Supplementary Information (SI) for New Journal of Chemistry.

Synthesis of aryl esters using carboxylic acids, triarylphosphites, and *N*-iodosuccinimide

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Supporting Information

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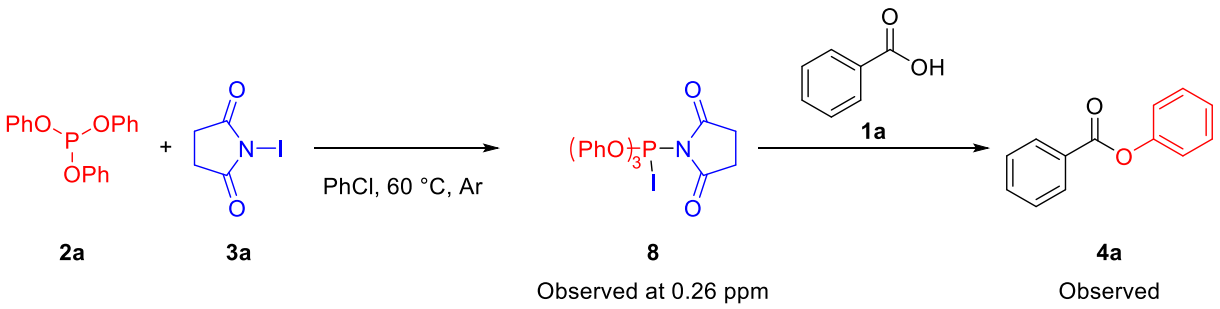
General Information

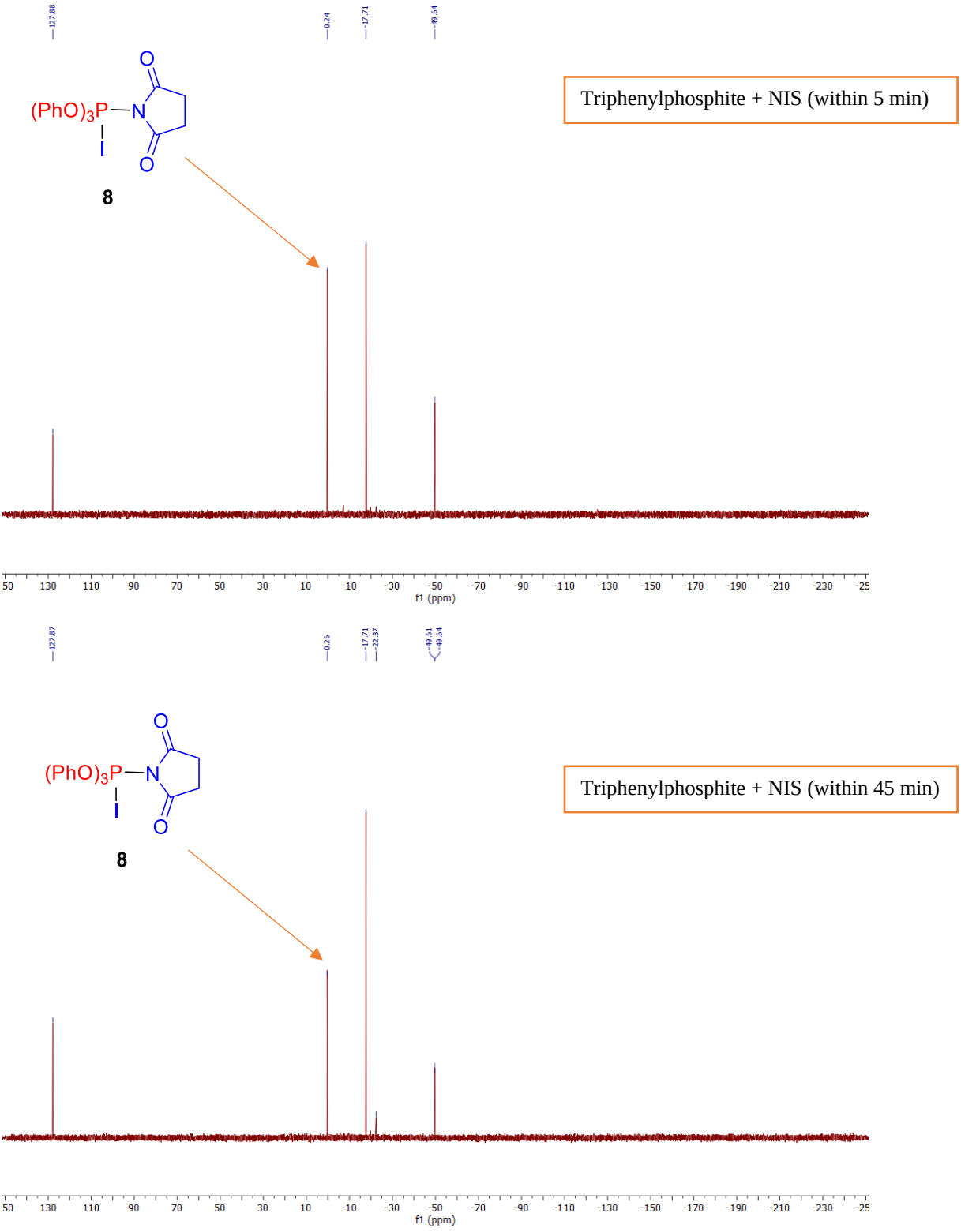
Unless otherwise specified, all reactions were monitored by TLC on silica gel aluminum plates (200 μm) and visualized by UV. Column chromatography was performed on silica gel (40-63 μm). NMR spectra were recorded on a 500 MHz (126 MHz for ^{13}C NMR, 202 MHz for ^{31}P NMR) or a 400 MHz (101 for ^{13}C NMR) or a 300 MHz spectrometer (75 MHz for ^{13}C NMR). ^{31}P NMR spectra were recorded using 85% H_3PO_4 as an external standard. Unless specified below, all chemicals are commercial products and were used as received.

Additional ^{31}P NMR Studies

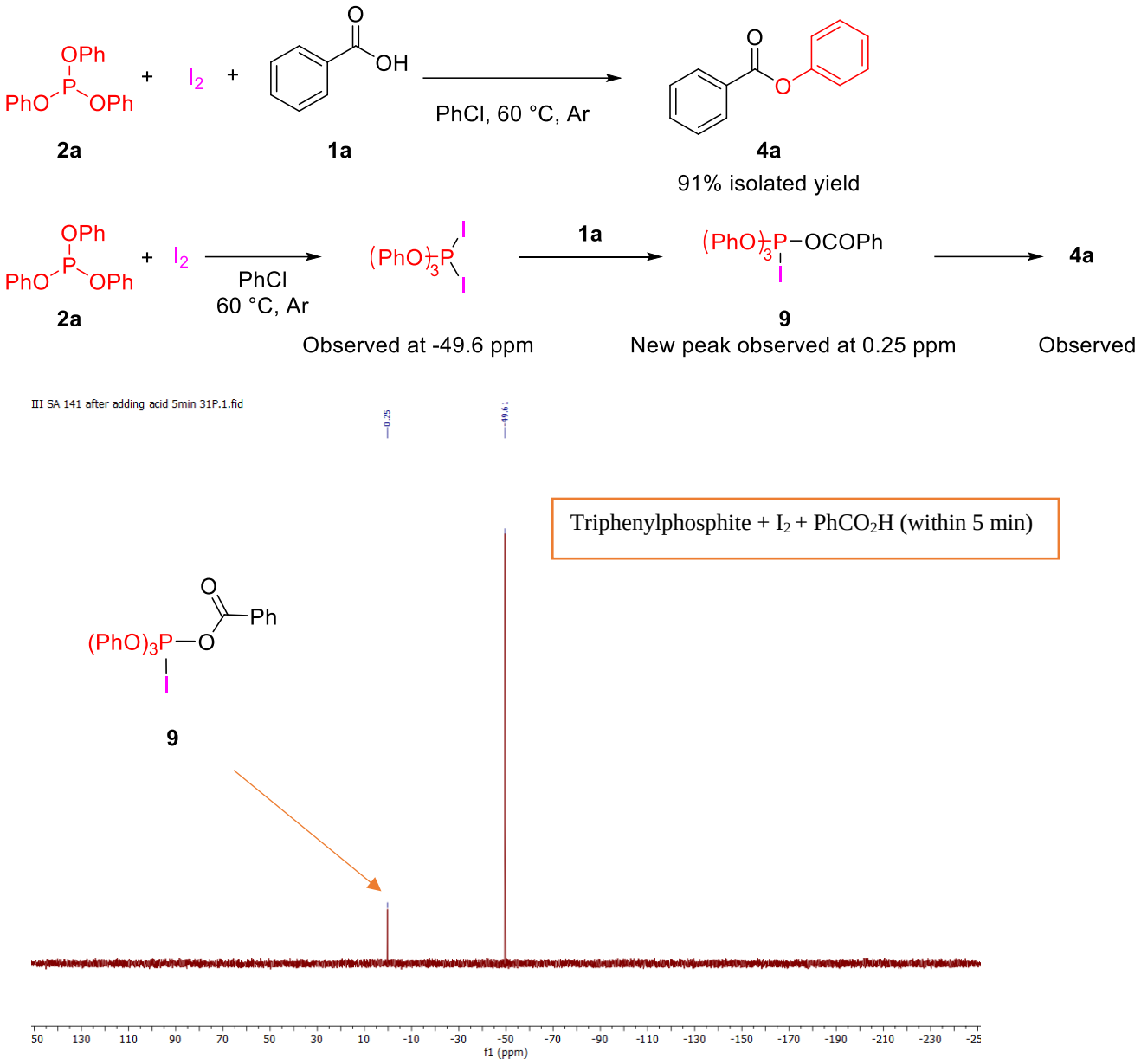
To verify that intermediate **8** indeed leads to the formation of the expected product **4a**, we generated the intermediate **8** by reacting triphenylphosphite (**2a**) and NIS (**3a**) first (Scheme S-1) and monitoring the intermediate formation with ^{31}P NMR. As the ^{31}P NMR spectra in Scheme S-2 show, formation of intermediate **8** (peak at 0.26 PPM) was evident immediately after triphenylphosphite (**2a**) and NIS (**3a**) was mixed and it persisted in the solution (see ^{31}P NMR at 45 min). After benzoic acid (**1a**) was added to this solution, the formation of the expected phenyl benzoate (**4a**) was observed.



Scheme S-1. Step-wise esterification of benzoic acid (**1a**) with ^{31}P NMR monitoring of the reaction intermediates.



Scheme S-2. ^{31}P NMR spectra of the reaction mixture of triphenylphosphite (**2a**) and NIS (**3a**).



Scheme S-3. Using I_2 instead of NIS in the esterification reaction and the observation of the presumed intermediate **9** using ^{31}P NMR.

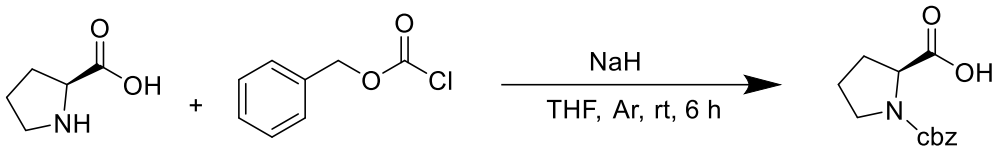
To clarify the role of the proposed intermediate **9** in this reaction, we conducted a similar step-wise esterification reaction using iodine (I_2) instead of NIS and monitored the reaction using ^{31}P NMR (Scheme S-3). The reaction of triphenylphosphite (**2a**) and I_2 should yield the triphenylphosphite diiodide intermediate.[15] Indeed, we found that a peak at -49.6 ppm showed up immediately, which was assigned for this intermediate. After adding benzoic acid (**1a**) to this solution, the formation of a new peak at 0.25 ppm was observed (Scheme S-3). This new peak was

tentatively assigned as the intermediate **9**. After stirring the reaction mixture at 60 °C for 6 h, formation of product **4a** was achieved. Thus, our new ^{31}P study indicates that presumed intermediates **8** and **9** have similar chemical shifts.

The above two experiments provided some evidence that both intermediates **8** and **9** can lead to the formation of the expected **4a**.

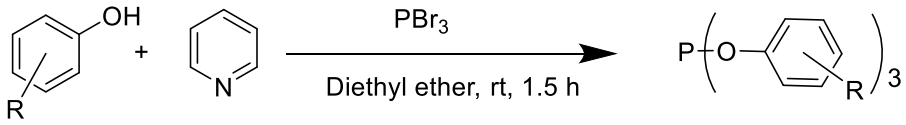
Experimental Procedures

Procedure for the synthesis of 1q



Compound **1b** was synthesized according to the reported procedure:[16] A mixture of L-proline (230 mg, 2.0 mmol) and NaH (72 mg, 3.0 mmol) in dry THF (10 mL) was stirred at room temperature under Ar for 3 h. Then benzyl chloroformate (513 mg, 0.43 mL, 3.0 mmol) was added dropwise over 5 min., and the resulting mixture was stirred at room temperature for 3 h. After the completion of reaction, water (10.0 mL) was added and the mixture was extracted with ethyl acetate (3 x 10.0 mL). The organic layer was separated and combined, washed with brine (20.0 mL), and dried over $MgSO_4$. The solvent was evaporated under reduced pressure. The crude mixture was purified by flash column chromatography using 30% ethyl acetate in hexane as the eluent to give compound **1q** (351 mg, 70%).

General procedure for the synthesis of phosphites 2c-2i



2c: R = 4-Me; **2d**: R = 4-*t*-Bu; **2e**: R = 4-MeO; **2f**: R = 4-I; **2g**: R = 4-NO_2;
2h: R = 2,6-Me_2; **2i**: R = 4-Ph

Phosphites **2c-2i** were prepared according to the reported procedure:[17] The respective phenol (15.76 mmol) was mixed with pyridine (1.58 g, 20.0 mmol) and Et_2O (20.0 mL) under argon. PBr_3 (0.38 mL, 1.08 g, 4.0 mmol) was added dropwise at room temperature over 15 min. The reaction

mixture was further stirred for 1.5 h. After the completion of reaction, the reaction mixture was quenched by adding water (10.0 mL) and extracted with ethyl acetate (3 x 20.0 mL). The combined organic layers were washed with water (30.0 mL) and brine (30.0 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The crude product obtained was purified by silica gel chromatography using 8 to10% ethyl acetate in hexane as the eluent**.**

In the case of **2g**, after the completion of reaction, the reaction mixture was quenched with water (10.0 mL). The white precipitate obtained after filtration was carefully washed with diethyl ether (5.0 mL × 3) and dried under vacuum, which gave the desired product that is pure enough for the esterification reaction. The compound is sparingly soluble in $CDCl_3$.

General experimental procedure for the esterification reaction

An oven-dried culture tube was backfilled with argon three times. To this tube, chlorobenzene (0.5 mL) and benzoic acid (**1a**, 122 mg, 1.0 mmol) were added while stirring. To the resulted solution, $P(OPh)_3$ (**2a**, 465 mg, 1.5 mmol) and NIS (**3a**, 225 mg, 1.0 mmol) were added in order immediately. The culture tube was then further stirred at 60 °C for 6 h (monitored by TLC). After the completion of the reaction, the organic volatiles were evaporated under reduced pressure. The crude reaction mixture obtained was purified by flash column chromatography using 3% EtOAc in hexane as the eluent to give product **4a** as a white solid (196 mg, 99%).

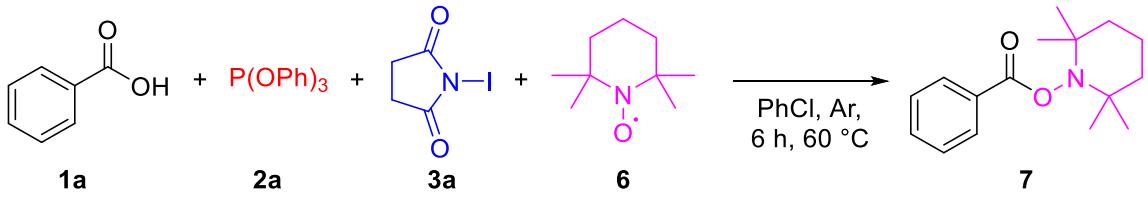
Procedure for the synthesis of products 4j, 4k, 4n, 4q, and 4ad

An oven-dried culture tube was backfilled with argon three times. To this tube, chlorobenzene (0.5 mL) and the respective acid (1.0 mmol) were added while stirring. Then the tube was placed in ice-water bath for 5 minutes. Afterwards DBU (2.0 mmol), $P(OPh)_3$ (1.5 mmol), and NIS (1.0 mmol) were added sequentially and the resulting mixture was further stirred at the same temperature for 10 min and then at room temperature for 16 h (monitored by TLC). The organic volatiles were evaporated under reduced pressure after the completion of the reaction. The crude reaction mixture obtained was purified by flash column chromatography using 3 to 7% EtOAc in hexane as the eluent to give the desired product.

Gram scale synthesis

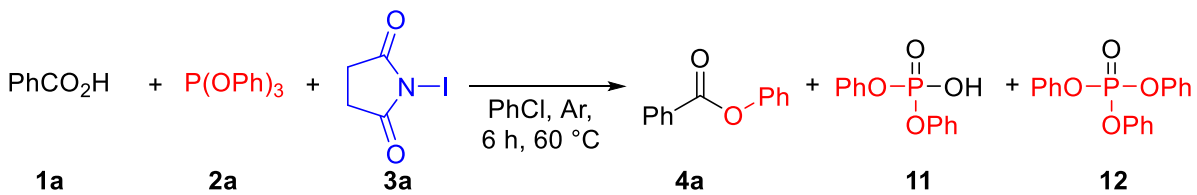
An oven-dried two neck round bottom flask was backfilled with argon three times. To this flask, chlorobenzene (4.0 mL) and benzoic acid (**1a**, 0.976 g, 8.0 mmol) were added while stirring. To the resulted solution, $P(OPh)_3$ (**2a**, 3.72 g, 12.0 mmol) and NIS (**3a**, 1.8 g, 8.0 mmol) were added in order immediately. The flask was then further stirred at 60 °C for 6 h (monitored by TLC). After the completion of the reaction, the organic volatiles were evaporated under reduced pressure. The crude reaction mixture obtained was purified by flash column chromatography using 3% EtOAc in hexane as the eluent to give product **4a** as a white solid (1.484 g, 94%).

TEMPO trapping experiment



An oven-dried culture tube was backfilled with argon three times. To this tube, chlorobenzene (0.5 mL) and benzoic acid (**1a**, 122 mg, 1.0 mmol) were added while stirring. To the resulted solution, $P(OPh)_3$ (**2a**, 465 mg, 1.5 mmol), TEMPO (**5**, 312 mg, 2.0 mmol) and NIS (**3a**, 225 mg, 1.0 mmol) were added in order immediately. The culture tube was then further stirred at 60 °C for 6 h (monitored by TLC). After the completion of the reaction, the organic volatiles were evaporated under reduced pressure. The crude reaction mixture obtained was purified by flash column chromatography using 3% EtOAc in hexane as the eluent to give product **7** as a light orange solid (118 mg, 45%).

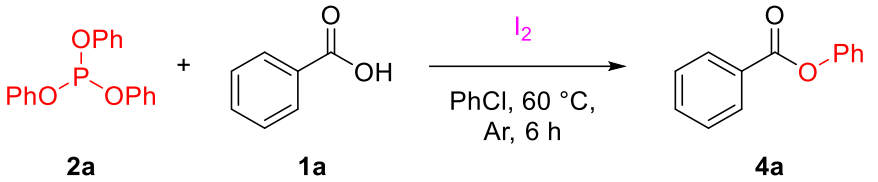
Isolation of triphenyl phosphate (12) and diphenyl phosphate (11)



The reaction was carried out according to the general experimental procedure for the esterification reaction as described above. After the separation of the ester product **4a** via column

chromatography using 3% EtOAc in hexane as the eluent, the column was further eluted with 10% and 50% EtOAc in hexane as the eluent sequentially to give product **12** [145 mg, 89% based on the excess of $Ph(OPh)_3$ (0.5 mmol)], and **11** [132 mg, 53% based on the reacted $Ph(OPh)_3$ (1.0 mmol)], respectively.

Procedure for the esterification reaction using I_2



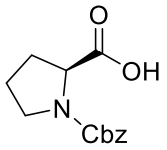
2a **1a** **4a**

91% isolated yield

An oven-dried 2-dram vial was backfilled three-times with argon. To this vial, chlorobenzene (0.5 mL) and benzoic acid (**1a**, 122 mg, 1.0 mmol) were added while stirring. To the resulted solution, $P(OPh)_3$ (**2a**, 465 mg, 1.5 mmol) and I_2 (254 mg, 1.0 mmol) were added in order immediately. The vial was capped and sealed with parafilm, placed immediately in an oil bath that was preheated at 60 °C and stirred for 6 h (monitored by TLC). After the completion of the reaction, the organic volatiles were evaporated under reduced pressure. The crude reaction mixture obtained was purified by flash column chromatography using 3% EtOAc in hexane as the eluent to give product **4a** as a white solid (181 mg, 91%).

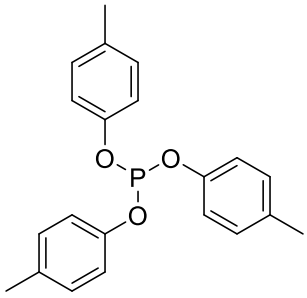
Compound Characterization Data

[(Benzyloxy)carbonyl]-L-proline (1q)[18]



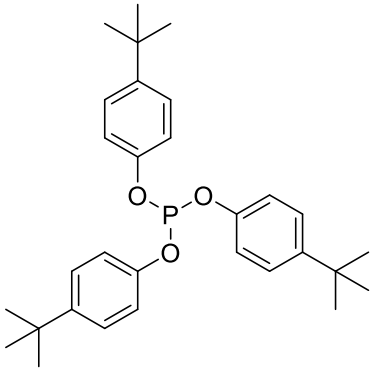
Viscous liquid; 351 mg, 70 % yield. The title compound was purified by using 30% ethyl acetate in hexane as an eluent. R_f = 0.25. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.61 (s, 1H), 7.47 – 7.13 (m, 5H), 5.28 – 5.07 (m, 2H), 4.52 – 4.31 (m, 1H), 3.69 – 3.40 (m, 2H), 2.34 – 1.81 (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.8 (178.3), 155.8 (154.6), 136.4 (136.6), 128.6 (128.5), 128.0 (128.2), 127.8 (128.0), 67.6 (67.3), 59.4 (58.7), 46.7 (47.0), 29.5 (31.0), 24.4 (23.5). Note: Numbers in parentheses are for the other rotamer.

Tris(4-methylphenyl) phosphite (2c)[19]



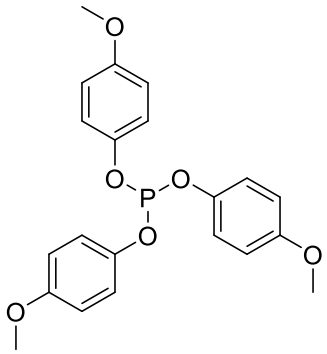
Colorless liquid; 1.2 g, 83 % yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.5. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.12 (d, *J* = 8.5 Hz, 6H), 7.03 (d, *J* = 8.3 Hz, 6H), 2.32 (s, 9H).^{13}C NMR (75 MHz, $CDCl_3$) δ 149.5 (d, $J_{C\text{-}P}$ = 3.8 Hz), 133.8, 130.3, 120.6 (d, $J_{C\text{-}P}$ = 6.8 Hz), 20.9.

Tris[(4-*tert*-butyl)phenyl] phosphite (2d)[19]



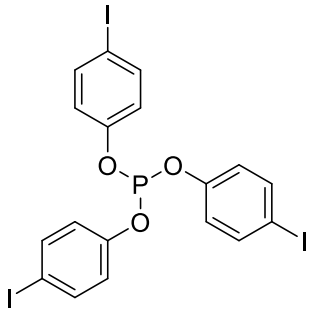
White solid; 1.7 g, 89 % yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.8. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 – 7.30 (m, 6H), 7.09 (dd, *J* = 8.8, 1.1 Hz, 6H), 1.31 (s, 27H).^{13}C NMR (75 MHz, $CDCl_3$) δ 149.4 (d, $J_{C\text{-}P}$ = 3.0 Hz), 147.0, 126.6, 120.2(d, $J_{C\text{-}P}$ = 6.8 Hz), 34.5, 31.6.

Tris(4-methoxyphenyl) phosphite (2e)[17]



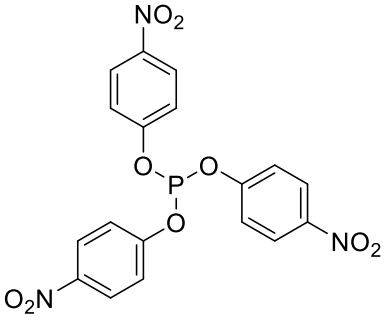
Colorless liquid; 1.2 g, 74 % yield. The title compound was purified by using 8% ethyl acetate in hexane as an eluent. R_f = 0.45. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.13 – 6.99 (m, 6H), 6.88 – 6.76 (m, 6H), 3.78 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.23, 145.1 (d, $J_{C\text{-}P}$ = 3.9 Hz), 121.7 (d, $J_{C\text{-}P}$ = 6.2 Hz) 114.73, 55.58.

Tris(4-iodophenyl) phosphite (2f)[17]



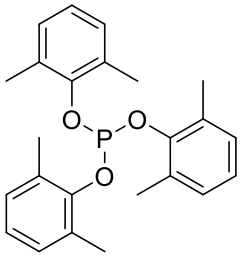
White solid; 2.1 g, 78% yield. The title compound was purified by using 10% ethyl acetate in hexane as an eluent. R_f = 0.55. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.66 – 7.58 (m, 6H), 6.90 – 6.81 (m, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.3 (d, $J_{C\text{-}P}$ = 3.2 Hz), 138.9, 122.9 (d, $J_{C\text{-}P}$ = 6.8 Hz), 88.1.

Tris(4-nitrophenyl) phosphite (2g)[20]



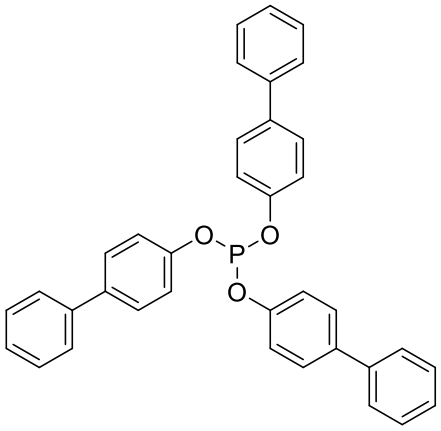
White solid; 1.3 g, 73% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.33 – 8.23 (m, 6H), 7.28 (t, *J* = 1.7 Hz, 3H), 7.25 (d, *J* = 1.1 Hz, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 155.9, 144.9, 126.2, 120.9 (d, $J_{C\text{-}P}$ = 7.5 Hz).

Tris(2,6-dimethylphenyl) phosphite (2h)[21]



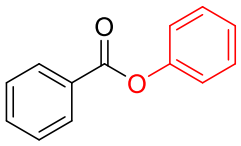
White solid; 1.3 g, 83% yield. The title compound was purified by using 8% ethyl acetate in hexane as an eluent. R_f = 0.5. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.08 (d, *J* = 6.5 Hz, 6H), 7.05 – 6.97 (m, 3H), 2.35 (s, 18H).^{13}C NMR (75 MHz, $CDCl_3$) δ 148.9, 130.7 (d, $J_{C\text{-}P}$ = 3.0 Hz), 129.0, 124.4, 17.8 (d, $J_{C\text{-}P}$ = 6.0 Hz).

Tri([1,1'-biphenyl]-4-yl) phosphite (2i)[19]



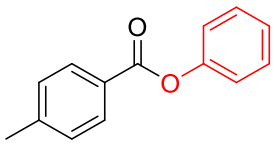
White solid; 1.8 g, 84% yield. The title compound was purified by using 5% ethyl acetate in hexane as an eluent. R_f = 0.4. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 – 7.50 (m, 12H), 7.49 – 7.39 (m, 6H), 7.38 – 7.30 (m, 3H), 7.30 – 7.27 (m, 3H), 7.25 (d, *J* = 1.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.2, 140.5, 137.6, 128.9, 128.6, 127.3, 127.1, 121.1 (d, $J_{C\text{-}P}$ = 6.8 Hz).

Phenyl benzoate (4a)[22]



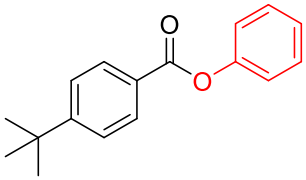
White solid; 196 mg, 99% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.4.^{1}H NMR (300 MHz, $CDCl_3$) δ 8.32 – 8.20 (m, 2H), 7.70 – 7.59 (m, 1H), 7.58 – 7.40 (m, 4H), 7.35 – 7.24 (m, 3H).). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.3, 151.1, 133.7, 130.3, 129.7, 129.6, 128.7, 126.0, 121.9.

Phenyl 4-methylbenzoate (4b)[23]



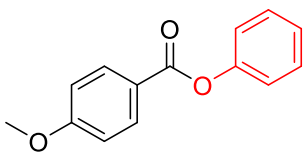
White solid; 180 mg, 85% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.4. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.3 Hz, 2H), 7.41 (ddt, *J* = 8.3, 6.6, 1.2 Hz, 2H), 7.33 – 7.27 (m, 2H), 7.25 – 7.13 (m, 3H), 2.43 (s, 3H).^{13}C NMR (75 MHz, CDCl3) δ 165.2, 151.1, 144.4, 130.2, 129.5, 129.3, 126.9, 125.8, 121.8, 21.7.

Phenyl 4-(*tert*-butyl)benzoate (4c)[23]



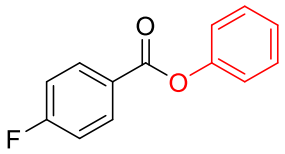
White solid; 210 mg, 83% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.4. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 – 8.17 (m, 2H), 7.60 – 7.52 (m, 2H), 7.50 – 7.40 (m, 2H), 7.28 (ddt, *J* = 8.5, 7.8, 1.2 Hz, 3H), 1.41 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.2, 157.4, 151.2, 130.2, 129.6, 127.0, 125.9, 125.7, 121.9, 35.3, 31.2.

Phenyl 4-methoxybenzoate (4d)[24]



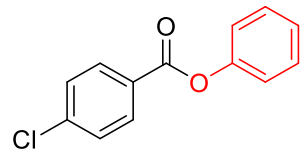
White solid; 207 mg, 91% yield. The title compound was purified by using 4% ethyl acetate in hexane as an eluent. R_f = 0.15. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 – 8.16 (m, 2H), 7.51 – 7.40 (m, 2H), 7.34 – 7.20 (m, 3H), 7.05 – 6.98 (m, 2H), 3.92 (s, 3H).). ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.9, 163.9, 151.1, 132.3, 129.5, 125.7, 121.9, 121.8, 113.9, 55.5.

Phenyl 4-fluorobenzoate (4e)[22]



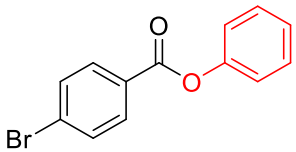
White solid; 188 mg, 87% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.7. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.32 – 8.20 (m, 2H), 7.48 (ddd, *J* = 8.2, 6.8, 1.2 Hz, 2H), 7.36 – 7.14 (m, 5H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.6 (d, J_{C-F} = 256.2 Hz), 164.2, 150.9, 132.8(d, J_{C-F} = 9.3 Hz), 129.6, 126.1, 125.8 (d, J_{C-F} = 3.1 Hz), 121.8, 115.8(d, J_{C-F} = 22.0 Hz).

Phenyl 4-chlorobenzoate (4f)[22]



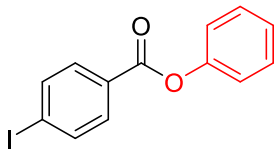
White solid; 194 mg, 84% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.7. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 (dt, *J* = 8.9, 2.2 Hz, 2H), 7.51 – 7.33 (m, 4H), 7.30 – 7.12 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.3, 150.8, 140.1, 131.6, 129.6, 129.0, 128.1, 126.1, 121.7.

Phenyl 4-bromobenzoate (4g)[25]



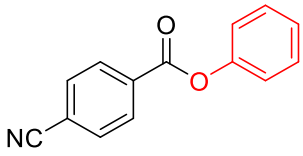
White solid; 166 mg, 60% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.7. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.2 Hz, 2H), 7.69 (d, *J* = 8.2 Hz, 2H), 7.47 (t, *J* = 7.7 Hz, 2H), 7.31 (dd, *J* = 14.7, 7.2 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 2H).^{13}C NMR (126 MHz, $CDCl_3$) δ 164.6, 150.9, 132.1, 131.8, 129.7, 129.0, 128.6, 126.2, 121.7.

Phenyl 4-iodobenzoate (4h)[25]



White solid; 275mg, 85% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.7. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 – 7.87 (m, 4H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.30 (dd, *J* = 13.3, 5.8 Hz, 1H), 7.23 (d, *J* = 8.0 Hz, 2H).^{13}C NMR (126 MHz, $CDCl_3$) δ 164.8, 150.8, 138.0, 131.6, 129.6, 129.1, 126.1, 121.6, 101.6.

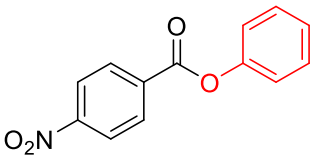
Phenyl 4-cyanobenzoate (4i)[25]



White solid; 140 mg, 63% yield. The title compound was purified by using 5% ethyl acetate in hexane as an eluent. R_f = 0.2. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.37 – 8.29 (m, 2H), 7.88 – 7.80 (m, 2H), 7.48 (dd, *J* =

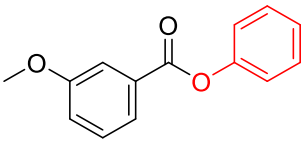
8.5, 7.1 Hz, 2H), 7.38 – 7.29 (m, 1H), 7.27 – 7.20 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.7, 150.6, 133.5, 132.5, 130.7, 129.7, 126.4, 121.5, 117.9, 117.1.

Phenyl 4-nitrobenzoate (4j)[22]



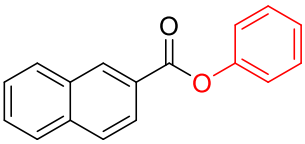
White solid; 70 mg, 29% yield. The title compound was purified by using 4% ethyl acetate in hexane as an eluent. R_f = 0.2. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 – 7.33 (m, 2H), 7.27 – 7.18 (m, 1H), 7.13 – 7.04 (m, 2H), 2.31 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.4, 151.0, 150.6, 135.1, 131.4, 129.8, 126.5, 123.8, 121.5.

Phenyl 3-methoxybenzoate (4k)[24]



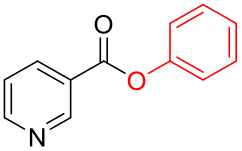
White solid; 205 mg, 90% yield. The title compound was purified by using 4% ethyl acetate in hexane as an eluent. R_f = 0.15. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.7 Hz, 1H), 7.71 (q, *J* = 2.0 Hz, 1H), 7.45 – 7.36 (m, 3H), 7.28 – 7.19 (m, 3H), 7.16 (ddd, *J* = 8.3, 2.6, 1.3 Hz, 1H), 3.84 (s, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 165.1, 159.8, 151.1, 130.9, 129.7, 129.6, 126.0, 122.6, 121.8, 120.2, 114.6, 55.5.

Phenyl 2-naphthoate (4l)[25]



White solid; 230mg, 93% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.6. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.77 (s, 1H), 8.18 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.98 (d, *J* = 8.1 Hz, 1H), 7.91 (dd, *J* = 14.3, 8.4 Hz, 2H), 7.64 – 7.52 (m, 2H), 7.43 (t, *J* = 8.0 Hz, 2H), 7.30 – 7.21 (m, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 165.4, 151.1, 135.9, 132.5, 132.0, 129.6,129.5, 128.7, 128.4, 127.9, 126.9, 126.8, 126.0, 125.5, 121.8.

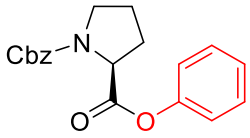
Phenyl nicotinate (4m)[22]



White solid; 125 mg, 63% yield. The title compound was purified by using 5% ethyl acetate in hexane as an eluent. R_f = 0.3. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.46 – 9.37 (m, 1H), 8.87 (dd, *J* = 4.9, 1.8 Hz, 1H), 8.47 (dt, *J* = 8.0, 1.9

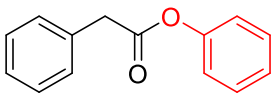
Hz, 1H), 7.52 – 7.40 (m, 3H), 7.36 – 7.20 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.9, 154.0, 151.4, 150.6, 137.7, 129.7, 126.3, 125.7, 123.6, 121.6.

1-Benzyl 2-phenyl (*S*)-pyrrolidine-1,2-dicarboxylate (4p)



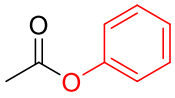
Colorless liquid; 198 mg, 61% yield. The title compound was purified by using 10 % ethyl acetate in hexane as an eluent. R_f = 0.2. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 – 6.99 (m, 9H), 6.76 – 6.67 (m, 1H), 5.24 – 4.94 (m, 2H), 4.50 (ddd, *J* = 15.6, 8.6, 4.1 Hz, 1H), 3.67 – 3.38 (m, 2H), 2.39 – 1.79 (m, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.4 (171.3), 154.4 (155.1), 150.5(150.8), 136.5(136.8), 129.5, 128.6 (128.5), 128.2, (128.1), 128.0 (128.1), 126.1 (126.0), 121.3 (121.5), 67.4 (67.2), 59.1 (59.6), 47.2 (46.6), 31.2 (30.1), 23.6 (24.6). Note: Numbers in parentheses are for the other rotamer. ν_{max} (acetone, cm^{-1}): 1766, 1700, 1410, 1350, 1117. HRMS (ESI, *m/z*) calcd. for $C_{19}H_{20}NO_4$ ($[M+H]^+$): 326.1387; found: 326.1391.

Phenyl 2-phenylacetate (4q)[22]



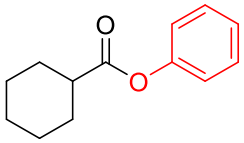
White solid; 188 mg, 89% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.3. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.61 – 7.29 (m, 7H), 7.28 – 7.20 (m, 1H), 7.14 – 7.04 (m, 2H), 3.89 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.0, 150.8, 133.5, 129.4, 129.3, 128.8, 127.4, 125.9, 121.5, 41.5.

Phenyl acetate (4r)[26]



Colorless liquid; 111 mg, 82% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.3. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 – 7.33 (m, 2H), 7.27 – 7.18 (m, 1H), 7.13 – 7.04 (m, 2H), 2.31 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.6, 150.8, 129.6, 126.0, 121.7, 21.3.

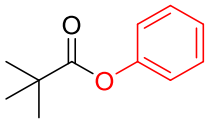
Phenyl cyclohexanecarboxylate (4s)[27]



Colorless liquid; 200 mg, 98% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.3. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 – 7.35 (m, 2H), 7.33 – 7.20 (m, 1H), 7.18 – 7.08 (m, 2H), 2.62 (tt, *J* = 11.1, 3.7 Hz, 1H), 2.20 – 2.07 (m, 2H), 1.98 – 1.82 (m, 2H), 1.80 – 1.60 (m, 3H), 1.53 –

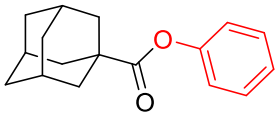
1.28 (m, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 174.3, 150.9, 129.3, 125.5, 121.5, 43.1, 28.9, 25.7, 25.3.

Phenyl pivalate (4t)[27]



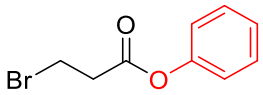
Pale yellow liquid; 158 mg, 89% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.3. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 (ddd, *J* = 8.3, 6.8, 1.3 Hz, 2H), 7.30 – 7.22 (m, 1H), 7.15 – 7.09 (m, 2H), 1.43 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.0, 151.2, 129.4, 125.6, 121.5, 39.1, 27.2.

Phenyl adamantane-1-carboxylate (4u)[28]



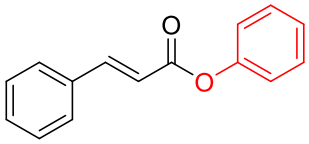
White solid; 205 mg, 80% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.4. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 – 7.37 (m, 2H), 7.30 – 7.22 (m, 1H), 7.15 – 7.08 (m, 2H), 2.14 (d, *J* = 2.6 Hz, 9H), 1.85 (t, *J* = 3.0 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 176.1, 151.2, 129.4, 125.6, 121.6, 41.1, 38.8, 36.5, 28.0.

Phenyl 3-bromopropanoate (4v)[29]



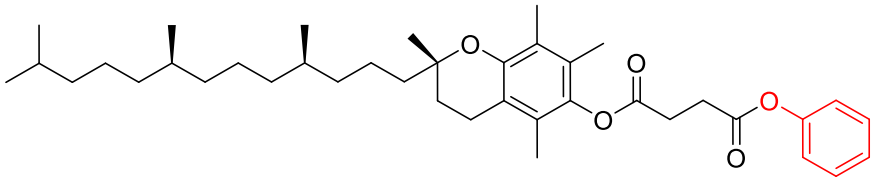
Colorless liquid; 119 mg, 52% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.2. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43 – 7.35 (m, 2H), 7.28 – 7.21 (m, 1H), 7.15 – 7.08 (m, 2H), 3.70 (t, *J* = 6.8 Hz, 2H), 3.18 (t, *J* = 6.7 Hz, 2H).^{13}C NMR (126 MHz, $CDCl_3$) δ 169.1, 150.5, 129.6, 126.1, 121.5, 37.8, 25.8.

Phenyl cinnamate (4w)[23]



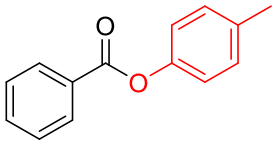
White solid; 203 mg, 91% yield. The title compound was purified by using 3% acetate in hexane as an eluent. R_f = 0.3. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 (d, *J* = 16.0 Hz, 1H), 7.59 (q, *J* = 3.2 Hz, 2H), 7.47 – 7.39 (m, 5H), 7.26 (dd, *J* = 26.9, 7.8 Hz, 3H), 6.67 (dd, *J* = 16.0, 1.6 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.5, 150.9, 146.7, 134.3, 130.8, 129.6, 129.1, 128.4, 125.9, 121.8, 117.5.

Phenyl {(*S*)-2,5,7,8-tetramethyl-2-[(4*R*,8*R*)-4,8,12-trimethyltridecyl]chroman-6-yl} succinate (4x)



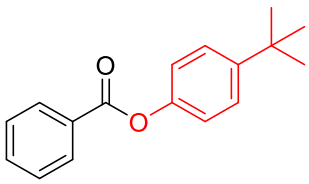
White solid; 466 mg, 77% yield. m.p. 59.3 °C. The title compound was purified by using 3% acetate in hexane as an eluent. R_f = 0.4. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 – 7.34 (m, 2H), 7.26 – 7.20 (m, 1H), 7.13 – 7.07 (m, 2H), 3.04 (td, *J* = 4.8, 1.8 Hz, 4H), 2.60 (t, *J* = 6.8 Hz, 2H), 2.12 – 1.97 (m, 9H), 1.88 – 1.70 (m, 2H), 1.62 – 1.01 (m, 24H), 0.93 – 0.82 (m, 12H).^{13}C NMR (126 MHz, $CDCl_3$) δ 171.0, 150.8, 149.6, 140.6, 129.6, 126.8, 126.0, 125.1, 123.2, 121.7, 117.5, 75.2, 39.5, 37.7, 37.6, 37.4, 32.9, 32.8, 29.5, 28.9, 28.1, 24.9, 24.6, 22.9, 22.8, 21.2, 20.7, 19.9, 19.8, 13.1, 12.3, 12.0. ν_{max} (neat, cm^{-1}): 1760, 1740, 1350,1200, 1120. HRMS (ESI, *m/z*) calcd. for $C_{39}H_{59}O_5$ ($[M+H]^+$): 607.4357; found: 607.4367.

4-Methylphenyl benzoate (4y)[28]



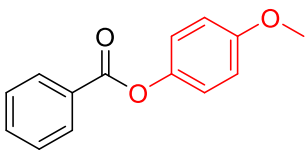
White solid; 175 mg, 83% yield. The title compound was purified by using 5% ethyl acetate in hexane as an eluent. R_f = 0.5.^{1}H NMR (300 MHz, $CDCl_3$) δ 8.29 – 8.22 (m, 2H), 7.71 – 7.63 (m, 1H), 7.60 – 7.51 (m, 2H), 7.28 (dd, *J* = 8.8, 0.8 Hz, 2H), 7.18 – 7.11 (m, 2H), 2.43 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.5, 148.8, 135.6, 133.6, 130.3, 130.1, 129.8, 128.7, 121.5, 21.0.

4-Tertbutylphenyl benzoate (4z)[25]



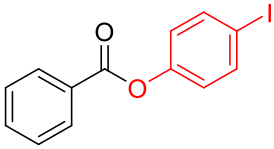
White solid; 193 mg, 76% yield. The title compound was purified by using 5% ethyl acetate in hexane as an eluent. R_f = 0.6. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.27 – 8.20 (m, 2H), 7.68 – 7.61 (m, 1H), 7.57 – 7.42 (m, 4H), 7.21 – 7.14 (m, 2H), 1.37 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.4, 148.8, 148.7, 133.6, 130.3, 129.8, 128.6, 126.5, 121.1, 34.6, 31.6.

4-Methoxyphenyl benzoate (4aa)[22]



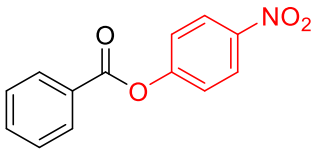
White solid; 209 mg, 92% yield. The title compound was purified by using 5% ethyl acetate in hexane as an eluent. R_f = 0.45. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24 – 8.17 (m, 2H), 7.67 – 7.60 (m, 1H), 7.55 – 7.46 (m, 2H), 7.15 (d, *J* = 9.2 Hz, 2H), 6.95 (d, *J* = 9.1 Hz, 2H), 3.83 (s, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 165.7, 157.4, 144.5, 133.6, 130.2, 129.8, 128.7, 122.6, 114.6, 55.7.

4-Iodophenyl benzoate (4ab)[25]



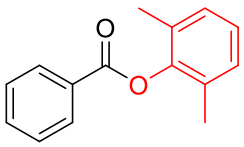
White solid; 155 mg, 48% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.7. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.23 – 8.15 (m, 2H), 7.79 – 7.70 (m, 2H), 7.69 – 7.62 (m, 1H), 7.52 (ddt, *J* = 8.3, 6.7, 1.1 Hz, 2H), 7.04 – 6.96 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.9, 151.0, 138.7, 133.9, 130.3, 129.3, 128.8, 124.1, 90.1.

4-Nitrophenyl benzoate (4ac)[25]



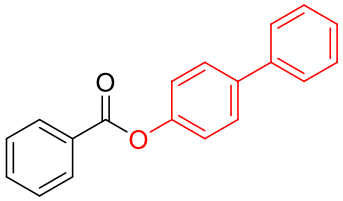
White solid; 186 mg, 76% yield. The title compound was purified by using 5% ethyl acetate in hexane as an eluent. R_f = 0.3. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.36 – 8.30 (m, 2H), 8.24 – 8.14 (m, 2H), 7.72 – 7.65 (m, 1H), 7.58 – 7.50 (m, 2H), 7.45 – 7.40 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.4, 155.9, 145.6, 134.4, 130.5, 128.9, 128.7, 125.4, 122.8.

2,6-Dimethylphenyl benzoate (4ad)[25]



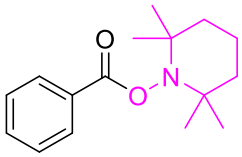
White solid; 95 mg, 42% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. R_f = 0.4. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.44 – 8.37 (m, 2H), 7.85 – 7.77 (m, 1H), 7.72 – 7.65 (m, 2H), 7.30 – 7.22 (m, 3H), 2.35 (d, *J* = 0.7 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.5, 148.5, 133.7, 130.5, 130.3, 129.5, 128.8, 128.7, 126.1, 16.5.

[1,1'-Biphenyl]-4-yl benzoate (4ae)[30]



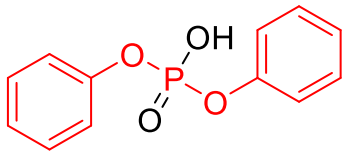
White solid; 241 mg, 88% yield. The title compound was purified by using 5% ethyl acetate in hexane as an eluent. R_f = 0.35. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.30 – 8.22 (m, 2H), 7.72 – 7.59 (m, 5H), 7.58 – 7.43 (m, 4H), 7.42 – 7.28 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.4, 150.5, 140.5, 139.2, 133.8, 130.3, 129.7, 128.9, 128.7, 128.4, 127.5, 127.3, 122.1.

2,2,6,6-Tetramethylpiperidin-1yl benzoate (7)[10]



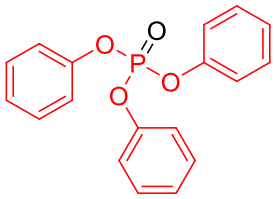
Light orange solid; 118 mg, 45% yield. The title compound was purified by using 3% ethyl acetate in hexane as an eluent. Rf = 0.35. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.19 (d, *J* = 7.4 Hz, 2H), 7.68 (t, *J* = 7.2 Hz, 1H), 7.57 (t, *J* = 7.5 Hz, 2H), 1.93 – 1.53 (m, 6H), 1.39 (s, 6H), 1.23 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.8, 131.3, 128.1, 128.0, 126.9, 58.8, 37.5, 30.4, 19.3, 15.4.

Diphenyl phosphate (11)[31]



White solid; 132 mg, 53% yield. The title compound was purified by using 50% ethyl acetate in hexane as an eluent. Rf = 0.1 (ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.64 (s, 1H), 7.29 (t, *J* = 7.8 Hz, 4H), 7.16 (dd, *J* = 8.3, 5.2 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.6 (d, J_{C-P} = 7.6 Hz), 129.8, 125.4, 120.3 (d, J_{C-P} = 5.0 Hz). ^{31}P NMR (202 MHz, $CDCl_3$) δ -10.4.

Triphenyl phosphate (12)[32]



Colorless liquid; 145 mg, 89% yield. The title compound was purified by using10% ethyl acetate in hexane as an eluent. Rf = 0.2. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 – 7.33 (m, 6H), 7.31 – 7.20 (m, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.6 (d, J_{C-P} = 7.6 Hz), 130.0, 125.7, 120.3 (d, J_{C-P} = 5.0 Hz). ^{31}P NMR (202 MHz, $CDCl_3$) δ -17.7.

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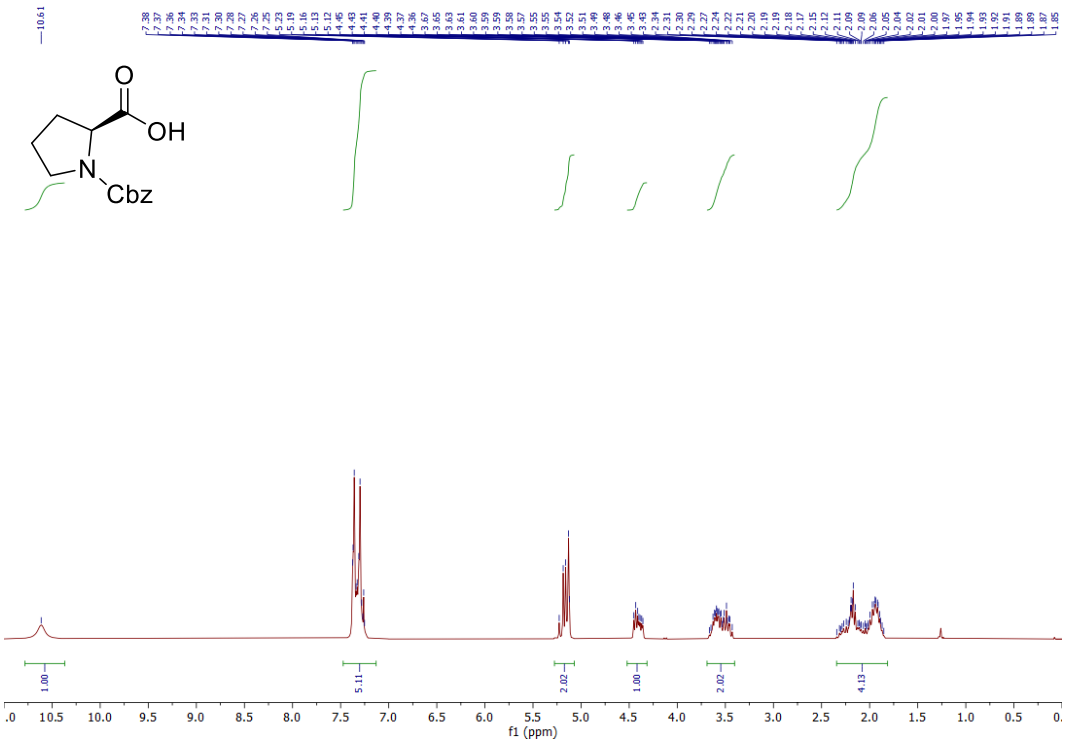
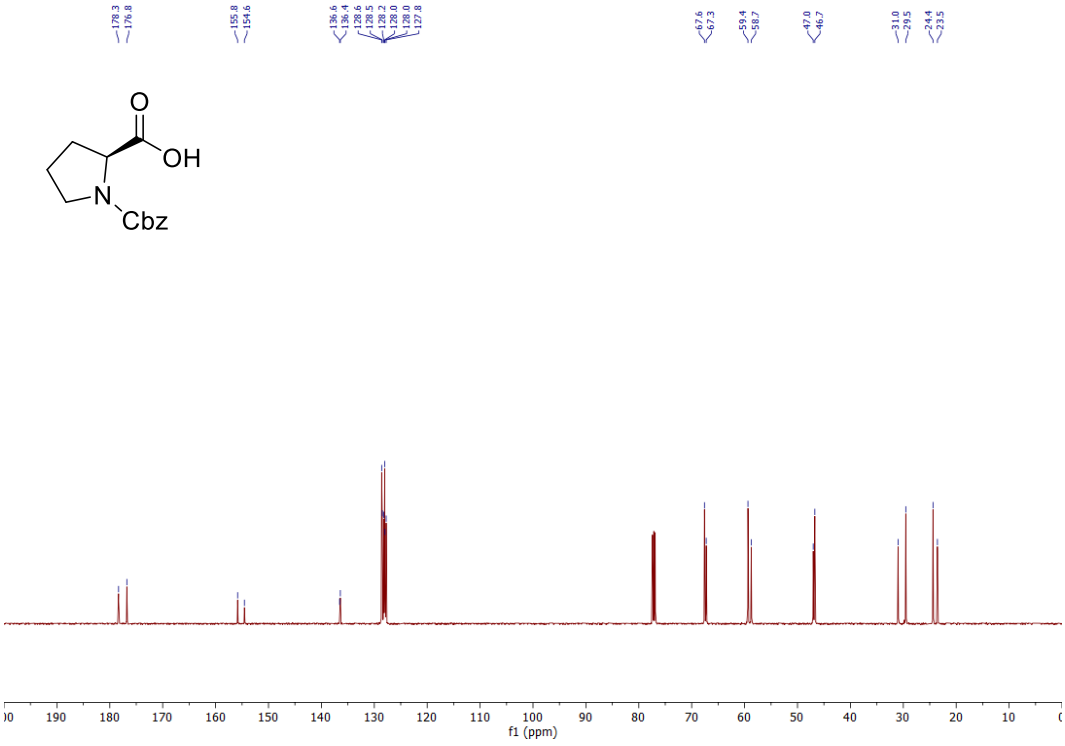
Copy of NMR spectra

^{1}H NMR spectrum of **1q** (300 MHz, $CDCl_3$)



^{13}C NMR spectrum of **1q** (126 MHz, $CDCl_3$)



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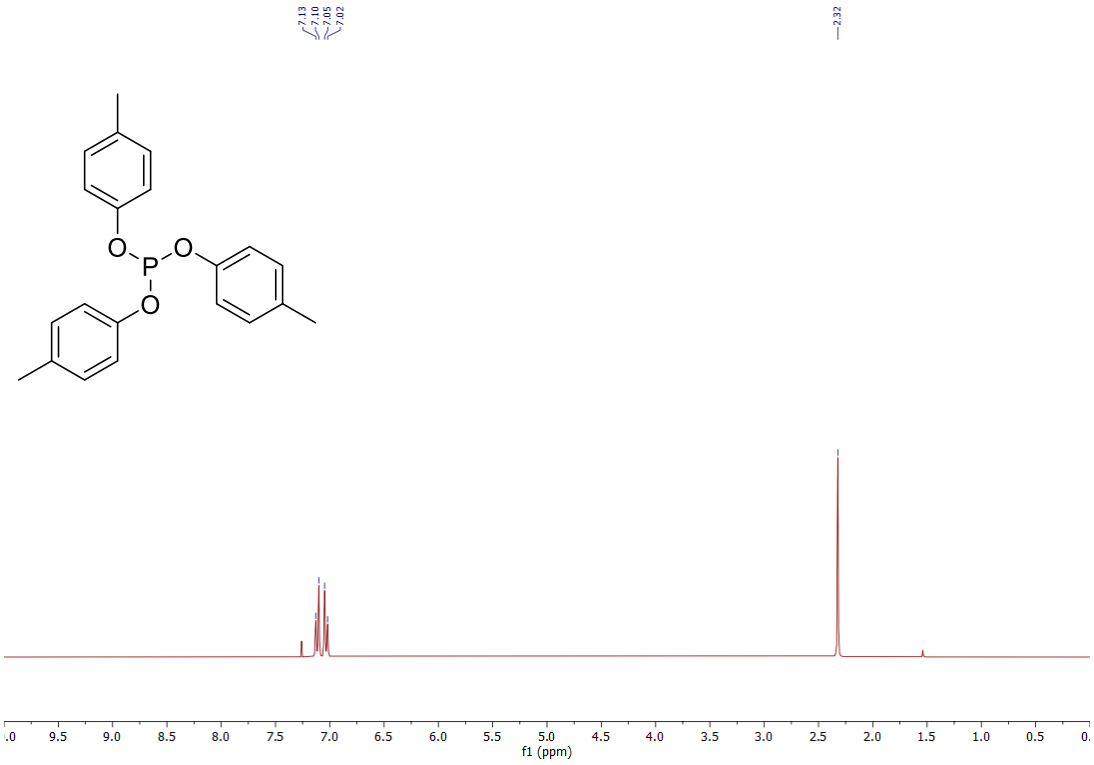
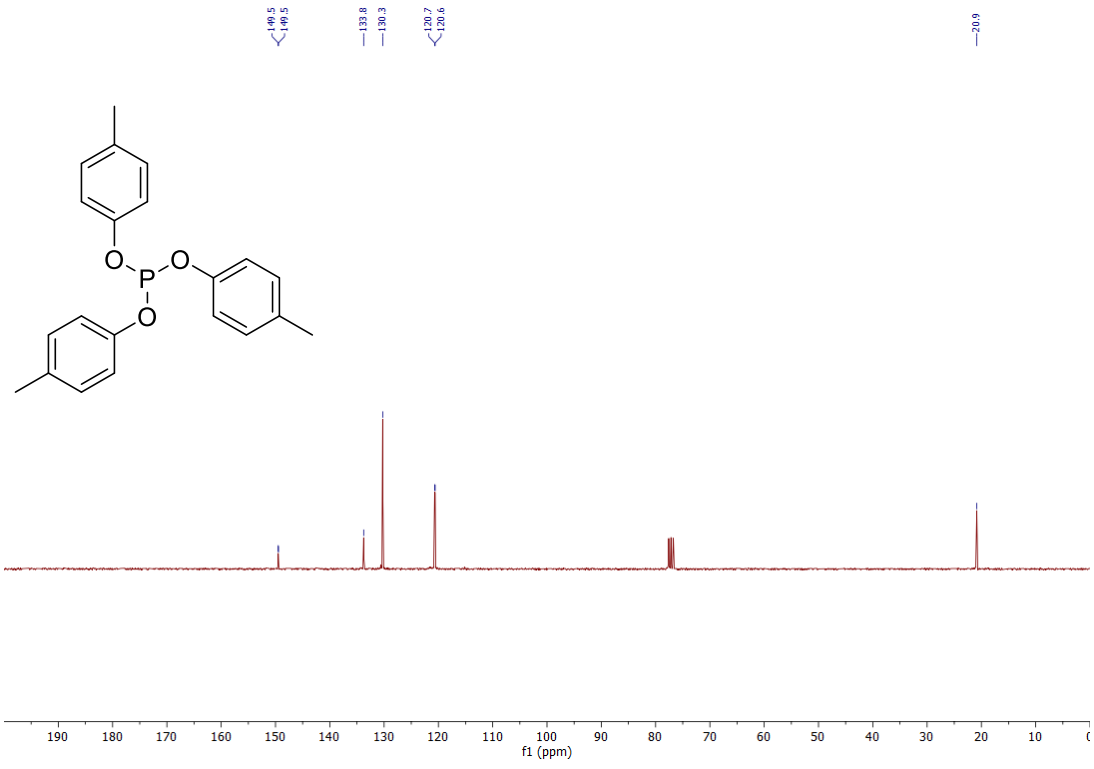


^{13}C NMR spectrum of **2c** (75 MHz, $CDCl_3$)



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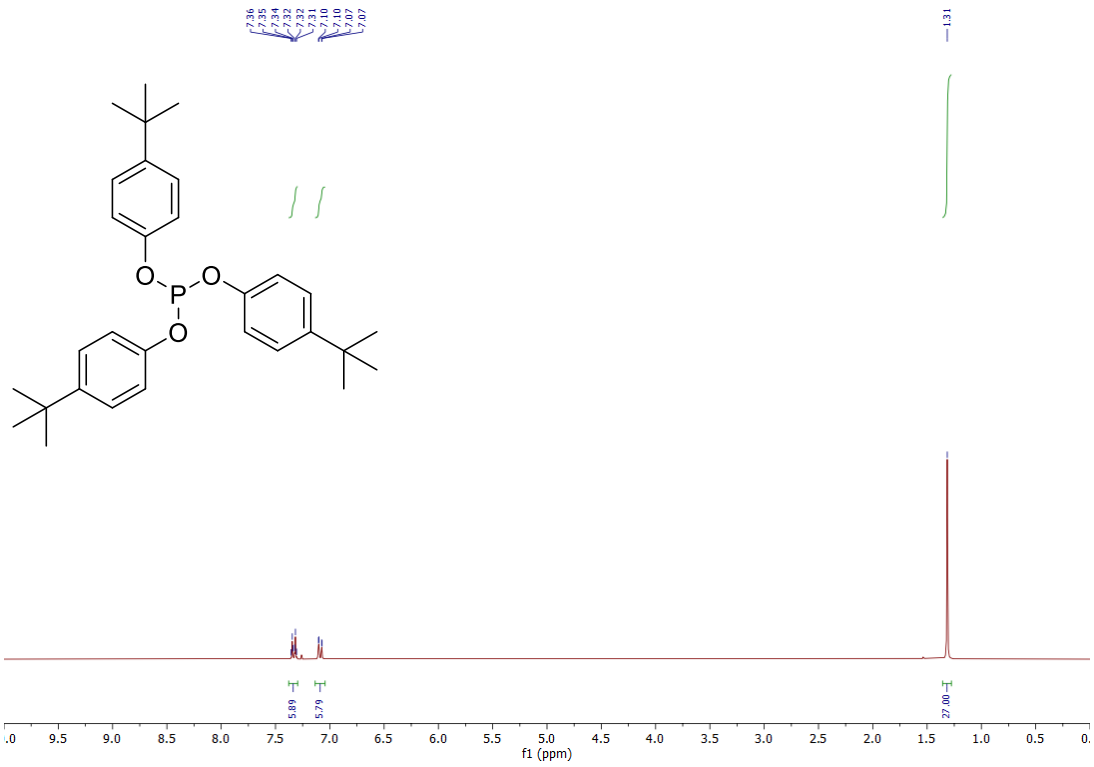

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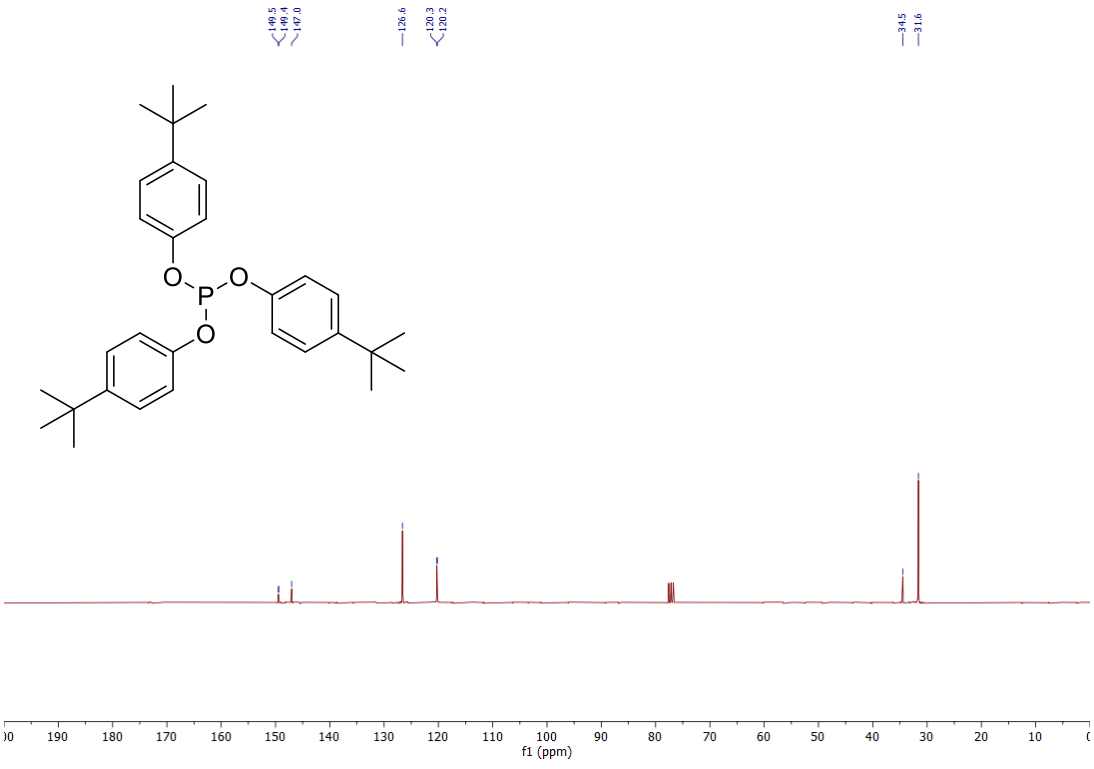

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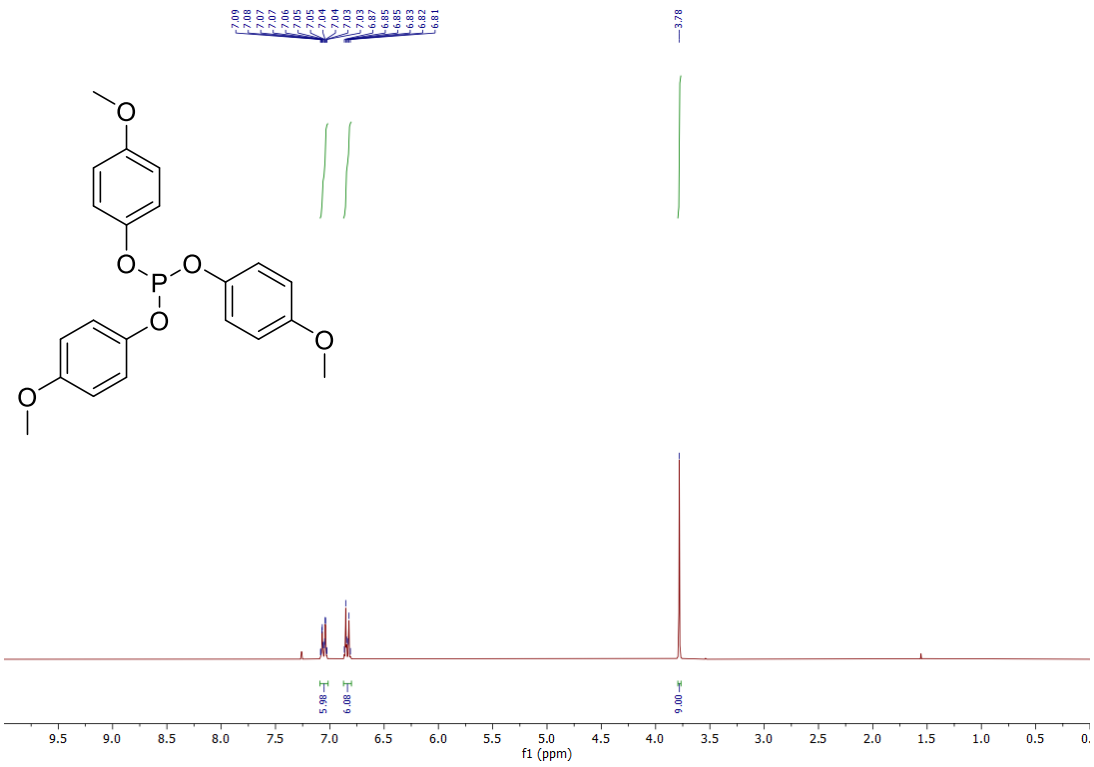
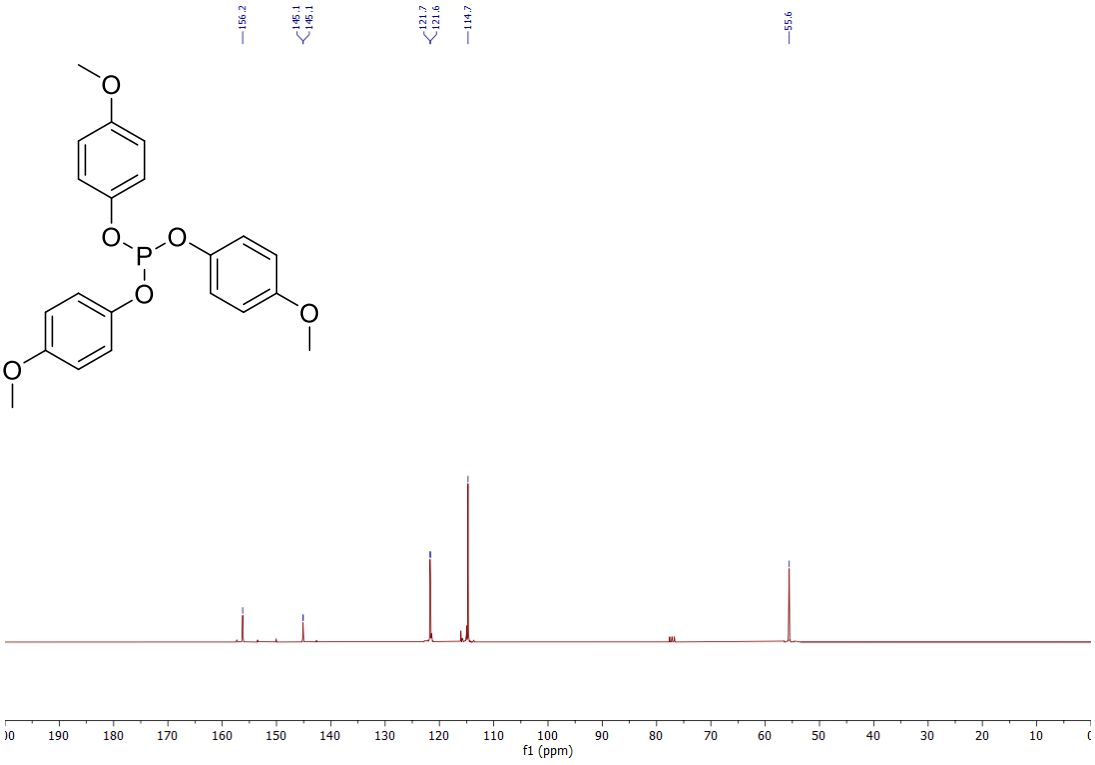


^{13}C NMR spectrum of **2e** (75 MHz, $CDCl_3$)



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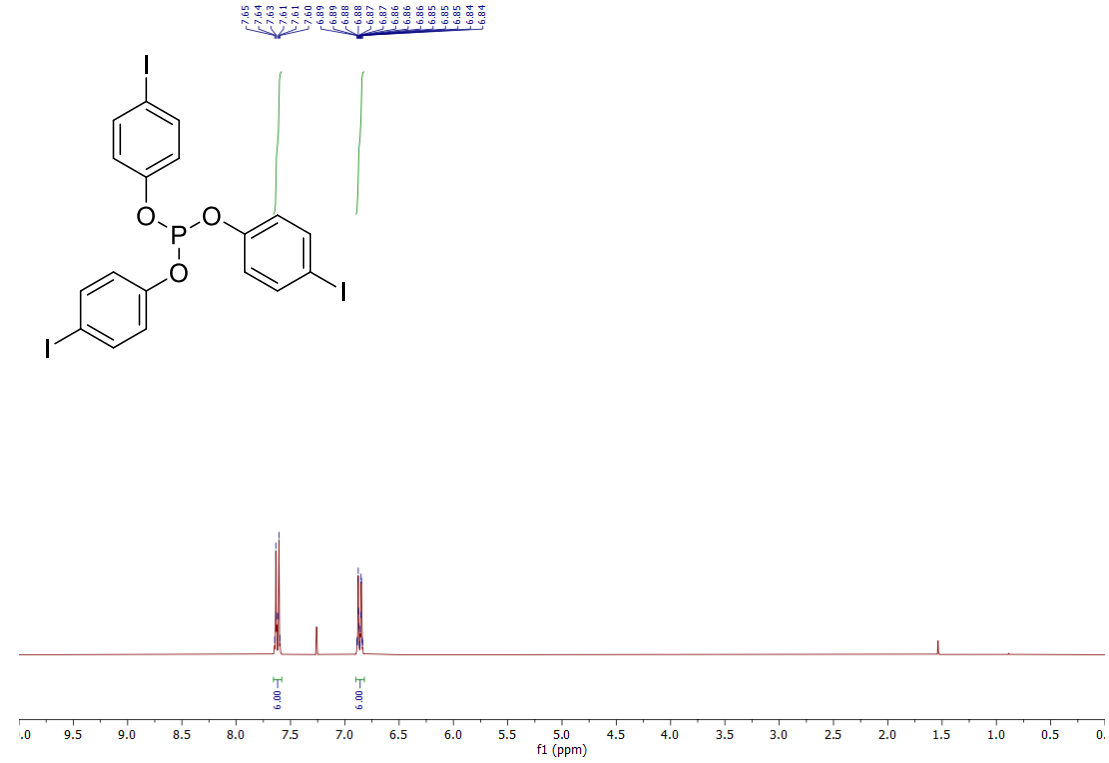

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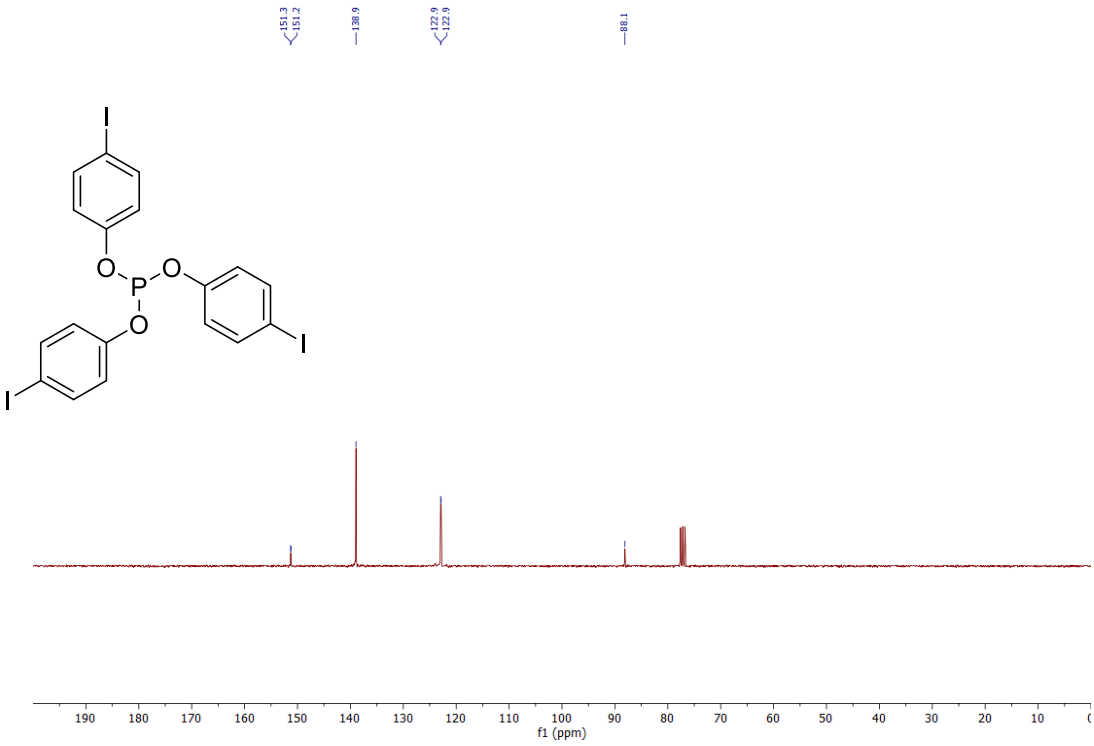

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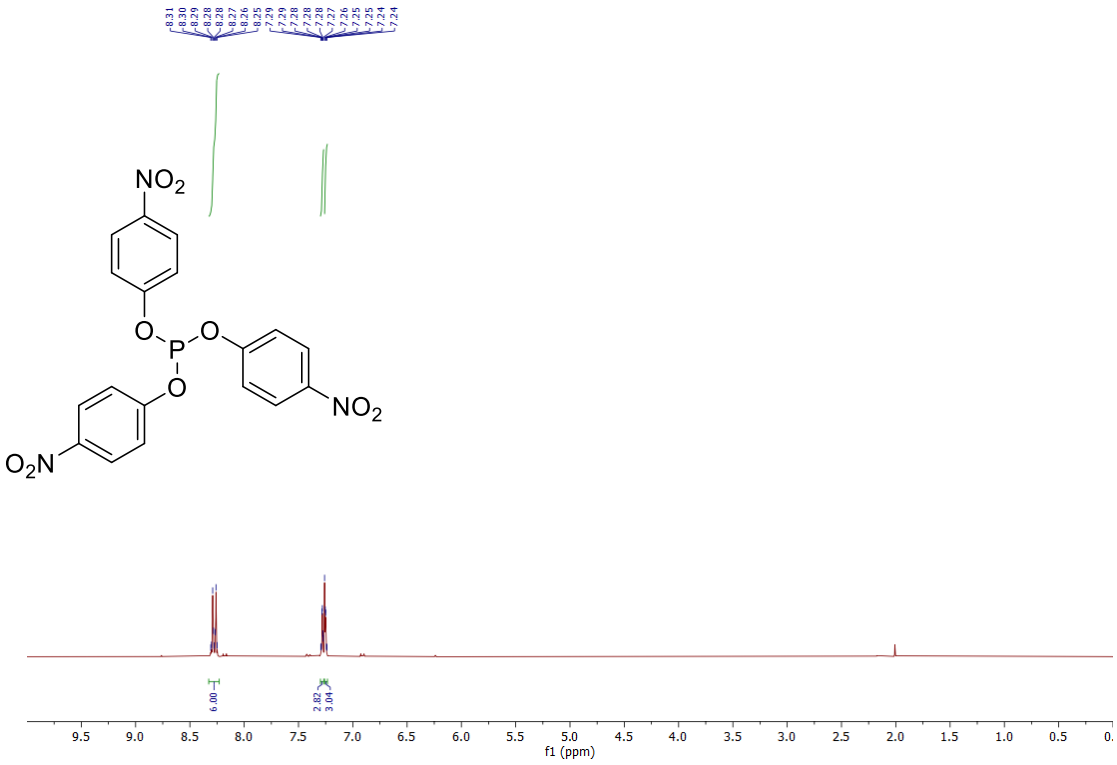
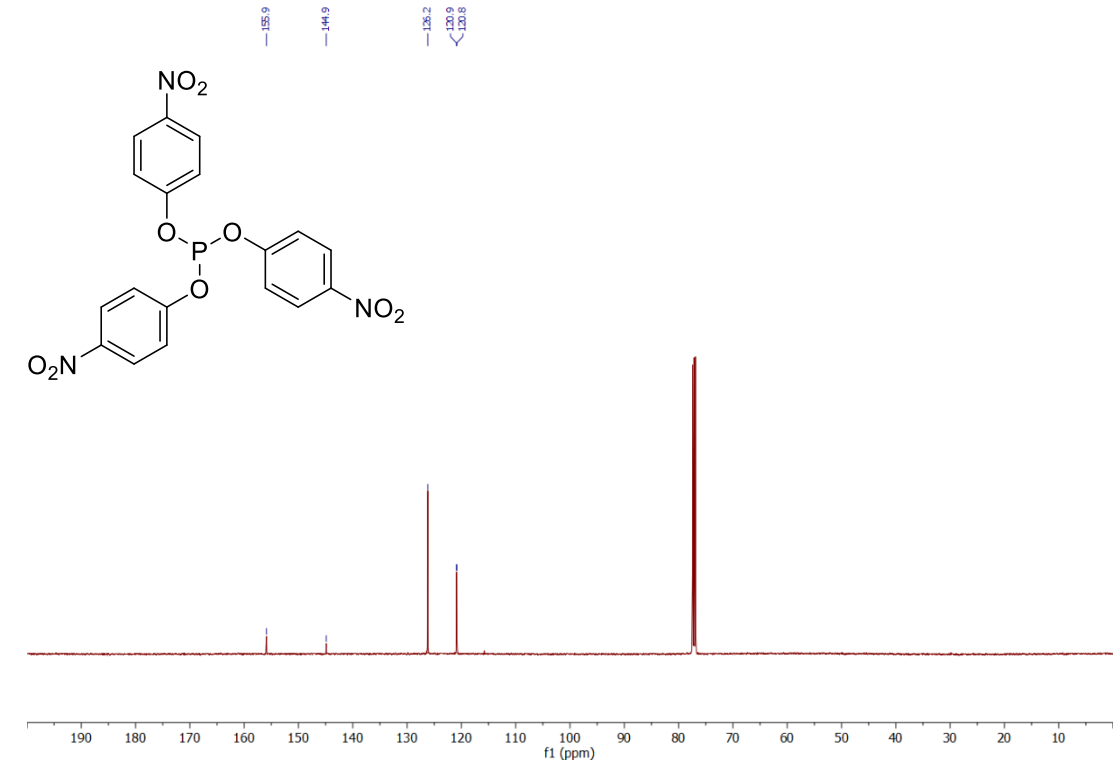
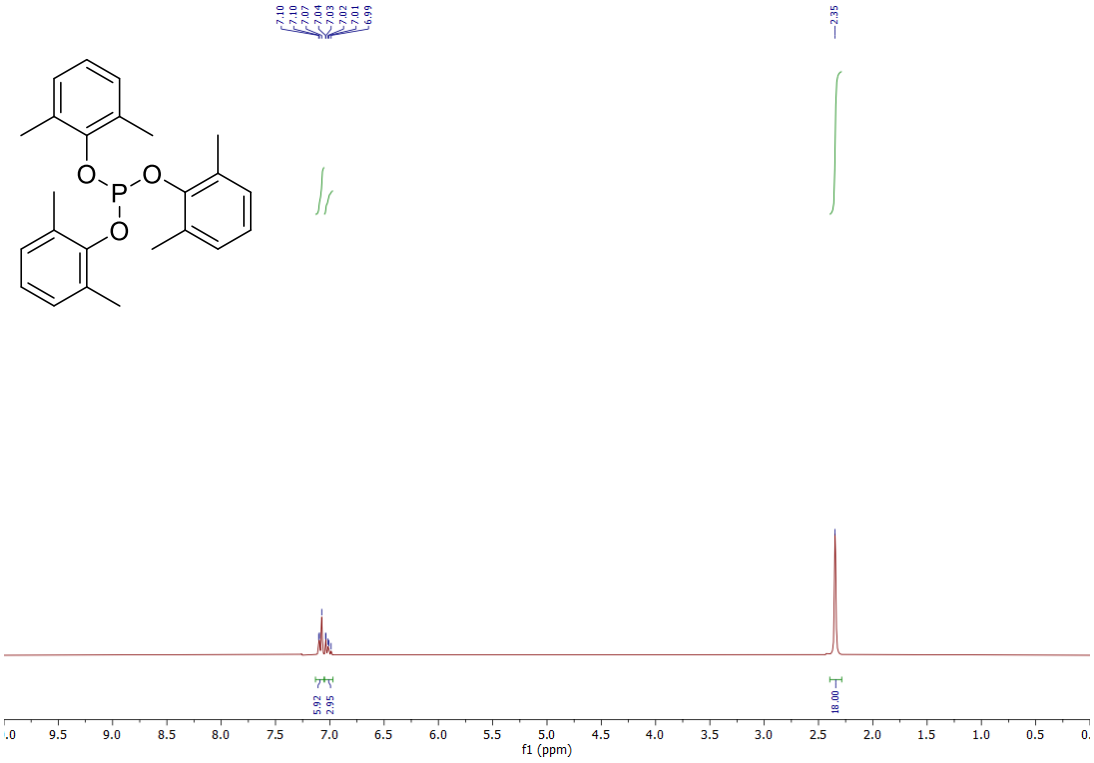


^{13}C NMR spectrum of **2g** (126 MHz, $CDCl_3$)



^{1}H NMR spectrum of **2h** (300 MHz, $CDCl_3$)



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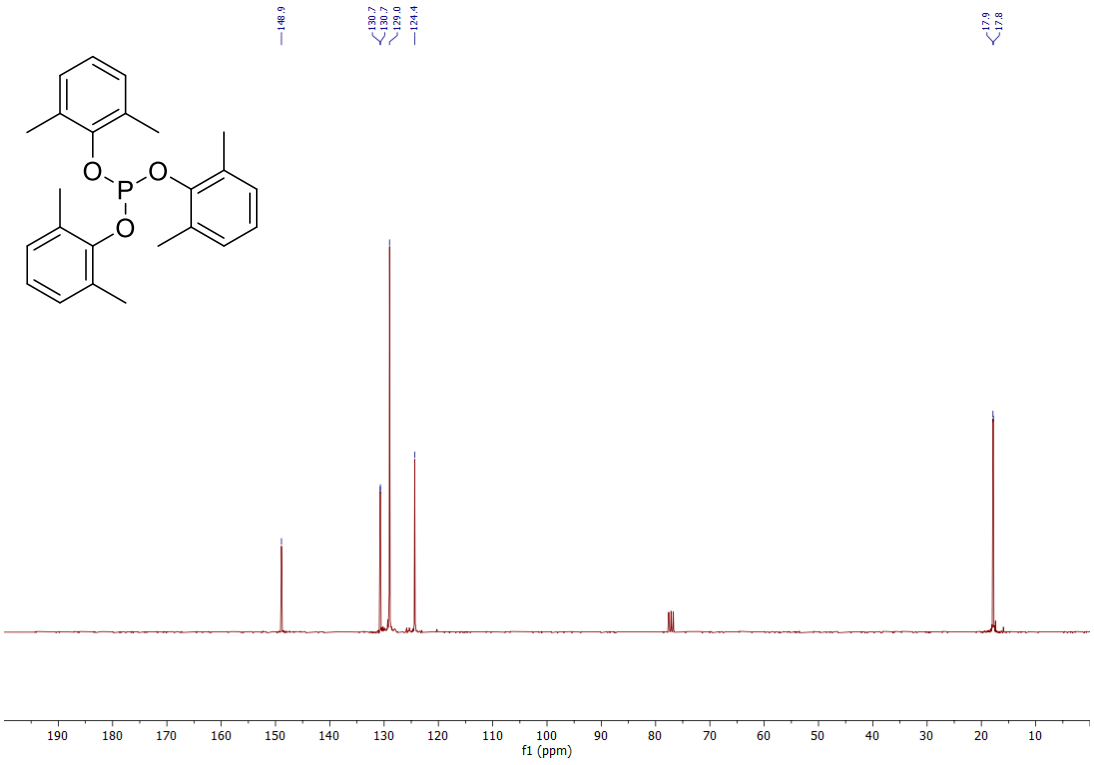
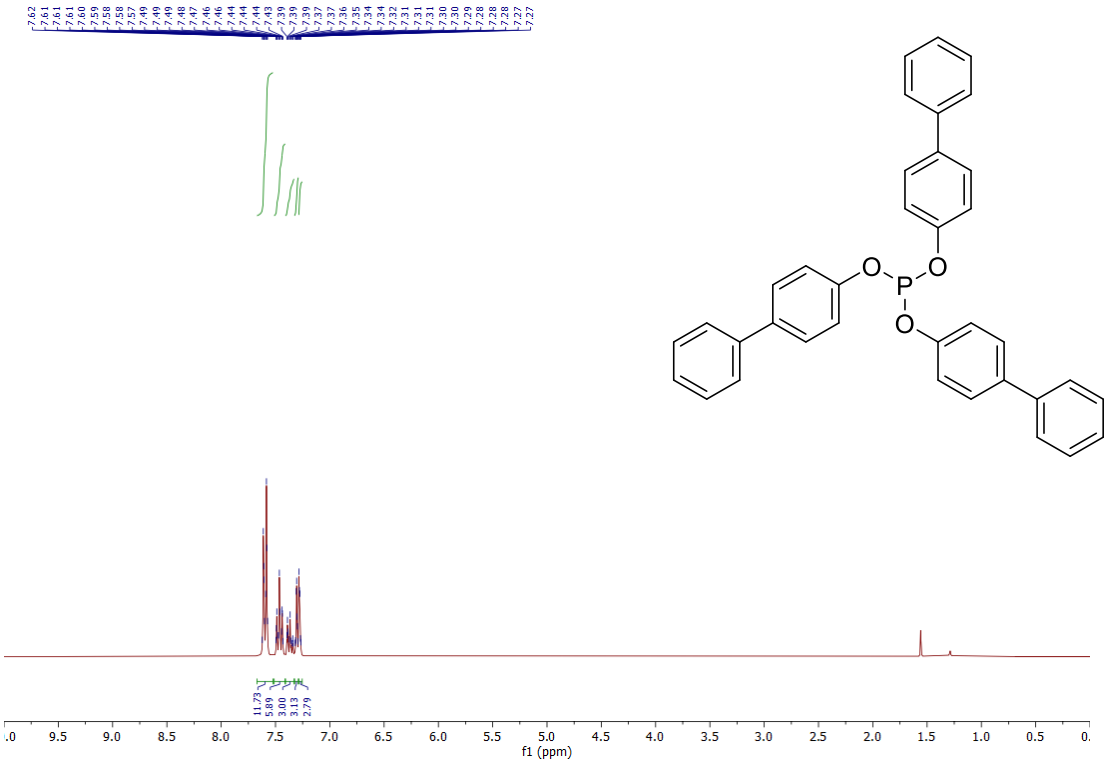


^{1}H NMR spectrum of **2i** (300 MHz, $CDCl_3$)



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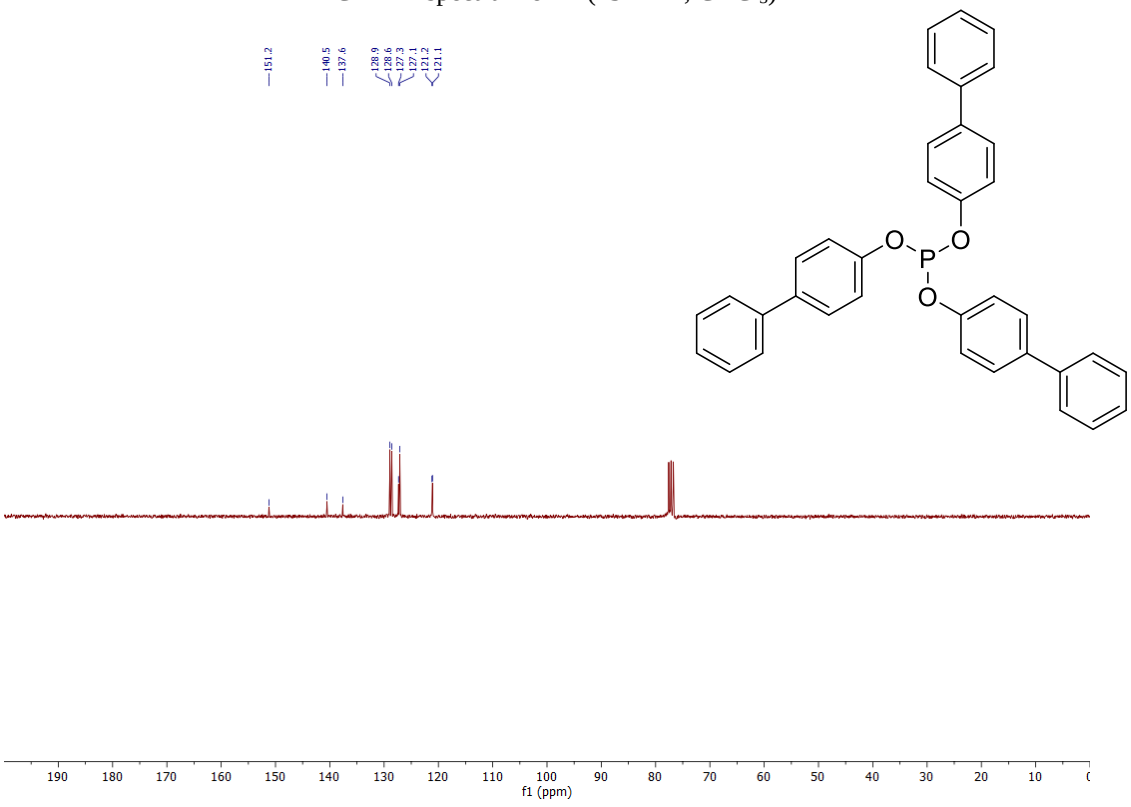

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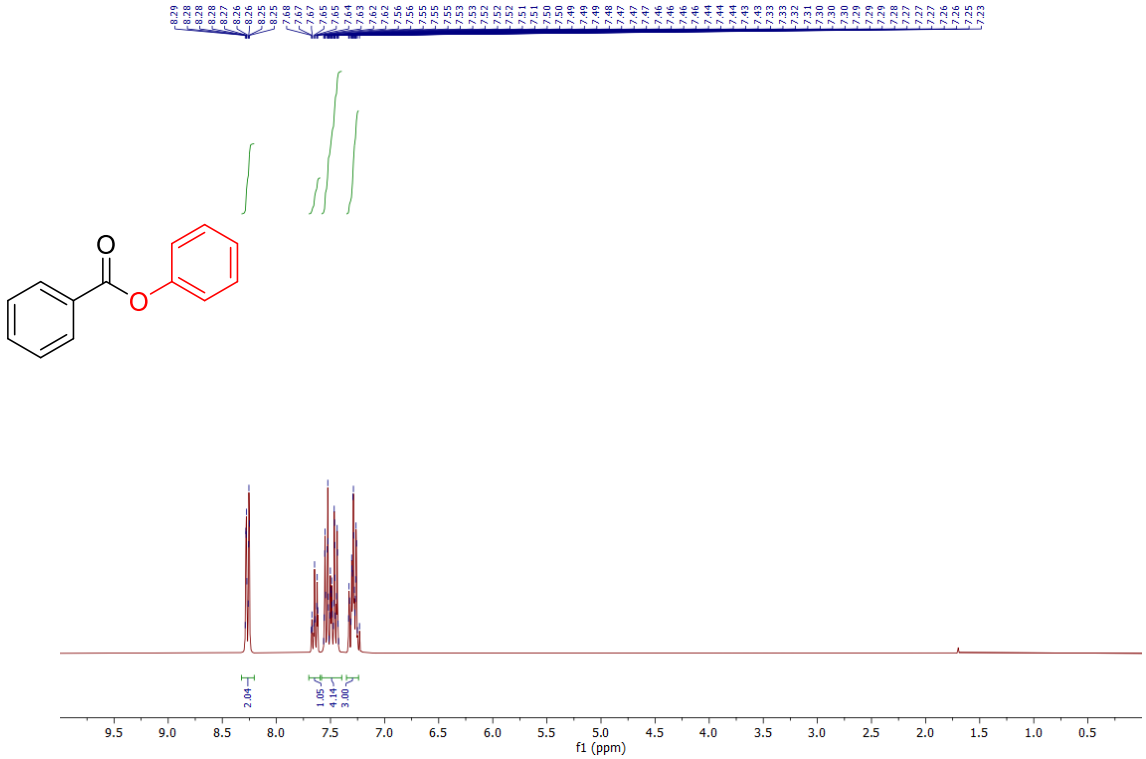

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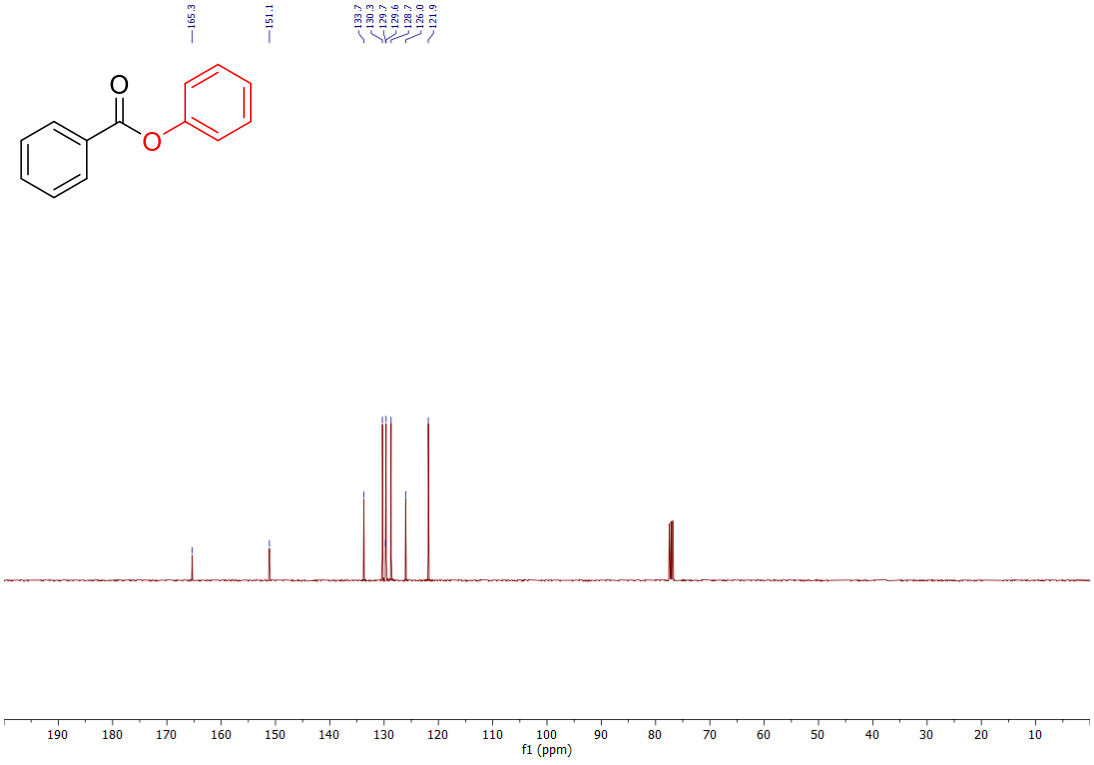
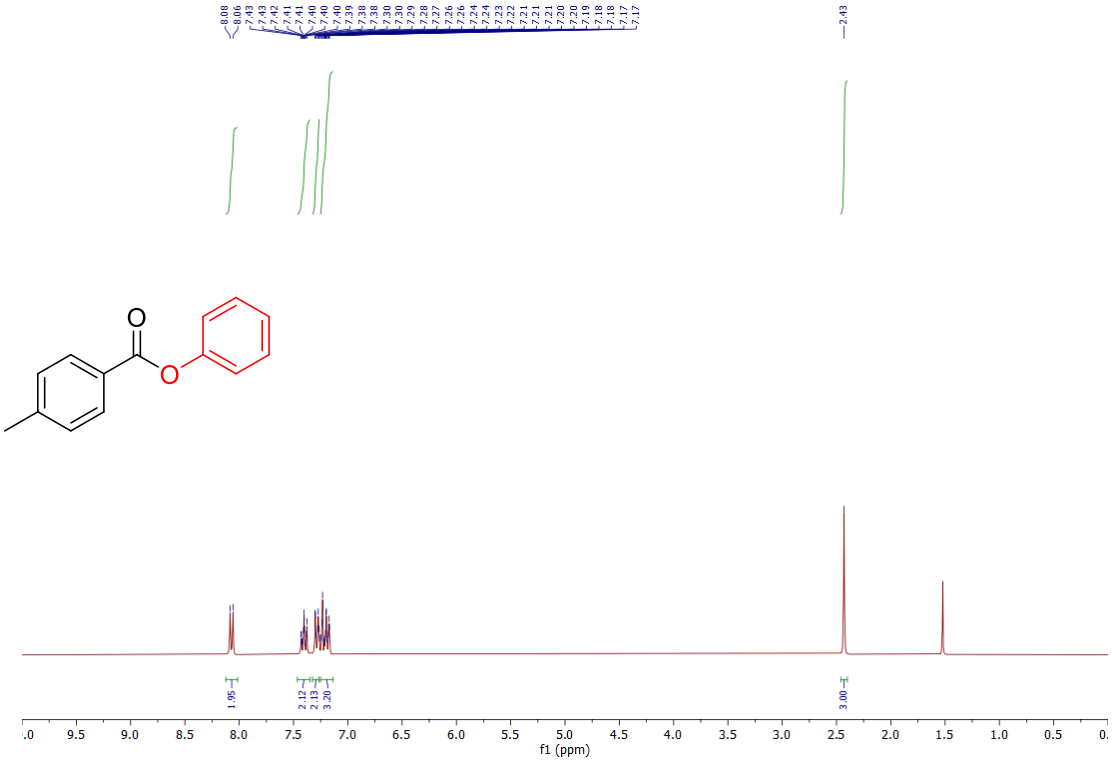
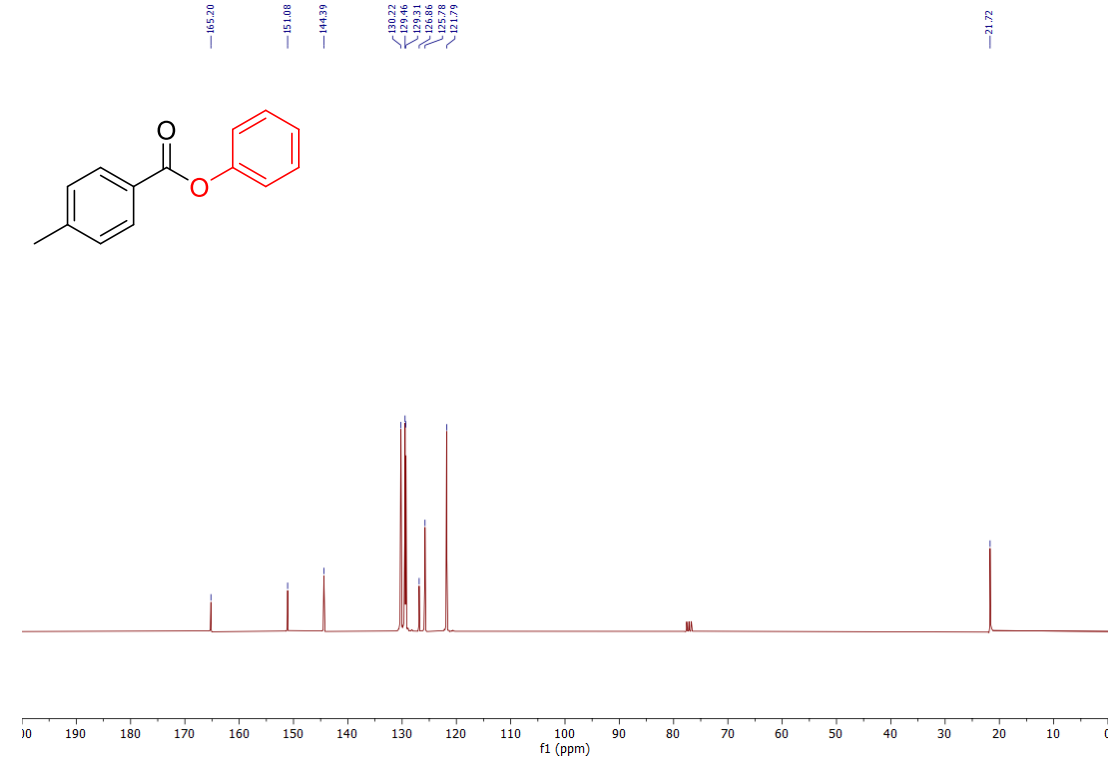


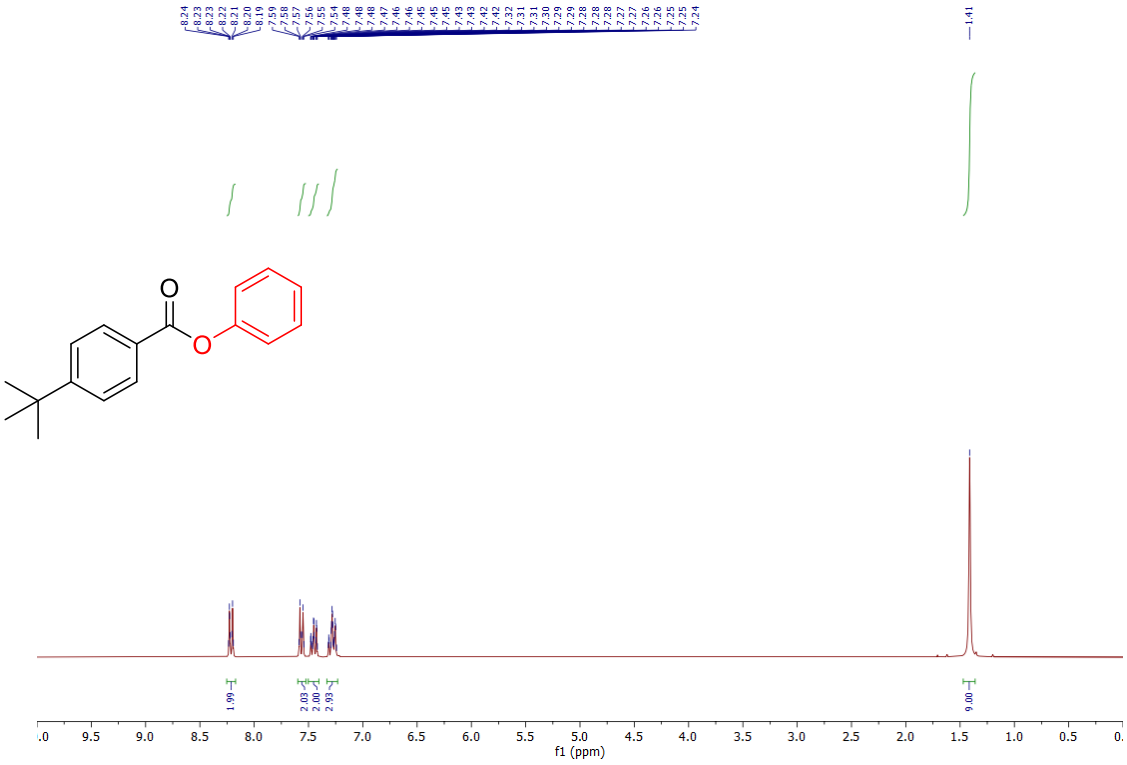
^{1}H NMR spectrum of **4b** (300 MHz, $CDCl_3$)



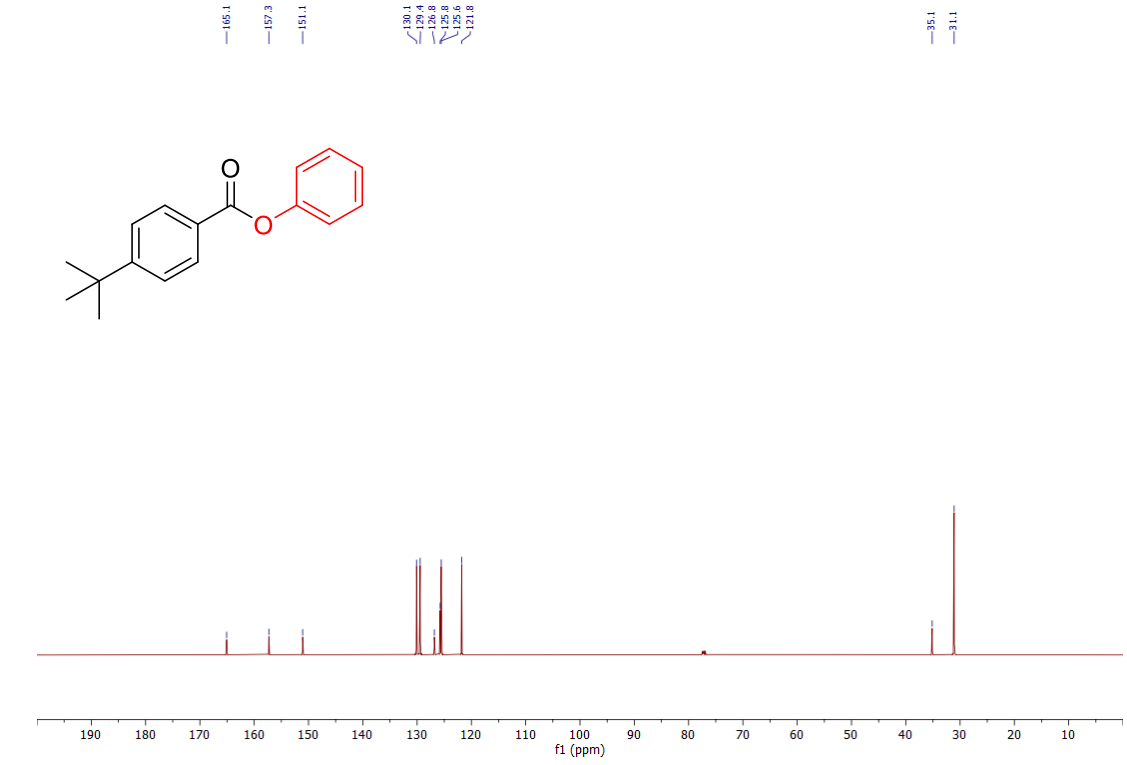
^{13}C NMR spectrum of **4b** (75 MHz, $CDCl_3$)



^{1}H NMR spectrum of **4c** (300 MHz, $CDCl_3$)



^{13}C NMR spectrum of **4c** (126 MHz, $CDCl_3$)



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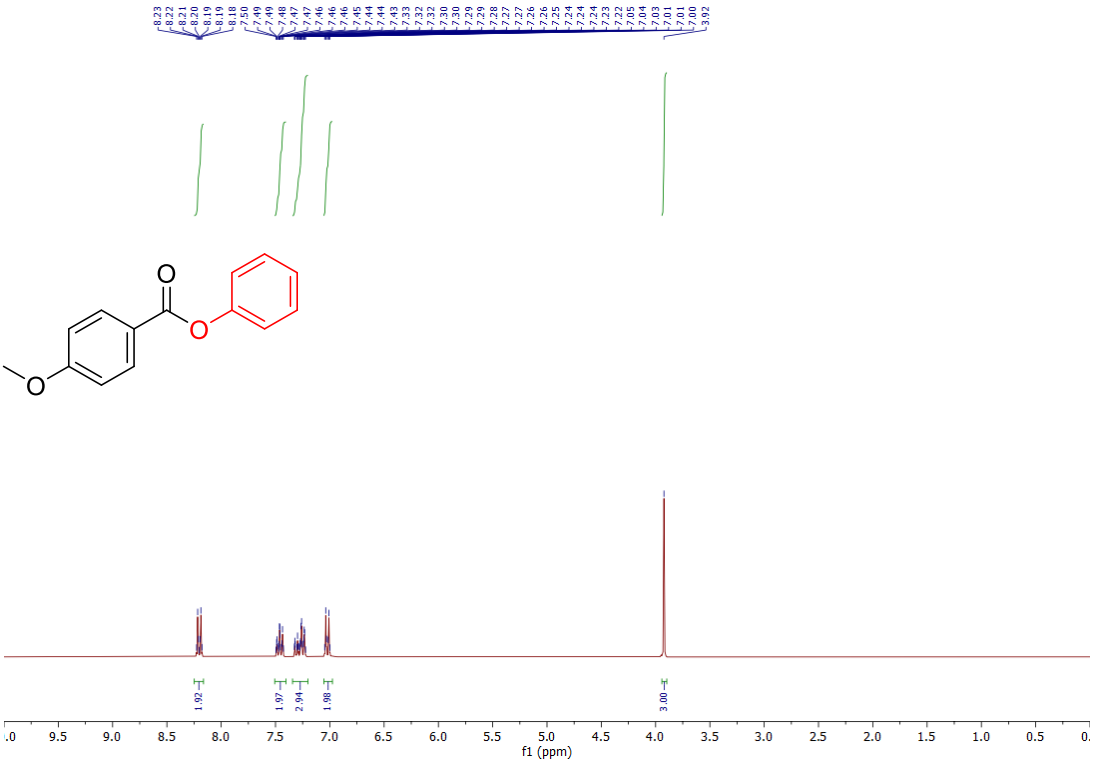
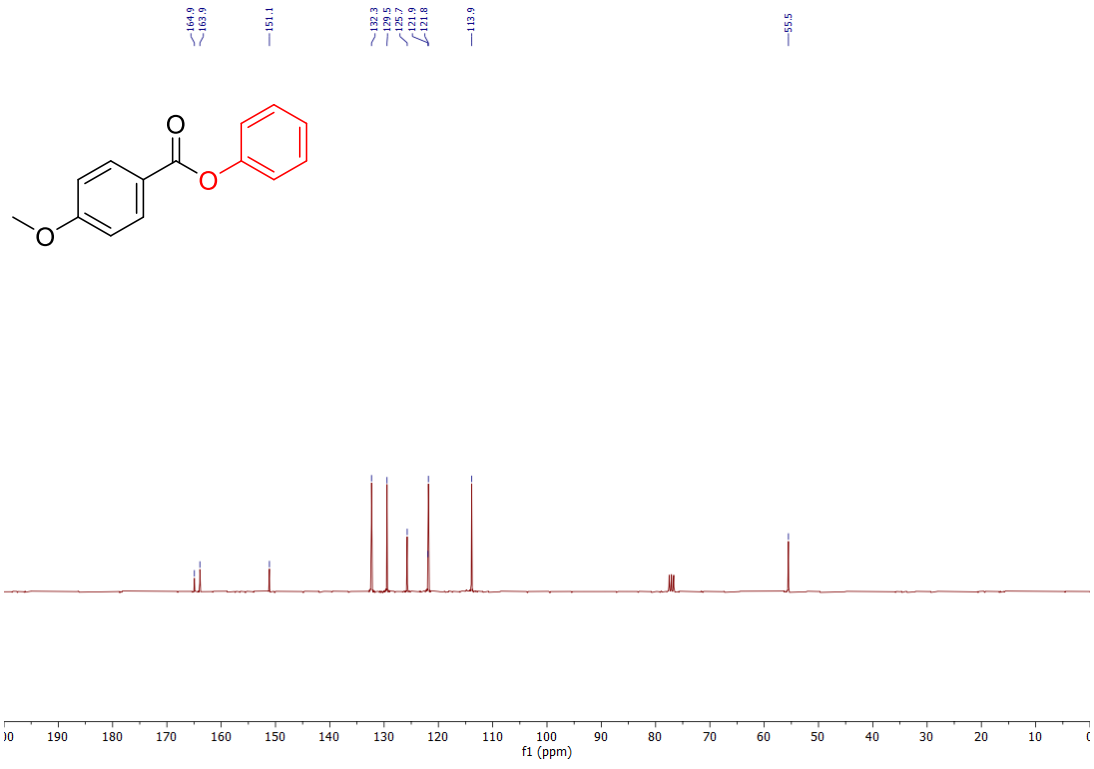


^{13}C NMR spectrum of **4d** (75 MHz, $CDCl_3$)



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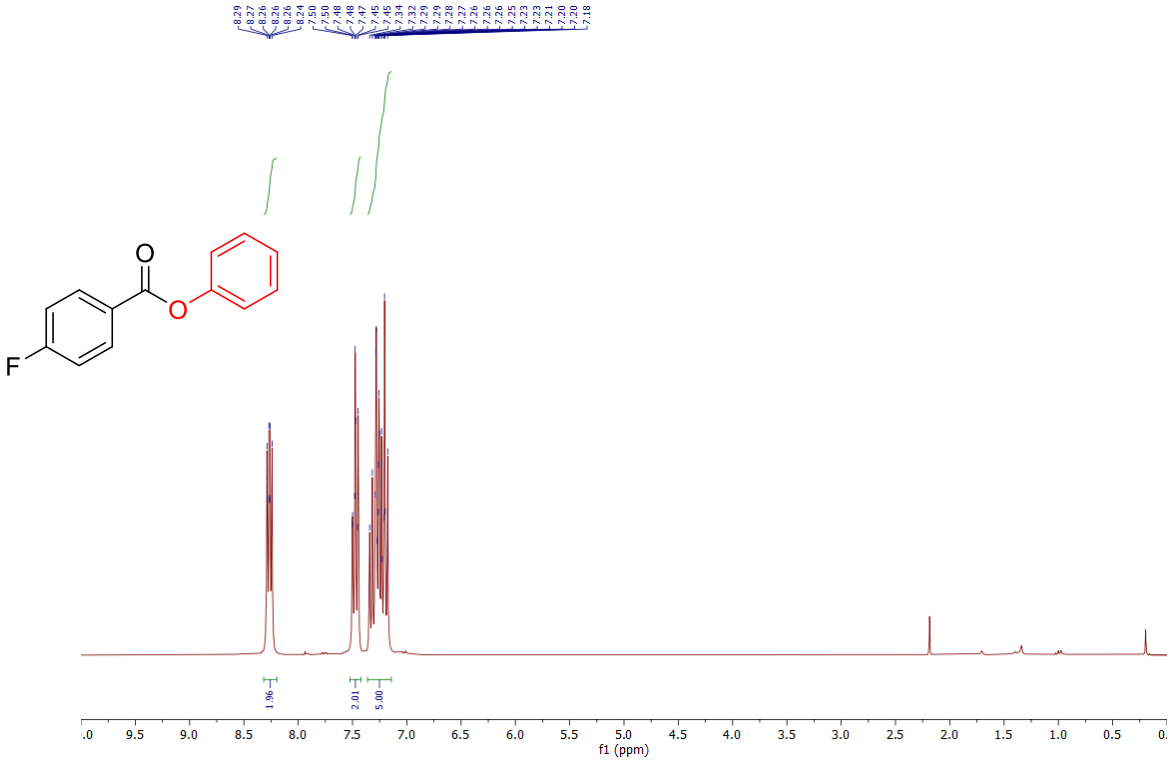

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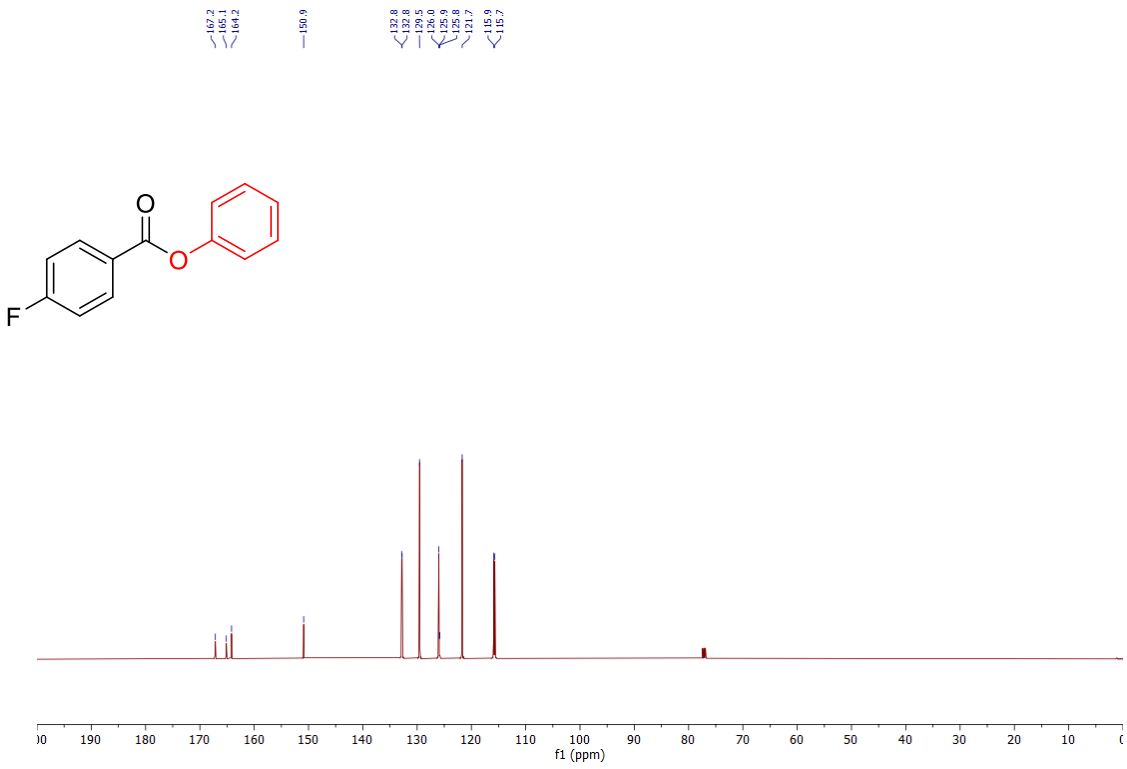
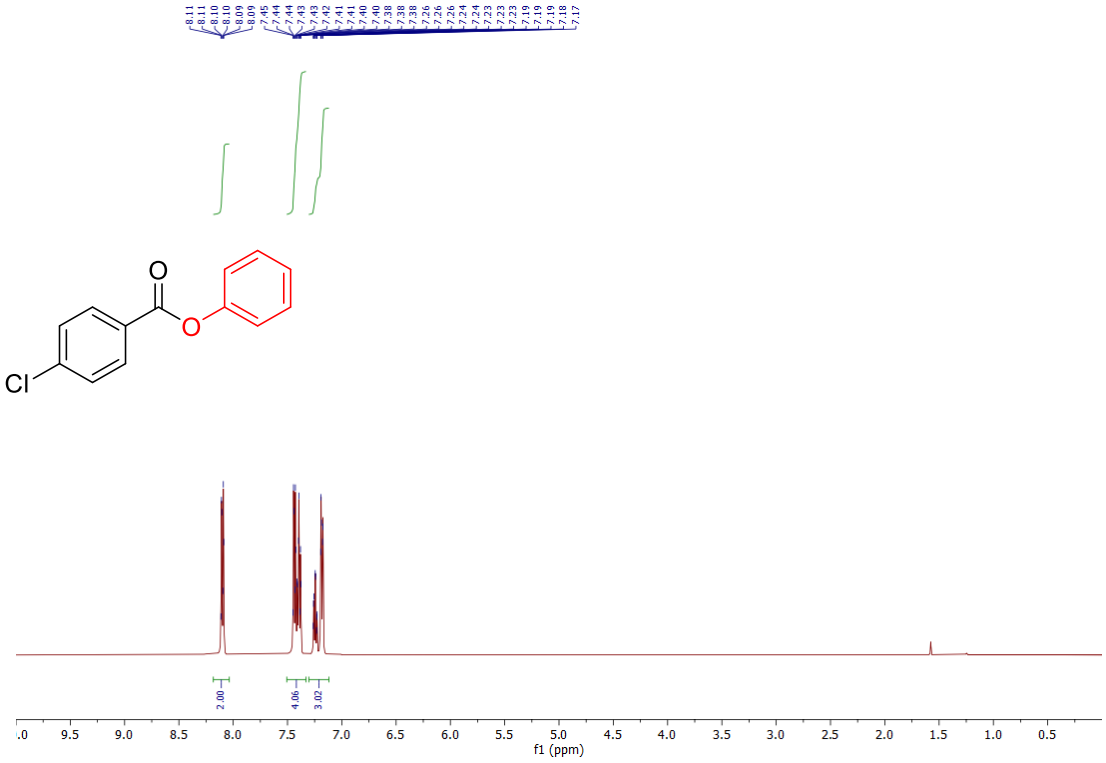
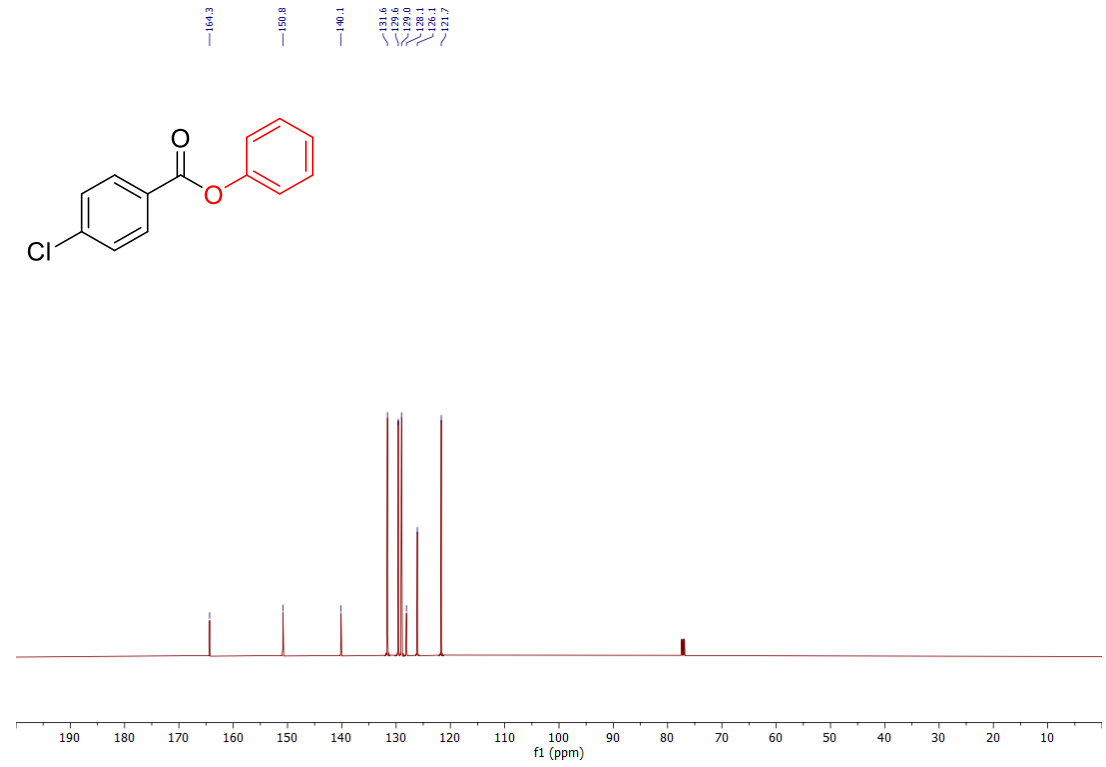


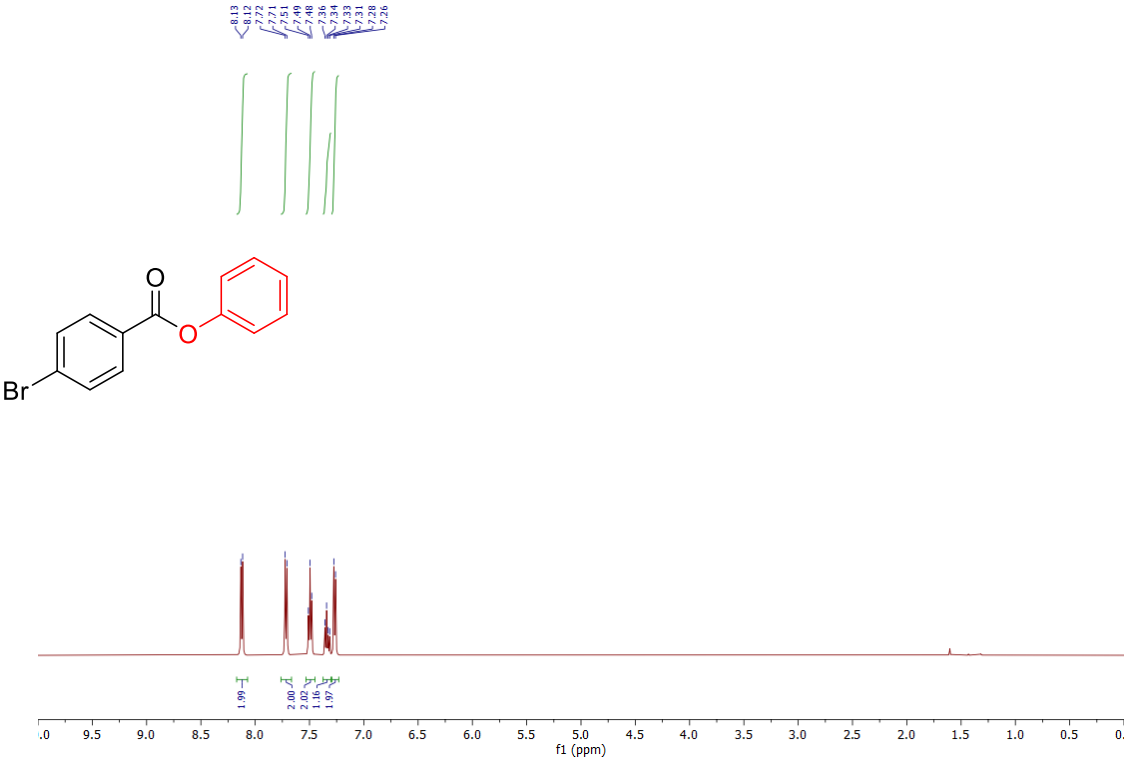
^{1}H NMR spectrum of **4f** (500 MHz, $CDCl_3$)



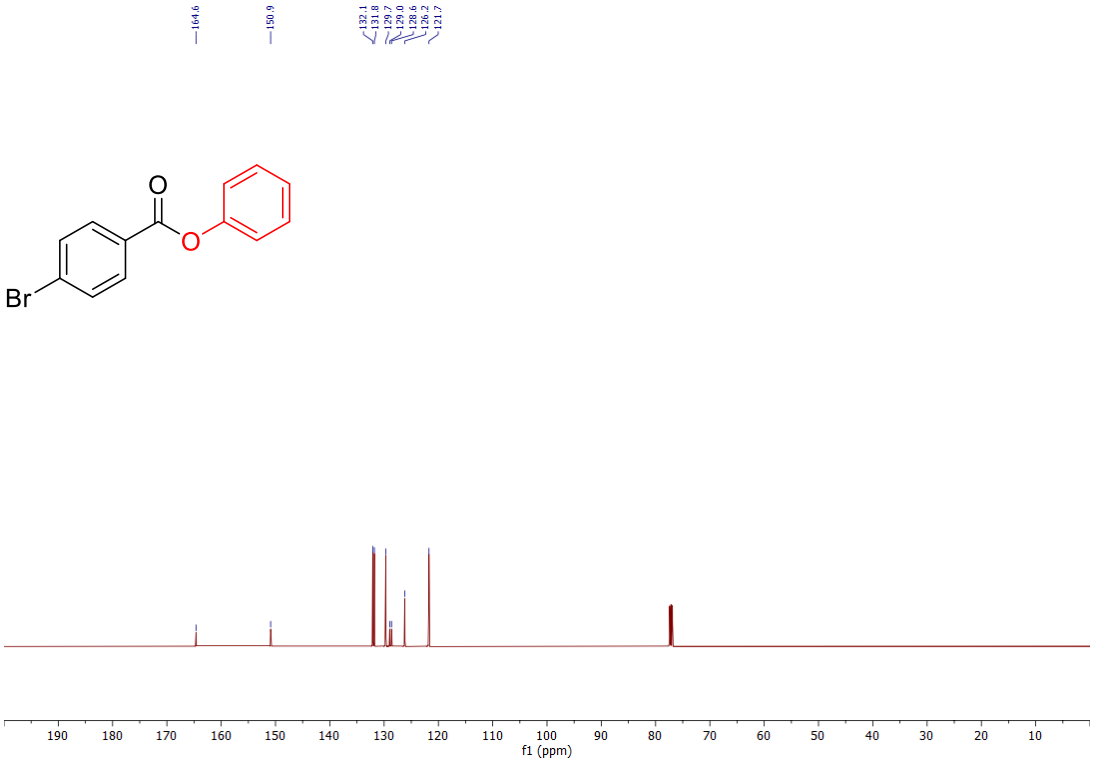
^{13}C NMR spectrum of **4f** (126 MHz, $CDCl_3$)



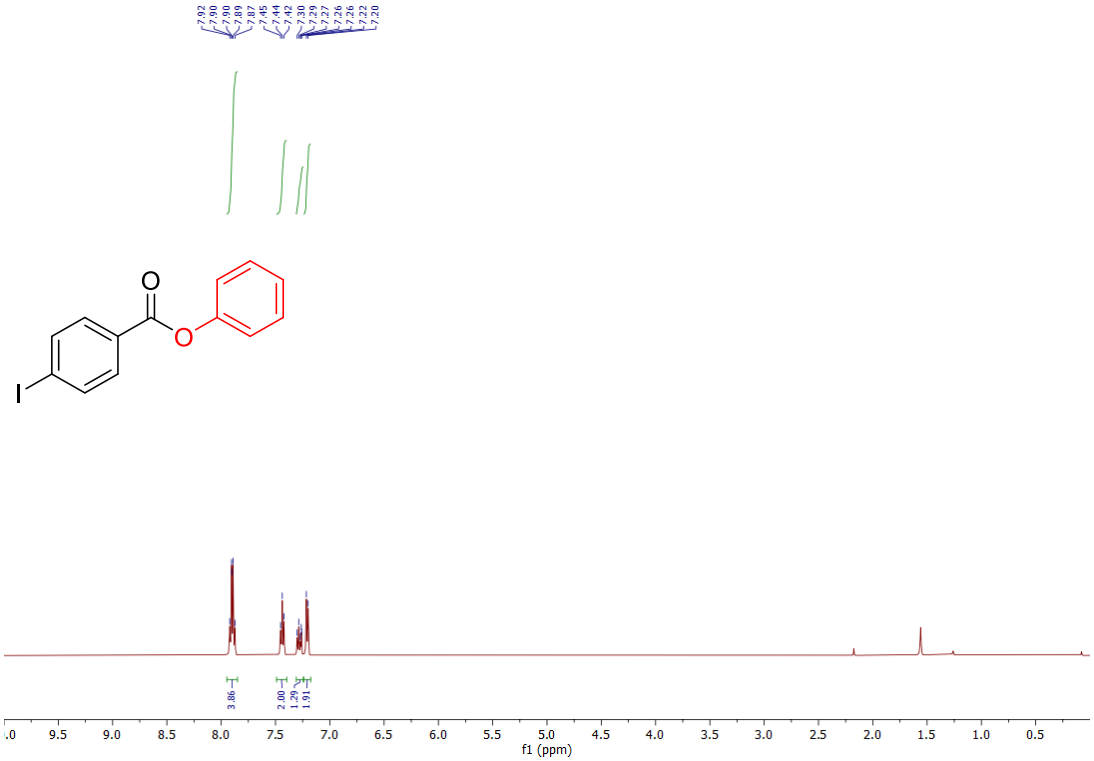
^{1}H NMR spectrum of **4g** (500 MHz, $CDCl_3$)



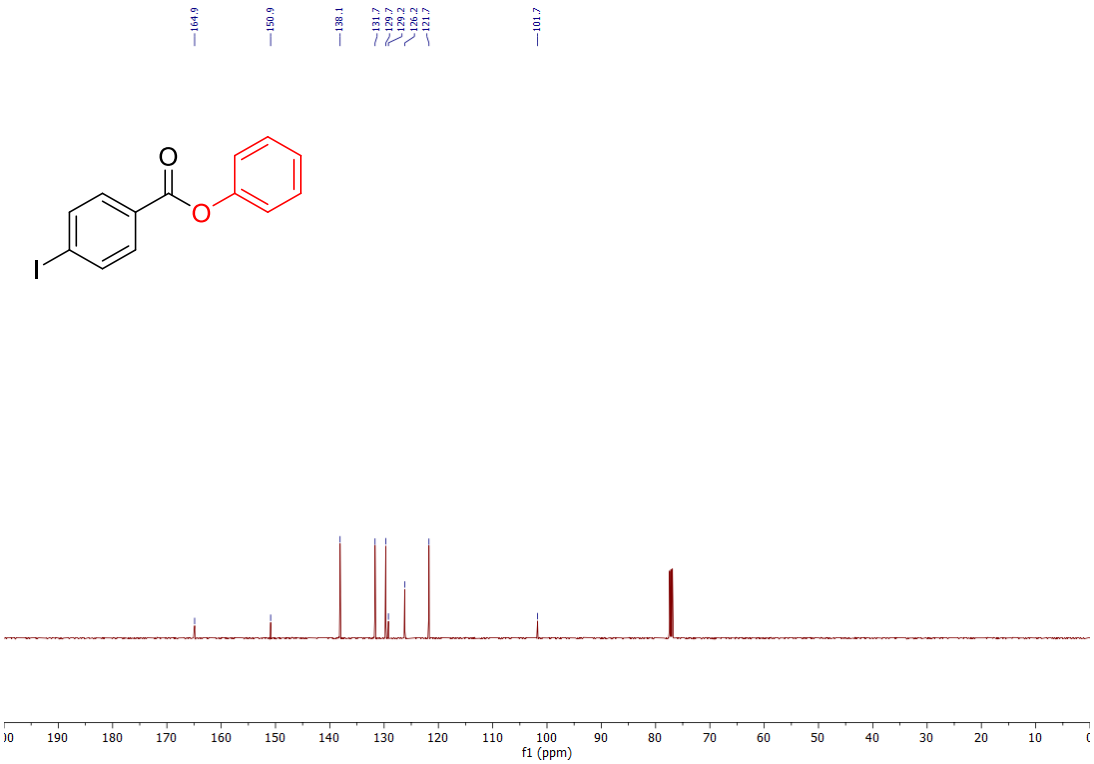
^{13}C NMR spectrum of **4g** (126 MHz, $CDCl_3$)



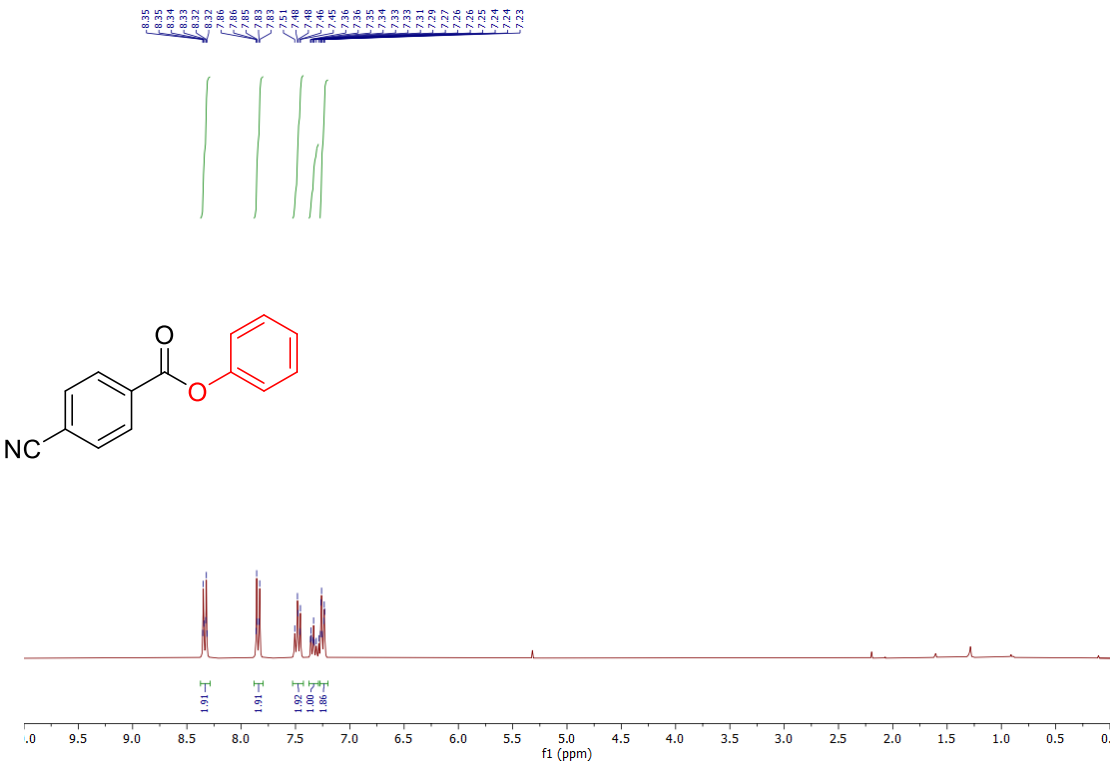
^{1}H NMR spectrum of **4h** (500 MHz, $CDCl_3$)



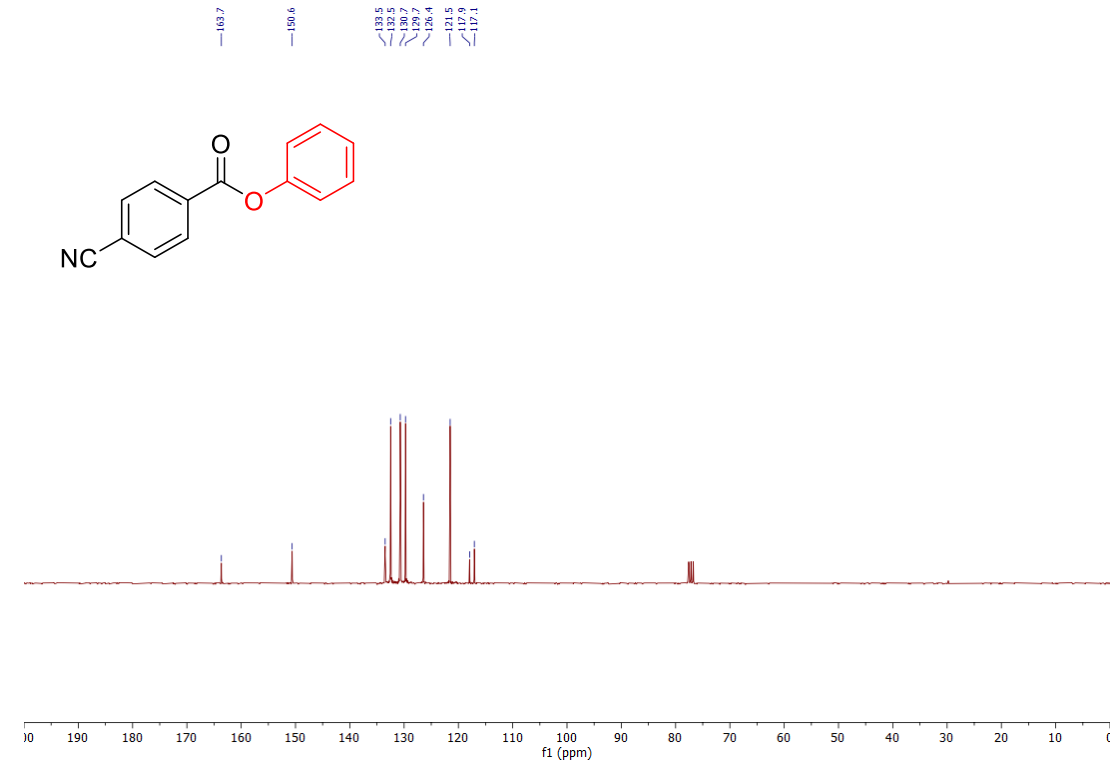
^{13}C NMR spectrum of **4h** (126 MHz, $CDCl_3$)



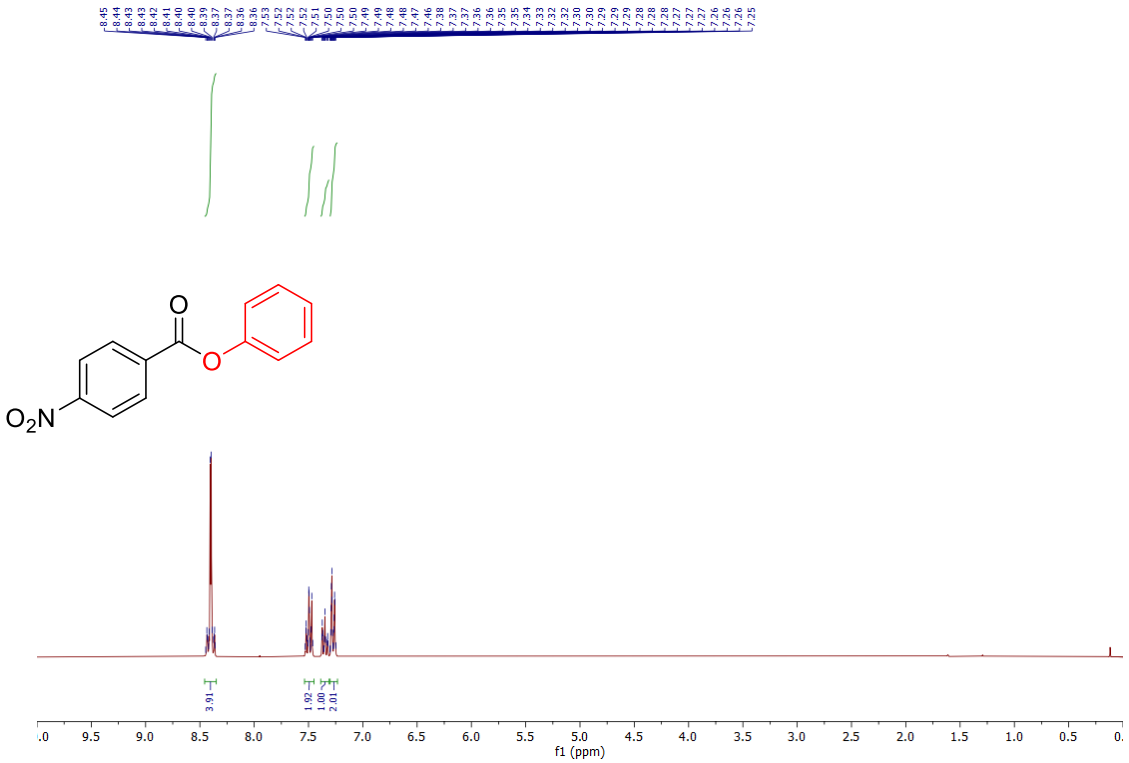
^{1}H NMR spectrum of **4i** (300 MHz, $CDCl_3$)



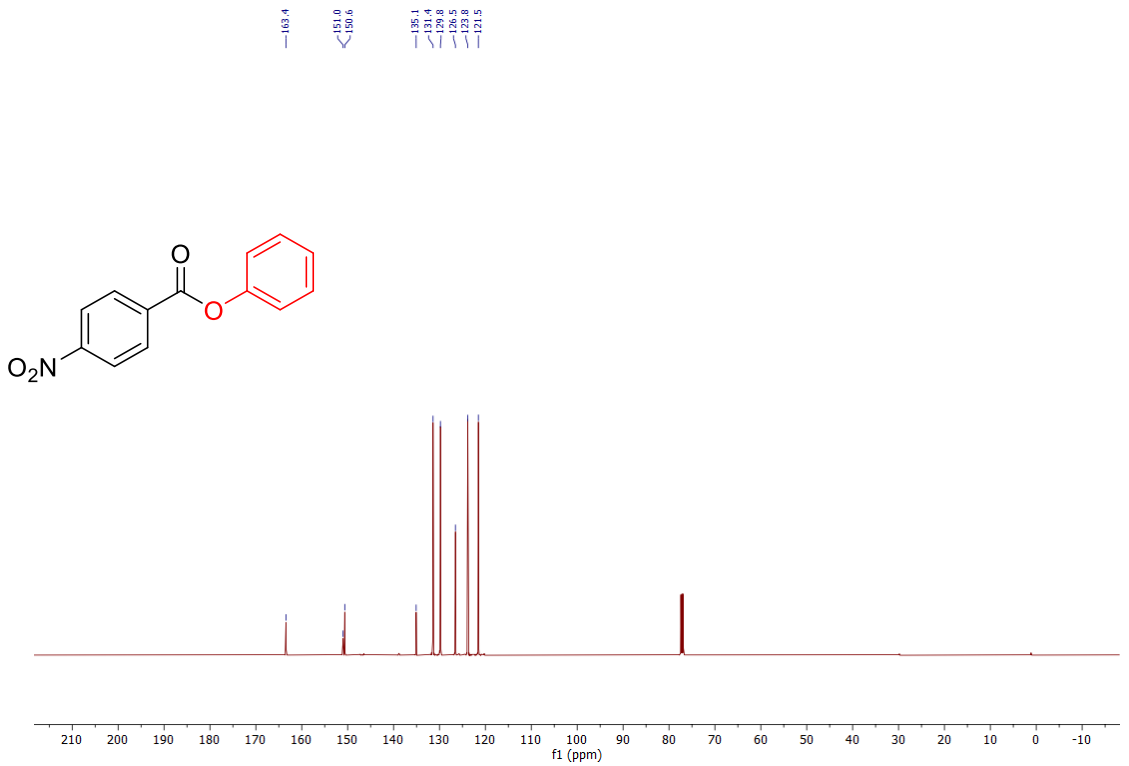
^{13}C NMR spectrum of **4i** (75 MHz, $CDCl_3$)



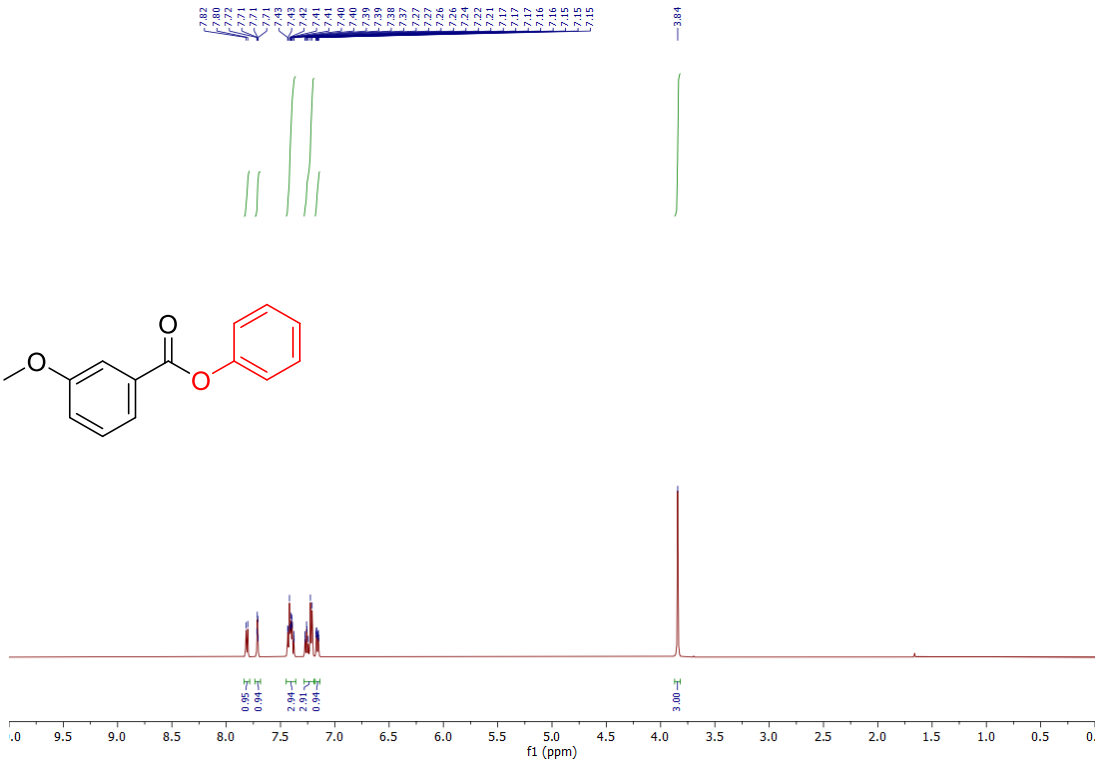
^{1}H NMR spectrum of **4j** (300 MHz, $CDCl_3$)



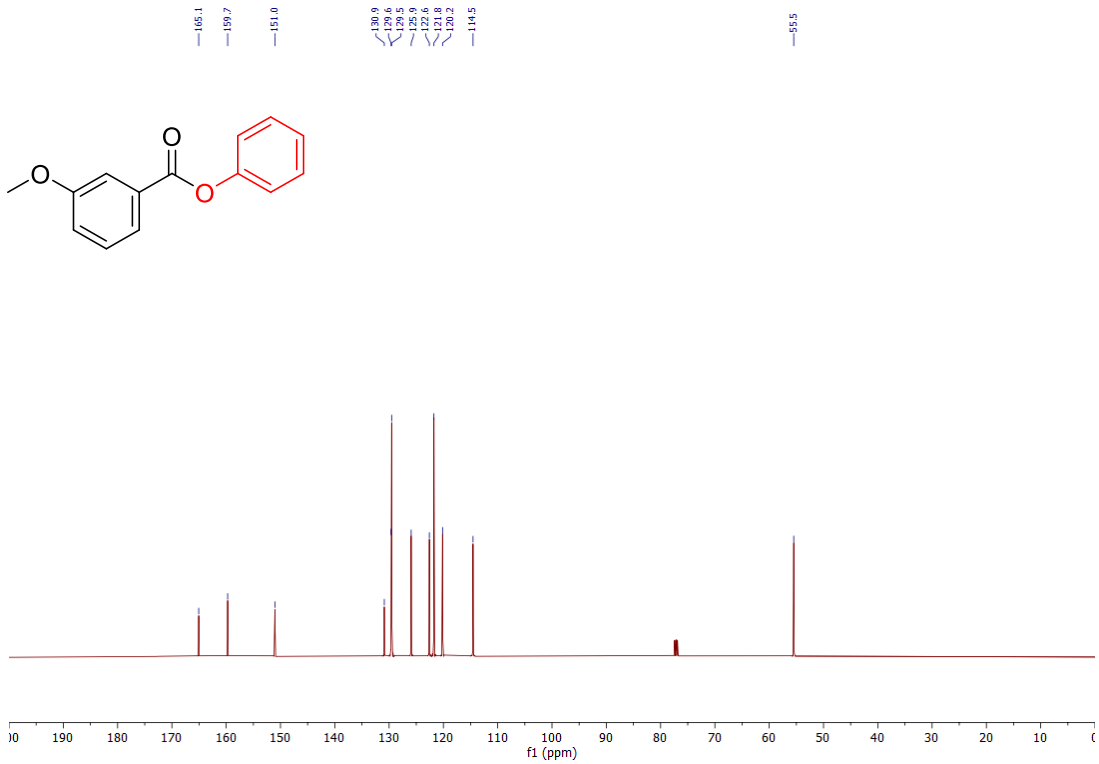
^{13}C NMR spectrum of **4j** (126 MHz, $CDCl_3$)



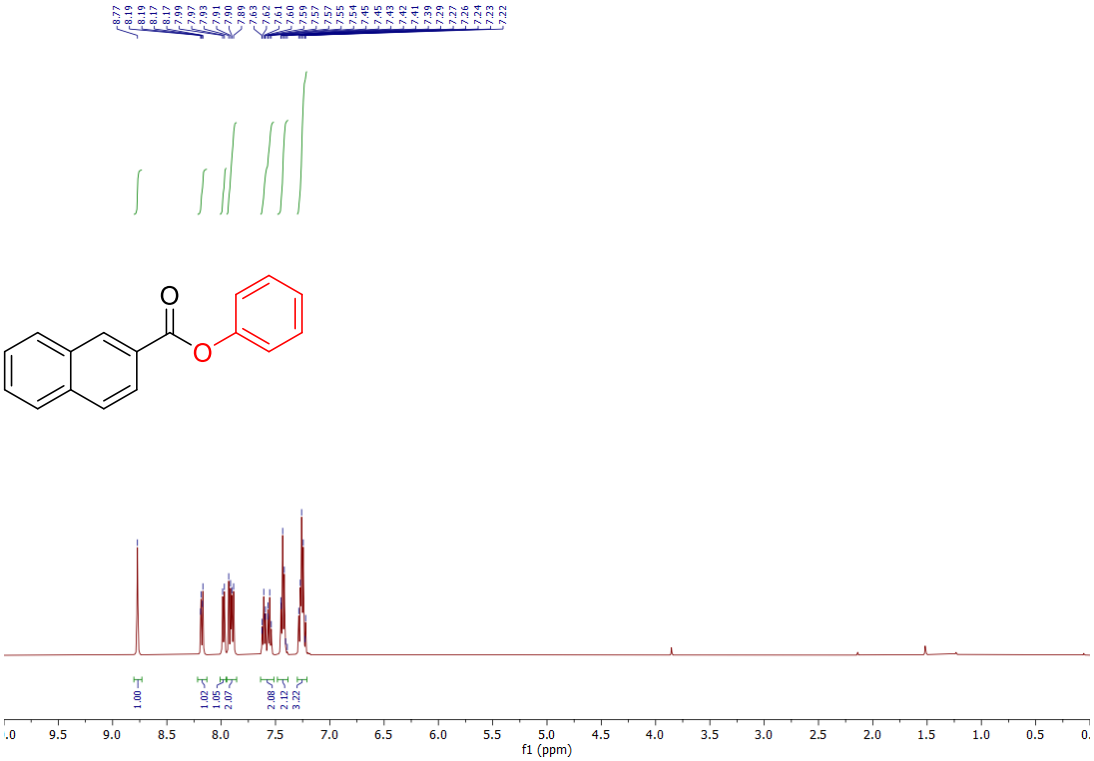
^{1}H NMR spectrum of **4k** (500 MHz, $CDCl_3$)



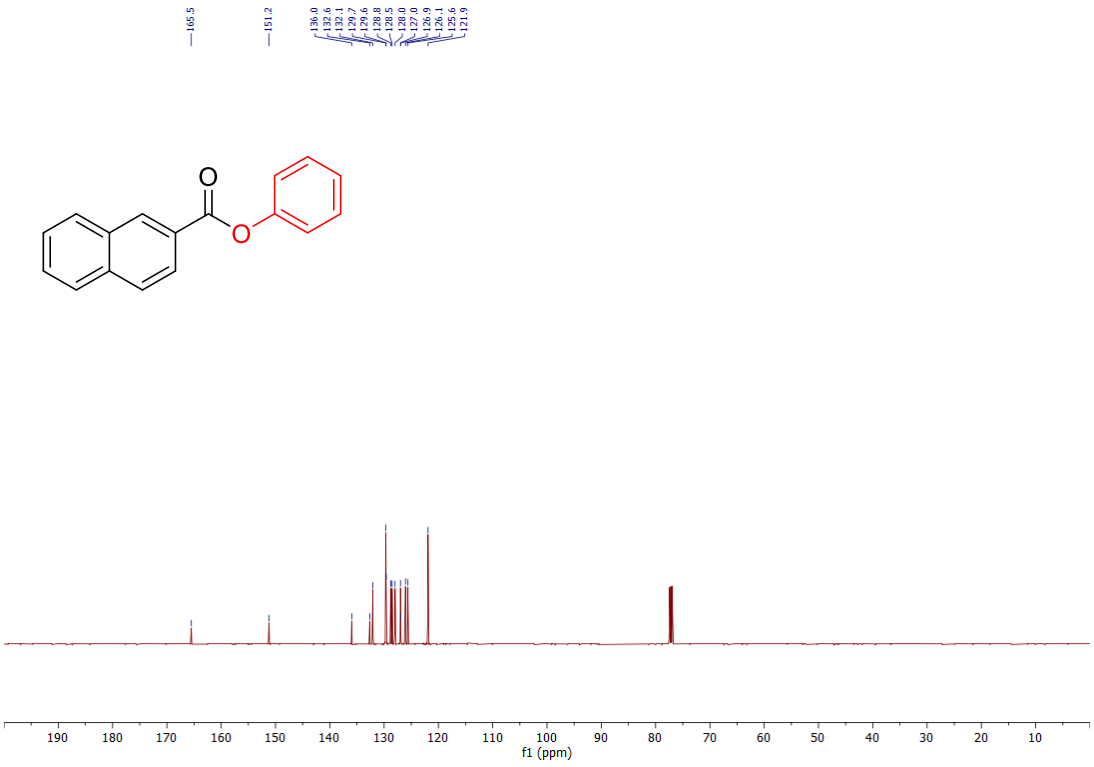
^{13}C NMR spectrum of **4k** (126 MHz, $CDCl_3$)



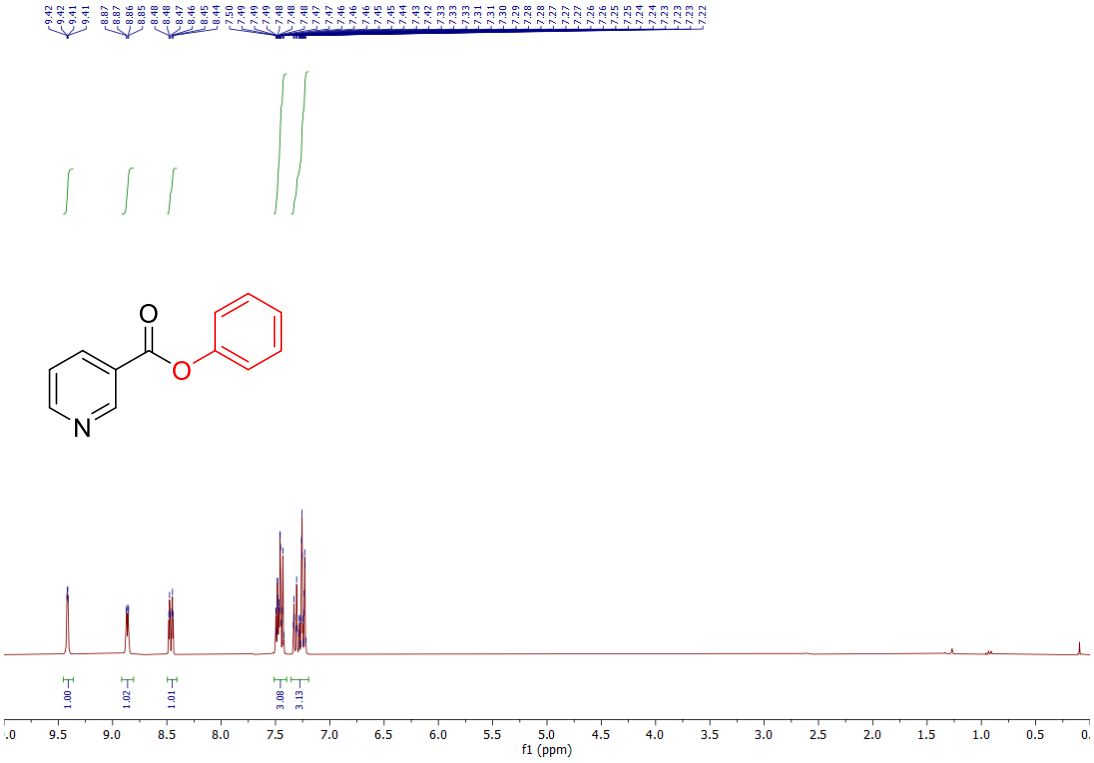
^{1}H NMR spectrum of **4l** (500 MHz, $CDCl_3$)



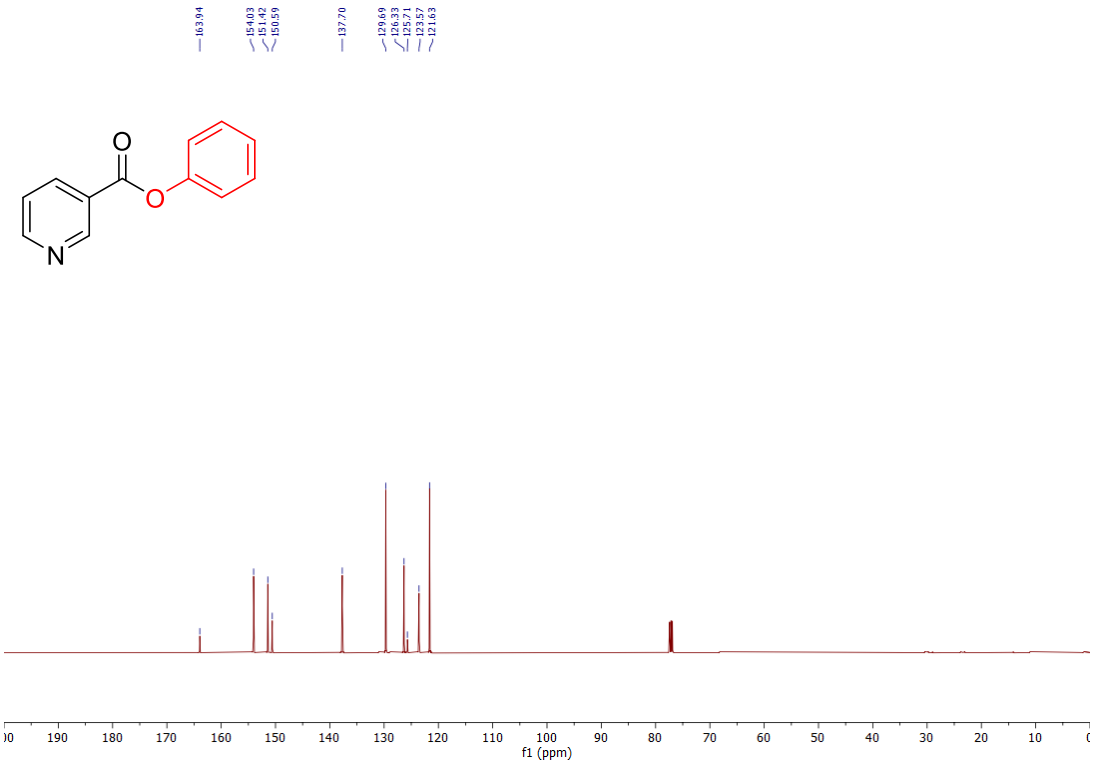
^{13}C NMR spectrum of **4l** (126 MHz, $CDCl_3$)



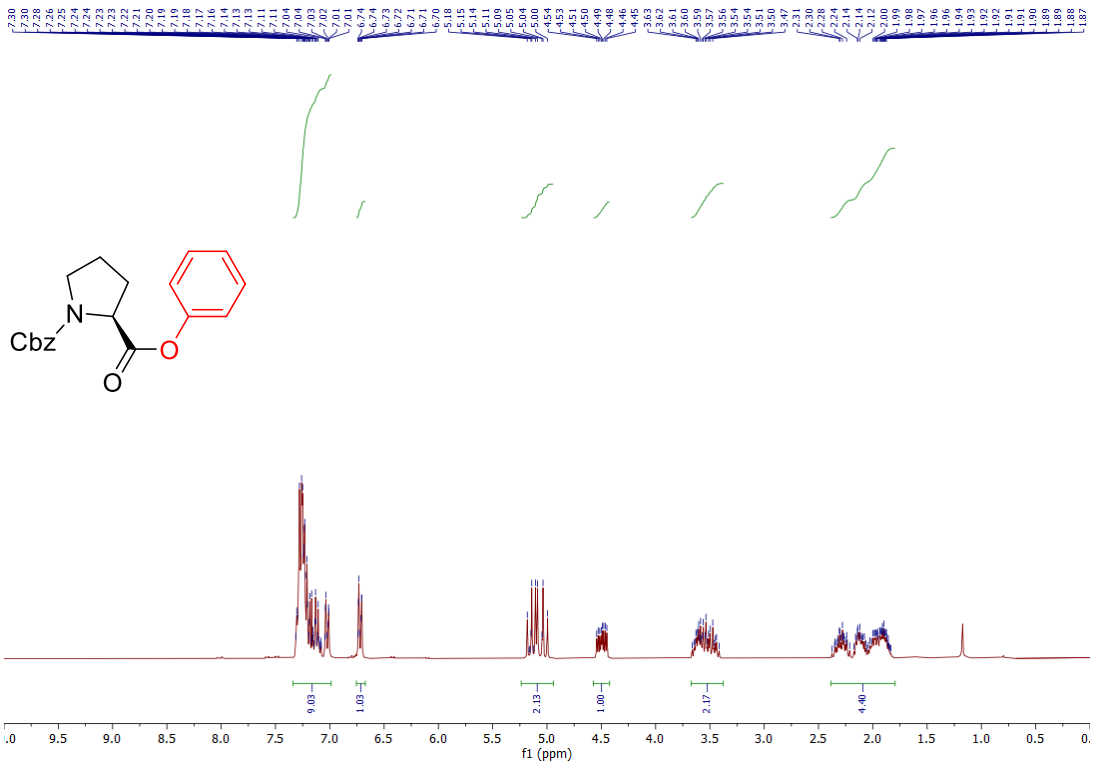
^{1}H NMR spectrum of **4m** (300 MHz, $CDCl_3$)



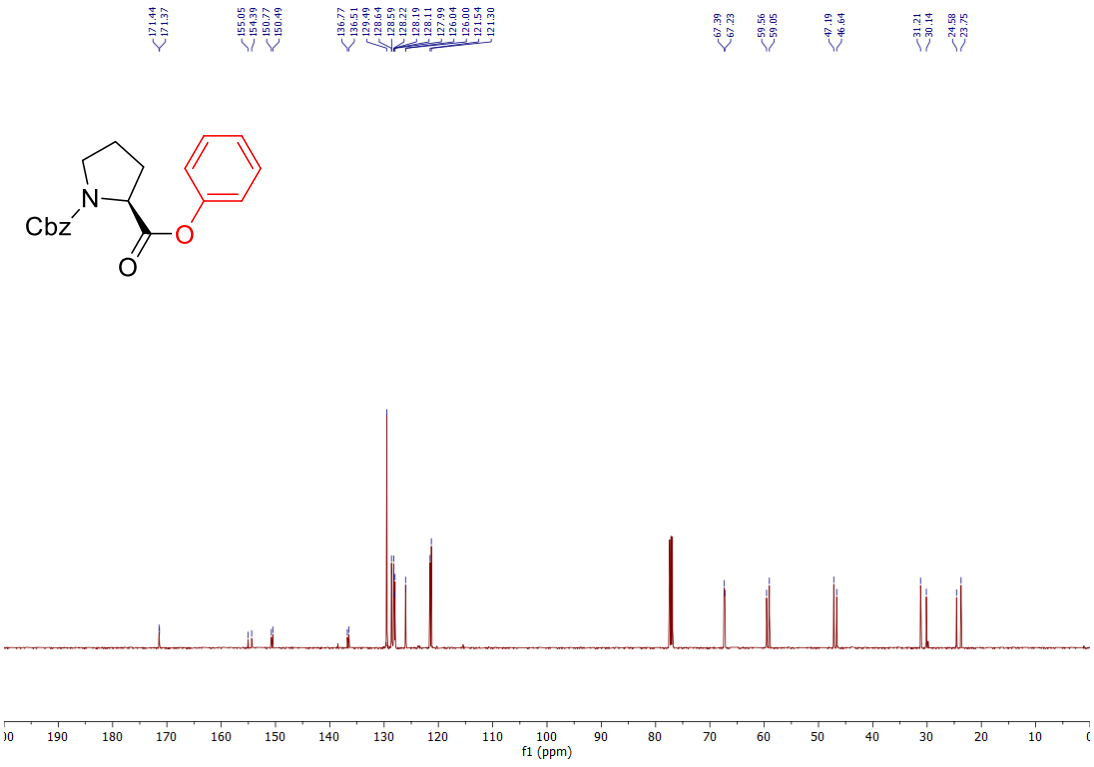
^{13}C NMR spectrum of **4m** (126 MHz, $CDCl_3$)



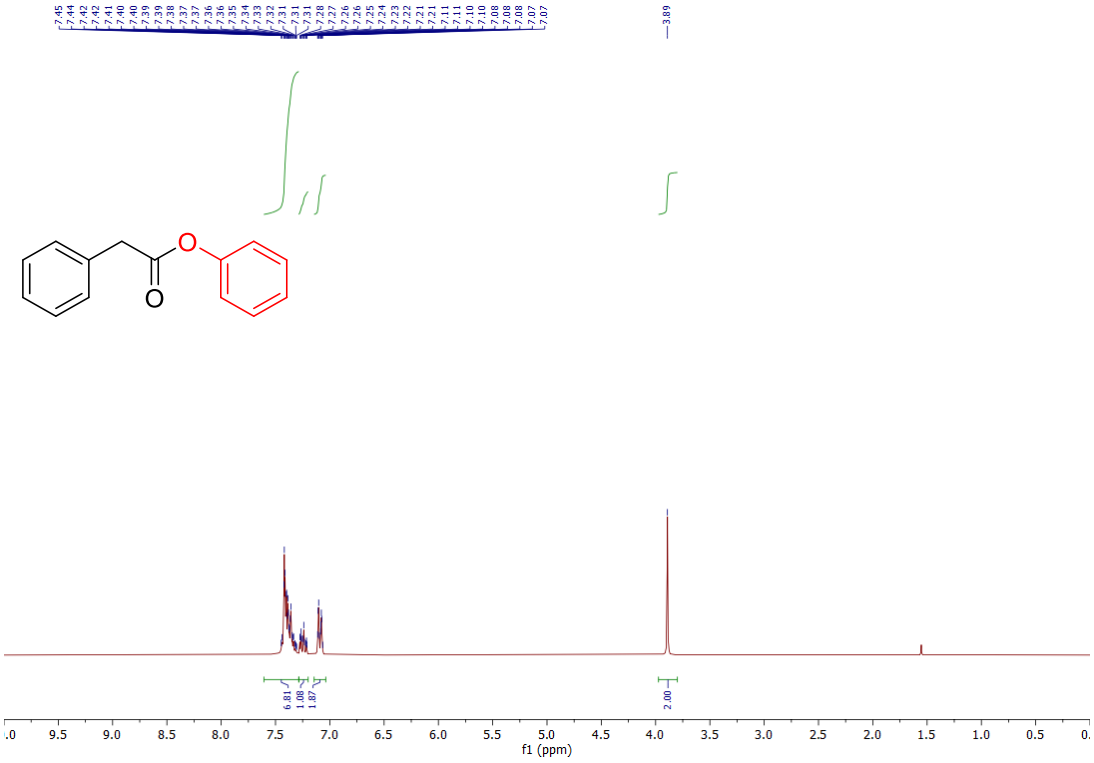
^{1}H NMR spectrum of **4p** (300 MHz, $CDCl_3$)



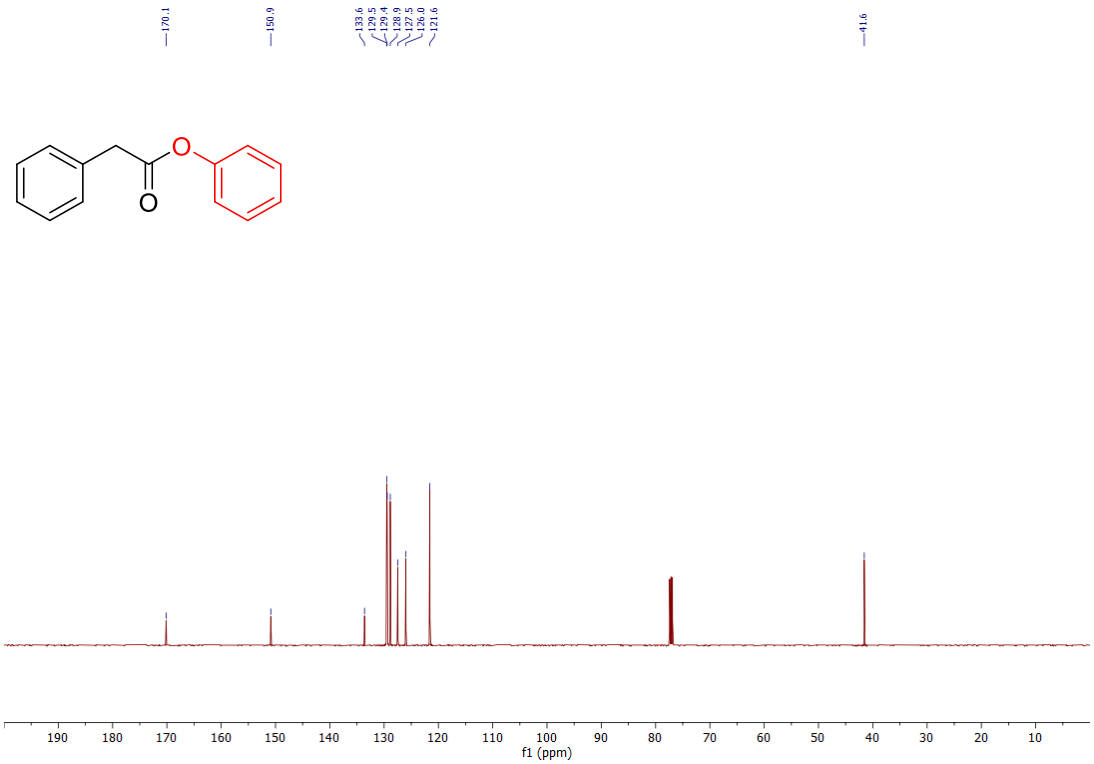
^{13}C NMR spectrum of **4p** (126 MHz, $CDCl_3$)



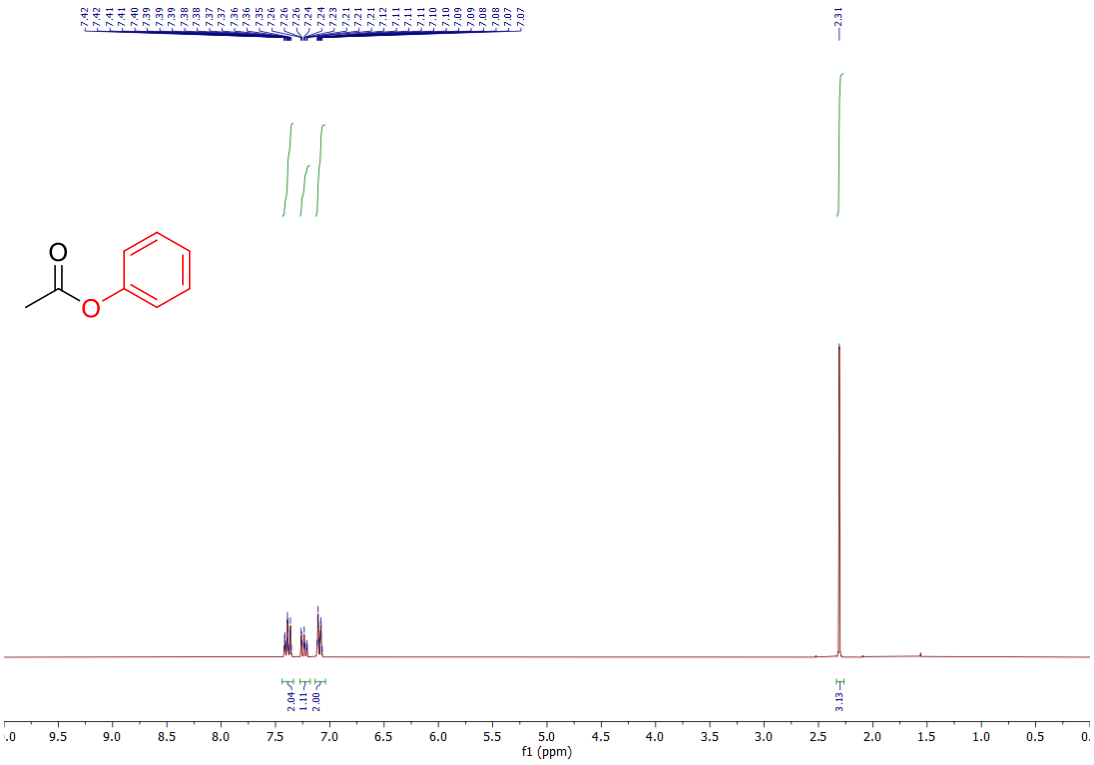
^{1}H NMR spectrum of **4q** (300 MHz, $CDCl_3$)



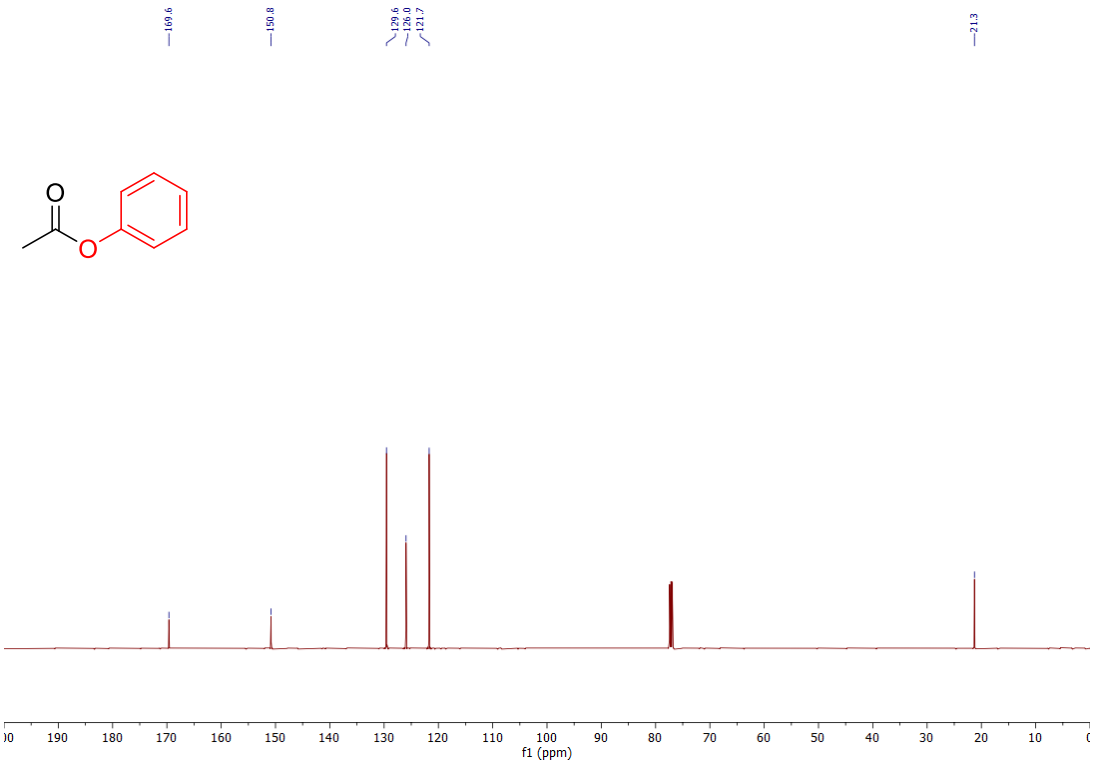
^{13}C NMR spectrum of **4q** (126 MHz, $CDCl_3$)



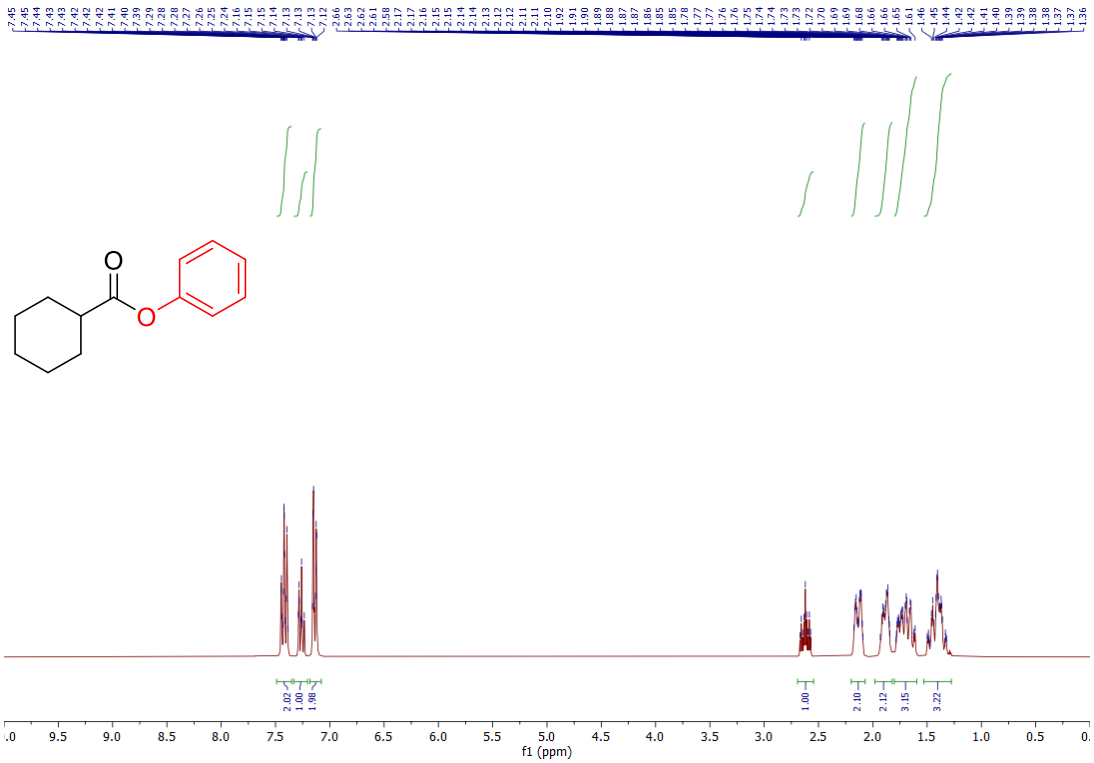
^{1}H NMR spectrum of **4r** (300 MHz, $CDCl_3$)



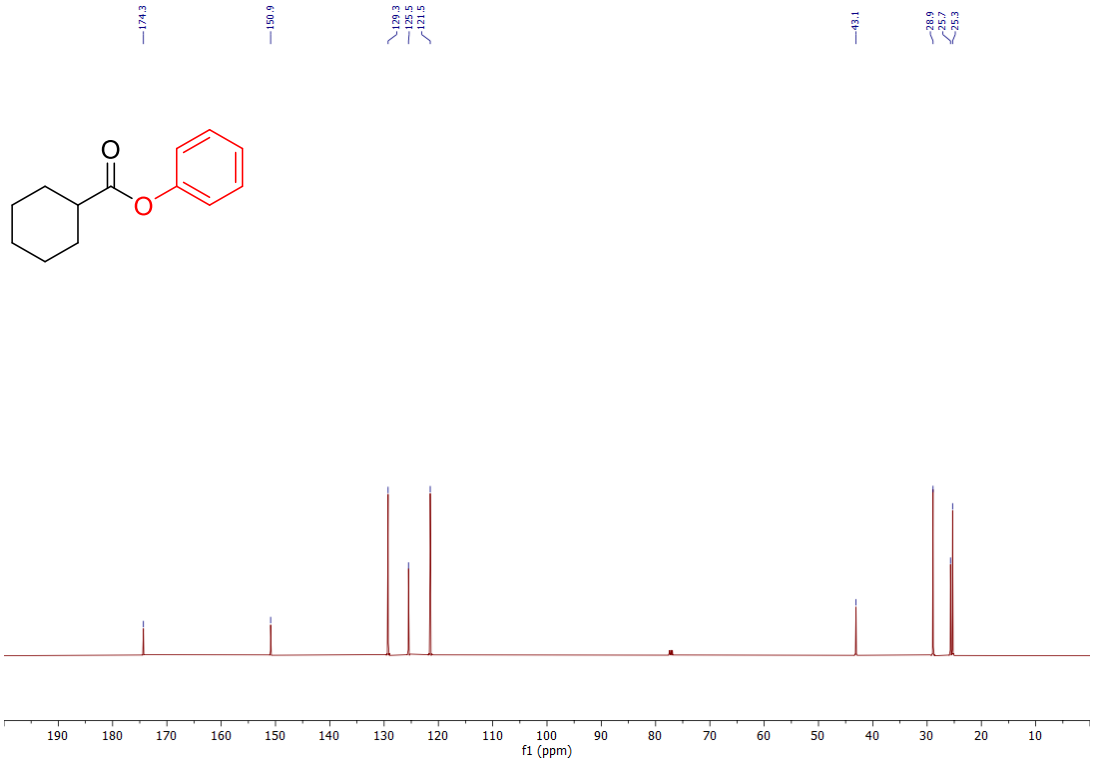
^{13}C NMR spectrum of **4r** (126 MHz, $CDCl_3$)



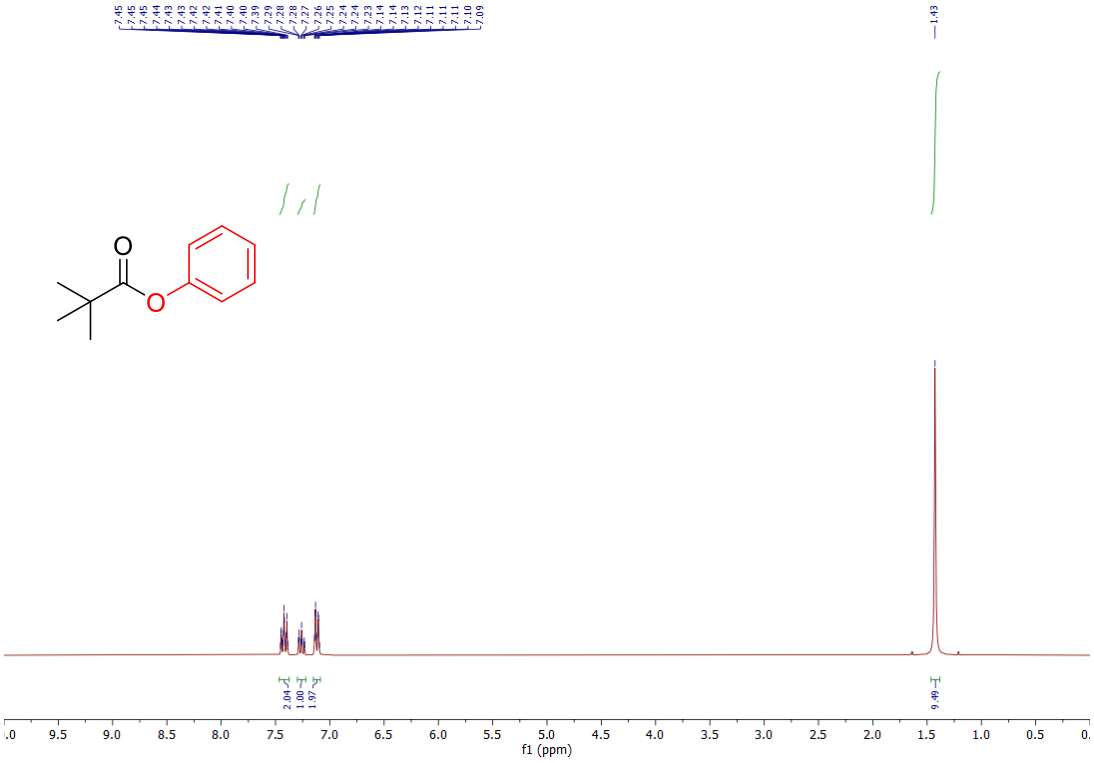
^{1}H NMR spectrum of **4s** (300 MHz, $CDCl_3$)



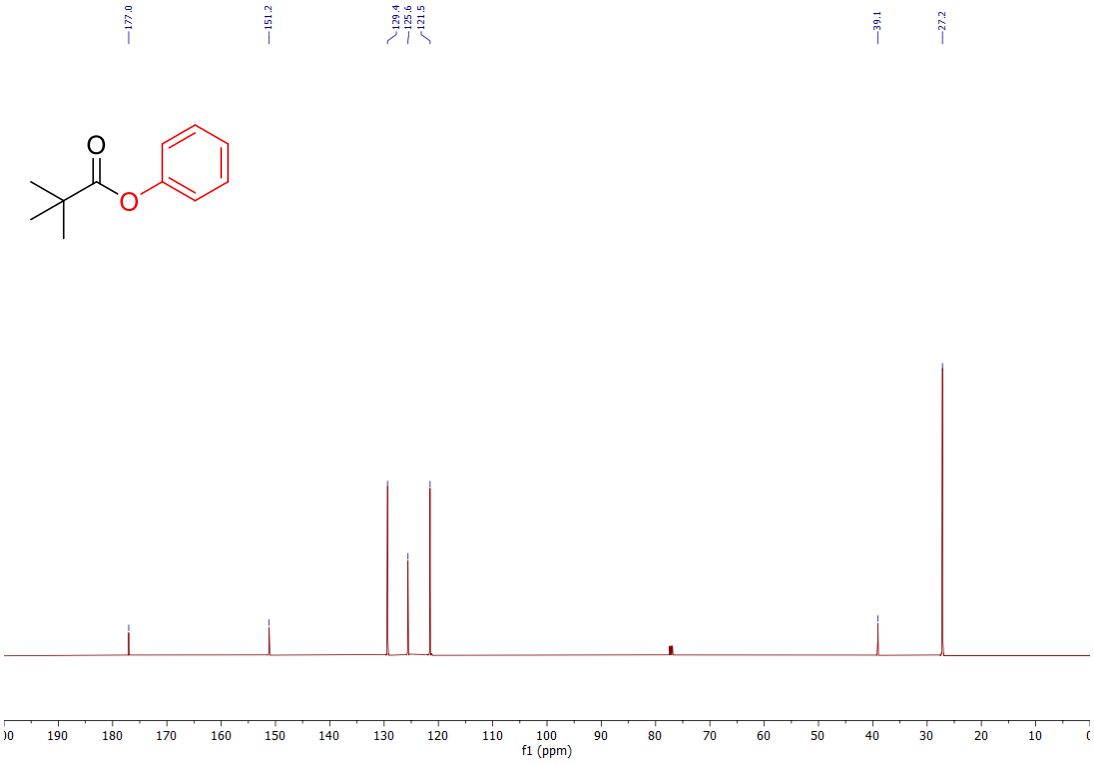
^{13}C NMR spectrum of **4s** (126 MHz, $CDCl_3$)



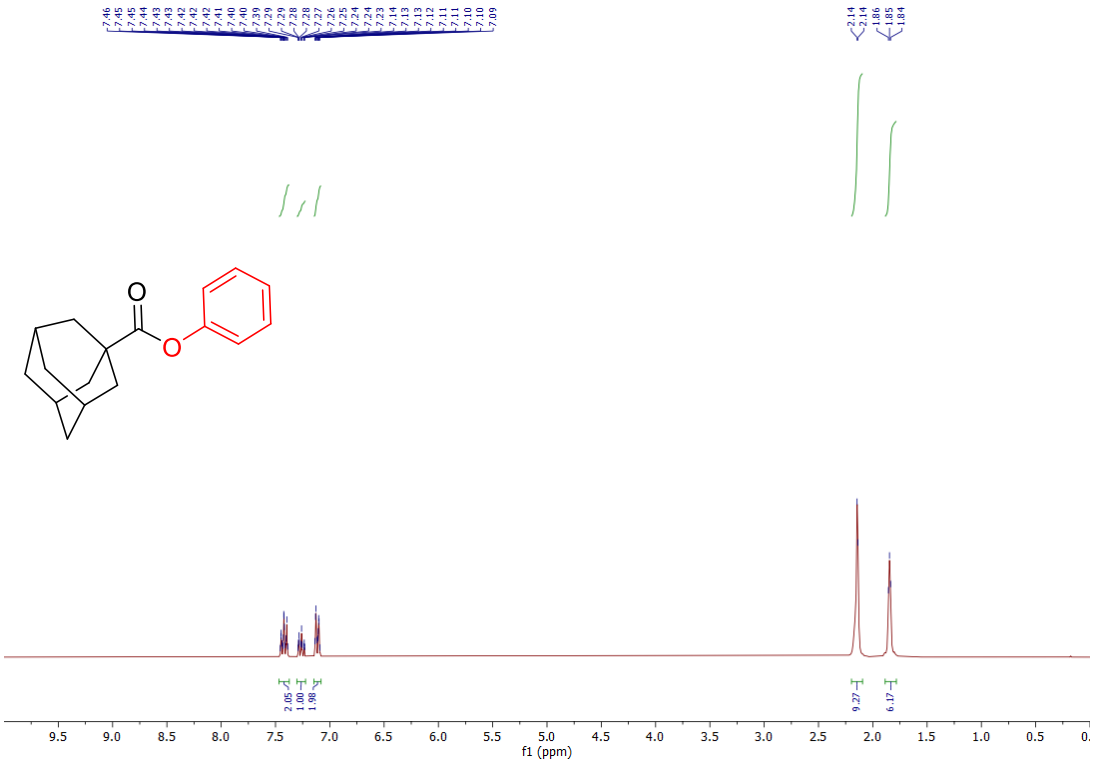
^{1}H NMR spectrum of **4t** (300 MHz, $CDCl_3$)



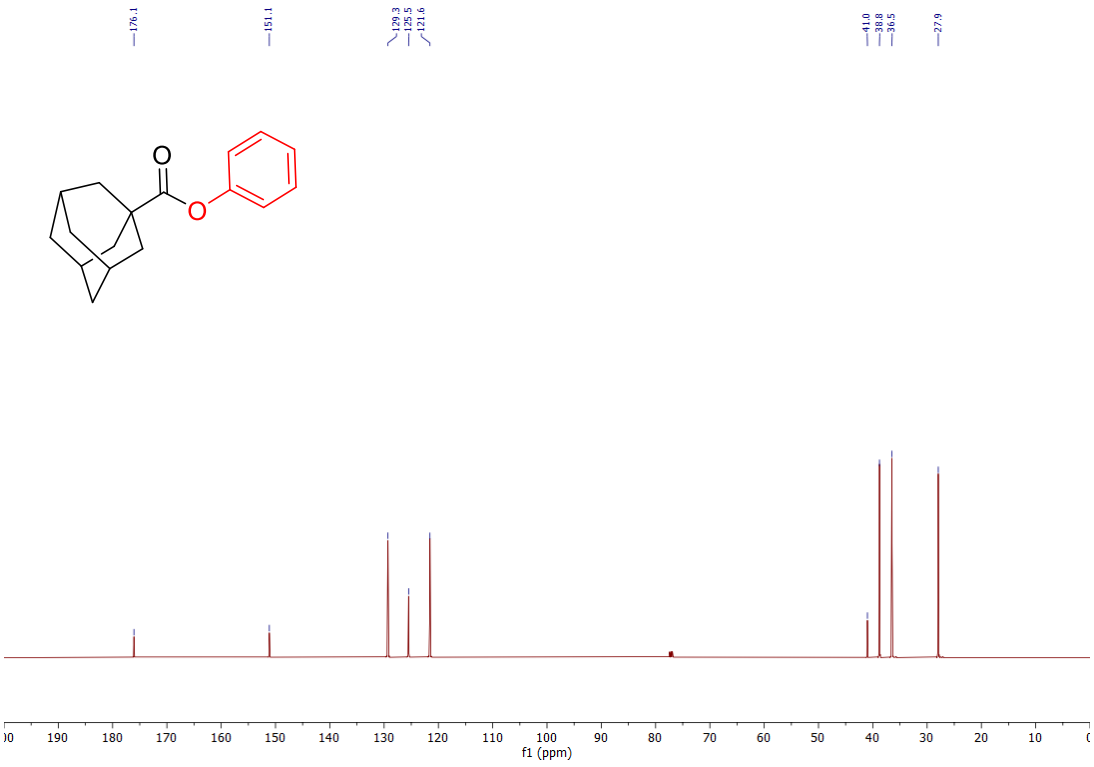
^{13}C NMR spectrum of **4t** (126 MHz, $CDCl_3$)



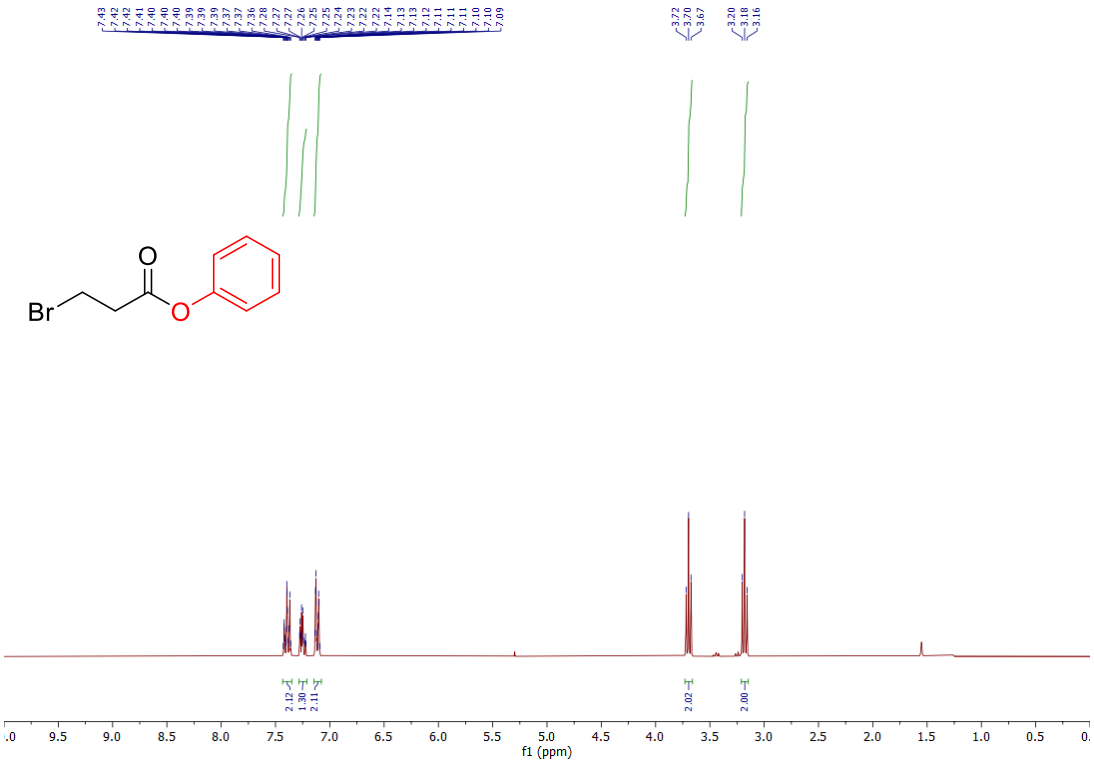
^{1}H NMR spectrum of **4u** (300 MHz, $CDCl_3$)



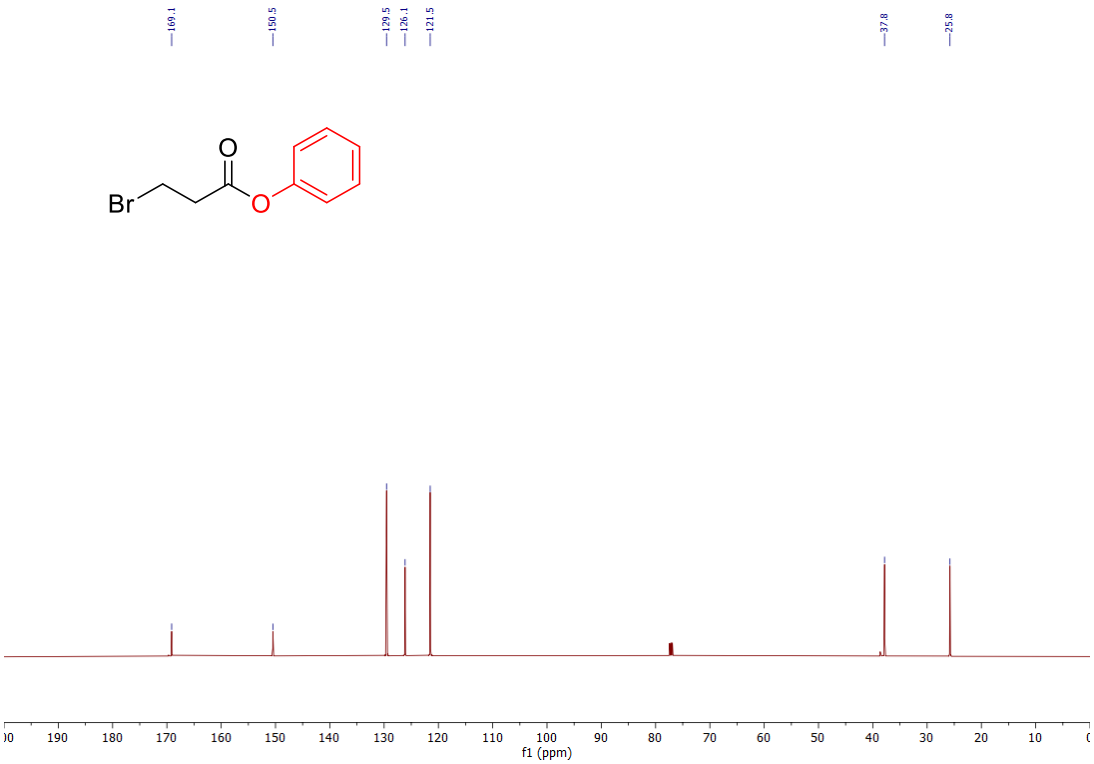
^{13}C NMR spectrum of **4u** (126 MHz, $CDCl_3$)



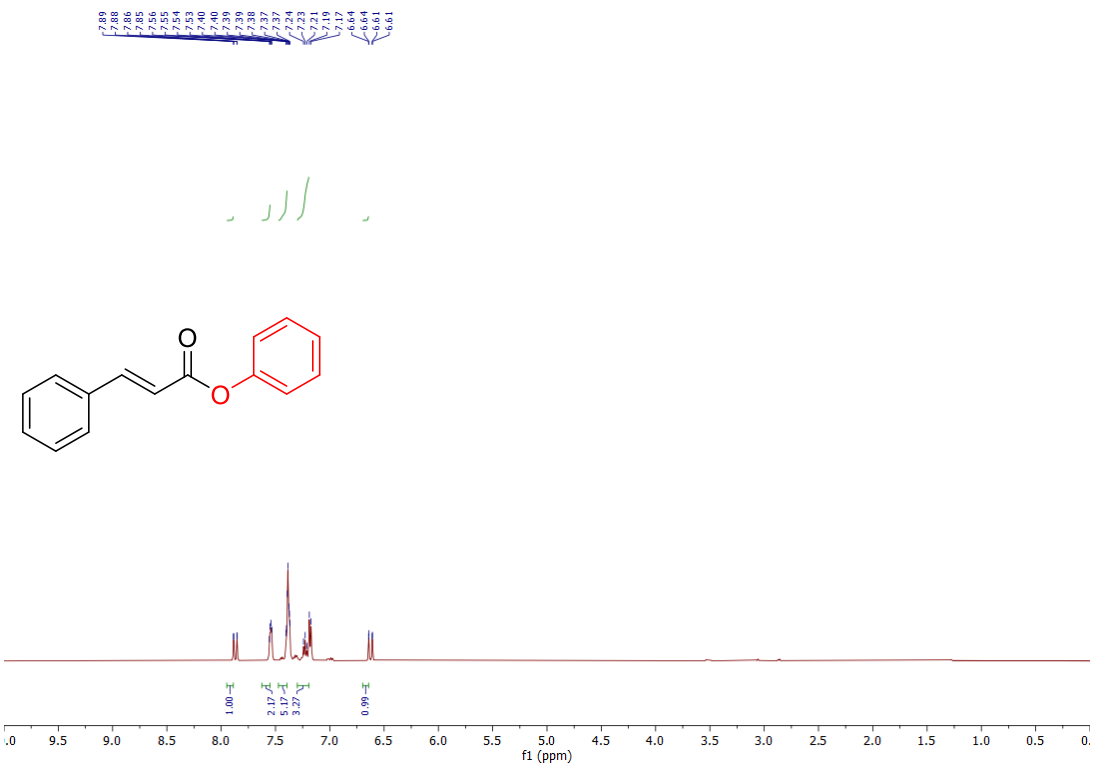
^{1}H NMR spectrum of **4v** (300 MHz, $CDCl_3$)



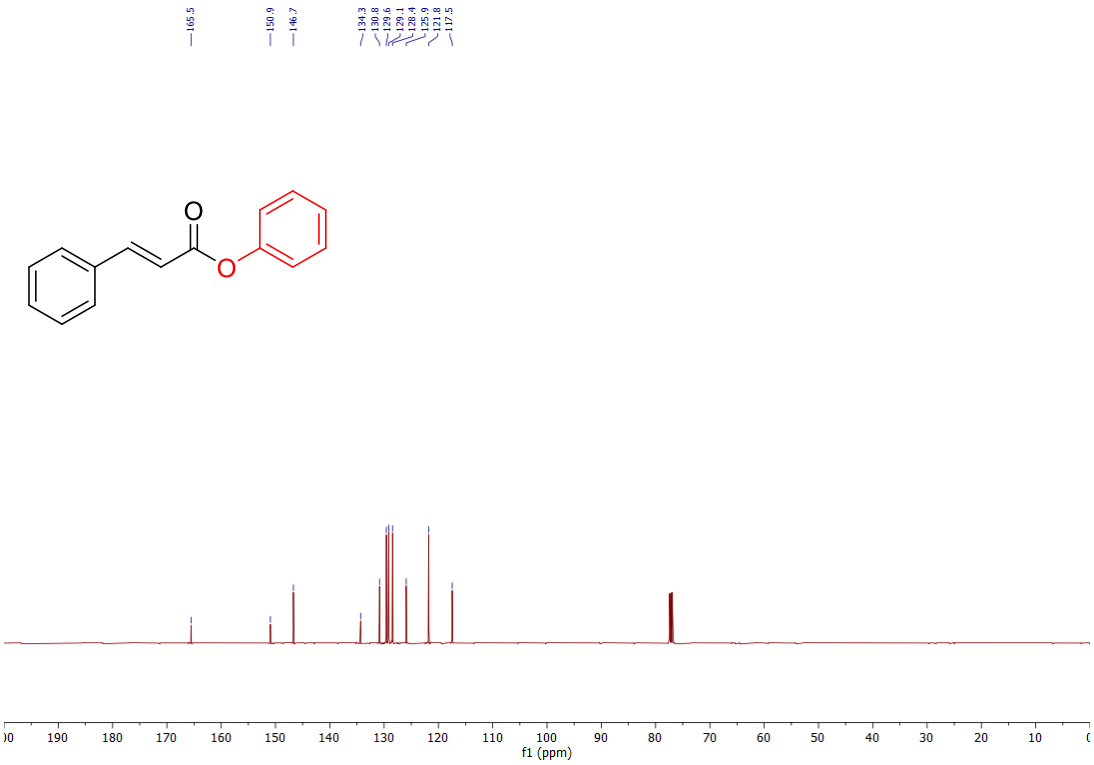
^{13}C NMR spectrum of **4v** (126 MHz, $CDCl_3$)



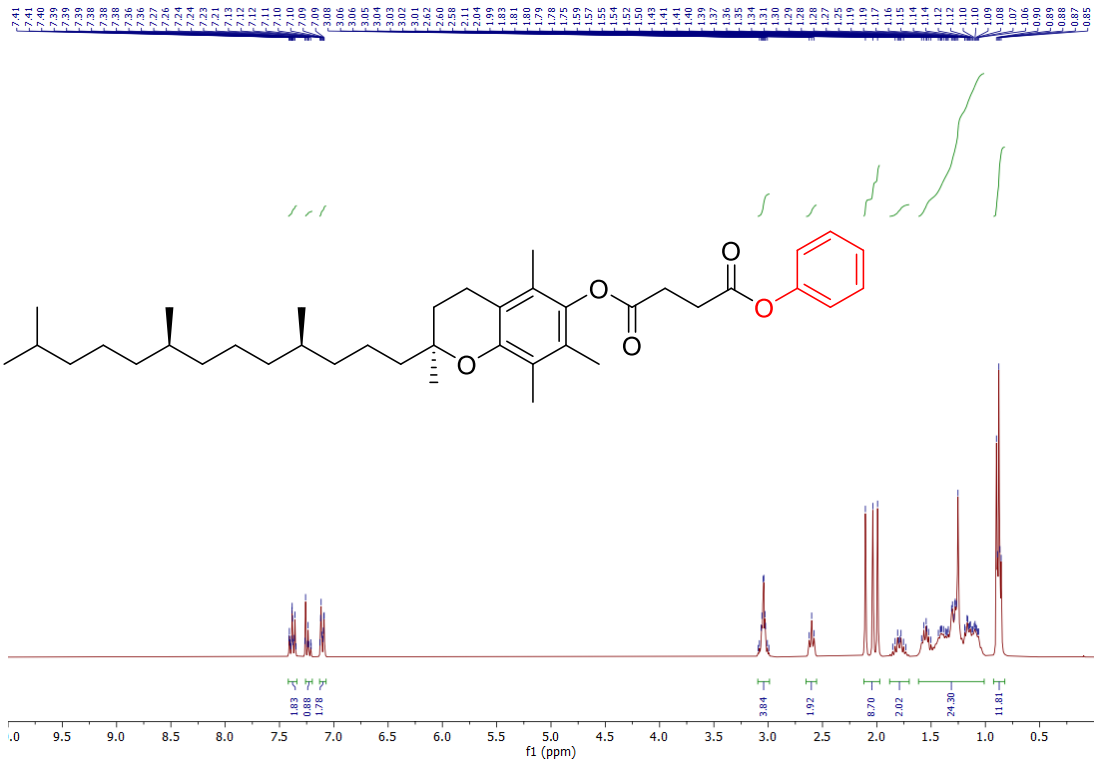
^{1}H NMR spectrum of **4w** (500 MHz, $CDCl_3$)



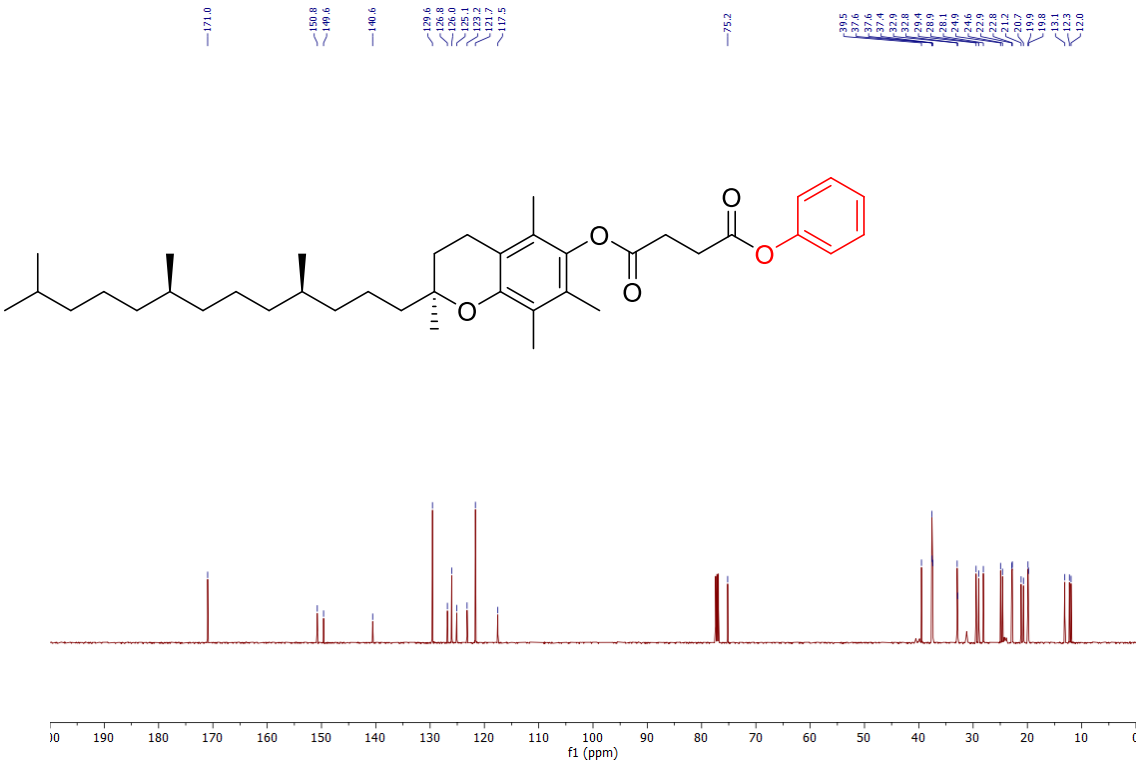
^{13}C NMR spectrum of **4w** (126 MHz, $CDCl_3$)



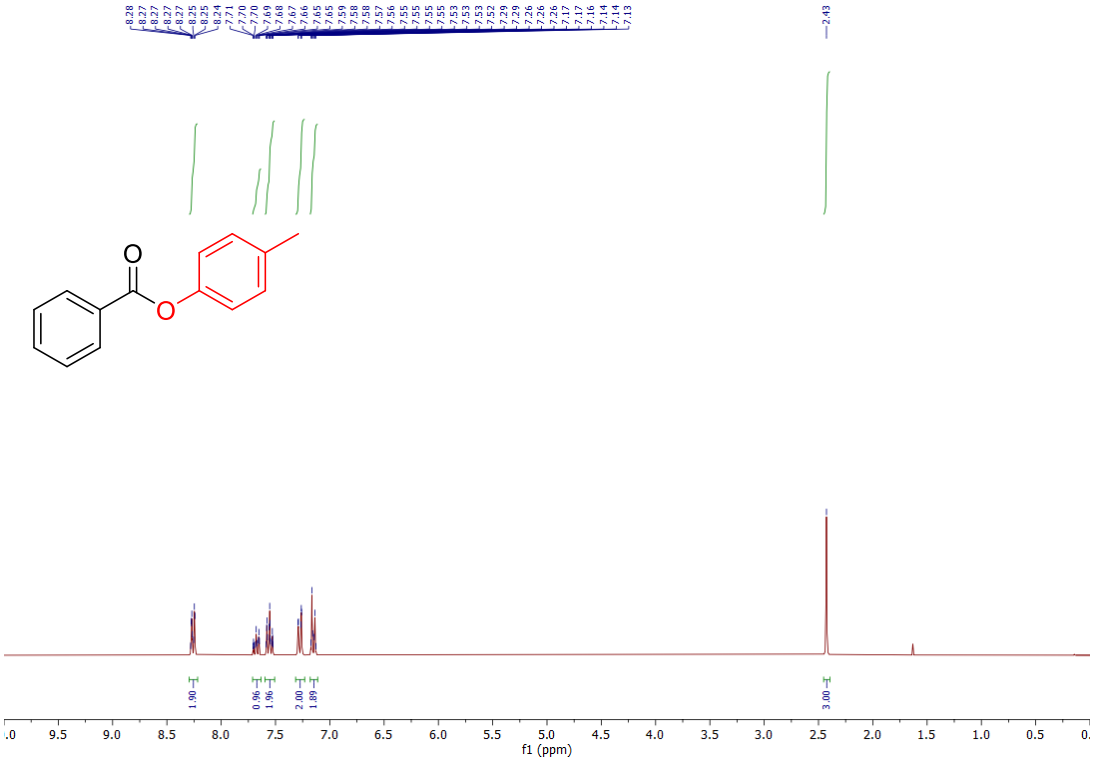
^{1}H NMR spectrum of **4x** (300 MHz, $CDCl_3$)



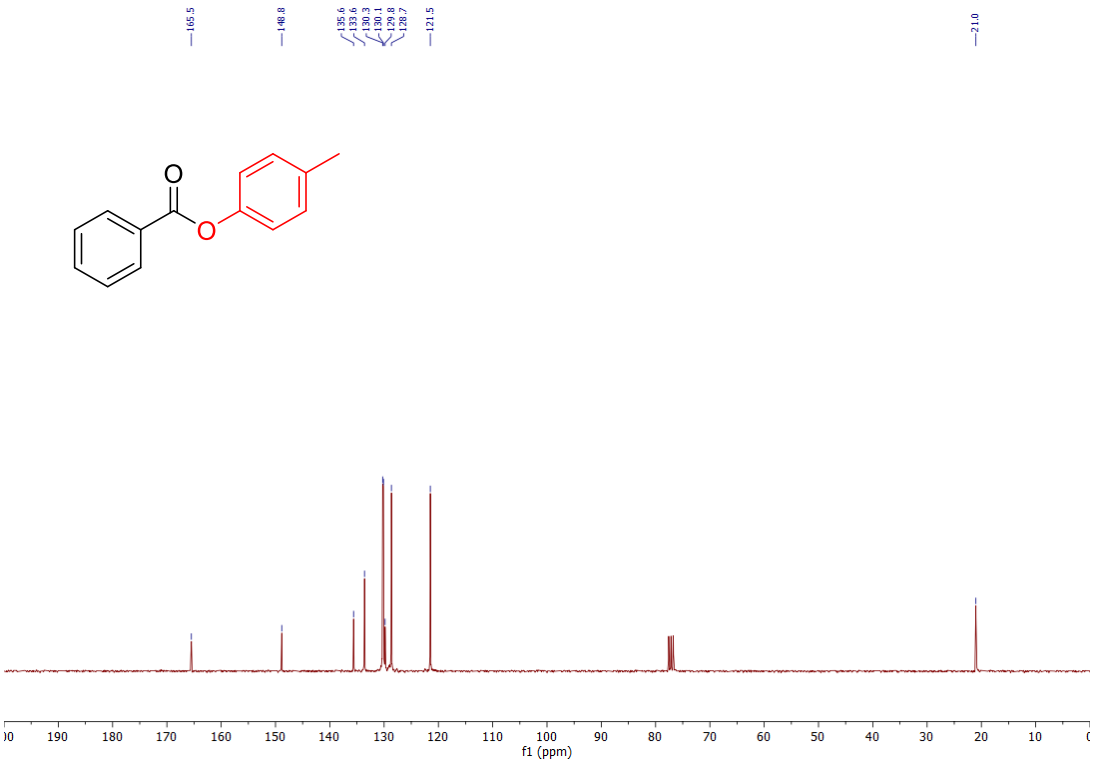
^{13}C NMR spectrum of **4x** (126 MHz, $CDCl_3$)



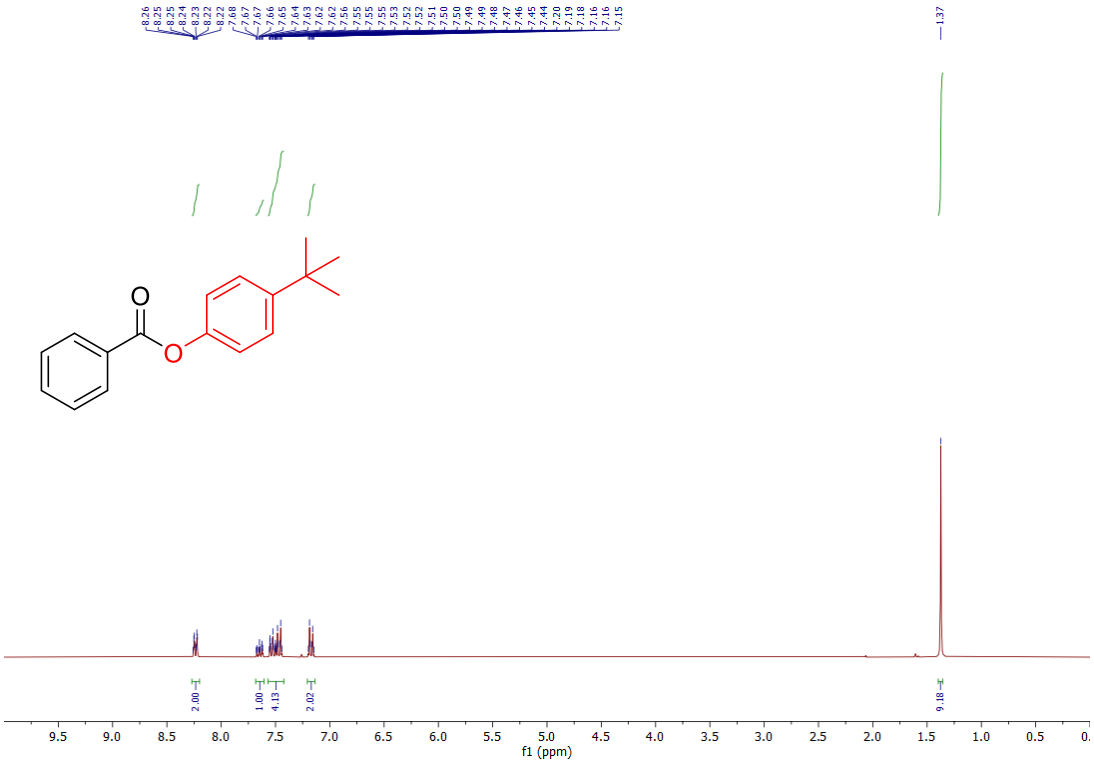
^{1}H NMR spectrum of **4y** (300 MHz, $CDCl_3$)



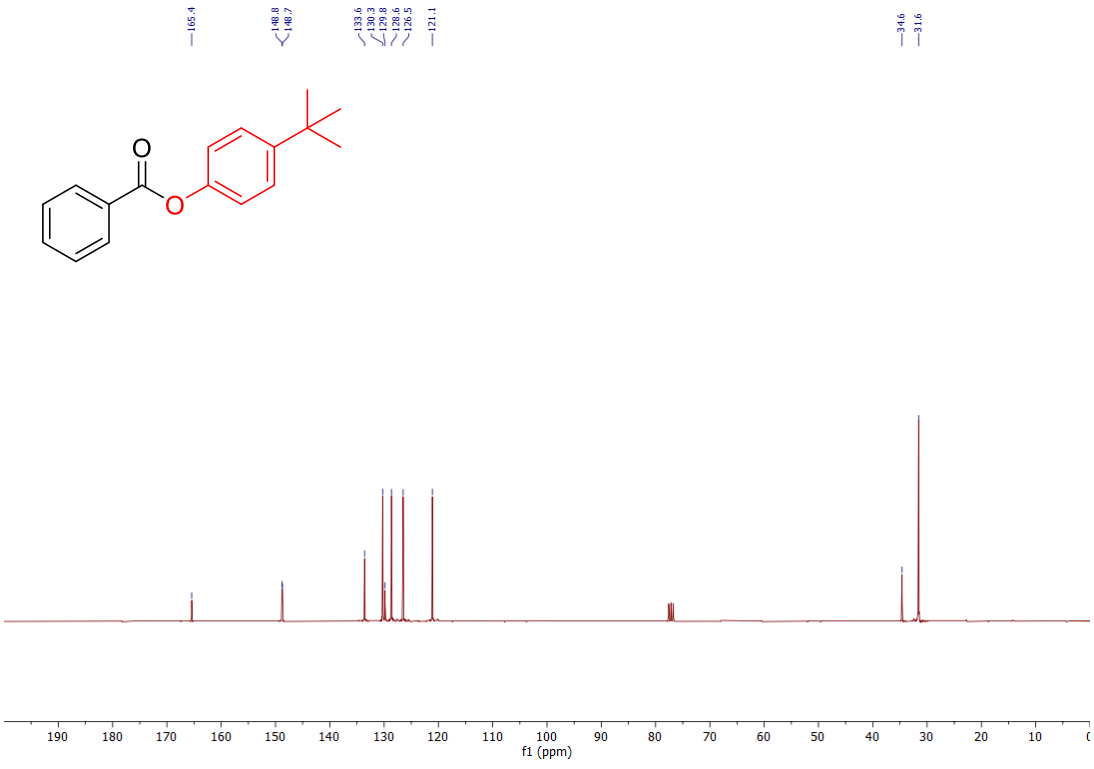
^{13}C NMR spectrum of **4y** (75 MHz, $CDCl_3$)



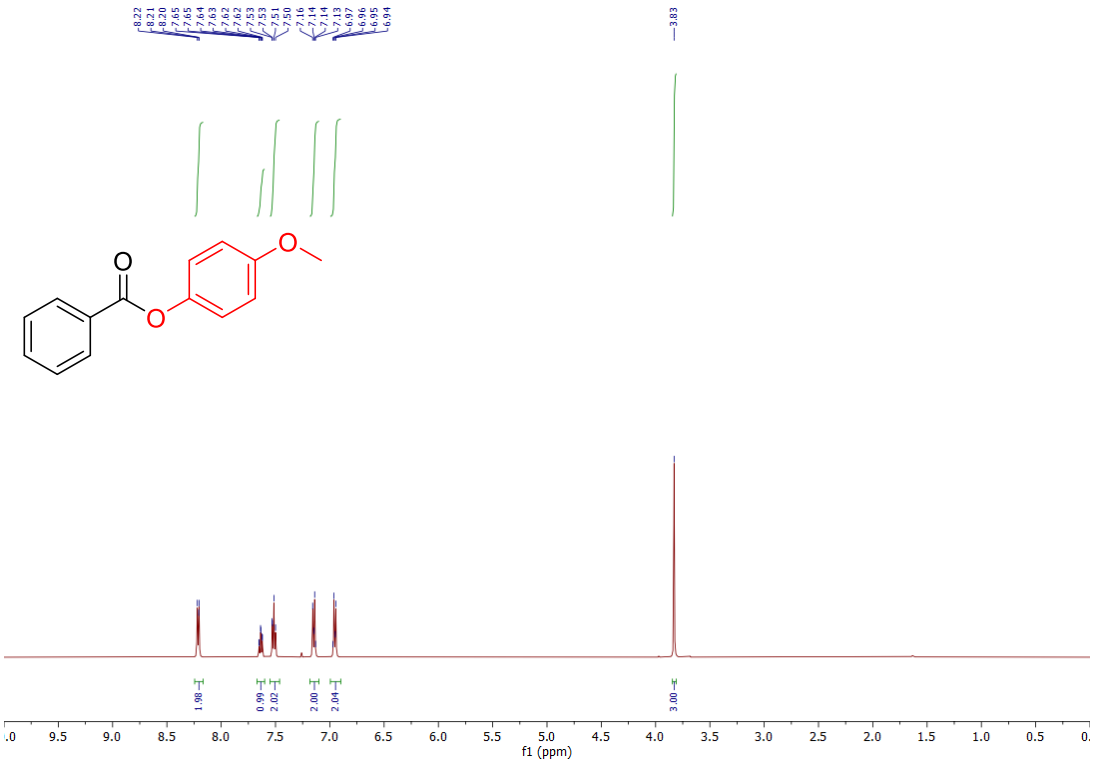
^{1}H NMR spectrum of **4z** (300 MHz, $CDCl_3$)



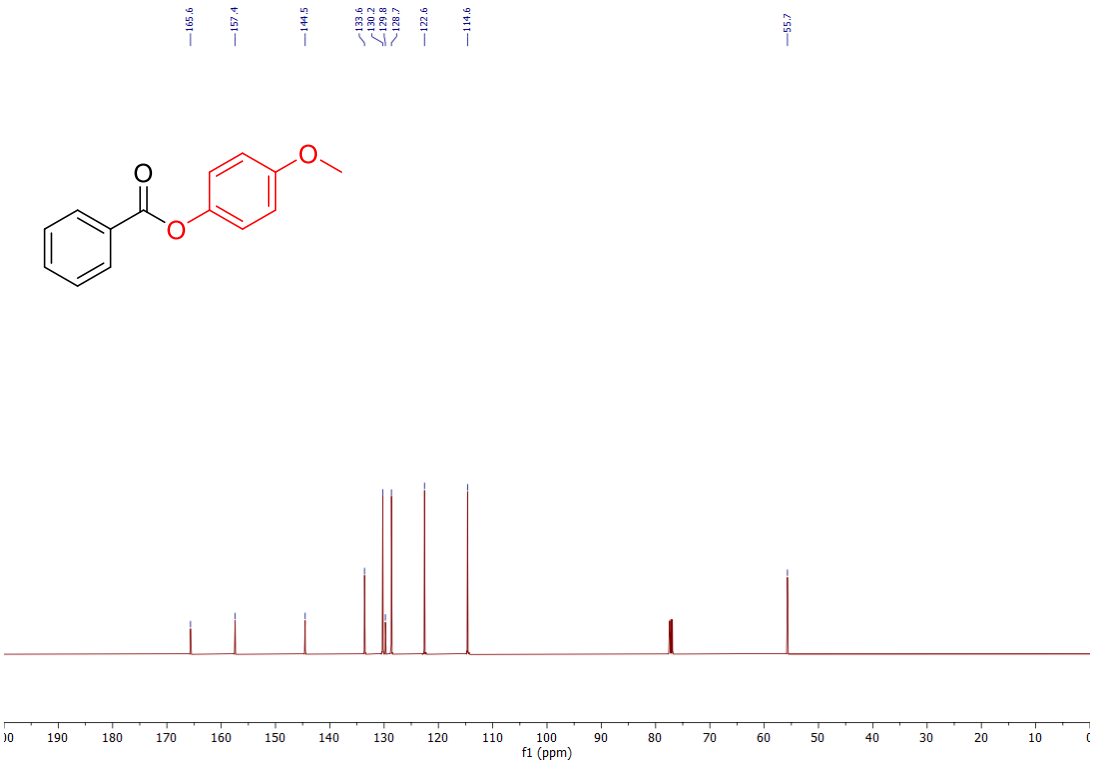
^{13}C NMR spectrum of **4z** (75 MHz, $CDCl_3$)



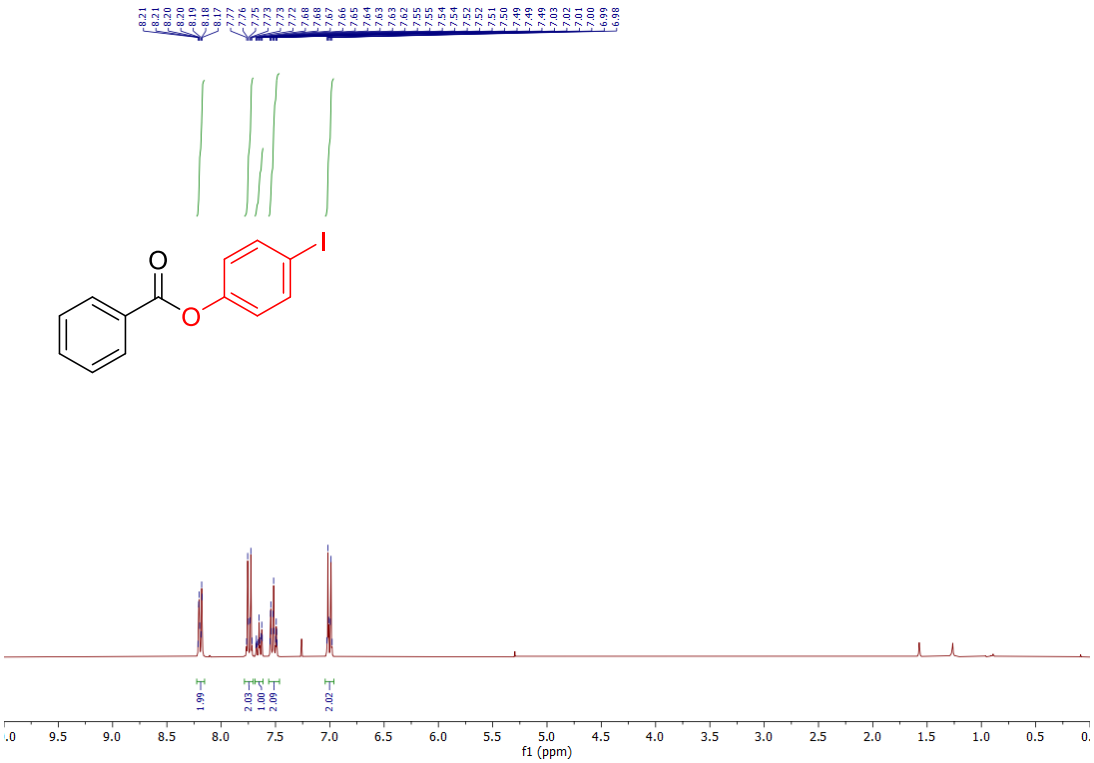
^{1}H NMR spectrum of **4aa** (500 MHz, $CDCl_3$)



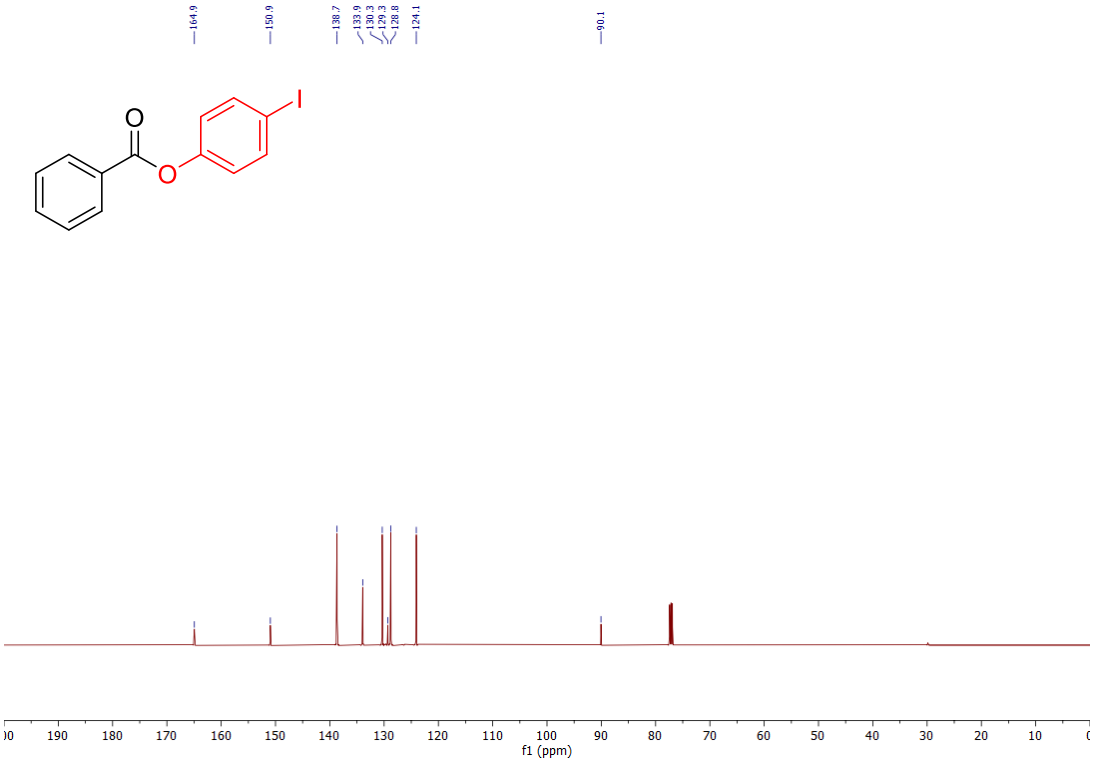
^{13}C NMR spectrum of **4aa** (126 MHz, $CDCl_3$)



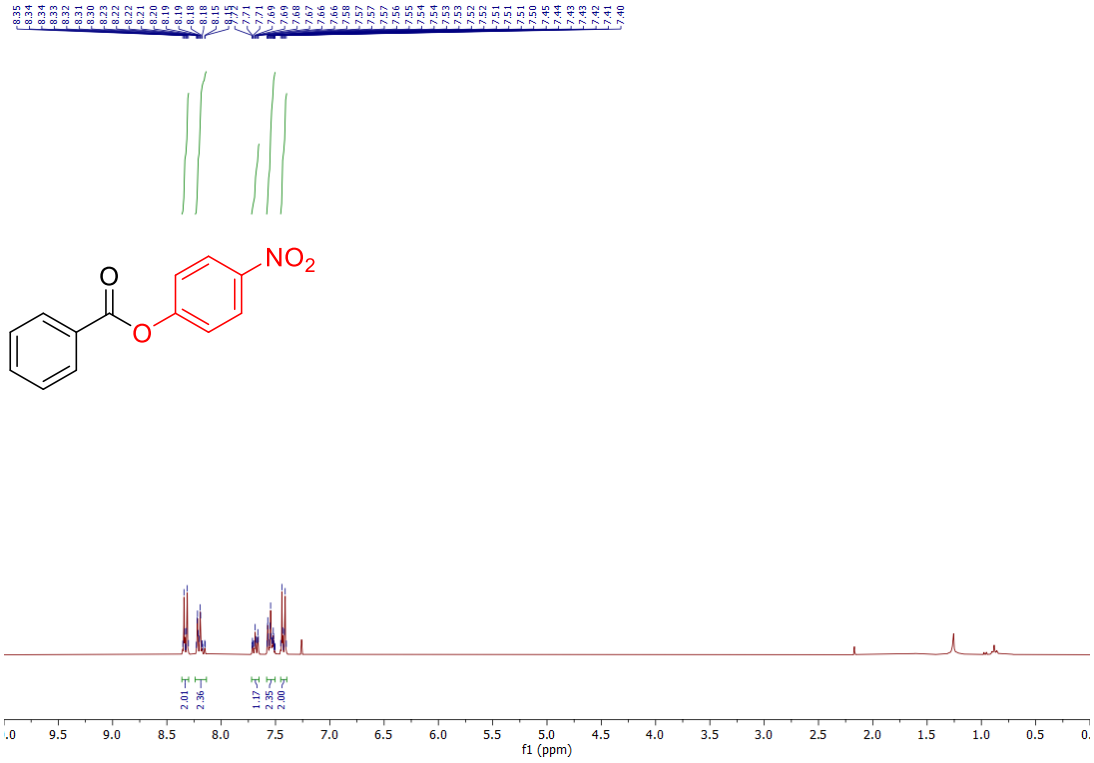
^{1}H NMR spectrum of **4ab** (300 MHz, $CDCl_3$)



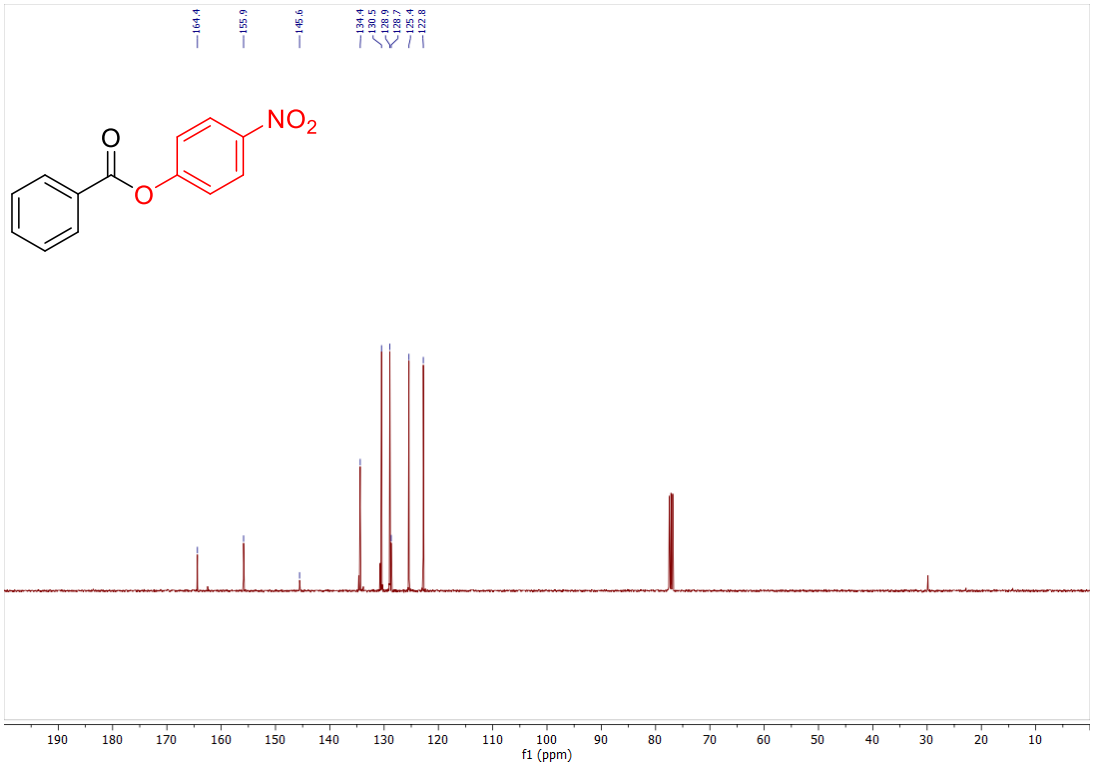
^{13}C NMR spectrum of **4ab** (126 MHz, $CDCl_3$)



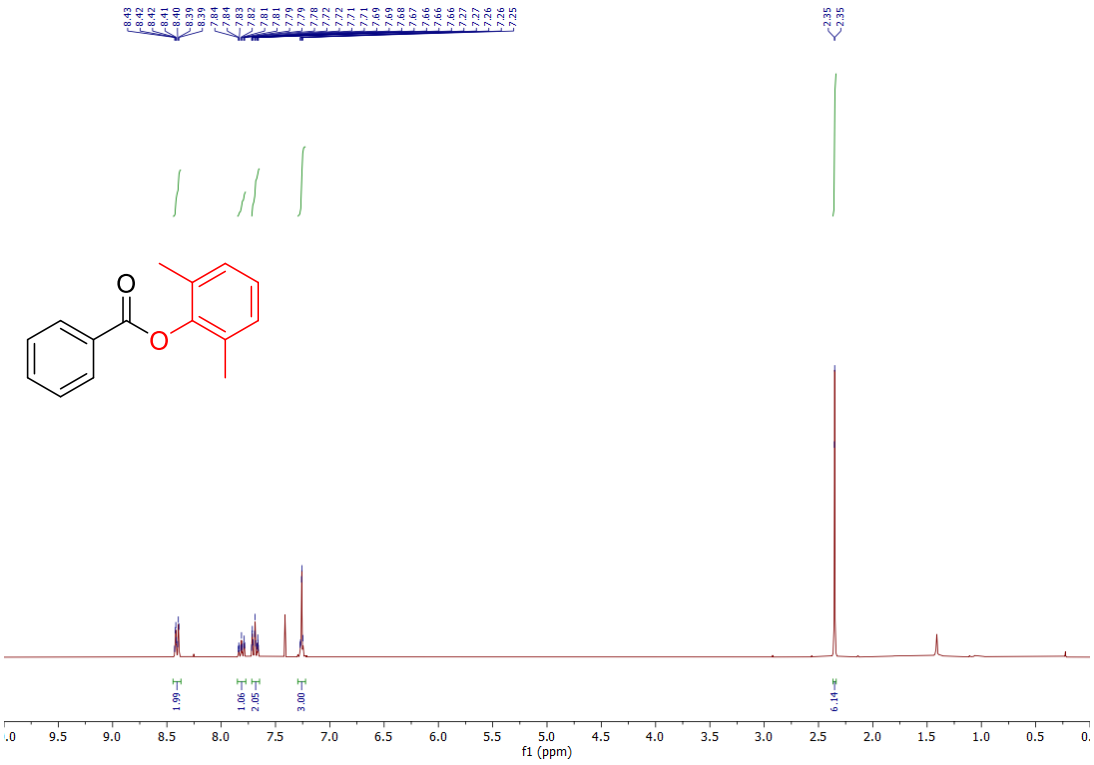
^{1}H NMR spectrum of **4ac** (300 MHz, $CDCl_3$)



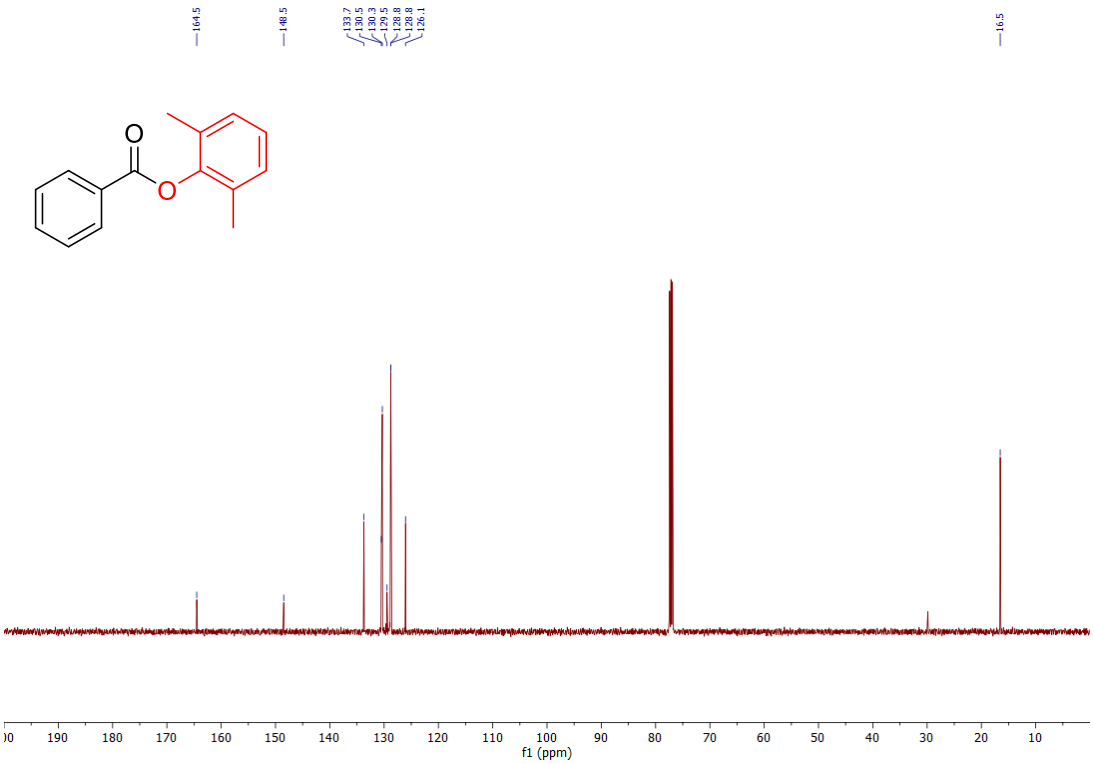
^{13}C NMR spectrum of **4ac** (101 MHz, $CDCl_3$)



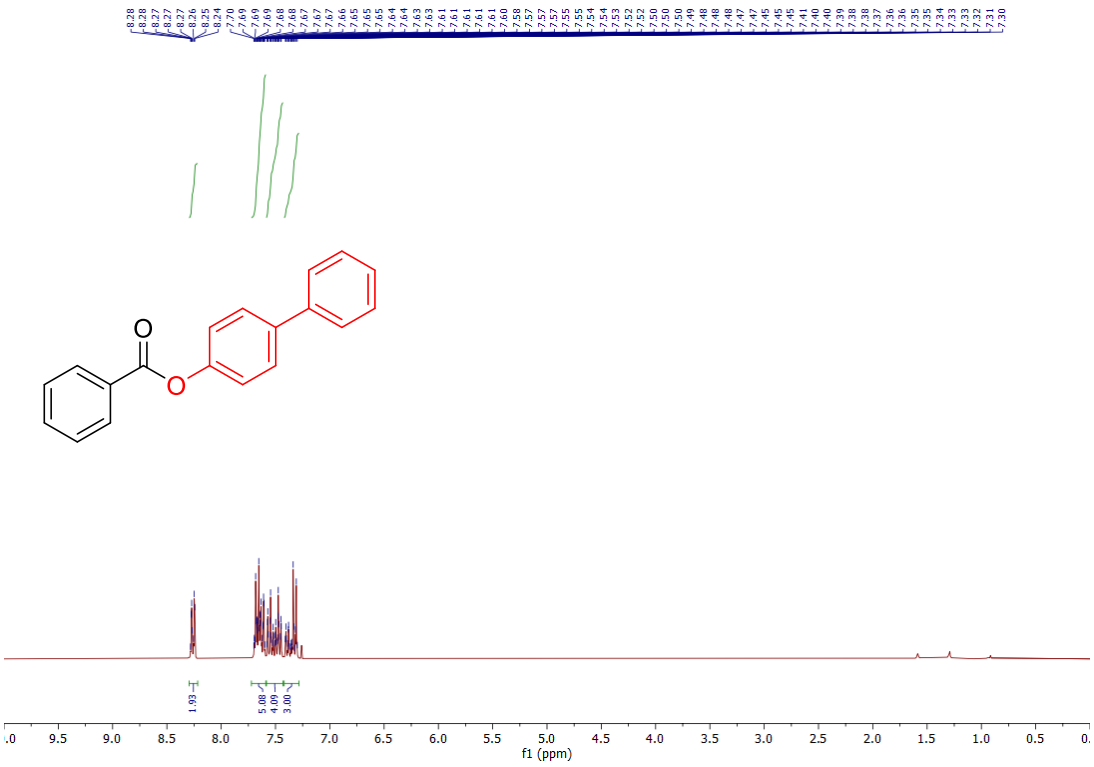
^{1}H NMR spectrum of **4ad** (300 MHz, $CDCl_3$)



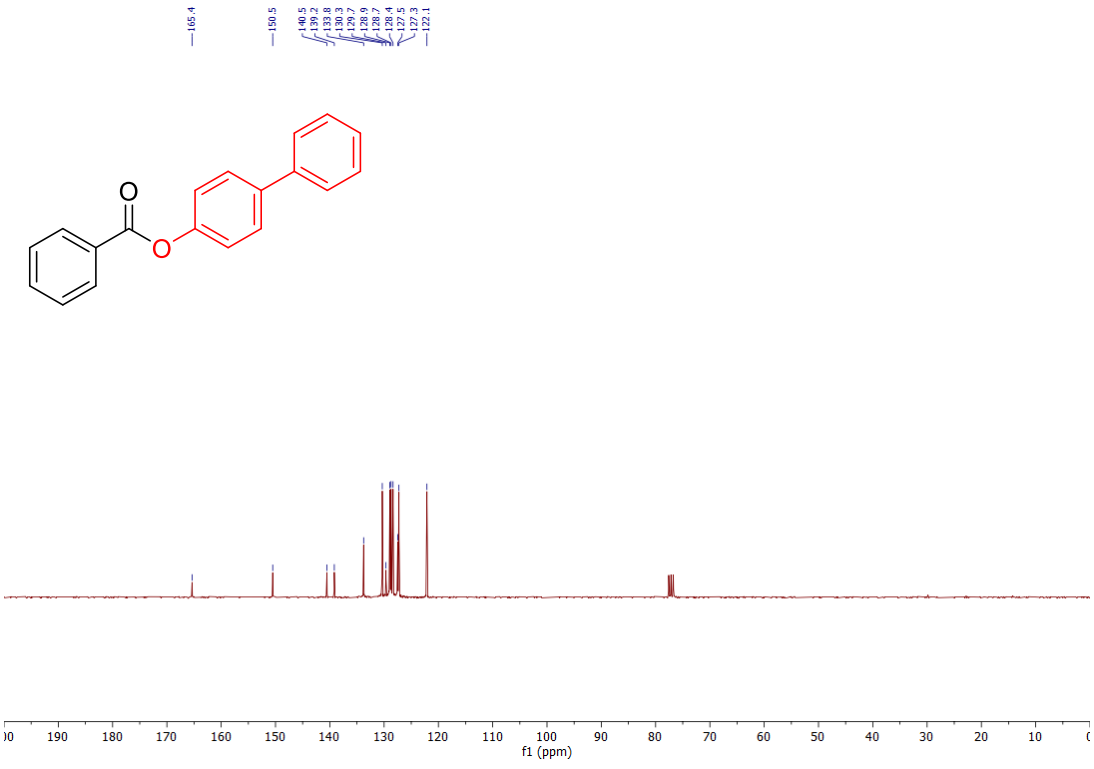
^{13}C NMR spectrum of **4ad** (126 MHz, $CDCl_3$)



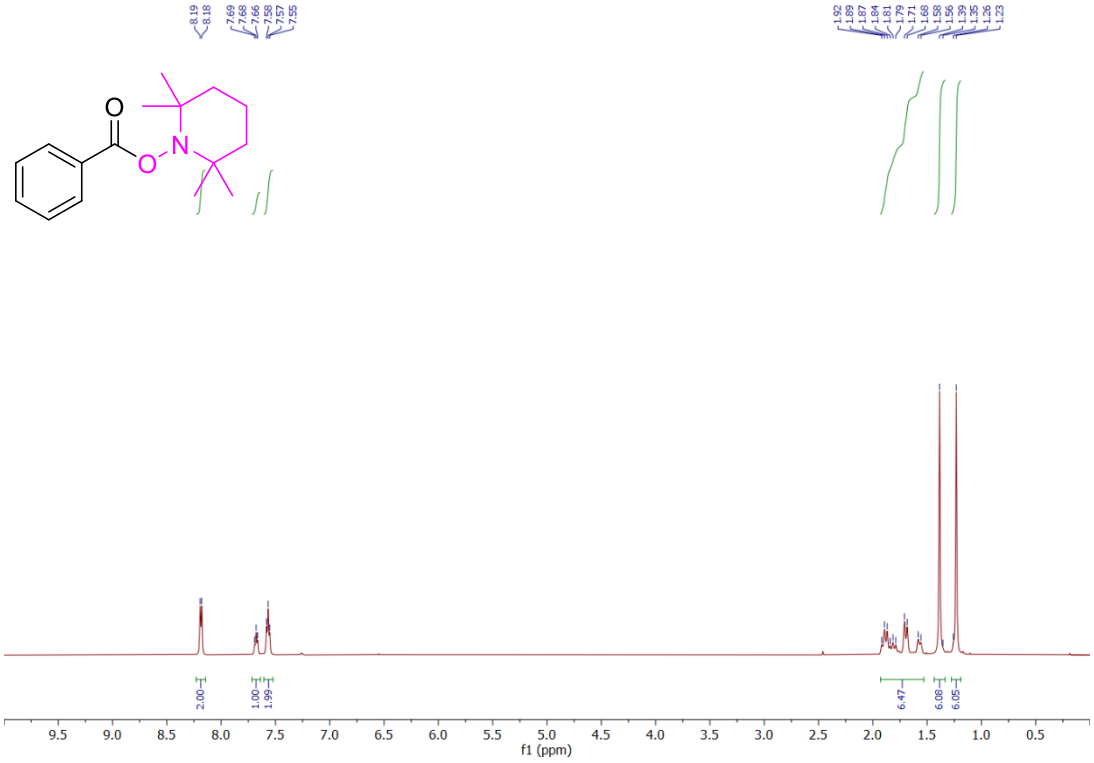
^{1}H NMR spectrum of **4ae** (300 MHz, $CDCl_3$)



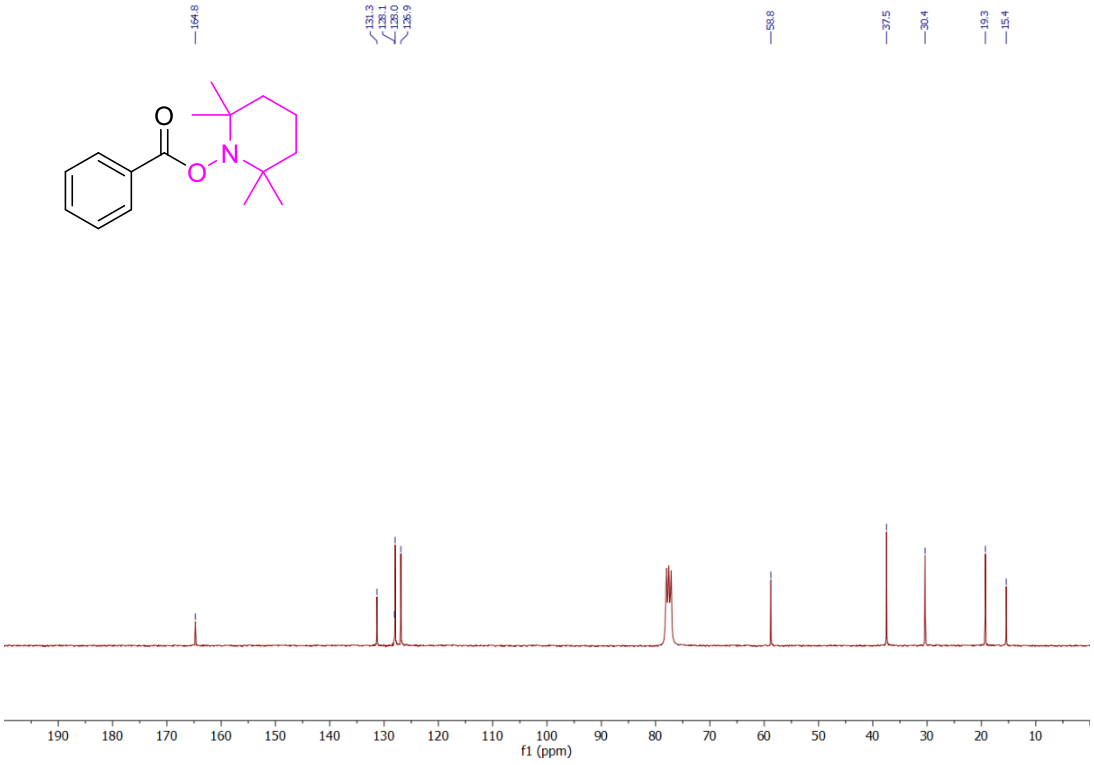
^{13}C NMR spectrum of **4ae** (75 MHz, $CDCl_3$)



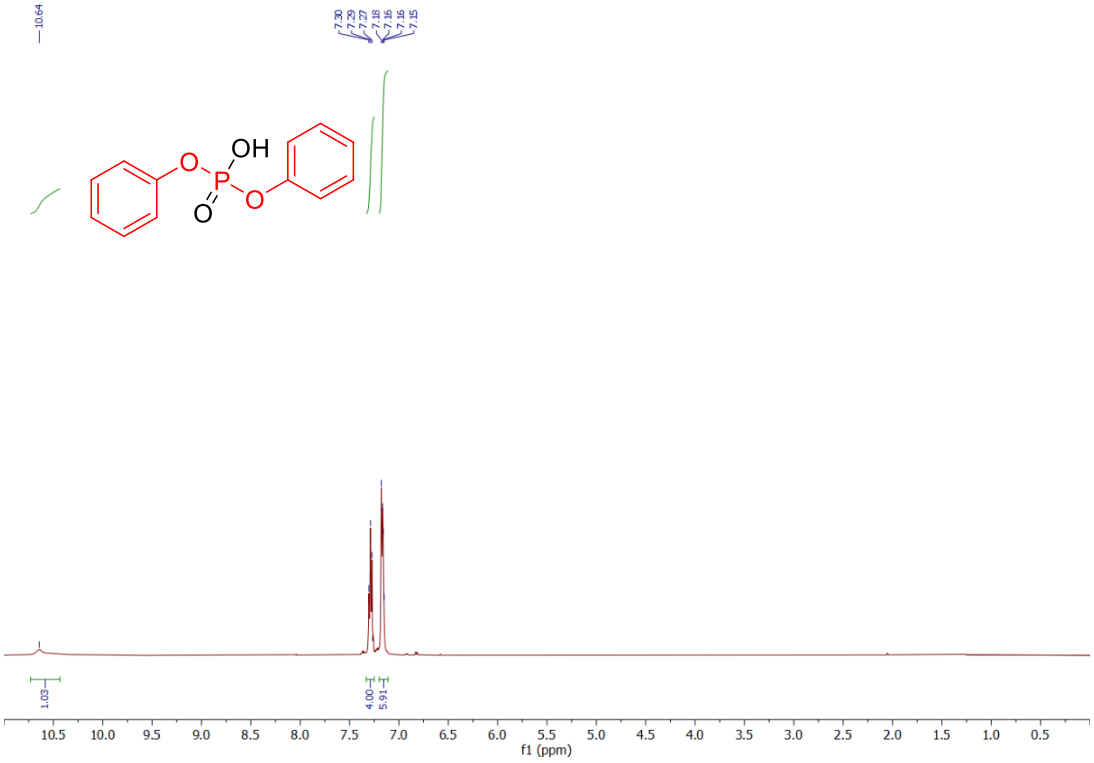
^{1}H NMR spectrum of **7** (500 MHz, $CDCl_3$)



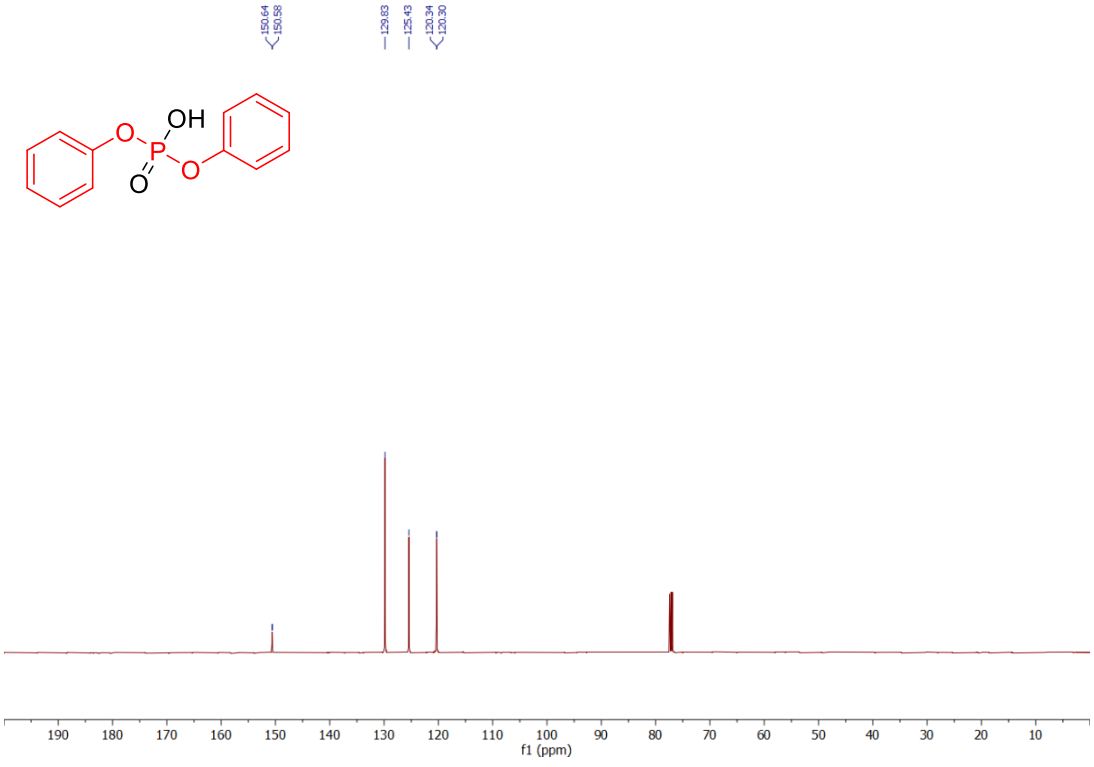
^{13}C NMR spectrum of **7** (75 MHz, $CDCl_3$)



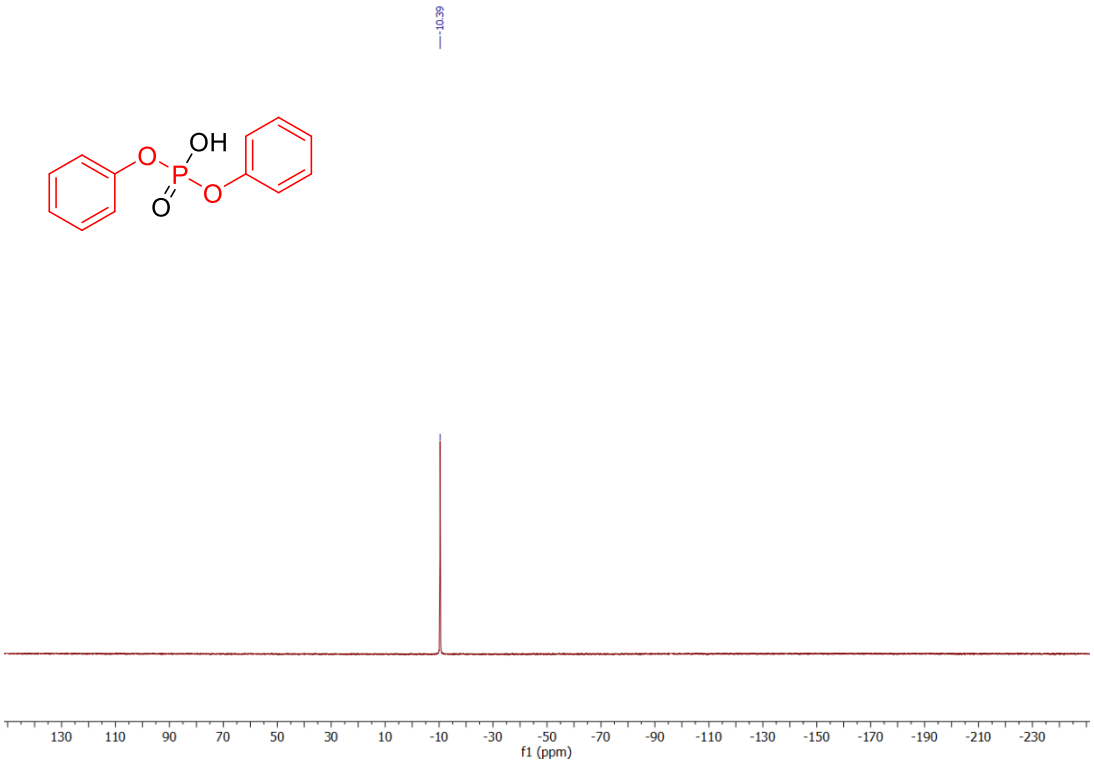
^{1}H NMR spectrum of **11** (500 MHz, $CDCl_3$)



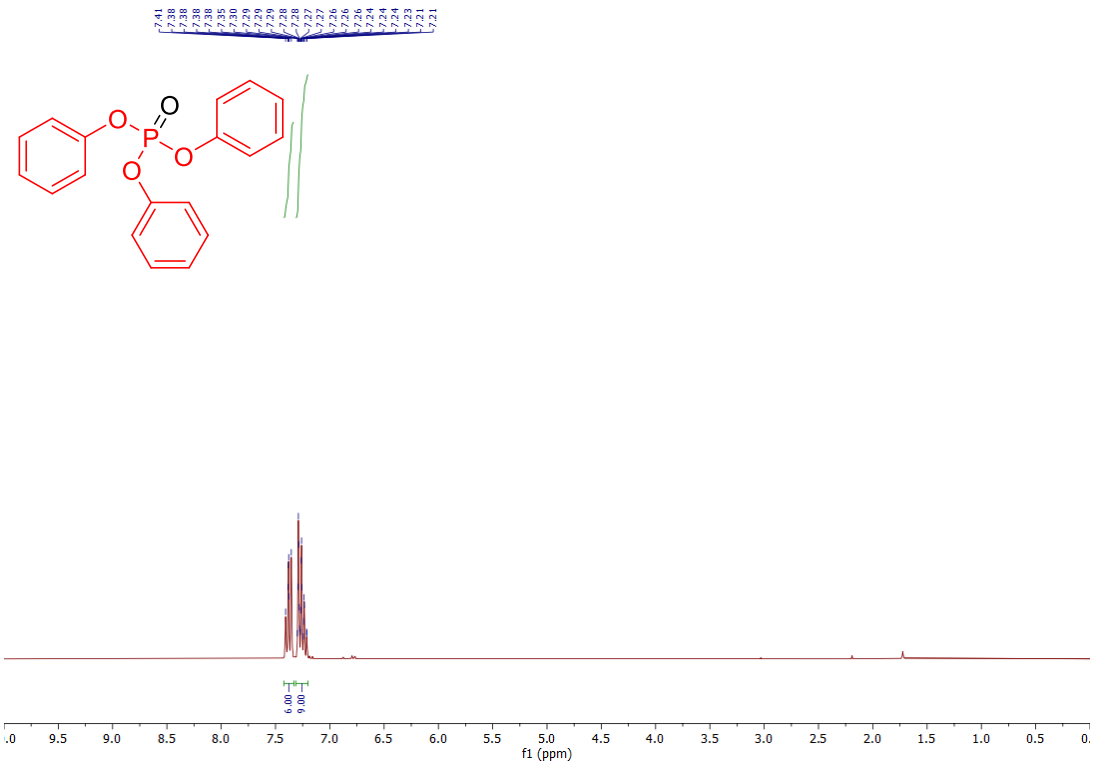
^{13}C NMR spectrum of **11** (126 MHz, $CDCl_3$)



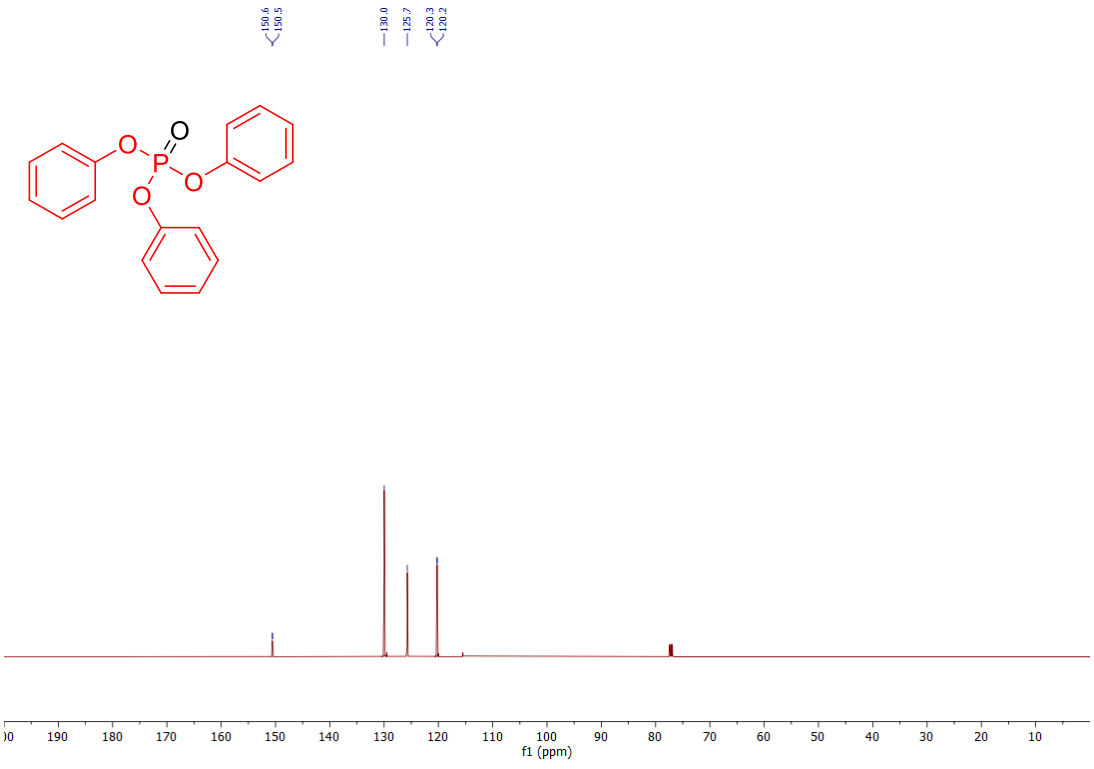
^{31}P NMR spectrum of **11** (202 MHz, $CDCl_3$)



^{1}H NMR spectrum of **12** (500 MHz, $CDCl_3$)



^{13}C NMR spectrum of **12** (126 MHz, $CDCl_3$)



^{31}P NMR spectrum of **12** (202 MHz, $CDCl_3$)