# Supporting Information

## Transition-metal and Photocatalyst-free Oxidative Cleavage of Aryl alkynes with PIDA/Iminoiodinanes in Visible Light

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## 1. General Information

The $^{1}H$ NMR and $^{13}C\{^{1}H\}$ NMR spectra were recorded using 400 MHz and 500 MHz spectrometers. $CDCl_3$ and DMSO-$d_6$ were used as NMR solvent and tetramethylsilane (TMS) was used as an internal standard. Chemical shifts are given in δ (ppm). The coupling constants, *J*, are reported in Hertz (Hz). Mass spectral data (HRMS) was recorded on electrospray ionization-time of-flight (ESI-TOF) reflectron. The reaction vessel is illuminated by a SynLED parallel Photoreactor (Catalog Number Z742680), which includes a 12 W blue LEDs, ($\lambda_{max}$=467 nm) operating at 700mA with a luminous output of 130-140 lumens. The photoreactor is also equipped with a build in cooling fan to ensure temperature stability. All reagents and solvents were of pure analytical grade. Thin-layer chromatography (TLC) was performed on 60 F254 silica gel, precoated on aluminium plates, and revealed with either a UV lamp ($\lambda_{max}$ = 254 nm) or iodine vapors. The products were purified by column chromatography on silica gel 100-200 mesh. Melting points were recorded on an Electrothermal digital melting point apparatus.

## 2. LED reaction setup:

The reaction vessel is illuminated by a SynLED parallel Photoreactor (Catalog Number Z742680), which includes a 12 W blue LEDs, ($\lambda_{max}$=467 nm).

**Figure S1:** Reaction setup

## 3. Detailed optimization studies (Tables S1-S4)

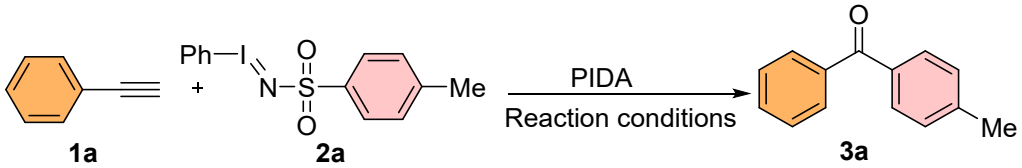



**Table S1**. Optimization of the solvent[a]

| Entry | Solvent | Yield(%)[b] |
|---|---|---|
| 1 | DCM | 68 |
| 2 | DCE | 41 |
| 3 | $CHCl_3$ | 49 |
| 4 | Acetone | NR |
| 5 | DMSO | NR |
| 6 | DMF | NR |
| 7 | Chlorobenzene | NR |
| 8 | toluene | NR |
| 9 | MeCN | 24 |
| 10 | MeOH | NR |
| 11 | THF | Traces |
| 12 | 1,4-dioxane | NR |

[a]Reaction conditions: **1a** (21 mg, 0.2 mmol, 1.0 equiv.), **2a** (89 mg, 0.24 mmol, 1.2 equiv.), and PIDA (45.0 mg, 0.14 mmol, 0.7 equiv.), Solvent = 3.0 mL, 12 W blue LEDs, rt, open-air, 8 h. [b]Isolated yield, NR = No reaction.

**Table S2**. Optimization of the stoichiometry of substrates[a]

| 1a (equiv.) | 2a (equiv.) | PIDA (equiv.) | Yield[b]% |
|---|---|---|---|
| 1.0 | 1.2 | - | Traces |
| 1.0 | 1.2 | 0.4 | 39 |
| 1.0 | 1.2 | 1.0 | 63 |
| 1.0 | 1.2 | 1.2 | 59 |

| | | | |
|---|---|---|---|
| 1.0 | - | 0.7 | NR |
| 1.0 | 0.6 | 0.7 | 34 |
| 1.0 | 2.0 | 0.7 | 69 |
| 2.0 | 1.2 | 0.7 | 67 |

[a]Reaction conditions: DCM = 3.0 mL, 12 W blue LEDs, rt, open-air, 8 h. [b]Isolated yield.

**Table S3**. Effect of light source and time[a]

| **Light Source** | **Time (h)** | **Yield(%)[b]** |
|---|---|---|
| 50 W blue LEDs | 8 | 45 |
| 12 W blue LEDs | 8 | 68 |
| Green LEDs | 8 | 21 |
| White light | 8 | 37 |
| 12 W blue LEDs | 4 | 40 |
| 12 W blue LEDs | 12 | 68 |
| 12 W blue LEDs | 20 | 68 |

[a]Reaction conditions: **1a** (21 mg, 0.2 mmol, 1.0 equiv.), **2a** (89 mg, 0.24 mmol, 1.2 equiv.), and PIDA (45.0 mg, 0.14 mmol, 0.7 equiv.), DCM = 3.0 mL, light source, rt, time. [b]Isolated yield, NR = No reaction.

**Table S4**. Optimization of the oxidant[a]

| **Entry** | Oxidant | **Yield(%)[b]** |
|---|---|---|
| 1 | $H_2O_2$ | NR |
| 2 | $K_2S_2O_8$ | NR |
| 3 | $K_3PO_4$ | NR |
| 4 | $(NH_4)_2S_2O_8$ | NR |
| 5 | TBHP | NR |

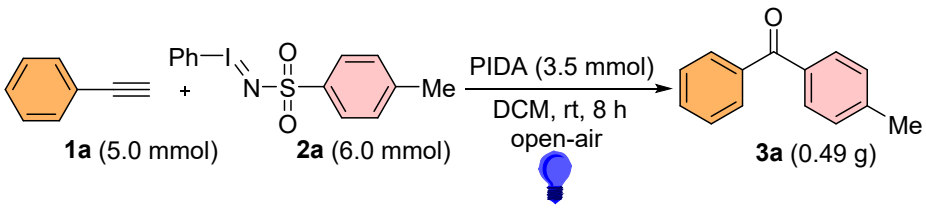
[a]Reaction conditions: **1a** (21 mg, 0.2 mmol, 1.0 equiv.), **2a** (89 mg, 0.24 mmol, 1.2 equiv.), and oxidant (0.14 mmol, 0.7 equiv.), DCM = 3.0 mL, 12 W blue LEDs, rt, open air, 8 h. [b]Isolated yield, NR = No reaction.

**4. Procedure A:** Synthesis of iminoiodinane (2)[1]

To a solution of $ArSO_2NH_2$ (1.0 equiv.), potassium hydroxide (2.5 equiv.) and methanol (120 ml) were stirred in a conical flask in an ice bath, ensuring the reaction mixture was below 10 °C. (It's not essential to have a solution, a suspension is more likely at this temp). Iodobenzene diacetate (1.0 equiv.) was added to the stirred mixture and the resulting yellow colored solution was stirred at room temperature for 3.5 h. The reaction mixture was poured into a large excess of iced water and stirred for 1 h. A yellow coloured solid precipitated on standing overnight. (It's important to allow the solid to stand as the particle size appears to increase giving higher yield on filtration). The light yellow solid was isolated by filtration and dried with a flow of air through the buchner funnel. Several portions of ether, in which the product is insoluble were used to wash away any iodobenzene present. The yellow solid was dissolved in a minimum of boiling methanol. The solution was placed in a freezer overnight whereupon an off-white solid (iminoiodinane, PhINTs) was recovered via filtration.

**5. Gram scale synthesis**

To an oven dried 250 mL round bottom flask, Phenyl acetylene **1a** (510 mg, 5.0 mmol, 1.0 equiv.), 4-methyl-*N*-(phenyl-$\lambda^3$-iodaneylidene)benzenesulfonamide **2a** (2.2 g, 6.0 mmol, 1.2 equiv.) and PIDA (1.1 g, 3.5 mmol, 0.7 equiv.) were dissolved in DCM (75 mL) in an oven-dried 100 mL round bottom flask equipped with a magnetic stirring bar, and irradiated using 12 W Blue LED at room temperature under air for 16 h. After completion of the reaction as monitored by TLC, the resulting residue was purified by column chromatography on silica gel (EtOAc/hexane = 1:99) to give desired product **3a** in 51% yield (0.49g).




**Scheme S1:** Scale-up experiment

**6. Crystallographic description of compounds 3i**

Single crystal X-ray diffraction data of compound **3i** was collected using a Bruker SMART APEX diffractometer equipped with a 3-axis goniometer. The crystal was grown by dissolving 10 mg of **3i** in 0.5 mL of chloroform. The clear solution was covered and kept at room temperature for 105 h.

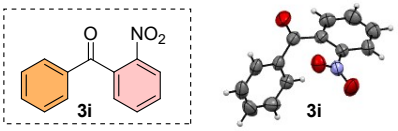



**Figure S2**: ORTEP diagram of compound **3i** (with 40% probability ellipsoids).

| | |
|---|---|
| Identification code | rajat_0ma_a |
| Empirical formula | $C_{13}H_9NO_3$ |
| Formula weight | 227.21 |
| Temperature | 152 K |
| Wavelength | 0.71073 Å |
| Crystal system | Monoclinic |
| Space group | P 1 21/c 1 |
| Unit cell dimensions | a = 8.146(4) Å; α = 90 ° |
| | b = 17.380(8) Å; β = 92.193(19) ° |
| | c = 7.782(4) Å; γ = 90 ° |
| Volume | 1100.9(9) $Å^3$ |
| Z | 4 |
| Density (calculated) | 1.371 $g/cm^3$ |
| Absorption coefficient | 0.099 |
| F(000) | 472.0 |
| Crystal size | 0.014 × 0.014 × 0.012 $mm^3$ |
| Theta range for data collection | 2.50 to 24.74 ° |
| Index ranges | -9 ≤ h ≤ 9, |

| | |
|---|---|
| | -20 ≤ k ≤ 20, |
| | -9 ≤ l ≤ 9 |
| Reflections collected | 8670 |
| Independent reflections | 2019 [Rint = 0.0885] |
| Completeness to theta = 25.418° | 99.6 % |
| Data / restraints / parameters | 2019 /0/ 154 |
| Goodness-of-fit on $F^2$ | 0.996 |
| Final R indices [I>2sigma(I)] | $R_1$ = 0.0845, |
| | $WR_2$ = 0.2256 |
| R indices (all data) | $R_1$ = 0.1289, |
| | $WR_2$ = 0.2657 |
| CCDC | 2447088 |

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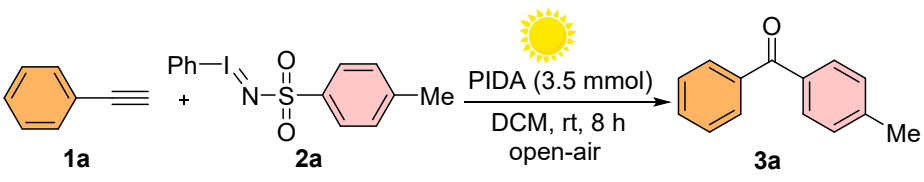
**7. Irradiation under natural sunlight**

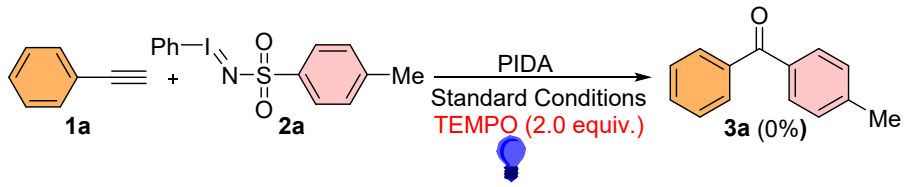
To an oven-dried reaction vial, phenyl acetylene **1a** (21.0 mg, 0.2 mmol), PhINTs **2a** (89.0 mg, 0.24 mmol) and PIDA (45.0 mg, 0.14 mmol) were dissolved in DCM (3 mL). The contents were kept in sunlight in an open atmosphere for 8 h (from 9:00 to 17:00 at IIT Delhi, India) without stirring. The reaction mixture was concentrated *in vacuo* and resulting residue was purified by column chromatography on silica gel (EtOAc/hexane = 1:99) to give desired product **3a** in 22% yield (8.6 mg).




**Scheme S2:** Synthesis of **3a** under natural sunlight

## 8. Mechanistic Studies

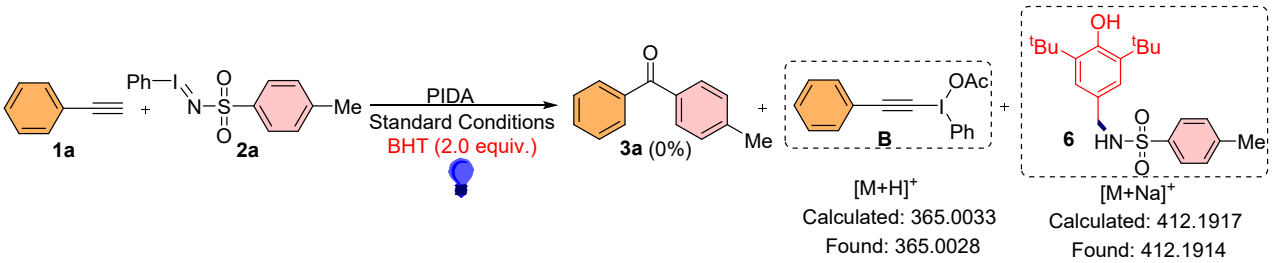
### a) Free radical-trapping experiment with TEMPO




**Scheme S3:** Free-radical trapping with TEMPO

Phenyl acetylene **1a** (21 mg, 0.20 mmol, 1.0 equiv.), 4-methyl-*N*-(phenyl-$\lambda^3$-iodaneylidene)benzenesulfonamide **2a** (89 mg, 0.24 mmol, 1.2 equiv.), PIDA (45.0 mg, 0.14 mmol, 0.7 equiv.) and 2,2,6,6-tetramethylpiperidinyloxy (TEMPO, 0.40 mmol, 2.0 equiv.) were dissolved in DCM (3.0 mL) in an oven-dried reaction vessel equipped with a magnetic stirring bar, and the reaction vessel was irradiated using 12 W Blue LED at room temperature under air for 8 h. The desired product **3a** was not detected.

### b) Free radical-trapping experiment with BHT




**Scheme S4:** Free-radical trapping with BHT

Phenyl acetylene **1a** (21 mg, 0.20 mmol, 1.0 equiv.), 4-methyl-*N*-(phenyl-$\lambda^3$-iodaneylidene)benzenesulfonamide **2a** (89 mg, 0.24 mmol, 1.2 equiv.), PIDA (45.0 mg, 0.14 mmol, 0.7 equiv.) and 2,6-ditert-butyl-4-methylphenol (BHT, 0.40 mmol, 2.0 equiv.) were dissolved in DCM (3.0 mL) in an oven-dried reaction vessel equipped with a magnetic stirring bar, and the reaction vessel was irradiated using 12 W Blue LED at room temperature under air for 8 h. The desired product **3a** was not detected and the adduct **B and 6** was identified by HRMS of the reaction mixture.

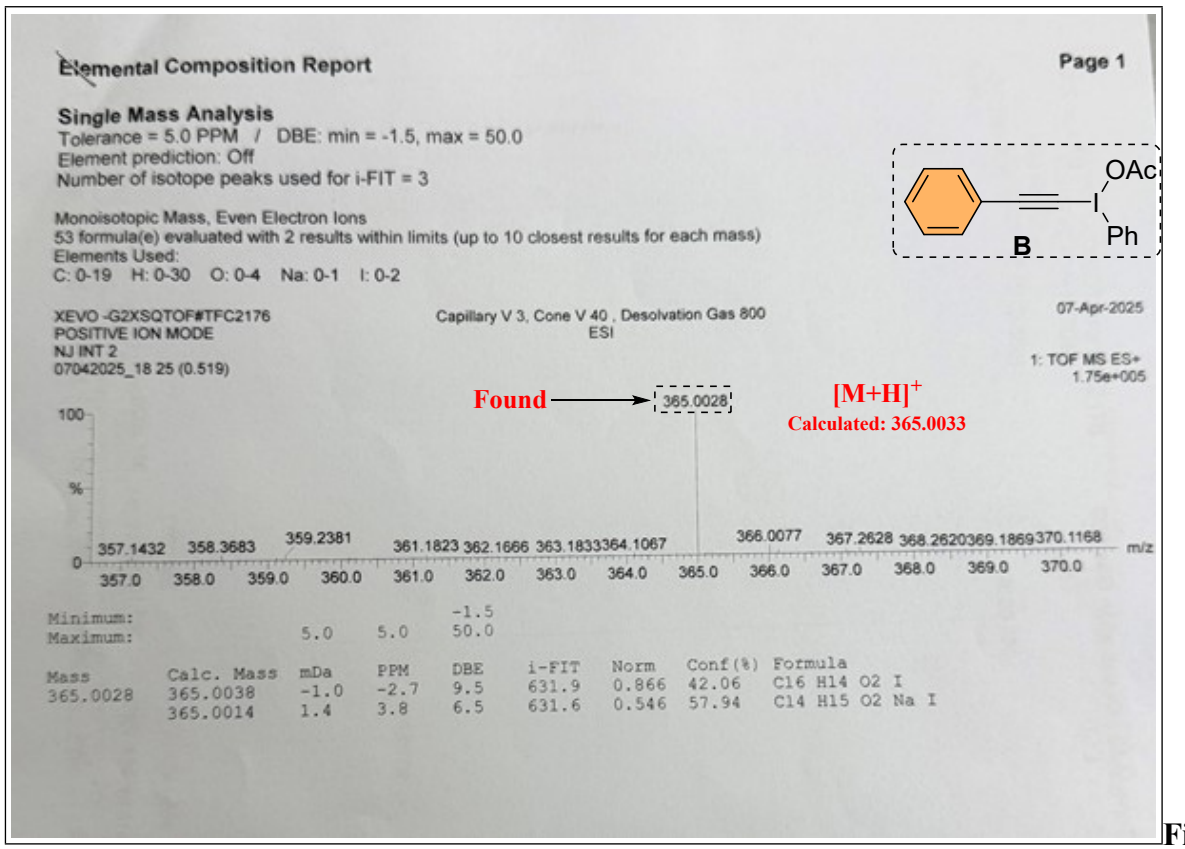



**Figure S3:** HRMS of the intermediate **B**

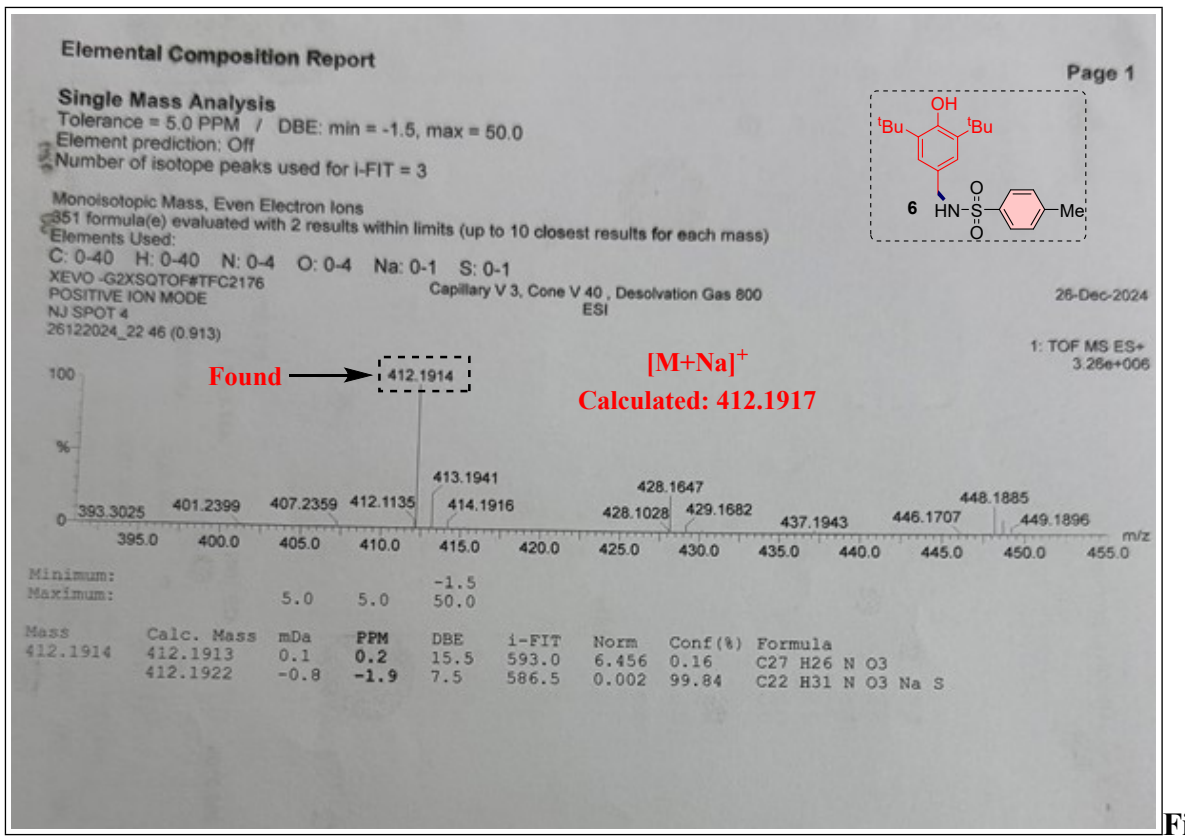
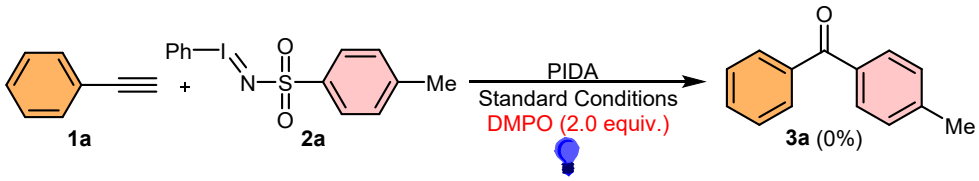



**Figure S4:** HRMS of the BHT adduct **6**

## c) EPR Studies

EPR measurements were performed on Bruker A300-9.5/12/S/W instrument using microwave strength 9.8 GHz, sweep time 60 seconds, and one scan. The EPR spectrum of the reaction mixture was recorded after 20 min of irradiation using 5,5- dimethyl-1-pyrroline N-oxide (DMPO) as a spin trap. The signal indicates the presence of paramagnetic species.




Reaction Conditions: **1a** (21.0 mg, 0.20 mmol, 1.0 equiv.), **2a** (89.0 mg, 0.24 mmol, 1.2 equiv.) and PIDA (45.0 mg, 0.14 mmol, 0.7 equiv.) DCM = 3.0 mL, 12 W blue LEDs, rt, open-air. After 20 min of stirring under 12 W blue LEDs, DMPO (2.0 equiv.) was added and EPR spectrum was recorded.

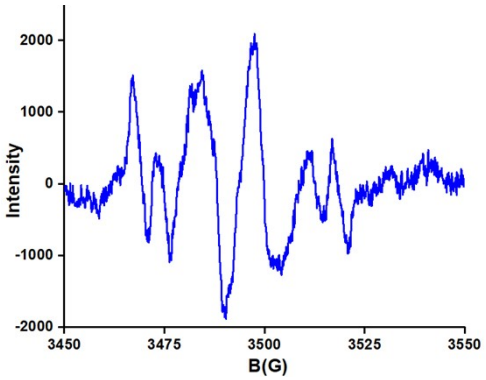
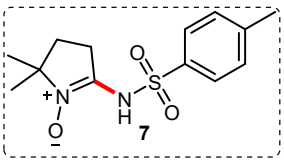



**Figure S5**: EPR spectrum of radical with spin trap (DMPO).

The adduct **7** was also identified by HRMS.



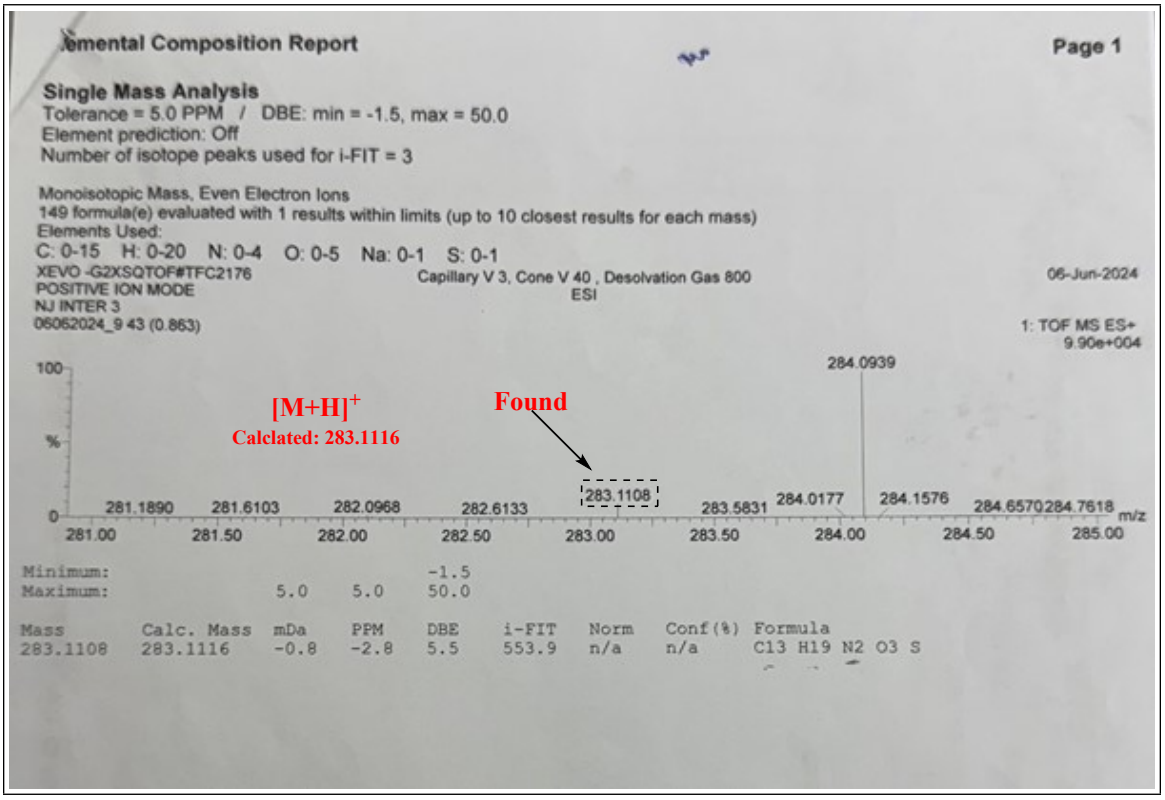



**Figure S6:** HRMS of the reaction mixture

**d) Detection of CO (Monitoring reaction by HS-GC)**

A sealed tube was charged with Phenyl acetylene **1a** (21 mg, 0.20 mmol, 1.0 equiv.), 4-methyl-*N*-(phenyl-$\lambda^3$-iodaneylidene)benzenesulfonamide **2a** (89 mg, 0.24 mmol, 1.2 equiv.), PIDA (45.0 mg, 0.14 mmol, 0.7 equiv.) were dissolved in DCM (3.0 mL) with constant stirring , and the reaction tube was irradiated using 12 W Blue LED at room temperature for 8 h. After completion of the reaction, the resulting gas from the reaction mixture was directly inserted into the HS-GC instrument via a syringe. A calibration curve was drawn between the retention time (min) of gases and response in mV. A peak was found at 4.4 min which shows the generation of CO in the reaction mixture. Gas quantification was carried out by gas chromatography (GC trace 1110 Thermo scientific with carboxen column, FID detector).

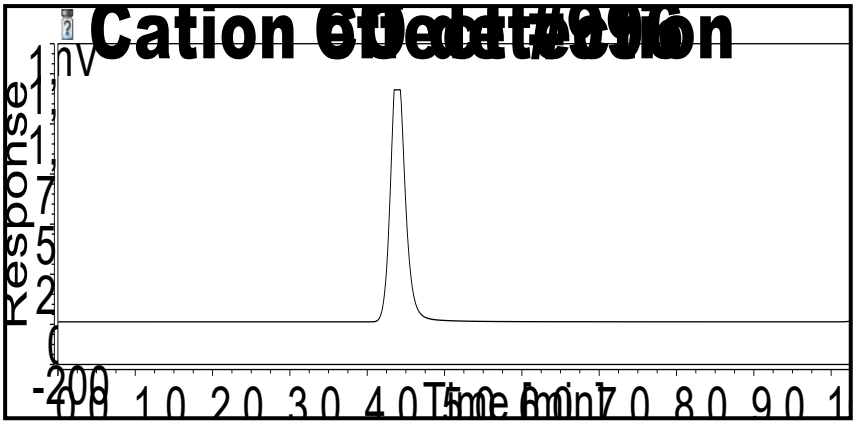



**Figure S7:** Calibration Curve for CO estimation

**e) *In-situ* IR analysis**

To detect the formation of product (**3a**) during the reaction, an *in-situ* FTIR experiment was conducted using a Mettler-Toledo React IR 700 (SN: C049640472) equipped with a TEMCT detector, DiComp (Dimond) probe, with a 9.5mm x 2m AgX fiber interface. Data was collected using the 2500 to 650 $cm^{-1}$ spectral window with 8 $cm^{-1}$ resolution and sampled in 60 second intervals. In a glass vial, Phenyl acetylene **1a** (21.0 mg, 0.20 mmol, 1.0 equiv.), 4-methyl-*N*-(phenyl-$\lambda^3$-iodaneylidene)benzenesulfonamide **2a** (89.0 mg, 0.24 mmol, 1.2 equiv.), PIDA (45.0 mg, 0.14 mmol, 0.7 equiv.) were dissolved in DCM (3.0 mL) with constant stirring. Then, a diamond probe was inserted in a solution containing a vial, *in-situ* FTIR spectra were recorded for 3.5 h and with the increase in the time, the formation of **3a** was increase. As depicted in Figure S8: (a) The signal intensity at 1703 $cm^{-1}$ (corresponding to C=O of **3a**) started increasing after 3.5 h, indicating the formation of carbonyl in product **3a**. (b) Depicts the 2D spectra for the reaction monitoring indicating the formation of **3a** during the reaction.

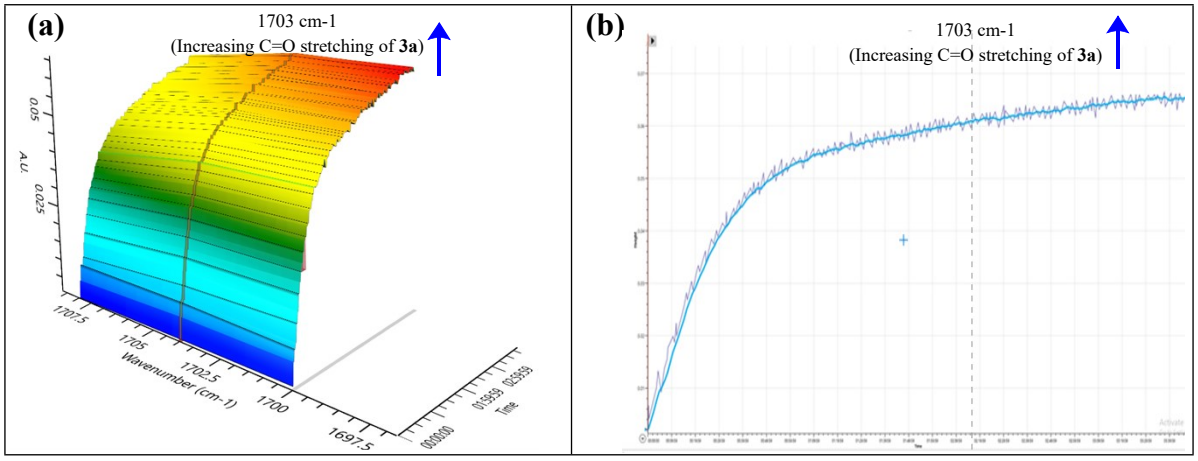



**Figure S8:** *In-situ* IR monitoring of the reaction, reaction conditions = **1a**:**2a:PIDA** (1:1.2:0.7) on a 0.2 mmol scale, DCM = 3 mL, 12 W blue LED, rt, open-air, 3.5 h. (a) The signal intensity at 1703 $cm^{-1}$ (corresponding to C=O of **3a**) started increasing after 3.5 h, indicating the formation of carbonyl in product **3a**. (b) Depicts the 2D spectra for the reaction monitoring indicating the formation of **3a** during the reaction.

### f) UV-Vis Experiments

Compounds **A-F** were measured in a 0.001 M solution in DCM at room temperature. All spectra were directly recorded after setting up the solutions.[2]

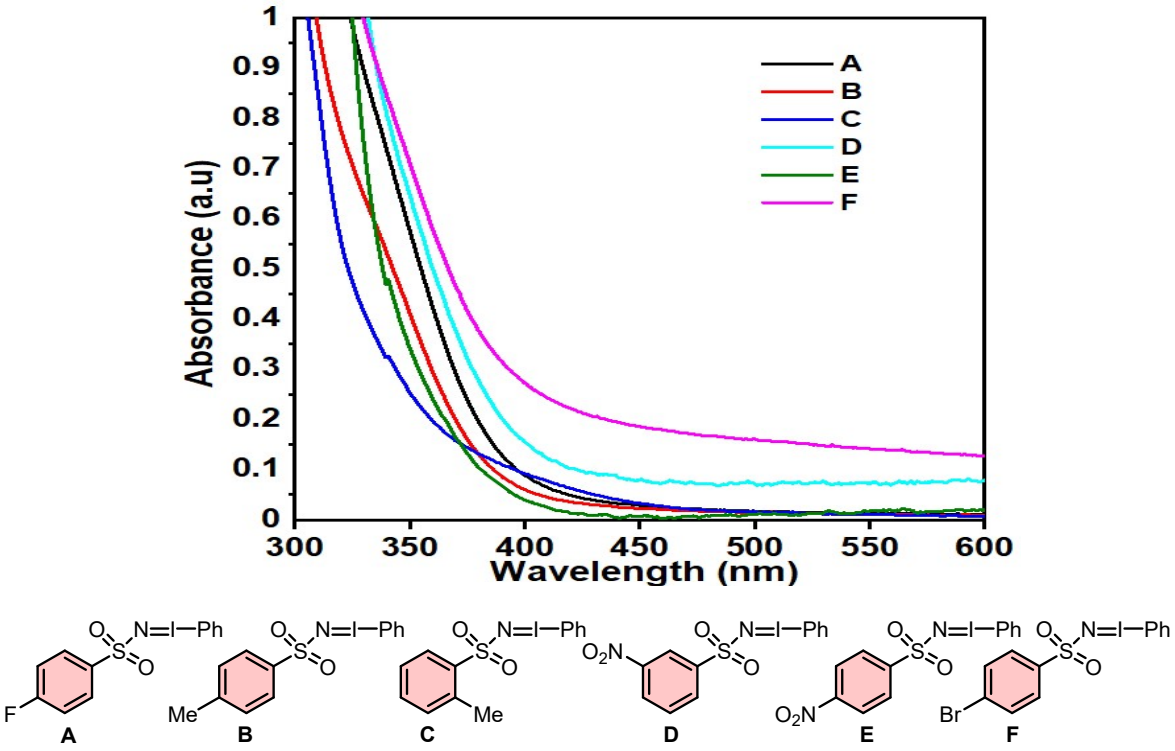



**Figure S9:**` UV-Vis spectra of different iminoiodonanes 0.001 M in DCM.

**g) Light ON/OFF experiment:**

A parallel eight reaction was conducted for the Light ON/OFF experiment. To an oven-dried reaction tube, phenyl acetylene **1a** (21 mg, 0.20 mmol, 1.0 equiv.), 4-methyl-*N*-(phenyl-$\lambda^3$-iodaneylidene)benzenesulfonamide **2a** (89 mg, 0.24 mmol, 1.2 equiv.), and PIDA (45.0 mg, 0.14 mmol, 0.7 equiv.) were dissolved in DCM (3.0 mL) and irradiated using 12 W Blue LED at room temperature for indicated time. The light was turned on and off every one hour. Upon the indicated time, the reaction mixture was concentrated under reduced pressure and analyzed by $^1$HNMR with 1,3,5-trimethoxybenzene as an internal standard to determine the yield of **3a**, and the results are shown below:

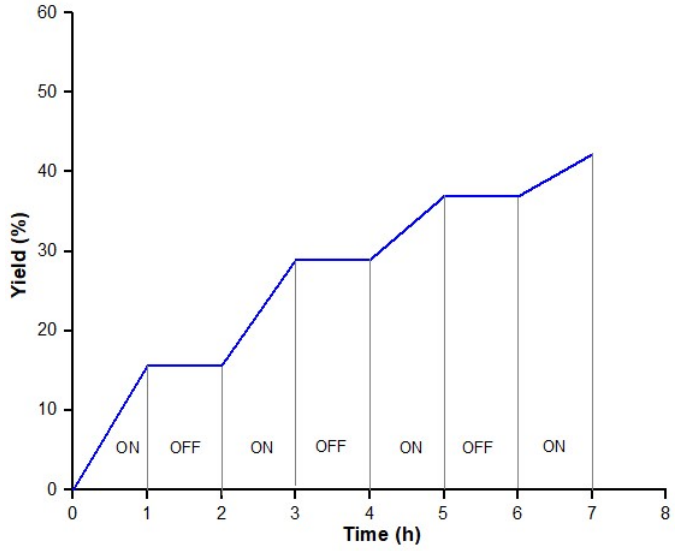


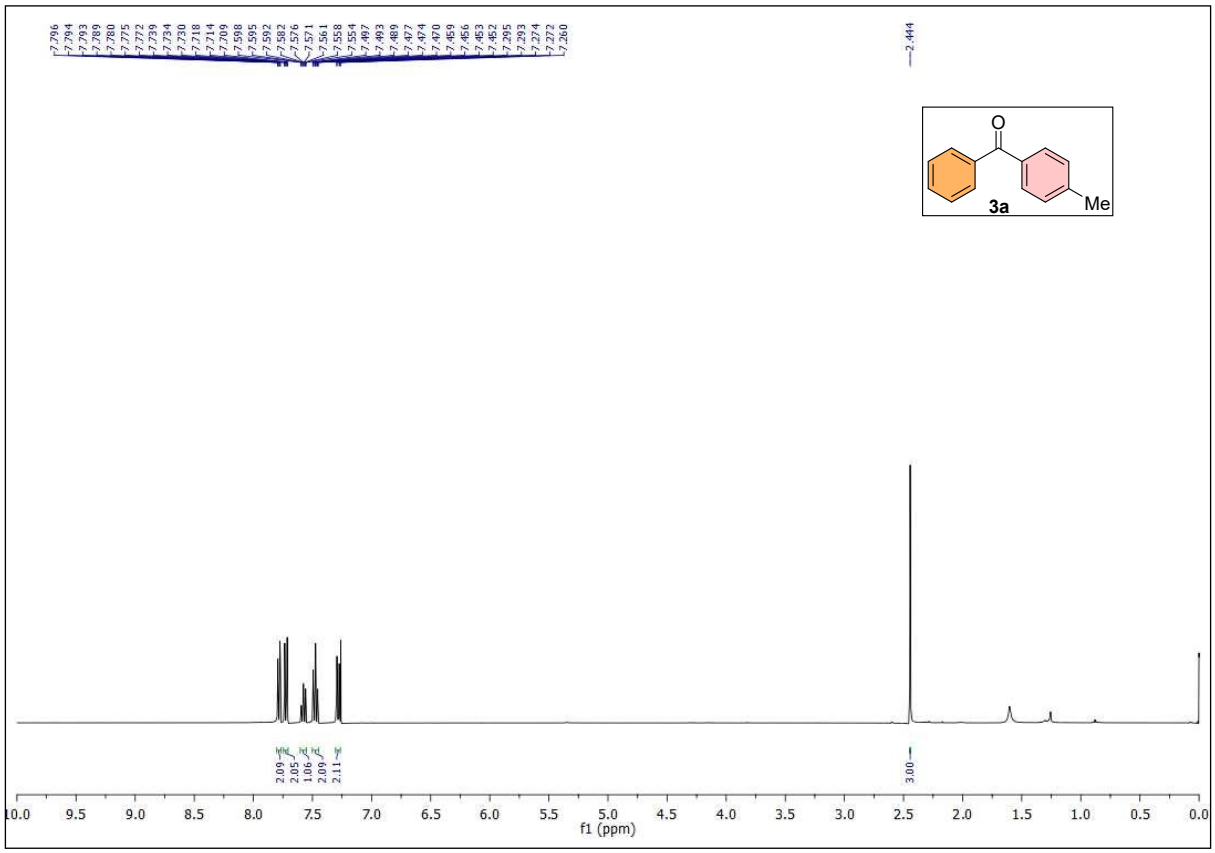

**Figure S10:** Light ON/OFF experiment

## 9. References

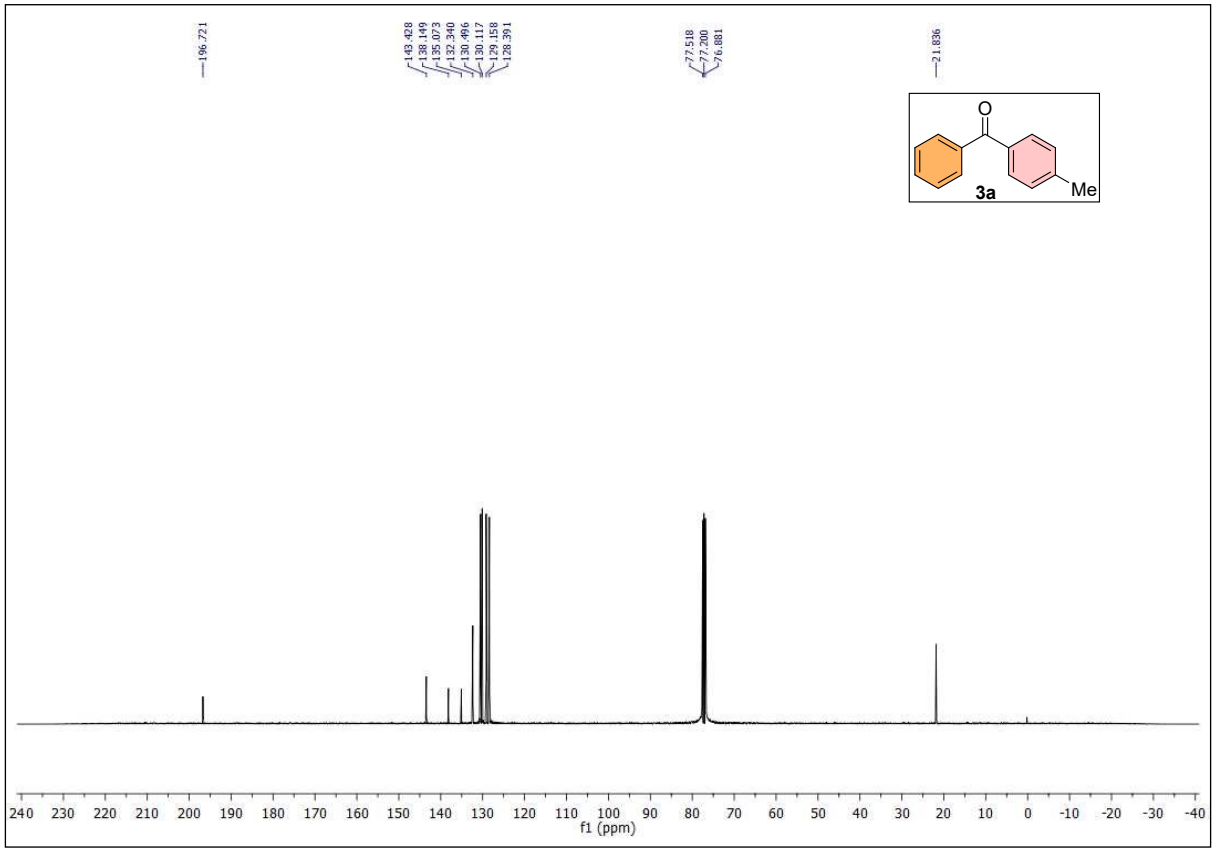
[1] Y. Guo, C. Pei, S. Jana, and R. M. Koenigs, *ACS Catal*. **2021**, *11*, 337−342.

[2] I. Jurberg, R. Nome, S. Crespi, T. D. Z. Atvars, B. Konig, *Adv. Synth. Catal*., 2022, **364**, 4061.

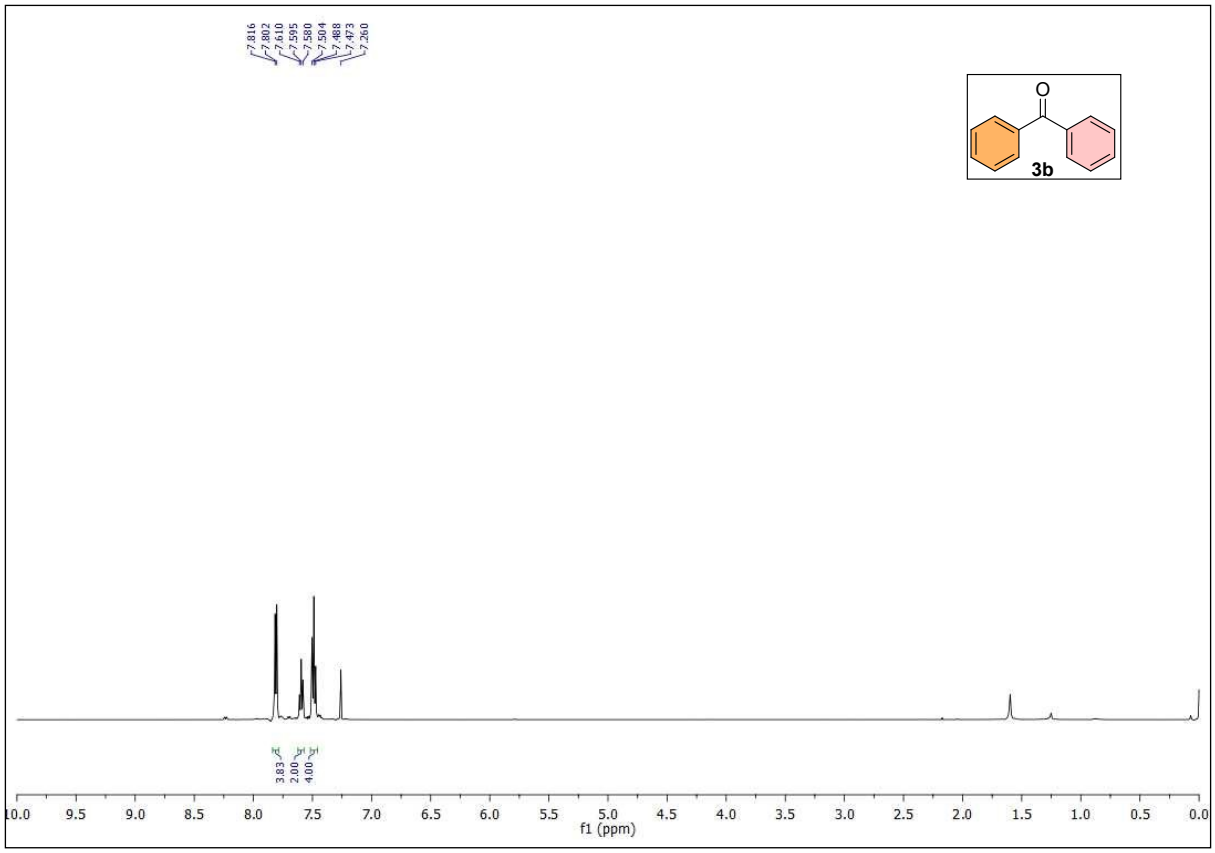
## 10. Copies of $^{1}H$ NMR and $^{13}C\{^{1}H\}$ NMR spectra of synthesized compounds



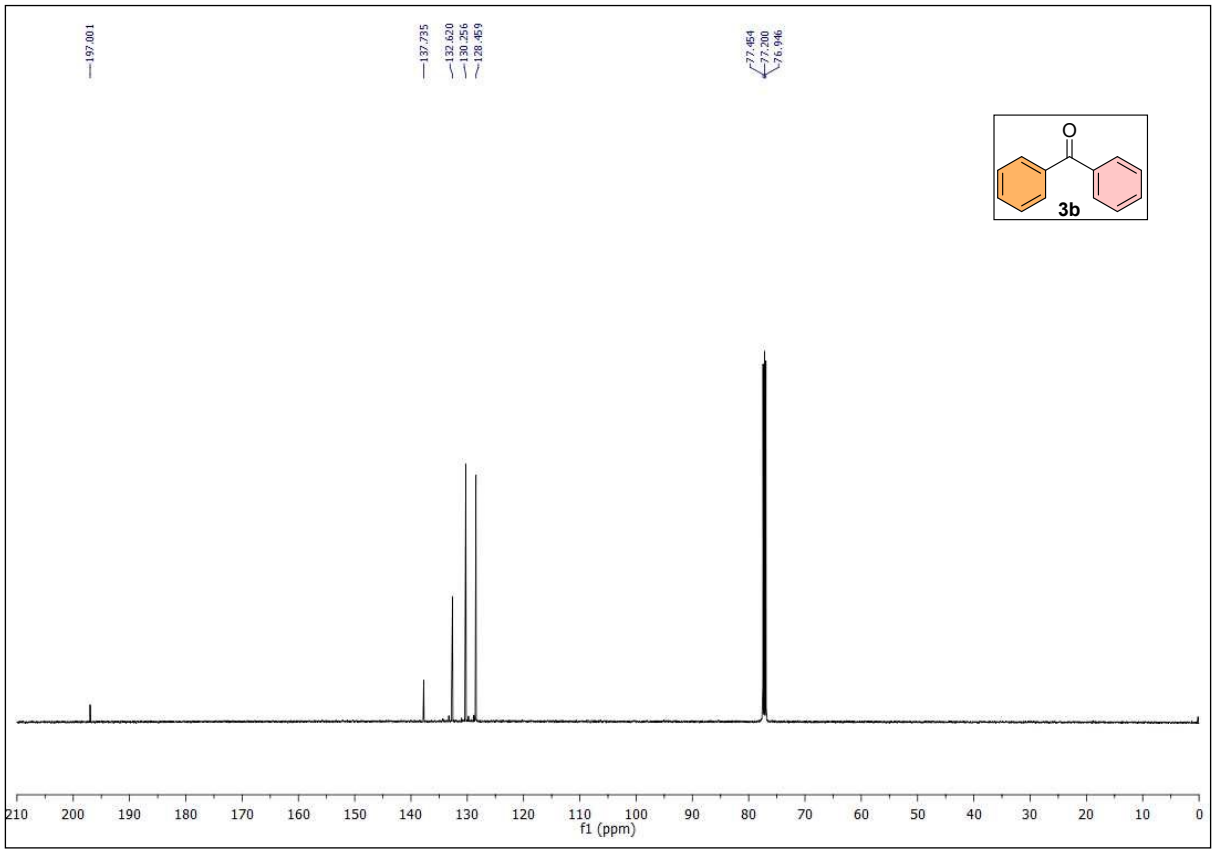

**Spectrum 1**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3a**



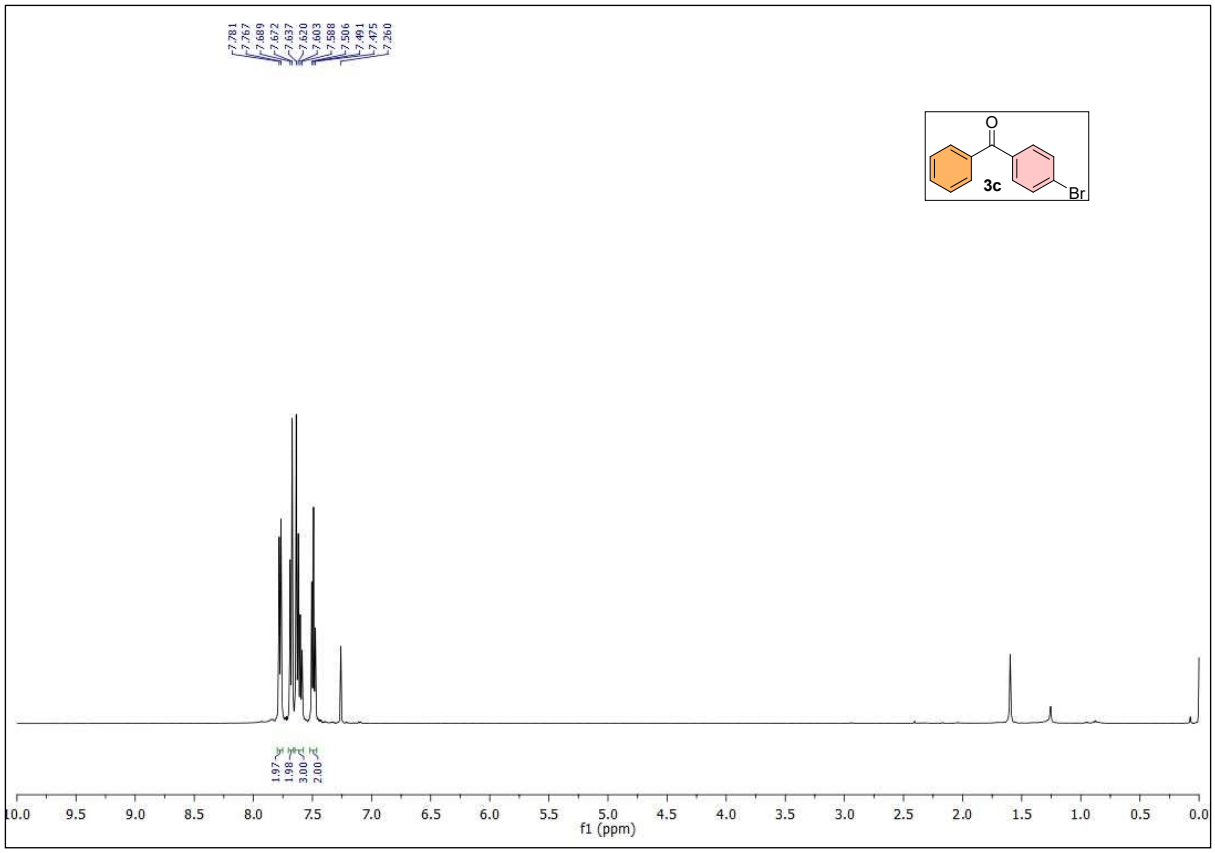

**Spectrum 2**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3a**



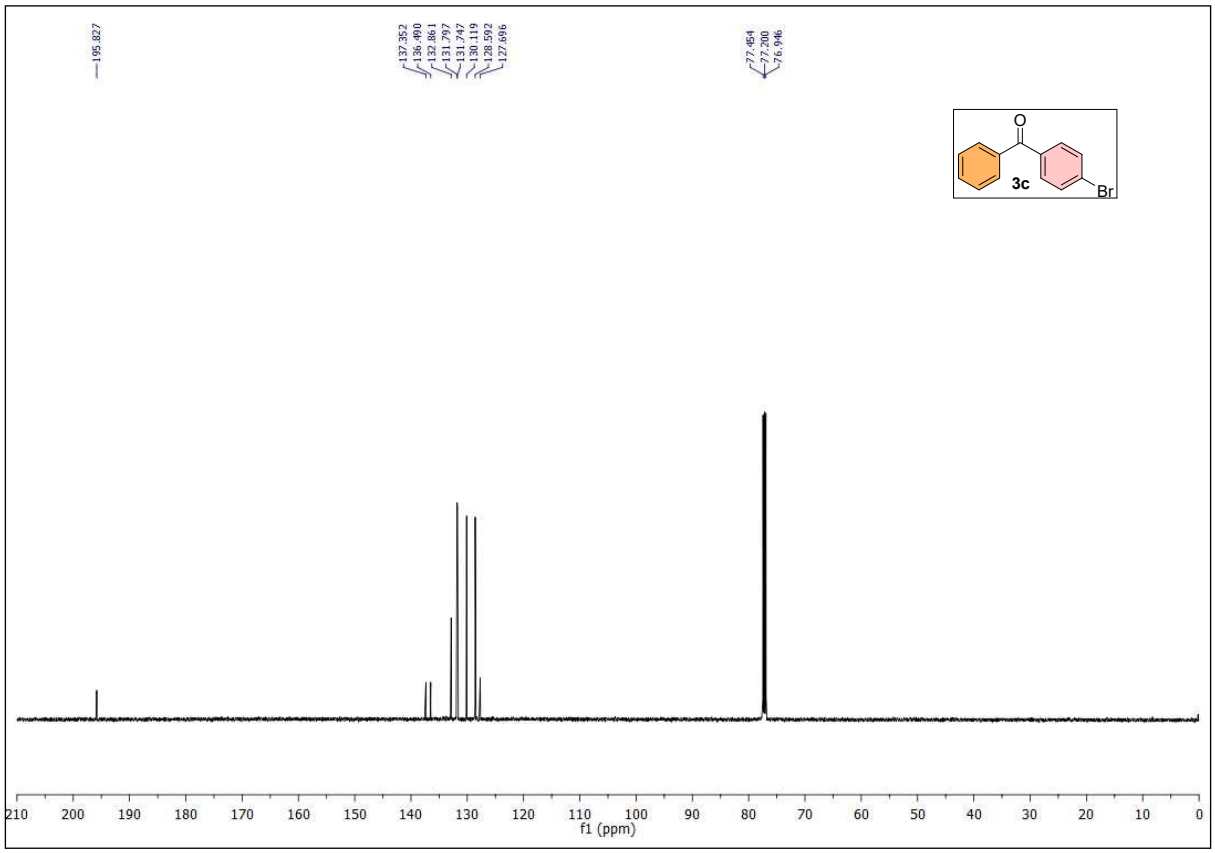

**Spectrum 3**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3b**



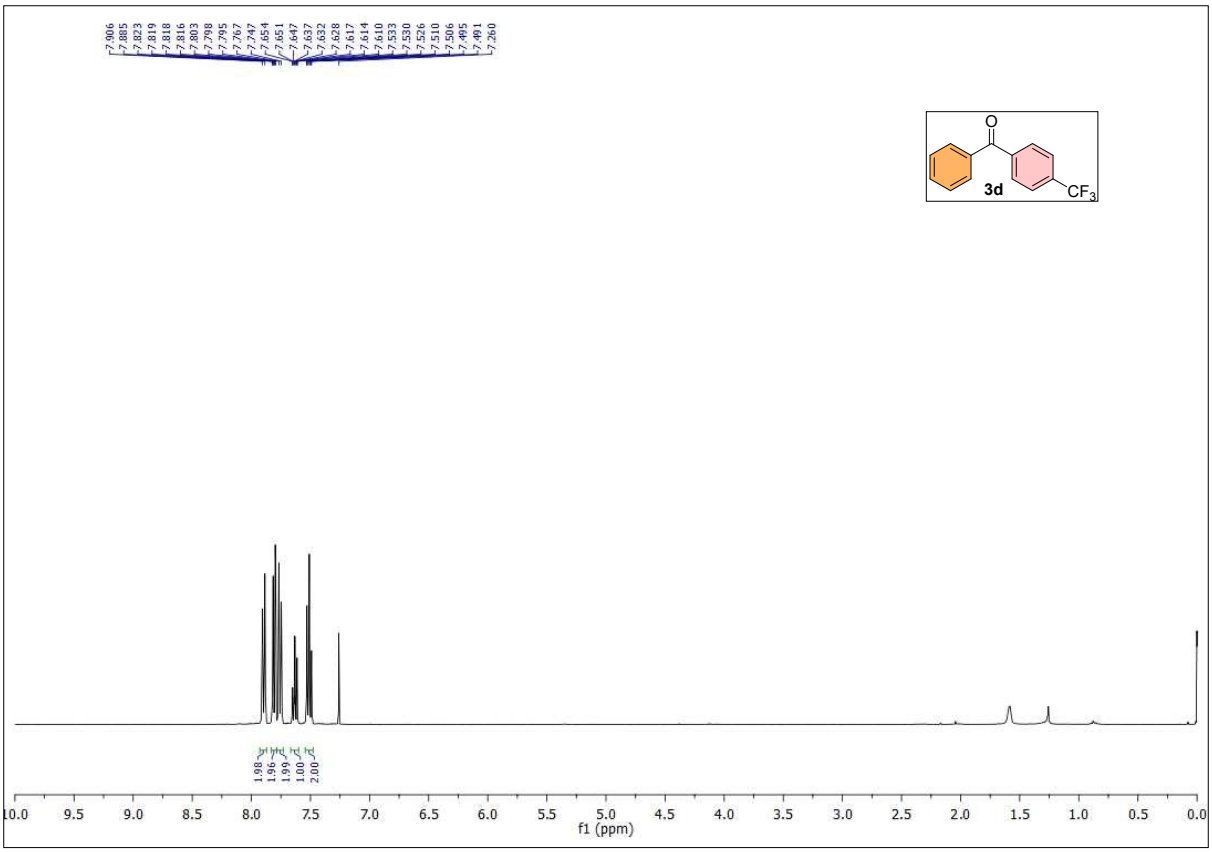

**Spectrum 4**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3b**



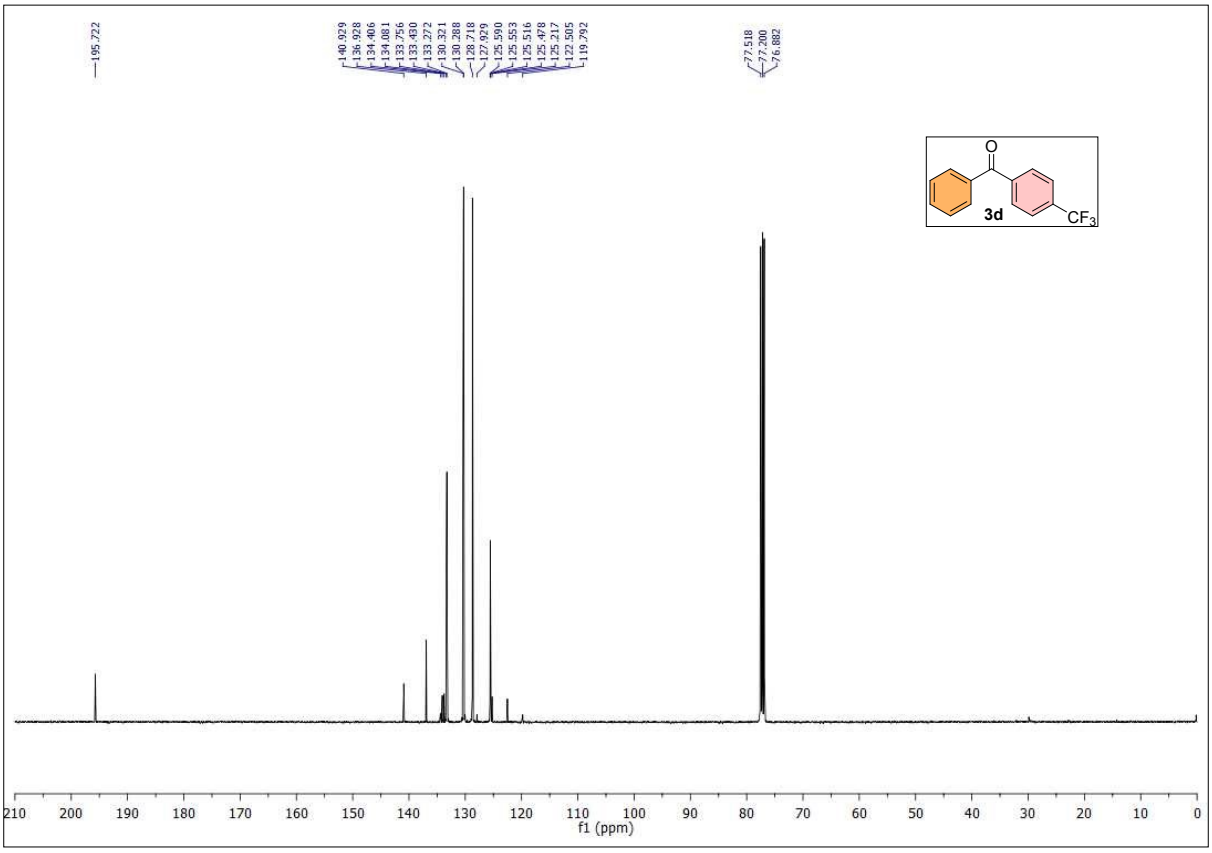

**Spectrum 5**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3c**



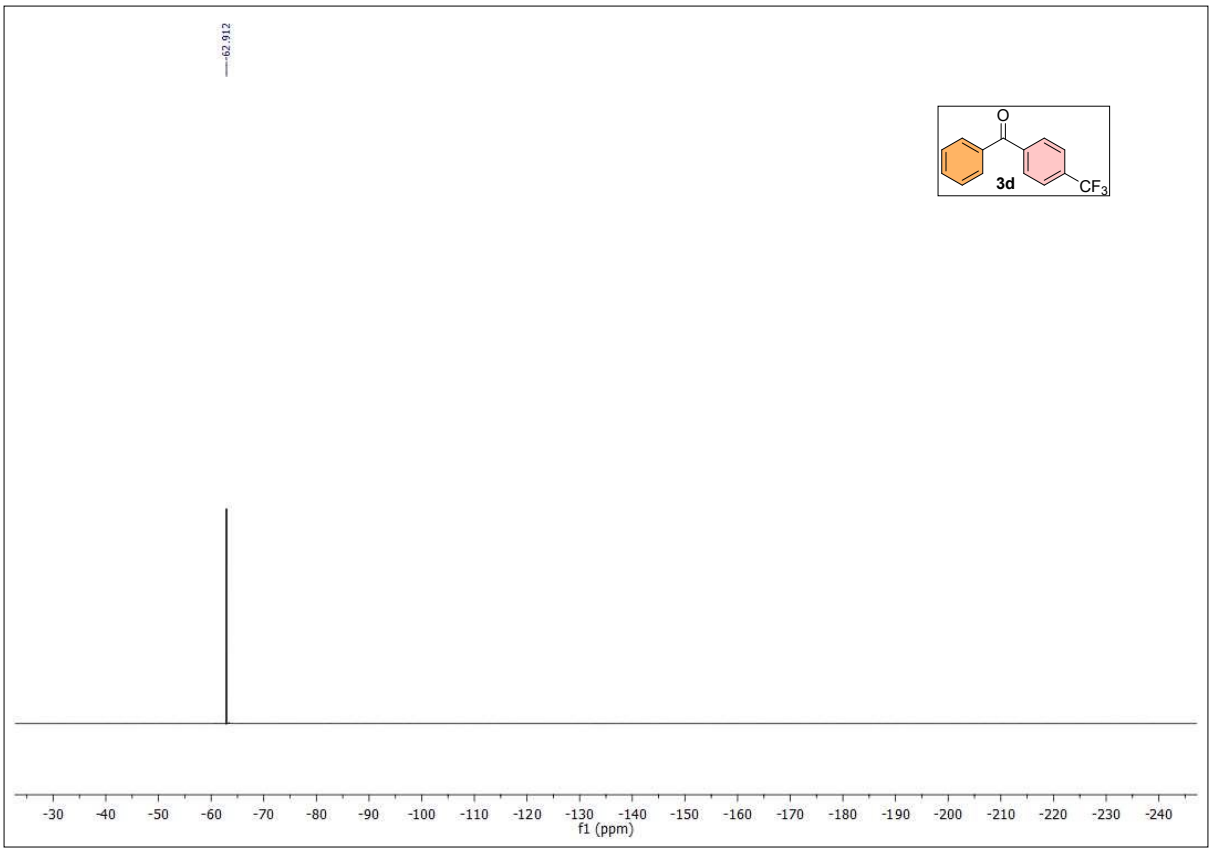

**Spectrum 6**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3c**



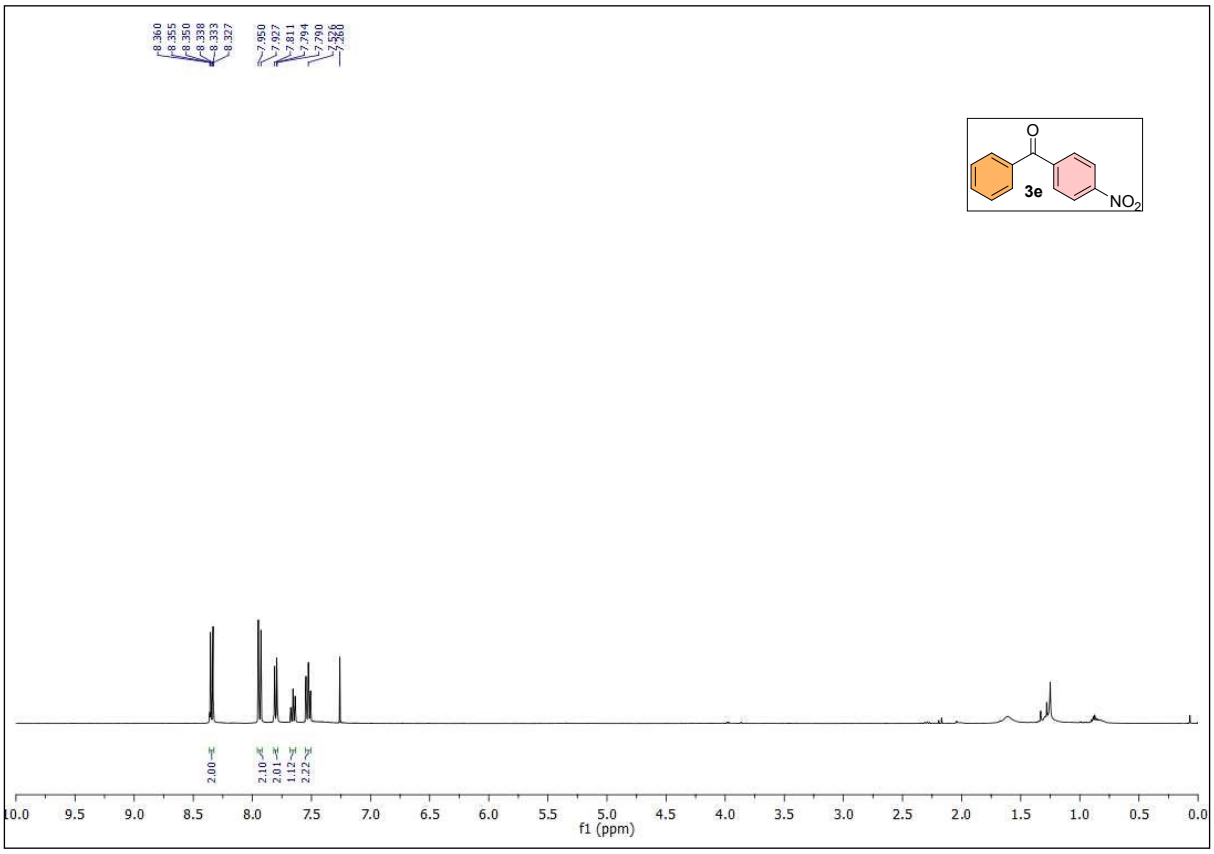

**Spectrum 7**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3d**



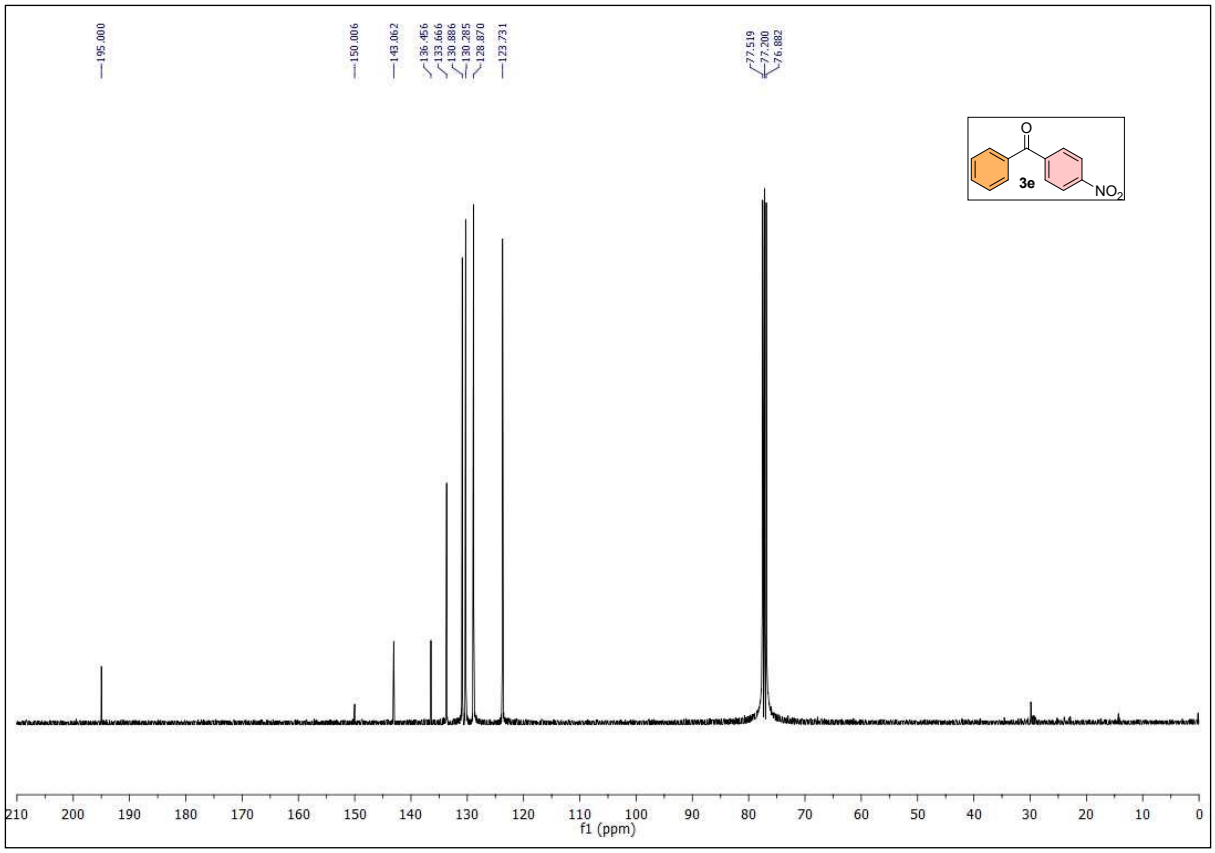

**Spectrum 8**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3d**



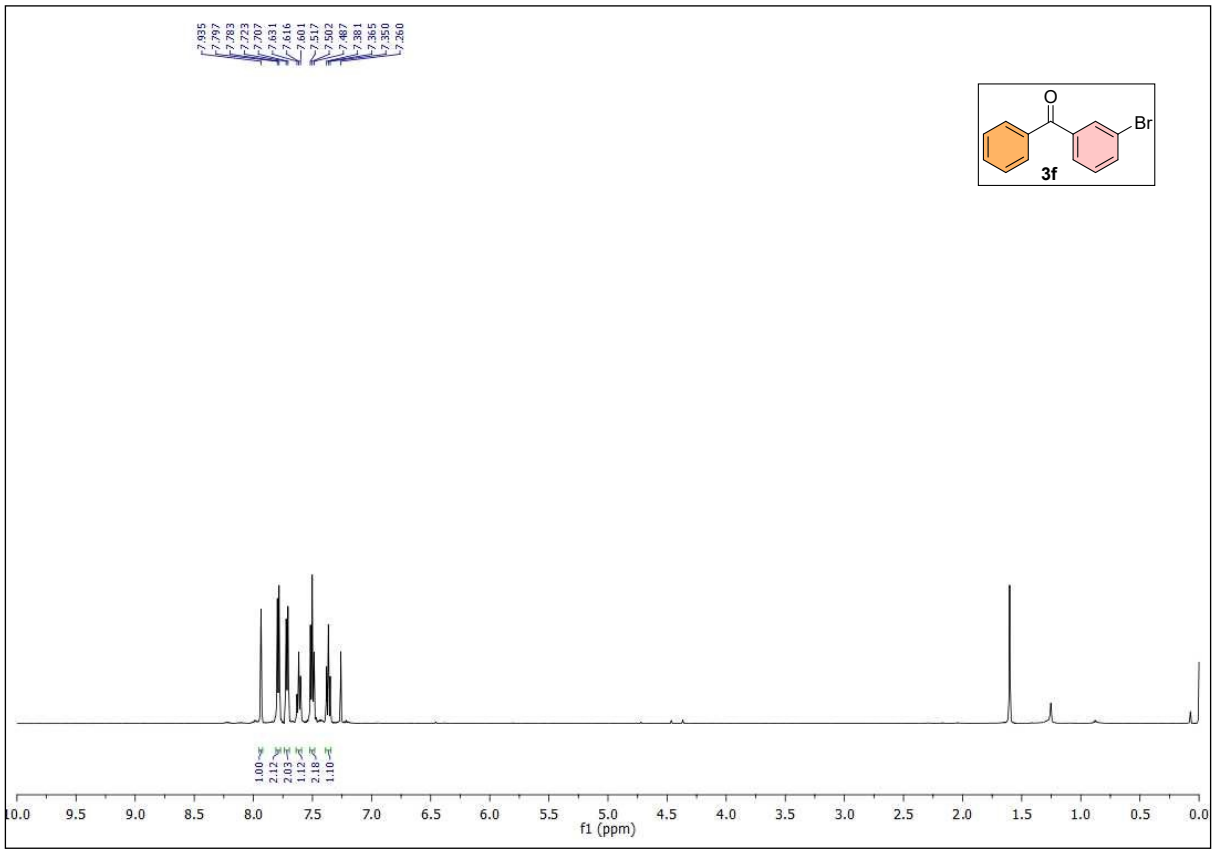

**Spectrum 9**. 376 MHz $^{19}F$ NMR ($CDCl_3$) of compound **3d**



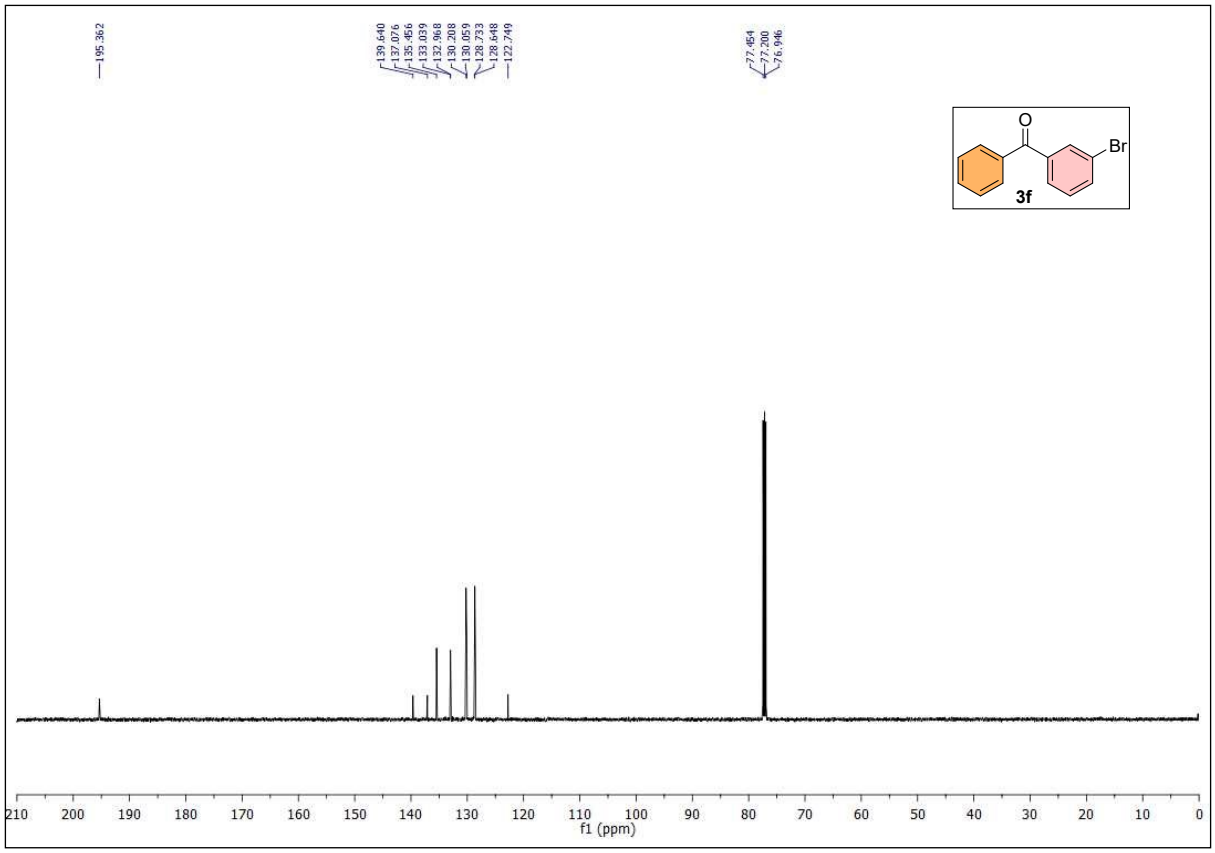

**Spectrum 10**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3e**



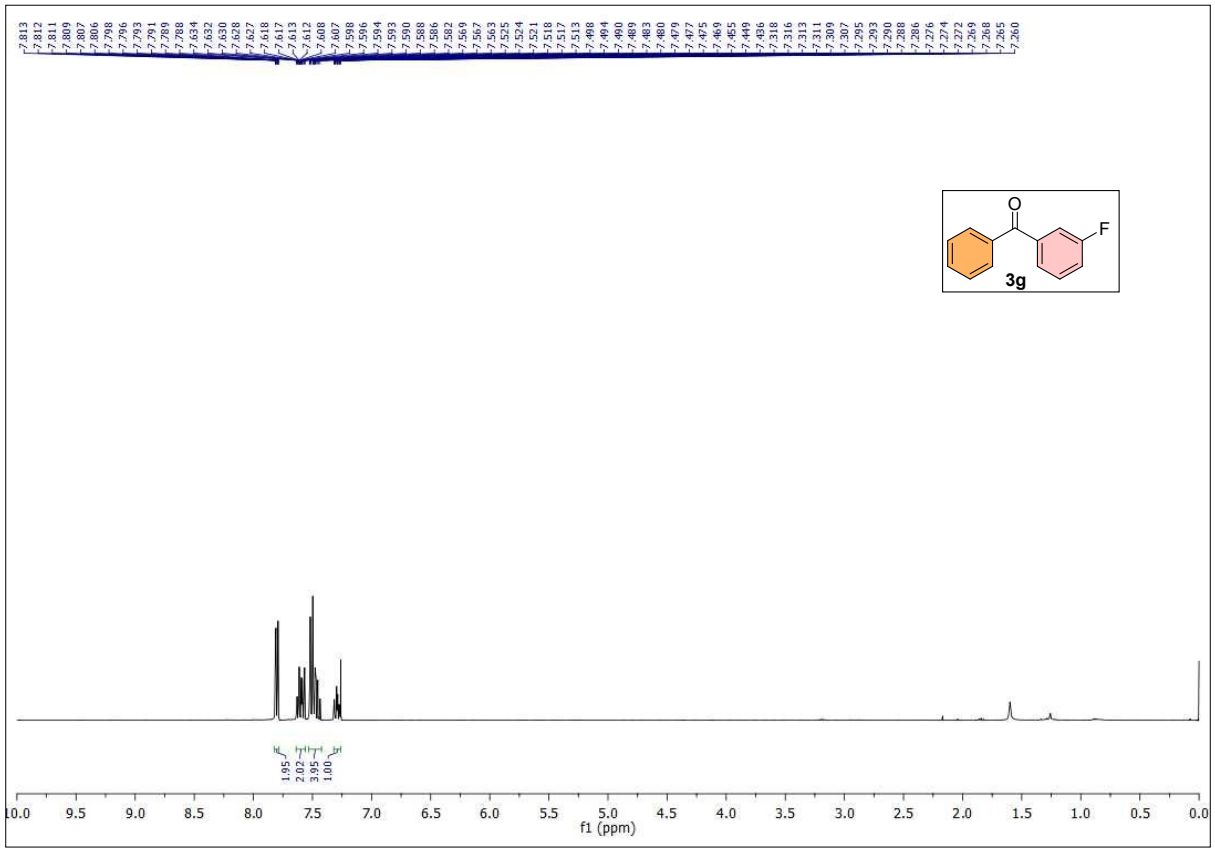

**Spectrum 11**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3e**



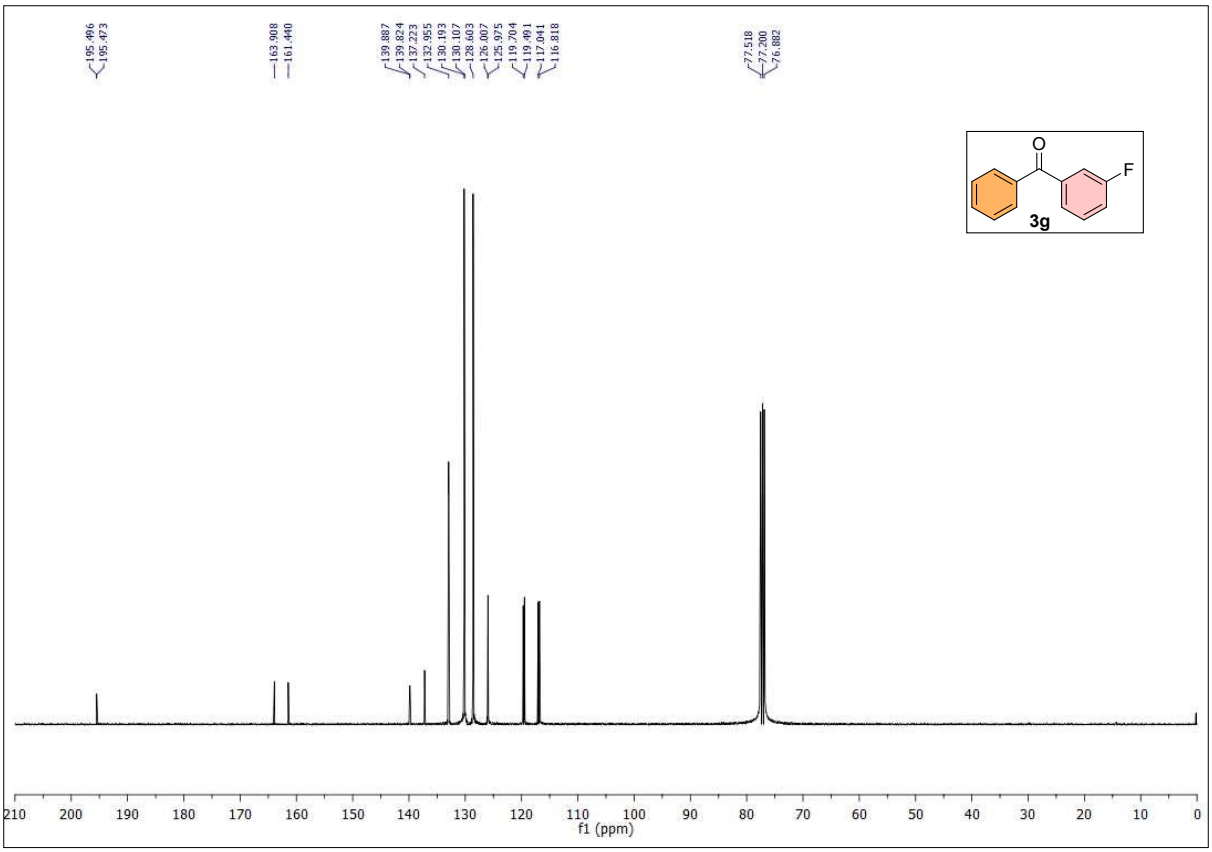

**Spectrum 12**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3f**



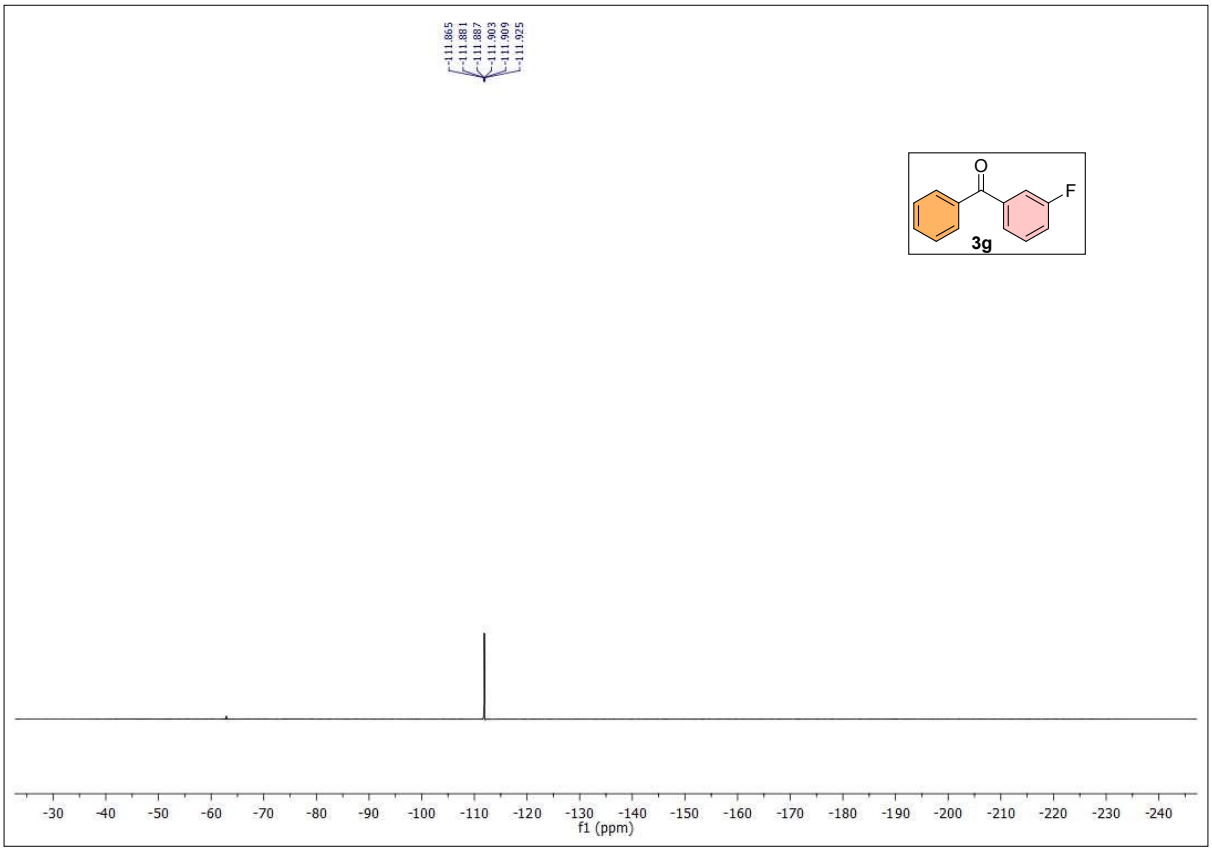

**Spectrum 13**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3f**



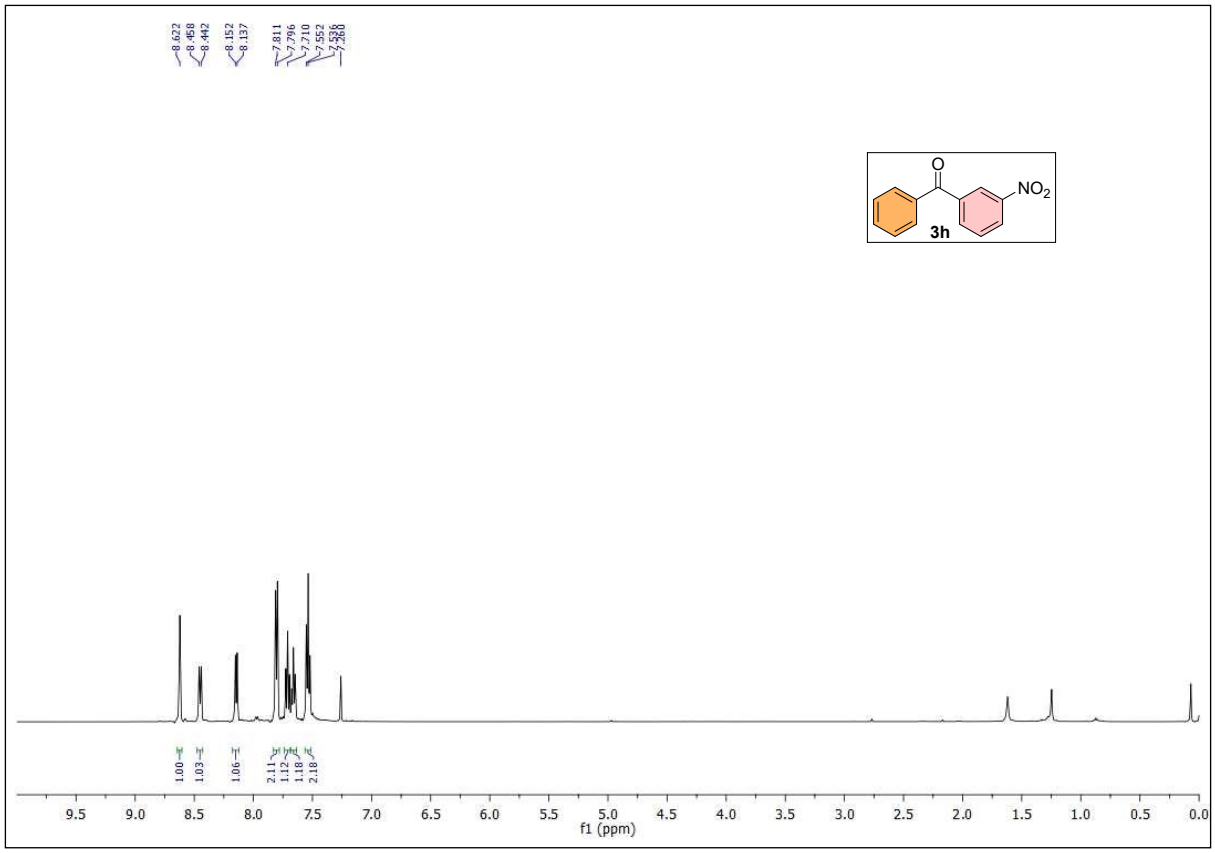

**Spectrum 14**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3g**



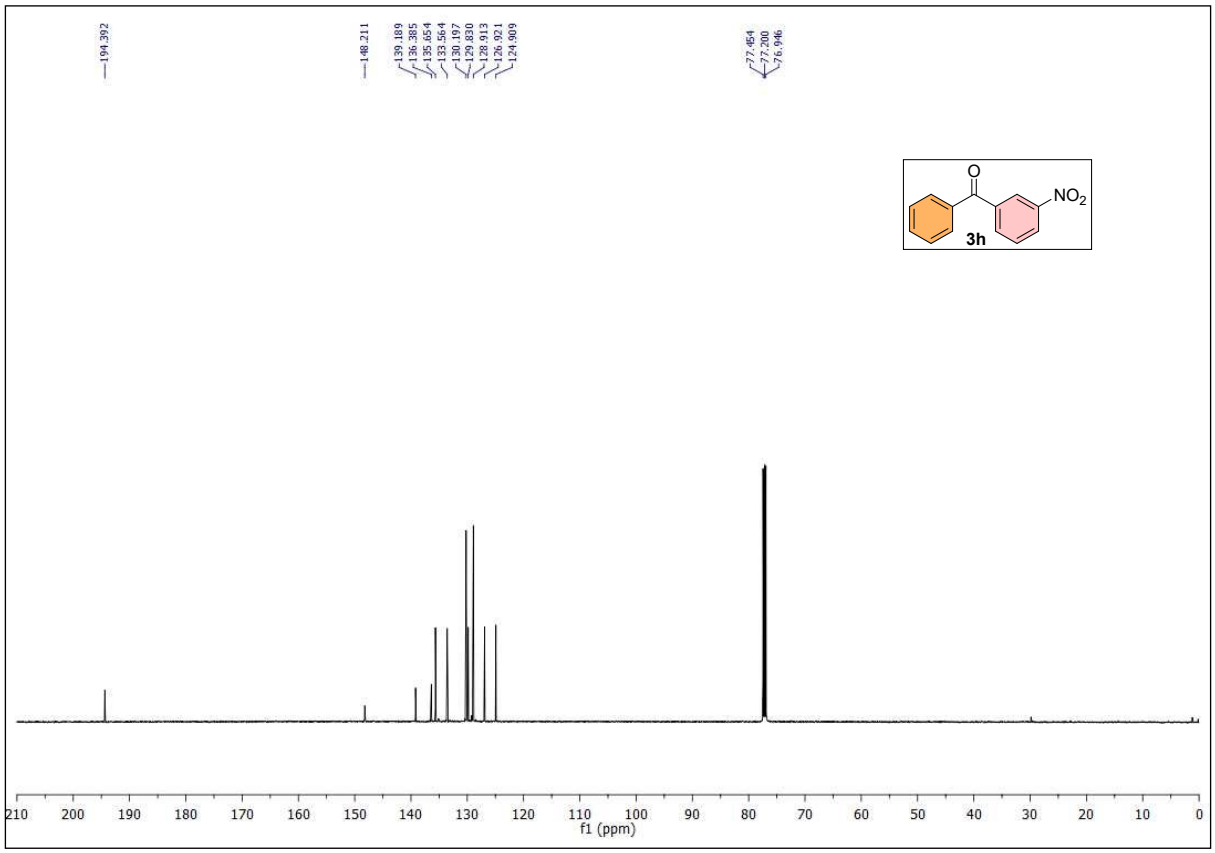

**Spectrum 15**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3g**



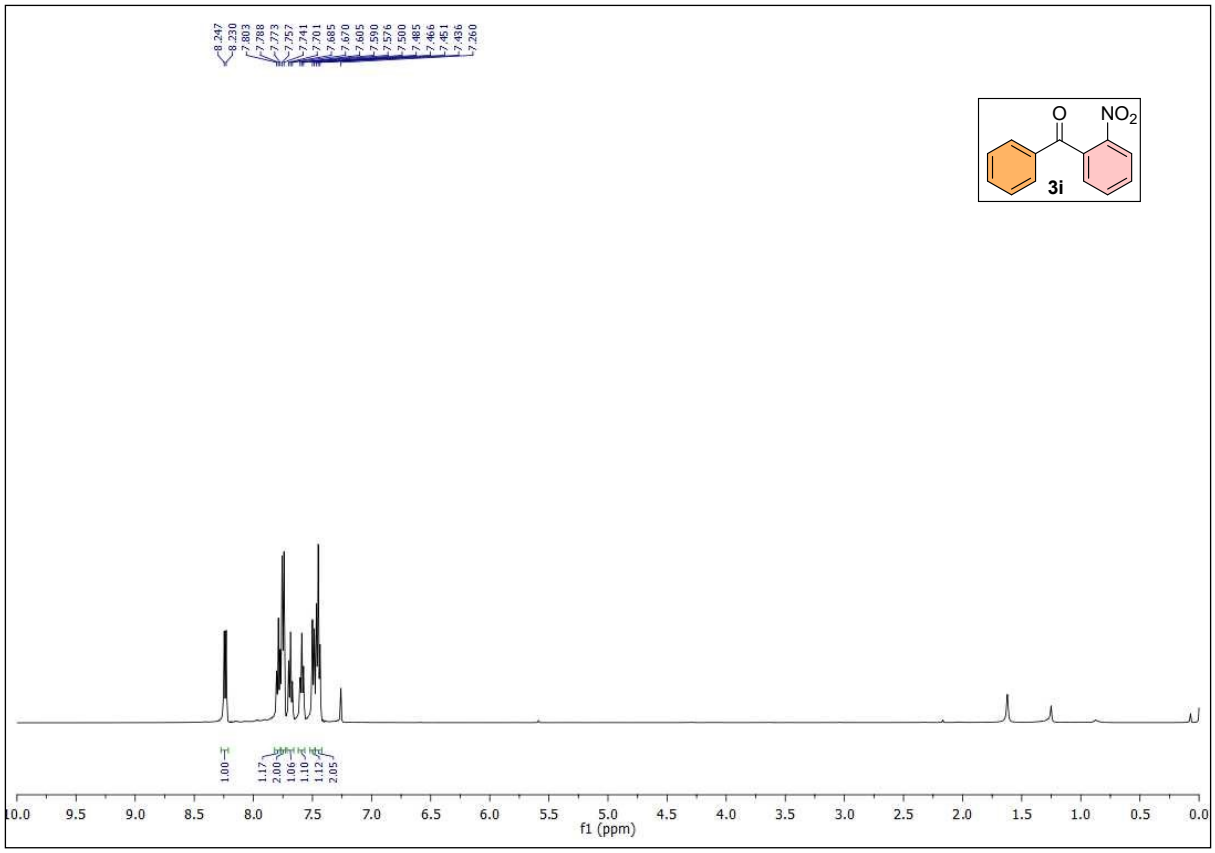

**Spectrum 16**. 376 MHz $^{19}F$ NMR ($CDCl_3$) of compound **3g**



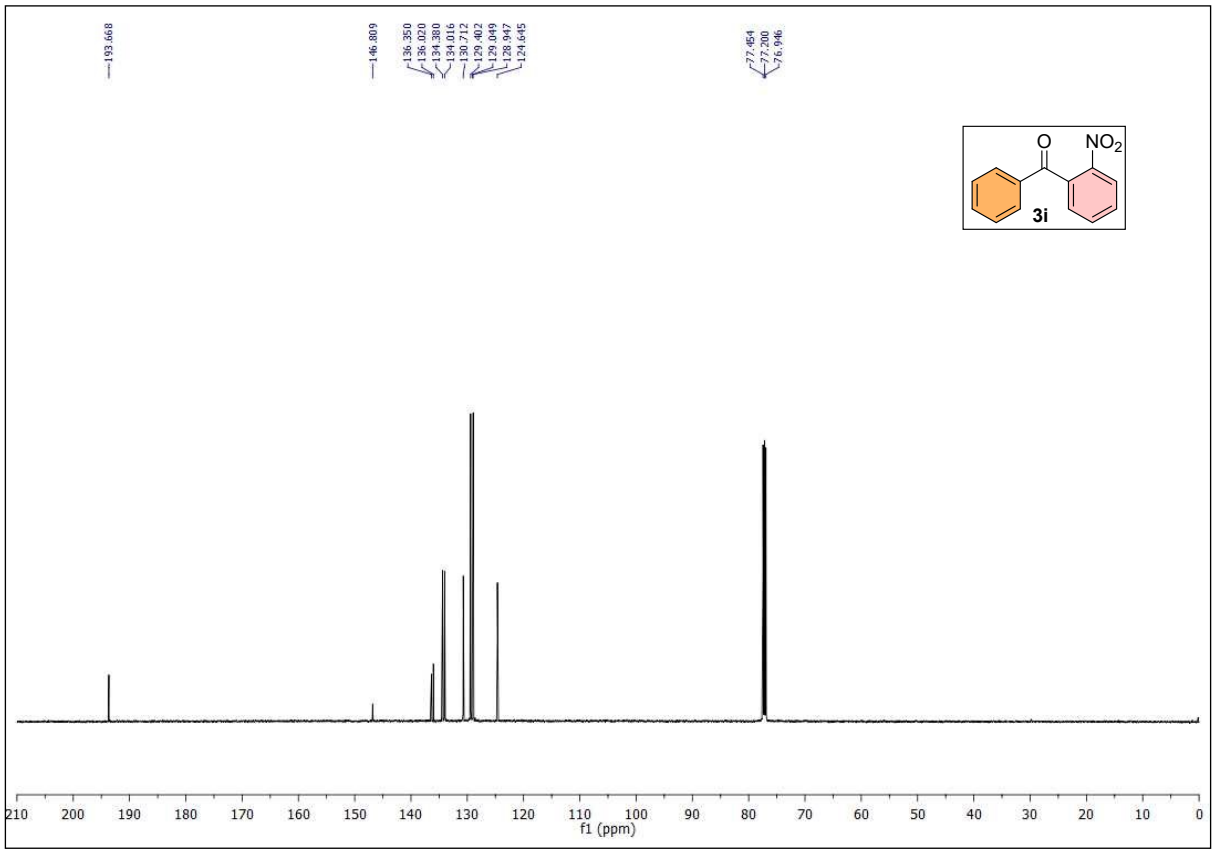

**Spectrum 17**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3h**



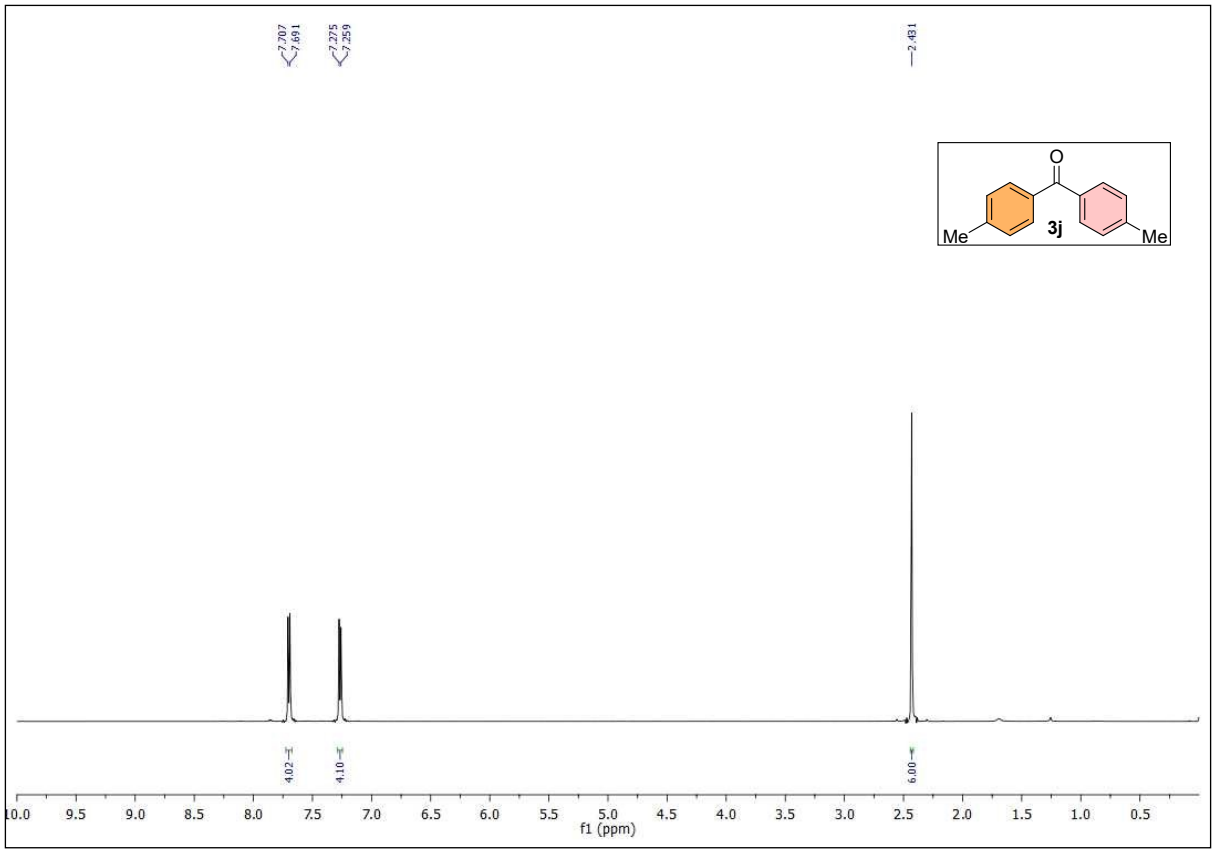

**Spectrum 18**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3h**



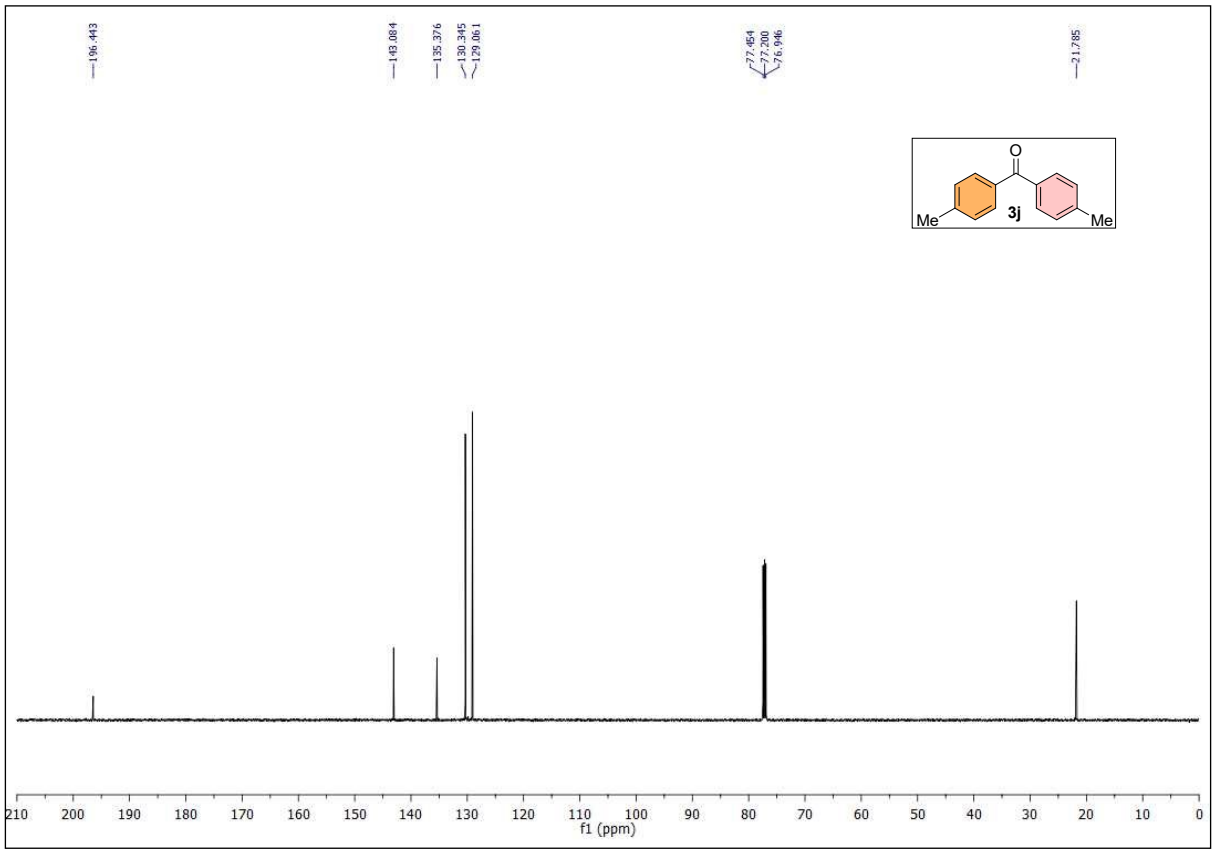

**Spectrum 19**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3i**



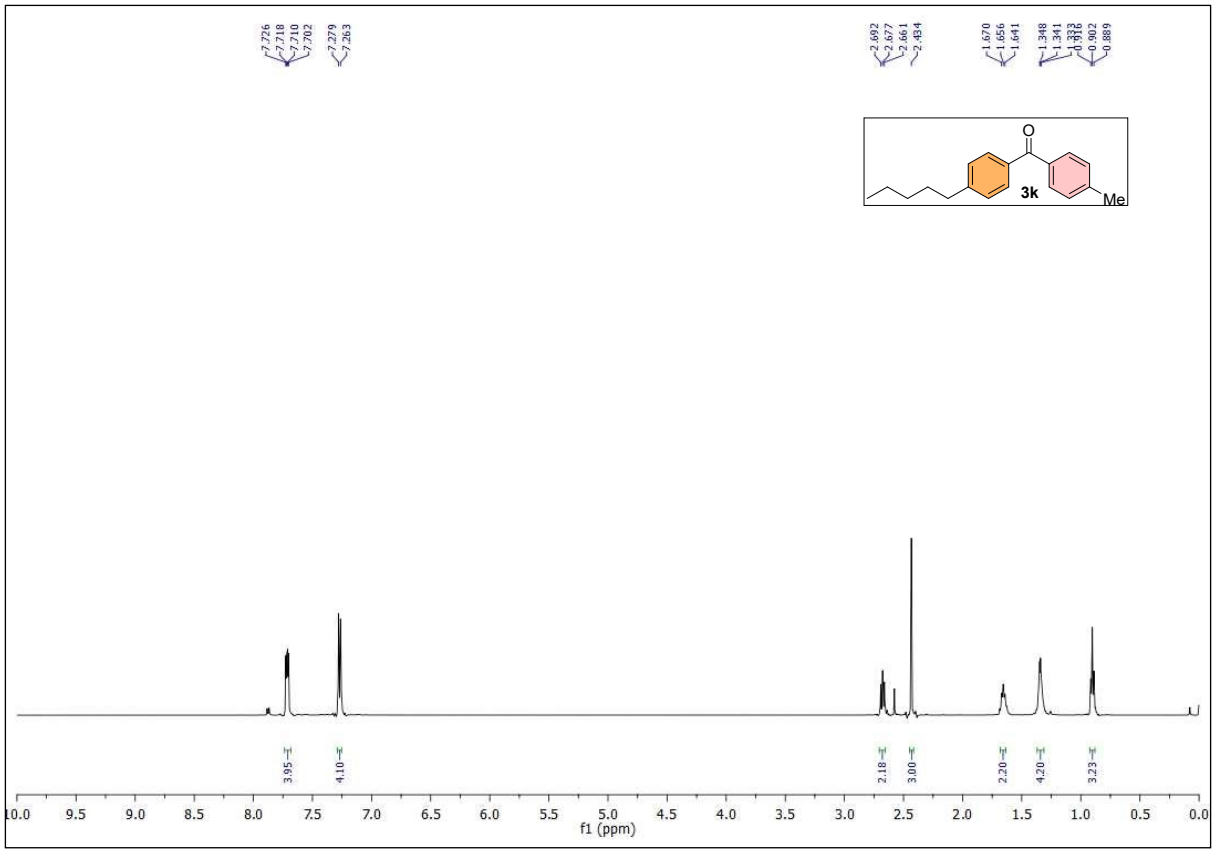

**Spectrum 20**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3i**



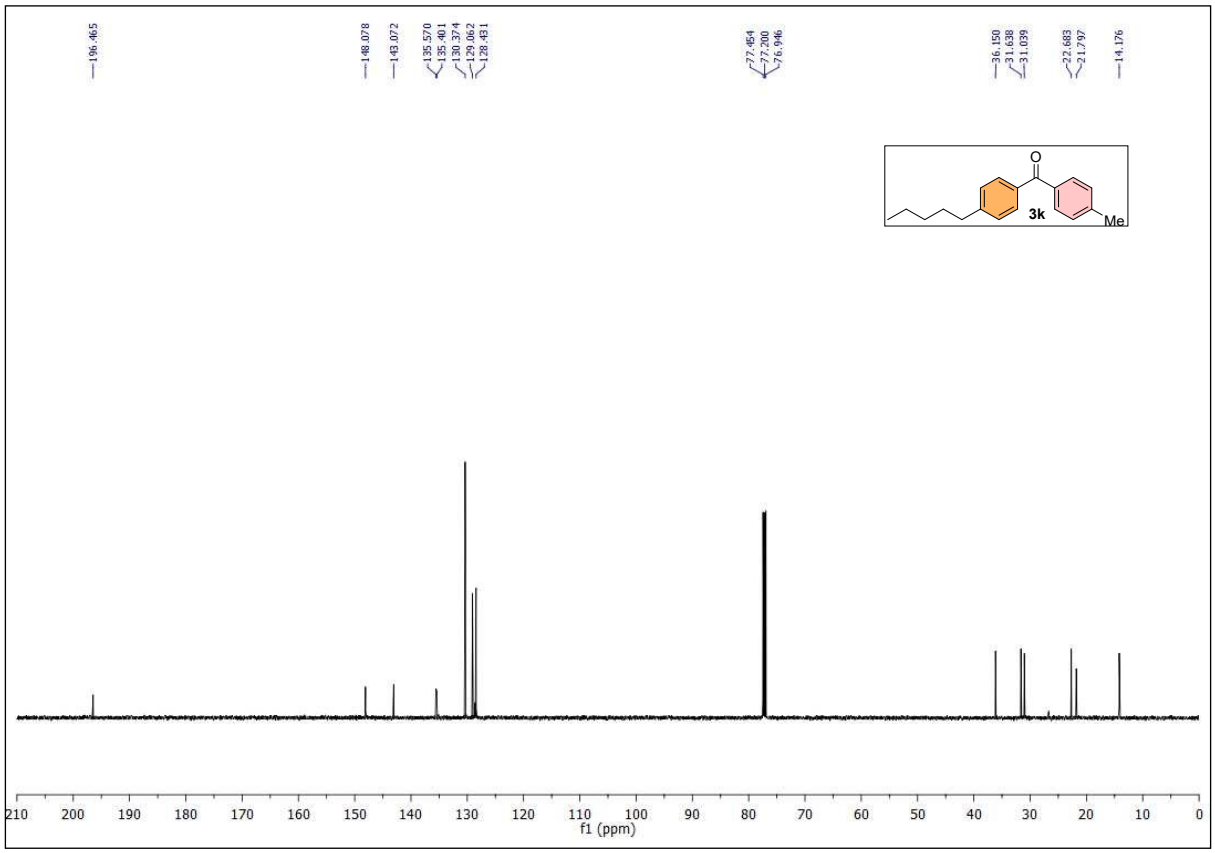

**Spectrum 21**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3j**



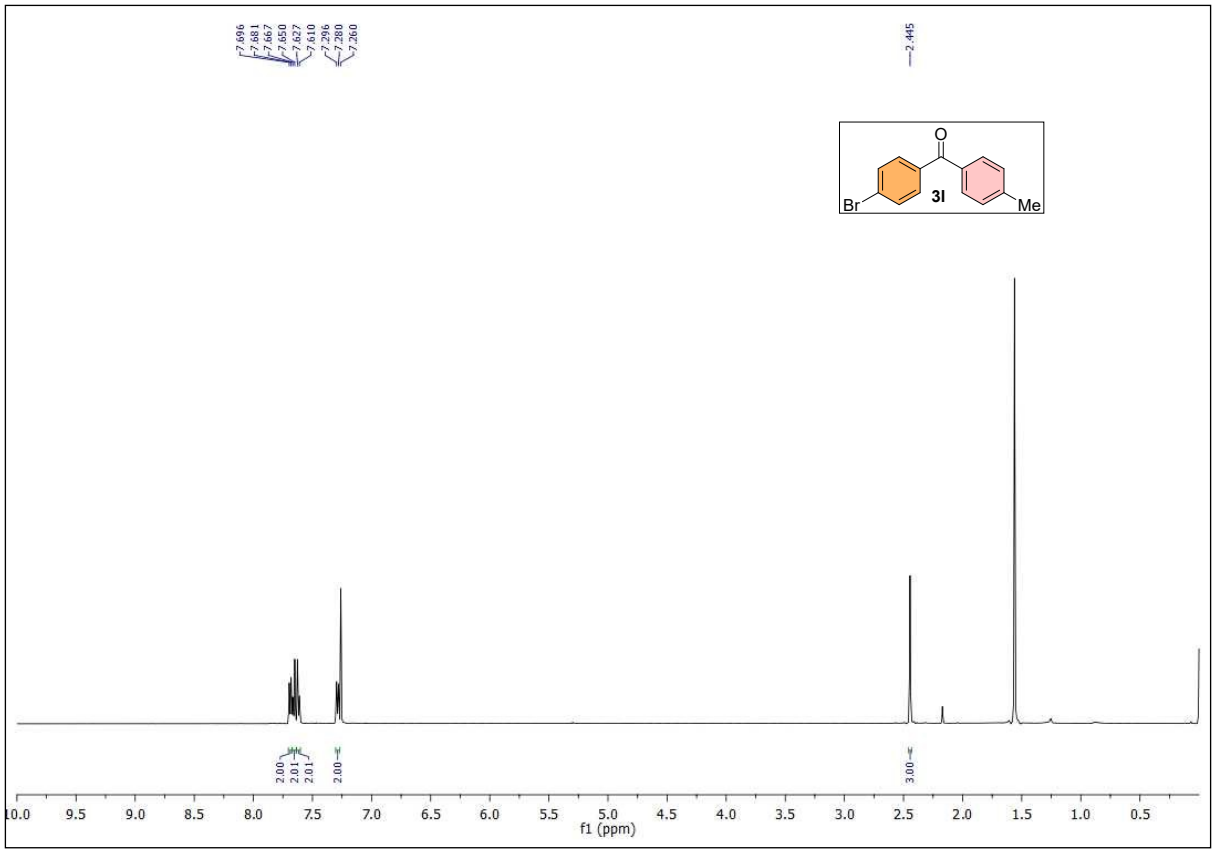

**Spectrum 22**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3j**



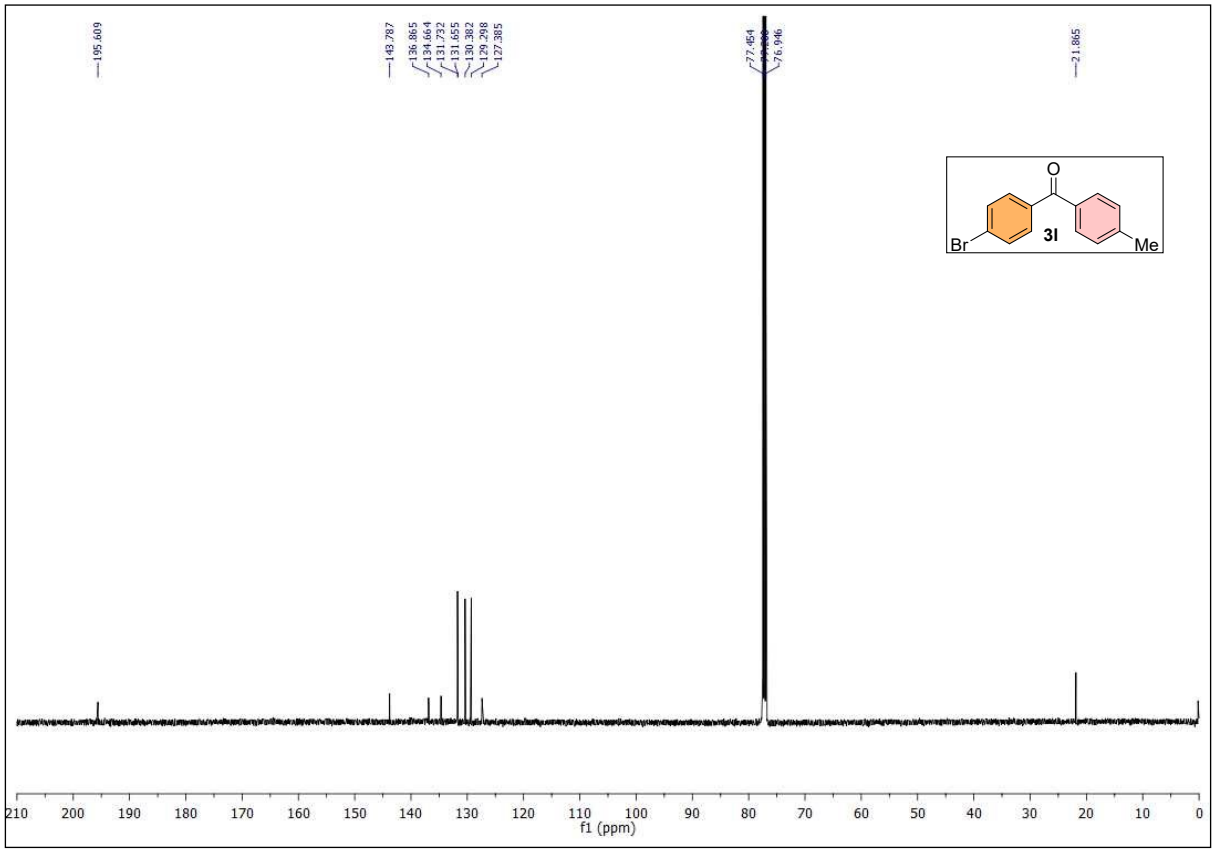

**Spectrum 23**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3k**



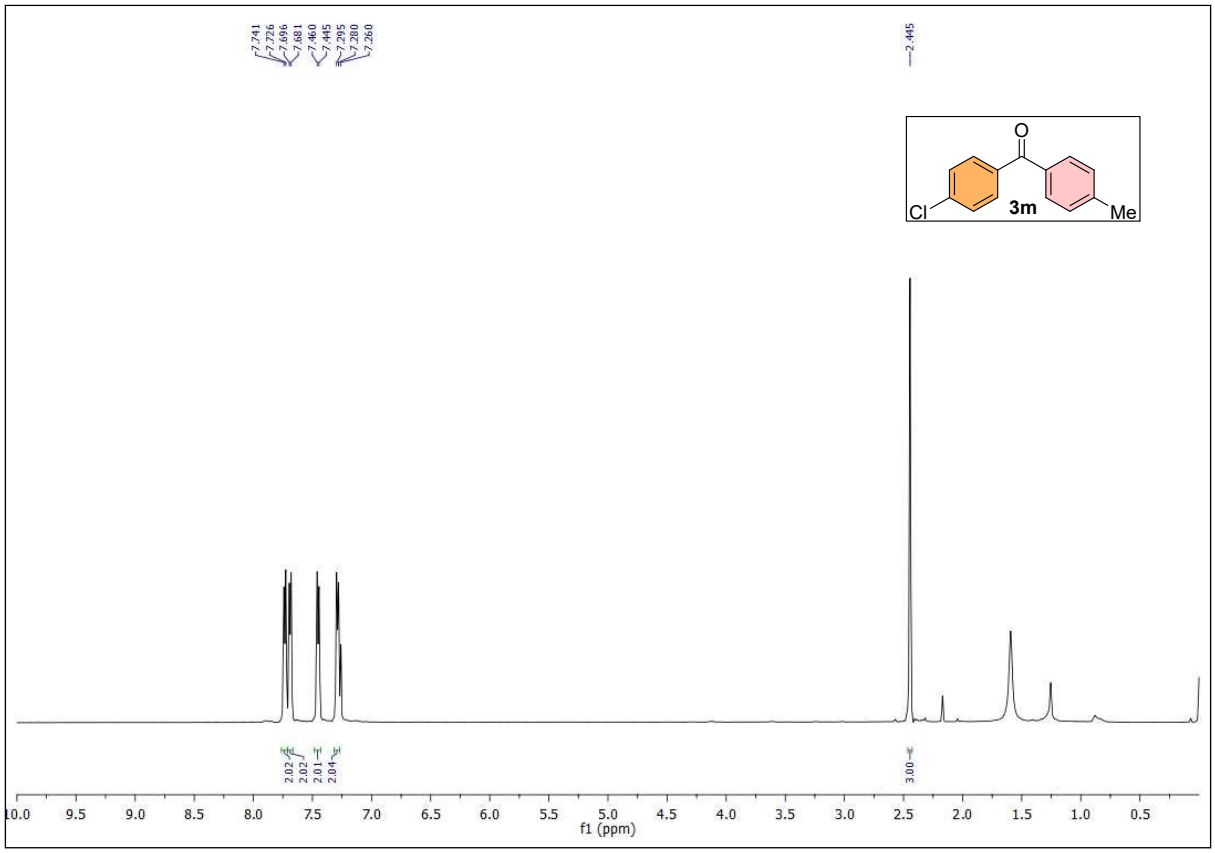

**Spectrum 24**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3k**



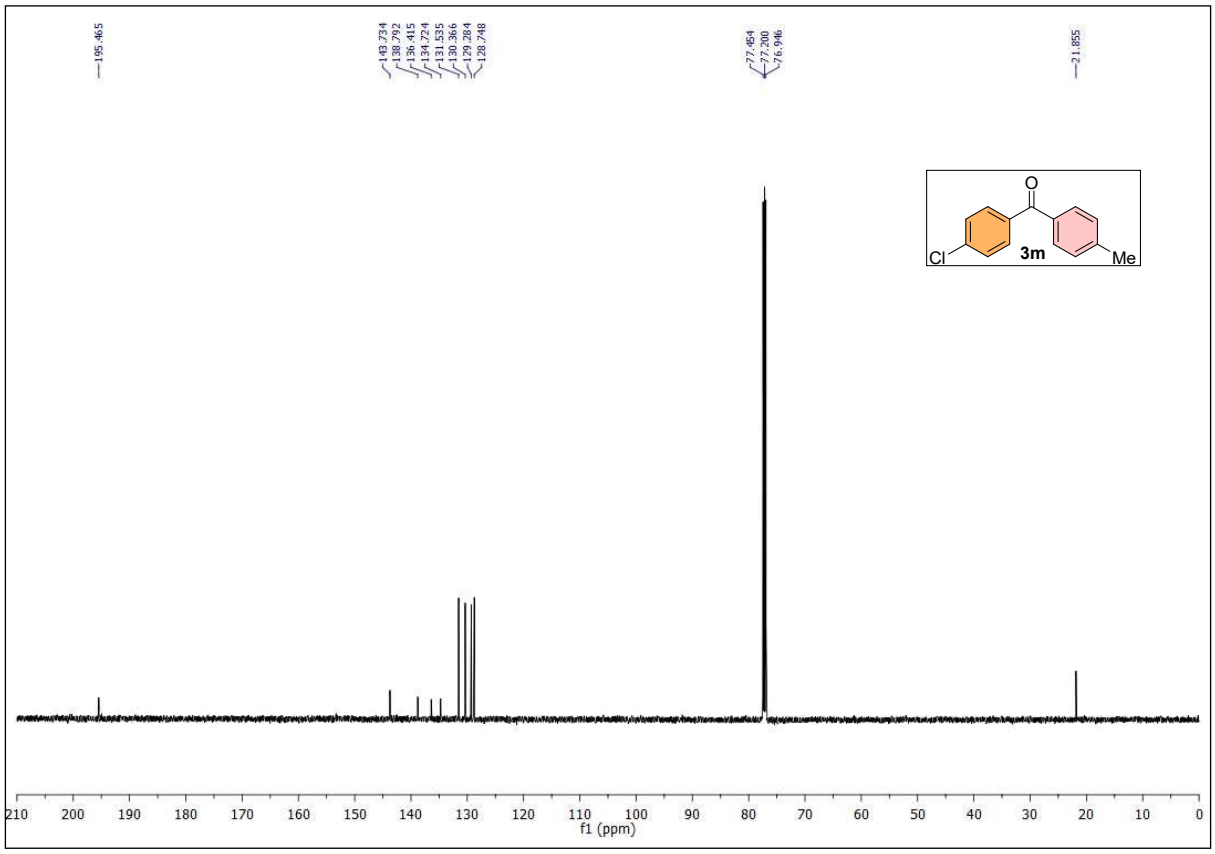

**Spectrum 25**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3l**



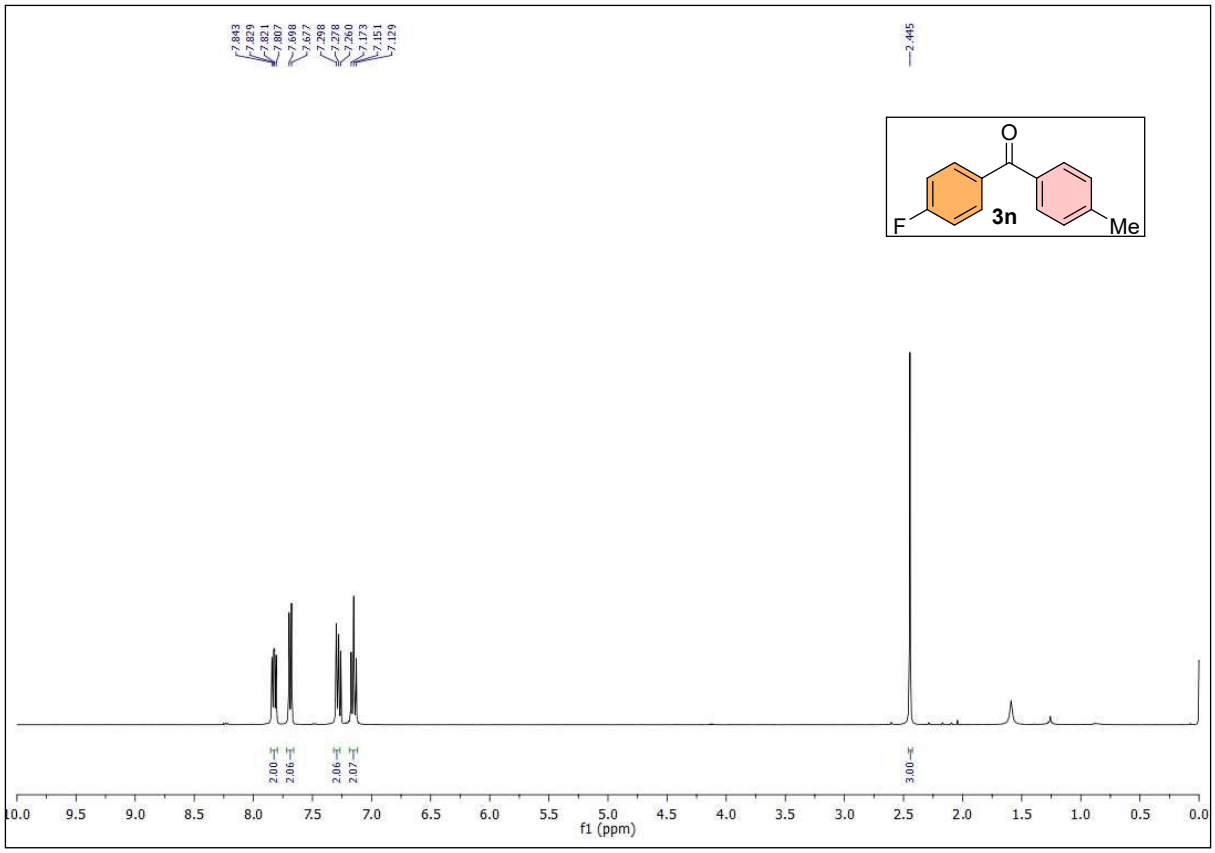

**Spectrum 26**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3l**



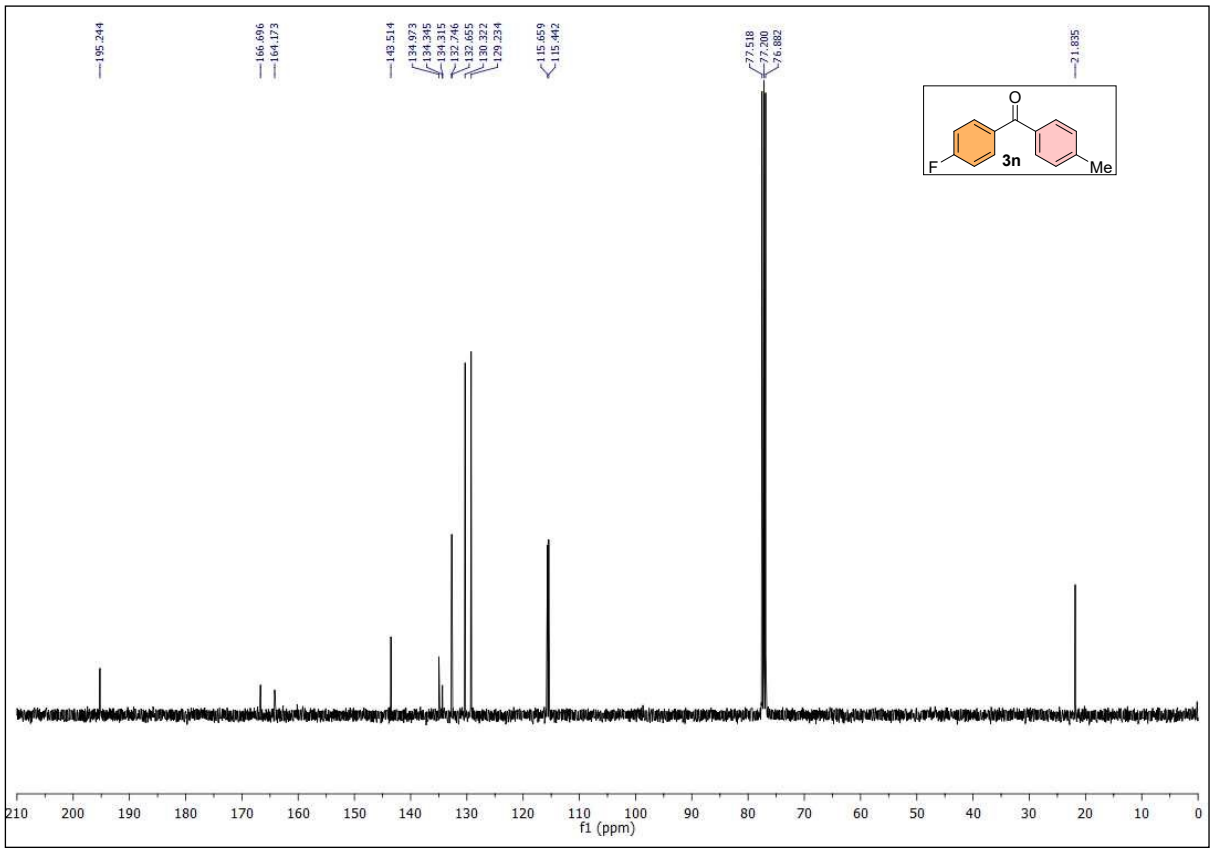

**Spectrum 27**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3m**



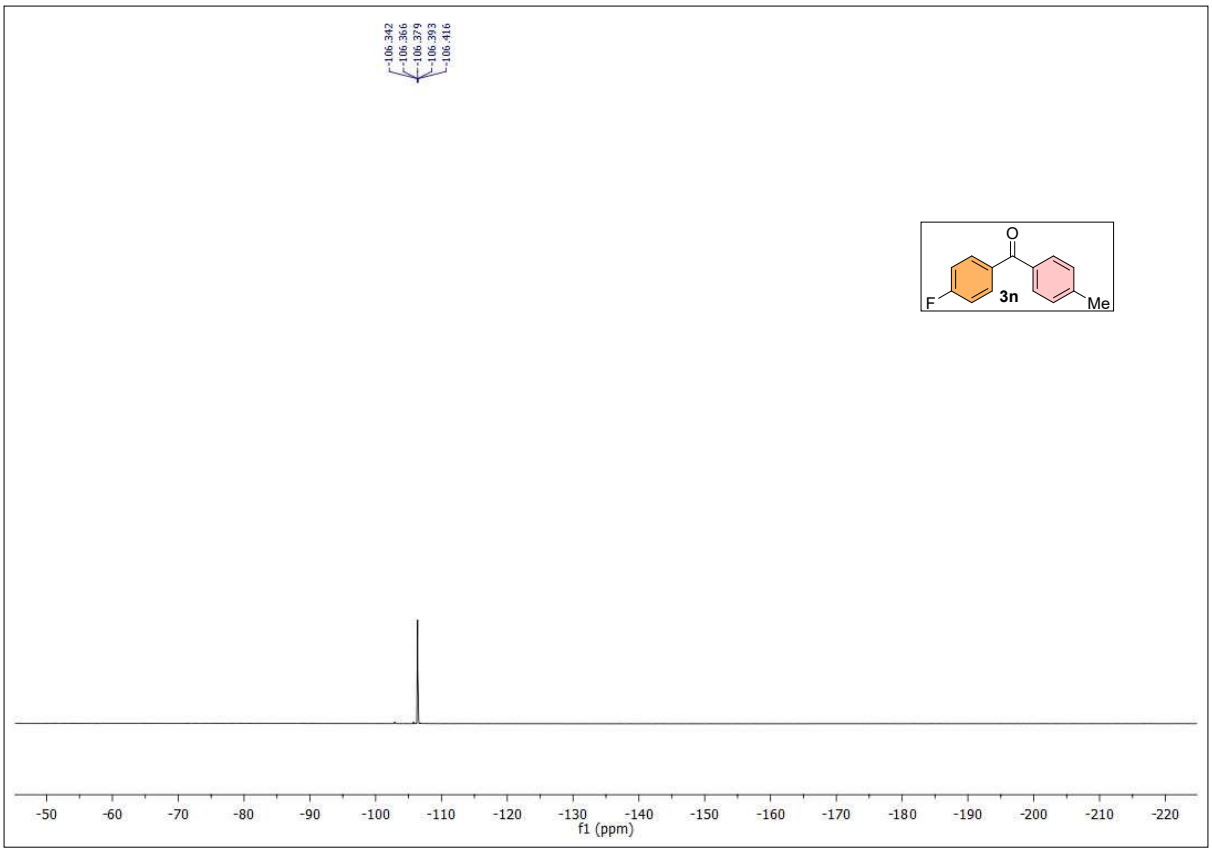

**Spectrum 28**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3m**



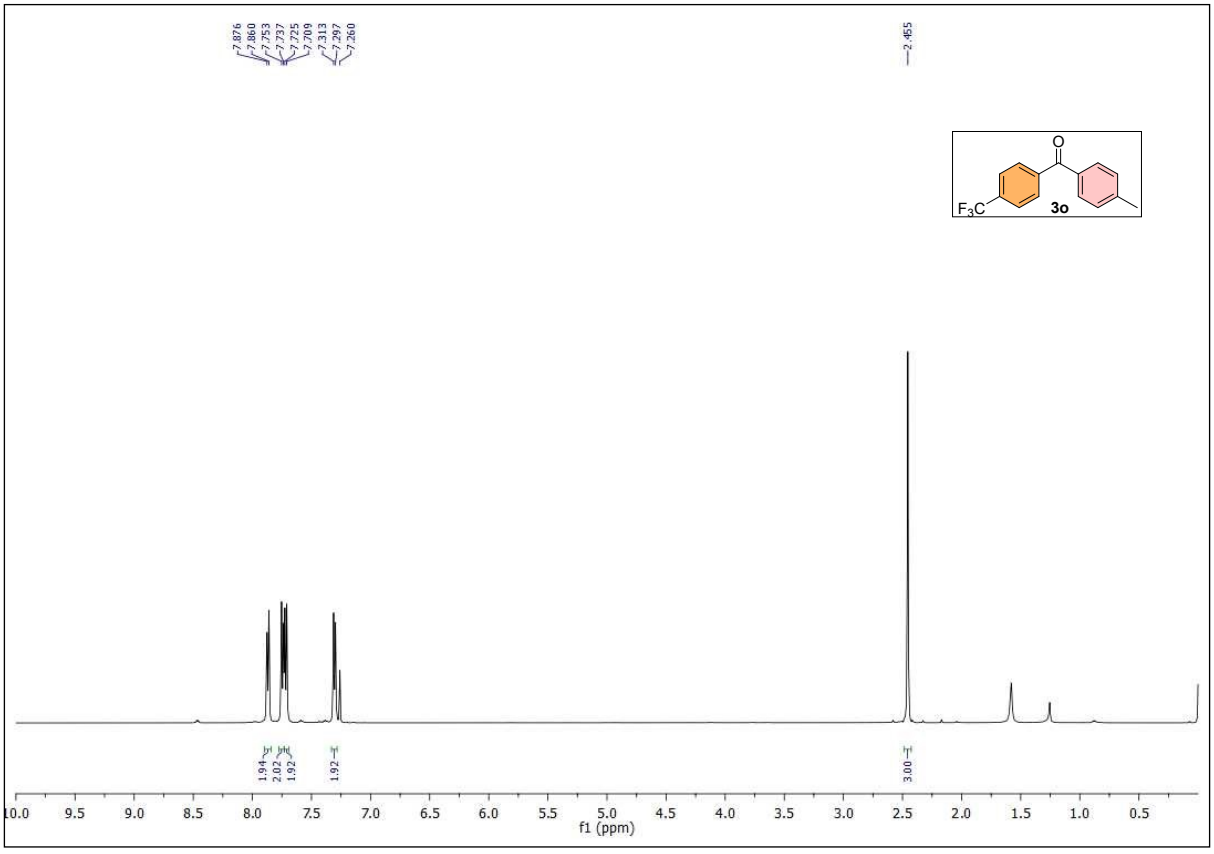

**Spectrum 29**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3n**



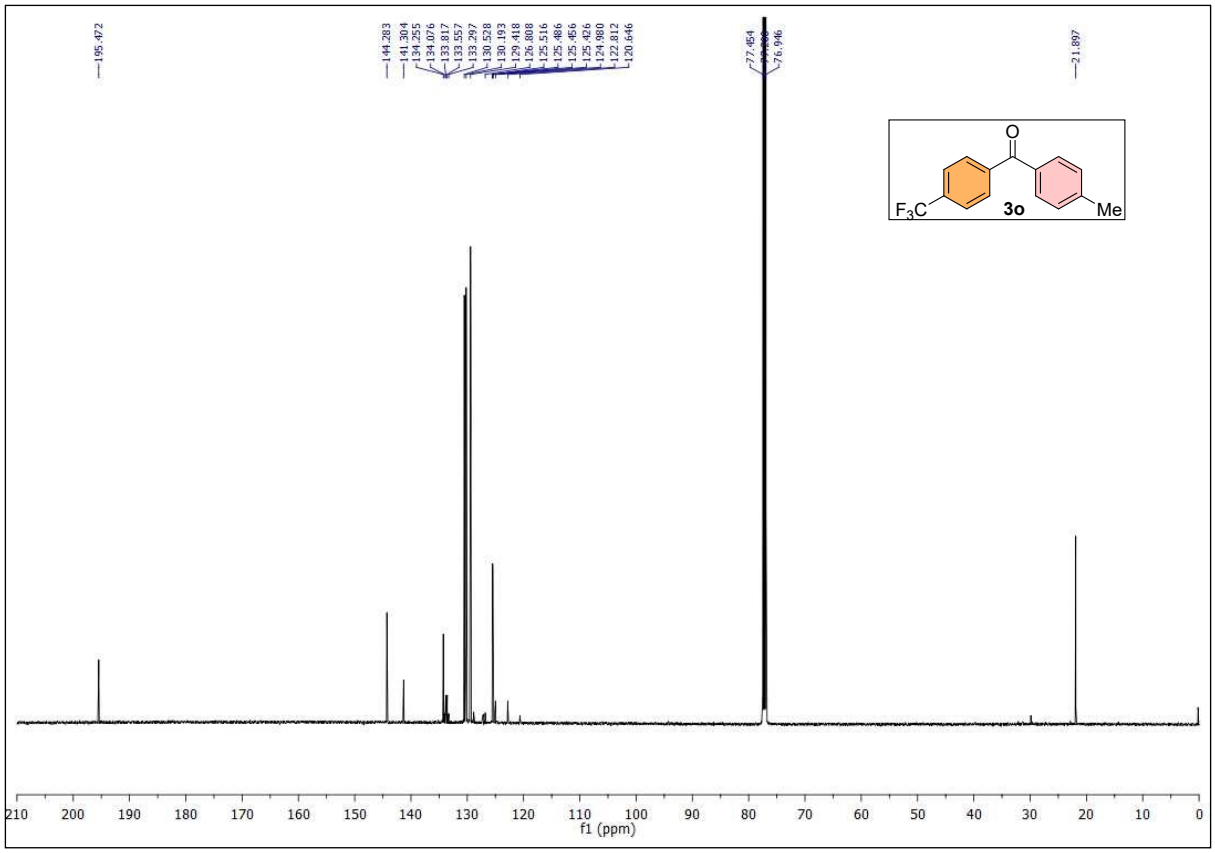

**Spectrum 30**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3n**



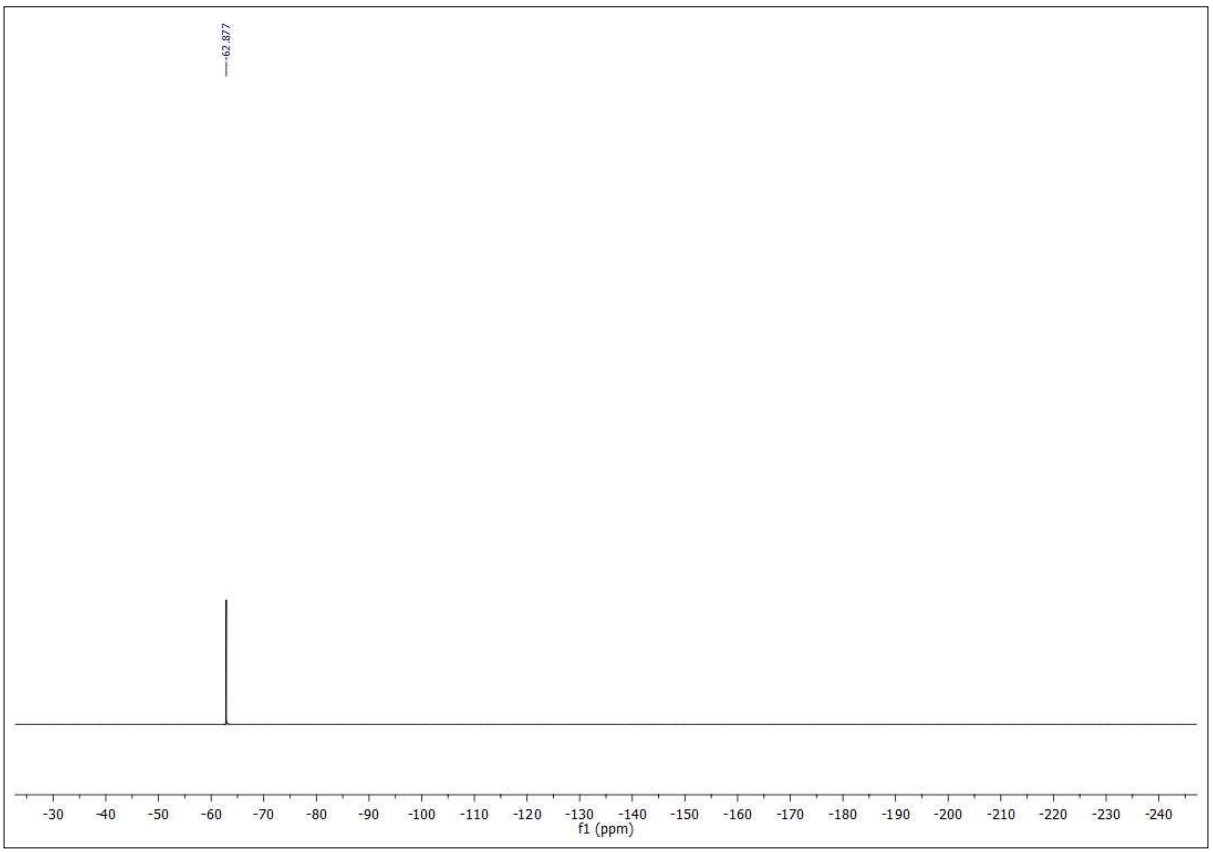

**Spectrum 31**. 376 MHz $^{19}F$ NMR ($CDCl_3$) of compound **3n**



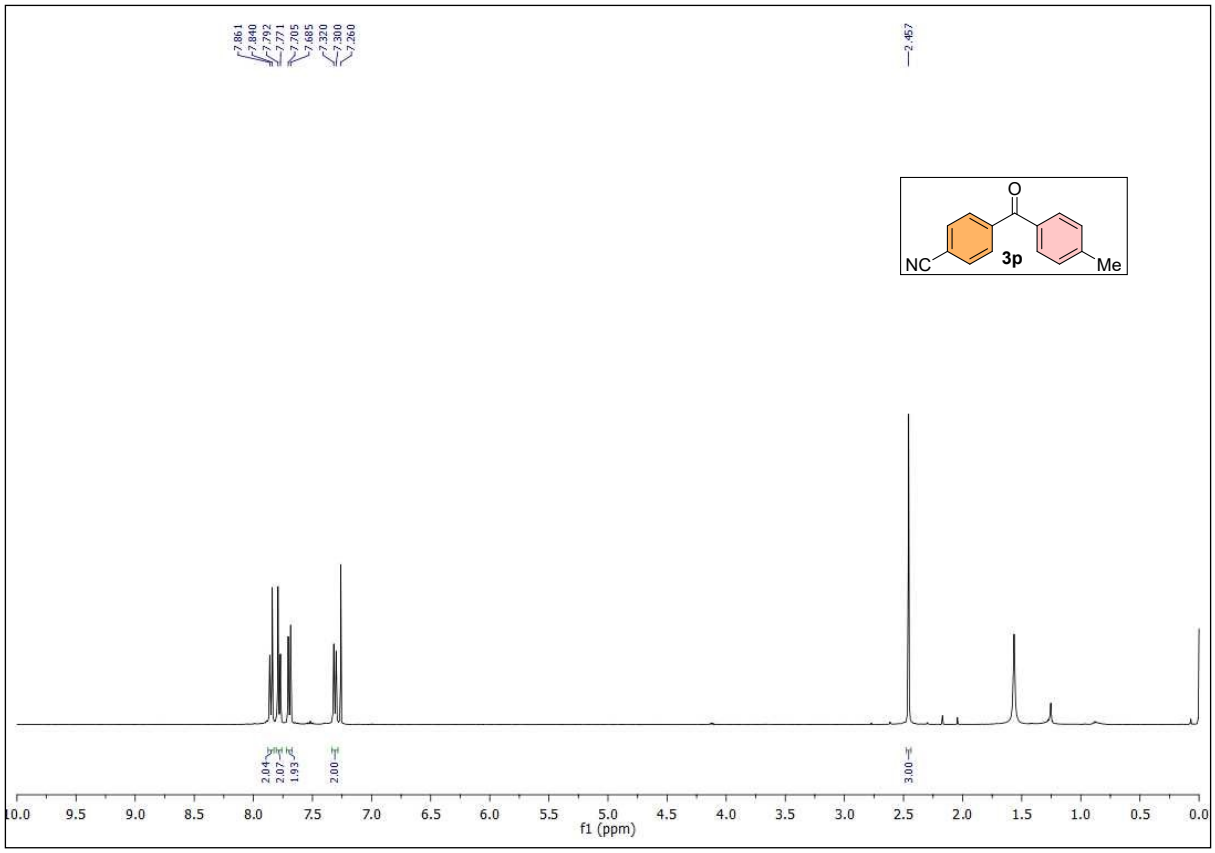

**Spectrum 32**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3o**



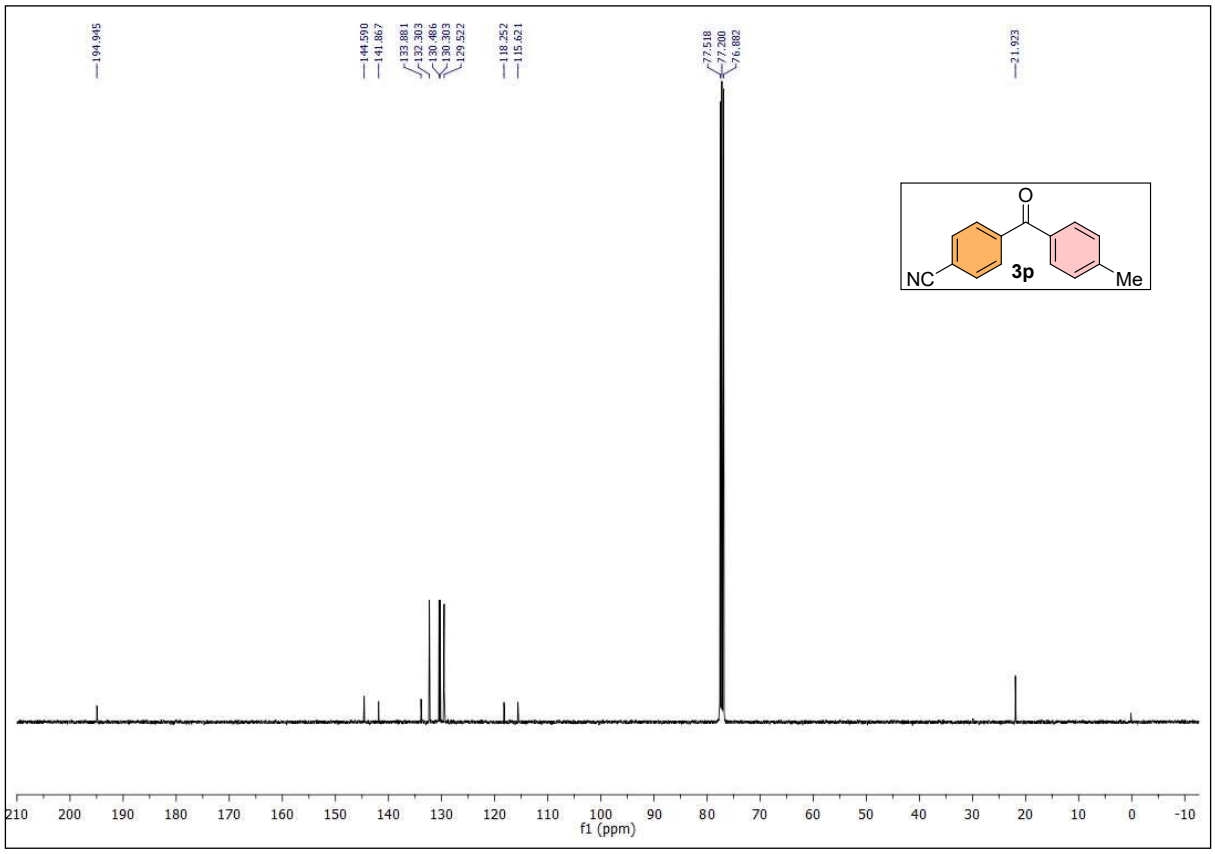

**Spectrum 33**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3o**



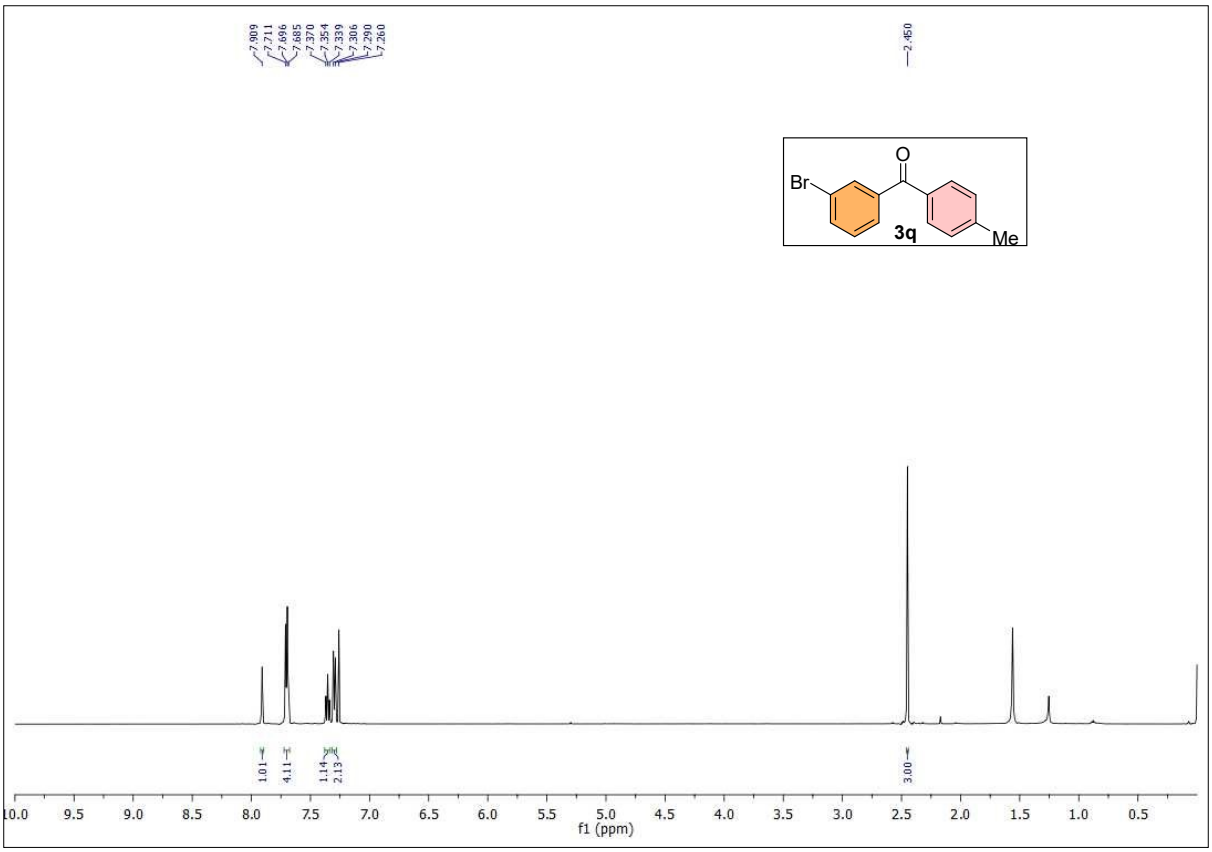

**Spectrum 34**. 376 MHz $^{19}F$ NMR ($CDCl_3$) of compound **3o**



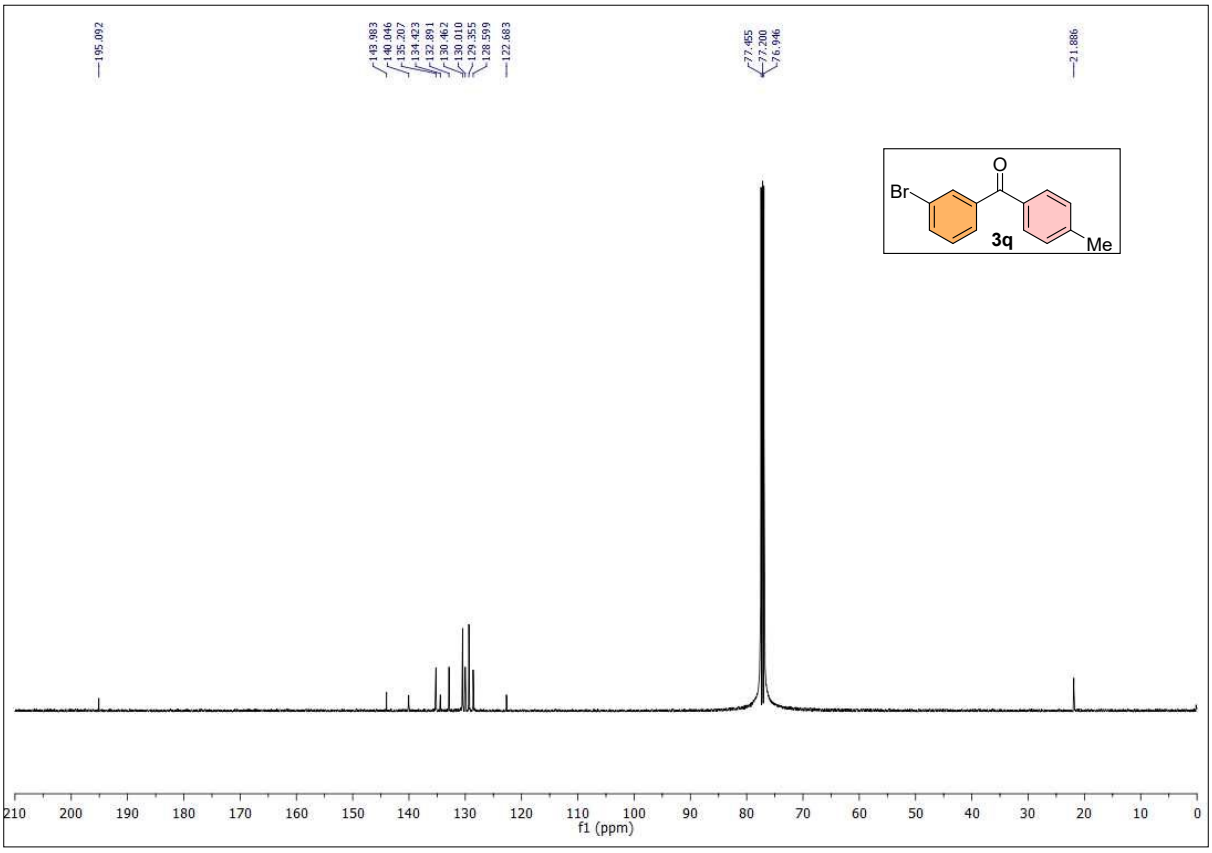

**Spectrum 35**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3p**



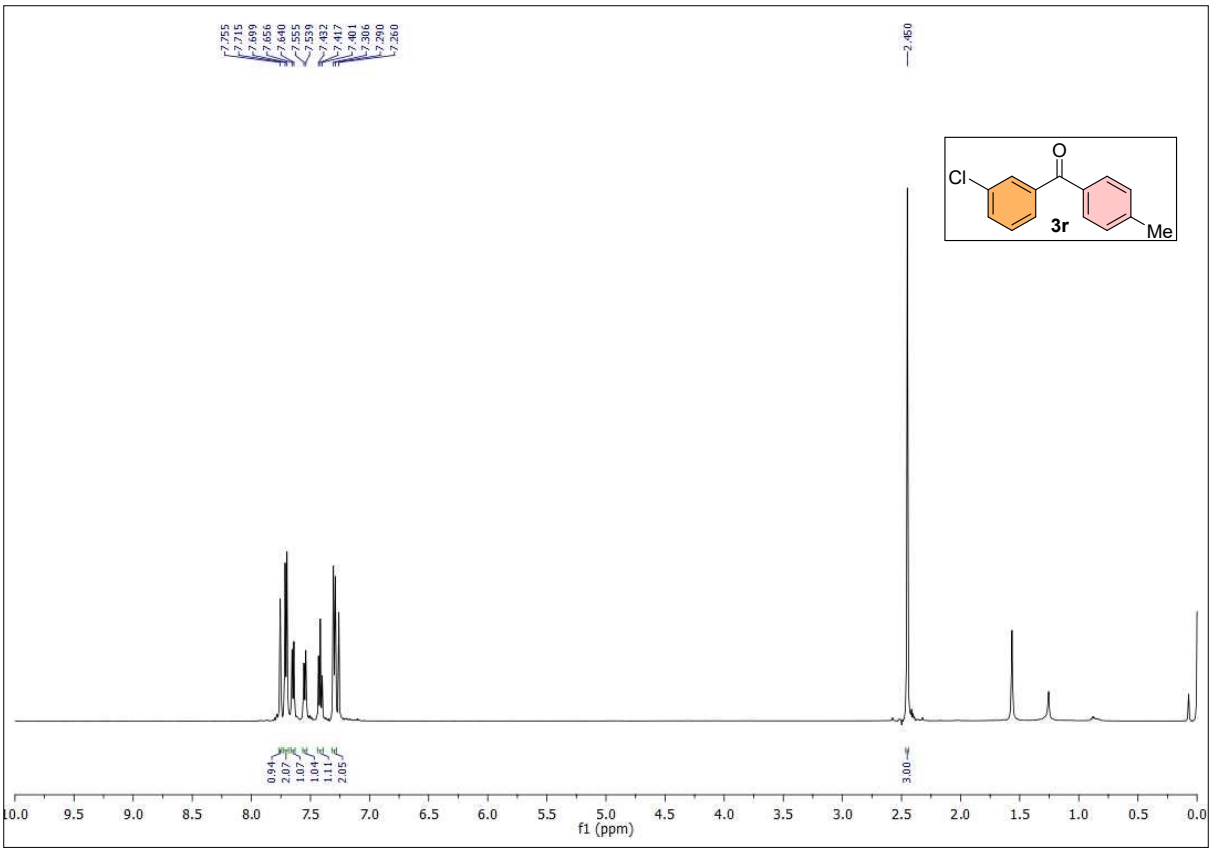

**Spectrum 36**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3p**



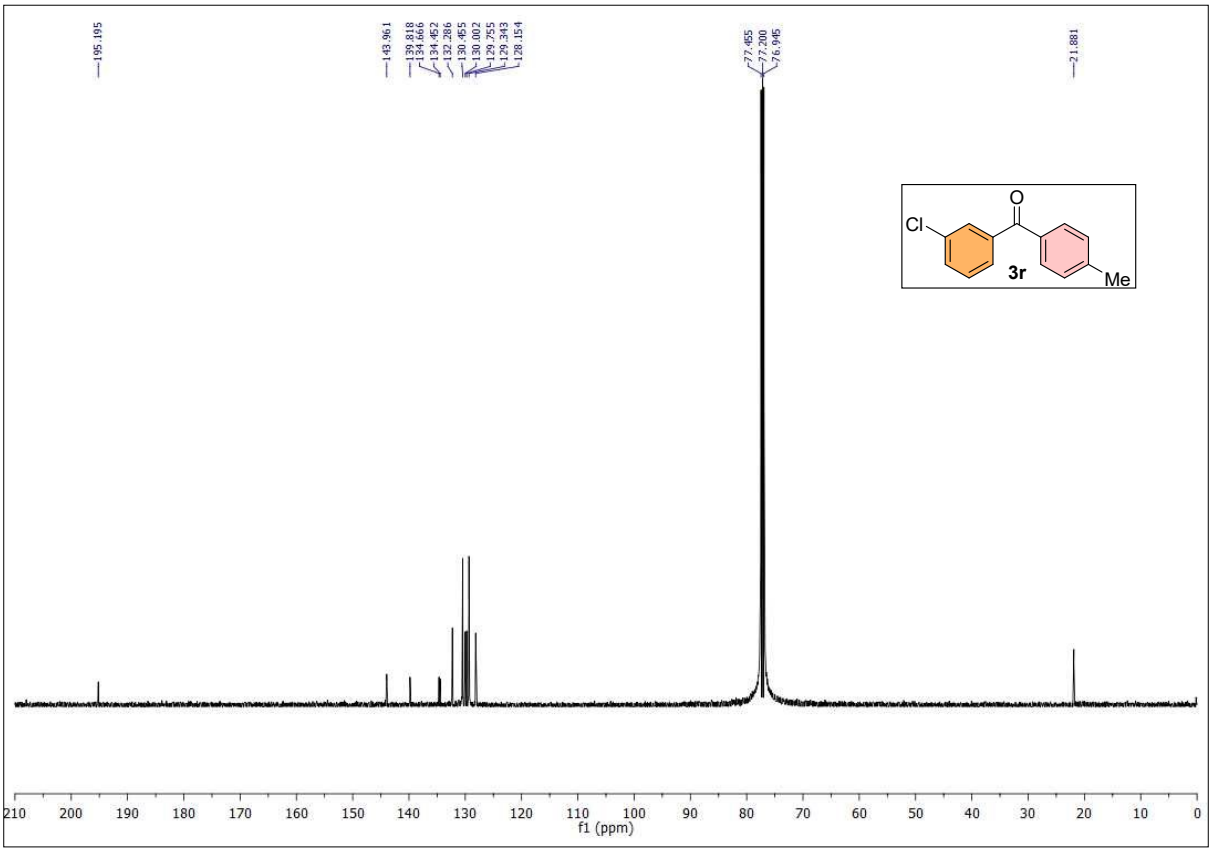

**Spectrum 37**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3q**



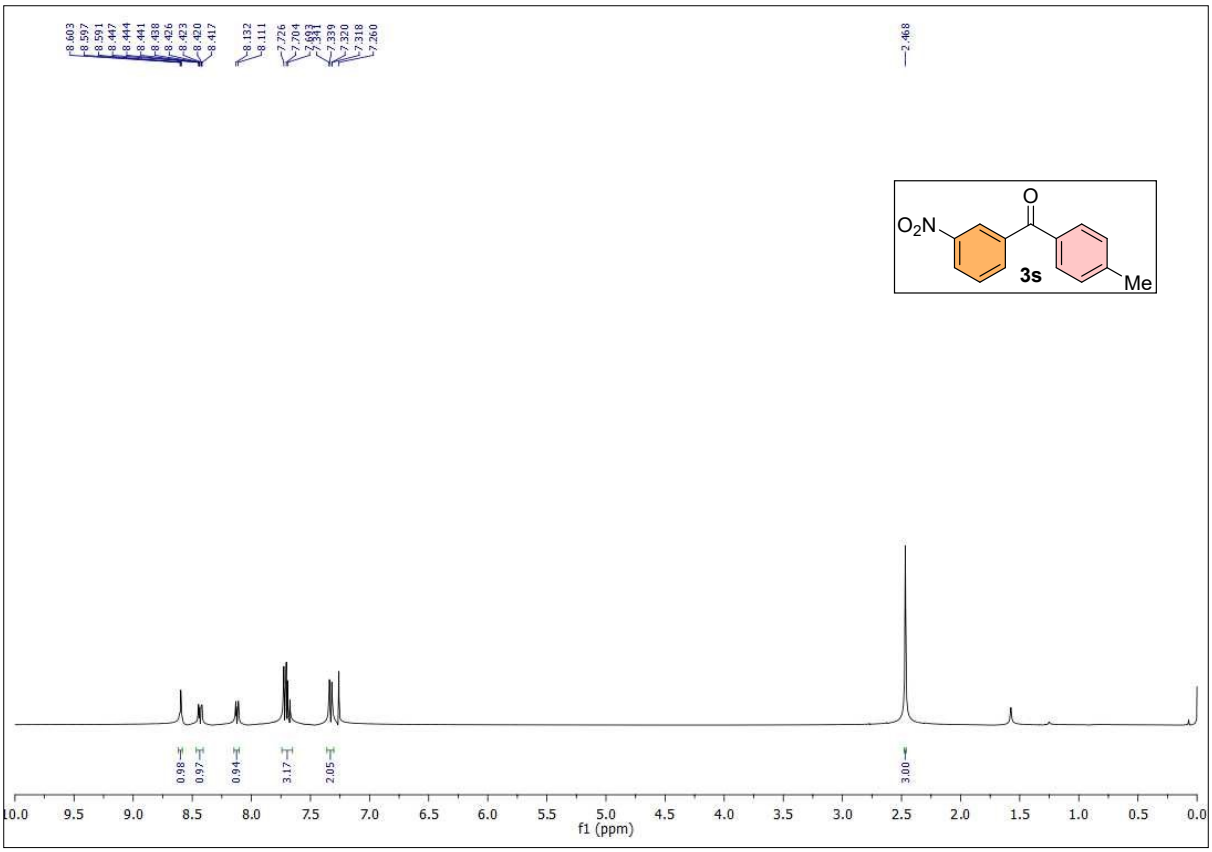

**Spectrum 38**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3q**



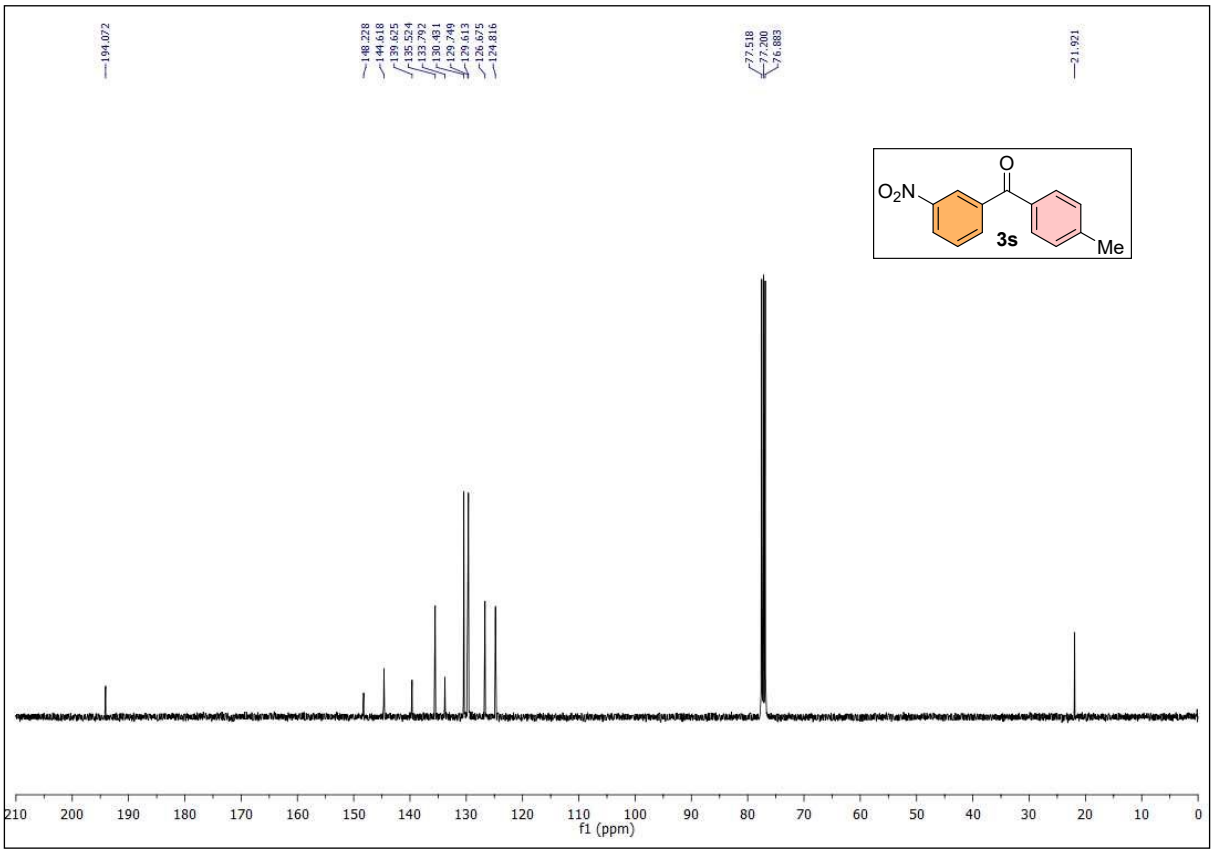

**Spectrum 39**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3r**



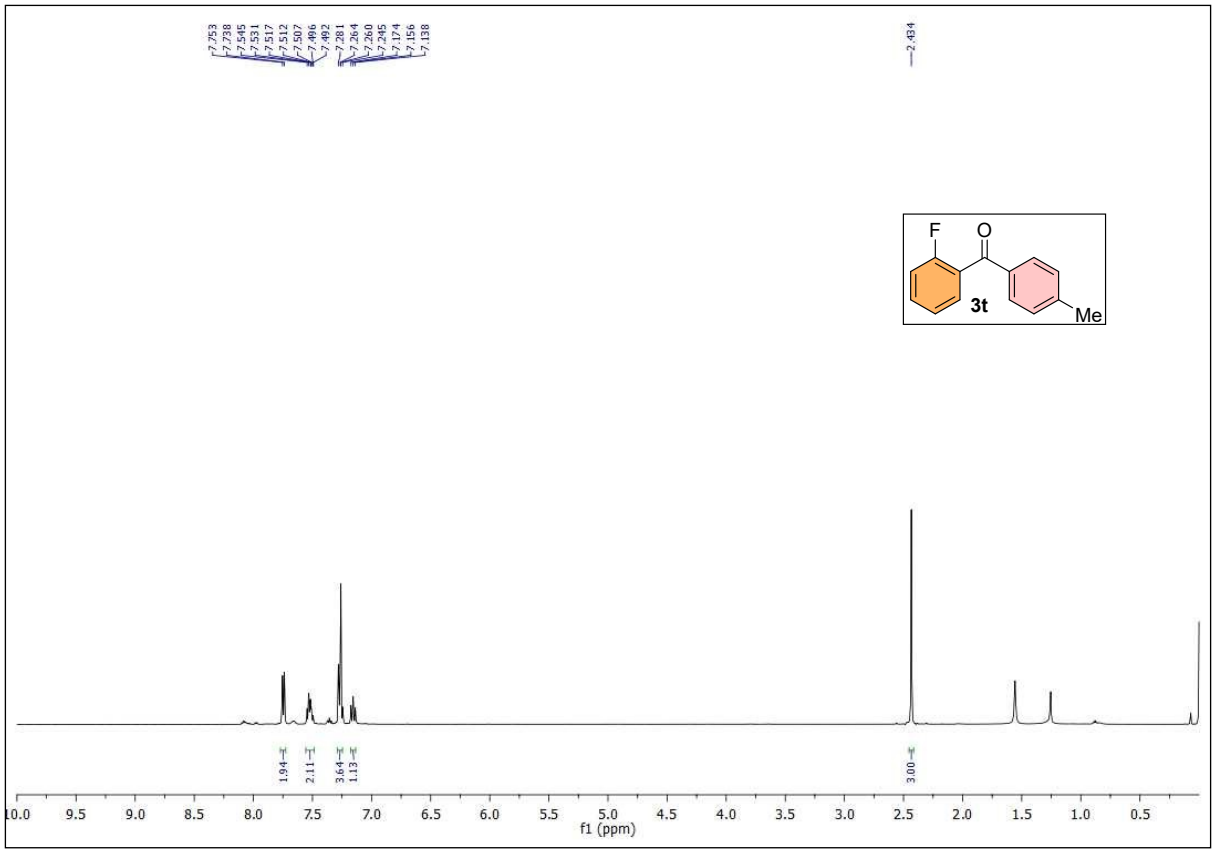

**Spectrum 40**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3r**



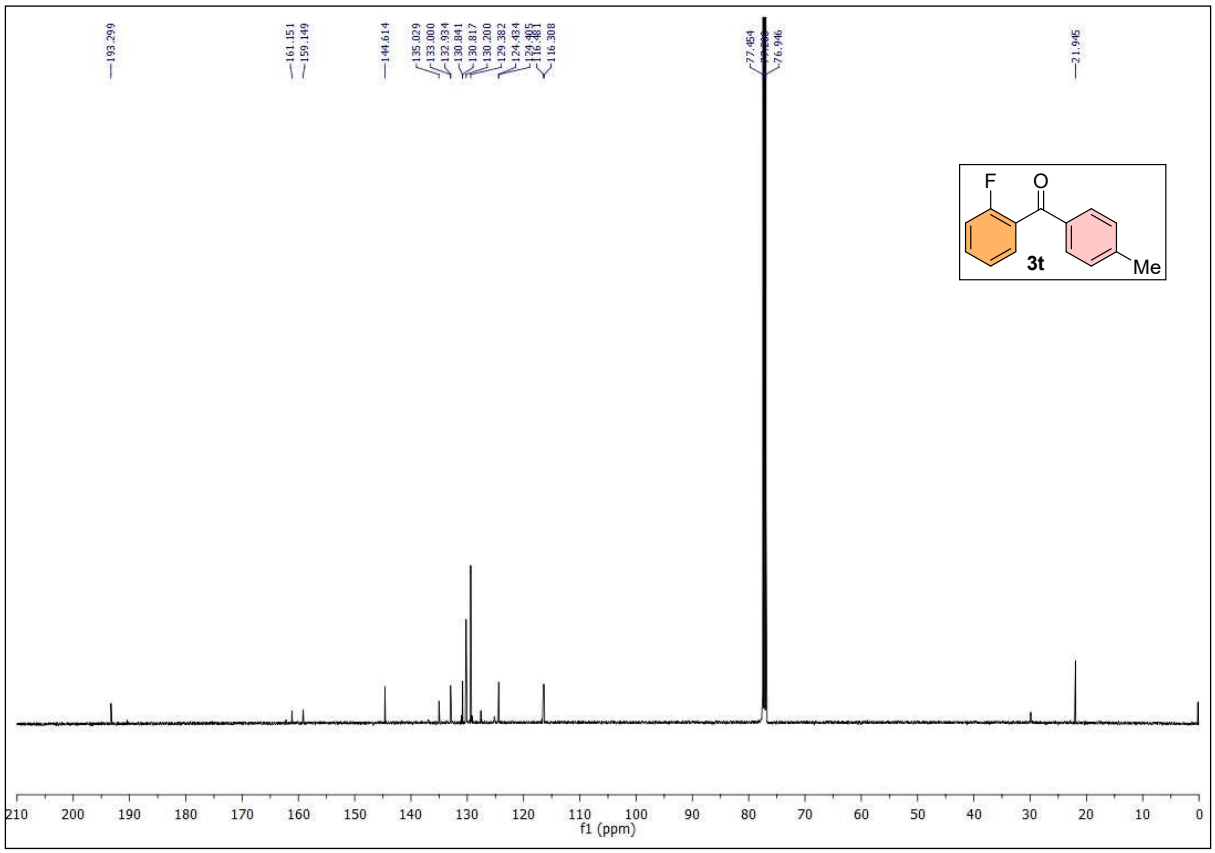

**Spectrum 41**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3s**



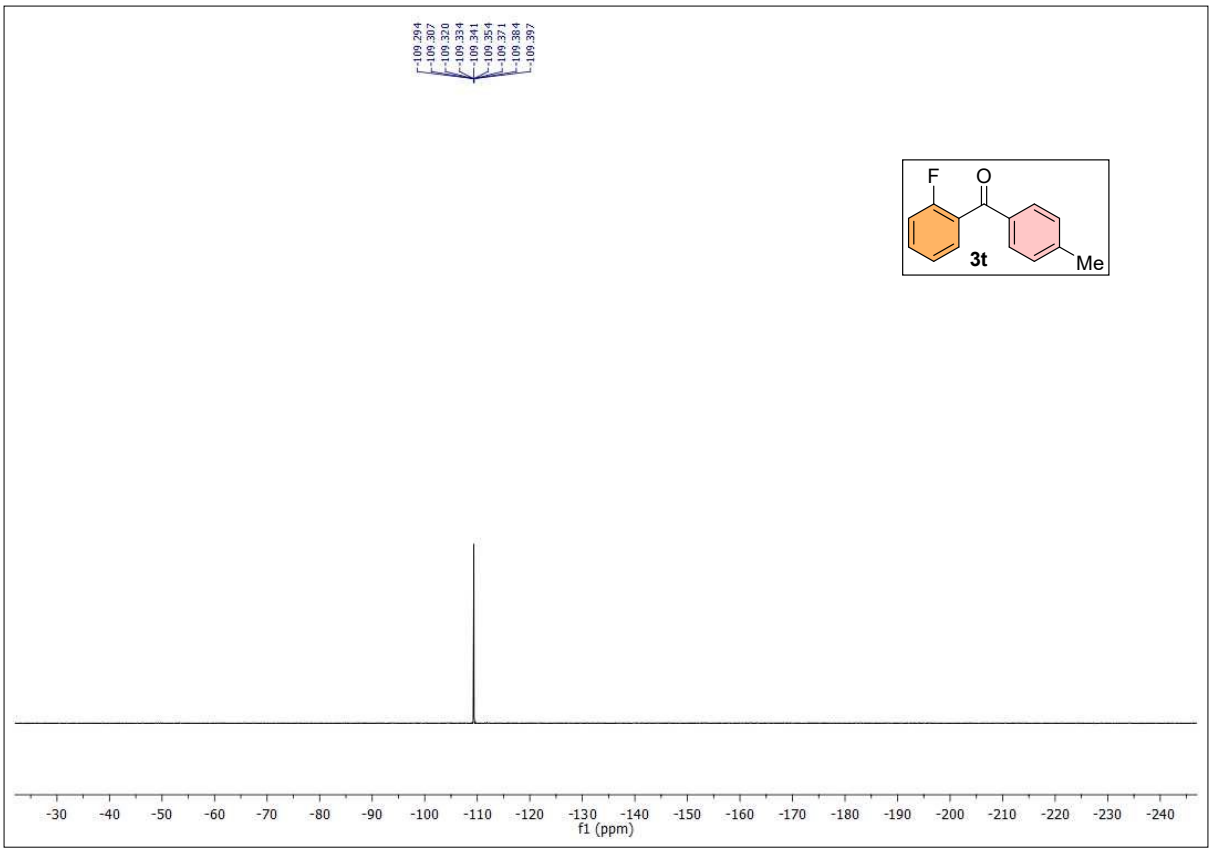

**Spectrum 42**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3s**



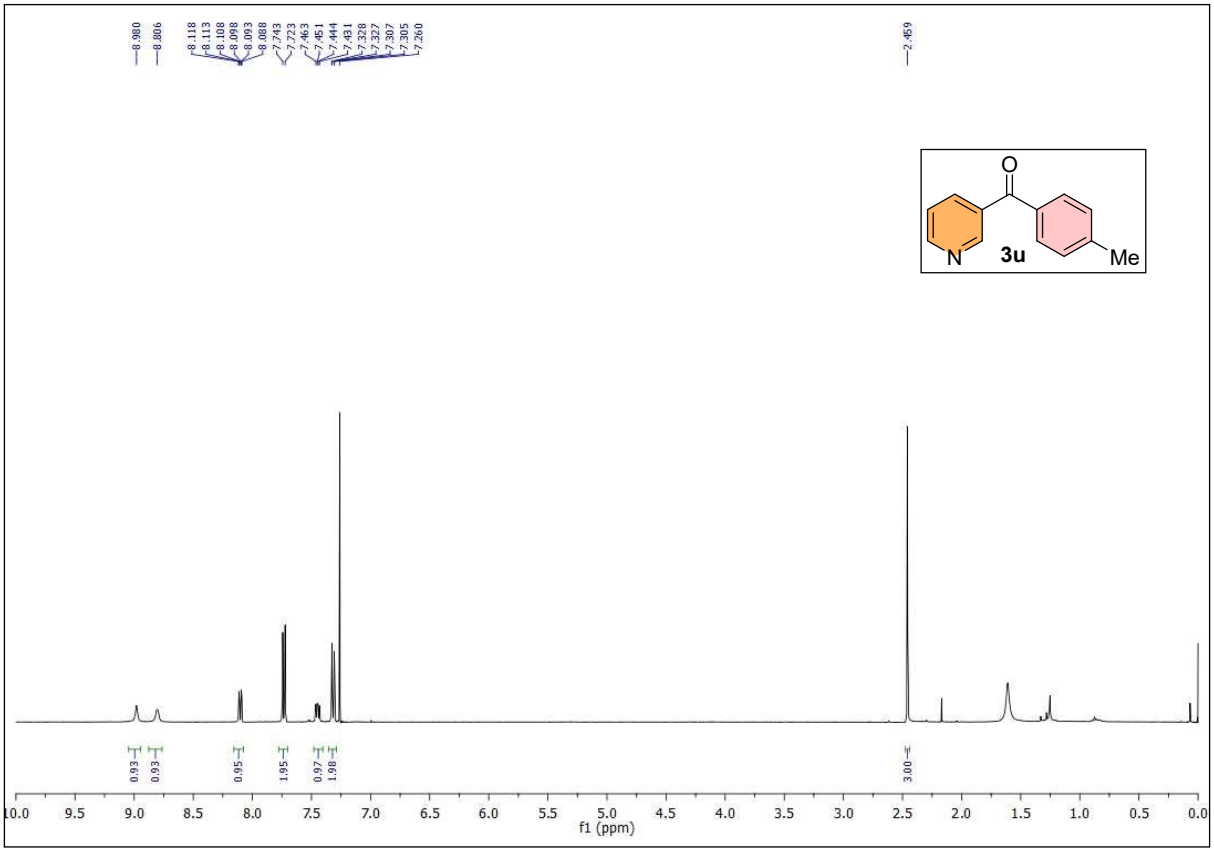

**Spectrum 43**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3t**



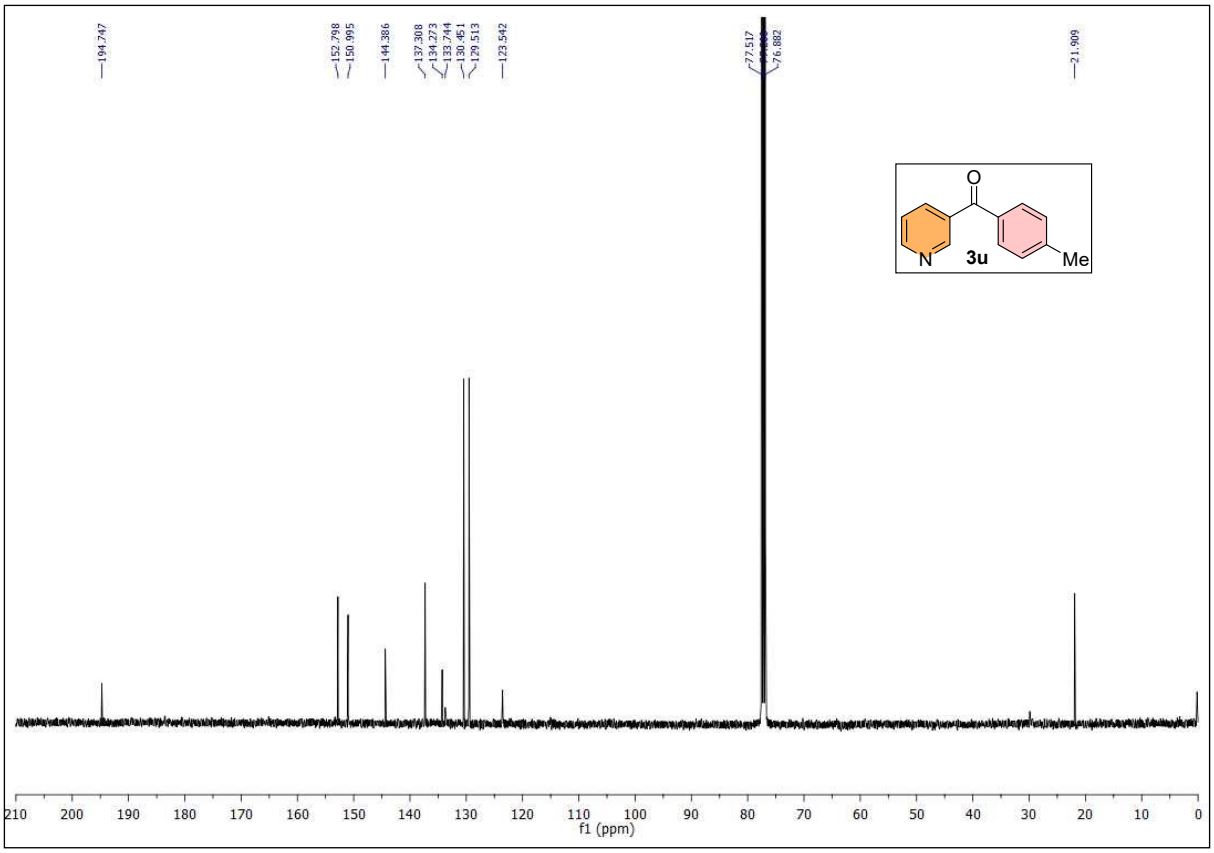

**Spectrum 44**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3t**



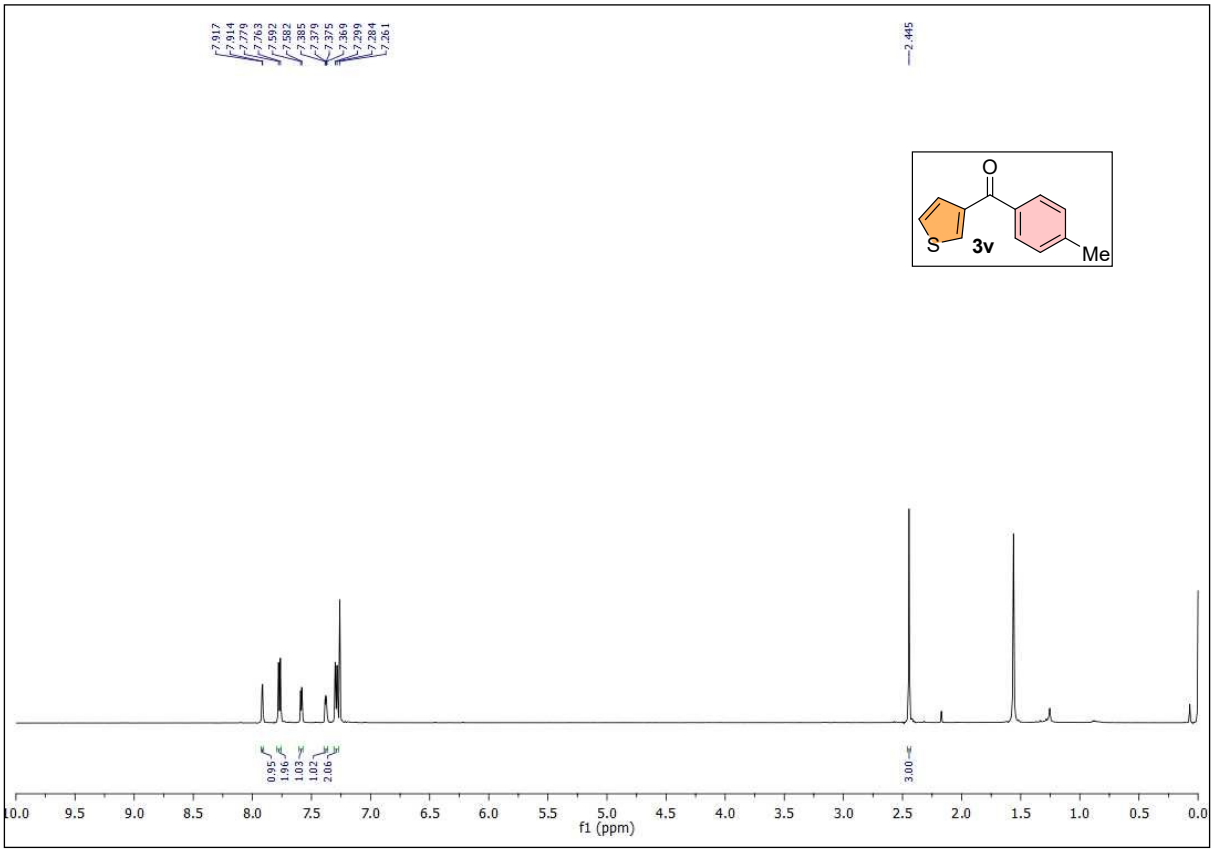

**Spectrum 45**. 376 MHz $^{19}F$ NMR ($CDCl_3$) of compound **3t**



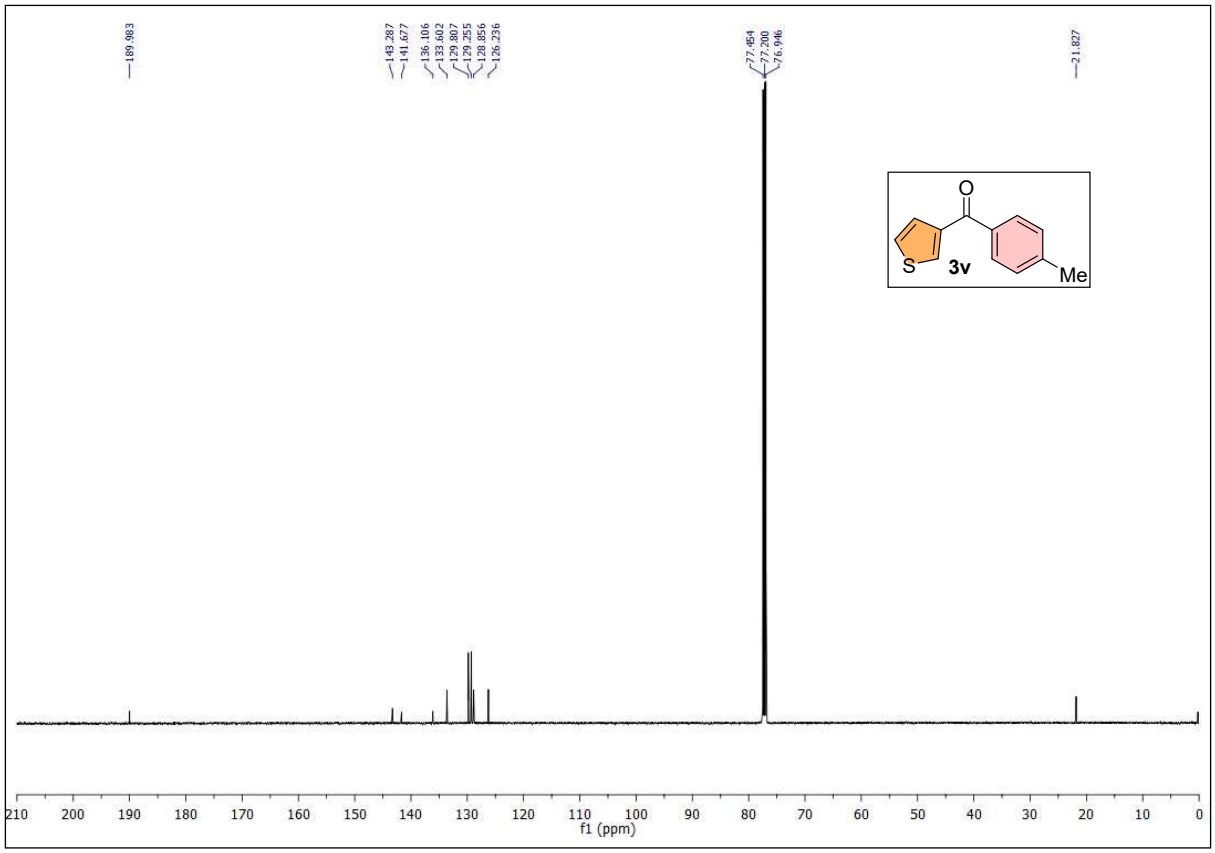

**Spectrum 46**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3u**



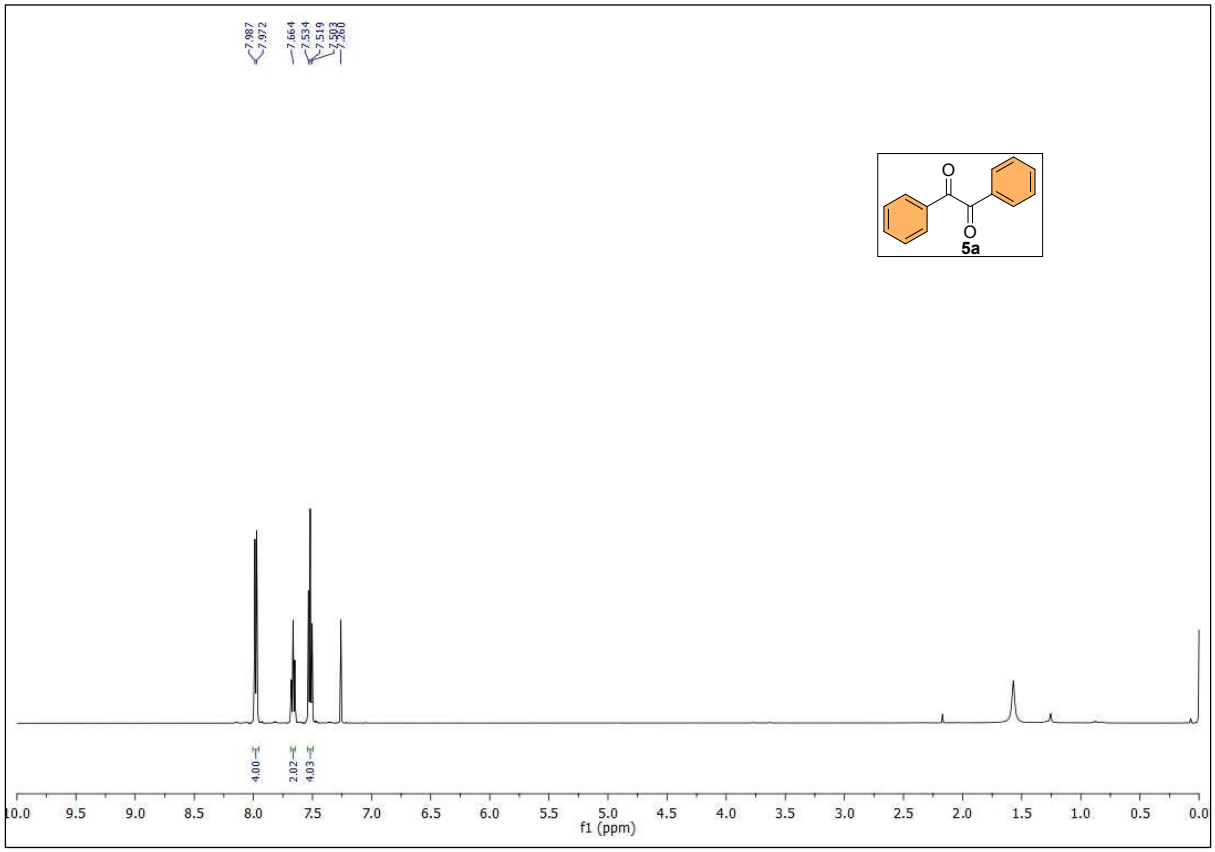

**Spectrum 47**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3u**



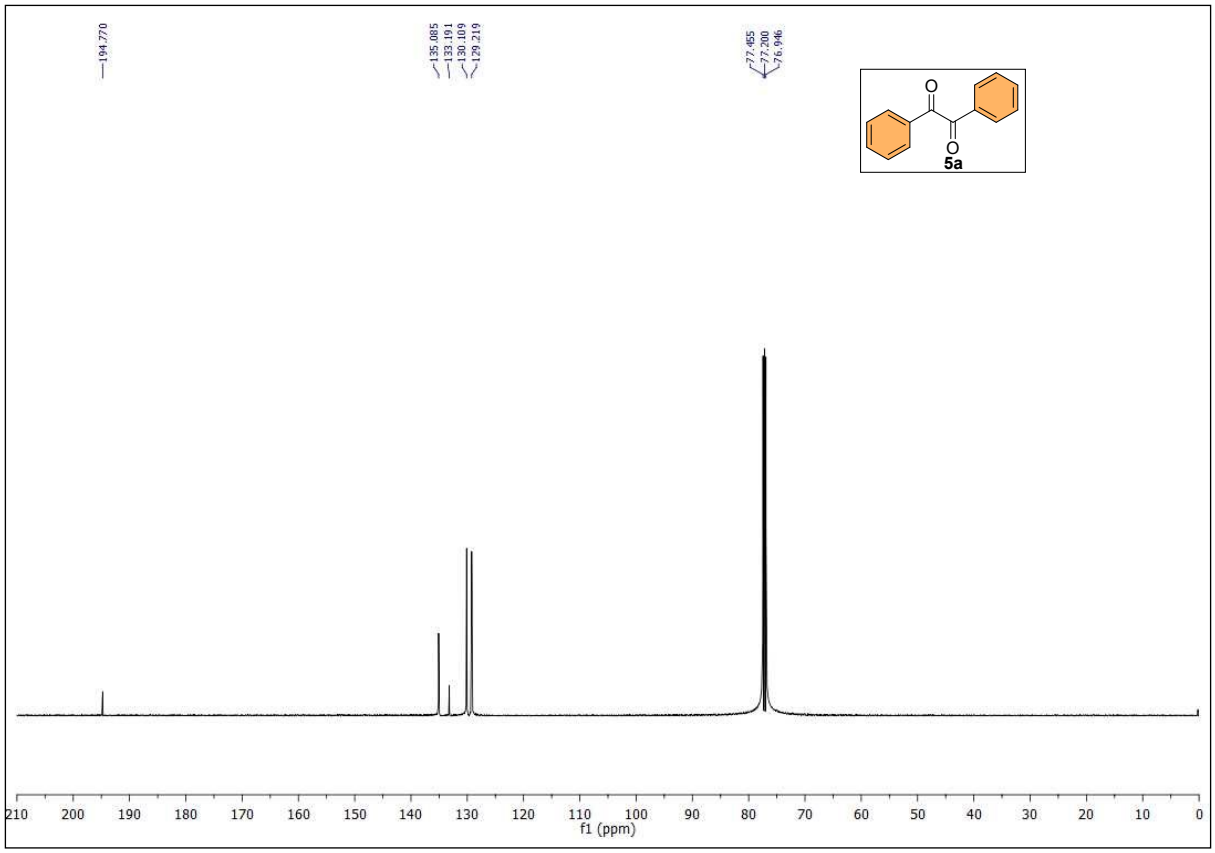

**Spectrum 48**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3v**



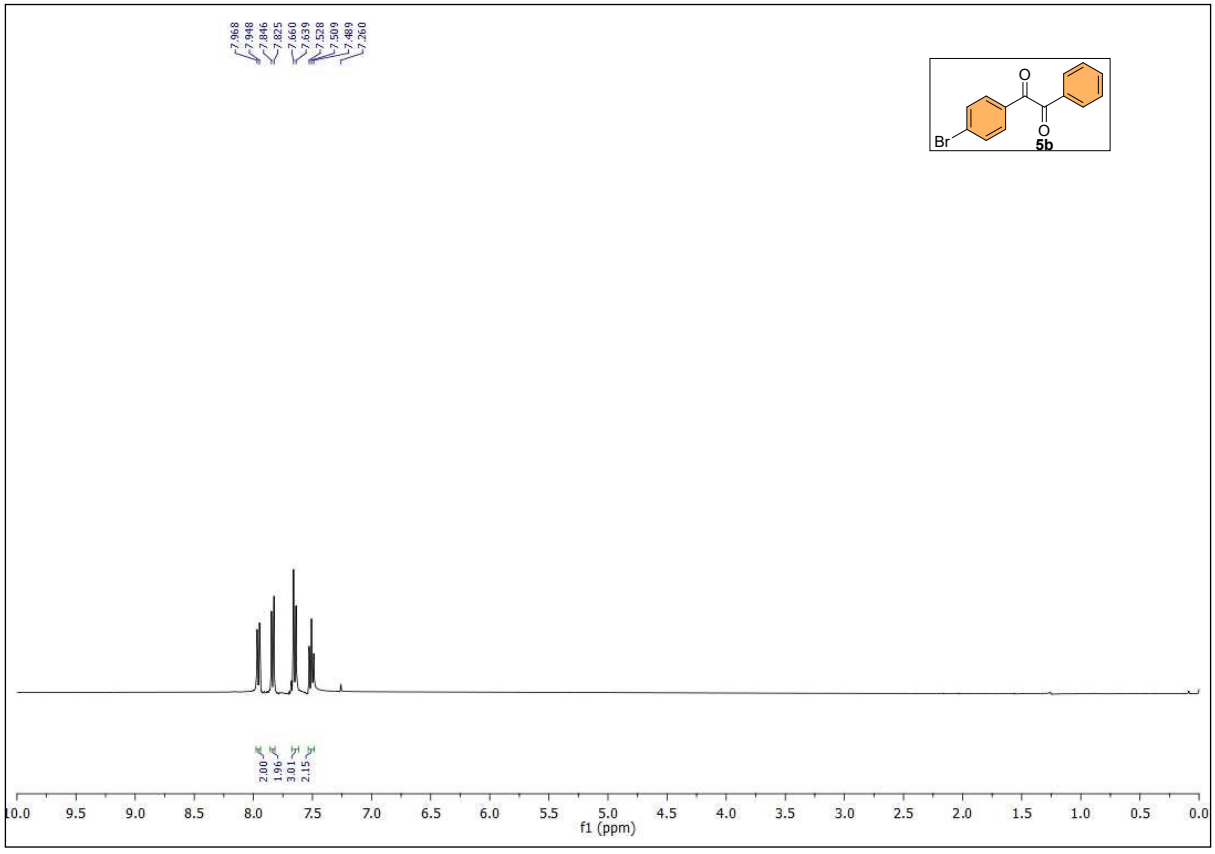

**Spectrum 49**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3v**



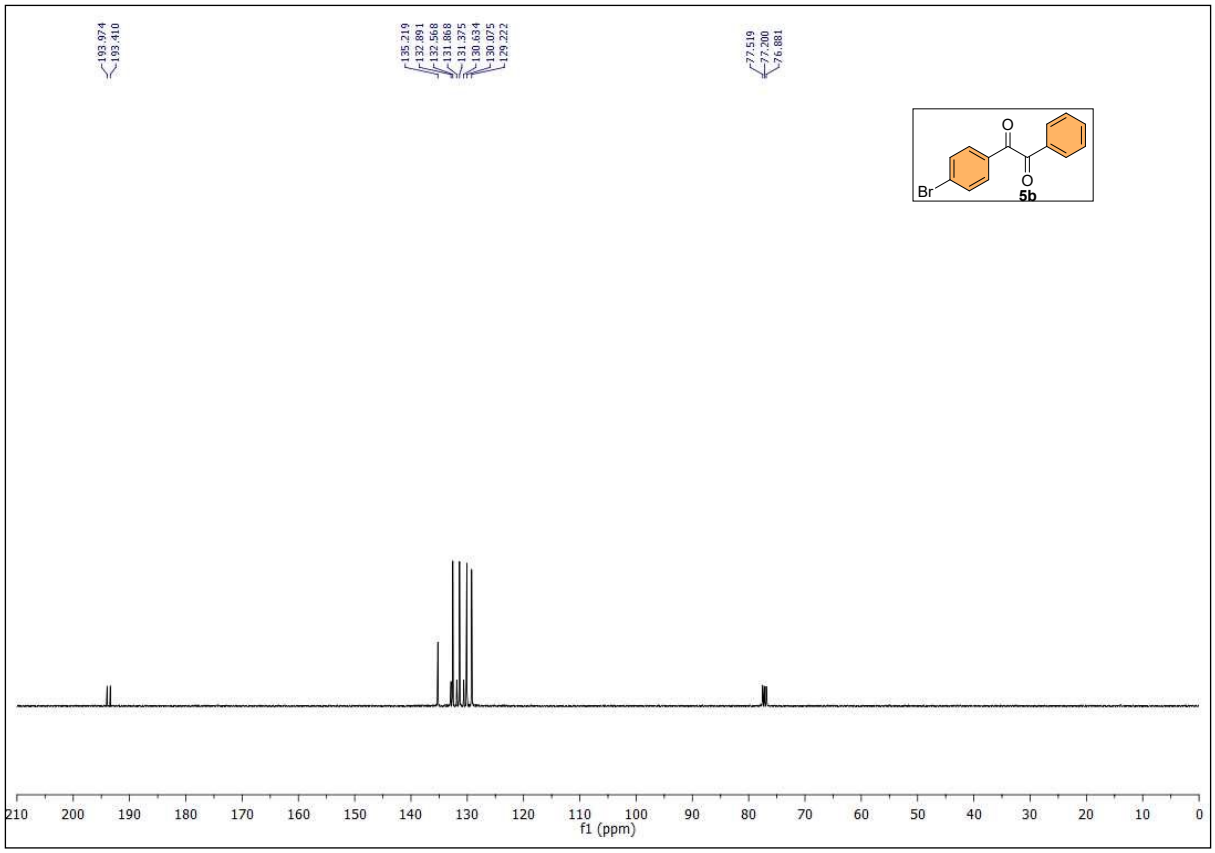

**Spectrum 50**. 500 MHz $^{1}H$ NMR ($CDCl_3$) of compound **5a**



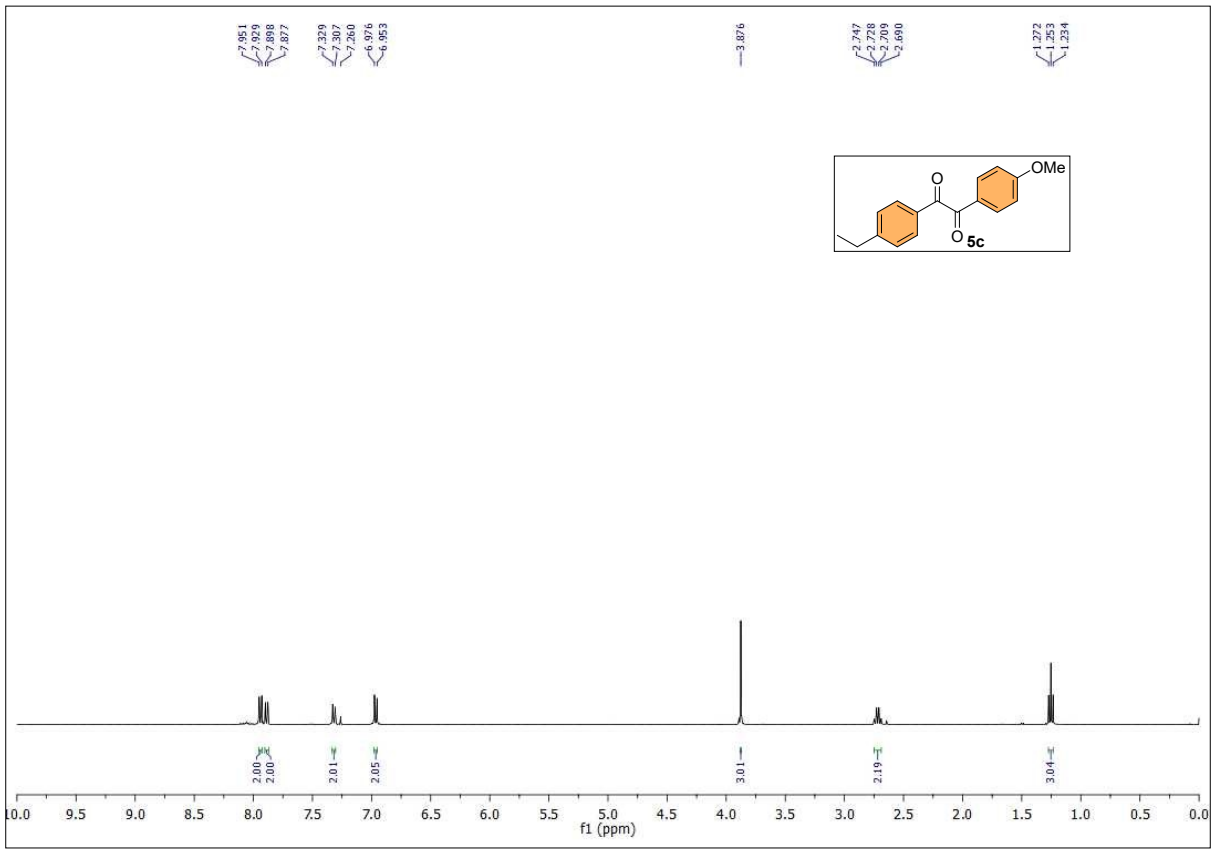

**Spectrum 51**. 125 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **5a**



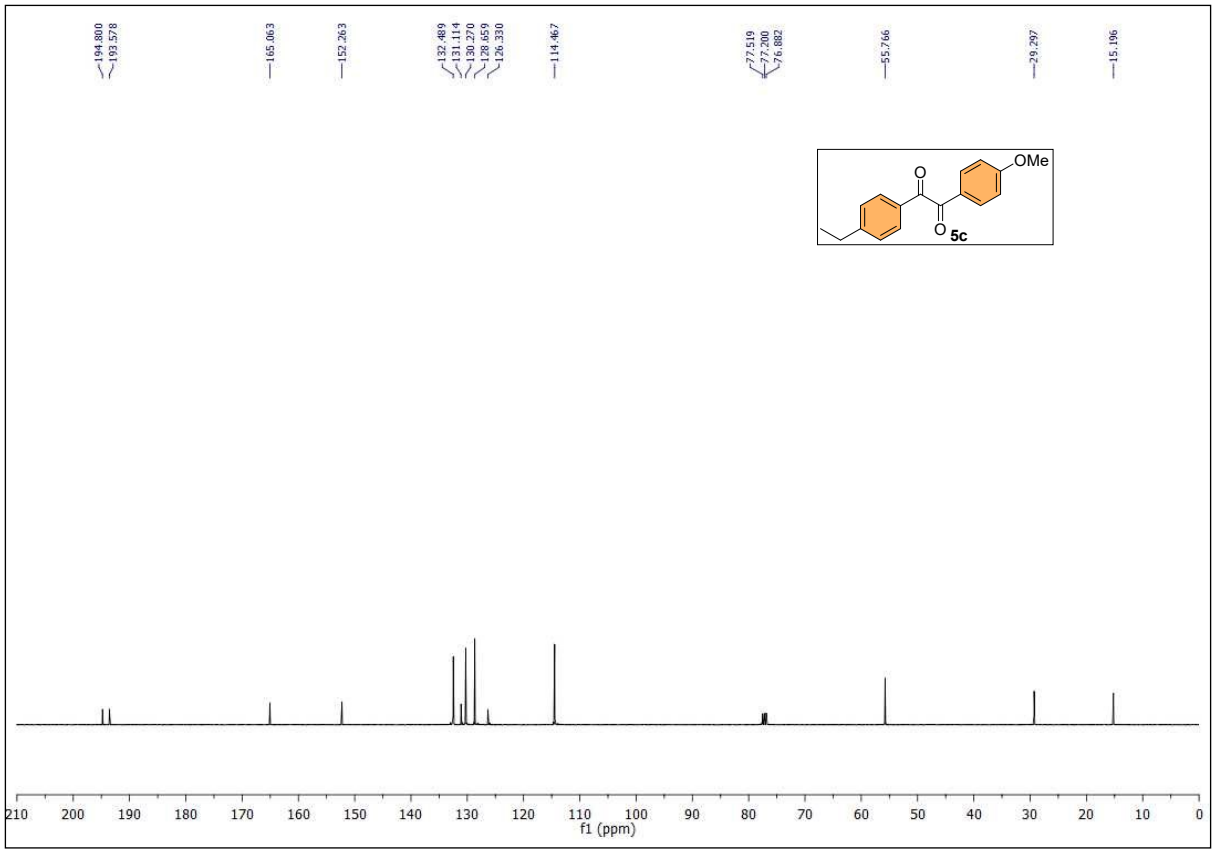

**Spectrum 52**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **5b**



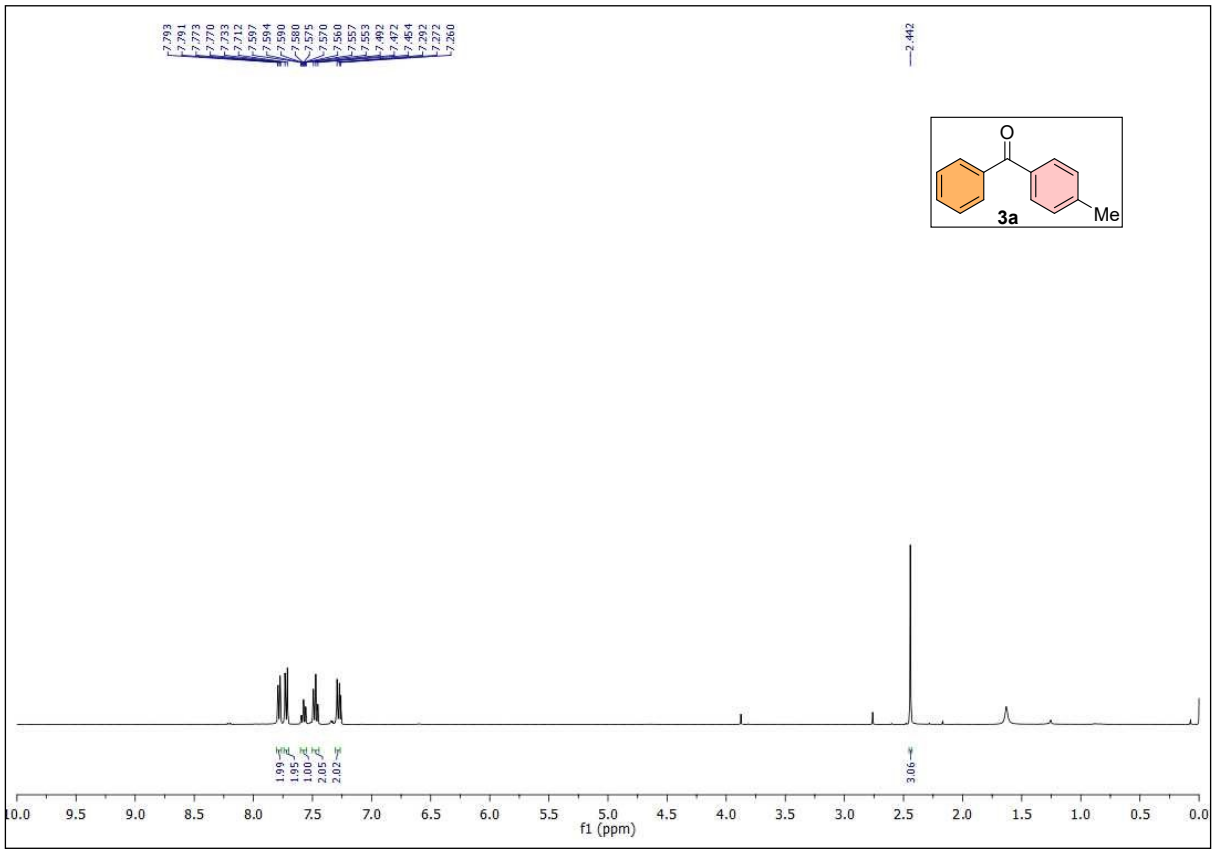

**Spectrum 53**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **5b**



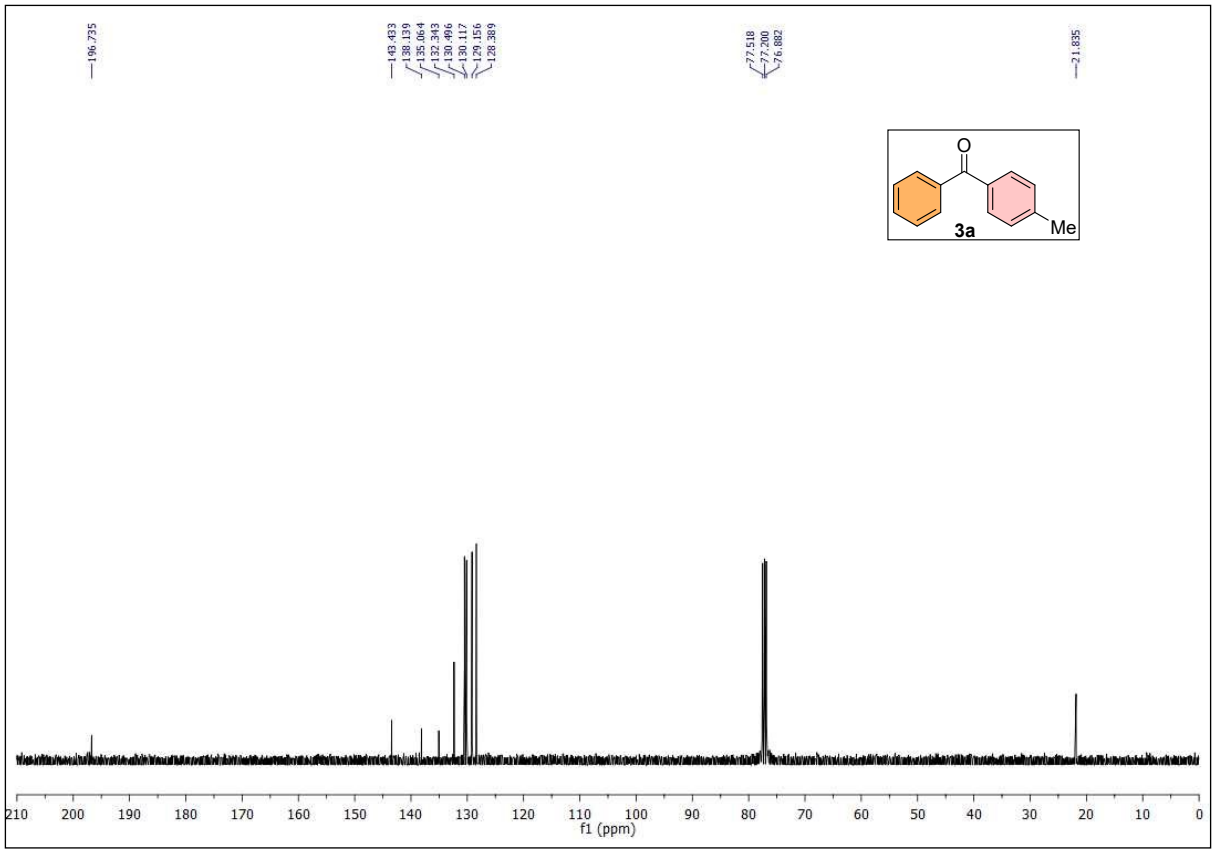

**Spectrum 54**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **5c**



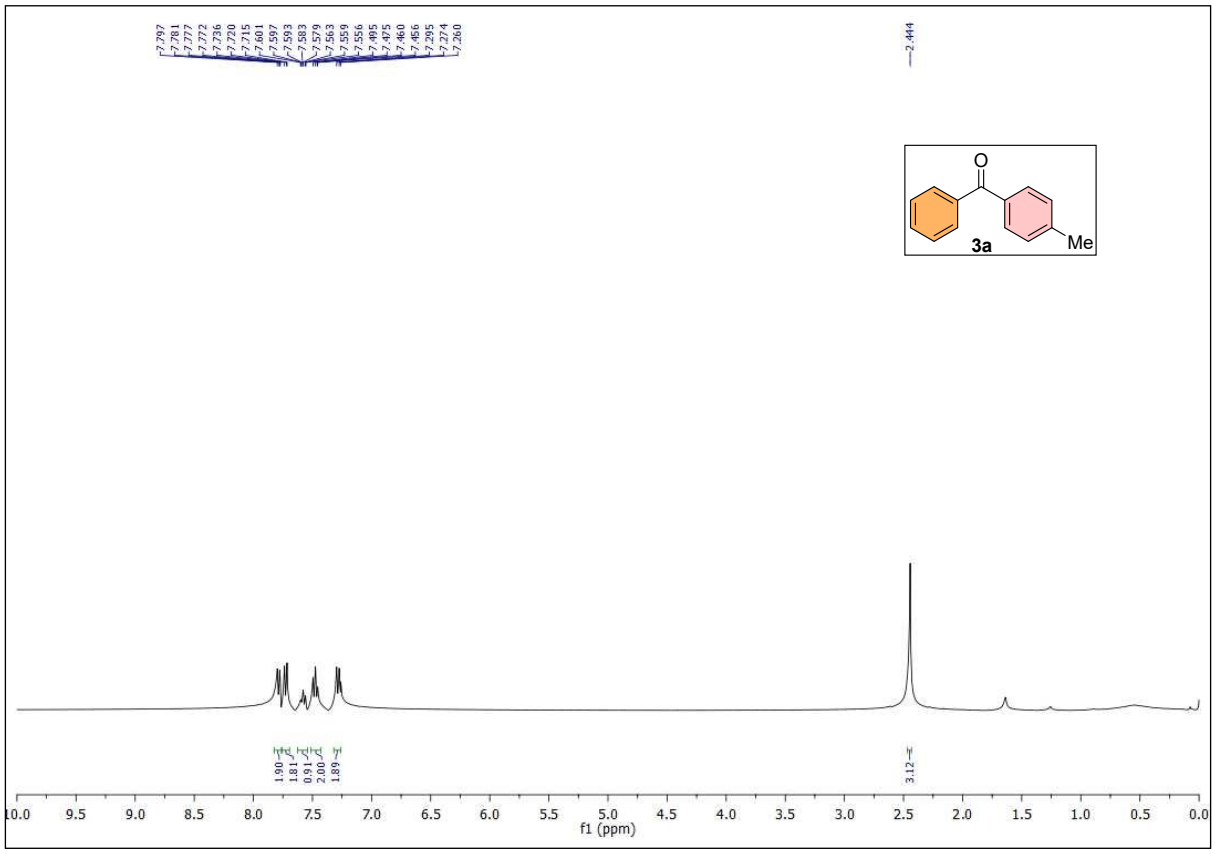

**Spectrum 55**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **5c**



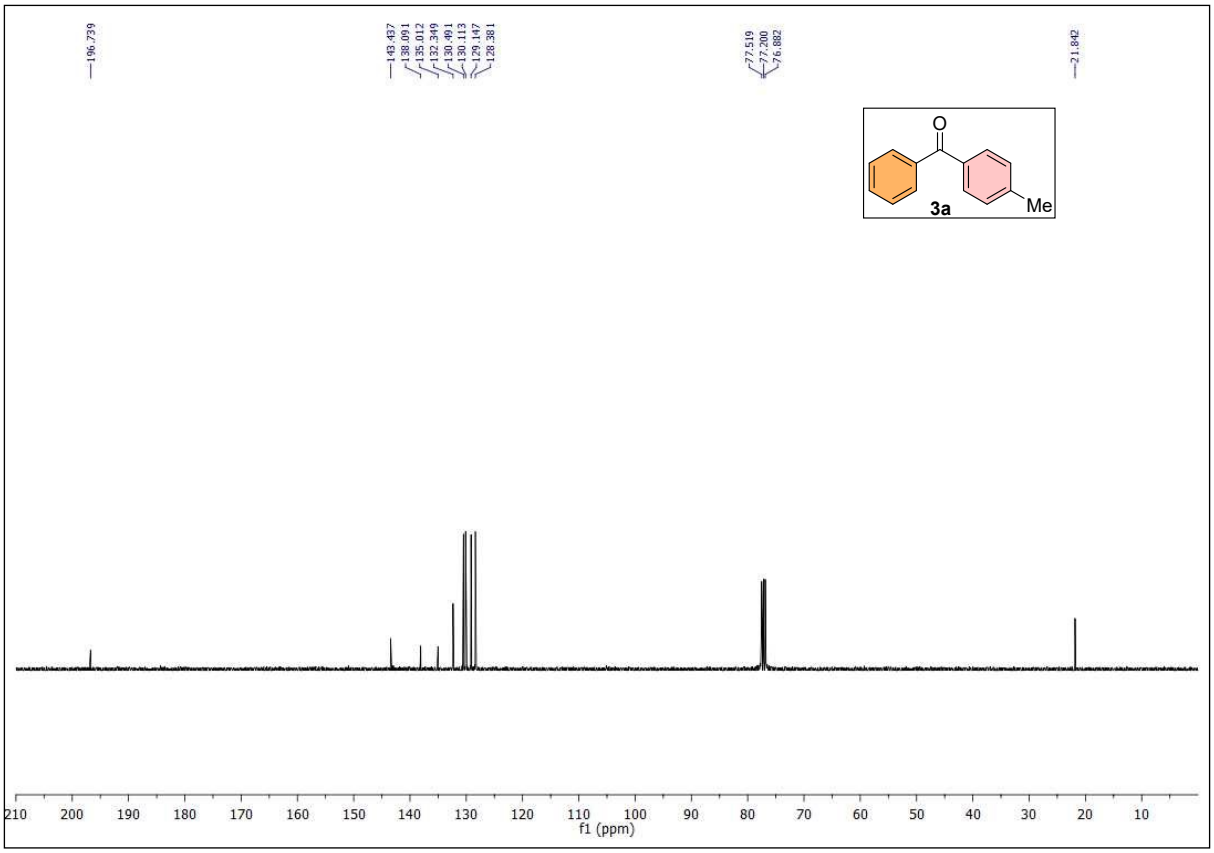

**Spectrum 56**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3a**




**Spectrum 57**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3a**




**Spectrum 58**. 400 MHz $^{1}H$ NMR ($CDCl_3$) of compound **3a**




**Spectrum 59**. 100 MHz $^{13}C\{^{1}H\}$ ($CDCl_3$) NMR of compound **3a**