

Supporting Information

Microwave-assisted direct oxidative synthesis of α -ketoamides from aryl methyl ketones and amines by a water soluble Cu(I)-complex

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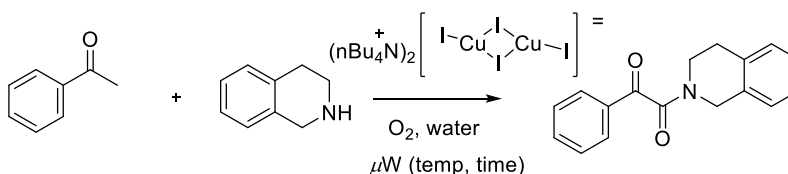
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Optimization studies for the synthesis of α -ketoamides under Microwave Irradiation

In order to rapidly access α -ketoamides, we explored microwave technology for the acceleration of the reaction of acetophenone (**1a**) with 1,2,3,4-tetrahydroquinoline (**2a**). By using Bis[(tetrabutylammonium) di- μ -iodo-diiododicuprate(I) complex in a microwave vial purged with oxygen (**Figure S1**), at 50 °C after 20 min, a conversion of 83% was observed, (Table 2, entry 3). Interestingly, at 60 °C, a conversion of 83% was observed after only 10 min. At this same temperature and duration, no improvement was obtained by switching to THF as the solvent (entry 10), but when the reaction was carried out in solvent-free mode, **3a** was isolated in a better yield (80%, entry 3). However, beyond heating at 60 °C towards 100 °C and 120 °C, the yields decreased exponentially and at 200 °C, no product was isolated. Compared to thermal reactions (50 °C), microwave irradiation (60 °C) gave better yield of **3a** (78% and 83% respectively) and significantly reduced the reaction times from 12h to 10 min. Thus, the microwave conditions were employed subsequently for all further reactions.

Table S1. Optimization studies under Microwave Irradiation^a



Entry	μ W Temp (°C)	Time (min)	Yield ^b
1	50	5	73
2	50	10	79
3	50	20	83
4	60	5	75
5	60	10	83
6	60	20	81
7	100	5	45
8	120	5	15
9	200	5	0
10	60	10	69 ^c
11	60	10	80 ^d

^aReaction conditions: **1a** (1 mmol), **2a** (2 mmol), catalyst (20 mol%) and solvent (2 mL) in a microwave vial purged with oxygen, ^bIsolated yields. ^cTHF, ^dsolvent-free.

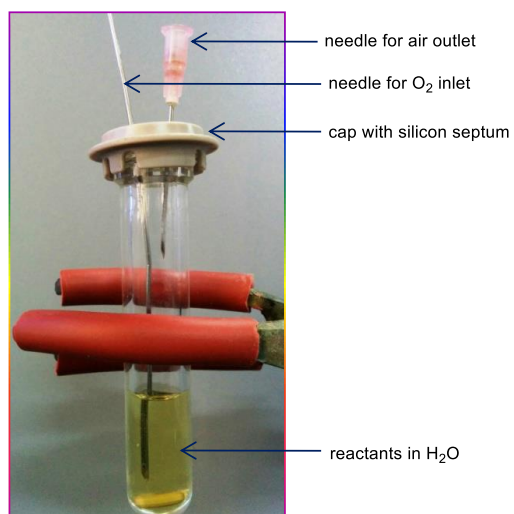
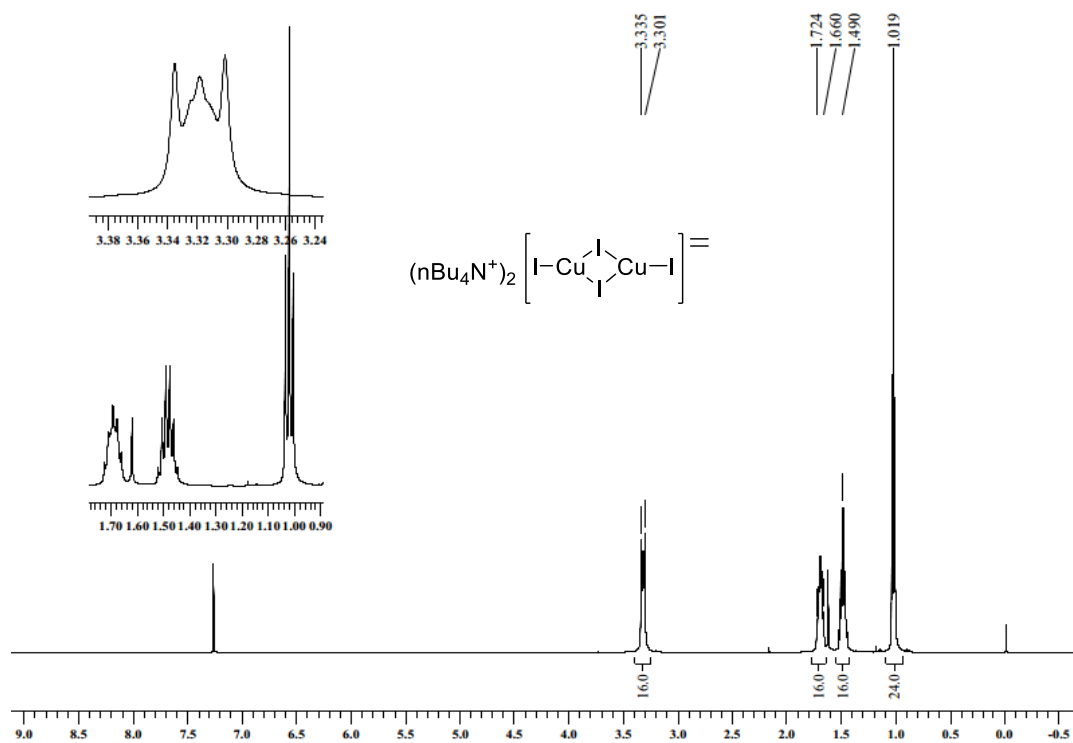
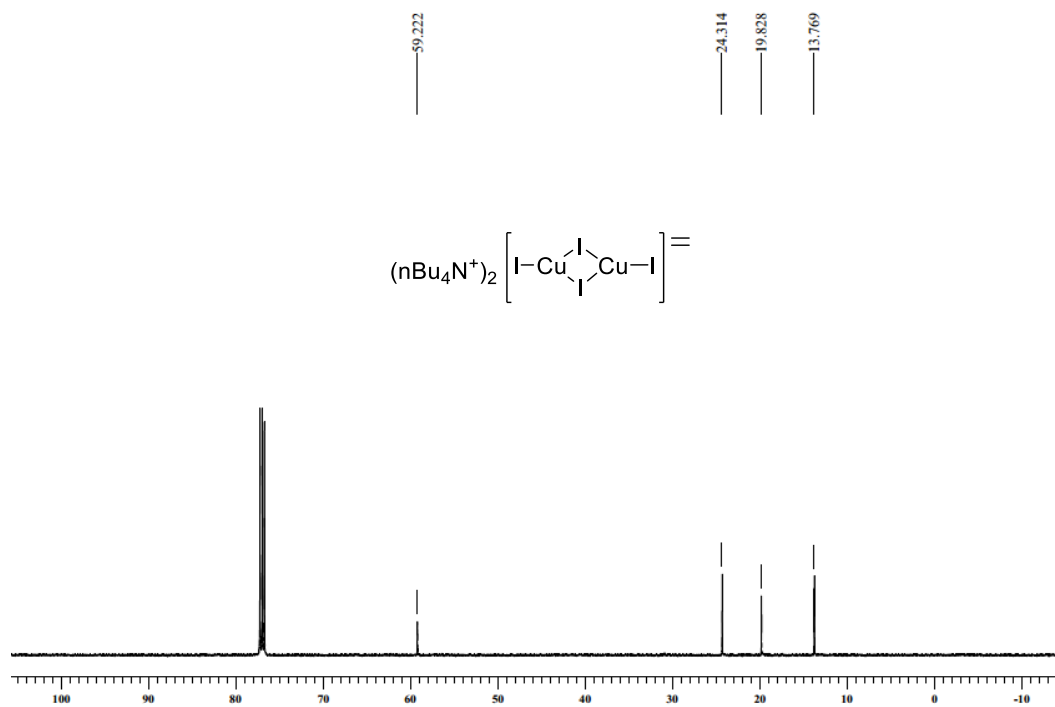


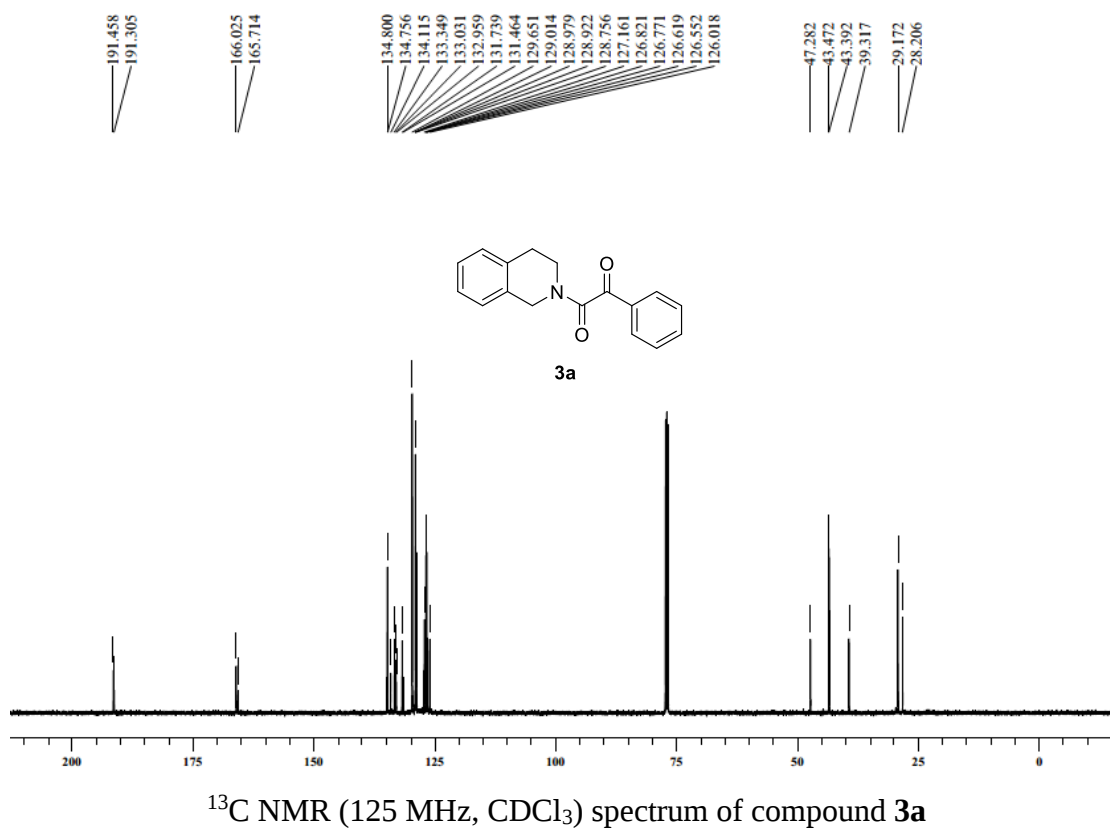
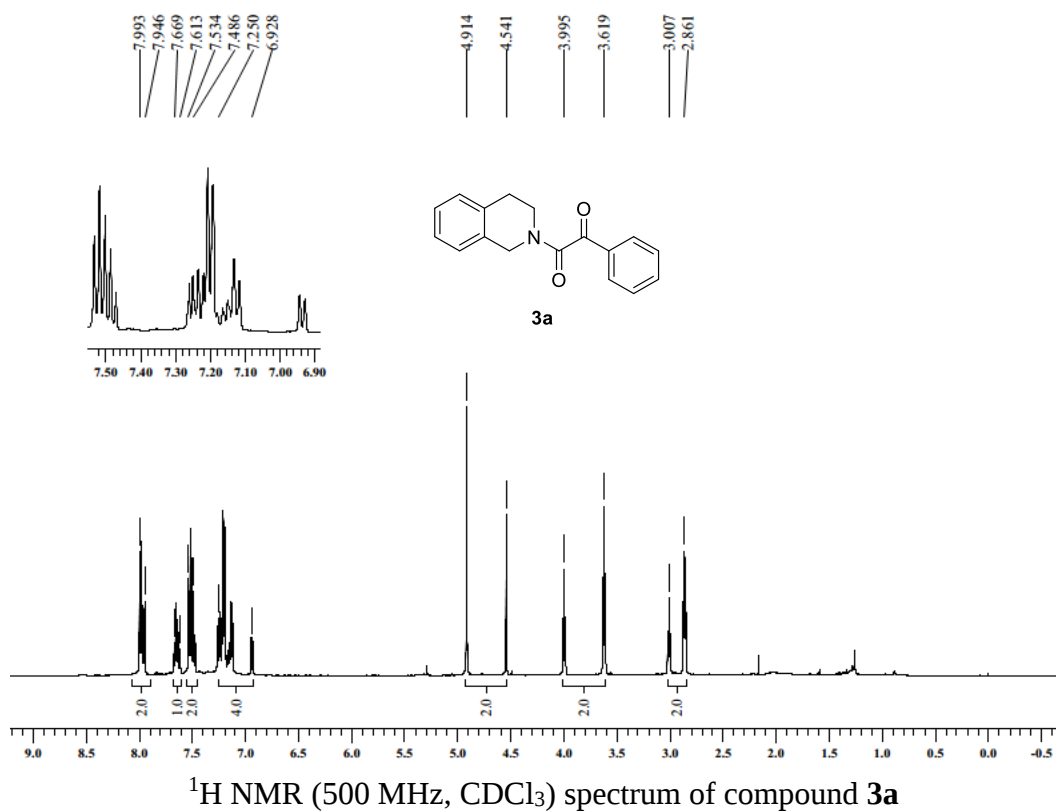
FIGURE S1. Capped monowave vial G10 (10 mL) with needles penetrating the silicon septum for creating oxygen atmosphere.

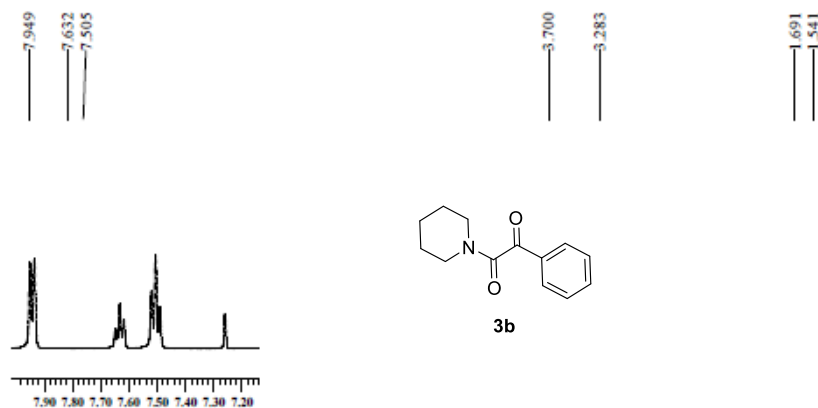


¹H NMR (500 MHz, CDCl₃) spectrum of $(n\text{Bu}_4\text{N}^+)_2(\text{Cu}_2\text{I}_4)^-$

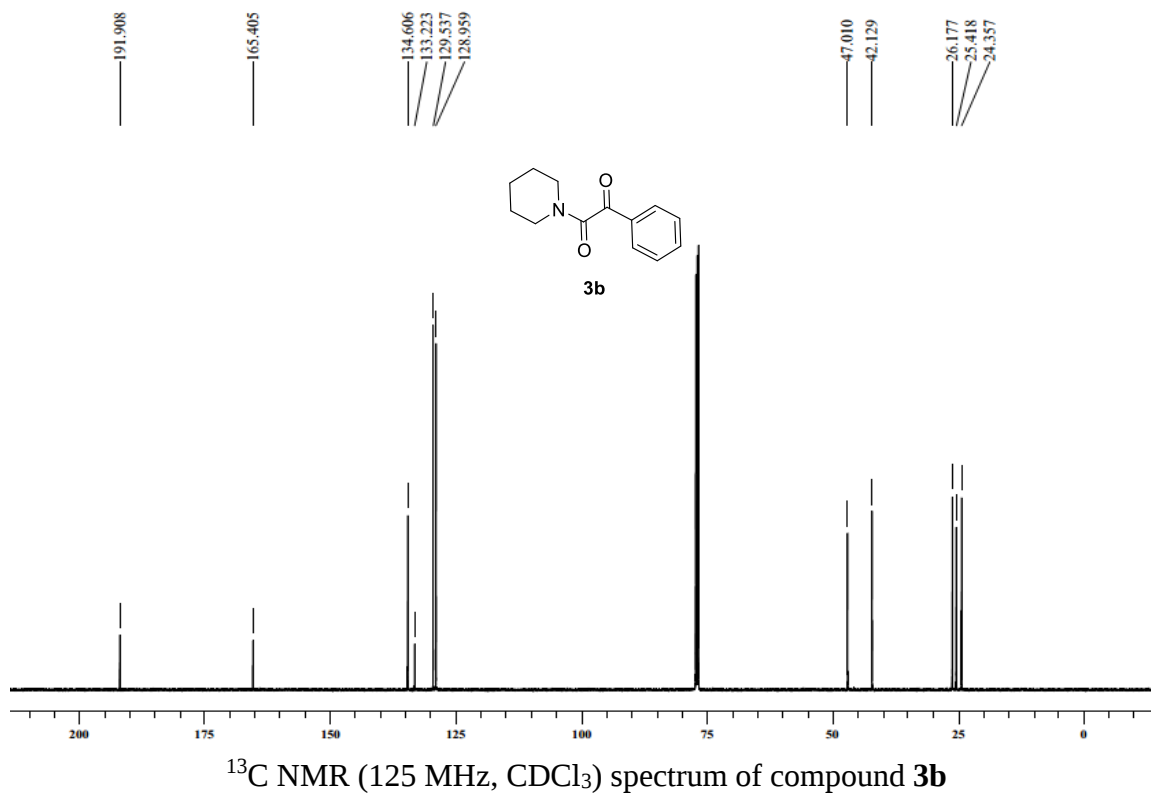


¹³C NMR (125 MHz, CDCl₃) spectrum of $(n\text{Bu}_4\text{N}^+)_2(\text{Cu}_2\text{I}_4)^-$

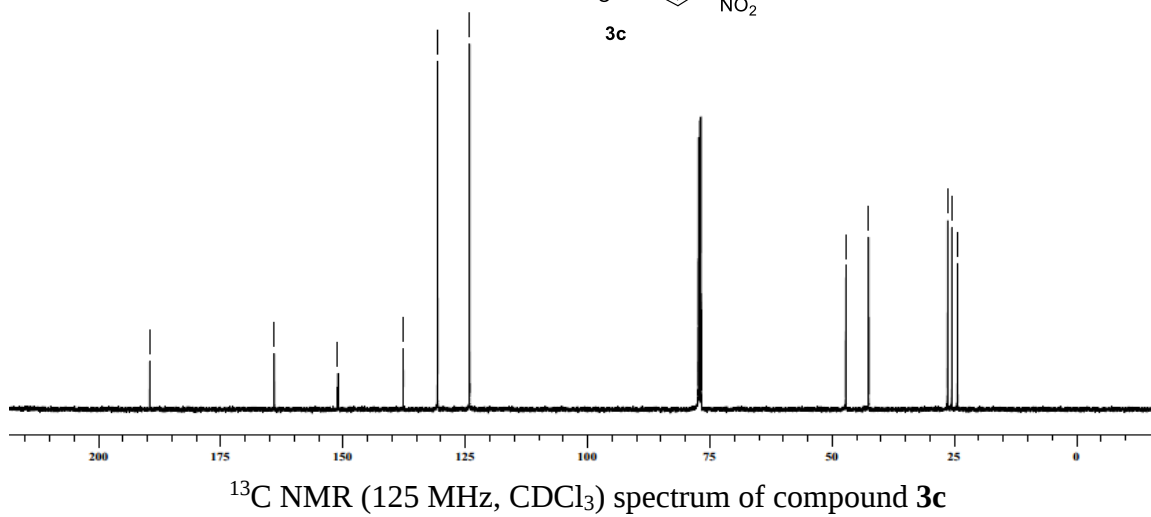
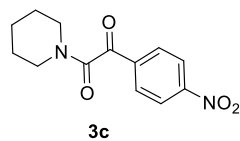
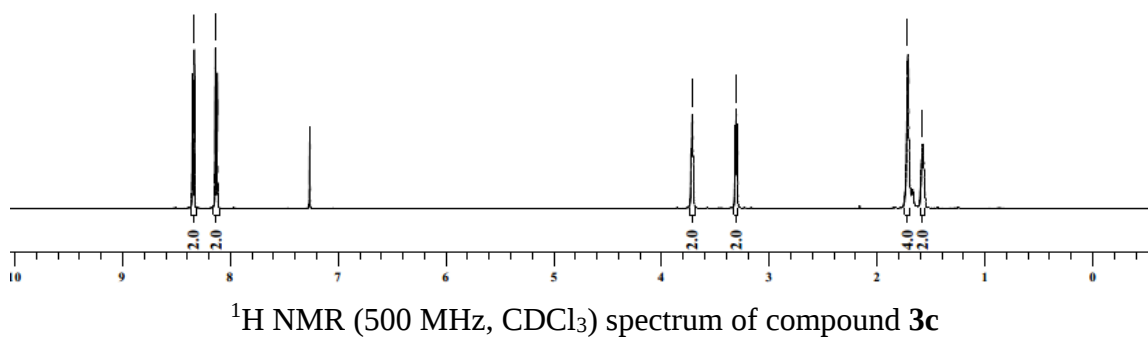
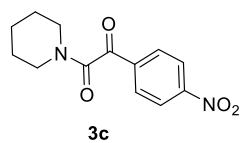


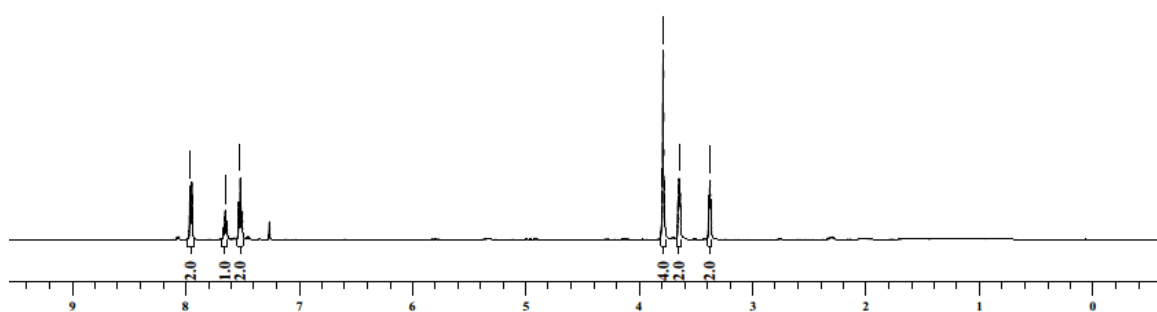
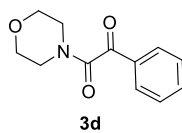
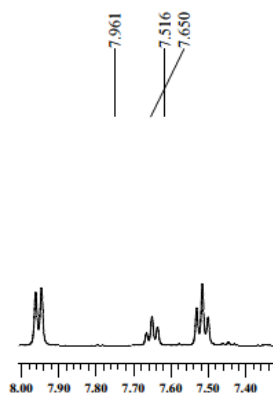


¹H NMR (500 MHz, CDCl₃) spectrum of compound **3b**

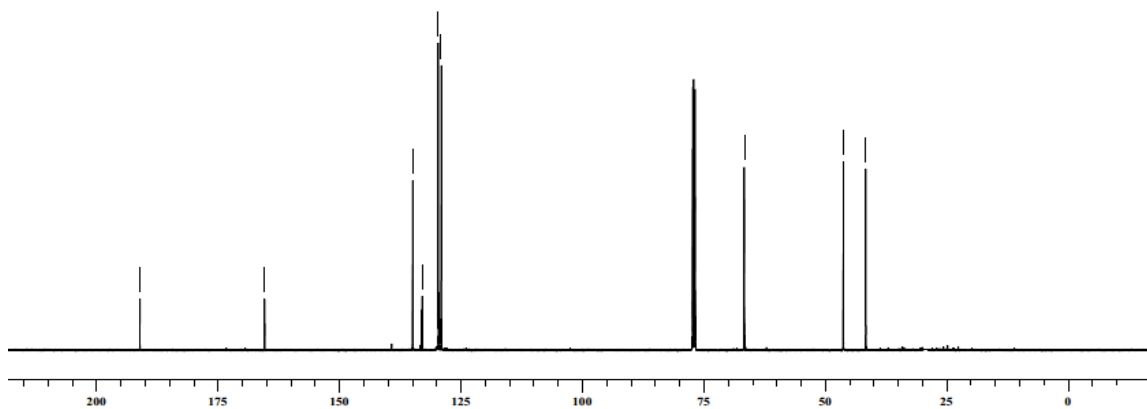
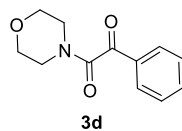
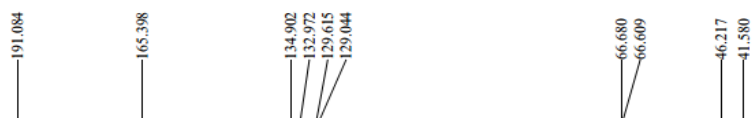


¹³C NMR (125 MHz, CDCl₃) spectrum of compound **3b**

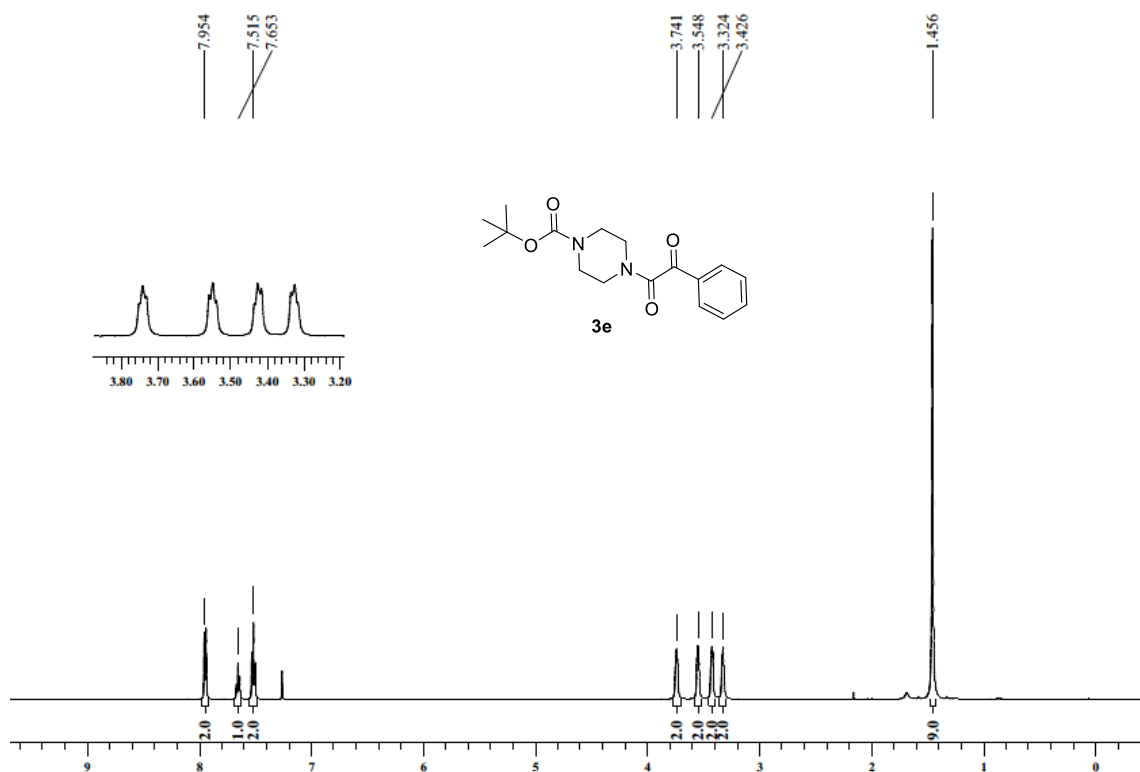




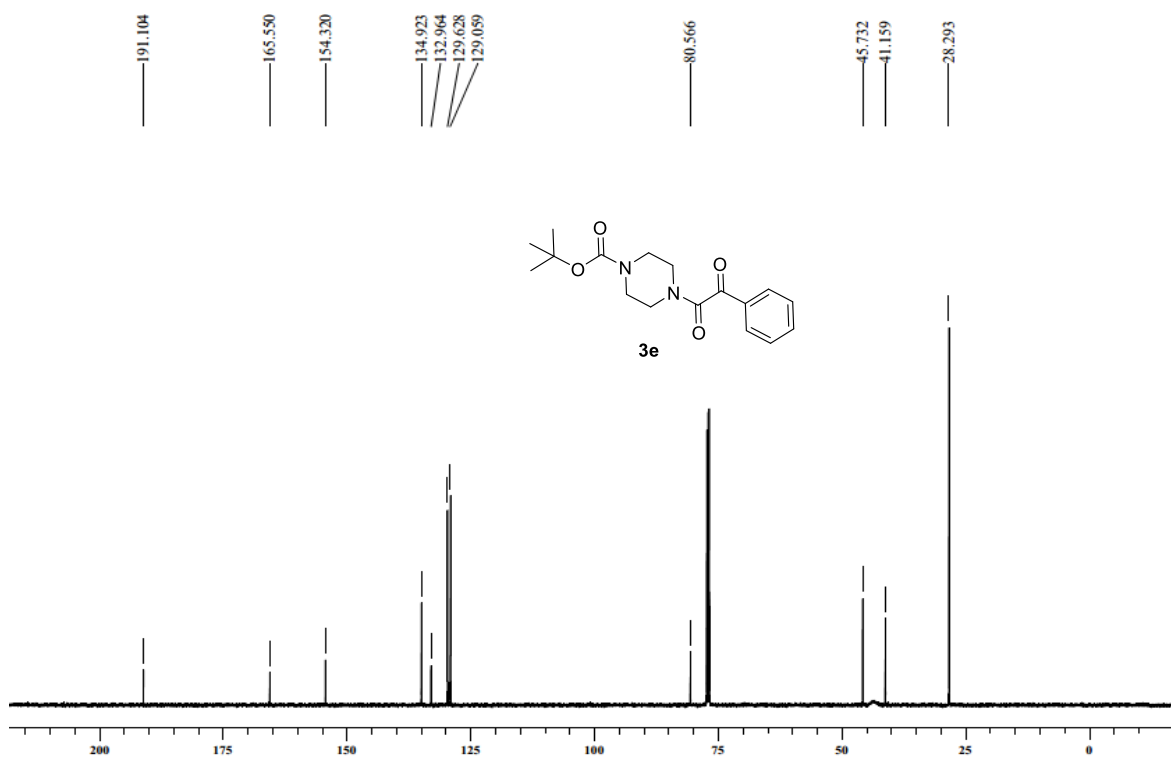
¹H NMR (500 MHz, CDCl₃) spectrum of compound **3d**



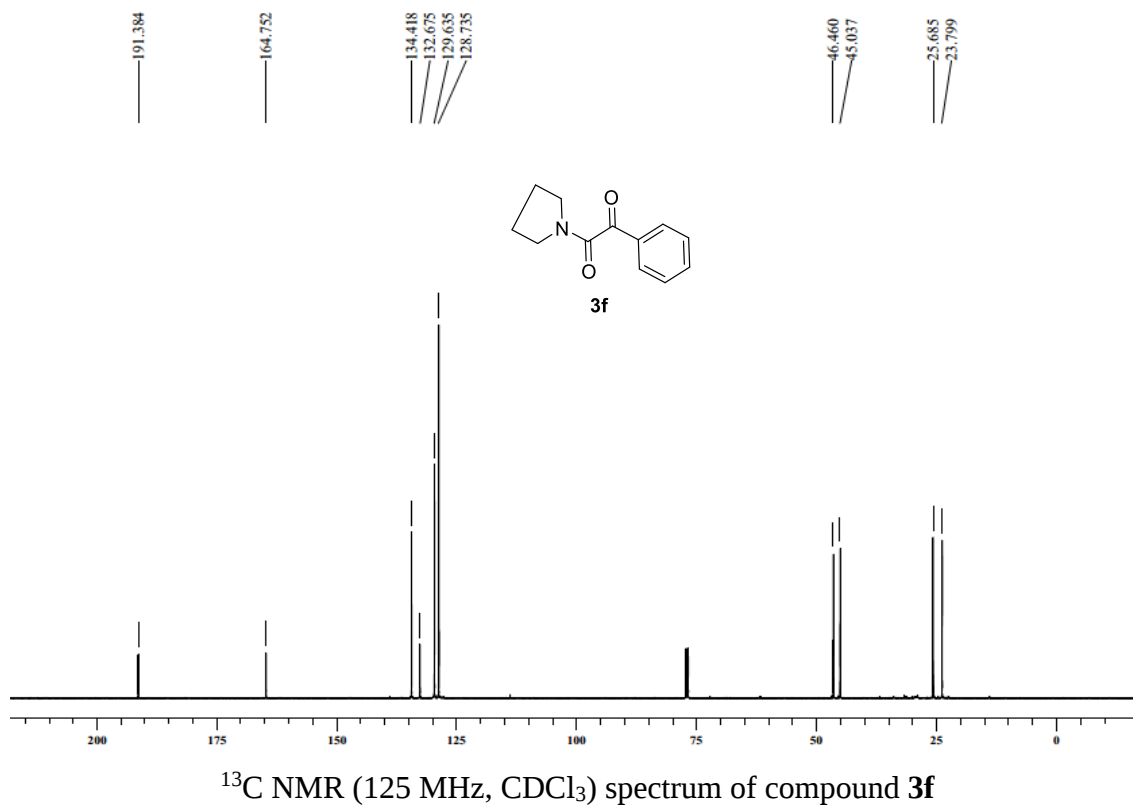
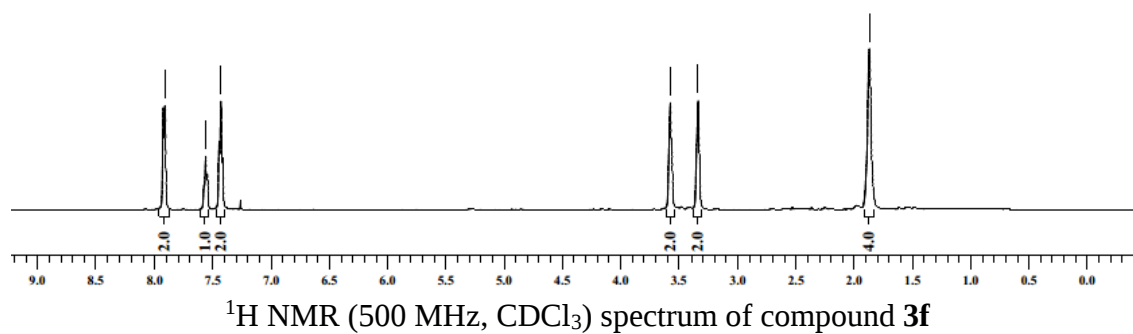
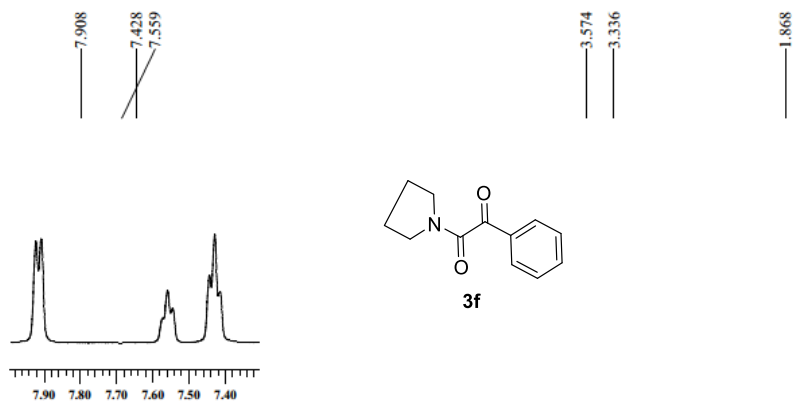
¹³C NMR (125 MHz, CDCl₃) spectrum of compound **3d**

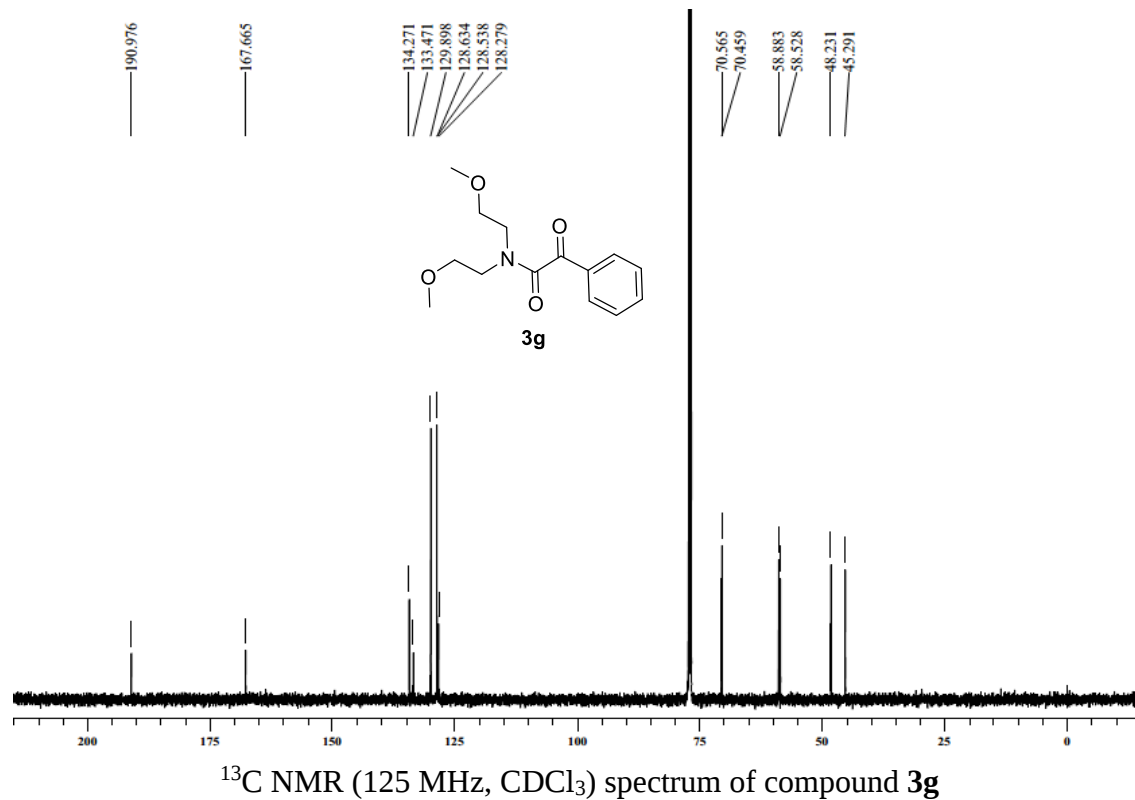
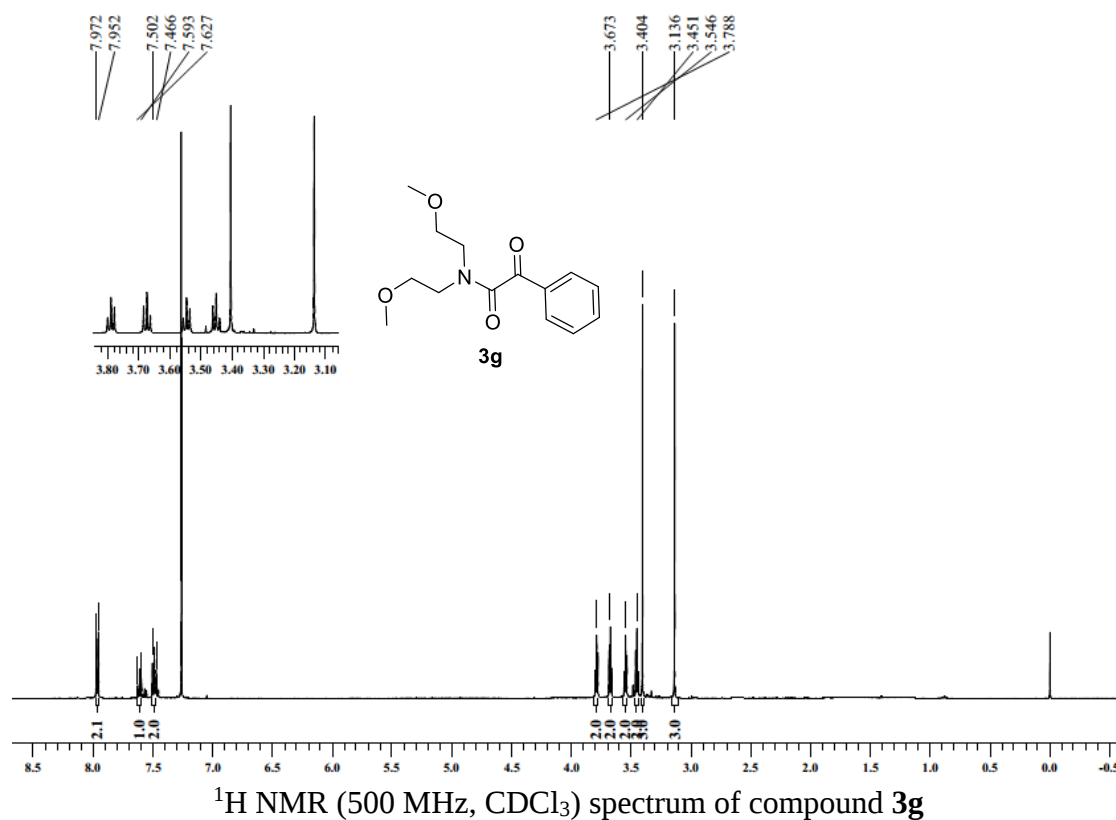


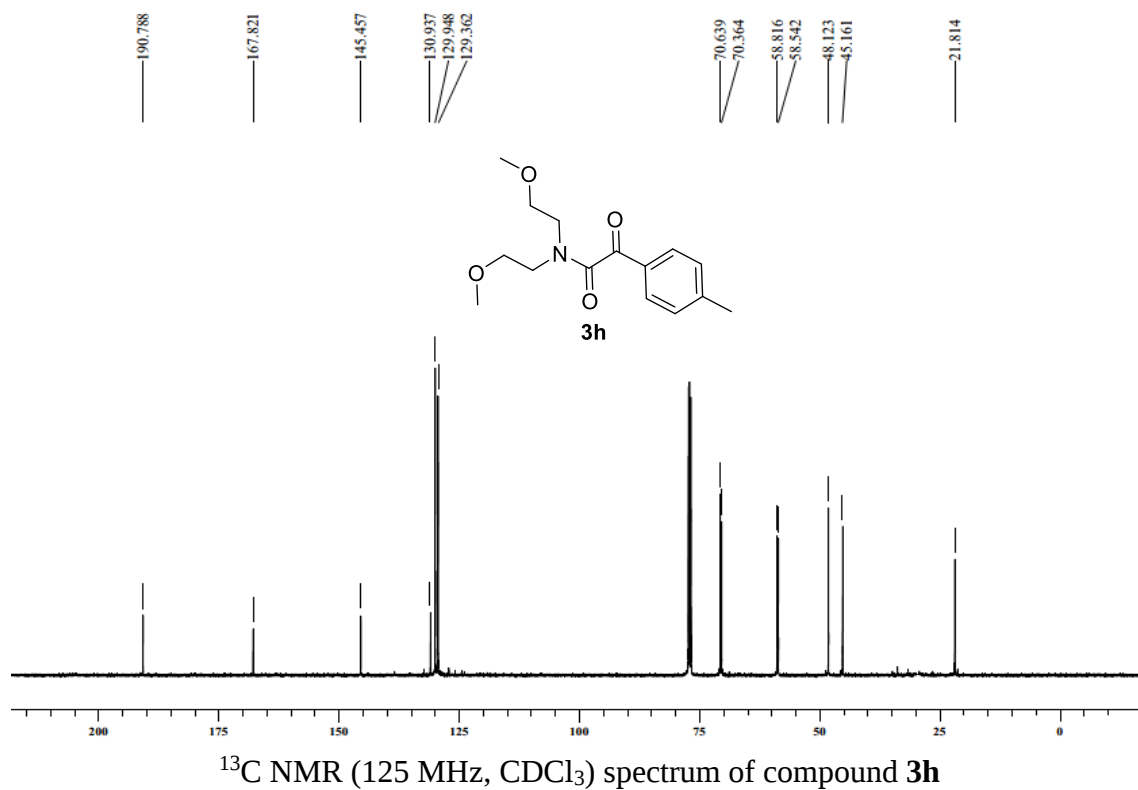
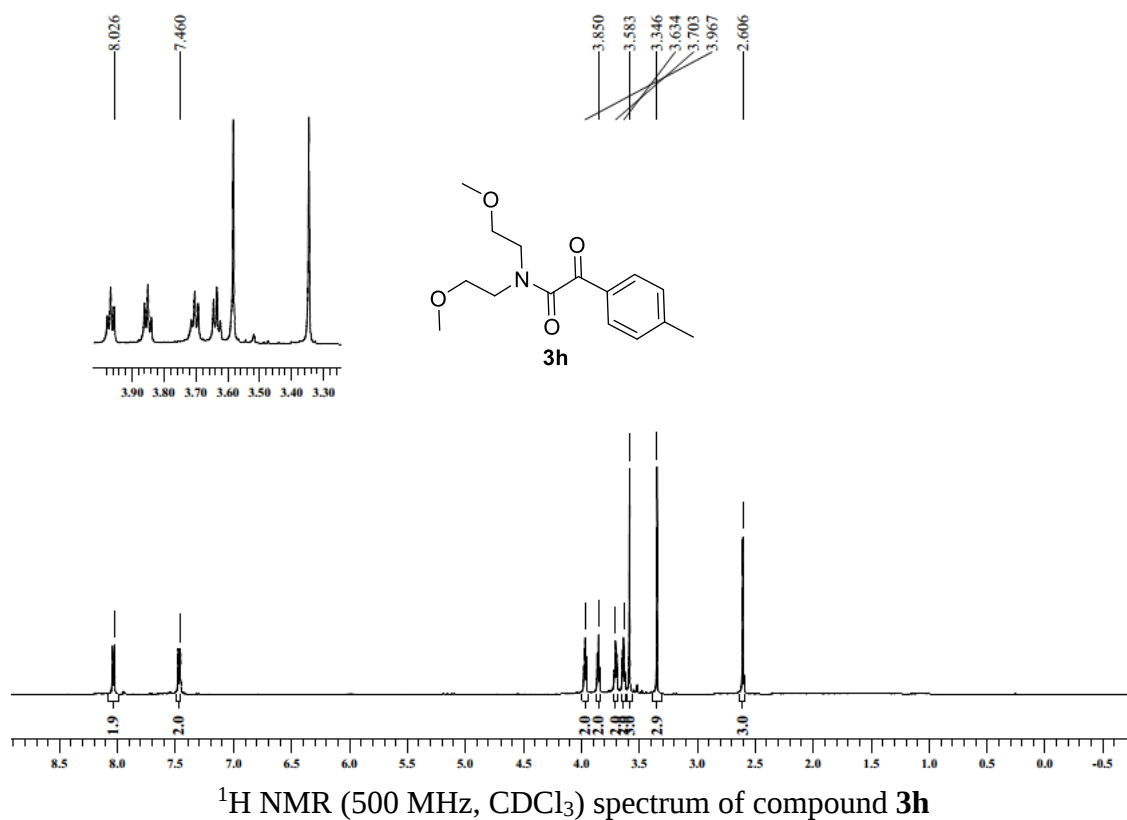
¹H NMR (500 MHz, CDCl₃) spectrum of compound **3e**

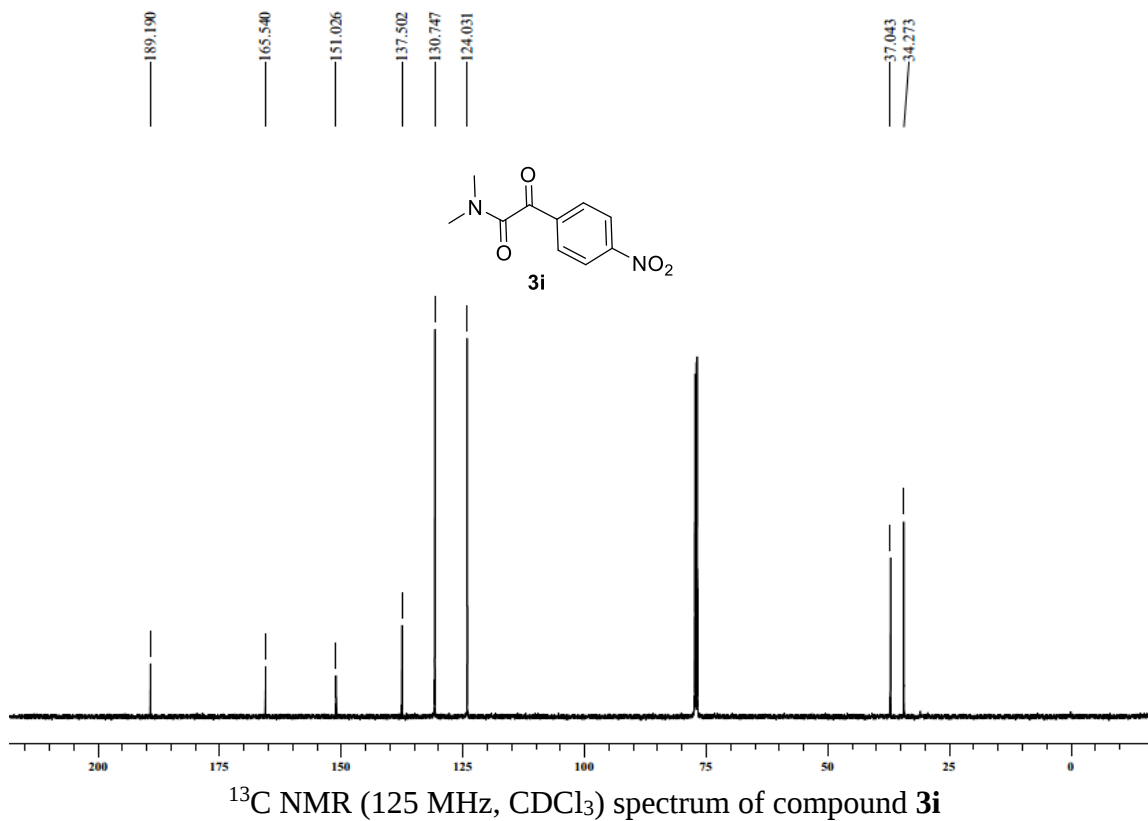
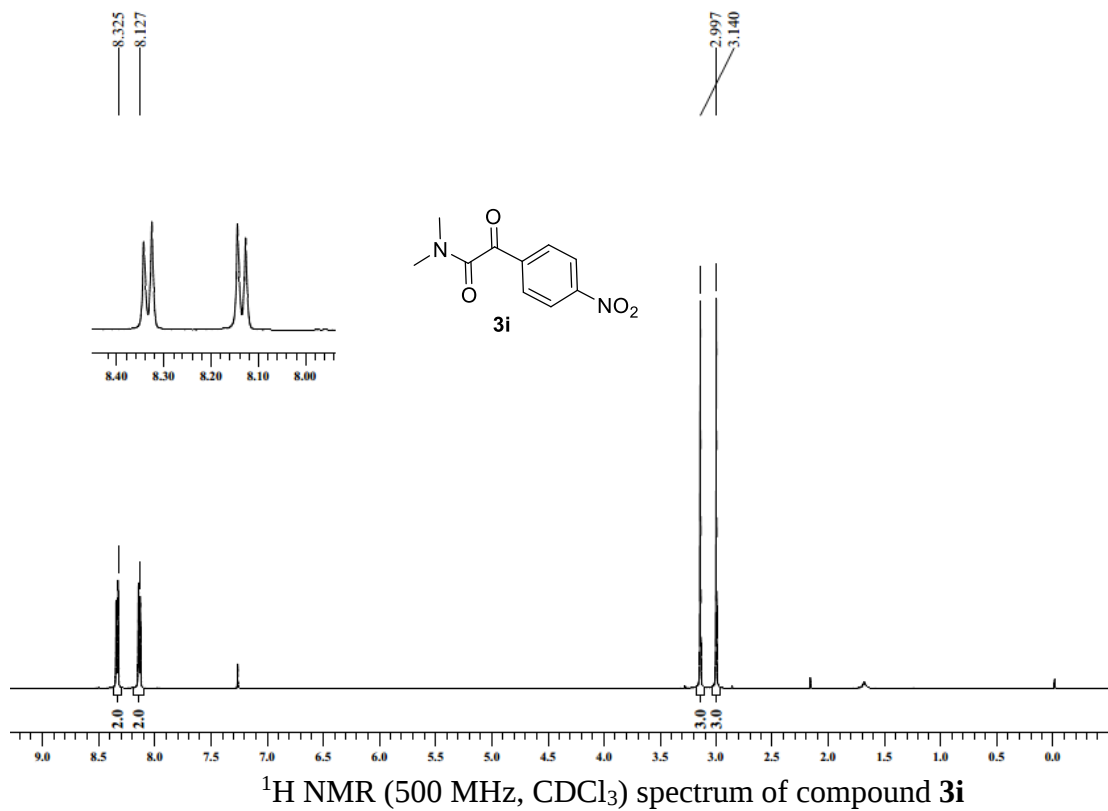


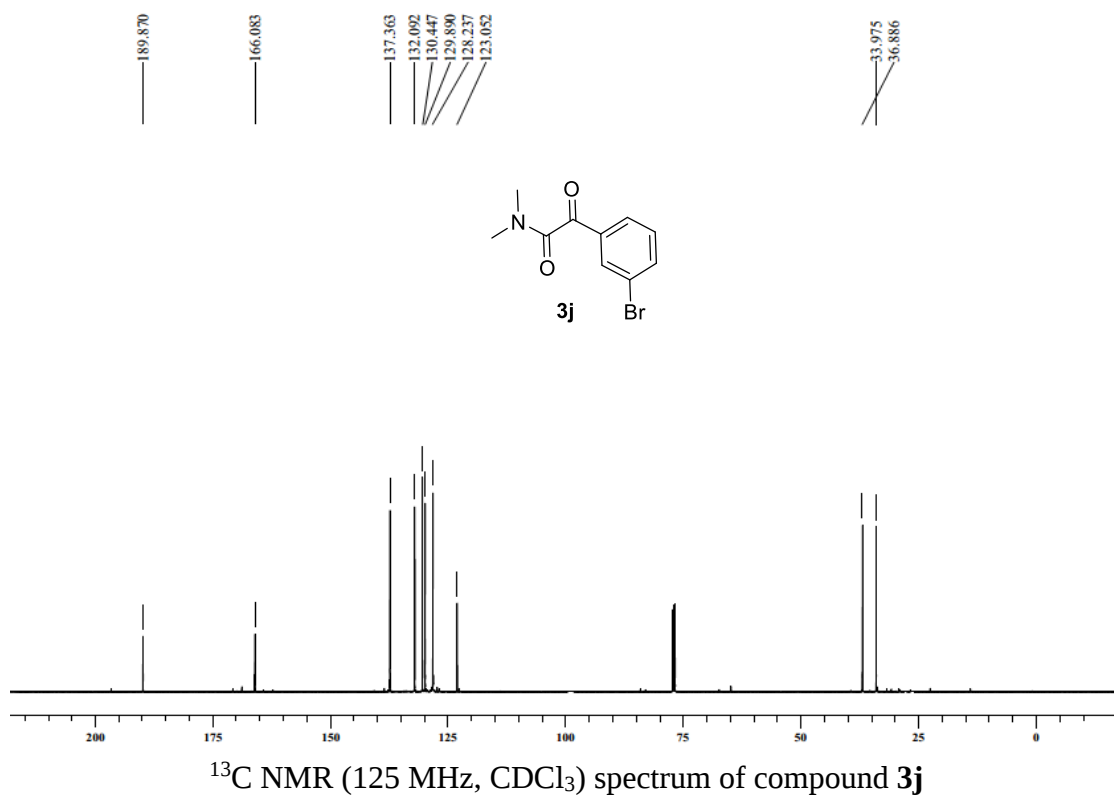
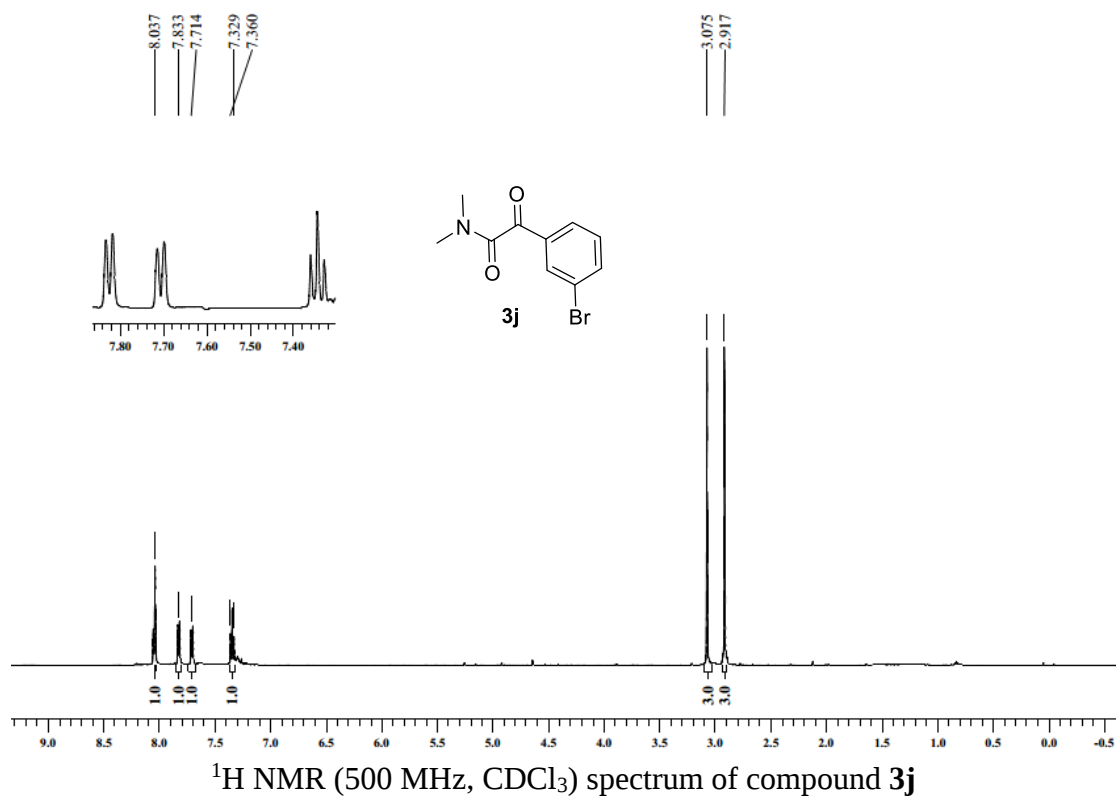
¹³C NMR (125 MHz, CDCl₃) spectrum of compound **3e**

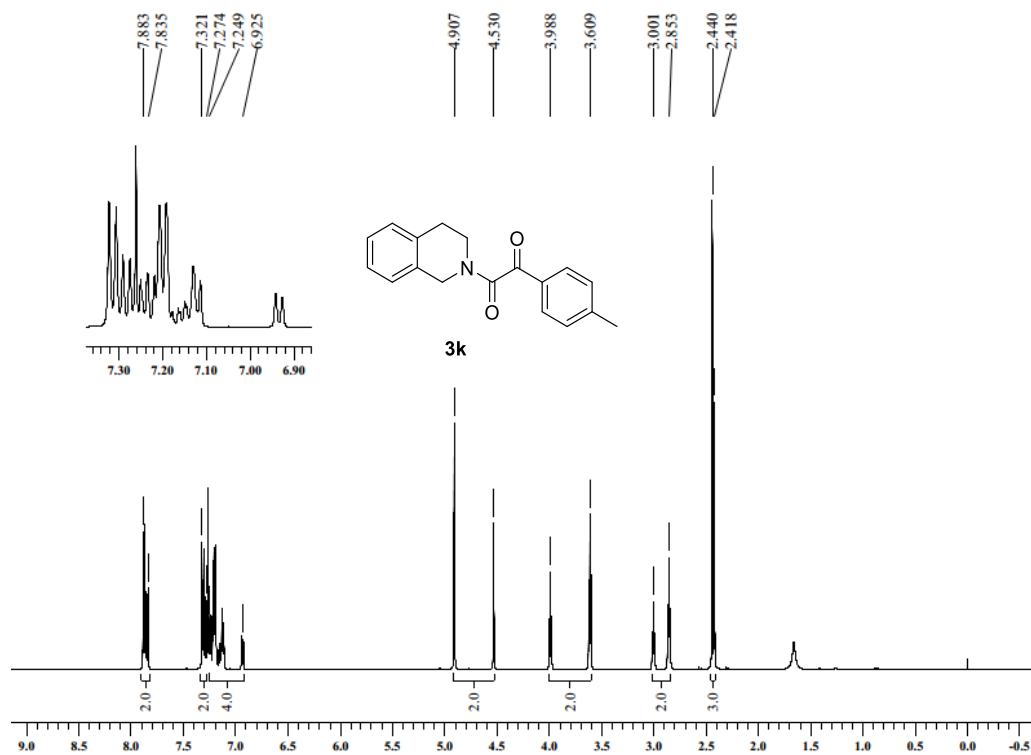




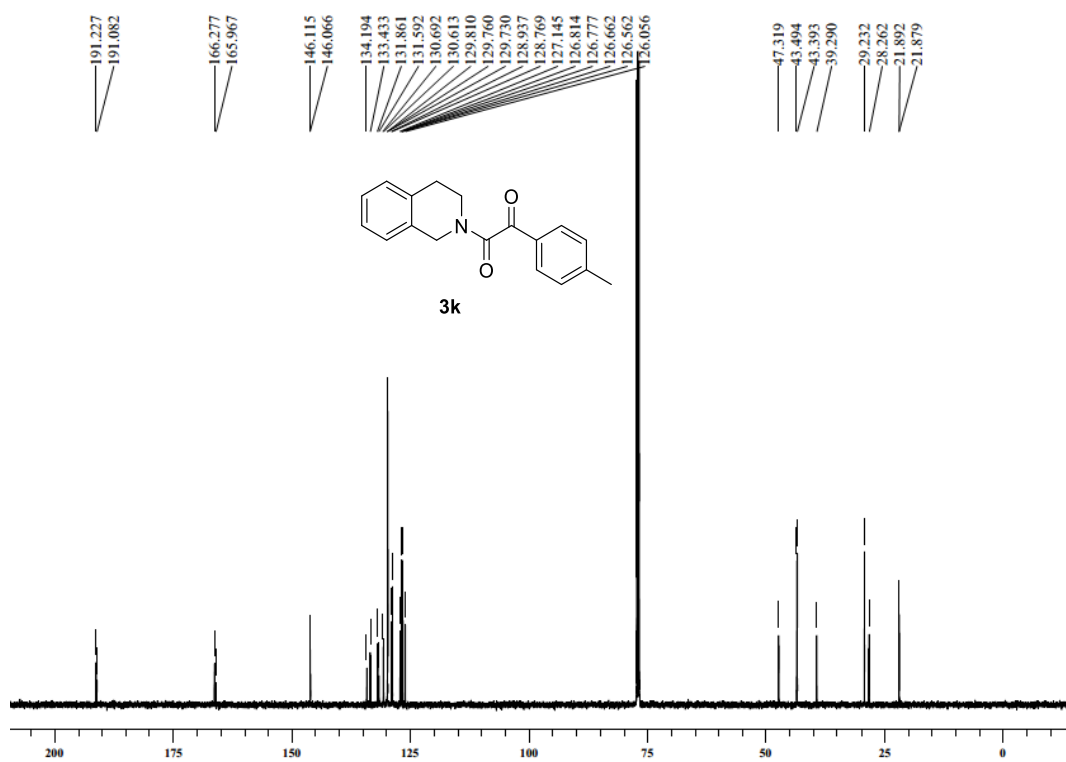




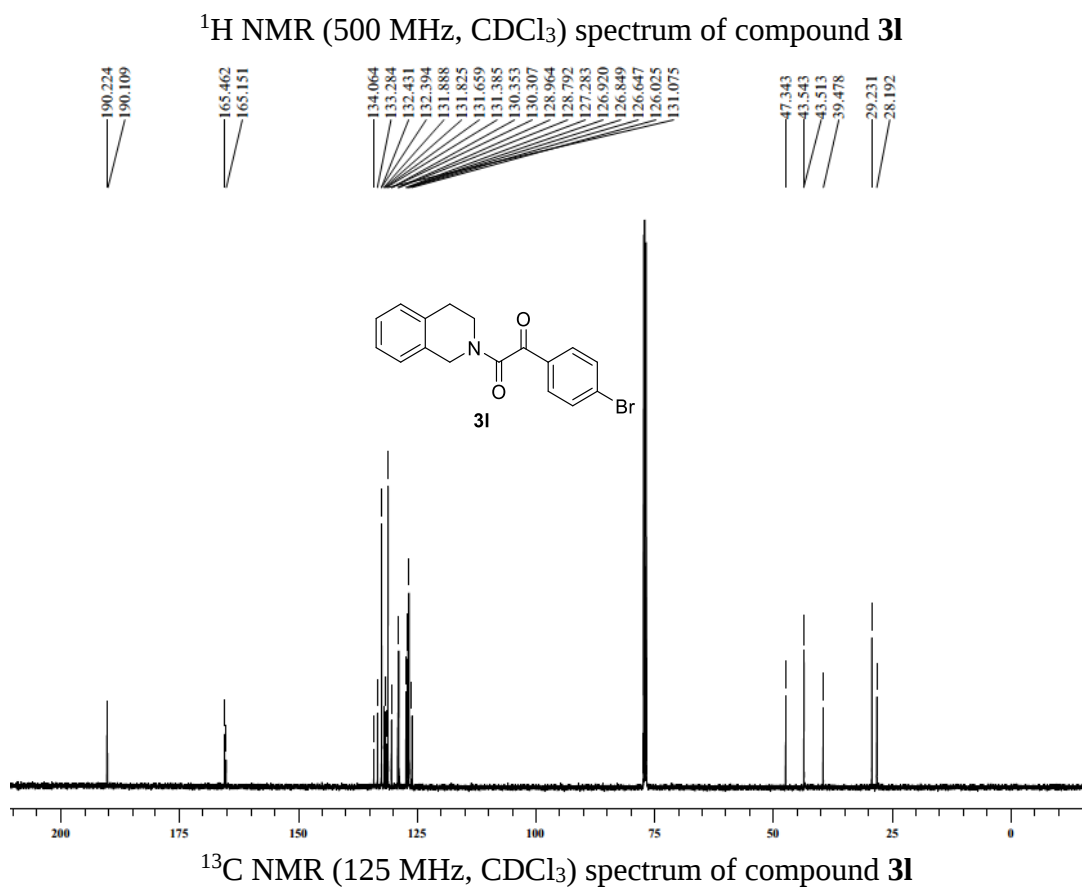
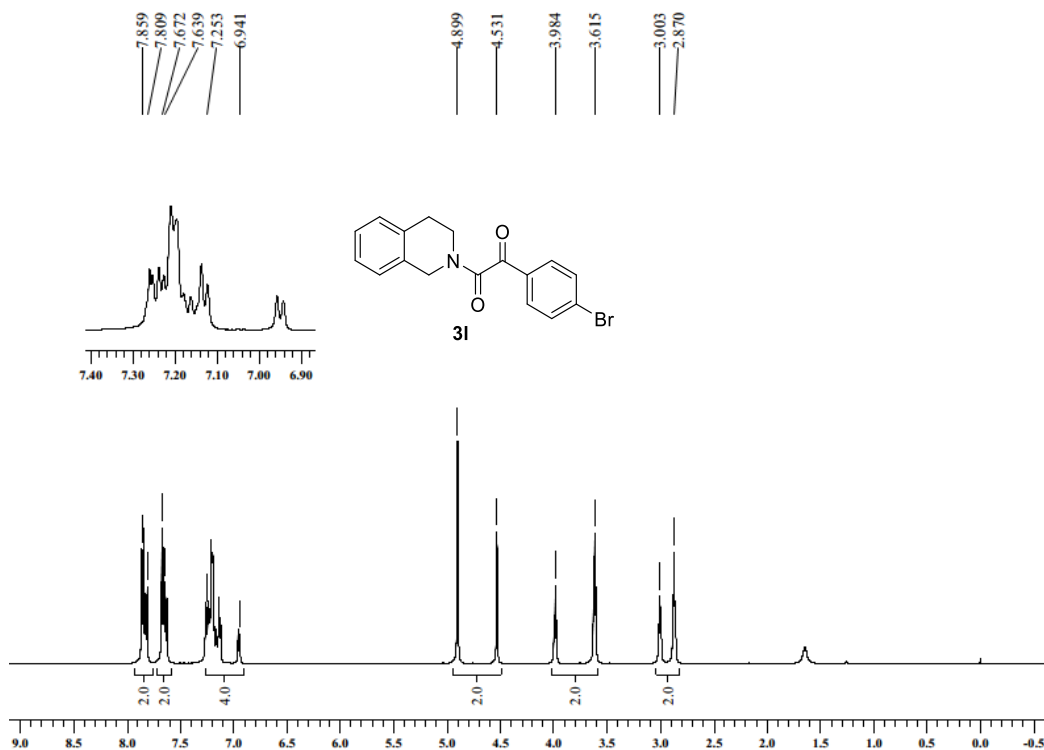


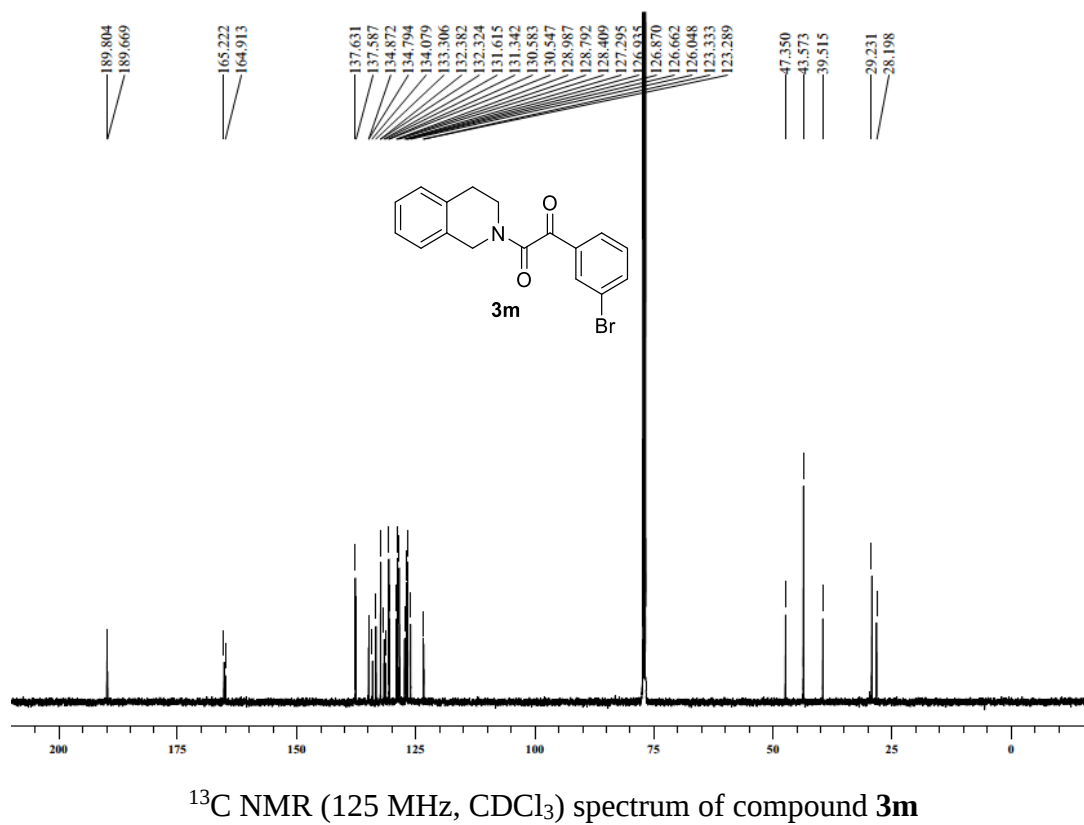
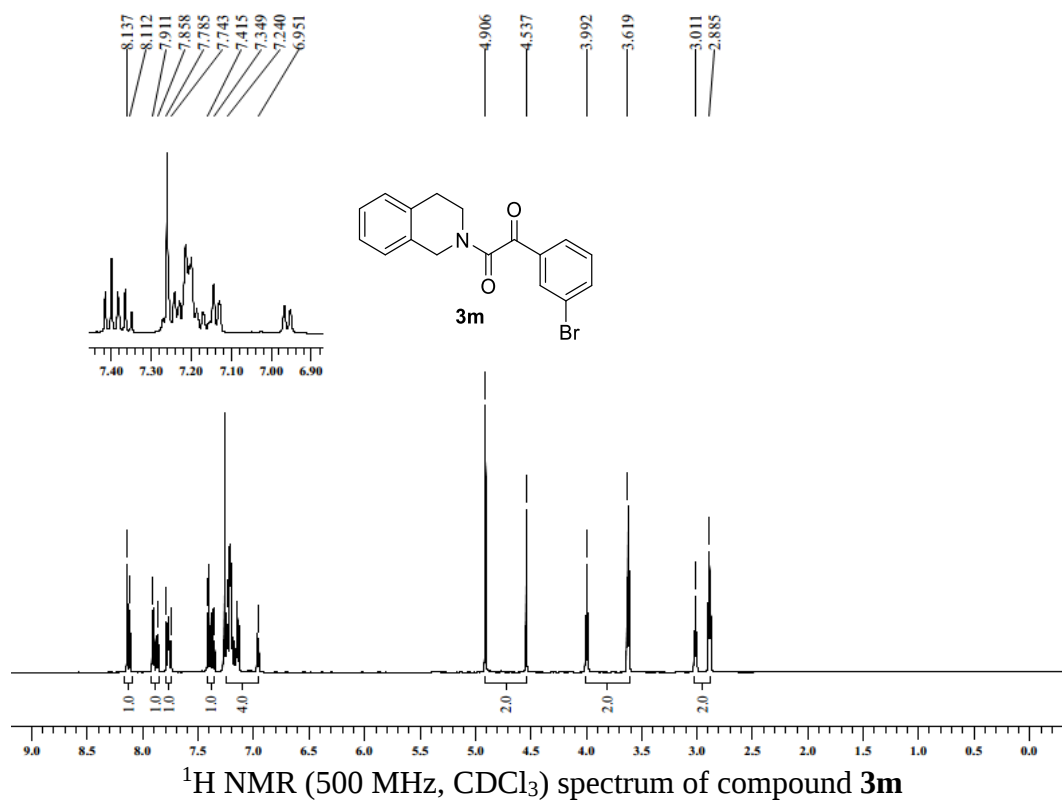


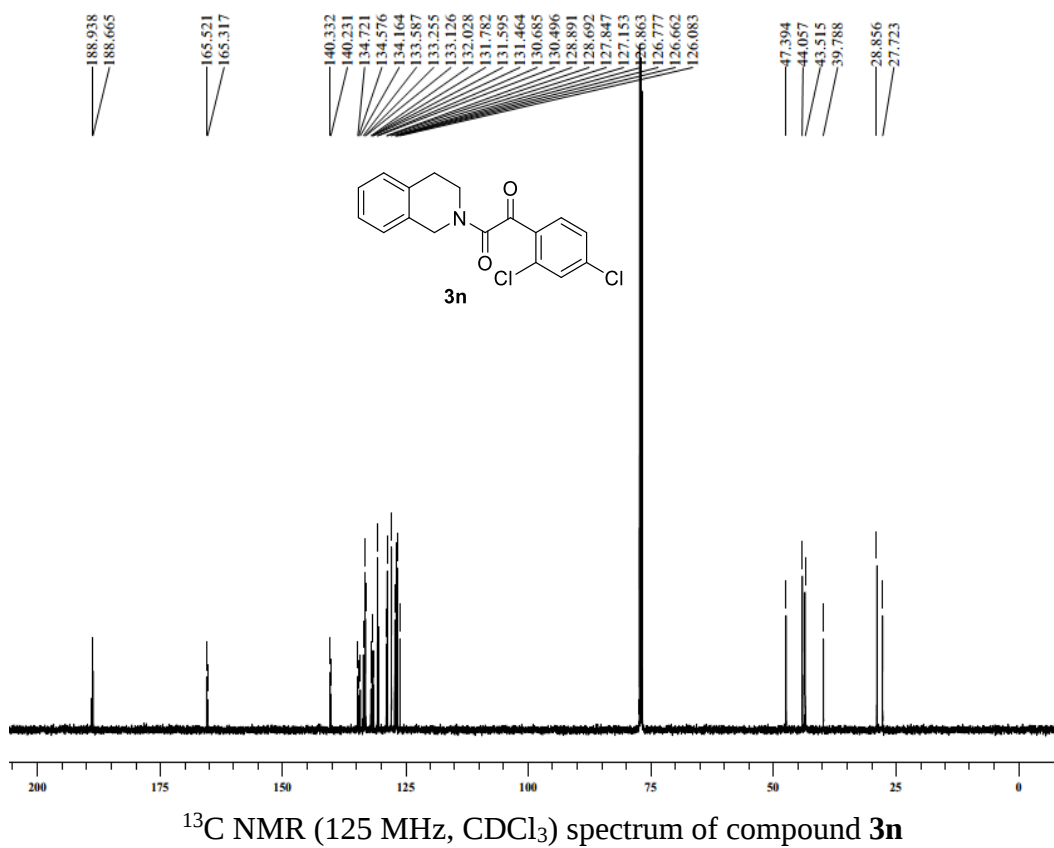
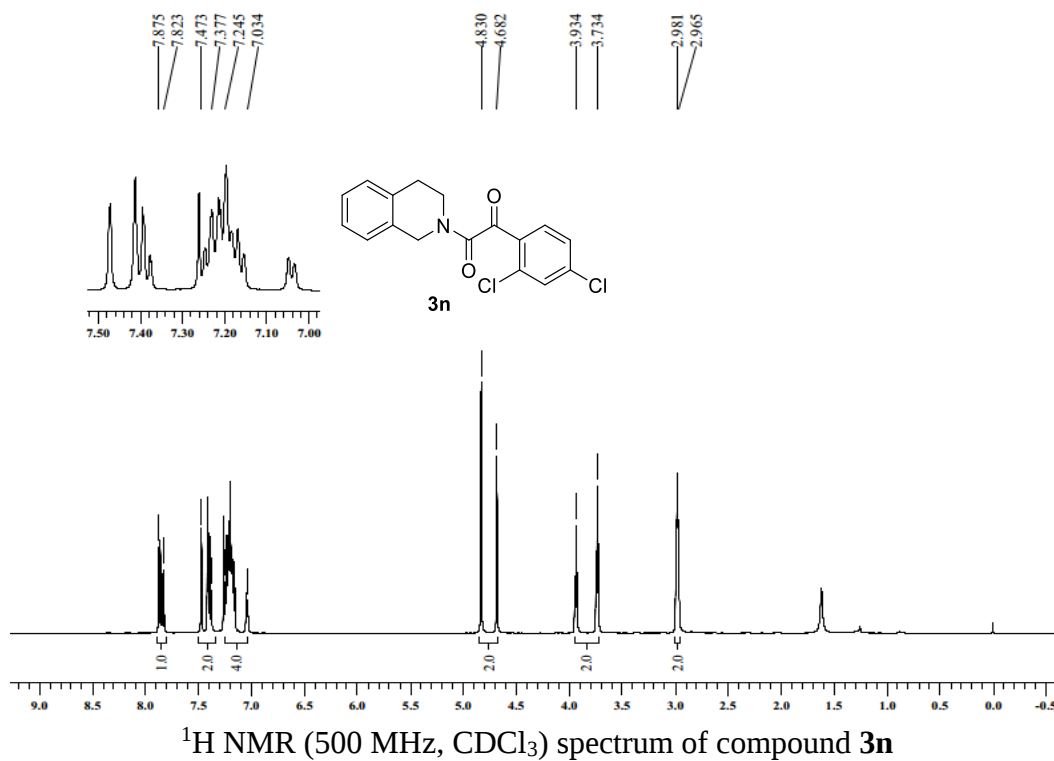
¹H NMR (500 MHz, CDCl₃) spectrum of compound **3k**

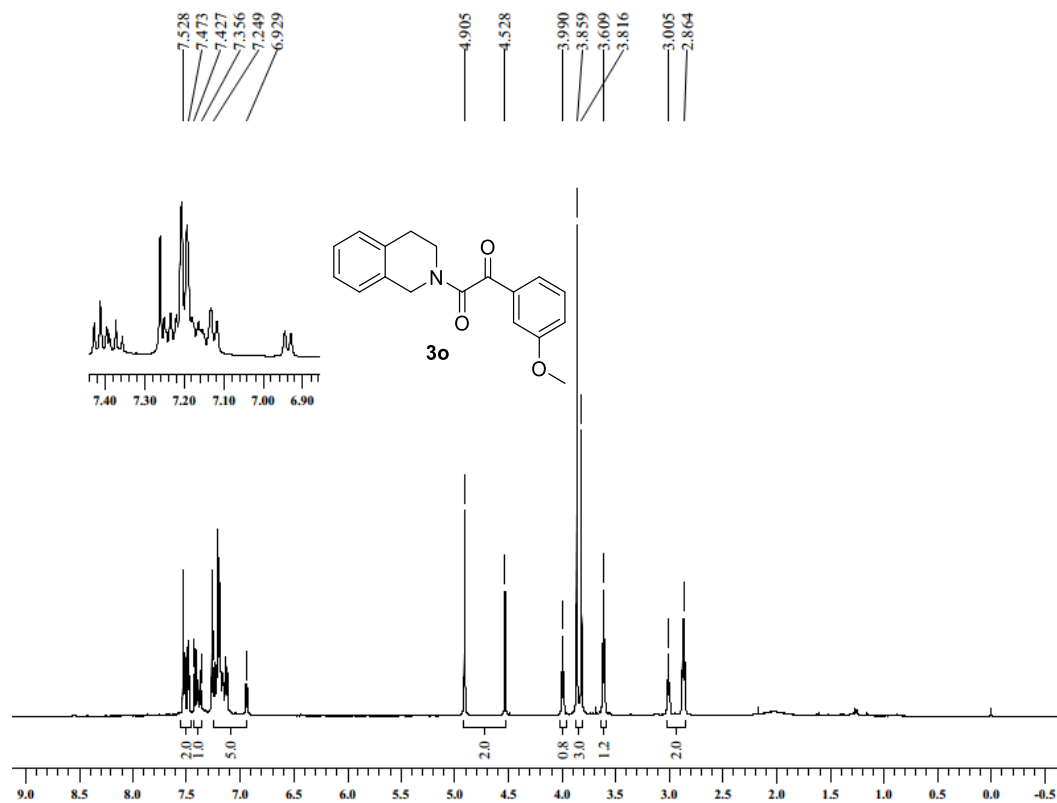


¹³C NMR (125 MHz, CDCl₃) spectrum of compound **3k**

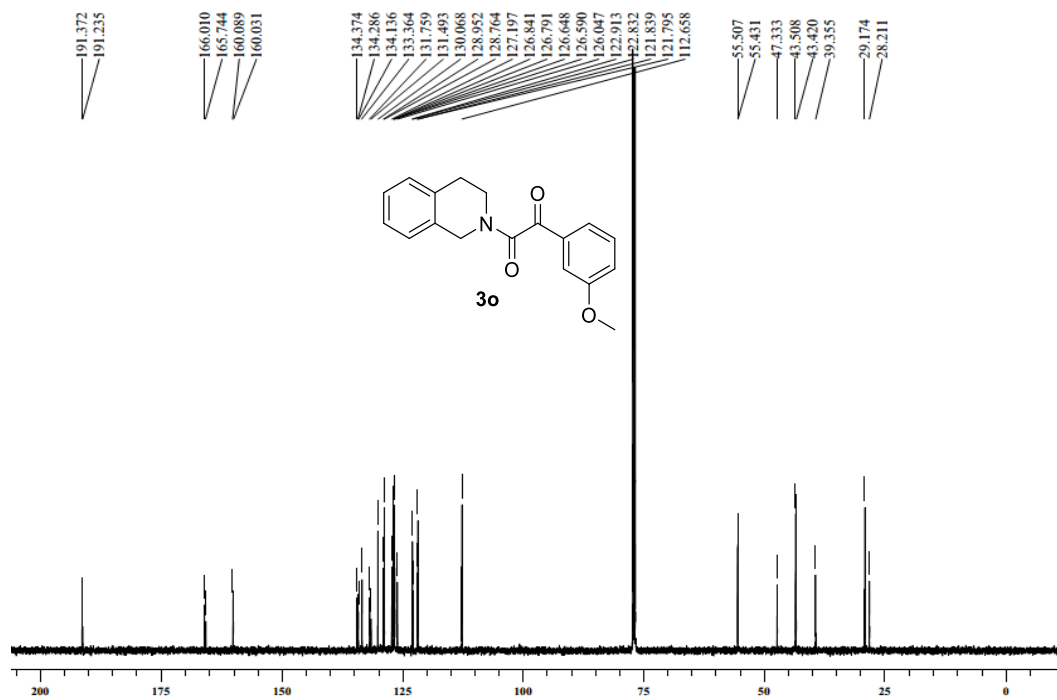




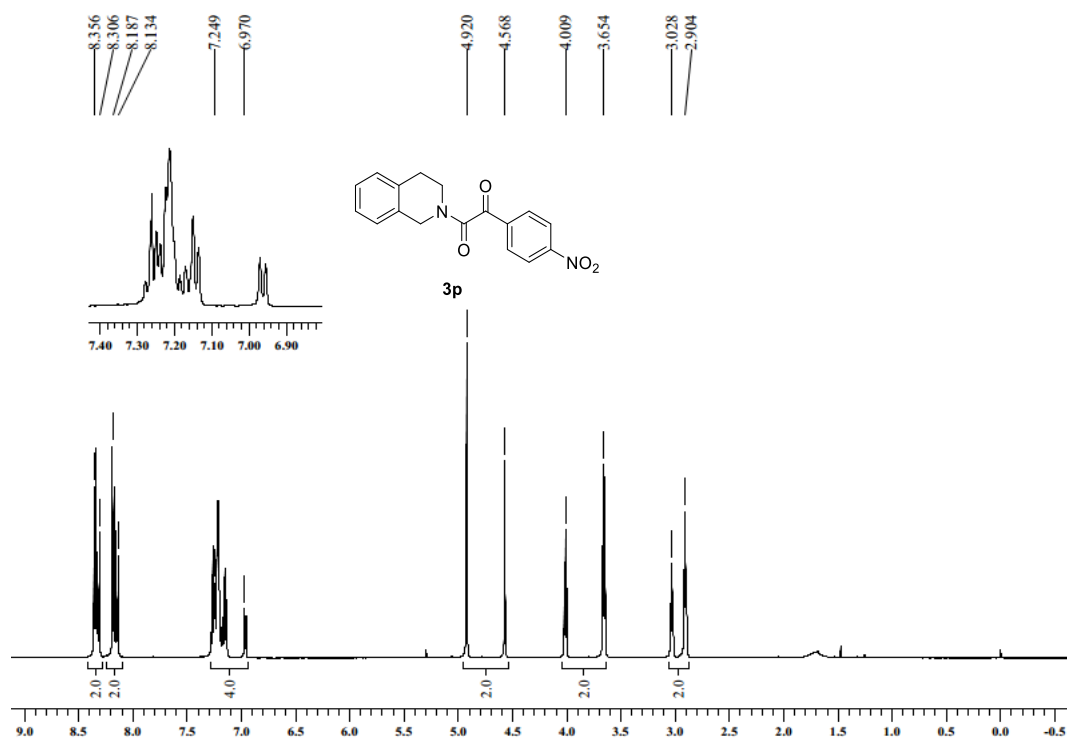




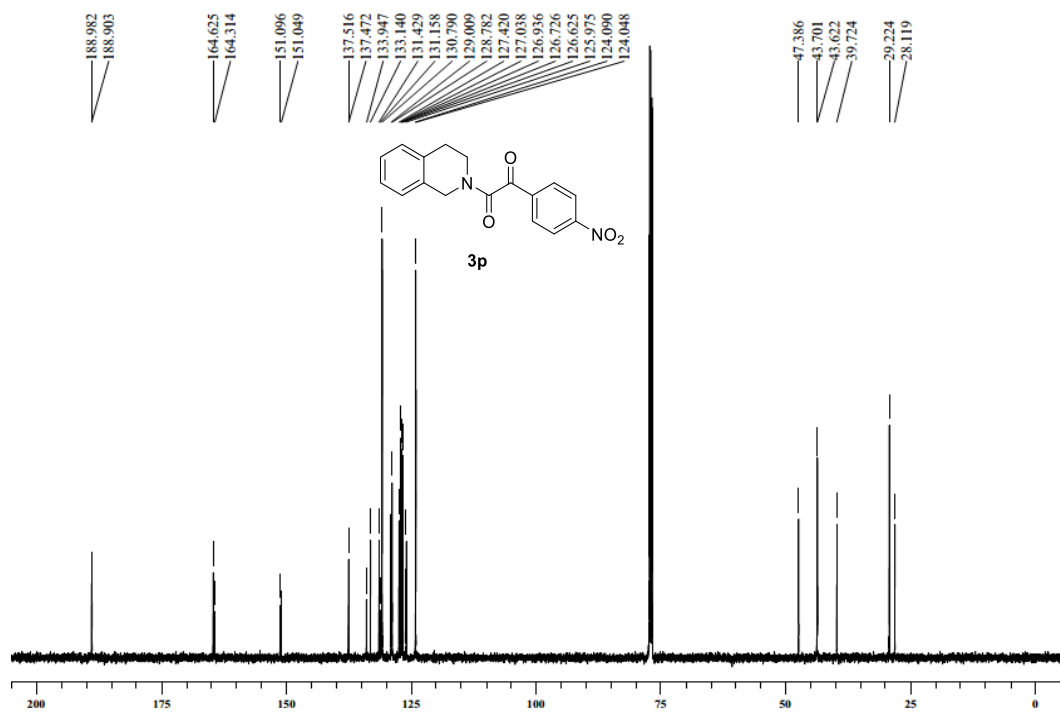
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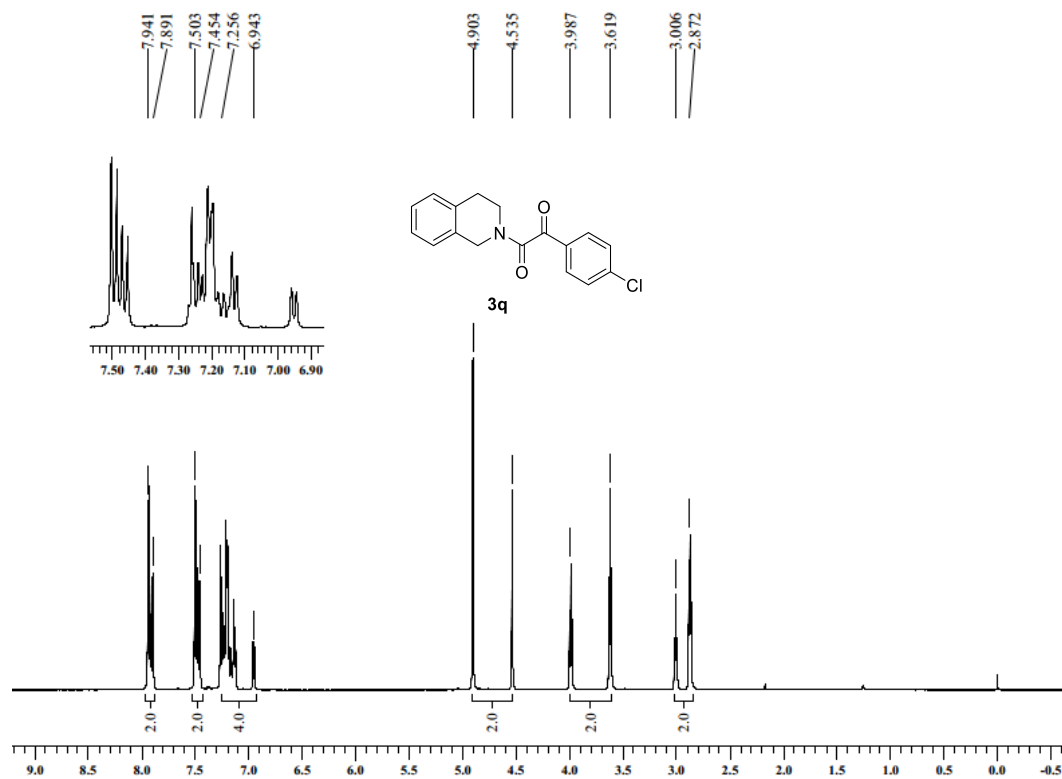
¹³C NMR (125 MHz, CDCl₃) spectrum of compound **3o**



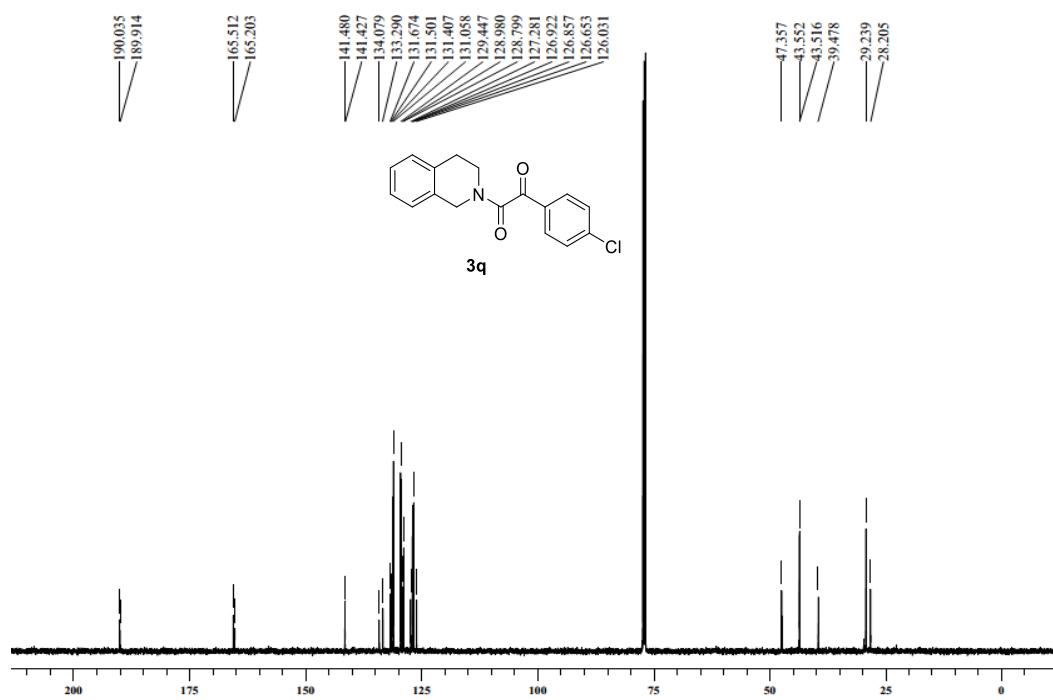
¹H NMR (500 MHz, CDCl₃) spectrum of compound **3p**



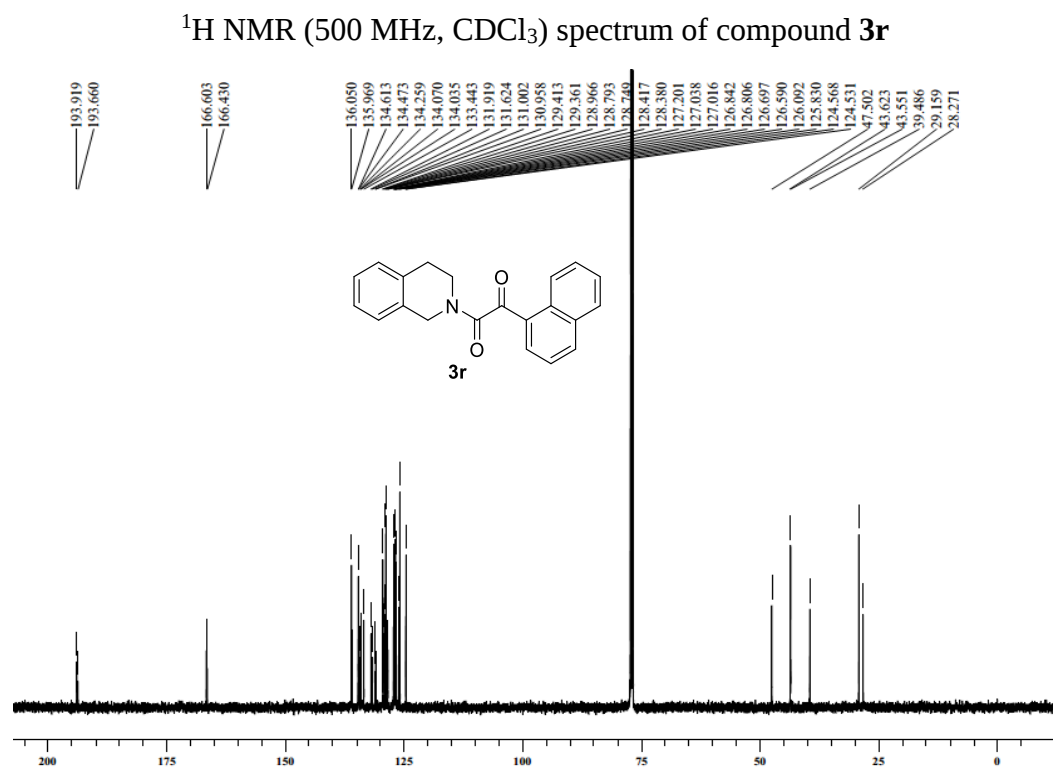
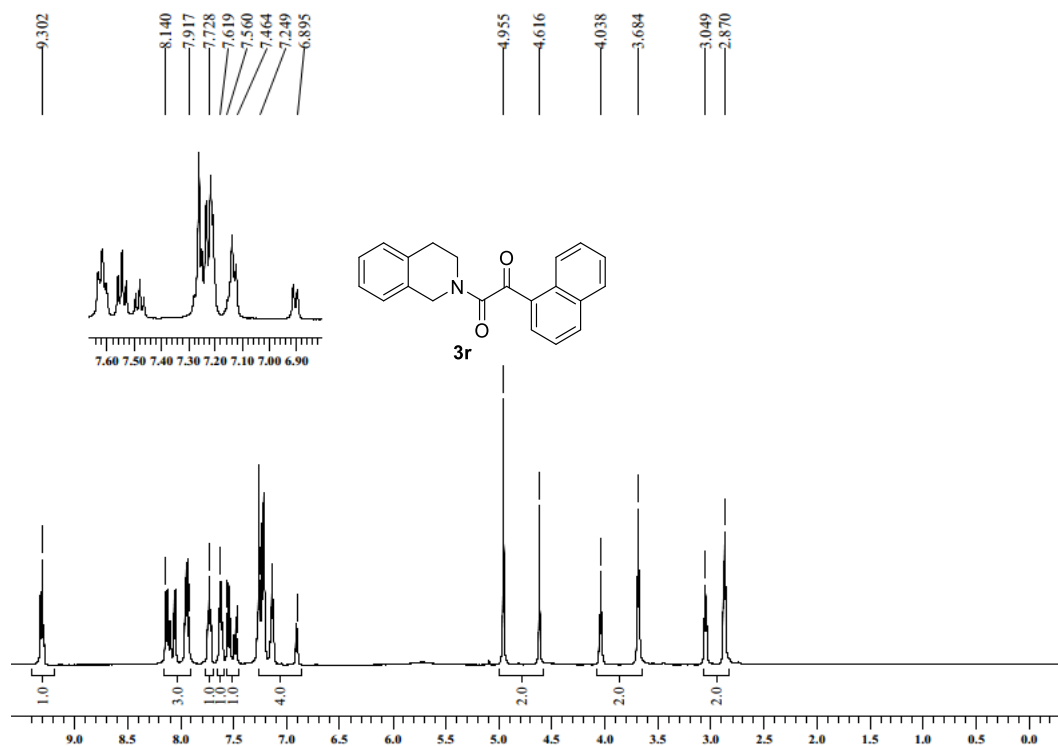
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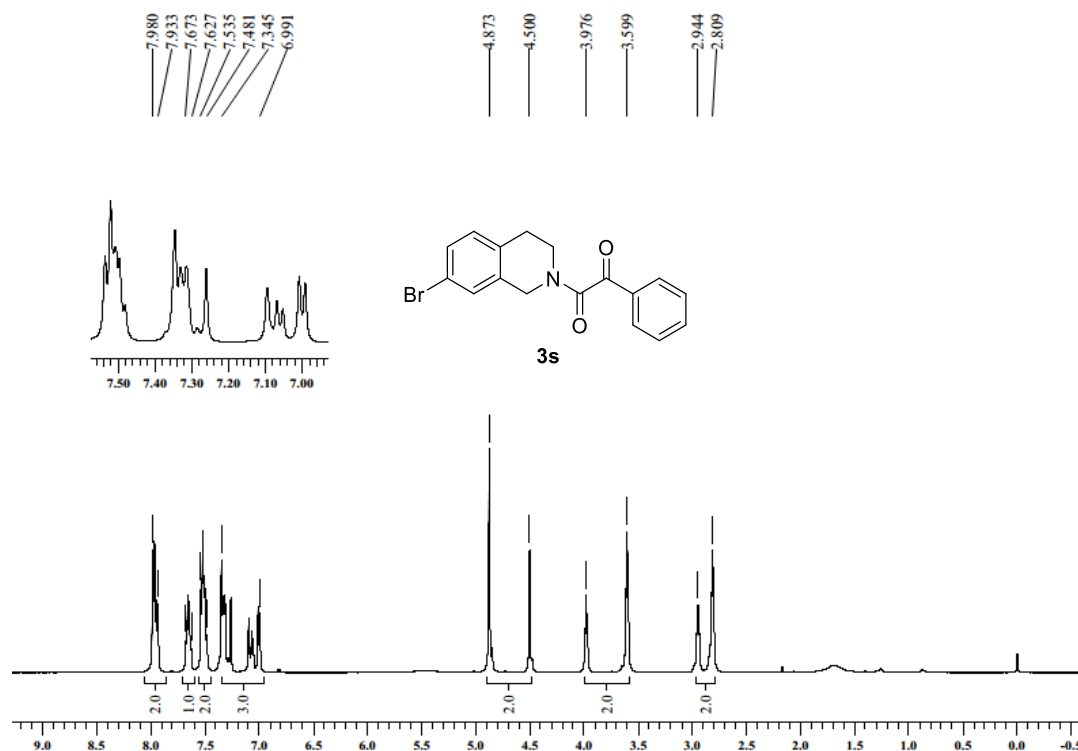
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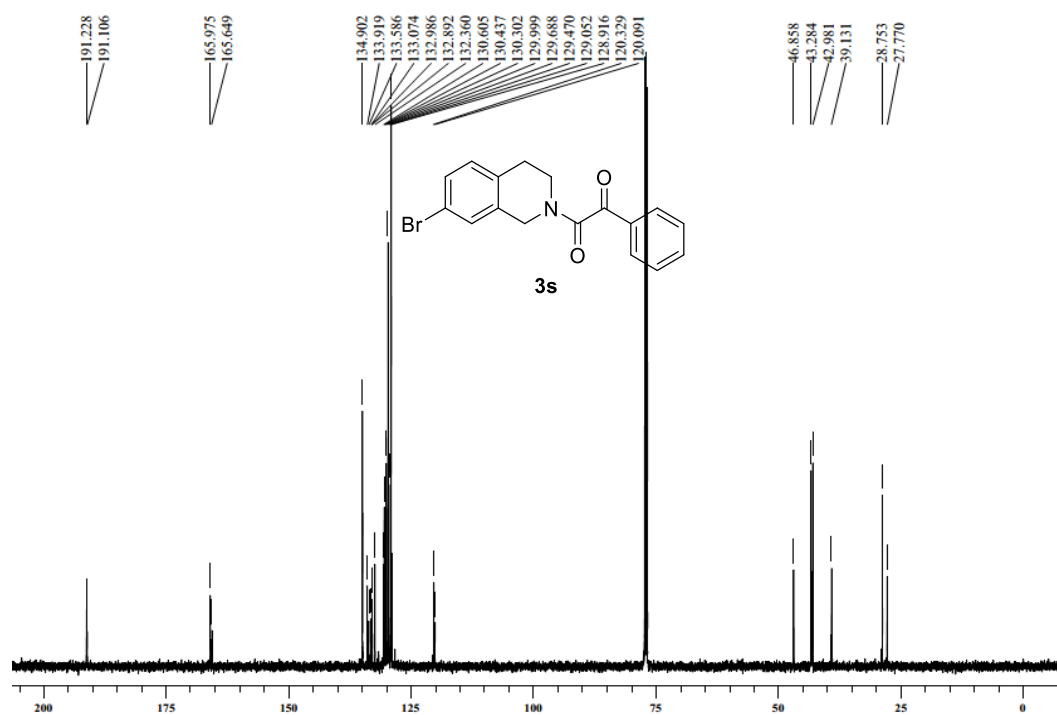
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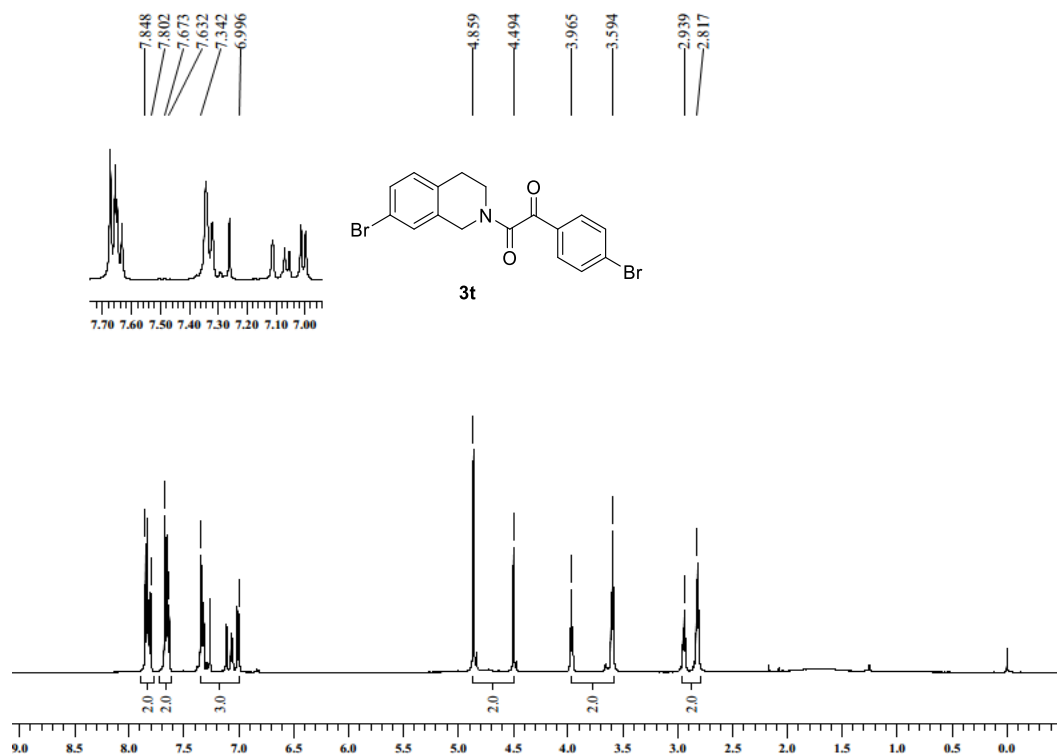
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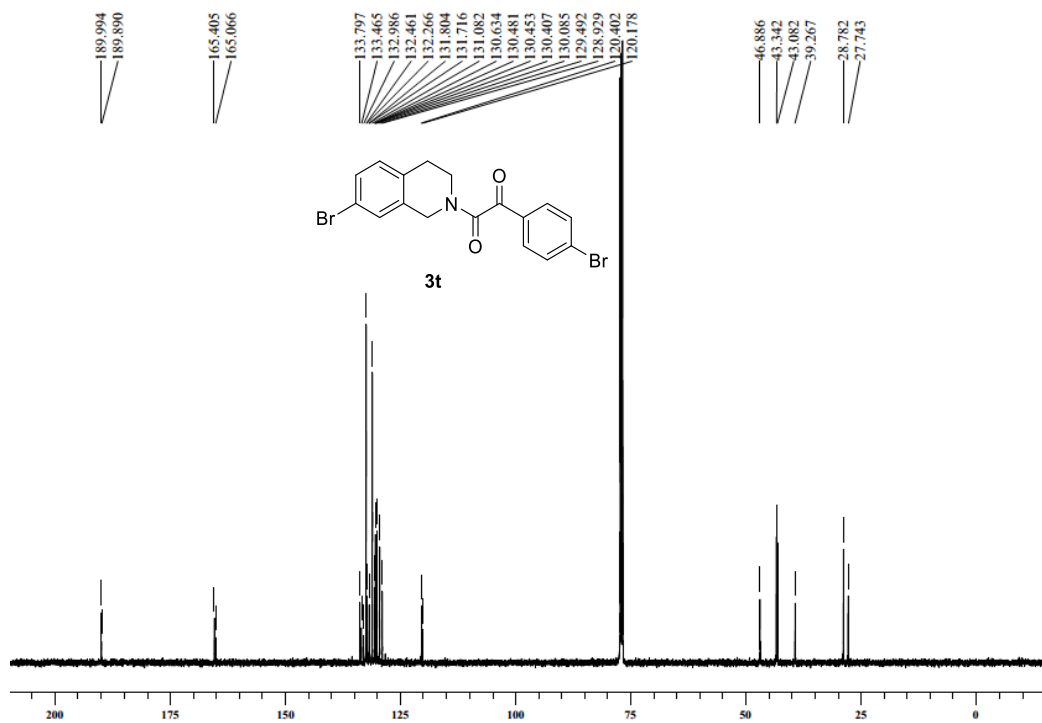
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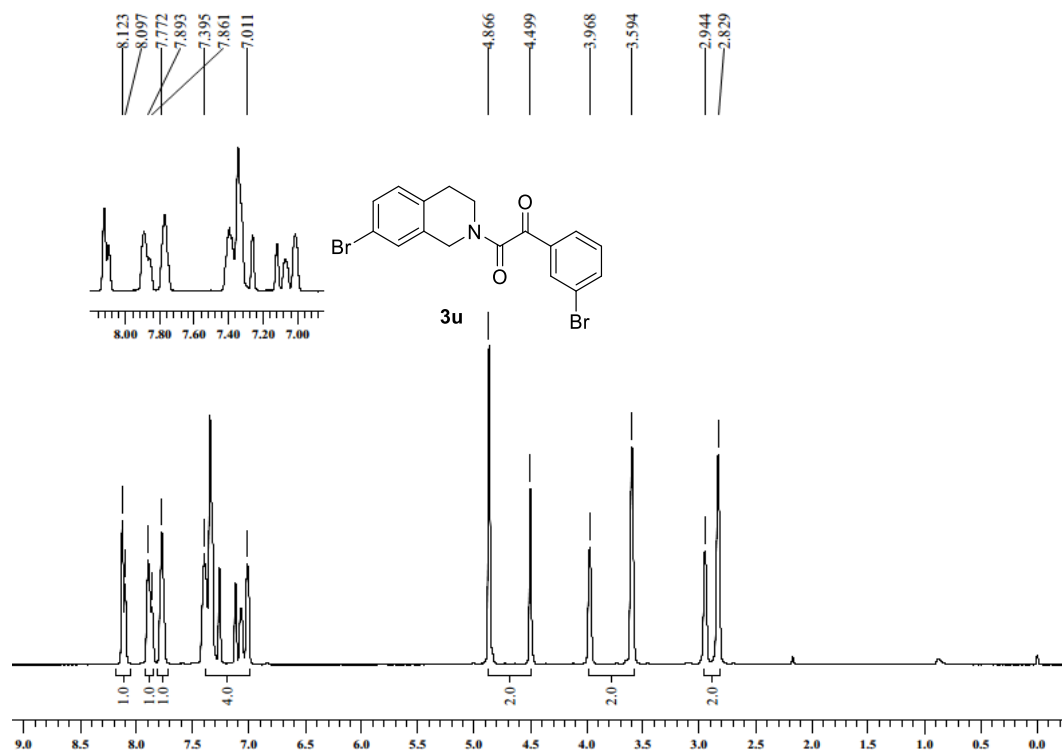
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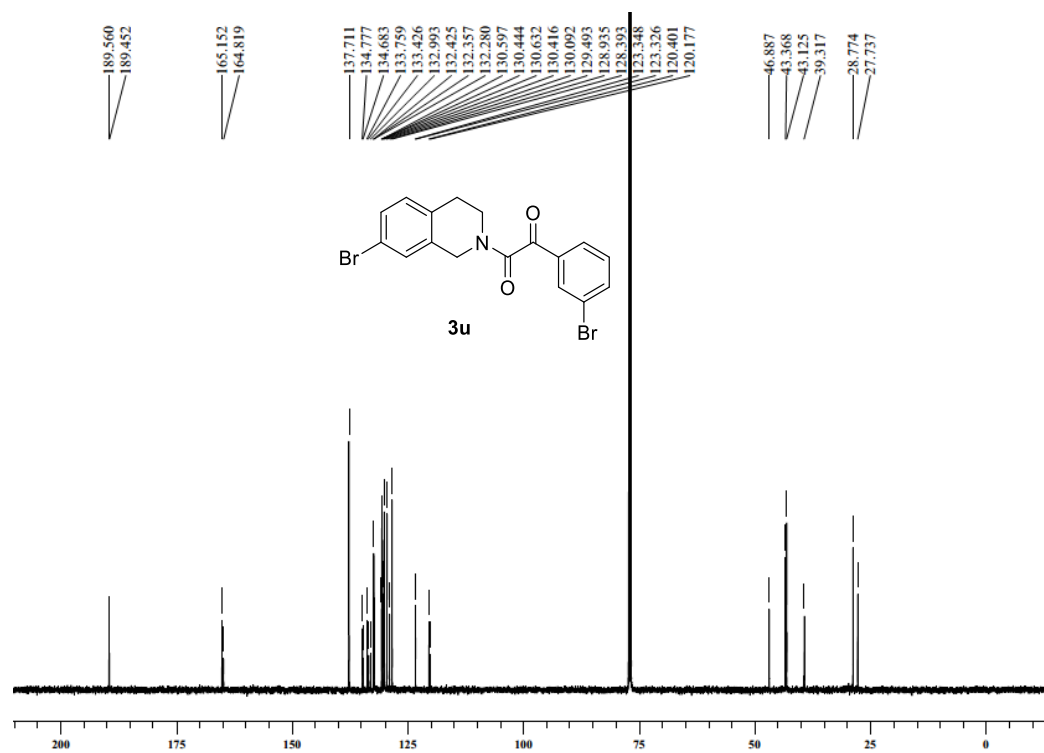
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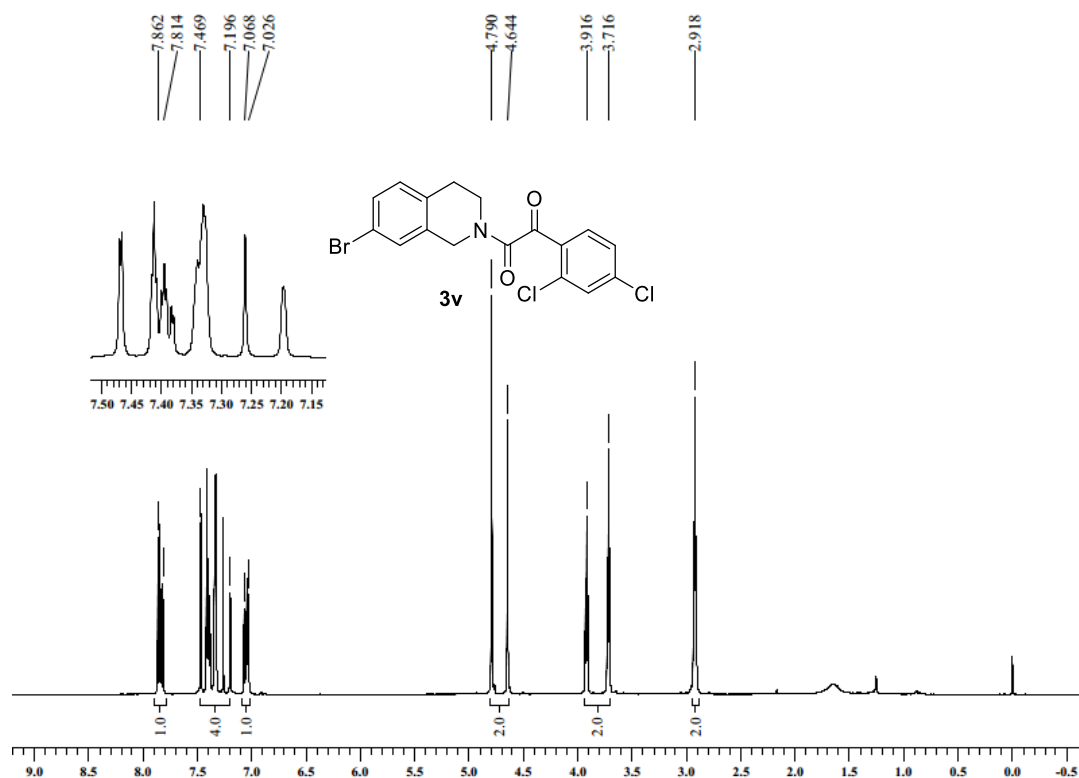
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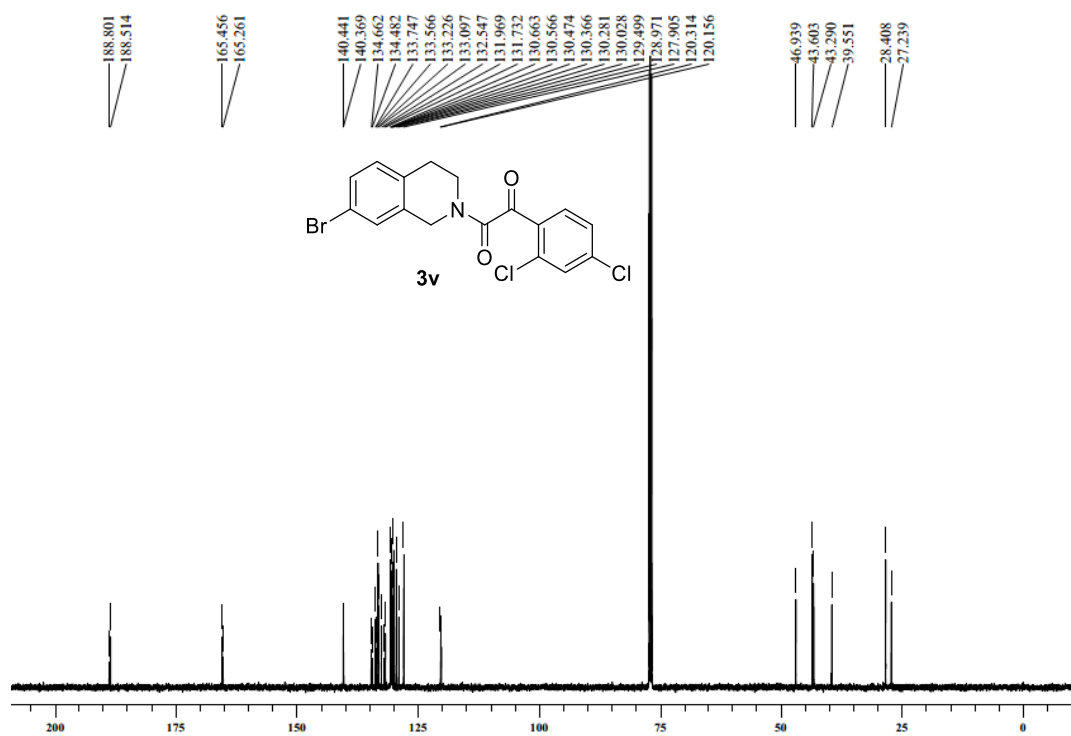
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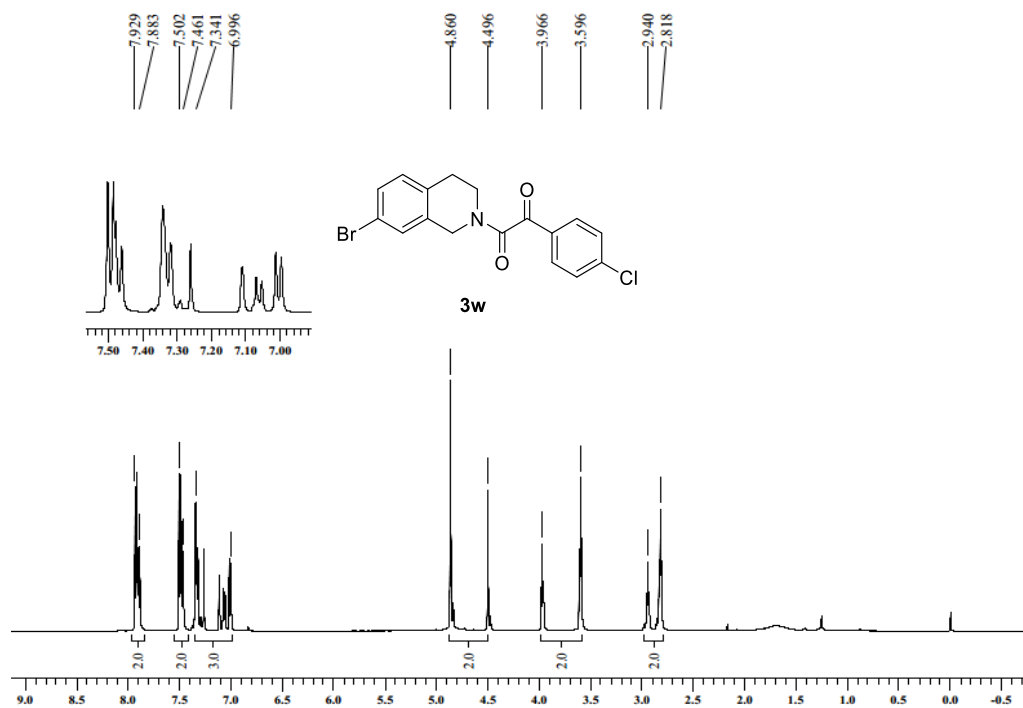
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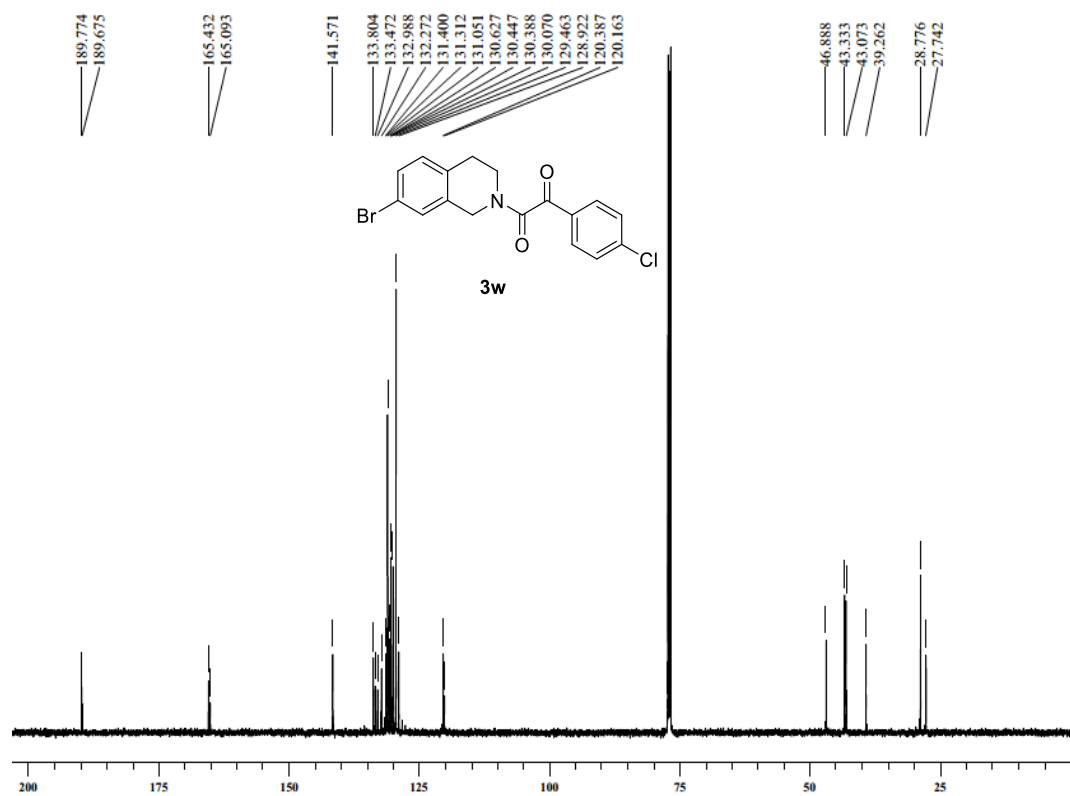
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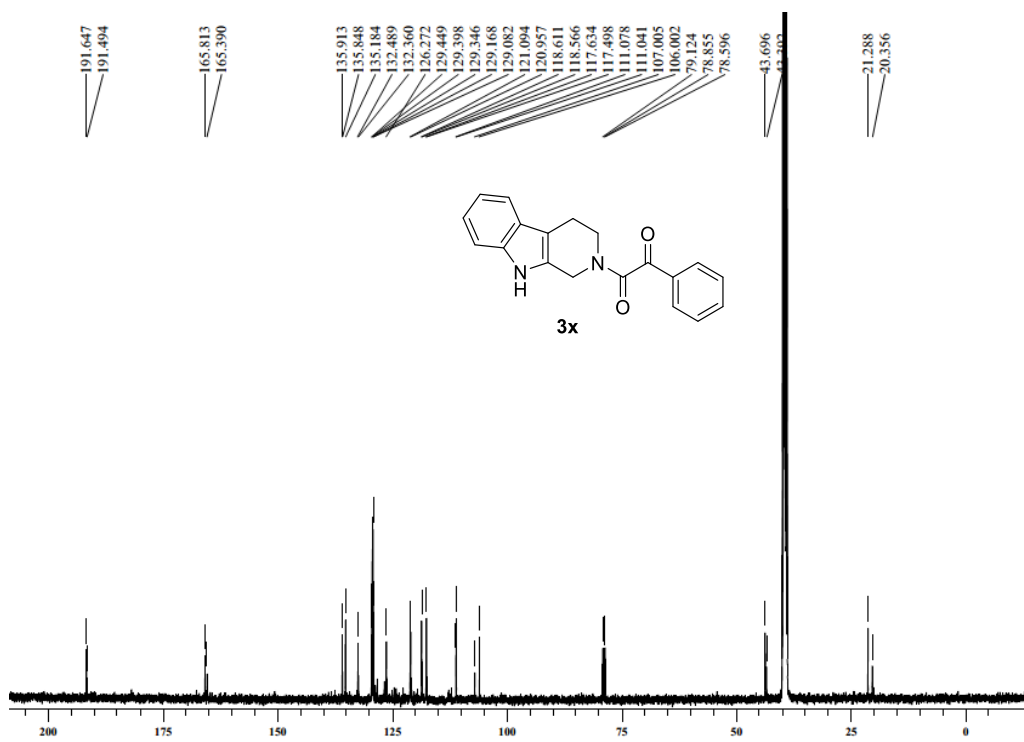
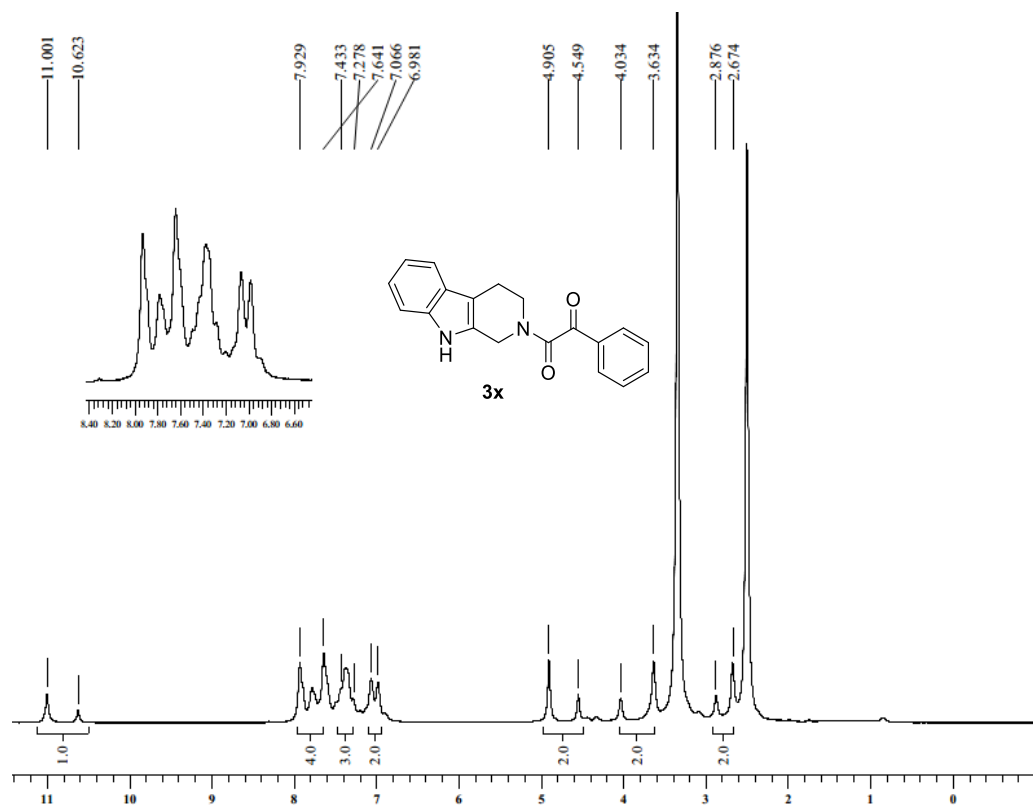
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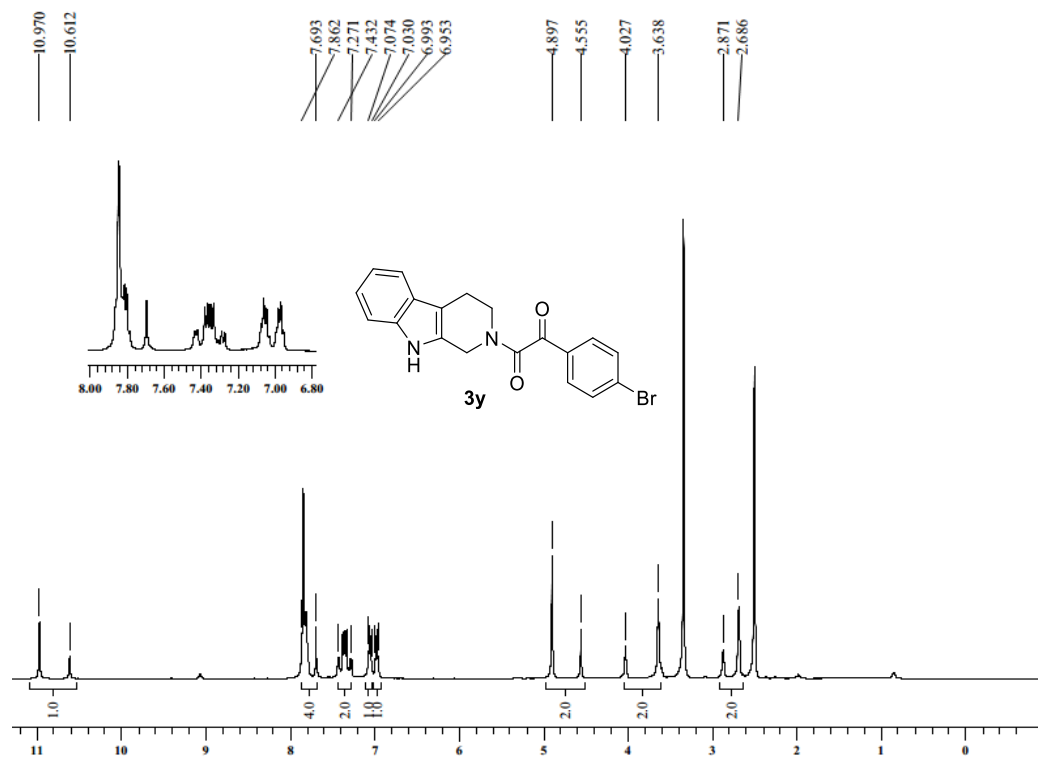


¹H NMR (500 MHz, CDCl₃) spectrum of compound **3w**

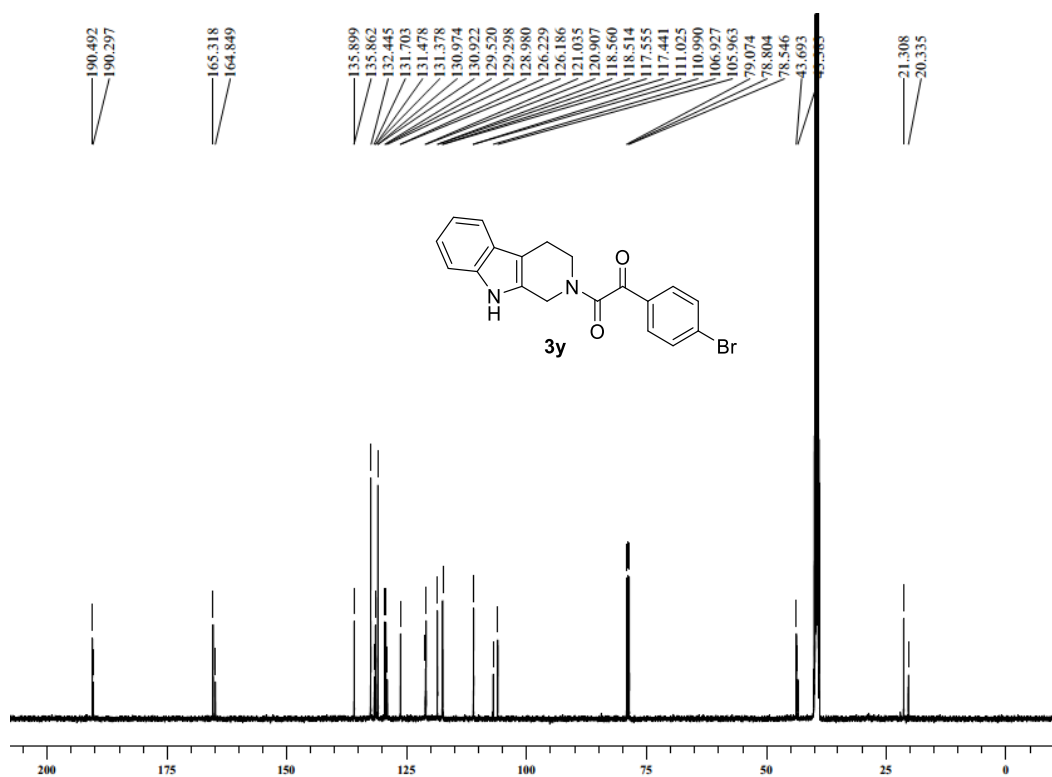


¹³C NMR (125 MHz, CDCl₃) spectrum of compound **3w**

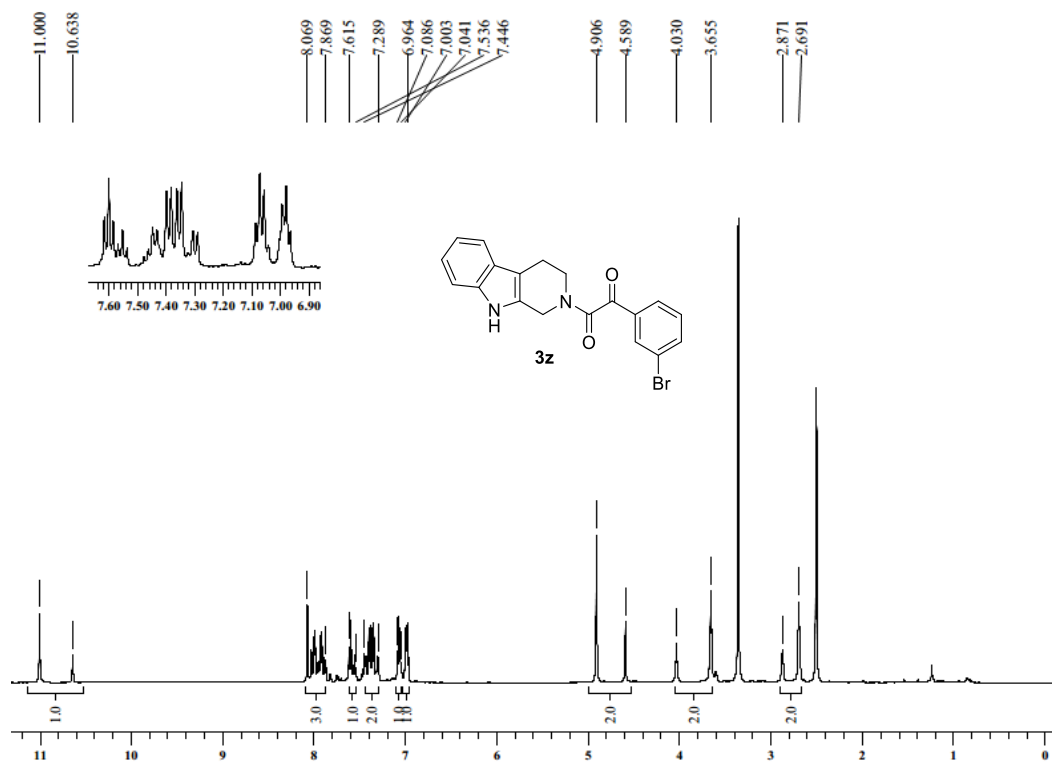




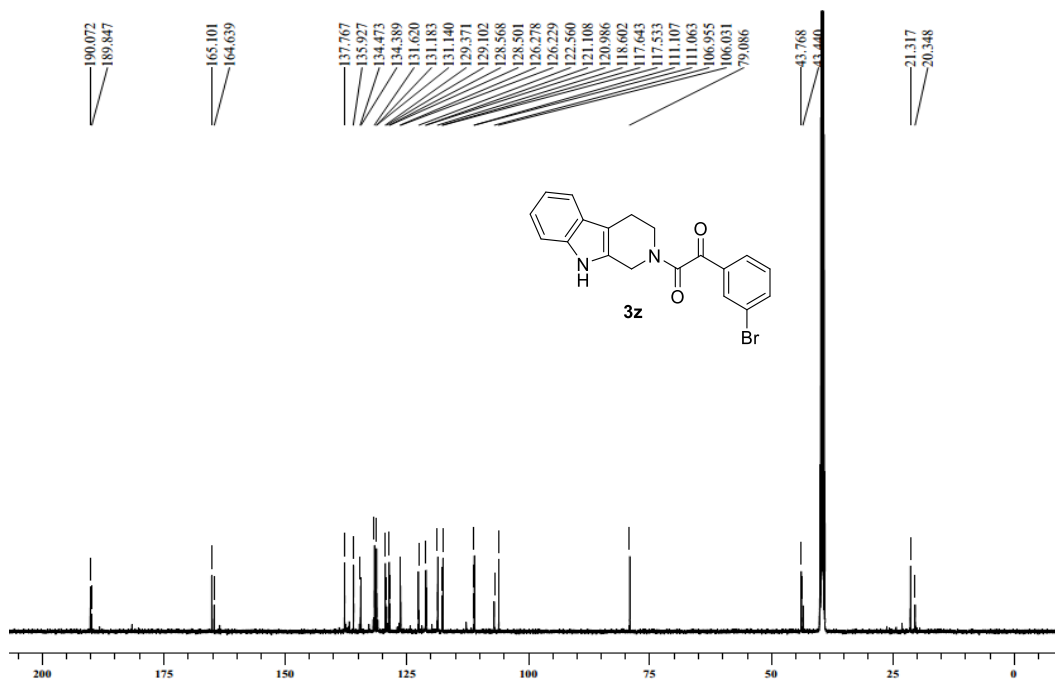
¹H NMR (500 MHz, DMSO-*d*₆) spectrum of compound **3y**



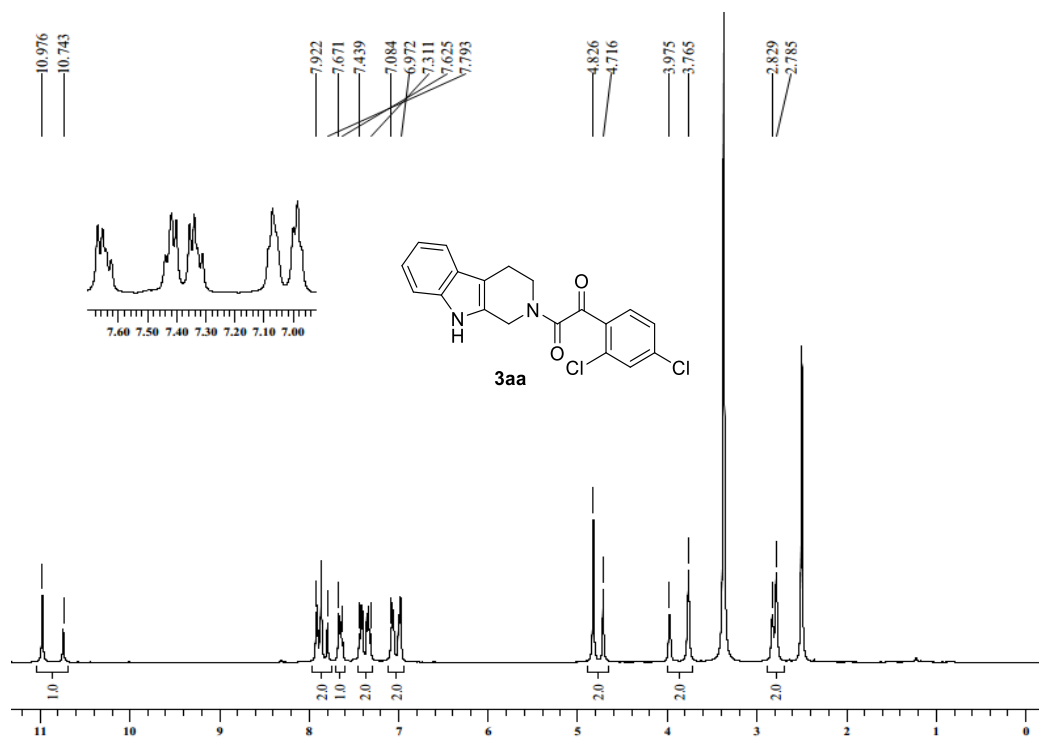
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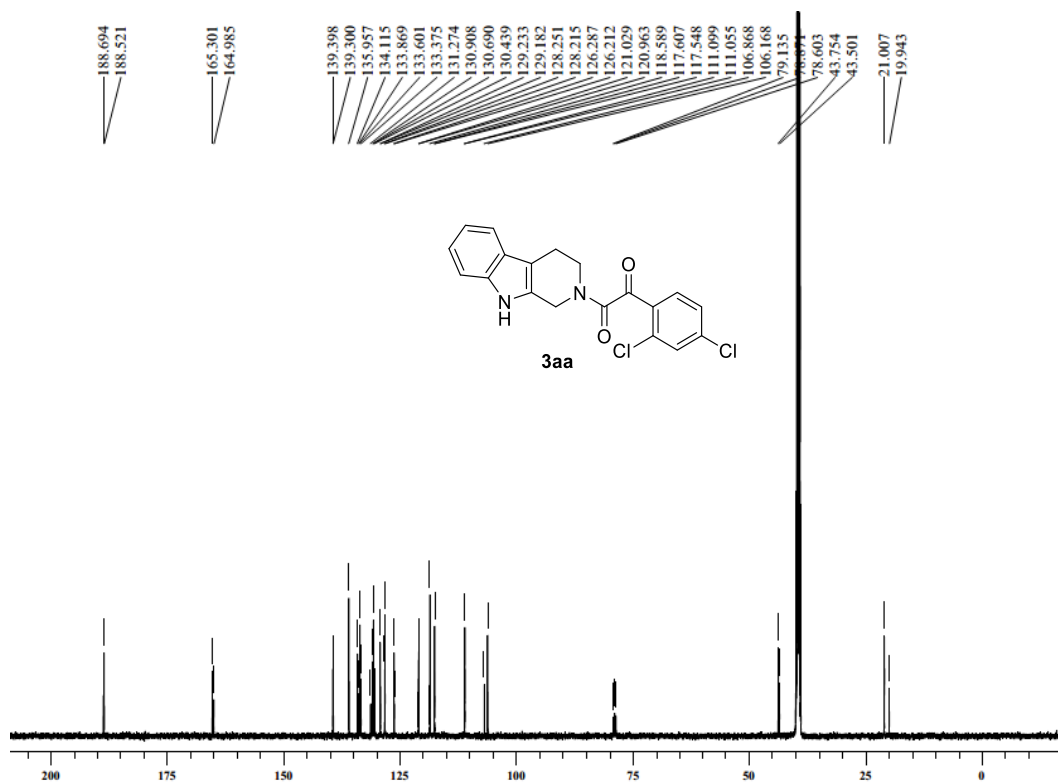
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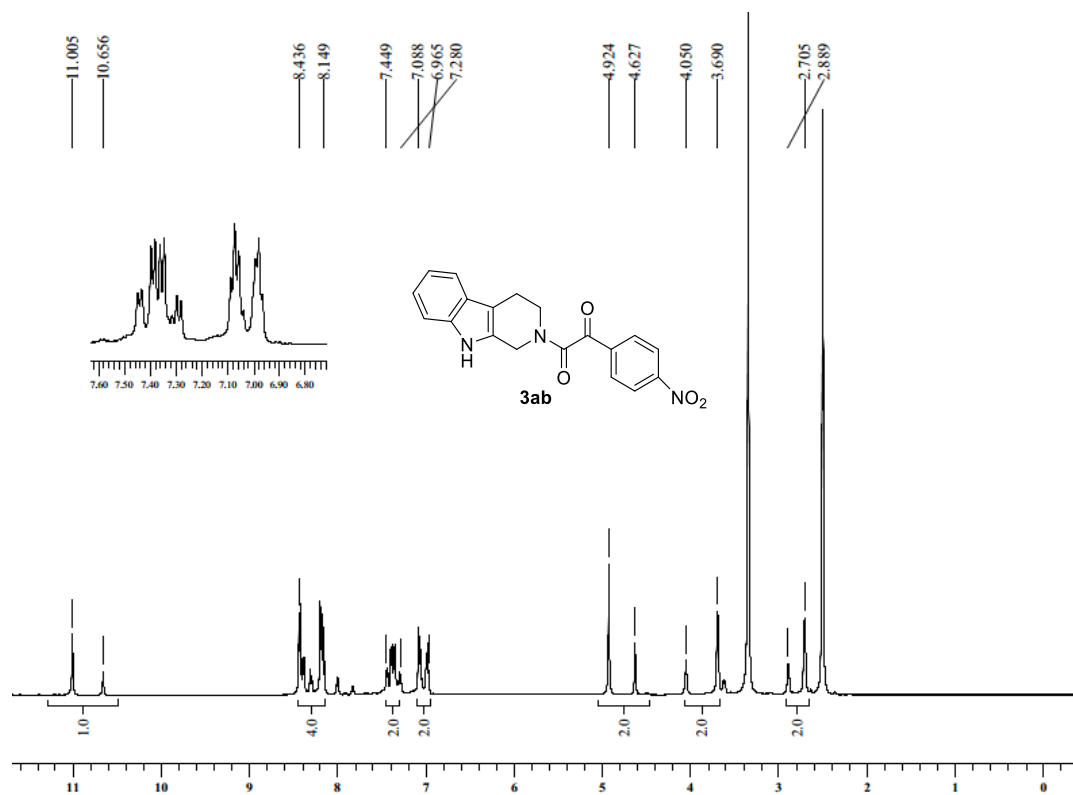
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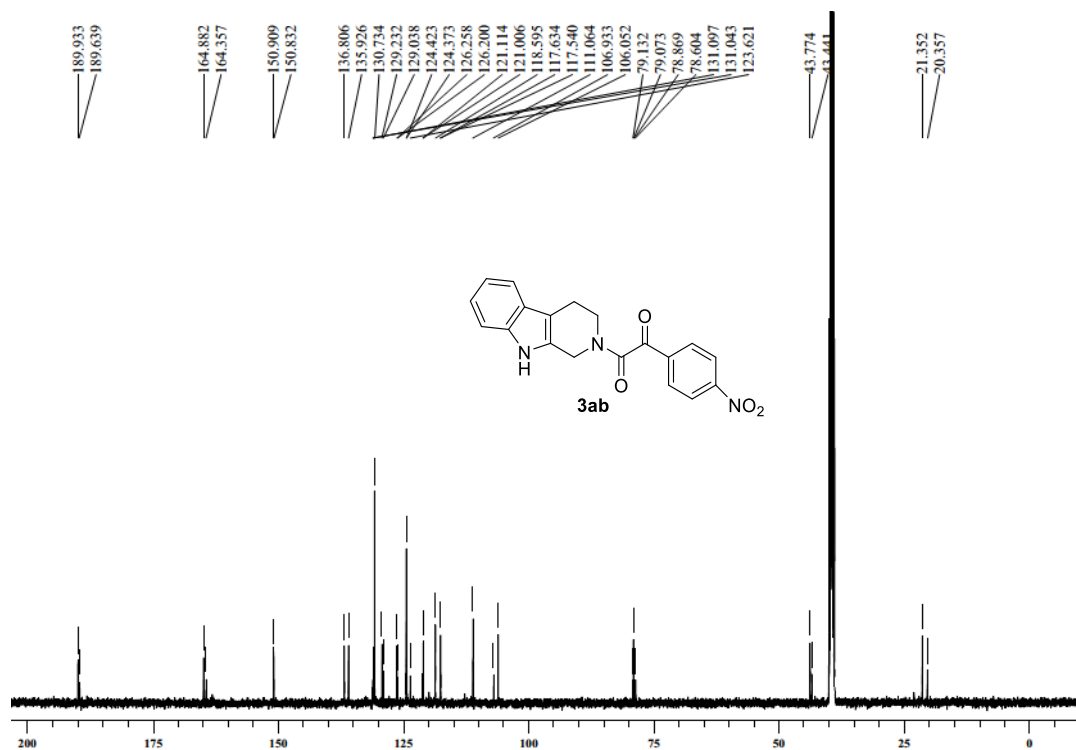
¹H NMR (500 MHz, DMSO-*d*₆) spectrum of compound **3aa**



¹³C NMR (125 MHz, DMSO-*d*₆) spectrum of compound **3aa**



¹H NMR (500 MHz, DMSO-*d*₆) spectrum of compound **3ab**



¹³C NMR (125 MHz, DMSO-*d*₆) spectrum of compound **3ab**

