

Supporting Information

for

Photoredox-Catalyzed 1,2-Oxo-Alkylation of Vinyl Arenes with 1,3-Diketones: An Approach to 1,4-Dicarbonyls via C-C Activation

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1. General Information

^1H NMR was recorded on a Bruker (500 MHz and 400 MHz), and ^{13}C NMR spectra were recorded on a Bruker (125 MHz/101 MHz). The chemical shift (δ) values are given in parts per million (ppm), and the coupling constants (J) are given in hertz (Hz). The spectra were recorded using CDCl_3 solvent. ^1H NMR chemical shifts are referenced to tetramethylsilane (TMS, 0 ppm), and ^{13}C NMR is referenced to CDCl_3 (77.0 ppm). HRMS was recorded with QTOF-ESI source M/S Bruker Daltonik GmbH, Germany, Waters - Xevo G2-XS-QTOF, Agilent 6530 Accurate-Mass Q-TOF LC/MS in ESI mode and Agilent 6230B, TOF in ESI mode. GC-MS was carried out on a GCMS - QP2010 Plus – Shimadzu. Cyclic voltammetry measurements were done with a computer-controlled potentiostat OriogaLys, SAS, Model OGF 500 (OriogaLys Electrochem, SAS, France). The progress of the reaction was monitored by TLC using Merck pre-coated TLC sheets. The Melting points of compounds were determined using a digital melting point apparatus (Model 935) from Deep Vision Electronics PVT. LTD. IP66 50 W Blue LED light from VistaRa fine lighting, China, irradiated the reaction mixture with a portable fan to maintain room temperature ($\sim 25\text{ }^\circ\text{C}$ to $30\text{ }^\circ\text{C}$). Column chromatography was performed on 100–120 mesh silica gel using hexane/ethyl acetate as eluent, and all the solvents were used without further distillation. All commercial chemicals were purchased from Sigma-Aldrich, Alfa Aesar, Spectrochem, Carbanio, Avra and SRL.

2. Parameter variation studies

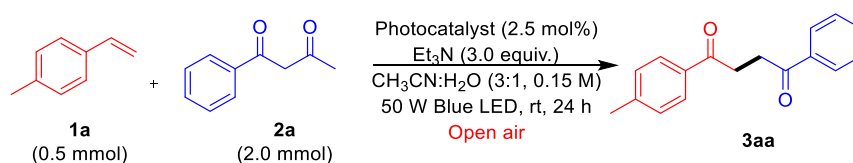
Table S1. 1,4-Diketone and Styrene equivalent variation studies^a



s.no	ratio of 2a:1a	yield of 3aa (%) ^b
1	1:1	20
2	1:2	24
3	2:1	57
4	3:1	78
5	3.5:1	79
6	4:1	84
7	4.5:1	42

^aAll reactions were carried out using 0.5 mmol of 1a. ^bIsolated yield

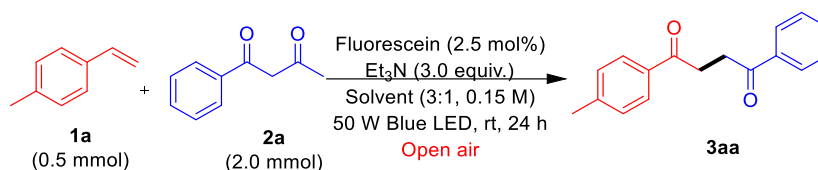
Table S2. Screening of photocatalysts^a



s.no	Photocatalyst	yield of 3aa (%) ^b
1	EY	60
2	RB	63
3	Methylene Blue	13
4	Riboflavin	38
5	Ru	63
6	4-CzIPN	64

^aAll reactions were carried out using 0.5 mmol of **1a**. ^bIsolated yield

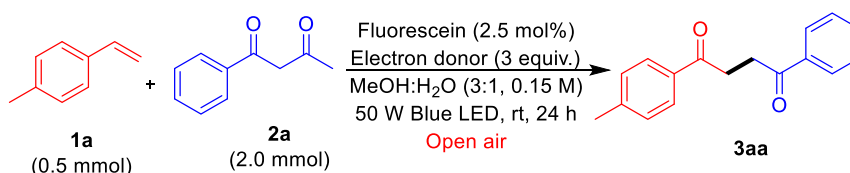
Table S3. Screening of solvents^a



s.no	solvent	yield of 3aa (%) ^b
1	MeOH:H ₂ O	90
2	Acetone:H ₂ O	57
3	DMSO:H ₂ O	N.D.
4	DMF:H ₂ O	N.D.
5	1,4-dioxane:H ₂ O	N.D.

^aAll reactions were carried out using 0.5 mmol of **1a**. ^bIsolated yield. N.D. Not detected.

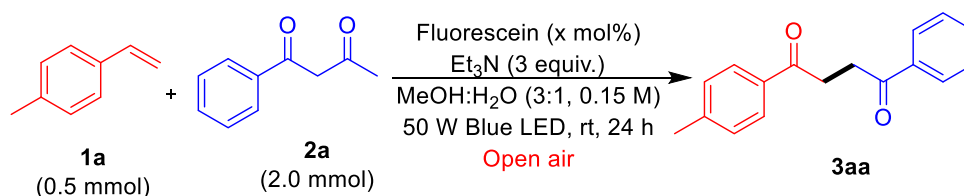
Table S4. Screening of electron donors^a



s.no	electron donor	yield of 3aa (%) ^b
1	Hantzsch Ester	N.D.
2	DIPEA	50
3	TEOA	15
4	DMAP	30

^aAll reactions were carried out using 0.5 mmol of **1a**. ^bIsolated yield. N.D. Not detected.

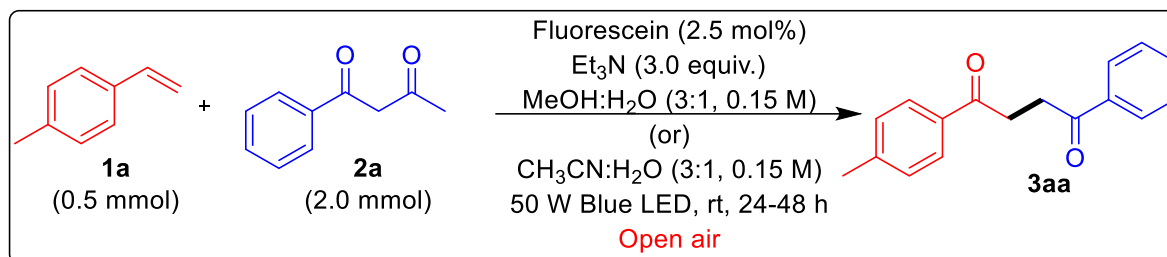
Table S5. Equivalent screening of electron donor and photocatalyst^a



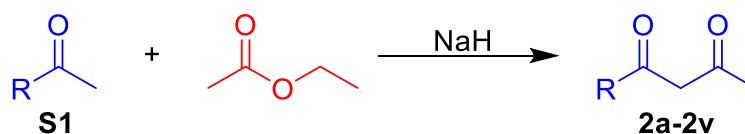
s.no	fluorescein (FL) (mol%)	Et ₃ N (equiv.)	yield of 3aa (%) ^b
1	2.5	Et ₃ N (2)	76
2	2.5	Et ₃ N (4)	78
3	2.0	Et ₃ N (3)	76
4	3.0	Et ₃ N (3)	61

^aAll reactions were carried out using 0.5 mmol of **1a**. ^bIsolated yield

Optimized conditions

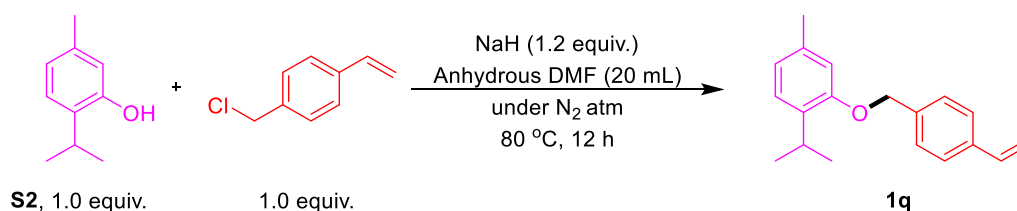


3. General experimental procedure for the synthesis of compounds 2a-v¹



Acetophenone or substituted acetophenone (**S1**, 10.0 mmol, 1.0 equiv.) in ethyl acetate (20 mL) was slowly added to a suspension of NaH (60% dispersion in mineral oil 40 mmol, 4.0 equiv.) in ethyl acetate (**S2**, 20 mL) at 0 °C. Then, the resultant mixture was stirred at room temperature for 12 h. After completion of the reaction, the reaction mixture was carefully treated with 10% aq. NH₄Cl (30 mL) and the pH were adjusted to 5 using diluted HCl (5 N). The aqueous phase was then separated and extracted with ethyl acetate. The combined ethyl acetate layers were dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to obtain the crude reaction product. The obtained crude was purified using column chromatography (silica gel, hexane/ethyl acetate) to afford the desired product.

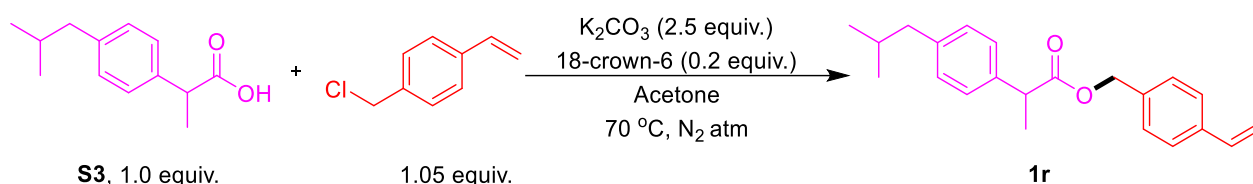
4. Experimental procedure for Synthesis of compound (1q)²



To a stirred solution of thymol (**S2**, 3.0 mmol) in anhydrous DMF (20 mL) was added NaH 60% in mineral oil (3.6 mmol) at 0 °C (ice bath). After stirring for 30 min, 1-(chloromethyl)-4-vinylbenzene (3.0 mmol) was added at 0 °C under N₂. The reaction mixture was stirred at 80 °C with an oil bath for 12 h. After the reaction,

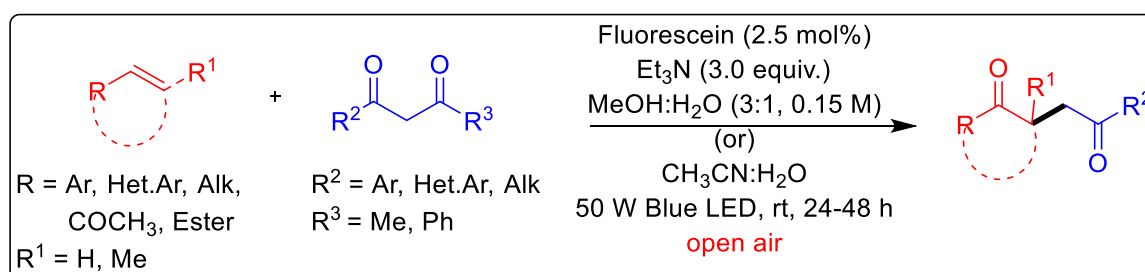
the mixture was quenched with 10 mL of water. The aqueous layer was extracted with ethyl acetate (3 x 10 mL). The combined ethyl acetate layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to get a crude compound. The obtained crude was purified using column chromatography by eluting hexane to afford the desired product (**1q**).

5. Experimental procedure for Synthesis of compound (**1r**)³



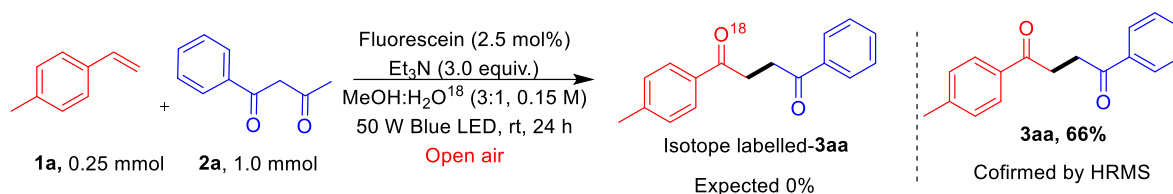
In a 25 mL round-bottom flask, ibuprofen (**S3**, 1.0 equiv.), K₂CO₃ (2.5 equiv.), and 18-crown-6 (0.2 equiv.) were added and dissolved in acetone. Then this mixture was purged with N₂ and added 1-(chloromethyl)-4-vinylbenzene (1.05 equiv.). The reaction solution was vigorously stirred under N₂ and heated at 70 °C. The completion of the reaction was monitored by TLC chromatography. After completion, the reaction mixture was diluted with 10 mL of water. The aqueous layer was extracted with ethyl acetate (3 x 10 mL). The combined ethyl acetate layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to get a crude compound. The obtained crude was purified using column chromatography by eluting hexane to afford the desired product (**1r**).

6. General experimental procedure (A) for 1,4-diketones **3aa-aw**, **3ba-gw**, **3na-3ra**

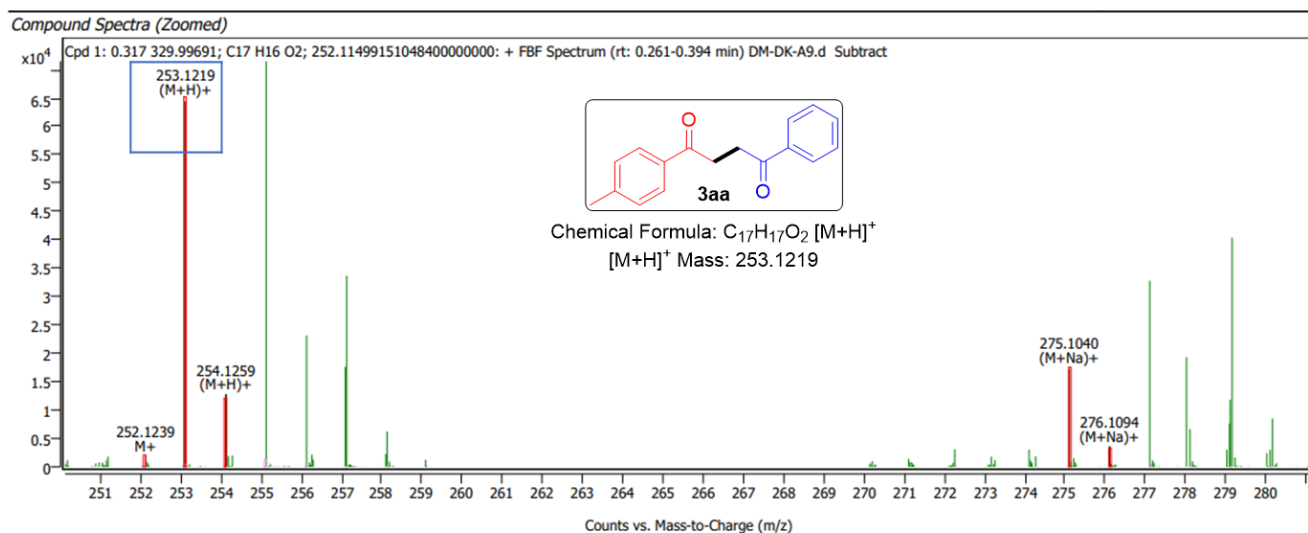


Compound **2a-2z** (2.0 mmol) was dissolved in 0.15 M of solvent (MeOH:H₂O or CH₃CN:H₂O (3:1, 3.3 mL) followed by the addition of triethylamine (3.0 equiv.), fluorescein (2.5 mol %), and alkenes **1a-1r** (0.5 mmol). The reaction mixture was stirred under the 50 W blue LED in the open air until completion of the reaction, monitored by TLC chromatography (~24-48 h). The reaction mixture was diluted with 10 mL of water. The aqueous layer was extracted with ethyl acetate (3 x 10 mL). The combined ethyl acetate layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to get a crude compound. The obtained crude was purified using column chromatography by eluting hexane/ethyl acetate to afford the desired product **3aa-aw**, **3ba-gw**, **3na-3ra** in average to good reaction yields.

7. Experimental procedure for the H₂O¹⁸ studies



The reaction was performed with standard conditions using MeOH:H₂O¹⁸ (3:1, 0.15M) on a 0.25mmol scale of **1a**. The reaction completion was monitored by TLC chromatography. The reaction mixture was diluted with 10 mL of water. The aqueous layer was extracted with ethyl acetate (3 x 10 mL). The combined ethyl acetate layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to get a crude compound. The obtained crude was purified using column chromatography by eluting hexane/ethyl acetate (3%) to afford the desired product **3aa** in 83 mg yield of 66% as a white solid. HRMS (ESI) calculated for C₁₇H₁₇O₂ [M+H]⁺: 253.1219 found 253.1219.



Compound Details

Cpd. 1: C17 H16 O2

Formula	m/z	Observed M/Z	Difference Da	Difference PPM	Score
C17 H16 O2	253.1219	253.12188922933	-0.0382445159345934	-0.151694708450181	85.07

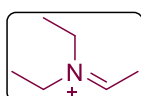
Figure S1. HRMS analysis data of H₂O¹⁸ studies

8. Reaction monitoring studies by GC-MS analysis

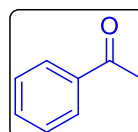
GC-MS chromatogram analysis of the reaction mixture of **3aa** was performed on GCMS - QP2010 Plus – Shimadzu during the courses of reaction (2 h, 6 h, 10 h, 18 h, 24 h) and the intermediates are verified. We have detected the following intermediates, product **3aa** and the minor side products. This strongly suggests that the mechanism goes via proposed in the manuscript.



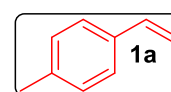
Chemical Formula: C₂H₄O
m/z: 44



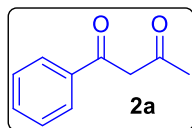
Chemical Formula: C₆H₁₄N⁺
m/z: 100



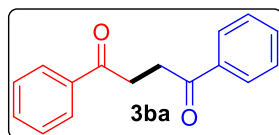
Chemical Formula: C₈H₈O
m/z: 120



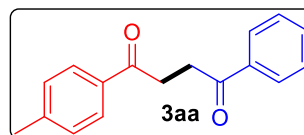
Chemical Formula: C₉H₁₀
m/z: 118



Chemical Formula: C₁₀H₁₀O₂
m/z: 162



Chemical Formula: C₁₇H₁₆O₂
m/z: 252



Chemical Formula: C₁₇H₁₆O₂
m/z: 252

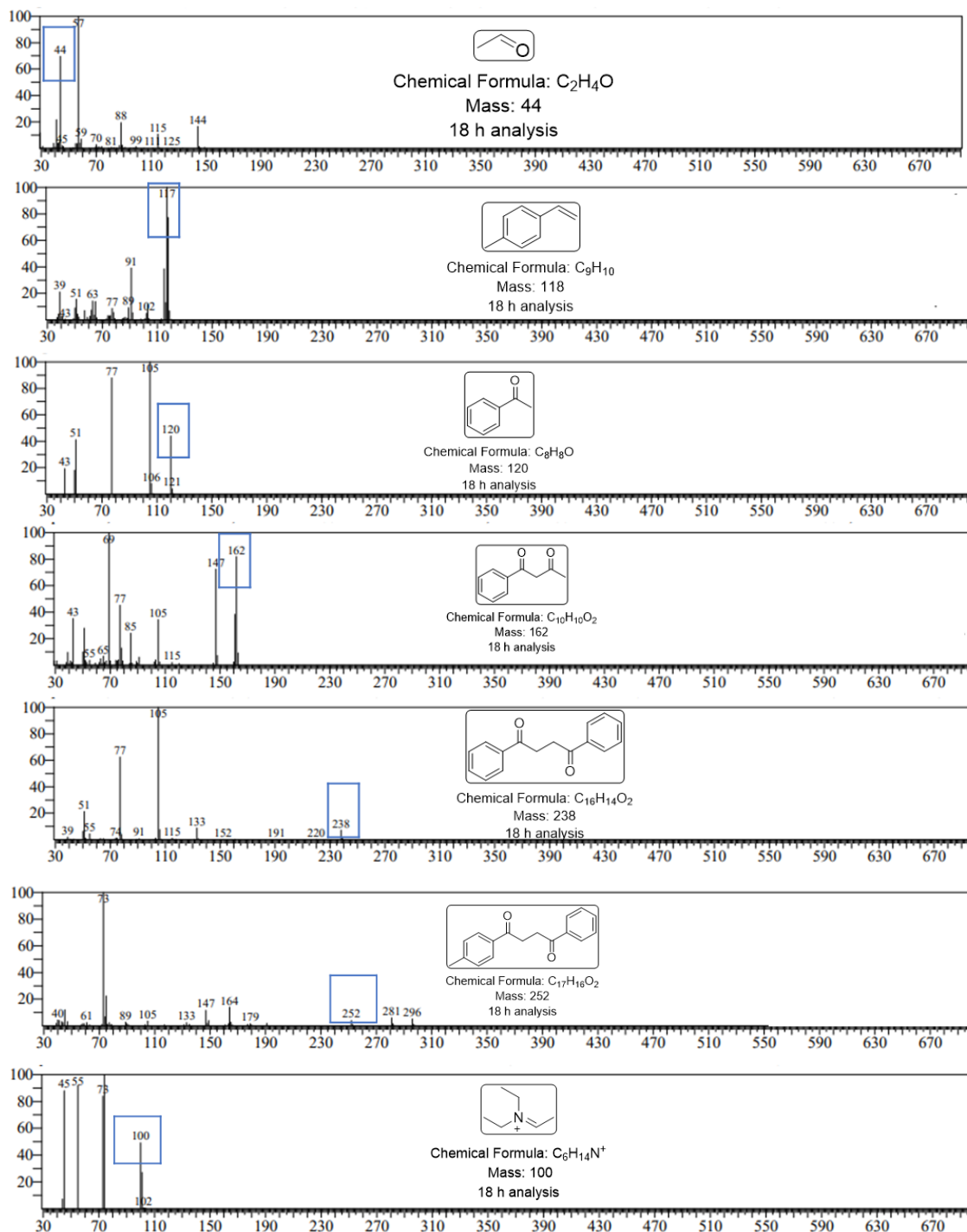
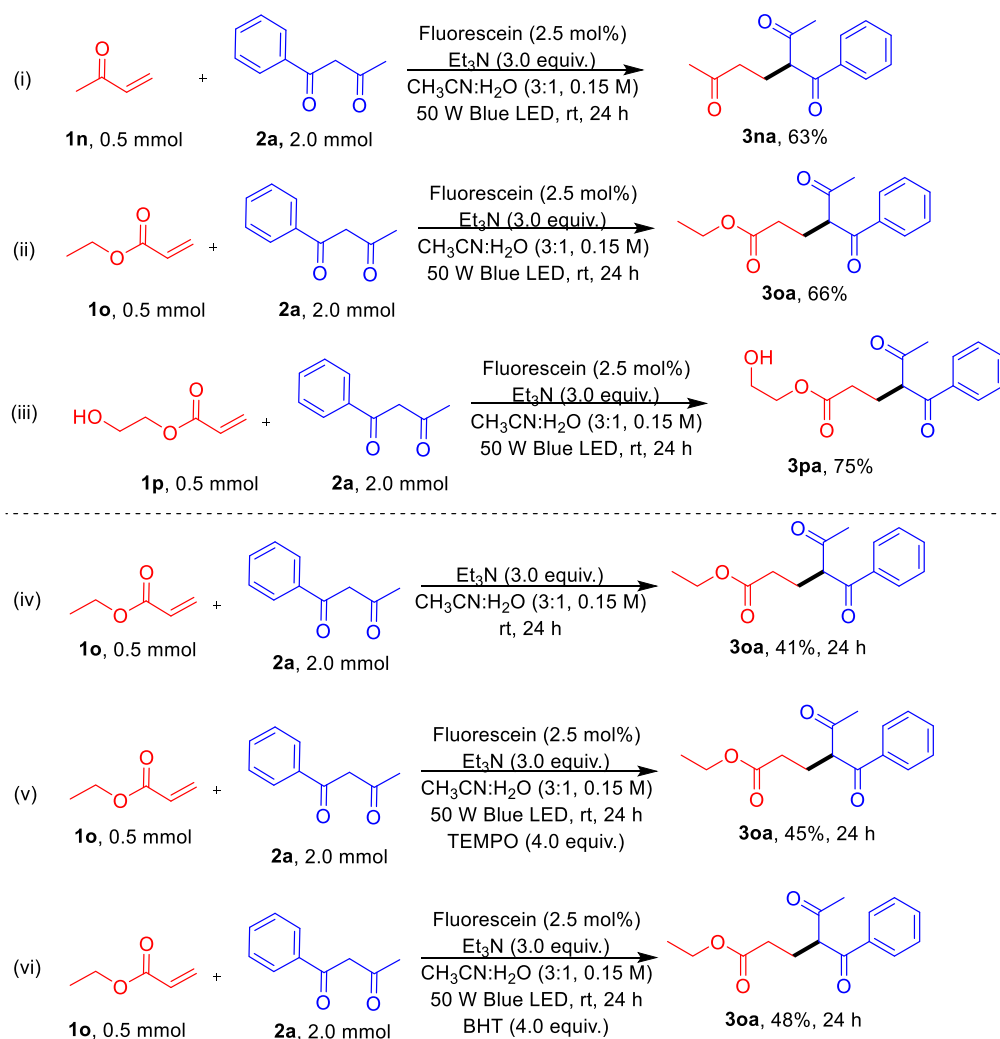


Figure S2. GC-MS analysis data of reaction

9. Control experiment for activated alkenes

While testing the scope of reaction with activated alkenes such as vinyl ketone (**2n**) and vinyl esters (**2o-p**). Interestingly, the reaction gave 1,2-hydroalkylation product **3oa-pa** in 63% - 75% yields. To confirm the role

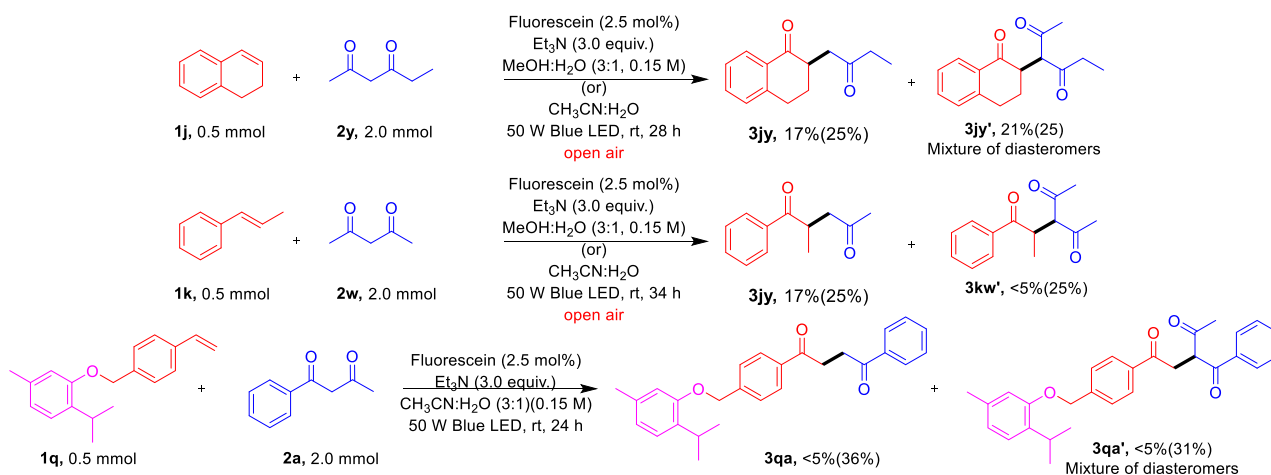
of visible-light photocatalysis in the 1,2-hydroalkylation reaction, we have performed the reaction of ethyl acrylate (**2o**) at room temperature without light/photocatalyst and also with radical scavengers (TEMPO & BHT). The reaction proceeded moderately, and the obtained reaction yields less than that of light-mediated reactions. These results weakly suggest that the visible light can drive to a better product yield.



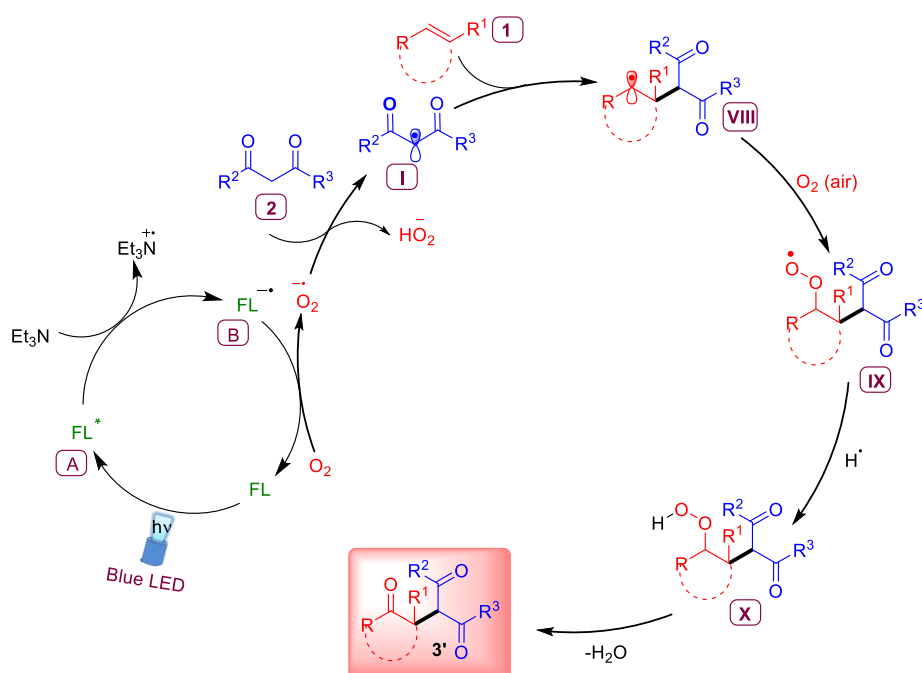
Scheme S1. Reaction of activated alkenes and control studies with ethylacrylate (**1o**).

10. Mechanism for minor side product

When we performed the reaction using CH₃CN:H₂O (Table 1, entry 5) conditions, we observed the formation of a 1,2-oxo-alkylation reaction of styrenes with 1,3-dicarbonyls before initiation of C-C activation to deliver the compounds **3jy'**, **3kw'** and **3qa'** as a side-product. In all the cases when the reaction was carried out in CH₃CN:H₂O, the formation of 1,2-oxo-alkylation was observed by trapping 1,3-diketone- α -carbon radical (**I**) with vinyl arenes before the C-C activation step to afford triketones in 5% to 10%, and it was traced in the case of MeOH:H₂O as a solvent. In this regard, we have isolated three derivatives which are formed more than 10% of the yield, as mentioned in Scheme S2. A plausible mechanism was also proposed for the triketones based on the reaction outcomes, as shown in Scheme S3.



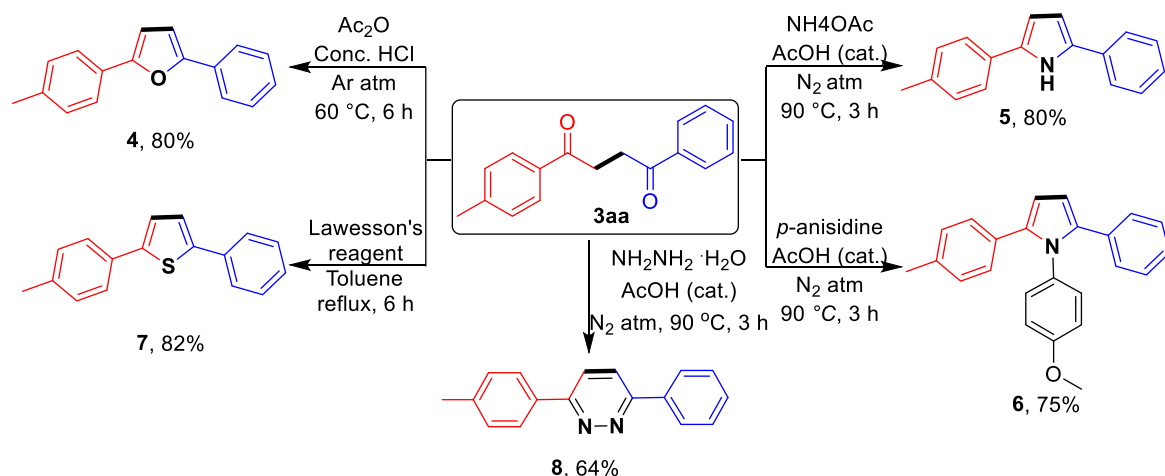
Scheme S2. Reactions for minor by-products. Yields mentioned in the brackets correspond to CH₃CN:H₂O condition



Scheme S3. Plausible reaction mechanism for the minor by-product

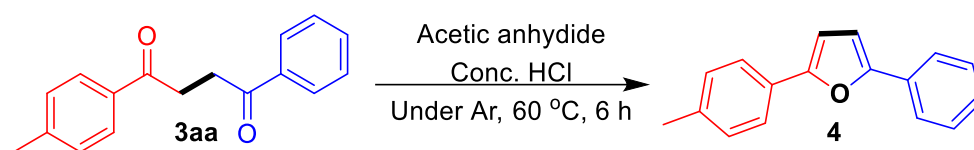
11. Synthetic applications

With the successful outcome of a variety of 1,4-diketones, we next stepped into their utilization as a potential building block for synthesizing five- and six-membered heterocyclic compounds. In this context, compound 3aa was chosen as a representative 1,4-diketone for synthesizing 2,4-disubstituted furan (**4**), 2,5-disubstituted pyrrole (**5&6**), 2,5-disubstituted thiophene (**7**) and 3,6-disubstituted pyridazine (**8**) with suitable coupling partners as shown in Scheme S4.^{4,5} All the reactions underwent smooth conversion to afford the above-said heterocycles in 64%-82% yields.



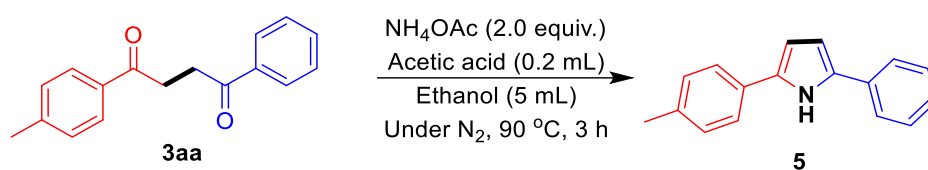
Scheme S4. Synthetic applications

12. Experimental procedure (B) for the synthesis of 2-Phenyl-5-(*p*-tolyl)furan (**4**)⁴



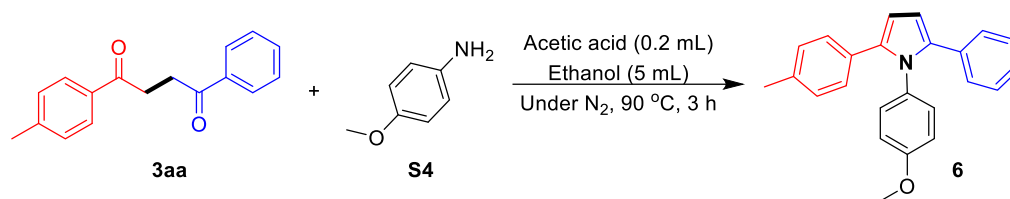
The title compound was synthesized following the modified procedure: The 2.5 mL of conc. HCl was added drop wise to a stirred suspension of **3aa** (0.5 mmol, 127 mg) in 2.5 mL of Ac₂O at 0 °C under an argon atmosphere, and the solution was allowed to stir at 60 °C for 6 h. The reaction was then neutralized with sat. NaHCO₃ and was extracted with ethyl acetate (3 x 10 mL). The combined ethyl acetate layer was given a water wash, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to get a crude compound. The obtained crude was purified using column chromatography by eluting hexane to afford the desired product in 80% yield.

13. Experimental procedure (C) for the synthesis of 2-Phenyl-5-(*p*-tolyl)-1*H*-pyrrole (**5**)⁵



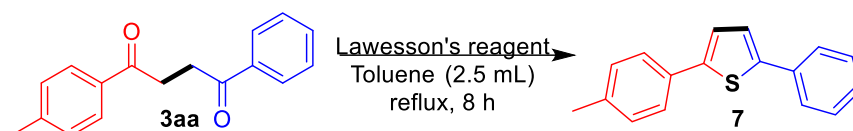
The title compound was synthesized following the modified procedure: The compound **3aa** (0.5 mmol, 127 mg) was dissolved in 5 mL of ethanol and then added NH₄OAc (1 mmol, 77 mg) followed by 0.2 mL acetic acid drop wise under nitrogen atmosphere. After adding acetic acid, the solution was stirred at 90 °C for 3 h. The reaction was neutralized with sat. NaHCO₃ and extracted with ethyl acetate (3 x 10 mL). The combined ethyl acetate layer was given a water wash, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to get a crude compound. The obtained crude was purified using column chromatography by eluting hexane to afford the desired product **5** in 80% yield.

14. Experimental procedure (D) for the synthesis of 1-(4-Methoxyphenyl)-2-phenyl-5-(*p*-tolyl)-1*H*-pyrrole (6)⁵



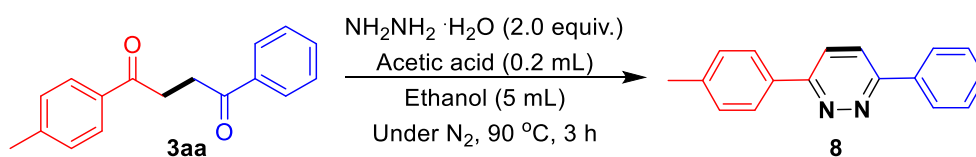
The title compound was synthesized following the modified procedure: The compound **3aa** (0.5 mmol, 127 mg) dissolved in 5 mL of ethanol and then added 4-methoxyaniline (**S4**, 1.0 mmol, 123 mg) followed by 0.2 mL acetic acid drop wise under nitrogen atmosphere. After adding acetic acid, the solution was stirred at 90 °C for 3 h. The reaction was neutralized with sat. NaHCO₃ was then extracted with ethyl acetate (3 x 10 mL). The combined ethyl acetate layer was given a water wash, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to get a crude compound. The obtained crude was purified using column chromatography by eluting hexane to afford the desired product **6** in 75% yield.

15. Experimental procedure (E) for the synthesis of 2-Phenyl-5-(*p*-tolyl)thiophenes (7)⁴



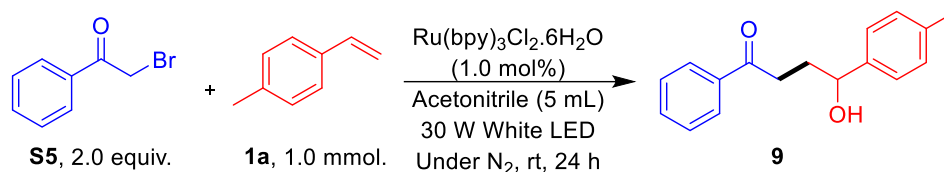
A mixture of **3aa** (0.5 mmol, 127 mg) and Lawesson's reagent (250 mg, 1.2 equiv) was heated under reflux in toluene (2.5 mL) for 6 h. The solution was allowed to cool to room temperature and then extracted with ethyl acetate (3 x 10 mL). The combined ethyl acetate layer was given a water wash, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to get a crude compound. The crude obtained was purified using column chromatography by eluting hexane to afford the desired product **7** of 82% yield.

16. Experimental procedure (F) for the synthesis of 3-Phenyl-6-(*p*-tolyl)pyridazines (8)⁵



The title compound was synthesized following the modified procedure: The compound **3aa** (0.5 mmol, 127 mg) dissolved in 5 mL of ethanol and then added NH₂NH₂·H₂O (1.0 mmol, 123 mg) followed by 0.2 mL acetic acid drop wise under nitrogen atmosphere. After adding acetic acid, the solution was stirred at 90 °C for 3 h. The reaction was neutralized with sat. NaHCO₃ was then extracted with ethyl acetate (3 x 10 mL). The combined ethyl acetate layer was given a water wash, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to get a crude compound. The obtained crude was purified using column chromatography by eluting hexane to afford the desired product **8** in 64% yield.

17. Experimental procedure (G) for the synthesis of 4-hydroxy-1-phenyl-4-(*p*-tolyl)butan-1-one (**9**)⁶



α -Bromoacetophenone (**S5**, 2.0 equiv.), *p*-methylstyrene (**1a**, 1.0 mmol.), Ru(bpy)₃Cl₂·6H₂O (1.0 mol%) and NaHCO₃ (1.0 equiv.) are weighed under a nitrogen atmosphere in a 25 mL glass vial. To this reaction mixture, acetonitrile (3.0 mL) and water (1.0 equiv.) were added and stirred for 24 h at room temperature under a 30 W white LED lamp. After the reaction is completed, the reaction mixture was diluted with 10 mL of water. The aqueous layer was extracted with ethyl acetate (3 x 10 mL). The combined ethyl acetate layer was dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to get a crude compound. The obtained crude was purified using column chromatography by eluting hexane/ethyl acetate to afford the desired product **9**.

18. Electrochemical measurements of 1-phenylbutane-1,3-dione (**2a**)

Samples for electrochemical measurements were prepared with 0.1 M of *tetra-n*-butylammonium hexafluorophosphate solution in MeOH:H₂O (3:1) and 0.05 M of 1-phenylbutane-1,3-dione (**2a**). Cyclic voltammetry measurements were done with a computer-controlled potentiostat OriogaLys, SAS, Model OGF 500. Cyclic Voltammetry was recorded using an undivided cell equipped with glassy carbon as the working electrode, platinum wire as the counter electrode and Ag/AgCl as a reference electrode. The ferrocene/ferrocenium couple (Fc/Fc⁺) was also measured in the same electrochemical system, and the electrode potential was reported as values referring to the system's apparent standard potential. Reductions were measured by scanning potentials in the negative direction and oxidations in the positive direction; the glassy carbon electrode was polished between scans. A scan rate was used, 100 mV/s.

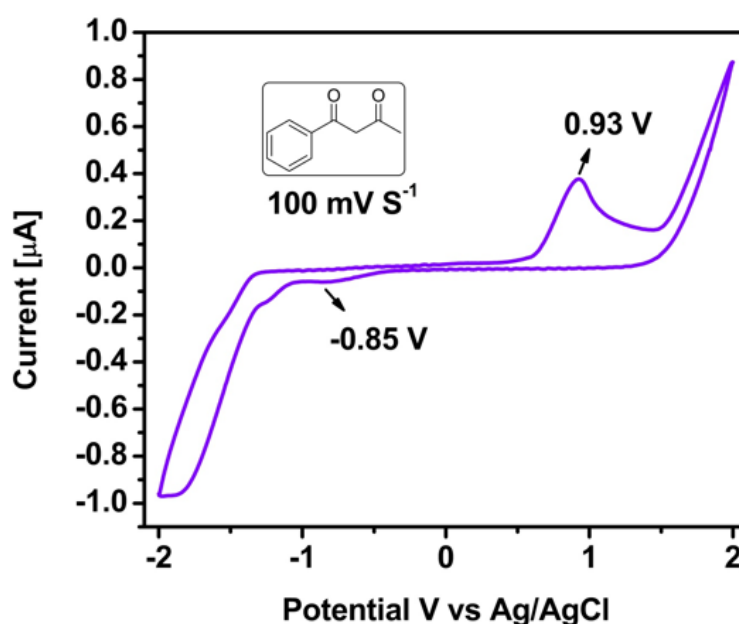
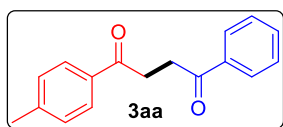


Figure S3. Cyclic Voltammogram of 1-phenylbutane-1,3-dione (**2a**).

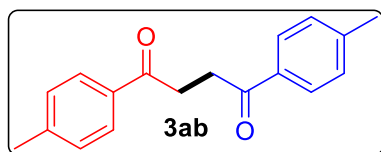
19. Spectral Characterization

1-Phenyl-4-(*p*-tolyl)butane-1,4-dione (3aa).⁷ The title compound was prepared according to the general



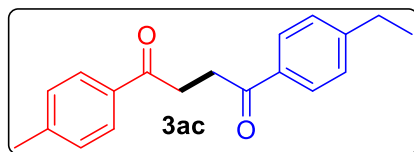
experimental procedure A on a 0.5 mmol for 24 h and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a white solid (MeOH:H₂O – 113 mg, 90% & CH₃CN:H₂O – 106 mg, 84%); mp 114-116 °C (lit. 116-118 °C); ¹H NMR (CDCl₃, 500 MHz): δ 8.06 – 8.03 (m, 2H), 7.94 (d, *J* = 8.0 Hz, 2H), 7.60 – 7.56 (m, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.28 (d, *J* = 8.3 Hz, 2H), 3.45 (s, 4H), 2.42 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 198.7, 198.2, 143.9, 136.8, 134.3, 133.1, 129.3, 128.6, 128.2, 128.1, 32.5, 32.4, 21.6; HRMS (ESI) calculated for C₁₇H₁₇O₂ [M+H]⁺: 253.1244 found 253.1244.

1,4-Di-*p*-tolylbutane-1,4-dione (3ab).⁸ The title compound was prepared according to the general



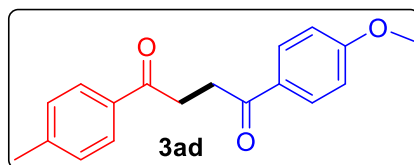
experimental procedure A on a 0.5 mmol for 30 h and the product was isolated by column chromatography (Ethyl acetate/Hexane: 2%) to afford a white solid (MeOH:H₂O – 118 mg, 89% & CH₃CN:H₂O – 104 mg, 78%); mp 158-159 °C (lit. 159-160 °C); ¹H NMR (CDCl₃, 500 MHz): δ 7.94 (d, *J* = 8.2 Hz, 4H), 7.29 – 7.25 (m, 4H), 3.43 (s, 4H), 2.42 (s, 6H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): 198.7, 144.1, 134.5, 129.5, 128.4, 32.7, 21.8; HRMS (ESI) calculated for C₁₈H₁₉O₂ [M+H]⁺: 267.1497 found 267.1497.

1-(4-Ethylphenyl)-4-(*p*-tolyl)butane-1,4-dione (3ac). The title compound was prepared according to the



general experimental procedure A on a 0.5 mmol for 36 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 2%) to afford a white solid (MeOH:H₂O – 131 mg, 97% & CH₃CN:H₂O – 119 mg, 85%); mp 117-118 °C; ¹H NMR (CDCl₃, 500 MHz): δ 7.96 (d, *J* = 8.0 Hz, 2H), 7.94 (d, *J* = 8.0 Hz, 2H), 7.3 (d, *J* = 8.0 Hz, 2H), 3.43 (s, 4H), 2.72 (q, *J* = 7.5 Hz, 2H), 2.42 (s, 3H), 1.27 (t, *J* = 7.5 Hz, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.7, 150.3, 144.1, 134.8, 134.5, 129.5, 128.5, 128.4, 128.3, 32.7, 29.1, 21.8, 15.4; HRMS (ESI) calculated for C₁₉H₂₀O₂Na [M+Na]⁺: 303.1361 found 303.1389.

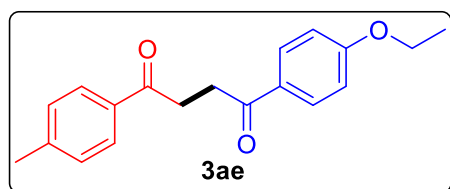
1-(4-Methoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (3ad).⁸ The title compound was prepared according to the



general experimental procedure A on a 0.5 mmol for 48 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 5%) to afford a pale white solid (MeOH:H₂O – 74 mg, 53% & CH₃CN:H₂O – 72 mg, 51%); mp 109-111 °C (lit. 108-109 °C); ¹H NMR (CDCl₃, 500 MHz):

δ 8.02 (d, *J* = 8.5 Hz, 2H), 7.94 (d, *J* = 7.8 Hz, 2H), 7.27 (d, *J* = 8.9 Hz, 2H), 6.95 (d, *J* = 8.5 Hz, 2H), 3.88 (s, 3H), 3.42 (s, 4H), 2.42 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.7, 197.5, 163.7, 144.0, 134.5, 130.5, 130.1, 129.4, 128.4, 113.9, 55.6, 32.7, 32.4, 21.8; HRMS (ESI) calculated for C₁₈H₁₉O₃ [M+H]⁺: 283.1335 found 283.1335.

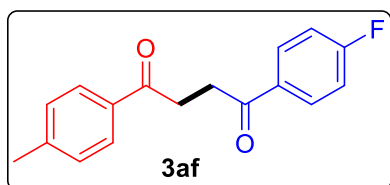
1-(4-Ethoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (3ae). The title compound was prepared according to the



general experimental procedure A on a 0.5 mmol for 48 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 5%) to afford a white solid (MeOH:H₂O – 55 mg, 37% & CH₃CN:H₂O – 58 mg, 39%); mp 121-122 °C; ¹H NMR (CDCl₃, 500

MHz): δ 8.01 (d, *J* = 8.8 Hz, 2H), 7.94 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 4.11 (q, *J* = 7.0 Hz, 2H), 3.46 – 3.38 (m, 4H), 2.42 (s, 3H), 1.45 (t, *J* = 7.0 Hz, 3H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 198.5, 197.3, 162.9, 143.8, 134.3, 130.3, 129.7, 129.2, 128.2, 114.1, 63.7, 32.5, 32.2, 21.6, 14.7; HRMS (ESI) calculated for C₁₉H₂₀O₃Na [M+Na]⁺: 319.1310 found 319.1302.

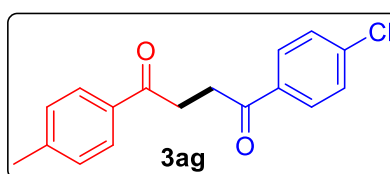
1-(4-Fluorophenyl)-4-(*p*-tolyl)butane-1,4-dione (3af).⁹ The title compound was prepared according to the



general experimental procedure A on a 0.5 mmol for 36 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a white solid (MeOH:H₂O – 75 mg, 56% & CH₃CN:H₂O – 68 mg, 50%); mp 120-122 °C; ¹H NMR (CDCl₃, 500 MHz): δ 8.10 – 8.05 (m, 2H),

7.94 (d, *J* = 8.1 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 7.18 – 7.13 (m, 2H), 3.47 – 3.39 (m, 4H), 2.42 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 198.2, 197.2, 165.7 (*J*_{C-F} = 253 Hz), 144.0, 134.2, 133.2, 130.7 (*J*_{C-F} = 9.25 Hz), 129.3, 128.2, 115.6 (*J*_{C-F} = 21.75 Hz), 32.4, 21.6; HRMS (ESI) calculated for C₁₇H₁₆FO₂ [M+H]⁺: 271.1124 found 271.1124.

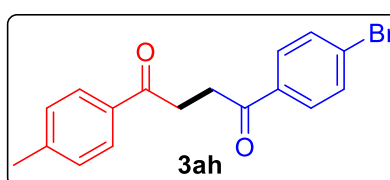
1-(4-Chlorophenyl)-4-(*p*-tolyl)butane-1,4-dione (3ag).¹⁰ The title compound was prepared according to the



general experimental procedure A on a 0.5 mmol for 36 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a pale yellow solid (MeOH:H₂O – 88 mg, 62%); mp 147-150 °C; ¹H

NMR (CDCl₃, 500 MHz): δ 7.99 – 7.96 (m, 2H), 7.93 (d, *J* = 8.0 Hz, 2H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 3.43 – 3.40 (m, 4H), 2.42 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 198.1, 197.6, 144.0, 139.5, 135.1, 134.1, 129.5, 129.3, 128.9, 128.2, 32.5, 32.4, 21.6; HRMS (ESI) calculated for C₁₇H₁₆ClO₂ [M+H]⁺: 287.0857 found 287.0857.

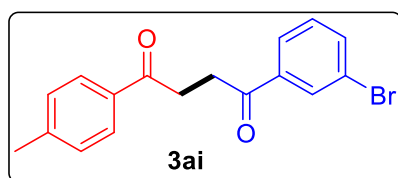
1-(4-Bromophenyl)-4-(*p*-tolyl)butane-1,4-dione (3ah).⁹ The title compound was prepared according to the



general experimental procedure A on a 0.5 mmol for 36 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a white crystalline solid (MeOH:H₂O – 82 mg, 49% & CH₃CN:H₂O – 80 mg, 48%); mp 165-167 °C; ¹H NMR (CDCl₃, 500 MHz): δ 7.93 (d, *J*

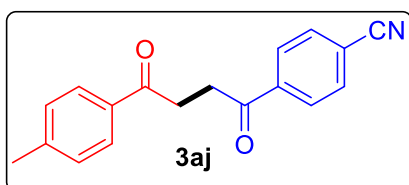
= 8.2 Hz, 2H), 7.91 (d, *J* = 8.3 Hz, 2H), 7.62 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.3 Hz, 2H), 3.46 – 3.39 (m, 4H), 2.42 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): ¹³C NMR (101 MHz, CDCl₃) δ 196.5, 196.2, 142.4, 133.9, 132.5, 130.3, 128.0, 127.7, 126.6, 30.9, 30.8, 20.0; HRMS (ESI) calculated for C₁₇H₁₆BrO₂ [M+H]⁺: 333.0298 found 333.0298.

1-(3-Bromophenyl)-4-(*p*-tolyl)butane-1,4-dione (3ai). The title compound was prepared according to the



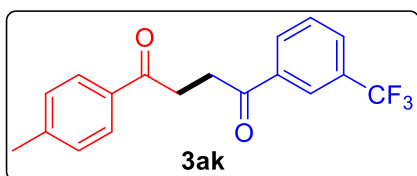
general experimental procedure A on a 0.5 mmol for 36 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a white crystalline solid (MeOH:H₂O – 116 mg, 70% & CH₃CN:H₂O – 118 mg, 71%); mp 108-110 °C; ¹H NMR (CDCl₃, 500 MHz): δ 8.16 (t, *J* = 1.7 Hz, 1H), 7.96 (d, *J* = 7.8 Hz, 1H), 7.93 (d, *J* = 8.2 Hz, 2H), 7.70 (d, *J* = 7.9 Hz, 1H), 7.39 – 7.34 (m, 1H), 7.28 (d, *J* = 8.1 Hz, 2H), 3.47 – 3.39 (m, 4H), 2.42 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.3, 197.7, 144.3, 138.7, 136.2, 134.3, 131.4, 130.4, 129.5, 128.4, 126.9, 123.2, 32.8, 32.6, 21.9; HRMS (ESI) calculated for C₁₇H₁₅BrO₂Na [M+Na]⁺: 353.0153 found 353.0183.

4-(4-Oxo-4-(*p*-tolyl)butanoyl)benzotrile (3aj).¹¹ The title compound was prepared according to the general



experimental procedure A on a 0.5 mmol for 48 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 5%) to afford a white solid (MeOH:H₂O – 50 mg, 39% & CH₃CN:H₂O – 54 mg, 36%); mp 132-134 °C (lit. 134-136 °C); ¹H NMR (CDCl₃, 500 MHz): δ 8.13 (d, *J* = 8.2 Hz, 2H), 7.93 (d, *J* = 7.9 Hz, 2H), 7.80 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 7.7 Hz, 2H), 3.48 – 3.42 (m, 4H), 2.43 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 197.8, 197.6, 144.2, 139.8, 133.9, 132.5, 129.3, 128.5, 128.2, 118.0, 116.3, 32.7, 32.4, 21.7; HRMS (ESI) calculated for C₁₈H₁₆NO₂ [M+H]⁺: 278.1178 found 278.1178.

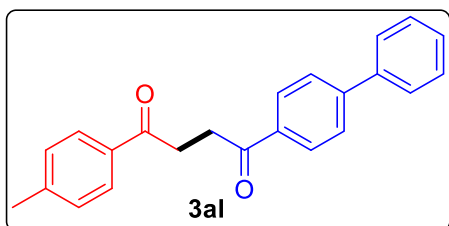
1-(*p*-Tolyl)-4-(3-(trifluoromethyl)phenyl)butane-1,4-dione (3ak). The title compound was prepared



according to the general experimental procedure A on a 0.5 mmol for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 4%) to afford a white solid (MeOH:H₂O – 52 mg, 33% & CH₃CN:H₂O – 88 mg, 55%); mp 94-96 °C; ¹H NMR (CDCl₃, 500 MHz):

δ 8.30 (s, 1H), 8.23 (d, *J* = 7.8 Hz, 1H), 7.94 (d, *J* = 8.2 Hz, 2H), 7.84 (d, *J* = 7.8 Hz, 1H), 7.64 (t, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 3.52 – 3.44 (m, 4H), 2.43 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.0, 197.5, 144.1, 137.3, 134.1, 131.3, 131.0, 129.5, 129.3, 128.2, 125.0, 122.3, 32.6, 32.4, 21.6; HRMS (ESI) calculated for C₁₈H₁₆F₃O₂ [M+H]⁺: 321.1102 found 321.1108.

1-([1,1'-biphenyl]-4-yl)-4-(*p*-tolyl)butane-1,4-dione (3al). The title compound was prepared according to

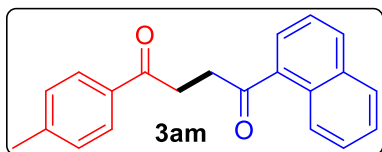


the general experimental procedure A on a 0.5 mmol for 48 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 6%) to afford a white solid (MeOH:H₂O – 91 mg, 55% & CH₃CN:H₂O – 77 mg, 47%); mp 179-181 °C; ¹H NMR (CDCl₃, 500 MHz): δ 8.12 (d, *J* = 8.4 Hz, 2H), 7.95 (d, *J* = 8.2 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.66

– 7.63 (m, 2H), 7.48 (t, *J* = 7.5 Hz, 2H), 7.40 (t, *J* = 7.3 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 3.48 (t, *J* = 3.7 Hz, 4H), 2.43 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 198.4, 198.4, 145.7, 143.9, 139.9, 135.5, 134.3, 129.3,

128.9, 128.7, 128.2, 127.3, 127.2, 32.6, 32.5, 21.7; HRMS (ESI) calculated for $C_{23}H_{20}O_2Na$ $[M+Na]^+$: 351.1361 found 351.1375.

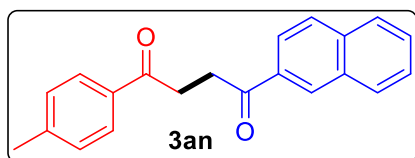
1-(Naphthalen-1-yl)-4-(*p*-tolyl)butane-1,4-dione (3am).⁹ The title compound was prepared according to the



general experimental procedure A on a 0.5 mmol for 48 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 5%) to afford a white solid (MeOH:H₂O – 78 mg, 52% & CH₃CN:H₂O – 74 mg, 49%); mp

98-100 °C; ¹H NMR (CDCl₃, 500 MHz): δ 8.59 (d, *J* = 8.6 Hz, 1H), 8.05 (d, *J* = 7.2 Hz, 1H), 7.99 (d, *J* = 8.2 Hz, 1H), 7.96 (d, *J* = 8.1 Hz, 2H), 7.87 (d, *J* = 8.1 Hz, 1H), 7.57 (t, *J* = 7.0 Hz, 1H), 7.52 (t, *J* = 7.0 Hz, 2H), 7.28 (d, *J* = 7.8 Hz, 2H), 3.54 – 3.48 (m, 4H), 2.42 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 203.0, 198.3, 144.0, 136.0, 134.3, 133.9, 132.6, 130.1, 129.3, 128.4, 128.3, 127.9, 127.7, 126.4, 125.9, 124.5, 77.4, 77.2, 76.9, 36.0, 32.98, 21.7; HRMS (ESI) calculated for $C_{21}H_{22}NO_2$ $[M+NH_4]^+$: 320.1636 found 320.1636.

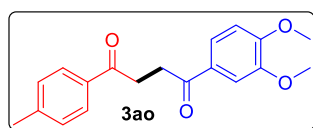
1-(Naphthalen-2-yl)-4-(*p*-tolyl)butane-1,4-dione (3an). The title compound was prepared according to the



general experimental procedure A on a 0.5 mmol for 48 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 6%) to afford a white solid (MeOH:H₂O – 101 mg, 67% & CH₃CN:H₂O – 63 mg, 42%); mp 155-157 °C; ¹H NMR (CDCl₃, 500 MHz): δ 8.59 (s, 1H), 8.08

(dd, *J* = 8.6, 1.4 Hz, 1H), 7.99 – 7.95 (m, 3H), 7.90 (d, *J* = 8.5 Hz, 1H), 7.88 (d, *J* = 8.5 Hz, 1H), 7.60 (t, *J* = 7.0 Hz, 1H), 7.55 (t, *J* = 7.0 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 3.59 (t, *J* = 6.4 Hz, 2H), 3.50 (t, *J* = 6.4 Hz, 2H), 2.42 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 199.0, 198.6, 144.2, 135.8, 134.5, 134.3, 132.7, 130.1, 129.8, 129.5, 128.6, 128.5, 128.0, 127.0, 124.1, 32.9, 32.8, 21.9; HRMS (ESI) calculated for $C_{21}H_{18}O_2Na$ $[M+Na]^+$: 325.1205 found 325.1222.

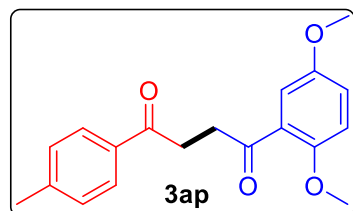
1-(3,4-Dimethoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (3ao).¹² The title compound was prepared according



to the general experimental procedure A on a 0.5 mmol for 48 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 8%) to afford a pale white solid (MeOH:H₂O – 51 mg, 33% & CH₃CN:H₂O – 56 mg, 36%); mp

114-118 °C; ¹H NMR (CDCl₃, 500 MHz): δ 7.95 (d, *J* = 8.1 Hz, 2H), 7.71 (dd, *J* = 8.4, 1.9 Hz, 1H), 7.57 (d, *J* = 1.8 Hz, 1H), 7.27 (d, *J* = 9.0 Hz, 2H), 6.92 (d, *J* = 8.4 Hz, 1H), 3.96 (s, 3H), 3.94 (s, 3H), 3.43 (s, 4H), 2.42 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.8, 197.6, 153.5, 149.1, 144.1, 134.5, 130.2, 129.5, 128.4, 123.0, 110.3, 110.2, 56.3, 56.2, 32.8, 32.4, 21.9; HRMS (ESI) calculated for $C_{19}H_{21}O_4$ $[M+H]^+$: 313.1434 found 313.1434.

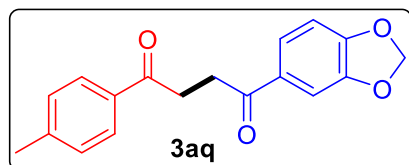
1-(2,5-dimethoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (3ap). The title compound was prepared according to



the general experimental procedure A on a 0.5 mmol for 48 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 8%) to afford a white solid (MeOH:H₂O – 53 mg, 34% & CH₃CN:H₂O – 68 mg, 44%); mp 74-76 °C; ¹H NMR (CDCl₃, 500 MHz): δ 7.94 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* =

3.1 Hz, 1H), 7.27 (d, $J = 7.5$ Hz, 2H), 7.04 (dd, $J = 8.9, 3.1$ Hz, 1H), 6.93 (d, $J = 9.0$ Hz, 1H), 3.89 (s, 3H), 3.79 (s, 3H), 3.46 (t, $J = 6.2$ Hz, 2H), 3.38 (t, $J = 6.2$ Hz, 2H), 2.42 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 200.2, 198.7, 153.4, 153.4, 143.7, 134.4, 129.2, 128.2, 127.9, 120.3, 113.9, 113.2, 56.1, 55.8, 38.0, 32.8, 21.6; HRMS (ESI) calculated for $\text{C}_{19}\text{H}_{20}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$; 335.1259 found 335.1256; HRMS (ESI) calculated for $\text{C}_{19}\text{H}_{21}\text{O}_4$ $[\text{M}+\text{H}]^+$: 313.1434 found 313.1434.

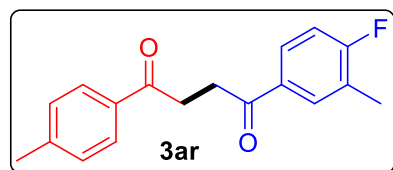
1-(Benzo[d][1,3]dioxol-5-yl)-4-(*p*-tolyl)butane-1,4-dione (3aq). The title compound was prepared according



to the general experimental procedure A on a 0.5 mmol for 40 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 6%) to afford a pale white solid (MeOH:H₂O – 104 mg, 70% & CH₃CN:H₂O – 90 mg, 61%); mp 153-155 °C; ^1H NMR (CDCl_3 , 500 MHz):

δ 7.93 (d, $J = 8.2$ Hz, 2H), 7.66 (dd, $J = 8.2, 1.6$ Hz, 1H), 7.48 (d, $J = 1.6$ Hz, 1H), 7.31 – 7.26 (m, 2H), 6.87 (d, $J = 8.2$ Hz, 1H), 6.05 (s, 2H), 3.43 – 3.35 (m, 4H), 2.42 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 198.4, 196.8, 151.7, 148.1, 143.9, 134.2, 131.6, 129.2, 128.2, 124.4, 107.94, 107.91, 101.8, 32.6, 32.3, 21.6; HRMS (ESI) calculated for $\text{C}_{18}\text{H}_{16}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$: 319.0946 found 319.0938.

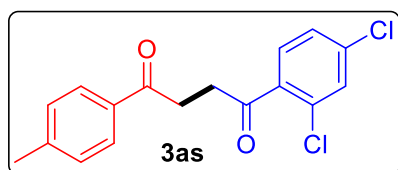
1-(4-Fluoro-3-methylphenyl)-4-(*p*-tolyl)butane-1,4-dione (3ar). The title compound was prepared



according to the general experimental procedure A on a 0.5 mmol for 48 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 5%) to afford a white solid (MeOH:H₂O – 69 mg, 49% & CH₃CN:H₂O – 55 mg, 39%); mp 100-102 °C; ^1H NMR (CDCl_3 , 500 MHz):

δ 7.99 – 7.83 (m, 4H), 7.27 (d, $J = 8.5$ Hz, 2H), 7.08 (t, $J = 8.8$ Hz, 1H), 3.46 – 3.38 (m, 4H), 2.42 (s, 3H), 2.34 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 101 MHz): δ 198.3, 197.5, 165.6, 163.1, 143.9, 134.2, 132.9, 131.9 ($J_{\text{CF}} = 6.5$ Hz), 128.7 ($J_{\text{CF}} = 105.2$ Hz), 128.04 ($J_{\text{CF}} = 9.2$ Hz), 125.3 ($J_{\text{CF}} = 18$ Hz), 115.2 ($J_{\text{CF}} = 22.9$ Hz), 32.5, 32.4, 21.6, 14.5; HRMS (ESI) calculated for $\text{C}_{18}\text{H}_{17}\text{FO}_2\text{Na}$ $[\text{M}+\text{Na}]^+$: 307.1110 found 307.1188.

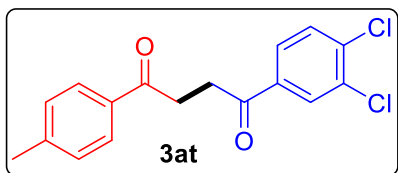
1-(2,4-Dichlorophenyl)-4-(*p*-tolyl)butane-1,4-dione (3as). The title compound was prepared according to



the general experimental procedure A on a 0.5 mmol continued for 36 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a white solid (MeOH:H₂O – 74 mg, 46% & CH₃CN:H₂O – 71 mg, 44%); mp 51-53 °C; ^1H NMR (CDCl_3 , 500 MHz): δ 7.90 (d, $J = 8.2$ Hz,

2H), 7.64 (d, $J = 8.3$ Hz, 1H), 7.44 (d, $J = 1.9$ Hz, 1H), 7.33 (dd, $J = 8.3, 2.0$ Hz, 1H), 7.26 (d, $J = 8.0$ Hz, 2H), 3.45 (dd, $J = 7.1, 5.4$ Hz, 2H), 3.32 (dd, $J = 6.7, 5.4$ Hz, 2H), 2.41 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz): δ 200.6, 197.7, 144.1, 137.4, 137.2, 133.9, 131.9, 130.6, 130.3, 129.3, 128.2, 127.3, 36.7, 33.1, 21.6; HRMS (ESI) calculated for $\text{C}_{17}\text{H}_{15}\text{Cl}_2\text{O}_2$ $[\text{M}+\text{H}]^+$: 321.0449 found 321.0451.

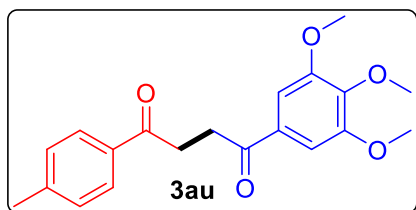
1-(3,4-Dichlorophenyl)-4-(*p*-tolyl)butane-1,4-dione (3at). The title compound was prepared according to



the general experimental procedure A on a 0.5 mmol for 48 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 5%) to afford a pale white solid (MeOH:H₂O – 55 mg, 34% & CH₃CN:H₂O – 75 mg, 47%); mp 115-119 °C; ¹H NMR (CDCl₃, 500 MHz): δ 8.11 (s, 1H),

7.92 (d, *J* = 7.9 Hz, 2H), 7.86 (d, *J* = 8.3 Hz, 1H), 7.57 (d, *J* = 8.3 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 2H), 3.45 (t, *J* = 5.8 Hz, 2H), 3.38 (d, *J* = 6.1 Hz, 2H), 2.42 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.0, 196.8, 144.3, 137.8, 136.6, 134.3, 133.5, 130.9, 130.3, 129.5, 128.4, 127.3, 32.7, 32.6, 21.8; HRMS (ESI) calculated for C₁₇H₁₅Cl₂O₂ [M+H]⁺: 321.0449 found 321.0446.

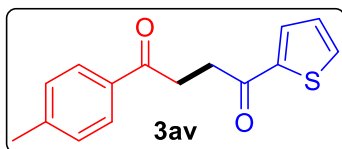
1-(*p*-Tolyl)-4-(3,4,5-trimethoxyphenyl)butane-1,4-dione (3au). The title compound was prepared according



to the general experimental procedure A on a 0.5 mmol for 36 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 10%) to afford a white solid (MeOH:H₂O – 75 mg, 44% & CH₃CN:H₂O – 80 mg, 47%); mp 108-111 °C; ¹H NMR (CDCl₃, 500 MHz): δ 7.95 (d, *J* = 8.0 Hz, 2H), 7.31 – 7.27 (m, 4H), 3.92 (s, 9H), 3.44 (s, 4H), 2.43 (s, 3H).

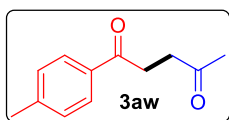
¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.6, 197.8, 153.3, 144.2, 142.8, 134.4, 132.3, 129.5, 128.4, 105.8, 61.1, 56.5, 32.8, 32.6, 21.9; HRMS (ESI) calculated for C₁₉H₂₁O₅ [M+H]⁺: 343.1546 found 343.1580.

1-(Thiophen-2-yl)-4-(*p*-tolyl)butane-1,4-dione (3av).¹⁵ The title compound was prepared according to the



general experimental procedure A on a 0.5 mmol for 48 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 5%) to afford a pale yellow solid (MeOH:H₂O – 88 mg, 68%); mp 120-122 °C (lit. 120 °C); ¹H NMR (CDCl₃, 500 MHz): δ 7.92 (d, *J* = 8.2 Hz, 2H), 7.85 – 7.81 (m, 1H), 7.64 (dd, *J* = 4.9, 1.0 Hz, 1H), 7.27 (d, *J* = 8.3 Hz, 2H), 7.16 (t, *J* = 5.0 Hz, 1H), 3.45 – 3.37 (m, 4H), 2.42 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.3, 192.0, 144.2, 134.3, 133.7, 132.2, 129.5, 128.4, 128.3, 33.4, 32.7, 21.9; HRMS (ESI) calculated for C₁₅H₁₅O₂S [M+H]⁺: 259.0808 found 259.0808.

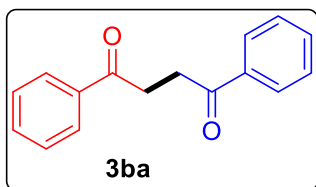
1-(*p*-Tolyl)pentane-1,4-dione (3aw).⁸ The title compound was prepared according to the general



experimental procedure A on a 0.5 mmol for 40 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 2%) to afford yellow oil (MeOH:H₂O – 51 mg, 54% & CH₃CN:H₂O – 47 mg, 49%); ¹H NMR (CDCl₃, 500 MHz): δ 7.88 (d, *J* = 7.7 Hz,

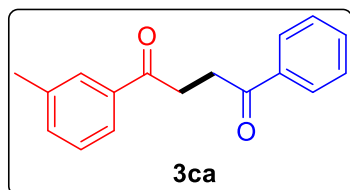
2H), 7.26 (d, *J* = 7.0 Hz, 2H), 3.26 (t, *J* = 5.5 Hz, 2H), 2.88 (t, *J* = 6.3 Hz, 2H), 2.41 (s, 3H), 2.26 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 207.6, 198.3, 144.0, 134.3, 129.3, 128.3, 37.2, 32.4, 30.2, 21.7; HRMS (ESI) calculated for C₁₂H₁₅O₂ [M+H]⁺: 191.1067 found 191.1071.

1,4-Diphenylbutane-1,4-dione (3ba).⁷ The title compound was prepared according to the general



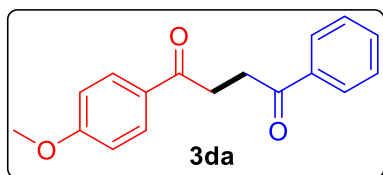
experimental procedure A on a 0.5 mmol for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 2%) to white solid (MeOH:H₂O – 71 mg, 76%); mp 144-145 °C (lit. 144-145 °C); ¹H NMR (CDCl₃, 500 MHz): δ 8.06 – 8.03 (m, 4H), 7.58 (tt, *J* = 6.8, 1.2 Hz, 2H), 7.48 – 7.46 (m, 4H), 3.46 (s, 4H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 198.7, 136.7, 133.1, 128.6, 128.1, 32.5; HRMS (ESI) calculated for C₁₂H₁₅O₃ [M+H]⁺: 229.0835 found 229.0836.

1-Phenyl-4-(*m*-tolyl)butane-1,4-dione (3ca).¹⁴ The title compound was prepared according to the general



experimental procedure A on a 0.5 mmol continued for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a white solid (MeOH:H₂O – 71 mg, 56% & CH₃CN:H₂O – 89 mg, 71%); mp 80-82 °C (No report); ¹H NMR (CDCl₃, 500 MHz): δ 8.05 (d, *J* = 7.4 Hz, 2H),

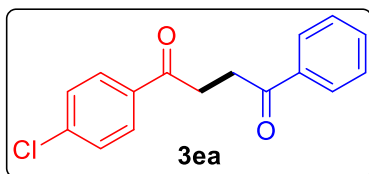
7.86-7.83 (m, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.41 – 7.35 (m, 2H), 3.46 (s, 4H), 2.43 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 198.9, 198.7, 138.3, 136.7, 133.9, 133.1, 128.6, 128.6, 128.5, 128.1, 125.3, 32.6, 21.4; HRMS (ESI) calculated for C₁₇H₁₇O₂ [M+H]⁺: 253.1239 found 253.1239.



1-(4-Methoxyphenyl)-4-phenylbutane-1,4-dione (3da).⁷ The title compound was prepared according to the general experimental procedure A on a 0.5 mmol for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 5%) to afford a yellow solid

(MeOH:H₂O – 90 mg, 67% & CH₃CN:H₂O – 95 mg, 71%); mp 102-104 °C (lit. 99-100 °C); ¹H NMR (CDCl₃, 500 MHz): δ 8.07 – 8.00 (m, 4H), 7.60 – 7.54 (m, 1H), 7.47 (t, *J* = 8.0 Hz, 2H), 6.95 (d, *J* = 9.0 Hz, 2H), 3.88 (s, 3H), 3.47 – 3.40 (m, 4H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 198.9, 197.2, 163.5, 136.8, 133.1, 130.4, 129.8, 128.6, 128.1, 113.7, 55.4, 32.6, 32.2; HRMS (ESI) calculated for C₁₇H₁₇O₃ [M+H]⁺: 269.1179 found 269.1179.

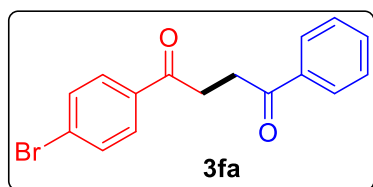
1-(4-Chlorophenyl)-4-phenylbutane-1,4-dione (3ea).⁷ The title compound was prepared according to the



general experimental procedure A on a 0.5 mmol for 36 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a white solid (MeOH:H₂O – 78 mg, 58% & CH₃CN:H₂O – 65 mg, 48%); mp 114-116 °C (lit. 114-115 °C); ¹H NMR (CDCl₃, 500 MHz): δ 8.06 – 8.03 (m,

2H), 7.98 (d, *J* = 8.6 Hz, 1H), 7.60 – 7.57 (m, 1H), 7.53 – 7.43 (m, 4H), 3.51 – 3.40 (m, 4H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.9, 198.7, 197.7, 139.8, 136.8, 135.3, 133.4, 133.4, 129.7, 129.1, 128.8, 128.3, 32.7; HRMS (ESI) calculated for C₁₆H₁₄ClO₂ [M+H]⁺: 273.0657 found 273.657.

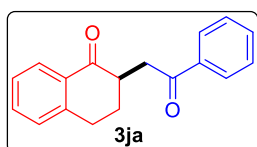
1-(4-Bromophenyl)-4-phenylbutane-1,4-dione (3fa).¹⁴ The title compound was prepared according to the



general experimental procedure A on a 0.5 mmol for 36 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a pale white solid (MeOH:H₂O – 119 mg, 75% & CH₃CN:H₂O – 95 mg, 60%); mp 100-103 °C; ¹H NMR (CDCl₃, 500 MHz): δ 8.03 (d, *J* = 7.8 Hz, 2H), 7.91

(d, *J* = 8.2 Hz, 2H), 7.63 (d, *J* = 8.2 Hz, 2H), 7.59 (t, *J* = 7.2 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 3.44 (dq, *J* = 11.6, 5.9 Hz, 4H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.7, 197.9, 136.8, 135.7, 133.4, 132.1, 129.9, 128.8, 128.5, 128.3, 32.7; HRMS (ESI) calculated for C₁₆H₁₄BrO₂ [M+H]⁺: 319.0135 found 319.0135.

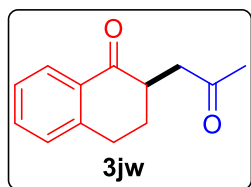
2-(2-Oxo-2-phenylethyl)-3,4-dihydronaphthalen-1(2H)-one (3ja).¹⁶ The title compound was prepared



according to the general experimental procedure A on a 0.5 mmol for 30 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 4%) to afford a white solid (CH₃CN:H₂O – 81 mg, 76%); mp 90-92 °C; ¹H NMR (CDCl₃, 500 MHz):

δ 8.04 (d, *J* = 7.7 Hz, 3H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.9 Hz, 3H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.28 – 7.24 (m, 1H), 3.86 (dd, *J* = 17.5, 4.7 Hz, 1H), 3.33 (ddt, *J* = 9.2, 7.1, 4.6 Hz, 1H), 3.23 – 3.15 (m, 1H), 3.02 – 2.95 (m, 2H), 2.30 (dt, *J* = 7.3, 4.4 Hz, 1H), 1.99 (qd, *J* = 13.0, 4.2 Hz, 1H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 199.1, 198.7, 144.2, 137.1, 133.5, 133.2, 132.4, 128.9, 128.7, 128.3, 127.6, 126.7, 44.3, 39.1, 29.7, 29.5; HRMS (ESI) calculated for C₁₈H₁₆O₂Na [M+Na]⁺: 287.1033 found 287.1033.

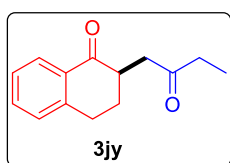
2-(2-Oxopropyl)-3,4-dihydronaphthalen-1(2H)-one (3jw).¹⁷ The title compound was prepared according to



the general experimental procedure A on a 0.5 mmol for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 4%) to afford a yellow oil (MeOH:H₂O – 45 mg, 44% & CH₃CN:H₂O – 34 mg, 33%); ¹H NMR (CDCl₃, 500

MHz): δ 8.00 (d, *J* = 7.8 Hz, 1H), 7.47 (t, *J* = 7.5 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.24 (d, *J* = 7.7 Hz, 1H), 3.23 – 3.10 (m, 3H), 3.00 – 2.92 (m, 1H), 2.45 (dt, *J* = 10.7, 5.5 Hz, 1H), 2.27 (s, 3H), 2.23 – 2.15 (m, 1H), 1.92 (qd, *J* = 13.0, 4.3 Hz, 1H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 207.4, 199.2, 144.3, 133.6, 132.4, 129.0, 127.6, 126.8, 44.4, 44.1, 30.7, 29.7, 29.6.

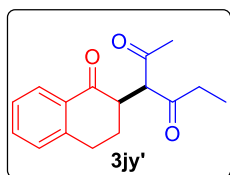
2-(2-Oxobutyl)-3,4-dihydronaphthalen-1(2H)-one (3jy).¹⁸ The title compound was prepared according to



the general experimental procedure A on a 0.5 mmol for 28 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 2-3%) to afford a yellow oil

(MeOH:H₂O – 18 mg, 17% & CH₃CN:H₂O – 27 mg, 25%); ¹H NMR (CDCl₃, 500 MHz): δ 8.00 (d, *J* = 7.8 Hz, 1H), 7.46 (t, *J* = 7.4 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 3.20 – 3.09 (m, 3H), 2.96 (m, 1H), 2.64 (m, 1H), 2.52 (m, 1H), 2.46 – 2.40 (m, 1H), 2.22 – 2.14 (m, 1H), 1.92 (qd, *J* = 12.9, 4.0 Hz, 1H), 1.11 (t, *J* = 7.3 Hz, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 210.0, 199.3, 144.2, 133.4, 132.3, 128.8, 127.5, 126.7, 44.3, 42.7, 36.6, 29.6, 29.5, 7.9.

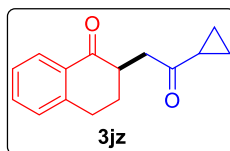
3-(1-Oxo-1,2,3,4-tetrahydronaphthalen-2-yl)hexane-2,4-dione (3jy'). The title compound was prepared



according to the general experimental procedure A on a 0.5 mmol continued for 28 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 6-8%) to afford a mixture of diastereomers as yellow oil (MeOH:H₂O – 27 mg, 21% & CH₃CN:H₂O – 32 mg, 25%); ¹H NMR (CDCl₃, 500 MHz): δ 7.98 – 7.92 (m, 2H), 7.52 –

7.46 (m, 2H), 7.30 (t, *J* = 7.6 Hz, 2H), 7.24 (d, *J* = 7.7 Hz, 2H), 4.23 (d, *J* = 8.8 Hz, 1H), 4.16 (d, *J* = 9.0 Hz, 1H), 3.64 – 3.56 (m, 2H), 3.20 – 3.10 (m, 2H), 3.02 – 2.95 (m, 2H), 2.84 (dq, *J* = 18.2, 7.2 Hz, 1H), 2.71 – 2.42 (m, 4H), 2.35 (s, 3H), 2.23 (s, 3H), 2.08 – 2.00 (m, 2H), 1.96 – 1.83 (m, 2H), 1.14 (t, *J* = 7.2 Hz, 3H), 1.07 (t, *J* = 7.2 Hz, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 206.1, 205.4, 203.7, 202.6, 198.0, 197.8, 143.8, 143.7, 133.9, 132.0, 128.8, 127.6, 126.9, 68.5, 68.2, 49.7, 49.6, 36.7, 36.6, 34.7, 34.2, 29.9, 29.8, 29.6, 29.5, 27.3, 22.4, 14.2, 7.9, 7.6. HRMS (ESI) calculated for C₁₆H₁₈O₃Na [M+Na]⁺: 281.1142 found 281.1142.

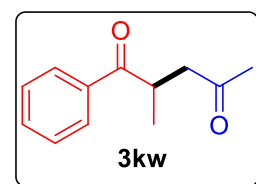
2-(2-Cyclopropyl-2-oxoethyl)-3,4-dihydronaphthalen-1(2H)-one (3jz). The title compound was prepared



according to the general experimental procedure A on a 0.5 mmol for 26 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 4%) to afford a yellow oil (MeOH:H₂O – 40 mg, 35% & CH₃CN:H₂O – 35 mg, 31%); ¹H NMR (CDCl₃, 500 MHz): δ 8.02 (d, *J* = 7.8 Hz, 1H), 7.46 (td, *J* = 7.5, 1.2 Hz, 1H), 7.30 (t, *J* = 7.6 Hz, 1H),

7.24 (d, *J* = 7.6 Hz, 1H), 3.37 (dd, *J* = 17.5, 5.0 Hz, 1H), 3.18 – 3.08 (m, 2H), 2.99 – 2.92 (m, 1H), 2.63 (dd, *J* = 17.5, 6.9 Hz, 1H), 2.23 – 2.17 (m, 1H), 2.07 – 1.99 (m, 1H), 1.91 (qd, *J* = 13.0, 4.3 Hz, 1H), 1.13 – 1.04 (m, 2H), 0.99 – 0.87 (m, 2H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 209.3, 199.1, 144.2, 133.4, 132.4, 128.8, 127.5, 126.7, 44.0, 43.6, 31.0, 29.5, 29.4, 21.1, 10.9. HRMS (ESI) calculated for C₁₅H₁₆O₂Na [M+Na]⁺: 251.1035 found 251.1035.

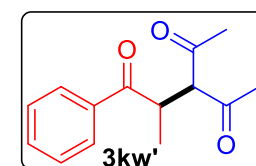
2-Methyl-1-phenylpentane-1,4-dione (3kw).¹⁹ The title compound was prepared according to the general



experimental procedure A on a 0.5 mmol for 34 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3-4%) to afford a yellow oil (MeOH:H₂O – 38 mg, 40% & CH₃CN:H₂O – 22 mg, 23%); ¹H NMR (CDCl₃, 500 MHz): δ 7.98 (dd, *J* = 8.3, 1.1 Hz, 2H), 7.59 – 7.53 (m, 1H), 7.47 (dd, *J* = 10.6, 4.6 Hz,

2H), 3.97 (dq, *J* = 14.4, 7.2, 5.1 Hz, 1H), 3.17 (dd, *J* = 18.1, 8.5 Hz, 1H), 2.56 (dd, *J* = 18.1, 5.05 Hz, 1H), 2.18 (s, 3H), 1.19 (d, *J* = 7.2 Hz, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 207.3, 203.5, 136.2, 133.2, 128.8, 128.7, 47.0, 36.4, 30.3, 18.0.

3-Acetyl-2-methyl-1-phenylpentane-1,4-dione (3kw'). The title compound was prepared according to the

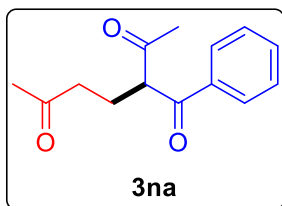


general experimental procedure A on a 0.5 mmol for 34 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 6-8%) to afford a yellow oil (MeOH:H₂O – <5% & CH₃CN:H₂O – 26 mg, 22%); ¹H NMR (CDCl₃, 500 MHz): δ 7.98 (d, *J* = 7.3 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.7 Hz, 2H), 4.33 (ddd, *J*

= 11.7, 10.6, 7.0 Hz, 2H), 2.34 (s, 3H), 2.26 (s, 3H), 1.15 (d, *J* = 6.9 Hz, 3H). ¹³C{¹H} NMR (CDCl₃, 101

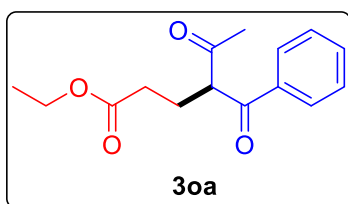
MHz): δ 203.0, 202.5, 202.1, 135.5, 133.5, 128.9, 128.7, 71.5, 41.6, 30.5, 30.4, 16.2. HRMS (ESI) calculated for $C_{14}H_{16}O_3Na$ $[M+Na]^+$: 255.0987 found 255.0987.

3-Benzoylheptane-2,6-dione (3na).²⁰ The title compound was prepared according to the general experimental



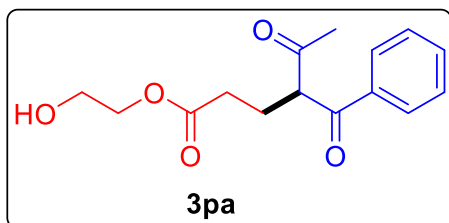
procedure A on a 0.5 mmol for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 6%) to afford a yellow oil (MeOH:H₂O – <5% & CH₃CN:H₂O – 78 mg, 63%); ¹H NMR (CDCl₃, 500 MHz): δ 8.05 – 8.01 (m, 2H), 7.63 – 7.58 (m, 1H), 7.53 – 7.47 (m, 2H), 4.60 – 4.54 (m, 1H), 2.63 – 2.44 (m, 2H), 2.28 – 2.16 (m, 2H), 2.15 (d, J = 2.1 Hz, 3H), 2.12 (d, J = 2.0 Hz, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 208.0, 204.1, 196.7, 136.2, 134.0, 129.0, 128.8, 61.2, 40.6, 30.1, 28.7, 22.5.

Ethyl 4-benzoyl-5-oxohexanoate (3oa).²¹ The title compound was prepared according to the general



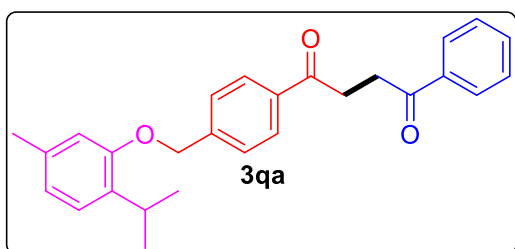
experimental procedure A on a 0.5 mmol for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 6%) to afford a yellow oil (MeOH:H₂O – <5% & CH₃CN:H₂O – 86 mg, 66%); ¹H NMR (CDCl₃, 500 MHz): δ 8.06 – 8.00 (m, 2H), 7.64 – 7.59 (m, 1H), 7.52 – 7.48 (m, 2H), 4.63 (t, J = 6.8 Hz, 1H), 4.13 (qd, J = 7.1, 1.2 Hz, 2H), 2.45 – 2.22 (m, 4H), 2.16 (s, 3H), 1.24 (td, J = 7.1, 1.3 Hz, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 203.7, 196.4, 172.9, 136.3, 134.0, 129.0, 128.9, 61.5, 60.7, 31.6, 28.6, 23.8, 14.3; HRMS (ESI) calculated for $C_{15}H_{18}O_4Na$ $[M+Na]^+$: 285.1085 found 285.1085.

2-Hydroxyethyl 4-benzoyl-5-oxohexanoate (3pa). The title compound was prepared according to the general



experimental procedure A on a 0.5 mmol for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 80-90%) to afford a yellow oil (MeOH:H₂O – <5% & CH₃CN:H₂O – 99 mg, 75%); ¹H NMR (CDCl₃, 500 MHz): δ 8.02 (d, J = 7.4 Hz, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.7 Hz, 2H), 4.62 (t, J = 6.9 Hz, 1H), 4.26 – 4.16 (m, 2H), 3.82 (t, J = 4.6 Hz, 2H), 2.51 – 2.23 (m, 5H), 2.16 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 203.9, 196.3, 173.1, 136.2, 134.1, 129.1, 128.9, 66.3, 61.5, 61.1, 31.6, 28.6, 23.8; HRMS (ESI) calculated for $C_{15}H_{18}O_5Na$ $[M+Na]^+$: 301.1047 found 301.1047.

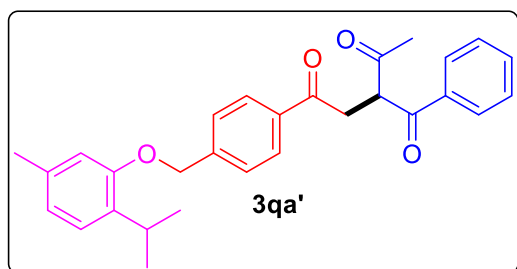
1-(4-((2-Isopropyl-5-methylphenoxy)methyl)phenyl)-4-phenylbutane-1,4-dione (3qa). The title



compound was prepared according to the general experimental procedure A on a 0.5 mmol for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 6%) to afford a white solid (MeOH:H₂O – <5% & CH₃CN:H₂O – 72 mg, 36%); mp 122-124 °C; ¹H NMR (CDCl₃, 500 MHz): δ 8.08 (d, J = 8.3 Hz, 2H), 8.06 – 8.03 (m, 2H), 7.61 – 7.54 (m, 3H), 7.48 (dd, J = 10.5, 4.8 Hz, 2H), 7.14 (d, J = 7.7 Hz, 1H), 6.79 (d, J = 7.7 Hz, 1H), 6.71 (s, 1H), 5.14 (s, 2H), 3.48 (s, 4H), 3.38 (dt, J = 13.8, 6.9 Hz, 1H), 2.32 (s, 3H), 1.24 (d, J = 6.9 Hz, 6H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.8, 198.4, 155.6, 143.2, 136.9, 136.5, 136.3,

134.4, 133.3, 128.7, 128.5, 128.2, 127.0, 126.2, 121.8, 112.7, 69.4, 32.7, 26.8, 22.9, 21.4; HRMS (ESI) calculated for $C_{27}H_{28}O_3Na$ $[M+Na]^+$: 423.1925 found 423.1925.

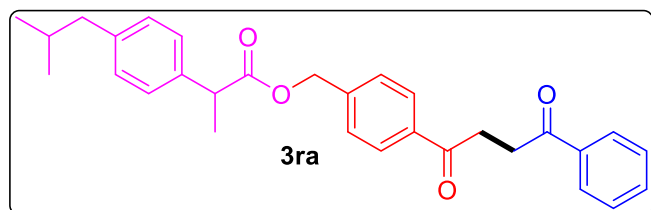
3-Benzoyl-1-(4-((2-isopropyl-5-methylphenoxy)methyl)phenyl)pentane-1,4-dione (3qa'). The title



compound was prepared according to the general experimental procedure A on a 0.5 mmol for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 6%) to afford a mixture of diastereomers as yellow oil (MeOH:H₂O – <5% & CH₃CN:H₂O – 51 mg, 23%); ¹H NMR (CDCl₃, 500 MHz): δ 8.09 (dd, *J* = 8.3, 1.1 Hz, 2H), 8.02 (d, *J* = 8.3 Hz, 2H), 7.67 – 7.61 (m, 1H),

7.57 – 7.51 (m, 4H), 7.13 (d, *J* = 7.7 Hz, 1H), 6.78 (d, *J* = 7.5 Hz, 1H), 6.69 (s, 1H), 5.31 (s, 1H), 5.13 (s, 2H), 3.80 (dd, *J* = 18.2, 6.8 Hz, 1H), 3.65 – 3.58 (m, 1H), 3.37 (dq, *J* = 13.8, 6.8 Hz, 1H), 2.31 (s, 3H), 2.25 (s, 3H), 1.24 (s, 3H), 1.22 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 202.4, 196.6, 196.4, 155.5, 143.7, 136.5, 136.2, 135.5, 134.4, 134.0, 129.1, 129.0, 128.8, 128.7, 128.3, 127.0, 126.2, 121.9, 112.6, 69.3, 57.0, 38.2, 29.7, 26.8, 22.9, 21.5. HRMS (ESI) calculated for $C_{29}H_{30}O_4Na$ $[M+Na]^+$: 465.2036 found 465.2025.

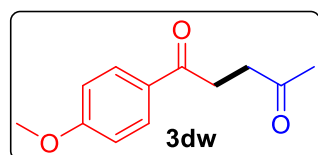
4-(4-Oxo-4-phenylbutanoyl)benzyl 2-(4-isobutylphenyl)propanoate (3ra). The title compound was



prepared according to the general experimental procedure A on a 0.5 mmol for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 6%) to afford a white solid

(MeOH:H₂O – <5% & CH₃CN:H₂O – 71 mg, 31%); mp 96-98 °C; ¹H NMR (CDCl₃, 500 MHz): δ 8.04 (d, *J* = 7.7 Hz, 2H), 7.96 (d, *J* = 8.2 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 5.23 – 5.12 (m, 2H), 3.78 (q, *J* = 7.1 Hz, 1H), 3.50 – 3.40 (m, 4H), 2.46 (d, *J* = 7.2 Hz, 2H), 1.86 (dp, *J* = 13.5, 6.8 Hz, 1H), 1.53 (d, *J* = 7.2 Hz, 3H), 0.91 (d, *J* = 6.6 Hz, 6H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 198.7, 198.3, 174.5, 141.6, 140.9, 137.5, 136.9, 136.4, 133.3, 129.5, 128.7, 128.4, 128.2, 127.5, 127.3, 65.6, 45.2, 45.1, 32.7, 30.3, 22.5, 18.4; HRMS (ESI) calculated for $C_{30}H_{33}O_4$ $[M+H]^+$: 457.2367 found 457.2367.

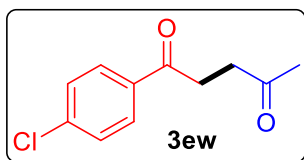
1-(4-Methoxyphenyl)pentane-1,4-dione (3dw).¹⁵ The title compound was prepared according to the general



experimental procedure A on a 0.5 mmol for 30 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 5%) to afford a pale white solid (MeOH:H₂O – 66 mg, 64% & CH₃CN:H₂O – 55 mg, 53%); mp 48-50 °C (lit. 51-

52 °C); ¹H NMR (CDCl₃, 500 MHz): δ 7.99 – 7.95 (m, 2H), 6.96 – 6.92 (m, 2H), 3.87 (s, 3H), 3.24 (t, *J* = 6.4 Hz, 2H), 2.87 (t, *J* = 6.3 Hz, 2H), 2.26 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 207.6, 197.0, 163.5, 130.3, 129.7, 113.7, 55.4, 37.1, 32.0, 30.1; HRMS (ESI) calculated for $C_{12}H_{15}O_2$ $[M+H]^+$: 191.1071 found 191.1067.

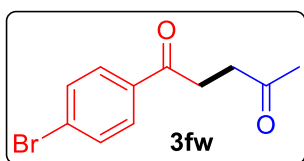
1-(4-Chlorophenyl)pentane-1,4-dione (3ew).¹⁵ The title compound was prepared according to the general



experimental procedure A on a 0.5 mmol for 30 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a white crystalline solid (MeOH:H₂O – 24 mg, 23% & CH₃CN:H₂O – 24 mg, 23%); mp 70-72 °C (lit. 69-71 °C); ¹H NMR (CDCl₃, 500 MHz): δ 7.92 (d, *J* = 8.5 Hz, 2H), 7.43 (d, *J* = 8.5

Hz, 2H), 3.24 (t, *J* = 6.2 Hz, 2H), 2.89 (t, *J* = 6.2 Hz, 2H), 2.26 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 207.2, 197.3, 139.6, 134.9, 129.4, 128.9, 37.0, 32.3, 30.1; HRMS (ESI) calculated for C₁₁H₁₁ClO₂Na [M+Na]⁺: 233.033 found 233.0333.

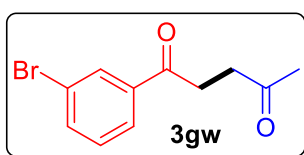
1-(4-Bromophenyl)pentane-1,4-dione (3fw).¹⁵ The title compound was prepared according to the general



experimental procedure A on a 0.5 mmol for 30 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a white crystalline solid (MeOH:H₂O – 83 mg, 65% & CH₃CN:H₂O – 46 mg, 36%); mp 86-89 °C (lit. 80-81 °C); ¹H NMR (CDCl₃, 500 MHz): δ 7.85 (d, *J* = 8.6 Hz, 2H), 7.61 (d, *J* = 8.6

Hz, 2H), 3.23 (t, *J* = 6.2 Hz, 2H), 2.89 (t, *J* = 6.2 Hz, 2H), 2.26 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 125 MHz): δ 207.3, 197.6, 135.5, 132.0, 129.7, 128.4, 37.1, 32.4, 30.2; HRMS (ESI) calculated for C₁₁H₁₂BrO₂ [M+H]⁺: 255.0013 found 255.0015.

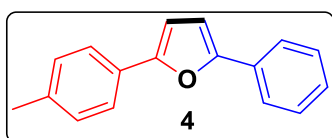
1-(3-Bromophenyl)pentane-1,4-dione (3gw).²² The title compound was prepared according to the general



experimental procedure A on a 0.5 mmol for 36 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford yellow oil (MeOH:H₂O – 45 mg, 35% & CH₃CN:H₂O – 51 mg, 40%); ¹H NMR (CDCl₃, 500

MHz): δ 8.10 (s, 1H), 7.89 (d, *J* = 7.8 Hz, 1H), 7.68 (d, *J* = 7.9 Hz, 1H), 7.34 (t, *J* = 7.9 Hz, 1H), 3.23 (t, *J* = 6.2 Hz, 2H), 2.89 (t, *J* = 6.2 Hz, 2H), 2.25 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 207.1, 197.3, 138.5, 136.1, 131.3, 130.3, 126.7, 123.1, 37.1, 32.6, 30.1; HRMS (ESI) calculated for C₁₁H₁₂BrO₂ [M+H]⁺: 255.0005 found 255.0015.

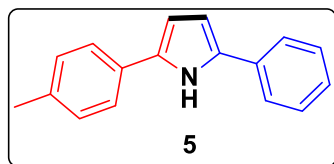
2-Phenyl-5-(*p*-tolyl)furan (4).²³ The title compound was prepared according to the general experimental



procedure B on a 0.5 mmol for 6 h, and the product was isolated by column chromatography (100% Hexane) to afford a white solid (94 mg, 80%); mp 105-107 °C (lit. 101-102 °C); ¹H NMR (CDCl₃, 500 MHz): δ 7.74 (d, *J* = 7.5 Hz, 2H),

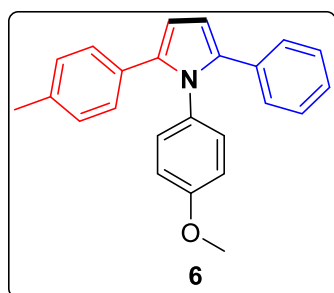
7.64 (d, *J* = 6.5 Hz, 2H), 7.40 (t, *J* = 8.0 Hz, 2H), 7.28 – 7.24 (m, 2H), 7.21 (d, *J* = 7.9 Hz, 2H), 6.74 – 6.67 (m, 2H), 2.38 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 153.6, 152.9, 137.2, 130.8, 129.3, 128.6, 128.1, 127.1, 123.7, 123.6, 107.1, 106.4, 21.2; HRMS (ESI) calculated for C₁₇H₁₅O [M+H]⁺: 235.1106 found 235.1106.

2-Phenyl-5-(*p*-tolyl)-1*H*-pyrrole (5).²⁴ The title compound was prepared according to the general



experimental procedure C on a 0.5 mmol for 3 h, and the product was isolated by column chromatography (100% Hexane) to afford a white solid (94 mg, 80%); mp 142-144 °C (lit. 142-143 °C); ¹H NMR (CDCl₃, 500 MHz): δ 8.54 (s, 1H), 7.54 – 7.50 (m, 2H), 7.43 (d, *J* = 8.0 Hz, 2H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.24 – 7.19 (m, 3H), 6.57 (t, *J* = 3.0 Hz, 1H), 6.53 (t, *J* = 2.5 Hz, 1H), 2.36 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 136.6, 133.7, 133.2, 133.0, 130.2, 130.1, 129.4, 126.7, 124.2, 124.2, 108.3, 107.8, 21.62 HRMS (ESI) calculated for C₁₇H₁₅N [M+H]⁺: 234.1255 found 234.1255.

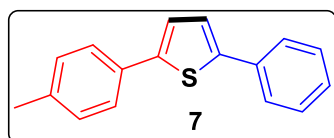
1-(4-Methoxyphenyl)-2-phenyl-5-(*p*-tolyl)-1*H*-pyrrole (6).²⁵ The title compound was prepared according to



the general experimental procedure D on a 0.5 mmol for 3 h, and the product was isolated by column chromatography (100% Hexane) to afford a white solid (128 mg, 75%); mp 195-197 °C; ¹H NMR (CDCl₃, 500 MHz): δ 7.19 – 7.11 (m, 3H), 7.08 (d, *J* = 7.0 Hz, 2H), 7.00 – 6.94 (m, 6H), 6.76 (d, *J* = 8.8 Hz, 2H), 6.45 (d, *J* = 3.5 Hz, 1H), 6.42 (d, *J* = 3.5 Hz, 1H), 3.78 (s, 3H), 2.28 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 158.4, 136.0, 135.8, 135.6, 133.4, 132.0, 130.4,

129.8, 128.6, 128.5, 127.8, 126.0, 113.9, 109.5, 109.2, 55.3, 21.1; HRMS (ESI) calculated for C₂₄H₂₂NO [M+H]⁺: 340.1698 found 340.1698.

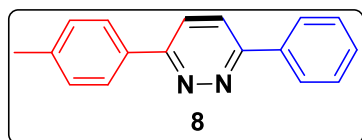
2-Phenyl-5-(*p*-tolyl)thiophene (7).²⁶ The title compound was prepared according to the general experimental



procedure E on a 0.5 mmol for 6 h, and the product was isolated by column chromatography (100% Hexane) to afford a white solid (102 mg, 82%); mp 146-148 °C (lit. 150-152 °C); ¹H NMR (CDCl₃, 500 MHz): δ 7.63 (d, *J* = 7.4 Hz, 2H),

7.53 (d, *J* = 8.0 Hz, 2H), 7.38 (t, *J* = 7.7 Hz, 2H), 7.30 – 7.24 (m, 4H), 7.20 (d, *J* = 7.9 Hz, 2H), 2.37 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 143.8, 143.0, 137.4, 134.3, 131.5, 129.5, 128.8, 127.3, 125.5, 123.9, 123.6, 123.4, 21.1; HRMS (ESI) calculated for C₁₇H₁₅S [M+H]⁺: 251.2878 found 251.2889.

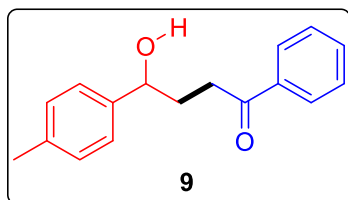
3-Phenyl-6-(*p*-tolyl)pyridazine (8).²⁷ The title compound was prepared according to the general experimental



procedure F on a 0.5 mmol for 3 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 3%) to afford a white solid (79 mg, 64%); mp 182-184 °C (lit. 187-189 °C); ¹H NMR (CDCl₃, 500 MHz): δ 8.16

(d, *J* = 7.1 Hz, 2H), 8.07 (d, *J* = 8.1 Hz, 2H), 7.91 (s, 2H), 7.56 – 7.49 (m, 3H), 7.35 (d, *J* = 7.9 Hz, 2H), 2.45 (s, 3H). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 157.5, 157.3, 140.2, 136.2, 133.2, 129.9, 129.7, 129.0, 126.8, 126.8, 124.1, 123.8, 21.3; HRMS (ESI) calculated for C₁₇H₁₅N₂ [M+H]⁺: 247.1230 found 247.1230.

4-Hydroxy-1-phenyl-4-(*p*-tolyl)butan-1-one (9).⁶ The title compound was prepared according to the general



experimental procedure G on a 1.0 mmol for 24 h, and the product was isolated by column chromatography (Ethyl acetate/Hexane: 6%) to afford a yellow oil; ¹H NMR (500 MHz, CDCl₃) δ 7.92 (d, *J* = 7.7 Hz, 2H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.42 (t, *J* = 7.7 Hz, 2H), 7.25 (d, *J* = 7.8 Hz, 2H), 7.14 (d, *J* = 7.8 Hz, 2H), 4.76 (t, *J* = 6.3 Hz, 1H), 3.07 (t, *J* = 7.0 Hz, 2H), 2.32 (s, 3H), 2.20 – 2.13 (m, 2H); HRMS (ESI) calculated for C₁₆H₁₃BrO₂ C₁₇H₁₉O₂ [M+H]⁺: 277.1235 found 277.1235.

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21. Copies of ^1H and ^{13}C Data

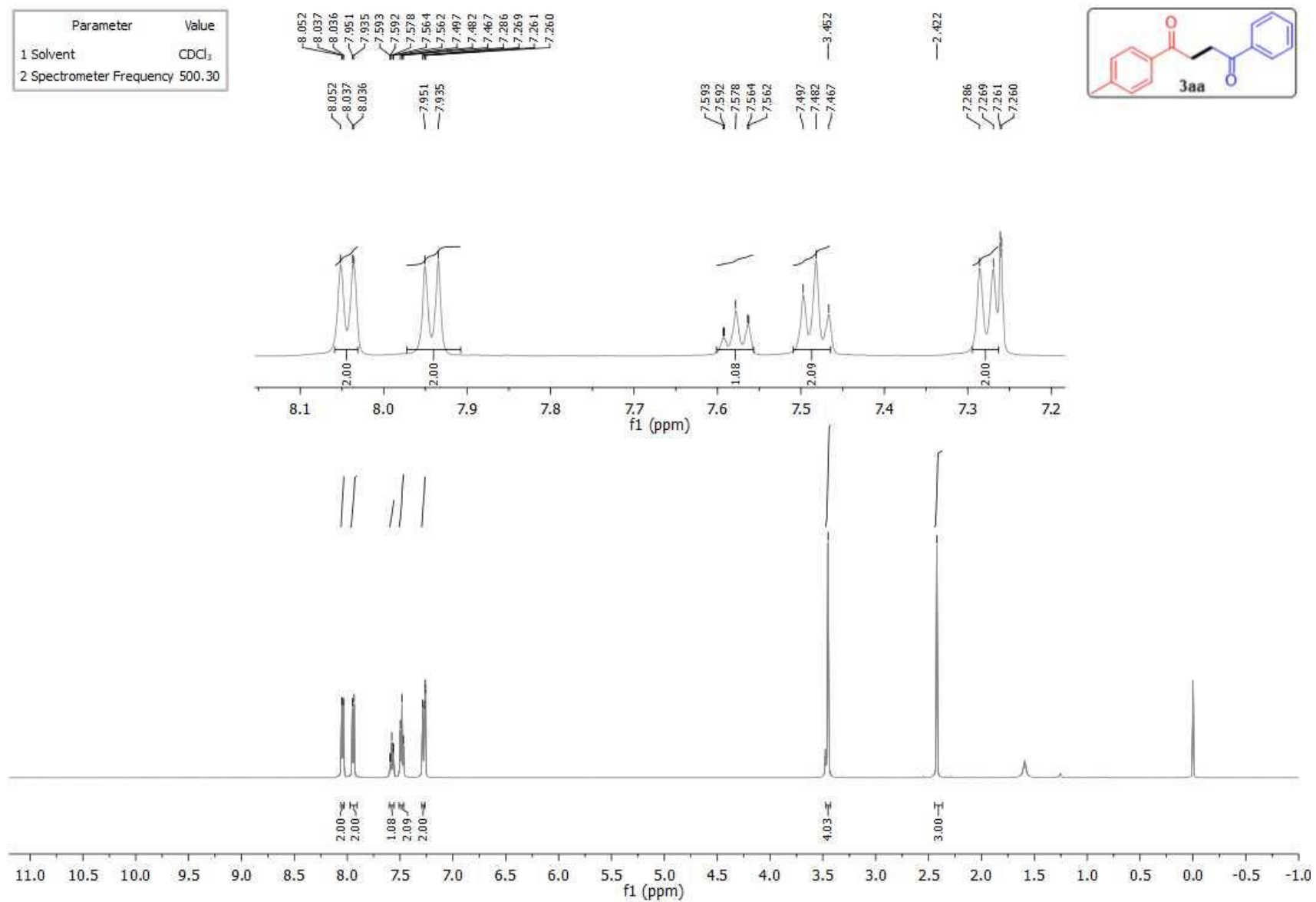


Figure S4. ^1H NMR spectrum of 1-Phenyl-4-(*p*-tolyl)butane-1,4-dione (**3aa**).

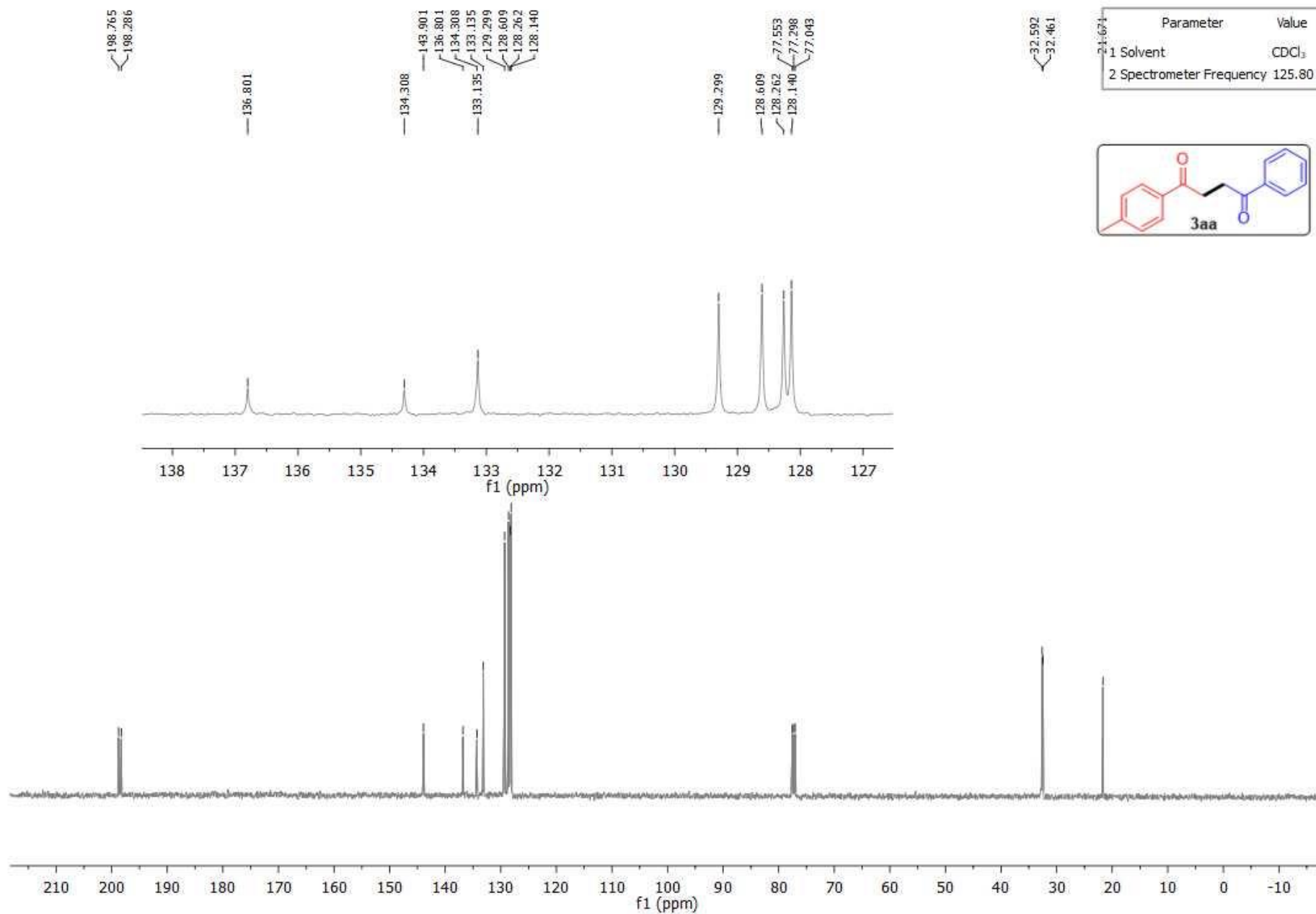


Figure S5. ¹³C NMR spectrum of 1-Phenyl-4-(*p*-tolyl)butane-1,4-dione (**3aa**).

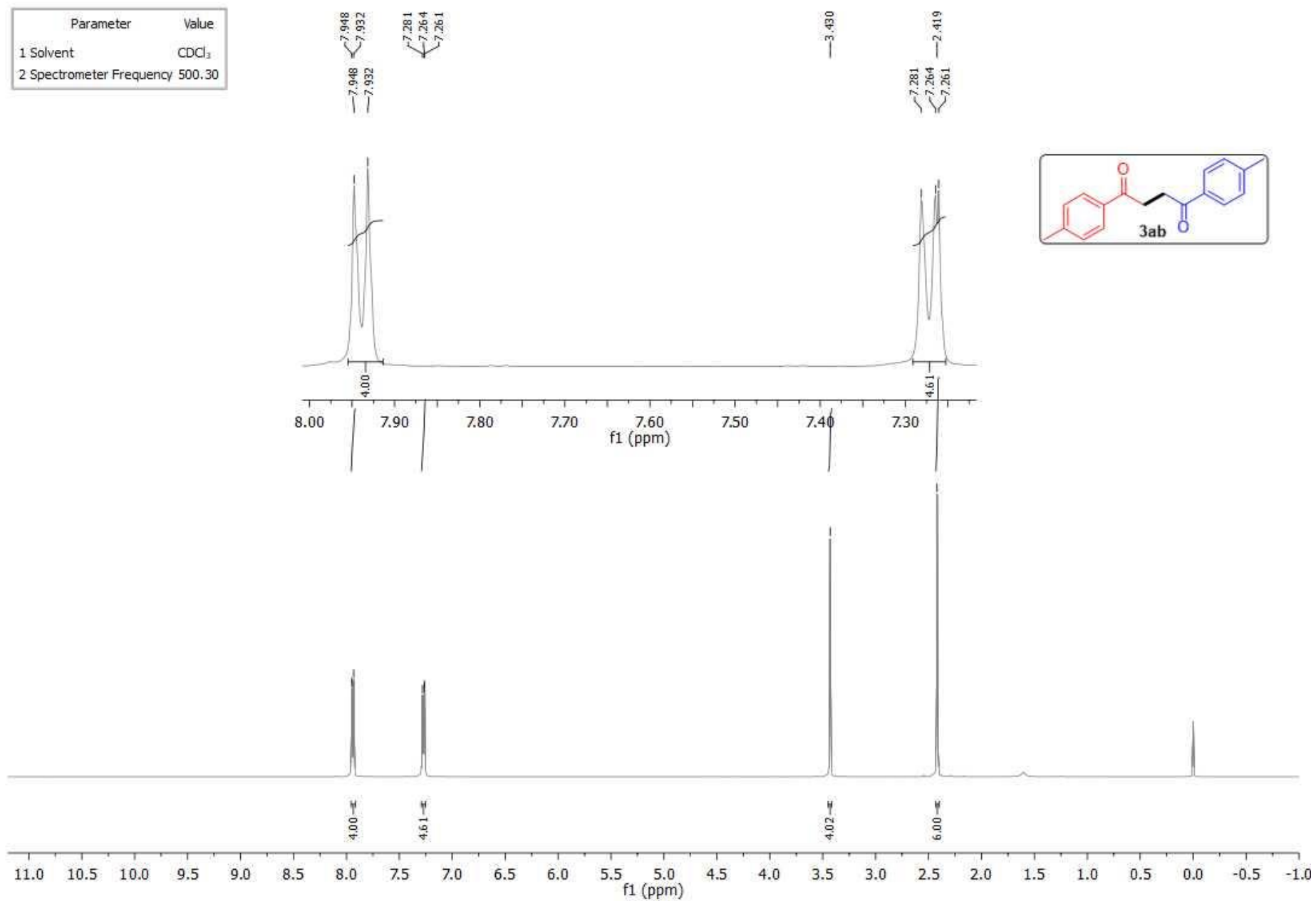


Figure S6. ¹H NMR spectrum of 1,4-Di-*p*-tolylbutane-1,4-dione (**3ab**).

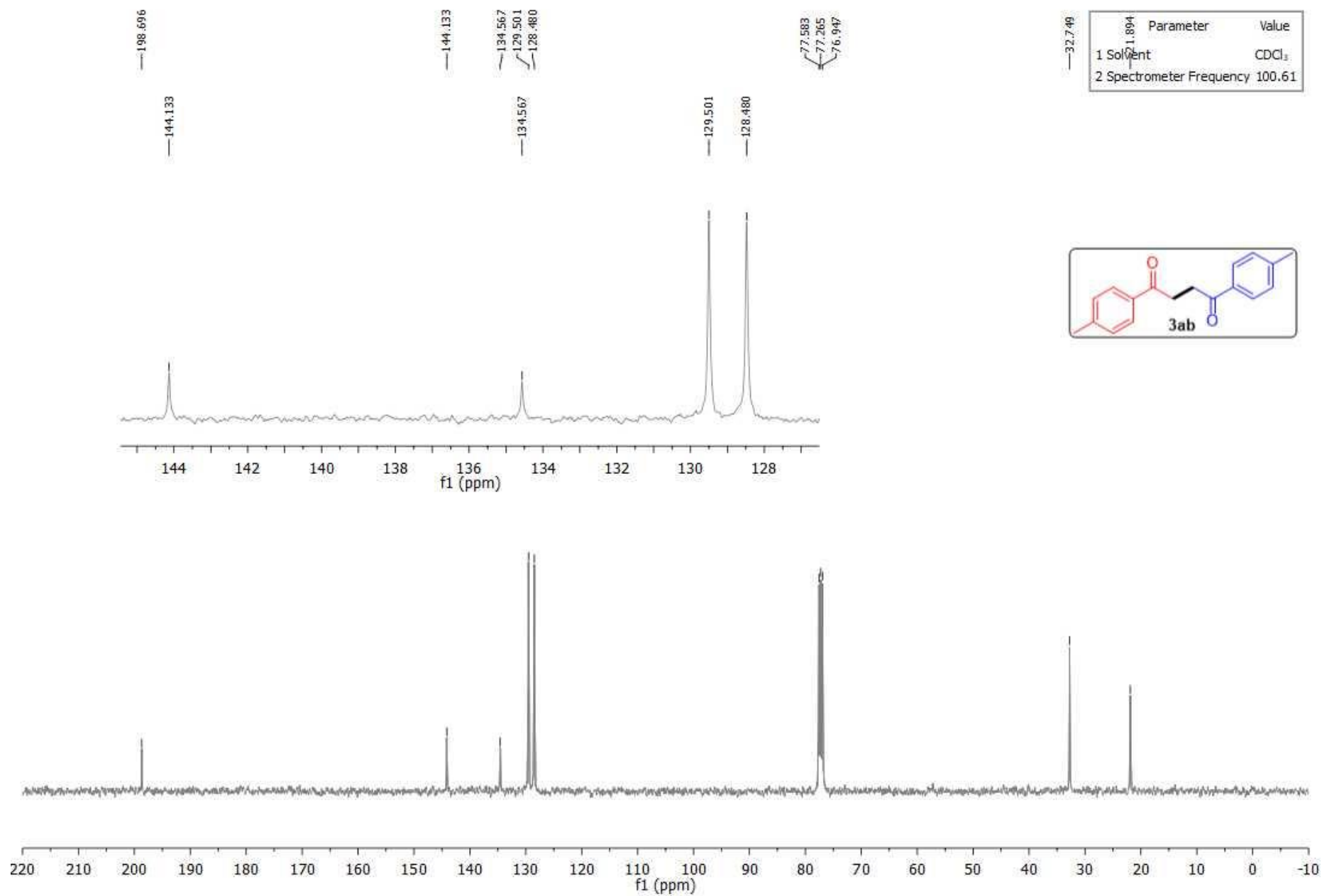


Figure S7. ¹³C NMR spectrum of 1,4-Di-*p*-tolylbutane-1,4-dione (**3ab**).

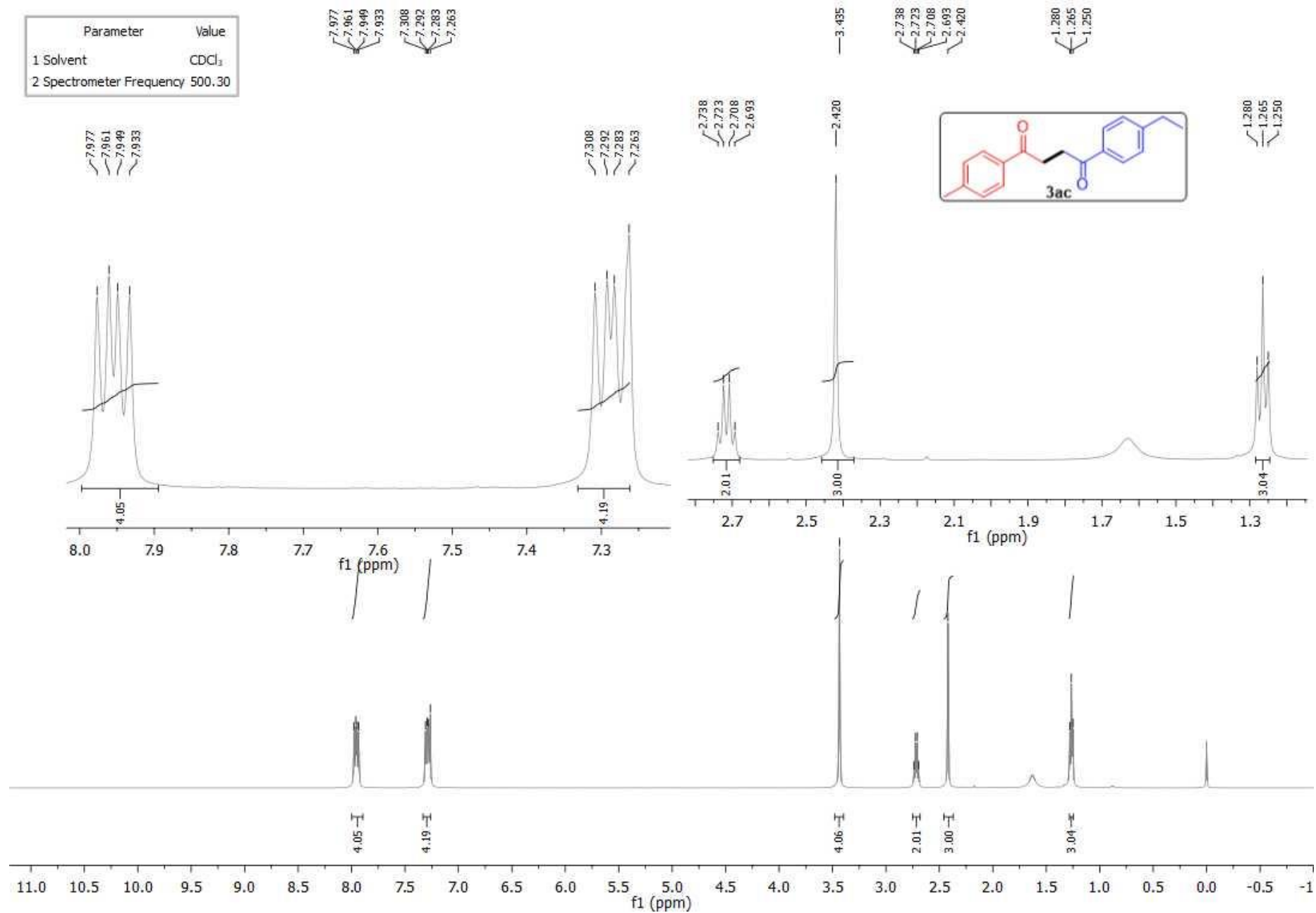


Figure S8. ¹H NMR spectrum of 1-(4-Ethylphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ac**).

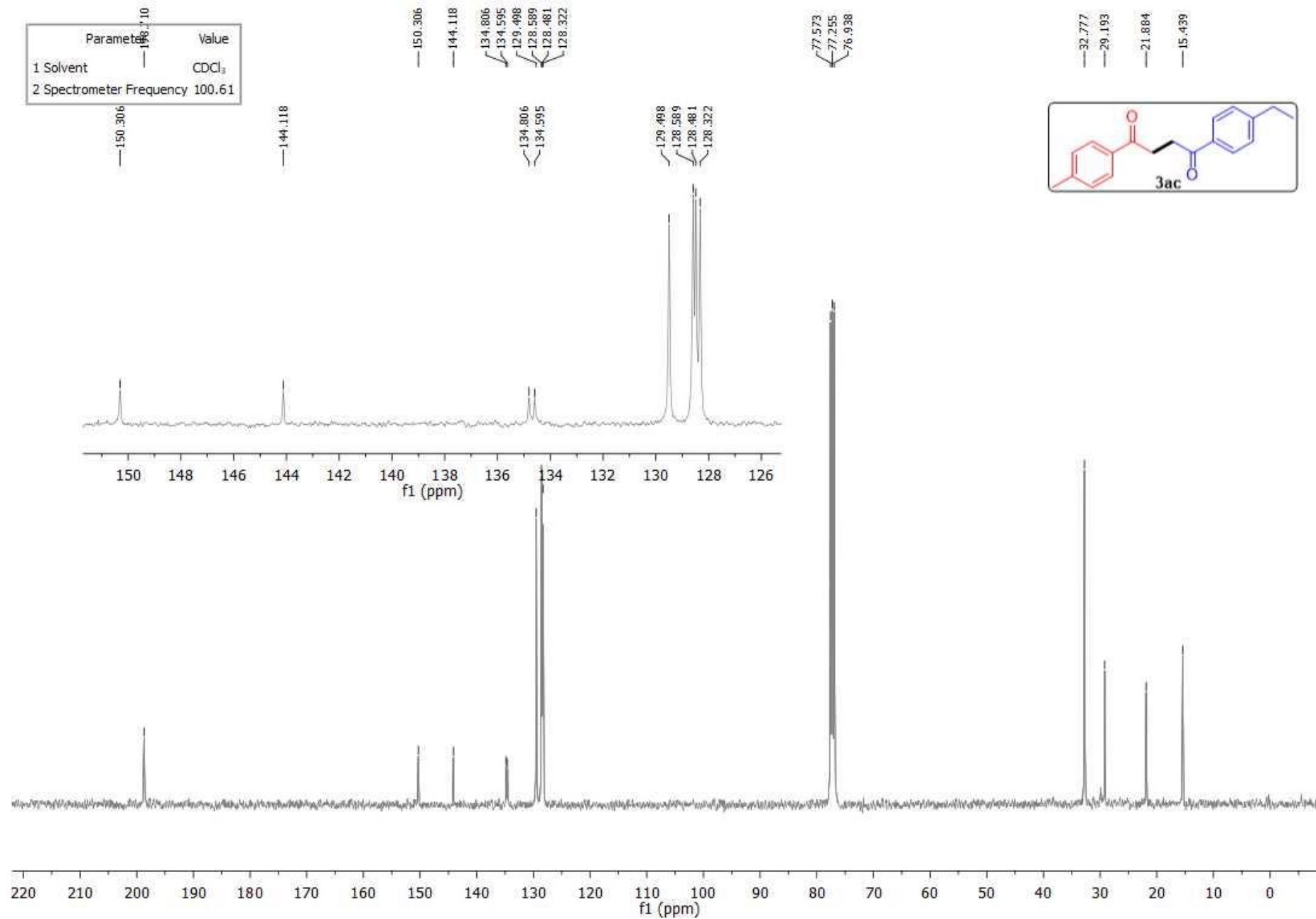


Figure S9. ¹³C NMR spectrum of 1-(4-Ethylphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ac**).

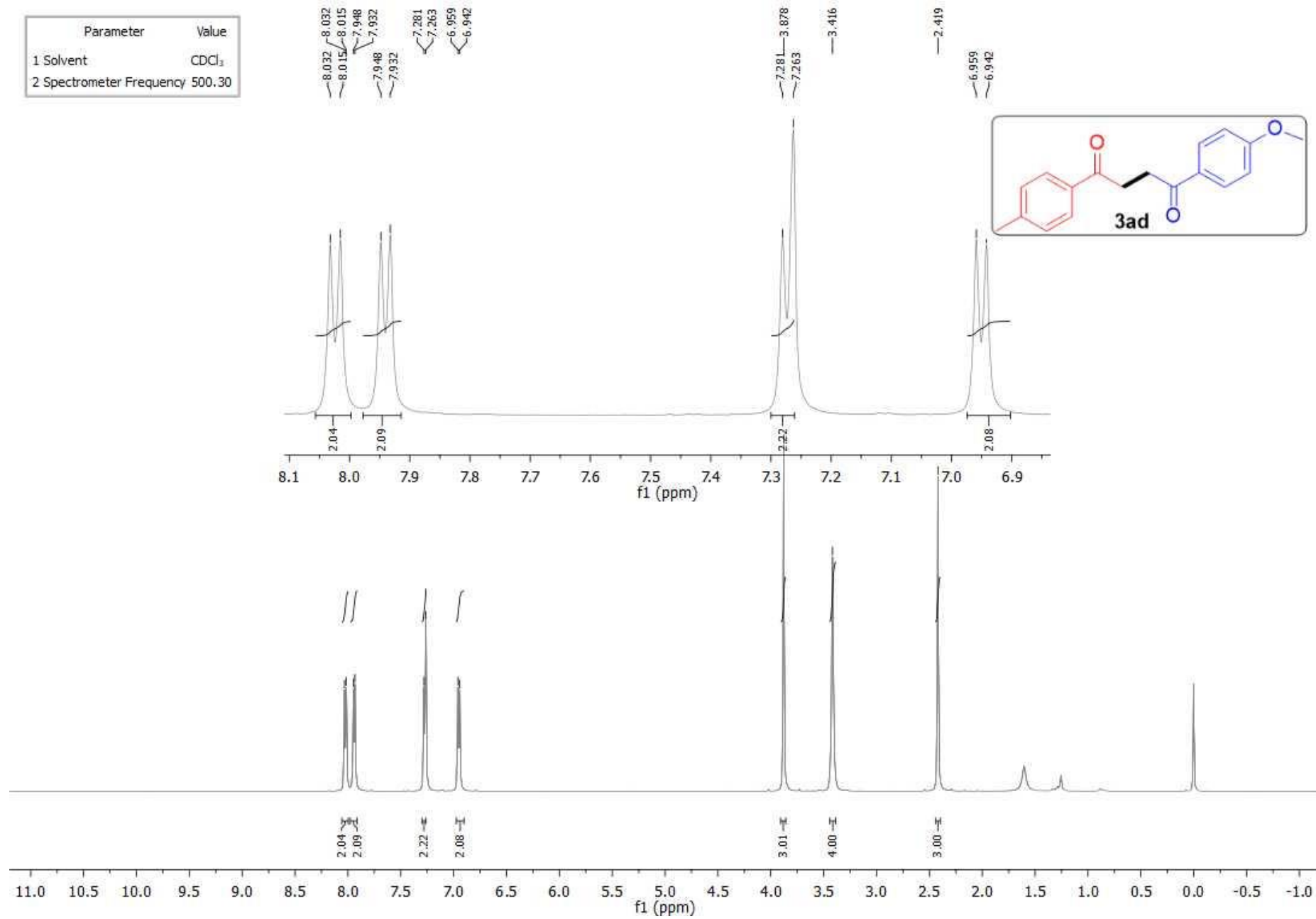


Figure S10. ¹H NMR spectrum of 1-(4-Methoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ad**).

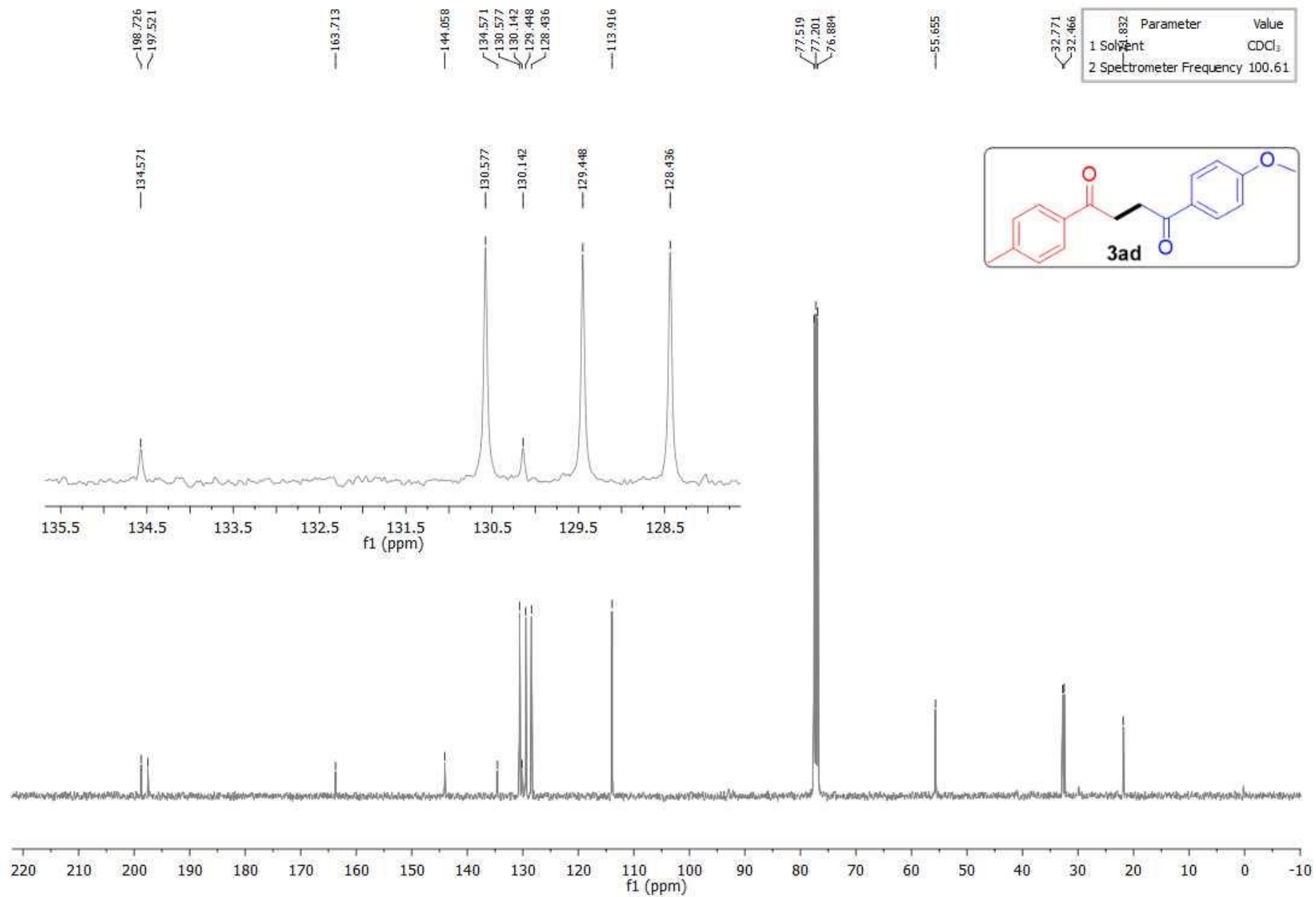


Figure S11. ^{13}C NMR spectrum of 1-(4-Methoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ad**).

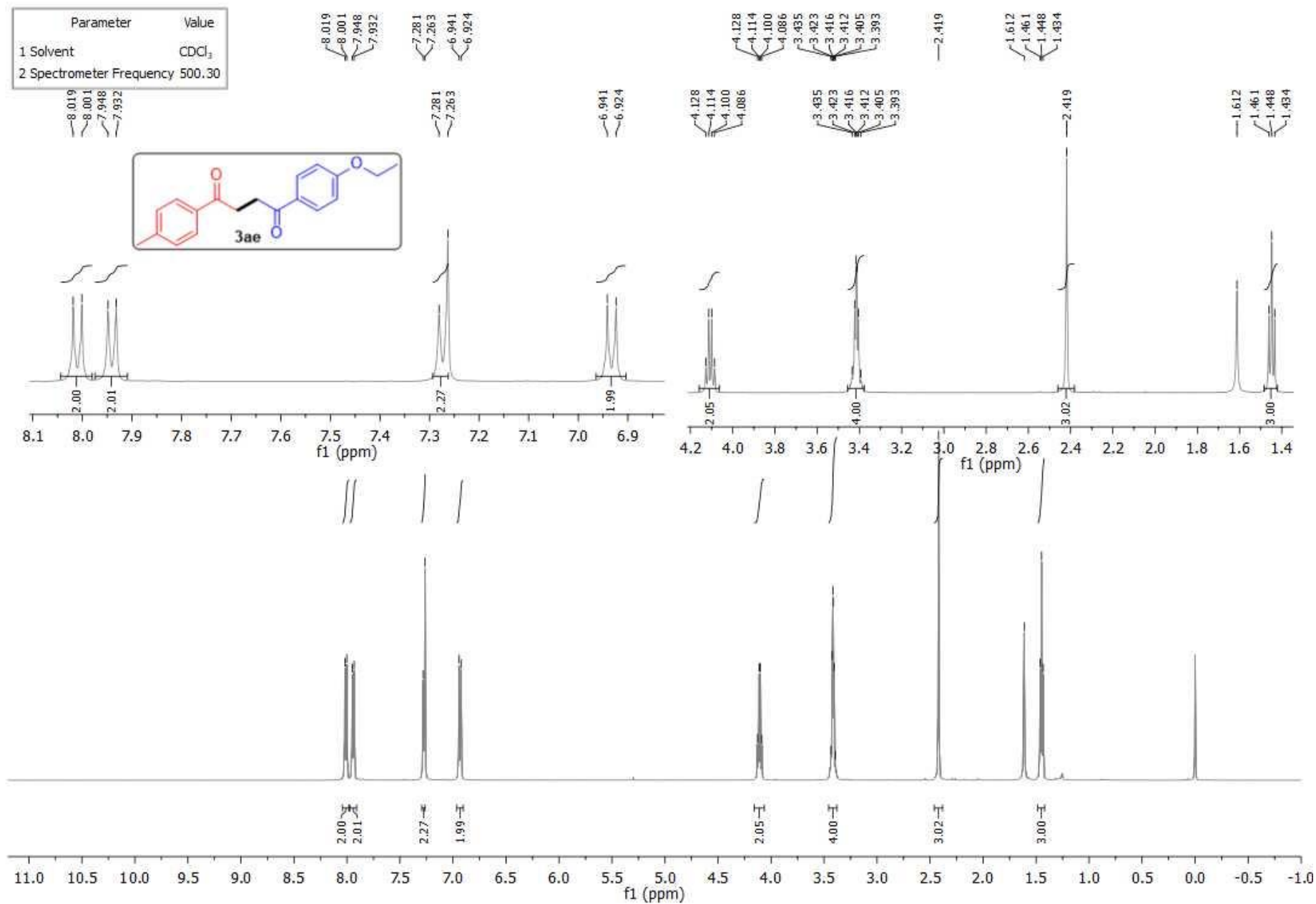


Figure S12. ¹H NMR spectrum of 1-(4-Ethoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ae**).

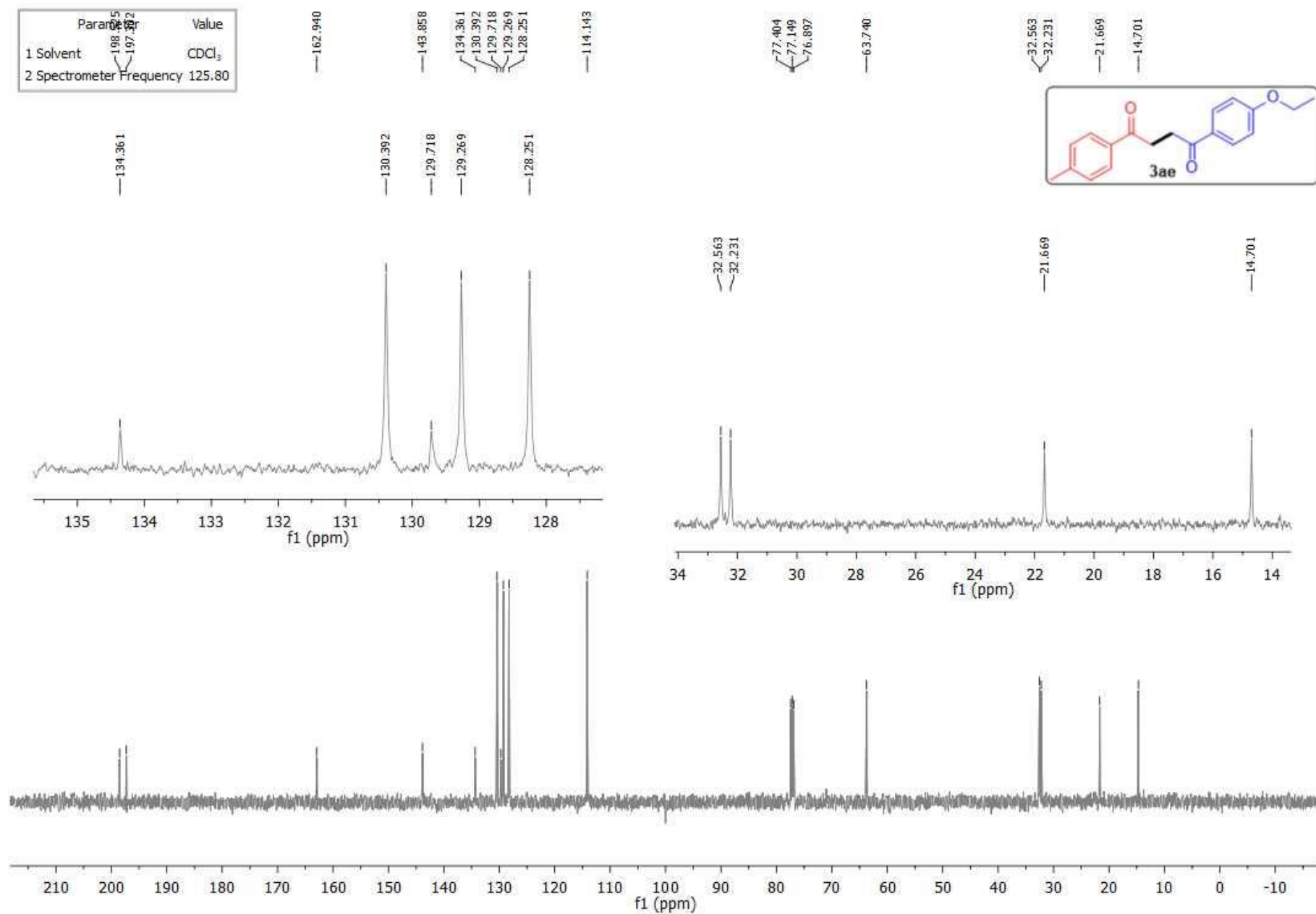


Figure S13. ¹³C NMR spectrum of 1-(4-Ethoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ae**).

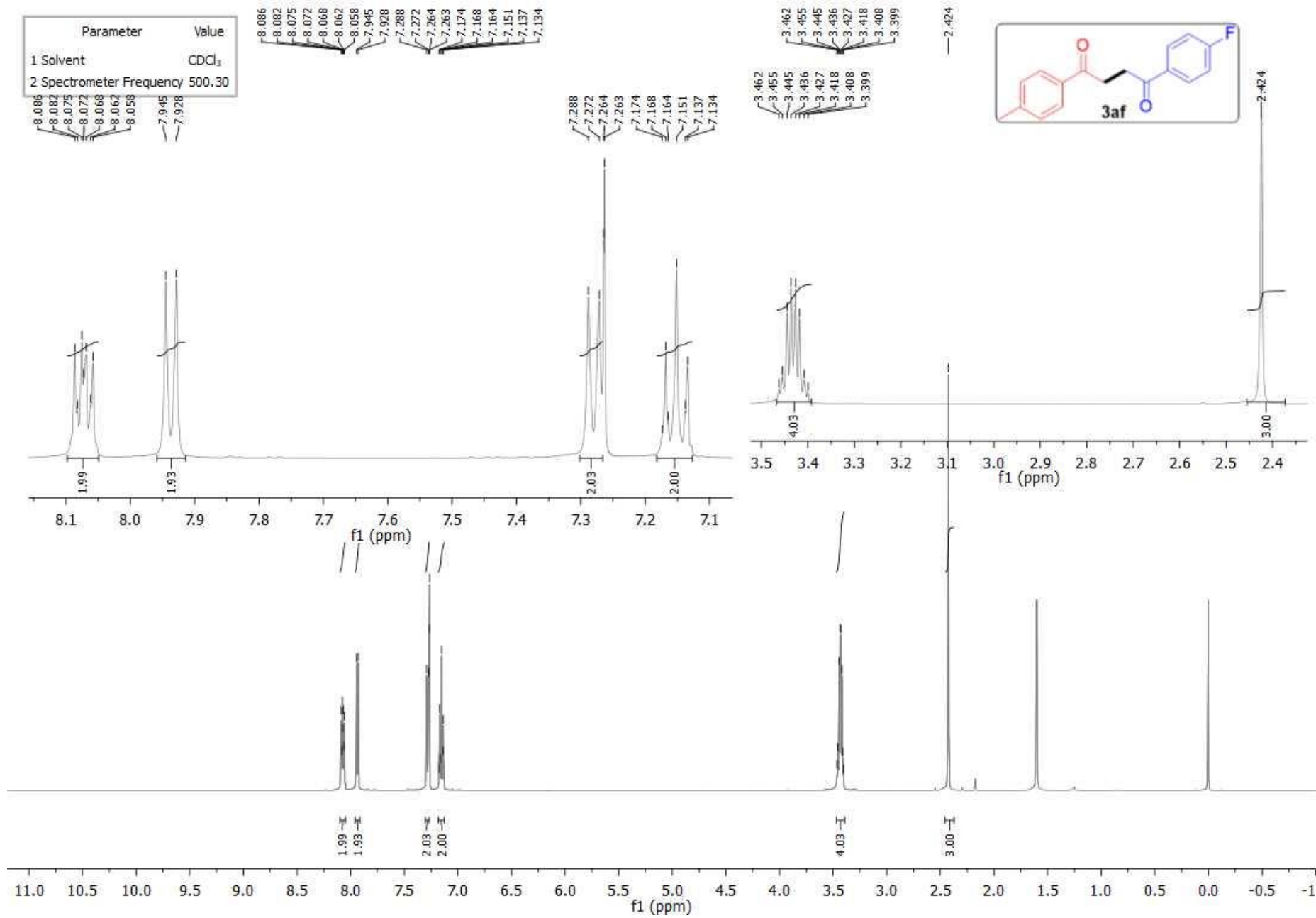


Figure S14. ¹H NMR spectrum of 1-(4-Fluorophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3af**).

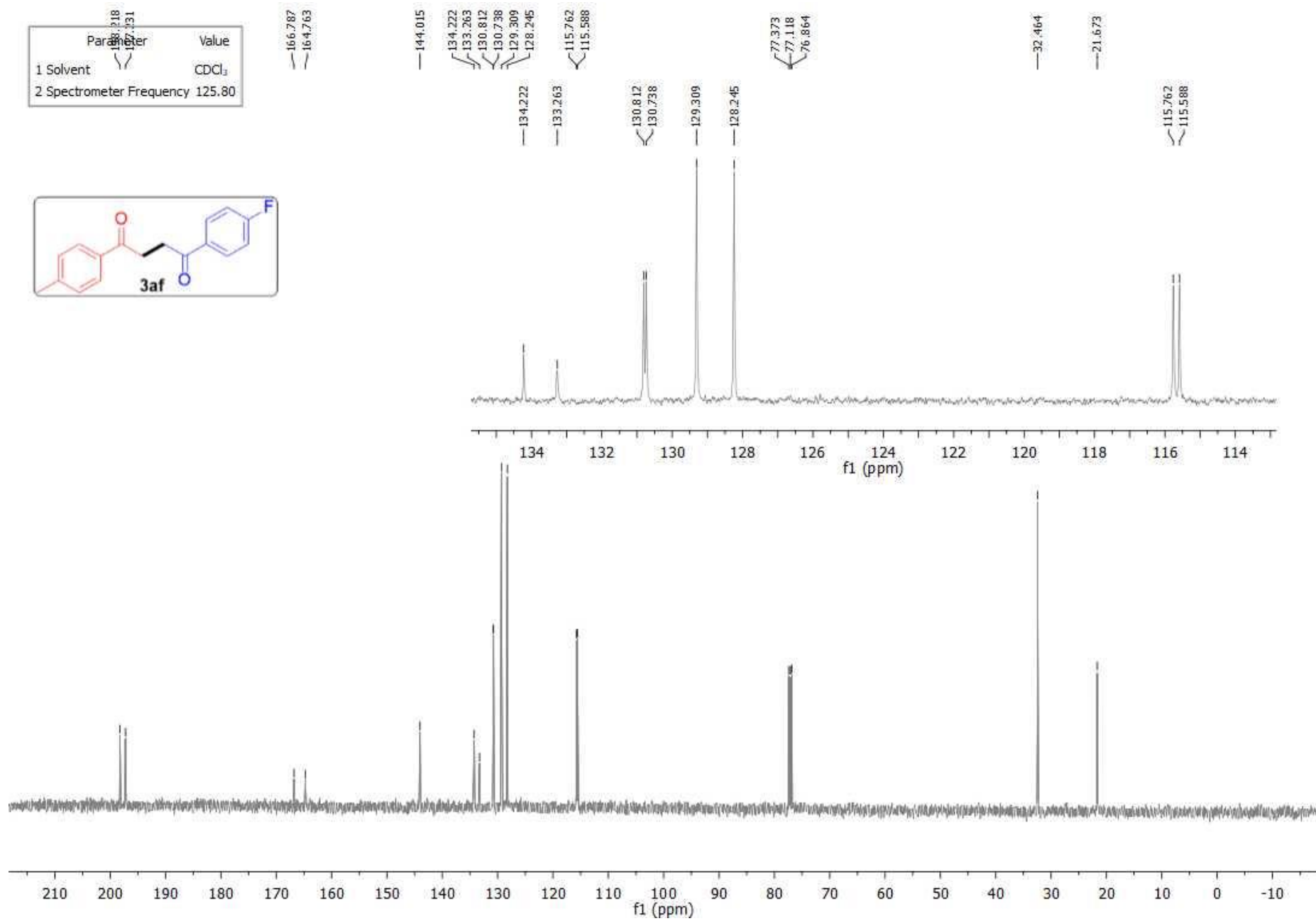


Figure S15. ¹³C NMR spectrum of 1-(4-Fluorophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3af**).

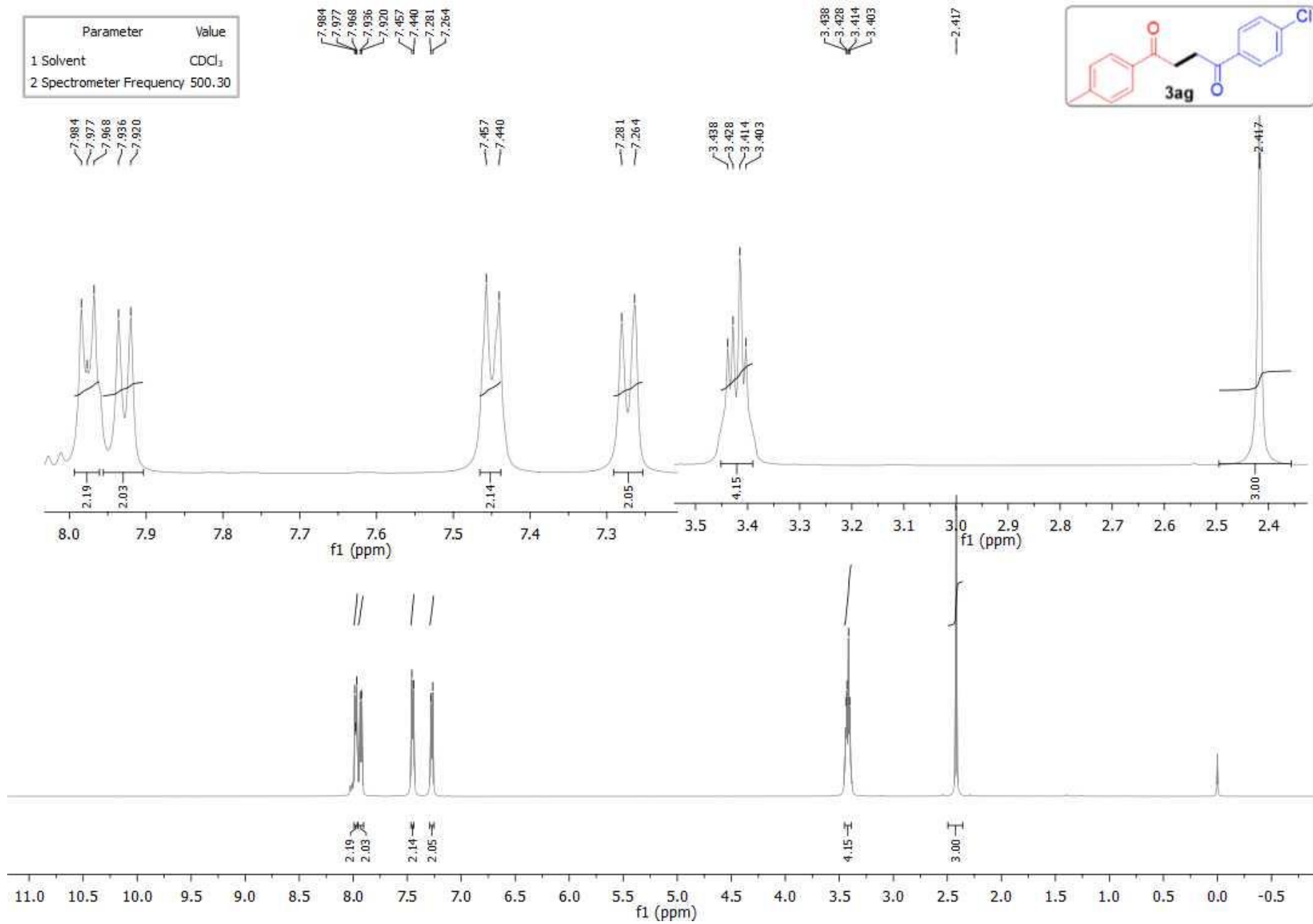


Figure S16. ¹H NMR spectrum of 1-(4-Chlorophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ag**).

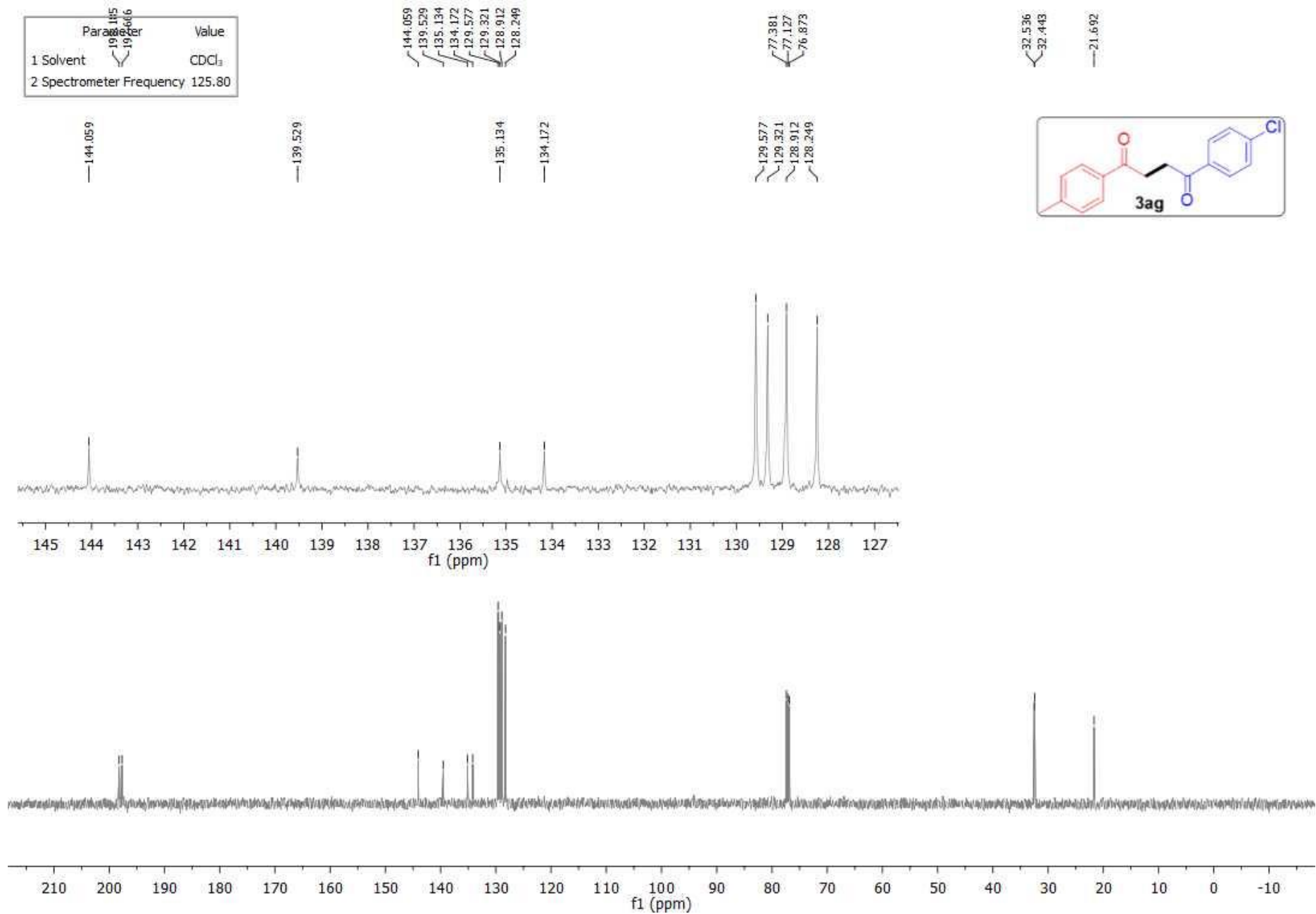


Figure S17. ¹³C NMR spectrum of 1-(4-Chlorophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ag**).

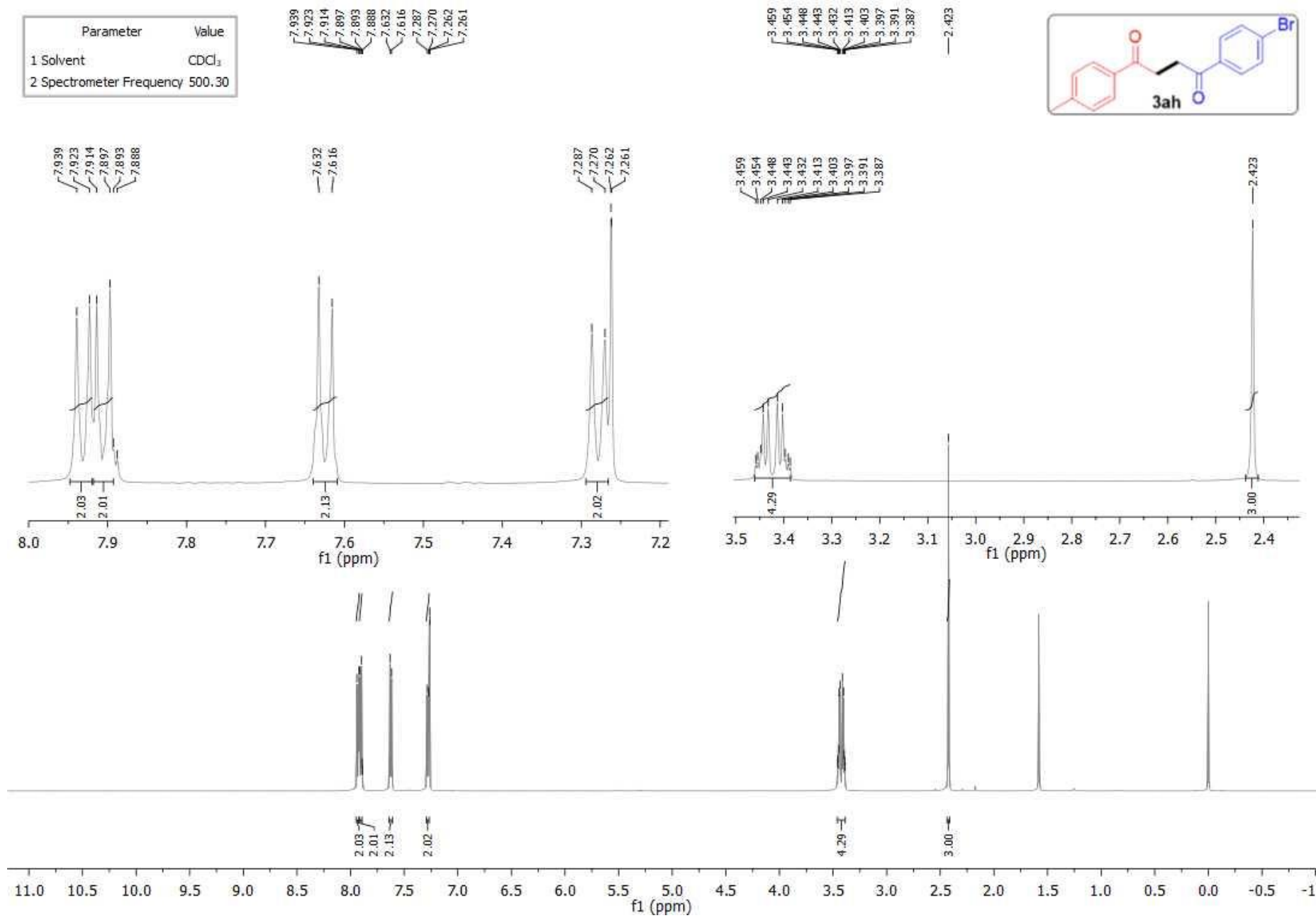


Figure S18. ¹H NMR spectrum of 1-(4-Bromophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ah**).

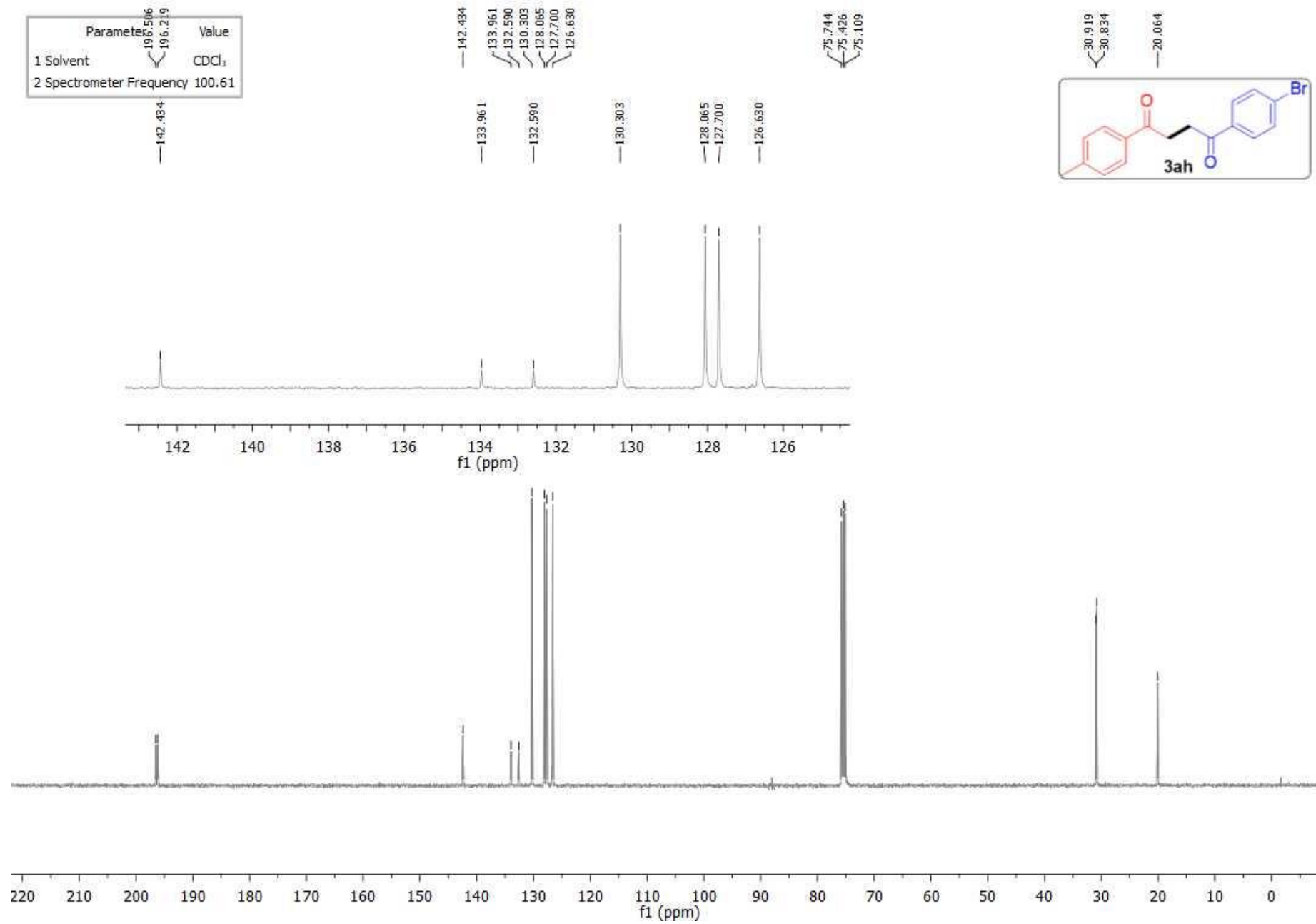


Figure S19. ¹³C NMR spectrum of 1-(4-Bromophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ah**).

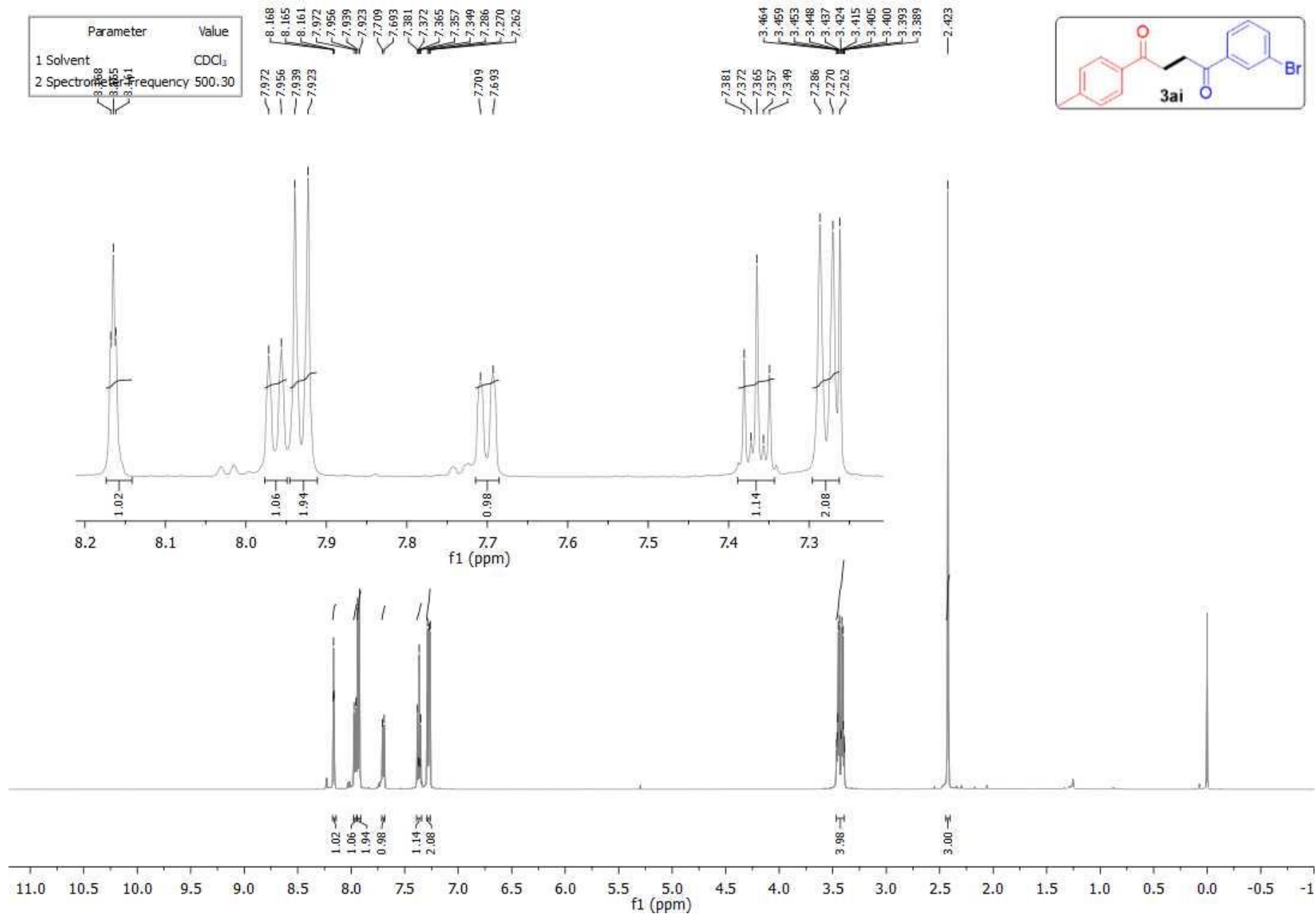


Figure S20. ¹H NMR spectrum of 1-(3-Bromophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ai**).

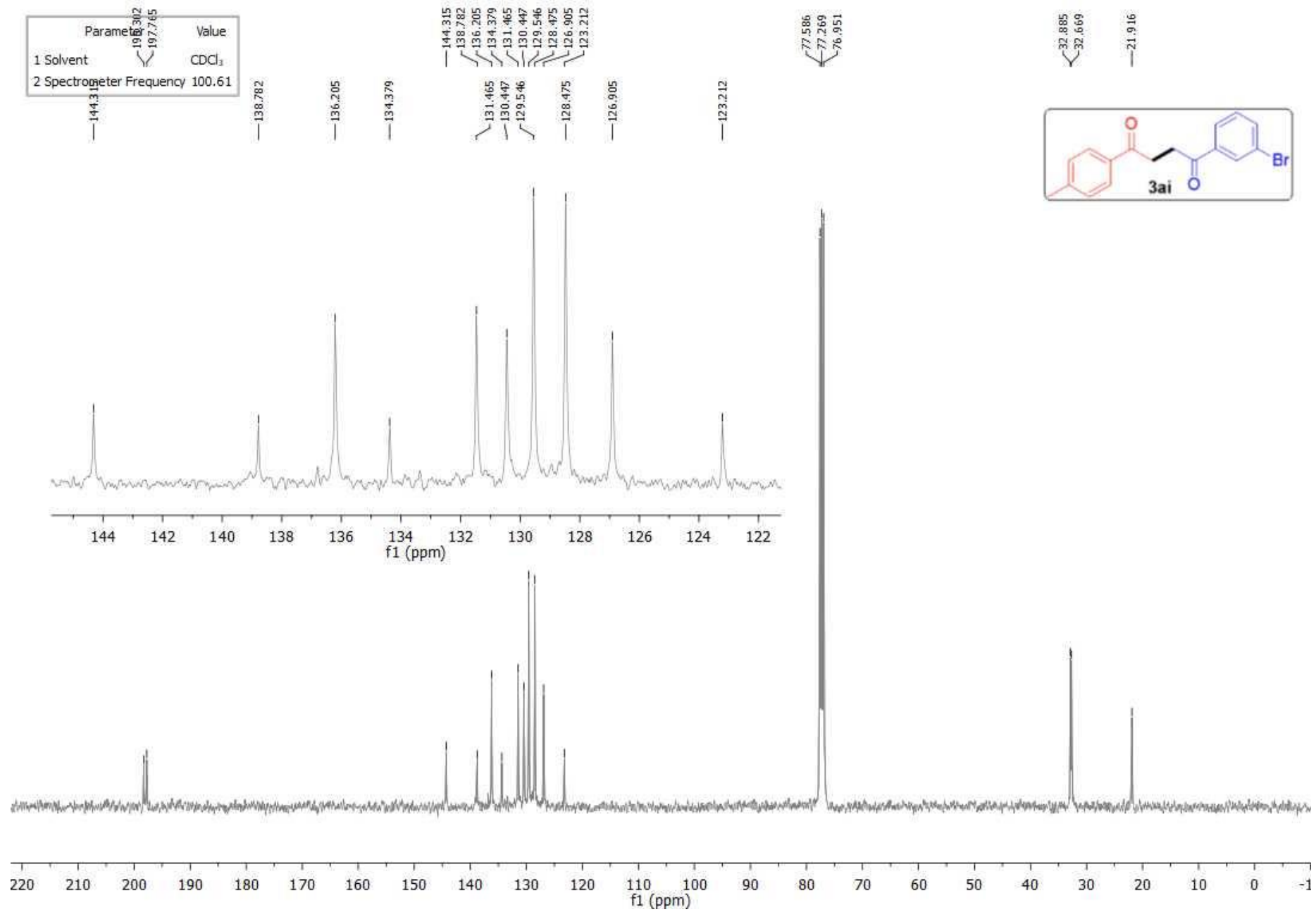


Figure S21. ¹³C NMR spectrum of 1-(3-Bromophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ai**).

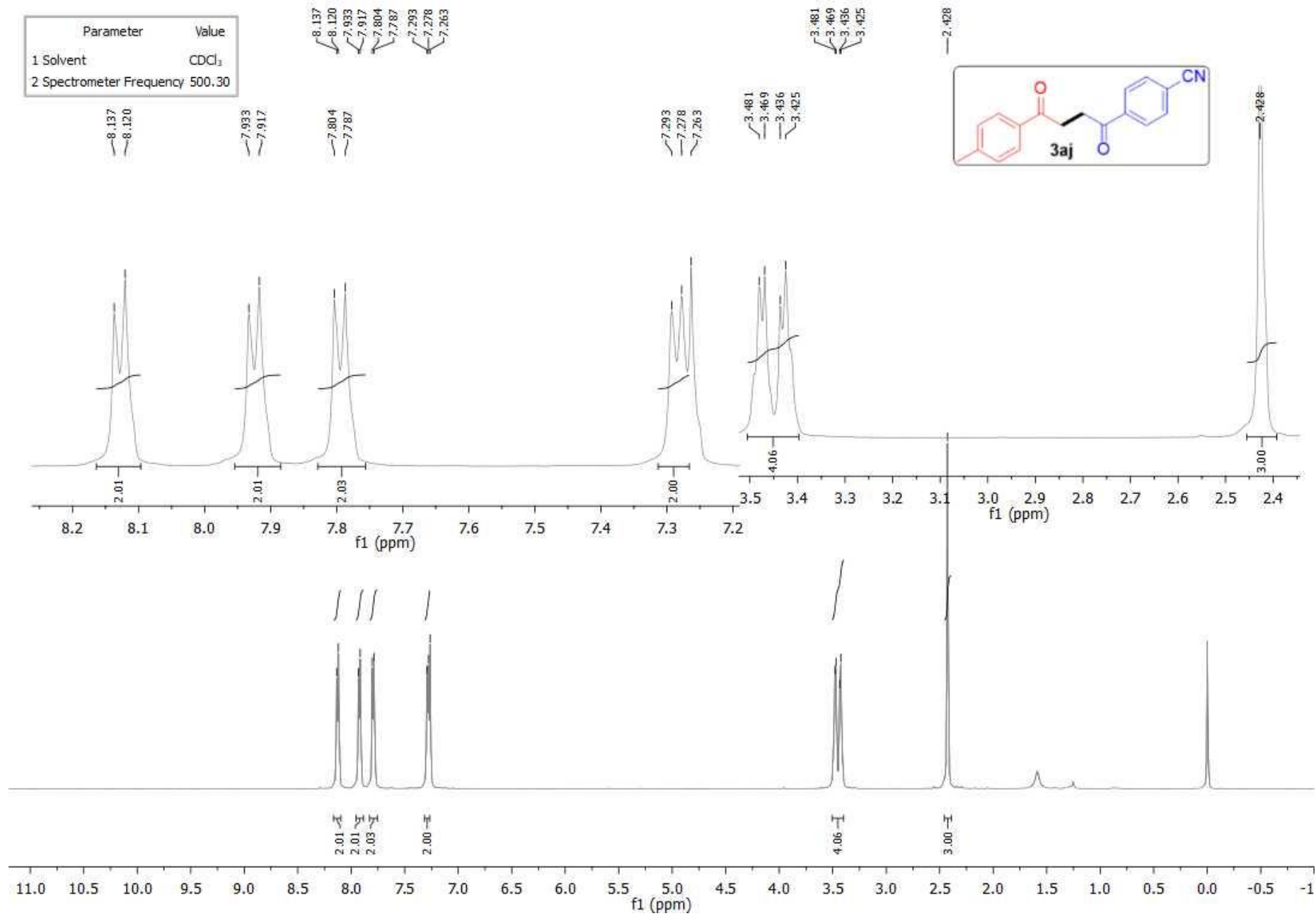


Figure S22. ¹H NMR spectrum of 4-(4-Oxo-4-(*p*-tolyl)butanoyl)benzonitrile (**3aj**).

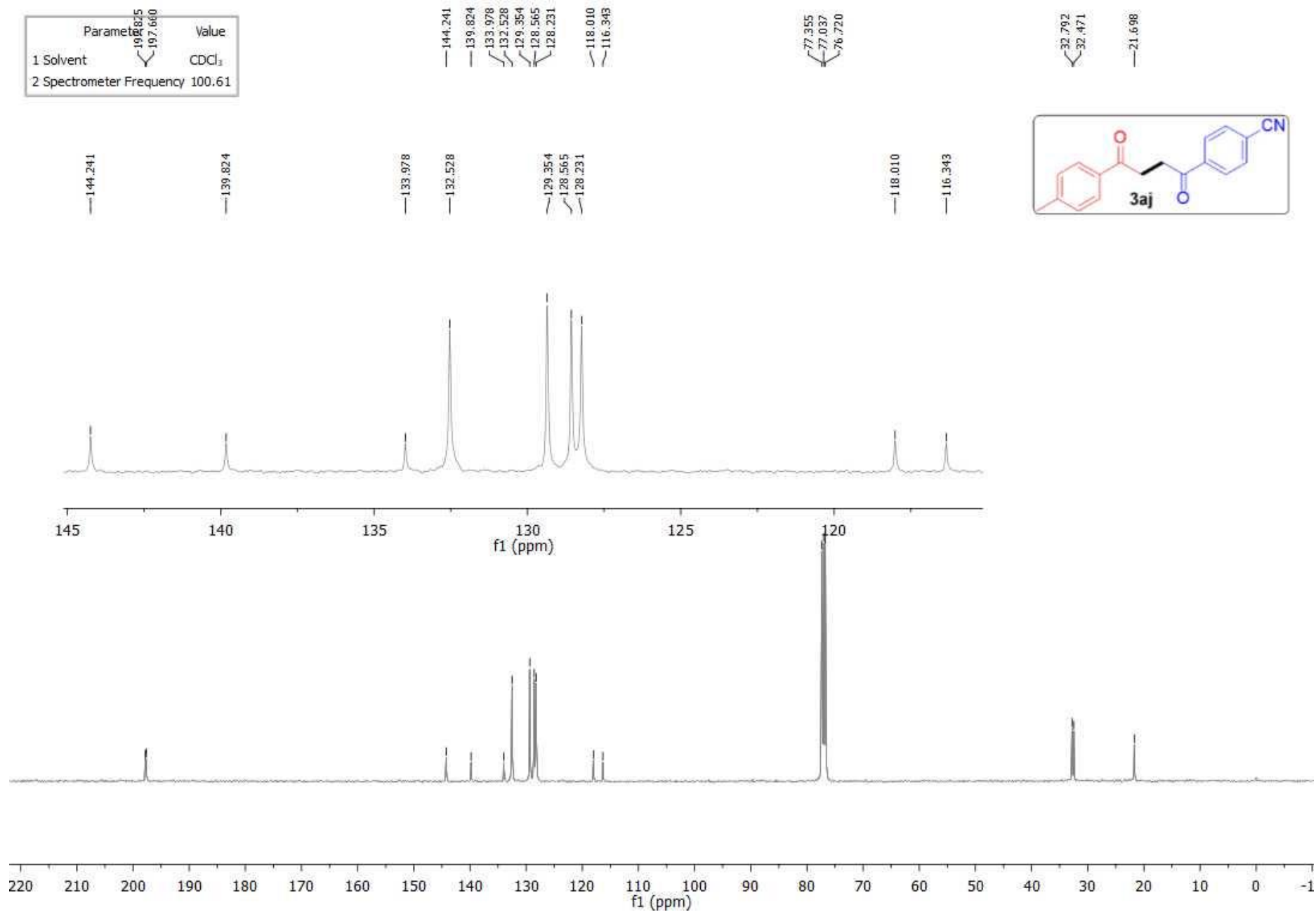


Figure S23. ¹³C NMR ¹spectrum of 4-(4-Oxo-4-(*p*-tolyl)butanoyl)benzotrile (**3aj**).

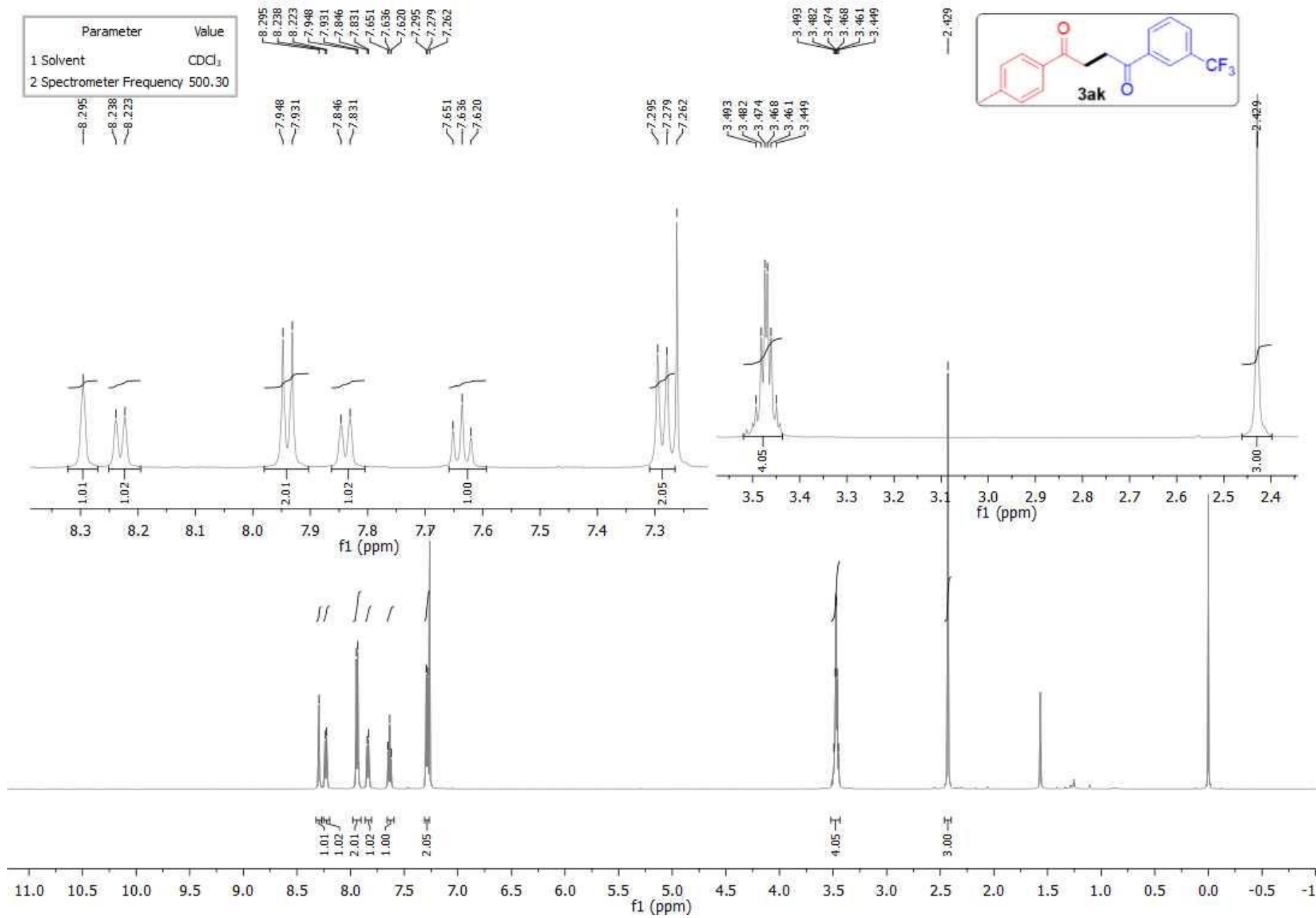


Figure S24. ¹H NMR spectrum of 1-(*p*-Tolyl)-4-(3-(trifluoromethyl)phenyl)butane-1,4-dione (**3ak**).

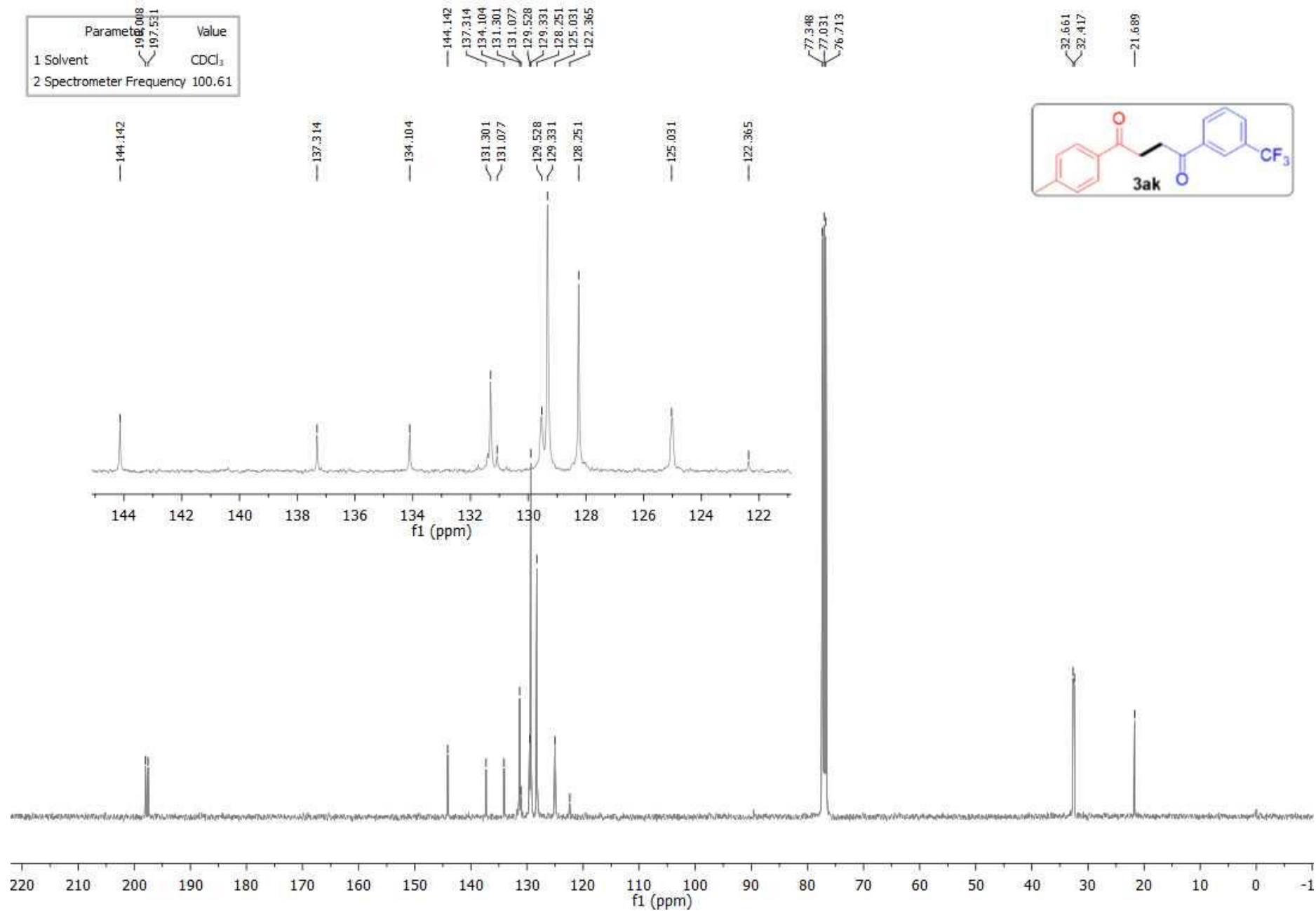


Figure S25. ¹³C NMR spectrum of 1-(*p*-Tolyl)-4-(3-(trifluoromethyl)phenyl)butane-1,4-dione (**3ak**).

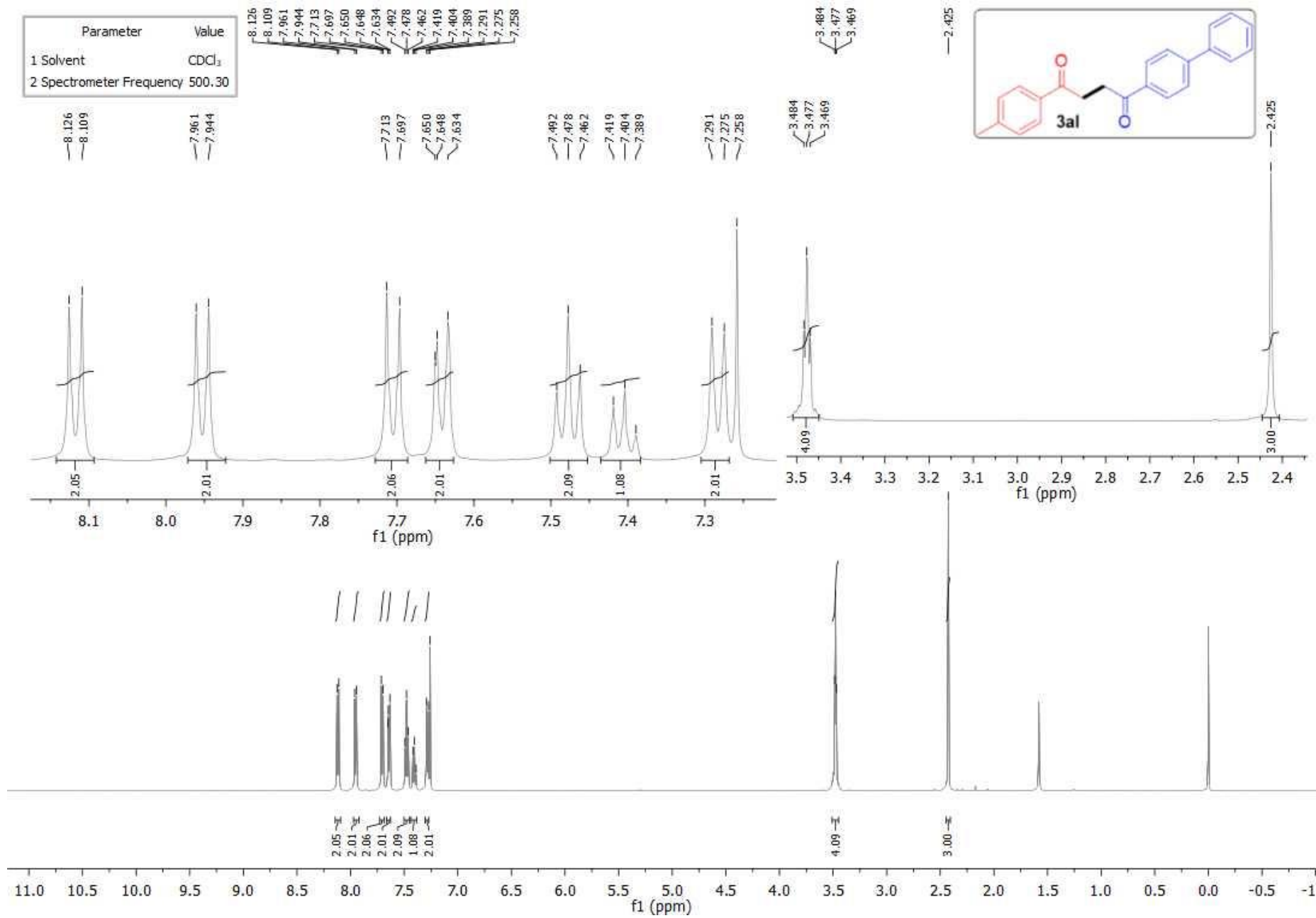


Figure S26. ¹H NMR spectrum of 1-([1,1'-Biphenyl]-4-yl)-4-(p-tolyl)butane-1,4-dione (**3al**).

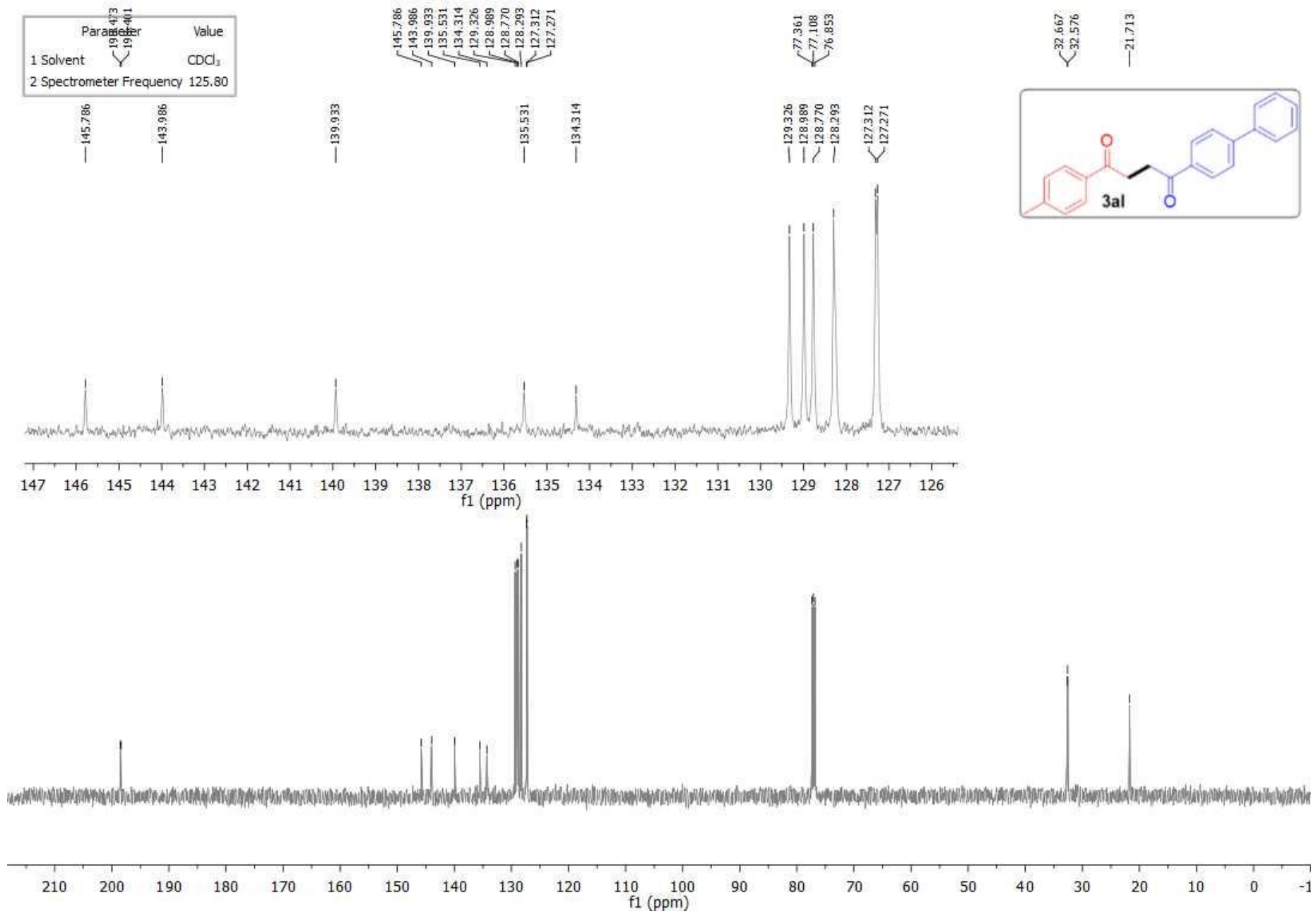


Figure S27. ¹³C NMR spectrum of 1-([1,1'-Biphenyl]-4-yl)-4-(p-tolyl)butane-1,4-dione (**3al**).

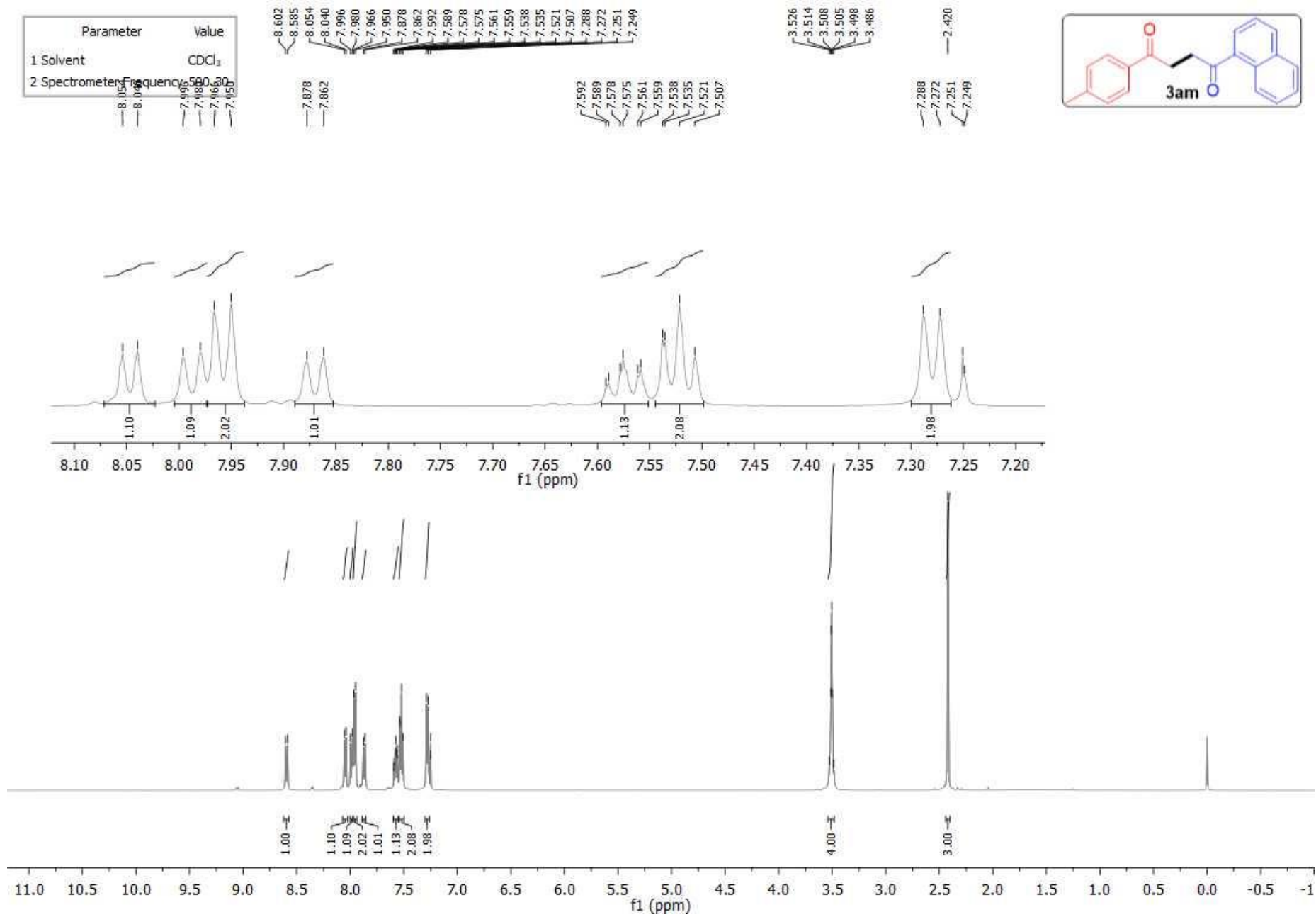


Figure S28. ¹H NMR spectrum of 1-(Naphthalen-1-yl)-4-(*p*-tolyl)butane-1,4-dione (**3am**).

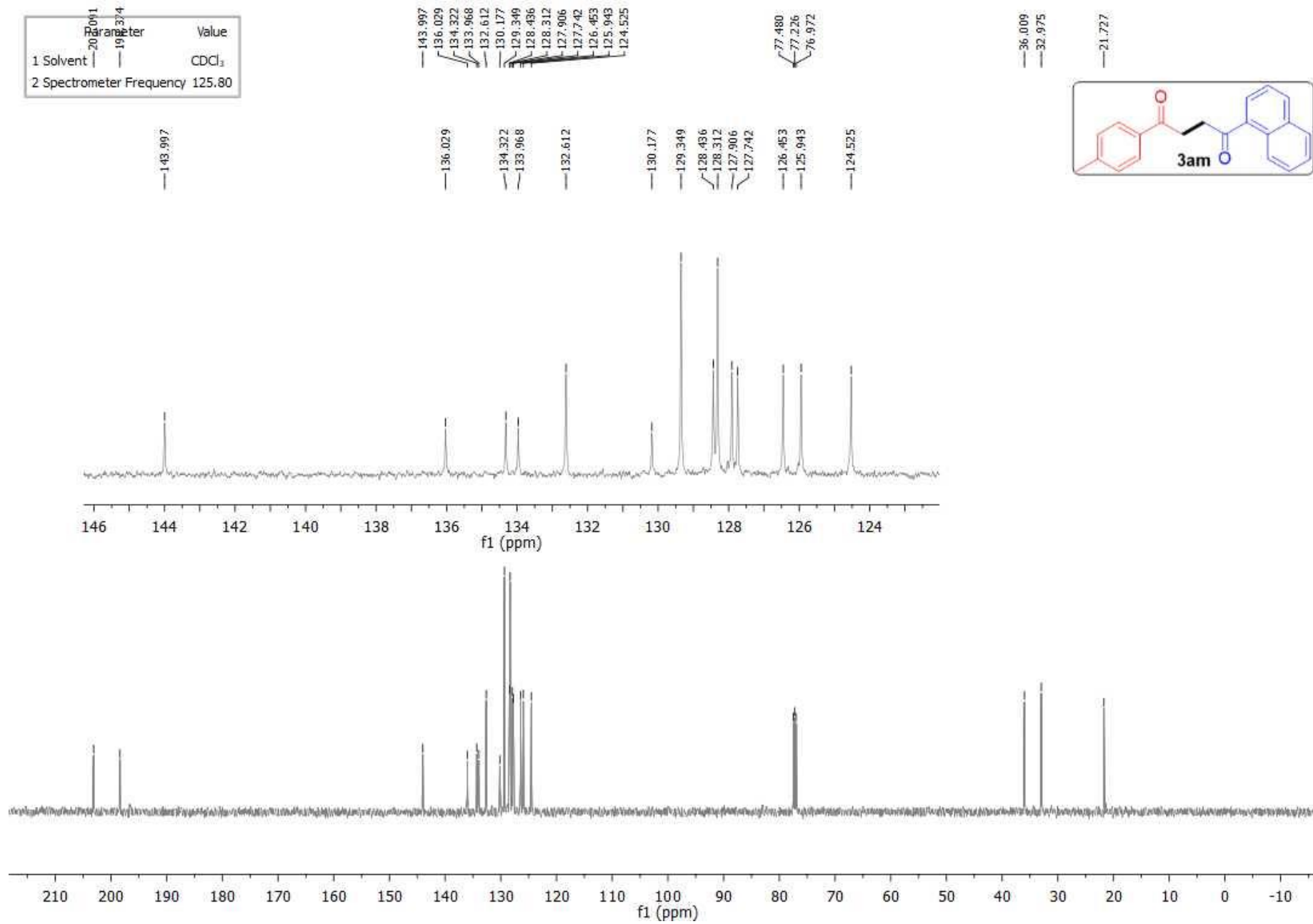


Figure S29. ¹³C NMR spectrum of 1-(Naphthalen-1-yl)-4-(*p*-tolyl)butane-1,4-dione (**3am**).

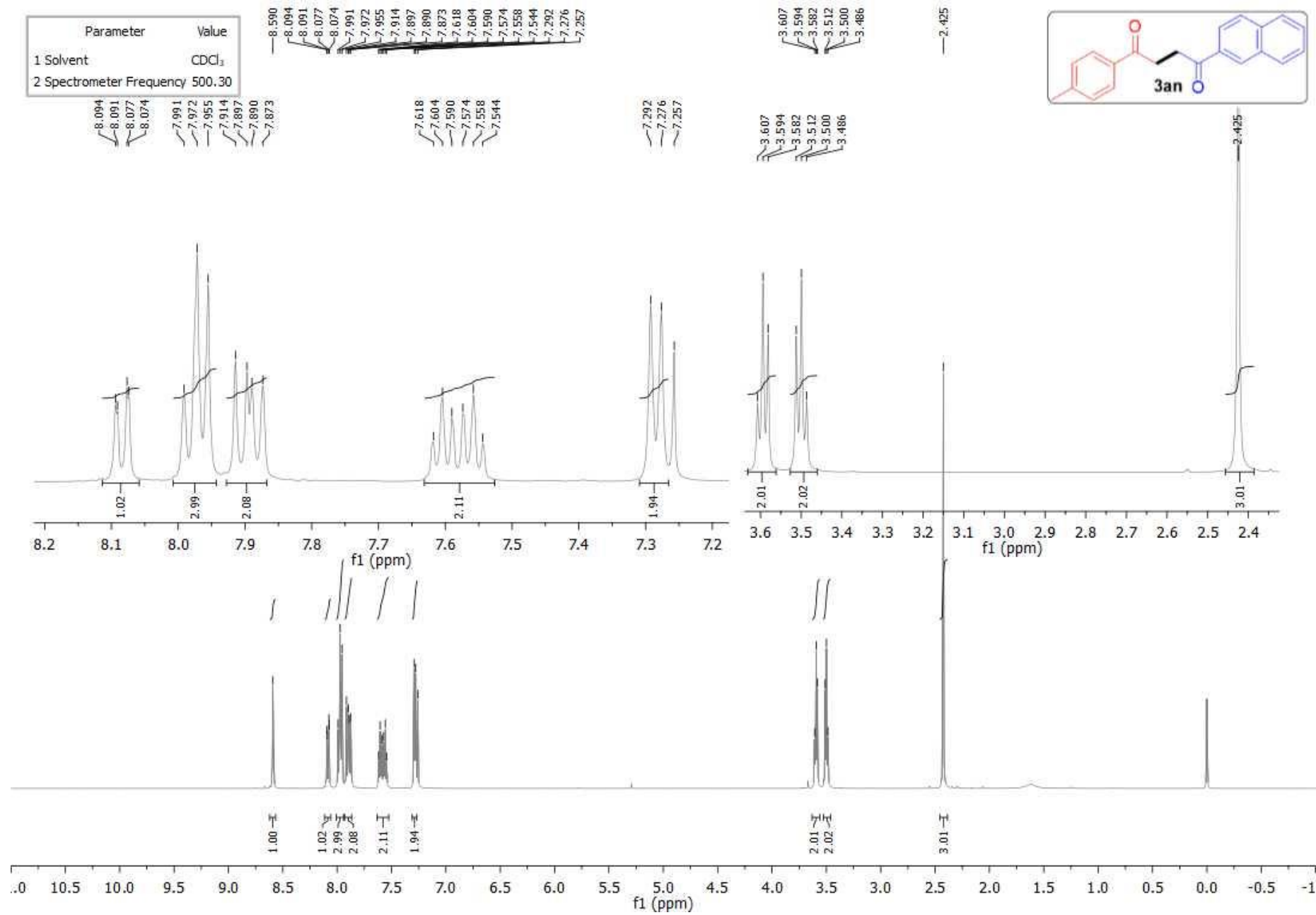


Figure S30. ¹H NMR spectrum of 1-(Naphthalen-2-yl)-4-(*p*-tolyl)butane-1,4-dione (**3an**).

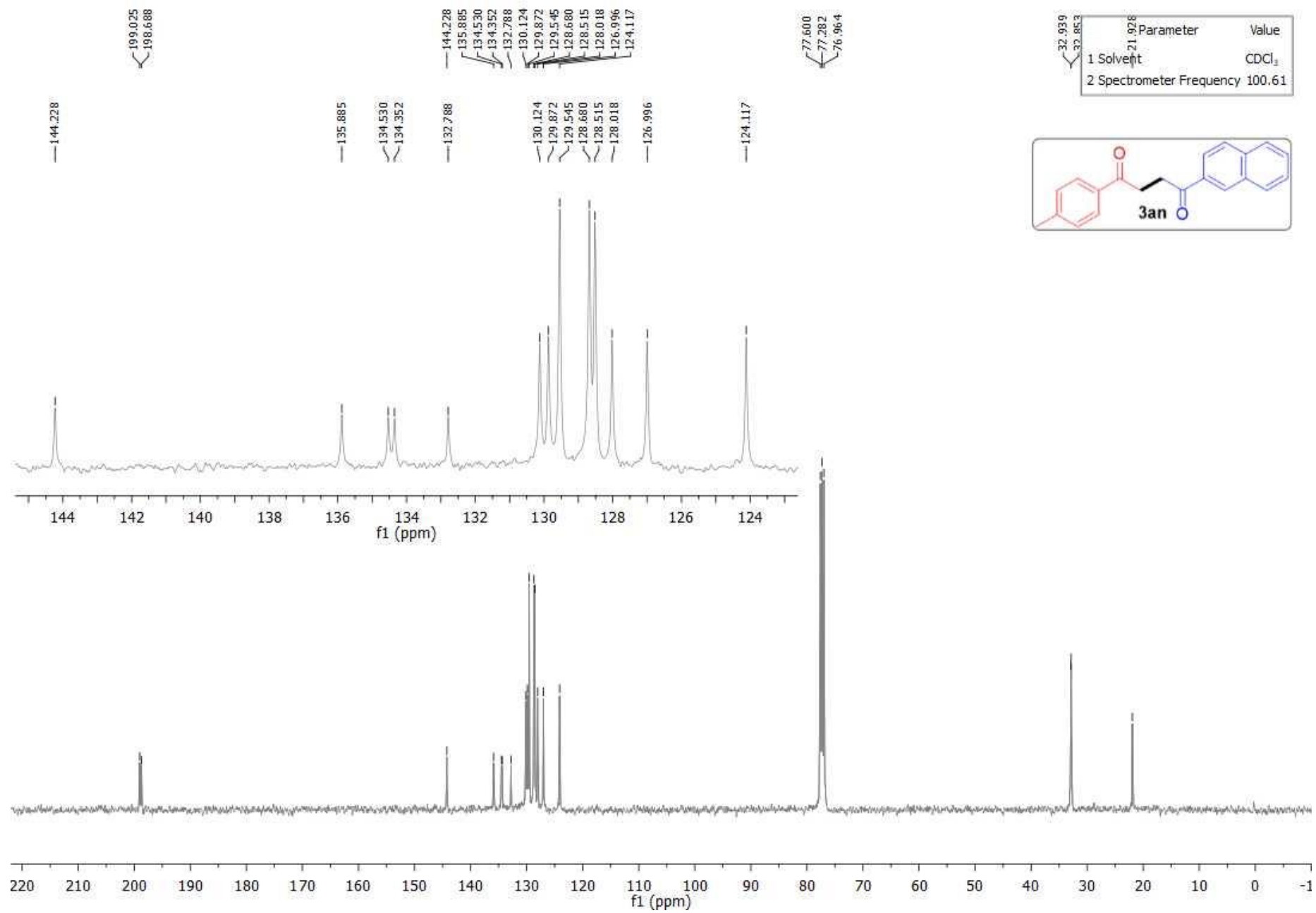


Figure S31. ¹³C NMR spectrum of 1-(Naphthalen-2-yl)-4-(*p*-tolyl)butane-1,4-dione (**3an**).

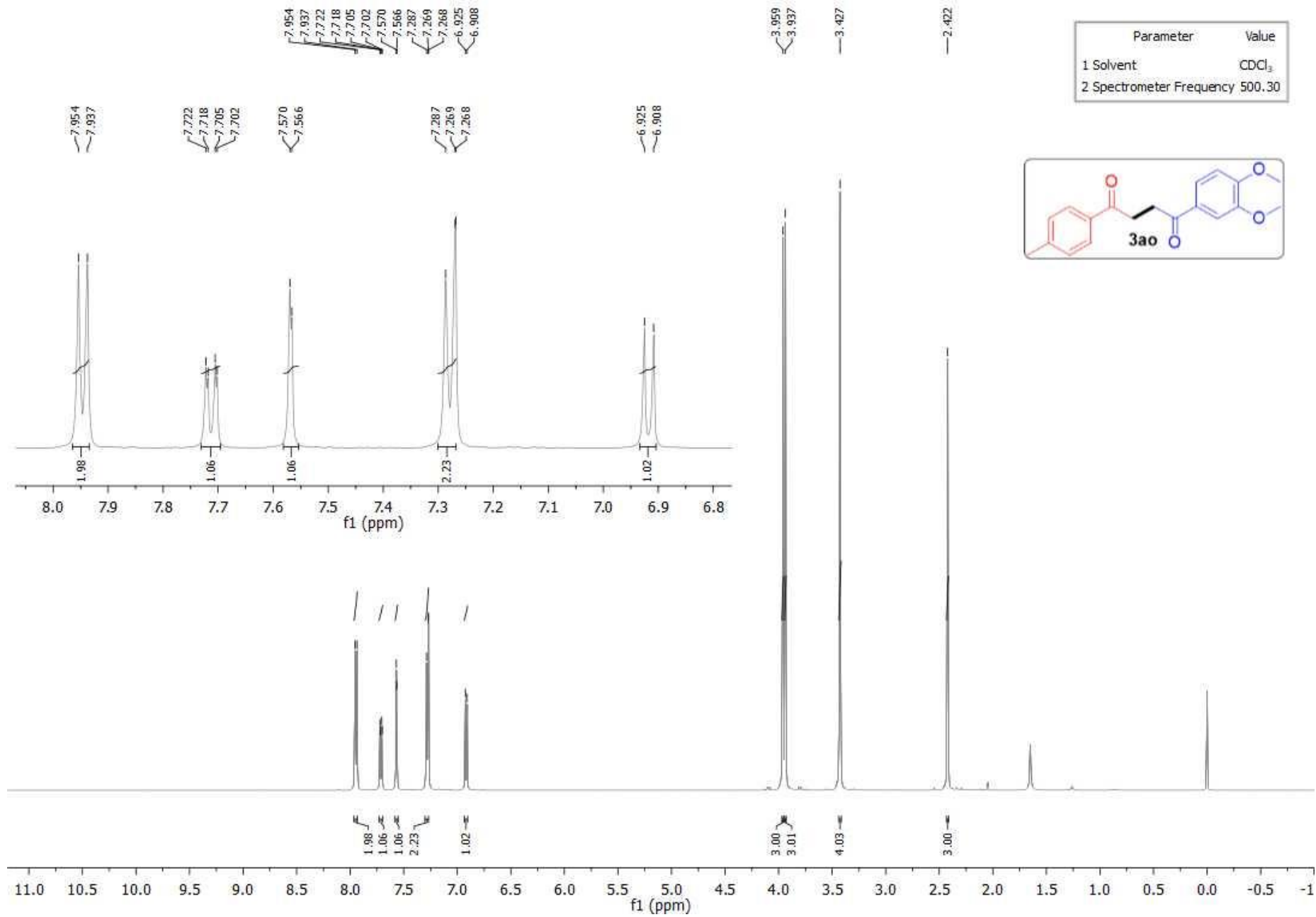


Figure S32. ¹H NMR spectrum of 1-(3,4-Dimethoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ao**).

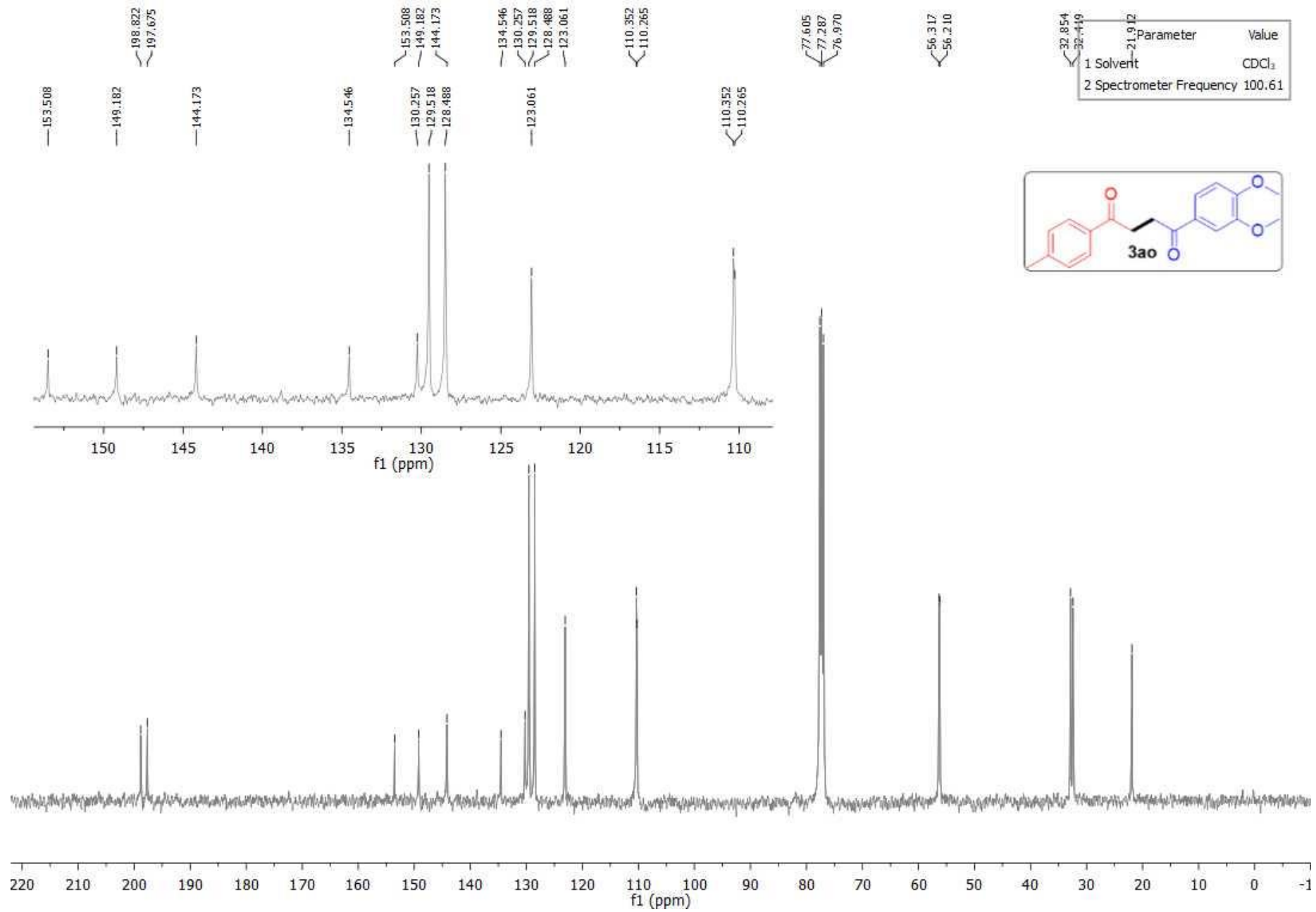


Figure S33. ¹³C NMR spectrum of 1-(3,4-Dimethoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ao**).

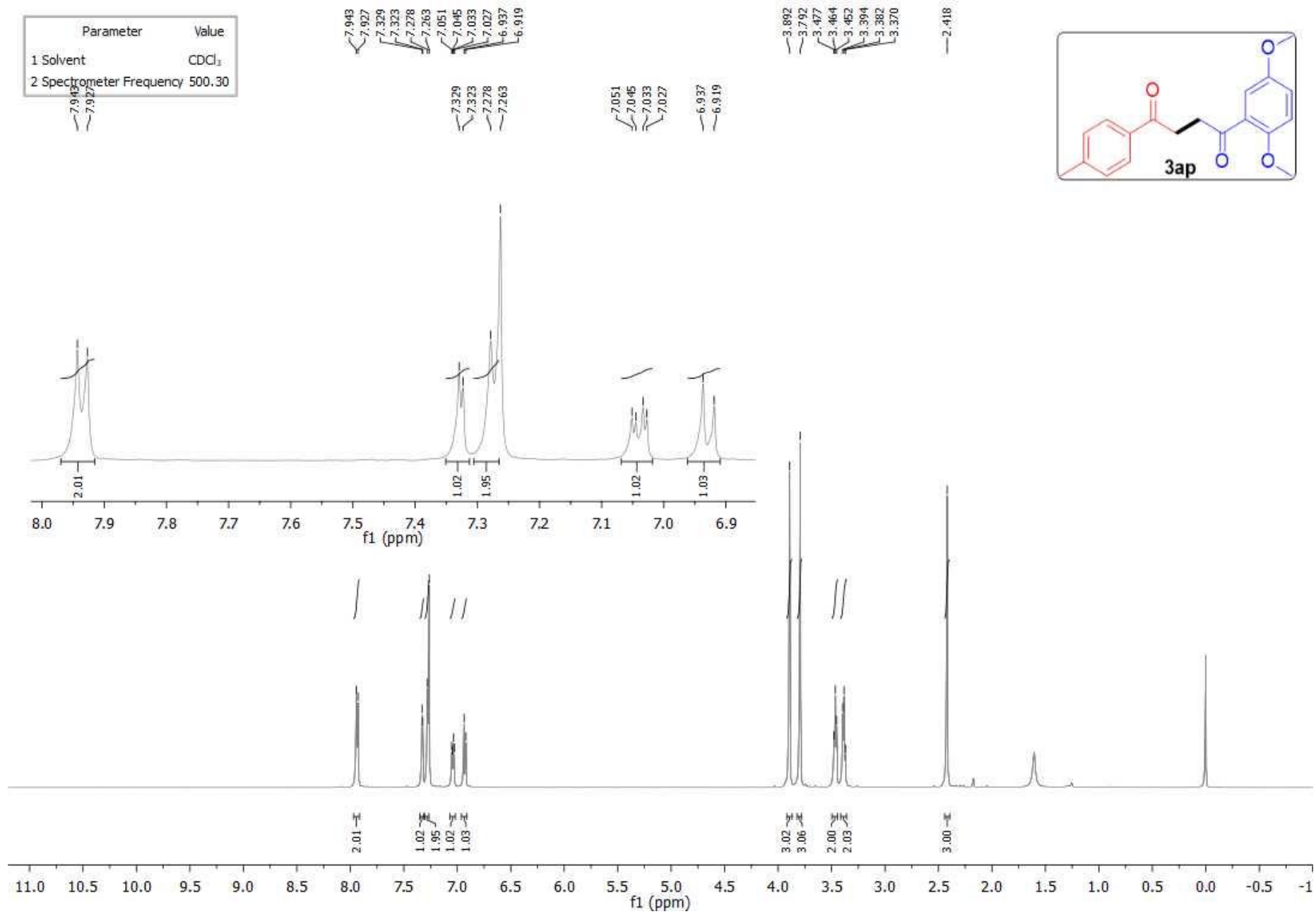


Figure S34. ¹H NMR spectrum of 1-(2,5-dimethoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ap**).

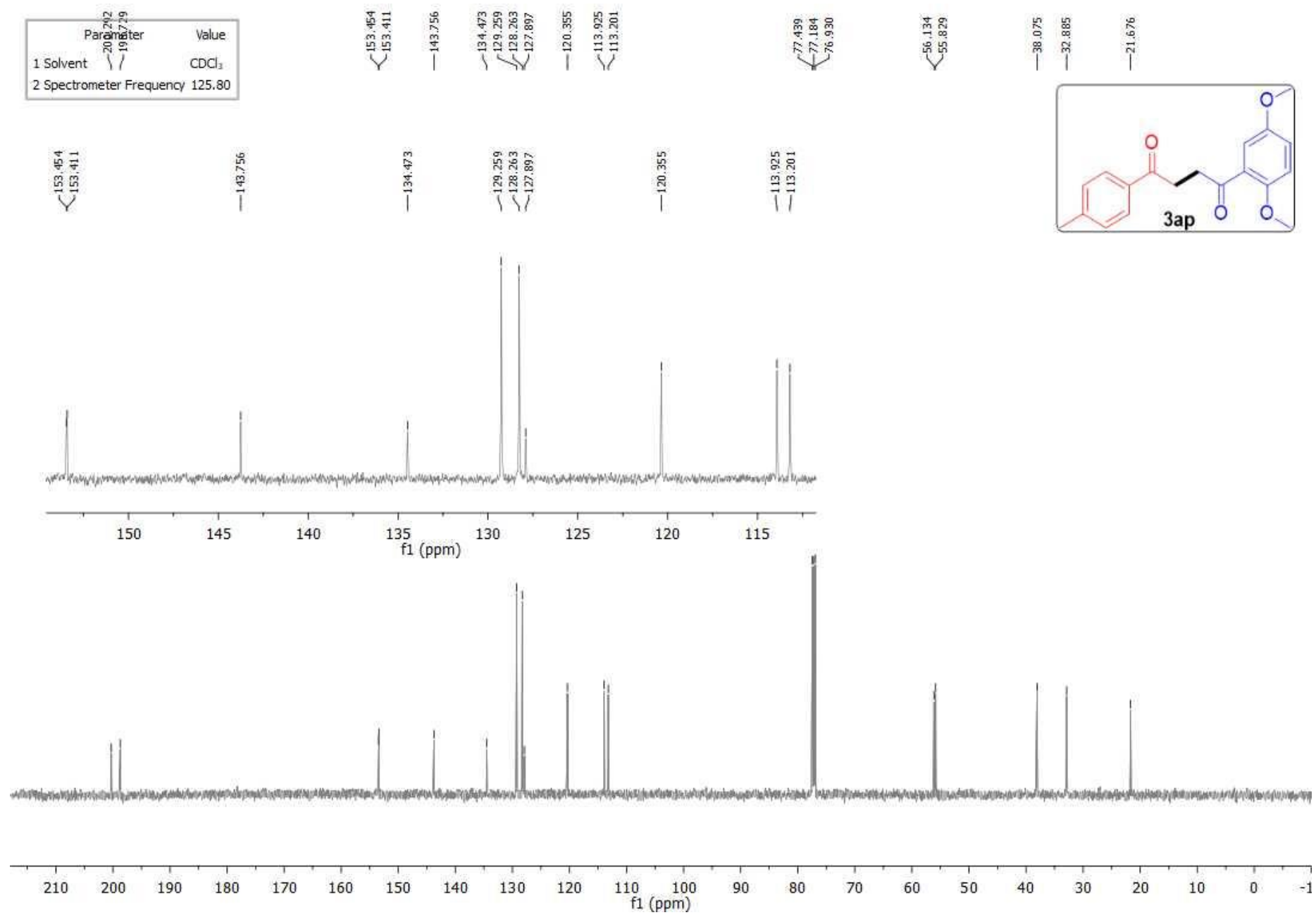


Figure S35. ¹³C NMR spectrum of 1-(2,5-dimethoxyphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ap**).

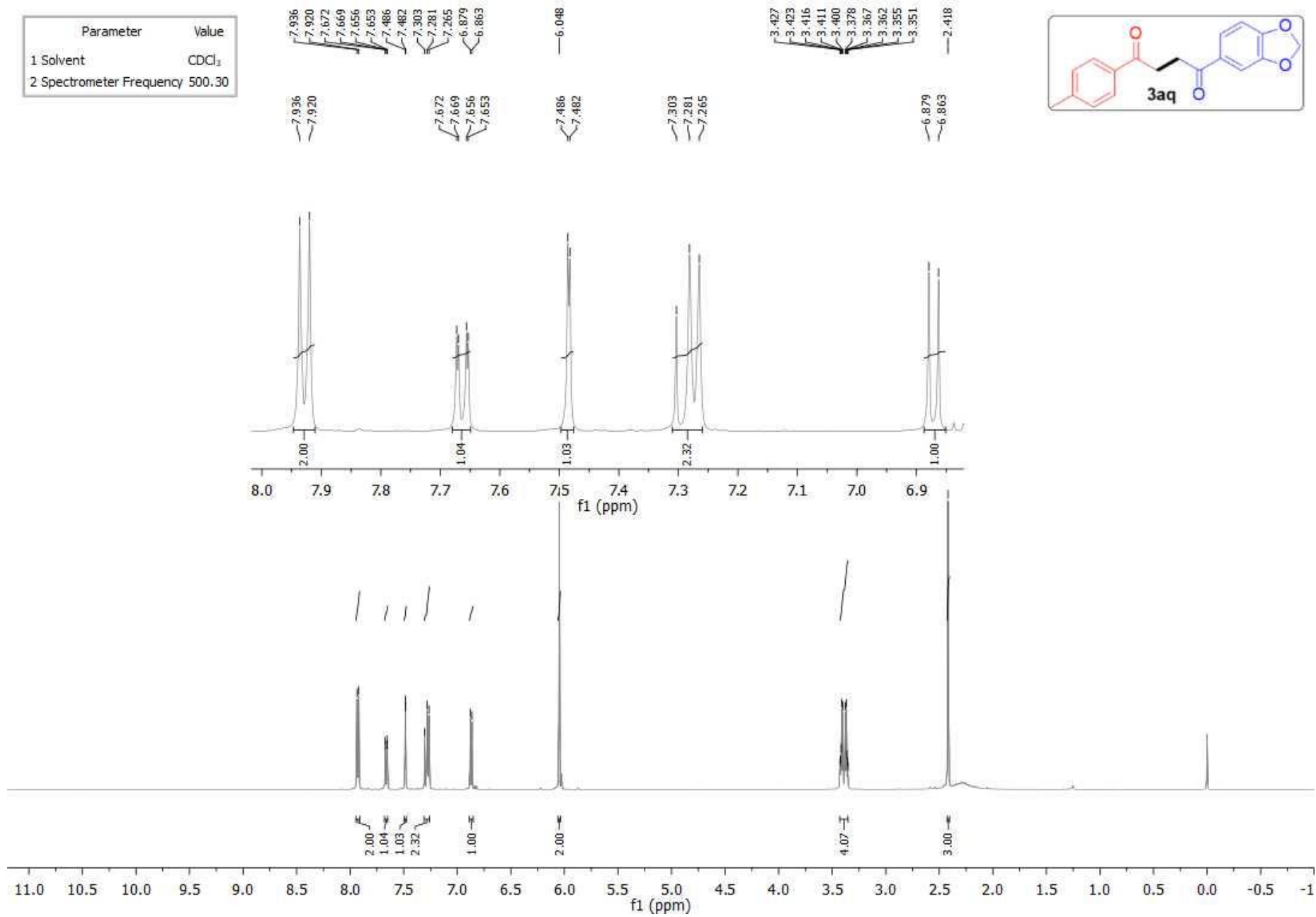


Figure S36. ¹H NMR spectrum of 1-(Benzo[*d*][1,3]dioxol-5-yl)-4-(*p*-tolyl)butane-1,4-dione (**3aq**).

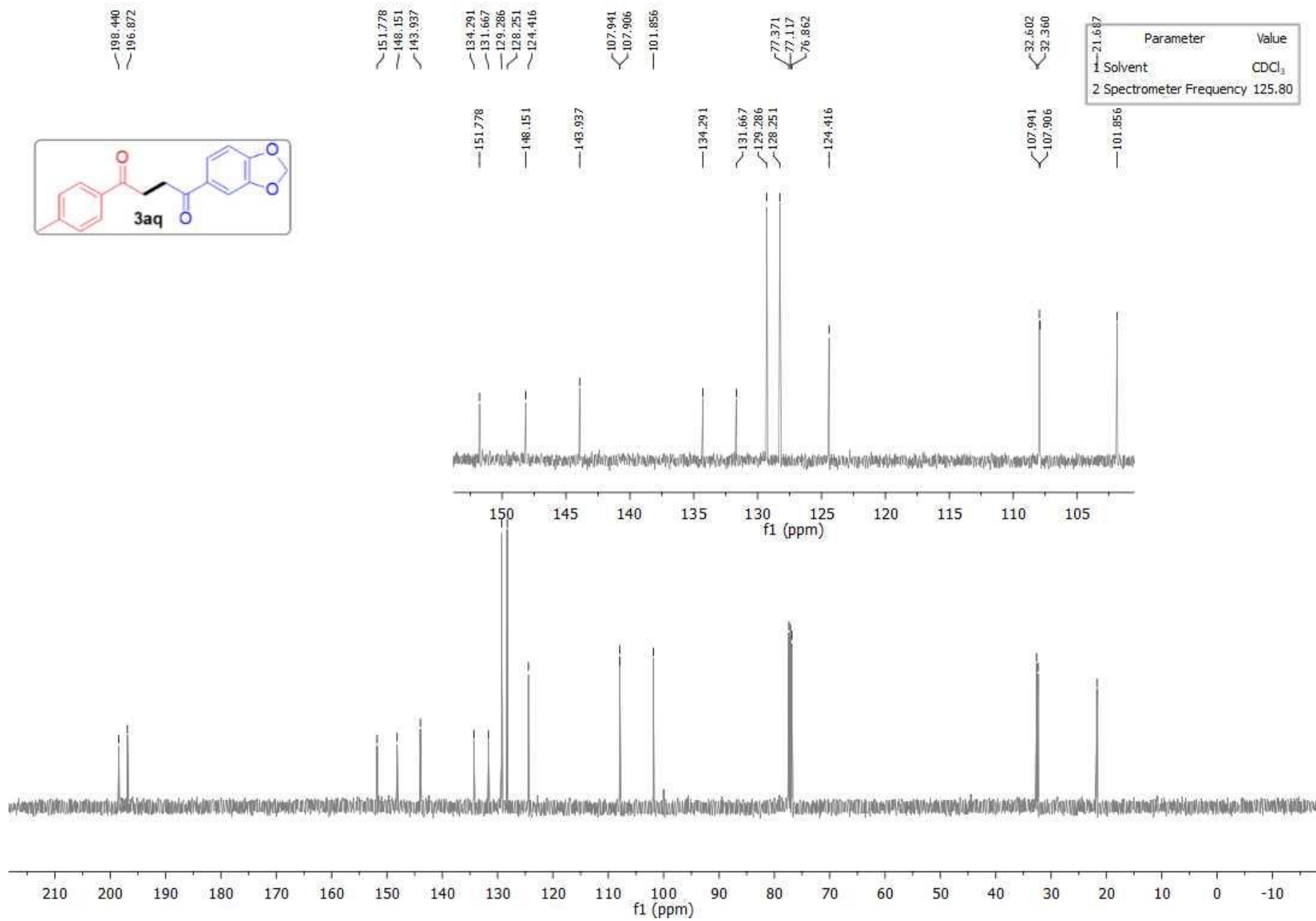


Figure S37. ¹³C NMR spectrum of 1-(Benzo[*d*][1,3]dioxol-5-yl)-4-(*p*-tolyl)butane-1,4-dione (**3aq**).

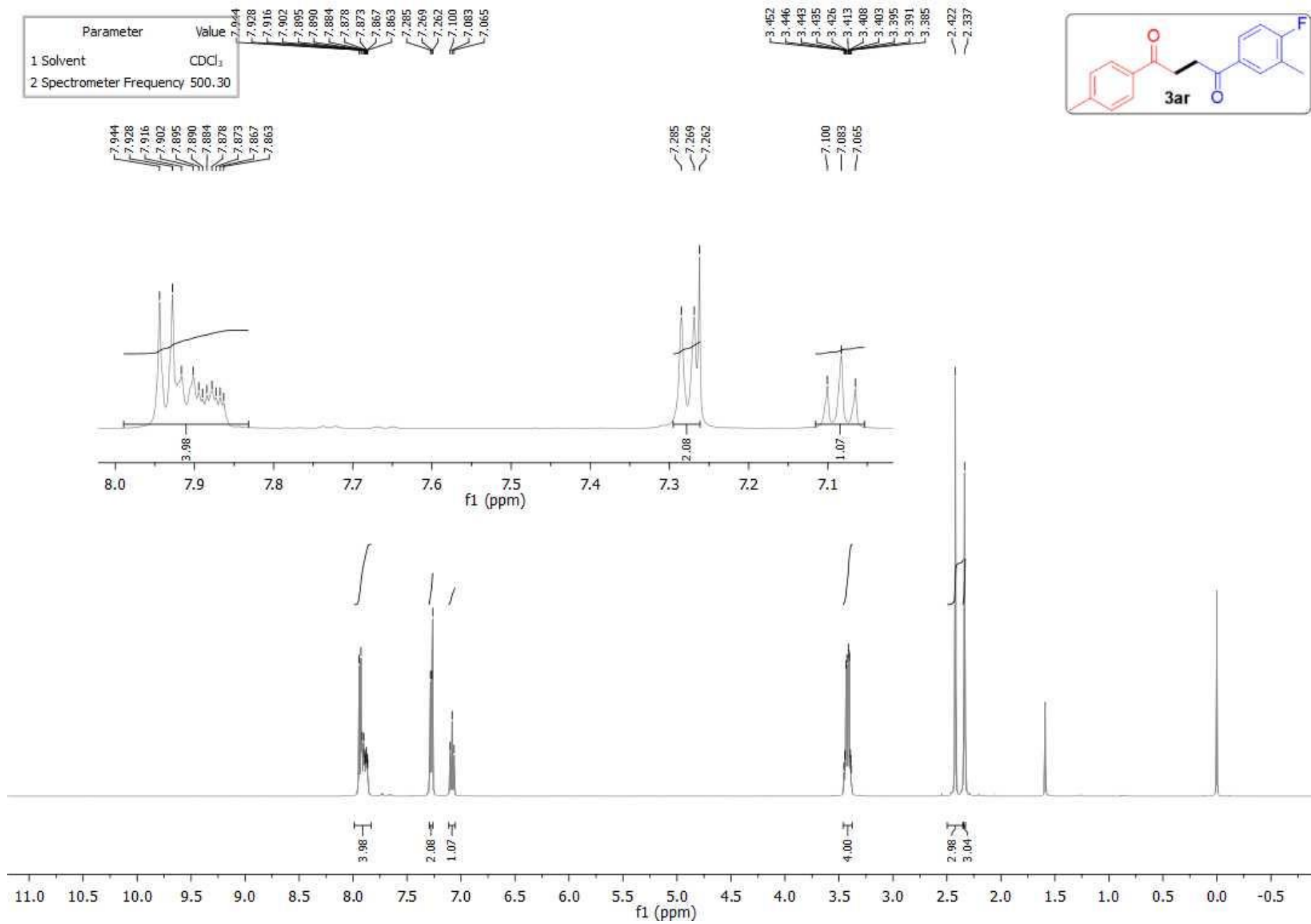


Figure S38. ¹H NMR spectrum of 1-(4-Fluoro-3-methylphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ar**).

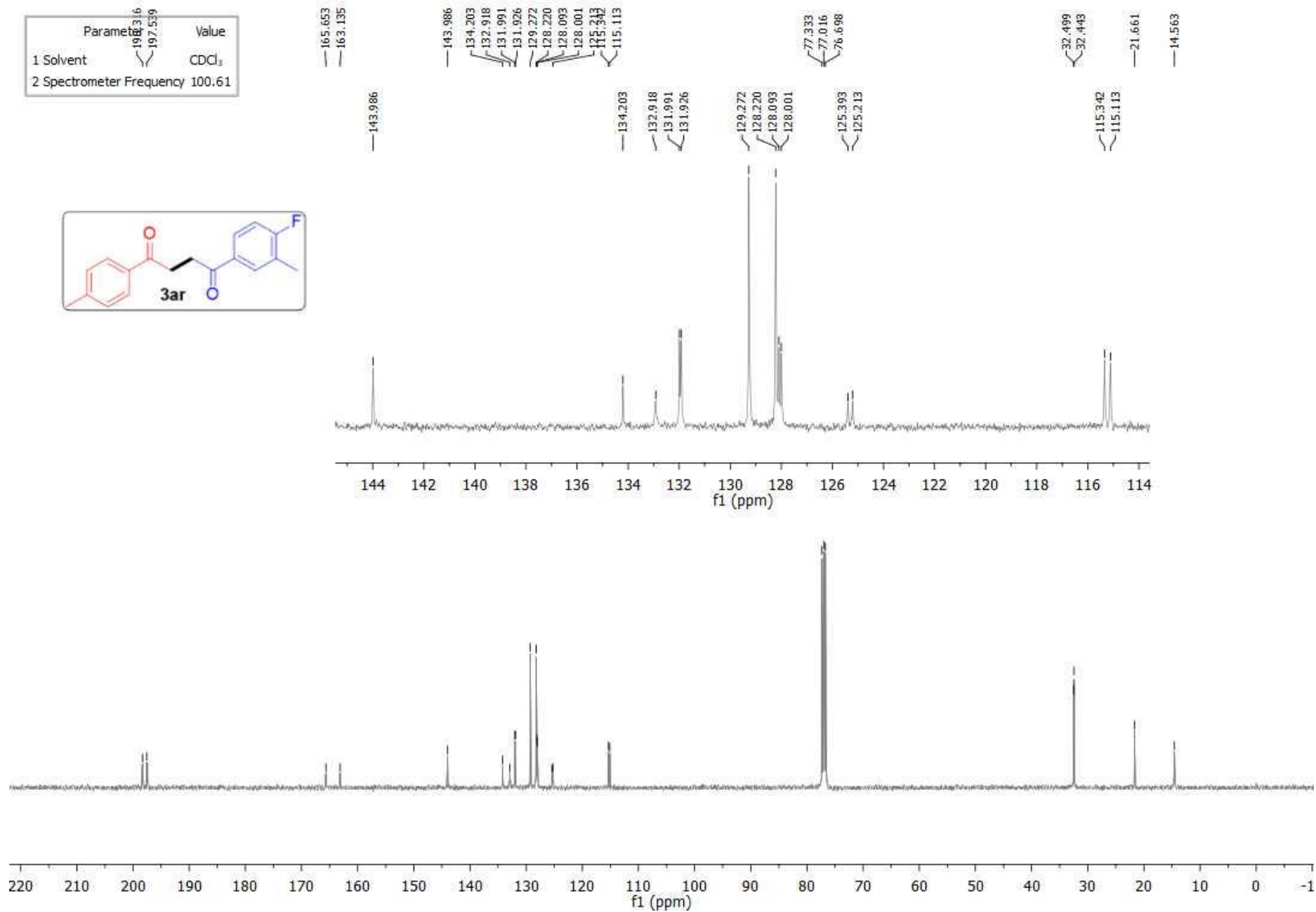


Figure S39. ¹³C NMR spectrum of 1-(4-Fluoro-3-methylphenyl)-4-(*p*-tolyl)butane-1,4-dione (**3ar**).

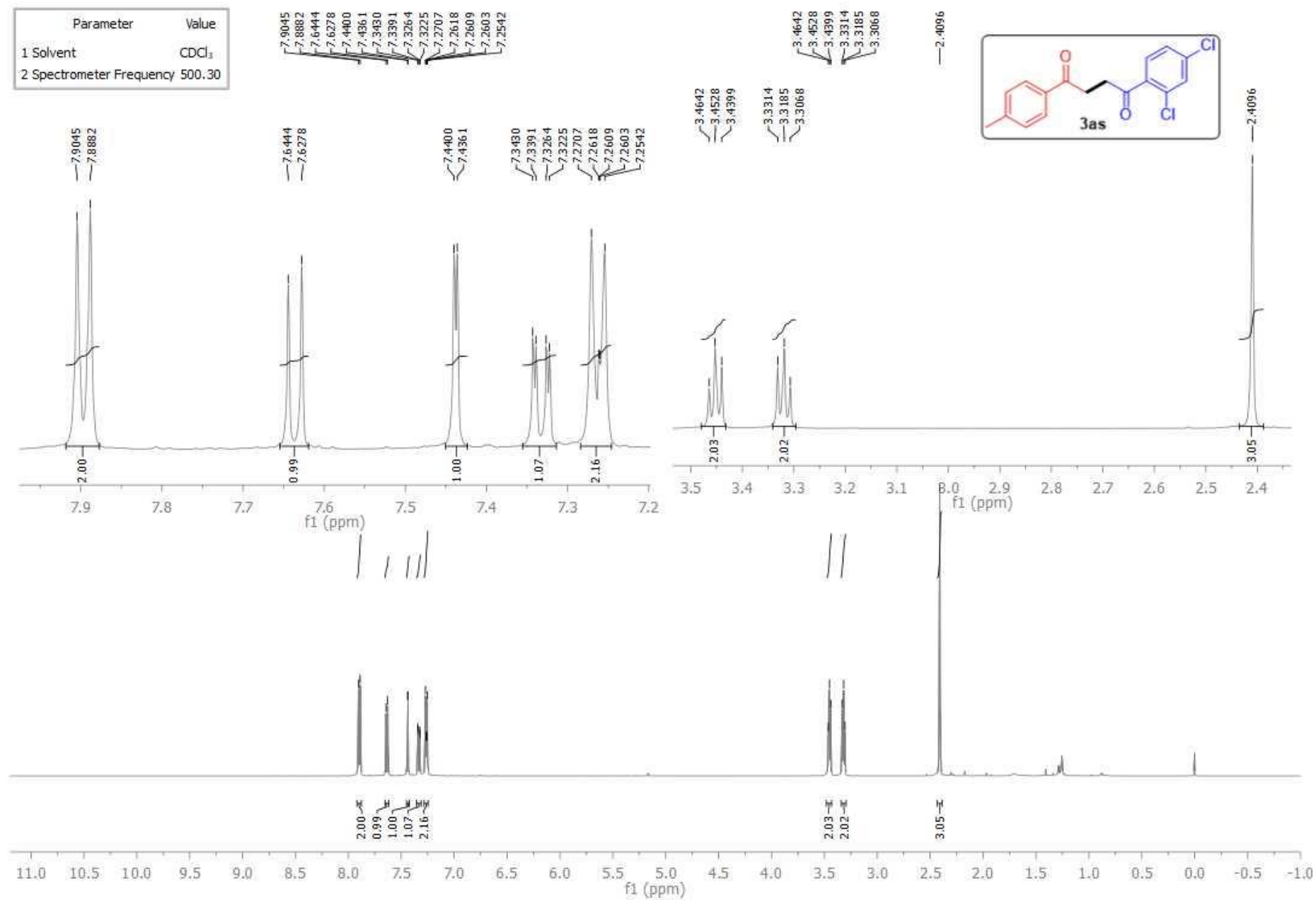


Figure S40. ¹H NMR spectrum of 1-(2,4-Dichlorophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3as**).

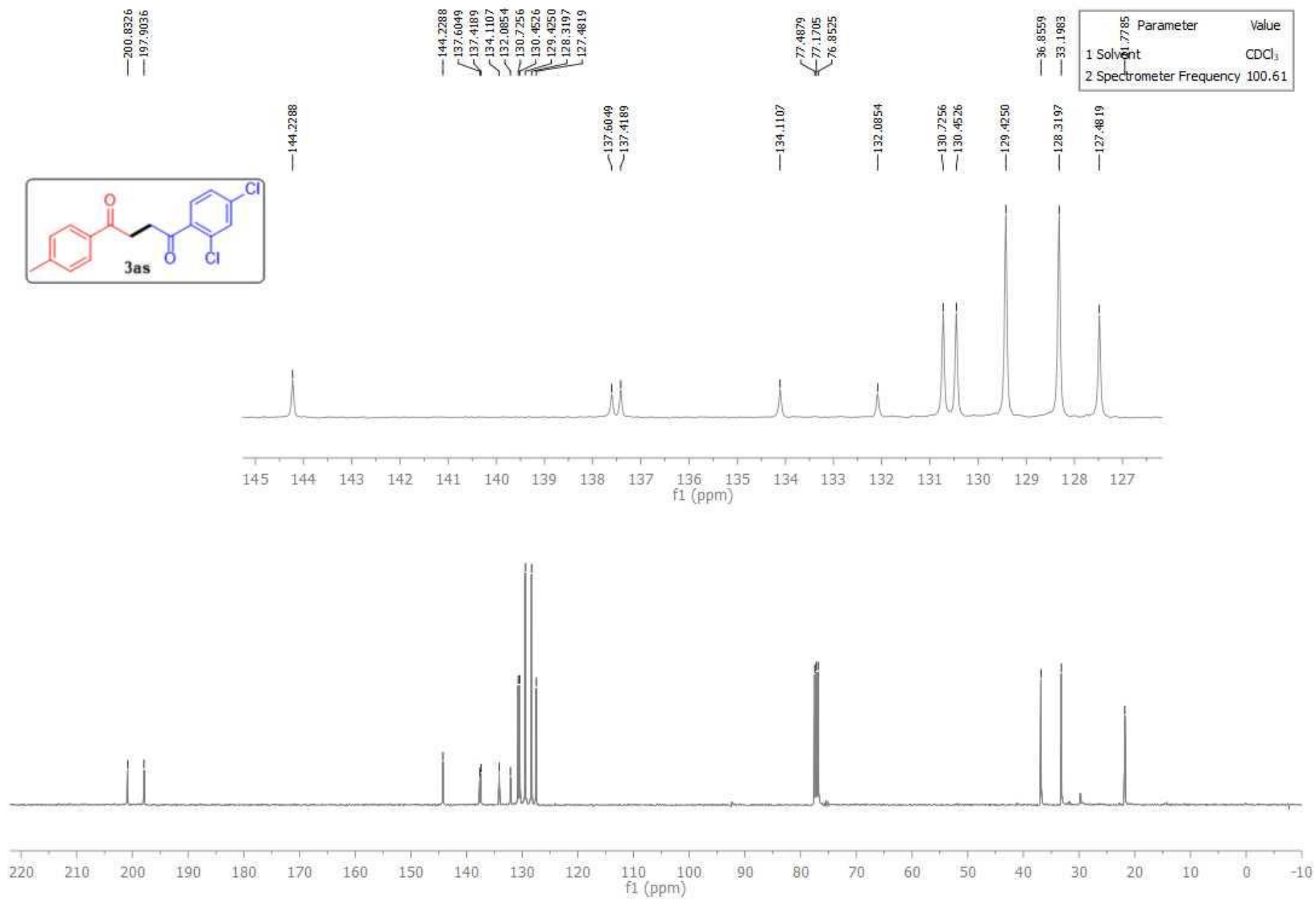


Figure S41. ¹³C NMR spectrum of 1-(2,4-Dichlorophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3as**).

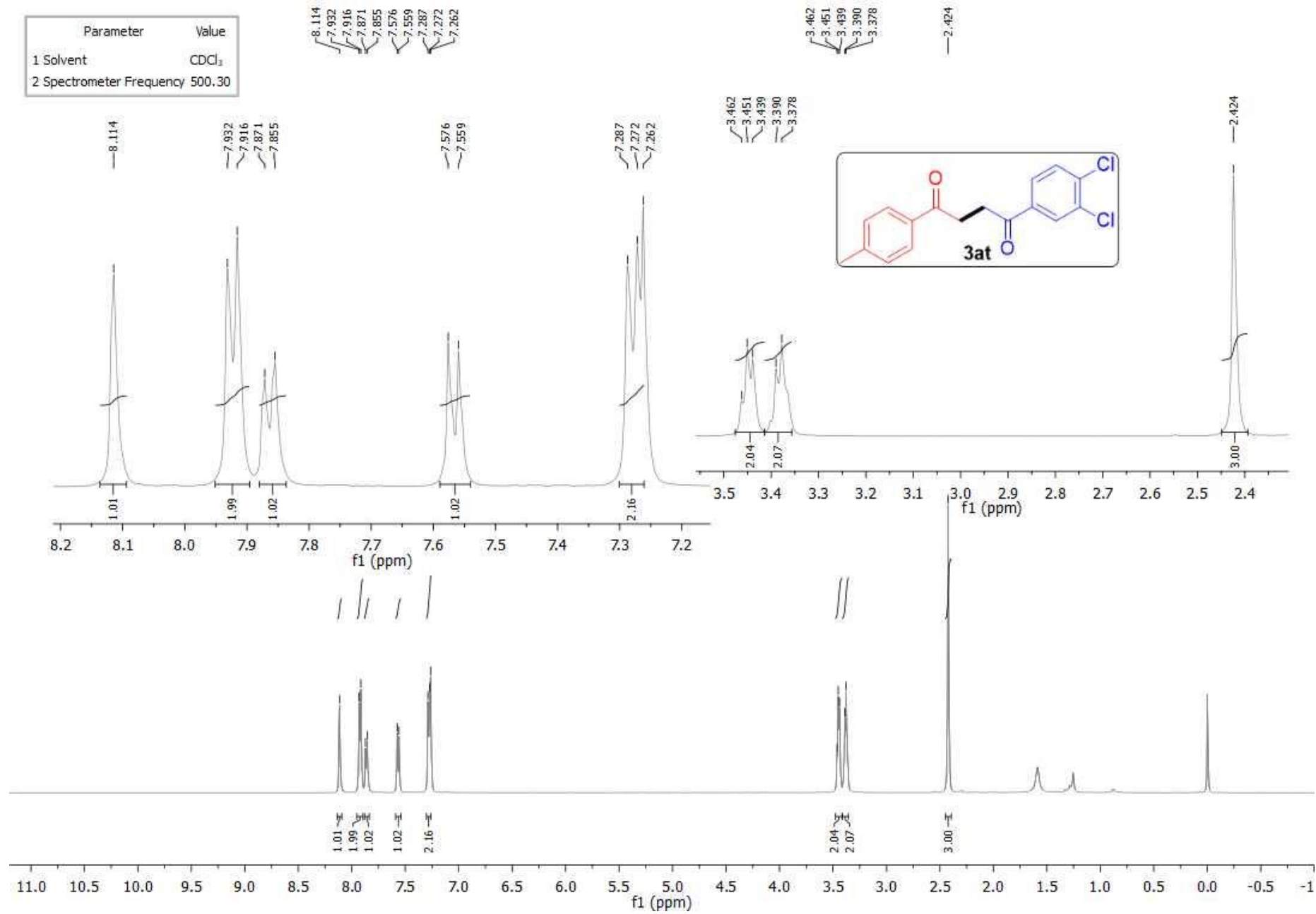


Figure S42. ¹H NMR spectrum of 1-(3,4-Dichlorophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3at**).

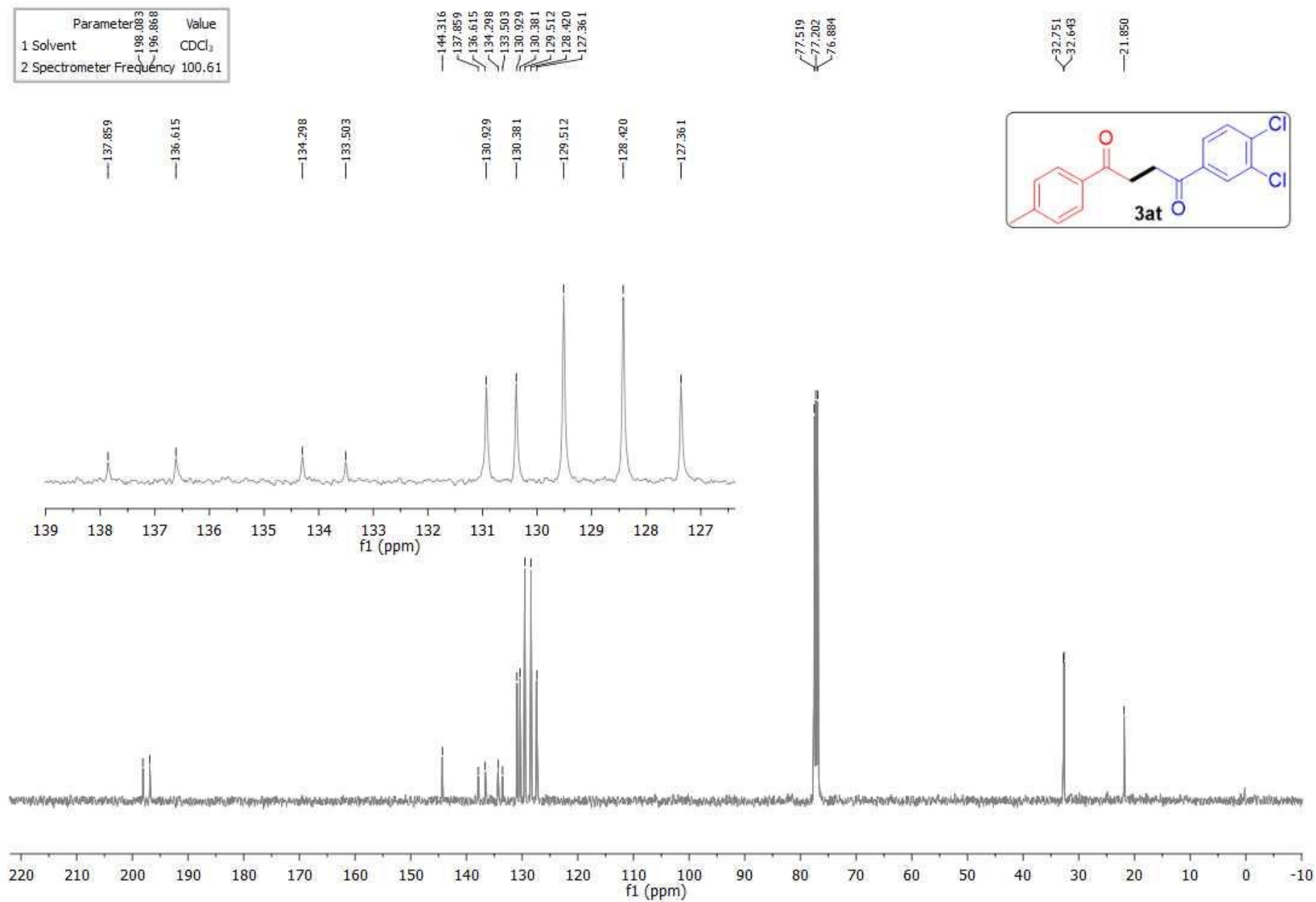


Figure S43. ¹³C NMR spectrum of 1-(3,4-Dichlorophenyl)-4-(*p*-tolyl)butane-1,4-dione (**3at**).

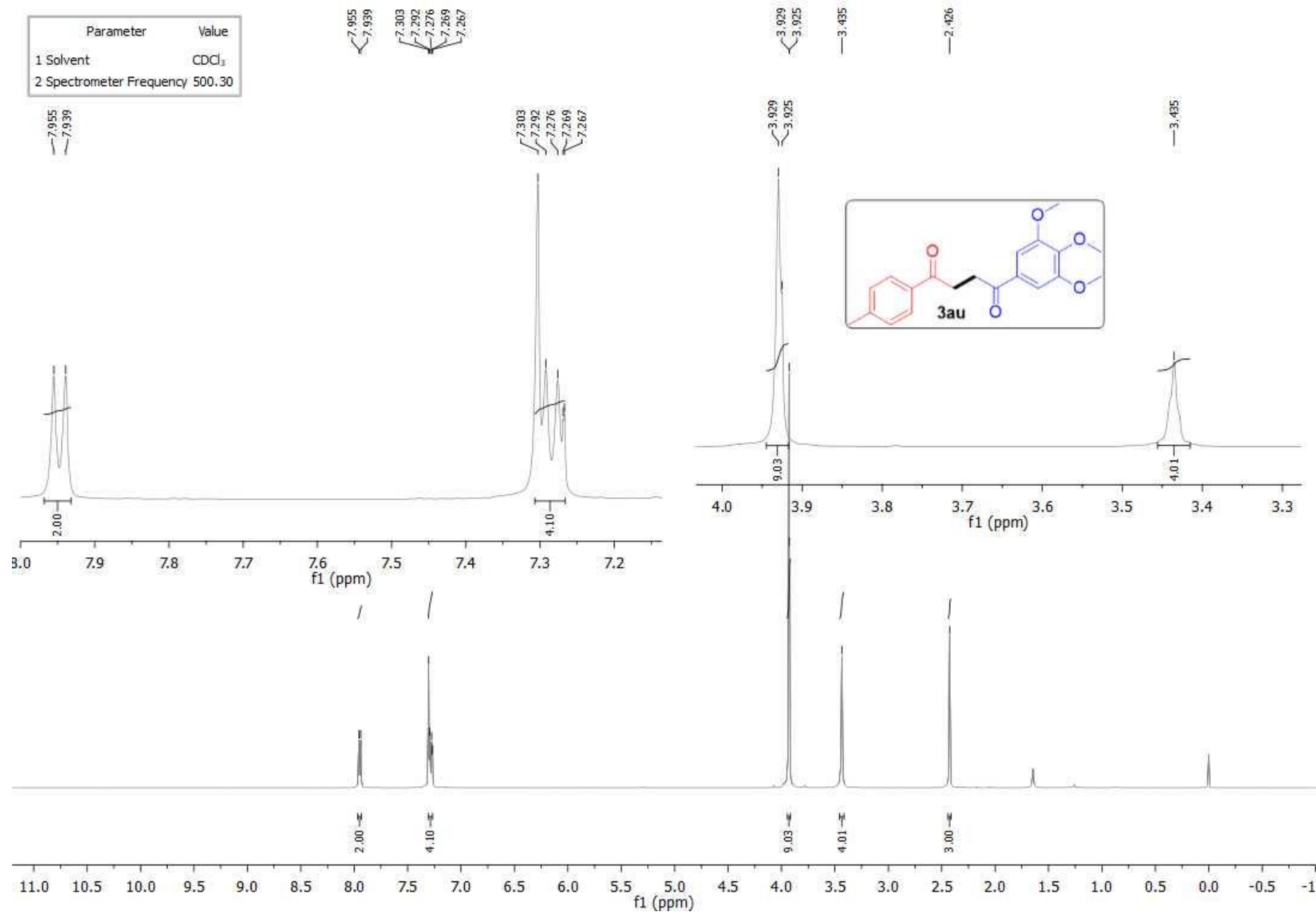


Figure S44. ¹H NMR spectrum of 1-(*p*-Tolyl)-4-(3,4,5-trimethoxyphenyl)butane-1,4-dione (**3au**).

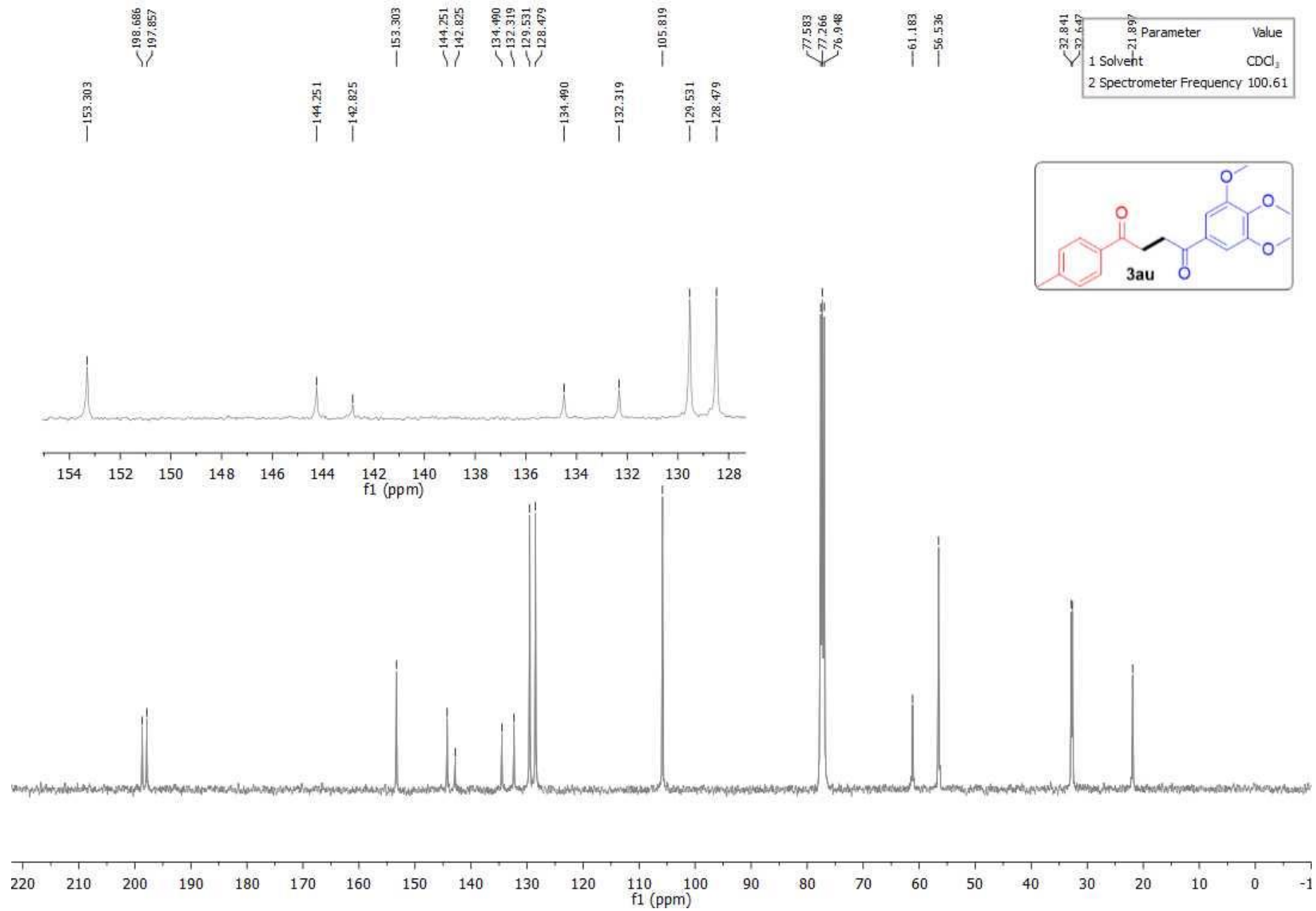


Figure S45. ¹³C NMR spectrum of 1-(*p*-Tolyl)-4-(3,4,5-trimethoxyphenyl)butane-1,4-dione (**3au**).

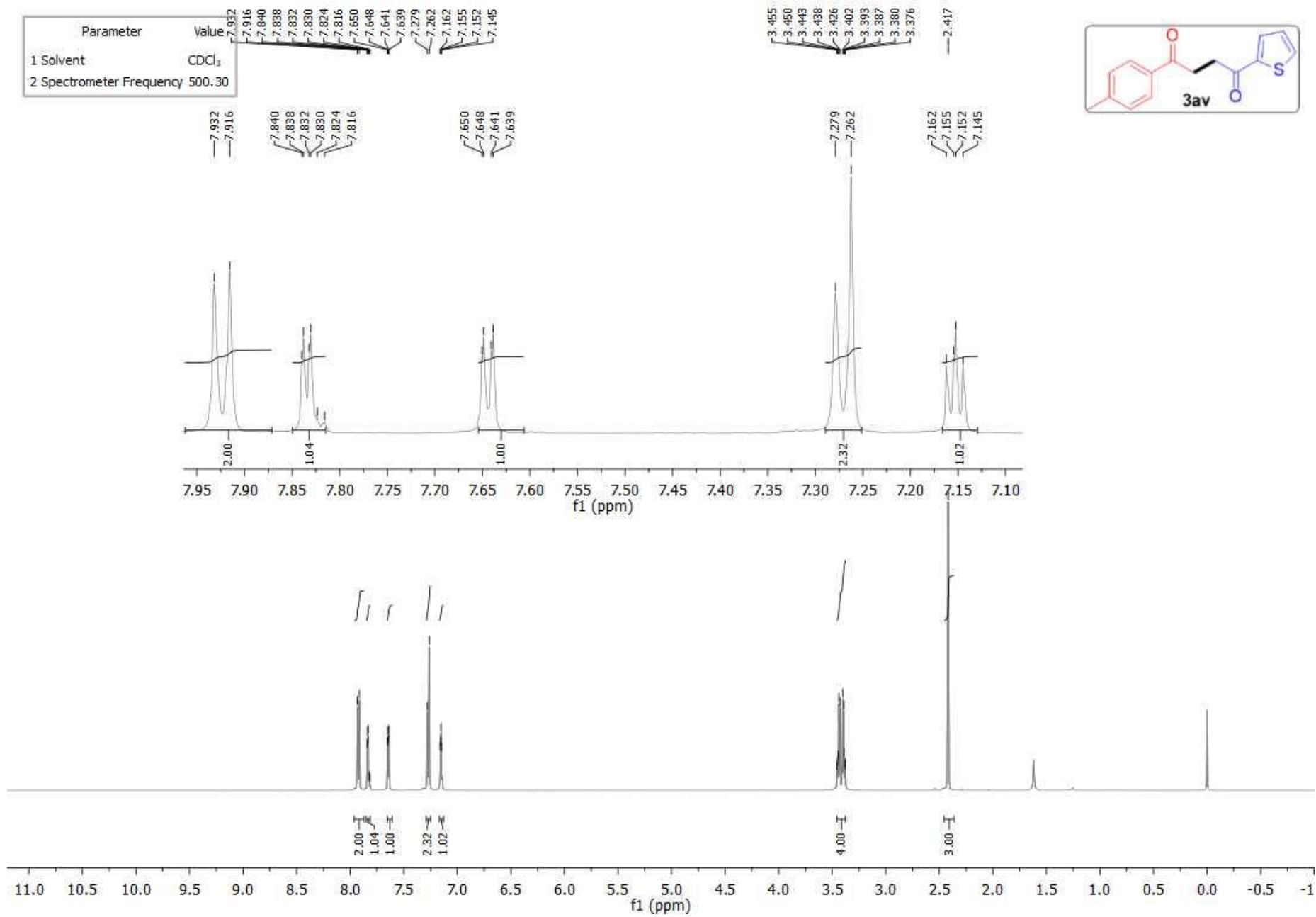


Figure S46. ¹H NMR spectrum of 1-(Thiophen-2-yl)-4-(*p*-tolyl)butane-1,4-dione (**3av**).

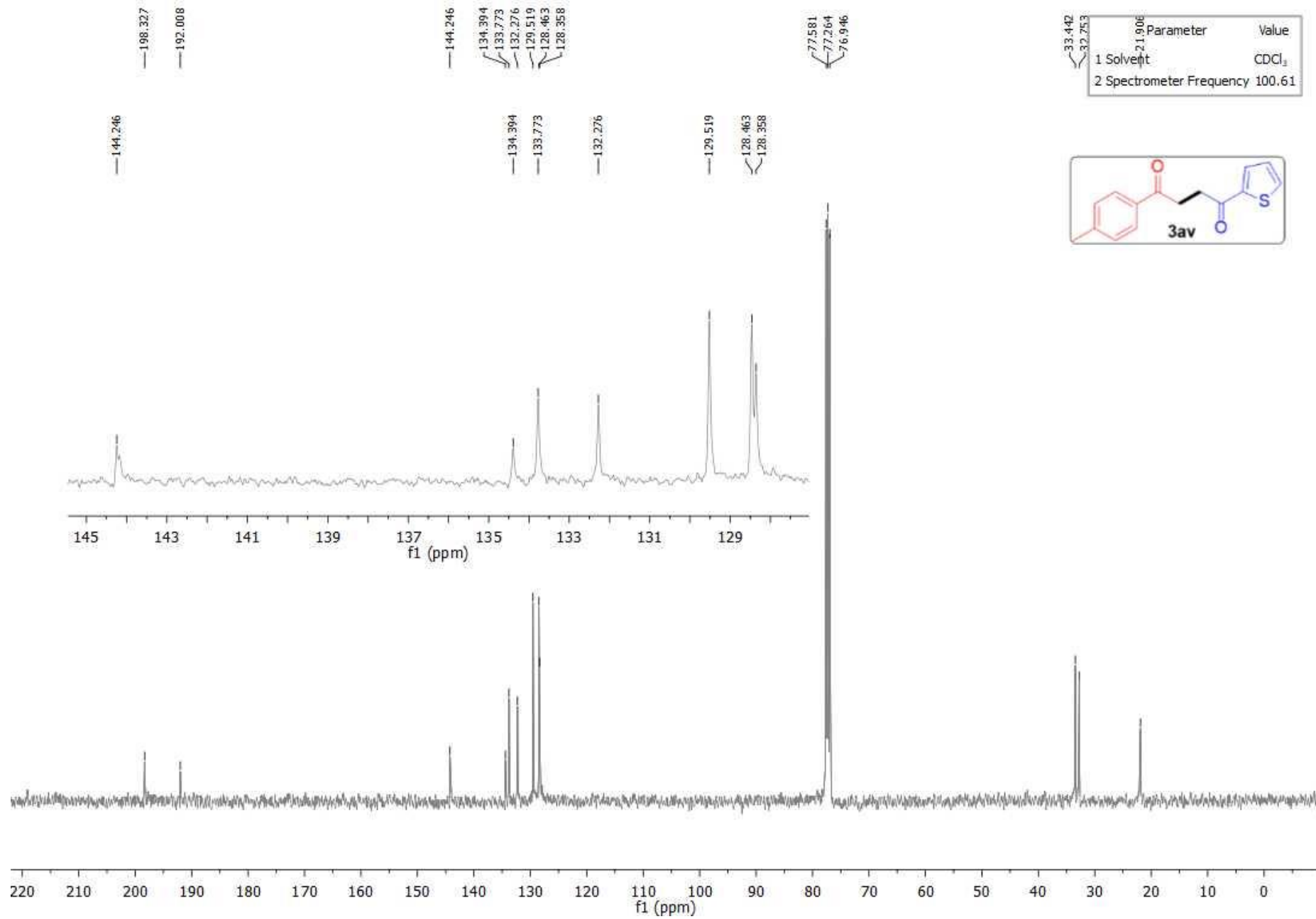


Figure S47. ¹³C NMR spectrum of 1-(Thiophen-2-yl)-4-(*p*-tolyl)butane-1,4-dione (**3av**).

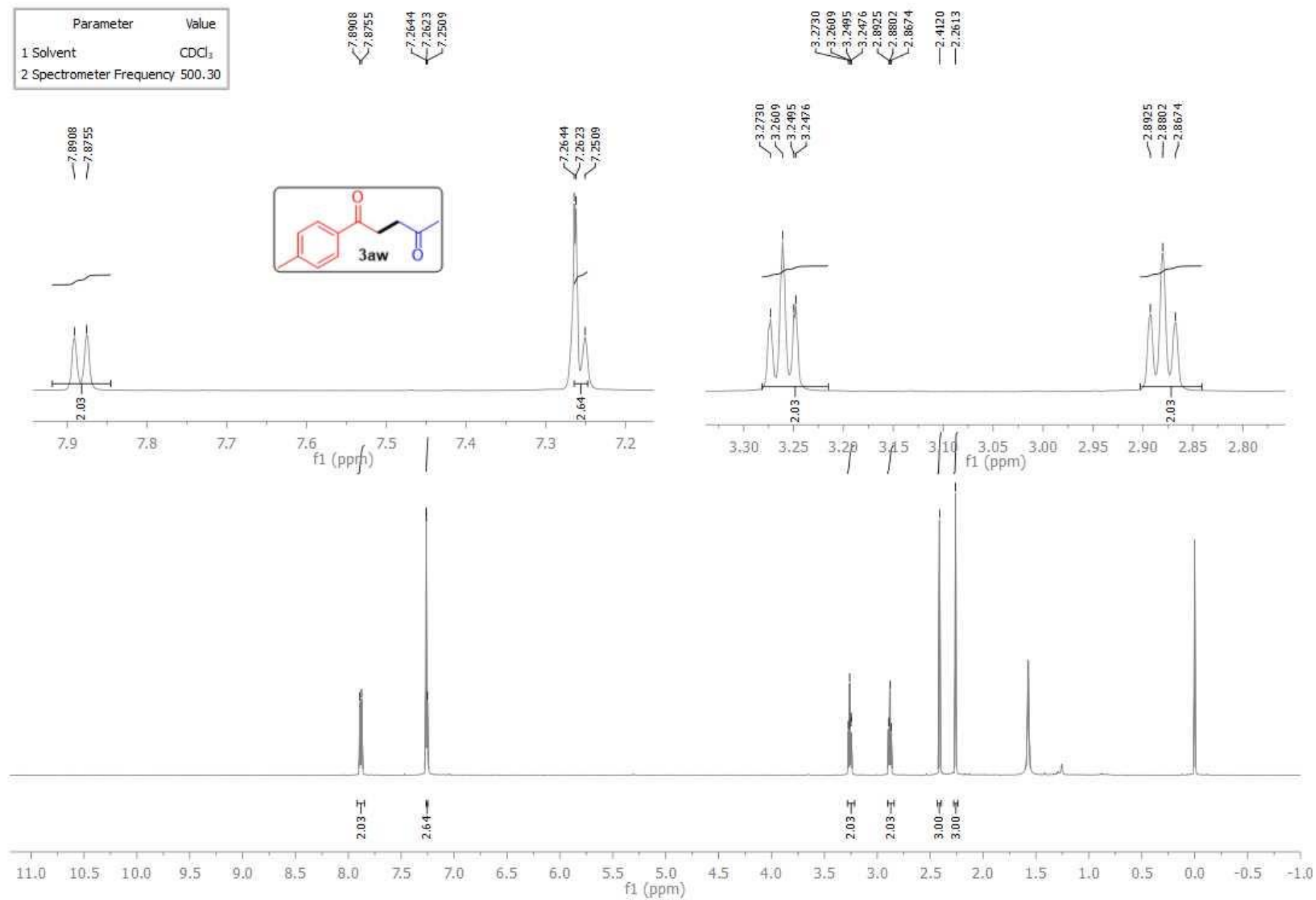


Figure S48. ¹H NMR spectrum of 1-(*p*-Tolyl)pentane-1,4-dione (**3aw**).

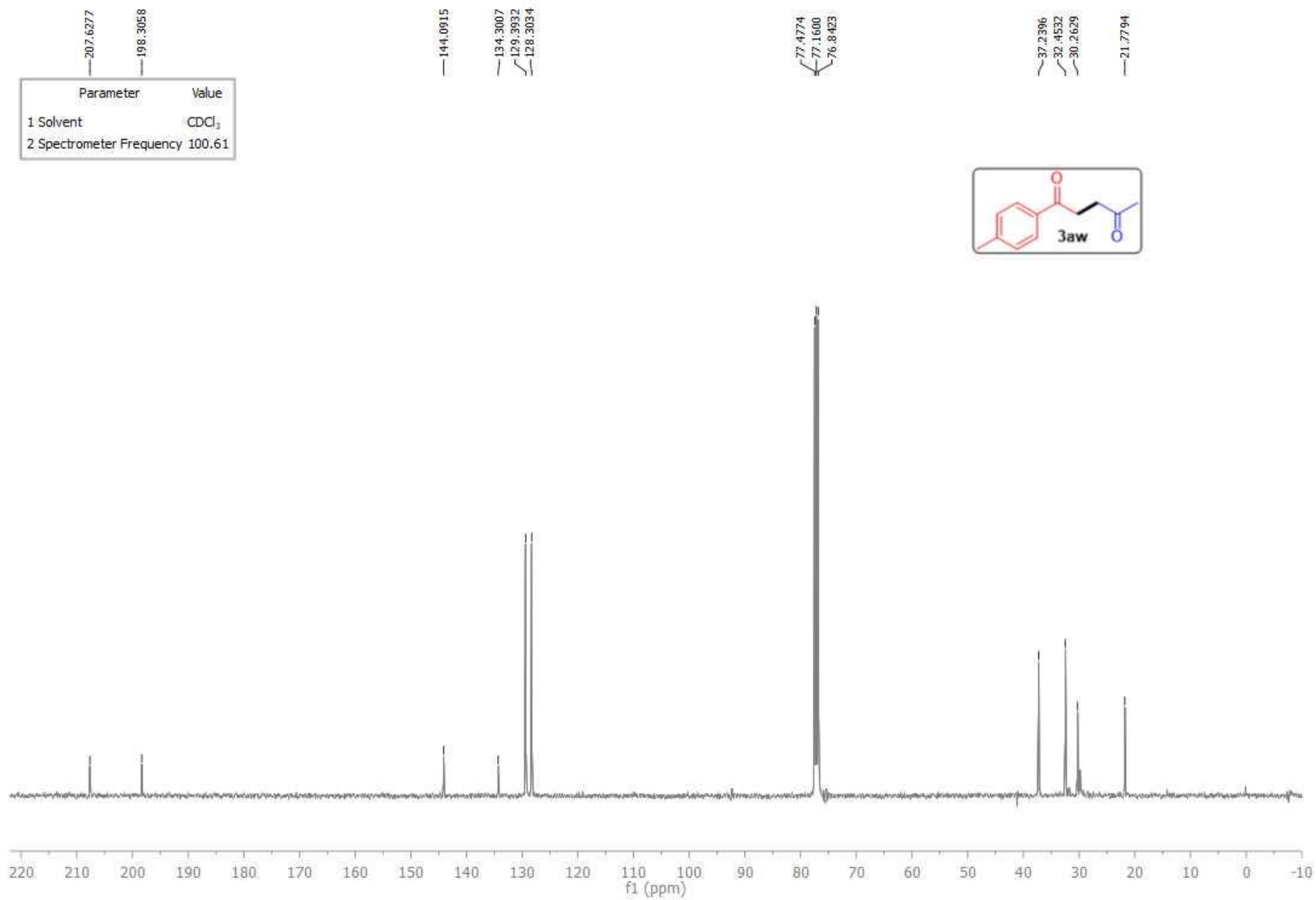


Figure S49. ¹³C NMR spectrum of 1-(*p*-Tolyl)pentane-1,4-dione (**3aw**).

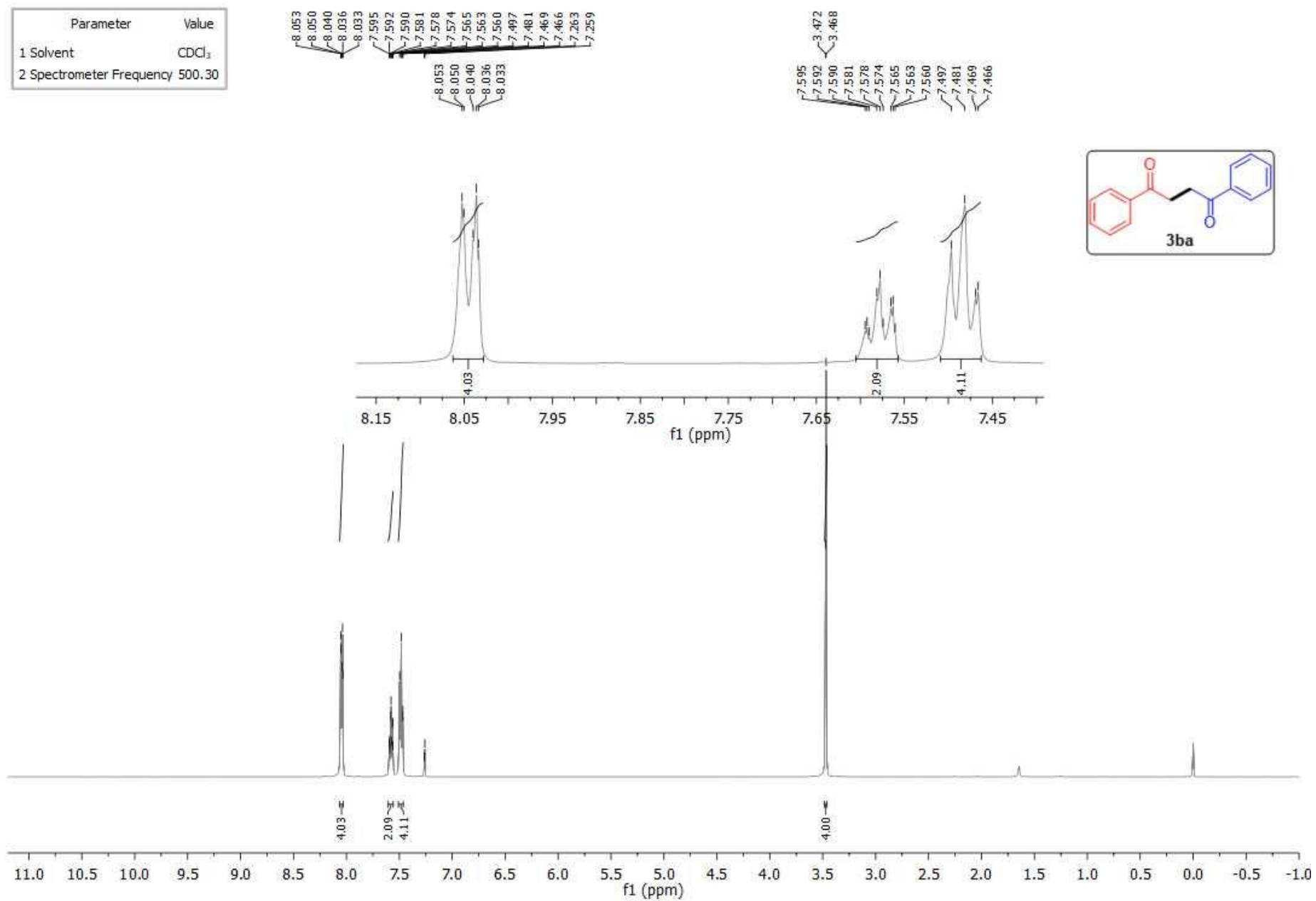


Figure S50. ¹H NMR spectrum of 1,4-Diphenylbutane-1,4-dione (**3ba**).

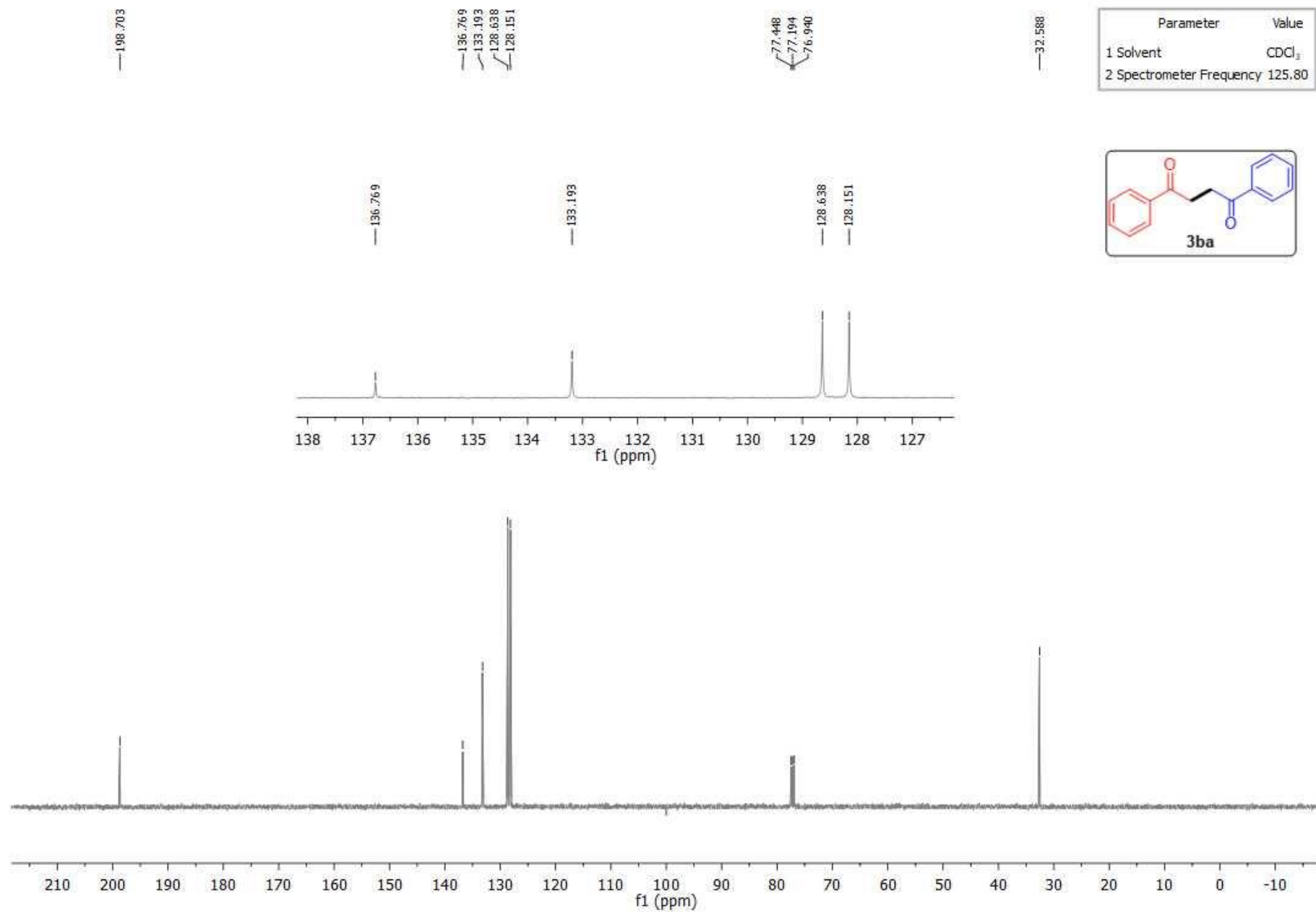


Figure S51. ¹³C NMR spectrum of 1,4-Diphenylbutane-1,4-dione (**3ba**).

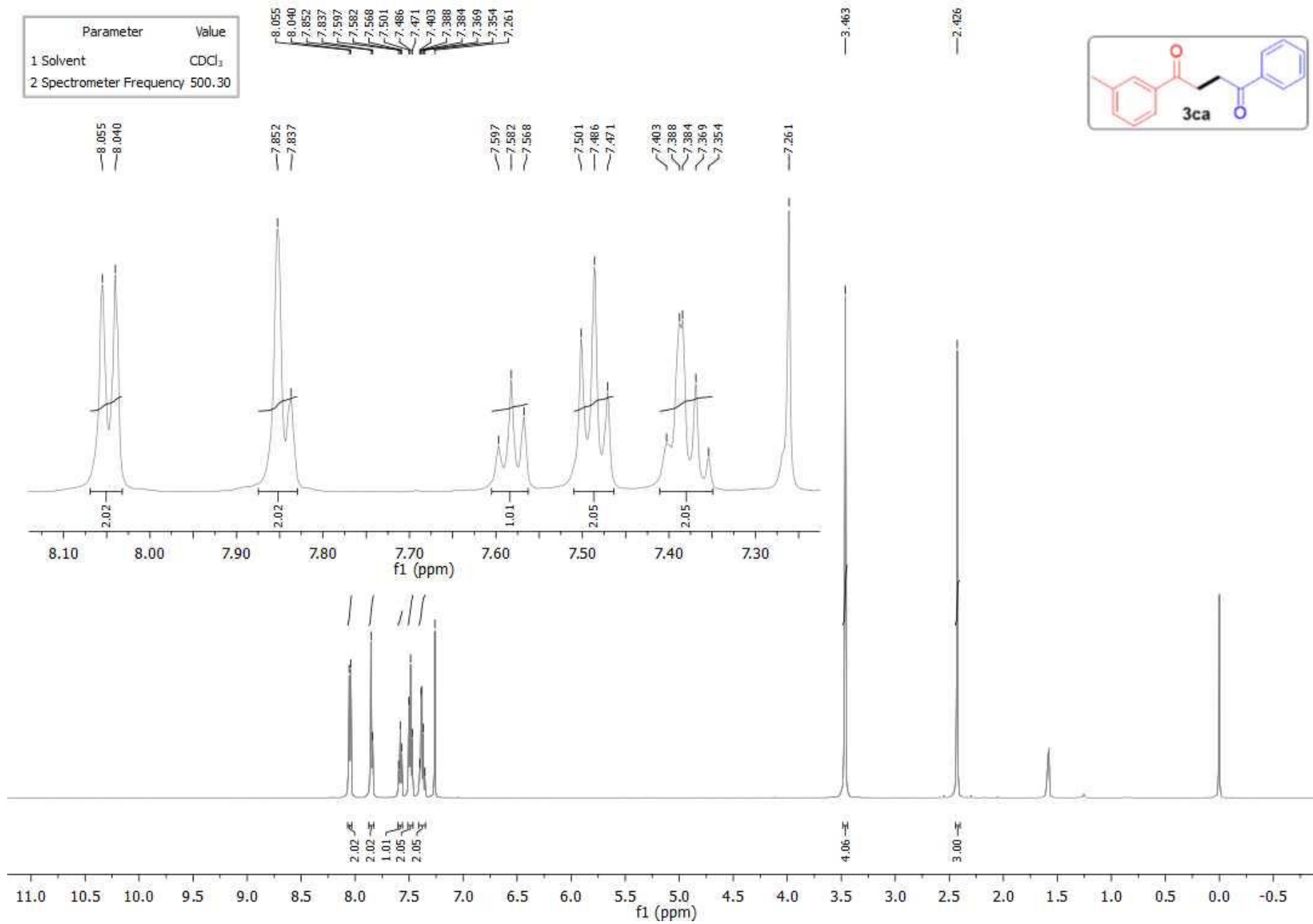


Figure S52. ¹H NMR spectrum of 1-Phenyl-4-(*m*-tolyl)butane-1,4-dione (**3ca**).

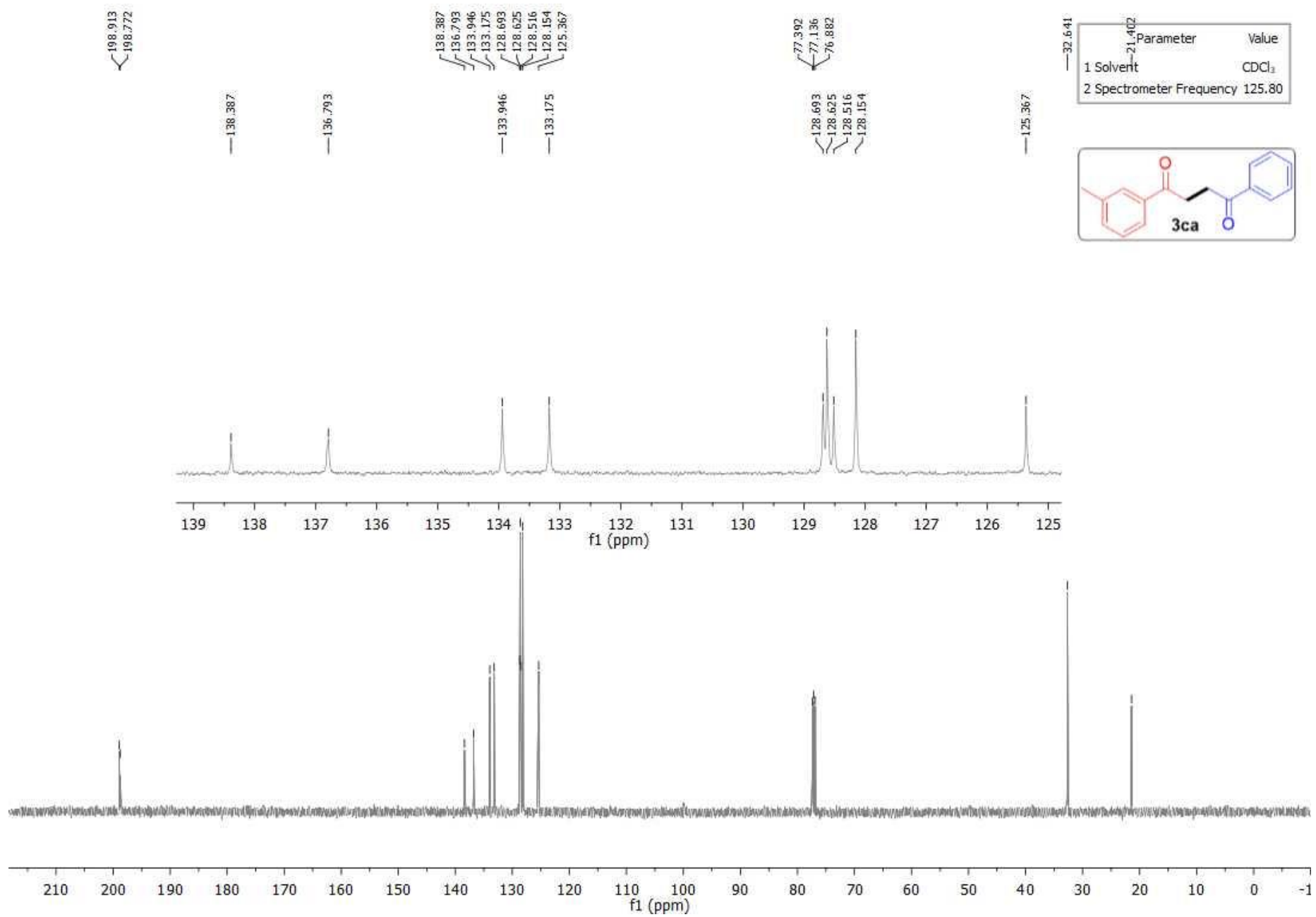


Figure S53. ¹³C NMR spectrum of 1-Phenyl-4-(*m*-tolyl)butane-1,4-dione (**3ca**).

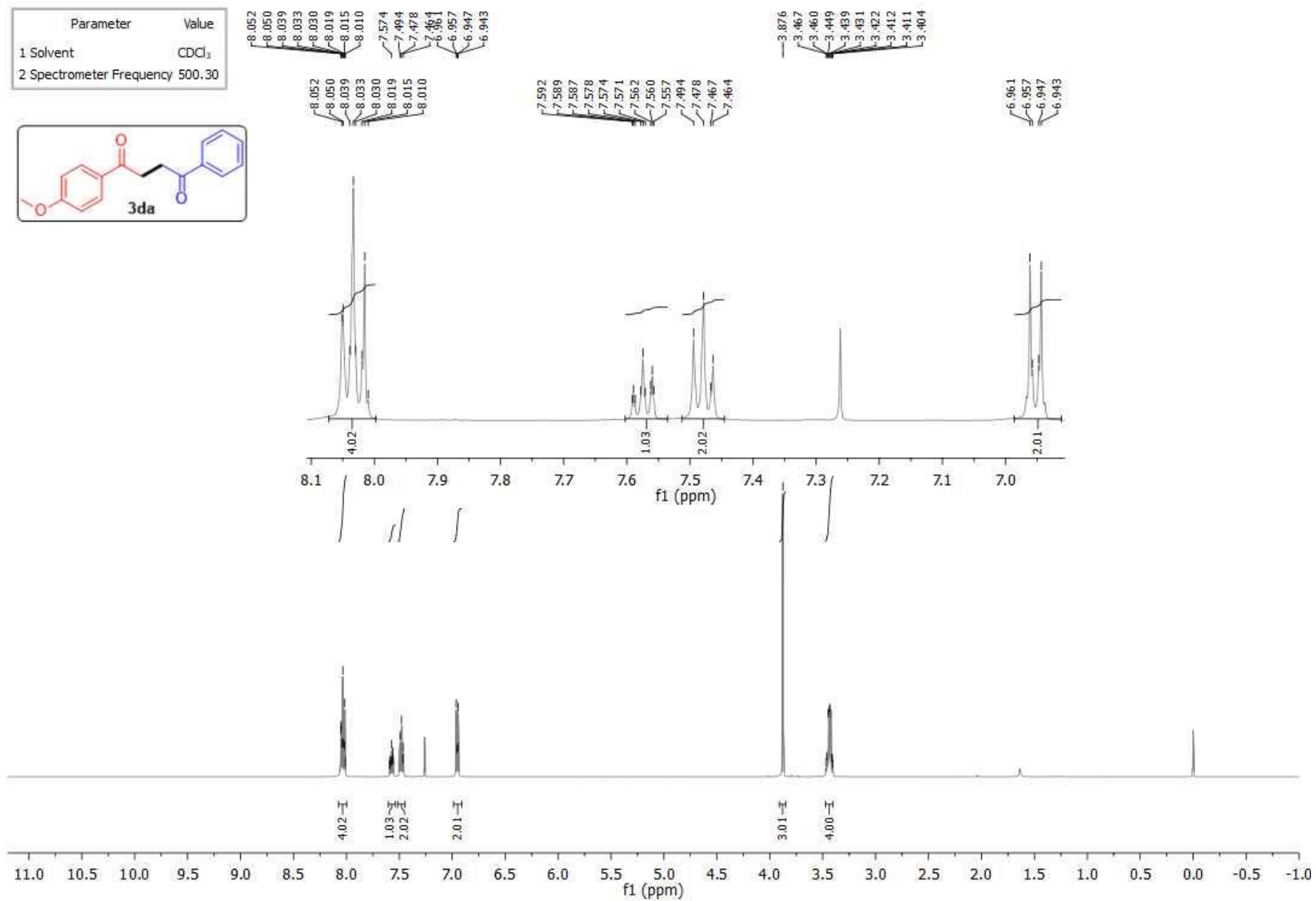


Figure S54. ¹H NMR spectrum of 1-(4-Methoxyphenyl)-4-phenylbutane-1,4-dione (**3da**).

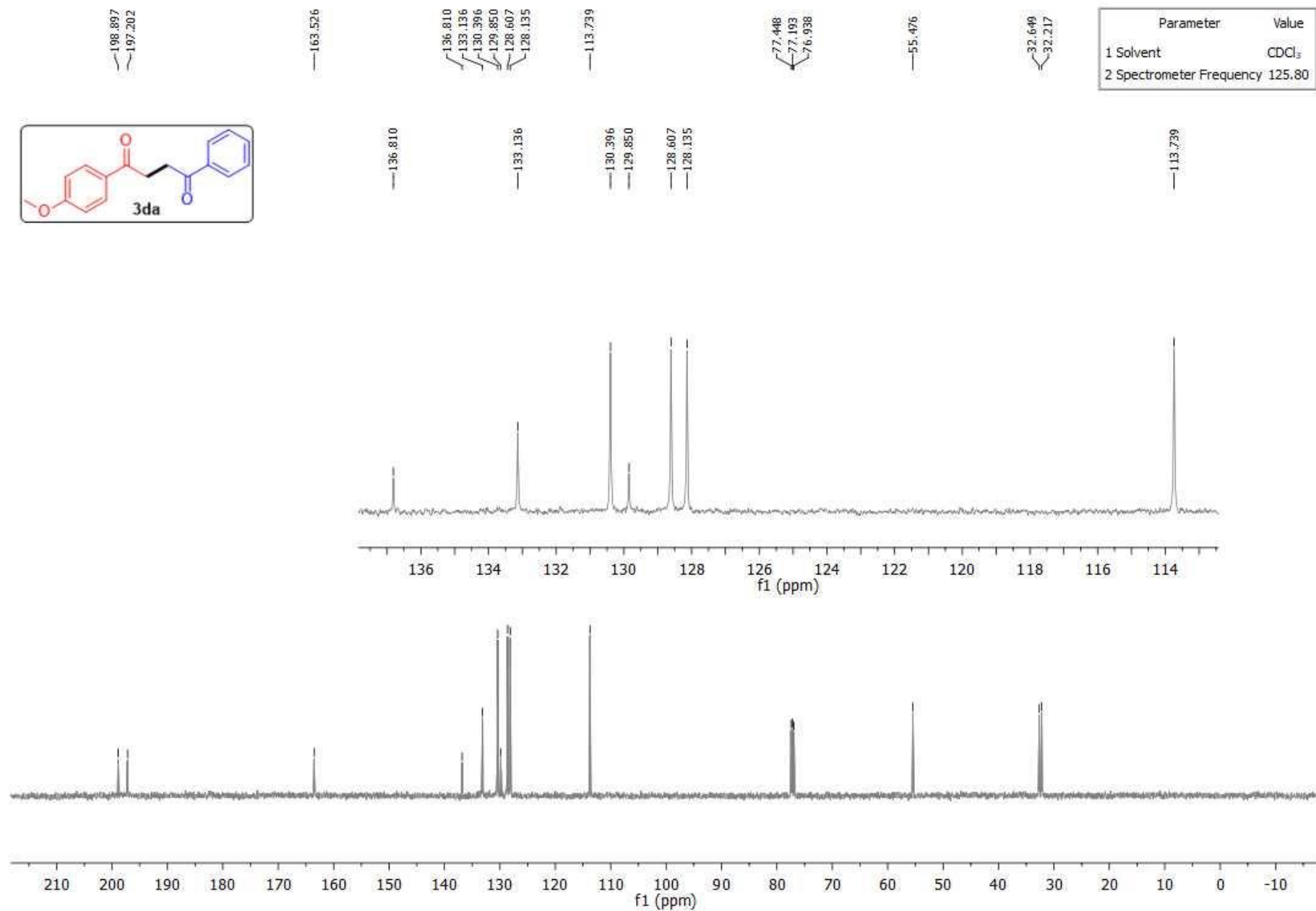


Figure S55. ¹³C NMR spectrum of 1-(4-Methoxyphenyl)-4-phenylbutane-1,4-dione (**3da**).

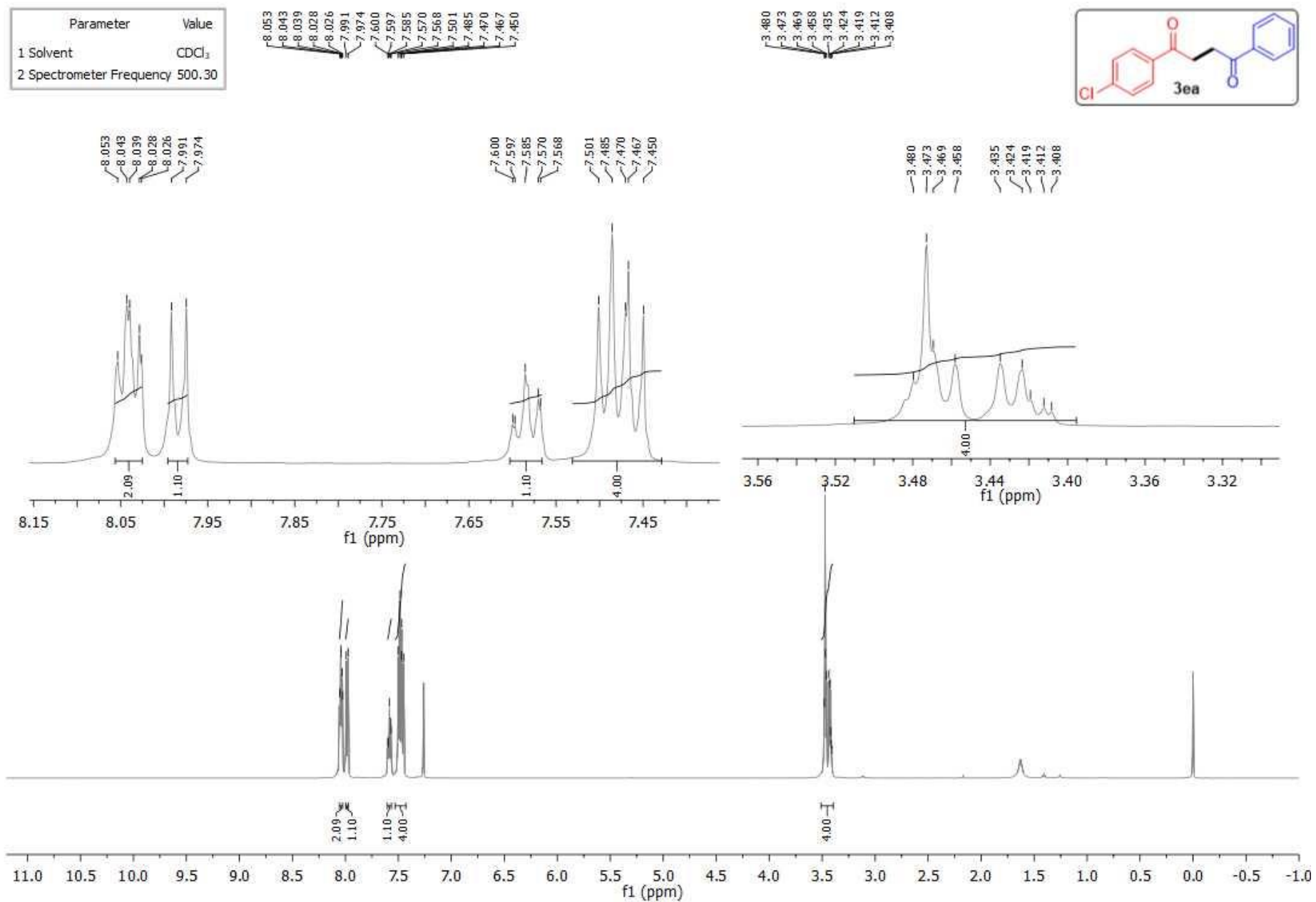


Figure S56. ¹H NMR spectrum of 1-(4-Chlorophenyl)-4-phenylbutane-1,4-dione (**3ea**).

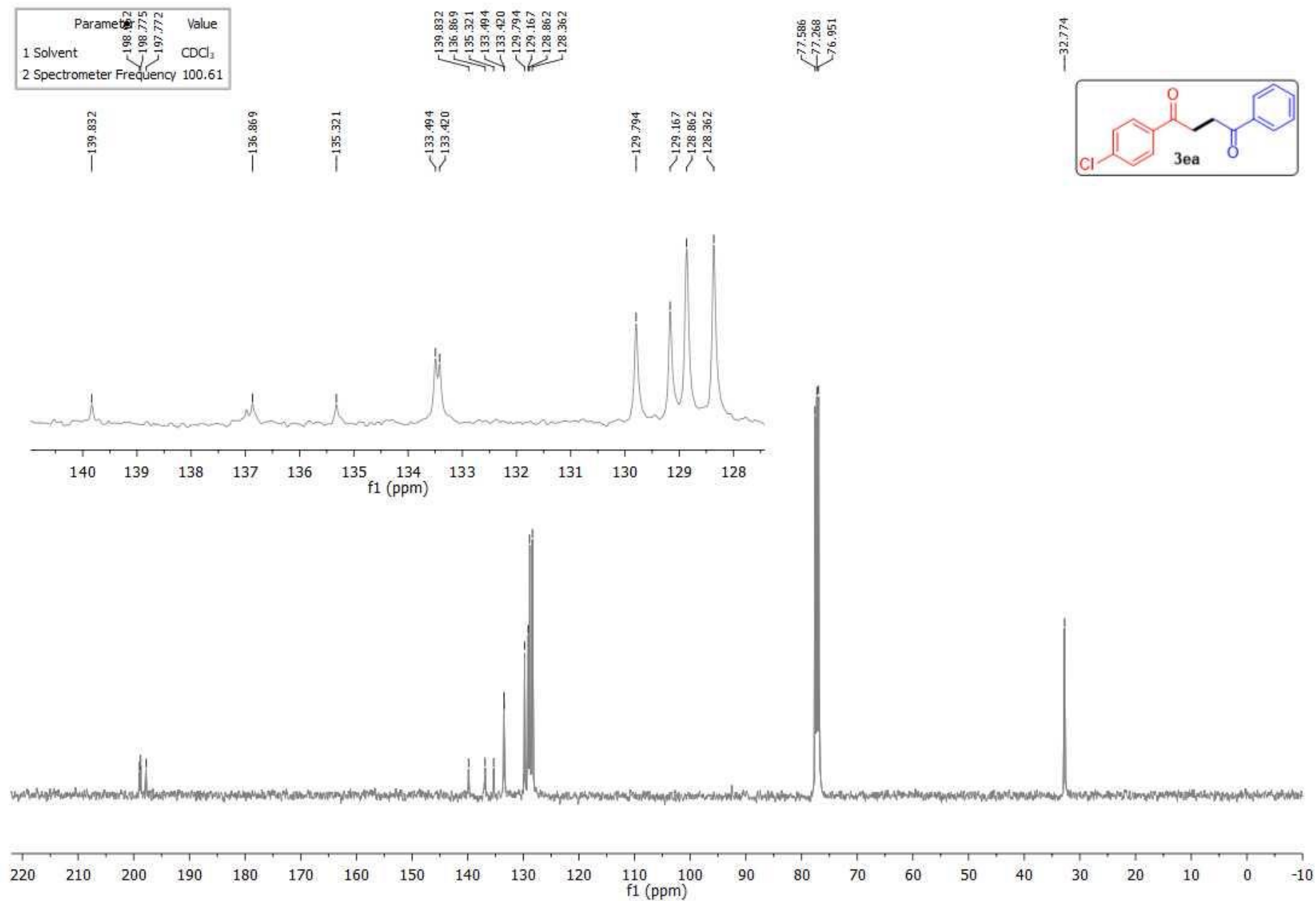


Figure S57. ¹³C NMR spectrum of 1-(4-Chlorophenyl)-4-phenylbutane-1,4-dione (**3ea**).

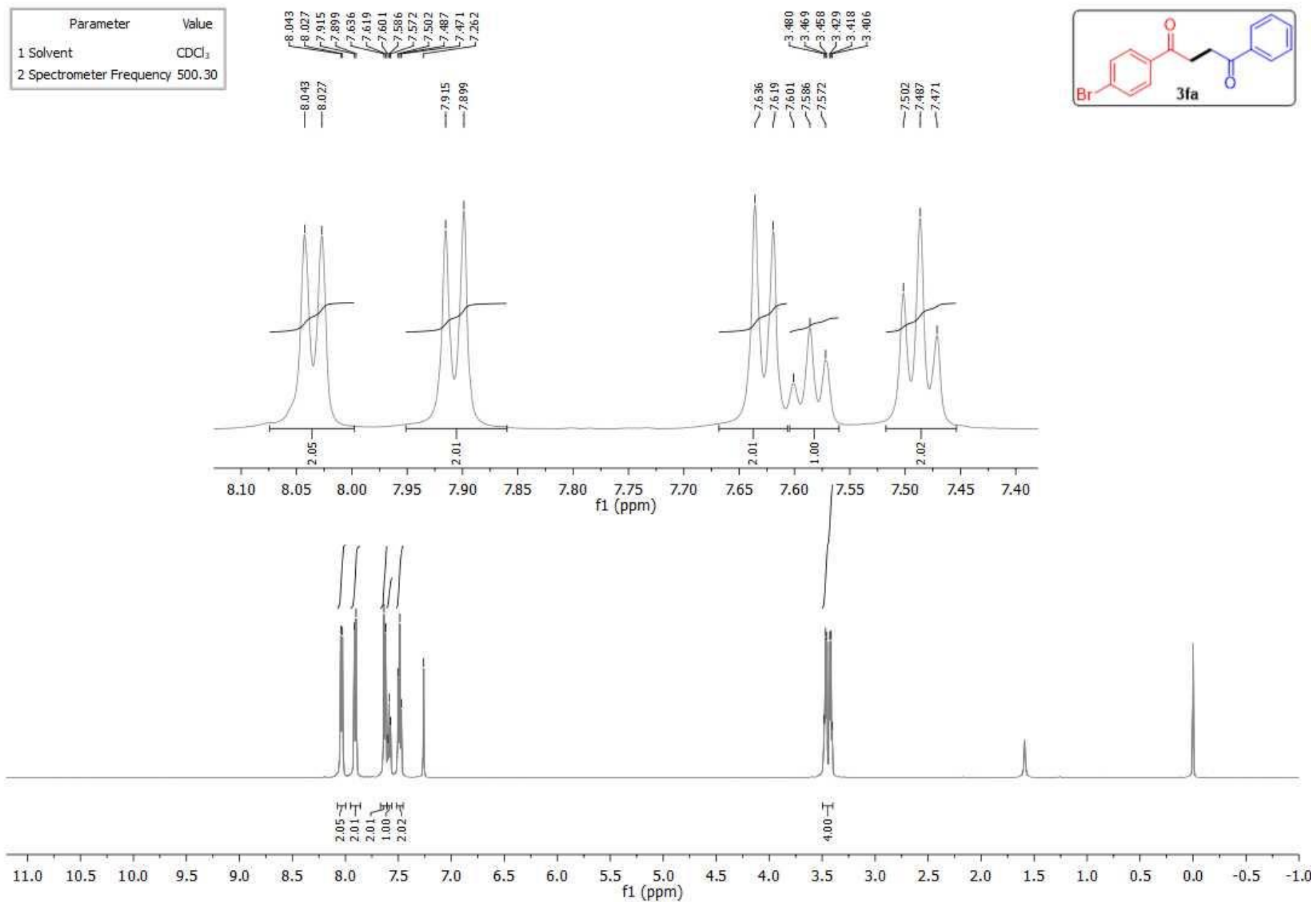


Figure S58. ¹H NMR spectrum of 1-(4-Bromophenyl)-4-phenylbutane-1,4-dione (**3fa**).

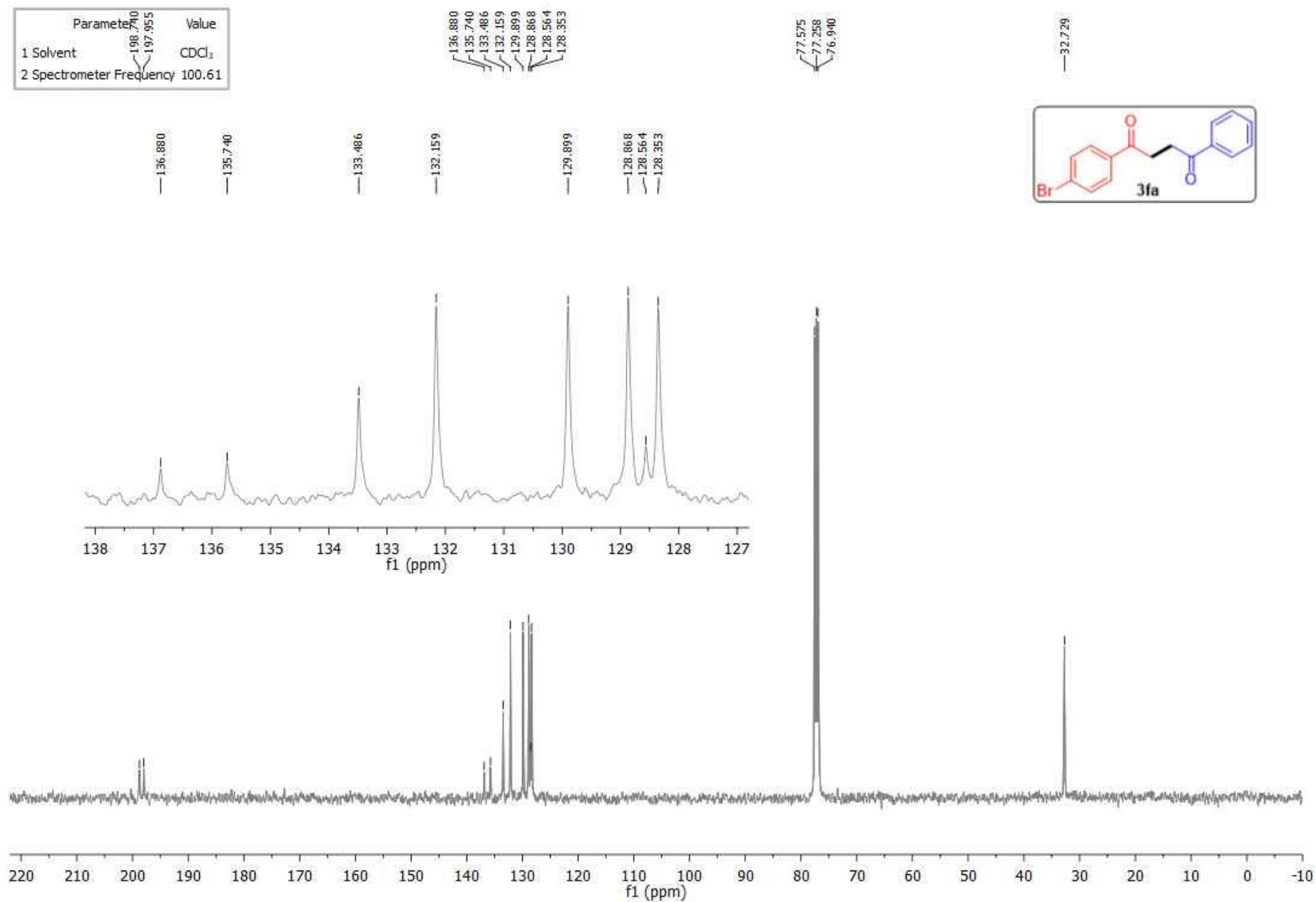


Figure S59. ^{13}C NMR spectrum of 1-(4-Bromophenyl)-4-phenylbutane-1,4-dione (**3fa**).

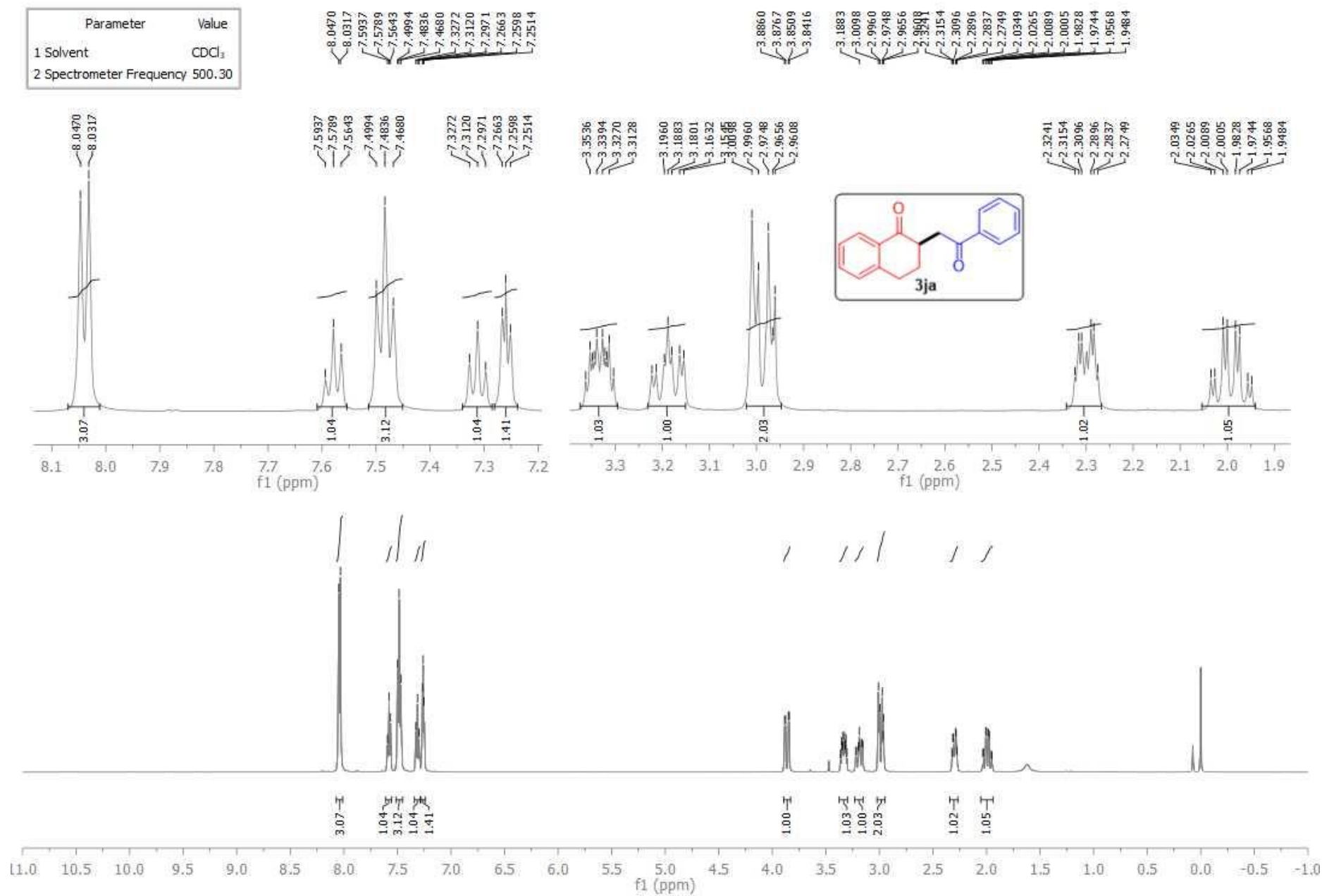


Figure S60. ¹H NMR spectrum of 2-(2-Oxo-2-phenylethyl)-3,4-dihydronaphthalen-1(2*H*)-one (**3ja**).

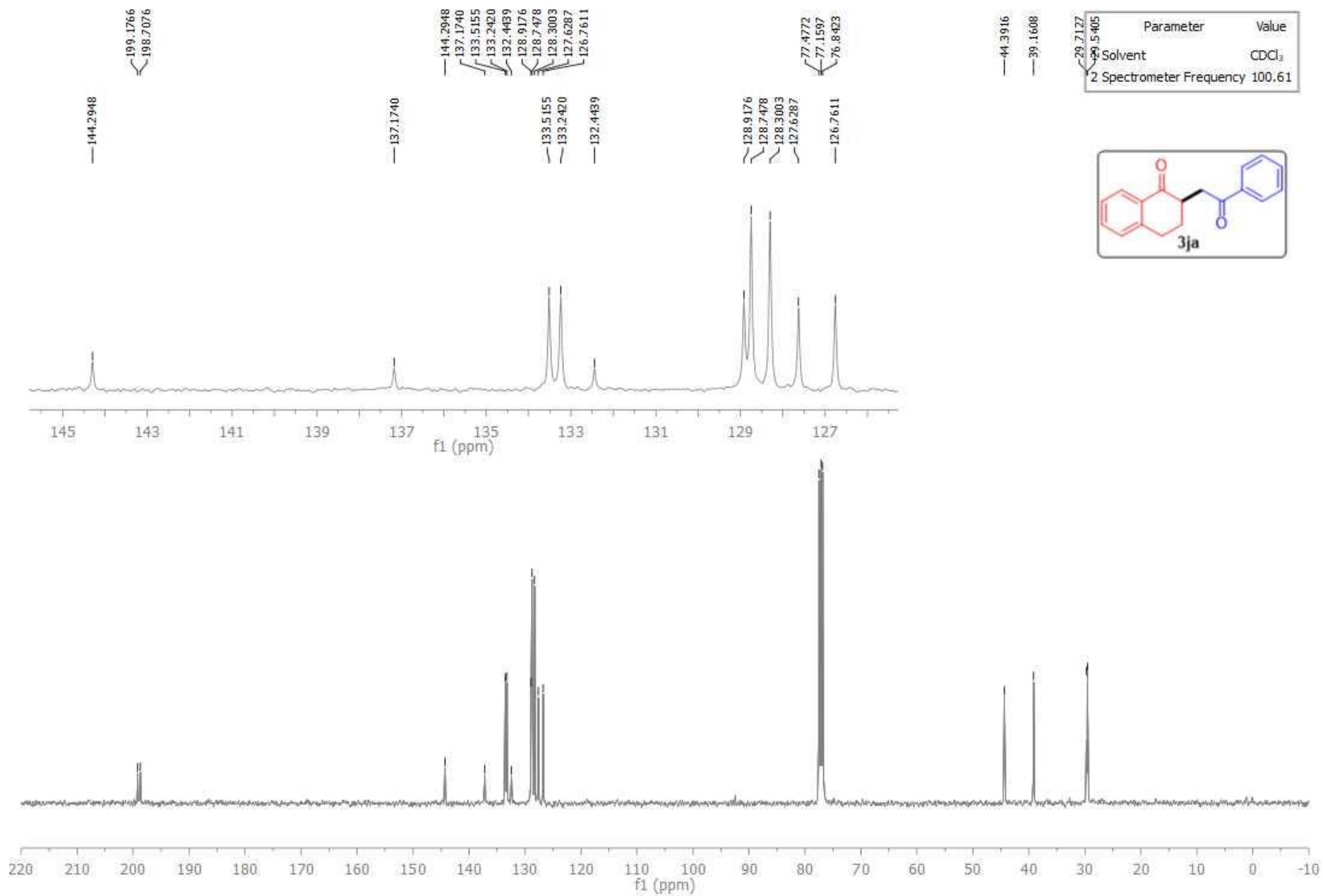


Figure S61. ¹³C NMR spectrum of 2-(2-Oxo-2-phenylethyl)-3,4-dihydronaphthalen-1(2H)-one (**3ja**).

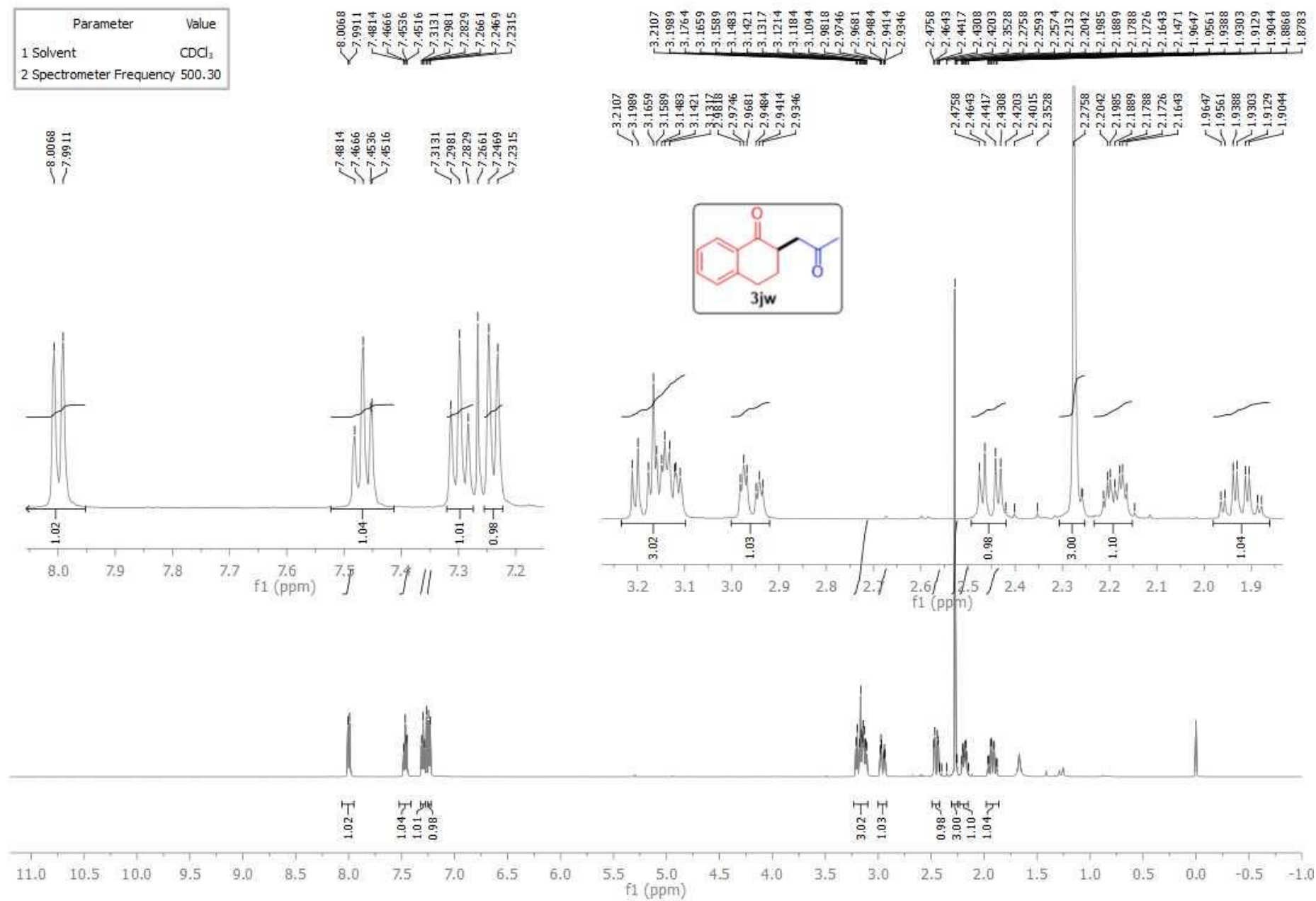


Figure S62. ¹H NMR spectrum of 2-(2-Oxopropyl)-3,4-dihydro-1H-naphthalen-1-one (**3jw**).

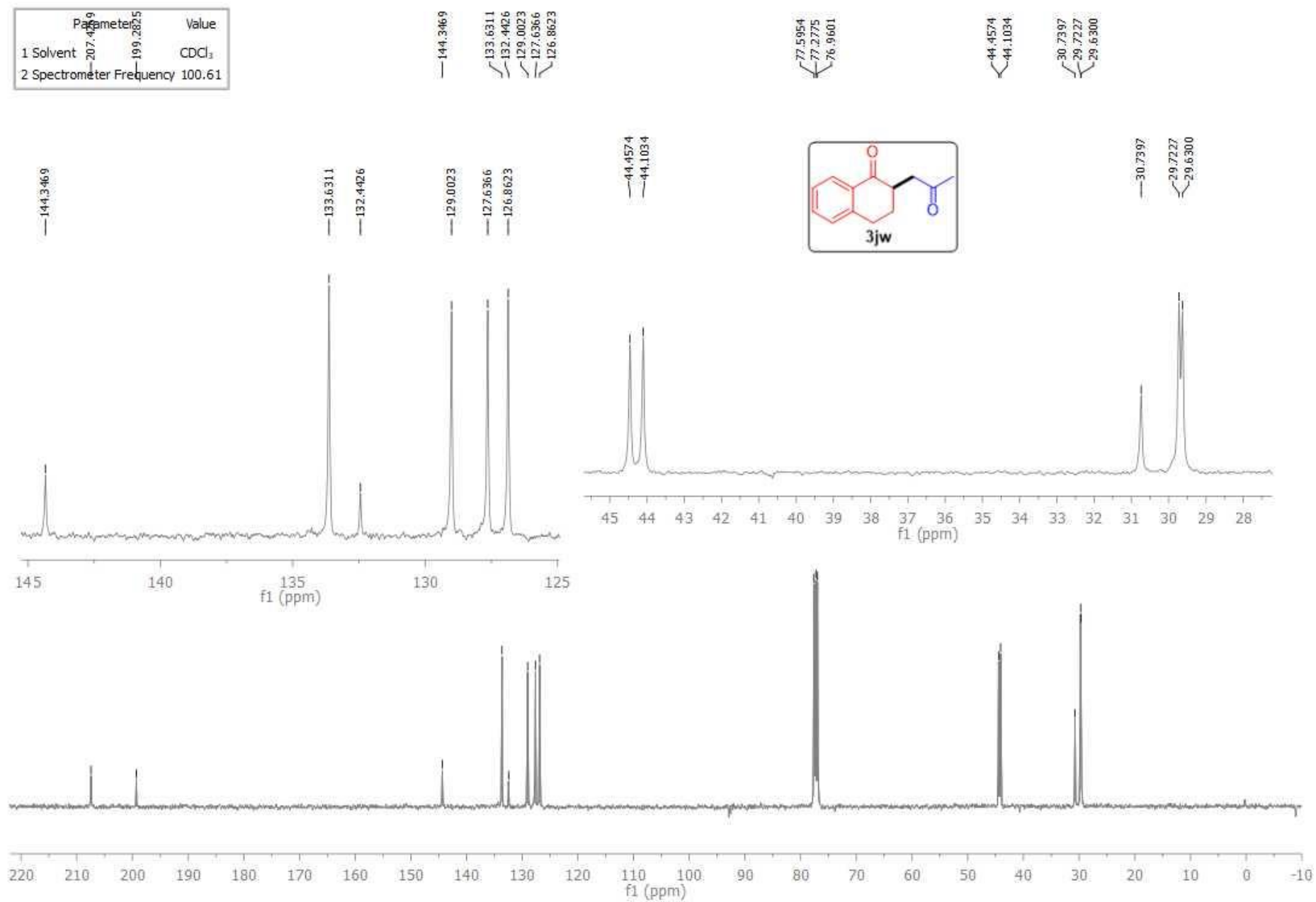


Figure S63. ¹³C NMR spectrum of 2-(2-Oxopropyl)-3,4-dihydronaphthalen-1(2H)-one (**3jw**).

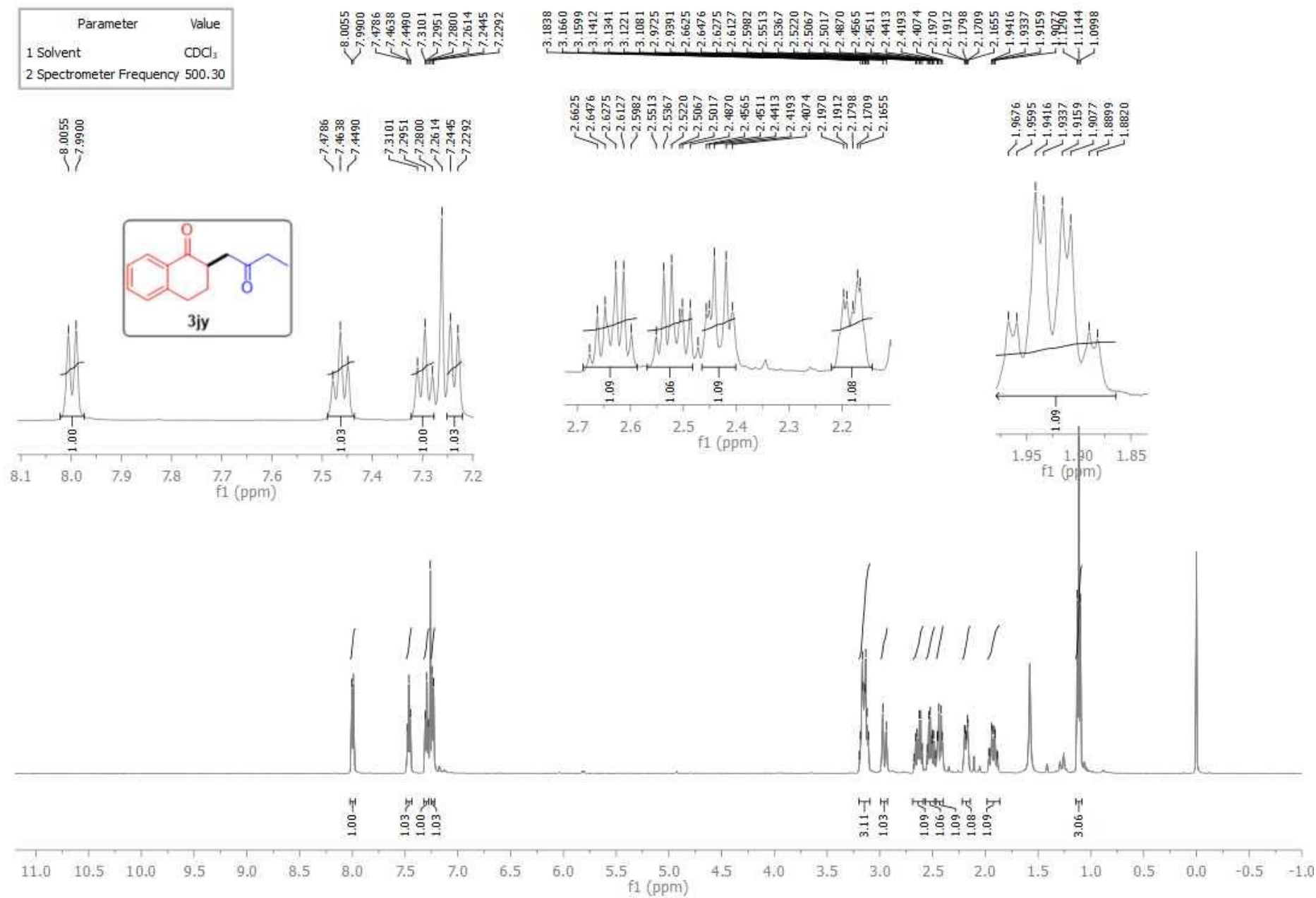


Figure S64. ¹H NMR spectrum of 2-(2-Oxobutyl)-3,4-dihydronaphthalen-1(2H)-one (**3jy**).

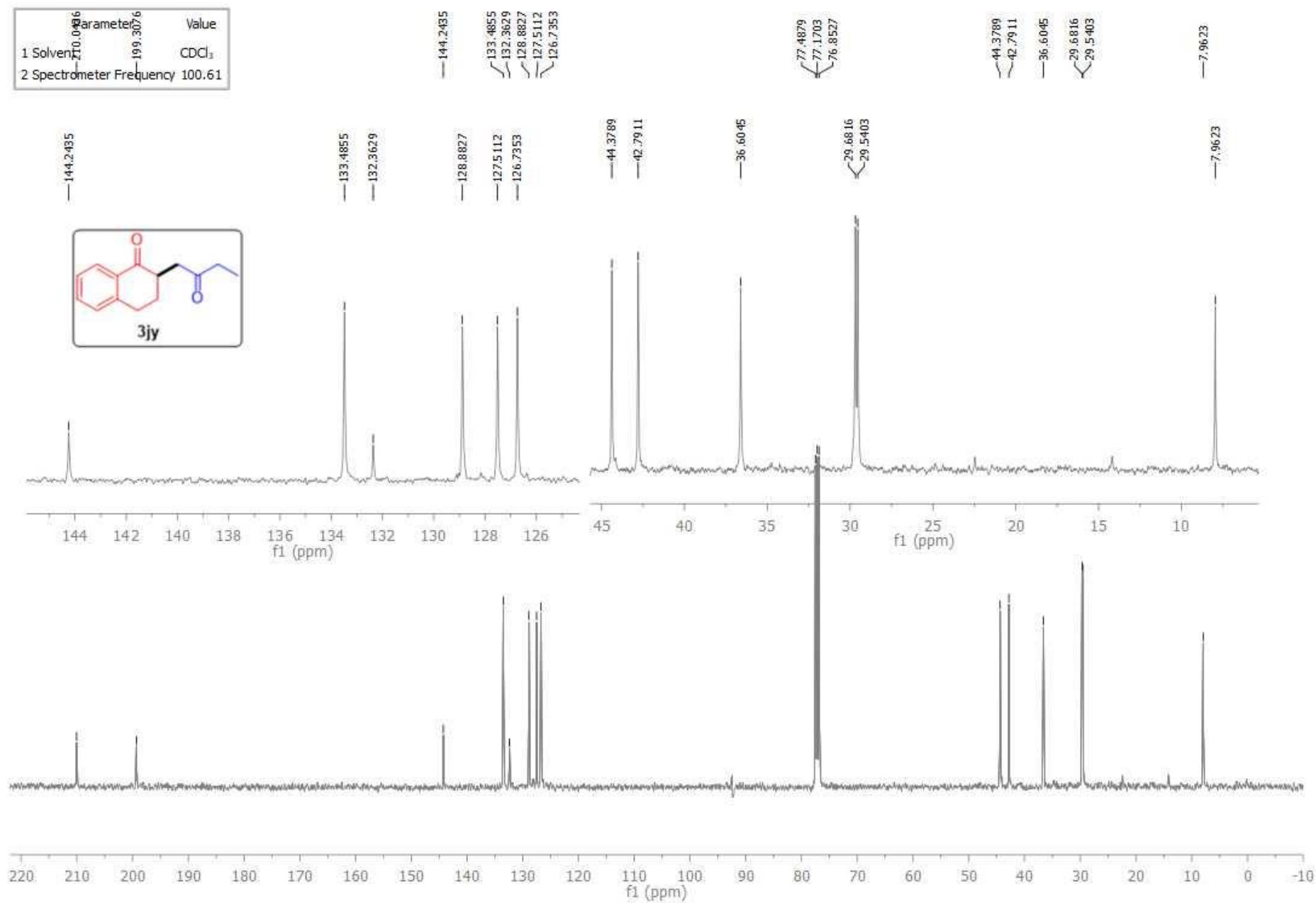


Figure S65. ¹³C NMR spectrum of 2-(2-Oxobutyl)-3,4-dihydronaphthalen-1(2*H*)-one (**3jy**).

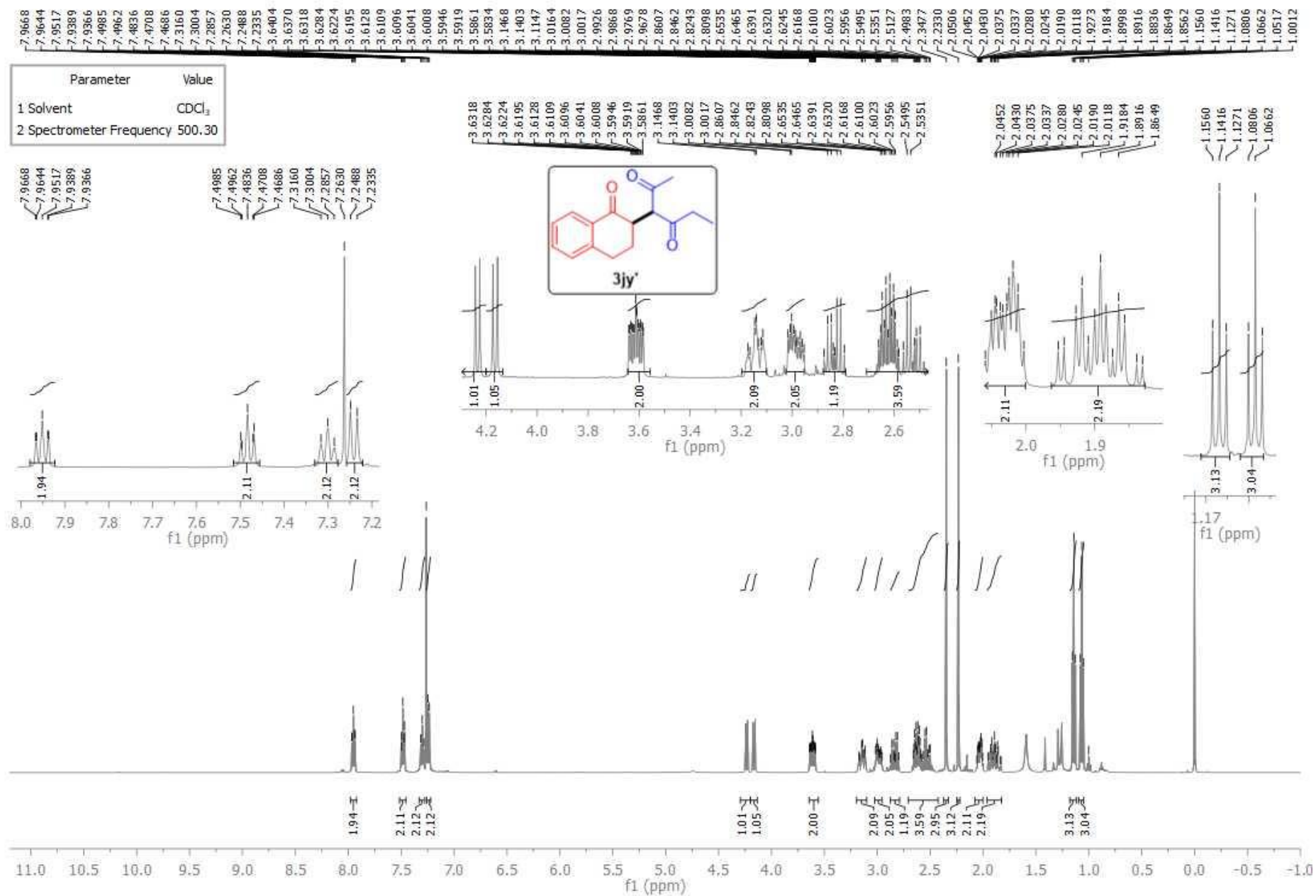


Figure S66. ¹H NMR spectrum of 3-(-1-Oxo-1,2,3,4-tetrahydronaphthalen-2-yl)hexane-2,4-dione (**3jy'**).

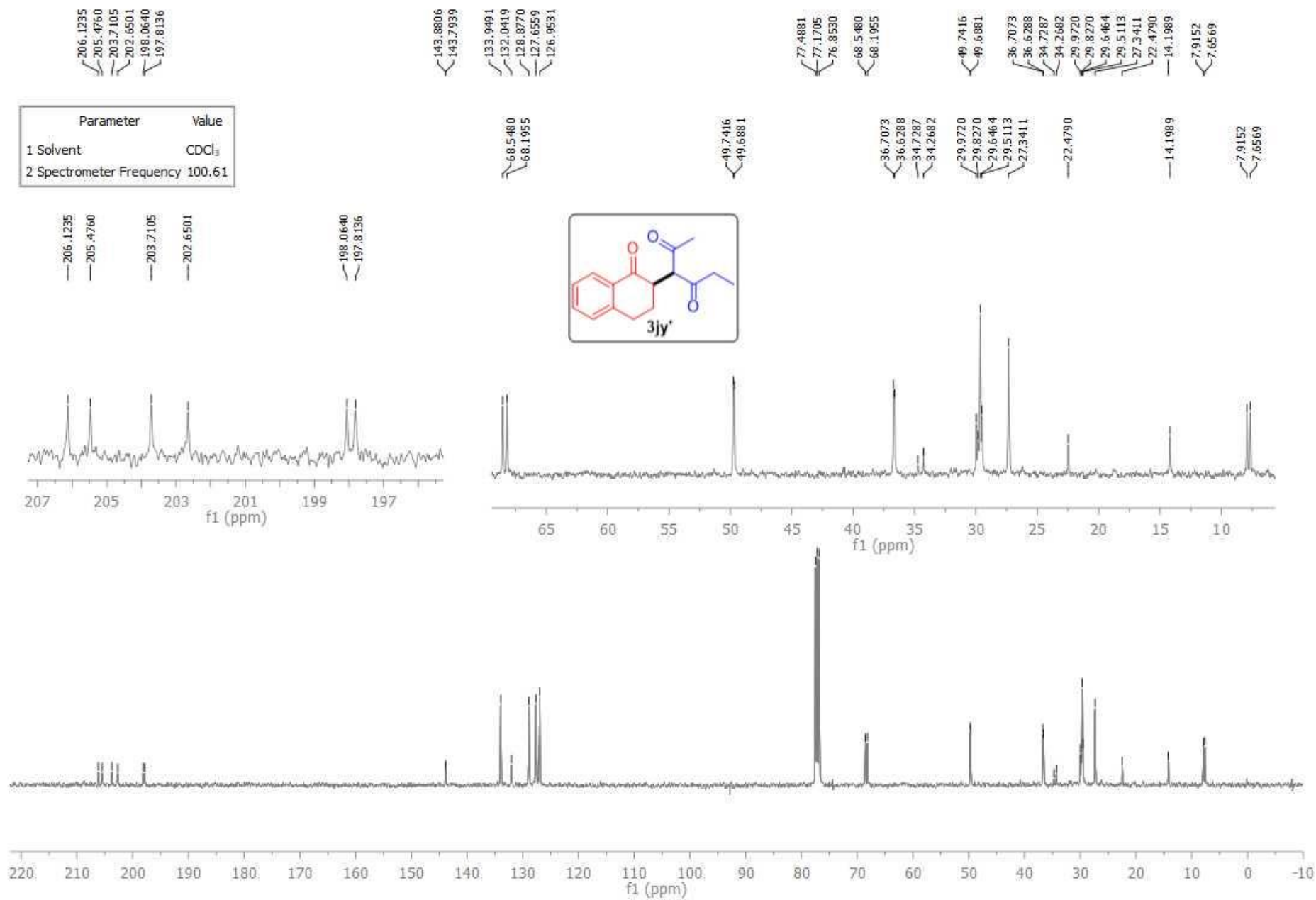


Figure S67. ¹³C NMR spectrum of 3-(-1-Oxo-1,2,3,4-tetrahydronaphthalen-2-yl)hexane-2,4-dione (**3jy'**).

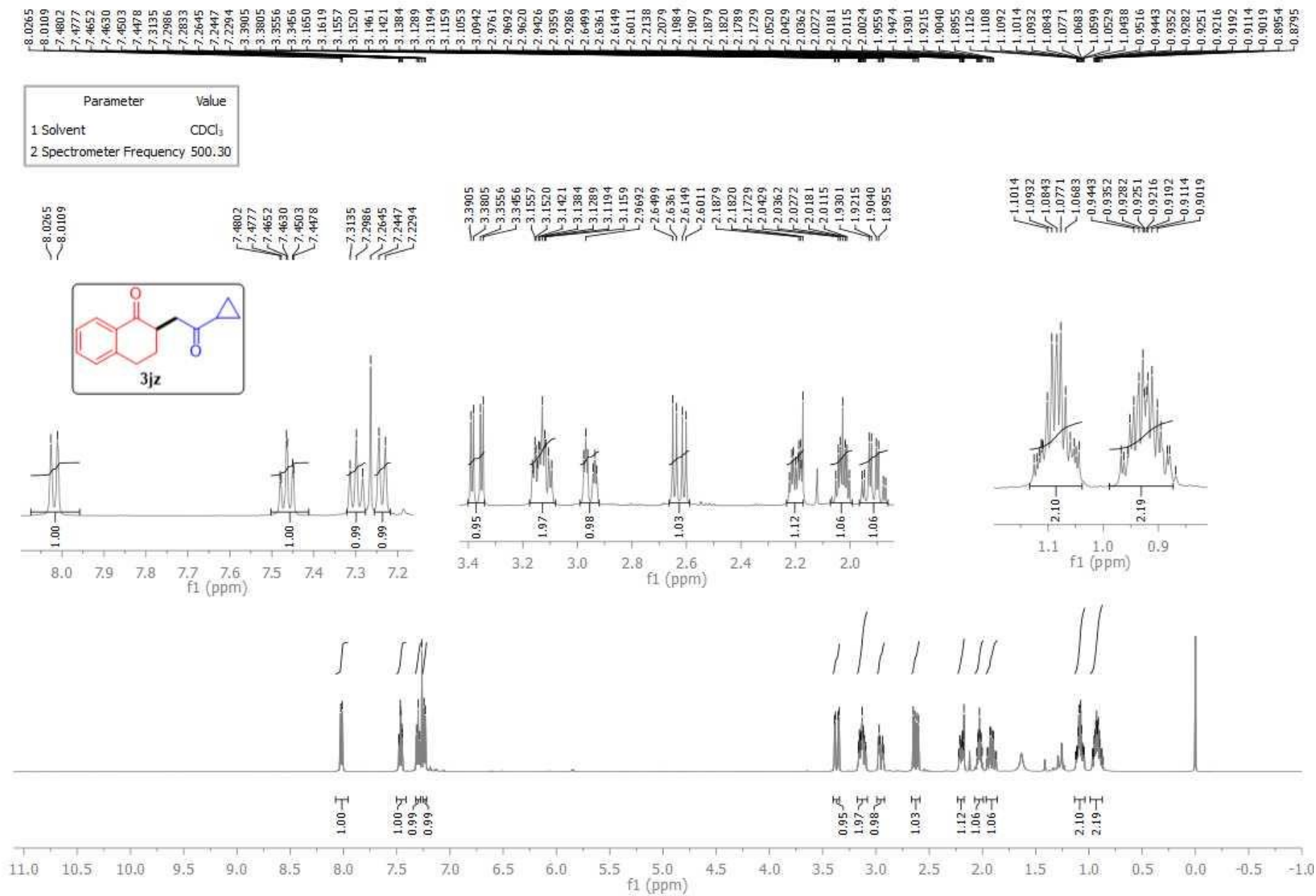


Figure S68. ¹H NMR spectrum of 2-(2-cyclopropyl-2-oxoethyl)-3,4-dihydronaphthalen-1(2H)-one (**3jz**).

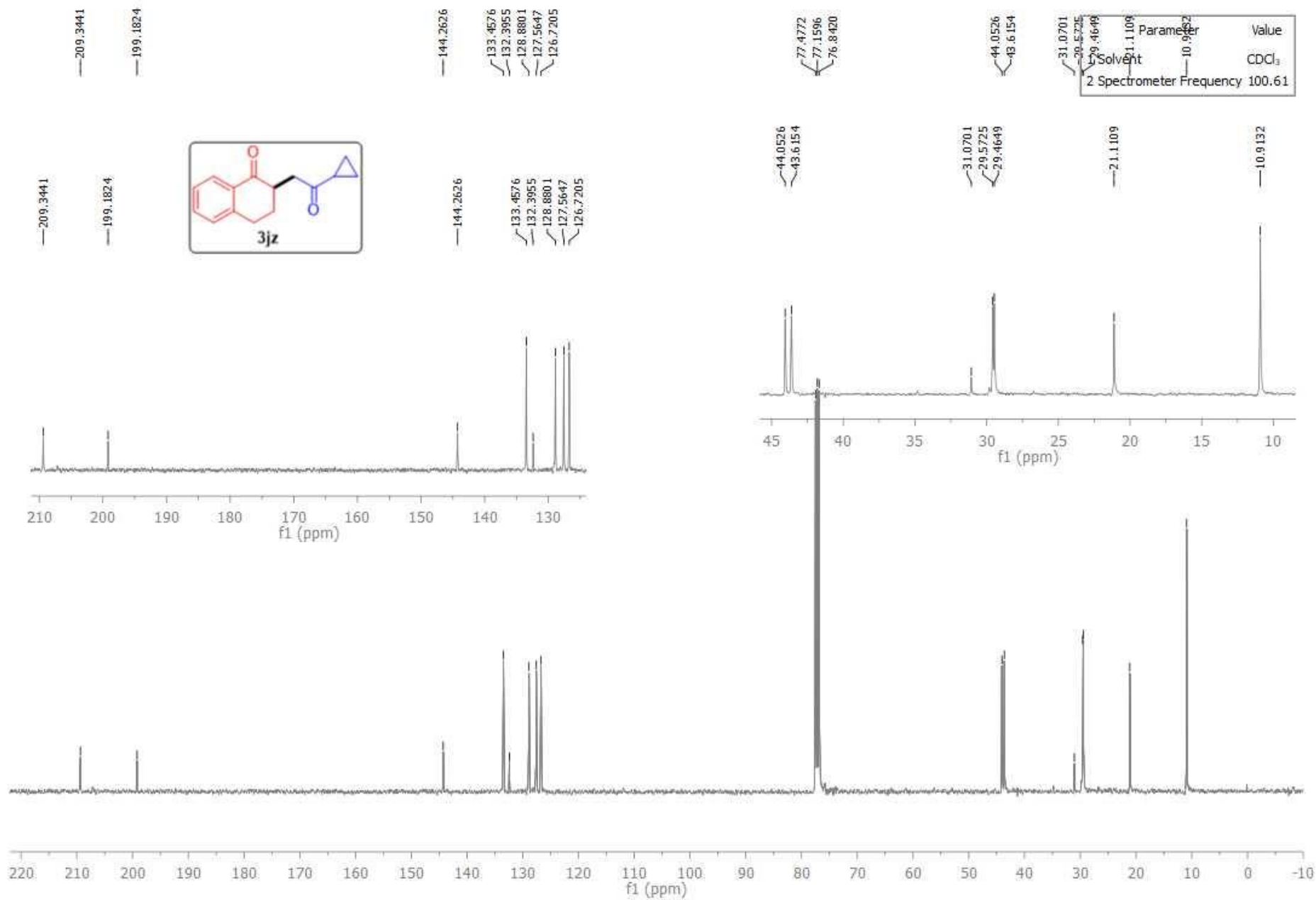


Figure S69. ¹³C NMR spectrum of 2-(2-cyclopropyl-2-oxoethyl)-3,4-dihydronaphthalen-1(2H)-one (**3jz**).

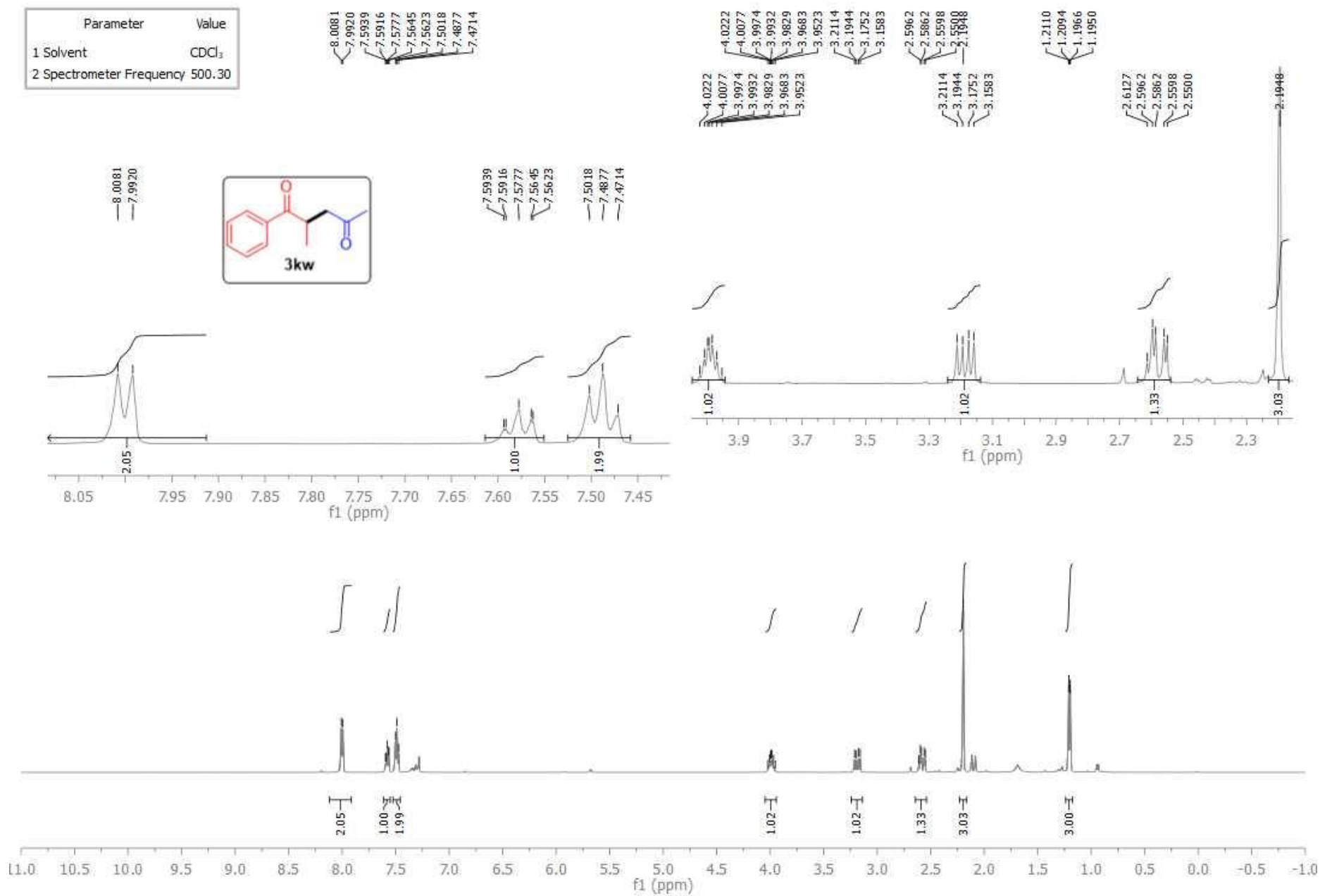


Figure S70. ¹H NMR spectrum of 2-Methyl-1-phenylpentane-1,4-dione (**3kw**).

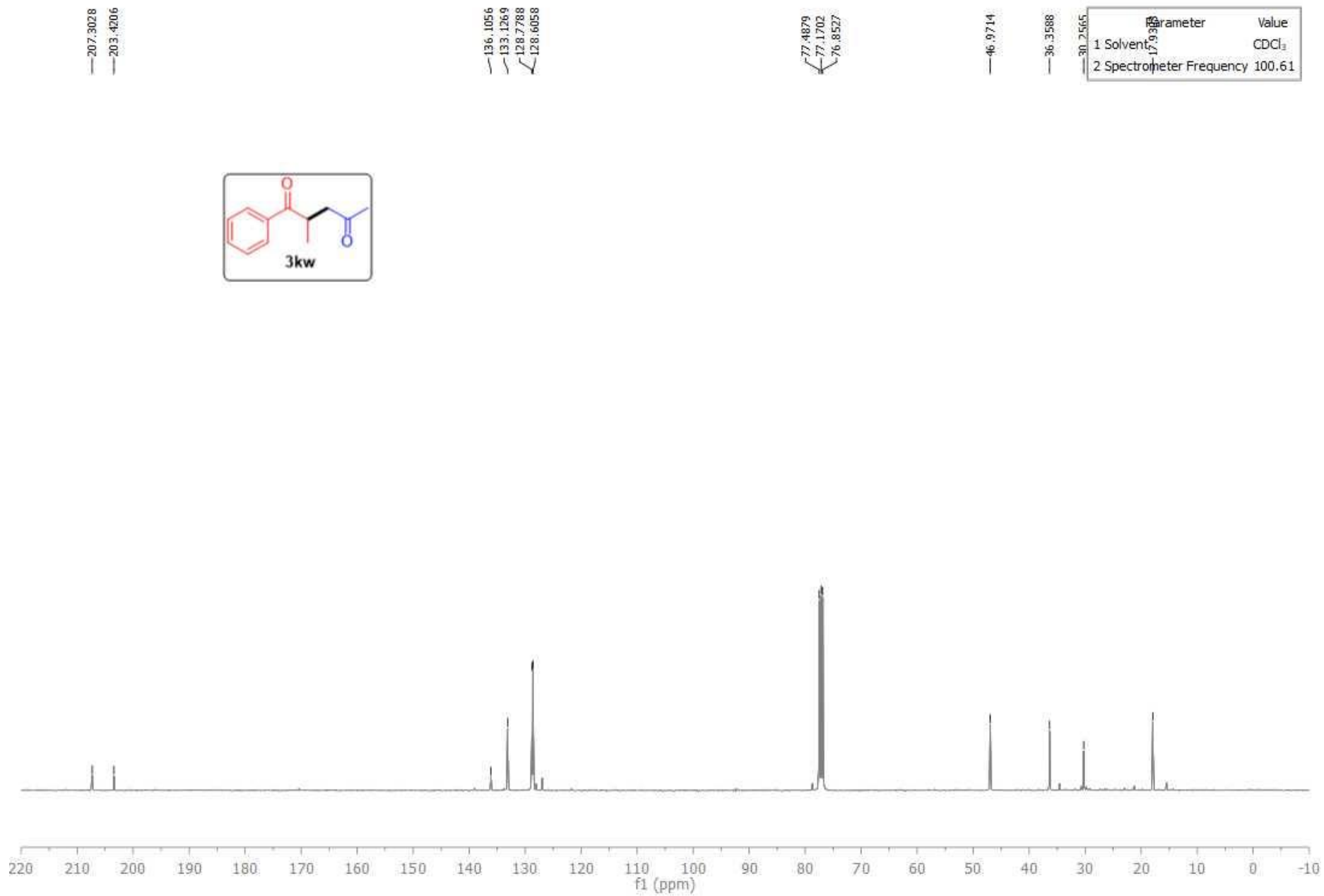


Figure S71. ¹³C NMR spectrum of 2-Methyl-1-phenylpentane-1,4-dione (**3kw**).

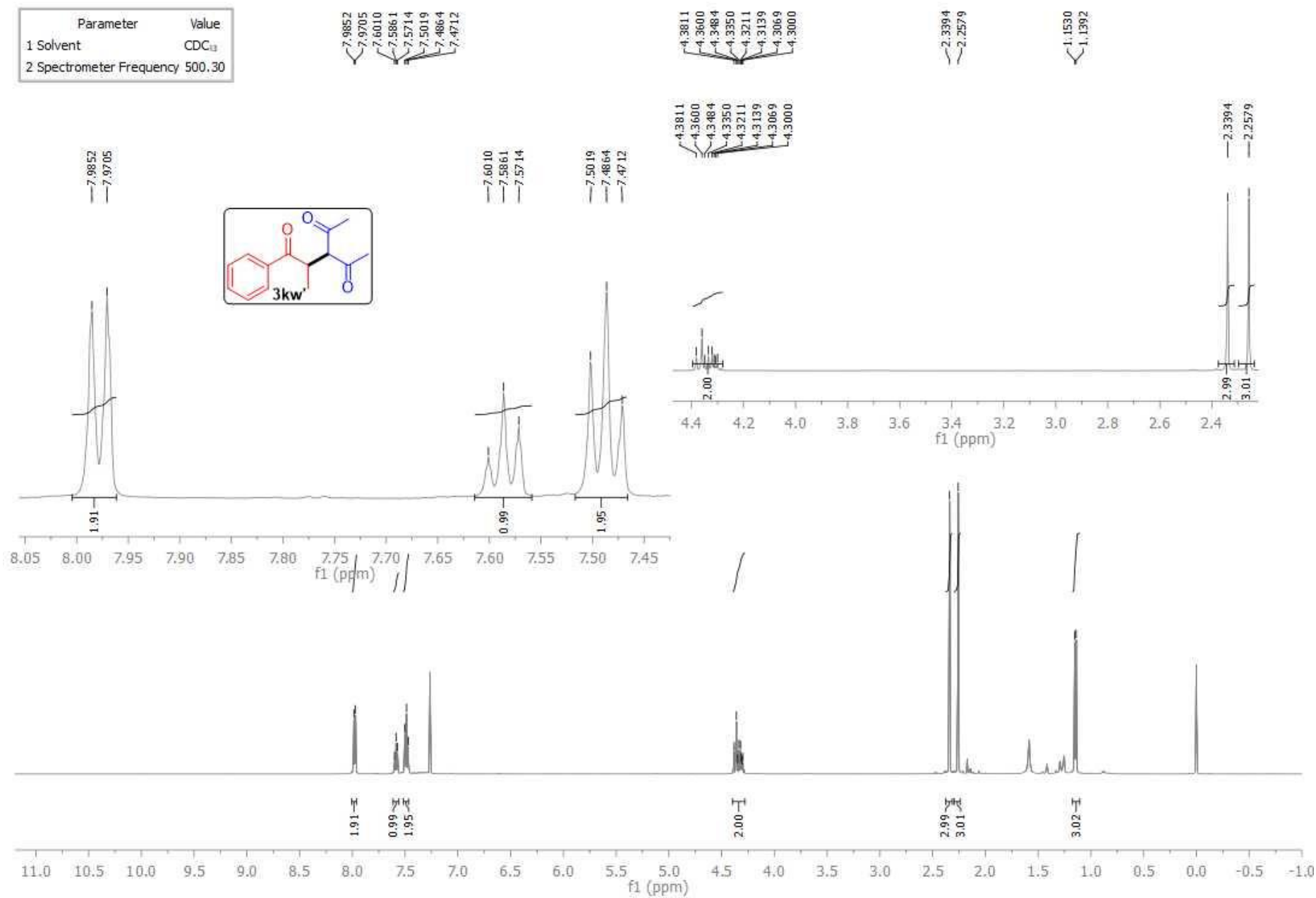


Figure S72. ^1H NMR spectrum of 3-Acetyl-2-methyl-1-phenylpentane-1,4-dione (**3kw'**).

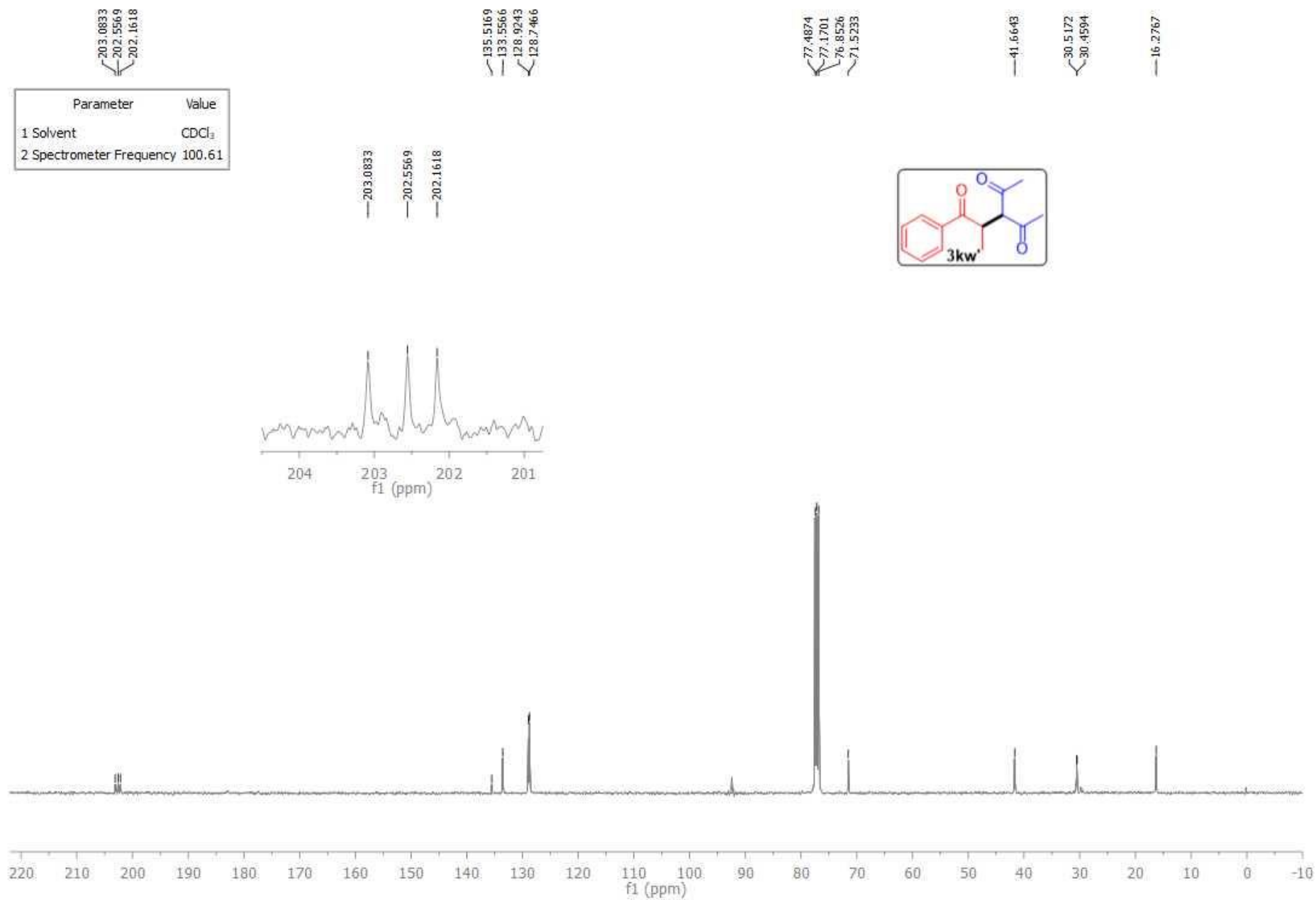


Figure S73. ¹³C NMR spectrum of 3-Acetyl-2-methyl-1-phenylpentane-1,4-dione (**3kw'**).

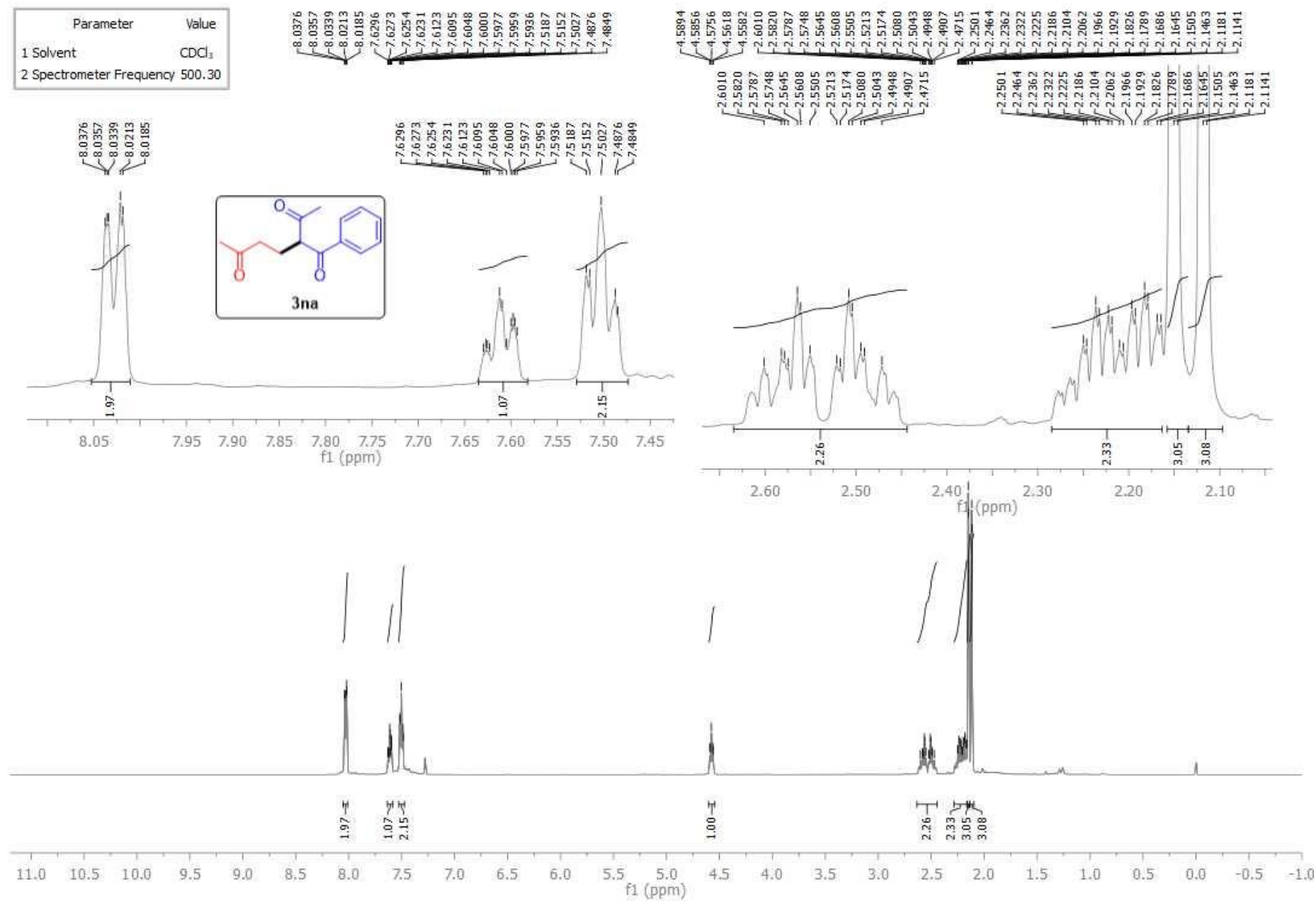


Figure S74. ¹H NMR spectrum of 3-Benzoylheptane-2,6-dione (**3na**).

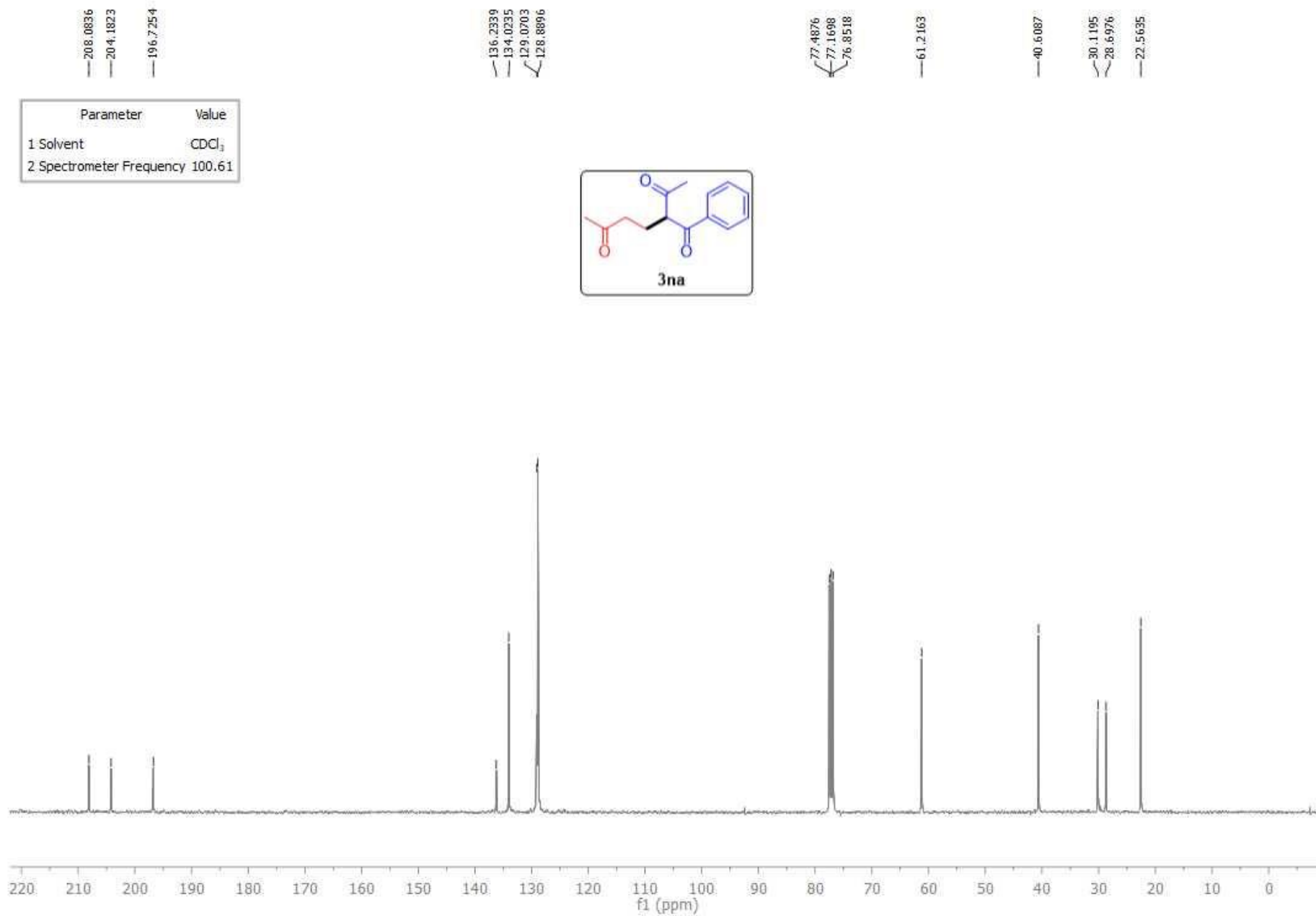


Figure S75. ¹³C NMR spectrum of 3-Benzoylheptane-2,6-dione (**3na**).

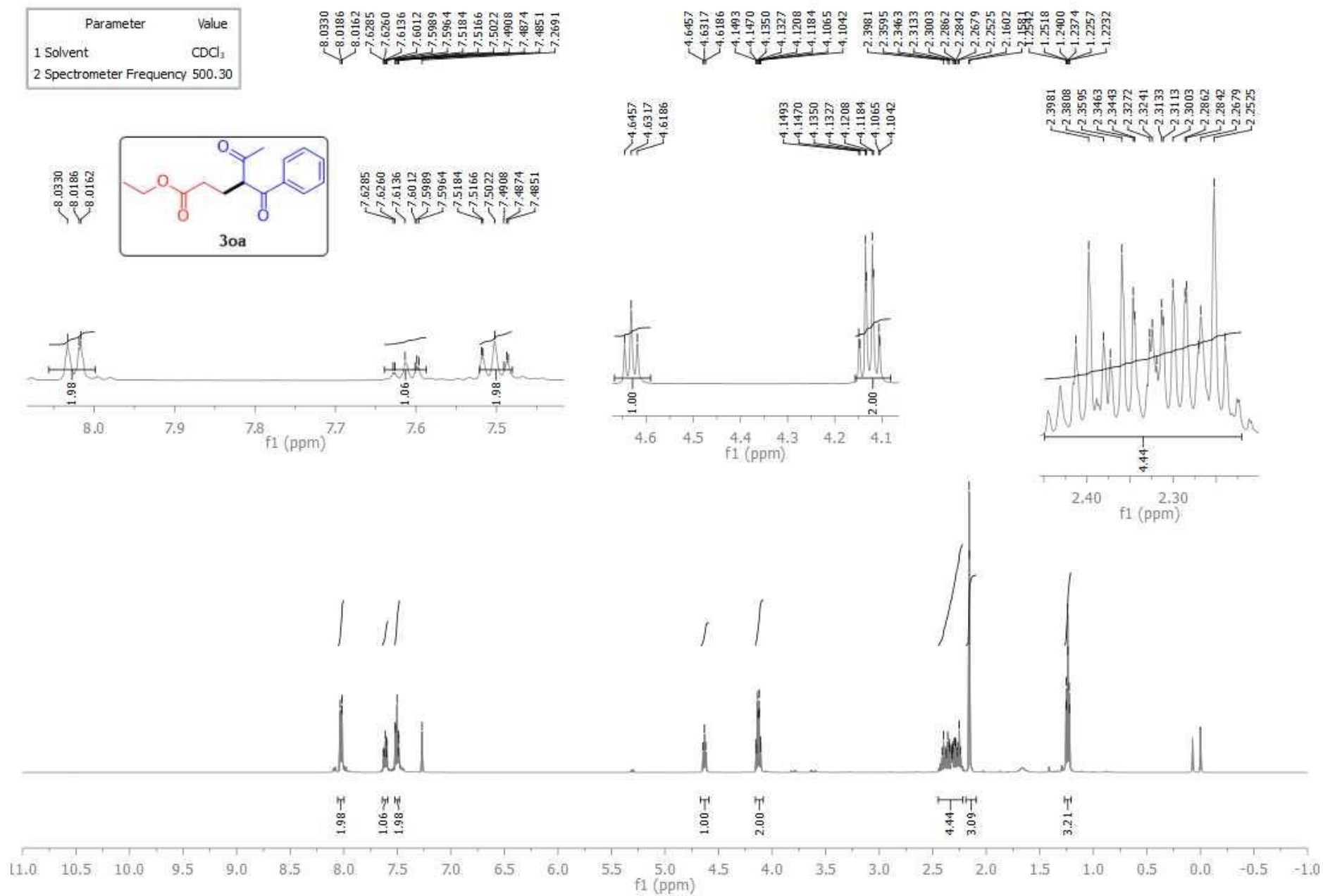


Figure S76. ¹H NMR spectrum of Ethyl 4-benzoyl-5-oxohexanoate (**30a**)

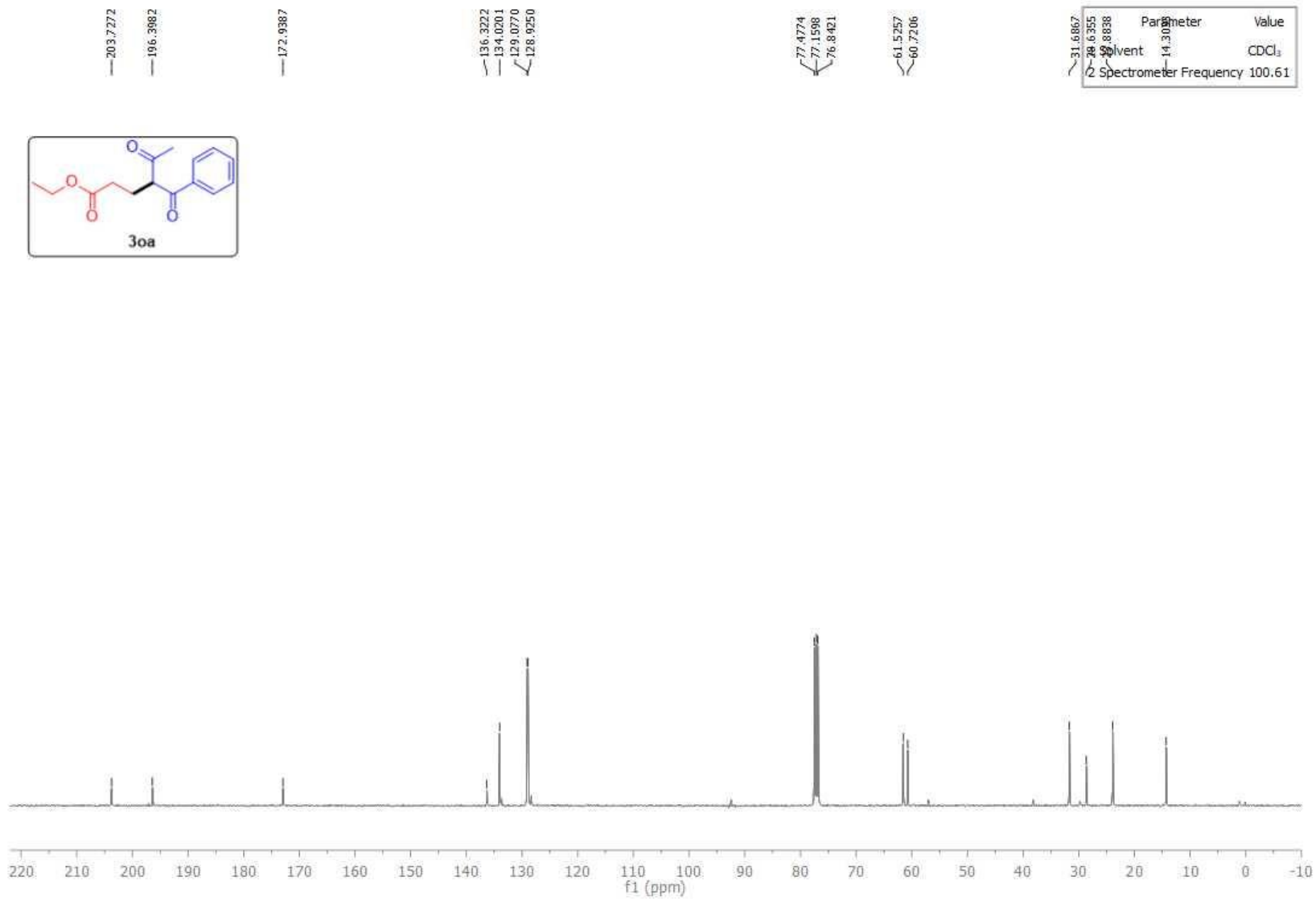


Figure S77. ¹³C NMR spectrum of Ethyl 4-benzoyl-5-oxohexanoate (**30a**).

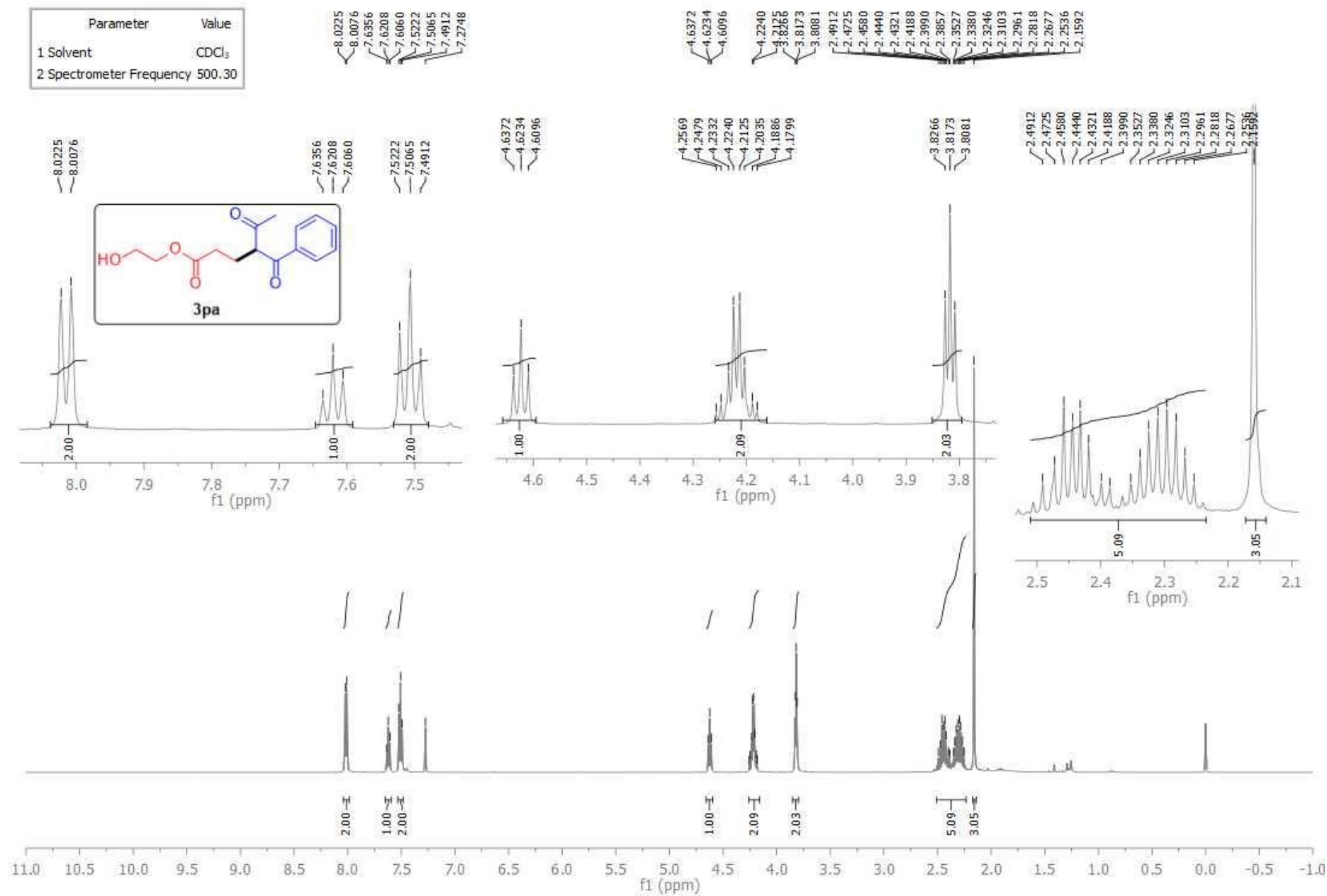


Figure S78. ¹H NMR spectrum of 2-Hydroxyethyl 4-benzoyl-5-oxohexanoate (**3pa**).

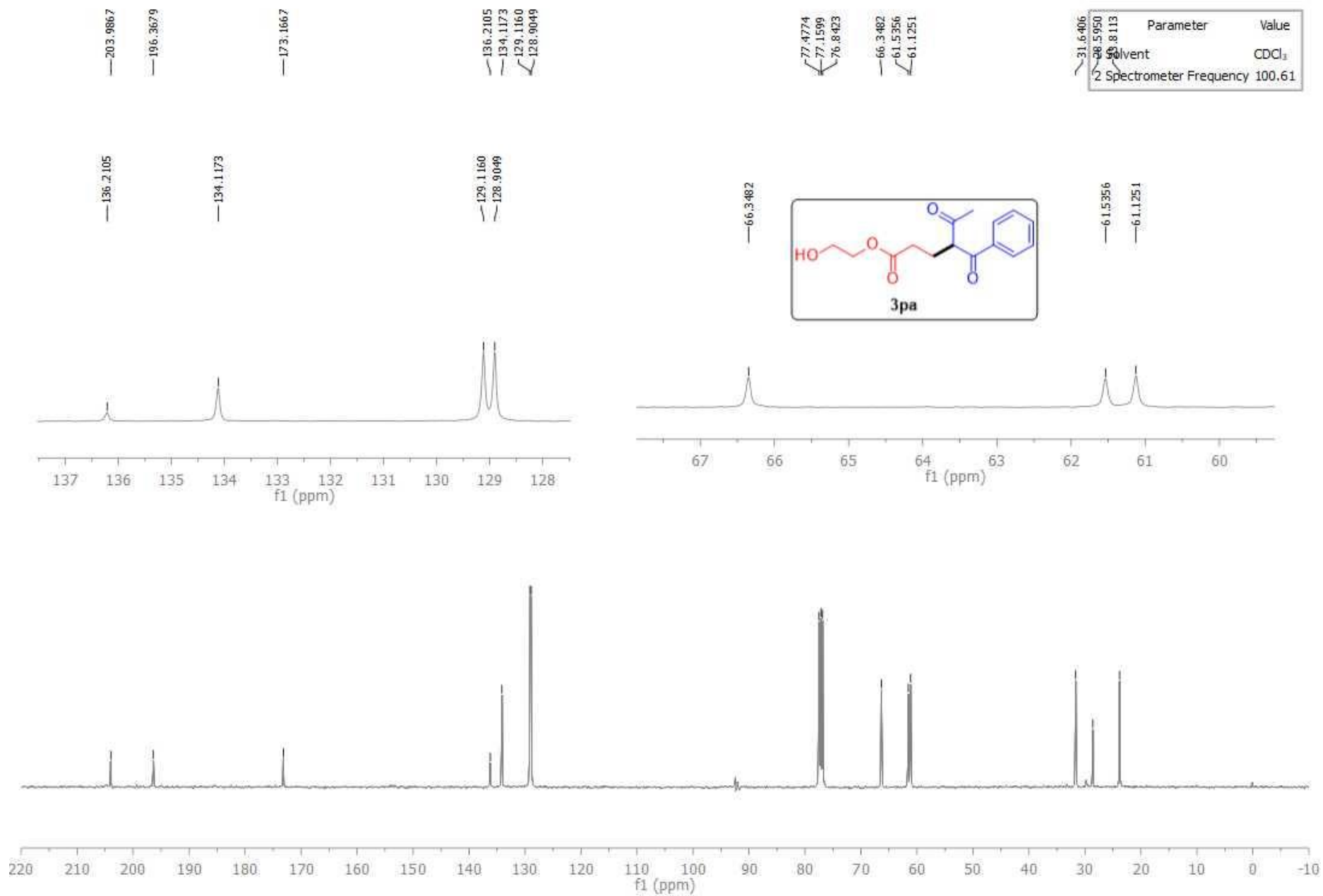


Figure S79. ¹³C NMR spectrum of 2-Hydroxyethyl 4-benzoyl-5-oxohexanoate (**3pa**).

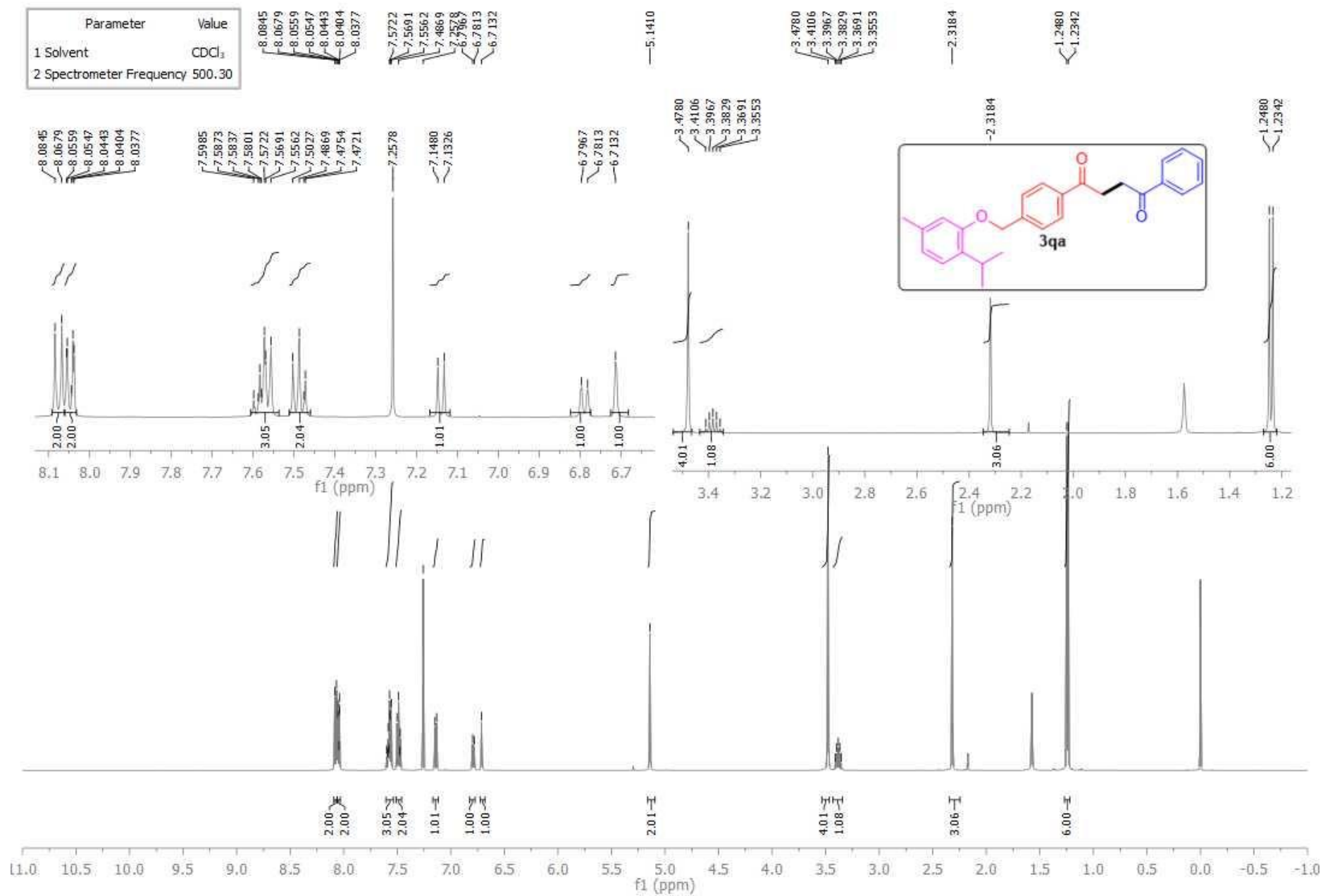


Figure S80. ¹H NMR spectrum of 1-(4-((2-Isopropyl-5-methylphenoxy)methyl)phenyl)-4-phenylbutane-1,4-dione (**3qa**).

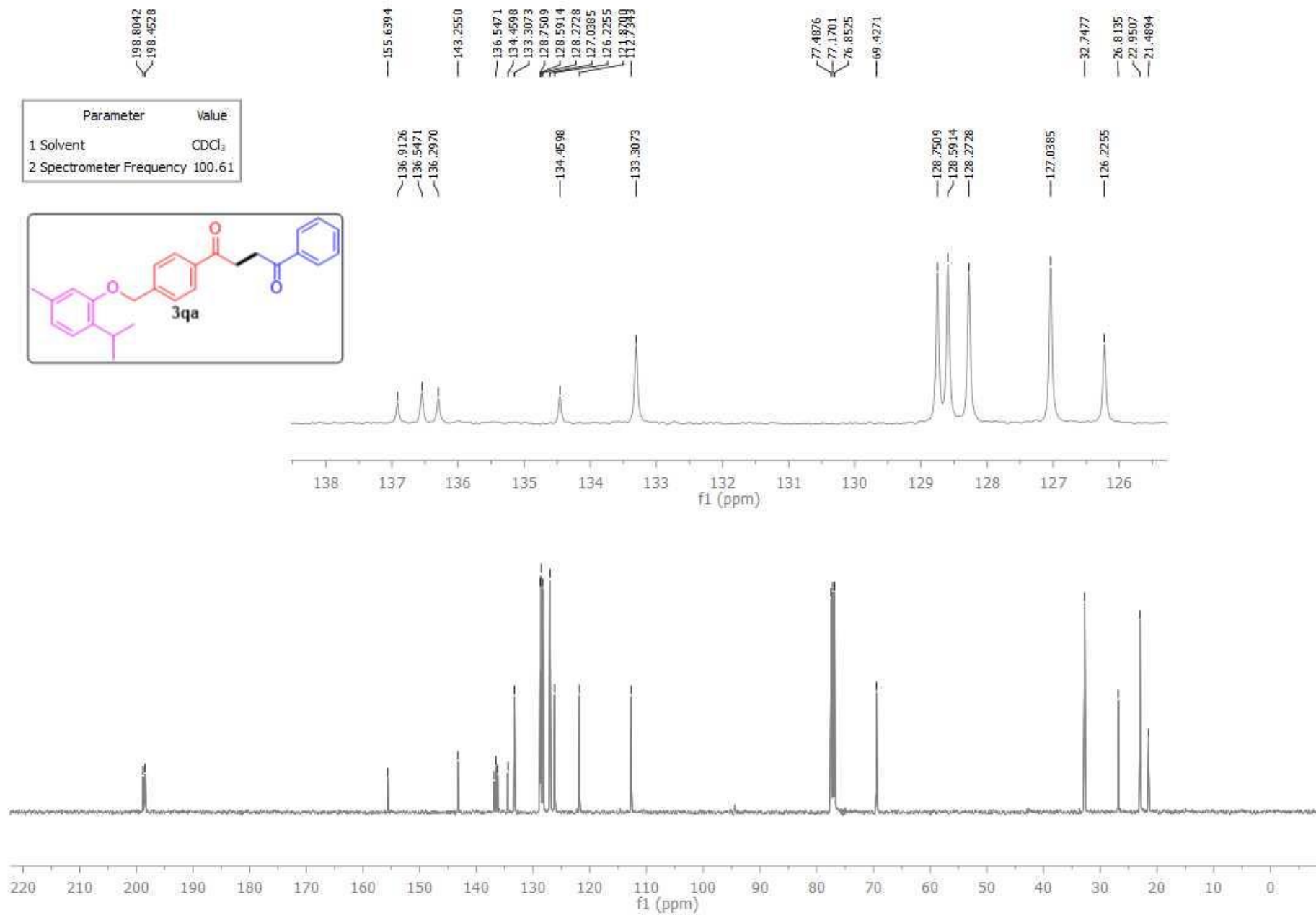


Figure S81. ¹³C NMR spectrum of 1-(4-((2-Isopropyl-5-methylphenoxy)methyl)phenyl)-4-phenylbutane-1,4-dione (**3qa**).

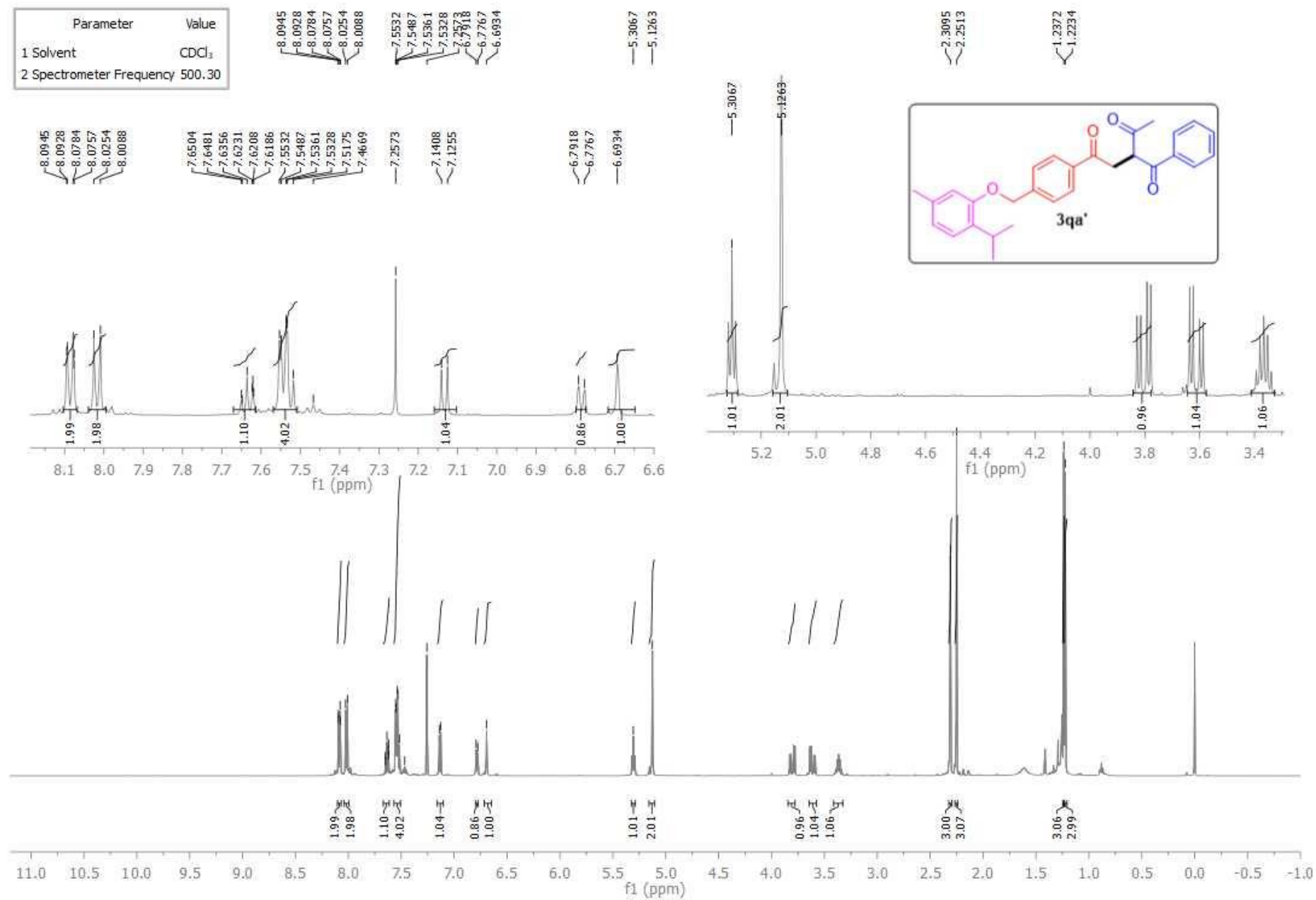


Figure S82. ¹H NMR spectrum of 3-Benzoyl-1-(4-((2-isopropyl-5-methylphenoxy)methyl)phenyl)pentane-1,4-dione (**3qa'**).

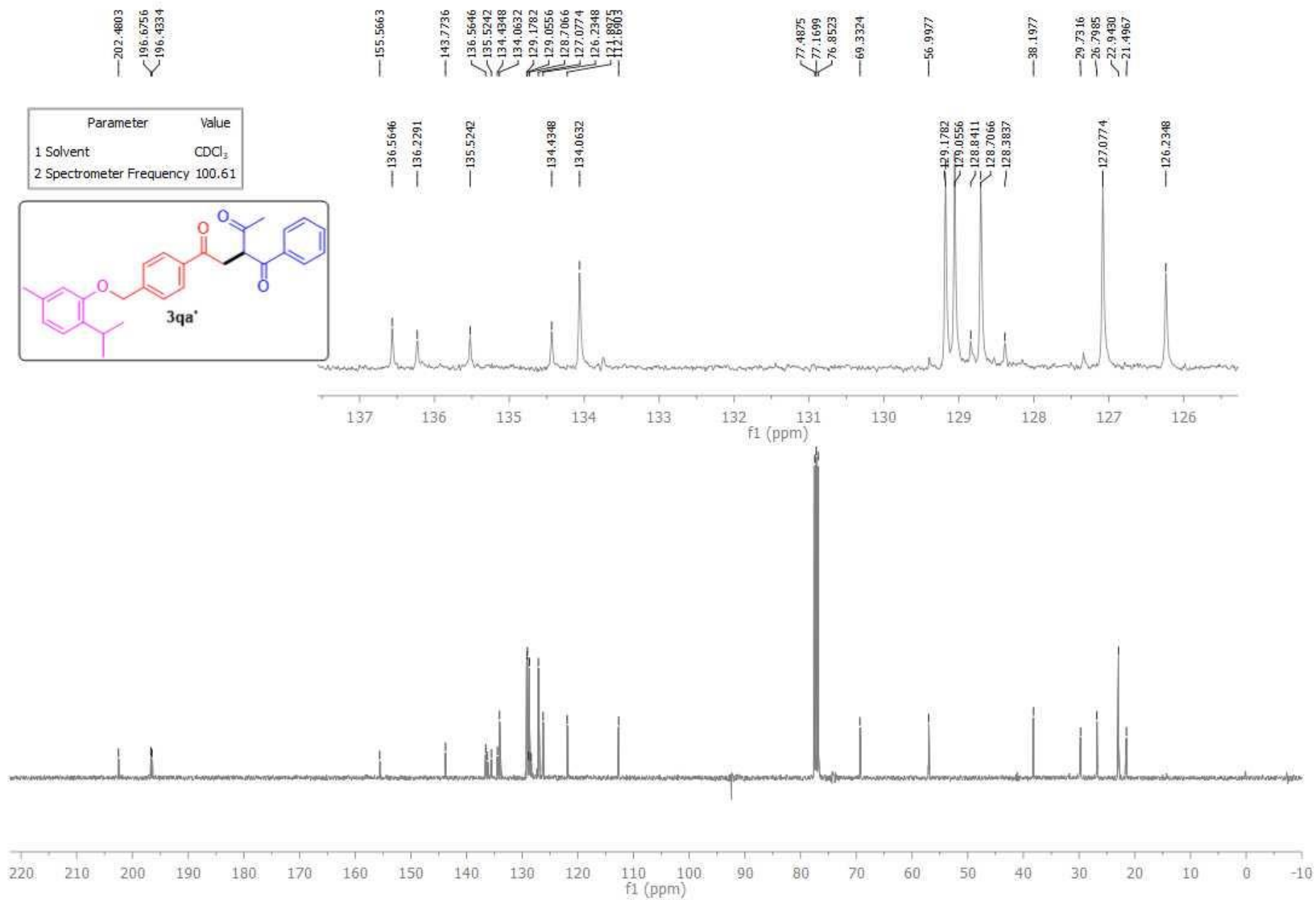


Figure S83. ¹³C NMR spectrum of 3-Benzoyl-1-(4-((2-isopropyl-5-methylphenoxy)methyl)phenyl)pentane-1,4-dione (**3qa'**).

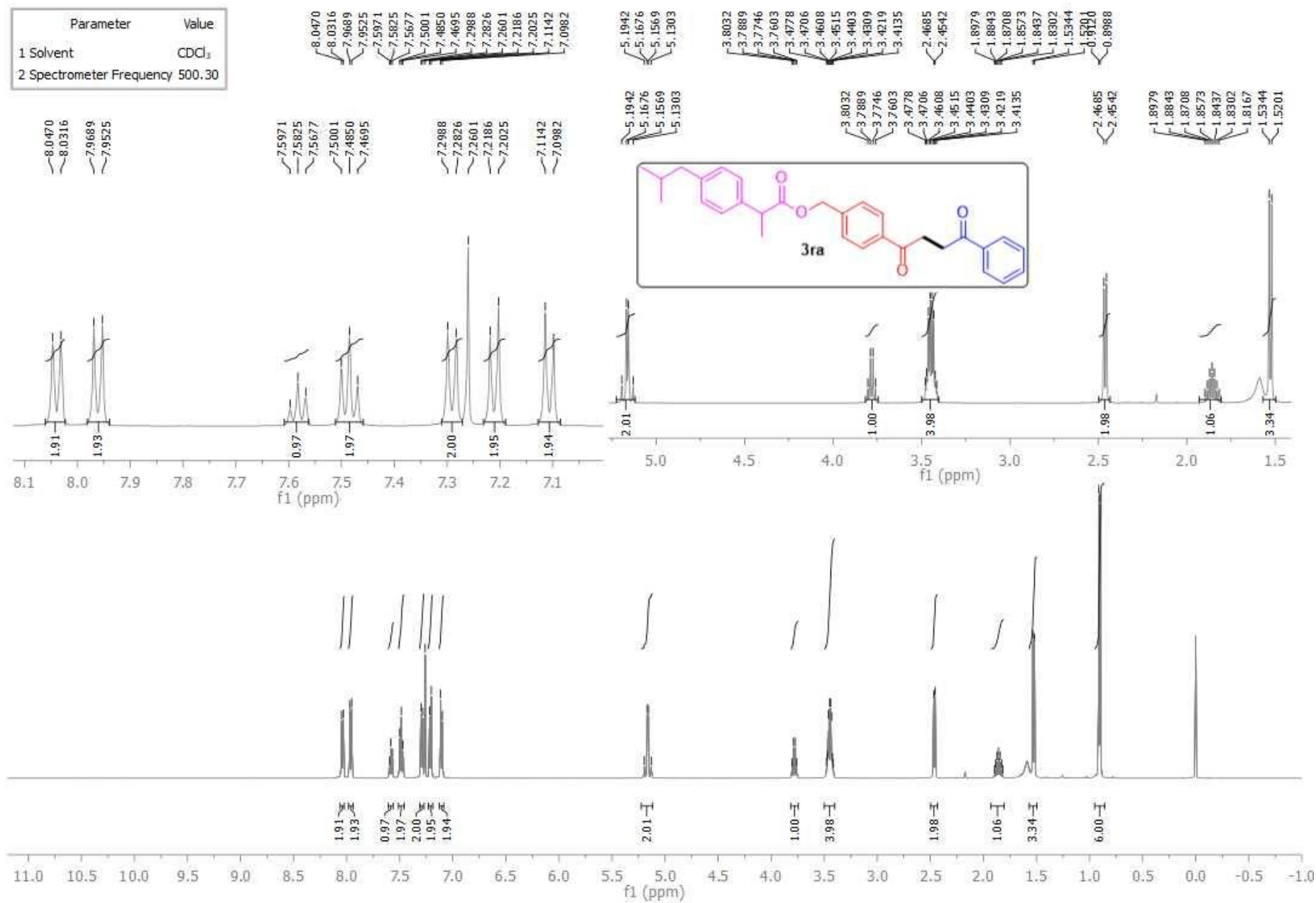


Figure S84. ¹H NMR spectrum of 4-(4-Oxo-4-phenylbutanoyl)benzyl 2-(4-isobutylphenyl)propanoate (**3ra**).

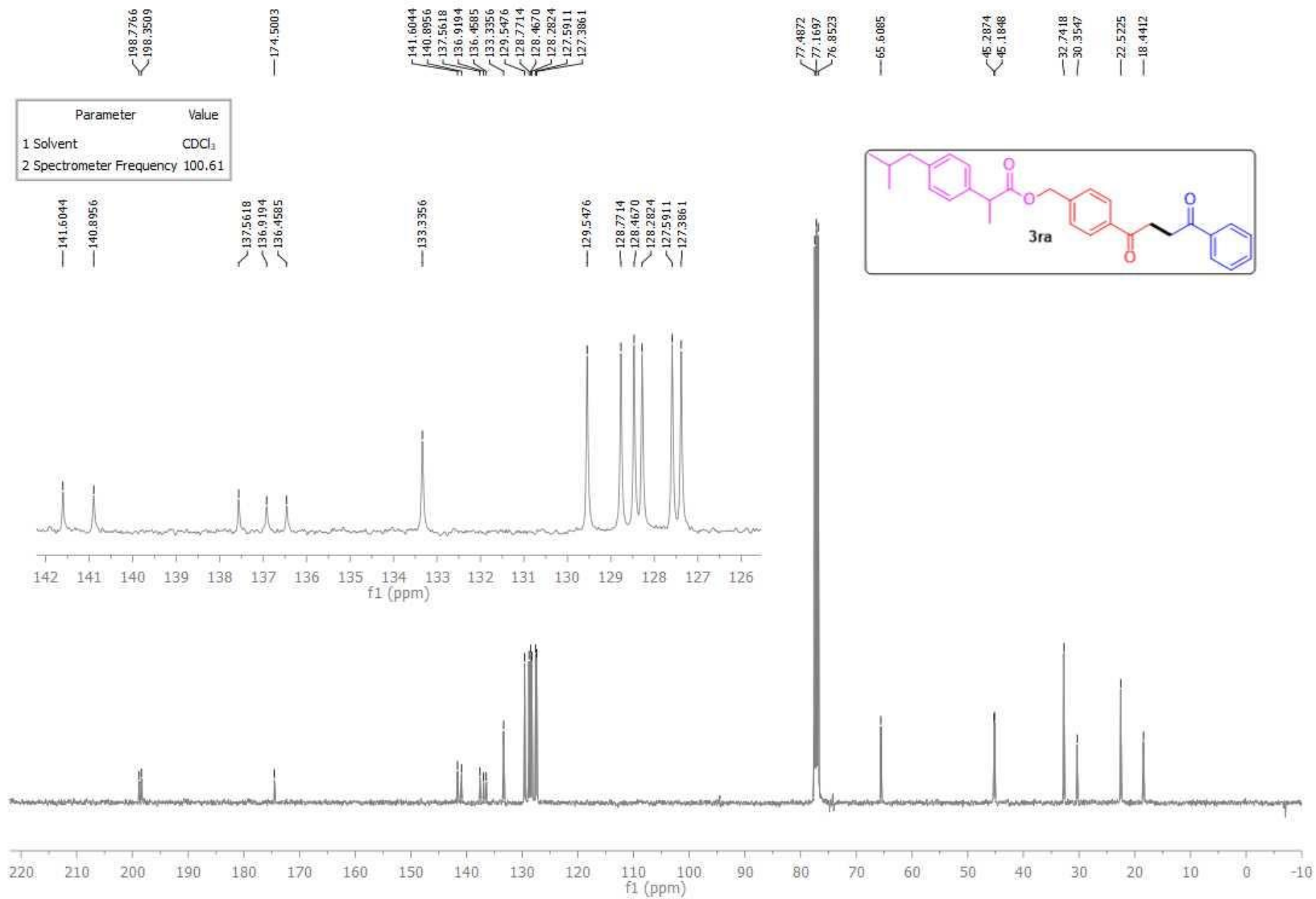


Figure S85. ¹³C NMR spectrum of 4-(4-Oxo-4-phenylbutanoyl)benzyl 2-(4-isobutylphenyl)propanoate (**3ra**).

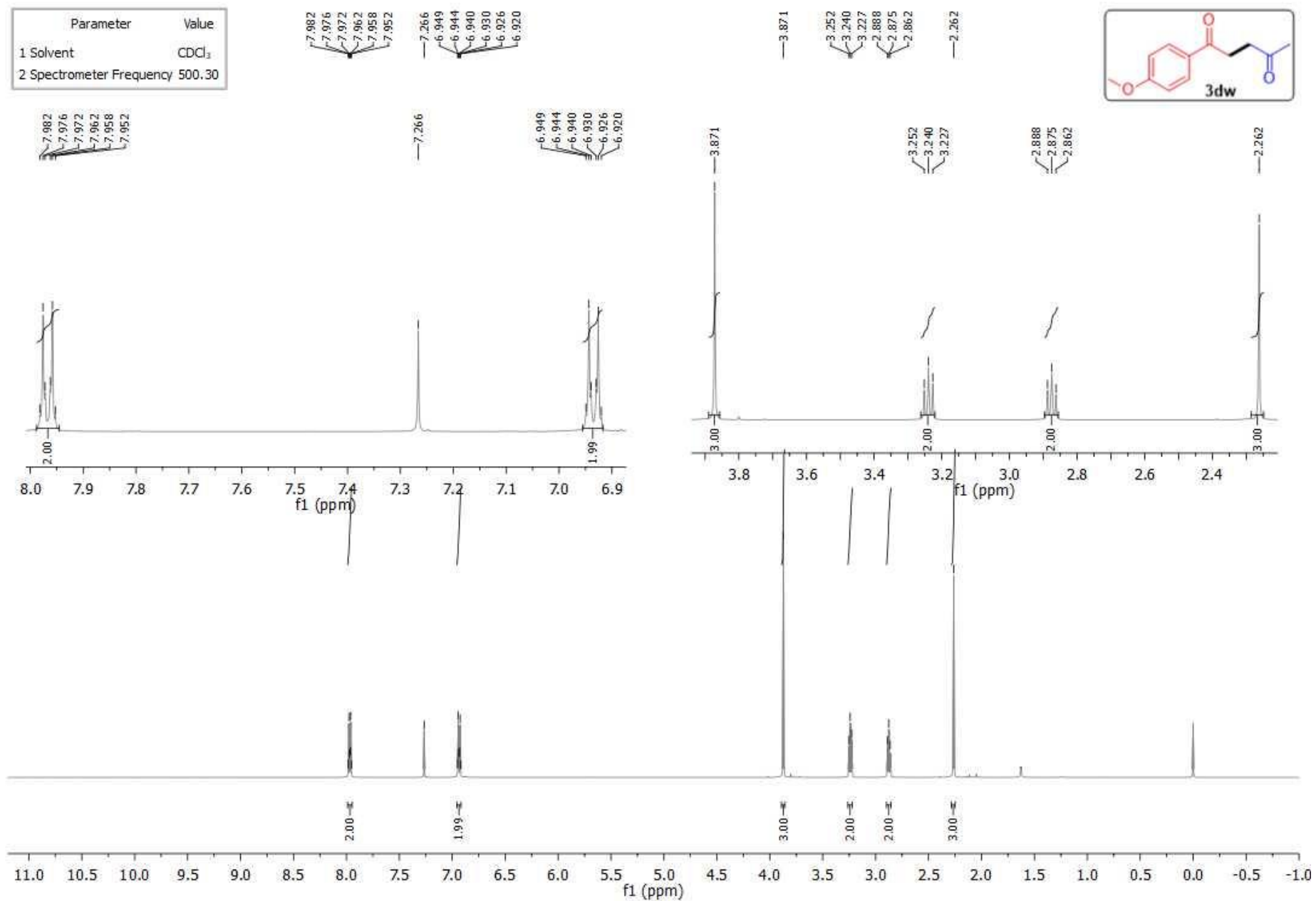


Figure S86. ¹H NMR spectrum of 1-(4-Methoxyphenyl)pentane-1,4-dione (**3dw**).

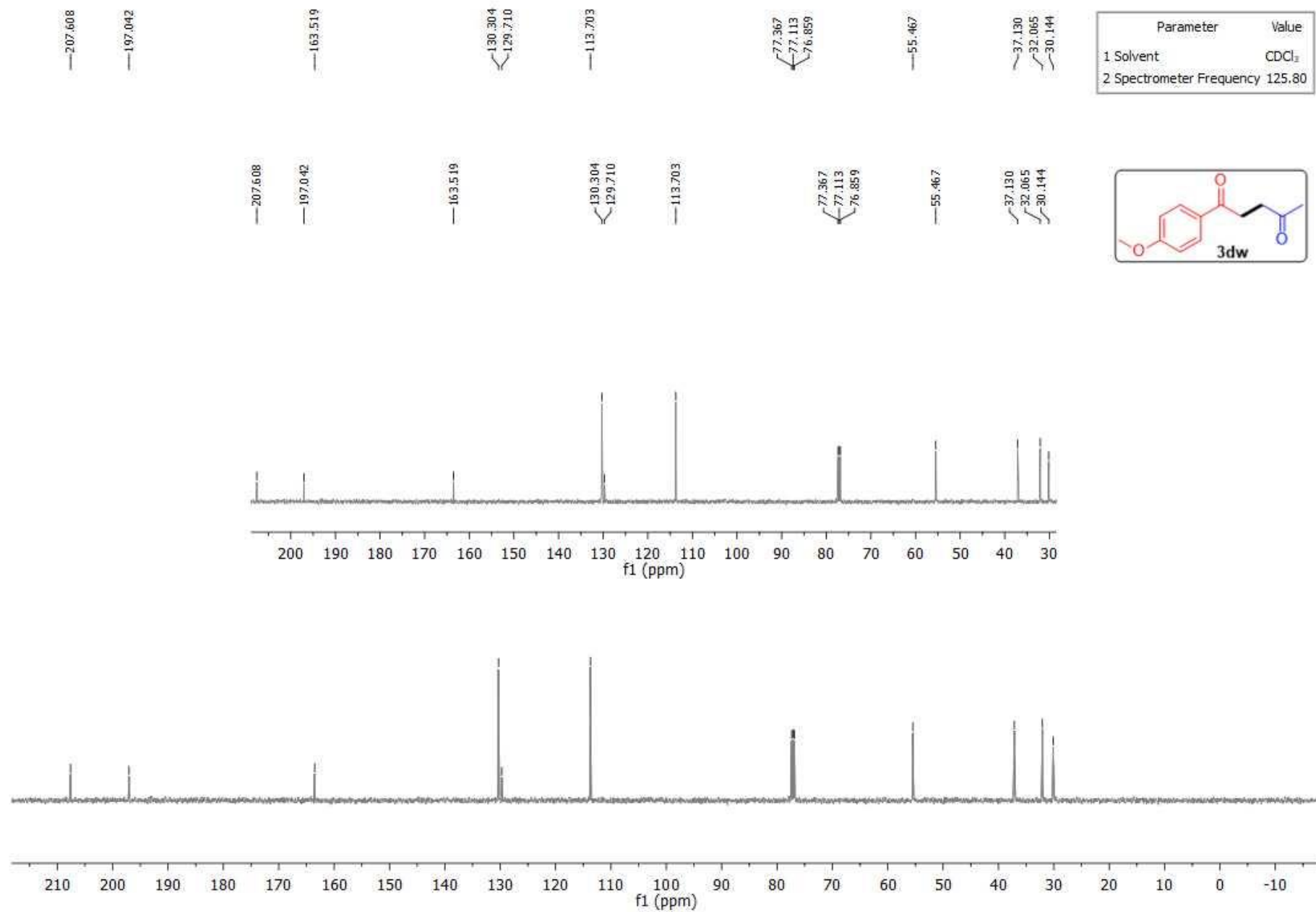


Figure S87. ¹³C NMR spectrum of 1-(4-Methoxyphenyl)pentane-1,4-dione (**3dw**).

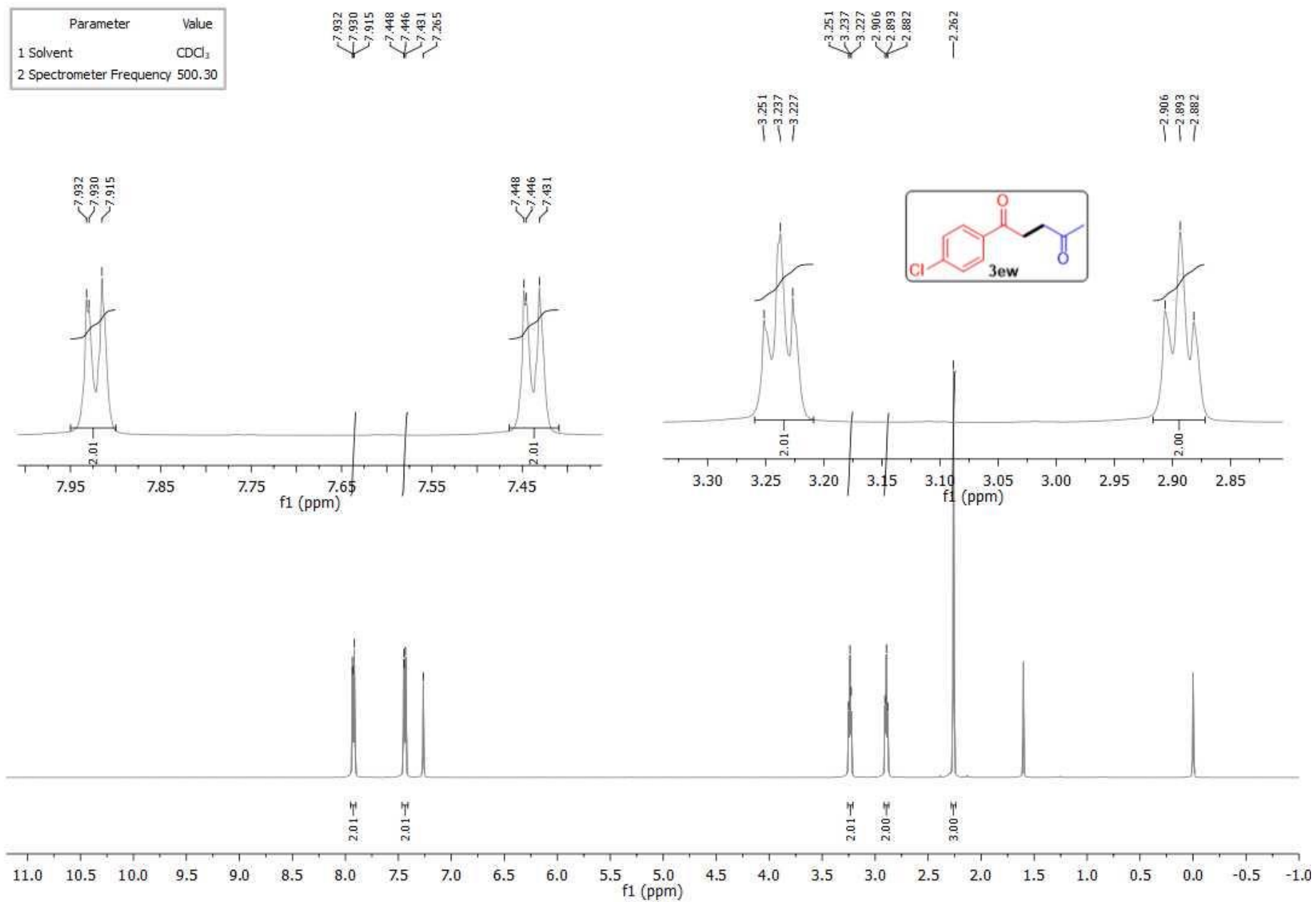


Figure S88. ¹H NMR spectrum of 1-(4-Chlorophenyl)pentane-1,4-dione (**3ew**).

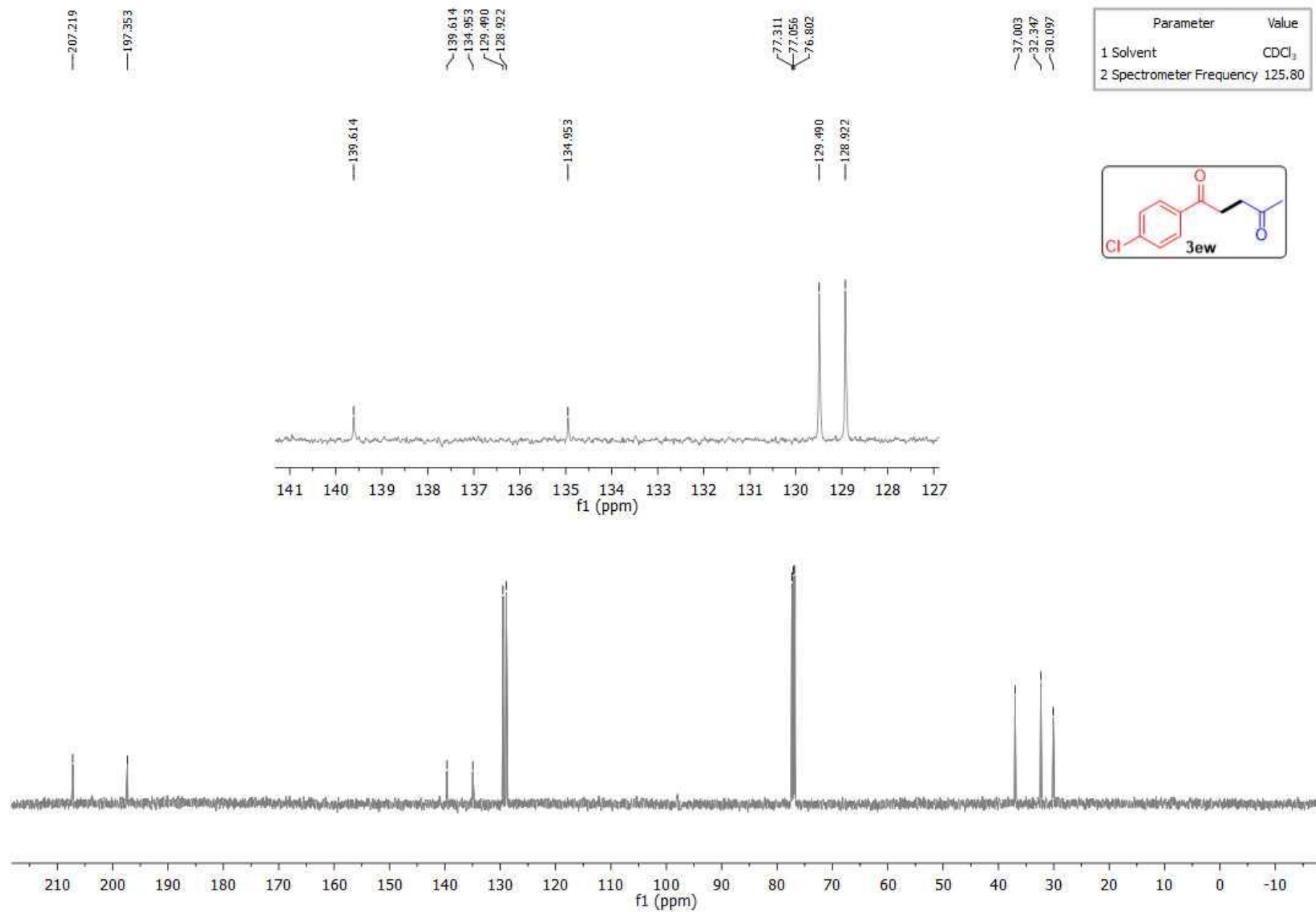


Figure S89. ¹³C NMR spectrum of 1-(4-Chlorophenyl)pentane-1,4-dione (**3ew**).

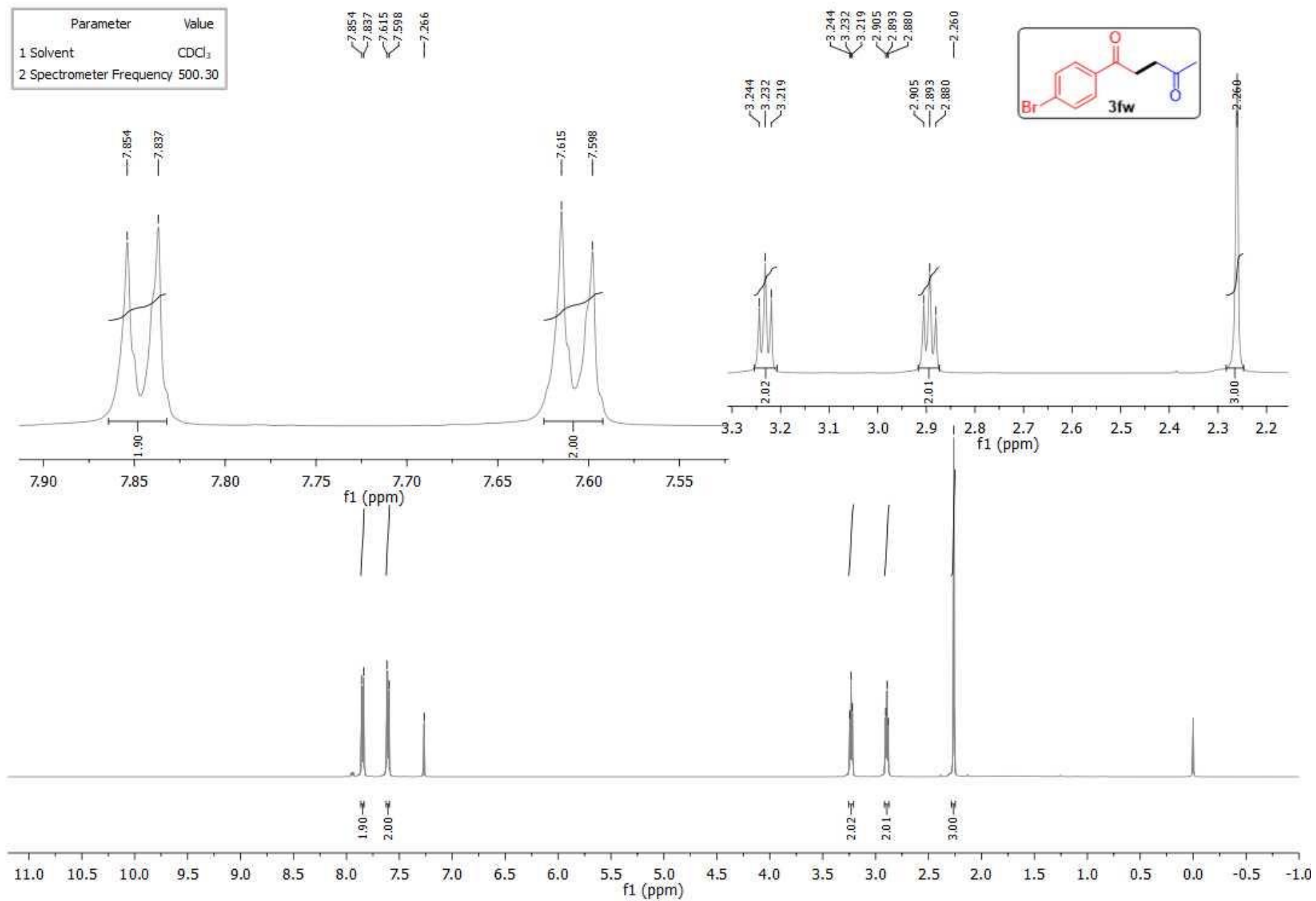


Figure S90. ¹H NMR spectrum of 1-(4-Bromophenyl)pentane-1,4-dione (**3fw**).

Parameter	Value
1 Solvent	CDCl ₃
2 Spectrometer Frequency	125.80

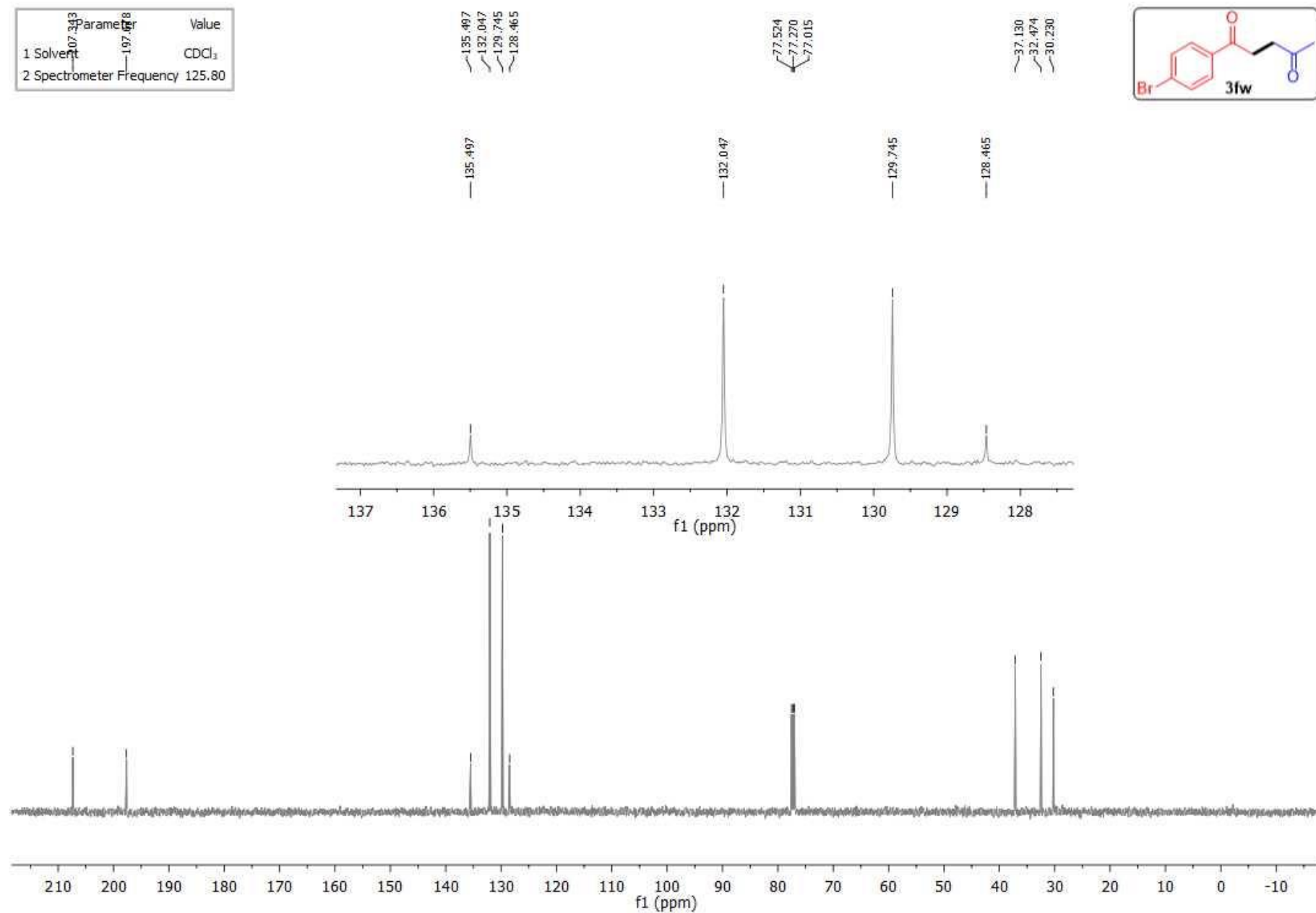


Figure S91. ¹³C NMR spectrum of 1-(4-Bromophenyl)pentane-1,4-dione (**3fw**).

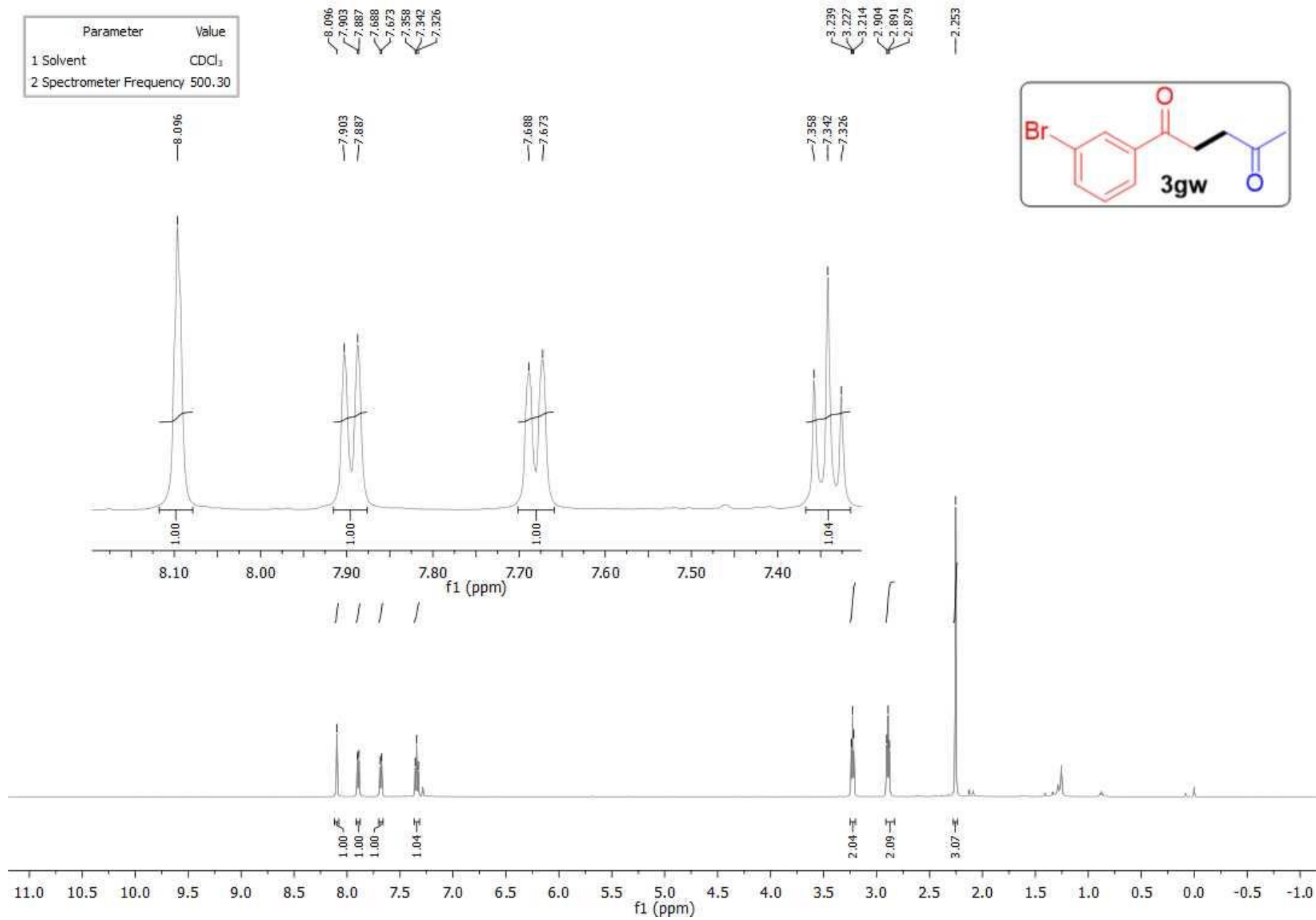


Figure S92. ¹H NMR spectrum of 1-(3-Bromophenyl)pentane-1,4-dione (**3gw**).

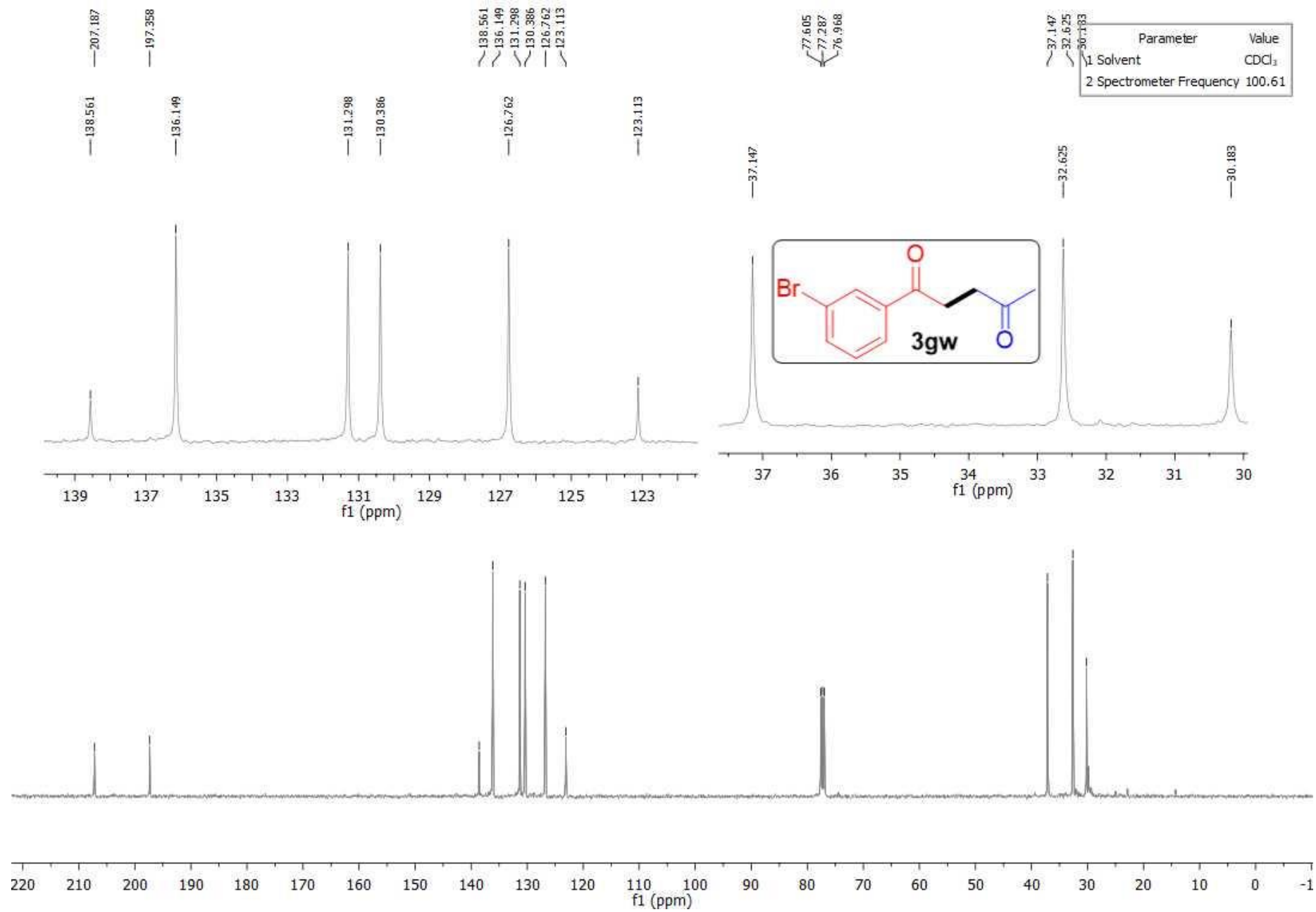


Figure S93. ¹³C NMR spectrum of 1-(3-Bromophenyl)pentane-1,4-dione (**3gw**).

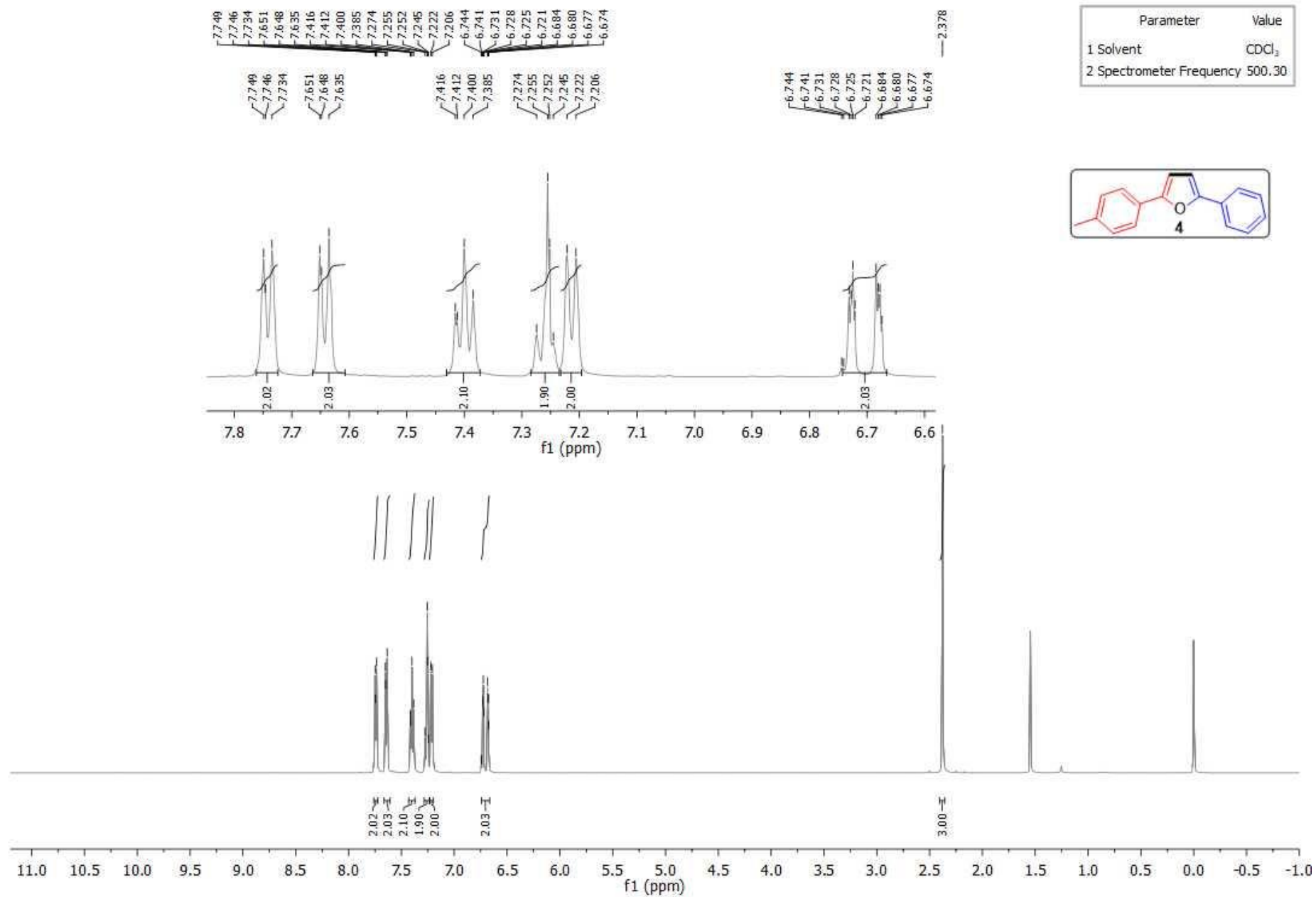


Figure S94. ¹H NMR spectrum of 2-Phenyl-5-(*p*-tolyl)furan (**4**).

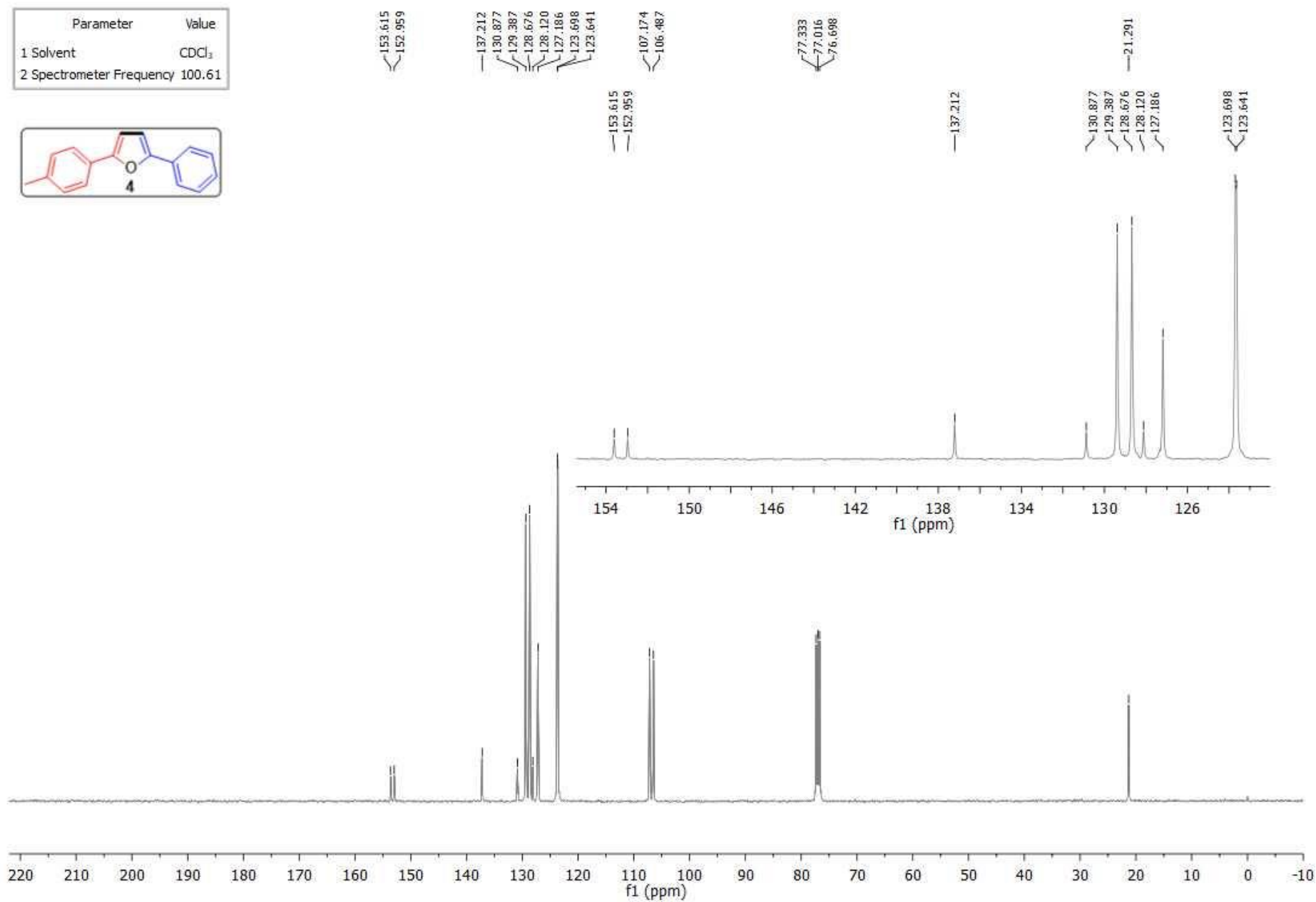


Figure S95. ¹³C NMR spectrum of 2-Phenyl-5-(*p*-tolyl)furan (**4**).

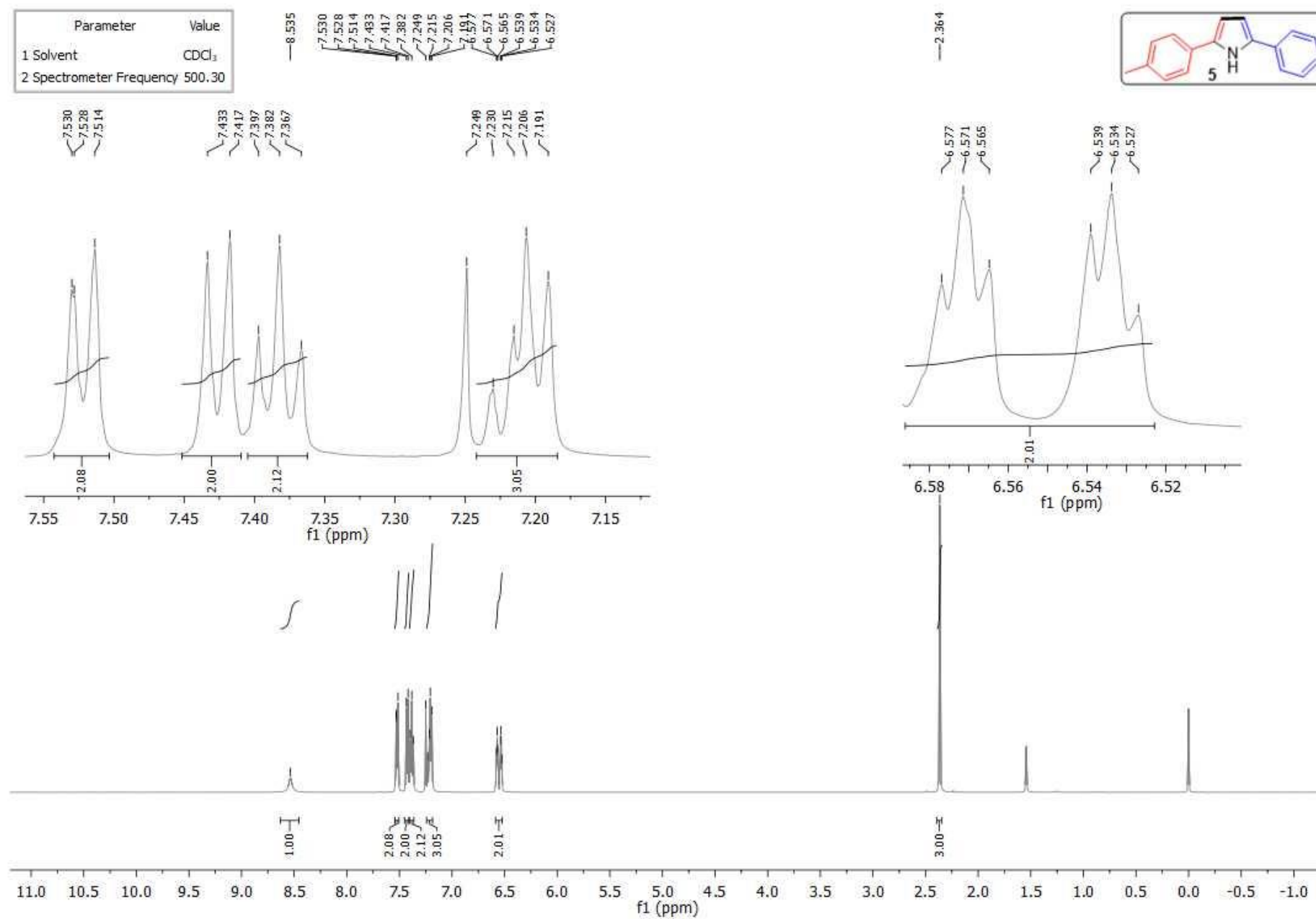


Figure S96. ¹H NMR spectrum of 2-Phenyl-5-(*p*-tolyl)-1*H*-pyrrole (5).

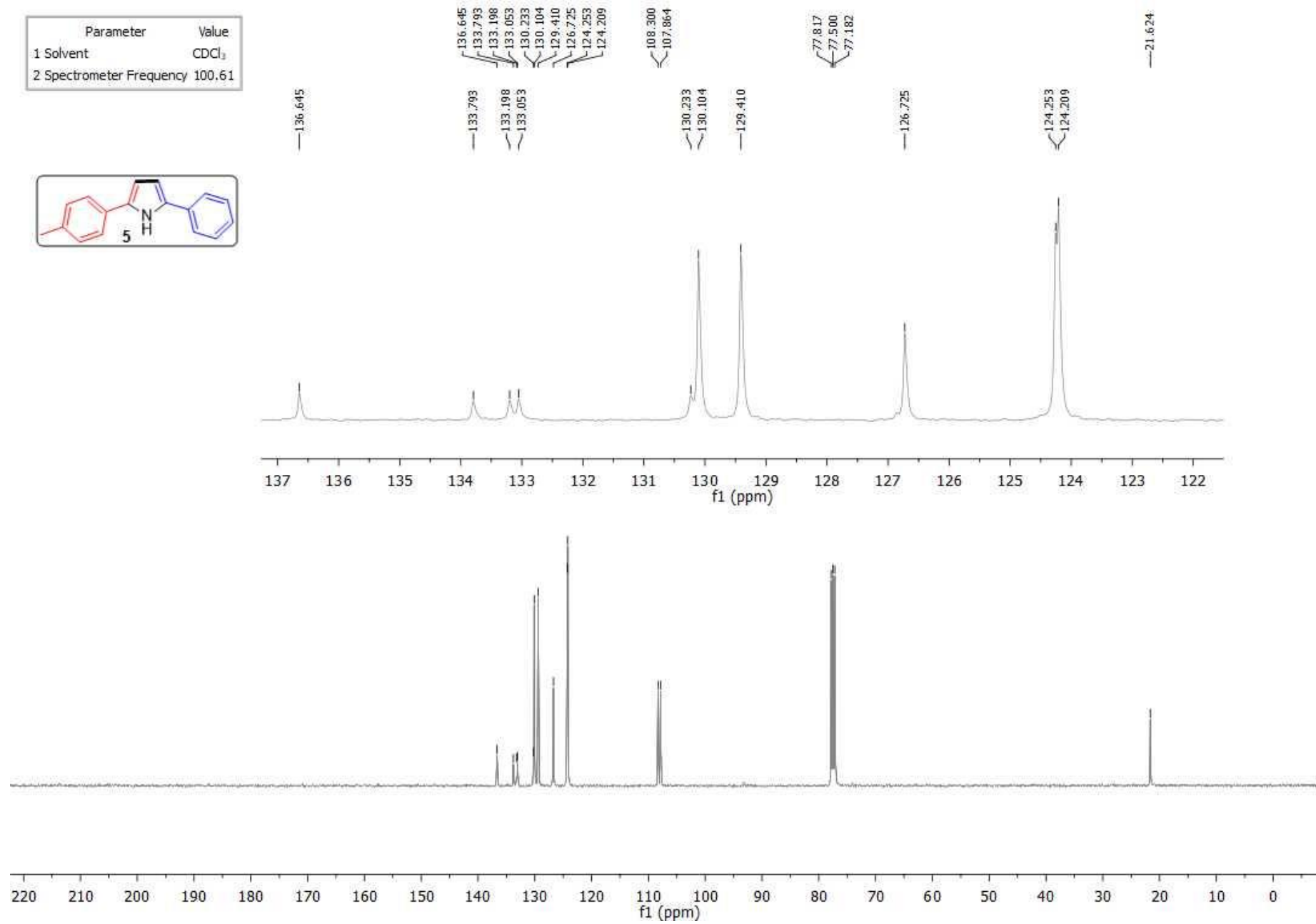


Figure S97. ¹³C NMR spectrum of 2-Phenyl-5-(*p*-tolyl)-1*H*-pyrrole (**5**).

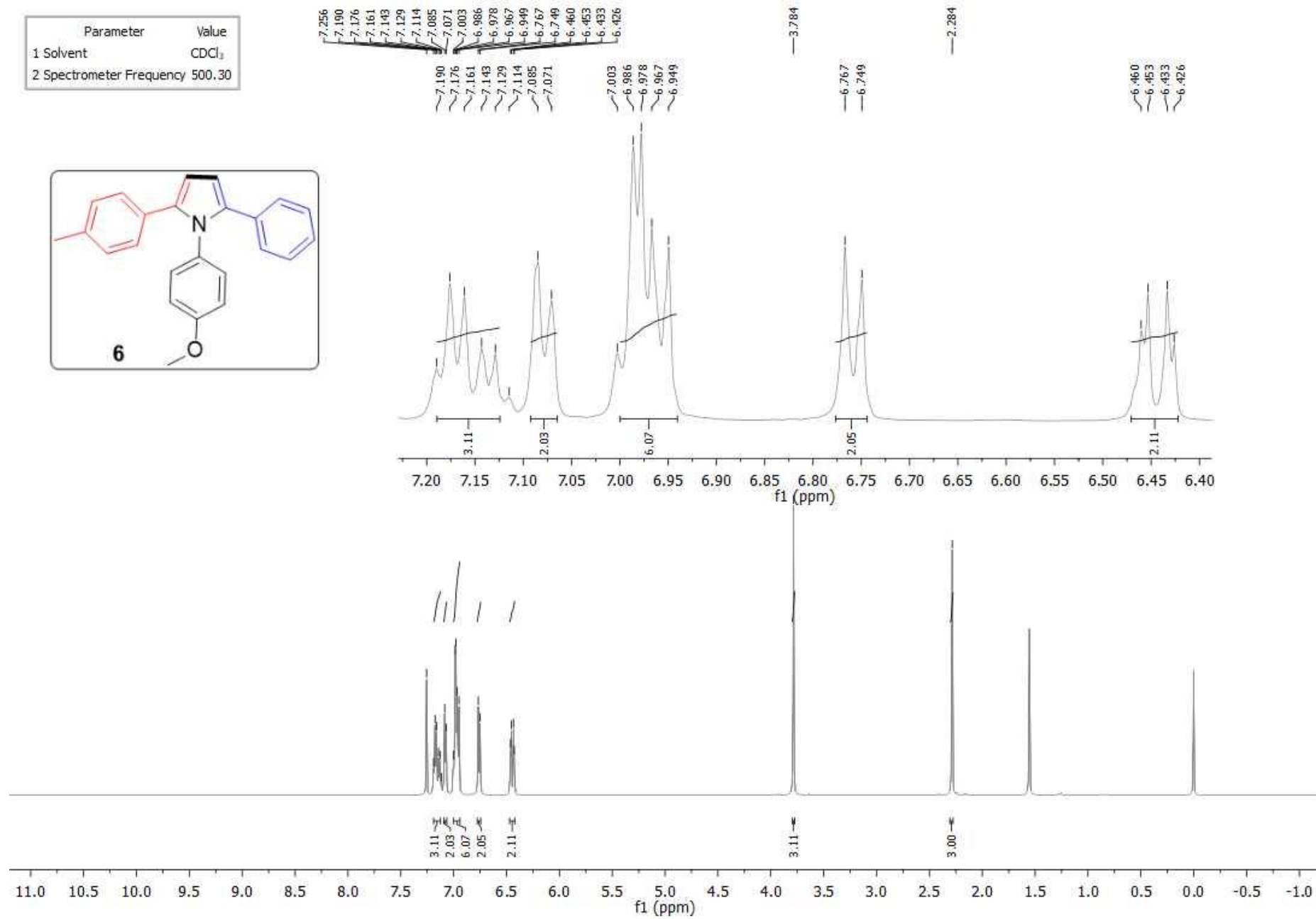


Figure S98. ¹H NMR spectrum of 1-(4-Methoxyphenyl)-2-phenyl-5-(*p*-tolyl)-1*H*-pyrrole (**6**).

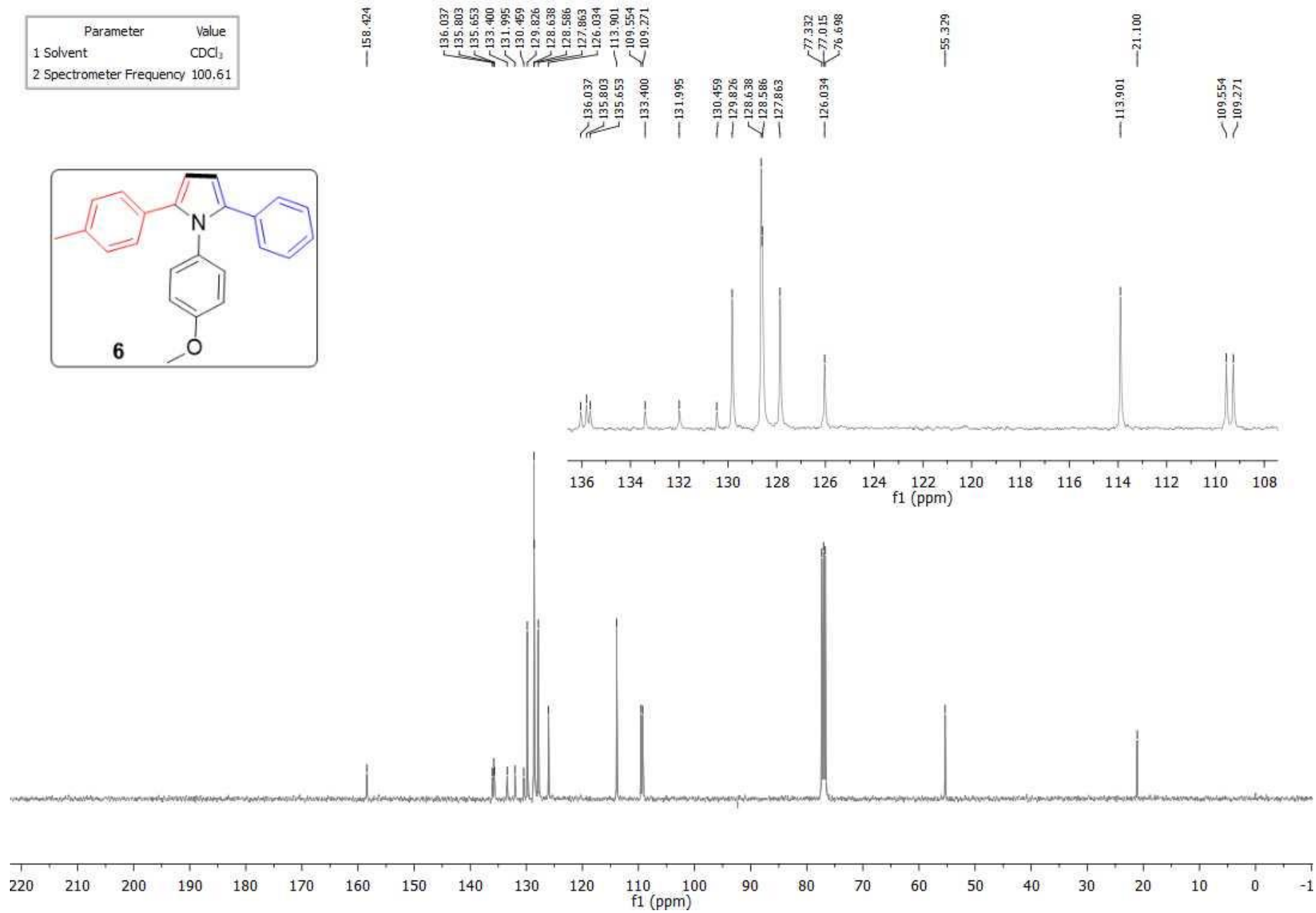


Figure S99. ¹³C NMR spectrum of 1-(4-Methoxyphenyl)-2-phenyl-5-(*p*-tolyl)-1*H*-pyrrole (**6**).

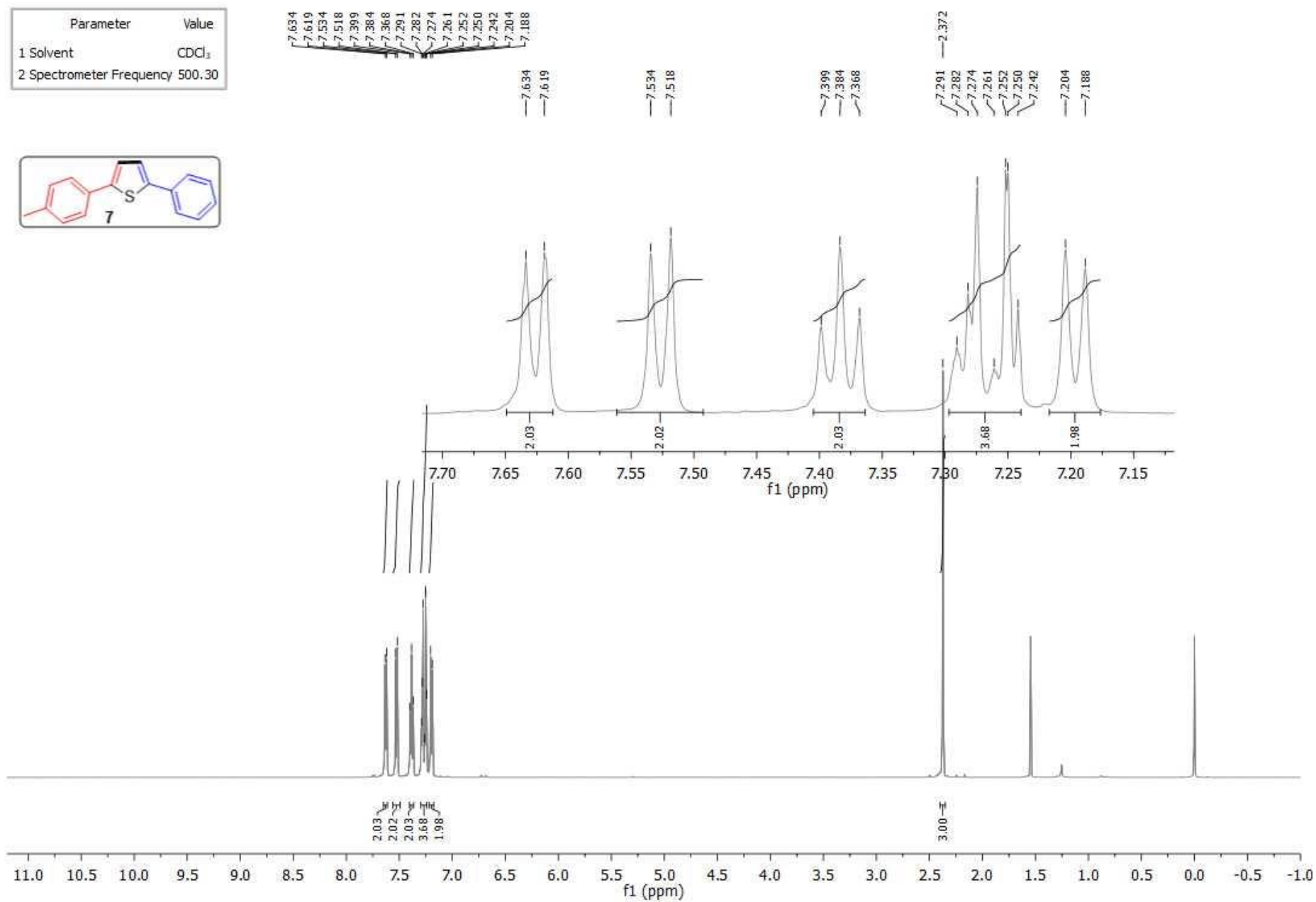


Figure S100. ¹H NMR spectrum of 2-Phenyl-5-(*p*-tolyl)thiophene (7).

Parameter	Value
1 Solvent	CDCl ₃
2 Spectrometer Frequency	100.61

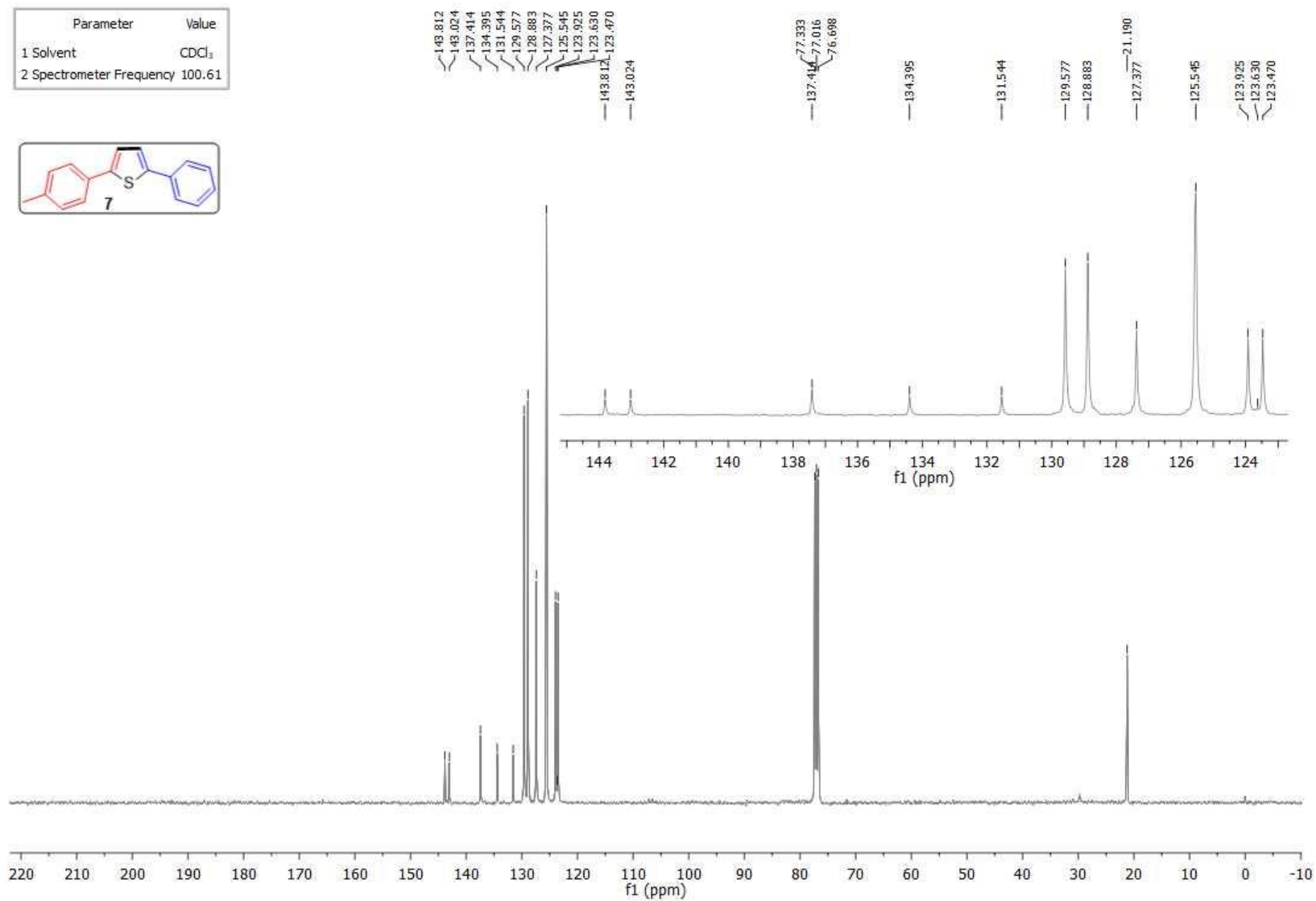


Figure S101. ¹³C NMR spectrum of 2-Phenyl-5-(*p*-tolyl)thiophene (7).

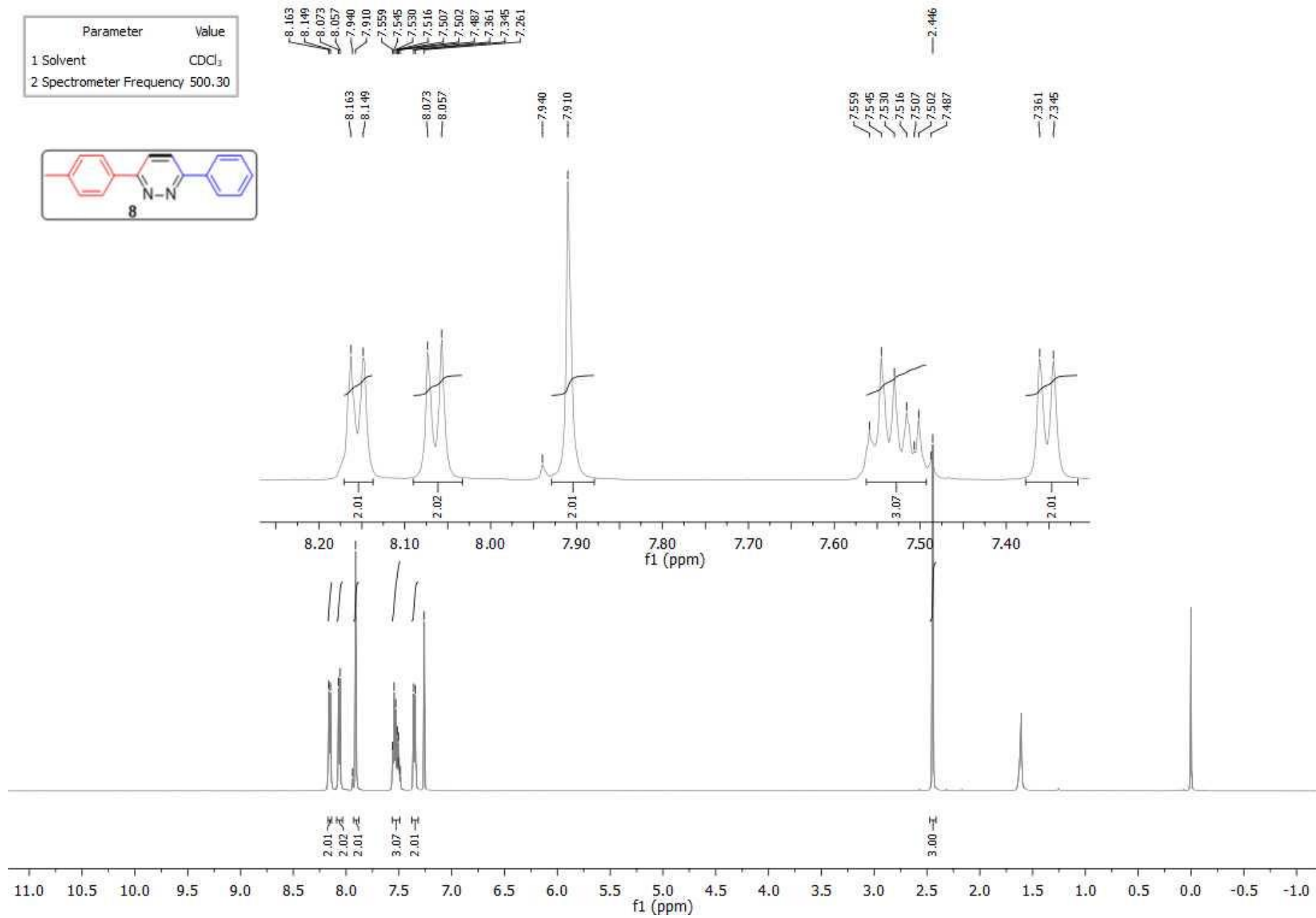


Figure S102. ¹H NMR spectrum of 3-Phenyl-6-(*p*-tolyl)pyridazine (8).

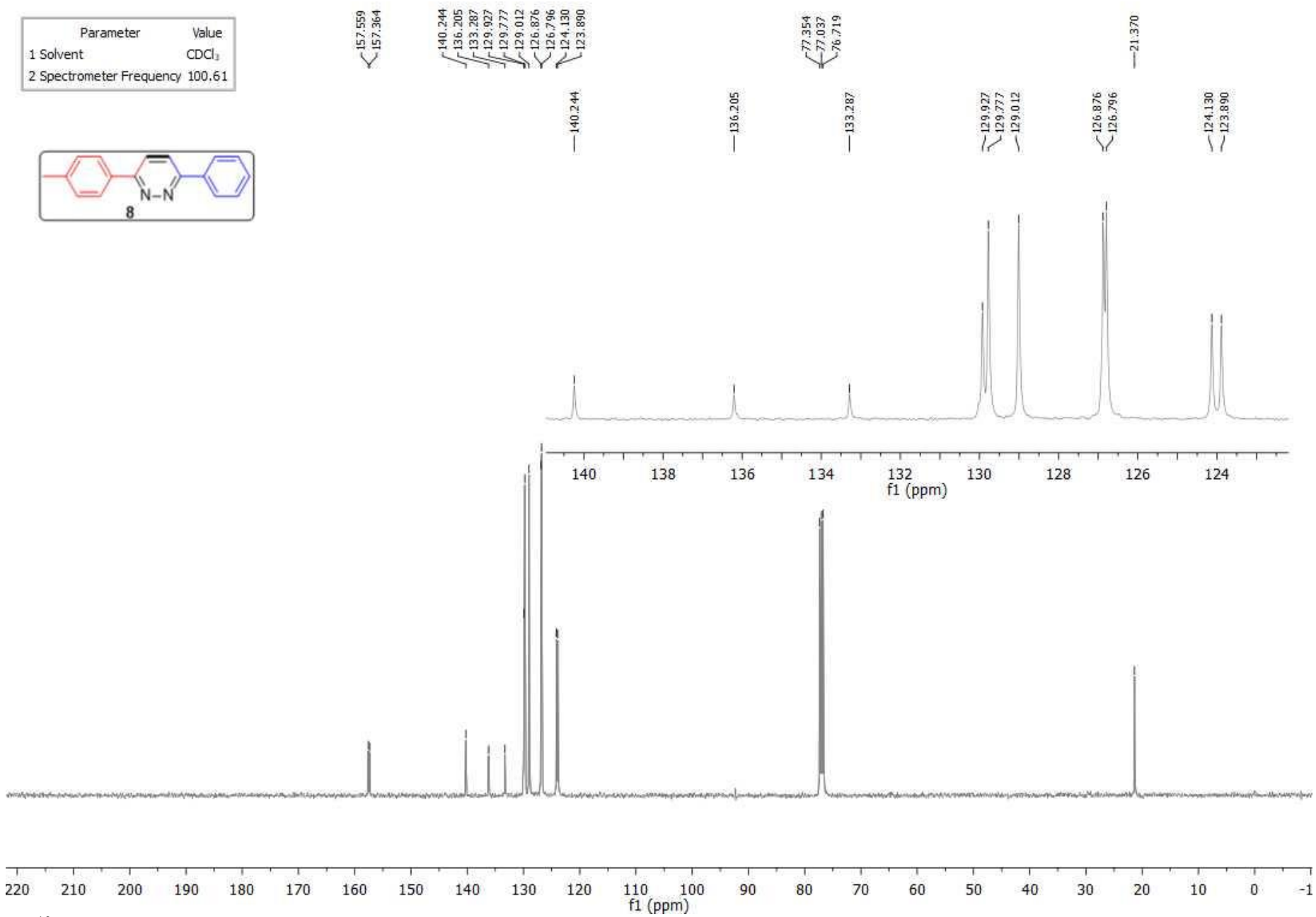


Figure S103. ¹³C NMR spectrum of 3-Phenyl-6-(*p*-tolyl)pyridazine (**8**)

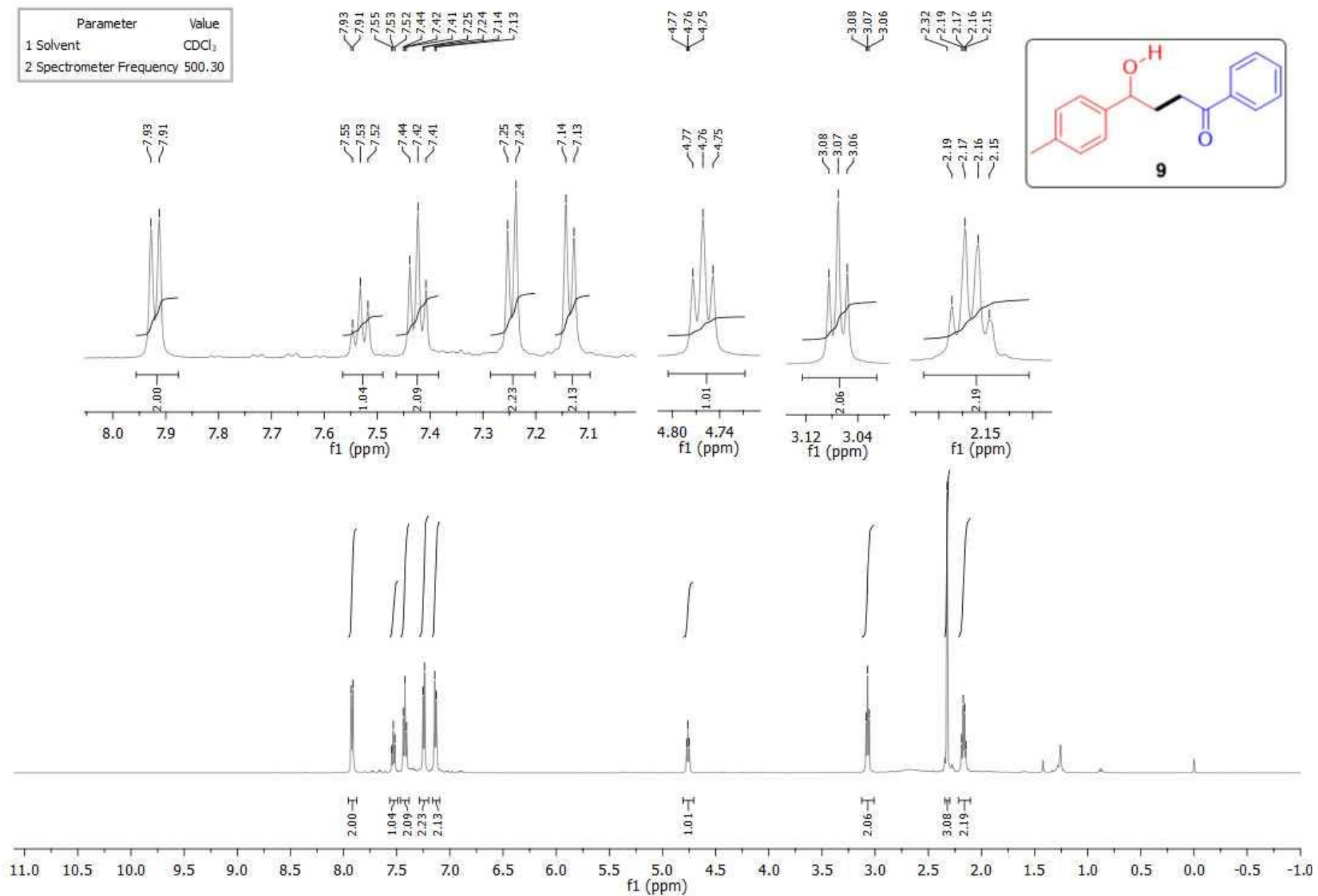


Figure S104. ¹H NMR spectrum of 4-Hydroxy-1-Phenyl-4-(*p*-tolyl)butane-1-one (**9**).

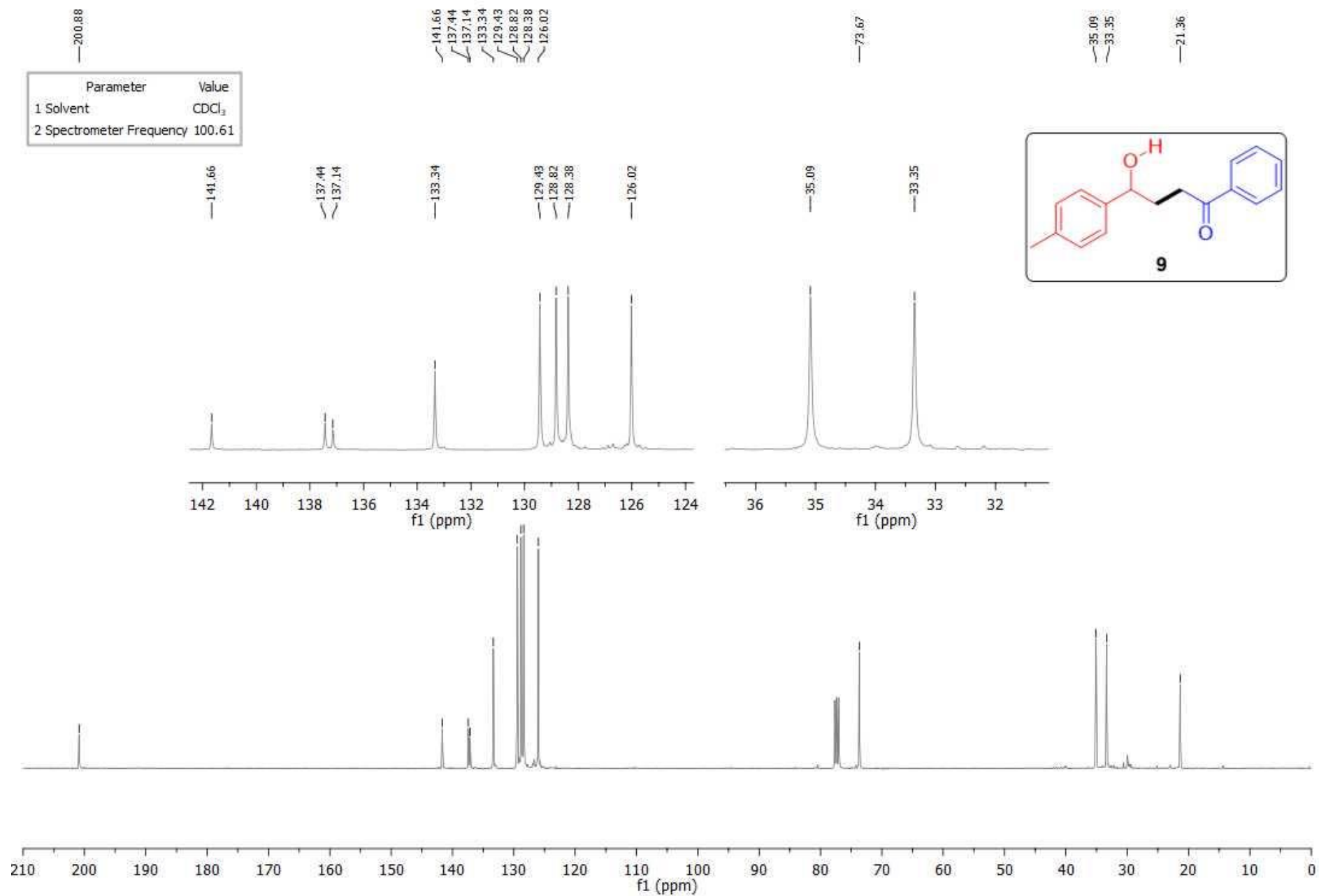


Figure S105. ¹³C NMR spectrum of 4-Hydroxy-1-Phenyl-4-(*p*-tolyl)butane-1-one (**9**).