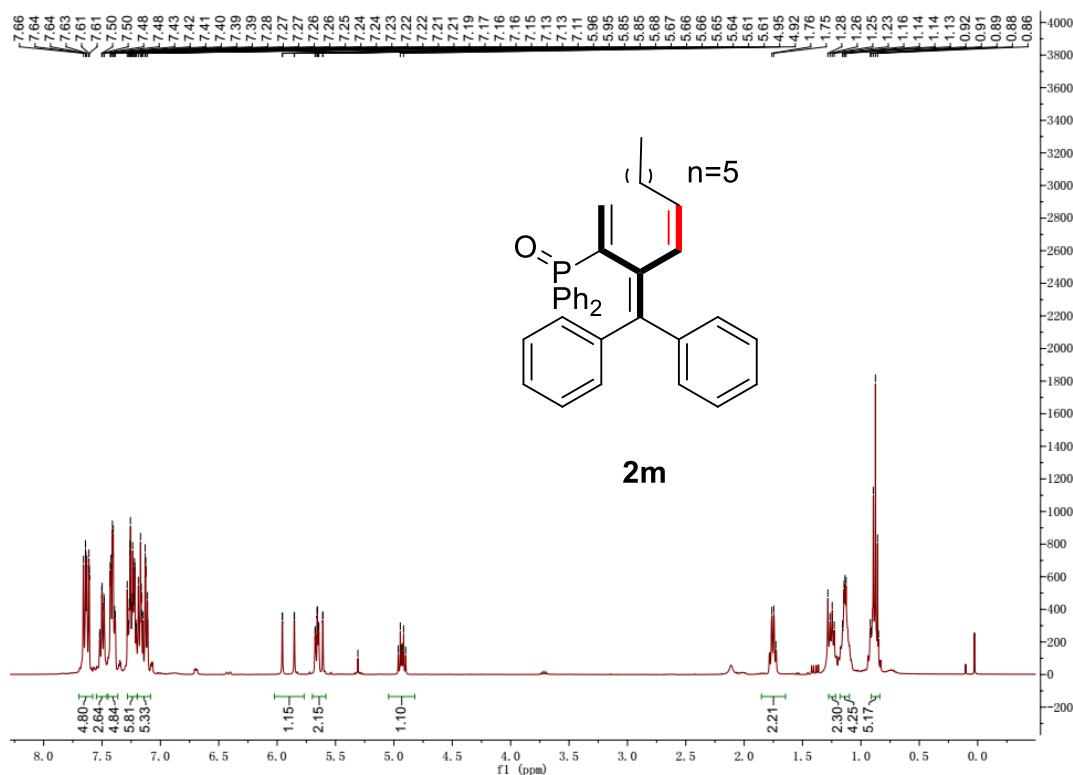


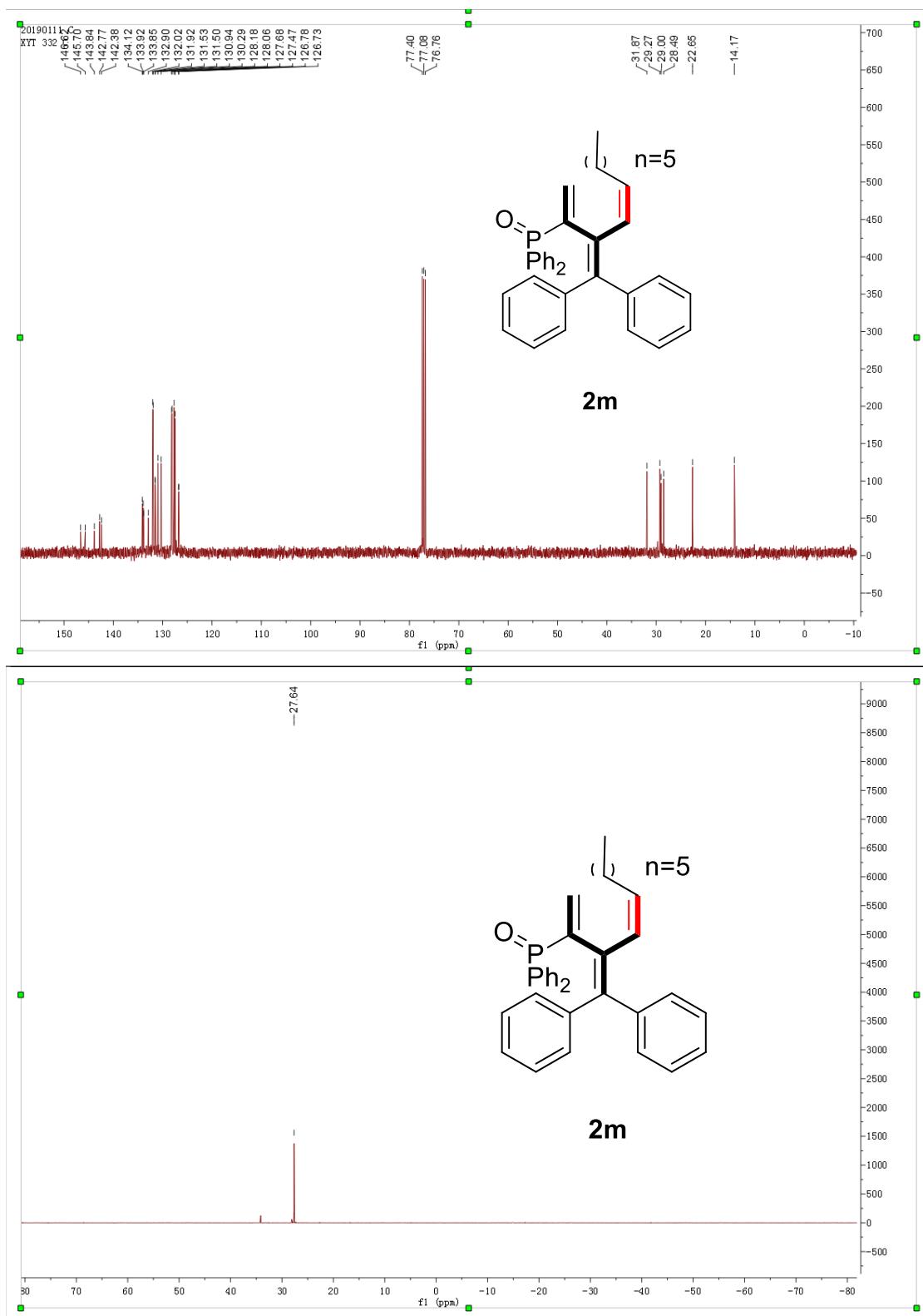


20181014-5 #35 RT: 0.51 AV: 1 NL: 5.92E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

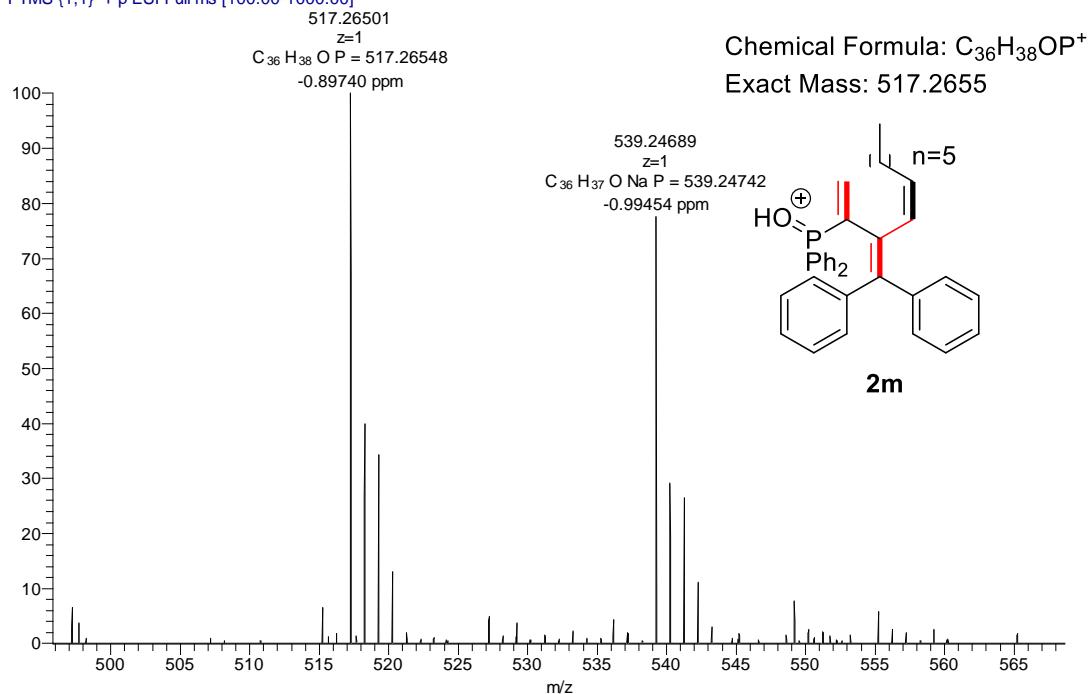


2m

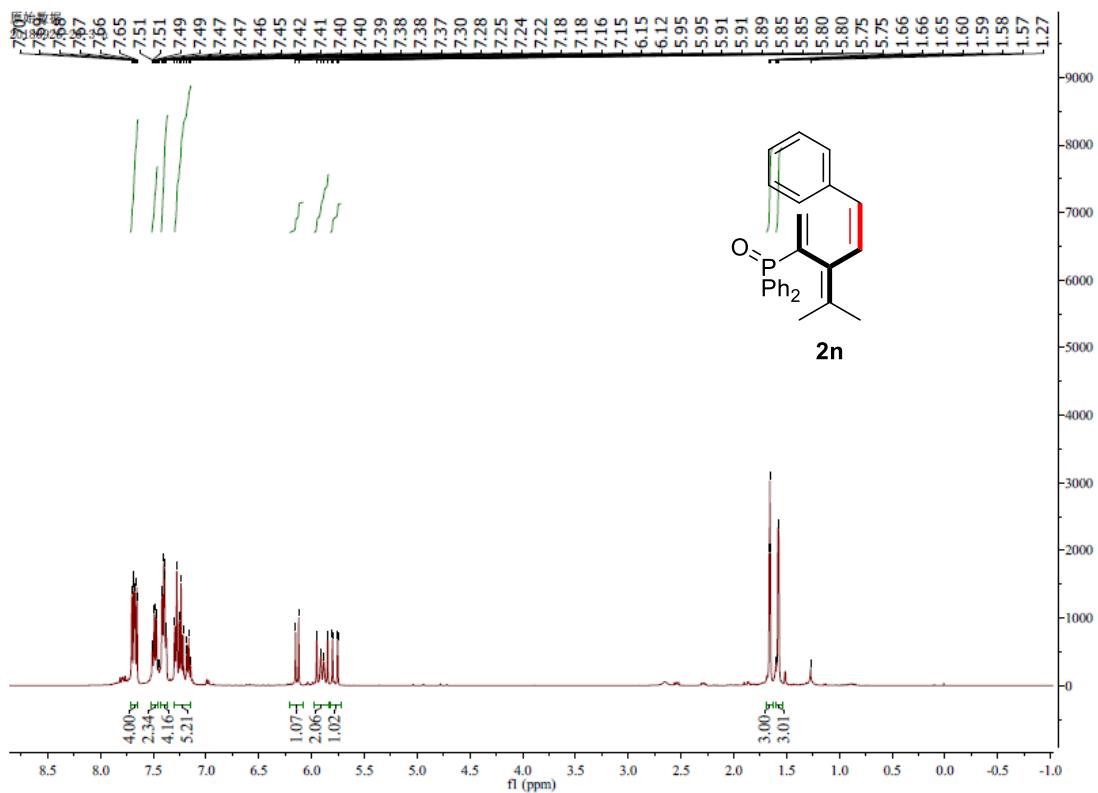


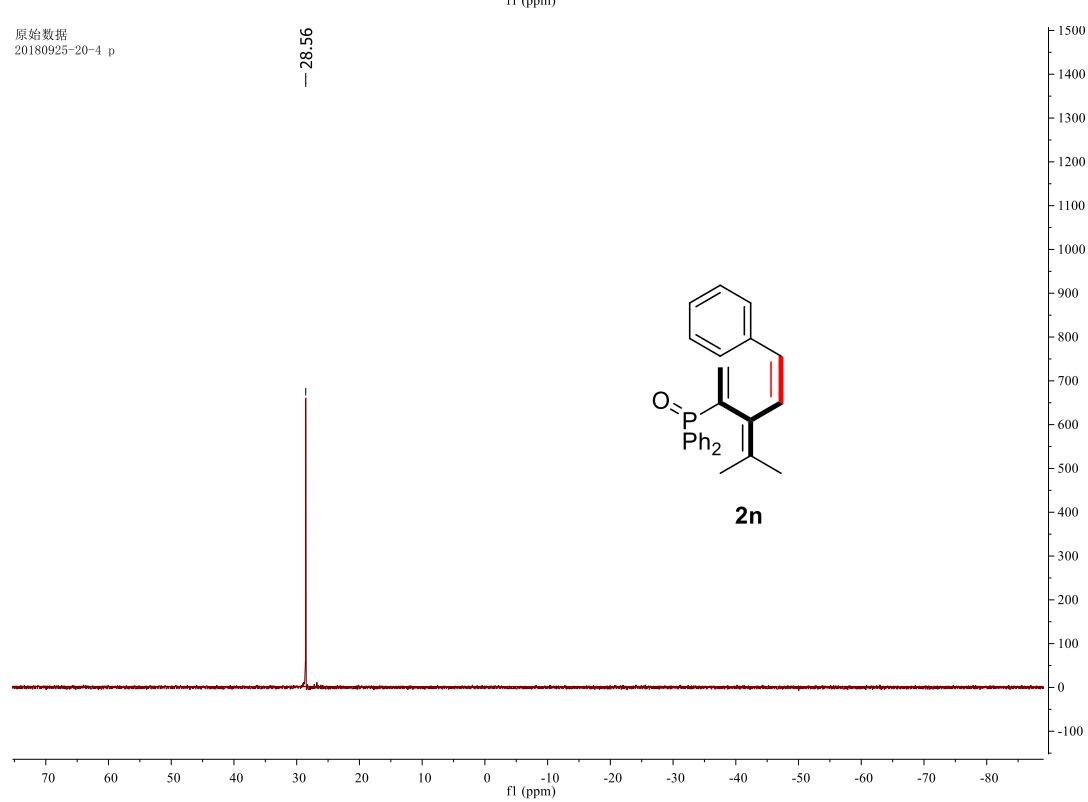
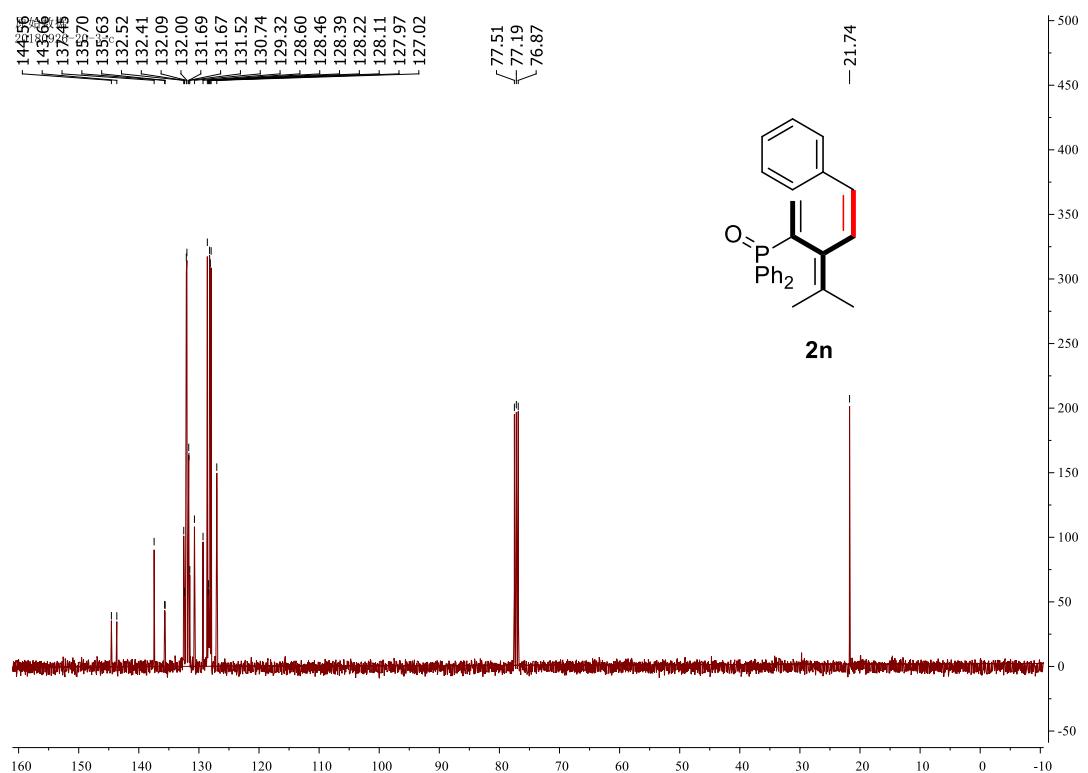


20190605-9 #25 RT: 0.39 AV: 1 NL: 2.17E4
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



2n

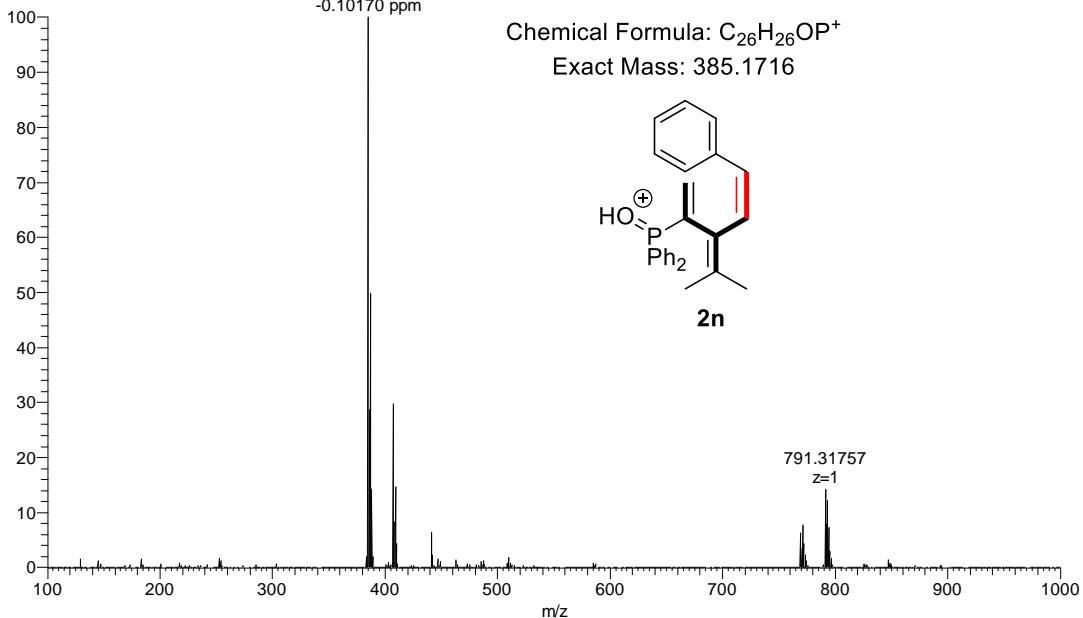
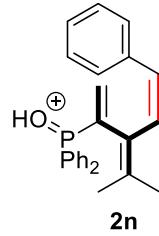




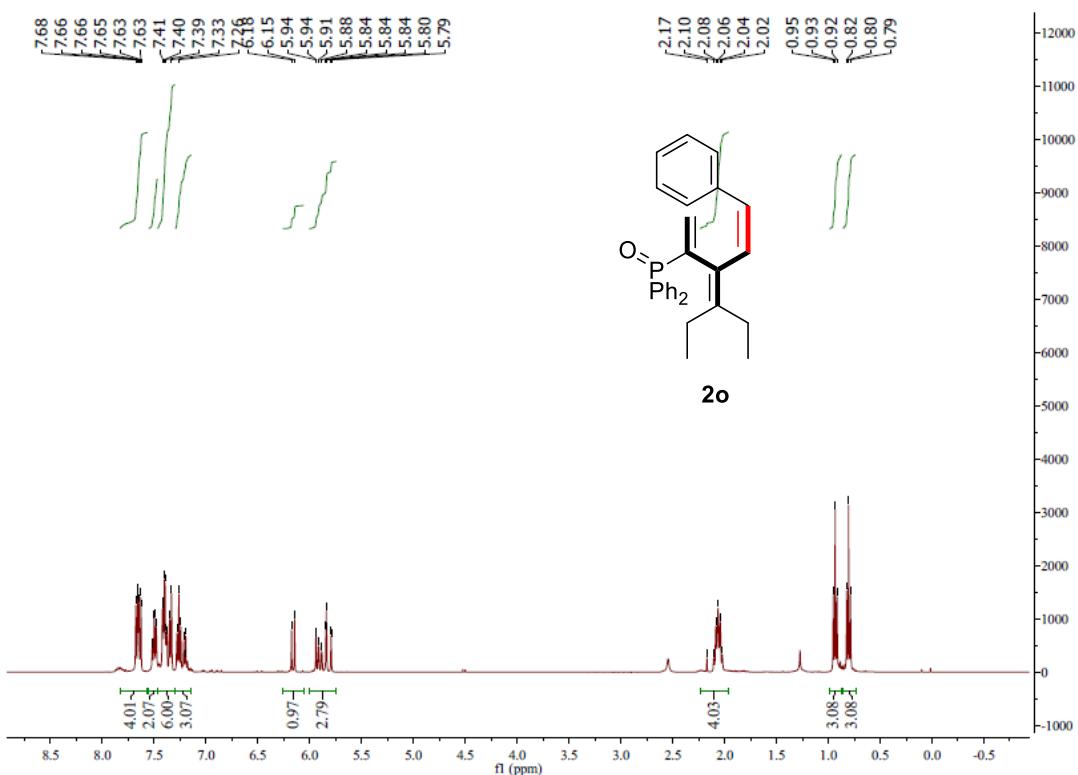
20181014-10 #37 RT: 0.55 AV: 1 NL: 2.15E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

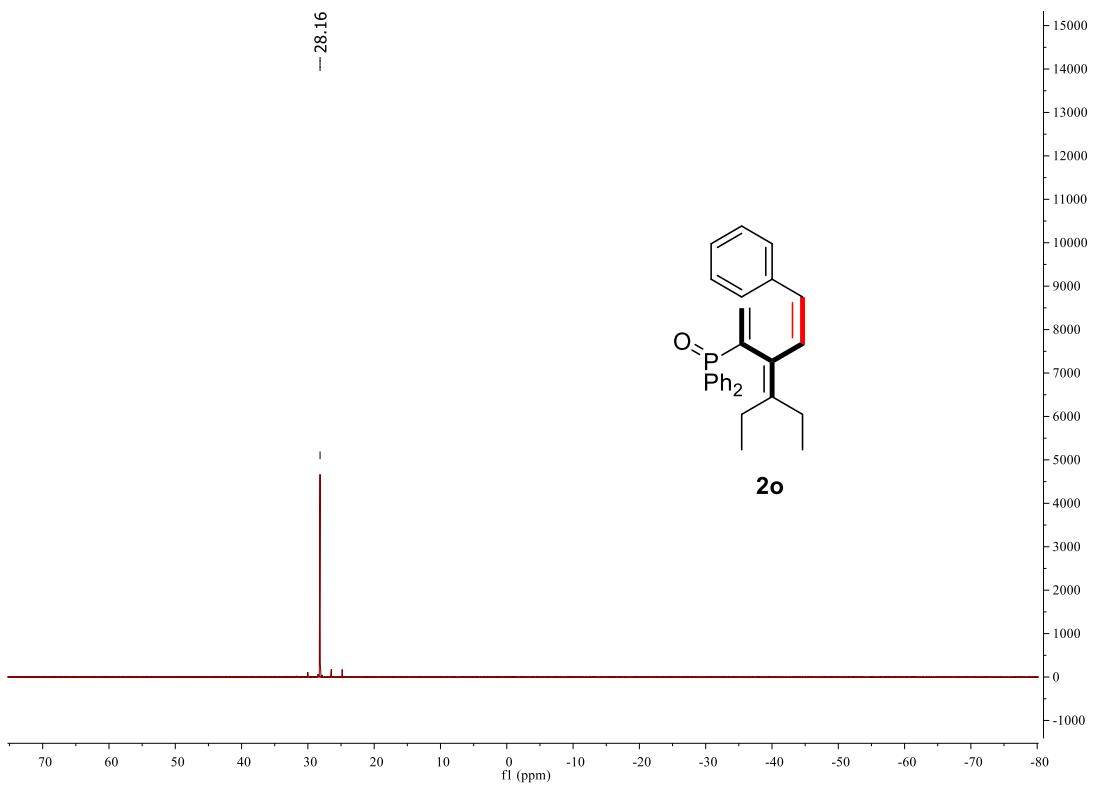
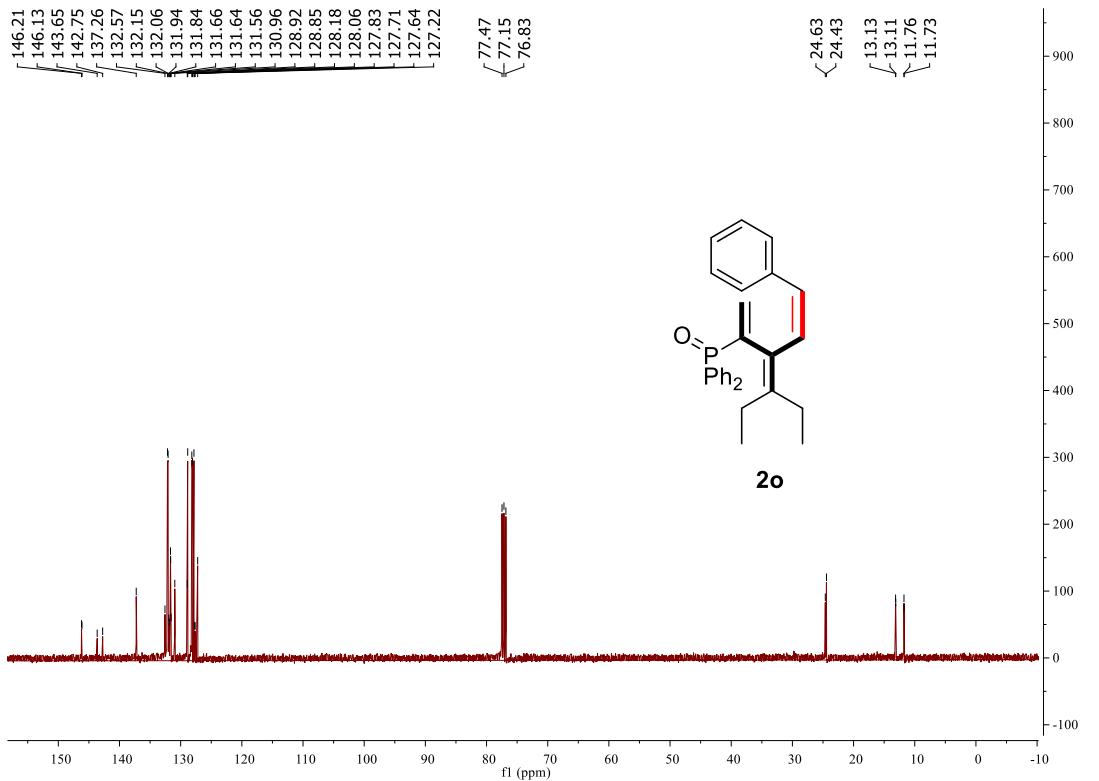
385.17154
 $z=?$
 $C_{26}H_{26}O_P = 385.17158$
-0.10170 ppm

Chemical Formula: $C_{26}H_{26}OP^+$
Exact Mass: 385.1716

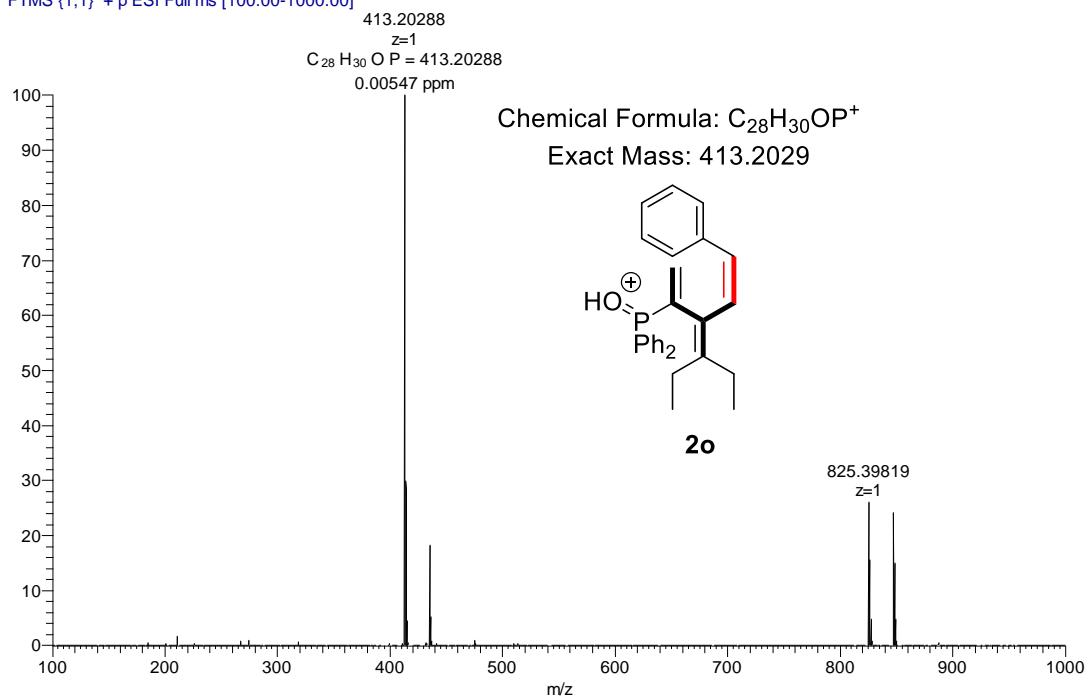


2o

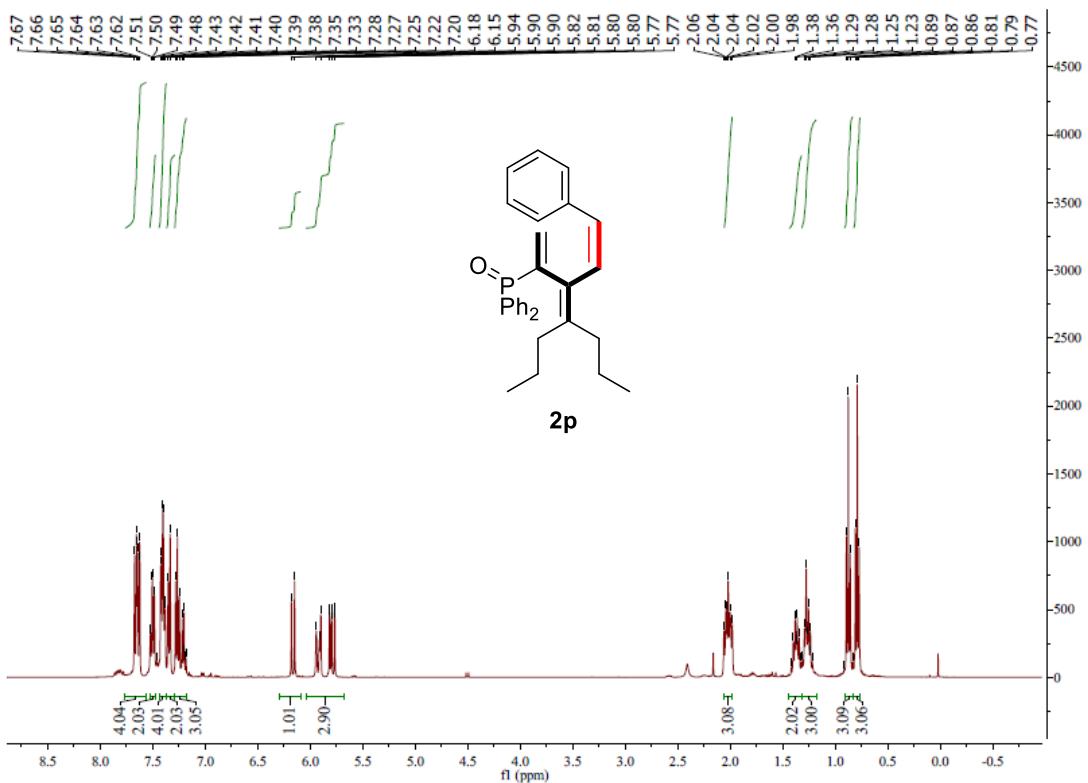


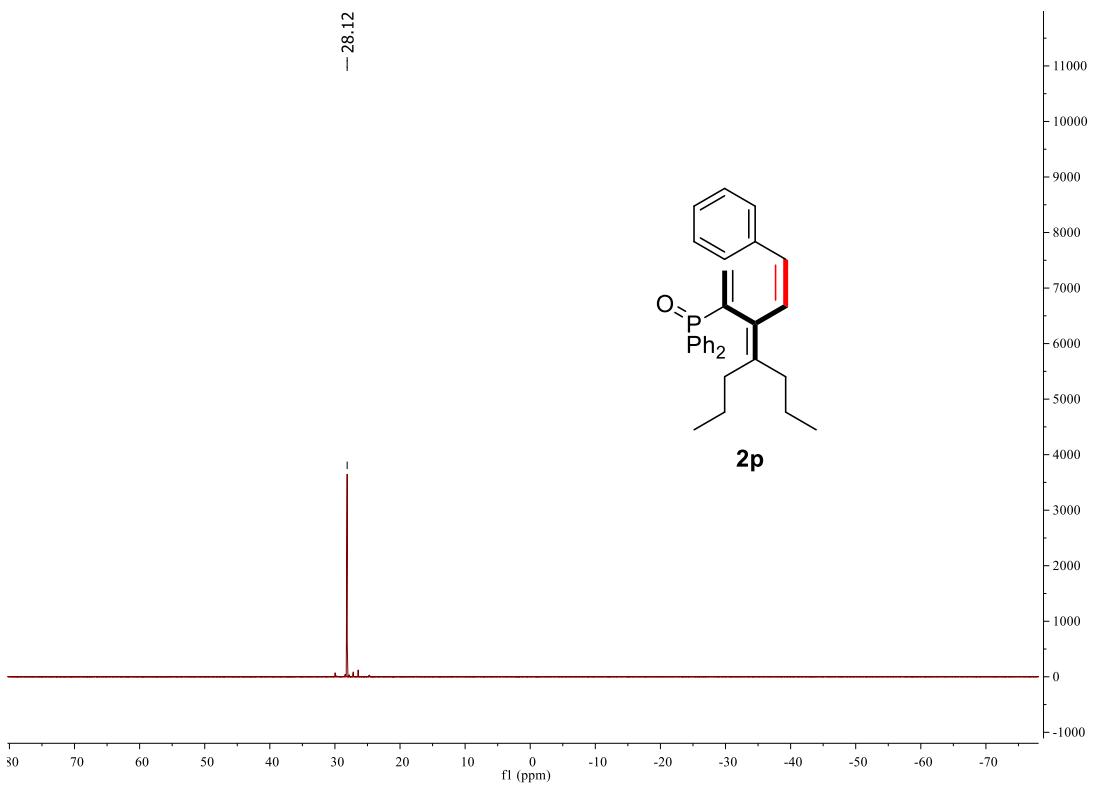
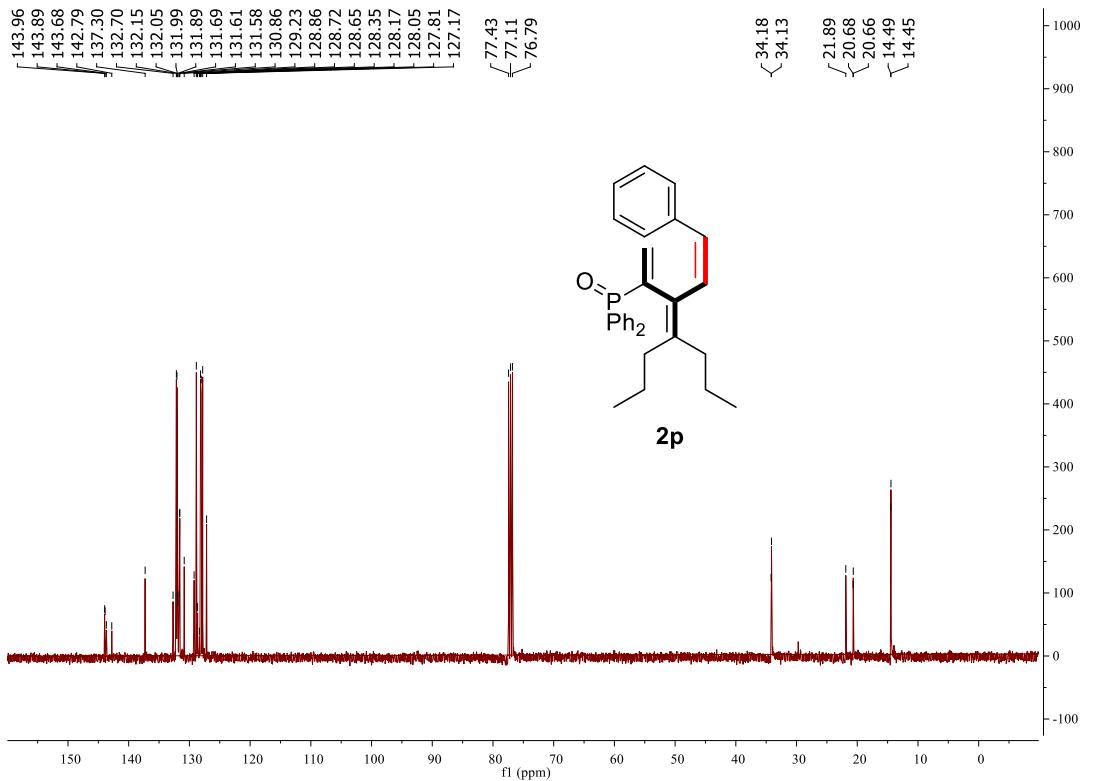


20181014-15 #25 RT: 0.36 AV: 1 NL: 8.33E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

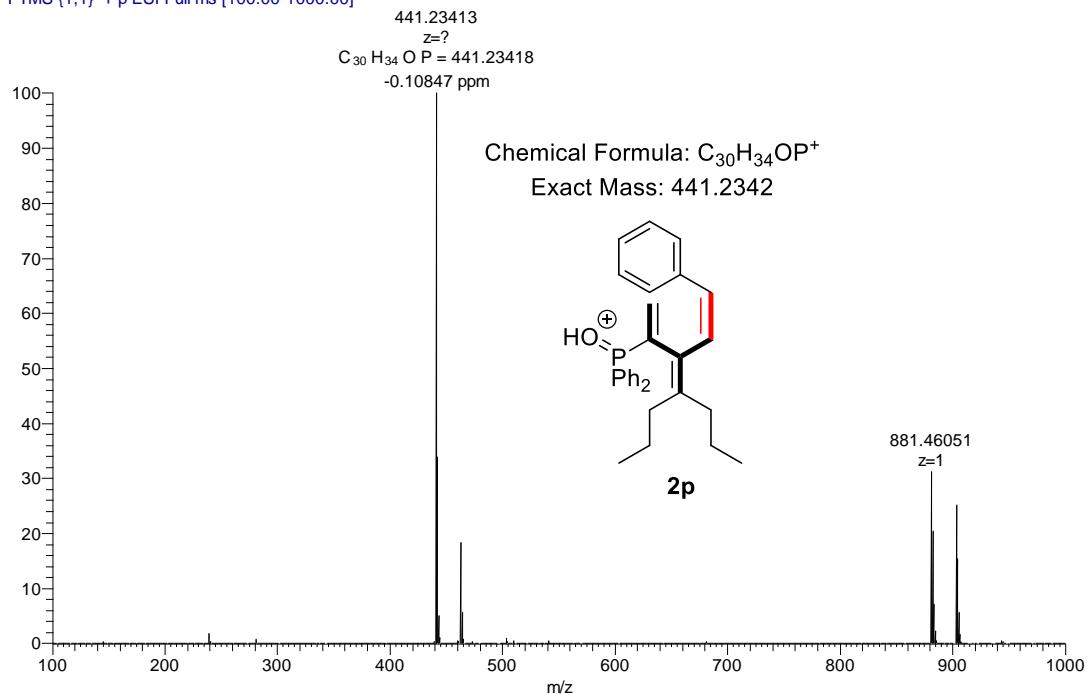


2p

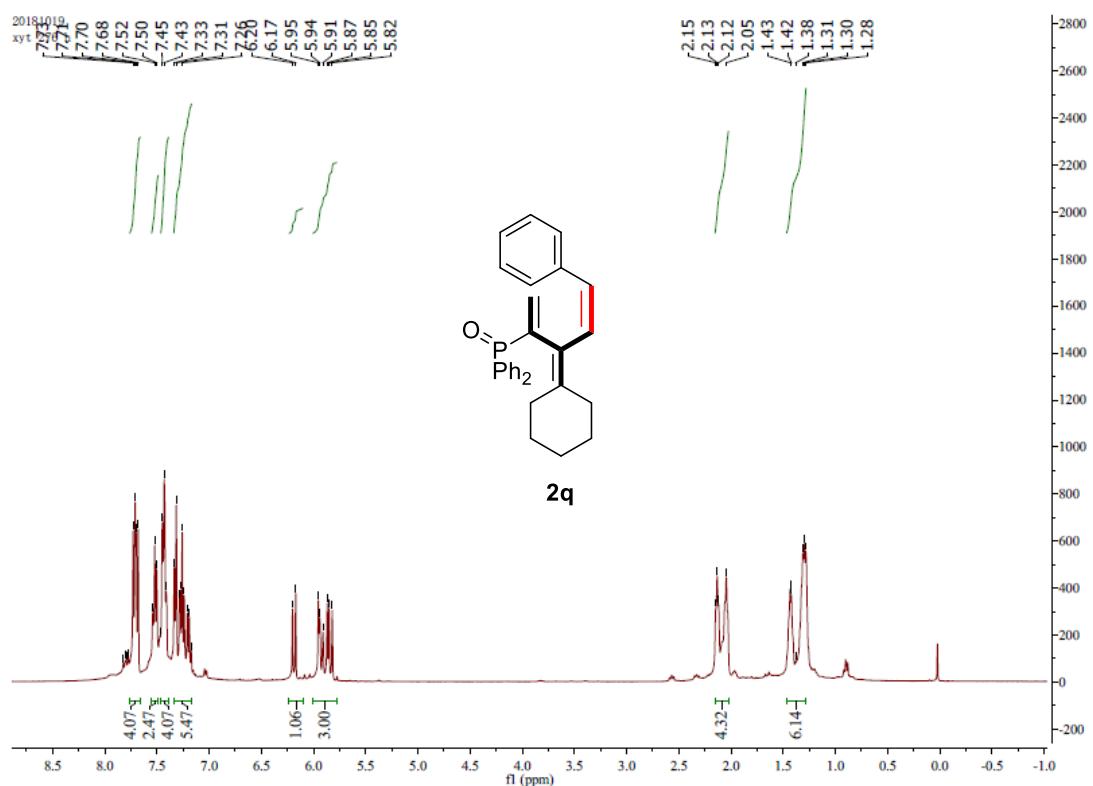


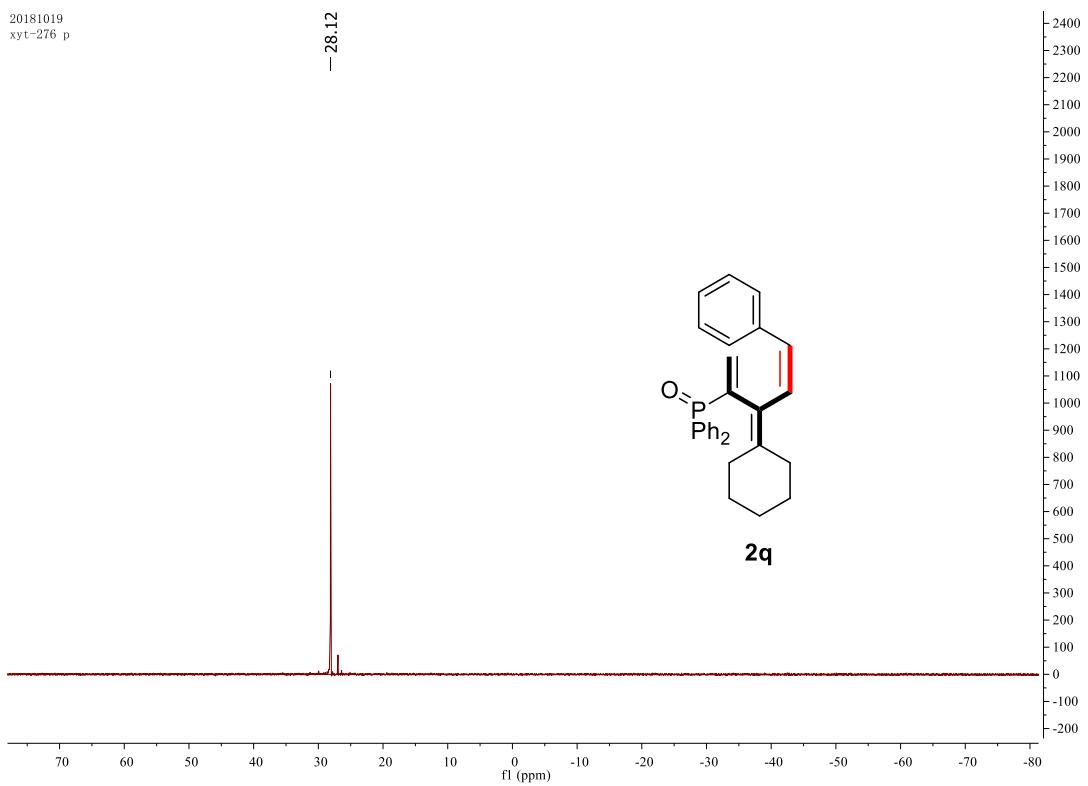
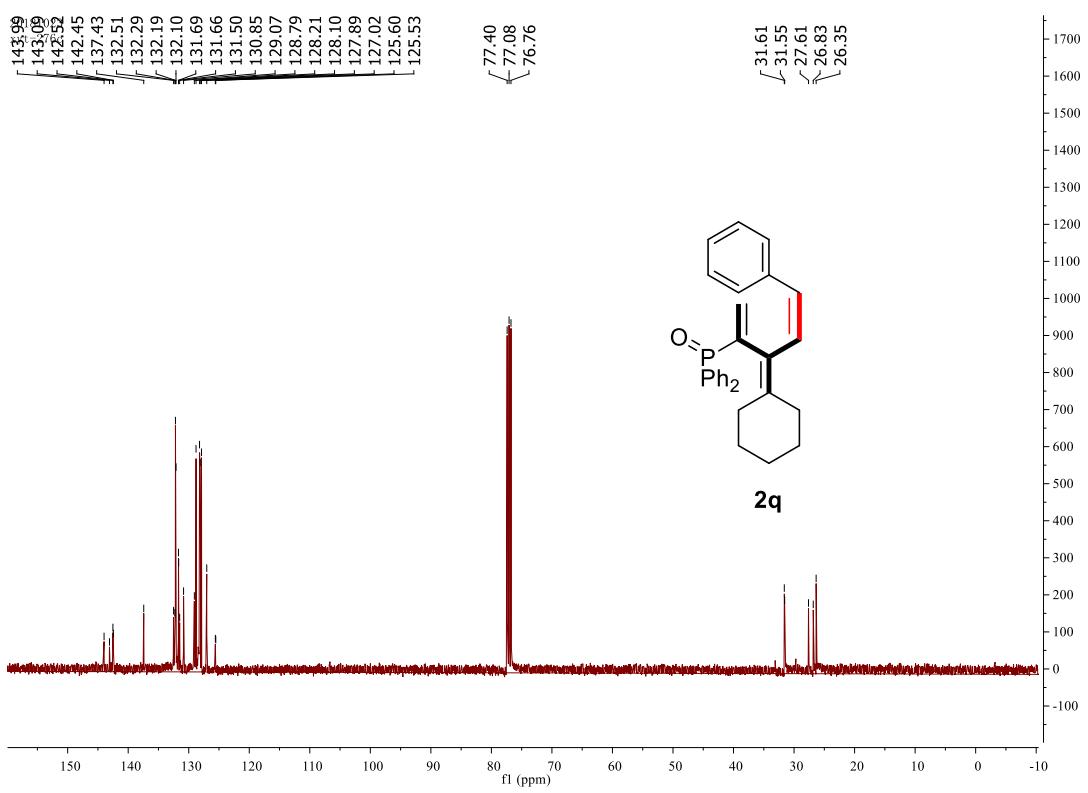


20181014-9 #53 RT: 0.77 AV: 1 NL: 7.72E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

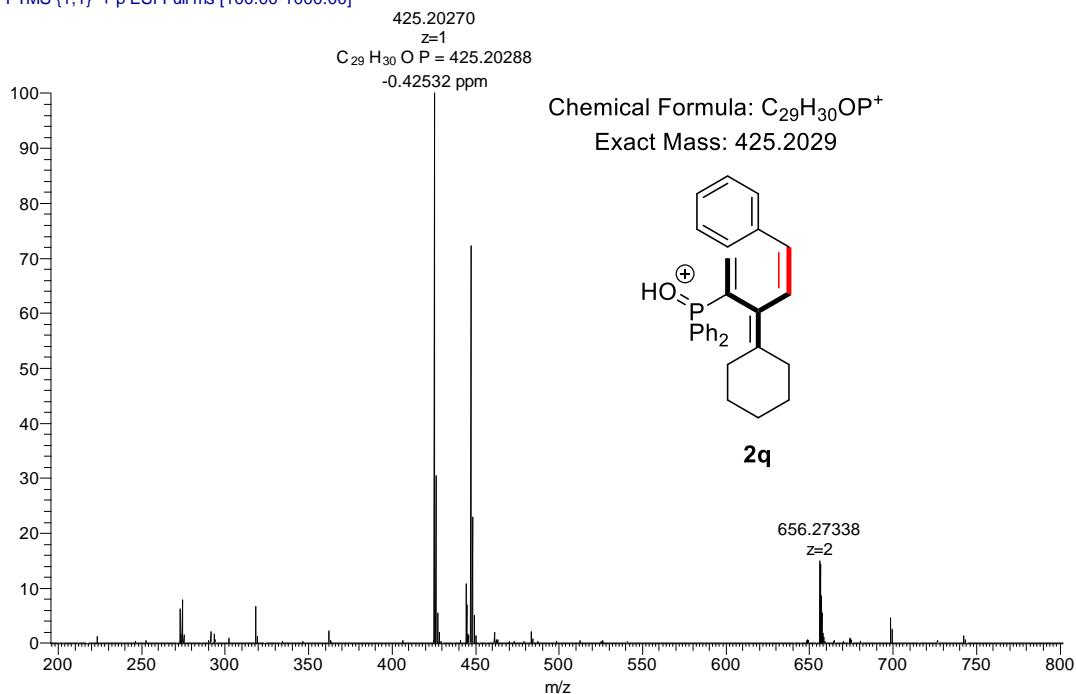


2q

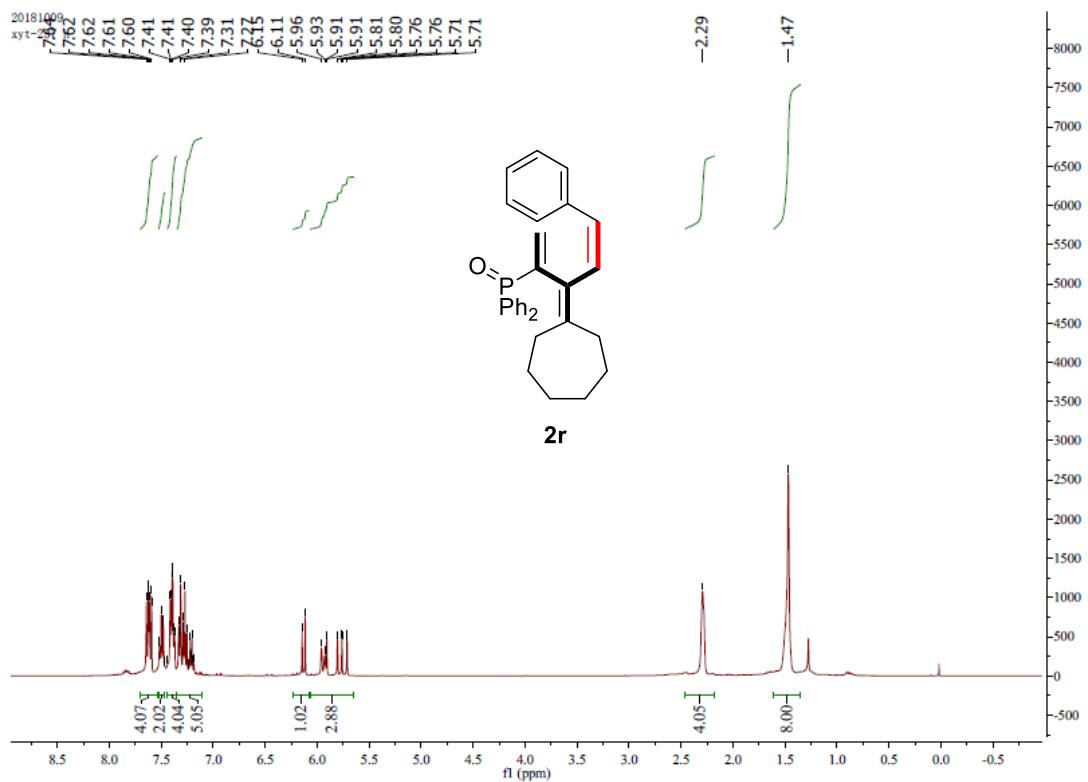


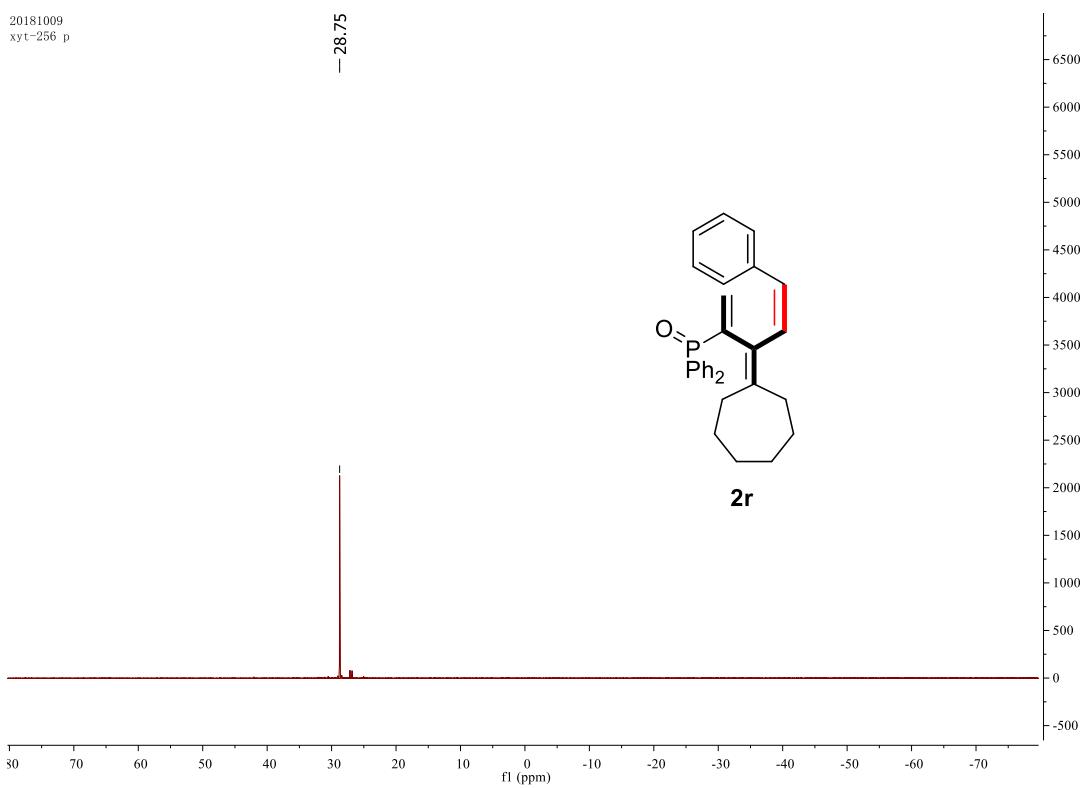
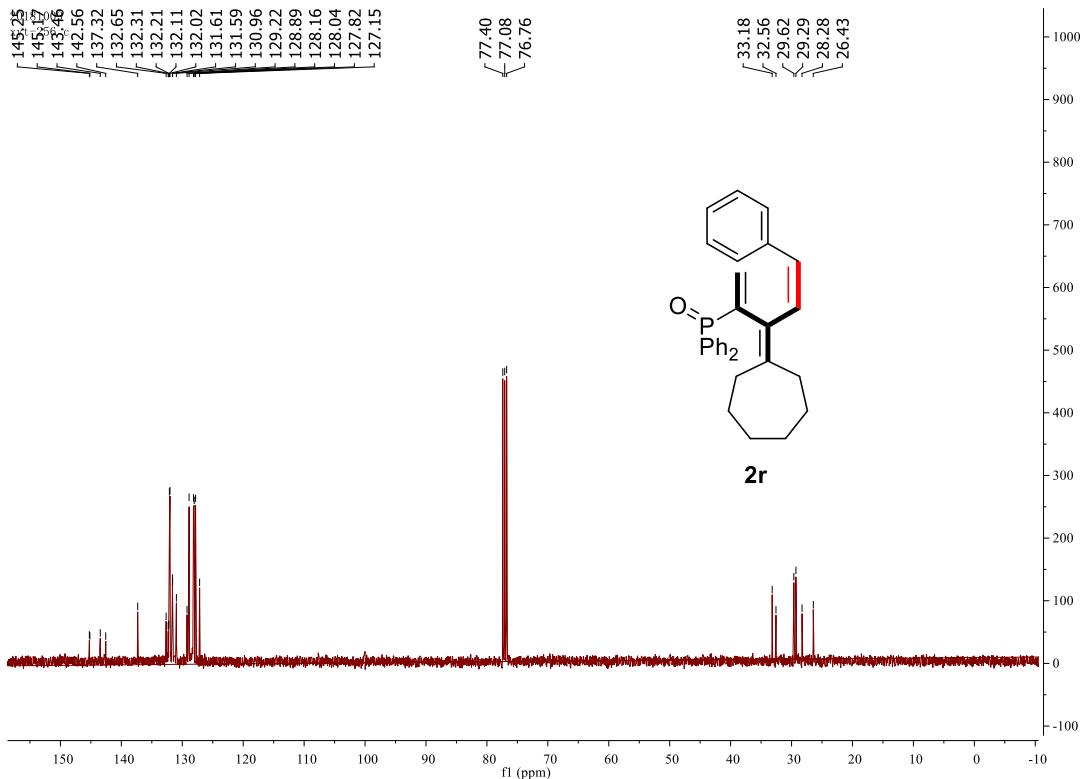


NJNY-3 #47 RT: 0.68 AV: 1 NL: 4.10E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

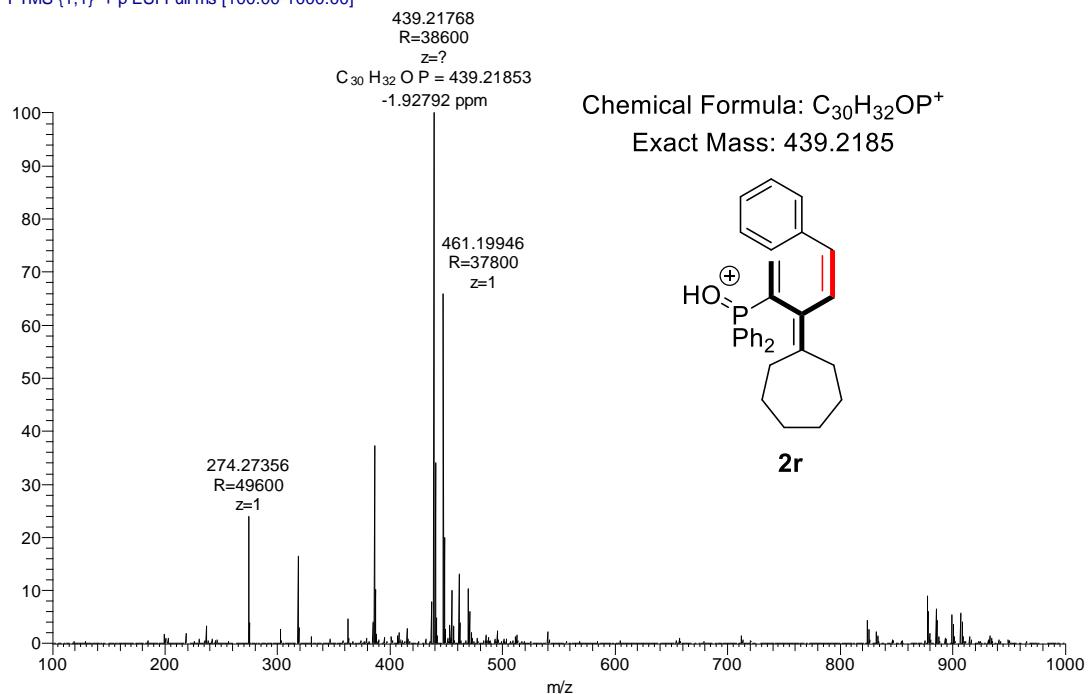


2r

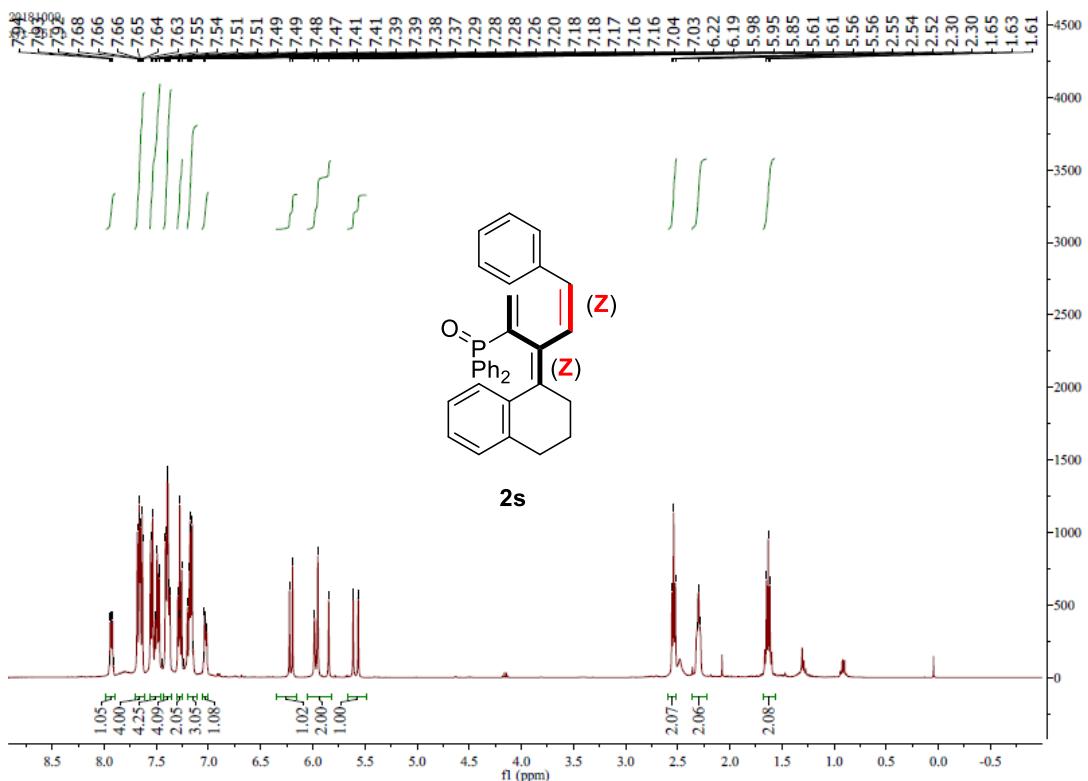


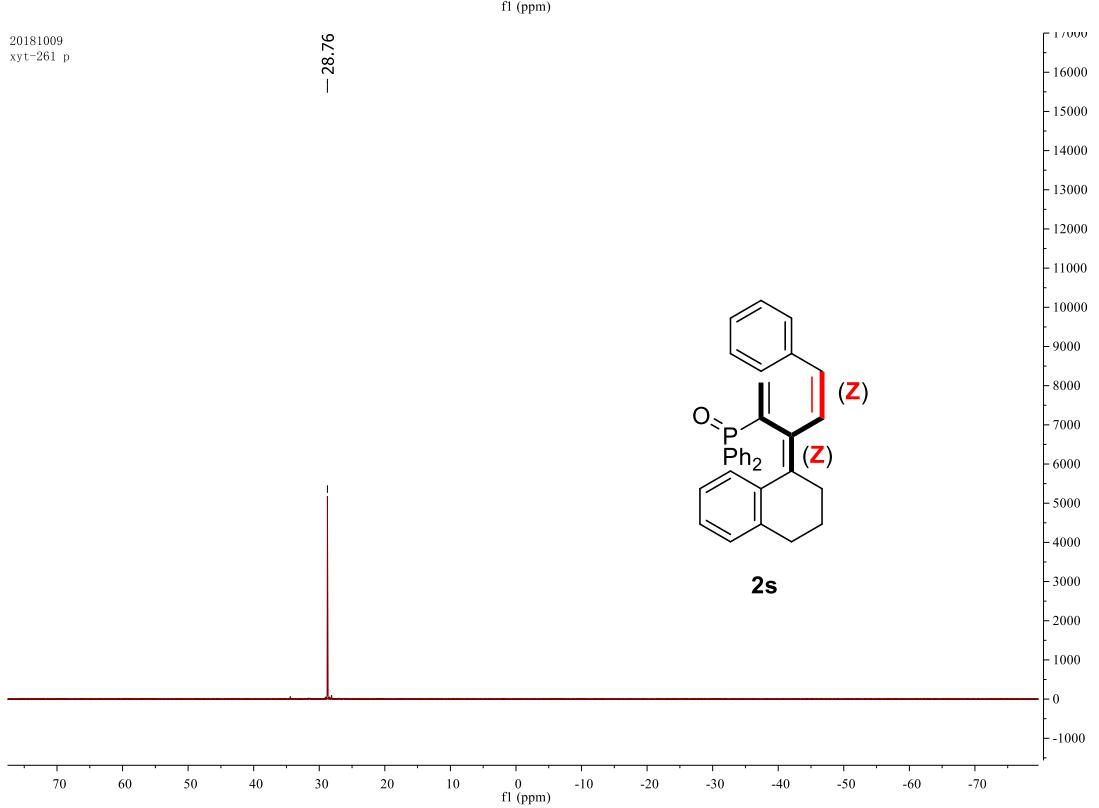
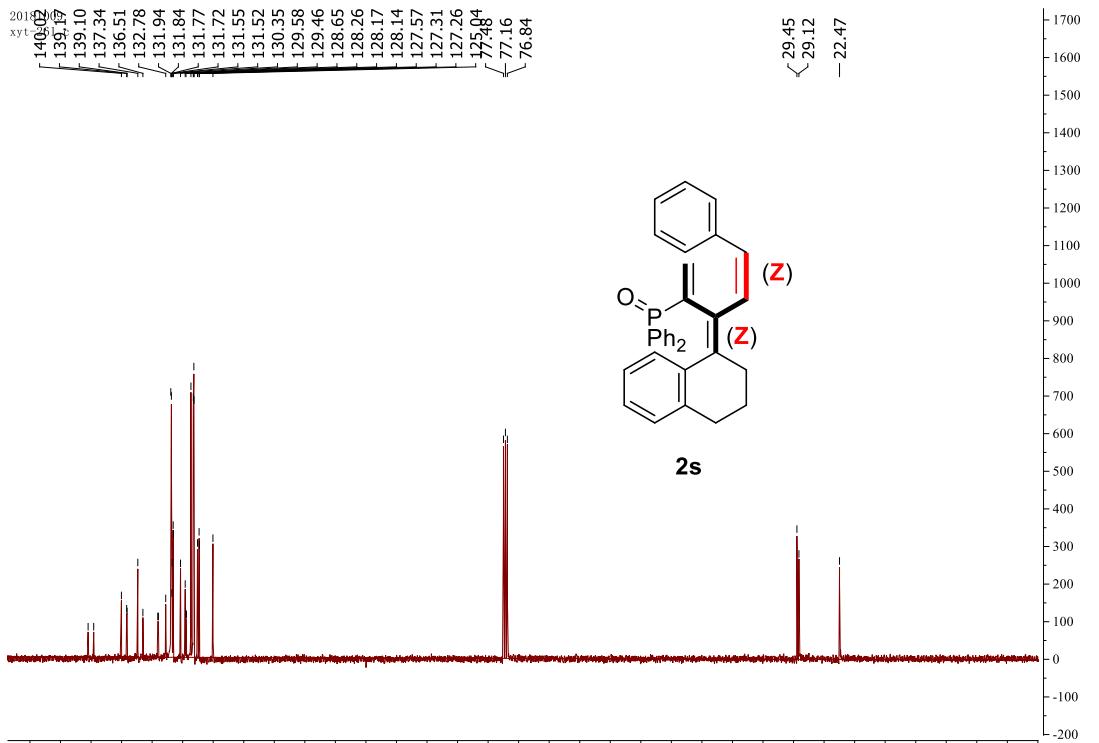


NJNY-20180812-5 #47 RT: 0.61 AV: 1 NL: 2.24E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

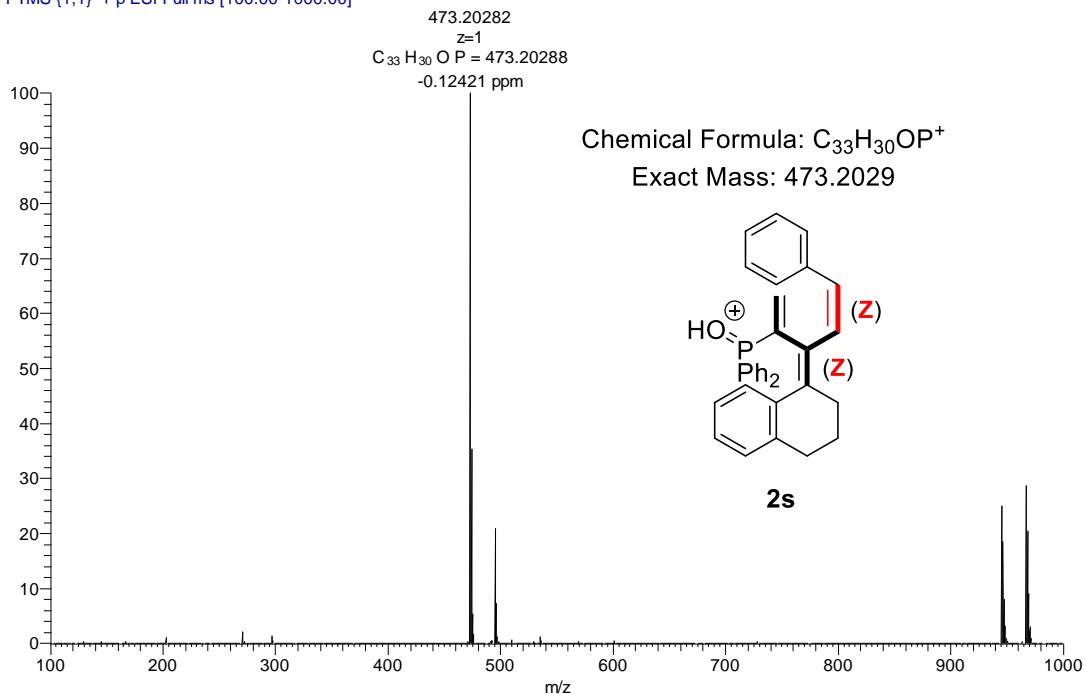


2s

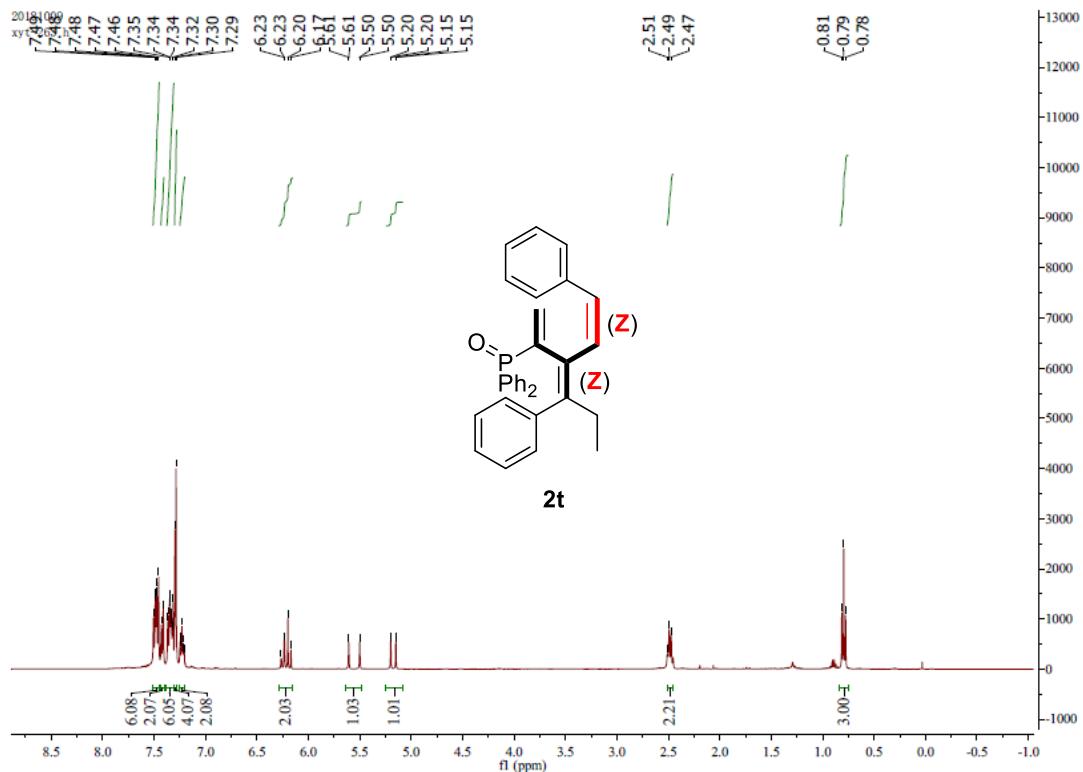


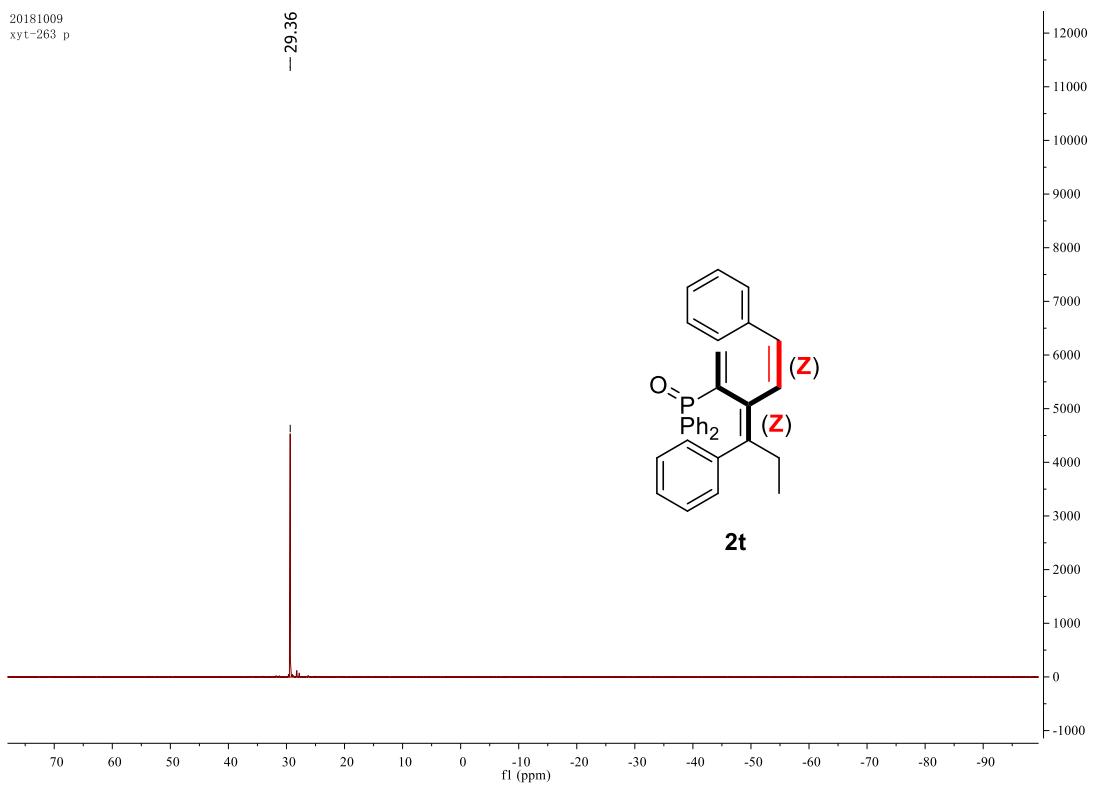
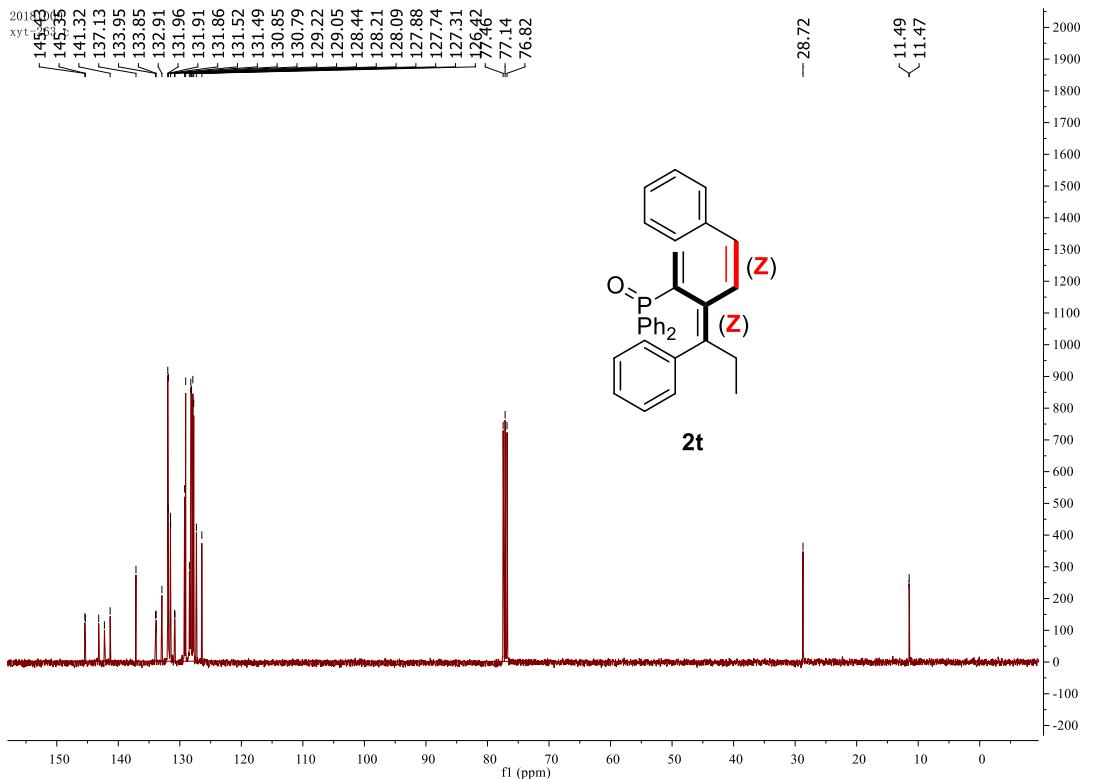


20181014-7 #49 RT: 0.71 AV: 1 NL: 5.70E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

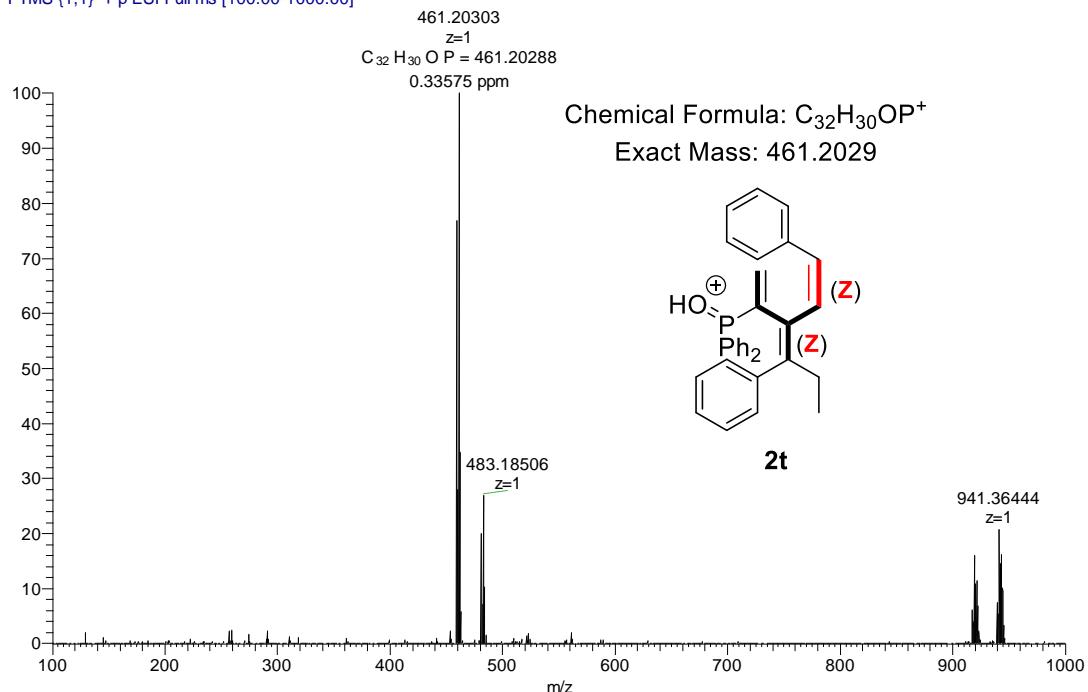


2t

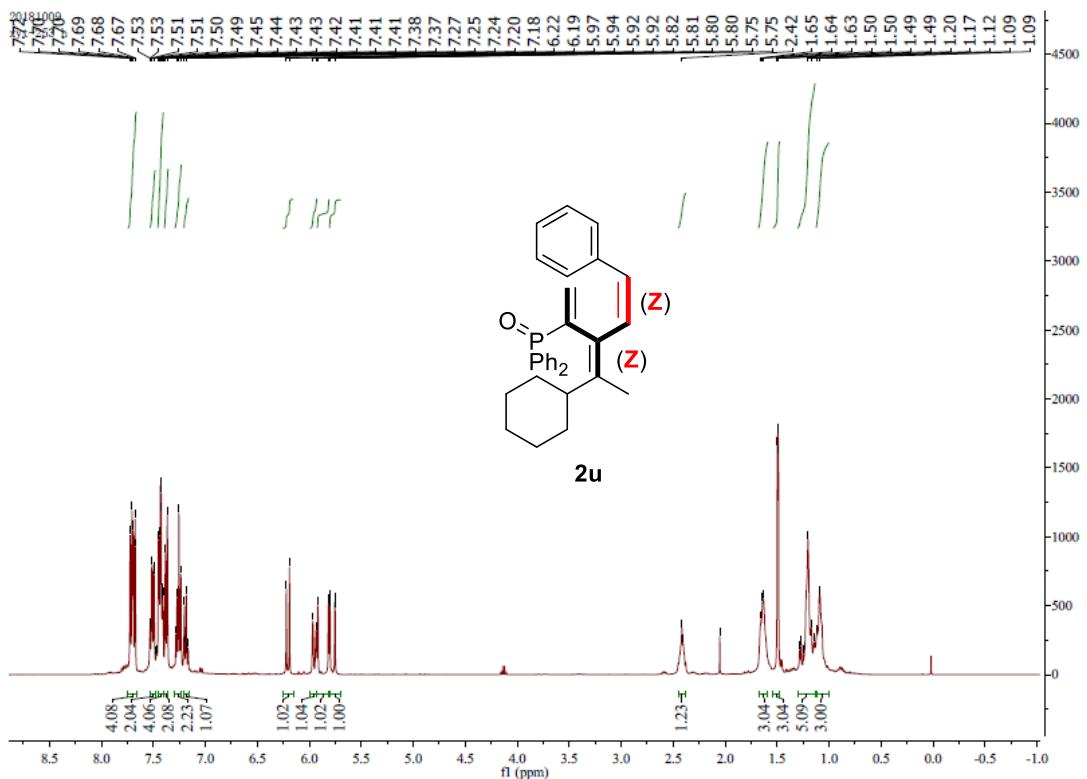


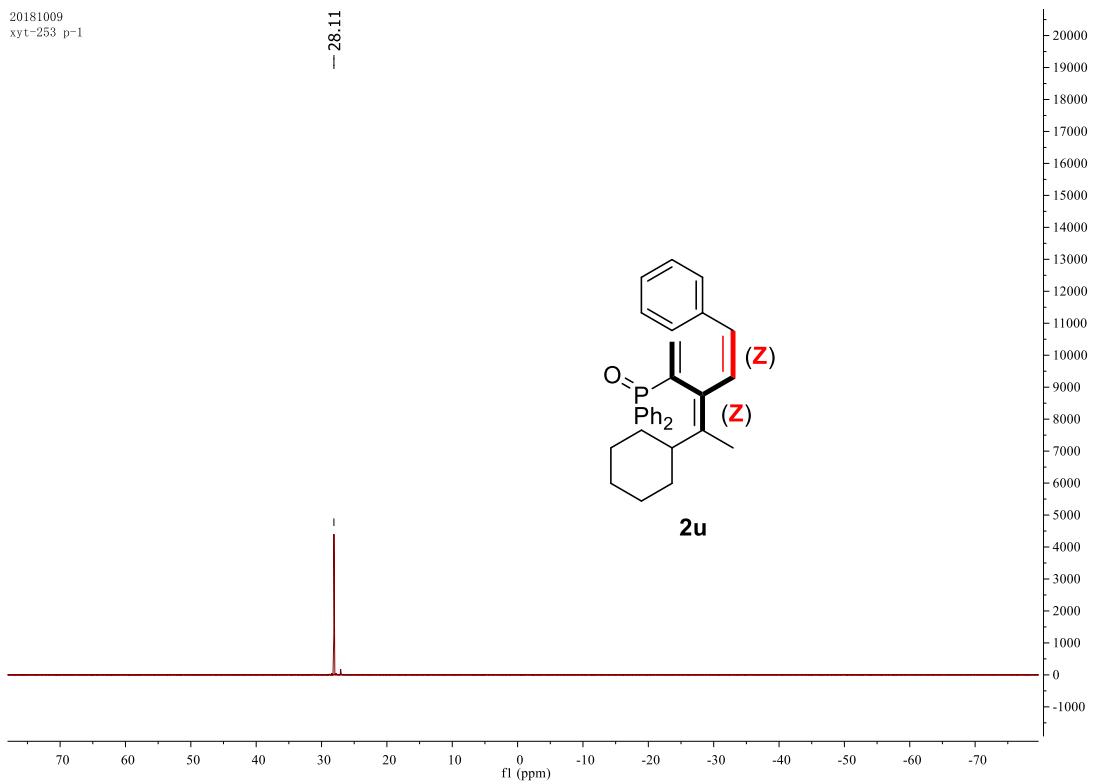
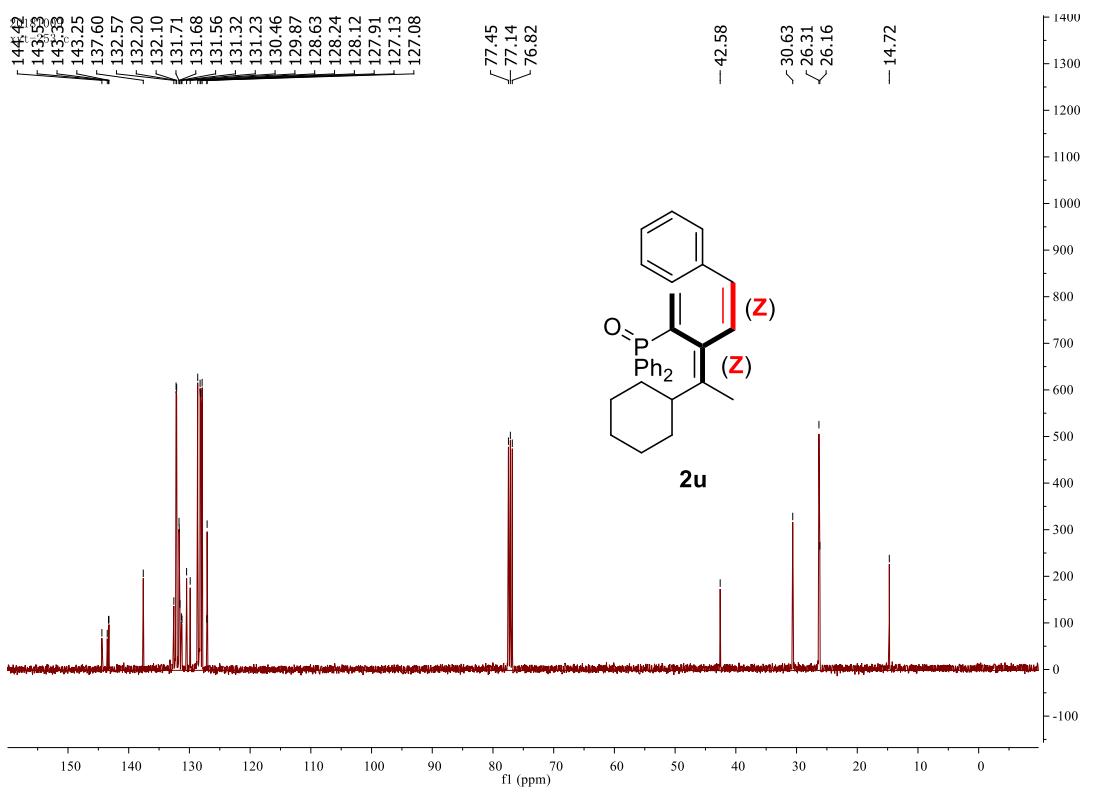


20181014-18 #27 RT: 0.40 AV: 1 NL: 1.67E6
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]



2u





20181014-16 #11 RT: 0.16 AV: 1 NL: 1.71E7
T: FTMS {1,1} + p ESI Full ms [100.00-1000.00]

