Supporting Information

Oxidative *ortho*-Alkenylation of Arylphosphine Oxides by Rhodium-Catalyzed Twofold C-H Bond Cleavages[‡,†]

Juntae Mo,[†] Sujin Lim,[†] Sangjune Park, Taekyu Ryu, Sanghyuck Kim and Phil Ho Lee*

Department of Chemistry, Kangwon National University, Chuncheon 200-701, Republic of Korea

FAX: (+)-82-33-253-7582. *e*-mail: phlee@kangwon.ac.kr

Contents

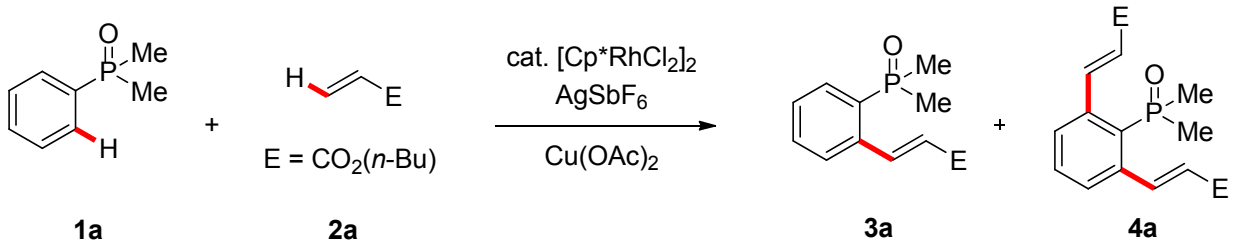
Experimental Section

General: Reactions were carried out in oven-dried glassware under a nitrogen atmosphere. $[Cp^*RhCl_2]_2$ were purchased from Anfenchem Co. $AgSbF_6$ and Ag_2CO_3 were purchased from Alfa Chemical Co. $Cu(OAc)_2$ was purchased from Aldrich Chemical Co. Other commercial available reagents were used without purification and all solvents were reaction grade.

All reaction mixtures were stirred magnetically and were monitored by thin-layer chromatography using Merck silica gel 60 F_{254} precoated glass plates, which were visualized with UV light and then, developed using either iodine or a solution of anisaldehyde. Flash column chromatography was carried out using Merck silica gel 60 (0.040-0.063 mm, 230-400 mesh). ^1H NMR (400 MHz), ^{13}C NMR (100 MHz), and ^{31}P NMR (161 MHz) spectra were recorded on a Brucker DPXFT spectrometer. Deuterated chloroform was used as the solvent, and chemical shift values (δ) are reported in parts per million relative to the residual signals of this solvent (δ 7.26 for ^1H and δ 77.0 for ^{13}C). Infrared spectra were recorded on FT-IR spectrometer as either a thin film pressed between two sodium chloride plates or as a solid suspended in a potassium bromide disk. Mass spectra were obtained from the KBSI on high resolution mass spectrometer. Melting points were determined in open capillary tube using Electrothermal 9100 apparatus.

Additional data for reaction optimization

Table 1. Solvent screening of oxidative alkenylation of phenyldimethylphosphine oxide with *n*-butyl acrylate[a]

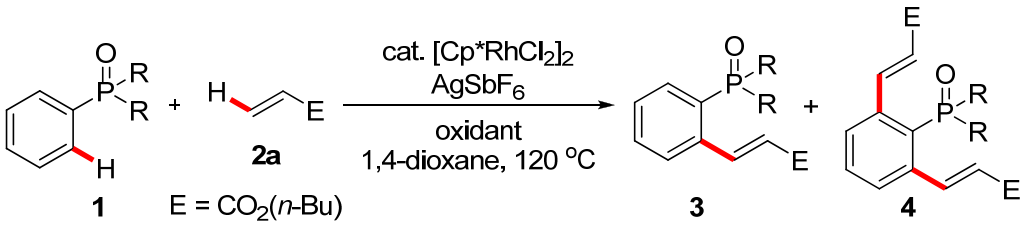


1a **2a** **3a** **4a**

entry	solvent	yield (%)[b]	entry	solvent	yield (%)[b]
1	CH_3CN	0	8	toluene	0
2	CH_3NO_2	0	9	xylene	4
3	MeOH	0	10	C_6F_6	2
4	*t*-BuOH	35 (6:1)[c]	11	THF	12
5	*t*-AmOH	12 (4:1)[c]	12	DMF	0
6	DCE	30 (4.5:1)[c]	13	DMSO	0
7	dioxane	50 (3:1)[c]	14[d]	dioxane	0

[a] Reaction conditions: $[Cp^*RhCl_2]_2$ (2 mol %), $AgSbF_6$ (8 mol %), **1a** (0.2 mmol), **2a** (0.4 mmol), $Cu(OAc)_2$ (2.0 equiv), solvent (0.8 mL) at 80 °C for 20 h. [b] Yields based on ^1H NMR integration relative to dibromethane internal standard. [c] The ratio of **3a** and **4a**. [d] $[Cp^*Rh(MeCN)_3](SbF_6)_2$ (2 mol %) was used.

Table 2. Reacivity study of dialkylphosphin oxide[a]

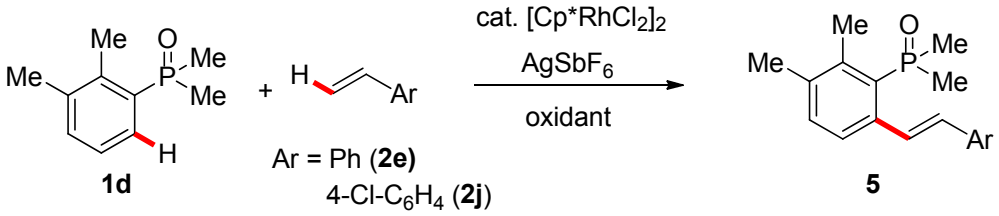


1 E = CO_2(*n*-Bu) **3** **4**

entry	R		time (h)		yield (%)[b]
1	Me	**1a**	8	**3a**	59 (22)[c]
2	*i*-Pr	**1j**	24	**3p**	45 (26)[d]
3	Ph		24		0

[a] Reaction condition: **1** (0.2 mmol), **2a** (0.4 mmol), $[Cp^*RhCl_2]Cl_2$ (2 mol %), $AgSbF_6$ (8 mol %), $Cu(OAc)_2$ (1.0 equiv), Ag_2CO_3 (1.0 equiv) at 120 °C for 24 h. [b] Isolated yield. [c] Number in parenthesis is yield of **4a**. [d] Recovery yield of starting material.

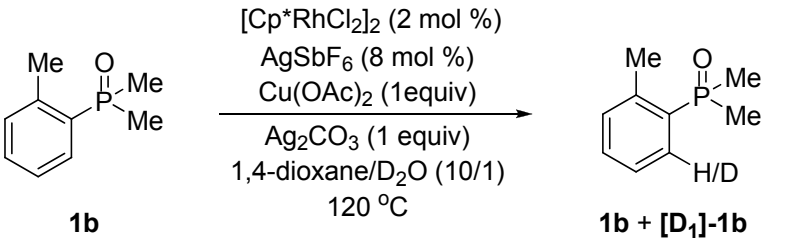
Table 3. Optimization of oxidative *ortho*-alkenylation of **1d** with styrene derivatives[a]



entry	**2** (equiv)	oxidant (equiv)	solvent	temp (^{o}C)	yield (%)[b]
1	**2e** (3)	$Cu(OAc)_2$ (1)/Ag_2CO_3 (1)	1,4-dioxane	120	60
2[c]	**2e** (3)	$Cu(OAc)_2$ (1)/Ag_2CO_3 (1)	1,4-dioxane	120	54
3	**2e** (3)	$Cu(OAc)_2$ (2)/Ag_2CO_3 (2)	1,4-dioxane	120	56
4[d]	**2e** (3)	$Cu(OAc)_2$ (1)/Ag_2CO_3 (1)	1,4-dioxane	120	57
5[e]	**2e** (3)	$Cu(OAc)_2$ (1)/Ag_2CO_3 (1)	1,4-dioxane	120	20
6	**2e** (6)	$Cu(OAc)_2$ (1)/Ag_2CO_3 (1)	1,4-dioxane	120	60
7	**2e** (3)	$Cu(OAc)_2$ (1)	1,4-dioxane	120	0
8	**2e** (3)	Ag_2CO_3 (1)	1,4-dioxane	120	60
9	**2e** (3)	Ag_2CO_3 (1)	THF	90	62
10	**2j** (3)	$Cu(OAc)_2$ (1)/Ag_2CO_3 (1)	1,4-dioxane	120	32
11	**2j** (1)	Ag_2CO_3 (1)	Ethyl acetate	120	38
12	**2j** (3)	Ag_2CO_3 (1)	*t*-BuOH	120	18
13	**2j** (3)	Ag_2CO_3 (1)	THF	120	54
14	**2j** (3)	Ag_2CO_3 (1)/AgOAc (1)	THF	120	14
15	**2j** (3)	$Cu(OAc)_2$ (1)/NaOAc (1)	THF	80	9
16	**2j** (3)	$Cu(OAc)_2$ (1)/Ag_2CO_3 (1)	THF	90	38

[a] Reaction condition: $[Cp^*RhCl_2]_2$ (2 mol %), $AgSbF_6$ (8 mol %), **1d** (0.2 mmol) in solvent (0.8 mL) for 24 h. [b] Isolated yield of product. [c] $AgSbF_6$ (16 mol %) was used. [d] 1,4-Dioxane (0.4 mL, 0.5 M). [e] Neat reaction

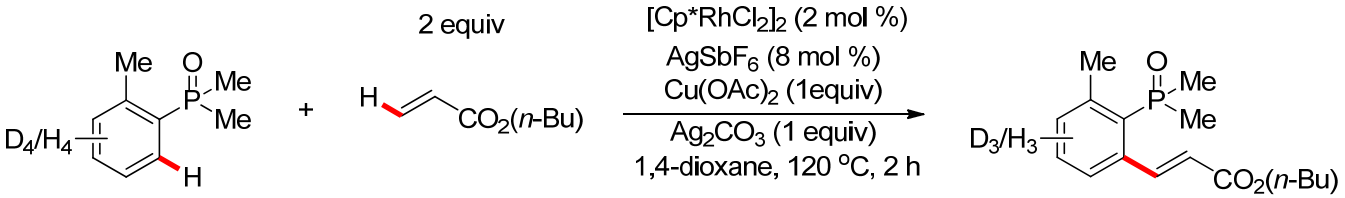
Studies with isotopically labeled compounds



1b **1b + [D$_1$]-1b**

1 h H 100% / D 0%
12 h H 100% / D 3.2%

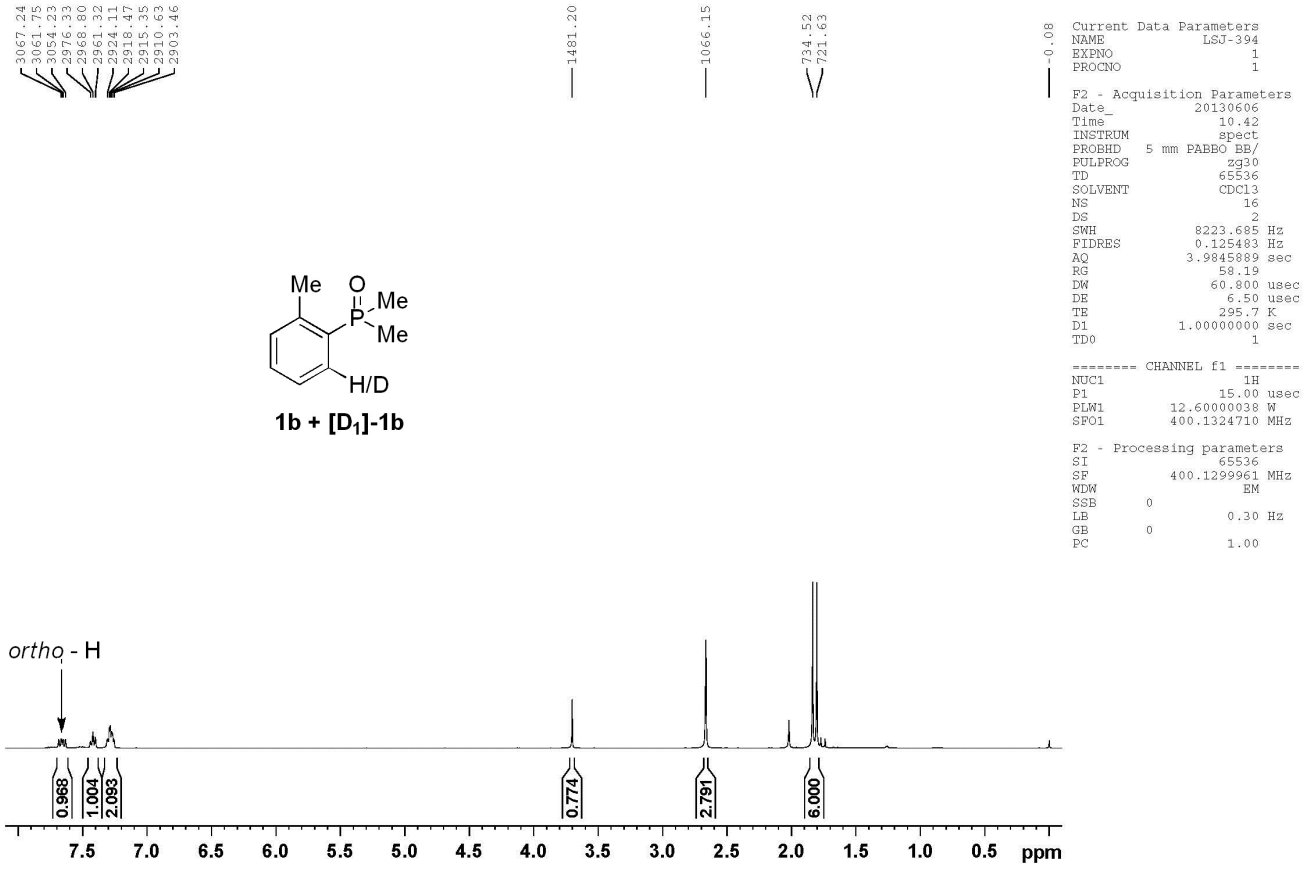
To an oven dried test tube were added $[Cp^*RhCl_2]_2$ (2.9 mg, 2 mol %), $AgSbF_6$ (5.5 mg, 8 mol %), $Cu(OAc)_2$ (36.3 mg, 0.2 mmol), Ag_2CO_3 (55.5 mg, 0.2 mmol) and dimethyl(*o*-tolyl)phosphine oxide (**1b**, 0.1 mmol) in 1,4-dioxane/D_2O (0.7 mL/0.07 mL). The resulting mixture was stirred under nitrogen at 120 °C (bath temperature) for 12 h. After celite filtration and evaporation of the solvent *in vacuo*, product was purified by column chromatography (methanol:dichloromethane = 1:20) on silica gel.



1b + [D$_4$]-1b **2a**

3c + [D$_3$]-3c
27%, k_H/k_D = 4.4

To an oven dried test tube were added $[Cp^*RhCl_2]_2$ (2.9 mg, 2 mol %), $AgSbF_6$ (5.5 mg, 8 mol %), $Cu(OAc)_2$ (36.3 mg, 0.2 mmol), Ag_2CO_3 (55.5 mg, 0.2 mmol), dimethyl(*o*-tolyl)phosphine oxide (**1b**, 0.1 mmol), dimethyl(*tetra*-deuterio-*o*-tolyl)phosphine oxide (**[D$_4$]-1b**, 0.1 mmol) and *n*-butyl acrylate (0.4 mmol) in 1,4-dioxane (0.8 mL). The resulting mixture was stirred under nitrogen at 120 °C (bath temperature) for 2 h. After celite filtration and evaporation of the solvent *in vacuo*, [**3c** + **[D$_3$]-3c]** (27%) was purified by column chromatography (methanol:dichloromethane = 1:20) on silica gel.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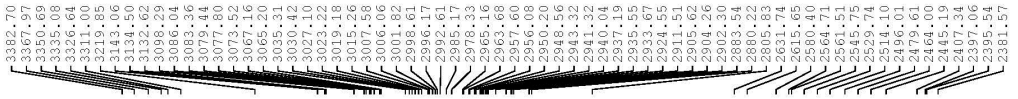

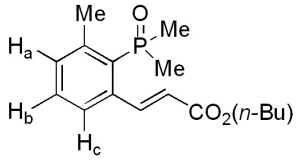

3c

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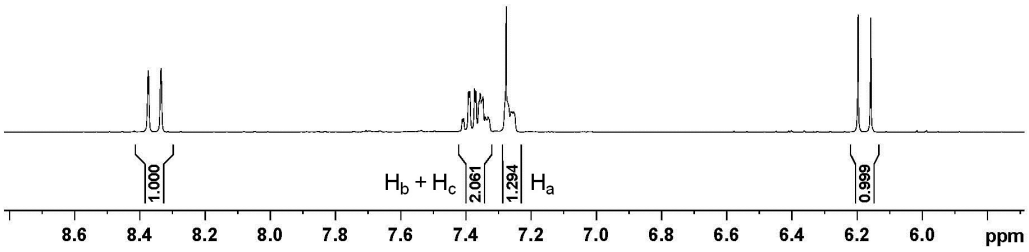

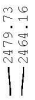

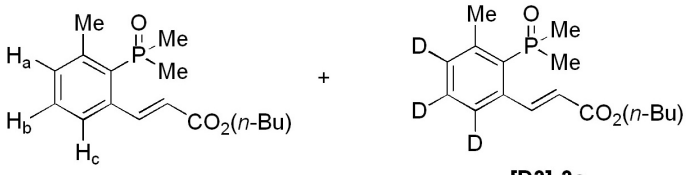

3c **[D3]-3c**

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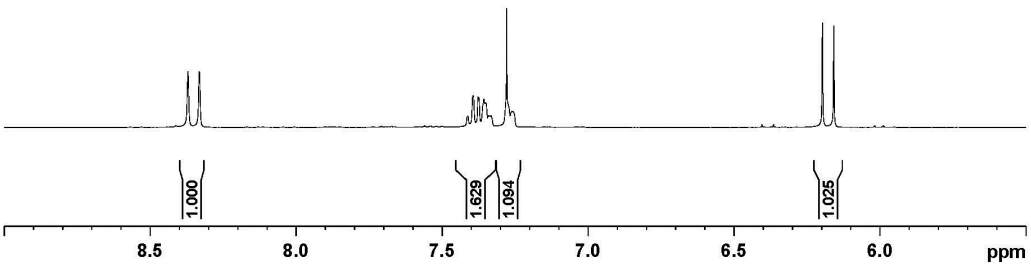
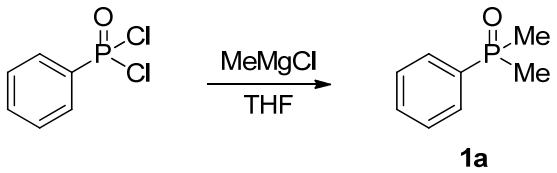
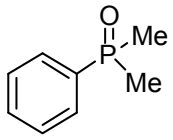


Preparation of arylphosphine oxide 1a.[1, 2]

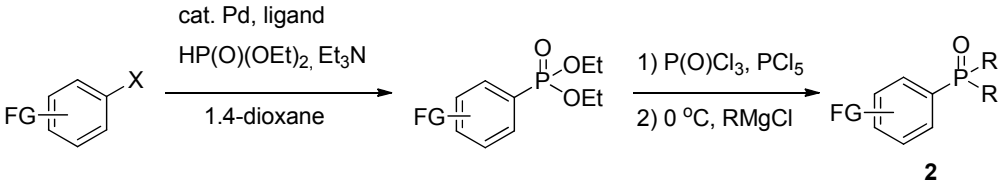


1a

To a solution of phenylphosphonic dichloride (0.7 mL, 5.0 mmols) in dry THF (25 mL) was added methylmagnesium chloride (3.0 M solution in THF, 4.17 mL, 12.5 mmol) under nitrogen atmosphere at 0 ^{o}C, The mixture was stirred at 0 ^{o}C for 1 h. When the reaction was complete, the solution was quenched with ammonium chloride at 0 ^{o}C . The aqueous layer was separated and extracted with CH_2Cl_2 (10 mL x 3) and the organic fraction was dried with $MgSO_4$, filtered, and concentrated by rotary evaporation. The resulting product was then purified by flash column chromatography (methanol : dichloromethane = 1 : 20) on silica gel.



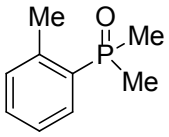
Dimethylphenyl phosphine oxide (1a)[1] : White solid. Melting point = 116-118 ^{o}C; R_f = 0.3 (methanol : dichloromethane = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77-7.72 (m, 2H), 7.53-7.50 (m, 3H), 1.74 (d, J = 12.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.8 (d, J_{CP} = 98.0 Hz), 131.6, 129.5(2C), 128.6(2C), 18.1 (d, J_{CP} = 70.0 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 33.80; IR (film) : 3056, 2984, 2913, 1659, 1437, 1294, 1176, 1118, 935, 865 cm^{-1}. HRMS (EI) calcd For $C_8H_{11}OP$: 154.0548; found : 154.0548.



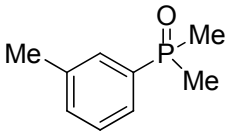
2

To a solution of tris(dibenzylideneacetone)dipalladium(0) (18.3 mg, 2 mol %), 4,5-*bis*(di-phenylphosphino)-9,9-dimethylxanthene (23.1 mg, 4 mol %) in dry 1,4-dioxane (10.0 mL) was added aryl halide (2.0 mmol), dimethyl phosphite (0.4 mL, 3.0 mmol), Et_3N (0.4 mL, 3.0 mmol) under nitrogen atmosphere. When the reaction was complete, the solution was quenched with water. The aqueous layer

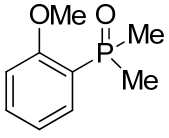
was separated and extracted with CH_2Cl_2 (10 mL x 3) and the organic fraction was dried with $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography. The corresponding diethyl phenylphosphonate (2.0 mmol), phosphoryl chloride (2.1 mmol), and phosphorous pentachloride (2.3 mmol) were reflux 4 h. The reaction solution was celite filtered and extracted with Et_2O (10 mL x 3) and concentrated under reduced pressure. The residue was diluted with THF (0.2 M) and dropwise arylmagnesium chloride (3.0 mmol) under nitrogen atmosphere at 0 ^oC The mixture was stirred at 0 ^oC for 1 h. When the reaction was complete, the solution was quenched with ammonium chloride at 0 ^oC . The aqueous layer was separated and extracted with CH_2Cl_2 (10 mL x 3) and the organic fraction was dried with $MgSO_4$, filtered, and concentrated by rotary evaporation. The resulting product was then purified by flash column chromatography (methanol : dichloromethane = 1 : 20) on silica gel.



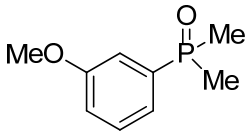
Dimethyl(*o*-tolyl)phosphine oxide (1b)[3] : White solid. Melting point = 79-81 ^oC; R_f = 0.3 (methanol : dichloromethane = 1 : 20); ^1H NMR (400 MHz, $CDCl_3$) δ 7.68-7.64 (m, 1H), 7.44-7.39 (m, 1H), 7.31-7.26 (m, 2H), 2.67 (s, 3H), 1.80 (d, J = 12.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.7, 132.6 (d, J_{CP} = 92.6 Hz), 131.9, 131.7(2C), 130.7, 21.4, 18.2 (d, J_{CP} = 71.3 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 35.09; IR (pellet) : 3056, 917, 1647, 1592, 1421, 1294, 1161, 1132, 932 cm^{-1}. HRMS (EI) calcd. For $C_9H_{13}OP$: 168.0704; found : 168.0704.



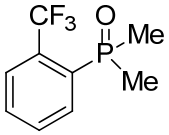
Dimethyl(*m*-tolyl)phosphine oxide (1c)[3] : White solid. Melting Point = 44-46 ^oC; R_f= 0.3 (methanol : dichloromethane = 1 : 20); ^1H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, J = 12.2 Hz, 1H), 7.49 (dd, J = 11.6 Hz, 7.2 Hz, 1H), 7.40-7.33 (m, 2H), 2.42 (s, 3H), 1.73 (d, J = 13.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.6, 134.6 (d, J_{CP} = 98.4 Hz), 132.4, 130.2, 128.6, 126.5, 21.4, 18.1 (d, J_{CP} = 71.4 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 33.90; IR (pellet) : 2983, 2917, 1648, 1420 ,1294, 1159, 938, 867, 785 cm^{-1}. HRMS (EI) calcd. For $C_9H_{13}OP$: 168.0704; found : 168.0706.



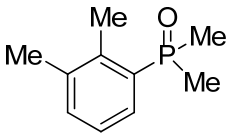
(2-Methoxyphenyl)dimethylphosphine oxide (1d)[3] : White solid. Melting point = 64-66 °C; R_f = 0.3 (methanol : dichloromethane = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.94 (m, 1H), 7.52 (t, J = 7.7 Hz, 1H), 7.13-7.09 (m, 1H), 6.95-6.92 (m, 1H), 3.89 (s, 3H), 1.75 (d, J = 13.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 133.8 (d, J_{CP} = 2.7 Hz), 133.7, 121.9 (d, J_{CP} = 95.0 Hz), 121.1 (d, J_{CP} = 10.8 Hz), 110.4 (d, J_{CP} = 3.0 Hz), 55.3 (d, J_{CP} = 2.9 Hz), 17.6 (d, J_{CP} = 72.2 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 32.59; IR (pellet) : 3069, 2980, 2943, 2840, 1650, 1590, 1479, 1274, 1243, 1162, 1079, 1018 cm^{-1}. HRMS (EI) calcd. For $C_9H_{13}O_2P$: 184.0653; found : 184.0650.



(3-Methoxyphenyl)dimethylphosphine oxide (1e)[3] : Colorless oil. R_f = 0.3 (methanol : dichloromethane = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.37 (m, 1H), 7.32 (d, J = 13.0 Hz, 1H), 7.25-7.19 (m, 1H), 7.06-7.04 (m, 1H), 3.85 (s, 3H), 1.73 (d, J = 13.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 136.2 (d, J_{CP} = 97.6 Hz), 130.0, 121.4, 117.7, 114.7, 55.5, 18.1 (d, J_{CP} = 71.4 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 34.13; IR (film) : 3064, 2979, 2913, 2837, 1652, 1591, 1576, 1485, 1416, 1288, 1242, 1167 cm^{-1}. HRMS (EI) calcd. For $C_9H_{13}O_2P$: 184.0653; found : 184.0650.

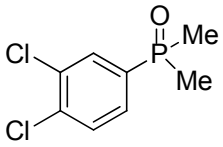


Dimethyl(2-(trifluoromethyl)phenyl)phosphine oxide (1f)[3] : Colorless oil. R_f = 0.3 (methanol : dichloromethane = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (dd, J = 13.1 Hz, 7.7 Hz, 1H), 7.81-7.79 (m, 1H), 7.76-7.72 (m, 1H), 7.69-7.65 (m, 1H), 1.86 (d, J = 13.5 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.0 (d, J_{CP} = 5.9 Hz), 133.2 (d, J_{CP} = 85.6 Hz), 132.1 (d, J_{CP} = 10.1 Hz), 131.8 (d, J_{CP} = 2.5 Hz), 130.5-130.2 (m, 1C), 127.1-126.9 (m, 1C), 124.0 (d, J_{CF} = 270 Hz), 18.8 (d, J_{CP} = 70 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 34.26; IR (film) : 3074, 2922, 1654, 1437, 1312, 1262, 1171, 1121, 1037, 935 cm^{-1}. HRMS (EI) calcd. For $C_9H_{10}F_3OP$: 222.0421; found : 222.0423.

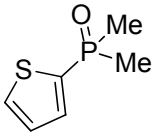


(2,3-Dimethylphenyl)dimethylphosphine oxide (1g) : White solid. Melting point = 108-110 °C; R_f = 0.3 (methanol : dichloromethane = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (dd, J = 13.0 Hz, 7.7 Hz, 1H), 7.31 (d, J = 7.5 Hz, 1H), 7.21-7.17 (m, 1H), 2.60 (s, 3H), 2.60 (s, 3H), 1.81 (d, J = 12.7 Hz, 6H); ^{13}C

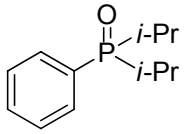
NMR (100 MHz, $CDCl_3$) δ 131.6, 131.5, 131.3, 129.5 (d, J_{CP} = 85.1 Hz), 128.3, 128.2, 25.2 (d, J_{CP} = 67.1 Hz, 2C), 16.0 (d, J_{CP} = 2.0 Hz), 14.9 (d, J_{CP} = 3.1 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ 36.00; IR (pellet) : 2978, 2916, 1647, 1448, 1421, 1294, 1187, 1167, 1145, 934 cm^{-1}. HRMS (EI) calcd. For $C_{10}H_{15}OP$: 182.0861; found : 182.0860.



(3,4-Dichlorophenyl)dimethylphosphine oxide (1h) : White solid. Melting point = 80-82 ^{o}C; R_f = 0.3 (methanol : dichloromethane = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (dd, J = 11.4 Hz, 1.7 Hz, 1H), 7.59-7.55 (m, 2H), 1.75 (d, J = 13.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.5 (d, J_{CP} = 3.0 Hz), 135.1 (d, J_{CP} = 96.1), 133.6 (d, J_{CP} = 15.5 Hz), 131.8 (d, J_{CP} = 11.1 Hz), 131.0 (d, J_{CP} = 12.7 Hz), 128.8 (d, J_{CP} = 9.6), 18.1 (d, J_{CP} = 72 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 32.66; IR (pellet) : 2984, 2913, 1655, 1465, 1364, 1295, 1174, 1032, 937 cm^{-1}. HRMS (EI) calcd. For $C_8H_9Cl_2OP$: 221.9768; found : 221.9765.



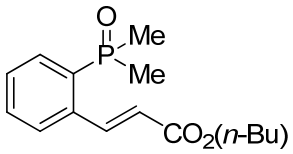
Dimethyl(thiophen-2-yl)phosphine oxide (1i) : White solid. Melting point = 100-102 ^{o}C; R_f = 0.3 (methanol : dichloromethane = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.68 (m, 1H), 7.59-7.56 (m, 1H), 7.22-7.19 (m, 1H), 1.81 (d, J = 13.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.6 (d, J_{CP} = 104.8 Hz), 133.9 (d, J_{CP} = 9.5 Hz), 132.3 (d, J_{CP} = 4.6 Hz), 128.4 (d, J_{CP} = 13.2 Hz), 19.7 (d, J_{CP} = 75.2 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 29.18; IR (pellet) : 3050, 2979, 2912, 1653, 1407, 1295, 1226, 1164, 1093 cm^{-1}. HRMS (EI) calcd. For C_6H_9OPS : 160.0112; found : 160.0112.



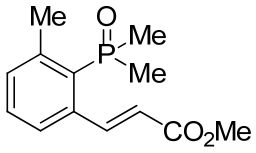
Diisopropyl(phenyl)phosphine oxide (1j) : White solid. Melting point = 46-49 ^{o}C; R_f = 0.3 (methanol : dichloromethane = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72-7.67 (m, 2H), 7.55-7.46 (m, 3H), 2.38-2.25 (m, 2H), 1.20 (dd, J = 14.8 Hz, 7.1 Hz, 6H), 1.06 (dd, J = 15.8 Hz, 7.2 Hz, 6H) ; ^{13}C NMR (100 MHz, $CDCl_3$) δ 131.6, 131.5, 131.4, 129.5 (d, J_{CP} = 84.3 Hz), 128., 128.2, 25.5, 24.9, 16.0(2C), 14.9(2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 50.74; IR (pellet) : 3056, 2966, 2935, 2874, 1640, 1466, 1173, 1150, 1110 cm^{-1}. HRMS (EI) calcd. For C12H19OP : 210.1174; found : 210.1174.

Rh-catalyzed oxidative *ortho*-alkenylation of arylphosphine oxide with arylates.

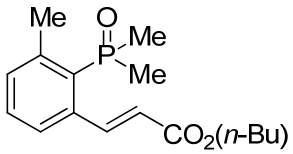
To an oven dried test tube were added $[Cp^*RhCl_2]_2$ (2.9 mg, 2 mol %), $AgSbF_6$ (5.5 mg, 8 mol %), $Cu(OAc)_2$ (36.3 mg, 0.2 mmol), Ag_2CO_3 (55.5 mg, 0.2 mmol), aryl phosphine oxide (0.2 mmol) and acrylate (0.4 mmol) in 1,4-dioxane (0.8 mL). The resulting mixture was stirred under nitrogen at 120 ^oC (bath temperature) for 12 h. After celite filtration and evaporation of the solvent *in vacuo*, product was purified by column chromatography on silica gel.



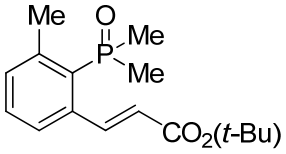
(*E*)-Butyl 3-(2-(dimethylphosphoryl)phenyl)acrylate (3a) : Yellow oil. R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^1H NMR (400 MHz, $CDCl_3$) δ 8.39 (d, J = 15.7 Hz, 1H), 7.91-7.86 (m, 1H), 7.69-7.67 (m, 1H), 7.57-7.48 (m, 2H), 6.38 (d, J = 15.7 Hz, 1H), 4.23 (t, J = 6.7 Hz, 2H), 1.83 (d, J = 12.9 Hz, 6H), 1.74-1.67 (m, 2H), 1.49-1.40 (m, 2H), 0.97 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.3, 142.1 (d, J_{CP} = 5.1 Hz), 137.2 (d, J_{CP} = 8.0 Hz), 133.8 (d, J_{CP} = 92.8 Hz), 132.1 (d, J_{CP} = 2.6 Hz), 131.6 (d, J_{CP} = 9.0 Hz), 129.5 (d, J_{CP} = 11.2 Hz), 127.7 (d, J_{CP} = 9.5 Hz), 122.1, 64.7, 30.7, 19.2 (d, J_{CP} = 16.4 Hz, 2C), 18.6, 13.7; ^{31}P NMR (161 MHz, $CDCl_3$) δ 33.93; IR (film) : 2959, 2934, 2873, 1712, 1634, 1468, 1313, 1267, 1178, 999, 933 cm^{-1}. HRMS (EI) calcd. For $C_{15}H_{21}O_3P$: 280.1228; found : 280.1232.



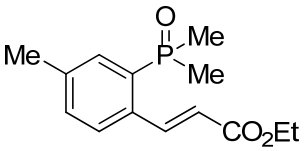
(*E*)-Methyl 3-(2-(dimethylphosphoryl)-3-methylphenyl)acrylate (3b) : Yellow solid. Melting point = 67-71 ^oC; R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^1H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, J = 15.7 Hz, 1H), 7.42-7.33 (m, 2H), 7.28-7.26 (m, 1H), 6.16 (d, J = 15.7 Hz, 1H), 3.81 (s, 3H), 2.67 (s, 3H), 1.89 (d, J = 12.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.9, 146.0 (d, J_{CP} = 4.8 Hz), 142.0 (d, J_{CP} = 9.0 Hz), 139.3 (d, J_{CP} = 9.0 Hz), 133.5 (d, J_{CP} = 10.4 Hz), 131.5 (d, J_{CP} = 90.0 Hz), 131.4 (d, J_{CP} = 2.3 Hz), 127.1 (d, J_{CP} = 9.7 Hz), 120.6, 51.9, 23.2 (d, J_{CP} = 3.8 Hz), 21.1 (d, J_{CP} = 70.0 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 37.14; IR (pellet) : 3054, 2987, 2952, 1632, 1580, 1451, 1436, 1313, 1221, 1168, 930 cm^{-1}. HRMS (EI) calcd. For $C_{13}H_{17}O_3P$: 252.0915; found : 252.0916.



(*E*)-Butyl 3-(2-(dimethylphosphoryl)-3-methylphenyl)acrylate (**3c**) : Yellow oil. R_f= 0.4 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, J = 15.6 Hz, 1H), 7.41-7.33 (m, 2H), 7.27-7.25 (m, 1H), 6.18 (d, J = 15.6 Hz, 1H), 4.21 (t, J = 6.7 Hz, 2H), 2.71 (s, 3H), 1.89 (d, J = 12.8 Hz, 6H), 1.73-1.66 (m, 2H), 1.48-1.39 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 145.5 (d, J_{CP} = 5.0 Hz), 142.7 (d, J_{CP} = 8.6 Hz), 138.8 (d, J_{CP} = 9.3 Hz), 133.6 (d, J_{CP} = 10.3 Hz), 131.6 (d, J_{CP} = 90.1 Hz), 131.3 (d, J_{CP} = 2.6 Hz), 127.0 (d, J_{CP} = 9.6 Hz), 121.2, 64.6, 30.7, 23.2 (d, J_{CP} = 3.5 Hz), 21.2 (d, J_{CP} = 70.1 Hz, 2C), 19.2, 13.7; ^{31}P NMR (121 MHz, $CDCl_3$) δ 37.01; IR (film) : 2959, 2934, 2873, 1633, 1452, 1308, 1168, 1063, 930 cm^{-1}. HRMS (EI) calcd. For $C_{16}H_{23}O_3P$: 294.1385; found : 294.1387.

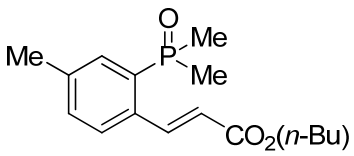


(*E*)-*tert*-Butyl 3-(2-(dimethylphosphoryl)-3-methylphenyl)acrylate (3d) : Red oil. R_f= 0.4 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, J = 15.5 Hz, 1H), 7.40-7.33 (m, 2H), 7.28-7.25 (m, 1H), 6.12 (d, J = 15.5 Hz, 1H), 2.73 (s, 3H), 1.93 (d, J = 12.8 Hz, 6H), 1.53 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 144.1 (d, J_{CP} = 5.1 Hz), 143.2 (d, J_{CP} = 8.2 Hz), 138.5 (d, J_{CP} = 10.0 Hz), 133.6 (d, J_{CP} = 10.3 Hz), 131.0 (d, J_{CP} = 91.9 Hz), 131.4 (d, J_{CP} = 2.0 Hz), 126.9 (d, J_{CP} = 9.7 Hz), 123.2, 81.0, 28.2(3C), 23.3 (d, J_{CP} = 3.4 Hz), 21.0 (d, J_{CP} = 69.9 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 39.07; IR (film) : 2975, 2918, 2850, 1707, 1632, 1365, 1316, 1153, 930 cm^{-1}. HRMS (EI) calcd. For $C_{16}H_{23}O_3P$: 294.1385; found : 294.1382.

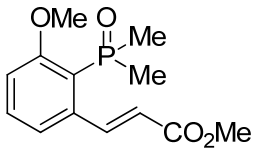


2-Ethoxy-4-*p*-tolyl-6-cyclohexylmethyl-1,2-oxaphosphorin 2-oxide (3e) : Yellow oil. R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (d, J = 15.7 Hz, 1H), 7.74 (dd, J = 13.3 Hz, 0.8 Hz, 1H), 7.59-7.56 (m, 1H), 7.35 (dd, J = 8.0 Hz, 0.5 Hz, 1H), 6.35 (d, J = 15.6 Hz, 1H), 4.28 (q, J = 7.1 Hz, 2H), 2.43 (s, 3H), 1.83 (d, J = 12.9 Hz, 6H), 1.34 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.4, 141.9 (d, J_{CP} = 5.1 Hz), 140.1 (d, J_{CP} = 11.0 Hz), 134.0, 133.2, 132.8, 132.5 (d, J_{CP} = 8.7 Hz), 127.7 (d, J_{CP} = 10.1 Hz), 121.1, 60.7, 21.3, 19.1 (d, J_{CP} = 71.2 Hz, 2C), 14.3; ^{31}P NMR

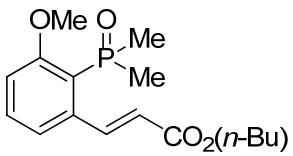
(161 MHz, $CDCl_3$) δ 33.49; IR (film) : 2981, 2920, 1711, 1633, 1313, 1266, 1178, 1036, 945 cm^{-1}. HRMS (EI) calcd. For $C_{14}H_{19}O_3P$: 266.1072; found : 266.1072.



(*E*)-Butyl 3-(2-(dimethylphosphoryl)-4-methylphenyl)acrylate (3f) : White solid. Melting point = 118-122 ^oC; R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^1H NMR (400 MHz, $CDCl_3$) δ 8.27 (d, J = 15.6 Hz, 1H), 7.75 (d, J = 12.6 Hz, 1H), 7.60-7.57 (m, 1H), 7.35 (d, J = 8.0 Hz, 1H), 6.35 (d, J = 15.6 Hz, 1H), 4.22 (t, J = 6.7 Hz, 2H), 2.42 (s, 3H), 1.83 (d, J = 12.9 Hz, 6H), 1.73-1.66 (m, 2H), 1.49-1.39 (m, 2H), 0.97 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 141.9 (d, J_{CP} = 5.0 Hz), 140.1 (d, J_{CP} = 11.0 Hz), 133.9 (d, J_{CP} = 9.5 Hz), 133.7 (d, J_{CP} = 91.9 Hz), 132.8 (d, J_{CP} = 2.3 Hz), 132.6 (d, J_{CP} = 8.6 Hz), 127.6 (d, J_{CP} = 10.1 Hz), 121.1, 64.7, 30.7, 21.4, 19.2, 19.1 (d, J_{CP} = 71.2 Hz, 2C), 13.7; ^{31}P NMR (161 MHz, $CDCl_3$) δ 33.36; IR (pellet) : 2959, 2933, 2873, 1711, 1633, 1479, 1311, 1175, 935 cm^{-1}. HRMS (EI) calcd. For $C_{16}H_{23}O_3P$: 294.1385; found : 294.1384.

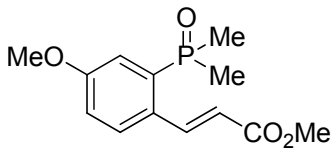


(*E*)-Methyl 3-(2-(dimethylphosphoryl)-3-methoxyphenyl)acrylate (3g) : Yellow oil. R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^1H NMR (400 MHz, $CDCl_3$) δ 9.19 (d, J = 15.9 Hz, 1H), 7.49 (t, J = 8.1 Hz, 1H), 7.20 (dd, J = 7.8 Hz, 2.7 Hz, 1H), 6.99-6.95 (m, 1H), 6.16 (d, J = 15.9 Hz, 1H), 3.90 (s, 3H), 3.79 (s, 3H), 1.83 (d, J = 13.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ; 167.1, 160.0 (d, J_{CP} = 4.4 Hz), 144.8, 142.7, 133.1, 121.7 (d, J_{CP} = 9.3 Hz), 120.7, 120.3 (d, J_{CP} = 92.7 Hz), 111.5 (d, J_{CP} = 6.0 Hz), 55.6, 51.7, 19.3 (d, J_{CP} = 73.0 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 39.91; IR (film) : 2950, 2917, 2842, 1716, 1567, 1465, 1258, 1167, 1064, 866 cm^{-1}. HRMS (EI) calcd. For $C_{13}H_{17}O_4P$: 268.0864; found : 268.0865.

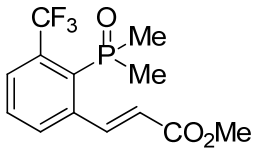


(*E*)-Butyl 3-(2-(dimethylphosphoryl)-3-methoxyphenyl)acrylate (3h) : Yellow oil. R_f= 0.4 (methanol : dichloromethan = 1 : 20); ^1H NMR (400 MHz, $CDCl_3$) δ 9.17 (d, J = 15.9 Hz, 1H), 7.47 (t, J = 8.1 Hz, 1H), 7.20 (dd, J = 7.7 Hz, 2.5 Hz, 1H), 6.98-6.95 (m, 1H), 6.16 (d, J = 15.9 Hz, 1H), 4.18 (t, J = 6.9 Hz,

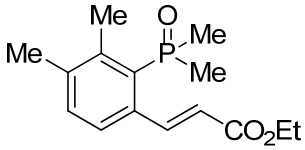
2H), 3.89 (s, 3H), 1.82 (d, J = 13.8 Hz, 6H), 1.73-1.66 (m, 2H), 1.46-1.37 (m, 2H), 0.94 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.7, 159.9 (d, J_{CP} = 4.4 Hz), 144.6 (d, J_{CP} = 2.6 Hz), 142.9 (d, J_{CP} = 4.3 Hz), 133.0, 121.8 (d, J_{CP} = 9.2 Hz), 121.1, 120.6 (d, J_{CP} = 90.5 Hz), 111.4 (d, J_{CP} = 5.9 Hz), 64.4, 55.5, 30.9, 30.8, 19.5 (d, J_{CP} = 68.2 Hz, 2C), 13.8; ^{31}P NMR (161 MHz, $CDCl_3$) δ 38.65; IR (film) : 2959, 2873, 1712, 1566, 1465, 1256, 1168, 1062, 931 cm^{-1}. HRMS (EI) calcd. For $C_{16}H_{23}O_4P$: 310.1334; found : 310.1335.



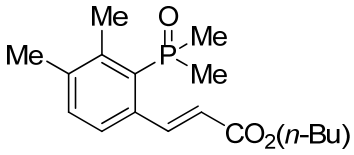
(*E*)-methyl 3-(2-(dimethylphosphoryl)-4-methoxyphenyl)acrylate (3i) : Yellow oil. R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 (d, J = 15.6 Hz, 1H), 7.58 (dd, J = 8.6 Hz, 4.0 Hz, 1H), 7.34 (d, J = 14.0 Hz, 1H), 6.99 (d, J = 8.5 Hz, 1H), 6.23 (d, J = 15.6 Hz, 1H), 3.80 (s, 3H), 3.72 (s, 3H), 1.79 (d, J = 12.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 160.6 (d, J_{CP} = 13.1 Hz), 141.5, 135.3 (d, J_{CP} = 90.8 Hz), 129.4 (d, J_{CP} = 10.3 Hz), 128.8 (d, J_{CP} = 6.3 Hz), 119.1, 117.7, 117.1 (d, J_{CP} = 9.0 Hz), 55.6, 51.8, 18.7 (d, J_{CP} = 71.2 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 37.39; IR (film) : 2953, 2919, 2849, 1713, 1593, 1240, 1172, 1035, 936, 863 cm^{-1}. HRMS (EI) calcd. For $C_{13}H_{17}O_4P$: 268.0864 ; found : 268.0864.



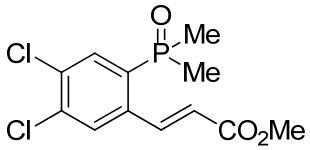
(*E*)-Methyl 3-(2-(dimethylphosphoryl)-3-(trifluoromethyl)phenyl)acrylate (3j) : Yellow oil. R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 (d, J = 15.7 Hz, 1H), 7.83-7.81 (m, 1H), 7.70-7.62 (m, 2H), 6.09 (d, J = 15.7 Hz, 1H), 3.81 (S, 3H), 1.94 (dd, J = 13.4 Hz, 1.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 146.2 (d, J_{CP} = 4.2 Hz), 143.2 (d, J_{CP} = 5.3 Hz), 133.7 (d, J_{CP} = 9.0 Hz), 132.7 (d, J_{CP} = 146.7 Hz), 131.6 (d, J_{CP} = 2.1 Hz), 128.0 (d, J_{CP} = 6.8 Hz), 125.2, 122.5, 120.5, 51.9, 20.5 (d, J_{CP} = 71.2 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 38.60; IR (film) : 2998, 2954, 2923, 1717, 1437, 1318, 1164, 933, 867 cm^{-1}. HRMS (EI) calcd. For $C_{13}H_{14}F_3O_3P$: 306.0633; found : 306.0632.



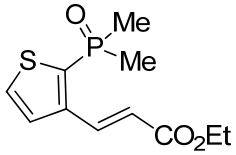
(*E*)-Ethyl 3-(2-(dimethylphosphoryl)-3,4-dimethylphenyl)acrylate (3k) : White solid. Melting point = 96-98 ^{o}C; R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, J = 15.6 Hz, 1H), 7.29-7.26 (m, 2H), 6.13 (d, J = 15.6 Hz, 1H), 4.26 (t, J = 14.2 Hz, 2H), 2.58 (s, 3H), 2.32 (s, 3H), 1.91 (d, J = 12.7 Hz, 6H), 1.33 (t, J = 7.14, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 146.0 (d, J_{CP} = 4.5 Hz), 140.8 (d, J_{CP} = 8.9 Hz), 140.0 (d, J_{CP} = 10.1 Hz), 137.1 (d, J_{CP} = 9.3 Hz), 133.1 (d, J_{CP} = 2.3 Hz), 131.8 (d, J_{CP} = 90.4 Hz), 126.8, 120.0, 60.6, 21.5 (d, J_{CP} = 70 Hz, 2C), 20.9, 18.9 (d, J_{CP} = 5.0 Hz), 14.3; ^{31}P NMR (161 MHz, $CDCl_3$) δ 37.49; IR (pellet) : 3050, 2980, 2921, 1709, 1630, 1446, 1308, 1225, 1170 cm^{-1}. HRMS (EI) calcd. For $C_{15}H_{21}O_3P$: 280.1228; found : 280.1229.



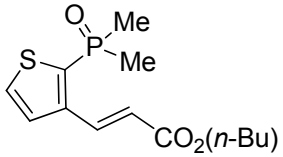
(*E*)-Butyl 3-(2-(dimethylphosphoryl)-3,4-dimethylphenyl)acrylate (3l) : Yellow oil. R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (d, J = 15.6 Hz, 1H), 7.29-7.26 (m, 2H), 6.14 (d, J = 15.6 Hz, 1H), 4.20 (t, J = 6.7 Hz, 2H), 2.59 (s, 3H), 2.32 (s, 3H), 1.90 (d, J = 12.7 Hz, 6H), 1.71-1.67 (m, 2H), 1.46-1.40 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ; 166.7 (d, J_{CP} = 15.6 Hz), 146.0 (d, J_{CP} = 4.9 Hz), 141.0, 140.0 (d, J_{CP} = 8.9 Hz), 136.9 (d, J_{CP} = 9.0 Hz), 133.0, 131.9 (d, J_{CP} = 9.1 Hz), 126.7 (d, J_{CP} = 10.5 Hz), 120.1, 64.5, 30.7, 21.5 (d, J_{CP} = 70 Hz, 2C), 20.9, 19.2, 19.0 (d, J_{CP} = 4.9 Hz), 13.7; ^{31}P NMR (161 MHz, $CDCl_3$) δ 37.48; IR (film) : 2959, 2873, 2873, 1713, 1631, 1456, 1308, 1169, 930 cm^{-1}. HRMS (EI) calcd. For $C_{17}H_{25}O_3P$: 308.1541; found : 308.1540.



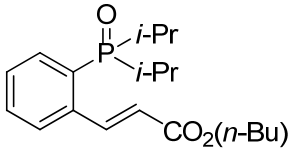
(*E*)-Methyl 3-(4,5-dichloro-2-(dimethylphosphoryl)phenyl)acrylate (3m) : Colorless oil. R_f = 0.3 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (d, J = 15.7 Hz, 1H), 7.94 (d, J = 12.7 Hz, 1H), 7.73 (d, J = 3.9 Hz, 1H), 6.38 (d, J = 15.7 Hz, 1H), 3.83 (s, 3H), 1.83 (d, J = 13.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.1, 139.9 (d, J_{CP} = 4.5 Hz), 136.7, 134.4, 133.9 (d, J_{CP} = 102.0 Hz), 133.7 (d, J_{CP} = 10.4 Hz), 129.6 (d, J_{CP} = 10.3 Hz), 123.1, 52.2, 18.9 (d, J_{CP} = 72 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 33.08; IR (film) : 2998, 2952, 2918, 1720, 1638, 1436, 1311, 1175, 938 cm^{-1}. HRMS (EI) calcd. For $C_{12}H_{13}Cl_2O_3P$: 305.9979; found : 305.9976.



(*E*)-Ethyl 3-(2-(dimethylphosphoryl)thiophen-3-yl)acrylate (3n) : Red solid. Melting point = 107-110 ^{o}C; R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (dd, J = 15.9 Hz, 0.5 Hz, 1H), 7.61 (t, J = 1.6 Hz, 1H), 7.43 (dd, J = 5.0 Hz, 2.0 Hz, 1H), 6.36 (d, J = 15.9 Hz, 1H), 4.27 (q, J = 7.1 Hz, 2H), 1.86 (d, J = 13.3 Hz, 6H), 1.33 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 143.2 (d, J_{CP} = 7.1 Hz), 135.7 (d, J_{CP} = 3.4 Hz), 134.8, 131.2 (d, J_{CP} = 5.7 Hz), 127.6 (d, J_{CP} = 11.2 Hz), 121.6, 60.8, 20.3 (d, J_{CP} = 74.9 Hz, 2C), 14.3; ^{31}P NMR (161 MHz, $CDCl_3$) δ 29.64; IR (pellet) : 3071, 2981, 2911, 1709, 1631, 1408, 1280, 1181, 1038, 862 cm^{-1}. HRMS (EI) calcd. For $C_{11}H_{15}O_3PS$: 258.0480; found : 258.0477.

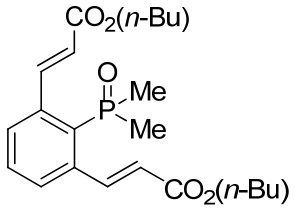


(*E*)-Butyl 3-(2-(dimethylphosphoryl)thiophen-3-yl)acrylate (3o) : Red solid. Melting point = 60-63 ^{o}C; R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, J = 15.9 Hz, 1H), 7.60 (t, J = 3.0 Hz, 1H), 7.43 (dd, J = 5.0 Hz, 1.9 Hz, 1H), 6.36 (d, J = 15.9 Hz, 1H), 4.21 (t, J = 6.7 Hz, 2H), 1.86 (d, J = 13.3 Hz, 6H), 1.73-1.66 (m, 2H), 1.48-1.38 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 143.2 (d, J_{CP} = 7.1 Hz), 135.7 (d, J_{CP} = 3.4 Hz), 134.8, 131.2 (d, J_{CP} = 5.7 Hz), 127.6 (d, J_{CP} = 11.2 Hz), 121.6, 64.7, 30.7, 20.3 (d, J_{CP} = 74.9 Hz, 2C), 19.7, 13.7; ^{31}P NMR (161 MHz, $CDCl_3$) δ 29.60; IR (pellet) : 3070, 2959, 2933, 2873, 1710, 1632, 1409, 1279, 1177, 1037, 862 cm^{-1}. HRMS (EI) calcd. For $C_{13}H_{19}O_3PS$: 286.0793; found : 286.0793.

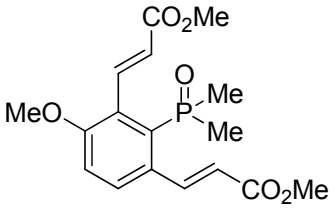


(*E*)-Butyl 3-(2-(diisopropylphosphoryl)phenyl)acrylate (3p) : Yellow oil. R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (d, J = 15.8 Hz, 1H), 7.72-7.68 (m, 2H), 7.55-7.45 (m, 2H), 6.33 (d, J = 15.8 Hz, 1H), 4.22 (t, J = 6.7 Hz, 2H), 2.41-2.32 (m, 2H), 1.74-1.47 (m, 2H), 1.74-1.67 (m, 2H), 1.48-1.39 (m, 2H), 1.30 (d, J = 7.0 Hz, 3H), 1.26 (d, J = 7.0 Hz, 3H), 1.05 (d, J = 7.2 Hz, 3H), 1.00 (d, J = 7.2 Hz, 3H), 0.96 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 143.5 (d, J_{CP} = 3.0 Hz), 139.4, 133.0 (d, J_{CP} = 7.7 Hz), 131.6 (d, J_{CP} = 2.4 Hz), 129.6 (d, J_{CP} = 78.3 Hz), 129.0 (d, J_{CP} = 10.3 Hz), 127.8 (d, J_{CP} = 8.8 Hz), 121.1, 64.6, 30.7, 27.4, 26.7, 19.2, 16.4 (d, J_{CP} = 2.2 Hz,

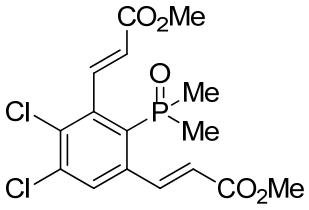
2C), 15.6 (d, J_{CP} = 3.1 Hz, 2C), 13.8; ^{31}P NMR (161 MHz, $CDCl_3$) δ 54.30; IR (film) : 2962, 2930, 2864, 1709, 1630, 1467, 1310, 1254, 1168, 997, 926 cm^{-1}. HRMS (EI) calcd. For C19H29O3P : 336.1854; found : 336.1854.



(2*E*,2'*E*)-Dibutyl 3,3'-(2-(dimethylphosphoryl)-1,3-phenylene)diacrylate (4a) : Yellow oil. R_f = 0.4 (methanol : dichloromethane = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (d, J = 15.6 Hz, 2H), 7.54 (d, J = 2.9 Hz, 3H), 6.22 (d, J = 15.6 Hz, 2H), 4.22 (t, J = 6.7 Hz, 4H), 1.91 (d, J = 12.8 Hz, 6H), 1.73-1.66 (m, 4H), 1.48-1.39 (m, 4H), 0.96 (t, J = 7.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.3(2C), 144.4 (d, J_{CP} = 4.7 Hz, 2C), 139.7 (d, J_{CP} = 7.9 Hz, 2C), 131.9, 130.0 (d, J_{CP} = 9.4 Hz, 2C), 128.3 (d, J_{CP} = 51.3 Hz), 122.1(2C), 64.8(2C), 30.7(2C), 21.3 (d, J_{CP} = 70.6 Hz, 2C), 19.2(2C), 13.7(2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 36.77; IR (film) : 2959, 2933, 2873, 1714, 1633, 1452, 1310, 1241, 1169, 863 cm^{-1}. HRMS (EI) calcd. For $C_{22}H_{31}O_5P$: 406.1909; found : 406.1906.



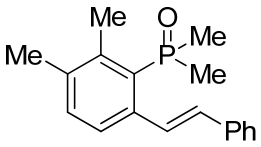
(2*E*,2'*E*)-Dimethyl 3,3'-(2-(dimethylphosphoryl)-4-methoxy-1,3-phenylene)diacrylate (4b) : Yellow oil. R_f = 0.4 (methanol : dichloromethane = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (d, J = 15.6 Hz, 1H), 7.88 (d, J = 16.1 Hz, 1H), 7.56-7.53 (m, 1H), 7.10 (d, J = 8.7 Hz, 1H), 6.38 (d, J = 16.1 Hz, 1H), 6.13 (d, J = 15.6 Hz, 1H), 3.89 (s, 2H), 3.82 (d, J = 10.8 Hz, 7H), 1.89 (d, J = 12.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 145.0, 139.8(2C), 132.5, 131.0 (d, J_{CP} = 11.1 Hz, 2C), 125.1, 121.8 (d, J_{CP} = 75.6 Hz), 119.3, 114.0(2C), 56.0, 51.9 (d, J_{CP} = 14.5 Hz, 2C), 21.5 (d, J_{CP} = 70.7, 2C); ^{31}P NMR (121 MHz, $CDCl_3$) δ 37.36; IR (film) : 2952, 2916, 2844, 1708, 1593, 1488, 1176, 937, 864, 716 cm^{-1}. HRMS (EI) calcd. For $C_{17}H_{21}O_6P$: 352.1076; found : 352.1076.



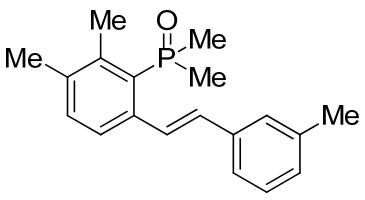
(2*E*,2'*E*)-Dimethyl 3,3'-(4,5-dichloro-2-(dimethylphosphoryl)-1,3-phenylene)diacrylate (4c) : Colorless oil. R_f = 0.4 (methanol : dichloromethane = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (d, J = 15.6 Hz, 1H), 7.86 (d, J = 16.3 Hz, 1H), 7.64 (d, J = 3.3 Hz, 1H), 6.22 (d, J = 15.7 Hz, 1H), 6.03 (t, J = 16.2 Hz, 1H), 3.84 (d, J = 13.3 Hz, 6H), 1.83 (d, J = 13.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.2, 165.5, 142.8 (d, J_{CP} = 3.8 Hz), 142.4 (d, J_{CP} = 3.5 Hz), 139.3 (d, J_{CP} = 7.8 Hz), 137.1, 134.3, 132.7 (d, J_{CP} = 87.9 Hz), 130.2 (d, J_{CP} = 10.3 Hz), 126.8, 122.3, 52.2, 52.1, 21.3 (d, J_{CP} = 70.6 Hz, 2C); ^{31}P NMR (161 MHz, $CDCl_3$) δ 37.07; IR (film) : 3024, 2956, 2904, 1714, 1633, 1448, 1321, 1256, 1100, 860, 726 cm^{-1}. HRMS (EI) calcd. For $C_{16}H_{17}O_5P$: 390.0191; found : 390.0190.

Rh-catalyzed oxidative *ortho*-alkenylation of arylphosphine oxide with styrene derivatives.

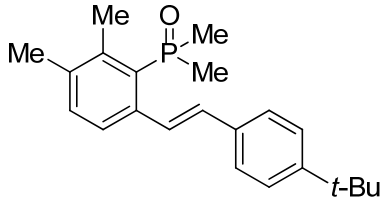
To an oven dried test tube were added $[Cp^*RhCl_2]_2$ (2.9 mg, 2 mol %), $AgSbF_6$ (5.5 mg, 8 mol %), Ag_2CO_3 (55.5 mg, 0.2 mmol), aryl phosphine oxide (0.2 mmol) and styrene (0.6 mmol) in THF (0.8 mL). The resulting mixture was stirred under nitrogen at 90 ^{o}C (bath temperature) for 24 h. After celite filtration and evaporation of the solvent *in vacuo*, product was purified by column chromatography on silica gel.



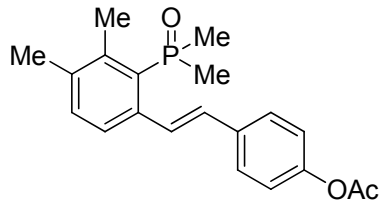
(*E*)-(2,3-Dimethyl-6-styrylphenyl)dimethylphosphine oxide (5a) : Pale yellow solid. Melting point = 156-158 ^{o}C; R_f = 0.4 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, J = 15.9 Hz, 1H), 7.50-7.48 (m, 2H), 7.37-7.33 (m, 3H), 7.29-7.26 (m, 2H), 6.77 (d, J = 15.9 Hz, 1H), 2.60 (s, 3H), 2.31 (s, 3H), 1.90 (d, J = 12.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.5 (d, J_{CP} = 9.2 Hz), 140.1 (d, J_{CP} = 9.9 Hz), 137.8 (d, J_{CP} = 10.4 Hz), 137.3, 133.0 (d, J_{CP} = 2.5 Hz), 130.8, 130.7 (d, J_{CP} = 92.4 Hz), 129.8 (d, J_{CP} = 4.6 Hz), 128.8(2C), 127.8, 126.6(2C), 126.3 (d, J_{CP} = 11.0 Hz), 21.7 (d, J_{CP} = 70.7 Hz, 2C), 20.8, 19.1 (d, J_{CP} = 5.1 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ 38.16; IR (pellet) : 2957, 2919, 2853, 1736, 1626, 1596, 1493, 1430, 1166, 936, 872 cm^{-1}. HRMS (EI) calcd. For $C_{18}H_{21}OP$: 284.1330; found : 284.1327.



(*E*)-(2,3-Dimethyl-6-(3-methylstyryl)phenyl)dimethylphosphine oxide (5b) : Brown oil. R_f = 0.3 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, J = 15.7 Hz, 1H), 7.34-7.23 (m, 5H), 7.09 (d, J = 7.4 Hz, 1H), 6.73 (d, J = 16.0 Hz, 1H), 2.61 (s, 3H), 2.37 (s, 3H), 2.31 (s, 3H), 1.90 (d, J = 12.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.6 (d, J_{CP} = 9.7 Hz), 140.0 (d, J_{CP} = 9.8 Hz), 138.4, 137.8 (d, J_{CP} = 10.3 Hz), 137.2, 133.0 (d, J_{CP} = 2.3 Hz), 131.1, 130.6 (d, J_{CP} = 94.9 Hz), 129.5 (d, J_{CP} = 4.6 Hz), 128.7(2C), 127.3, 126.4 (d, J_{CP} = 11.0 Hz), 123.8, 21.8 (d, J_{CP} = 61.9 Hz, 2C), 21.4, 20.9, 19.1 (d, J_{CP} = 5.1 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ 38.34; IR (film) : 2919, 2859, 1601, 1452, 1292, 1169, 928, 861 cm^{-1}. HRMS (EI) calcd. For $C_{19}H_{23}OP$: 298.1487; found : 298.1484.

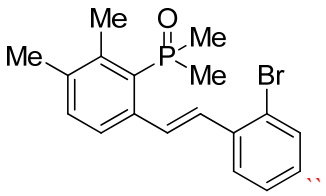


(*E*)-(6-(4-(tert-Butyl)styryl)-2,3-dimethylphenyl)dimethylphosphine oxide (5c) : Brown solid. R_f = 0.3 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 16.0 Hz, 1H), 7.44-7.38 (m, 4H), 7.33-7.26 (m, 2H), 6.75 (d, J = 16.0 Hz, 1H), 2.63 (s, 3H), 2.31 (s, 3H), 1.90 (d, J = 12.7 Hz, 6H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.1, 140.9 (d, J_{CP} = 9.0 Hz), 139.8 (d, J_{CP} = 10.1 Hz), 137.7 (d, J_{CP} = 10.3 Hz), 134.4, 133.0 (d, J_{CP} = 2.7 Hz), 130.8, 130.6 (d, J_{CP} = 92.7 Hz), 128.9 (d, J_{CP} = 5.0 Hz), 126.4(2C), 126.2, 125.8(2C), 34.6, 31.3(3C), 21.8 (d, J_{CP} = 70.2 Hz, 2C), 20.9, 19.0 (d, J_{CP} = 5.0 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ 38.27; IR (pellet) : 2961, 2914, 2864, 1511, 1459, 1291, 1170, 928 cm^{-1}. HRMS (EI) calcd. For $C_{22}H_{29}OP$: 340.1956 ; found : 340.1953.

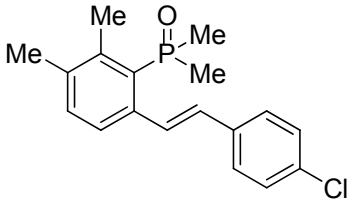


(*E*)-4-(2-(Dimethylphosphoryl)-3,4-dimethylstyryl)phenyl acetate (5d) : colorless oil. R_f = 0.3 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, J = 16.0 Hz, 1H), 7.50 (d, J = 8.5 Hz, 2H), 7.35-7.27 (m, 2H), 7.07 (d, J = 8.6 Hz, 2H), 6.74 (d, J = 16.0 Hz, 1H), 2.57 (s, 3H), 2.31 (s, 3H), 2.30 (s, 3H), 1.91 (d, J = 12.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.5, 150.2, 140.2 (d, J_{CP} = 9.6 Hz), 140.1 (d, J_{CP} = 9.6 Hz), 137.9 (d, J_{CP} = 10.3 Hz), 135.1 133.1 (d, J_{CP} = 2.4 Hz), 130.0 (d, J_{CP} = 4.7 Hz), 129.7, 127.7 (d, J_{CP} = 94.4 Hz), 127.6, 127.2, 126.4 (d, J_{CP} = 11.0 Hz), 121.9, 115.5 (d, J_{CP} = 20.2 Hz), 21.7 (d, J_{CP} = 70.0 Hz, 2C), 21.3, 20.9 (d, J_{CP} = 1.0 Hz), 19.3 (d, J_{CP} = 5.2 Hz); ^{31}P NMR (161

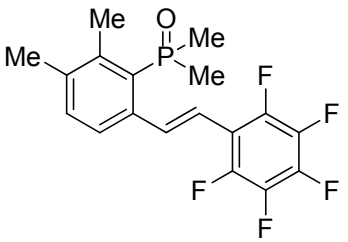
MHz, $CDCl_3$) δ 38.86; IR (film) : 3005, 2923, 1760, 1601, 1505, 1369, 1195, 1166 cm^{-1}. HRMS (EI) calcd. For $C_{20}H_{23}O_3P$: 342.1385; found : 342.1386.



(*E*)-(6-(2-Bromostyryl)-2,3-dimethylphenyl)dimethylphosphine oxide (5e) : Brown oil. R_f = 0.3 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 15.9 Hz, 1H), 7.74 (dd, J = 7.8 Hz, 1.6 Hz, 1H), 7.56 (dd, J = 8.0 Hz, 1.2 Hz, 1H), 7.42-7.39 (m, 1H), 7.32-7.29 (m, 2H), 7.12-7.07 (m, 2H), 2.52 (s, 3H), 2.32 (s, 3H), 1.92 (d, J = 12.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.6 (d, J_{CP} = 9.1 Hz), 139.4 (d, J_{CP} = 9.9 Hz), 138.0 (d, J_{CP} = 10.4 Hz), 137.3, 133.2 (d, J_{CP} = 2.7 Hz), 132.9(2C), 130.6 (d, J_{CP} = 92.2 Hz), 128.9, 128.8, 127.8, 127.3, 127.0 (d, J_{CP} = 10.9 Hz), 123.9, 21.6 (d, J_{CP} = 70.2 Hz, 2C), 20.9, 19.4 (d, J_{CP} = 5.2 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ 38.37; IR (film) : 2917, 2857, 1468, 1436, 1292, 1021, 920 cm^{-1}. HRMS (EI) calcd. For $C_{18}H_{20}BrOP$: 362.0435; found : 362.0433.



(*E*)-(6-(4-Chlorostyryl)-2,3-dimethylphenyl)dimethylphosphine oxide (5f) : Brown solid. Melting point = 86-90 °C; R_f = 0.3 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 16.0 Hz, 1H), 7.43 (d, J = 8.5 Hz, 2H), 7.37-7.28 (m, 4H), 6.70 (d, J = 16.0 Hz, 1H), 2.53 (s, 3H), 2.31 (s, 3H), 1.91 (d, J = 12.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.4 (d, J_{CP} = 8.9 Hz), 139.6 (d, J_{CP} = 9.9 Hz), 137.9 (d, J_{CP} = 10.5 Hz), 136.0, 133.2, 131.1 (d, J_{CP} = 2.5 Hz), 130.7 (d, J_{CP} = 4.5 Hz), 130.6 (d, J_{CP} = 92.2 Hz), 129.0, 128.9(2C), 127.9(2C), 126.4 (d, J_{CP} = 11.0 Hz), 21.7 (d, J_{CP} = 70.0 Hz), 20.8, 19.4 (d, J_{CP} = 5.2 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ 39.0; IR (pellet) : 2919, 2854, 1735, 1589, 1469, 1437, 1292, 1171, 1021 cm^{-1}. HRMS (EI) calcd. For $C_{18}H_{20}ClOP$: 318.0940; found : 318.0937.



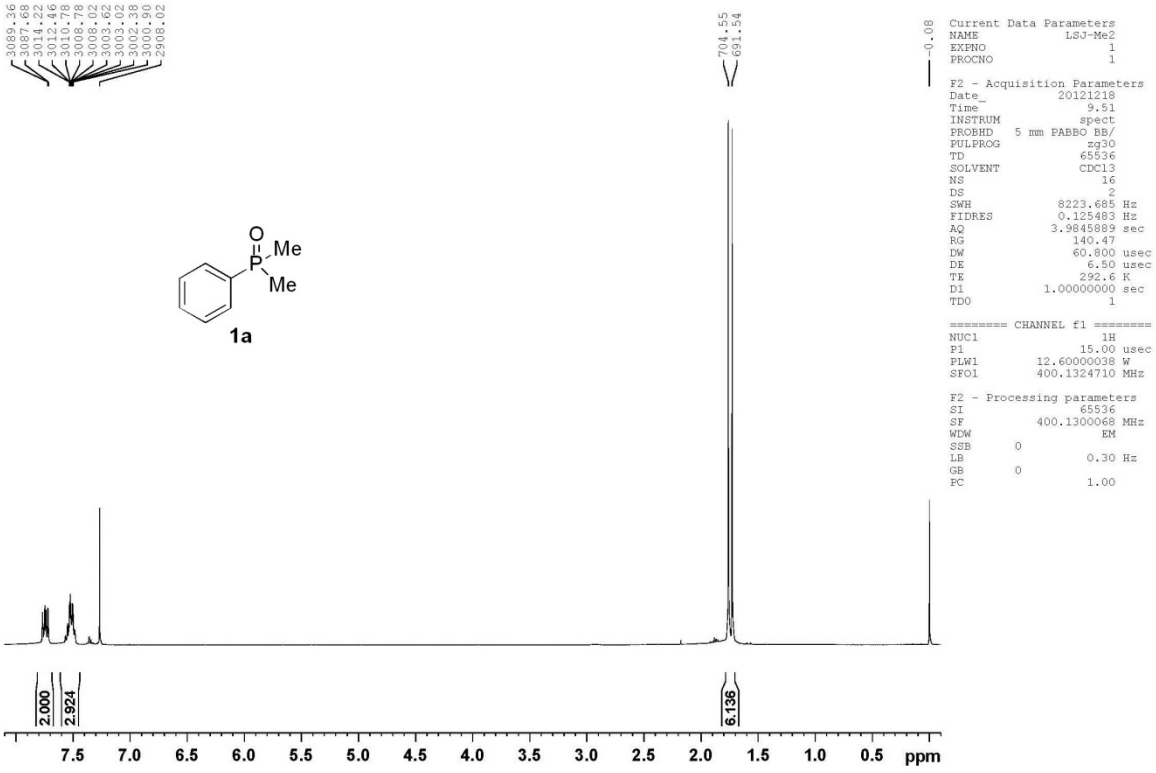
(*E*)-(2,3-Dimethyl-6-(2-(perfluorophenyl)vinyl)phenyl)dimethylphosphine oxide (5g) : Yellow solid. Melting point = 114-116 ^{o}C; R_f = 0.35 (methanol : dichloromethan = 1 : 20); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (d, J = 16.4 Hz, 1H), 7.34 (s, 2H), 6.65 (d, J = 16.4 Hz, 1H), 2.54 (s, 3H), 2.33 (s, 3H), 1.92 (d, J = 12.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.1, 143.6, 140.0 (d, J_{CP} = 8.8 Hz), 139.9 (d, J_{CP} = 9.6 Hz), 139.0 (d, J_{CP} = 10.3 Hz), 138.8, 136.5, 133.2(2C), 131.1 (d, J_{CP} = 91.0 Hz), 126.6, 114.2(2C), 112.5, 21.4 (d, J_{CP} = 70.0 Hz), 21.7 (d, J_{CP} = 70.0 Hz), 20.9, 19.3 (d, J_{CP} = 5.2 Hz); ^{31}P NMR (161 MHz, $CDCl_3$) δ 38.2; IR (pellet) : 3056, 2953, 2928, 2842, 1709, 1614, 1410, 1269, 1167, 1028, 906, 732 cm^{-1}. HRMS (EI) calcd. For $C_{18}H_{16}F_5OP$: 374.0859; found : 374.0857.

References

1. S. N. Tverdomed, J. Kolanowski, E. Lock, G-V. Roschenthaler, *Tetrahedron,* 2011, **67**, 3887.

2. A. J. Blomfield, S. B. Herzon, *Org. Lett*., **2012**, *14*, 4370.

3. W-R. Zheng, J-L. Xu, T. Huang, Z-C. Chen, G. Wang, *Computational & Theoretical chemistry,* 2011, **968**, 1.

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TE                    292.6 K
D1                 1.00000000 sec
TD0                         1

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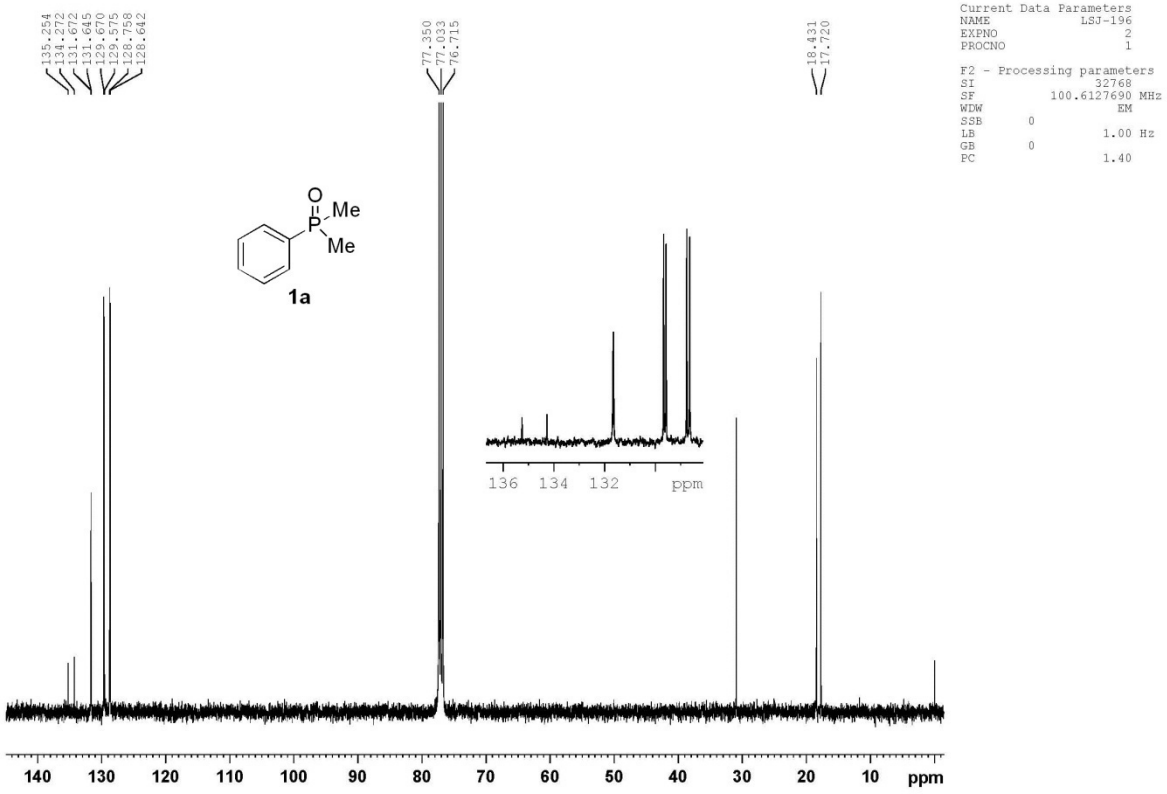


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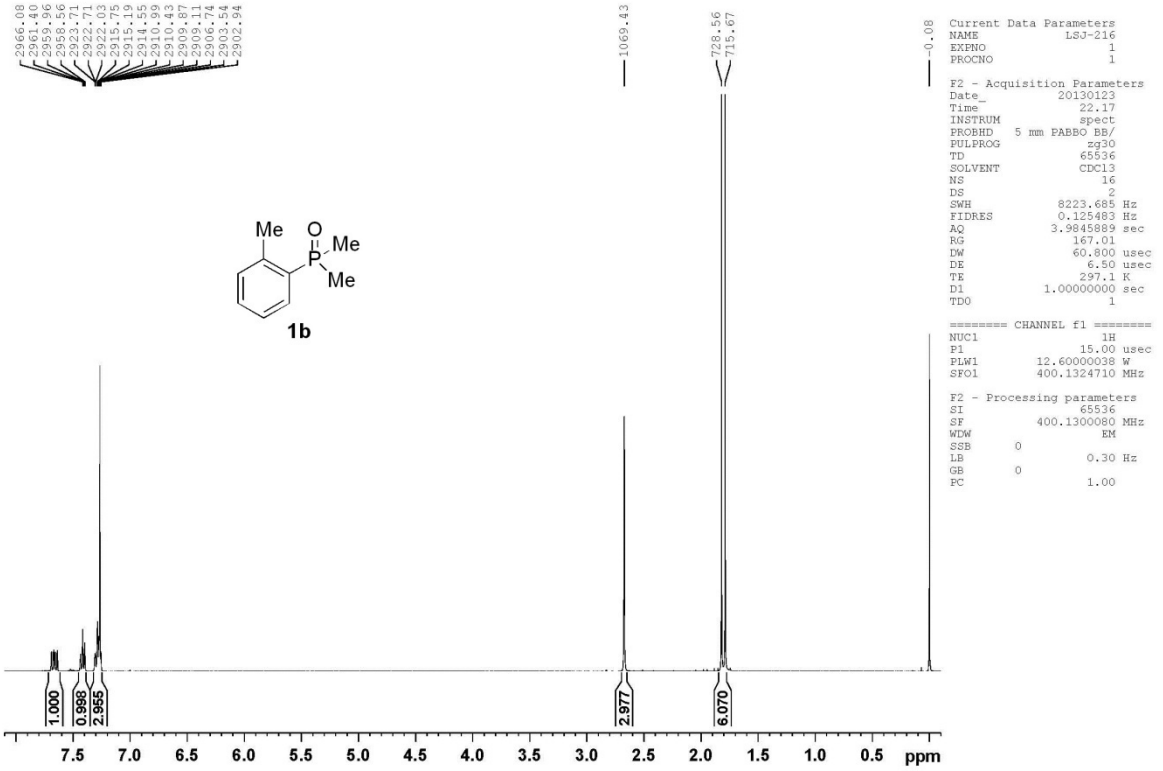
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2909.87
2909.11
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2902.94
1069.43
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719.67
-0.08
Me
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P
Me
1b
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0.998
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TE 297.1 K
D1 1.00000000 sec
TD0 1
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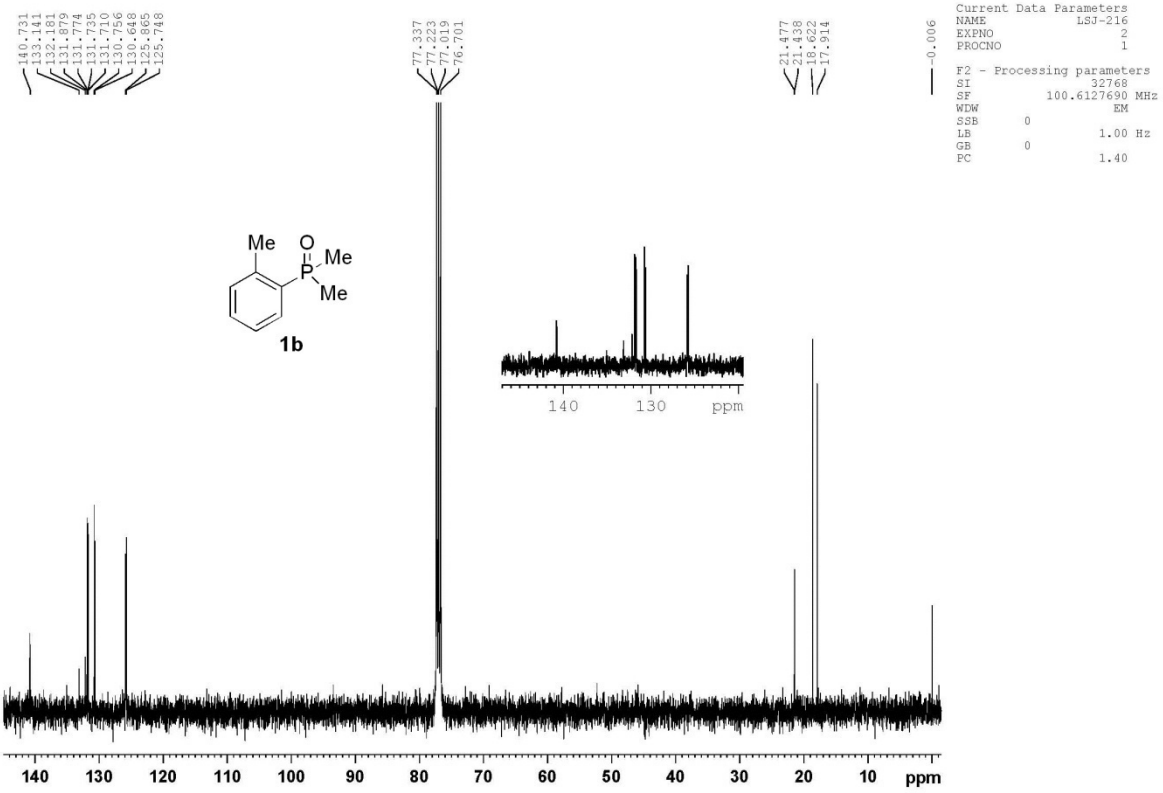
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1b
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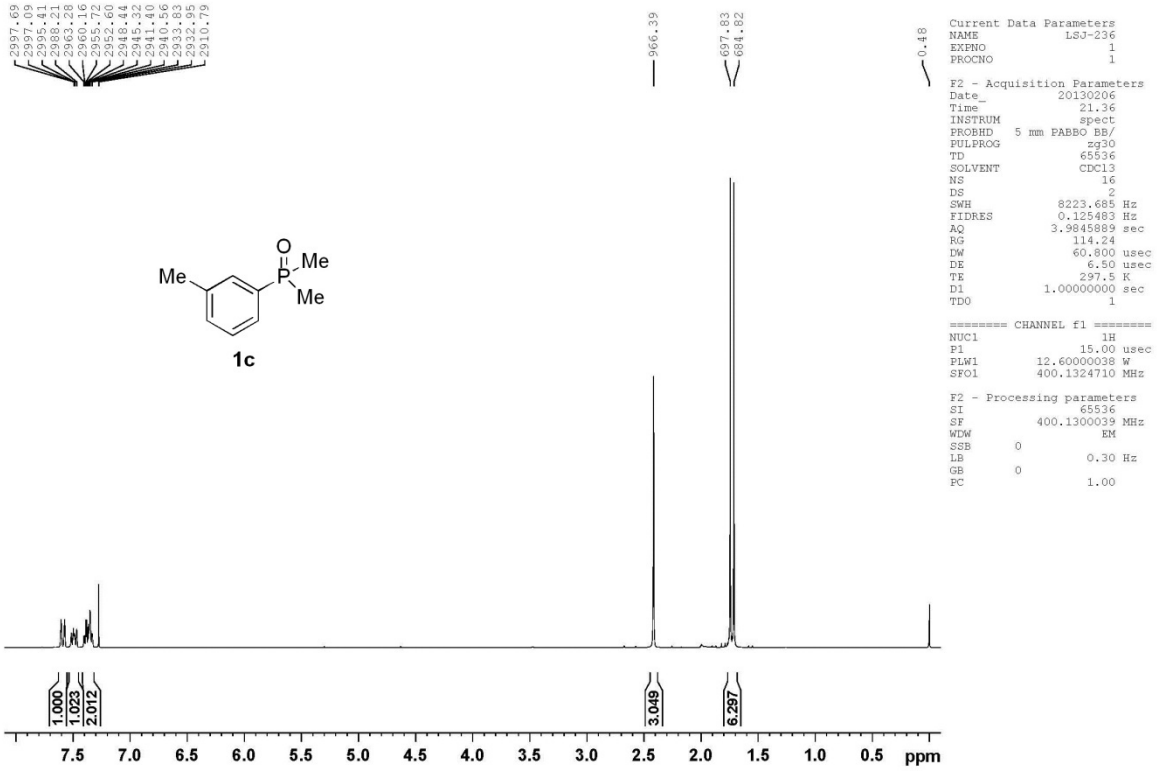


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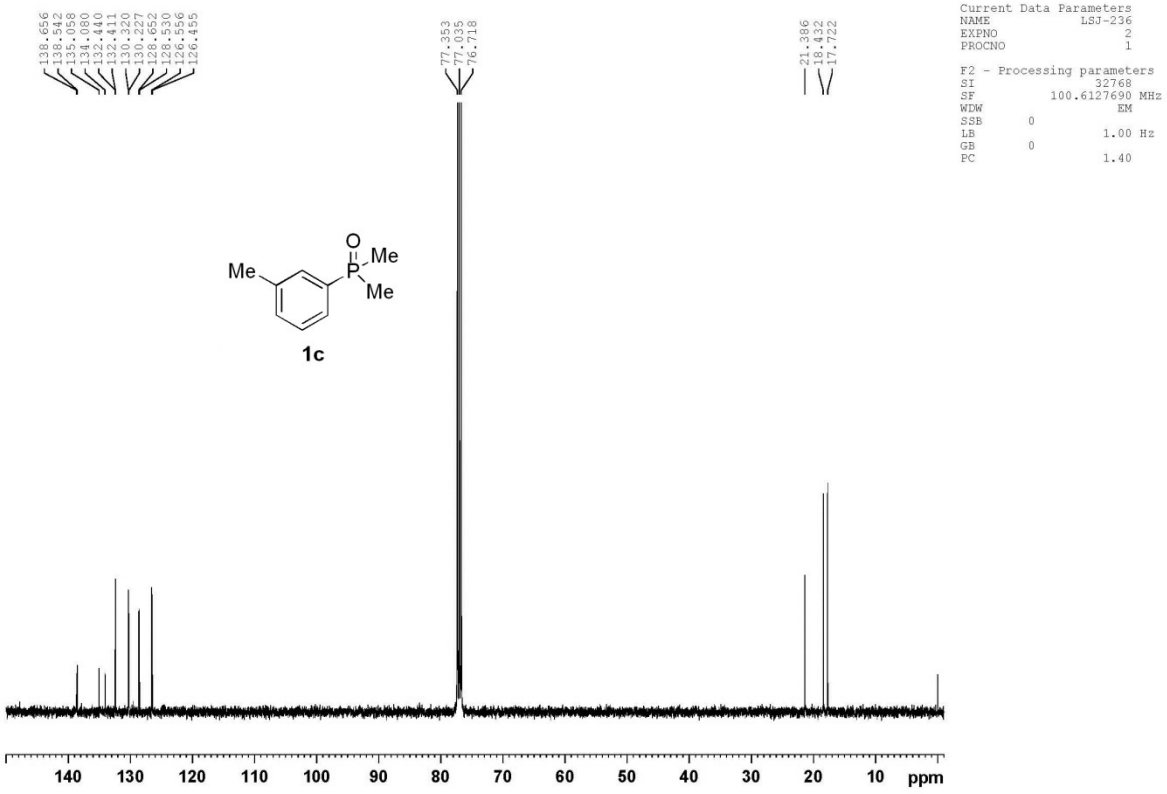


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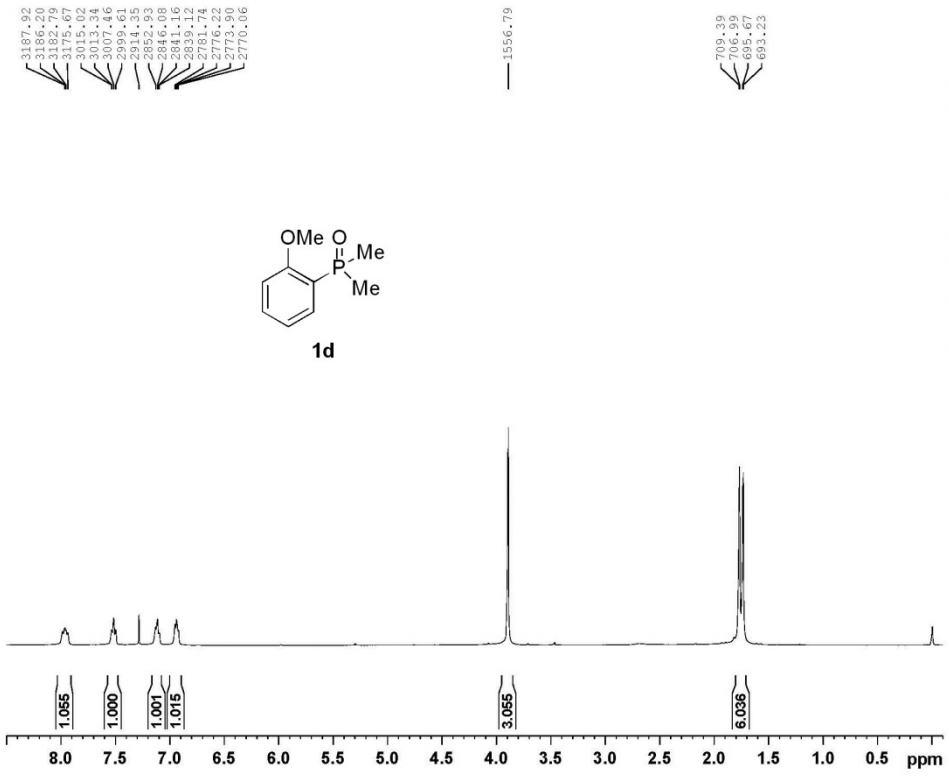


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PROCNO                      1

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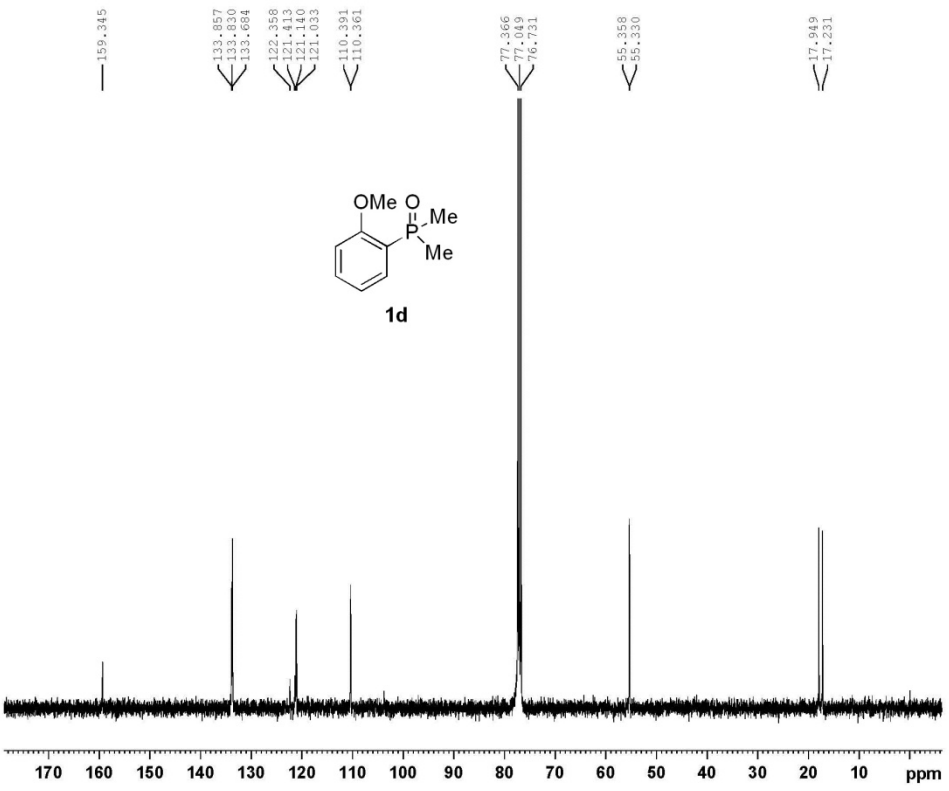


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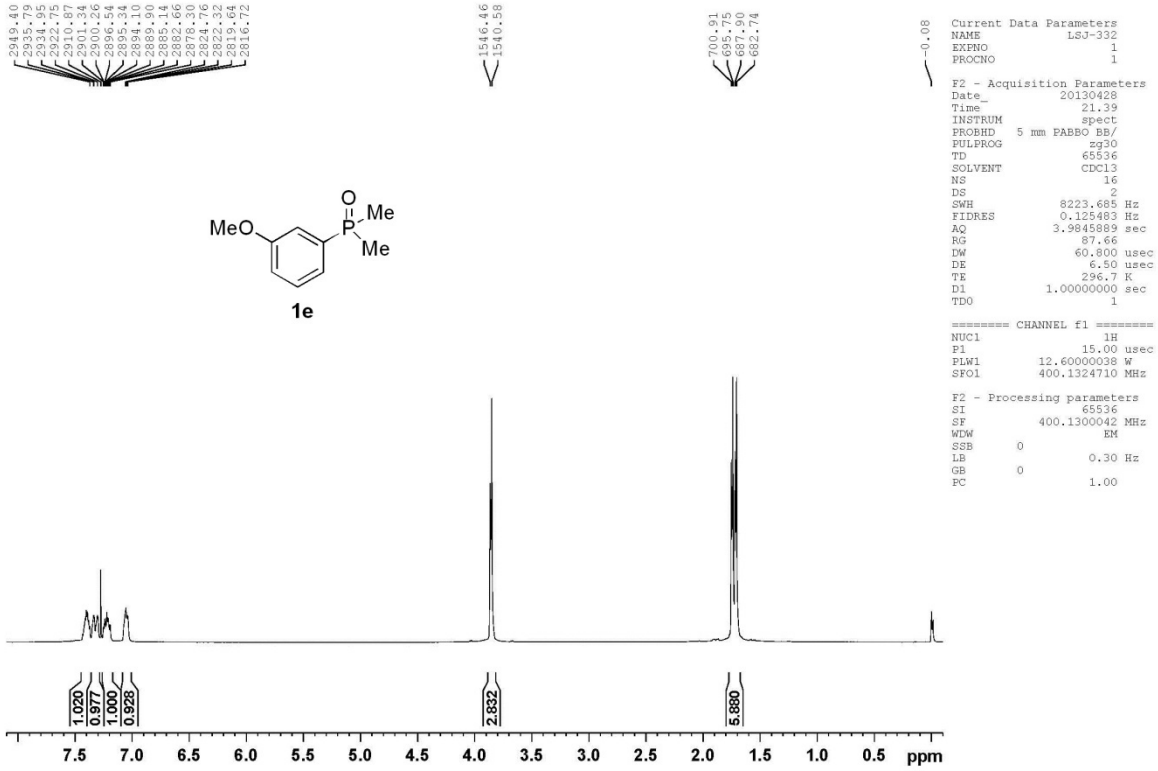
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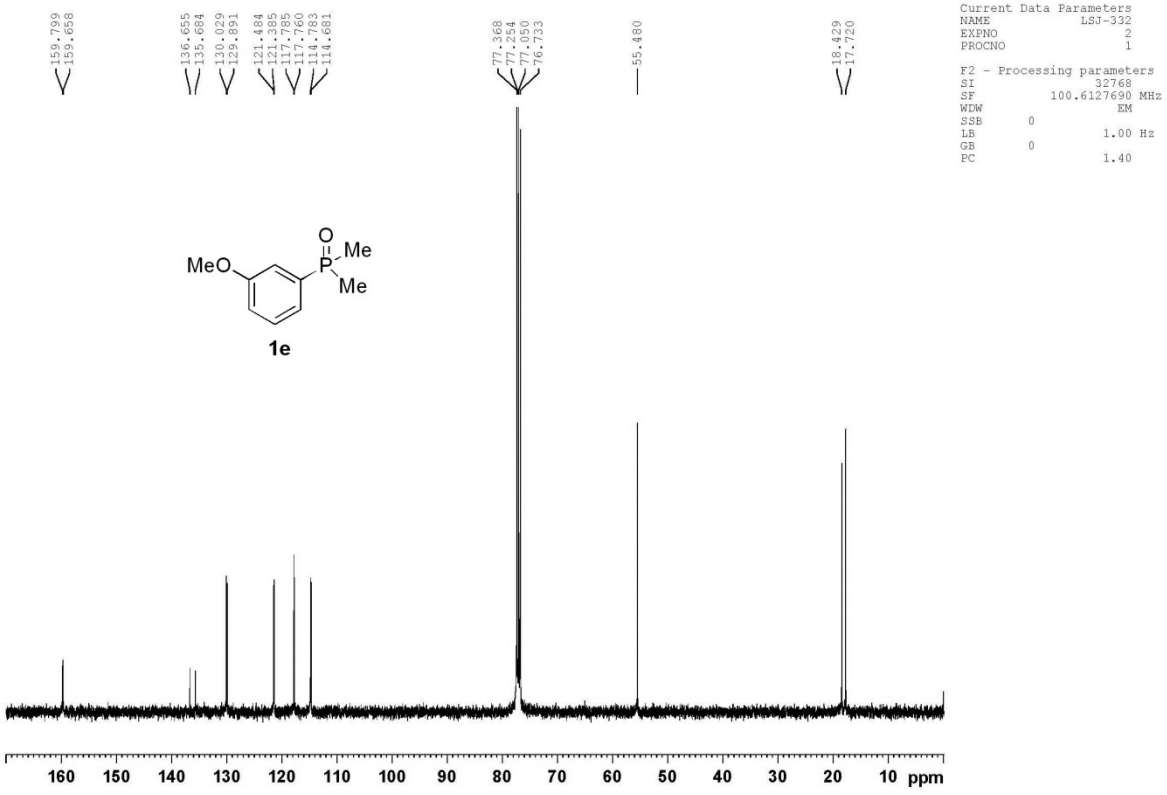
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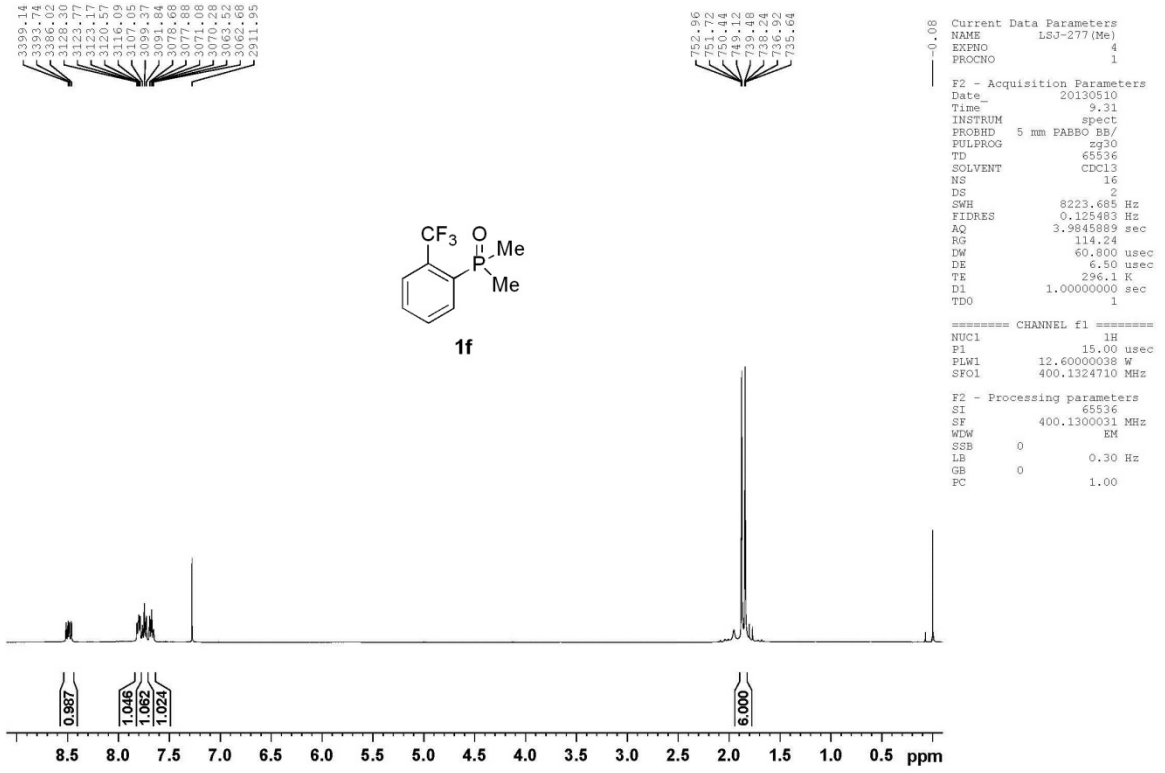
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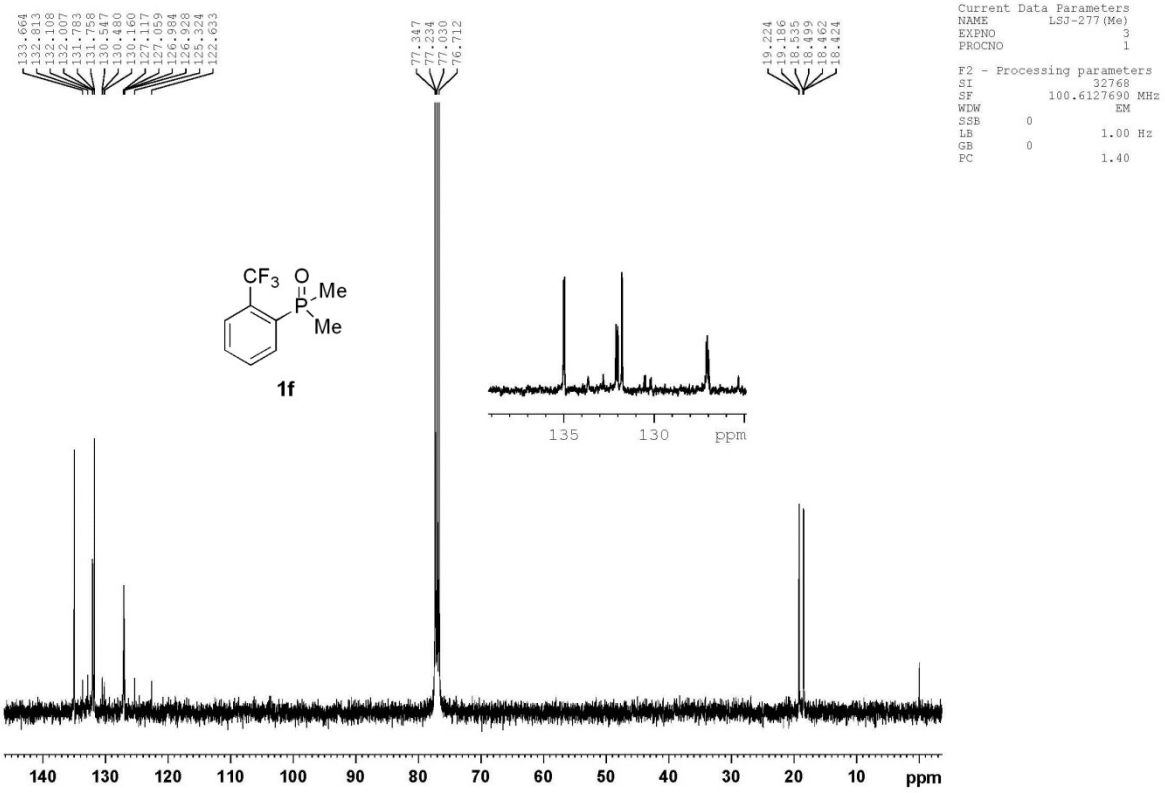
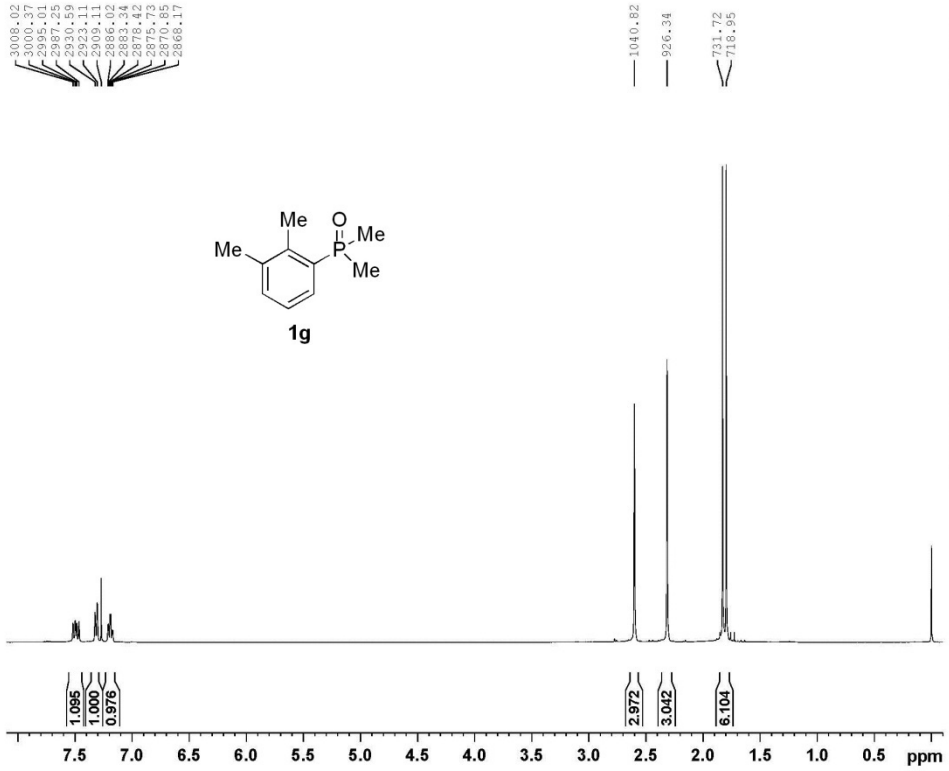
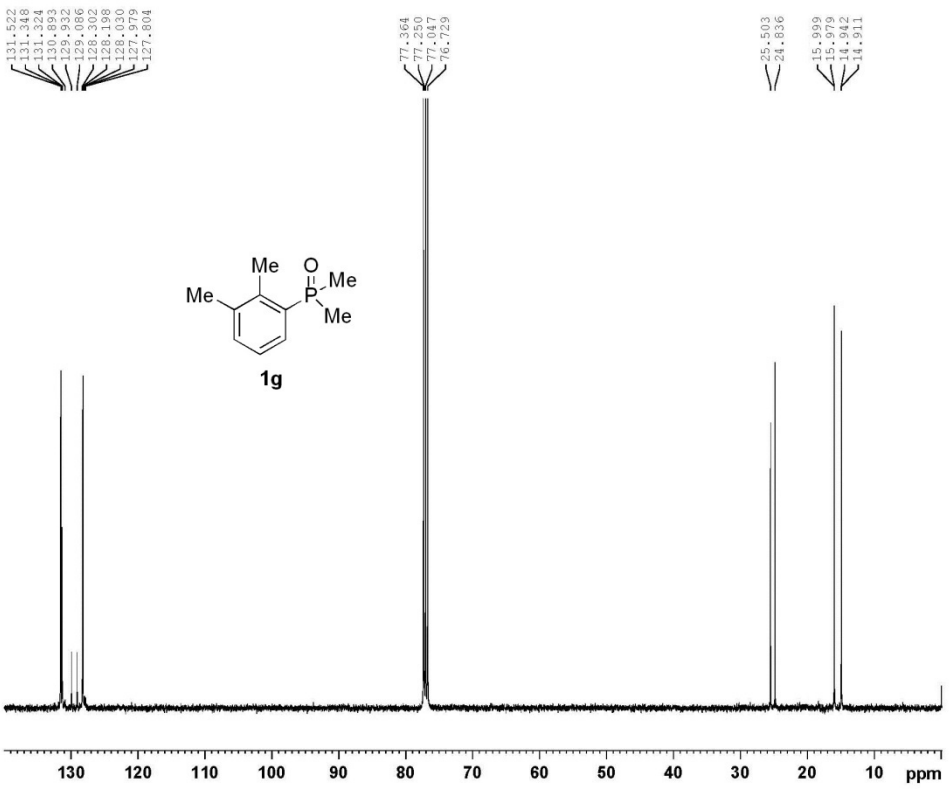
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

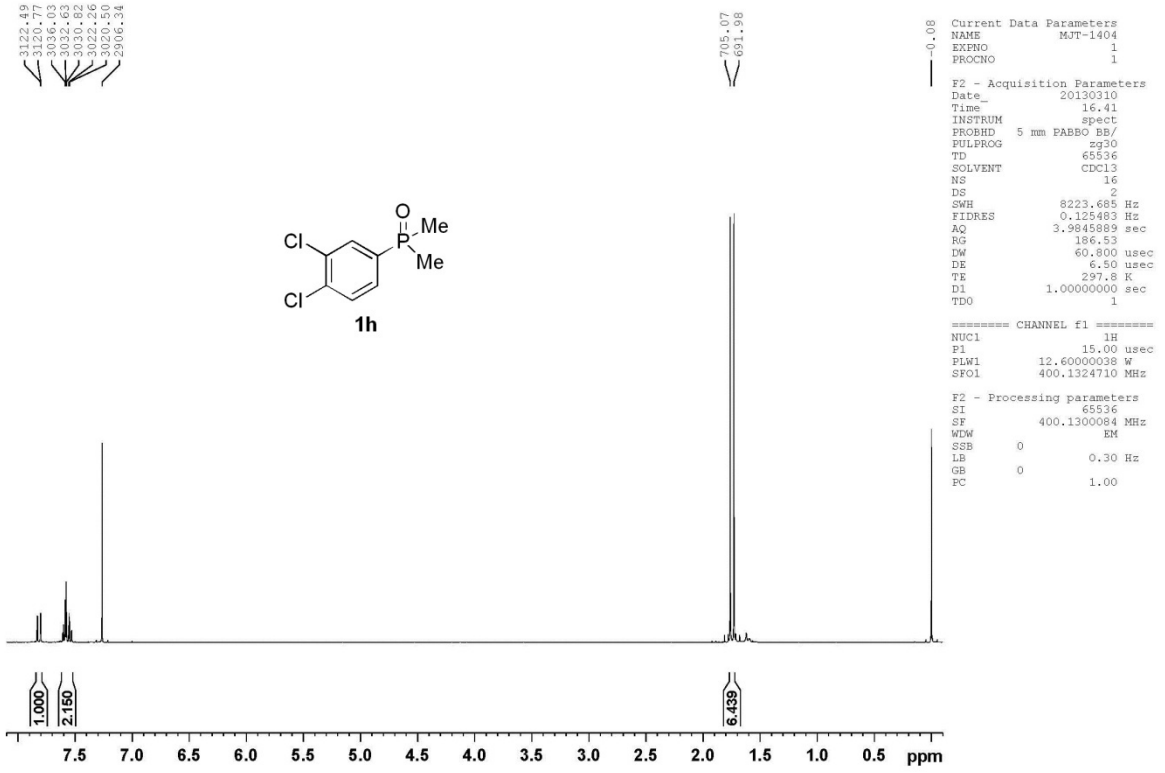

133.664
132.813
132.108
132.007
131.783
131.758
130.547
130.480
130.160
127.117
127.059
126.984
126.938
126.324
122.633
77.347
77.234
77.030
76.712
19.224
19.186
18.535
18.499
18.462
18.424
Current Data Parameters
NAME LSJ-277(Me)
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
CF3
O
Me
P
Me
1f
135 130 ppm
140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



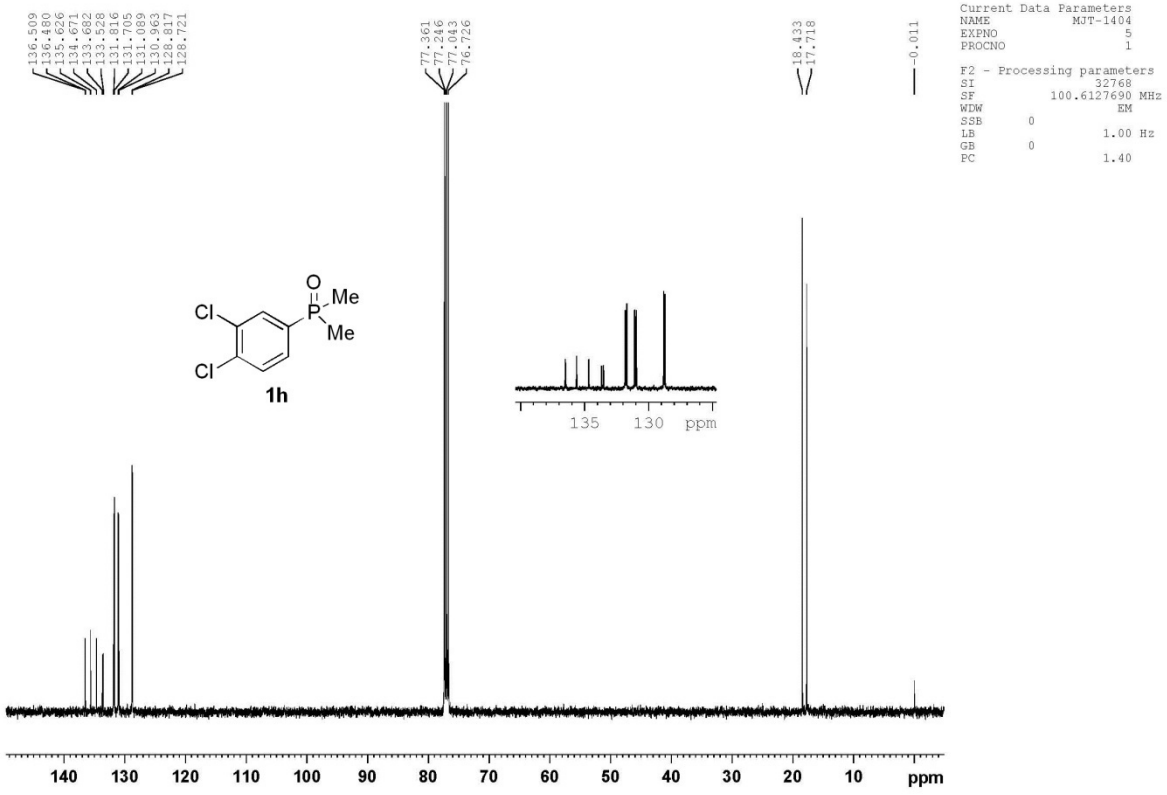

```
Current Data Parameters
NAME                  LSJ-360
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130515
Time                  14.16
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9845889 sec
RG                   114.24
DW                   60.800 usec
DE                    6.50 usec
TE                   296.0 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PLW1             12.60000038 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF               400.1300057 MHz
WDW                         EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



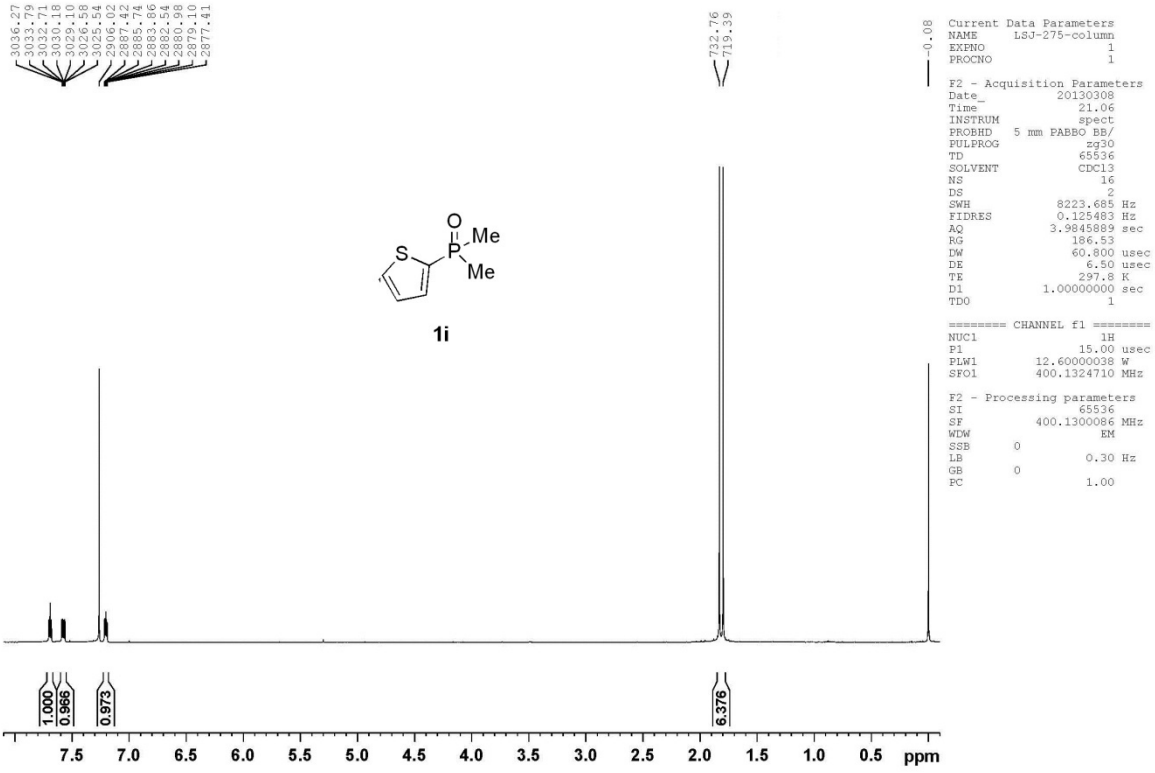

```
Current Data Parameters
NAME                  LSJ-360
EXPNO                       2
PROCNO                      1

F2 - Processing parameters
SI                        32768
SF               100.6127690 MHz
WDW                         EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

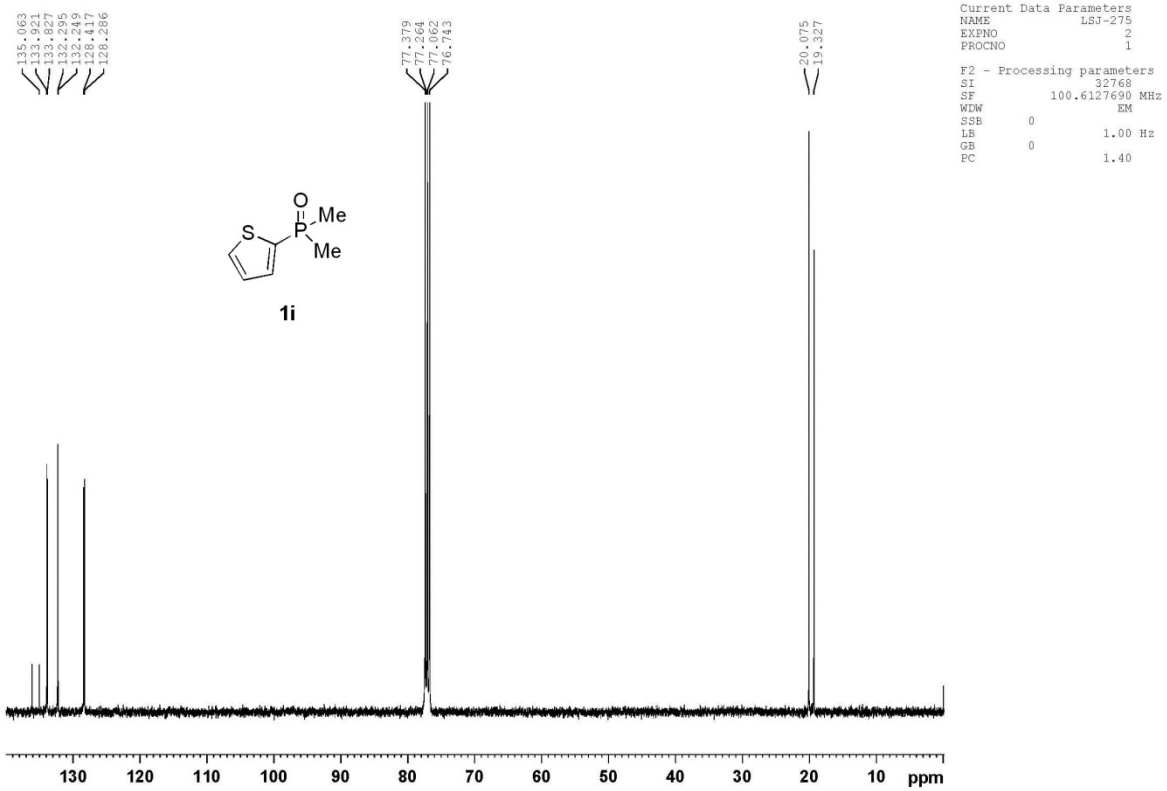


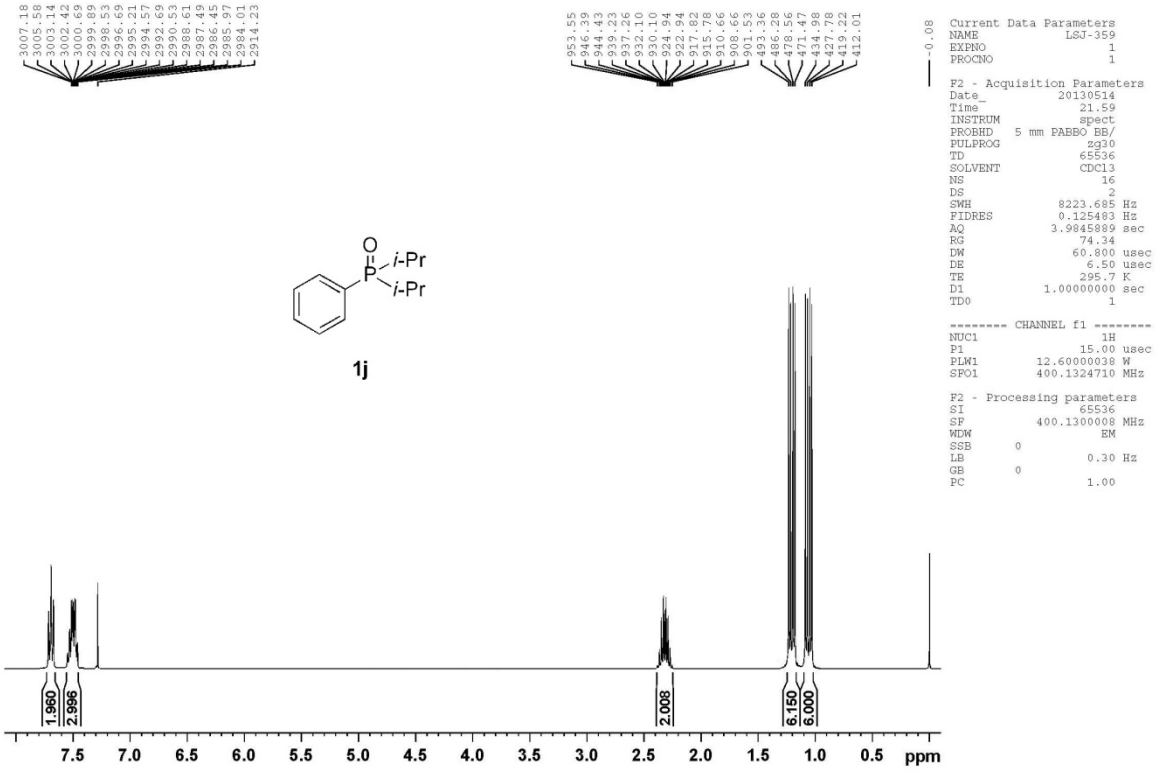

```
Current Data Parameters
NAME                  MJT-1404
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20130310
Time                   16.41
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9845889 sec
RG                    186.53
DW                    60.800 usec
DE                     6.50 usec
TE                    297.8 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PLW1              12.60000038 W
SFO1              400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF                400.1300084 MHz
WDW                         EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```

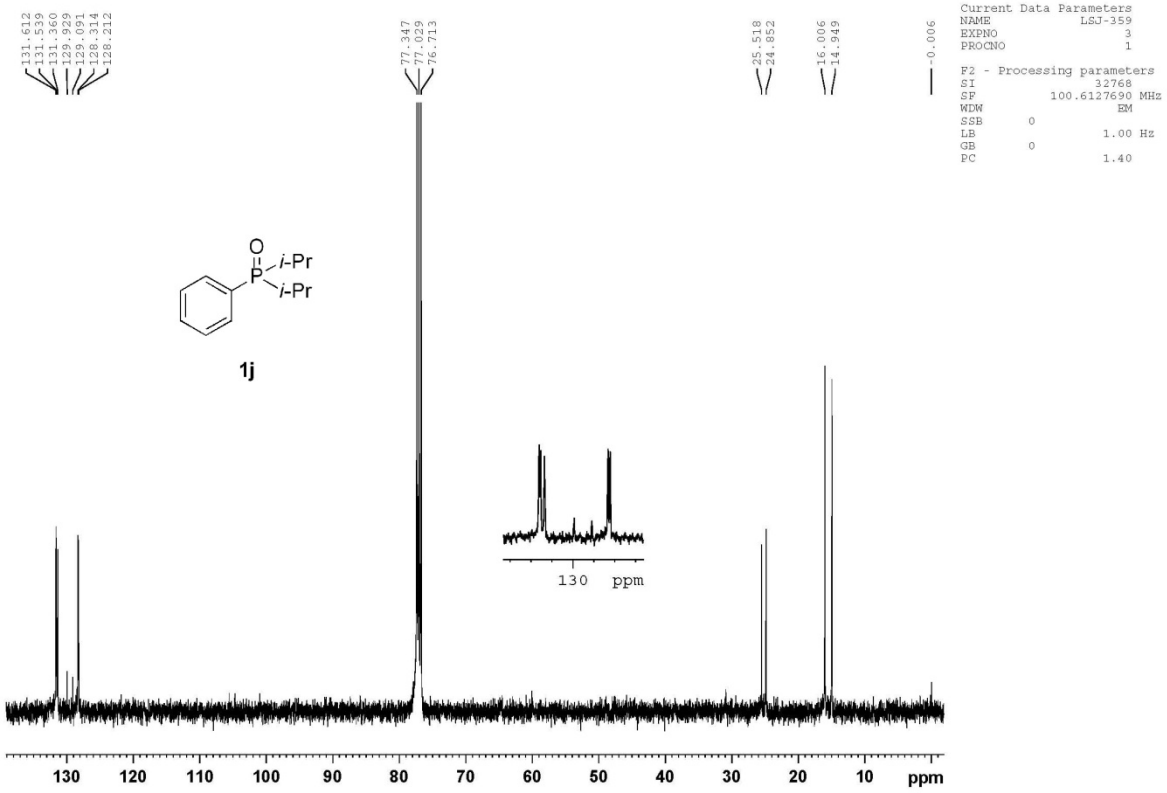


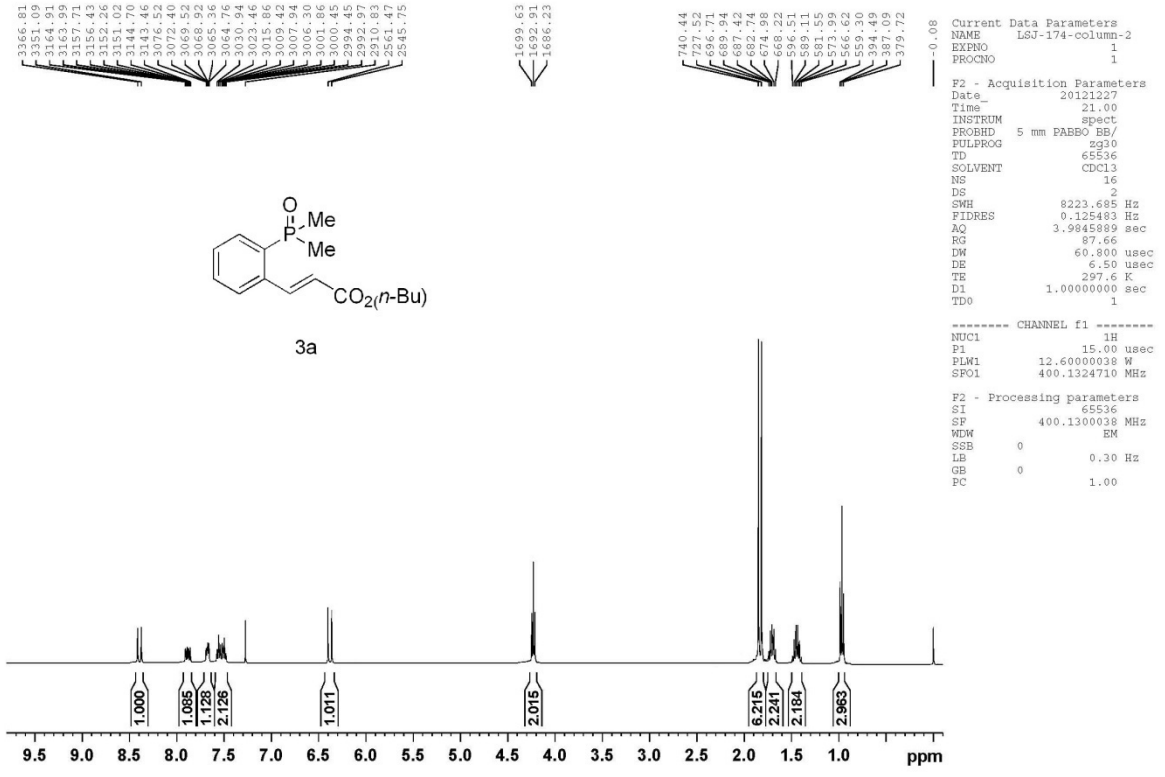

```
Current Data Parameters
NAME                  MJT-1404
EXPNO                        5
PROCNO                       1

F2 - Processing parameters
SI                        32768
SF                100.6127690 MHz
WDW                         EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```


3036.27
3033.79
3032.71
3030.18
3029.10
3026.58
3025.54
2906.02
2887.42
2885.74
2883.86
2882.54
2880.98
2879.10
2877.41
732.76
719.39
-0.08
Current Data Parameters
NAME LSJ-275-column
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130308
Time 21.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 186.53
DW 60.800 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
O
S
P
1i
1.000
0.966
0.973
6.376
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

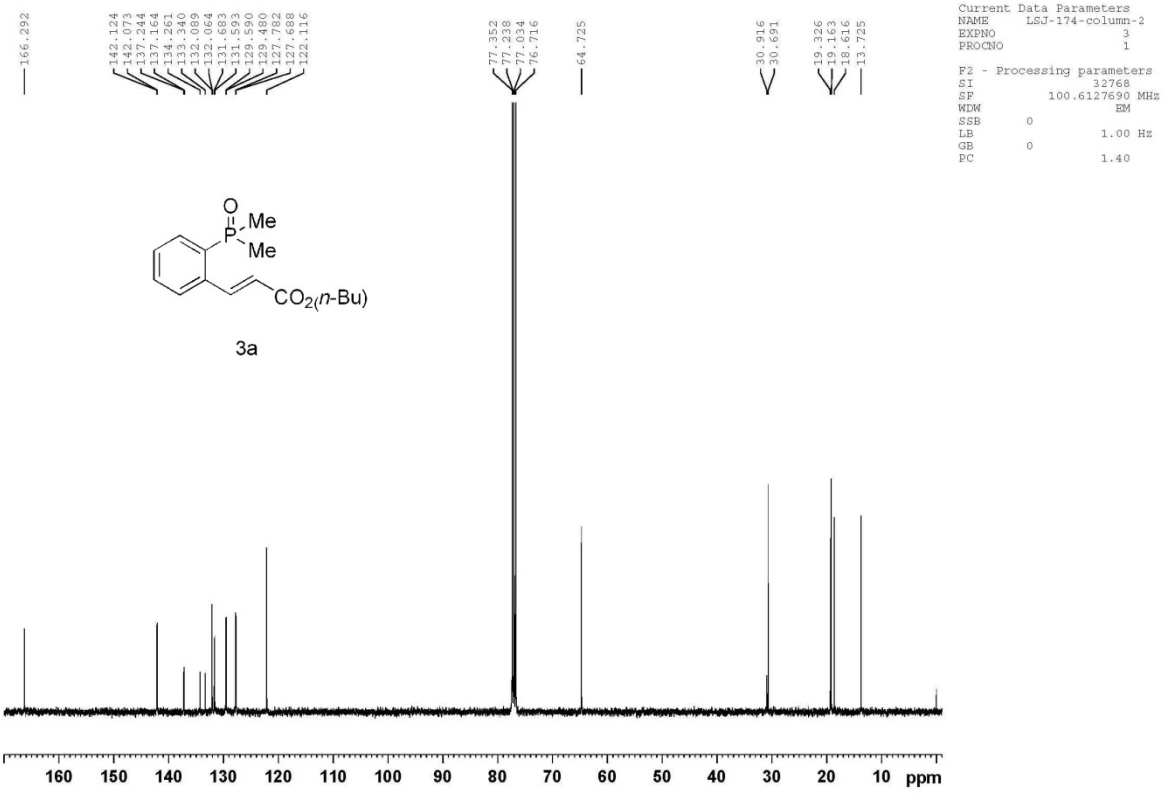

135.063
133.921
133.827
132.295
132.249
128.417
128.286
77.379
77.264
77.062
76.743
20.075
19.327
Current Data Parameters
NAME LSJ-275
EXPNO 2
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
Me
O
S
P
1i
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


3007.18
3005.58
3003.14
3002.42
3000.69
2999.89
2998.53
2996.69
2995.21
2994.57
2992.69
2990.53
2988.61
2987.49
2986.45
2985.97
2984.01
2914.23
953.55
946.39
944.43
939.23
937.26
932.10
930.10
924.94
922.94
917.82
915.78
910.66
908.66
901.53
493.36
486.28
478.56
471.47
434.98
427.78
419.22
412.01
-0.08
Current Data Parameters
NAME LSJ-359
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130514
Time 21.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 74.34
DW 60.800 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SPO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300008 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
P
i-Pr
i-Pr
1j
1.960
2.996
2.008
6.150
6.000
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

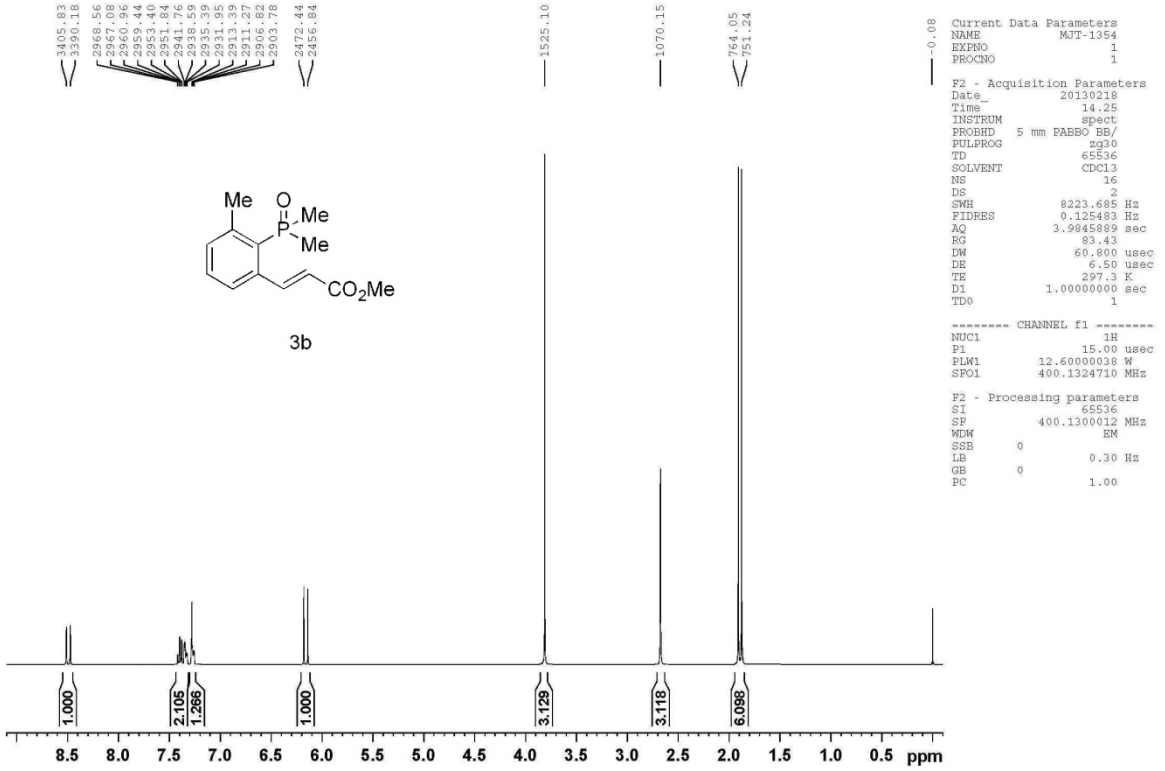

131.612
131.539
131.360
129.929
129.091
128.314
128.212
77.347
77.029
76.713
25.518
24.852
16.006
14.949
-0.006
Current Data Parameters
NAME LSJ-359
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
P
i-Pr
i-Pr
1j
130 ppm
130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



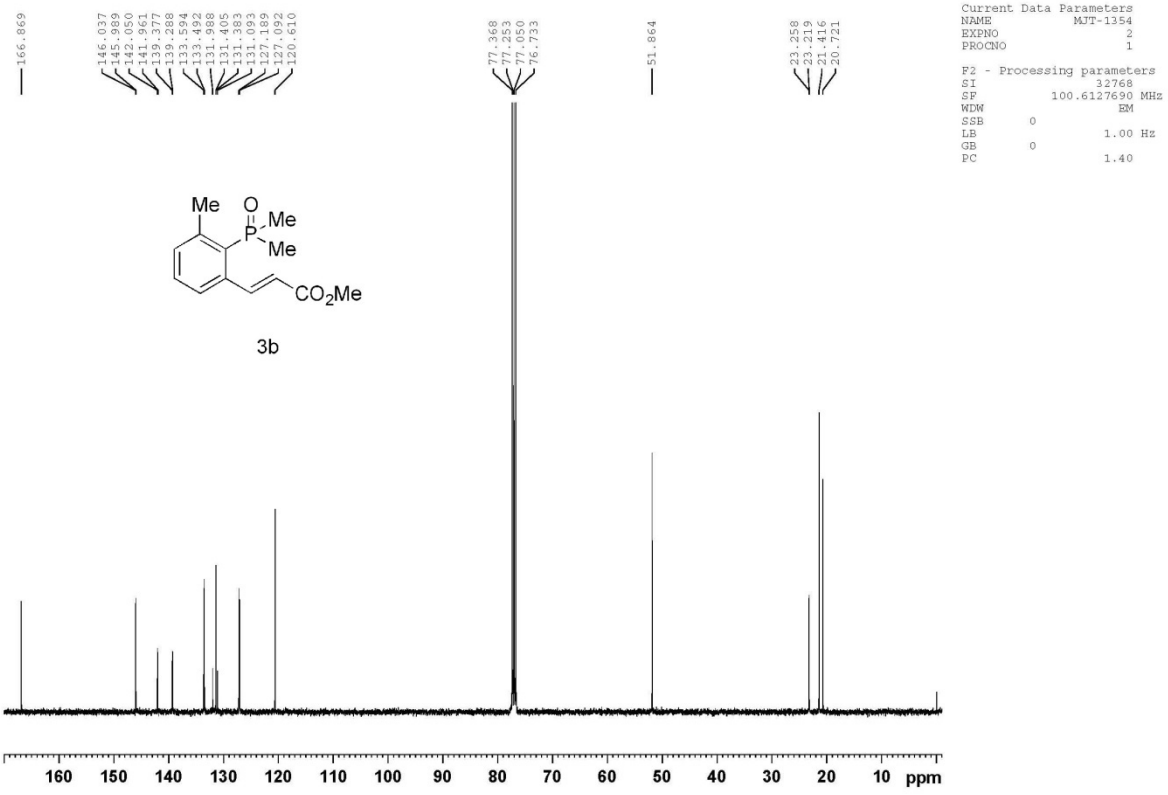

```
Current Data Parameters
NAME      LSJ-174-column-2
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20121227
Time_              21.00
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9845889 sec
RG                 87.66
DW                60.800 usec
DE                 6.50 usec
TE                297.6 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                   1H
P1                 15.00 usec
PLW1         12.60000038 W
SPO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300038 MHz
WDW                   EM
SSB        0
LB                 0.30 Hz
GB         0
PC                 1.00
```



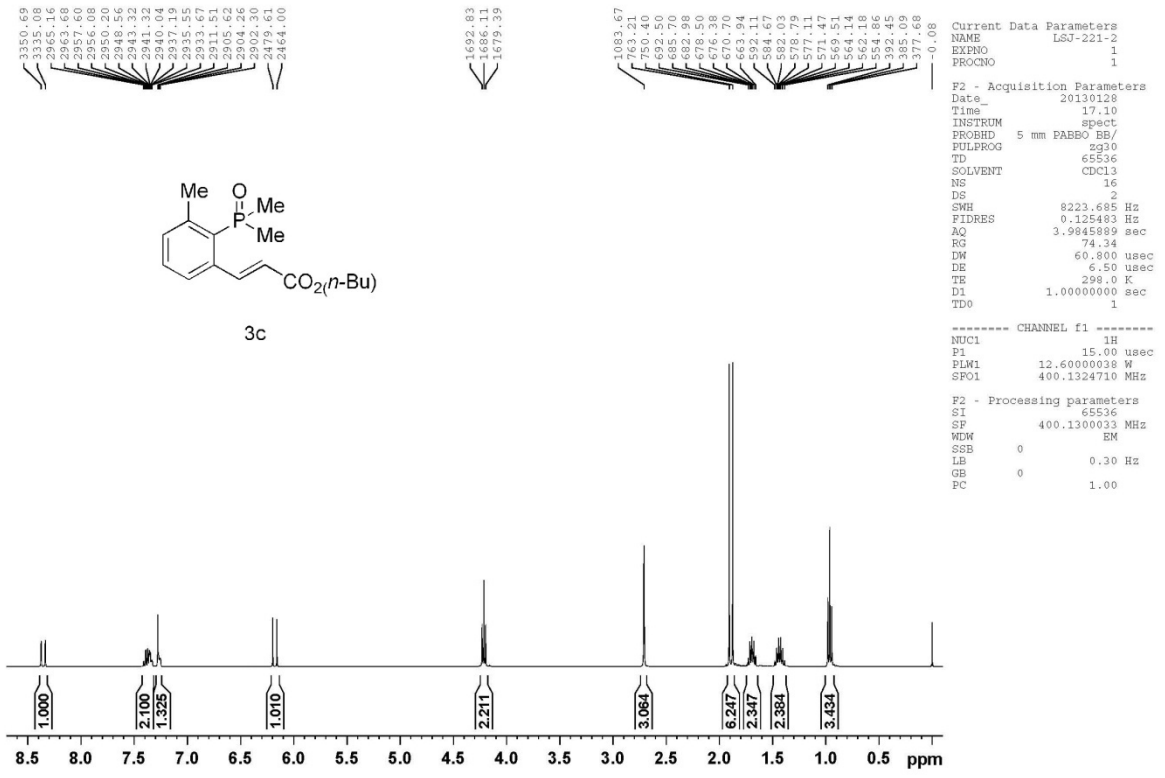

```
Current Data Parameters
NAME      LSJ-174-column-2
EXPNO                    3
PROCNO                   1

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB        0
LB                 1.00 Hz
GB         0
PC                 1.40
```



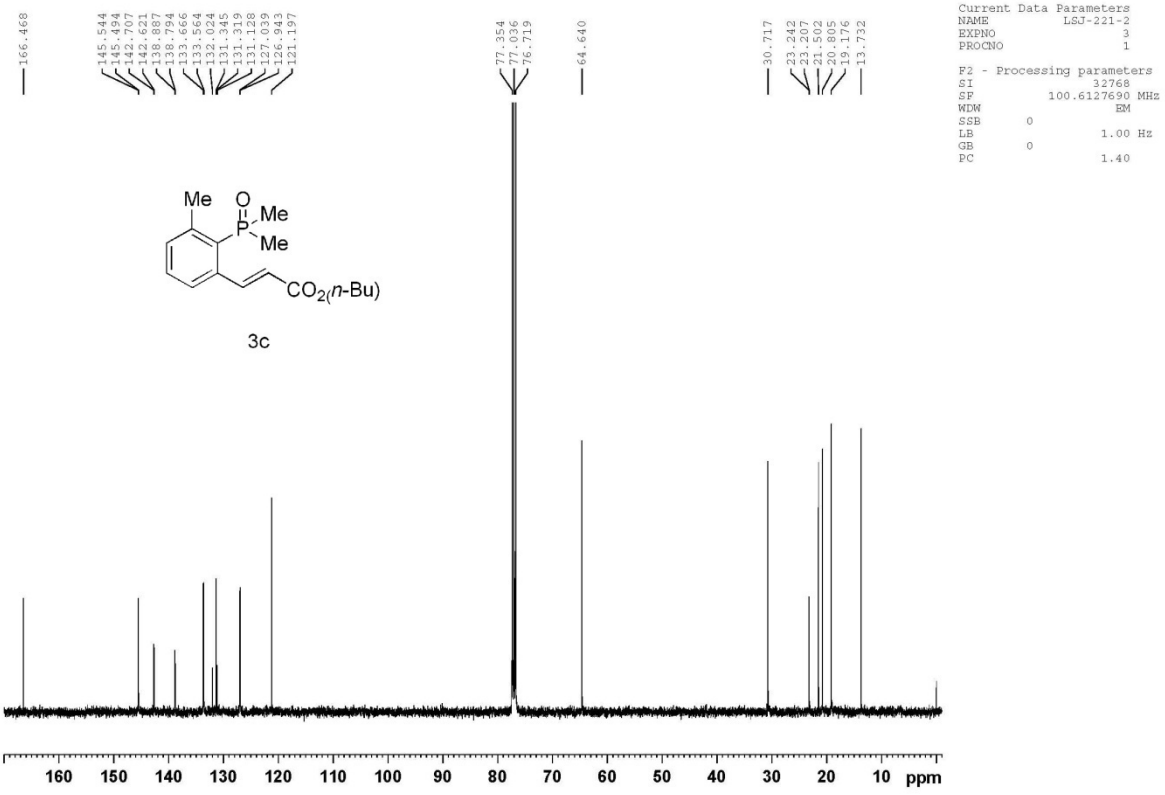

```
Current Data Parameters
NAME                  MJT-1354
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20130218
Time                   14.25
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9845889 sec
RG                     83.43
DW                    60.800 usec
DE                     6.50 usec
TE                    297.3 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PLW1             12.60000038 W
SPO1            400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF               400.1300012 MHz
WDW                         EM
SSB                  0
LB                     0.30 Hz
GB                   0
PC                     1.00
```



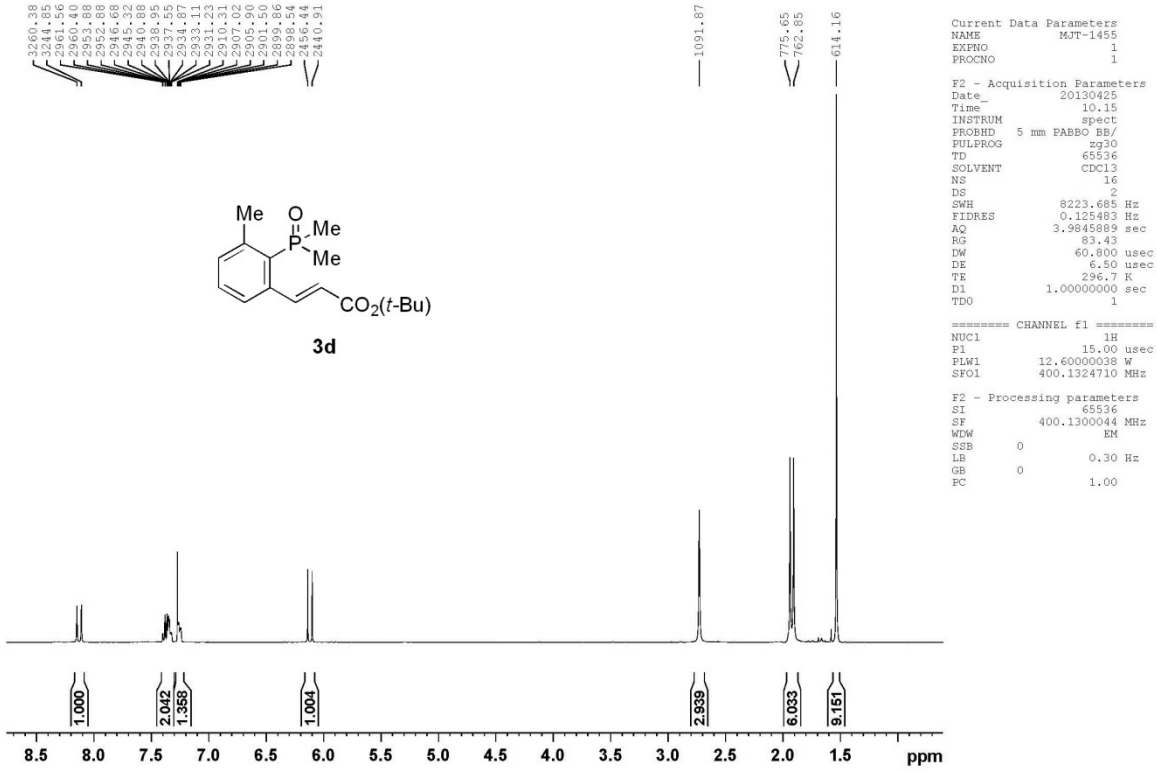

```
Current Data Parameters
NAME                  MJT-1354
EXPNO                        2
PROCNO                       1

F2 - Processing parameters
SI                        32768
SF               100.6127690 MHz
WDW                         EM
SSB                  0
LB                     1.00 Hz
GB                   0
PC                     1.40
```



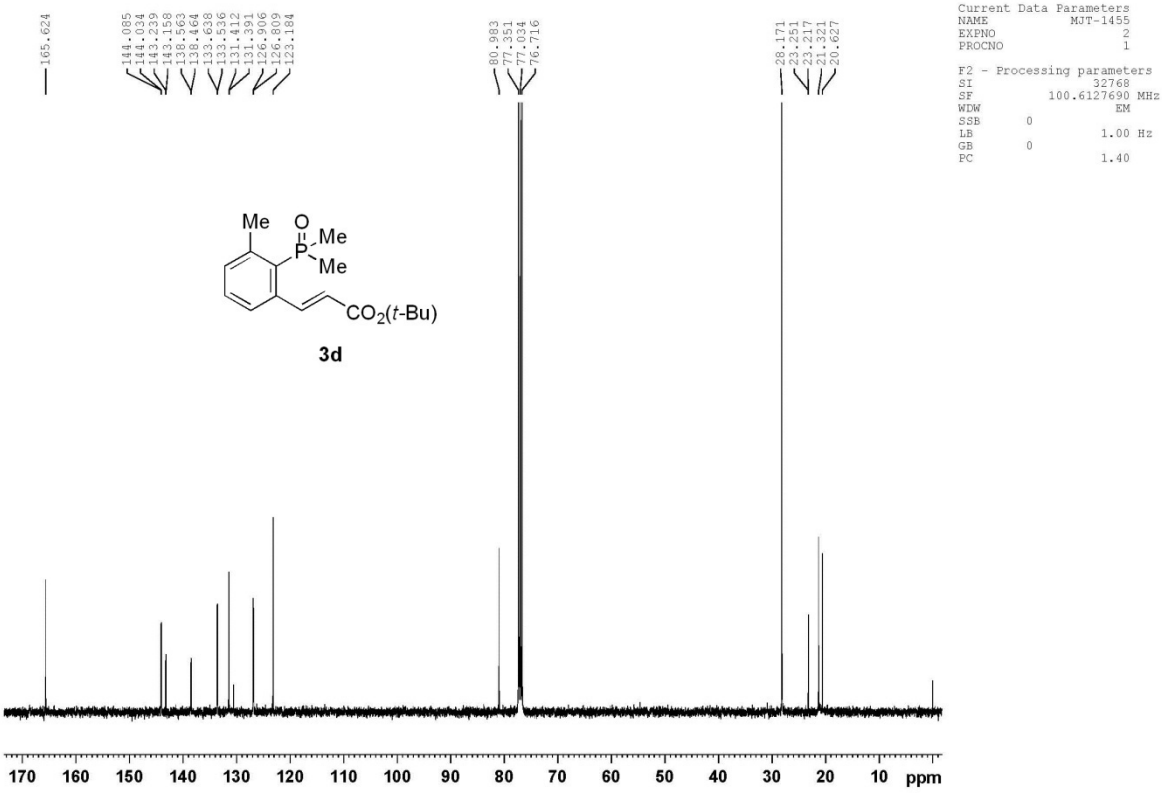

```
Current Data Parameters
NAME              LSJ-221-2
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20130128
Time                  17.10
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9845889 sec
RG                     74.34
DW                   60.800 usec
DE                    6.50 usec
TE                   298.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PLW1             12.60000038 W
SPO1            400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF               400.1300033 MHz
WDW                       EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```



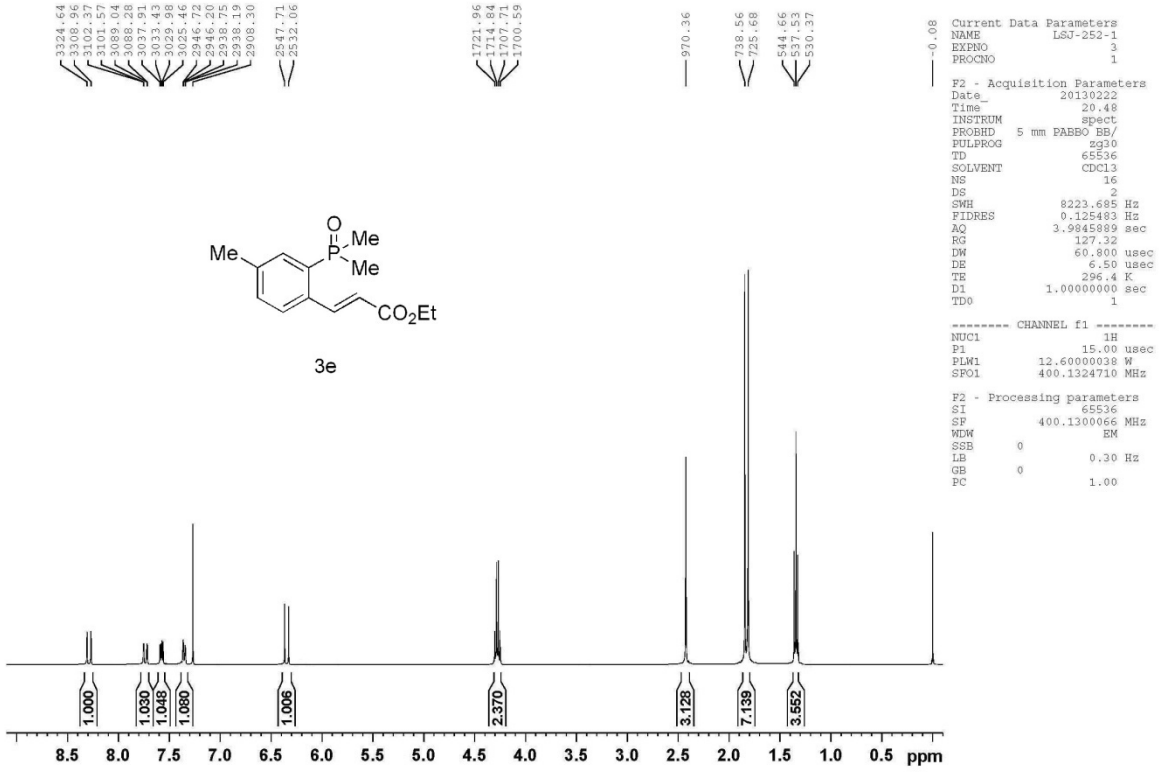

```
Current Data Parameters
NAME              LSJ-221-2
EXPNO                     3
PROCNO                    1

F2 - Processing parameters
SI                        32768
SF               100.6127690 MHz
WDW                       EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```



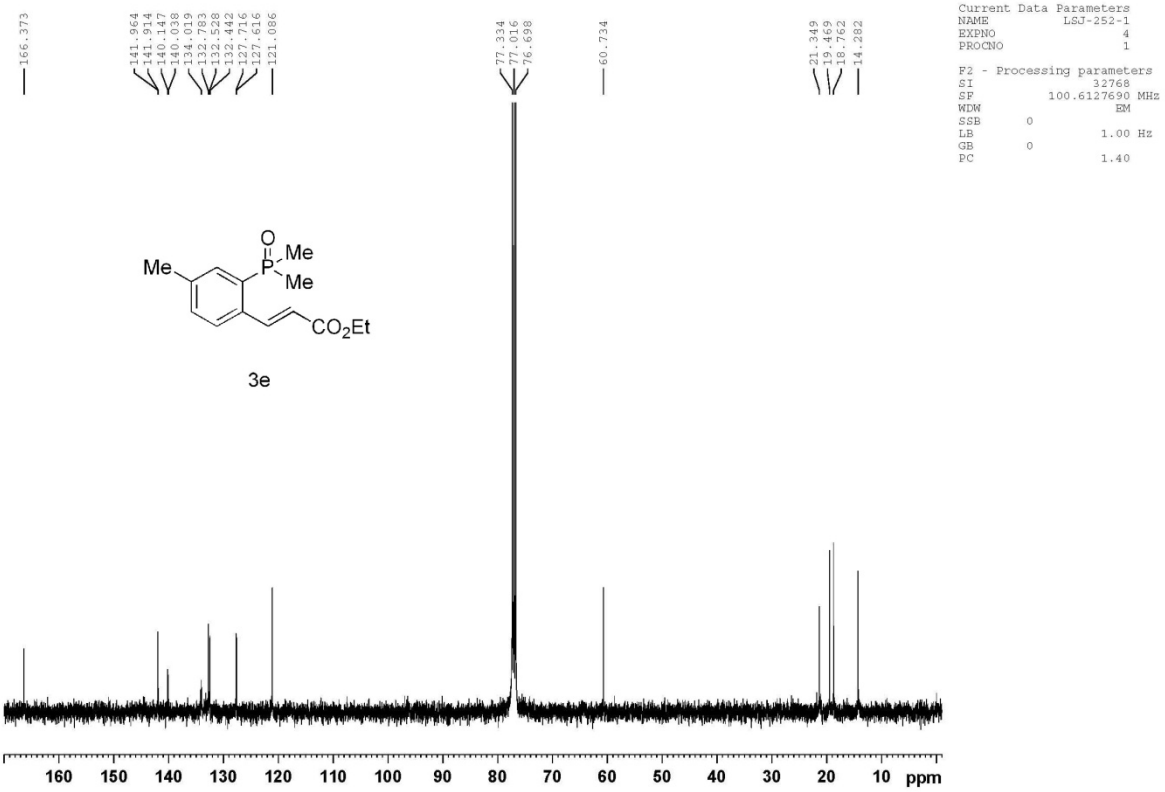

```
Current Data Parameters
NAME                  MJT-1455
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20130425
Time                   10.15
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES                0.125483 Hz
AQ                 3.9845889 sec
RG                     83.43
DW                    60.800 usec
DE                     6.50 usec
TE                    296.7 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.00 usec
PLW1               12.60000038 W
SFO1              400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF                400.1300044 MHz
WDW                         EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



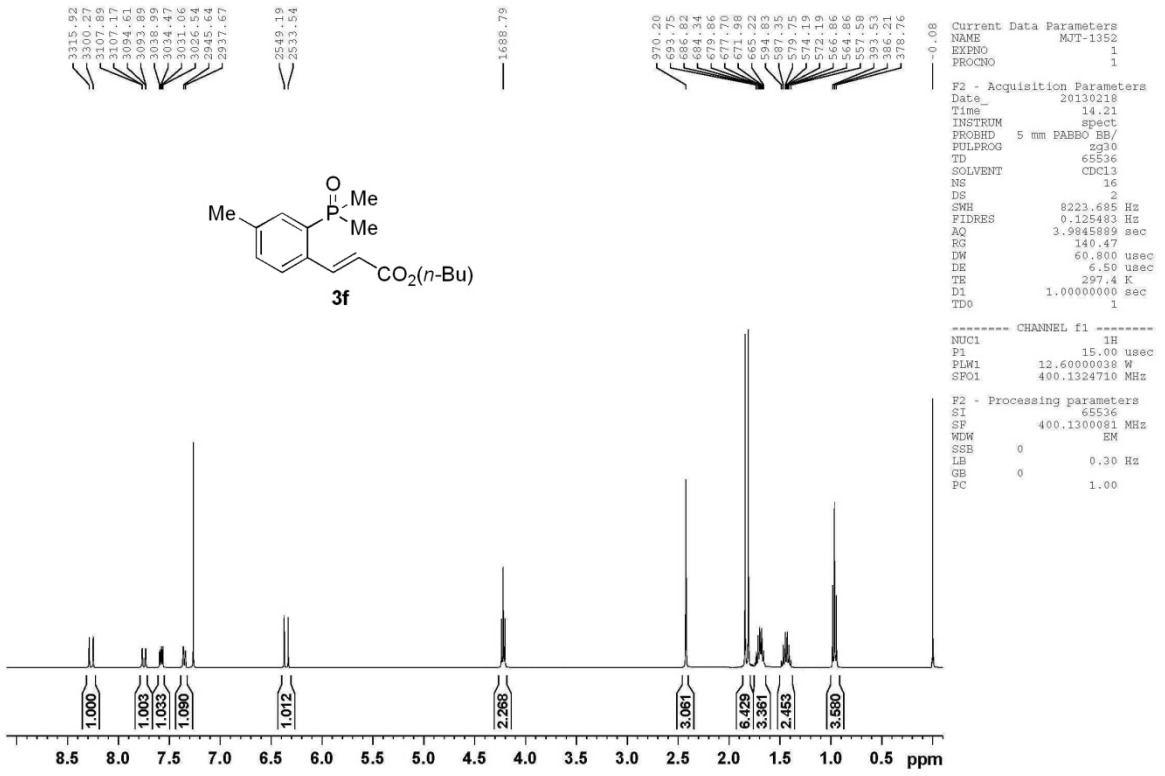

```
Current Data Parameters
NAME                  MJT-1455
EXPNO                        2
PROCNO                       1

F2 - Processing parameters
SI                        32768
SF                100.6127690 MHz
WDW                         EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

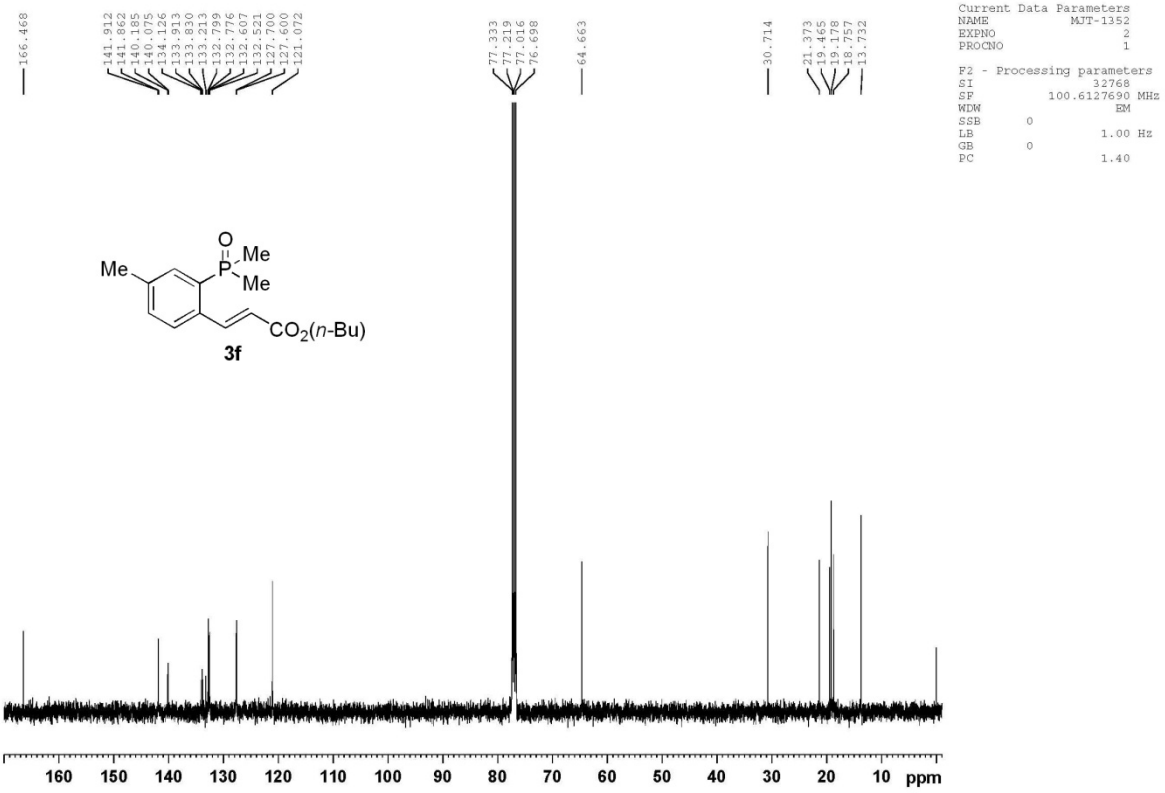


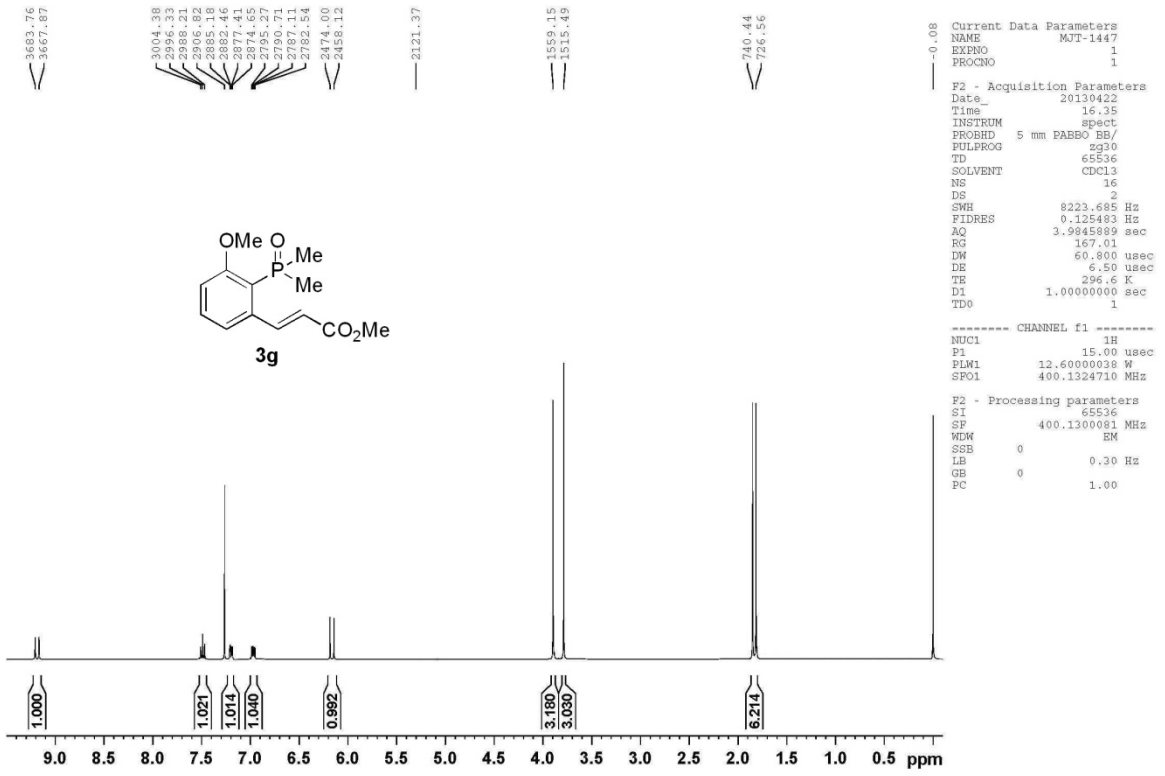

```
Current Data Parameters
NAME                  LSJ-252-1
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_               20130222
Time                    20.48
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9845889 sec
RG                     127.32
DW                     60.800 usec
DE                      6.50 usec
TE                     296.4 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.00 usec
PLW1               12.60000038 W
SPO1               400.1324710 MHz

F2 - Processing parameters
SI                         65536
SF                 400.1300066 MHz
WDW                           EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```

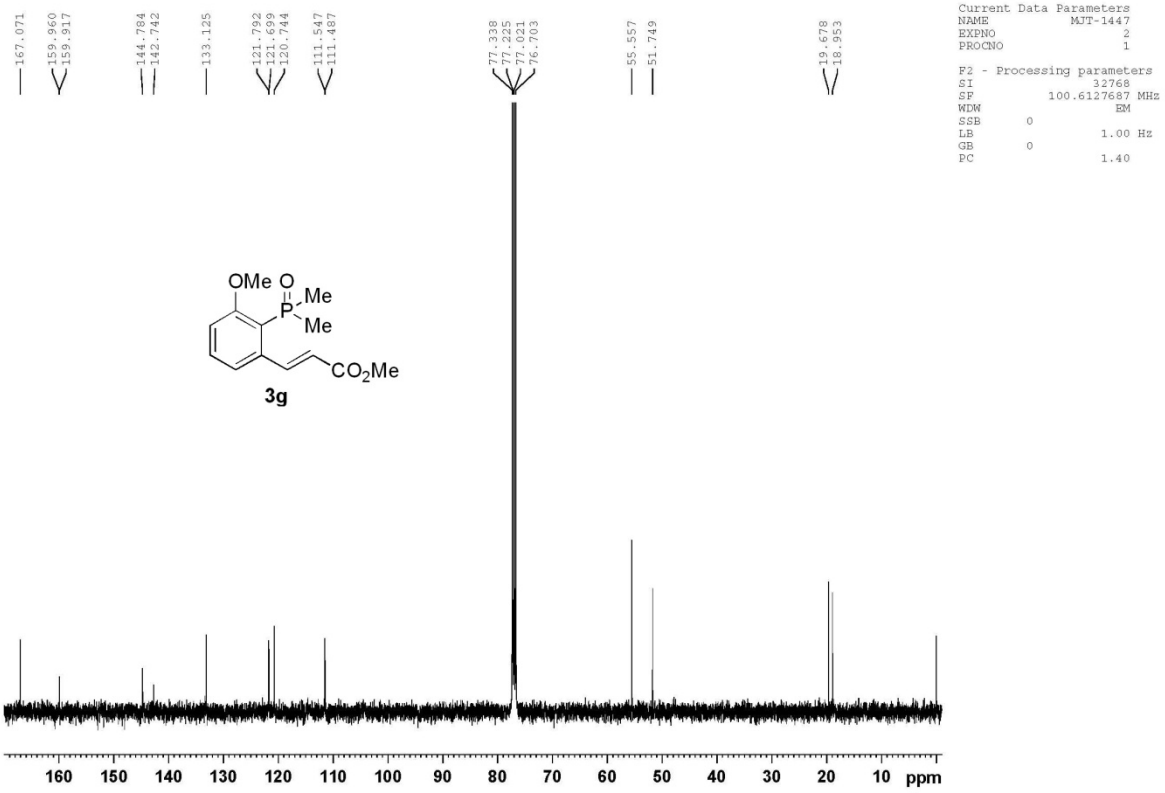


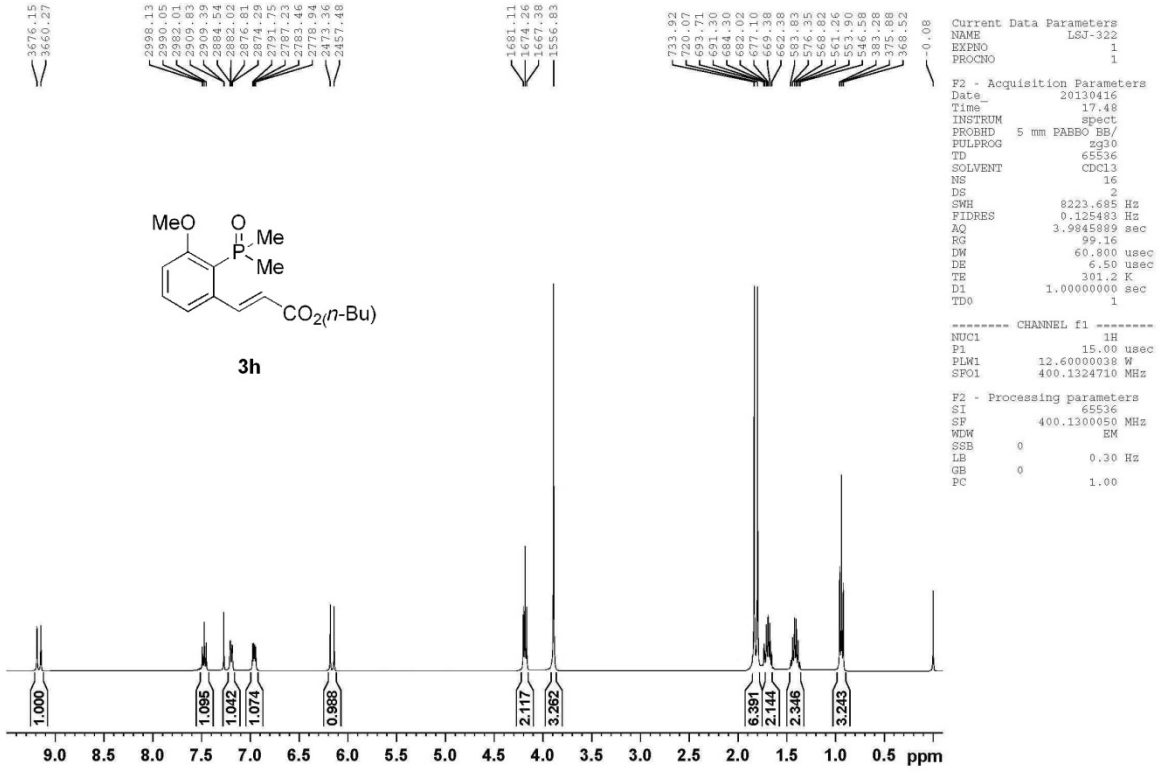

```
Current Data Parameters
NAME                  LSJ-252-1
EXPNO                         4
PROCNO                        1

F2 - Processing parameters
SI                         32768
SF                 100.6127690 MHz
WDW                           EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

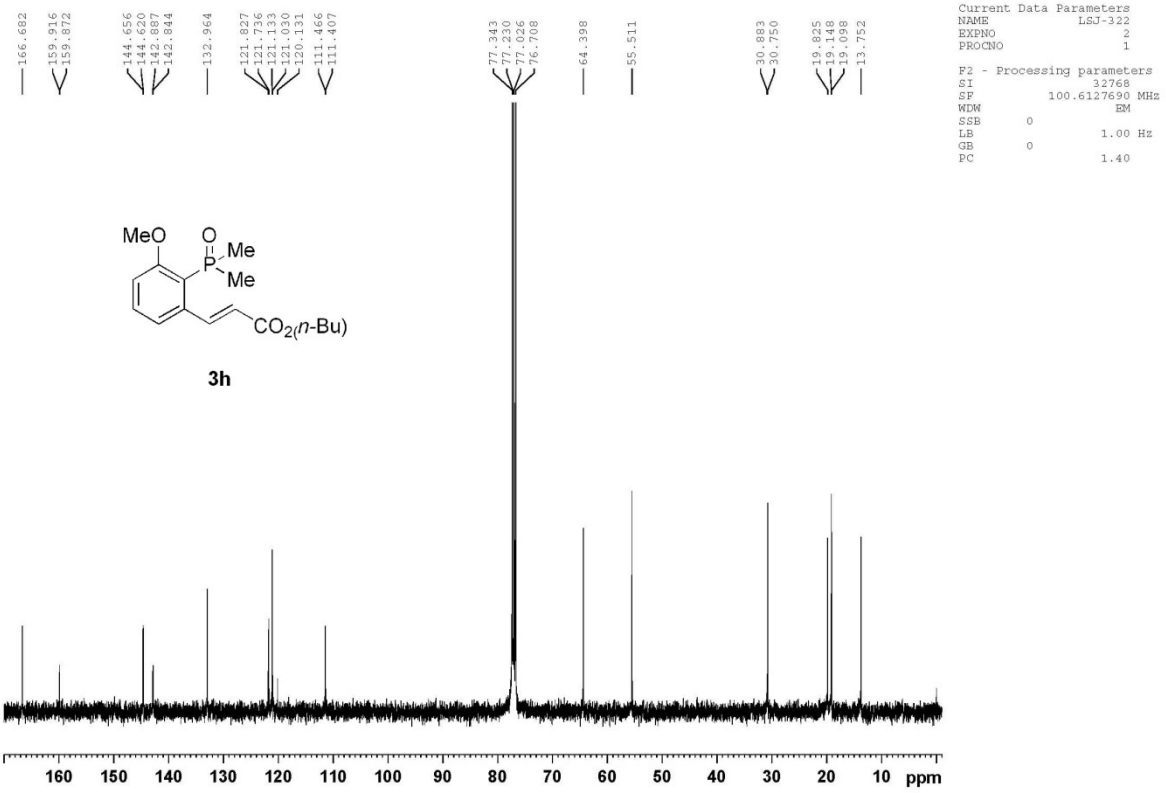

3315.92
3300.27
3107.89
3107.17
3094.61
3093.89
3038.99
3034.47
3031.06
3026.54
2945.64
2937.67
2549.19
2533.54
1688.79
970.20
693.75
686.82
684.34
679.86
677.70
671.98
665.22
594.83
587.35
579.75
574.19
572.19
566.86
564.86
557.58
393.53
386.21
378.76
-0.08
Current Data Parameters
NAME MJT-1352
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130218
Time 14.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 140.47
DW 60.800 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SPO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
O
P
Me
Me
CO2(n-Bu)
3f
1.000
1.003
1.033
1.090
1.012
2.268
3.061
6.429
3.361
2.453
3.580
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

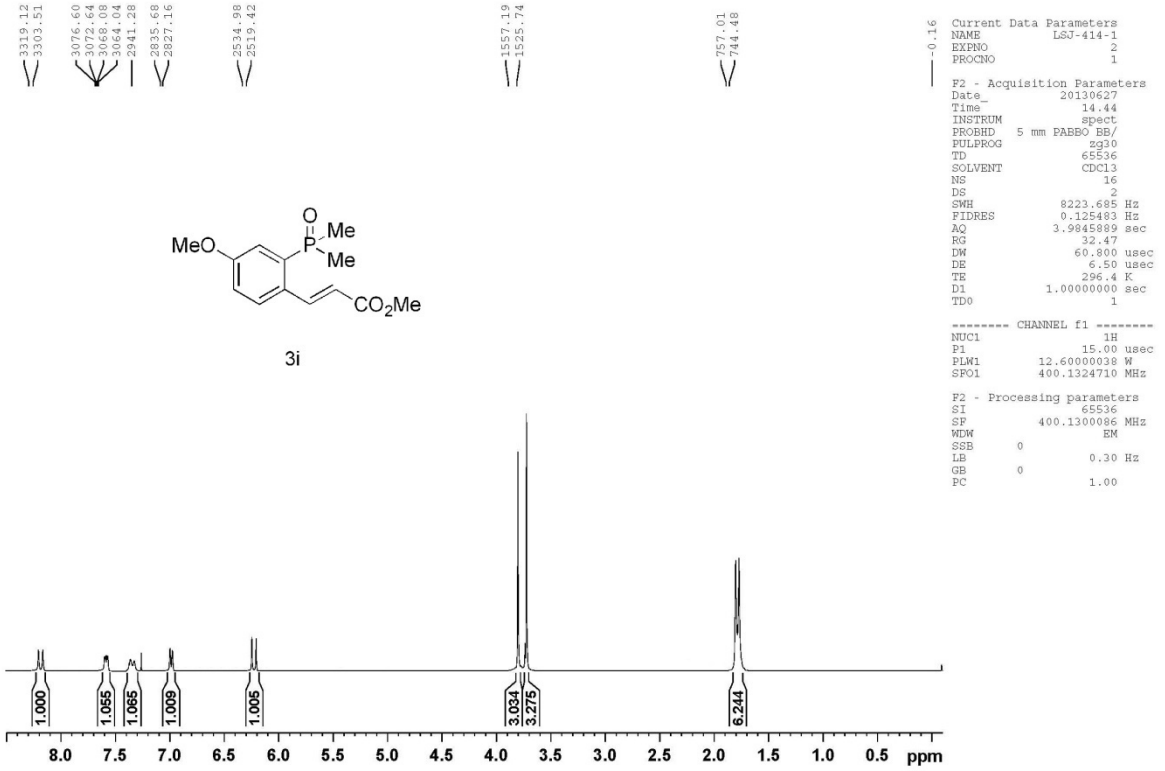

166.468
141.912
141.862
140.185
140.075
134.126
133.913
133.830
133.213
132.799
132.776
132.607
132.521
127.700
127.600
121.072
77.333
77.219
77.016
76.698
64.663
30.714
21.373
19.465
19.178
18.757
13.732
Current Data Parameters
NAME MJT-1352
EXPNO 2
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me
O
P
Me
Me
CO2(n-Bu)
3f
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


3683.76
3667.87
3004.38
2996.33
2988.21
2906.82
2885.18
2882.46
2877.41
2874.65
2795.27
2790.71
2787.11
2782.54
2474.00
2458.12
2121.37
1559.15
1515.49
740.44
726.56
-0.08
OMe O
Me
P
Me
CO2Me
3g
Current Data Parameters
NAME MJT-1447
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130422
Time 16.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 167.01
DW 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.000
1.021
1.014
1.040
0.992
3.180
3.030
6.214
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

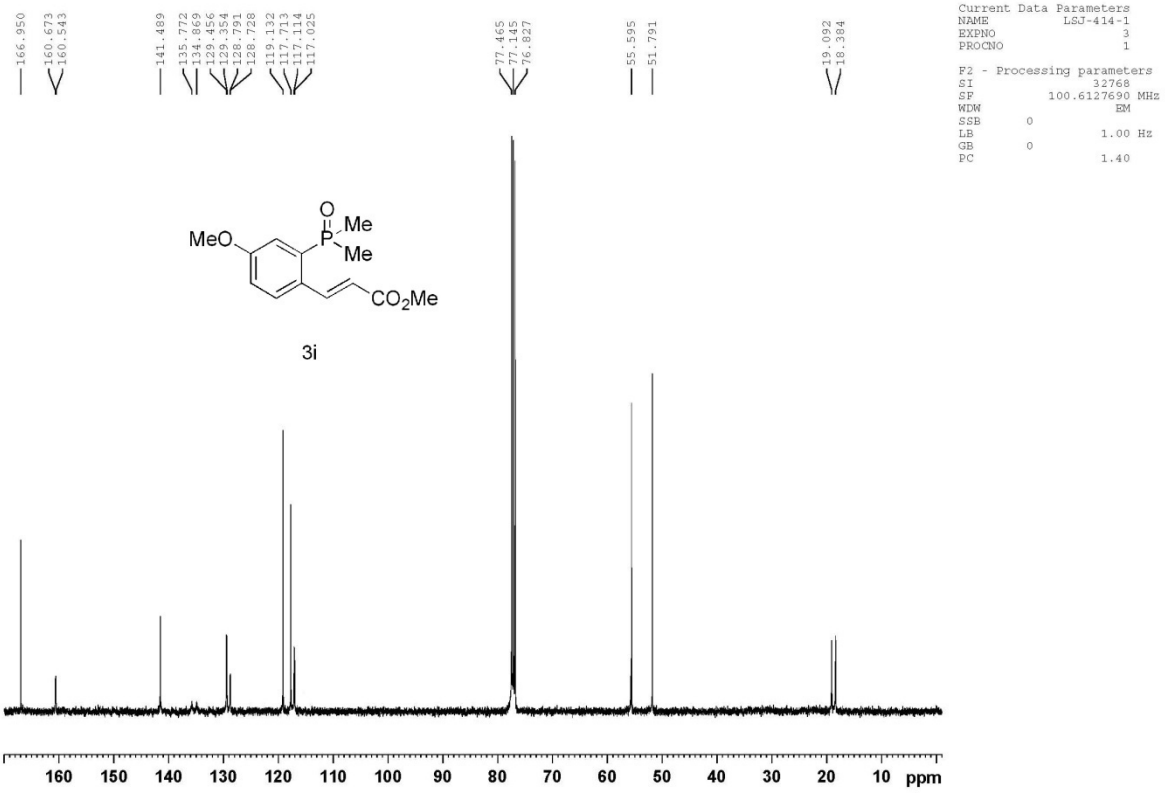

167.071
159.960
159.917
144.784
142.742
133.125
121.792
121.699
120.744
111.547
111.487
77.338
77.225
77.021
76.703
55.557
51.749
19.678
18.953
OMe O
Me
P
Me
CO2Me
3g
Current Data Parameters
NAME MJT-1447
EXPNO 2
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127687 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


3676.15
3660.27
2998.13
2990.05
2982.01
2909.83
2909.39
2884.54
2882.02
2876.81
2874.29
2791.75
2787.23
2783.46
2778.94
2747.36
2457.48
1681.11
1674.26
1667.38
1556.83
733.92
720.07
693.71
691.30
684.30
682.02
677.10
669.38
662.38
583.83
576.35
568.82
561.26
553.90
546.58
383.28
375.88
368.52
-0.08
MeO
O
P
Me
Me
CO2(n-Bu)
3h
Current Data Parameters
NAME LSJ-322
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130416
Time 17.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 99.16
DW 60.800 usec
DE 6.50 usec
TE 301.2 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SPO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.000
1.095
1.042
1.074
0.988
2.117
3.262
6.391
2.144
2.346
3.243
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

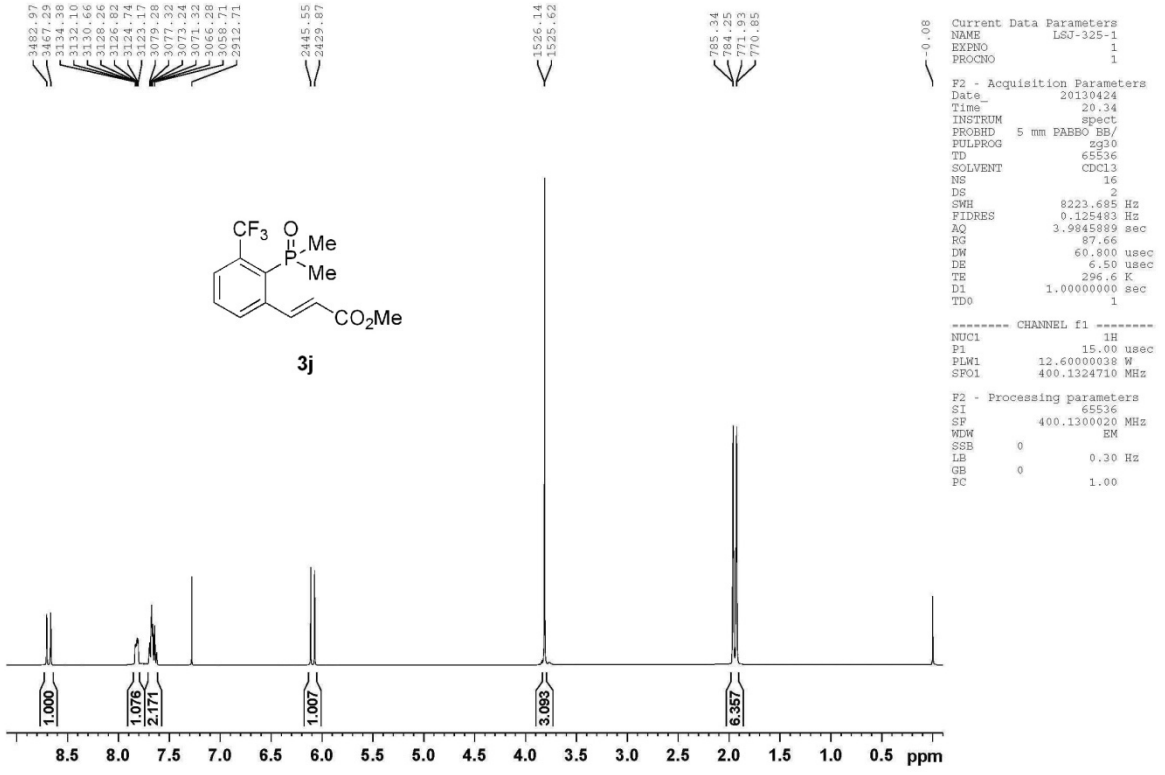

166.682
159.916
159.872
144.656
144.620
142.887
142.844
132.964
121.827
121.736
121.133
121.030
120.131
111.466
111.407
77.343
77.230
77.026
76.708
64.398
55.511
30.883
30.750
19.825
19.148
19.098
13.752
MeO
O
P
Me
Me
CO2(n-Bu)
3h
Current Data Parameters
NAME LSJ-322
EXPNO 2
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

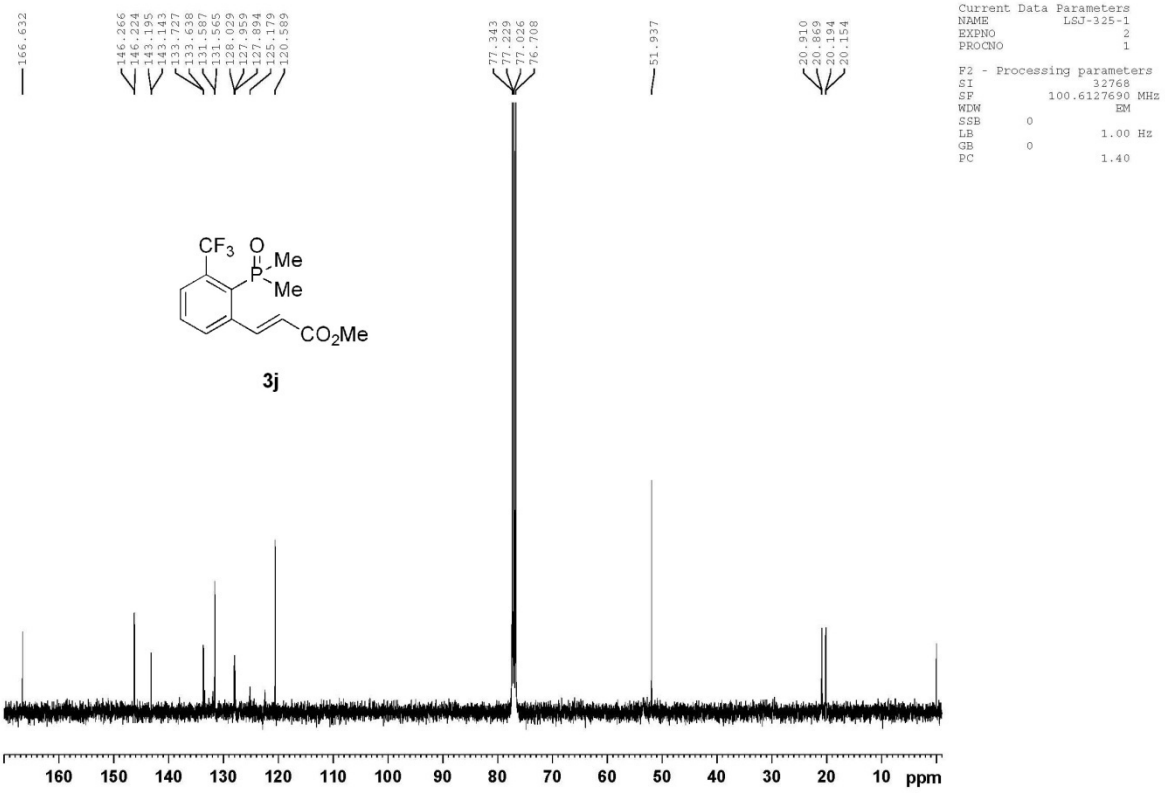


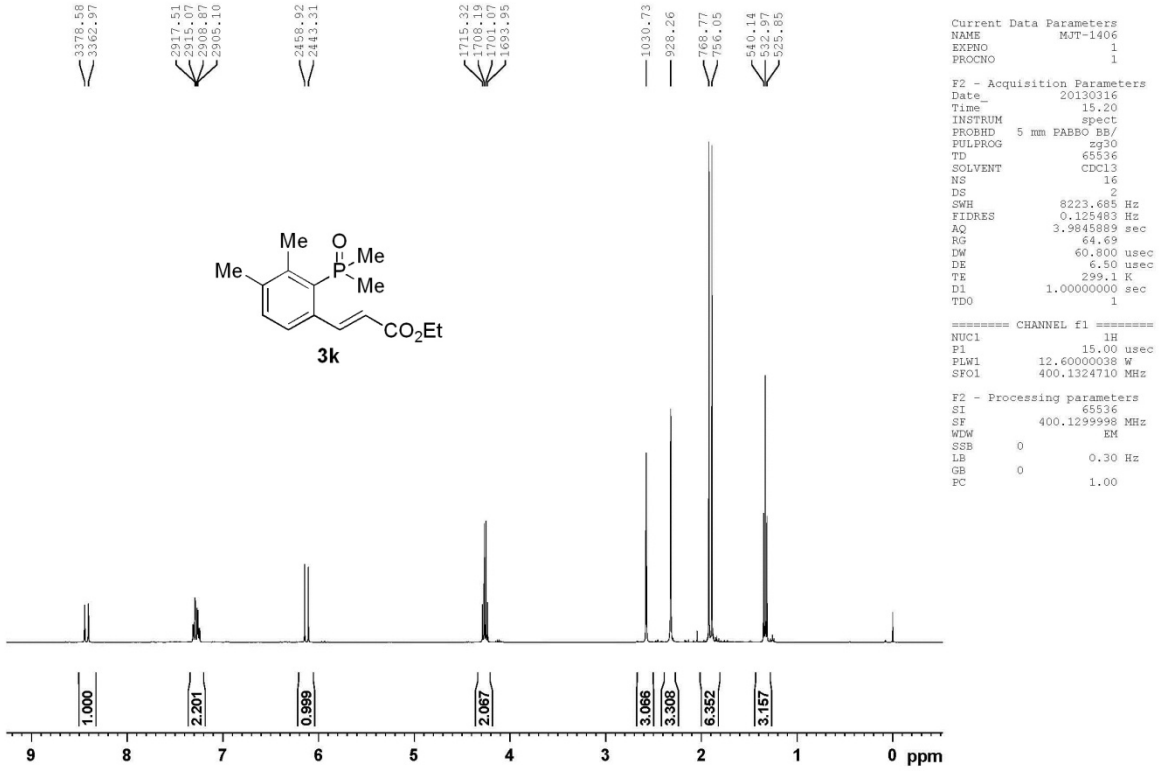

```
Current Data Parameters
NAME                  LSJ-414-1
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_               20130627
Time                    14.44
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9845889 sec
RG                       32.47
DW                      60.800 usec
DE                       6.50 usec
TE                      296.4 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        1H
P1                       15.00 usec
PLW1                12.60000038 W
SPO1                400.1324710 MHz

F2 - Processing parameters
SI                          65536
SF                  400.1300086 MHz
WDW                            EM
SSB                 0
LB                           0.30 Hz
GB                  0
PC                           1.00
```



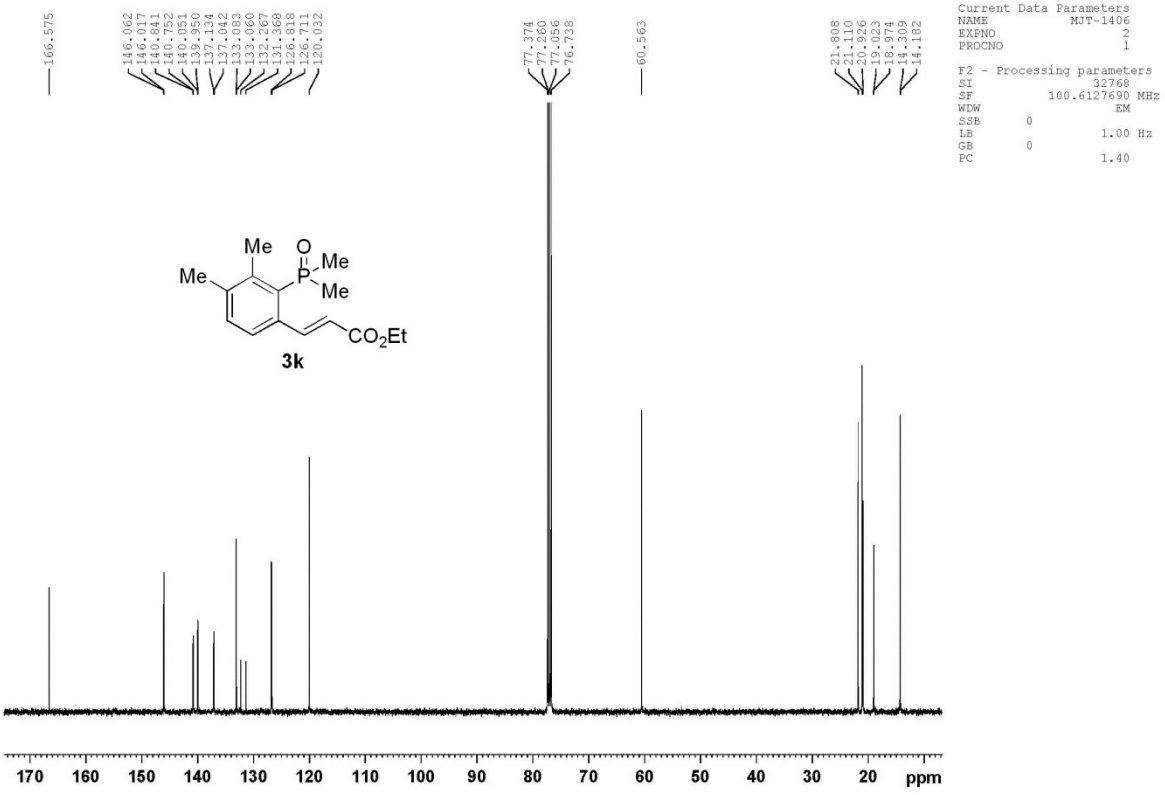

```
Current Data Parameters
NAME                  LSJ-414-1
EXPNO                         3
PROCNO                        1

F2 - Processing parameters
SI                          32768
SF                  100.6127690 MHz
WDW                            EM
SSB                 0
LB                           1.00 Hz
GB                  0
PC                           1.40
```


3j
Current Data Parameters
NAME LSJ-325-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130424
Time 20.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 87.66
DW 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SPO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300020 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.000
1.076
2.171
1.007
3.093
6.357
ppm

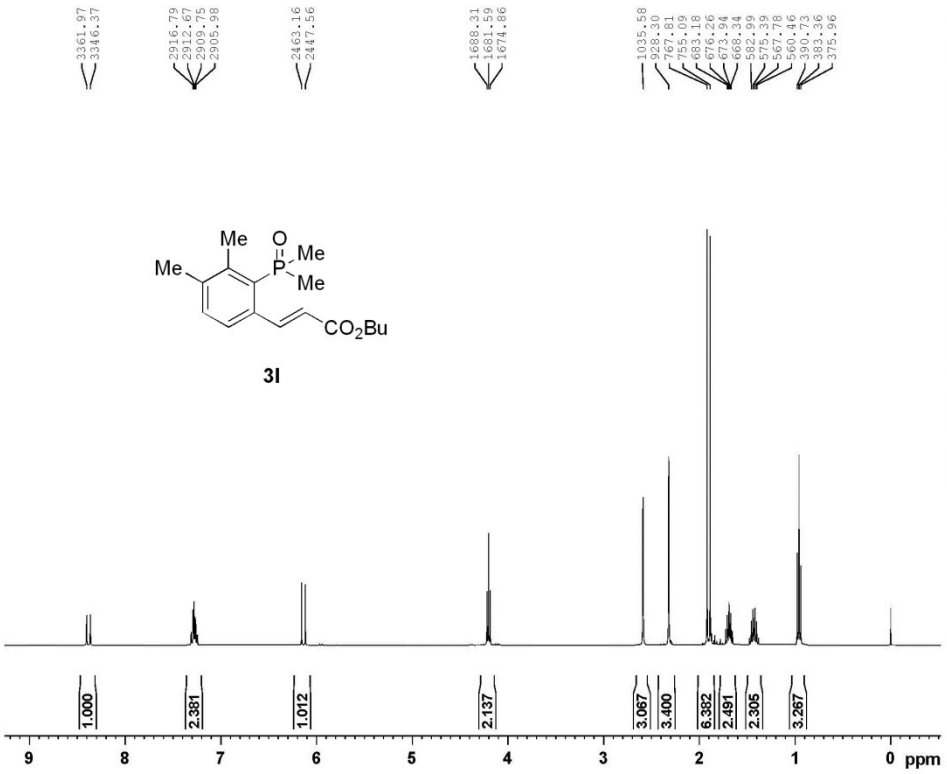

3j
Current Data Parameters
NAME LSJ-325-1
EXPNO 2
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm



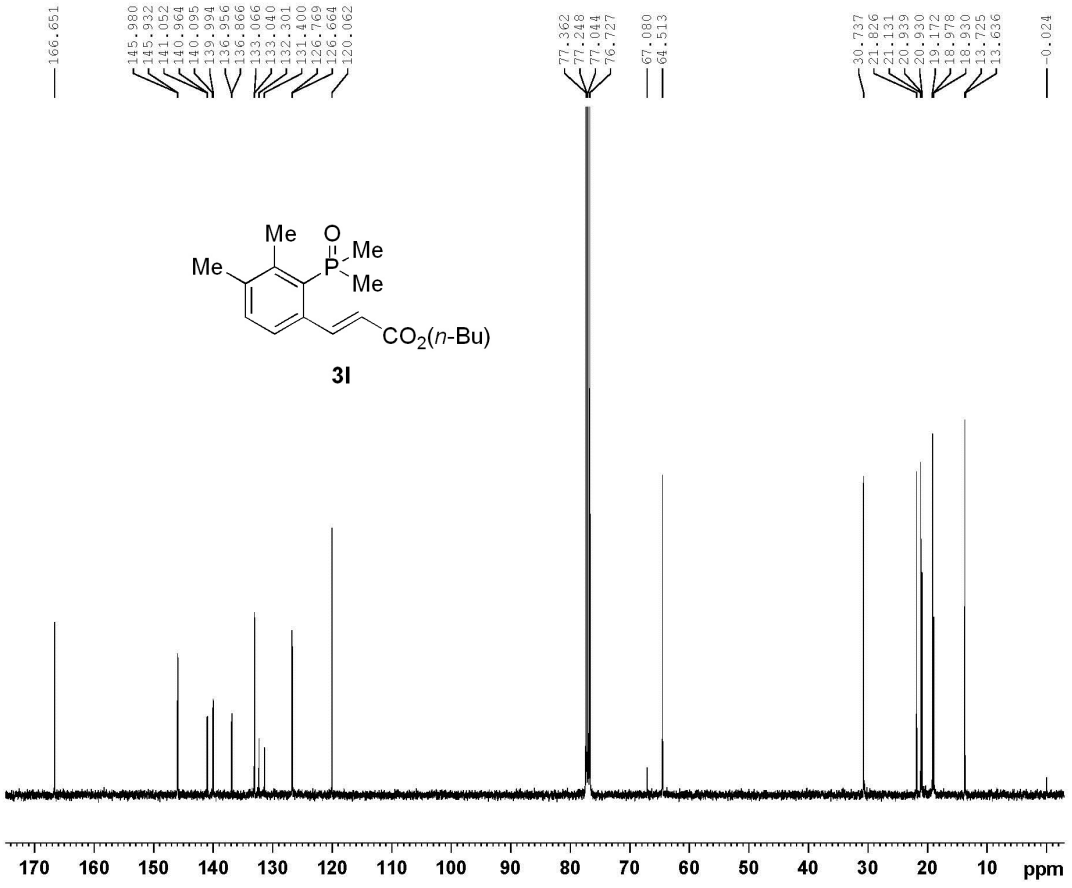

```
Current Data Parameters
NAME                  MJT-1406
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20130316
Time                   15.20
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9845889 sec
RG                     64.69
DW                    60.800 usec
DE                     6.50 usec
TE                   299.1 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.00 usec
PLW1              12.60000038 W
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF               400.1299998 MHz
WDW                      EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



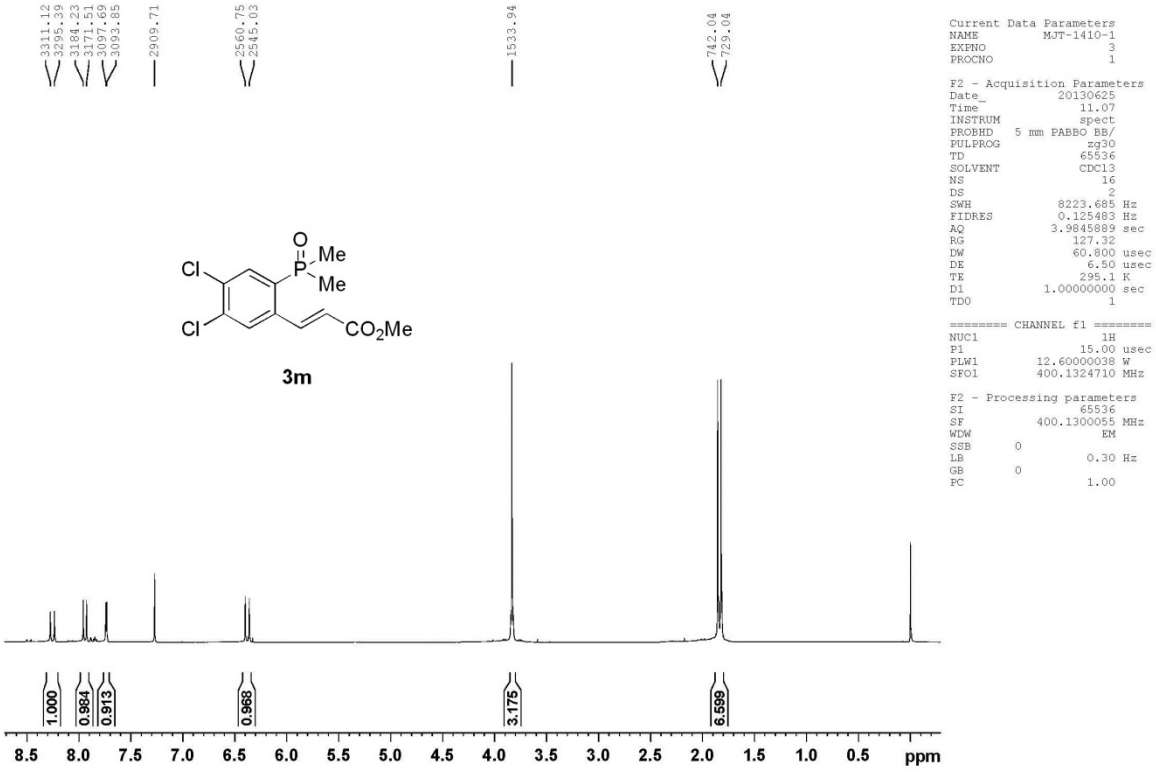

```
Current Data Parameters
NAME                  MJT-1406
EXPNO                        2
PROCNO                       1

F2 - Processing parameters
SI                    32768
SF               100.6127690 MHz
WDW                      EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```



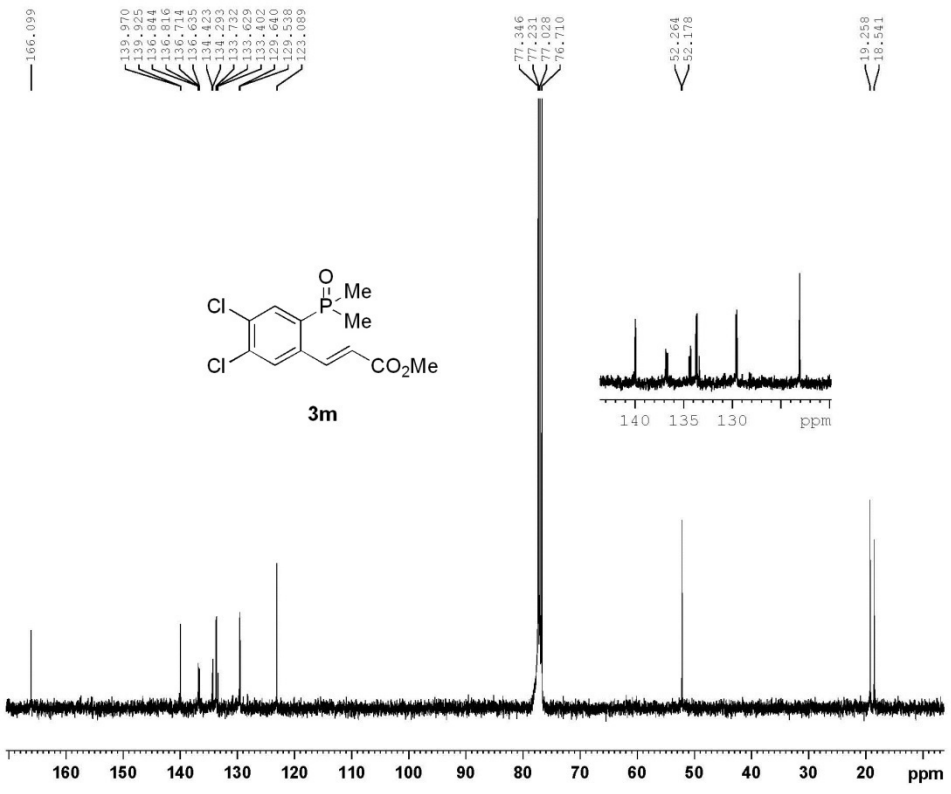

```
Current Data Parameters
NAME                  MJT-1405
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20130316
Time                   15.15
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9845889 sec
RG                    64.69
DW                   60.800 usec
DE                    6.50 usec
TE                   299.1 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PLW1             12.60000038 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF               400.1300022 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                    1.00
```



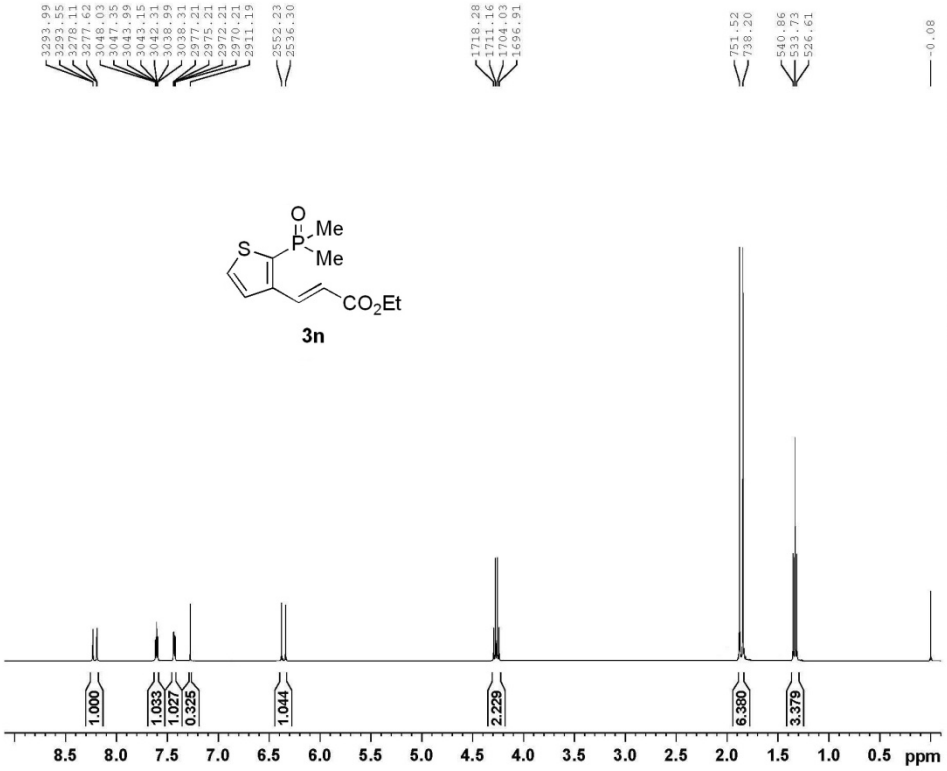

```
Current Data Parameters
NAME                  MJT-1405
EXPNO                        2
PROCNO                       1

F2 - Processing parameters
SI                        32768
SF               100.6127690 MHz
WDW                      EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```



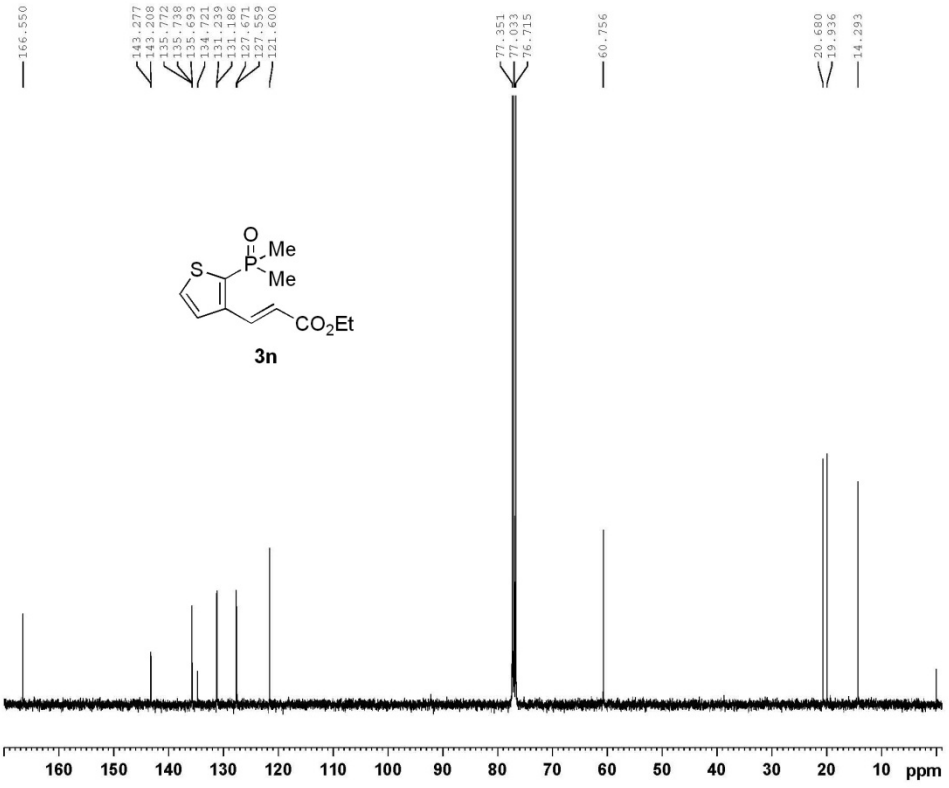

```
Current Data Parameters
NAME            MJT-1410-1
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_            20130625
Time                11.07
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9845889 sec
RG                 127.32
DW                 60.800 usec
DE                  6.50 usec
TE                 295.1 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PLW1          12.60000038 W
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF            400.1300055 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



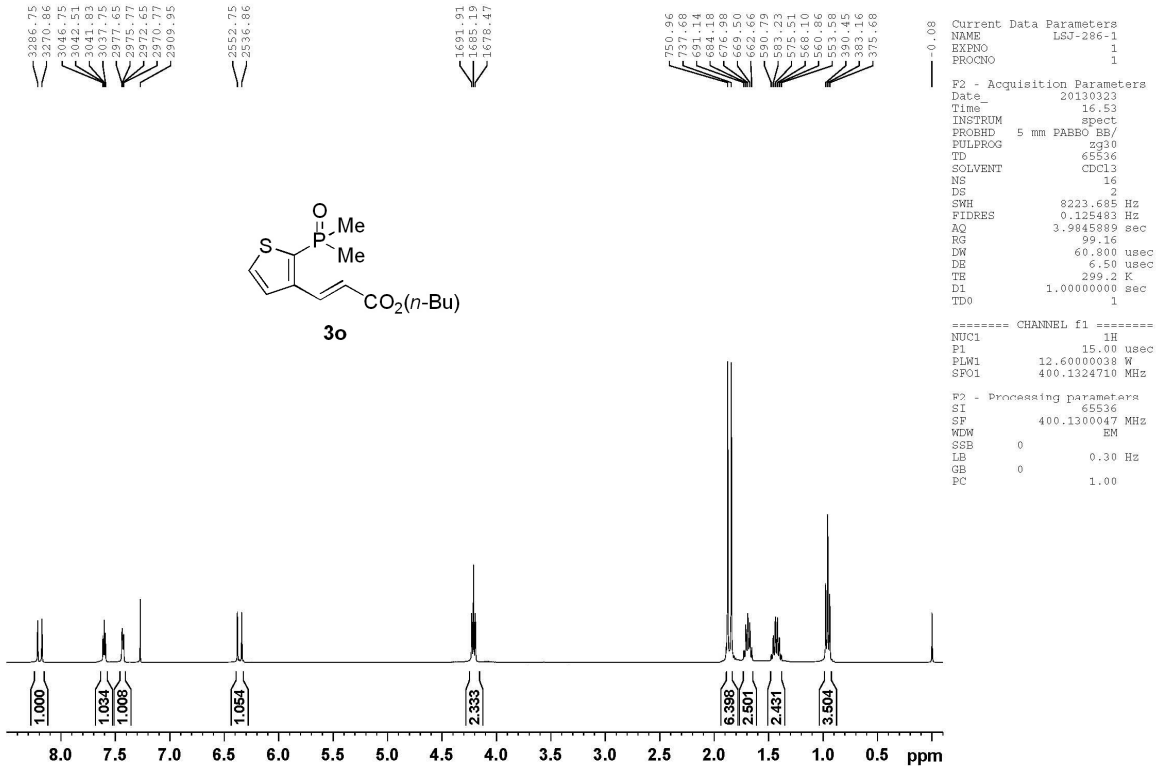

```
Current Data Parameters
NAME            MJT-1410-1
EXPNO                    5
PROCNO                   1

F2 - Processing parameters
SI                 32768
SF            100.6127690 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



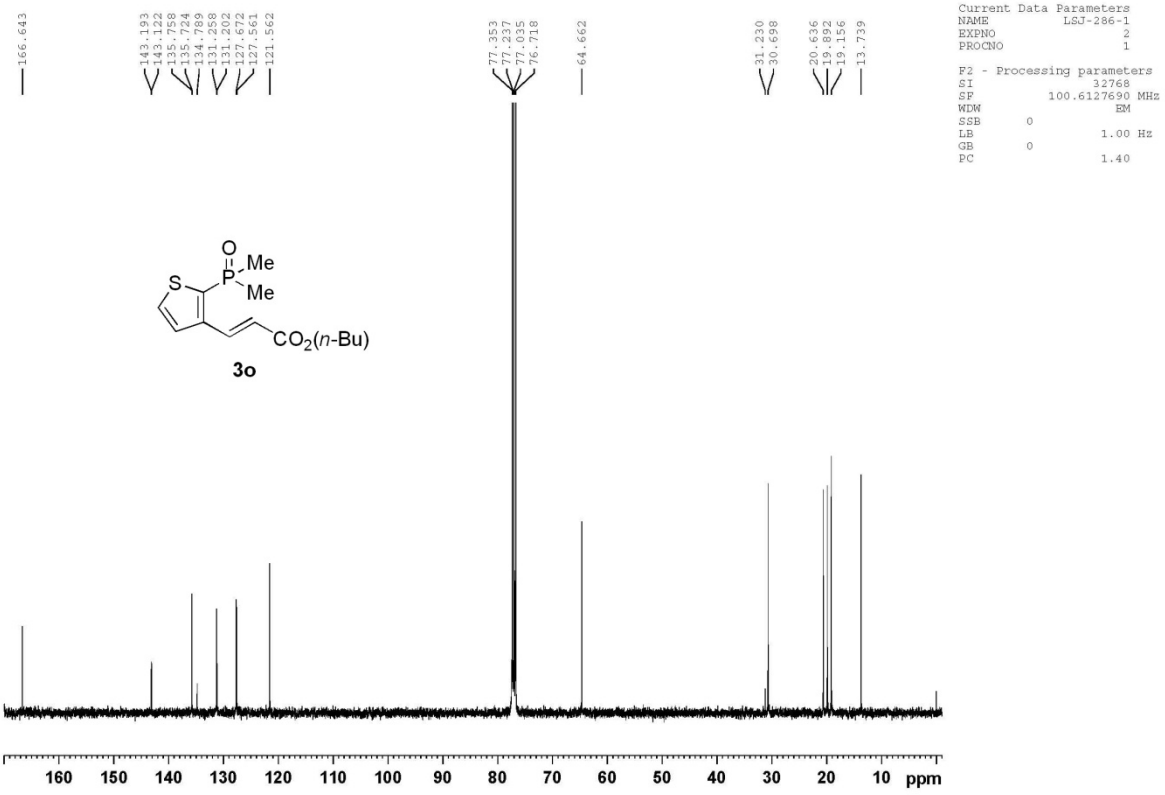

```
Current Data Parameters
NAME                 LSJ-287-1
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20130323
Time                   17.04
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9845889 sec
RG                     99.16
DW                    60.800 usec
DE                     6.50 usec
TE                    299.3 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.00 usec
PLW1               12.60000038 W
SFO1              400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF                400.1300035 MHz
WDW                          EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```



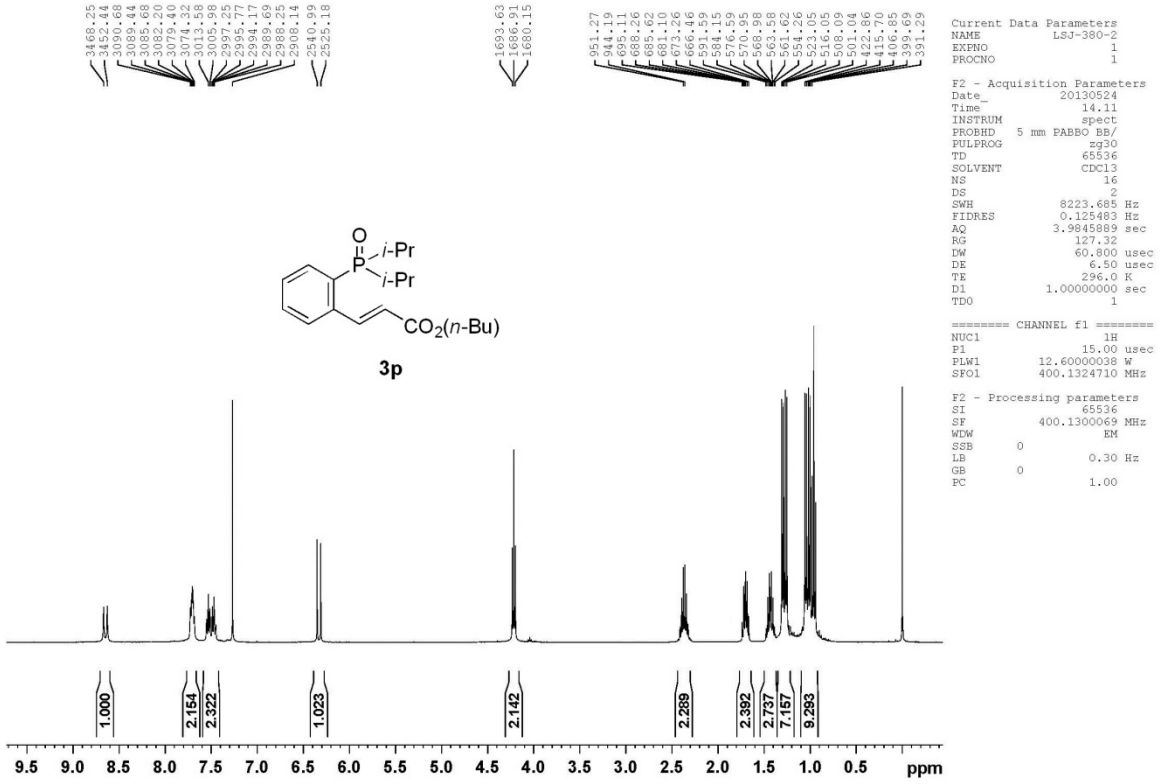

```
Current Data Parameters
NAME                 LSJ-287-1
EXPNO                        2
PROCNO                       1

F2 - Processing parameters
SI                        32768
SF                100.6127690 MHz
WDW                          EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
```



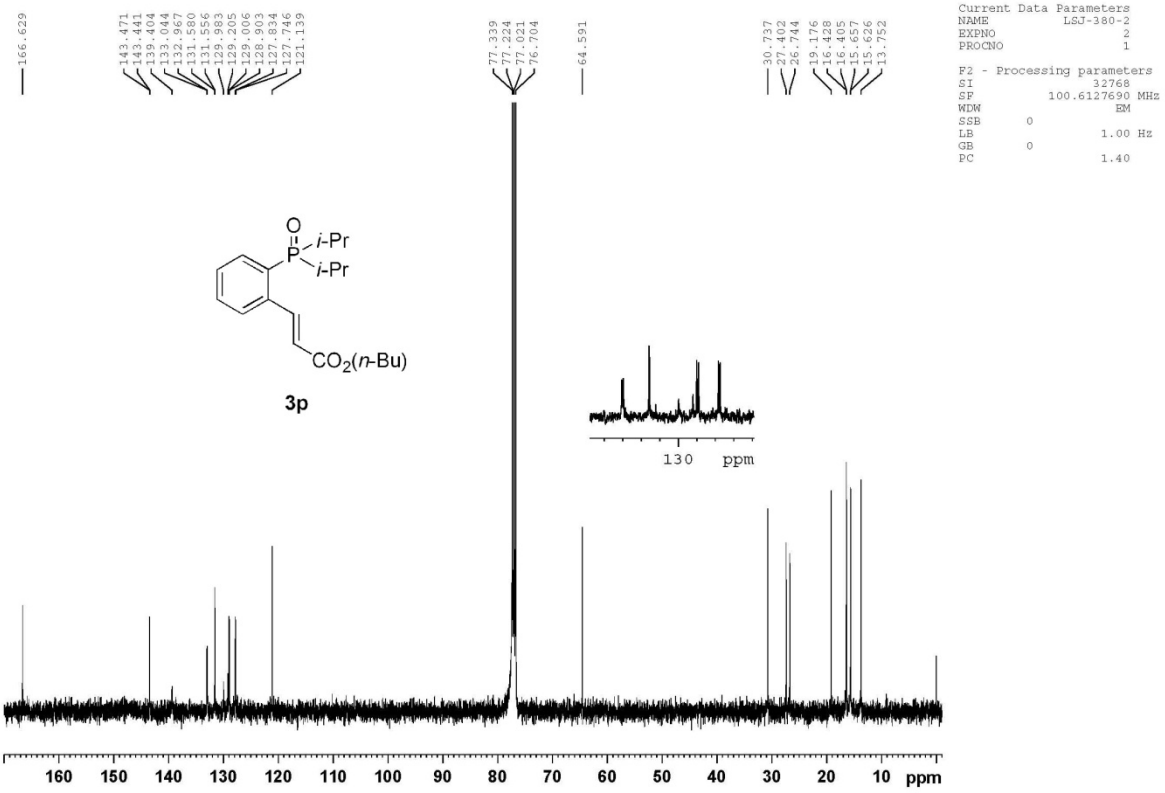

```
Current Data Parameters
NAME                  LSJ-286-1
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_               20130323
Time                   16.53
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9845889 sec
RG                     99.16
DW                    60.800 usec
DE                     6.50 usec
TE                    299.2 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PLW1              12.60000038 W
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF               400.1300047 MHz
WDW                      EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```



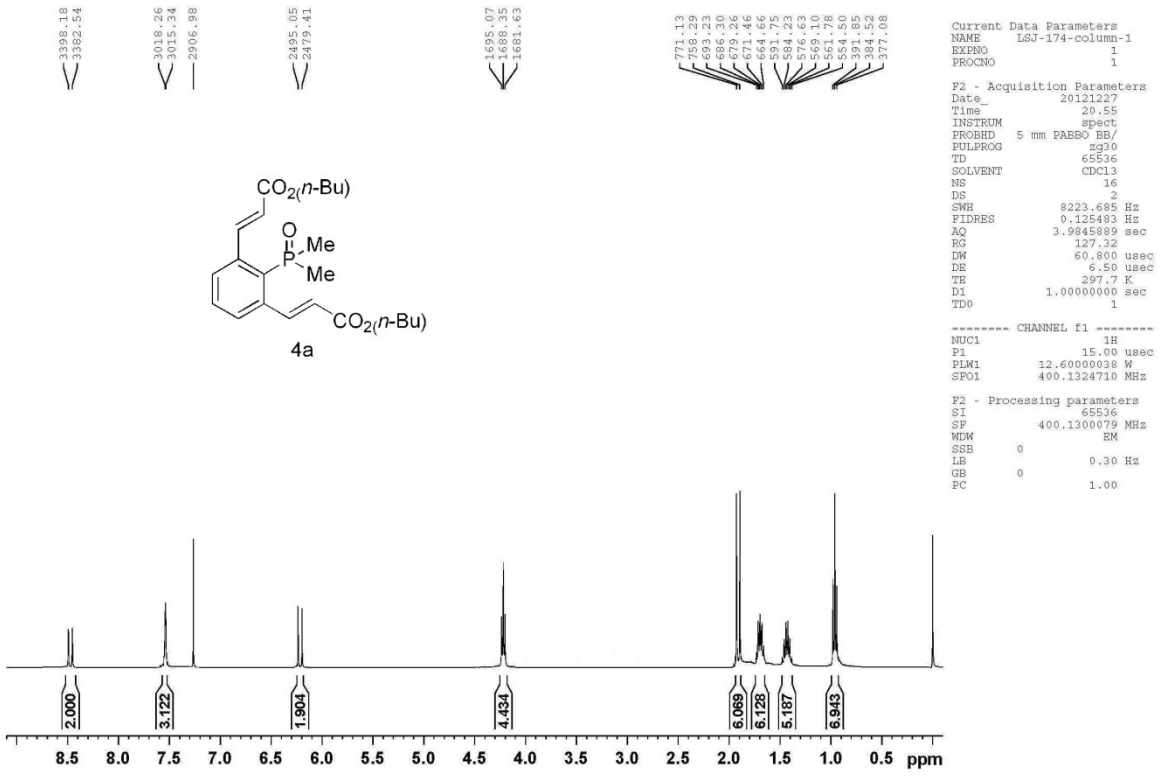

```
Current Data Parameters
NAME                  LSJ-286-1
EXPNO                         2
PROCNO                        1

F2 - Processing parameters
SI                        32768
SF               100.6127690 MHz
WDW                      EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

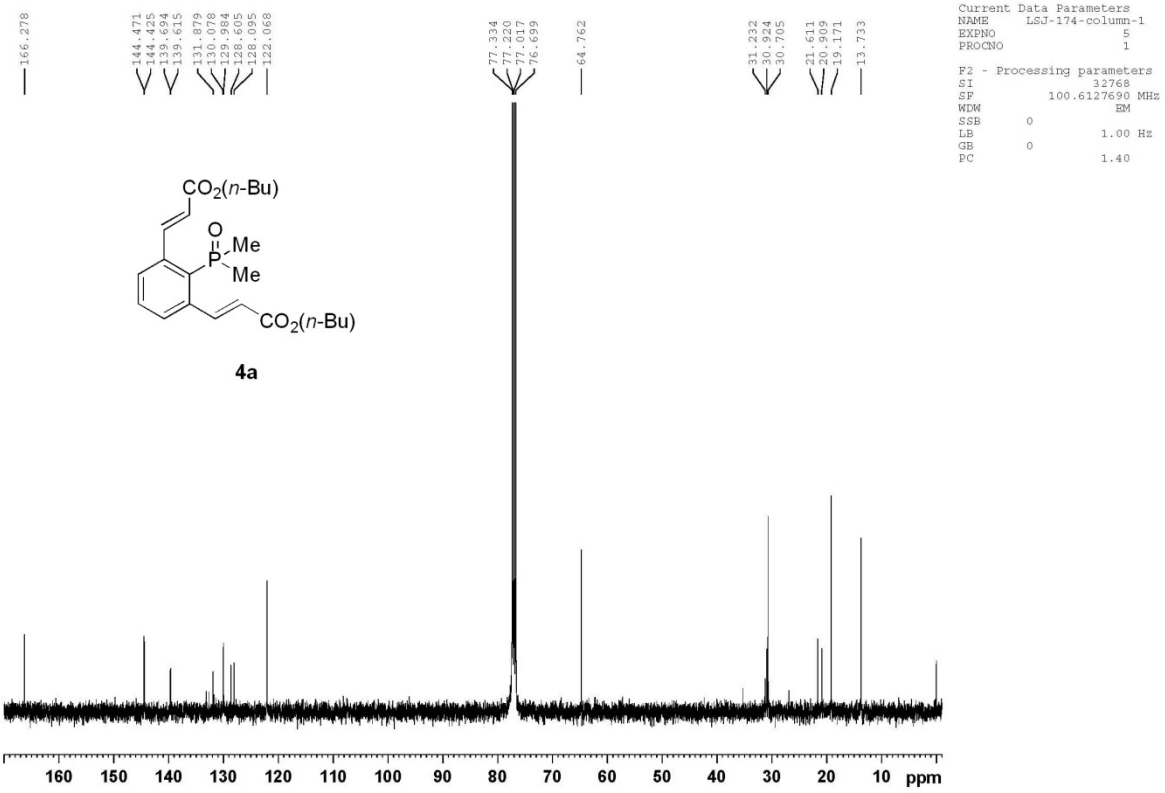


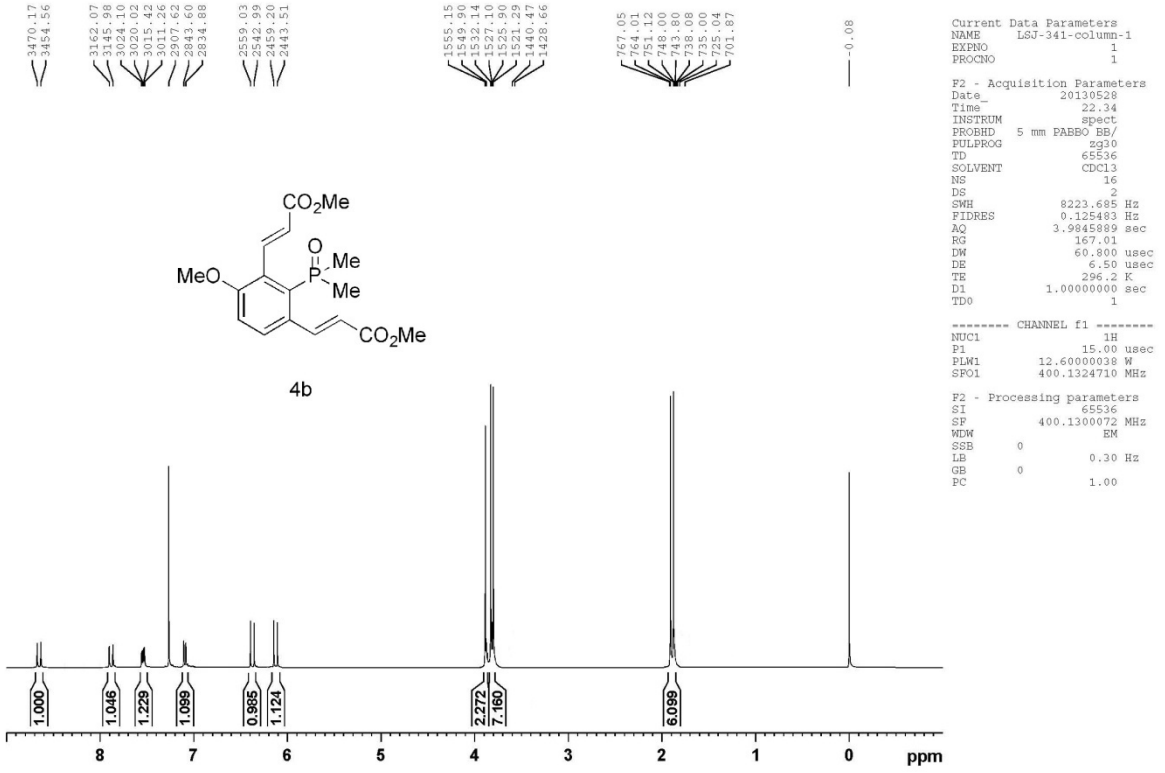

```
Current Data Parameters
NAME              LSJ-380-2
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20130524
Time                  14.11
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9845889 sec
RG                   127.32
DW                   60.800 usec
DE                    6.50 usec
TE                   296.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    15.00 usec
PLW1             12.60000038 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF               400.1300069 MHz
WDW                      EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```

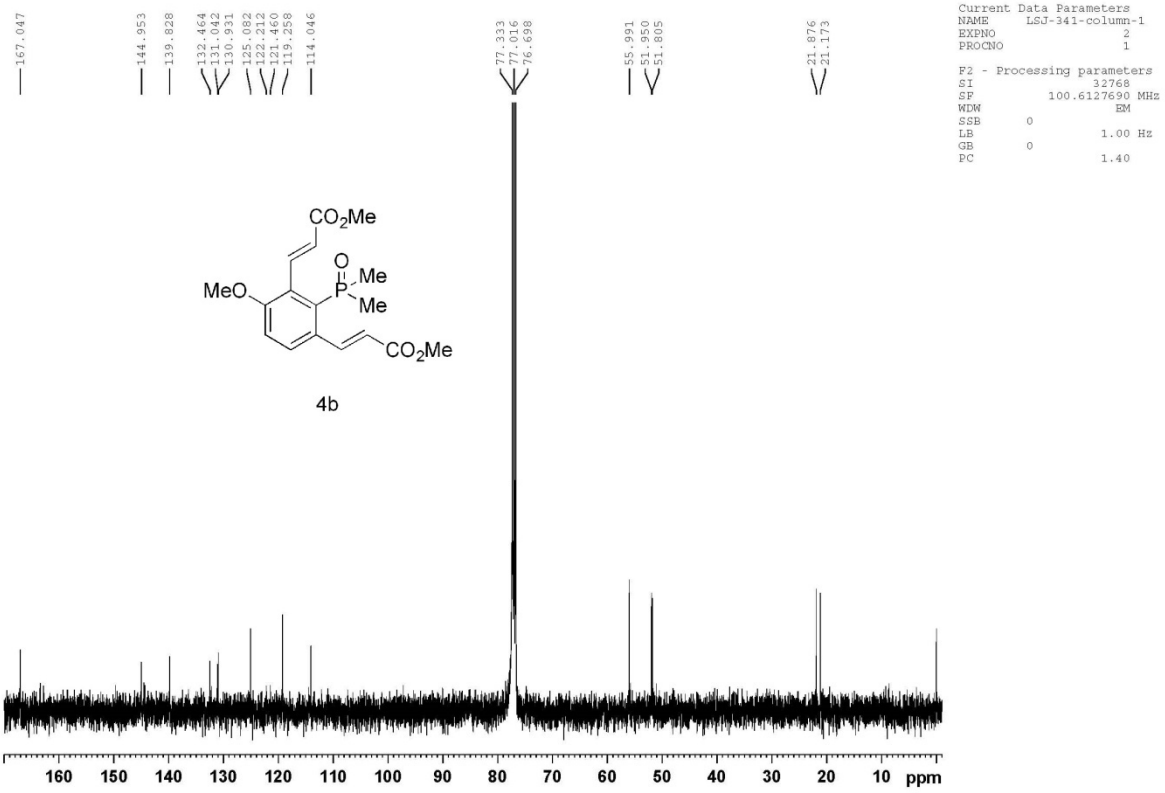


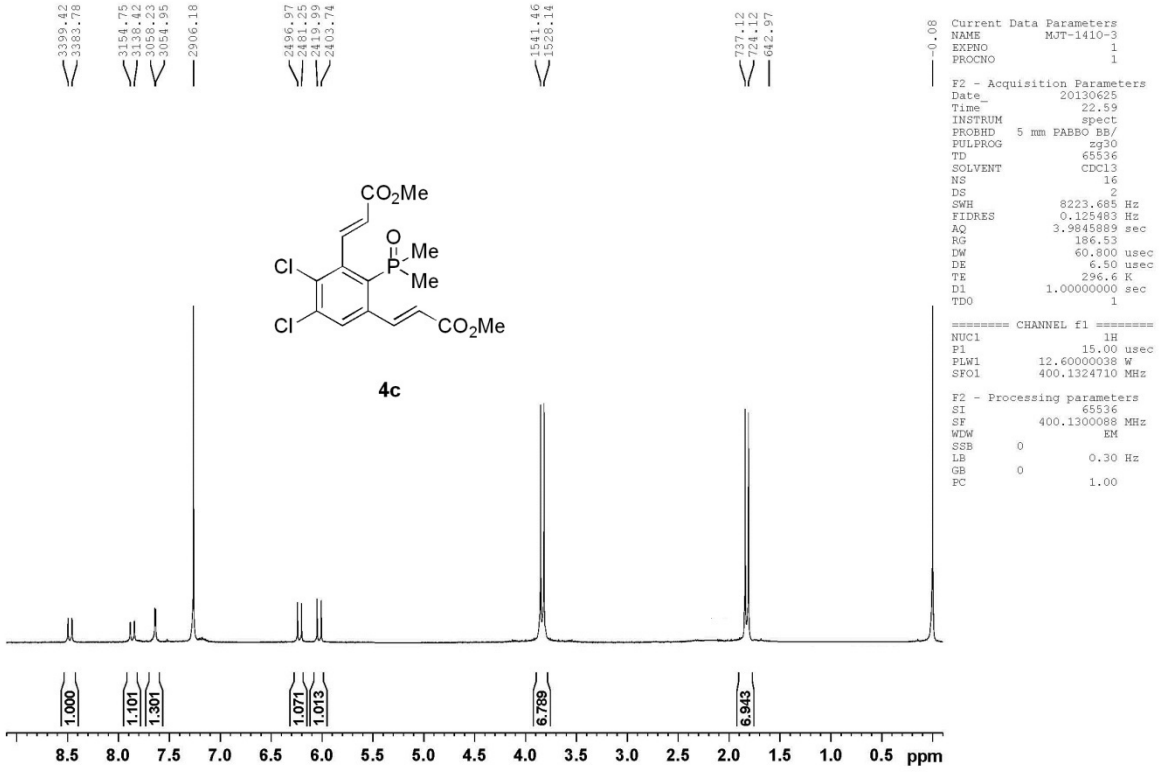

```
Current Data Parameters
NAME              LSJ-380-2
EXPNO                     2
PROCNO                    1

F2 - Processing parameters
SI                    32768
SF               100.6127690 MHz
WDW                      EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```

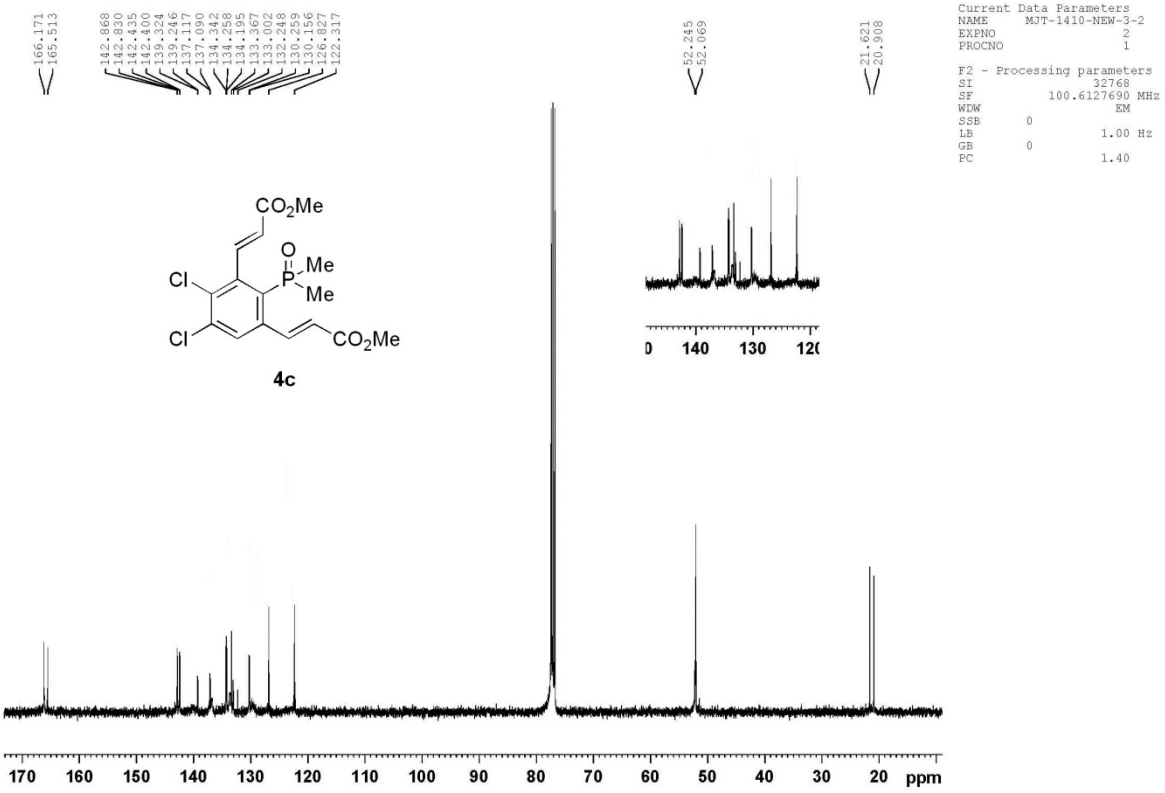

3398.18
3382.54
3018.26
3015.34
2906.98
2495.05
2479.41
1695.07
1688.35
1681.63
771.13
758.29
693.23
686.30
679.26
671.46
664.66
591.75
584.23
576.43
569.10
561.78
554.50
391.85
384.52
377.08
CO2(n-Bu)
O
P
Me
Me
CO2(n-Bu)
4a
Current Data Parameters
NAME LSJ-174-column-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121227
Time 20.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 127.32
DW 60.800 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SPO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300079 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.000
3.122
1.904
4.434
6.069
6.128
5.187
6.943
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

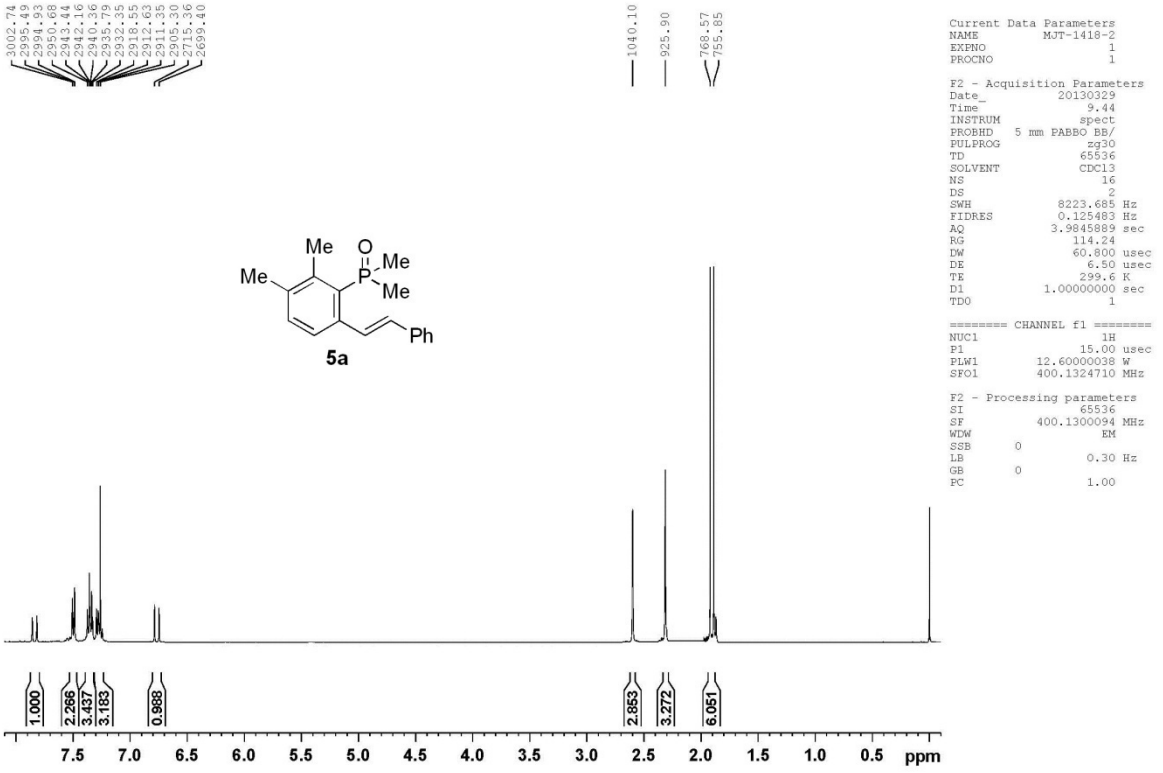

166.278
144.471
144.425
139.694
139.615
131.879
130.078
129.984
128.605
128.095
122.068
77.334
77.220
77.017
76.699
64.762
31.232
30.924
30.705
21.611
20.909
19.171
13.733
CO2(n-Bu)
O
P
Me
Me
CO2(n-Bu)
4a
Current Data Parameters
NAME LSJ-174-column-1
EXPNO 5
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

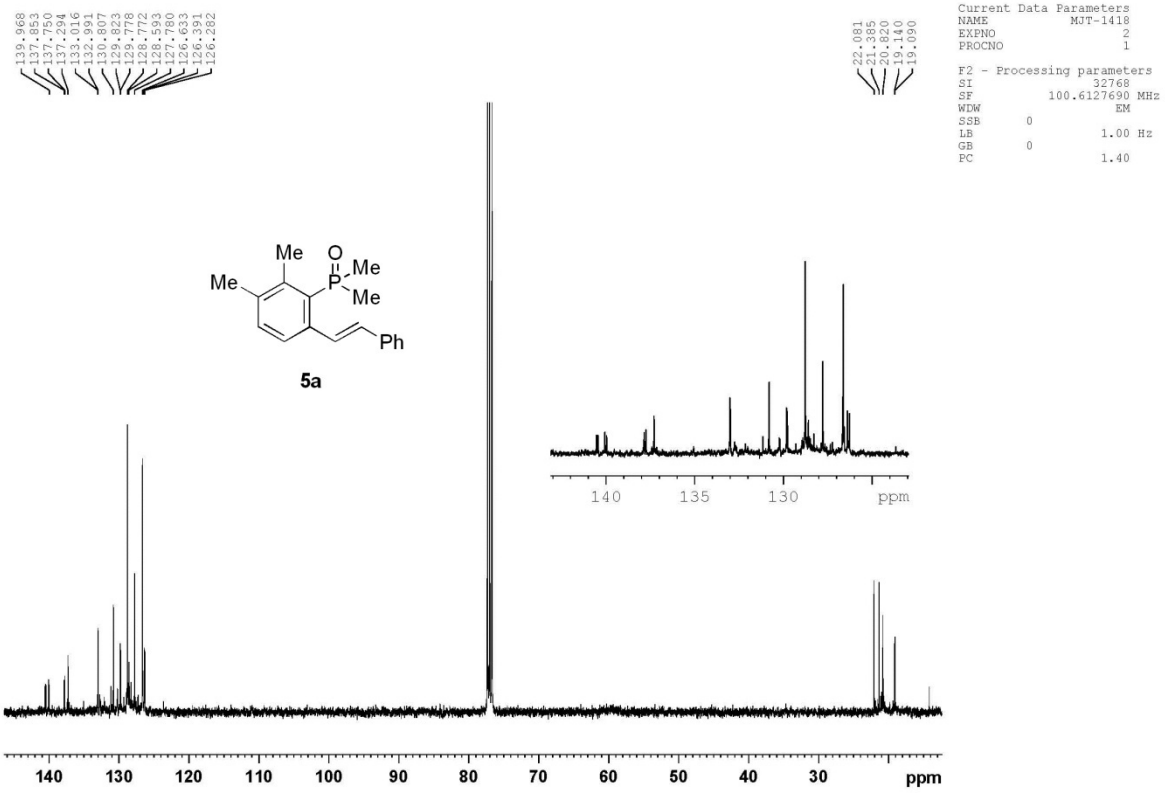

3470.17
3454.56
3162.07
3145.98
3024.10
3020.02
3015.42
3011.26
2907.62
2843.60
2834.88
2559.03
2542.99
2459.20
2443.51
1555.15
1549.90
1532.14
1527.10
1525.90
1521.29
1440.47
1428.66
767.05
764.01
751.12
749.00
743.08
738.08
735.00
725.04
701.87
-0.08
CO2Me
O
Me
P
Me
MeO
CO2Me
4b
1.000
1.046
1.229
1.099
0.985
1.124
2.272
7.160
6.099
8
7
6
5
4
3
2
1
0
ppm
Current Data Parameters
NAME LSJ-341-column-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130528
Time 22.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 167.01
DW 60.800 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SPO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300072 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

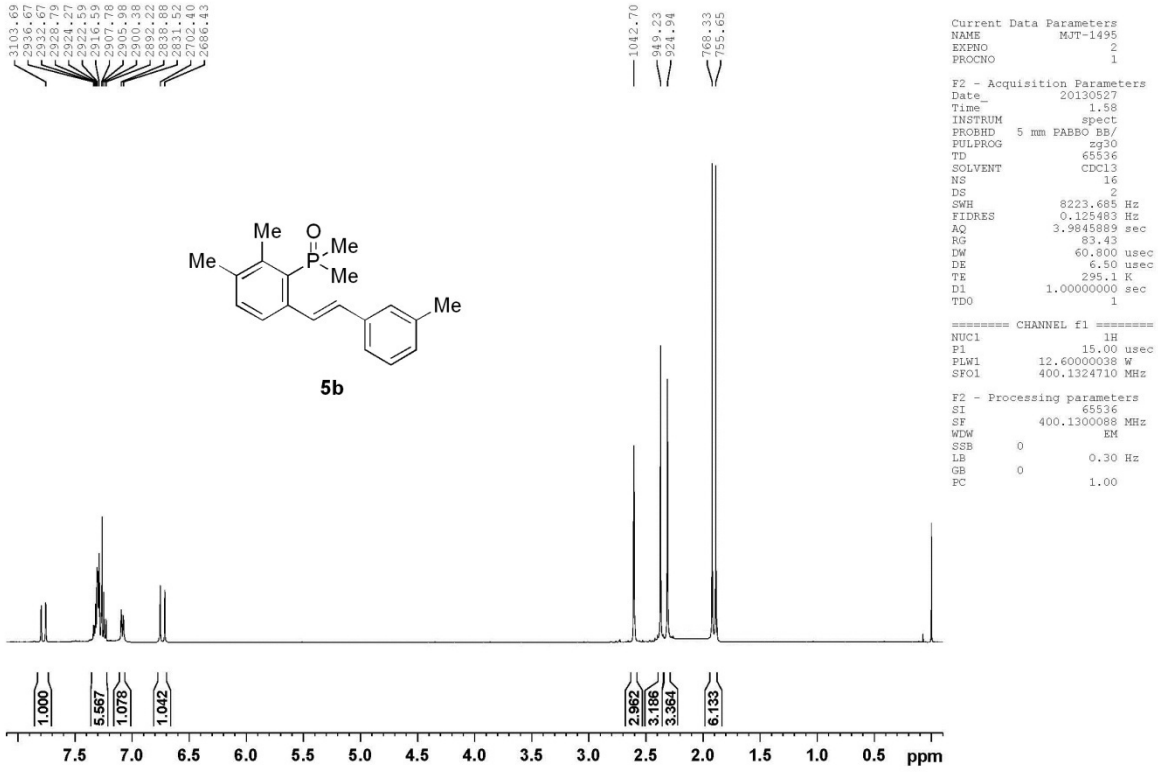

167.047
144.953
139.828
132.464
131.042
130.931
125.082
122.212
121.460
119.258
114.046
77.333
77.016
76.698
55.991
51.950
51.805
21.876
21.173
CO2Me
O
Me
P
Me
MeO
CO2Me
4b
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
Current Data Parameters
NAME LSJ-341-column-1
EXPNO 2
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

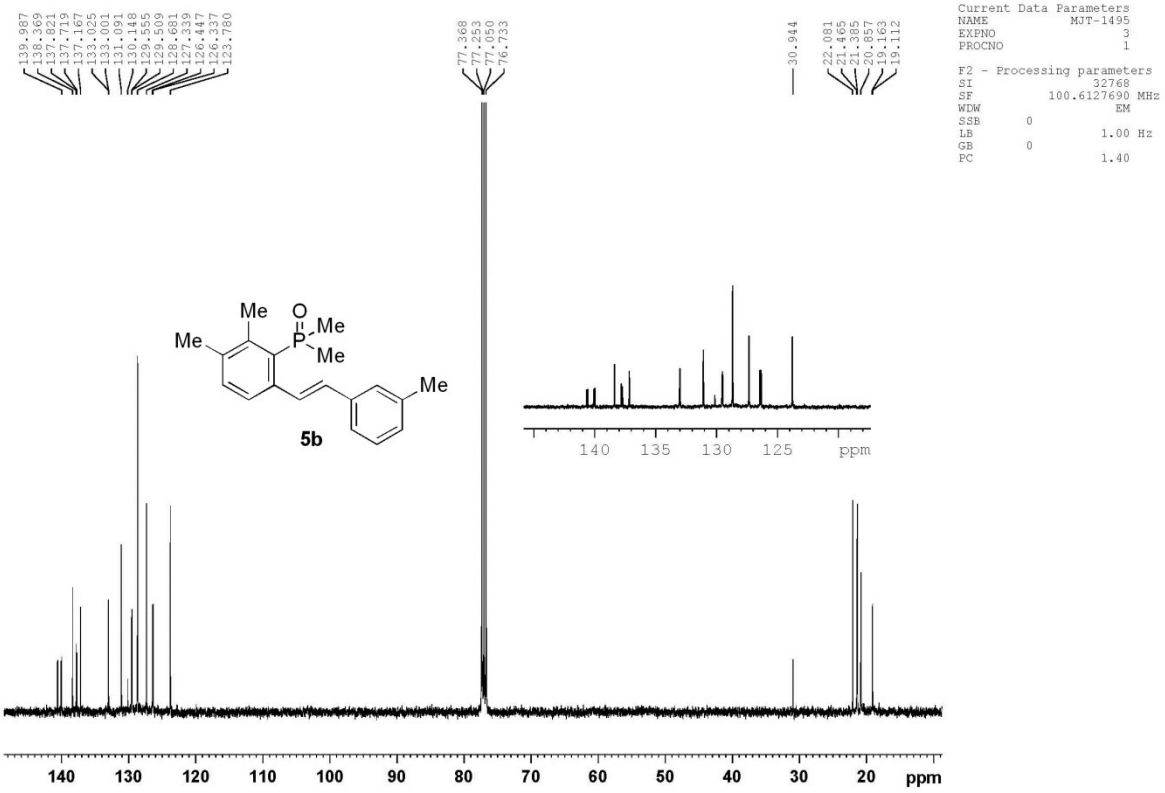

3399.42
3383.78
3154.75
3138.42
3058.23
3054.95
2906.18
2496.97
2481.25
2419.99
2403.74
1541.46
1528.14
737.12
724.12
642.97
-0.08
Current Data Parameters
NAME MJT-1410-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130625
Time 22.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 186.53
DW 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
CO2Me
Cl
O
P
Me
Me
Cl
CO2Me
4c
1.000
1.101
1.301
1.071
1.013
6.789
6.943
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

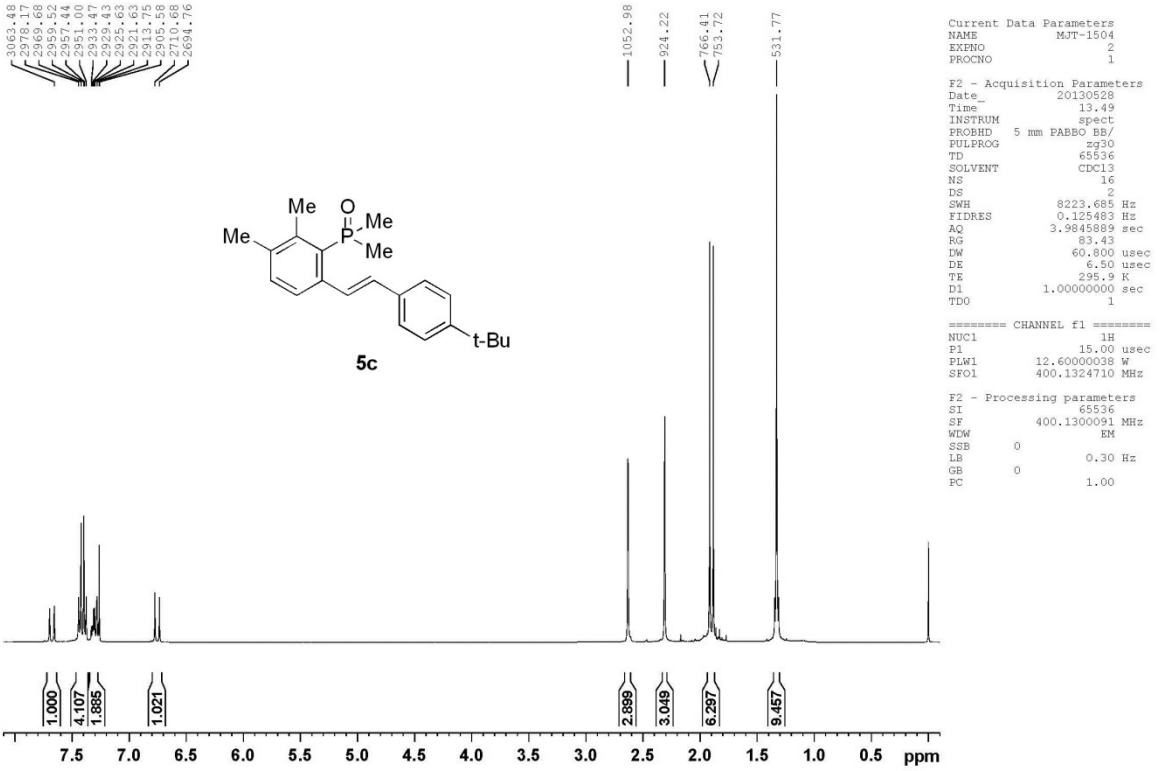

166.171
165.513
142.868
142.830
142.435
142.400
139.324
139.246
137.117
137.090
134.352
134.258
133.195
133.367
133.002
132.248
130.259
130.156
126.827
122.317
52.245
52.069
21.621
20.908
Current Data Parameters
NAME MJT-1410-NEW-3-2
EXPNO 2
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
CO2Me
Cl
O
P
Me
Me
Cl
CO2Me
4c
0 140 130 120
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

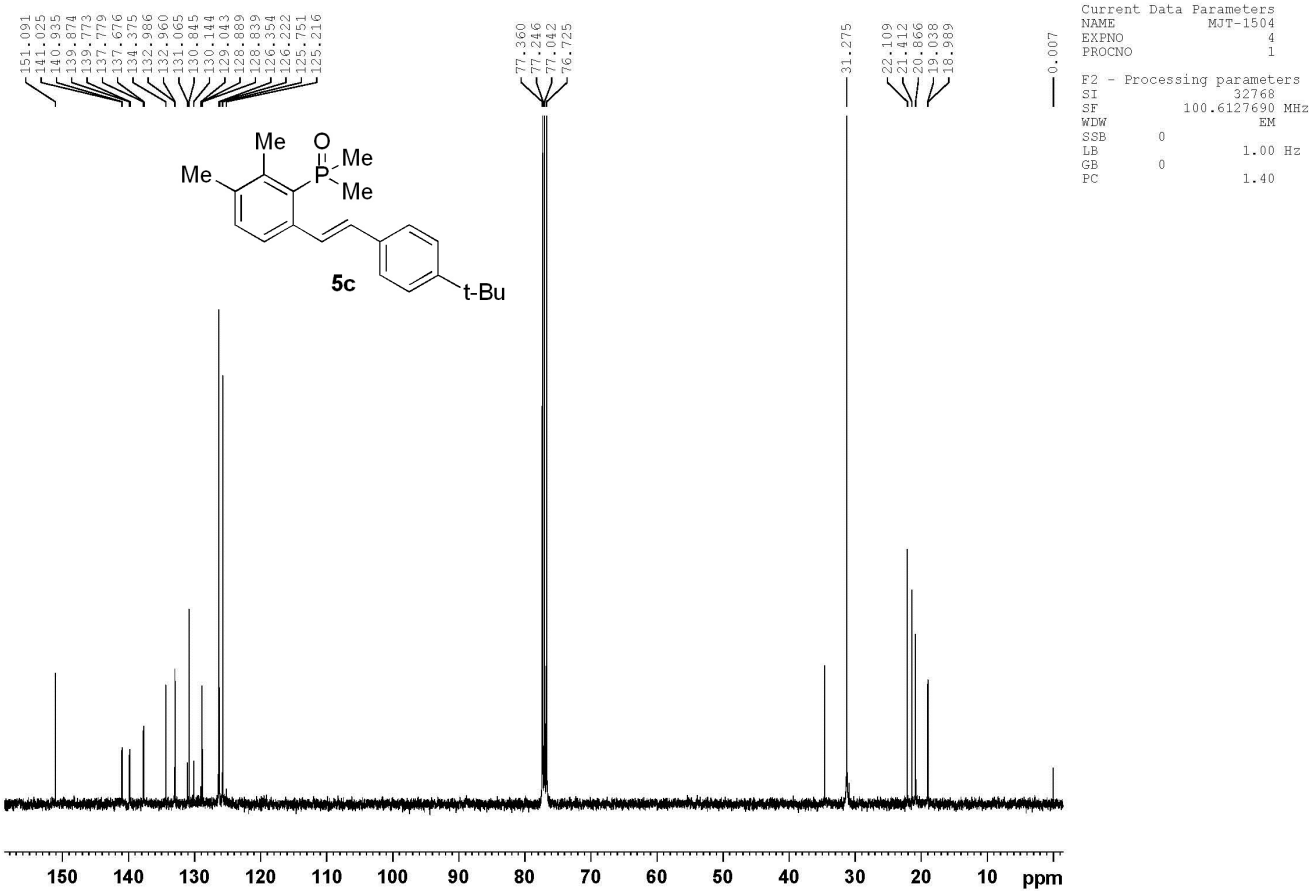

3002.74
2995.49
2994.93
2950.68
2943.44
2942.16
2940.36
2935.79
2932.35
2918.55
2912.63
2911.35
2905.30
2715.36
2699.40
1040.10
925.90
768.57
755.85
Me
O
Me
Me
P
Me
Ph
5a
Current Data Parameters
NAME MJT-1418-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130329
Time 9.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 114.24
DW 60.800 usec
DE 6.50 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.000
2.266
3.437
3.183
0.988
2.853
3.272
6.051
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

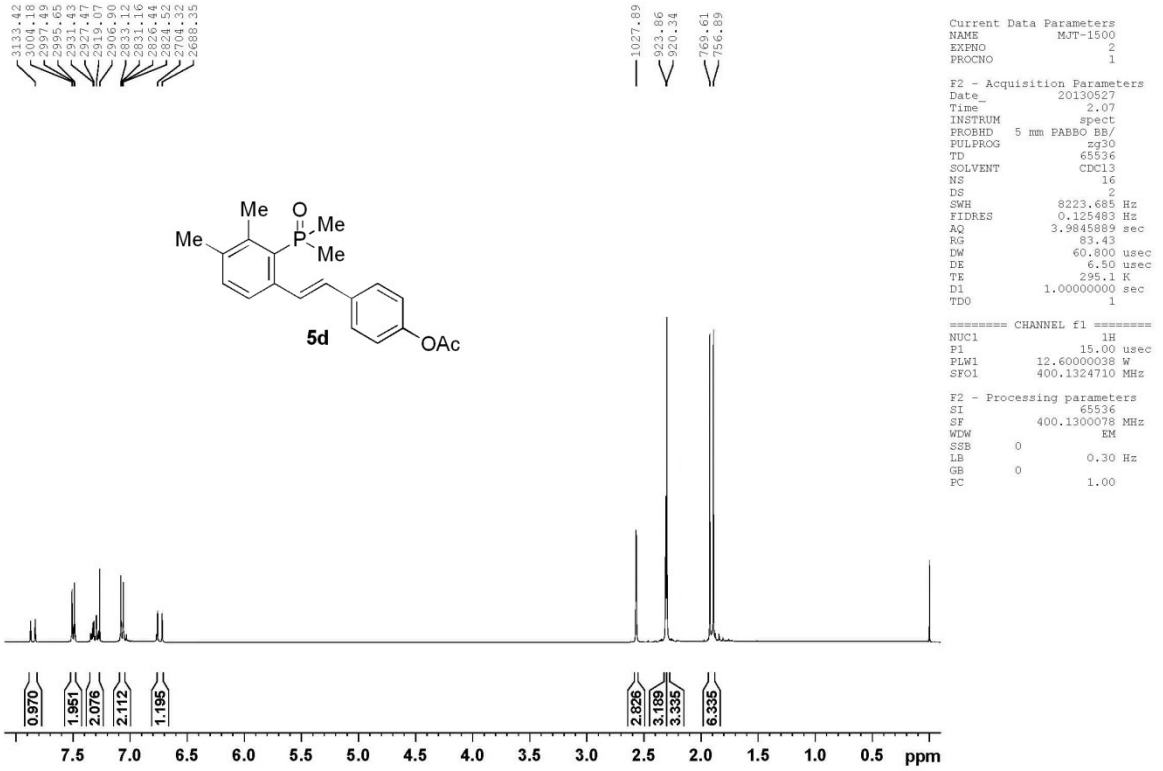

139.968
137.853
137.750
137.294
133.016
132.991
130.807
129.273
128.772
128.593
127.780
126.633
126.391
126.282
22.081
21.385
20.820
19.140
19.090
Me
O
Me
Me
P
Me
Ph
5a
Current Data Parameters
NAME MJT-1418
EXPNO 2
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
140 135 130 ppm
140 130 120 110 100 90 80 70 60 50 40 30 ppm


3103.69
2936.67
2932.67
2928.79
2924.27
2922.59
2916.59
2907.78
2905.98
2900.38
2892.22
2838.88
2831.52
2702.40
2686.43
1042.70
949.23
924.94
768.33
755.65
Me
O
Me
P
Me
Me
Me
5b
Current Data Parameters
NAME MJT-1495
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130527
Time 1.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 83.43
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.000
5.567
1.078
1.042
2.962
3.186
3.364
6.133
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

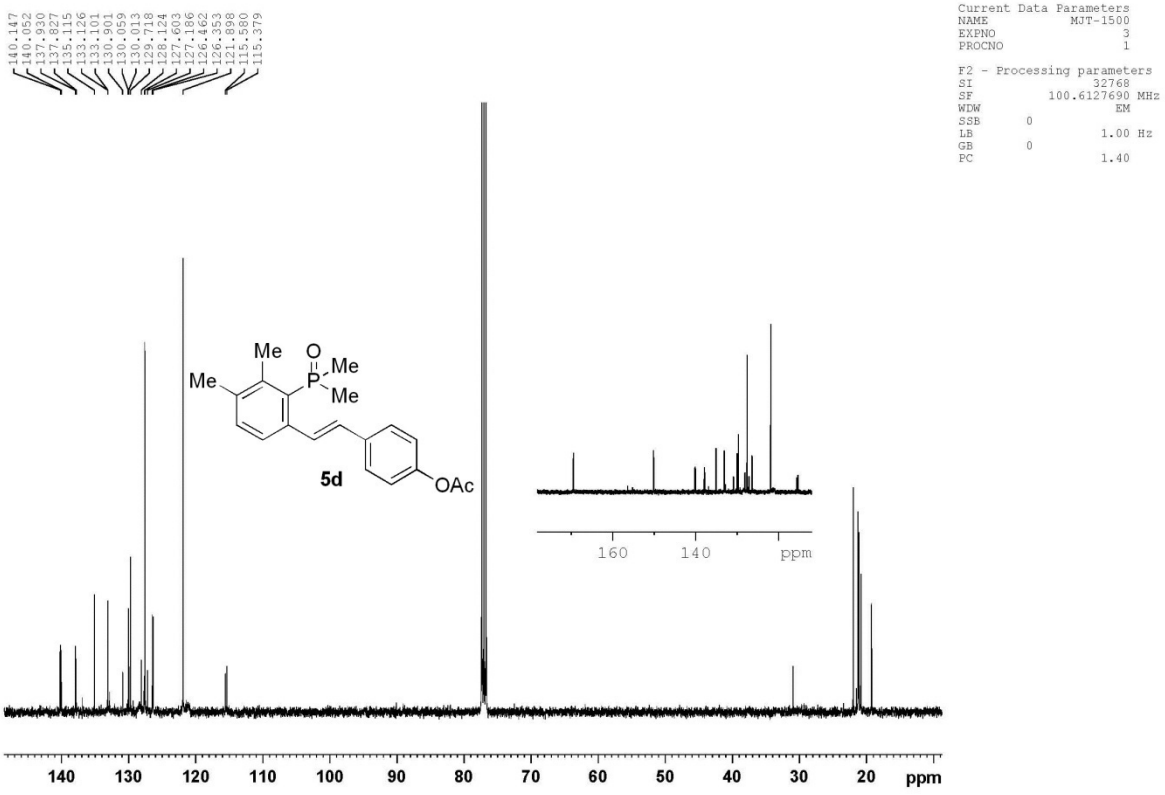

139.987
138.369
137.821
137.719
137.167
133.025
133.001
131.091
130.148
129.555
129.509
128.681
127.339
126.447
126.237
125.780
77.368
77.253
77.050
76.733
30.944
22.081
21.465
21.385
20.857
19.163
19.112
Me
O
Me
P
Me
Me
Me
5b
140
135
130
125
ppm
Current Data Parameters
NAME MJT-1495
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


3063.48
2978.17
2969.68
2959.52
2957.44
2951.00
2933.47
2929.43
2925.63
2921.63
2913.75
2905.58
2710.68
2694.76
1052.98
924.22
766.41
753.72
531.77
Me
O
Me
Me
P
Me
t-Bu
5c
Current Data Parameters
NAME MJT-1504
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130528
Time 13.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 83.43
DW 60.800 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.000
4.107
1.885
1.021
2.899
3.049
6.297
9.457
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

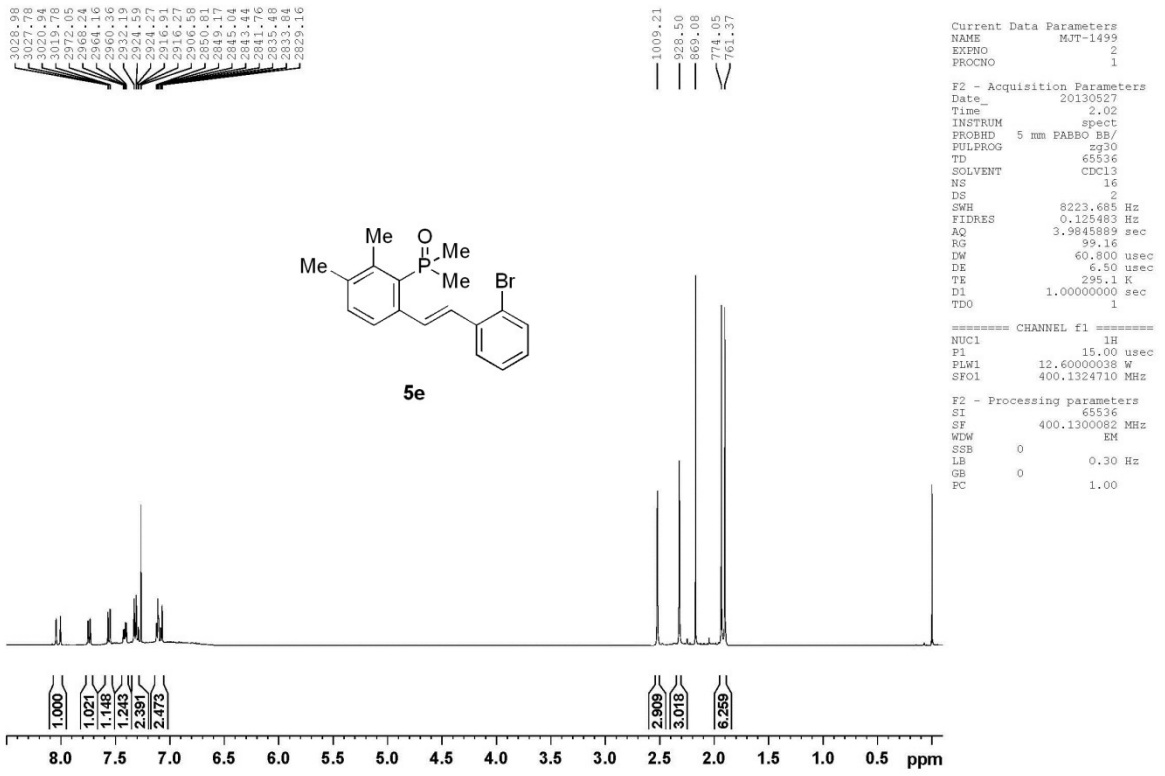

151.091
141.025
140.935
139.874
139.773
137.779
137.676
134.375
132.986
132.960
131.065
130.845
130.144
129.043
128.889
128.839
126.354
126.222
125.751
125.216
77.360
77.246
77.042
76.725
31.275
22.109
21.412
20.866
19.038
18.989
0.007
Me
O
Me
Me
P
Me
t-Bu
5c
Current Data Parameters
NAME MJT-1504
EXPNO 4
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

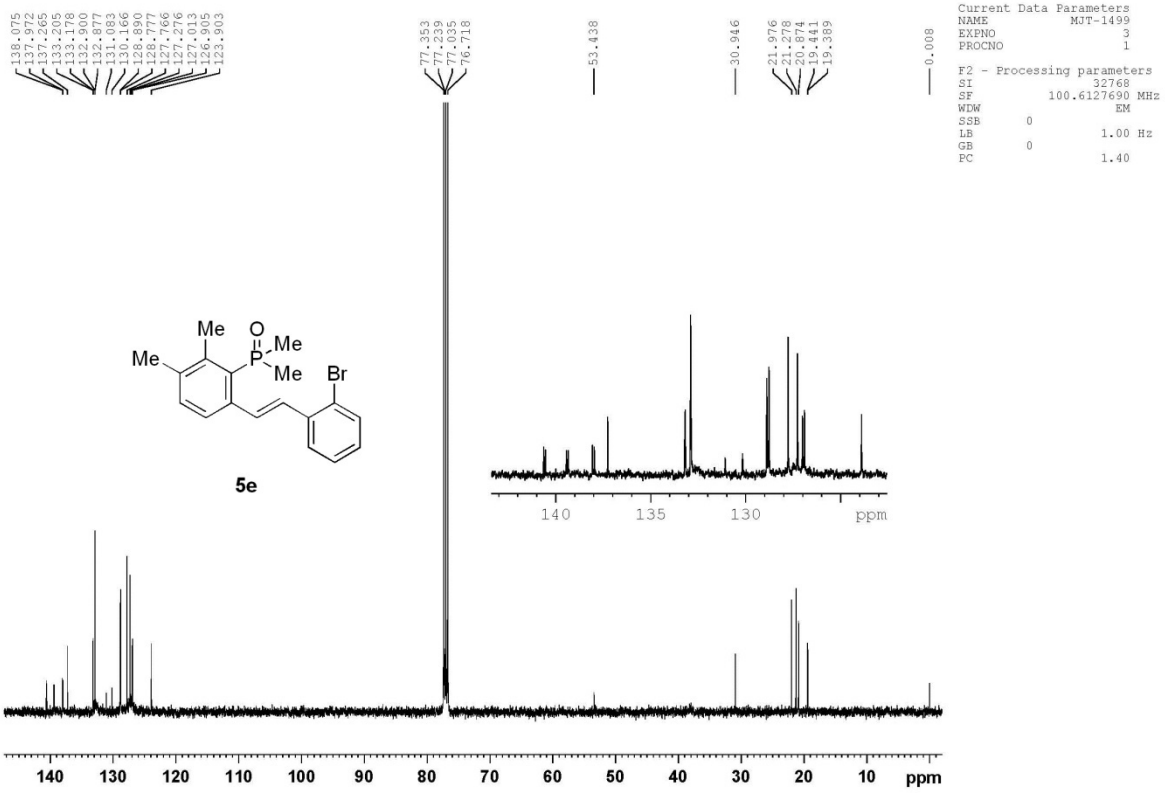


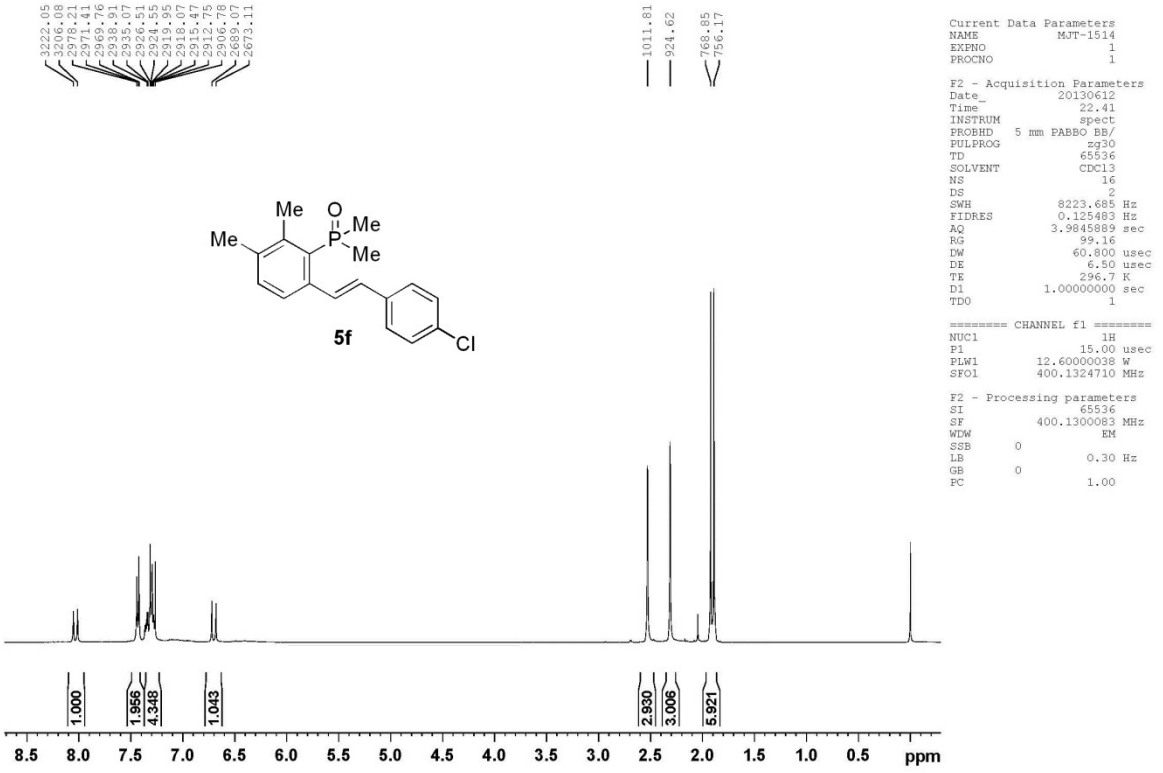

```
Current Data Parameters
NAME                  MJT-1500
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_               20130527
Time                    2.07
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9845889 sec
RG                      83.43
DW                     60.800 usec
DE                      6.50 usec
TE                     295.1 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                      15.00 usec
PLW1               12.60000038 W
SFO1              400.1324710 MHz

F2 - Processing parameters
SI                         65536
SF                400.1300078 MHz
WDW                           EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



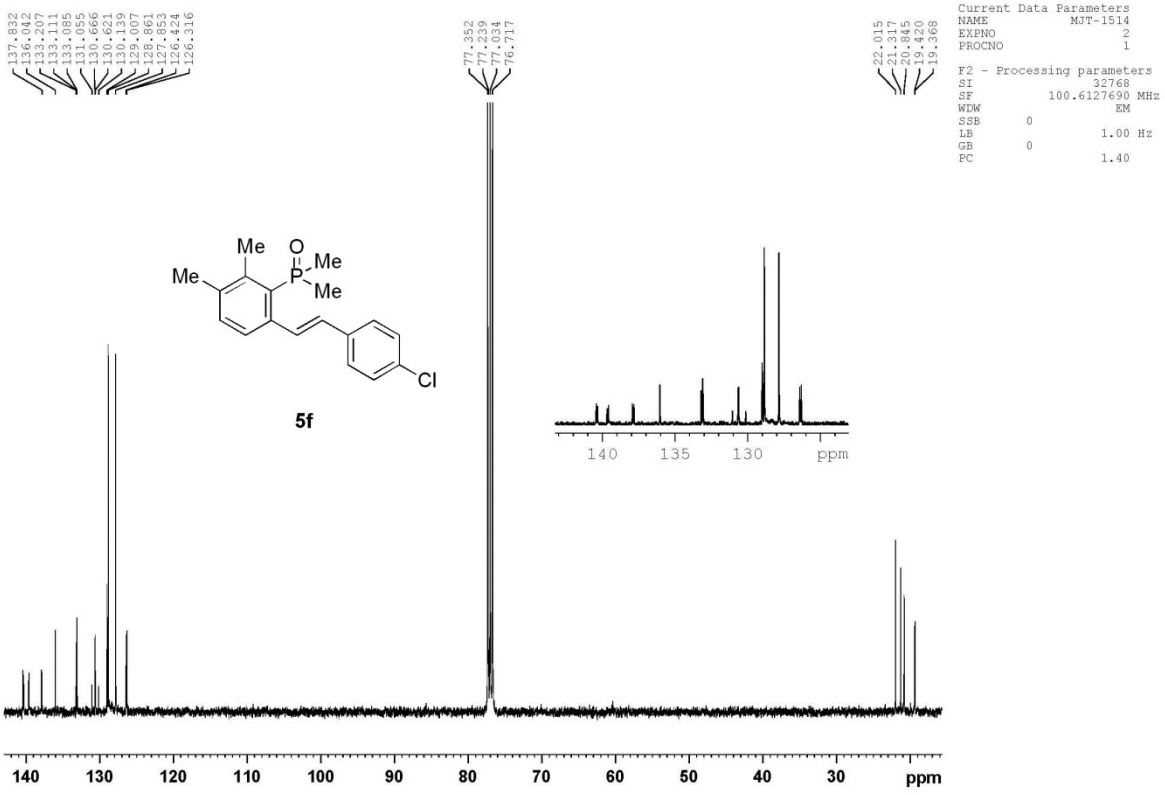

```
Current Data Parameters
NAME                  MJT-1500
EXPNO                        3
PROCNO                       1

F2 - Processing parameters
SI                         32768
SF                100.6127690 MHz
WDW                           EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```


3028.98
3027.78
3020.94
3019.78
2972.05
2968.24
2964.16
2960.36
2932.19
2924.59
2924.27
2916.91
2916.27
2906.58
2850.81
2849.17
2845.04
2843.44
2841.76
2835.48
2833.84
2829.16
1009.21
928.50
869.08
774.05
761.37
Me
O
Me
P
Me
Me
Br
5e
1.000
1.021
1.148
1.243
2.391
2.473
2.909
3.018
6.259
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
Current Data Parameters
NAME MJT-1499
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130527
Time 2.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 99.16
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

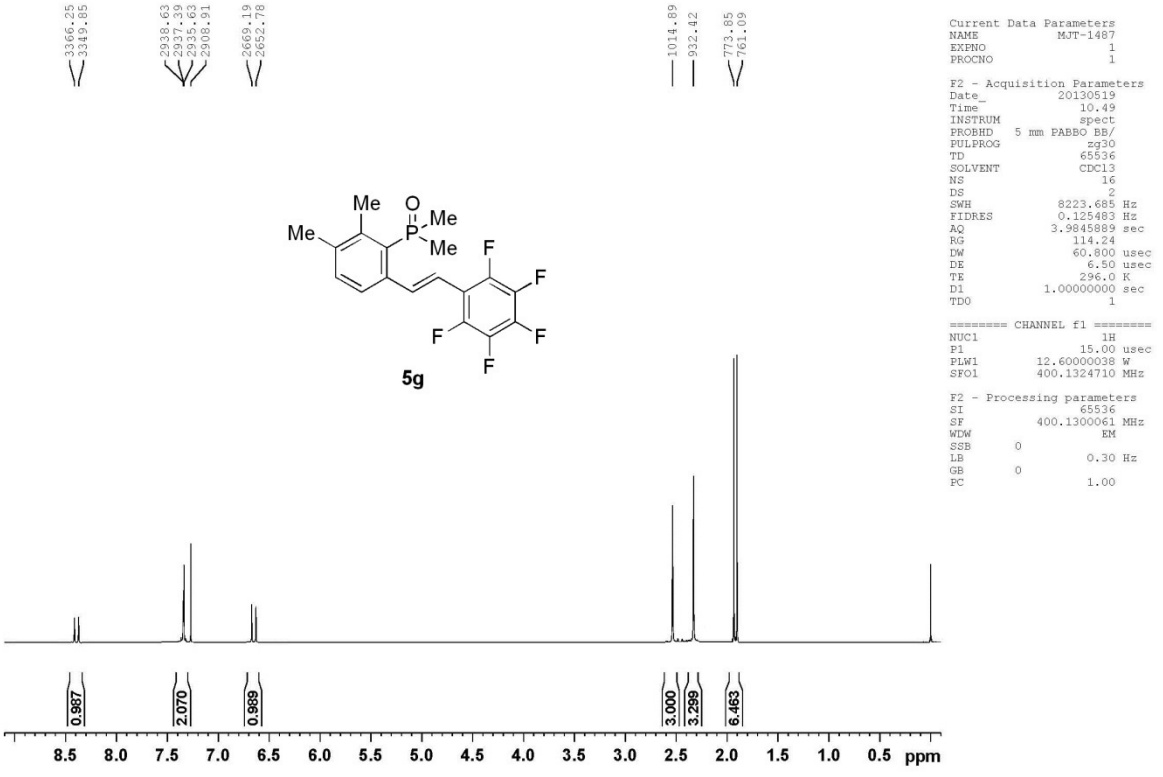

138.075
137.972
137.265
133.205
133.178
132.900
132.877
131.083
130.166
128.890
128.777
127.766
127.276
127.013
126.905
123.903
77.353
77.239
77.035
76.718
53.438
30.946
21.976
21.278
20.874
19.441
19.389
0.008
Me
O
Me
P
Me
Me
Br
5e
140
135
130
ppm
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
Current Data Parameters
NAME MJT-1499
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

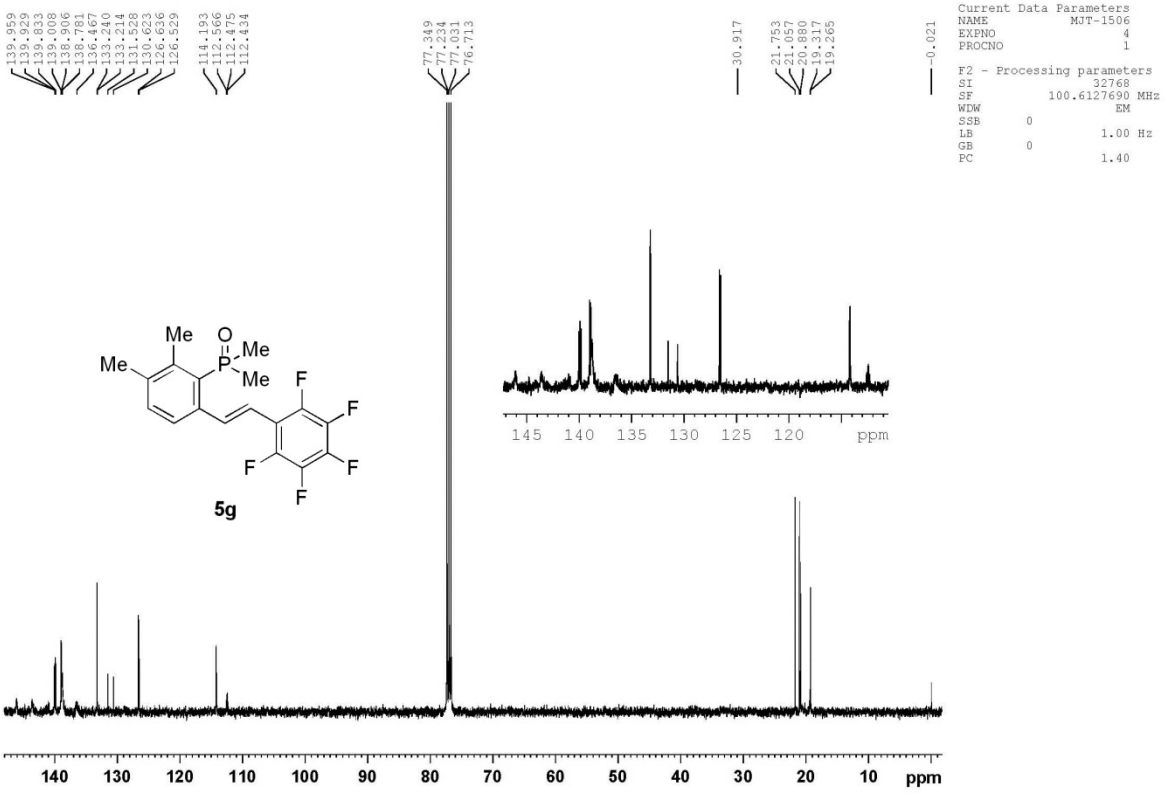



```
Current Data Parameters
NAME                  MJT-1514
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20130612
Time                   22.41
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9845889 sec
RG                    99.16
DW                   60.800 usec
DE                    6.50 usec
TE                   296.7 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.00 usec
PLW1             12.60000038 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF               400.1300083 MHz
WDW                      EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```




```
Current Data Parameters
NAME                  MJT-1514
EXPNO                        2
PROCNO                       1

F2 - Processing parameters
SI                    32768
SF               100.6127690 MHz
WDW                      EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```


3366.25
3349.85
2938.63
2937.39
2935.63
2908.91
2669.19
2652.78
1014.89
932.42
773.85
761.09
Me
O
Me
Me
P
Me
F
F
F
F
F
5g
Current Data Parameters
NAME MJT-1487
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130519
Time 10.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 114.24
DW 60.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.987
2.070
0.989
3.000
3.299
6.463
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm


139.959
139.929
139.833
139.008
138.906
138.781
136.467
133.240
133.214
131.528
130.623
126.636
126.529
114.193
112.566
112.475
112.434
77.349
77.234
77.031
76.713
30.917
21.753
21.057
20.860
19.317
19.265
-0.021
Me
O
Me
Me
P
Me
F
F
F
F
F
5g
145 140 135 130 125 120 ppm
Current Data Parameters
NAME MJT-1506
EXPNO 4
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm