

Catalyst-free Thiophosphorylation of in Situ Formed *ortho*-Quinone Methides

Jeffrey Ash and Jun Yong Kang*

Department of Chemistry and Biochemistry, University of Nevada Las Vegas, 4505 South Maryland
Parkway, Las Vegas, Nevada, 89154-4003, United States

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1. General information

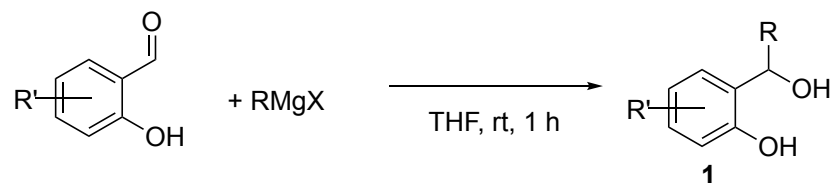
All reactions were carried out under an air atmosphere in oven-dried glassware with a magnetic stirring bar. Dry solvents (THF, toluene, ACN, diethyl ether, and DCM) were obtained by solvent purification system under argon. All commercially available reagents were used as received without further purification. The tubes used for the reaction were showed in **Figure S1**. Purification of reaction products was carried out by flash column chromatography using silica gel 60 (230-400 mesh). Analytical thin-layer chromatography was performed on 0.25 mm aluminum-backed silica gel 60-F plates. Visualization was accompanied with UV light and KMnO₄ solution. Concentration under reduced pressure refers to the removal of volatiles using a rotary evaporator attached to a dry diaphragm pump (10-15 mm Hg) followed by pumping to a constant weight with an oil pump (<300 mTorr). Infrared (IR) spectra were recorded on an IR spectrometer with KBr wafers or a film on a KBr plate. High-resolution mass spectra (HRMS) were recorded on LCMS-IT-TOF mass spectrometer using ESI (electrospray ionization) or APCI (Atmospheric Pressure Chemical Ionization). ¹H NMR spectra were recorded in CDCl₃ on 400 MHz NMR spectrometer. The ¹H chemical shifts are referenced to residual solvent signals at δ 7.26 (CHCl₃) or δ 0.00 (TMS). ¹H NMR coupling constants (*J*) are reported in Hertz (Hz) and multiplicities are indicated as follows: s (singlet), bs (broad singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublets), dt (doublet of triplets), td (triplet of doublets), tt (triplet of triplets). ¹³C NMR spectra were proton decoupled and recorded in CDCl₃ on 100.5 MHz NMR spectrometer. The ¹³C chemical shifts are referenced to solvent signals at δ 77.16 (CDCl₃). ³¹P NMR spectra were proton decoupled and recorded in CDCl₃ on 162 MHz NMR spectrometer. ³¹P chemical shifts are reported relative to 85% H₃PO₄ (0.00 ppm) as an external standard.



Figure S1. A pictorial description of reaction tubes for the reaction.

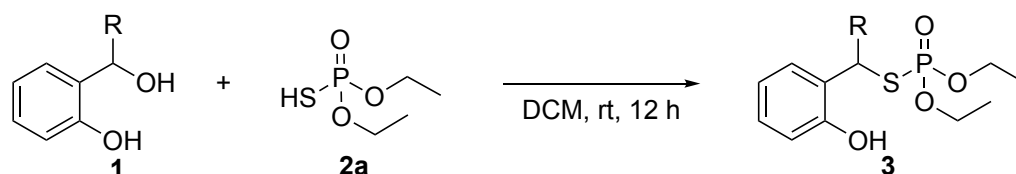
2. General experimental procedure

2.1. General procedure for the synthesis of diaryl alcohol **1**



Diaryl alcohol starting materials **1a-1n** were synthesized via Grignard reaction according to the following procedure.¹ To a suspension of Mg (10.0 mmol) in THF (8.0 mL) was added a crystal of iodine followed by dropwise addition of halide (10.0 mmol) dissolved in THF (3.0 mL). The reaction mixture was refluxed for 1 hour. The Grignard reagent (10.0 mmol) was cooled to 0°C and aldehyde (3.33 mmol) in THF (3.0 mL) was added dropwise. The reaction mixture was stirred for 1 hour at room temperature. After stirring for 1 hour, the reaction mixture was quenched with NH₄Cl and extracted with dichloromethane (3x 10mL). The organic layer was washed with brine, dried over sodium sulfate, and concentrated under reduced pressure. The residue was subjected to column chromatography on silica gel to give the corresponding diaryl alcohols **1**.

2.2. General procedure for the synthesis of thiophosphate **3**



To a solution of O,O-diethyl S-hydrogen phosphorothioate **2a** (0.15 mmol) in DCM (0.5 mL) was added diaryl phenol **1** (0.10 mmol) at room temperature. The reaction mixture was stirred for 12 hours. After stirring for 12 hours at room temperature, the reaction mixture was subjected to column chromatography on silica gel to give the corresponding thiophosphate products **3**.

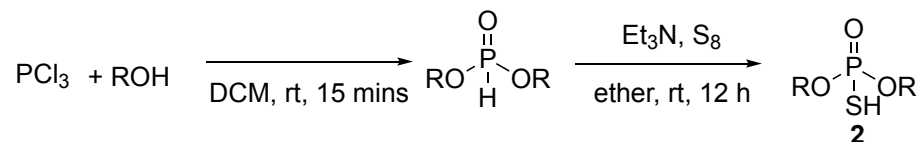
2.3. General procedure for the synthesis of thiophosphate **4**



To a solution of thiophosphoric acid **2** (0.15 mmol) in DCM (0.5 mL) was added 2-(hydroxy(phenyl)methyl)-4-methylphenol **1a** (0.10 mmol) at room temperature. The reaction mixture was stirred for 12 h. After

stirring for 12 hours at room temperature, the reaction mixture was subjected to column chromatography on silica gel to give the corresponding thiophosphate products **4**.

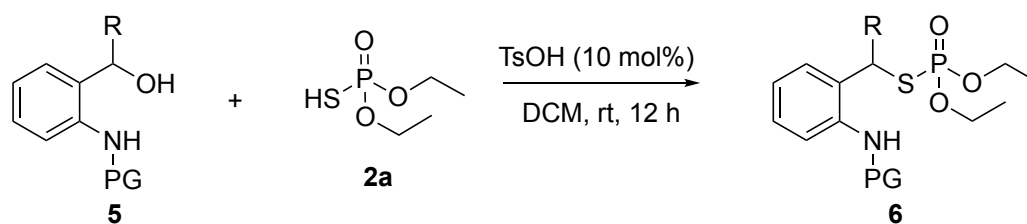
2.4. General procedure for the synthesis of thiophosphoric acid **2**



Step one: Alkoxyphosphites were prepared by a modified version of the following procedure.² To a solution of phosphorous trichloride (3.0 mmol) in DCM (200 μL) was added alkyl alcohol (9.0 mmol) dropwise at 0°C under an argon atmosphere. The reaction mixture was allowed to warm to room temperature and was stirred for 15 minutes at room temperature. After stirring for 15 minutes at room temperature, the reaction mixture was concentrated under reduced pressure and dried under a high vacuum to give the corresponding alkoxyphosphite in quantitative yield.

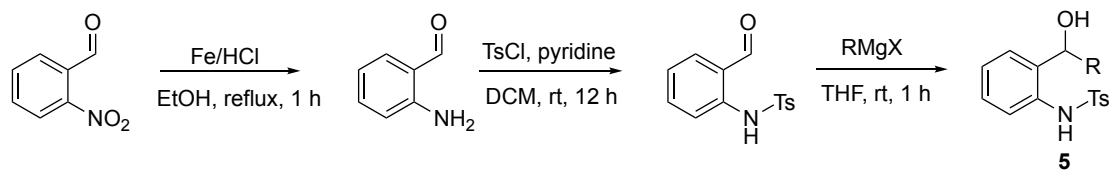
Step two: Thiophosphoric acids were prepared according to the following procedure.³ To a solution of alkoxy phosphite (10.0 mmol) and sulfur (12.0 mmol) in ether (25.0 mL) was added triethylamine (11.0 mmol). The reaction mixture was stirred for 12 hours. After stirring for 12 hours at room temperature, the reaction mixture was diluted with ether (10.0 mL) and washed with 1 M HCl. The organic layer was dried with sodium sulfate and concentrated under reduced pressure to give the thiophosphoric acid **2**.

2.5. General procedure for the synthesis of sulfonamido thiophosphate **6**



To a solution of thiophosphoric acid **2a** (0.2 mmol) and sulfonamido alcohol **5** (0.1 mmol) in DCM (0.5 mL) was added p-toluenesulfonic acid (0.01 mmol) at room temperature. The reaction mixture was stirred for 12 hours at room temperature. After stirring for 12 hours at room temperature, the reaction mixture was subjected to column chromatography on silica gel to give the corresponding sulfonamido thiophosphate product **6**.

2.6. General procedure for the synthesis of diaryl sulfonamido alcohol **5**

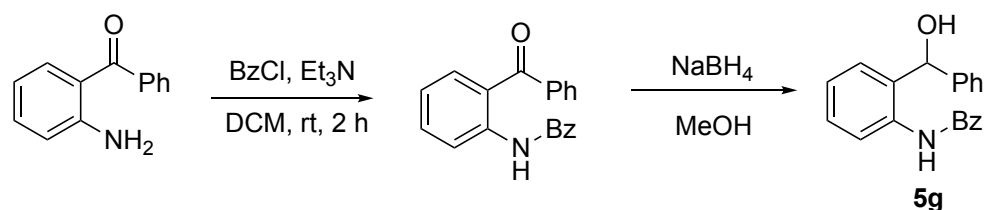


Step one: 2-amino benzaldehyde was prepared according to the following procedure.⁴ To a solution of 2-nitrobenzaldehyde (8.5 mmol) and iron powder (51 mmol) in ethanol (50 mL) was added concentrated HCl (0.5 mL). The reaction mixture was refluxed for 1 hour. After stirring for 1 hour under reflux, the iron was removed by filtration over celite. The yellow reaction mixture was concentrated under reduced pressure to dryness and dissolved in water. The residue was extracted with ethyl acetate (3x 10 mL) and the organic layer was washed with brine, dried with sodium sulfate, and concentrated under reduced pressure. The crude amine was subjected to the following reaction without further purification.

Step two: To a solution of 2-aminobenzaldehyde (2.15 mmol) and tosyl chloride (2.36 mmol) in DCM (5.8 mL) was added pyridine (4.73 mmol). The reaction mixture was stirred for 12 hours at room temperature. After stirring for 12 hours at room temperature, the reaction mixture was diluted with water and extracted with DCM (3x 10mL). The organic layer was washed with brine, dried over sodium sulfate, and concentrated under reduced pressure. The residue was subjected to column chromatography on silica gel to give the sulfonamido aldehyde.

Step three: To a suspension of Mg (3.0 mmol) and a crystal of iodine in THF (2.0 mL) was added halide (3.0 mmol) dissolved in THF (1.0 mL) dropwise. The reaction mixture was refluxed for 1 hour. The Grignard reagent (3.0 mmol) was cooled to 0°C and aldehyde (1.0 mmol) in THF (1.0 mL) was added dropwise. The reaction mixture was stirred for 1 hour at room temperature. After stirring for 1 hour, the reaction mixture was quenched with NH₄Cl and extracted with dichloromethane (3x 10mL). The organic layer was washed with brine, dried over sodium sulfate, and concentrated under reduced pressure. The residue was subjected to column chromatography on silica gel to give the corresponding sulfonamido alcohol **5**.

2.7 Procedure for the synthesis of **5g**

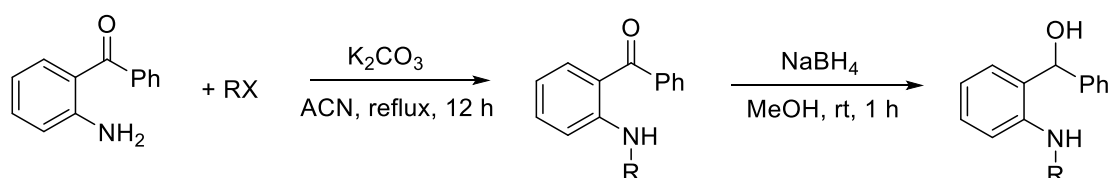


Step one: To a solution of (2-aminophenyl)(phenyl)methanone (1.0 mmol) and benzoyl chloride (1.5 mmol)

in DCM (5.0 mL) was added triethylamine (1.5 mmol). The reaction mixture was stirred for 2 hours at room temperature. After stirring for 2 hours at room temperature, the reaction mixture was subjected to column chromatography on silica gel to give *N*-(2-benzoylphenyl)benzamide.

Step two: To a solution of *N*-(2-benzoylphenyl)benzamide (0.63 mmol) in methanol (3.0 mL) was added sodium borohydride (1.55 mmol) portion wise at 0°C. The reaction mixture was stirred for 1 hour at room temperature. After stirring for 1 hour, the reaction mixture was quenched by water and extracted with DCM (3x 10mL). The organic layer was washed with brine, dried over sodium sulfate, and concentrated under reduced pressure to give *N*-(2-(hydroxy(phenyl)methyl)phenyl)benzamide **5g**.

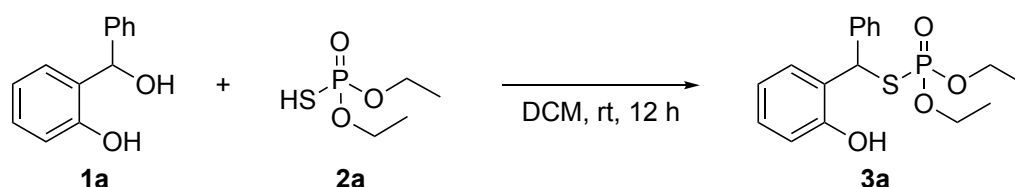
2.8 General procedure for the synthesis of 5h-5j



Step one: To a solution of (2-aminophenyl)(phenyl)methanone (3.0 mmol) and potassium carbonate (6.0 mmol) in acetonitrile (10.0 mL) was added halide (3.6 mmol). The reaction mixture was refluxed for 12 hours. After stirring for 12 hours of refluxing, the crude mixture was filtered through celite and concentrated under reduced pressure. The reaction mixture was then subjected to column chromatography on silica gel to give an alkylated product.

Step two: To a solution of secondary amine (1.00 mmol) in methanol (5.0 mL) was added sodium borohydride (2.50 mmol) portionwise at 0°C. The reaction mixture was stirred for 1 hour at room temperature. After stirring for 1 hour, reaction mixture was quenched by water and extracted with DCM (3x 10mL). The organic layer was washed with brine, dried over sodium sulfate, and concentrated under reduced pressure to give product **5h-5j**.

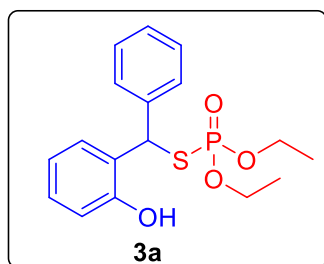
2.9. Scale-up experiment



To a solution of thiophosphoric acid **2a** (4.5 mmol) in DCM (15 mL) was added diaryl phenol **1a** (3.0 mmol). The reaction mixture was stirred for 12 h. After stirring for 12 hours, the mixture was concentrated under reduced pressure. The residue was subjected to column chromatography on silica gel to give the

corresponding product **3a** (922.8 mg) with 87% yield.

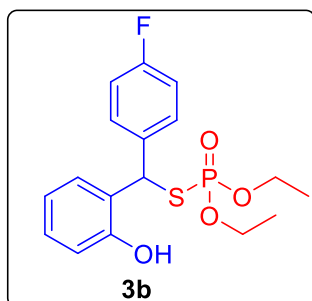
3. New compound characterization



O,O-diethyl S-((2-hydroxyphenyl)(phenyl)methyl) phosphorothioate

(3a). 31.5 mg, 91%; as an oil; **IR** ν (thin film, cm^{-1}): 3322, 2986, 1597, 1480, 1450, 1226, 1018, 972, 756, 702; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.47 (d, $J = 8.0$ Hz, 2H), 7.32-7.28 (m, 2H), 7.24-7.21 (m, 2H), 7.11-7.07 (m, 1H), 6.92 (d, $J = 4.0$ Hz, 1H), 6.83 (t, $J = 7.2$ Hz, 1H), 6.14 (d, $J = 12.4$ Hz, 1H). 4.07-3.91 (m, 3H), 3.90- 3.83 (m, 1H), 1.22-1.13 (m, 6H); **$^{13}\text{C NMR}$** (100.5 MHz, CDCl_3) δ 153.8, 140.7 (d, $J = 5.2$ Hz), 129.0, 128.8,

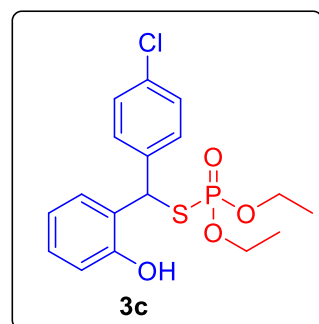
128.5 (d, $J = 5.2$ Hz), 128.4, 128.3, 127.3, 120.4, 117.2, 64.1 (d, $J = 5.9$ Hz), 64.0 (d, $J = 5.9$ Hz), 48.1 (d, $J = 3.0$ Hz), 15.7 (d, $J = 7.4$ Hz); **$^{31}\text{P NMR}$** (162 MHz, CDCl_3): δ 28.62; **HRMS** (ESI): m/z calcd. for $\text{C}_{17}\text{H}_{21}\text{O}_4\text{PS}$ ($[\text{M}+\text{Na}]^+$): 375.0790; Found: 375.0769.



O,O-diethyl S-((4-fluorophenyl)(2-hydroxyphenyl)methyl)

phosphorothioate (3b). 28.2 mg, 76%; as an oil; **IR** ν (thin film, cm^{-1}): 3140, 2985, 1597, 1504, 1411, 1342, 1234, 1157, 1095, 702; 78%; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 8.18 (br s, 1H), 7.45-7.42 (m, 2H), 7.20 (dd, $J = 7.6$ Hz, $J = 1.6$ Hz, 1H), 7.13-7.08 (m, 1H), 6.99-6.95 (m, 2H), 6.91 (dd, $J = 8.4$ Hz, $J = 1.2$ Hz, 1H), 6.84 (td, $J = 7.6$ Hz, $J = 1.2$ Hz, 1H), 6.14 (d, $J = 12.4$ Hz, 1H), 4.05-3.94 (m, 3H), 3.93-3.86 (m, 1H) 1.22-1.14 (m, 6H); **$^{13}\text{C NMR}$** (100.5 MHz, CDCl_3) δ 161.8 (d, $J = 246.0$ Hz), 153.7, 136.8,

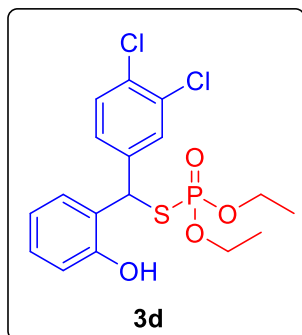
136.7 (d, $J = 5.9$ Hz), 129.9 (d, $J = 8.2$ Hz), 128.8 (d, $J = 16.4$ Hz), 128.3 (d, $J = 5.2$ Hz), 120.2, 117.1, 115.2 (d, $J = 20.8$ Hz), 64.1 (ap t, $J = 5.9$ Hz), 47.3 (d, $J = 12.0$ Hz) 15.7 (d, $J = 7.5$ Hz); **$^{31}\text{P NMR}$** (162 MHz, CDCl_3): δ 28.03; **HRMS** (ESI): m/z calcd. for $\text{C}_{17}\text{H}_{20}\text{O}_4\text{FPS}$ ($[\text{M}+\text{Na}]^+$): 393.0696; Found: 393.0688.



S-((4-chlorophenyl)(2-hydroxyphenyl)methyl) O,O-diethyl

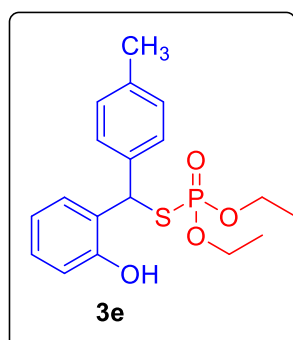
phosphorothioate (3c). 24.2 mg, 63%; as an oil; **IR** ν (thin film, cm^{-1}): 3224, 1597, 1489, 1226, 1018, 756; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 8.14 (br s, 1H), 7.42-7.38 (m, 2H), 7.28-7.25 (m, 2H), 7.21 (dd, $J = 7.6$ Hz, $J = 1.2$ Hz, 1H), 7.13-7.09 (m, 1H), 6.90 (dd, $J = 8.4$ Hz, $J = 1.2$ Hz, 1H), 6.84 (td, $J = 8.4$ Hz, $J = 1.2$ Hz, 1H), 6.12 (d, $J = 12.8$ Hz, 1H), 4.06-3.95 (m, 3H), 3.93-3.86 (m, 1H), 1.23-1.48 (m, 6H); **$^{13}\text{C NMR}$** (100.5 MHz, CDCl_3) δ 153.7, 139.5 (d, $J = 5.2$ Hz), 133.1, 129.6, 128.9, 128.7, 128.5, 128.1 (d, $J = 5.2$ Hz), 120.3, 117.2, 64.1 (ap t, $J = 5.9$ Hz), 47.4 (d, $J =$

2.9 Hz), 15.7 (d, $J = 7.4$ Hz); **$^{31}\text{P NMR}$** (162 MHz, CDCl_3): δ 28.00; **HRMS** (ESI): m/z calcd. for $\text{C}_{17}\text{H}_{20}\text{O}_4\text{CIPS}$ ($[\text{M}+\text{Na}]^+$): 409.0401; Found: 409.0378.



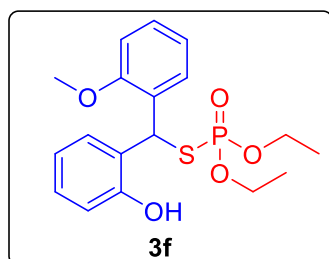
S-((3,4-dichlorophenyl)(2-hydroxyphenyl)methyl) O,O-diethyl phosphorothioate (3d). 33.2 mg, 82%; as a white solid; mp 118-120 °C
IR ν (thin film, cm^{-1}) 3224, 2985, 1597, 1458, 1226, 1018, 756; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.57 (s, 1H), 7.35-7.24 (m, 3H), 7.14-7.10 (m, 1H), 6.91-6.84 (m, 2H), 6.12 (d, $J = 12.4$ Hz, 1H), 4.07-4.00 (m, 3H), 3.99-3.89 (m, 1H), 1.25-1.16 (m, 6H); **$^{13}\text{C NMR}$** (100.5 MHz, CDCl_3) δ 153.7, 141.5 (d, $J = 5.3$ Hz), 132.3, 131.3, 130.2 ($J = 7.4$ Hz), 129.1, 128.6, 127.7, 127.3, 127.2, 120.3, 116.8, 64.2 (ap t, $J = 5.9$ Hz), 46.8 (d, $J = 3.0$ Hz), 15.8 (d, $J = 7.5$ Hz); **$^{31}\text{P NMR}$** (162 MHz, CDCl_3): δ 27.34; **HRMS** (ESI): m/z calcd.

for $\text{C}_{17}\text{H}_{19}\text{O}_4\text{PSCl}_2$ ($[\text{M}+\text{Na}]^+$): 443.0011; Found: 443.0075.



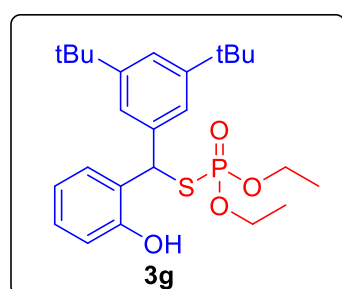
O,O-diethyl S-((2-hydroxyphenyl)(p-tolyl)methyl) phosphorothioate (3e). 28.7 mg, 80%; as an oil; **IR** ν (thin film, cm^{-1}): 3232, 2985, 2924, 1597, 1504, 1450, 1219, 1018, 756; 80%; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.93 (br s, 1H), 7.34 (d, $J = 8.0$ Hz, 2H), 7.18 (dd, $J = 8.0$ Hz, $J = 1.2$ Hz, 1H), 7.13-7.09 (m, 2H), 7.08 (d, $J = 1.6$ Hz, 1H), 6.92 (dd, $J = 8.0$ Hz, $J = 1.2$ Hz, 1H), 6.82 (td, $J = 6.8$ Hz, $J = 1.2$ Hz, 1H), 6.07 (d, $J = 12.4$ Hz, 1H), 4.09-3.94 (m, 3H), 3.89-3.82 (m, 1H), 2.32 (s, 3H), 1.23-1.12 (m, 6H); **$^{13}\text{C NMR}$** (100.5 MHz, CDCl_3) δ 153.6, 137.5 (d, $J = 6.0$ Hz), 137.1, 129.2, 129.1, 129.0, 128.7, 128.1, 120.5, 117.7, 64.1 (d, $J = 6.7$ Hz), 64.0 (d, $J = 5.9$ Hz),

48.0 (d, $J = 3.0$ Hz), 21.0, 15.8 (d, $J = 3.0$ Hz), 15.7 (d, $J = 3.0$ Hz); **$^{31}\text{P NMR}$** (162 MHz, CDCl_3): δ 28.88; **HRMS** (ESI): m/z calcd. for $\text{C}_{18}\text{H}_{23}\text{O}_4\text{PS}$ ($[\text{M}+\text{Na}]^+$): 389.0947; Found: 389.0926.



O,O-diethyl S-((2-hydroxyphenyl)(2-methoxyphenyl)methyl) phosphorothioate (3f). 29.5 mg, 81%; as a white solid; mp 123-125 °C; **IR** ν (thin film, cm^{-1}): 3232, 2985, 1597, 1489, 1018, 756; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.82 (br s, 1H), 7.60 (dd, $J = 7.6$, 1.6 Hz, 1H), 7.28-7.24 (m, 1H), 7.17 (dd, $J = 8.0$, $J = 2.0$ Hz, 1H), 7.11-7.07 (m, 1H), 6.99 (td, $J = 8.0$ Hz, $J = 1.2$ Hz, 1H), 6.91 (dd, $J = 8.0$ Hz, $J = 1.2$ Hz, 1H), 6.86 (dd, $J = 8.4$ Hz, $J = 0.8$ Hz, 1H), 6.78 (td, $J = 7.6$ Hz, $J = 1.2$ Hz, 1H),

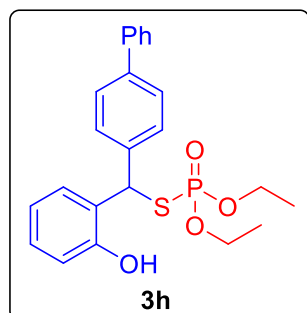
6.27 (d, $J = 14.0$ Hz, 1H), 4.08-3.94 (m, 3H), 3.93-3.86 (m, 1H), 3.78 (s, 3H), 1.20-1.15 (m, 6H); **$^{13}\text{C NMR}$** (100.5 MHz, CDCl_3) δ 155.8, 155.3, 129.4, 129.0, 128.94, 128.90, 128.7, 128.6, 128.3, 128.2, 117.7, 111.0, 63.9 (ap t, $J = 5.9$ Hz), 42.8 (d, $J = 3.0$ Hz), 15.8 (d, $J = 4.4$ Hz), 15.7 (d, $J = 4.5$ Hz); **$^{31}\text{P NMR}$** (162 MHz, CDCl_3): δ 29.40; **HRMS** (ESI): m/z calcd. for $\text{C}_{18}\text{H}_{23}\text{O}_5\text{PS}$ ($[\text{M}+\text{Na}]^+$): 405.0896; Found: 405.0885.



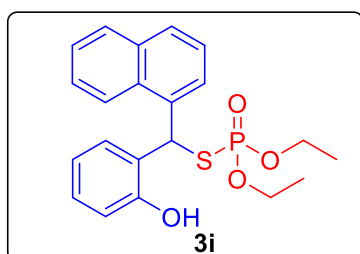
S-((3,5-di-tert-butylphenyl)(2-hydroxyphenyl)methyl) O,O-diethyl phosphorothioate (3g). 46.4 mg, 69% as a white solid; mp 142-145 °C **IR** ν (thin film, cm^{-1}) 3224, 2962, 1597, 1458, 1226, 1018, 756; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.30 (s, 3H), 7.18-7.16 (m, 1H), 7.11-7.07 (m, 1H), 6.94 (d, $J = 8.4$ Hz, 1H), 6.81 (t, $J = 8.0$ Hz, 1H), 6.05 (d, $J = 12.8$ Hz, 1H), 4.04-3.93 (m, 3H), 3.86-3.82 (m, 1H), 1.29 (s, 18H), 1.18-1.09 (m, 6H); **$^{13}\text{C NMR}$** (100.5 MHz, CDCl_3) δ 153.7, 150.8, 139.3 (d, $J = 5.9$ Hz), 129.1 (d, $J = 4.5$ Hz), 129.0, 128.7, 122.7, 121.3, 120.4,

117.7, 64.1 (d, $J = 5.9$ Hz), 63.9 (d, $J = 5.9$ Hz), 49.0 (d, $J = 2.9$ Hz), 34.8, 31.4, 15.7 (d, $J = 7.4$ Hz); **^{31}P**

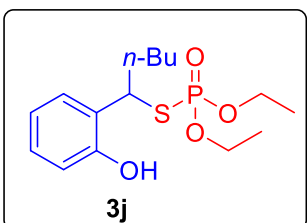
NMR (162 MHz, CDCl₃): δ 29.35; **HRMS** (ESI): m/z calcd. for C₂₅H₃₇NO₅PS ([M+Na]⁺): 487.2042; Found: 487.2024.



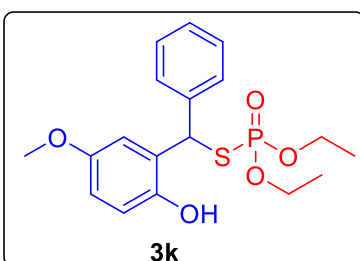
S-([1,1'-biphenyl]-4-yl(2-hydroxyphenyl)methyl) O,O-diethyl phosphorothioate (3h). 32.5 mg, 78%; as a white solid; mp 128-130 °C; **IR** ν (thin film, cm⁻¹): 3209, 2978, 1597, 1481, 1220, 1018, 750; **¹H NMR** (400 MHz, CDCl₃) δ 8.0 (br s, 1H), 7.77-7.51 (m, 6H), 7.44-7.39 (m, 2H), 7.35-7.31 (m, 1H), 7.27-7.15 (m, 1H), 7.14-7.11 (m, 1H), 6.94 (dd, *J* = 8.0 Hz, *J* = 1.2 Hz, 1H), 6.86 (td, *J* = 7.6 Hz, *J* = 1.2 Hz, 1H), 6.17 (d, *J* = 12.8 Hz, 1H), 4.10-3.97 (m, 3H), 3.92-3.86 (m, 1H), 1.23-1.14 (m, 6H); **¹³C NMR** (100.5 MHz, CDCl₃) δ 153.7, 140.4, 140.2, 139.6 (d, *J* = 5.9 Hz), 129.0, 128.9, 128.8 (d, *J* = 4.4 Hz), 128.76, 128.7, 127.3, 127.1, 127.0, 120.5, 117.6, 64.2 (d, *J* = 6.7 Hz), 64.1 (d, *J* = 6.0 Hz), 47.9 (d, *J* = 2.3 Hz), 15.8 (ap t, *J* = 2.2 Hz); **³¹P NMR** (162 MHz, CDCl₃): δ 28.56; **HRMS** (ESI): m/z calcd. for C₂₃H₂₅O₄PS ([M+Na]⁺): 451.1103; Found: 451.1097.



O,O-diethyl S-((2-hydroxyphenyl)(naphthalen-1-yl)methyl) phosphorothioate (3i). 35.9 mg, 92%; as an oil; **IR** ν (thin film, cm⁻¹) 3217, 2985, 1597, 1458, 1219, 1018, 756; **¹H NMR** (400 MHz, CDCl₃) δ 8.52 (br s, 1H), 8.08-8.06 (m, 1H), 7.87 (d, *J* = 7.2 Hz, 1H), 7.84-7.79 (m, 2H), 7.53-7.49 (m, 1H), 7.44-7.40 (m, 2H), 7.12-7.05 (m, 2H), 7.0 (dd, *J* = 8.0 Hz, *J* = 1.2 Hz, 1H), 6.83 (d, *J* = 12.4 Hz, 1H), 6.75-6.71 (m, 1H), 4.14-3.81 (m, 4H), 1.21-1.09 (m, 6H); **¹³C NMR** (100.5 MHz, CDCl₃) δ 153.2, 136.4 (d, *J* = 6.7 Hz), 133.7, 130.6, 129.4, 129.3, 129.0, 128.9, 128.6, 128.5, 126.7, 125.9, 124.8, 123.7, 120.7, 118.3, 64.3 (d, *J* = 6.7 Hz), 64.1 (d, *J* = 6.0 Hz), 45.7 (d, *J* = 2.3 Hz), 15.7 (ap t, *J* = 7.4 Hz); **³¹P NMR** (162 MHz, CDCl₃): δ 29.29; **HRMS** (ESI): m/z calcd. for C₂₁H₂₃O₄PS ([M+Na]⁺): 425.0947; Found: 425.0927.

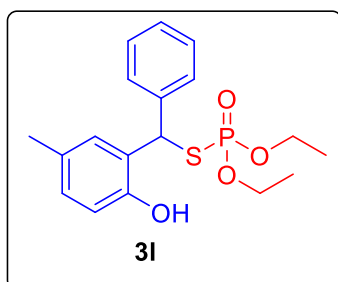


O,O-diethyl S-(1-(2-hydroxyphenyl)pentyl) phosphorothioate (3j). 11.1 mg, 34%; as an oil; **IR** ν (thin film, cm⁻¹): 3224, 2954, 1597, 1458, 1219, 1018, 972, 756; **¹H NMR** (400 MHz, CDCl₃) δ 8.10 (br s, 1H), 7.27-7.25 (m, 2H), 7.16-7.11 (m, 1H), 6.96-6.91 (m, 2H), 4.73-4.66 (m, 1H), 4.23-4.10 (m, 2H), 3.83-3.67 (m, 2H), 2.08-2.00 (m, 2H), 1.44-1.24 (m, 6H), 1.12-1.07 (m, 3H), 0.90-0.85 (m, 3H); **¹³C NMR** (100.5 MHz, CDCl₃) δ 153.7, 130.6, 128.6, 126.9, 121.2, 119.1, 64.1 (d, *J* = 6.7 Hz), 64.0 (d, *J* = 6.0 Hz), 44.9 (d, *J* = 3.7 Hz), 36.3 (d, *J* = 9.7 Hz), 29.8, 22.0, 15.9 (d, *J* = 7.4 Hz), 15.6 (d, *J* = 7.5 Hz), 13.8; **³¹P NMR** (162 MHz, CDCl₃): δ 30.85; **HRMS** (ESI): m/z calcd. for C₁₅H₂₅O₄PS ([M+Na]⁺): 355.1103; Found: 355.1087.

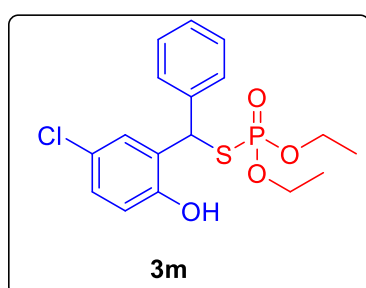


O,O-diethyl S-((2-hydroxy-5-methoxyphenyl)(phenyl)methyl) phosphorothioate (3k). 34.3 mg, 90%; as a white solid; mp 119-121 °C **IR** ν (thin film, cm⁻¹) 3248, 2985, 1597, 1435, 1103, 720; **¹H NMR** (400 MHz, CDCl₃) δ 7.47-7.45 (m, 2H), 7.34-7.30 (m, 2H), 7.27-7.25 (m, 1H), 6.88 (d, *J* = 8.4 Hz, 1H), 6.73 (d, *J* = 2.8 Hz, 1H), 6.69-6.66 (m, 1H), 6.07 (d, *J* = 12.0 Hz, 1H), 4.10-3.95 (m, 3H), 3.87-3.81 (m, 1H), 3.62 (s, 3H), 1.25-1.31 (m, 6H); **¹³C NMR** (100.5 MHz, CDCl₃) δ 153.5, 147.4, 140.2 (d, *J* = 6.7 Hz), 130.4 (d, *J* = 3.7 Hz), 128.4, 128.1, 127.4, 118.9, 114.5,

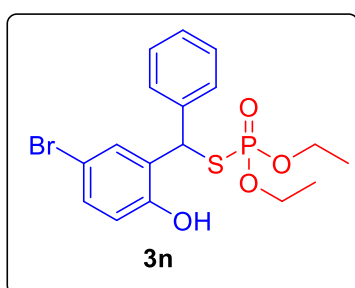
114.0, 64.5 (d, $J = 6.0$ Hz), 64.2 (d, $J = 6.7$ Hz), 55.6, 48.2 (d, $J = 3.0$ Hz), 15.8 (d, $J = 6.7$ Hz), 15.7 (d, $J = 7.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 28.73; HRMS (ESI): m/z calcd. for $\text{C}_{18}\text{H}_{23}\text{O}_5\text{PS}$ ($[\text{M}+\text{Na}]^+$): 405.0896; Found: 405.0886.



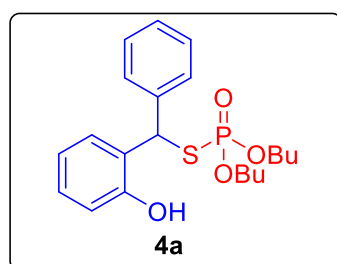
O,O-diethyl S-((2-hydroxy-5-methylphenyl)(phenyl)methyl) phosphorothioate (3l). 27.4 mg, 78%; as an oil; IR ν (thin film, cm^{-1}) 3232, 2985, 1504, 1226, 1018, 810; ^1H NMR (400 MHz, CDCl_3) δ 7.48-7.45 (m, 2H), 7.34-7.29 (m, 2H), 7.27-7.23 (m, 2H), 6.96-6.89 (m, 1H), 6.82 (d, $J = 8.0$ Hz, 1H), 6.06 (d, $J = 12.8$ Hz, 1H); 4.07-3.95 (m, 3H), 3.90-3.81 (m, 1H), 2.19 (s, 3H), 1.23-1.12 (m, 6H); ^{13}C NMR (100.5 MHz, CDCl_3) δ 151.2, 140.5 (d, $J = 6.0$ Hz), 129.8, 129.5, 129.3, 128.8 (d, $J = 3.7$ Hz), 128.4, 128.2, 127.3, 117.7, 64.1 (d, $J = 6.0$ Hz), 64.0 (d, $J = 5.9$ Hz), 48.2 (d, $J = 3.0$ Hz), 20.6, 15.8 (d, $J = 4.5$ Hz), 15.7 (d, $J = 4.4$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 28.76; HRMS (ESI): m/z calcd. for $\text{C}_{18}\text{H}_{23}\text{O}_4\text{PS}$ ($[\text{M}+\text{Na}]^+$): 389.0947; Found: 389.0935.



S-((5-chloro-2-hydroxyphenyl)(phenyl)methyl) O,O-diethyl phosphorothioate (3m). 31.0 mg, 84%; as an oil; IR ν (thin film, cm^{-1}) 3201, 2985, 1597, 1489, 1219, 1018; ^1H NMR (400 MHz, CDCl_3) δ 7.43 (d, $J = 7.6$ Hz, 2H), 7.31 (t, $J = 8.0$ Hz, 2H), 7.28-7.25 (m, 1H), 7.21 (d, $J = 2.4$ Hz, 1H), 7.03 (dd, $J = 8.8$ Hz, $J = 2.8$ Hz, 1H), 6.84 (d, $J = 8.8$ Hz, 1H), 6.09 (d, $J = 12.4$ Hz, 1H), 4.06-3.98 (m, 3H), 3.96-3.91 (m, 1H), 1.20 (q, $J = 6.8$ Hz, 6H); ^{13}C NMR (100.5 MHz, CDCl_3) δ 152.6, 139.9 (d, $J = 6.7$ Hz), 130.5, 130.4, 128.7, 128.6, 128.1, 127.6, 124.7, 118.7, 64.3 (ap t, $J = 6.7$ Hz), 47.7 (d, $J = 2.2$ Hz), 15.7 (d, $J = 7.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 27.91; HRMS (ESI): m/z calcd. for $\text{C}_{17}\text{H}_{20}\text{NO}_4\text{PSCl}$ ($[\text{M}+\text{Na}]^+$): 409.0401; Found: 409.0377.

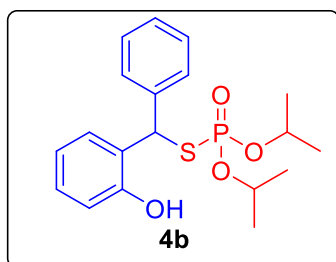


S-((5-bromo-2-hydroxyphenyl)(phenyl)methyl) O,O-diethyl phosphorothioate (3n). 31.1 mg, 72%; as an oil; IR ν (thin film, cm^{-1}) 3170, 2985, 1589, 1489, 1219, 1018, 748; ^1H NMR (400 MHz, CDCl_3) δ 7.43-7.41 (m, 2H), 7.39-7.25 (m, 4H), 7.16 (dd, $J = 8.4$ Hz, $J = 2.4$ Hz, 1H), 6.77 (d, $J = 8.4$ Hz, 1H), 6.11 (d, $J = 12.4$ Hz, 1H), 4.07-4.0 (m, 3H), 3.97-3.90 (m, 1H), 1.22-1.17 (m, 6H); ^{13}C NMR (100.5 MHz, CDCl_3) δ 153.2, 140.0 (d, $J = 6.7$ Hz), 131.4, 130.72, 130.68, 128.6, 128.2, 127.6, 118.8, 111.7, 64.3 (ap t, $J = 6.0$ Hz), 47.6 (d, $J = 3.0$ Hz), 15.7 (d, $J = 7.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 28.01; HRMS (ESI): m/z calcd. for $\text{C}_{17}\text{H}_{20}\text{O}_4\text{BrPS}$ ($[\text{M}+\text{Na}]^+$): 452.9895; Found: 452.9882.



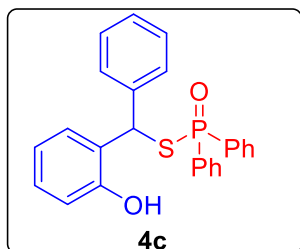
O,O-dibutyl S-((2-hydroxyphenyl)(phenyl)methyl) phosphorothioate (4a). 33.4 mg, 82%; as an oil; IR ν (thin film, cm^{-1}) 3224, 2962, 1597, 1458, 1219, 1018, 748; ^1H NMR (400 MHz, CDCl_3) δ 7.47-7.45 (m, 2H), 7.33-7.30 (m, 2H), 7.27-7.23 (m, 1H), 7.16 (dd, $J = 7.6$ Hz, $J = 1.6$ Hz, 1H), 7.12-7.09 (m, 1H), 6.94-6.92 (m, 1H), 6.82 (td, $J = 7.6$ Hz, $J = 1.2$ Hz, 1H), 6.11 (d, $J = 12.8$ Hz, 1H), 3.99-3.87 (m, 3H), 3.82-3.76 (m, 1H), 1.58-1.44 (m, 4H), 1.30-1.21 (m, 4H), 0.86-0.82 (m, 6H); ^{13}C NMR (100.5 MHz, CDCl_3) δ 153.7, 140.5 (d, $J = 6.0$ Hz), 129.2, 129.1, 129.0, 128.8, 128.4,

128.2, 127.3, 126.8, 67.9 (d, $J = 6.7$ Hz), 67.7 (d, $J = 6.0$ Hz), 48.2 (d, $J = 3.0$ Hz), 31.9 (d, $J = 2.2$ Hz), 31.8 (d, $J = 1.5$ Hz), 18.5 (d, $J = 3.0$ Hz), 13.4; ^{31}P NMR (162 MHz, CDCl_3): δ 29.29; HRMS (ESI): m/z calcd. for $\text{C}_{21}\text{H}_{25}\text{O}_4\text{PS}$ ($[\text{M}+\text{Na}]^+$): 431.1416; Found: 431.1404.

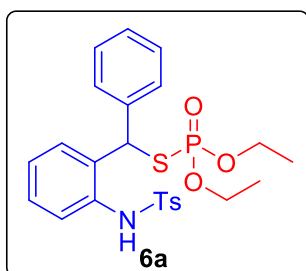


S-((2-hydroxyphenyl)(phenyl)methyl) O,O-diisopropyl phosphorothioate (4b). 35.7 mg, 93%; as an oil; IR ν (thin film, cm^{-1}) 3217, 2978, 1597, 1458, 1219, 987, 748; ^1H NMR (400 MHz, CDCl_3) δ 8.39 (br s, 1H), 7.46 (d, $J = 7.2$ Hz, 2H) 7.31 (t, $J = 7.2$ Hz, 2H), 7.24 (d, $J = 5.6$ Hz, 1H), 7.15 (d, $J = 7.6$ Hz, 1H), 7.12-7.07 (m, 1H), 6.94 (d, $J = 8.0$ Hz, 1H), 6.81 (d, $J = 7.2$ Hz, 1H), 6.14 (d, $J = 13.2$ Hz, 1H), 4.61-4.48 (m, 2H), 1.21-1.19 (m, 9H), 1.12 (d, $J = 6.0$ Hz, 3H); ^{13}C

NMR (100.5 MHz, CDCl_3) δ 153.9, 140.7 (d, $J = 6.7$ Hz), 129.3 (d, $J = 3.8$ Hz), 129.0, 128.7, 128.4, 128.3, 127.2, 120.3, 117.9, 73.5 (ap t, $J = 6.7$ Hz), 48.3 (d, $J = 2.3$ Hz); 23.6 (d, $J = 3.7$ Hz), 23.5 (d, $J = 4.5$ Hz), 23.4 (d, $J = 2.2$ Hz), 23.3 (d, $J = 3.0$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 26.76; HRMS (ESI): m/z calcd. for $\text{C}_{19}\text{H}_{25}\text{O}_4\text{PS}$ ($[\text{M}+\text{Na}]^+$): 403.1103; Found: 403.1091.

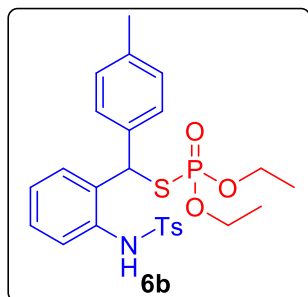


S-((2-hydroxyphenyl)(phenyl)methyl) diphenylphosphinothioate (4c). 21.2 mg, 51%; as a white solid; mp 172-174°C; IR ν (thin film, cm^{-1}) 3062, 2990, 1450, 1382, 1220, 823, 761, 615; ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 9.60 (br s, 1H), 7.72-7.66 (m, 4H), 7.57-7.53 (m, 2H), 7.47-7.43 (m, 4H), 7.31 (d, $J = 7.6$ Hz, 1H), 7.23-7.21 (m, 2H), 7.17-7.10 (m, 3H), 7.04-7.00 (m, 1H), 6.74-6.69 (m, 2H), 5.87 (d, $J = 12.0$ Hz, 1H); ^{13}C NMR (100.5 MHz, $\text{DMSO}-d_6$) δ 154.1, 142.1 (d, $J = 3.0$ Hz), 133.5 (d, $J = 104.9$ Hz), 132.8 (d, $J = 3.0$ Hz), 132.7 (d, $J = 2.3$ Hz), 131.4 (d, $J = 6.0$ Hz), 131.3 (d, $J = 6.0$ Hz), 129.2 (d, $J = 72.9$ Hz), 129.1 (d, $J = 12.7$ Hz), 128.7, 128.3 (d, $J = 17.8$ Hz), 127.1, 119.4, 115.6, 46.3; ^{31}P NMR (162 MHz, $\text{DMSO}-d_6$): δ 35.91; HRMS (ESI): m/z calcd. for $\text{C}_{25}\text{H}_{21}\text{O}_2\text{PS}$ ($[\text{M}+\text{Na}]^+$): 439.0892; Found: 439.0882.

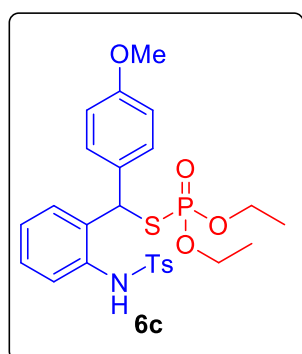


O,O-diethyl S-((2-((4-methylphenyl)sulfonamido)phenyl)(phenyl)methyl) phosphorothioate (6a). 45.3 mg, 90%; as a white solid; mp 139-141°C; IR ν (thin film, cm^{-1}) 3116, 2985, 1597, 1489, 1334, 1226, 1165, 1018, 748, 563; ^1H NMR (400 MHz, CDCl_3) δ 8.82 (s, 1H), 7.71 (d, $J = 8.4$ Hz, 1H), 7.57 (d, $J = 8.0$ Hz, 1H), 7.24-7.17 (m, 4H), 7.17-7.12 (m, 2H), 7.08 (td, $J = 8.4$, $J = 1.2$ Hz, 1H), 7.03 (dd, $J = 8.4$ Hz, $J = 1.6$ Hz, 1H), 6.80 (dd, $J = 8.4$ Hz, $J = 1.6$ Hz, 2H), 5.59 (d, $J = 12.8$ Hz, 1H), 4.13-4.01 (m, 2H), 3.97-

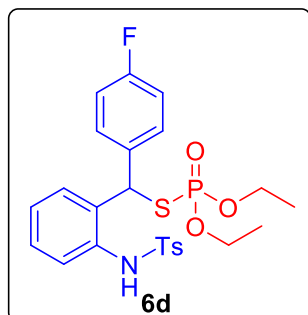
3.93 (m, 1H), 3.83-3.77 (m, 1H), 2.33 (s, 3H), 1.22-1.11 (m, 6H); ^{13}C NMR (100.5 MHz, CDCl_3) δ 143.2, 139.1, 139.0, 137.6, 136.6, 136.5, 133.8, 129.6, 128.4, 128.2, 127.3, 127.2, 126.8, 126.5, 64.5 (d, $J = 6.0$ Hz), 64.2 (d, $J = 6.7$ Hz), 48.4 (d, $J = 3.0$ Hz), 21.4, 15.8 (d, $J = 6.7$ Hz), 15.7 (d, $J = 7.5$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 27.72; HRMS (ESI): m/z calcd. for $\text{C}_{24}\text{H}_{28}\text{NO}_5\text{PS}_2$ ($[\text{M}+\text{Na}]^+$): 528.1039; Found: 528.1037.

O,O-diethyl S-((2-((4-methylphenyl)sulfonamido)phenyl)(p-tolyl)methyl) phosphorothioate (6b).

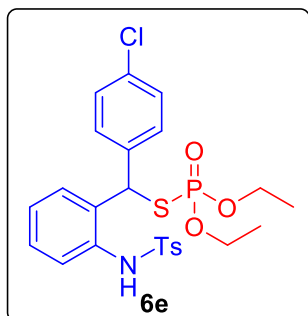
46.4 mg, 90%; as a white solid; mp 135-137 °C; **IR** ν (thin film, cm^{-1}) 3132, 2985, 1597, 1473, 1342, 1234, 1157, 1018, 756, 563; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.72 (s, 1H), 7.64 (d, $J = 8.4$ Hz, 2H), 7.21 (d, $J = 2.0$ Hz, 1H), 7.16-7.12 (m, 5H), 7.10-7.02 (m, 4H), 5.48 (d, $J = 12.0$ Hz, 1H), 4.01-3.95 (m, 2H), 3.87-3.77 (m, 2H), 2.34 (s, 3H), 2.31 (s, 3H), 1.15-1.08 (m, 6H); **$^{13}\text{C NMR}$** (100.5 MHz, CDCl_3) δ 143.5, 142.7 (d, $J = 5.2$ Hz), 137.9 (d, $J = 5.9$ Hz), 137.3, 137.2, 136.1, 129.5, 129.3, 129.2, 127.9, 127.3, 124.4, 120.4, 119.8, 63.6 (d, $J = 6.0$ Hz), 53.5 (d, $J = 2.9$ Hz), 21.5, 21.0, 15.8 (d, $J = 2.9$ Hz), 15.7 (d, $J = 3.0$ Hz); **$^{31}\text{P NMR}$** (162 MHz, CDCl_3): δ 25.64; **HRMS** (ESI): m/z calcd. for $\text{C}_{25}\text{H}_{30}\text{NO}_5\text{PS}_2$ ($[\text{M}+\text{Na}]^+$): 542.1195; Found: 542.1200.

**O,O-diethyl S-((4-methoxyphenyl)(2-((4-methylphenyl)sulfonamido)phenyl)methyl) phosphorothioate (6c).**

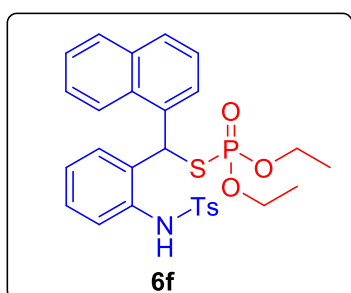
42.4 mg, 82% as a white solid; mp 145-148 °C **IR** ν (thin film, cm^{-1}) 3248, 2985, 1604, 1512, 1473, 1334, 1249, 1165, 1018; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.63 (d, $J = 8.4$ Hz, 2H), 7.39 (br s, 1H), 7.20-7.14 (m, 6H), 7.10 (d, $J = 8.0$ Hz, 1H), 7.02 (dt, $J = 8.0$ Hz, $J = .8$ Hz, 1H), 6.80 (d, $J = 8.8$ Hz, 2H), 5.50 (d, $J = 11.2$ Hz, 1H), 4.01-3.96 (m, 2H), 3.95-3.80 (m, 2H), 3.78 (s, 3H), 2.38 (s, 3H), 1.16-1.10 (m, 6H); **$^{13}\text{C NMR}$** (100.5 MHz, CDCl_3) δ 158.8, 143.6, 142.9 (d, $J = 5.2$ Hz), 137.1, 136.0, 132.9 (d, $J = 6.0$ Hz), 129.5, 129.4, 129.2, 127.3, 124.5, 120.5, 119.3, 113.8, 63.5 (d, $J = 5.9$ Hz), 55.2, 53.3 (d, $J = 3.0$ Hz), 21.5, 15.8 (d, $J = 3.7$ Hz), 15.7 (d, $J = 3.7$ Hz); **$^{31}\text{P NMR}$** (162 MHz, CDCl_3): δ 25.54; **HRMS** (ESI): m/z calcd. for $\text{C}_{25}\text{H}_{30}\text{NO}_6\text{PS}_2$ ($[\text{M}+\text{Na}]^+$): 558.1144; Found: 558.1117.

**O,O-diethyl S-((4-fluorophenyl)(2-((4-methylphenyl)sulfonamido)phenyl)methyl) phosphorothioate (6d).**

42.7 mg, 81%; as an oil; **IR** ν (thin film, cm^{-1}) 3140, 2985, 1597, 1505, 1411, 1342, 1157, 702, 633 %; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ 7.70 (s, 1H), 7.63 (d, $J = 8.4$ Hz, 2H), 7.27-7.24 (m, 2H), 7.16 (d, $J = 8.0$ Hz, 4H), 7.09 (d, $J = 6.8$ Hz, 1H), 7.04-7.01 (m, 1H), 6.97-6.93 (m, 2H), 5.53 (d, $J = 11.6$ Hz, 1H), 4.01-3.95 (m, 2H), 3.89-3.82 (m, 2H), 2.35 (s, 3H), 1.16-1.15 (m, 6H); **$^{13}\text{C NMR}$** (100.5 MHz, CDCl_3) δ 161.9 (d, $J = 245.6$ Hz), 143.7, 142.3 (d, $J = 6.0$ Hz), 137.4, 136.8 (dd, $J = 5.9$ Hz, 3.7 Hz), 136.0, 129.8 (d, $J = 8.2$ Hz), 129.5 (d, $J = 2.2$ Hz), 127.9, 124.3, 120.3, 120.0, 115.4, 115.2, 63.8 (d, $J = 3.0$ Hz), 63.7 (d, $J = 3.0$ Hz), 52.9 (d, $J = 2.9$ Hz), 21.4, 15.8 (d, $J = 2.3$ Hz), 15.7 (d, $J = 2.2$ Hz); **$^{31}\text{P NMR}$** (162 MHz, CDCl_3): δ 25.17; **HRMS** (ESI): m/z calcd. for $\text{C}_{24}\text{H}_{27}\text{NO}_5\text{FPS}_2$ ($[\text{M}+\text{Na}]^+$): 546.0944; Found: 546.0944.

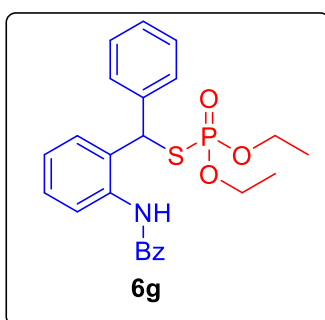


S-((4-chlorophenyl)(2-((4-methylphenyl)sulfonamido)phenyl)methyl)O,O-diethyl phosphorothioate (6e). 42.3 mg, 78% as a white solid; mp 131-133 °C; IR ν (thin film, cm^{-1}) 3140, 2985, 1597, 1489, 1404, 1342, 1234, 1157, 1095, 1018; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.63-7.60 (m, 2H), 7.54 (s, 1H), 7.25-7.20 (m, 4H), 7.15-7.13 (m, 4H), 7.08 (d, $J = 8.0$ Hz, 1H), 7.04-7.001 (m, 1H), 5.51 (d, $J = 11.6$ Hz, 1H), 4.02-3.96 (m, 2H), 3.89-3.83 (m, 2H), 2.36 (s, 3H), 1.17-1.12 (m, 6H); $^{13}\text{C NMR}$ (100.5 MHz, CDCl_3) δ 143.7, 142.1 (d, $J = 5.9$ Hz), 139.6, 139.5, 137.3, 136.0, 133.3, 129.6, 129.5, 128.6, 127.2, 124.3, 120.2, 120.1, 63.8 (d $J = 2.2$ Hz), 63.7 (d, $J = 2.3$ Hz), 52.9 (d, $J = 3.0$ Hz), 21.4, 15.7 (ap t, $J = 6.0$ Hz); $^{31}\text{P NMR}$ (162 MHz, CDCl_3): δ 25.01; **HRMS** (ESI): m/z calcd. for $\text{C}_{24}\text{H}_{27}\text{NO}_5\text{PS}_2\text{Cl}$ ($[\text{M}+\text{Na}]^+$): 562.0649; Found: 562.0640.



O,O-diethyl S-((2-((4-methylphenyl)sulfonamido)phenyl)(naphthalen-1-yl)methyl) phosphorothioate (6f). 41.3 mg, 74% as a white solid; mp 175-178 °C; IR ν (thin film, cm^{-1}) 3140, 2985, 1597, 1473, 1334, 1234, 1165, 1018, 786; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.06 (d, $J = 8.0$ Hz, 1H), 7.86 (d, $J = 7.6$ Hz, 1H), 7.79 (d, $J = 8.0$ Hz, 1H), 7.54 (d, $J = 8.0$ Hz, 1H), 7.49-7.42 (m, 4H), 7.41-7.39 (m, 1H), 7.28 (br s, 1H), 7.19-7.12 (m, 2H), 7.04-7.00 (m, 3H), 6.32 (d, $J = 12.4$ Hz, 1H), 4.03-

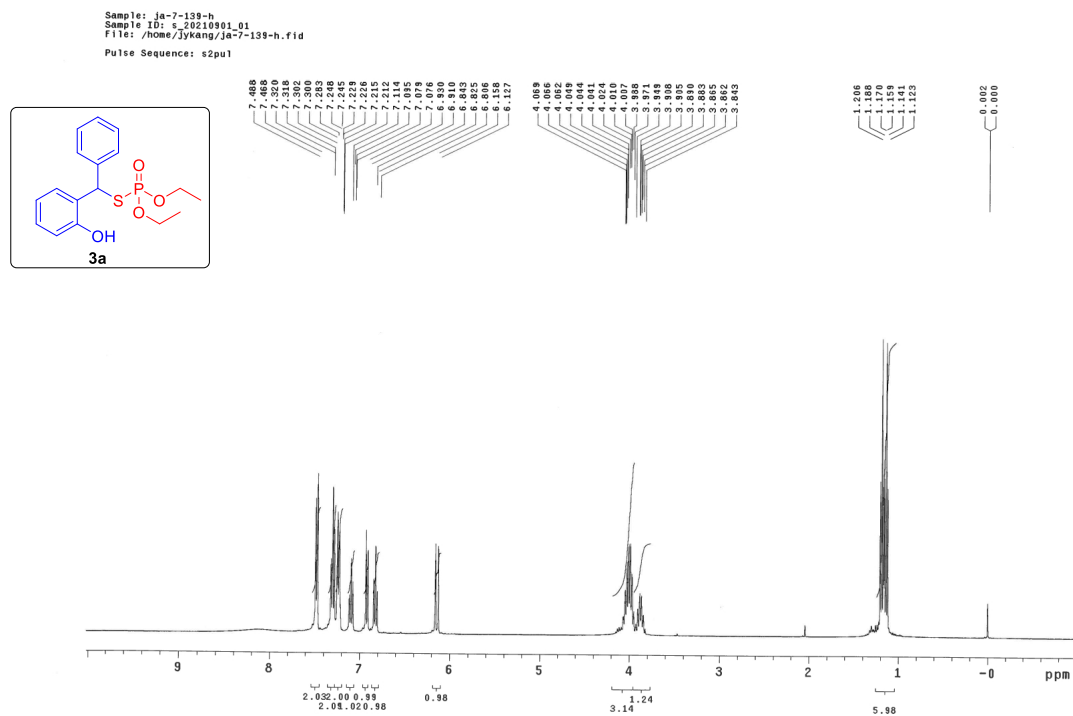
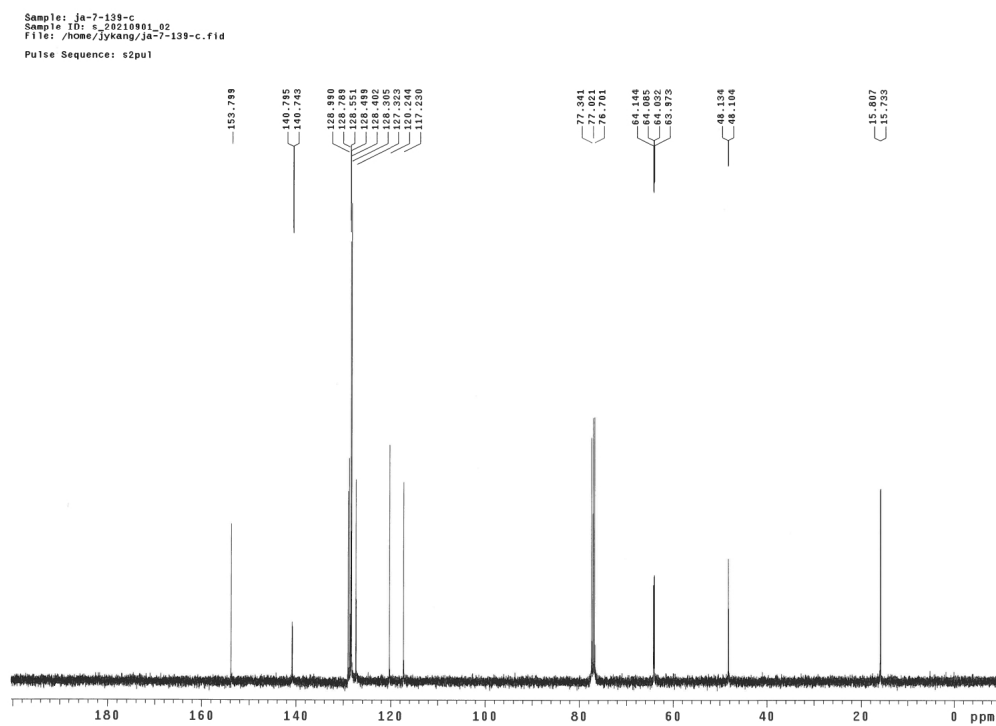
3.96 (m, 2H), 3.95-3.82 (m, 2H), 2.29 (s, 3H), 1.13-1.07 (m, 6H); $^{13}\text{C NMR}$ (100.5 MHz, CDCl_3) δ 143.5, 142.3 (d, $J = 3.8$ Hz), 137.2, 136.1 (d, $J = 5.9$ Hz), 135.9, 133.9, 130.3, 129.5, 129.4, 128.8, 128.6, 127.2, 126.8, 126.4, 125.8, 125.0, 124.8, 123.6, 120.8, 119.9, 63.8 (ap t, $J = 6.0$ Hz), 50.5, 21.4, 15.8 (d, $J = 5.2$ Hz), 15.7 (d, $J = 6.0$ Hz); $^{31}\text{P NMR}$ (162 MHz, CDCl_3): δ 25.64; **HRMS** (ESI): m/z calcd. for $\text{C}_{28}\text{H}_{30}\text{NO}_5\text{PS}_2$ ($[\text{M}+\text{Na}]^+$): 578.1195; Found: 578.1157.



S-((2-benzamidophenyl)(phenyl)methyl)O,O-diethyl phosphorothioate (6g). 15.3 mg, 34%; as an oil; IR ν (thin film, cm^{-1}) 3278, 2985, 1674, 1581, 1519, 1450, 1296, 1018, 748; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 9.64 (br s, 1H), 8.09-8.07 (m, 2H), 7.77 (d, $J = 8.8$ Hz, 1H), 7.56-7.44 (m, 3H), 7.42-7.34 (m, 3H), 7.30-7.23 (m, 3H), 7.21-7.17 (m, 2H), 6.08 (d, $J = 10.4$, 1H), 4.20-4.03 (m, 2H), 3.76-3.61 (m, 2H), 1.30-1.26 (m, 3H), 1.05-1.00 (m, 3H); $^{13}\text{C NMR}$ (100.5 MHz, CDCl_3) δ 166.1, 139.4 (d, $J = 8.9$ Hz), 139.1 (d, $J = 2.3$ Hz), 134.7, 134.2, 131.7, 129.9, 128.6, 128.5, 128.2, 127.8, 127.7, 127.6, 126.9, 126.3, 64.3 (d,

$J = 6.0$ Hz), 63.6 (d, $J = 6.7$ Hz), 48.8 (d, $J = 3.0$ Hz), 15.9 (d, $J = 7.4$ Hz), 15.6 (d, $J = 8.2$ Hz); $^{31}\text{P NMR}$ (162 MHz, CDCl_3): δ 26.96; **HRMS** (ESI): m/z calcd. for $\text{C}_{24}\text{H}_{26}\text{NO}_4\text{PS}$ ($[\text{M}+\text{Na}]^+$): 478.1212; Found: 478.1209.

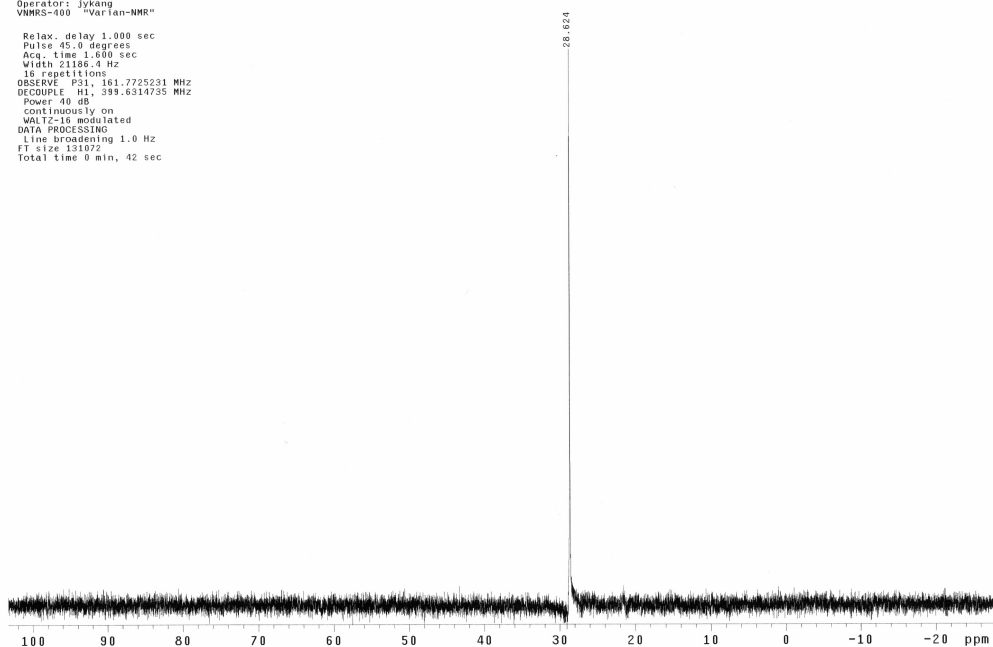
4. ^1H , ^{13}C , and ^{31}P NMR spectra

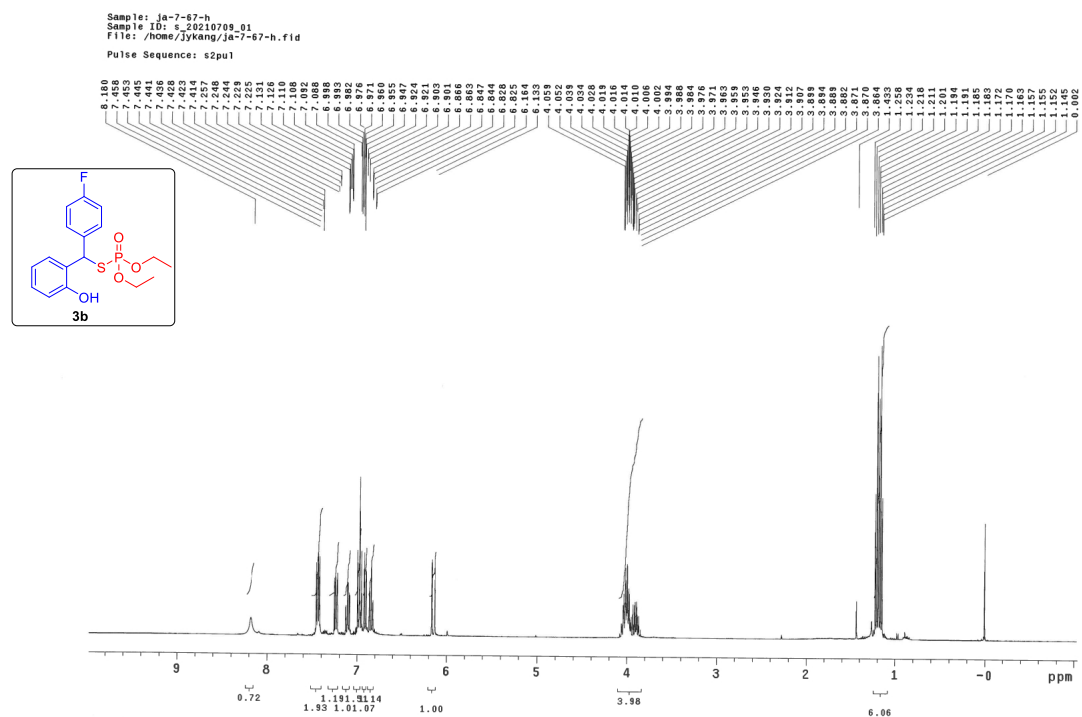
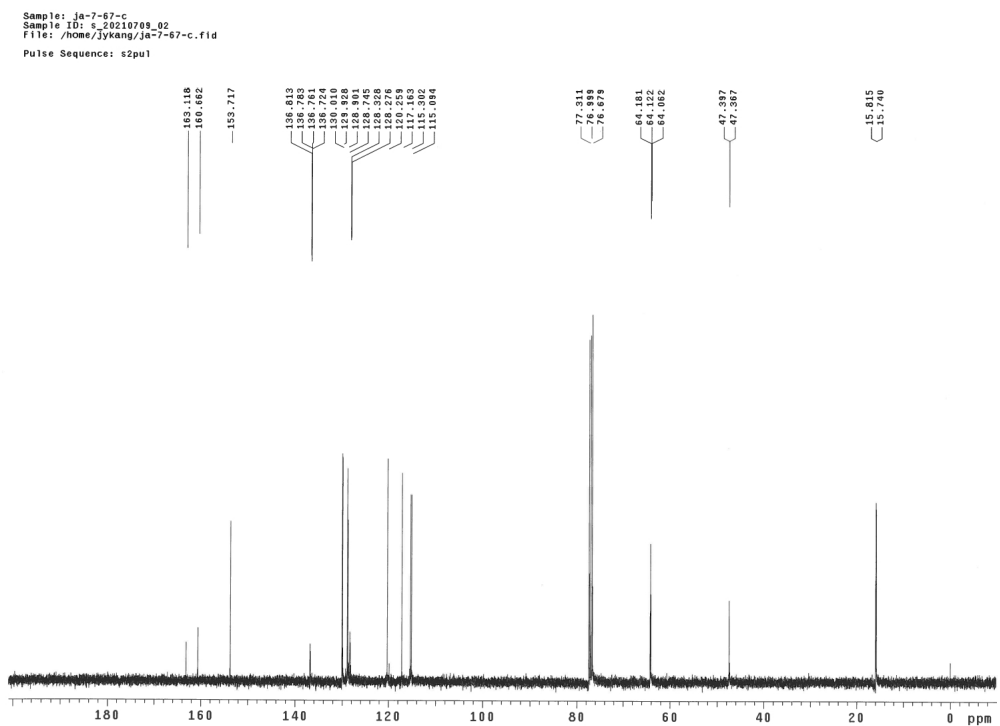
¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

Sample: ja-7-139-p
File: exp
Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: jykang
VNMR-400 "Varian-NMR"

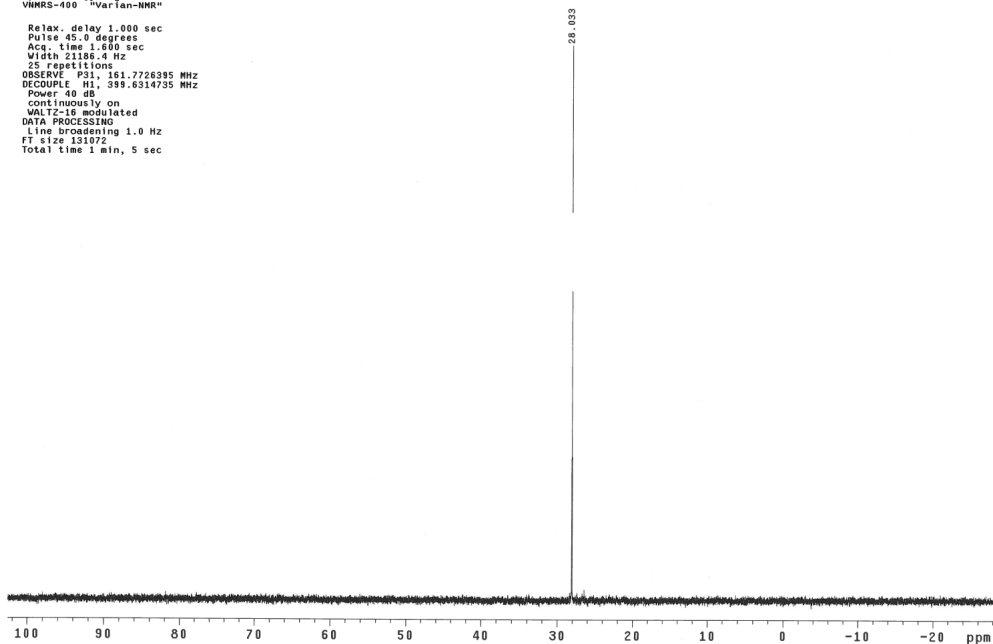
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21166.0 Hz
16 repetitions
OBSERVE P31, 161.725231 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 0 min, 42 sec

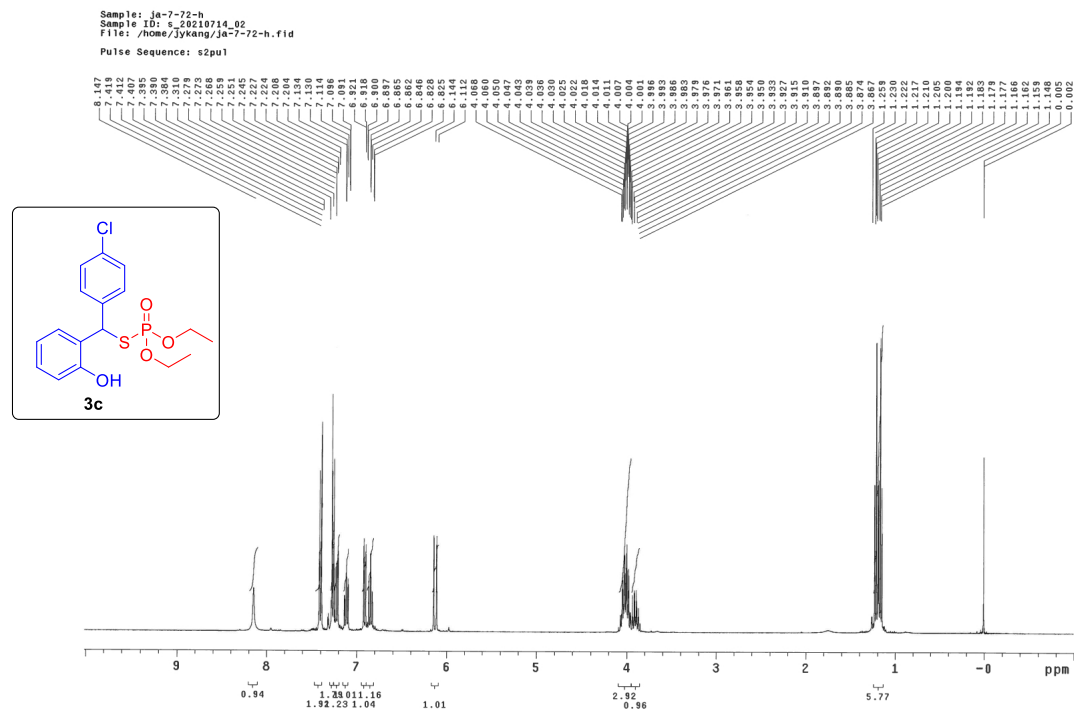
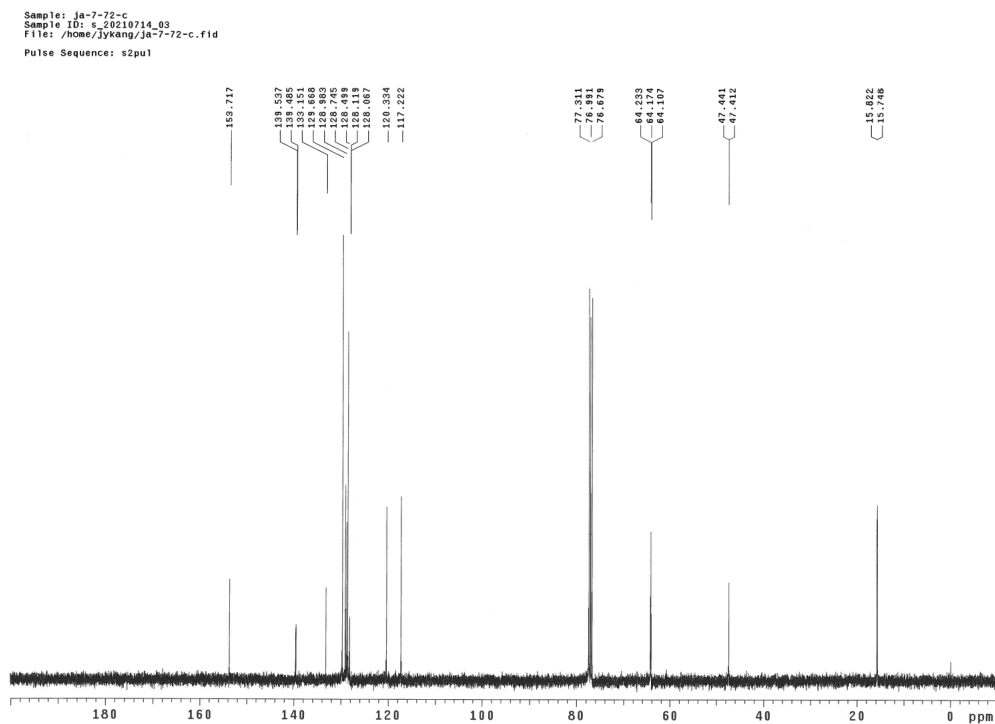


¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

Sample: ja-7-67-p
File: exp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: jykang
VMDS-600 "Varian-NMR"
Relax. delay 1.000 sec
Pulse 45.0 degree
Acq. time 1.600 sec
Width 21186.4 Hz
25 repetitions
OBSERVE F31, 161.7726395 MHz
DECOUPLE H1, 399.8314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

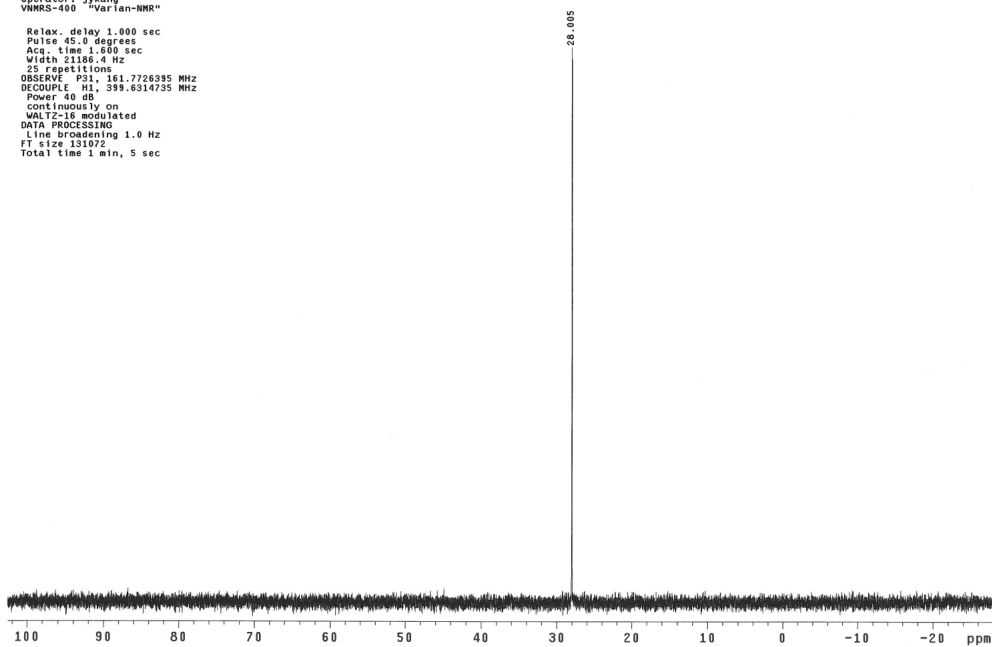


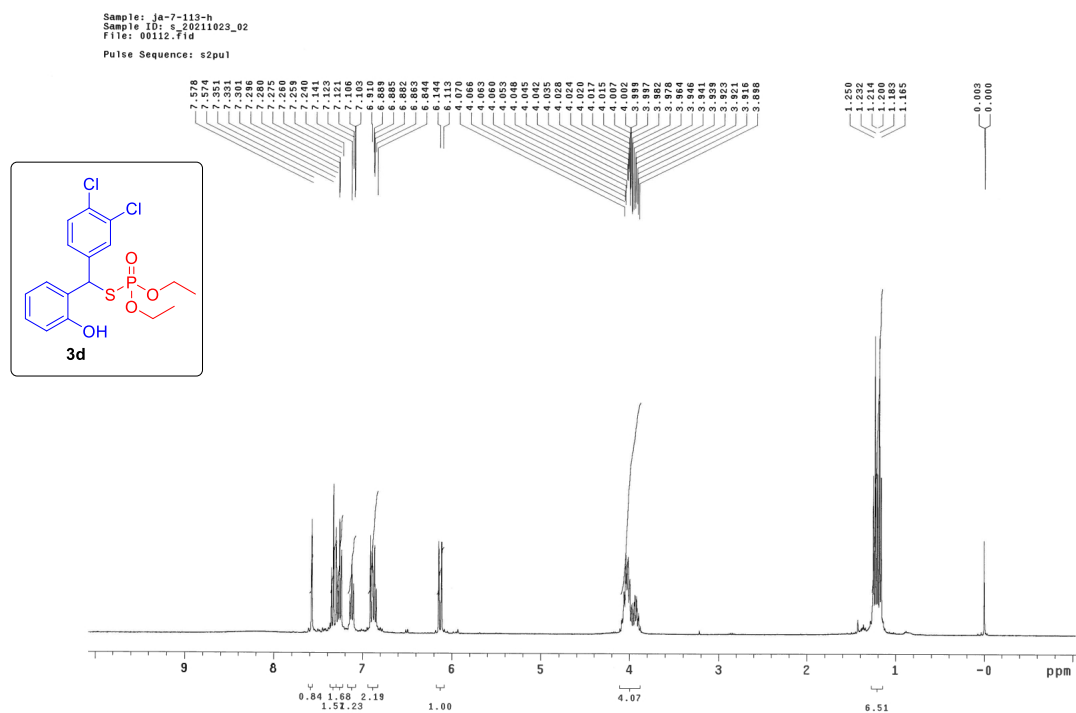
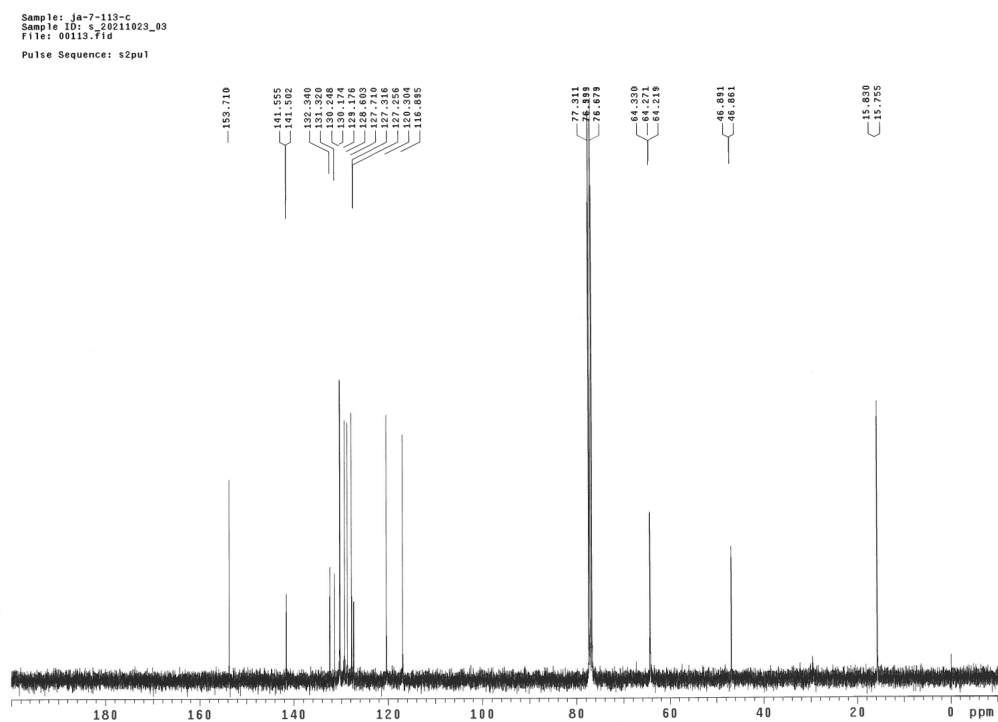
^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

^{31}P NMR (162 MHz) in CDCl_3

Sample: ja-7-72-p
File: exp
Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: jykang
VNMR5-000 "Varian-NMR"

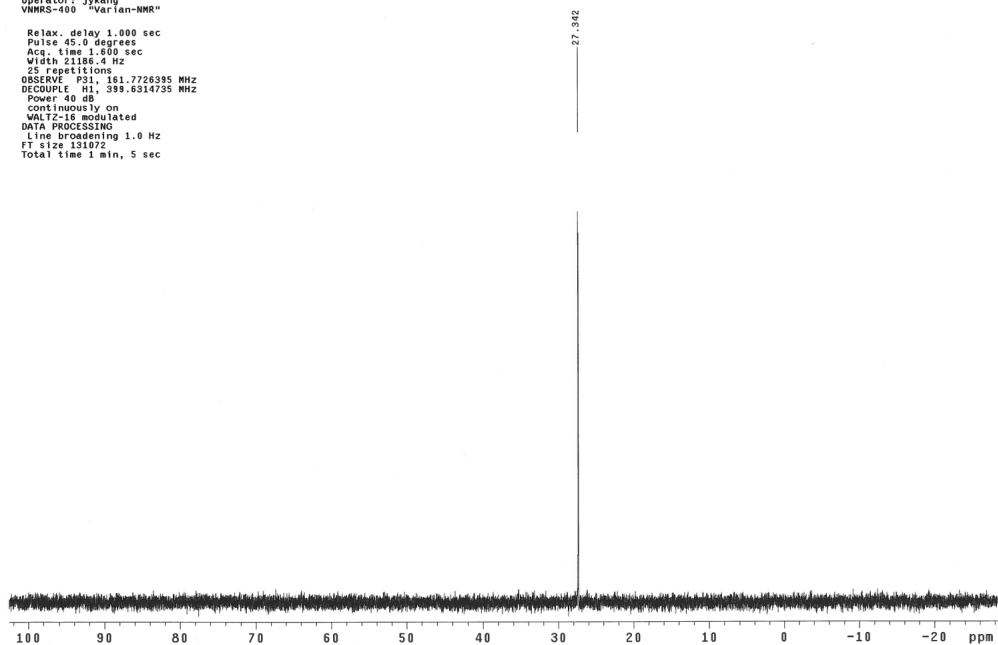
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 2186.4 Hz
25 repetitions
OBSERVE F31: 161.7726395 MHz
DECOUPLE H1: 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec



¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

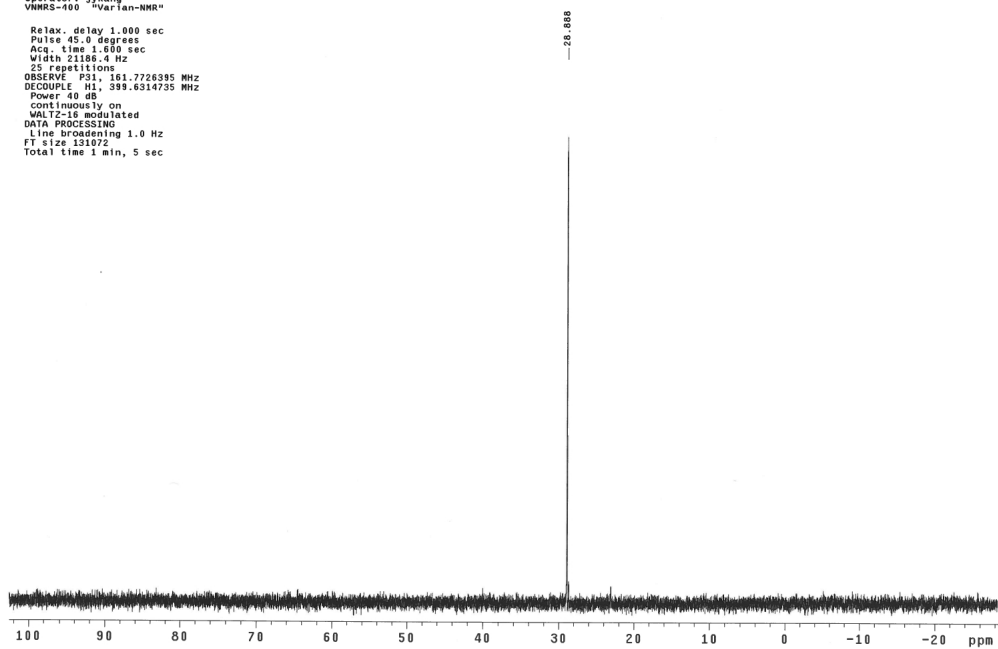
Sample: ja-7-113-p
File: exp
Pulse Sequence: s2pu1
Solvent: cdc13
Temp: 25.0 C / 298.1 K
Operator: jykang
VMRS-400 "Varian-NMR"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21188.4 Hz
25 repetitions
OBSERVE P31, 161.7726395 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

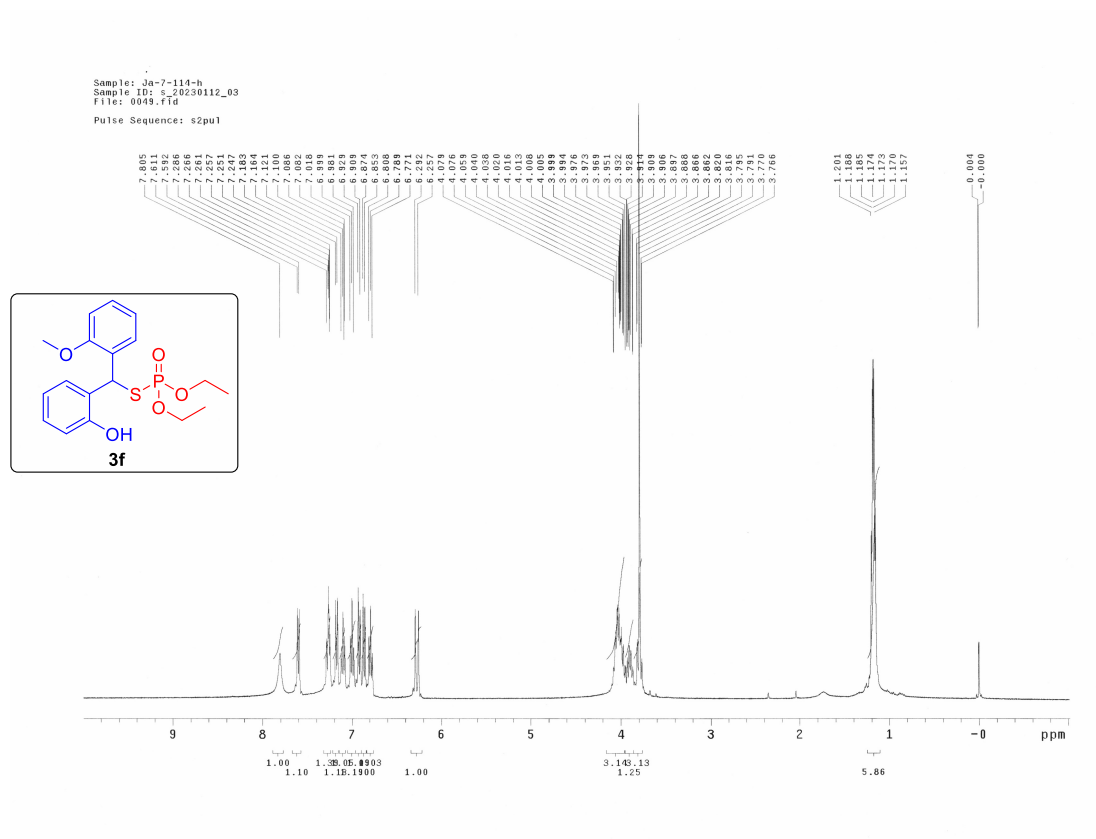
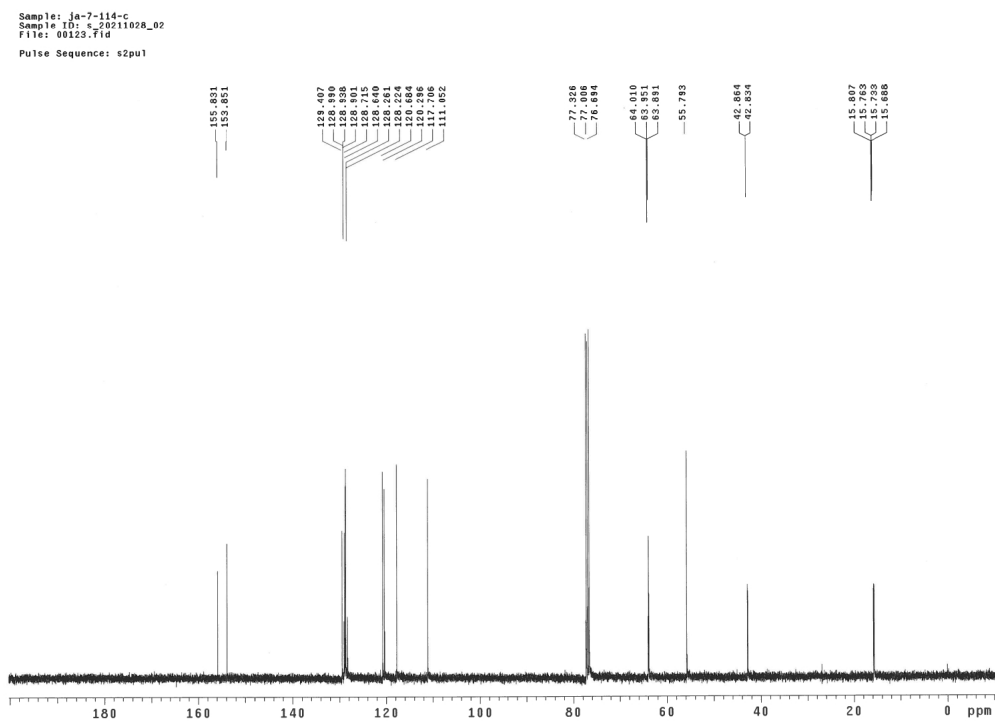


^{31}P NMR (162 MHz) in CDCl_3

Sample: ja-7-63-p
File: exp
Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: jykeng
VNMR5-900 "Varian-NMR"

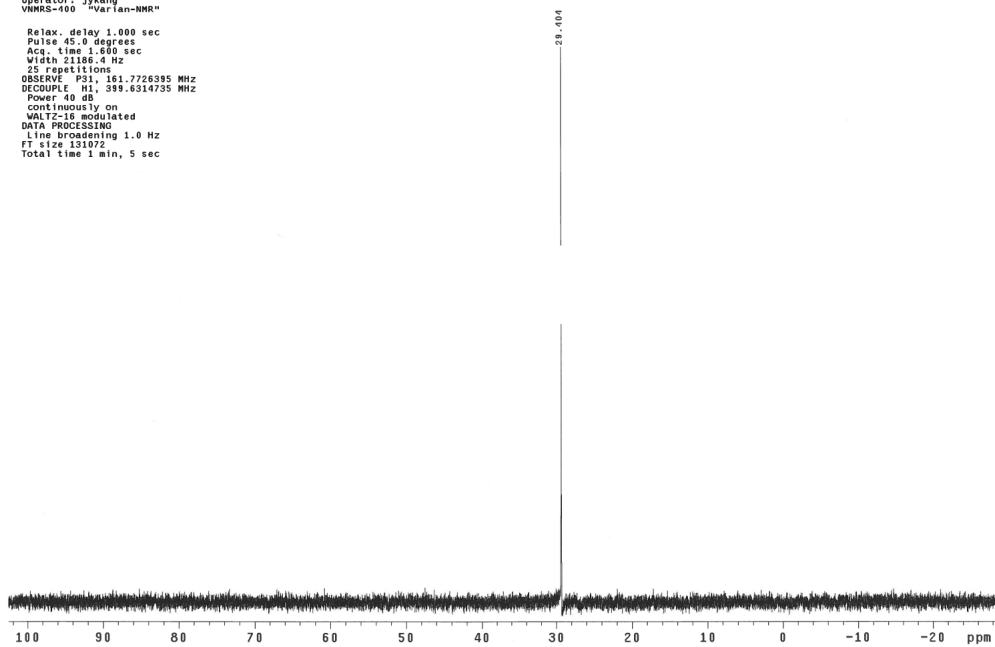
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 2186.4 Hz
25 repetitions
OBSERVE P31, 161.7726395 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

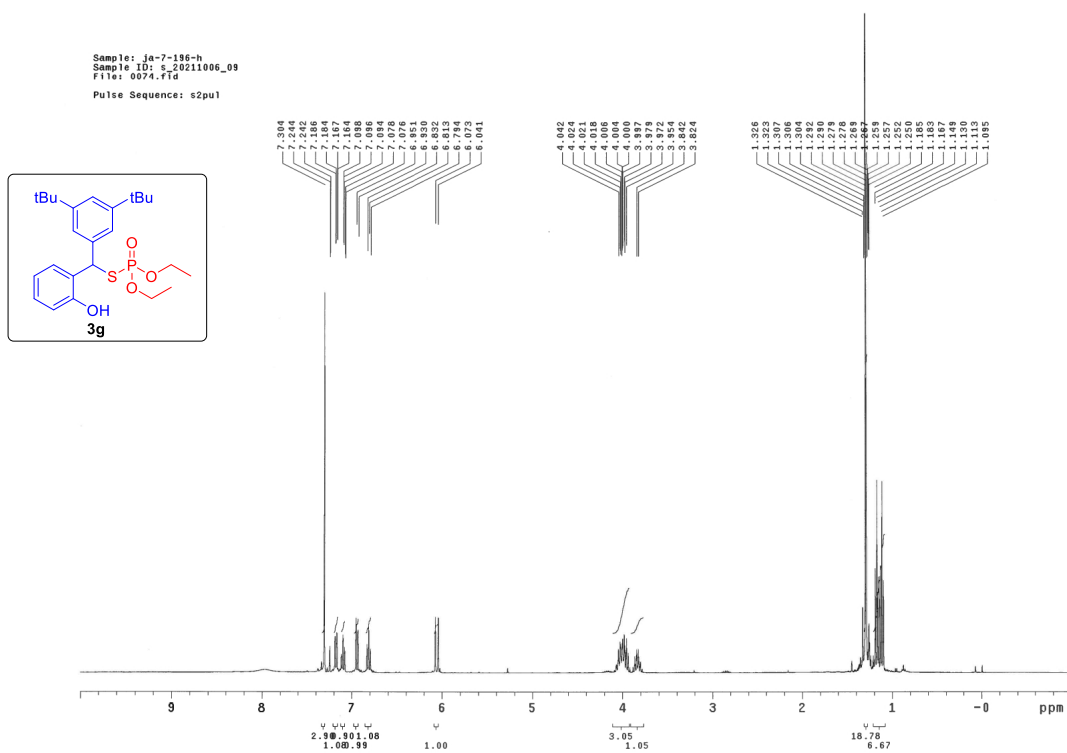
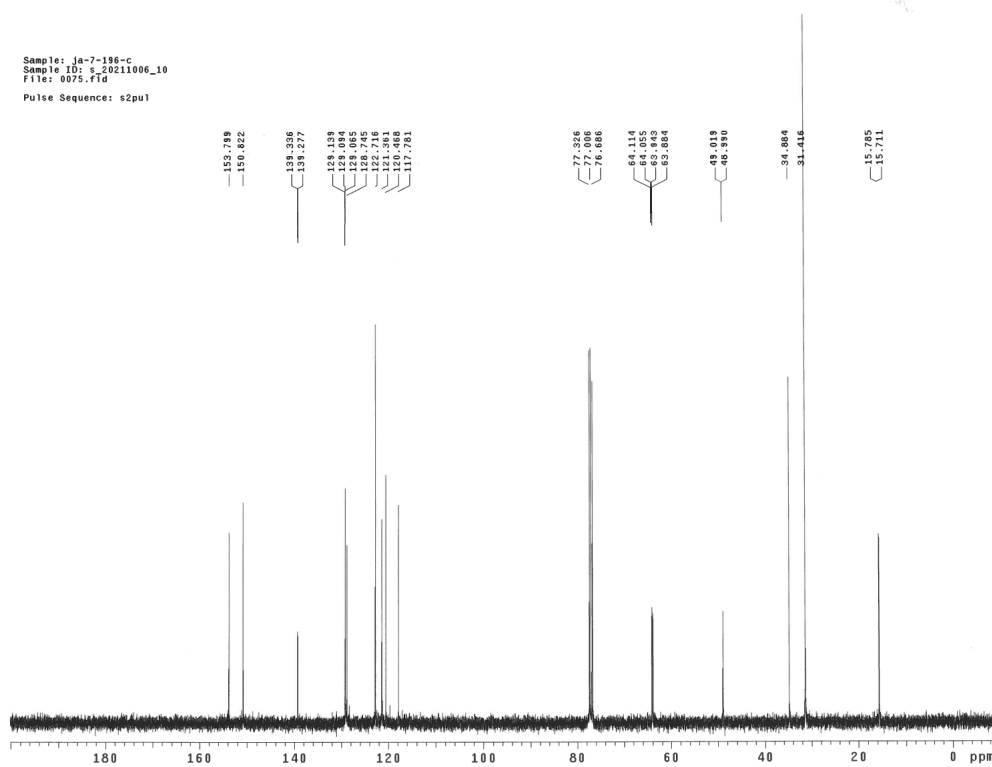


¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

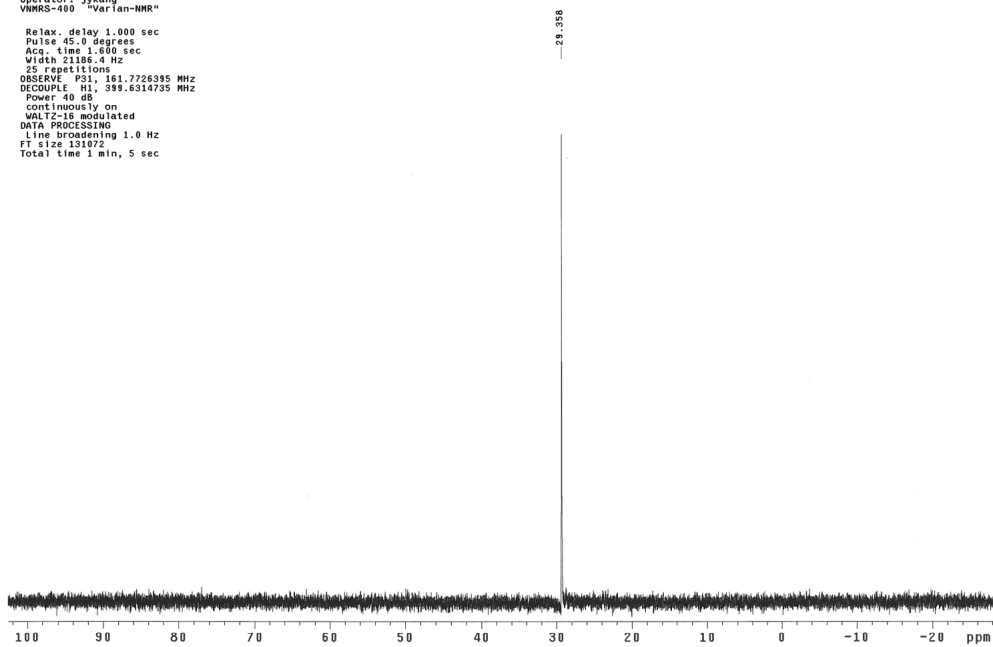
Sample: ja-7-114-p
File: exp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: Jykang
VNMR5-400 "Varian-NMR"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.800 sec
Width 21186.4 Hz
25 repetitions
OBSERVE P31, 161.7726395 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

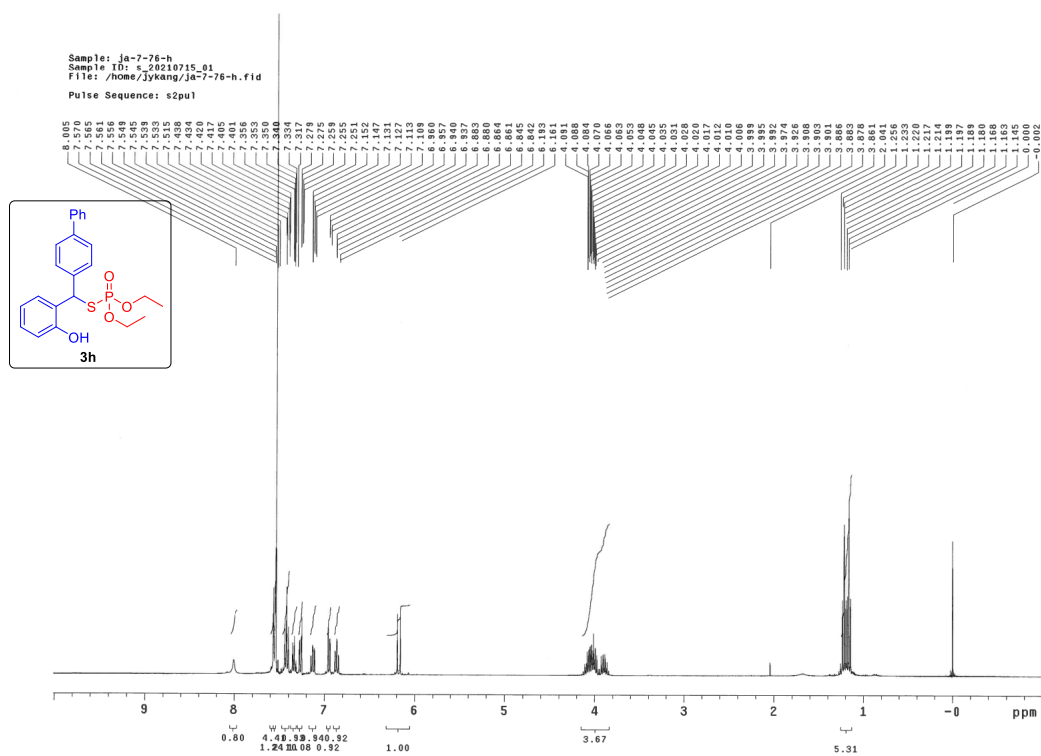
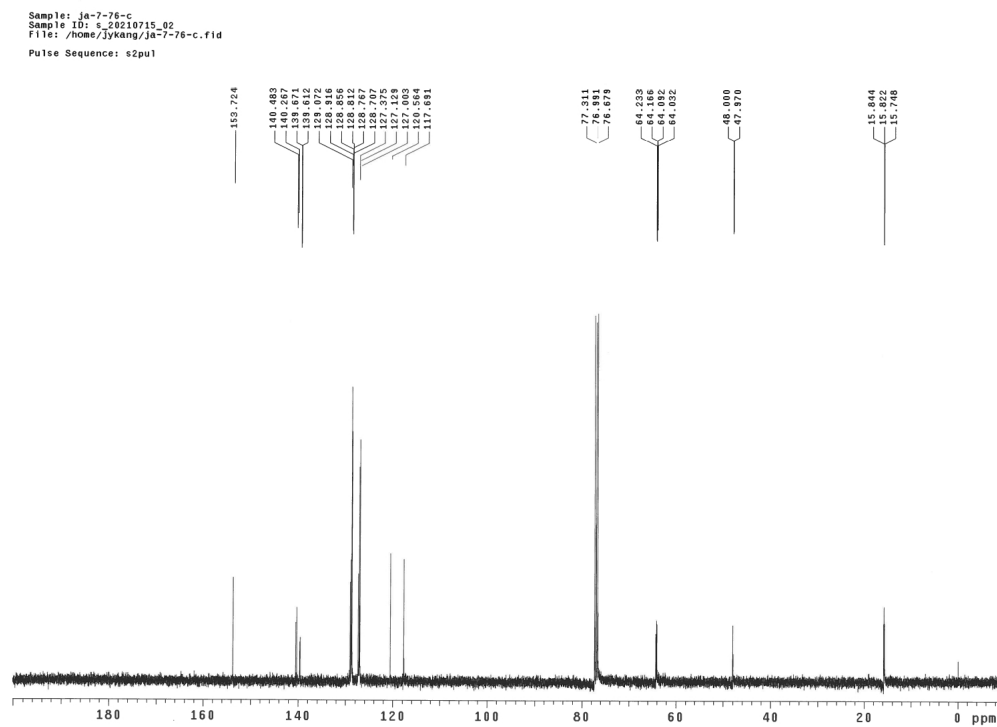


^1H NMR (400 MHz) in CDCl_3  ^{13}C NMR (100.5 MHz) in CDCl_3 

^{31}P NMR (162 MHz) in CDCl_3

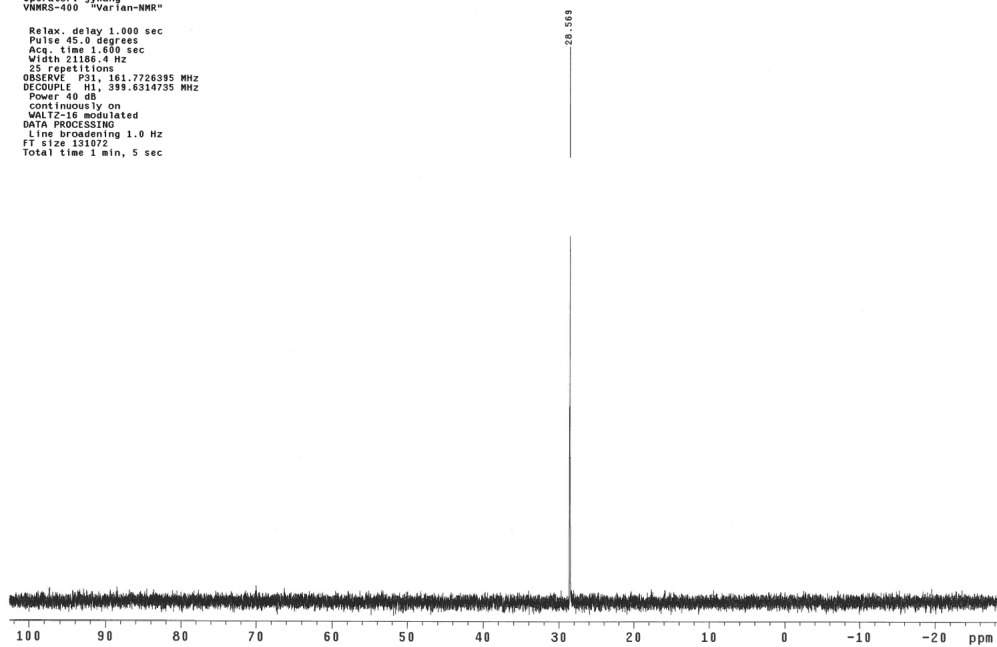
Sample: ja-7-196-p
File: exp
Pulse Sequence: s2pu1
Solvent: cdc13
Temp: 25.0 C / 298.1 K
Operator: jykang
VMRS-400 "Varian-NMR"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21186.4 Hz
25 repetitions
OBSERVE P31, 161.7726395 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

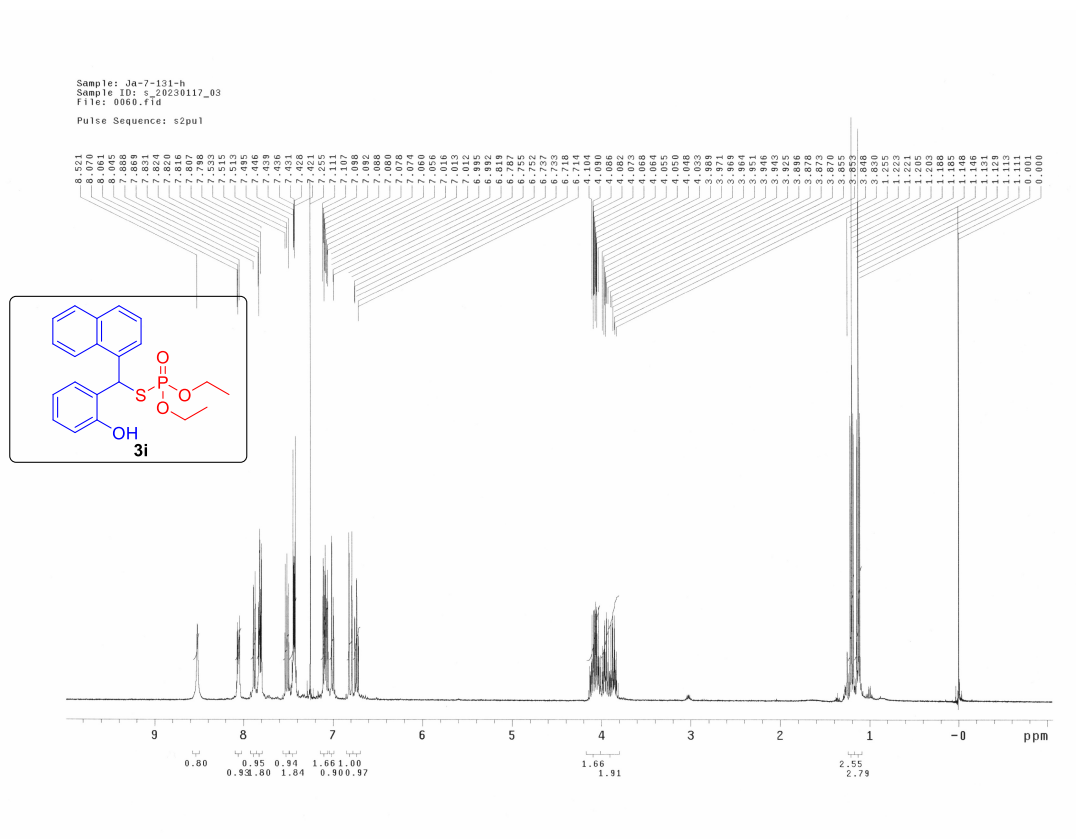
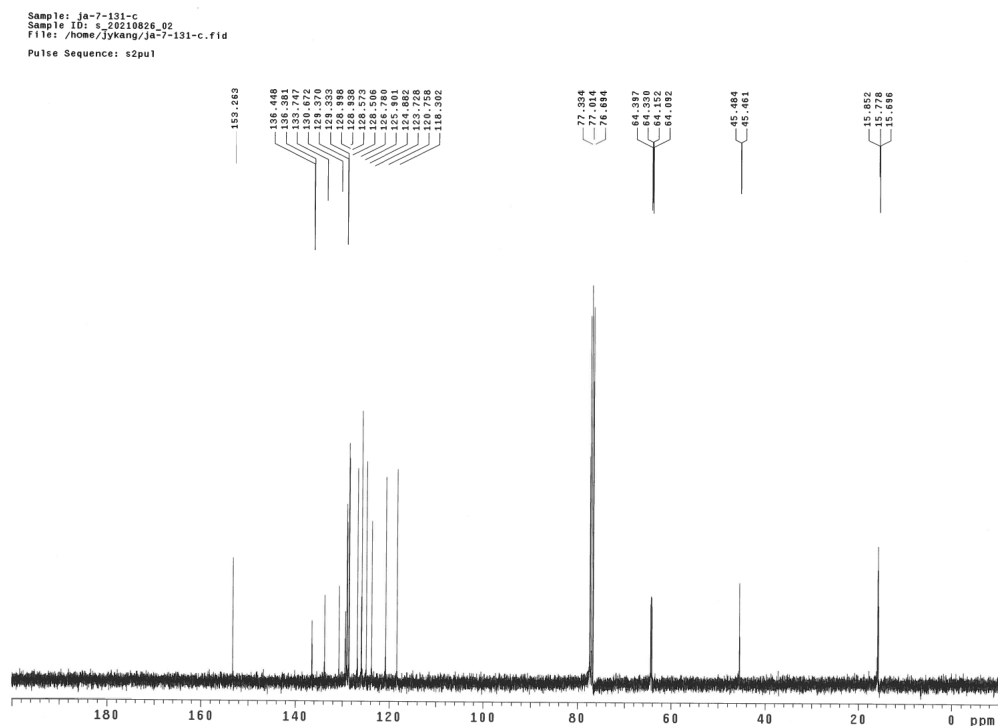


¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

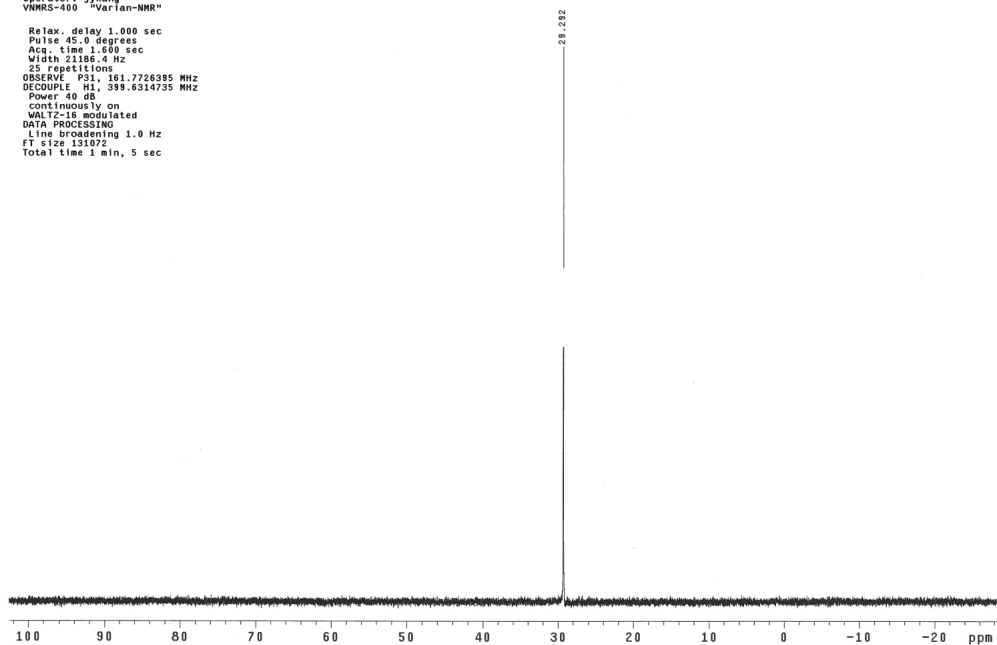
Sample: ja-7-76-p
File: exp
Pulse Sequence: s2pul
Solvent: cdc13
Temp: 25.0 C / 298.1 K
Operator: Jywang
VNMR5-600 Varian-NMR®
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21186.4 Hz
25 repetitions
OBSERVE P31, 161.7726395 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 321072
Total time 1 min, 5 sec

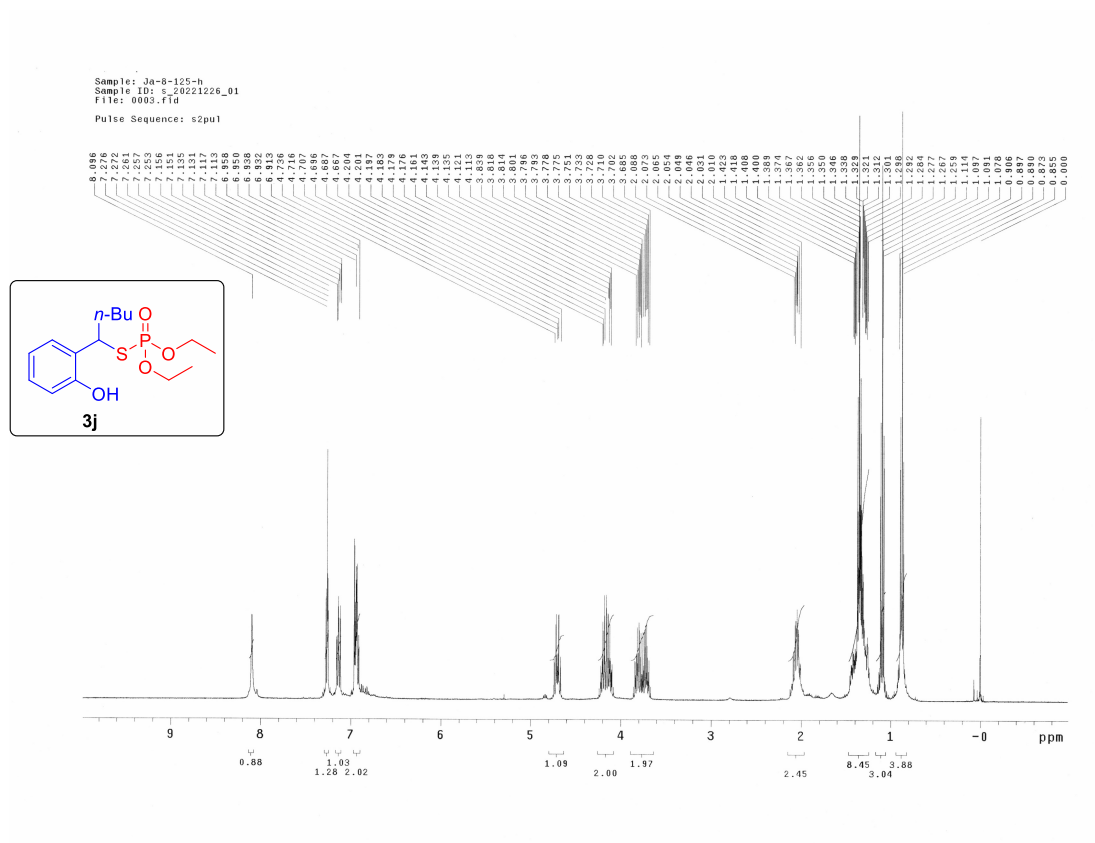
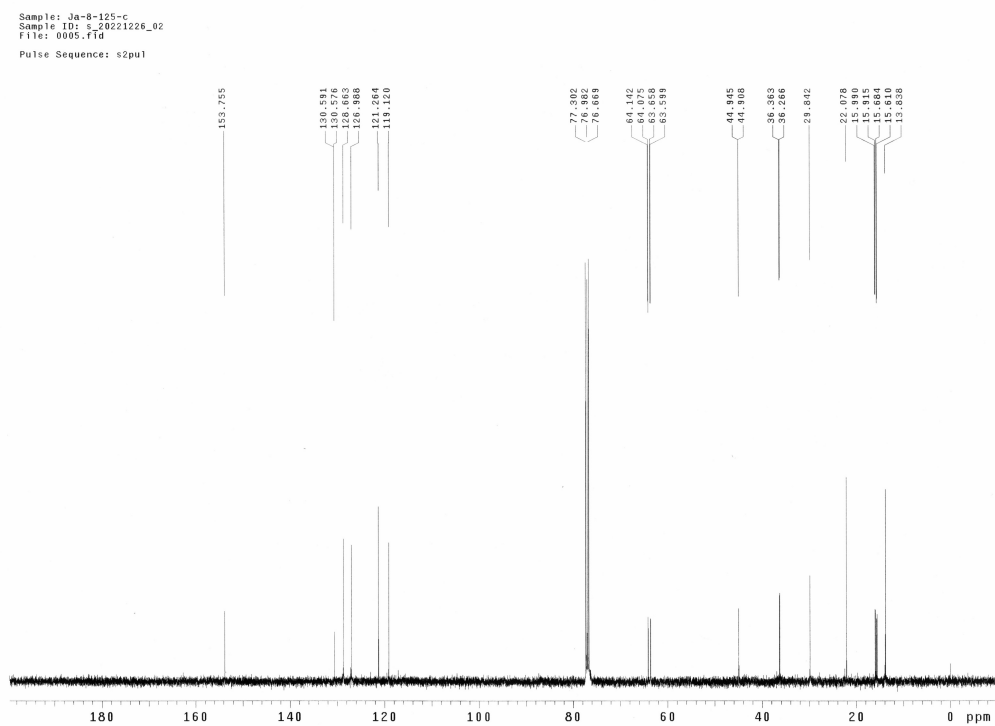


¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

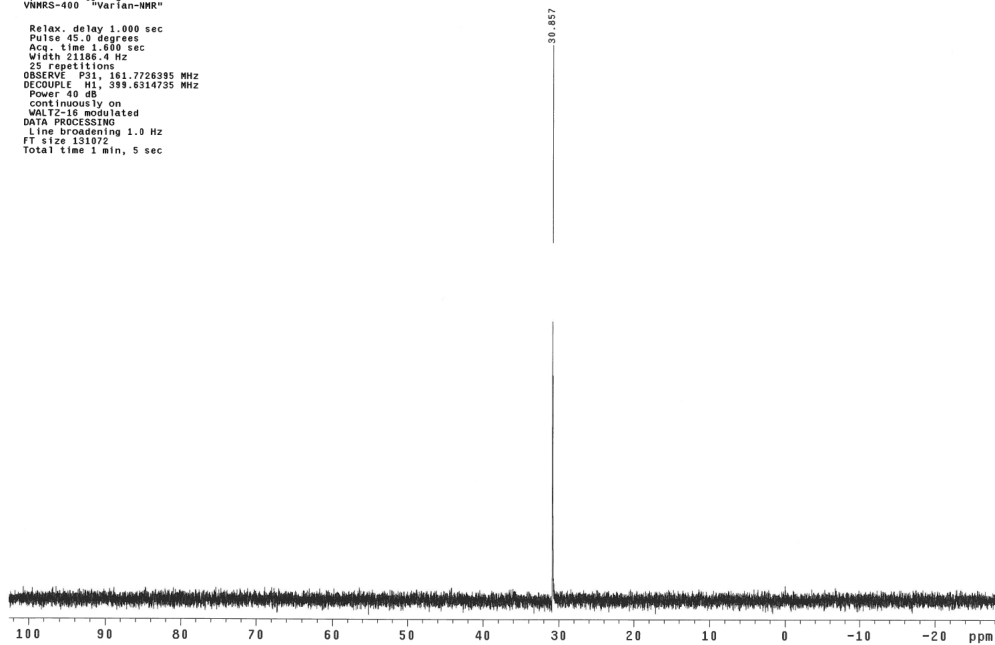
Sample: ja-7-131-p
File: exp
Pulse Sequence: sZpu1
Solvent: cdc13
Temp. 25.0 C / 298.1 K
Operator: Jywang
VNMR5-600 Varian-NMR
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 2186.4 Hz
25 repetitions
OBSERVE F31: 161.7726385 MHz
DECUPLE H1: 398.9314735 MHz
Power 40 dB
continuous on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

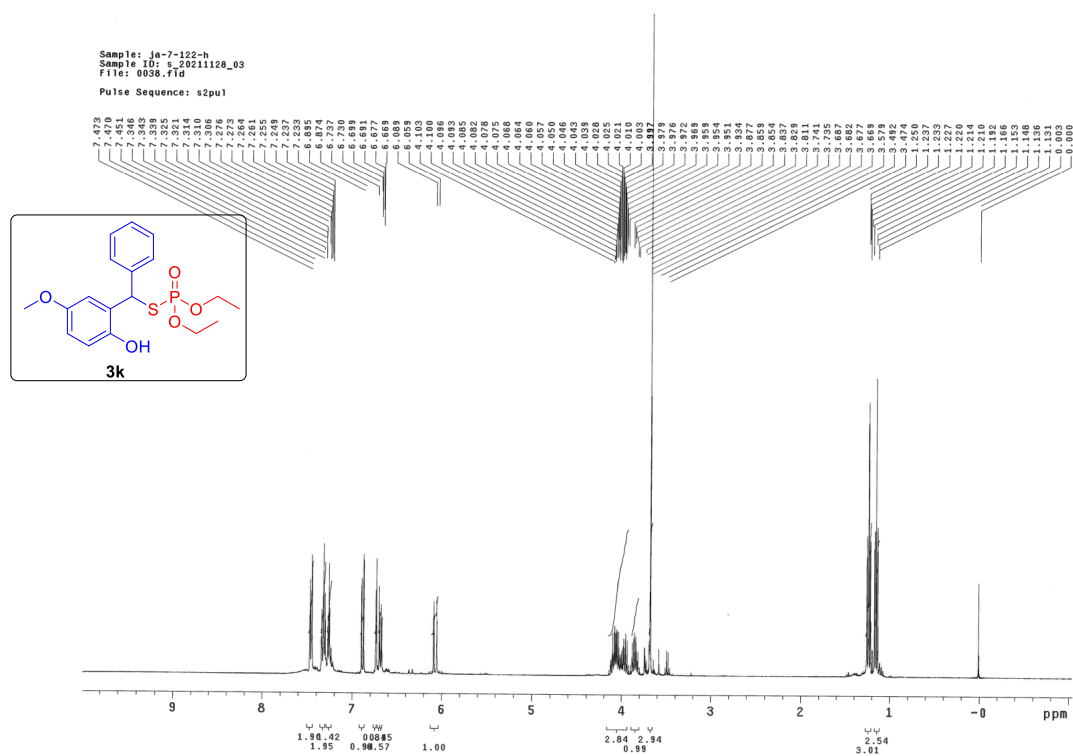
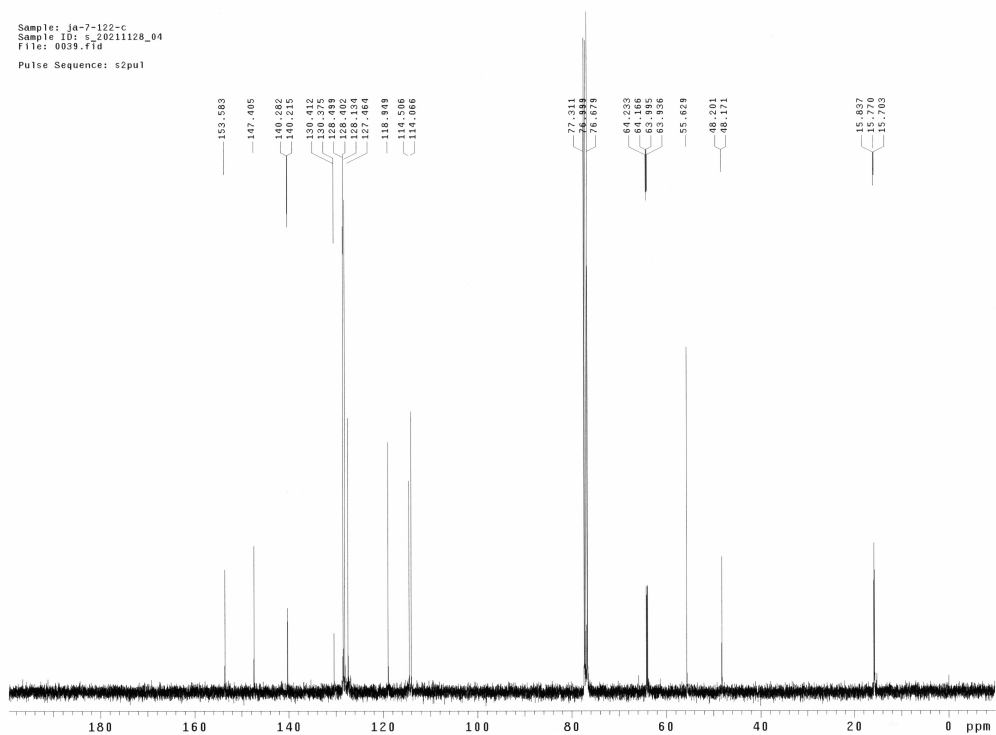


¹H NMR (400 MHz) in CDCl₃¹³C NMR (100.5 MHz) in CDCl₃

^{31}P NMR (162 MHz) in CDCl_3

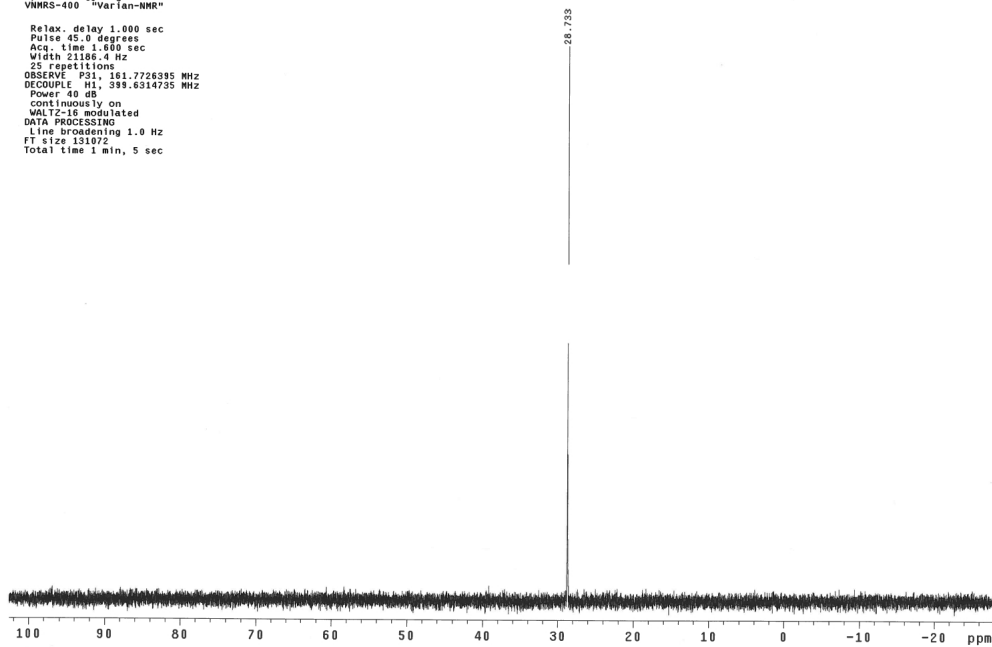
Sample: ja-8-125-p
File: exp
Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: jykang
VNMR-400 "Varian-NMR"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.800 sec
Width 21186.4 Hz
25 repetitions
OBSERVE P31, 161.7726395 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
cont inously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

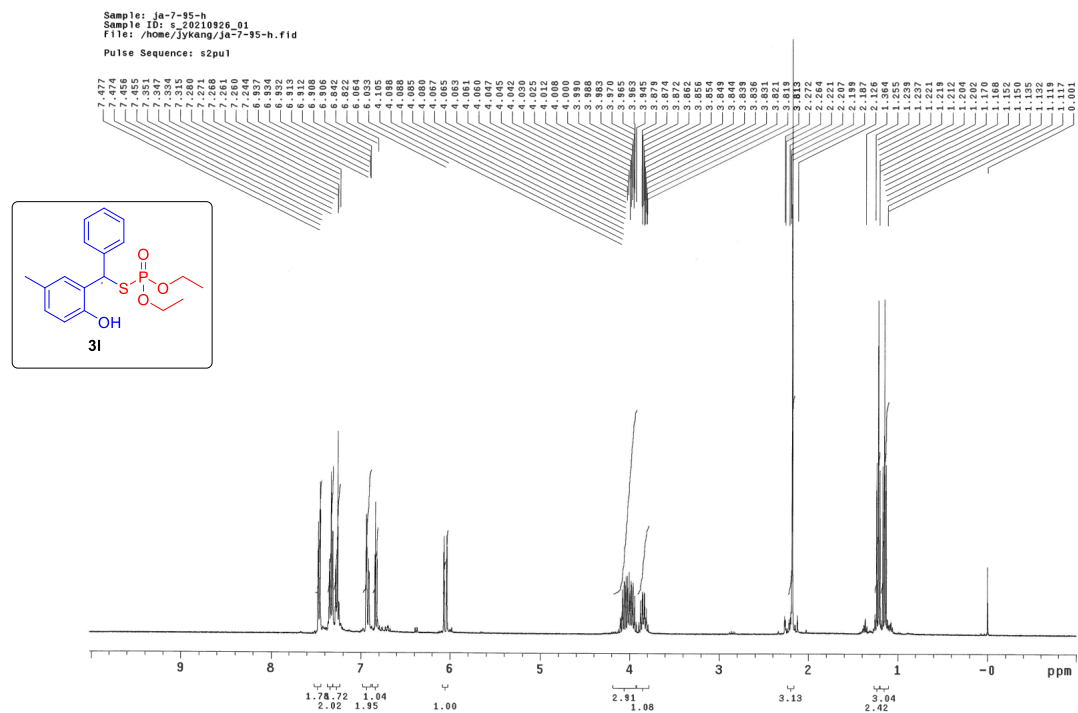
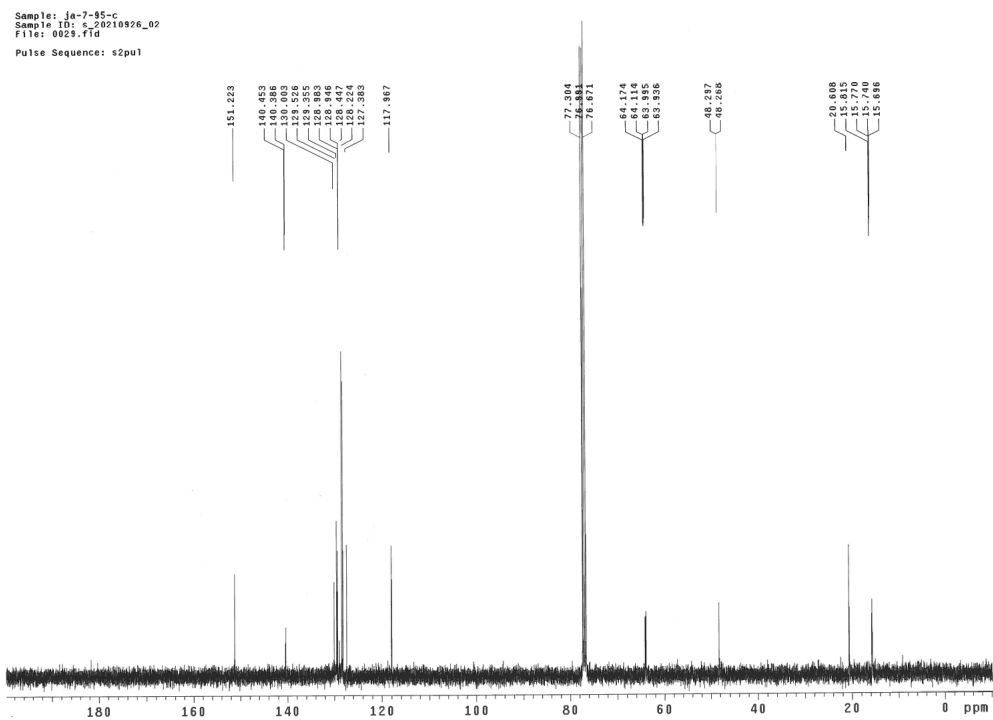


¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

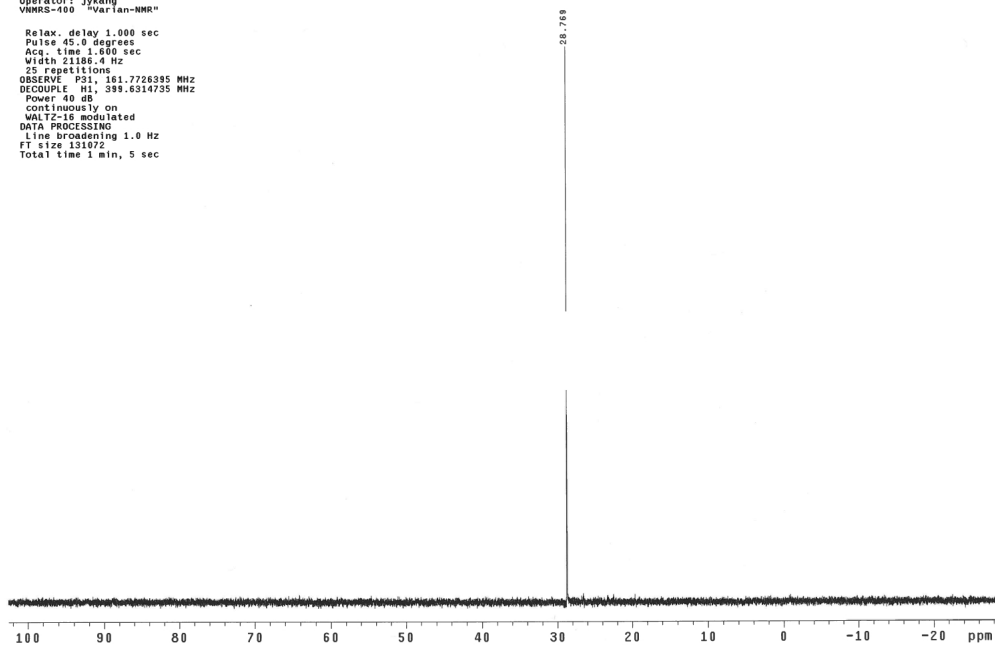
Sample: ja-7-122-p
File: exp
Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: jykang
VNMRS-400 "Varian-NMR"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.800 sec
Width 21186.4 Hz
25 repetitions
OBSERVE P31, 161.7726395 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
continously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

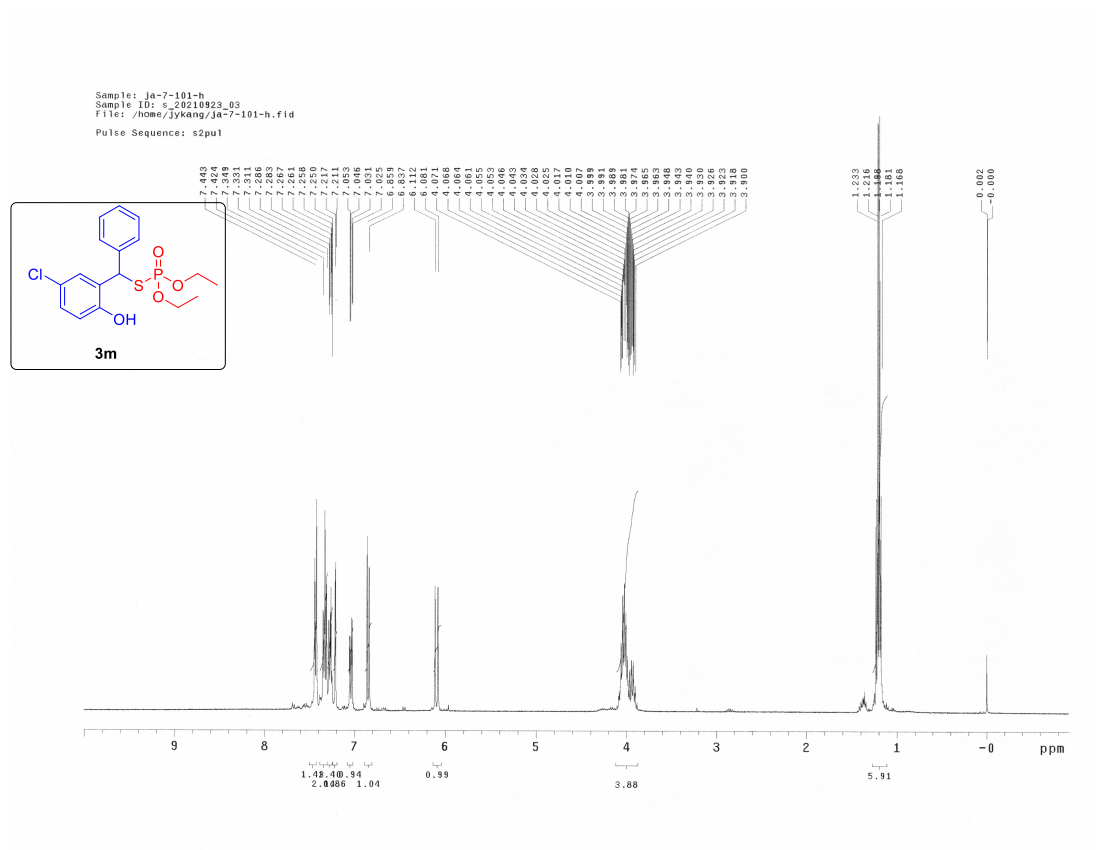
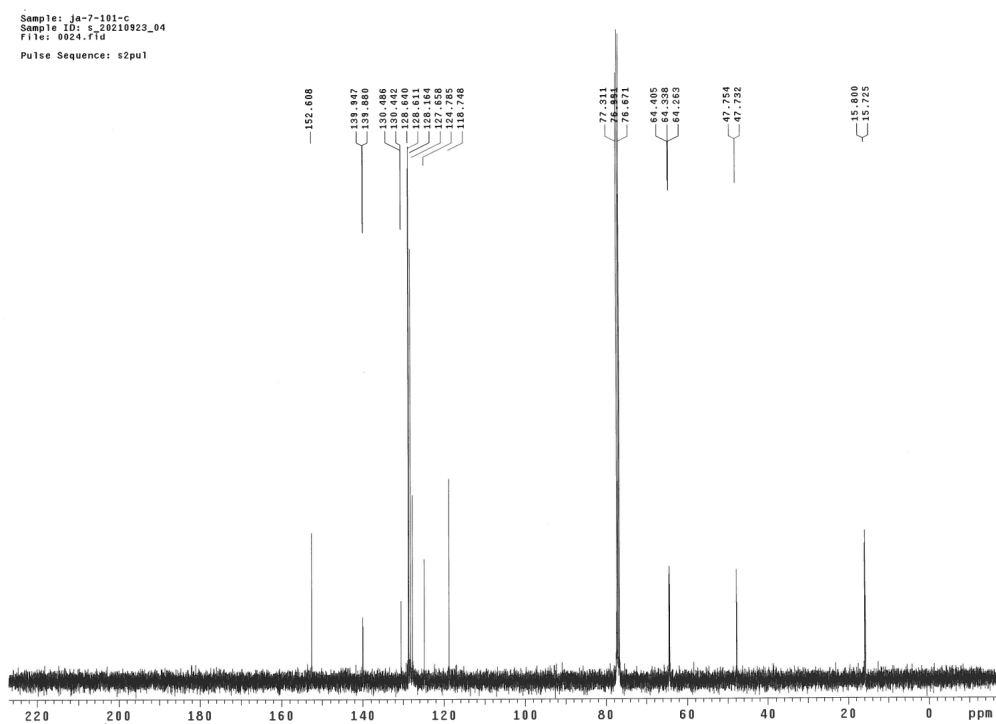


¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

Sample: ja-7-85-p
File: exp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: jykang
VMRS-400 "Varian-NMR"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.800 sec
Width 21186.4 Hz
25 repetitions
OBSERVE P31, 161.7726395 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
Ft size 131072
Total time 1 min, 5 sec

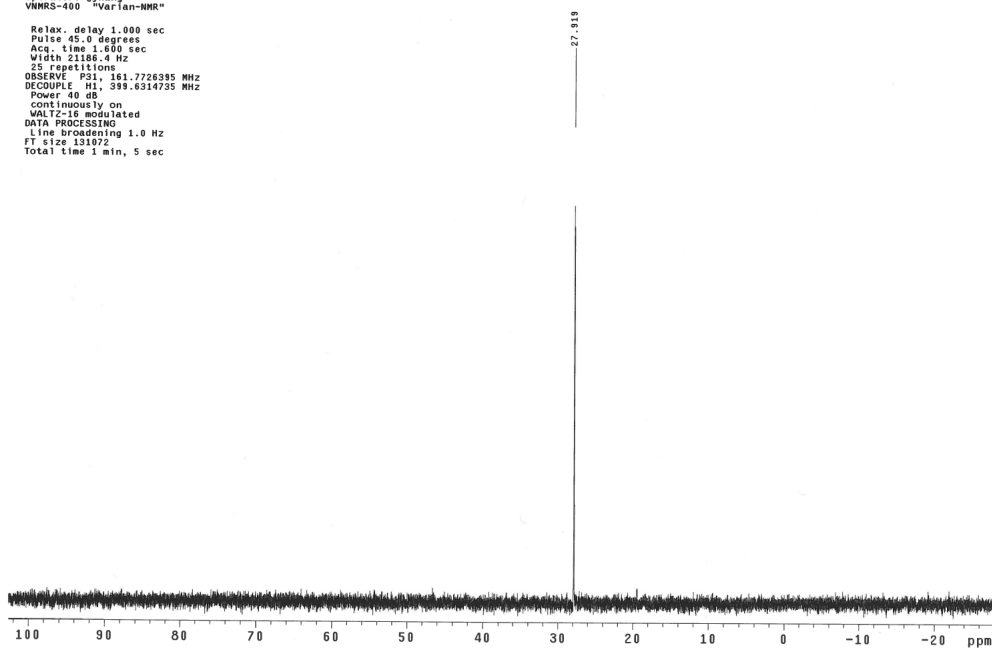


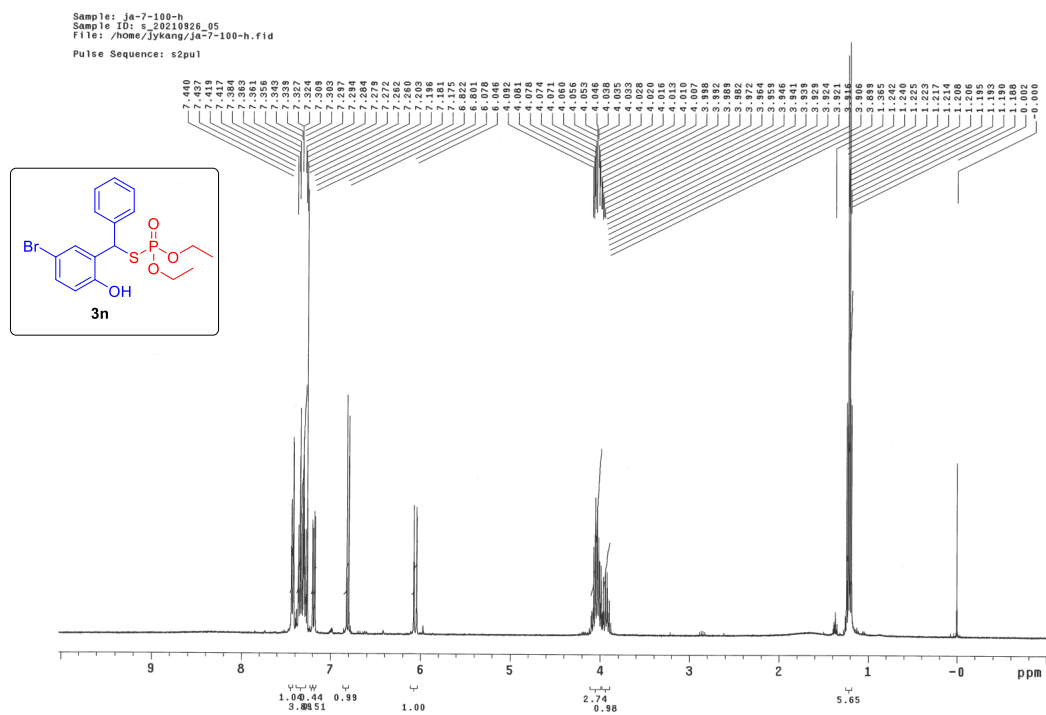
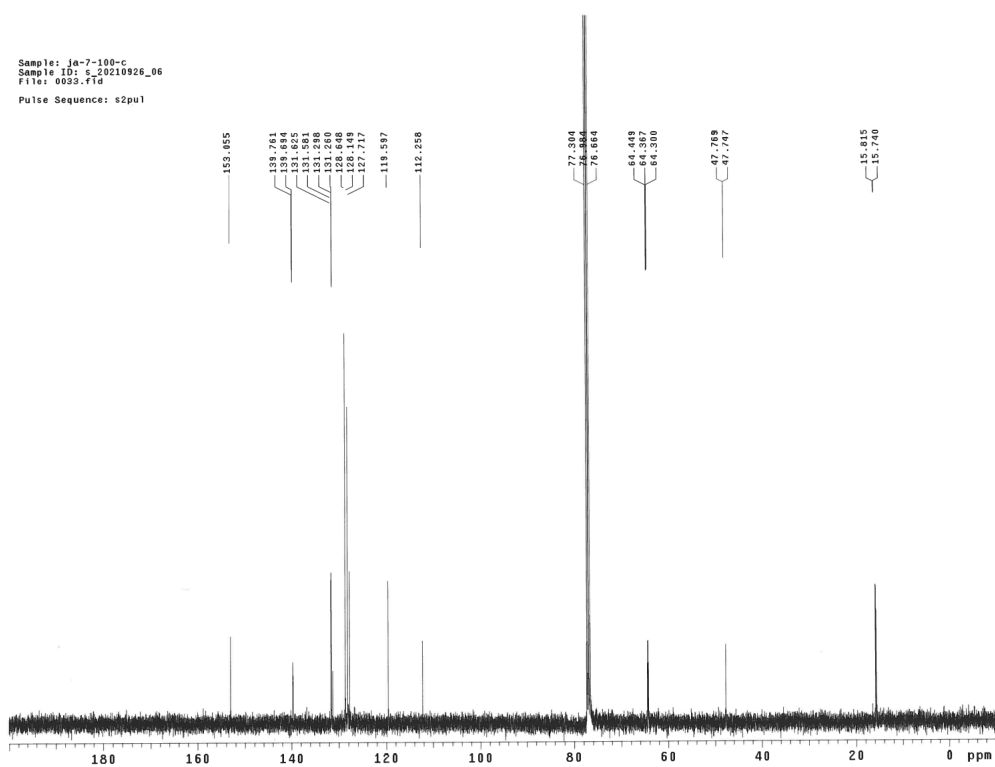
¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

Sample: ja-7-101-p
File: exp
Pulse Sequence: s2pul
Solvent: cdc13
Temp: 25.0 C / 298.1 K
Operator: jykeng
VNMR5-900 "Varian-NMR"

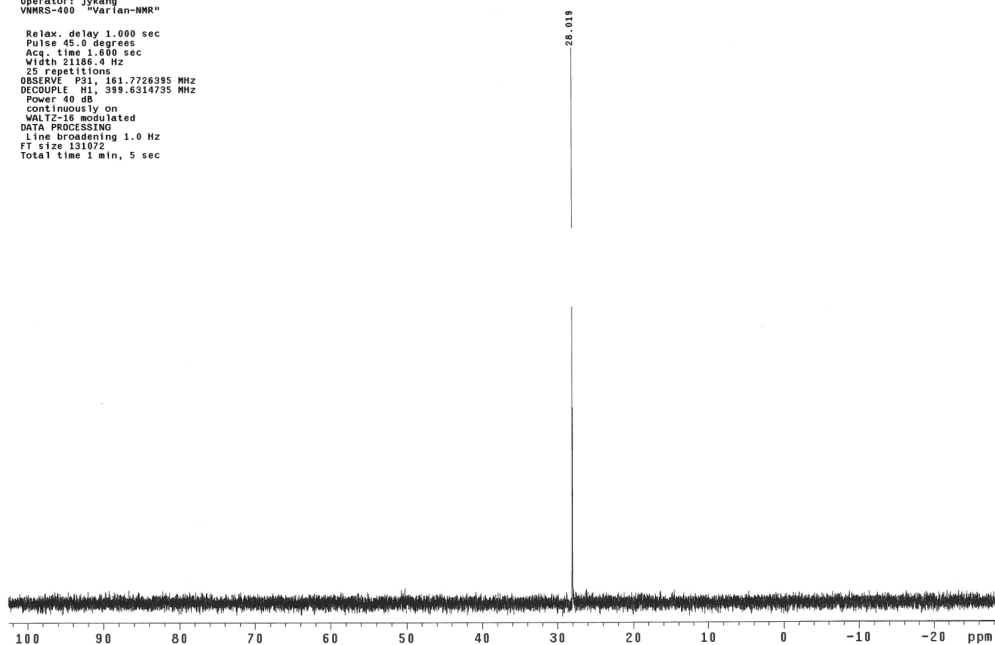
Relax: delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21186.4 Hz
25 repetitions
OBSERVE P21, 161.7726385 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 db
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec



¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

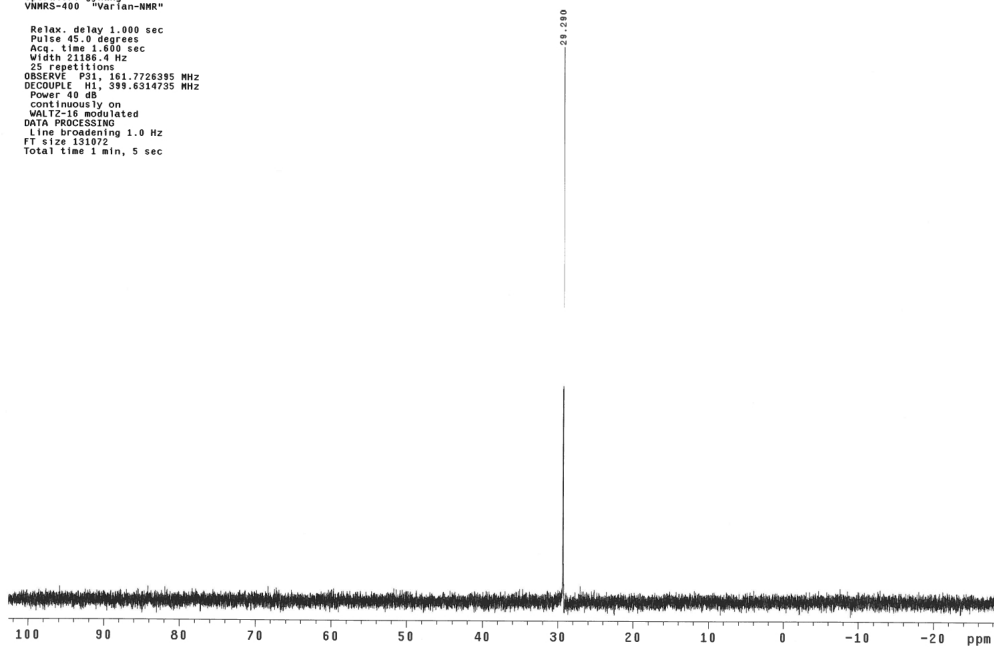
^{31}P NMR (162 MHz) in CDCl_3

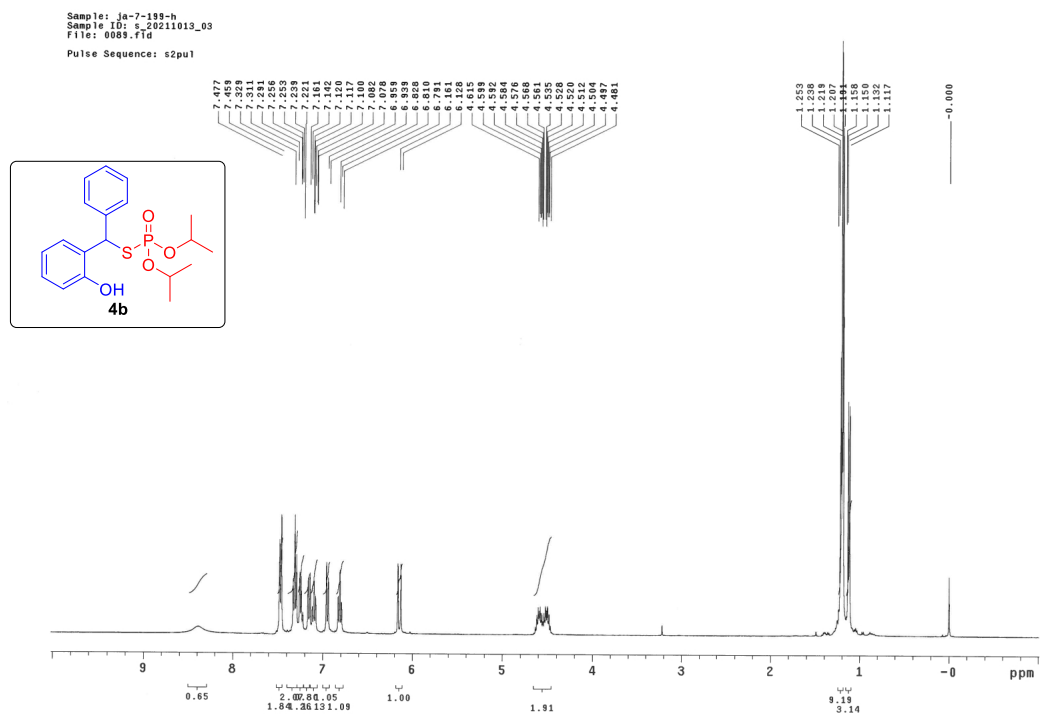
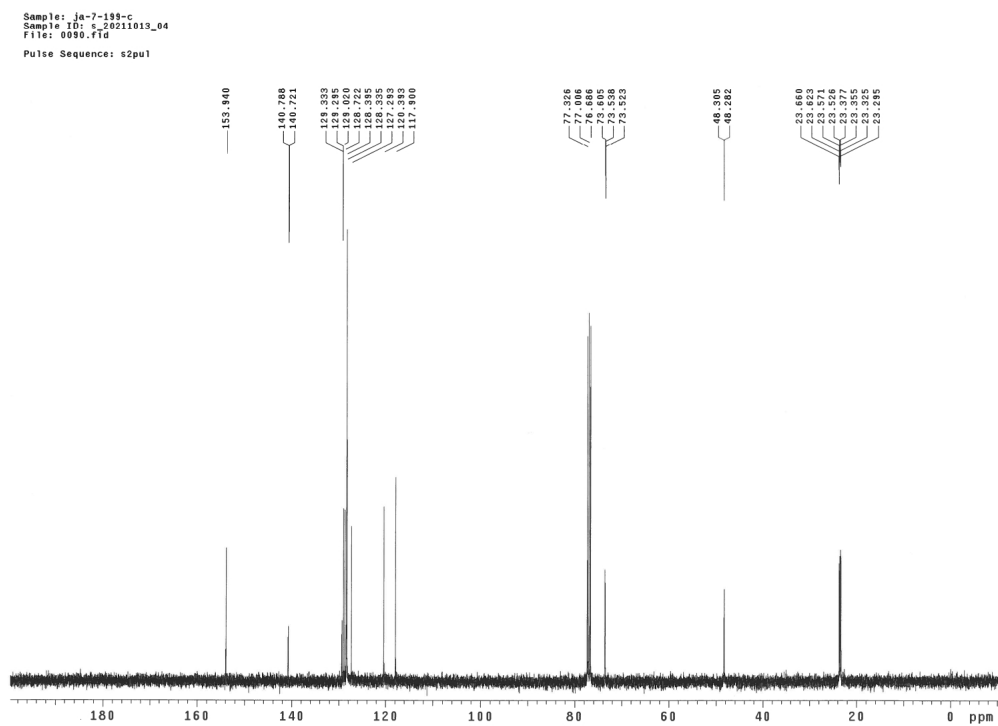
Sample: ja-7-100-p
File: exp
Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: Jykwang
VMRCS-000 Varian-NMR®
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21386.4 Hz
25 repetitions
OBSERVE F31: 161.7726385 MHz
DECOUPLE H1: 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec



^{31}P NMR (162 MHz) in CDCl_3

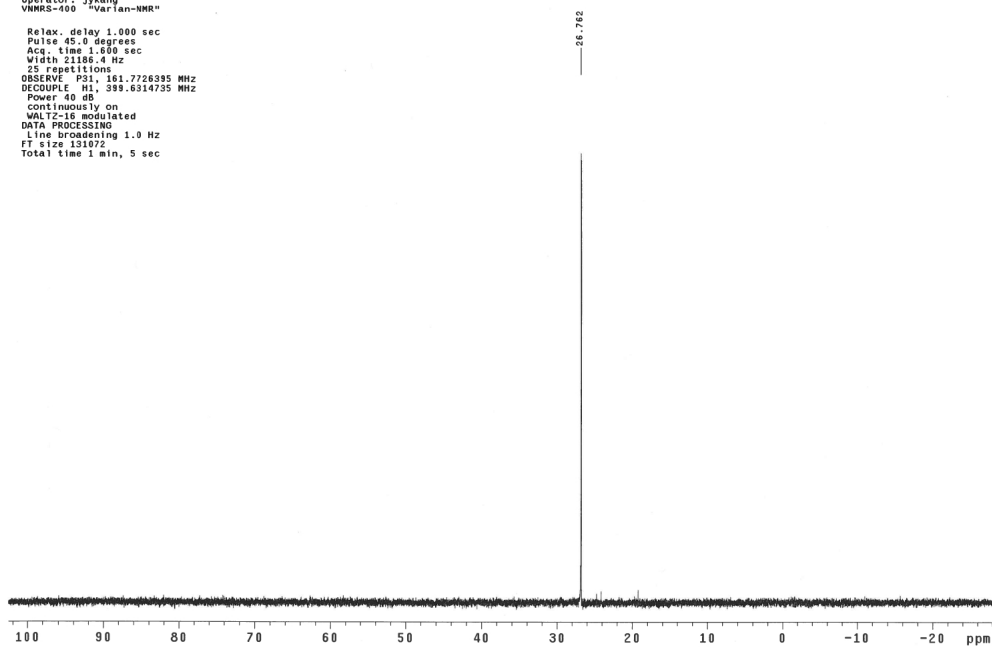
Sample: ja-7-198-p
File: exp
Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: jykeng
VNMRS-400 "Varian-NMR"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21186.4 Hz
25 repetitions
OBSERVE F21, 161.7726395 MHz
DECOUPLE H1, 399.6314735 MHz
Power 48 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

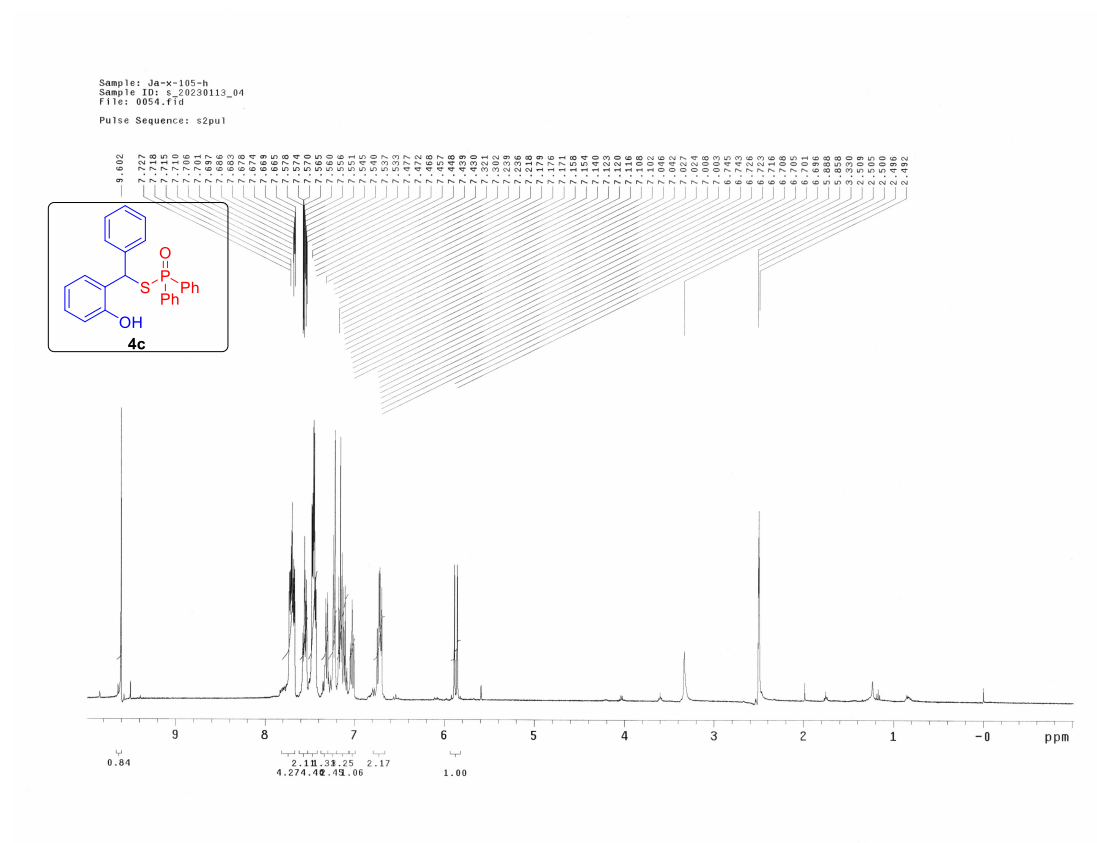
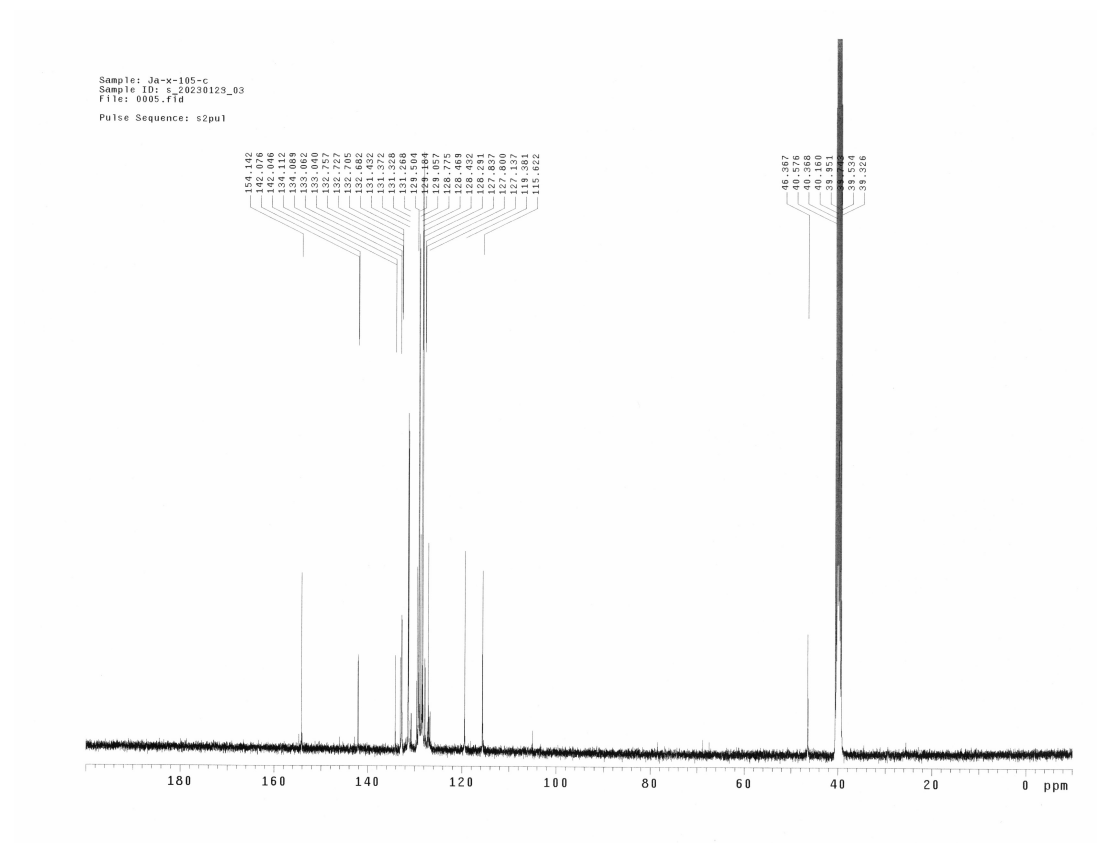


¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

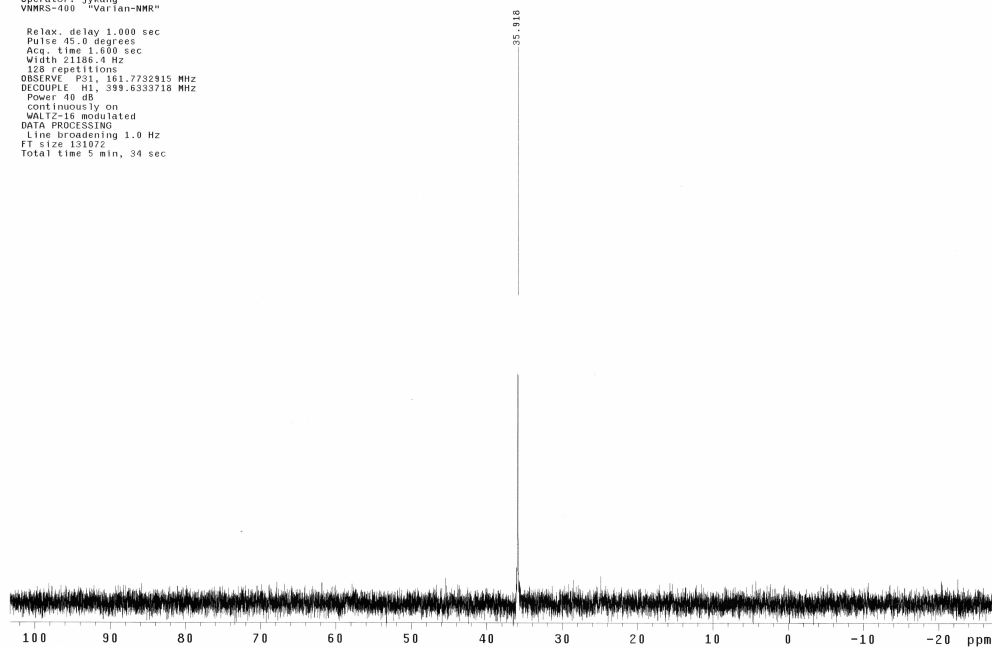
Sample: ja-7-199-p
File: exp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JyKang
VNMR5-600 Varian-NMR
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 23386.4 Hz
25 repetitions
OBSERVE P31 161.7726385 MHz
DECOUPLE H1 599.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

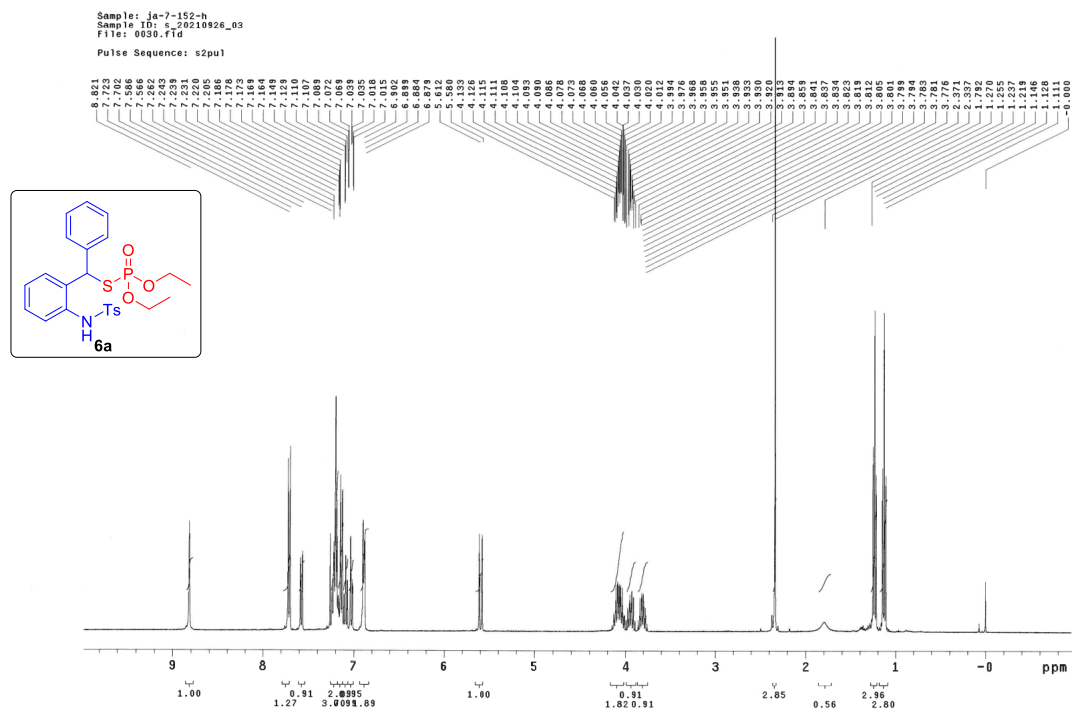
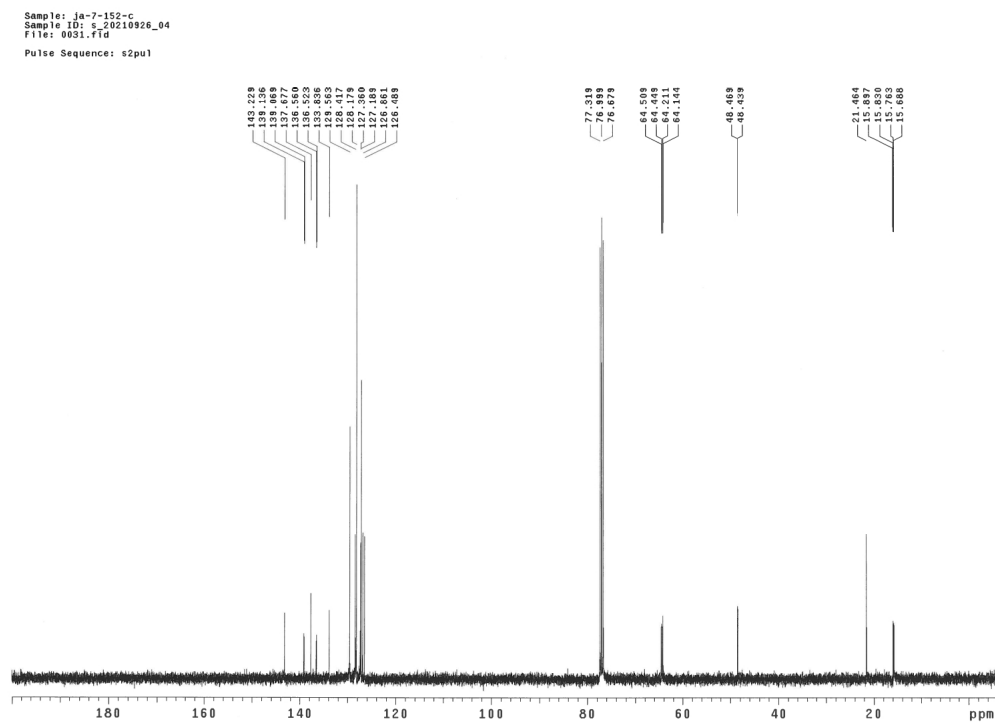


¹H NMR (400 MHz) in DMSO-d₆**¹³C NMR (100.5 MHz) in DMSO-d₆**

^{31}P NMR (162 MHz) in DMSO-d₆

Sample: ja-x-105-p
File: exp
Pulse Sequence: s2pul
Solvent: dmsc
Temp: 25.0 C / 298.1 K
Operator: jykang
VMRS-400 "Varian-NMR"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21186.4 Hz
128 repetitions
OBSERVE P31, 161.7792915 MHz
DECUPLE H1, 509.6333716 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 5 min, 34 sec

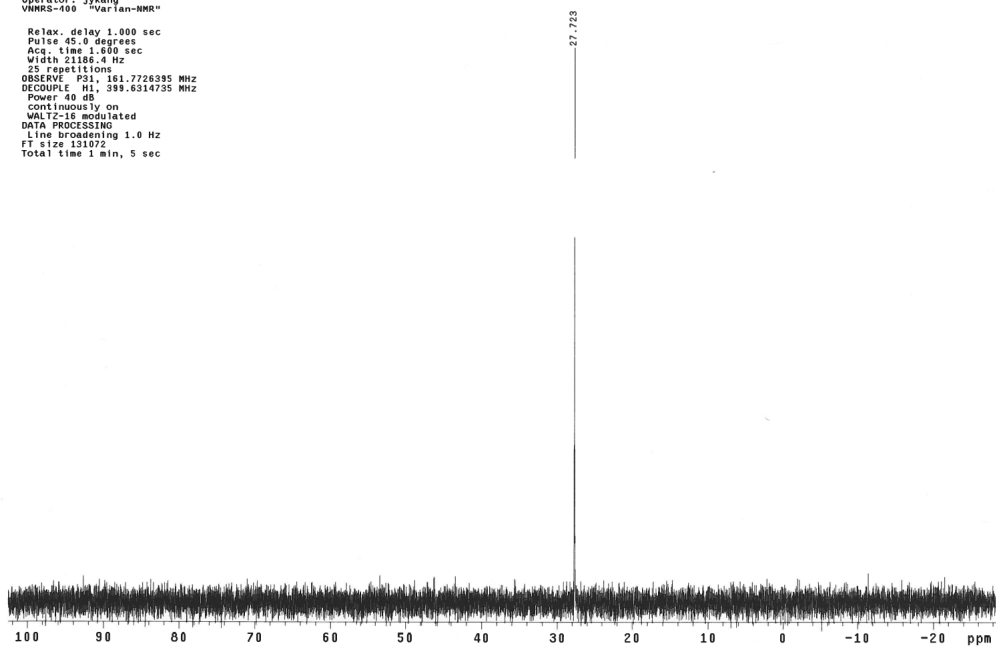


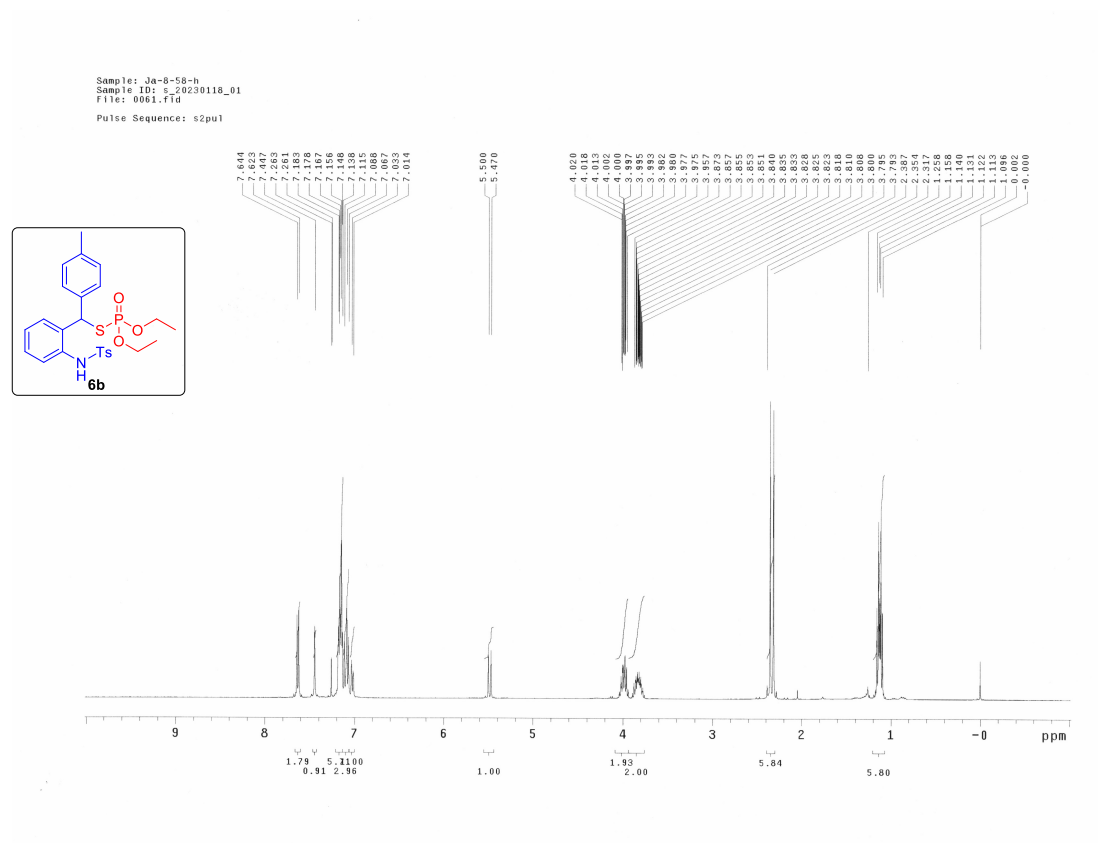
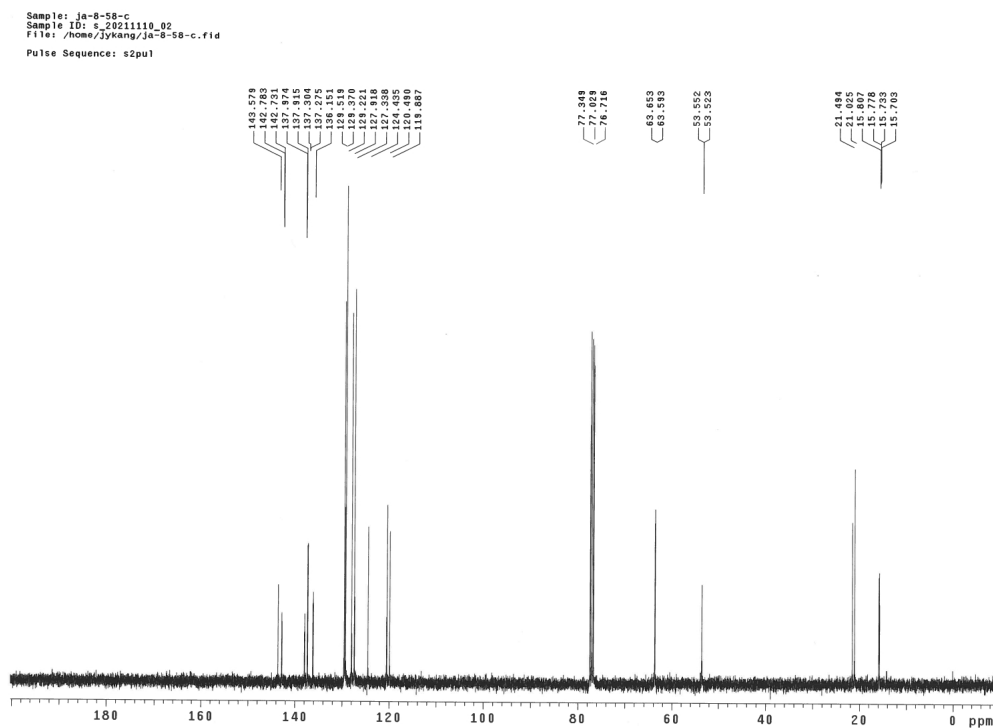
¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

Sample: ja-7-152-p
File: exp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JyKang
VNMR3-000 Varian-NMR®

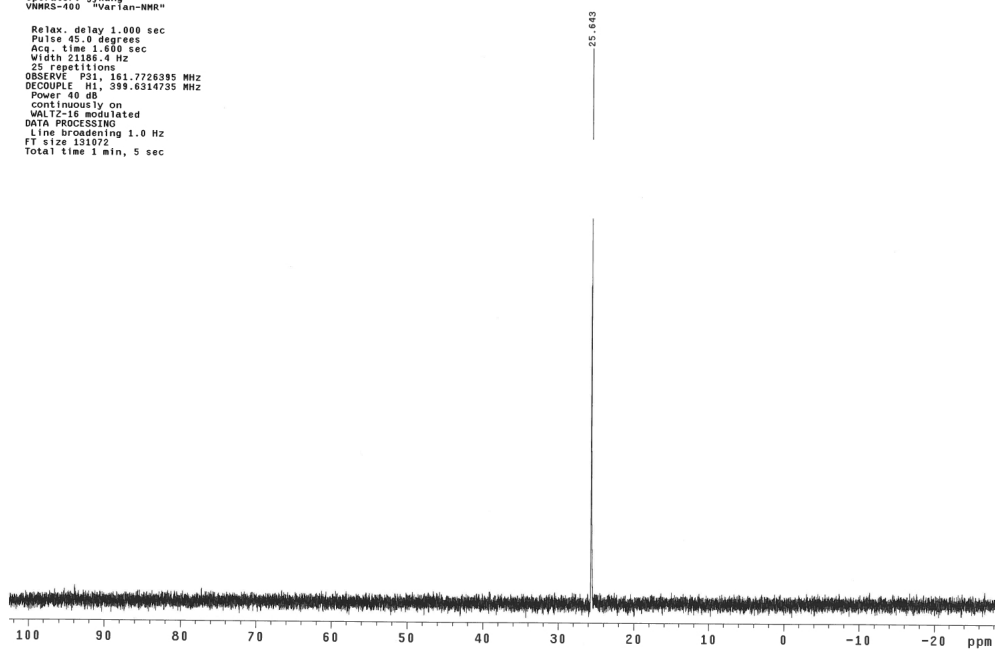
Relax. delay 1.000 sec
Pulse 45.0 degree
Acq. time 1.600 sec
Width 21386.4 Hz
25 repetitions
OBSERVE F31 161.7726395 MHz
DECOUPLE H1 599.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 size 131072
Total time 1 min, 5 sec

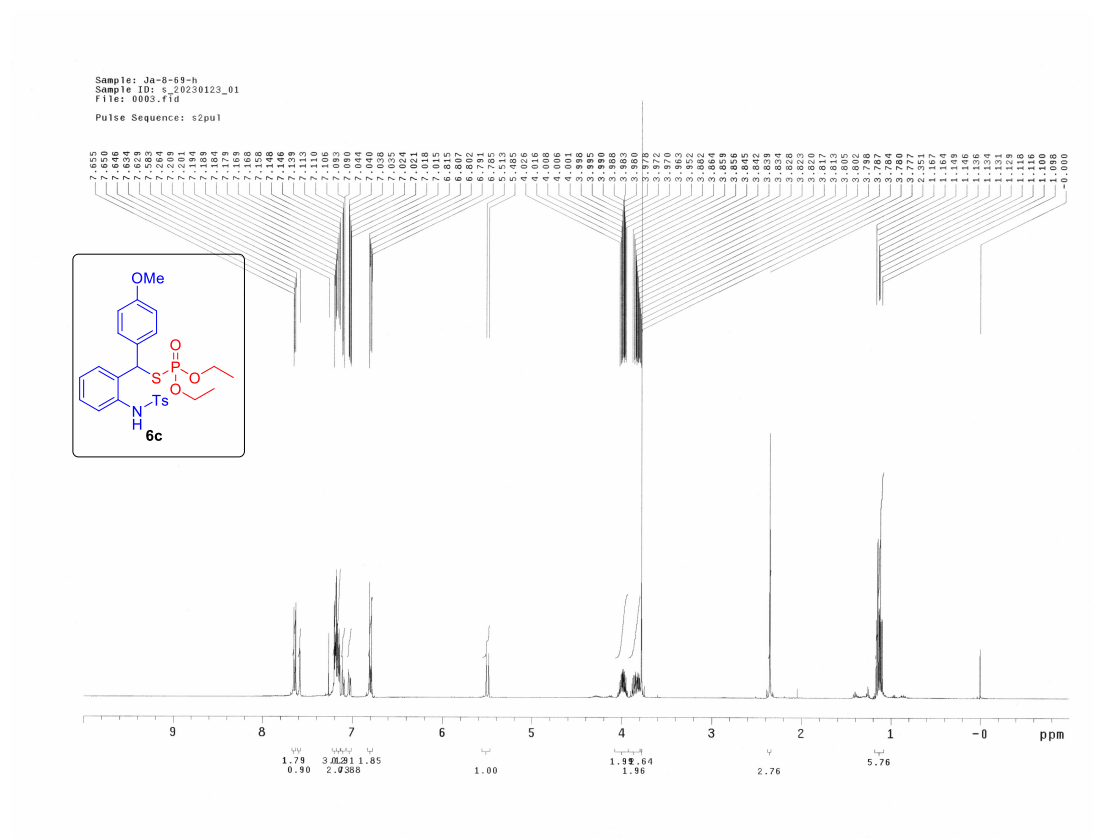
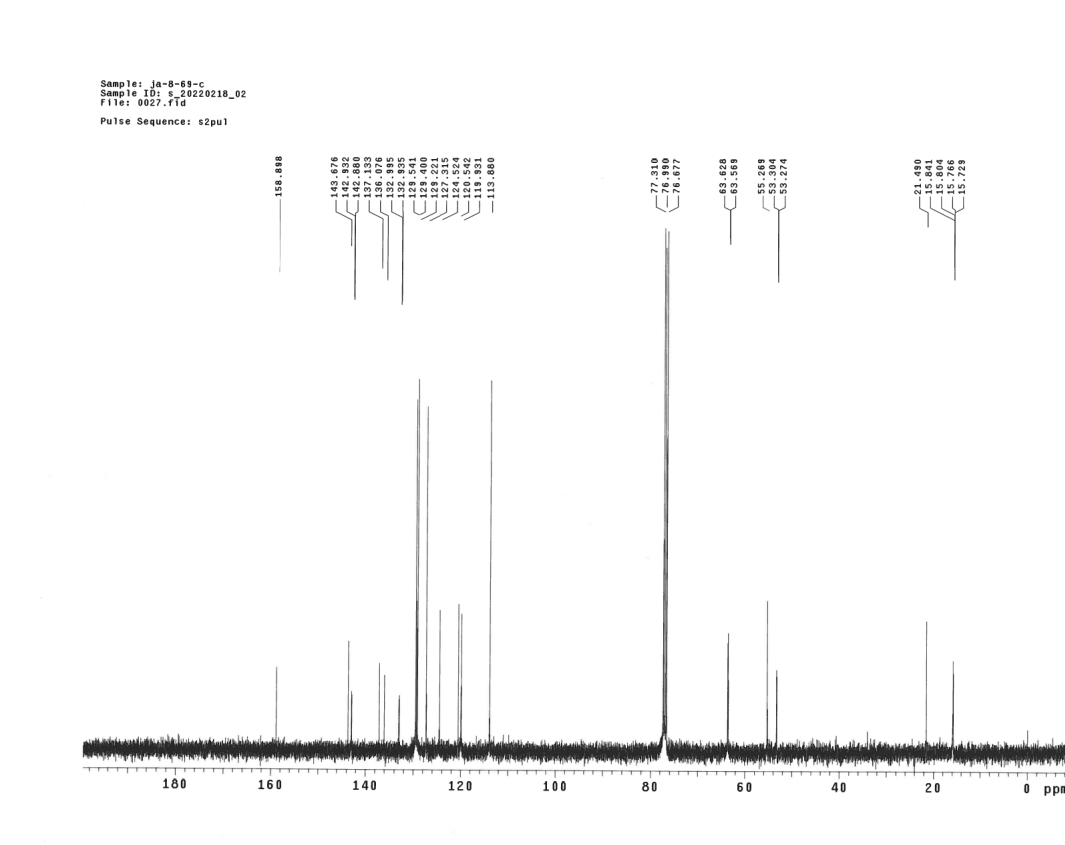


¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

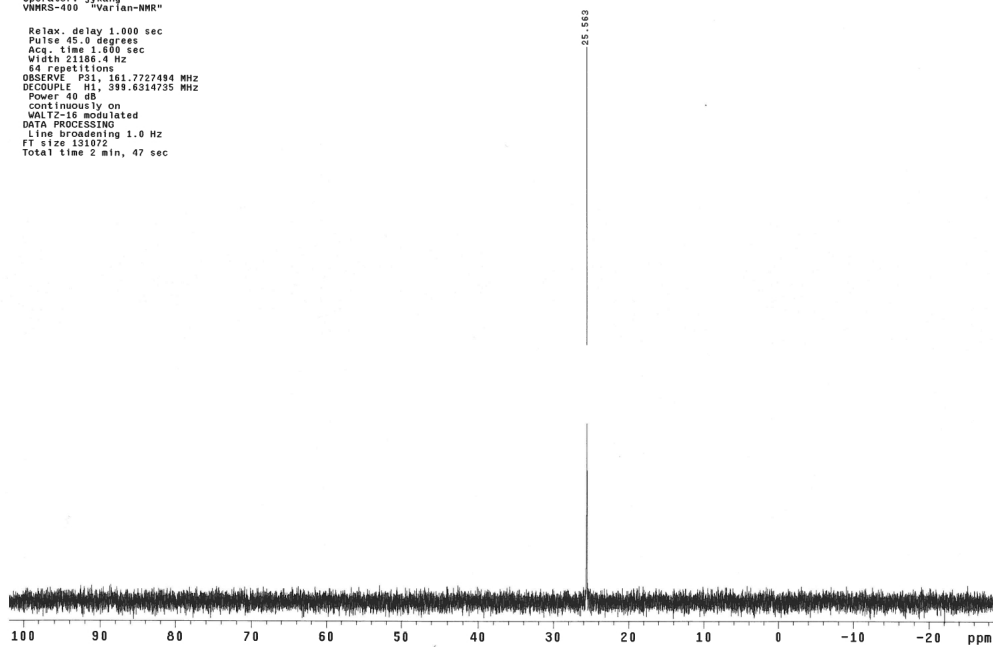
Sample: ja-8-58-p
File: exp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: Jykeng
VNMR5-900 "Varian-NMR"
Relax: delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 2186.4 Hz
25 repetitions
OBSERVE F21: 161.7726385 MHz
DECOUPLE H1: 399.6314735 MHz
Power 40 db
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

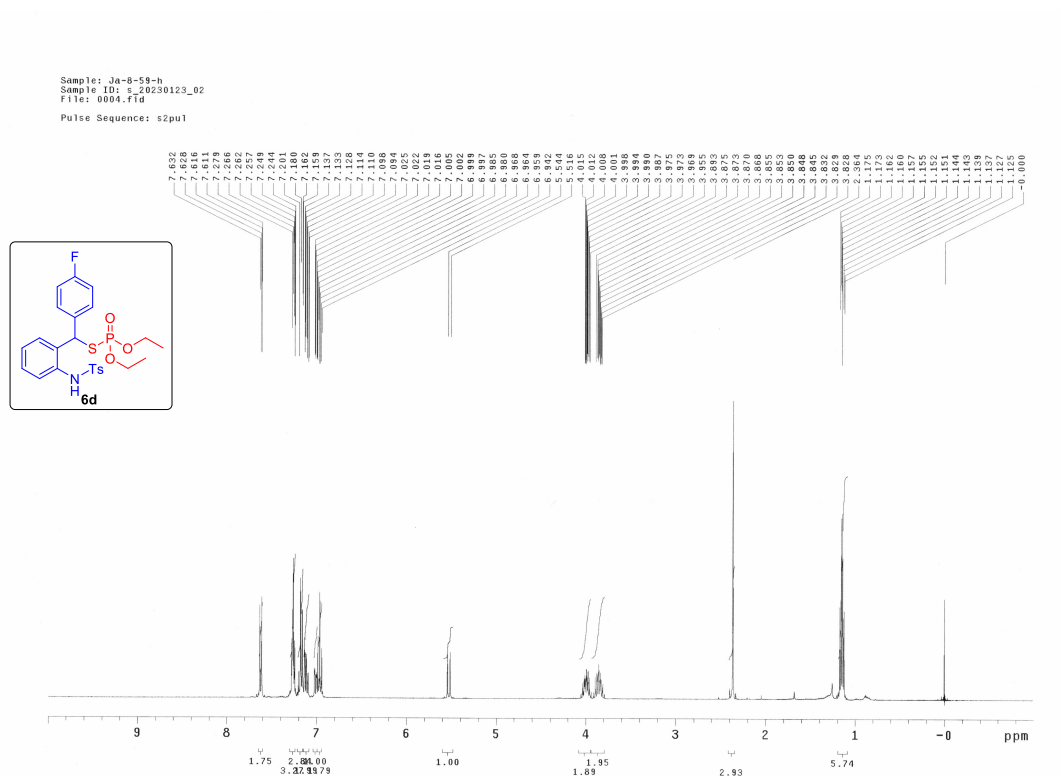
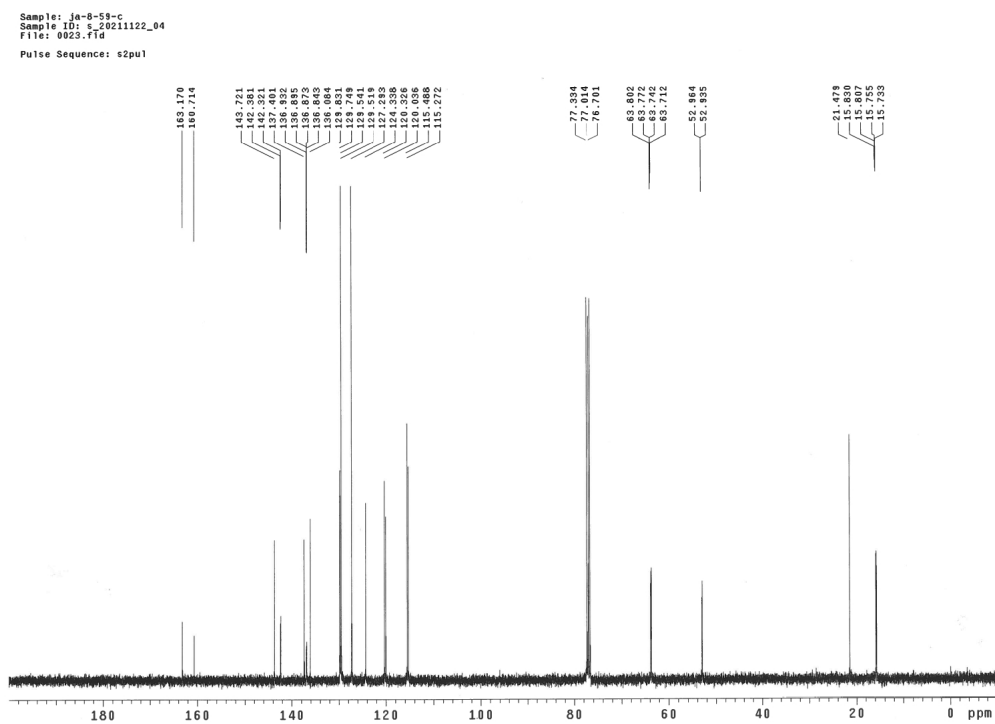


¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

Sample: Ja-8-68-p
File: exp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: JyKeng
VNMR5-400 "Varian-NMR"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.800 sec
Width 21186.4 Hz
64 repetitions
OBSERVE P31, 161.7727484 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 151072
Total time 2 min, 47 sec

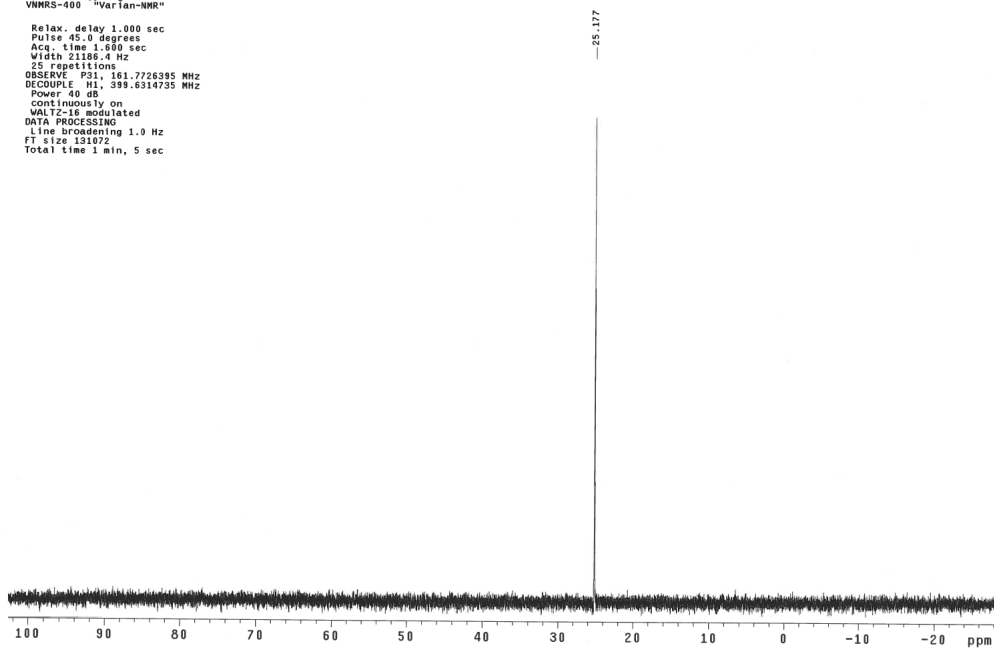


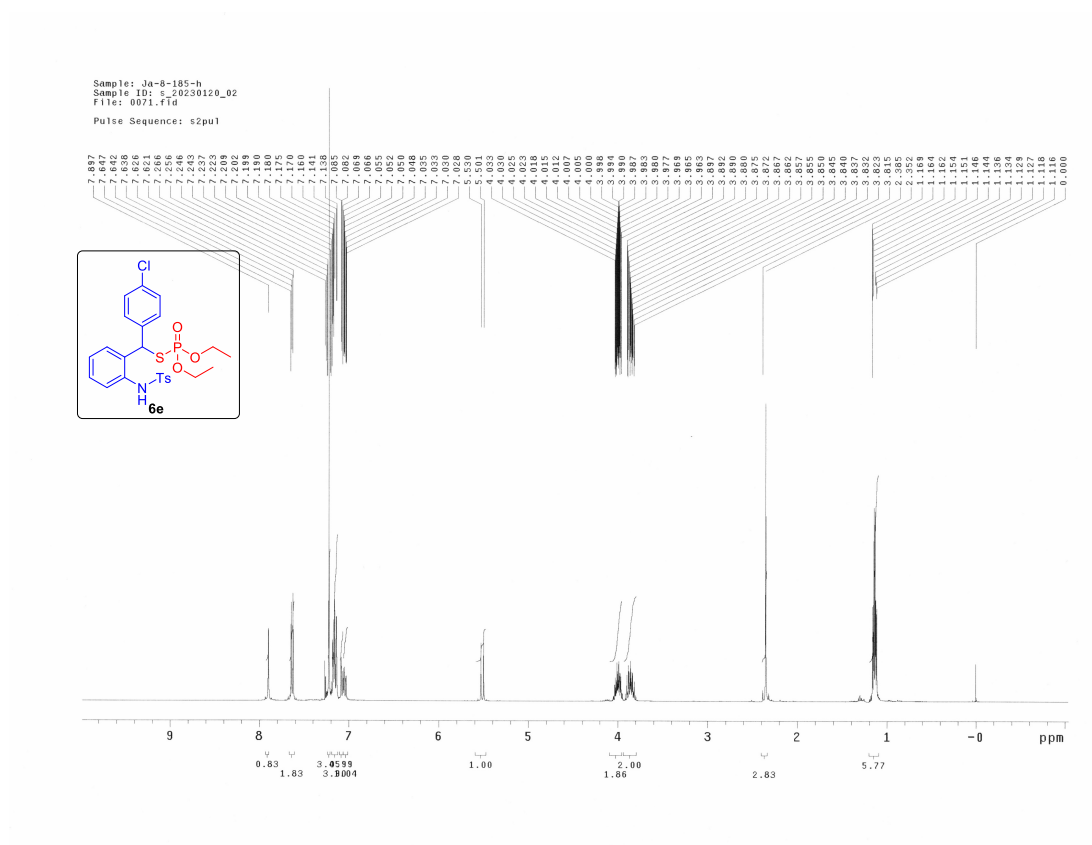
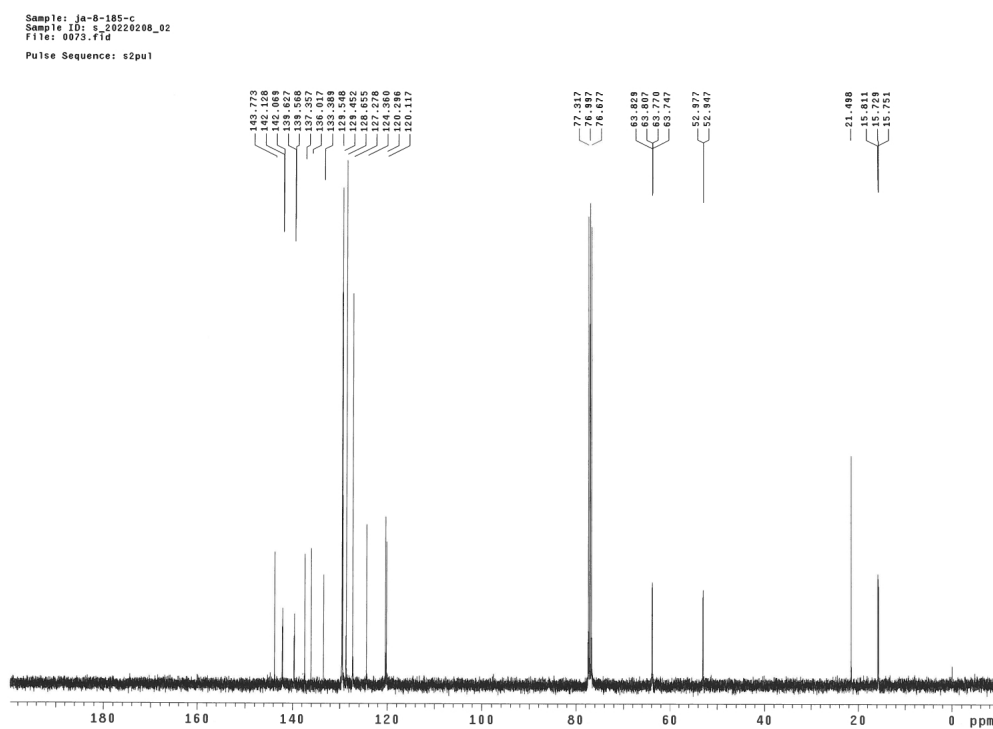
¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

Sample: ja-6-59-p
File: exp
Pulse Sequence: s2pul
Solvent: cdc13
Temp: 25.0 C / 298.1 K
Operator: lykeng
VNMR5-400 "Varian-NMR"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21186.4 Hz
25 repetitions
OBSERVE F31, 161.7726395 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

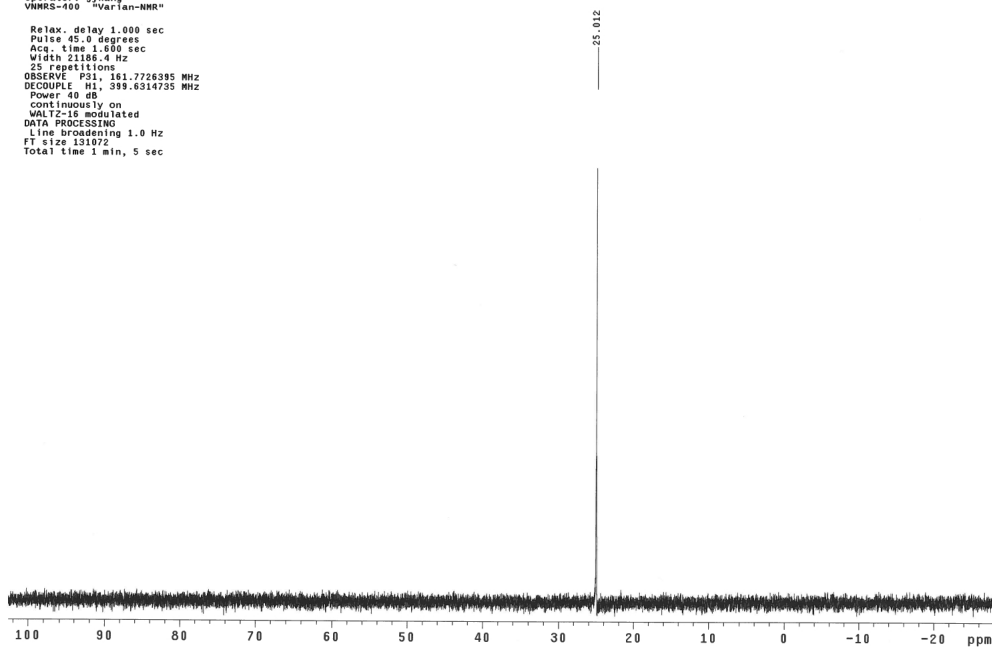


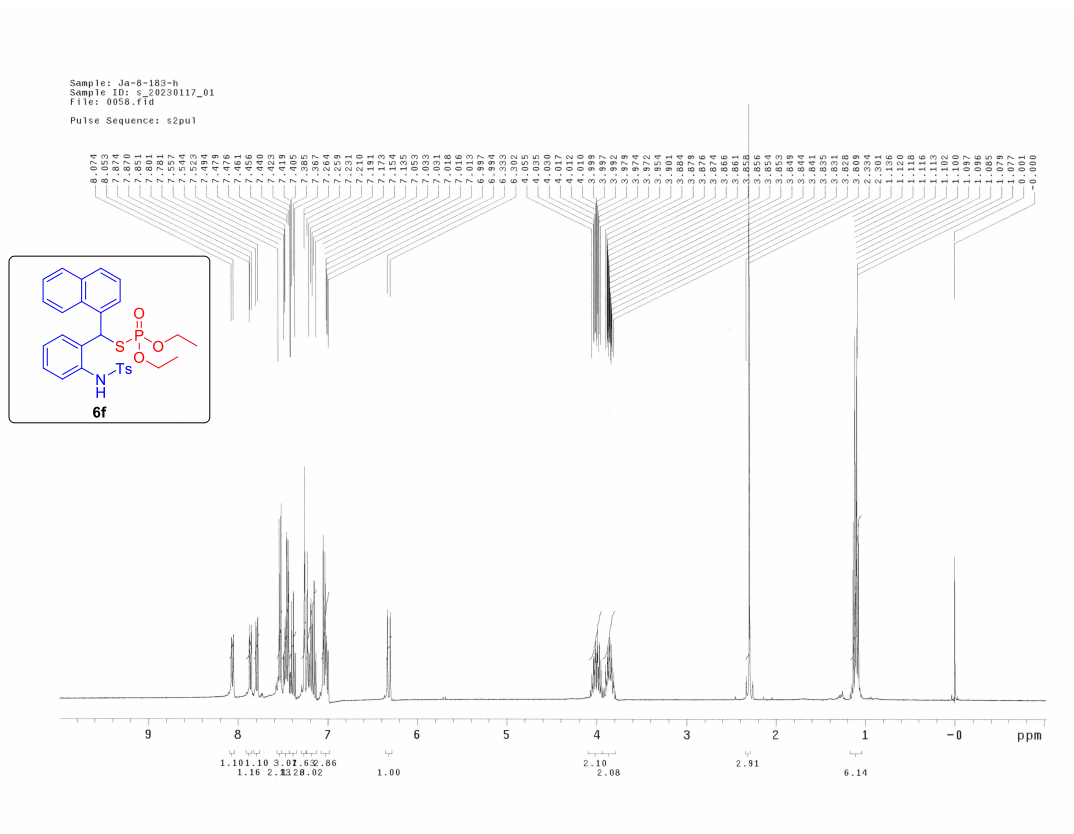
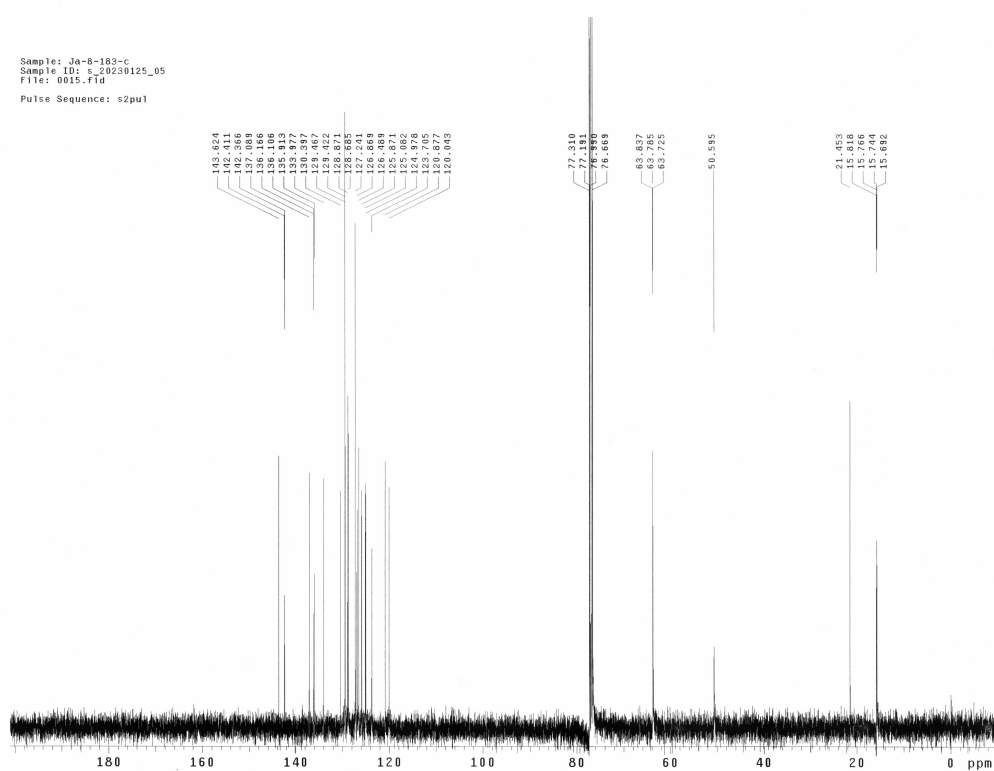
¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

Sample: ja-8-185-p
File: exp
Pulse Sequence: s2pul
Solvent: cdc13
Temp: 25.0 C / 288.1 K
Operator: JyKeng
VNMR5-900 "Varian-NMR"

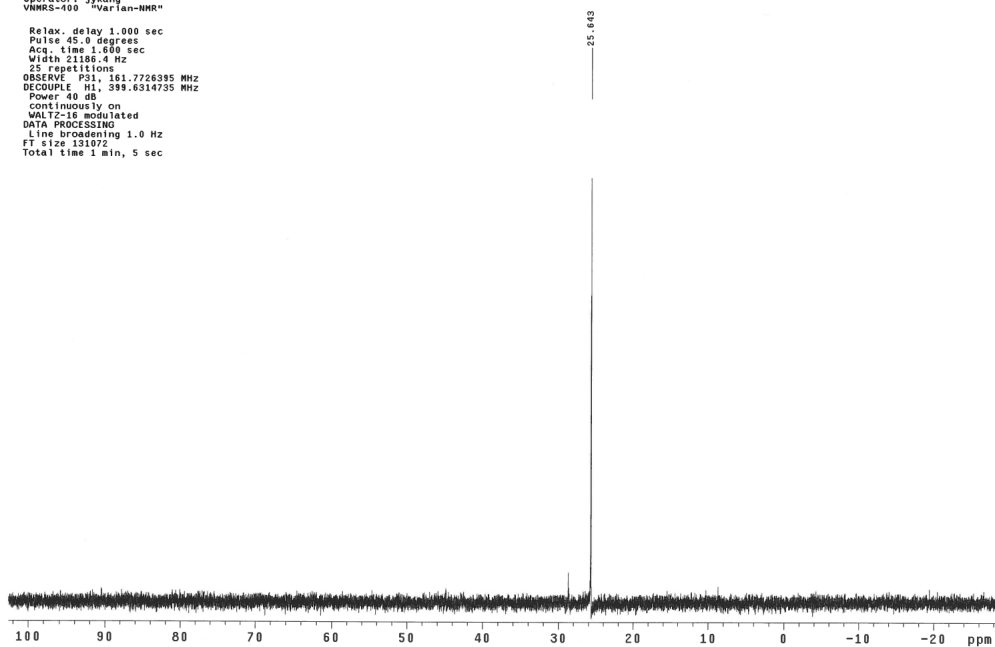
Relax: delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21186.4 Hz
25 repetitions
OBSERVE P21, 161.7726385 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 db
CONTINUOUSLY on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

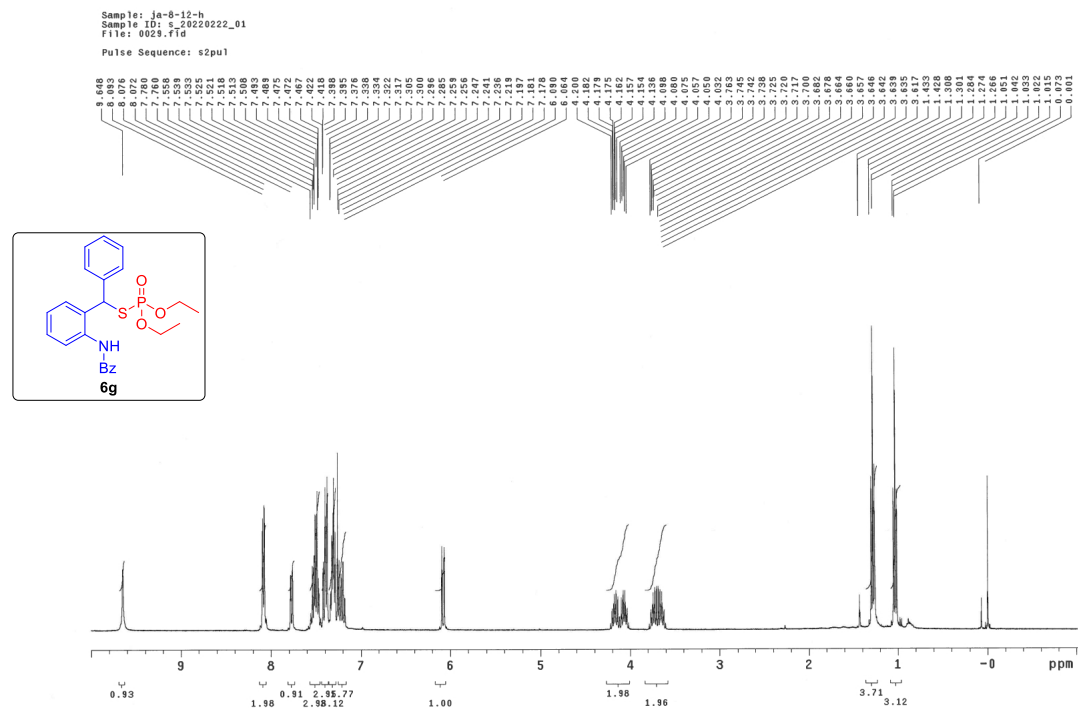
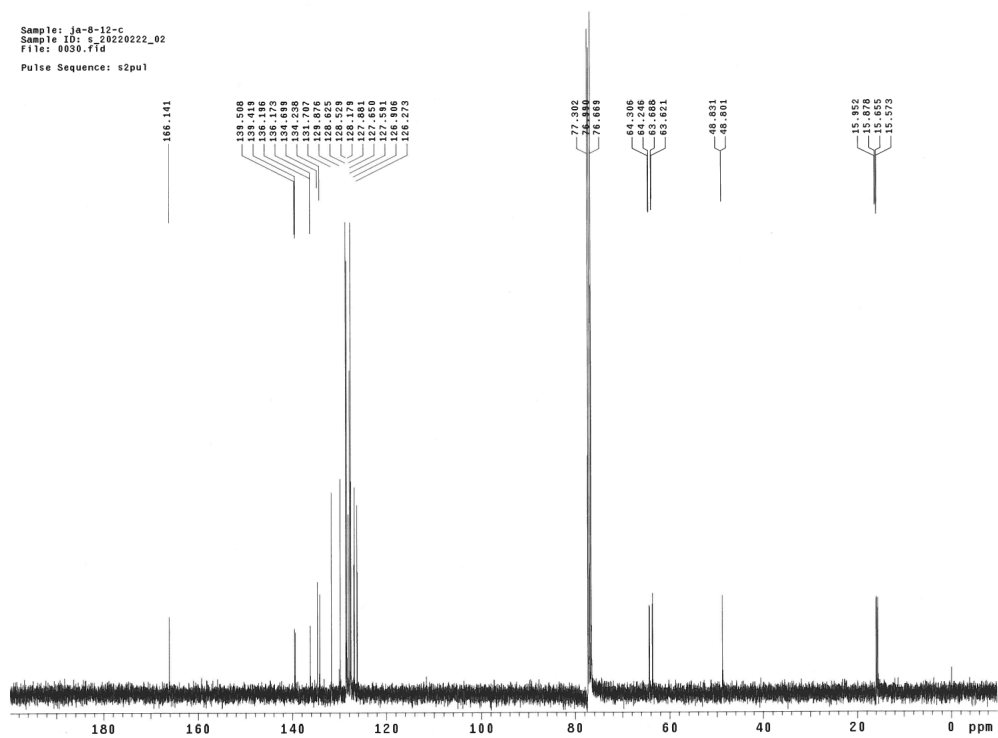


¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

Sample: ja-8-183-p
File: exp
Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: Jywang
VNMRS-600 Varian-NMR
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.600 sec
Width 21186.4 Hz
25 repetitions
OBSERVE P31, 161.7726385 MHz
DECOUPLE H1, 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec

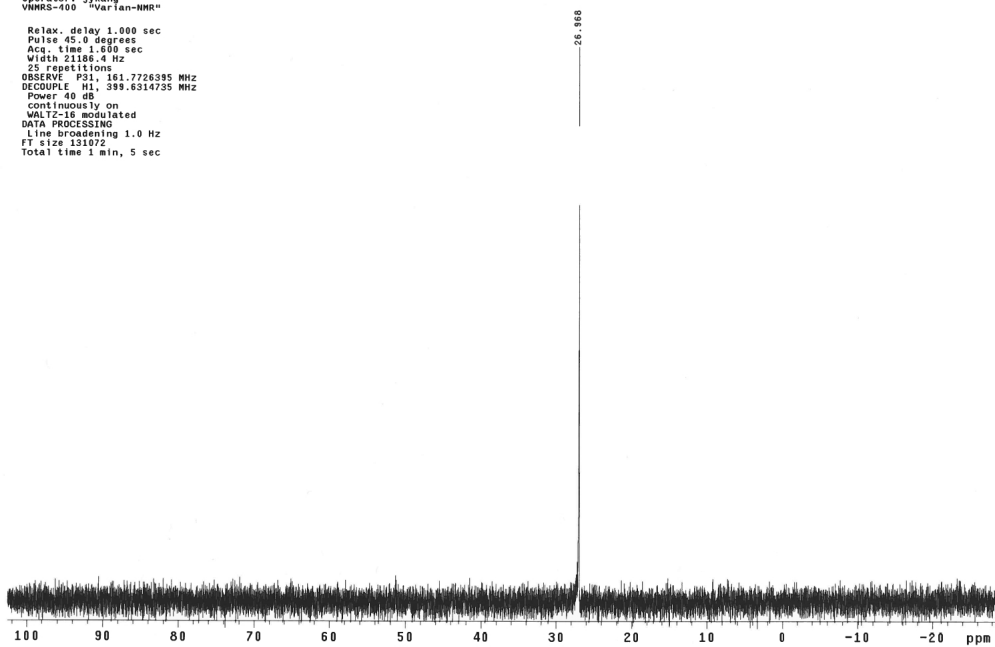


¹H NMR (400 MHz) in CDCl₃**¹³C NMR (100.5 MHz) in CDCl₃**

^{31}P NMR (162 MHz) in CDCl_3

Sample: ja-8-12-p
File: exp
Pulse Sequence: s2pu1
Solvent: cdcl3
Temp: 25.0 C / 298.1 K
Operator: lykang
VNMR5-600 "Varian-NMR"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.800 sec
Width 21186.4 Hz
25 Repetitions
OBSERVE F31: 161.7726395 MHz
DECOUPLE H1: 399.6314735 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 5 sec



References

1. H. Huang and J. Y. Kang, *Org. Lett.*, 2017, **19**, 5988-5991.
2. H. Fakhraian and A. Mirzaei, *Org. Process Res. Dev.*, 2004, **8**, 401-404.
3. Y. Guo, Y. Luo, S. Mu, J. Xu and Q. Song, *Org. Lett.*, 2021, **23**, 6729-6734.
4. F. Yang, X. Zhou, Y. Wei, L. Wang and J. Jiang, *Org. Chem. Front.* , 2021, **8**, 5064-5070.