*Supporting Information*

# Electrochemical Synthesis of Phosphorylated Oxazolines from *N*-Allylamides

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**Experimental:**

**General Method:**

IR spectra were recorded on a Bruker Tensor 37 (FTIR) spectrophotometer. $^{1}H$ NMR spectra were recorded on Bruker Avance 400 (400 MHz), 600 (600 MHz) spectrometers at 295 K in $CDCl_3$; chemical shifts ($\delta$ ppm) and coupling constants (Hz) are reported in a standard fashion concerning either internal standard tetramethylsilane (TMS) ($\delta_H$ = 0.00 ppm) or $CDCl_3$ ($\delta_H$ = 7.26 ppm). In the $^{1}H$-NMR, the following abbreviations were used throughout: s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, sept = septet, dd = doublet of doublet, m = multiplet, and br. s = broad singlet. $^{13}C\{^{1}H\}$ NMR spectra were recorded on Bruker Avance 400 (101 MHz) and Bruker Avance 600 (151 MHz) spectrometers at room temperature in $CDCl_3$; chemical shifts ($\delta$ ppm) are reported relative to $CDCl_3$ [$\delta_C$ = 77.16 ppm (central line of the triplet)]. In the $^{13}C\{^{1}H\}$ NMR, the nature of carbons (C, CH, $CH_2$, and $CH_3$) was determined by recording the DEPT-135 spectra. The assignment of signals was confirmed by $^{1}H$, $^{13}C\{^{1}H\}$ CPD, and DEPT spectra. $^{31}P$ NMR spectra were recorded on a Bruker Avance 600 (243 MHz) spectrometer at room temperature in $CDCl_3$. $^{19}F$ NMR spectra were recorded on a Bruker Avance 600 (243 MHz) spectrometer at room temperature in $CDCl_3$. High-resolution mass spectra (HR-MS) were recorded on an Agilent 6538 UHD Q-TOF electron spray ionization (ESI) mode and atmospheric pressure chemical ionization (APCI) modes. All small-scale reactions were carried out using a Schlenk tube. Electrochemical reactions were carried out using an IKA ElectraSyn 2.0. Cyclic voltammetry experiments were carried out on a CH-instrument (Model: CHI1210C). Reactions were monitored by TLC on silica gel using a combination of hexane and ethyl acetate as eluents. Solvents were distilled before use; petroleum ether, with a boiling range of 60-80 °C, was used. Acetophenones, NaH, methyltriphenylphosphonium bromide, benzoic acids, oxalyl chloride, aryl bromides, diethyl phosphite, magnesium, iodine, electrolytes, ferrocene, triethylamine, were purchased from Sigma-Aldrich/TCI/local and used as received. Acme's silica gel (60-120 mesh) was used for column chromatography (approximately 20 g per 1 g of crude material).

**General procedure for cyclic voltammetry (CV):**

Cyclic voltammetry experiments were carried out on a CH instrument. Cyclic voltammetry was performed in a three-electrode cell connected to a 10 mL vial at room temperature. The working electrode was a steady glassy carbon electrode, while the counter electrode was a platinum electrode. The reference was an $Ag/AgNO_3$ electrode, and 10 mL of $CH_3CN$ containing 0.1 M of

$^{n}Bu_4NPF_6$ was poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 50 mV/s, ranging from -0.5 V to 1.5 V.

**The polishing material and method:** We initially cleaned the electrodes with distilled water, ethanol, and acetone, and the working electrode was polished with a polishing pad that contained a small amount of alumina powder and a small amount of distilled water.

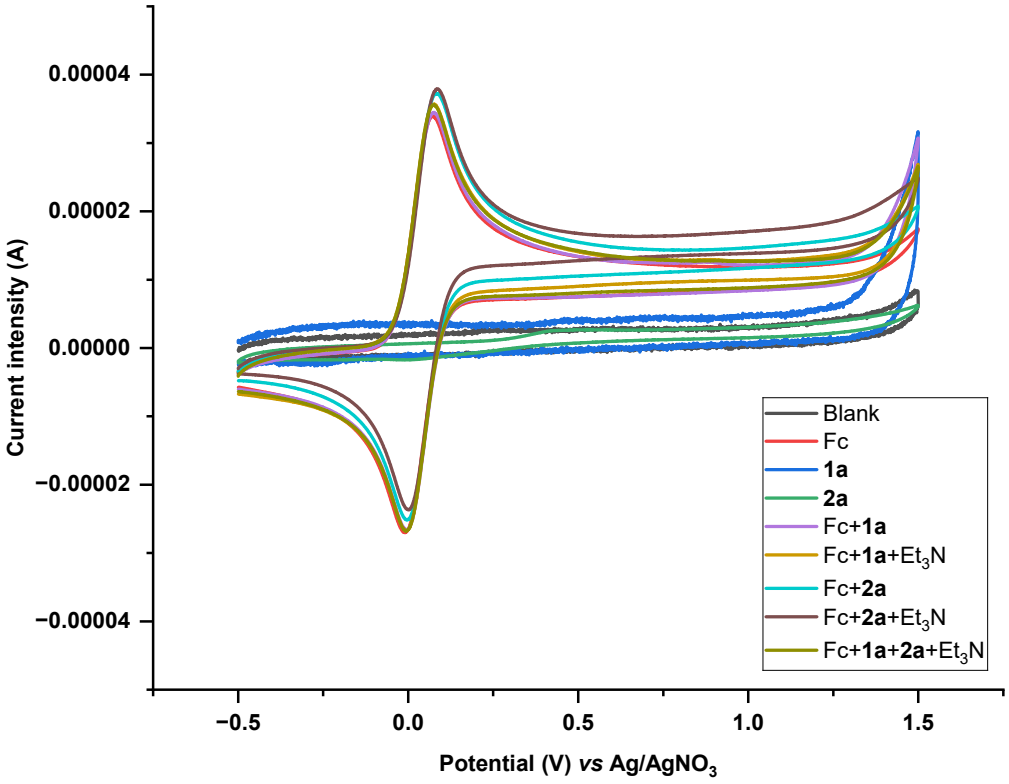
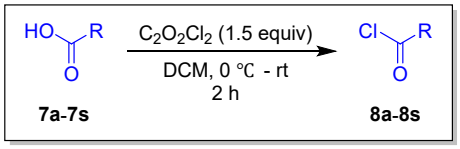



**Figure S1: Cyclic Voltammetry**

**General Procedure 1 (GP-1) for the preparation of acyl chlorides:**




To an oven-dried round-bottomed flask equipped with a magnetic stir bar, were added acid **7a-7s** (122.12 - 250.33 mg, 1 mmol, 1 equiv), oxalyl chloride (190.4 mg, 1.5 equiv), DMF (catalytic, 0.1 mL), and DCM at 0 ℃. Then, the reaction mixture was stirred at room temperature for 2 hours. Then, the solvent was concentrated under reduced pressure, and the crude product was used in the further step without purification.

**General Procedure 2 (GP-2) for the synthesis of *N*-allylamides 1:**[1–4]

To an oven-dried round-bottomed flask equipped with a magnetic stir bar, were added methyl/ethyl triphenylphosphonium bromide (2.14 g, 6.0 mmol, 1.2 equiv) and THF (5 mL). The suspension was cooled to 0 °C, NaH (60% dispersion in mineral oil, 400 mg, 10 mmol, 2 equiv) was added, and the resulting yellow suspension was stirred at 0 °C for 45 min. To this suspension, a solution of ketone/aldehyde **9a-9h** (602.0 - 995.25 mg, 5.0 mmol, 1.0 equiv) in THF (0.5 M) was added dropwise, and the resulting mixture was warmed gradually to room temperature and 12 h. The reaction was quenched with water and extracted with DCM (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The crude material was purified by column chromatography on silica gel using petroleum ether/ethyl acetate (petroleum ether: ethyl acetate = 100:00 to 99:01) to obtain the pure products **10a-10h** (55%-75%), as colorless oils.

To an oven-dried round-bottomed flask equipped with a magnetic stir bar, were added **10a-10h** (118.2 - 492.7 mg, 2.5 mmol, 1.0 equiv), *N*-Bromosuccinimide (489.4 mg, 2.75 mmol, 1.1 equiv), pTsOH (43.0 mg, 0.25 mmol, 0.1 equiv), and dry THF (3 mL). The reaction mixture was stirred at 100 ℃ for 4 h. The reaction was quenched with water and extracted with DCM (3 × 10 mL). The combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The crude material was purified by column chromatography on silica gel using petroleum ether/ethyl acetate (petroleum ether: ethyl acetate = 100:00 to 99:01) to obtain the pure products **11a-11h** (65%-80%), as colorless oils.

To an oven-dried round-bottomed flask equipped with a magnetic stir bar, were added **11a-11h** (197.1 - 276 mg, 1 mmol, 1.0 equiv), $NaN_3$ (78.0 mg, 1.2 mmol, 1.2 equiv), and THF (3 mL) and

$H_2O$ (1 mL). The reaction mixture was stirred at 50 ℃ for 2 h. The solution was cooled to room temperature, then $PPh_3$ (393.4 mg, 1.5 mmol, 1.5 equiv) was added, and the mixture was stirred for 20 h at room temperature. Most of the THF was removed under vacuum, and the residue was dissolved with aqueous HCl (5 mL, 1 M). The aqueous layer was extracted with diethyl ether (3 × 20 mL), then basified with solid NaOH and again extracted with diethyl ether (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure, which was directly used without further purification.

To an oven-dried round-bottomed flask equipped with a magnetic stir bar, were added **12a**-**12h** (133.2 - 212.1 mg, 0.5 mmol, 1.0 equiv), triethylamine (101.2 mg, 1.0 mmol, 2.0 equiv), DMAP (12.2 mg, 0.1 mmol, 20 mol%), and DCM (5 mL). The reaction was cooled in an ice bath. To it was added the corresponding acyl chloride **8a**-**8s** (77.3 - 147.8 mg, 0.55 mmol, 1.1 equiv) dropwise at 0 °C. The reaction mixture was stirred at room temperature for 2 h. It was then quenched with saturated aqueous $NH_4Cl$ solution and extracted with DCM (3 × 10 mL). The combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The crude material was purified by column chromatography on silica gel using petroleum ether/ethyl acetate (petroleum ether: ethyl acetate = 80:20 to 70:30) to obtain the pure product **1a-1w** (75%-88%), as white solids.

To an oven-dried round-bottomed flask equipped with a magnetic stir bar, was added **1a** (118.6 mg, 0.5 mmol, 1.0 equiv), Lawesson reagent (303.6 mg, 0.75 mmol, 1.5 equiv), and THF (1 mL). The reaction mixture was stirred at 60 °C for 4 h. Then, the concentrated reaction mixture and crude material were purified by column chromatography on silica gel using petroleum ether/ethyl acetate (petroleum ether: ethyl acetate = 99:01 to 98:02) to obtain the pure product **1x** (45%), as a yellow oil.

**Table S1**: The following *N*-allylamides are prepared by using the literature reports **1a-1x**.[1–4]

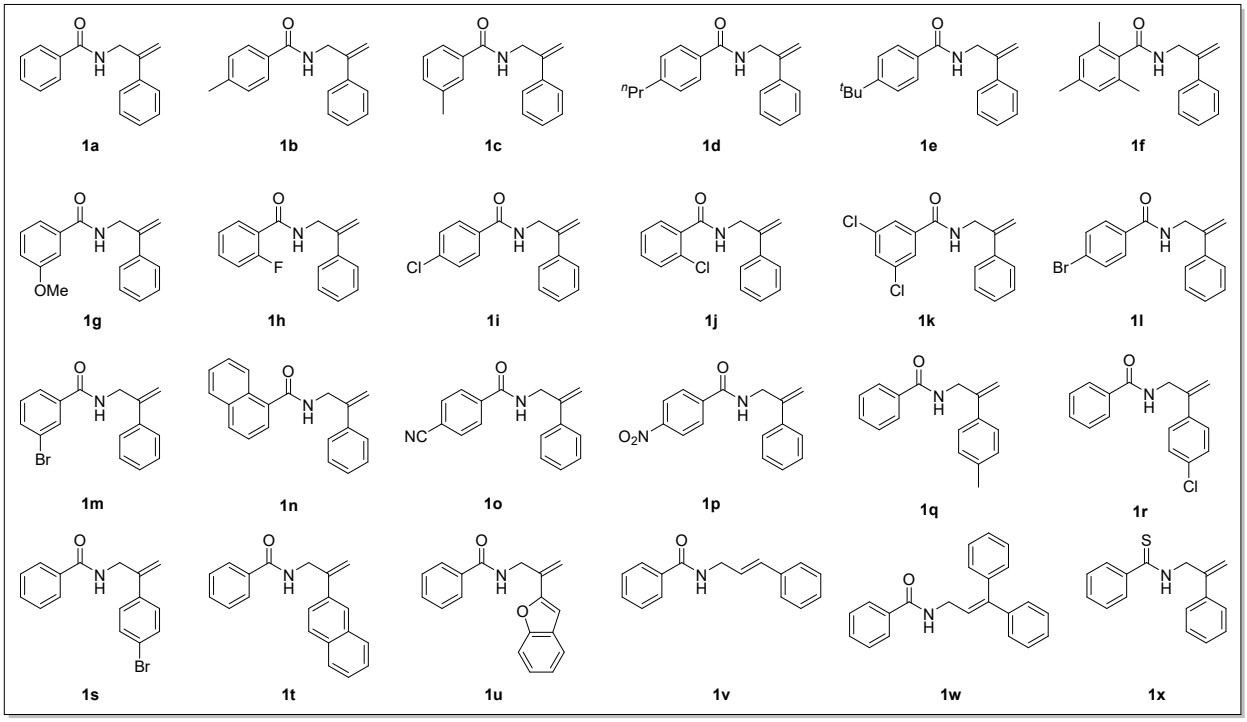
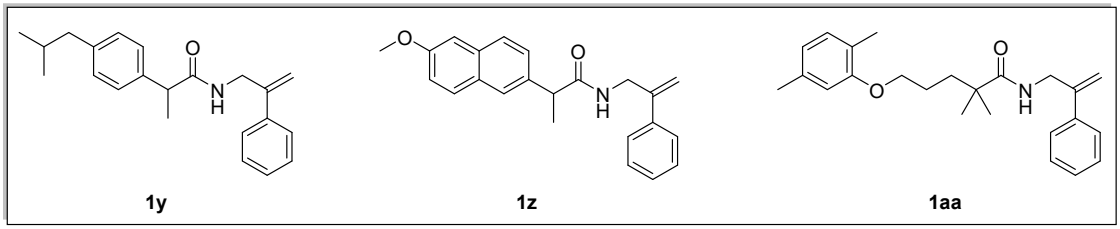


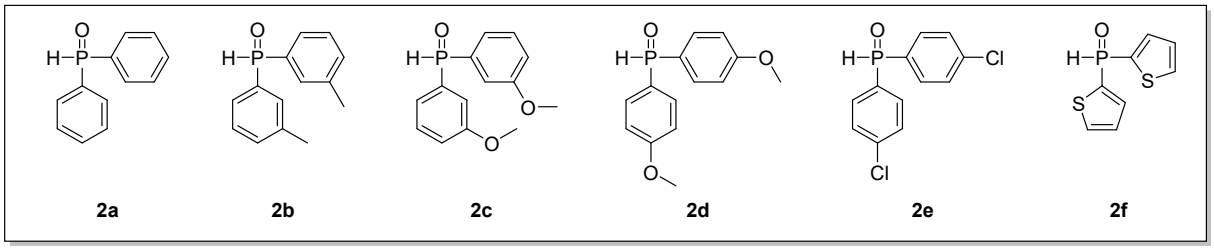

**Table S2:** The following carbazates **1y**, **1z**, and **1aa** (Table S2) are unreported.




**General Procedure 3 (GP-3) for the preparation of diarylphosphine oxides 2:**[5–8]

To an oven-dried round-bottomed flask equipped with a magnetic stir bar, were added magnesium (364.5 mg, 15 mmol, 3 equiv), a catalytic amount of iodine (190.3 mg, 1.5 mmol, 10 mol%), and dry THF (10 mL) under a nitrogen atmosphere. Afterwards, aryl bromide **13a-13f** (2.3 g to 2.9 g, 15 mmol, 3 equiv) was also added dropwise. The reaction mixture was refluxed for 30 minutes before being cooled to 0 °C. The solution of diethyl phosphite (690.5 mg, 5 mmol, 1.0 equiv.) in 5 mL dry THF was added dropwise to the reaction system at 0 °C, and the reaction mixture was stirred for 12 h. It was then quenched with saturated aqueous $NH_4Cl$ solution and extracted with ethyl acetate (3 × 10 mL). The combined organic layers were dried over anhydrous sodium sulfate and concentrated under reduced pressure. The crude material was purified by column chromatography on silica gel using petroleum ether/ethyl acetate (DCM: MeOH = 99:01 to 98:02) to obtain the pure products **2a-2e** (55%-75%), as white solids (Table S1).

**Table S3:** The following starting materials, diarylphosphine oxides **2a**-**2f** (Table S3), are known in the literature.[5–8]




**General procedure (GP-4) for electrochemical synthesis of phosphorylated oxazolines 3:**

To an oven-dried ElectraSyn 2.0 undivided cell (6 mL) equipped with a magnetic stir bar, were added *N*-allylamides **1** (0.2 mmol), diarylphosphine oxides **2** (0.4 mmol), ferrocene (20 mol%), $Et_3N$ (0.2 mmol), $^nBu_4OAc$ (0.1 M), and a mixture of $CH_3CN$ and MeOH (5 mL, v:v = 4:1). The ElectraSyn vial cap equipped with a platinum plate (5.2 cm × 0.8 cm × 0.2 cm) as the cathode and a graphite plate (5.2 cm × 0.8 cm × 0.2 cm) as the anode was inserted into the reaction mixture. The reaction mixture was stirred and electrolyzed at a constant current of 5 mA at room temperature for 4 h. After the reaction was complete, the reaction mixture was concentrated under reduced pressure. Purifying the crude product by column chromatography on silica gel using DCM/MeOH (DCM: MeOH = 99:01 to 98:02) furnished the products **3aa-3aaa** (58 to 86%), as white solids.

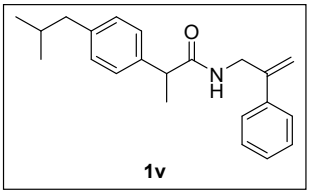
**Photographic guide for electrochemical reaction:**

**Electrochemical set-up**


**Left:** Select New “Experiment”; **Middle:** Select “Constant Current”; **Right:** Select “5 mA”


**Left:** Reference electrode chosen “No”; **Middle:** Select “Time”; **Right:** Select “4 hours”


**Left:** Select “0.2 mmol”; **Middle:** Alternate the polarity Choose “No”; **Right:** Select “Start the experiment”



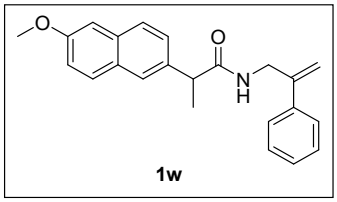

**2-(4-Isobutylphenyl)-*N*-(2-phenylallyl)propenamide (1v): GP-1** was carried out with ibuprofen (206.3 mg, 1 mmol), oxalyl chloride (190.4 mg, 1.5 mmol, 1.5 equiv), DMF (catalytic, 0.1 mL) in 5 mL of DCM at room temperature for 2 h. Followed by **GP-2** was carried out with 2-phenylprop-2-en-1-amine **12a** (133.2 mg, 0.5 mmol, 1.0 equiv), triethylamine (101.2 mg, 1.0 mmol, 2.0 equiv), DMAP (12.2 mg, 0.1 mmol, 20 mol%), 2-(4-isobutylphenyl)propanoyl chloride **8q** (123.6 mg, 0.55 mmol, 1.1 equiv) in 5 mL of DCM at room temperature for 2 h. Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate: 80:20 to 70:30) furnished the product **1v** (143.0 mg, 89%) as a white solid; mp = 131-132 °C. [TLC (petroleum ether/ethyl acetate) 70:30, $R_f$(**12a**) = 0.10, $R_f$(**8q**) = 0.90, $R_f$(**1v**) = 0.40, UV detection].

IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3054, 2343, 1735, 1654, 1552, 1268, 1186, 1085, 978, 901, 850, 730, 623, 532 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.35 – 7.27 (m, 5H), 7.10 – 7.01 (m, 4H), 5.41 (brs, 1H), 5.32 (d, $J$ = 0.8 Hz, 1H), 5.02 (d, $J$ = 0.8 Hz, 1H), 4.31 (dd, $J$ = 15.8, 6.0 Hz, 1H), 4.22 (dd, $J$ = 15.8, 5.5 Hz, 1H), 3.51 (q, $J$ = 7.2 Hz, 1H), 2.43 (d, $J$ = 7.2 Hz, 2H), 1.88 – 1.77 (m, 1H), 1.48 (d, $J$ = 7.2 Hz, 3H), 0.89 (d, $J$ = 6.6 Hz, 6H) ppm.

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) $\delta$ 174.4, 144.7, 140.8, 138.6, 138.3, 129.7 (2C), 128.5 (2C), 128.1, 127.4 (2C), 126.2 (2C), 113.4, 46.8, 45.1, 43.3, 30.3, 22.5 (2C), 18.4 ppm.

HRMS (ESI) m/z: $[(M)]^+$ calcd for $C_{22}H_{27}NO$ 321.2087; Found 321.2085.




**2-(6-Methoxynaphthalen-2-yl)-*N*-(2-phenylallyl)propenamide (1w): GP-1** was carried out with naproxen (230.2 mg, 2 mmol), oxalyl chloride (190.4 mg, 1.5 mmol, 1.5 equiv), DMF (catalytic, 0.1 mL) in 5 mL of DCM at room temperature for 2 h. Followed by **GP-2** was carried out with 2-phenylprop-2-en-1-amine **12a** (133.2 mg, 0.5 mmol, 1.0 equiv), triethylamine (101.2 mg, 1.0 mmol, 2.0 equiv), DMAP (12.2 mg, 0.1 mmol, 20 mol%), 2-(6-methoxynaphthalen-2-yl)propanoyl chloride **8r** (136.8 mg, 0.55 mmol, 1.1 equiv) in 5 mL of DCM at room temperature

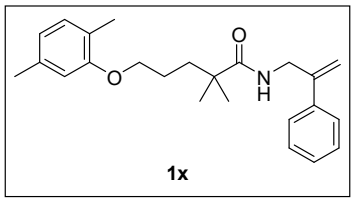
for 2 h. Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate: 80:20 to 70:30) furnished the product **1w** (141.6 mg, 82%) as a white solid; mp = 133-134 °C. [TLC (petroleum ether/ethyl acetate) 70:30, $R_f$(**12a**) = 0.10, $R_f$(**8r**) = 0.90, $R_f$(**1w**) = 0.40, UV detection].

IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3053, 2932, 2342, 1651, 1500, 1440, 1274, 1190, 1110, 982, 905, 617, 533 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.64 (d, $J$ = 8.6 Hz, 2H), 7.56 (d, $J$ = 1.1 Hz, 1H), 7.33 – 7.23 (m, 6H), 7.15 (dd, $J$ = 8.9, 2.5 Hz, 1H), 7.11 (d, $J$ = 2.4 Hz, 1H), 5.61 (t, $J$ = 4.8 Hz, 1H), 5.31 (d, $J$ = 0.7 Hz, 1H), 5.04 (d, $J$ = 0.8 Hz, 1H), 4.31 (dd, $J$ = 15.8, 6.0 Hz, 1H), 4.23 (dd, $J$ = 15.8, 5.6 Hz, 1H), 3.91 (s, 3H), 3.67 (q, $J$ = 7.2 Hz, 1H), 1.57 (d, $J$ = 7.2 Hz, 3H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 174.2, 157.7, 144.5, 138.4, 136.3, 133.7, 129.3, 128.9, 128.4 (2C), 128.0, 127.5, 126.3, 126.1 (3C), 119.1, 113.4, 105.6, 55.4, 47.0, 43.2, 18.4 ppm.

HRMS (ESI) m/z: $[(M + K)]^+$ calcd for $C_{23}H_{23}KNO_2$ 384.1360; Found 384.1336.



**1x**

**5-(2,5-Dimethylphenoxy)-2,2-dimethyl-*N*-(2-phenylallyl)pentanamide (1x): GP-1** was carried out with gemfibrozil (250.3 mg, 2 mmol), oxalyl chloride (190.4 mg, 1.5 mmol, 1.5 equiv), DMF (catalytic, 0.1 mL) in 5 mL of DCM at room temperature for 2 h. Followed by **GP-2** was carried out with 2-phenylprop-2-en-1-amine **12a** (133.2 mg, 0.5 mmol, 1.0 equiv), triethylamine (101.2 mg, 1.0 mmol, 2.0 equiv), DMAP (12.2 mg, 0.1 mmol, 20 mol%), 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoyl chloride **8s** (147.8 mg, 0.55 mmol, 1.1 equiv) in 5 mL of DCM at room temperature for 2 h. Purification of the crude material by silica gel column chromatography (petroleum ether/ethyl acetate: 80:20 to 70:30) furnished the product **1x** (155.3 mg, 85%) as a white solid; mp = 134-135 °C. [TLC (petroleum ether/ethyl acetate) 70:30, $R_f$(**12a**) = 0.10, $R_f$(**8s**) = 0.90, $R_f$(**1x**) = 0.40, UV detection].

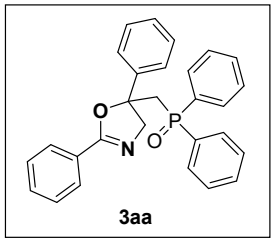
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3054, 2571, 1651, 1500, 12275, 1190, 1110, 905, 829, 749, 701, 619, 530 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.32 – 7.23 (m, 2H), 7.16 (dd, $J$ = 8.1, 6.8 Hz, 2H), 7.11 (d, $J$ = 7.3 Hz, 1H), 6.86 (d, $J$ = 7.5 Hz, 1H), 6.52 (d, $J$ = 7.4 Hz, 1H), 6.45 (s, 1H), 5.90 (t, $J$ = 5.5 Hz, 1H),

5.28 (d, $J$ = 0.4 Hz, 1H), 5.07 (d, $J$ = 0.9 Hz, 1H), 4.19 (d, $J$ = 5.7 Hz, 2H), 3.65 (t, $J$ = 5.5 Hz, 2H), 2.18 (s, 3H), 2.05 (s, 3H), 1.50 (s, 4H), 1.02 (s, 6H) ppm.

$^{13}$C NMR (101 MHz, $CDCl_3$) $\delta$ 177.1, 156.8, 144.9, 138.4, 136.2, 130.2, 128.3 (2C), 127.8, 126.0 (2C), 123.2, 120.6, 113.2, 111.8, 67.7, 43.0, 41.7, 37.3, 25.4 (2C), 24.8, 21.3, 15.8 ppm.

HRMS (ESI) m/z: $[(M + H_2O)]^+$ calcd for $C_{24}H_{33}NO_3$ 383.2455; Found 383.2457.



**3aa**

**((2,5-Diphenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide) (3aa): GP-4** was carried out with *N*-(2-phenylallyl)benzamide **1a** (47.5 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^{n}Bu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3aa** (74.4 mg, 85%), as a white solid; mp = 139-140 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1a**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3aa**) = 0.40, UV detection].

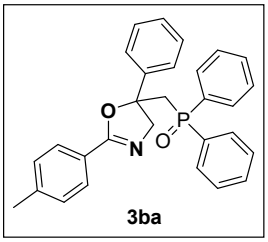
IR (MIR-ATR, 4000-400 cm$^{-1}$) $\nu_{max}$ = 3419, 3058, 2935, 2309, 2183, 1291, 1249, 1186, 1108, 1035, 910, 699, 533 cm$^{-1}$.

$^1$H NMR (600 MHz, $CDCl_3$) $\delta$ 7.60 – 7.53 (m, 4H), 7.50 (dd, $J$ = 11.4, 8.2 Hz, 2H), 7.34 – 7.25 (m, 4H), 7.23 – 7.16 (m, 7H), 7.12 (dd, $J$ = 7.5, 7.4 Hz, 2H), 7.06 (d, $J$ = 6.9 Hz, 1H), 4.92 (d, $J$ = 14.9 Hz, 1H), 4.19 (d, $J$ = 14.9 Hz, 1H), 3.14 – 3.00 (m, 2H) ppm.

$^{13}$C{$^1$H} NMR (151 MHz, $CDCl_3$) $\delta$ 162.2, 144.5 (d, $J$ = 5.3 Hz), 133.5 (d, $J$ = 100.6 Hz), 133.4 (d, $J$ = 100.8 Hz), 131.4 (d, $J$ = 2.0 Hz), 131.4 (d, $J$ = 2.2 Hz), 130.6 (d, $J$ = 9.6 Hz) (2C), 130.5 (d, $J$ = 8.9 Hz) (2C), 128.5 (2C), 128.5 (d, $J$ = 12.0 Hz) (2C), 128.4 (d, $J$ = 11.9 Hz) (2C), 128.2 (2C), 128.1 (2C), 127.6, 127.4, 124.5 (2C), 86.7 (d, $J$ = 3.6 Hz), 67.5 (d, $J$ = 3.1 Hz), 41.5 (d, $J$ = 68.5 Hz) ppm.

$^{31}$P NMR (243 MHz, $CDCl_3$) $\delta$ 25.6 ppm.

HRMS (ESI) m/z: $[(M + K)]^+$ calcd for $C_{28}H_{24}KNO_2P$ 476.1176; Found 476.1186.



3ba

**Diphenyl((5-phenyl-2-(*p*-tolyl)-4,5-dihydrooxazol-5-yl)methyl)phosphine oxide (3ba): GP-4** was carried out with 4-methyl-*N*-(2-phenylallyl)benzamide **1b** (50.3 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ba** (72.2 mg, 80%), as a white solid; mp = 141-142 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1b**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ba**) = 0.40, UV detection].

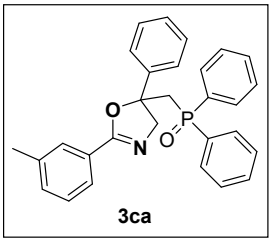
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3421, 3057, 2922, 2856, 1653, 1592, 1272, 1184, 996, 899, 617, 534 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.70 – 7.57 (m, 4H), 7.56 (d, $J$ = 8.3 Hz, 2H), 7.42 – 7.26 (m, 8H), 7.24 – 7.17 (m, 2H), 7.17 – 7.13 (m, 1H), 7.11 (d, $J$ = 7.9 Hz, 2H), 4.97 (d, $J$ = 14.9 Hz, 1H), 4.27 (d, $J$ = 14.9 Hz, 1H), 3.23 – 3.10 (m, 2H), 2.36 (s, 3H) ppm.

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) $\delta$ 162.4, 144.5 (d, $J$ = 5.5 Hz), 141.62, 133.6 (d, $J$ = 101.0 Hz), 133.5 (d, $J$ = 100.6 Hz), 131.4 (d, $J$ = 2.5 Hz), 131.4 (d, $J$ = 2.5 Hz), 130.7 (d, $J$ = 9.4 Hz) (2C), 130.6 (d, $J$ = 9.2 Hz) (2C), 128.7, 128.5, 128.5 (d, $J$ = 11.6 Hz) (2C), 128.4 (d, $J$ = 10.6 Hz) (2C), 128.2, 127.7, 124.7, 86.6 (d, $J$ = 3.7 Hz), 67.6 (d, $J$ = 3.1 Hz), 41.5 (d, $J$ = 68.7 Hz), 21.7 ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.5 ppm.

HRMS (ESI) m/z: $[(M + K)]^+$ calcd for $C_{29}H_{26}KNO_2P$ 490.1333; Found 490.1289.




**Diphenyl((5-phenyl-2-(*m*-tolyl)-4,5-dihydrooxazol-5-yl)methyl)phosphine oxide (3ca): GP-4** was carried out with 3-methyl-*N*-(2-phenylallyl)benzamide **1c** (50.3 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ca** (72.0 mg, 78%), as a white solid; mp = 141-142 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1c**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ca**) = 0.40, UV detection].

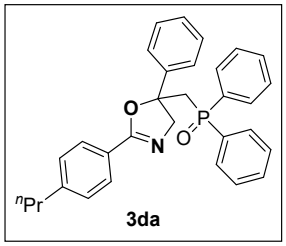
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3652, 3395, 2859, 2312, 1589, 1348, 1276, 1184, 1111, 1097, 986, 618, 524 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.73 (d, $J$ = 7.3 Hz, 2H), 7.69 – 7.64 (m, 2H), 7.63 – 7.57 (m, 2H), 7.46 – 7.42 (m, 1H), 7.39 (ddd, $J$ = 7.4, 7.5, 1.2 Hz, 1H), 7.36 – 7.27 (m, 7H), 7.18 (d, $J$ = 8.4 Hz, 1H), 7.14 – 7.10 (m, 2H), 6.96 (d, $J$ = 7.6 Hz, 1H), 5.02 (d, $J$ = 14.7 Hz, 1H), 4.31 (d, $J$ = 14.7 Hz, 1H), 3.52 – 2.87 (m, 2H), 2.24 (s, 3H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 162.5, 144.6, 137.7, 133.5 (d, $J$ = 100.7 Hz), 133.4 (d, $J$ = 101.3 Hz), 132.1, 131.5 (d, $J$ = 2.1 Hz), 131.4(d, $J$ = 1.8 Hz), 130.7 (d, $J$ = 9.6 Hz) (2C), 130.5 (d, $J$ = 9.5 Hz) (2C), 128.7, 128.6 (2C), 128.5 (d, $J$ = 12.0 Hz) (2C), 128.5 (d, $J$ = 11.7 Hz) (2C), 128.5, 128.1, 127.8, 125.4, 124.5 (2C), 86.6 (d, $J$ = 3.6 Hz), 67.6 (d, $J$ = 2.3 Hz), 41.4 (d, $J$ = 68.8 Hz), 21.3 ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 26.2 ppm.

HRMS (ESI) m/z: $[(M + H)]^+$ calcd for $C_{29}H_{27}NO_2P$ 452.1774; Found 452.1755.



3da

**Diphenyl((5-phenyl-2-(4-propylphenyl)-4,5-dihydrooxazol-5-yl)methyl)phosphine oxide (3da): GP-4** was carried out with *N*-(2-phenylallyl)-4-propylbenzamide **1d** (55.9 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3da** (71.0 mg, 74%), as a white solid; mp = 145-146 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1d**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3da**) = 0.40, UV detection].

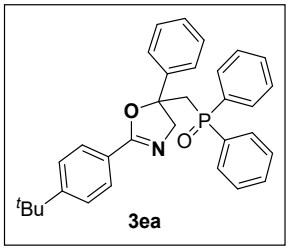
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3304, 2928, 2128, 1909, 1275, 1182, 1110, 1022, 985, 903, 620, 527 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.69 – 7.63 (m, 2H), 7.62 – 7.58 (m, 2H), 7.57 (d, $J$ = 8.1 Hz, 2H), 7.41 – 7.34 (m, 3H), 7.34 – 7.26 (m, 5H), 7.24 – 7.19 (m, 2H), 7.16 (d, $J$ = 7.3 Hz, 1H), 7.11 (d, $J$ = 8.2 Hz, 2H), 4.97 (d, $J$ = 14.8 Hz, 1H), 4.26 (d, $J$ = 14.8 Hz, 1H), 3.28 – 3.02 (m, 2H), 2.63 – 2.55 (m, 2H), 1.69 – 1.60 (m, 2H), 0.94 (t, $J$ = 7.3 Hz, 3H) ppm.

$^{13}C$ NMR (151 MHz, $CDCl_3$) $\delta$ 162.4, 146.4, 144.6 (d, $J$ = 5.7 Hz), 133.6 (d, $J$ = 100.6 Hz), 133.5 (d, $J$ = 101.4 Hz), 131.5 (d, $J$ = 2.5 Hz), 131.4 (d, $J$ = 2.3 Hz), 130.7 (d, $J$ = 9.6 Hz) (2C), 130.6 (d, $J$ = 8.8 Hz) (2C), 128.6 (2C), 128.5 (d, $J$ = 14.3 Hz) (2C), 128.5 (d, $J$ = 12.0 Hz) (2C), 128.3 (2C), 128.2 (2C), 127.7, 124.8, 124.6 (2C), 86.6 (d, $J$ = 3.6 Hz), 67.6 (d, $J$ = 3.2 Hz), 41.6(d, $J$ = 68.7 Hz), 38.1, 24.5, 13.8 ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.7 ppm.

HRMS (ESI) m/z: $[(M + H)]^+$ calcd for $C_{31}H_{31}NO_2P$ 480.2087; Found 480.2078.



3ea

**((2-(4-(*tert*-Butyl)phenyl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3ea): GP-4** was carried out with 4-(*tert*-butyl)-*N*-(2-phenylallyl)benzamide **1e** (58.7 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^{n}Bu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ea** (78.9 mg, 82%), as a white solid; mp = 145-146 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1e**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ea**) = 0.40, UV detection].

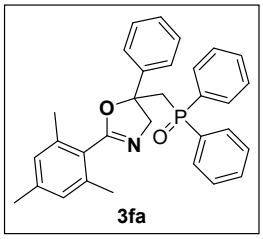
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3052, 2856, 1719, 1347, 1270, 1180, 1106, 1081, 985, 920, 697, 531 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.71 – 7.54 (m, 6H), 7.41 – 7.27 (m, 10H), 7.24 – 7.13 (m, 3H), 4.97 (d, $J$ = 14.8 Hz, 1H), 4.27 (d, $J$ = 14.8 Hz, 1H), 3.59 – 2.73 (m, 2H), 1.33 (d, $J$ = 6.4 Hz, 9H) ppm.

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) $\delta$ 162.3, 154.7, 144.5 (d, $J$ = 5.8 Hz), 133.3 (d, $J$ = 100.9 Hz), 133.2 (d, $J$ = 101.2 Hz), 131.4 (d, $J$ = 2.8 Hz), 131.4 (d, $J$ = 2.8 Hz), 130.6 (d, $J$ = 9.4 Hz) (2C), 130.4 (d, $J$ = 9.3 Hz) (2C), 128.5 (2C), 128.5 (d, $J$ = 11.6 Hz) (2C), 128.4 (d, $J$ = 12.5 Hz) (2C), 128.0 (2C), 127.7, 125.0 (2C), 124.5 (2C), 124.4, 86.5 (d, $J$ = 4.1 Hz), 67.5 (d, $J$ = 2.9 Hz), 41.4 (d, $J$ = 68.7 Hz), 34.9, 31.2 (3C) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.6 ppm.

HRMS (ESI) m/z: $[(M + Na)]^+$ calcd for $C_{32}H_{32}NNaO_2P$ 516.2063; Found 516.2034.



3fa

**((2-Mesityl-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3fa): GP-4** was carried out with 2,4,6-trimethyl-*N*-(2-phenylallyl)benzamide **1f** (58.7 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3fa** (69.0 mg, 74%), as a white solid; mp = 144-145 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1f**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3fa**) = 0.40, UV detection].

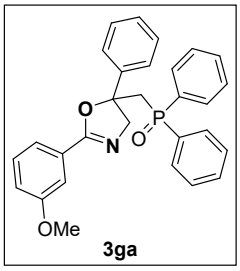
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3315, 2929, 2184, 2058, 1902, 1262, 1213, 1163, 1074, 700, 543 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.68 – 7.60 (m, 2H), 7.51 – 7.33 (m, 7H), 7.31 – 7.26 (m, 1H), 7.21– 7.14 (m, 2H), 7.09 – 6.97 (m, 3H), 6.82 (s, 2H), 4.94 (d, $J$ = 15.1 Hz, 1H), 4.55 (d, $J$ = 15.1 Hz, 1H), 3.23 – 3.08 (m, 2H), 2.27 (s, 3H), 2.19 (s, 6H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 162.9, 142.1 (d, $J$ = 1.5 Hz), 139.4, 137.2 (2C), 134.6 (d, $J$ = 100.7 Hz) (2C), 131.7 (d, $J$ = 2.7 Hz), 131.1 (d, $J$ = 2.9 Hz), 130.4 (d, $J$ = 9.3 Hz) (2C), 130.3 (d, $J$ = 9.3 Hz) (2C), 128.7 (d, $J$ = 11.8 Hz) (2C), 128.3 (2C), 128.2 (d, $J$ = 12.9 Hz) (2C), 128.0, 125.8 (2C), 87.2, 66.1, 42.3 (d, $J$ = 68.6 Hz), 21.3, 20.0 (2C) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.3 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{31}H_{34}N_2O_2P$ 497.2352; Found 497.2343.



3ga

**((2-(3-Methoxyphenyl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3ga): GP-4** was carried out with 3-methoxy-*N*-(2-phenylallyl)benzamide **1g** (53.5 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ga** (74.8 mg, 80%), as a white solid; mp = 147-148 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1g**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ga**) = 0.40, UV detection].

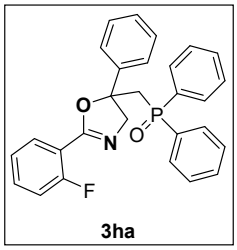
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3290, 2920, 2114, 1647, 1267, 1112, 1077, 902, 702, 541 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.70 – 7.63 (m, 2H), 7.63 – 7.56 (m, 2H), 7.43 – 7.26 (m, 9H), 7.26 – 7.19 (m, 4H), 7.19 – 7.15 (m, 1H), 7.01 – 6.93 (m, 1H), 5.02 (d, $J$ = 14.9 Hz, 1H), 4.28 (d, $J$ = 14.9 Hz, 1H), 3.81 (s, 3H), 3.23 – 3.08 (m, 2H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 162.3, 159.3, 144.5 (d, $J$ = 6.0 Hz), 133.5 (d, $J$ = 100.6 Hz), 133.4 (d, $J$ = 100.9 Hz), 131.5 (d, $J$ = 2.3 Hz), 131.5 (d, $J$ = 2.4 Hz), 130.7 (d, $J$ = 9.6 Hz) (2C), 130.5 (d, $J$ = 8.9 Hz) (2C), 129.2, 128.6 (3C), 128.54 (d, $J$ = 12.0 Hz) (2C), 128.52 (d, $J$ = 12.0 Hz) (2C), 127.9, 124.6 (2C), 120.8, 118.1, 112.6, 86.9 (d, $J$ = 3.5 Hz), 67.3, 55.5, 41.5 (d, $J$ = 68.7 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.7 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{29}H_{30}N_2O_3P$ 485.1989; Found 485.2009.




3ha

**((2-(2-Fluorophenyl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3ha): GP-4** was carried out with 2-fluoro-*N*-(2-phenylallyl)benzamide **1h** (51.0 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ha** (68.3 mg, 75%), as a white solid; mp = 137-138 ℃. [TLC (DCM/MeOH) 98:02, $R_f$(**1h**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ha**) = 0.40, UV detection].

IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3051, 2868, 1660, 1598, 1506, 1263, 1190, 1037, 701, 532 $cm^{-1}$.

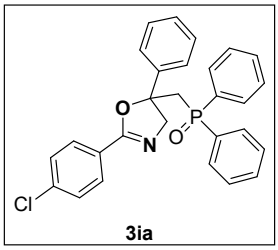
$^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.59 – 7.52 (m, 2H), 7.51 – 7.41 (m, 3H), 7.31 – 7.24 (m, 4H), 7.22 – 7.15 (m, 5H), 7.11 – 7.05 (m, 2H), 7.05 – 6.99 (m, 1H), 6.97 – 6.91 (m, 2H), 4.95 (d, $J$ = 15.2 Hz, 1H), 4.22 (d, $J$ = 15.2 Hz, 1H), 3.11 – 2.91 (m, 2H) ppm.

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) $\delta$ 161.2 (d, $J$ = 258.8 Hz), 158.9 (d, $J$ = 5.4 Hz), 144.4 (d, $J$ = 5.6 Hz), 133.4 (d, $J$ = 100.7 Hz), 133.2 (d, $J$ = 100.7 Hz), 132.8 (d, $J$ = 8.6 Hz), 131.5 (d, $J$ = 2.3 Hz), 131.4 (d, $J$ = 2.3 Hz), 131.2, 130.7 (d, $J$ = 9.6 Hz) (2C), 130.5 (d, $J$ = 9.5 Hz) (2C), 128.6, 128.5 (d, $J$ = 11.8 Hz) (2C), 128.4 (d, $J$ = 12.0 Hz) (2C), 127.8, 124.7, 123.7 (d, $J$ = 3.4 Hz), 116.5 (d, $J$ = 21.9 Hz), 115.6 (d, $J$ = 10.4 Hz), 86.5 (d, $J$ = 3.5 Hz), 67.4 (d, $J$ = 1.9 Hz), 41.6 (d, $J$ = 68.3 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.7 ppm.

$^{19}F$ NMR (565 MHz, $CDCl_3$) $\delta$ -108.9 ppm.

HRMS (ESI) m/z: $[(M + K)]^+$ calcd for $C_{28}H_{23}FKNO_2P$ 494.1082; Found 494.1090.



3ia

**((2-(4-Chlorophenyl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3ia): GP-4** was carried out with 4-chloro-*N*-(2-phenylallyl)benzamide **1i** (54.3 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ia** (80.2 mg, 85%), as a white solid; mp = 166-167 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1i**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ia**) = 0.40, UV detection].

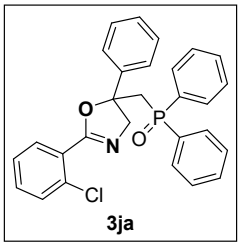
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3364, 3057, 2335, 1274, 1185, 1025, 982, 900, 816, 630, 533 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.64 – 7.51 (m, 4H), 7.51 – 7.46 (m, 2H), 7.33 (dd, $J$ = 7.4, 1.4 Hz, 1H), 7.30 – 7.06 (m, 12H), 4.96 (d, $J$ = 15.0 Hz, 1H), 4.19 (d, $J$ = 15.0 Hz, 1H), 3.20 – 2.97 (m, 2H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 161.4, 144.7 (d, $J$ = 6.0 Hz), 137.5, 133.7 (d, $J$ = 101.0 Hz), 133.4 (d, $J$ = 100.8 Hz), 131.6 (d, $J$ = 2.2 Hz), 131.5 (d, $J$ = 2.4 Hz), 130.8 (d, $J$ = 9.0 Hz) (2C), 130.5 (d, $J$ = 9.3 Hz) (2C), 129.6 (2C), 128.7 (2C), 128.6 (d, $J$ = 12.0 Hz) (2C), 128.6 (d, $J$ = 11.8 Hz) (2C), 128.4 (2C), 127.9 (2C), 124.5 (2C), 87.1 (d, $J$ = 3.8 Hz), 67.7, 41.5 (d, $J$ = 68.3 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 26.1 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{28}H_{27}ClN_2O_2P$ 489.1493; Found 489.1524.



3ja

**((2-(2-Chlorophenyl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3ja): GP-4** was carried out with 2-chloro-*N*-(2-phenylallyl)benzamide **1j** (54.3 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ja** (74.6 mg, 79%), as a white solid; mp = 168-169 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1j**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ja**) = 0.40, UV detection].

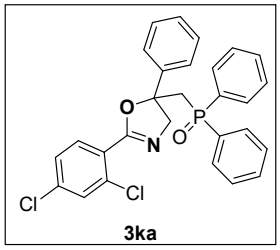
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3307, 2295, 1922, 1279, 1165, 1092, 1030, 988, 898, 817, 745, 583 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.69 – 7.64 (m, 2H), 7.61 – 7.52 (m, 3H), 7.44 – 7.27 (m, 10H), 7.23 – 7.11 (m, 4H), 5.04 (d, $J$ = 15.1 Hz, 1H), 4.36 (d, $J$ = 15.1 Hz, 1H), 3.26 – 3.06 (m, 2H) ppm.

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) $\delta$ 160.7, 144.0 (d, $J$ = 4.9 Hz), 133.6, 133.5 (d, $J$ = 101.5 Hz), 133.3 (d, $J$ = 100.7 Hz), 131.9, 131.6, 131.5 (d, $J$ = 2.5 Hz), 131.4 (d, $J$ = 2.8 Hz), 130.8, 130.7 (d, $J$ = 9.6 Hz) (2C), 130.5 (d, $J$ = 9.5 Hz) (2C), 128.5 (2C), 128.5 (d, $J$ = 12.6 Hz) (2C), 128.5 (d, $J$ = 11.8 Hz) (2C), 127.9, 126.7, 126.4, 125.0 (2C), 87.2 (d, $J$ = 2.6 Hz), 67.4 (d, $J$ = 2.2 Hz), 41.7 (d, $J$ = 68.8 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.7 ppm.

HRMS (ESI) m/z: $[(M + Na)]^+$ calcd for $C_{28}H_{23}ClNNaO_2P$ 494.1047; Found 494.1064.



**3ka**

**((2-(2,4-Dichlorophenyl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3ka): GP-4** was carried out with 2,4-dichloro-*N*-(2-phenylallyl)benzamide **1k** (61.2 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^{n}Bu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ka** (81.0 mg, 80%), as a white solid; mp = 165-165 ℃. [TLC (DCM/MeOH) 98:02, $R_f$(**1k**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ka**) = 0.40, UV detection].

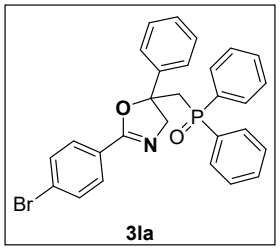
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3293, 2923, 1891, 1353, 1269, 1178, 1106, 1012, 904, 823, 700, 531 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.60 (dd, $J$ = 10.6, 8.5 Hz, 2H), 7.51 (dd, $J$ = 10.5, 8.5 Hz, 2H), 7.45 (dd, $J$ = 8.4, 1.8 Hz, 1H), 7.37 – 7.21 (m, 9H), 7.20 (d, $J$ = 2.0 Hz, 1H), 7.18 – 7.09 (m, 3H), 4.98 (dd, $J$ = 15.2, 1.5 Hz, 1H), 4.28 (dd, $J$ = 15.2, 1.4 Hz, 1H), 3.26 – 2.89 (m, 2H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 159.9, 144.2 (d, $J$ = 5.3 Hz), 137.1, 134.5, 133.4 (d, $J$ = 100.4 Hz), 133.4 (d, $J$ = 100.5 Hz), 131.5 (d, $J$ = 2.4 Hz) (2C), 130.7 (d, $J$ = 10.3 Hz) (2C), 130.7, 130.5 (d, $J$ = 8.9 Hz) (2C), 128.4 (d, $J$ = 11.7 Hz) (2C), 128.5 (d, $J$ = 12.1 Hz) (2C), 128.0, 126.8, 125.2, 124.8, 87.3, 67.5, 41.7 (d, J = 68.2 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.6 ppm.

HRMS (ESI) m/z: $[(M + H)]^+$ calcd for $C_{28}H_{23}Cl_2NO_2P$ 506.0838; Found 506.0818.



3la

**((2-(4-Bromophenyl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3la): GP-4** was carried out with 4-bromo-*N*-(2-phenylallyl)benzamide **1l** (63.2 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3la** (84.7 mg, 82%), as a white solid; mp = 169-170 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1l**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3la**) = 0.40, UV detection].

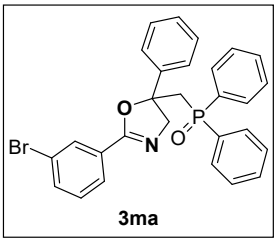
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3294, 2955, 2349, 1657, 1375, 1228, 1160, 1111, 1061, 952, 834, 524 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.62 – 7.51 (m, 4H), 7.43 – 7.39 (m, 2H), 7.37 – 7.31 (m, 3H), 7.29 – 7.10 (m, 10H), 4.50 (d, $J$ = 15.0 Hz, 1H), 4.26 (d, $J$ = 15.0 Hz, 1H), 3.20 – 2.16 (m, 2H) ppm.

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) $\delta$ 161.6, 144.6 (d, $J$ = 6.4 Hz), 133.5 (d, $J$ = 100.8 Hz), 133.1 (d, $J$ = 102.1 Hz), 131.7 (d, $J$ = 2.2 Hz), 131.6 (d, $J$ = 2.2 Hz), 131.4 (2C), 130.8 (d, $J$ = 9.6 Hz) (2C), 130.5 (d, $J$ = 8.9 Hz) (2C), 129.8 (2C), 128.7 (2C), 128.7 (d, $J$ = 11.5 Hz) (2C), 128.6 (d, $J$ = 11.4 Hz) (2C), 127.9, 126.3, 126.0, 124.4 (2C), 87.05 (d, $J$ = 4.5 Hz), 67.7 (d, $J$ = 2.0 Hz), 41.4 (d, $J$ = 68.6 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.8 ppm.

HRMS (ESI) m/z: $[(M + H)]^+$ calcd for $C_{28}H_{24}Br^{79}NO_2P$ 516.0723; Found 516.0679.; $[(M + H)]^+$ calcd for $C_{28}H_{24}Br^{81}NO_2P$ 518.0702; Found 518.0682.



3ma

**((2-(3-Bromophenyl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3ma): GP-4** was carried out with 3-bromo-*N*-(2-phenylallyl)benzamide **1m** (63.2 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^{n}Bu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ma** (82.6 mg, 80%), as a white solid; mp = 171-172 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1m**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ma**) = 0.40, UV detection].

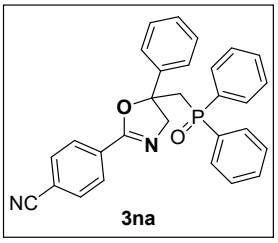
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3302, 2928, 2320, 1910, 1272, 1184, 1108, 1012, 900, 826, 698, 532 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.71 – 7.65 (m, 3H), 7.65 – 7.59 (m, 3H), 7.54 (ddd, $J$ = 8.0, 2.0, 1.0 Hz, 1H), 7.46 – 7.39 (m, 1H), 7.39 – 7.33 (m, 5H), 7.33 –7.27 (m, 2H), 7.24 – 7.14 (m, 2H), 5.10 (d, $J$ = 15.0 Hz, 1H), 4.27 (d, $J$ = 15.0 Hz, 1H), 3.21 – 3.09 (m, 2H) ppm.

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) $\delta$ 160.9, 144.8 (d, $J$ = 6.7 Hz), 134.2, 133.8 (d, $J$ = 109.2 Hz), 133.2 (d, $J$ = 101.0 Hz), 131.7 (d, $J$ = 2.9 Hz), 131.6 (d, $J$ = 3.0 Hz), 131.0, 130.7 (d, $J$ = 9.4 Hz) (2C), 130.4 (d, $J$ = 9.2 Hz) (2C), 129.7, 129.3, 128.7 (2C), 128.6 (d, $J$ = 12.0 Hz) (2C), 128.5 (d, $J$ = 12.1 Hz) (2C), 127.9, 126.9, 124.4 (2C), 122.1, 87.1 (d, $J$ = 4.3 Hz), 67.4 (d, $J$ = 2.4 Hz), 41.4 (d, $J$ = 68.3 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.3 ppm.

HRMS (ESI) m/z: $[(M + H)]^+$ calcd for $C_{28}H_{24}Br^{79}NO_2P$ 516.0723; Found 516.0694.; $[(M + H)]^+$ calcd for $C_{28}H_{24}Br^{81}NO_2P$ 518.0702; Found 518.0667.



3na

**4-(5-((Diphenylphosphoryl)methyl)-5-phenyl-4,5-dihydrooxazol-2-yl)benzonitrile (3na):** **GP-4** was carried out with 4-cyano-*N*-(2-phenylallyl)benzamide **1n** (52.5 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^{n}Bu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3na** (65.7 mg, 71%), as a white solid; mp = 164-165 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1n**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3na**) = 0.40, UV detection].

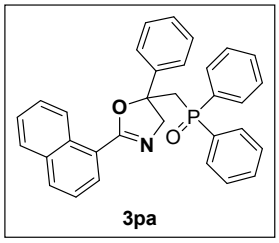
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3392, 2344, 2298, 1729, 1273, 1187, 1107, 1080, 1012, 976, 902, 826, 532 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.70 (d, $J$ = 8.2 Hz, 2H), 7.69 – 7.64 (m, 2H), 7.64 – 7.59 (m, 2H), 7.58 (d, $J$ = 8.2 Hz, 2H), 7.47 – 7.39 (m, 1H), 7.36 – 7.33 (m, 4H), 7.31 – 7.26 (m, 5H), 7.23 – 7.17 (m, 1H), 5.10 (d, $J$ = 15.3 Hz, 1H), 4.30 (d, $J$ = 15.3 Hz, 1H), 3.25 – 3.03 (m, 2H) ppm.

$^{13}C$ NMR (151 MHz, $CDCl_3$) $\delta$ 160.5, 144.4 (d, $J$ = 6.9 Hz), 133.4 (d, $J$ = 100.7 Hz), 132.9 (d, $J$ = 101.1 Hz), 131.8 (2C), 131.6 (d, $J$ = 2.0 Hz), 131.4 (d, $J$ = 2.2 Hz), 131.3, 130.6 (d, $J$ = 9.5 Hz) (2C), 130.2 (d, $J$ = 9.5 Hz) (2C), 128.7 (2C), 128.6 (2C), 128.6 (d, $J$ = 11.3 Hz) (2C), 128.5 (d, $J$ = 12.0 Hz) (2C), 127.9, 124.2 (2C), 118.3, 114.4, 87.3 (d, $J$ = 4.5 Hz), 67.5, 41.2 (d, $J$ = 68.1 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.8 ppm.

HRMS (ESI) m/z: $[(M + H)]^+$ calcd for $C_{29}H_{24}N_2O_2P$ 463.1570; Found 463.1583.



3pa

**((2-(Naphthalen-1-yl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3pa): GP-4** was carried out with *N*-(2-phenylallyl)-1-naphthamide **1p** (57.5 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^{n}Bu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3pa** (70.2 mg, 72%), as a white solid; mp = 155-156 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1p**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3pa**) = 0.40, UV detection].

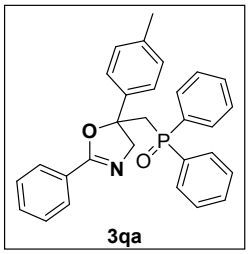
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3393, 2858, 2343, 1601, 1346, 1225, 1185, 1108, 979, 732, 532 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 9.23 (d, $J$ = 8.6 Hz, 1H), 7.90 (d, $J$ = 8.2 Hz, 1H), 7.84 (d, $J$ = 8.0 Hz, 1H), 7.72 – 7.54 (m, 6H), 7.53 – 7.48 (m, 1H), 7.47 – 7.42 (m, 2H), 7.40 – 7.27 (m, 4H), 7.27 – 7.23 (m, 3H), 7.22 – 7.14 (m, 3H), 5.18 (d, $J$ = 14.9 Hz, 1H), 4.47 (d, $J$ = 15.0 Hz, 1H), 3.35 – 3.08 (m, 2H) ppm.

$^{13}C$ NMR (151 MHz, $CDCl_3$) $\delta$ 161.9, 144.7 (d, $J$ = 5.8 Hz), 133.7, 133.6 (d, $J$ = 100.7 Hz), 133.4 (d, $J$ = 100.3 Hz), 132.0, 131.49 (d, $J$ = 2.2 Hz), 131.4 (d, $J$ = 2.8 Hz), 131.3, 130.8 (d, $J$ = 9.6 Hz) (2C), 130.6 (d, $J$ = 9.5 Hz) (2C), 129.2, 128.7 (2C), 128.5 (d, $J$ = 11.6 Hz) (2C), 128.6 (d, $J$ = 11.8 Hz) (2C), 128.4, 127.8, 127.5, 126.8, 126.1, 124.8 (2C), 124.5, 123.8, 85.4 (d, $J$ = 4.0 Hz), 68.4 (d, $J$ = 2.3 Hz), 41.6 (d, $J$ = 68.4 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.9 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{32}H_{30}N_2O_2P$ 505.2039; Found 505.2044.



3qa

**Diphenyl((2-phenyl-5-(*p*-tolyl)-4,5-dihydrooxazol-5-yl)methyl)phosphine oxide (3qa): GP-4** was carried out with *N*-(2-(*p*-tolyl)allyl)benzamide **1q** (50.2 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^{n}Bu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3qa** (77.6 mg, 86%), as a white solid; mp = 143-144 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1q**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3qa**) = 0.40, UV detection].

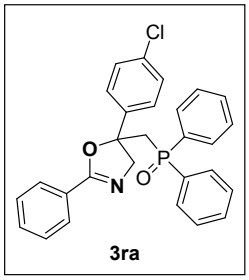
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3239, 2856, 2341, 1657, 1266, 1179, 1106, 1036, 970, 821, 531 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.70 – 7.62 (m, 4H), 7.61 – 7.56 (m, 2H), 7.42 – 7.37 (m, 2H), 7.35 – 7.26 (m, 7H), 7.26 – 7.22 (m, 2H), 7.01 (d, $J$ = 7.9 Hz, 2H), 4.97 (d, $J$ = 14.8 Hz, 1H), 4.27 (d, $J$ = 14.8 Hz, 1H), 3.22 – 3.06 (m, 2H), 2.25 (s, 3H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 162.3, 141.2 (d, $J$ = 5.1 Hz), 137.5, 133.5 (d, $J$ = 100.6 Hz), 133.4 (d, $J$ = 101.1 Hz), 131.4 (d, $J$ = 2.2 Hz), 131.3 (d, $J$ = 2.2 Hz), 131.2, 130.7 (d, $J$ = 9.4 Hz) (2C), 130.5 (d, $J$ = 9.0 Hz) (2C), 129.2 (2C), 128.5 (d, $J$ = 11.8 Hz) (2C), 128.5 (d, $J$ = 11.4 Hz) (2C), 128.2 (2C), 128.1 (2C), 127.4, 124.6 (2C), 86.7 (d, $J$ = 3.5 Hz), 67.4 (d, $J$ = 2.3 Hz), 41.7 (d, $J$ = 68.9 Hz), 21.1 ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.7 ppm.

HRMS (ESI) m/z: $[(M)]^+$ calcd for $C_{29}H_{26}NO_2P$ 451.1696; Found 451.1691.



3ra

**((5-(4-Chlorophenyl)-2-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3ra): GP-4** was carried out with *N*-(2-(4-chlorophenyl)allyl)benzamide **1r** (63.2 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ra** (71.7 mg, 76%), as a white solid; mp = 162-163 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1r**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ra**) = 0.40, UV detection].

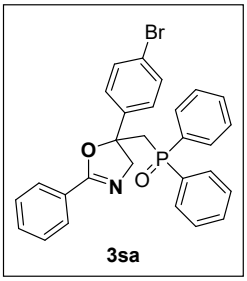
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3257, 2856, 2341, 1656, 1343, 1268, 1175, 1105, 970, 695, 531 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.73 – 7.53 (m, 6H), 7.46 – 7.39 (m, 2H), 7.38 – 7.27 (m, 9H), 7.17 – 7.09 (m, 2H), 4.92 (d, $J$ = 15.0 Hz, 1H), 4.27 (d, $J$ = 15.0 Hz, 1H), 3.15 (d, $J$ = 10.7 Hz, 2H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 162.3, 142.3 (d, $J$ = 4.2 Hz), 133.8, 133.5 (d, $J$ = 101.1 Hz), 133.0 (d, $J$ = 101.3 Hz), 131.6 (d, $J$ = 2.9 Hz), 131.5 (d, $J$ = 2.3 Hz), 131.5, 130.7 (2C), 130.6 (d, $J$ = 9.2 Hz) (4C), 128.6 (2C), 128.6 (d, $J$ = 11.9 Hz) (2C), 128.6 (d, $J$ = 11.9 Hz) (2C), 128.3 (2C), 127.3, 126.5 (2C), 86.6 (d, $J$ = 2.6 Hz), 67.6 (d, $J$ = 3.2 Hz), 41.7 (d, $J$ = 68.9 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.7 ppm.

HRMS (ESI) m/z: $[(M + H)]^+$ calcd for $C_{28}H_{24}ClNO_2P$ 472.1228; Found 472.1216.



3sa

**((5-(4-Bromophenyl)-2-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3sa): GP-4** was carried out with *N*-(2-(4-bromophenyl)allyl)benzamide **1s** (63.2 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3sa** (80.5 mg, 78%), as a white solid; mp = 169-170 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1s**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3sa**) = 0.40, UV detection].

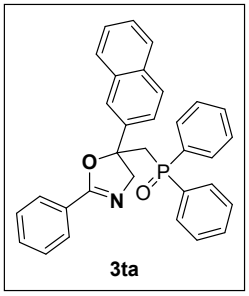
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3263, 2310, 1732, 1232, 1170, 1104, 1035, 970, 878, 820, 528 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.65 (d, $J$ = 7.3 Hz, 2H), 7.62 – 7.55 (m, 2H), 7.50 (dd, $J$ = 11.5, 7.4 Hz, 2H), 7.40 – 7.30 (m, 3H), 7.30 – 7.23 (m, 6H), 7.23 – 7.14 (m, 4H), 4.87 (d, $J$ = 14.9 Hz, 1H), 4.22 (d, $J$ = 14.9 Hz, 1H), 3.08 (d, $J$ = 10.6 Hz, 2H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 162.3, 142.8 (d, $J$ = 4.8 Hz), 133.5 (d, $J$ = 101.6 Hz), 133.0 (d, $J$ = 100.8 Hz), 131.6 (d, $J$ = 2.9 Hz) (2C), 131.6, 131.5 (d, $J$ = 4.0 Hz), 130.6 (d, $J$ = 9.4 Hz) (4C), 128.6 (d, $J$ = 11.7 Hz) (2C), 128.6 (d, $J$ = 11.5 Hz) (2C), 128.3 (2C), 128.2 (2C), 127.3, 126.8 (2C), 122.0, 86.6 (d, $J$ = 2.3 Hz), 67.6 (d, $J$ = 3.0 Hz), 41.7 (d, $J$ = 68.8 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.3 ppm.

HRMS (ESI) m/z: $[(M + H)]^+$ calcd for $C_{28}H_{24}Br^{79}NO_2P$ 516.0723; Found 516.0756.; $[(M + H)]^+$ calcd for $C_{28}H_{24}Br^{81}NO_2P$ 518.0702; Found 518.0729.




**((5-(Naphthalen-2-yl)-2-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3ta): GP-4** was carried out with *N*-(2-(naphthalen-2-yl)allyl)benzamide **1t** (57.5 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^{n}Bu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ta** (81.9 mg, 84%), as a white solid; mp = 156-157 ℃. [TLC (DCM/MeOH) 98:02, $R_f$(**1t**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ta**) = 0.40, UV detection].

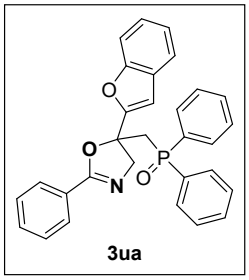
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3315, 2339, 2113, 1267, 1219, 1163, 1111, 1021, 814, 735, 697, 531 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.80 – 7.74 (m, 3H), 7.74 – 7.70 (m, 2H), 7.70 – 7.63 (m, 3H), 7.56 – 7.52 (m, 2H), 7.49 – 7.41 (m, 4H), 7.37 – 7.29 (m, 3H), 7.29 – 7.24 (m, 2H), 7.23 – 7.20 (m, 1H), 7.19 – 7.14 (m, 2H), 5.08 (d, $J$ = 14.9 Hz, 1H), 4.43 (d, $J$ = 14.9 Hz, 1H), 3.35 – 3.07 (m, 2H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 162.4, 141.2 (d, $J$ = 5.0 Hz), 133.4 (d, $J$ = 101.2 Hz), 133.0 (d, $J$ = 100.5 Hz), 132.8, 132.7, 131.4 (d, $J$ = 2.4 Hz), 131.3, 131.2 (d, $J$ = 2.2 Hz), 130.6 (d, $J$ = 9.5 Hz) (2C), 130.4 (d, $J$ = 9.4 Hz) (2C), 128.5 (d, $J$ = 11.6 Hz) (2C), 128.3 (d, $J$ = 11.5 Hz) (2C), 128.31, 128.3 (2C), 128.23, 128.0 (2C), 127.5, 127.4, 126.4, 126.3, 123.6, 122.9, 87.0 (d, $J$ = 3.4 Hz), 67.3 (d, $J$ = 1.9 Hz), 41.6 (d, $J$ = 68.5 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.7 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{32}H_{30}N_2O_2P$ 505.2039; Found 505.2085.



**3ua**

**((5-(Benzofuran-2-yl)-2-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3ua): GP-4** was carried out with *N*-(2-(benzofuran-2-yl)allyl)benzamide **1u** (55.5 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ua** (68.8 mg, 72%), as a white solid; mp = 156-157 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1u**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ua**) = 0.40, UV detection].

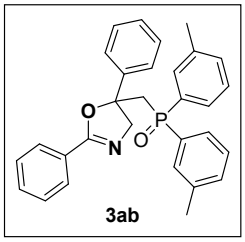
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3303, 2343, 1381, 1266, 1168, 1113, 1029, 985, 855, 815, 533 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.80 – 7.76 (m, 2H), 7.75 – 7.70 (m, 2H), 7.57 (ddd, $J$ = 11.7, 7.5, 1.7 Hz, 2H), 7.44 – 7.30 (m, 7H), 7.22 – 7.16 (m, 2H), 7.15 – 7.08 (m, 4H), 6.67 (s, 1H), 4.89 (d, $J$ = 15.6 Hz, 1H), 4.59 (d, $J$ = 15.6 Hz, 1H), 3.51 (dd, $J$ = 14.9, 9.9 Hz, 1H), 3.31 (dd, $J$ = 14.9, 12.0 Hz, 1H) ppm.

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) $\delta$ 155.14 (d, $J$ = 3.1 Hz), 154.8 (2C), 133.4 (d, $J$ = 101.7 Hz) (2C), 131.8 (d, $J$ = 2.6 Hz), 131.5, 131.2 (d, $J$ = 2.8 Hz), 130.6 (d, $J$ = 9.4 Hz) (2C), 130.5 (d, $J$ = 9.5 Hz) (2C), 128.7 (d, $J$ = 11.9 Hz) (2C), 128.3, 128.2 (4C), 128.0 (d, $J$ = 12.0 Hz) (2C), 127.4, 124.6, 122.9, 121.5, 111.4, 104.5, 77.4, 64.4 (d, $J$ = 5.1 Hz), 38.4 (d, $J$ = 68.7 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.0 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{30}H_{28}N_2O_3P$ 495.1832; Found 495.1826.




**((2,5-Diphenyl-4,5-dihydrooxazol-5-yl)methyl)di-*m*-tolylphosphine oxide (3ab): GP-4** was carried out with *N*-(2-phenylallyl)benzamide **1a** (47.5 mg, 0.2 mmol), di-*p*-tolylphosphine oxide **2b** (92.1 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ab** (70.8 mg, 76%), as a white solid; mp = 148-149 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1a**) = 0.80, $R_f$(**2b**) = 0.50, $R_f$(**3ab**) = 0.40, UV detection].

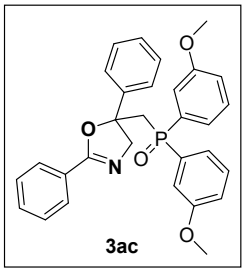
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3308, 2343, 1905, 1266, 1178, 1114, 1084, 1029, 985, 921, 855, 741, 533 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.70 – 7.62 (m, 2H), 7.51 (d, $J$ = 12.3 Hz, 1H), 7.45 – 7.35 (m, 6H), 7.33 – 7.27 (m, 2H), 7.25 – 7.05 (m, 2H), 5.02 (d, $J$ = 14.9 Hz, 1H), 4.26 (d, $J$ = 14.9 Hz, 1H), 3.25 – 3.05 (m, 2H), 2.27 (s, 3H), 2.18 (s, 3H) ppm.

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) $\delta$ 162.3, 144.8 (d, $J$ = 6.2 Hz), 138.5 (d, $J$ = 5.7 Hz), 138.3 (d, $J$ = 5.7 Hz), 133.4 (d, $J$ = 100.5 Hz), 133.1 (d, $J$ = 100.7 Hz), 132.3 (d, $J$ = 2.8 Hz), 132.2 (d, $J$ = 2.9 Hz), 131.3 (d, $J$ = 9.1 Hz), 131.3, 131.0 (d, $J$ = 9.0 Hz), 128.5 (2C), 128.4 (d, $J$ = 12.5 Hz), 128.3 (d, $J$ = 12.7 Hz), 128.2 (2C), 128.1 (2C), 127.7, 127.6 (d, $J$ = 10.0 Hz), 127.5 (d, $J$ = 11.9 Hz), 127.3, 124.5 (2C), 86.72 (d, $J$ = 4.1 Hz), 67.43 (d, $J$ = 2.9 Hz), 41.50 (d, $J$ = 68.2 Hz), 21.4, 21.3 ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.9 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{30}H_{32}N_2O_2P$ 483.2196; Found 483.2197.




**((2,5-Diphenyl-4,5-dihydrooxazol-5-yl)methyl)bis(3-methoxyphenyl)phosphine oxide (3ac):** **GP-4** was carried out with *N*-(2-phenylallyl)benzamide **1a** (47.5 mg, 0.2 mmol), bis(3-methoxyphenyl)phosphine oxide **2c** (104.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ac** (82.6 mg, 83%), as a white solid; mp = 162-163 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1a**) = 0.80, $R_f$(**2c**) = 0.50, $R_f$(**3ac**) = 0.40, UV detection].

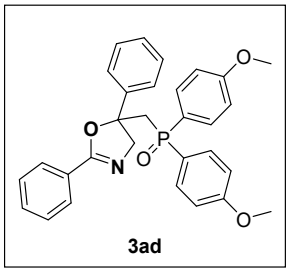
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3267, 2230, 1731, 1269, 1163, 1026, 917, 855, 809, 629, 528 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.71 – 7.63 (m, 2H), 7.45 – 7.39 (m, 1H), 7.38 – 7.35 (m, 2H), 7.35 – 7.28 (m, 2H), 7.27 – 7.13 (m, 9H), 6.95 – 6.89 (m, 1H), 6.87 – 6.79 (m, 1H), 5.03 (d, $J$ = 14.9 Hz, 1H), 4.27 (d, $J$ = 14.9 Hz, 1H), 3.75 (s, 3H), 3.62 (s, 3H), 3.26 – 3.08 (m, 2H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 162.3, 159.6 (d, $J$ = 8.3 Hz), 159.5 (d, $J$ = 8.2 Hz), 144.8 (d, $J$ = 6.3 Hz), 135.0 (d, $J$ = 100.2 Hz), 134.7 (d, $J$ = 100.6 Hz), 131.3, 129.8 (d, $J$ = 13.6 Hz), 129.7 (d, $J$ = 14.0 Hz), 128.6 (2C), 128.2 (2C), 128.1 (2C), 127.8, 127.4, 124.6 (2C), 122.7 (d, $J$ = 9.7 Hz), 122.5 (d, $J$ = 9.1 Hz), 117.7 (d, $J$ = 2.2 Hz) (2C), 115.7 (d, $J$ = 9.8 Hz), 115.54 (d, $J$ = 10.2 Hz), 86.7 (d, $J$ = 3.8 Hz), 67.6 (d, $J$ = 2.6 Hz), 55.5, 55.3, 41.7 (d, $J$ = 68.4 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.6 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{30}H_{32}N_2O_4P$ 515.2094; Found 515.2096.



3ad

**((2,5-Diphenyl-4,5-dihydrooxazol-5-yl)methyl)bis(4-methoxyphenyl)phosphine oxide (3ad):** **GP-4** was carried out with *N*-(2-phenylallyl)benzamide **1a** (47.5 mg, 0.2 mmol), bis(4-methoxyphenyl)phosphine oxide **2d** (104.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ad** (67.7 mg, 68%), as a white solid; mp = 165-166 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1a**) = 0.80, $R_f$(**2d**) = 0.50, $R_f$(**3ad**) = 0.40, UV detection].

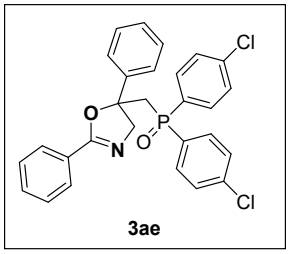
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3213, 2149, 1274, 1218, 1163, 1110, 1027, 967, 919, 853, 697, 532 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.68 (dd, $J$ = 8.3, 1.2 Hz, 2H), 7.60 – 7.54 (m, 2H), 7.54 – 7.49 (m, 2H), 7.45 – 7.40 (m, 1H), 7.39 – 7.34 (m, 2H), 7.33 – 7.29 (m, 2H), 7.29 – 7.23 (m, 2H), 7.21 – 7.17 (m, 1H), 6.85 – 6.81 (m, 2H), 6.77 – 6.71 (m, 2H), 5.06 (d, $J$ = 14.9 Hz, 1H), 4.24 (d, $J$ = 14.9 Hz, 1H), 3.78 (s, 3H), 3.68 (s, 3H), 3.13 – 3.02 (m, 2H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 162.3, 162.2 (d, $J$ = 2.9 Hz), 162.1 (d, $J$ = 3.0 Hz), 145.2 (d, $J$ = 6.9 Hz), 132.7 (d, $J$ = 10.9 Hz) (2C), 132.4 (d, $J$ = 10.6 Hz) (2C), 131.2, 128.7 (2C), 128.3 (2C), 128.1 (2C), 127.7, 127.5, 125.2 (d, $J$ = 107.6 Hz), 124.5 (2C), 124.4 (d, $J$ = 109.9 Hz), 114.1 (d, $J$ = 13.3 Hz) (2C), 114.0 (d, $J$ = 13.7 Hz (2C), 86.8 (d, $J$ = 3.7 Hz), 67.3 (d, $J$ = 2.8 Hz), 55.4, 55.2, 42.0 (d, $J$ = 69.3 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.8 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{30}H_{32}N_2O_4P$ 515.2094; Found 515.2082.



3ae

**Bis(4-chlorophenyl)((2,5-diphenyl-4,5-dihydrooxazol-5-yl)methyl)phosphine oxide (3ae): GP-4** was carried out with *N*-(2-phenylallyl)benzamide **1a** (47.5 mg, 0.2 mmol), bis(4-methoxyphenyl)phosphine oxide **2e** (104.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ae** (78.9 mg, 78%), as a white solid; mp = 170-171 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1a**) = 0.80, $R_f$(**2e**) = 0.50, $R_f$(**3ae**) = 0.40, UV detection].

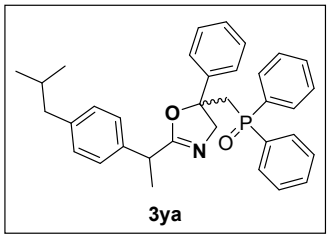
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3212, 2854, 2340, 1649, 1278, 1165, 1110, 1024, 969, 850, 749, 524 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.60 – 7.55 (m, 2H), 7.47 (ddd, $J$ = 26.3, 11.2, 8.5 Hz, 4H), 7.40 – 7.35 (m, 1H), 7.30 – 7.21 (m, 6H), 7.21 – 7.11 (m, 5H), 4.91 (d, $J$ = 14.9 Hz, 1H), 4.91 (d, $J$ = 14.9 Hz, 1H), 4.18 (d, $J$ = 14.9 Hz, 1H), 4.18 (d, $J$ = 14.9 Hz, 1H), 3.21 – 2.90 (m, 2H), 3.19 – 2.93 (m, 2H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 162.2, 144.4 (d, $J$ = 6.7 Hz), 138.4 (d, $J$ = 3.5 Hz), 138.4 (d, $J$ = 3.3 Hz), 132.0 (d, $J$ = 10.7 Hz) (2C), 131.8 (d, $J$ = 9.8 Hz) (2C), 131.7 (d, $J$ = 104.2 Hz), 131.6, 131.2 (d, $J$ = 104.0 Hz), 129.0 (d, $J$ = 12.3 Hz) (2C), 129.0 (d, $J$ = 12.8 Hz) (2C), 128.7 (2C), 128.26 (2C), 128.1 (2C), 127.9, 126.9, 124.4 (2C), 86.5 (d, $J$ = 4.5 Hz), 67.7 (d, $J$ = 3.1 Hz), 41.5 (d, $J$ = 69.7 Hz) ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 24.8 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{28}H_{26}Cl_2N_2O_2P$ 523.1103; Found 523.1097.



3ya

**((2-(1-(4-Isobutylphenyl)ethyl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3ya): GP-4** was carried out with 2-(4-isobutylphenyl)-*N*-(2-phenylallyl)propenamide **1v** (64.3 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3ya** (75.6 mg, 65%), as a white solid; mp = 149-150 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1y**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3ya**) = 0.40, UV detection]. The mixture of two inseparable diastereomers (1:0.25).

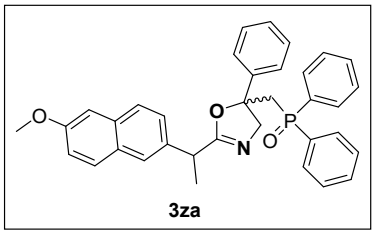
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3297, 3061, 2342, 1653, 1272, 1163, 1110, 1026, 969, 855, 745, 533 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 7.79 – 7.71 (m, 2H, major and minor), 7.70 – 7.56 (m, 3H, major and minor), 7.55 – 7.38 (m, 6H, major and minor), 7.37 – 7.26 (m, 3H, major and minor), 7.25 – 6.94 (m, 11H, major and minor), 6.90 (dd, *J* = 7.8, 1.5 Hz, 2H, major and minor), 4.84 (dd, *J* = 14.4, 1.5 Hz, 1H, major and minor), 4.03 (d, *J* = 14.4, 1.5 Hz, 1H, major and minor), 3.15 – 2.84 (m, 4H, major and minor), 2.44 (dd, *J* = 7.1, 3.1 Hz, 3H, major and minor), 1.90 – 1.78 (m, 1H, major and minor), 1.39 (d, *J* = 7.2 Hz, 2H, major and minor), 1.37 (d, *J* = 9.7 Hz, 1H, major and minor), 0.93 – 0.87 (m, 8H, major and minor) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 168.2, 168.2, 145.1 (d, *J* = 7.0 Hz) (2C), 140.4, 140.3, 138.6, 138.3, 133.8 (d, *J* = 100.5 Hz) (2C), 133.6 (d, *J* = 100.4 Hz) (2C), 131.6 (d, *J* = 2.5 Hz), 131.5 (d, *J* = 2.1 Hz), 131.2 (d, *J* = 3.0 Hz) (4C), 131.0 (d, *J* = 9.6 Hz) (2C), 130.5 (d, *J* = 9.3 Hz) (2C), 130.4 (d, *J* = 9.4 Hz) (4C), 129.2 (2C), 129.1 (2C), 128.5 (d, *J* = 11.8 Hz) (4C), 128.5 (d, *J* = 12.0 Hz) (4C), 128.3 (2C), 128.2 (2C), 127.4 (2C), 127.3 (2C), 124.9 (2C), 124.3 (2C), 86.8 (d, *J* = 2.0 Hz), 86.2 (d, *J* = 3.9 Hz), 67.0, 66.7, 45.1, 45.0, 41.4 (d, *J* = 68.4 Hz), 41.2 (d, *J* = 69.2 Hz), 39.1, 38.7, 30.3, 30.3, 22.5, 22.5, 22.4, 22.3, 18.7, 18.6 ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 26.5, 26.2 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{34}H_{40}N_2O_2P$ 539.2822; Found 539.2875.



3za

**(2-(1-(6-Methoxynaphthalen-2-yl)ethyl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3za): GP-4** was carried out with 2-(6-methoxynaphthalen-2-yl)-*N*-(2-phenylallyl)propenamide **1z** (69.1 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3za** (63.3 mg, 58%), as a white solid; mp = 172-173 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1z**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3za**) = 0.40, UV detection]. The mixture of two inseparable diastereomers (1:0.45).

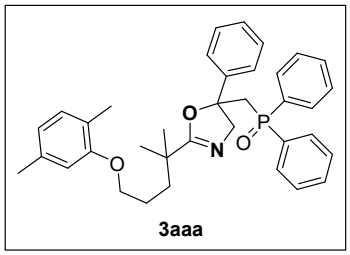
IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3292, 2923, 2062, 1654, 1269, 1185, 1084, 1011, 979, 837, 699, 617, 530 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.76 – 7.70 (m, 2H, major and minor), 7.7 (dd, $J$ = 8.6, 3.2 Hz, 1H major and minor), 7.65 – 7.60 (m, 1H, major and minor), 7.59 – 7.51 (m, 4H, major and minor), 7.49 – 7.43 (m, 3H, major and minor), 7.42 – 7.34 (m, 3H, major and minor), 7.34 – 7.29 (m, 3H, major and minor), 7.23 – 7.18 (m, 2H, major and minor), 7.16 – 7.08 (m, 4H major and minor), 7.07 – 7.00 (m, 2H, major and minor), 6.99 – 6.94 (m, 2H, major and minor), 6.93 – 6.89 (m, 2H, major and minor), 4.85 (dd, $J$ = 14.5, 1.7 Hz, 1H, major and minor), 4.16 (dd, $J$ = 14.4, 0.6 Hz, 1H, major and minor), 3.92 (s, 1H, minor), 3.92 (s, 3H, major), 3.23 (q, $J$ = 7.0 Hz, 1H, major and minor), 3.02 – 2.97 (m, 3H, major and minor), 1.48 (d, $J$ = 7.2 Hz, 3H, major and minor) ppm.

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) $\delta$ 168.2, 168.0, 157.6, 157.6, 144.8 (d, $J$ = 6.6 Hz) (2C), 136.6, 136.4, 133.8, 133.7 (d, $J$ = 99.8 Hz) (4C), 133.7, 131.61 (d, $J$ = 2.6 Hz), 131.5 (d, $J$ = 2.5 Hz) , 131.21 (d, $J$ = 2.3 Hz) (2C), 131.0 (d, $J$ = 9.4 Hz) (2C), 130.5 (d, $J$ = 9.0 Hz) (2C), 130.5 (d, $J$ = 9.4 Hz) (2C), 130.5 (d, $J$ = 9.2 Hz) (2C), 130.5 (d, $J$ = 9.2 Hz) (2C), 129.4, 129.3, 128.9 (2C), 128.6 (2C), 128.4 (d, $J$ = 11.8 Hz) (8C), 128.3, 128.2, 127.7, 127.5, 127.1, 127.0, 126.6, 126.5, 126.05, 126.01, 125.0 (2C), 124.4 (2C), 118.9, 118.9, 105.7, 105.6, 86.5 (d, $J$ = 4.1 Hz) (2C), 66.8, 66.6, 55.4, 55.4, 41.4 (d, $J$ = 68.4 Hz), 41.3 (d, $J$ = 68.2 Hz), 39.5, 39.2, 19.0, 18.9 ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.7, 25.4 ppm.

HRMS (ESI) m/z: $[(M + NH_4)]^+$ calcd for $C_{35}H_{36}N_2O_3P$ 563.2458; Found 563.2425.



3aaa

**((2-(5-(2,5-Dimethylphenoxy)-2-methylpentan-2-yl)-5-phenyl-4,5-dihydrooxazol-5-yl)methyl)diphenylphosphine oxide (3aaa): GP-4** was carried out with 5-(2,5-dimethylphenoxy)-2,2-dimethyl-*N*-(2-phenylallyl)pentanamide **1aa** (73.1 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), and $^nBu_4OAc$ (0.1 M) in 5 mL of $CH_3CN$/MeOH (4/1) at room temperature for 4 h. Purification of the crude material by silica gel column chromatography (DCM/MeOH: 99:01 to 98:02) furnished the product **3aaa** (67.9 mg, 60%), as a white solid; mp = 177-178 °C. [TLC (DCM/MeOH) 98:02, $R_f$(**1aa**) = 0.80, $R_f$(**2a**) = 0.50, $R_f$(**3aaa**) = 0.40, UV detection].

IR (MIR-ATR, 4000-400 $cm^{-1}$) $\nu_{max}$ = 3057, 2856, 2340, 1348, 1270, 1183, 1083, 906, 837, 731, 621, 530 $cm^{-1}$.

$^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 7.72 – 7.57 (m, 2H), 7.50 – 7.43 (m, 3H), 7.41 – 7.29 (m, 5H), 7.28 – 7.23 (m, 2H), 7.16 – 7.05 (m, 3H), 6.99 (d, $J$ = 7.5 Hz, 1H), 6.65 (d, $J$ = 7.5 Hz, 1H), 6.57 (s, 1H), 4.68 (d, $J$ = 14.4 Hz, 1H), 4.22 (d, $J$ = 14.4 Hz, 1H), 3.93 – 3.75 (m, 2H), 3.16 – 2.85 (m, 2H), 2.29 (s, 3H), 2.16 (s, 3H), 1.74 – 1.57 (m, 4H), 1.16 (s, 3H), 1.12 (s, 3H) ppm.

$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) $\delta$ 157.1 (2C), 143.8 (d, $J$ = 3.6 Hz), 136.5 (2C), 134.3 (d, $J$ = 101.3 Hz), 133.3 (d, $J$ = 100.6 Hz), 131.6 (d, $J$ = 1.8 Hz), 131.3 (d, $J$ = 2.1 Hz), 130.6 (d, $J$ = 9.1 Hz) (2C), 130.5 (d, $J$ = 9.1 Hz) (2C), 130.3, 128.6 (d, $J$ = 11.5 Hz) (2C), 128.4 (d, $J$ = 11.9 Hz) (2C), 128.3 (2C), 127.8, 125.0 (2C), 123.6, 120.7, 112.0, 86.6, 68.1, 66.5, 41.7 (d, $J$ = 68.9 Hz), 36.7 (d, $J$ = 96.7 Hz), 25.7, 25.4, 24.9 (2C), 21.5, 17.0 ppm.

$^{31}P$ NMR (243 MHz, $CDCl_3$) $\delta$ 25.9 ppm.

HRMS (ESI) m/z: $[(M + Na)]^+$ calcd for $C_{36}H_{40}NNaO_3P$ 588.2638; Found 588.2652.

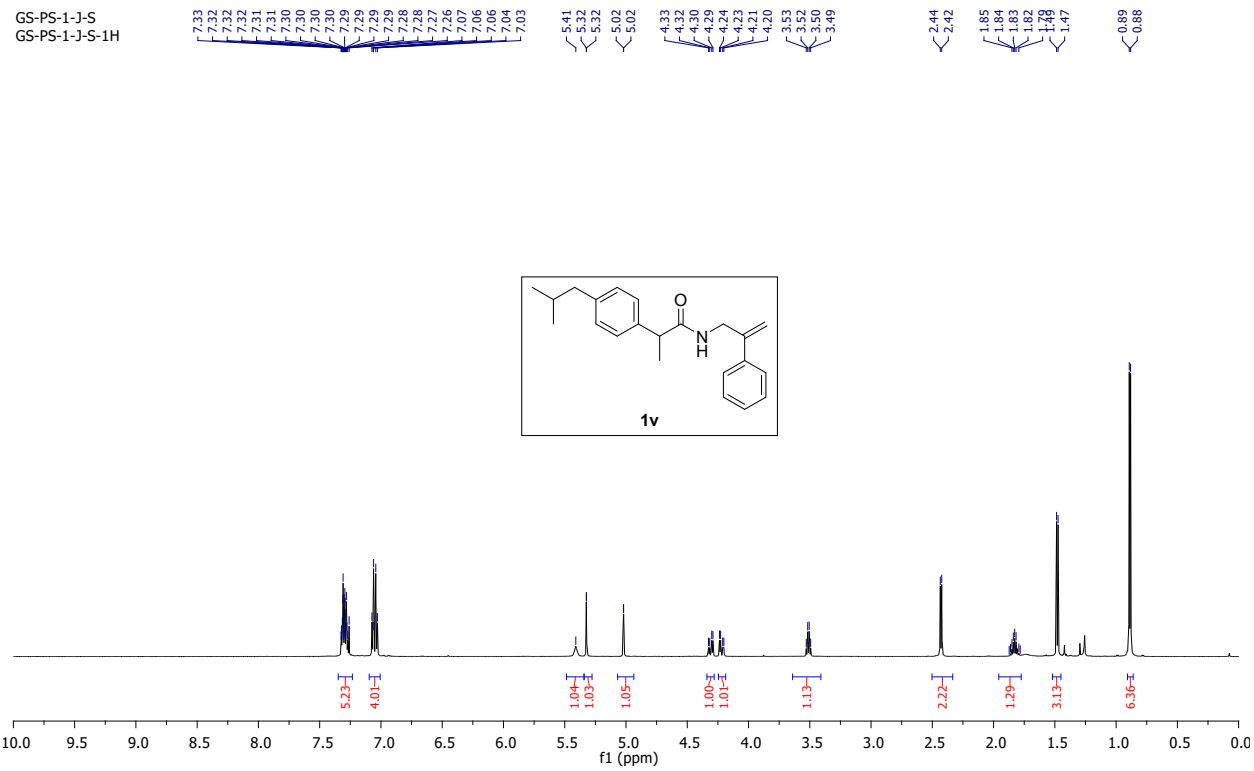
**Scale-up reaction:**

To an oven-dried ElectraSyn 2.0 undivided cell (10 mL) equipped with a magnetic stir bar, were added *N*-(2-phenylallyl)benzamide **1a** (0.5 g, 2.1 mmol), diphenylphosphine oxide **2a** (0.85 g, 4.2 mmol, 2 equiv), ferrocene (0.08 g, 0.42 mmol, 20 mol%), $Et_3N$ (0.21 g, 2.1 mmol, 1 equiv), $^nBu_4OAc$ (0.3 g, 0.1 M), and a mixture of $CH_3CN$ and MeOH (10 mL, v:v = 8:2). The ElectraSyn vial cap equipped with a platinum plate (5.2 cm × 0.8 cm × 0.2 cm) as the cathode and a graphite

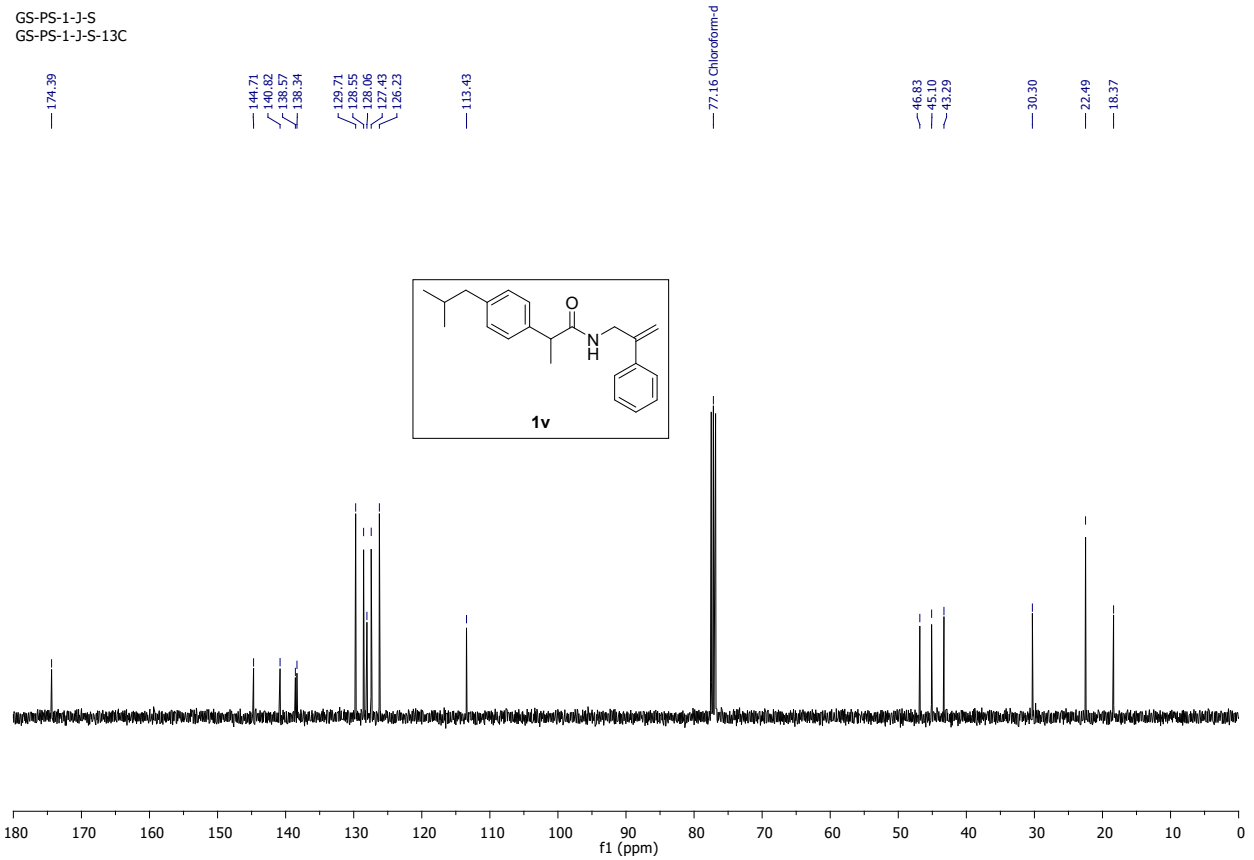
plate (5.2 cm × 0.8 cm × 0.2 cm) as the anode was inserted into the reaction mixture. The reaction mixture was stirred and electrolyzed at a constant current of 25 mA at room temperature for 12 h. After the reaction was complete, the reaction mixture was concentrated under reduced pressure. Purification of the crude product by column chromatography on silica gel using DCM/MeOH (DCM: MeOH= 99:01 to 98:02) furnished the product **3aa** (0.66 g, 72%), white solid.

**Control experiments:**

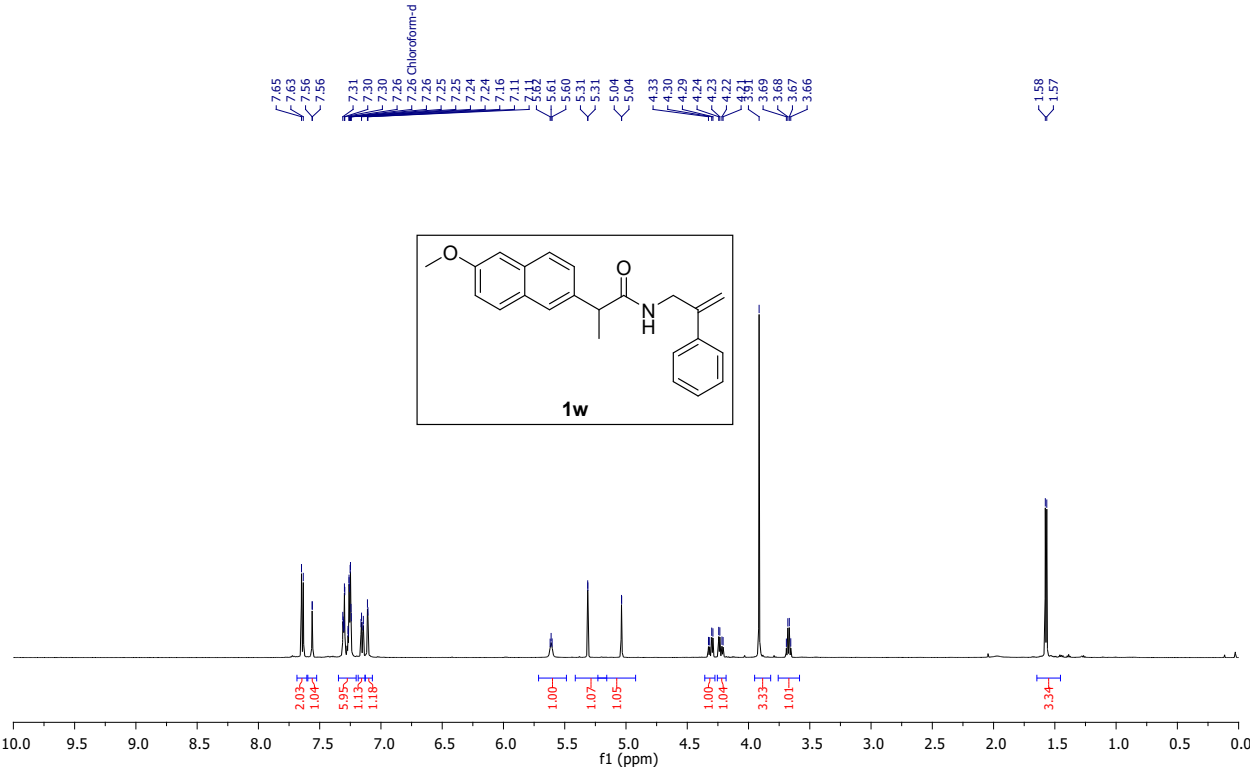
To an oven-dried ElectraSyn 2.0 undivided cell (6 mL) equipped with a magnetic stir bar, were added *N*-(2-phenylallyl)benzamide **1a** (47.5 mg, 0.2 mmol), diphenylphosphine oxide **2a** (80.9 mg, 0.4 mmol), ferrocene (7.4 mg, 0.04 mmol), $Et_3N$ (20.2 mg, 0.2 mmol), $^{n}Bu_4OAc$ (0.1 M), radical scavenger (TEMPO, butylated hydroxytoluene (BHT), or (1-cyclopropylvinyl)benzene) (0.4 mmol, 2 equiv) and a mixture of $CH_3CN$ and MeOH (5 mL, v:v = 4:1). The ElectraSyn vial cap equipped with a platinum plate (5.2 cm × 0.8 cm × 0.2 cm) as the cathode and a graphite plate (5.2 cm × 0.8 cm × 0.2 cm) as the anode was inserted into the reaction mixture. The reaction mixture was stirred and electrolyzed at a constant current of 5 mA at room temperature for 4 h. After the reaction was complete, the reaction mixture was concentrated under reduced pressure. The crude mixtures were subjected to mass spectrometric analysis to confirm the formation of adducts **4**, **5**, and **6**.



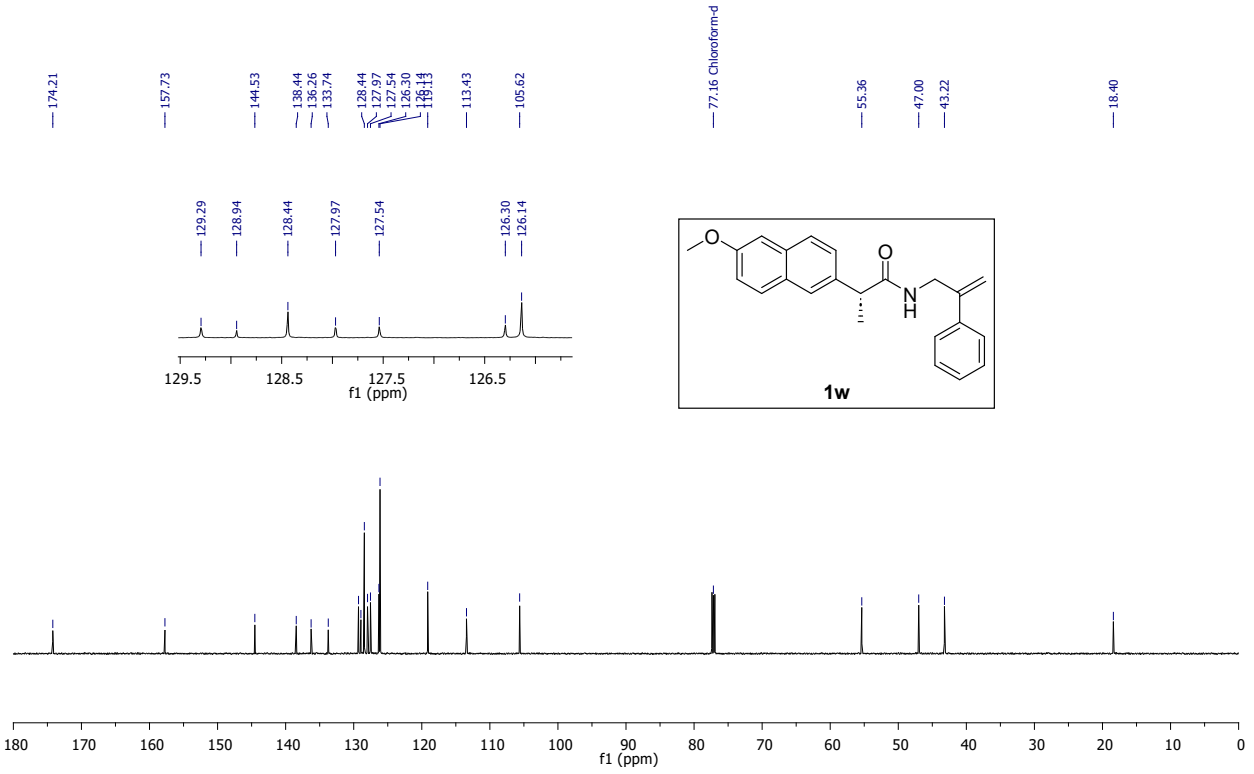

$^{1}$H NMR (600 MHz) spectrum of **1v** in $CDCl_3$



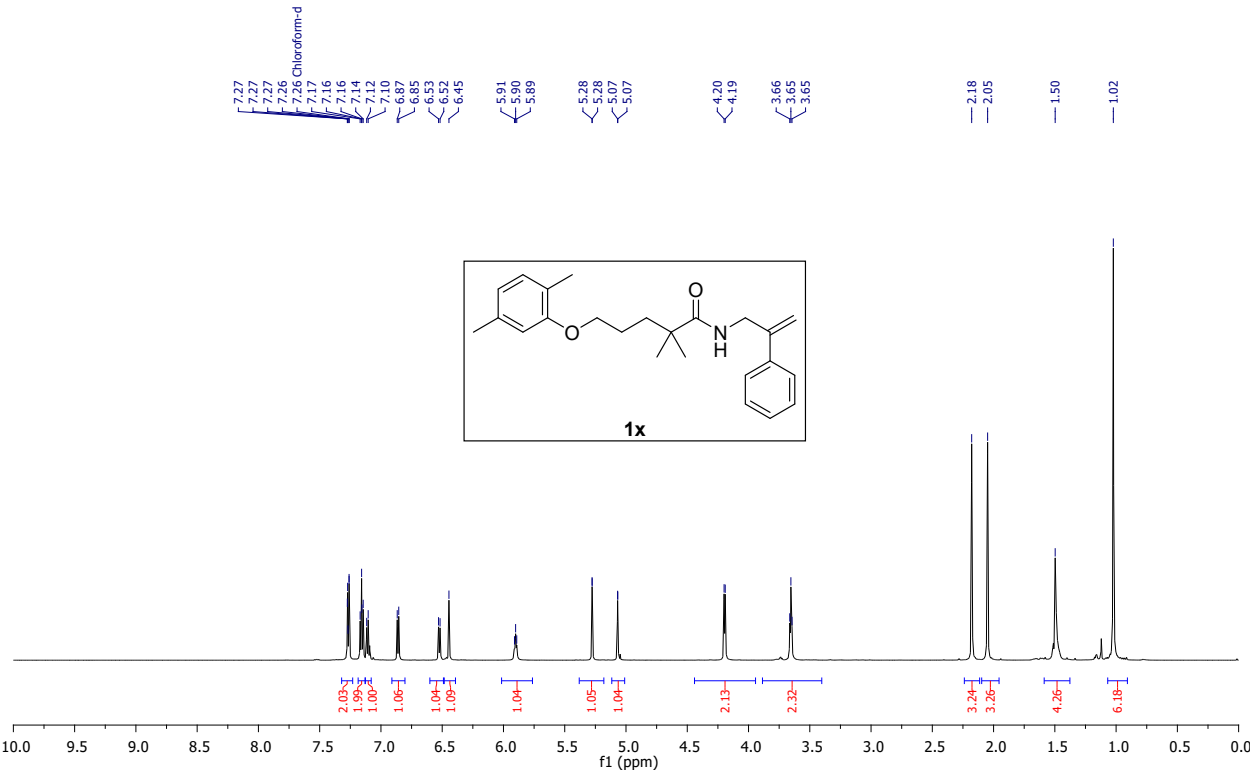

$^{13}C\{^{1}H\}$ NMR (101 MHz) spectrum of **1v** in $CDCl_3$



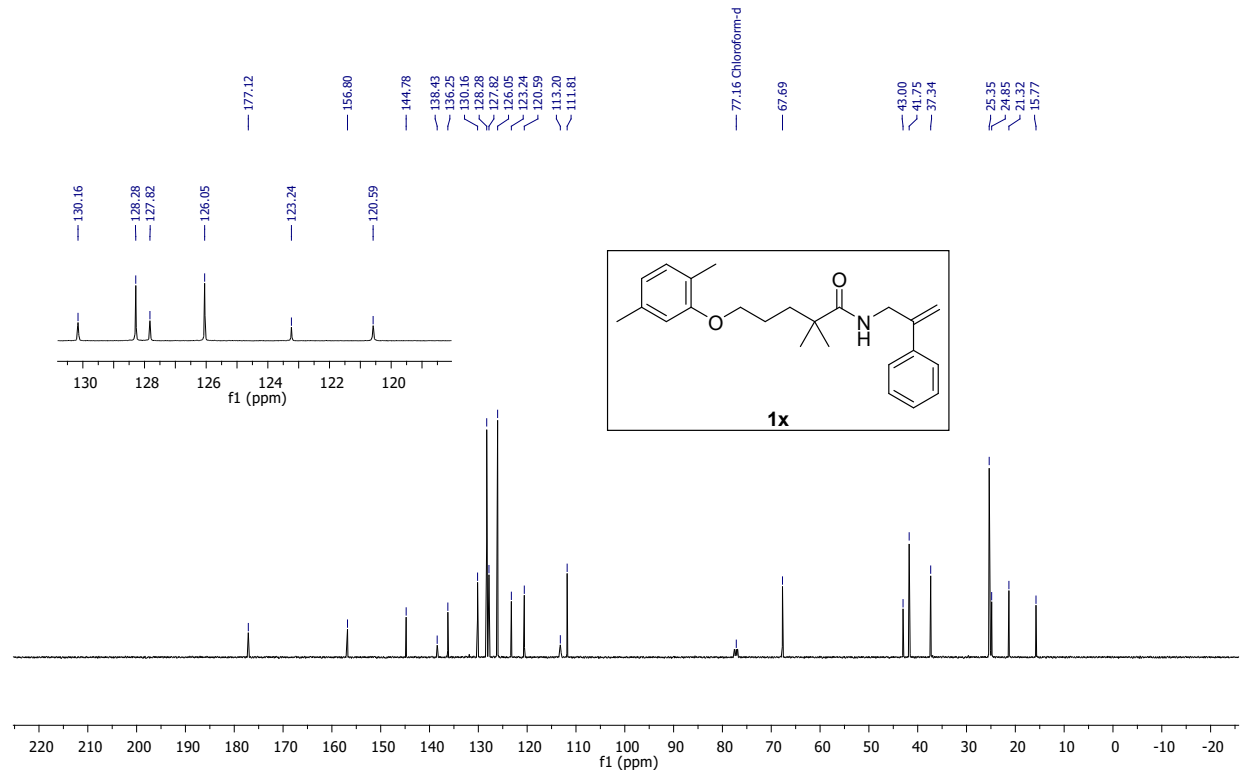

$^{1}$H NMR (600 MHz) spectrum of **1w** in $CDCl_3$



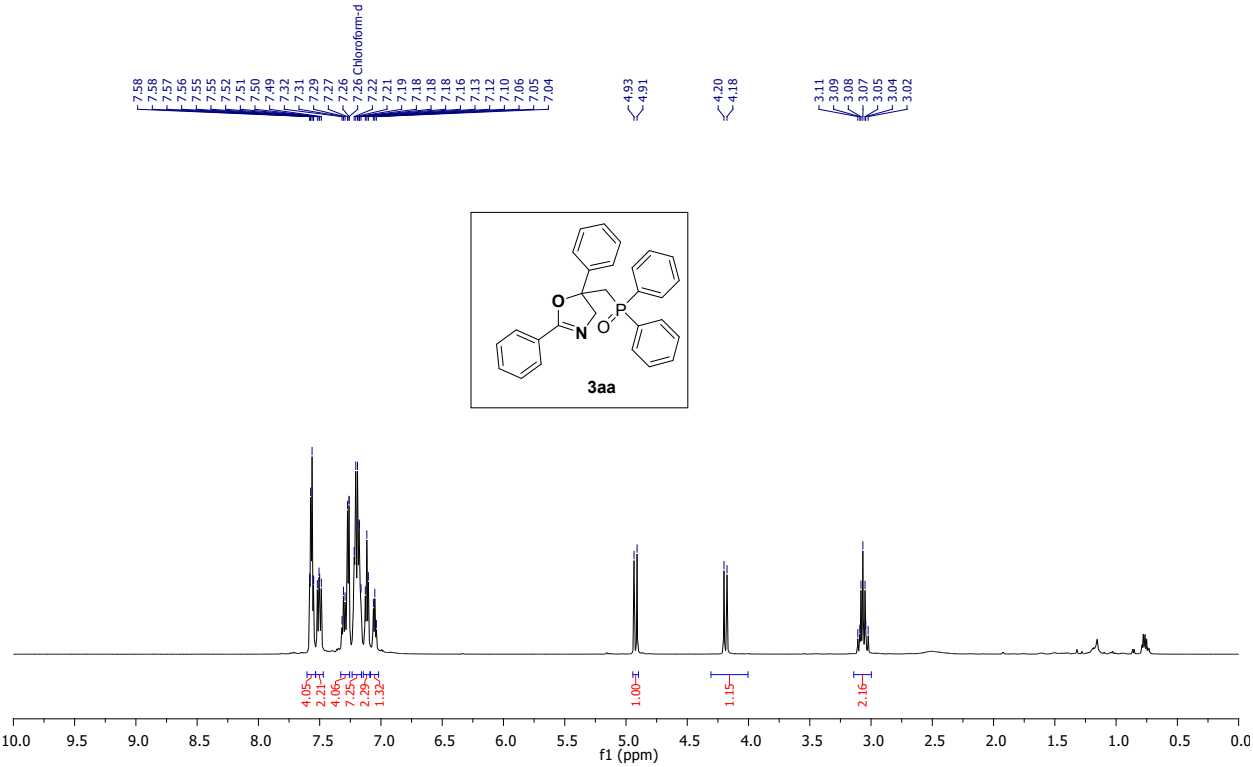

$^{13}$C{$^{1}$H} NMR (151 MHz) spectrum of **1w** in $CDCl_3$



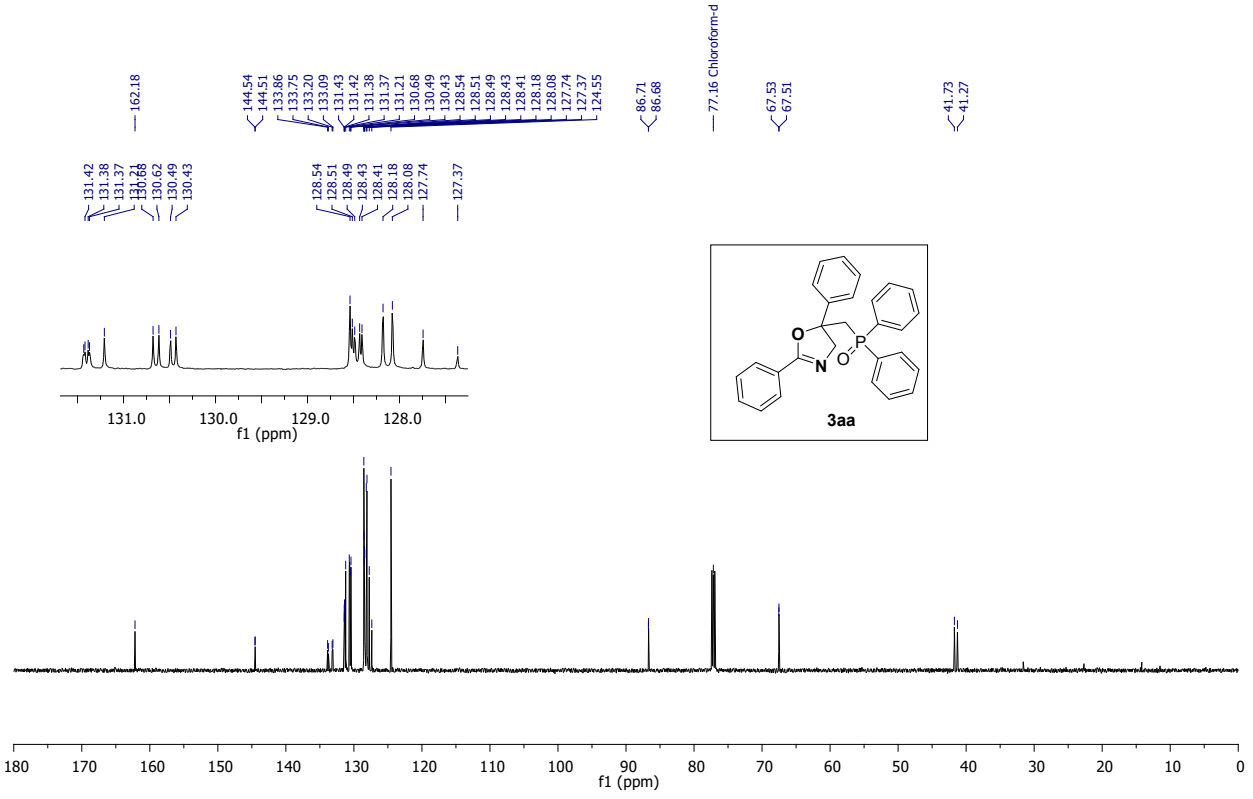

$^{1}$H NMR (600 MHz) spectrum of **1x** in $CDCl_3$



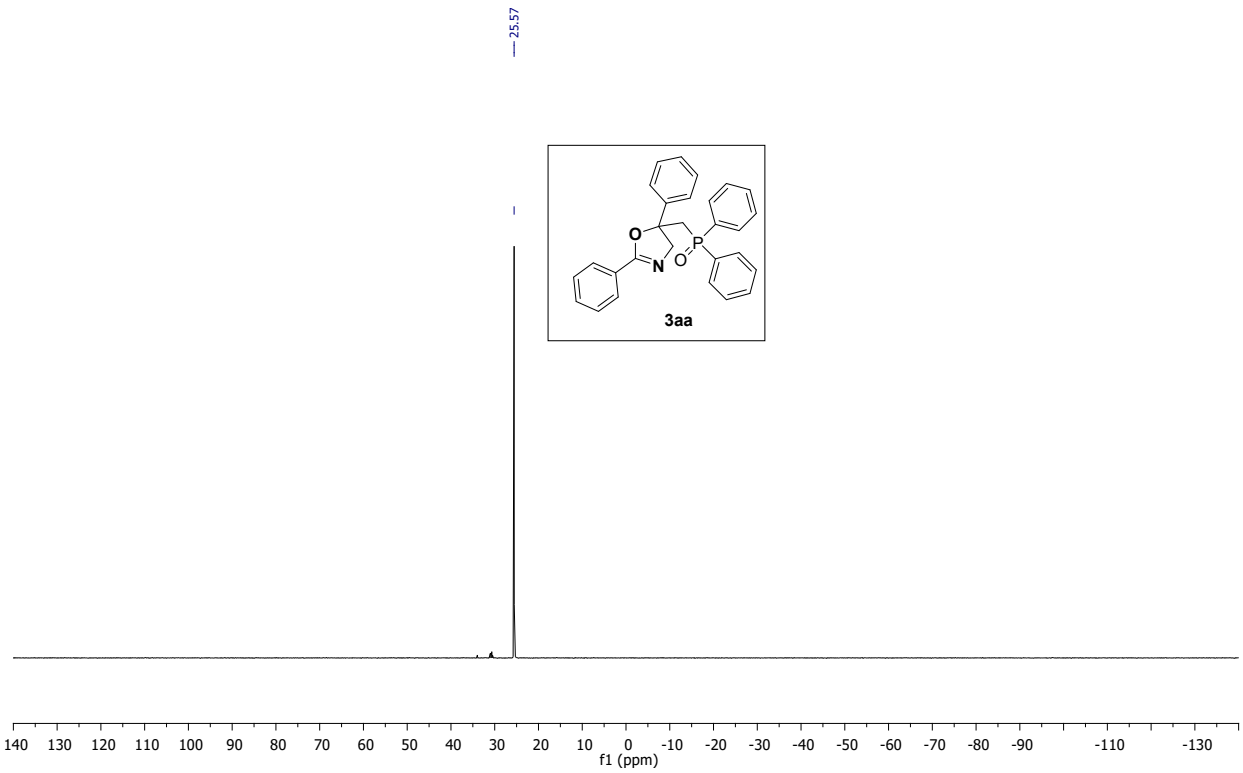

$^{13}$C {$^{1}$H} NMR (101 MHz) spectrum of **1x** in $CDCl_3$



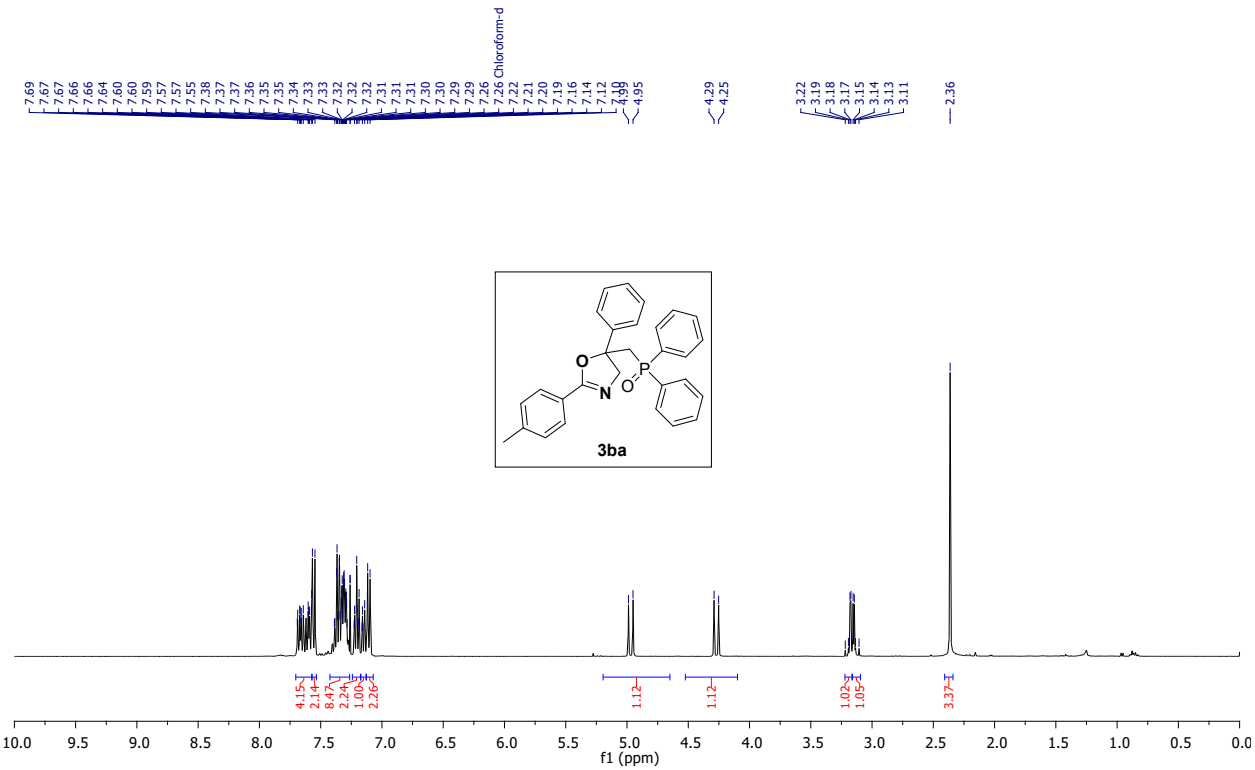

$^{1}$H NMR (600 MHz) spectrum of **3aa** in $CDCl_3$



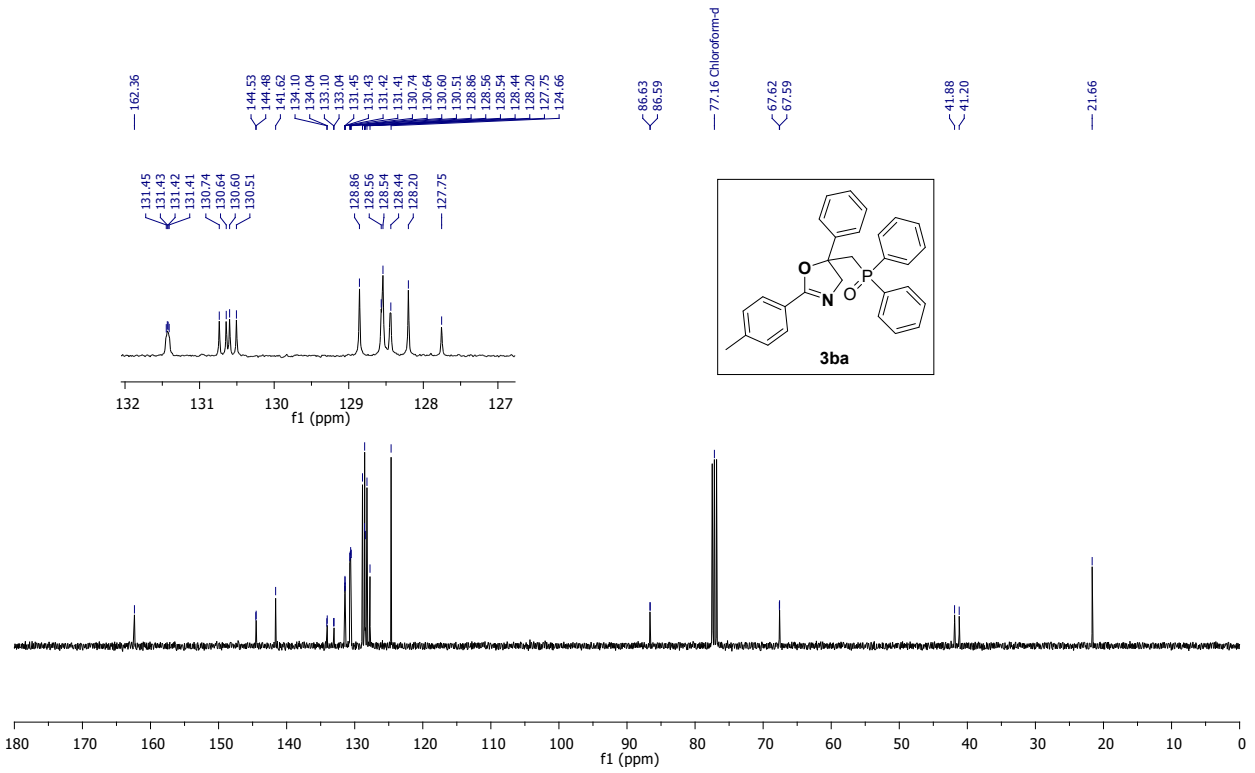

$^{13}$C{$^{1}$H} NMR (151 MHz) spectrum of **3aa** in $CDCl_3$



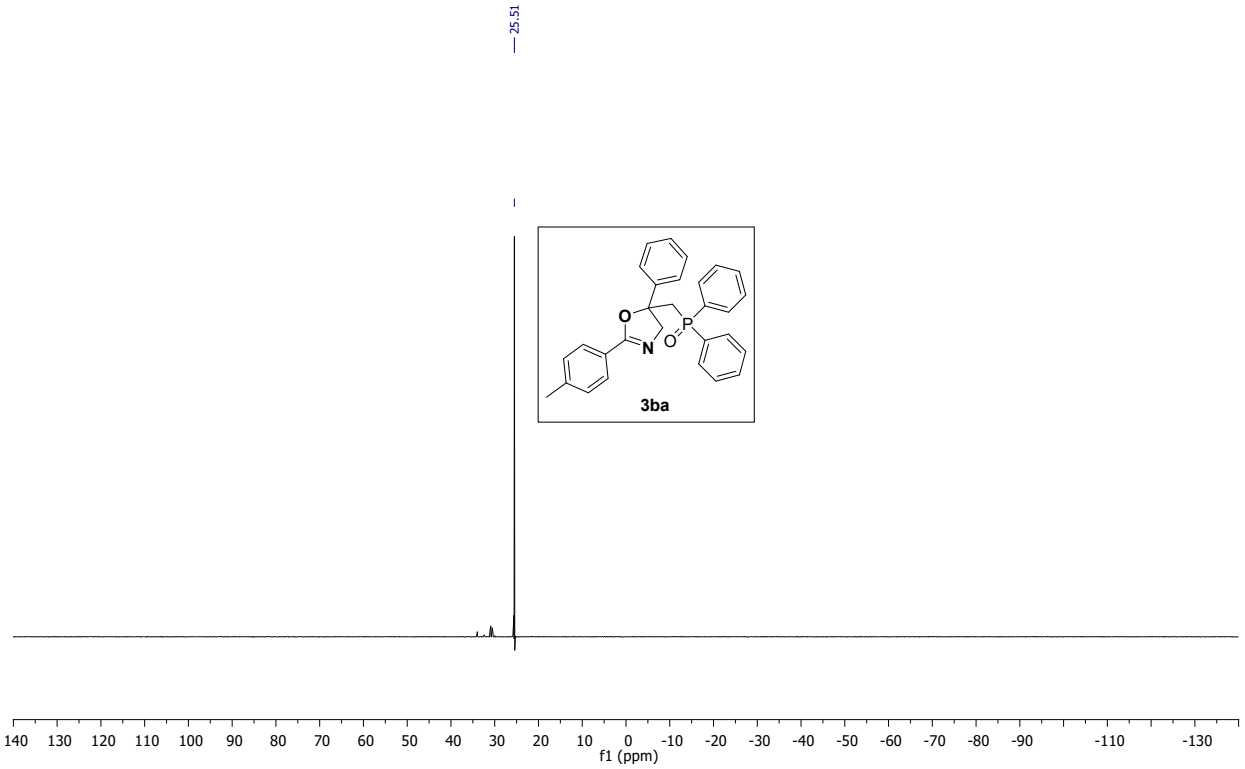

$^{31}$P NMR (243 MHz) spectrum of **3aa** in $CDCl_3$



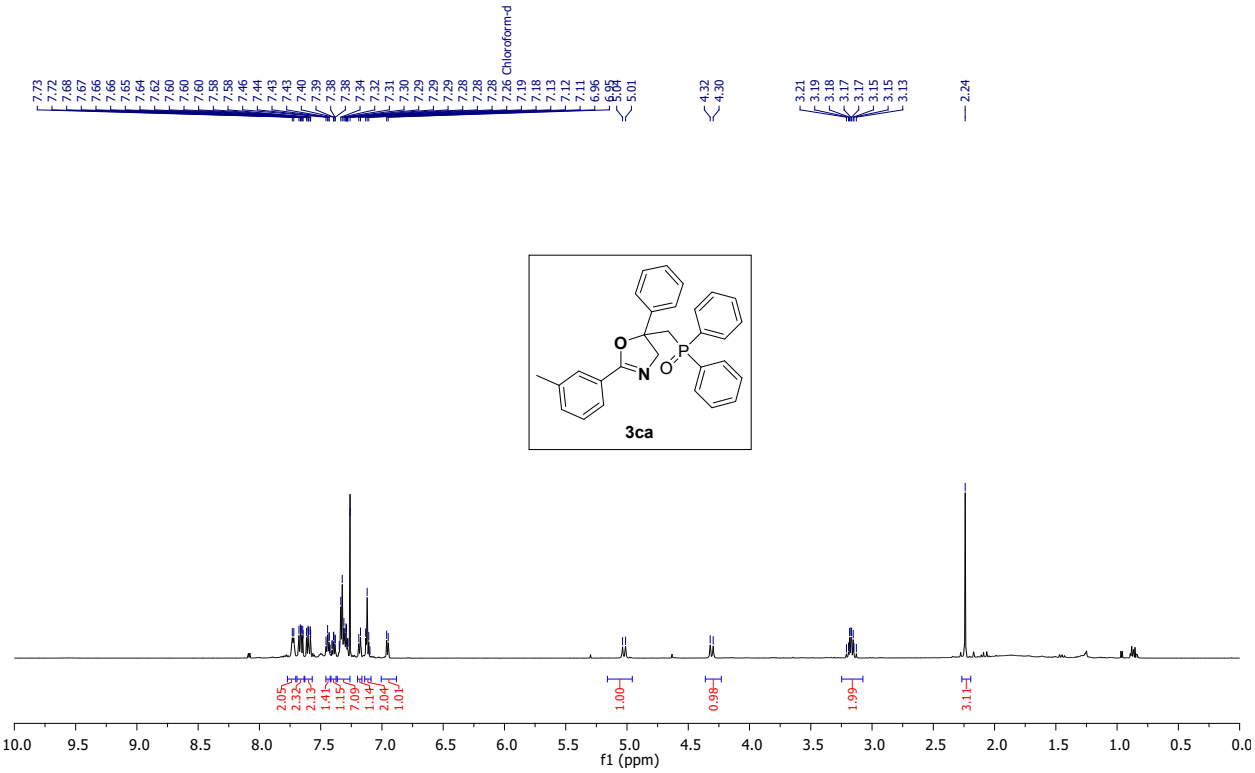

$^{1}$H NMR (400 MHz) spectrum of **3ba** in $CDCl_3$



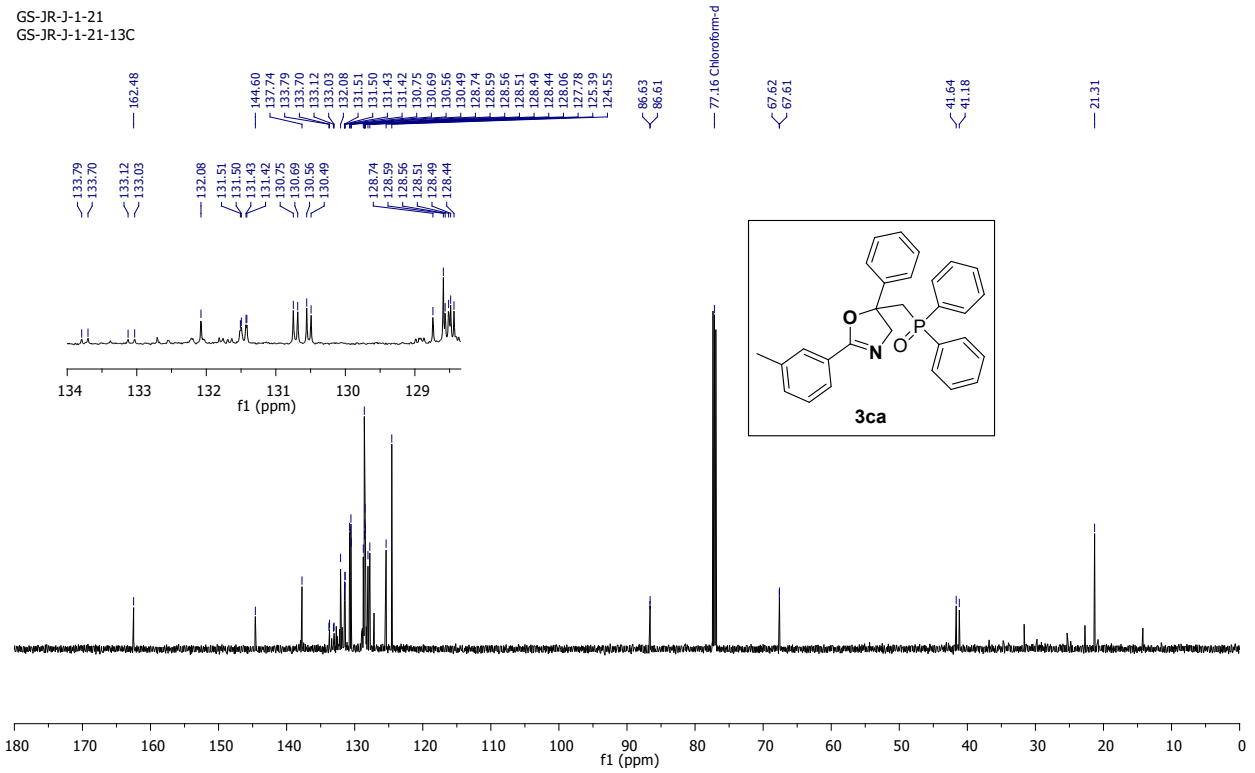

$^{13}C\{^{1}H\}$ NMR (101 MHz) spectrum of **3ba** in $CDCl_3$



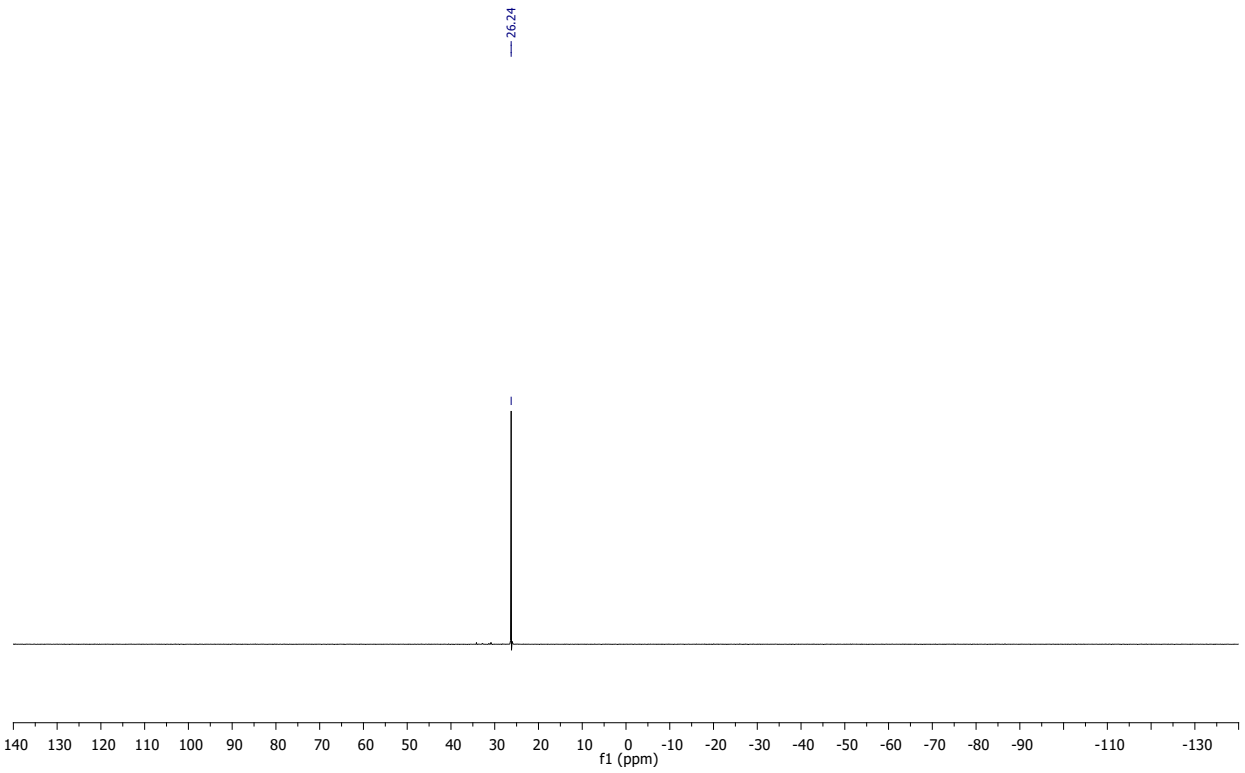

$^{31}P$ NMR (243 MHz) spectrum of **3ba** in $CDCl_3$



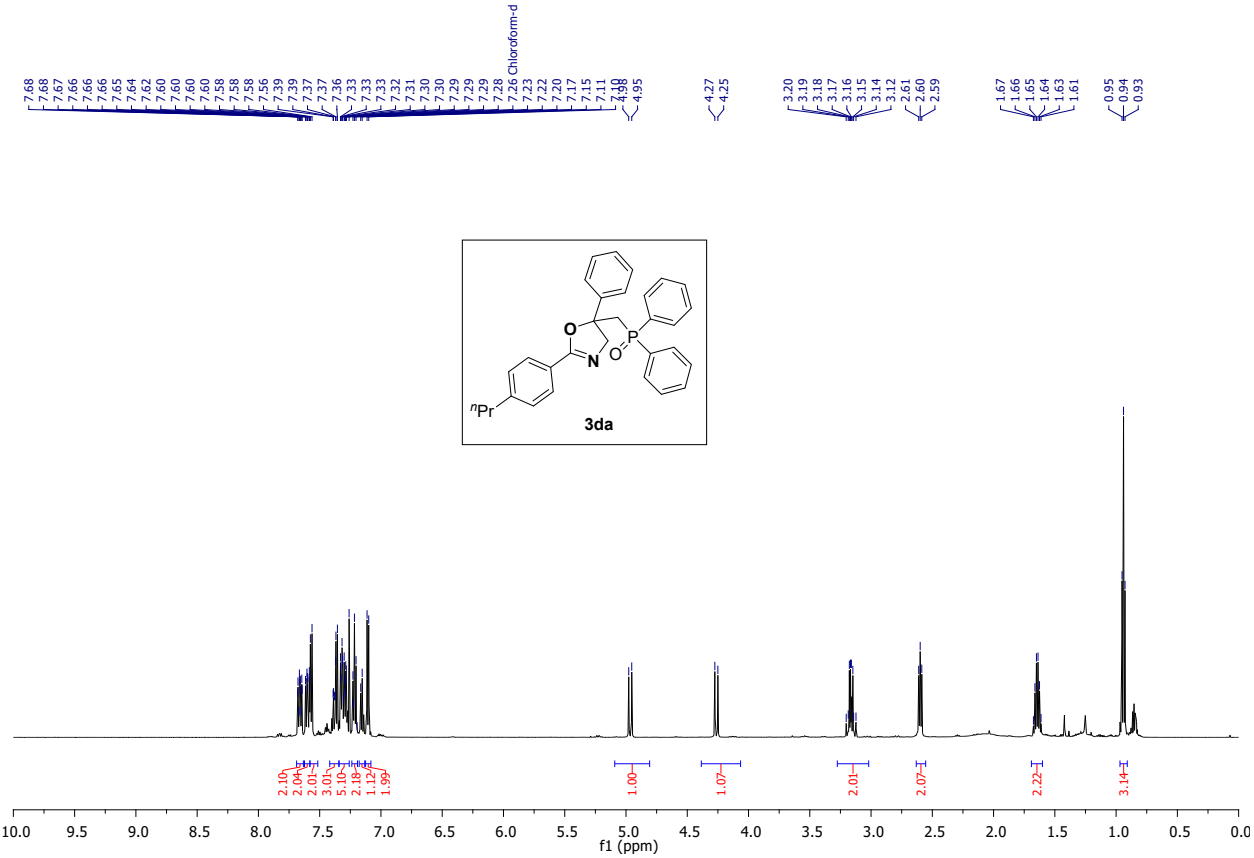

$^{1}$H NMR (600 MHz) spectrum of **3ca** in $CDCl_3$



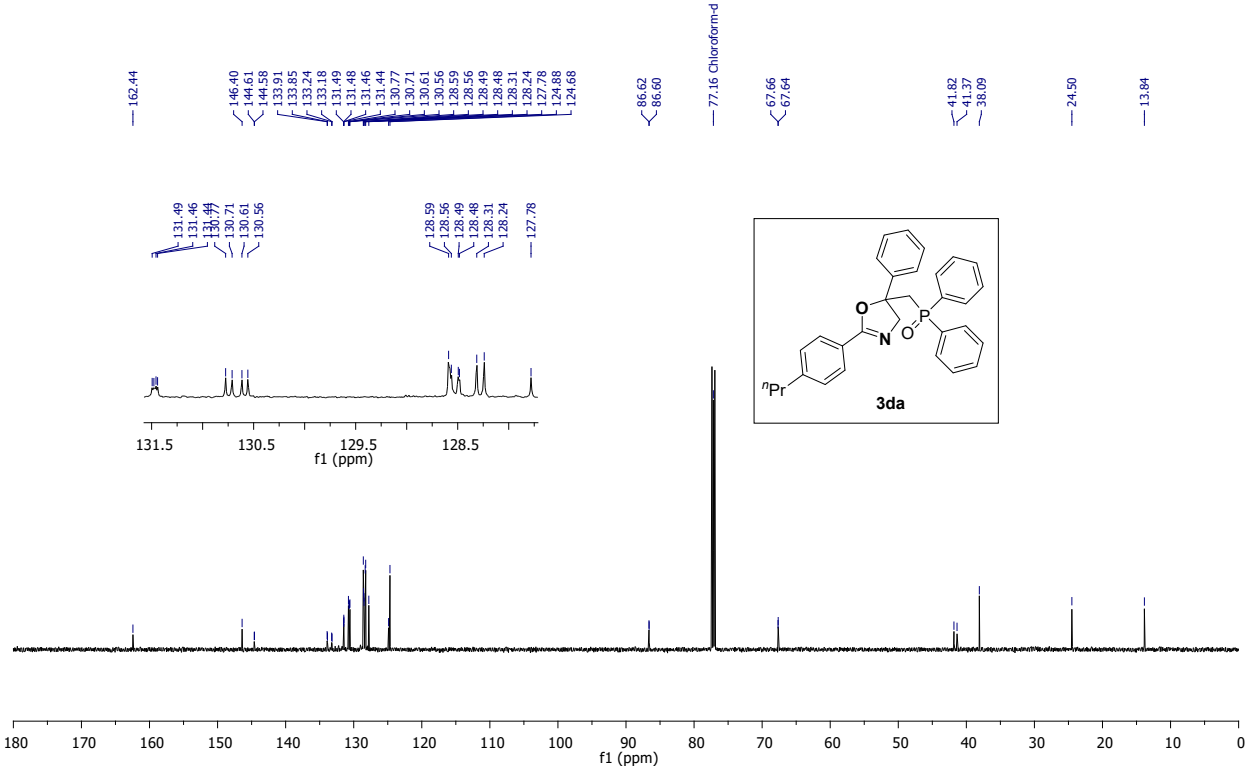

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3ca** in $CDCl_3$



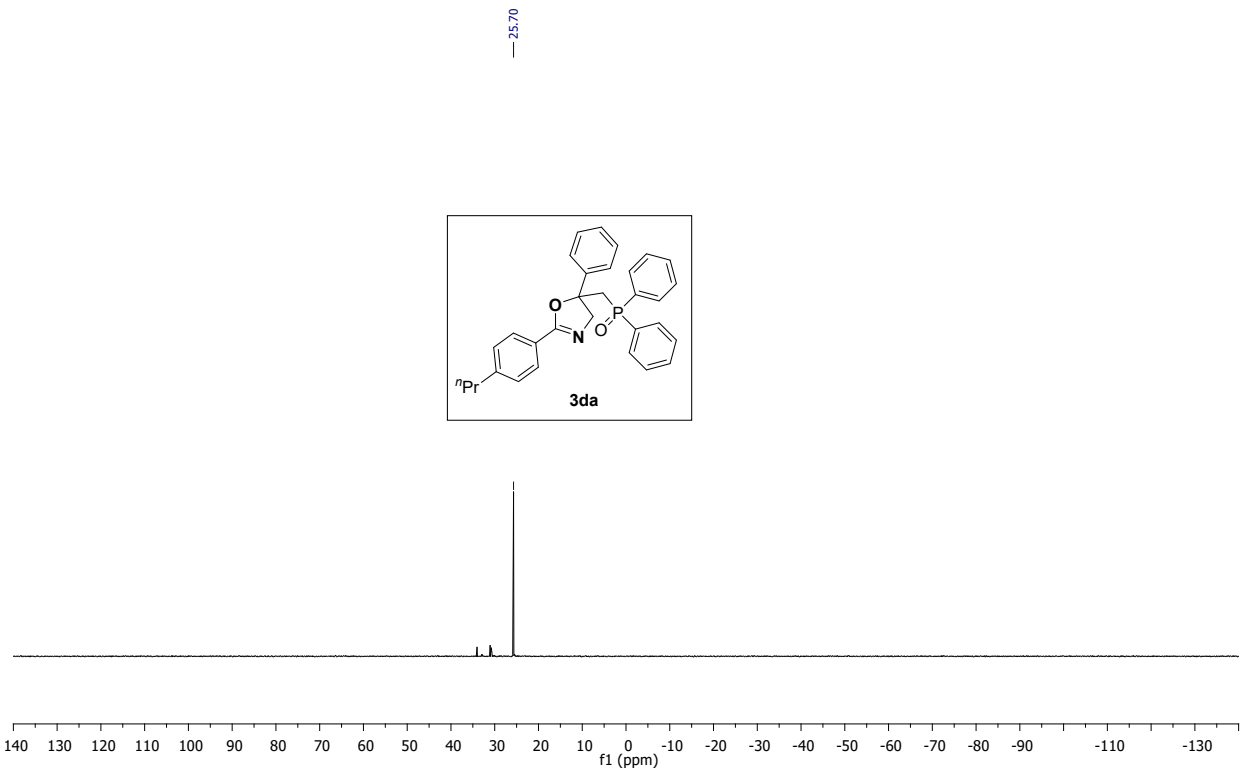

$^{31}$P NMR (243 MHz) spectrum of **3ca** in $CDCl_3$



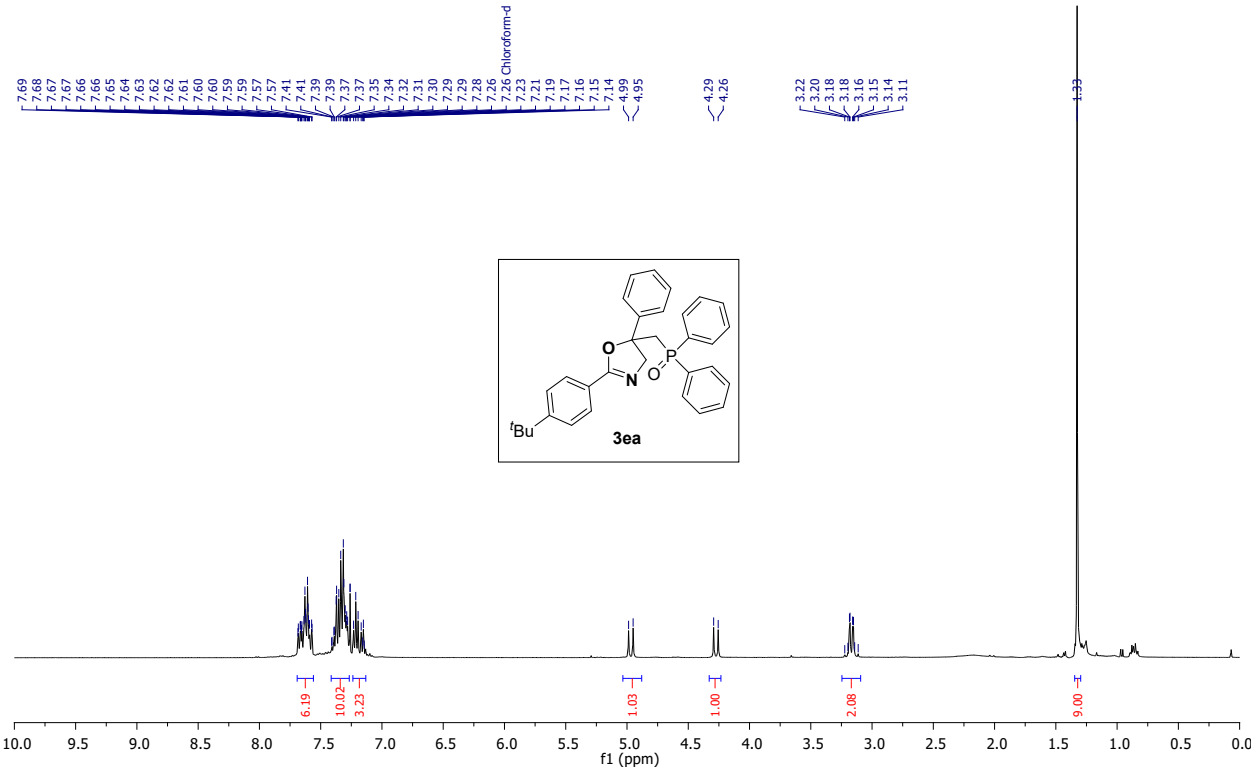

$^{1}$H NMR (600 MHz) spectrum of **3da** in $CDCl_3$



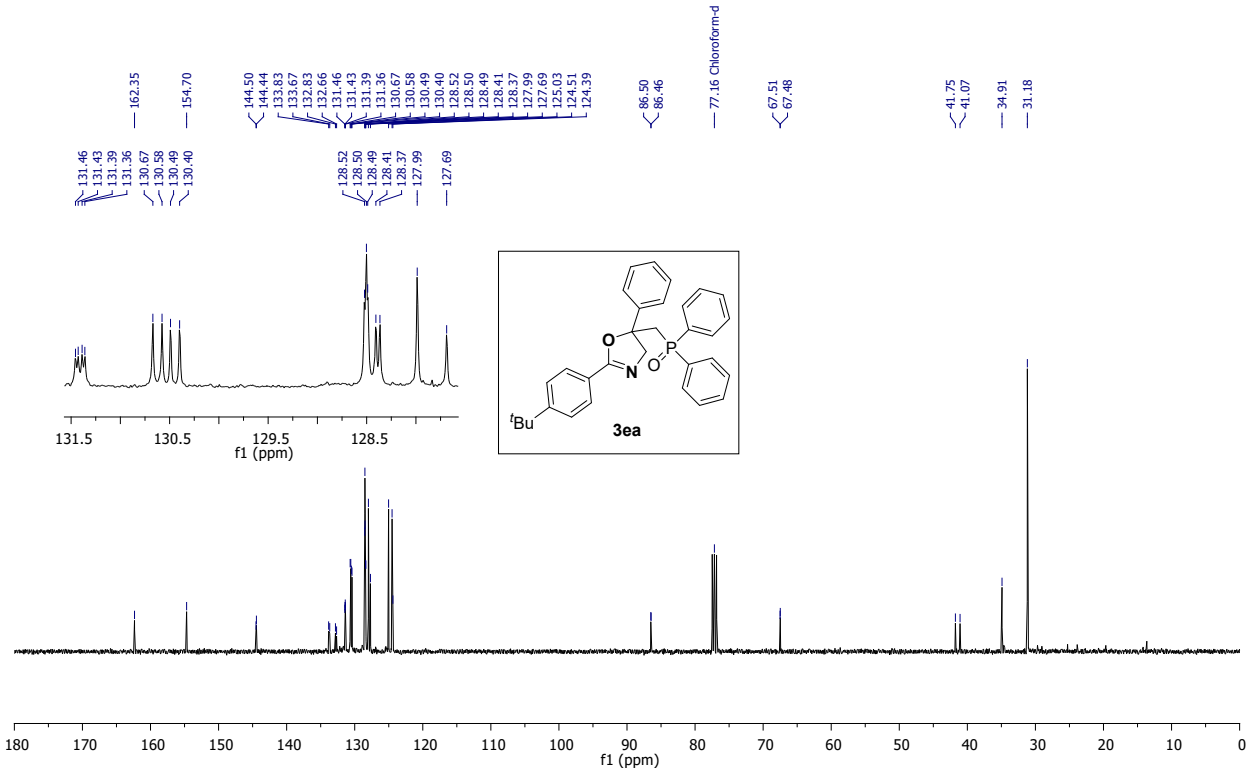

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3da** in $CDCl_3$



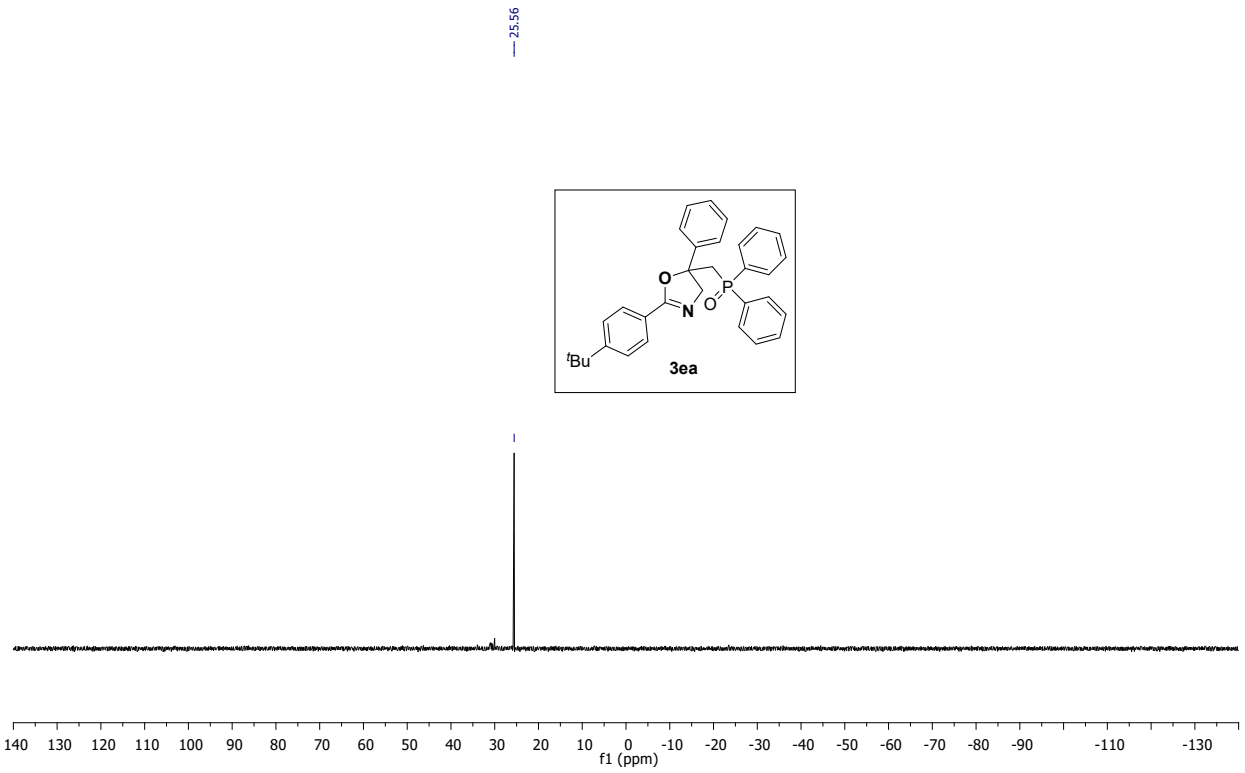

$^{31}$P NMR (243 MHz) spectrum of **3da** in $CDCl_3$



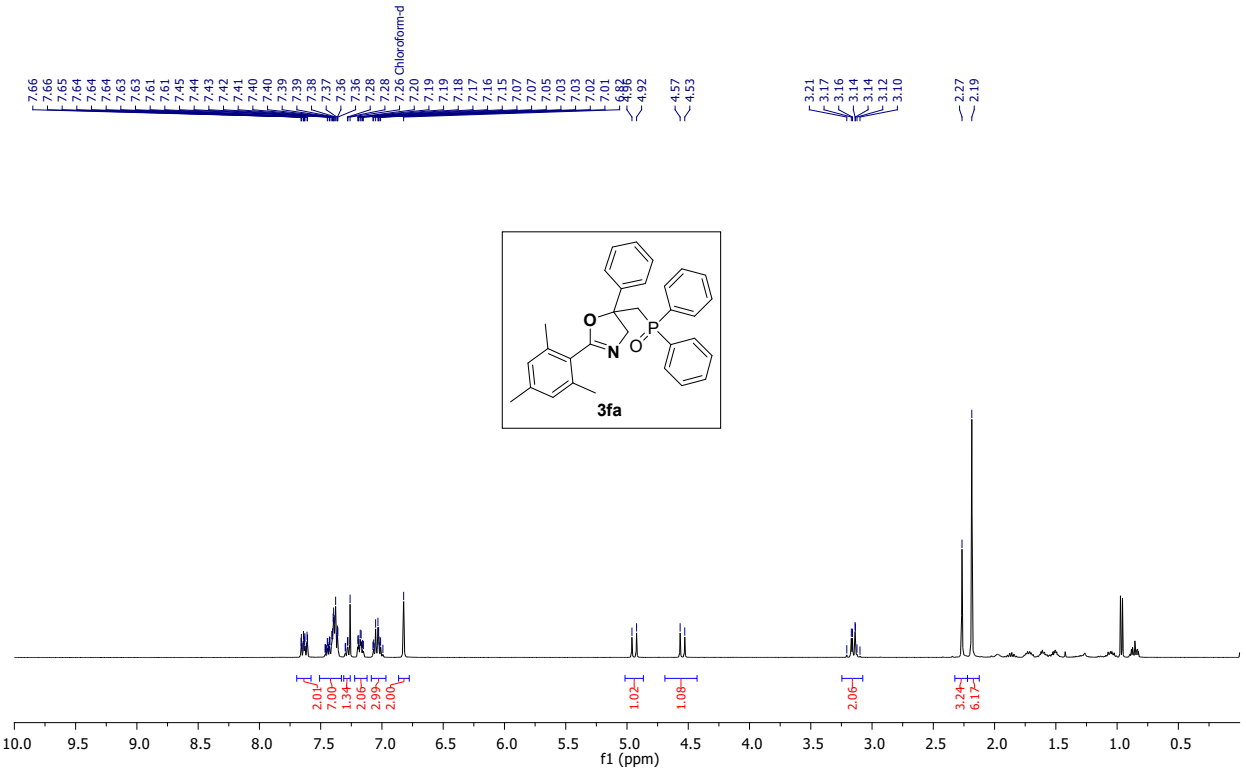

$^{1}$H NMR (600 MHz) spectrum of **3ea** in $CDCl_3$



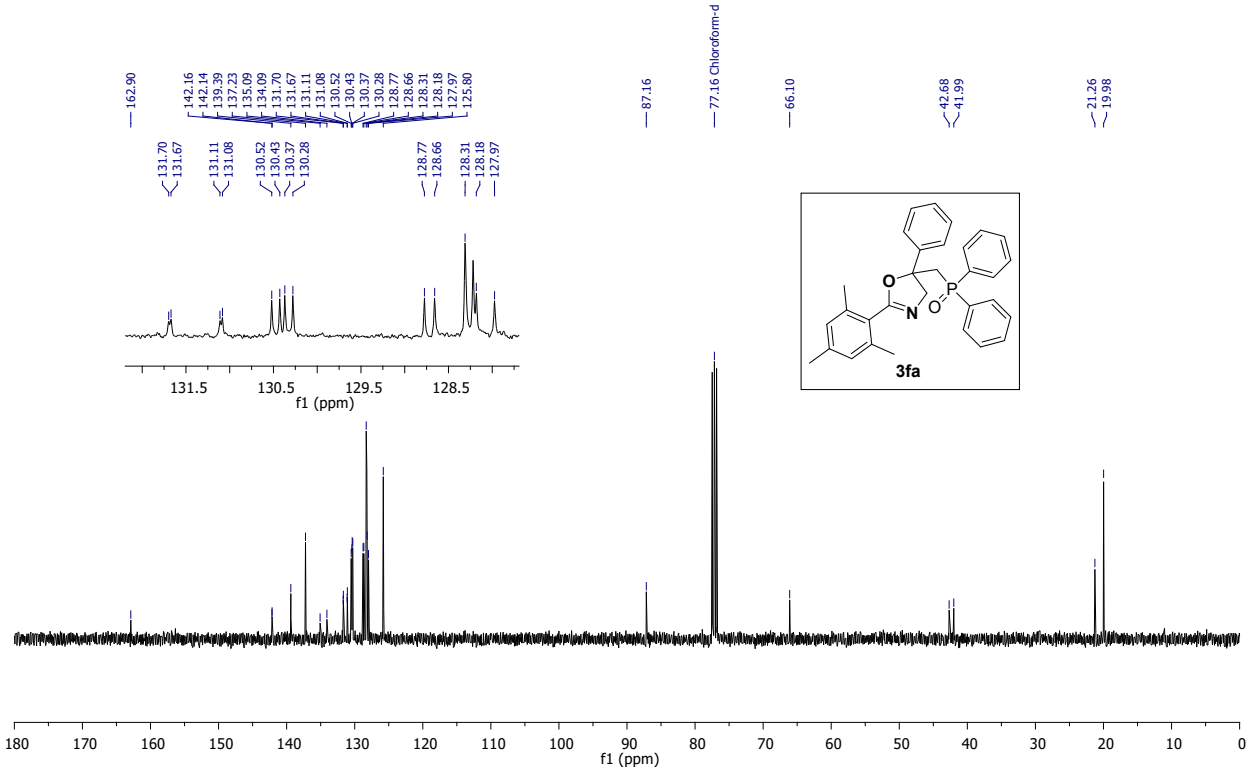

$^{13}$C {$^{1}$H} NMR (101 MHz) spectrum of **3ea** in $CDCl_3$



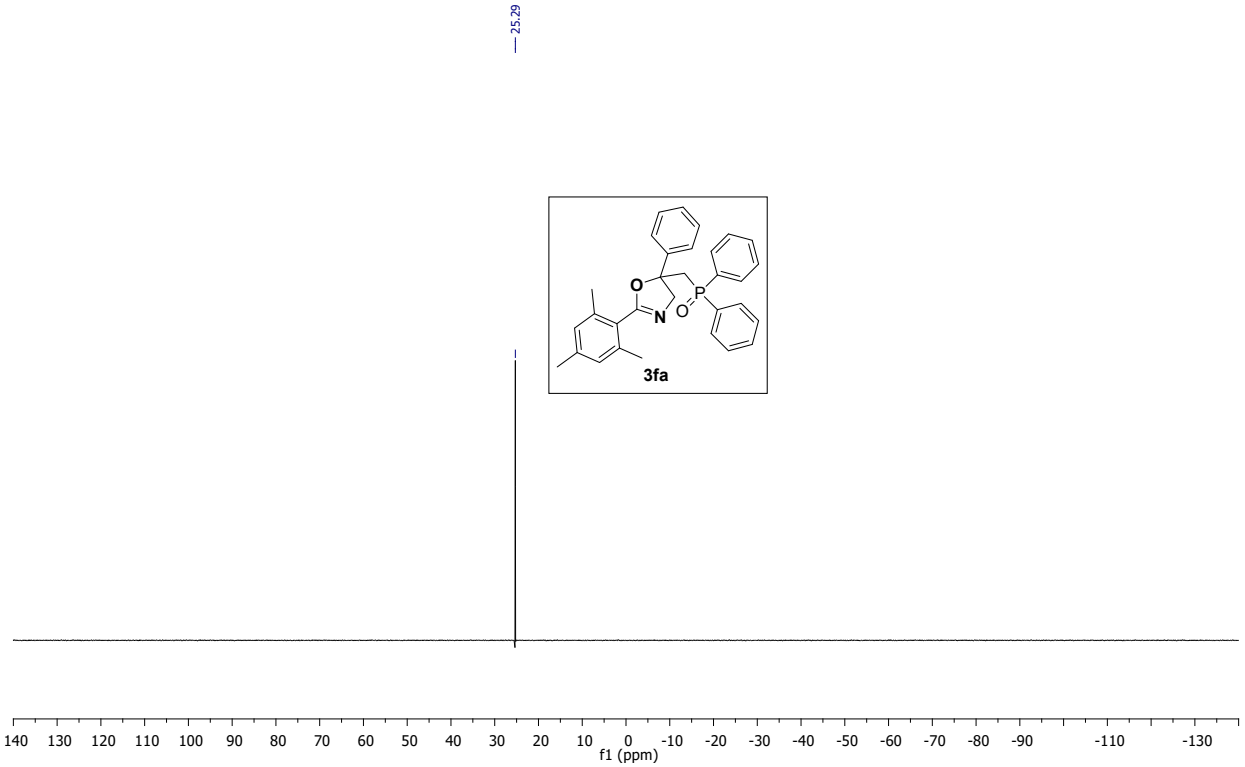

$^{31}$P NMR (243 MHz) spectrum of **3ea** in $CDCl_3$



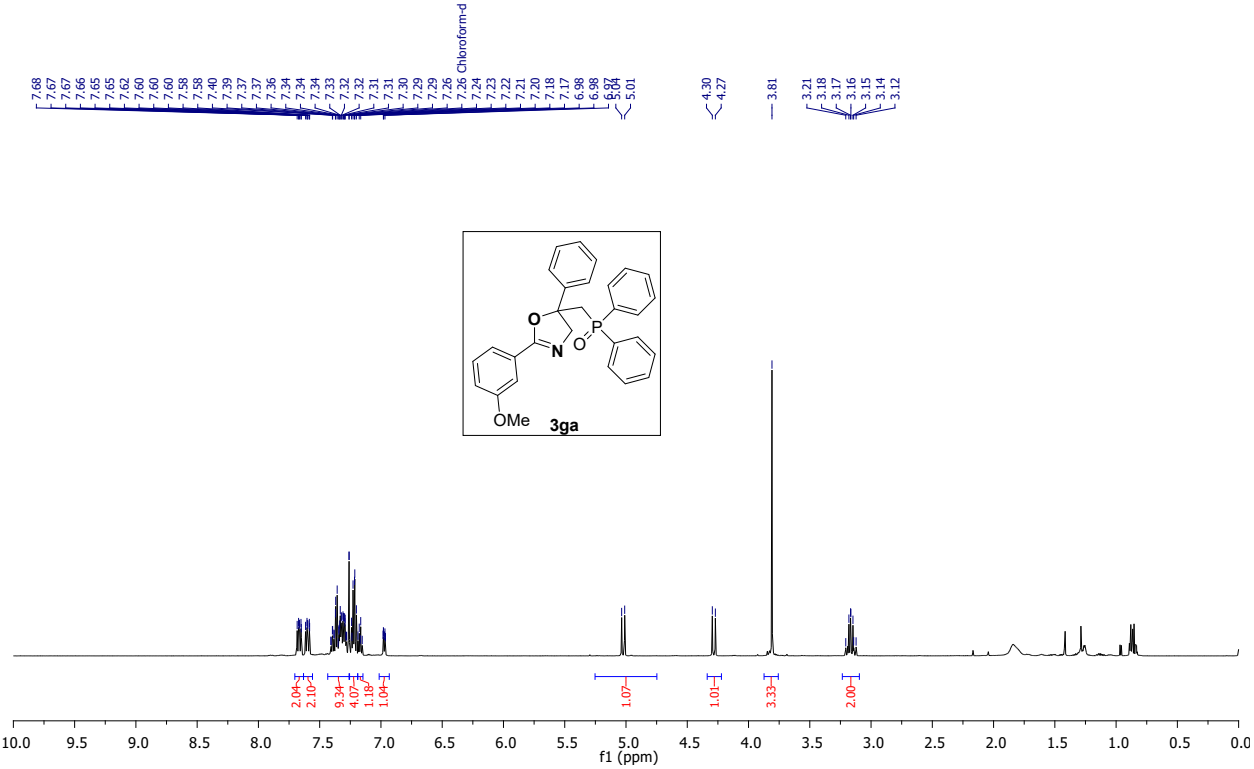

$^{1}$H NMR (400 MHz) spectrum of **3fa** in $CDCl_3$



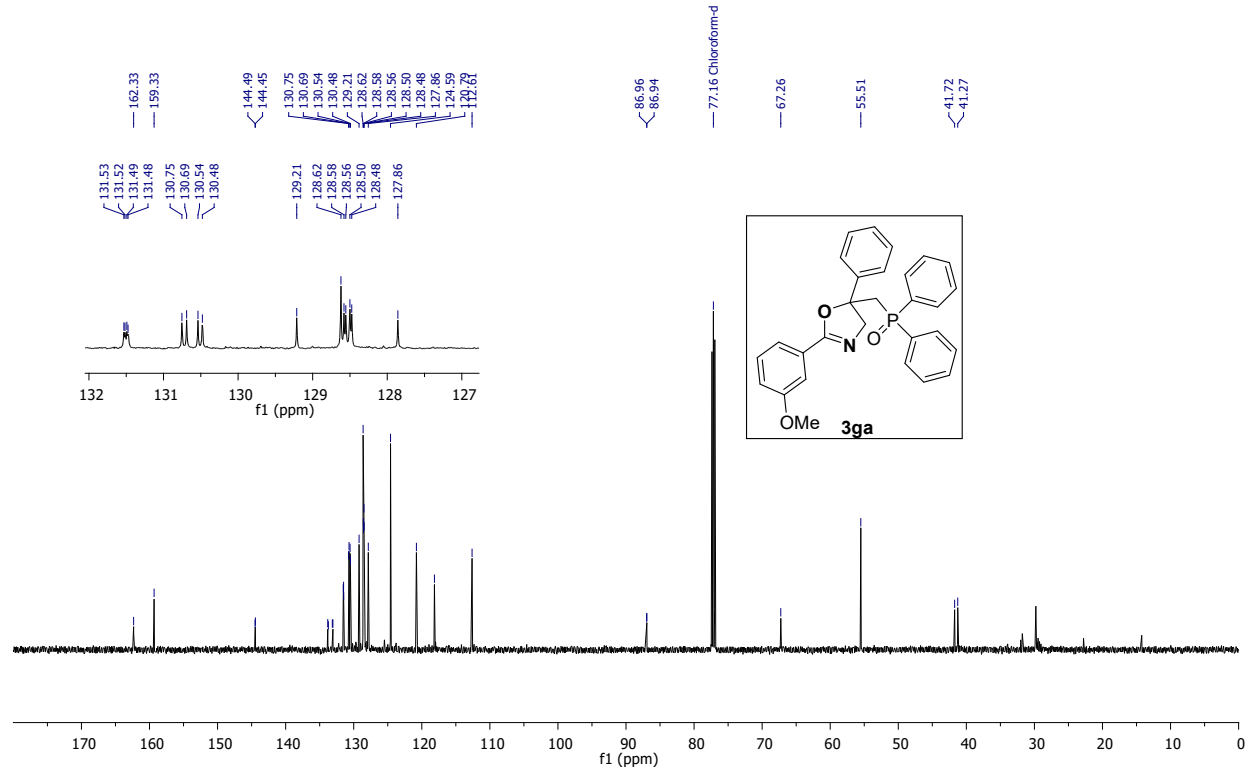

$^{13}$C {$^{1}$H} NMR (101 MHz) spectrum of **3fa** in $CDCl_3$



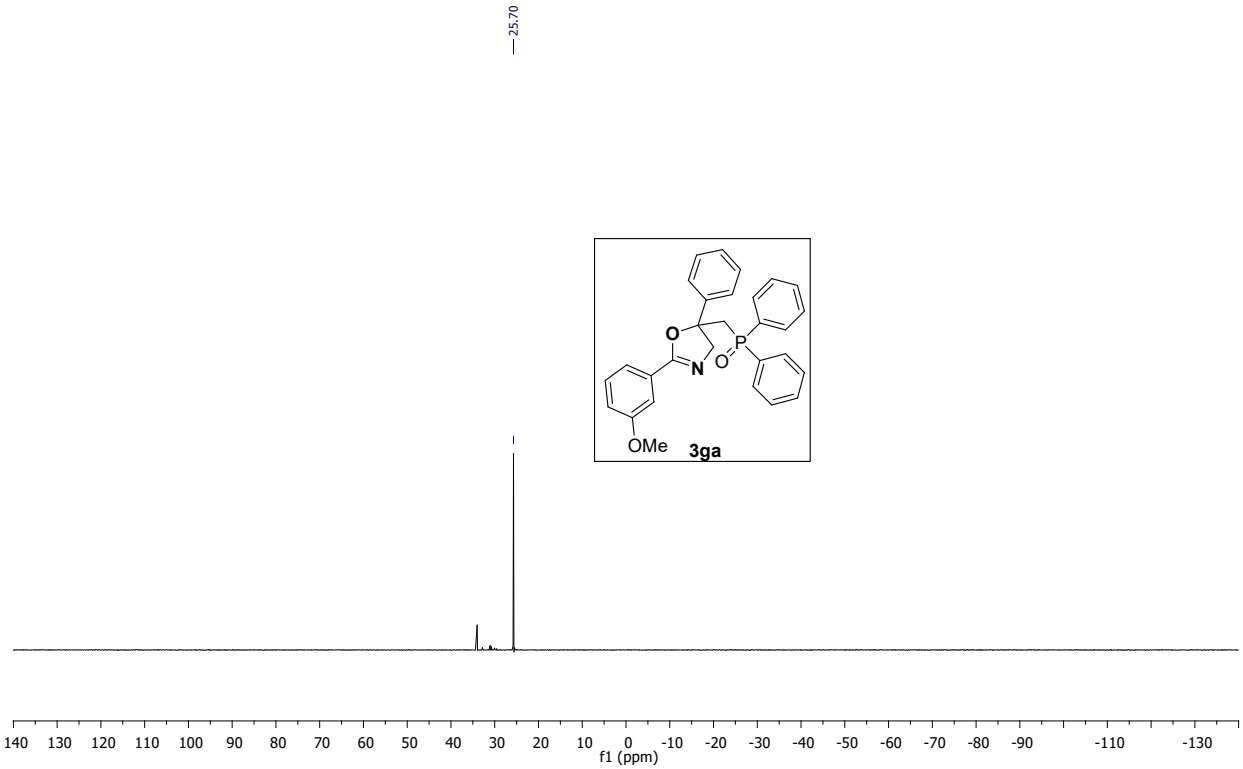

$^{31}$P NMR (243 MHz) spectrum of **3fa** in $CDCl_3$



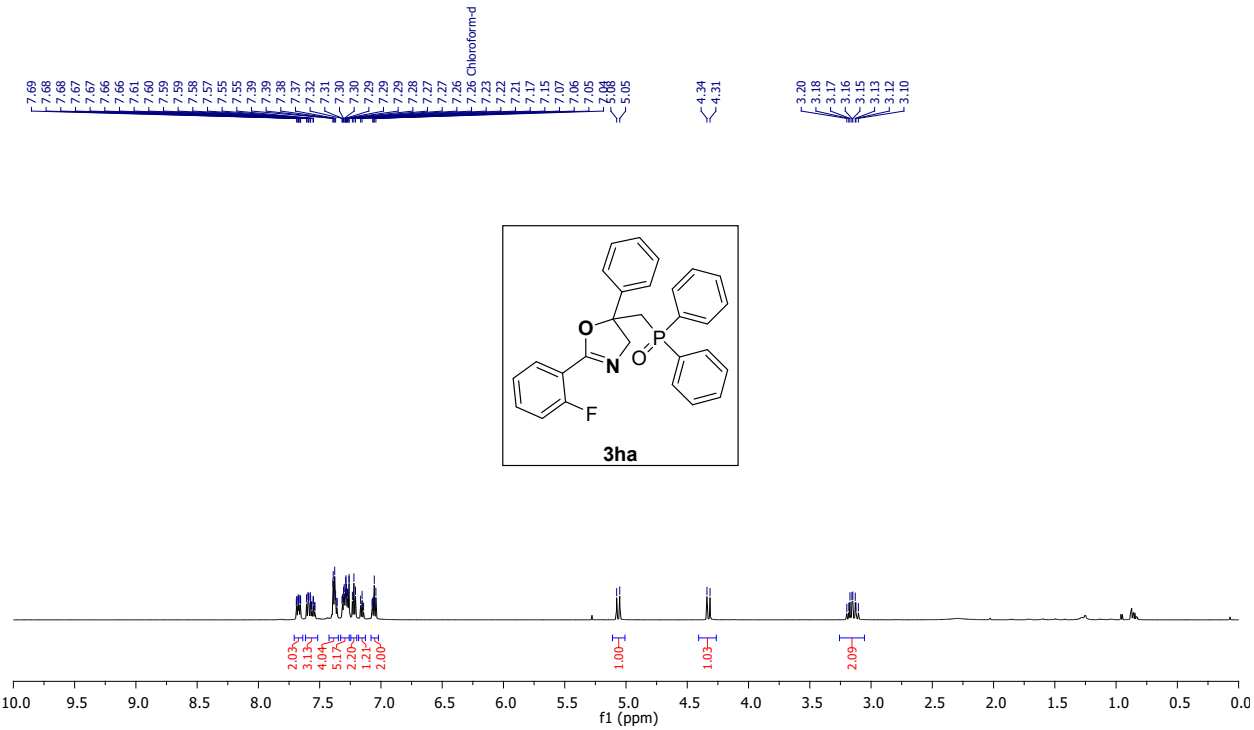

$^{1}$H NMR (600 MHz) spectrum of **3ga** in $CDCl_3$



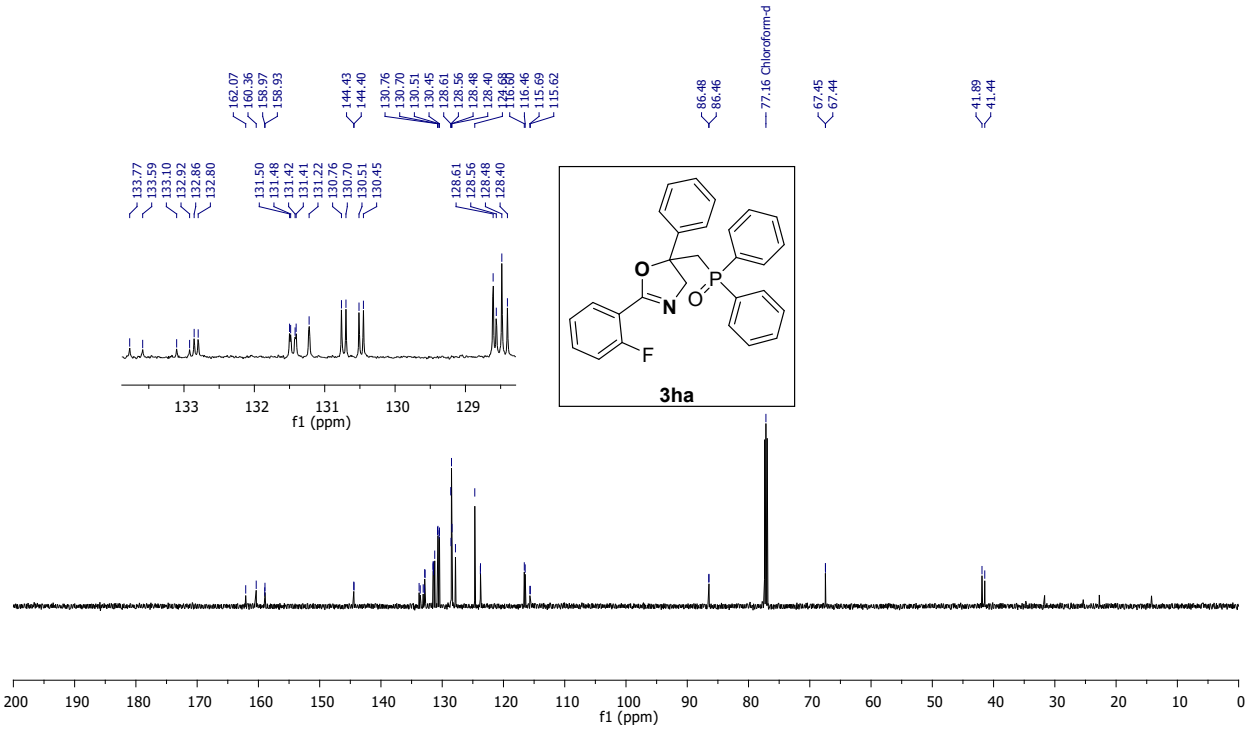

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3ga** in $CDCl_3$



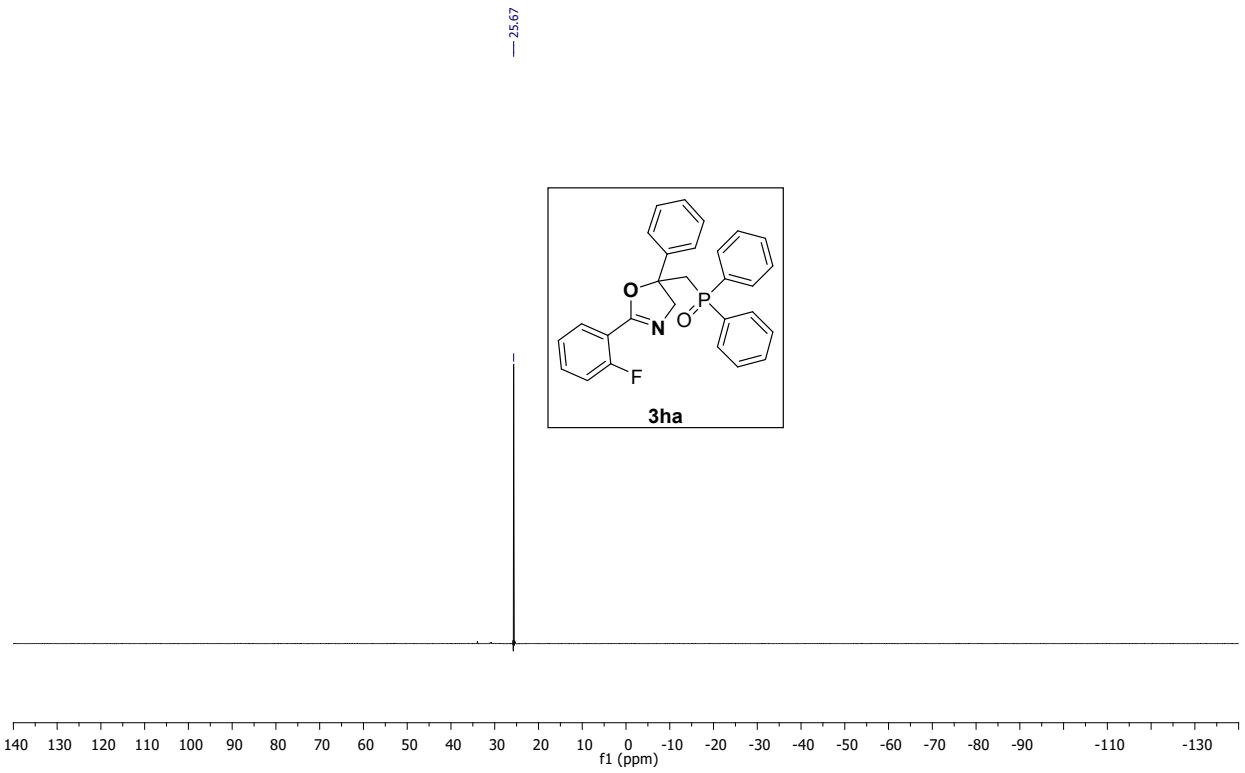

$^{31}$P NMR (243 MHz) spectrum of **3ga** in $CDCl_3$



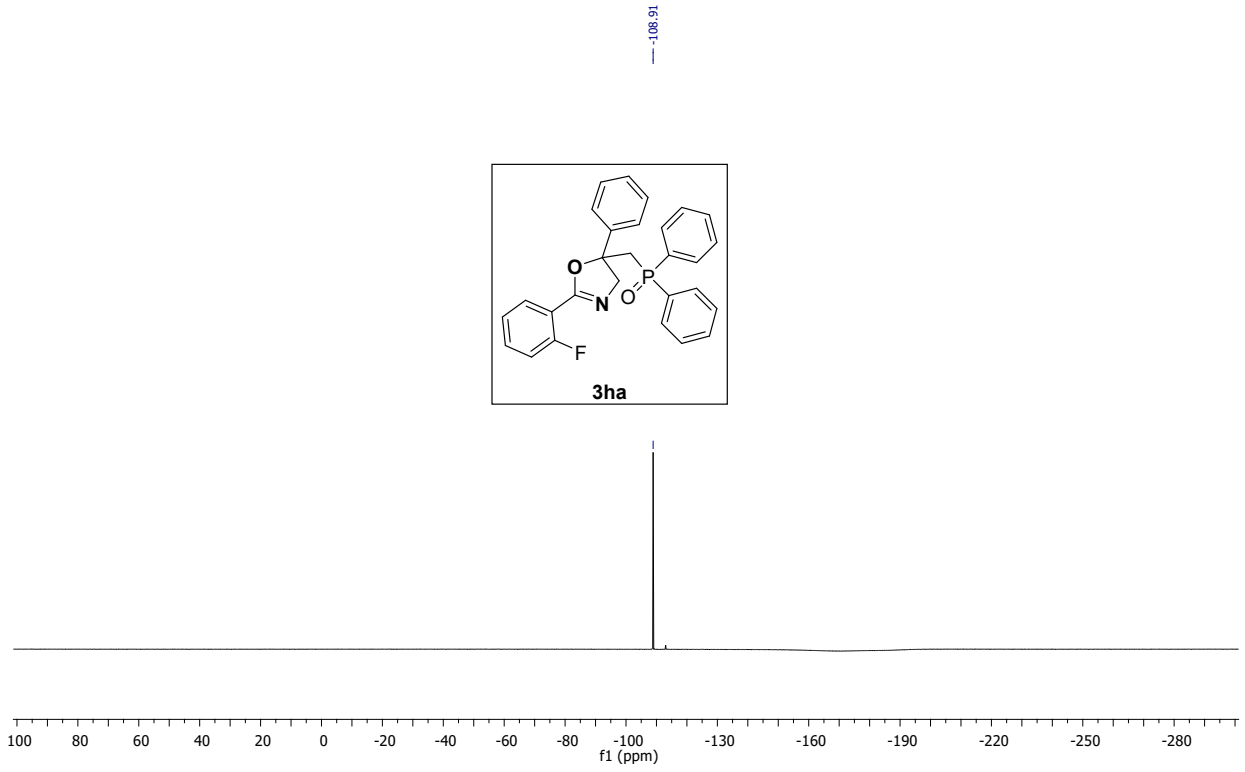

$^{1}$H NMR (400 MHz) spectrum of **3ha** in $CDCl_3$



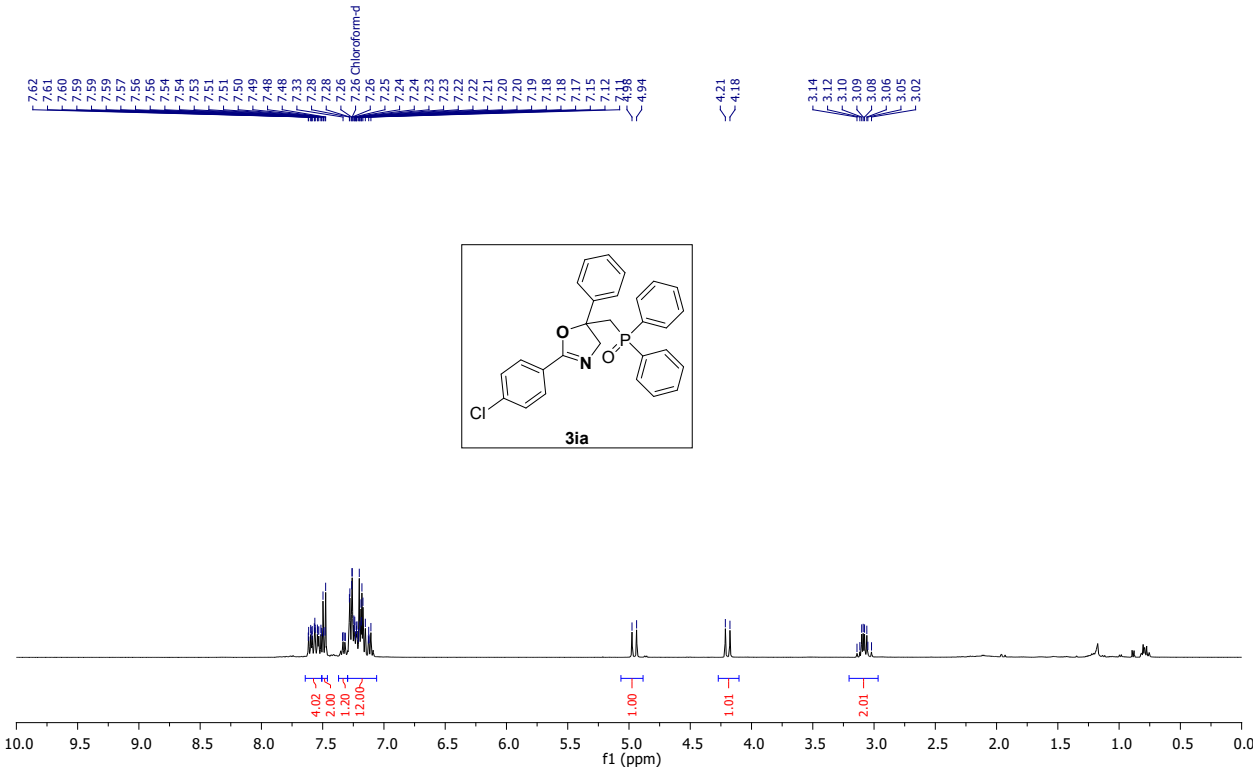

$^{13}$C {$^{1}$H} NMR (101 MHz) spectrum of **3ha** in $CDCl_3$



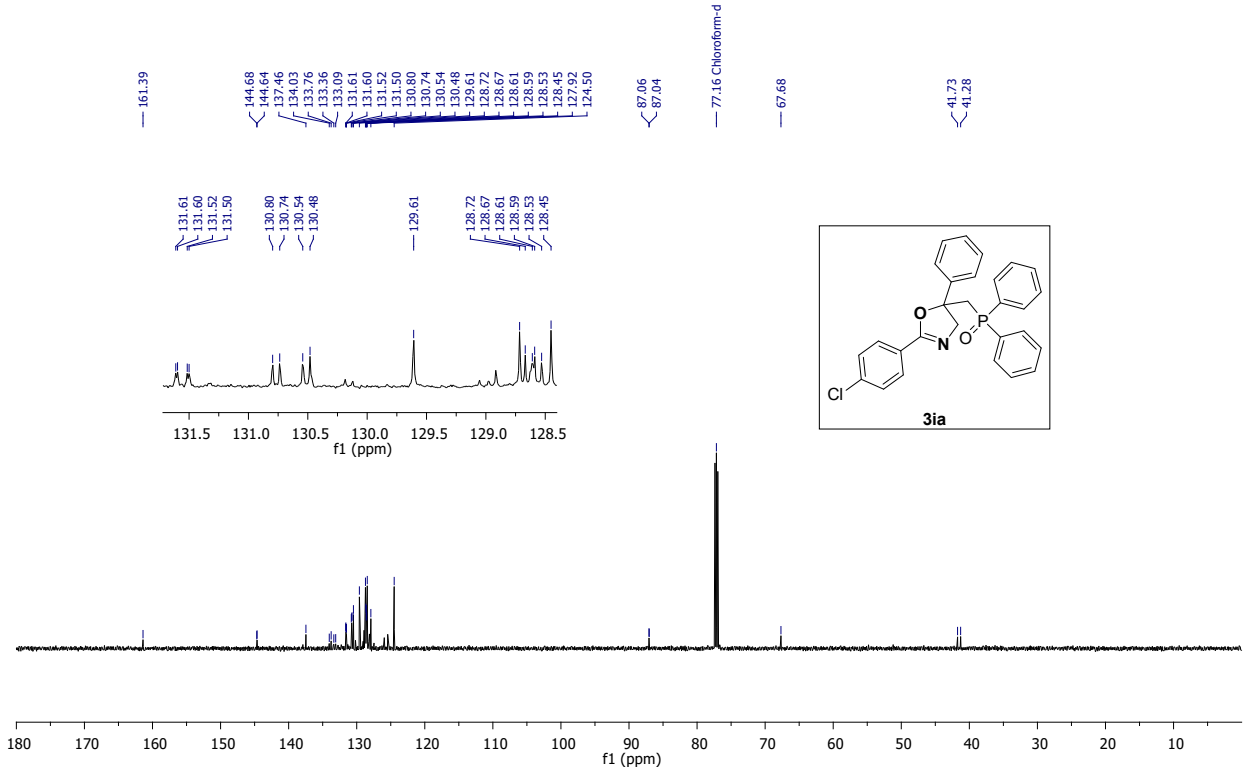

$^{31}$P NMR (243 MHz) spectrum of **3ha** in $CDCl_3$



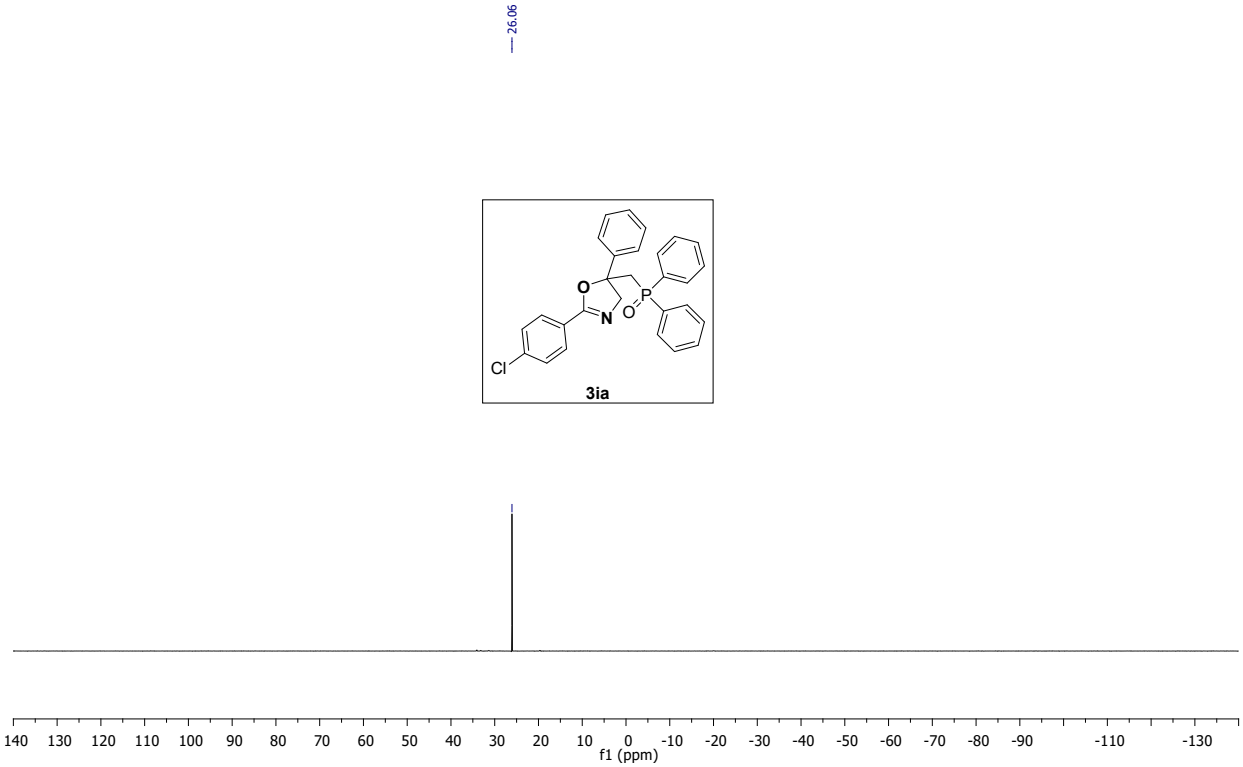

$^{19}$F NMR (565 MHz, $CDCl_3$) spectrum of **3ha** in $CDCl_3$



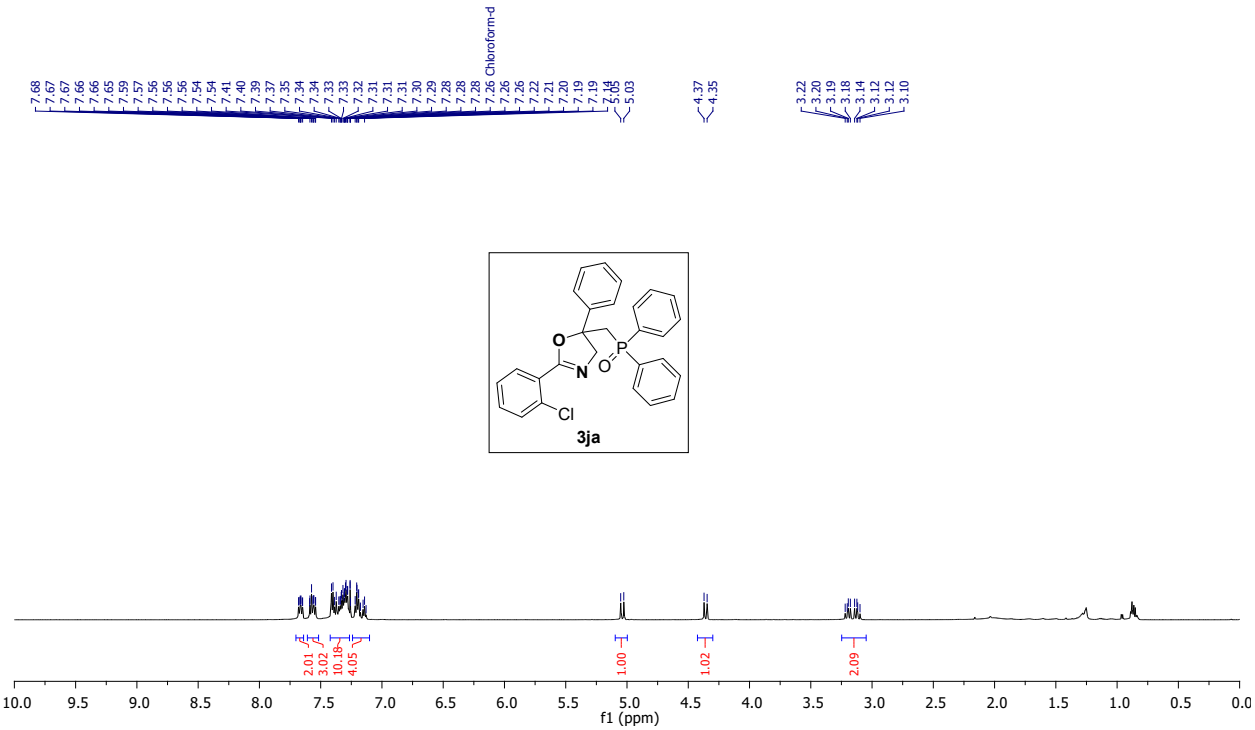

$^{1}$H NMR (400 MHz) spectrum of **3ia** in $CDCl_3$



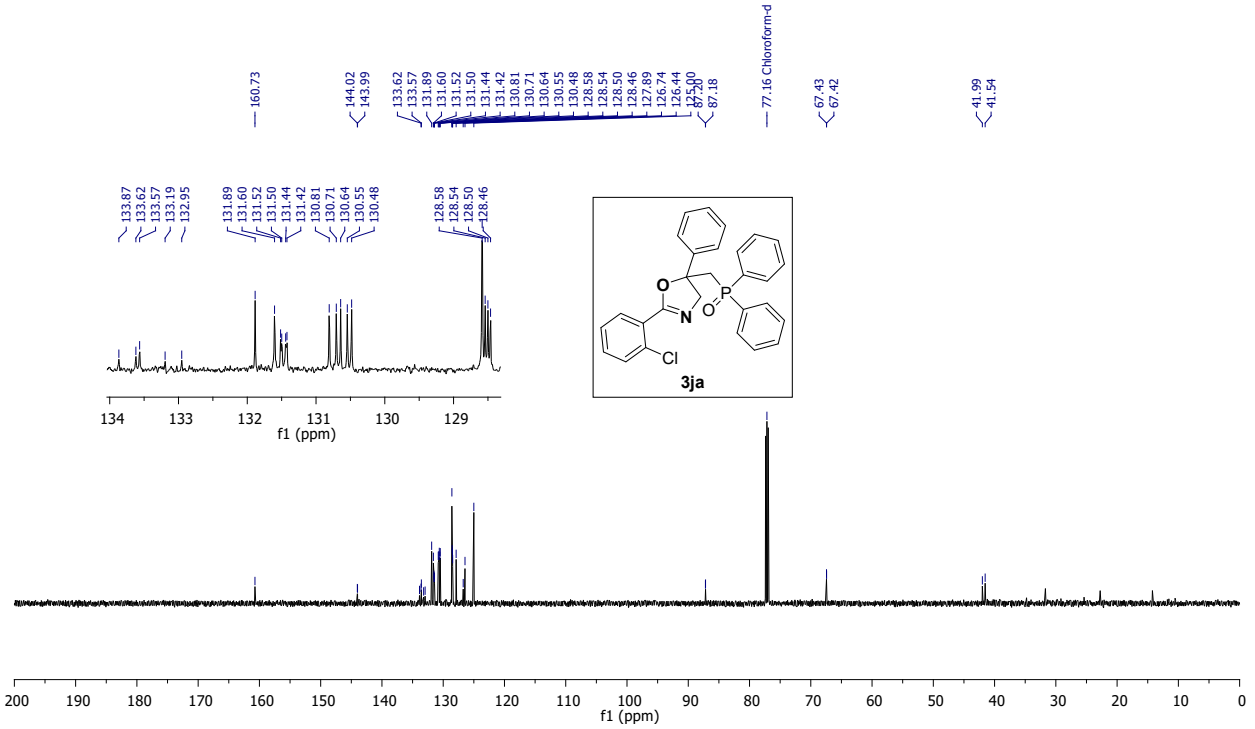

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3ia** in $CDCl_3$



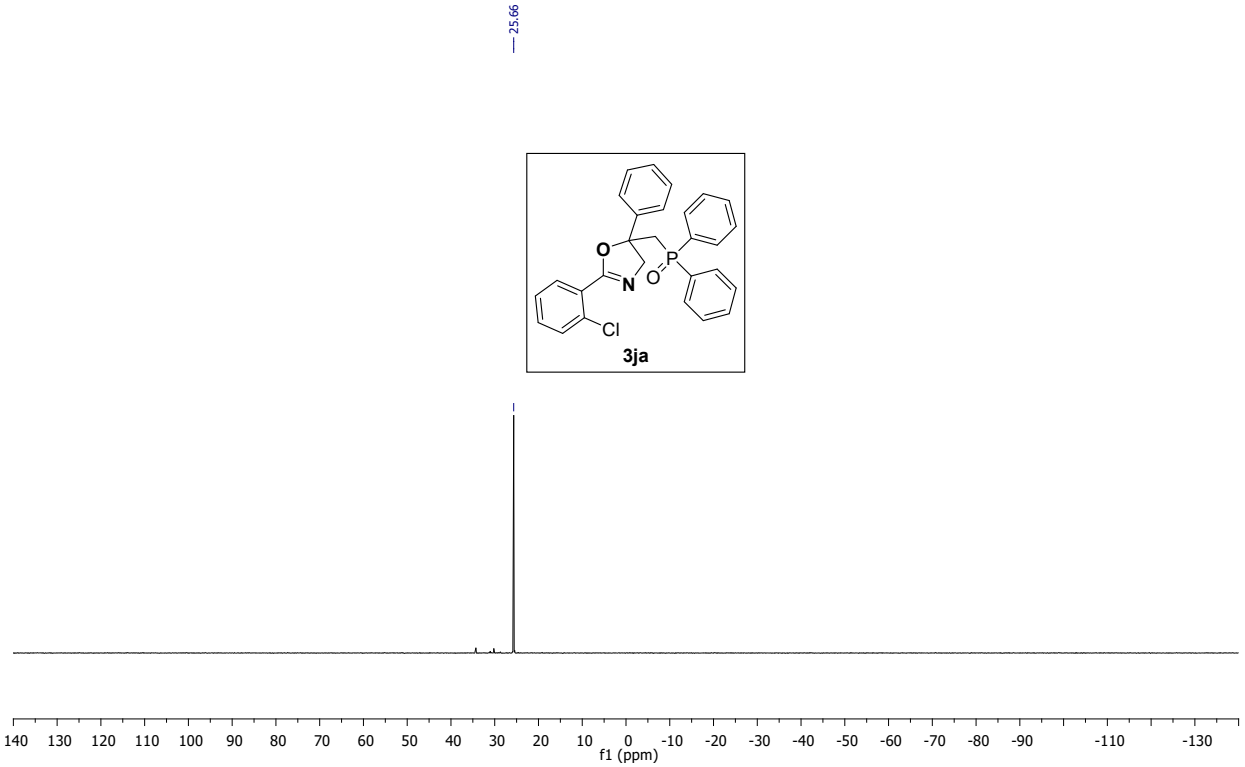

$^{31}$P NMR (243 MHz) spectrum of **3ia** in $CDCl_3$



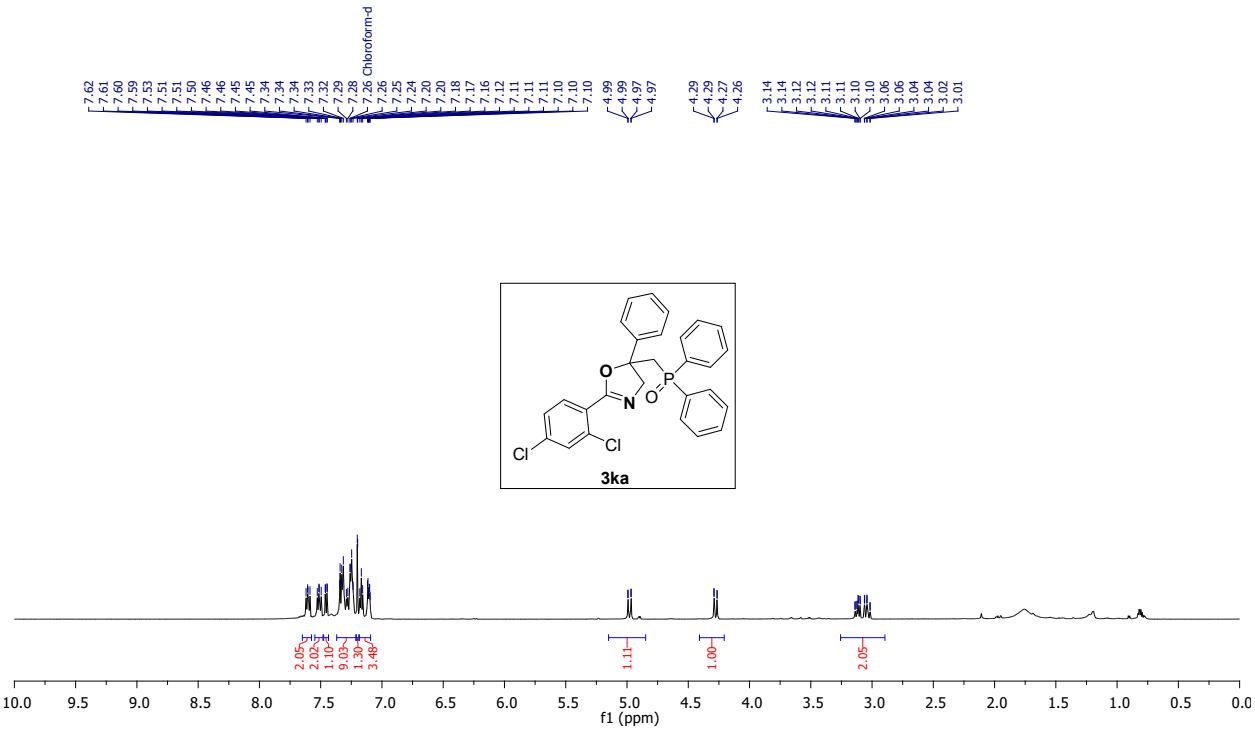

$^{1}$H NMR (400 MHz) spectrum of **3ja** in $CDCl_3$



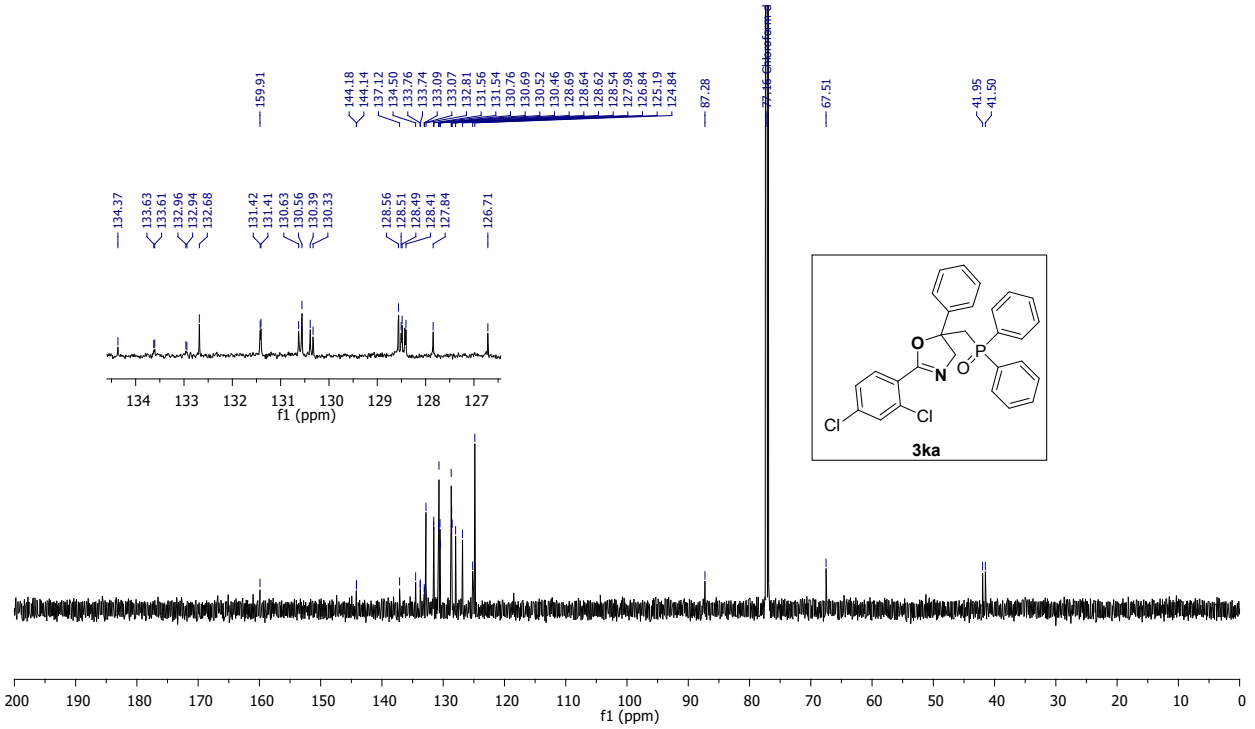

$^{13}$C {$^{1}$H} NMR (101 MHz) spectrum of **3ja** in $CDCl_3$



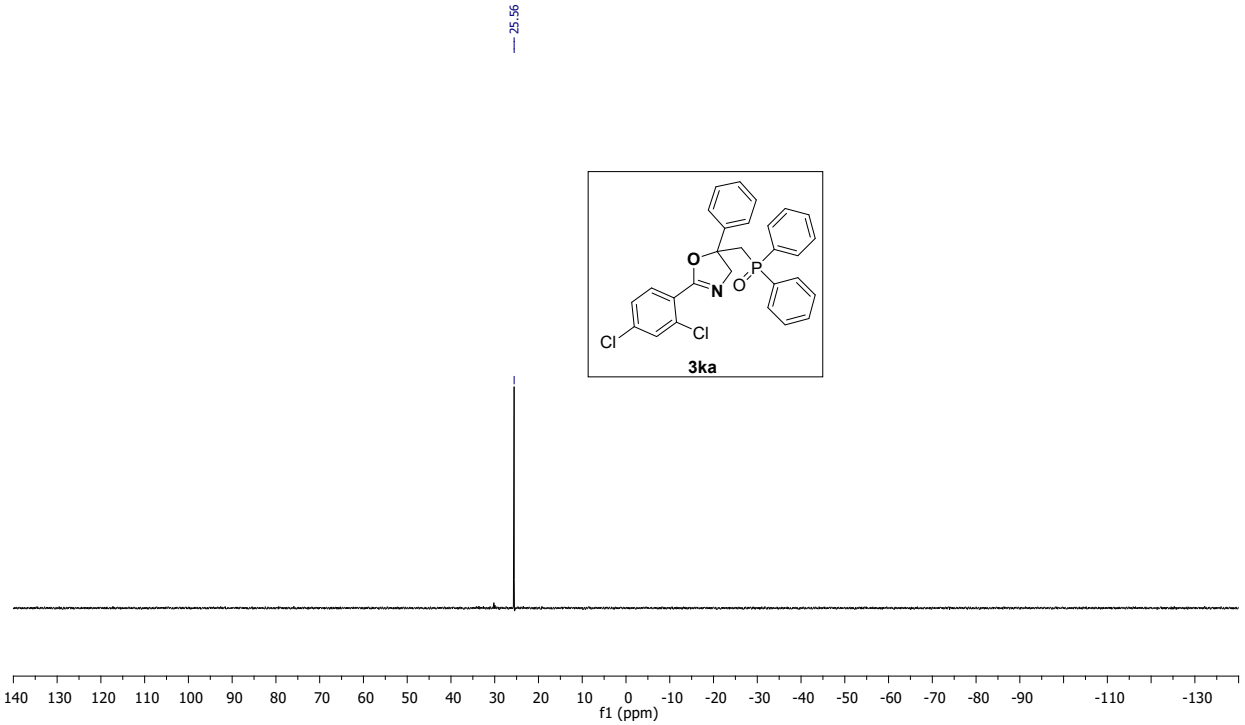

$^{31}$P NMR (243 MHz) spectrum of **3ja** in $CDCl_3$



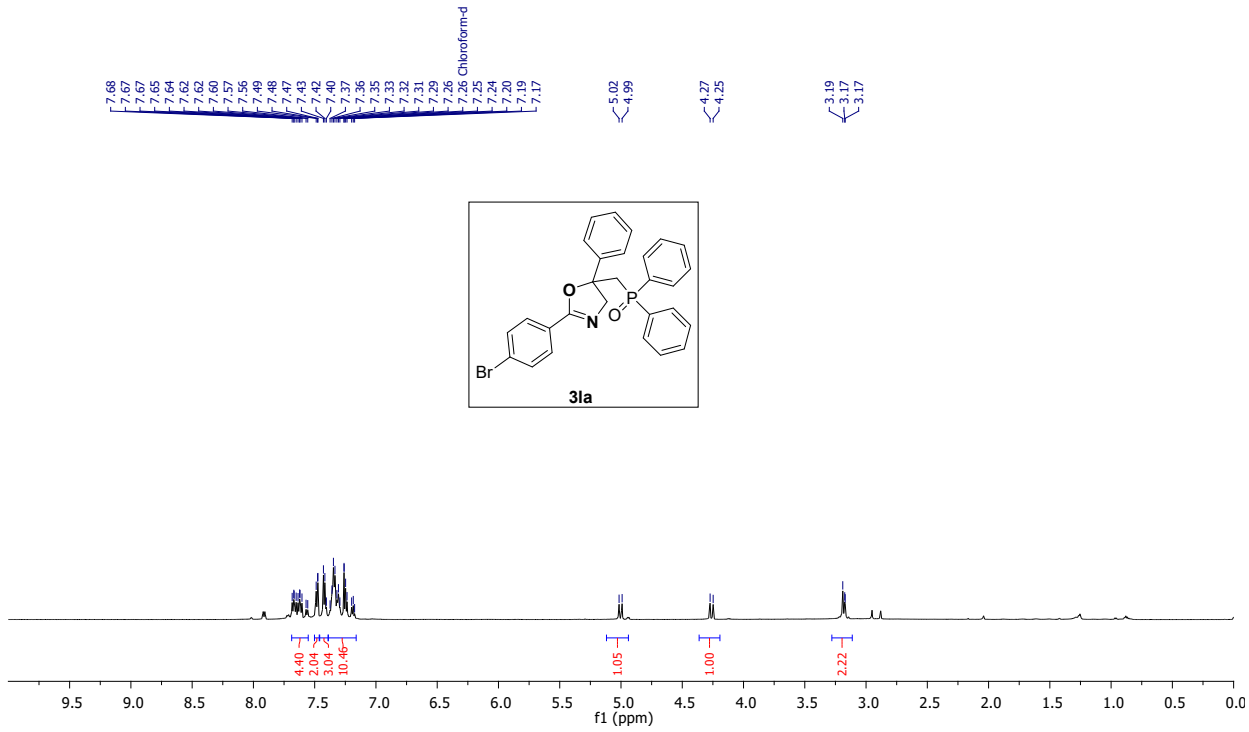

$^{1}$H NMR (400 MHz) spectrum of **3ka** in $CDCl_3$



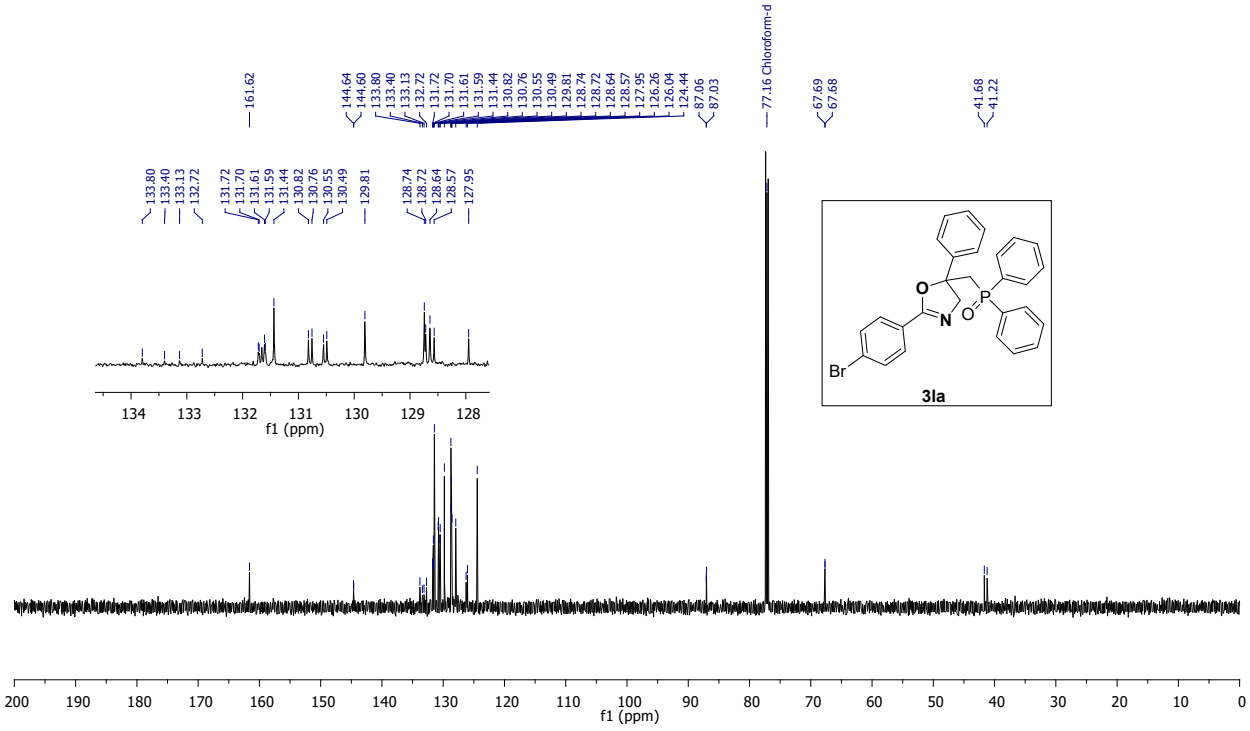

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3ka** in $CDCl_3$



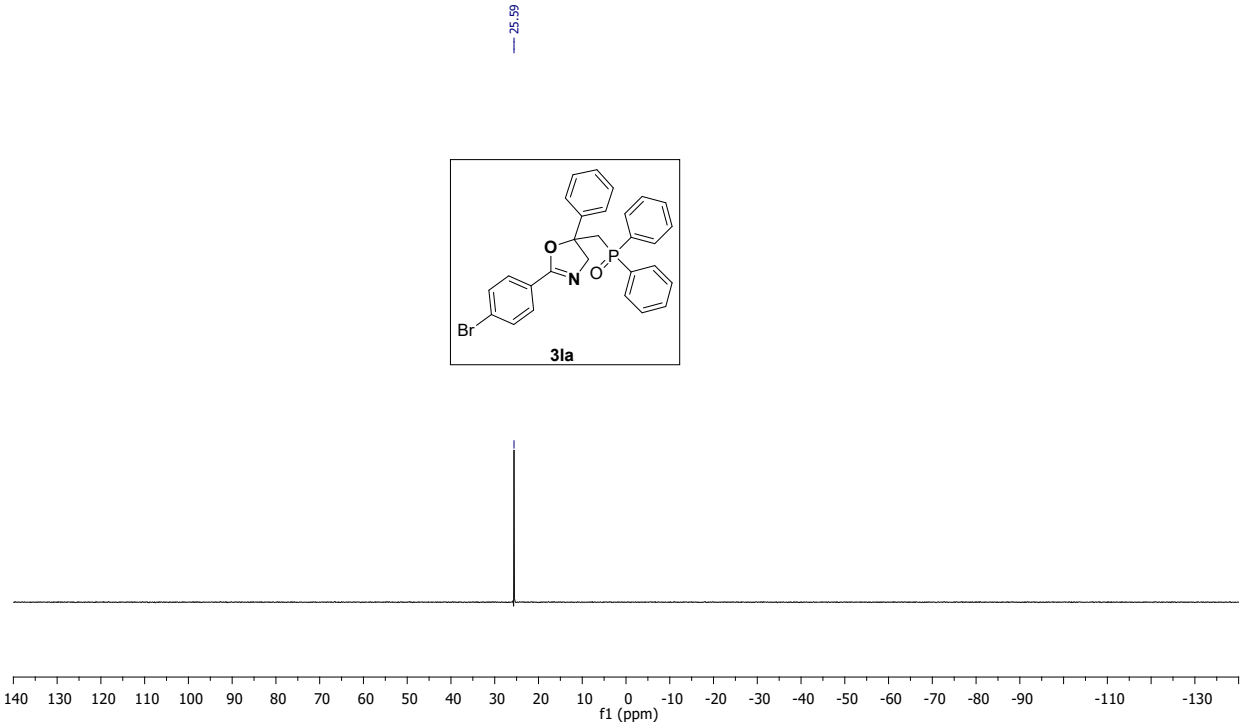

$^{31}$P NMR (243 MHz) spectrum of **3ka** in $CDCl_3$



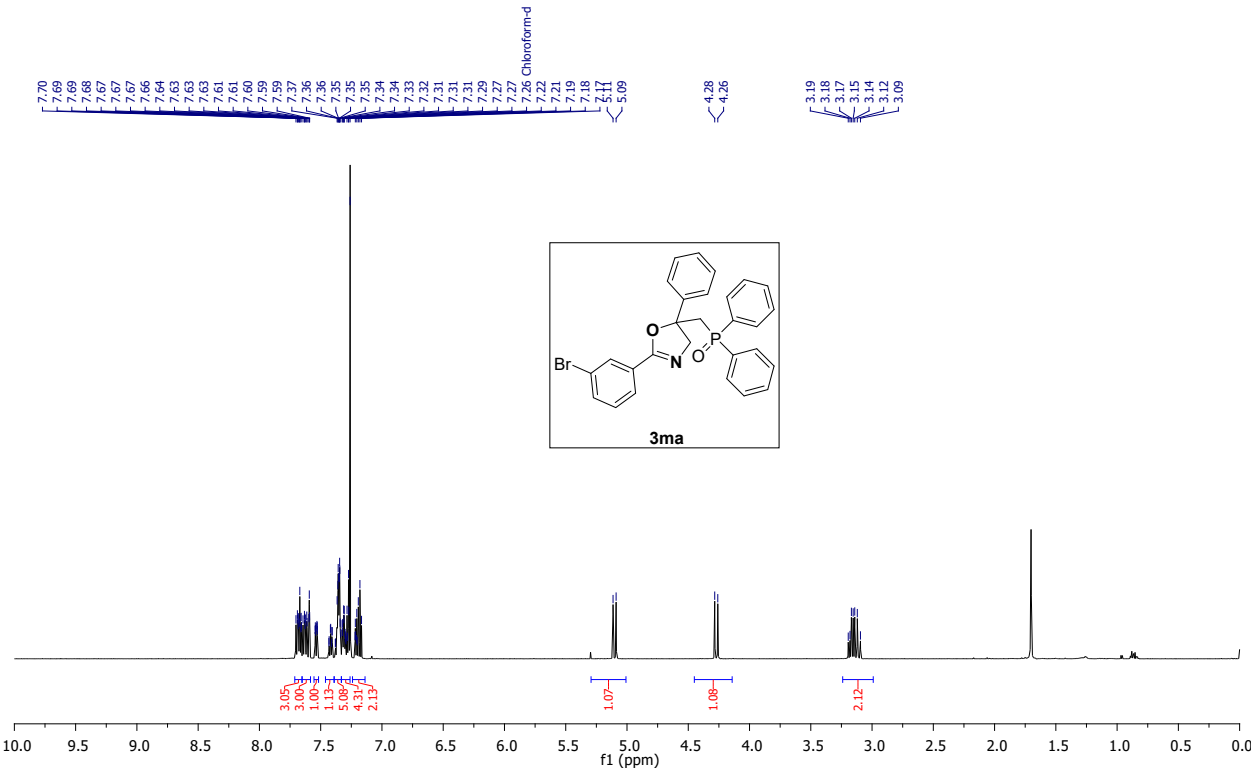

$^{1}$H NMR (400 MHz) spectrum of **3la** in $CDCl_3$



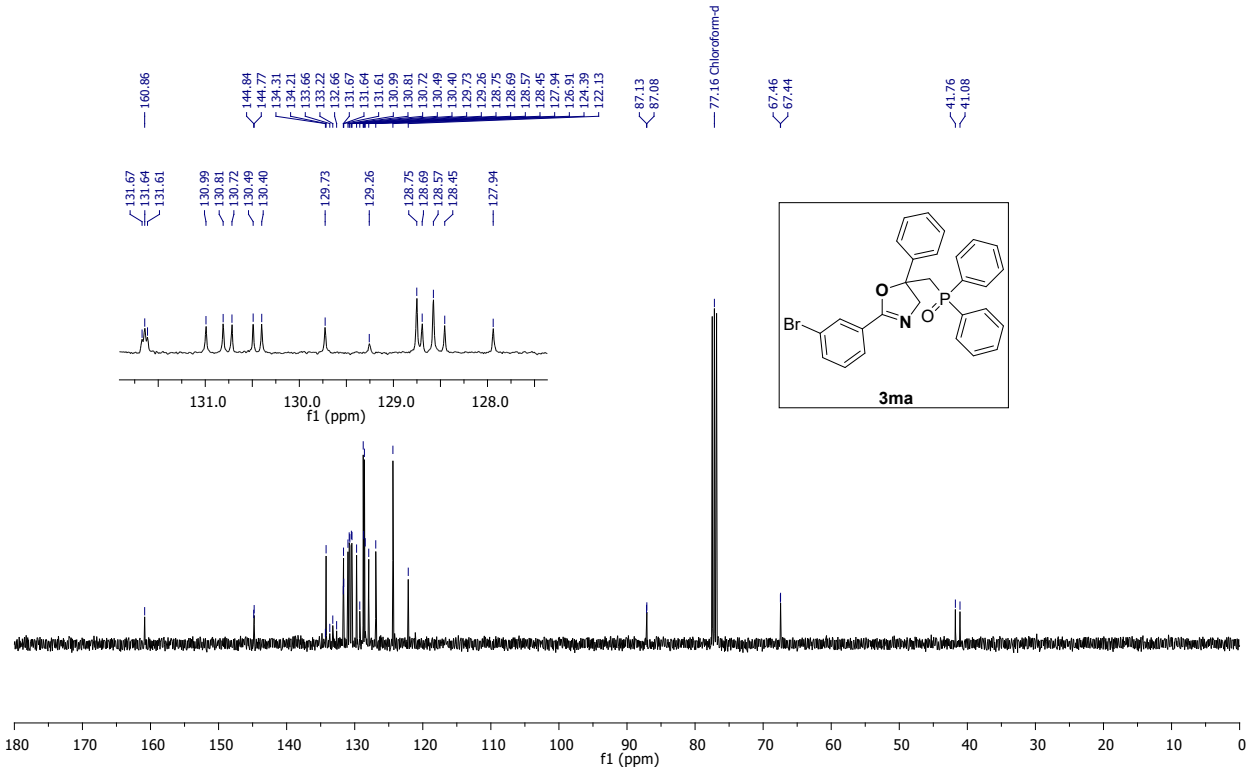

$^{13}$C {$^{1}$H} NMR (101 MHz) spectrum of **3la** in $CDCl_3$



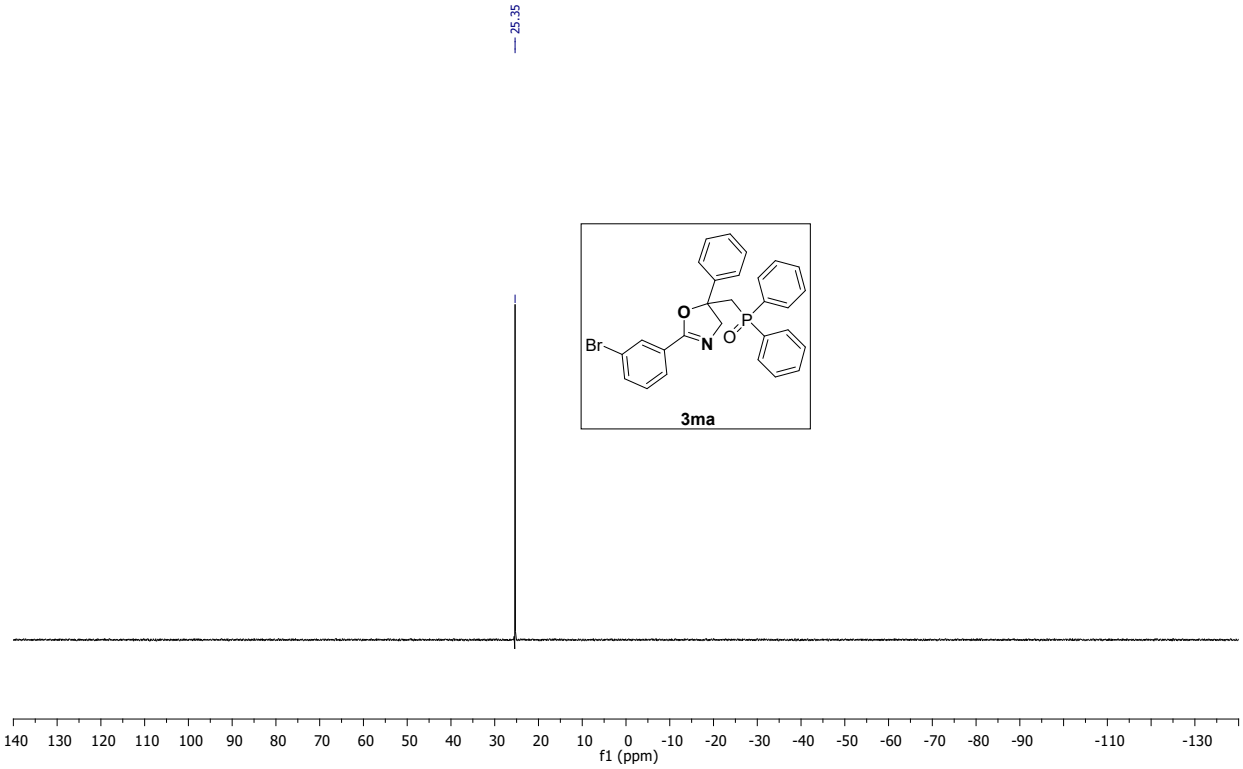

$^{31}$P NMR (243 MHz) spectrum of **3la** in $CDCl_3$



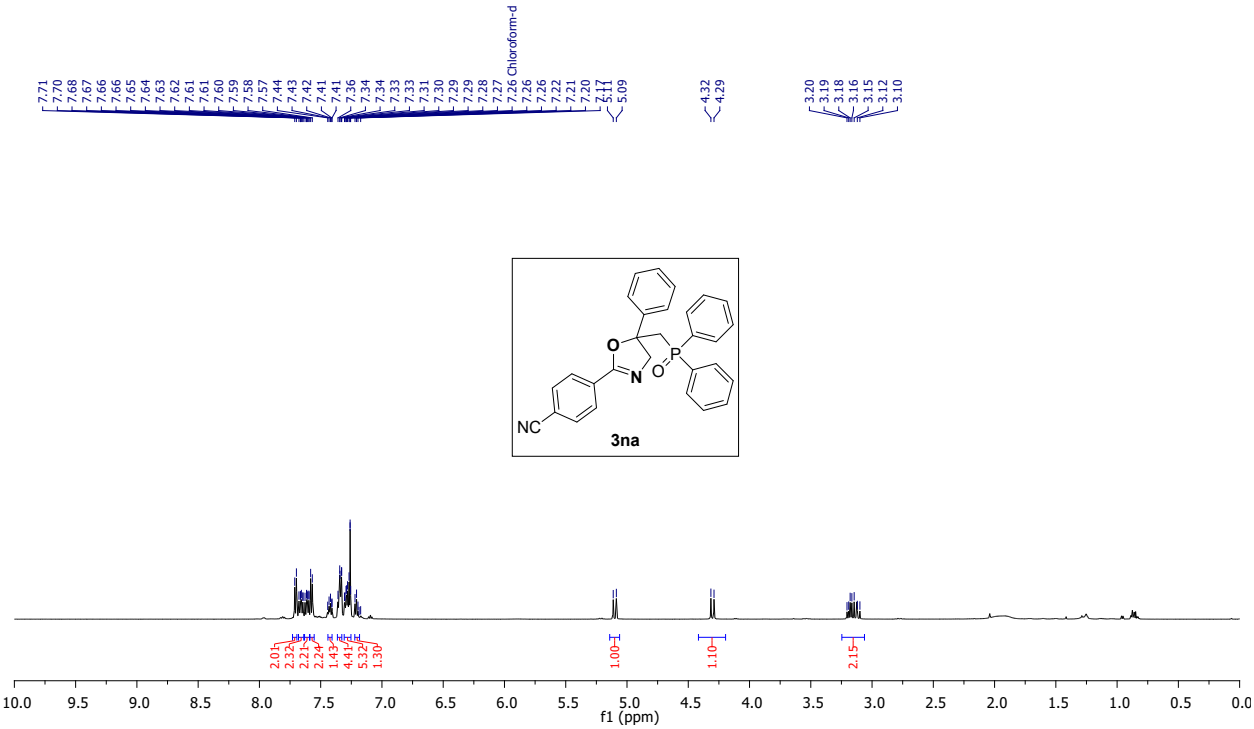

$^{1}$H NMR (600 MHz) spectrum of **3ma** in $CDCl_3$



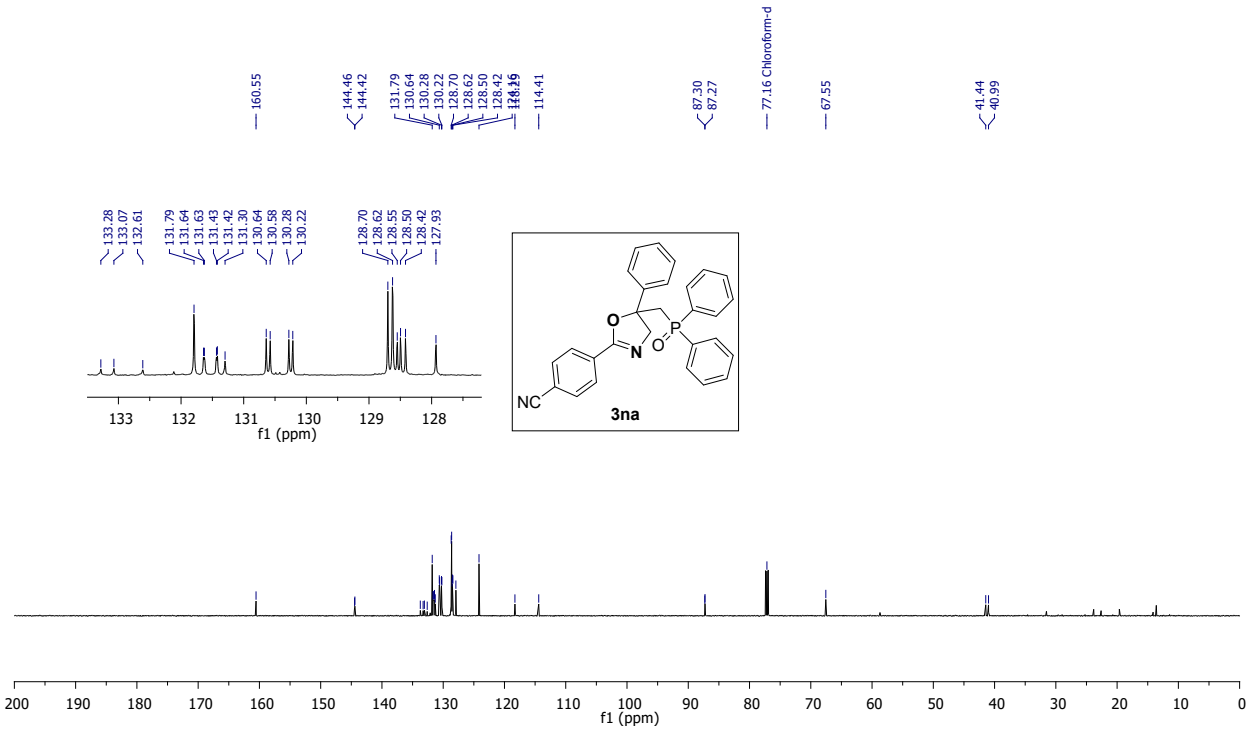

$^{13}$C {$^{1}$H} NMR (101 MHz) spectrum of **3ma** in $CDCl_3$



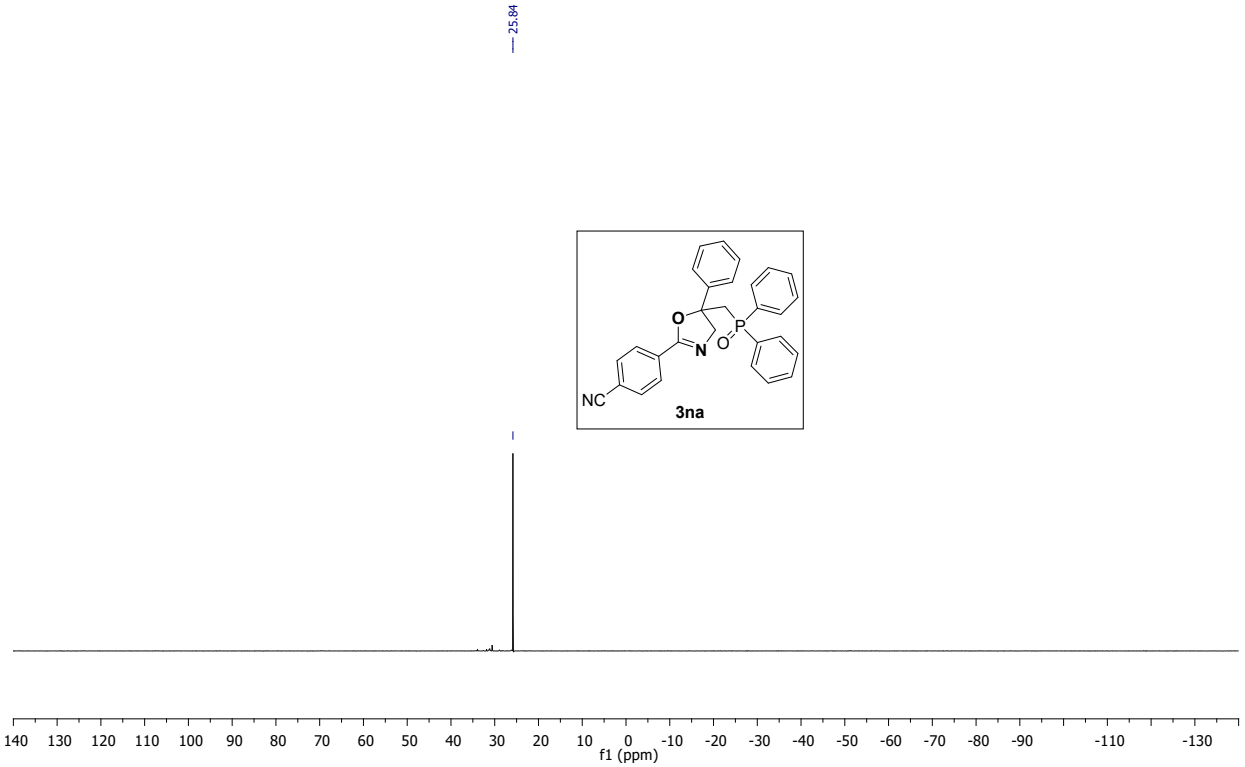

$^{31}$P NMR (243 MHz) spectrum of **3ma** in $CDCl_3$



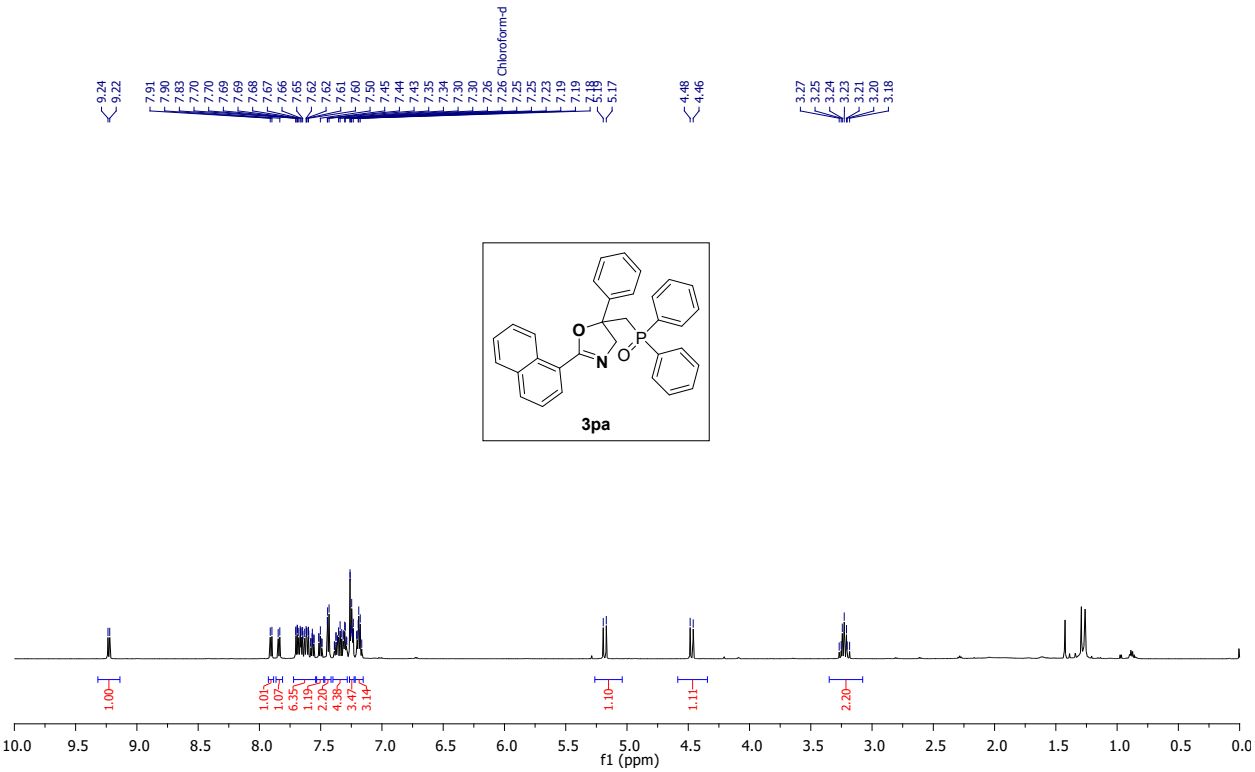

$^{1}$H NMR (400 MHz) spectrum of **3na** in $CDCl_3$



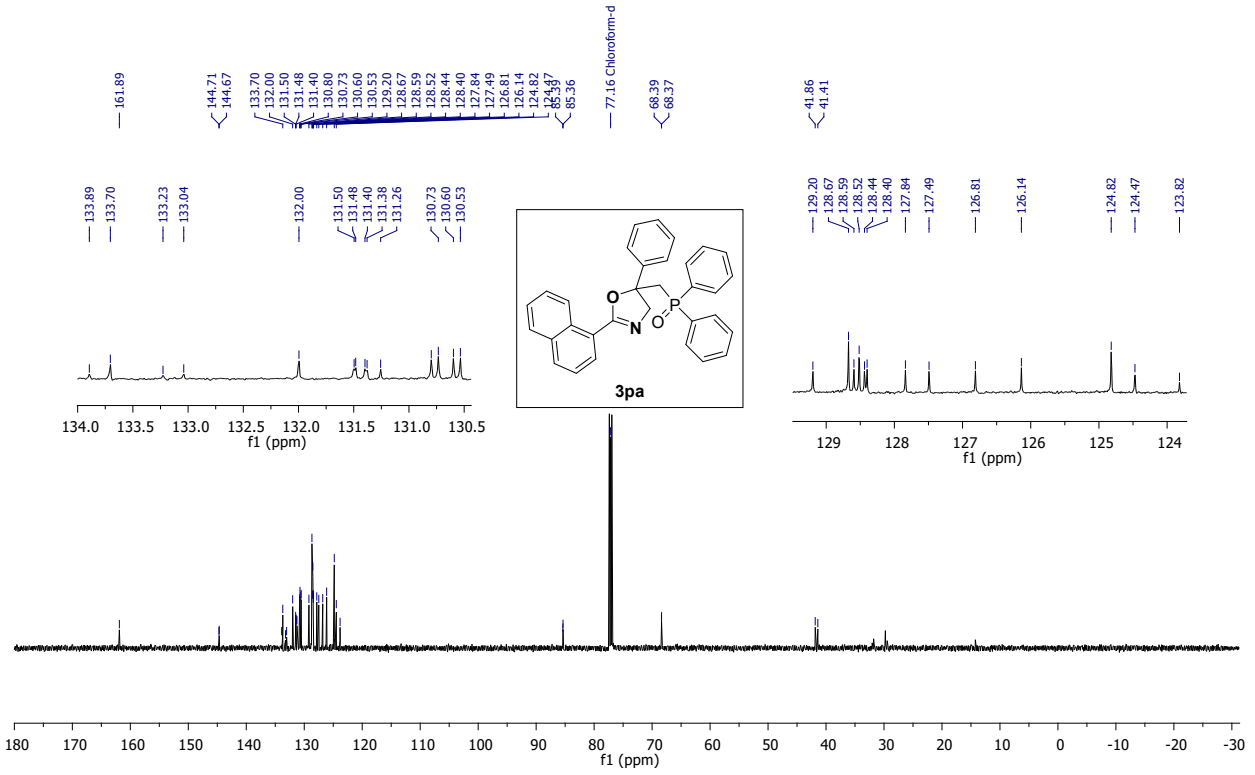

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3na** in $CDCl_3$



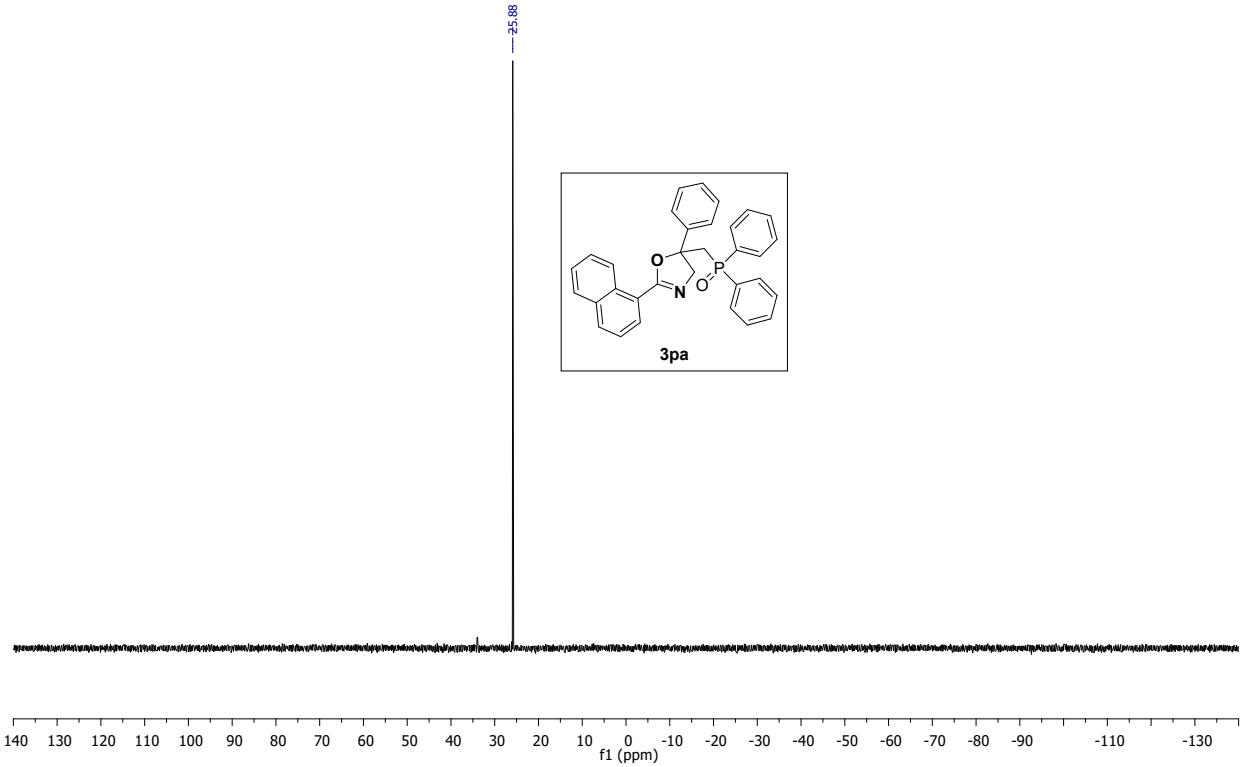

$^{31}$P NMR (243 MHz) spectrum of **3na** in $CDCl_3$



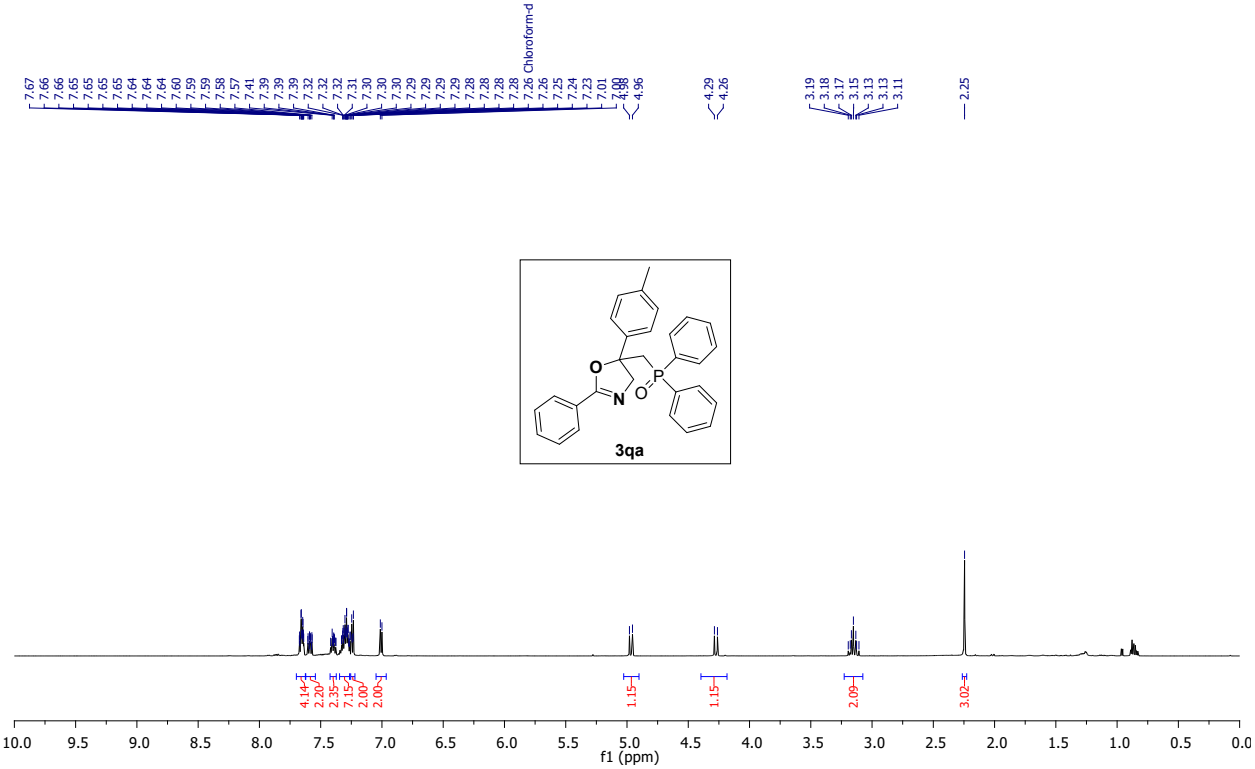

$^{1}$H NMR (600 MHz) spectrum of **3pa** in $CDCl_3$



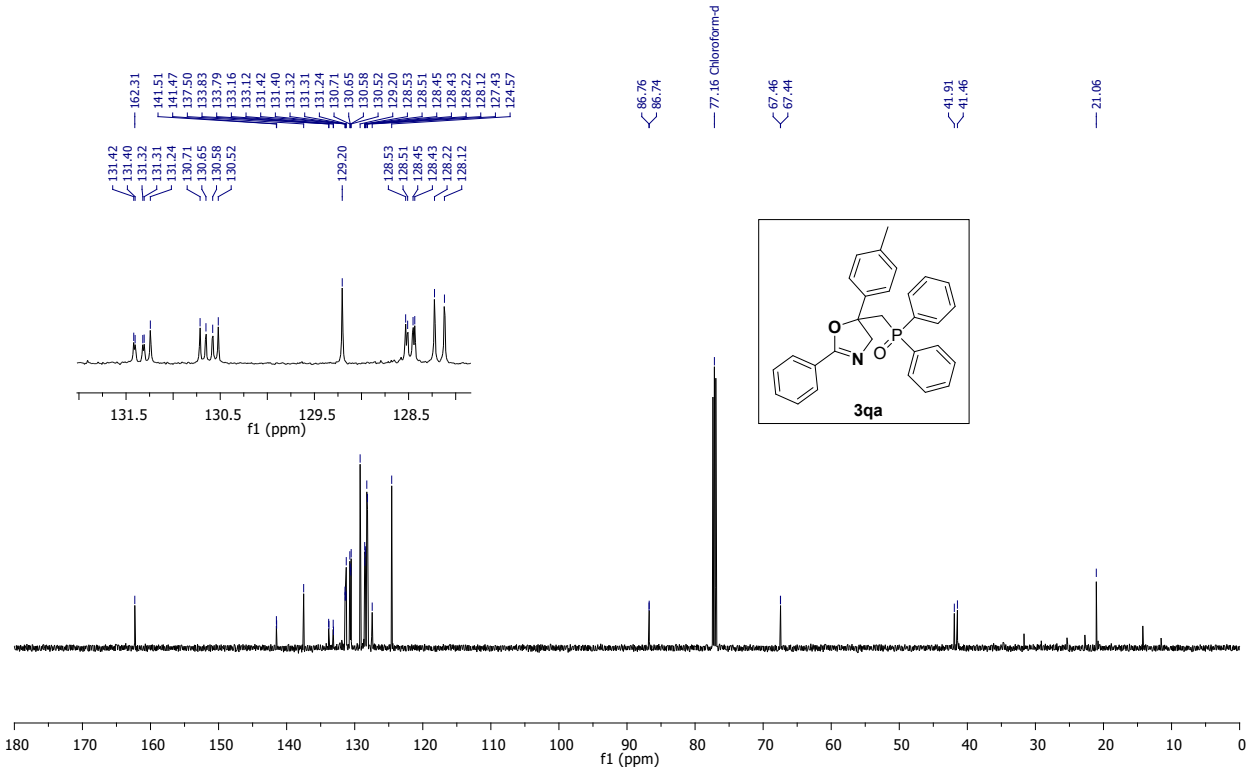

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3pa** in $CDCl_3$



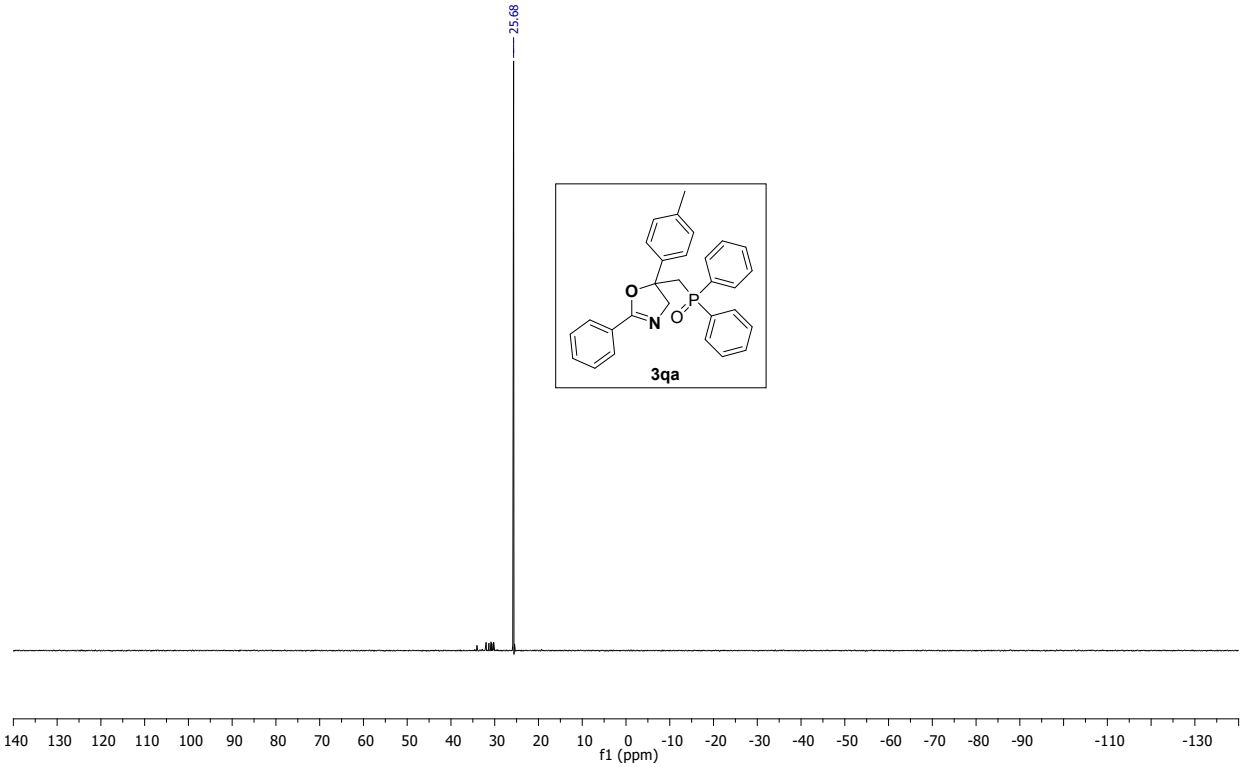

$^{31}$P NMR (243 MHz) spectrum of **3pa** in $CDCl_3$



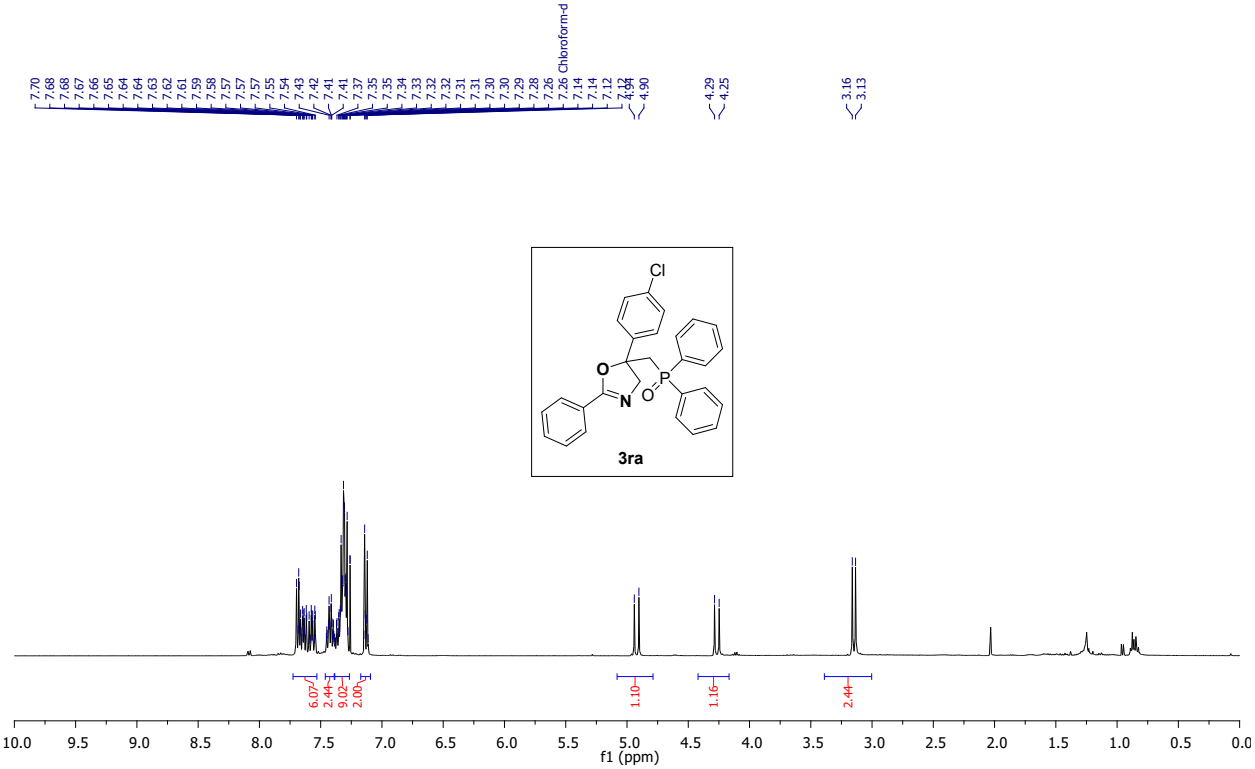

$^{1}$H NMR (600 MHz) spectrum of **3qa** in $CDCl_3$



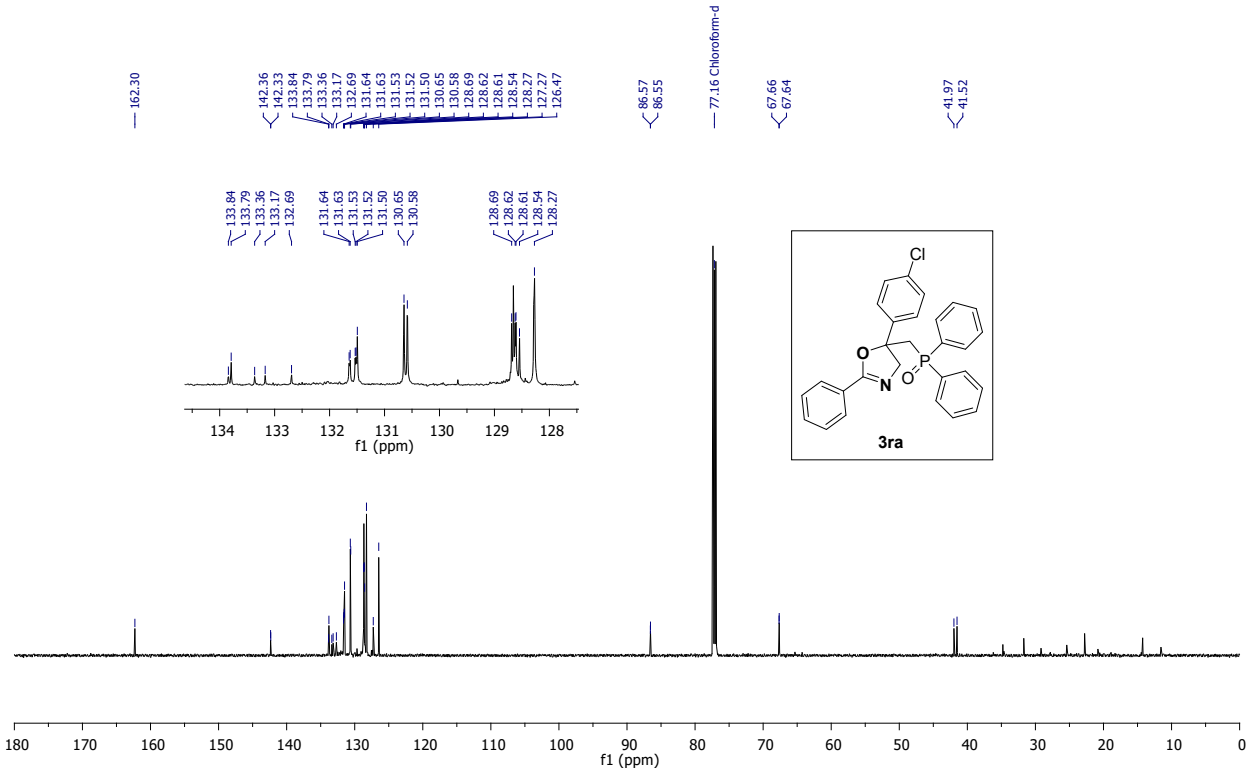

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3qa** in $CDCl_3$



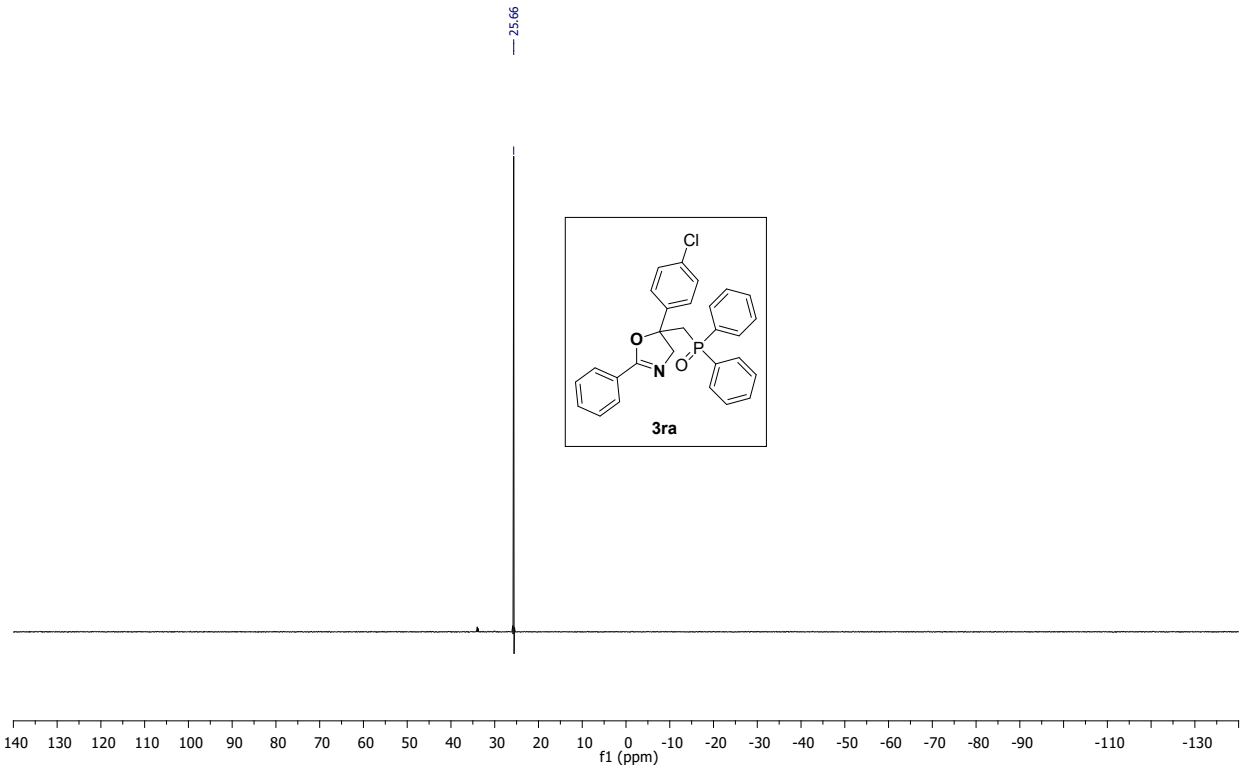

$^{31}$P NMR (243 MHz) spectrum of **3qa** in $CDCl_3$



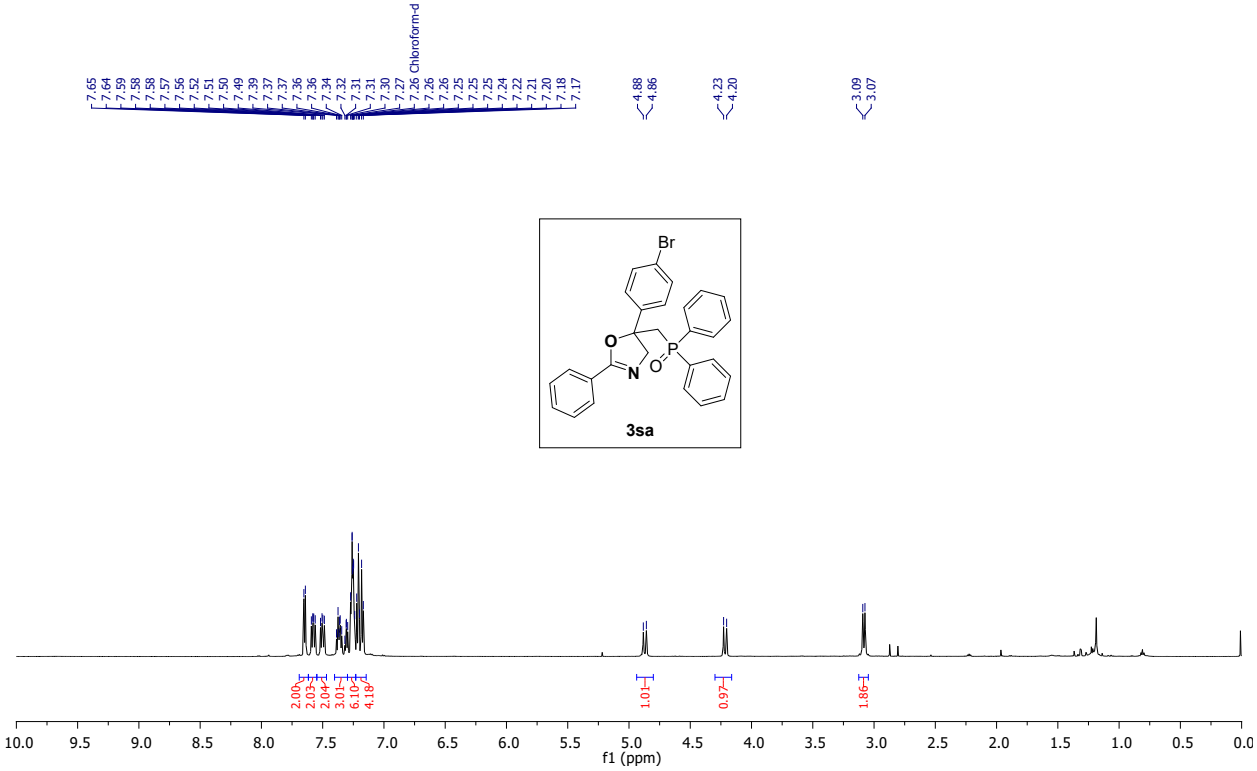

$^{1}$H NMR (600 MHz) spectrum of **3ra** in $CDCl_3$



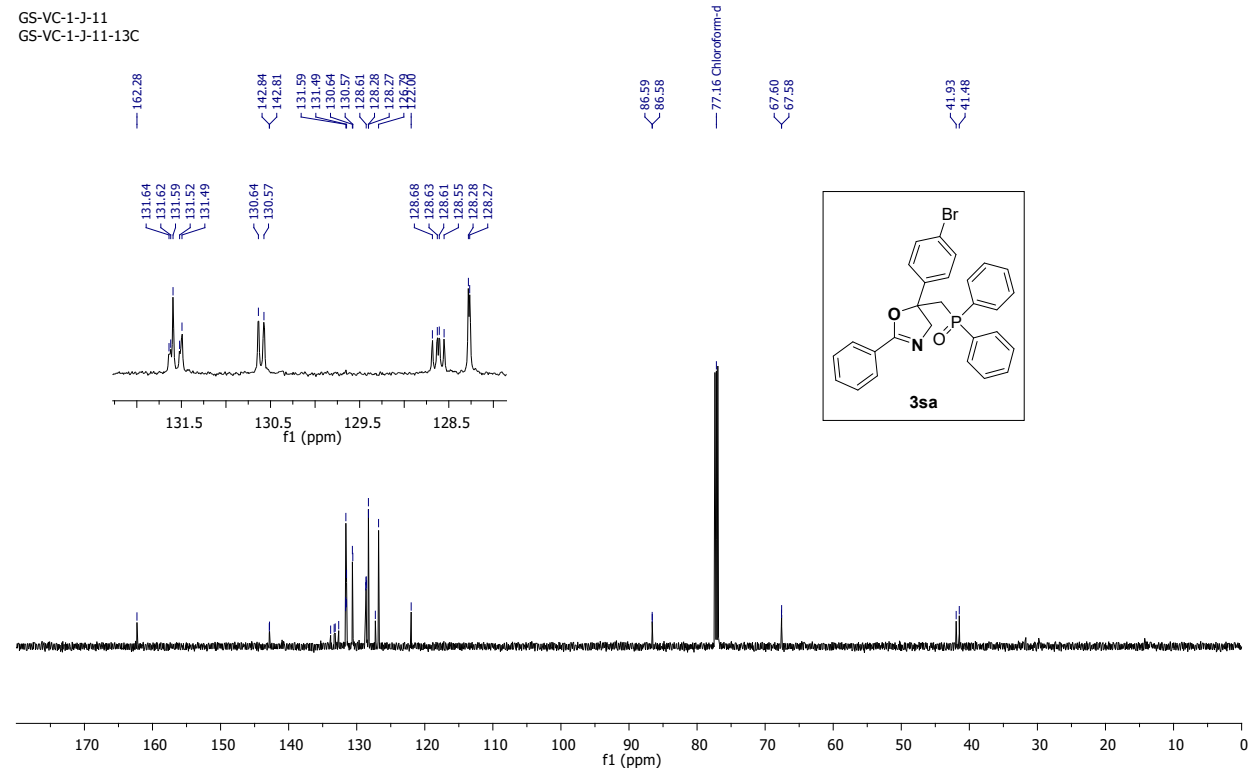

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3ra** in $CDCl_3$



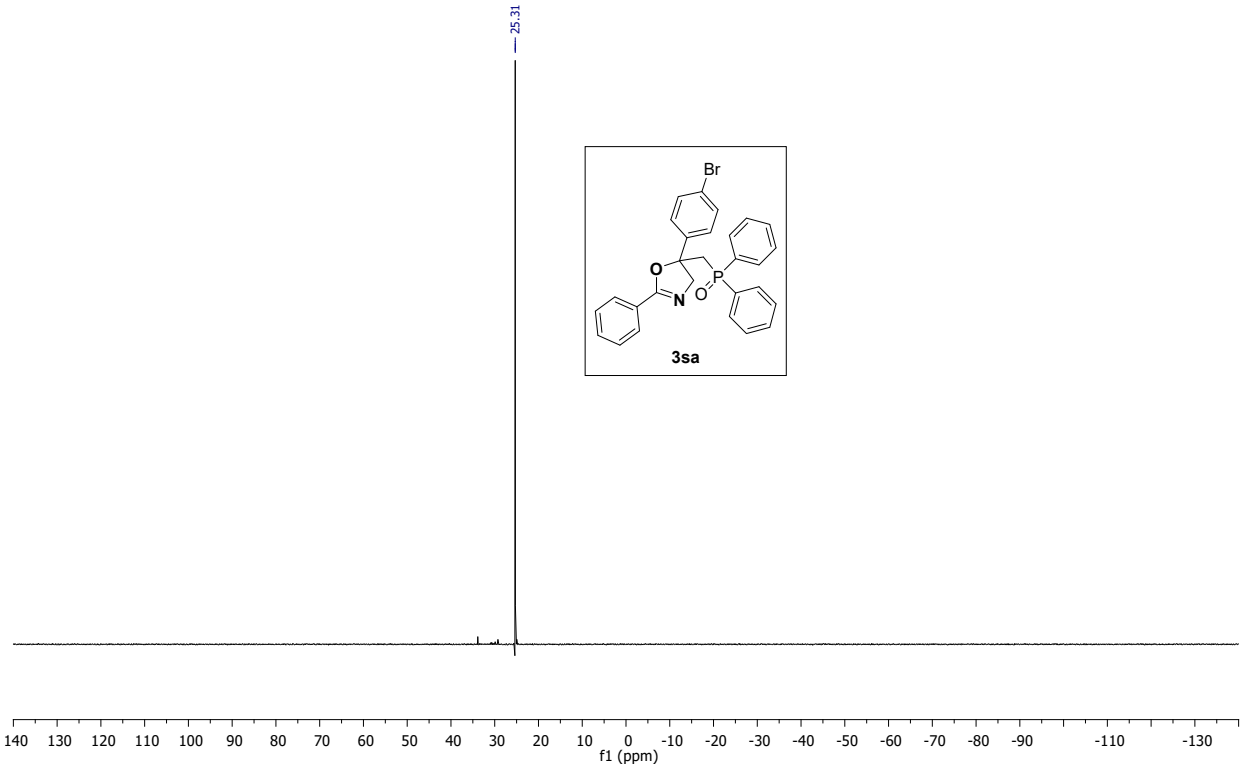

$^{31}$P NMR (243 MHz) spectrum of **3ra** in $CDCl_3$



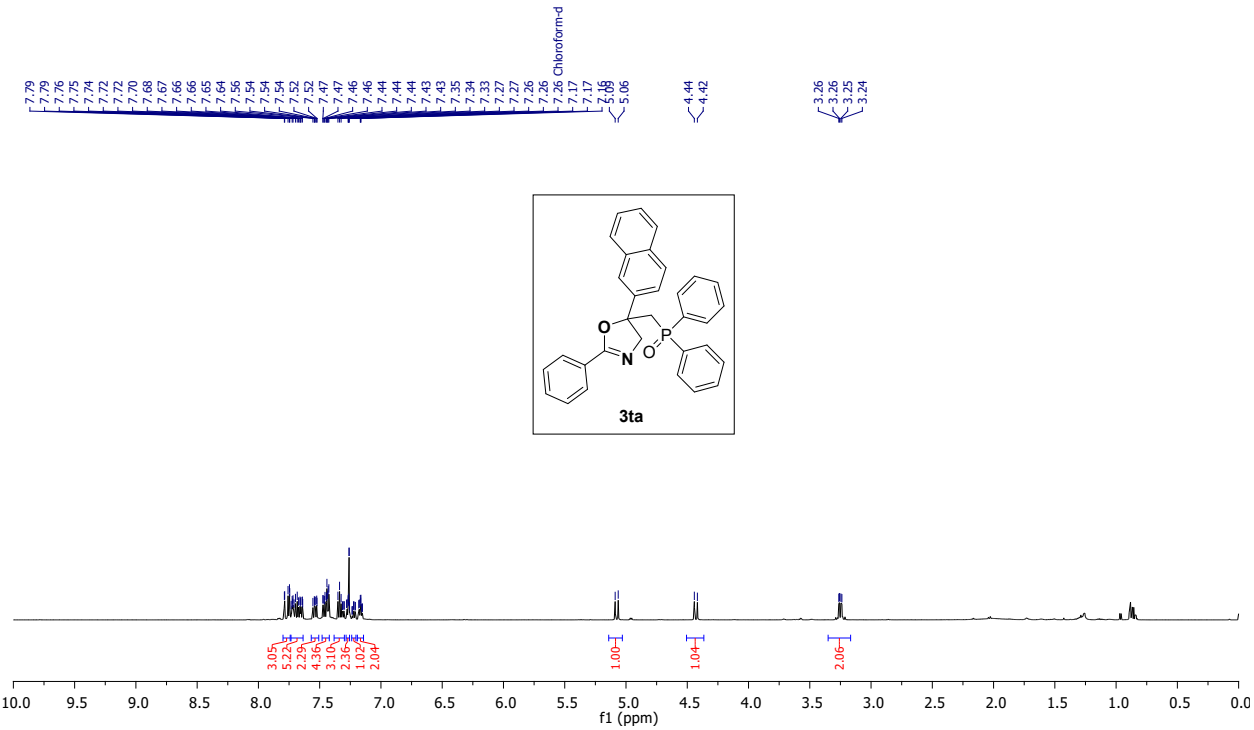

$^{1}$H NMR (600 MHz) spectrum of **3sa** in $CDCl_3$



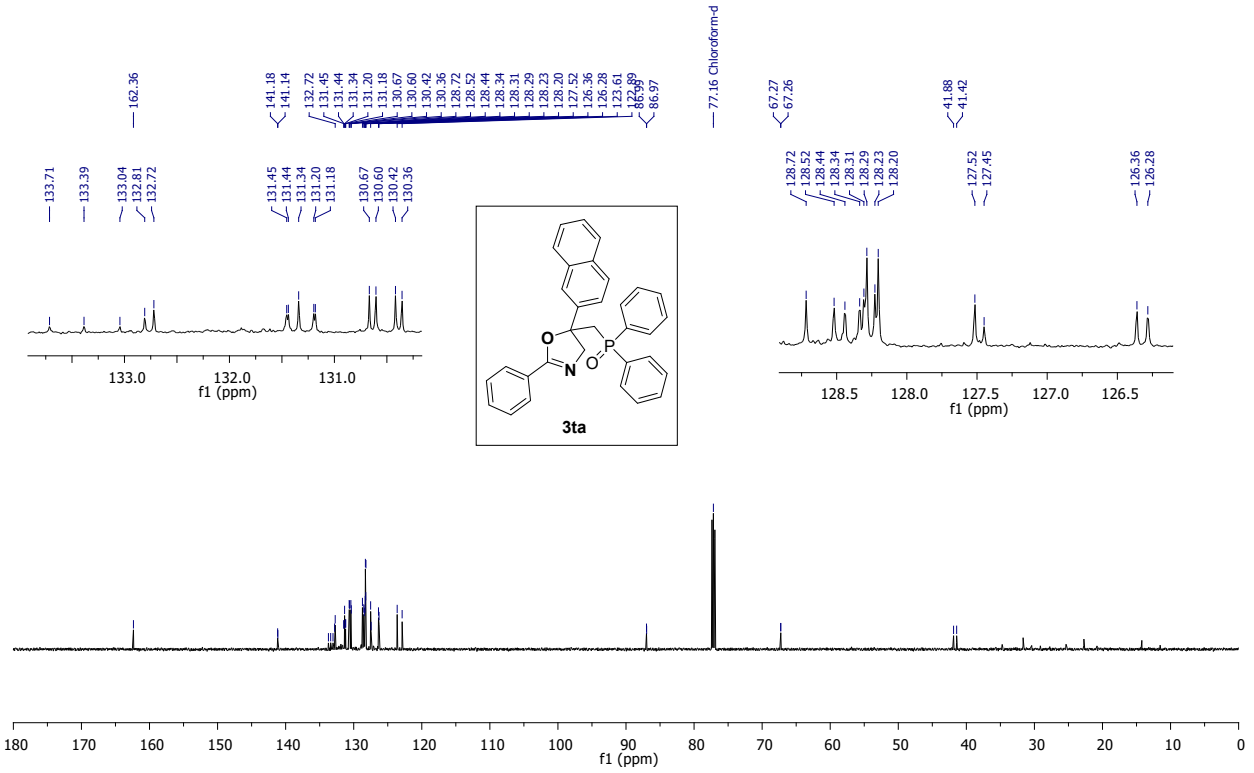

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3sa** in $CDCl_3$



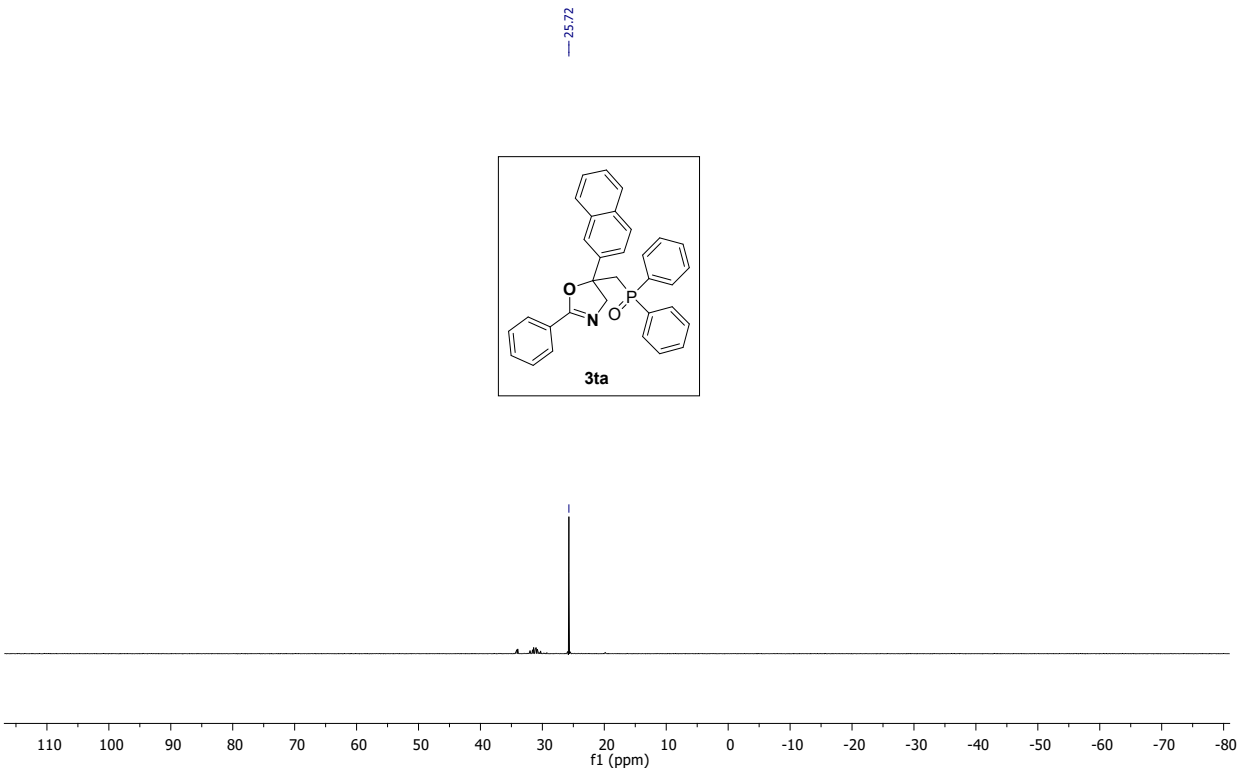

$^{31}P$ NMR (243 MHz) spectrum of **3sa** in $CDCl_3$



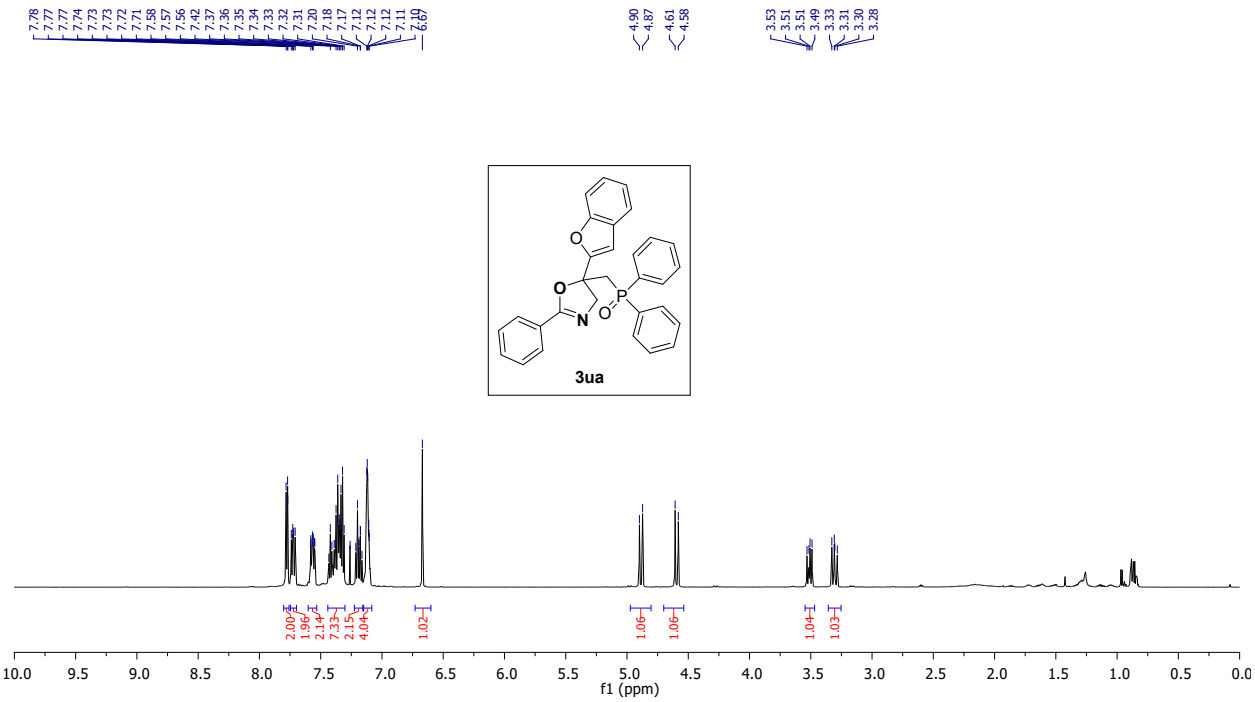

$^{1}$H NMR (400 MHz) spectrum of **3ta** in $CDCl_3$



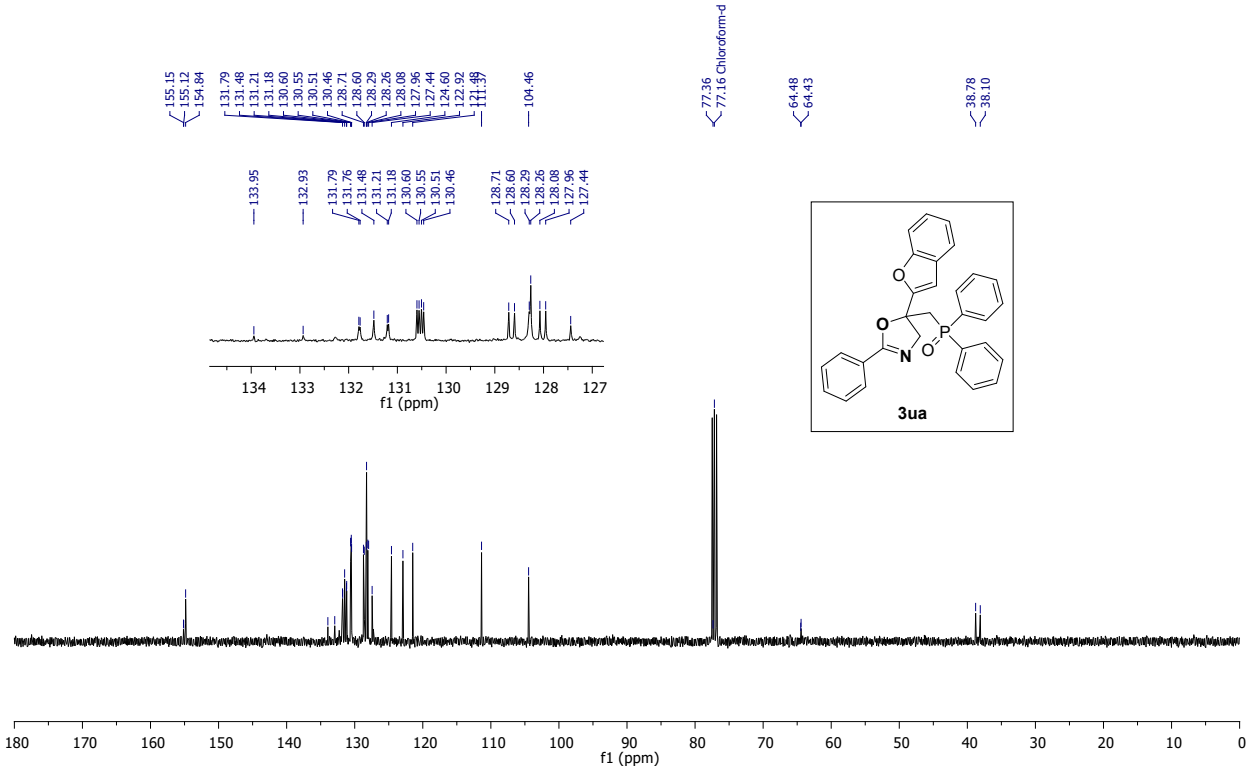

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3ta** in $CDCl_3$



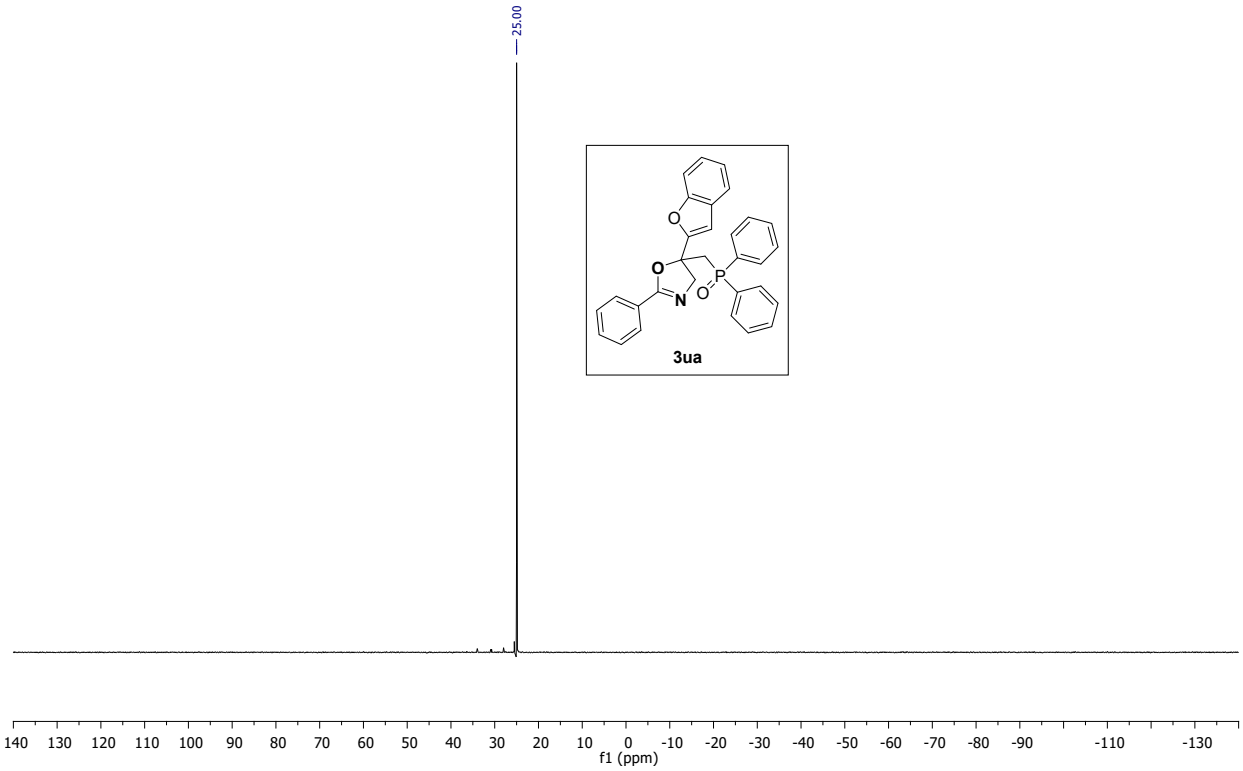

$^{31}P$ NMR (243 MHz) spectrum of **3ta** in $CDCl_3$



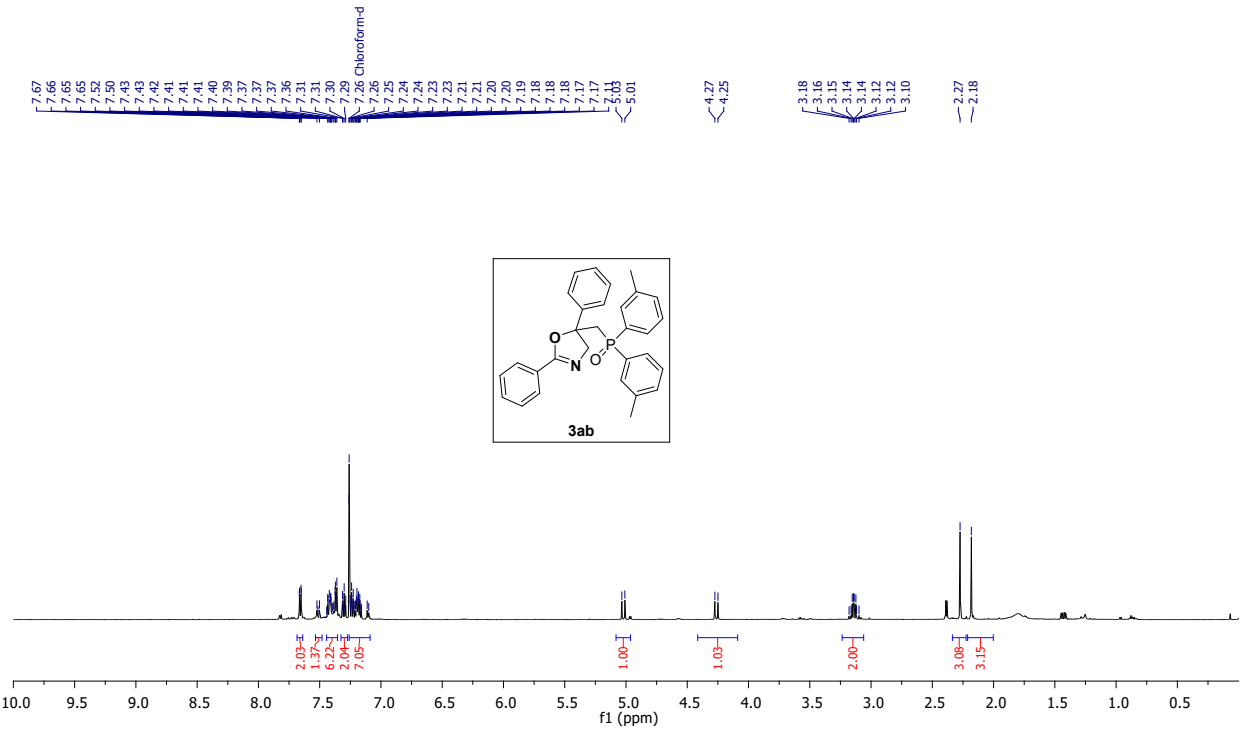

$^{1}$H NMR (600 MHz) spectrum of **3ua** in $CDCl_3$



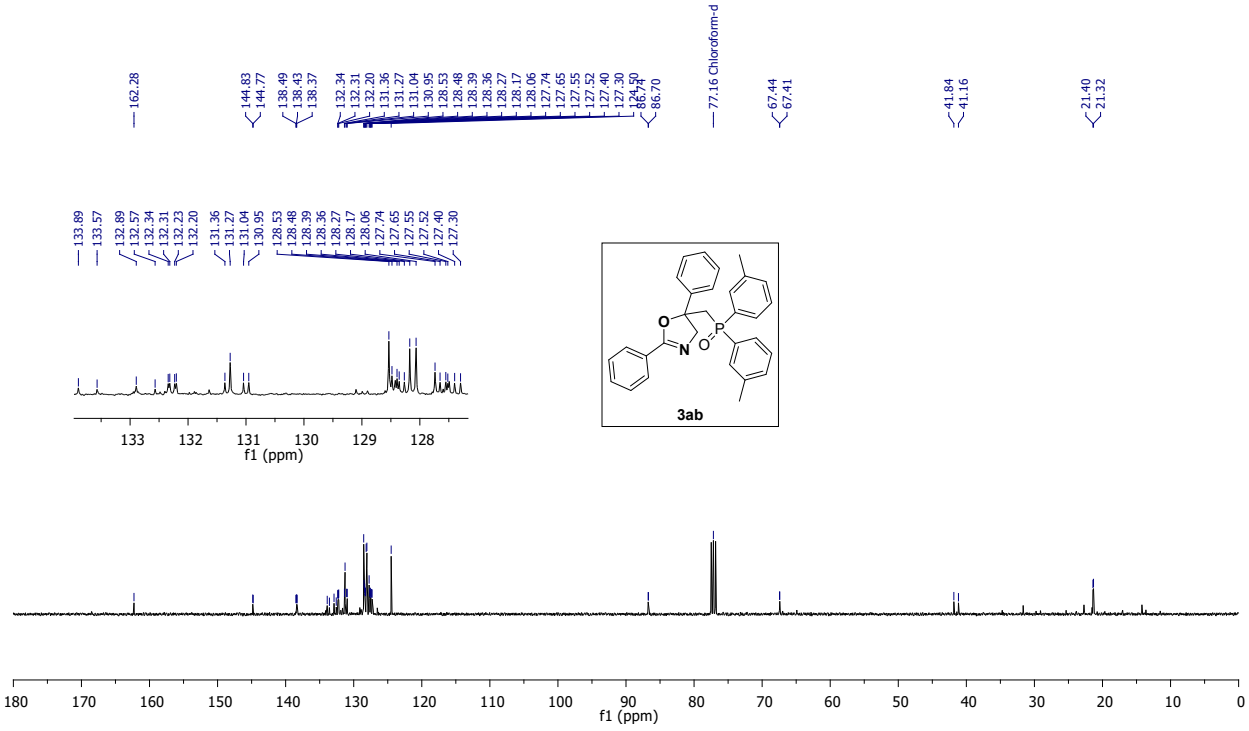

$^{13}$C {$^{1}$H} NMR (101 MHz) spectrum of **3ua** in $CDCl_3$



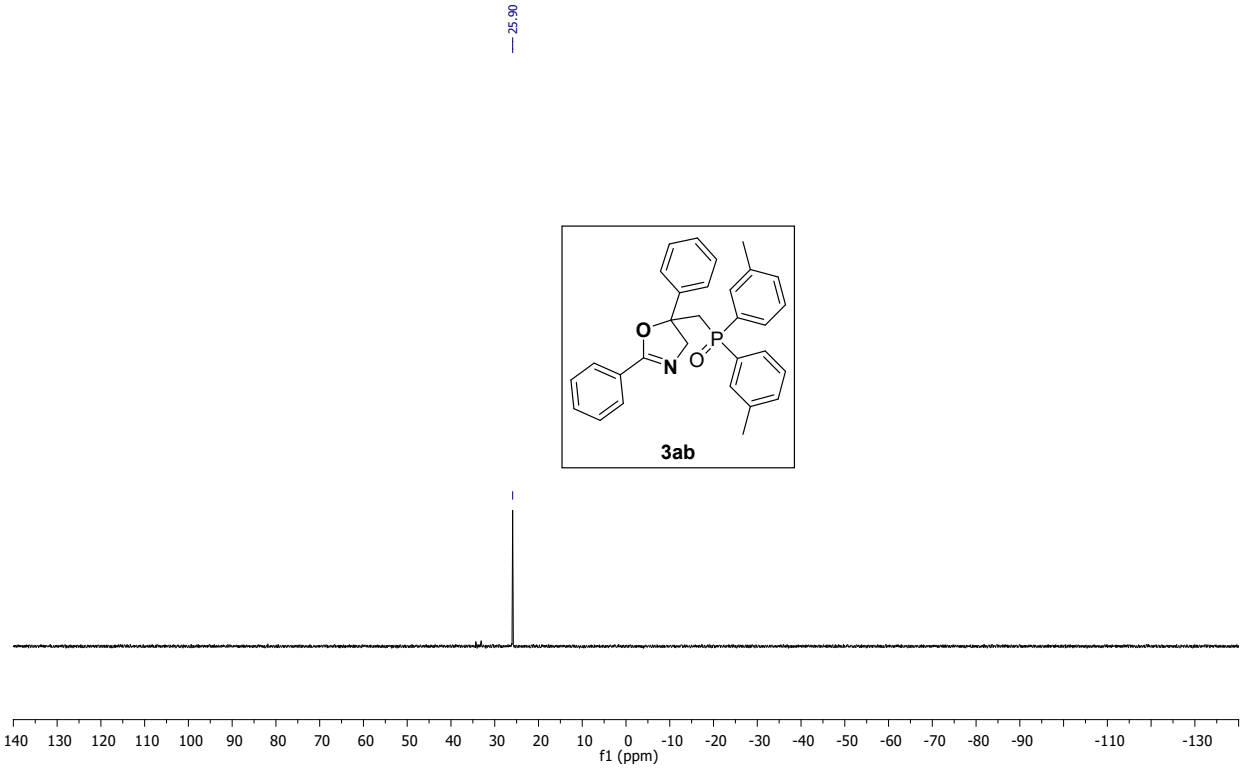

$^{31}$P NMR (243 MHz) spectrum of **3ua** in $CDCl_3$



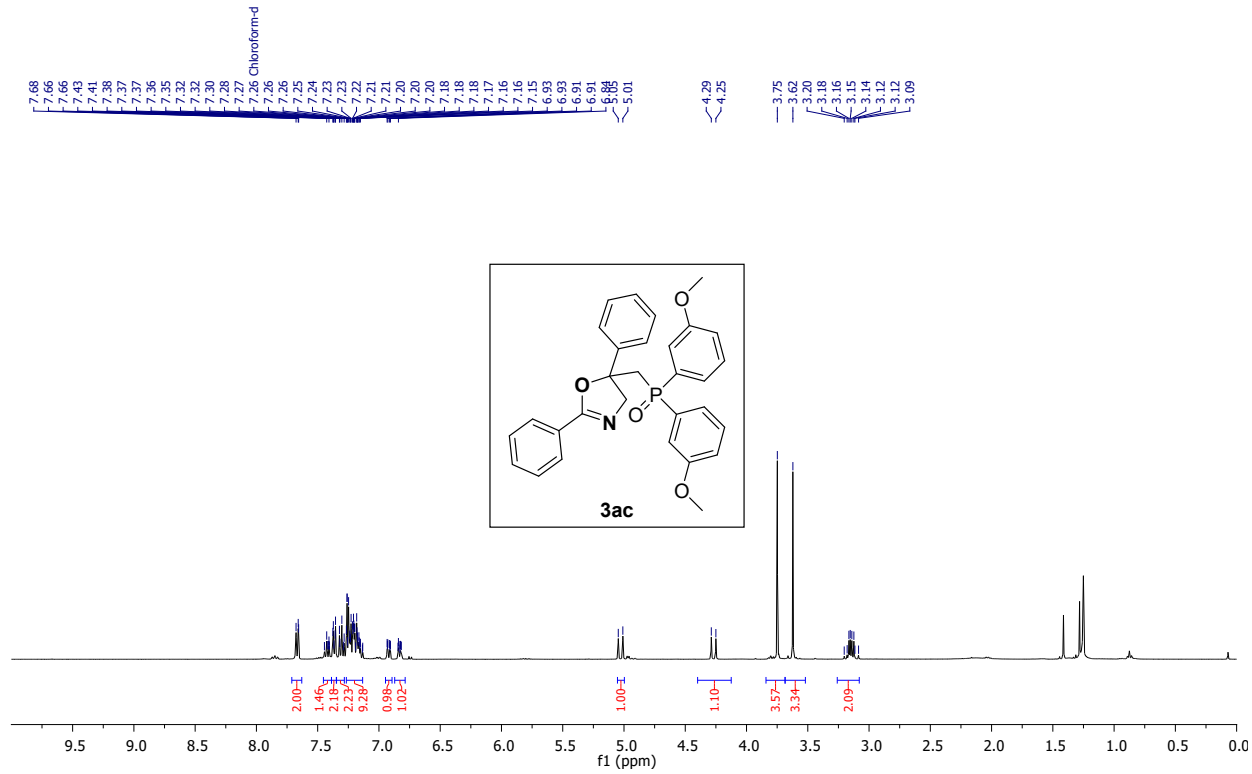

$^{1}$H NMR (400 MHz) spectrum of **3ab** in $CDCl_3$



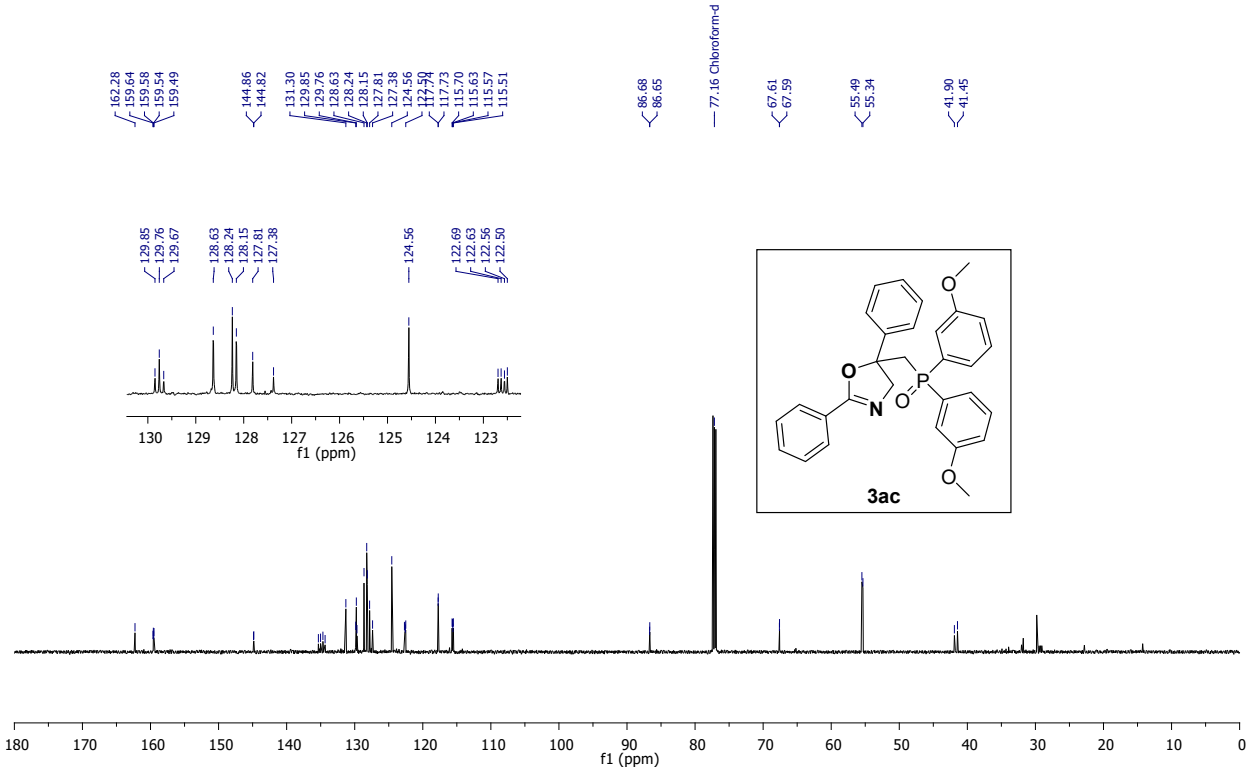

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3ab** in $CDCl_3$



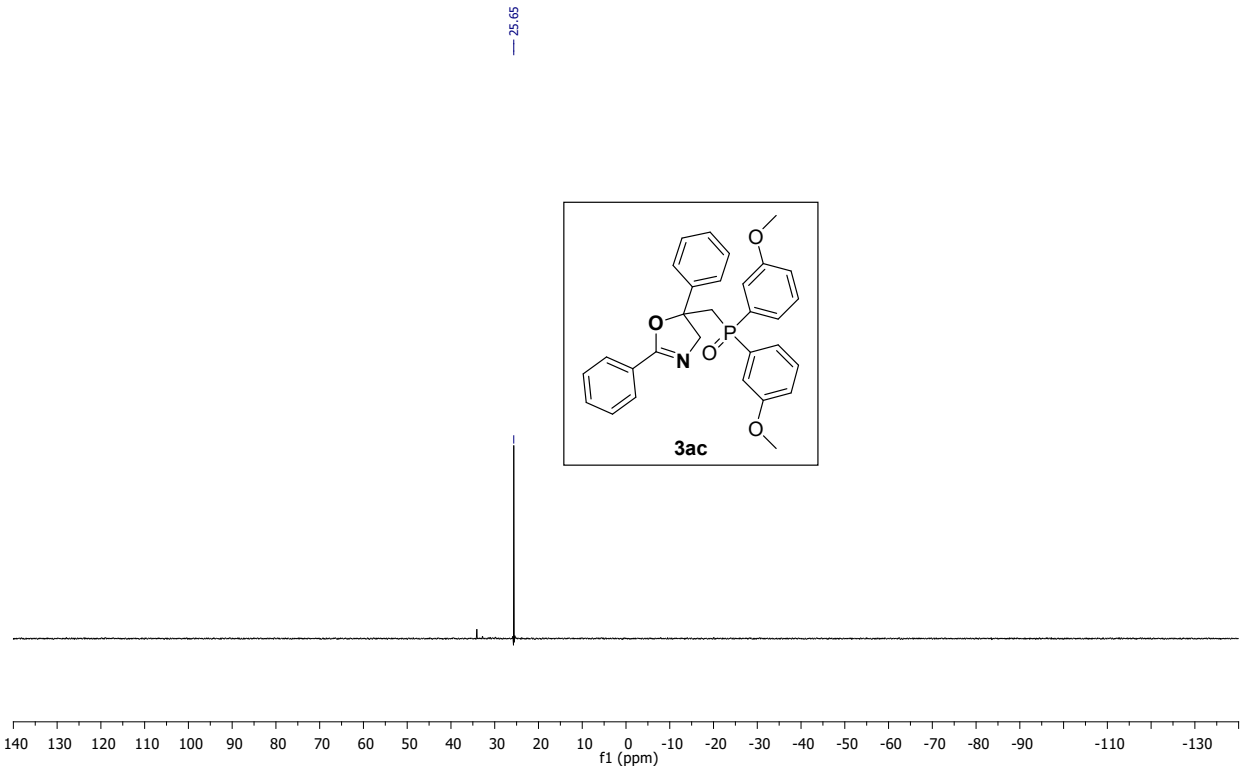

$^{31}$P NMR (243 MHz) spectrum of **3ab** in $CDCl_3$




$^{1}$H NMR (400 MHz) spectrum of **3ac** in $CDCl_3$




$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3ac** in $CDCl_3$




$^{31}$P NMR (243 MHz) spectrum of **3ac** in $CDCl_3$

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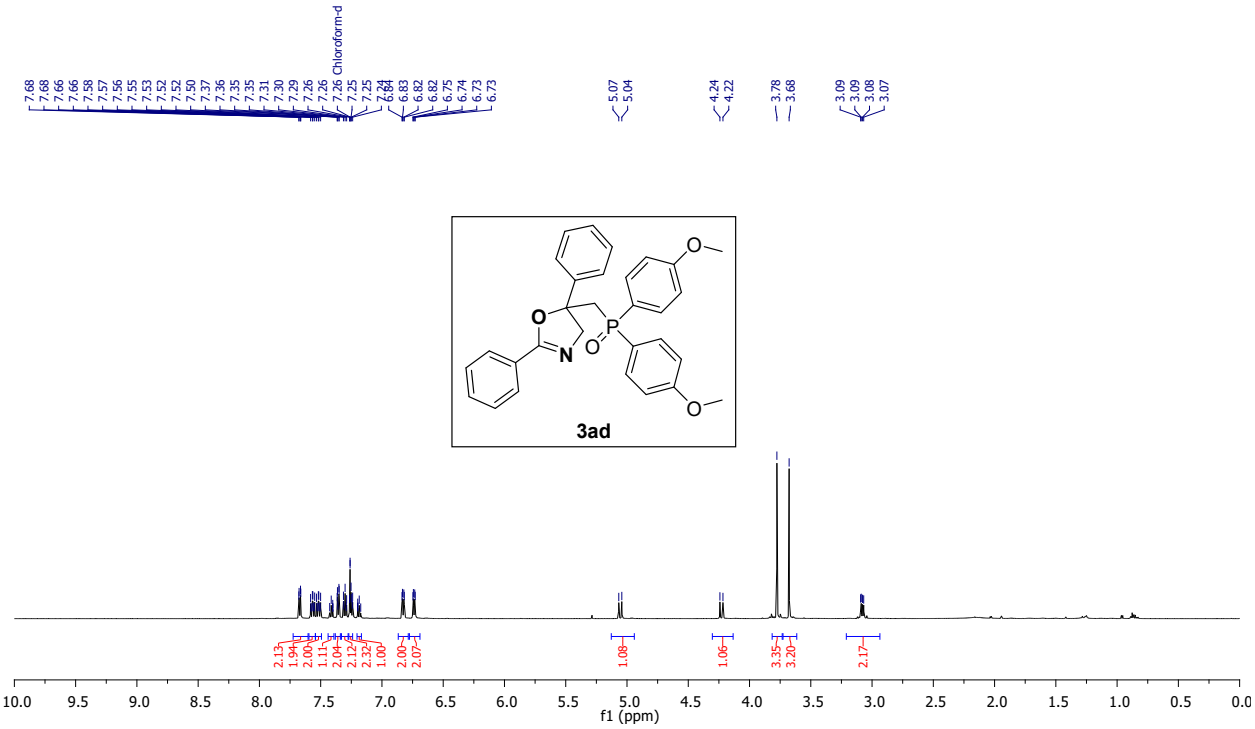
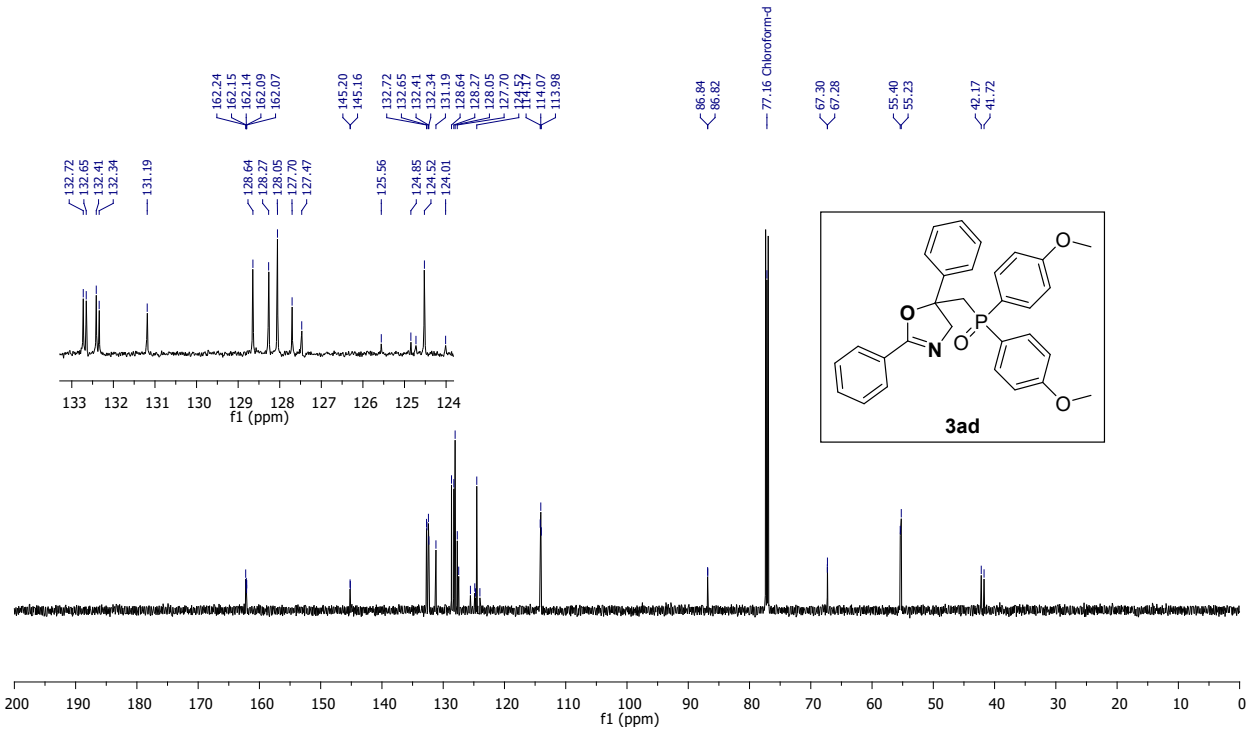
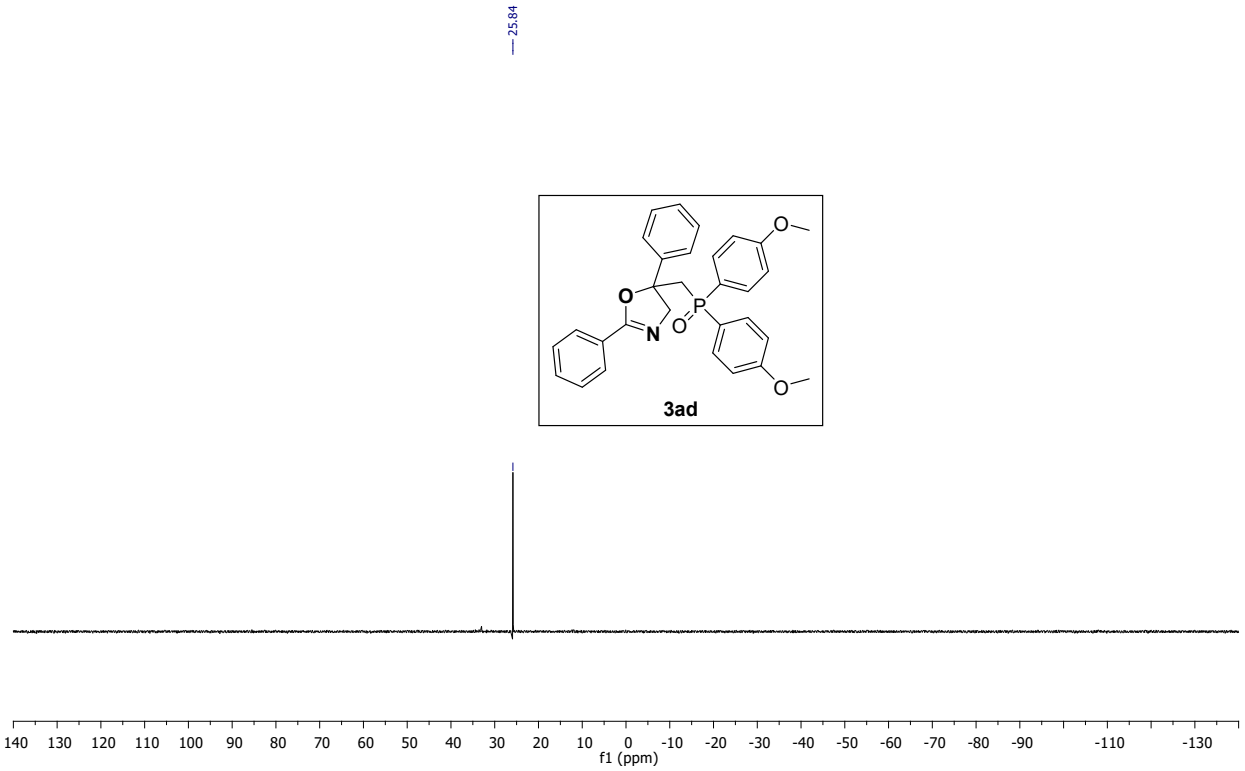


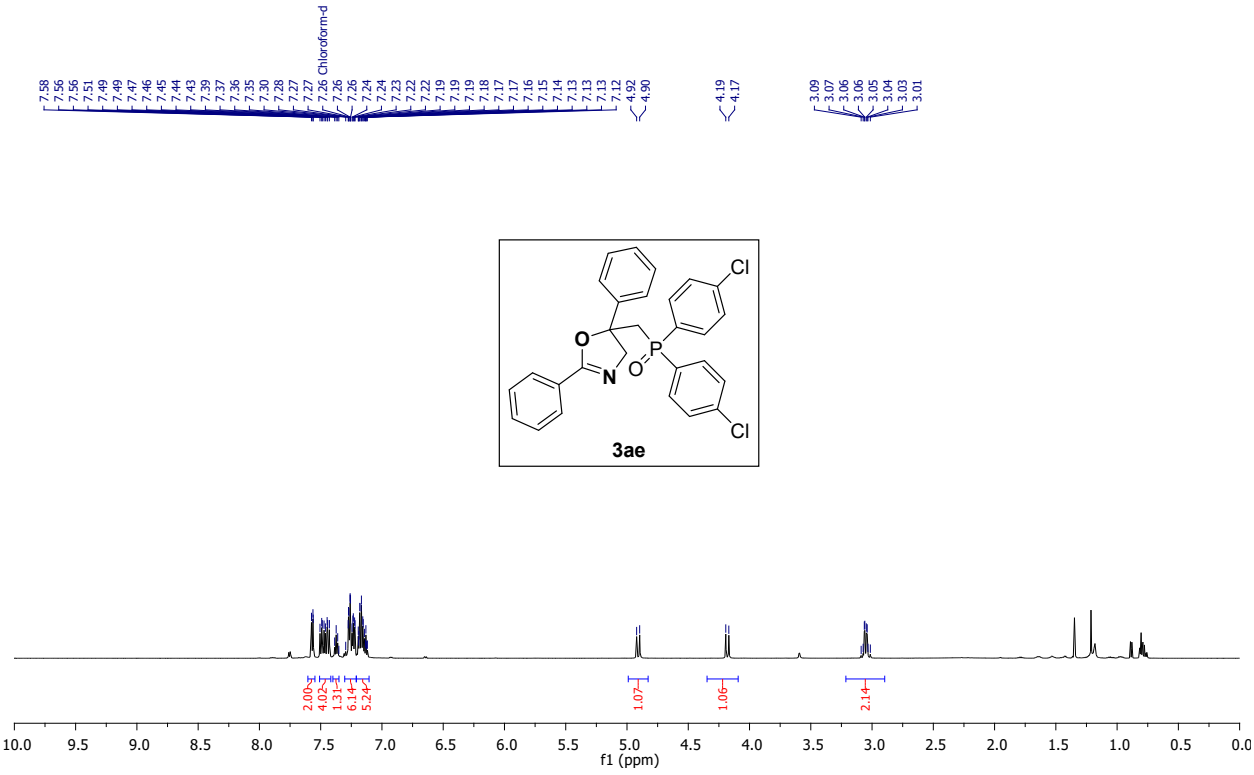

$^{1}$H NMR (400 MHz) spectrum of **3ad** in $CDCl_3$



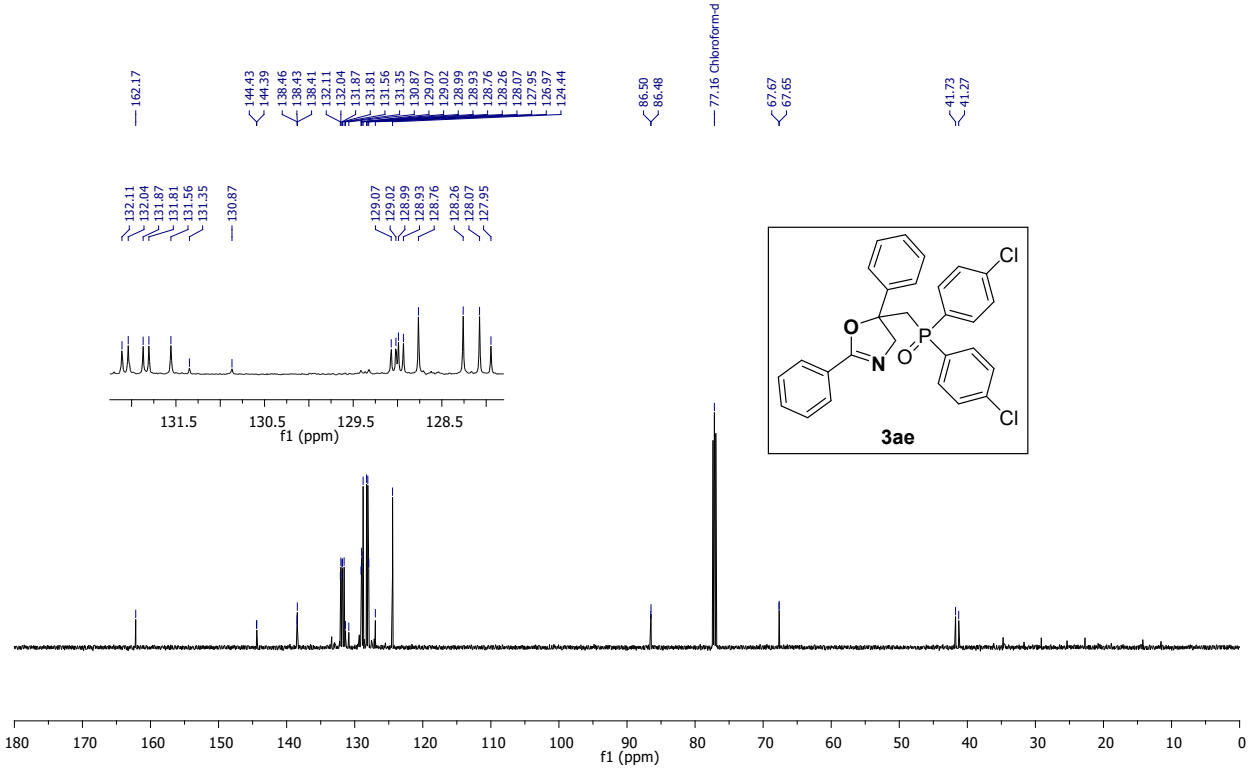

$^{13}$C {$^{1}$H} NMR (101 MHz) spectrum of **3ad** in $CDCl_3$



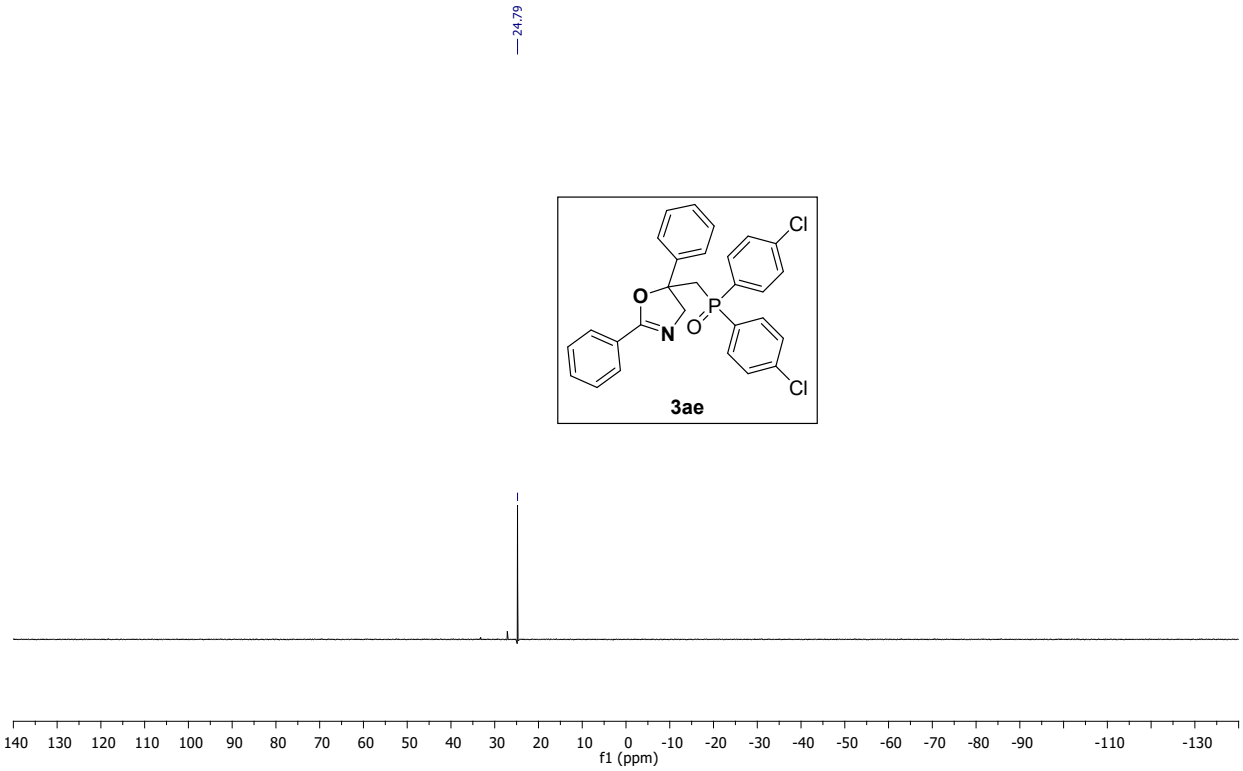

$^{31}$P NMR (243 MHz) spectrum of **3ad** in $CDCl_3$



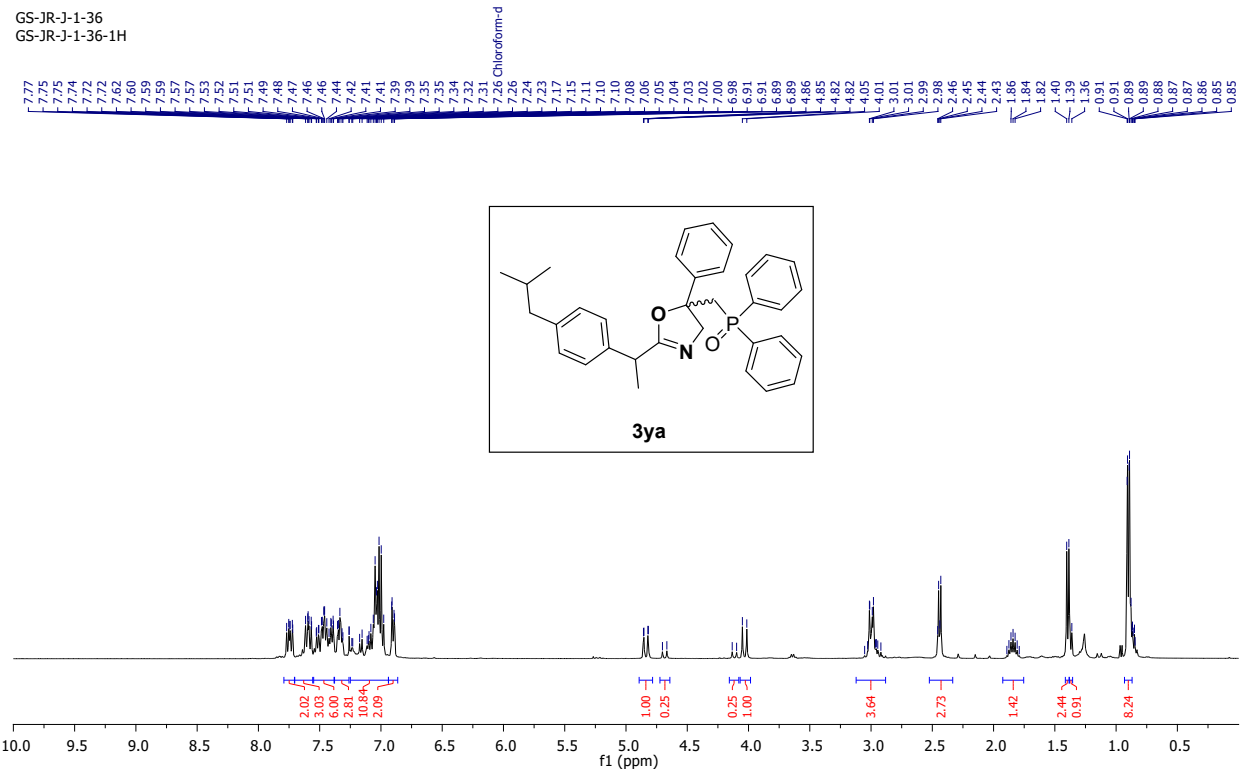

$^{1}$H NMR (600 MHz) spectrum of **3ae** in $CDCl_3$



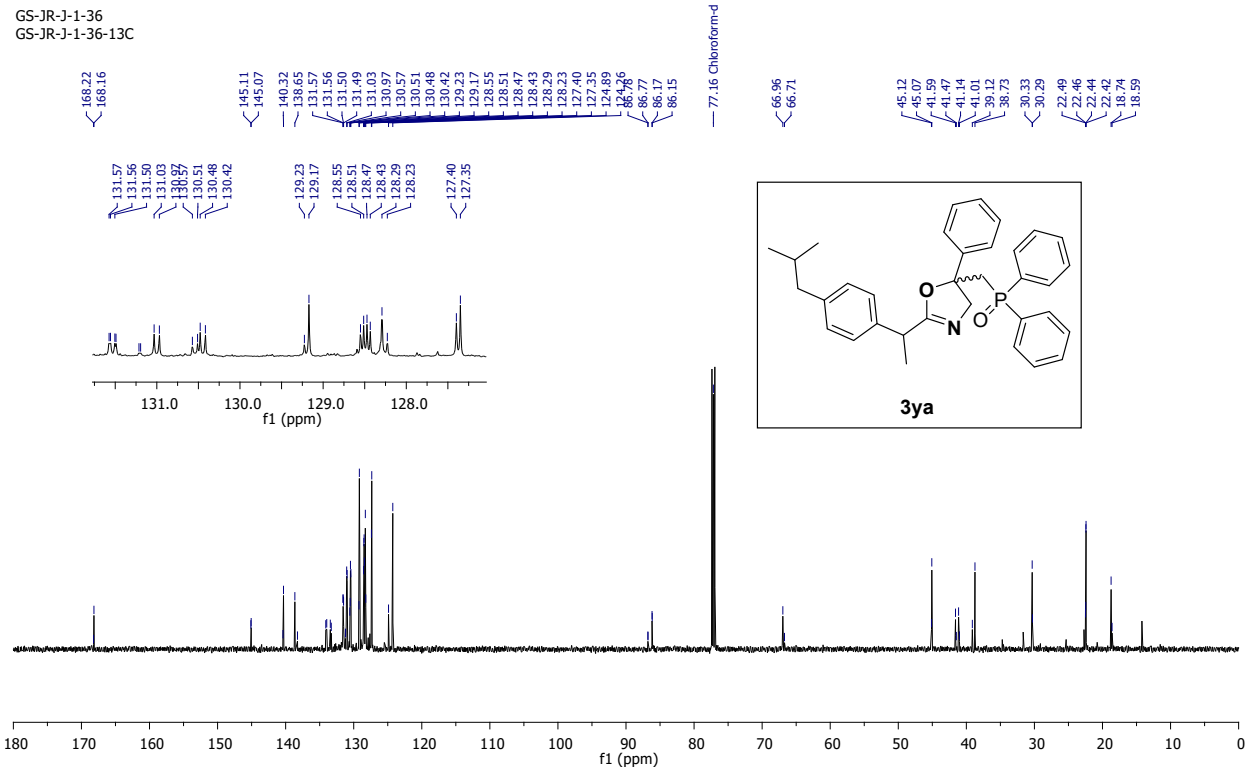

$^{13}C\{^{1}H\}$ NMR (151 MHz) spectrum of **3ae** in $CDCl_3$



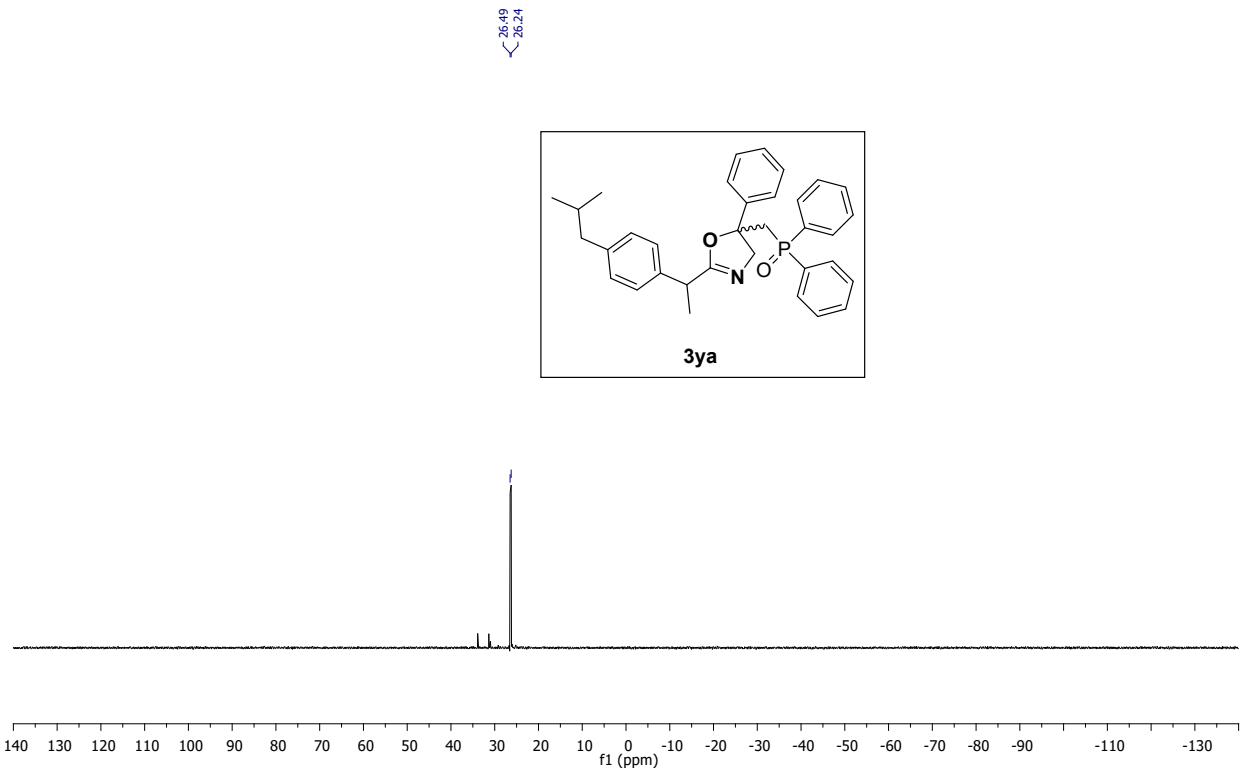

$^{31}$P NMR (243 MHz) spectrum of **3ae** in $CDCl_3$



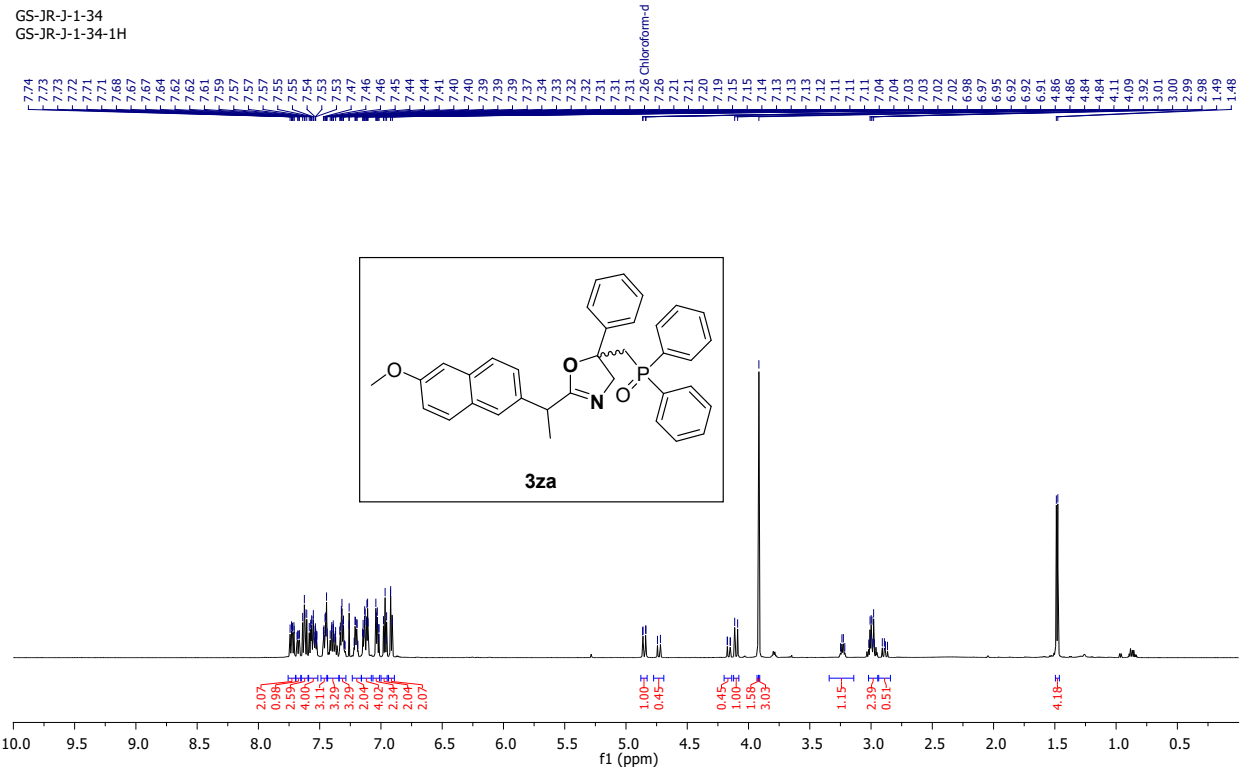

$^{1}$H NMR (600 MHz) spectrum of **3ya** in $CDCl_3$



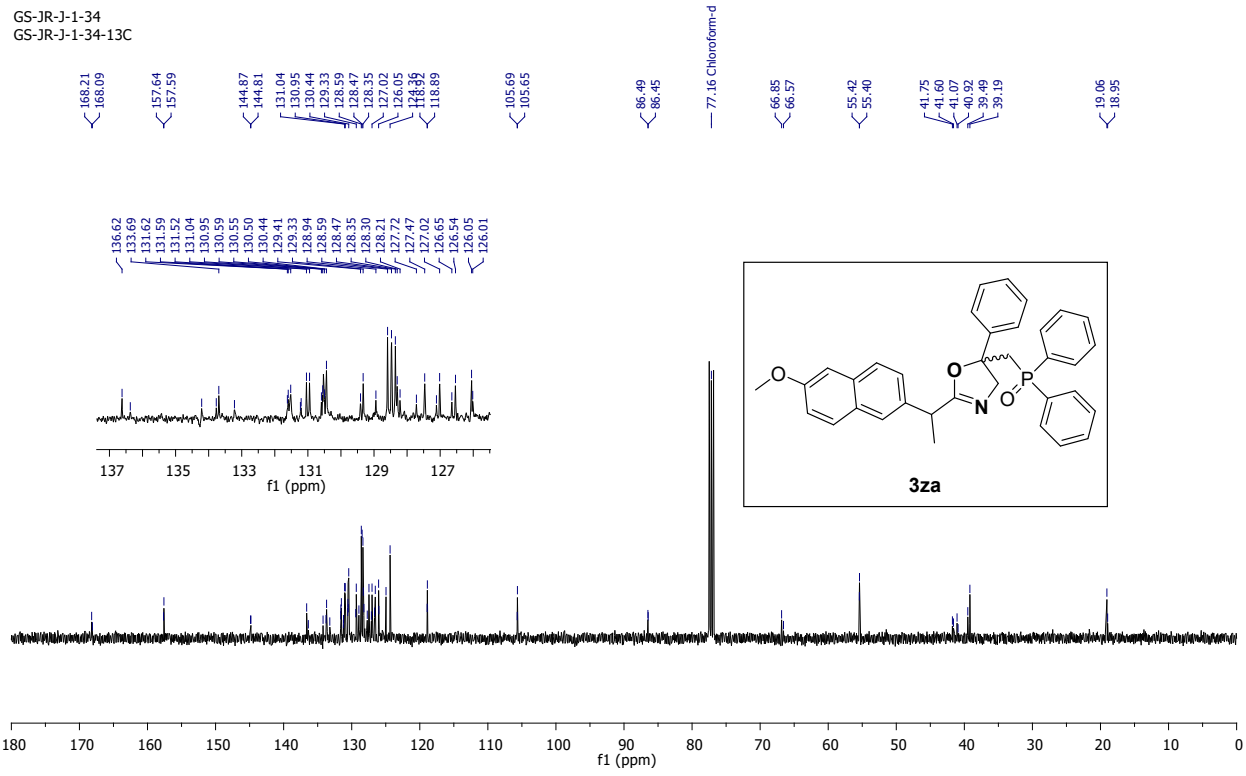

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3ya** in $CDCl_3$



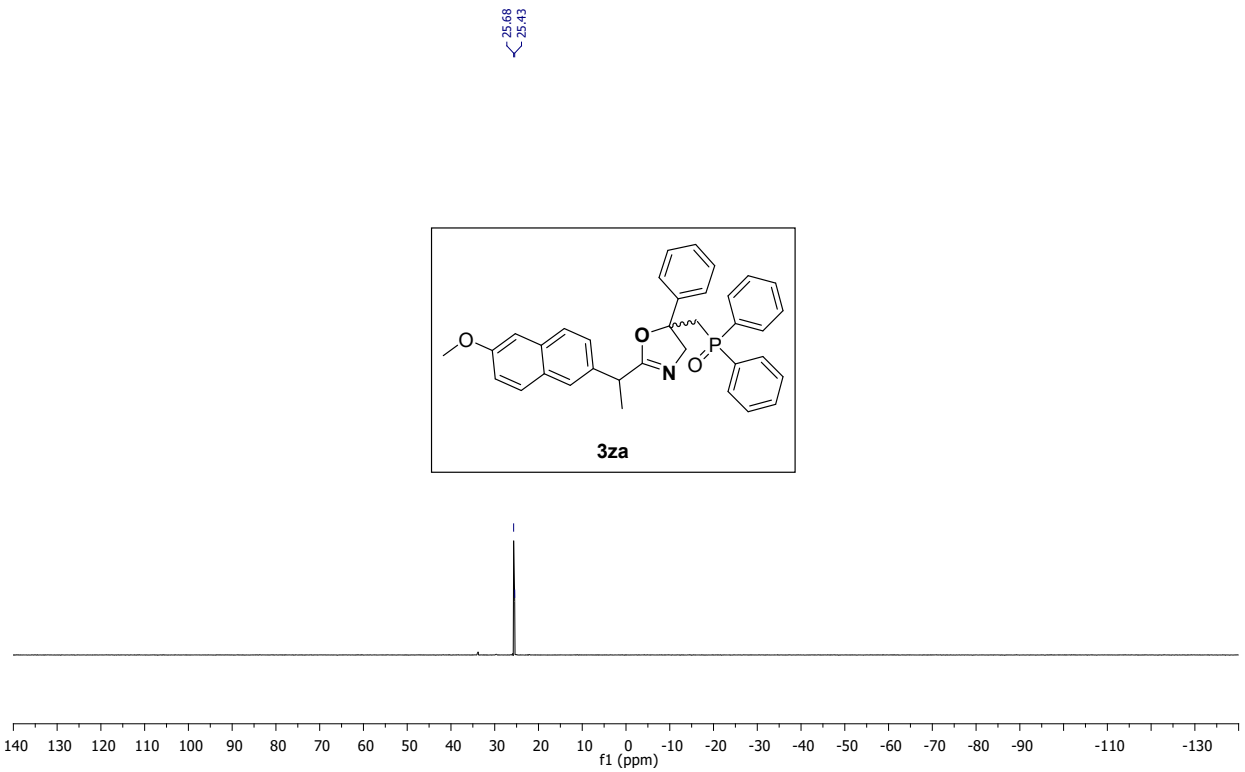

$^{31}$P NMR (243 MHz) spectrum of **3ya** in $CDCl_3$



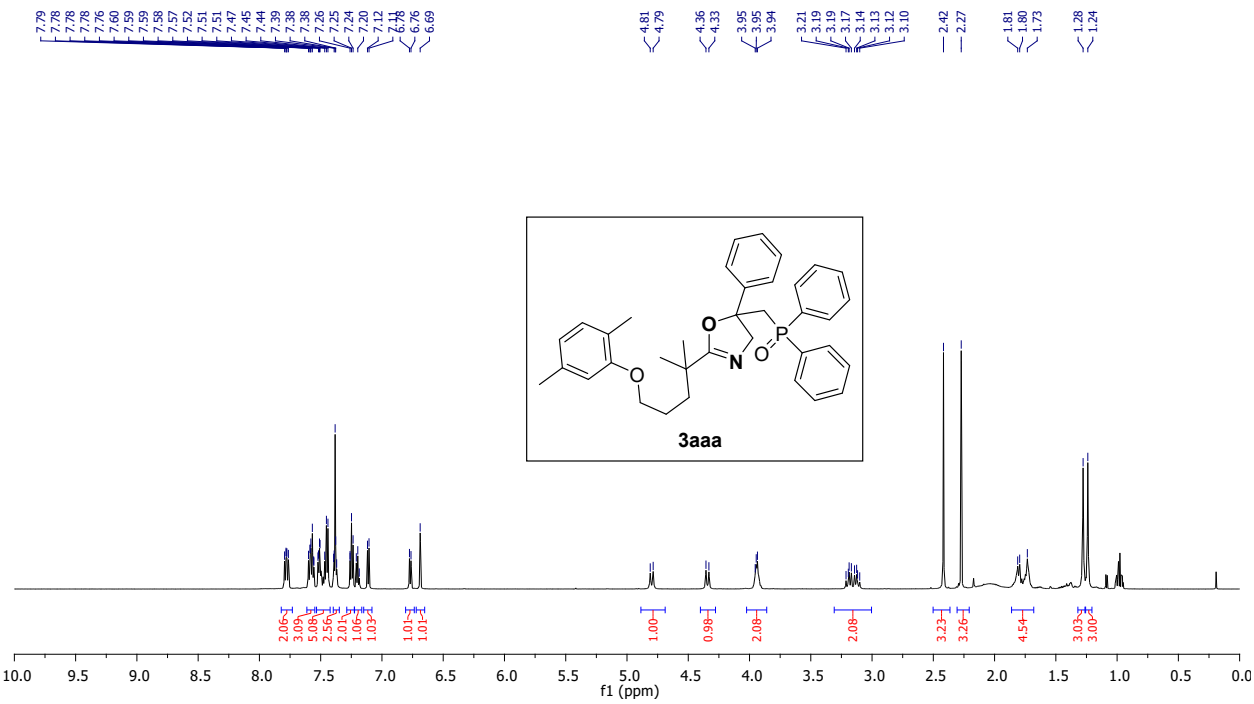

$^{1}$H NMR (600 MHz) spectrum of **3za** in $CDCl_3$



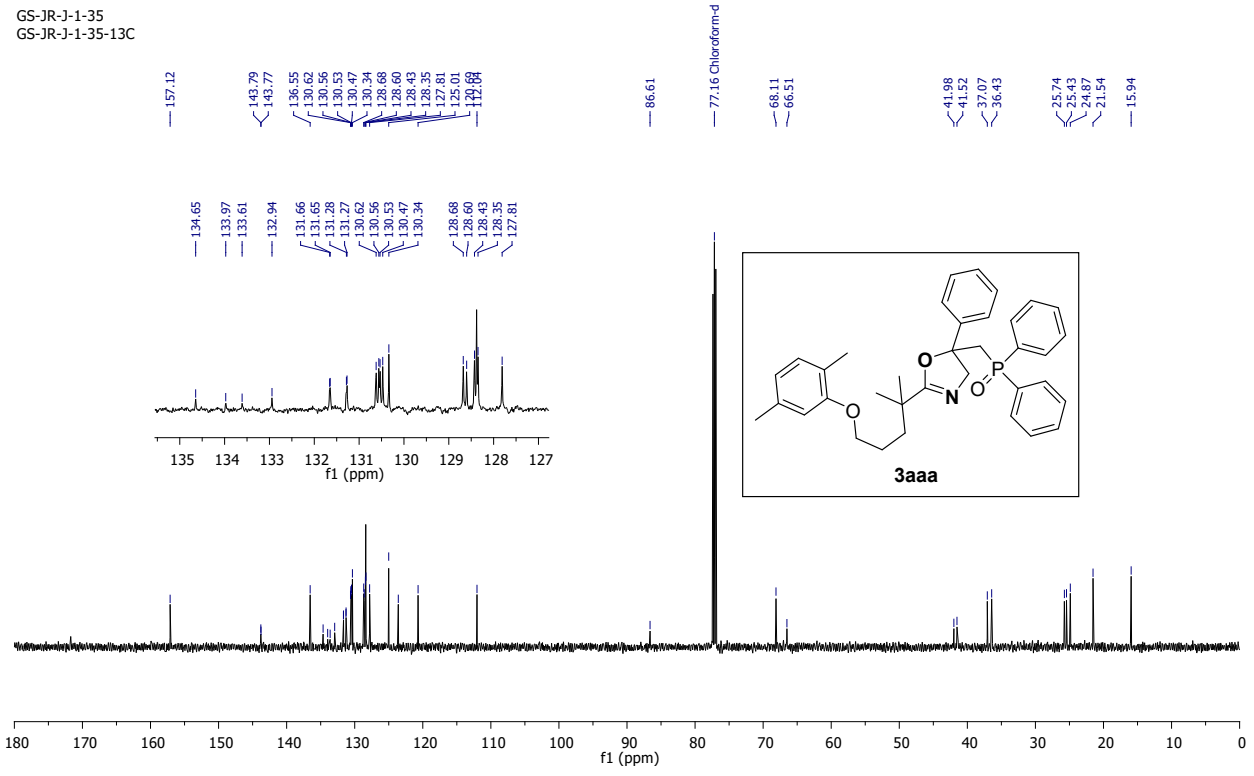

$^{13}$C {$^{1}$H} NMR (101 MHz) spectrum of **3za** in $CDCl_3$



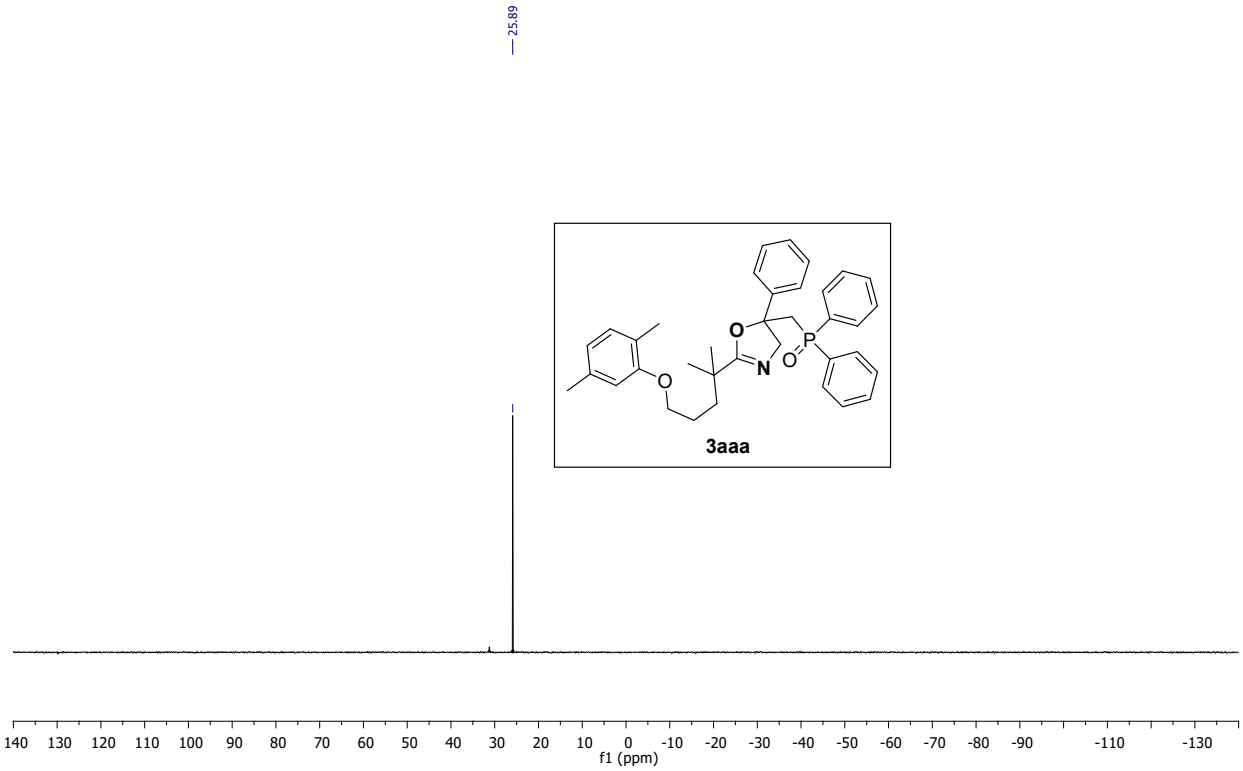

$^{31}$P NMR (243 MHz) spectrum of **3za** in $CDCl_3$




$^{1}$H NMR (600 MHz) spectrum of **3aaa** in $CDCl_3$



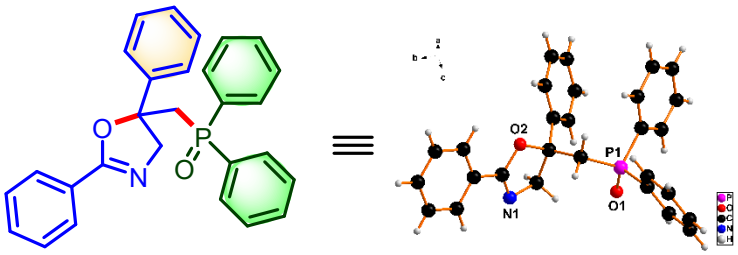

$^{13}$C {$^{1}$H} NMR (151 MHz) spectrum of **3aaa** in $CDCl_3$




$^{31}$P NMR (243 MHz) spectrum of **3aaa** in $CDCl_3$

**X-ray Diffraction Analysis of Compound 3aa:**

A Crystal of compound **3aa** was obtained by dissolving the product in a mixture of Acetonitrile and DCM in a 3:1 ratio, allowing the solvent to slowly evaporate at room temperature. The crystal structure information for this compound has been deposited at the Cambridge Crystallographic Data Centre. CCDC No: 2466394 contains the crystal structure information of this compound and can be obtained free of charge via http://www.ccdc.cam.ac.uk.




**3aa**

**Figure S2**: X-ray structure of the product **3aa** with the ellipsoids drawn at the 50% probability level.

**Table S4: Crystal data and structure refinement for 3aa.**

| | |
|---|---|
| Identification code | **3aa** |
| Empirical formula | $C_{28}H_{24}NO_2P$ |
| Formula weight | 437.45 |
| Temperature/K | 298 |
| Crystal system | monoclinic |
| Space group | Pn |
| a/Å | 6.0125(10) |
| b/Å | 12.501(2) |
| c/Å | 15.039(3) |
| α/° | 90 |
| β/° | 96.181(6) |
| γ/° | 90 |
| Volume/Å$^3$ | 1123.8(4) |
| Z | 2 |
| $\rho_{calc}$g/cm$^3$ | 1.293 |
| μ/mm$^{-1}$ | 0.148 |

| | |
|---|---|
| F(000) | 460.0 |
| Crystal size/mm$^3$ | 0.28 × 0.23 × 0.15 |
| Radiation | MoKα (λ = 0.71073) |
| 2Θ range for data collection/° | 4.246 to 54.21 |
| Index ranges | -7 ≤ h ≤ 7, -16 ≤ k ≤ 16, -19 ≤ l ≤ 19 |
| Reflections collected | 25702 |
| Independent reflections | 4708 [$R_{int}$ = 0.0825, $R_{sigma}$ = 0.0621] |
| Data/restraints/parameters | 4708/2/289 |
| Goodness-of-fit on $F^2$ | 1.019 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0431, $wR_2$ = 0.0840 |
| Final R indexes [all data] | $R_1$ = 0.0717, $wR_2$ = 0.0937 |
| Largest diff. peak/hole / e Å$^{-3}$ | 0.12/-0.19 |
| Flack parameter | 0.20(7) |

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