Electronic Supplementary Material (ESI) for RSC Advances.


# Facile Synthesis of *O*-acylhydroxamates via Reaction of Oxime Chlorides with Carboxylic Acids

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# Supporting Information

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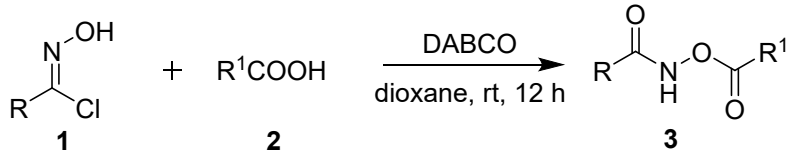
## 1. General methods

NMR data were obtained for $^{1}H$ at 400 MHz MHz, and for $^{13}C$ at 100 MHz. Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard in $CDCl_3$ solution. ESI HRMS was recorded on a Waters SYNAPT G2. Column chromatography was performed on silica gel (200-300 mesh) eluting with ethyl acetate/petroleum ether. TLC was performed on glass-backed silica plates. UV light, $I_2$, and solution of potassium permanganate were used to visualize products. All chemicals were used without purification as commercially available unless otherwise noted. Petroleum ether and ethyl acetate were distilled. THF was freshly distilled from sodium/benzophenone. Unless otherwise noted, experiments involving moisture and/or air sensitive components were performed under a positive pressure of argon in oven-dried glassware equipped with a rubber septum inlet. Dried solvents and liquid reagents were transferred by oven-dried syringes. The hydroximoyl chloride **1** [1]and isolable 2,4,6-trimethylbenzonitrile oxide **1a'** [2]were prepared according to the literature procedures.

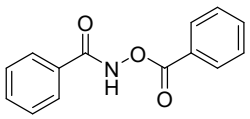

(1) (a) R. J. B. Schäfer, M. R. Monaco, M. Li, A. Tirla, P. Rivera-Fuentes and H. Wennemers, *J. Am. Chem. Soc.*, 2019, **141**, 18644; (b) M. J. H. Ong and R. J. Hewitt, *ChemistrySelect*, 2019, **4**, 10532; (c) Q. V. Vo, C. Trenerry, S. Rochfort, J. Wadeson, C. Leyton and A. B. Hughes, *Bioorgan. Med. Chem.*, 2013, **21**, 5945.

(2) (a) G. Zhao, L. Liang, C. H. E. Wen and R. Tong, *Org. Lett.*, 2019, **21**, 315; (b) O. Altintas, M. Glassner, C. Rodriguez-Emmenegger, A. Welle, V. Trouillet and C. Barner-Kowollik, *Angew. Chem., Int. Ed.*, 2015, **54**, 5777.


## 2. General procedure for synthesis of *O*-acylhydroxamate 3

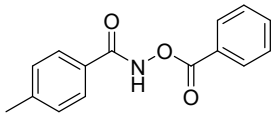



To a solution of oxime chlorides **1** (0.22 mmol), carboxylic acids **2** (0.2 mmol) in dioxane (1 mL) was added DABCO (0.22 mmol). The solution was stirred at rt for 12 h. After completion, product **3** was obtained by flash chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 to 10:1).



**3a**

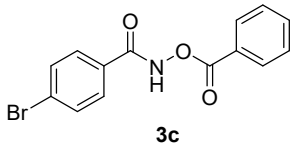
**3a**, 38.6 mg, 80% yield, white solid; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 9.80 (s, 1H), 8.16 (d, $J$ = 8.0 Hz, 2H), 7.89 – 7.86 (m, 2H), 7.66 (t, $J$ = 7.6 Hz, 1H), 7.60 – 7.58 (m, 1H), 7.53 – 7.46 (m, 4H). $^{13}C$ NMR (100 MHz, $CDCl_3$) δ 166.6, 165.3, 134.4, 132.9, 130.9, 130.1, 128.9, 128.8, 127.6, 126.6. ESI-HRMS: $C_{14}H_{11}NO_3$+$H^+$ 242.0812, found 242.0808.



**3b**

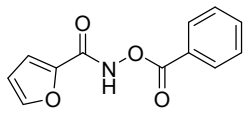
**3b**, 37.7 mg, 74% yield, white solid; $^{1}H$ NMR (400 MHz, $CDCl_3$) δ 9.78 (s, 1H), 8.15 (d, $J$ = 8.0 Hz, 2H), 7.77 (d, $J$ = 8.0 Hz, 2H), 7.64 (t, $J$ = 7.6 Hz, 1H), 7.49 (t, $J$ = 7.6 Hz, 2H), 7.26 (d, $J$ = 5.2 Hz, 2H), 2.41 (s, 3H). $^{13}C$ NMR (100 MHz, $CDCl_3$)

δ 166.7, 165.4, 143.5, 134.3, 130.1, 129.5, 128.7, 128.0, 127.6, 126.7, 21.6. ESI-HRMS: $C_{15}H_{13}NO_3$+$H^+$ 256.0968, found 256.0966.



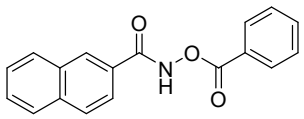
**3c**

**3c**, 48.6 mg, 76% yield, white solid; $^1$H NMR (400 MHz, CDCl3) δ 10.01 (s, 1H), 8.10 (d, *J* = 8.0 Hz, 2H), 7.71 (d, *J* = 8.4 Hz, 2H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.56 (d, *J* = 8.4 Hz, 2H), 7.49 (t, *J* = 7.6 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 165.7, 165.2, 134.4, 132.1, 130.1, 129.6, 129.1, 128.8, 127.7, 126.4. ESI-HRMS: $C_{14}H_{10}BrNO_3$+$H^+$ 319.9917 ($^{79}$Br) and 321.9896 ($^{81}$Br), found 319.9914, 321.9893.



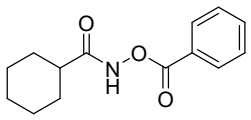
**3d**

**3d**, 32.3 mg, 70% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.72 (s, 1H), 8.15 (d, *J* = 7.6 Hz, 2H), 7.66 (t, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 7.27 (d, *J* = 4.0 Hz, 2H), 6.38 (d, *J* = 3.6 Hz, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 164.9, 155.6, 144.2, 140.0, 134.4, 130.1, 128.8, 126.3, 119.1, 109.1. ESI-HRMS: $C_{12}H_9NO_4$+$H^+$ 232.0604, found 232.0607.



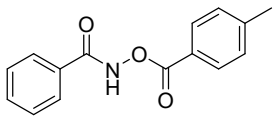
**3e**

**3e**, 44.2 mg, 76% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 10.01 (s, 1H), 8.41 (s, 1H), 8.16 (d, *J* = 7.6 Hz, 2H), 7.92 – 7.86 (m, 4H), 7.66 – 7.53 (m, 3H), 7.49 (t, *J* = 7.6 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.7, 165.4, 135.3, 134.3, 132.5, 130.1, 129.1, 128.8, 128.7, 128.6, 128.3, 128.0, 127.8, 127.0, 126.6, 123.5. ESI-HRMS: $C_{18}H_{13}NO_3$+$H^+$ 292.0968, found 292.0963.



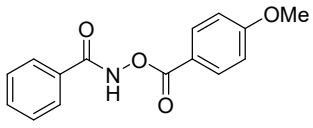
**3f**

**3f**, 34.1 mg, 69% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.27 (s, 1H), 8.09 (d, *J* = 8.0 Hz, 2H), 7.62 (t, *J* = 7.2 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 2.30 (t, *J* = 11.2 Hz, 1H), 1.93 (d, *J* = 12.8 Hz, 2H), 1.82 (d, *J* = 7.6 Hz, 2H), 1.69 – 1.53 (m, 3H), 1.34 – 1.23 (m, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 174.5, 165.2, 134.2, 130.0, 128.7, 126.7, 42.6, 29.2, 25.53, 25.50. ESI-HRMS: $C_{14}H_{17}NO_3$+$H^+$ 248.1281, found 248.1278.



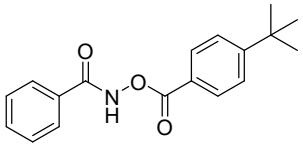
**3g**

**3g**, 34.7 mg, 68% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.83 (s, 1H), 8.04 (d, *J* = 8.4 Hz, 2H), 7.87 (d, *J* = 7.6 Hz, 2H), 7.57 (t, *J* = 7.6 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 2.44 (s, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.5, 165.3, 145.3, 132.8, 130.9, 130.1, 129.5, 128.8, 127.6, 123.7, 21.8. ESI-HRMS: $C_{15}H_{13}NO_3$+$H^+$ 256.0968, found 256.0963.



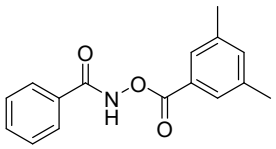
**3h**

**3h**, 35.8 mg, 66% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.91 (s, 1H), 8.09 (d, *J* = 8.4 Hz, 2H), 7.85 (d, *J* = 8.0 Hz, 2H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.44 (t, *J* = 7.2 Hz, 2H), 6.95 (d, *J* = 8.4 Hz, 2H), 3.88 (s, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.5, 165.0, 164.4, 132.7, 132.3, 131.0, 128.8, 127.5, 118.7, 114.1, 55.6. ESI-HRMS: $C_{15}H_{13}NO_4$+$H^+$ 272.0917, found 272.0914.



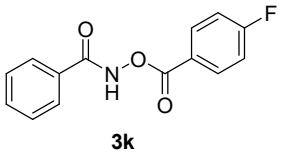
**3i**

**3i**, 38.0 mg, 64% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.95 (s, 1H), 8.07 (d, *J* = 8.4 Hz, 2H), 7.86 (d, *J* = 8.0 Hz, 2H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 2H), 7.45 (t, *J* = 7.6 Hz, 2H), 1.35 (s, 9H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.6, 165.3, 158.3, 132.7, 130.9, 130.0, 128.8, 127.6, 125.8, 123.7, 35.3, 31.0. ESI-HRMS: $C_{18}H_{19}NO_3$+$H^+$ 298.1438, found 298.1435.



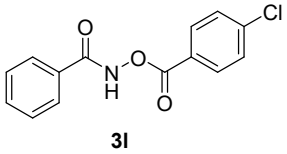
**3j**

**3j**, 36.0 mg, 67% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.82 (s, 1H), 7.87 (d, *J* = 7.6 Hz, 2H), 7.77 (s, 2H), 7.58 (t, *J* = 7.2 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.27 (s, 1H), 2.38 (s, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.5, 165.6, 138.5, 136.1, 132.8, 130.9, 128.9, 127.7, 127.6, 126.3, 21.1. ESI-HRMS: $C_{16}H_{15}NO_3$+$H^+$ 270.1125, found 270.1121.



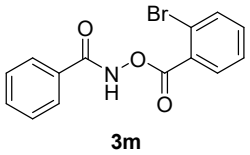
**3k**

**3k,** 38.3 mg, 74% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.84 (s, 1H), 8.16 (dd, *J* = 8.4, 5.6 Hz, 2H), 7.86 (d, *J* = 8.0 Hz, 2H), 7.57 (t, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.2 Hz, 2H), 7.16 (t, *J* = 8.4 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.6, 166.5 (d, *J* = 254.5 Hz), 164.3, 134.3, 132.9 (d, *J* = 2.2 Hz), 132.7, 130.7, 130.0, 128.9, 128.7, 127.6, 122.8 (d, *J* = 3.1 Hz), 116.1 (d, *J* = 22.1 Hz). ESI-HRMS: $C_{14}H_{10}FNO_3$+$H^+$ 260.0717, found 260.0714.



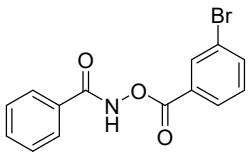
**3l**

**3l,** 38.5 mg, 70% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.76 (s, 1H), 8.08 (d, *J* = 8.0 Hz, 2H), 7.86 (d, *J* = 7.6 Hz, 2H), 7.59 (t, *J* = 6.8 Hz, 1H), 7.48 (d, *J* = 6.0 Hz, 4H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.6, 164.5, 141.0, 132.9, 131.4, 130.7, 129.2, 128.9, 127.6, 125.0. ESI-HRMS: $C_{14}H_{10}ClNO_3$+$H^+$ 276.0422, found 276.0417.



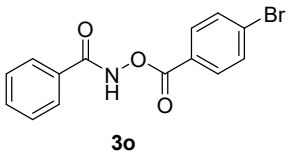
**3m**

**3m,** 42.9 mg, 67% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.72 (s, 1H), 8.08 (dd, *J* = 7.2, 4.8 Hz, 1H), 7.88 (d, *J* = 7.6 Hz, 2H), 7.73 – 7.71 (m, 1H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.50 – 7.41 (m, 4H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.7, 164.7, 134.6, 133.9, 132.9, 132.2, 130.6, 128.9, 128.4, 127.6, 127.5, 122.5. ESI-HRMS: $C_{14}H_{10}BrNO_3$+$H^+$ 319.9917 ($^{79}$Br) and 321.9896 ($^{81}$Br), found 319.9914, 321.9895.



**3n**

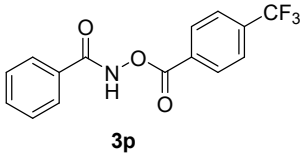
**3n,** 42.9 mg, 67% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.70 (s, 1H), 8.29 (s, 1H), 8.09 (d, *J* = 7.6 Hz, 1H), 7.87 (d, *J* = 7.6 Hz, 2H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.39 (t, *J* = 7.6 Hz, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.6, 164.1, 137.3, 133.0, 132.9, 130.6, 130.3, 130.1, 129.0, 128.8, 128.6, 128.5, 127.6, 122.8. ESI-HRMS: $C_{14}H_{10}BrNO_3$+$H^+$ 319.9917 ($^{79}$Br) and 321.9896 ($^{81}$Br), found 319.9912, 321.9891.



**3o**

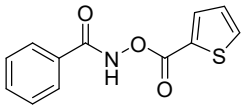
**3o,** 44.8 mg, 70% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.97 (d, *J* = 8.4 Hz, 2H), 7.85 (d, *J* = 8.0 Hz, 2H), 7.62 (d, *J* = 8.4 Hz, 2H), 7.56 (t,

$J$ = 7.2 Hz, 1H), 7.44 (t, $J$ = 7.2 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.6, 164.6, 132.9, 132.1, 131.4, 130.6, 129.7, 128.9, 127.6, 125.4. ESI-HRMS: $C_{14}H_{10}BrNO_3$+$H^+$ 319.9917 ($^{79}$Br) and 321.9896 ($^{81}$Br), found 319.9913, 321.9895.



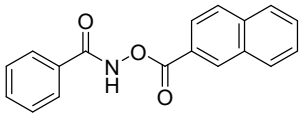
**3p**

**3p,** 38.3 mg, 62% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.79 (s, 1H), 8.27 (d, $J$ = 8.0 Hz, 2H), 7.87 (d, $J$ = 7.6 Hz, 2H), 7.77 (d, $J$ = 8.4 Hz, 2H), 7.60 (t, $J$ = 7.2 Hz, 1H), 7.48 (t, $J$ = 7.6 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.7, 164.2, 135.7 (q, $J$ = 32.6 Hz), 134.4, 133.0, 130.5, 129.4 (q, $J$ = 14.9 Hz), 129.0, 127.6, 125.8 (q, $J$ = 3.6 Hz), 123.4 ((q, $J$ = 271.2 Hz). ESI-HRMS: $C_{15}H_{10}F_3NO_3$+$H^+$ 310.0686, found 310.0679.



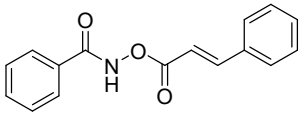
**3q**

**3q,** 36.6 mg, 74% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.80 (s, 1H), 7.98 (d, $J$ = 3.2 Hz, 1H), 7.85 (d, $J$ = 7.6 Hz, 2H), 7.70 (d, $J$ = 4.8 Hz, 1H), 7.55 (t, $J$ = 7.6 Hz, 1H), 7.44 (t, $J$ = 7.6 Hz, 2H), 7.16 (t, $J$ = 4.0 Hz, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.7, 160.9, 135.5, 134.5, 132.8, 130.6, 128.8, 128.2, 127.6. ESI-HRMS: $C_{12}H_9NO_3S$+$H^+$ 248.0376, found 248.0374.



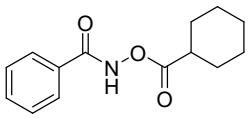
**3r**

**3r,** 41.3 mg, 71% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.97 (s, 1H), 8.76 (s, 1H), 8.21 – 7.91 (m, 7H), 7.64 – 7.49 (m, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.6, 165.5, 136.1, 134.3, 132.9, 132.4, 132.2, 130.9, 130.1, 129.6, 128.9, 128.7, 127.9, 127.6, 127.1, 124.9, 123.6. ESI-HRMS: $C_{18}H_{13}NO_3$+$H^+$ 292.0968, found 292.0963.



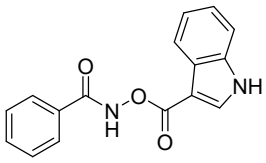
**3s**

**3s,** 37.4 mg, 70% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.69 (s, 1H), 7.91 (d, $J$ = 16.0 Hz, 1H), 7.87 (d, $J$ = 8.0 Hz, 2H), 7.59 (d, $J$ = 6.8 Hz, 3H), 7.50 (t, $J$ = 8.0 Hz, 2H), 7.44 (d, $J$ = 6.0 Hz, 3H), 6.64 (d, $J$ = 16.0 Hz, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.3, 165.5, 148.4, 133.7, 132.8, 131.3, 130.9, 129.1, 128.9, 128.5, 127.5, 113.2. ESI-HRMS: $C_{16}H_{13}NO_3$+$Na^+$ 290.0788, found 290.0795.



**3t**

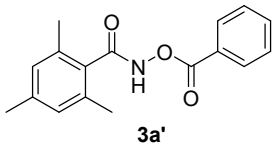
**3t,** 34.1 mg, 69% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.57 (s, 1H), 7.81 (d, $J$ = 8.0 Hz, 2H), 7.56 (t, $J$ = 7.2 Hz, 1H), 7.45 (t, $J$ = 7.6 Hz, 2H), 2.60 (tt, $J$ = 11.2, 3.6 Hz, 1H), 2.01 (d, $J$ = 13.2 Hz, 2H), 1.82 – 1.79 (m, 2H), 1.69 – 1.65 (m, 1H), 1.62 – 1.52 (m, 2H), 1.38 – 1.25 (m, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 174.5, 166.5, 132.7, 130.9, 128.8, 127.5, 41.2, 28.8, 25.5, 25.2. ESI-HRMS: $C_{14}H_{17}NO_3$+$H^+$ 248.1281, found 248.1278.



**3u**

**3u,** 35.8 mg, 64% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 8.16 (d, $J$ = 8.0 Hz, 2H), 7.88 (d, $J$ = 8.0 Hz, 2H), 7.65 (t, $J$ = 6.8 Hz, 1H), 7.59 (t, $J$ = 7.2 Hz, 1H), 7.53-7.46 (m, 4H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 166.6, 165.3,

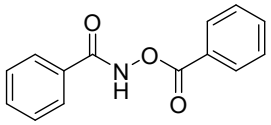
134.3, 132.8, 130.9, 130.1, 128.9, 128.8, 127.6, 126.6. ESI-HRMS: $C_{16}H_{12}N_2O_3$+$H^+$ 281.0921, found 281.0917.



3a'

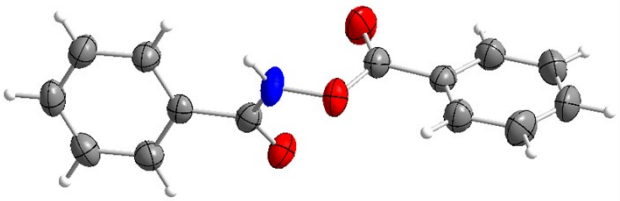
**3a',** 48.1 mg, 85% yield, white solid; $^1$H NMR (400 MHz, $CDCl_3$) δ 9.33 (s, 1H), 8.14 (d, *J* = 7.6 Hz, 2H), 7.65 (t, *J* = 7.2 Hz, 1H), 7.50 (t, *J* = 7.2 Hz, 2H), 6.86 (s, 2H), 2.39 (s, 6H), 2.28 (s, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 167.7, 165.0, 139.9, 135.9, 134.3, 130.1, 129.8, 128.7, 128.4, 126.5, 21.2, 19.2. ESI-HRMS: $C_{17}H_{17}NO_3$+$H^+$ 284.1281, found 284.1275.

## 3. Crystal data and structural refinement for 3a



3a

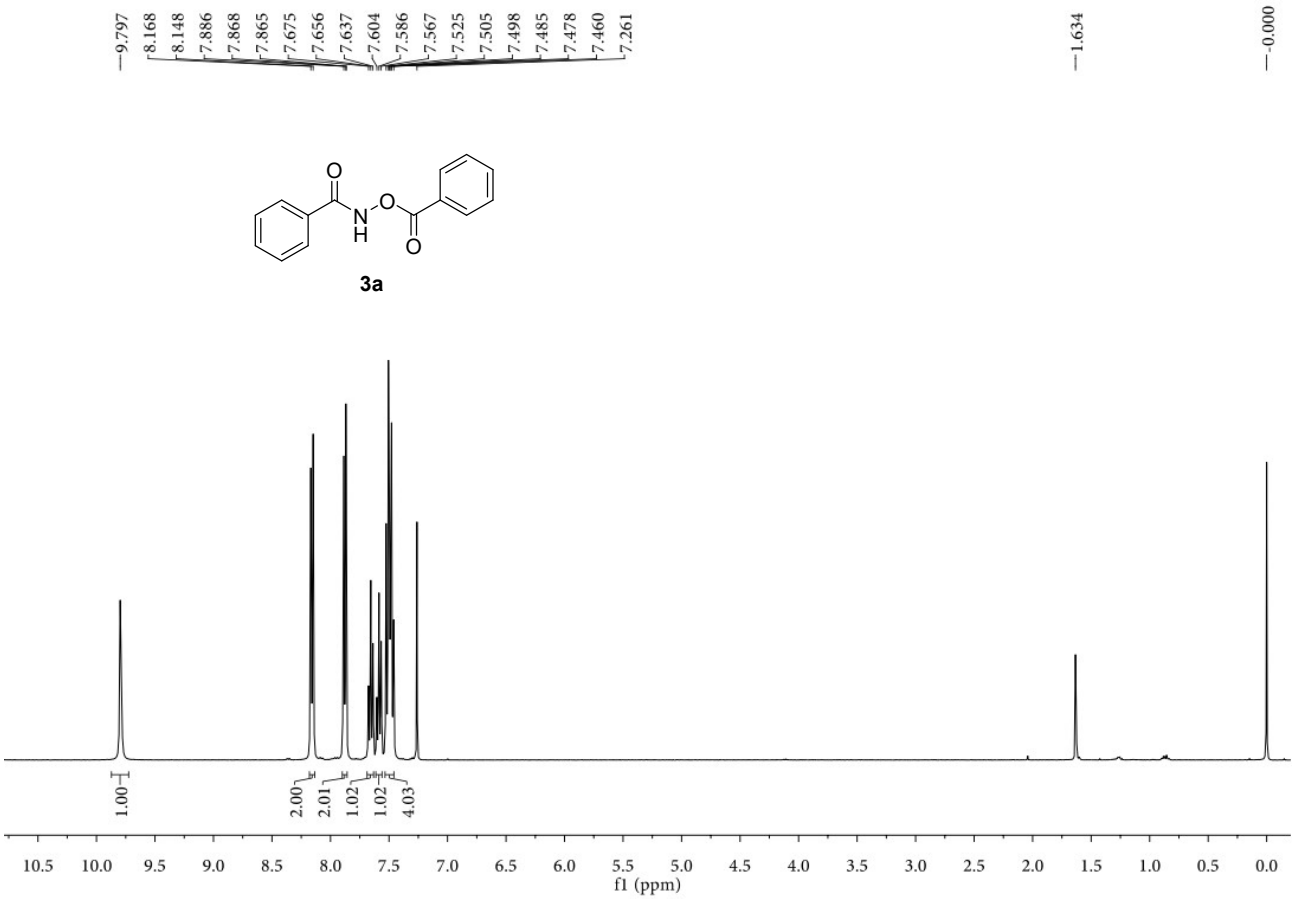
≡

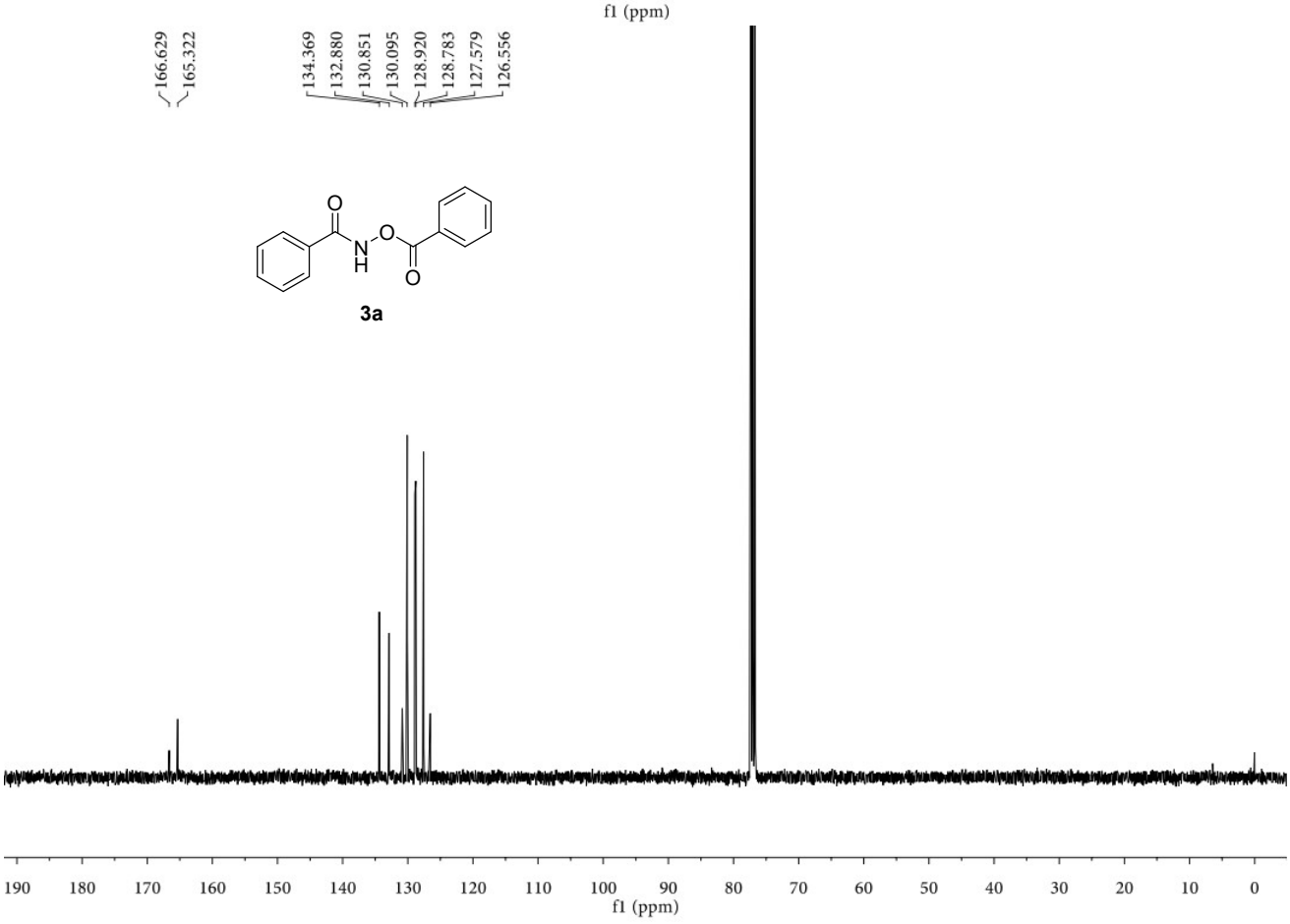

CCDC 1974489

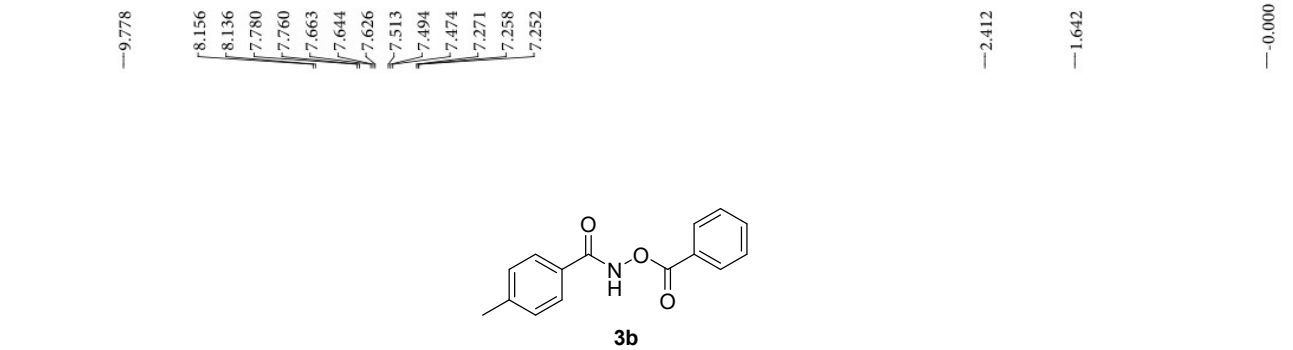
| | |
|---|---|
| Identification code | **3a** |
| Empirical formula | $C_{14}H_{11}NO_3$ |
| Formula weight | 241.24 |
| Temperature/K | 296(2) |
| Crystal system | Orthorhombic |
| Space group | P212121 |
| a/Å | 8.9979(11) |
| b/Å | 9.4469(11) |
| c/Å | 14.0932(14) |
| α/° | 90 |
| β/° | 90 |
| γ/° | 90 |
| Volume/Å$^3$ | 1198.0(2) |
| Z | 4 |
| $\rho_{calc}$g/cm$^3$ | 1.338 |
| μ/mm$^{-1}$ | 0.095 |

| | |
|---|---|
| F(000) | 504.0 |
| Crystal size/$mm^3$ | 0.26 × 0.26 × 0.25 |
| Radiation | MoKα (λ = 0.71073) |
| 2Θ range for data collection/° | 2.60 to 24.992 |
| Index ranges | $-10 \leq h \leq 10$, $-9 \leq k \leq 11$, $-16 \leq l \leq 16$ |
| Reflections collected | 6084 |
| Independent reflections | 2107 [$R_{int}$ = 0.0180] |
| Data/restraints/parameters | 1859 / 0 / 164 |
| Goodness-of-fit on $F^2$ | 1.017 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0326, $wR_2$ = 0.0940 |
| R indices (all data) | $R_1$ = 0.0384, $wR_2$ = 0.0983 |
| Largest diff. peak and hole | 0.127 /-0.153 |

## 4. NMR spectra

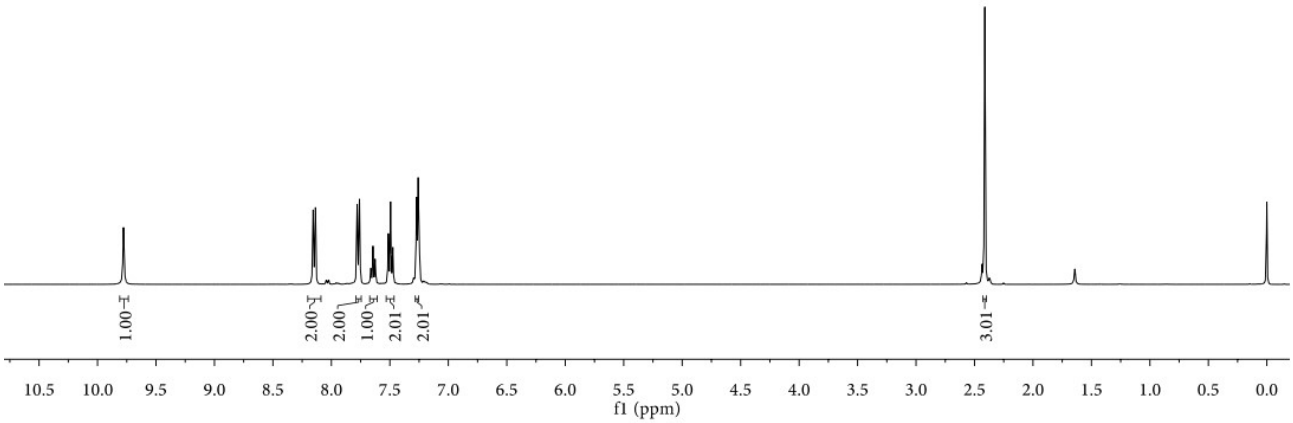


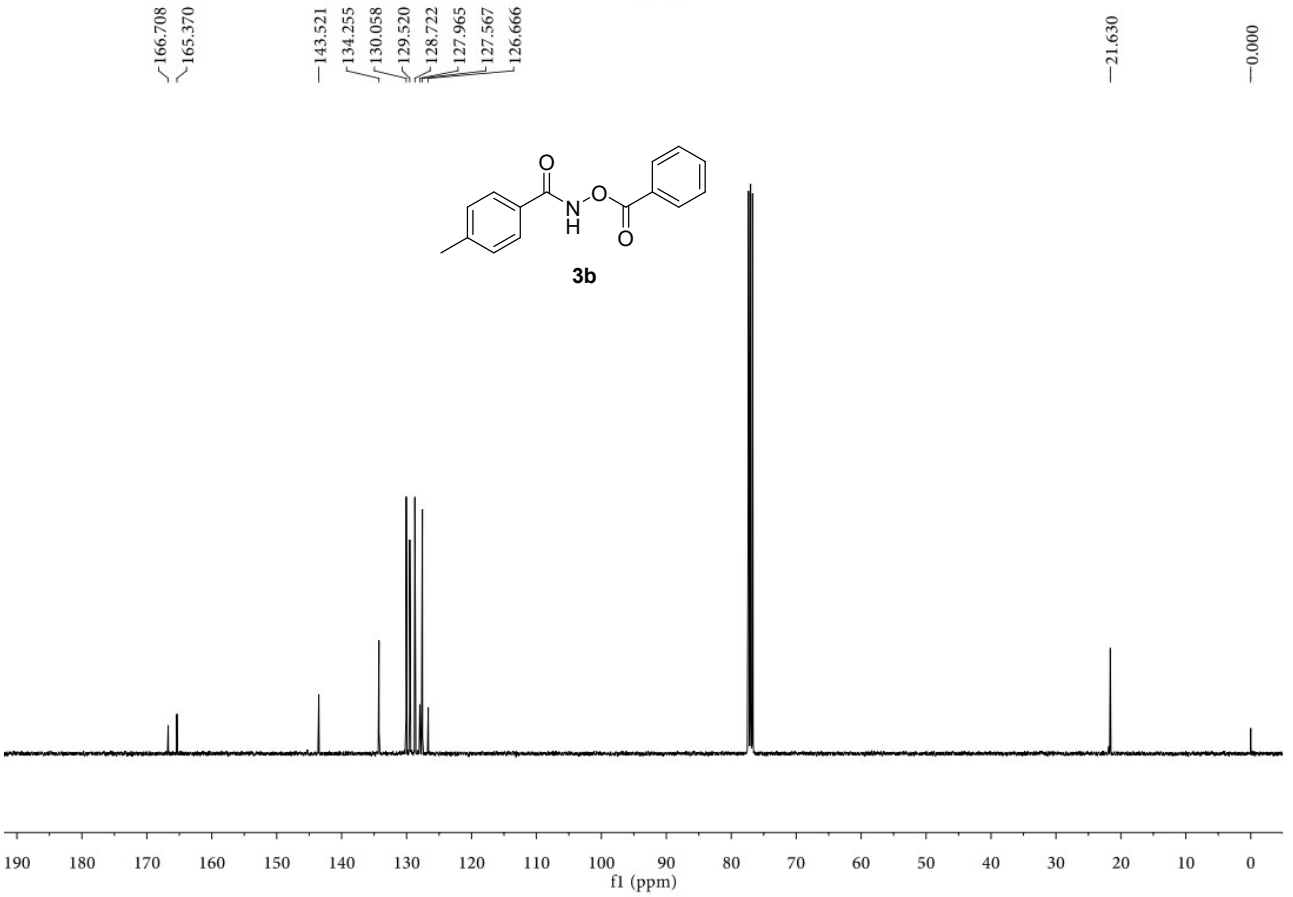




9.778
8.156
8.136
7.780
7.760
7.663
7.644
7.626
7.513
7.494
7.474
7.271
7.258
7.252
2.412
1.642
0.000



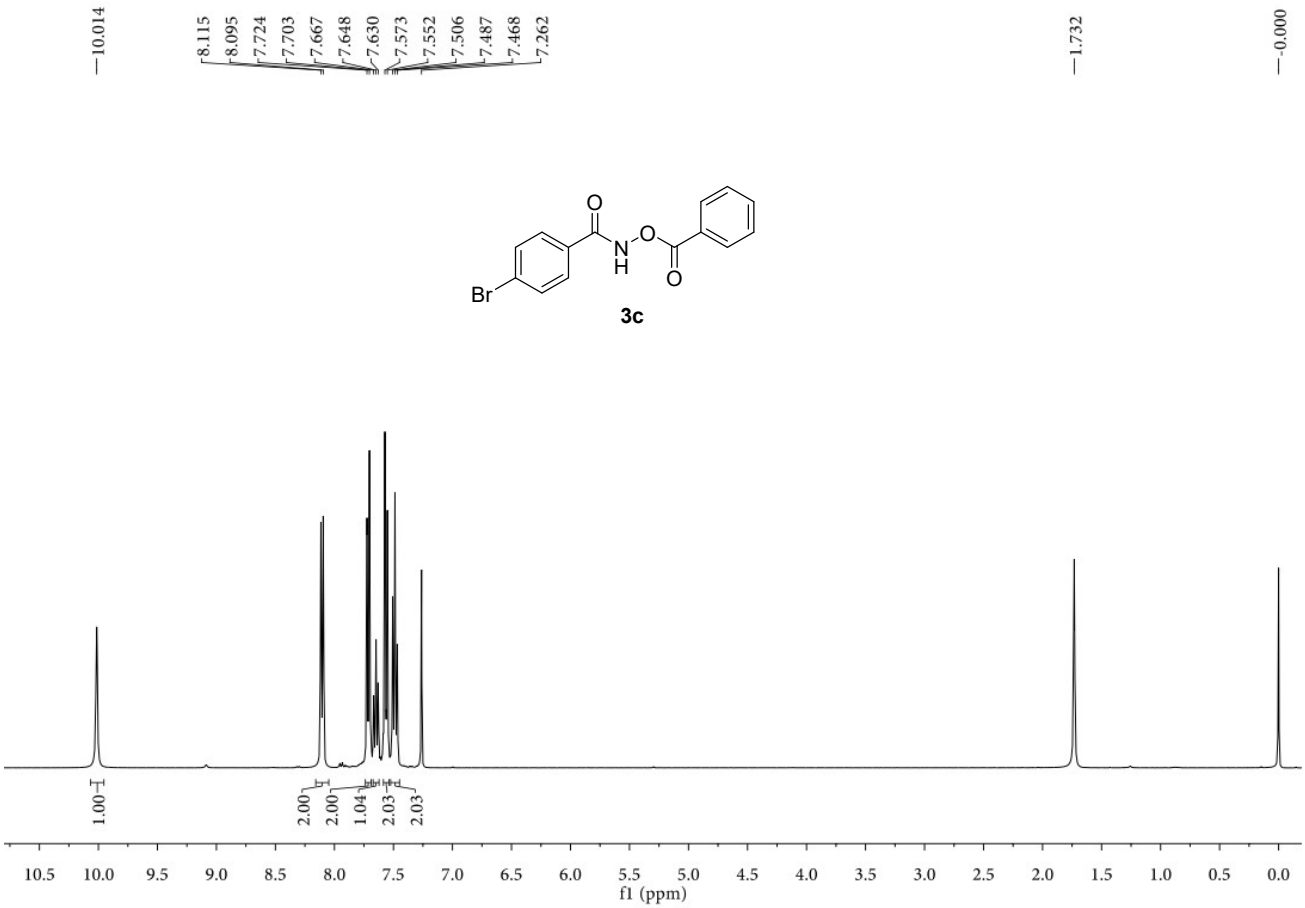
**3b**

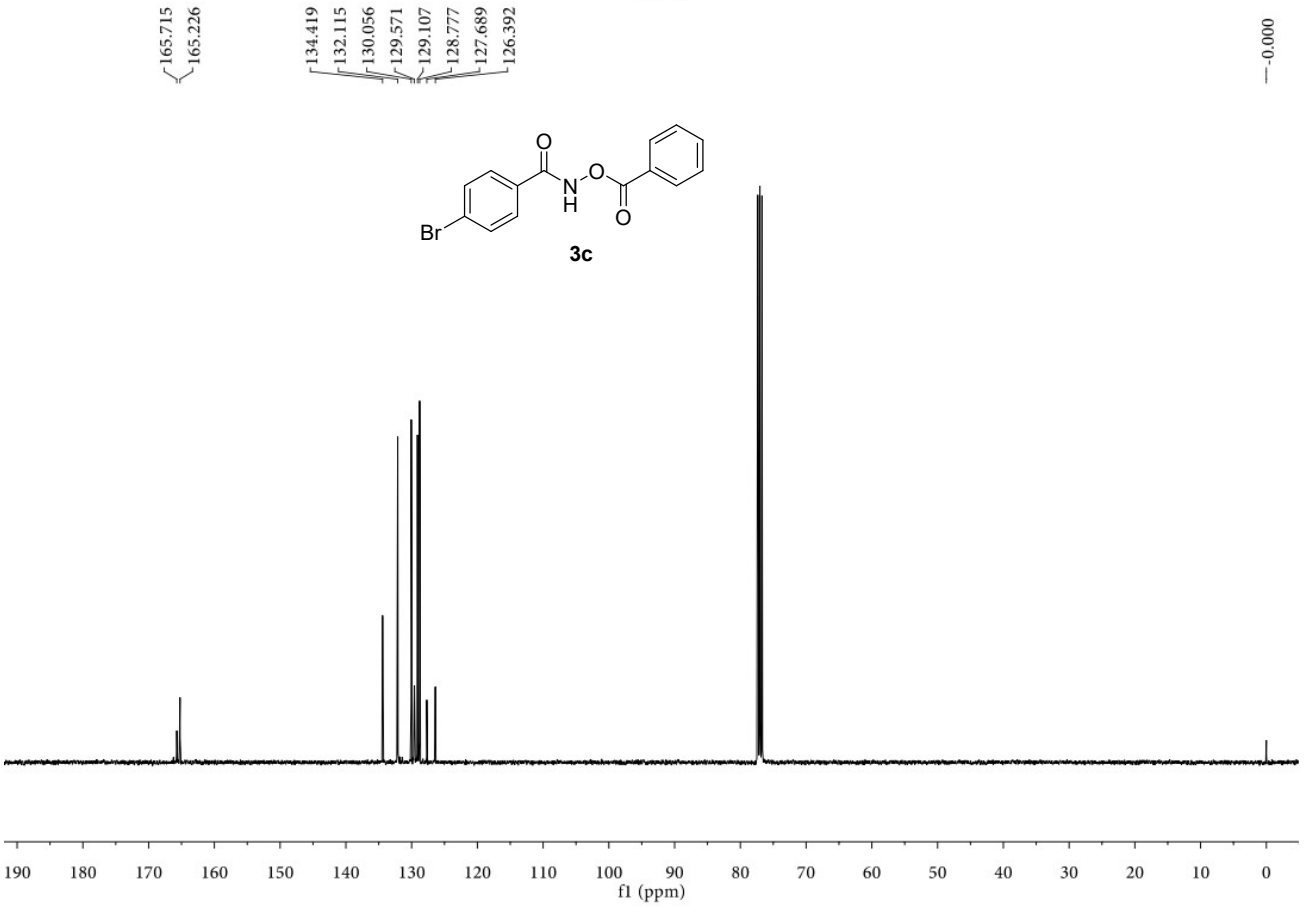

1.00
2.00
2.00
1.00
2.01
2.01
3.01
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

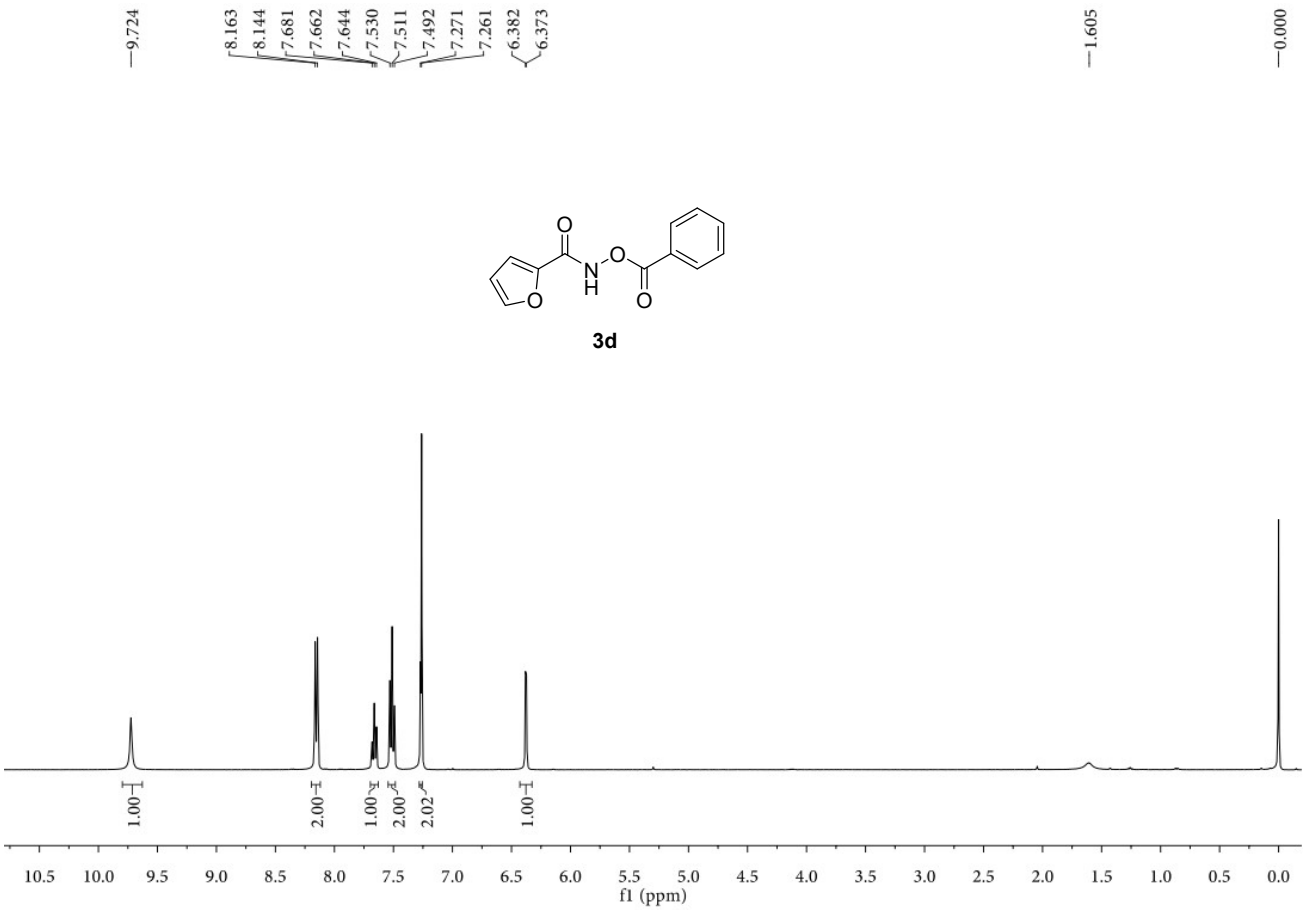

166.708
165.370
143.521
134.255
130.058
129.520
128.722
127.965
127.567
126.666
21.630
0.000
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

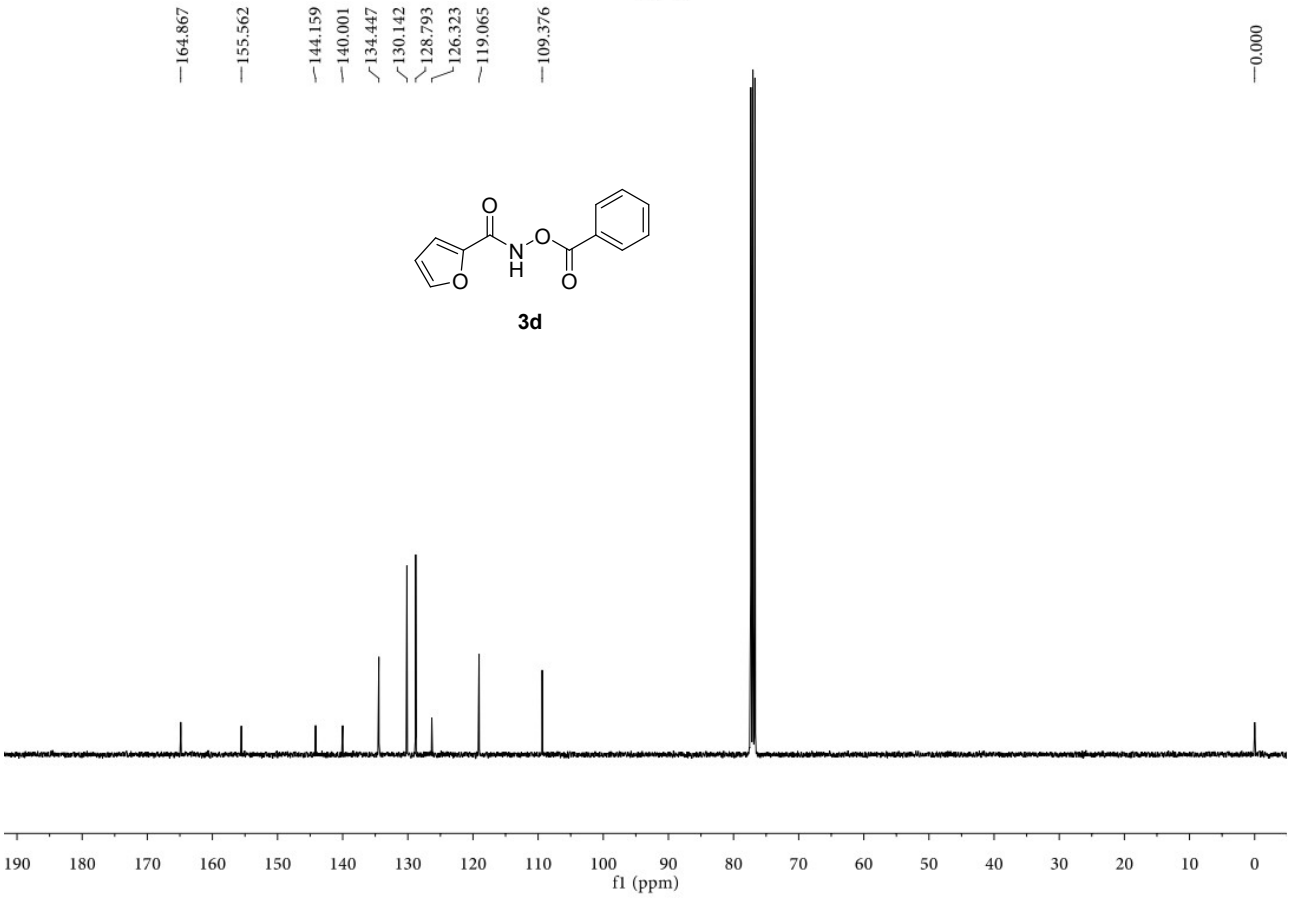


**3b**

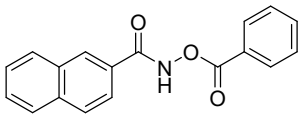

10.014
8.115
8.095
7.724
7.703
7.667
7.648
7.630
7.573
7.552
7.506
7.487
7.468
7.262
1.732
0.000
Br
3c
1.00
2.00
2.00
1.04
2.03
2.03
f1 (ppm)


165.715
165.226
134.419
132.115
130.056
129.571
129.107
128.777
127.689
126.392
0.000
Br
3c
f1 (ppm)

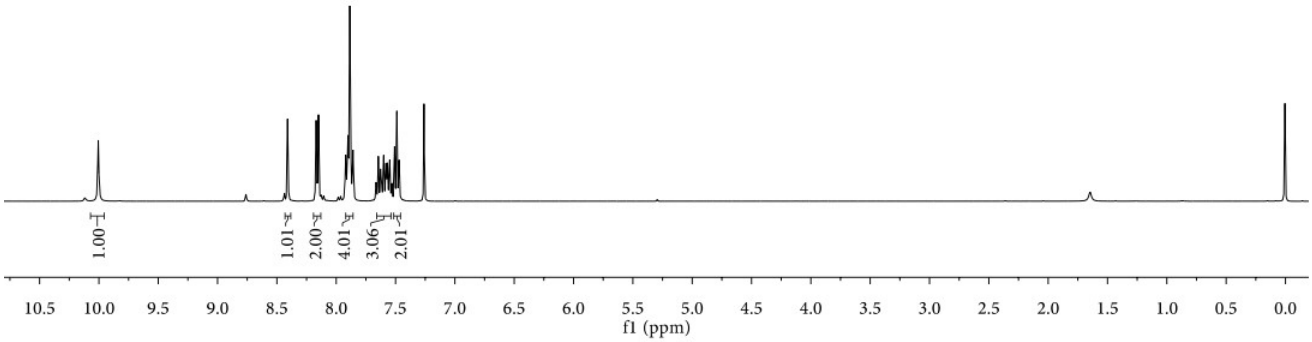

9.724
8.163
8.144
7.681
7.662
7.644
7.530
7.511
7.492
7.271
7.261
6.382
6.373
1.605
0.000
3d
1.00
2.00
1.00
2.00
2.02
1.00
f1 (ppm)


164.867
155.562
144.159
140.001
134.447
130.142
128.793
126.323
119.065
109.376
0.000
3d
f1 (ppm)

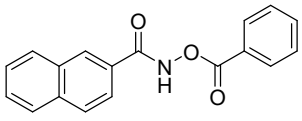

10.005
8.410
8.169
8.150
7.920
7.901
7.886
7.859
7.664
7.645
7.627
7.600
7.580
7.568
7.549
7.531
7.508
7.489
7.470
7.260
1.647
0.005



**3e**


1.00
1.01
2.00
4.01
3.06
2.01
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

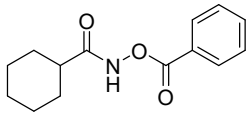

166.735
165.361
135.308
134.303
132.463
130.079
129.119
128.838
128.736
128.584
128.287
127.965
127.843
127.024
126.588
123.477
0.000



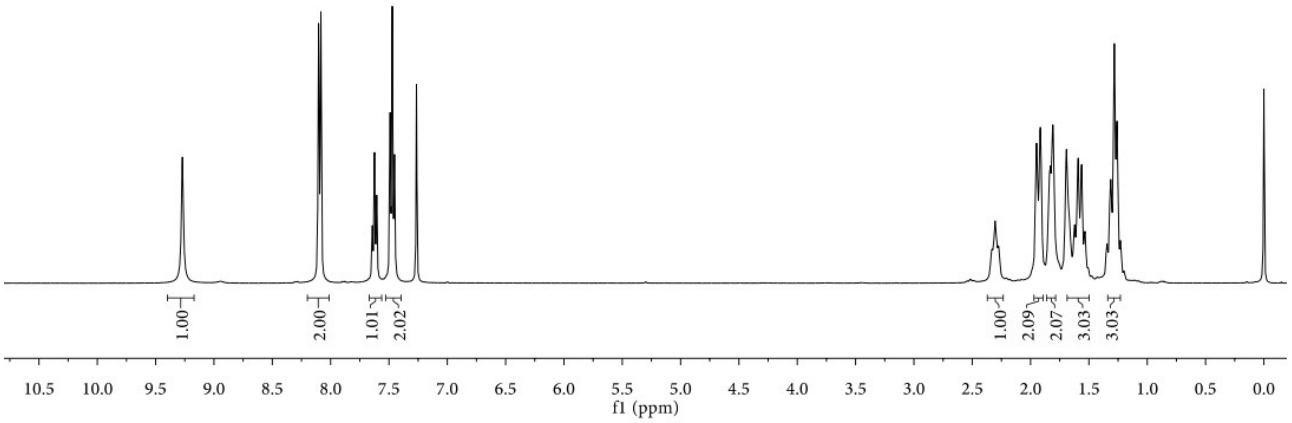
**3e**

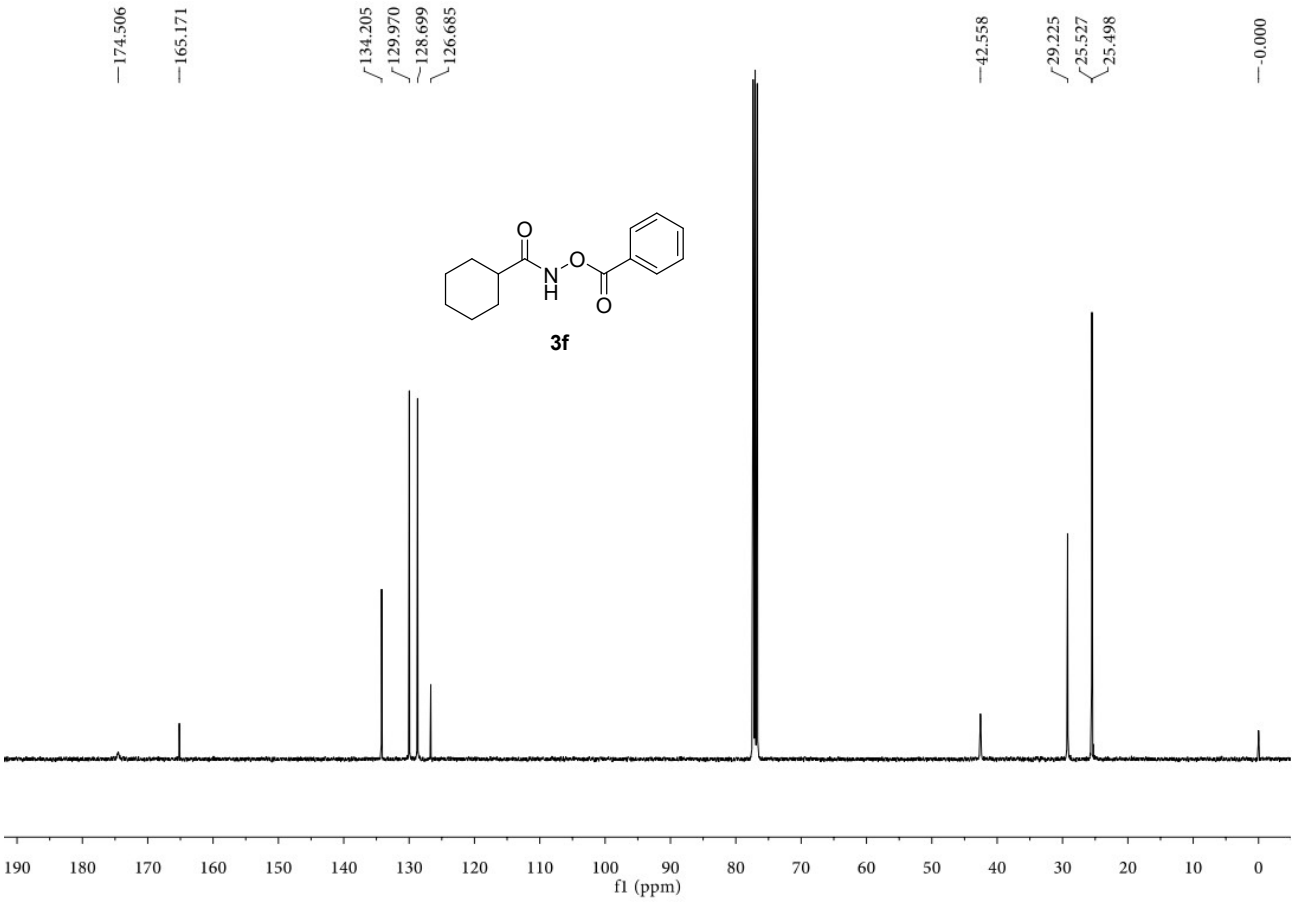

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


9.270
8.103
8.083
7.642
7.624
7.605
7.490
7.471
7.452
7.264
2.331
2.303
2.276
1.948
1.916
1.829
1.810
1.693
1.621
1.592
1.562
1.533
1.344
1.313
1.282
1.259
1.230
0.000



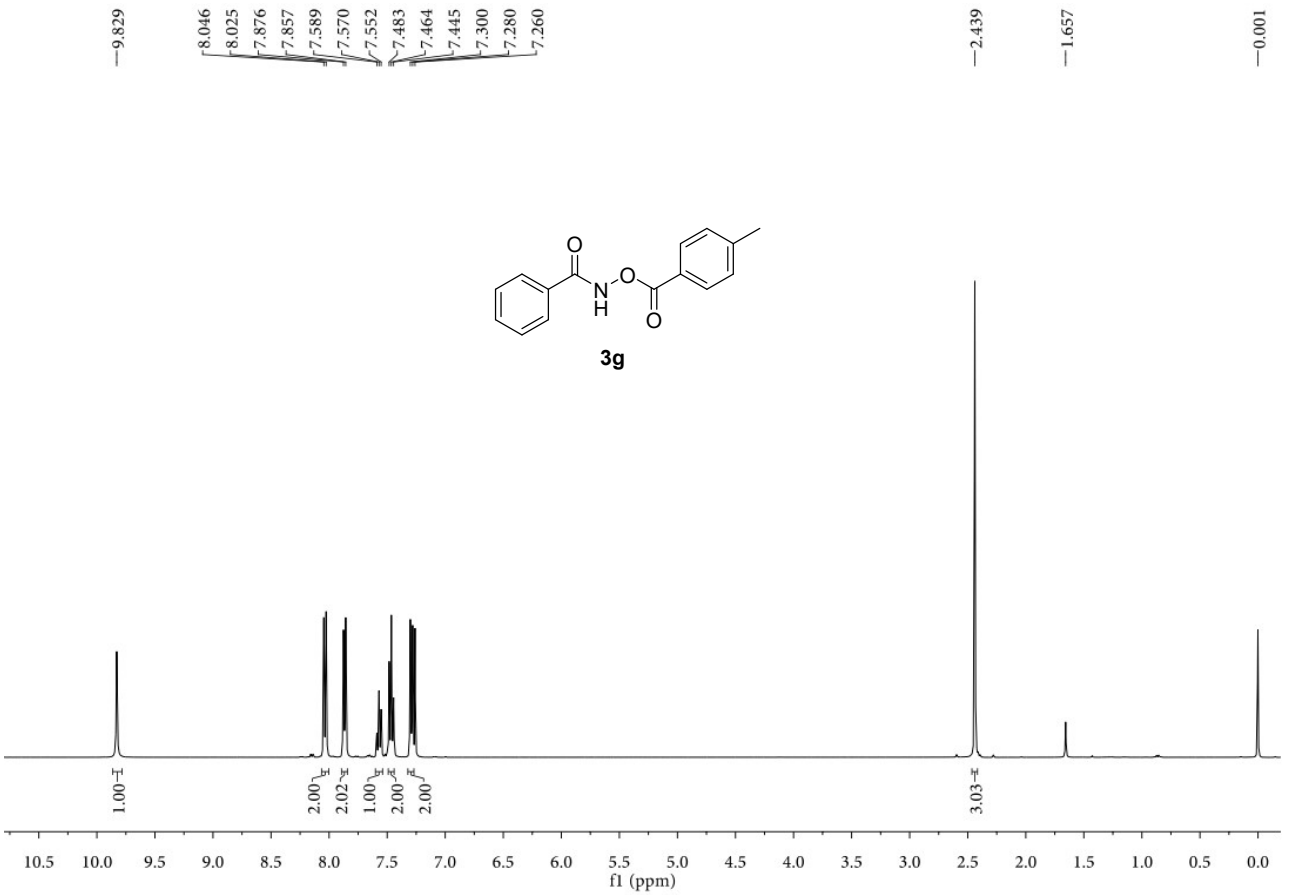
**3f**

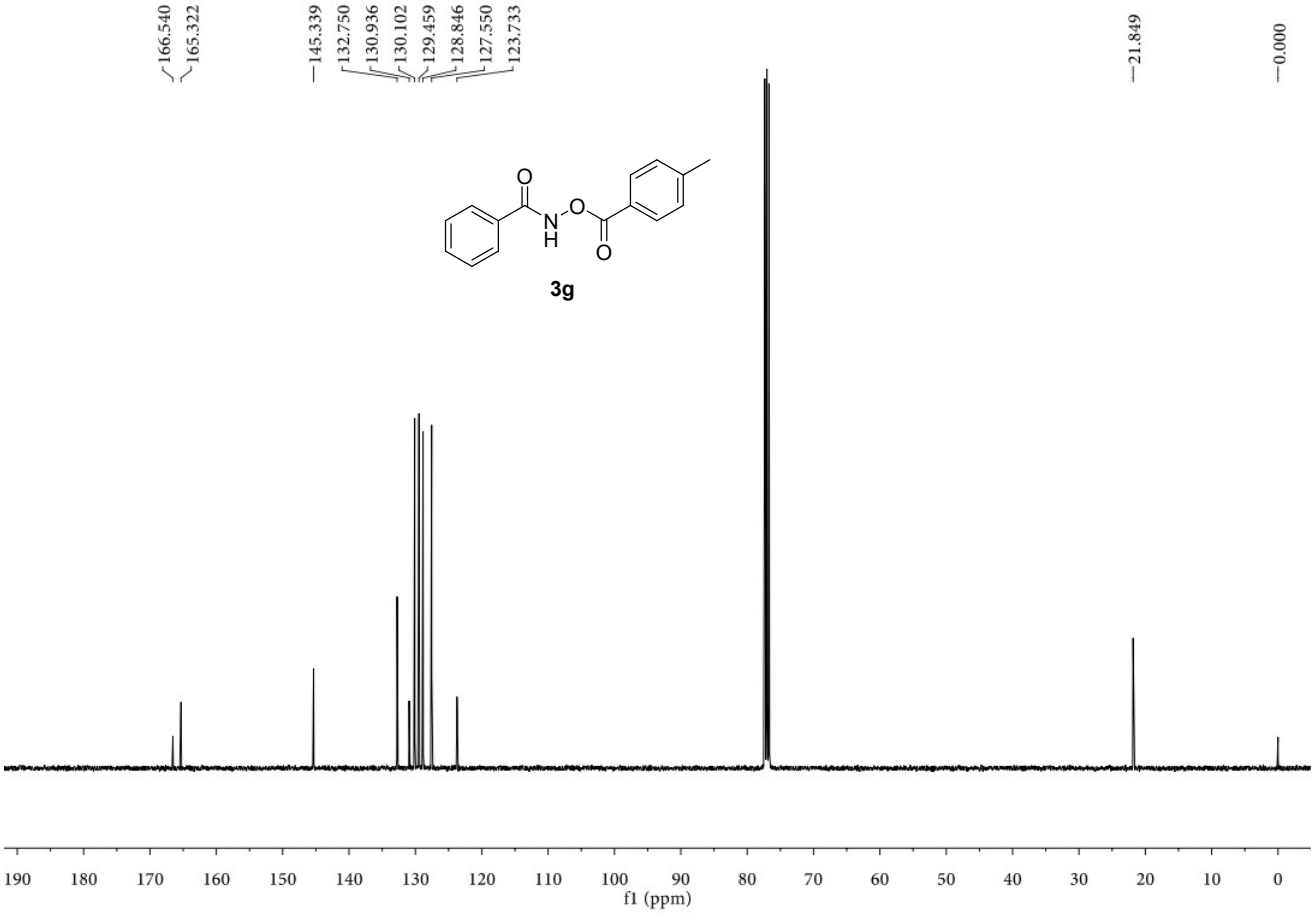

1.00
2.00
1.01
2.02
1.00
2.09
2.07
3.03
3.03
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

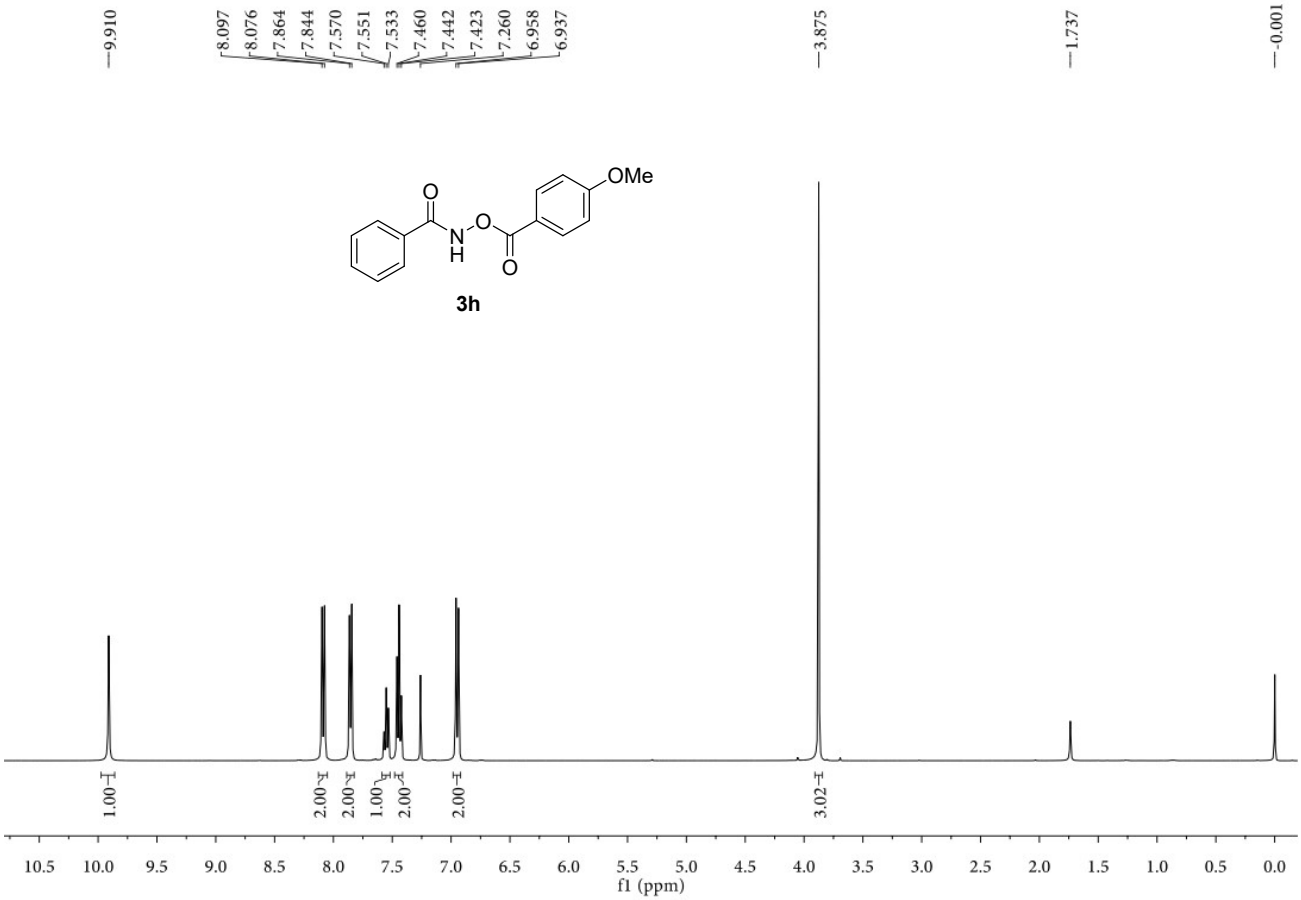

174.506
165.171
134.205
129.970
128.699
126.685
42.558
29.225
25.527
25.498
0.000
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

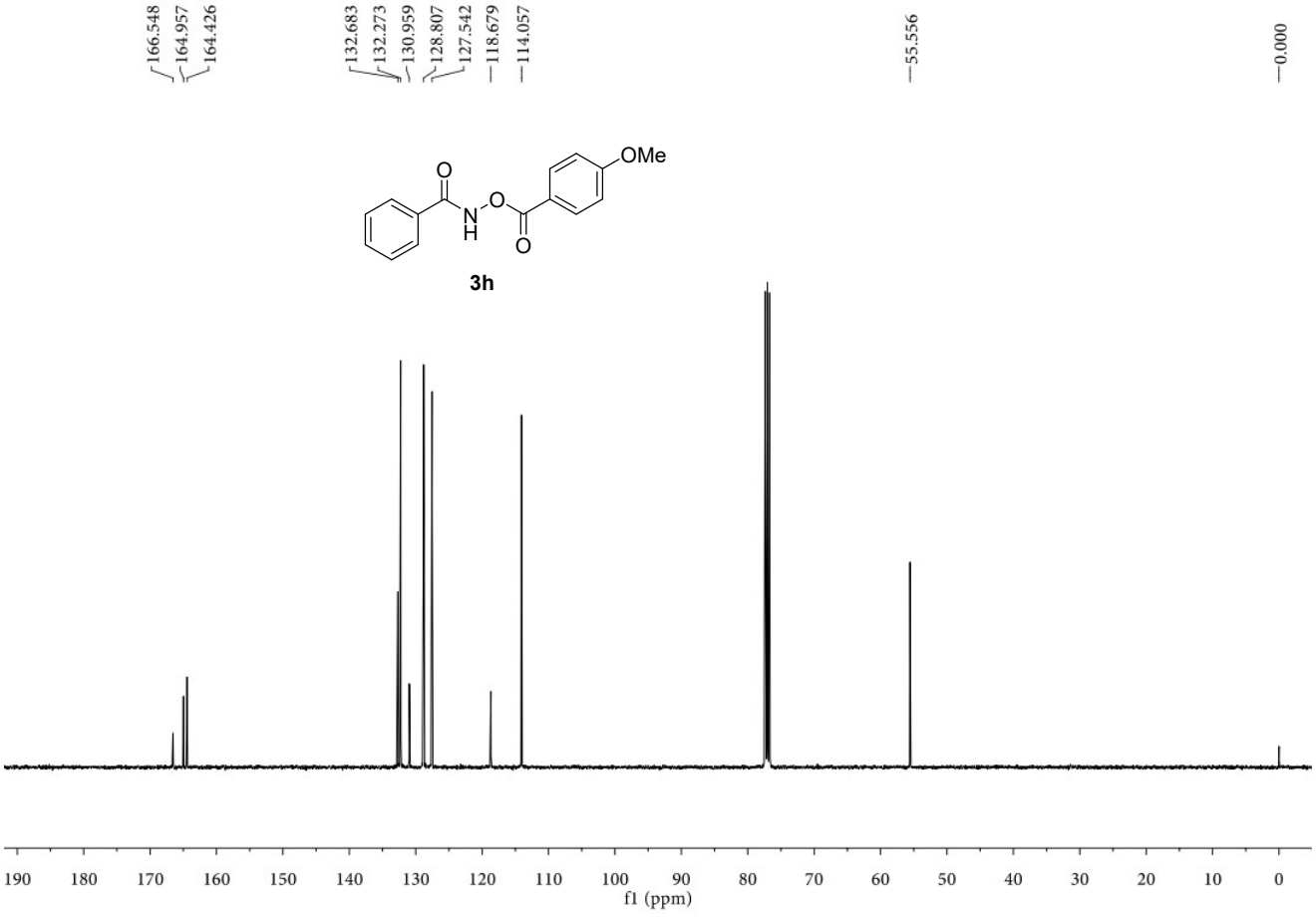


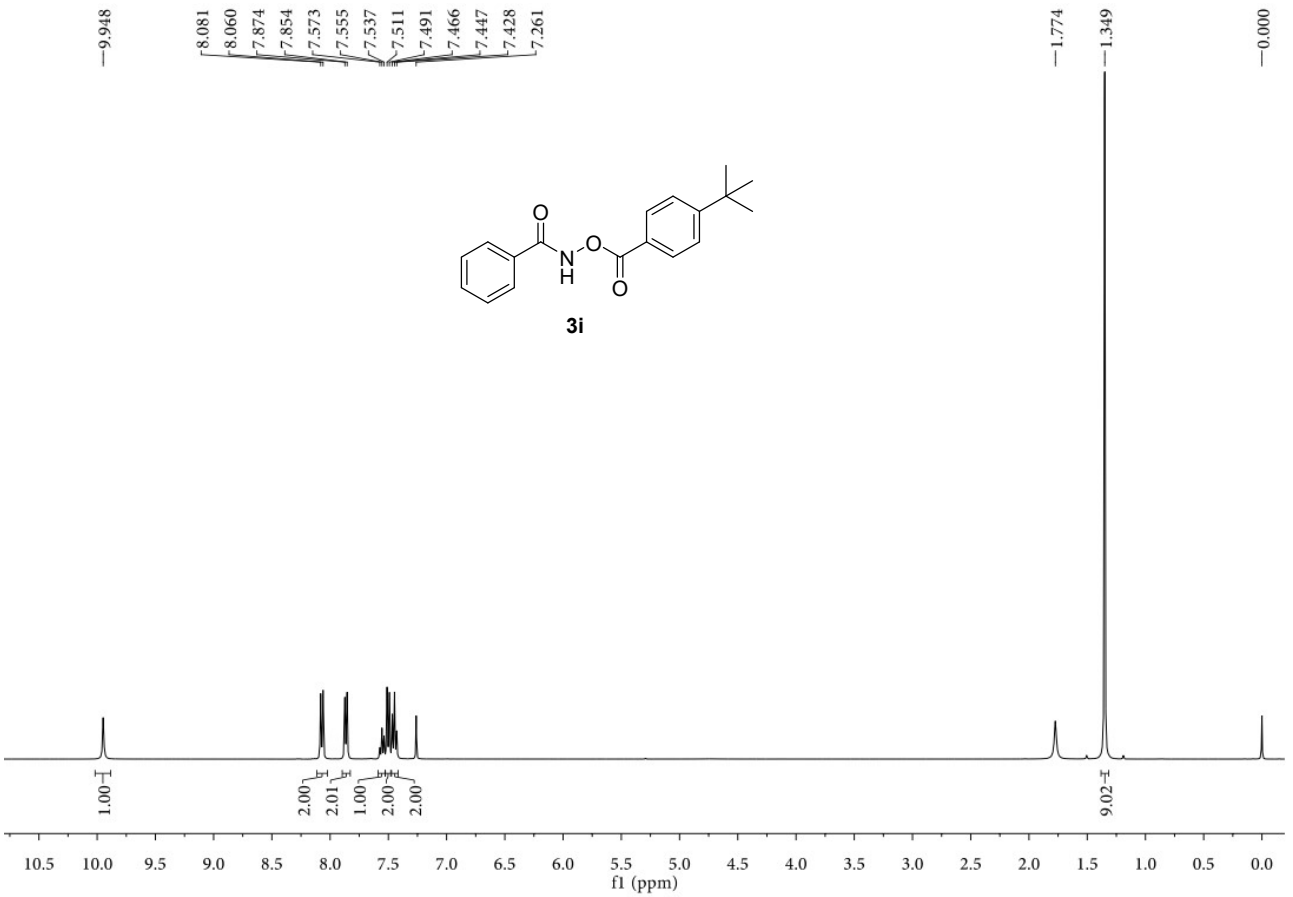
**3f**

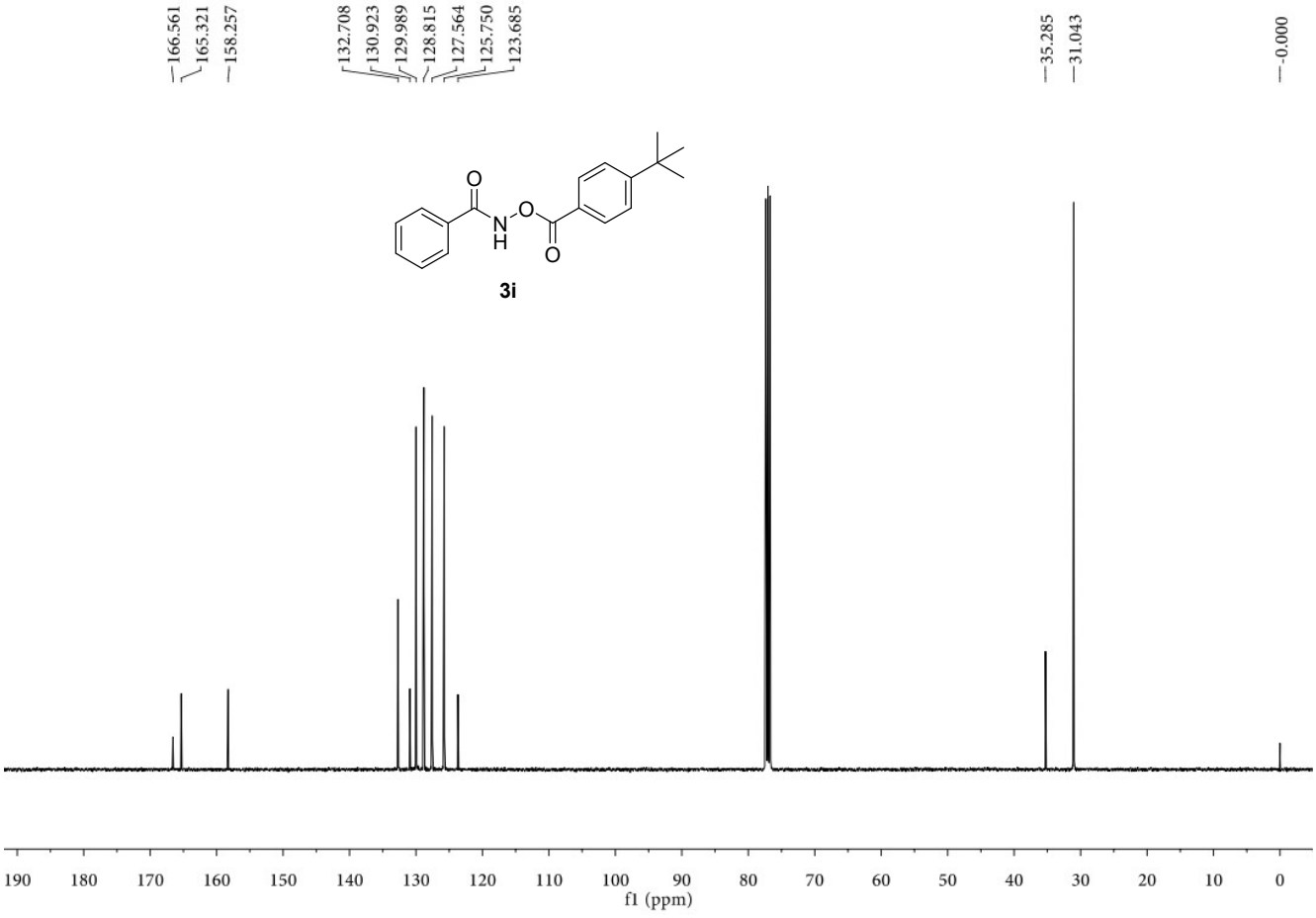

9.829
8.046
8.025
7.876
7.857
7.589
7.570
7.552
7.483
7.464
7.445
7.300
7.280
7.260
2.439
1.657
0.001
3g
1.00
2.00
2.02
1.00
2.00
2.00
3.03
fl (ppm)

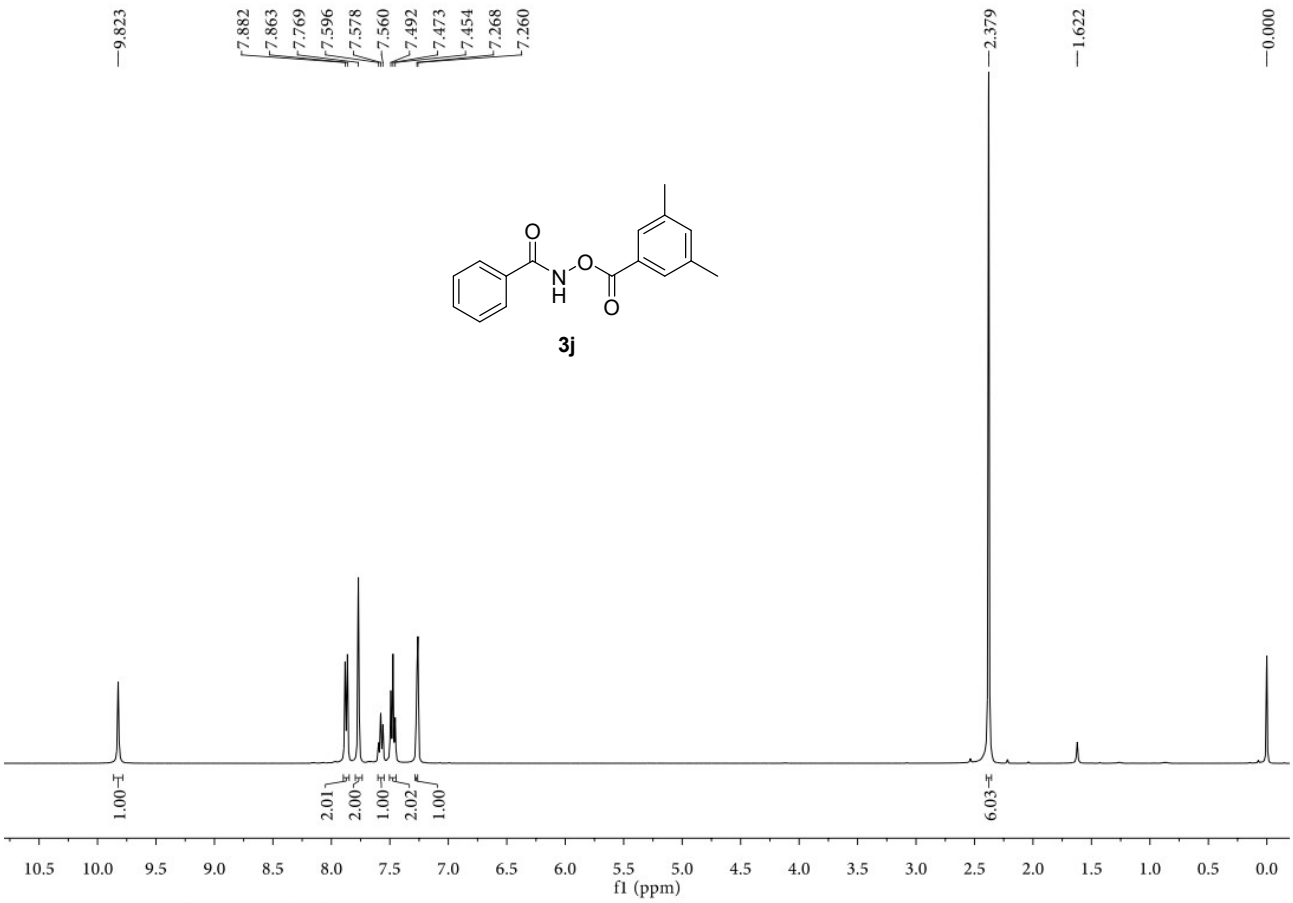

166.540
165.322
145.339
132.750
130.936
130.102
129.459
128.846
127.550
123.733
21.849
0.000
3g
fl (ppm)


9.910
8.097
8.076
7.864
7.844
7.570
7.551
7.533
7.460
7.442
7.423
7.260
6.958
6.937
3.875
1.737
-0.001
OMe
O
N
H
O
O
3h
1.00
2.00
2.00
1.00
2.00
2.00
3.02
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

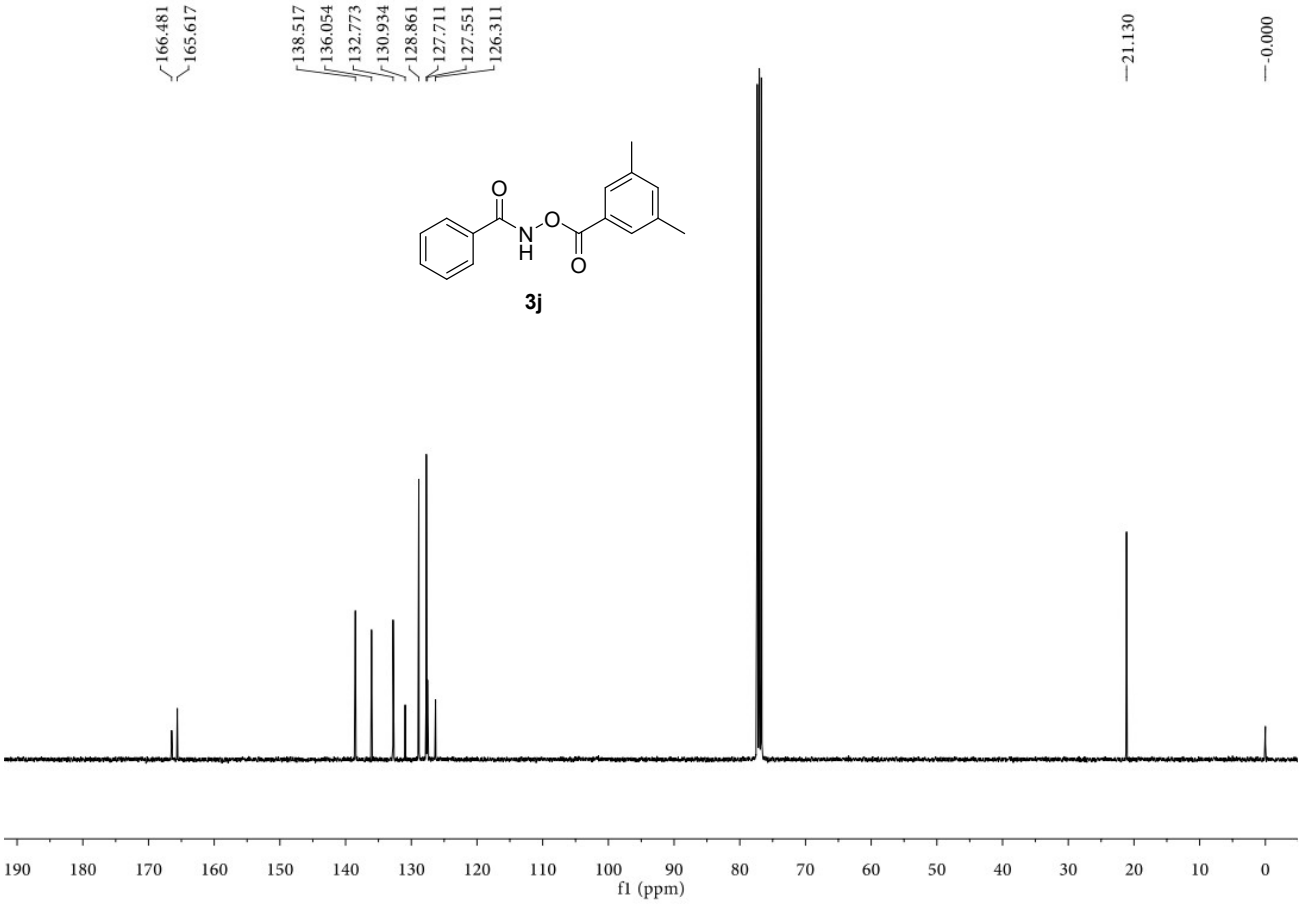

166.548
164.957
164.426
132.683
132.273
130.959
128.807
127.542
118.679
114.057
55.556
0.000
OMe
O
N
H
O
O
3h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


9.948
8.081
8.060
7.874
7.854
7.573
7.555
7.537
7.511
7.491
7.466
7.447
7.428
7.261
1.774
1.349
0.000
3i
1.00
2.00
2.01
1.00
2.00
2.00
9.02
f1 (ppm)

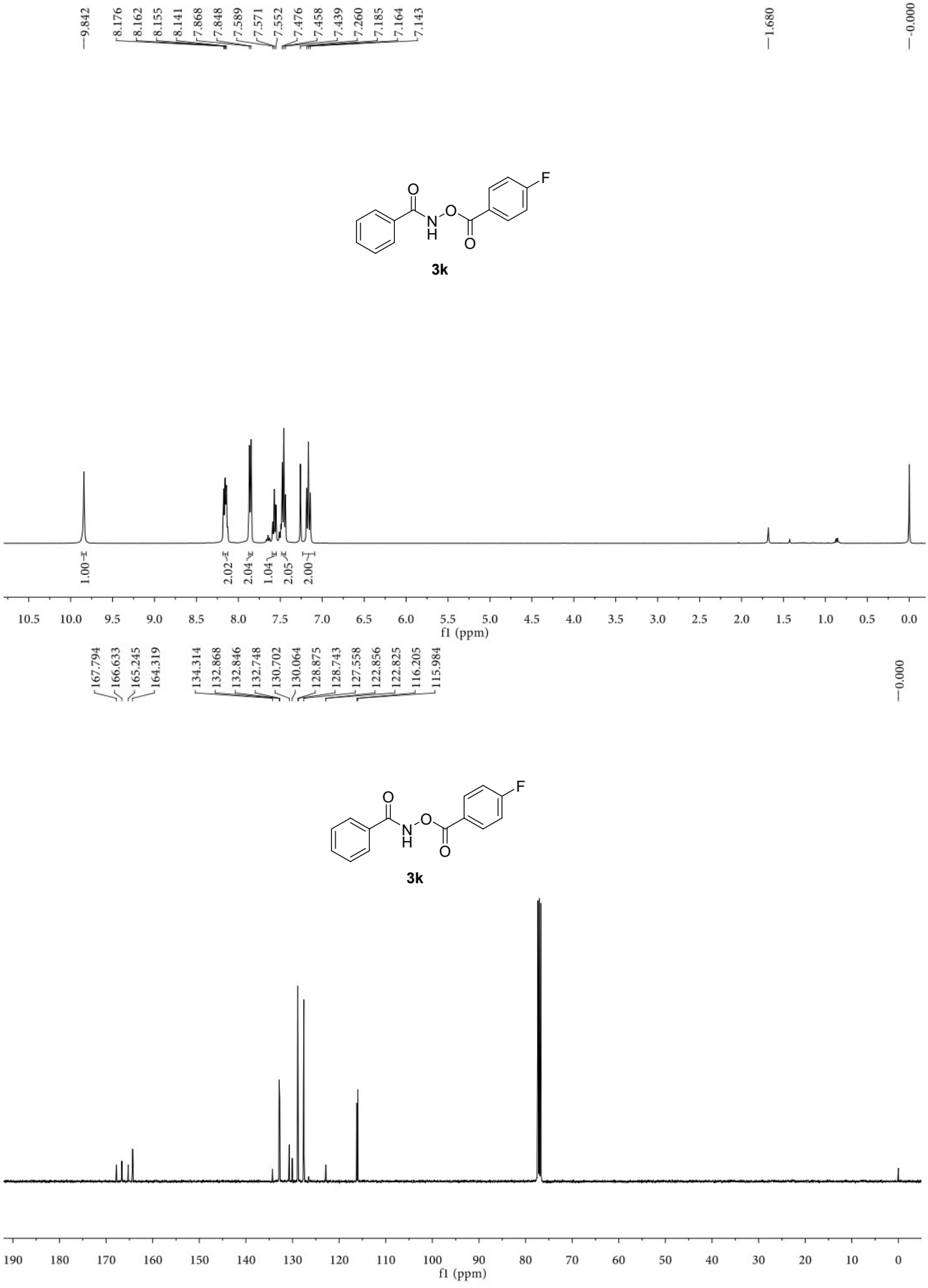

166.561
165.321
158.257
132.708
130.923
129.989
128.815
127.564
125.750
123.685
35.285
31.043
-0.000
3i
f1 (ppm)


9.823
7.882
7.863
7.769
7.596
7.578
7.560
7.492
7.473
7.454
7.268
7.260
2.379
1.622
0.000
1.00
2.01
2.00
1.00
2.02
1.00
6.03
f1 (ppm)

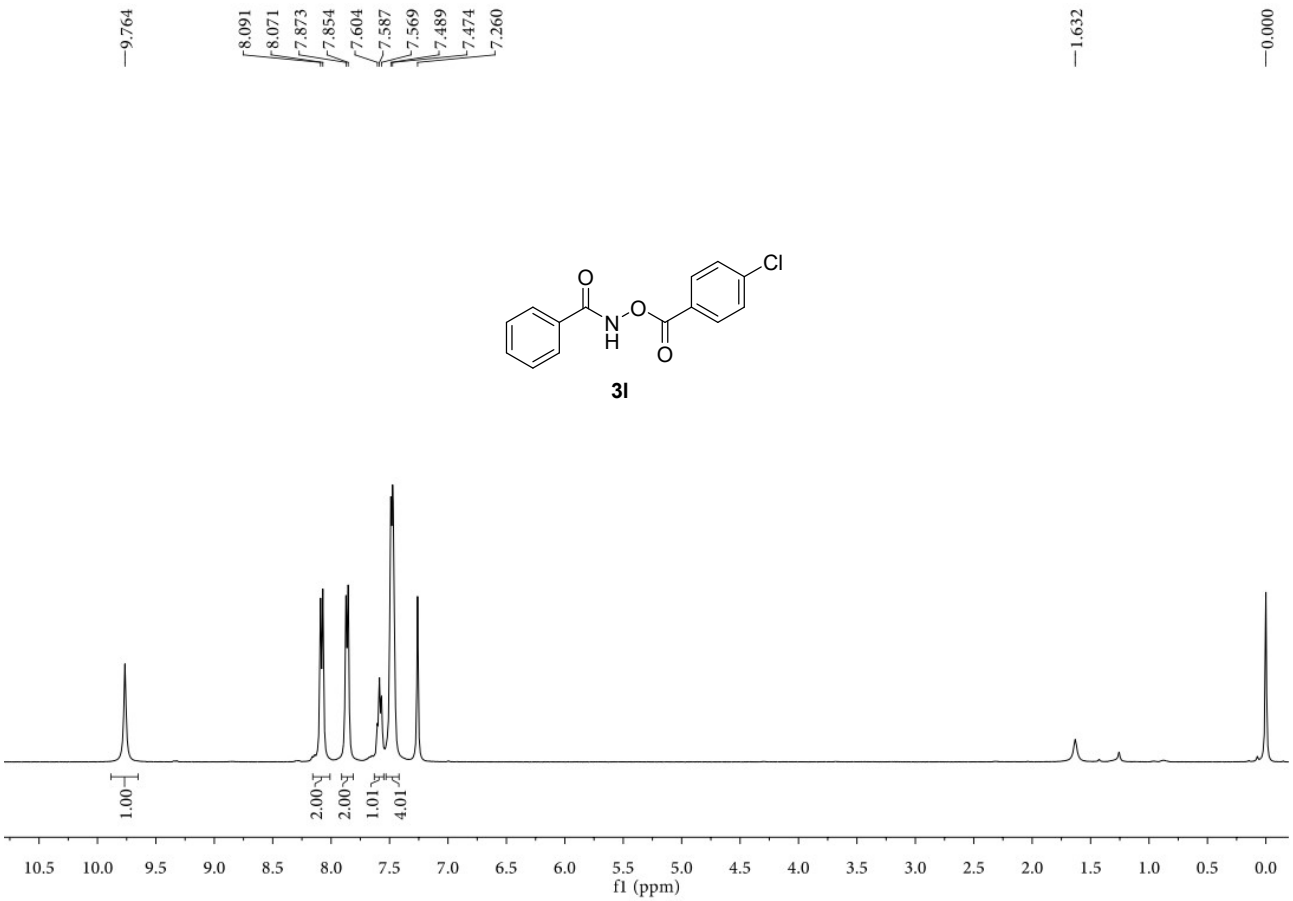
**3j**

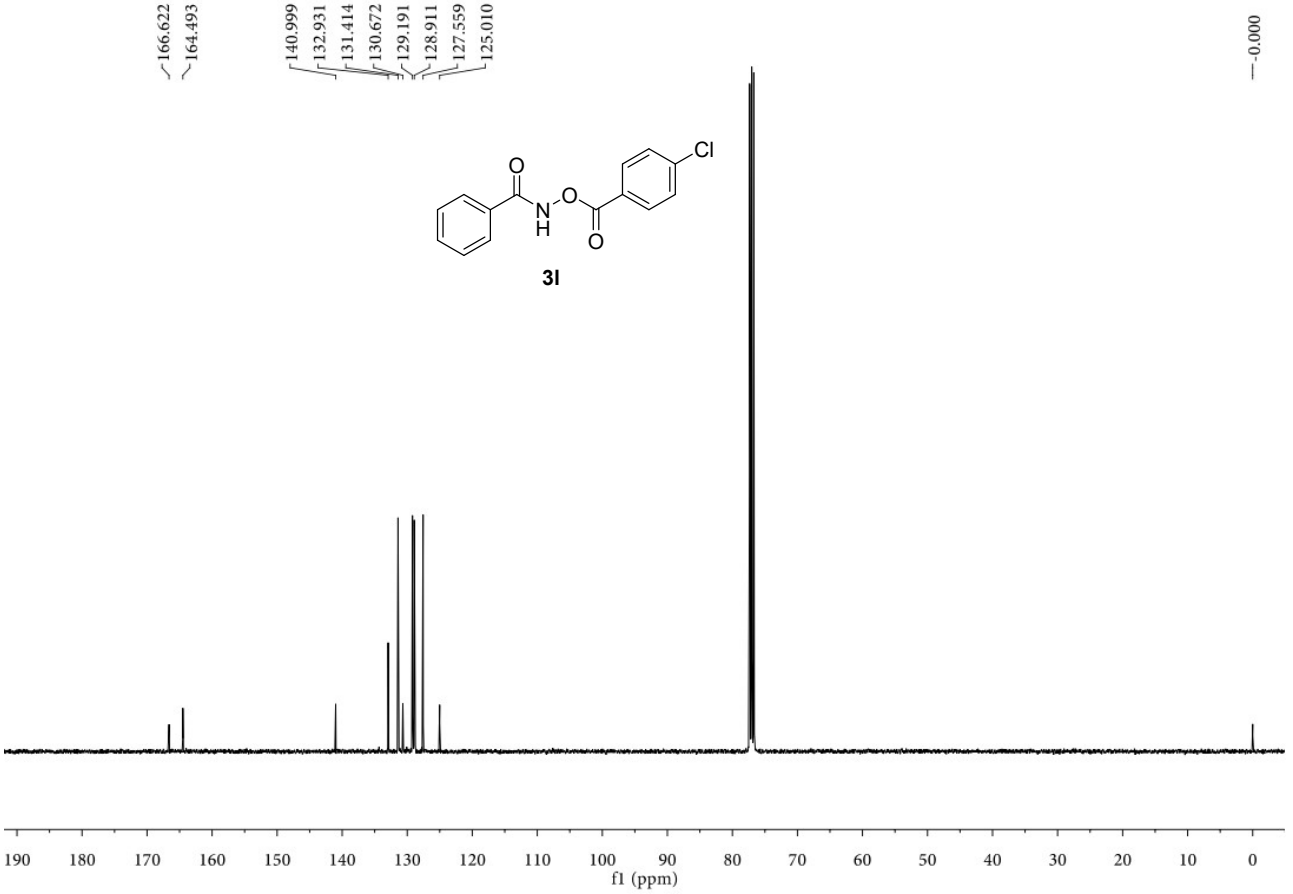

166.481
165.617
138.517
136.054
132.773
130.934
128.861
127.711
127.551
126.311
21.130
-0.000
f1 (ppm)

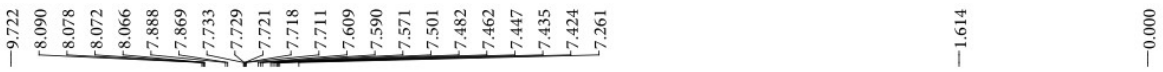
**3j**

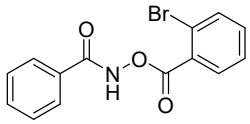

9.842
8.176
8.162
8.155
8.141
7.868
7.848
7.589
7.571
7.552
7.476
7.458
7.439
7.260
7.185
7.164
7.143
1.680
0.000
F
O
N
H
O
O
3k
1.00
2.02
2.04
1.04
2.05
2.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
167.794
166.633
165.245
164.319
134.314
132.868
132.846
132.748
130.702
130.064
128.875
128.743
127.558
122.856
122.825
116.205
115.984
0.000
F
O
N
H
O
O
3k
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-102.645
O
N
H
O
O
F
3k
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

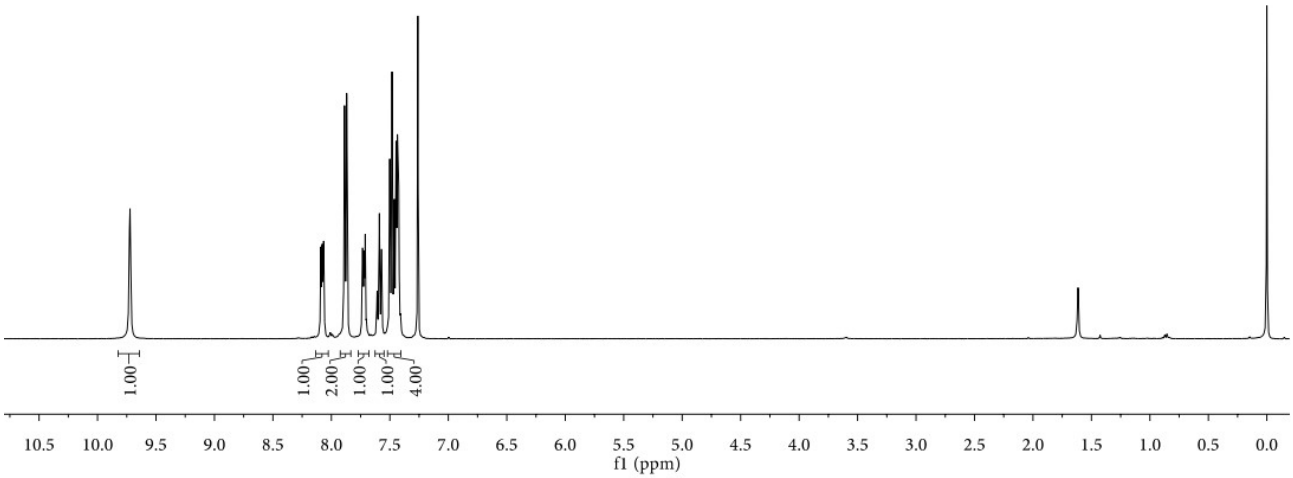

9.764
8.091
8.071
7.873
7.854
7.604
7.587
7.569
7.489
7.474
7.260
1.632
0.000
Cl
O
N
H
O
O
3l
1.00
2.00
2.00
1.01
4.01
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.622
164.493
140.999
132.931
131.414
130.672
129.191
128.911
127.559
125.010
-0.000
Cl
O
N
H
O
O
3l
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

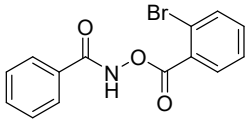

9.722
8.090
8.078
8.072
8.066
7.888
7.869
7.733
7.729
7.721
7.718
7.711
7.609
7.590
7.571
7.501
7.482
7.462
7.447
7.435
7.424
7.261
1.614
0.000


Br
O
N
H
O
O

**3m**


1.00
1.00
2.00
1.00
1.00
4.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

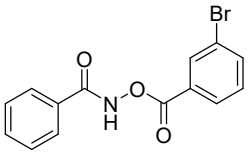

166.743
164.664
134.609
133.941
132.945
132.178
130.605
128.904
128.411
127.567
127.456
122.544
-0.000


Br
O
N
H
O
O

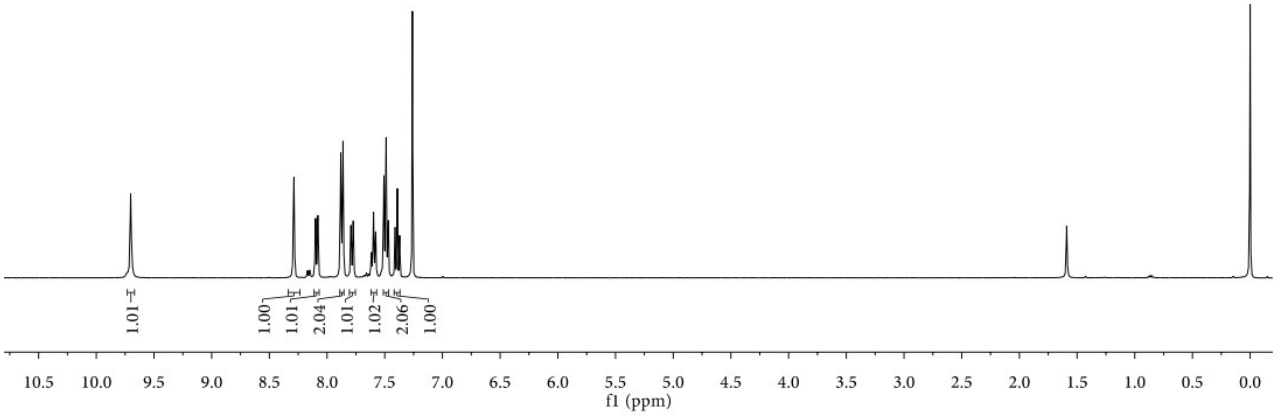
**3m**

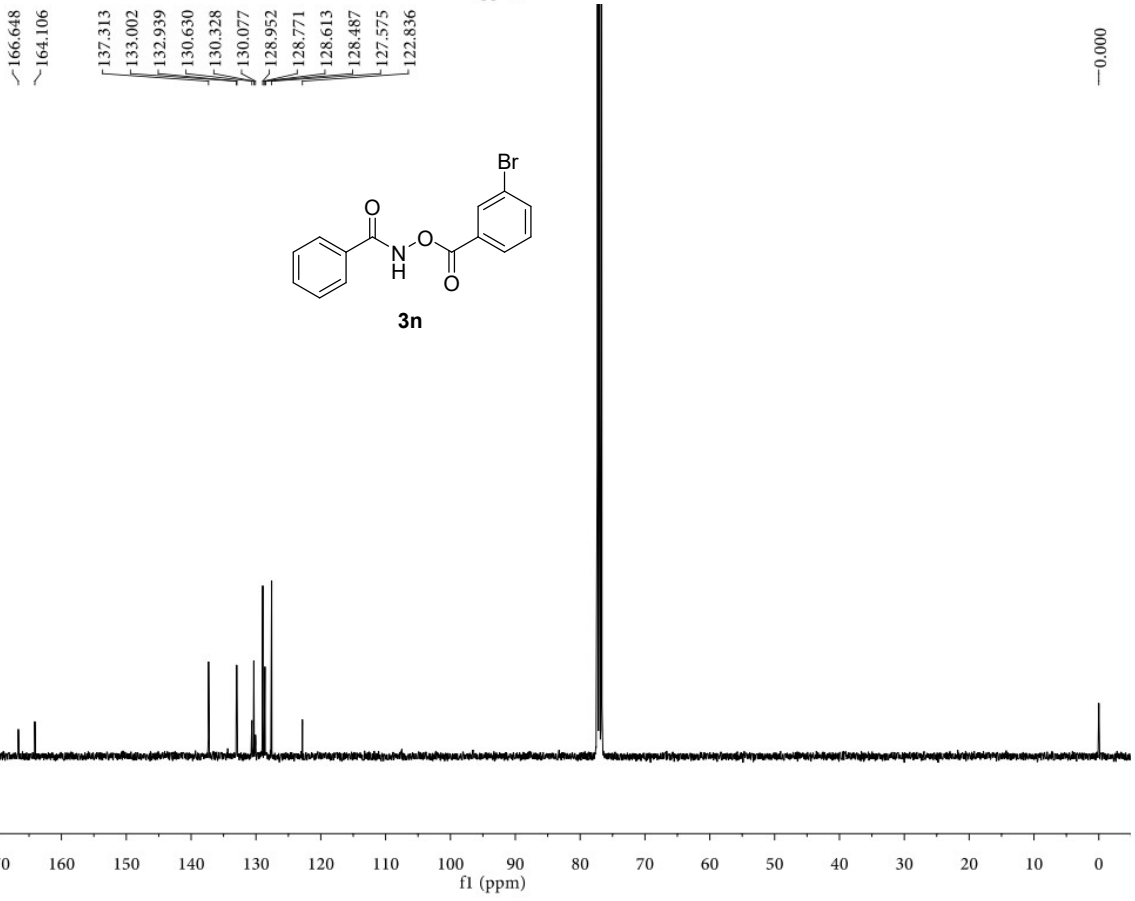

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


9.702
8.287
8.099
8.080
7.881
7.862
7.793
7.773
7.616
7.597
7.579
7.507
7.488
7.469
7.410
7.391
7.371
7.260
1.590
-0.000

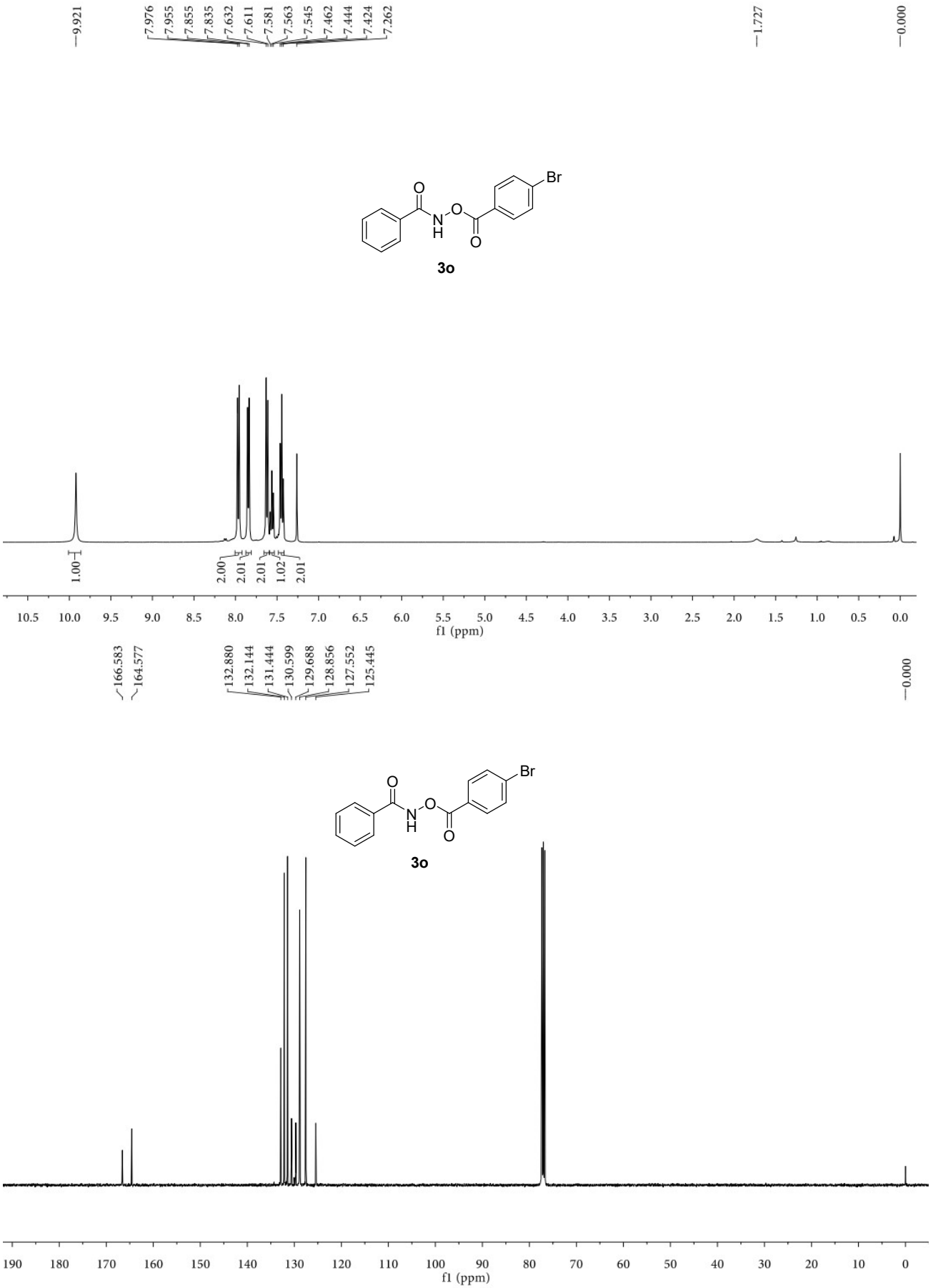

Br
O
N
H
O
O

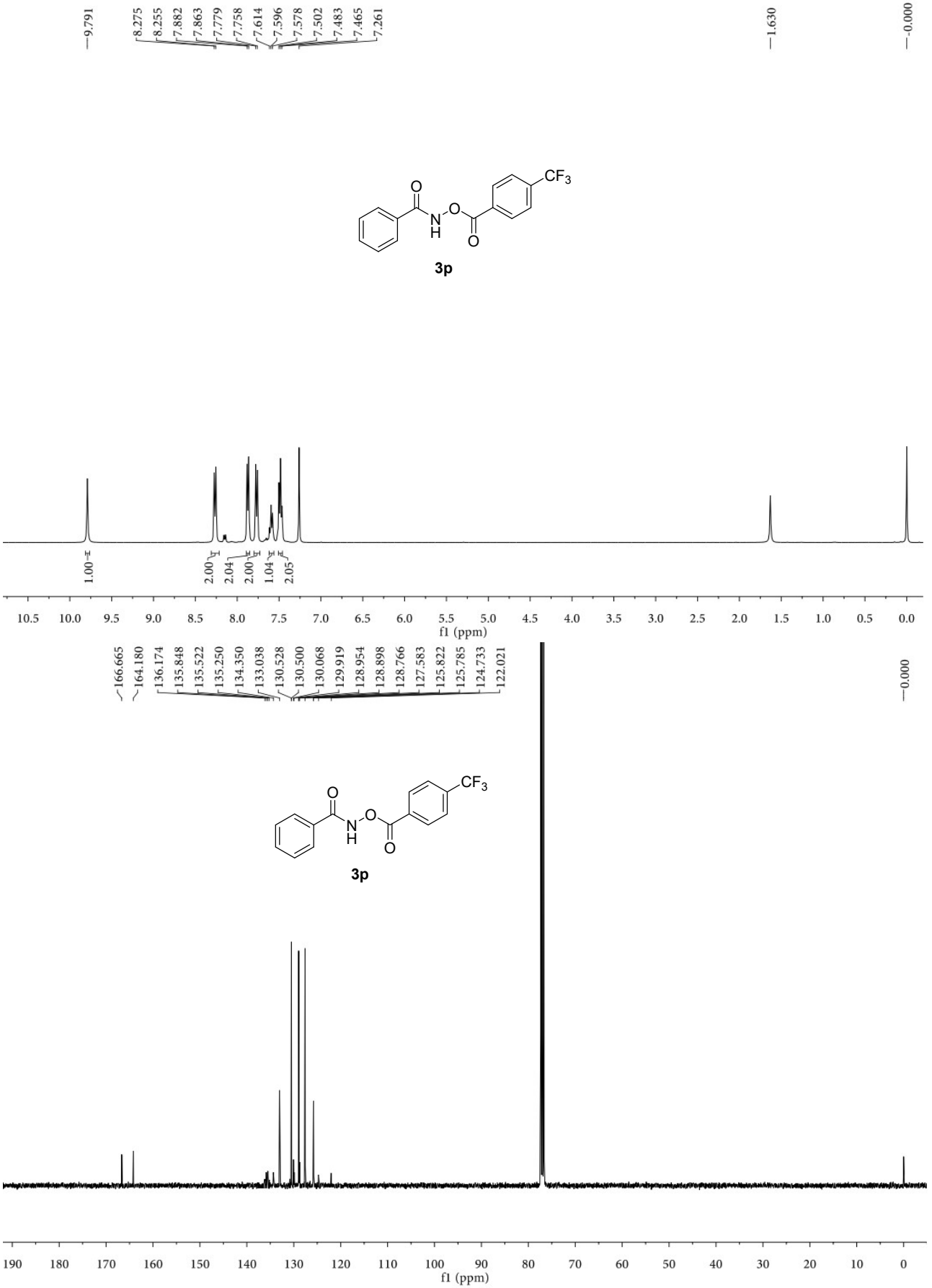
**3n**


1.01
1.00
1.01
2.04
1.01
1.02
2.06
1.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.648
164.106
137.313
133.002
132.939
130.630
130.328
130.077
128.952
128.771
128.613
128.487
127.575
122.836
-0.000
Br
O
N
H
O
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

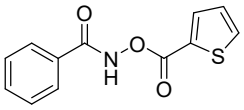
**3n**


9.921
7.976
7.955
7.855
7.835
7.632
7.611
7.581
7.563
7.545
7.462
7.444
7.424
7.262
1.727
0.000
Br
O
N
H
O
O
3o
1.00
2.00
2.01
2.01
1.02
2.01
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
166.583
164.577
132.880
132.144
131.444
130.599
129.688
128.856
127.552
125.445
0.000
Br
O
N
H
O
O
3o
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

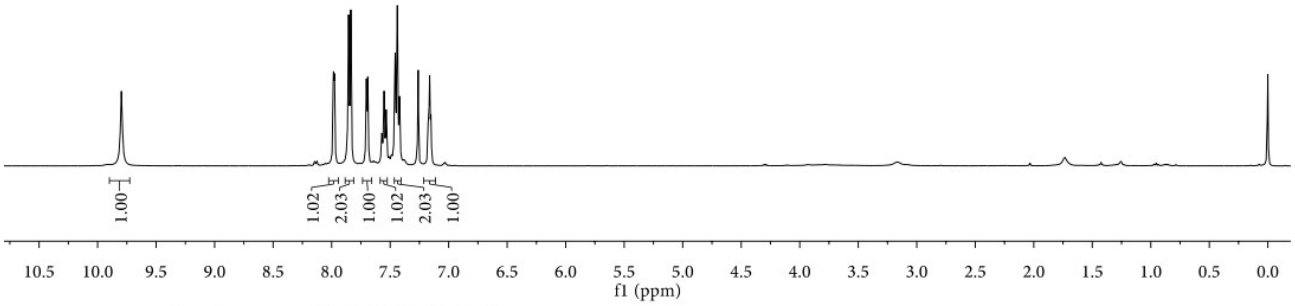

9.791
8.275
8.255
7.882
7.863
7.779
7.758
7.614
7.596
7.578
7.502
7.483
7.465
7.261
1.630
-0.000
CF3
O
N
H
O
O
3p
1.00
2.00
2.04
2.00
1.04
2.05
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
166.665
164.180
136.174
135.848
135.522
135.250
134.350
133.038
130.528
130.500
130.068
129.919
128.954
128.898
128.766
127.583
125.822
125.785
124.733
122.021
0.000
CF3
O
N
H
O
O
3p
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-63.318
O
N
H
O
O
CF3
3p
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

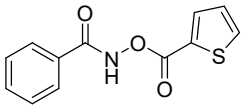

9.796
7.984
7.976
7.855
7.836
7.703
7.691
7.571
7.552
7.534
7.457
7.438
7.420
7.261
7.172
7.162
7.152
0.000



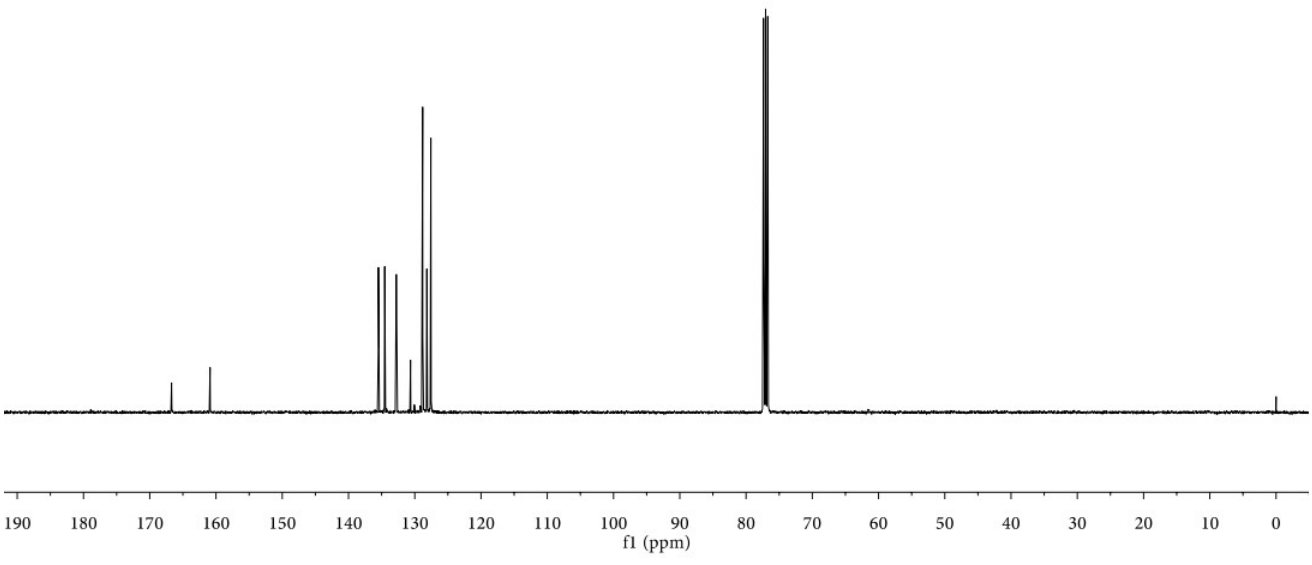
**3q**

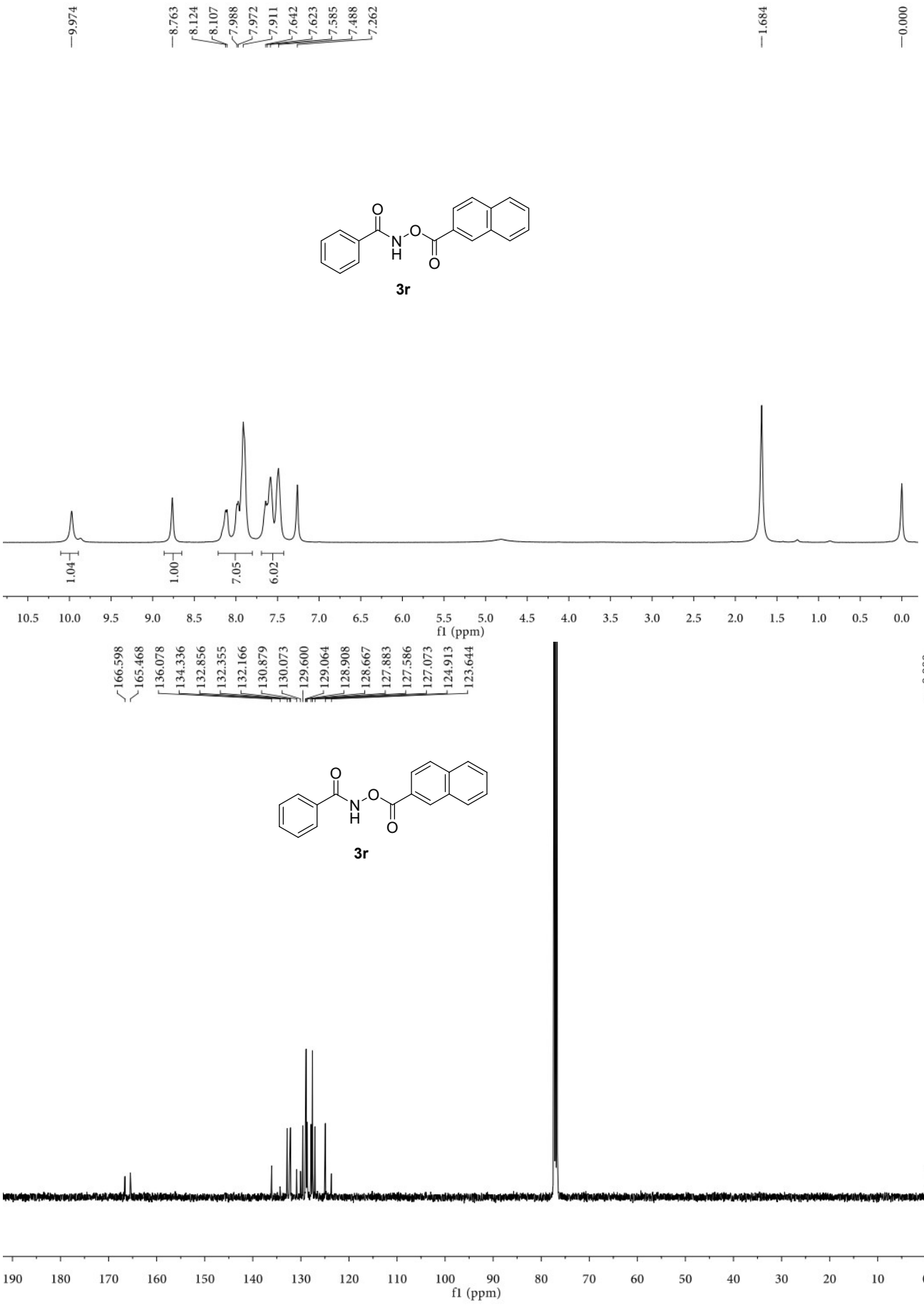

1.00
1.02
2.03
1.00
1.02
2.03
1.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

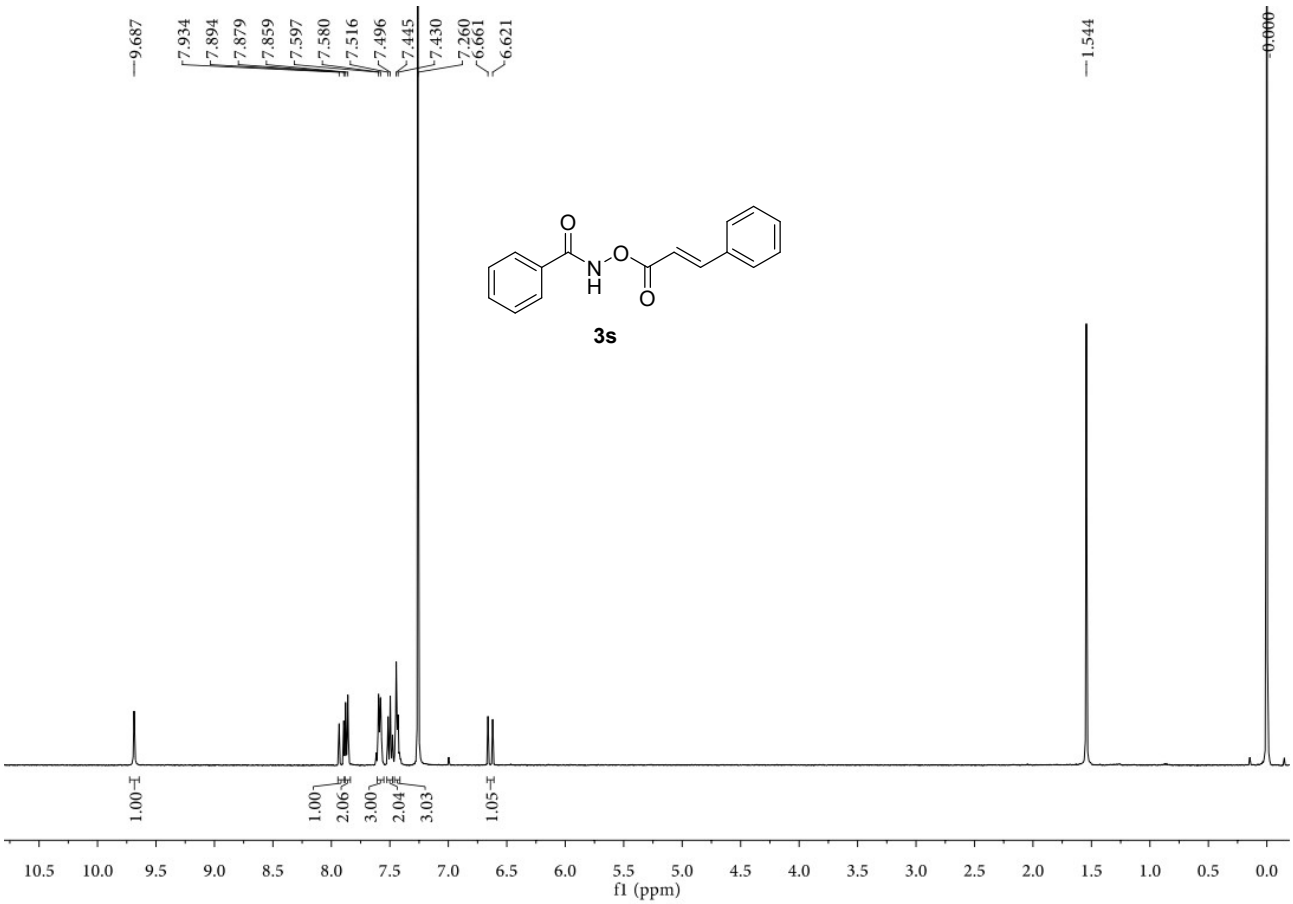

166.709
160.891
135.488
134.531
132.782
130.638
128.809
128.181
127.574
0.000



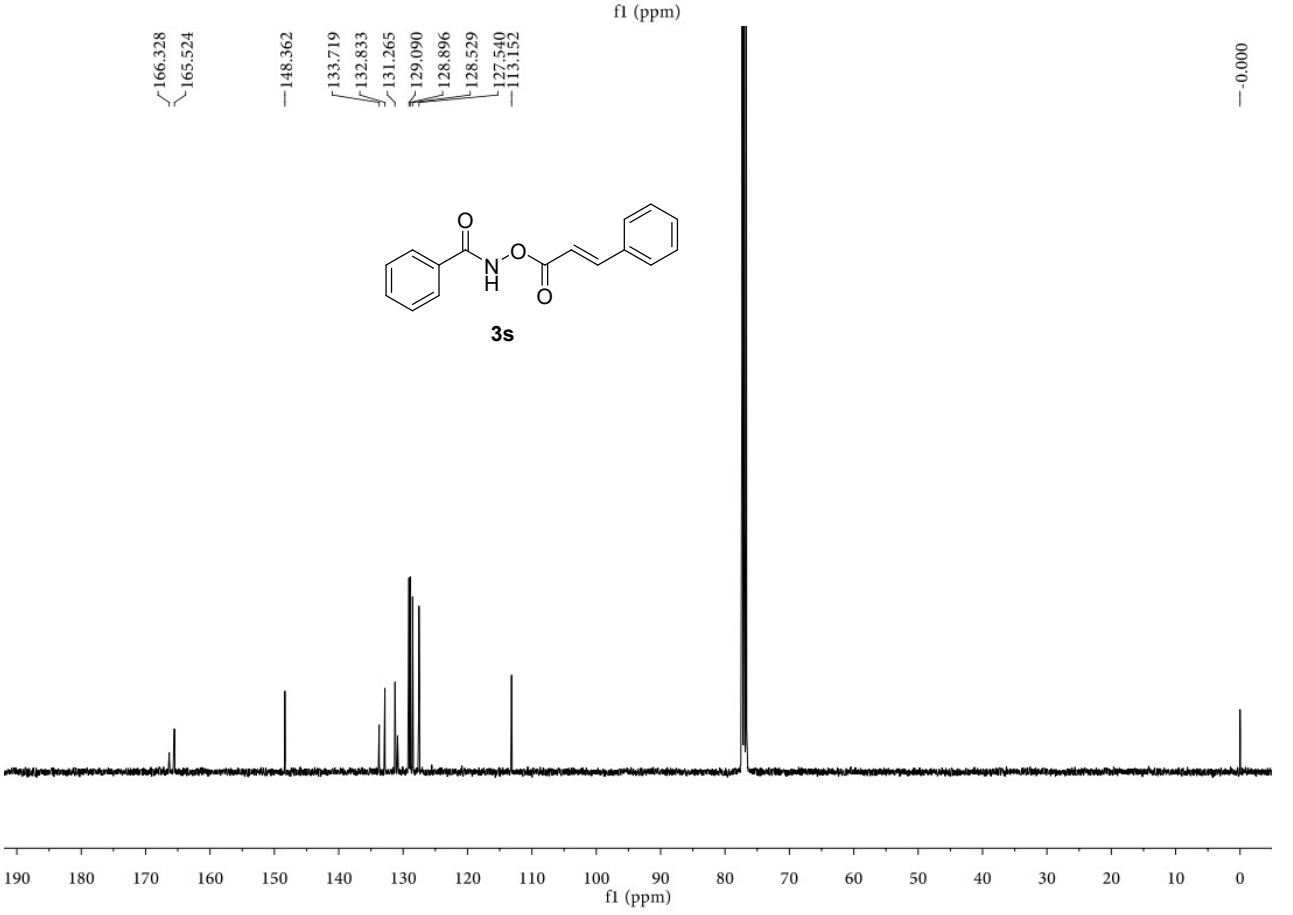
**3q**


190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


9.974
8.763
8.124
8.107
7.988
7.972
7.911
7.642
7.623
7.585
7.488
7.262
1.684
0.000
3r
1.04
1.00
7.05
6.02
f1 (ppm)
166.598
165.468
136.078
134.336
132.856
132.355
132.166
130.879
130.073
129.600
129.064
128.908
128.667
127.883
127.586
127.073
124.913
123.644
-0.000
3r
f1 (ppm)

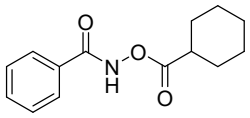

9.687
7.934
7.894
7.879
7.859
7.597
7.580
7.516
7.496
7.445
7.430
7.260
6.661
6.621
1.544
0.000
1.00
2.06
3.00
2.04
3.03
1.05
f1 (ppm)

**3s**

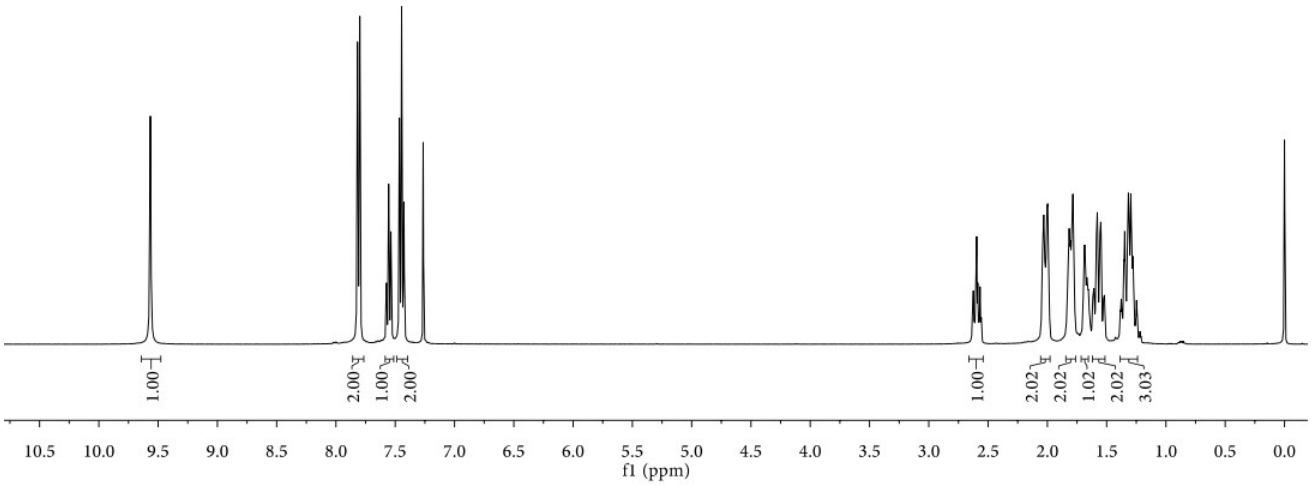

166.328
165.524
148.362
133.719
132.833
131.265
129.090
128.896
128.529
127.540
113.152
-0.000
f1 (ppm)

**3s**

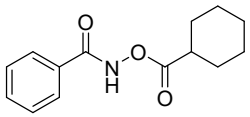

9.565
7.819
7.799
7.574
7.556
7.537
7.465
7.446
7.427
7.264
2.633
2.624
2.615
2.605
2.596
2.587
2.577
2.568
2.559
2.030
1.997
1.816
1.785
1.579
1.549
1.317
1.296
0.000



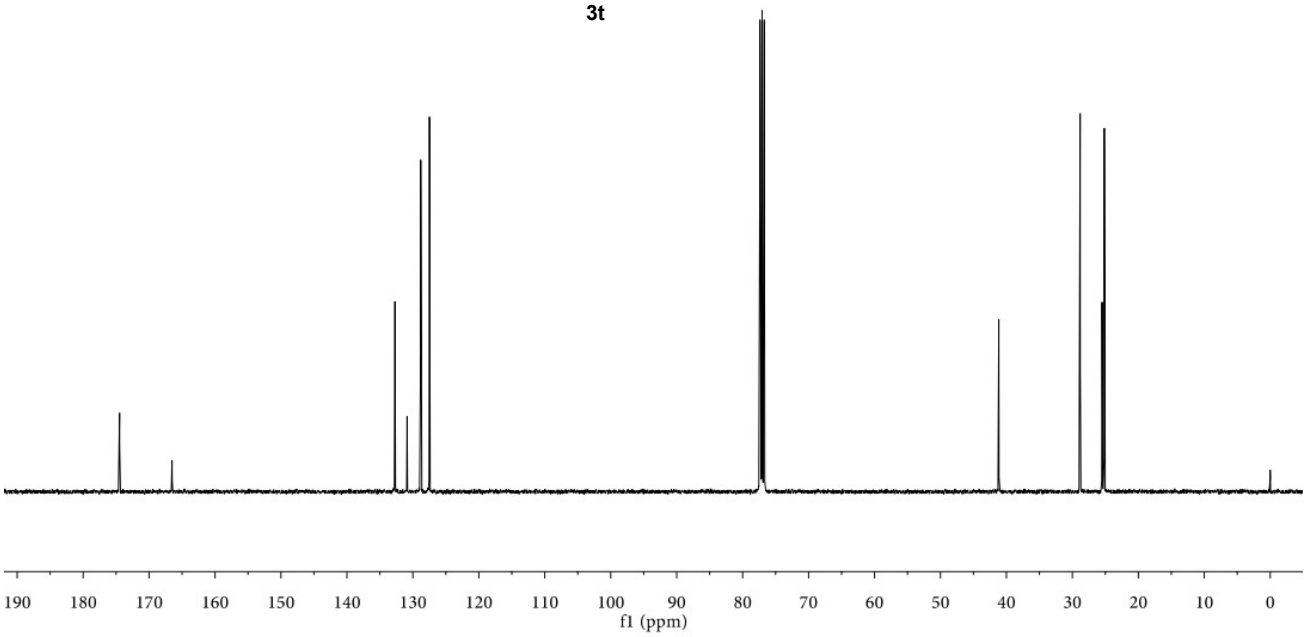
**3t**

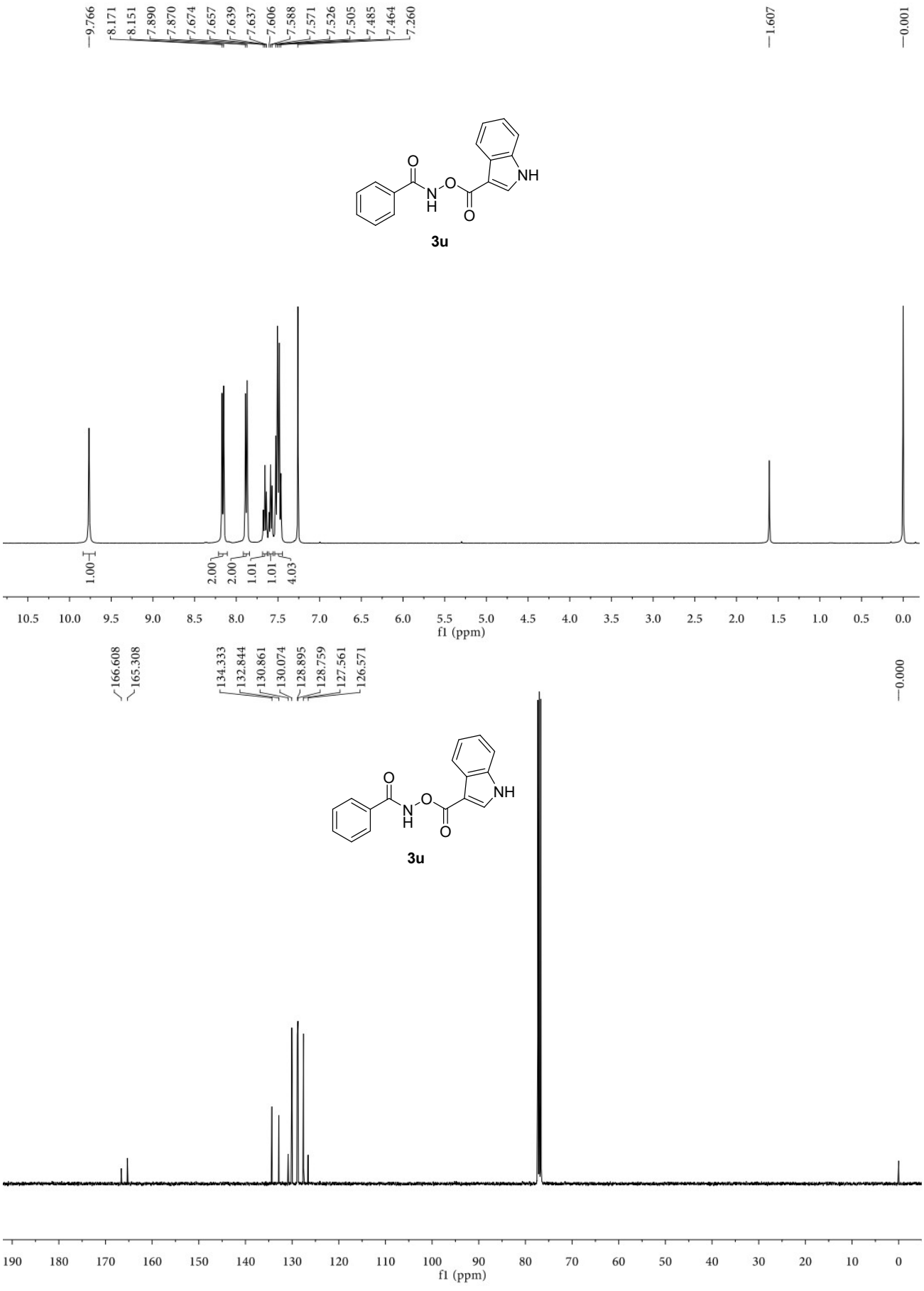

1.00
2.00
1.00
2.00
1.00
2.02
2.02
1.02
2.02
3.03
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

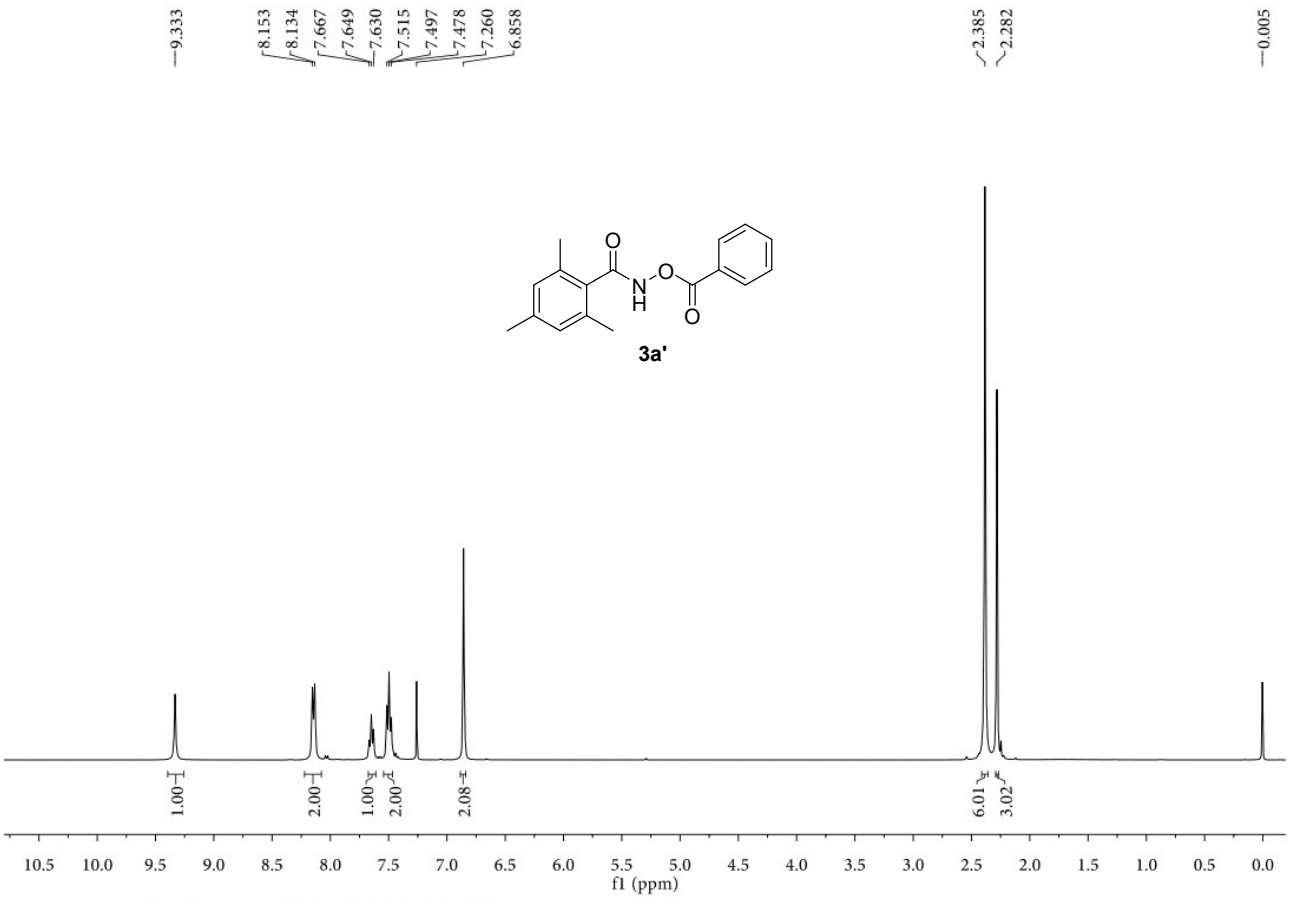

174.471
166.522
132.715
130.873
128.811
127.480
41.157
28.820
25.501
25.159
0.000

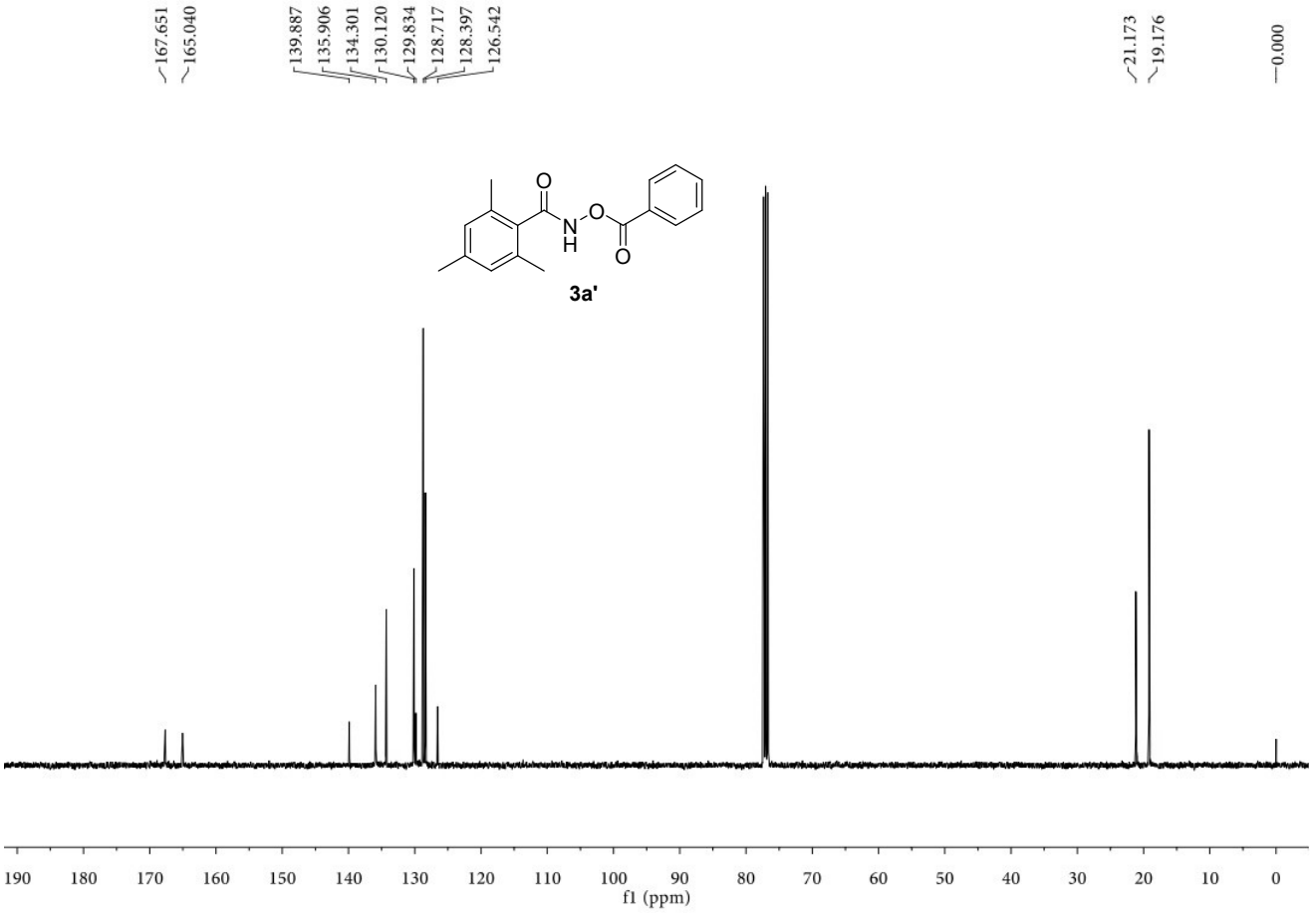


**3t**


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


9.766
8.171
8.151
7.890
7.870
7.674
7.657
7.639
7.637
7.606
7.588
7.571
7.526
7.505
7.485
7.464
7.260
1.607
0.001
NH
O
N
H
O
O
3u
1.00
2.00
2.00
1.01
1.01
4.03
f1 (ppm)
166.608
165.308
134.333
132.844
130.861
130.074
128.895
128.759
127.561
126.571
0.000
NH
O
N
H
O
O
3u
f1 (ppm)


9.333
8.153
8.134
7.667
7.649
7.630
7.515
7.497
7.478
7.260
6.858
2.385
2.282
0.005
3a'
1.00
2.00
1.00
2.00
2.08
6.01
3.02
f1 (ppm)


167.651
165.040
139.887
135.906
134.301
130.120
129.834
128.717
128.397
126.542
21.173
19.176
0.000
3a'
f1 (ppm)