Electronic Supplementary Material (ESI) for Chemical Communications.

Catalyst-Free Diazo Cross-coupling to Access Useful 3(2*H*)-Furanone Derivatives

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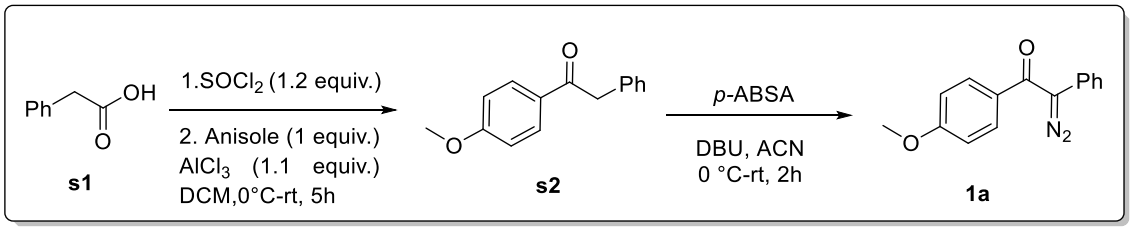
Content

1.1 General Procedure.

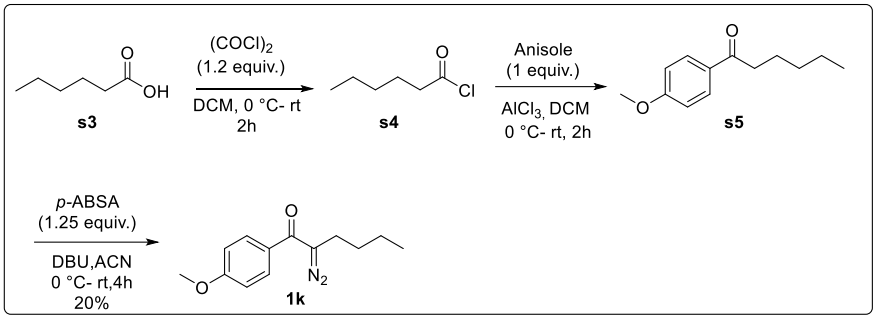
Unless otherwise noted, all the preparations of substrates were performed in oven-dried glassware under a nitrogen atmosphere with freshly distilled solvents. The catalytic reactions were performed under a nitrogen atmosphere. DCE, DCM and Ether were distilled from CaH_2 under nitrogen. THF was re-distilled from Na metal under nitrogen. All other commercial reagents were used without further purification unless otherwise indicated. ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker 500, 600 MHz and Varian 500,700 MHz spectrometers using chloroform-*d* ($CDCl_3$) as the internal standards. High-resolution mass spectral analysis (HRMS) data were measured on JMS-T100LP4G (JEOL) mass spectrometer or a TOF mass analyzer equipped with the ESI source and Magnetic Sector Mass Analyzer (MStation) equipped with the EI source. Brand: JEOL Model: JMS-T200GC AccuTOF GCx, Source mode: FD(field desorption). Single-crystal X-ray diffraction intensity data were collected on a Bruker X8 APEX diffractometer equipped with a CCD area detector and Mo Kα radiation (λ = 0.71073 Å) at 100 K; all data calculations were performed by using the PC version of the APEX2 program package. Final R indices were obtained using those reflections $I > 2\sigma(I)$.

1.2 1. General synthetic procedures for preparation of diazo ketone [s1]



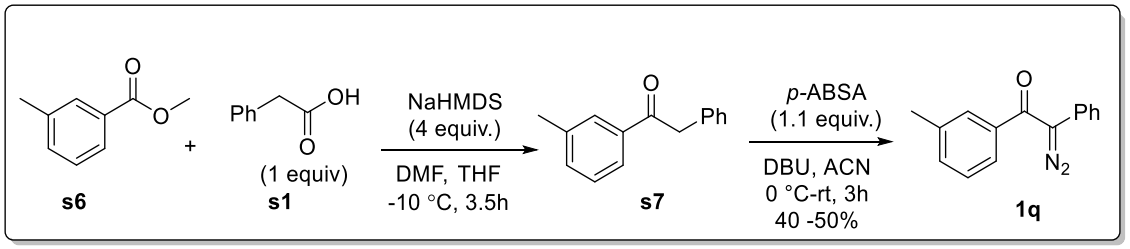
To a DCM (60 ml) solution of substituted phenylacetic acid (**s1**) (3.0 g, 22.03 mmol) was added $SOCl_2$ (1.91 ml, 26.44 mmol) dropwise at 0 °C. The mixture was warm to room temperature and stirred for 2 h. The resulting solution was cooled to 0 °C, followed by the addition of $AlCl_3$ (2.93 g, 22.03 mmol) and Anisole (2.39 ml, 22.03 mmol). The mixture was stirred at room temperature for 3h. After completion of the reaction. The reaction was quenched with H_2O, extracted with ethyl acetate (2 x 50 mL), and washed with brine (25 mL). The combined organic layers were dried over $MgSO_4$, concentrated under reduced pressure, and purified by a silica column (EA/Hexane = 15/85) to afford 1-(4-methoxyphenyl)-2-phenylethanone (**s2**) white solid (4.0 g, 17.67 mmol, 80%). To an acetonitrile (23 ml) solution of 1-(4-methoxyphenyl)-2-phenylethanone (1.0 g, 44.19 mmol) was added *p*-ABSA (1.27 g, 53.03 mmol). The solution was cooled to 0 °C and DBU (0.86 ml, 57.45 mmol) was added dropwise to the above mixture and stirred at room temperature for 2 h. The reaction was quenched with H_2O, followed by extraction with ether (2 x 50 mL), and washed with brine (25 mL). The combined organic layers were dried over $MgSO_4$, concentrated under reduced pressure, and purified by a silica column (EA/Hexane = 15/85) to afford 2-diazo-1-(4-methoxyphenyl)-2-phenylethan-1-one (**1a**) (0.836 g, 33.14 mmol, 76%) as yellow solid.

Substrates (**1b – 1j**) were synthesized according to the reported literature above procedure [s1-a]



To a DCM (30 ml) solution of substituted hexanoic acid (**s3**) (2.0 g, 17.2 mmol) was added $(COCl)_2$ (2.95 ml, 34.4 mmol) dropwise at 0 °C. The mixture was warm to room temperature and stirred for 2 h. The resulting solution was cooled to 0 °C, followed by the addition of $AlCl_3$ (2.52 g, 18.9 mmol) and Anisole (1.86 ml, 17.2 mmol). The mixture was stirred at room temperature for 2h. After completion of the reaction. The reaction was quenched with H_2O, extracted with DCM (2 x 50 mL), and washed with brine (25 mL). The combined organic layers were dried over $MgSO_4$, concentrated under reduced pressure, and purified by a silica column (EA/Hexane = 15/85) to afford 1-(4-methoxyphenyl)hexan-1-one (**s5**) white solid (3.50 g, 16.97 mmol, 98%). To an acetonitrile (20 ml) solution of 1-(4-methoxyphenyl)hexan-1-one (1.5 g, 7.27 mmol) was added *p*-ABSA (2.18 g, 9.09 mmol, 1.25 equiv.). The solution was cooled to 0 °C and DBU (1.47 ml, 8.72 mmol, 1.3 equiv.) was added dropwise to the above mixture and stirred at room temperature for 2 h. The reaction was quenched with H_2O, followed by extraction with ether (2 x 50 mL), and washed with brine (25 mL). The combined organic layers were dried over $MgSO_4$, concentrated under reduced pressure, and purified by a silica column (EA/Hexane = 15/85) to afford 2-diazo-1-(4-methoxyphenyl)hexan-1-one (**1k**) (400 mg, 1.72 mmol, 24%) as yellow oil.

Substrate **1l** synthesized according to above procedure [s1-b]



To a solution of aromatic acetic acid **s1** (0.9 gm, 6.66 mmol, 1.0 eq.) and aromatic methyl ester **s6** (1 gm, 6.66 mmol, 1.0 eq.) in DMF (3 mL) was added NaHMDS (2.0 M in THF) (5.43 mL, 4.0 eq.) at -10℃ over 1 min. The resulting mixture was stirred at -10℃ for 3.5 hours. To the resulting solution was then added saturated aqueous NH_4Cl solution. The resulting mixture was extracted with EtOAc (2x30 ml). The combined organic phase was washed with brine, dried over sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatography to afford desired ketone **s7**. The compound **s7** was dissolved in ACN at 0 °C

and *p*-ABSA (0.60 gm, 2.51 mmol, 1.2 equiv.) was added to above solution and DBU (0.38 mL, 1.21 equiv.) was added dropwise to the above mixture at 0 °C and stirred at room temperature for 3h. The reaction was quenched with H_2O, followed by extraction with ether (3 x 50 mL), and washed with brine (25 mL). The combined organic layers were dried over $MgSO_4$, concentrated under reduced pressure, and purified by a silica column (EA/Hexane = 15/85) to afford 2-diazo-2-phenyl-1-(*m*-tolyl) ethan-1-one (**1q**) (0.24 g, 1.02 mmol, 48%) as yellow solid.

Substrates (**1r –1s**) were synthesized according to the reported literature above procedure [s1-c]

Substrate **1s** having non-separable impurities and it used as without further purification.

Substrates (**1m-1p)** were synthesized according to reported literature. [s1-d]

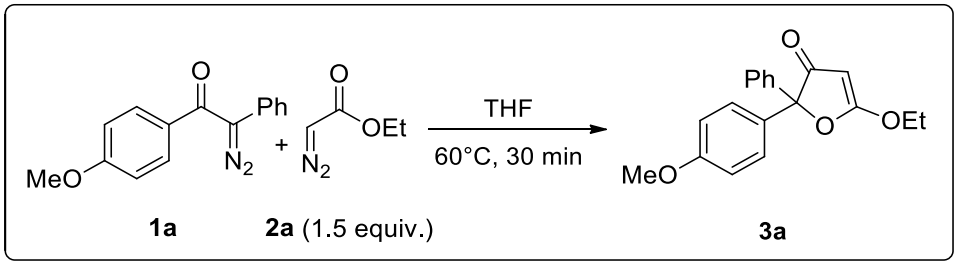
Substrates **1t**, and **1u** were synthesized according to reported literature. [s1-e]

1.2.3 Preparation of α-diazo esters.

2a is commercially available from Sigma-Aldrich.

All α-diazo esters (**2b-2i**) were prepared from the reported procedure in the literature. [**s2**]

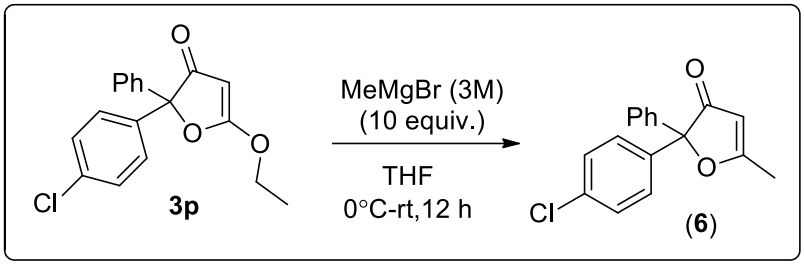
2. Standard reaction procedures



A Schlenk tube charged with freshly distilled out THF (2 mL) and heated up to the 65°C in oil bath, then to that pre-heated solution of THF, suspension of **1a** (50 mg, 0.198 mmol) and **2a** (34 mg, 0.297 mmol, 1.5 equiv.) in THF (1 mL) was added quickly at 60°C and resultant reaction mixture continue for 30 min at same temperature. Reaction monitor by using TLC and after completion of reaction solvent was removed under reduced pressure and eluted through a silica gel column with ethyl acetate/ hexane (25/75) to afford 5-ethoxy-2-(4-methoxyphenyl)-2-phenylfuran-3(*2H*)-one **(3a)** (50 mg, 0.161 mmol, 81%) as a colorless oil.

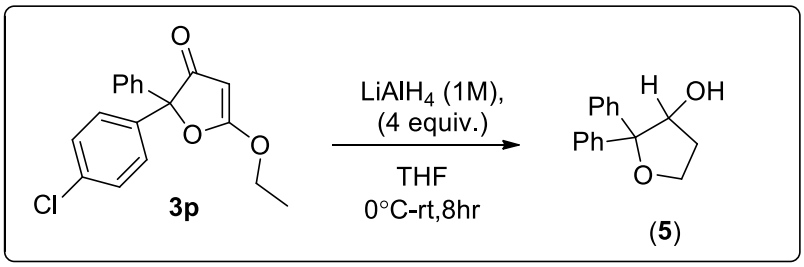
3. Chemical functionalization procedure and Large scale synthesis

3.1. Synthesis 2-(4-chlorophenyl)-5-methyl-2-phenylfuran-3(*2H*)-one (6)



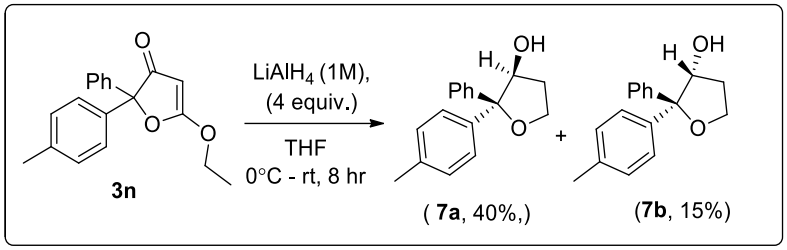
To a THF (3 mL) solution of 2-(4-chlorophenyl)-5-ethoxy-2-phenylfuran-3(*2H*)-one **(3p)** (30 mg, 0.095 mmol) was added MeMgBr (0.31 mL, 10 equiv.) at 0°C and the reaction is stirred at room temperature for 12 h. After completion of the reaction, reaction was quenched by saturated NH_4Cl solution (2 mL), followed by extraction with ether (3 x 5 mL), and washed with brine (5 mL). The combined organic layers were dried over $MgSO_4$, concentrated under reduced pressure, and purified by a silica column (EA/Hexane = 15/85) to afford 2-(4-chlorophenyl)-5-methyl-2-phenylfuran-3(*2H*)-one **(6)** (12.2 mg, 0.042 mmol, 45%) as a colorless oil.

3.2. Synthesis 2,2-diphenyltetrahydrofuran-3-ol (5)



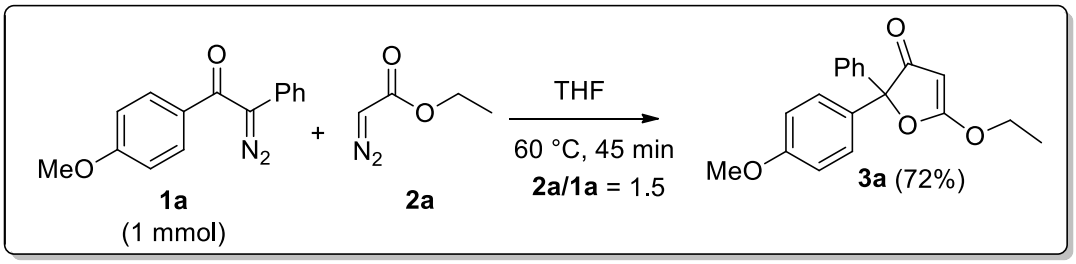
To a THF (3 mL) solution of 2-(4-chlorophenyl)-5-ethoxy-2-phenylfuran-3(*2H*)-one **(3p)** (30 mg, 0.095 mmol) was added LAH (1M, 0.38 mL, 4 equiv.) at 0°C and the reaction is stirred at room temperature for 8 h. After completion of the reaction, reaction was quenched by H_2O (2 mL), followed by extraction with ether (3 x 5 mL), and washed with brine (5 mL). The combined organic layers were dried over $MgSO_4$, concentrated under reduced pressure, and purified by a silica column (EA/Hexane = 15/85) to afford 2,2-diphenyltetrahydrofuran-3-ol **(5)** (11.2 mg, 0.046 mol, 49%) as a colorless oil.

3.3. Synthesis (2*R*,3*S*)-2-phenyl-2-(*p*-tolyl)tetrahydrofuran-3-ol (7a) and (2*R*,3*R*)-2-phenyl-2-(*p*-tolyl)tetrahydrofuran-3-ol (7b)



To a THF (3 mL) solution of (*R*)-5-ethoxy-2-phenyl-2-(p-tolyl)furan-3(*2H*)-one **(3n)** (60 mg, 0.20 mmol) was added LAH (1M, 0.85 mL, 4 equiv.) at 0°C and the reaction is stirred at room temperature for 8 h. After completion of the reaction, reaction was quenched by H_2O (2 mL), followed by extraction with ether (3 x 5 mL), and washed with brine (5 mL). The combined organic layers were dried over $MgSO_4$, concentrated under reduced pressure, and purified by a silica column (EA/Hexane = 20/80) to afford (2*R*,3*S*)-2-phenyl-2-(*p*-tolyl)tetrahydrofuran-3-ol **(7a)** (21 mg, 0.077 mol, 40%) as white solid and (2*R*,3*R*)-2-phenyl-2-(*p*-tolyl)tetrahydrofuran-3-ol **(7b)** (8 mg, 0.031 mmol, 15%) as colorless oil.

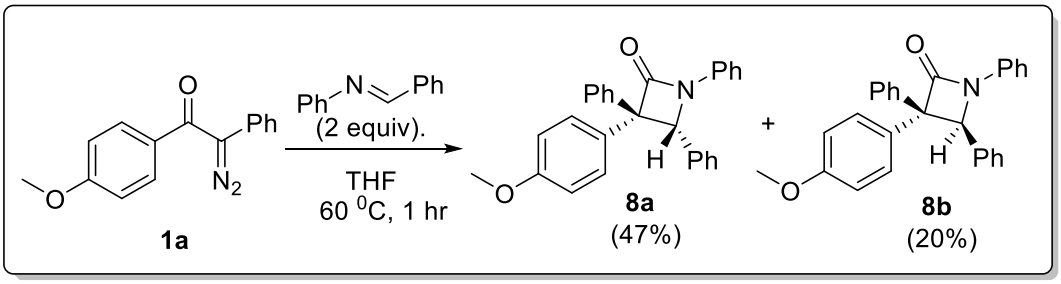
3.4. Large scale synthesis.



A Schlenk tube charged with freshly distilled out THF (8 mL) and heated up to the 65°C in oil bath, then to that pre-heated solution of THF, suspension of **1a** (252.3 mg, 1.00 mmol) and **2a** (171.1 mg, 1.50 mmol, 1.5 equiv.) in THF (3 mL) was added quickly at 60°C and resultant reaction mixture continue for 45 min at same temperature. Reaction monitor by using TLC and after completion of reaction solvent was removed under reduced pressure and eluted through a silica gel column with ethyl acetate/ hexane (25/75) to afford 5-ethoxy-2-(4-methoxyphenyl)-2-phenylfuran-3(*2H*)-one **(3a)** (224 mg, 0.72 mmol, 72%) as a colorless oil.

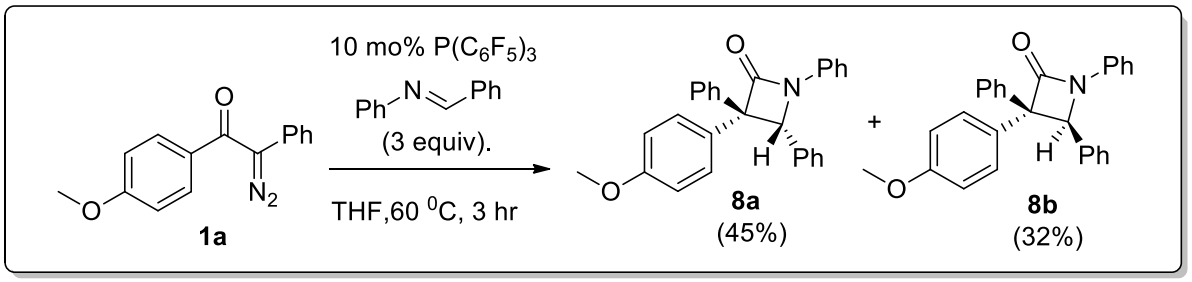
4. Verification of ketene intermediates

1. Synthesis of (3*S*,4*R*)-3-(4-methoxyphenyl)-1,3,4-triphenylazetidin-2-one (8a) and (3*S*,4*S*)-3-(4-methoxyphenyl)-1,3,4-triphenylazetidin-2-one (8b)



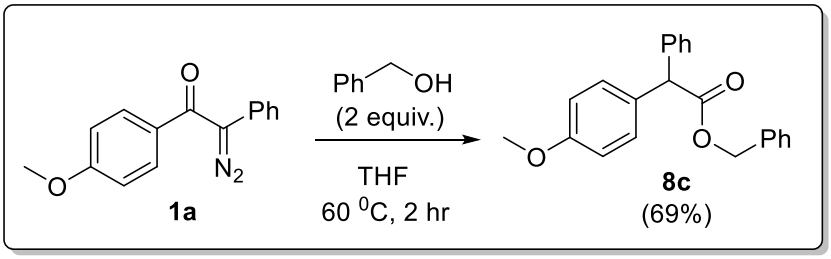
A Schlenk tube charged with freshly distilled out THF (2 mL) and heated up to the 60 °C in oil bath, then to that pre-heated solution of THF, suspension of **1a** (50 mg, 0.198 mmol) and (*E*)-N,1-diphenylmethanimine (71.8 mg, 0.39 mmol, 2 equiv.) in THF (1mL) was added quickly at 60°C and resultant reaction mixture continue for 1 hr at same temperature. Reaction monitor by using TLC and after completion of reaction solvent was removed under reduced pressure and eluted through a silica gel column with ethyl acetate/ hexane (20/80) to afford (3*S*,4*R*)-3-(4-methoxyphenyl)-1,3,4-triphenylazetidin-2-one **(8a)** (37.8 mg, 0.093 mmol, 47%) as white solid and (3*S*,4*S*)-3-(4-methoxyphenyl)-1,3,4-triphenylazetidin-2-one **(8b)** (16.1 mg, 0.039 mmol, 20%) as white solid.

2. Synthesis of (3*S*,4*R*)-3-(4-methoxyphenyl)-1,3,4-triphenylazetidin-2-one (8a) and (3*S*,4*S*)-3-(4-methoxyphenyl)-1,3,4-triphenylazetidin-2-one (8b)



A Schlenk tube charged with 10 mol% $P(C_6F_5)_3$ and freshly distilled out THF (1 mL) was added and stirred at rt heated up to the 65 °C in oil bath, then to that pre-heated solution of THF, suspension of **1a** (50 mg, 0.198 mmol) and (*E*)-N,1-diphenylmethanimine (107 mg, 0.59 mmol, 2 equiv.) in THF (2mL) was added quickly at 60 °C and resultant reaction mixture continue for 3 hr at same temperature. Reaction monitor by using TLC and after completion of reaction solvent was removed under reduced pressure and eluted through a silica gel column with ethyl acetate/ hexane (20/80) to afford (3*S*,4*R*)-3-(4-methoxyphenyl)-1,3,4-triphenylazetidin-2-one **(8a)** (36.2 mg, 0.089 mmol, 47%) as white solid and (3*S*,4*S*)-3-(4-methoxyphenyl)-1,3,4-triphenylazetidin-2-one **(8b)** (25.7 mg, 0.063 mmol, 33%) as white solid.

3. Synthesis of benzyl 2-(4-methoxyphenyl)-2-phenylacetate (8c)



A Schlenk tube charged with freshly distilled out THF (2 mL) and heated up to the 65°C in oil bath, then to that pre-heated solution of THF, suspension of **1a** (50 mg, 0.198 mmol) and Benzyl alcohol (42 mg, 0.39 mmol, 2 equiv.) in THF (1mL) was added quickly at 60°C and resultant reaction mixture continue for 2hr at same temperature. Reaction monitor by using TLC and after completion of reaction solvent was removed under reduced pressure and eluted through a silica gel column with ethyl acetate/ hexane (10/90) to afford 2-(4-methoxyphenyl)-2-phenylacetate **(8c)** (45.4 mg, 0.0136 mmol, 69%) as a colorless oil.

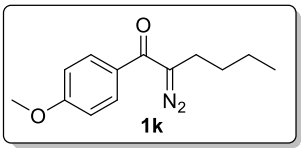
4. References

[s1] (a) S. A. More, V. A. Sadaphal, T-C Kuo, M-J Cheng, and R.-S. Liu, *Chem. Commun*., 2022, **58**,10064–10067; (b) X. L. Yan, N. Zhang, Z. Q. Hao, Z. H. Ma, Z. G. Han, X. Z. Zheng, and J. Lin. *Polyhedron* **147** (2018) 75–79; (c) 1) G. Wu, W. Yin. H. C. Shen, and Y. Huang, *Green Chem.*, 2012, **14**, 580-585; 2) S. Ghosh, I. Banerjee, and S. Baul, *Tetrahedron* **55** (1999) 11537-11546; (d) 1) C. N. Chen, W. M. Cheng, J. K. Wang, T. H. Chao, M. J. Cheng, and R.-S. Liu, *Angew. Chem. Int. Ed*. **2021**, 60, 4479-4484; *Angew. Chem*. 2021, **133**, 4529-4534; 2) J. Vaitala, A. Bayer, and K. H. Hopman, *Angew. Chem. Int. Ed*. 2017, **56**, 4277-4281; (e) Q. Lu, S. Mondal, S. Cembellín, and F. Glorius, *Angew. Chem. Int. Ed*. 2018, **57**, 10732-10736.

[s2] (1) D. M. Carminati, D. Intrieri, A. Caselli, S. Le Gac, B. Boitrel, L. Toma, L. Legnani, and E. Gallo, *Chem. Eur. J*. 2016, **22**, 13599-13612; (2) H. S. A. Mandour, Y. Nakagawa, M. Tone, H.Inoue, N. Otog, I. Fujisawa, S. Chanthamath, and S. I. Beilstein, *J. Org. Chem*. 2019, **15**, 357–363; (3) J.-H. Chu, X-H. Xu, S-M. Kang, N. Liu, and Z-Q. Wu, *J. Am. Chem. Soc*. 2018, **140**, 17773−17781; (4) T. Xiao, P. Peng, Y. Xie, Z.-Y. Wang, and L. Zhou, *Org. Lett*. 2015, **17**, 4332−4335; (5) R. D. Kardile, and R.-S. Liu, *Org. Lett*. 2019, **21**, 6452−6456; (6) A. S. Narode, and R-S. Liu, *Org. Lett*. 2022, **24**, 2165−2169; (7) T. Torna, J. Shimokawa, and T. Fukuyama, *Org. Lett*. 2007, **9**, 3195–3197; (8) H. Mao, A. Lin, Y. Shi, Z. Mao, X. Zhu, W. Li, H. Hu, Y. Cheng, and C. Zhu, *Angew. Chem. Int. Ed*. 2013, **52**, 6288–6292; (9) D. M. Hodgson, and D. Angrish, *Chem. Eur. J*. 2007, **13**, 3470-3479. (10) S. P. Bew, P.-A. Ashford, and D. U. Bachera, *Synthesis* 2013, **45**, 903–912

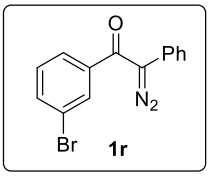
5. Spectral data of key compounds

Spectral data for 2-diazo-1-(4-methoxyphenyl)hexan-1-one (1k)



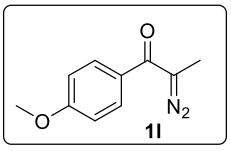
Yellow oil, (400 mg, 1.72 mmol, 24%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.55 (d, *J* = 10 Hz, 2H), 7.89 (d, *J* = 9.1 Hz, 2H), 3.82 (s, 3H), 2.49 (t, *J* = 10 Hz, 2H), 1.54 ~ 1.51 (m, 2H), 1.43 ~ 1.38 (m, 2H), 0.93 (t, *J* = 5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 188.6, 161.9, 130.4, 129.2, 113.6, 55.3, 29.1, 23.3, 21.9, 13.7 , ($C=N_2$) Peak not showing because of low intensity; HRMS- ESI+ calcd. for $C_{13}H_{16}O_2N_2$ (M+Na): 255.1109; found: 255.1107.

Spectral data for 1-(3-bromophenyl)-2-diazo-2-phenylethan-1-one (1r)



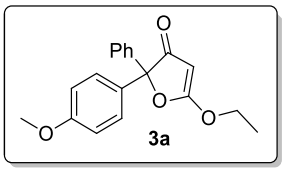
Brown solide, (350 mg, 1.16 mmol, 64%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.74 (t, *J* = 2.0 Hz, 2H), 7.61 ~ 7.59 (m,1H), 7.49 ~ 7.46 (m, 1H), 7.43 ~ 7.37 (m, 4H), 7.28 ~ 7.23 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 186.5, 139.6, 134.5, 130.8, 129.9, 129.1, 127.3, 126.2, 126.2, 125.6, 122.7, 73.3; HRMS-FD+ calcd. for $C_{14}H_9ON_2Br$: 299.9903; found: 299.9896.

Spectral data for 2-diazo-1-(4-methoxyphenyl)propan-1-one (1l)



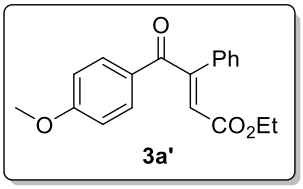
Yellow solide, (220 mg, 1.16 mmol, 38%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (d, *J* = 9.2 Hz, 2H), 6.89 (d, *J* = 9.2 Hz, 2H), 3.81 (s, 3H), 2.11 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.0, 162.0, 130.2, 129.2, 113.6, 62.0, 55.3, 9.7; HRMS-FD+ calcd. for $C_{10}H_{10}O_2N_2$: 190.0747; found: 190.0742.

Spectral data for 5-ethoxy-2-(4-methoxyphenyl)-2-phenylfuran-3(*2H*)-one (3a)



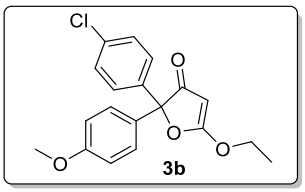
colorless oil, (50 mg, 0.161 mmol, 81%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.45 (d, *J* = 7.0 Hz, 2H), 7.36 (d, *J* = 9.1 Hz, 2H), 7.33 ~ 7.29 (m, 3H), 6.84 (d, *J* = 8.4 Hz, 2H), 4.80 (s, 1H), 4.29(q, *J* = 7.0 Hz, 2H), 3.77 (s, 3H), 1.46 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.9, 183.3, 159.7, 138.0, 130.1, 128.3, 128.3, 126.4, 113.7, 94.2, 79.0, 55.2, 14.2; HRMS- ESI+ calcd. for $C_{19}H_{18}O_4$ (M+Na): 333.1103; found: 333.1166.

Spectral data for ethyl (*E*)-4-(4-methoxyphenyl)-4-oxo-3-phenylbut-2-enoate (3a')



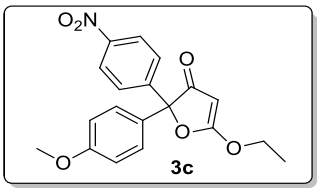
colorless oil, (24.6 mg, 0.079 mmol, 40%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.0 Hz, 2H), 7.38 (d, *J* = 4.5 Hz, 2H), 7.32 (d, *J* = 5.0 Hz, 3H), 6.89 (d, *J* = 8.5 Hz, 2H), 6.16 (s, 1H), 4.08(q, *J* = 8.0 Hz, 2H), 3.83 (s, 3H), 1.11 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 194.1, 165.4, 164.0, 152.7, 134.4, 132.5, 128.8, 128.3, 128.0, 122.3, 113.9, 113.9, 60.7, 55.5, 13.8; HRMS-ESI+ calcd. for $C_{19}H_{18}O_4$ (M+Na): 333.1102; found: 333.1103

Spectral data for 2-(4-chlorophenyl)-5-ethoxy-2-(4-methoxyphenyl)furan-3(*2H*)-one (3b)



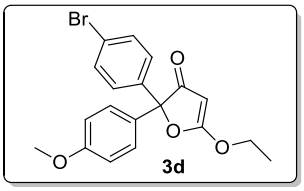
colorless oil, (45.7 mg, 0.132 mmol, 76%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.40 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 6.84 (d, *J* = 8.4 Hz, 2H), 4.80 (s, 1H), 4.29 (q, *J* = 7.0 Hz, 2H), 3.77 (s, 3H), 1.46 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.5, 183.4, 159.8, 136.5, 134.4, 129.8, 128.5, 128.2, 127.8, 113.8, 93.5, 78.9, 68.1, 55.3, 14.2; HRMS-ESI+ calcd. for $C_{19}H_{17}ClO_4$ (M+Na): 367.0713; found: 367.0727

Spectral data for 5-ethoxy-2-(4-methoxyphenyl)-2-(4-nitrophenyl)furan-3(*2H*)-one (3c)



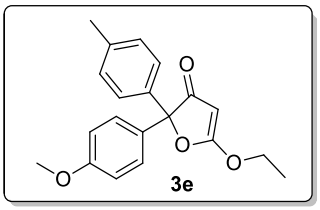
colorless oil, (49.6 mg, 0.139 mmol, 83%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.16 (d, *J* = 9.1 Hz, 2H), 7.68 (d, *J* = 9.1 Hz, 2H), 7.33 (d, *J* = 9.1 Hz, 2H), 6.85 (d, *J* = 9.1 Hz, 2H), 4.82 (s, 1H), 4.32 (q, *J* = 7.0 Hz, 2H), 3.77 (s, 3H), 1.48 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 196.3, 183.4, 160.1, 147.7, 144.8, 129.3, 128.3, 127.0, 123.4, 114.0, 93.0, 78.9, 68.5, 55.3, 14.2; HRMS-ESI+ calcd. for $C_{19}H_{17}O_6$ (M+Na): 355.1056; found: 355.1057.

Spectral data for 2-(4-bromophenyl)-5-ethoxy-2-(4-methoxyphenyl)furan-3(*2H*)-one (3d)



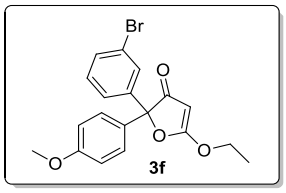
colorless oil, (47 mg, 0.120 mmol, 80%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.44 (d, *J* = 8.4 Hz, 2H), 7.35 ~ 7.32 (m, 2H), 6.84 (d, *J* = 9.1 Hz, 2H), 4.79 (s, 1H), 4.29 (q, *J* = 7.0 Hz, 2H), 3.76 (s, 3H), 1.45 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.4, 183.3, 159.8, 137.0, 131.4, 129.7, 128.2, 128.0, 122.6, 113.8, 93.5, 78.9, 68.1, 55.2, 14.2; HRMS-ESI+ calcd. for $C_{19}H_{17}O_4Br$: 388.0310; found: 388.0309

Spectral data for 5-ethoxy-2-(4-methoxyphenyl)-2-(*p*-tolyl)furan-3(*2H*)-one (3e)



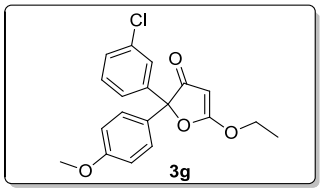
colorless oil, (51.8 mg, 0.159 mmol, 85%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.35 (d, *J* = 9.1 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 8.4 Hz, 2H), 6.84 (d, *J* = 8.4 Hz, 2H), 4.79 (s, 1H), 4.29 (q, *J* = 7.0 Hz, 2H), 3.76 (s, 3H), 2.31 (s, 3H), 1.45 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 198.1 183.3, 159.6, 138.2, 135.1, 130.2, 129.0, 128.2, 126.4, 113.7, 93.3, 78.9, 67.8, 55.2, 21.0, 14.2; HRMS-ESI+ calcd. for $C_{20}H_{20}O_4$: 324.1362; found: 324.1367

Spectral data for 2-(3-bromophenyl)-5-ethoxy-2-(4-methoxyphenyl)furan-3(*2H*)-one (3f)



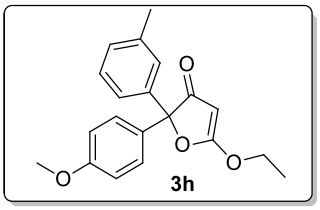
colorless oil, (45.8 mg, 0.117 mmol, 78%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.62 (s, 1H), 7.62 ~ 7.40 (m, 2H), 7.33 (d, *J* = 9.1 Hz, 2H), 7.19 (t, *J* = 7.7 Hz, 1H), 6.85 (d, *J* = 9.1 Hz, 2H), 4.80 (s, 1H), 4.32 ~ 4.29 (m, 2H), 3.77 (s, 3H), 1.47 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.2 183.3, 159.8, 140.2, 131.4, 129.8, 129.6, 129.1, 128.2, 125.2, 122.4, 113.9, 93.1, 78.9, 68.2, 55.3, 14.2; HRMS-ESI+ calcd. for $C_{19}H_{17}BrO_4$: 388.0310; found: 388.0313

Spectral data for 2-(3-chlorophenyl)-5-ethoxy-2-(4-methoxyphenyl)furan-3(*2H*)-one (3g)



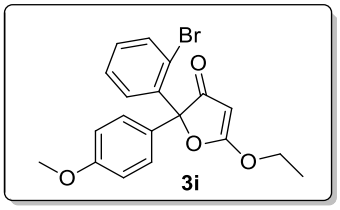
colorless oil, (45.1 mg, 0.130 mmol, 75%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.47 (s, 1H), 7.46 ~ 7.34 (m, 3H), 7.28 ~ 7.24 (m, 2H), 6.85 (d, *J* = 9.1 Hz, 2H), 4.80 (s, 1H), 4.32 ~ 4.29 (m, 2H), 3.77 (s, 3H), 1.47 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.2 183.3, 159.8, 139.9, 134.3, 129.6, 129.5, 128.5, 128.2, 126.3, 124.7, 113.9, 93.2, 78.9, 68.2, 55.3, 14.2; HRMS-ESI+ calcd. for $C_{19}H_{17}ClO_4$: 344.0815; found: 344.0813

Spectral data for 5-ethoxy-2-(4-methoxyphenyl)-2-(*m*-tolyl)furan-3(*2H*)-one (3h)



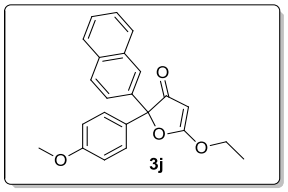
colorless oil, (48.7 mg, 0.150 mmol, 80%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.35 (d, *J* = 9.1 Hz, 2H), 7.25 (d, *J* = 9.1 Hz, 2H), 7.20 (t, *J* = 7.7 Hz, 1H), 7.10 (d, *J* = 7.0 Hz, 1H), 6.83 (d, *J* = 9.1 Hz, 2H), 4.79 (s, 1H), 4.28 (q, *J* = 7.0 Hz, 2H), 3.76 (s, 3H), 2.30 (s, 3H), 1.45 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 198.0 183.3, 159.6, 138.0, 137.9, 130.2, 129.1, 128.2, 128.1, 127.0, 123.5, 113.7, 94.3, 79.0, 67.9, 55.2, 21.4, 14.2; HRMS-ESI+ calcd. for $C_{20}H_{20}O_4$: 324.1362; found: 324.1364.

Spectral data for 2-(2-bromophenyl)-5-ethoxy-2-(4-methoxyphenyl)furan-3(*2H*)-one (3i)



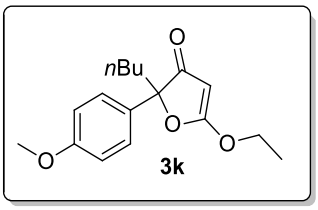
colorless oil, (50.2 mg, 0.128 mmol, 65%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.65 (d, *J* = 7.7 Hz, 1H), 7.60 (d, *J* = 8.4 Hz, 1H), 7.28 (t, *J* = 7.7 Hz, 1H), 7.21 ~ 7.16 (m, 3H), 6.84 (d, *J* = 9.1 Hz, 2H), 4.83 (s, 1H), 4.35 (q, *J* = 7.0 Hz, 2H), 3.76 (s, 3H), 1.47 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.1 183.5, 159.4, 135.5, 135.4, 130.9, 130.4, 128.9, 126.9, 126.8, 124.3, 113.7, 94.8, 79.3, 68.0, 55.2, 14.3; HRMS-ESI+ calcd. for $C_{19}H_{17}BrO_4$: 388.0310; found: 388.0308.

Spectral data for 5-ethoxy-2-(4-methoxyphenyl)-2-(naphthalen-2-yl)furan-3(*2H*)-one (3j)



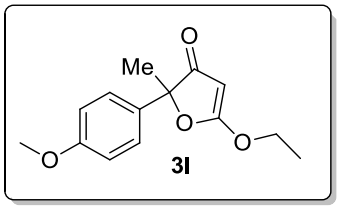
colorless oil, (37 mg, 0.102 mmol, 62%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.99 (s, 1H), 7.82 ~ 7.78 (m, 3H), 7.51 (dd, *J* = 8.4, 1.4 Hz, 1H), 7.47 ~ 7.45 (m, 2H), 7.38 (d, *J* = 9.1 Hz, 2H), 6.85 (d, *J* = 8.4 Hz, 2H), 4.85 (s, 1H), 4.32 (q, *J* = 7.0 Hz, 2H), 3.77 (s, 3H), 1.48 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.8 183.4, 159.7, 135.2, 133.0, 132.7, 130.0, 128.4, 128.3, 128.2, 127.5, 126.5, 126.2, 125.4, 124.4, 113.8, 94.4, 79.1, 68.0, 55.2, 14.2; HRMS-ESI+ calcd. for $C_{23}H_{20}O_4$: 360.1362; found: 360.1364.

Spectral data for 2-butyl-5-ethoxy-2-(4-methoxyphenyl)furan-3(*2H*)-one (3k)



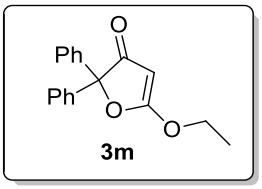
colorless oil, (28.1 mg, 0.089 mmol, 45%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.44 (d, *J* = 9.1 Hz, 2H), 6.85 (d, *J* = 9.1 Hz, 2H), 4.68 (s, 1H), 4.27 (q, *J* = 5.6 Hz, 2H), 3.77 (s, 3H), 2.03 (q, *J* = 11.2 Hz, 2H), 1.46 (t, *J* = 7.0 Hz, 3H), 1.30 ~ 1.23 (m, 4H), 0.84 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 199.8 183.3, 159.3, 129.6, 125.9, 113.7, 94.4, 79.0, 67.7, 55.2, 37.8, 25.3, 22.6, 14.2, 13.8; HRMS-ESI+ calcd. for $C_{17}H_{22}O_4$: 290.1518; found: 290.1516.

Spectral data for 5-ethoxy-2-(4-methoxyphenyl)-2-methylfuran-3(*2H*)-one (3l)



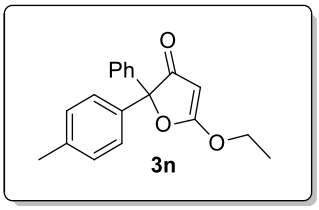
colorless oil, (40.5 mg, 0.163 mmol, 62%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.41 (d, *J* = 8.4 Hz, 2H), 6.86 (d, *J* = 9.1 Hz, 2H), 4.70 (s, 1H), 4.29 (q, *J* = 7.0 Hz, 2H), 3.77 (s, 3H), 1.75 (s, 3H), 1.45 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 200.2, 183.1, 159.5, 129.9, 126.1, 113.8, 91.8, 77.9, 67.7, 55.2, 24.1, 14.2; HRMS-ESI+ calcd for $C_{14}H_{16}O_4Na$ $[M+Na]^+$: 271.0946, found: 271.0948.

Spectral data for 5-ethoxy-2,2-diphenylfuran-3(*2H*)-one (3m)



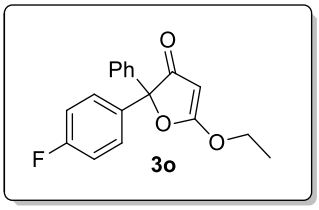
colorless oil, (47.3 mg, 0.168 mmol, 75%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.47 (t, *J* = 7.7 Hz, 4H), 7.34 ~ 7.29 (m, 6H), 4.81 (s, 1H), 4.30 (q, *J* = 7.0 Hz, 2H), 1.46 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.6, 183.4, 137.9, 128.4, 128.3, 126.5, 94.1, 79.0, 68.0, 14.2; HRMS-ESI+ calcd. for $C_{18}H_{16}O_3$ (M+Na): 280.1099; found: 280.1103.

Spectral data for 5-ethoxy-2-phenyl-2-(p-tolyl)furan-3(*2H*)-one (3n)



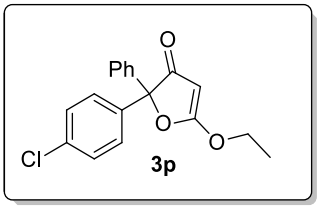
colorless oil, (51.7 mg, 0.175 mmol, 83%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.47 (t, *J* = 8.4 Hz, 2H), 7.35 ~ 7.33 (m, 2H), 7.32 ~ 7.29 (m, 3H), 7.13 (d, *J* = 7.7 Hz, 2H), 4.80 (s, 1H), 4.29 (q, *J* = 7.0 Hz, 2H), 2.31 (s, 3H), 1.46 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.8 183.4, 138.3, 137.9, 135.0, 129.0, 128.3, 126.6, 126.5, 94.2, 78.9, 67.9, 21.0, 14.2; HRMS-ESI+ calcd. for $C_{19}H_{18}O_3$: 294.1256; found: 294.1252

Spectral data for 5-ethoxy-2-(4-fluorophenyl)-2-phenylfuran-3(*2H*)-one (3o)



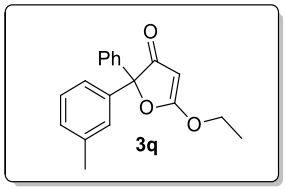
colorless oil, (50.9 mg, 0.170 mmol, 82%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.48 ~ 7.44 (m, 4H), 7.34 ~ 7.30 (m, 3H), 7.01 (t, *J* = 8.4 Hz, 2H), 4.81 (s, 1H), 4.30 (q, *J* = 7.0 Hz, 2H), 1.47 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.4 183.4, 163.4, 162.0, 137.7, 133.8, 128.6, 128.5, 128.4, 126.4, 115.3, 115.2, 93.5, 78.9, 68.1, 14.2; ^{19}F NMR (500 MHz, $CDCl_3$): -112.093; HRMS-EI+ calcd. for $C_{18}H_{15}FO_3$: 298.1005; found: 298.1003.

Spectral data for 2-(4-chlorophenyl)-5-ethoxy-2-phenylfuran-3(*2H*)-one (3p)



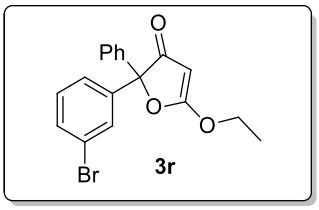
colorless oil, (53.4 mg, 0.169 mmol, 87%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45 ~ 7.42 (m, 4H), 7.34 ~ 7.29 (m, 5H), 4.80 (s, 1H), 4.30 (q, *J* = 5.5 Hz, 2H), 1.47 (t, *J* = 5.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 197.1, 183.4, 137.5, 136.4, 134.5, 128.6, 128.5, 128.4, 127.9, 126.5, 93.3, 78.9, 68.2, 14.2; HRMS-ESI+ calcd. for $C_{18}H_{15}ClO_3$: 314.0709; found: 314.0714.

Spectral data for 5-ethoxy-2-phenyl-2-(m-tolyl)furan-3(*2H*)-one (3q)



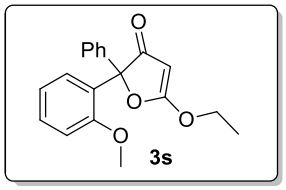
colorless oil, (49.2 mg, 0.167 mmol, 79%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.46 (d, *J* = 7.0 Hz, 2H), 7.33 ~ 7.30 (m, 3H), 7.27 (d, *J* = 14.0 Hz, 2H), 7.22 (t, *J* = 7.0 Hz, 1H), 7.12 (d, *J* = 7.7 Hz, 1H), 4.80 (s, 1H), 4.30 (q, *J* = 7.7 Hz, 2H), 2.30 (s, 3H), 1.47 (t, *J* = 7.7 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.7 183.4, 138.0, 137.9, 137.8, 129.2, 128.4, 128.3, 128.2, 127.1, 126.5, 123.7, 94.2, 79.0, 68.0, 21.5, 14.2; HRMS-ESI+ calcd. for $C_{19}H_{18}O_3$ (M+Na): 317.1153; found: 317.1145

Spectral data for 2-(3-bromophenyl)-5-ethoxy-2-phenylfuran-3(*2H*)-one (3r)



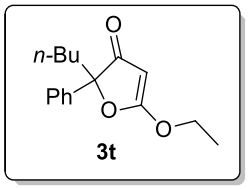
colorless oil, (44.7 mg, 0.124 mmol, 75%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.64 (d, *J* = 7.0 Hz, 1H), 7.45 ~ 7.43 (m, 4H), 7.34 ~ 7.31 (m, 3H), 7.19 (t, *J* = 7.7 Hz, 1H), 4.80 (s, 1H), 4.31 (q, *J* = 7.0 Hz, 2H), 1.47 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 196.8 183.4, 140.0, 137.4, 131.5, 129.9, 129.2, 128.7, 128.5, 126.5, 125.3, 122.5, 93.0, 78.9, 68.3, 14.2; ; HRMS-ESI+ calcd. for $C_{18}H_{15}O_3Br$ (M+Na): 381.0102; found: 381.0102.

Spectral data for 5-ethoxy-2-(2-methoxyphenyl)-2-phenylfuran-3(*2H*)-one (3s)



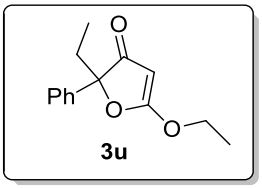
colorless oil, (46.1 mg, 0.148 mmol, 75%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.51 (d, *J* = 7.0 Hz, 2H), 7.35 ~ 7.29 (m, 4H), 7.02 (d, *J* = 9.1 Hz, 1H), 6.88 (d, *J* = 7.7 Hz, 1H), 6.84 (t, *J* = 7.7 Hz, 1H), 4.79 (s, 1H), 4.27 (q, *J* = 7.0 Hz, 2H), 3.64 (s, 3H), 1.45 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 198.8 183.1, 158.9, 137.4, 131.0, 130.8, 128.0, 127.9, 126.1, 125.7, 120.3, 112.3, 93.2, 78.2, 67.6, 55.7, 14.2; ; HRMS-ESI+ calcd. for $C_{19}H_{18}O_4$: 310.1205; found: 310.1207.

Spectral data for 2-butyl-5-ethoxy-2-phenylfuran-3(*2H*)-one (3t)



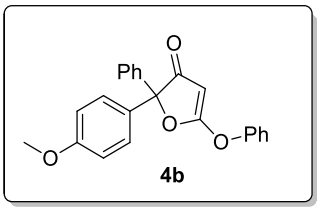
colorless oil, (9.7 mg, 0.037 mmol, 15%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.53 (t, *J* = 7.0 Hz, 2H), 7.33 (t, *J* = 7.0 Hz, 2H), 7.27 (d, *J* = 7.0 Hz, 1H), 4.68 (s, 1H), 4.31 ~ 4.26 (m, 2H), 2.11 ~ 2.03 (m, 2H), 1.47 (t, *J* = 7.0 Hz, 3H), 1.31 ~ 1.28 (m, 4H), 0.85 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 199.4, 183.4, 137.4, 128.3, 127.9, 124.5, 94.4, 78.7, 67.8 38.0, 25.3, 22.5, 14.2, 13.8; ; HRMS-ESI+ calcd. for $C_{16}H_{20}O_3$: 260.1412; found: 260.1411.

Spectral data for 5-ethoxy-2-ethyl-2-phenylfuran-3(*2H*)-one (3u)



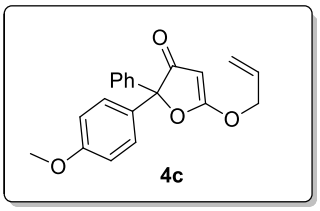
colorless oil, (6.7 mg, 0.028 mmol, 10%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.52 (d, *J* = 8.4 Hz, 2H), 7.33 (t, *J* = 7.0 Hz, 2H), 7.27 (t, *J* = 7.0 Hz, 1H), 4.70 (s, 1H), 4.30 ~ 4.27 (m, 2H), 2.12 (q, *J* = 7.7 Hz, 2H), 1.47 (t, *J* = 7.0 Hz, 3H), 0.87 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 199.3, 183.5, 137.2, 128.3, 127.9, 124.5, 94.7, 78.9, 67.8 31.3, 14.2, 7.73; ; HRMS-ESI+ calcd. for $C_{14}H_{16}O_3$: 232.1099; found: 232.1095.

Spectral data for 2-(4-methoxyphenyl)-5-phenoxy-2-phenylfuran-3(*2H*)-one (4b)



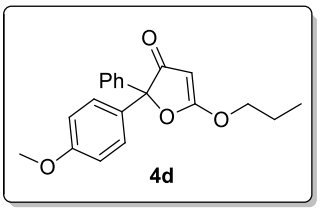
colorless oil, (63.2 mg, 0.176 mmol, 89%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.15 (t, *J* = 7.0 Hz, 2H), 7.42 ~ 7.40 (m, 4H), 7.33 ~ 7.31 (m, 4H), 7.21 (d, *J* = 8.4 Hz, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 4.63 (s, 1H), 3.78 (s, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.7, 183.6, 159.8, 152.3, 137.8, 130.1, 129.9, 128.4, 128.3, 128.2, 127.2, 126.3, 120.3, 113.8, 95.2, 80.9, 55.2; HRMS-ESI+ calcd. for $C_{23}H_{18}O_4$ (M+Na): 381.1102; found: 381.1103

Spectral data for 5-(allyloxy)-2-(4-methoxyphenyl)-2-phenylfuran-3(*2H*)-one (4c)



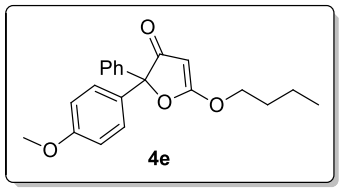
colorless oil, (44.7 mg, 0.138 mmol, 70%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45 (t, *J* = 7.0 Hz, 2H), 7.35 (d, *J* = 8.5 Hz, 2H), 7.40 ~ 7.30 (m, 3H), 6.84 (d, *J* = 9.0 Hz, 2H), 6.02 ~ 5.94 (m, 1H), 5.41 (q, *J* = 7.5 Hz, 2H), 4.82 (s, 1H), 4.73 (d, *J* = 5.5 Hz, 2H), 3.77 (s, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 198.0, 183.2, 159.7, 137.8, 130.0, 129.9, 128.6, 128.4, 128.1, 127.1, 126.4, 120.6, 113.7, 94.6, 79.5, 55.2; HRMS-ESI+ calcd. for $C_{20}H_{18}O_4$ (M+Na): 345.1102; found: 345.1104

Spectral data for 2-(4-methoxyphenyl)-2-phenyl-5-propoxyfuran-3(*2H*)-one (4d)



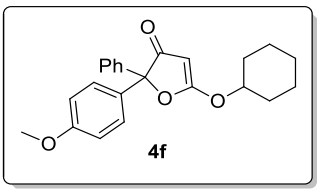
colorless oil, (47.6 mg, 0.146 mmol, 74%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.46 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 8.5 Hz, 2H), 7.34 ~ 7.29 (m, 3H), 6.85 (d, *J* = 9.0 Hz, 2H), 4.81 (s, 1H), 4.18 (t, *J* = 6.5 Hz, 2H), 3.77 (s, 3H), 1.87 ~ 1.80 (m, 2H), 1.02 (t, 7.5 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.9, 183.5, 159.6, 137.9, 130.1, 128.3, 128.3, 128.3, 126.4, 113.7, 94.2, 79.0, 73.4, 55.2, 22.0, 10.1; HRMS-ESI+ calcd. for $C_{20}H_{20}O_4$ (M+Na): 347.1259; found: 347.1260

Spectral data for 5-butoxy-2-(4-methoxyphenyl)-2-phenylfuran-3(*2H*)-one (4e)



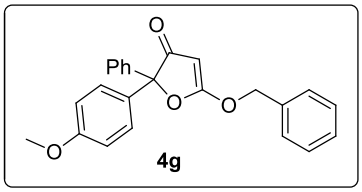
colorless oil, (49 mg, 0.144 mmol, 73%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.45 (d, *J* = 7.7 Hz, 2H), 7.36 (d, *J* = 8.4 Hz, 2H), 7.33 ~ 7.28 (m, 3H), 6.84 (d, *J* = 8.4 Hz, 2H), 4.80 (s, 1H), 4.23 (t, *J* = 7.0 Hz, 2H), 3.77 (s, 3H), 1.81 ~ 1.77 (m, 2H), 1.49 ~ 1.44 (m, 2H), 0.95 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.9, 183.5, 159.6, 138.0, 130.1, 128.3, 128.3, 128.2, 126.4, 113.7, 94.2, 79.0, 71.8, 55.2, 30.5, 18.7, 13.5; HRMS-ESI+ calcd. for $C_{21}H_{22}O_4$ (M+Na): 361.1415; found: 361.1415

Spectral data for 5-(cyclohexyloxy)-2-(4-methoxyphenyl)-2-phenylfuran-3(*2H*)-one (4f)



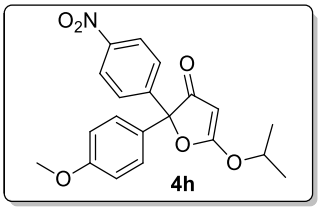
colorless oil, (58.5 mg, 0.160 mmol, 81%);); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.45 (d, *J* = 7.0 Hz, 2H), 7.36 (d, *J* = 9.0 Hz, 2H), 7.34 ~ 7.28 (m, 3H), 6.84 (d, *J* = 9.0 Hz, 2H), 4.78 (s, 1H), 4.51 ~ 4.46 (m, 1H), 3.76 (s, 3H), 1.99 ~ 1.97 (m, 2H), 1.80 ~ 1.87 (m, 2H), 1.68 ~ 1.61 (m, 2H), 1.56 ~ 1.53 (m, 1H), 1.41 ~ 1.30 (m, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.9, 182.7, 159.6, 138.1, 130.2, 128.3, 128.2, 126.4, 113.7, 93.8, 81.3, 79.3, 55.2, 31.3, 31.3, 24.9, 23.1; HRMS-ESI+ calcd. for $C_{23}H_{24}O_4$ (M+Na): 387.1572; found: 387.1578

Spectral data for 5-(benzyloxy)-2-(4-methoxyphenyl)-2-phenylfuran-3(*2H*)-one (4g)



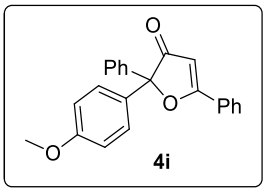
colorless oil, (45.1 mg, 0.121 mmol, 61%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.43 ~ 7.41 (m, 2H), 7.41 ~ 7.38 (m, 5H), 7.34 ~ 7.30 (m, 5H), 6.84 (d, *J* = 9.0 Hz, 2H), 4.78 (s, 1H), 5.26 (s, 2H), 5.90 (s, 1H), 3.77 (s, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 197.9, 183.1, 159.7, 137.8, 133.4, 130.0, 129.2, 128.9, 128.4, 128.3, 128.3, 128.1, 126.4, 113.7, 94.7, 79.9, 73.2, 55.3; HRMS-ESI+ calcd. for $C_{24}H_{20}O_4$ (M+Na): 395.1259; found: 395.1261

Spectral data for 5-isopropoxy-2-(4-methoxyphenyl)-2-(4-nitrophenyl)furan-3(*2H*)-one (4h)



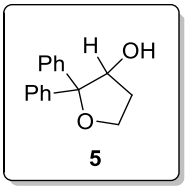
White Solid, (54.7 mg, 0.148 mmol, 88%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 8.17 (d, *J* = 8.4 Hz, 2H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.33 (d, *J* = 8.4 Hz, 2H), 6.85 (d, *J* = 8.4 Hz, 2H), 4.80 (s, 1H), 4.75 (m, 1H), 3.77 (s, 3H), 1.45 (d, *J* = 3.5 Hz, 3H), 1.44 (d, *J* = 3.5 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 196.3, 182.7, 160.0, 147.6, 145.0, 129.4, 128.3, 127.0, 123.4, 114.0, 92.6, 79.2, 76.8, 55.3, 21.8, 21.8; ; HRMS-ESI+ calcd. for $C_{20}H_{21}O_6$ (M+Na): 369.1212; found: 369.1218.

Spectral data for 2-(4-methoxyphenyl)-2,5-diphenylfuran-3(*2H*)-one (4i)



colorless oil, (30.3 mg, 0.088 mmol, 55%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 – 7.92 (m, 2H), 7.57 – 7.55 (m, 1H), 7.52 – 7.48 (m, 4H), 7.44 – 7.41 (m, 2H), 7.36 – 7.30 (m, 3H), 6.87 (dd, J_1 = 6.8 Hz, J_2 = 1.6 Hz, 1H), 6.07 (s, 1H), 3.77 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.4, 184.0, 159.6, 138.4, 132.9, 130.6, 128.9, 128.7, 128.3, 128.2, 127.2, 126.5, 113.8, 99.7, 93.0, 55.2; HRMS-ESI+ calcd for $C_{23}H_{18}O_3Na$ $[M+Na]^+$: 365.1153, found: 365.1151.

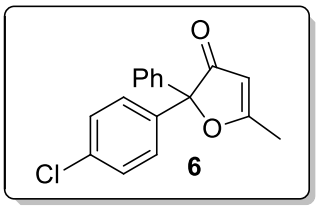
Spectral data for 2,2-diphenyltetrahydrofuran-3-ol (5)



5

White solid, (11.3 mg, 0.047 mmol, 49%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.50 (t, *J* = 8.4 Hz, 4H), 7.32 ~ 7.22 (m, 4H), 7.21 ~ 7.19 (m, 2H), 4.96 (s, 1H), 4.28 (q, *J* = 7.7 Hz, 1H), 3.97 (q, *J* = 7.0 Hz, 1H), 2.07 ~ 2.05 (m, 2H), 1.52 (bs, 1H) ; ^{13}C NMR (175 MHz, $CDCl_3$): δ 143.9, 141.4, 128.7, 128.5, 128.4, 128.3, 127.2, 127.1, 126.5, 125.5, 90.9, 76.3, 65.1, 33.3; HRMS-ESI+ calcd. for $C_{16}H_{16}O_2$ (M+Na): 263.1048; found: 263.1053

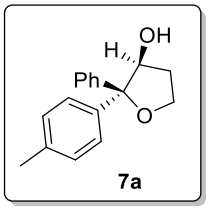
Spectral data for 2-(4-chlorophenyl)-5-methyl-2-phenylfuran-3(*2H*)-one (6)



6

colorless oil, (12.2 mg, 0.042 mmol, 45%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.38 ~ 7.32 (m, 4H), 7.31 ~ 7.23 (m, 5H), 5.48 (s, 1H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 202.1, 189.0, 137.8, 136.7, 134.4, 128.6, 128.5, 128.5, 127.9, 126.4, 103.3, 92.3, 17.0; HRMS-ESI+ calcd. for $C_{17}H_{13}O_2Cl$: 284.0604; found: 284.0607

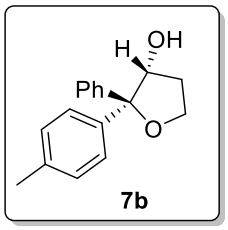
Spectral data for (2*R*,3*S*)-2-phenyl-2-(*p*-tolyl)tetrahydrofuran-3-ol (7a)



7a

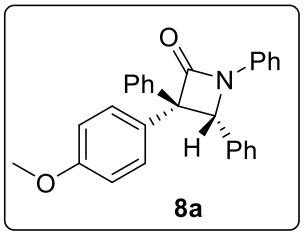
White solid, (21 mg, 0.077 mol, 40%) ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.48 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.30 (t, *J* = 8.4 Hz, 2H), 7.19 (t, *J* = 7.0 Hz, 1H), 7.11 (d, *J* = 8.4 Hz, 2H), 4.95 ~ 4.93 (m, 1H), 4.25 (q, *J* = 7.7 Hz, 1H), 3.97 ~ 3.93 (m, 1H), 2.28 (d, *J* = 8.4 Hz, 3H), 2.08 ~ 2.04 (m, 2H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 141.6, 141.0, 136.8, 129.2, 129.1, 128.4, 128.4, 127.1, 126.5, 126.4, 125.4, 125.4, 90.9, 76.2, 65.1, 33.4, 20.9 ; HRMS-ESI+ calcd. for $C_{17}H_{18}O_2$ (M+Na): 277.1204; found: 277.1219

Spectral data for (2*R*,3*R*)-2-phenyl-2-(*p*-tolyl)tetrahydrofuran-3-ol (7b)



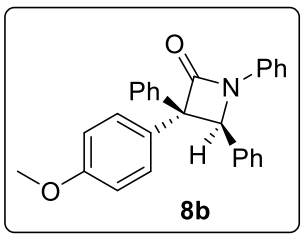
colorless oil, (8 mg, 0.029 mmol, 15%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.49 (d, *J* = 7.7 Hz, 2H), 7.37 (d, *J* = 7.7 Hz, 2H), 7.30 (t, *J* = 7.7 Hz, 2H), 7.20 (t, *J* = 7.0 Hz, 1H), 7.11 (d, *J* = 7.7 Hz, 2H), 4.94 (d, *J* = 2.8 Hz, 1H), 4.26 (q, *J* = 7.7 Hz, 1H), 3.95 (q, *J* = 7.7 Hz, 1H), 2.28 (d, *J* = 8.4 Hz, 3H), 2.06 ~ 2.04 (m, 2H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 144.1, 138.4, 136.9, 129.2, 129.1, 128.4, 128.4, 127.0, 126.4, 126.4, 125.4, 125.4, 90.9, 76.2, 65.1, 33.3, 20.9; HRMS-ESI+ calcd. for $C_{17}H_{18}O_2$ (M+Na): 277.1204; found: 277.1203.

Spectral data for (3*S*,4*R*)-3-(4-methoxyphenyl)-1,3,4-triphenylazetidin-2-one (8a)



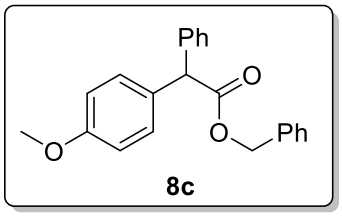
White solid, (37.8 mg, 0.093 mmol, 47%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.55 (d, *J* = 8.5 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.23 (t, *J* = 7.5 Hz, 2H), 7.11 (s, 5H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.04 ~ 6.97 (m, 4H), 6.89 (d, *J* = 7.0 Hz, 2H), 5.74 (s, 1H), 3.77 (s, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.3, 158.8, 137.5, 137.4, 134.9, 132.8, 129.0, 128.4, 128.3, 128.3,128.0, 127.8, 127.5, 126.6, 124.0, 117.4, 114.1, 71.7, 67.3, 55.3; HRMS-ESI+ calcd. for $C_{28}H_{23}NO_2$ (M+Na): 428.1626; found: 428.1626.

Spectral data for (3*S*,4*S*)-3-(4-methoxyphenyl)-1,3,4-triphenylazetidin-2-one (8b)



White solid, (16 mg, 0.039 mmol, 20%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.62 (d, *J* = 7.5 Hz, 2H), 7.36 (t, *J* = 8.5 Hz, 4H), 7.27 ~ 7.21 (m, 4H), 7.12 (s, 5H), 7.04 ~ 6.99 (m, 3H), 6.52 (d, *J* = 8.5 Hz, 2H), 5.76 (s, 1H), 3.63 (s, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ 167.3, 158.2, 141.0, 137.5, 134.9, 129.5, 129.3, 129.0, 128.7, 128.3, 128.1, 127.5, 127.3, 127.2, 124.0, 117.4, 113.2, 71.7, 67.1, 55.0; HRMS-ESI+ calcd. for $C_{28}H_{23}NO_2$ (M+Na): 428.1626; found: 428.1626.

Spectral data for benzyl 2-(4-methoxyphenyl)-2-phenylacetate (8c)

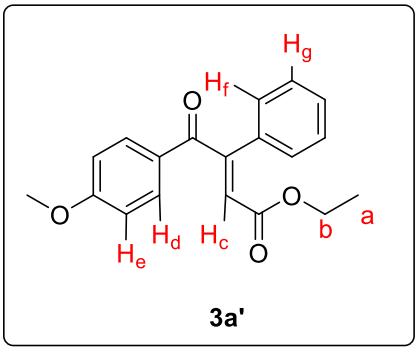


8c

colorless oil, (47 mg, 0.141 mmol, 72%); ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.48 ~ 7.36 (m, 10H), 7.34 (d, *J* = 8.5 Hz, 2H), 6.95 (d, *J* = 9.0 Hz, 2H), 5.29 (s, 2H), 5.14 (s, 1H), 3.89 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 172.5, 158.7, 138.8, 135.6, 130.6, 129.6, 128.5, 128.4, 128.4, 128.1, 128.1, 127.1, 113.9, 66.8, 56.1, 55.2; HRMS-ESI+ calcd. for $C_{22}H_{20}O_3$ (M+Na): 428.1626; found: 428.1626.

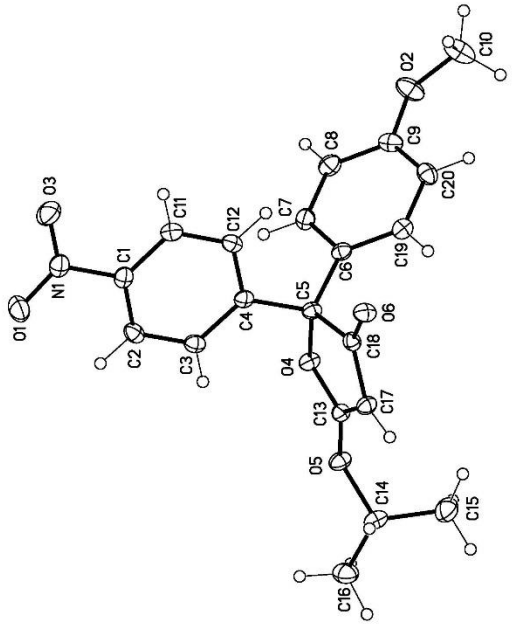
6. NOE of compound 3a’

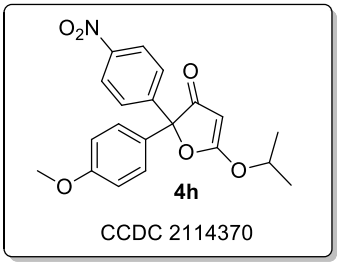
Irradite	Enhancements
H^a (δ, 1.11)	H^b (δ, 4.08, 1.32%)
H^b (δ, 4.08)	H^a (δ, 1.11, 0.89%)
H^c (δ, 6.16)	H^d (δ, 7.90, 0.83%)
H^d (δ, 7.90)	H^c (δ, 6.16, 0.51%), H^e (δ, 6.89, 2.22%),
H^e (δ, 6.89)	H^d (δ, 7.90, 2.53%)
H^f (δ, 7.32)	H^g (δ, 7.38, 1.82%)



3a'

7. X-ray crystallographic data of compound 4h





Ellipsoid contour % probability level = 50%

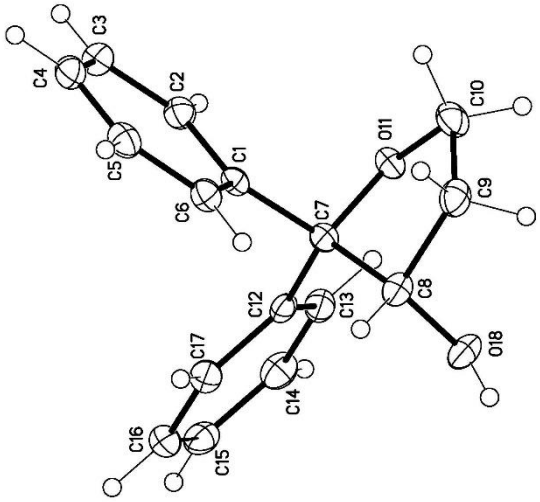
Experimental: The sample was dissolved in appropriate amount of dichlomethane followed by the addition of pentane to furnish a saturated solution. Afterwards, the mixture was allowed to stand at room temperature to form the crystals.

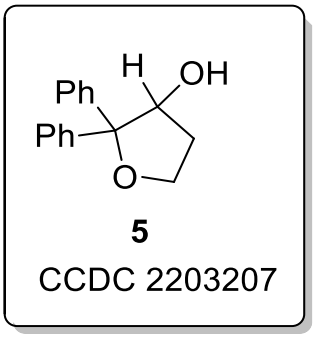
Table 1. Crystal data and structure refinement for 210951LT_0M.

Identification code	210951lt_0m	
Empirical formula	C20 H19 N O6	
Formula weight	369.36	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 6.4622(4) Å	α = 86.413(3)°.
	b = 10.4948(8) Å	β = 89.241(3)°.
	c = 14.1200(10) Å	γ = 73.335(3)°.

Volume	915.58(11) $Å^3$
Z	2
Density (calculated)	1.340 Mg/m^3
Absorption coefficient	0.100 mm^{-1}
F(000)	388
Crystal size	0.18 x 0.06 x 0.06 mm^3
Theta range for data collection	2.029 to 26.433°.
Index ranges	-8<=h<=7, -13<=k<=13, -17<=l<=17
Reflections collected	13572
Independent reflections	3726 [R(int) = 0.0240]
Completeness to theta = 25.242°	99.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7454 and 0.7164
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3726 / 0 / 247
Goodness-of-fit on F^2	1.040
Final R indices [I>2sigma(I)]	R1 = 0.0358, wR2 = 0.0860
R indices (all data)	R1 = 0.0415, wR2 = 0.0897
Extinction coefficient	n/a
Largest diff. peak and hole	0.356 and -0.245 $e.Å^{-3}$

X-ray crystallographic data of compound 5





Ellipsoid contour % probability level = 50%

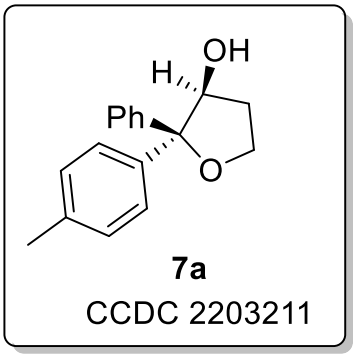
Experimental: The sample was dissolved in appropriate amount of dichlomethane followed by the addition of pentane to furnish a saturated solution. Afterwards, the mixture was allowed to stand at room temperature to form the crystals.

Table 2 Crystal data and structure refinement for 220747lt_auto.

Identification code	220747lt_auto
Empirical formula	$C_{16}H_{16}O_2$
Formula weight	240.29
Temperature/K	100.01(10)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	11.41961(18)

b/Å	5.65053(12)
c/Å	19.2947(3)
α/°	90
β/°	101.7637(17)
γ/°	90
Volume/Å^3	1218.88(4)
Z	4
ρ_{calc}g/cm^3	1.309
μ/mm^{-1}	0.675
F(000)	512.0
Crystal size/mm^3	0.13 × 0.12 × 0.1
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	7.908 to 134.154
Index ranges	$-10 \leq h \leq 13$, $-6 \leq k \leq 5$, $-22 \leq l \leq 23$
Reflections collected	8591
Independent reflections	2167 [R_{int} = 0.0186, R_{sigma} = 0.0179]
Data/restraints/parameters	2167/0/165
Goodness-of-fit on F^2	1.070
Final R indexes [I>=2σ (I)]	R_1 = 0.0322, wR_2 = 0.0796
Final R indexes [all data]	R_1 = 0.0345, wR_2 = 0.0813
Largest diff. peak/hole / e Å^{-3}	0.23/-0.18

X-ray crystallographic data of compound 7a



Ellipsoid contour % probability level = 50%

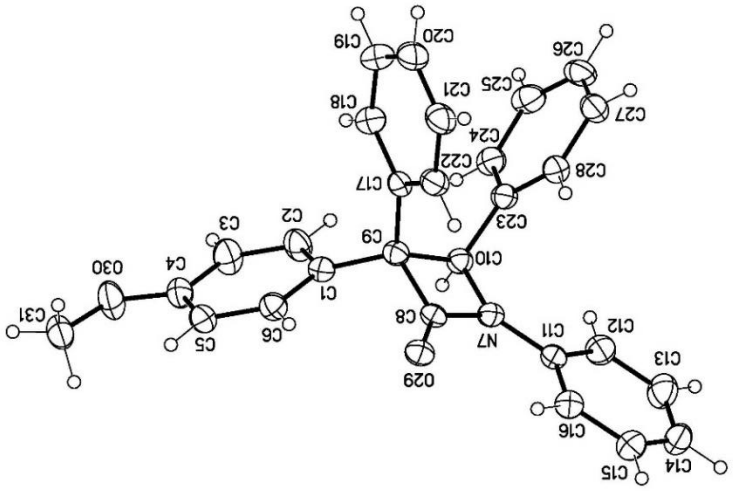
Experimental: The sample was dissolved in appropriate amount of dichlomethane followed by the addition of pentane to furnish a saturated solution. Afterwards, the mixture was allowed to stand at room temperature to form the crystals.

Table 3 Crystal data and structure refinement for 220784lt_auto.

Identification code	220784lt_auto
Empirical formula	$C_{34}H_{36}O_4$
Formula weight	508.63

Temperature/K	99.99(10)
Crystal system	triclinic
Space group	P-1
a/Å	5.94355(19)
b/Å	13.5482(4)
c/Å	17.6441(5)
α/°	75.901(3)
β/°	80.614(3)
γ/°	79.208(3)
Volume/Å^3	1343.28(8)
Z	2
ρ_{calc}g/cm^3	1.258
μ/mm^{-1}	0.640
F(000)	544.0
Crystal size/mm^3	0.16 × 0.07 × 0.02
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	5.204 to 134.152
Index ranges	-7 ≤ h ≤ 5, -16 ≤ k ≤ 16, -21 ≤ l ≤ 21
Reflections collected	15811
Independent reflections	4759 [R_{int} = 0.0297, R_{sigma} = 0.0310]
Data/restraints/parameters	4759/0/348
Goodness-of-fit on F^2	1.043
Final R indexes [I>=2σ (I)]	R_1 = 0.0407, wR_2 = 0.1038
Final R indexes [all data]	R_1 = 0.0505, wR_2 = 0.1088
Largest diff. peak/hole / e Å^{-3}	0.21/-0.24

X-ray crystallographic data of compound 8b



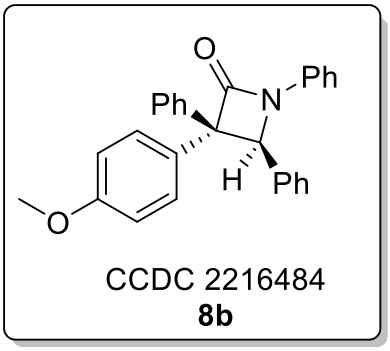
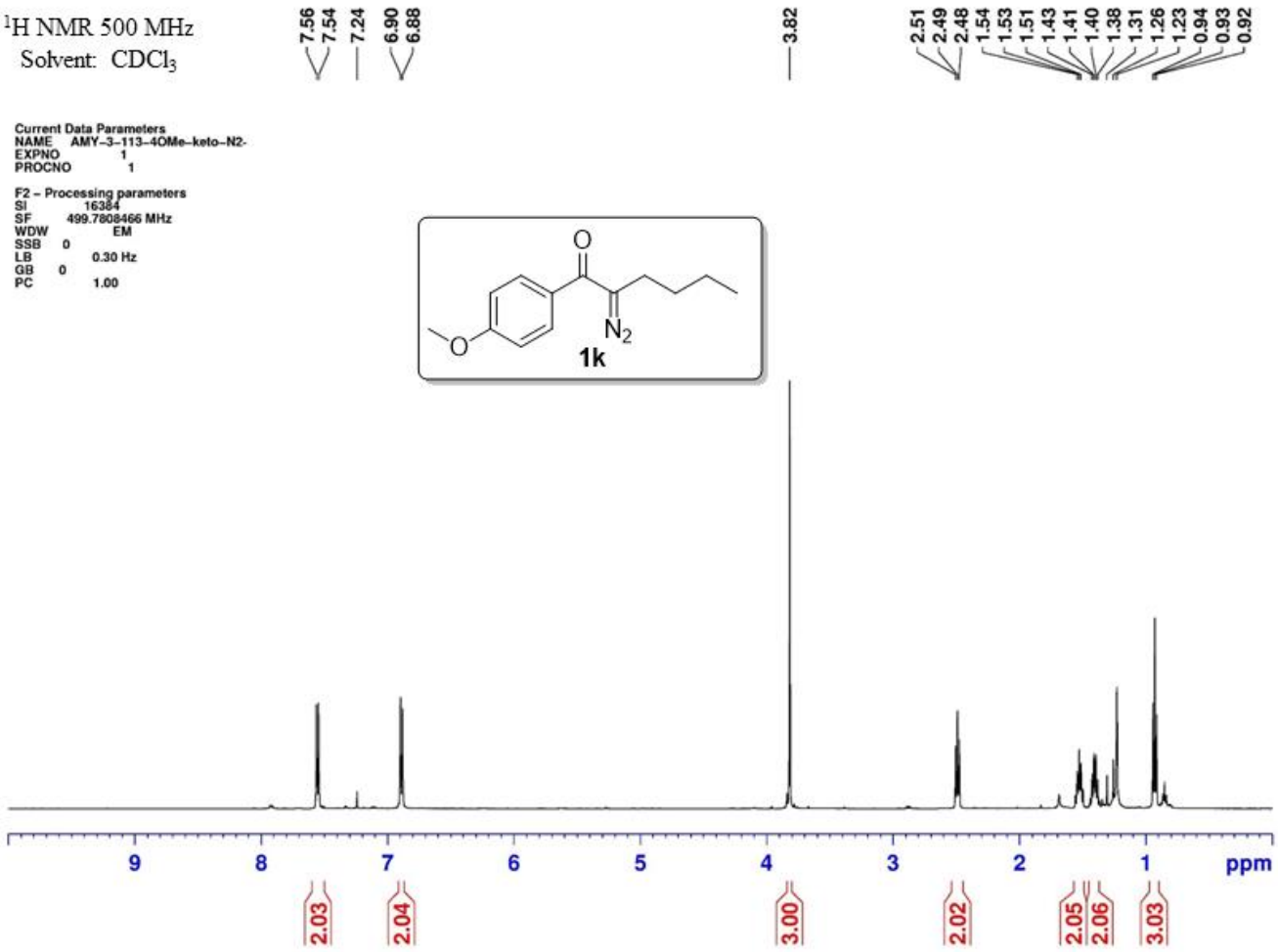


Table 1 Crystal data and structure refinement for 2210100LT_auto.

Identification code	2210100LT_auto
Empirical formula	$C_{28}H_{23}NO_2$
Formula weight	405.47
Temperature/K	100.00(11)
Crystal system	orthorhombic
Space group	Pbca
a/Å	13.66639(16)
b/Å	17.3063(2)
c/Å	18.4966(2)
α/°	90

β/°	90
γ/°	90
Volume/Å^3	4374.70(9)
Z	8
ρ_{calc}g/cm^3	1.231
μ/mm^{-1}	0.606
F(000)	1712.0
Crystal size/mm^3	0.09 × 0.03 × 0.02
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	9.532 to 134.156
Index ranges	-16 ≤ h ≤ 15, -20 ≤ k ≤ 20, -22 ≤ l ≤ 21
Reflections collected	26604
Independent reflections	3913 [R_{int} = 0.0251, R_{sigma} = 0.0179]
Data/restraints/parameters	3913/0/282
Goodness-of-fit on F^2	1.049
Final R indexes [I>=2σ (I)]	R_1 = 0.0330, wR_2 = 0.0791
Final R indexes [all data]	R_1 = 0.0386, wR_2 = 0.0820
Largest diff. peak/hole / e Å^{-3}	0.20/-0.15

8. ^{1}H and ^{13}C Spectra of Key Compounds

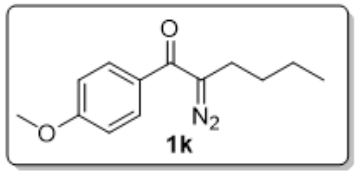


^{13}C NMR; 125 MHz
Solvent: $CDCl_3$

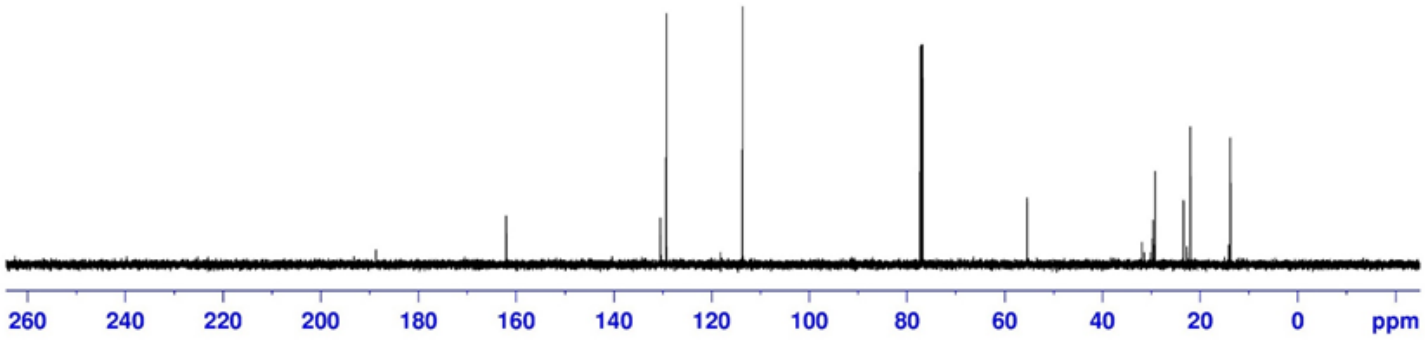
188.67 161.99 130.46 129.22 113.64 77.25 77.00 76.75 55.35 29.18 23.35 21.96 13.76

Current Data Parameters
NAME AMY-3-113-4OMe-keto-N2-Bu-13C.fi
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 125.6728843 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

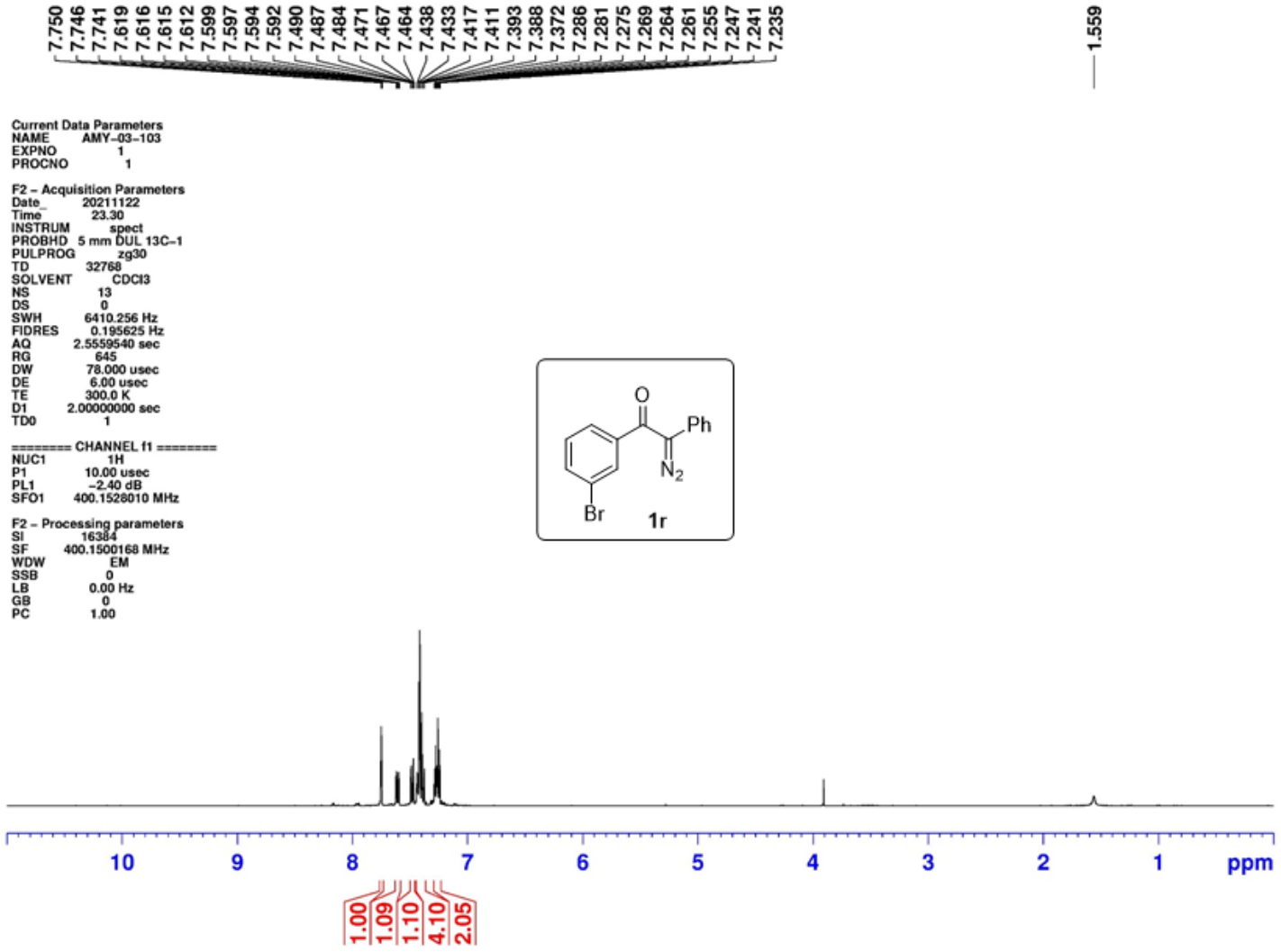


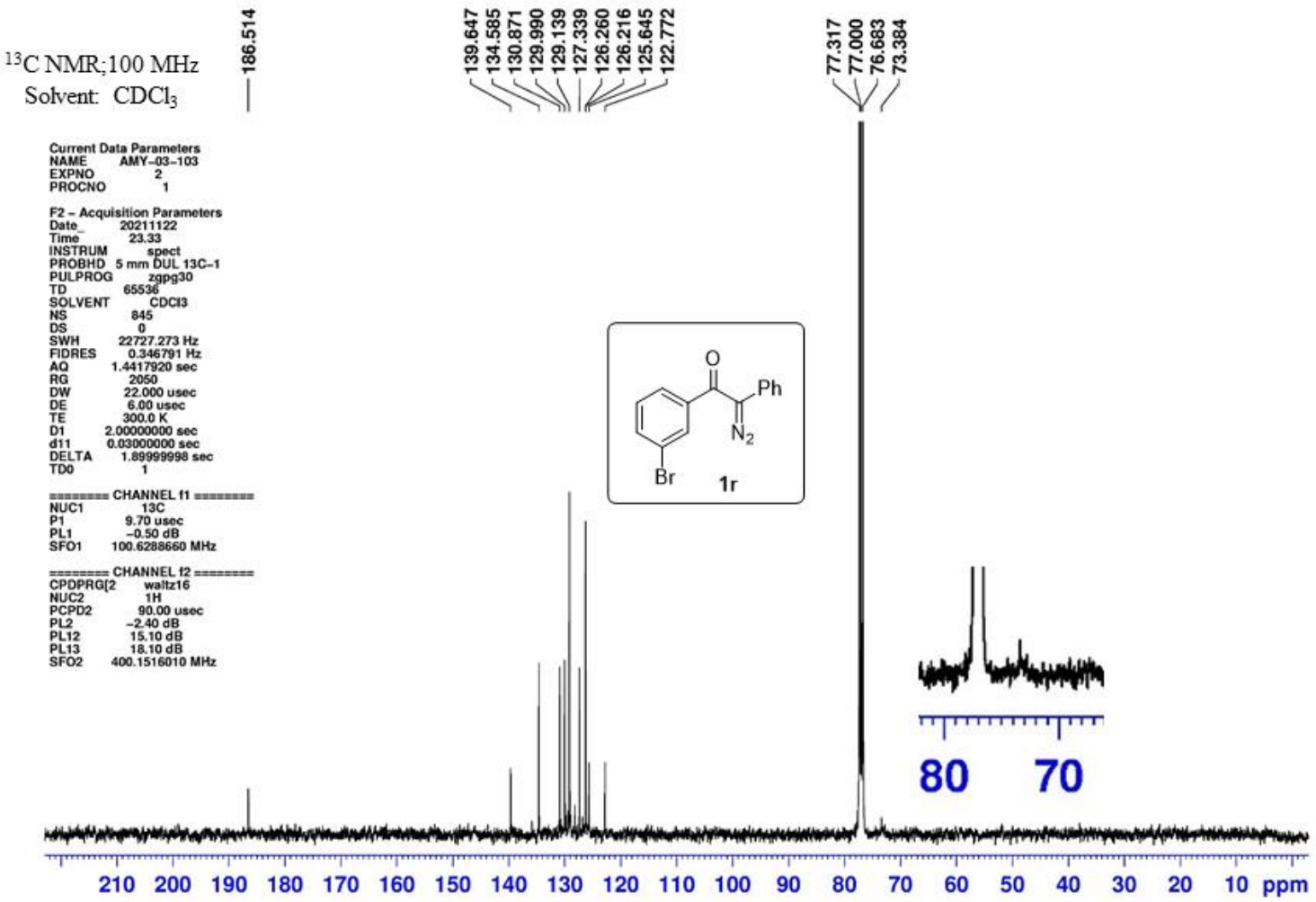
1k



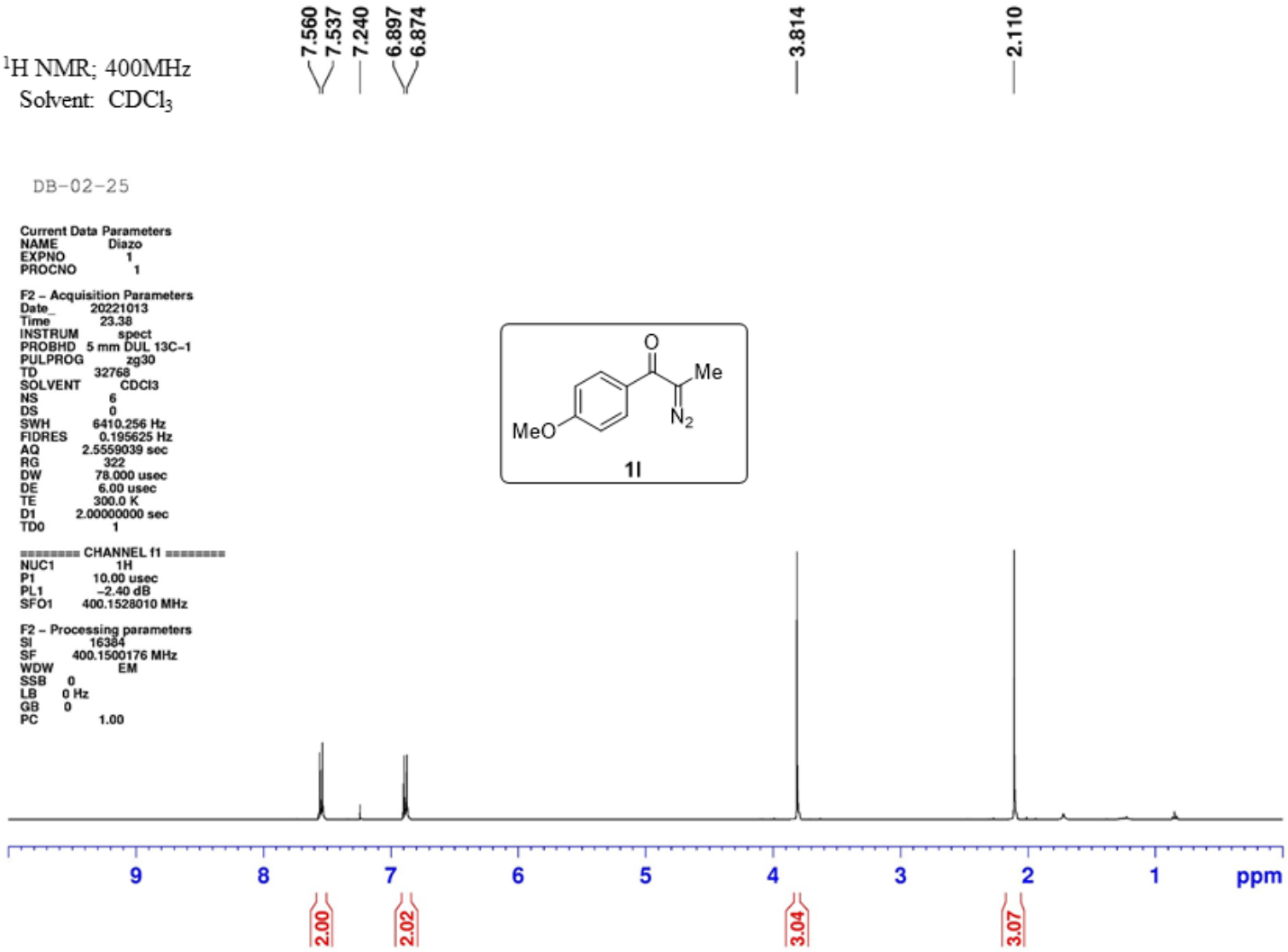
^{1}H NMR; 400 MHz

Solvent: $CDCl_3$

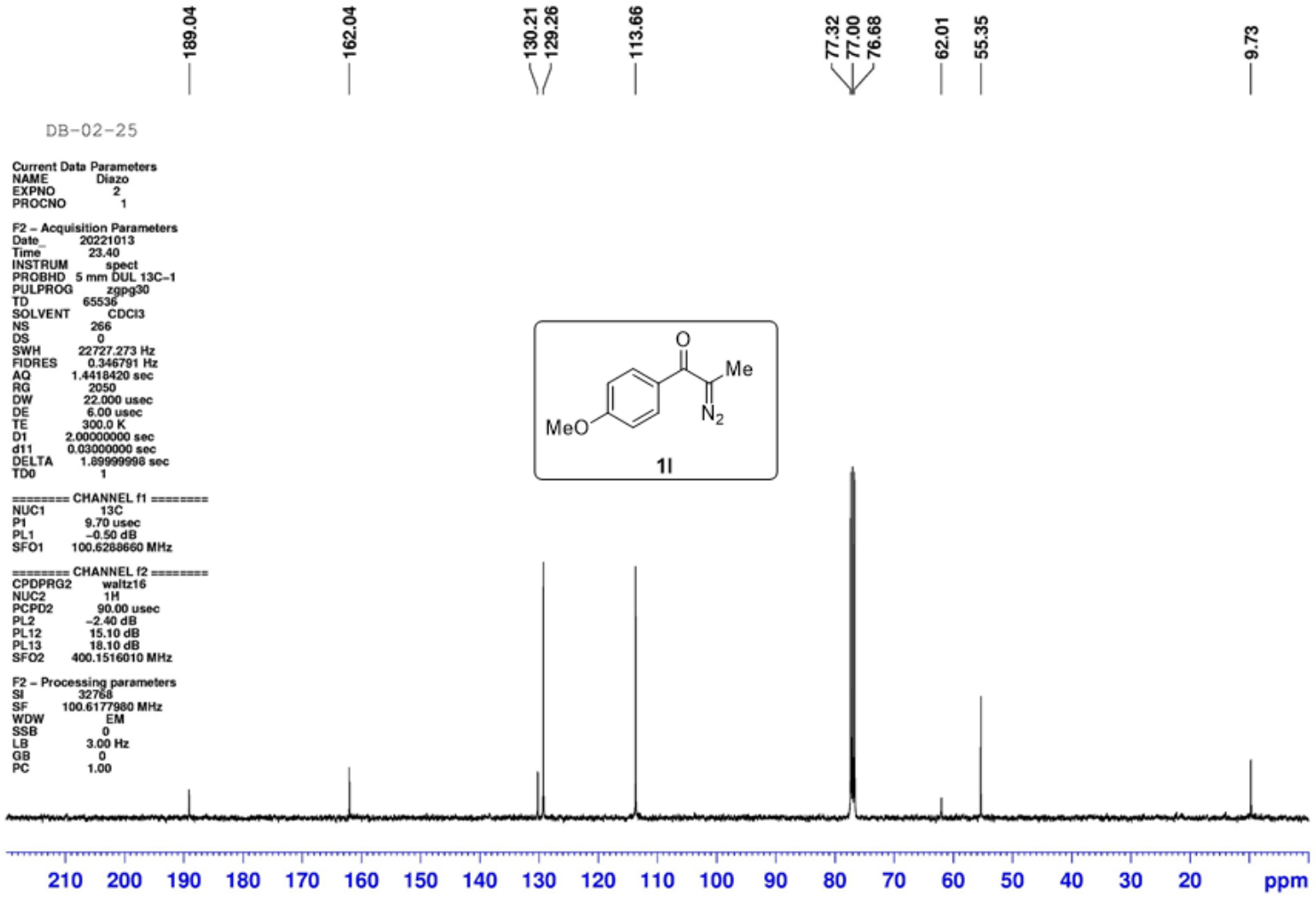



^{13}C NMR;100 MHz
Solvent: $CDCl_3$
186.514
139.647
134.585
130.871
129.990
129.139
127.339
126.260
126.216
125.645
122.772
77.317
77.000
76.683
73.384
Current Data Parameters
NAME AMY-03-103
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20211122
Time 23.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 845
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4417920 sec
RG 2050
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
O
Ph
N_2
Br
1r
80
70
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

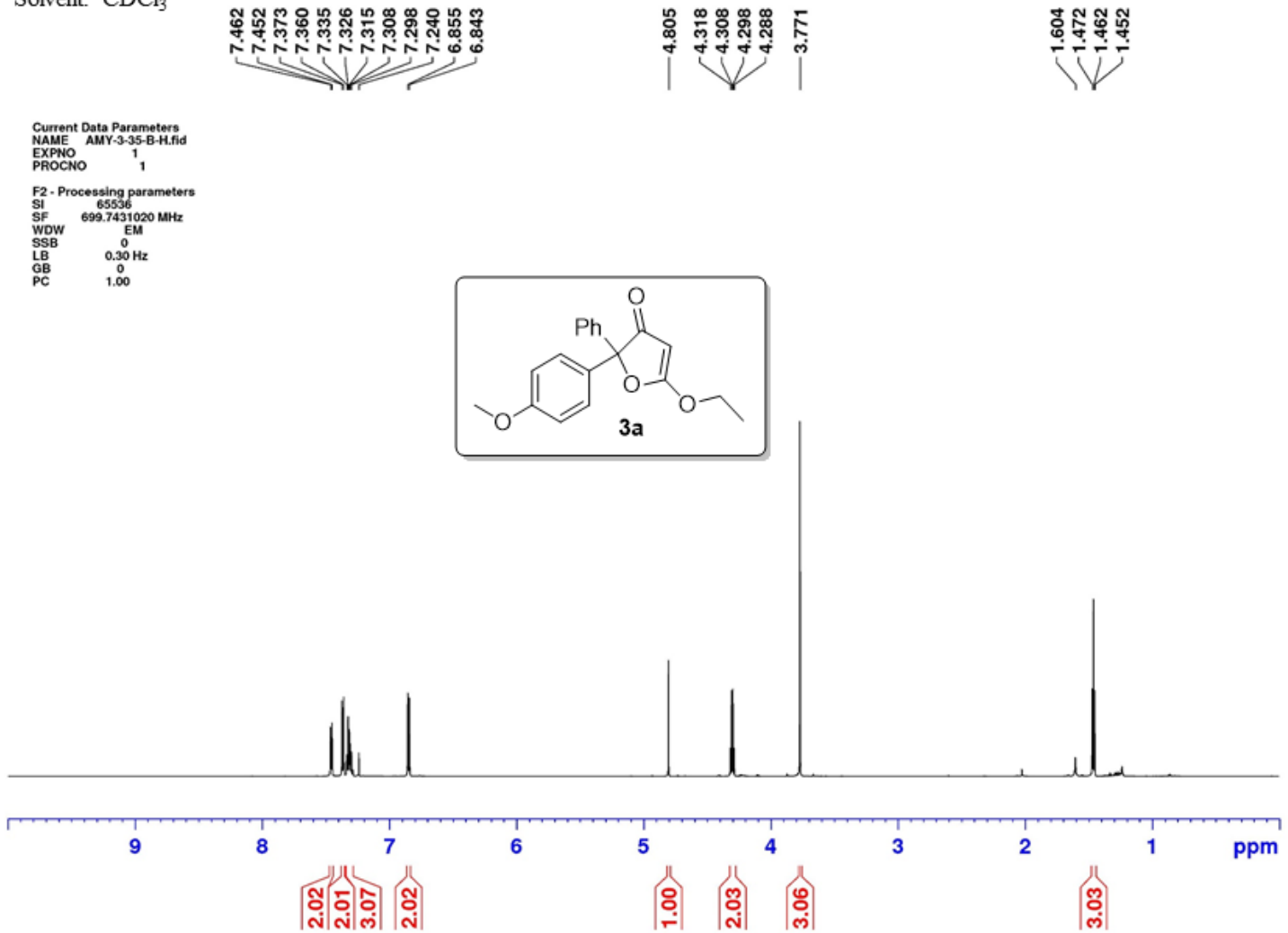
^{1}H NMR; 400MHz
Solvent: $CDCl_3$



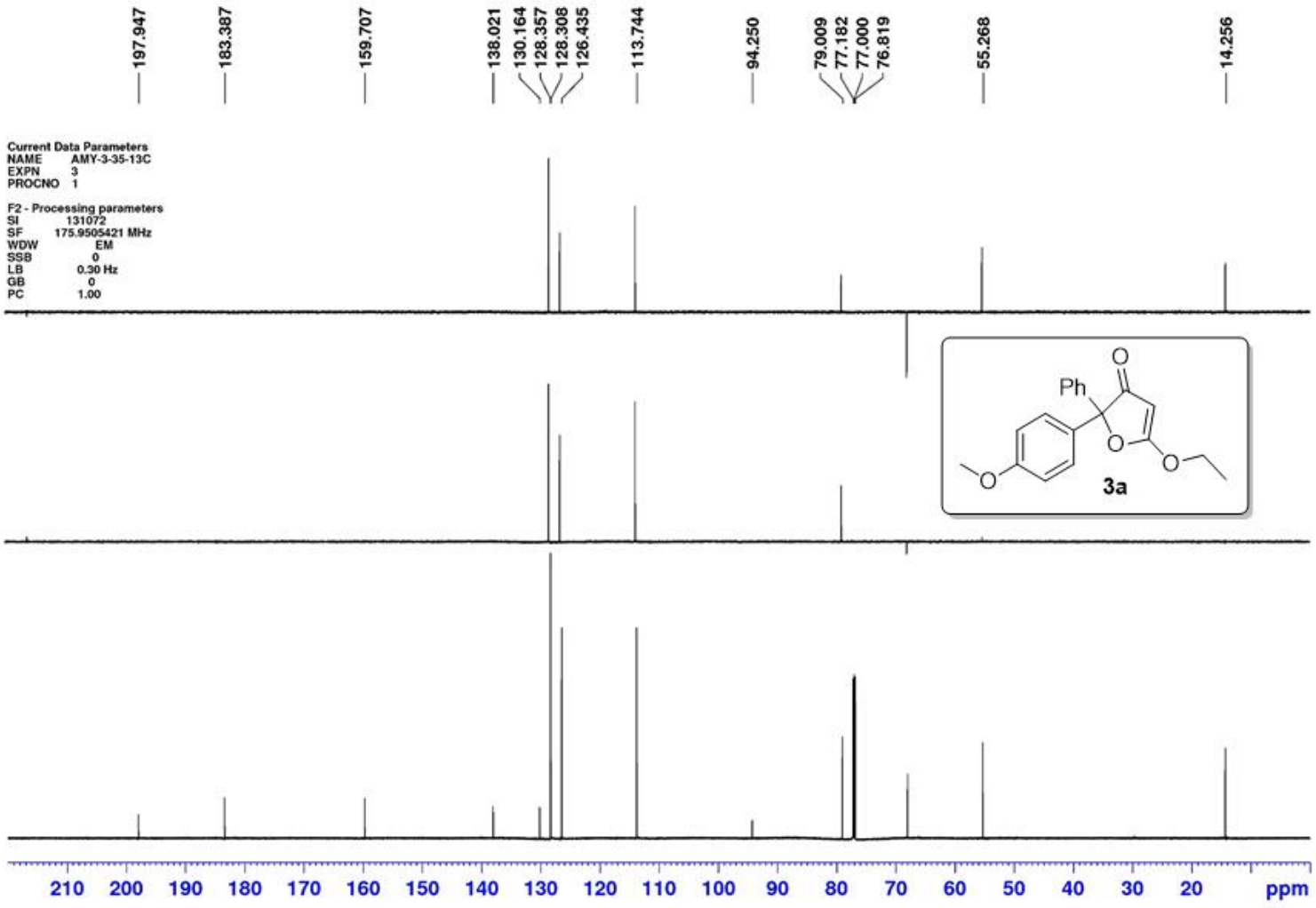
^{13}C NMR; 100 MHz
Solvent: $CDCl_3$

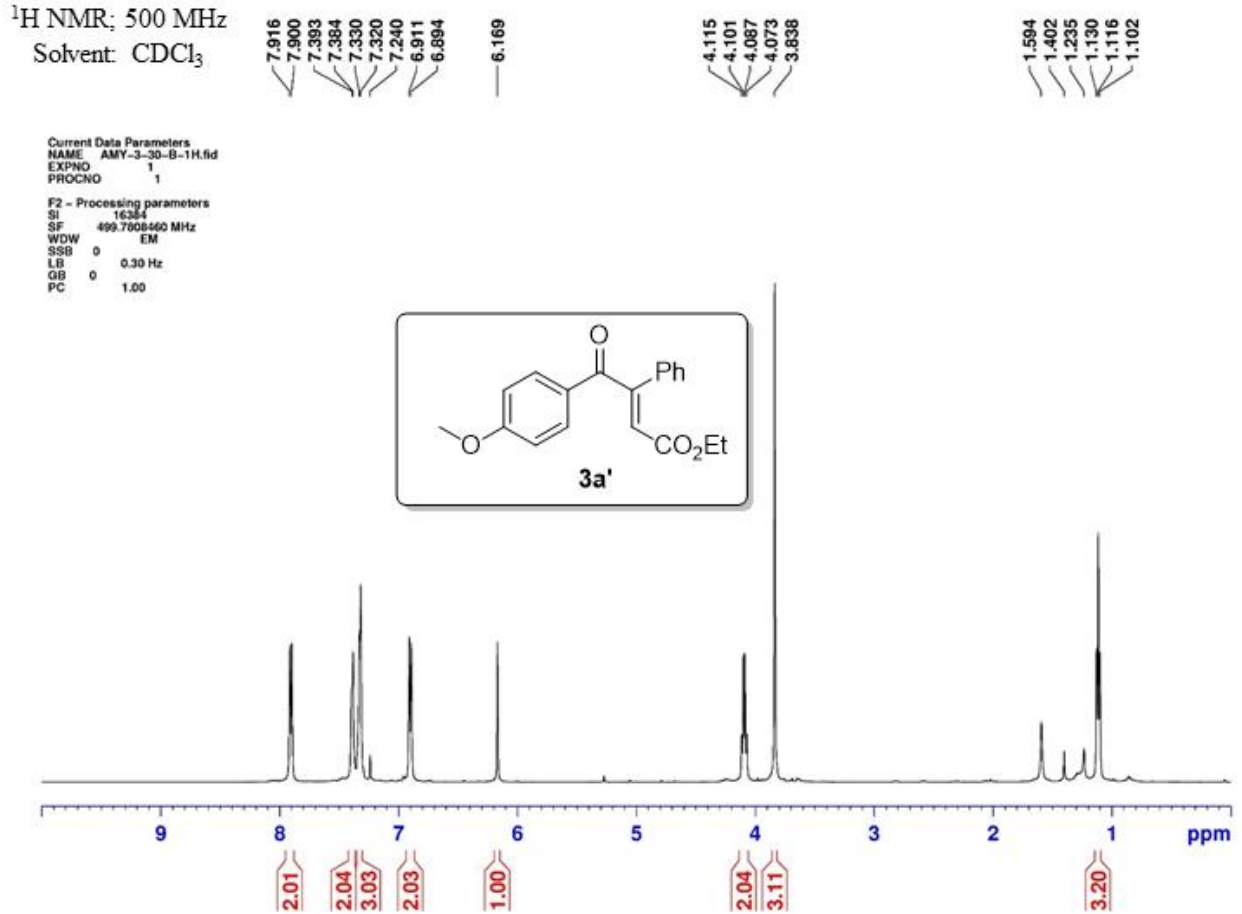


^{1}H NMR; 700 MHz
Solvent: $CDCl_3$

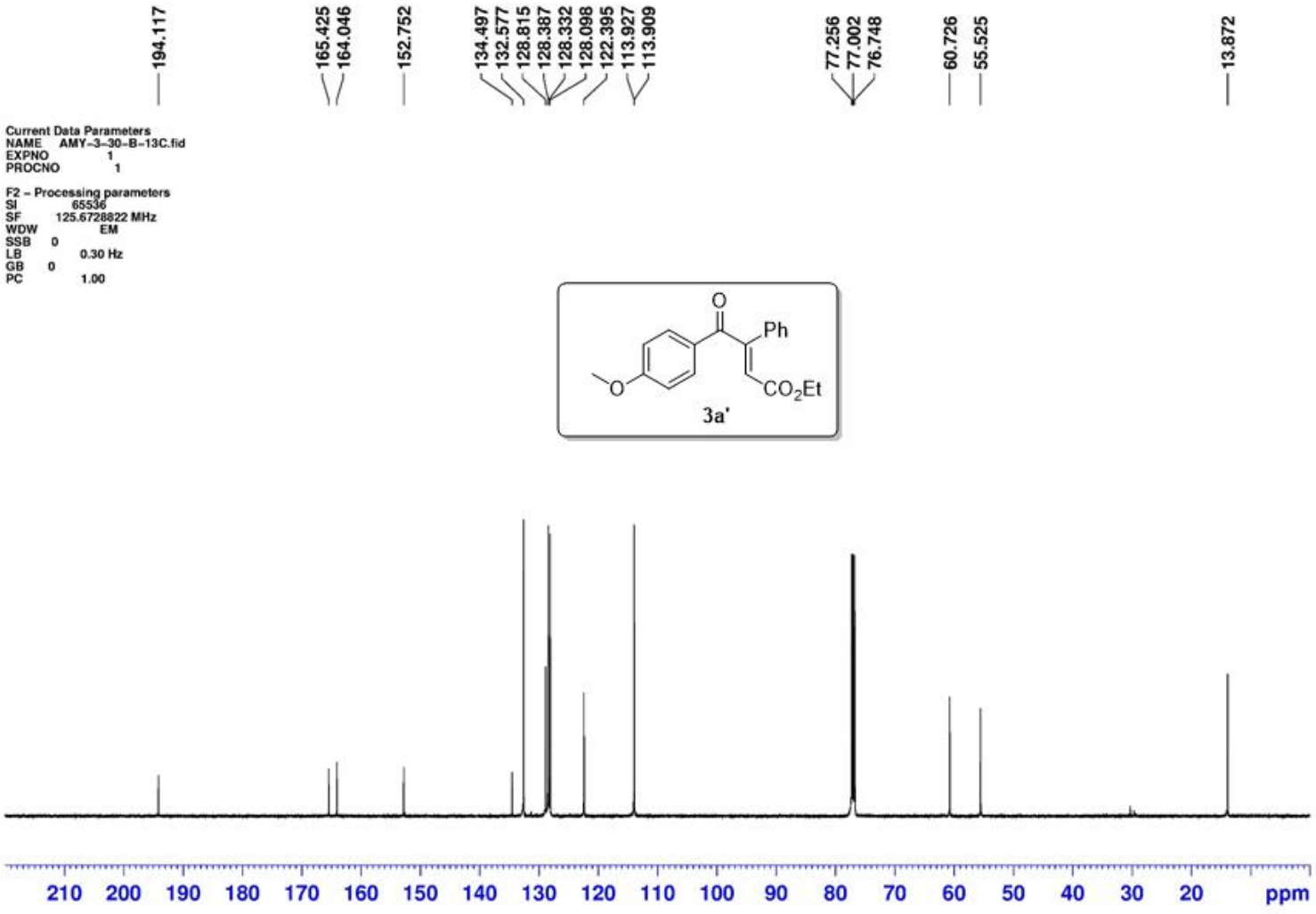


^{13}C NMR; 175 MHz
Solvent: $CDCl_3$



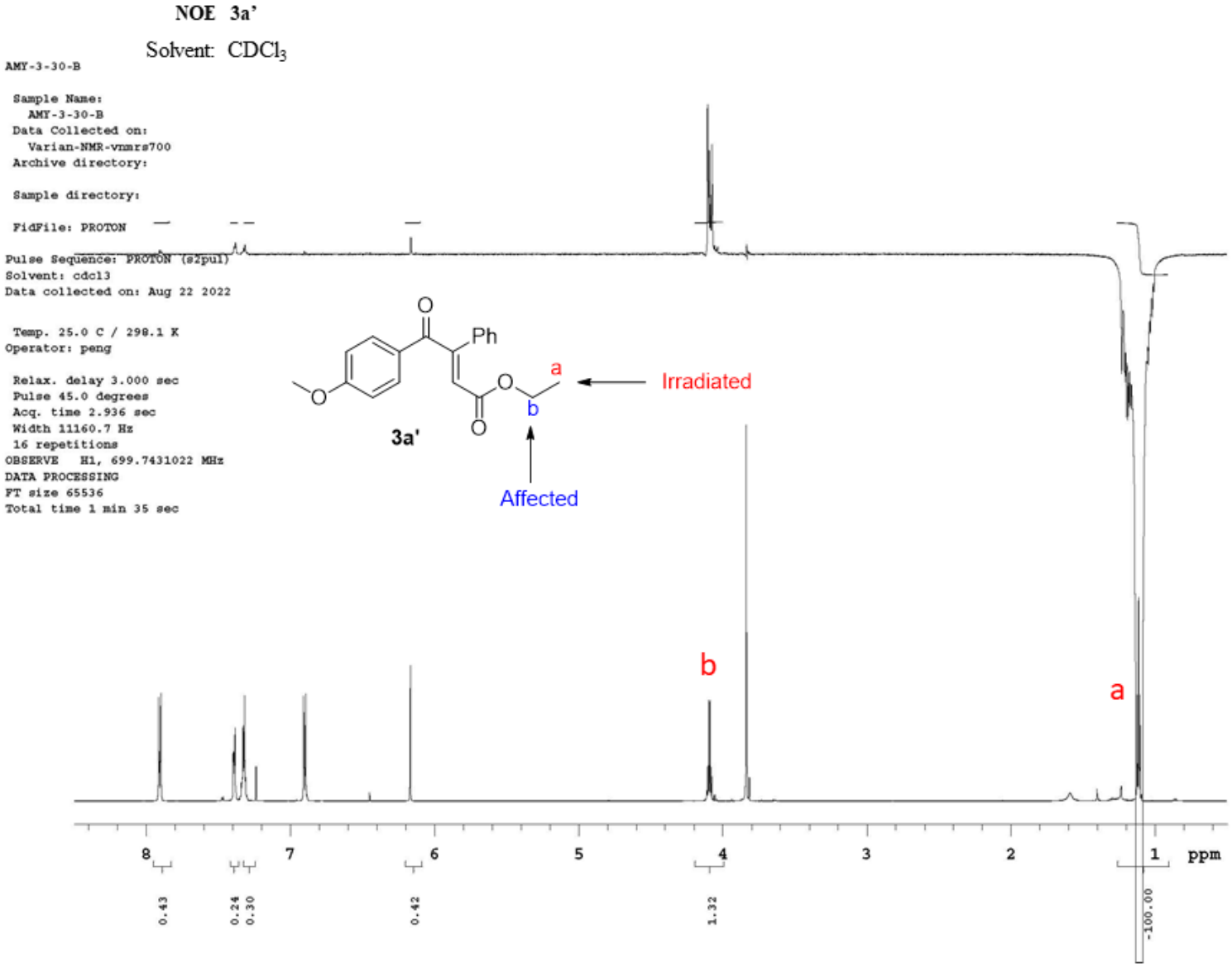

1H NMR; 500 MHz
Solvent: CDCl3
7.916
7.900
7.393
7.384
7.330
7.320
7.240
6.911
6.894
6.169
4.115
4.101
4.087
4.073
3.838
1.594
1.402
1.235
1.130
1.116
1.102
Current Data Parameters
NAME AMY-3-30-B-1H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 16384
SF 499.7808460 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
Ph
O
CO2Et
3a'
9
8
7
6
5
4
3
2
1
ppm
2.01
2.04
3.03
2.03
1.00
2.04
3.11
3.20

^{13}C NMR; 125 MHz
Solvent: $CDCl_3$



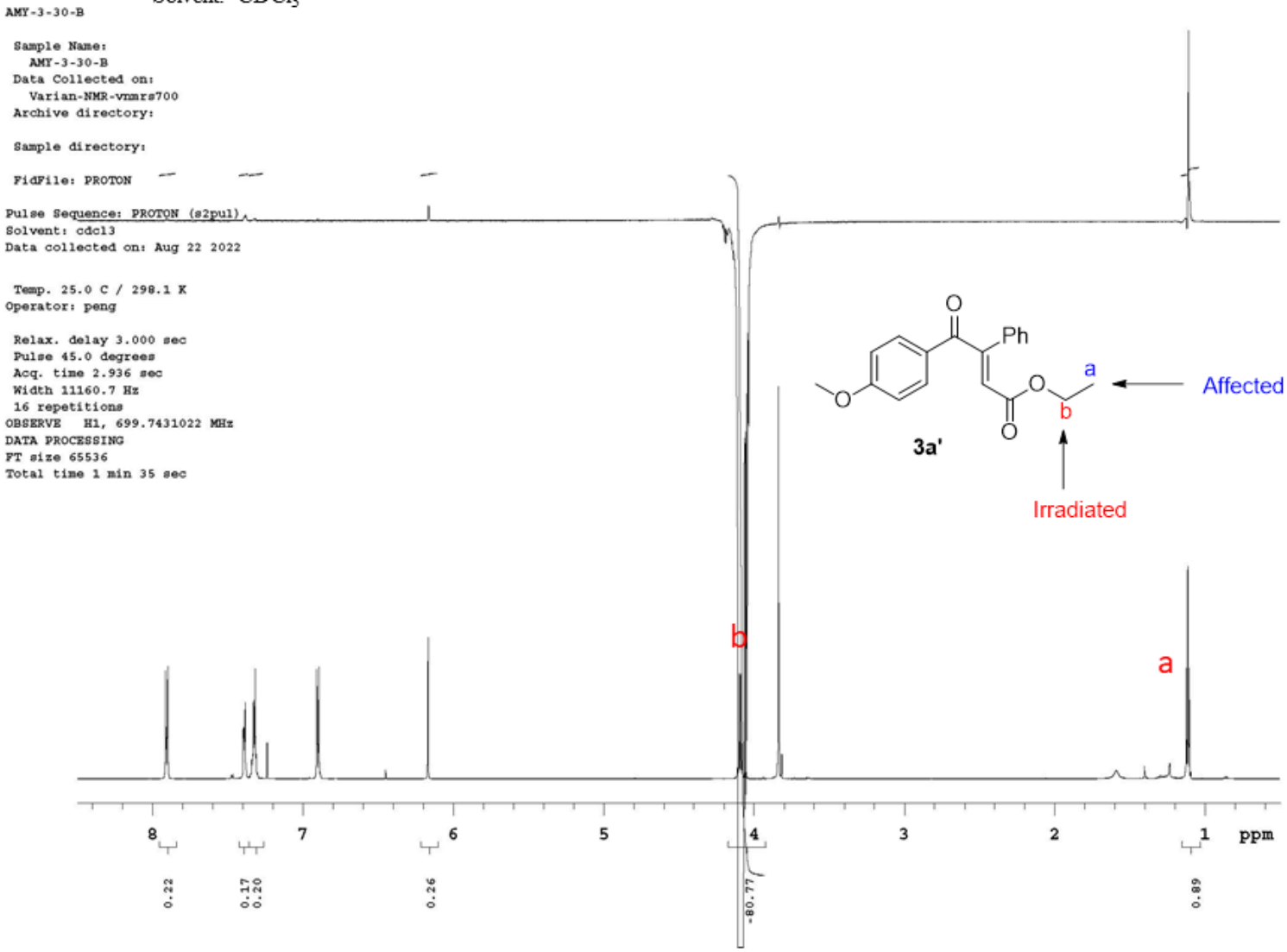
NOE 3a'

Solvent: $CDCl_3$



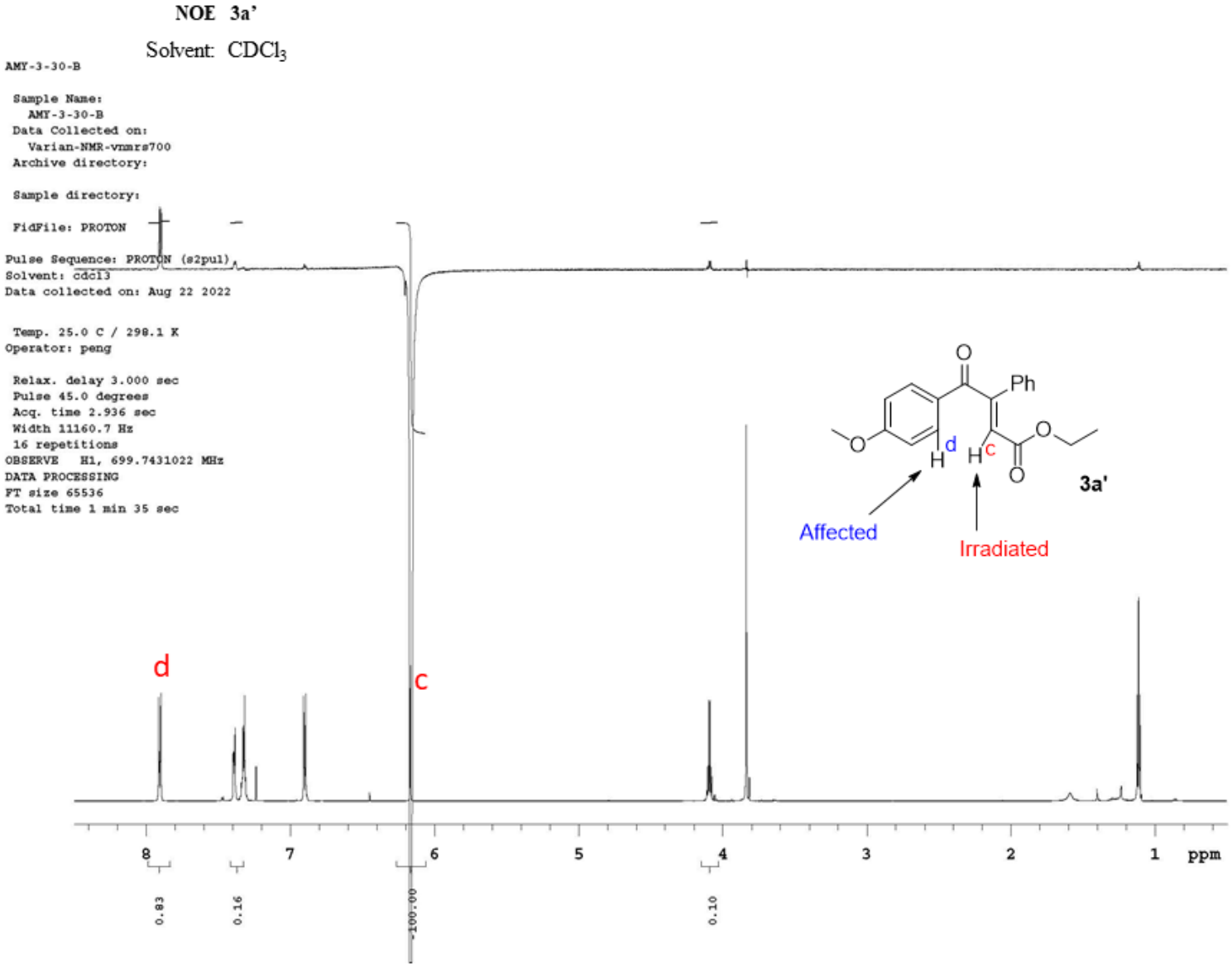
NOE 3a'

Solvent: $CDCl_3$



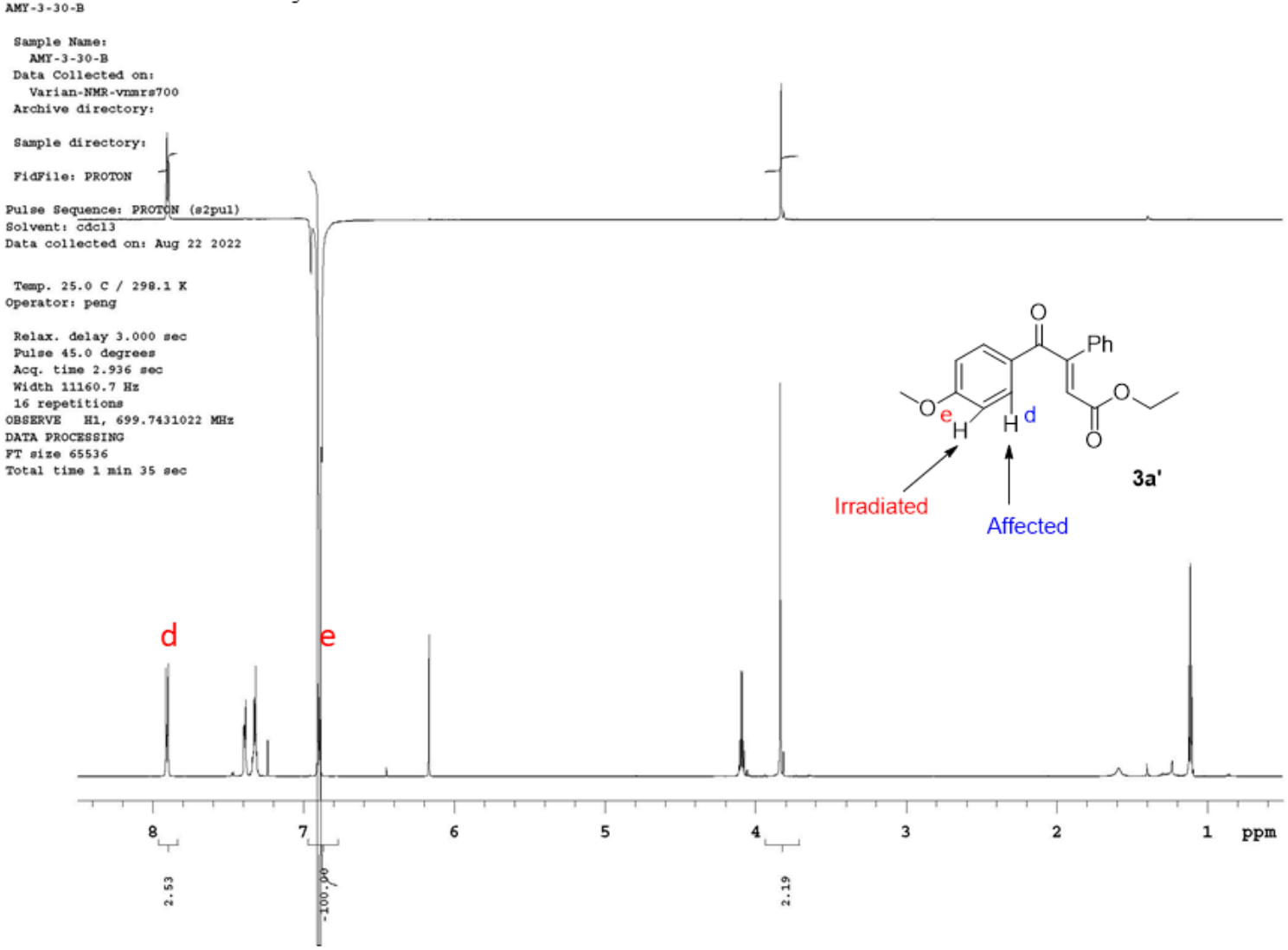
NOE 3a'

Solvent: $CDCl_3$



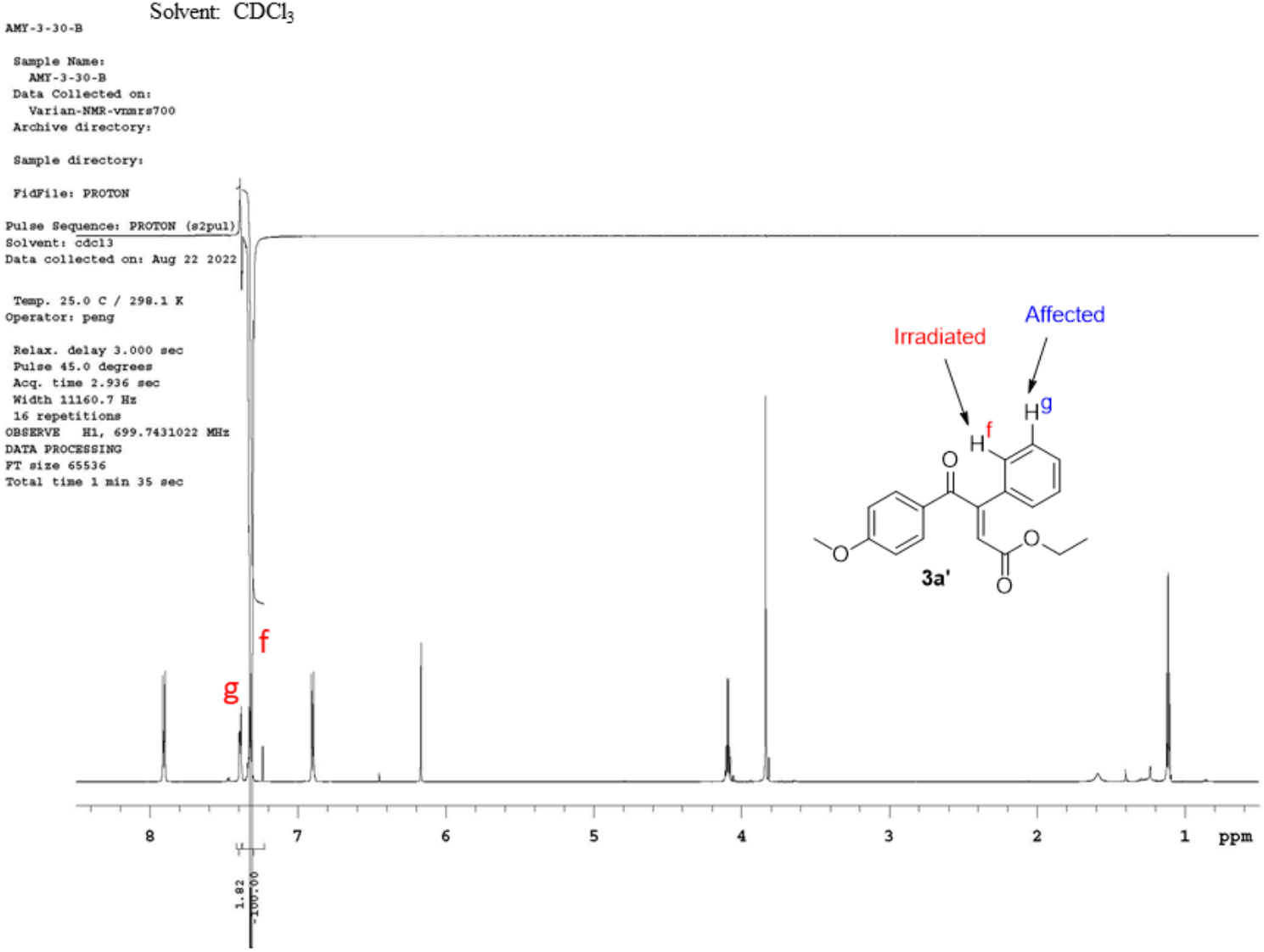
NOE 3a'

Solvent: $CDCl_3$



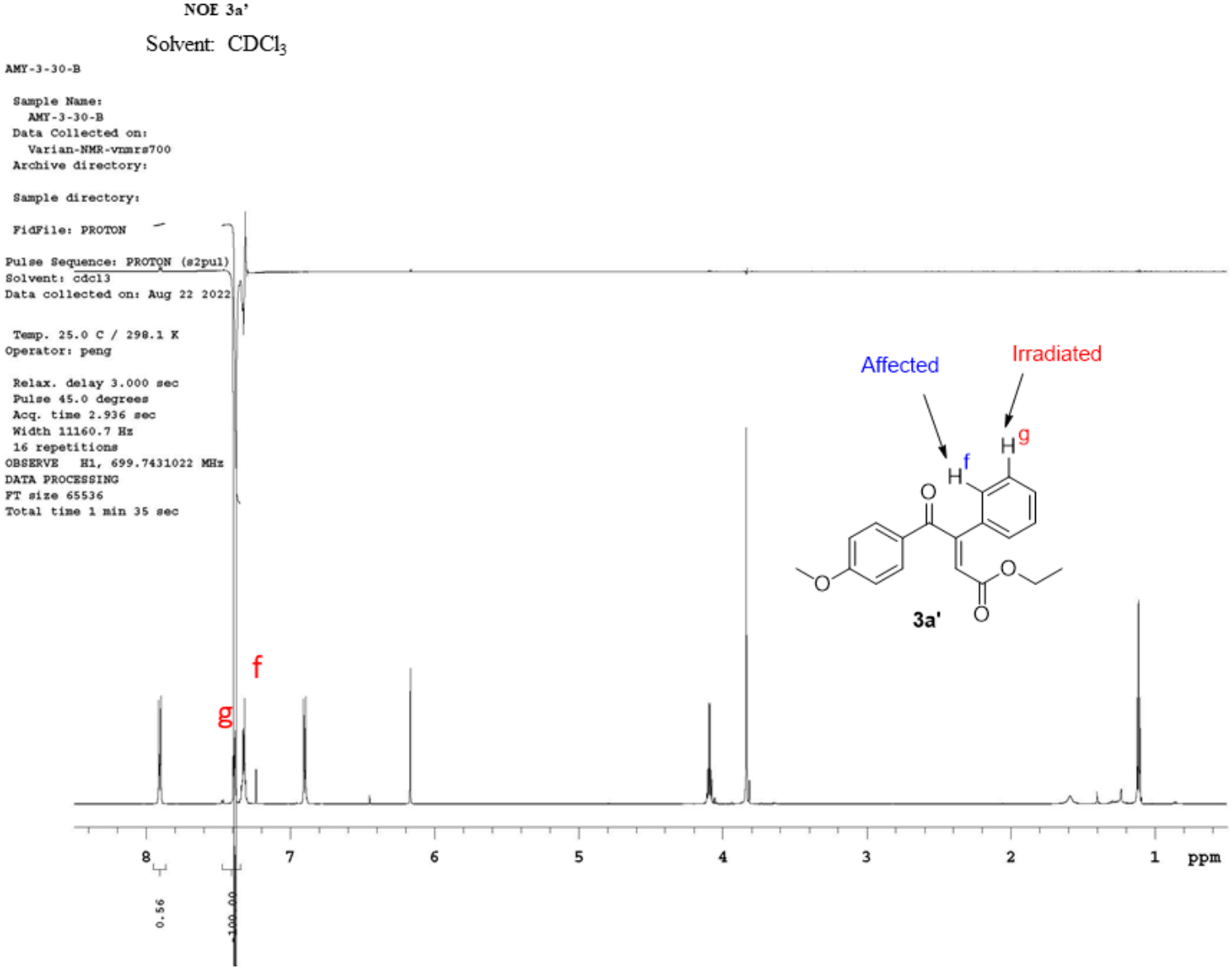
NOE 3a'

Solvent: $CDCl_3$

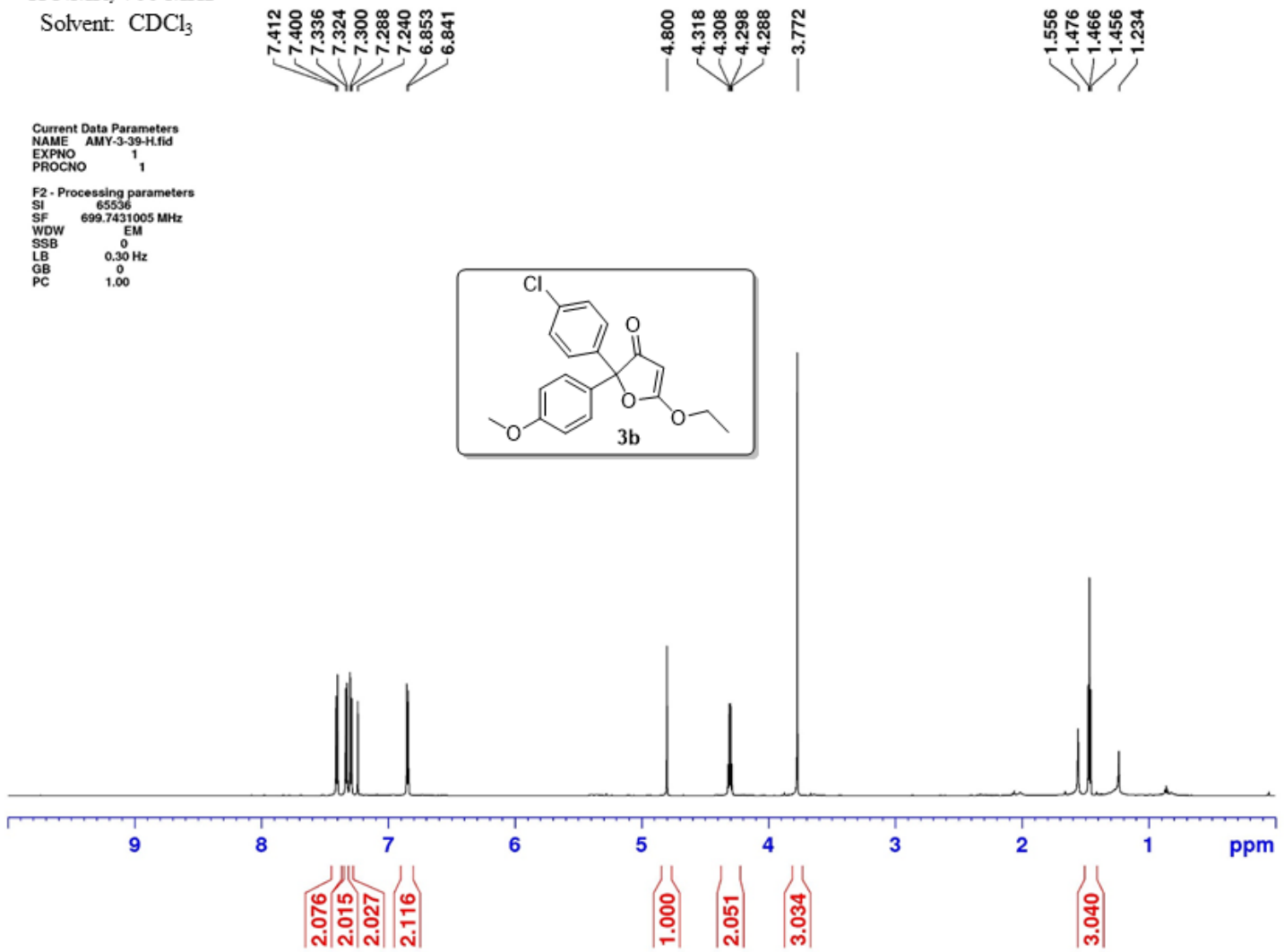


NOE 3a'

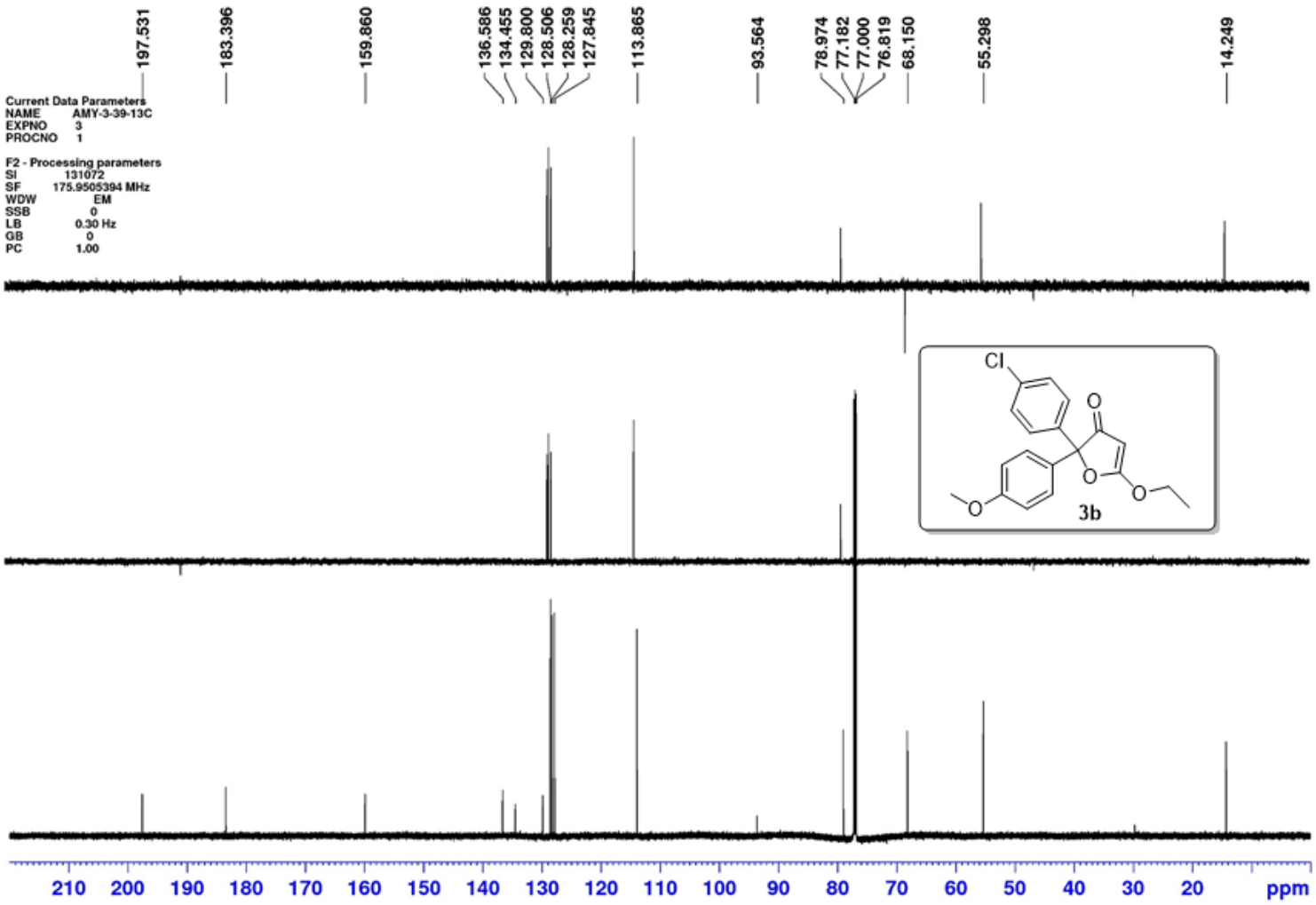
Solvent: $CDCl_3$

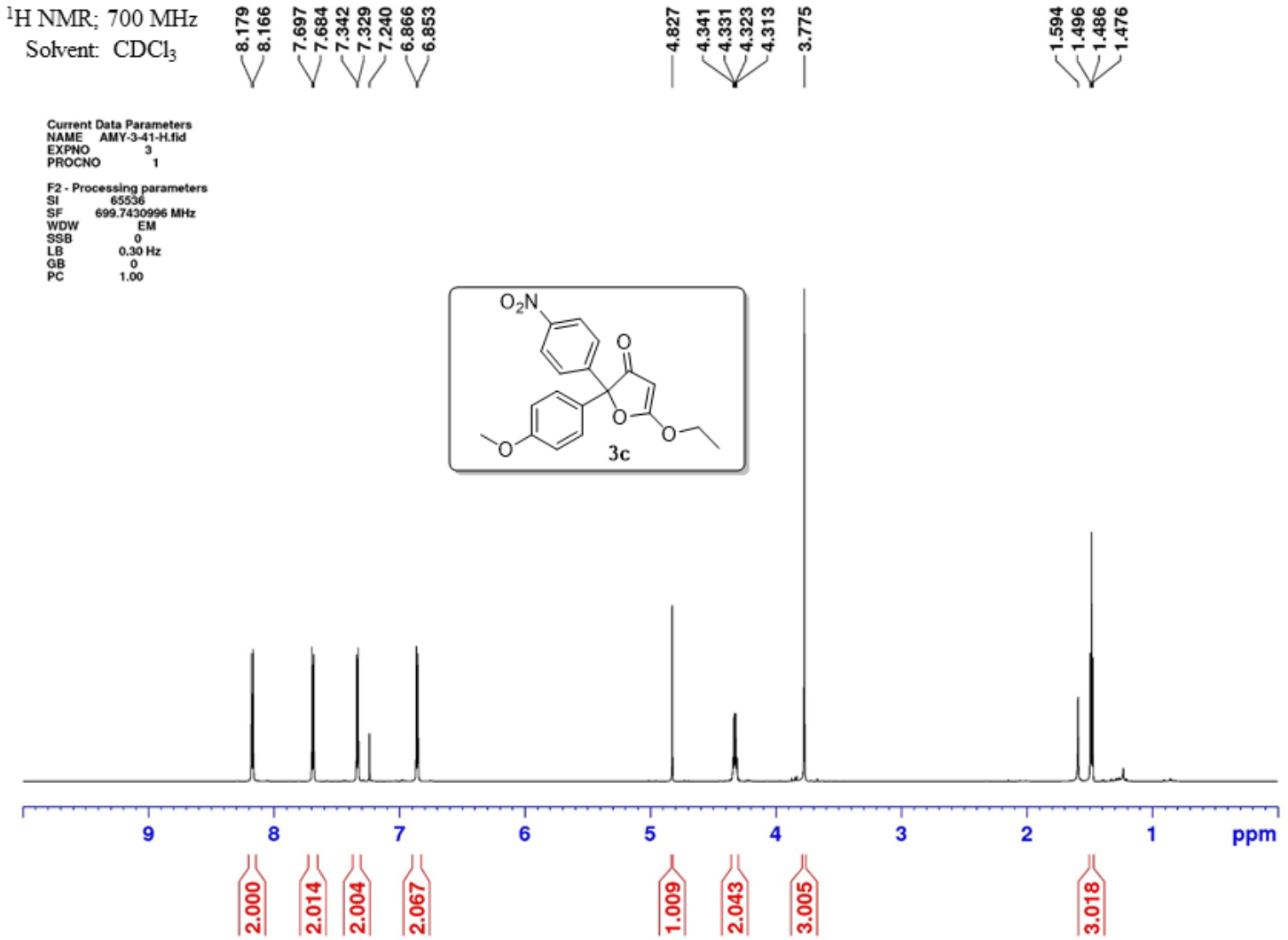


^{1}H NMR; 700 MHz
Solvent: $CDCl_3$

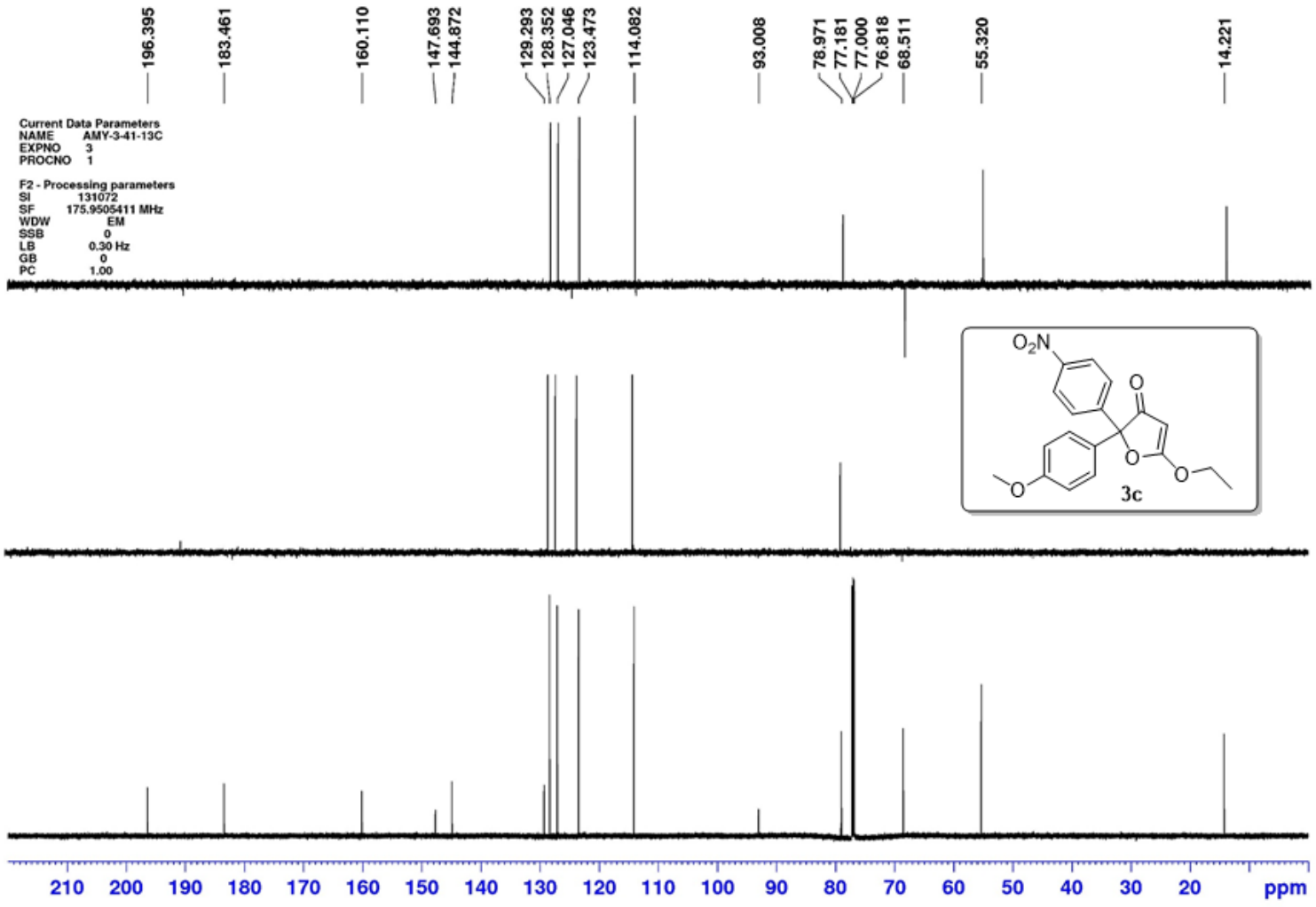


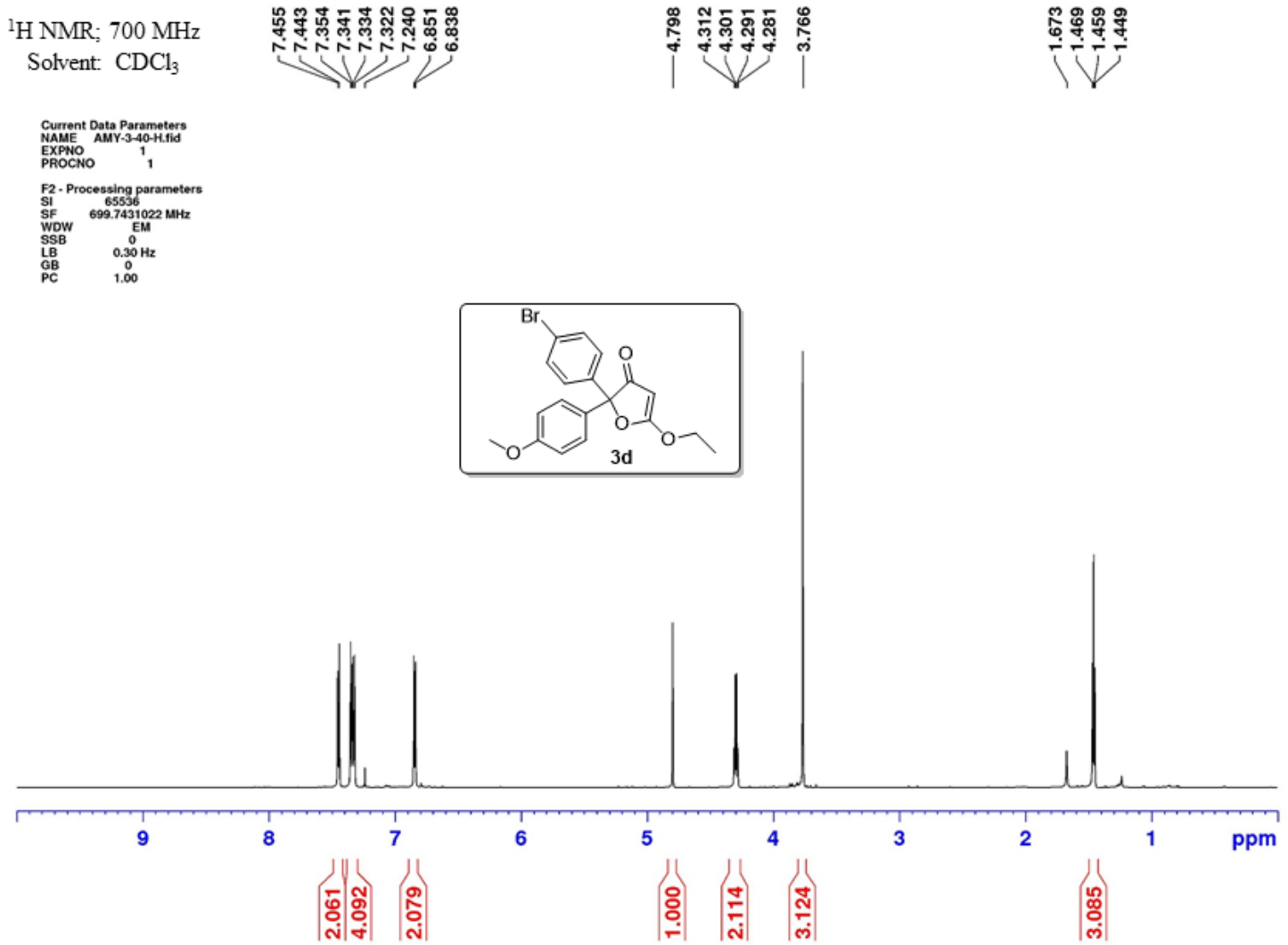
^{13}C NMR; 175 MHz
Solvent: $CDCl_3$



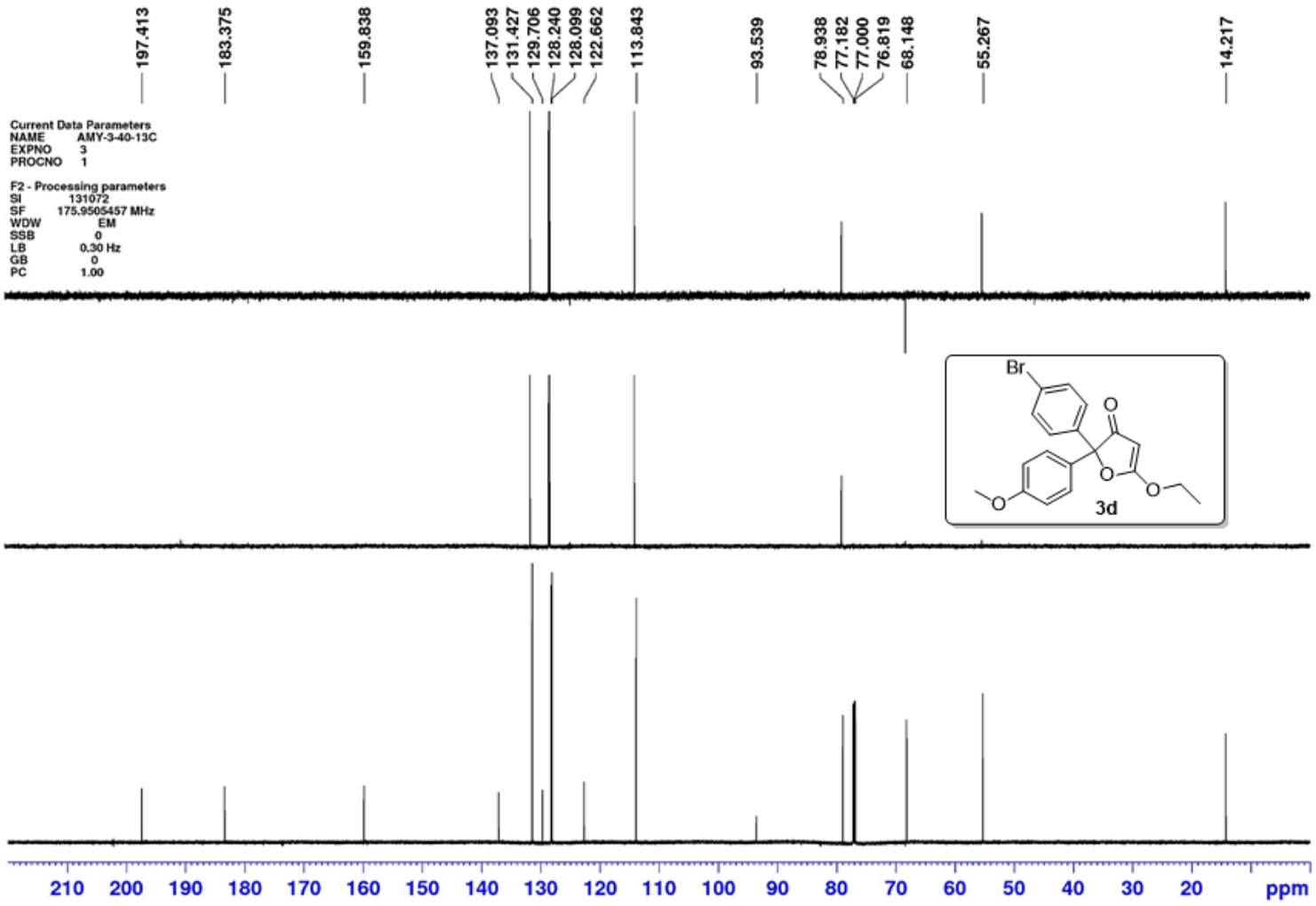

1H NMR; 700 MHz
Solvent: CDCl3
8.179
8.166
7.697
7.684
7.342
7.329
7.240
6.866
6.853
4.827
4.341
4.331
4.323
4.313
3.775
1.594
1.496
1.486
1.476
Current Data Parameters
NAME AMY-3-41-H.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7430996 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O2N
O
O
O
O
3c
9
8
7
6
5
4
3
2
1
ppm
2.000
2.014
2.004
2.067
1.009
2.043
3.005
3.018

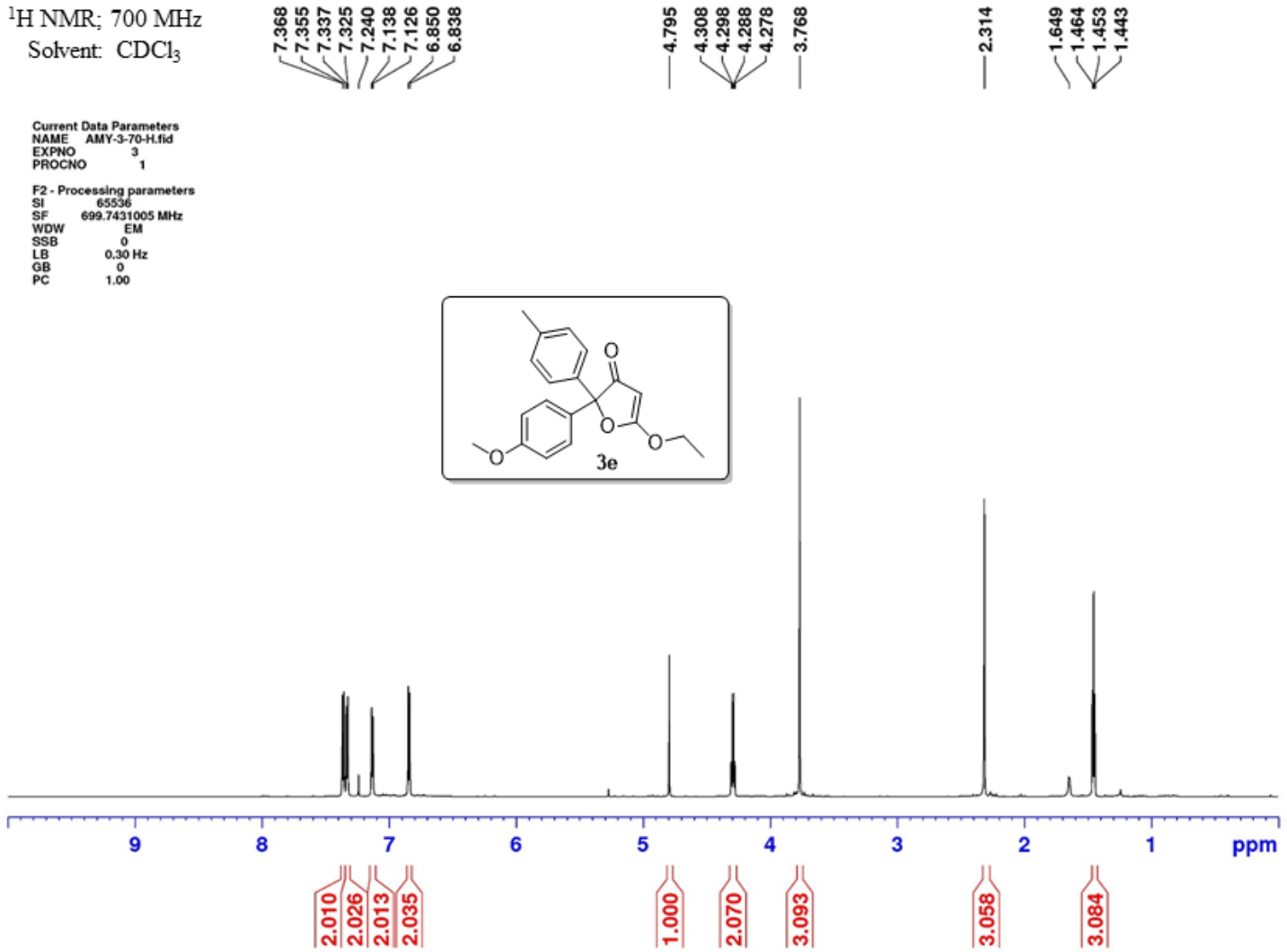
^{13}C NMR; 175 MHz
Solvent: $CDCl_3$

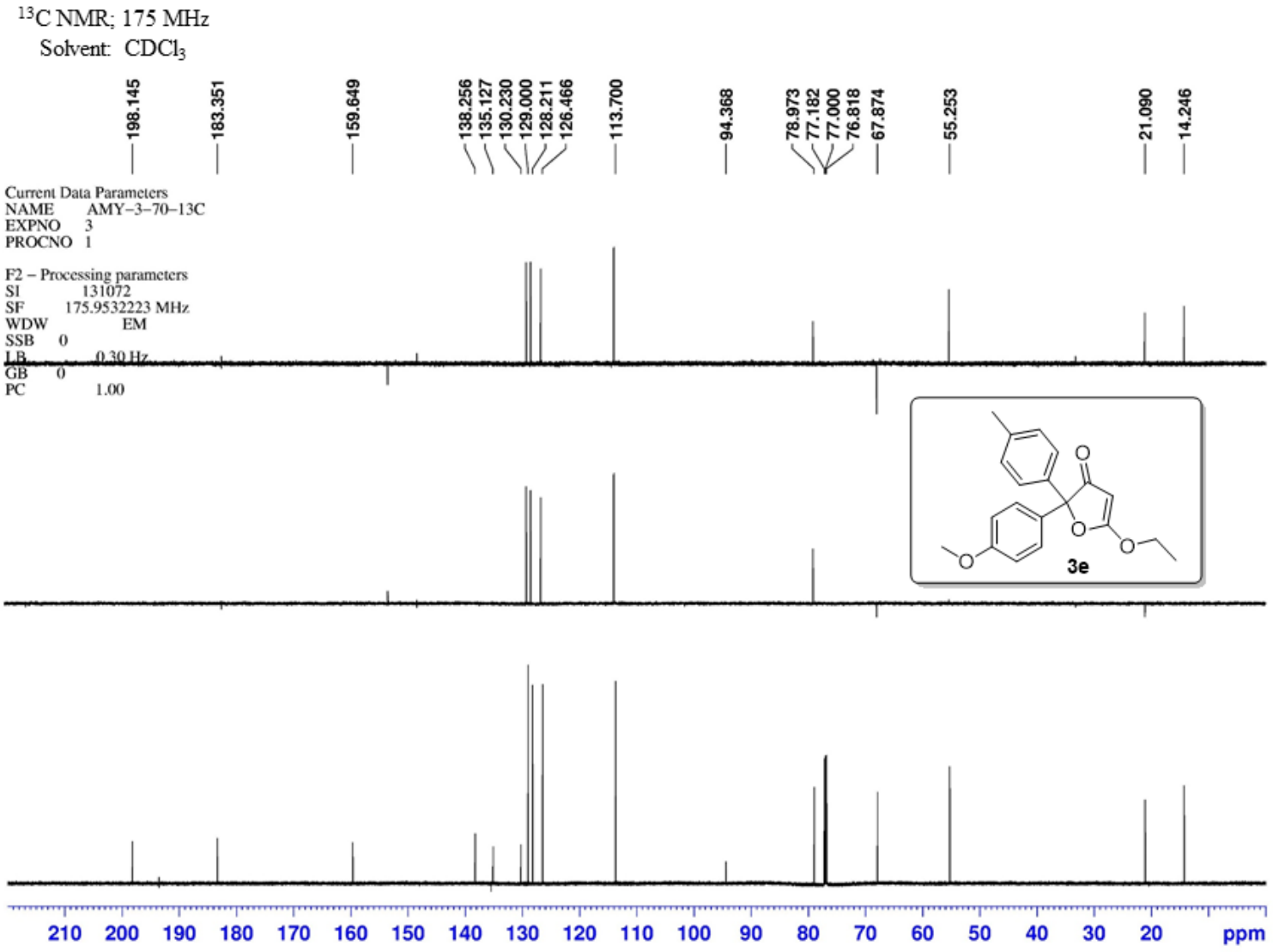



^{1}H NMR; 700 MHz
Solvent: $CDCl_3$
7.455
7.443
7.354
7.341
7.334
7.322
7.240
6.851
6.838
4.798
4.312
4.301
4.291
4.281
3.766
1.673
1.469
1.459
1.449
Current Data Parameters
NAME AMY-3-40-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7431022 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
O
O
O
O
3d
9
8
7
6
5
4
3
2
1
ppm
2.061
4.092
2.079
1.000
2.114
3.124
3.085

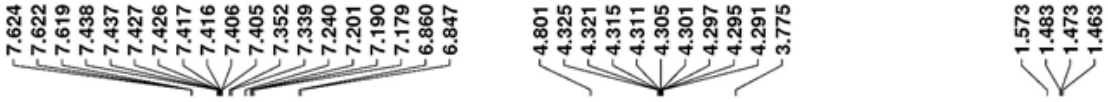
^{13}C NMR; 175 MHz
Solvent: $CDCl_3$




^{1}H NMR; 700 MHz
Solvent: $CDCl_3$
7.368
7.355
7.337
7.325
7.240
7.138
7.126
6.850
6.838
4.795
4.308
4.298
4.288
4.278
3.768
2.314
1.649
1.464
1.453
1.443
Current Data Parameters
NAME AMY-3-70-H.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7431005 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3e
9
8
7
6
5
4
3
2
1
ppm
2.010
2.026
2.013
2.035
1.000
2.070
3.093
3.058
3.084

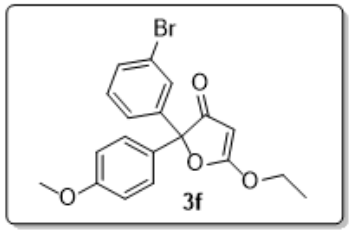

^{13}C NMR; 175 MHz
Solvent: $CDCl_3$
198.145
183.351
159.649
138.256
135.127
130.230
129.000
128.211
126.466
113.700
94.368
78.973
77.182
77.000
76.818
67.874
55.253
21.090
14.246
Current Data Parameters
NAME AMY-3-70-13C
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9532223 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3e
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

^{1}H NMR; 700 MHz
Solvent: $CDCl_3$

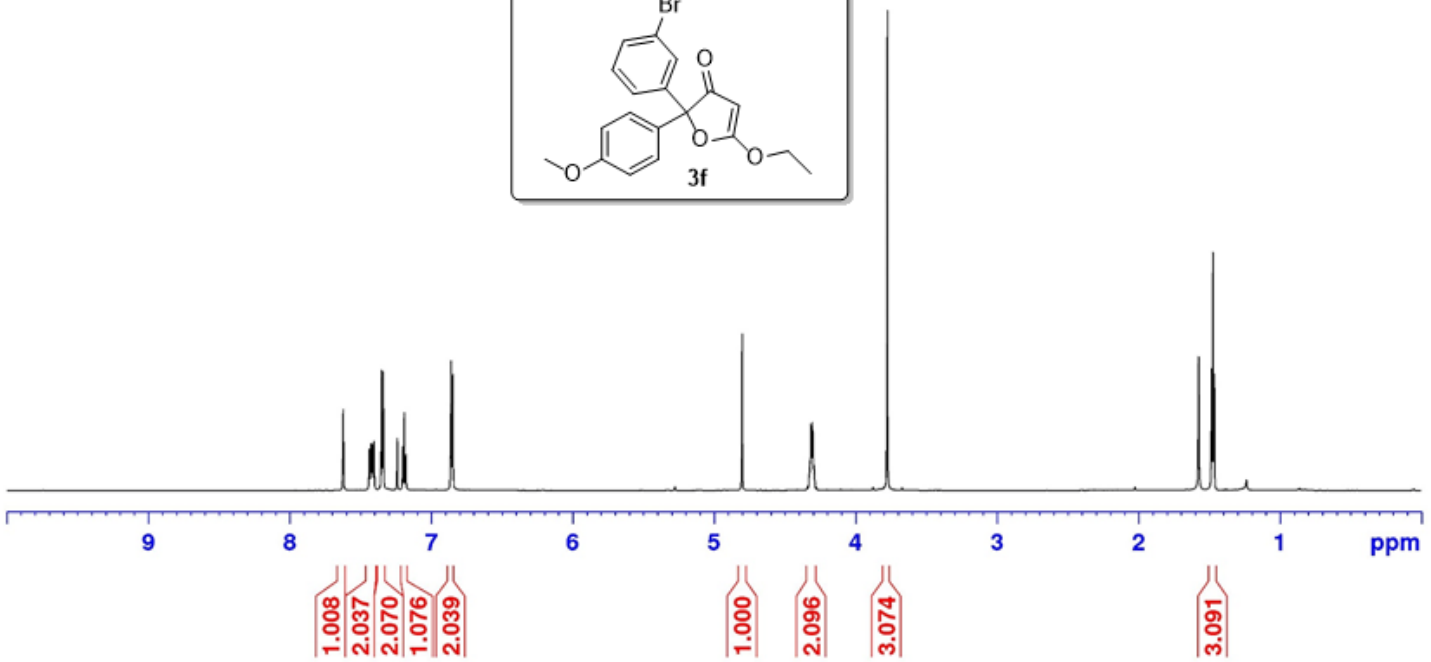


Current Data Parameters
NAME AMY-3-69-H.fid
EXPNO 3
PROCNO 1

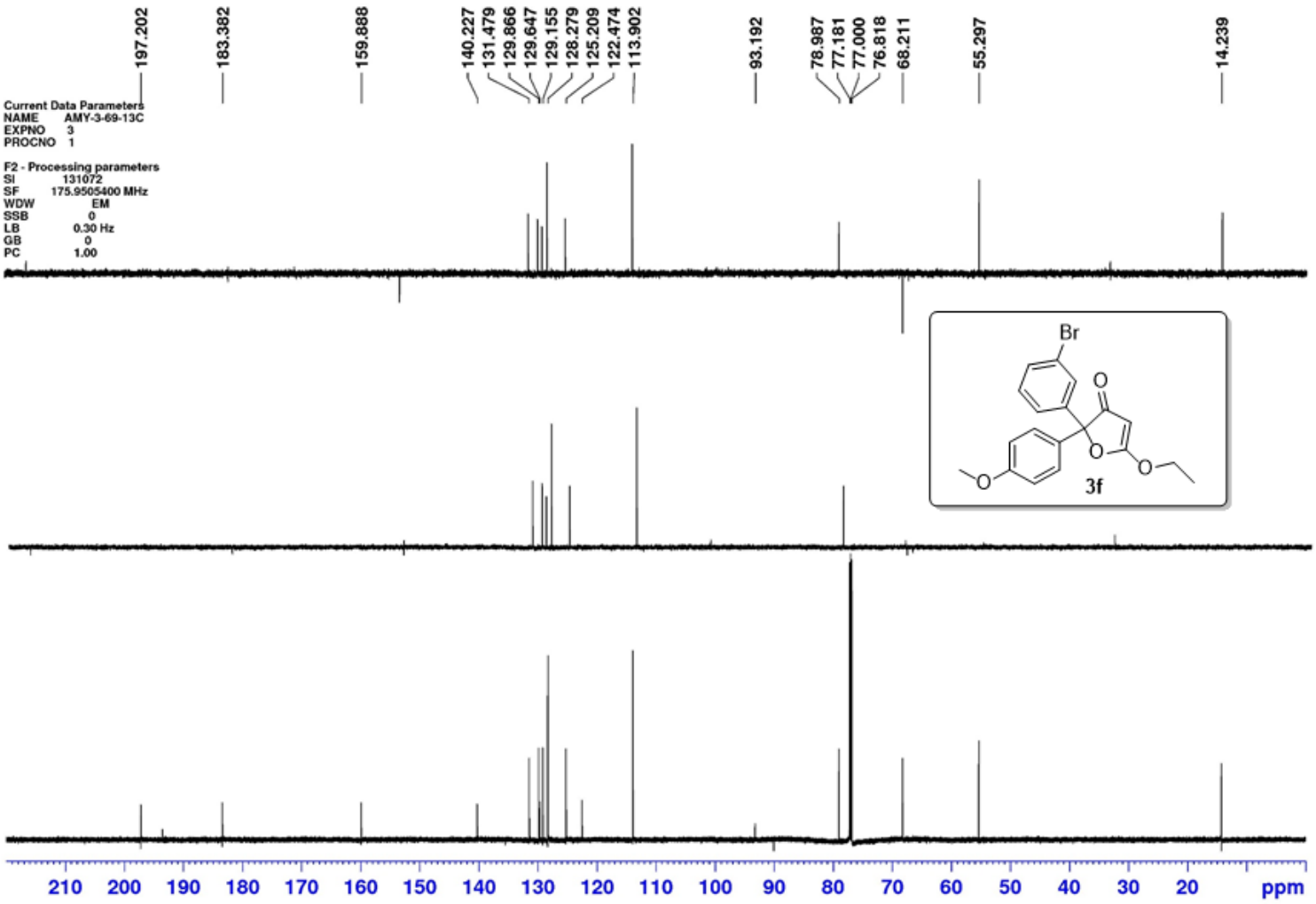
F2 - Processing parameters
SI 65536
SF 699.7431001 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



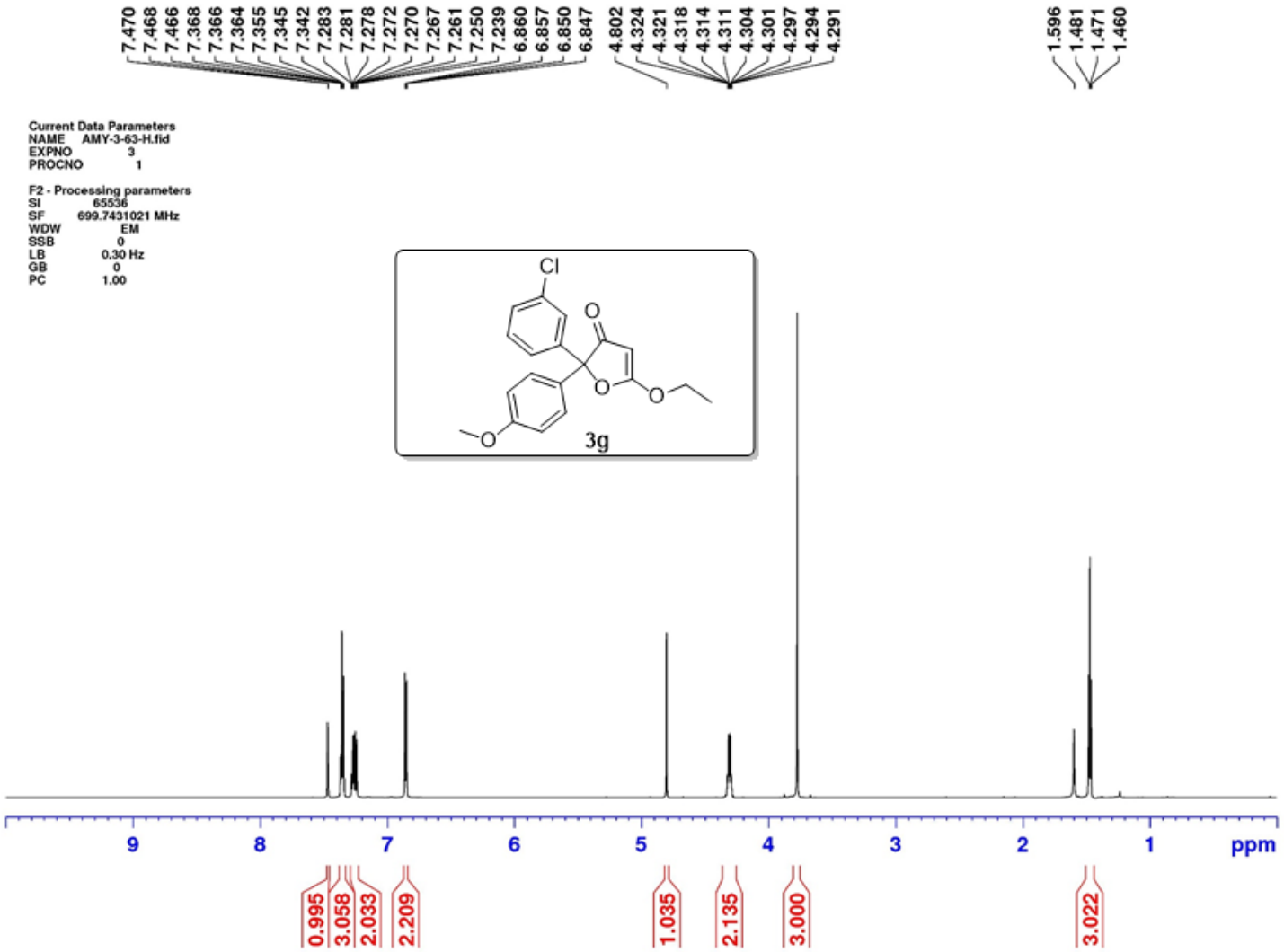
3f



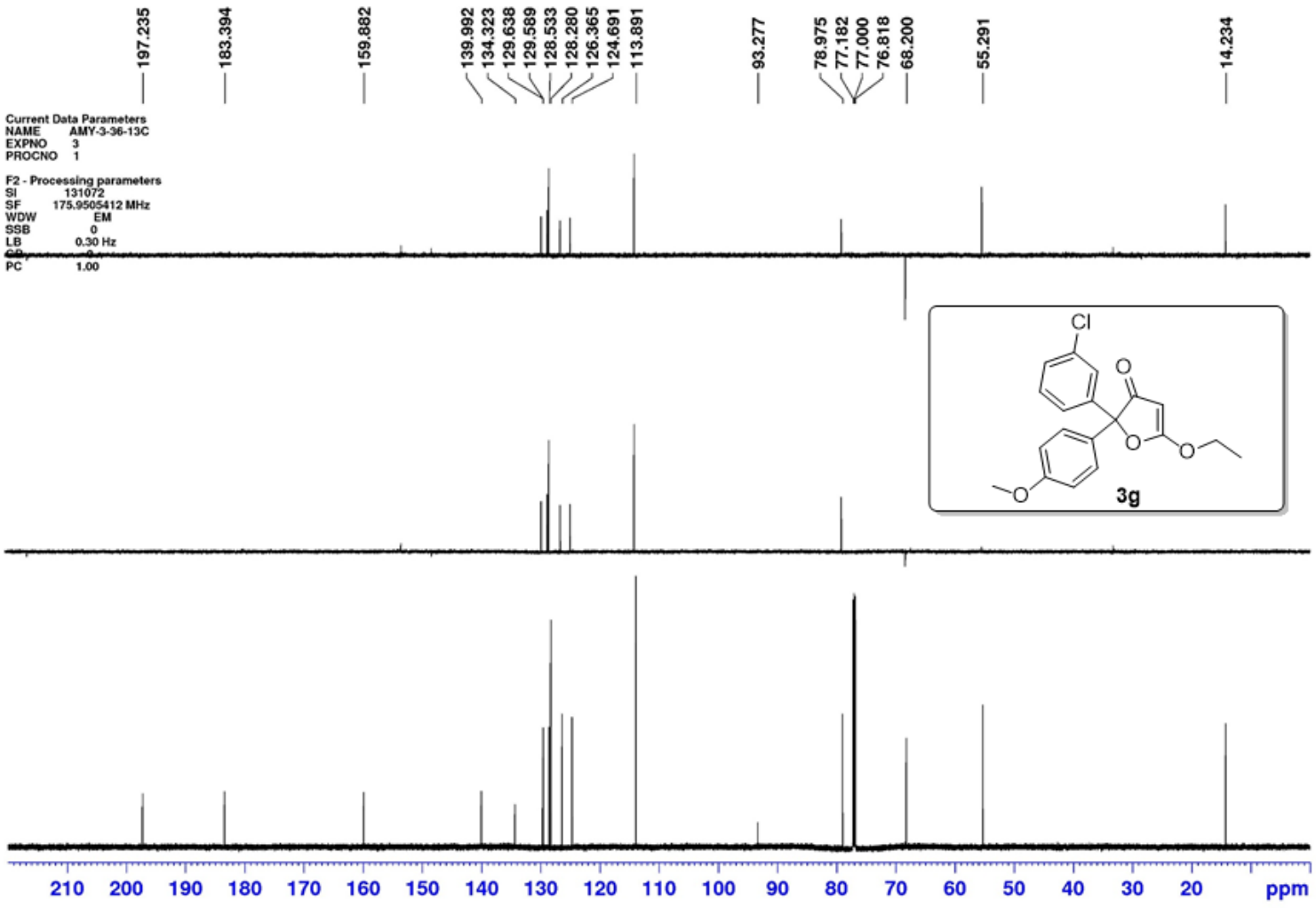
^{13}C NMR; 175 MHz
Solvent: $CDCl_3$

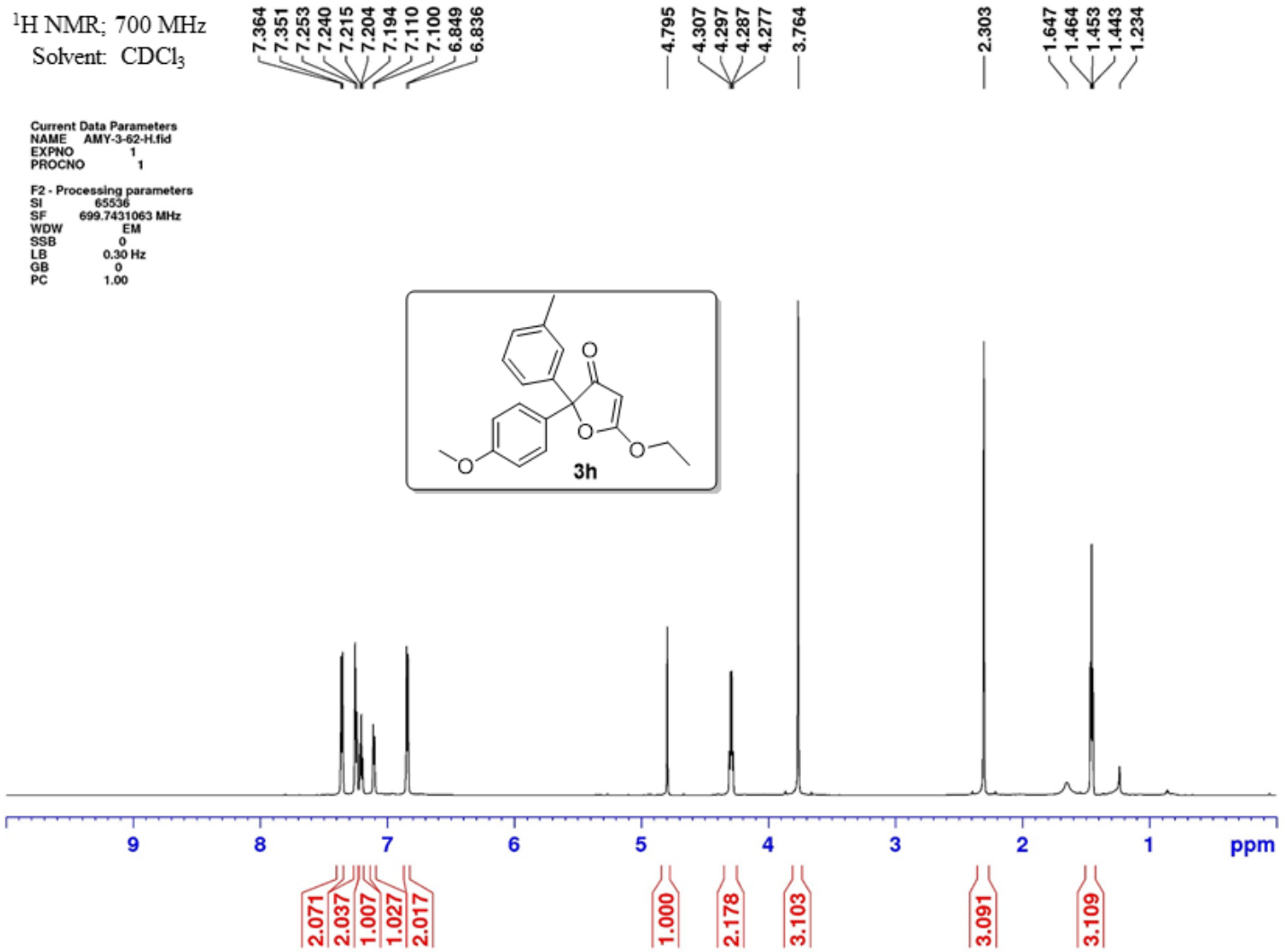


^{1}H NMR; 700 MHz
Solvent: $CDCl_3$

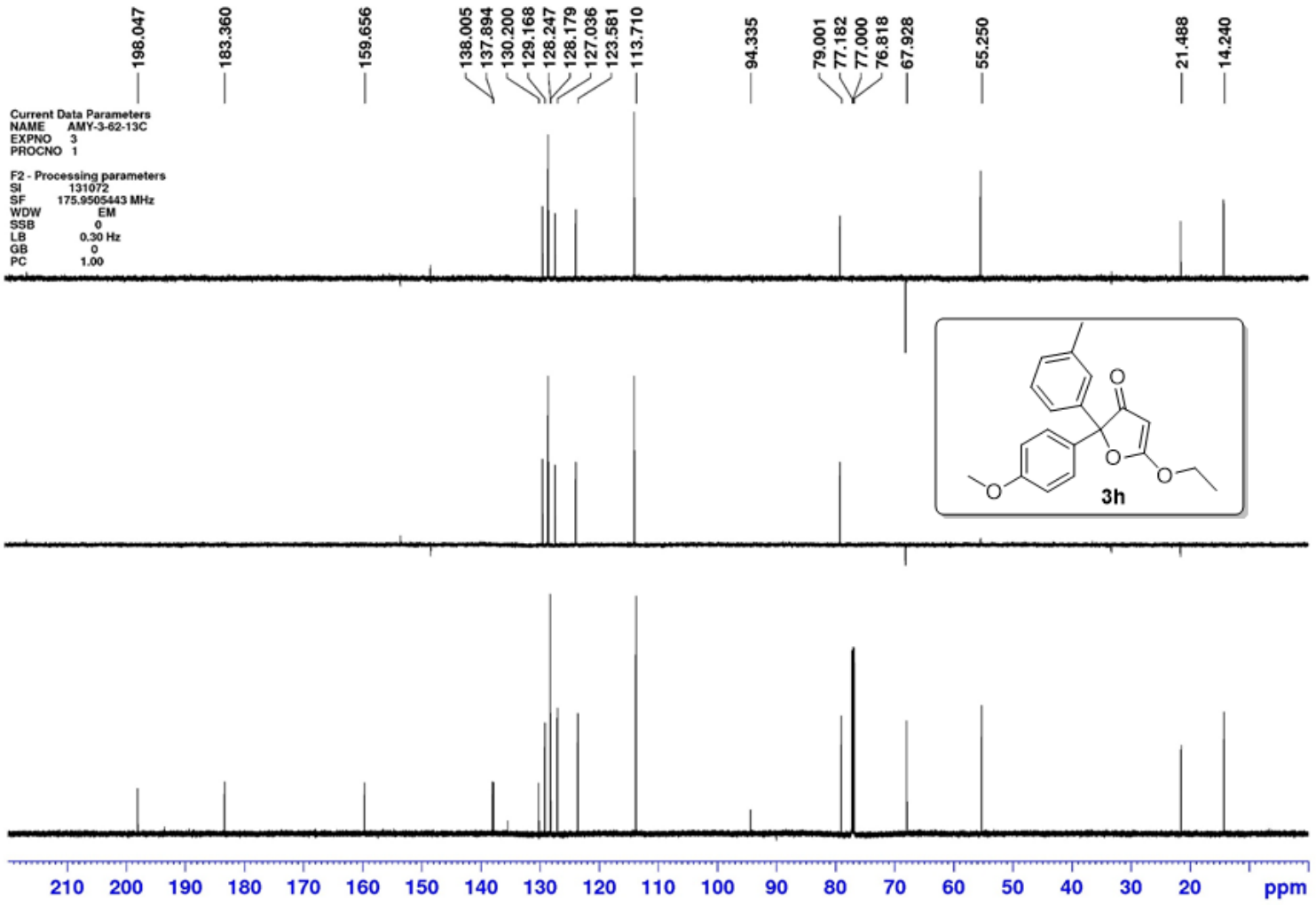


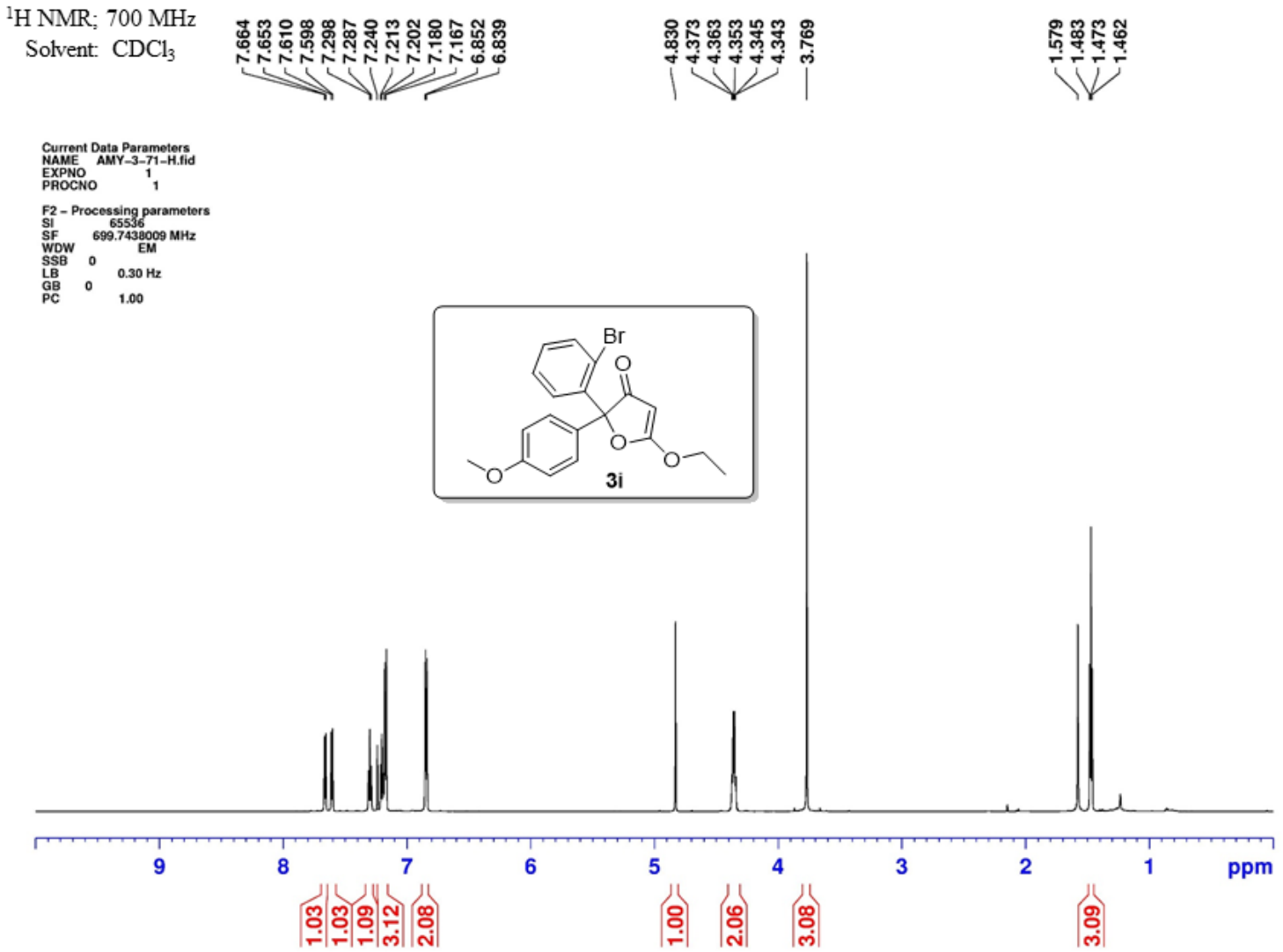
^{13}C NMR; 175 MHz
Solvent: $CDCl_3$

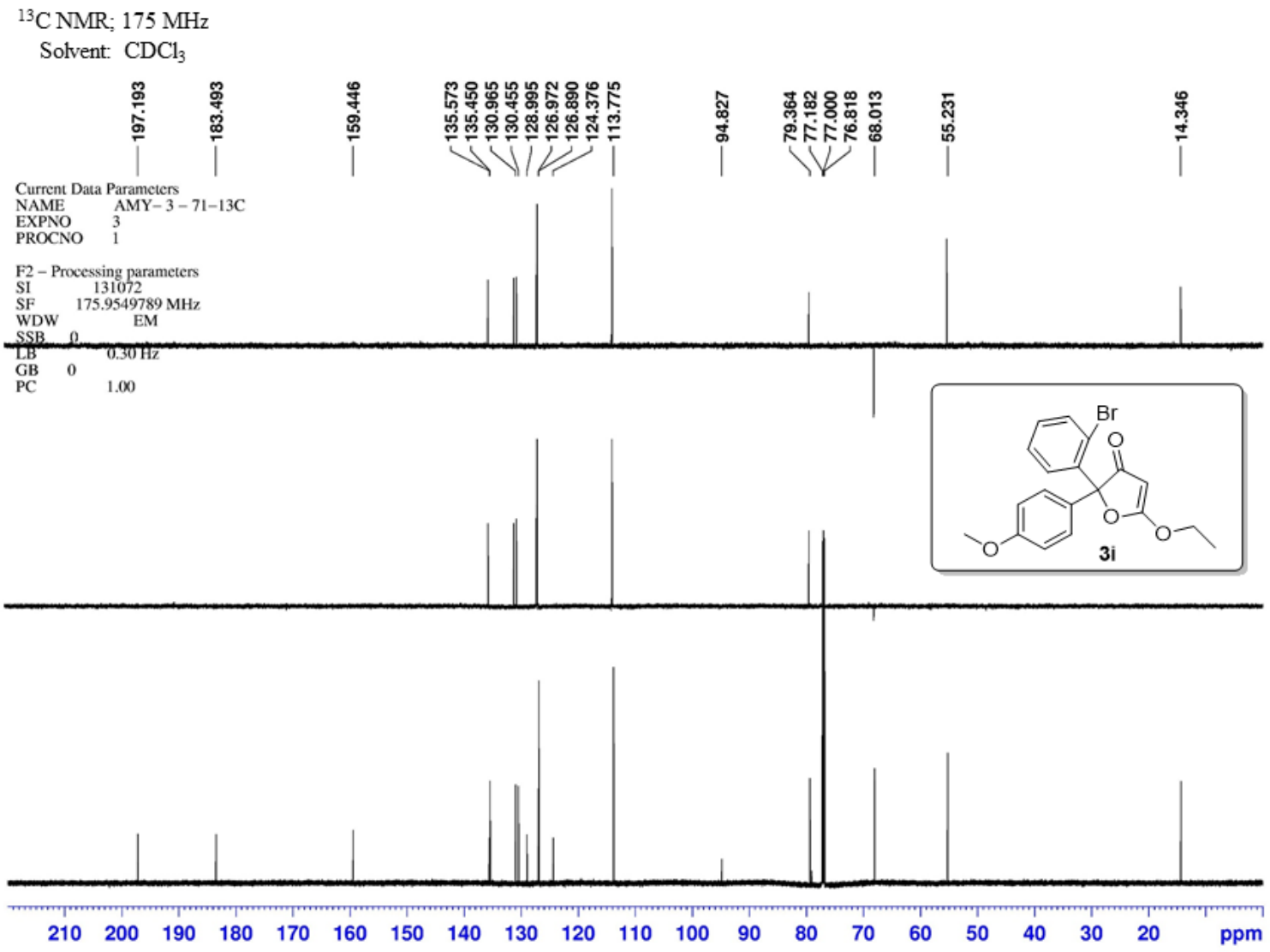



^{1}H NMR; 700 MHz
Solvent: $CDCl_3$
7.364
7.351
7.253
7.240
7.215
7.204
7.194
7.110
7.100
6.849
6.836
4.795
4.307
4.297
4.287
4.277
3.764
2.303
1.647
1.464
1.453
1.443
1.234
Current Data Parameters
NAME AMY-3-62-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7431063 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3h
9
8
7
6
5
4
3
2
1
ppm
2.071
2.037
1.007
1.027
2.017
1.000
2.178
3.103
3.091
3.109

^{13}C NMR; 175 MHz
Solvent: $CDCl_3$

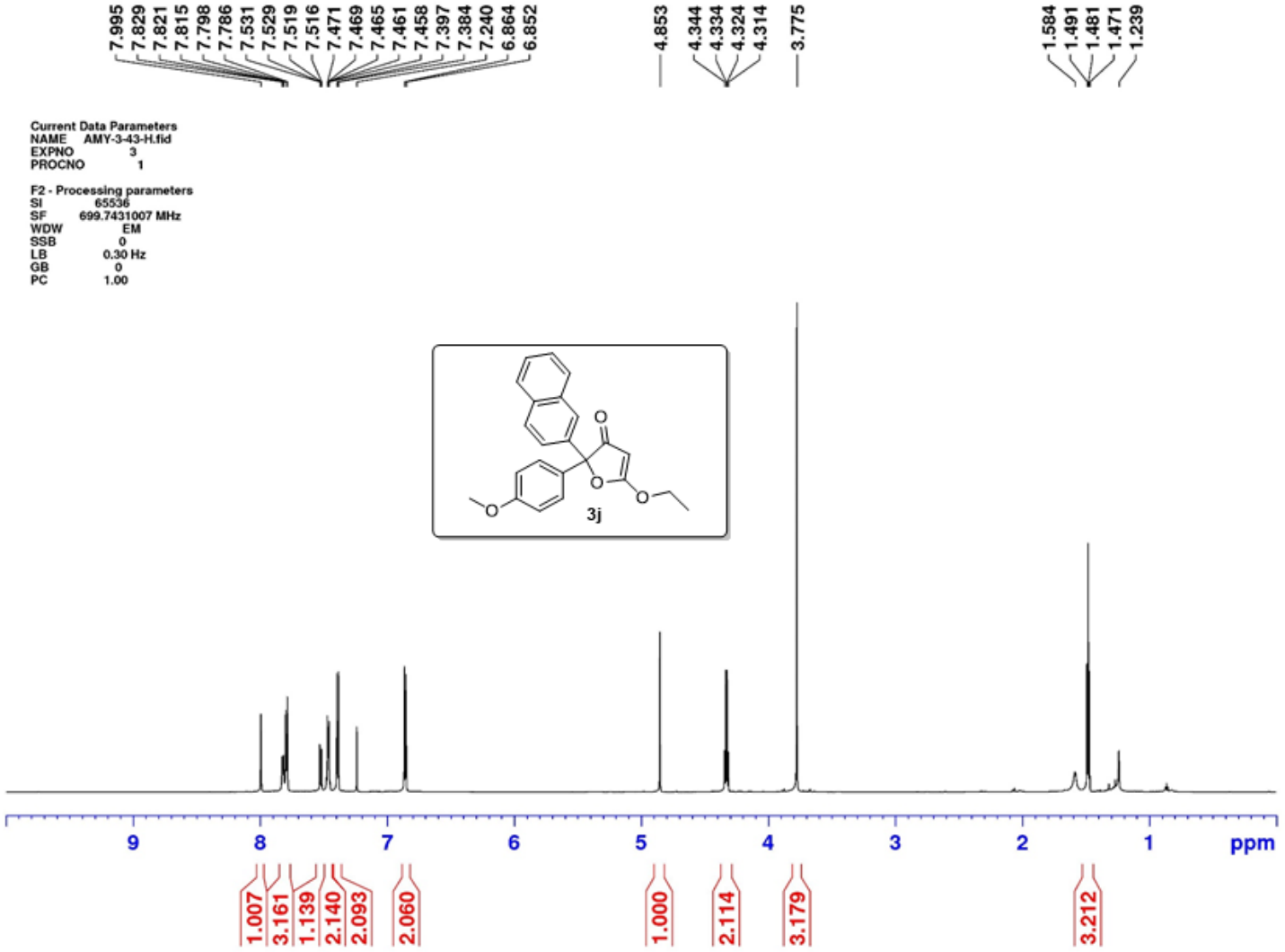



^{1}H NMR; 700 MHz
Solvent: $CDCl_3$
7.664
7.653
7.610
7.598
7.298
7.287
7.240
7.213
7.202
7.180
7.167
6.852
6.839
4.830
4.373
4.363
4.353
4.345
4.343
3.769
1.579
1.483
1.473
1.462
Current Data Parameters
NAME AMY-3-71-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7438009 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
O
O
O
O
3i
9
8
7
6
5
4
3
2
1
ppm
1.03
1.03
1.09
3.12
2.08
1.00
2.06
3.08
3.09

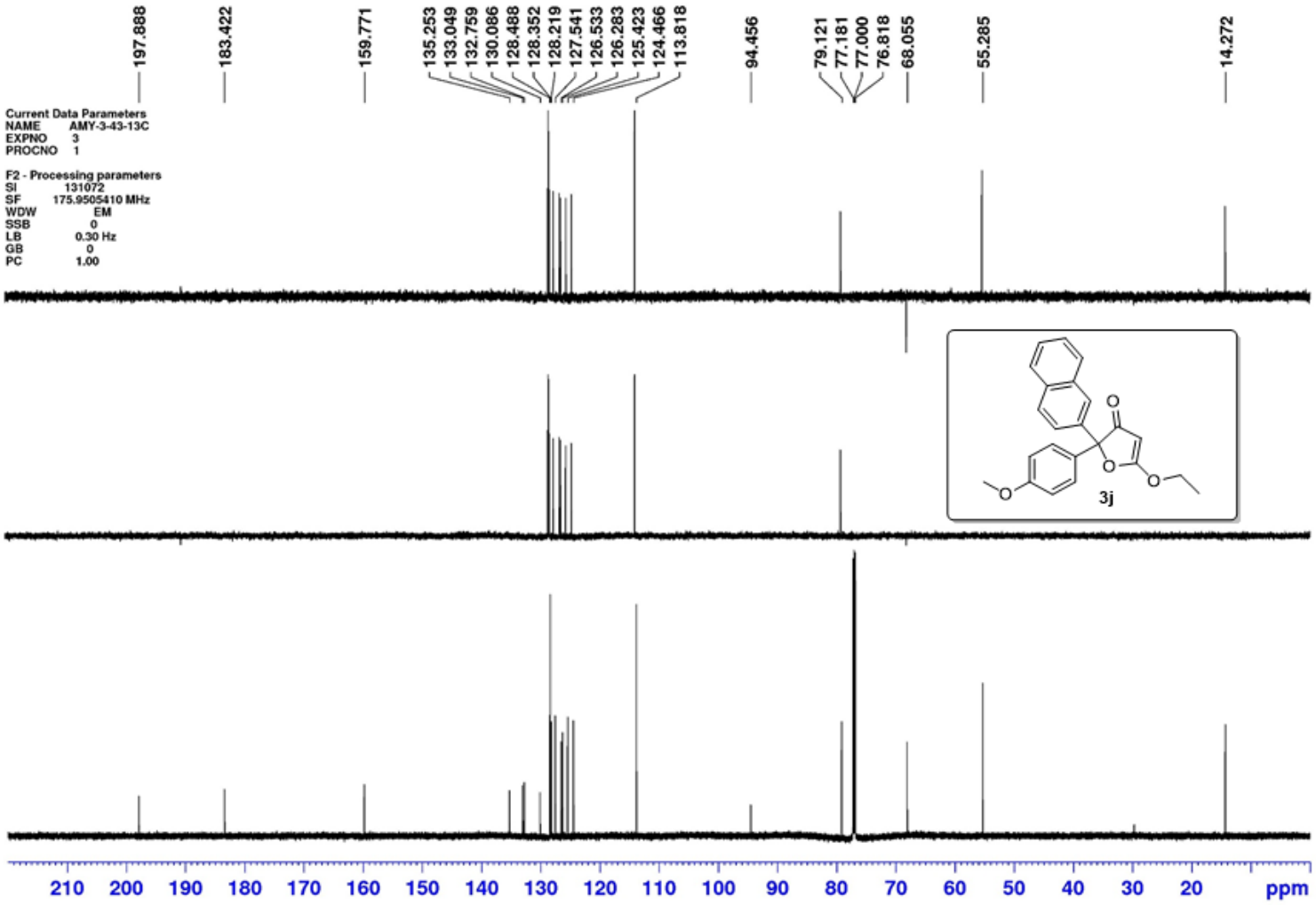

^{13}C NMR; 175 MHz
Solvent: $CDCl_3$
197.193
183.493
159.446
135.573
135.450
130.965
130.455
128.995
126.972
126.890
124.376
113.775
94.827
79.364
77.182
77.000
76.818
68.013
55.231
14.346
Current Data Parameters
NAME AMY– 3 – 71–13C
EXPNO 3
PROCNO 1
F2 – Processing parameters
SI 131072
SF 175.9549789 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
O
O
O
O
3i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

^{1}H NMR; 700 MHz

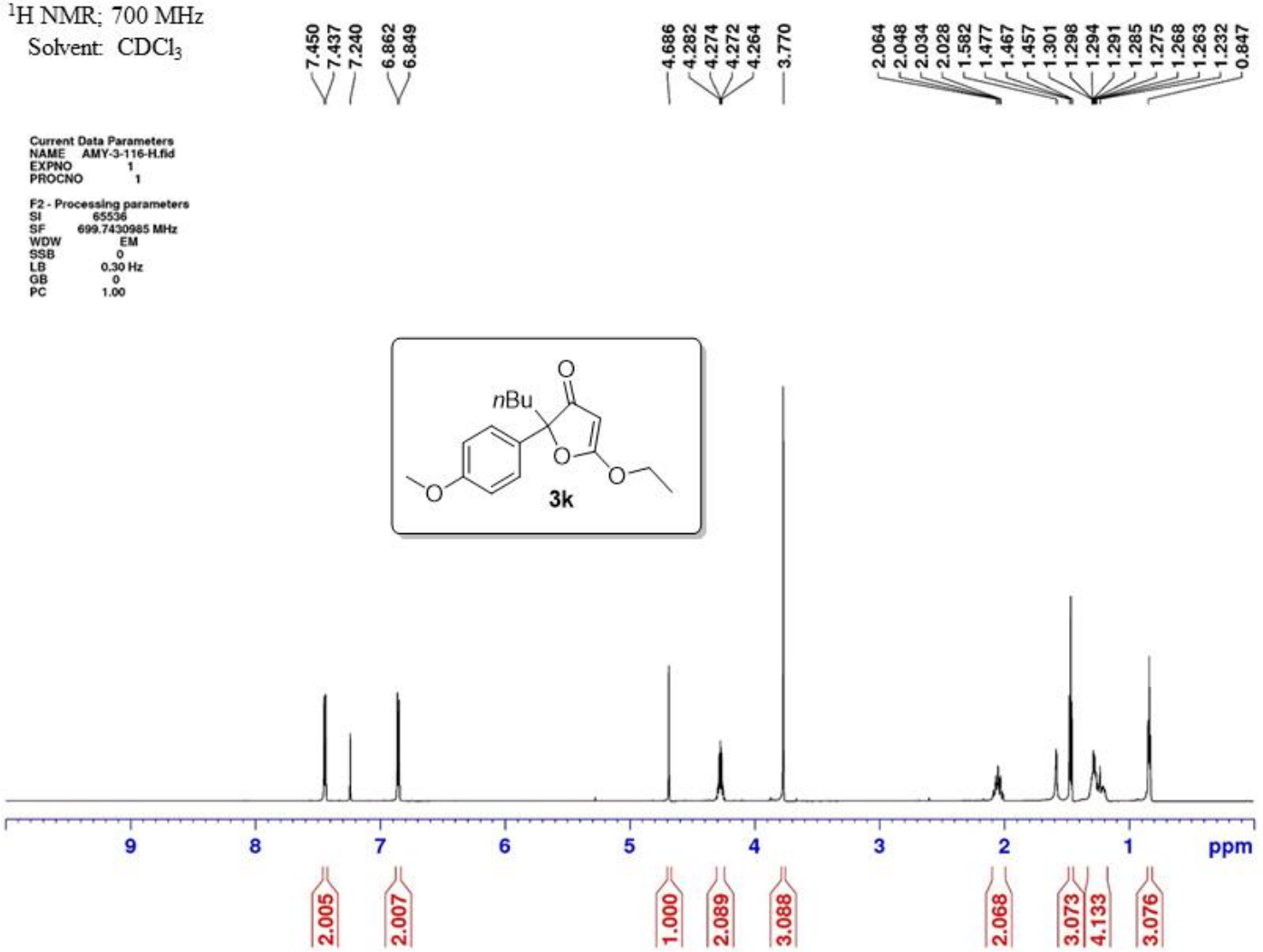
Solvent: $CDCl_3$



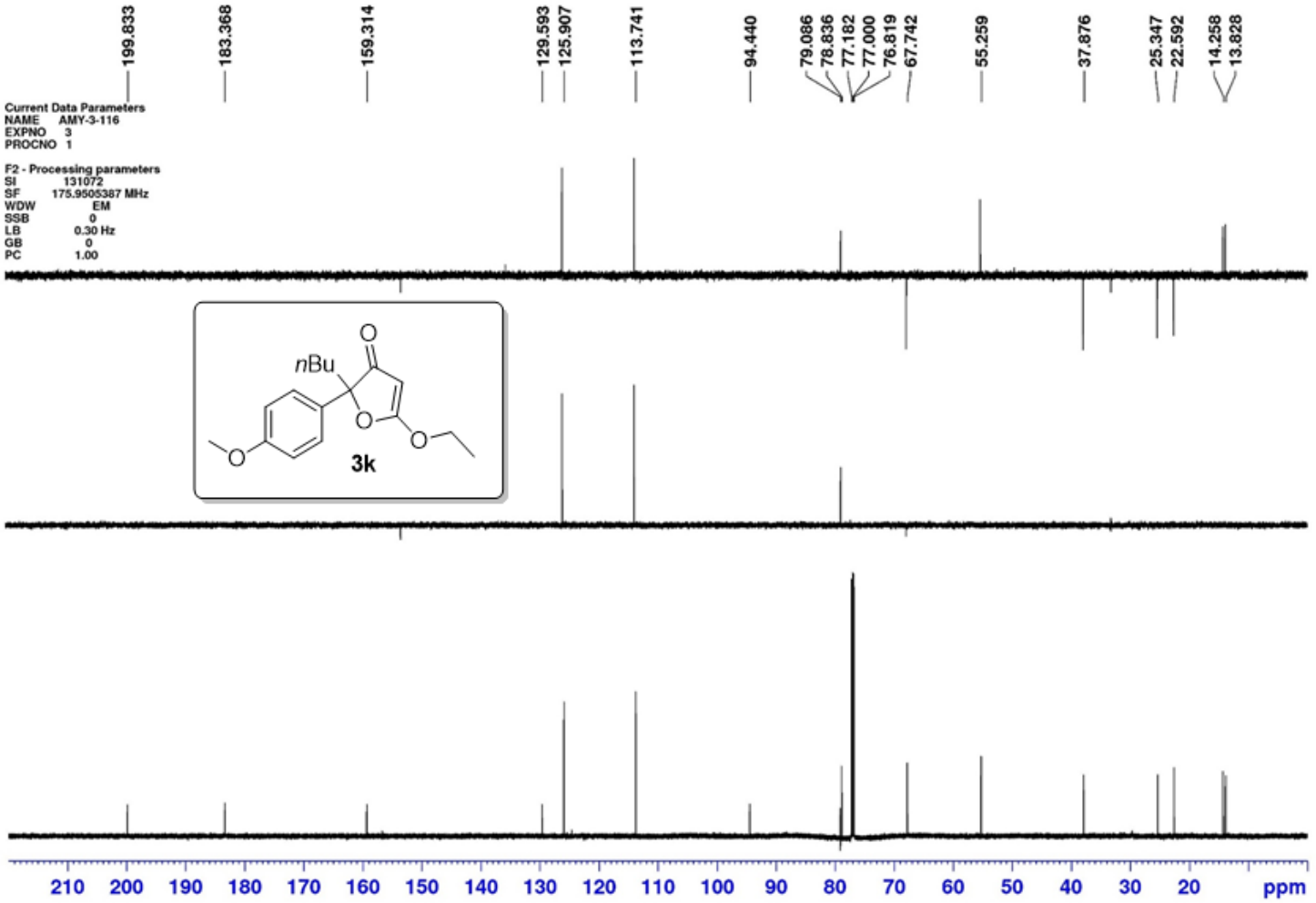
^{13}C NMR; 175 MHz
Solvent: $CDCl_3$



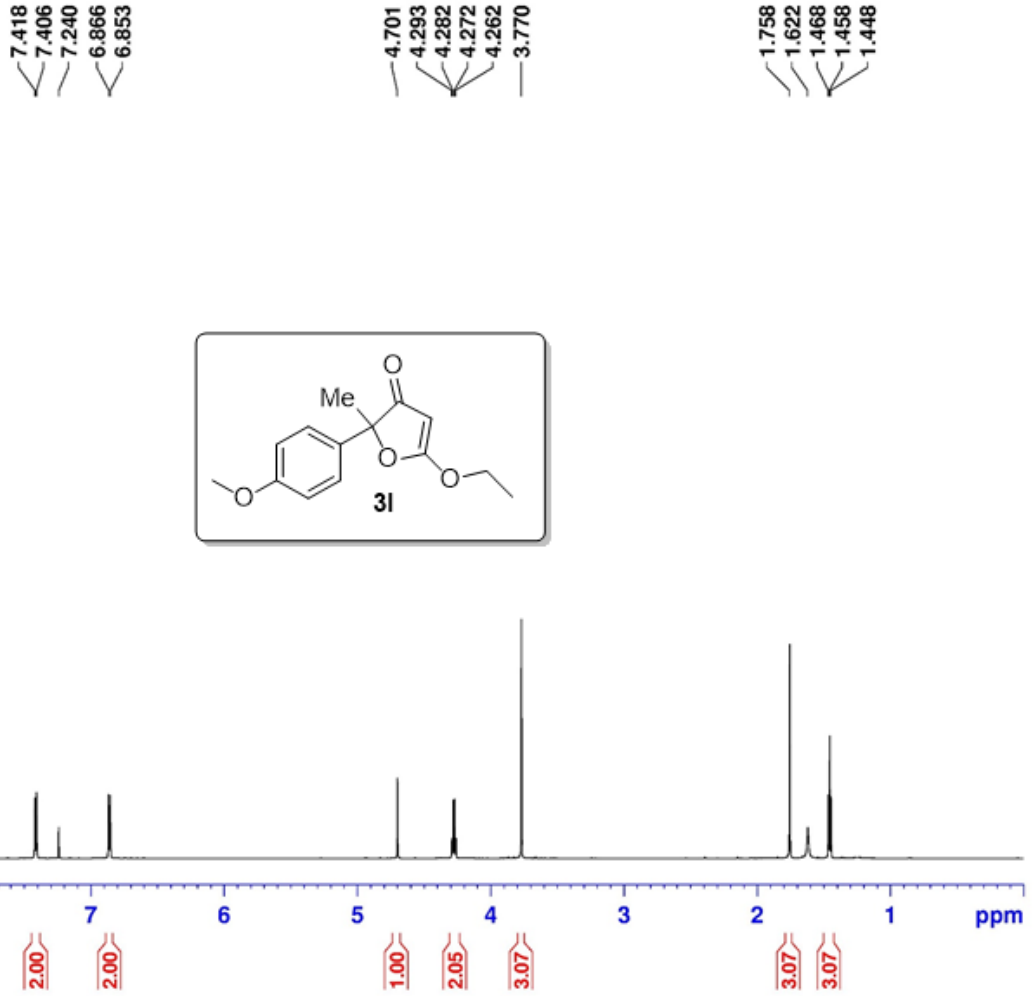
^{1}H NMR; 700 MHz
Solvent: $CDCl_3$



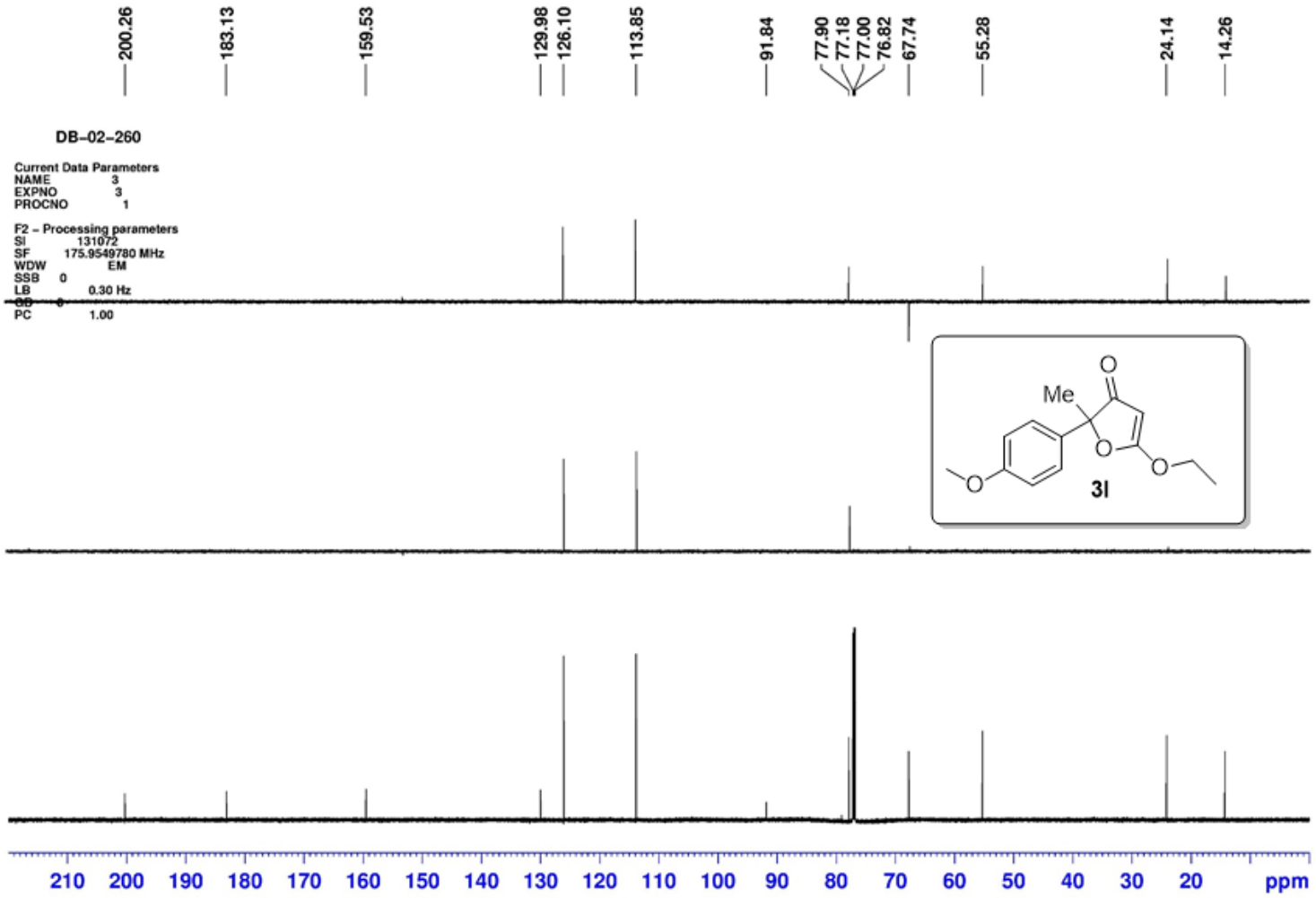
^{13}C NMR; 175 MHz
Solvent: $CDCl_3$

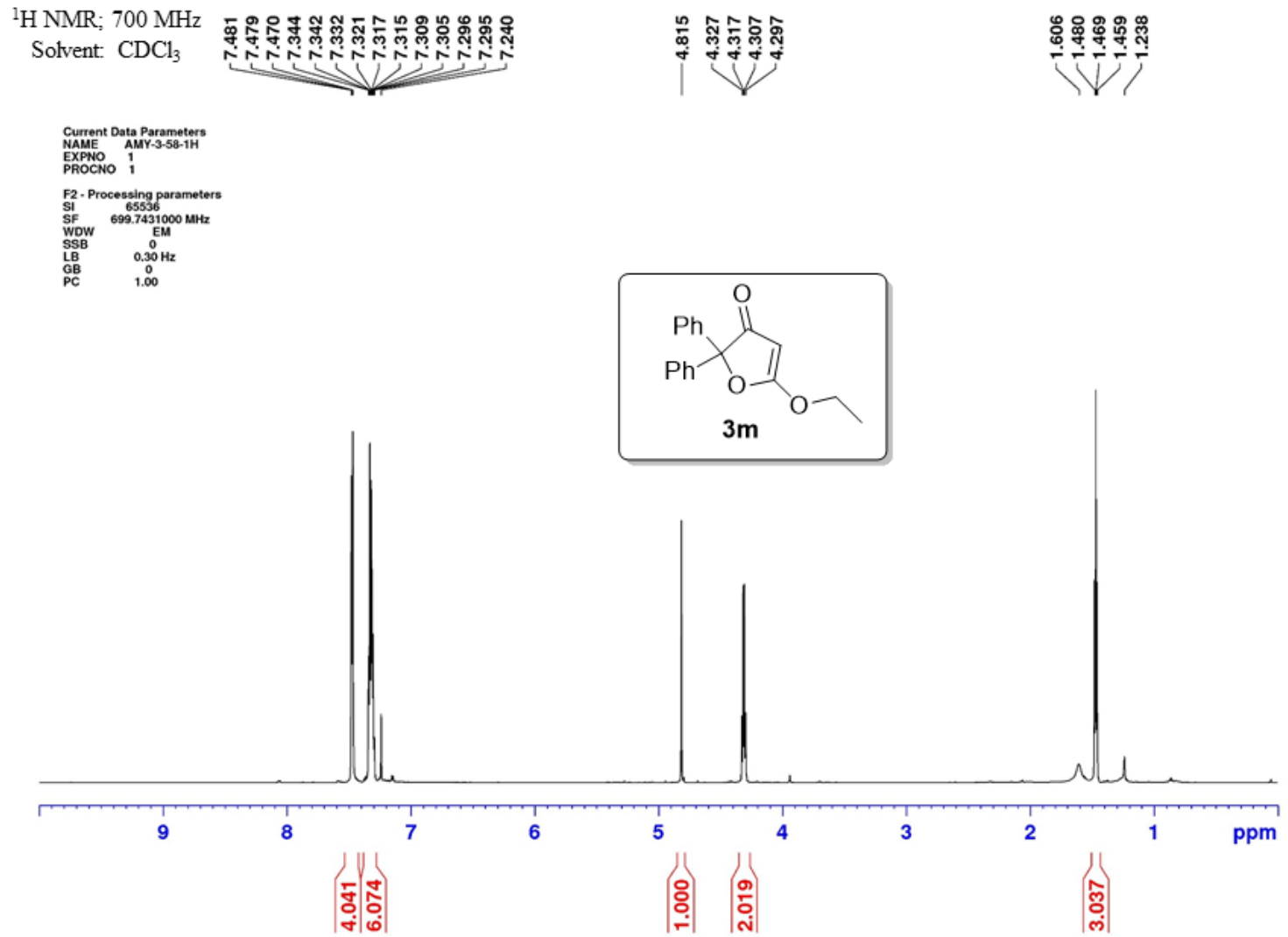


^{1}H NMR; 700 MHz
Solvent: $CDCl_3$

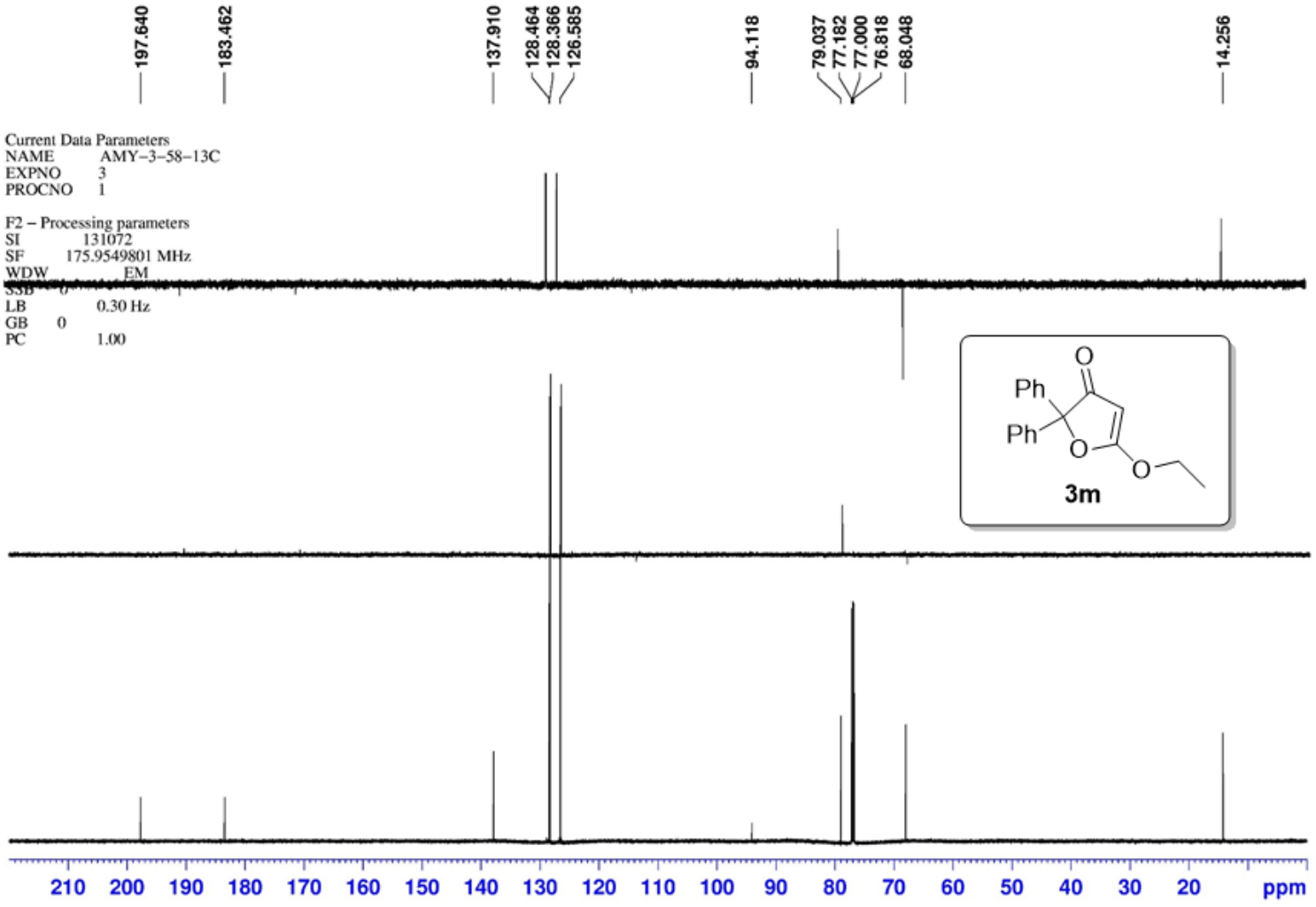


^{13}C NMR; 175 MHz
Solvent: $CDCl_3$

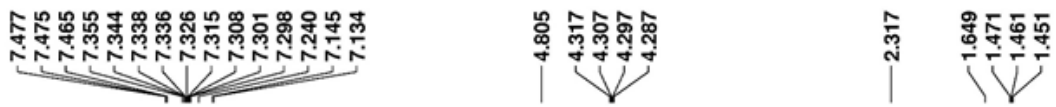



^{1}H NMR; 700 MHz
Solvent: $CDCl_3$
7.481
7.479
7.470
7.344
7.342
7.332
7.321
7.317
7.315
7.309
7.305
7.296
7.295
7.240
4.815
4.327
4.317
4.307
4.297
1.606
1.480
1.469
1.459
1.238
Current Data Parameters
NAME AMY-3-58-1H
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7431000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
Ph
O
O
O
3m
9
8
7
6
5
4
3
2
1
ppm
4.041
6.074
1.000
2.019
3.037

^{13}C NMR; 175 MHz
Solvent: $CDCl_3$

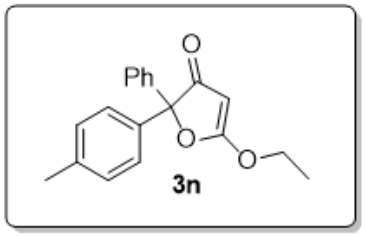


^{1}H NMR; 700 MHz
Solvent: $CDCl_3$

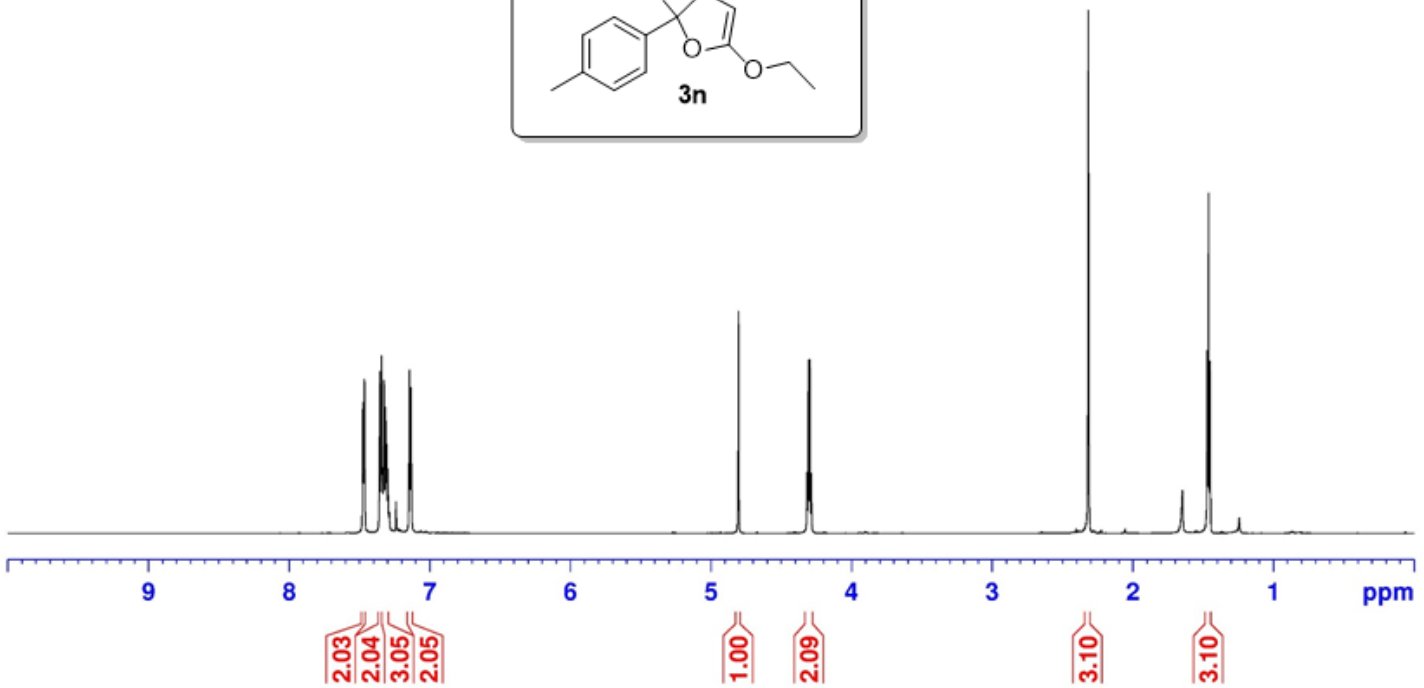


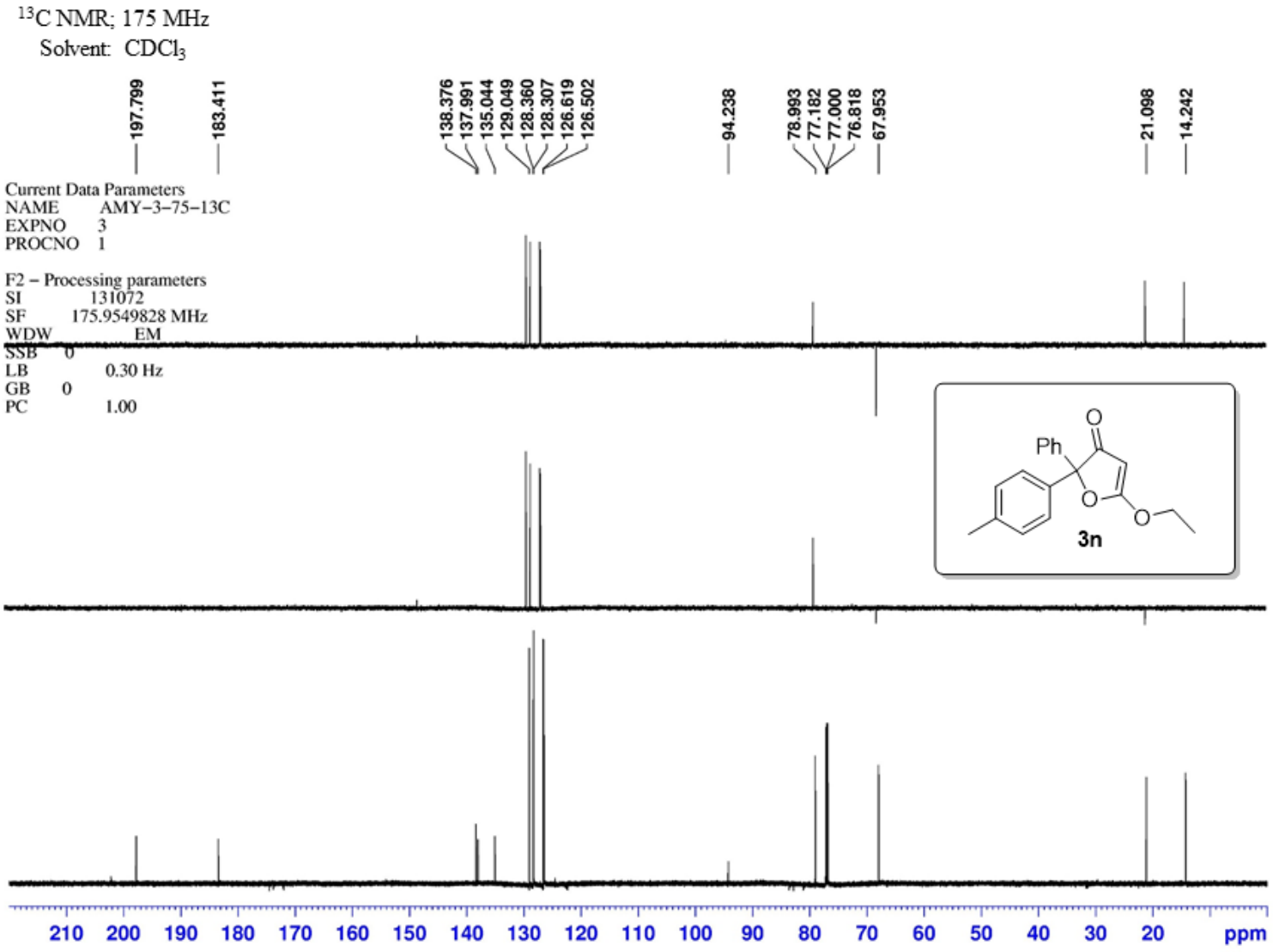
Current Data Parameters
NAME AMY-3-75-H.fid
EXPNO 1
PROCNO 1

F2 – Processing parameters
SI 65536
SF 699.7441505 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3n




^{13}C NMR; 175 MHz
Solvent: $CDCl_3$
197.799
183.411
138.376
137.991
135.044
129.049
128.360
128.307
126.619
126.502
94.238
78.993
77.182
77.000
76.818
67.953
21.098
14.242
Current Data Parameters
NAME AMY-3-75-13C
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9549828 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
O
O
O
3n
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

^{1}H NMR; 700 MHz
Solvent: $CDCl_3$

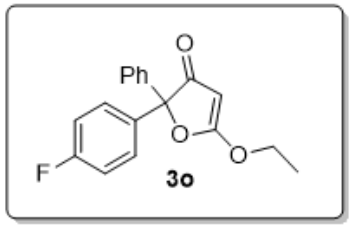
7.480 7.477 7.466 7.458 7.453 7.445 7.442 7.344 7.335 7.323 7.321 7.311 7.303 7.240 7.025 7.013 7.001

4.814 4.325 4.315 4.305 4.295

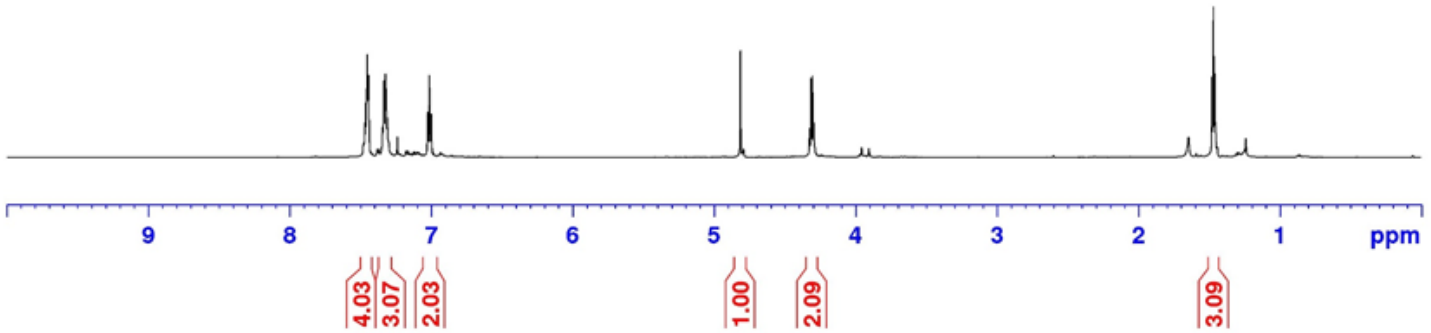
1.645 1.480 1.470 1.460

Current Data Parameters
NAME AMY-3-76-H.fid
EXPNO 1
PROCNO 1

F2 - Processing parameters
SI 65536
SF 699.7431012 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

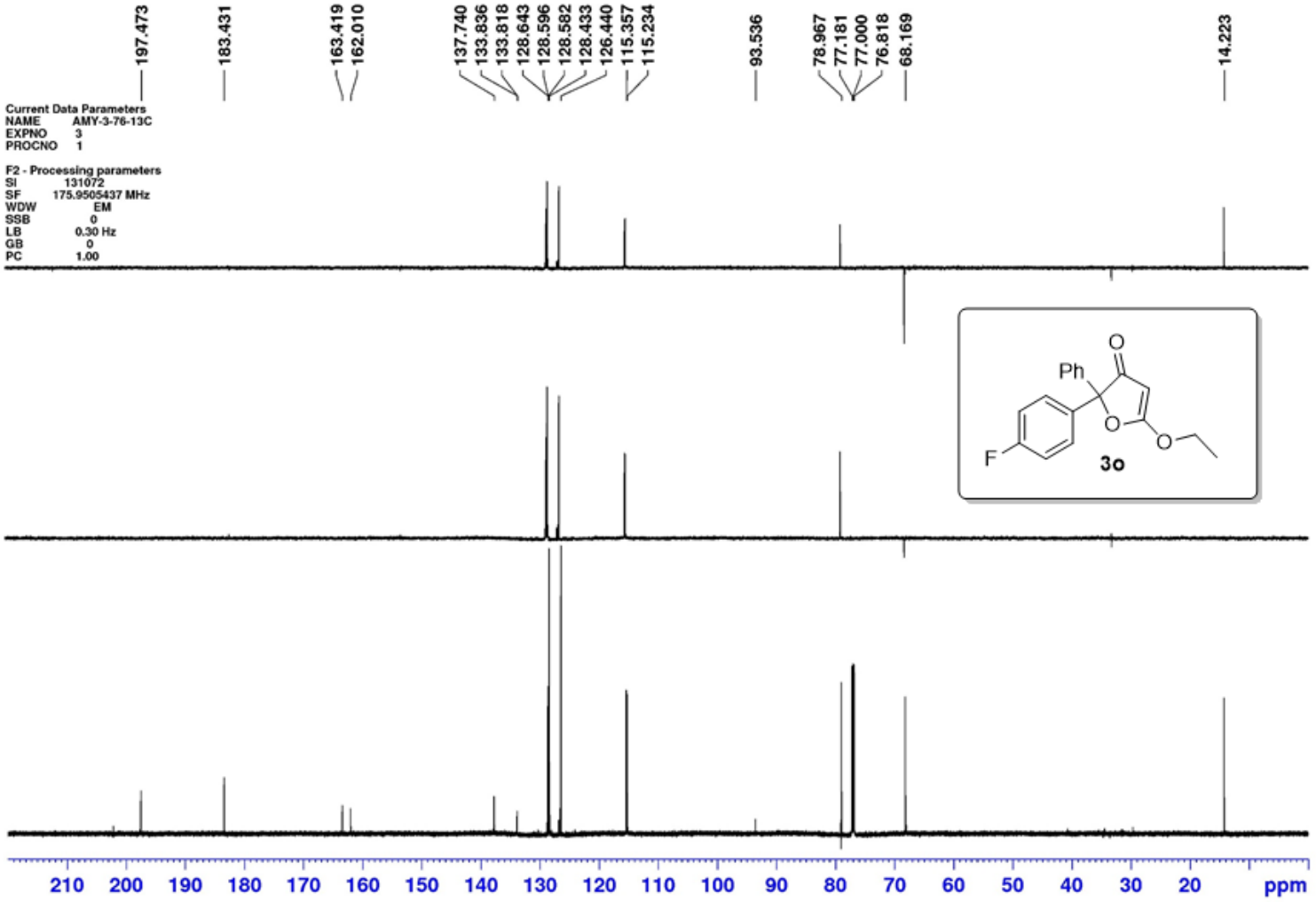


3o

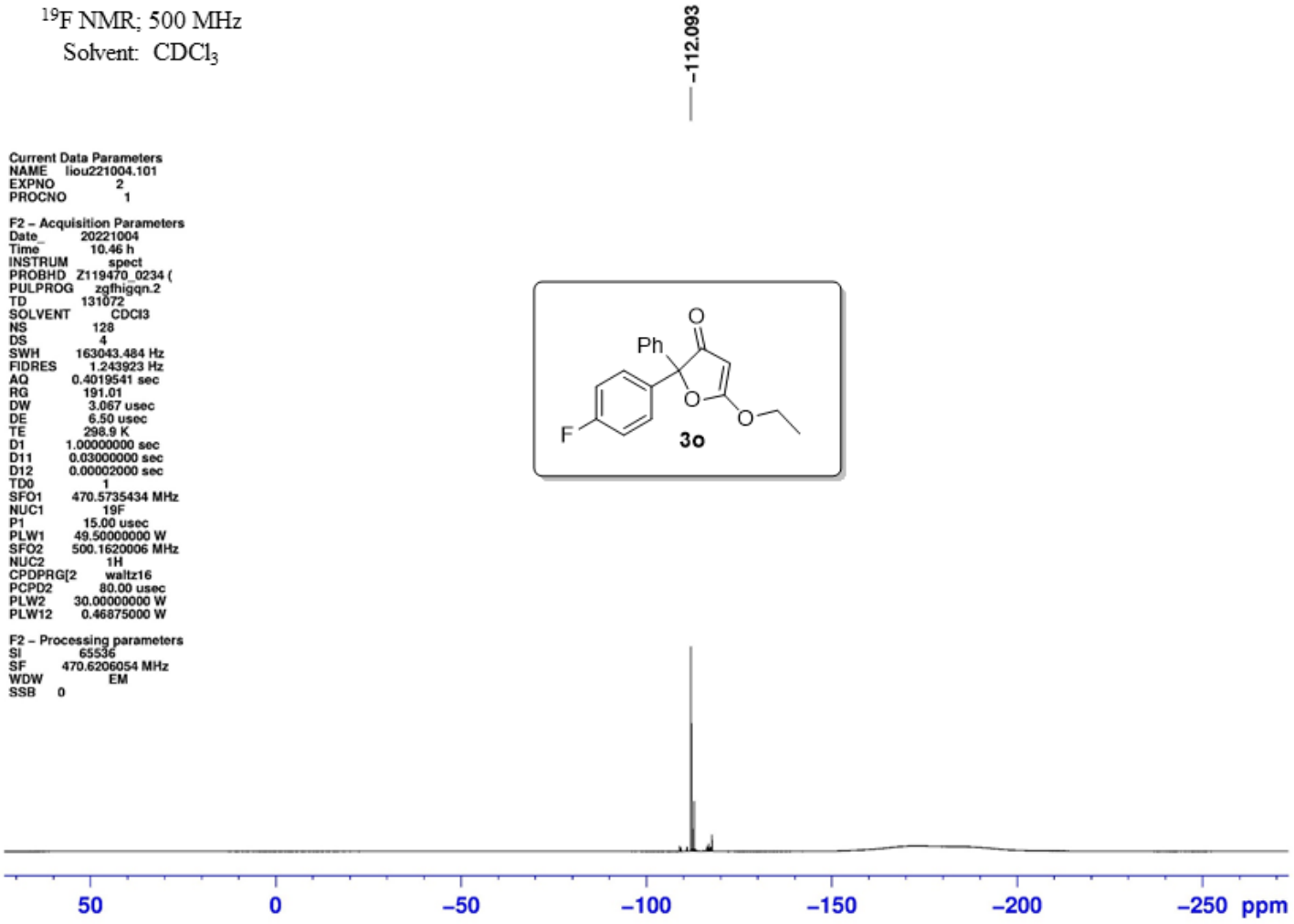


^{13}C NMR; 175 MHz

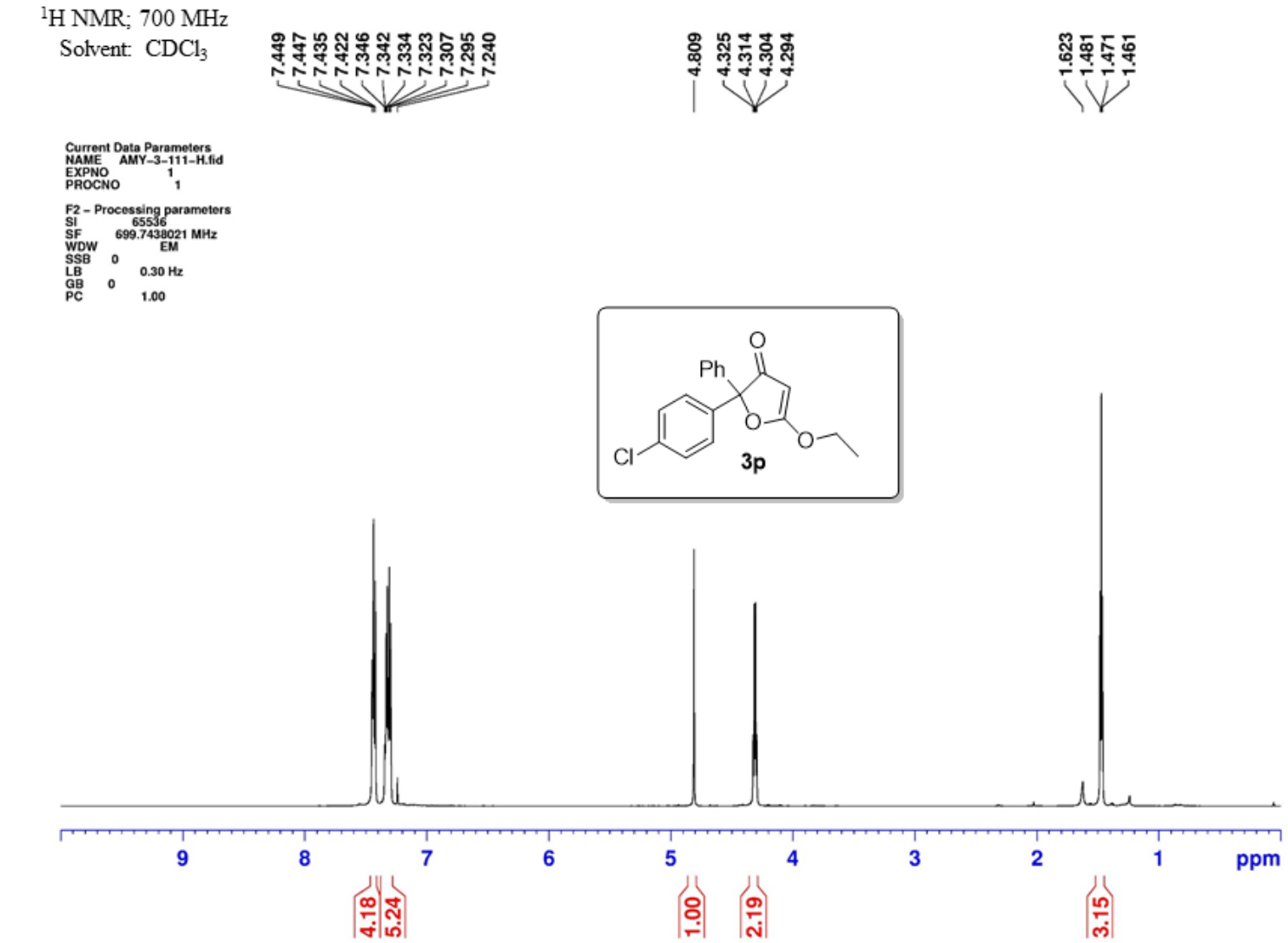
Solvent: $CDCl_3$



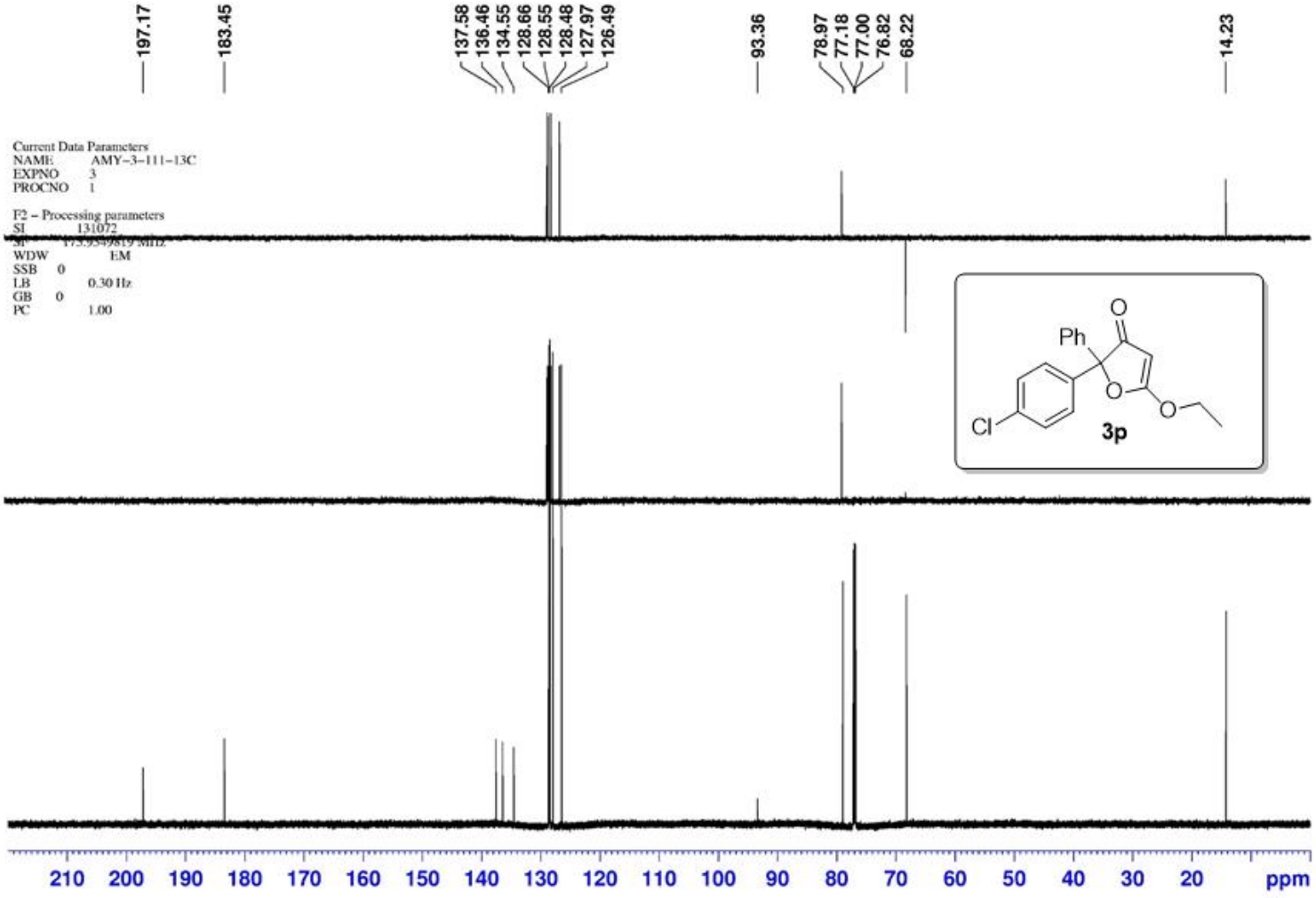
^{19}F NMR; 500 MHz
Solvent: $CDCl_3$

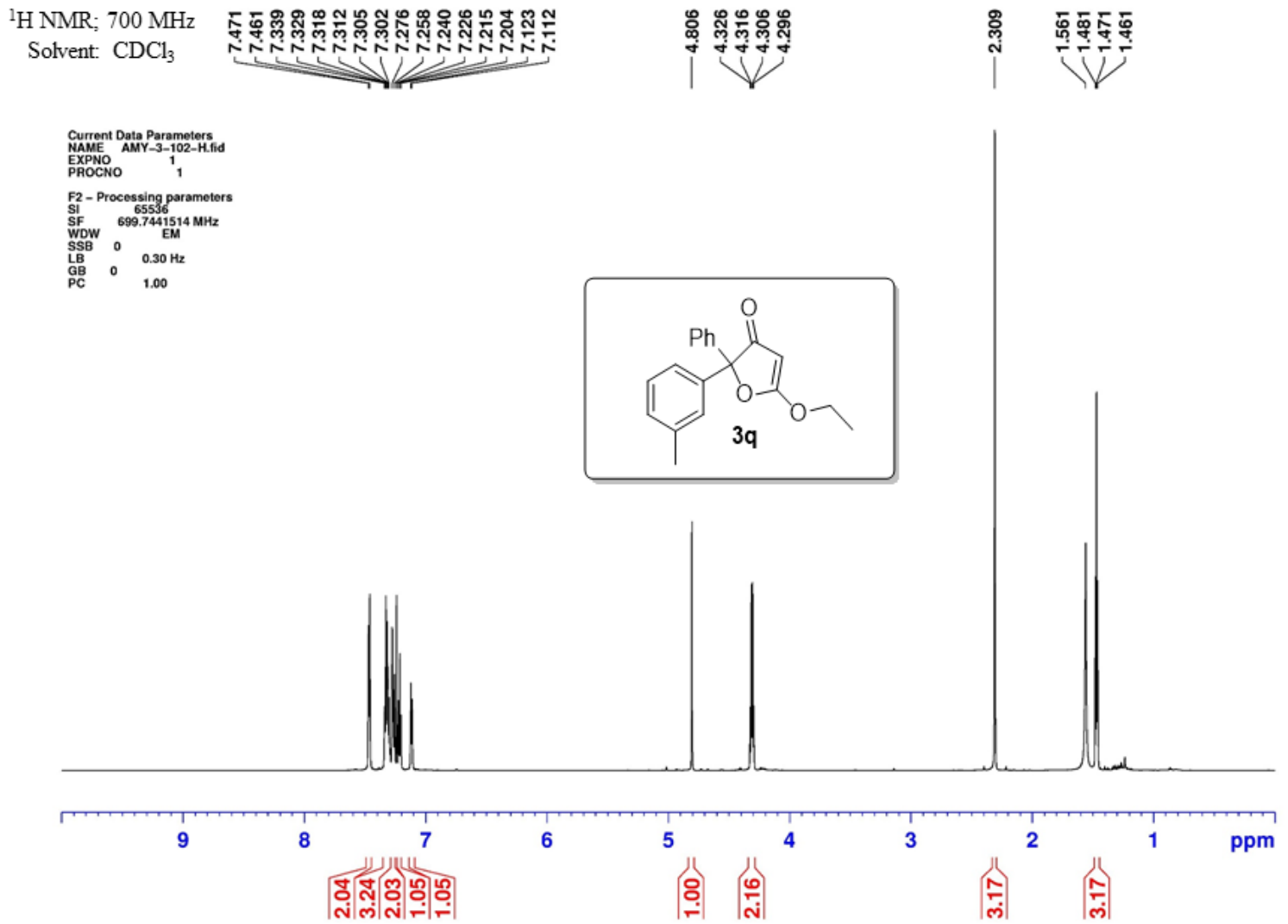


^{1}H NMR; 700 MHz
Solvent: $CDCl_3$

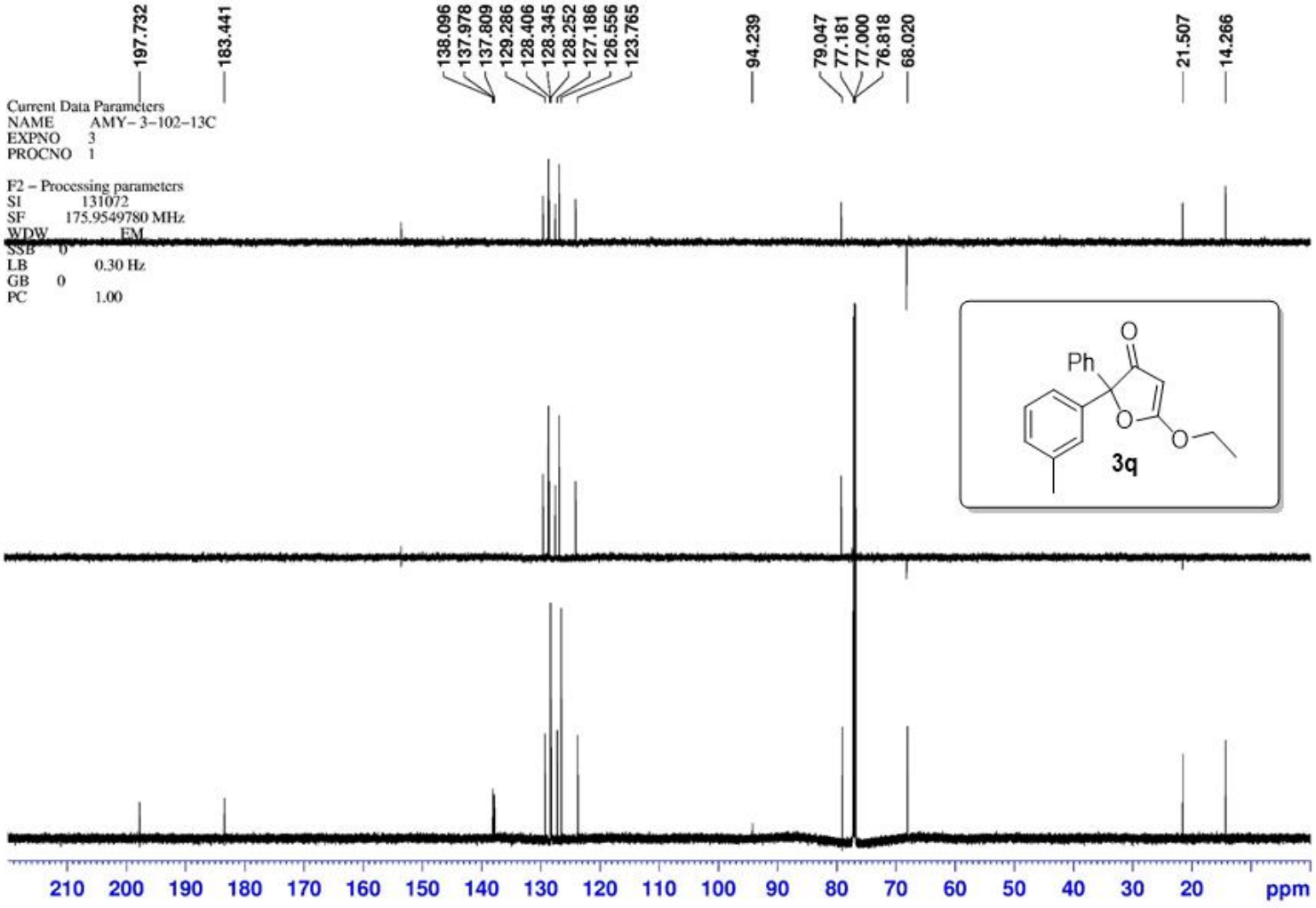


^{13}C NMR; 175 MHz
Solvent: $CDCl_3$

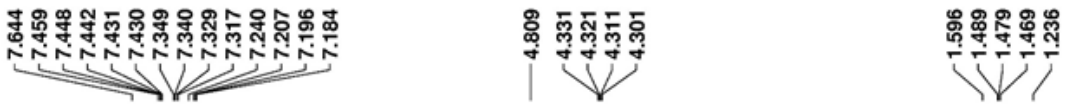



^{1}H NMR; 700 MHz
Solvent: $CDCl_3$
7.471
7.461
7.339
7.329
7.318
7.312
7.305
7.302
7.276
7.258
7.240
7.226
7.215
7.204
7.123
7.112
4.806
4.326
4.316
4.306
4.296
2.309
1.561
1.481
1.471
1.461
Current Data Parameters
NAME AMY-3-102-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7441514 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
O
O
O
3q
9
8
7
6
5
4
3
2
1
ppm
2.04
3.24
2.03
1.05
1.05
1.00
2.16
3.17
3.17

^{13}C NMR; 175 MHz
Solvent: $CDCl_3$

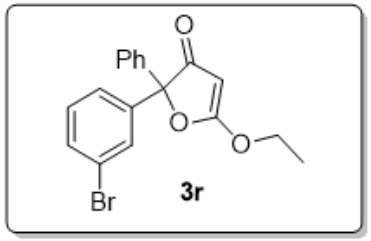


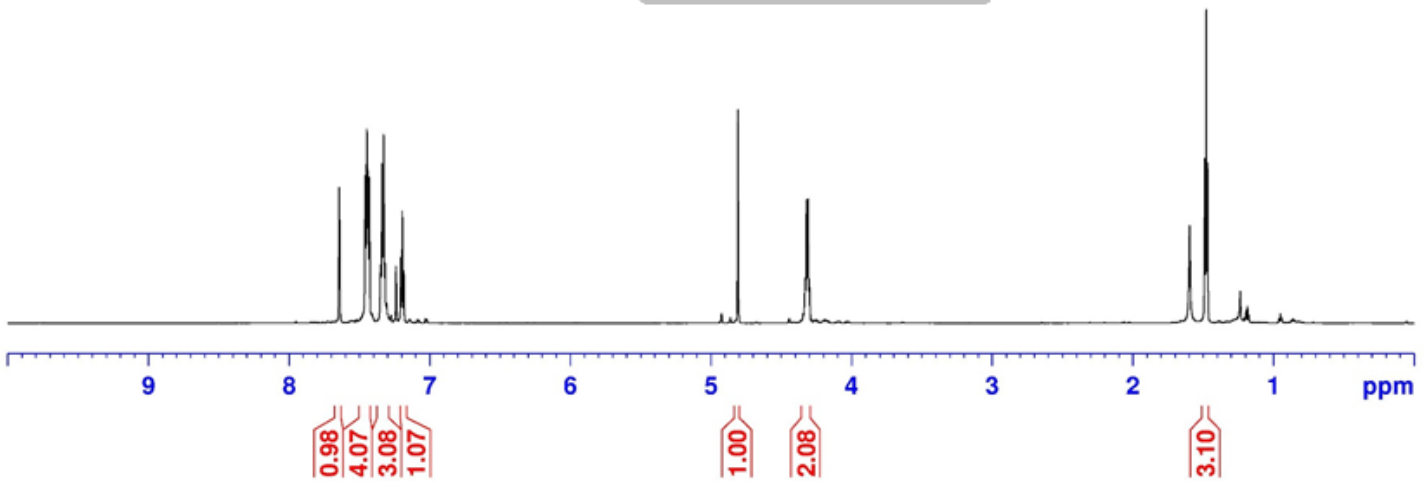
^{1}H NMR; 700 MHz
Solvent: $CDCl_3$



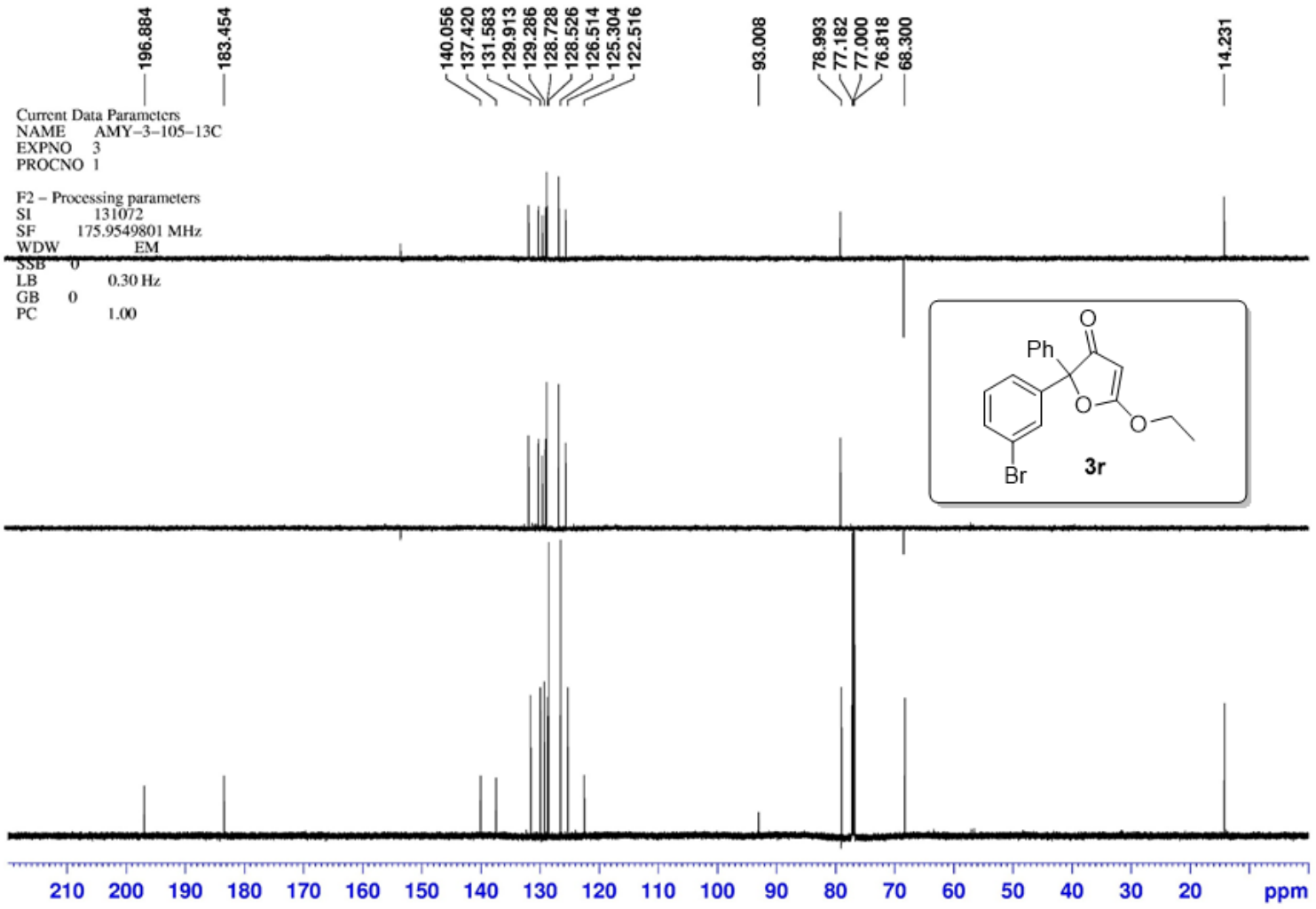
Current Data Parameters
NAME AMY-3-105-H.fid
EXPNO 1
PROCNO 1

F2 – Processing parameters
SI 65536
SF 699.7441520 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

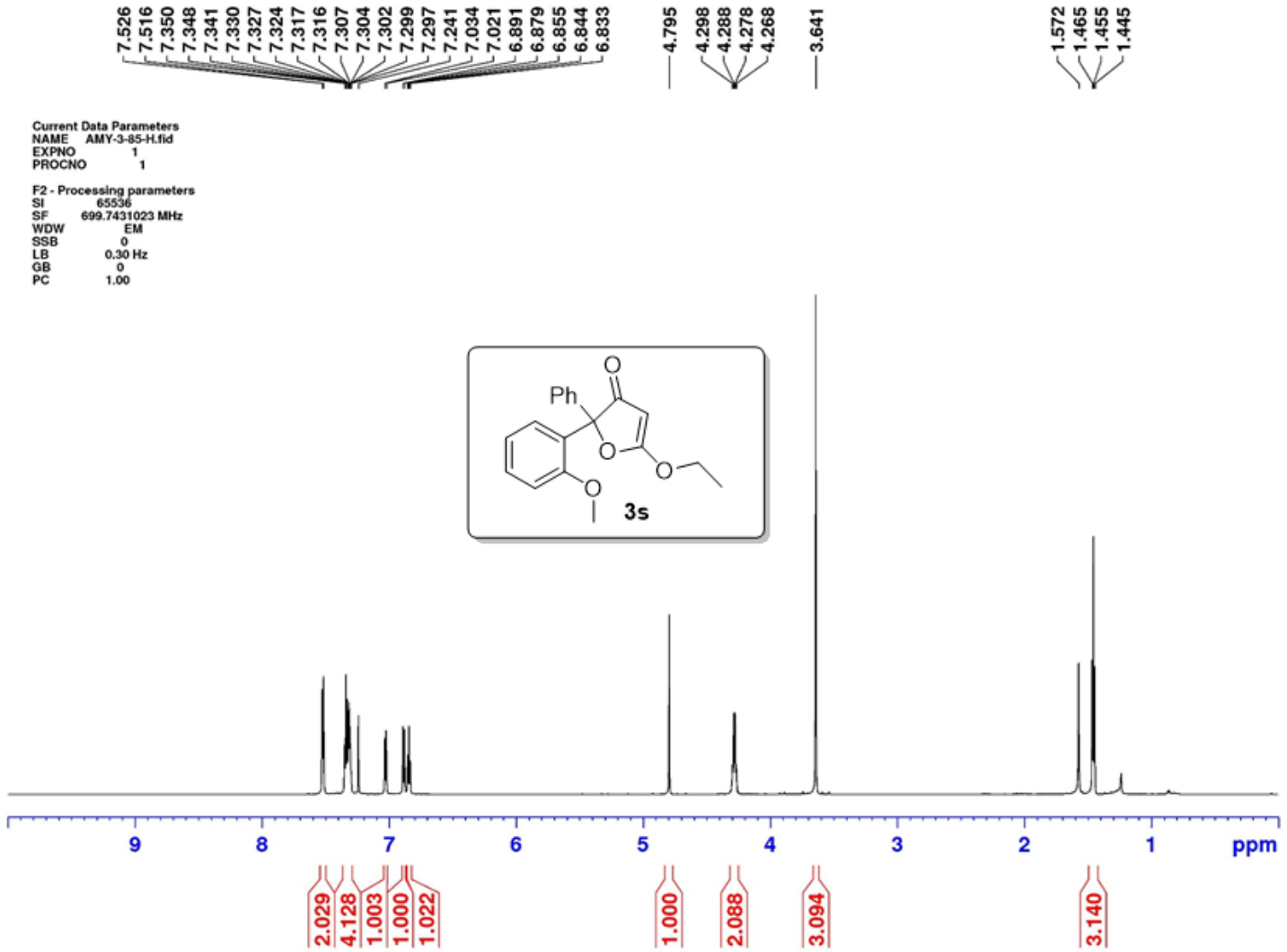




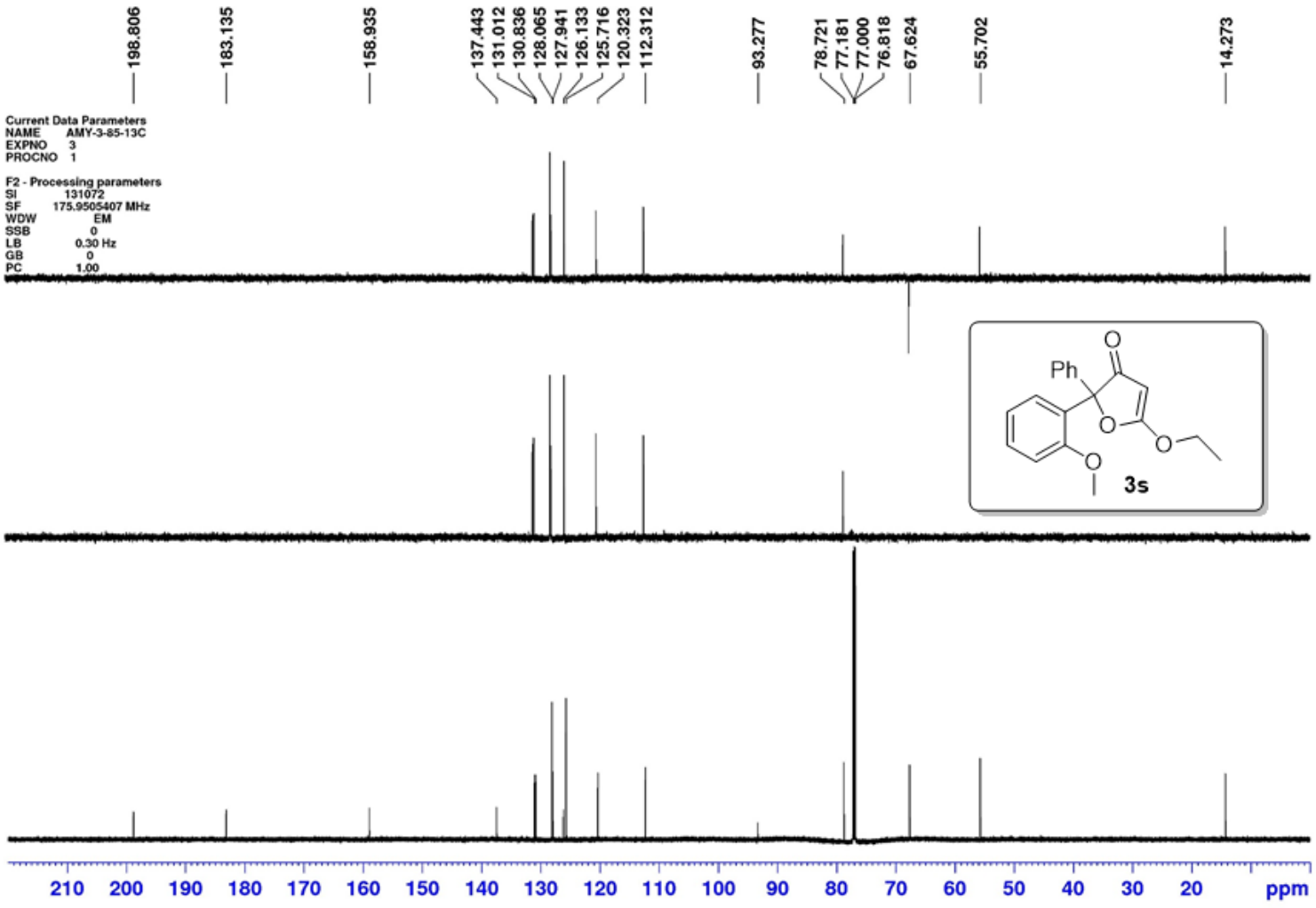
^{13}C NMR; 175 MHz
Solvent: $CDCl_3$



^{1}H NMR; 700 MHz
Solvent: $CDCl_3$



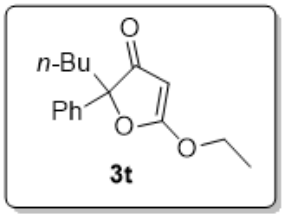
^{13}C NMR; 175 MHz
Solvent: $CDCl_3$



^{1}H NMR; 700 MHz
Solvent: $CDCl_3$

Current Data Parameters
NAME AMY-3-96-H.fid
EXPNO 1
PROCNO 1

F2 – Processing parameters
SI 65536
SF 699.7438011 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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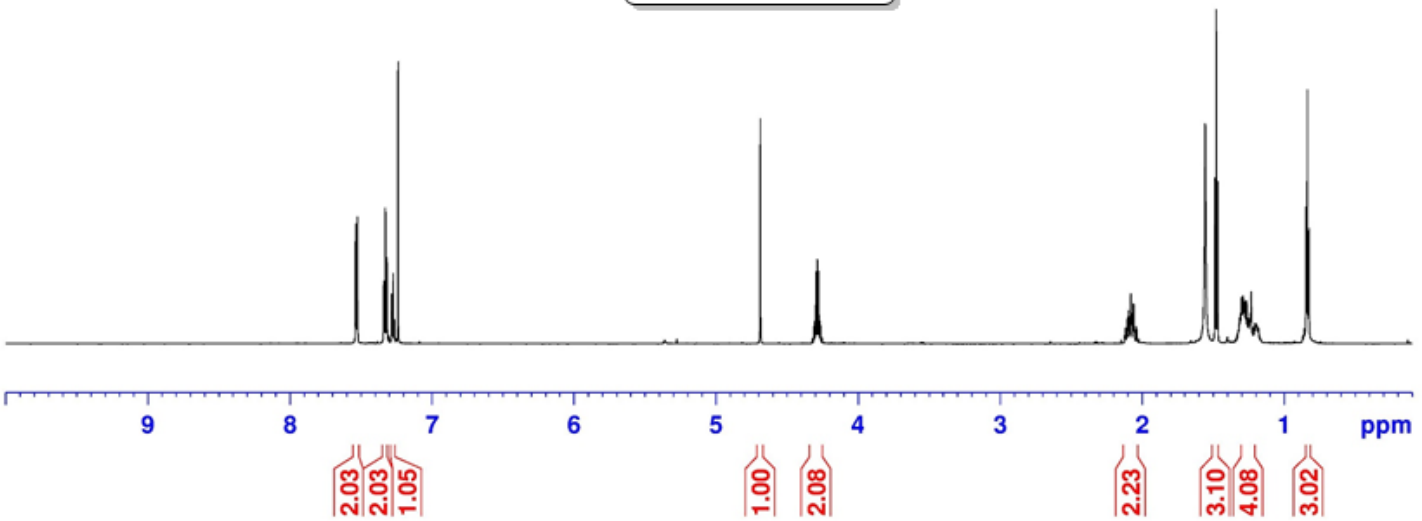
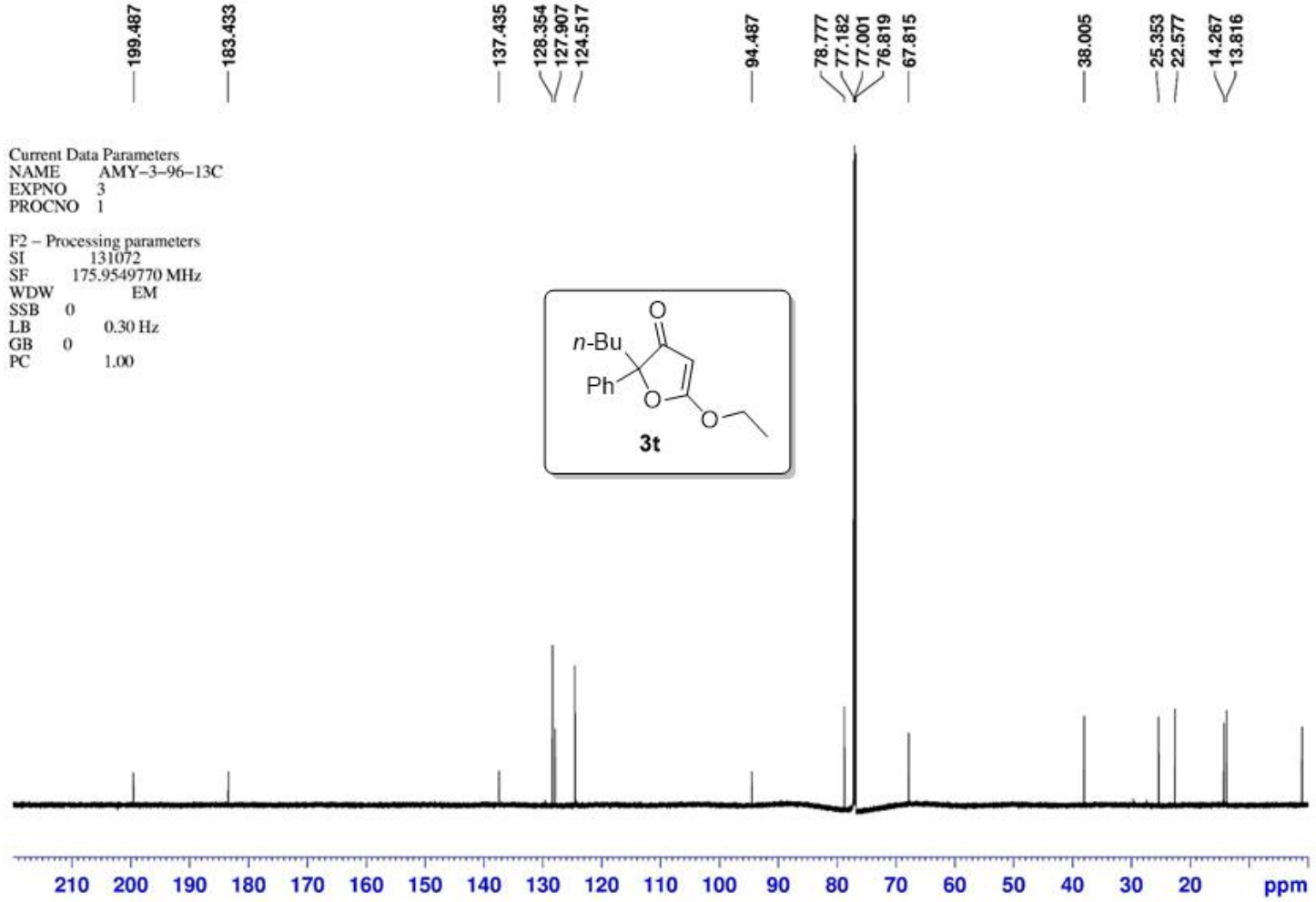
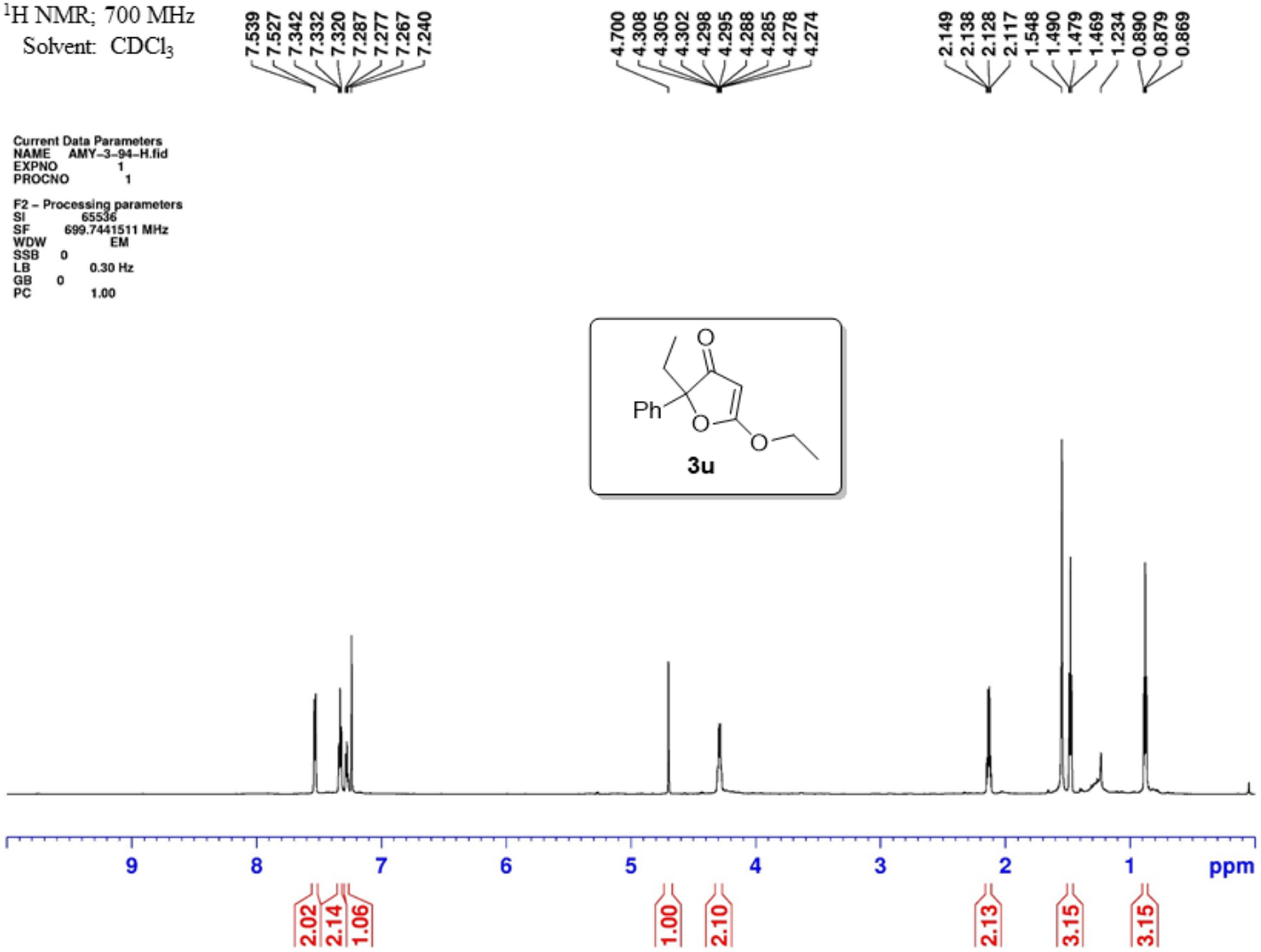
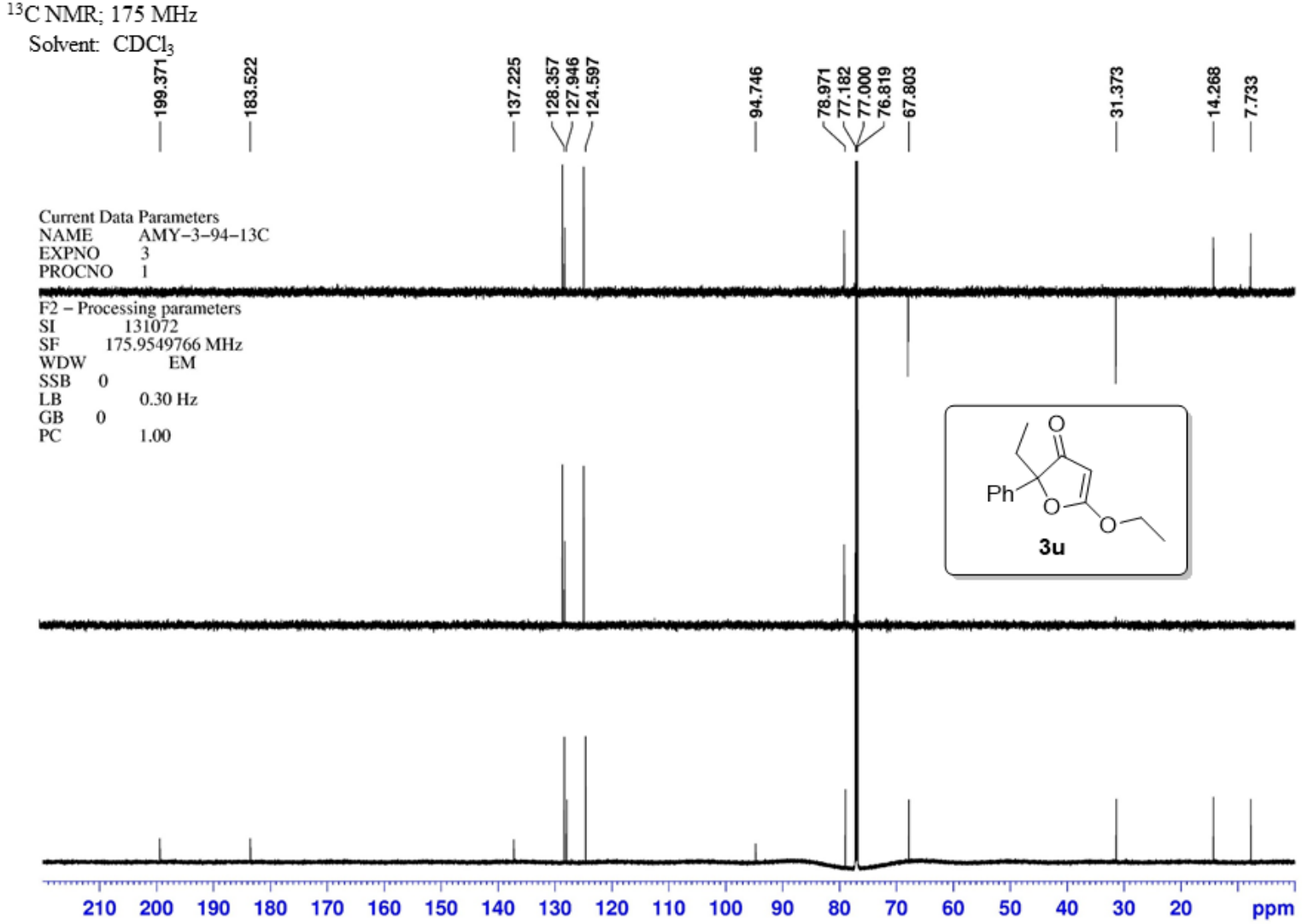


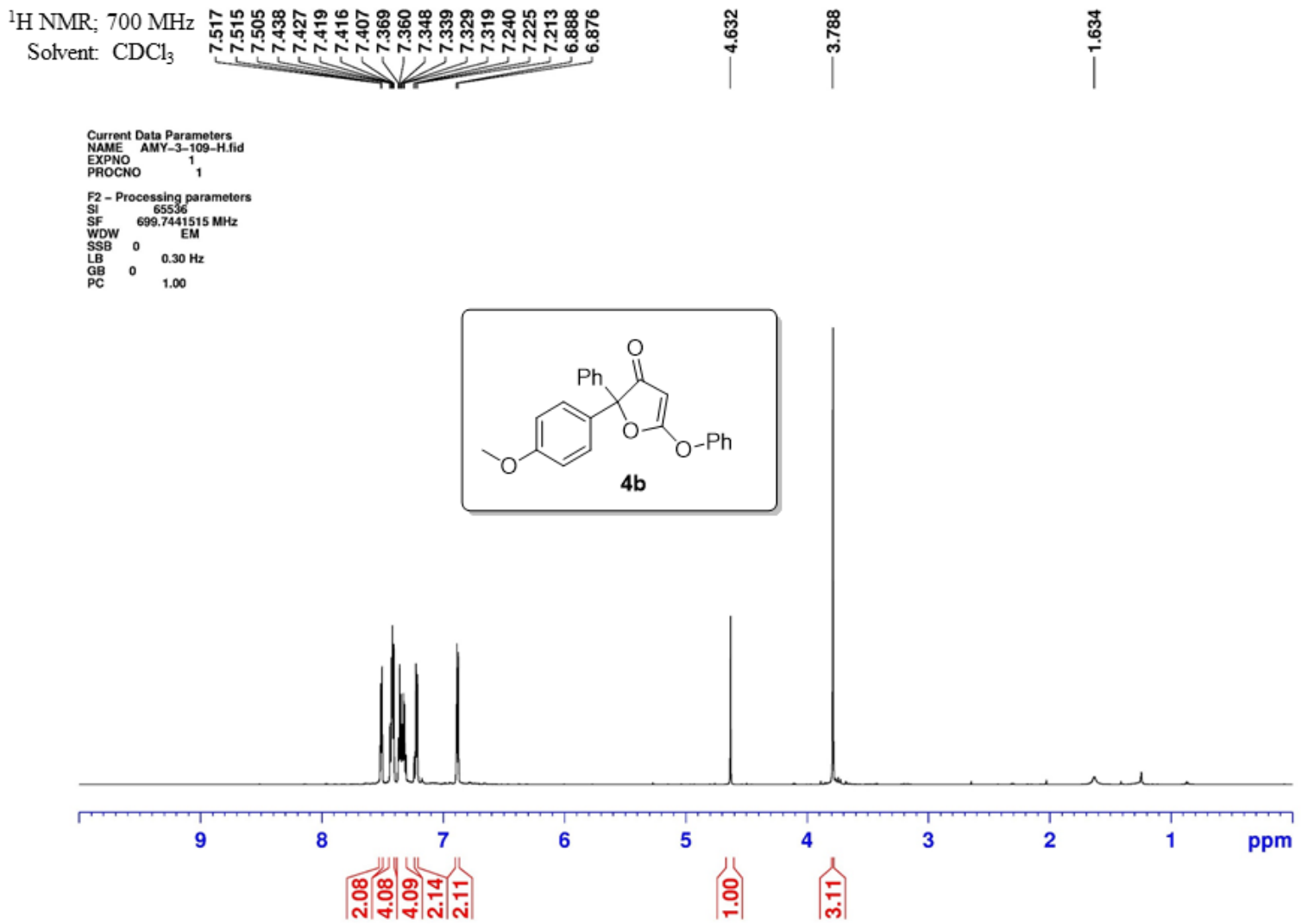
^{13}C NMR; 175 MHz
Solvent: $CDCl_3$



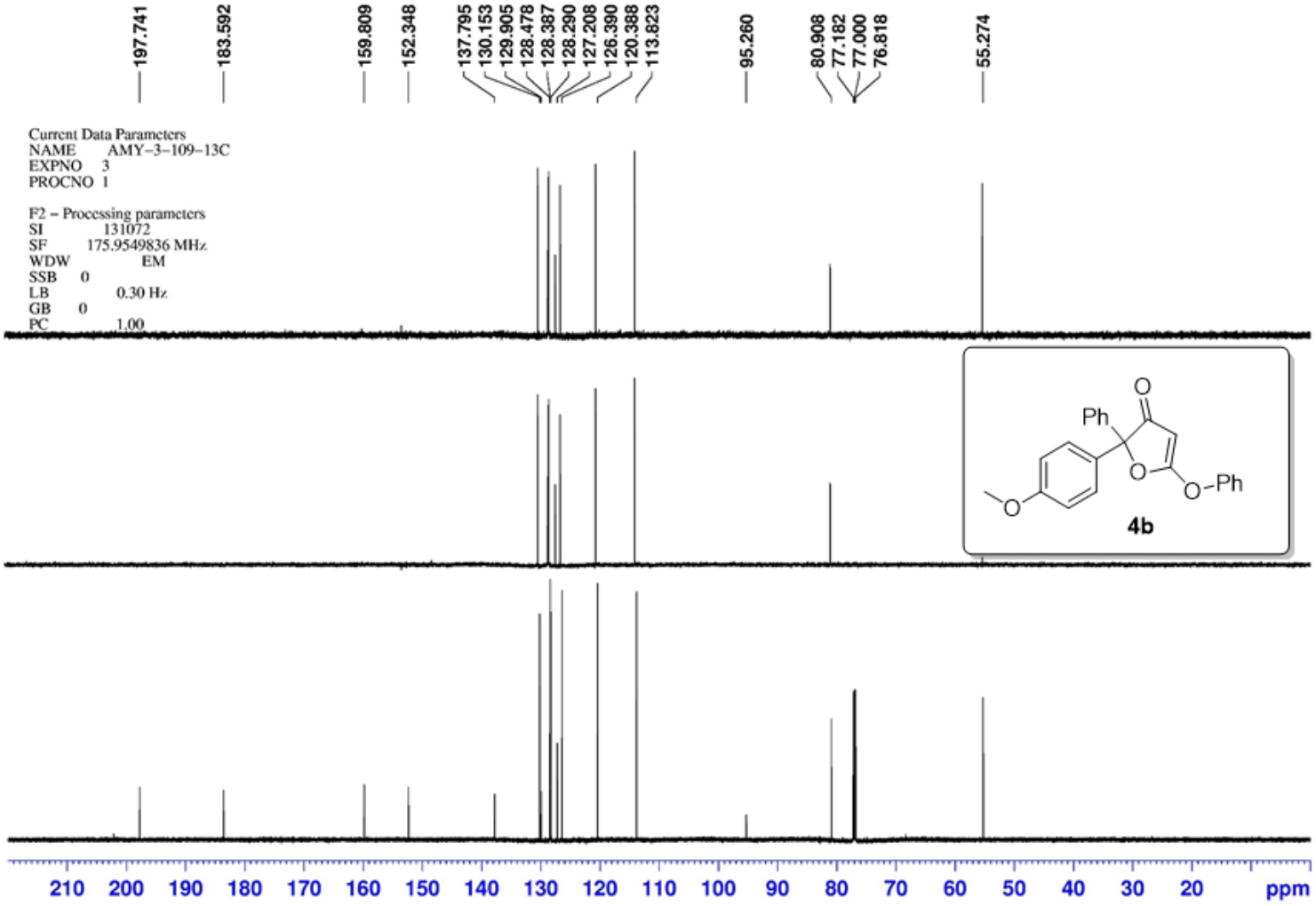
^{1}H NMR; 700 MHz
Solvent: $CDCl_3$




^{13}C NMR; 175 MHz
Solvent: $CDCl_3$
199.371
183.522
137.225
128.357
127.946
124.597
94.746
78.971
77.182
77.000
76.819
67.803
31.373
14.268
7.733
Current Data Parameters
NAME AMY-3-94-13C
EXPNO 3
PROCNO 1
F2 – Processing parameters
SI 131072
SF 175.9549766 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
O
O
O
3u
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


^{1}H NMR; 700 MHz
Solvent: $CDCl_3$
7.517
7.515
7.505
7.438
7.427
7.419
7.416
7.407
7.369
7.360
7.348
7.339
7.329
7.319
7.240
7.225
7.213
6.888
6.876
4.632
3.788
1.634
Current Data Parameters
NAME AMY-3-109-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7441515 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
O
O
O
O-Ph
4b
9
8
7
6
5
4
3
2
1
ppm
2.08
4.08
4.09
2.14
2.11
1.00
3.11

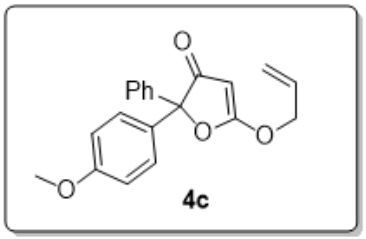
^{13}C NMR; 175 MHz
Solvent: $CDCl_3$



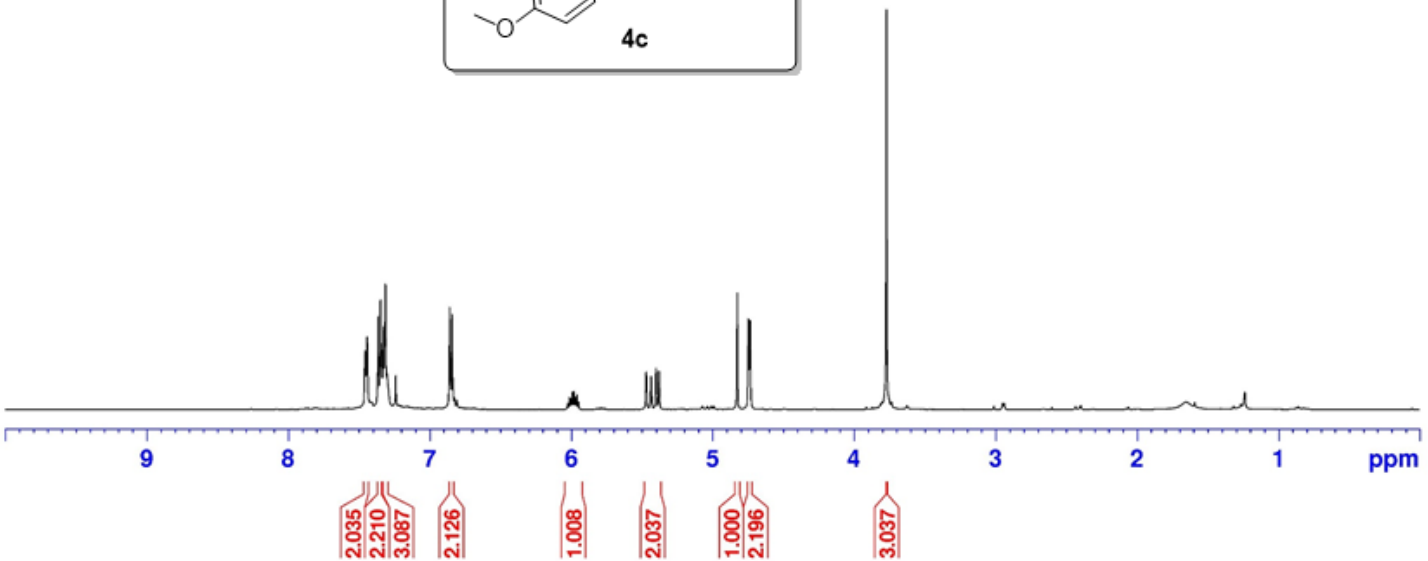
^{1}H NMR; 500 MHz
Solvent: $CDCl_3$

Current Data Parameters
NAME AMY-3-167-1H.fid
EXPNO 1
PROCNO 1

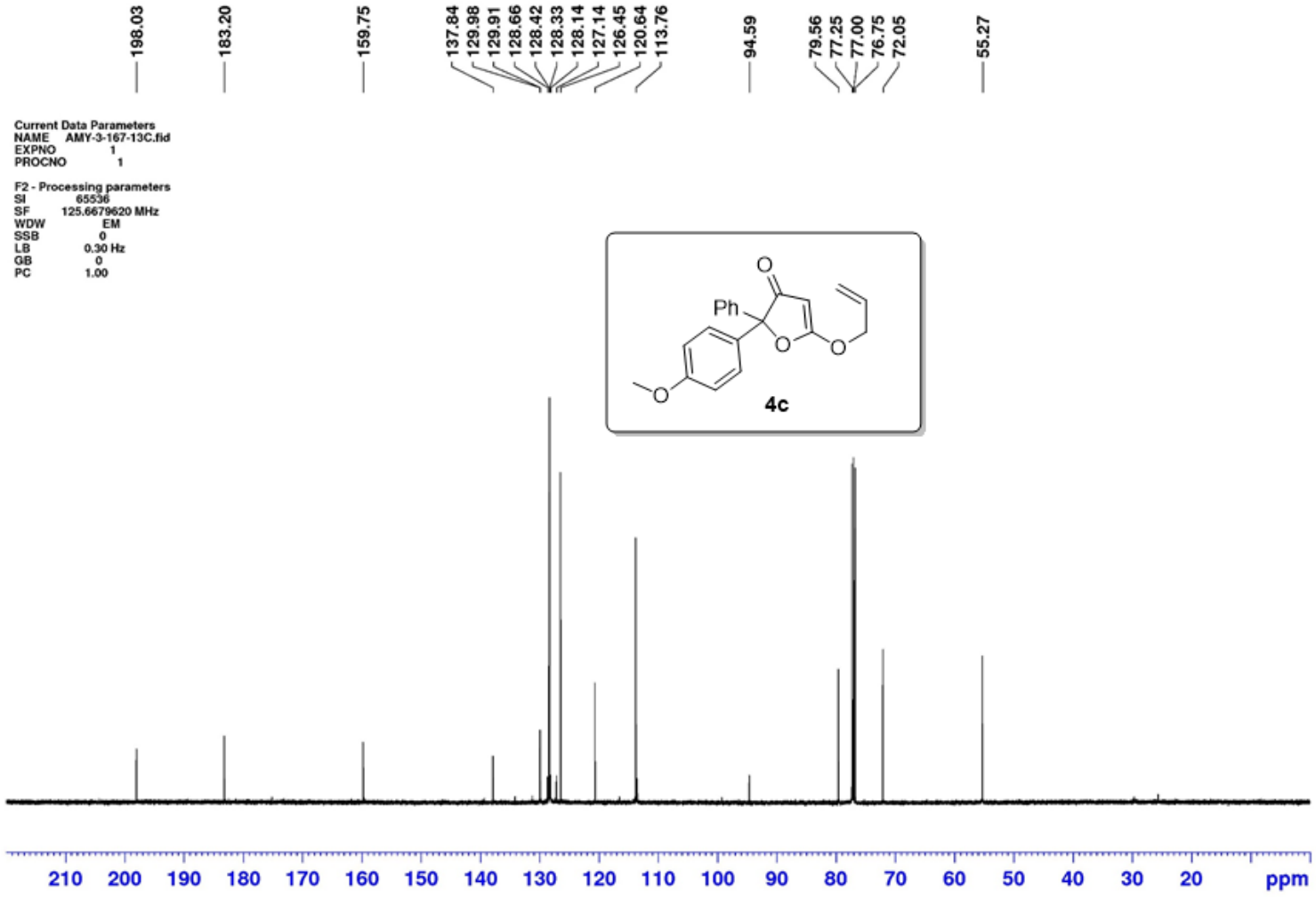
F2 - Processing parameters
SI 32768
SF 499.7727654 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

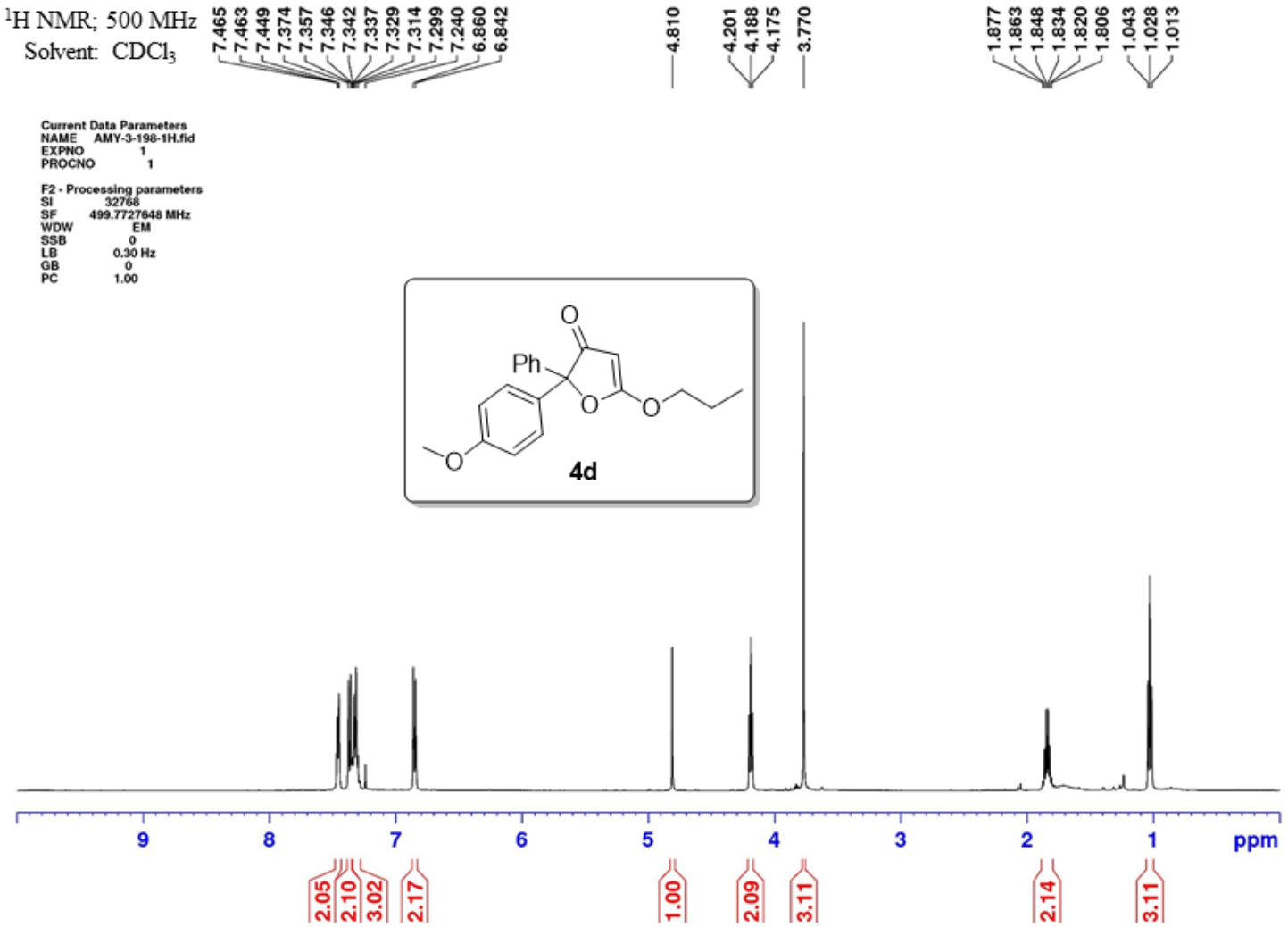


4c

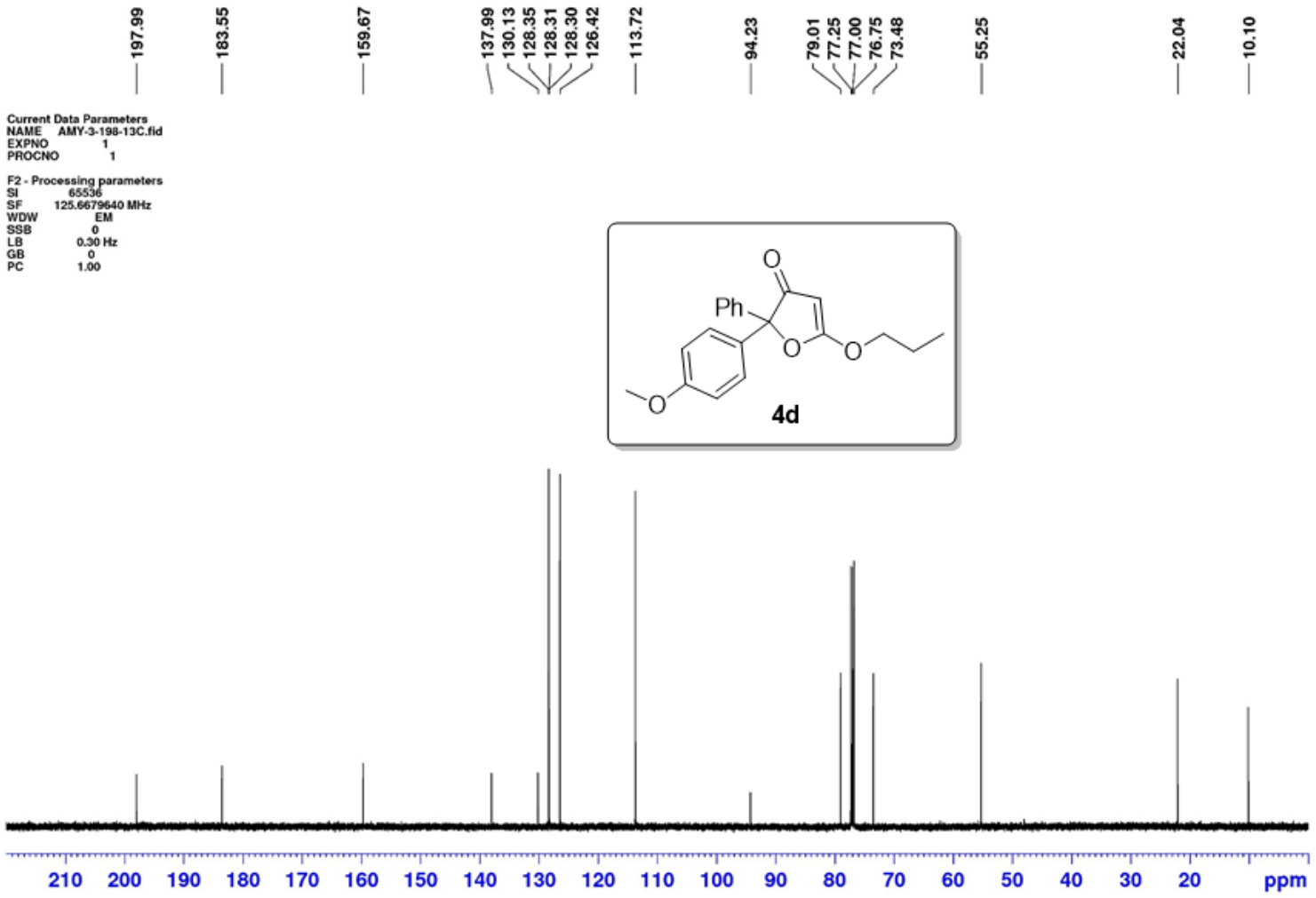


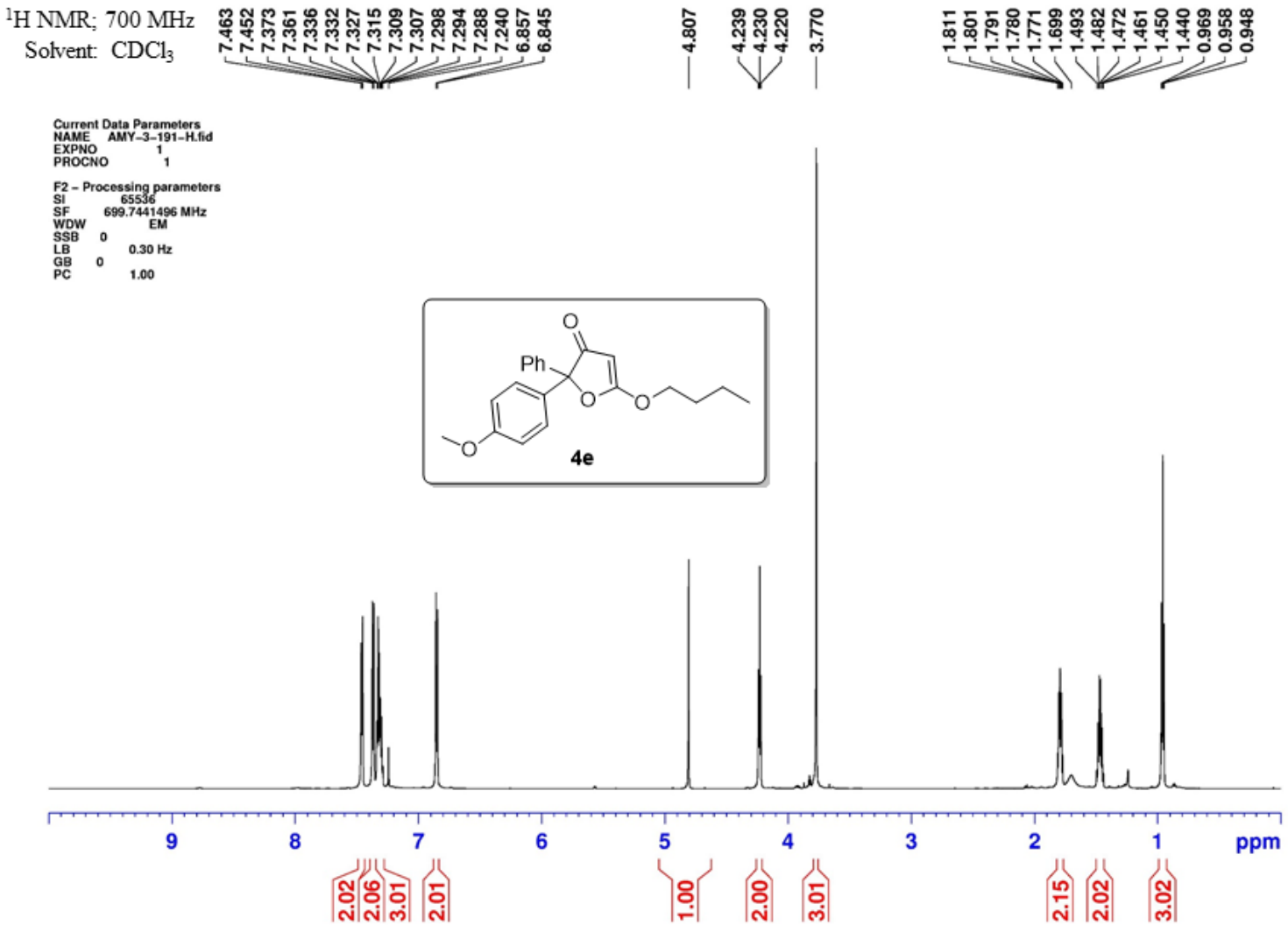
^{13}C NMR; 125 MHz
Solvent: $CDCl_3$



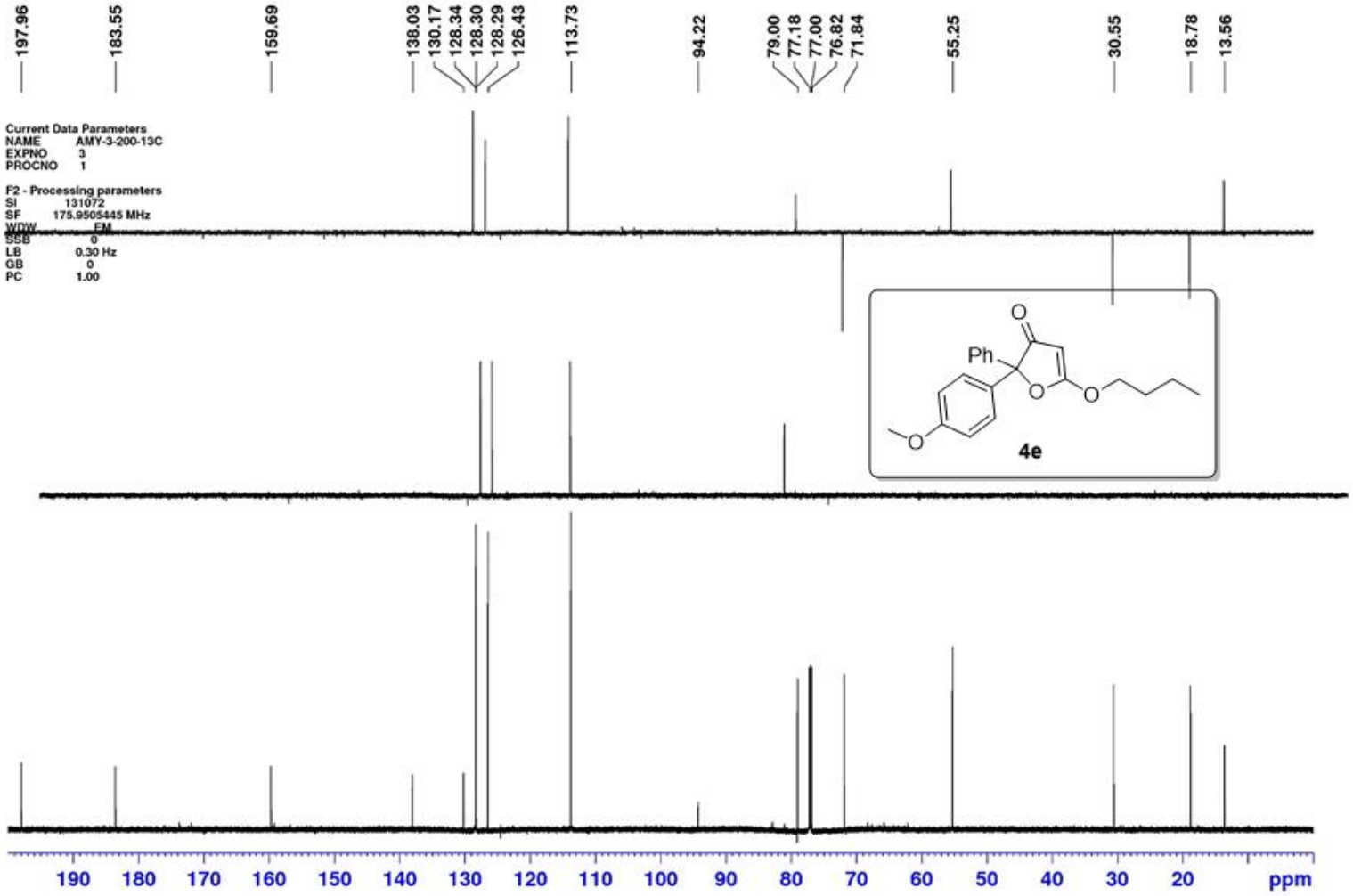

^{1}H NMR; 500 MHz
Solvent: $CDCl_3$
7.465
7.463
7.449
7.374
7.357
7.346
7.342
7.337
7.329
7.314
7.299
7.240
6.860
6.842
4.810
4.201
4.188
4.175
3.770
1.877
1.863
1.848
1.834
1.820
1.806
1.043
1.028
1.013
Current Data Parameters
NAME AMY-3-198-1H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 32768
SF 499.7727648 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
O
O
O
O
4d
9
8
7
6
5
4
3
2
1
ppm
2.05
2.10
3.02
2.17
1.00
2.09
3.11
2.14
3.11

^{13}C NMR; 125 MHz
Solvent: $CDCl_3$




^{1}H NMR; 700 MHz
Solvent: $CDCl_3$
7.463
7.452
7.373
7.361
7.336
7.332
7.327
7.315
7.309
7.307
7.298
7.294
7.288
7.240
6.857
6.845
4.807
4.239
4.230
4.220
3.770
1.811
1.801
1.791
1.780
1.771
1.699
1.493
1.482
1.472
1.461
1.450
1.440
0.969
0.958
0.948
Current Data Parameters
NAME AMY-3-191-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7441496 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
O
O
O
O
4e
9
8
7
6
5
4
3
2
1
ppm
2.02
2.06
3.01
2.01
1.00
2.00
3.01
2.15
2.02
3.02

^{13}C NMR; 175 MHz
Solvent: $CDCl_3$



^{1}H NMR; 500 MHz
Solvent: $CDCl_3$

7.461 7.447 7.375 7.357 7.345 7.342 7.337 7.329 7.313 7.297 7.284 7.240 6.860 6.843 6.826

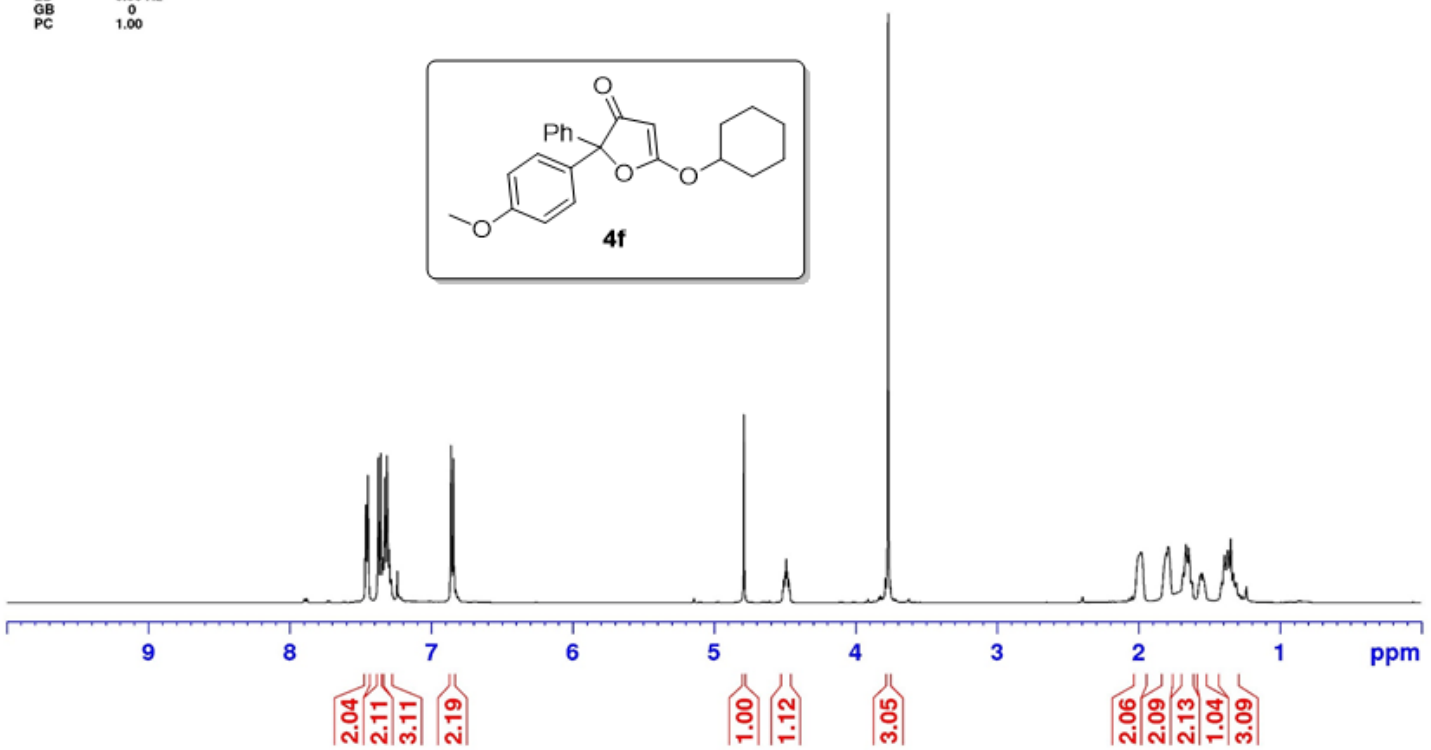
4.789 4.515 4.507 4.498 4.490 4.482 4.472 4.465 3.769

1.999 1.982 1.977 1.804 1.787 1.682 1.663 1.644 1.626 1.619 1.564 1.558 1.546 1.535 1.411 1.391 1.369 1.347 1.328

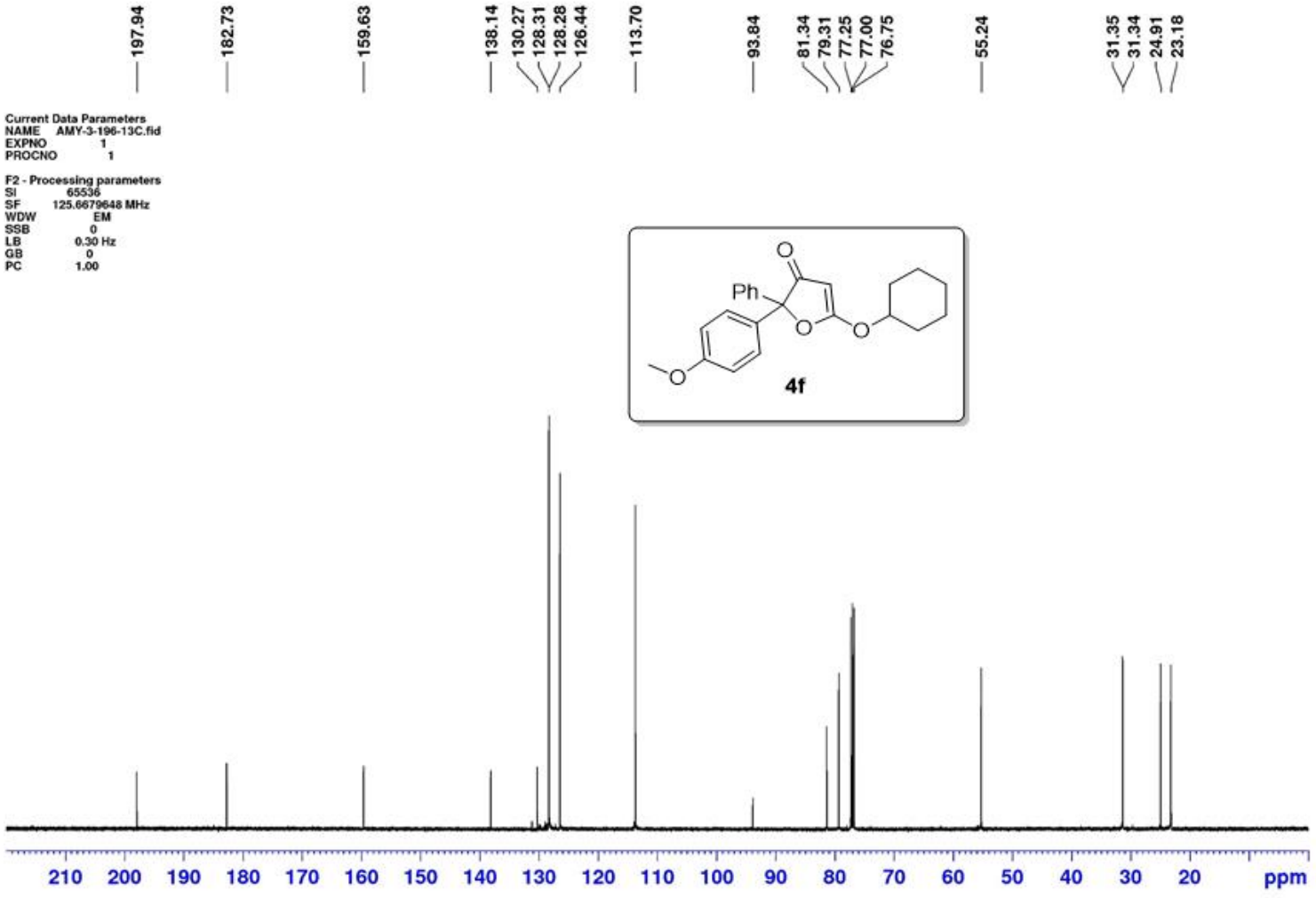
Current Data Parameters
NAME AMY-3-196-1H.fid
EXPNO 1
PROCNO 1

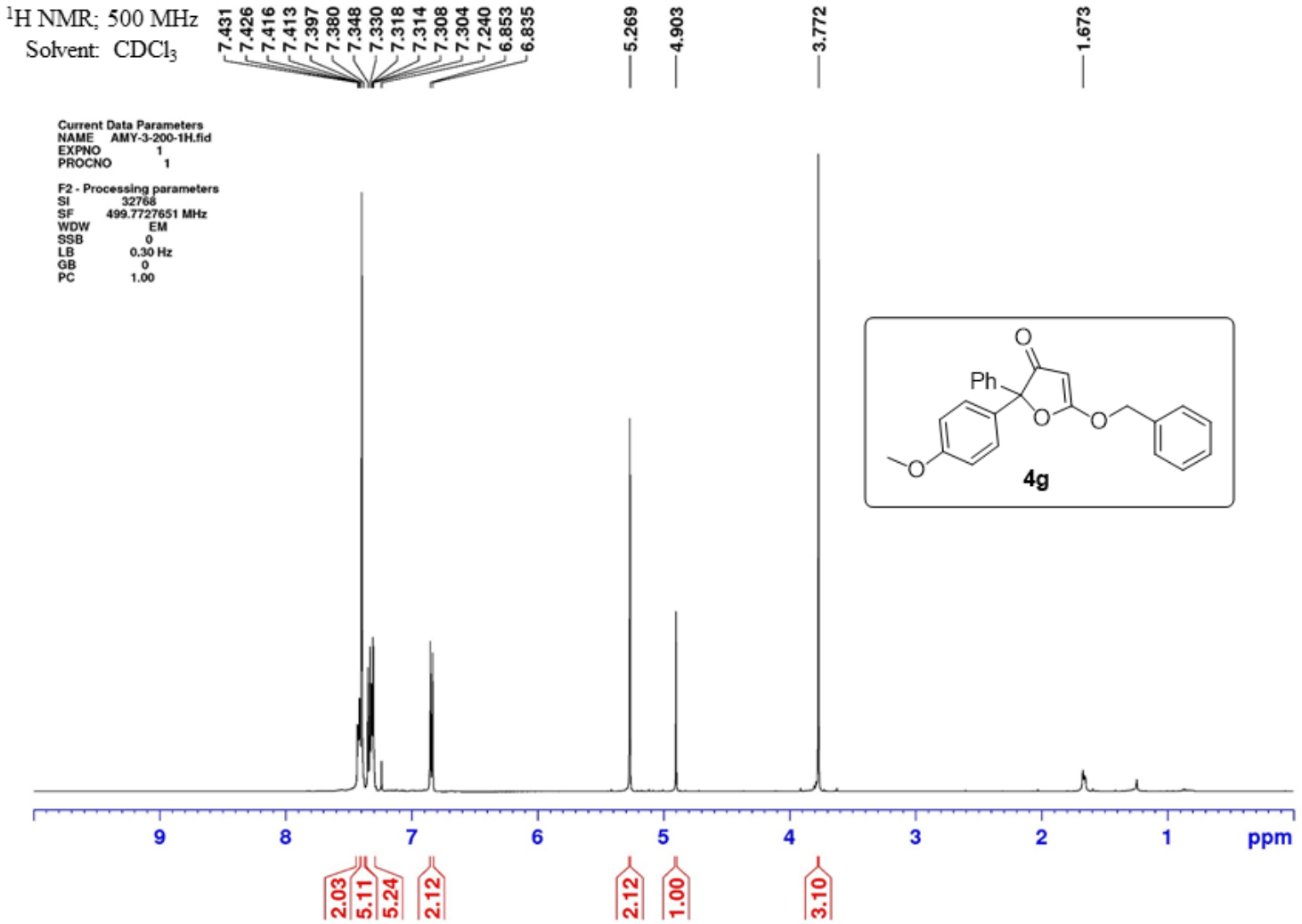
F2 - Processing parameters
SI 32768
SF 499.7727648 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

4f

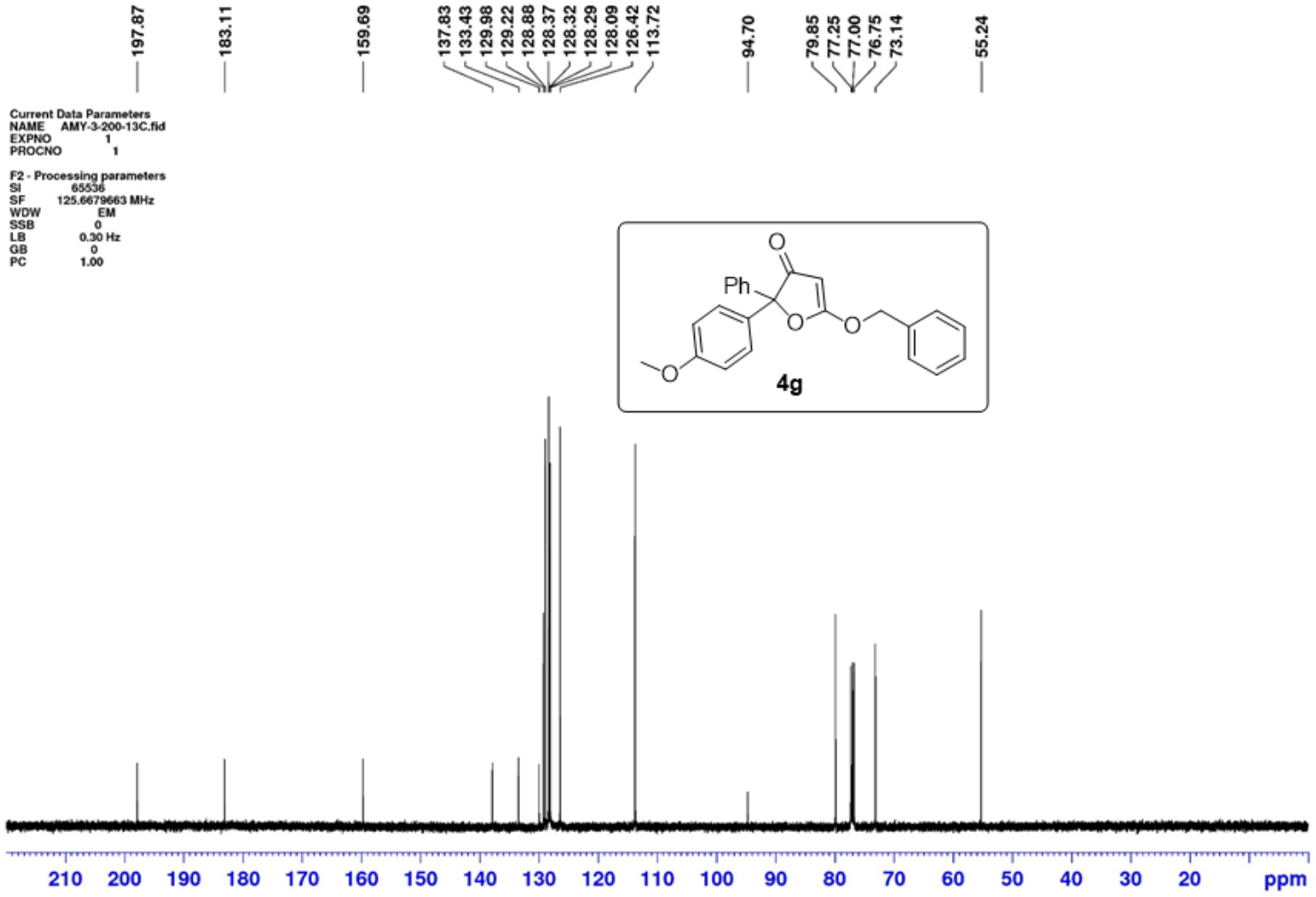


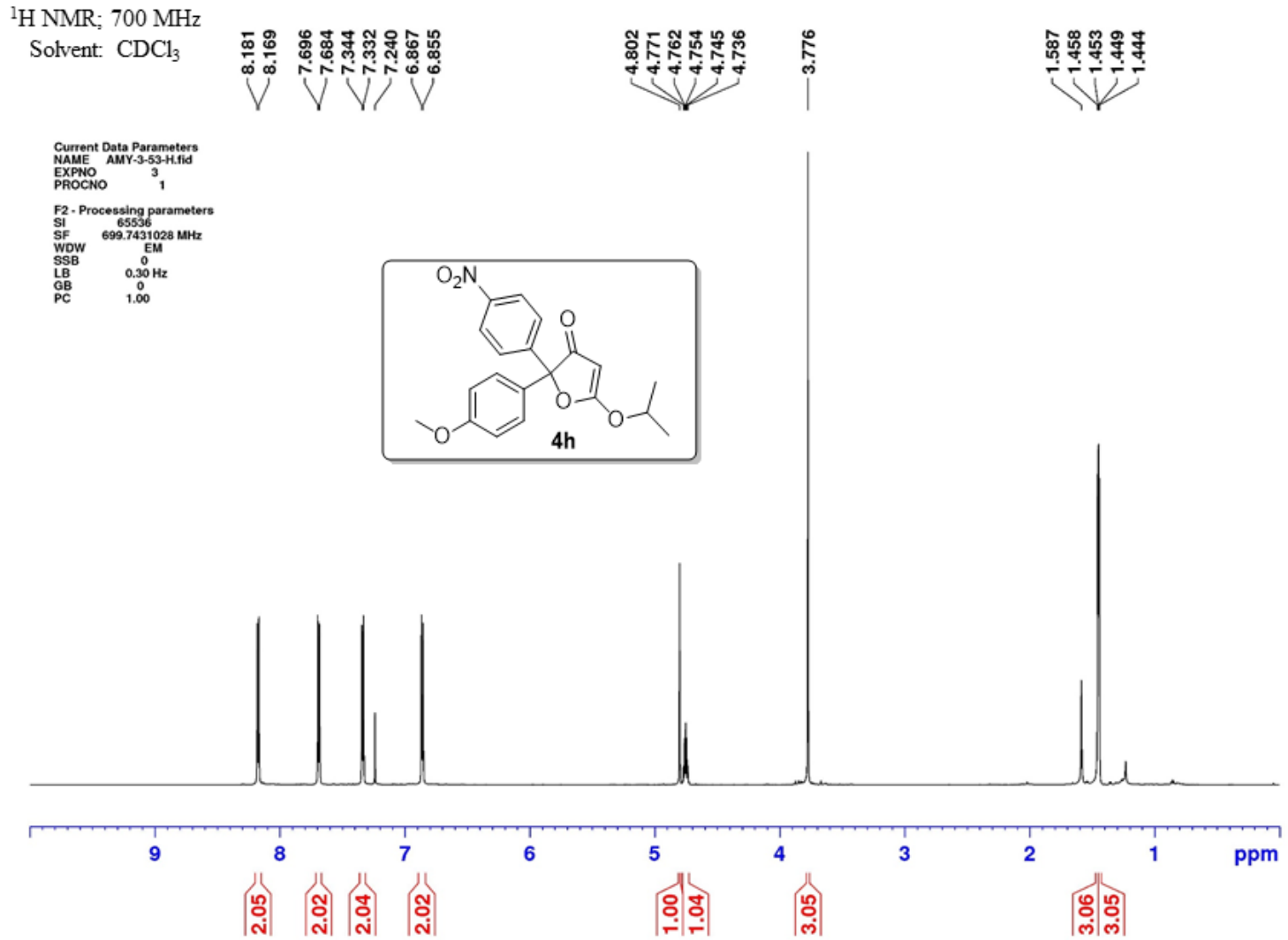
^{13}C NMR; 125 MHz
Solvent: $CDCl_3$



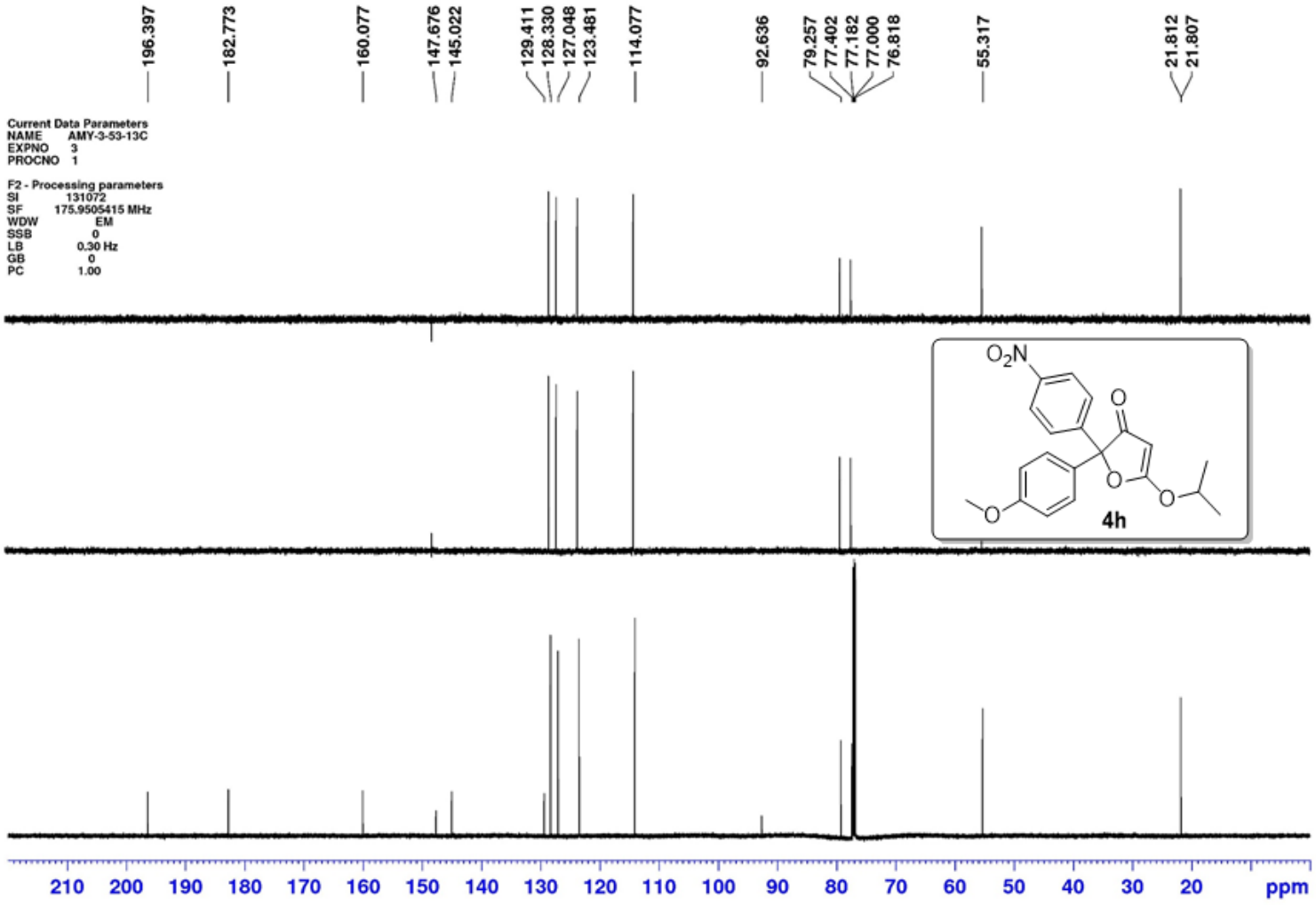

^{1}H NMR; 500 MHz
Solvent: $CDCl_3$
7.431
7.426
7.416
7.413
7.397
7.380
7.348
7.330
7.318
7.314
7.308
7.304
7.240
6.853
6.835
5.269
4.903
3.772
1.673
Current Data Parameters
NAME AMY-3-200-1H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 32768
SF 499.7727651 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
O
O
O
O
4g
9
8
7
6
5
4
3
2
1
ppm
2.03
5.11
5.24
2.12
2.12
1.00
3.10

^{13}C NMR; 125 MHz
Solvent: $CDCl_3$



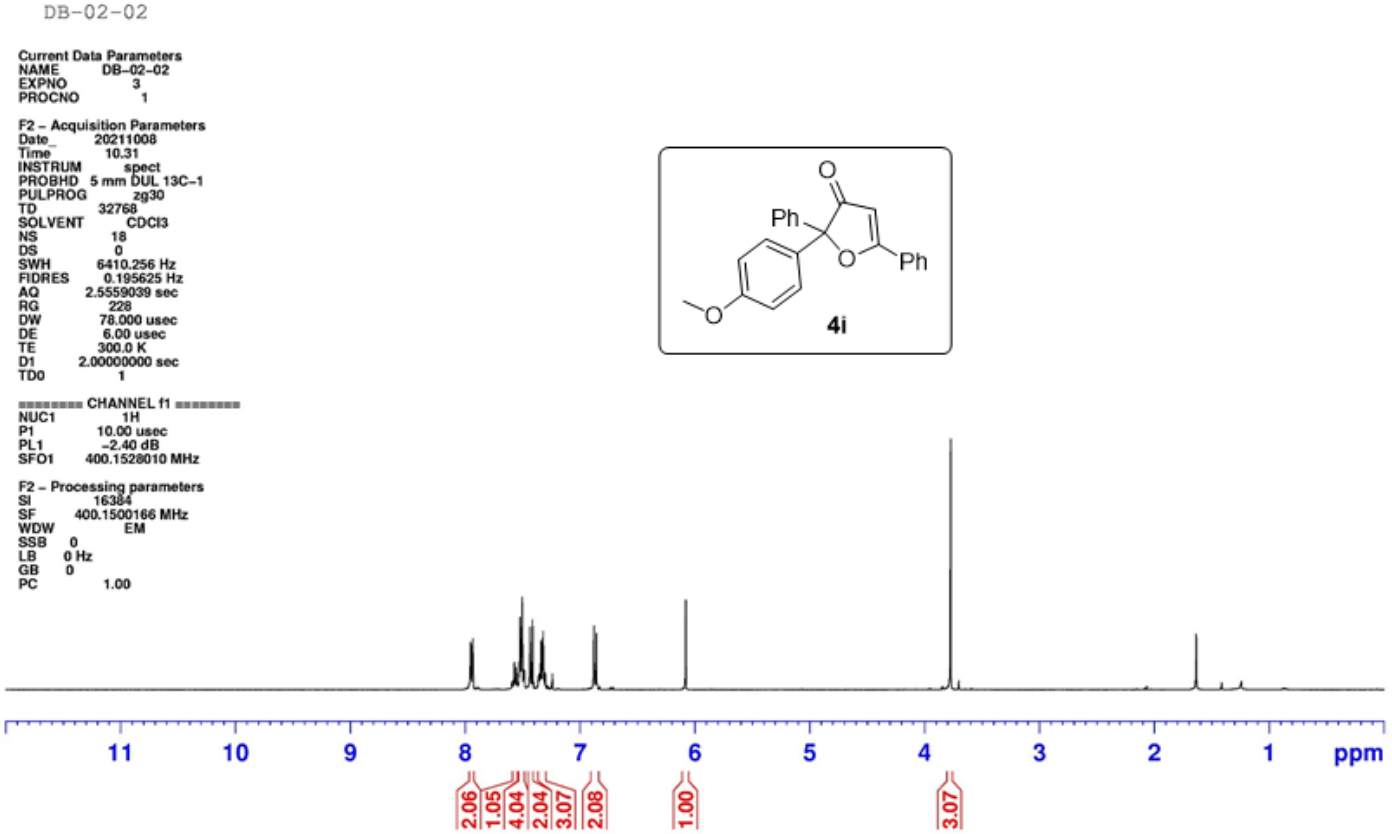

^{1}H NMR; 700 MHz
Solvent: $CDCl_3$
8.181
8.169
7.696
7.684
7.344
7.332
7.240
6.867
6.855
4.802
4.771
4.762
4.754
4.745
4.736
3.776
1.587
1.458
1.453
1.449
1.444
Current Data Parameters
NAME AMY-3-53-H.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7431028 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O_2N
O
O
O
O
4h
9
8
7
6
5
4
3
2
1
ppm
2.05
2.02
2.04
2.02
1.00
1.04
3.05
3.06
3.05

^{13}C NMR; 175 MHz
Solvent: $CDCl_3$

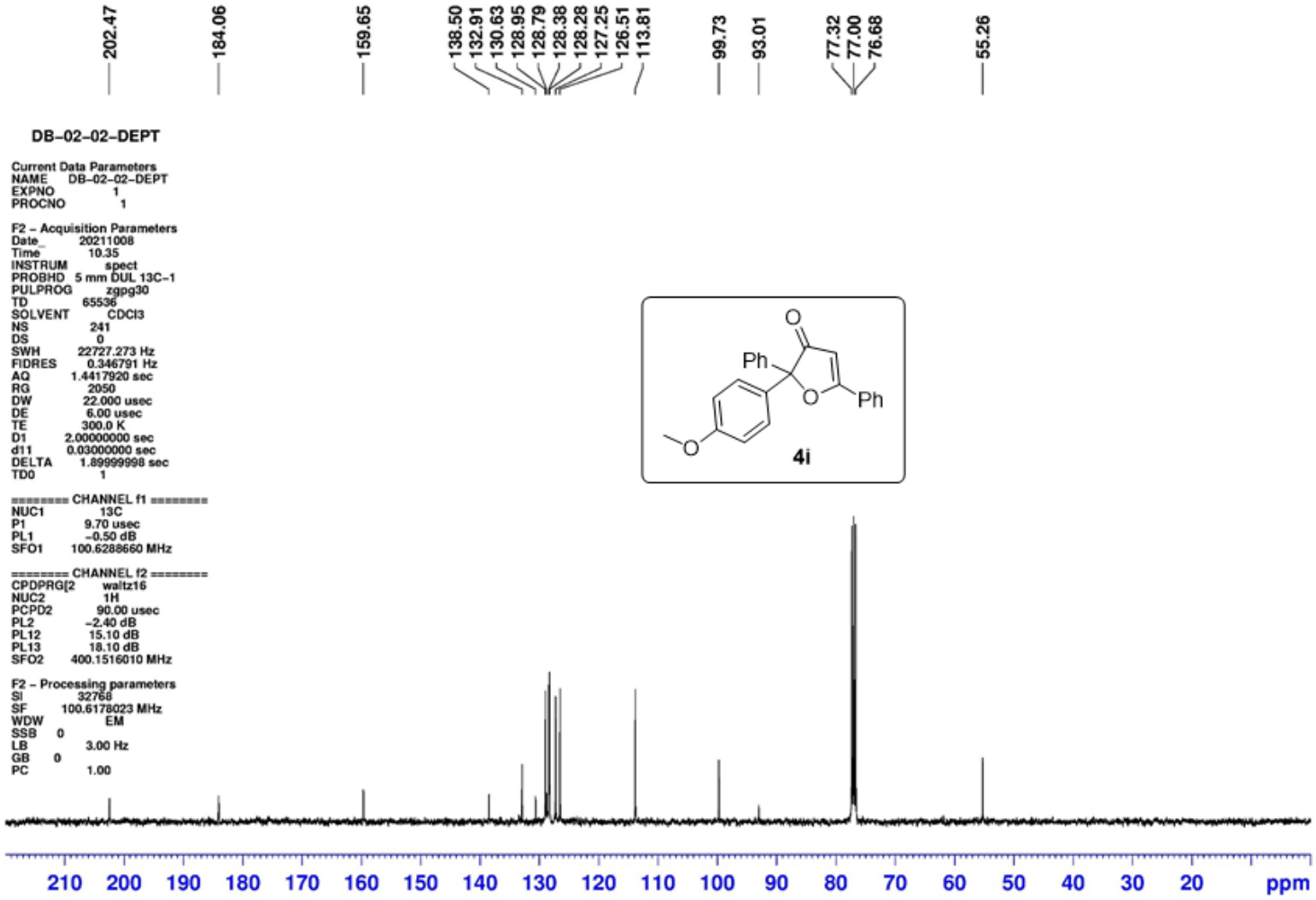


^{1}H NMR; 400 MHz

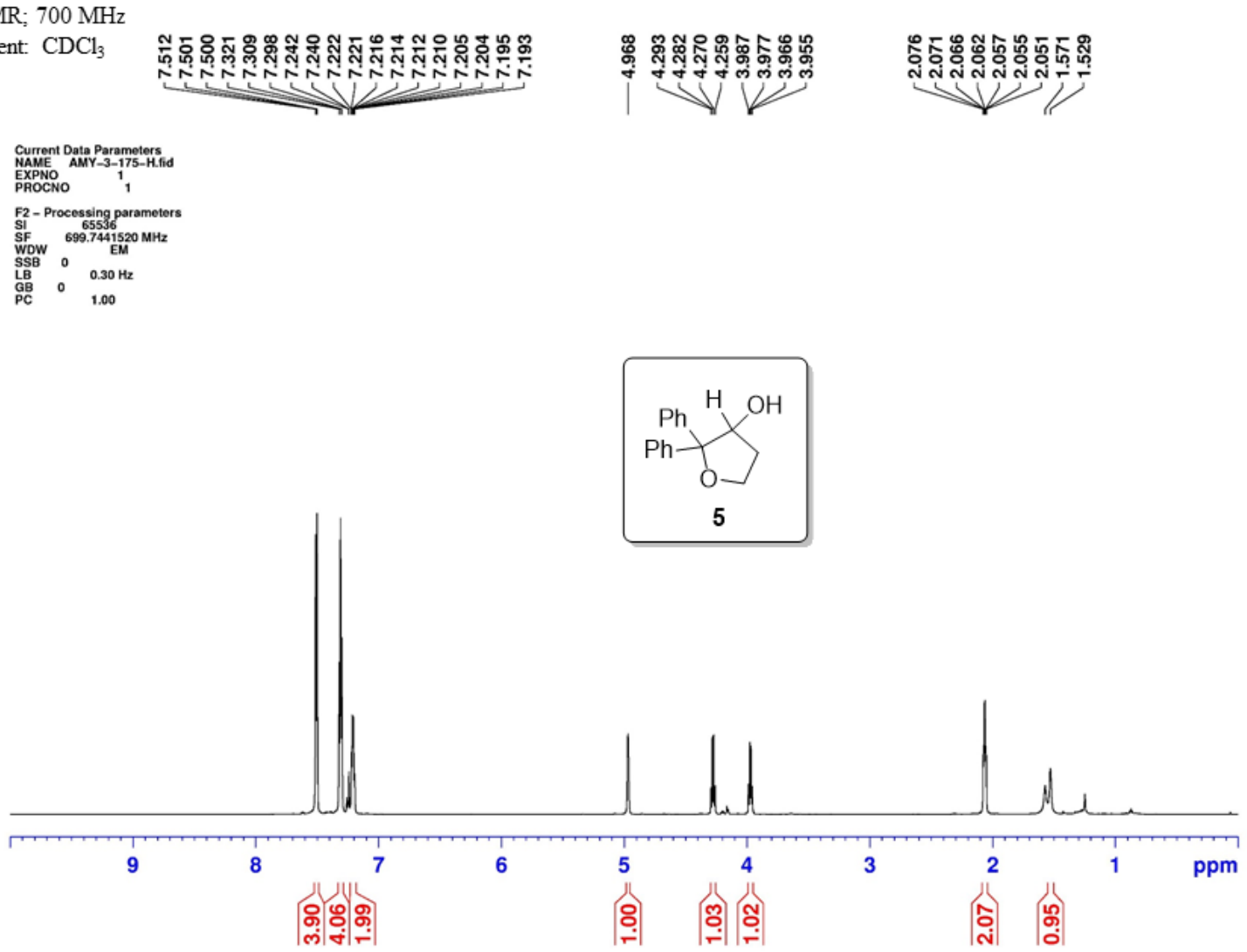
Solvent: $CDCl_3$

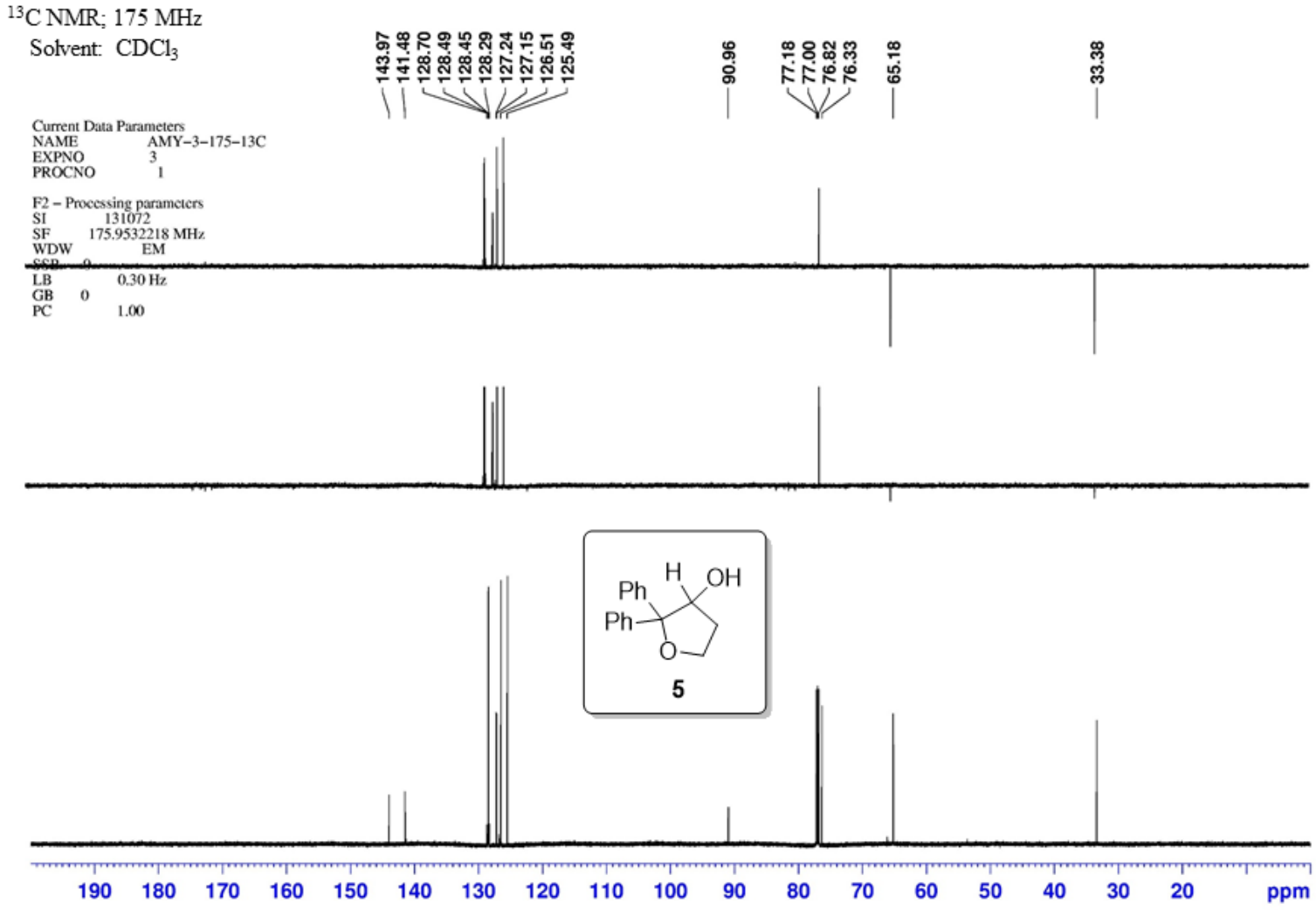


^{13}C NMR; 100 MHz
Solvent: $CDCl_3$

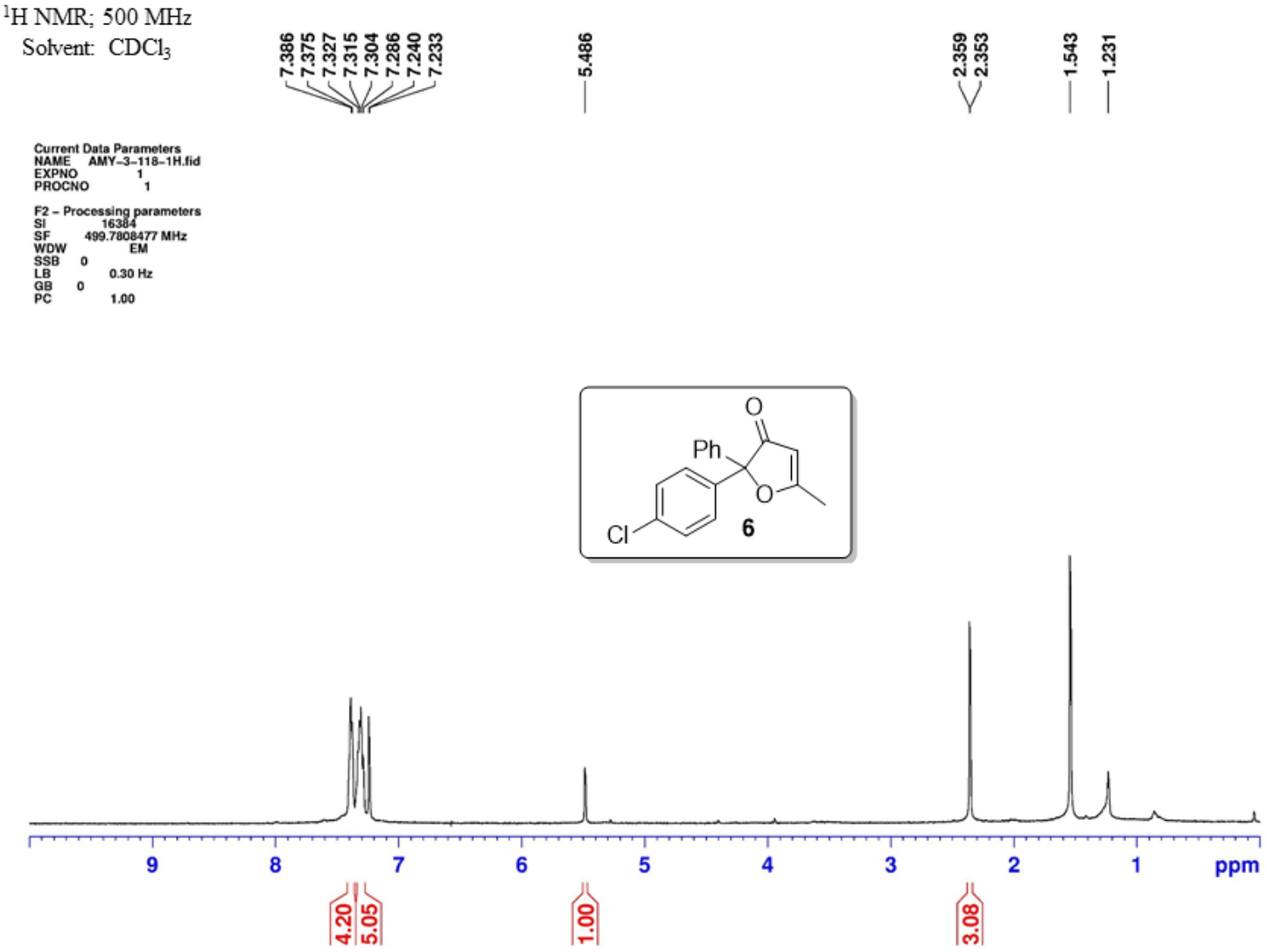


^{1}H NMR; 700 MHz
Solvent: $CDCl_3$

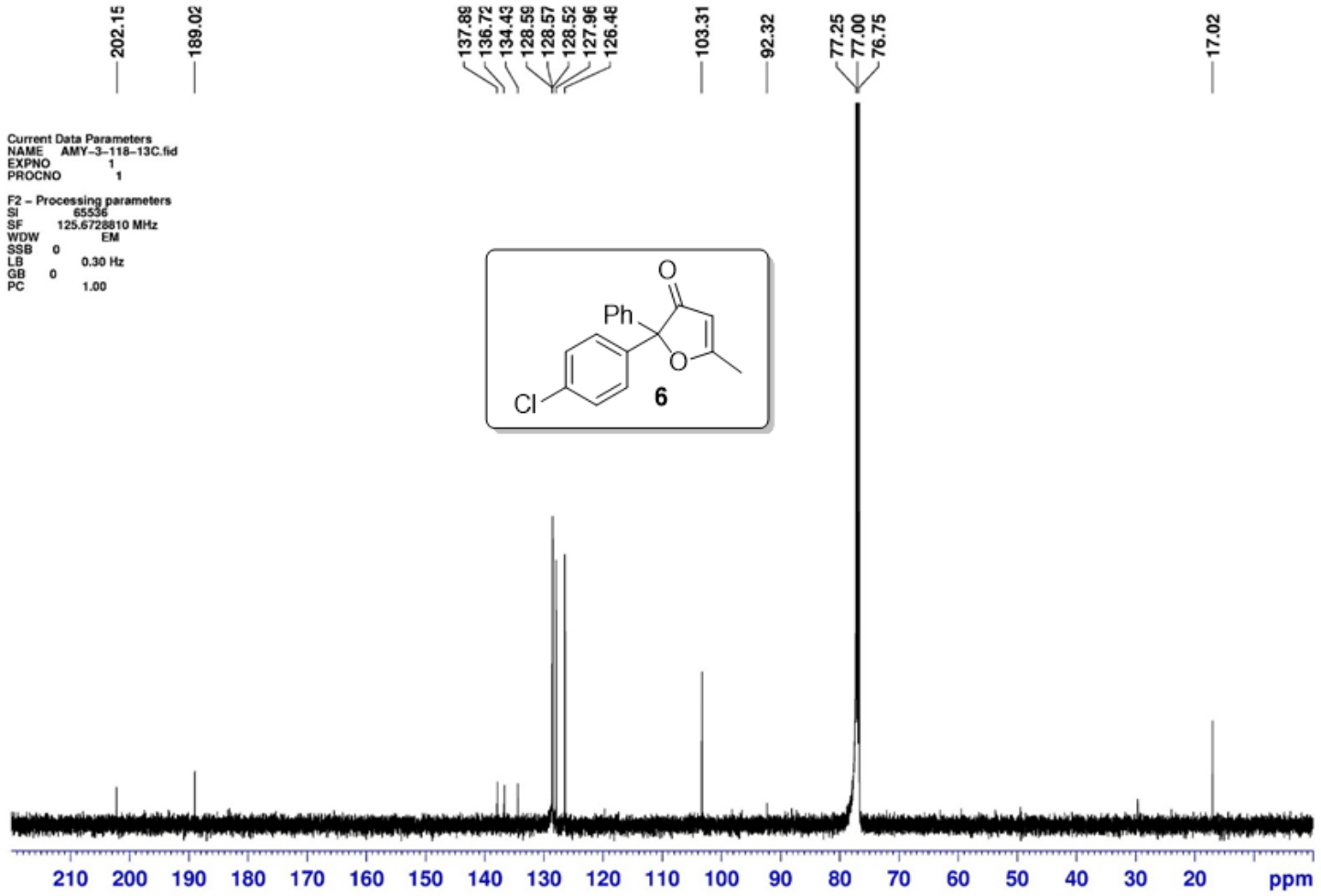



^{13}C NMR; 175 MHz
Solvent: $CDCl_3$
143.97
141.48
128.70
128.49
128.45
128.29
127.24
127.15
126.51
125.49
90.96
77.18
77.00
76.82
76.33
65.18
33.38
Current Data Parameters
NAME AMY-3-175-13C
EXPNO 3
PROCNO 1
F2 – Processing parameters
SI 131072
SF 175.9532218 MHz
WDW EM
LB 0.30 Hz
GB 0
PC 1.00
H
OH
Ph
Ph
O
5
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

^{1}H NMR; 500 MHz
Solvent: $CDCl_3$

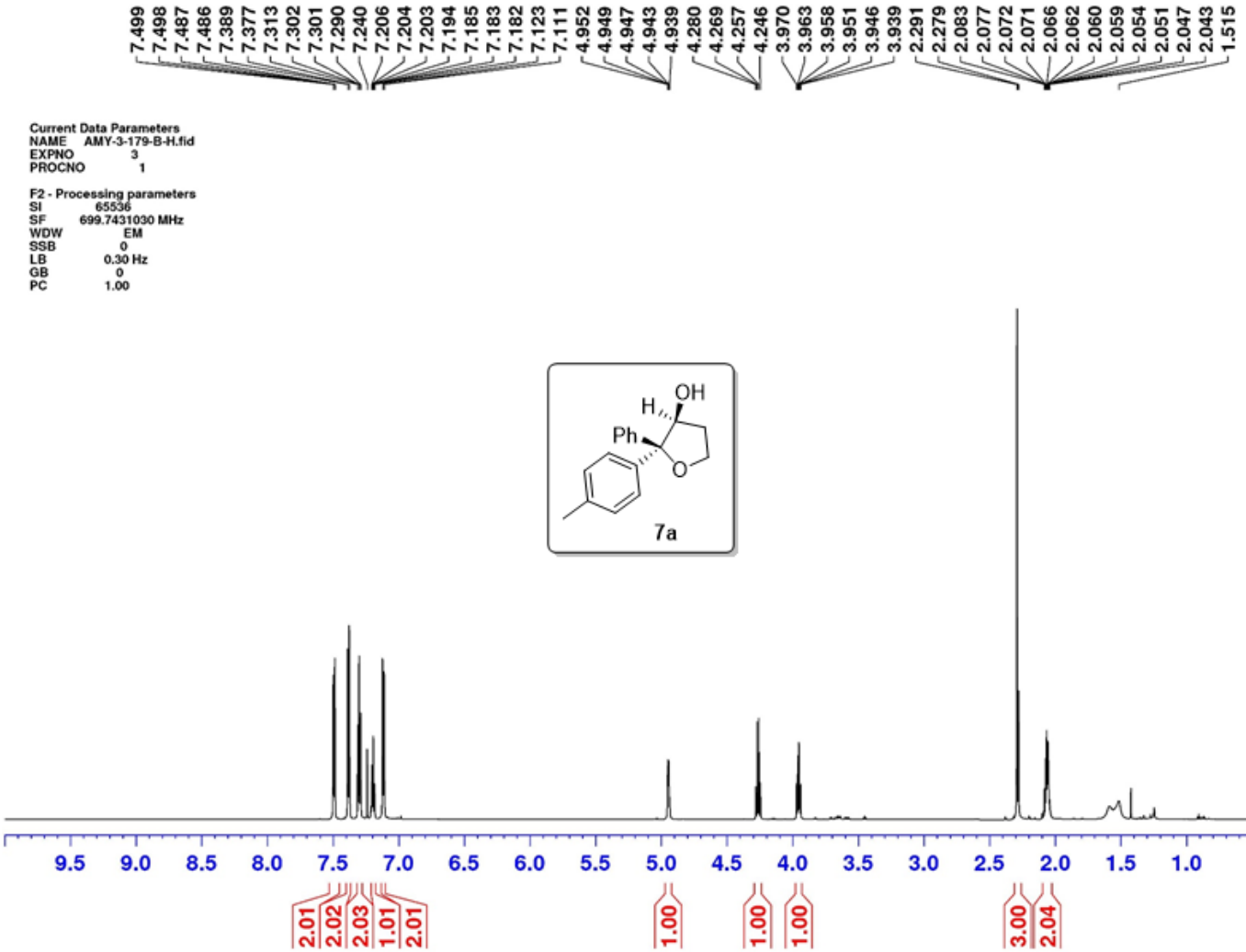


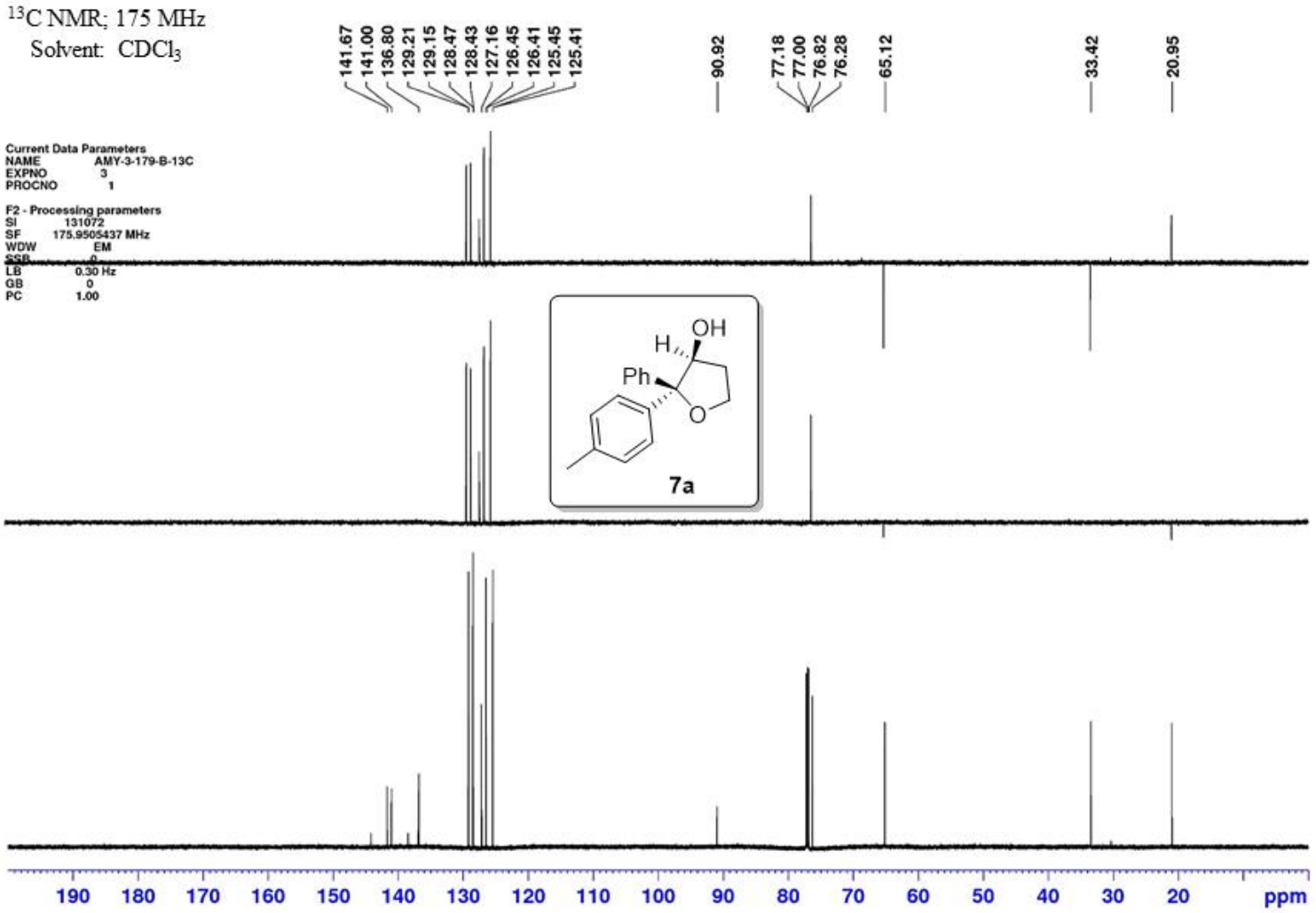
^{13}C NMR; 125 MHz
Solvent: $CDCl_3$



^{1}H NMR; 700 MHz

Solvent: $CDCl_3$




^{13}C NMR; 175 MHz
Solvent: $CDCl_3$
141.67
141.00
136.80
129.21
129.15
128.47
128.43
127.16
126.45
126.41
125.45
125.41
90.92
77.18
77.00
76.82
76.28
65.12
33.42
20.95
Current Data Parameters
NAME AMY-3-179-B-13C
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505437 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
H
Ph
O
7a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

^{1}H NMR; 700 MHz
Solvent: $CDCl_3$

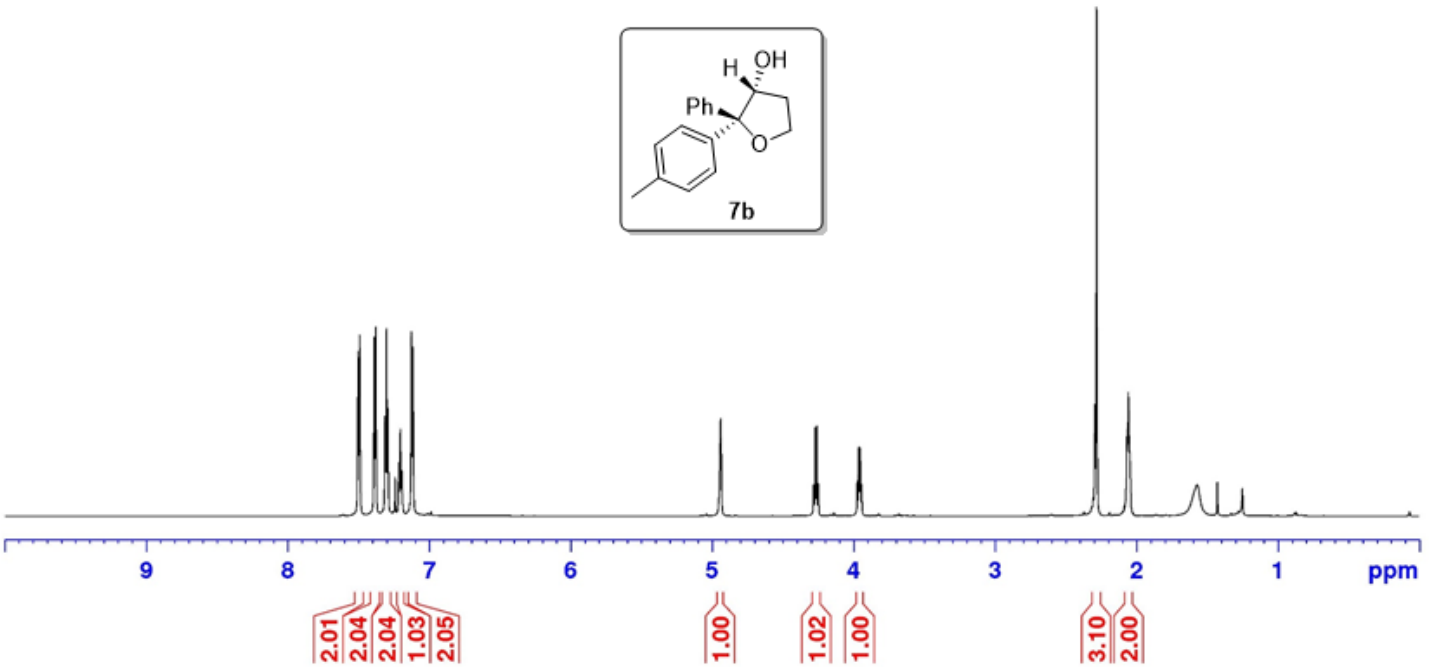
7.503 7.492 7.389 7.378 7.314 7.303 7.292 7.240 7.214 7.204 7.194 7.126 7.115

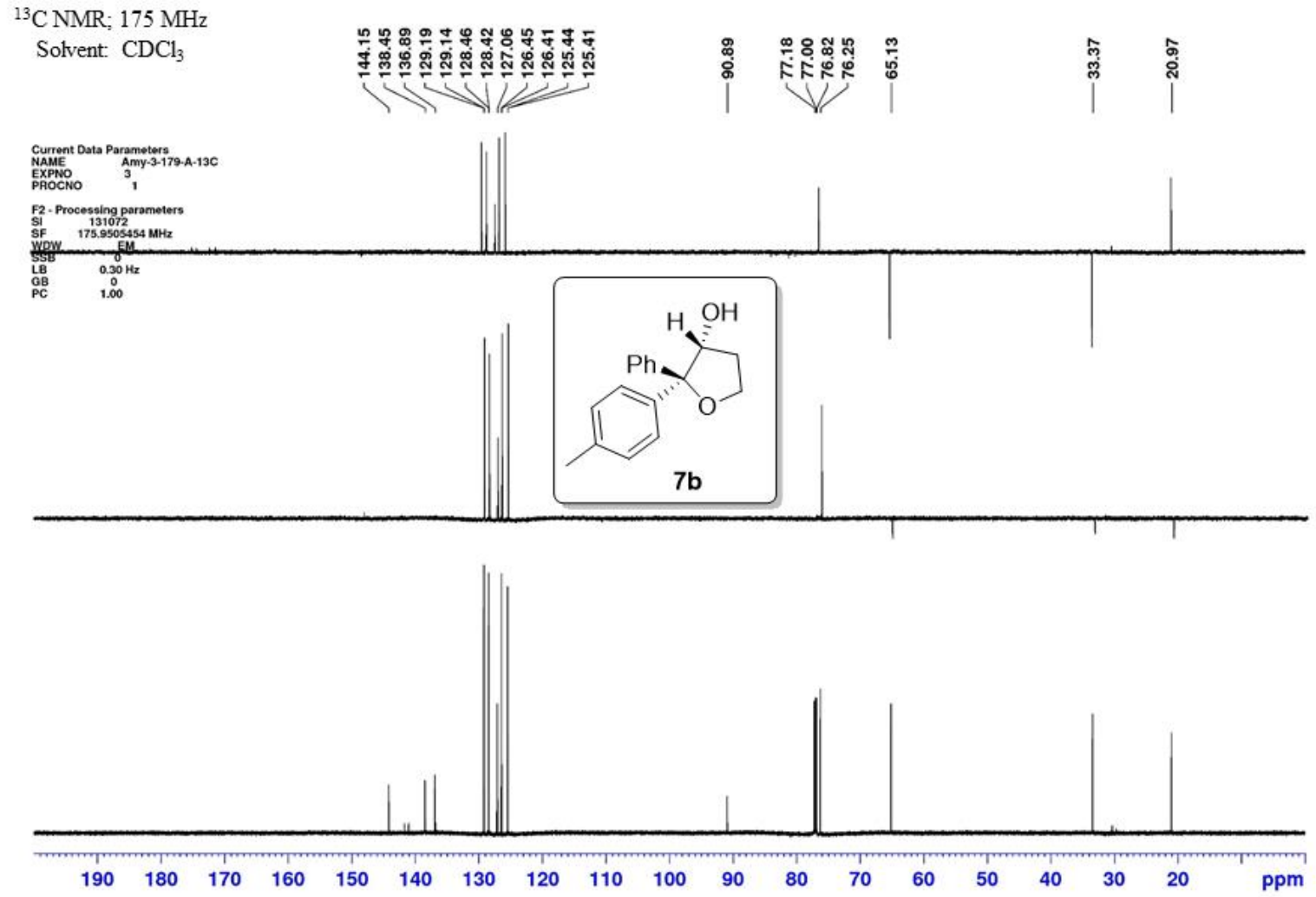
4.944 4.940 4.283 4.271 4.260 4.249 3.973 3.962 3.953 3.942

2.294 2.282 2.068 2.063 2.057 2.052 2.048 1.571

Current Data Parameters
NAME AMY-3-179-A-H.fid
EXPNO 3
PROCNO 1

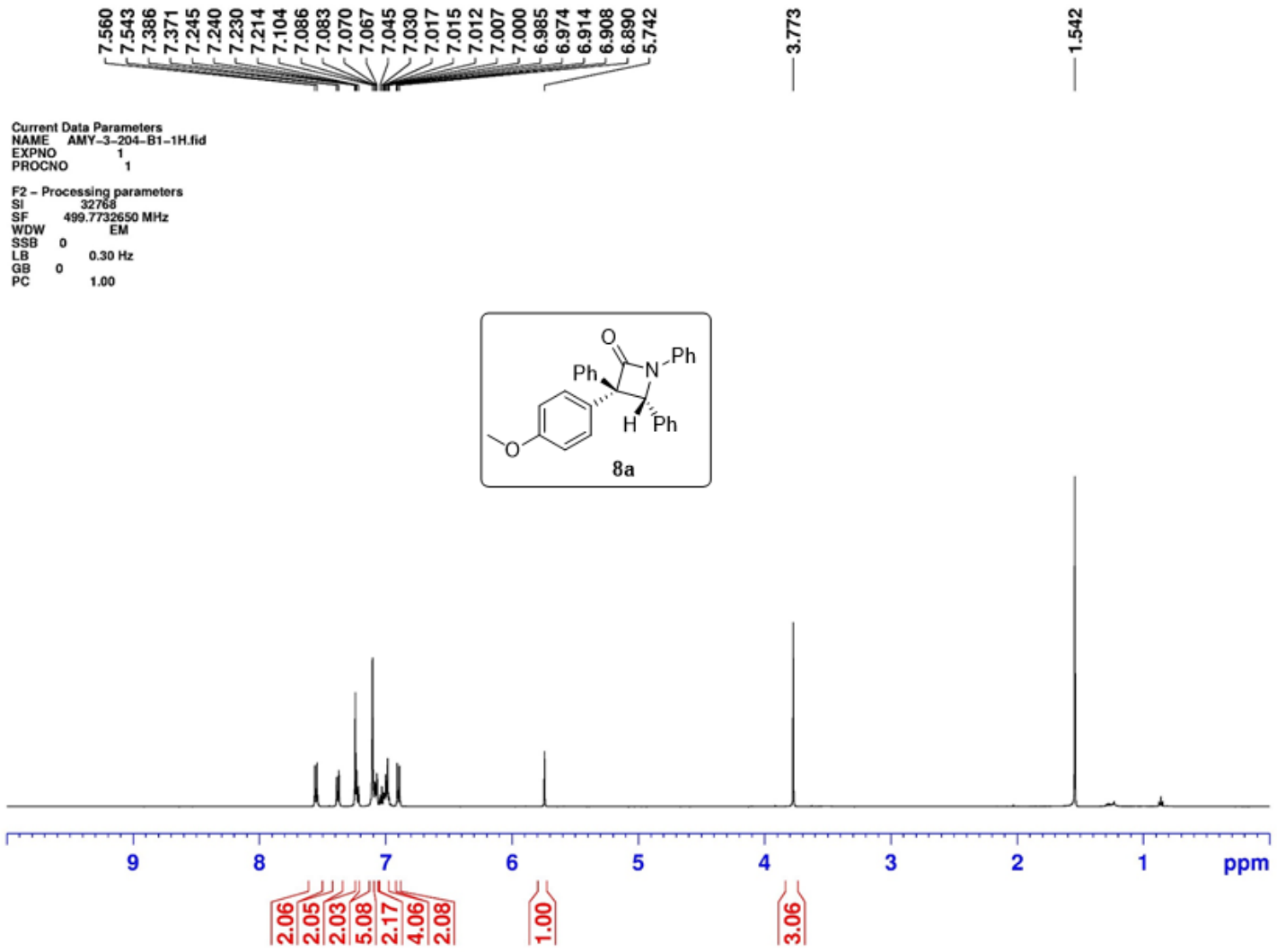
F2 - Processing parameters
SI 65536
SF 699.7431036 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

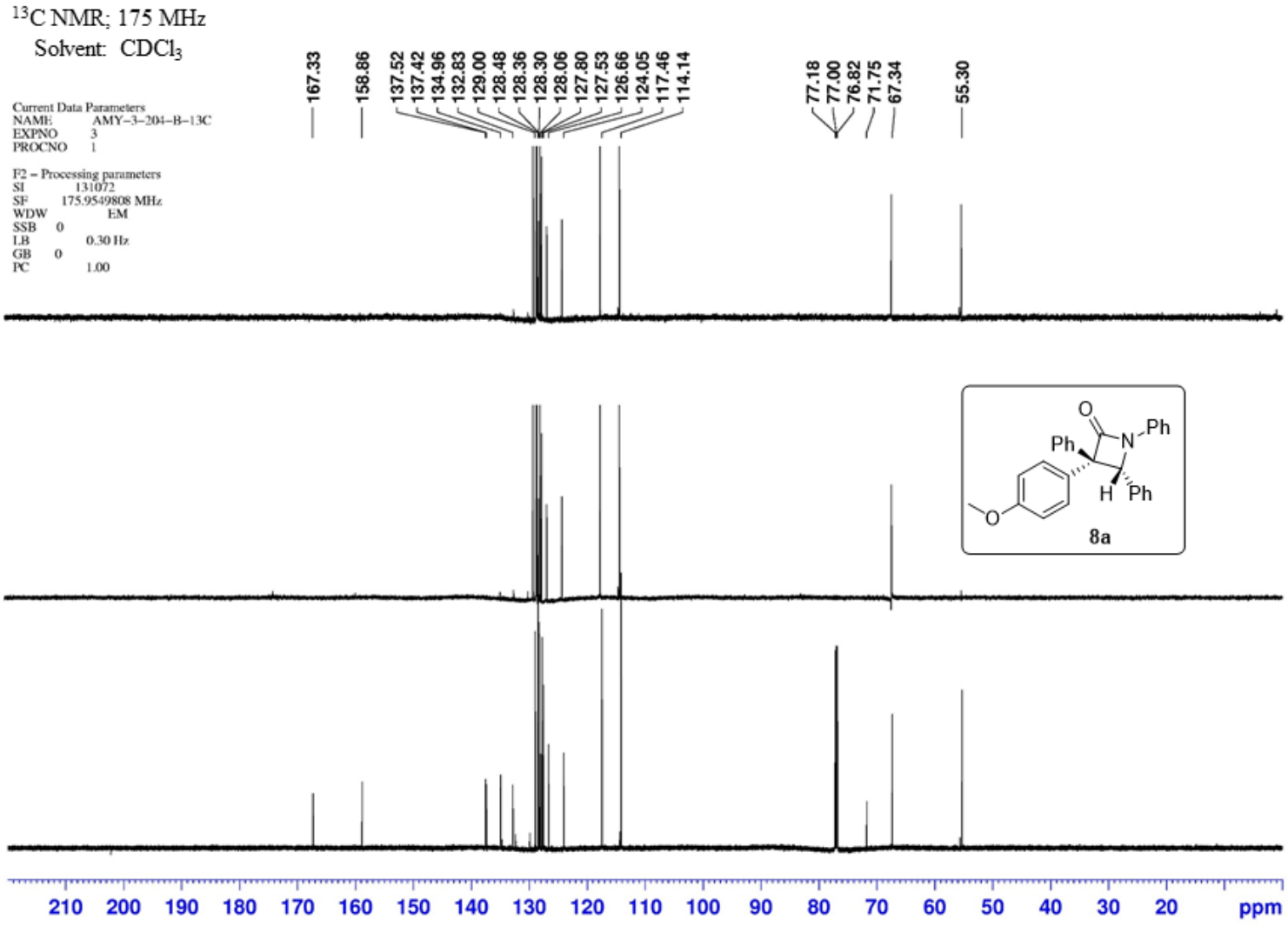



^{13}C NMR; 175 MHz
Solvent: $CDCl_3$
144.15
138.45
136.89
129.19
129.14
128.46
128.42
127.06
126.45
126.41
125.44
125.41
90.89
77.18
77.00
76.82
76.25
65.13
33.37
20.97
Current Data Parameters
NAME Amy-3-179-A-13C
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505454 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H
OH
Ph
O
7b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

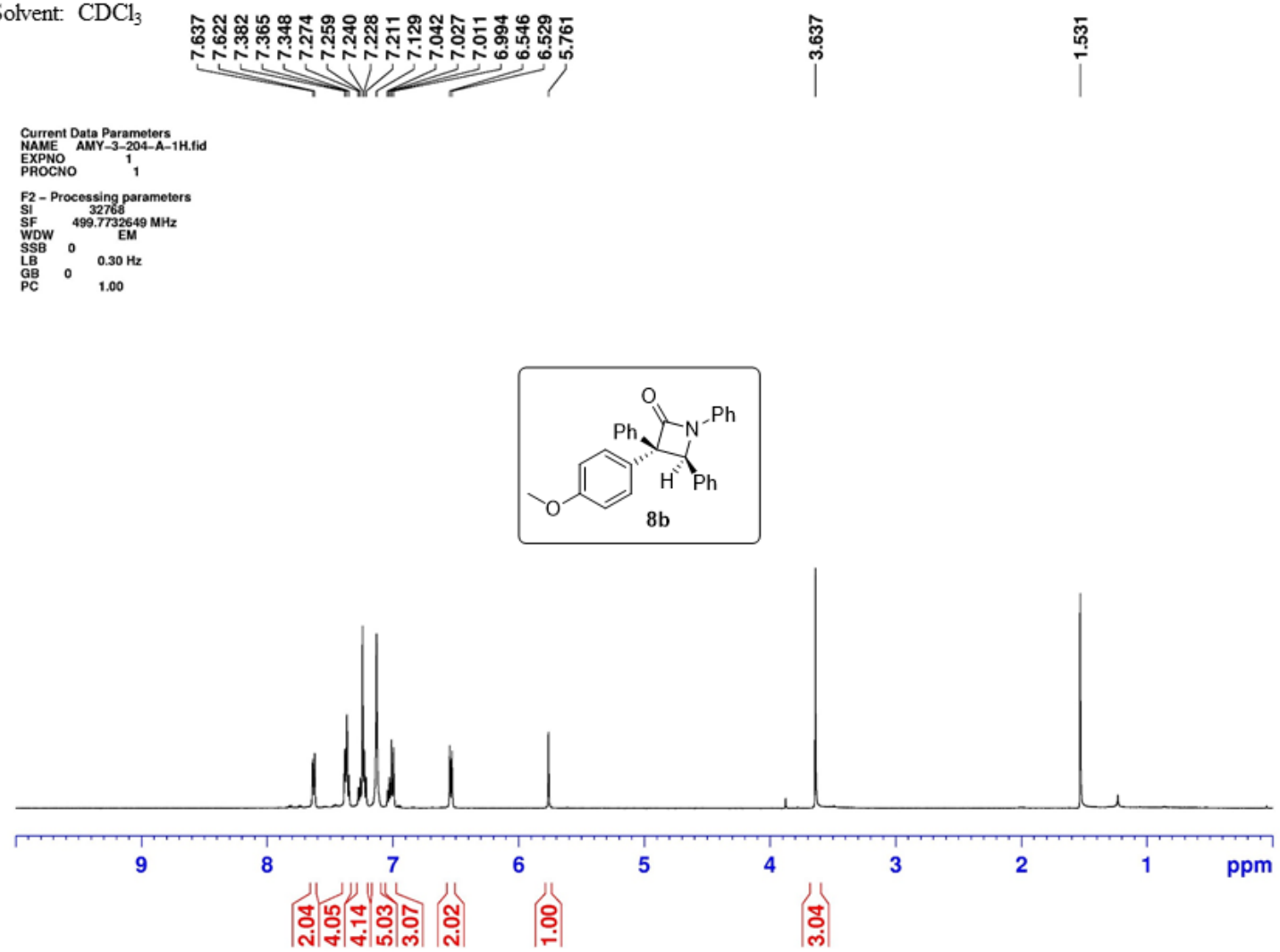
^{1}H NMR; 500 MHz

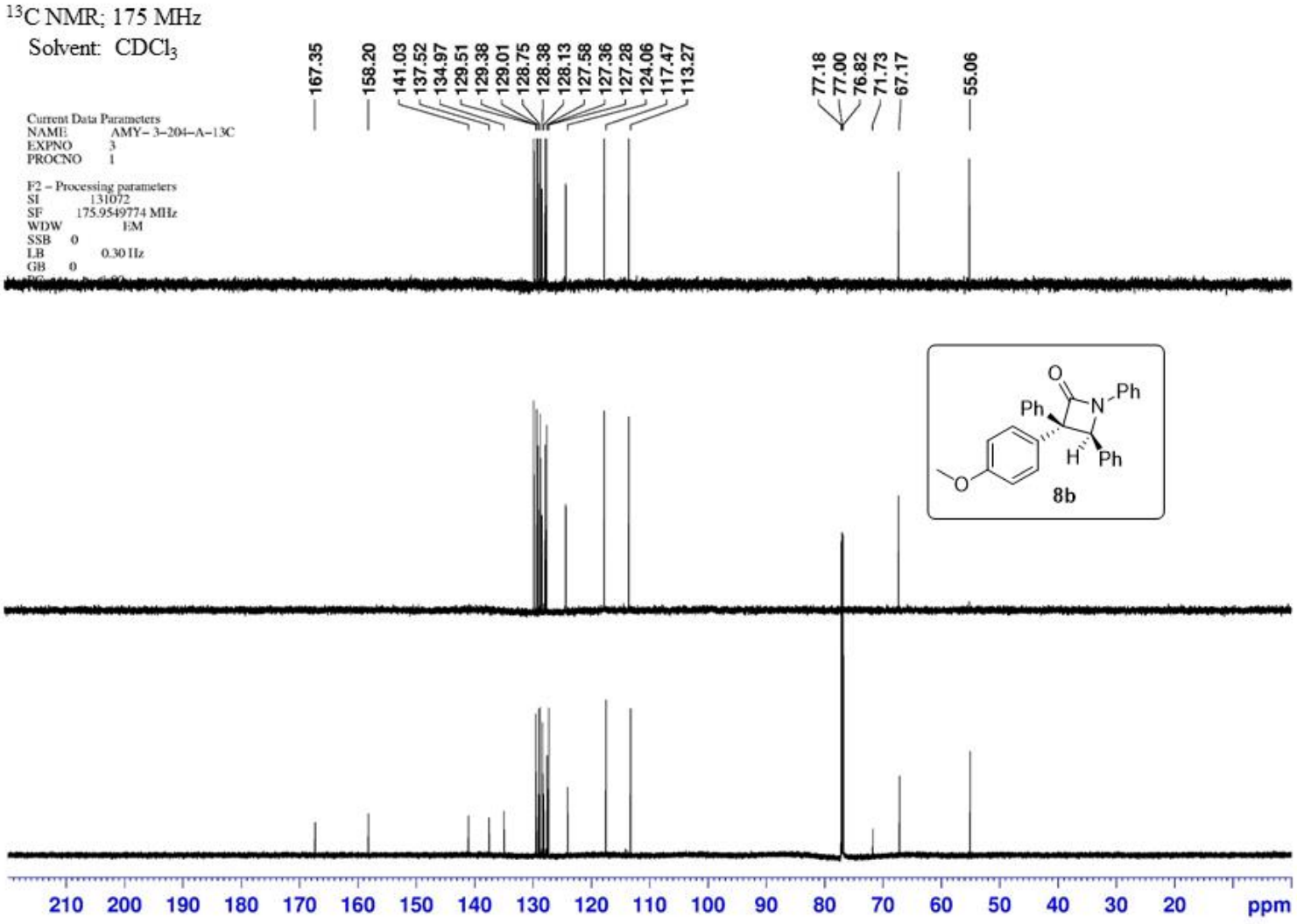
Solvent: $CDCl_3$




13C NMR; 175 MHz
Solvent: CDCl3
Current Data Parameters
NAME AMY-3-204-B-13C
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9549808 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
167.33
158.86
137.52
137.42
134.96
132.83
129.00
128.48
128.36
128.30
128.06
127.80
127.53
126.66
124.05
117.46
114.14
77.18
77.00
76.82
71.75
67.34
55.30
O
N
Ph
Ph
H
Ph
O
8a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

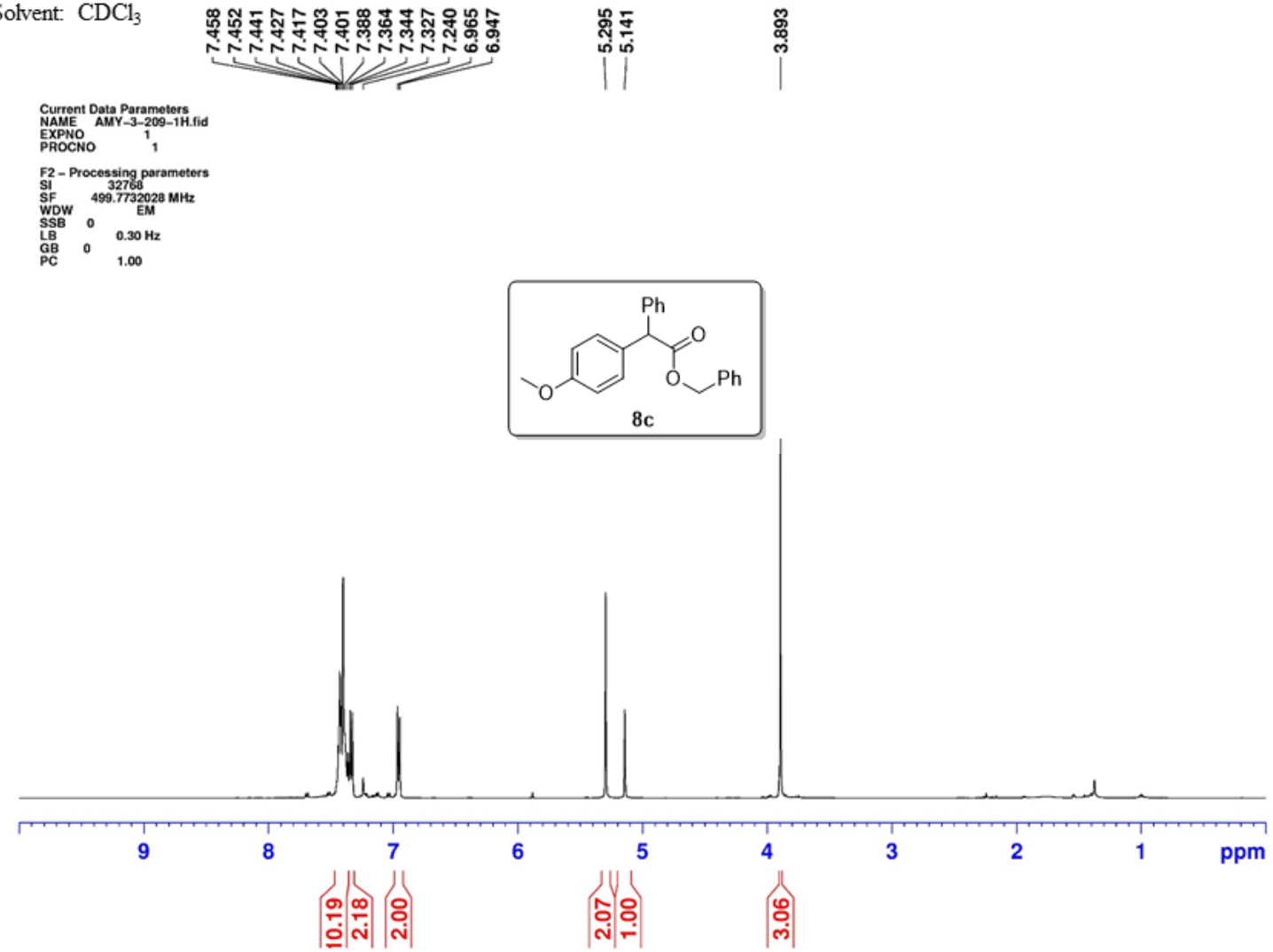
^{1}H NMR; 500 MHz
Solvent: $CDCl_3$




13C NMR; 175 MHz
Solvent: CDCl3
167.35
158.20
141.03
137.52
134.97
129.51
129.38
129.01
128.75
128.38
128.13
127.58
127.36
127.28
124.06
117.47
113.27
77.18
77.00
76.82
71.73
67.17
55.06
Current Data Parameters
NAME AMY-3-204-A-13C
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9549774 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
O
Ph
N
Ph
Ph
H
O
8b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

^{1}H NMR; 500 MHz

Solvent: $CDCl_3$



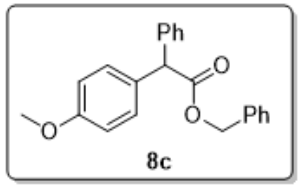
^{13}C NMR; 125 MHz

Solvent: $CDCl_3$

Current Data Parameters
NAME AMY-3-209-13C.fid
EXPNO 1
PROCNO 1

F2 – Processing parameters
SI 65536
SF 125.6698772 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

172.56 158.72 138.87 135.68 130.65 129.68 128.53 128.47 128.46 128.18 128.13 127.15 113.92 77.25 77.00 76.75 66.83 56.16 55.22



8c