

Supporting Information
For

Chemodivergent Phosphonylation of Diazocarboxylates: Light-On Vs Light-Off Reaction

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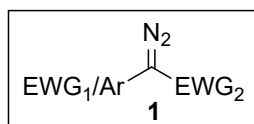
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1. General Information

Unless mentioned otherwise, all reactions were carried out under nitrogen atmosphere in flame-dried glassware. All reactions were monitored by TLC, visualization was effected with UV and/or by developing in iodine. The NMR Chemical shifts are reported in δ (ppm) relative to TMS as the internal standard for ^1H and $^{13}\text{C}\{\text{H}\}$, TFA as the internal standard for $^{19}\text{F}\{\text{H}\}$ and phosphoric acid as the external standard for $^{31}\text{P}\{\text{H}\}$. To describe spin multiplicity, standard abbreviations such as s, d, t, q, m, dd referring to singlet, doublet, triplet, quartet, multiplet and doublet of doublet respectively, are used.

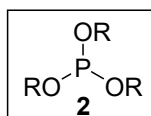
The aryldiazoacetates (**1a-1q**)¹ as well as other diazo substrates (**1r-1u**)²⁻⁴ were synthesized following reported literature protocols. All the trialkyl phosphites (**2a-2d**) were commercially available. All other chemicals and catalysts were purchased from commercial sources and used as received. The structure of substrates **1a-1u** and **2a-2d** have been provided in Table S1 and S2, respectively.

Table S1. Substitutions in diazo substrates **1**



Compound	Ar/EWG ₁	EWG ₂	Compound	Ar/EWG ₁	EWG ₂
1a	Ph	CO ₂ Me	1l	2-NO ₂ -Ph	CO ₂ Me
1b	2-OMe-Ph	-do-	1m	1-naphthyl	-do-
1c	4-OMe-Ph	-do-	1n	3,4-(OCH ₂ O)-Ph	CO ₂ Et
1d	4-OCF ₃ -Ph	-do-	1o	3-thienyl	-do-
1e	4-Ph-Ph	-do-	1p	Ph	CO ₂ allyl
1f	4-Cl-Ph	-do-	1q	Ph	CO ₂ propargyl
1g	4-Br-Ph	-do-	1r	Ph	COPh
1h	4-F-Ph	-do-	1s	Ph	CF ₃
1i	3-CF ₃ -Ph	-do-	1t	CO ₂ Et	CO ₂ Et
1j	4-CF ₃ -Ph	-do-	1u	COPh	COPh
1k	2-NO ₂ -Ph	-do-			

Table S2. Substitutions in alkyl phosphites **2**



Compound	R	Compound	R
2a	Me	2c	<i>i</i> -propyl
2b	Et	2d	allyl

2. Instrumentation Details

Melting points were recorded on a Precision melting point apparatus and are uncorrected. NMR spectra were recorded on a Bruker Avance spectrometer at 400/500 MHz (^1H), 100/125 MHz ($^{13}\text{C}\{\text{H}\}$), 162 MHz ($^{31}\text{P}\{\text{H}\}$) and 376 MHz ($^{19}\text{F}\{\text{H}\}$). The ESI-HRMS spectra were recorded on Agilent 6520-Q-ToF LC/MS system. The reactions were irradiated using high power blue LEDs (make: Original Oplunt America, power = 3 W, $\lambda_{\text{max}} = 455$ nm, luminous flux/radiant flux = 687 mW at 700 mA) fitted in an aluminum block. The block contains 6 holes to hold vials and water inlet-outlet nozzles. The vials placed in holes on the block were irradiated from bottom at a distance of ≈ 2 cm. The reaction temperature can be maintained by circulating water through the aluminum block via inlet-outlet nozzles (Figure S1).

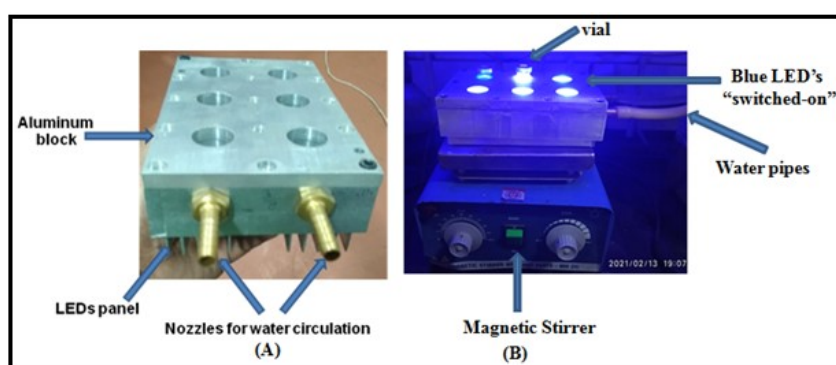


Figure S1. (A) Picture of the visible light photoreactor; (B) Experimental set-up

3. General Procedures

3.1. General procedure for thermal phosphorylation

In an oven dried 5 mL snap vial equipped with a magnetic stir bar, diazo compound **1** (0.2 mmol) and trialkyl phosphite **2** (0.6 mmol) were dissolved in anhydrous acetonitrile (2.0 mL). The solution was purged with nitrogen and stirred at room temperature (25-30 °C) for 12 h. Upon completion of the reaction (TLC monitoring), the reaction mixture was concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (100-200 mesh) with hexane/ethyl acetate as eluent, furnishing the pure product **3**.

3.2. General procedure for photocatalytic phosphorylation

In an oven dried 5 mL snap vial equipped with a magnetic stir bar, diazo compound **1** (0.4 mmol) and trialkyl phosphite (0.2 mmol) were dissolved in dichloromethane (2.0 mL). The resulting reaction mixture was degassed by three “pump-freeze-thaw” cycles via a syringe needle and stirred at room temperature (25-30 °C) under the irradiation of 455 nm, 3W blue LEDs for 6 h. Upon completion of the reaction (TLC monitoring), the reaction mixture was concentrated under reduced pressure. The crude product was purified by column chromatography on silica gel (100-200 mesh) with hexane/ethyl acetate as eluent, furnishing the pure product **4**.

4. Details of X-ray Analysis of 3k

A good quality single crystal of size 0.39 x 0.15 x 0.15 mm, was selected under a polarizing microscope and mounted on a glass fiber for data collection. Single crystal X-ray data for compound **3k** were collected on the Rigaku XtaLAB Synergy-S single crystal X-ray diffractometer equipped with a HyPix-6000HE Hybrid Photon Counting (HPC) detector, dual Mo and Cu microfocus sealed X-ray source with kappa goniometer at 294 (2) K. Data collection cell determination, and data reduction was performed using the CrysAlisPro⁵ software. Structure solution and refinement were performed by using SHELX-97.⁶ Refinement of coordinates and anisotropic thermal parameters of non-hydrogen atoms were carried out by the full-matrix least-squares method. The hydrogen atoms attached to carbons were generated with idealized geometries and isotropically refined using a riding model.

Crystallization: Crystals of compound **3k** were grown from the solvent chloroform by slow evaporation method (Figure S2, Table S3).

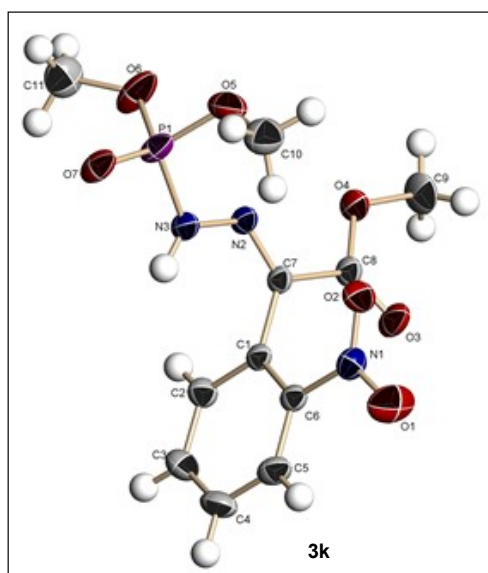


Figure S2. ORTEP diagram drawn with 30% ellipsoid probability for non-H atoms of the crystal structure of compound **3k** determined at 294K.

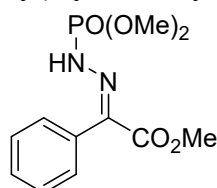
Table S3. Crystal data and structure refinement details for compound **3k**

Compound	3k
Empirical formula	C ₁₁ H ₁₄ N ₃ O ₇ P
Formula weight	331.22
Crystal System	Monoclinic
Space group	P 2 ₁ /c
<i>a</i> (Å)	9.6313(2)
<i>b</i> (Å)	8.3277(2)
<i>c</i> (Å)	18.8433(3)

α (°)	90.00
β (°)	95.670(2)
γ (°)	90.00
V (Å ³)	1503.96(5)
Z	4
D_c (g/cm ³)	1.463
F_{000}	688
μ (mm ⁻¹)	2.001
θ_{\max} (°)	77.58
Total reflections	10723
Unique reflections	3050
Reflections [$I > 2\sigma(I)$]	2716
Parameters	203
R_{int}	0.0372
Goodness-of-fit	1.065
R [$F^2 > 2\sigma(F^2)$]	0.0649
wR (F^2 , all data)	0.1805
CCDC No.	2342436

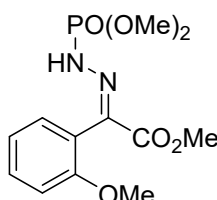
5. Characterization Data

Methyl (*E*)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-phenylacetate (**3a**)⁷



White solid; isolated yield 78% (45 mg). R_f 0.50 (50% EtOAc/hexane); Mp 93-95 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.49-7.54 (m, 3H), 7.26 (d, $J = 7.8$ Hz, 2H merged with solvent peak), 7.08 (d, $^2J_{\text{H-P}} = 30.5$ Hz, 1H), 3.87 (d, $^3J_{\text{H-P}} = 12.2$ Hz, 6H merged with s at 3.84), 3.84 (s, 3H merged with d at 3.87); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 163.95, 143.66 (d, $^3J_{\text{C-P}} = 16.9$ Hz), 130.39, 129.70, 128.40, 128.29, 54.42 (d, $^2J_{\text{C-P}} = 5.9$ Hz), 52.77; ³¹P{¹H} NMR (162 MHz, CDCl₃) δ 1.62; HRMS for C₁₁H₁₆N₂O₅P⁺: calcd. [M+H]⁺: 287.0791, found: 287.0792

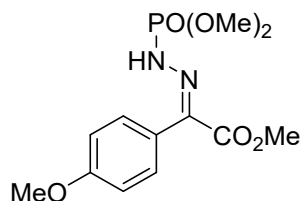
Methyl (*E*)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-(2-methoxyphenyl)acetate (**3b**)



White solid; isolated yield 76% (51 mg). R_f 0.50 (60% EtOAc/hexane); Mp 144-145 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.44-7.48 (m, 1H), 7.13 (dd, $J = 7.4$ Hz, 1.5 Hz, 1H), 7.07 (t, $J = 7.3$ Hz, 1H), 7.01 (d, $J = 8.4$ Hz, 1H), 6.91 (d, $^2J_{\text{H-P}} = 30.5$ Hz, 1H), 3.85 (d, $^3J_{\text{H-P}} = 11.4$ Hz, 6H), 3.82 (s, 3H), 3.80 (s, 3H); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 164.06, 156.70, 141.61

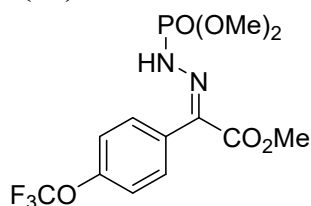
(d, $^3J_{C-P} = 17.4$ Hz), 132.08, 129.84, 121.46, 117.08, 111.78, 55.64, 54.28 (d, $^2J_{C-P} = 5.8$ Hz), 52.63; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.81; HRMS for $\text{C}_{12}\text{H}_{17}\text{N}_2\text{O}_6\text{PNa}^+$: calcd. $[\text{M}+\text{Na}]^+$: 339.0722, found: 339.0718

Methyl (E)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-(4-methoxyphenyl)acetate (3c)⁷



White solid; isolated yield 70% (47 mg). R_f 0.50 (60% EtOAc/hexane); Mp 158-160 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.18-7.22 (m, 2H), 7.13 (d, $^2J_{H-P} = 30.4$ Hz, 1H), 7.01-7.04 (m, 2H), 3.86 (s, 3H), 3.85 (d, $^3J_{H-P} = 11.4$ Hz, 6H), 3.83 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 164.21, 160.95, 143.57 (d, $^3J_{C-P} = 17.2$ Hz), 129.88, 120.11, 115.15, 55.43, 54.39 (d, $^2J_{C-P} = 5.9$ Hz), 52.74; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.82; HRMS for $\text{C}_{12}\text{H}_{17}\text{N}_2\text{O}_6\text{PNa}^+$: calcd. $[\text{M}+\text{Na}]^+$: 339.0722, found: 339.0719

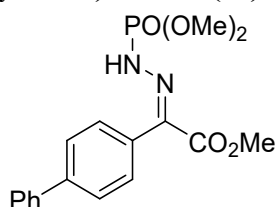
Methyl (E)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-(4-(trifluoromethoxy)phenyl)acetate (3d)



White solid; isolated yield 69% (51 mg). R_f 0.50 (40% EtOAc/hexane); Mp 135-137 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.38 (d, $J = 8.2$ Hz, 2H), 7.31-7.33 (m, 2H), 7.05 (d, $^2J_{H-P} = 30.1$ Hz, 1H), 3.86 (d, $^3J_{H-P} = 11.5$ Hz, 6H), 3.85 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 163.71, 150.43, 142.03 (d, $^3J_{C-P} = 16.8$ Hz), 130.38, 126.81, 122.00, 120.34 (q, $^1J_{C-F} = 257.2$ Hz), 54.51 (d, $^2J_{C-P} = 5.9$ Hz), 52.87; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.27; $^{19}\text{F}\{\text{H}\}$ NMR (376 MHz, CDCl_3) δ -57.70; HRMS for $\text{C}_{12}\text{H}_{15}\text{F}_3\text{N}_2\text{O}_6\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 371.0614, found: 371.0619

Methyl (E)-2-([1,1'-biphenyl]-4-yl)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)acetate (3e)

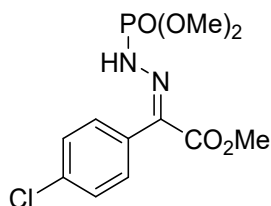
(E)-2-([1,1'-biphenyl]-4-yl)-2-(2-



White solid; isolated yield 65% (47 mg). R_f 0.50 (50% EtOAc/hexane); Mp 133-134 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.72 (d, $J = 8.2$ Hz, 2H), 7.60 (d, $J = 7.2$ Hz, 2H), 7.48 (t, $J = 7.5$ Hz, 2H), 7.41 (t, $J = 7.3$ Hz, 1H), 7.34 (d, $J = 8.2$ Hz, 2H), 7.16 (d, $^2J_{H-P} = 30.4$ Hz, 1H), 3.87 (d, $^3J_{H-P} = 11.4$ Hz, 6H), 3.86 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 164.02, 143.44, 143.43 (d, $^3J_{C-P} = 16.9$ Hz), 140.03, 128.98, 128.79, 128.46, 128.04, 127.26, 127.03, 54.46 (d,

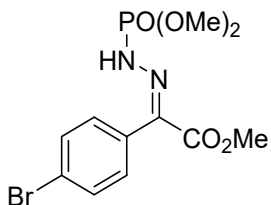
$^2J_{C-P} = 5.9$ Hz), 52.83; $^{31}P\{H\}$ NMR (162 MHz, $CDCl_3$) δ 1.64; HRMS for $C_{17}H_{20}N_2O_5P^+$: calcd. $[M+H]^+$: 363.1104, found: 363.1109

Methyl (E)-2-(4-chlorophenyl)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)acetate (3f)⁷



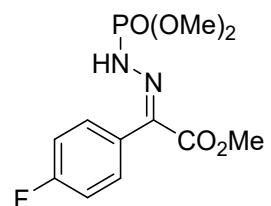
White solid; isolated yield 75% (51 mg). R_f 0.50 (50% EtOAc/hexane); Mp 160-161 °C; 1H NMR (400 MHz, $CDCl_3$) δ 7.50-7.52 (m, 2H), 7.20-7.22 (m, 2H), 7.03 (d, $^2J_{H-P} = 30.1$ Hz, 1H), 3.86 (d, $^3J_{H-P} = 11.4$ Hz, 6H), 3.84 (s, 3H); $^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$) δ 163.70, 142.31 (d, $^3J_{C-P} = 16.9$ Hz), 136.67, 130.04, 129.89, 126.66, 54.49 (d, $^2J_{C-P} = 6.0$ Hz), 52.85; $^{31}P\{H\}$ NMR (162 MHz, $CDCl_3$) δ 1.33; HRMS for $C_{11}H_{14}ClN_2O_5PNa^+$: calcd. $[M+Na]^+$: 343.0227, found: 343.0217

Methyl (E)-2-(4-bromophenyl)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)acetate (3g)⁷



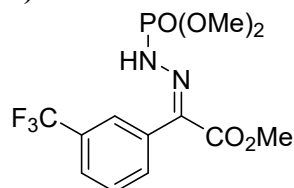
White solid; isolated yield 70% (54 mg). R_f 0.50 (50% EtOAc/hexane); Mp 150-152 °C; 1H NMR (400 MHz, $CDCl_3$) δ 7.64-7.66 (m, 2H), 7.12-7.14 (m, 2H), 7.08 (d, $^2J_{H-P} = 31.2$ Hz, 1H), 3.84 (d, $^3J_{H-P} = 11.4$ Hz, 6H merged with s at 3.82), 3.82 (s, 3H); $^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$) δ 163.62, 142.33 (d, $^3J_{C-P} = 16.6$ Hz), 133.00, 130.06, 127.14, 124.92, 54.50 (d, $^2J_{C-P} = 5.9$ Hz), 52.85; $^{31}P\{H\}$ NMR (162 MHz, $CDCl_3$) δ 1.30; HRMS for $C_{11}H_{14}BrN_2O_5PNa^+$: calcd. $[M+Na]^+$: 386.9721, found: 386.9711

Methyl (E)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-(4-fluorophenyl)acetate (3h)⁷



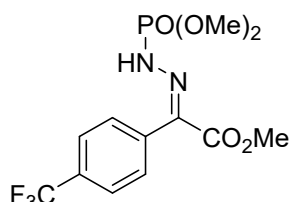
White solid; isolated yield 85% (55 mg). R_f 0.50 (60% EtOAc/hexane); Mp 144-146 °C; 1H NMR (400 MHz, $CDCl_3$) δ 7.20-7.29 (m, 4H), 7.06 (d, $^2J_{H-P} = 30.2$ Hz, 1H), 3.86 (d, $^3J_{H-P} = 11.4$ Hz, 6H), 3.84 (s, 3H); $^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$) δ 163.86, 163.58 (d, $^1J_{C-F} = 250.0$ Hz), 142.50, (d, $^3J_{C-P} = 16.8$ Hz), 130.65 (d, $^3J_{C-F} = 8.5$ Hz), 124.24 (d, $^4J_{C-F} = 3.5$ Hz), 116.98 (d, $^2J_{C-F} = 21.8$ Hz), 54.46 (d, $^2J_{C-P} = 5.9$ Hz), 52.81; $^{31}P\{H\}$ NMR (162 MHz, $CDCl_3$) δ 1.41; $^{19}F\{H\}$ NMR (376 MHz, $CDCl_3$) δ -108.96; HRMS for $C_{11}H_{14}FN_2O_5PNa^+$: calcd. $[M+Na]^+$: 327.0522, found: 327.0528

Methyl (E)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-(3-(trifluoromethyl)phenyl)acetate (3i)



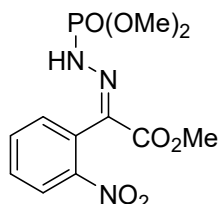
White solid; isolated yield 62% (44 mg). R_f 0.50 (40% EtOAc/hexane); Mp 159-160 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.74 (d, $J = 7.9$ Hz, 1H), 7.65 (t, $J = 7.7$ Hz, 1H), 7.51 (s, 1H), 7.44 (d, $J = 7.6$ Hz, 1H), 7.12 (d, $^2J_{\text{H-P}} = 29.9$ Hz, 1H), 3.83 (d, $^3J_{\text{H-P}} = 11.4$ Hz, 6H), 3.82 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 163.56, 141.67 (d, $^3J_{\text{C-P}} = 17.0$ Hz), 132.15 (q, $^2J_{\text{C-F}} = 32.8$ Hz), 131.90, 130.19, 129.52, 127.12 (q, $J_{\text{C-F}} = 3.6$ Hz), 125.55 (q, $J_{\text{C-F}} = 3.6$ Hz), 123.49 (q, $^1J_{\text{C-F}} = 271.0$ Hz), 54.47 (d, $^2J_{\text{C-P}} = 5.9$ Hz), 52.86; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.09; $^{19}\text{F}\{\text{H}\}$ NMR (376 MHz, CDCl_3) δ -62.84; HRMS for $\text{C}_{12}\text{H}_{14}\text{F}_3\text{N}_2\text{O}_5\text{PNa}^+$: calcd. $[\text{M}+\text{Na}]^+$: 377.0490, found: 377.0487

Methyl (E)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-(4-(trifluoromethyl)phenyl)acetate (3j)



White solid; isolated yield 76% (54 mg). R_f 0.50 (40% EtOAc/hexane); Mp 156-157 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.80 (d, $J = 8.1$ Hz, 2H), 7.41 (d, $J = 8.0$ Hz, 2H), 7.01 (d, $^2J_{\text{H-P}} = 29.9$ Hz, 1H), 3.86 (d, $^3J_{\text{H-P}} = 11.4$ Hz, 6H), 3.85 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 163.49, 141.86 (d, $^3J_{\text{C-P}} = 16.9$ Hz), 132.41 (q, $J_{\text{C-F}} = 32.5$ Hz), 132.23, 129.11, 126.65 (q, $J_{\text{C-F}} = 3.6$ Hz), 123.52 (q, $^1J_{\text{C-F}} = 271.1$ Hz), 54.52 (d, $^2J_{\text{C-P}} = 6.0$ Hz), 52.90; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.09; $^{19}\text{F}\{\text{H}\}$ NMR (376 MHz, CDCl_3) δ -63.18; HRMS for $\text{C}_{12}\text{H}_{14}\text{F}_3\text{N}_2\text{O}_5\text{PNa}^+$: calcd. $[\text{M}+\text{Na}]^+$: 377.0490, found: 377.0498

Methyl (E)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-(2-nitrophenyl)acetate (3k)

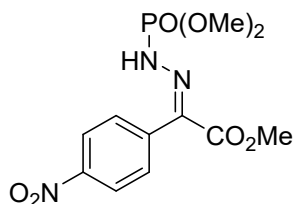


White solid; isolated yield 33% (22 mg). R_f 0.50 (40% EtOAc/hexane); Mp 186-188 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.33 (dd, $J = 8.2$ Hz, 1.2 Hz, 1H), 7.78-7.82 (m, 1H), 7.70-7.74 (m, 1H), 7.34 (dd, $J = 7.5$ Hz, 1.4 Hz, 1H), 7.03 (d, $^2J_{\text{H-P}} = 29.1$ Hz, 1H), 3.86 (d, $^3J_{\text{H-P}} = 11.3$ Hz, 3H), 3.81 (s, 3H), 3.78 (d, $^3J_{\text{H-P}} = 11.6$ Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 162.92, 147.66, 140.34 (d, $^3J_{\text{C-P}} = 17.4$ Hz), 134.85, 131.48, 130.56, 125.75, 125.26, 54.57 (d, $^2J_{\text{C-P}} = 6.0$ Hz), 54.21 (d, $^2J_{\text{C-P}} = 5.7$ Hz), 52.93; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.10; HRMS for $\text{C}_{11}\text{H}_{14}\text{N}_3\text{O}_7\text{PNa}^+$: calcd. $[\text{M}+\text{Na}]^+$: 354.0467, found: 354.0467

Selected X-ray crystallographic data for 3k, $\text{C}_{11}\text{H}_{14}\text{N}_3\text{O}_7\text{P}$, $M = 331.22$, Monoclinic, $P 2_1/c$, $a = 9.6313$ (2) Å, $b = 8.3277$ (2) Å, $c = 18.8433$ (3) Å, $V = 1503.96$ (5) Å³, $\alpha = 90.00^\circ$, β

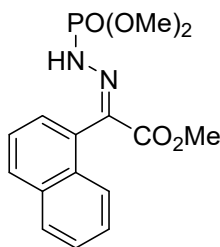
= 95.670° (2), γ = 90.00°, Z = 4, D_c = 1.463 g cm⁻³, μ (Mo-K α) = 2.001 mm⁻¹, $F(000)$ = 688, Reflections collected/unique 10723/3050, $[R(\text{int}) = 0.0372]$. Final R indices $[I > 2\sigma(I)]$, $R1 = 0.0649$, $wR = 0.1805$

Methyl (*E*)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-(4-nitrophenyl)acetate (3l)



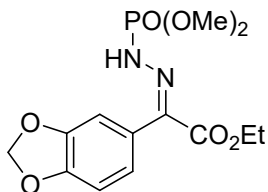
Yellow solid; isolated yield 55% (36 mg). R_f 0.50 (40% EtOAc/hexane); Mp 82-83 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.39 (d, J = 8.7 Hz, 2H), 7.49 (d, J = 8.7 Hz, 2H), 6.96 (d, $^2J_{\text{H-P}}$ = 29.6 Hz, 1H), 3.87 (d, $^3J_{\text{H-P}}$ = 11.1 Hz, 6H merged with s at 3.86), 3.86 (s, 3H); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 158.49, 144.06, 136.19, 130.29, 125.26, 119.95, 49.87 (d, $^2J_{\text{C-P}}$ = 6.1 Hz), 48.26; ³¹P{¹H} NMR (162 MHz, CDCl₃) δ 0.76; HRMS for C₁₁H₁₄N₃O₇PK⁺: calcd. $[M+K]^+$: 370.0206, found: 370.0197

Methyl (*E*)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-(naphthalen-1-yl)acetate (3m)



White solid; isolated yield 63% (45 mg). R_f 0.50 (50% EtOAc/hexane); Mp 176-178 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.00 (d, J = 8.2 Hz, 1H), 7.95 (d, J = 7.8 Hz, 1H), 7.51-7.61 (m, 4H), 7.38 (dd, J = 7.0 Hz, 1.1 Hz, 1H), 6.75 (d, $^2J_{\text{H-P}}$ = 30.7 Hz, 1H), 3.86 (d, $^3J_{\text{H-P}}$ = 11.4 Hz, 3H), 3.83 (s, 3H), 3.82 (d, $^3J_{\text{H-P}}$ = 11.4 Hz, 3H); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 164.11, 143.23 (d, $^3J_{\text{C-P}}$ = 17.2 Hz), 133.89, 130.86, 129.71, 129.14, 127.65, 127.27, 126.96, 126.23, 125.72, 123.74, 54.47 (d, $^2J_{\text{C-P}}$ = 6.0 Hz), 54.26 (d, $^2J_{\text{C-P}}$ = 5.8 Hz), 52.83; ³¹P{¹H} NMR (162 MHz, CDCl₃) δ 1.45; HRMS for C₁₅H₁₇N₂O₅PNa⁺: calcd. $[M+Na]^+$: 359.0773, found: 359.0780

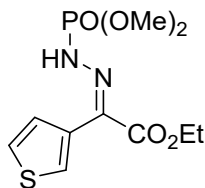
Ethyl (*E*)-2-(benzo[d][1,3]dioxol-5-yl)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)acetate (3n)



White solid; isolated yield 70% (48 mg). R_f 0.50 (40% EtOAc/hexane); Mp 104-106 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.13 (d, $^2J_{\text{H-P}}$ = 30.4 Hz, 1H), 6.92 (d, J = 7.8 Hz, 1H), 6.73 (d appearing as s, 1H, merged with s at 6.70), 6.70 (s, 1H), 6.03 (s, 2H), 4.29 (q, J = 7.0 Hz, 2 H), 3.85 (d, $^3J_{\text{H-P}}$ = 11.3 Hz, 6H), 1.31 (t, J = 7.1 Hz, 3H); ¹³C{¹H} NMR (100 MHz, CDCl₃) δ 163.51, 149.15, 148.76, 143.33 (d, $^3J_{\text{C-P}}$ = 16.9 Hz), 122.34, 121.54, 109.40, 108.68, 101.69,

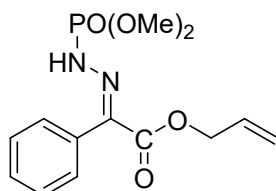
61.83, 54.47 (d, $^2J_{C-P} = 6.0$ Hz), 14.17; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.53; HRMS for $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_7\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 345.0846, found: 345.0854

Ethyl (*E*)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-(thiophen-3-yl)acetate (3o)



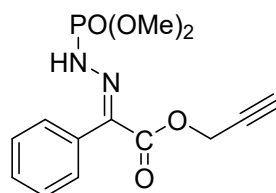
Yellow liquid; isolated yield 55% (34 mg). R_f 0.50 (40% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.51-7.53 (m, 2H), 7.34 (d, $^2J_{H-P} = 30.2$ Hz, 1H), 7.12 (dd, $J = 4.4$ Hz, 1.8 Hz, 1H), 4.31 (q, $J = 7.1$ Hz, 2 H), 3.87 (d, $^3J_{H-P} = 11.3$ Hz, 6H), 1.33 (t, $J = 7.1$ Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 163.39, 139.38 (d, $^3J_{C-P} = 17.1$ Hz), 127.95, 127.65, 127.28, 126.91, 61.83, 54.53 (d, $^2J_{C-P} = 6.1$ Hz), 14.15; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.51; HRMS for $\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_5\text{PS}^+$: calcd. $[\text{M}+\text{H}]^+$: 307.0512, found: 307.0504

Allyl (*E*)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-phenylacetate (3p)



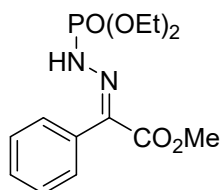
White solid; isolated yield 63% (39 mg). R_f 0.50 (40% EtOAc/hexane); Mp 44-46 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.48-7.54 (m, 3H), 7.25-7.27 (m, 2H), 7.10 (d, $^2J_{H-P} = 30.5$ Hz, 1H), 5.90-5.99 (m, 1H), 5.29-5.33 (m, 1H), 5.22-5.25 (m, 1H), 4.72-4.74 (m, 2H), 3.86 (d, $^3J_{H-P} = 11.3$ Hz, 6H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 163.10, 143.56 (d, $^3J_{C-P} = 16.9$ Hz), 131.70, 130.35, 129.67, 128.40, 128.29, 118.44, 66.18, 54.50 (d, $^2J_{C-P} = 6.0$ Hz); $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.33; HRMS for $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_5\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 313.0948, found: 313.0947

Prop-2-yn-1-yl (*E*)-2-(2-(dimethoxyphosphoryl)hydrazineylidene)-2-phenylacetate (3q)



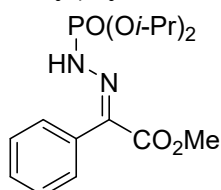
White solid; isolated yield 60% (37 mg). R_f 0.50 (40% EtOAc/hexane); Mp 54-56 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.47-7.55 (m, 3H), 7.26-7.28 (m, 2H), 7.16 (d, $^2J_{H-P} = 30.5$ Hz, 1H), 4.83 (d, $J = 2.32$ Hz, 2H), 3.87 (d, $^3J_{H-P} = 11.3$ Hz, 6H), 2.48 (s, 1H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 162.71, 142.82 (d, $^3J_{C-P} = 16.9$ Hz), 130.48, 129.71, 128.36, 128.00, 77.30, 75.24, 54.56 (d, $^2J_{C-P} = 6.0$ Hz), 53.00; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.16; HRMS for $\text{C}_{13}\text{H}_{15}\text{N}_2\text{O}_5\text{PNa}^+$: calcd. $[\text{M}+\text{Na}]^+$: 333.0616, found: 333.0613

Methyl (*E*)-2-(2-(diethoxyphosphoryl)hydrazineylidene)-2-phenylacetate (3r)⁷



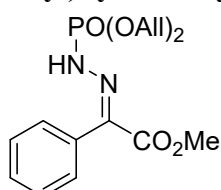
White solid; isolated yield 72% (45 mg). R_f 0.50 (40% EtOAc/hexane); Mp 60-62 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.48-7.54 (m, 3H), 7.23-7.26 (m, 2H), 7.07 (d, $^2J_{\text{H-P}} = 30.4$ Hz, 1H), 4.13-4.28 (m, 4H), 3.83 (s, 3H), 1.39 (td, $J = 7.1$ Hz, 0.8 Hz, 6H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 164.09, 143.05 (d, $^3J_{\text{C-P}} = 17.0$ Hz), 130.29, 129.67, 128.56, 128.34, 64.13 (d, $^2J_{\text{C-P}} = 5.8$ Hz), 52.67, 16.11 (d, $^3J_{\text{C-P}} = 6.5$ Hz); $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ -0.93; HRMS for $\text{C}_{13}\text{H}_{20}\text{N}_2\text{O}_5\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 315.1104, found: 315.1102

Methyl (*E*)-2-(2-(diisopropoxyphosphoryl)hydrazineylidene)-2-phenylacetate (3s)⁷



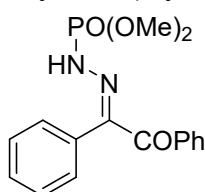
White solid; isolated yield 65% (44 mg). R_f 0.50 (40% EtOAc/hexane); Mp 70-72 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.47-7.53 (m, 3H), 7.23 (d, $J = 6.6$ Hz, 2H), 7.05 (d, $^2J_{\text{H-P}} = 30.4$ Hz, 1H), 4.67-4.75 (m, 2H), 3.83 (s, 3H), 1.39 (d, $J = 6.2$ Hz, 6H), 1.36 (d, 6.2 Hz, 6H); $^{13}\text{C}\{\text{H}\}$ NMR (125 MHz, CDCl_3) δ 164.22, 142.37 (d, $^3J_{\text{C-P}} = 17.0$ Hz), 130.17, 129.64, 128.76, 128.36, 73.05 (d, $^2J_{\text{C-P}} = 5.8$ Hz), 52.53, 23.77 (d, $^3J_{\text{C-P}} = 4.6$ Hz), 23.54 (d, $^3J_{\text{C-P}} = 5.0$ Hz); $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ -2.94; HRMS for $\text{C}_{15}\text{H}_{23}\text{N}_2\text{O}_5\text{PNa}^+$: calcd. $[\text{M}+\text{Na}]^+$: 365.1237, found: 365.1239

Methyl (*E*)-2-(2-(bis(allyloxy)phosphoryl)hydrazineylidene)-2-phenylacetate (3t)⁷



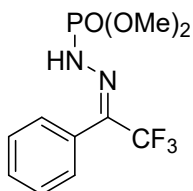
White solid; isolated yield 68% (46 mg). R_f 0.50 (40% EtOAc/hexane); Mp 100-102 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.48-7.53 (m, 3H), 7.23 (d, $J = 6.4$ Hz, 2H), 7.09 (d, $^2J_{\text{H-P}} = 30.8$ Hz, 1H), 5.94-6.03 (m, 2H), 5.40 (d, $J = 17.0$ Hz, 2H), 5.27 (d, $J = 10.2$ Hz, 2H), 4.62-4.67 (m, 4H), 3.83 (s, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (125 MHz, CDCl_3) δ 164.01, 143.29 (d, $^3J_{\text{C-P}} = 16.9$ Hz), 132.33 (d, $^3J_{\text{C-P}} = 5.6$ Hz), 130.32, 129.65, 128.45, 128.32, 118.60, 68.49 (d, $^2J_{\text{C-P}} = 5.7$ Hz), 52.68; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ -0.77; HRMS for $\text{C}_{15}\text{H}_{20}\text{N}_2\text{O}_5\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 339.1104, found: 339.1091

Dimethyl (*E*)-2-(2-(2-oxo-1,2-diphenylethylidene)hydrazineyl)phosphonate (3u)⁷



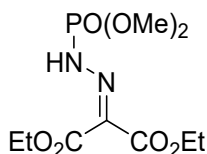
White solid; isolated yield 70% (46 mg). R_f 0.50 (50% EtOAc/hexane); Mp 150-152 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.99 (d, $J = 7.4$ Hz, 2H), 7.37-7.50 (m, 6H), 7.25 (d, $J = 6.8$ Hz, 2H), 7.10 (d, $^2J_{\text{H-P}} = 30.5$ Hz, 1H), 3.73 (d, $^3J_{\text{H-P}} = 11.2$ Hz, 6H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 190.61, 150.25 (d, $^3J_{\text{C-P}} = 16.5$ Hz), 136.62, 132.68, 130.63, 130.30, 129.69, 128.61, 128.43, 127.99, 54.44 (d, $^2J_{\text{C-P}} = 6.1$ Hz); $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.40; HRMS for $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_4\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 333.0999, found: 333.0999

Dimethyl (*E*)-(2-(2,2,2-trifluoro-1-phenylethylidene)hydrazineyl)phosphonate (3v)



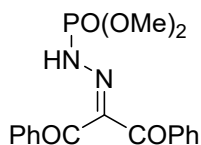
White solid; isolated yield 65% (51 mg). R_f 0.50 (40% EtOAc/hexane); Mp 90-92 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.55 (br s, 3H), 7.32 (br s, 2H), 6.93 (d, $^2J_{\text{H-P}} = 29.4$ Hz, 1H), 3.85 (d, $^3J_{\text{H-P}} = 11.3$ Hz, 6H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 140.78 (dd, $^2J_{\text{C-F}} = 34.9$ Hz, $^3J_{\text{C-P}} = 17.7$ Hz), 131.26, 130.03, 128.40, 125.52, 120.38 (q, $^1J_{\text{C-F}} = 272.4$ Hz), 54.47 (d, $^2J_{\text{C-P}} = 5.9$ Hz); $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 1.36; $^{19}\text{F}\{\text{H}\}$ NMR (376 MHz, CDCl_3) δ -68.06; HRMS for $\text{C}_{10}\text{H}_{13}\text{F}_3\text{N}_2\text{O}_3\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 297.0610, found: 297.0604

Diethyl 2-(2-(dimethoxyphosphoryl)hydrazineylidene)malonate (3w)

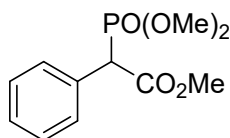


Colorless viscous liquid; isolated yield 56% (33 mg). R_f 0.50 (40% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 10.87 (d, $^2J_{\text{H-P}} = 34.6$ Hz, 1H), 4.31-4.37 (m, 4), 3.86 (d, $^3J_{\text{H-P}} = 11.4$ Hz, 6H), 1.35 (t, $J = 7.0$ Hz, 3H), 1.34 (t, $J = 7.0$ Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 162.26, 160.43, 130.80 (d, $^3J_{\text{C-P}} = 14.8$ Hz), 62.22, 61.93, 54.62 (d, $^2J_{\text{C-P}} = 5.9$ Hz), 13.99, 13.88; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ -0.13; HRMS for $\text{C}_9\text{H}_{18}\text{N}_2\text{O}_7\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 297.0846, found: 297.0830

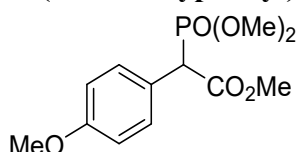
Dimethyl (2-(1,3-dioxo-1,3-diphenylpropan-2-ylidene)hydrazineyl)phosphonate (3x)



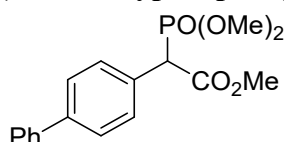
White solid; isolated yield 80% (57 mg). R_f 0.50 (40% EtOAc/hexane); Mp 102-104 °C; ^1H NMR (400 MHz, CDCl_3) δ 10.03 (d, $^2J_{\text{H-P}} = 33.1$ Hz, 1H), 8.10-8.12 (m, 2H), 7.72-7.74 (m, 2H), 7.62-7.65 (m, 1H), 7.55-7.59 (m, 1H), 7.51 (t, $J = 7.7$ Hz, 2H), 7.42 (t, $J = 7.8$ Hz, 2H), 3.83 (d, $^3J_{\text{H-P}} = 11.4$ Hz, 6H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 191.42, 190.06, 143.18 (d, $^3J_{\text{C-P}} = 13.8$ Hz), 135.93, 135.12, 134.19, 133.65, 130.67, 128.88, 128.69, 128.45, 54.67 (d, $^2J_{\text{C-P}} = 5.9$ Hz); $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 0.26; HRMS for $\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_5\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 361.0948, found: 361.0944

Methyl 2-(dimethoxyphosphoryl)-2-phenylacetate (4a)⁸

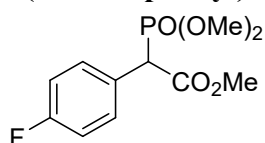
Colorless viscous liquid; isolated yield 55% (40 mg). R_f 0.50 (40% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.49-7.51 (m, 2H), 7.32-7.39 (m, 3H), 4.29 (d, $^2J_{\text{H-P}} = 23.5$ Hz, 1H), 3.77 (s, 3H), 3.73 (d, $^3J_{\text{H-P}} = 11.0$ Hz, 3H), 3.67 (d, $^3J_{\text{H-P}} = 10.9$ Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 168.01 (d, $^2J_{\text{C-P}} = 3.0$ Hz), 130.57 (d, $J_{\text{C-P}} = 8.6$ Hz), 129.56 (d, $J_{\text{C-P}} = 6.2$ Hz), 128.71 (d, $J_{\text{C-P}} = 2.3$ Hz), 128.18 (d, $J_{\text{C-P}} = 2.9$ Hz), 54.08 (d, $^2J_{\text{C-P}} = 6.7$ Hz), 53.70 (d, $^2J_{\text{C-P}} = 7.1$ Hz), 52.95, 51.65 (d, $^1J_{\text{C-P}} = 135.2$ Hz); $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 21.11; HRMS for $\text{C}_{11}\text{H}_{16}\text{O}_5\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 259.0730, found: 259.0730

Methyl 2-(dimethoxyphosphoryl)-2-(4-methoxyphenyl)acetate (4b)

Colorless viscous liquid; isolated yield 60% (35 mg). R_f 0.50 (40% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.40-7.43 (m, 2H), 6.89 (d, $J = 8.5$ Hz, 2H), 4.23 (d, $^2J_{\text{H-P}} = 23.5$ Hz, 1H), 3.80 (s, 3H), 3.76 (s, 3H), 3.73 (d, $^3J_{\text{H-P}} = 11.0$ Hz, 3H), 3.67 (d, $^3J_{\text{H-P}} = 10.8$ Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 168.23 (d, $^2J_{\text{C-P}} = 2.5$ Hz), 159.51 (d, $J_{\text{C-P}} = 2.7$ Hz), 130.68 ($J_{\text{C-P}} = 6.2$ Hz), 122.38 ($J_{\text{C-P}} = 8.7$ Hz), 114.16 ($J_{\text{C-P}} = 2.2$ Hz), 55.26, 54.08 (d, $^2J_{\text{C-P}} = 6.6$ Hz), 53.69 (d, $^2J_{\text{C-P}} = 7.2$ Hz), 52.91, 50.69 (d, $^1J_{\text{C-P}} = 136.3$ Hz); $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 21.43; HRMS for $\text{C}_{12}\text{H}_{18}\text{O}_6\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 289.0836, found: 289.0814

Methyl 2-([1,1'-biphenyl]-4-yl)-2-(dimethoxyphosphoryl)acetate (4c)

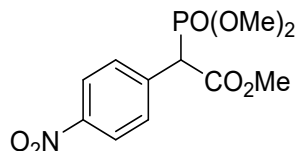
Colorless viscous liquid; isolated yield 52% (35 mg). R_f 0.50 (40% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.55-7.61 (m, 6H), 7.42-7.46 (m, 2H), 7.33-7.37 (m, 1H), 4.34 (d, $^2J_{\text{H-P}} = 23.5$ Hz, 1H), 3.79 (s, 3H), 3.76 (d, $^3J_{\text{H-P}} = 11.0$ Hz, 3H), 3.72 (d, $^3J_{\text{H-P}} = 10.9$ Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 168.01 (d, $^2J_{\text{C-P}} = 3.3$ Hz), 141.07 (d, $J_{\text{C-P}} = 2.9$ Hz), 140.40, 129.94 (d, $J_{\text{C-P}} = 6.4$ Hz), 129.53 (d, $J_{\text{C-P}} = 8.7$ Hz), 128.81, 127.51, 127.41 (d, $J_{\text{C-P}} = 2.2$ Hz), 127.08, 54.13 (d, $^2J_{\text{C-P}} = 6.6$ Hz), 53.78 (d, $^2J_{\text{C-P}} = 7.0$ Hz), 53.02, 51.32 (d, $^1J_{\text{C-P}} = 135.2$ Hz); $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 21.08; HRMS for $\text{C}_{17}\text{H}_{20}\text{O}_5\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 335.1043, found: 335.1029

Methyl 2-(dimethoxyphosphoryl)-2-(4-fluorophenyl)acetate (4d)

Colorless viscous liquid; isolated yield 48% (26 mg). R_f 0.50 (40% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.47-7.51 (m, 2H), 7.05 (t, $J = 8.5$ Hz, 2H), 4.27 (d, $^2J_{\text{H-P}} = 23.8$ Hz, 1H), 3.77 (s, 3H), 3.74 (d, $^3J_{\text{H-P}} = 11.0$ Hz, 3H), 3.68 (d, $^3J_{\text{H-P}} = 10.9$ Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR

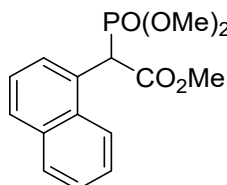
(100 MHz, CDCl₃) δ 167.89 (d, ²J_{C-P} = 4.0 Hz), 162.64 (d, ¹J_{C-F} = 249.2 Hz), 131.30 (dd, ³J_{C-F} = 7.9 Hz, J_{C-P} = 6.7 Hz), 126.39 (dd, ⁴J_{C-F} = 3.0 Hz, J_{C-P} = 8.5 Hz), 115.66 (dd, ²J_{C-F} = 21.3 Hz, J_{C-P} = 2.2 Hz), 54.06 (d, ²J_{C-P} = 6.6 Hz), 53.74 (d, ²J_{C-P} = 7.0 Hz), 53.02, 50.75 (d, ¹J_{C-P} = 135.4 Hz); ³¹P{H} NMR (162 MHz, CDCl₃) δ 20.84 (d, J_{P-F} = 5.0 Hz); ¹⁹F{H} NMR (376 MHz, CDCl₃) δ -113.96 (d, J_{F-P} = 4.4 Hz); HRMS for C₁₁H₁₅FO₅P⁺: calcd. [M+H]⁺: 277.0636, found: 277.0627

Methyl 2-(dimethoxyphosphoryl)-2-(4-nitrophenyl)acetate (4e)⁸



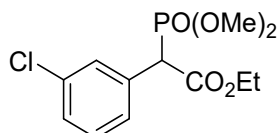
Colorless viscous liquid; isolated yield 45% (27 mg). *R_f* 0.50 (40% EtOAc/hexane); ¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 8.6 Hz, 2H), 7.70 (dd, *J* = 8.9 Hz, 2.2 Hz, 2H), 4.41 (d, ²J_{H-P} = 24.1 Hz, 1H), 3.81 (s, 3H), 3.77 (d, ³J_{H-P} = 11.1 Hz, 3H), 3.73 (d, ³J_{H-P} = 11.0 Hz, 3H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 166.98 (d, ²J_{C-P} = 4.4 Hz), 147.73 (d, J_{C-P} = 3.2 Hz), 138.04 (J_{C-P} = 8.5 Hz), 130.62 (J_{C-P} = 6.3 Hz), 123.72 (J_{C-P} = 2.2 Hz), 54.19 (d, ²J_{C-P} = 6.6 Hz), 53.99 (d, ²J_{C-P} = 6.9 Hz), 53.33, 51.44 (d, ¹J_{C-P} = 133.4 Hz); ³¹P{H} NMR (162 MHz, CDCl₃) δ 19.43; HRMS for C₁₁H₁₅NO₇P⁺: calcd. [M+H]⁺: 304.0581, found: 304.0576

Methyl 2-(dimethoxyphosphoryl)-2-(naphthalen-1-yl)acetate (4f)



Colorless viscous liquid; isolated yield 50% (31 mg). *R_f* 0.50 (40% EtOAc/hexane); ¹H NMR (400 MHz, CDCl₃) δ 8.06 (d, *J* = 8.5 Hz, 1H), 8.00 (dd, *J* = 7.1 Hz, 2.5 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 8.2 Hz, 1H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.49-7.53 (m, 2H), 5.17 (d, ²J_{H-P} = 24.1 Hz, 1H), 3.74 (s, 3H), 3.73 (d, ³J_{H-P} = 11.0 Hz, 3H), 3.58 (d, ³J_{H-P} = 10.8 Hz, 3H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 168.44 (d, ²J_{C-P} = 2.4 Hz), 134.03, 131.51 (J_{C-P} = 6.8 Hz), 129.10, 128.89 (d, J_{C-P} = 2.9 Hz), 127.95 (J_{C-P} = 6.3 Hz), 126.81, 126.68 (d, J_{C-P} = 7.9 Hz), 125.81, 125.40 (d, J_{C-P} = 3.0 Hz), 122.77, 54.01 (d, ²J_{C-P} = 6.6 Hz), 53.69 (d, ²J_{C-P} = 7.0 Hz), 53.04, 46.25 (d, ¹J_{C-P} = 139.3 Hz); ³¹P{H} NMR (162 MHz, CDCl₃) δ 21.67; HRMS for C₁₅H₁₈O₅P⁺: calcd. [M+H]⁺: 309.0886, found: 309.0889

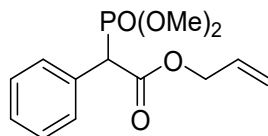
Ethyl 2-(3-chlorophenyl)-2-(dimethoxyphosphoryl)acetate (4g)



Colorless viscous liquid; isolated yield 45% (28 mg). *R_f* 0.50 (40% EtOAc/hexane); ¹H NMR (400 MHz, CDCl₃) δ 7.43 (s, 1H), 7.31-7.34 (m, 1H), 7.20-7.21 (m, 2H), 4.08-4.20 (m, 3H), 3.66 (d, ³J_{H-P} = 11.0 Hz, 3H), 3.63 (d, ³J_{H-P} = 11.0 Hz, 3H), 1.19 (t, 3H); ¹³C{H} NMR (100 MHz, CDCl₃) δ 166.70 (d, ²J_{C-P} = 3.6 Hz), 134.09 (d, J_{C-P} = 2.7 Hz), 132.45 (d, J_{C-P} = 8.5 Hz), 129.55 (J_{C-P} = 2.3 Hz), 129.34 (J_{C-P} = 6.4 Hz), 128.03 (J_{C-P} = 2.8 Hz), 127.59 (J_{C-P} = 6.3 Hz), 61.90, 53.79 (d, ²J_{C-P} = 6.6 Hz), 53.51 (d, ²J_{C-P} = 7.1 Hz), 51.05 (d, ¹J_{C-P} = 134.4 Hz),

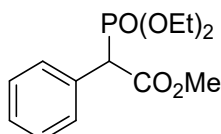
13.71; $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 20.59; HRMS for $\text{C}_{12}\text{H}_{17}\text{ClO}_5\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 307.0497, found: 307.0491

Allyl 2-(dimethoxyphosphoryl)-2-phenylacetate (4h)⁸



Colorless viscous liquid; isolated yield 66% (38 mg). R_f 0.50 (50% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.42-7.44 (m, 2H), 7.23-7.30 (m, 3H), 5.77-5.87 (m, 1H), 5.24 (dd, $J = 17.2$ Hz, 1.4 Hz, 1H), 5.15 (dd, $J = 10.4$ Hz, 1.1 Hz, 1H), 4.53-4.63 (m, 2H), 4.24 (d, $^2J_{\text{H-P}} = 23.5$ Hz, 1H), 3.64 (d, $^3J_{\text{H-P}} = 11.0$ Hz, 3H), 3.59 (d, $^3J_{\text{H-P}} = 10.9$ Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 167.18 (d, $^2J_{\text{C-P}} = 3.1$ Hz), 131.41, 130.55 ($J_{\text{C-P}} = 8.6$ Hz), 129.58 ($J_{\text{C-P}} = 6.5$ Hz), 128.68 ($J_{\text{C-P}} = 2.2$ Hz), 128.15 ($J_{\text{C-P}} = 3.0$ Hz), 118.76, 66.38, 54.03 (d, $^2J_{\text{C-P}} = 6.6$ Hz), 53.69 (d, $^2J_{\text{C-P}} = 6.8$ Hz), 51.75 (d, $^1J_{\text{C-P}} = 134.9$ Hz); $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 21.14; HRMS for $\text{C}_{13}\text{H}_{18}\text{O}_5\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 285.0886 found: 285.0882

Methyl 2-(diethoxyphosphoryl)-2-phenylacetate (4i)⁸



Colorless viscous liquid; isolated yield 60% (35 mg). R_f 0.50 (40% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 7.43-7.46 (m, 2H), 7.24-7.30 (m, 3H), 4.19 (d, $^2J_{\text{H-P}} = 23.6$ Hz, 1H), 3.87-4.06 (m, 4H), 3.69 (s, 3H), 1.19 (t, $J = 7.1$ Hz, 3H), 1.12 (t, $J = 7.1$ Hz, 3H); $^{13}\text{C}\{\text{H}\}$ NMR (100 MHz, CDCl_3) δ 168.16 (d, $^2J_{\text{C-P}} = 3.4$ Hz), 130.86 (d, $J_{\text{C-P}} = 8.3$ Hz), 129.64 ($J_{\text{C-P}} = 6.5$ Hz), 128.56 ($J_{\text{C-P}} = 2.3$ Hz), 128.02 ($J_{\text{C-P}} = 2.9$ Hz), 63.49 (d, $^2J_{\text{C-P}} = 6.8$ Hz), 63.16 (d, $^2J_{\text{C-P}} = 7.0$ Hz), 52.80, 52.13 (d, $^1J_{\text{C-P}} = 133.6$ Hz), 16.29 (d, $^3J_{\text{C-P}} = 6.4$ Hz), 16.23 (d, $^3J_{\text{C-P}} = 6.4$ Hz); $^{31}\text{P}\{\text{H}\}$ NMR (162 MHz, CDCl_3) δ 18.54; HRMS for $\text{C}_{13}\text{H}_{20}\text{O}_5\text{P}^+$: calcd. $[\text{M}+\text{H}]^+$: 287.1043 found: 287.1043

6. References

1. S. Xia, Y. Jian, L. Zhang, C. Zhang, Y. An and Y. Wang, *RSC Adv.*, 2023, **13**, 14501-14505.
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3. X. Lv, Z. Kang, D. Xing, and W. Hu, *Org. Lett.*, 2018, **20**, 4843-4847.
4. J.-L. Zhu and Y.-T. Tsai, *J. Org. Chem.*, 2021, **86**, 813-828.
5. CrysAlisPro, Oxford Diffraction /Agilent Technologies UK Ltd, Yarnton, England
6. G. M. Sheldrick, *Acta Crystallogr. Sect. A*, 2008, **64**, 112-122.
7. H. Jiang, H. Jin, A. Abdukader, A. Lin, Y. Chenga and C. Zhu, *Org. Biomol. Chem.*, 2013, **11**, 3612-3615.
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7. Copies of ^1H , $^{13}\text{C}\{\text{H}\}$, $^{31}\text{P}\{\text{H}\}$, $^{19}\text{F}\{\text{H}\}$ NMR Spectra & ESMS of crude 4a

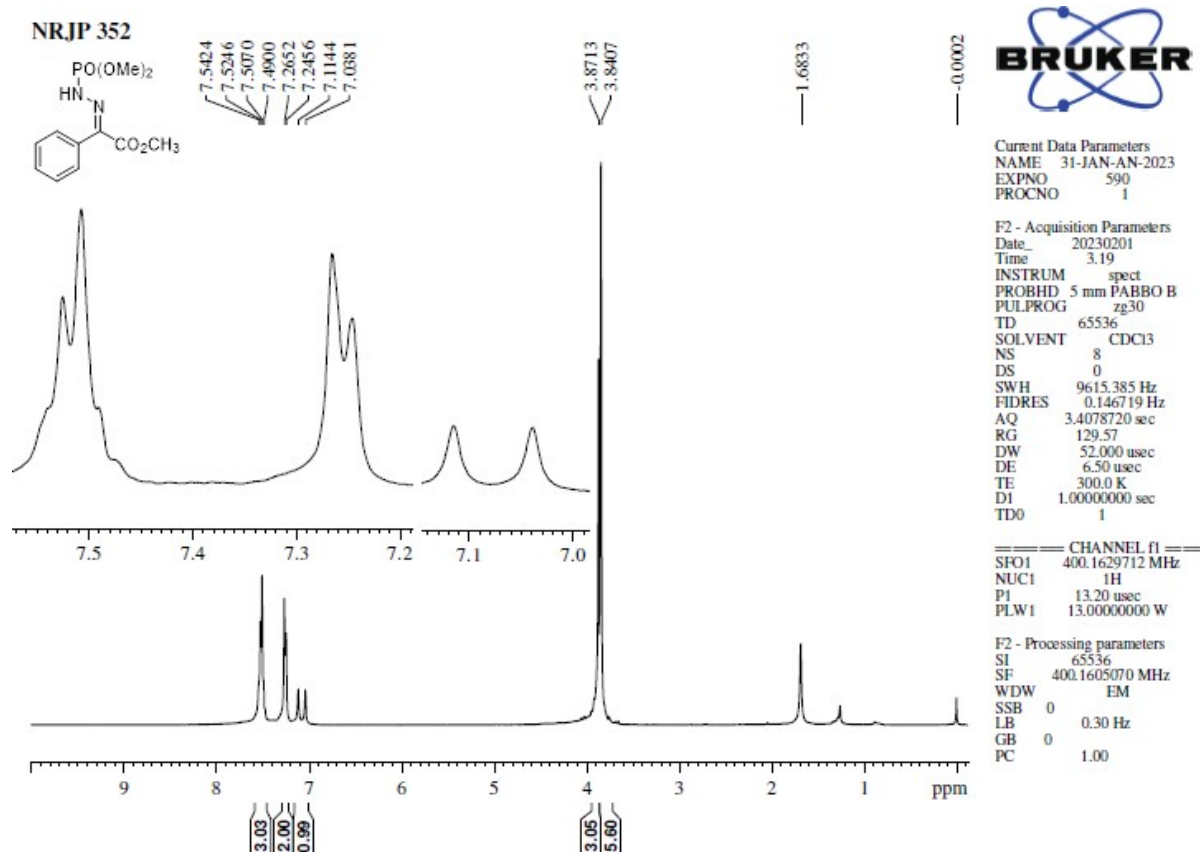


Figure S3: ^1H NMR spectrum of 3a

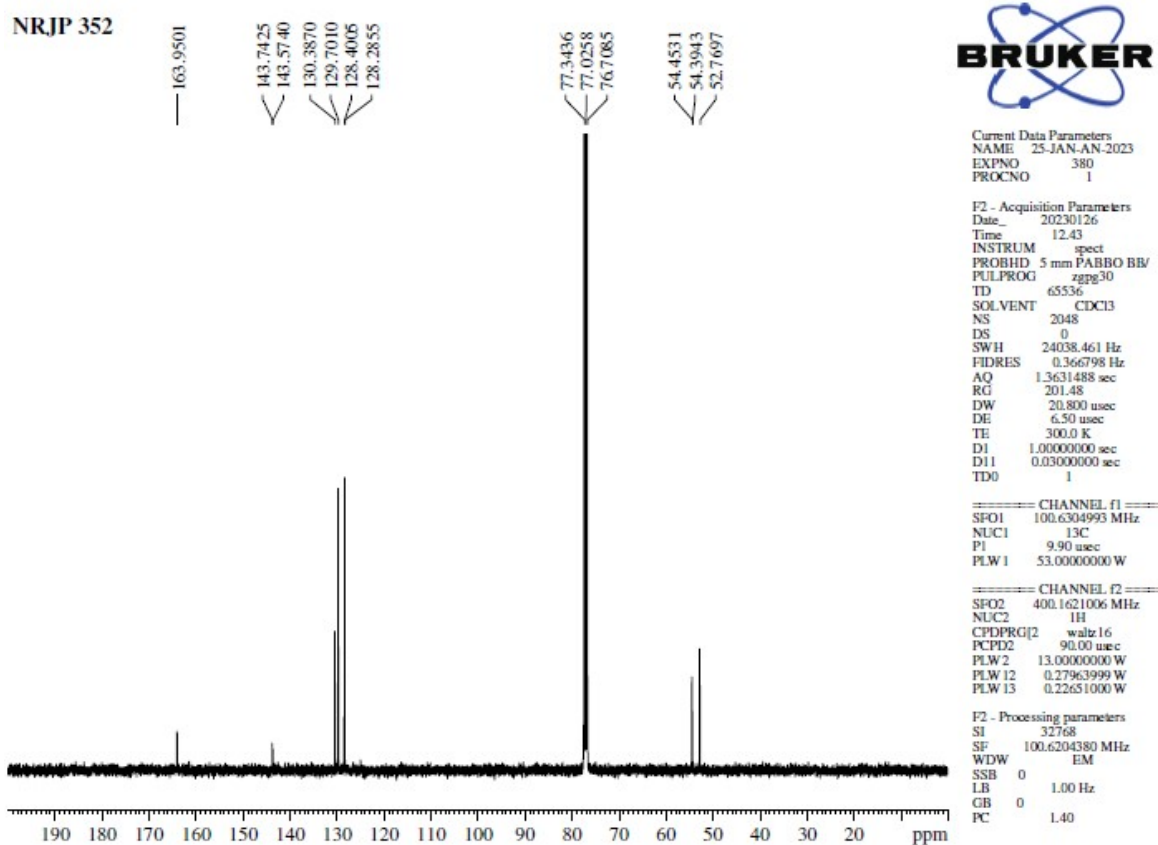
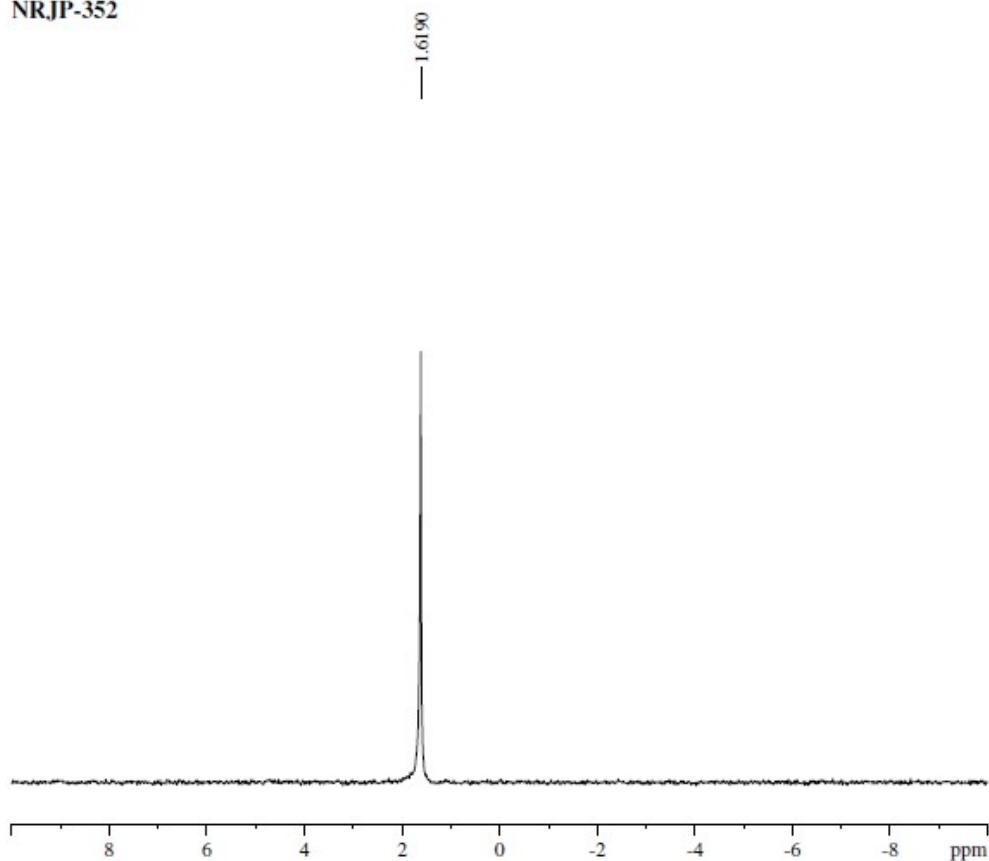


Figure S4: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3a

NRJP-352



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 PROCNO 1

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 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
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 D1 2.00000000 sec
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 TD0 1

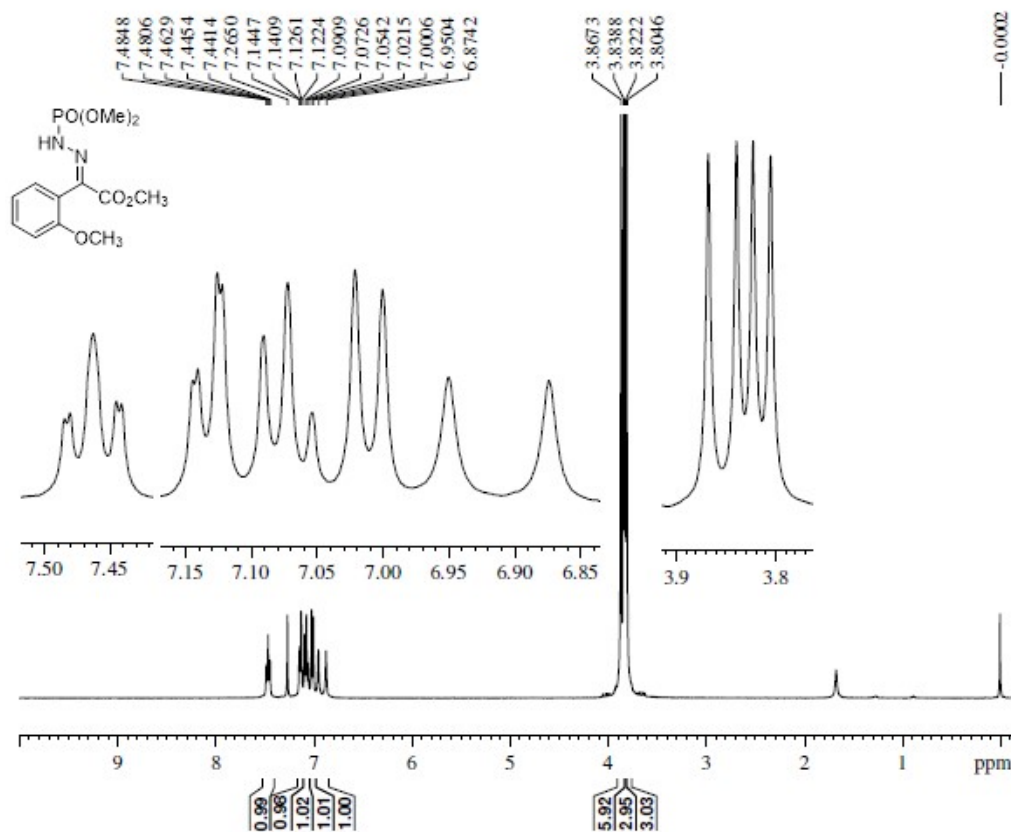
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 PLW1 12.00000000 W

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 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S5: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3a

NRJP-386



Current Data Parameters
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 PROCNO 1

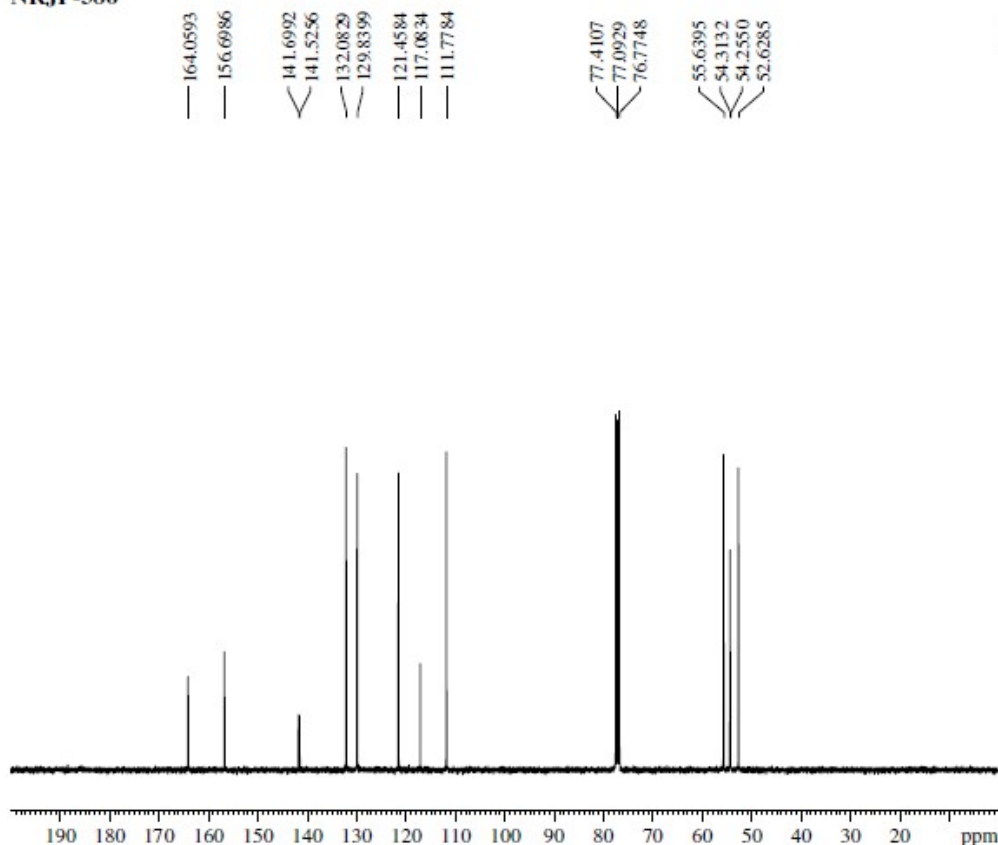
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 SOLVENT CDCl3
 NS 8
 DS 0
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 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 129.57
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605074 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S6: ^1H NMR spectrum of 3b

NRJP-386



Current Data Parameters
 NAME 29-Mar-AN-2023
 EXPNO 400
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230330
 Time 5.23
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 D11 0.0300000 sec
 TDO 1

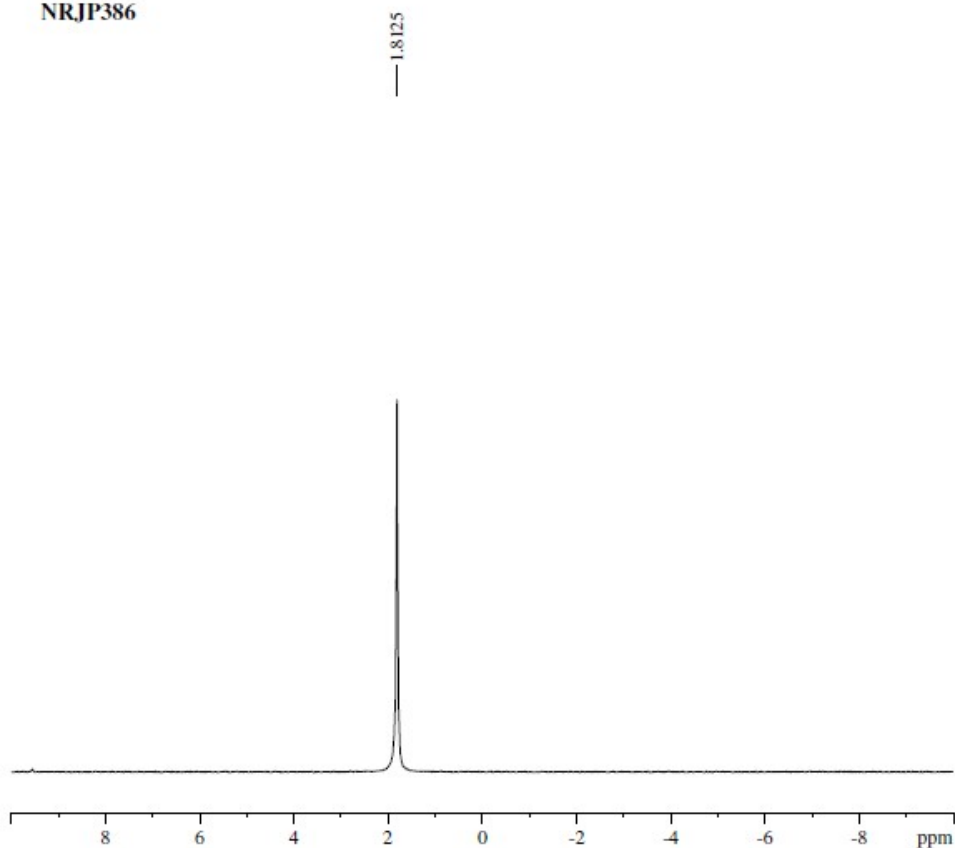
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 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S7: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3b

NRJP386



Current Data Parameters
 NAME 27-MAR-AN-2023
 EXPNO 380
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230327
 Time 19.48
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

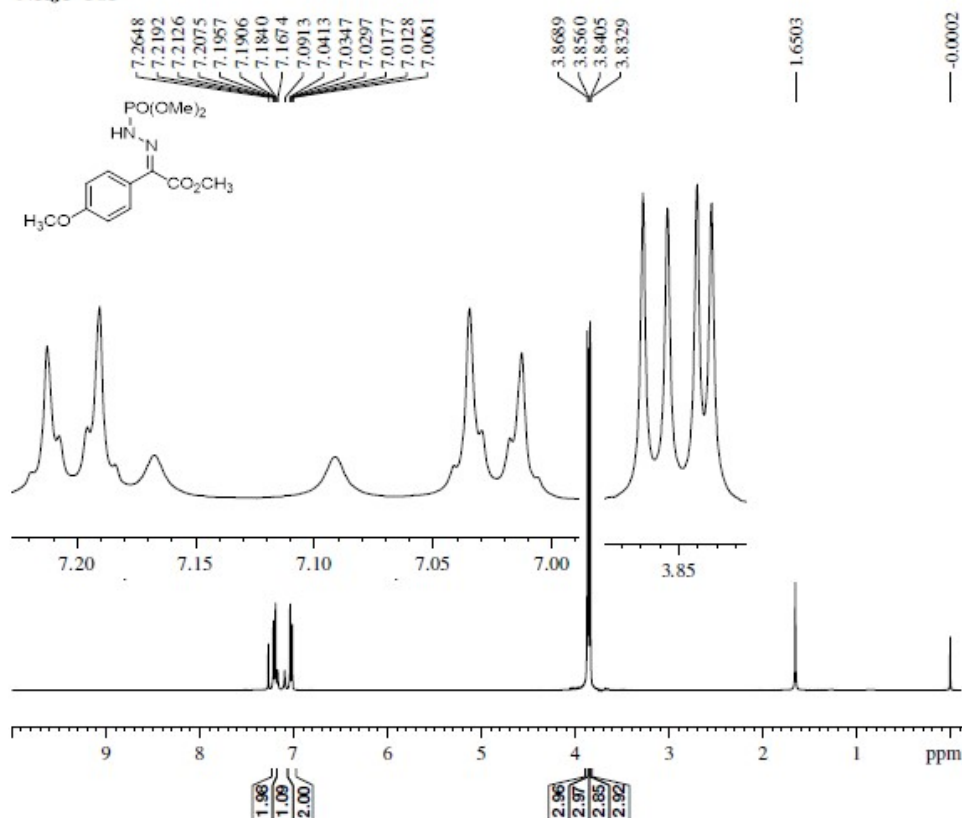
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 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S8: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3b

NRJP-385



Current Data Parameters
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 PROCNO 1

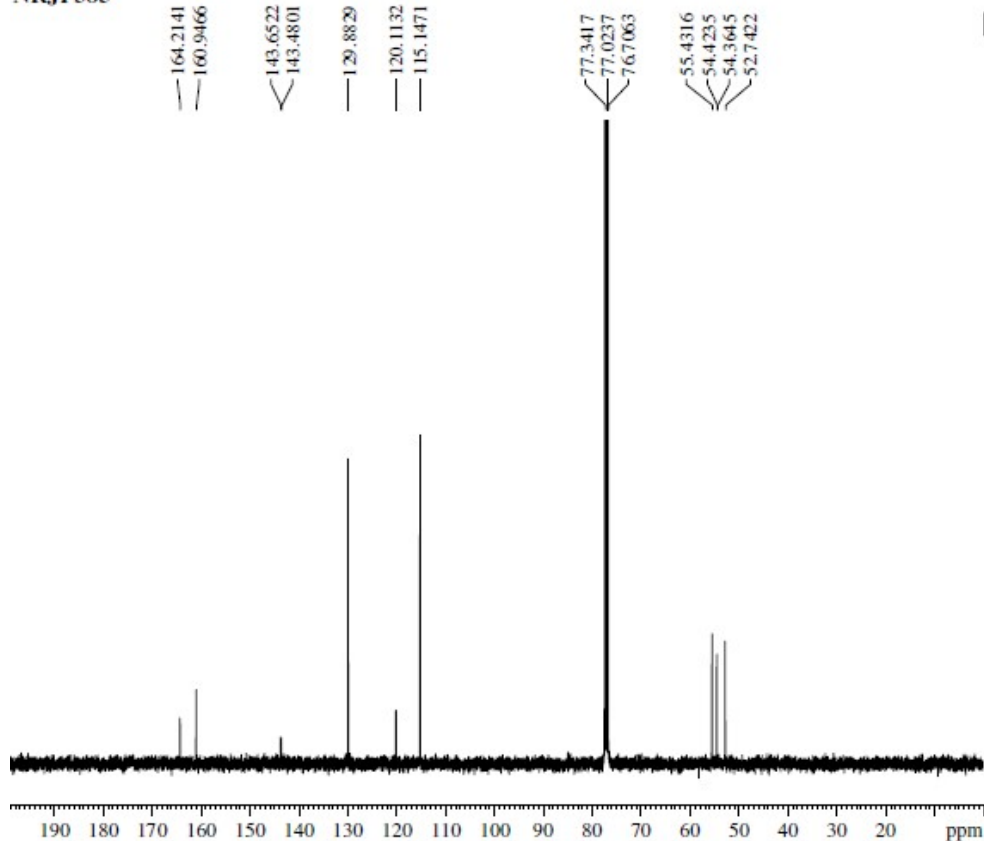
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 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 145.29
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
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 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605078 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S9: ¹H NMR spectrum of 3c

NRJP385



Current Data Parameters
 NAME 27-MAR-AN-2023
 EXPNO 400
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230328
 Time 6.39
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

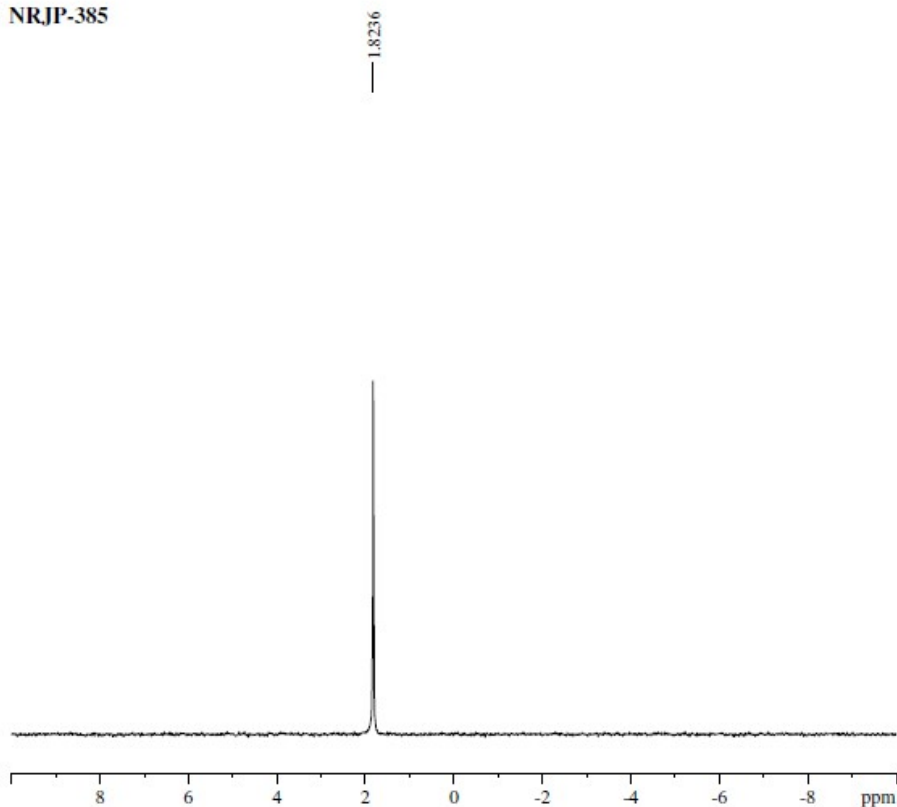
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 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
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 SSB 0
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 GB 0
 PC 1.40

Figure S10: ¹³C{H} NMR spectrum of 3c

NRJP-385



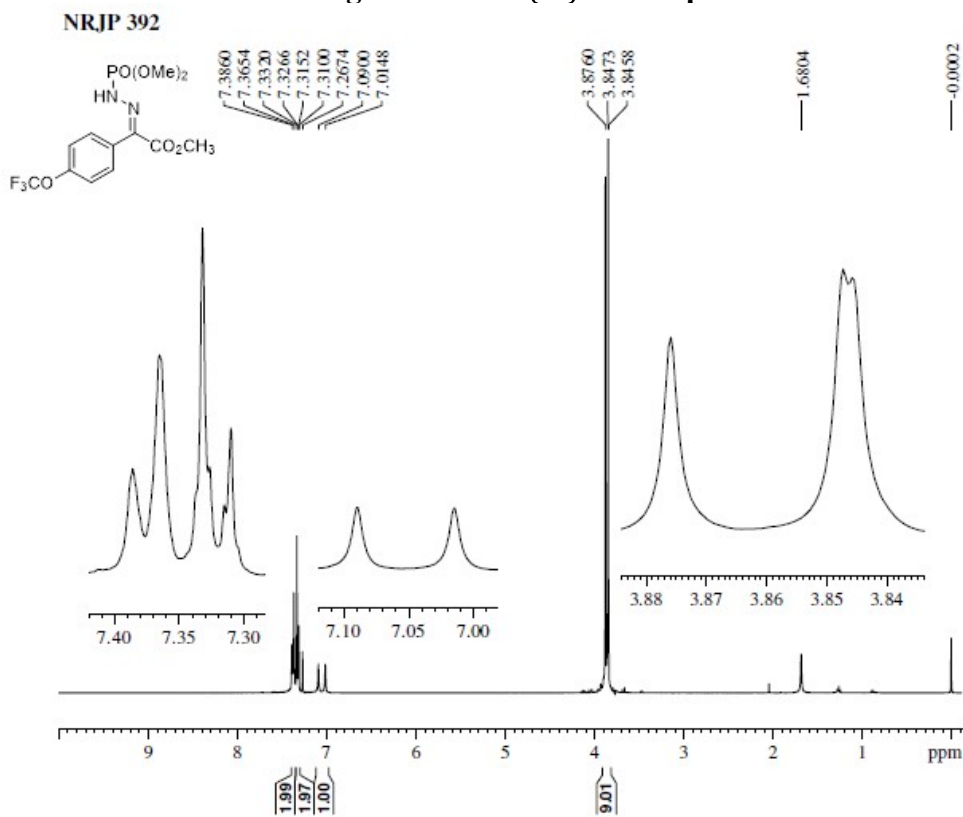
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 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

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 NUC1 31P
 P1 15.00 usec
 PLW1 12.0000000 W

==== CHANNEL f2 ====
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 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
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 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S11: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3c

Current Data Parameters
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 PROCNO 1

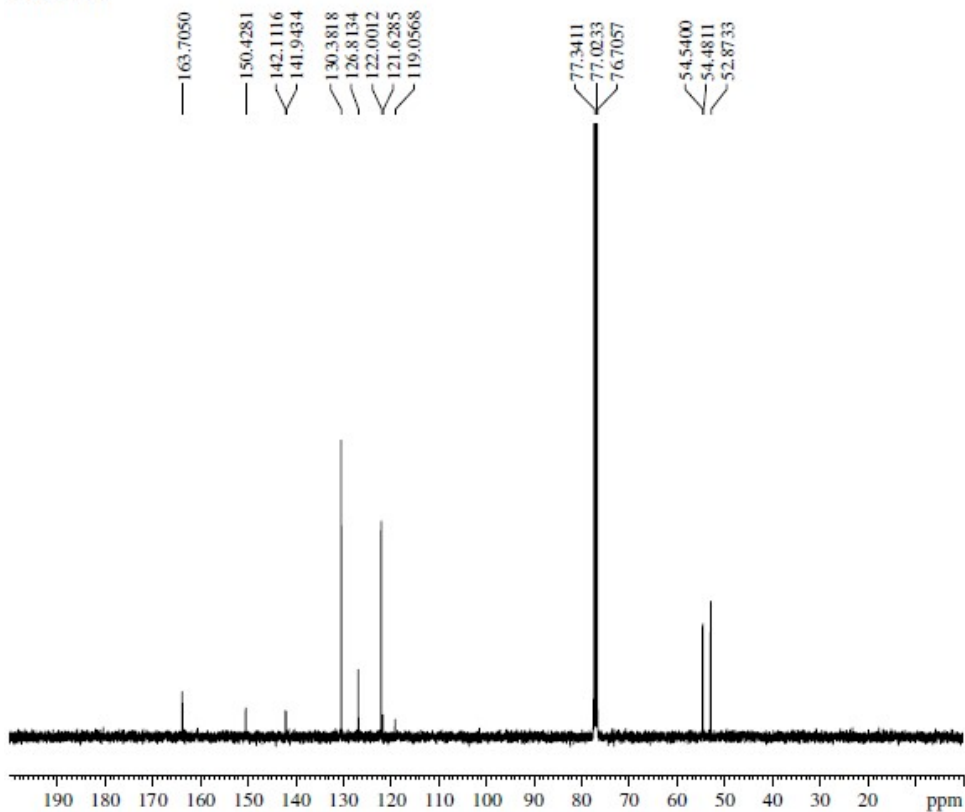
F2 - Acquisition Parameters
 Date_ 20230406
 Time 1.23
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 129.57
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.0000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605068 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S12: ^1H NMR spectrum of 3d

NRJP 392



Current Data Parameters
 NAME 05-APR-AN-2023
 EXPNO 450
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230406
 Time 1.49
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TDO 1

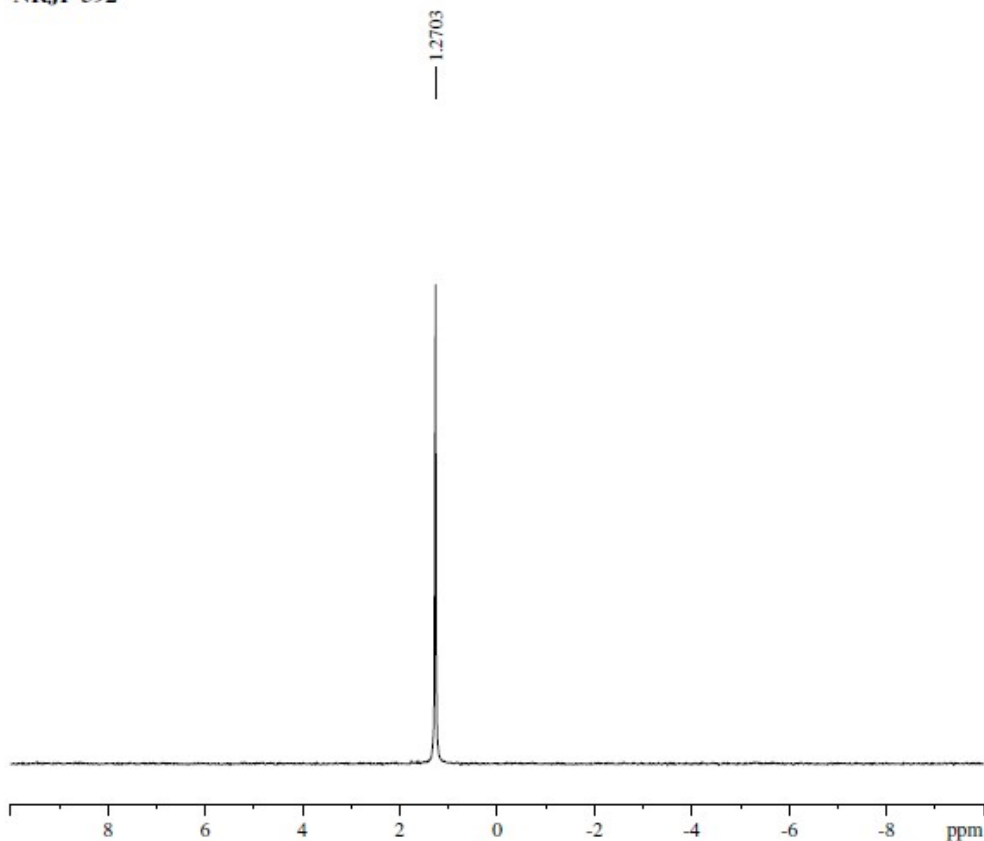
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S13: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3d

NRJP 392



Current Data Parameters
 NAME 05-APR-AN-2023
 EXPNO 460
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230406
 Time 1.54
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

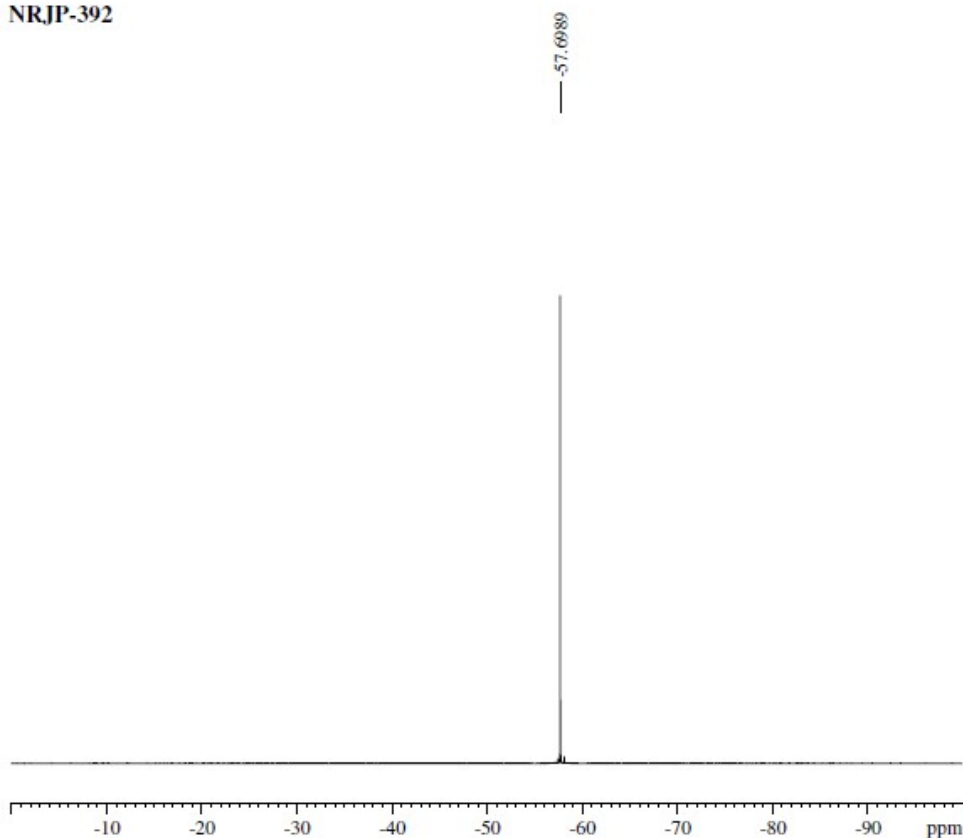
==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

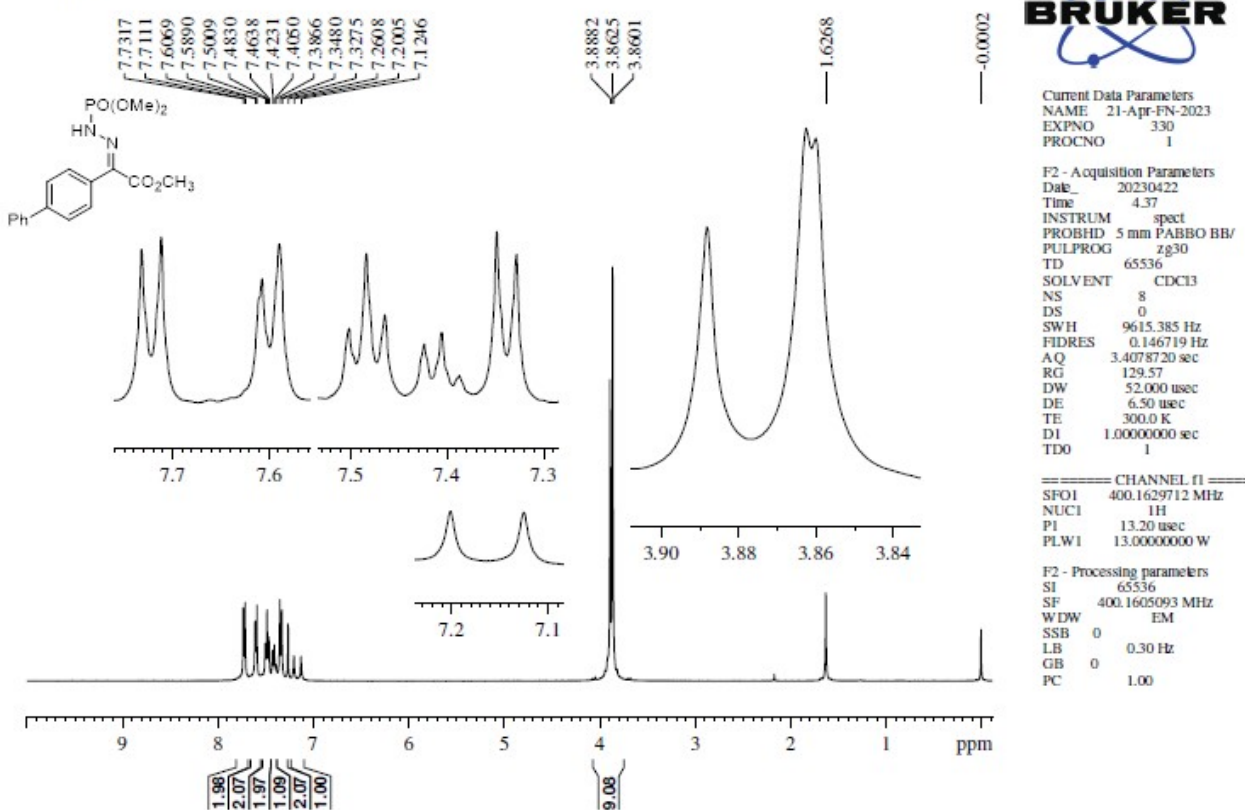
F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S14: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3d

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Figure S15: $^{19}\text{F}\{\text{H}\}$ NMR spectrum of 3d

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Figure S16: ^1H NMR spectrum of 3e

NRJP 39 1

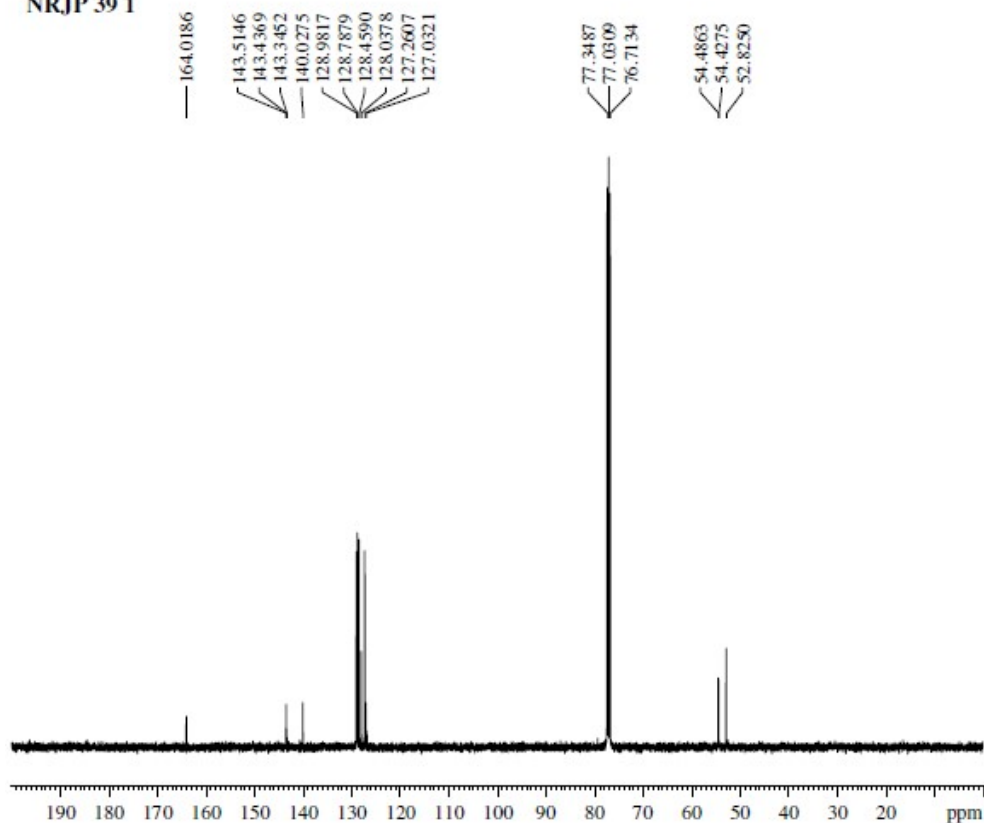


Figure S17: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3e



Current Data Parameters
 NAME 06-Apr-AN-2023
 EXPNO 360
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20230407
 Time 12.31
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 100.6304993 MHz
 NUC1 ^{13}C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

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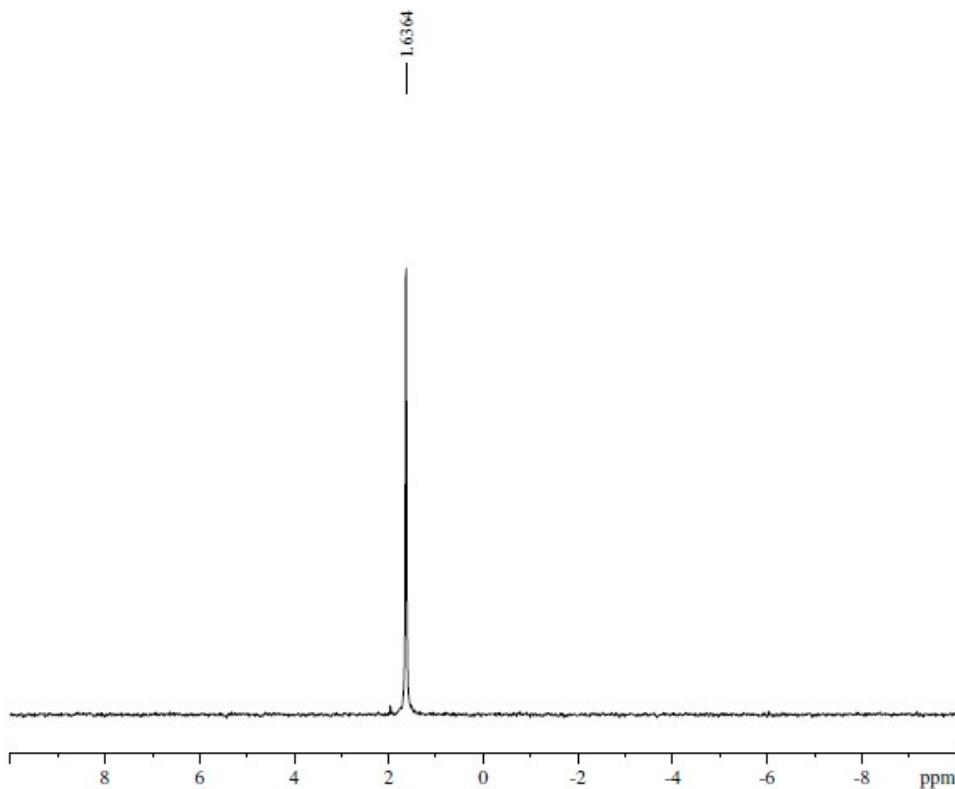


Figure S18: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3e



Current Data Parameters
 NAME 25-Apr-FN-2023
 EXPNO 410
 PROCNO 1

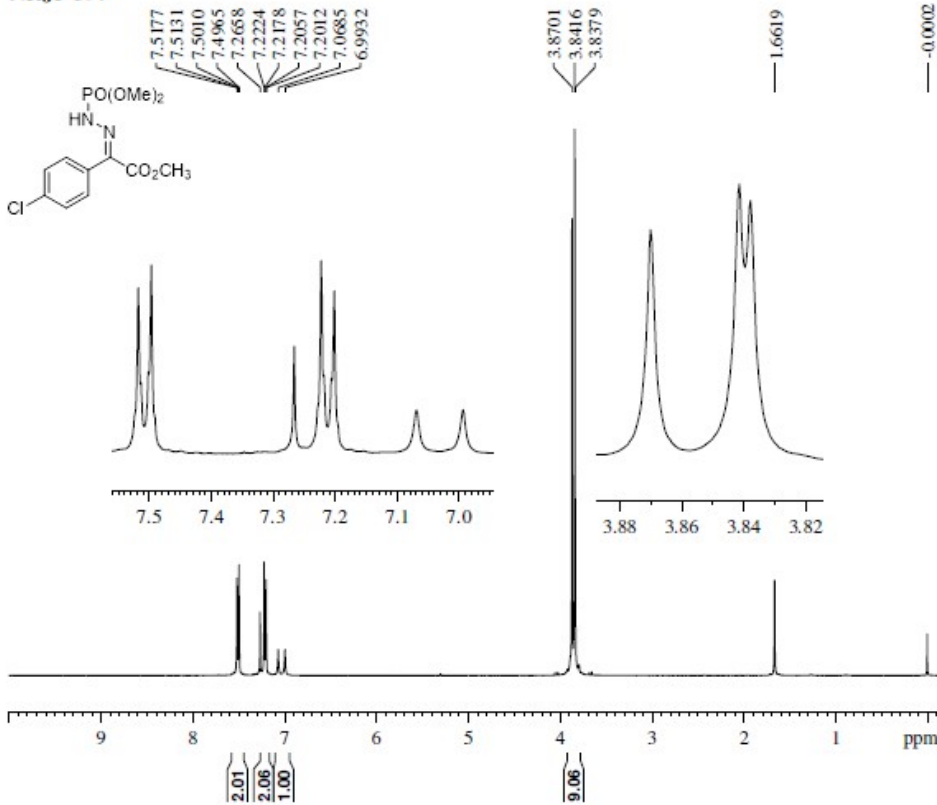
F2 - Acquisition Parameters
 Date_ 20230425
 Time 11.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 161.9798402 MHz
 NUC1 ^{31}P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP 397



Current Data Parameters
 NAME 18-Apr-AN-2023
 EXPNO 450
 PROCNO 1

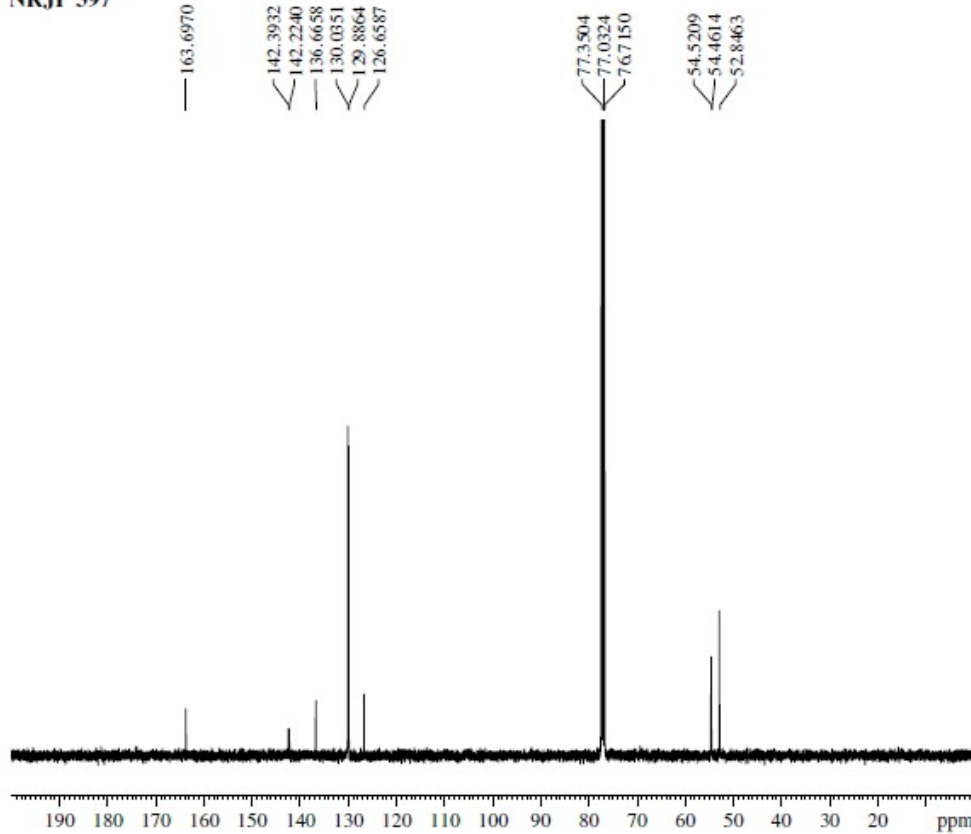
F2 - Acquisition Parameters
 Date_ 20230419
 Time 9.24
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 A Q 3.4078720 sec
 RG 145.29
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605073 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S19: ¹H NMR spectrum of 3f

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Current Data Parameters
 NAME 18-Apr-AN-2023
 EXPNO 470
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230419
 Time 2.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 A Q 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TDO 1

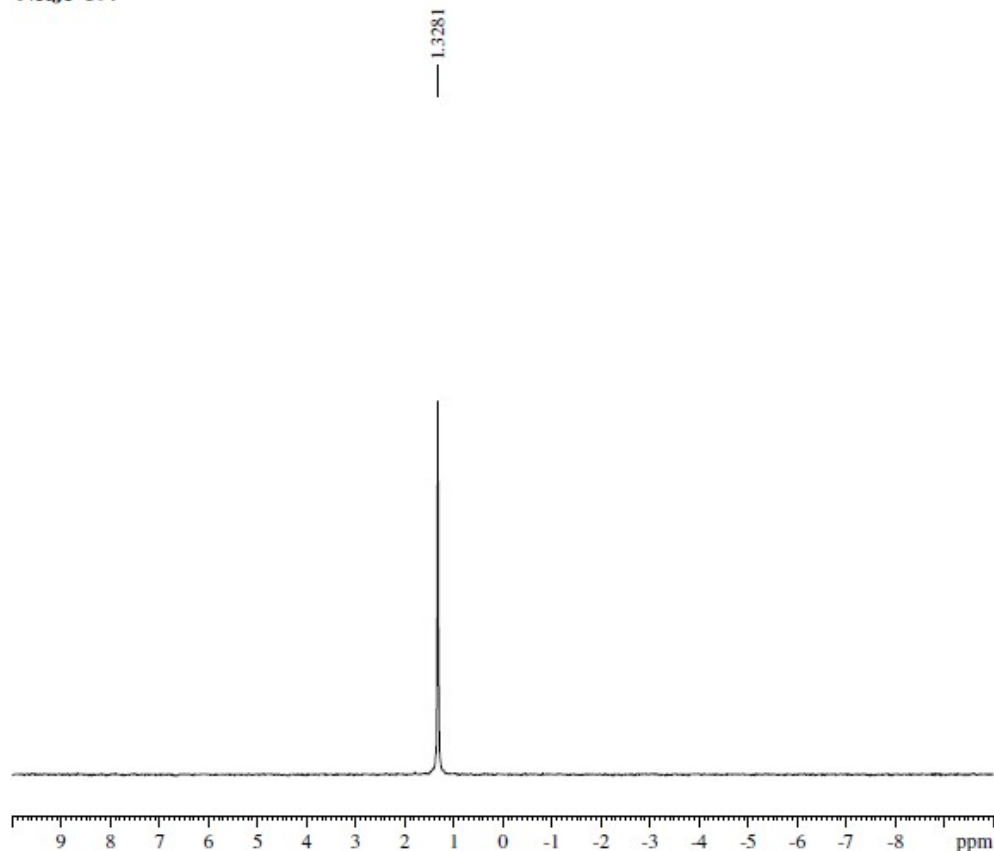
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S20: ¹³C{¹H} NMR spectrum of 3f

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Figure S21: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3f

Current Data Parameters
 NAME 18-Apr-AN-2023
 EXPNO 460
 PROCNO 1

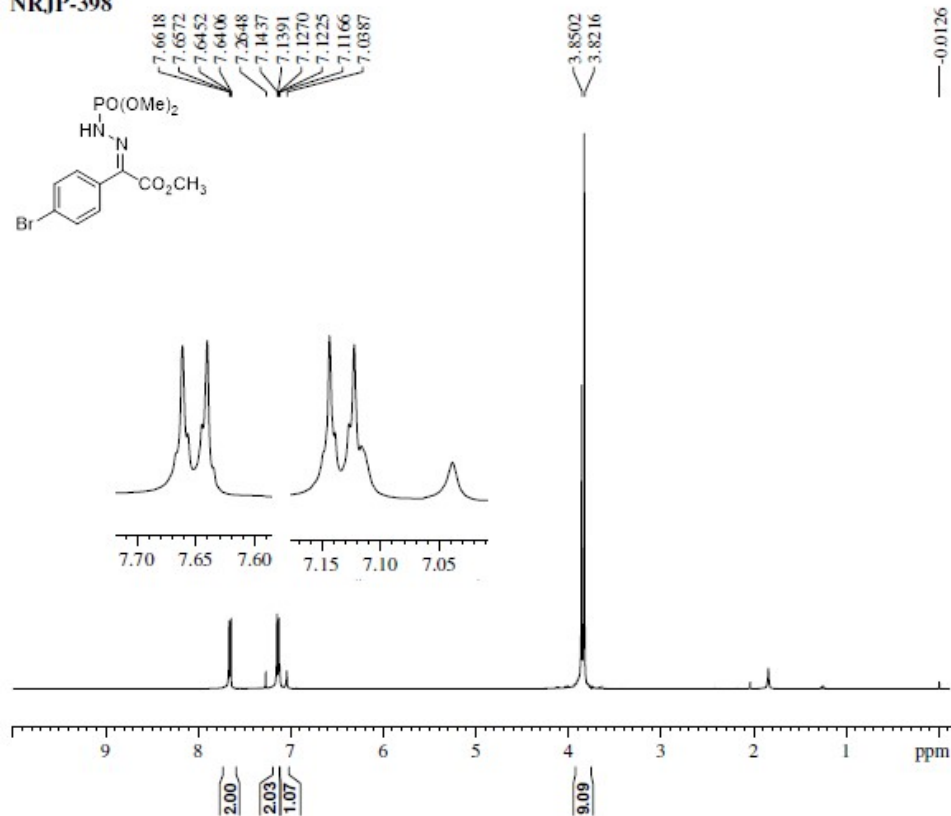
F2 - Acquisition Parameters
 Date_ 20230419
 Time 9.26
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 ^{31}P
 P1 15.00 usec
 PLW1 12.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

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Figure S22: ^1H NMR spectrum of 3g

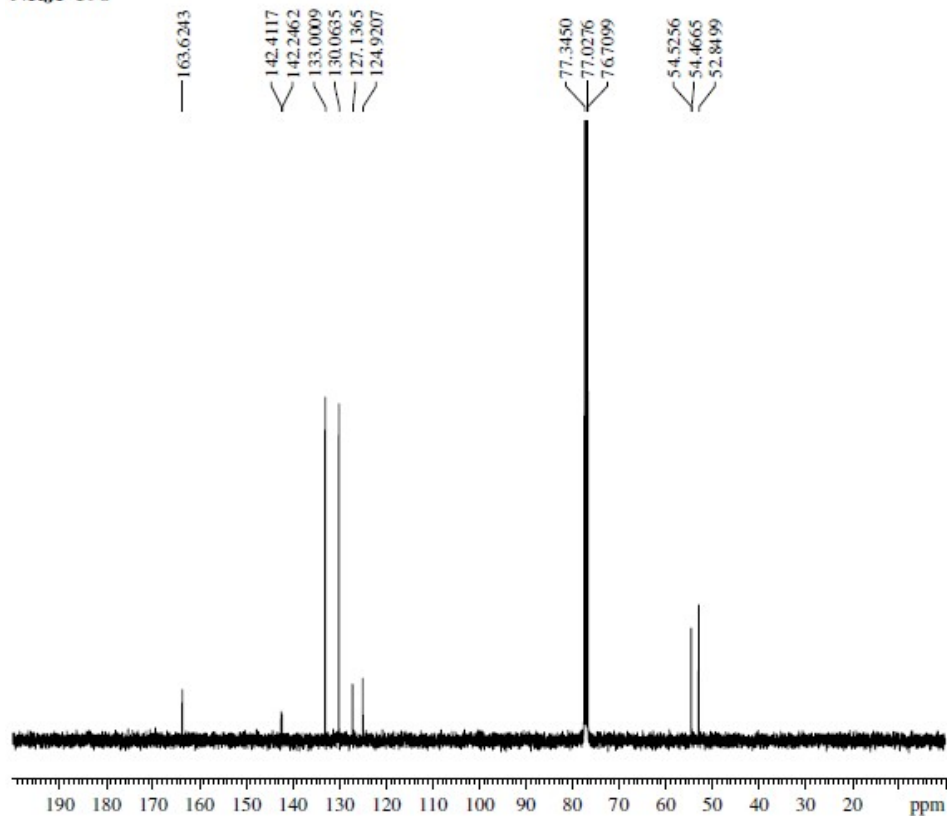
Current Data Parameters
 NAME 14-JUL-AN-2023
 EXPNO 430
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230715
 Time 11.26
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 89.7
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 ^1H
 P1 13.20 usec
 PLW1 13.0000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605077 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NRJP 398

Figure S23: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3g

Current Data Parameters
NAME 17-Apr-AN-2023
EXPNO 390
PROCNO 1

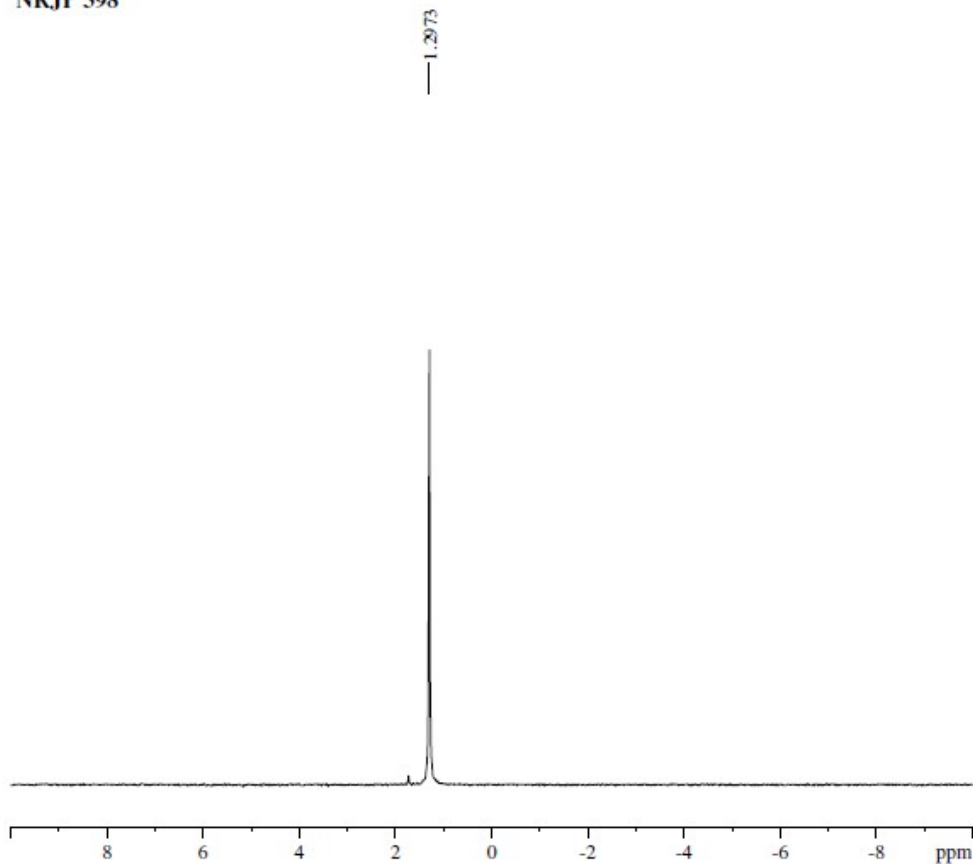
F2 - Acquisition Parameters
Date_ 20230418
Time 0.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 201.48
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.0000000 sec
D11 0.0300000 sec
TDO 1

==== CHANNEL f1 ====
SFO1 100.6304993 MHz
NUC1 13C
P1 9.90 usec
PLW1 53.0000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 13.0000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 100.6204380 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

NRJP 398

Figure S24: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3g

Current Data Parameters
NAME 17-Apr-AN-2023
EXPNO 380
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230418
Time 0.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 64102.563 Hz
FIDRES 0.978127 Hz
AQ 0.5111808 sec
RG 201.48
DW 7.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.0000000 sec
D11 0.0300000 sec
TDO 1

==== CHANNEL f1 ====
SFO1 161.9798402 MHz
NUC1 31P
P1 15.00 usec
PLW1 12.0000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 13.0000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 161.9879400 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

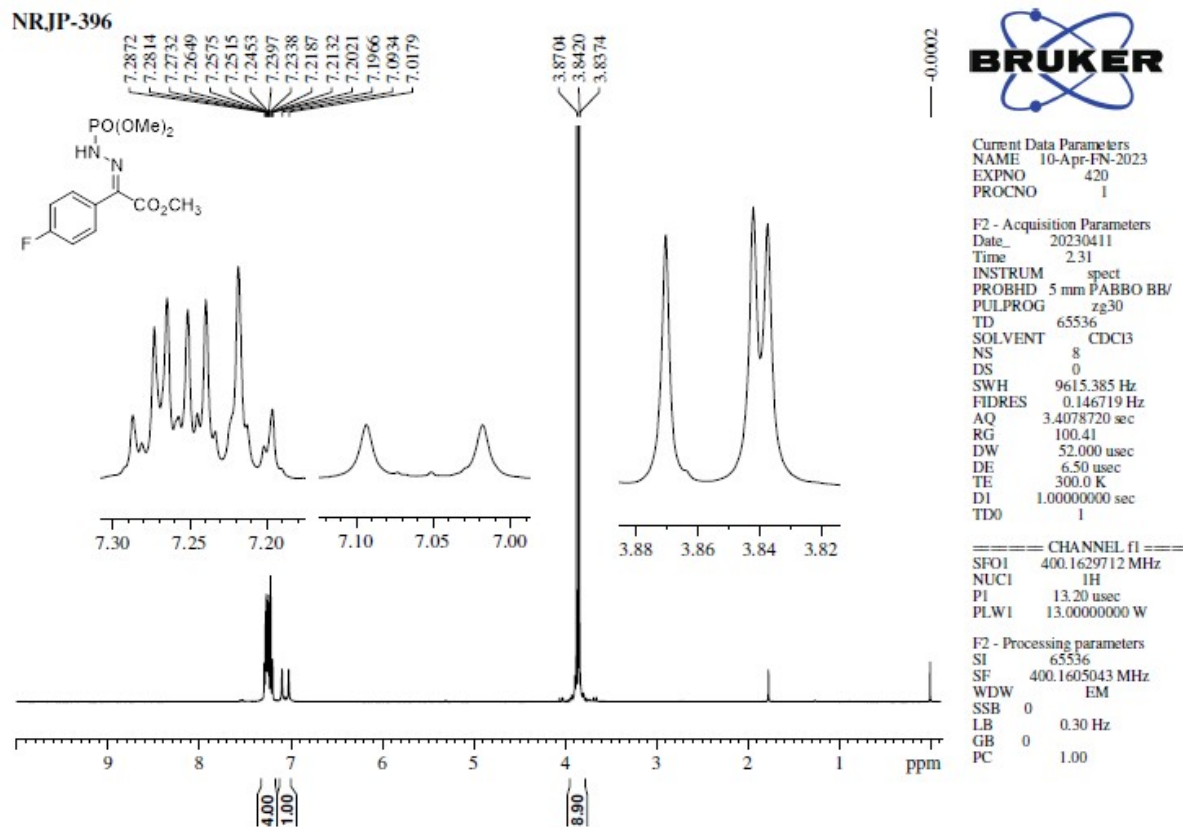


Figure S25: ^1H NMR spectrum of 3h

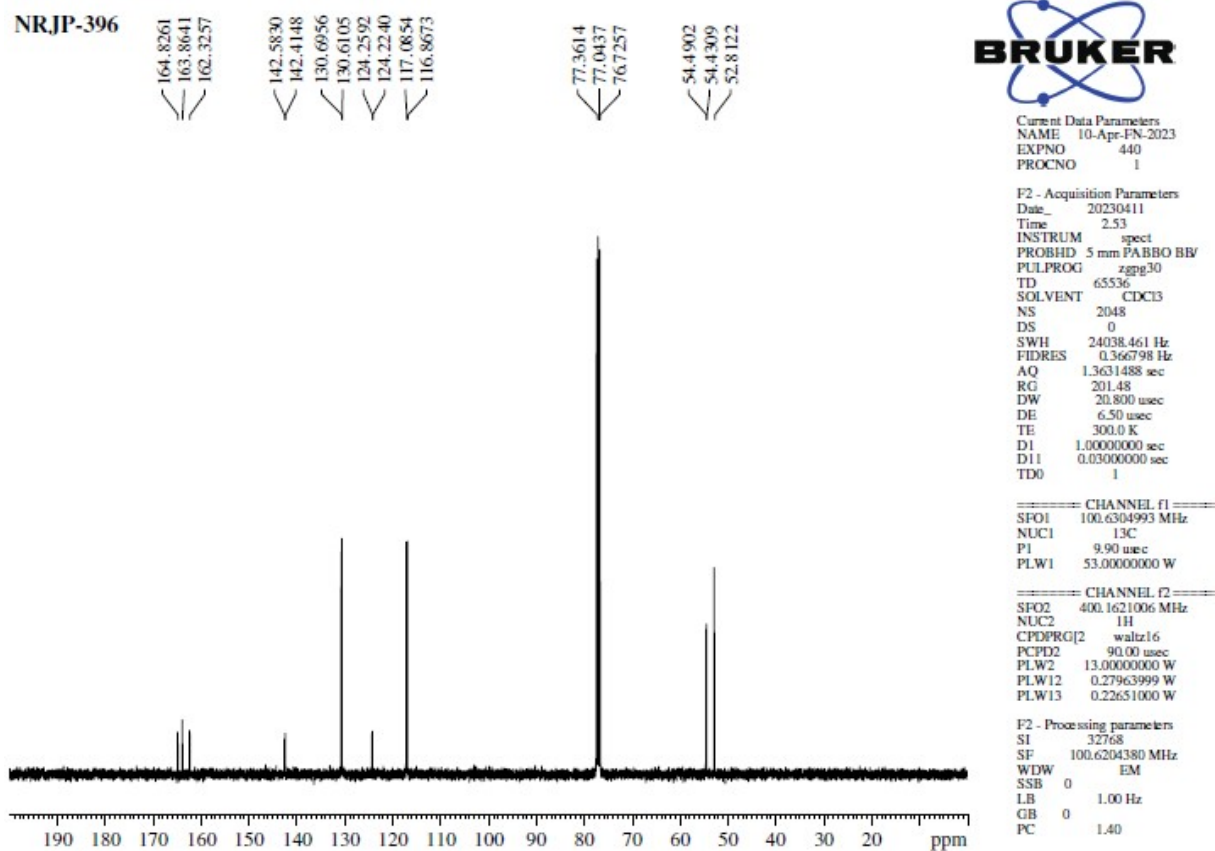
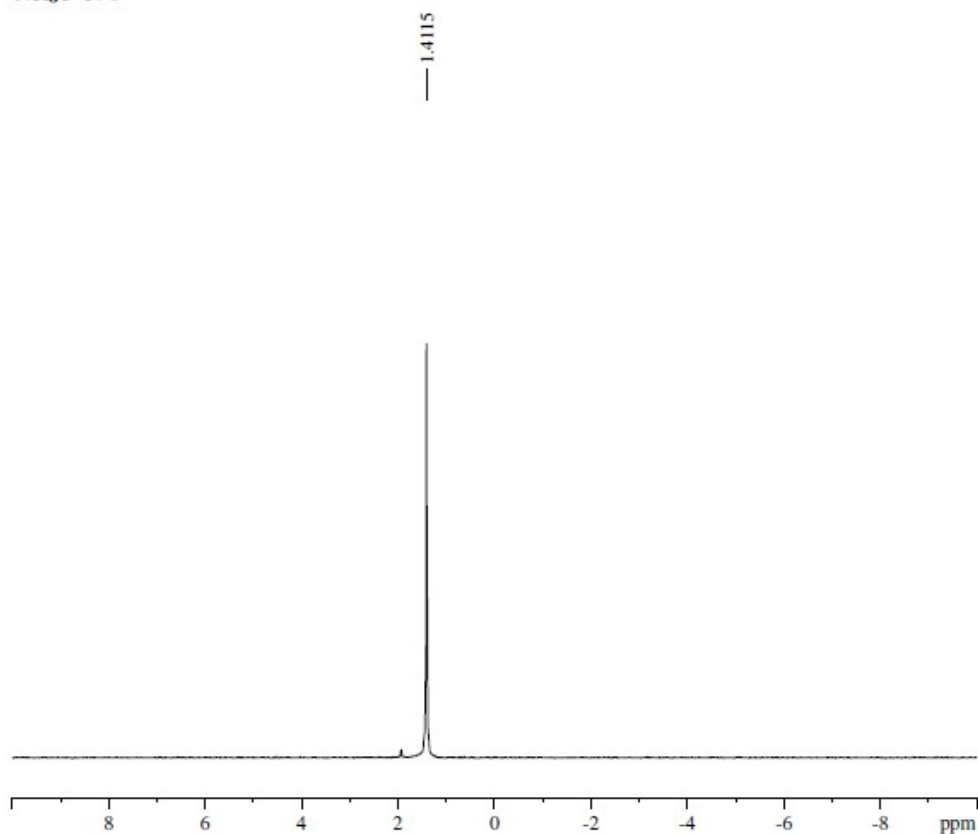


Figure S26: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of 3h

NRJP-396



Current Data Parameters
NAME 10-Apr-FN-2023
EXPNO 430
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230411
Time 2.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 64102.563 Hz
FIDRES 0.978127 Hz
AQ 0.5111808 sec
RG 201.48
DW 7.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.0000000 sec
D11 0.03000000 sec
TD0 1

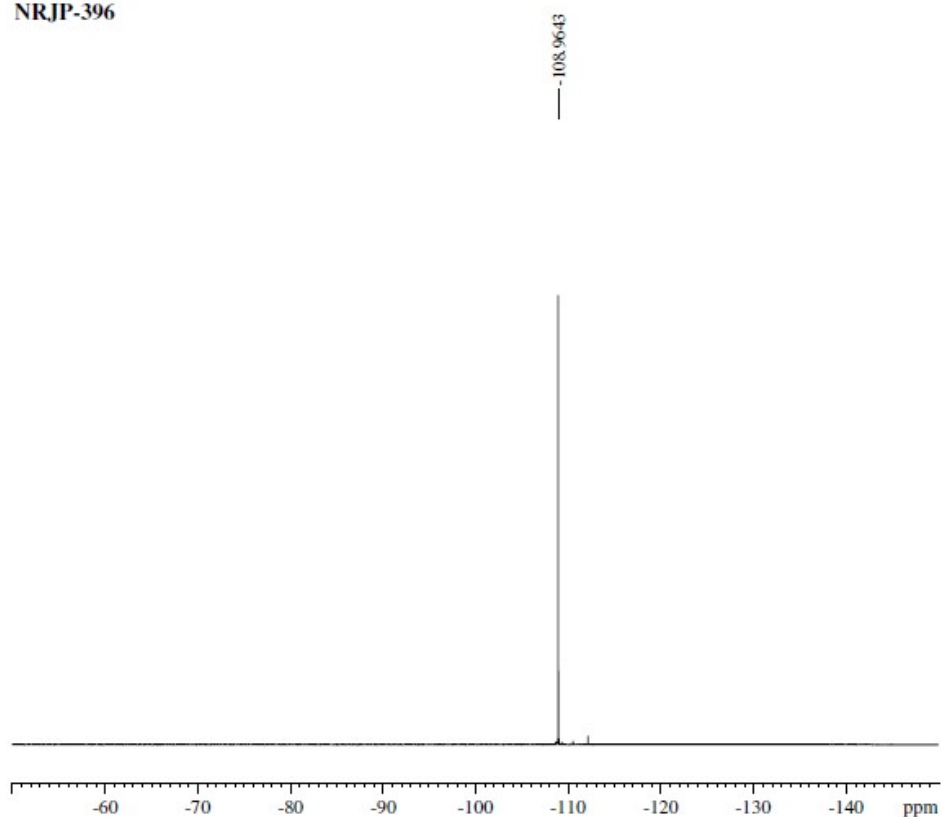
==== CHANNEL f1 ====
SFO1 161.9798402 MHz
NUC1 ^{31}P
P1 15.00 usec
PLW1 12.0000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 ^1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 13.0000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 161.9879400 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S27: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3h

NRJP-396



Current Data Parameters
NAME 02-May-AN-2023
EXPNO 500
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230503
Time 0.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg/hzgcn.2
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 89285.711 Hz
FIDRES 0.681196 Hz
AQ 0.7340032 sec
RG 201.48
DW 5.600 usec
DE 6.50 usec
TE 300.1 K
D1 1.0000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
TD0 1

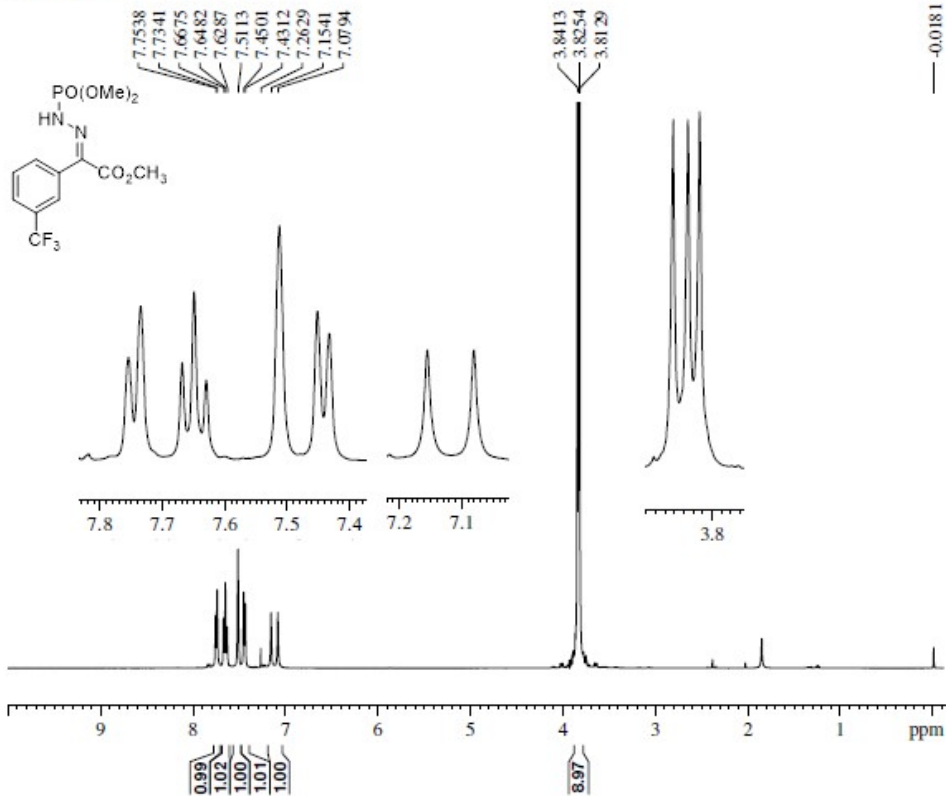
==== CHANNEL f1 ====
SFO1 376.4894122 MHz
NUC1 ^{19}F
P1 15.00 usec
PLW1 21.0000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 ^1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 13.0000000 W
PLW12 0.27963999 W

F2 - Processing parameters
SI 65536
SF 376.5270650 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S28: $^{19}\text{F}\{\text{H}\}$ NMR spectrum of 3h

NRJP383



Current Data Parameters
 NAME 24-MAR-AN-2023
 EXPNO 330
 PROCNO 1

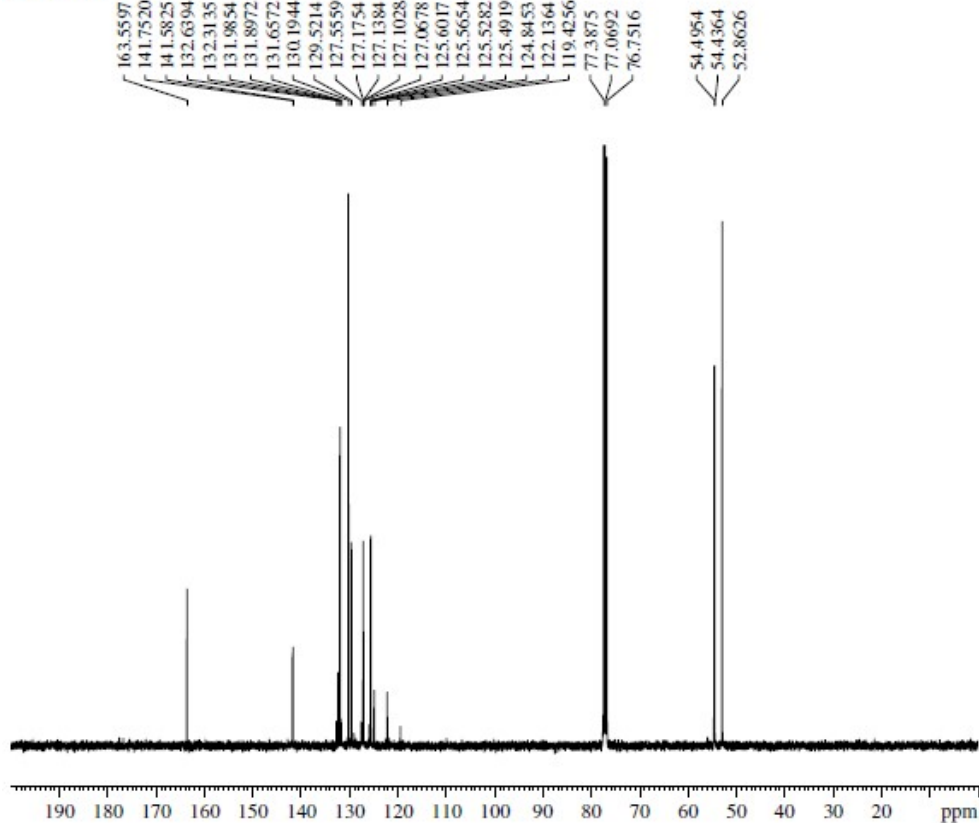
F2 - Acquisition Parameters
 Date_ 20230325
 Time 5.02
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 80.54
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605085 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S29: ¹H NMR spectrum of 3i

NRJP-383



Current Data Parameters
 NAME 29-Mar-AN-2023
 EXPNO 420
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230330
 Time 6.01
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S30: ¹³C{H} NMR spectrum of 3i

NRJP383

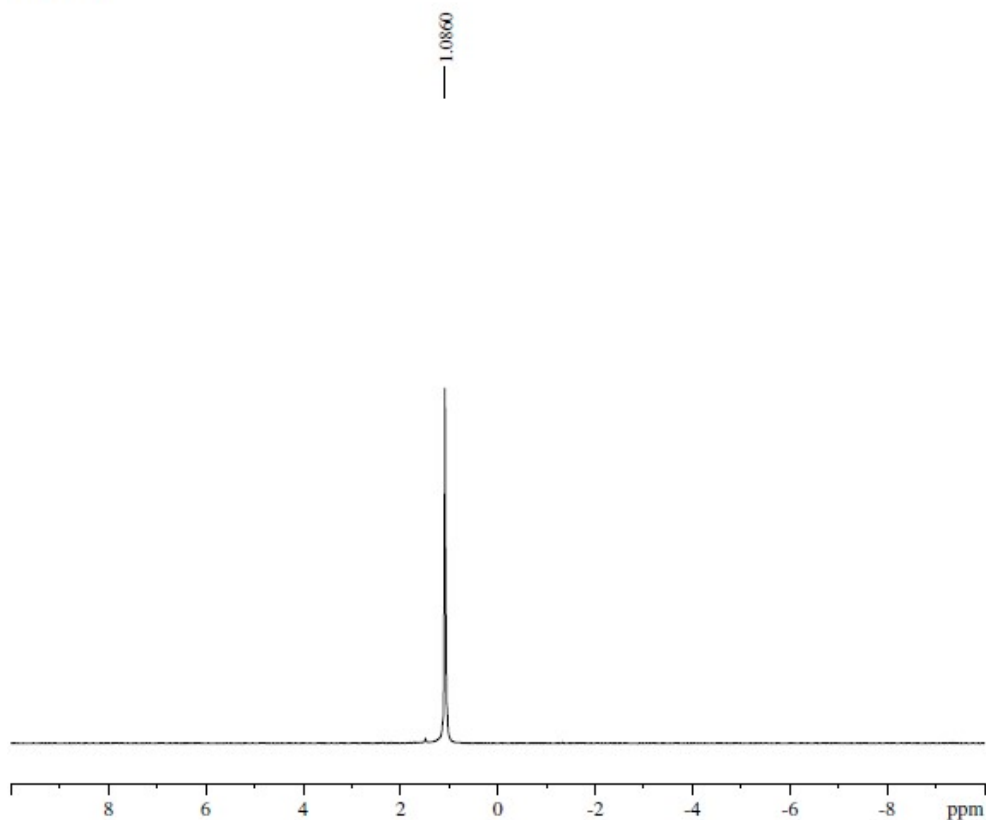


Figure S31: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3i



Current Data Parameters
NAME 24-MAR-AN-2023
EXPNO 340
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230325
Time 5.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 64102.563 Hz
FIDRES 0.978127 Hz
AQ 0.5111808 sec
RG 201.48
DW 7.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 161.9798402 MHz
NUC1 ^{31}P
P1 15.00 usec
PLW1 12.0000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 ^1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 13.0000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 161.9879400 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

NRJP-383

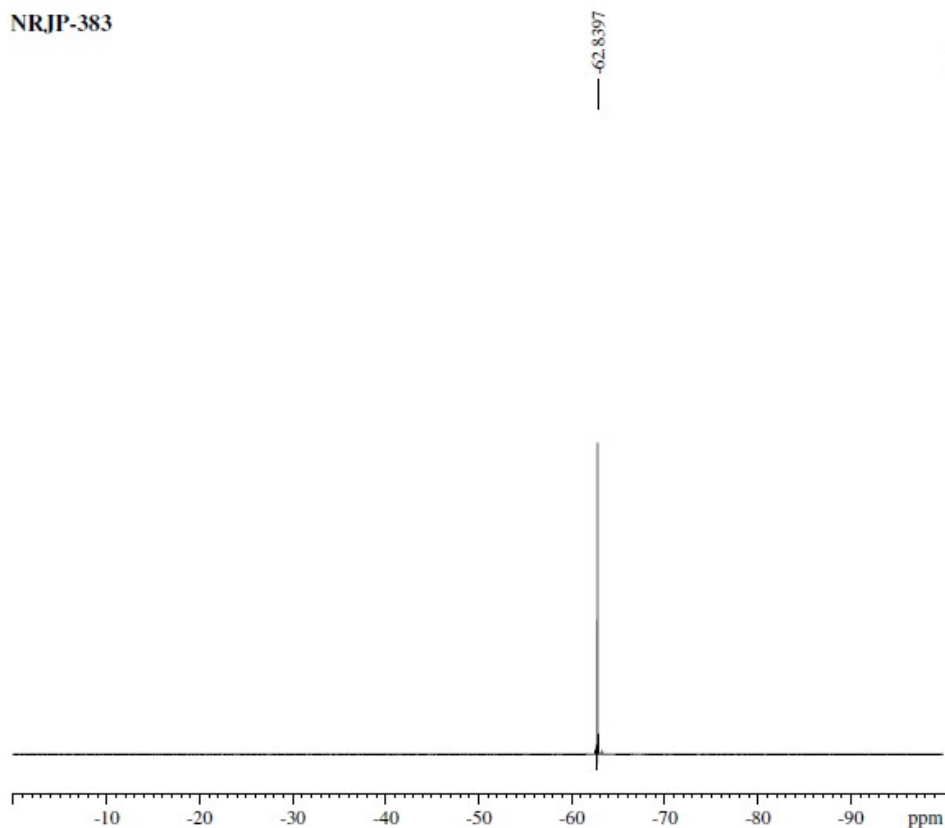


Figure S32: $^{19}\text{F}\{\text{H}\}$ NMR spectrum of 3i



Current Data Parameters
NAME 29-Mar-AN-2023
EXPNO 410
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230330
Time 5.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgfbigqn.2
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 89285.711 Hz
FIDRES 0.681196 Hz
AQ 0.7340032 sec
RG 201.48
DW 5.600 usec
DE 6.50 usec
TE 300.0 K
D1 1.0000000 sec
D11 0.0300000 sec
D12 0.0002000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 376.4894122 MHz
NUC1 ^{19}F
P1 15.00 usec
PLW1 21.0000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 ^1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 13.0000000 W
PLW12 0.27963999 W

F2 - Processing parameters
SI 65536
SF 376.5270650 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

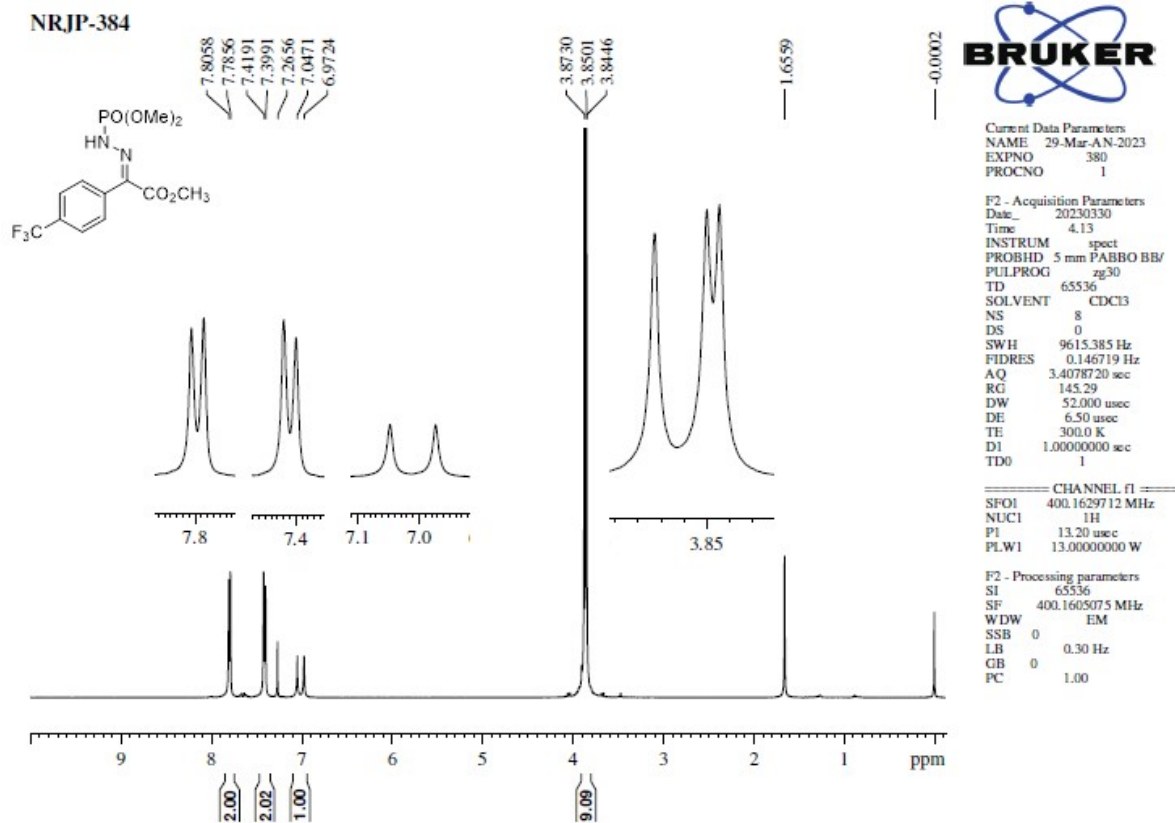


Figure S33: ^1H NMR spectrum of 3j

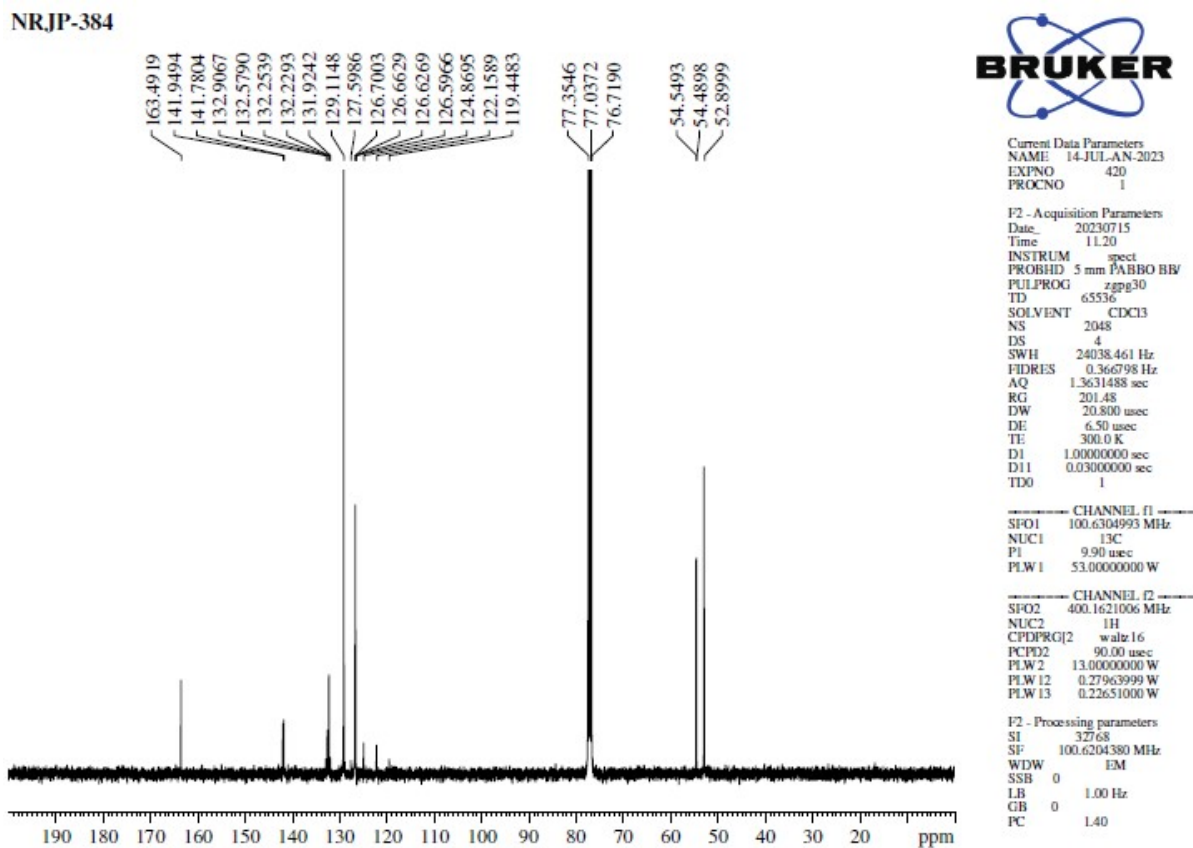
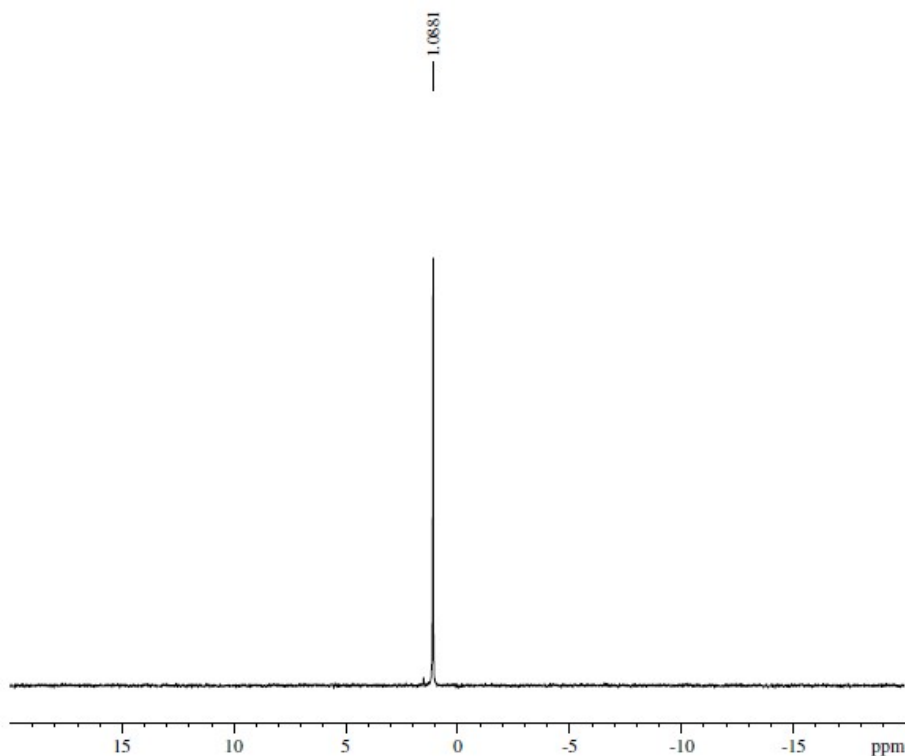


Figure S34: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of 3j

NRJP384

Figure S35: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3j

Current Data Parameters
 NAME 31-Mar-AN-2023
 EXPNO 350
 PROCNO 1

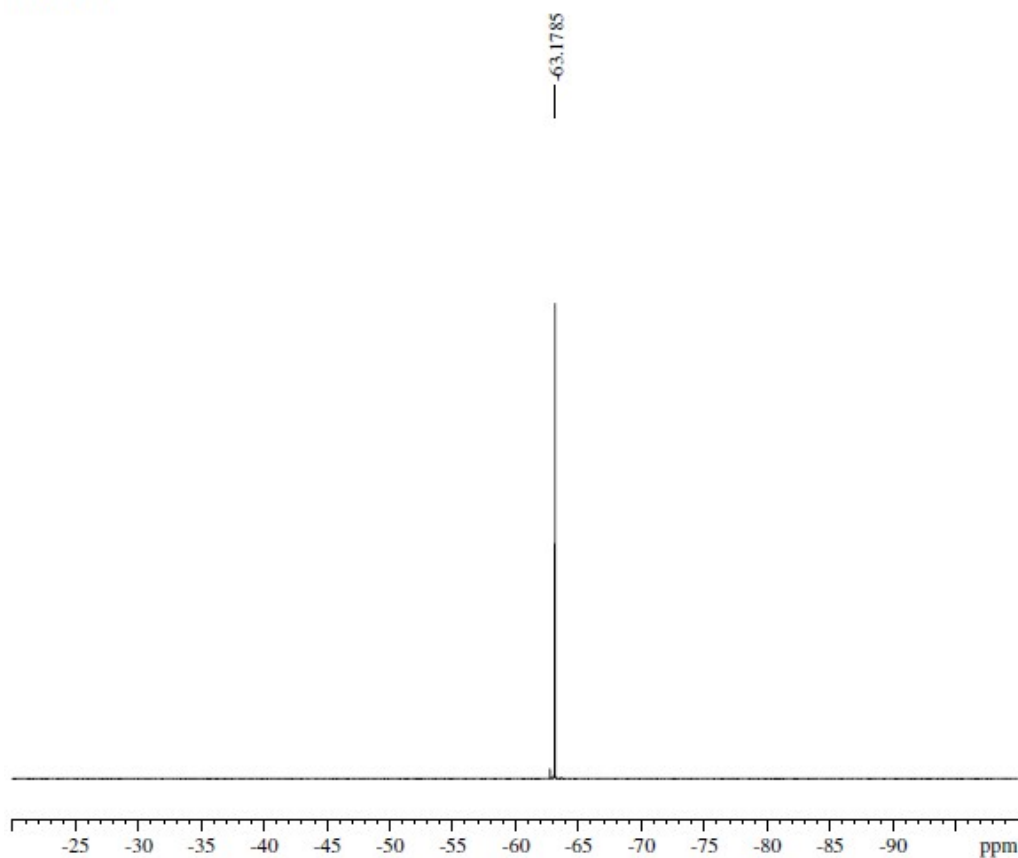
F2 - Acquisition Parameters
 Date_ 20230401
 Time 6.20
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 ^{31}P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP384

Figure S36: $^{19}\text{F}\{\text{H}\}$ NMR spectrum of 3j

Current Data Parameters
 NAME 31-Mar-AN-2023
 EXPNO 340
 PROCNO 1

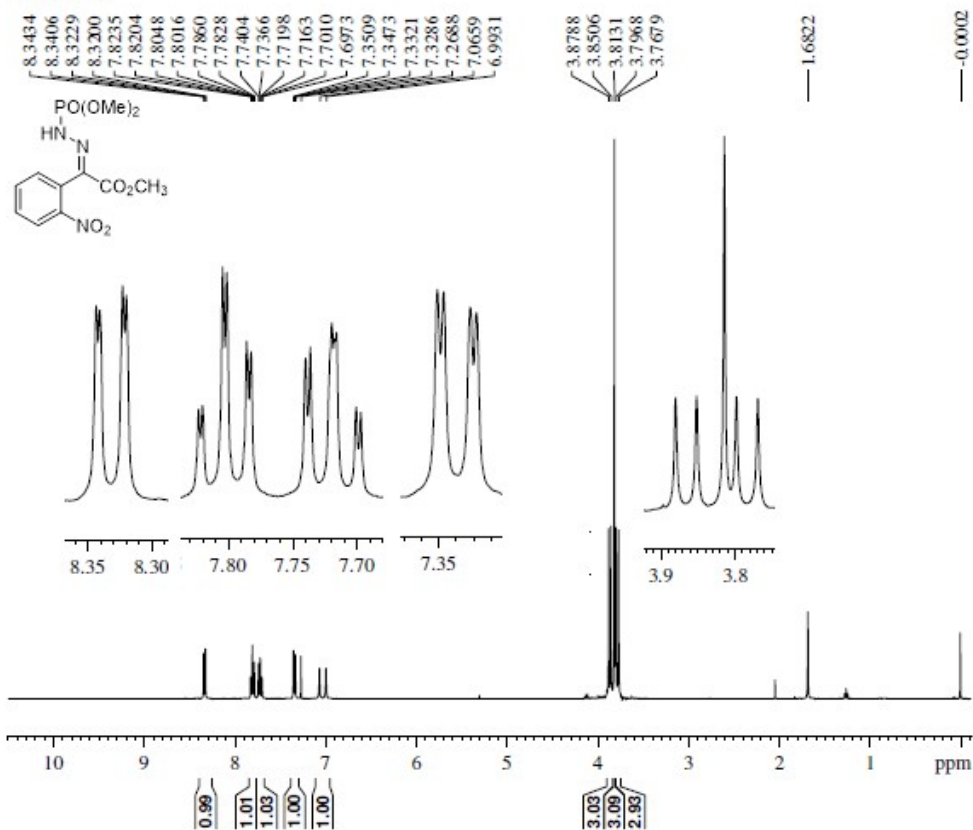
F2 - Acquisition Parameters
 Date_ 20230401
 Time 6.19
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgfbigqn.2
 TD 131072
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 89285.711 Hz
 FIDRES 0.681196 Hz
 AQ 0.7340032 sec
 RG 201.48
 DW 5.600 usec
 DE 6.50 usec
 TE 300.1 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 376.4894122 MHz
 NUC1 ^{19}F
 P1 15.00 usec
 PLW1 21.00000000 W

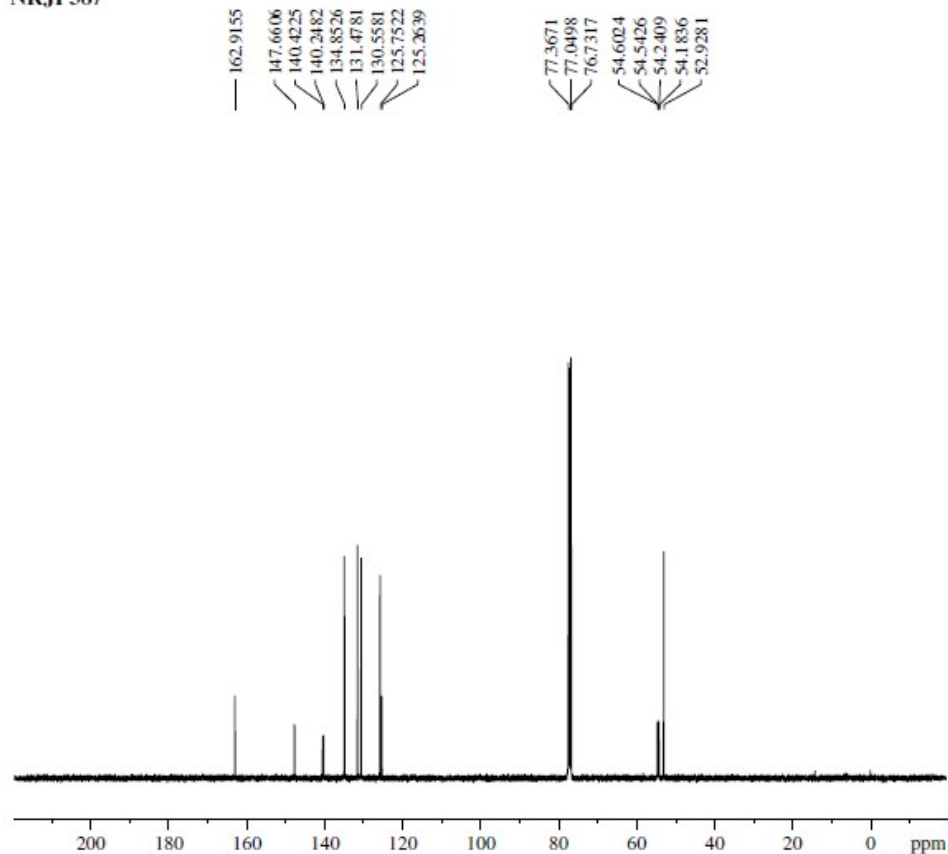
==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W

F2 - Processing parameters
 SI 65536
 SF 376.5270650 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NRJP387

Figure S37: ^1H NMR spectrum of 3k

NRJP387

Figure S38: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of 3k

Current Data Parameters
 NAME 03-Apr-AN-2023
 EXPNO 330
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230404
 Time 2.24
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 129.57
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 ^1H
 P1 13.20 usec
 PLW1 13.0000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605061 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME 31-Mar-AN-2023
 EXPNO 330
 PROCNO 1

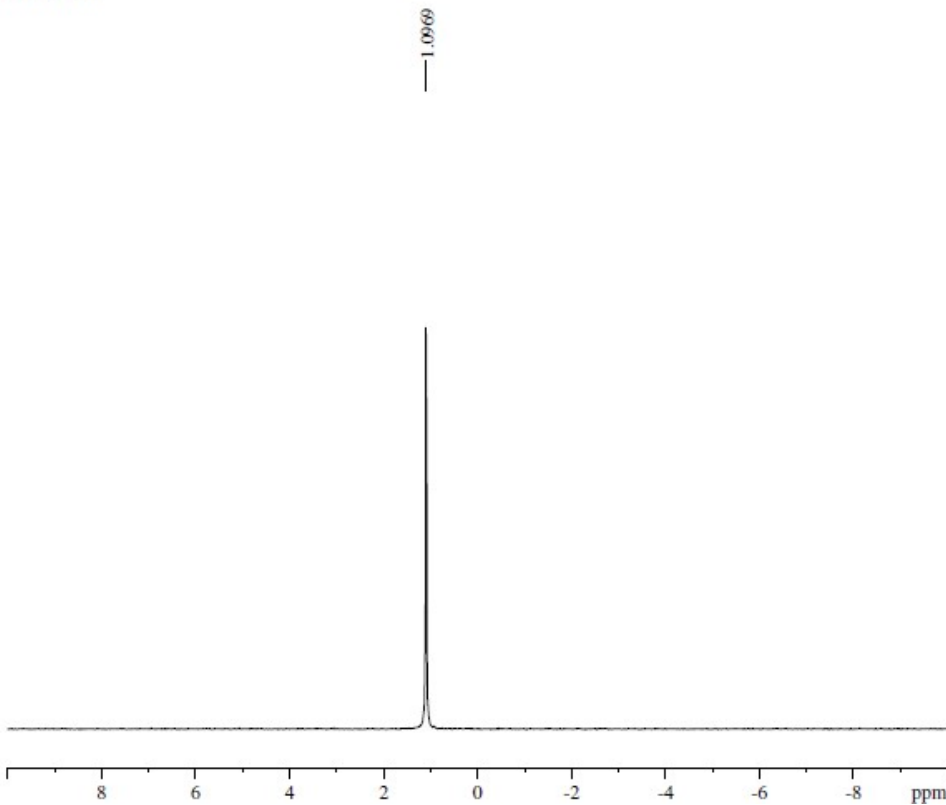
F2 - Acquisition Parameters
 Date_ 20230401
 Time 6.15
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 291.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 ^{13}C
 P1 9.90 usec
 PLW1 53.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP387



Current Data Parameters
 NAME 31-Mar-AN-2023
 EXPNO 320
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230401
 Time 5.21
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

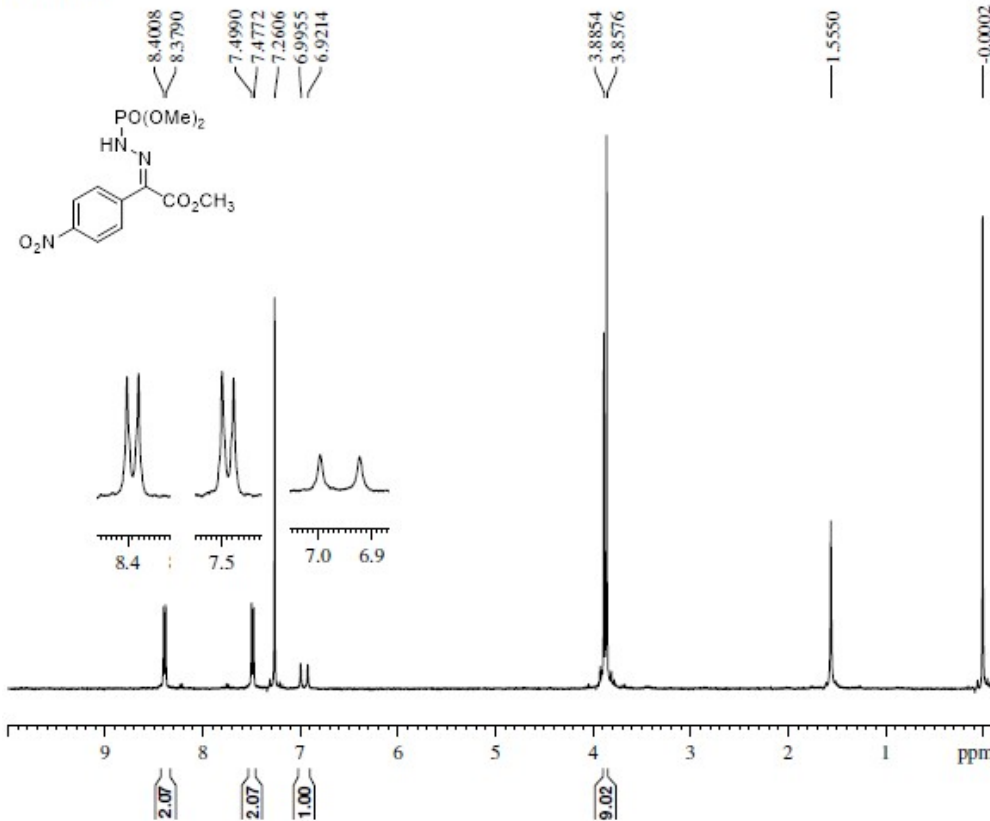
==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S39: ³¹P{¹H} NMR spectrum of 3k

NRJP-389



Current Data Parameters
 NAME 26-APR-AN-2023
 EXPNO 530
 PROCNO 1

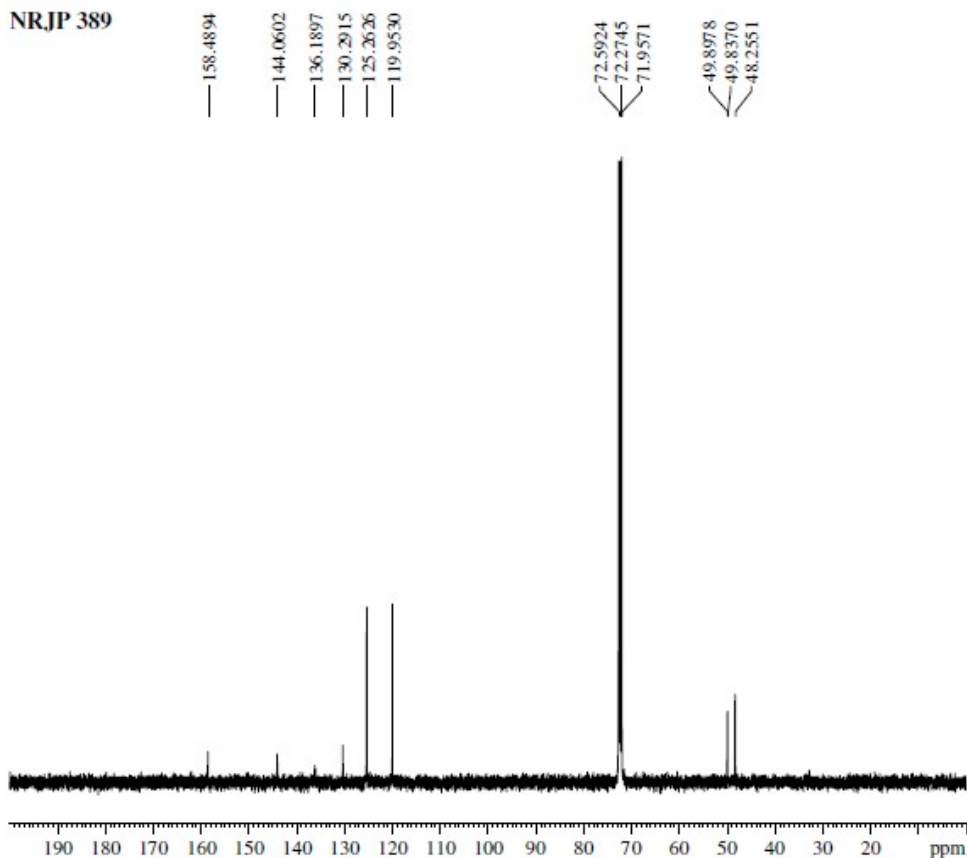
F2 - Acquisition Parameters
 Date_ 20230426
 Time 12.30
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 201.48
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605092 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S40: ¹H NMR spectrum of 3l

NRJP 389



Current Data Parameters
NAME 03-May-AN-2023
EXPNO 330
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230504
Time 8.08
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 201.48
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1

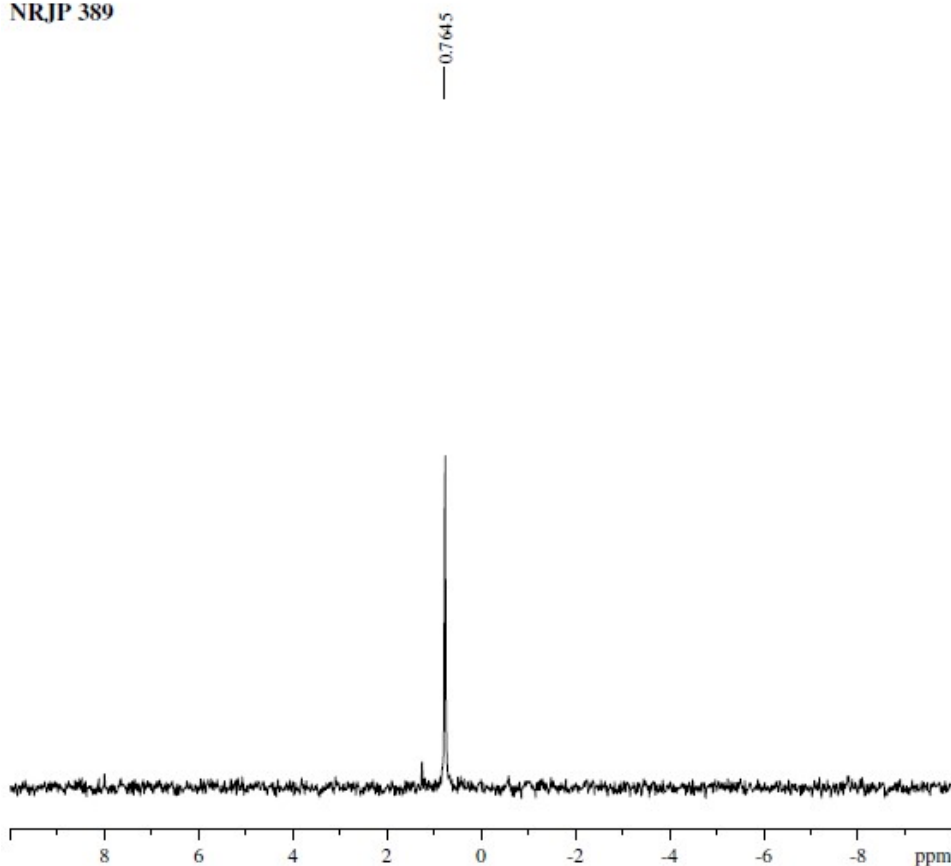
==== CHANNEL f1 ====
SFO1 100.6304993 MHz
NUC1 ^{13}C
P1 9.90 usec
PLW1 53.00000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 ^1H
CPDPRG[2] walz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 100.6204380 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S41: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3l

NRJP 389



Current Data Parameters
NAME 27-APR-AN-2023
EXPNO 320
PROCNO 1

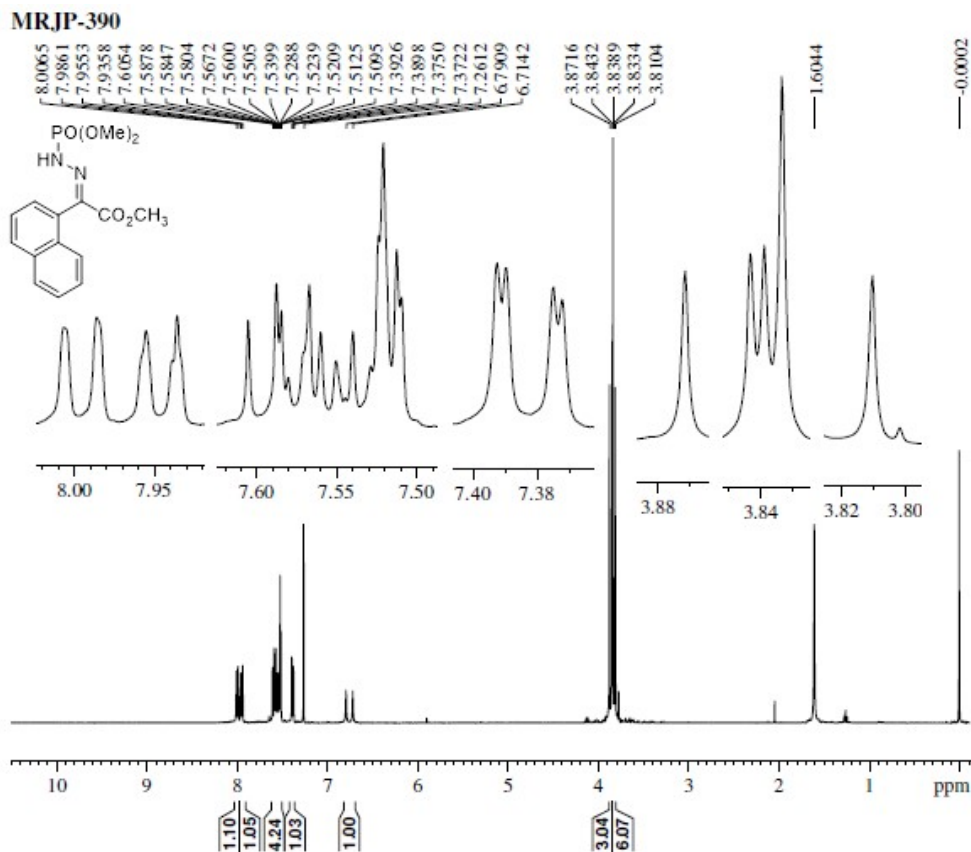
F2 - Acquisition Parameters
Date_ 20230427
Time 23.28
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 64102.563 Hz
FIDRES 0.978127 Hz
AQ 0.5111808 sec
RG 201.48
DW 7.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 161.9798402 MHz
NUC1 ^{31}P
P1 15.00 usec
PLW1 12.00000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 ^1H
CPDPRG[2] walz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 161.9879400 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S42: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3l



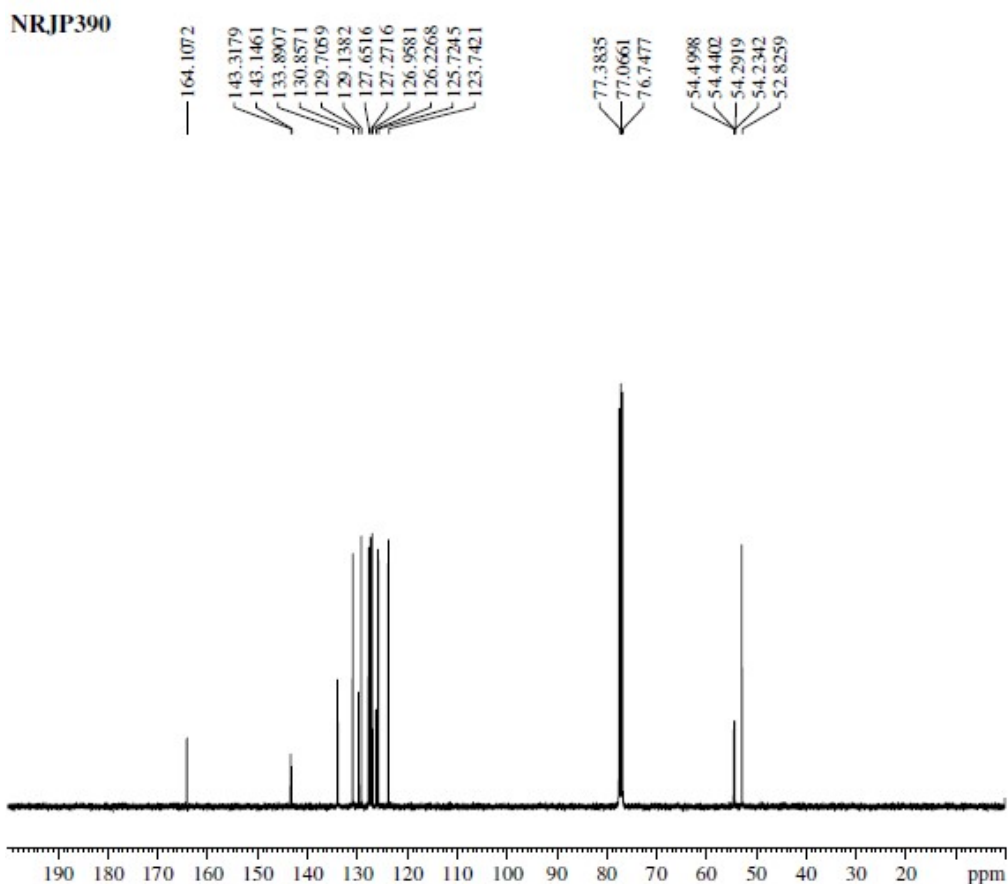
Current Data Parameters
 NAME 01-Aug-AN-2023
 EXPNO 470
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230801
 Time 20.34
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 159.22
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605091 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S43: ¹H NMR spectrum of 3m



Current Data Parameters
 NAME 03-Apr-AN-2023
 EXPNO 350
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230404
 Time 3.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TDO 1

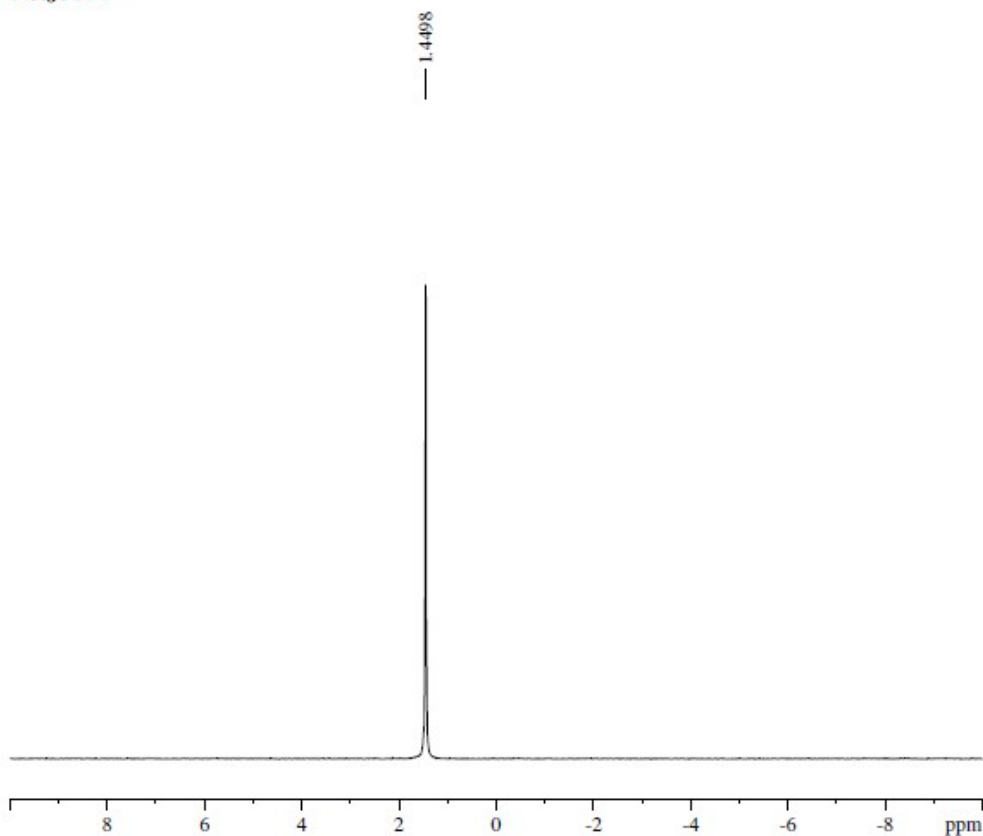
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S44: ¹³C{¹H} NMR spectrum of 3m

NRJP390



Current Data Parameters
 NAME 03-Apr-AN-2023
 EXPNO 360
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230404
 Time 3.16
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

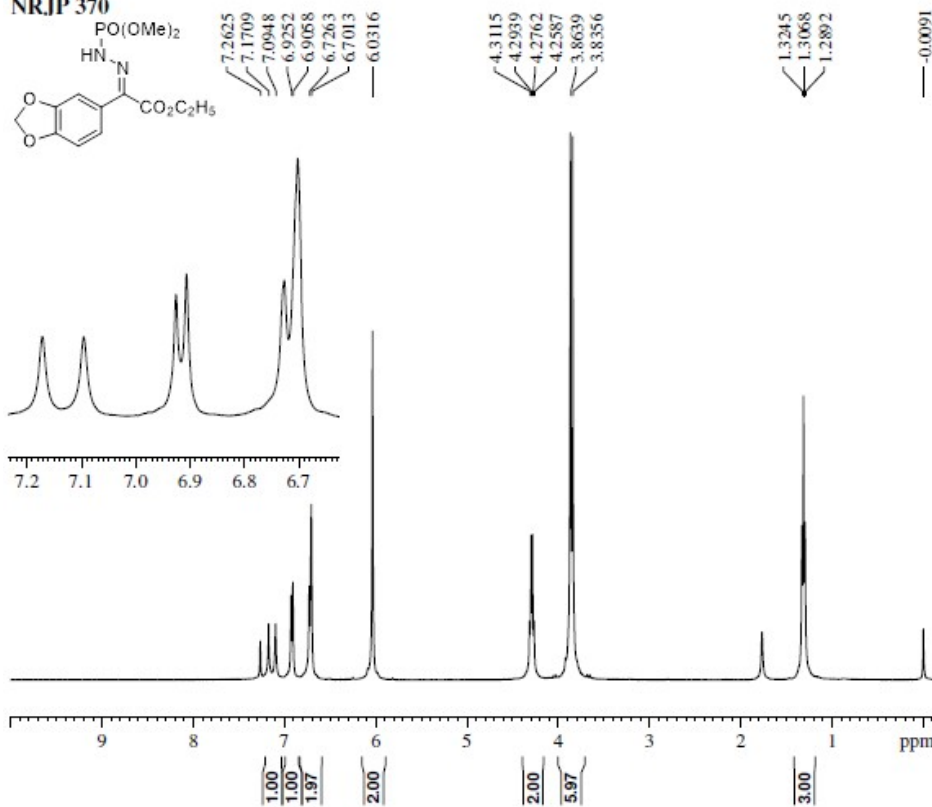
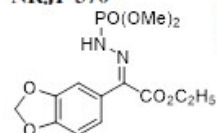
==== CHANNEL f1 =====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S45: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3m

NRJP 370



Current Data Parameters
 NAME 31-MAY-AN-2023
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230531
 Time 18.46
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 114.26
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 =====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605089 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S46: ^1H NMR spectrum of 3n

NRJP 370

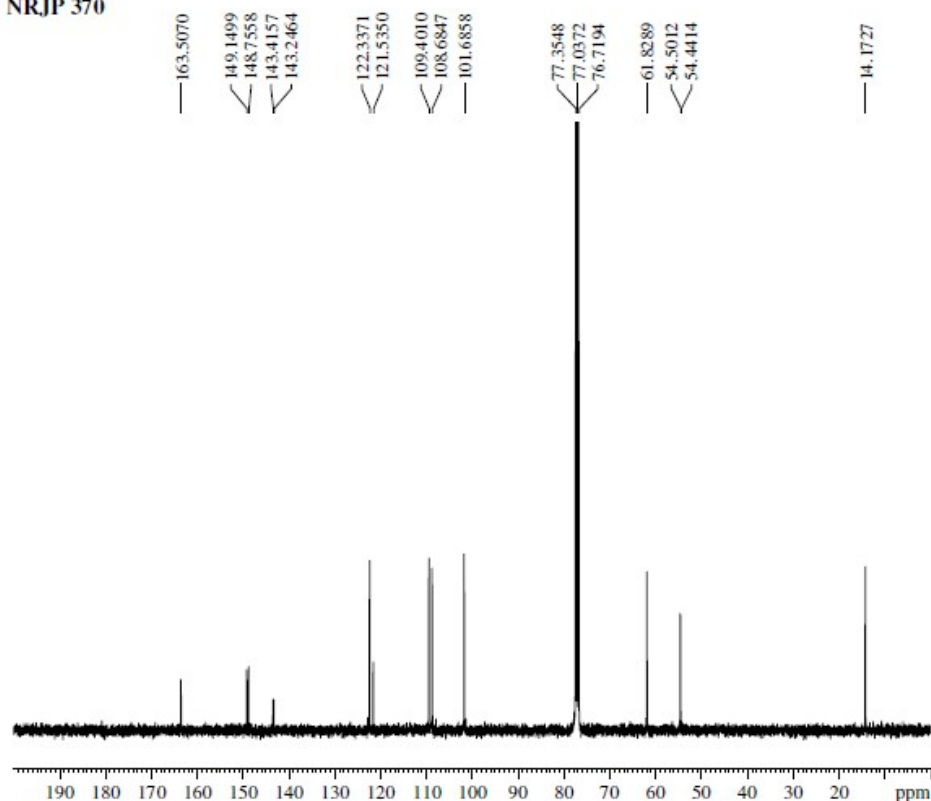


Figure S47: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3n



Current Data Parameters
 NAME 06-JUN-AN-2023
 EXPNO 330
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230607
 Time 0.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 ^{13}C
 P1 9.90 usec
 PLW1 53.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP 370

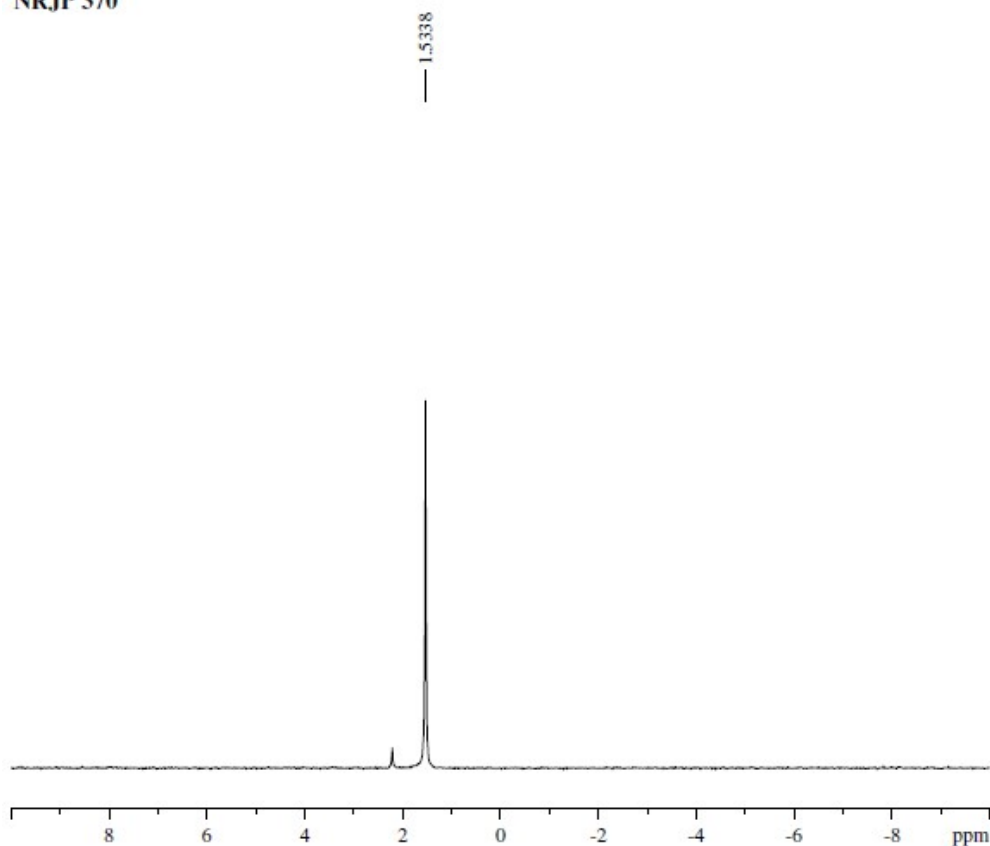


Figure S48: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3n



Current Data Parameters
 NAME 07-JUN-AN-2023
 EXPNO 340
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230607
 Time 22.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 ^{31}P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

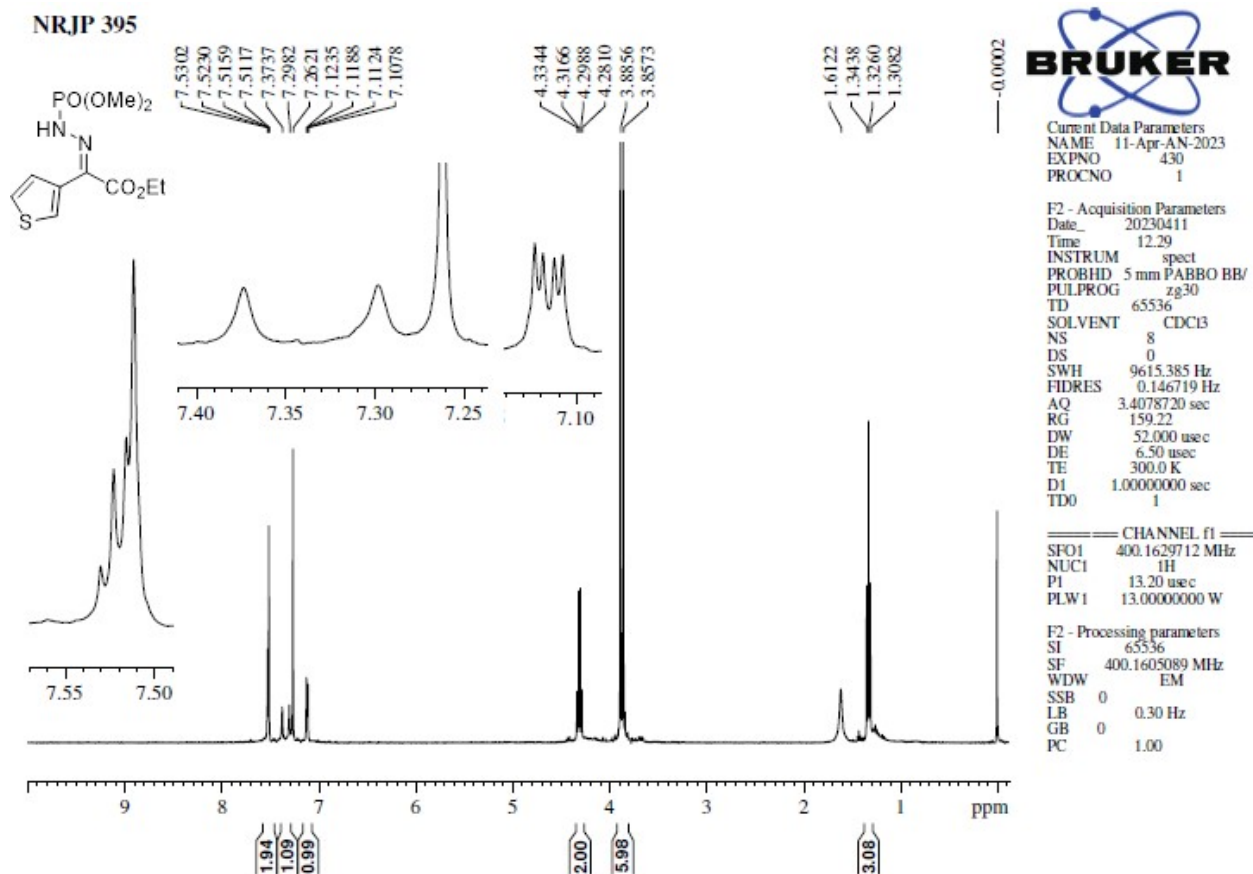


Figure S49: ¹H NMR spectrum of 3o

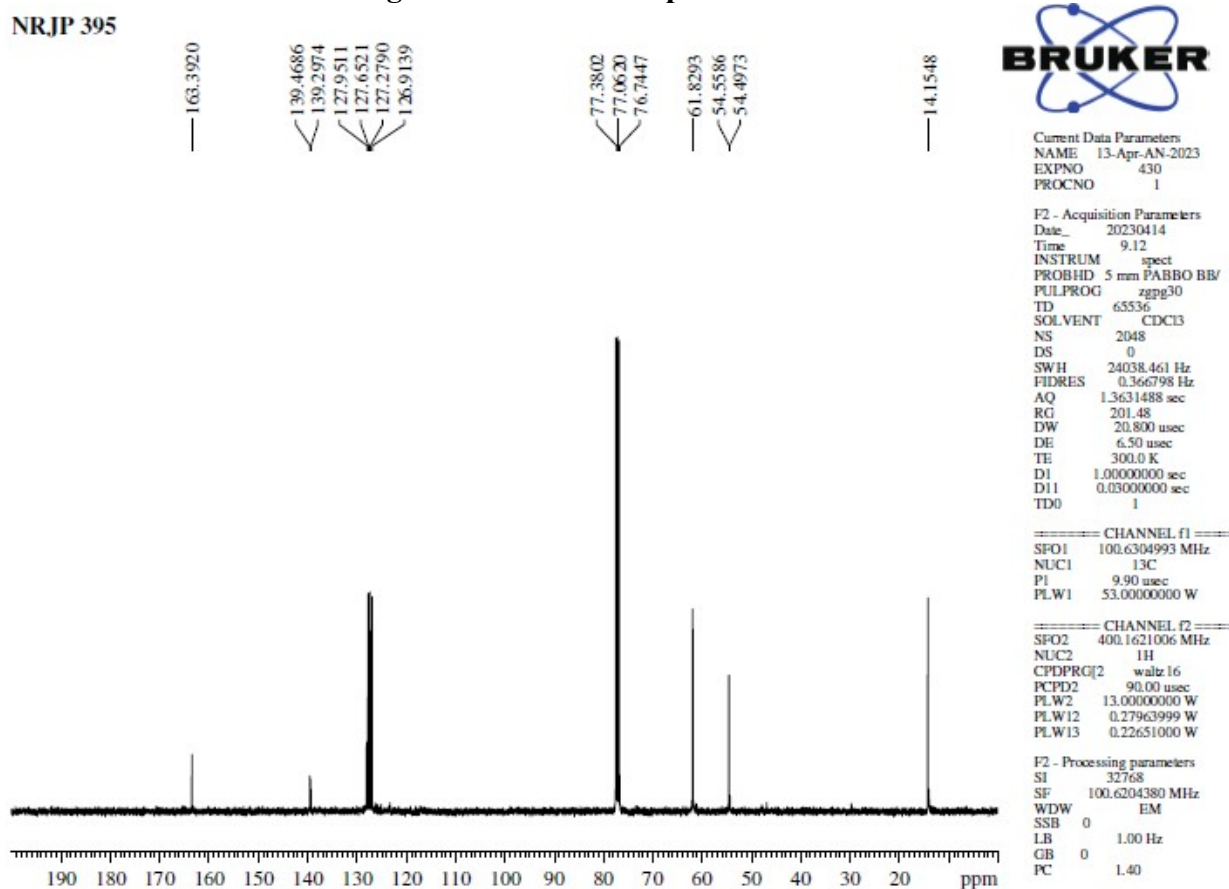
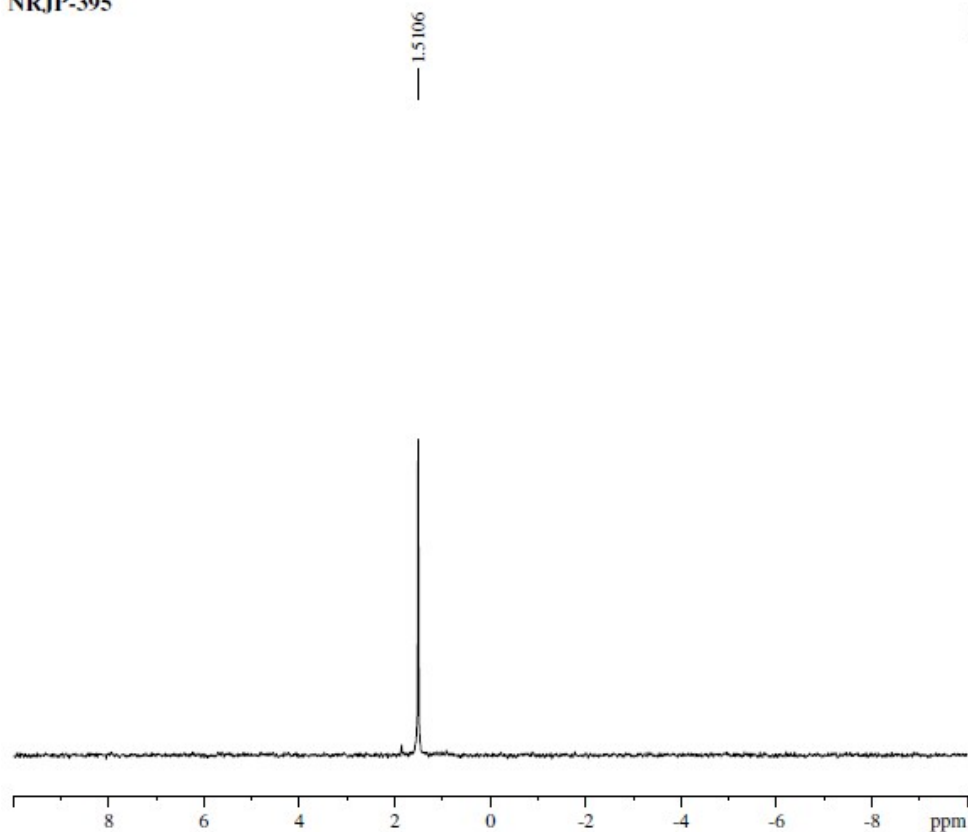


Figure S50: ¹³C{¹H} NMR spectrum of 3o

NRJP-395

Figure S51: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3o

Current Data Parameters
 NAME 12-Apr-2023
 EXPNO 330
 PROCNO 1

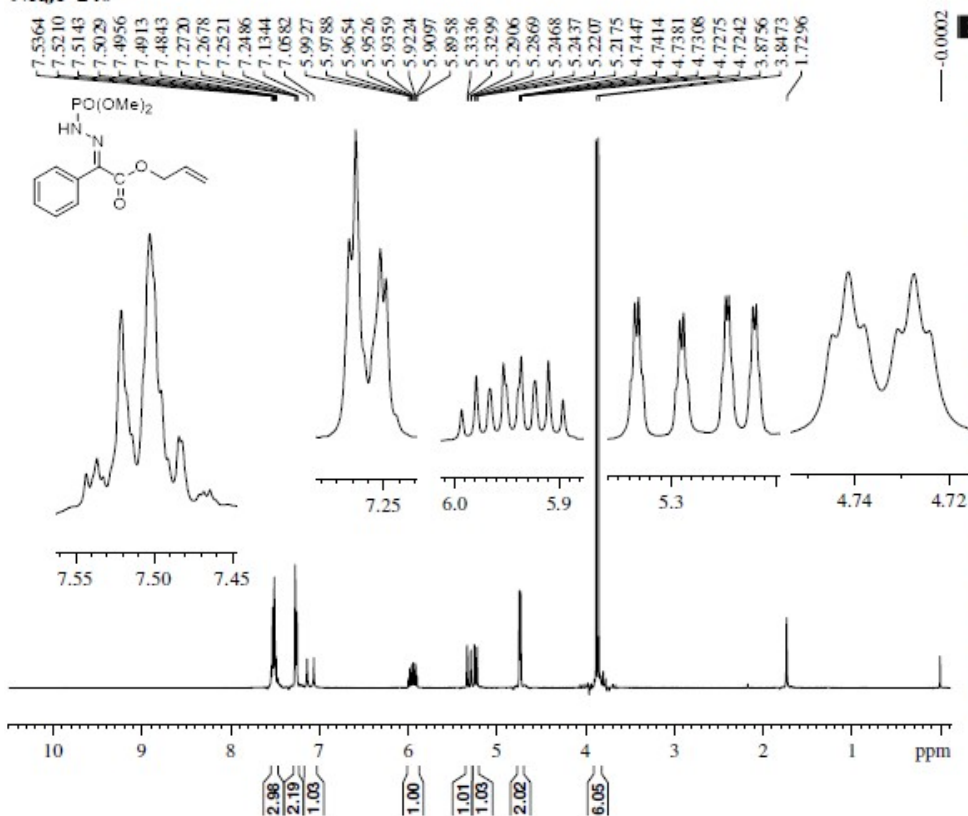
F2 - Acquisition Parameters
 Date_ 20230412
 Time 19.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 ^{31}P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CDPORG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP 246

Figure S52: ^1H NMR spectrum of 3p

Current Data Parameters
 NAME 24-Apr-AN-2023
 EXPNO 420
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230424
 Time 22.28
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 100.41
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 ^1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605065 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NRJP 246

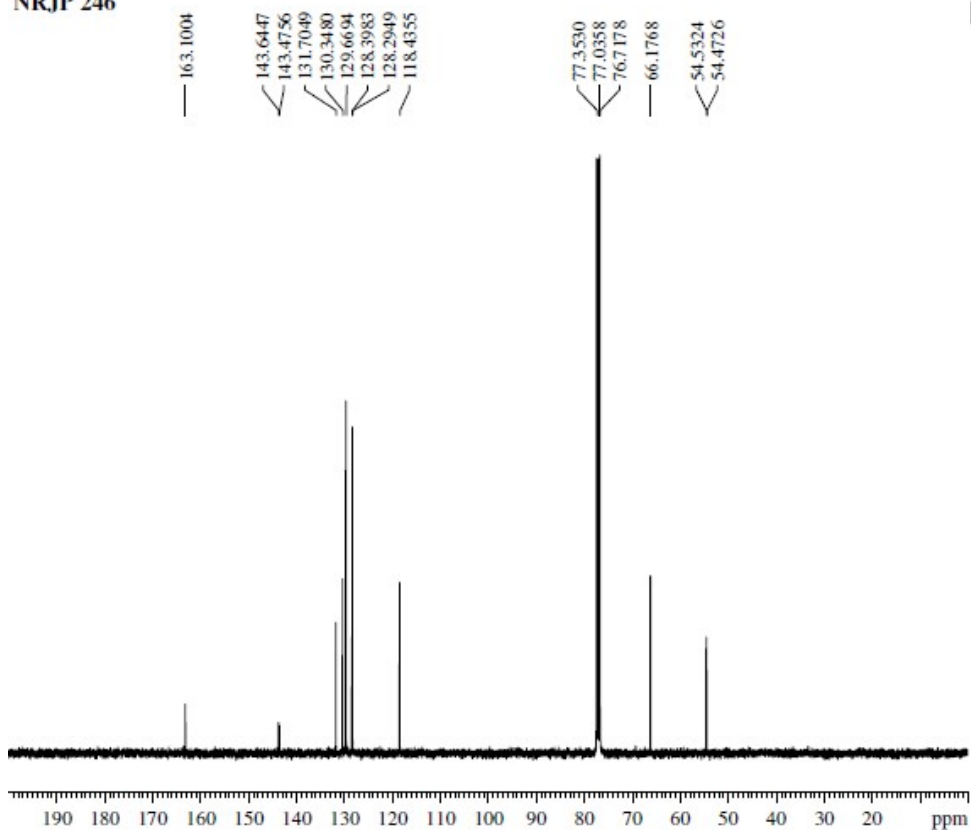


Figure S53: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3p



Current Data Parameters
NAME 24-Apr-AN-2023
EXPNO 430
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230424
Time 23.05
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 201.48
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 100.6304993 MHz
NUC1 ^{13}C
P1 9.90 usec
PLW1 53.0000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 ^1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 13.0000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 100.6204380 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

NRPK-246

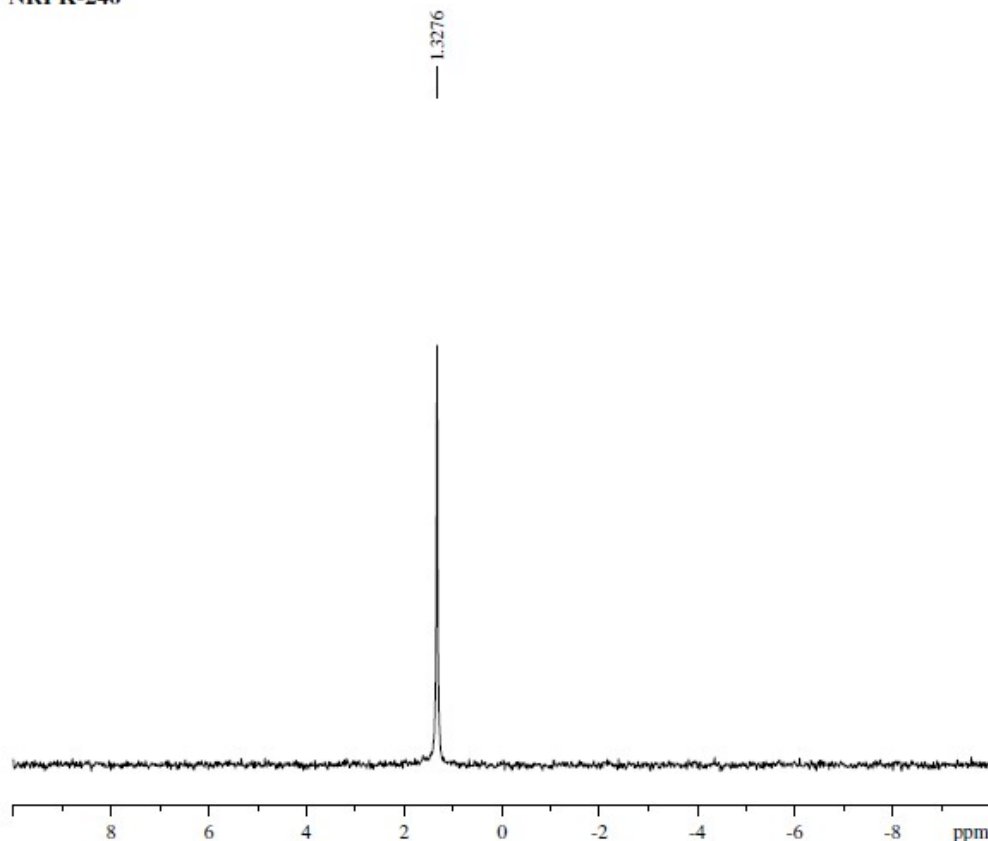


Figure S54: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3p



Current Data Parameters
NAME 20-Mar-AN-2023
EXPNO 470
PROCNO 1

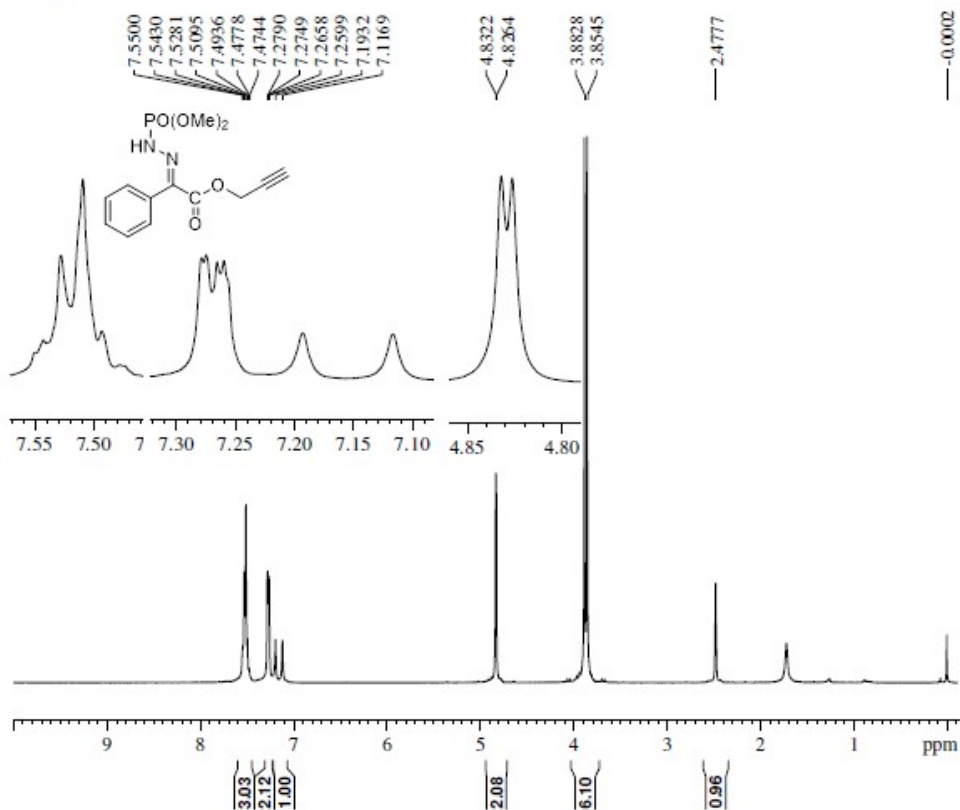
F2 - Acquisition Parameters
Date_ 20230321
Time 2.24
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 64102.563 Hz
FIDRES 0.978127 Hz
AQ 0.5111808 sec
RG 201.48
DW 7.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 ====
SFO1 161.9798402 MHz
NUC1 ^{31}P
P1 15.00 usec
PLW1 12.0000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 ^1H
CPDPRG2 waltz16
PCPD2 90.00 usec
PLW2 13.0000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 161.9879400 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

NRJP-379



BRUKER

Current Data Parameters
 NAME 16-Mar-AN-2023
 EXPNO 360
 PROCNO 1

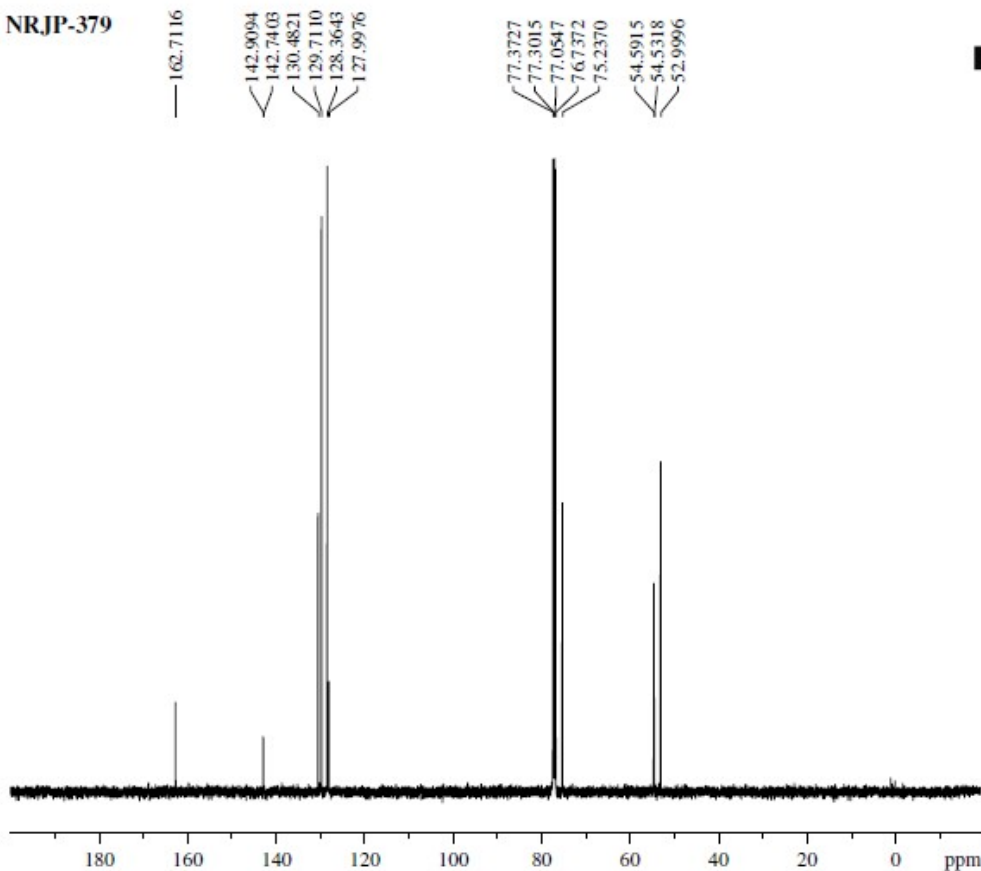
F2 - Acquisition Parameters
 Date_ 20230316
 Time 20.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 129.57
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 =====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605071 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S55: ¹H NMR spectrum of 3q

NRJP-379



BRUKER

Current Data Parameters
 NAME 13-Mar-AN-2023
 EXPNO 340
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230314
 Time 3.08
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TDO 1

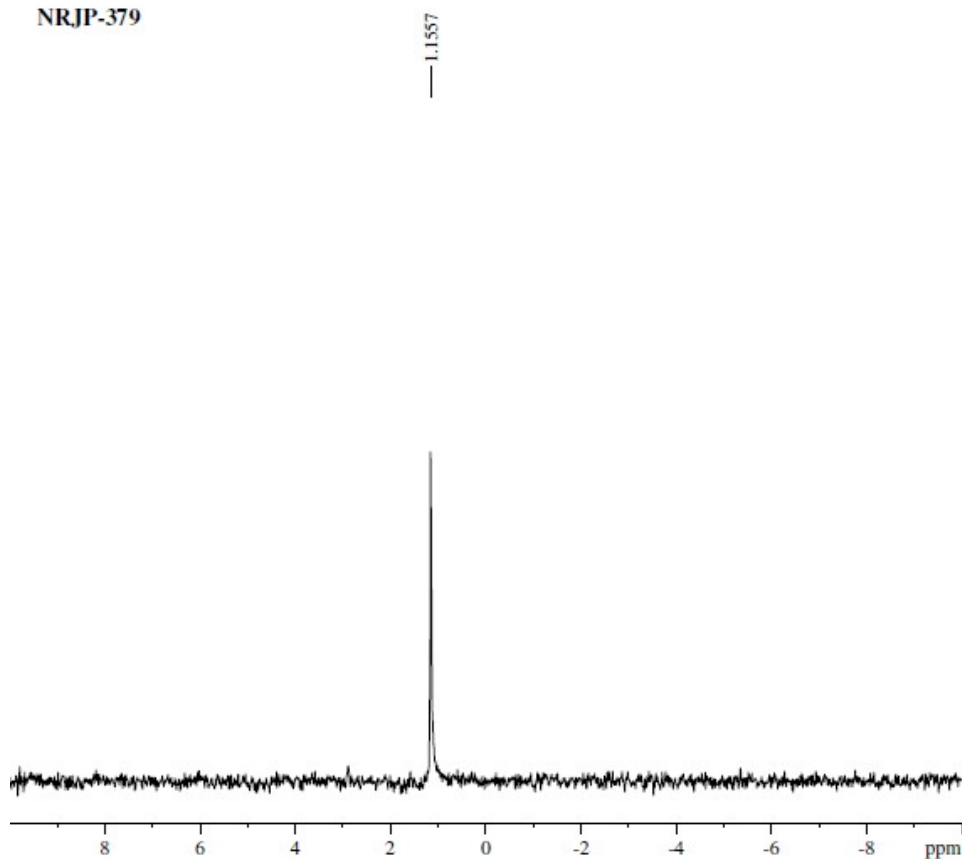
==== CHANNEL f1 =====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S56: ¹³C{¹H} NMR spectrum of 3q

NRJP-379

Figure S57: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3q

Current Data Parameters
 NAME 22-Mar-AN-2023
 EXPNO 310
 PROCNO 1

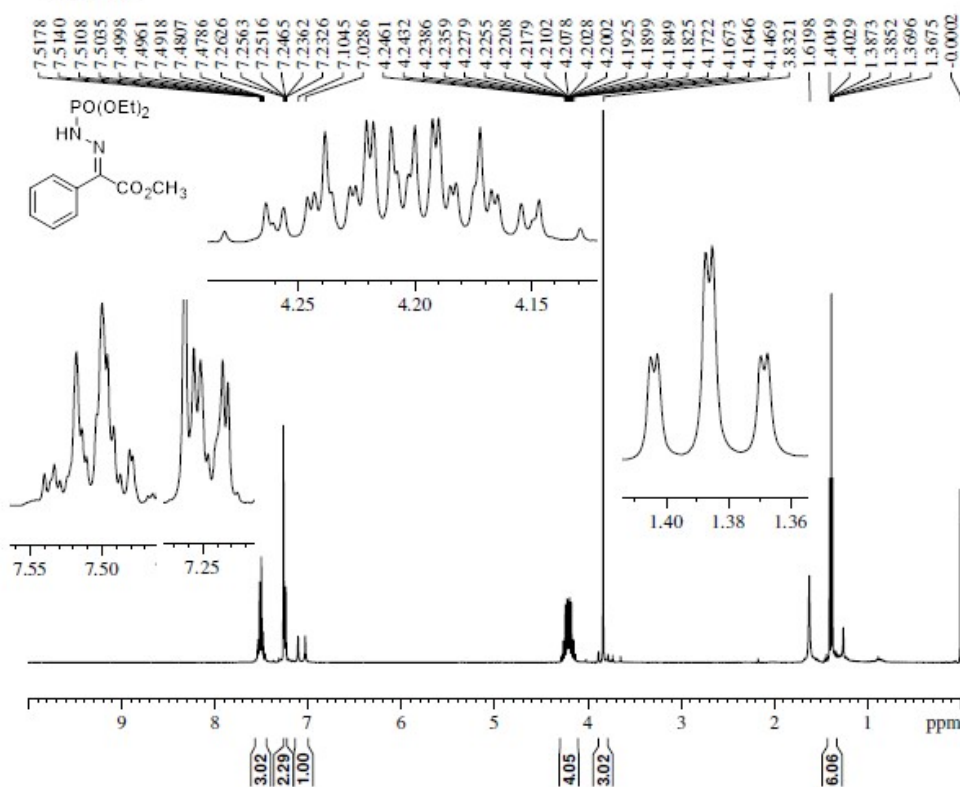
F2 - Acquisition Parameters
 Date_ 20230322
 Time 16.03
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 ^{31}P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP 371

Figure S58: ^1H NMR spectrum of 3r

Current Data Parameters
 NAME 03-May-AN-2023
 EXPNO 320
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230504
 Time 7.32
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 145.29
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 ^1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605087 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

NRJP371

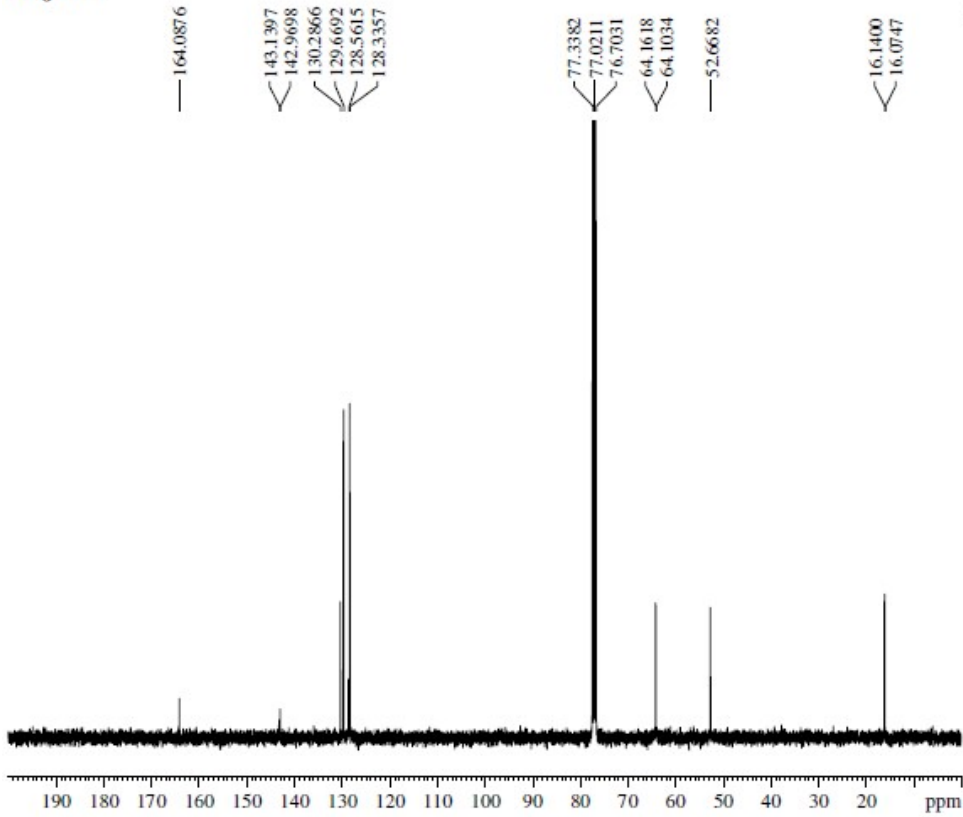


Figure S59: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3r



Current Data Parameters
 NAME 20-FEB-AN-2023
 EXPNO 430
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230221
 Time 4.34
 INSTRUM spect
 PROBHD 5 mm PABBO BH
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 ^{13}C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP 371

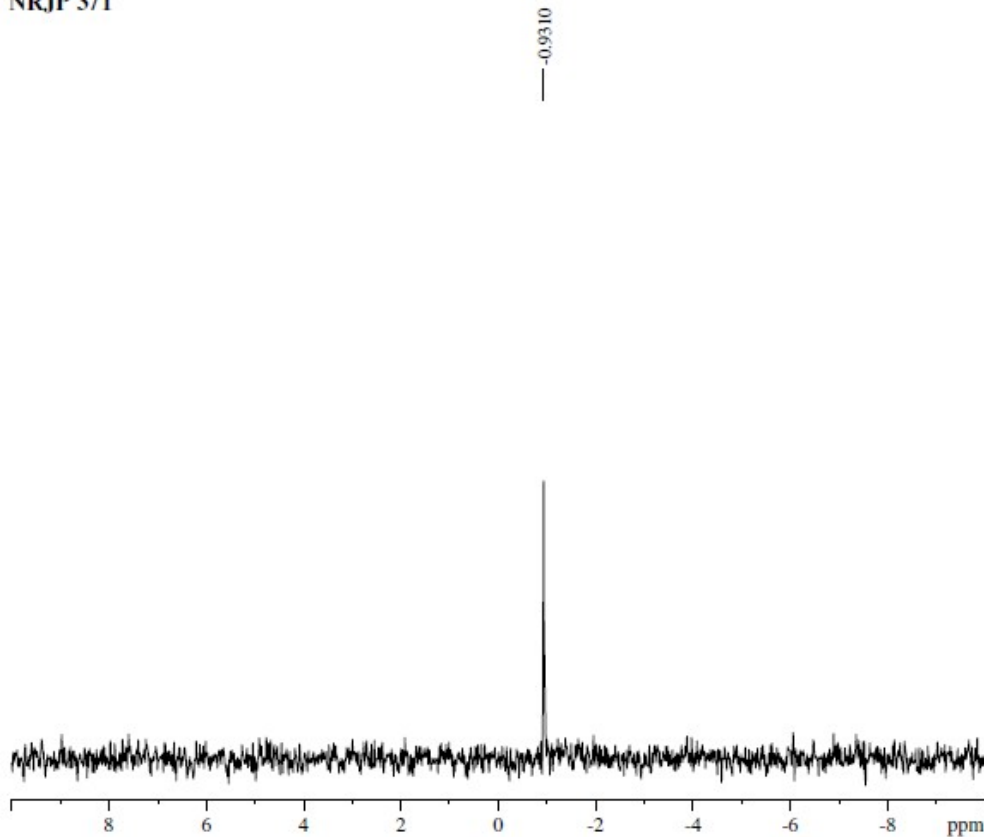


Figure S60: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3r



Current Data Parameters
 NAME 24-Apr-AN-2023
 EXPNO 410
 PROCNO 1

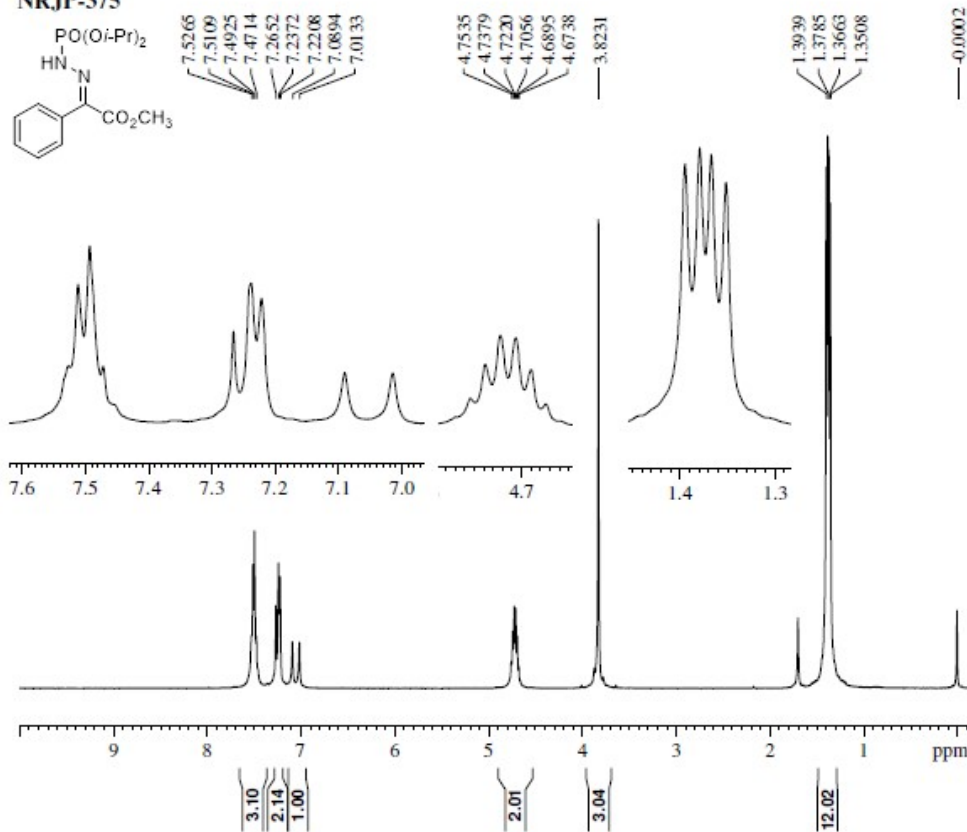
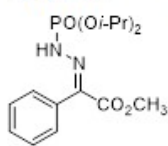
F2 - Acquisition Parameters
 Date_ 20230424
 Time 22.23
 INSTRUM spect
 PROBHD 5 mm PABBO BH
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 ^{31}P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP-375



Current Data Parameters
 NAME 27-Apr-FN-2023
 EXPNO 500
 PROCNO 1

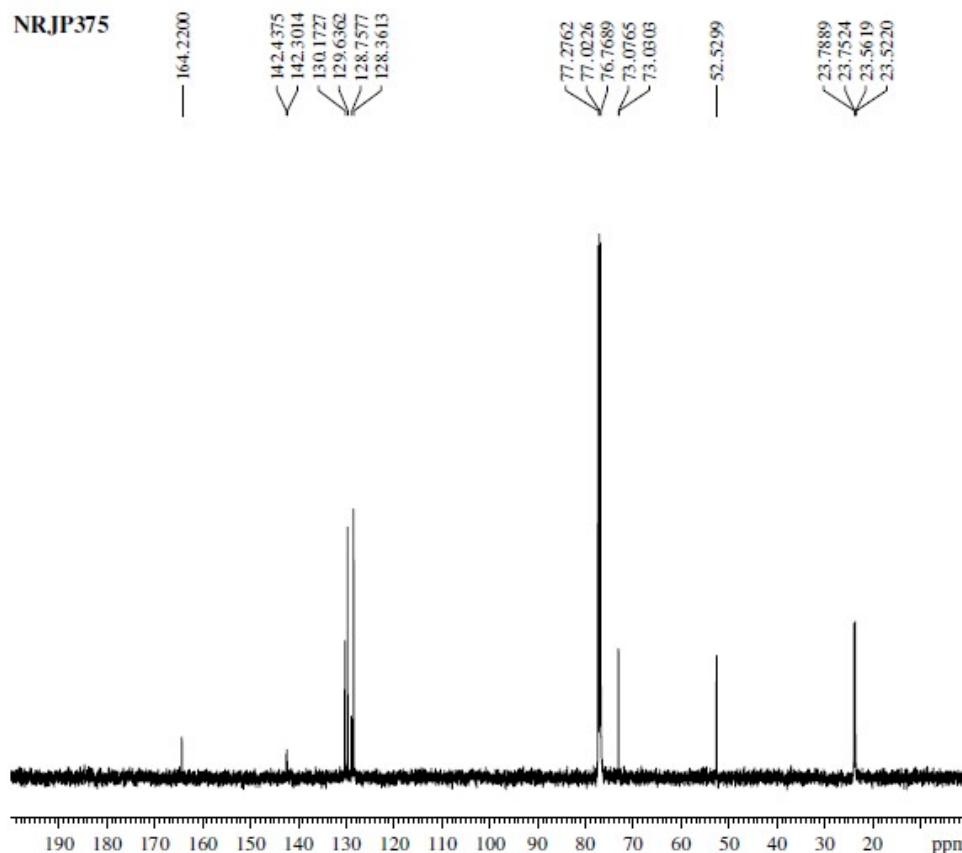
F2 - Acquisition Parameters
 Date_ 20230427
 Time 12.50
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 114.26
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605074 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S61: ¹H NMR spectrum of 3s

NRJP375



Current Data Parameters
 NAME 27-Apr-FN-2023
 EXPNO 510
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230427
 Time 11.08
 INSTRUM spect
 PROBHD 5 mm PATX1 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 107.78
 DW 16.800 usec
 DE 6.50 usec
 TE 303.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

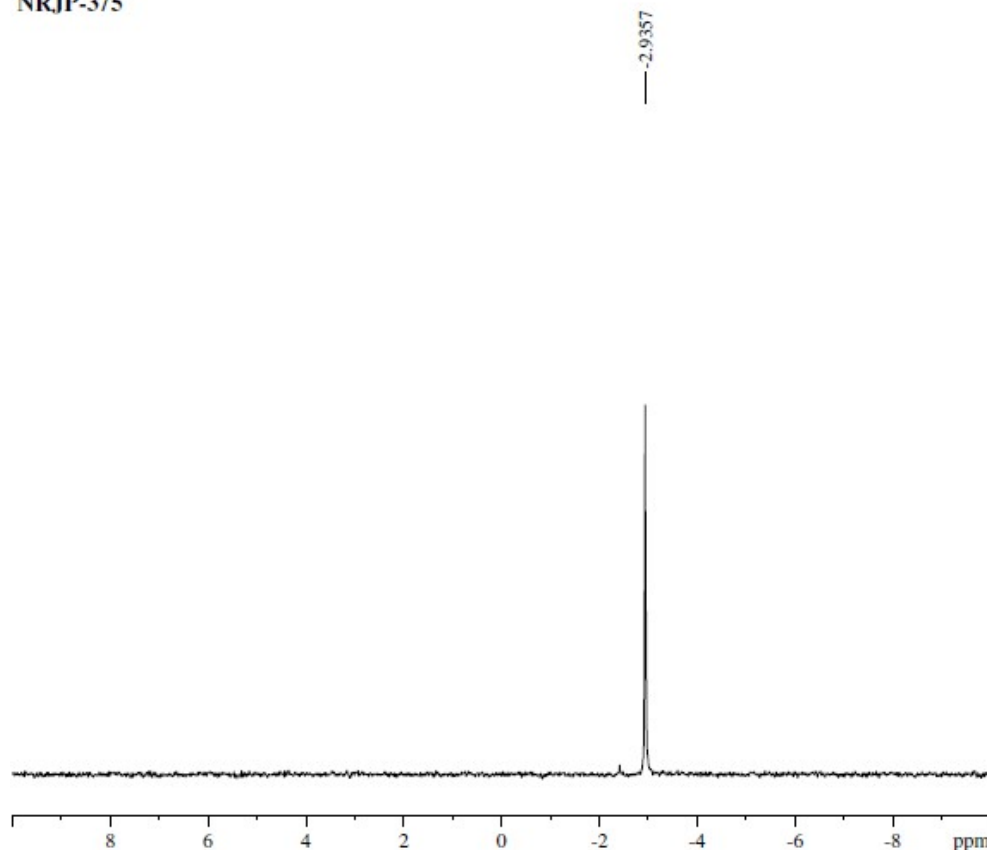
==== CHANNEL f1 ====
 SFO1 125.9077573 MHz
 NUC1 13C
 P1 9.23 usec
 PLW1 244.00000000 W

==== CHANNEL f2 ====
 SFO2 500.6783527 MHz
 NUC2 1H
 CPDPRG2 walz16
 PCPD2 80.00 usec
 PLW2 13.60000038 W
 PLW12 0.08840500 W
 PLW13 0.05657900 W

F2 - Processing parameters
 SI 32768
 SF 125.8951680 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

Figure S62: ¹³C{H} NMR spectrum of 3s

NRJP-375



Current Data Parameters
 NAME 21-Mar-AN-2023
 EXPNO 430
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230321
 Time 21.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

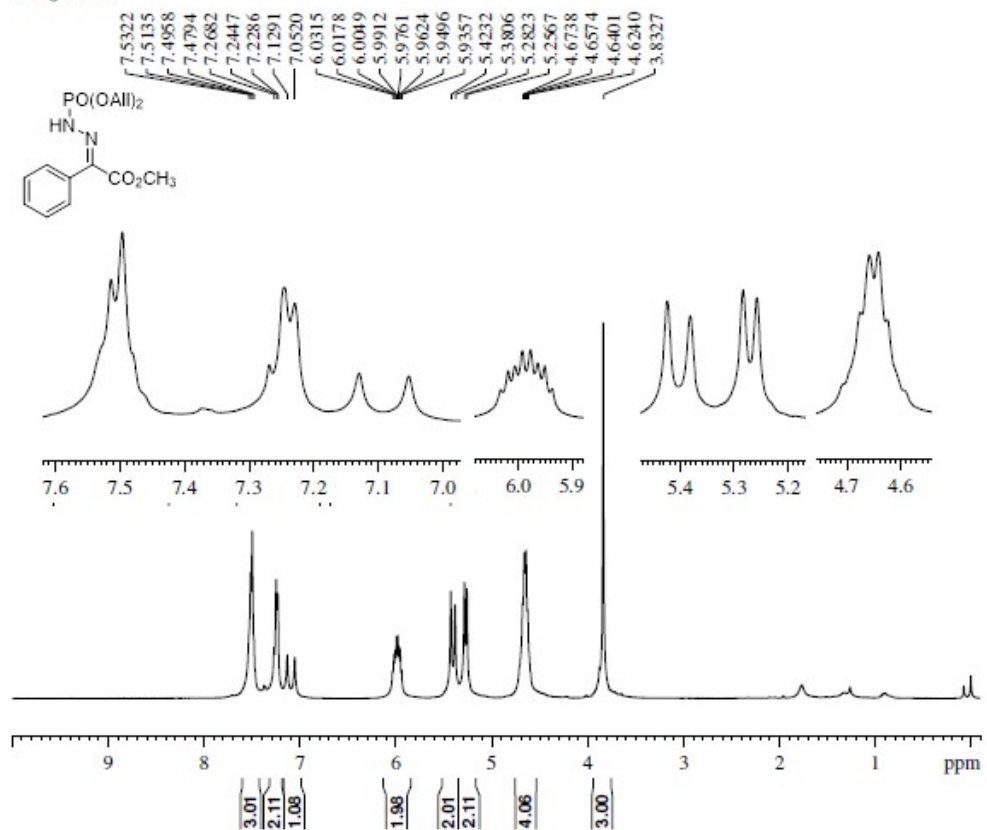
==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S63: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3s

NRJP-507



Current Data Parameters
 NAME 19-DEC-AN-2023
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20231219
 Time 17.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 89.7
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605065 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S64: ^1H NMR spectrum of 3t

NRJP-507

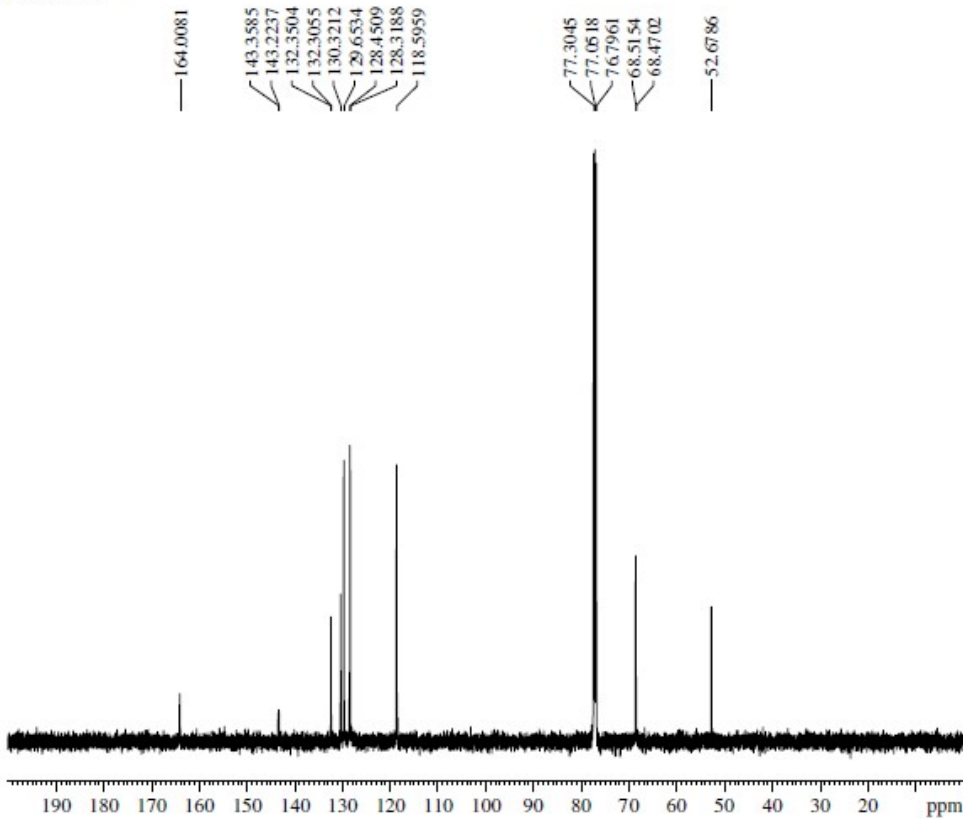


Figure S65: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3t



Current Data Parameters
 NAME 19-DEC-AN-2023
 EXPNO 330
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20231219
 Time 13.36
 INSTRUM spect
 PROBHD 5 mm PATXI 1H/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010048 sec
 RG 107.78
 DW 16.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 125.9077573 MHz
 NUC1 ^{13}C
 P1 9.23 usec
 PLW1 244.0000000 W

==== CHANNEL f2 =====
 SFO2 500.6783527 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 80.00 usec
 PLW2 13.60000038 W
 PLW12 0.08840500 W
 PLW13 0.05657900 W

F2 - Processing parameters
 SI 32768
 SF 125.8951680 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP-507

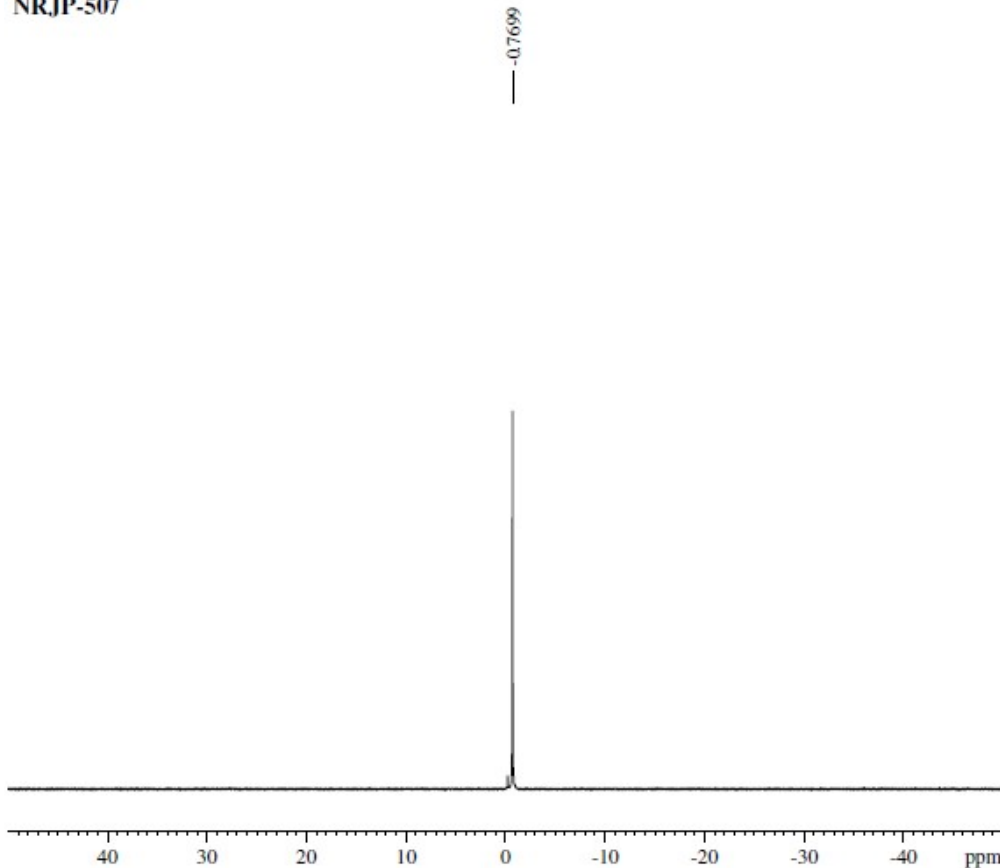


Figure S66: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3t



Current Data Parameters
 NAME 19-DEC-AN-2023
 EXPNO 320
 PROCNO 1

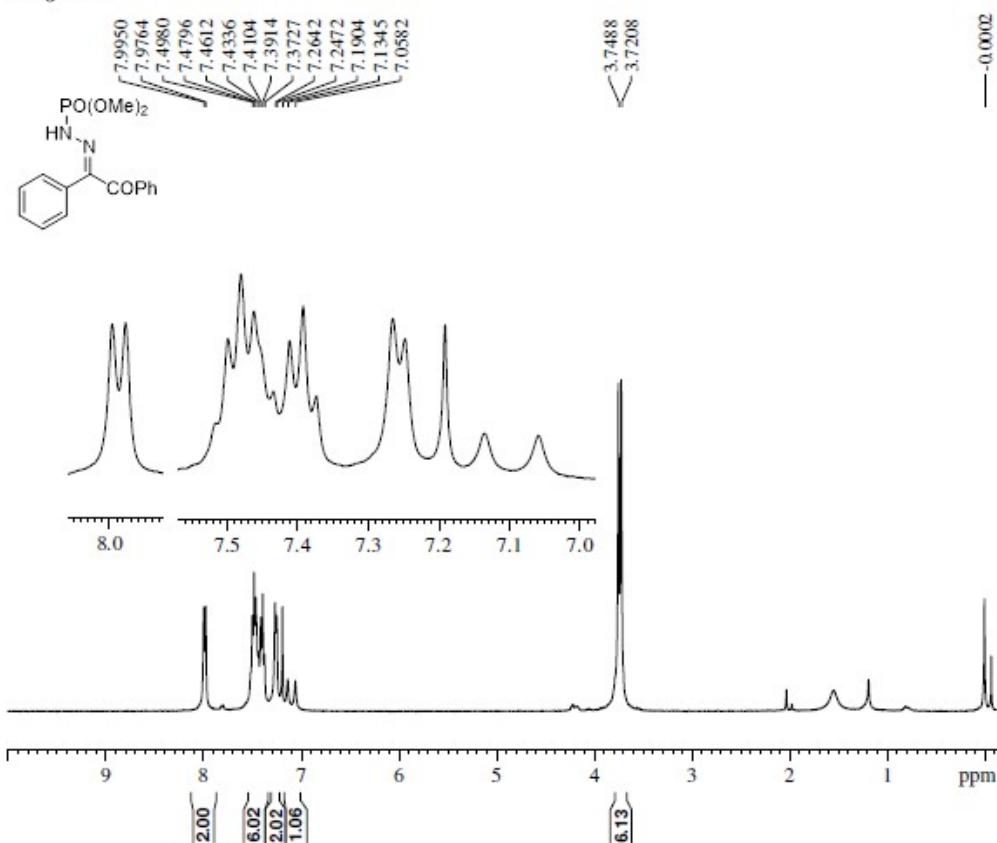
F2 - Acquisition Parameters
 Date_ 20231219
 Time 17.13
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 161.9798402 MHz
 NUC1 ^{31}P
 P1 15.00 usec
 PLW1 12.0000000 W

==== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP-527



Current Data Parameters
 NAME 19-FEB-AN-2023
 EXPNO 360
 PROCNO 1

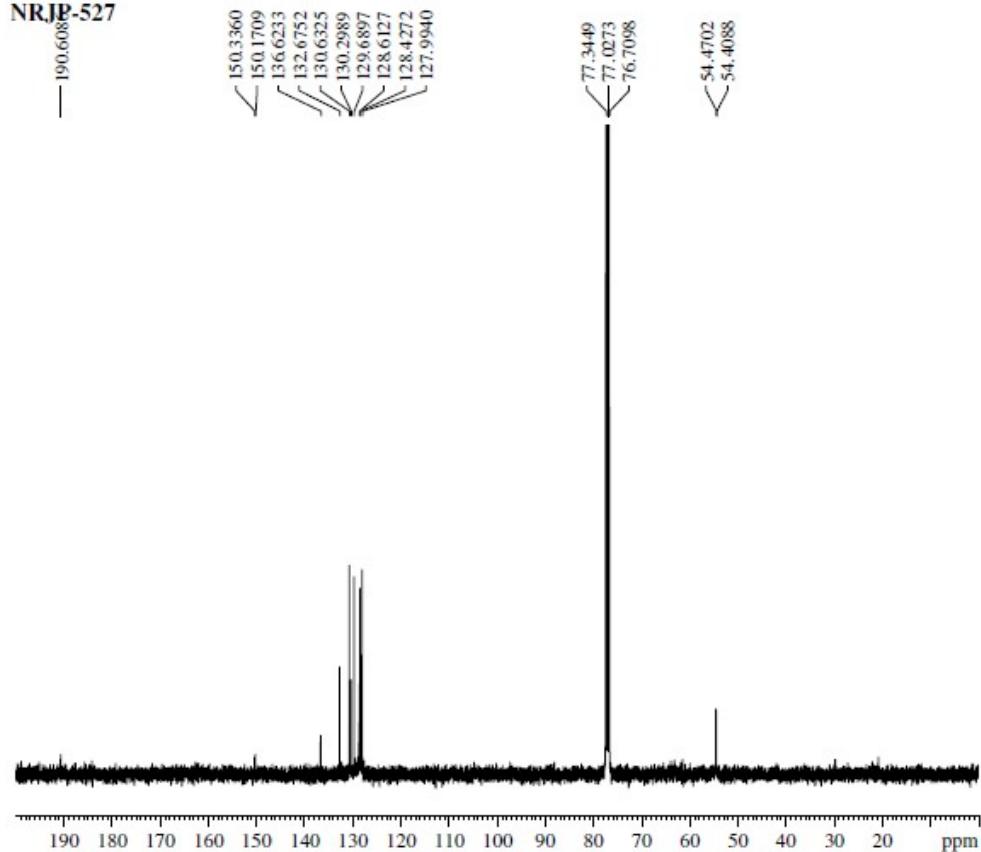
F2 - Acquisition Parameters
 Date_ 20240219
 Time 21.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 145.29
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TDO 1

CHANNEL f1
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605377 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S67: ¹H NMR spectrum of 3u

NRJP-527



Current Data Parameters
 NAME 22-FEB-AN-2024
 EXPNO 320
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20240222
 Time 17.17
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 575
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 D11 0.0300000 sec
 TDO 1

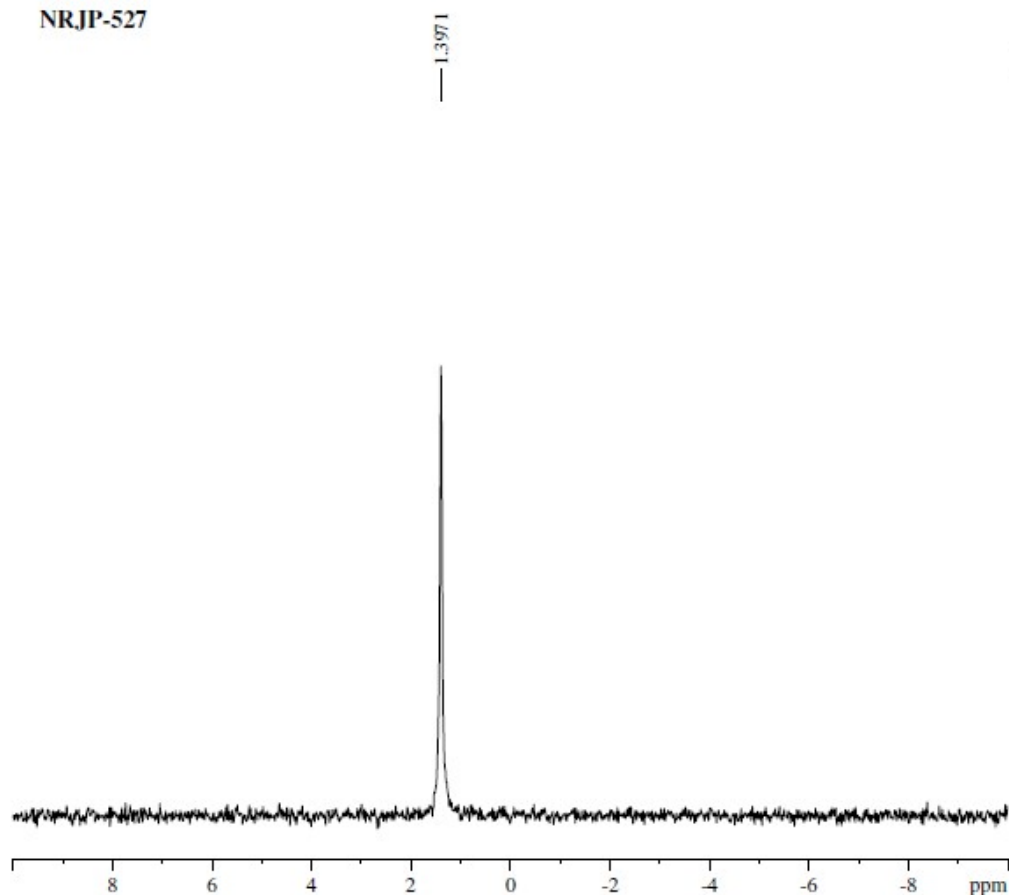
CHANNEL f1
 SFO1 100.6204380 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

CHANNEL f2
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S68: ¹³C{¹H} NMR spectrum of 3u

NRJP-527



Current Data Parameters
 NAME 21-FEB-FN-2024
 EXPNO 480
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20240221
 Time 12.03
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

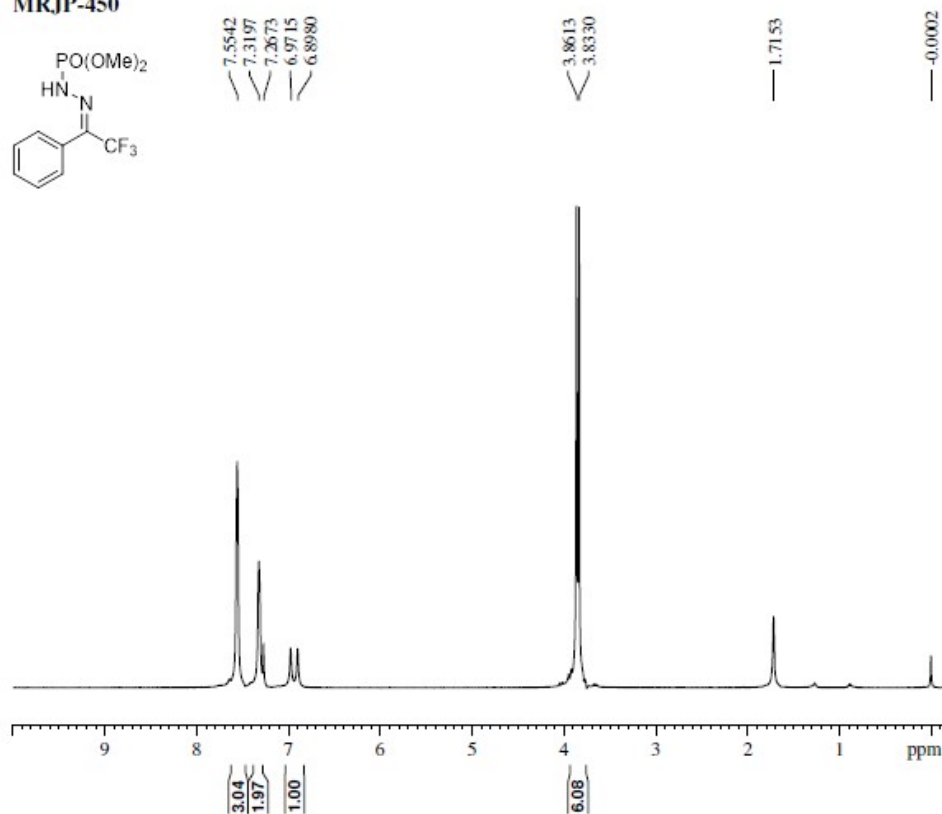
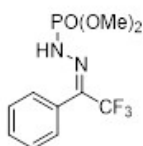
==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S69: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3u

MRJP-450



Current Data Parameters
 NAME 25-Jul--AN-2023
 EXPNO 460
 PROCNO 1

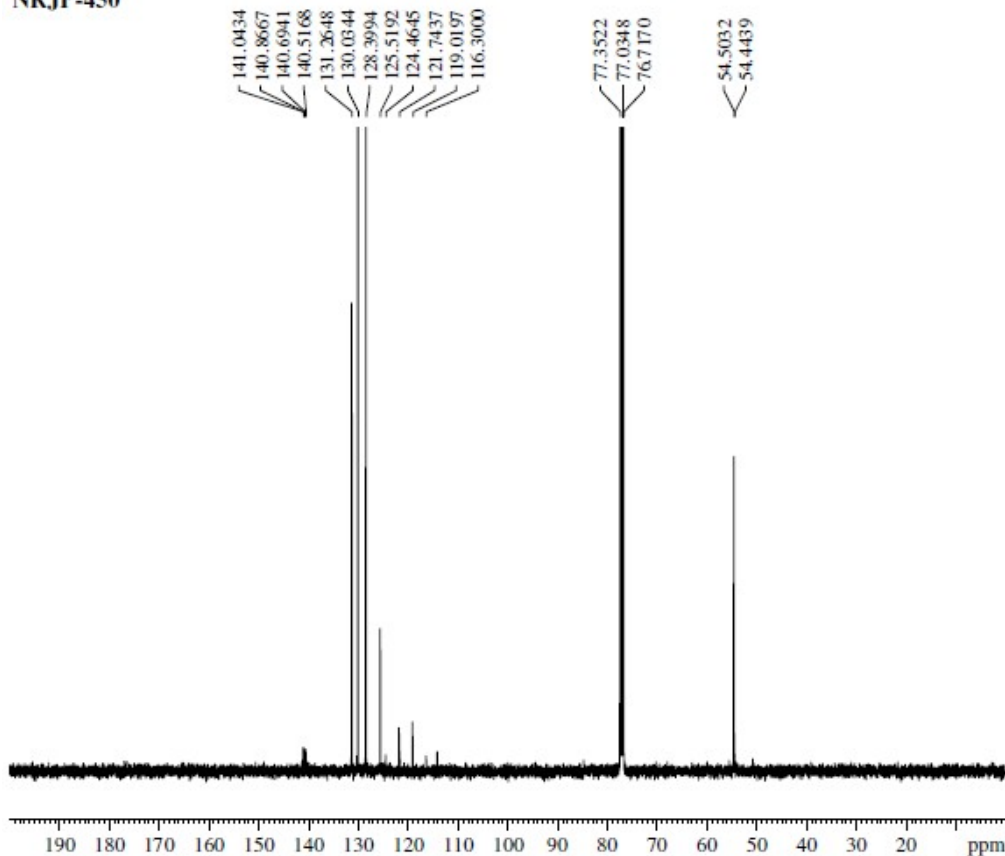
F2 - Acquisition Parameters
 Date_ 20230726
 Time 0.07
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 114.26
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.0000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605065 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S70: ^1H NMR spectrum of 3v

NRJP-450



Current Data Parameters
 NAME 27-JUL-AN-2023
 EXPNO 320
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230727
 Time 19.23
 INSTRUM spect
 PROBHD 5 mm PABBO BBV
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TDO 1

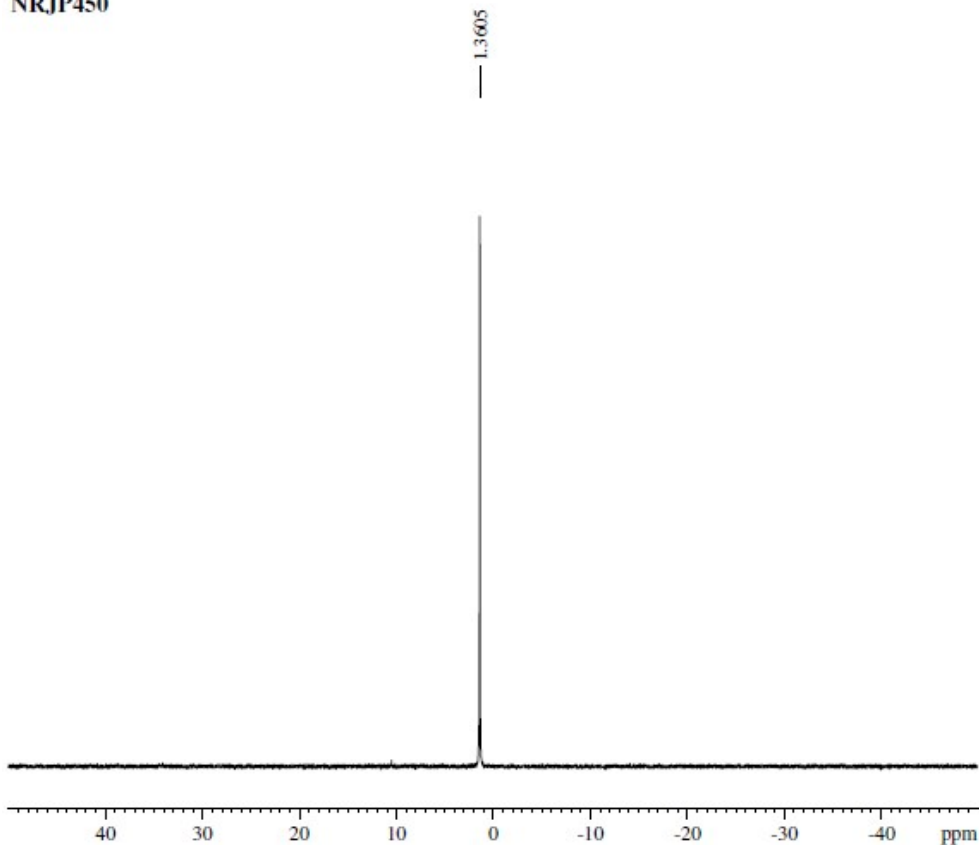
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S71: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3v

NRJP450



Current Data Parameters
 NAME 26-JUL-AN-2023
 EXPNO 360
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230726
 Time 17.49
 INSTRUM spect
 PROBHD 5 mm PABBO BBV
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.2 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TDO 1

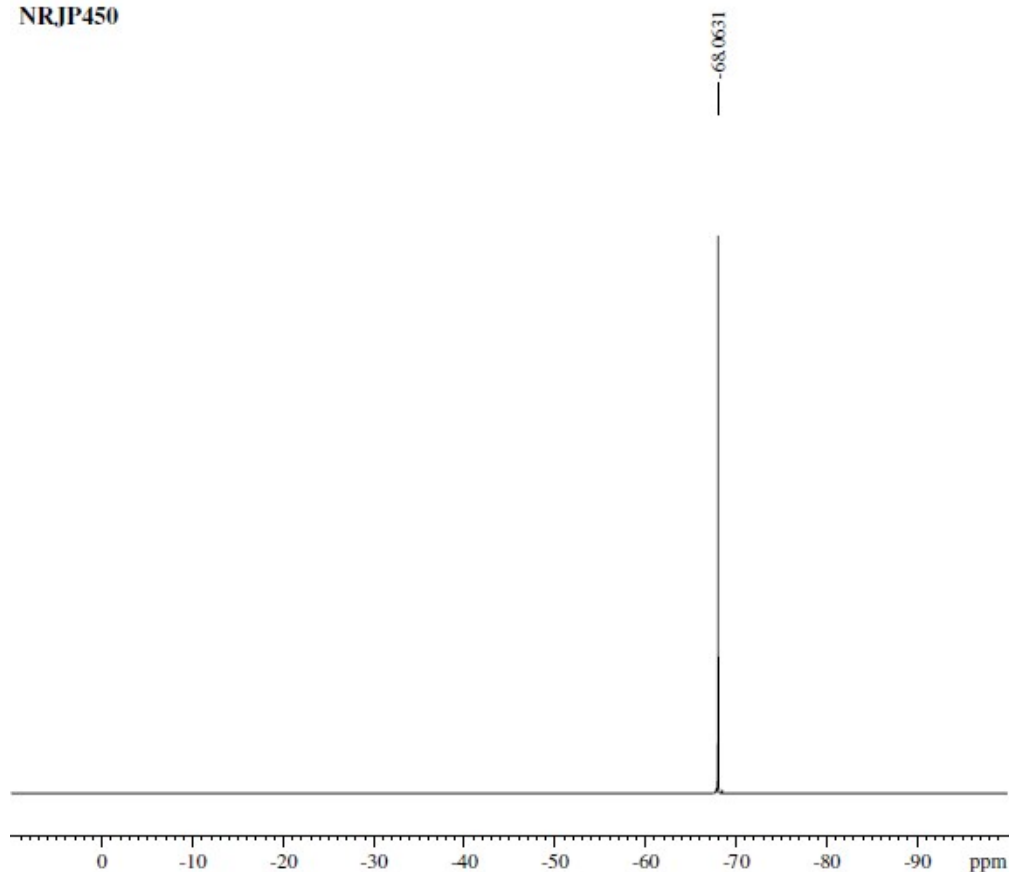
==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S72: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3v

NRJP450



Current Data Parameters
 NAME 26-JUL-AN-2023
 EXPNO 350
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230726
 Time 17.48
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 89285.711 Hz
 FIDRES 0.681196 Hz
 AQ 0.734032 sec
 RG 201.48
 DW 5.600 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 D12 0.00002000 sec
 TD0 1

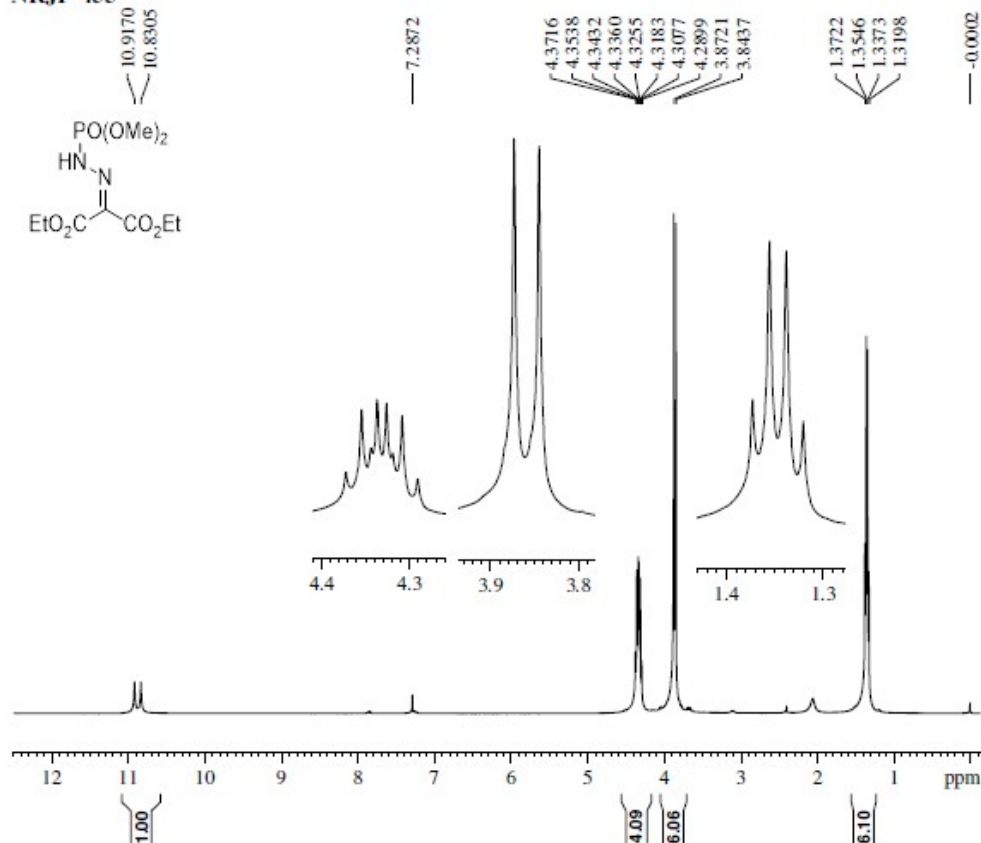
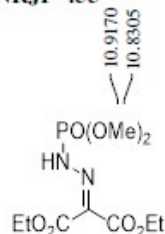
==== CHANNEL f1 ====
 SFO1 376.4894122 MHz
 NUC1 19F
 P1 15.00 usec
 PLW1 21.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 walz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W

F2 - Processing parameters
 SI 65536
 SF 376.5270650 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S73: $^{19}\text{F}\{\text{H}\}$ NMR spectrum of 3v

NRJP-455



Current Data Parameters
 NAME 07-AUG-AN-2023
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230807
 Time 17.09
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 73.53
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1604984 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S74: ^1H NMR spectrum of 3v

NRJP-455

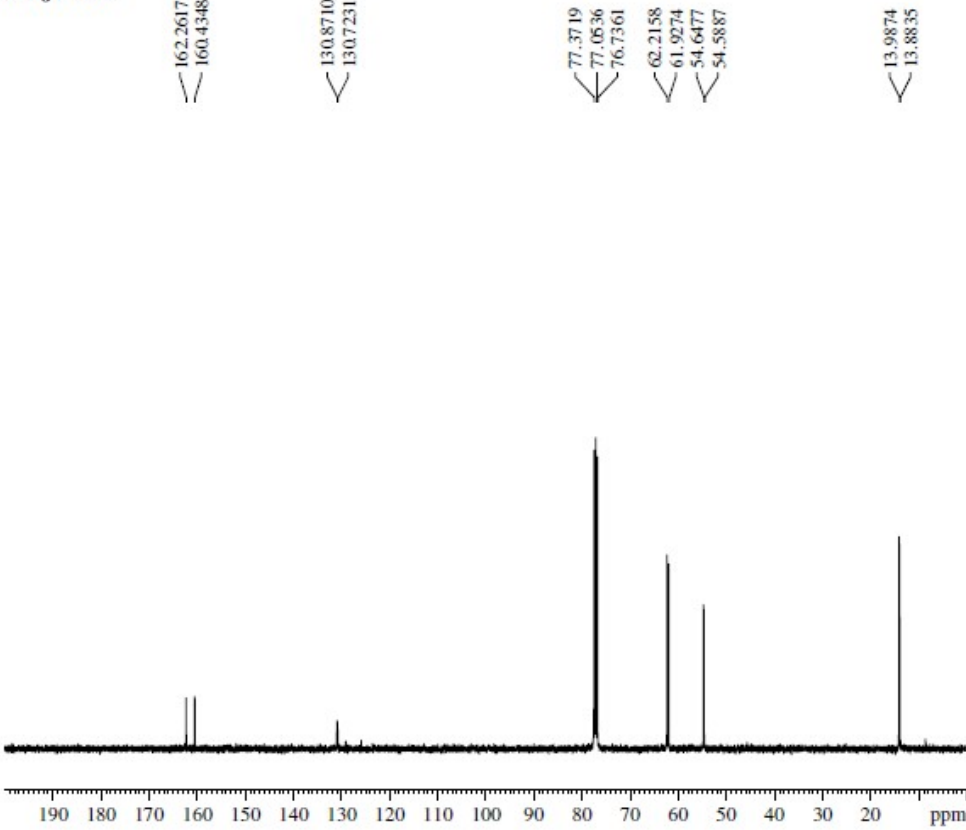


Figure S75: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 3w



Current Data Parameters
 NAME 08-AUG-AN-2023
 EXPNO 350
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230808
 Time 19.13
 INSTRUM spect
 PROBHD 5 mm PA-BBO BBV
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 ^{13}C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP-455

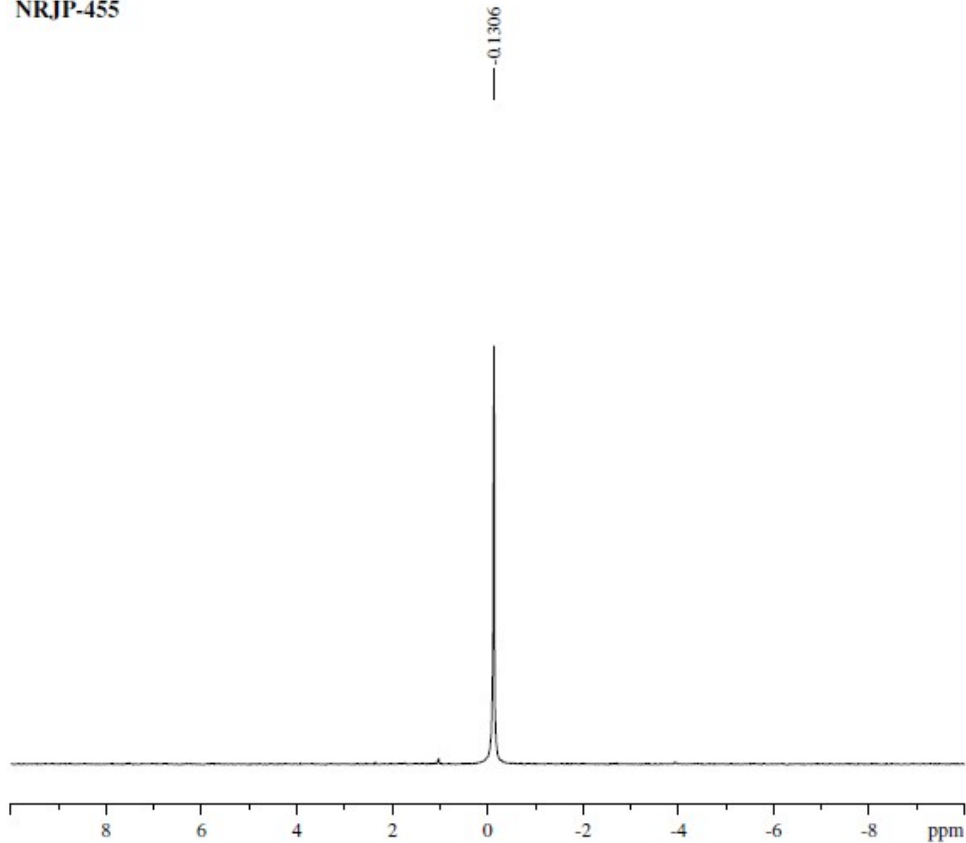


Figure S76: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3w



Current Data Parameters
 NAME 09-AUG-AN-2023
 EXPNO 440
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230809
 Time 23.28
 INSTRUM spect
 PROBHD 5 mm PA-BBO BBV
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 ^{31}P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 ^1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

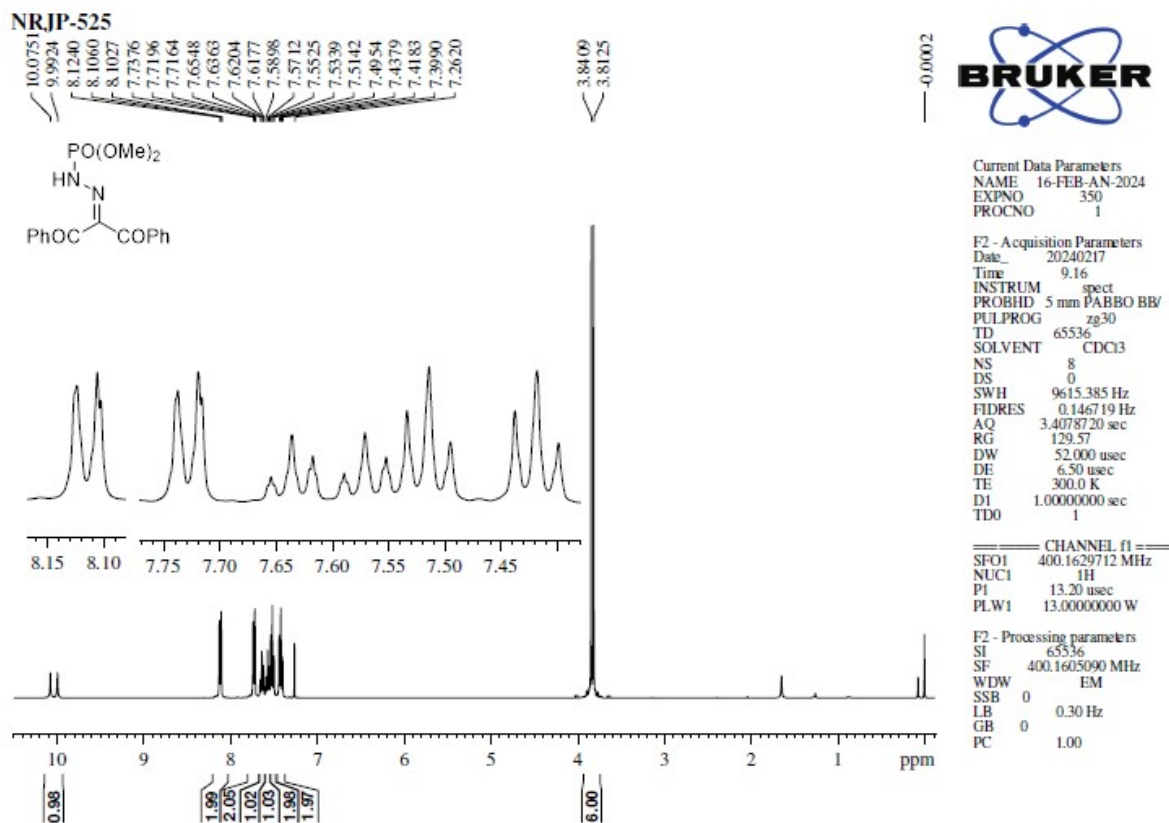


Figure S77: ¹H NMR spectrum of 3x

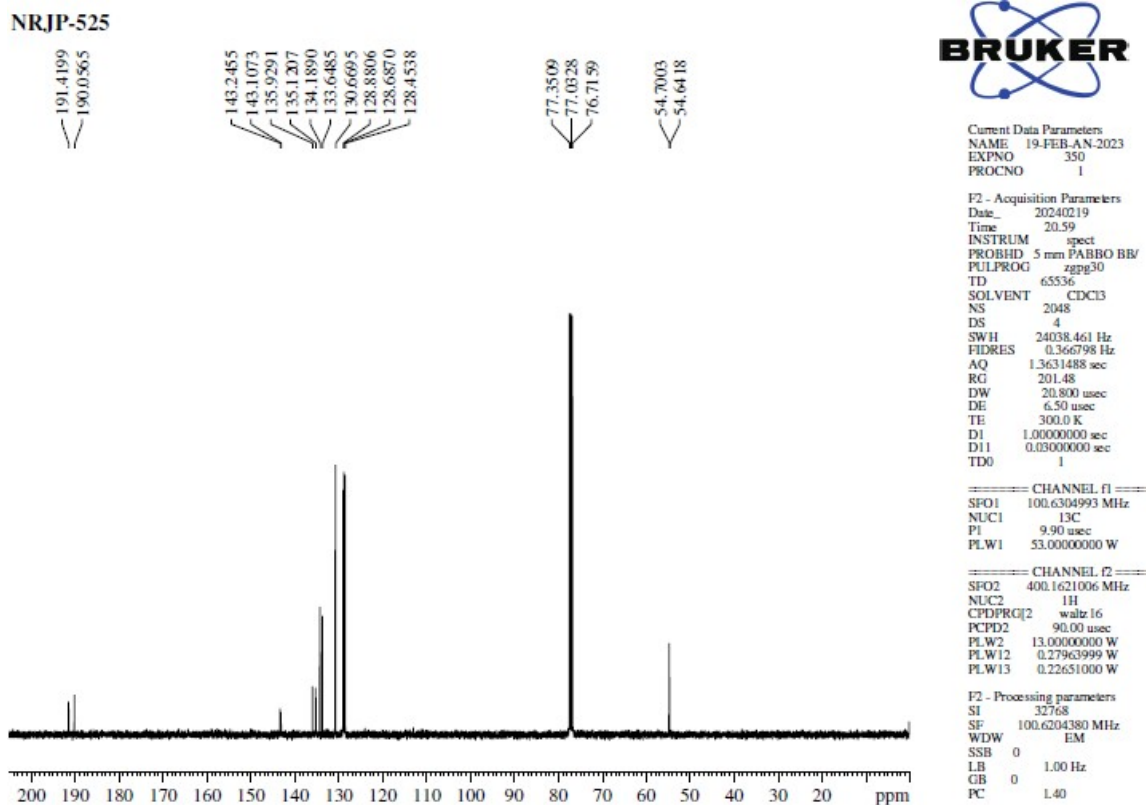
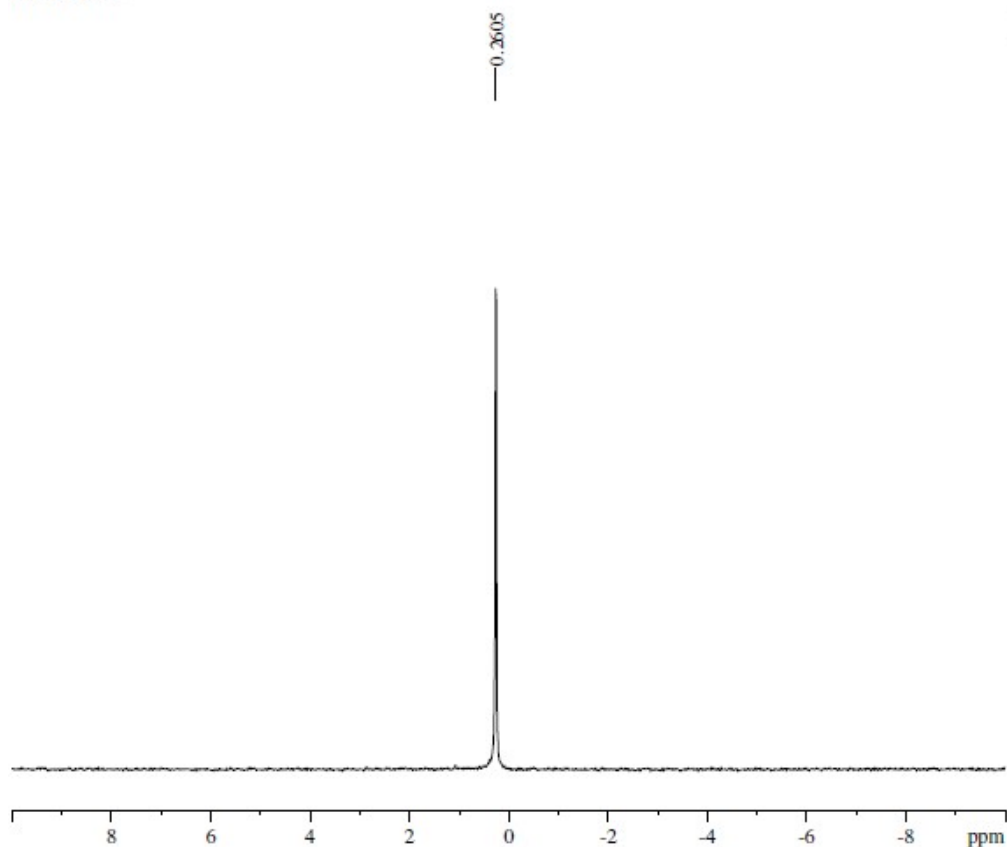


Figure S78: ¹³C{¹H} NMR spectrum of 3x

NRJP-525



Current Data Parameters
 NAME 16-FEB-AN-2024
 EXPNO 360
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20240217
 Time 9.19
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 A Q 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

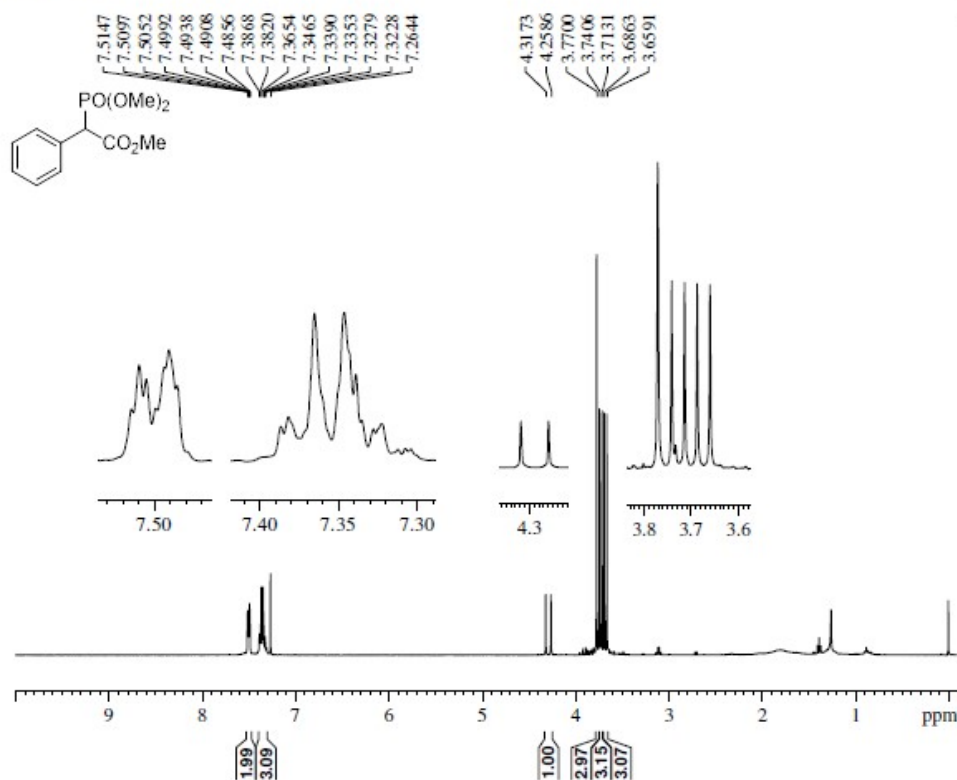
==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.279639999 W
 PLW13 0.226510000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S79: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 3x

NRJP-353



Current Data Parameters
 NAME 12-Apr-FN-2023
 EXPNO 320
 PROCNO 1

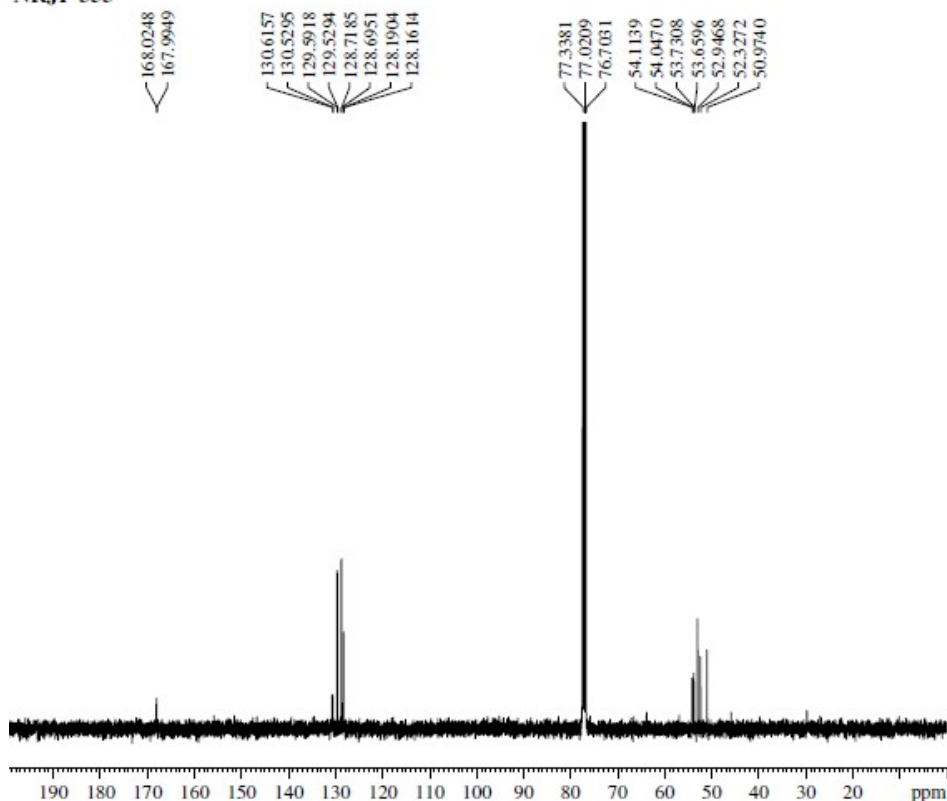
F2 - Acquisition Parameters
 Date_ 20230412
 Time 19.33
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 A Q 3.4078720 sec
 RG 129.57
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605079 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S80: ^1H NMR spectrum of 4a

NRJP 353



Current Data Parameters
 NAME 18-Apr-AN-2023
 EXPNO 480
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230419
 Time 2.40
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

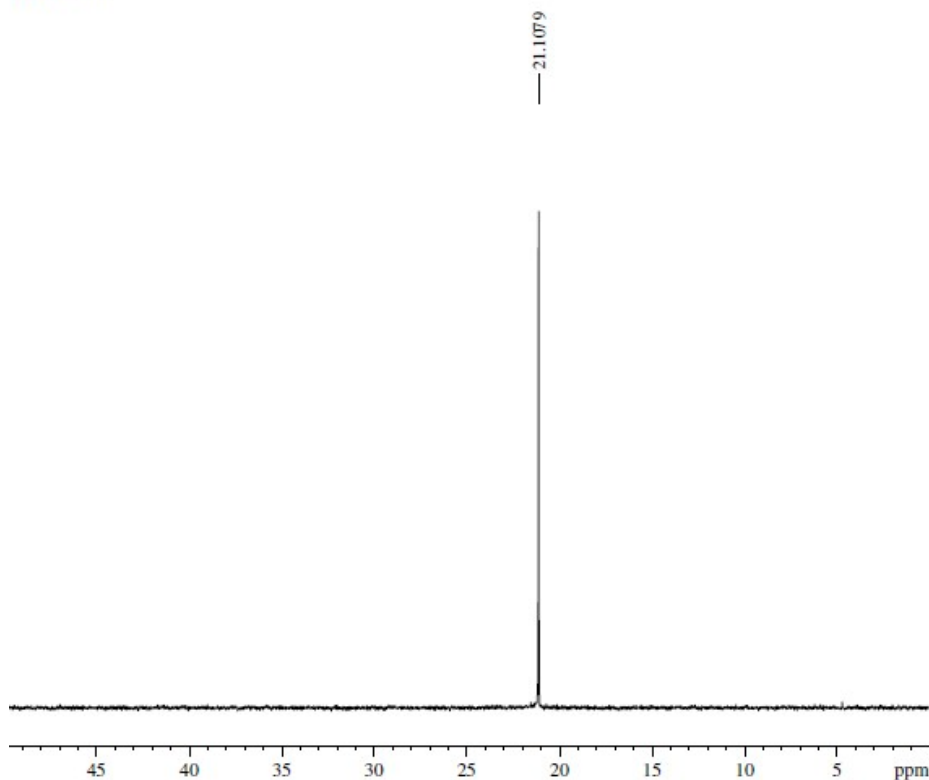
==== CHANNEL f1 =====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S81: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 4a

NRJP 353



Current Data Parameters
 NAME 13-Apr-AN-2023
 EXPNO 420
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230414
 Time 8.18
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

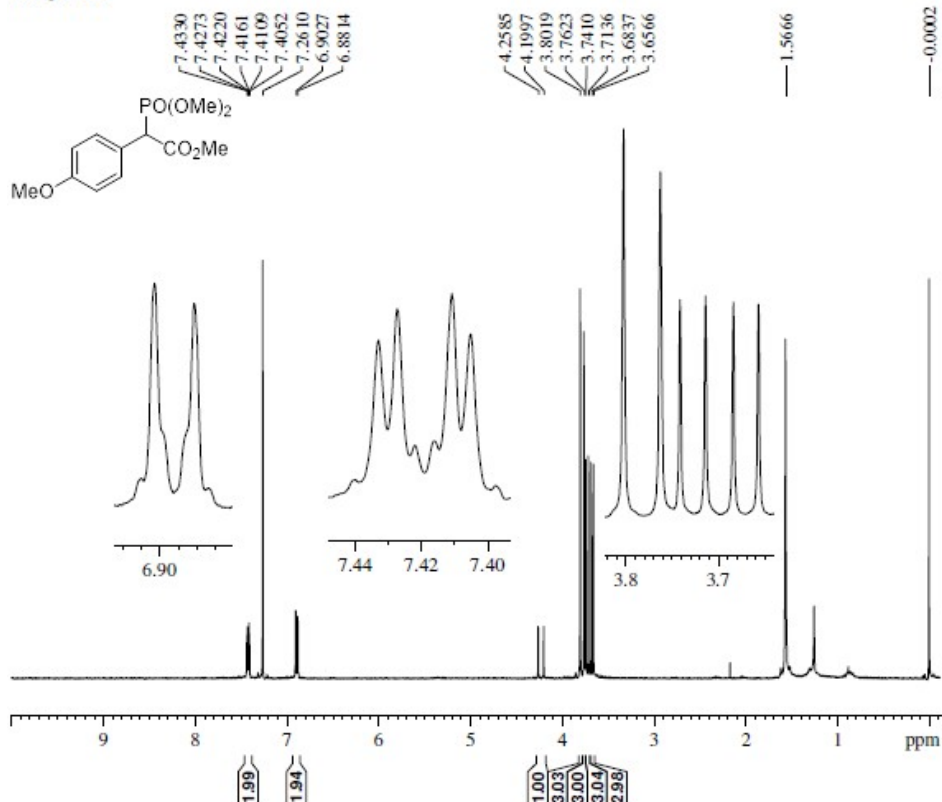
==== CHANNEL f1 =====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S82: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 4a

NRJP 415



Current Data Parameters
 NAME 31-MAY-AN-2023
 EXPNO 320
 PROCNO 1

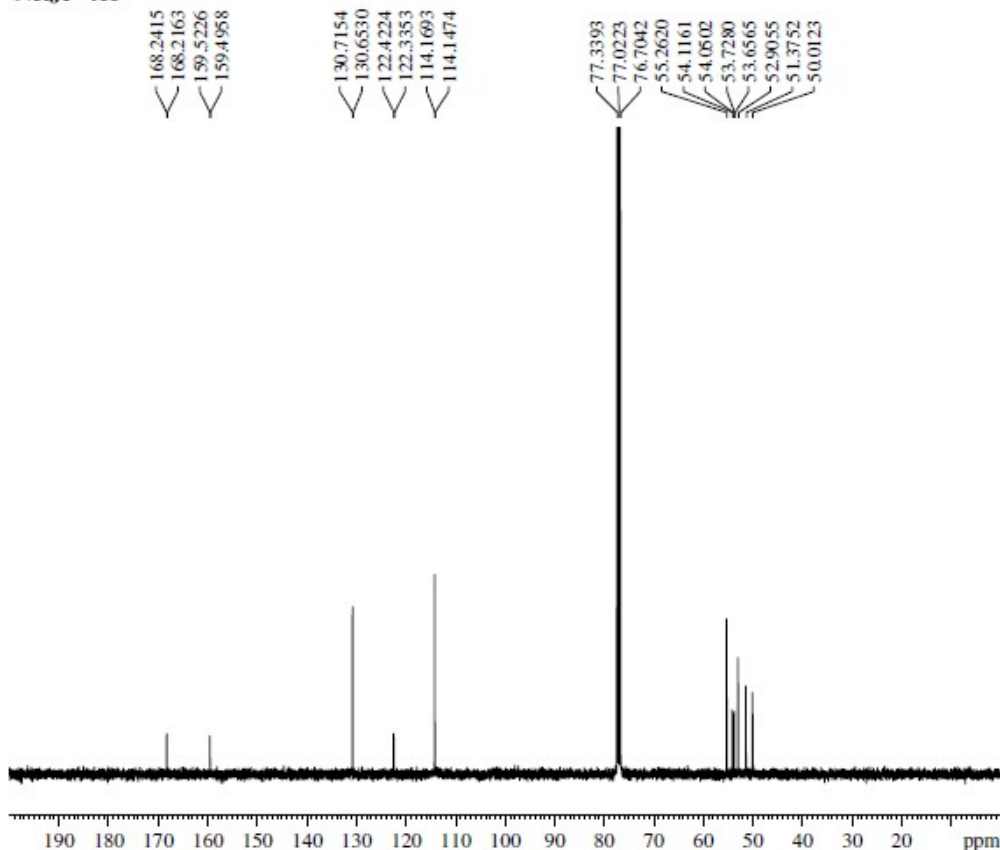
F2 - Acquisition Parameters
 Date_ 20230531
 Time 18.51
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 179.93
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605094 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S83: ¹H NMR spectrum of 4b

NRJP-415



Current Data Parameters
 NAME 22-May-AN-2023
 EXPNO 310
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230522
 Time 19.47
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

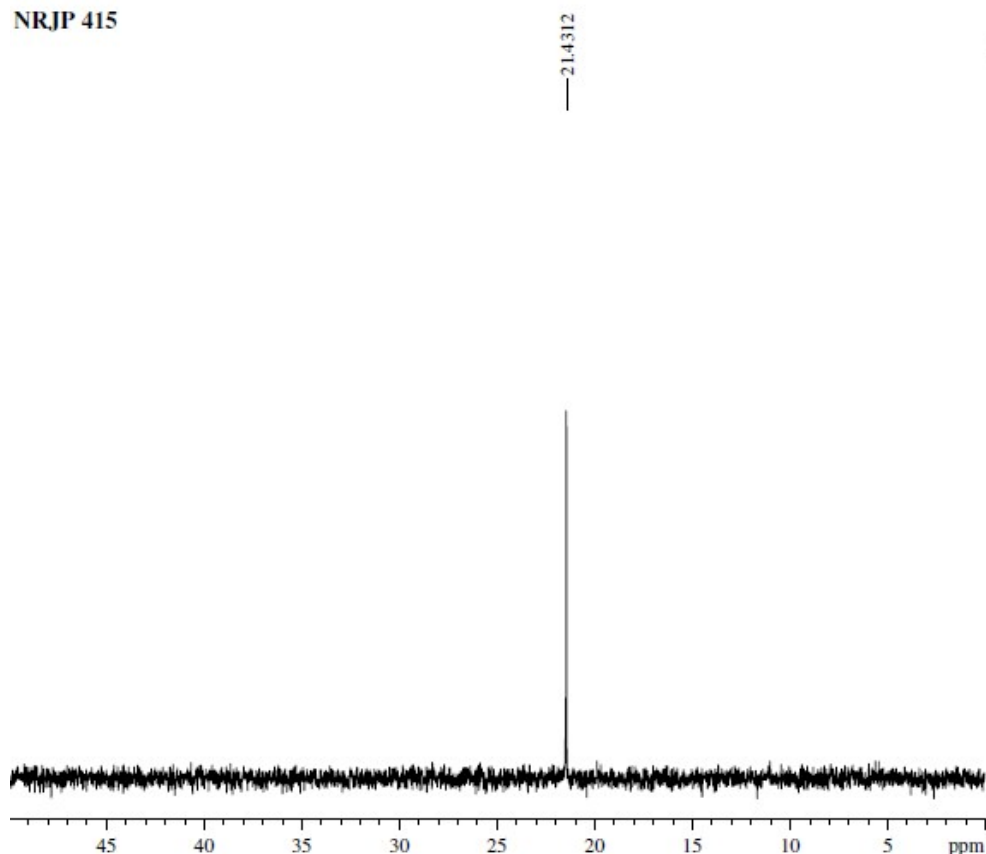
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S84: ¹³C{¹H} NMR spectrum of 4b

NRJP 415



Current Data Parameters
 NAME 07-JUN-AN-2023
 EXPNO 350
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230607
 Time 22.09
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

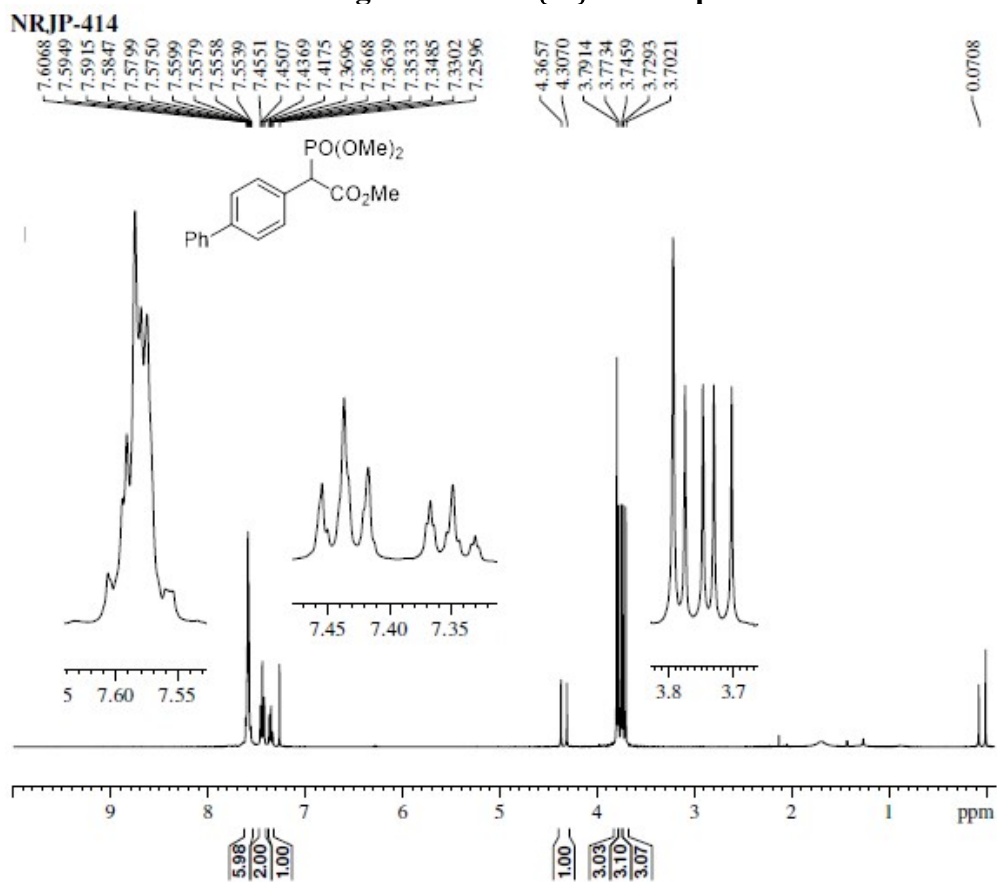
==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 walz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S85: ³¹P{H} NMR spectrum of 4b

NRJP-414



Current Data Parameters
 NAME 18-Dec-AN-2023
 EXPNO 350
 PROCNO 1

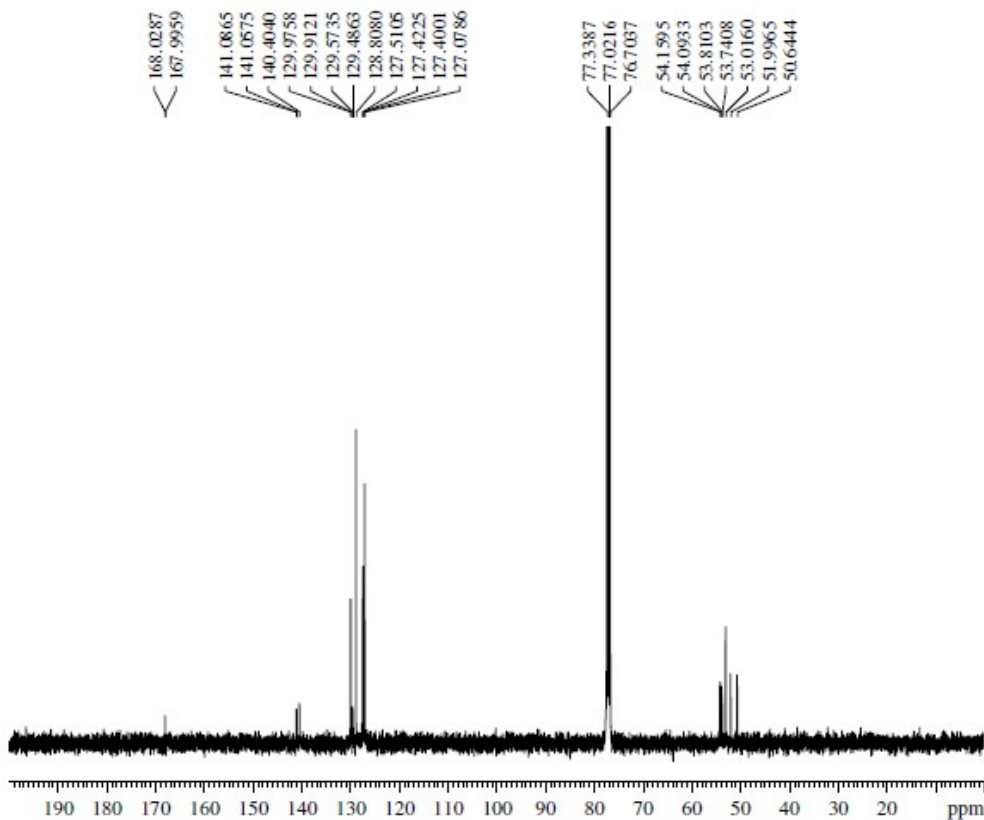
F2 - Acquisition Parameters
 Date_ 20231218
 Time 21.45
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 145.29
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605097 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S86: ¹H NMR spectrum of 4c

NRJP-414



Current Data Parameters
 NAME 19-May-AN-2023
 EXPNO 420
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230520
 Time 14.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.363798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TDO 1

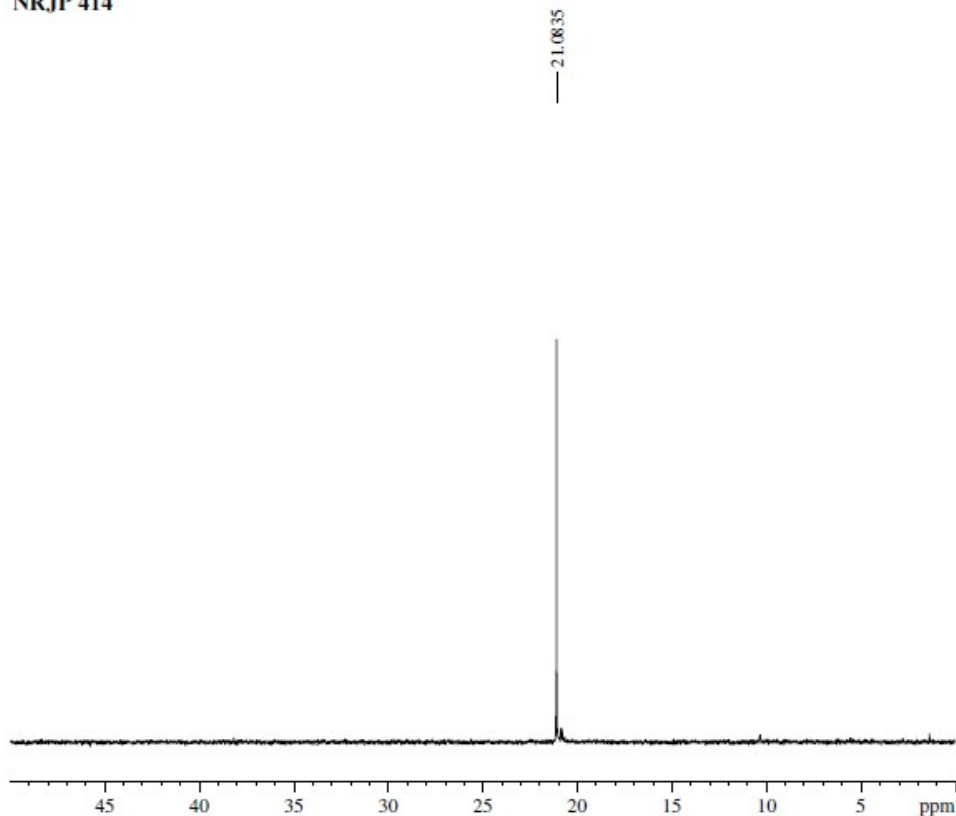
==== CHANNEL f1 =====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.0000000 W

==== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S87: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 4c

NRJP 414



Current Data Parameters
 NAME 23-May-AN-2023
 EXPNO 580
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230523
 Time 13.36
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

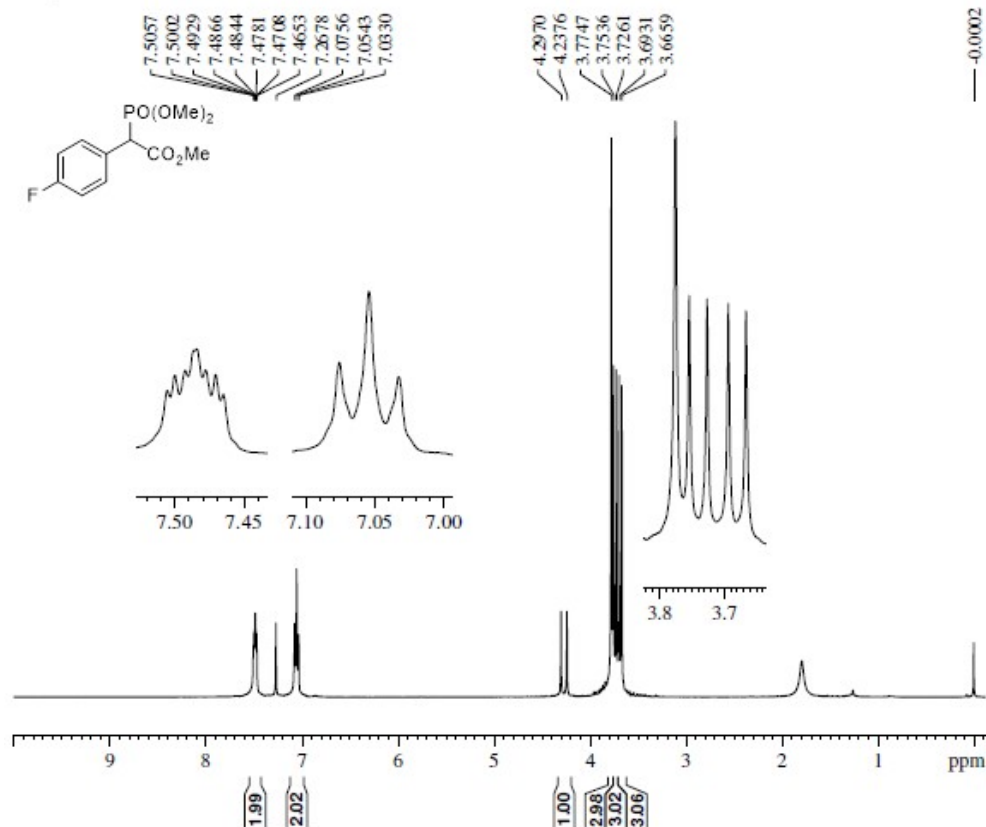
==== CHANNEL f1 =====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.0000000 W

==== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S88: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 4c

NRJP 407



Current Data Parameters
 NAME 06-JUL-AN-2023
 EXPNO 400
 PROCNO 1

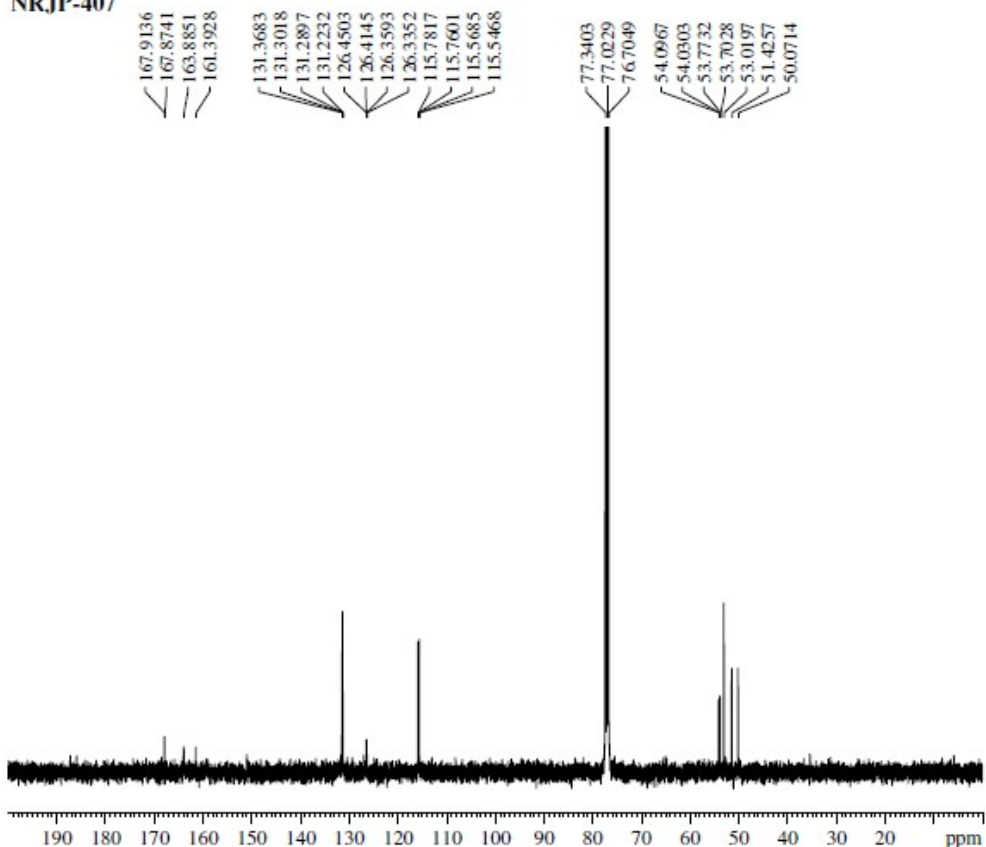
F2 - Acquisition Parameters
 Date_ 20230706
 Time 22.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 129.57
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

CHANNEL f1 ==
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605067 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S89: ^1H NMR spectrum of 4d

NRJP-407



Current Data Parameters
 NAME 04-JUL-AN-2023
 EXPNO 410
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230704
 Time 22.34
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

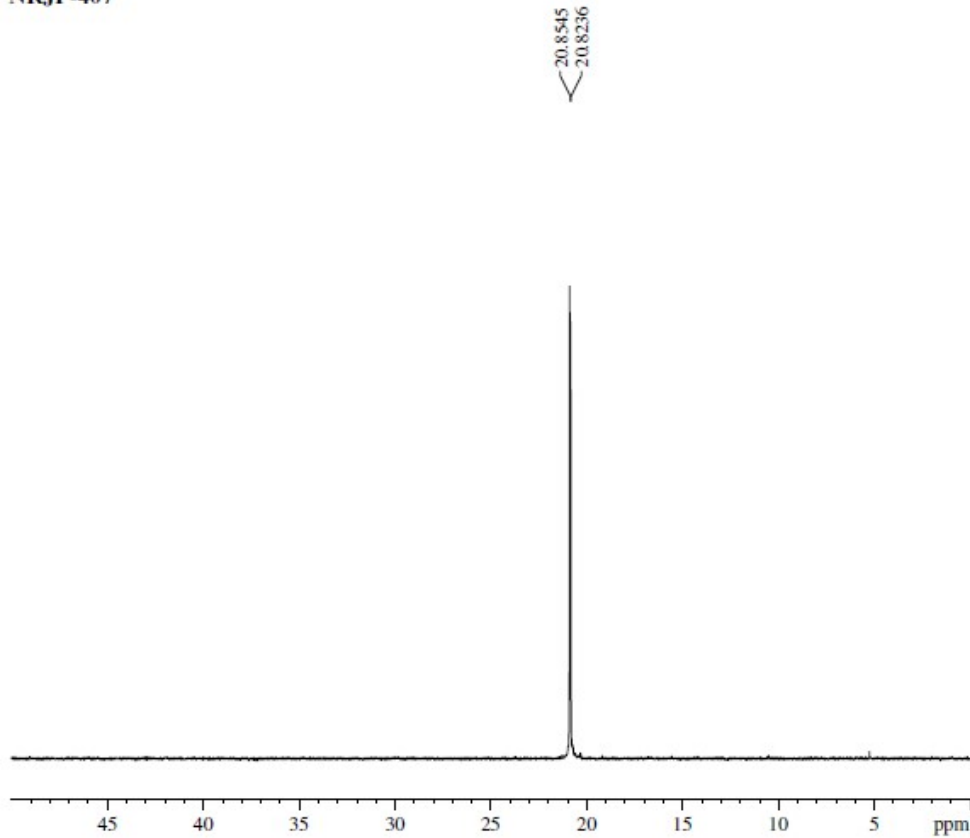
CHANNEL f1 ==
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

CHANNEL f2 ==
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 walz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S90: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of 4d

NRJP-407



Current Data Parameters
NAME 23-May-AN-2023
EXPNO 400
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230523
Time 19.47
INSTRUM spect
PROBHD 5 mm PABBO BV
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 64102.563 Hz
FIDRES 0.978127 Hz
AQ 0.5111808 sec
RG 201.48
DW 7.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

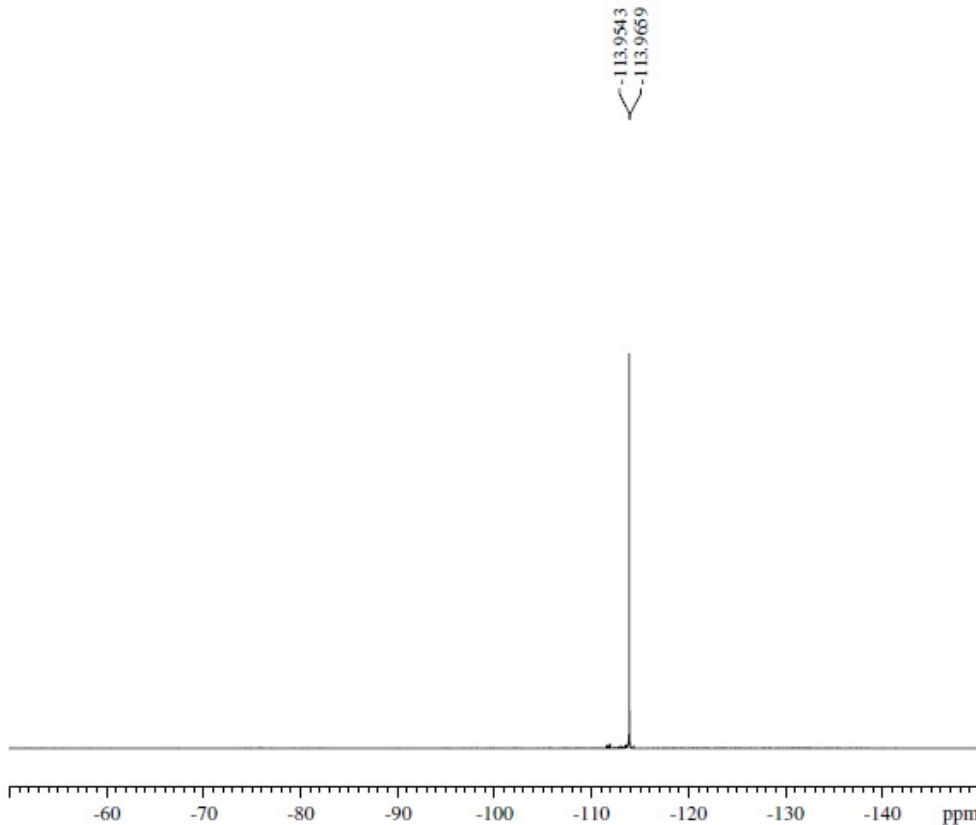
==== CHANNEL f1 ====
SFO1 161.9798402 MHz
NUC1 31P
P1 15.00 usec
PLW1 12.00000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.27963999 W
PLW13 0.22651000 W

F2 - Processing parameters
SI 32768
SF 161.9879400 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S91: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 4d

NRJP 407



Current Data Parameters
NAME 19-May-A-N-2023
EXPNO 430
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230519
Time 12.25
INSTRUM spect
PROBHD 5 mm PABBO BV
PULPROG zgfhgqn.2
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 89285.711 Hz
FIDRES 0.681196 Hz
AQ 0.7340032 sec
RG 201.48
DW 5.600 usec
DE 6.50 usec
TE 300.1 K
D1 1.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
TDO 1

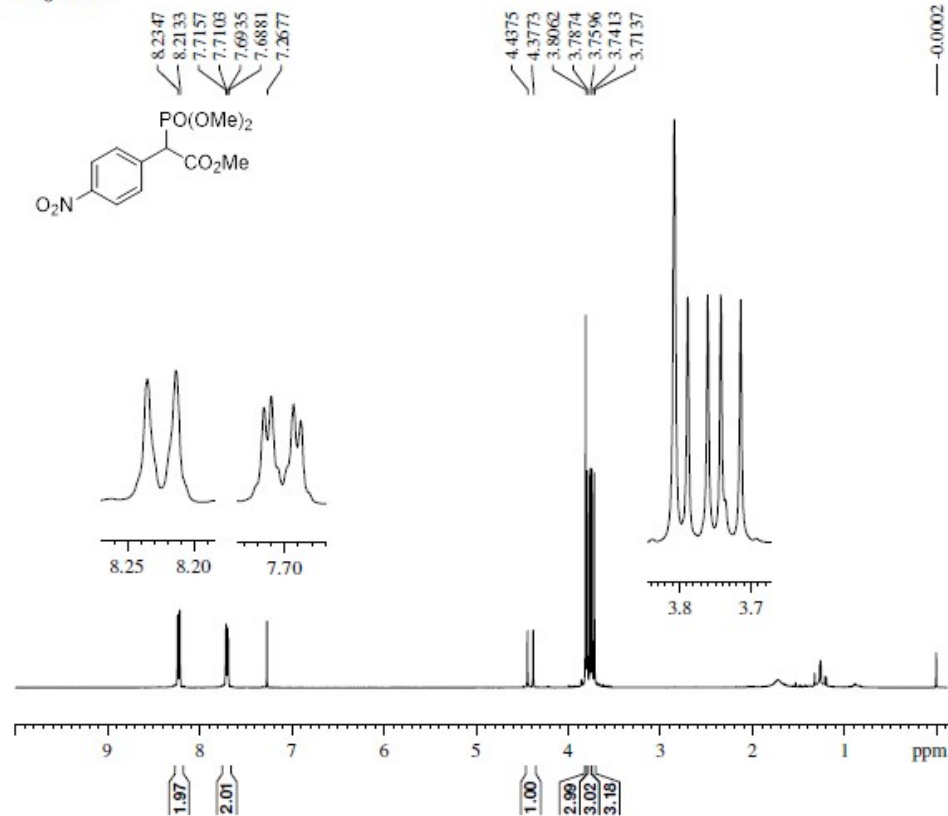
==== CHANNEL f1 ====
SFO1 376.4894122 MHz
NUC1 19F
P1 15.00 usec
PLW1 21.00000000 W

==== CHANNEL f2 ====
SFO2 400.1621006 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.27963999 W

F2 - Processing parameters
SI 65536
SF 376.5270650 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S92: $^{19}\text{F}\{\text{H}\}$ NMR spectrum of 4d

NRJP-419



Current Data Parameters
 NAME 16-MAY-AN-2023
 EXPNO 460
 PROCNO 1

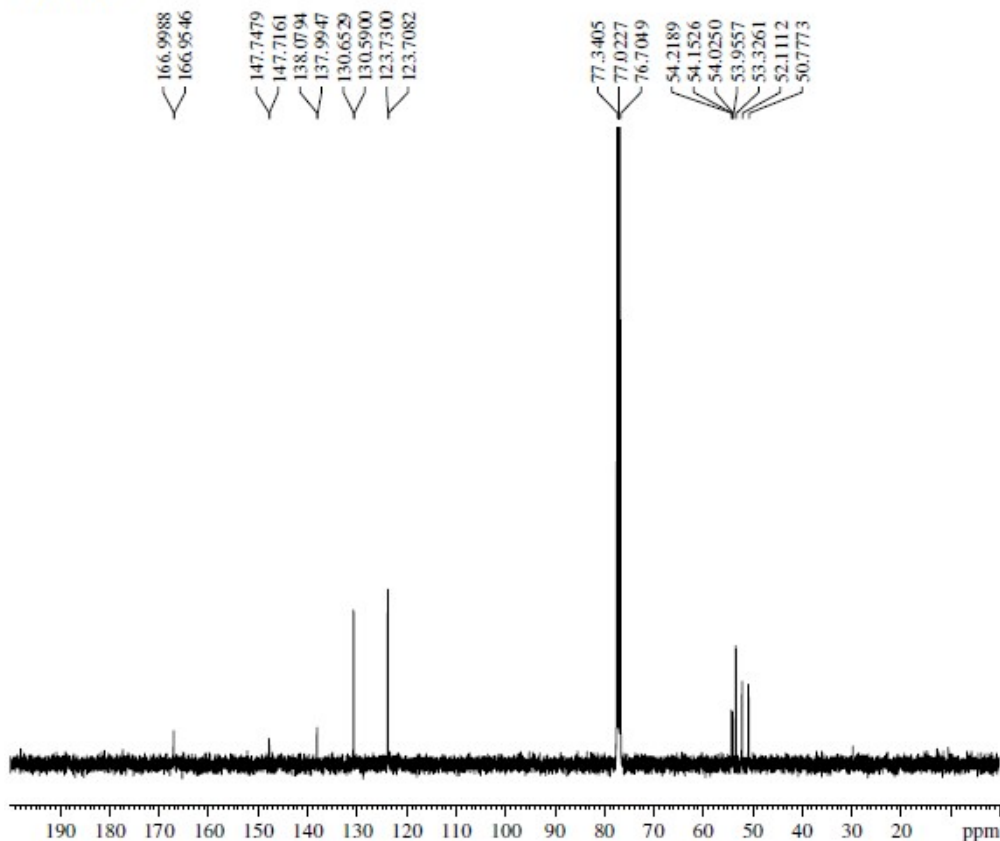
F2 - Acquisition Parameters
 Date_ 20230516
 Time 23.44
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 129.57
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D10 1

===== CHANNEL f1 =====
 SFO1 400.1629712 MHz
 NUC1 1H
 PI 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605066 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S93: ¹H NMR spectrum of 4e

NRJP-419



Current Data Parameters
 NAME 18-May-AN-2023
 EXPNO 400
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230519
 Time 2.40
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

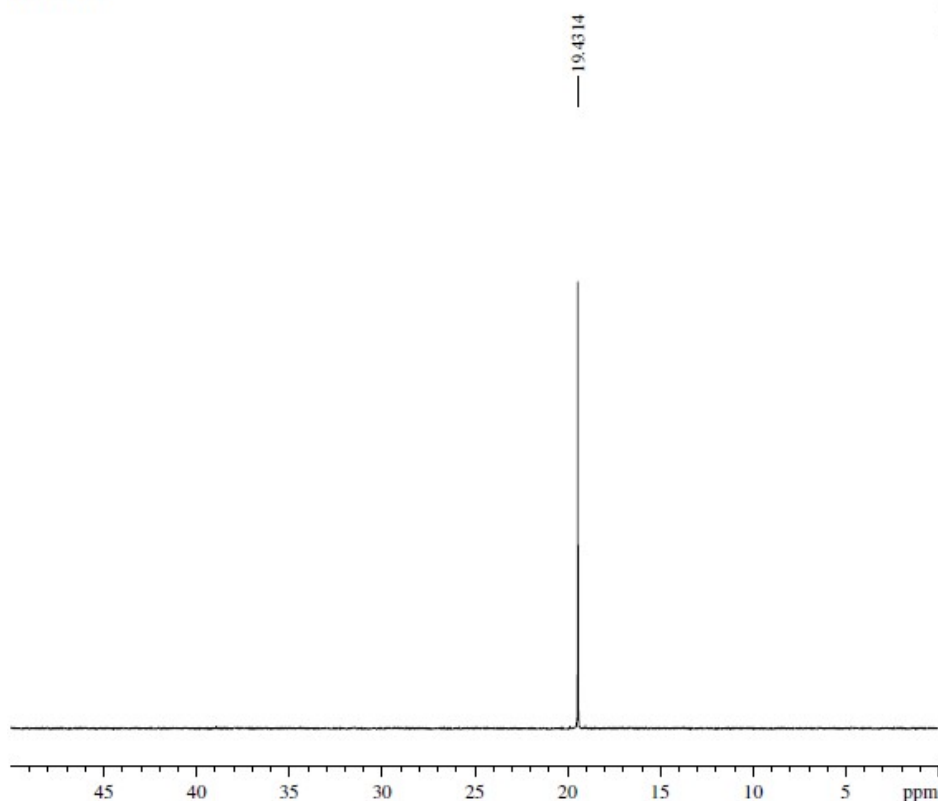
===== CHANNEL f1 =====
 SFO1 100.6304993 MHz
 NUC1 13C
 PI 9.90 usec
 PLW1 53.00000000 W

===== CHANNEL f2 =====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S94: ¹³C{¹H} NMR spectrum of 4e

NRJP 419



Current Data Parameters
 NAME 23-May-AN-2023
 EXPNO 560
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230523
 Time 13.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.511808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TD0 1

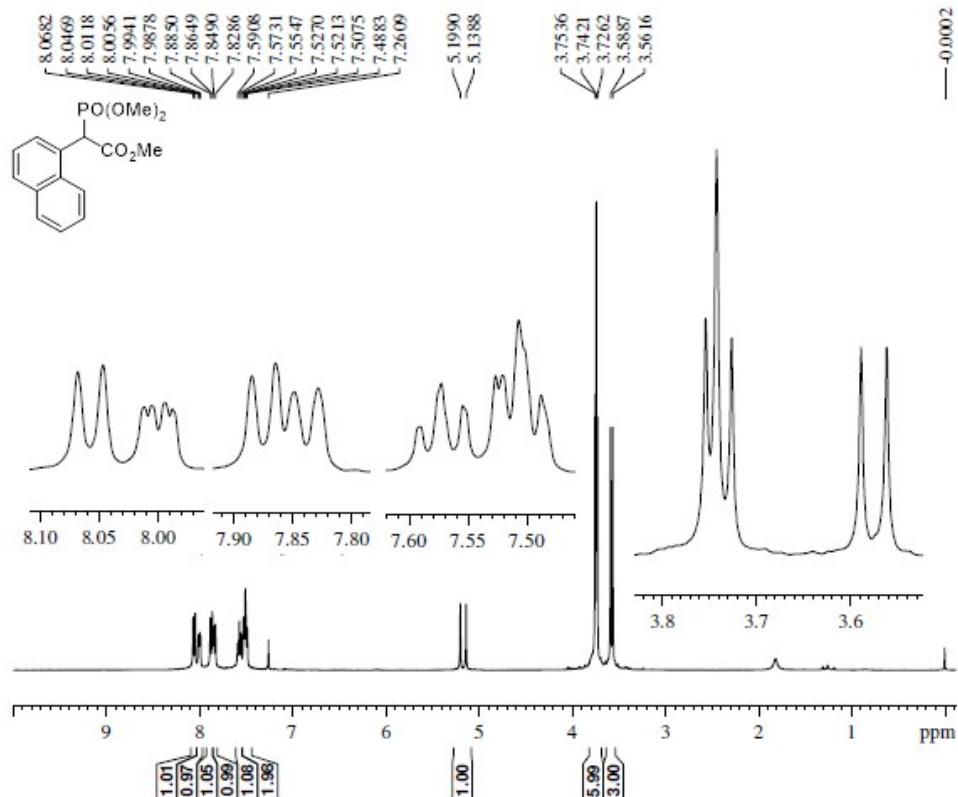
==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S95: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 4e

NRJP-421



Current Data Parameters
 NAME 16-MAY-AN-2023
 EXPNO 450
 PROCNO 1

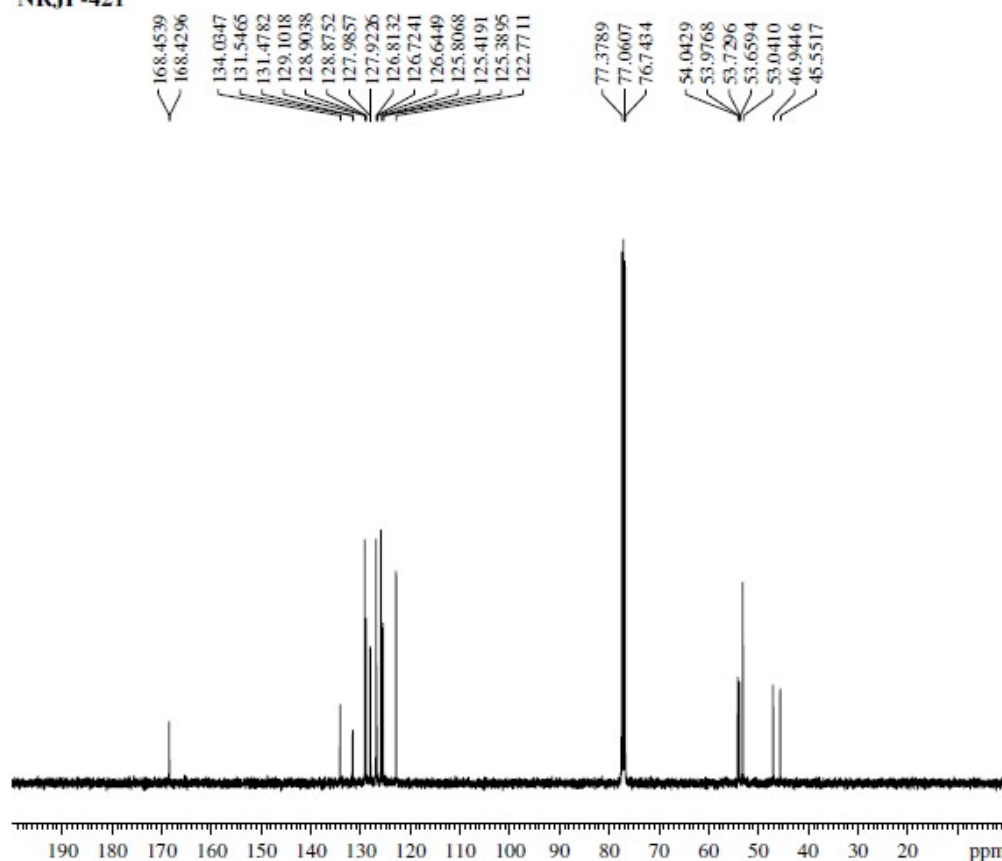
F2 - Acquisition Parameters
 Date_ 20230516
 Time 23.39
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 80.54
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605092 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S96: ^1H NMR spectrum of 4f

NRJP-421



Current Data Parameters
 NAME 19-May-AN-2023
 EXPNO 390
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230520
 Time 13.18
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 0
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TDO 1

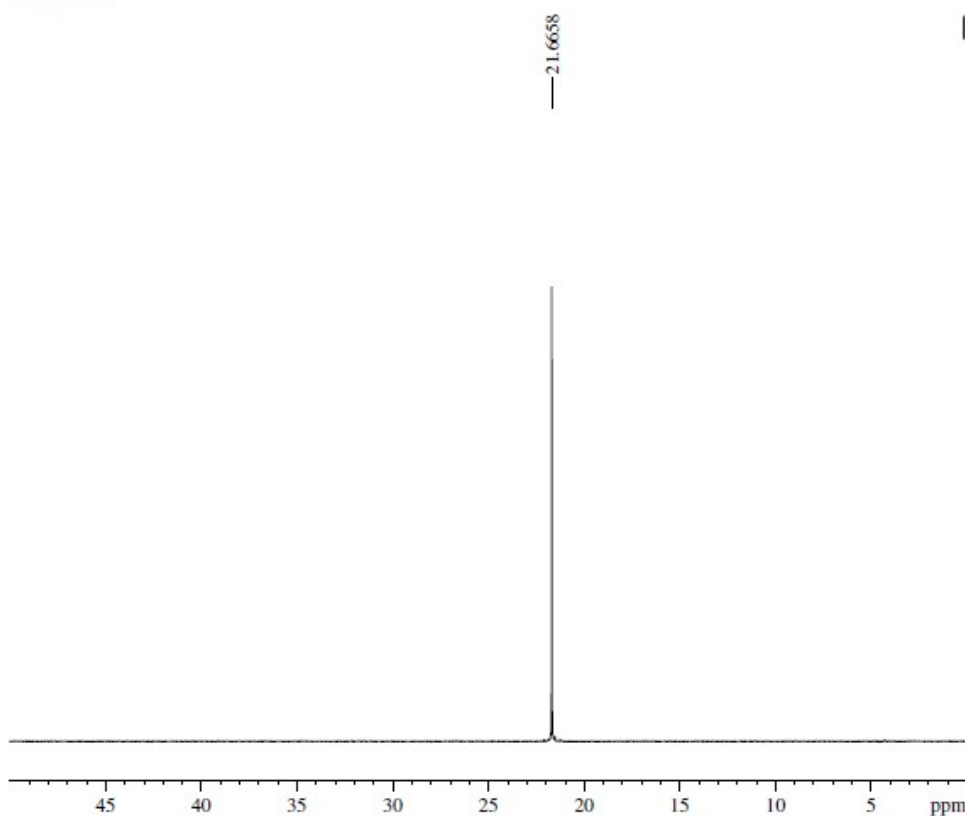
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S97: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 4f

NRJP 421



Current Data Parameters
 NAME 23-May-FN-2023
 EXPNO 570
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230523
 Time 13.31
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

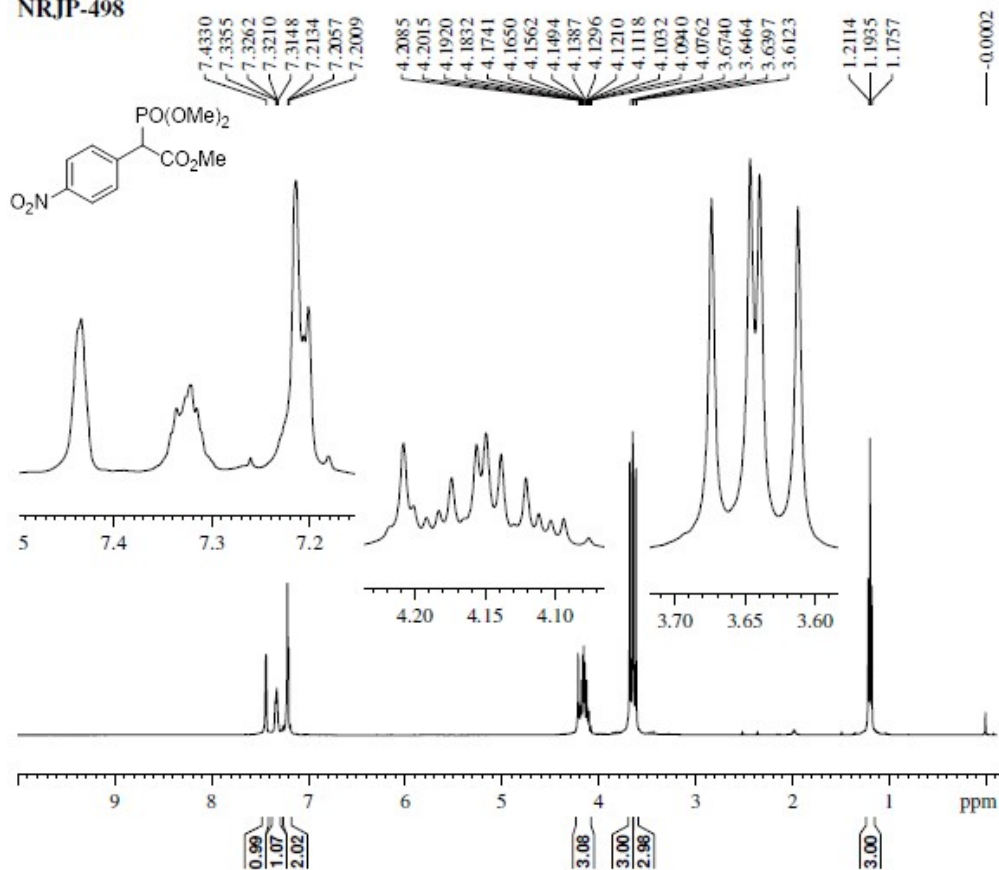
==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S98: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 4f

NRJP-498



Current Data Parameters
 NAME 25-OCT-AN-2023
 EXPNO 420
 PROCNO 1

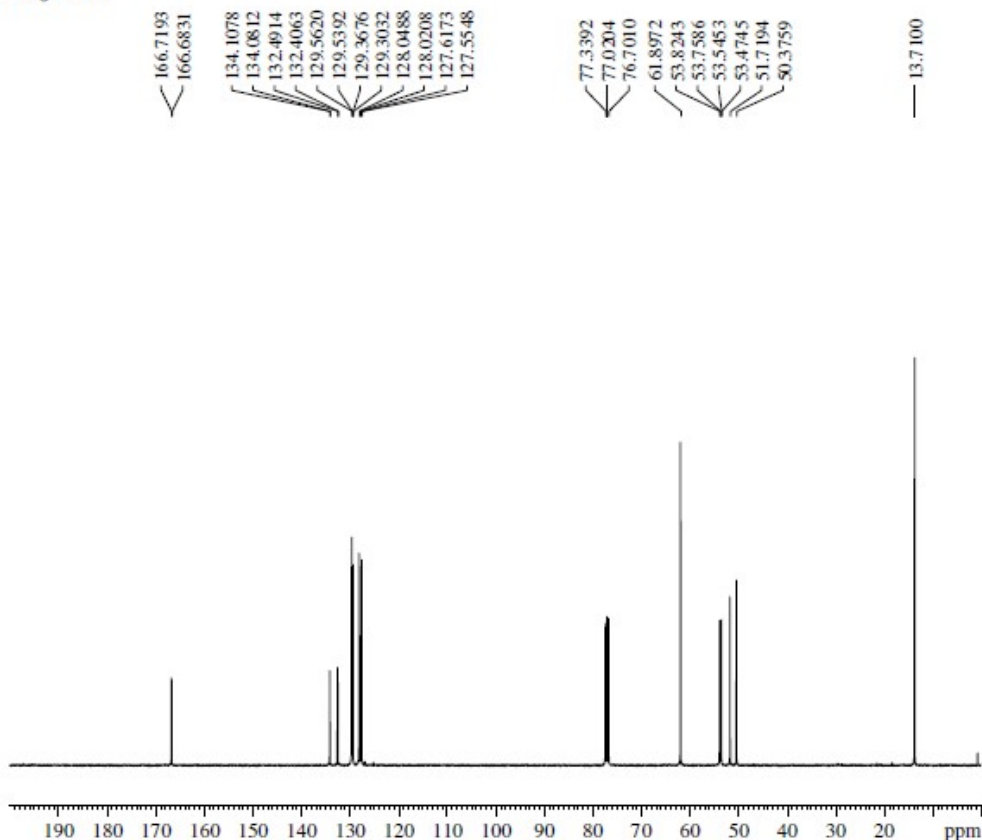
F2 - Acquisition Parameters
 Date_ 20231025
 Time 23.09
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 19.45
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.0000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605090 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S99: ¹H NMR spectrum of 4g

NRJP-498



Current Data Parameters
 NAME 27-OCT-AN-2023
 EXPNO 340
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20231028
 Time 1.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TDO 1

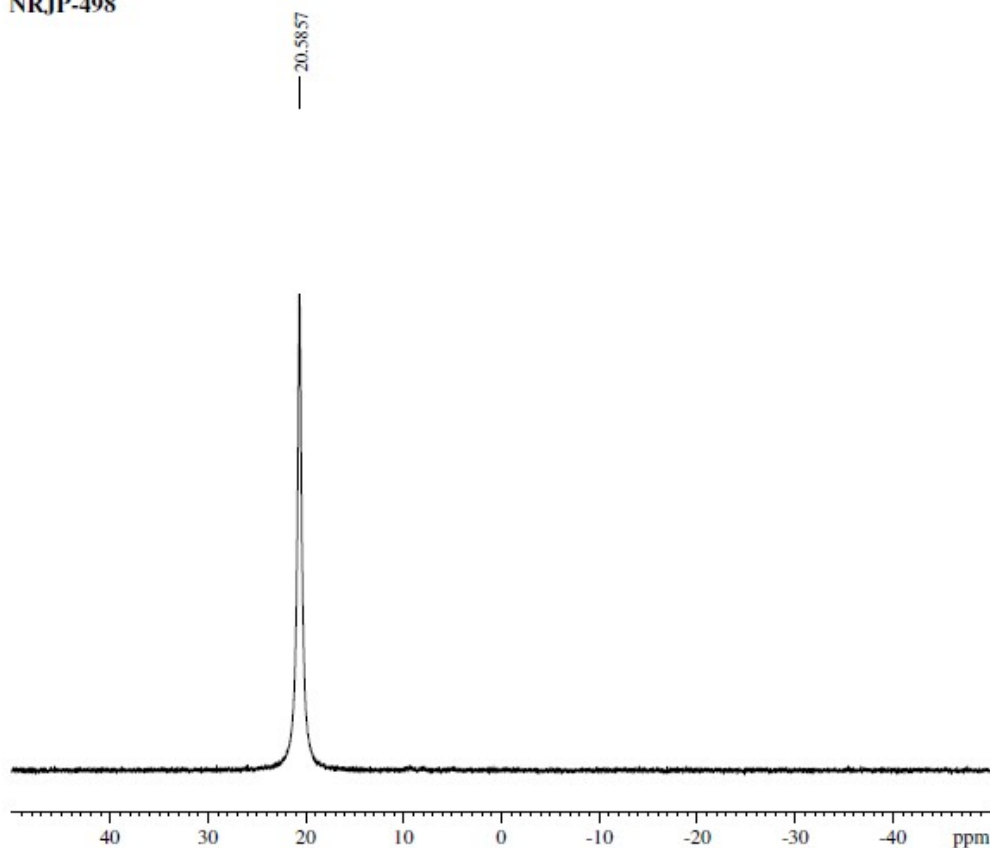
==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204579 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S100: ¹³C{¹H} NMR spectrum of 4g

NRJP-498



Current Data Parameters
 NAME 26-OCT-AN-2023
 EXPNO 370
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20231026
 Time 16.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

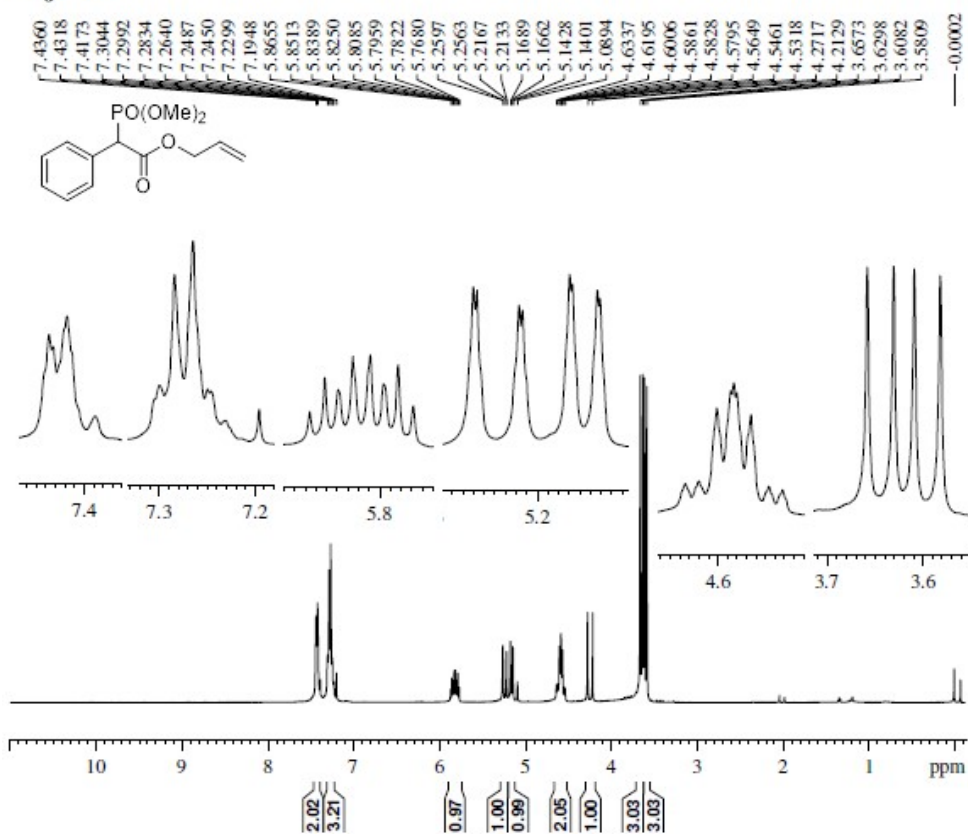
==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S101: ³¹P{H} NMR spectrum of 4g

NRJP-489



Current Data Parameters
 NAME 25-OCT-AN-2023
 EXPNO 410
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20231025
 Time 23.05
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 80.54
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.0000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605356 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S102: ¹H NMR spectrum of 4h

NRJP-489

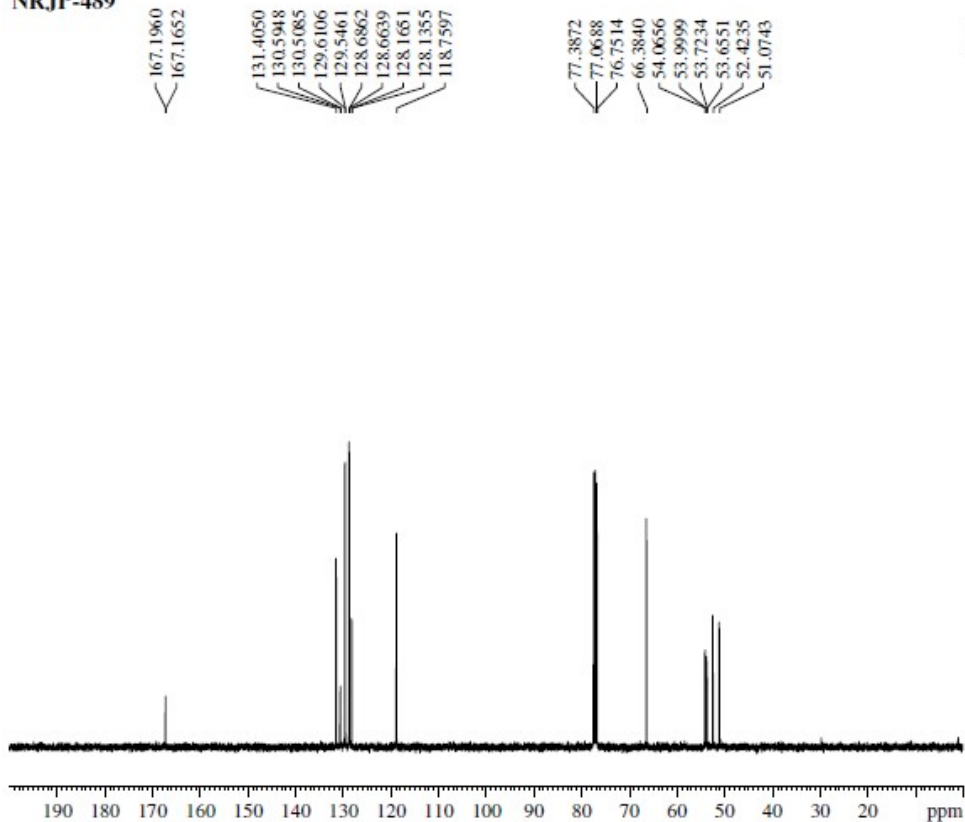


Figure S103: $^{13}\text{C}\{\text{H}\}$ NMR spectrum of 4h



Current Data Parameters
 NAME 21-DEC-AN-2023
 EXPNO 380
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20231222
 Time 8.56
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 D11 0.0300000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP-489

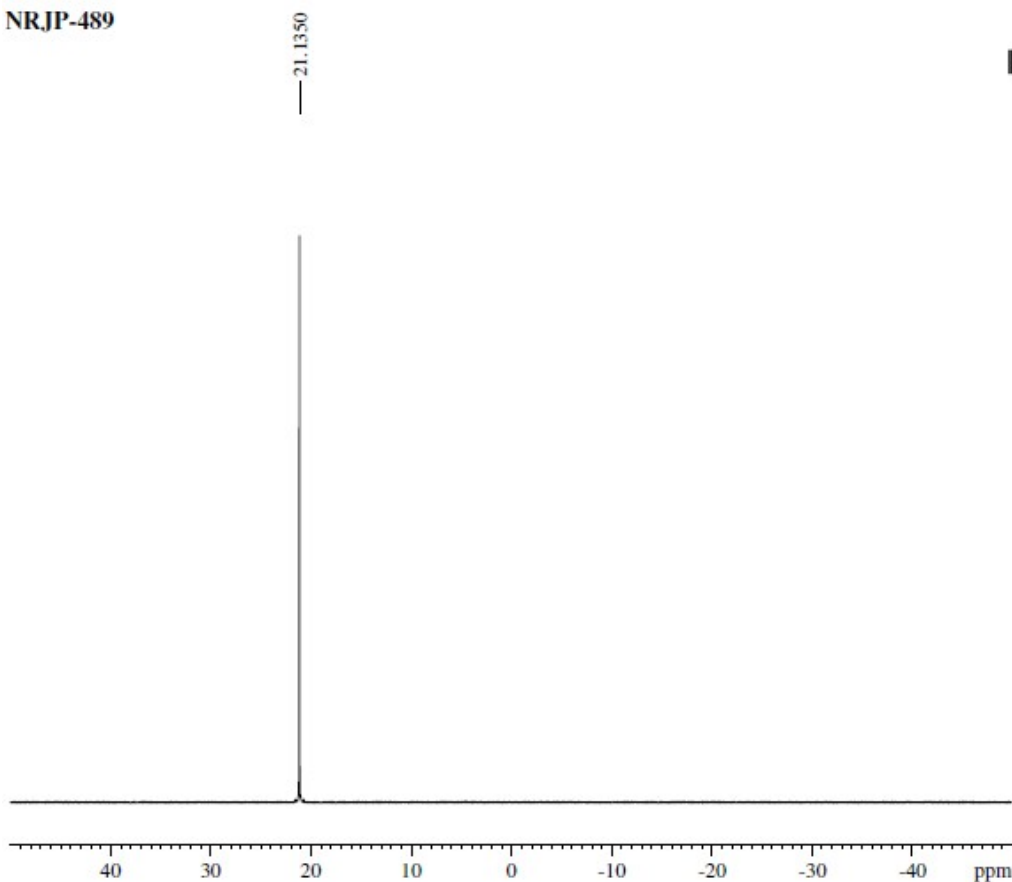


Figure S104: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 4h



Current Data Parameters
 NAME 26-OCT-AN-2023
 EXPNO 350
 PROCNO 1

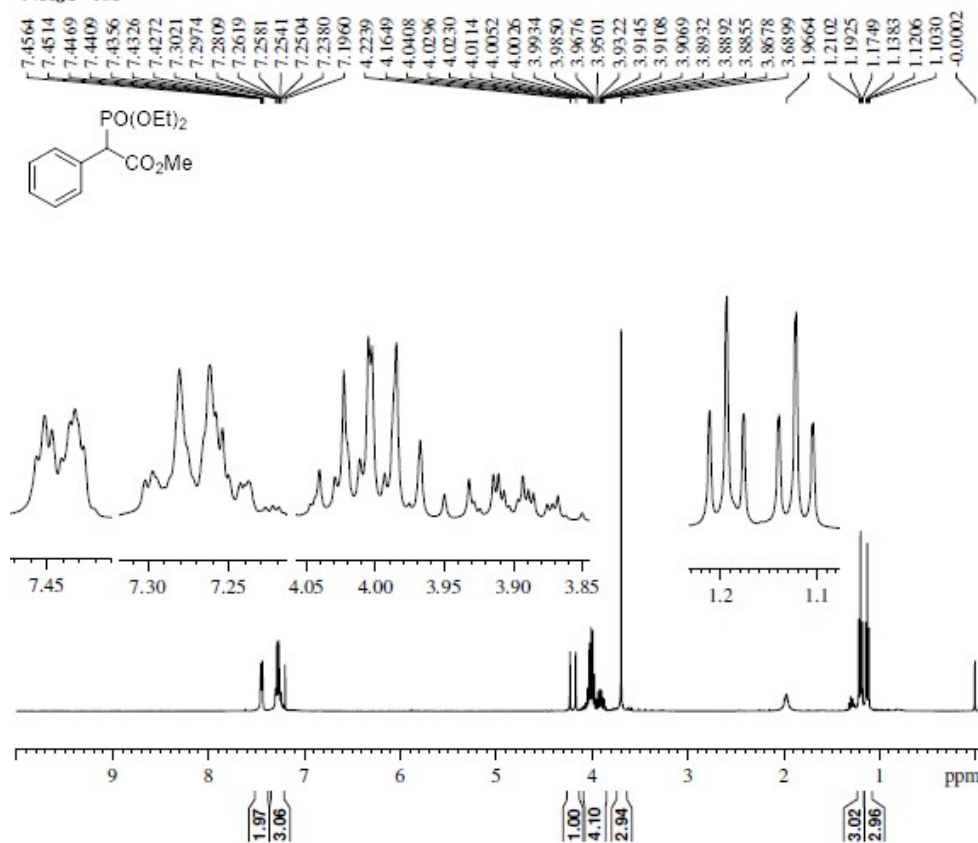
F2 - Acquisition Parameters
 Date_ 20231026
 Time 16.49
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.1 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.0000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.0000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NRJP 485



Current Data Parameters
 NAME 25-OCT-FN-2023
 EXPNO 540
 PROCNO 1

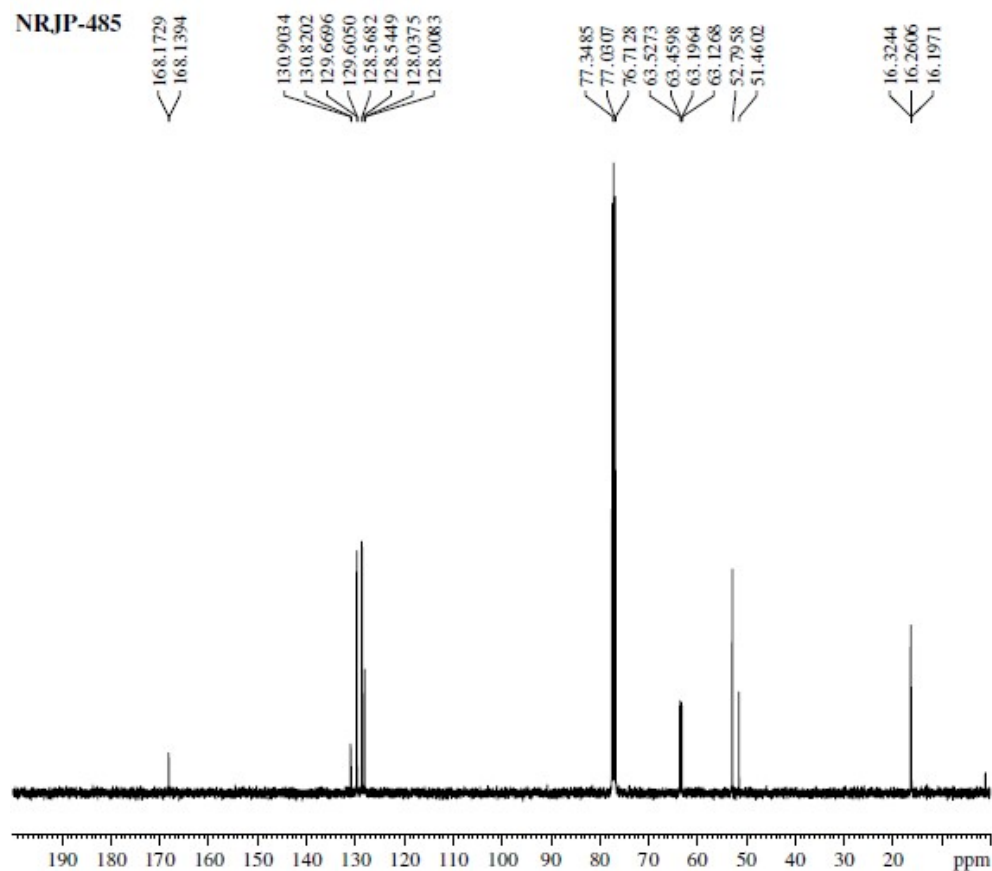
F2 - Acquisition Parameters
 Date_ 20231025
 Time 13.44
 INSTRUM spect
 PROBHD 5 mm PABBO BB
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 9615.385 Hz
 FIDRES 0.146719 Hz
 AQ 3.4078720 sec
 RG 100.41
 DW 52.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 400.1629712 MHz
 NUC1 1H
 P1 13.20 usec
 PLW1 13.00000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1605352 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S105: ¹H NMR spectrum of 4i

NRJP-485



Current Data Parameters
 NAME 26-OCT-AN-2023
 EXPNO 330
 PROCNO 1

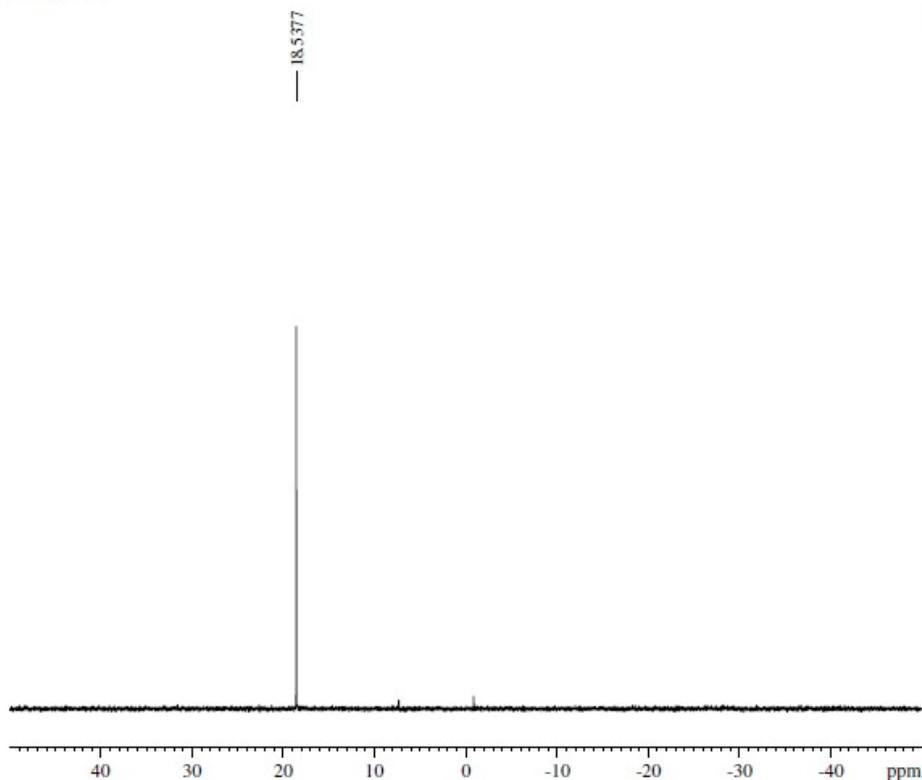
F2 - Acquisition Parameters
 Date_ 20231026
 Time 16.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 201.48
 DW 20.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 ====
 SFO1 100.6304993 MHz
 NUC1 13C
 P1 9.90 usec
 PLW1 53.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 100.6204380 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S106: ¹³C{H} NMR spectrum of 4i



Current Data Parameters
 NAME 13-OCT-AN-2023
 EXPNO 330
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20231016
 Time 9:39
 INSTRUM spect
 PROBHD 5 mm F4BBO BIV
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 64102.563 Hz
 FIDRES 0.978127 Hz
 AQ 0.5111808 sec
 RG 201.48
 DW 7.800 usec
 DE 6.50 usec
 TE 300.3 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

==== CHANNEL f1 ====
 SFO1 161.9798402 MHz
 NUC1 31P
 P1 15.00 usec
 PLW1 12.00000000 W

==== CHANNEL f2 ====
 SFO2 400.1621006 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.00000000 W
 PLW12 0.27963999 W
 PLW13 0.22651000 W

F2 - Processing parameters
 SI 32768
 SF 161.9879400 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

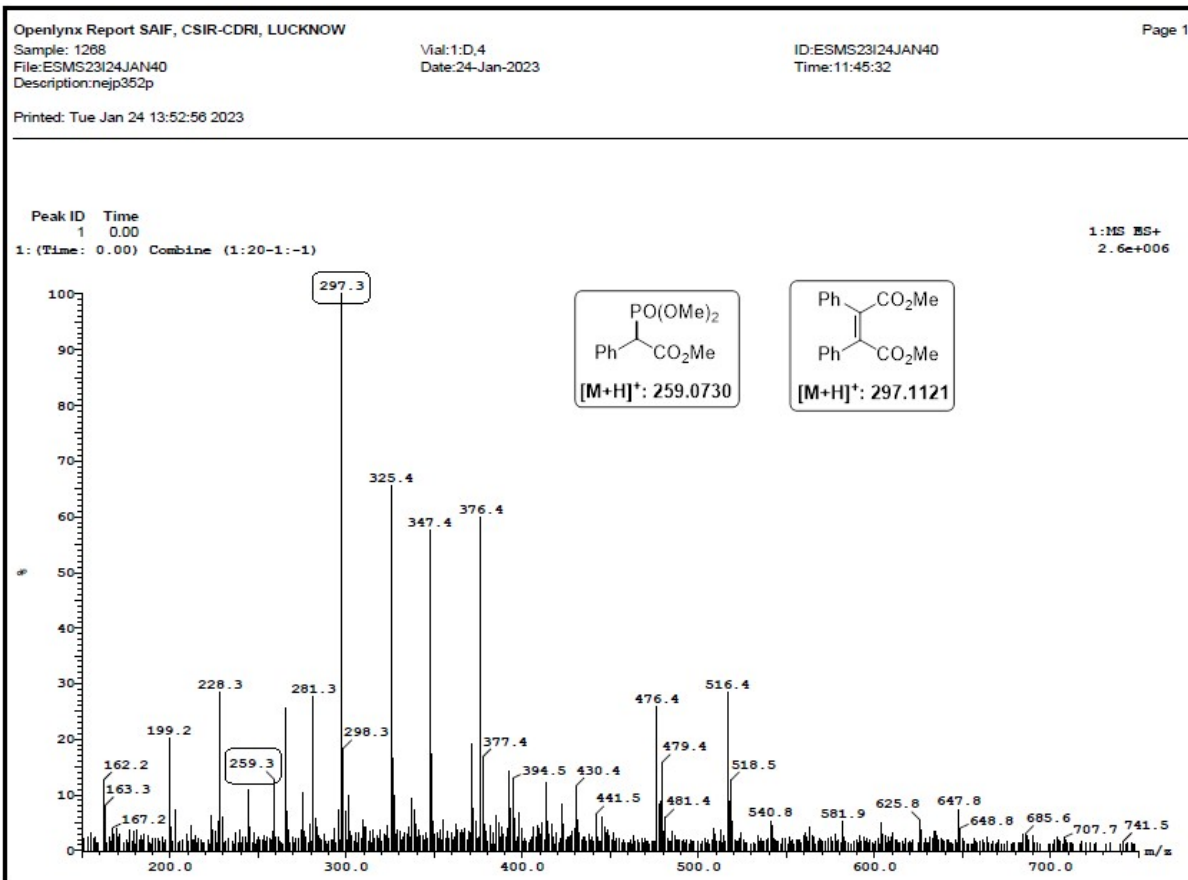
Figure S107: $^{31}\text{P}\{\text{H}\}$ NMR spectrum of 4i

Figure S108: ESMS of crude 4a