Stereo- and Regiocontrol in Intermolecular [2+2] Cycloadditions between Diarylketenes and Allenamides to Access Substituted α-Methylenecyclobutanones.

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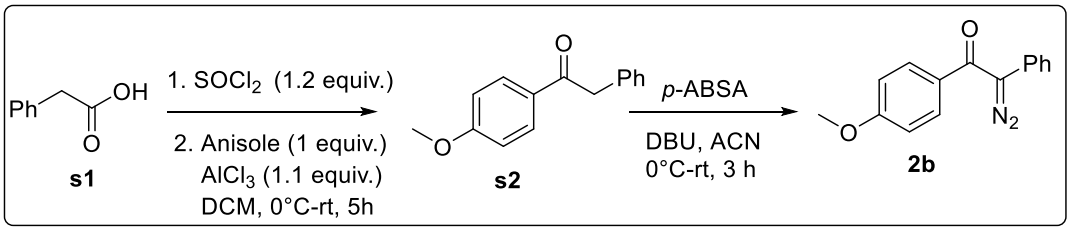
Content

1. (a) 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 CaH2 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 13C NMR spectra were recorded on a Bruker 500, 600 MHz and Varian 500,700 MHz spectrometers using chloroform-d (CDCl3) 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 $I > 2\sigma(I)$.

(b) General synthetic procedures for preparation of diazo ketones[s1] and allene substrates[s2]

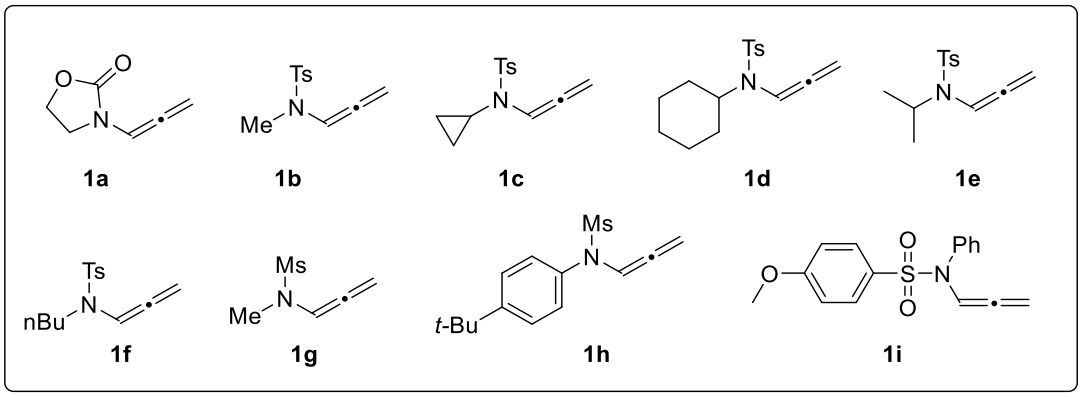


To a DCM (60 ml) solution of substituted phenylacetic acid **(s1)** (5.0 g, 36.72 mmol) was added $SOCl_2$ (3.20 ml, 44.07 mmol) dropwise at 0 °C. The mixture was warm to room temperature and stirred for 2 h. The resulting solution was cooled to 0 °C, followed by the addition of $AlCl_3$ (5.39 g, 40.40 mmol) and Anisole (3.99 ml, 36.72 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 = 10/90) to afford 1-(4-methoxyphenyl)-2-phenylethanone **(s2)** white solid (7.0 g, 30.94 mmol,

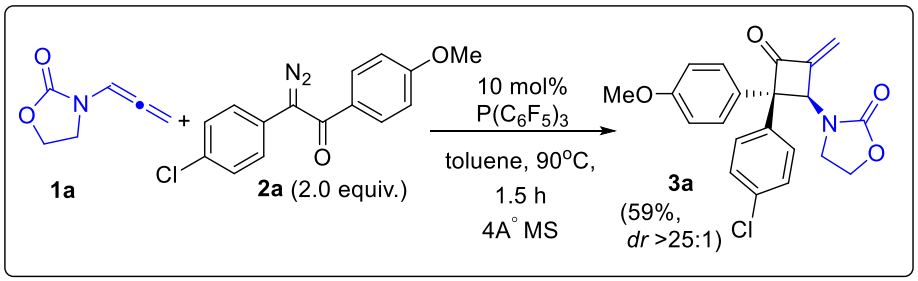
84%). To an acetonitrile (23 ml) solution of 1-(4-methoxyphenyl)-2-phenylethanone **(s2)** (3.0 g, 13.26 mmol) was added p-ABSA (3.5 g, 14.59 mmol). The solution was cooled to 0 °C and DBU (2.34 ml, 17.24 mmol) was added dropwise to the above mixture and stirred at room temperature for 3 h. The reaction was quenched with water, followed by extraction with ether (2 x 100 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 **(2b)** (2.4 g, 9.51 mmol, 72%) as a yellow solid.

All diazo ketones **(2a – 2m)** were synthesized according to the our recently reported literature above procedure [S1]

All the allenamide **(1a-1i)** substrates were known compounds and synthesized according to related reported procedures of literature. [S2]



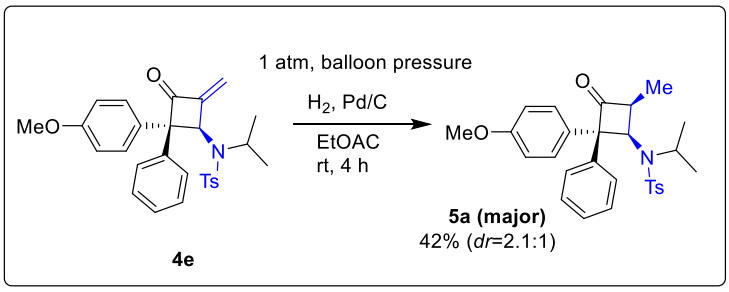
2. Standard catalytic reaction procedure:



A suspension of $P(C_6F_5)_3$ (21.4 mg, 0.040 mmol) in dry toluene (1 mL) was fitted with N_2 balloon. To this solution was added a toluene (1 mL) solution of 3-(propa-1,2-dien-1-yl)oxazolidin-2-one **1a** (50 mg, 0.402 mmol) and 2-(4-chlorophenyl)-2-diazo-1-(4-methoxyphenyl)ethan-1-one **2a** (231 mg, 0.805 mmol) at room temperature. After addition reaction mixture was stirred in pre-heated oil bath 90°C for 1.5 h, reaction progress was monitored by TLC, after completion then reaction mixture was filtered over a short celite bed, concentrated, and purified through a silica column using ethyl acetate/hexane (30: 70) as the eluent to afforded compound 3-((1R,2R)-2-(4-chlorophenyl)-2-(4-methoxyphenyl)-4-methylene-3-oxocyclobutyl)oxazolidin-2-one **3a** (92 mg, 0.239 mmol, 59%) as a pale yellow oil.

3. Synthetic procedures for chemical functionalizations:

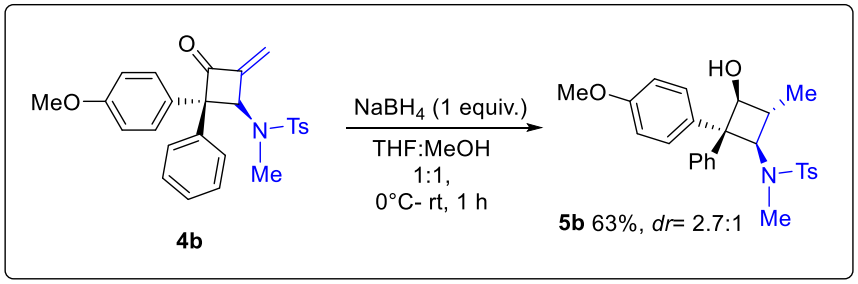
(a) Synthesis of N-isopropyl-N-((1R,2S,4S)-2-(4-methoxyphenyl)-4-methyl-3-oxo-2-phenylcyclobutyl)-4-methylbenzenesulfonamide (5a):



A suspension of **4e** (50 mg, 0.105 mmol) in an ethyl acetate (2 mL) was degassed with N_2 for 10min. To this solution Palladium on carbon (Pd/C, 30.0 mg, 10%Wt), was added then reaction vessel was evacuated and then fitted with H_2(g) balloon (1 atm) and reaction was stirred at room temperature for 4 h. Reaction progress was monitored by TLC, after completion then reaction mixture was filtered over a short celite bed, concentrated, and purified through a silica column using ethyl acetate/hexane (10: 90) as the eluent to afford N-

isopropyl-N-((1R,2S,4S)-2-(4-methoxyphenyl)-4-methyl-3-oxo-2-phenylcyclobutyl)-4-methylbenzenesulfonamide **5a** (57 mg, 0.043 mmol, 42%, *dr*=2.1:1) as a colourless oil.

(b) Synthesis of N-((1R,2S,3S,4R)-3-hydroxy-2-(4-methoxyphenyl)-4-methyl-2-phenylcyclobutyl)-N,4-dimethylbenzenesulfonamide (5b):



A suspension of **4b** (50 mg, 0.111 mmol) in an Methanol (1 mL): THF (1mL) was cooled to 0°C then $NaBH_4$ (4.23 mg, 0.111mmol) was added, then reaction mixture was stirred at 0°C for 1 h. Reaction progress was monitored by TLC, after completion then reaction mixture was quenched with saturated solution of NH_4Cl (2mL) and extracted with ethyl acetate (2X10mL) dried over sodium sulfate, concentrated, and purified through a silica column using ethyl acetate/hexane (30: 70) as the eluent to afford N-((1R,2S,3S,4R)-3-hydroxy-2-(4-methoxyphenyl)-4-methyl-2-phenylcyclobutyl)-N,4-dimethylbenzenesulfonamide **5b** (31 mg, 0.070 mmol, 63% *dr*=2.7:1) as a colourless oil.

2. References:

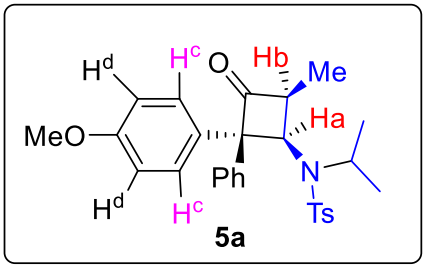
[S1] (a) A. V. Sasane and R.-S Liu, *Chem. Commun.*, 2023, **59**, 1086-1089; (b) S. A. More, V. A. Sadaphal, T-C Kuo, M-J Cheng and R.-S Liu, *Chem. Commun.*, 2022, **58**, 10064–10067; (c) C. N. Chen, W. M. Cheng, J. K. Wang, T. H. Chao, M. J. Cheng and R.-S. Liu, *Angew. Chem. Int. Ed.* 2021, **60**, 4479-4484; (d) Q. Lu, S. Mondal, S. Cembellín and F. Glorius, *Angew. Chem. Int. Ed.* 2018, **57**, 10732- 10736.

[S2] (a) J. He, L. Yang, X. Zhang, W. Xu, H. Wang, M. Lang, J. Wang and S. Peng, *ACS Catal.*, 2022, **12**, 23, 14647–14653; (b) Y. B. Pandit and R.-S. Liu., *Org. Lett.*, 2022, ***24*** (2), 548-553; (c) R. R. Singh, S. K. Pawar, M.-J. Huang and R.-S. Liu, *Chem. Commun.*, 2016, **52**, 11434-11437; (d) S.-P. Samuel, C. Hernandez-Diaz, M. Piedrafita, E. Rubio and J. M. Gonzalez., *Adv. Synth. Catal.* 2012, **354**, 1651−1657; (e) Suárez-Pantiga, Samuel, C. Hernández-Díaz, E. Rubio,

and J. M. González., *Angew. Chem., Int. Ed.* 2012, **51**, 11552−11555; (f) Yang, Xiaoyu, and F. D. Toste., *Chem. Sci.* 2016, **7**, 2653−2656; (g) I. Chakrabarty, S. M. Inamdar, M. O. Akram, A. B. Gade, S. Banerjee, S. Berac and N. T. Patil., *Chem. Commun.*, 2017, **53**, 196−199; (h) H.-H. Li, X.-X. Li, Z.-G. Zhao, X. Yuan and C.-Y. Sun., *Org. Biomol. Chem.*, 2017, **15**, 4005−4013; (i) H. Fuwa and M. A Sasaki., *Org. Biomol. Chem.* 2007, **5**, 2214−2218; (j) W.-F. Zheng, G.-J. Sun, L. Chen and Q. Kang., *Adv. Synth. Catal.*, 2018, **360**, 1790−1794; (k) R. Blieck, L. A. Perego, I. Ciofini, L. Grimaud, M. Taillefer and F. Monnier., *Synthesis* 2019, **51**, 1225−1234; (l) L.-L. Wei, J. A. Mulder, H. Xiong, C. A. Zificsak, C. J. Douglas and R. P. Hsung., *Tetrahedron* 2001, **57**, 459−466; (m) S. Peng, S. Cao and J. Sun., *Org. Lett.*, 2017, **19**, 524−527; (n) S. Guo, J. Chen, M. Yi, L. Dong, A. Lin and H. Yao., *Org. Chem. Front.*, 2021, **8**, 1783−1788; (o) L. Rout and A. M. Harned., *Chem. Eur. J.*, 2009, **15**, 12926−12928; (p) R. Kuppusamy, R. Santhoshkumar, R. Boobalan, H.-R. Wu and C.-H. Cheng., *ACS Catal.*, 2018, **8**, 1880−1883; (q) X. Huang, W. Thor, X. Feng, L. Kang, M. Yang, C.-S. Lee, Y.-K. Cheng and S. He., *Org. Chem. Front.*, 2020, **7**, 255−260; (r) Y. Liu, Z. Yi, X. Yang, H. Wang, C. Yin, M. Wang, X.-Q. Dong and X. Zhang., *ACS Catal.*, 2020, **10**, 11153−11161.

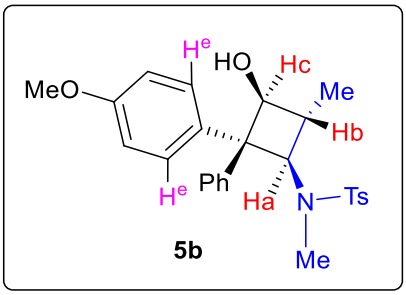
5. NOE of compound 5a and 5b

(a) NOE for compound 5a:



Sr. No.	Irradiation	Intensity increase % (Key peaks)
1.	Me (δ 1.1)	H^b (δ 3.5, 1.48%), H^a (not affected)
2.	H^a (δ 4.8)	H^b (δ 7.35, 0.63%), H^c (δ 7.25, 4.18%)
3.	H^b (δ 3.5)	H^a (δ 4.8, 0.77%), Me (1.1, 2.66%) and (isopropyl group affected)
4.	H^c (δ 7.25)	H^d (δ 6.79, 3.86%), H^a (δ 4.8, 2.94%)

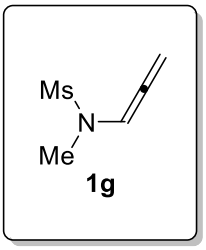
(b) NOE for compound 5b:



Sr. No.	Irradiation	Intensity increase % (Key peaks)
1.	Me (δ 0.6)	H^a (δ 4.2, 1.03%), H^c (δ 3.7, 1.09%), H^b (δ 2.6, 1.77%) (H^a, H^c and H^c all affected, cis to Me group)
2.	H^a (δ 4.2)	H^c (δ 3.7, 2.63%), Me (δ 0.6, 1.88%), H^b (δ 2.6, very less affected, 0.60%), H^e (δ 7.4, 3.93%)
3.	H^b (δ 2.6)	H^a (δ 4.2, 0.72%) and H^c (3.7, 0.75%) (very less affected) Me (δ 0.6, 3.27 %)

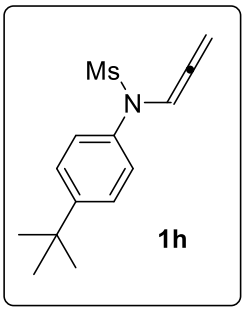
6. Spectral Data:

Spectral data for N-methyl-N-(propa-1,2-dien-1-yl)methanesulfonamide (1g)



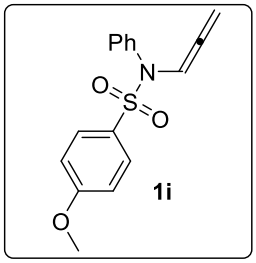
(Compound **1i**) was purified on silica gel column using ethyl acetate/hexane: (10: 90) as the eluent; brown oil (300 mg, 2.04 mmol, 75%);^{1}H NMR (400 MHz, $CDCl_3$): 6.70 ~ 6.67 (m, 1H), 5.37 (d, *J* = 10.5 Hz, 2H), 2.84 (s, 3H), 2.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$):200.7, 101.0, 87.8, 35.5, 33.0; HRMS-ESI+ m/z: $[M+Na]^+$ calcd $C_5H_9NO_2S$: 170.02517; found: 170.02518.

Spectral data for N-(4-(tert-butyl)phenyl)-N-(propa-1,2-dien-1-yl)methanesulfonamide (1h)



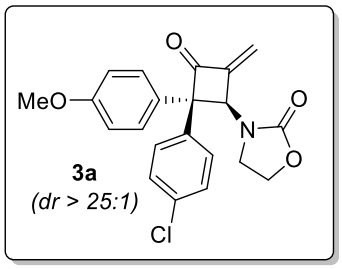
(Compound **1h**) was purified on silica gel column using ethyl acetate/hexane: (10: 90) as the eluent; brown oil (350 mg, 1.32 mmol, 70%);^{1}H NMR (400 MHz, $CDCl_3$):7.40 ~ 7.37 (m, 2H), 7.25 ~ 7.23 (m, 2H), 6.90 (t, *J* = 11.2 Hz, 1H), 5.15 (d, *J* = 11.2 Hz, 2H), 2.98 (s, 3H), 1.30 (s, 9H); ^{13}C NMR (175 MHz, $CDCl_3$): 201.0, 151.8, 134.5, 128.4, 126.1, 101.7, 87.5, 38.0, 34.6, 31.2, four carbons are merged; HRMS-ESI- m/z: [M-H] calcd $C_{14}H_{19}NO_2S$: 264.10582; found: 264.10616.

Spectral data for 4-methoxy-N-phenyl-N-(propa-1,2-dien-1-yl)benzenesulfonamide(1i)



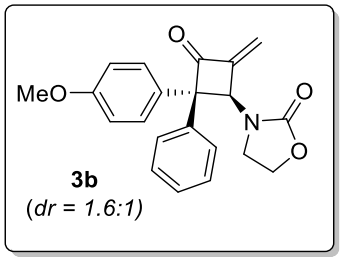
(Compound **1i**) was purified on silica gel column using ethyl acetate/hexane: (10: 90) as the eluent; brown oil (280 mg, 0.929 mmol, 56%); ^{1}H NMR (700 MHz, $CDCl_3$): 7.56 (d, *J* = 9.1 Hz, 2H), 7.27 ~ 7.23 (m, 3H), 7.06 (t, *J* = 6.3 Hz, 1H), 6.98 ~ 6.97 (m, 2H), 6.91 (d, *J* = 9.1 Hz, 2H), 4.99 (d, *J* = 6.3 Hz, 2H), 3.83 (s, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): 201.0, 163.1, 137.2, 129.5, 128.6, 128.5, 113.9, 102.3, 87.3, 55.5, six carbons are merged; HRMS-ESI+ m/z: $[M+Na]^+$ calcd $C_{16}H_{15}NO_3S$: 324.06703; found: 324.06727.

Spectral Data for 3-((*1R,2R*)-2-(4-chlorophenyl)-2-(4-methoxyphenyl)-4-methylene-3-oxocyclobutyl)oxazolidin-2-one (3a):



(Compound **3a**, *diastereomeric ratio > 25:1*) was purified on silica gel column using ethyl acetate/hexane: (25: 75) as the eluent; pale yellow oil (92 mg, 0.239 mmol, 59%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.62 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.28 (s, 1H), 5.85 (s, 1H), 5.44 (s, 1H), 4.17 ~ 4.13 (m, 1H), 3.81 ~ 3.76 (m, 1H), 3.74 (s, 3H), 3.30 ~ 3.26 (m, 1H), 2.27 ~ 2.25 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ; 197.3, 158.9, 158.4, 149.6, 136.9, 133.6, 131.9, 129.2, 128.25, 128.21, 120.3, 114.4, 77.6, 62.5, 57.9, 55.2, 41.2, four carbons are merged with aromatic region; HRMS (FD) m/z: $[M]^+$ calcd for $C_{21}H_{18}ClNO_4$: 383.09298; found: 383.09315.

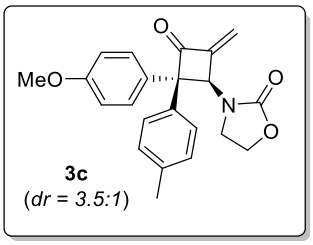
Spectral Data for 3-((*1R,2S*)-2-(4-methoxyphenyl)-4-methylene-3-oxo-2-phenylcyclobutyl)oxazolidin-2-one (3b):



Compound **3b** (*diastereomeric ratio 1.6:1*) was purified on silica gel column using ethyl acetate/hexane: (30: 70) as the eluent; brown oil (86.0 mg, 0.246 mmol, 60%);^{1}H NMR (700 MHz, $CDCl_3$) for major isomer: 7.66 (d, *J* = 7.1 Hz, 2H), 7.29~7.26 (m, 2H), 7.24 (d, *J* = 7.7 Hz, 2H), 7.21 ~ 7.16 (m, 3H), 6.86 (d, *J* = 8.4 Hz, 2H), 6.26 (s, 1H), 5.86 (s, 1H), 5.41 (s, 1H), 4.14 ~ 4.07 (m, 1H), 3.74 (s, 3H), 3.71 ~ 3.67 (m, 1H), 3.26 ~ 3.20 (m, 1H), 2.15 ~ 2.11 (m, 1H).;^{1}H NMR (700 MHz, $CDCl_3$) for minor isomer:7.72 (d, *J* = 7.7 Hz, 2H), 7.32 (t, *J* = 8.4 Hz, 2H), 6.82 (d, *J* = 9.1 Hz, 2H), 5.89 (s, 1H), 3.80~3.76 (m, 1H), 2.24 ~ 2.20 (m, 1H) rest of peaks are merged.; ^{13}C NMR (175 MHz, $CDCl_3$) for major isomer: 197.90, 158.7, 158.42, 149.94, 140.4, 138.3, 132.3, 130.0, 129.0, 128.8, 128.3, 128.0, 127.4, 127.1, 127.0, 126.8, 119.71, 114.2, 78.3, 62.51, 57.9, 55.23, 41.08.;^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer: 197.97, 158.9, 158.42, 149.96,

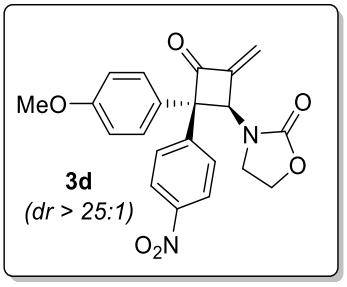
119.74, 114.46, 78.4, 62.59, 57.6, 55.23, 41.1, rest of carbons are merged.; HRMS-ESI+ m/z: $[M+Na]^+$ calcd for $C_{21}H_{19}NO_4$: 372.12118; found: 372.12115.

Spectral Data for 3-((*1R,2S*)-2-(4-methoxyphenyl)-4-methylene-3-oxo-2-(*p-tolyl*)cyclobutyl)oxazolidin-2-one (3c):



Compound **3c** (*diastereomeric ratio 3.5:1*) was purified on silica gel column using ethyl acetate/hexane: (25: 75) as the eluent; white solid (111 mg, 0.305 mmol, 76%); ^{1}H NMR (700 MHz, $CDCl_3$) for major isomer: 7.64 (d, *J* = 9.1 Hz, 2H), 7.16 ~ 7.11 (m, 3H), 7.08 (d, *J* = 8.4 Hz, 2H), 6.85 (d, *J* = 9.1 Hz, 2H), 6.25 ~ 6.24 (m, 1H), 5.84 (s, 1H), 5.40 (s, 1H), 4.13 ~ 4.08 (m, 1H), 3.78 ~ 3.71 (m, 6H), 3.26 ~ 3.21 (m, 1H), 2.25 (s, 3H), 2.21 ~ 2.19 (m, 1H).;^{1}H NMR (700 MHz, $CDCl_3$) for minor isomer: 7.59 (d, *J* = 8.4 Hz, 1H), 6.81 (d, *J* = 8.4 Hz, 1H), 5.86 (s, 1H), 2.26 (s, 3H), rest of peaks are merged with major isomer.;^{13}C NMR (175 MHz, $CDCl_3$) for major isomer: 198.1, 158.7, 158.45, 150.0, 137.2, 135.3, 132.6, 130.2, 129.7, 129.5, 128.2, 128.01, 126.8, 126.7, 119.5, 114.2, 78.09, 62.54, 57.8, 55.23, 41.09, 21.01;^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer: 158.8, 158.42, 136.9, 119.6, 114.4, 78.1, 62.5, 57.7, 55.22, 41.15, 20.9, rest of carbons are merged; HRMS-ESI+ m/z: $[M+Na]^+$ calcd for $C_{22}H_{21}NO_4$: 386.13683; found: 386.13653.

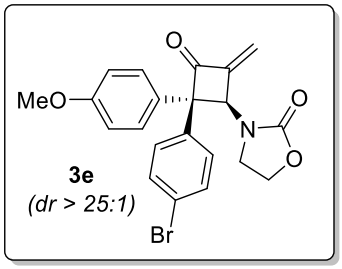
Spectral Data for 3-((*1R,2S*)-2-(4-methoxyphenyl)-4-methylene-2-(4-nitrophenyl)-3-oxocyclobutyl)oxazolidin-2-one (3d):



Compound **3d** (*diastereomeric ratio > 25:1*) was purified on silica gel column using ethyl acetate/hexane: (30: 70) as the eluent; yellow solid (90 mg, 0.228 mmol, 57%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.62 (d, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 8.4 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 6.86

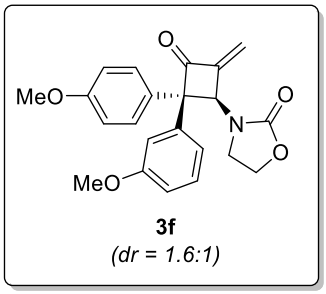
(d, *J* = 8.4 Hz, 2H), 6.28 (s, 1H), 5.85 (s, 1H), 5.44 (s, 1H), 4.17 ~ 4.13 (m, 1H), 3.82 ~ 3.79 (m, 1H), 3.74 (s, 3H), 3.30 ~ 3.26 (m, 1H), 2.29 ~ 2.26 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ; 197.3, 158.9, 158.4, 149.6, 137.4, 132.2, 131.8, 128.5, 128.2, 121.7, 120.3, 114.4, 77.6, 62.5, 57.9, 55.2, 41.2, rest of carbons are merged with aromatic region; HRMS (FD) m/z: $[M]^+$ calcd for $C_{21}H_{18}NO_6$: 394.11703; found: 394.11758.

Spectral Data for 3-((*1R,2R*)-2-(4-bromophenyl)-2-(4-methoxyphenyl)-4-methylene-3-oxocyclobutyl)oxazolidin-2-one (3e):



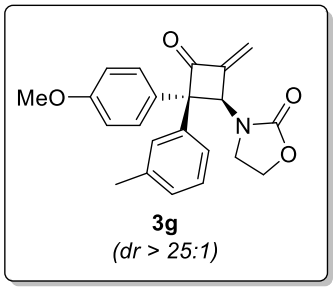
(Compound **3e**, *diastereomeric ratio > 25:1*) was purified on silica gel column using ethyl acetate/hexane: (25: 75) as the eluent; brown solid (79.9 mg, 0.186 mmol, 46%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.62 (d, *J* = 9.1 Hz, 2H), 7.42 (d, *J* = 8.4 Hz, 2H), 7.16 (d, *J* = 9.1 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 2H), 6.28 (dd, *J* = 2.1, 1.4 Hz, 1H), 5.85 (*t*, *J* = 2.8 Hz, 1H), 5.44 (dd, *J* = 1.4, 1.4 Hz, 1H), 4.17 ~ 4.13 (m, 1H), 3.83 ~ 3.78 (m, 1H), 3.74 (s, 3H), 3.30 ~ 3.26 (m, 1H), 2.29 ~ 2.26 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ; 197.3, 158.9, 158.4, 149.6, 137.4, 132.2, 131.8, 128.5, 128.2, 121.7, 120.3, 114.4, 77.6, 62.5, 57.9, 55.2, 41.2, rest of carbons are merged with aromatic region; HRMS (FD) m/z: $[M]^+$ calcd for $C_{21}H_{18}BrNO_4$: 427.04247; found: 427.04198.

Spectral Data for 3-((*1R,2R*)-2-(3-methoxyphenyl)-2-(4-methoxyphenyl)-4-methylene-3-oxocyclobutyl)oxazolidin-2-one (3f):



Compound **3f** (*diastereomeric ratio 1.6:1*) was purified on silica gel column using ethyl acetate/hexane: (20: 80) as the eluent; yellow solid (83.0 mg, 0.218 mmol, 54%); ^{1}H NMR (700 MHz, $CDCl_3$) for major isomer: 7.66 (d, *J* = 9.1 Hz, 2H), 7.32 ~ 7.31(m, 1H), 7.23 ~ 7.17 (m, 3H), 6.87 ~ 6.84 (m, 3H), 6.82 (d, *J* = 8.4 Hz, 1H), 6.77 ~ 6.74 (m, 3H), 6.26 ~ 6.25 (m, 1H), 5.84 (s, 1H), 5.41 (s, 1H), 4.13 ~ 4.10 (m, 1H), 3.74 (s, 3H), 3.71 (s, 3H), 3.27 ~ 3.23 (m, 1H), 2.26 ~ 2.19 (m, 1H); ^{1}H NMR (700 MHz, $CDCl_3$) for minor isomer: 5.87 (s, 1H), 5.40 (s, 1H), 4.13 ~ 4.10 (m, 1H), 3.81 ~ 3.78 (m, 3H), 3.73 (s, 3H) 3.27~3.23 (m, 1H), 2.26 ~ 2.19 (m, 1H), rest of peaks are merged with major isomer.;^{13}C NMR (175 MHz, $CDCl_3$) for major isomer: 197.6, 160.0, 158.8, 158.49, 149.9, 141.9, 139.7, 132.2, 130.0, 129.8, 128.2, 119.7, 118.8, 114.2, 113.4, 111.8, 78.2, 62.5, 57.92, 55.24, 55.20, 41.1; ^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer: 197.8, 159.8, 158.9, 158.44, 129.9, 128.0, 119.6, 119.2, 114.4, 113.3, 112.2, 78.4, 62.6, 57.94, 55.28, 55.24, other carbons are merged; HRMS-ESI+ m/z: $[M+Na]^+$ calcd for $C_{22}H_{21}NO_5$: 402.13174; found:402.13187.

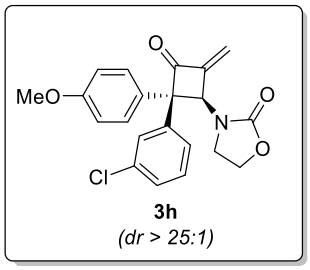
Spectral Data for 3-((*1R,2R*)-2-(4-methoxyphenyl)-4-methylene-3-oxo-2-(m-tolyl)cyclobutyl)oxazolidin-2-one (3g):



3g
(dr > 25:1)

(Compound **3g,** *diastereomeric ratio > 25:1*) was purified on silica gel column using ethyl acetate/hexane: (30: 70) as the eluent; yellow oil (96.5 mg, 0.264 mmol, 66%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.65 (d, *J* = 8.4 Hz, 2H), 7.16 (*t*, *J* = 7.0 Hz, 1H), 7.04 ~ 7.01 (m, 3H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.25 (d, *J* = 2.8 Hz, 1H), 5.85 (*t*, *J* = 2.8 Hz, 1H), 5.40 (s, 1H), 4.12 ~ 4.08 (m, 1H), 3.74 (s, 3H), 3.71 ~ 3.68 (m, 1H), 3.24 ~ 3.20 (m, 1H), 3.26 (s, 3H), 2.15 ~ 2.12 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ; 198.0, 158.7, 158.4, 149.9, 138.8, 138.2, 132.4, 128.9, 128.28, 128.2, 127.2, 123.9, 119.5, 114.2, 78.3, 62.5, 57.8, 55.2, 41.0, 21.4, two carbons merged with aromatic region; HRMS (FD) m/z: $[M]^+$ calcd for $C_{22}H_{21}NO_4$: 363.14761; found: 363.14750.

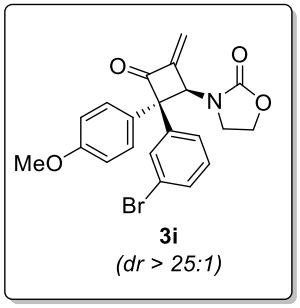
Spectral Data for 3-((*1R,2R*)-2-(3-chlorophenyl)-2-(4-methoxyphenyl)-4-methylene-3-oxocyclobutyl)oxazolidin-2-one (3h):



3h
(dr > 25:1)

(Compound **3h**, *diastereomeric ratio > 25:1*) was purified on silica gel column using ethyl acetate/hexane: (25: 75) as the eluent; yellow solid (86 mg, 0.224 mmol, 56%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.64 (d, *J* = 9.1 Hz, 2H), 7.31 (s, 1H), 7.23 ~ 7.19 (m, 2H), 7.11 (d, *J* = 7.7 Hz, 1H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.28 (t, *J* = 1.4 Hz, 1H), 5.86 (s, 1H), 5.44 (s, 1H), 4.17 ~ 4.14 (m, 1H), 3.81 ~ 3.77 (m, 1H), 3.75 (s, 3H), 3.31 ~ 3.27 (m, 1H), 2.25 ~ 2.22 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ; 197.0, 159.0, 158.4, 149.5, 140.3, 134.8, 131.6, 130.3, 128.2, 127.7, 126.6, 125.3, 120.3, 114.4, 77.7, 62.5, 58.0, 55.2, 41.2, three carbons are merged with aromatic region; HRMS-ESI+ m/z: $[M+Na]^+$ calcd for $C_{21}H_{18}ClNO_4$: 406.08220; found: 406.08235.

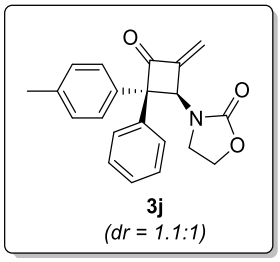
Spectral Data for 3-((*1R,2R*)-2-(3-bromophenyl)-2-(4-methoxyphenyl)-4-methylene-3-oxocyclobutyl)oxazolidin-2-one (3i):



3i
(dr > 25:1)

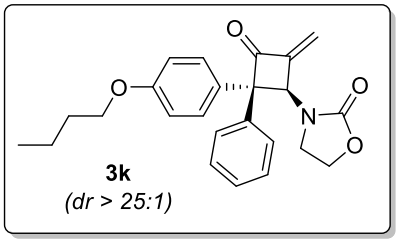
(Compound **3i**, *diastereomeric ratio > 25:1*) was purified on silica gel column using ethyl acetate/hexane: (15: 85) as the eluent; pale yellow solid (103.0 mg, 0.240 mmol, 60%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.63 (d, *J* = 8.4 Hz, 2H), 7.46 (s, 1H), 7.36 ~ 7.35 (m, 1H), 7.17 ~ 7.16 (m, 2H) 6.88 (d, *J* = 8.4 Hz, 2H), 6.28 (s, 1H), 5.86 (s, 1H), 5.44 (s, 1H), 4.17 ~ 4.14 (m, 1H), 3.82 ~ 3.77 (m, 1H), 3.75 (s, 3H), 3.31 ~ 3.27 (m, 1H), 2.24 ~ 2.21 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ; 196.9, 159.0, 158.4, 149.5, 140.5, 131.6, 130.66, 130.64, 129.5, 128.2, 125.7, 122.9, 120.3, 114.4, 77.6, 62.5, 58.0, 55.2, 41.2, two carbons are merged with aromatic region; HRMS (FD) m/z: $[M]^+$ calcd for $C_{21}H_{18}BrNO_4$: 427.0419; found: 427.0425.

Spectral Data for 3-((*1R,2S*)-4-methylene-3-oxo-2-phenyl-2-(p-tolyl)cyclobutyl)oxazolidin-2-one (3j):



(Compound **3j**, *diastereomeric ratio 1.1:1*) was purified on silica gel column using ethyl acetate/hexane: (20: 80) as the eluent; colourless oil (81.0 mg, 0.242 mmol, 61%);^{1}H NMR (700 MHz, $CDCl_3$) for major isomer: 7.62 (d, *J* = 8.4 Hz, 2H), 7.28 ~ 7.25 (m, 4H), 7.14 (d, *J* =7.7 Hz, 4H), 6.25 (s, 1H), 5.90 ~ 5.89 (m, 1H), 5.41 (s, 1H), 4.12 ~ 4.07 (m, 1H), 3.70 ~ 3.67 (m, 1H), 3.25 ~ 3.21 (m, 1H), 2.27 (s, 3H), 2.20 ~ 2.17 (m, 1H); ^{1}H NMR (700 MHz, $CDCl_3$) for minor isomer: 7.73 (d, *J* = 7.7 Hz, 2H), 7.31 (t, *J* = 8.4 Hz, 2H), 7.21 ~ 7.18 (m, 2H), 7.09 (d, *J* = 8.4 Hz, 2H), 3.75 ~ 3.72 (m, 1H), 2.26 (s, 3H), 2.14 ~ 2.11 (m, 1H), rest of peaks are merged with major isomer; ^{13}C NMR (175 MHz, $CDCl_3$) for major isomer: 197.8, 158.40, 149.84, 140.3, 138.2, 137.0, 135.0, 129.7, 129.5, 128.8, 127.4, 127.0, 126.9, 126.8, 126.7, 119.7, 78.70, 62.4, 57.7, 41.0, 20.95; ^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer: 197.9, 158.41, 149.8, 137.3, 137.2, 127.1, 119.6, 78.71, 62.5, 57.6, 41.0, 20.9, rest of carbons are merged; HRMS (FD) m/z: $[M]^+$ calcd for $C_{21}H_{19}NO_3$: 333.13704; found: 333.13668.

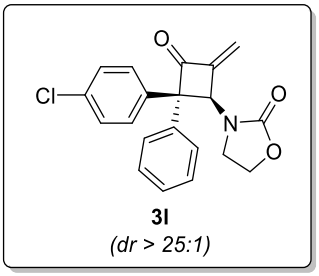
Spectral Data for 3-((*1R,2S*)-2-(4-butoxyphenyl)-4-methylene-3-oxo-2-phenylcyclobutyl)oxazolidin-2-one (3k):



Compound **3k**, (*diastereomeric ratio > 25:1*) was purified on silica gel column using ethyl acetate/hexane: (15: 85) as the eluent; white solid (82.0 mg, 0.209 mmol, 52%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.62 (d, *J* = 8.4 Hz, 2H), 7.26 ~ 7.23 (m, 4H), 7.20 ~ 7.18 (m, 1H), 6.84 (d, *J* = 9.1 Hz, 2H), 6.25 (s, 1H), 5.85 (s, 1H), 5.40 (s, 1H), 4.09 ~ 4.06 (m, 1H), 3.88 (t, *J* = 6.3 Hz, 2H), 3.69 ~ 3.66 (m, 1H), 3.23 ~ 3.19 (m, 1H), 2.14 ~ 2.11 (m, 1H), 1.71 ~ 1.67 (m, 2H), 1.44 ~ 1.41

(m, 2H), 0.91 (t, *J* = 7.7 Hz, 3H); ^{13}C NMR (175 MHz, $CDCl_3$): δ; 197.9, 158.4, 158.3, 149.9, 138.4, 132.0, 129.0, 128.2, 127.4, 126.8, 119.6, 114.8, 78.3, 67.6, 62.5, 57.9, 41.0, 31.2, 19.1, 13.7, four carbons merged with aromatic region; HRMS (FD) m/z: $[M]^+$ calcd for $C_{24}H_{25}NO_4$: 391.17891; found: 391.17823.

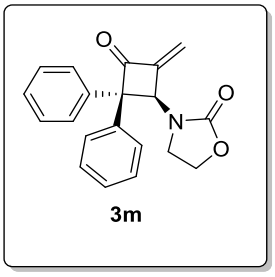
Spectral Data for 3-((*1R,2S*)-2-(4-chlorophenyl)-4-methylene-3-oxo-2-phenylcyclobutyl)oxazolidin-2-one (3l):



3l
(dr > 25:1)

Compound **3l**, (*diastereomeric ratio > 25:1*) was purified on silica gel column using ethyl acetate/hexane: (20: 80) as the eluent; white solid (93 mg, 0.254 mmol, 63%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.71 (d, *J* = 7.7 Hz, 2H), 7.33 (*t*, *J* = 7.7 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.23 ~ 7.21 (m, 3H), 6.29 (s, 1H), 5.91 (s, 1H), 5.45 (s, 1H), 4.17 ~ 4.14 (m, 1H), 3.83 ~ 3.79 (m, 1H), 3.31 ~ 3.27 (m, 1H), 2.28 ~ 2.24 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ; 197.1, 158.3, 149.5, 139.7, 136.6, 133.7, 129.2, 129.0, 128.2, 127.5, 126.9, 120.4, 78.2, 62.5, 57.8, 41.2, four carbons are merged with aromatic region; HRMS-ESI+ m/z: $[M+Na]^+$ calcd for $C_{20}H_{16}ClNO_3$: 376.07164; found: 376.07177.

Spectral Data for (*R*)-3-(4-methylene-3-oxo-2,2-diphenylcyclobutyl)oxazolidin-2-one (3m):

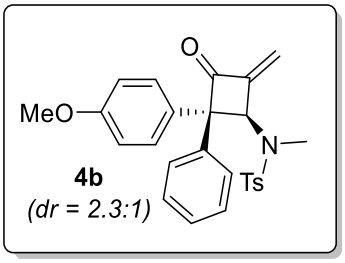


3m

(Compound **3m**) was purified on silica gel column using ethyl acetate/hexane: (15: 85) as the eluent; yellow oil (91.0 mg, 0.284 mmol, 71%); ^{1}H NMR (700 MHz, $CDCl_3$): δ 7.75 (d, *J* = 7.7 Hz, 2H), 7.33 (*t*, *J* = 7.7 Hz, 2H), 7.29 ~ 7.25 (m, 4H), 7.22 ~ 7.20 (m, 2H), 6.26 (s, 1H), 5.92 (s, 1H), 5.42 (s, 1H), 4.11 ~ 4.08 (m, 1H), 3.72 ~ 3.68 (m, 1H), 3.25 ~3.21 (m, 1H), 2.14 ~ 2.10 (m,

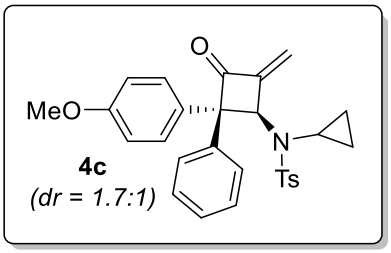
1H); ^{13}C NMR (175 MHz, $CDCl_3$): δ; 197.6, 158.4, 149.7, 140.1, 138.0, 129.0, 128.8, 127.5, 127.2, 127.0, 126.9, 119.8, 79.0, 62.5, 57.7, 41.0, one carbon is merged with aromatic region; HRMS-ESI+ m/z: $[M+Na]^+$ calcd for $C_{20}H_{17}NO_4$: 342.11061; found: 342.11023.

Spectral Data for *N*-((*1R,2S*)-2-(4-methoxyphenyl)-4-methylene-3-oxo-2-phenylcyclobutyl)-N,4-dimethylbenzenesulfonamide (4b):



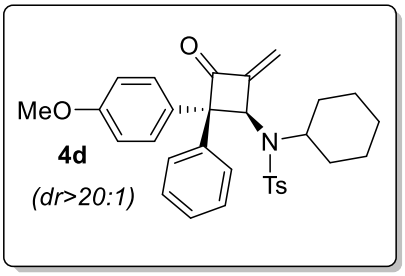
Compound **4b**, (*diastereomeric ratio 2.3:1*) was purified on silica gel column using ethyl acetate/hexane: (12: 88) as the eluent; yellow solid (65.2 mg, 0.145 mmol, 65%); ^{1}H NMR (700 MHz, $CDCl_3$) for major isomer: 7.77 ~ 7.75 (m, 2H), 7.64 (d, *J* = 9.1 Hz, 2H), 7.40 (d, *J* = 7.7 Hz, 2H), 7.32 ~ 7.31 (m, 3H), 7.27 ~ 7.23 (m, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 5.95 (t, *J* = 2.8 Hz, 1H), 5.89 (d, *J* = 2.8 Hz, 1H), 4.14 (d, *J* = 1.4 Hz, 1H), 3.74 (s, 3H), 2.42 (s, 3H), 2.01 (s, 3H). ^{1}H NMR (700 MHz, $CDCl_3$) for minor isomer: 7.71(d, *J* = 7.7 Hz, 2H), 7.20 ~ 7.17 (m, 2H), 6.81 (d, *J* = 9.1 HZ, 2H), 5.97 (t, *J* = 2.1 Hz, 1H), 4.12 (d, *J* = 2.1 Hz, 1H), 3.73 (s, 3H), 2.05 (s, 3H), rest of peaks are merged with major isomer.;^{13}C NMR (175 MHz, $CDCl_3$) for major isomer: 197.9, 158.6, 149.85, 143.94, 141.9, 138.01, 135.9, 133.8, 129.9, 128.8, 128.2, 127.7, 127.1, 127.0, 126.4, 119.0, 114.3, 76.9, 61.9, 55.2, 30.9, 21.5, three carbons merged with aromatic region; ^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer: 198.03, 158.7, 149.82, 143.95, 135.8, 128.8, 127.2, 127.1, 119.05, 114.2, 61.6, 55.1, 31.05, other carbons merged with major isomer.; HRMS-ESI+ m/z: $[M+Na]^+$ calcd for $C_{26}H_{25}NO_4S$: 470.14020; found: 470.14006.

Spectral Data for *N*-cyclopropyl-*N*-((*1R,2S*)-2-(4-methoxyphenyl)-4-methylene-3-oxo-2-phenylcyclobutyl)-4-methylbenzenesulfonamide (4c):



Compound **4c**, (*diastereomeric ratio 1.7:1*) was purified on silica gel column using ethyl acetate/hexane: (15: 85) as the eluent; yellow solid (68.5 mg, 0.144 mmol, 72%); ^{1}H NMR (700 MHz, $CDCl_3$) for major isomer: 7.83 ~ 7.82 (m, 2H), 7.67 (d, *J* = 9.1 Hz, 2H), 7.62 (d, *J* = 7.0 Hz, 2H), 7.35 (d, *J* = 7.7 Hz, 2H), 7.27 (t, *J* = 7.7 Hz, 2H), 7.18 ~ 7.14 (m, 1H), 6.85 (d, *J* = 11.9 Hz, 2H), 5.94 ~ 5.92 (m, 1H), 5.90 ~ 5.89 (m, 1H), 4.38 (s, 1H), 3.73 (s, 3H), 2.44 (s, 3H), 1.65 ~ 1.62 (s, 1H), 0.75 ~ 0.71 (m, 1H), 0.63 ~ 0.56 (m, 1H), 0.01 ~ -0.04 (m, 1H), -0.47 ~ -0.50 (m, 1H); ^{1}H NMR (700 MHz, $CDCl_3$) for minor isomer: 7.73 (d, *J* = 7.7 Hz, 2H), 7.56 (d, *J* = 8.4 Hz, 2H), 7.30 (t, *J* = 7.7 Hz, 2H), 6.82 (d, *J* = 7 Hz, 2H), 0.81 ~ 0.78 (m, 1H), -0.38 ~ -0.42 (m, 1H), rest of peaks are merged with major isomer; ^{13}C NMR (175 MHz, $CDCl_3$) for major isomer: 197.70, 158.6, 149.67, 144.0, 142.7, 139.7, 135.15, 134.5, 131.6, 129.8, 129.0, 128.39, 127.5, 127.0, 126.9, 126.3, 118.64, 114.4, 113.8, 77.4, 65.3, 55.2, 29.5, 21.5, 9.51, three carbons are merged with major isomer; ^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer: 197.8, 158.4, 149.69, 135.12, 128.34, 126.8, 118.60, 65.1, 55.1, 29.7, 9.6, rest of carbons are merged.; HRMS-ESI+ m/z: $[M+Na]^+$ calcd for $C_{28}H_{27}NO_4S$: 496.15585; found: 496.15548.

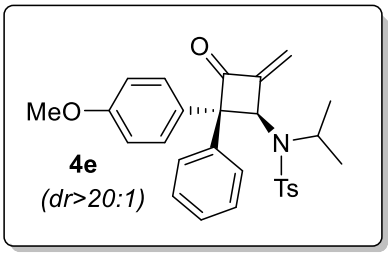
Spectral data for N-cyclohexyl-N-((1R,2S)-2-(4-methoxyphenyl)-4-methylene-3-oxo-2-phenylcyclobutyl)-4-methylbenzenesulfonamide (4d)



Compound **4d**, (*diastereomeric ratio 20:1*) was purified on silica gel column using ethyl acetate/hexane: (15: 85) as the eluent; pale yellow solid (65.5 mg, 0.127 mmol, 74%); ^{1}H NMR (700 MHz, $CDCl_3$); 7.82 (s, 2H), 7.46 (d, *J* = 8.4 Hz, 2H), 7.33 ~ 7.28 (m, 4H), 7.26 ~ 7.24 (m, 2H), 7.17 (t, *J* = 7 Hz, 1H), 6.82 (d, *J* = 9.1 Hz, 2H), 6.04 (s, 1H), 5.71 (s, 1H), 4.92 (s, 1H), 3.74

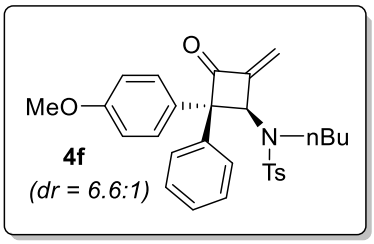
(s, 3H), 2.50 ~ 2.45 (m, 1H), 2.43 (s, 3H), 1.86 ~ 1.81 (m, 1H), 1.66 ~ 1.59 (m, 2H), 1.48 (s, 1H), 1.41 ~ 1.37 (m, 1H), 1.29 ~ 1.37 (m, 2H), 0.98 ~ 0.86 (m, 2H), 0.57 ~ 0.50 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$); 194.5, 158.6, 152.2, 143.4, 139.1, 133.7, 129.6, 128.6, 128.0, 127.7, 119.5, 114.2, 78.3, 63.5, 62.1, 55.2, 32.6, 32.2, 28.8, 27.1, 26.6, 25.2, 21.5 rest of carbons are merged; HRMS-ESI+ calcd for $C_{31}H_{33}NO_4S$ $[M+Na]^+$: 538.20280, found: 538.20302

Spectral data for N-isopropyl-N-((1R,2S)-2-(4-methoxyphenyl)-4-methylene-3-oxo-2-phenylcyclobutyl)-4-methylbenzenesulfonamide (4e);



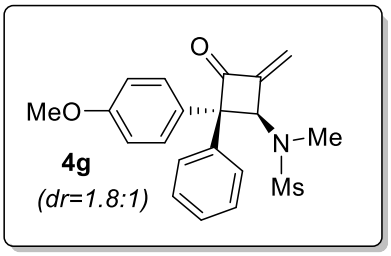
Compound **4e**, (*diastereomeric ratio 20:1*) was purified on silica gel column using ethyl acetate/hexane: (15: 85) as the eluent; yellow solid (55.9 mg, 0.117 mmol, 59%); ^{1}H NMR (700 MHz, $CDCl_3$); 7.81 ~ 7.80 (m, 2H), 7.51 (d, *J* = 7.7 Hz, 1H), 7.44 (d, *J* = 8.4 Hz, 1H), 7.36 (d, *J* = 7.7 Hz, 1H), 7.31 ~ 7.25 (m, 5H), 7.17 (t, *J* = 7 Hz, 1H), 6.82 ~ 6.79 (m, 2H), 6.04 (d, *J* = 2.8 Hz, 1H), 5.70 (d, *J* = 13.3 Hz, 1H), 4.94 (s, 1H), 3.73 (s, 3H), 3.03 ~ 2.98 (m, 1H), 2.43 (s, 3H), 1.16 ~ 1.14 (m, 3H), 0.56 ~ 0.47 (m, 3H);^{13}C NMR (175 MHz, $CDCl_3$) for major isomer:198.5, 158.6, 152.0, 143.4, 141.9, 138.9, 138.8, 133.7, 130.8, 129.5, 129.0, 128.5, 128.5, 127.8, 127.9, 127.7, 127.6, 126.6, 119.6, 114.2, 78.0, 63.5, 55.1, 53.0, 22.0, 21.4, two carbons are merged. ^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer: 198.8, 128.9, 127.11, 114.0, 78.4, 22.2, rest of carbons are merged. HRMS-ESI+ calcd for $C_{28}H_{29}NO_4S$ $[M+Na]^+$: 498.17150, found: 498.17089.

Spectral Data for N-butyl-N-((1R,2S)-2-(4-methoxyphenyl)-4-methylene-3-oxo-2-phenylcyclobutyl)-4-methylbenzenesulfonamide (4f);



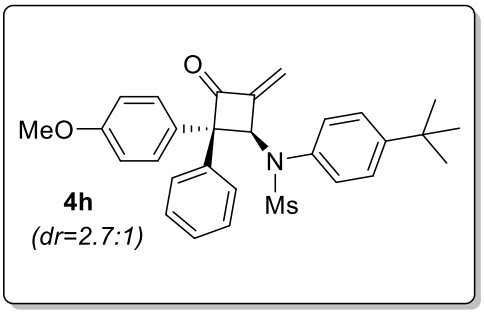
Compound **4f**, (*diastereomeric ratio 6.6:1*) was purified on silica gel column using ethyl acetate/hexane: (15: 85) as the eluent; pale yellow solid (72.0 mg, 0.147 mmol, 78%); ^{1}H NMR (700 MHz, $CDCl_3$) for major isomer: 7.80 (d, *J* = 7.7 Hz, 2H), 7.61 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 7.0 Hz, 2H), 7.33 ~ 7.25 (m, 5H), 7.19 ~ 7.17 (m, 1H), 6.85 (d, *J* = 8.4 Hz, 2H), 5.92 (s, 1H), 4.34 (s, 1H), 3.74 (s, 3H), 2.59 ~ 2.54 (m, 1H), 2.43 (s, 3H), 2.32 ~ 2.28 (m, 1H), 1.27 ~ 1.22 (m, 1H), 0.81 ~ 0.78 (m, 1H), 0.66 ~ 0.62 (m, 1H), 0.60 ~ 0.54 (m, 3H), 0.35 ~ 0.32 (m, 1H); ^{1}H NMR (700 MHz, $CDCl_3$) for minor isomer: 7.68 (d, *J* = 7.7 Hz, 2H), 6.81 (d, *J* = 9.1 Hz, 2H), 3.73 (s, 1H), 0.47 ~ 0.41 (m, 1H) rest of peaks are merged with major isomer; ^{13}C NMR (175 MHz, $CDCl_3$) for major isomer: 198.7, 158.6, 151.2, 143.8, 138.3, 136.8, 134.05, 129.9, 128.7, 127.9, 127.7, 127.3, 127.1, 119.1, 114.2, 77.8, 63.8, 55.2, 47.6, 31.2, 21.5,20.3, 13.3, six carbons are merged; HRMS-ESI+ calcd for $C_{29}H_{31}NO_4S$ $[M+Na]^+$: 512.18715, found: 512.18748.

Spectral data for N-((1R,2S)-2-(4-methoxyphenyl)-4-methylene-3-oxo-2-phenylcyclobutyl)-N-methylmethanesulfonamide (4g)



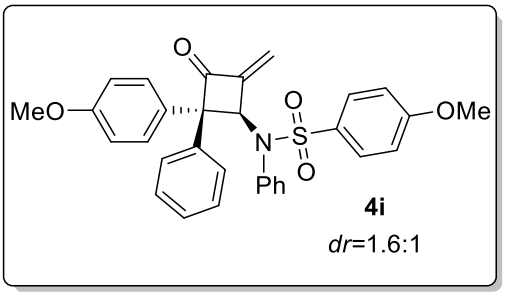
Compound **4g**, (*diastereomeric ratio 1.8:1*) was purified on silica gel column using ethyl acetate/hexane: (12: 88) as the eluent; yellow oil (85.8 mg, 0.230 mmol, 68%);^{1}H NMR (700 MHz, $CDCl_3$) for major isomer: 7.57 (d, *J* = 9.1 Hz, 2H), 7.35 ~ 7.18 (m, 5H), 6.84 ~ 6.81 (m, 2H). 6.27 (s, 1H), 5.95 (s, 1H), 5.31 (s, 1H), 3.73 (s, 3H), 2.91 (s, 3H), 2.12 (s, 3H);^{1}H NMR (700 MHz, $CDCl_3$) for minor isomer: 7.65 (d, *J* = 7.7 Hz, 2H), 5.97 (s, 1H), 2.93 (s, 3H), 2.17 (s, 3H) rest of peaks are merged with major isomer; ^{13}C NMR (175 MHz, $CDCl_3$) for major isomer: 198.11, 158.6, 150.65, 141.2, 138.0, 133.1, 129.7, 128.82, 128.4, 127.8, 127.2, 127.1, 126.5, 119.0, 114.25, 77.6, 61.5, 55.2, 38.1, 30.6; ^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer: 198.17, 158.8, 150.64, 128.85, 127.3, 114.28, 77.7, 61.2, 55.1, 38.0, 30.7 rest of the carbons are merged.; HRMS-ESI- calcd for $C_{20}H_{21}NO_4S$ [M-H]: 370.11130, found: 370.11098.

Spectral data for N-(4-(tert-butyl)phenyl)-N-((1R,2S)-2-(4-methoxyphenyl)-4-methylene-3-oxo-2-phenylcyclobutyl)methanesulfonamide (4h)



Compound **4h**, (*diastereomeric ratio 2.7:1*) was purified on silica gel column using ethyl acetate/hexane: (15: 85) as the eluent; white solid (57.2 mg, 0.116 mmol, 62%);^{1}H NMR (700 MHz, $CDCl_3$) for major isomer: 7.62 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 7.7 Hz, 2H), 7.33 ~ 7.20 (m, 3H), 7.14 ~ 7.11 (m, 2H), 6.86 ~ 6.83 (m, 2H), 6.50 (d, *J* = 7.7 Hz, 2H), 6.25 (s, 1H), 6.07 (s, 1H), 5.31 (s, 1H), 3.75 (s, 3H), 2.68 (s, 3H), 1.23 (s, 9H); ^{1}H NMR (700 MHz, $CDCl_3$) for minor isomer: 7.69 (d, *J* = 7.7 Hz, 2H), 6.56 (d, *J* = 7.7 Hz, 2H), 6.28 (s, 1H), 3.78 (s, 3H), 2.73 (s, 3H) other peaks are merged;^{13}C NMR (175 MHz, $CDCl_3$) for major isomer:198.7, 158.7, 152.0, 151.07, 140.9, 139.2, 133.5, 132.7, 131.65, 131.1, 129.9, 128.81, 128.5, 128.4, 127.3, 127.2, 127.1, 125.92, 121.63, 114.2, 79.1, 63.9, 55.2, 39.6, 34.59, 31.17, three carbons are merged; ^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer:198.9, 158.8, 151.9, 151.1, 133.6, 131.61, 128.85, 125.93, 121.60, 113.8, 79.3, 63.7, 55.3, 39.7, 34.58, 31.18, rest of carbons are merged; HRMS-ESI+ calcd for $C_{29}H_{31}NO_4S$ $[M+Na]^+$: 512.18715, found: 512.18737.

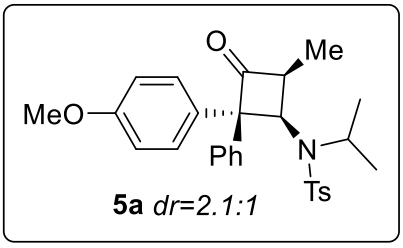
Spectral data for 4-methoxy-N-((1R,2S)-2-(4-methoxyphenyl)-4-methylene-3-oxo-2-phenylcyclobutyl)-N-phenylbenzenesulfonamide (4i)



Compound **4i**, (*diastereomeric ratio 1.6:1*) was purified on silica gel column using ethyl acetate/hexane: (20: 80) as the eluent; white solid (65 mg, 0.162 mmol, 77%);^{1}H NMR (700 MHz, $CDCl_3$) for major isomer:7.65 ~ 7.64 (m, 2H), 7.59 ~ 7.56 (m, 2H), 7.30 ~ 7.28 (m, 2H), 7.23 ~ 7.17 (m, 3H), 7.08 ~ 7.05 (m, 1H), 6.94 ~ 6.86 (m, 6H), 6.34 (dd, *J* = 2.1 Hz, 1H), 6.12 (d, *J* = 8.4 Hz, 2H), 5.99 (s, 1H), 4.95 (s, 1H), 3.83 (s, 3H), 3.74 (s, 3H); ^{1}H NMR (700 MHz, $CDCl_3$) for

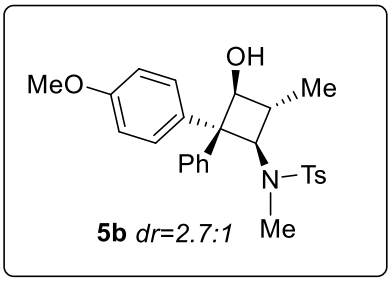
minor isomer: 7.72 (d, *J* = 7.7 Hz, 2H), 7.33 (t, *J* = 7.7 Hz, 2H), 6.75 (dd, *J* = 1.4 Hz, 2H), 6.20 (d, *J* = 7.7 Hz, 2H), 4.93 (s, 3H), 3.75 (s, 3H), rest of peaks are merged with major isomer; ^{13}C NMR (175 MHz, $CDCl_3$) for major isomer:198.5, 163.11, 158.71, 151.90, 141.68, 138.84, 136.0, 133.5, 132.58, 131.5, 130.7, 129.8, 129.4, 128.9, 128.5, 128.21, 128.0, 127.0, 126.9, 121.07, 114.3, 114.0, 113.9, 78.6, 64.1, 55.6, 55.23, three carbons are merged;^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer:198.8, 163.12, 158.78, 151.97, 141.69, 138.85, 136.2, 132.51, 131.6, 128.24, 127.1, 121.02, 114.03, 78.8, 64.0, 55.27, rest of carbons are merged; HRMS-ESI+ calcd for $C_{31}H_{27}NO_5S$ $[M+Na]^+$: 548.15076, found: 548.15126.

Spectral data for N-isopropyl-N-((1R,2S,4S)-2-(4-methoxyphenyl)-4-methyl-3-oxo-2-phenylcyclobutyl)-4-methylbenzenesulfonamide (5a)



Compound **5a**, (*diastereomeric ratio 2.1:1*) was purified on silica gel column using ethyl acetate/hexane: (10: 90) as the eluent; colourless oil (57 mg, 0.043 mmol, 42%); ^{1}H NMR (700 MHz, $CDCl_3$) for major isomer:7.76 ~ 7.75 (m, 2H), 7.33 (d, *J* = 9.1 Hz, 2H), 7.28 ~ 7.23 (m, 5H), 7.21 ~ 7.14 (m, 2H), 6.80 ~ 6.78 (m, 2H), 4.87 ~ 4.85 (m, 1H), 3.76 ~ 3.74 (m, 3H), 3.54 ~ 3.46 (m, 1H), 3.01 ~ 2.94 (m, 1H), 2.41 (s, 3H), 1.20 ~ 1.17 (m, 3H), 0.99 (d, *J* = 7 Hz, 3H), 0.87 (d, *J* = 7 Hz, 3H).; ^{1}H NMR (700 MHz, $CDCl_3$) for minor isomer: 7.38 (d, *J* = 7.7 Hz, 2H), 7.07 (d, *J* = 9.1 Hz, 2H), 3.05 ~ 3.01 (m, 1H), 0.95 (d, *J* = 6.3 Hz, 3H), rest of peaks are merged with major isomer; ^{13}C NMR (175 MHz, $CDCl_3$) for major isomer: 210.74, 158.4, 143.5, 141.8, 138.64, 133.8, 130.3, 129.5, 129.3, 128.7, 128.1, 127.9, 127.2,126.7, 113.9, 77.9, 61.9, 55.46, 55.1,51.91, 22.2, 22.10, 21.4, 13.4, four carbons are merged; ^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer: 210.73, 158.7, 138.62, 128.5, 126.8, 114.1, 78.01, 61.6, 55.42, 55.2, 51.94, 22.4, 22.15, 13.3, rest of carbons are merged; HRMS-ESI+ calcd for $C_{28}H_{31}NO_4S$ $[M+Na]^+$: 500.18715, found: 500.18661.

Spectral data for N-((1R,2S,3S,4R)-3-hydroxy-2-(4-methoxyphenyl)-4-methyl-2-phenylcyclobutyl)-N,4-dimethylbenzenesulfonamide (5b)



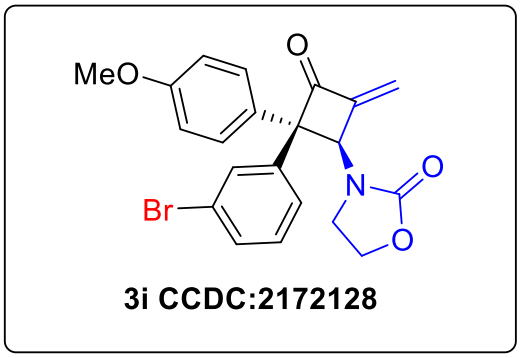
Compound **5b**, (*diastereomeric ratio 2.7:1*) was purified on silica gel column using ethyl acetate/hexane: (30: 70) as the eluent; colourless oil (31 mg, 0.070 mmol, 63%);^{1}H NMR (700 MHz, $CDCl_3$) for major isomer:7.68 ~ 7.67 (m, 2H), 7.65 (d, *J* = 7.7 Hz, 2H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.27 ~ 7.24 (m, 3H), 7.20 (t, *J* = 7.7 Hz, 2H), 6.86 (d, *J* = 9.1 Hz, 2H), 4.27 ~ 4.25 (m, 1H), 3.78 (s, 3H), 3.74 (s, 1H), 2.70 ~ 2.62 (m, 1H), 2.46 (s, 1H), 2.39 (s, 3H), 1.91 (s, 3H), 0.68 (d, *J* = 6.3 Hz, 3H);^{1}H NMR (700 MHz, $CDCl_3$) for minor isomer: 7.58 ~ 7.56 (m, 2H), 7.32 (t, *J* = 7.7 Hz, 2H), 7.16 ~ 7.14 (m, 2H), 6.76 (d, *J* = 8.4 Hz, 2H), 1.97 (s, 3H), rest of peaks are merged with major isomer; ^{13}C NMR (175 MHz, $CDCl_3$) for major isomer:157.7,149.3, 143.3, 141.6, 140.5, 135.8, 131.5, 130.4, 129.7, 129.5, 128.5, 127.8, 127.45, 127.3, 127.2, 126.2, 126.1, 113.8, 80.0, 62.1, 58.4, 55.2, 41.14, 31.1, 21.4, 16.69; ^{13}C NMR (175 MHz, $CDCl_3$) for minor isomer: 157.9,132.2,127.47, 126.0, 113.3, 79.6, 62.0, 58.1, 55.1, 41.12, 31.2, 16.66, rest of carbons are merged; HRMS-ESI+ calcd for $C_{26}H_{29}NO_4S$ $[M+Na]^+$: 474.17150, found: 474.17158.

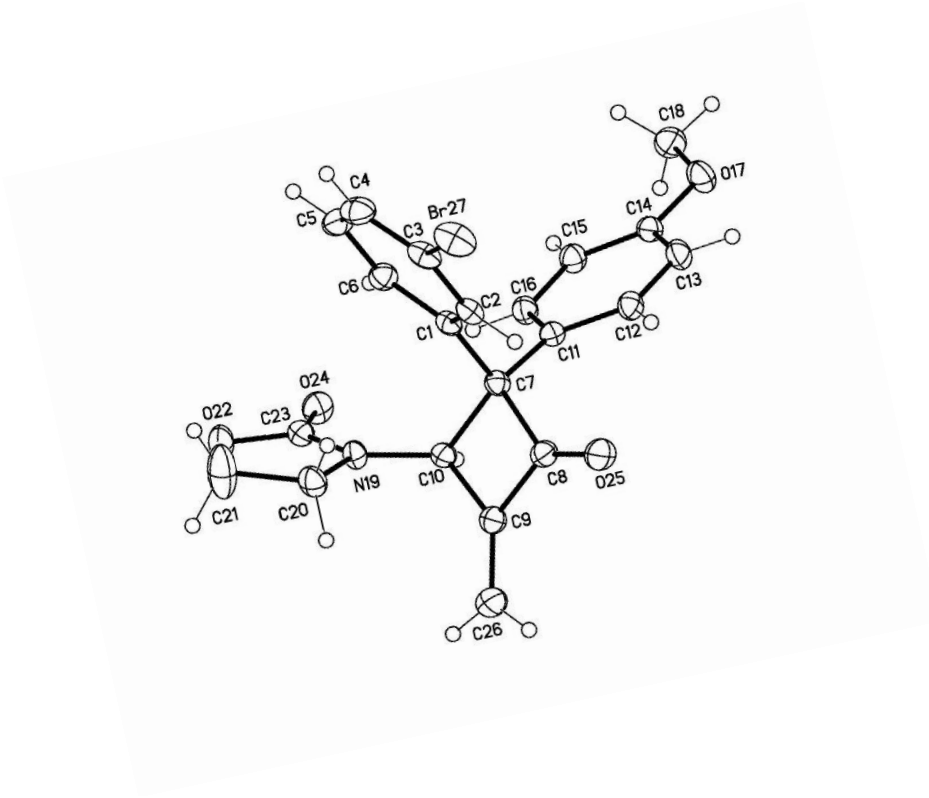
7. X-ray crystallographic structure and data for compound (3i, 3l, 3m and 4f):

(a) X-ray crystallographic data of compound (3i):

Ellipsoid contour %probability level = 50%

Experimental: The sample was dissolved in appropriate amount of Dichloromethane 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.





220428lt_auto

Table S1 Crystal data and structure refinement for 220428lt_auto.

Identification code	220428lt_auto
Empirical formula	$C_{21}H_{18}BrNO_4$
Formula weight	428.27
Temperature/K	100.00(10)
Crystal system	triclinic
Space group	P-1

a/Å	8.5723(2)
b/Å	10.1918(3)
c/Å	10.7219(3)
α/°	91.770(2)
β/°	98.537(2)
γ/°	96.489(2)
Volume/$Å^3$	919.34(4)
Z	2
c_{alcd}/cm^3	1.547
μ/mm^{-1}	3.282
F(000)	436.0
Crystal size/mm^3	0.05 × 0.04 × 0.04
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	8.348 to 149.922
Index ranges	$-10 \leq h \leq 10$, $-11 \leq k \leq 12$, $-13 \leq l \leq 12$
Reflections collected	10098
Independent reflections	3579 [R_{int} = 0.0236, R_{sigma} = 0.0280]
Data/restraints/parameters	3579/0/245
Goodness-of-fit on F^2	1.063
Final R indexes [I>=2σ (I)]	R_1 = 0.0296, wR_2 = 0.0722
Final R indexes [all data]	R_1 = 0.0344, wR_2 = 0.0743
Largest diff. peak/hole / e $Å^{-3}$	0.37/-0.54

Table S2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 220428lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Br27	4948.9(3)	2240.5(2)	6479.6(2)	32.73(9)
C1	4224(2)	6134(2)	7152.3(18)	17.7(4)
C2	4039(3)	4760(2)	7034.2(19)	19.8(4)
C3	5245(3)	4113(2)	6653.1(19)	23.6(5)
C4	6639(3)	4795(2)	6402(2)	27.7(5)
C5	6809(3)	6166(2)	6504(2)	27.1(5)
C6	5617(2)	6837(2)	6864(2)	20.9(4)
C7	2935(2)	6886.7(19)	7547.6(19)	17.0(4)
C8	1778(2)	6097(2)	8314(2)	19.9(4)
C9	2286(2)	7003(2)	9451(2)	20.4(4)
C10	3457(2)	7849(2)	8778.6(19)	17.6(4)
C11	2108(2)	7526(2)	6409.3(19)	18.3(4)
C12	878(2)	6781(2)	5591(2)	20.0(4)
C13	201(3)	7292(2)	4487(2)	22.8(4)
C14	746(2)	8559(2)	4165.3(19)	20.4(4)
C15	1939(2)	9327(2)	4986(2)	20.7(4)
C16	2608(2)	8799(2)	6096.2(19)	20.0(4)
C18	654(3)	10190(2)	2612(2)	28.1(5)
C20	5832(3)	7001(2)	10145(2)	23.8(5)
C21	7494(3)	7704(3)	10529(3)	46.3(8)
C23	6093(2)	9073(2)	9280.9(19)	20.8(4)
C26	1876(3)	7030(2)	10594(2)	28.4(5)
N19	5092(2)	7965.4(16)	9360.5(16)	18.3(4)

Table S2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 220428lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O17	27.5(18)	8951.9(15)	3038.8(14)	25.9(3)
O22	7534.0(18)	8971.2(16)	9967.7(15)	27.3(3)
O24	5817.1(18)	10033.0(15)	8687.5(14)	25.2(3)
O25	832.5(19)	5111.3(15)	8073.3(15)	27.9(4)

Table S3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 220428lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Br27	50.57(18)	20.91(13)	27.73(14)	-0.68(9)	3.49(11)	12.45(10)
C1	19.9(10)	19.8(10)	13.0(9)	1.4(7)	0.4(8)	3.5(8)
C2	23.4(11)	20.6(10)	14.3(9)	0.4(8)	0.2(8)	1.8(8)
C3	33.6(12)	21.6(11)	15.7(10)	1.1(8)	0.7(9)	7.7(9)
C4	31.0(12)	32.2(12)	23.4(11)	1.8(9)	8.0(9)	13.9(10)
C5	24.8(11)	34.8(13)	23.6(11)	4.3(9)	9.0(9)	4.4(9)
C6	22.8(11)	20.4(10)	19.7(10)	2.4(8)	3.9(8)	1.4(8)
C7	18.6(10)	15.2(9)	16.5(10)	-1.0(8)	2.4(8)	-0.4(8)
C8	18.8(10)	20.8(10)	20.5(10)	0.2(8)	4.5(8)	2.2(8)
C9	20.2(10)	20.7(10)	19.8(10)	0.3(8)	3.5(8)	-0.7(8)
C10	18.0(10)	18.1(10)	16.4(10)	-1.1(8)	3.0(8)	1.3(8)
C11	16.9(10)	21.6(10)	16.6(10)	-0.5(8)	2.9(8)	3.5(8)
C12	18.5(10)	18.4(10)	22.2(10)	-2.4(8)	2.2(8)	1.2(8)
C13	20.4(10)	24.2(11)	21.7(11)	-4.3(9)	-1.1(8)	0.3(8)
C14	17.9(10)	27.1(11)	16.8(10)	0.7(8)	2.0(8)	7.0(8)

Table S3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 220428lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C15	20.1(10)	19.9(10)	21.5(10)	2.1(8)	2.2(8)	0.9(8)
C16	18.6(10)	20.5(10)	19.9(10)	0.5(8)	1.1(8)	0.1(8)
C18	26.5(12)	35.2(13)	23.4(11)	9.1(9)	3.9(9)	5.2(10)
C20	24.2(11)	25.1(11)	21.0(11)	5.8(9)	-0.5(9)	2.2(9)
C21	30.7(14)	40.6(16)	59.9(19)	21.6(14)	-16.1(13)	-3.5(11)
C23	20.0(10)	23.6(11)	17.8(10)	-1.6(8)	3.3(8)	-0.7(8)
C26	26.0(12)	32.9(13)	25.8(12)	-1.0(10)	7.2(9)	-2.0(9)
N19	17.6(9)	17.2(8)	18.6(8)	1.9(7)	-0.5(7)	-0.3(7)
O17	26.1(8)	29.6(8)	20.4(8)	4.1(6)	-2.9(6)	4.0(6)
O22	19.9(8)	29.9(8)	28.8(8)	4.6(7)	-2.4(6)	-4.2(6)
O24	25.9(8)	21.4(8)	27.8(8)	4.8(6)	4.5(6)	-1.8(6)
O25	27.8(8)	25.5(8)	28.4(9)	-1.8(6)	6.9(7)	-8.5(7)

Table S4 Bond Lengths for 220428lt_auto.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br27	C3	1.896(2)	C10	N19	1.437(3)
C1	C2	1.392(3)	C11	C12	1.400(3)
C1	C6	1.402(3)	C11	C16	1.387(3)
C1	C7	1.517(3)	C12	C13	1.383(3)
C2	C3	1.391(3)	C13	C14	1.395(3)
C3	C4	1.379(3)	C14	C15	1.393(3)
C4	C5	1.387(3)	C14	O17	1.365(2)
C5	C6	1.385(3)	C15	C16	1.393(3)

Table S4 Bond Lengths for 220428lt_auto.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C7	C8	1.552(3)	C18	O17	1.430(3)
C7	C10	1.597(3)	C20	C21	1.513(3)
C7	C11	1.521(3)	C20	N19	1.452(3)
C8	C9	1.488(3)	C21	O22	1.441(3)
C8	O25	1.212(3)	C23	N19	1.352(3)
C9	C10	1.525(3)	C23	O22	1.357(3)
C9	C26	1.324(3)	C23	O24	1.210(3)

Table S5 Bond Angles for 220428lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	C1	C6	119.04(19)	N19	C10	C7	117.68(16)
C2	C1	C7	121.51(18)	N19	C10	C9	115.74(17)
C6	C1	C7	119.42(18)	C12	C11	C7	119.38(18)
C3	C2	C1	119.48(19)	C16	C11	C7	122.14(18)
C2	C3	Br27	118.56(17)	C16	C11	C12	118.30(19)
C4	C3	Br27	119.60(17)	C13	C12	C11	120.8(2)
C4	C3	C2	121.8(2)	C12	C13	C14	120.27(19)
C3	C4	C5	118.4(2)	C15	C14	C13	119.55(19)
C6	C5	C4	121.0(2)	O17	C14	C13	115.49(19)
C5	C6	C1	120.2(2)	O17	C14	C15	124.96(19)
C1	C7	C8	115.15(17)	C16	C15	C14	119.47(19)
C1	C7	C10	115.56(16)	C11	C16	C15	121.52(19)
C1	C7	C11	109.59(16)	N19	C20	C21	101.32(17)

Table S5 Bond Angles for 220428lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C8	C7	C10	86.56(14)	O22	C21	C20	106.98(19)
C11	C7	C8	113.90(17)	N19	C23	O22	110.03(18)
C11	C7	C10	114.65(16)	O24	C23	N19	127.4(2)
C9	C8	C7	92.52(16)	O24	C23	O22	122.54(19)
O25	C8	C7	133.47(19)	C10	N19	C20	125.99(16)
O25	C8	C9	134.0(2)	C23	N19	C10	121.33(17)
C8	C9	C10	91.53(16)	C23	N19	C20	112.56(17)
C26	C9	C8	133.3(2)	C14	O17	C18	117.37(17)
C26	C9	C10	135.2(2)	C23	O22	C21	109.10(17)
C9	C10	C7	89.39(15)				

Table S6 Torsion Angles for 220428lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
Br27	C3	C4	C5	178.32(17)	C9	C10	N19	C20	27.4(3)
C1	C2	C3	Br27	−179.20(15)	C9	C10	N19	C23	−148.34(19)
C1	C2	C3	C4	0.9(3)	C10	C7	C8	C9	−0.17(16)
C1	C7	C8	C9	116.52(18)	C10	C7	C8	O25	−179.7(3)
C1	C7	C8	O25	−63.0(3)	C10	C7	C11	C12	−144.38(18)
C1	C7	C10	C9	−116.14(18)	C10	C7	C11	C16	40.5(3)
C1	C7	C10	N19	2.9(3)	C11	C7	C8	C9	−115.66(18)
C1	C7	C11	C12	83.8(2)	C11	C7	C8	O25	64.8(3)
C1	C7	C11	C16	−91.3(2)	C11	C7	C10	C9	114.93(18)

Table S6 Torsion Angles for 220428lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C2	C1	C6	C5	-2.0(3)	C11	C7	C10	N19	-126.06(19)
C2	C1	C7	C8	24.0(3)	C11	C12	C13	C14	0.6(3)
C2	C1	C7	C10	122.6(2)	C12	C11	C16	C15	-1.4(3)
C2	C1	C7	C11	-106.0(2)	C12	C13	C14	C15	-2.4(3)
C2	C3	C4	C5	-1.7(3)	C12	C13	C14	O17	178.14(19)
C3	C4	C5	C6	0.8(3)	C13	C14	C15	C16	2.2(3)
C4	C5	C6	C1	1.1(3)	C13	C14	O17	C18	-174.00(19)
C6	C1	C2	C3	1.0(3)	C14	C15	C16	C11	-0.4(3)
C6	C1	C7	C8	−157.89(18)	C15	C14	O17	C18	6.5(3)
C6	C1	C7	C10	-59.2(2)	C16	C11	C12	C13	1.2(3)
C6	C1	C7	C11	72.2(2)	C20	C21	O22	C23	-1.2(3)
C7	C1	C2	C3	179.16(18)	C21	C20	N19	C10	-176.8(2)
C7	C1	C6	C5	179.83(19)	C21	C20	N19	C23	-0.7(3)
C7	C8	C9	C10	0.18(16)	C26	C9	C10	C7	179.0(3)
C7	C8	C9	C26	-179.0(3)	C26	C9	C10	N19	58.3(3)
C7	C10	N19	C20	-76.5(3)	N19	C20	C21	O22	1.1(3)
C7	C10	N19	C23	107.8(2)	N19	C23	O22	C21	0.7(3)
C7	C11	C12	C13	−174.04(19)	O17	C14	C15	C16	-178.33(19)
C7	C11	C16	C15	173.77(19)	O22	C23	N19	C10	176.29(17)
C8	C7	C10	C9	0.17(15)	O22	C23	N19	C20	0.0(2)
C8	C7	C10	N19	119.17(18)	O24	C23	N19	C10	-4.6(3)
C8	C7	C11	C12	-46.8(2)	O24	C23	N19	C20	179.1(2)
C8	C7	C11	C16	138.1(2)	O24	C23	O22	C21	-178.4(2)

Table S6 Torsion Angles for 220428lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C8	C9	C10	C7	-0.17(16)	O25	C8	C9	C10	179.7(3)
C8	C9	C10	N19	120.88(18)	O25	C8	C9	C26	0.5(5)

Table S7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 220428lt_auto.

Atom	*x*	*y*	*z*	U(eq)
H2	3095.73	4266.89	7212.59	24
H4	7463.84	4337.11	6165.24	33
H5	7755.96	6651.34	6324.66	32
H6	5744.34	7776.05	6914.46	25
H10	3105.59	8740.9	8626.77	21
H12	505.17	5912.9	5797.08	24
H13	-639.73	6777.19	3945.76	27
H15	2292.74	10202.79	4790.97	25
H16	3424.82	9323.31	6651.37	24
H18A	74.45	10337.26	1779.41	42
H18B	1782.23	10177.06	2554.04	42
H18C	534.39	10904.09	3211.57	42
H20A	5839.51	6159.74	9661.19	29
H20B	5292.27	6824.31	10888.04	29
H21A	7746.65	7820.78	11460.44	56
H21B	8281.68	7183.87	10223.84	56
H26A	1103.51	6366.27	10807.92	34

Table S7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 220428lt_auto.

Atom	*x*	*y*	*z*	U(eq)
H26B	2354.81	7712.58	11199.19	34

Experimental

Single crystals of $C_{21}H_{18}BrNO_4$ **[220428lt_auto]** were **[]**. A suitable crystal was selected and **[]** on a **XtaLAB Synergy R, DW system, HyPix-Arc 150** diffractometer. The crystal was kept at 100.00(10) K during data collection. Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program using Intrinsic Phasing and refined with the SHELXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [220428lt_auto]

Crystal Data for $C_{21}H_{18}BrNO_4$ (M =428.27 g/mol): triclinic, space group P-1 (no. 2), a = 8.5723(2) Å, b = 10.1918(3) Å, c = 10.7219(3) Å, α = 91.770(2)°, β = 98.537(2)°, γ = 96.489(2)°, V = 919.34(4) Å^3, Z = 2, T = 100.00(10) K, μ(Cu Kα) = 3.282 mm^{-1}, *Dcalc* = 1.547 g/cm^3, 10098 reflections measured (8.348° ≤ 2Θ ≤ 149.922°), 3579 unique (R_{int} = 0.0236, R_{sigma} = 0.0280) which were used in all calculations. The final R_1 was 0.0296 (I > 2σ(I)) and wR_2 was 0.0743 (all data).

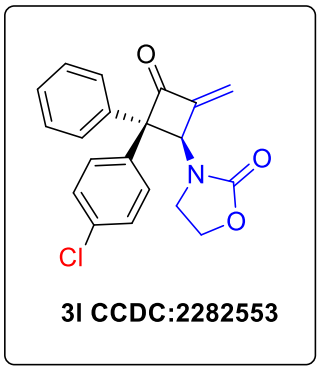
Refinement model description

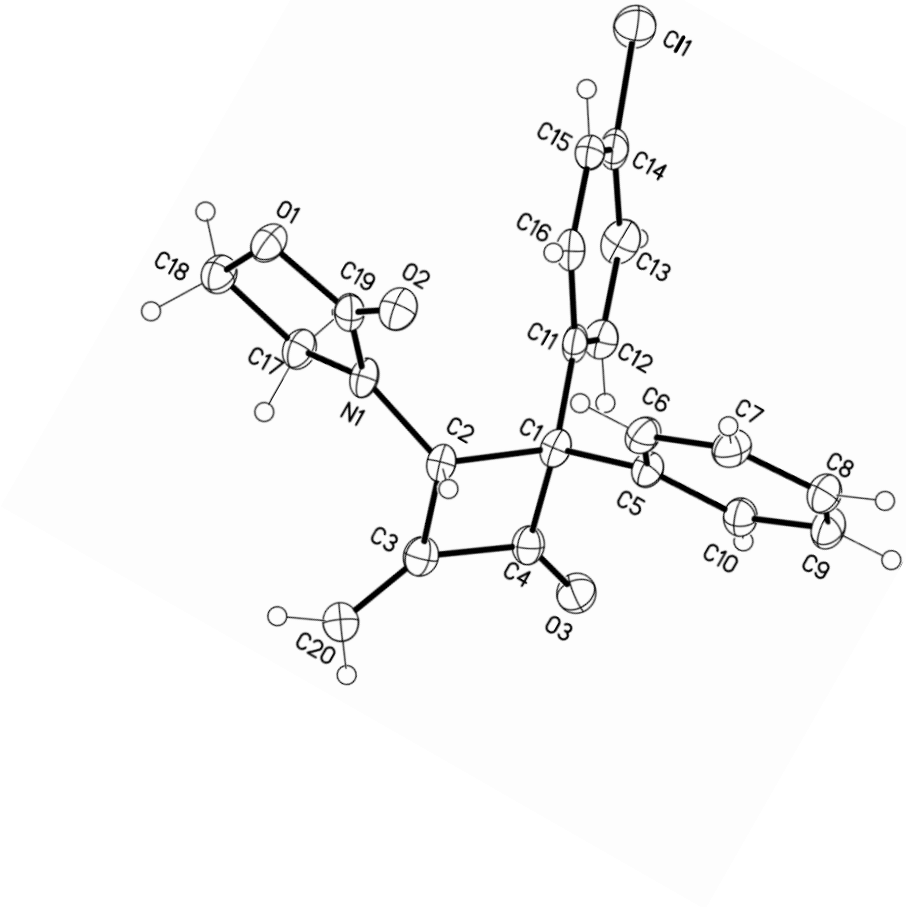
Number of restraints - 0, number of constraints - unknown.

Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups, All C(H,H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Ternary CH refined with riding coordinates:
 C10(H10)
2.b Secondary CH2 refined with riding coordinates:
 C20(H20A,H20B), C21(H21A,H21B)
2.c Aromatic/amide H refined with riding coordinates:
 C2(H2), C4(H4), C5(H5), C6(H6), C12(H12), C13(H13), C15(H15), C16(H16)
2.d X=CH2 refined with riding coordinates:
 C26(H26A,H26B)
2.e Idealised Me refined as rotating group:
 C18(H18A,H18B,H18C)
```

(b) X-ray crystallographic data of compound (3l):





Ellipsoid contour %probability level = 50%

Experimental: The sample was dissolved in appropriate amount of Dichloromethane 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.

230734lt_auto

Table S8 Crystal data and structure refinement for 230734lt_auto.

Identification code	230734lt_auto
Empirical formula	$C_{20}H_{16}ClNO_3$

Formula weight	353.79
Temperature/K	99.98(10)
Crystal system	monoclinic
Space group	Pc
a/Å	9.0167(3)
b/Å	11.7335(4)
c/Å	8.0325(2)
α/°	90
β/°	104.733(3)
γ/°	90
Volume/Å^3	821.88(4)
Z	2
ρ_{calc}g/cm^3	1.430
μ/mm^{-1}	2.223
F(000)	368.0
Crystal size/mm^3	0.12 × 0.09 × 0.04
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	7.534 to 133.126
Index ranges	$-10 \leq h \leq 10$, $-13 \leq k \leq 13$, $-9 \leq l \leq 5$
Reflections collected	5108
Independent reflections	2012 [R_{int} = 0.0316, R_{sigma} = 0.0357]
Data/restraints/parameters	2012/2/227
Goodness-of-fit on F^2	1.094
Final R indexes [I>=2σ (I)]	R_1 = 0.0408, wR_2 = 0.1030
Final R indexes [all data]	R_1 = 0.0422, wR_2 = 0.1038
Largest diff. peak/hole / e Å^{-3}	0.36/-0.44

Flack parameter 0.03(3)

Table S9 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 230734lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C1	403(4)	-2693(3)	3861(5)	19.7(8)
C2	15(4)	-1859(3)	5252(5)	19.3(8)
C3	597(5)	-2752(3)	6638(5)	21.8(8)
C4	994(4)	-3544(4)	5368(5)	20.2(8)
C5	1675(4)	-2254(4)	3070(5)	21.4(9)
C6	1750(4)	-1112(4)	2646(5)	23.4(9)
C7	2850(5)	-747(4)	1798(6)	23.9(9)
C8	3842(4)	-1536(4)	1359(5)	26.4(10)
C9	3769(5)	-2668(4)	1794(5)	26.4(9)
C10	2693(4)	-3043(4)	2656(5)	21.9(8)
C11	-953(4)	-3091(4)	2429(5)	19.4(8)
C12	-1337(5)	-4239(4)	2160(5)	22.3(9)
C13	-2568(4)	-4562(4)	803(5)	26.7(10)
C14	-3401(4)	-3732(4)	-275(5)	23.5(9)
C15	-3018(4)	-2599(4)	-33(5)	21.3(8)
C16	-1795(4)	-2285(3)	1322(5)	19.8(8)
C17	-2778(4)	-2128(3)	5486(5)	22.0(8)
C18	-3963(5)	-1180(3)	5429(5)	22.8(9)
C19	-2017(4)	-465(3)	4396(5)	19.9(8)
C20	705(5)	-2837(4)	8311(5)	25.2(9)
Cl1	-4970.8(11)	-4127.1(9)	-1928.7(12)	31.4(3)

Table S9 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 230734lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
N1	-1543(4)	-1472(3)	5069(4)	19.8(7)
O1	-3496(3)	-268(3)	4452(3)	23.4(6)
O2	-1301(3)	244(2)	3775(4)	25.8(6)
O3	1563(3)	-4483(3)	5461(4)	26.8(7)

Table S10 Anisotropic Displacement Parameters (Å^2×10^3) for 230734lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	18.1(19)	18(2)	25(2)	-1.0(16)	9.6(16)	0.1(16)
C2	18.7(18)	20(2)	21.6(18)	-1.0(16)	9.5(15)	1.0(16)
C3	21.3(19)	20(2)	25(2)	-1.0(17)	8.7(16)	-2.4(16)
C4	20.3(19)	20(2)	22.6(19)	-1.3(17)	9.3(15)	-2.4(16)
C5	16.5(19)	27(2)	22(2)	-1.1(17)	7.3(16)	0.3(16)
C6	17.3(19)	28(2)	26(2)	1.7(17)	7.4(16)	0.7(16)
C7	19(2)	25(2)	28(2)	4.1(18)	7.5(17)	-2.4(16)
C8	18(2)	37(3)	26(2)	-0.2(19)	9.8(16)	-1.5(19)
C9	19.9(19)	33(2)	29(2)	-2.2(19)	10.9(16)	-0.8(18)
C10	21.0(19)	23(2)	23(2)	-2.2(16)	7.9(16)	-1.6(16)
C11	17.7(19)	24(2)	20.3(18)	-3.0(16)	11.2(15)	0.8(16)
C12	22(2)	22(2)	25(2)	-1.4(18)	8.7(17)	-0.5(17)
C13	24(2)	22(2)	36(2)	-5.4(19)	10.3(18)	-3.0(16)
C14	21(2)	29(2)	23(2)	-4.4(17)	11.8(17)	0.7(17)
C15	17.2(18)	30(2)	18.4(18)	-2.3(17)	6.8(15)	1.4(16)

Table S10 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 230734lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C16	22.6(19)	18(2)	22.6(18)	-2.8(16)	12.3(15)	-1.9(15)
C17	20.3(19)	22(2)	27(2)	1.7(17)	10.9(16)	-0.5(17)
C18	22(2)	22(2)	25(2)	2.7(17)	9.0(16)	1.4(16)
C19	20.6(19)	21(2)	19.8(19)	-3.2(16)	8.5(15)	0.0(16)
C20	25(2)	29(2)	23(2)	-1.3(18)	7.5(16)	1.3(18)
Cl1	23.9(5)	36.9(6)	32.3(5)	-9.3(5)	5.4(4)	-5.2(4)
N1	18.6(16)	20.2(17)	24.1(17)	-0.3(14)	12.0(13)	-0.5(14)
O1	18.5(14)	24.6(16)	28.9(15)	3.1(13)	9.2(11)	2.4(12)
O2	26.4(15)	22.3(15)	30.7(16)	1.2(13)	11.1(12)	0.7(13)
O3	30.5(16)	23.6(16)	26.7(16)	2.6(12)	8.1(12)	6.1(13)

Table S11 Bond Lengths for 230734lt_auto.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	C2	1.590(5)	C9	C10	1.398(5)
C1	C4	1.555(6)	C11	C12	1.393(6)
C1	C5	1.535(5)	C11	C16	1.385(6)
C1	C11	1.523(5)	C12	C13	1.396(6)
C2	C3	1.521(6)	C13	C14	1.390(6)
C2	N1	1.447(5)	C14	C15	1.374(6)
C3	C4	1.490(6)	C14	Cl1	1.740(4)
C3	C20	1.326(6)	C15	C16	1.387(6)
C4	O3	1.209(5)	C17	C18	1.534(5)
C5	C6	1.389(6)	C17	N1	1.461(5)

Table S11 Bond Lengths for 230734lt_auto.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C5	C10	1.402(6)	C18	O1	1.451(5)
C6	C7	1.405(6)	C19	N1	1.325(5)
C7	C8	1.394(6)	C19	O1	1.366(4)
C8	C9	1.379(6)	C19	O2	1.234(5)

Table S12 Bond Angles for 230734lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C4	C1	C2	86.6(3)	C8	C9	C10	120.8(4)
C5	C1	C2	113.9(3)	C9	C10	C5	119.2(4)
C5	C1	C4	113.2(3)	C12	C11	C1	122.3(4)
C11	C1	C2	116.2(3)	C16	C11	C1	118.6(4)
C11	C1	C4	116.2(3)	C16	C11	C12	119.1(4)
C11	C1	C5	109.3(3)	C11	C12	C13	120.1(4)
C3	C2	C1	89.6(3)	C14	C13	C12	119.5(4)
N1	C2	C1	120.2(3)	C13	C14	Cl1	119.6(3)
N1	C2	C3	115.8(3)	C15	C14	C13	120.9(4)
C4	C3	C2	91.6(3)	C15	C14	Cl1	119.5(3)
C20	C3	C2	135.7(4)	C14	C15	C16	119.2(4)
C20	C3	C4	132.7(4)	C11	C16	C15	121.3(4)
C3	C4	C1	92.2(3)	N1	C17	C18	100.2(3)
O3	C4	C1	133.6(4)	O1	C18	C17	104.8(3)
O3	C4	C3	134.3(4)	N1	C19	O1	110.8(3)
C6	C5	C1	120.9(3)	O2	C19	N1	128.2(3)

Table S12 Bond Angles for 230734lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C6	C5	C10	120.2(4)	O2	C19	O1	121.0(4)
C10	C5	C1	118.7(4)	C2	N1	C17	126.1(3)
C5	C6	C7	119.8(4)	C19	N1	C2	121.7(3)
C8	C7	C6	119.9(4)	C19	N1	C17	112.2(3)
C9	C8	C7	120.0(4)	C19	O1	C18	108.2(3)

Table S13 Torsion Angles for 230734lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C1	C2	C3	C4	0.8(3)	C6	C7	C8	C9	-1.6(6)
C1	C2	C3	C20	-178.2(5)	C7	C8	C9	C10	0.9(6)
C1	C2	N1	C17	78.3(5)	C8	C9	C10	C5	0.3(6)
C1	C2	N1	C19	-98.1(4)	C10	C5	C6	C7	0.0(6)
C1	C5	C6	C7	-174.9(4)	C11	C1	C2	C3	116.8(3)
C1	C5	C10	C9	174.3(4)	C11	C1	C2	N1	-3.2(5)
C1	C11	C12	C13	-178.4(3)	C11	C1	C4	C3	-116.9(3)
C1	C11	C16	C15	178.4(3)	C11	C1	C4	O3	63.5(6)
C2	C1	C4	C3	0.8(3)	C11	C1	C5	C6	91.4(4)
C2	C1	C4	O3	-178.8(5)	C11	C1	C5	C10	-83.6(4)
C2	C1	C5	C6	-40.5(5)	C11	C12	C13	C14	0.1(6)
C2	C1	C5	C10	144.5(4)	C12	C11	C16	C15	0.7(6)
C2	C1	C11	C12	-119.8(4)	C12	C13	C14	C15	0.7(6)
C2	C1	C11	C16	62.5(5)	C12	C13	C14	Cl1	-178.1(3)
C2	C3	C4	C1	-0.8(3)	C13	C14	C15	C16	-0.9(6)

Table S13 Torsion Angles for 230734lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C2	C3	C4	O3	178.8(5)	C14	C15	C16	C11	0.2(6)
C3	C2	N1	C17	-27.6(5)	C16	C11	C12	C13	-0.8(6)
C3	C2	N1	C19	156.0(4)	C17	C18	O1	C19	-16.6(4)
C4	C1	C2	C3	-0.8(3)	C18	C17	N1	C2	167.2(3)
C4	C1	C2	N1	-120.8(4)	C18	C17	N1	C19	-16.1(4)
C4	C1	C5	C6	-137.4(4)	C20	C3	C4	C1	178.2(5)
C4	C1	C5	C10	47.6(5)	C20	C3	C4	O3	-2.2(8)
C4	C1	C11	C12	-20.1(5)	Cl1	C14	C15	C16	178.0(3)
C4	C1	C11	C16	162.3(3)	N1	C2	C3	C4	124.5(3)
C5	C1	C2	C3	-114.7(3)	N1	C2	C3	C20	-54.4(6)
C5	C1	C2	N1	125.3(4)	N1	C17	C18	O1	18.9(4)
C5	C1	C4	C3	115.4(3)	N1	C19	O1	C18	6.9(4)
C5	C1	C4	O3	-64.2(6)	O1	C19	N1	C2	-176.5(3)
C5	C1	C11	C12	109.5(4)	O1	C19	N1	C17	6.6(5)
C5	C1	C11	C16	-68.1(4)	O2	C19	N1	C2	3.7(6)
C5	C6	C7	C8	1.2(6)	O2	C19	N1	C17	-173.2(4)
C6	C5	C10	C9	-0.8(6)	O2	C19	O1	C18	-173.2(3)

Table S14 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 230734lt_auto.

Atom	*x*	*y*	*z*	U(eq)
H2	728.31	-1191.86	5440.41	23
H6	1059.32	-578.33	2927.63	28
H7	2917.73	36.04	1525.05	29

Table S14 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 230734lt_auto.

Atom	*x*	*y*	*z*	U(eq)
H8	4568.45	-1294.5	758.67	32
H9	4458.04	-3199.41	1505.08	32
H10	2652.5	-3823.19	2956.7	26
H12	-761.52	-4801.39	2900.43	27
H13	-2833.73	-5343.7	617.17	32
H15	-3584.16	-2037.76	-783.98	26
H16	-1529.87	-1502.84	1492.89	24
H17A	-2449.46	-2479.34	6641.88	26
H17B	-3173.04	-2726.28	4617.56	26
H18A	-5007.65	-1449.31	4852.27	27
H18B	-3949.91	-920.82	6605.26	27
H20A	1104.44	-3511.85	8913.62	30
H20B	383.09	-2223.14	8905.86	30

Experimental

Single crystals of $C_{20}H_{16}ClNO_3$ **[230734lt_auto]** were **[]**. A suitable crystal was selected and **[]** on a **XtaLAB Synergy R, DW system, HyPix-Arc 150** diffractometer. The crystal was kept at 99.98(10) K during data collection. Using Olex2 [1], the structure was solved with the SHELXS [2] structure solution program using Direct Methods and refined with the SHELXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [230734lt_auto]

Crystal Data for $C_{20}H_{16}ClNO_3$ (M =353.79 g/mol): monoclinic, space group Pc (no. 7), a = 9.0167(3) Å, b = 11.7335(4) Å, c = 8.0325(2) Å, β = 104.733(3)°, V = 821.88(4) Å^3, Z = 2, T = 99.98(10) K, μ(Cu Kα) = 2.223 mm^{-1}, *Dcalc* = 1.430 g/cm^3, 5108 reflections measured (7.534° ≤ 2Θ ≤ 133.126°), 2012 unique (R_{int} = 0.0316, R_{sigma} = 0.0357) which were used in all calculations. The final R_1 was 0.0408 (I > 2σ(I)) and wR_2 was 0.1038 (all data).

Refinement model description

Number of restraints - 2, number of constraints - unknown.

Details:

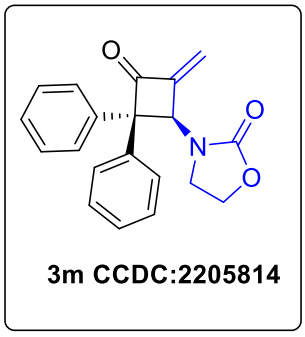
```
1. Twinned data refinement
 Scales: 0.97(3)
```

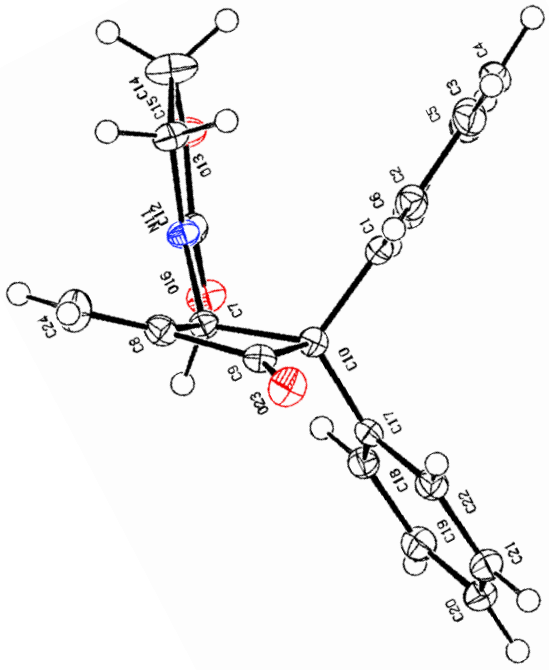
```
 0.03(3)
2. Fixed Uiso
 At 1.2 times of:
  All C(H) groups, All C(H,H) groups
3.a Ternary CH refined with riding coordinates:
 C2(H2)
3.b Secondary CH2 refined with riding coordinates:
 C17(H17A,H17B), C18(H18A,H18B)
3.c Aromatic/amide H refined with riding coordinates:
 C6(H6), C7(H7), C8(H8), C9(H9), C10(H10), C12(H12), C13(H13), C15(H15),
 C16(H16)
3.d X=CH2 refined with riding coordinates:
 C20(H20A,H20B)
```

(c) X-ray crystallographic data of compound (3m):

Ellipsoid contour %probability level = 50%

Experimental: The sample was dissolved in appropriate amount of Dichloromethane 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.





220677lt_auto

Table S15 Crystal data and structure refinement for 220677lt_auto.

Identification code	220677lt_auto
Empirical formula	$C_{20}H_{17}NO_3$
Formula weight	319.35
Temperature/K	100.00(10)
Crystal system	orthorhombic
Space group	Pbca
a/Å	8.86145(10)
b/Å	18.8062(2)
c/Å	19.06566(19)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	3177.30(6)
Z	8
ρ_{calc}g/cm^3	1.335
μ/mm^{-1}	0.729
F(000)	1344.0
Crystal size/mm^3	0.23 × 0.22 × 0.19
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	9.278 to 134.15
Index ranges	$-10 \leq h \leq 10$, $-22 \leq k \leq 22$, $-17 \leq l \leq 22$
Reflections collected	23337
Independent reflections	2834 [R_{int} = 0.0211, R_{sigma} = 0.0125]

Data/restraints/parameters	2834/0/218
Goodness-of-fit on F^2	1.039
Final R indexes [I>=2σ (I)]	R_1 = 0.0325, wR_2 = 0.0784
Final R indexes [all data]	R_1 = 0.0340, wR_2 = 0.0793
Largest diff. peak/hole / e $Å^{-3}$	0.26/-0.17

Table S16 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters ($Å^2$×10^3) for 220677lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C1	3713.6(13)	3564.0(6)	3862.6(6)	16.6(2)
C2	5189.9(14)	3397.2(7)	4046.9(6)	21.5(3)
C3	6392.9(14)	3751.6(8)	3735.9(7)	26.8(3)
C4	6137.3(15)	4278.2(7)	3244.3(7)	27.0(3)
C5	4668.7(15)	4435.1(7)	3040.3(6)	23.9(3)
C6	3465.1(14)	4077.3(6)	3344.0(6)	19.9(3)
C7	1339.7(13)	3735.8(6)	4666.9(6)	15.7(2)
C8	1926.0(13)	3376.3(6)	5329.2(6)	18.6(3)
C9	2733.8(13)	2829.7(6)	4910.7(6)	17.7(3)
C10	2362.9(13)	3207.1(6)	4206.6(6)	15.8(2)
C12	809.3(13)	4904.5(6)	4168.0(6)	17.8(3)
C14	2721.1(16)	5594.5(7)	4597.2(8)	30.4(3)
C15	2876.5(14)	4862.7(6)	4927.5(6)	20.0(3)
C17	1526.9(13)	2742.8(6)	3681.7(6)	16.1(2)
C18	297.3(13)	2994.4(6)	3302.6(6)	18.7(3)
C19	-401.6(14)	2565.4(7)	2805.0(6)	21.9(3)
C20	123.5(14)	1879.7(7)	2684.5(6)	22.6(3)

Table S16 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 220677lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C21	1352.1(14)	1628.5(6)	3057.6(6)	22.1(3)
C22	2058.7(13)	2055.7(6)	3552.4(6)	19.2(3)
C24	1883.5(15)	3508.8(7)	6010.3(6)	25.5(3)
N11	1594.6(11)	4491.9(5)	4620.1(5)	16.2(2)
O13	1419.6(10)	5565.9(4)	4144.7(5)	23.2(2)
O16	-286.6(9)	4742.4(4)	3822.1(4)	22.3(2)
O23	3382.6(10)	2285.1(4)	5056.8(4)	24.0(2)

Table S17 Anisotropic Displacement Parameters (Å^2×10^3) for 220677lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	17.2(6)	15.9(6)	16.9(5)	-3.8(4)	0.8(4)	-0.8(4)
C2	18.7(6)	24.6(6)	21.3(6)	-3.0(5)	-0.4(5)	1.9(5)
C3	16.1(6)	38.8(8)	25.7(6)	-8.1(6)	0.6(5)	-1.3(5)
C4	23.6(6)	35.0(7)	22.3(6)	-7.5(5)	6.8(5)	-11.6(6)
C5	28.8(7)	23.5(6)	19.5(6)	-0.8(5)	4.0(5)	-5.4(5)
C6	19.4(6)	20.7(6)	19.6(6)	-1.1(5)	1.0(5)	-0.5(5)
C7	16.8(5)	13.1(5)	17.1(5)	0.5(4)	0.8(4)	0.0(4)
C8	19.2(6)	15.8(6)	20.8(6)	3.7(5)	0.7(5)	0.1(5)
C9	17.3(6)	15.8(6)	19.9(6)	1.4(5)	-1.0(5)	-1.8(5)
C10	15.2(6)	14.6(5)	17.5(5)	0.7(4)	-0.2(4)	1.8(4)
C12	19.6(6)	14.8(5)	19.0(6)	-0.3(4)	1.9(5)	1.5(5)
C14	30.8(7)	21.4(7)	39.1(8)	4.8(6)	-14.9(6)	-6.7(6)

Table S17 Anisotropic Displacement Parameters ($Å^2×10^3$) for 220677lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C15	19.8(6)	16.6(6)	23.7(6)	-2.1(5)	-3.8(5)	-1.7(5)
C17	16.6(5)	16.1(6)	15.8(5)	1.4(4)	3.0(4)	-2.5(4)
C18	19.2(6)	16.9(6)	20.1(6)	0.5(5)	1.5(5)	0.1(5)
C19	20.7(6)	24.4(6)	20.4(6)	1.5(5)	-1.9(5)	-1.0(5)
C20	24.7(6)	23.1(6)	19.9(6)	-3.5(5)	0.5(5)	-6.0(5)
C21	25.7(6)	16.2(6)	24.4(6)	-2.0(5)	3.7(5)	-1.1(5)
C22	19.4(6)	17.4(6)	20.8(6)	1.8(5)	0.7(5)	0.0(5)
C24	31.2(7)	25.0(6)	20.3(6)	2.7(5)	0.3(5)	6.2(5)
N11	16.8(5)	13.3(5)	18.3(5)	0.2(4)	-2.1(4)	0.0(4)
O13	25.4(5)	14.5(4)	29.6(5)	3.4(3)	-6.1(4)	-1.9(3)
O16	21.4(4)	19.7(4)	25.8(4)	0.7(3)	-6.0(4)	1.1(3)
O23	27.6(5)	19.3(4)	25.1(5)	3.0(4)	-3.0(4)	6.2(4)

Table S18 Bond Lengths for 220677lt_auto.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	C2	1.3904(17)	C10	C17	1.5206(16)
C1	C6	1.3993(17)	C12	N11	1.3524(15)
C1	C10	1.5209(16)	C12	O13	1.3570(14)
C2	C3	1.3900(18)	C12	O16	1.2129(15)
C3	C4	1.382(2)	C14	C15	1.5198(17)
C4	C5	1.3900(19)	C14	O13	1.4412(15)
C5	C6	1.3877(17)	C15	N11	1.4561(15)
C7	C8	1.5236(16)	C17	C18	1.3906(16)

Table S18 Bond Lengths for 220677lt_auto.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C7	C10	1.6065(15)	C17	C22	1.3974(16)
C7	N11	1.4426(14)	C18	C19	1.3907(17)
C8	C9	1.4851(16)	C19	C20	1.3902(18)
C8	C24	1.3229(17)	C20	C21	1.3837(18)
C9	C10	1.5537(15)	C21	C22	1.3884(17)
C9	O23	1.2071(14)			

Table S19 Bond Angles for 220677lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	C1	C6	118.83(11)	C17	C10	C1	110.67(9)
C2	C1	C10	122.14(10)	C17	C10	C7	116.10(9)
C6	C1	C10	119.03(10)	C17	C10	C9	114.17(9)
C3	C2	C1	120.38(12)	N11	C12	O13	109.98(10)
C4	C3	C2	120.47(12)	O16	C12	N11	127.90(11)
C3	C4	C5	119.69(12)	O16	C12	O13	122.12(10)
C6	C5	C4	119.99(12)	O13	C14	C15	106.66(10)
C5	C6	C1	120.56(11)	N11	C15	C14	101.32(9)
C8	C7	C10	89.18(8)	C18	C17	C10	121.90(10)
N11	C7	C8	115.80(9)	C18	C17	C22	119.15(11)
N11	C7	C10	119.21(9)	C22	C17	C10	118.87(10)
C9	C8	C7	91.50(9)	C17	C18	C19	120.41(11)
C24	C8	C7	136.08(11)	C20	C19	C18	120.12(11)
C24	C8	C9	132.18(11)	C21	C20	C19	119.68(11)

Table S19 Bond Angles for 220677lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C8	C9	C10	92.64(9)	C20	C21	C22	120.44(11)
O23	C9	C8	133.86(11)	C21	C22	C17	120.20(11)
O23	C9	C10	133.45(11)	C7	N11	C15	124.70(9)
C1	C10	C7	113.99(9)	C12	N11	C7	121.63(10)
C1	C10	C9	114.06(9)	C12	N11	C15	112.53(9)
C9	C10	C7	85.99(8)	C12	O13	C14	109.49(9)

Table S20 Torsion Angles for 220677lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C1	C2	C3	C4	0.56(19)	C10	C7	C8	C24	168.32(15)
C1	C10	C17	C18	-94.03(12)	C10	C7	N11	C12	93.20(13)
C1	C10	C17	C22	82.72(12)	C10	C7	N11	C15	-73.67(14)
C2	C1	C6	C5	-2.71(17)	C10	C17	C18	C19	177.25(11)
C2	C1	C10	C7	114.13(12)	C10	C17	C22	C21	−177.68(10)
C2	C1	C10	C9	17.54(15)	C14	C15	N11	C7	169.40(11)
C2	C1	C10	C17	−112.82(12)	C14	C15	N11	C12	1.49(13)
C2	C3	C4	C5	-2.47(19)	C15	C14	O13	C12	-0.53(14)
C3	C4	C5	C6	1.77(19)	C17	C18	C19	C20	0.18(18)
C4	C5	C6	C1	0.84(18)	C18	C17	C22	C21	-0.83(17)
C6	C1	C2	C3	2.01(17)	C18	C19	C20	C21	-0.54(18)
C6	C1	C10	C7	-65.83(13)	C19	C20	C21	C22	0.21(18)
C6	C1	C10	C9	−162.43(10)	C20	C21	C22	C17	0.48(18)

Table S20 Torsion Angles for 220677lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C6	C1	C10	C17	67.21(13)	C22	C17	C18	C19	0.51(17)
C7	C8	C9	C10	6.53(9)	C24	C8	C9	C10	−168.45(14)
C7	C8	C9	O23	−171.00(14)	C24	C8	C9	O23	14.0(2)
C7	C10	C17	C18	37.94(15)	N11	C7	C8	C9	−128.66(10)
C7	C10	C17	C22	−145.31(10)	N11	C7	C8	C24	46.0(2)
C8	C7	C10	C1	−108.55(10)	N11	C7	C10	C1	10.84(14)
C8	C7	C10	C9	6.04(8)	N11	C7	C10	C9	125.42(10)
C8	C7	C10	C17	121.04(10)	N11	C7	C10	C17	−119.58(11)
C8	C7	N11	C12	−162.20(10)	N11	C12	O13	C14	1.50(13)
C8	C7	N11	C15	30.93(15)	O13	C12	N11	C7	−170.28(10)
C8	C9	C10	C1	108.32(10)	O13	C12	N11	C15	−1.95(13)
C8	C9	C10	C7	−6.20(9)	O13	C14	C15	N11	−0.55(13)
C8	C9	C10	C17	−123.07(10)	O16	C12	N11	C7	10.02(19)
C9	C10	C17	C18	135.66(11)	O16	C12	N11	C15	178.35(12)
C9	C10	C17	C22	−47.59(14)	O16	C12	O13	C14	−178.78(12)
C10	C1	C2	C3	−177.96(11)	O23	C9	C10	C1	−74.13(16)
C10	C1	C6	C5	177.26(11)	O23	C9	C10	C7	171.35(14)
C10	C7	C8	C9	−6.31(9)	O23	C9	C10	C17	54.48(18)

Table S21 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 220677lt_auto.

Atom	*x*	*y*	*z*	U(eq)
H2	5377.46	3039.06	4387.24	26
H3	7397.93	3630.98	3862.09	32
H4	6961.48	4531.65	3046.75	32
H5	4488.91	4787.29	2692.99	29
H6	2463.91	4181.67	3198.57	24
H7	245.61	3625.81	4598.06	19
H14A	2580.44	5960.83	4964.32	37
H14B	3636.37	5713.23	4323.37	37
H15A	3843.46	4634.65	4797.08	24
H15B	2796.87	4885.9	5444.95	24
H18	-67.66	3462.14	3383.94	22
H19	-1239.41	2741.38	2547.08	26
H20	-358.71	1584.97	2347.54	27
H21	1714.46	1160.54	2974.75	26
H22	2906.32	1880.44	3803.77	23
H24A	2448.16	3222.45	6325.9	31
H24B	1288.3	3890.36	6183.86	31

Experimental

Single crystals of $C_{20}H_{17}NO_3$ **[220677lt_auto]** were **[]**. A suitable crystal was selected and **[]** on a **XtaLAB Synergy R, DW system, HyPix-Arc 150** diffractometer. The crystal was kept at 100.00(10) K during data collection. Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program using Intrinsic Phasing and refined with the SHELXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [220677lt_auto]

Crystal Data for $C_{20}H_{17}NO_3$ (*M* =319.35 g/mol): orthorhombic, space group Pbca (no. 61), *a* = 8.86145(10) Å, *b* = 18.8062(2) Å, *c* = 19.06566(19) Å, *V* = 3177.30(6) Å^3, *Z* = 8, *T* = 100.00(10) K, μ(Cu Kα) = 0.729 mm^{-1}, *Dcalc* = 1.335 g/cm^3, 23337 reflections measured (9.278° ≤ 2Θ ≤ 134.15°), 2834 unique (R_{int} = 0.0211, R_{sigma} = 0.0125) which were used in all calculations. The final R_1 was 0.0325 (I > 2σ(I)) and wR_2 was 0.0793 (all data).

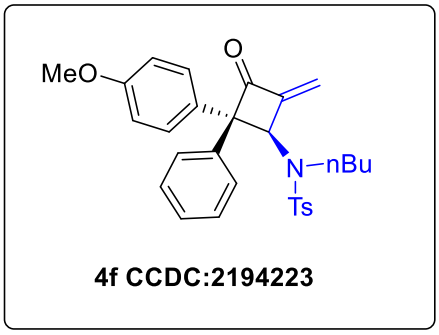
Refinement model description

Number of restraints - 0, number of constraints - unknown.

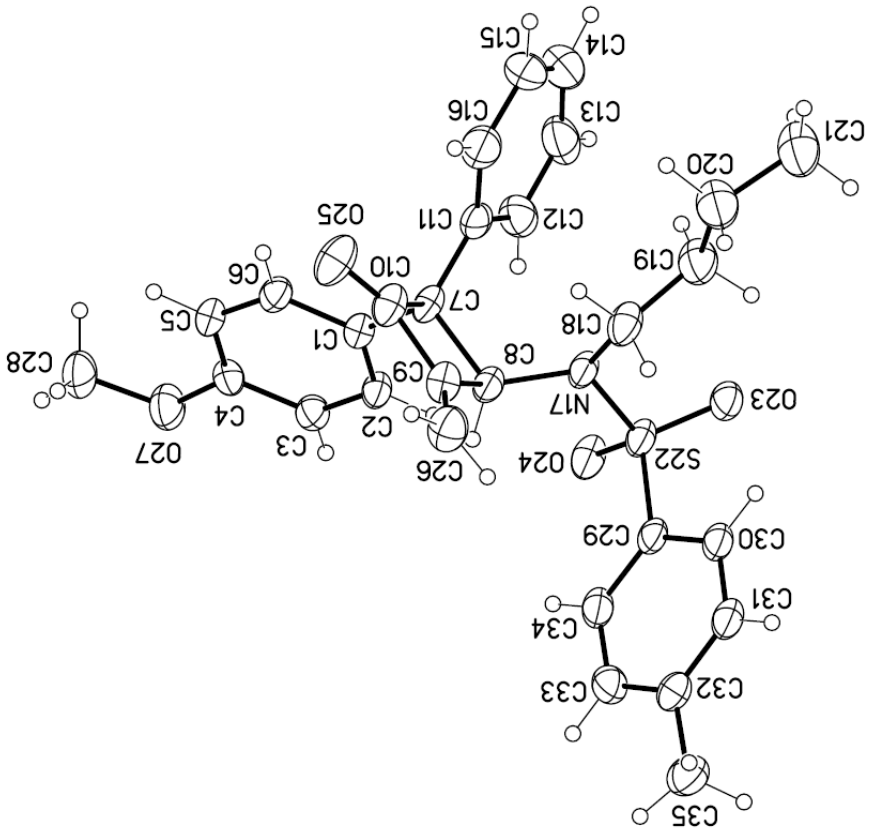
Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups, All C(H,H) groups
2.a Ternary CH refined with riding coordinates:
 C7(H7)
2.b Secondary CH2 refined with riding coordinates:
 C14(H14A,H14B), C15(H15A,H15B)
2.c Aromatic/amide H refined with riding coordinates:
 C2(H2), C3(H3), C4(H4), C5(H5), C6(H6), C18(H18), C19(H19), C20(H20),
 C21(H21), C22(H22)
2.d X=CH2 refined with riding coordinates:
 C24(H24A,H24B)
```

(d) X-ray crystallographic data of compound (4f):



4f CCDC:2194223



Ellipsoid contour %probability level = 50%

Experimental: The sample was dissolved in appropriate amount of Dichloromethane 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.

220766lt_auto

Table S22 Crystal data and structure refinement for 220766lt_auto.

Identification code	220766lt_auto
Empirical formula	$C_{29}H_{29}NO_4S$
Formula weight	487.59
Temperature/K	99.99(10)
Crystal system	triclinic
Space group	P-1
a/Å	7.73568(18)
b/Å	12.3470(3)
c/Å	14.4133(3)
α/°	69.991(2)
β/°	83.0268(19)
γ/°	79.978(2)
Volume/Å^3	1270.97(5)
Z	2
ρ_{calc}g/cm^3	1.274
μ/mm^{-1}	1.414
F(000)	516.0

Crystal size/mm^3	0.13 × 0.11 × 0.08
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	6.542 to 134.038
Index ranges	-9 ≤ h ≤ 9, -14 ≤ k ≤ 14, -17 ≤ l ≤ 15
Reflections collected	13857
Independent reflections	4513 [R_{int} = 0.0206, R_{sigma} = 0.0239]
Data/restraints/parameters	4513/0/320
Goodness-of-fit on F^2	1.065
Final R indexes [I>=2σ (I)]	R_1 = 0.0593, wR_2 = 0.1650
Final R indexes [all data]	R_1 = 0.0639, wR_2 = 0.1686
Largest diff. peak/hole / e Å^{-3}	0.83/-0.40

Table S23 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 220766lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C1	4732(3)	7877(2)	3994.7(19)	27.2(6)
C2	2928(3)	7971(2)	3909.8(19)	28.8(6)
C3	1714(4)	8385(2)	4540.3(19)	31.3(6)
C4	2279(4)	8704(2)	5278.5(19)	31.6(6)
C5	4062(4)	8641(3)	5359(2)	33.6(6)
C6	5267(4)	8234(2)	4717.1(19)	30.4(6)
C7	6116(3)	7426(2)	3312(2)	28.8(6)
C8	5594(4)	6351(2)	3080.0(19)	29.5(6)
C9	7044(4)	5547(3)	3722(2)	34.3(6)
C10	7593(4)	6547(3)	3911(2)	35.6(6)
C11	6711(4)	8407(3)	2424(2)	34.3(6)

Table S23 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 220766lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C12	5489(4)	9188(3)	1805(2)	41.2(7)
C13	5974(5)	10119(3)	998(2)	51.3(9)
C14	7717(6)	10277(3)	791(3)	58.1(10)
C15	8947(5)	9499(4)	1361(3)	55.2(9)
C16	8482(4)	8554(3)	2190(3)	48.2(8)
C18	7442(4)	6118(3)	1574(2)	39.1(7)
C19	7880(4)	6822(3)	529(2)	47.4(8)
C20	9848(4)	6567(4)	268(3)	54.9(9)
C21	10330(5)	7125(4)	-823(3)	58.9(10)
C26	7629(5)	4411(3)	4020(2)	44.4(7)
C28	1596(5)	9296(3)	6705(2)	48.8(8)
C29	3641(3)	4868(3)	1994.1(19)	30.6(6)
C30	4563(4)	4155(3)	1476(2)	32.3(6)
C31	4353(4)	2996(3)	1782(2)	35.0(6)
C32	3215(4)	2523(3)	2597(2)	35.7(6)
C33	2297(4)	3252(3)	3101(2)	38.5(7)
C34	2505(4)	4413(3)	2814(2)	35.5(6)
C35	2971(5)	1264(3)	2897(2)	45.8(8)
N17	5711(3)	6444(2)	2032.4(16)	30.3(5)
O23	4182(2)	6724.9(18)	536.8(14)	35.1(5)
O24	2484(2)	6939.5(18)	2052.0(15)	37.0(5)
O25	8743(3)	6644(2)	4364.8(18)	48.4(6)
O27	1013(3)	9034(2)	5911.0(15)	43.6(5)

Table S23 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for 220766lt_auto. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
S22	3910.1(8)	6350.0(6)	1592.2(5)	30.6(2)

Table S24 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 220766lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	29.1(13)	29.7(13)	25.2(12)	-11.5(10)	-4.1(10)	-3.8(10)
C2	29.9(13)	34.8(14)	25.6(12)	-13.7(11)	-5.4(10)	-4.9(11)
C3	28.8(14)	37.9(15)	28.7(13)	-12.4(11)	-0.6(11)	-7.0(11)
C4	37.6(15)	34.3(14)	24.2(13)	-11.9(11)	3.9(11)	-8.1(11)
C5	40.6(16)	39.8(15)	27.1(13)	-15.9(12)	-4.0(11)	-11.3(12)
C6	29.4(14)	35.4(14)	30.3(13)	-13.6(11)	-6.1(11)	-6.4(11)
C7	25.6(13)	34.6(14)	32.9(14)	-18.6(12)	-7.6(11)	-2.5(11)
C8	29.9(13)	34.2(14)	30.2(13)	-16.7(11)	-3.8(11)	-5.8(11)
C9	36.3(15)	37.1(15)	32.2(14)	-15.5(12)	-5.4(12)	-1.6(12)
C10	31.5(15)	42.3(16)	38.2(15)	-20.3(13)	-9.0(12)	0.1(12)
C11	38.1(15)	40.3(16)	35.1(15)	-25.5(13)	6.5(12)	-12.9(12)
C12	46.0(17)	41.5(17)	36.7(16)	-15.5(13)	3.5(13)	-6.5(13)
C13	74(2)	44.7(18)	34.7(16)	-15.7(14)	6.1(16)	-8.0(17)
C14	79(3)	57(2)	41.0(18)	-15.0(16)	9.1(18)	-25(2)
C15	55(2)	73(2)	46.6(19)	-26.3(18)	17.7(17)	-35.2(19)
C16	42.3(18)	65(2)	46.6(18)	-27.9(17)	3.3(14)	-15.9(16)
C18	26.5(14)	59.1(19)	42.0(16)	-29.7(15)	-4.4(12)	-4.8(13)
C19	35.1(16)	71(2)	41.1(17)	-22.7(16)	-3.9(13)	-9.6(15)

Table S24 Anisotropic Displacement Parameters ($Å^2×10^3$) for 220766lt_auto. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C20	32.0(17)	93(3)	43.7(18)	-25.0(19)	-1.7(14)	-14.6(17)
C21	35.9(18)	99(3)	44.8(19)	-28(2)	-4.6(14)	-7.6(18)
C26	52.1(19)	38.7(17)	40.5(17)	-12.8(14)	-5.6(14)	-0.2(14)
C28	53(2)	65(2)	37.9(17)	-30.2(16)	5.3(14)	-12.4(17)
C29	26.2(13)	44.4(16)	28.9(13)	-18.8(12)	-3.6(11)	-9.8(11)
C30	28.5(14)	46.3(16)	29.1(13)	-19.6(12)	-2.0(11)	-9.0(12)
C31	33.6(15)	45.9(17)	33.2(15)	-21.5(13)	-6.0(12)	-6.0(12)
C32	33.5(15)	45.4(17)	32.9(14)	-13.8(13)	-11.7(12)	-9.6(12)
C33	33.1(15)	54.3(19)	30.4(14)	-14.1(13)	0.1(12)	-13.2(13)
C34	29.6(14)	54.3(18)	30.0(14)	-22.3(13)	-0.5(11)	-9.2(13)
C35	49.1(19)	46.6(18)	45.1(18)	-12.6(15)	-13.0(14)	-14.2(15)
N17	27.0(11)	41.2(13)	31.1(12)	-21.3(10)	-3.4(9)	-6.8(10)
O23	31.2(10)	48.6(12)	30.3(10)	-16.9(9)	-4.7(8)	-8.9(9)
O24	28.1(10)	50.8(12)	40.9(11)	-27.8(10)	-5.2(8)	-0.3(9)
O25	40.2(12)	55.6(14)	59.4(14)	-29.7(12)	-22.9(11)	2.9(10)
O27	40.8(12)	58.6(14)	37.2(11)	-25.3(10)	2.2(9)	-5.5(10)
S22	25.4(4)	43.3(4)	31.3(4)	-21.5(3)	-3.6(3)	-5.8(3)

Table S25 Bond Lengths for 220766lt_auto.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	C2	1.397(4)	C13	C14	1.378(6)
C1	C6	1.390(4)	C14	C15	1.355(6)
C1	C7	1.528(4)	C15	C16	1.417(5)

Table S25 Bond Lengths for 220766lt_auto.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C2	C3	1.382(4)	C18	C19	1.491(4)
C3	C4	1.393(4)	C18	N17	1.474(3)
C4	C5	1.384(4)	C19	C20	1.528(4)
C4	O27	1.361(3)	C20	C21	1.514(5)
C5	C6	1.386(4)	C28	O27	1.431(4)
C7	C8	1.600(4)	C29	C30	1.394(4)
C7	C10	1.548(4)	C29	C34	1.396(4)
C7	C11	1.519(4)	C29	S22	1.762(3)
C8	C9	1.529(4)	C30	C31	1.379(4)
C8	N17	1.467(3)	C31	C32	1.397(4)
C9	C10	1.489(4)	C32	C33	1.394(4)
C9	C26	1.330(4)	C32	C35	1.504(4)
C10	O25	1.211(3)	C33	C34	1.382(4)
C11	C12	1.385(4)	N17	S22	1.636(2)
C11	C16	1.398(4)	O23	S22	1.431(2)
C12	C13	1.395(5)	O24	S22	1.435(2)

Table S26 Bond Angles for 220766lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	C1	C7	122.5(2)	C11	C12	C13	122.0(3)
C6	C1	C2	118.0(2)	C14	C13	C12	120.0(4)
C6	C1	C7	119.5(2)	C15	C14	C13	119.2(3)
C3	C2	C1	120.9(2)	C14	C15	C16	121.7(3)

Table S26 Bond Angles for 220766lt_auto.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	C3	C4	120.2(3)	C11	C16	C15	119.5(3)
C5	C4	C3	119.7(2)	N17	C18	C19	117.5(3)
O27	C4	C3	117.0(3)	C18	C19	C20	110.0(3)
O27	C4	C5	123.3(2)	C21	C20	C19	112.3(3)
C4	C5	C6	119.6(2)	C30	C29	C34	120.2(3)
C5	C6	C1	121.7(2)	C30	C29	S22	119.2(2)
C1	C7	C8	112.7(2)	C34	C29	S22	120.6(2)
C1	C7	C10	110.8(2)	C31	C30	C29	119.6(3)
C10	C7	C8	86.6(2)	C30	C31	C32	121.2(3)
C11	C7	C1	112.1(2)	C31	C32	C35	120.1(3)
C11	C7	C8	116.2(2)	C33	C32	C31	118.4(3)
C11	C7	C10	116.1(2)	C33	C32	C35	121.5(3)
C9	C8	C7	89.2(2)	C34	C33	C32	121.3(3)
N17	C8	C7	116.0(2)	C33	C34	C29	119.3(3)
N17	C8	C9	117.9(2)	C8	N17	C18	118.2(2)
C10	C9	C8	91.4(2)	C8	N17	S22	116.52(18)
C26	C9	C8	136.0(3)	C18	N17	S22	120.03(18)
C26	C9	C10	132.6(3)	C4	O27	C28	117.0(2)
C9	C10	C7	92.7(2)	N17	S22	C29	107.67(12)
O25	C10	C7	133.2(3)	O23	S22	C29	107.51(12)
O25	C10	C9	134.1(3)	O23	S22	N17	107.48(11)
C12	C11	C7	120.1(3)	O23	S22	O24	119.75(12)
C12	C11	C16	117.6(3)	O24	S22	C29	107.46(13)
C16	C11	C7	122.3(3)	O24	S22	N17	106.46(11)

Table S27 Torsion Angles for 220766lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C1	C2	C3	C4	-0.6(4)	C10	C7	C8	N17	-118.9(2)
C1	C7	C8	C9	-109.1(2)	C10	C7	C11	C12	175.6(2)
C1	C7	C8	N17	130.0(2)	C10	C7	C11	C16	-3.6(4)
C1	C7	C10	C9	110.8(2)	C11	C7	C8	C9	119.6(2)
C1	C7	C10	O25	-68.6(4)	C11	C7	C8	N17	-1.3(3)
C1	C7	C11	C12	-55.7(3)	C11	C7	C10	C9	-119.8(2)
C1	C7	C11	C16	125.2(3)	C11	C7	C10	O25	60.8(4)
C2	C1	C6	C5	1.9(4)	C11	C12	C13	C14	0.6(5)
C2	C1	C7	C8	-40.1(3)	C12	C11	C16	C15	1.9(4)
C2	C1	C7	C10	-135.3(3)	C12	C13	C14	C15	2.1(5)
C2	C1	C7	C11	93.2(3)	C13	C14	C15	C16	-2.7(6)
C2	C3	C4	C5	2.0(4)	C14	C15	C16	C11	0.7(5)
C2	C3	C4	O27	-176.0(2)	C16	C11	C12	C13	-2.6(4)
C3	C4	C5	C6	-1.4(4)	C18	C19	C20	C21	171.9(3)
C3	C4	O27	C28	176.4(3)	C18	N17	S22	C29	76.4(2)
C4	C5	C6	C1	-0.6(4)	C18	N17	S22	O23	-39.1(3)
C5	C4	O27	C28	-1.5(4)	C18	N17	S22	O24	-168.6(2)
C6	C1	C2	C3	-1.4(4)	C19	C18	N17	C8	-142.8(3)
C6	C1	C7	C8	141.4(2)	C19	C18	N17	S22	63.7(3)
C6	C1	C7	C10	46.3(3)	C26	C9	C10	C7	-177.4(3)
C6	C1	C7	C11	-85.3(3)	C26	C9	C10	O25	2.0(6)
C7	C1	C2	C3	-179.9(2)	C29	C30	C31	C32	0.8(4)
C7	C1	C6	C5	-179.5(2)	C30	C29	C34	C33	-0.4(4)

Table S27 Torsion Angles for 220766lt_auto.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C7	C8	C9	C10	-2.2(2)	C30	C29	S22	N17	-82.0(2)
C7	C8	C9	C26	177.5(4)	C30	C29	S22	O23	33.5(2)
C7	C8	N17	C18	82.9(3)	C30	C29	S22	O24	163.6(2)
C7	C8	N17	S22	-122.6(2)	C30	C31	C32	C33	-0.3(4)
C7	C11	C12	C13	178.3(3)	C30	C31	C32	C35	178.2(3)
C7	C11	C16	C15	-179.0(3)	C31	C32	C33	C34	-0.5(4)
C8	C7	C10	C9	-2.1(2)	C32	C33	C34	C29	0.9(4)
C8	C7	C10	O25	178.4(4)	C34	C29	C30	C31	-0.4(4)
C8	C7	C11	C12	75.9(3)	C34	C29	S22	N17	98.7(2)
C8	C7	C11	C16	-103.2(3)	C34	C29	S22	O23	-145.8(2)
C8	C9	C10	C7	2.2(2)	C34	C29	S22	O24	-15.6(3)
C8	C9	C10	O25	-178.3(4)	C35	C32	C33	C34	-179.0(3)
C8	N17	S22	C29	-77.5(2)	N17	C8	C9	C10	117.1(2)
C8	N17	S22	O23	166.91(19)	N17	C8	C9	C26	-63.2(5)
C8	N17	S22	O24	37.4(2)	N17	C18	C19	C20	165.8(3)
C9	C8	N17	C18	-21.1(4)	O27	C4	C5	C6	176.4(3)
C9	C8	N17	S22	133.4(2)	S22	C29	C30	C31	-179.7(2)
C10	C7	C8	C9	2.1(2)	S22	C29	C34	C33	178.8(2)

Table S28 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 220766lt_auto.

Atom	*x*	*y*	*z*	U(eq)
H2	2529.38	7748.91	3412.3	35
H3	491.44	8450.61	4469.82	38

Table S28 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 220766lt_auto.

Atom	*x*	*y*	*z*	U(eq)
H5	4457.37	8873.96	5850.87	40
H6	6487.92	8198.97	4772.32	36
H8	4406.18	6174.74	3397.95	35
H12	4283.4	9085.92	1934.7	49
H13	5102.4	10644.65	591.19	62
H14	8051.01	10924.46	255.71	70
H15	10153.5	9589.18	1200.19	66
H16	9365.49	8023.37	2583.72	58
H18A	7526.12	5296.22	1607.14	47
H18B	8356.48	6152.72	1983.91	47
H19A	7551.2	7660.79	441.68	57
H19B	7202.78	6629.96	80.14	57
H20A	10510.44	6859.82	656.9	66
H20B	10201.27	5713.97	454.28	66
H21A	9818.55	6753.78	-1205.95	88
H21B	11613.39	7024.72	-942.17	88
H21C	9870.83	7957.22	-1027.31	88
H28A	2317.52	9926.66	6434.2	73
H28B	2297.24	8601.73	7127.23	73
H28C	573.65	9538.78	7097.24	73
H30	5330.16	4465.49	915.94	39
H31	4993.05	2509.45	1432.89	42
H33	1511.44	2944.51	3653.22	46

Table S28 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 220766lt_auto.

Atom	*x*	*y*	*z*	U(eq)
H34	1881.96	4896.52	3170.84	43
H35A	2283.38	1047.04	3534.36	69
H35B	4123.42	777.61	2959.33	69
H35C	2346.18	1148.35	2394.11	69

Experimental

Single crystals of $C_{29}H_{29}NO_4S$ **[220766lt_auto]** were **[]**. A suitable crystal was selected and **[]** on a **XtaLAB Synergy R, DW system, HyPix-Arc 150** diffractometer. The crystal was kept at 99.99(10) K during data collection. Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program using Intrinsic Phasing and refined with the SHELXL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.

Crystal structure determination of [220766lt_auto]

Crystal Data for $C_{29}H_{29}NO_4S$ (M =487.59 g/mol): triclinic, space group P-1 (no. 2), a = 7.73568(18) Å, b = 12.3470(3) Å, c = 14.4133(3) Å, α = 69.991(2)°, β = 83.0268(19)°, γ = 79.978(2)°, V = 1270.97(5) Å^3, Z = 2, T = 99.99(10) K, μ(Cu Kα) = 1.414 mm^{-1}, *Dcalc* = 1.274 g/cm^3, 13857 reflections measured (6.542° ≤ 2Θ ≤ 134.038°), 4513 unique (R_{int} = 0.0206, R_{sigma} = 0.0239) which were used in all calculations. The final R_1 was 0.0593 (I > 2σ(I)) and wR_2 was 0.1686 (all data).

Refinement model description

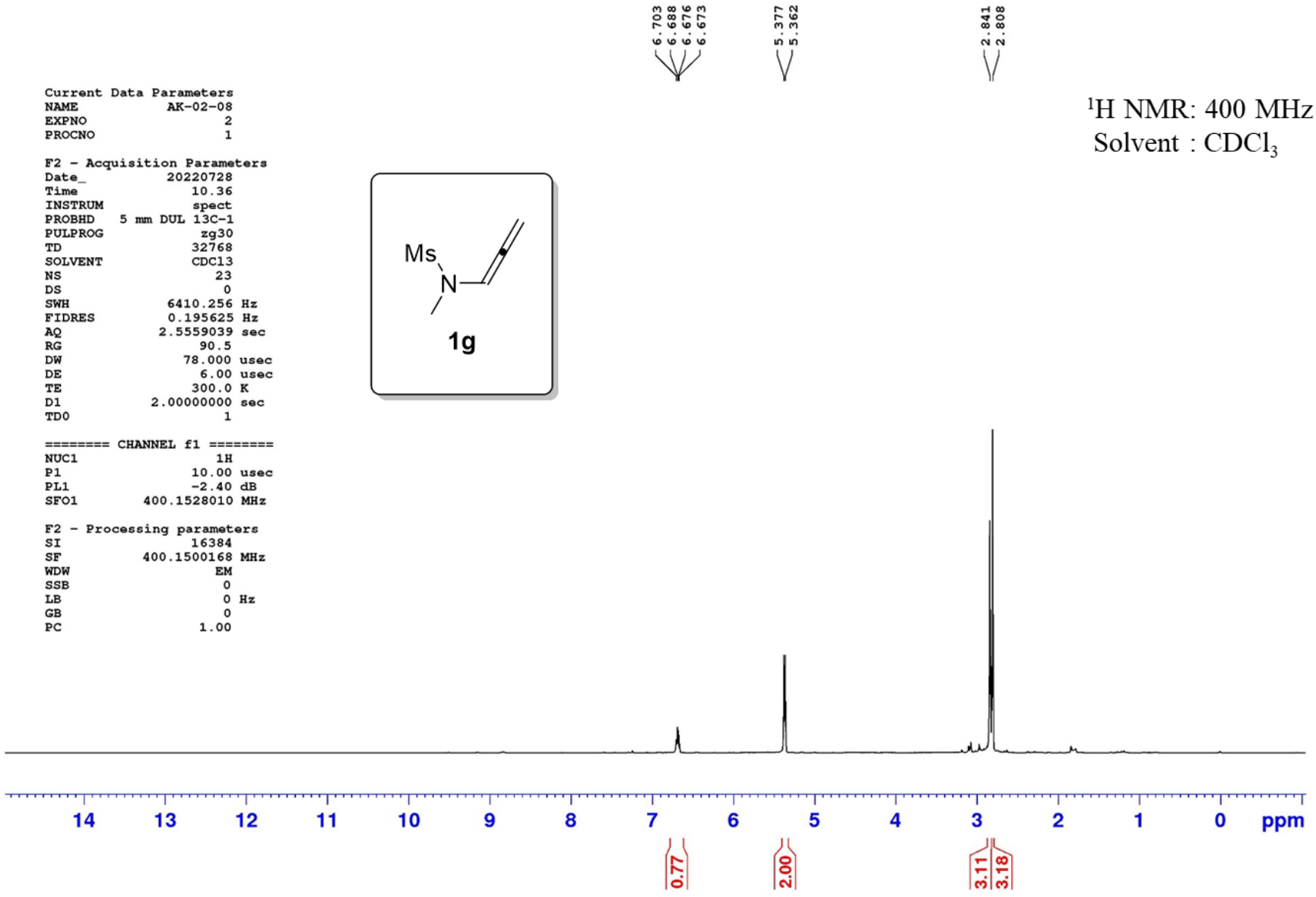
Number of restraints - 0, number of constraints - unknown.

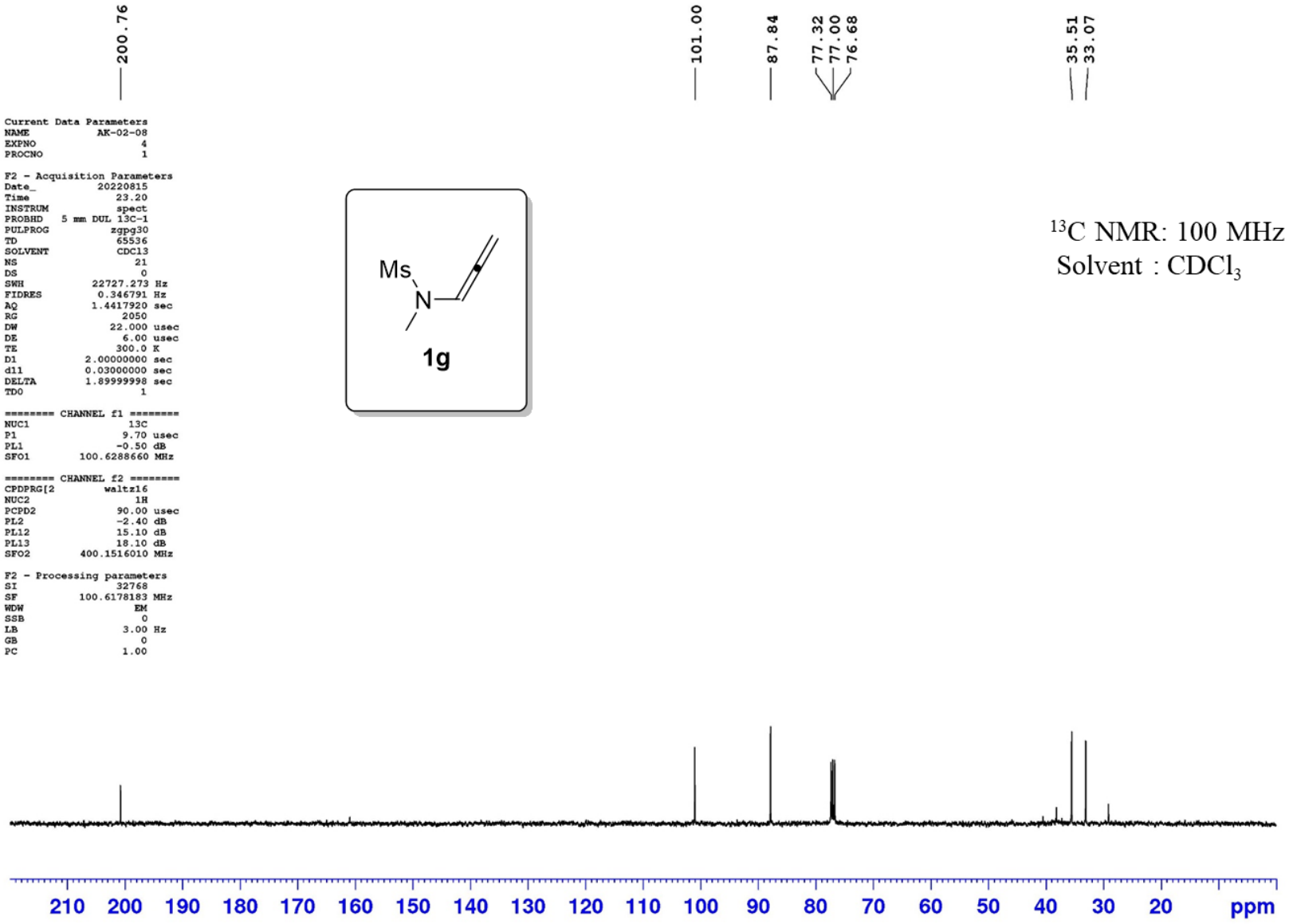
Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups, All C(H,H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Ternary CH refined with riding coordinates:
 C8(H8)
2.b Secondary CH2 refined with riding coordinates:
 C18(H18A,H18B), C19(H19A,H19B), C20(H20A,H20B)
2.c Aromatic/amide H refined with riding coordinates:
 C2(H2), C3(H3), C5(H5), C6(H6), C12(H12), C13(H13), C14(H14), C15(H15),
 C16(H16), C30(H30), C31(H31), C33(H33), C34(H34)
2.d Idealised Me refined as rotating group:
 C21(H21A,H21B,H21C), C28(H28A,H28B,H28C), C35(H35A,H35B,H35C)
```

.

8. ^{1}H, ^{13}C spectra and NOE of key compounds:





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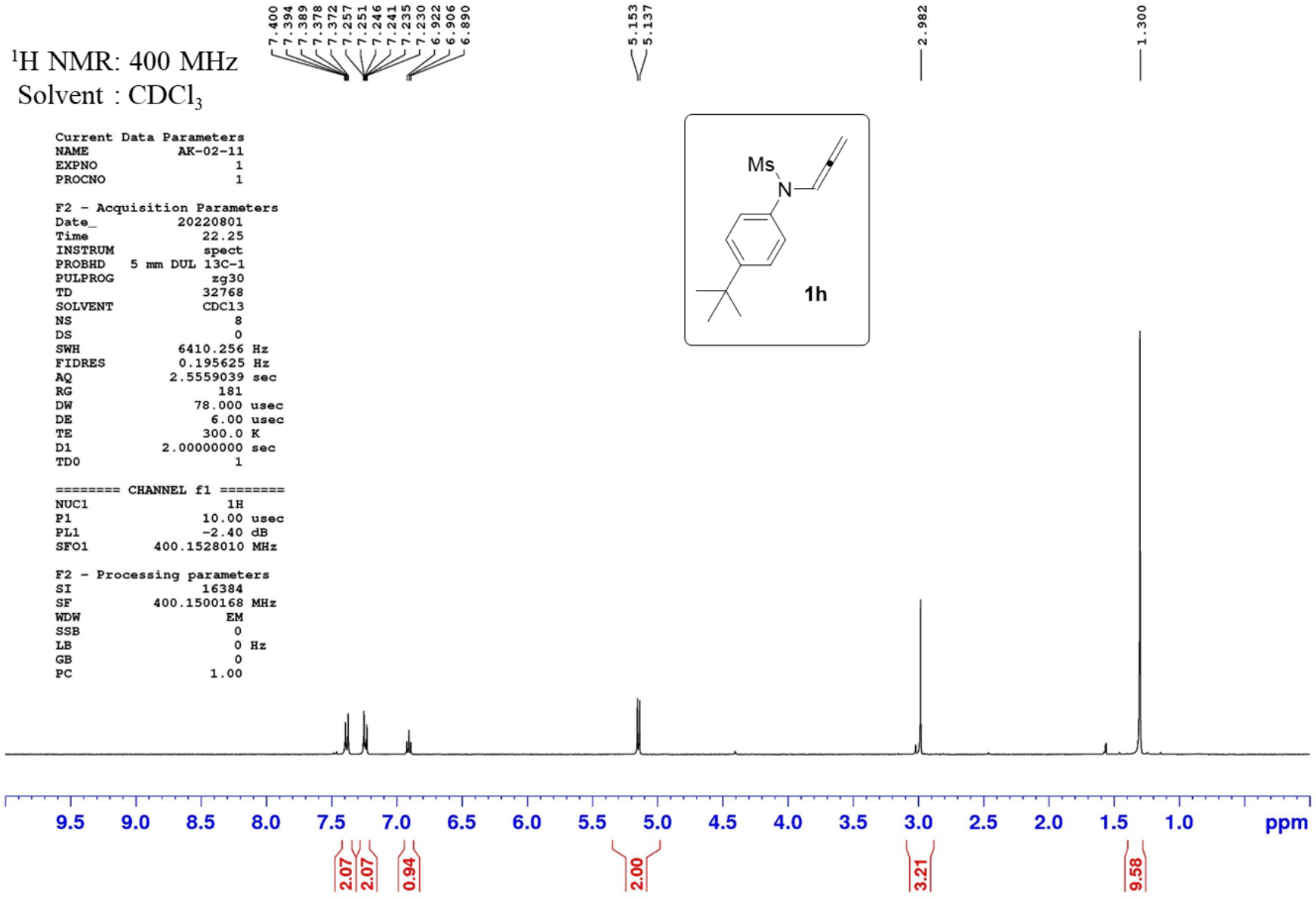
Solvent : $CDCl_3$

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

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PROCNO                 1

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TD                 32768
SOLVENT            CDC13
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DS                     0
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FIDRES          0.195625 Hz
AQ             2.5559039 sec
RG                   181
DW                78.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
TD0                    1

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P1                10.00 usec
PL1               -2.40 dB
SFO1         400.1528010 MHz

F2 - Processing parameters
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WDW                   EM
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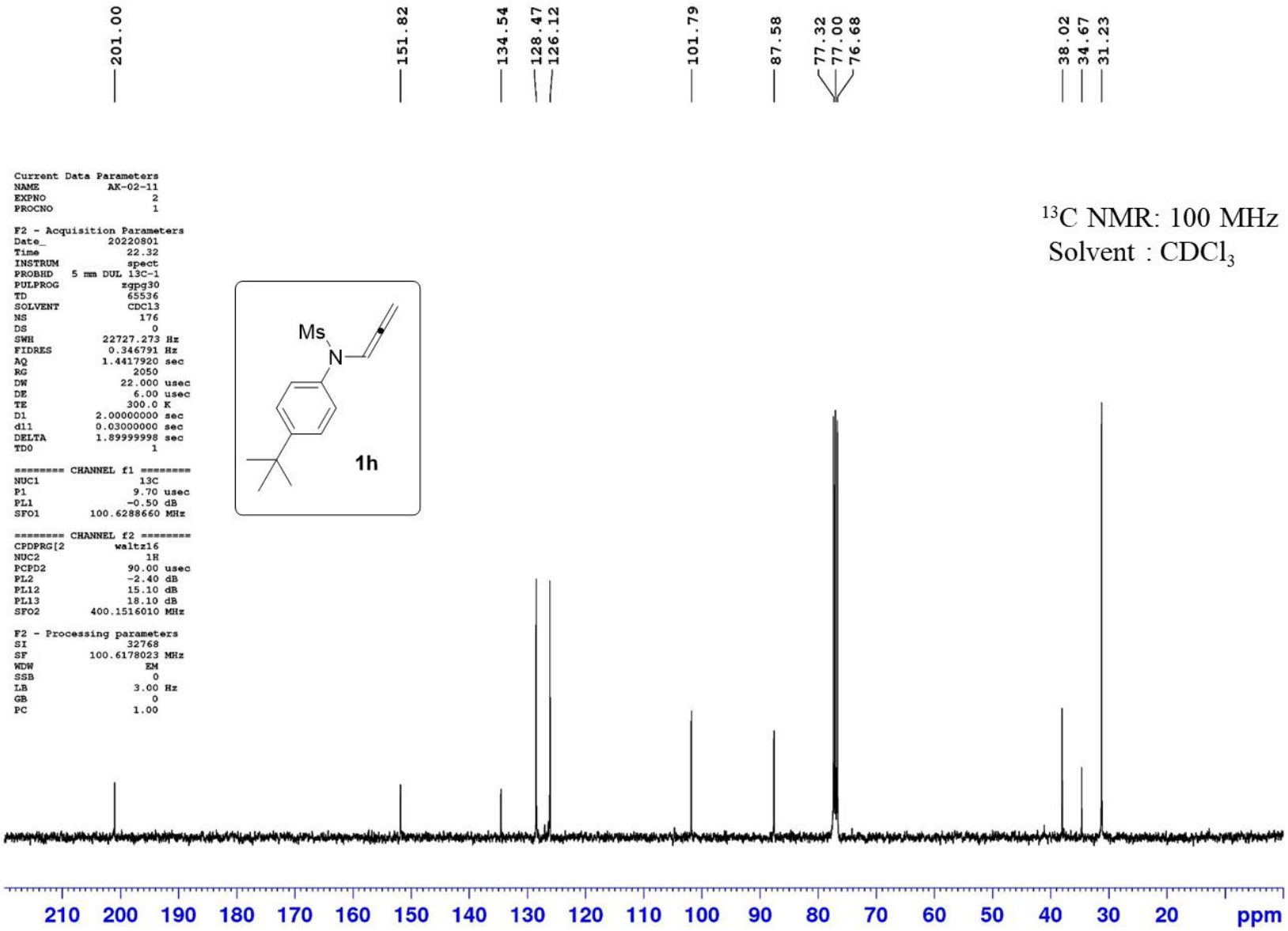
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77.32
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76.68
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34.67
31.23
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PROCNO 1
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Time 22.32
INSTRUM spect
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 176
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
F2 - Processing parameters
SI 32768
SF 100.6178023 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
Ms
N
1h
13C NMR: 100 MHz
Solvent : CDCl3
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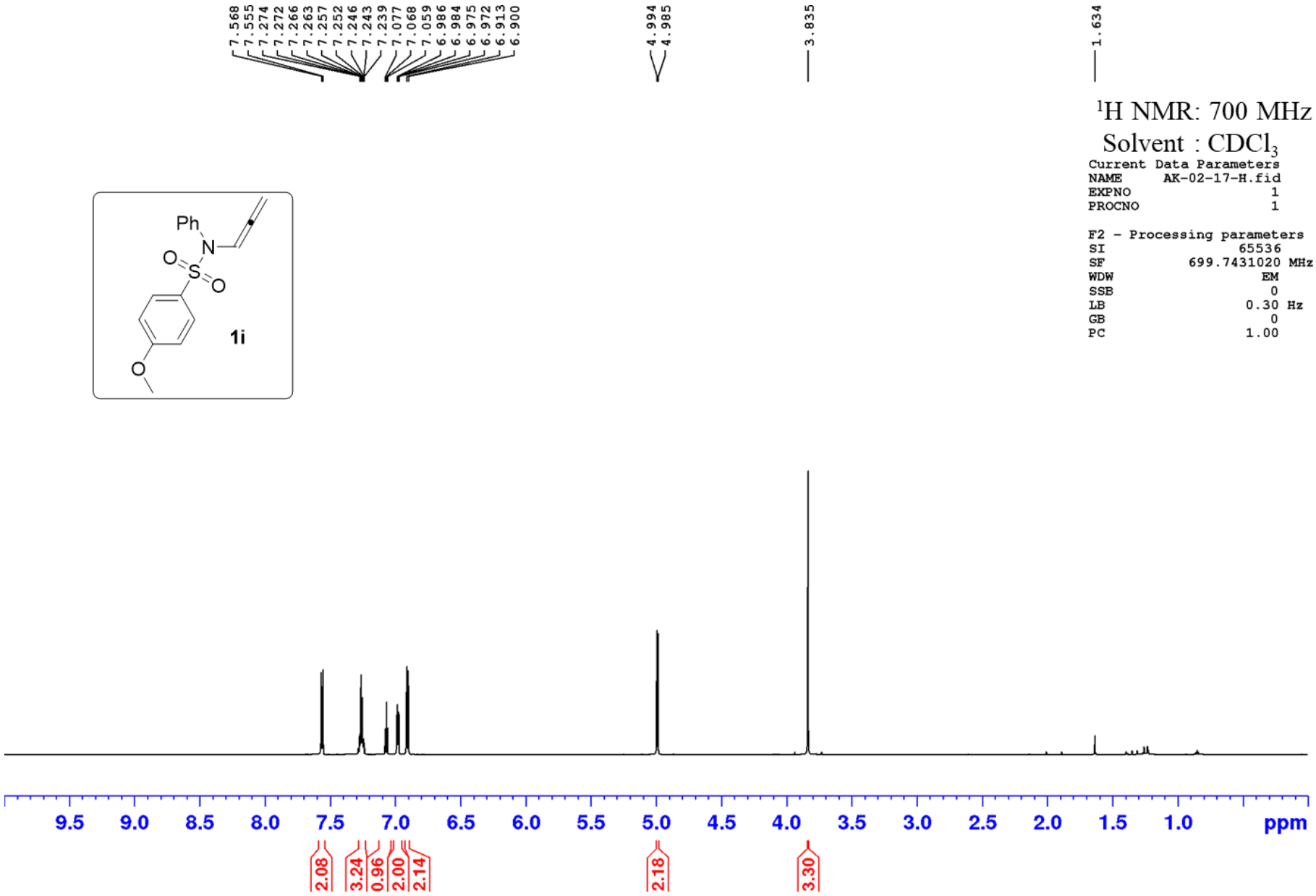
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7.274
7.272
7.266
7.263
7.257
7.252
7.246
7.243
7.239
7.077
7.068
7.059
6.986
6.984
6.975
6.972
6.913
6.900
4.994
4.985
3.835
1.634
1H NMR: 700 MHz
Solvent : CDCl3
Current Data Parameters
NAME AK-02-17-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7431020 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
N
O
S
O
O
1i
9.5
9.0
8.5
8.0
7.5
7.0
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5.0
4.5
4.0
3.5
3.0
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2.0
1.5
1.0
ppm
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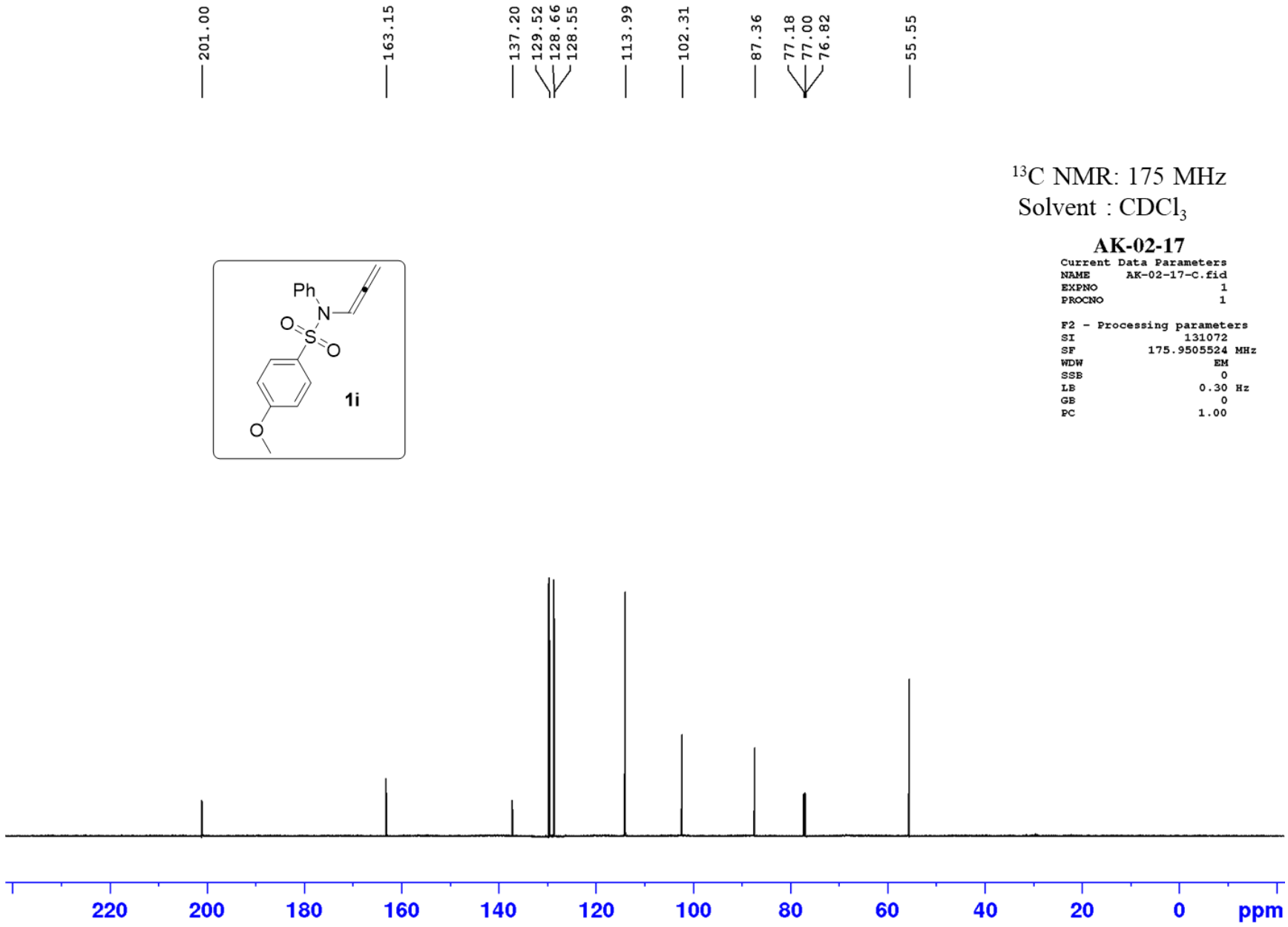
3.24
0.96
2.00
2.14
2.18
3.30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128.55
113.99
102.31
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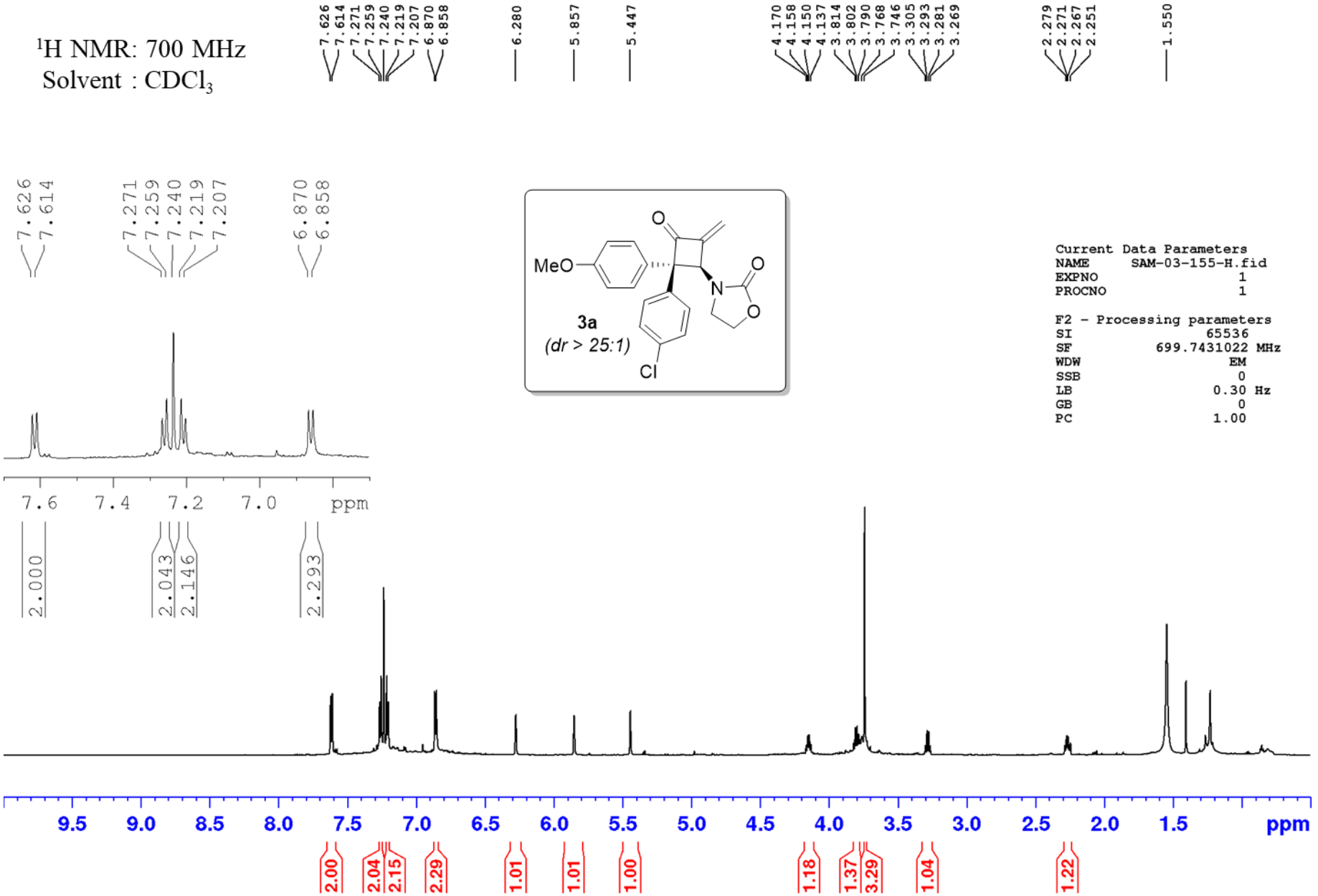
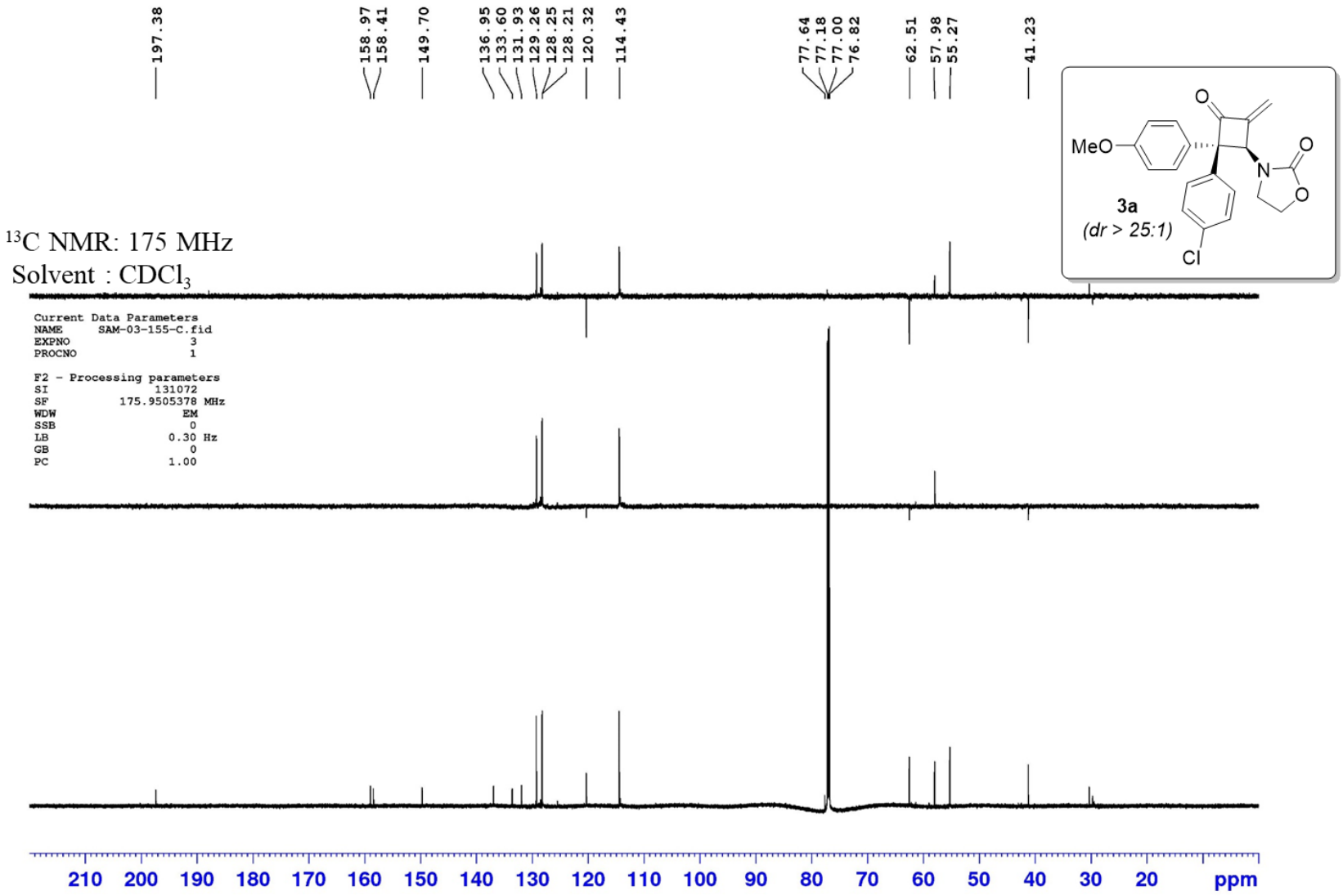
77.18
77.00
76.82
55.55
^{13}C NMR: 175 MHz
Solvent : $CDCl_3$
AK-02-17
Current Data Parameters
NAME AK-02-17-C.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505524 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
N
O
S
O
O
1i
220
200
180
160
140
120
100
80
60
40
20
0
ppm

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

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136.95
133.60
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128.21
120.32
114.43
77.64
77.18
77.00
76.82
62.51
57.98
55.27
41.23
MeO
O
N
O
O
Cl
3a
(dr > 25:1)
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
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PROCNO 1
F2 - Processing parameters
SI 131072
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WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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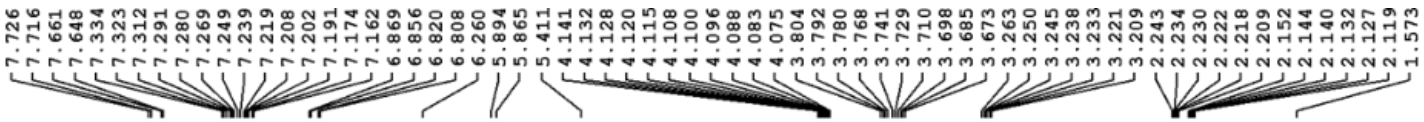
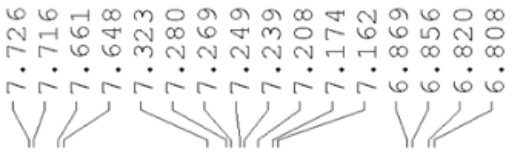
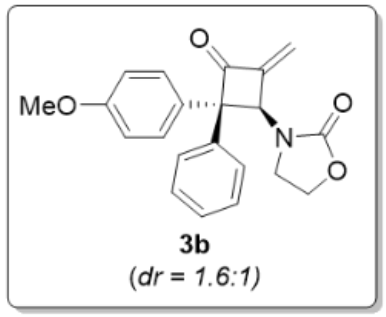


^{1}H NMR: 700 MHz
Solvent : $CDCl_3$





3b
(*dr = 1.6:1*)

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PROCNO                   1

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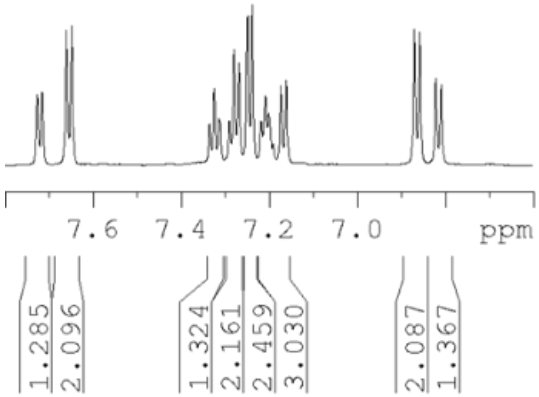

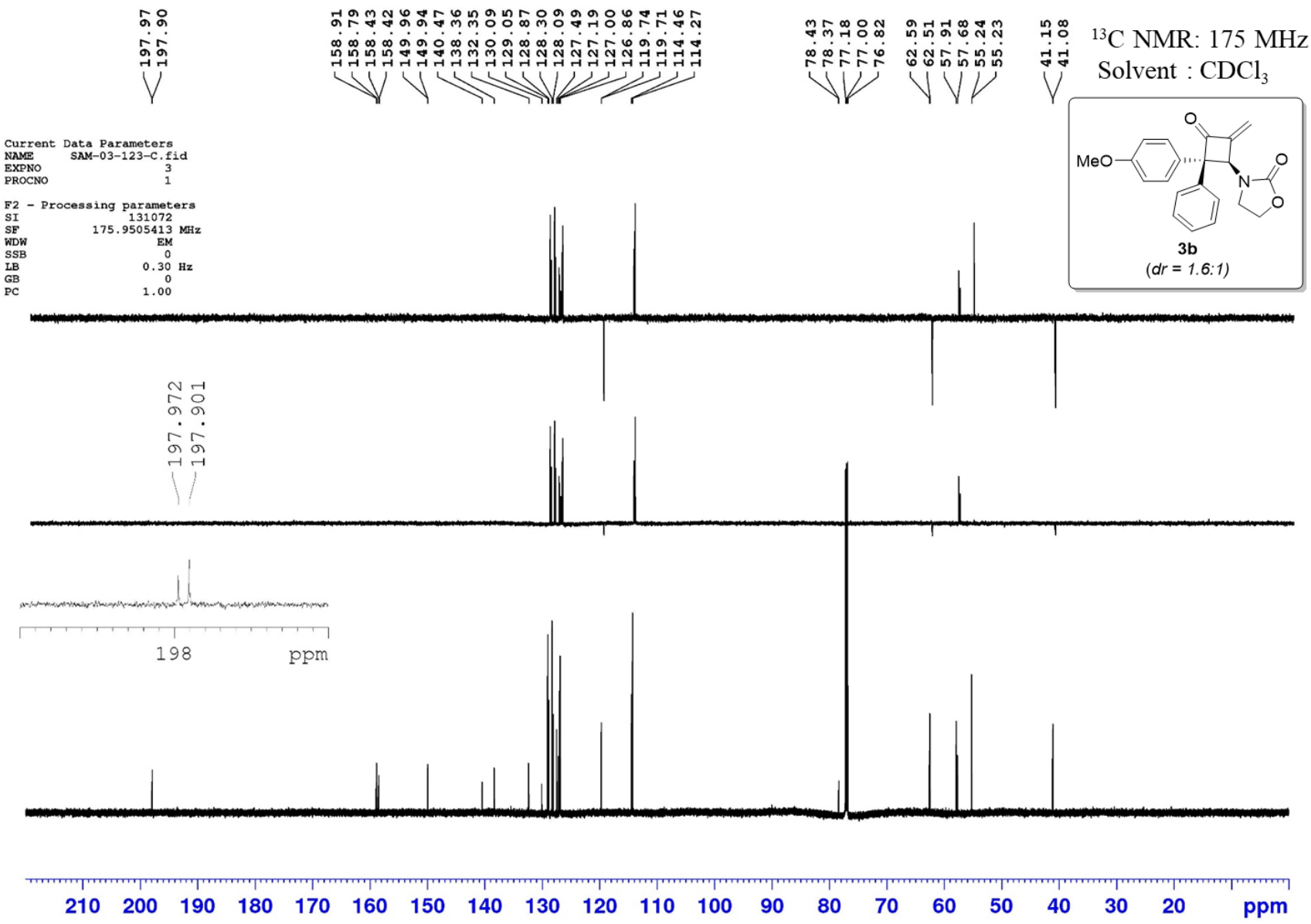
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119.71
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114.27
78.43
78.37
77.18
77.00
76.82
62.59
62.51
57.91
57.68
55.24
55.23
41.15
41.08
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
NAME SAM-03-123-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
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LB 0.30 Hz
GB 0
PC 1.00
MeO
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O
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ppm
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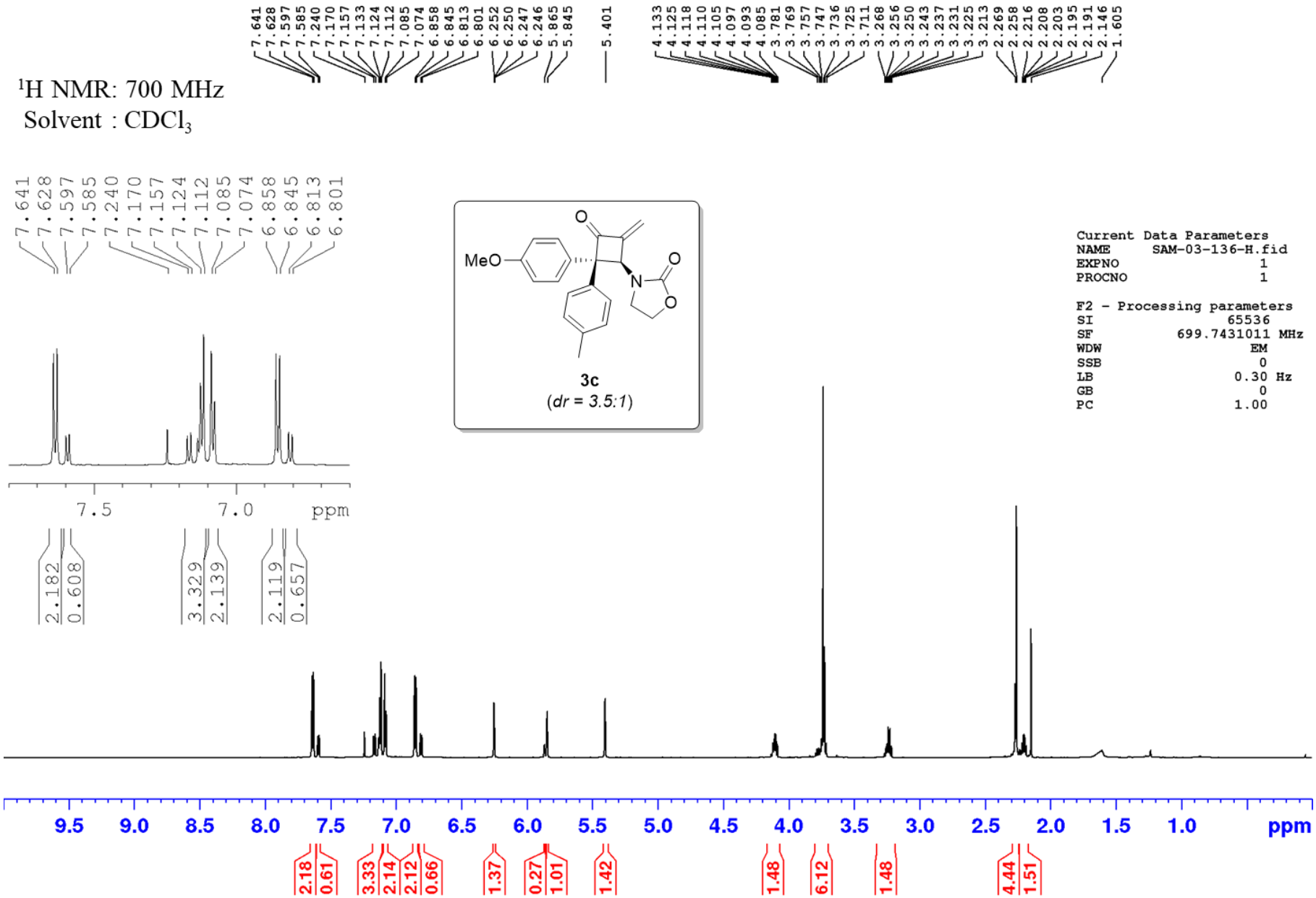
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4.093
4.085
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3.769
3.757
3.747
3.736
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3.250
3.243
3.237
3.231
3.225
3.213
2.269
2.258
2.216
2.208
2.203
2.195
2.191
2.146
1.605
1H NMR: 700 MHz
Solvent : CDCl3
MeO
O
N
O
O
3c
(dr = 3.5:1)
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PROCNO 1
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GB 0
PC 1.00
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ppm
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2.119
0.657
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
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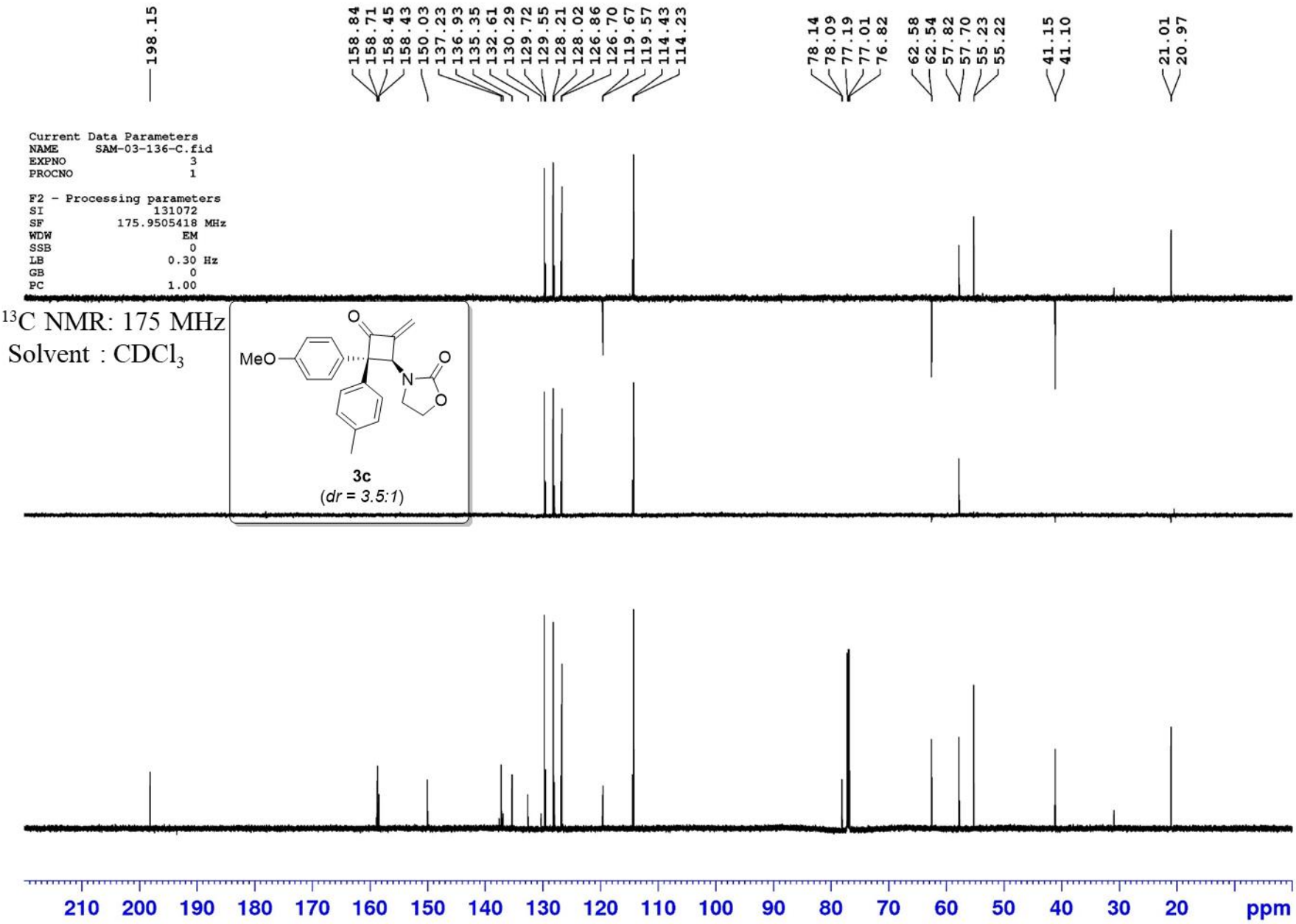
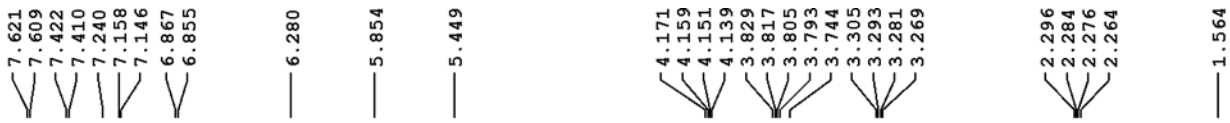
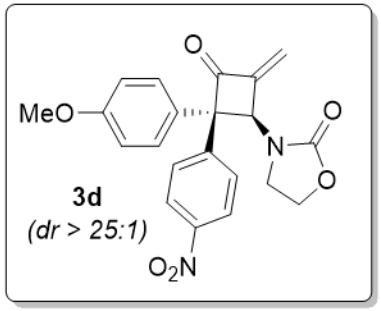
3.33
2.14
2.12
0.66
1.37
0.27
1.01
1.42
1.48
6.12
1.48
4.44
1.51

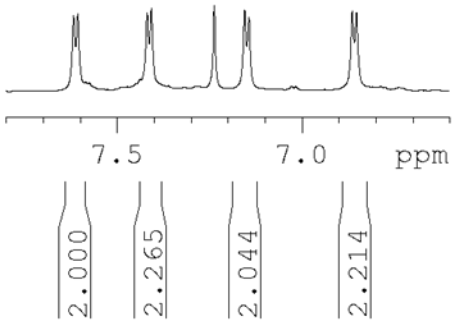

198.15
158.84
158.71
158.45
158.43
150.03
137.23
136.93
135.35
132.61
130.29
129.72
129.55
128.21
128.02
126.86
126.70
119.67
119.57
114.43
114.23
78.14
78.09
77.19
77.01
76.82
62.58
62.54
57.82
57.70
55.23
55.22
41.15
41.10
21.01
20.97
Current Data Parameters
NAME SAM-03-136-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505418 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13C NMR: 175 MHz
Solvent : CDCl3
MeO
O
N
O
O
3c
(dr = 3.5:1)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

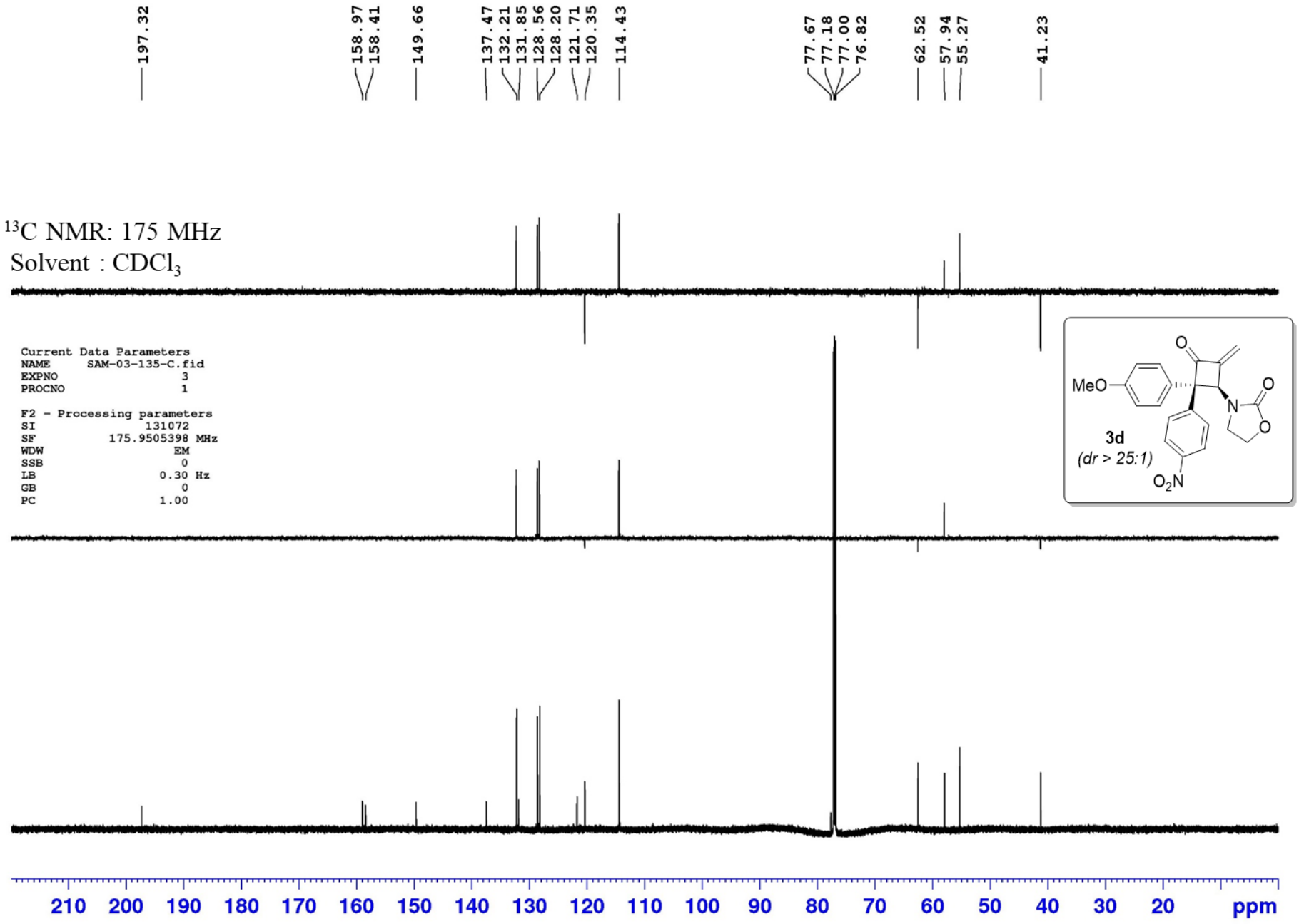
^{1}H NMR: 700 MHz
Solvent : $CDCl_3$

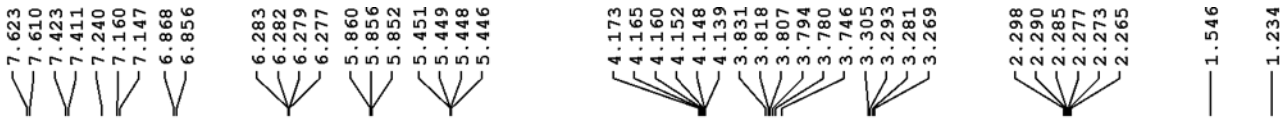





```
Current Data Parameters
NAME      SAM-03-135-H.fid
EXPNO                    1
PROCNO                   1

F2 - Processing parameters
SI                   65536
SF          699.7431058 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

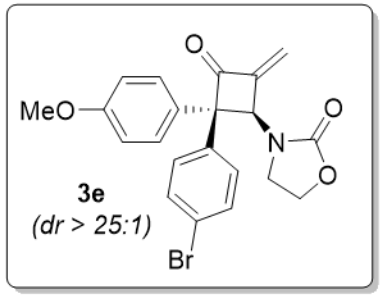



197.32
158.97
158.41
149.66
137.47
132.21
131.85
128.56
128.20
121.71
120.35
114.43
77.67
77.18
77.00
76.82
62.52
57.94
55.27
41.23
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
NAME SAM-03-135-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505398 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
O
N
O
O
O2N
3d
(dr > 25:1)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

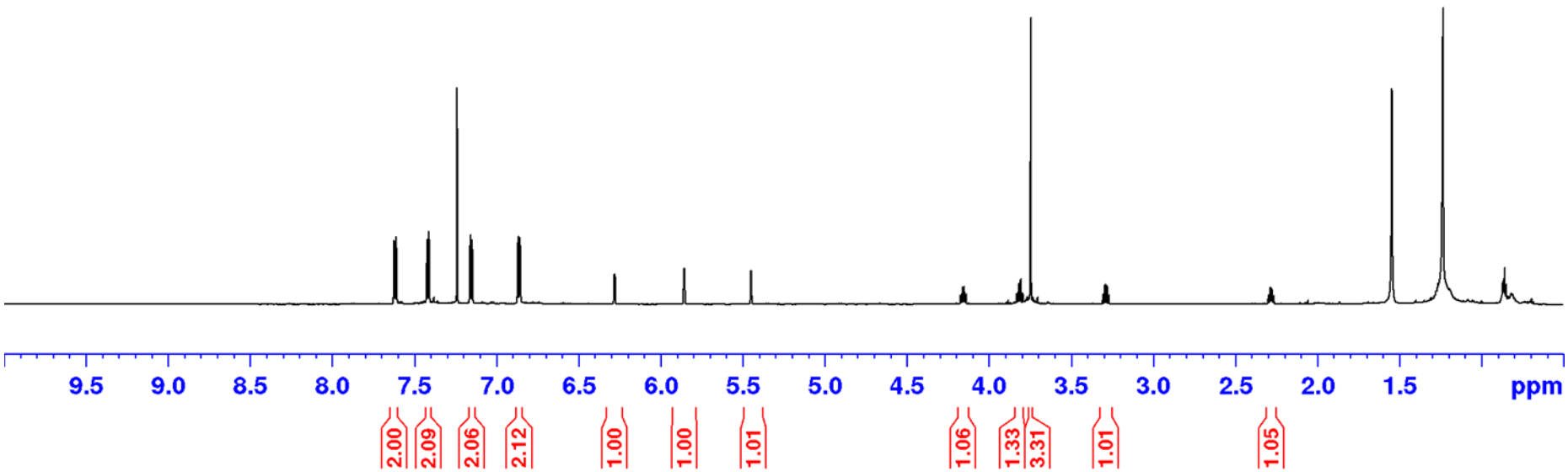


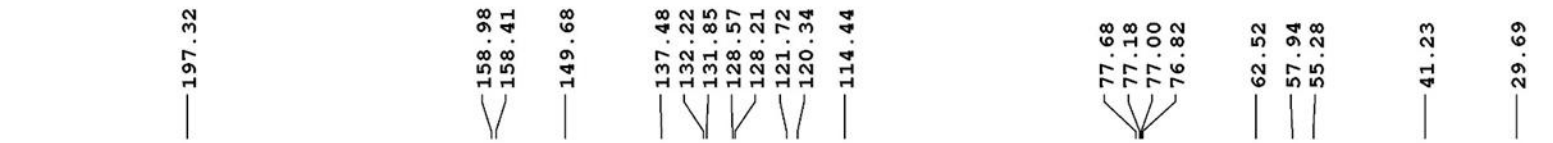
^{1}H NMR: 700 MHz
Solvent : $CDCl_3$

```
Current Data Parameters
NAME      SAM-03-141-H.fid
EXPNO                    1
PROCNO                   1

F2 - Processing parameters
SI                   65536
SF          699.7431002 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



3e
(dr > 25:1)

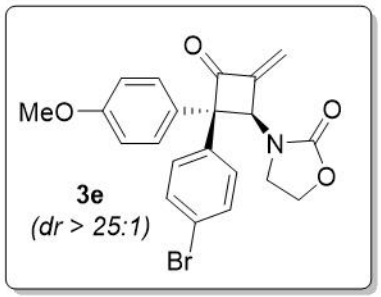




^{13}C NMR: 175 MHz

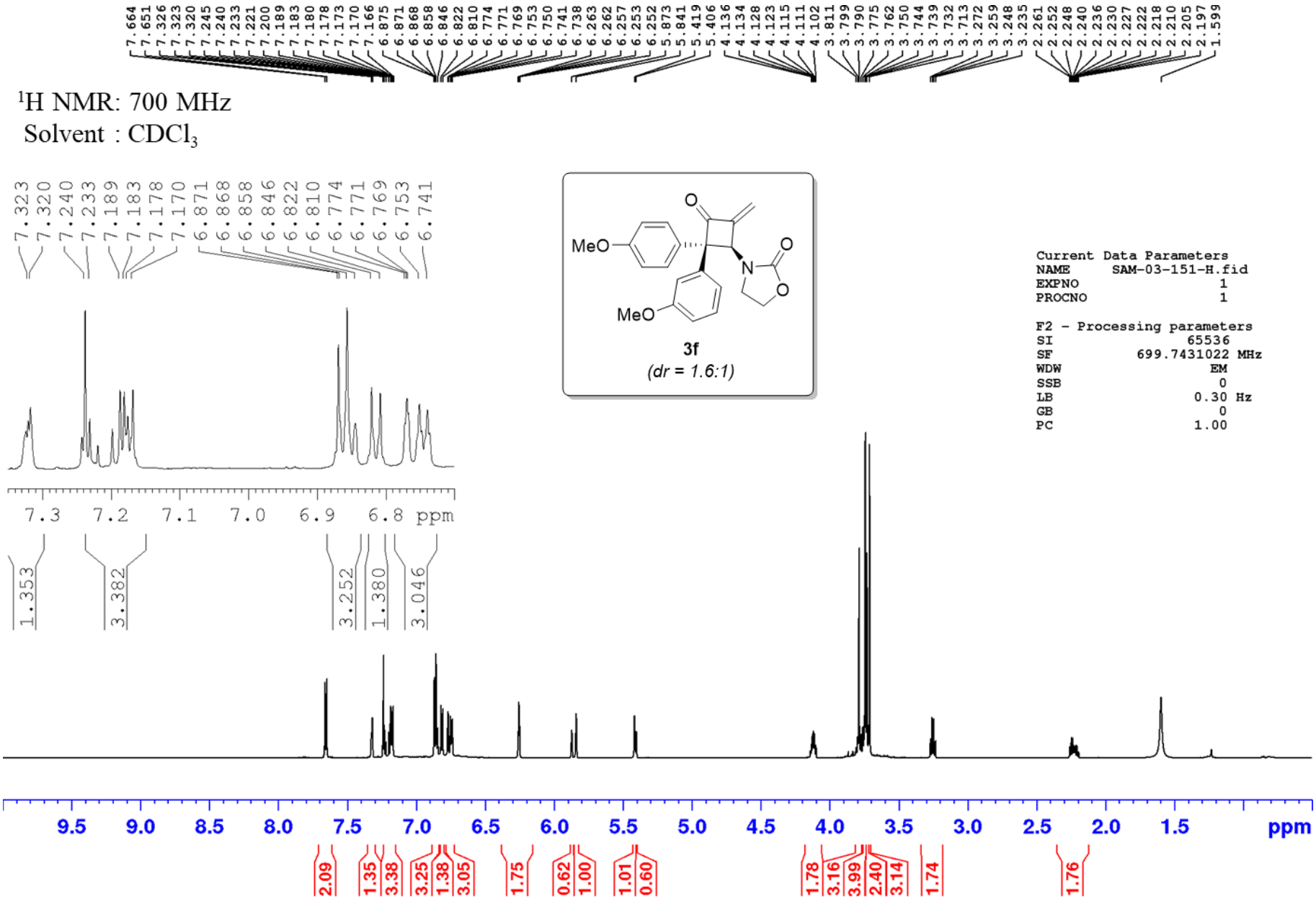
Solvent : $CDCl_3$

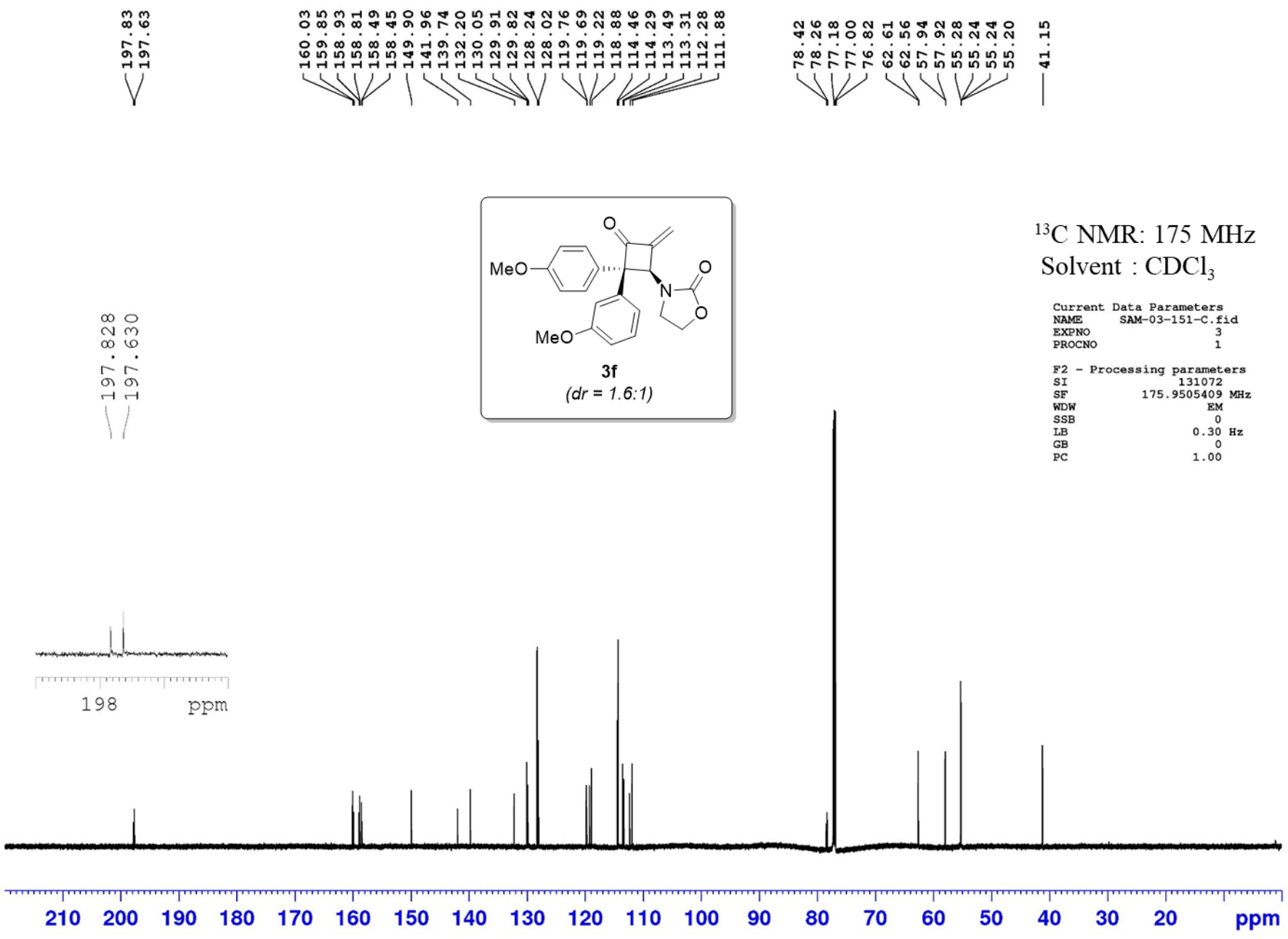
```
Current Data Parameters
NAME      SAM-03-141-C.fid
EXPNO                    3
PROCNO                   1

F2 - Processing parameters
SI                  131072
SF          175.9505382 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



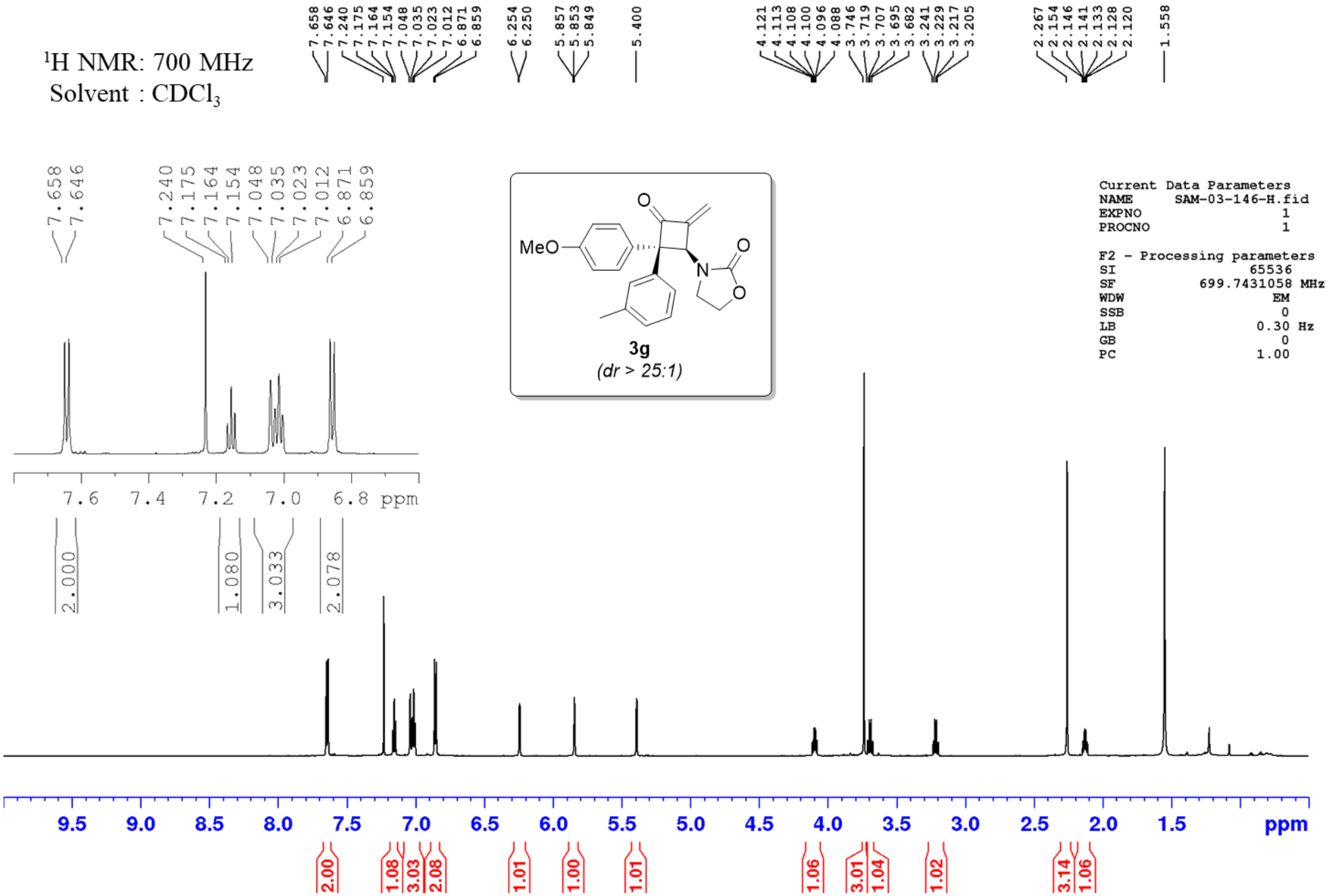
3e

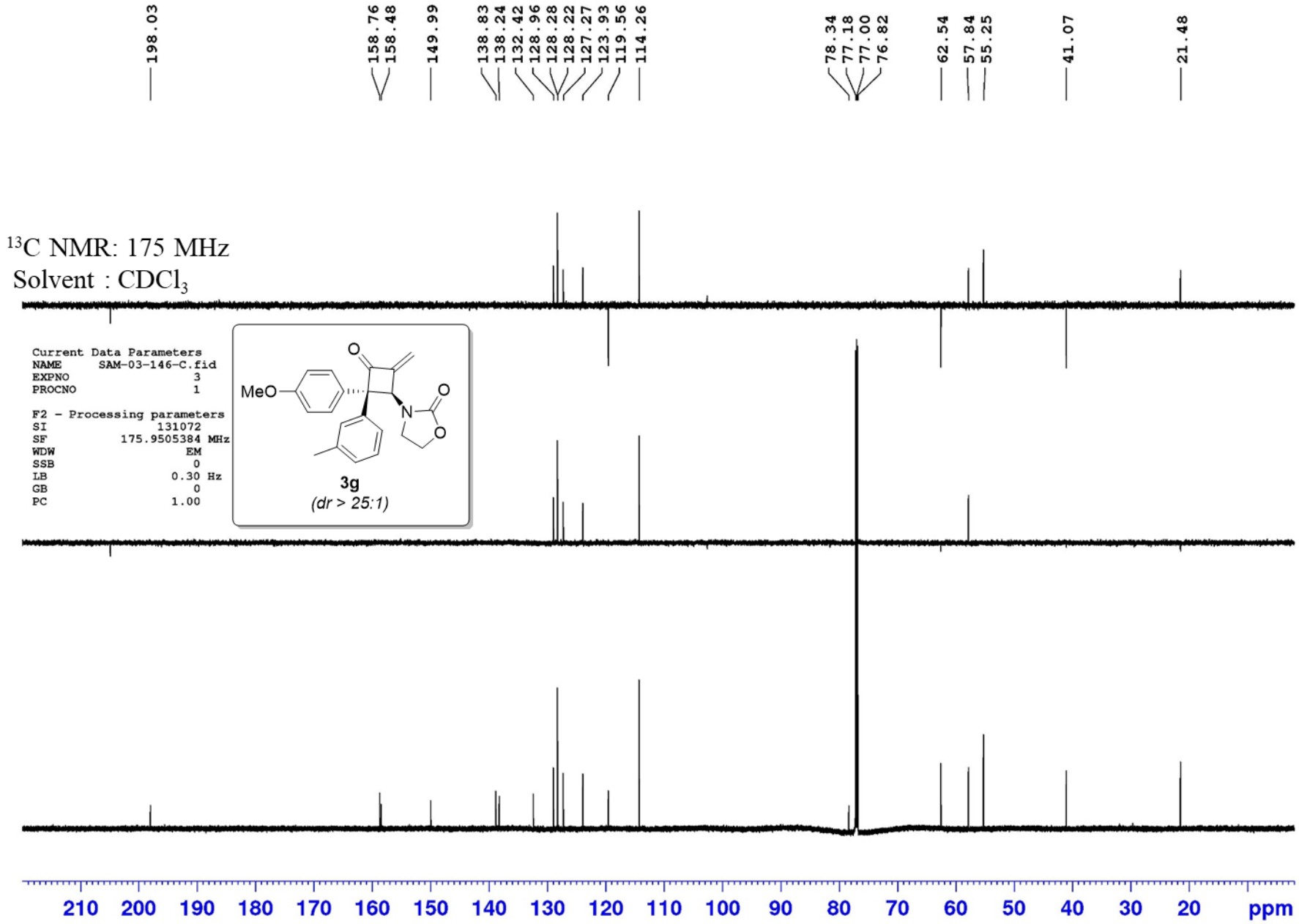
(dr > 25:1)


7.664 7.651 7.326 7.323 7.320 7.245 7.240 7.233 7.221 7.200 7.189 7.183 7.180 7.178 7.173 7.170 7.166 6.875 6.871 6.868 6.858 6.846 6.822 6.810 6.774 6.771 6.769 6.753 6.750 6.741 6.738 6.263 6.262 6.257 6.253 6.252 5.873 5.841 5.419 5.406 4.136 4.134 4.128 4.123 4.115 4.111 4.102 3.811 3.799 3.790 3.775 3.762 3.750 3.744 3.739 3.732 3.713 3.272 3.259 3.248 3.235 2.261 2.252 2.248 2.240 2.236 2.230 2.227 2.222 2.218 2.210 2.205 2.197 1.599
1H NMR: 700 MHz
Solvent : CDCl3
7.323 7.320 7.240 7.233 7.189 7.183 7.178 7.170 6.871 6.868 6.858 6.846 6.822 6.810 6.774 6.771 6.769 6.753 6.741
MeO
MeO
O
O
N
O
3f
(dr = 1.6:1)
Current Data Parameters
NAME SAM-03-151-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
7.3 7.2 7.1 7.0 6.9 6.8 ppm
1.353 3.382 3.252 1.380 3.046
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm
2.09 1.35 3.38 3.25 1.38 3.05 1.75 0.62 1.00 1.01 0.60 1.78 3.16 3.99 2.40 3.14 1.74 1.76

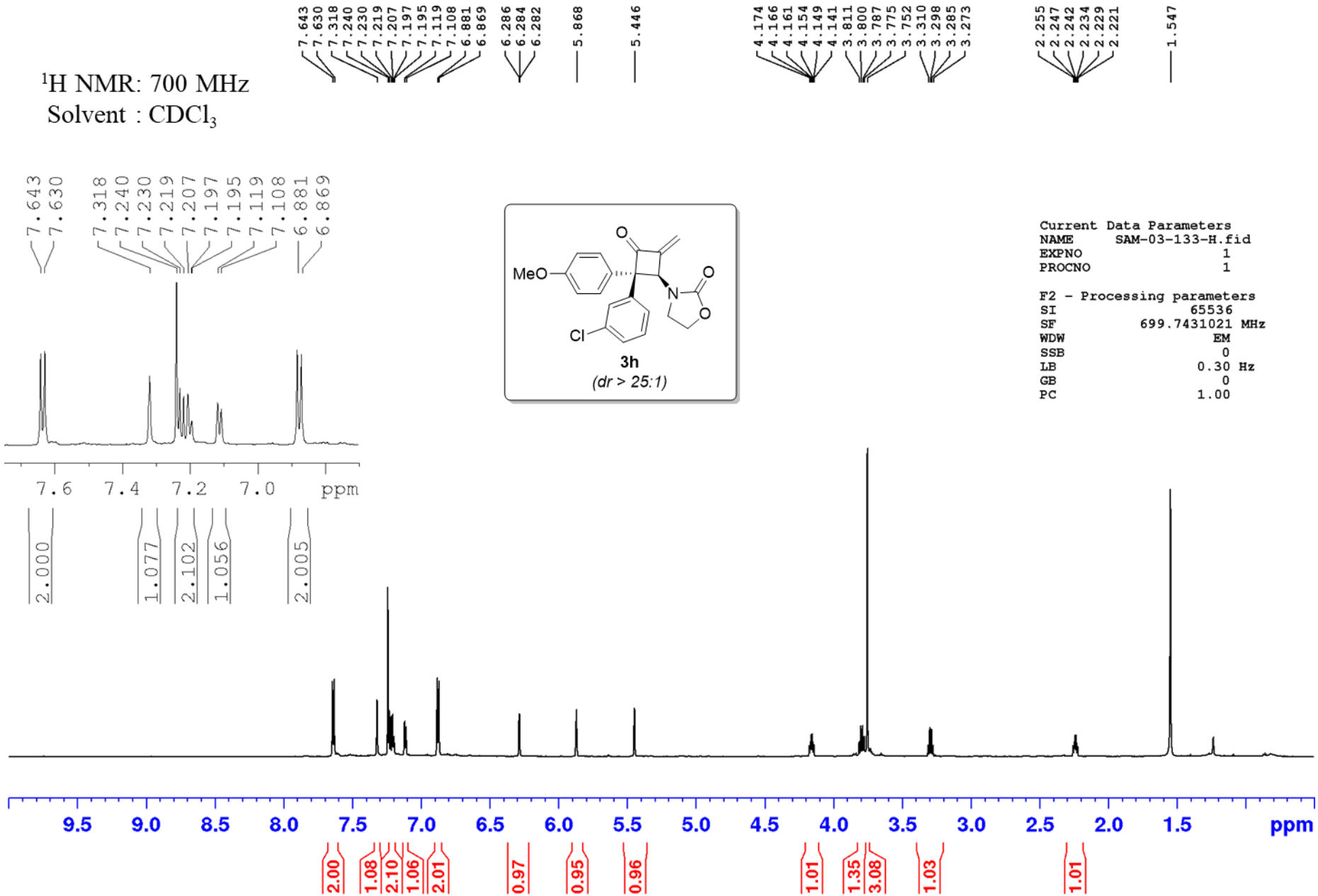

197.83
197.63
160.03
159.85
158.93
158.81
158.49
158.45
149.90
141.96
139.74
132.20
130.05
129.91
129.82
128.24
128.02
119.76
119.69
119.22
118.88
114.46
114.29
113.49
113.31
112.28
111.88
78.42
78.26
77.18
77.00
76.82
62.61
62.56
57.94
57.92
55.28
55.24
55.24
55.20
41.15
MeO
MeO
O
O
N
O
3f
(dr = 1.6:1)
197.828
197.630
198
ppm
^{13}C NMR: 175 MHz
Solvent : $CDCl_3$
Current Data Parameters
NAME SAM-03-151-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505409 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

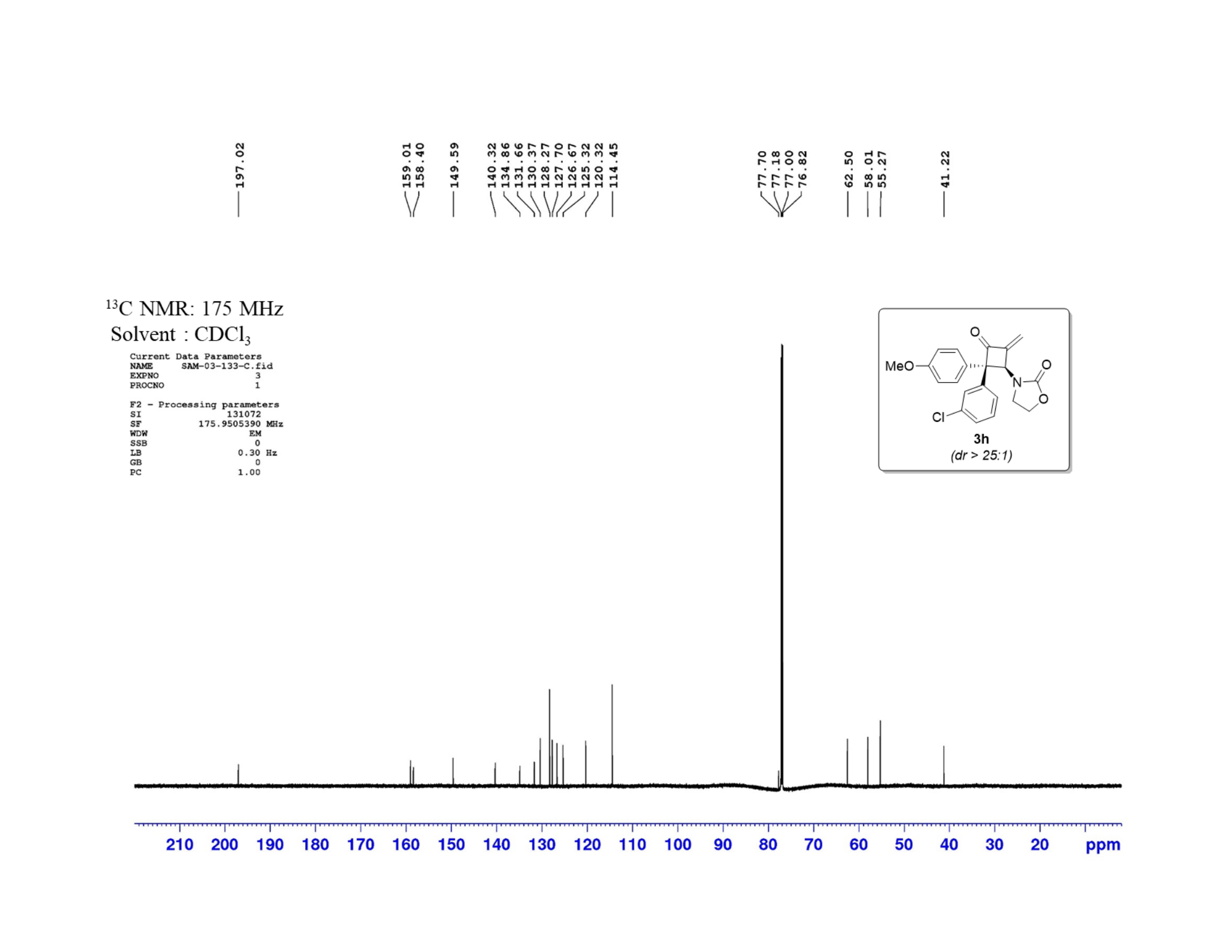
^{1}H NMR: 700 MHz
Solvent : $CDCl_3$



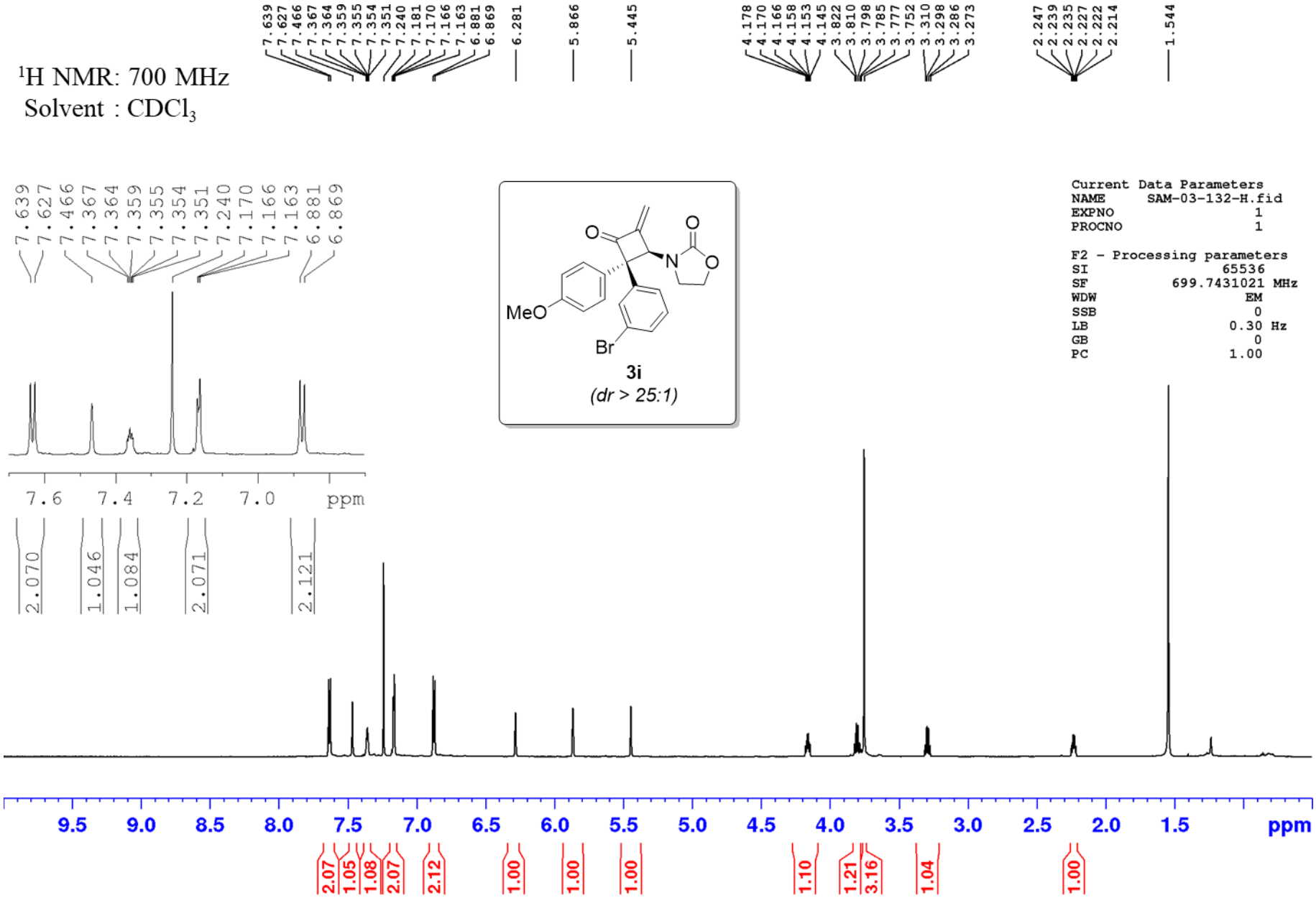

198.03
158.76
158.48
149.99
138.83
138.24
132.42
128.96
128.28
128.22
127.27
123.93
119.56
114.26
78.34
77.18
77.00
76.82
62.54
57.84
55.25
41.07
21.48
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
NAME SAM-03-146-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505384 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
O
N
O
O
3g
(dr > 25:1)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

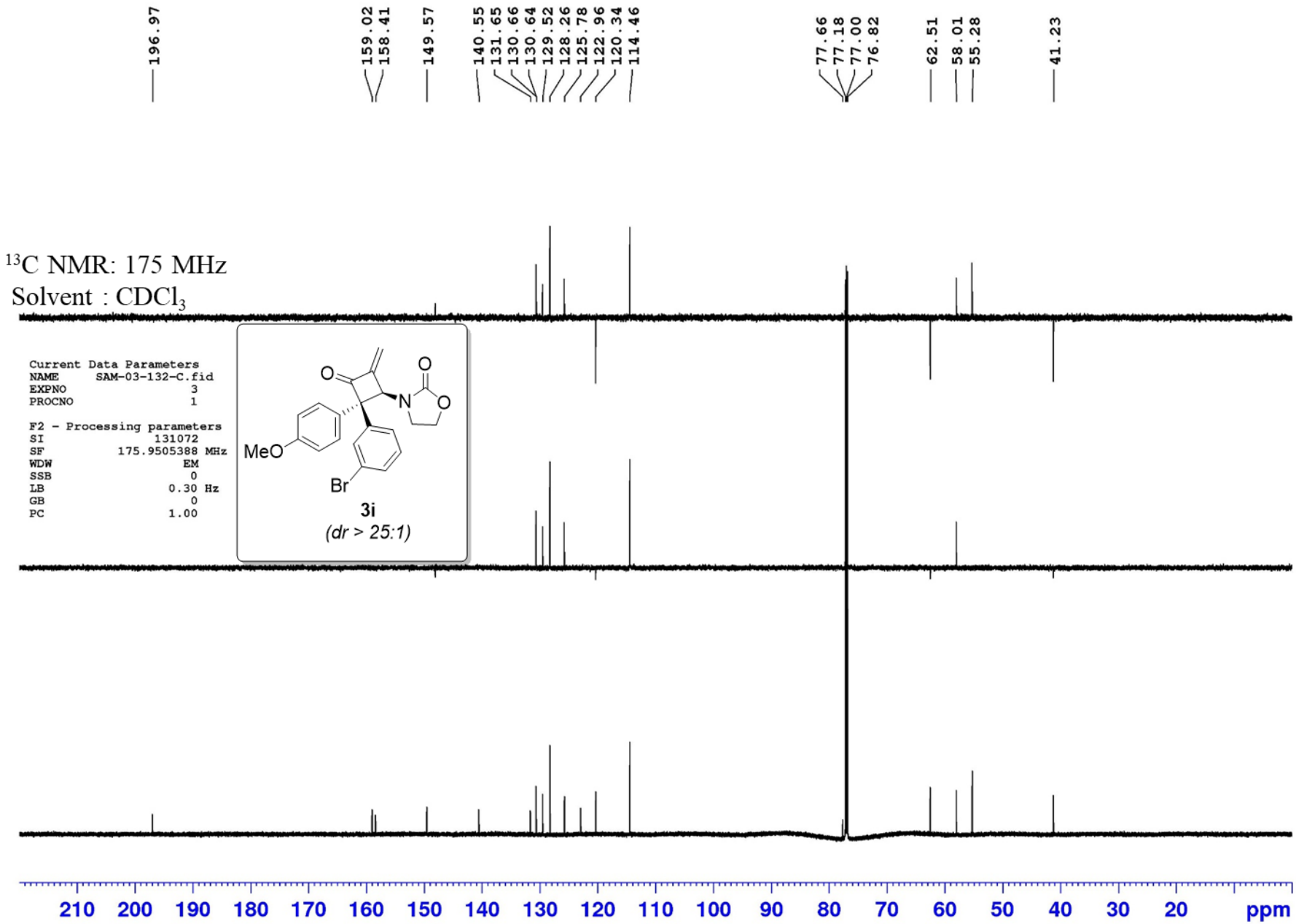
^{1}H NMR: 700 MHz
Solvent : $CDCl_3$

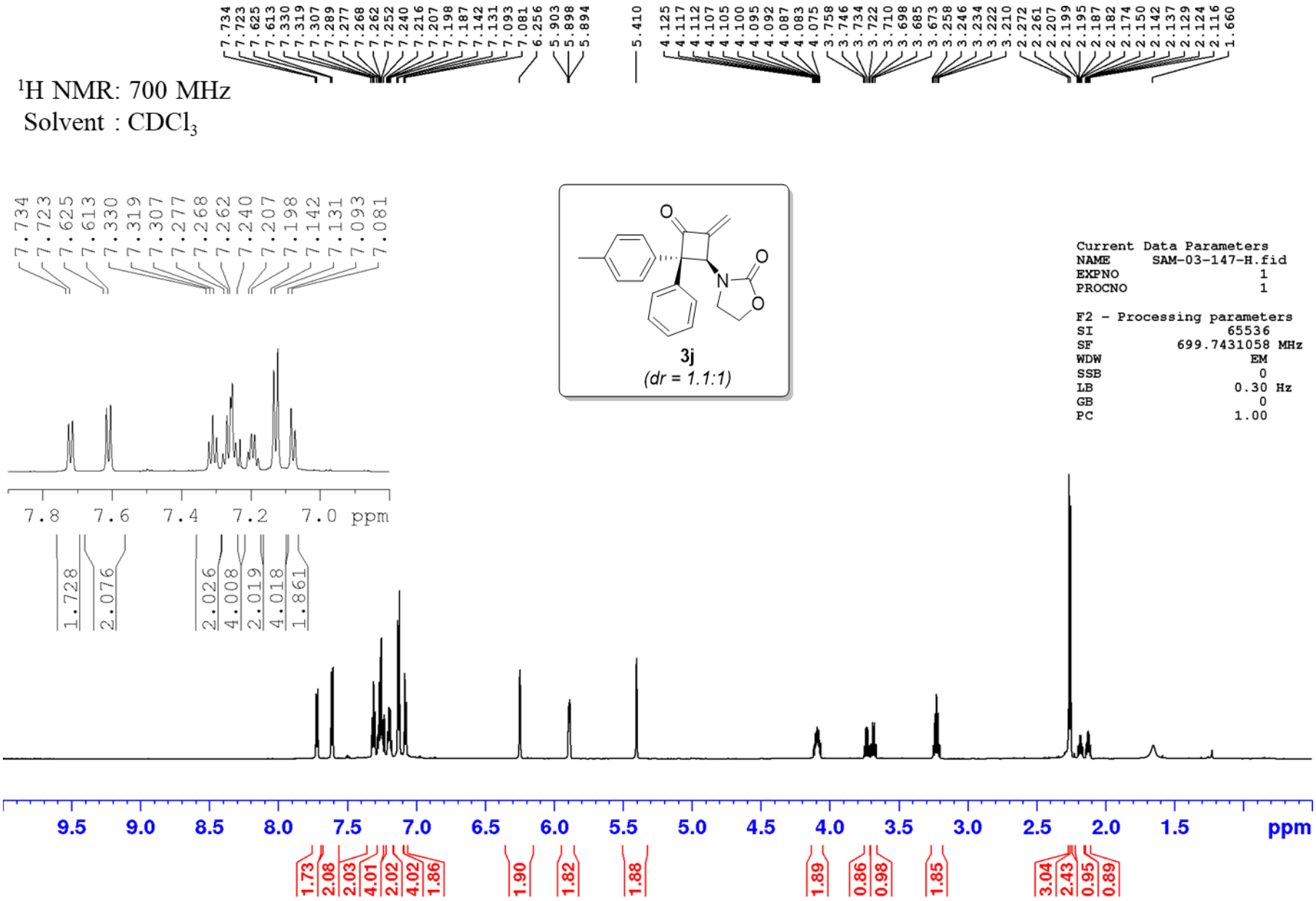


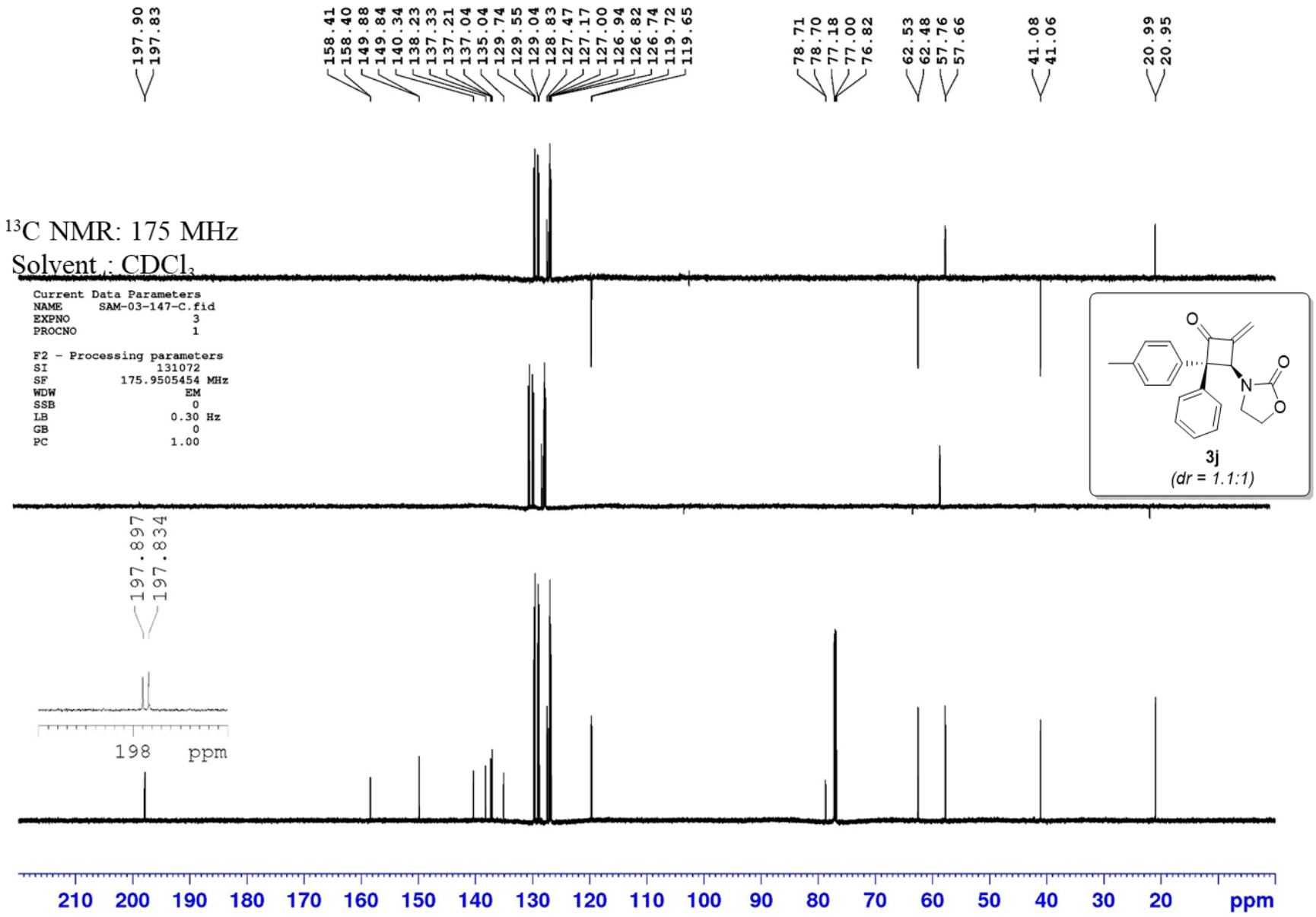

197.02
159.01
158.40
149.59
140.32
134.86
131.66
130.37
128.27
127.70
126.67
125.32
120.32
114.45
77.70
77.18
77.00
76.82
62.50
58.01
55.27
41.22
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
NAME SAM-03-133-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505390 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
Cl
O
N
O
O
3h
(dr > 25:1)
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 : $CDCl_3$

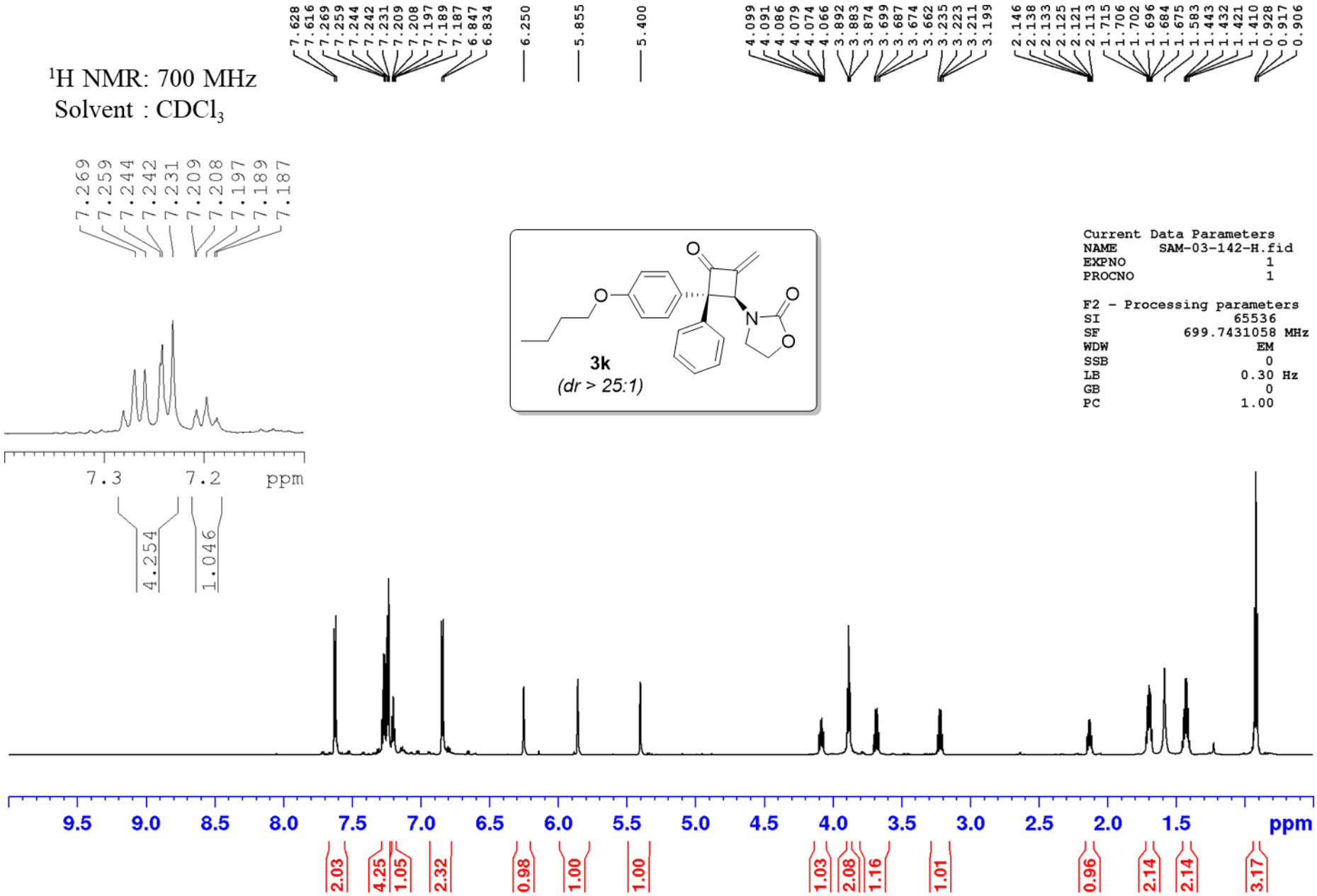


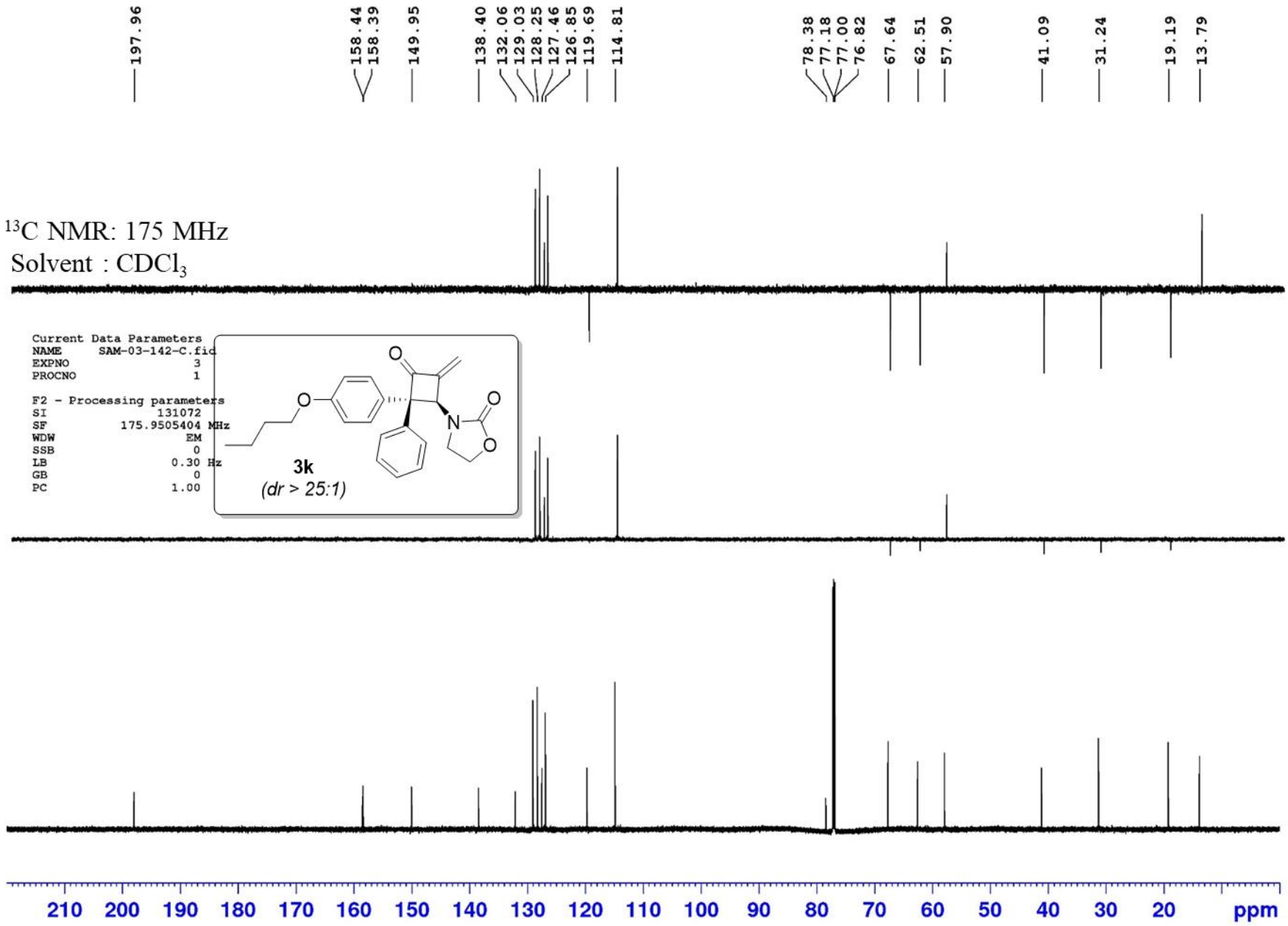

196.97
159.02
158.41
149.57
140.55
131.65
130.66
130.64
129.52
128.26
125.78
122.96
120.34
114.46
77.66
77.18
77.00
76.82
62.51
58.01
55.28
41.23
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
NAME SAM-03-132-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505388 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
Br
3i
(dr > 25:1)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.734
7.723
7.625
7.613
7.330
7.319
7.307
7.289
7.277
7.268
7.262
7.252
7.240
7.216
7.207
7.198
7.187
7.142
7.131
7.093
7.081
6.256
5.903
5.898
5.894
5.410
4.125
4.117
4.112
4.107
4.105
4.100
4.095
4.092
4.087
4.083
4.075
3.758
3.746
3.734
3.722
3.710
3.698
3.685
3.673
3.258
3.246
3.234
3.222
3.210
2.272
2.261
2.207
2.199
2.195
2.187
2.182
2.174
2.150
2.142
2.137
2.129
2.124
2.116
1.660
1H NMR: 700 MHz
Solvent : CDCl3
3j
(dr = 1.1:1)
Current Data Parameters
NAME SAM-03-147-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7431058 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
1.73
2.08
2.03
4.01
2.02
4.02
1.86
1.90
1.82
1.88
1.89
0.86
0.98
1.85
3.04
2.43
0.95
0.89

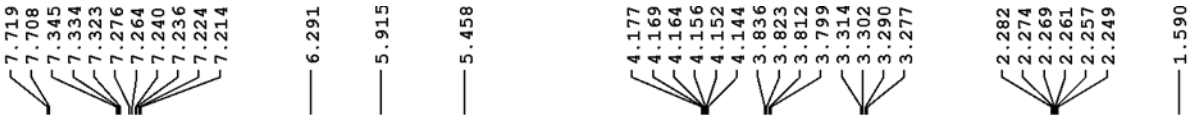

197.90
197.83
158.41
158.40
149.88
149.84
140.34
138.23
137.33
137.21
137.04
135.04
129.74
129.55
129.04
128.83
127.47
127.17
127.00
126.94
126.82
126.74
119.72
119.65
78.71
78.70
77.18
77.00
76.82
62.53
62.48
57.76
57.66
41.08
41.06
20.99
20.95
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
NAME SAM-03-147-C.fid
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
3j
(dr = 1.1:1)
197.897
197.834
198 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

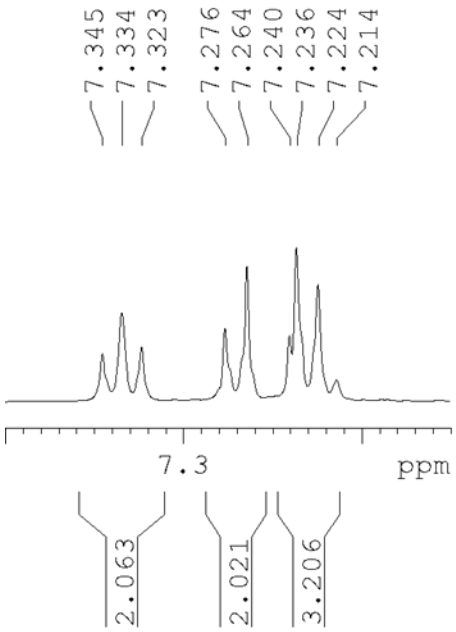
^{1}H NMR: 700 MHz
Solvent : $CDCl_3$

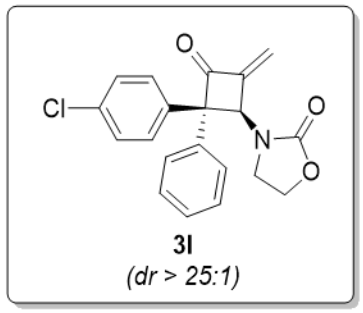



197.96
158.44
158.39
149.95
138.40
132.06
129.03
128.25
127.46
126.85
119.69
114.81
78.38
77.18
77.00
76.82
67.64
62.51
57.90
41.09
31.24
19.19
13.79
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
NAME SAM-03-142-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505404 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3k
(dr > 25:1)
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 : $CDCl_3$

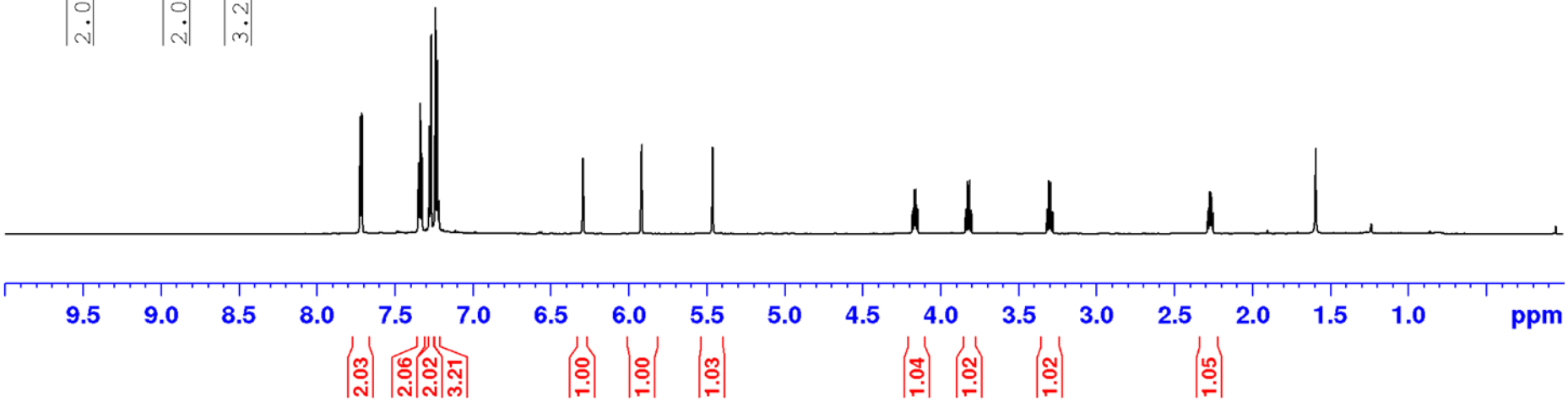


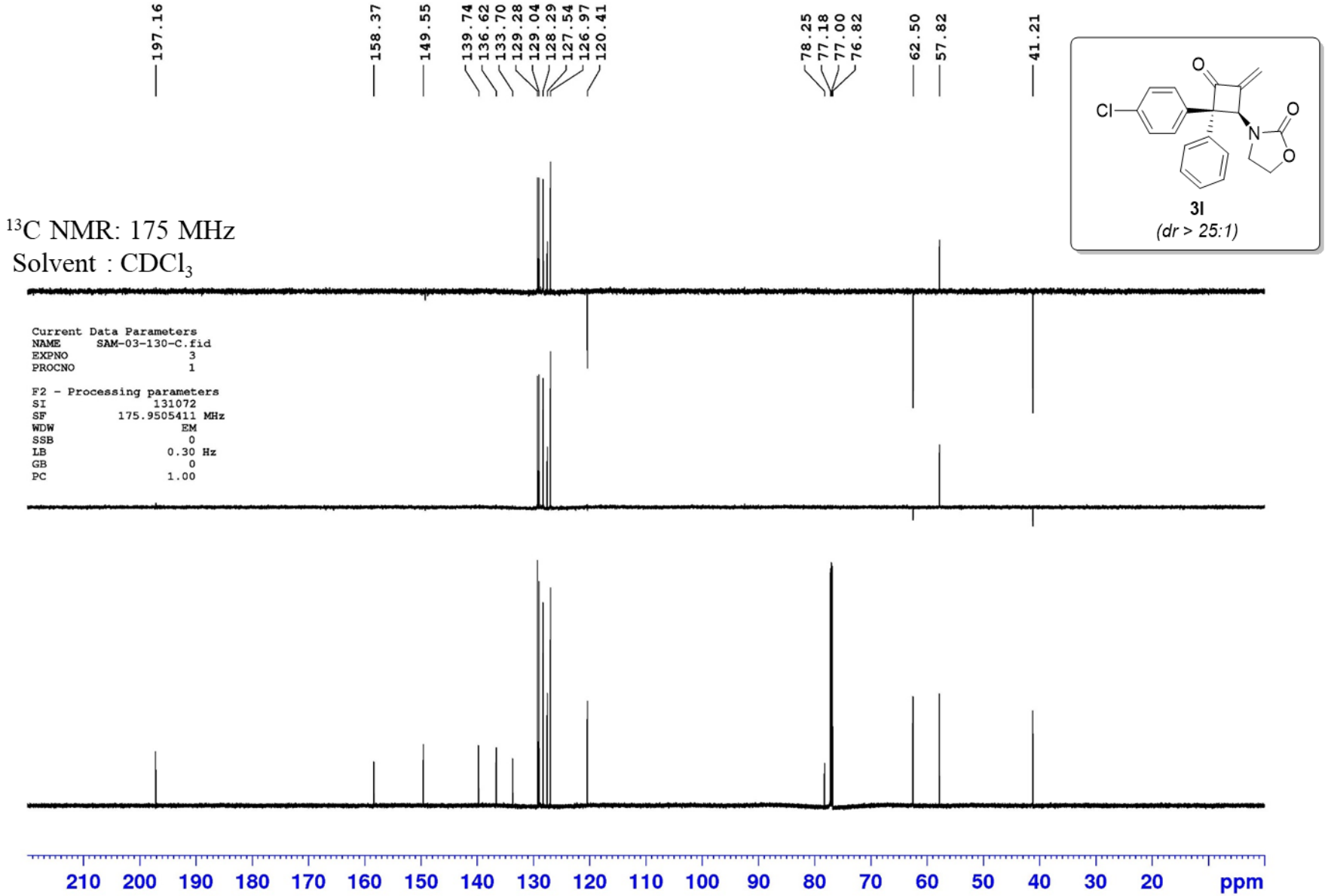




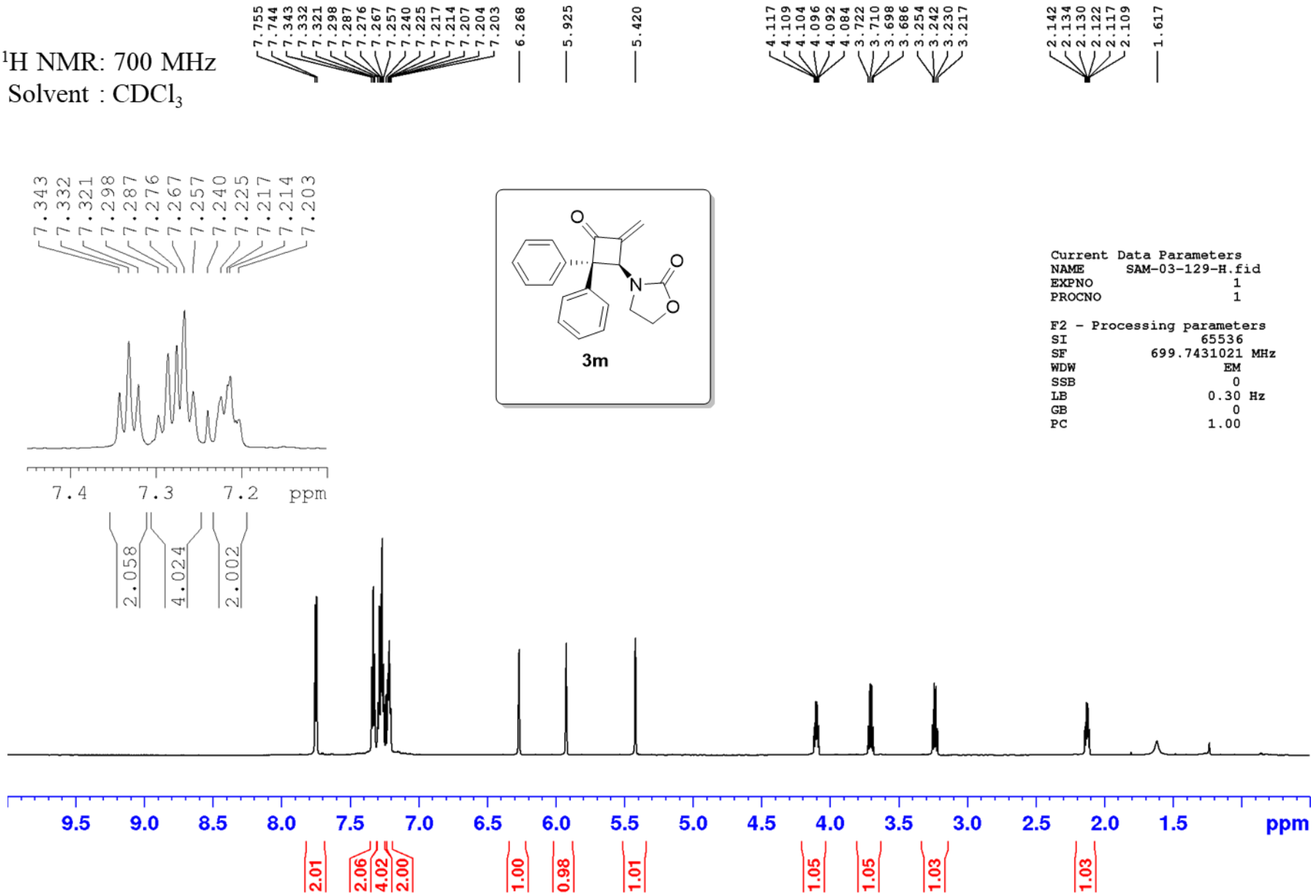
3l
(dr > 25:1)

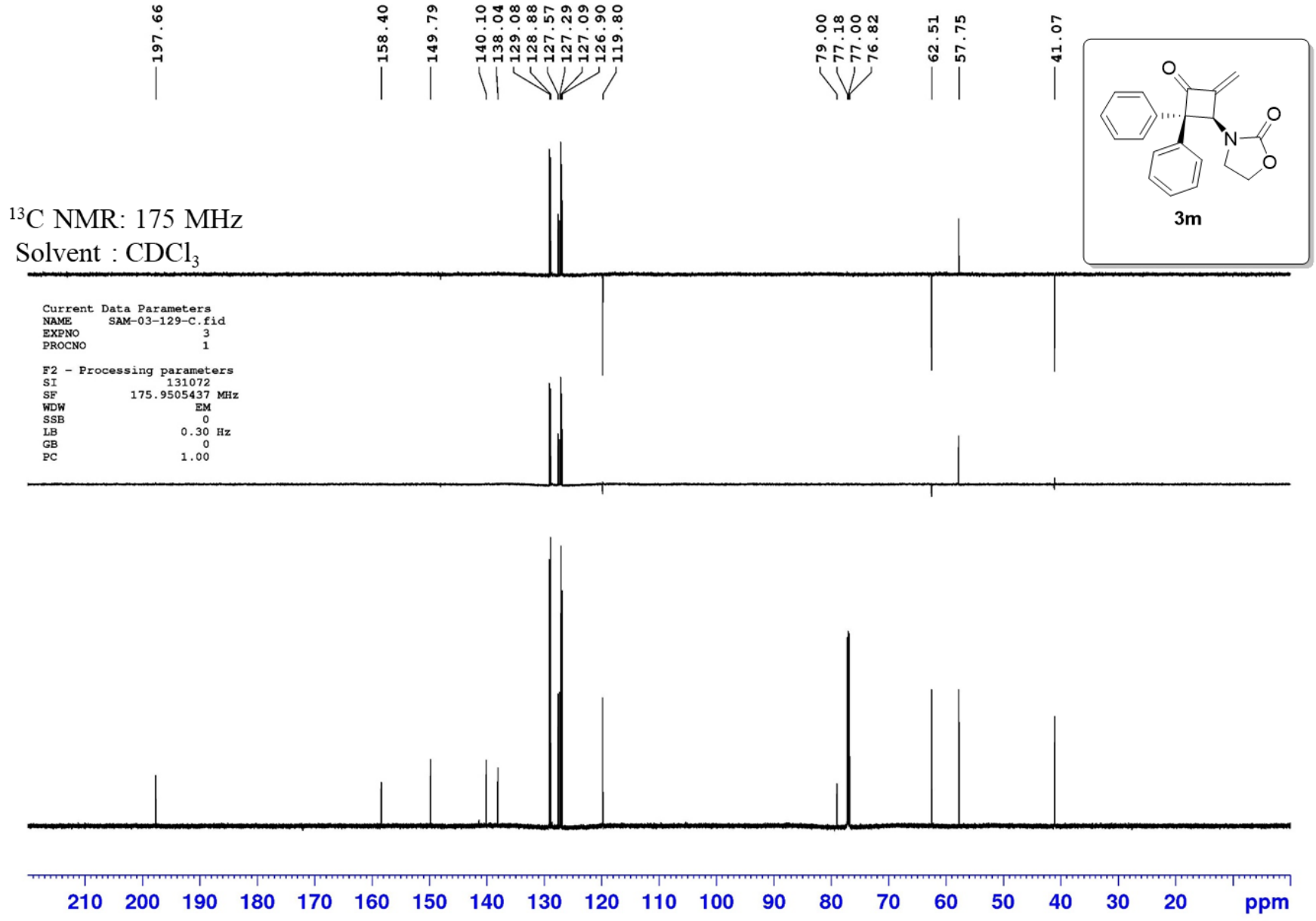
```
Current Data Parameters
NAME      SAM-03-130-H.fid
EXPNO                    1
PROCNO                   1

F2 - Processing parameters
SI                   65536
SF          699.7430960 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



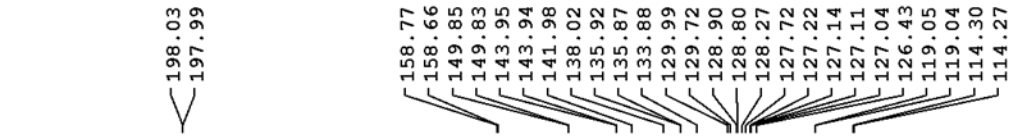

197.16
158.37
149.55
139.74
136.62
133.70
129.28
129.04
128.29
127.54
126.97
120.41
78.25
77.18
77.00
76.82
62.50
57.82
41.21
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
NAME SAM-03-130-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505411 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Cl
O
N
O
O
3l
(dr > 25:1)
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 : $CDCl_3$

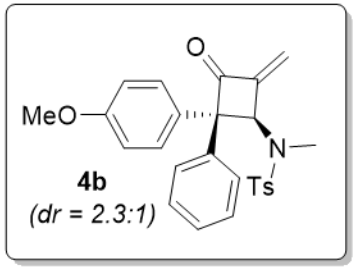



197.66
158.40
149.79
140.10
138.04
129.08
128.88
127.57
127.29
127.09
126.90
119.80
79.00
77.18
77.00
76.82
62.51
57.75
41.07
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
NAME SAM-03-129-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505437 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3m
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

7.770
7.763
7.759
7.751
7.717
7.706
7.646
7.633
7.402
7.391
7.326
7.323
7.319
7.313
7.271
7.260
7.249
7.244
7.238
7.231
7.207
7.196
7.186
7.176
6.870
6.858
6.810
6.797
5.978
5.974
5.970
5.960
5.956
5.953
5.898
5.894
4.140
4.138
4.123
4.120
3.744
3.730
2.429
2.059
2.013
1.586
MeO
O
N
Ts
4b
(dr = 2.3:1)
^{1}H NMR: 700 MHz
Solvent : $CDCl_3$
Current Data Parameters
NAME 1
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
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 ppm
3.232
1.142
2.221
2.054
5.596
2.868
1.768
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm
3.23
1.14
2.22
2.05
5.60
2.87
1.77
2.11
1.18
0.45
1.02
1.52
1.02
0.42
3.09
1.30
4.39
1.33
3.04

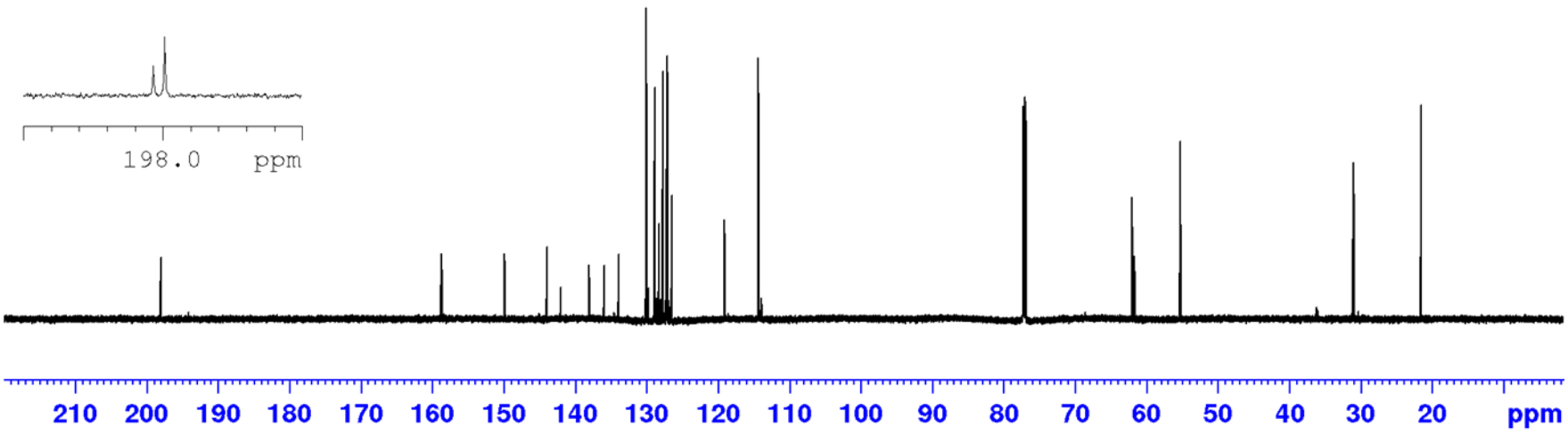


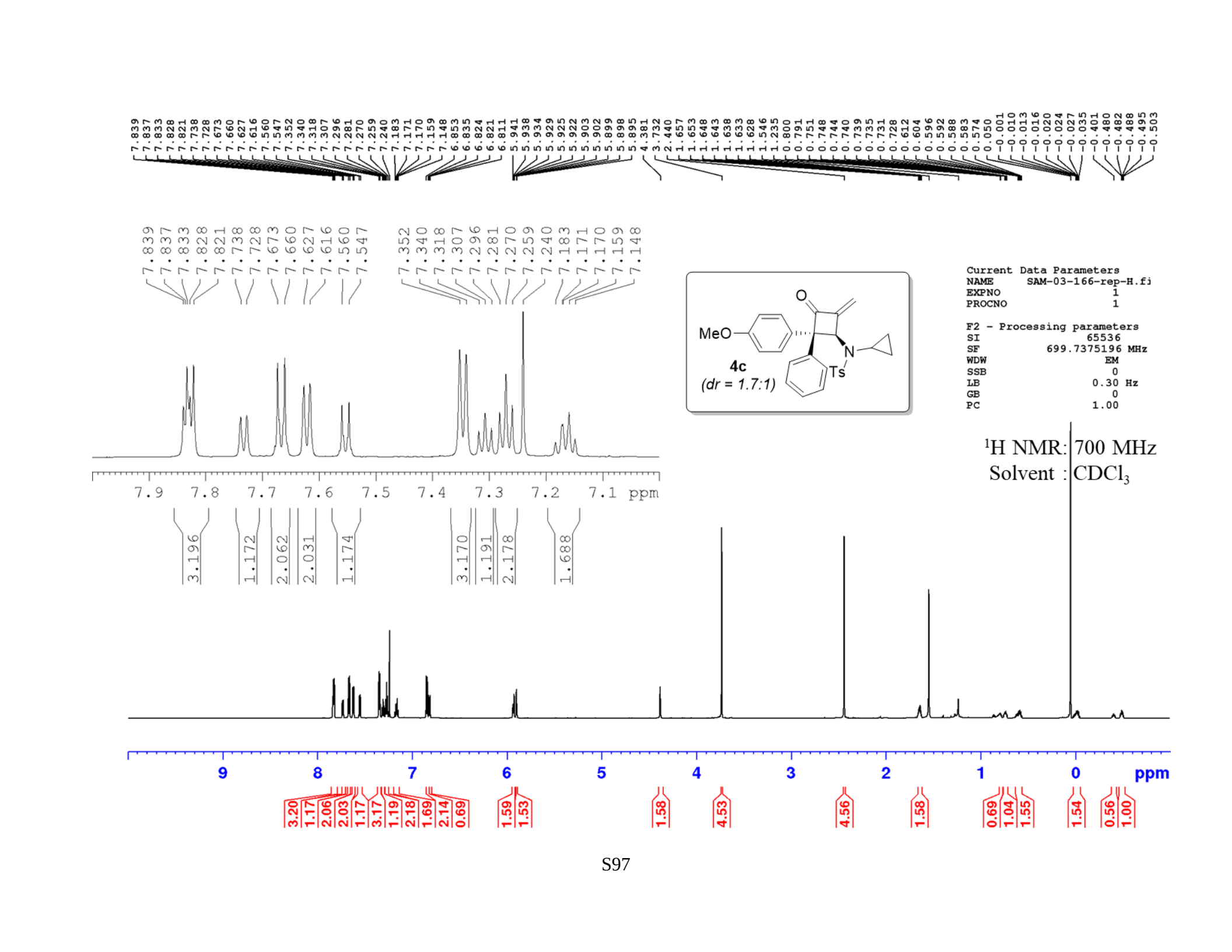
^{13}C NMR: 175 MHz
Solvent : $CDCl_3$

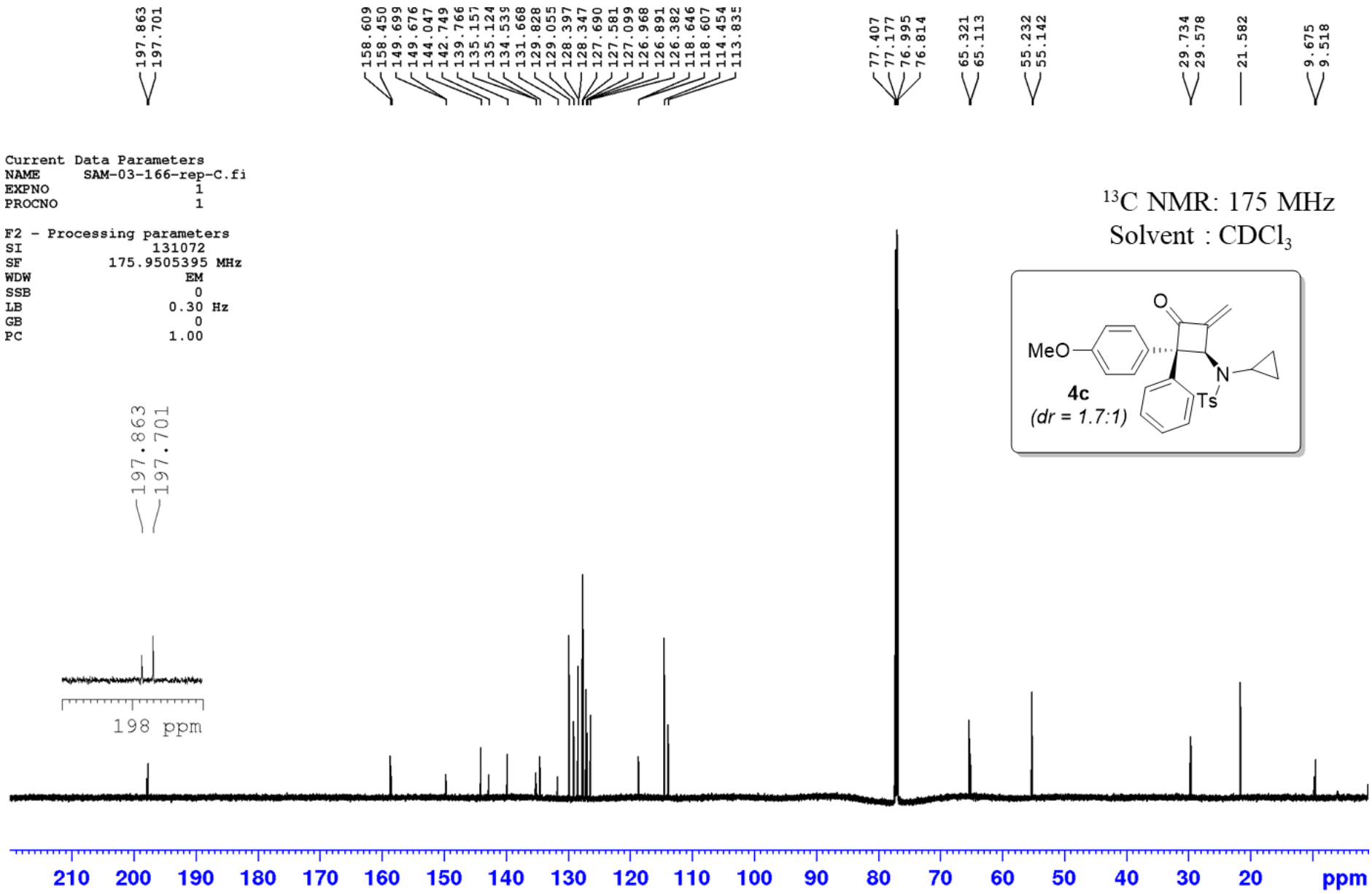


198.032
197.991

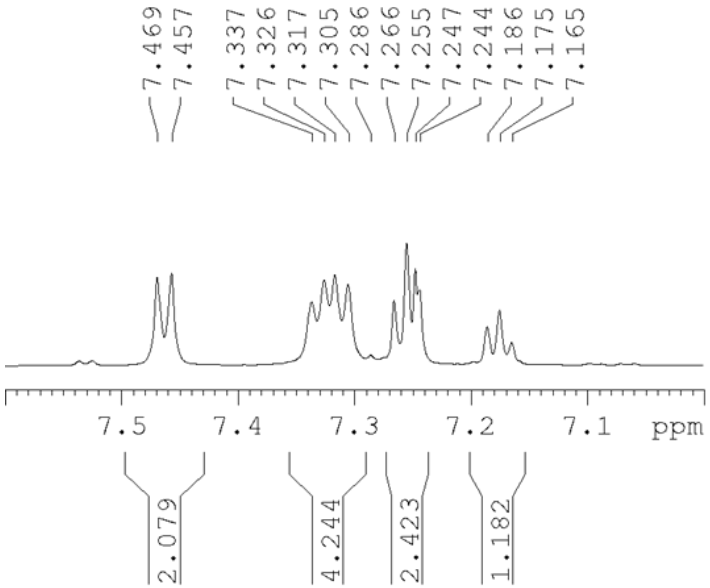
```
Current Data Parameters
NAME      SAM-03-153-C.fid
EXPNO                    1
PROCNO                   1

F2 - Processing parameters
SI                  131072
SF          175.9505381 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

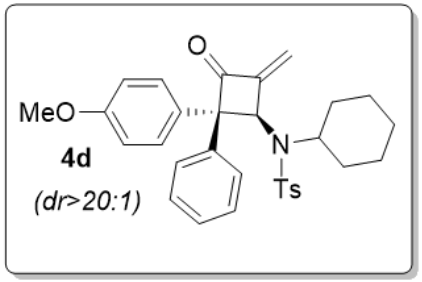



7.839 7.837 7.833 7.828 7.821 7.738 7.728 7.673 7.660 7.627 7.616 7.560 7.547 7.352 7.340 7.318 7.307 7.296 7.281 7.270 7.259 7.240 7.183 7.171 7.170 7.159 7.148 6.853 6.835 6.824 6.821 6.811 5.941 5.938 5.934 5.929 5.925 5.922 5.903 5.902 5.899 5.898 5.895 4.381 3.732 2.440 1.657 1.653 1.648 1.643 1.638 1.633 1.628 1.546 1.235 0.800 0.791 0.751 0.748 0.744 0.740 0.739 0.735 0.731 0.728 0.612 0.604 0.596 0.592 0.588 0.583 0.574 0.050 -0.001 -0.010 -0.013 -0.016 -0.020 -0.024 -0.027 -0.035 -0.401 -0.480 -0.482 -0.488 -0.495 -0.503
MeO
O
N
Ts
4c
(dr = 1.7:1)
Current Data Parameters
NAME SAM-03-166-rep-H.fi
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7375196 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1H NMR: 700 MHz
Solvent : CDCl3
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 ppm
3.196 1.172 2.062 2.031 1.174 3.170 1.191 2.178 1.688
9 8 7 6 5 4 3 2 1 0 ppm
3.20 1.17 2.06 2.03 1.17 3.17 1.19 2.18 1.69 2.14 0.69 1.59 1.53 1.58 4.53 4.56 1.58 0.69 1.04 1.55 1.54 0.56 1.00

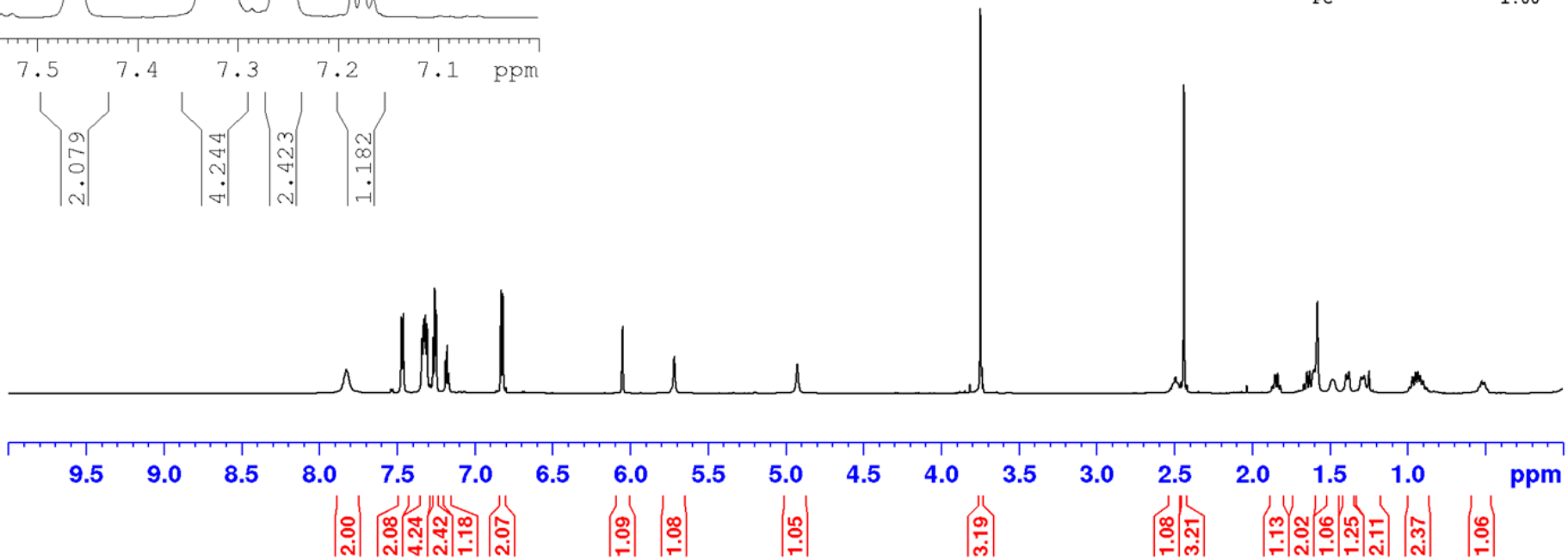

197.863
197.701
158.609
158.450
149.699
149.676
144.047
142.749
139.766
135.157
135.124
134.539
131.668
129.828
129.055
128.397
128.347
127.690
127.581
127.099
126.968
126.891
126.382
118.646
118.607
114.454
113.835
77.407
77.177
76.995
76.814
65.321
65.113
55.232
55.142
29.734
29.578
21.582
9.675
9.518
Current Data Parameters
NAME SAM-03-166-rep-C.fi
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505395 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13C NMR: 175 MHz
Solvent : CDCl3
MeO
O
N
Ts
4c
(dr = 1.7:1)
198 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

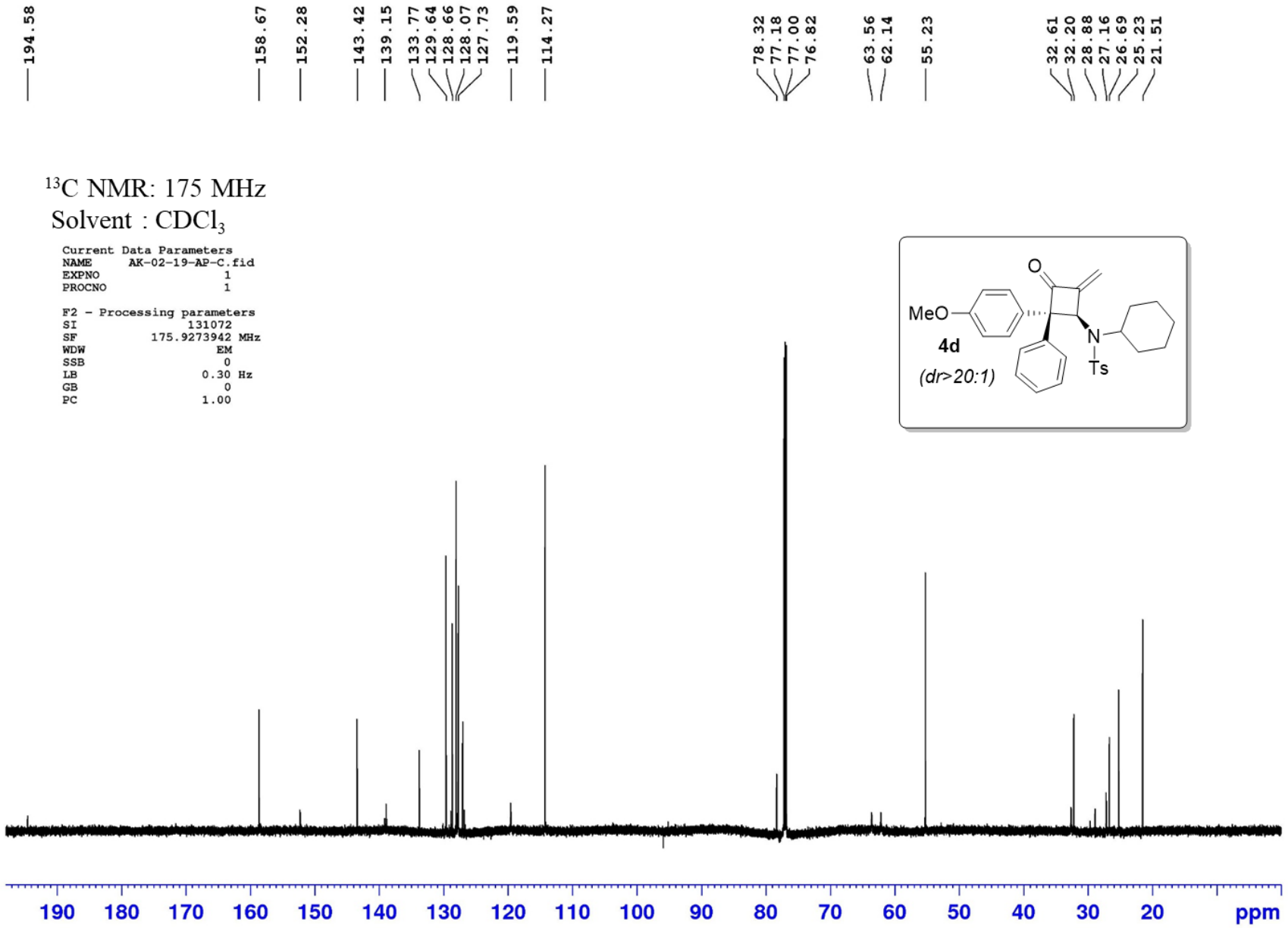
^{1}H NMR: 700 MHz
Solvent : $CDCl_3$

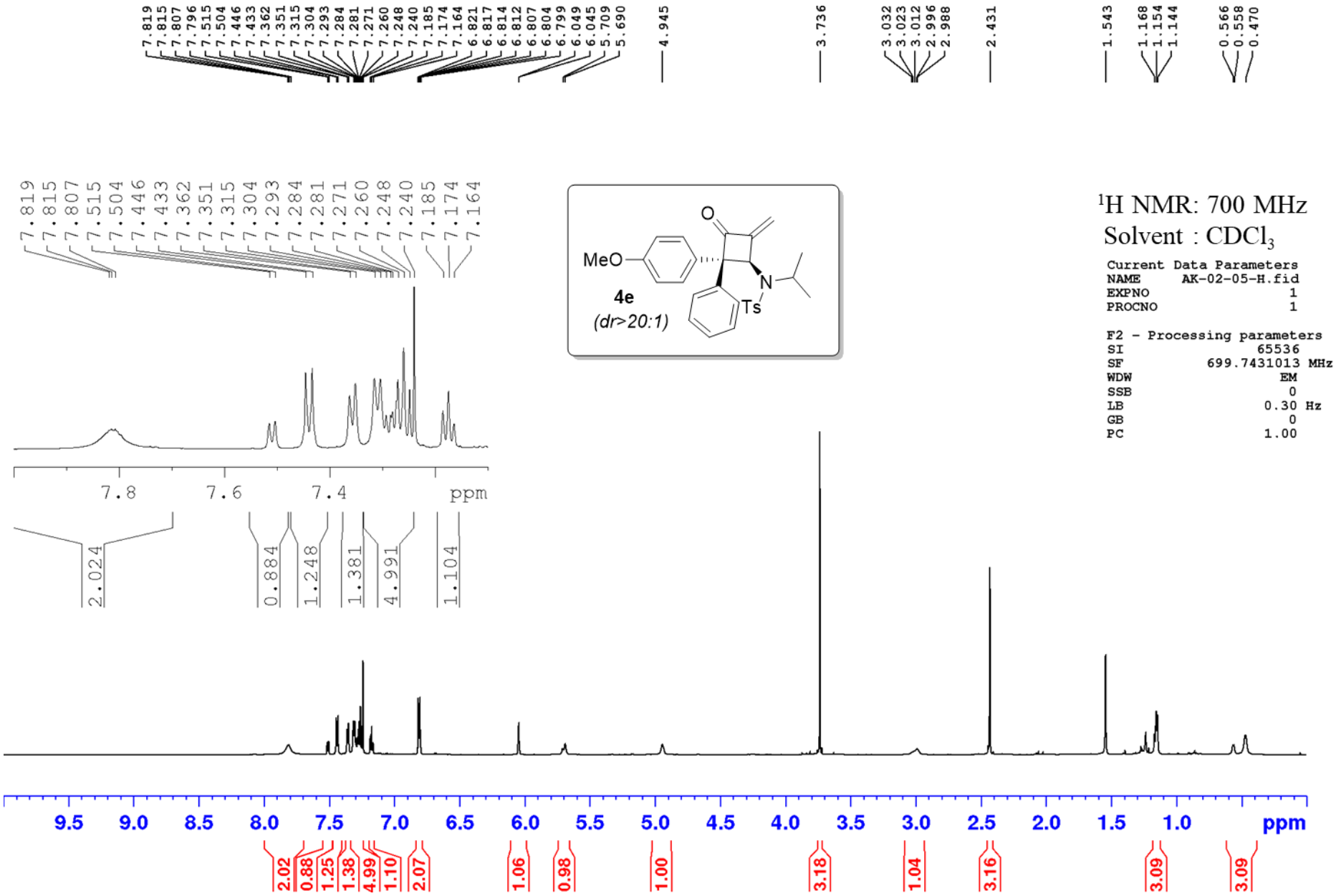





```
Current Data Parameters
NAME      AK-02-19-AP-H.fid
EXPNO                     1
PROCNO                    1

F2 - Processing parameters
SI                    65536
SF          699.7430945 MHz
WDW                      EM
SSB                       0
LB                 0.30 Hz
GB                        0
PC                     1.00
```




194.58
158.67
152.28
143.42
139.15
133.77
129.64
128.66
128.07
127.73
119.59
114.27
78.32
77.18
77.00
76.82
63.56
62.14
55.23
32.61
32.20
28.88
27.16
26.69
25.23
21.51
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
NAME AK-02-19-AP-C.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9273942 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
4d
(dr>20:1)
O
N
Ts
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

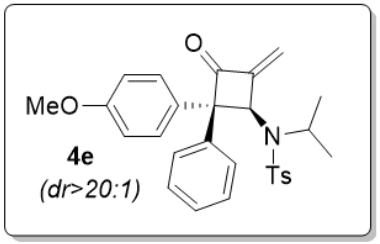

7.819
7.815
7.807
7.796
7.515
7.504
7.446
7.433
7.362
7.351
7.315
7.304
7.293
7.284
7.281
7.271
7.260
7.248
7.240
7.185
7.174
7.164
6.821
6.817
6.814
6.812
6.807
6.804
6.799
6.049
6.045
5.709
5.690
4.945
3.736
3.032
3.023
3.012
2.996
2.988
2.431
1.543
1.168
1.154
1.144
0.566
0.558
0.470
MeO
O
N
Ts
4e
(dr>20:1)
1H NMR: 700 MHz
Solvent : CDCl3
Current Data Parameters
NAME AK-02-05-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7431013 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
2.024
0.884
1.248
1.381
4.991
1.104
2.02
0.88
1.25
1.38
4.99
1.10
2.07
1.06
0.98
1.00
3.18
1.04
3.16
3.09
3.09

198.85
198.59
158.60
152.07
143.46
141.91
138.96
138.84
133.71
130.82
129.60
129.04
128.83
128.58
127.91
127.80
127.66
127.06
127.02
126.63
119.61
114.22
114.03
78.32
78.08
77.18
77.00
76.82
63.58
55.15
53.05
22.22
22.00
21.44

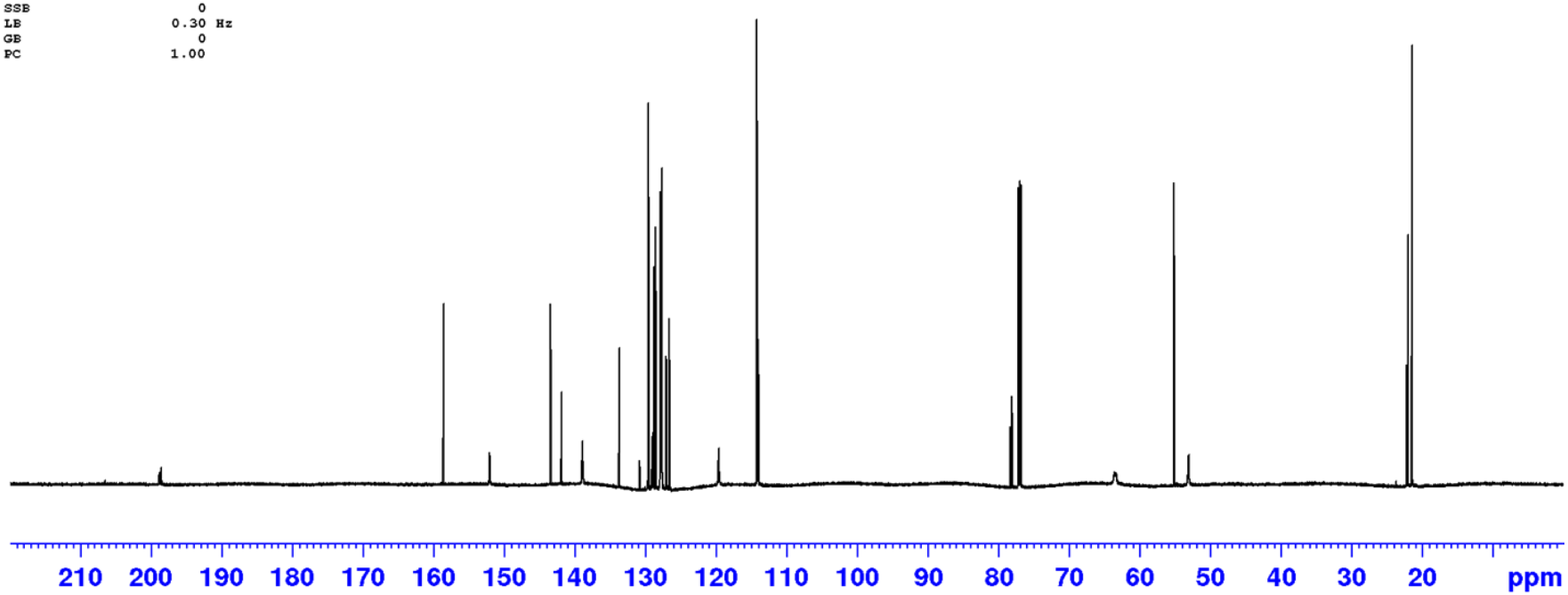
^{13}C NMR: 175 MHz
Solvent : $CDCl_3$

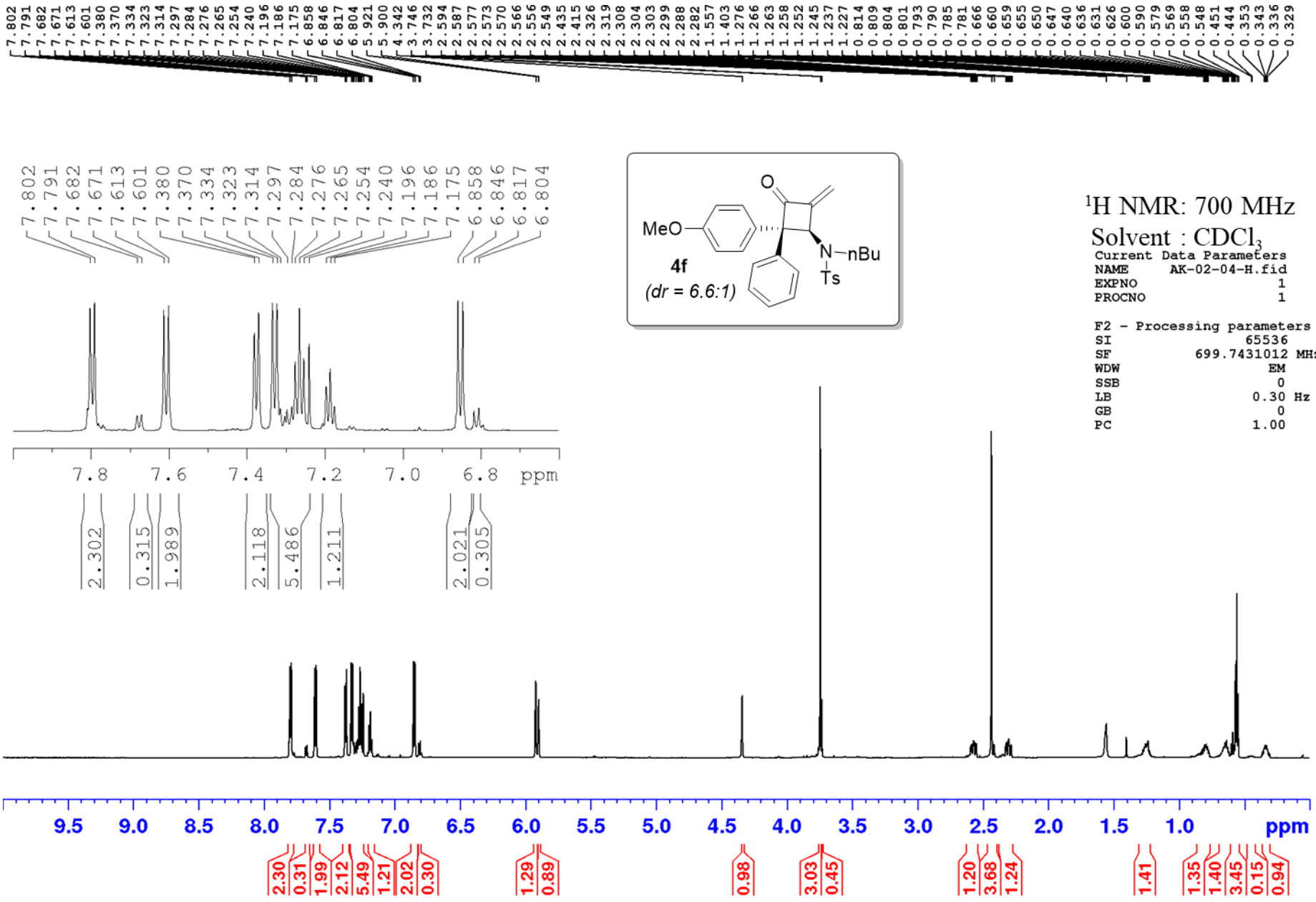
AK-02-203

```
Current Data Parameters
NAME      AK-02-203-C.fid
EXPNO                   3
PROCNO                  1

F2 - Processing parameters
SI                 131072
SF          175.9505559 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```

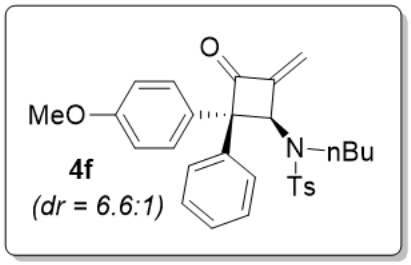


4e
(dr>20:1)




7.802 7.791 7.682 7.671 7.613 7.601 7.380 7.370 7.334 7.323 7.314 7.297 7.284 7.276 7.265 7.254 7.240 7.196 7.186 7.175 6.858 6.846 6.817 6.804 5.921 5.900 4.342 3.746 3.732 2.594 2.587 2.577 2.573 2.570 2.566 2.556 2.549 2.435 2.415 2.326 2.319 2.308 2.304 2.303 2.299 2.288 2.282 1.557 1.403 1.276 1.266 1.263 1.258 1.252 1.245 1.237 1.227 0.814 0.809 0.804 0.801 0.793 0.790 0.785 0.781 0.666 0.660 0.659 0.655 0.650 0.647 0.640 0.636 0.631 0.626 0.600 0.590 0.579 0.569 0.558 0.548 0.451 0.444 0.353 0.343 0.336 0.329
7.802 7.791 7.682 7.671 7.613 7.601 7.380 7.370 7.334 7.323 7.314 7.297 7.284 7.276 7.265 7.254 7.240 7.196 7.186 7.175 6.858 6.846 6.817 6.804
7.8 7.6 7.4 7.2 7.0 6.8 ppm
2.302 0.315 1.989 2.118 5.486 1.211 2.021 0.305
MeO
4f
(dr = 6.6:1)
N–nBu
Ts
O
1H NMR: 700 MHz
Solvent : CDCl3
Current Data Parameters
NAME AK-02-04-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
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.30 0.31 1.99 2.12 5.49 1.21 2.02 0.30 1.29 0.89 0.98 3.03 0.45 1.20 3.68 1.24 1.41 1.35 1.40 3.45 0.15 0.94

198.70
158.65
151.24
143.84
138.39
136.87
134.06
129.92
128.72
127.95
127.70
127.31
127.15
119.19
114.28
77.83
77.20
77.02
76.83
63.88
55.26
47.68
31.24
21.56
20.36
13.31



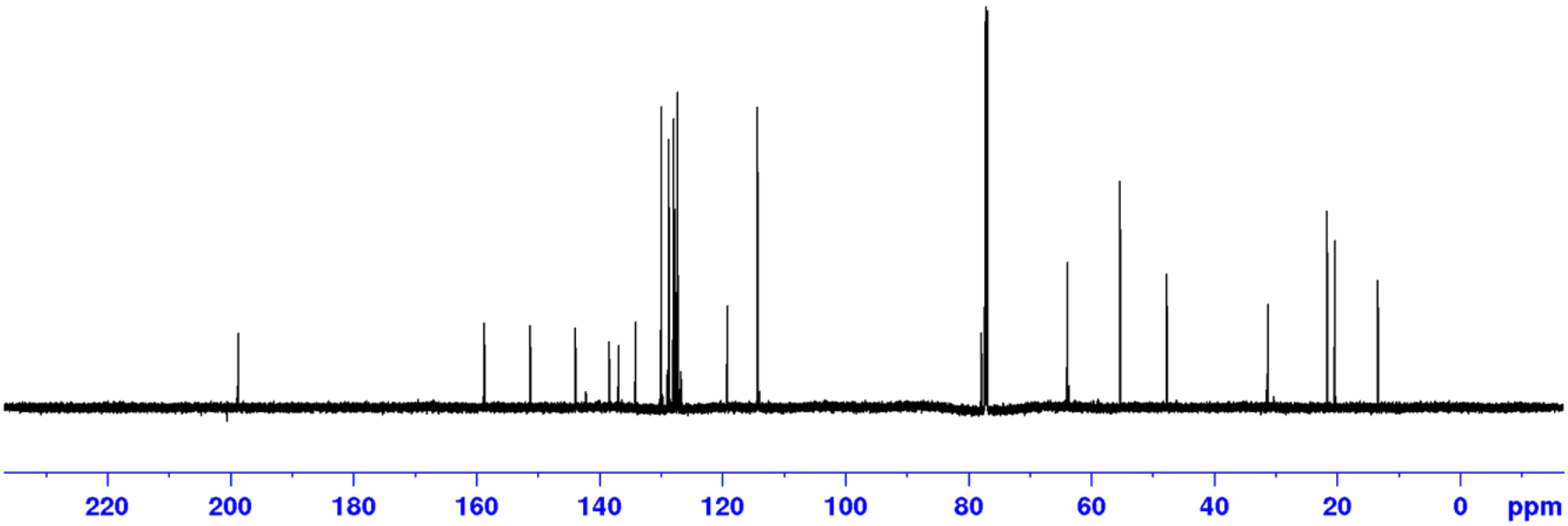
4f

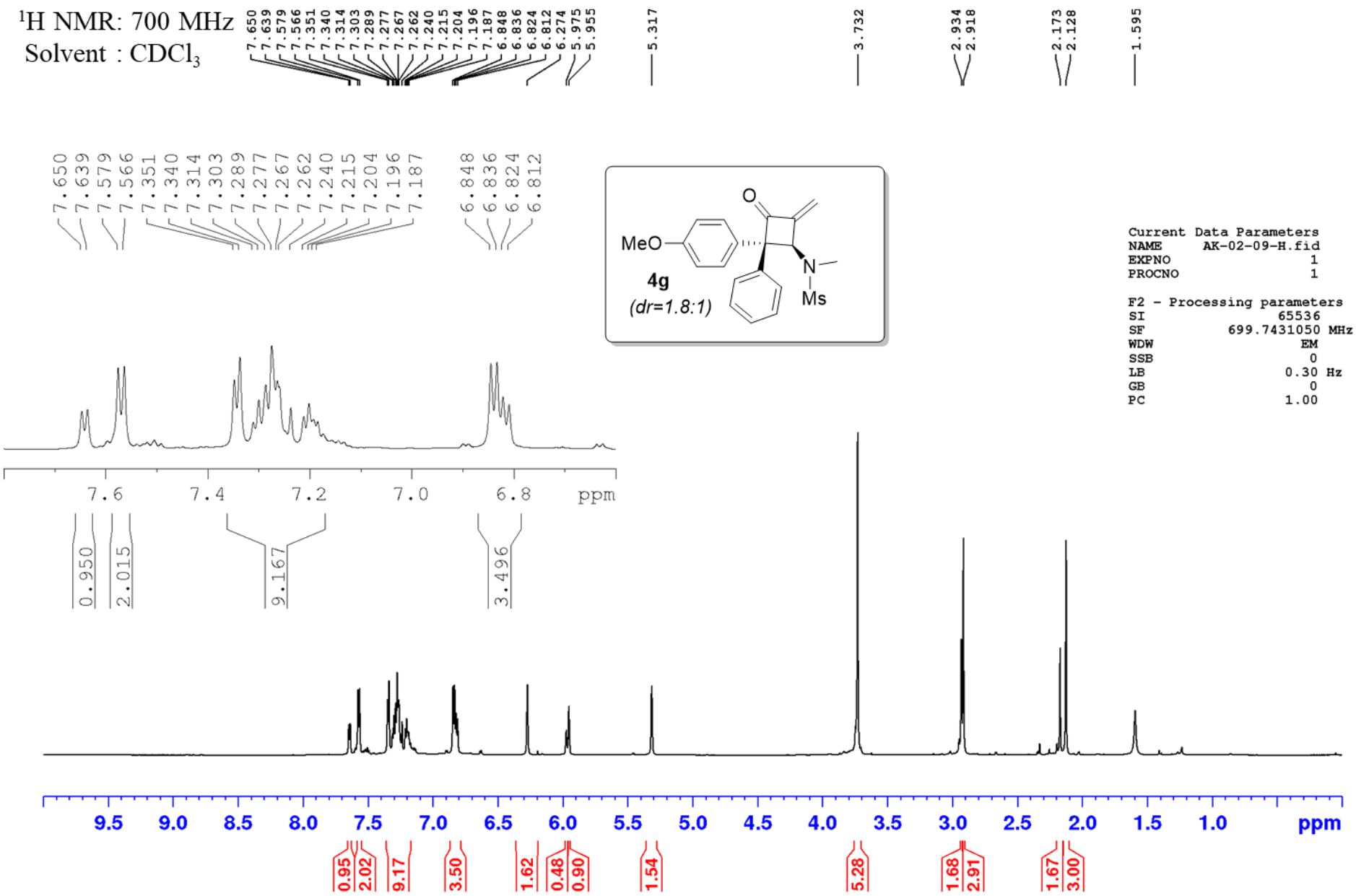
(dr = 6.6:1)

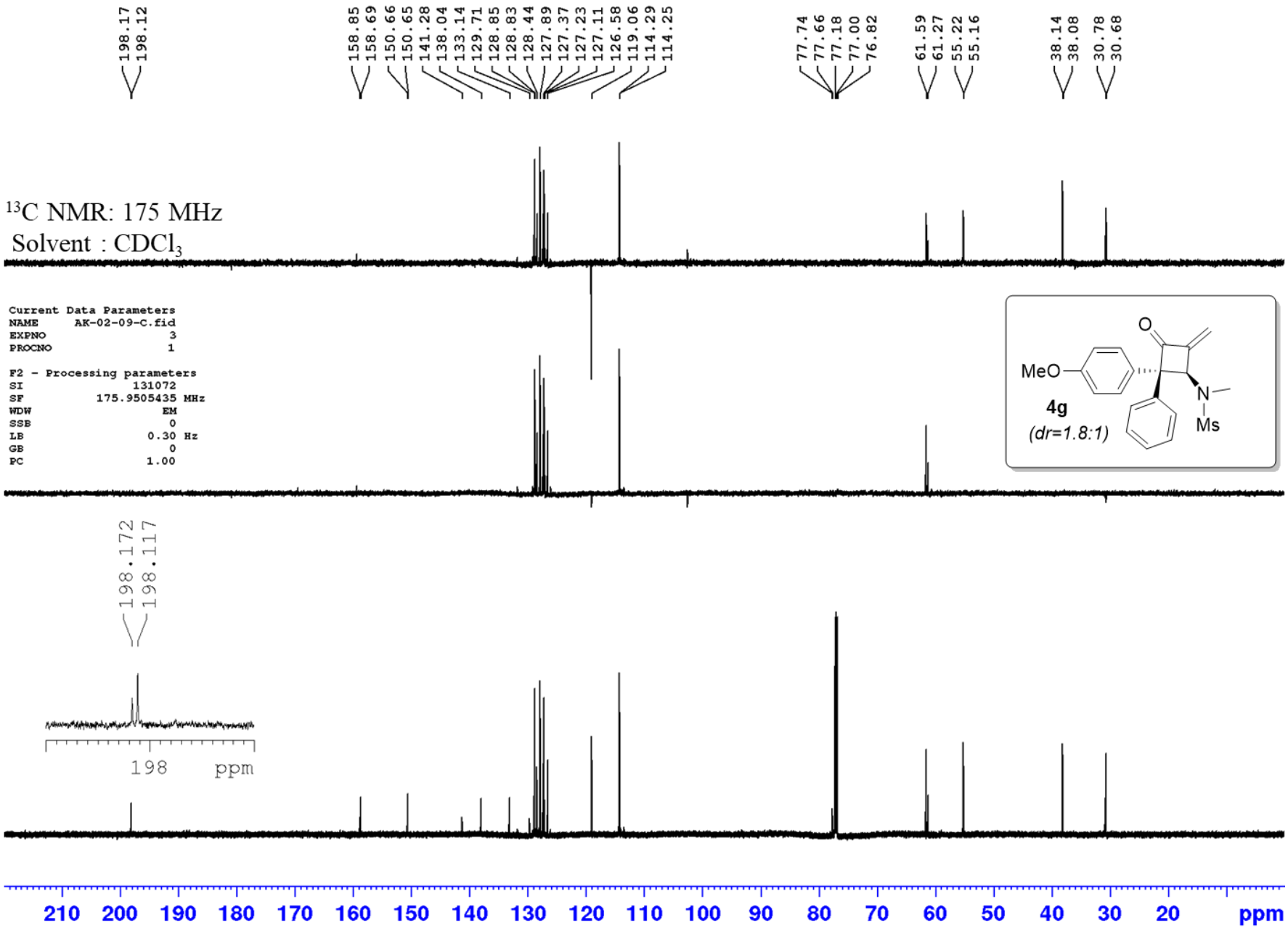
```
Current Data Parameters
NAME      AK-02-04-C.fid
EXPNO                  1
PROCNO                 1

F2 - Processing parameters
SI                131072
SF          175.9505378 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

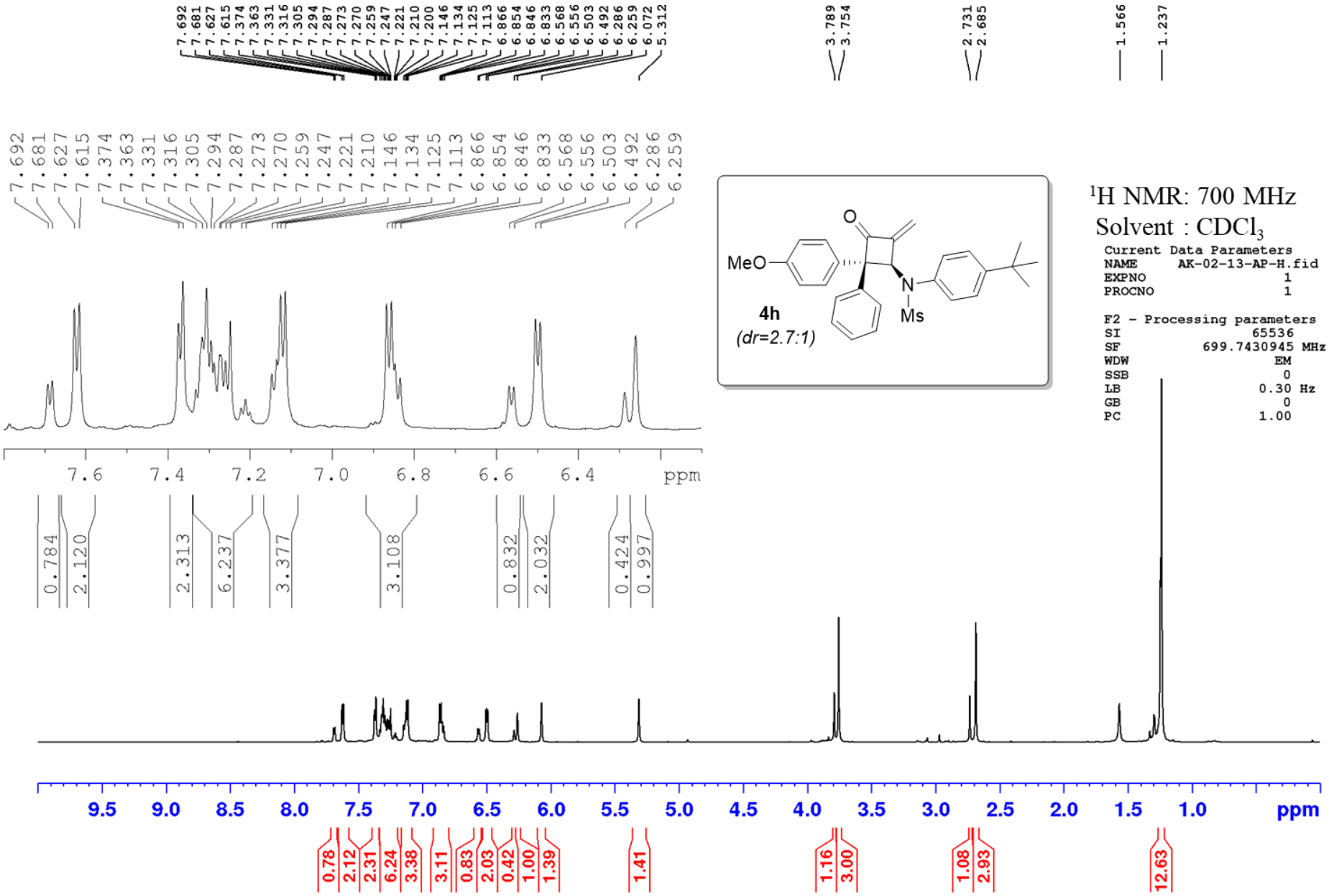
^{13}C NMR: 175 MHz

Solvent : $CDCl_3$




1H NMR: 700 MHz
Solvent : CDCl3
7.650
7.639
7.579
7.566
7.351
7.340
7.314
7.303
7.289
7.277
7.267
7.262
7.240
7.215
7.204
7.196
7.187
6.848
6.836
6.824
6.812
6.274
5.975
5.955
5.317
3.732
2.934
2.918
2.173
2.128
1.595
MeO
4g
(dr=1.8:1)
O
N
Ms
Current Data Parameters
NAME AK-02-09-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7431050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
0.950
2.015
9.167
3.496
0.95
2.02
9.17
3.50
1.62
0.48
0.90
1.54
5.28
1.68
2.91
1.67
3.00

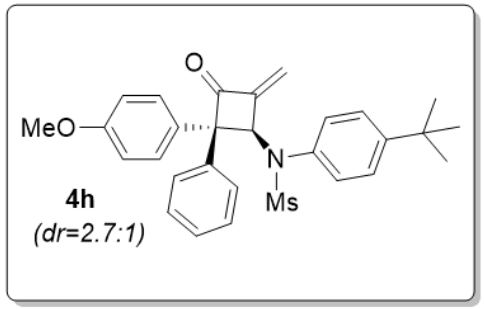

198.17
198.12
158.85
158.69
150.66
150.65
141.28
138.04
133.14
129.71
128.85
128.83
128.44
127.89
127.37
127.23
127.11
126.58
119.06
114.29
114.25
77.74
77.66
77.18
77.00
76.82
61.59
61.27
55.22
55.16
38.14
38.08
30.78
30.68
13C NMR: 175 MHz
Solvent : CDCl3
Current Data Parameters
NAME AK-02-09-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505435 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
O
N
Ms
4g
(dr=1.8:1)
198.172
198.117
198 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.692
7.681
7.627
7.615
7.374
7.363
7.331
7.316
7.305
7.294
7.287
7.273
7.270
7.259
7.247
7.221
7.210
7.200
7.146
7.134
7.125
7.113
6.866
6.854
6.846
6.833
6.568
6.556
6.503
6.492
6.286
6.259
6.072
5.312
3.789
3.754
2.731
2.685
1.566
1.237
MeO
O
N
Ms
4h
(dr=2.7:1)
1H NMR: 700 MHz
Solvent : CDCl3
Current Data Parameters
NAME AK-02-13-AP-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7430945 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
0.784
2.120
2.313
6.237
3.377
3.108
0.832
2.032
0.424
0.997
0.78
2.12
2.31
6.24
3.38
3.11
0.83
2.03
0.42
1.00
1.39
1.41
1.16
3.00
1.08
2.93
12.63

198.99
198.74
158.86
158.75
152.00
151.99
151.10
151.07
140.97
139.28
133.68
133.57
132.79
131.65
131.61
131.15
129.99
128.85
128.81
128.56
128.43
127.33
127.26
127.19
125.93
125.93
121.63
121.60
114.24
113.84
79.38
79.17
77.20
77.02
76.83
63.92
63.71
55.30
55.24
39.74
39.68
34.59
34.58
31.18
31.17

Current Data Parameters
NAME AK-02-13-AP-C.fid
EXPNO 3
PROCNO 1

F2 - Processing parameters
SI 131072
SF 175.9505386 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

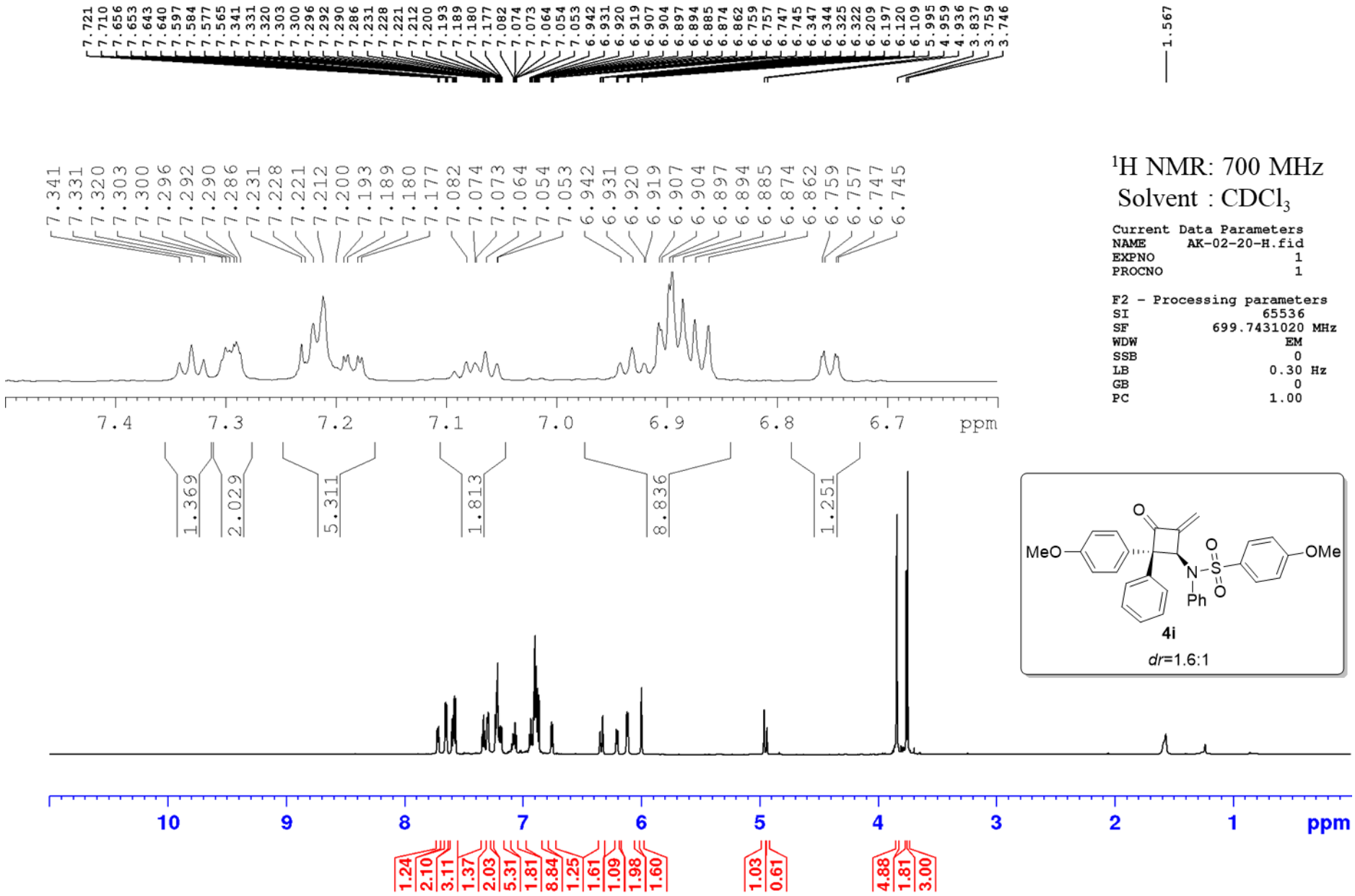


4h
(dr=2.7:1)

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

198.992
198.744
199 ppm

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



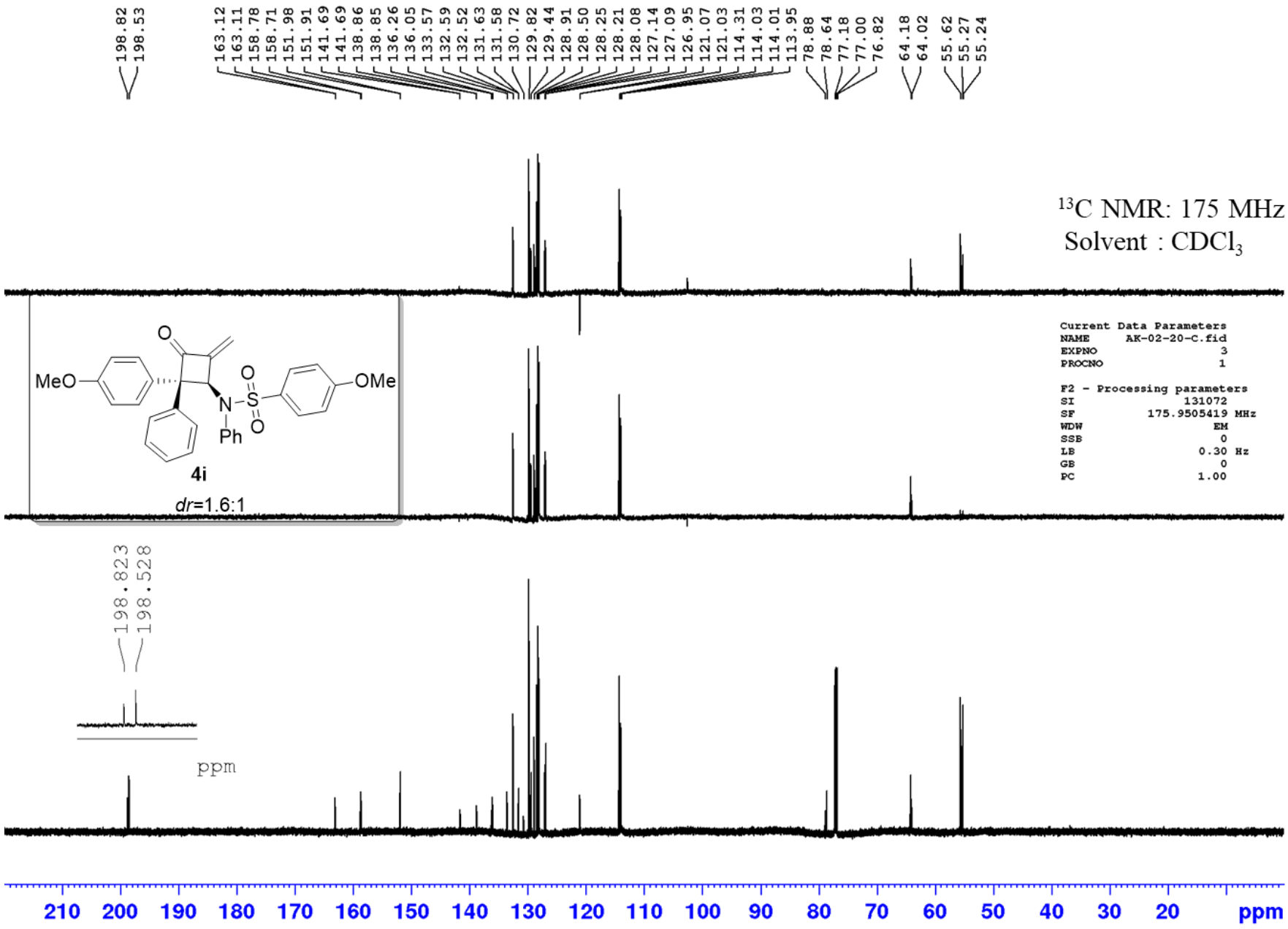
^{1}H NMR: 700 MHz
Solvent : $CDCl_3$

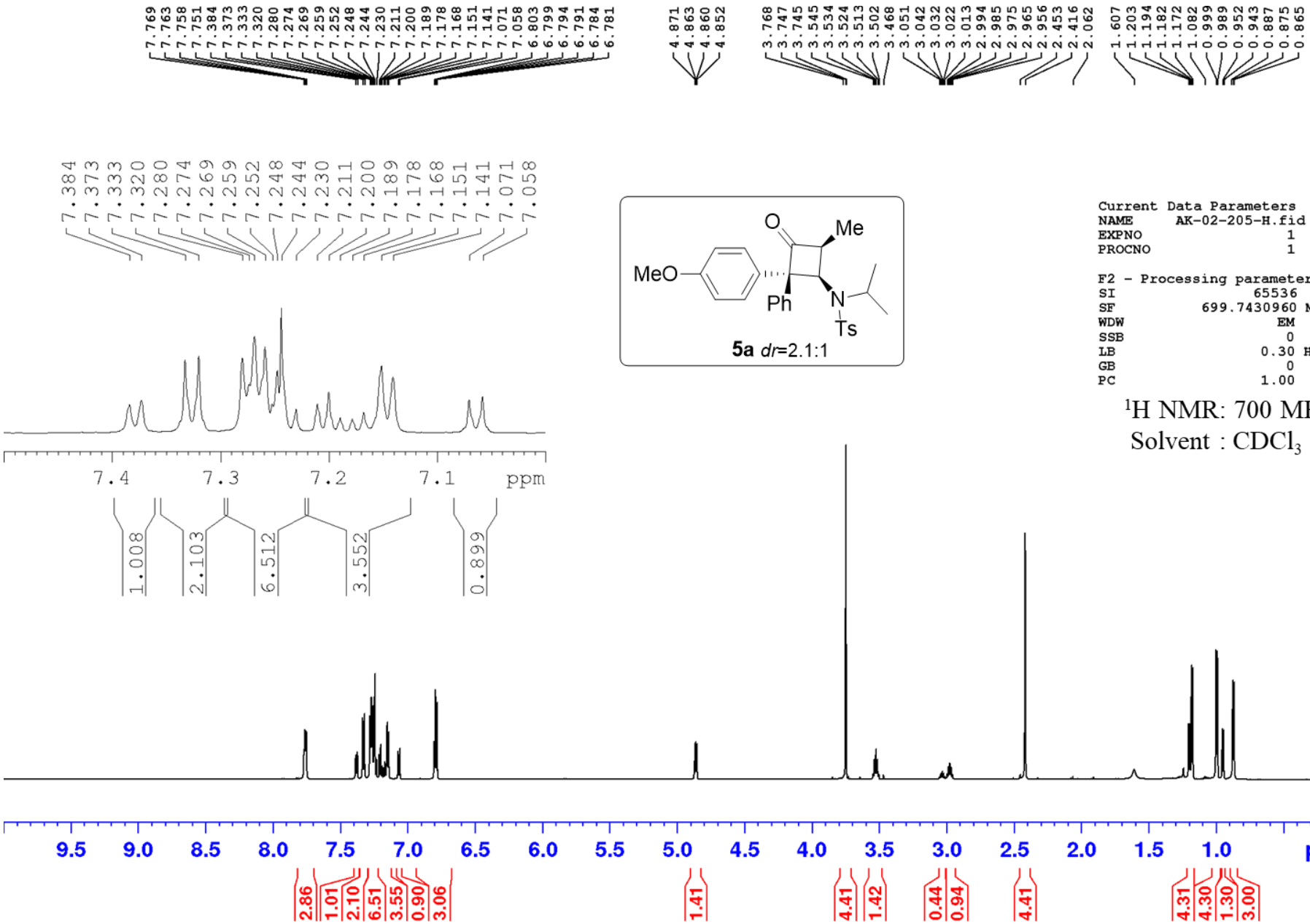
Current Data Parameters

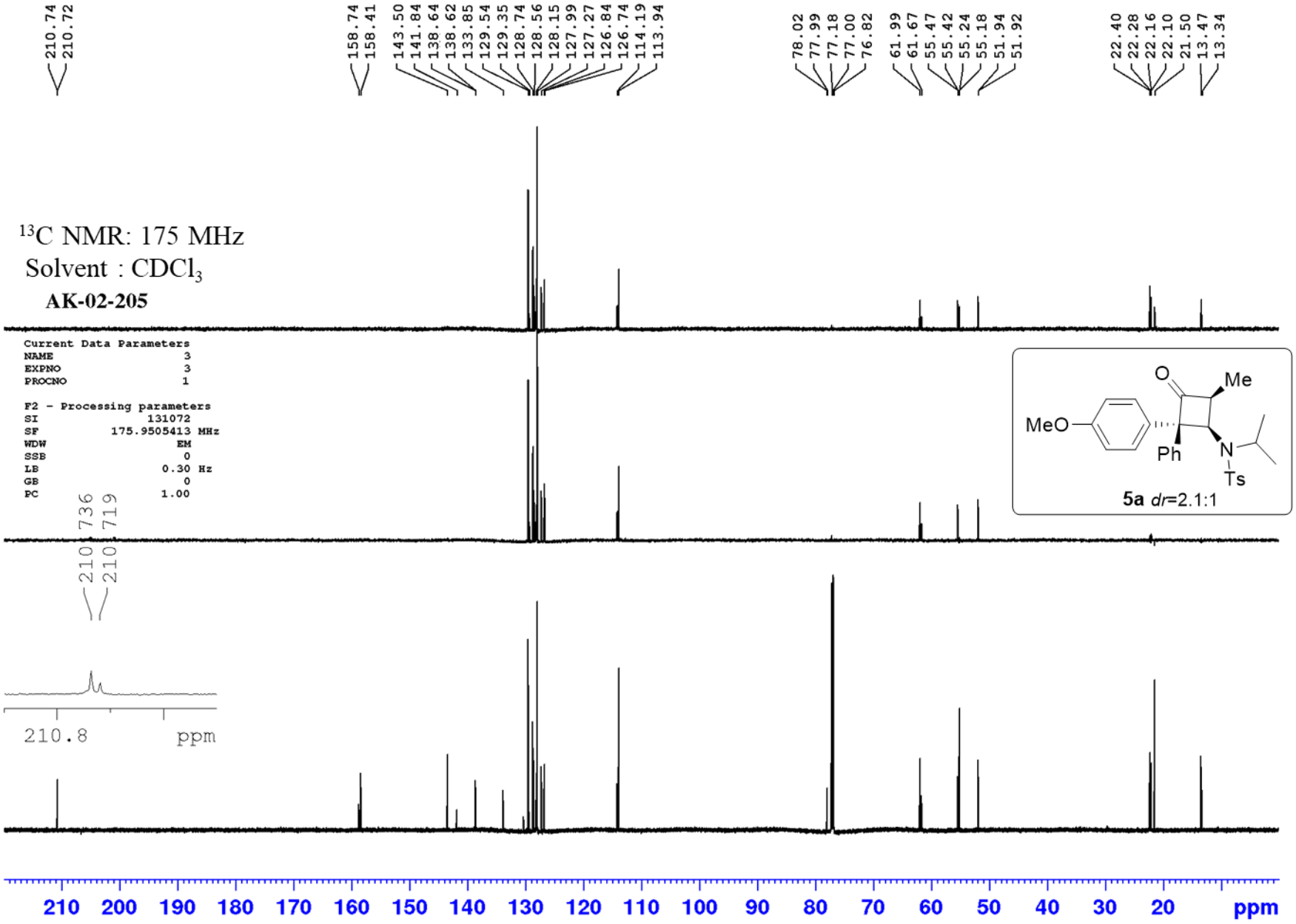
NAME	AK-02-20-H.fid
EXPNO	1
PROCNO	1

F2 - Processing parameters

SI	65536
SF	699.7431020 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00


198.82
198.53
163.12
163.11
158.78
158.71
151.98
151.91
141.69
141.69
138.86
138.85
136.26
136.05
133.57
132.59
132.52
131.63
131.58
130.72
129.82
129.44
128.91
128.50
128.25
128.21
128.08
127.14
127.09
126.95
121.07
121.03
114.31
114.03
114.01
113.95
78.88
78.64
77.18
77.00
76.82
64.18
64.02
55.62
55.27
55.24
13C NMR: 175 MHz
Solvent : CDCl3
MeO
OMe
N
S
Ph
4i
dr=1.6:1
Current Data Parameters
NAME AK-02-20-C.fid
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505419 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
198.823
198.528
ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.769 7.763 7.758 7.751 7.384 7.373 7.333 7.320 7.280 7.274 7.269 7.259 7.252 7.248 7.244 7.230 7.211 7.200 7.189 7.178 7.168 7.151 7.141 7.071 7.058 6.803 6.799 6.794 6.791 6.784 6.781
4.871 4.863 4.860 4.852
3.768 3.747 3.745 3.545 3.534 3.524 3.513 3.502 3.468 3.051 3.042 3.032 3.022 3.013 2.994 2.985 2.975 2.965 2.956 2.453 2.416 2.062
1.607 1.203 1.194 1.182 1.172 1.082 0.999 0.989 0.952 0.943 0.887 0.875 0.865
7.384 7.373 7.333 7.320 7.280 7.274 7.269 7.259 7.252 7.248 7.244 7.230 7.211 7.200 7.189 7.178 7.168 7.151 7.141 7.071 7.058
7.4 7.3 7.2 7.1 ppm
1.008 2.103 6.512 3.552 0.899
O Me MeO Ph N Ts
5a dr=2.1:1
Current Data Parameters
NAME AK-02-205-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7430960 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1H NMR: 700 MHz
Solvent : CDCl3
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.86 1.01 2.10 6.51 3.55 0.90 3.06 1.41 4.41 1.42 0.44 0.94 4.41 4.31 4.30 1.30 3.00


210.74
210.72
158.74
158.41
143.50
141.84
138.64
138.62
133.85
129.54
129.35
128.74
128.56
128.15
127.99
127.27
126.84
126.74
114.19
113.94
78.02
77.99
77.18
77.00
76.82
61.99
61.67
55.47
55.42
55.24
55.18
51.94
51.92
22.40
22.28
22.16
22.10
21.50
13.47
13.34
13C NMR: 175 MHz
Solvent : CDCl3
AK-02-205
Current Data Parameters
NAME 3
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505413 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
210.736
210.719
210.8
ppm
MeO
O
Me
Ph
N
Ts
5a dr=2.1:1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

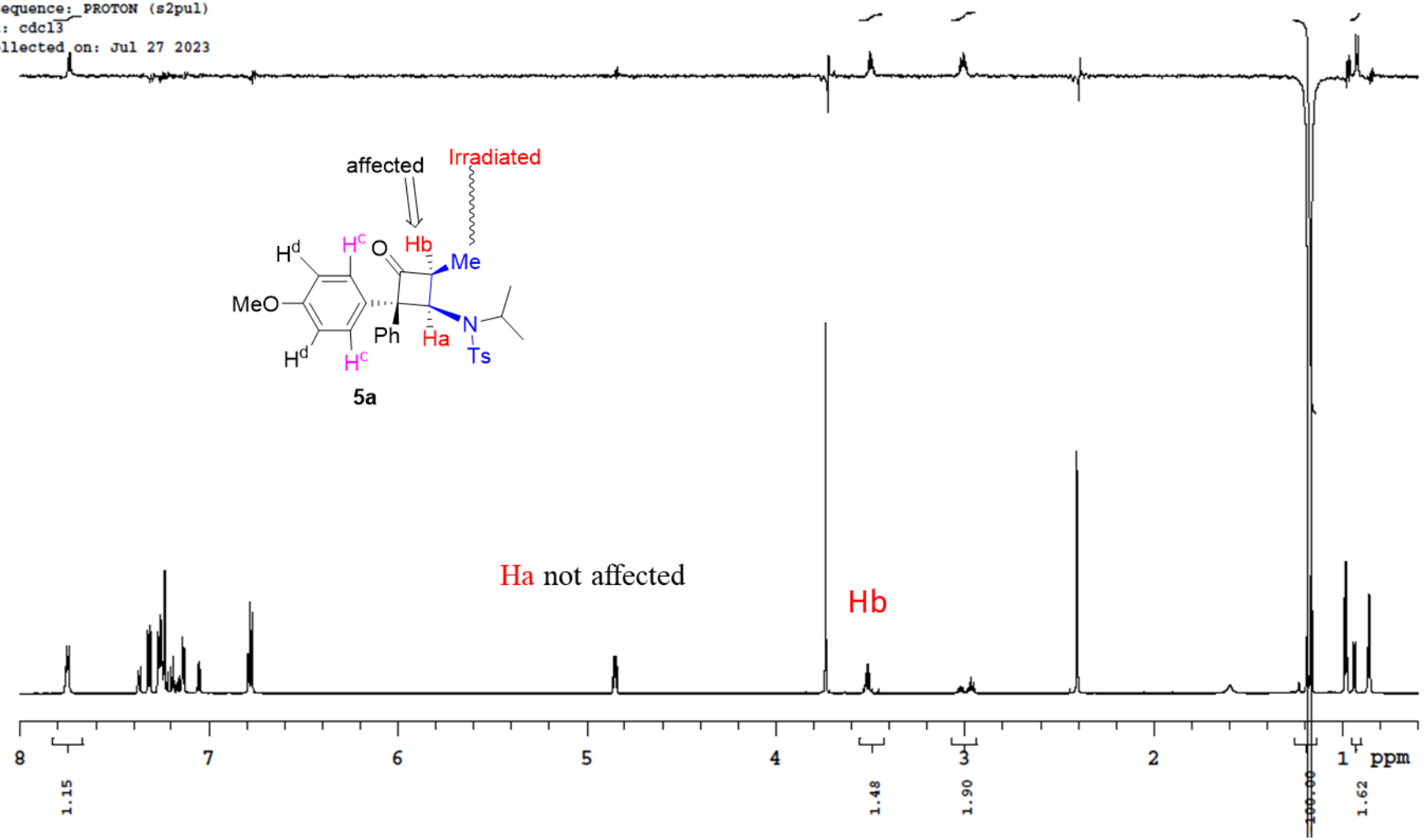
AK-02-205

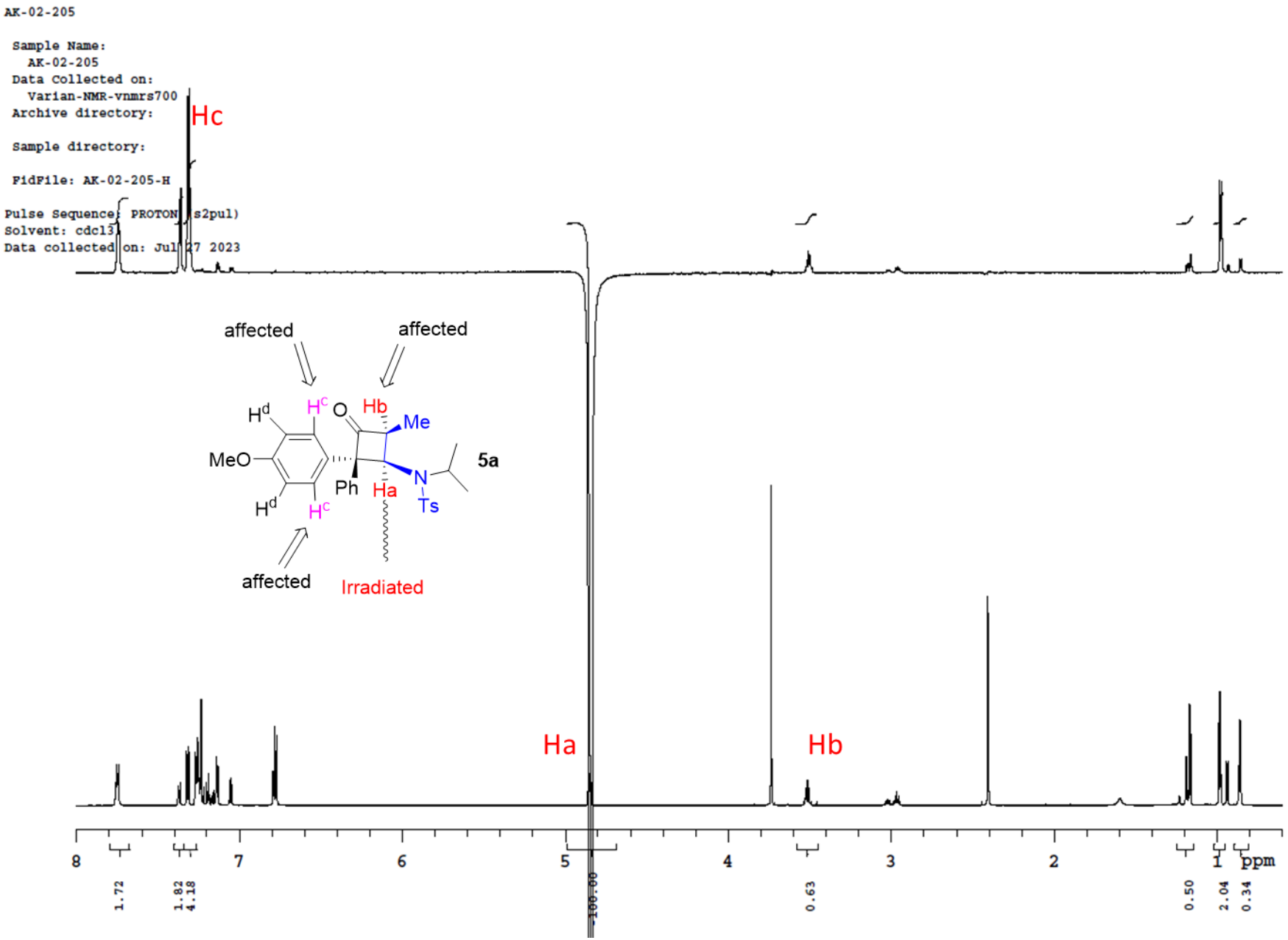
Sample Name:
AK-02-205
Data Collected on:
Varian-NMR-vnmrs700
Archive directory:

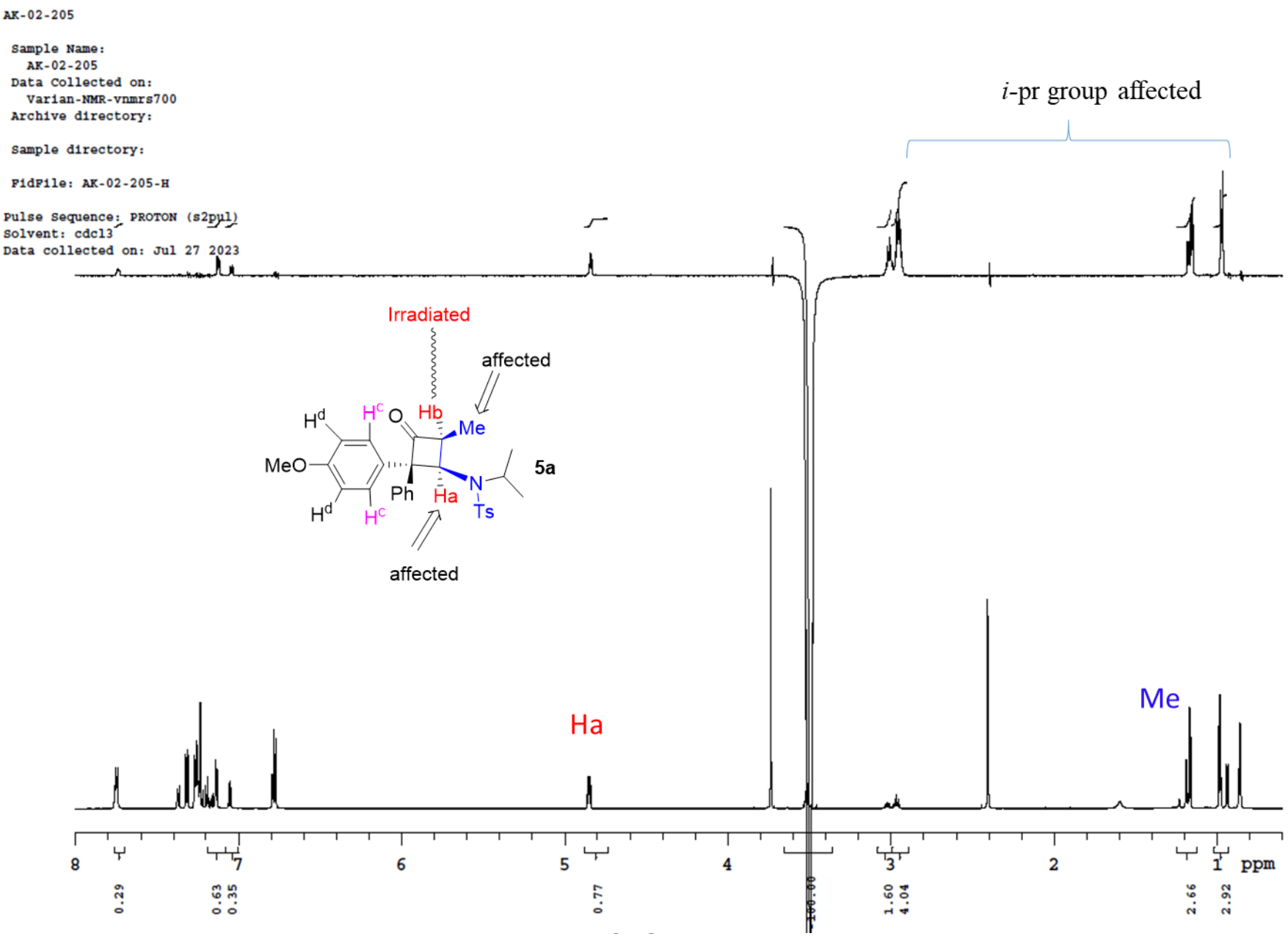
Sample directory:

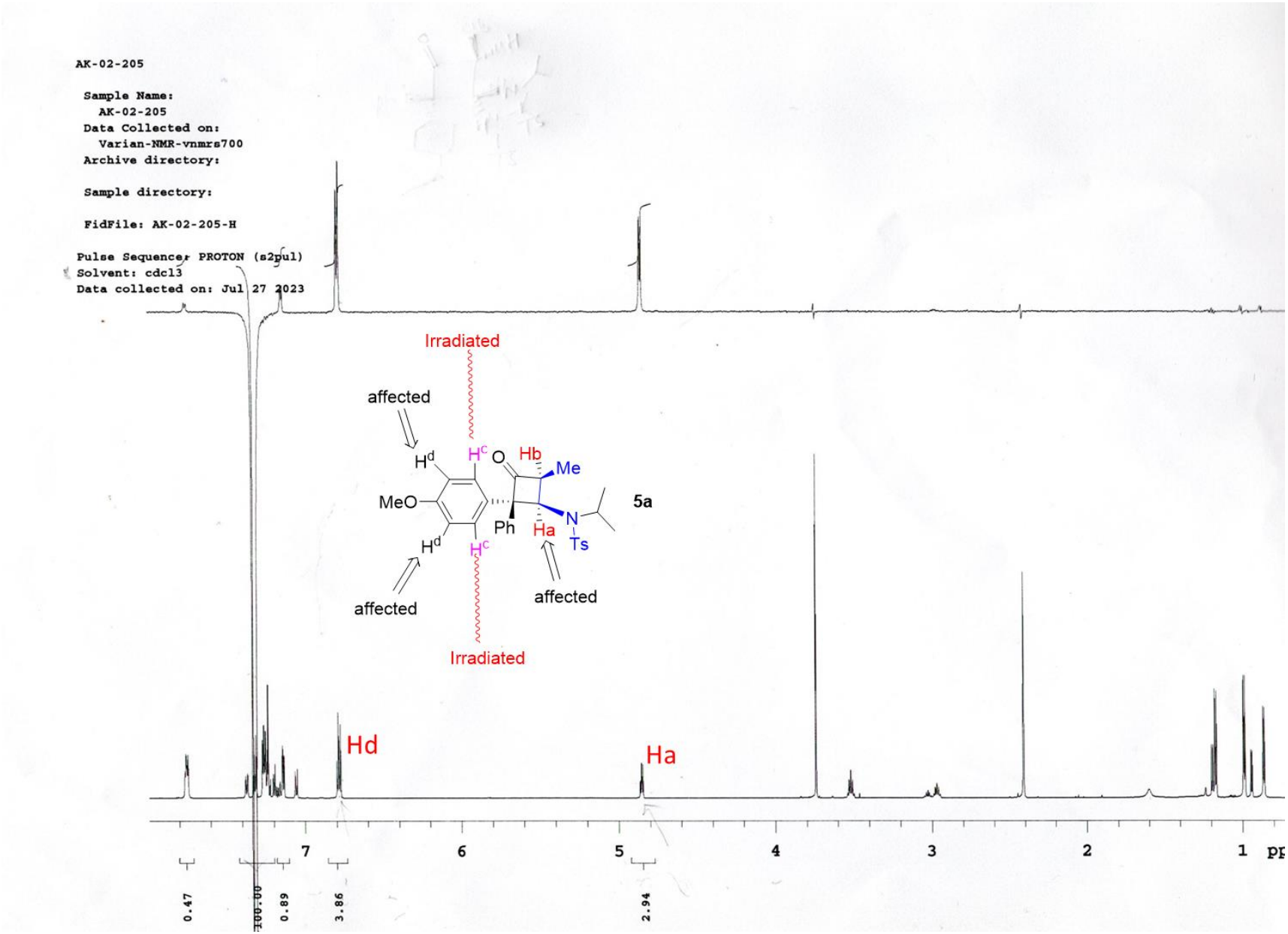
FidFile: AK-02-205-H

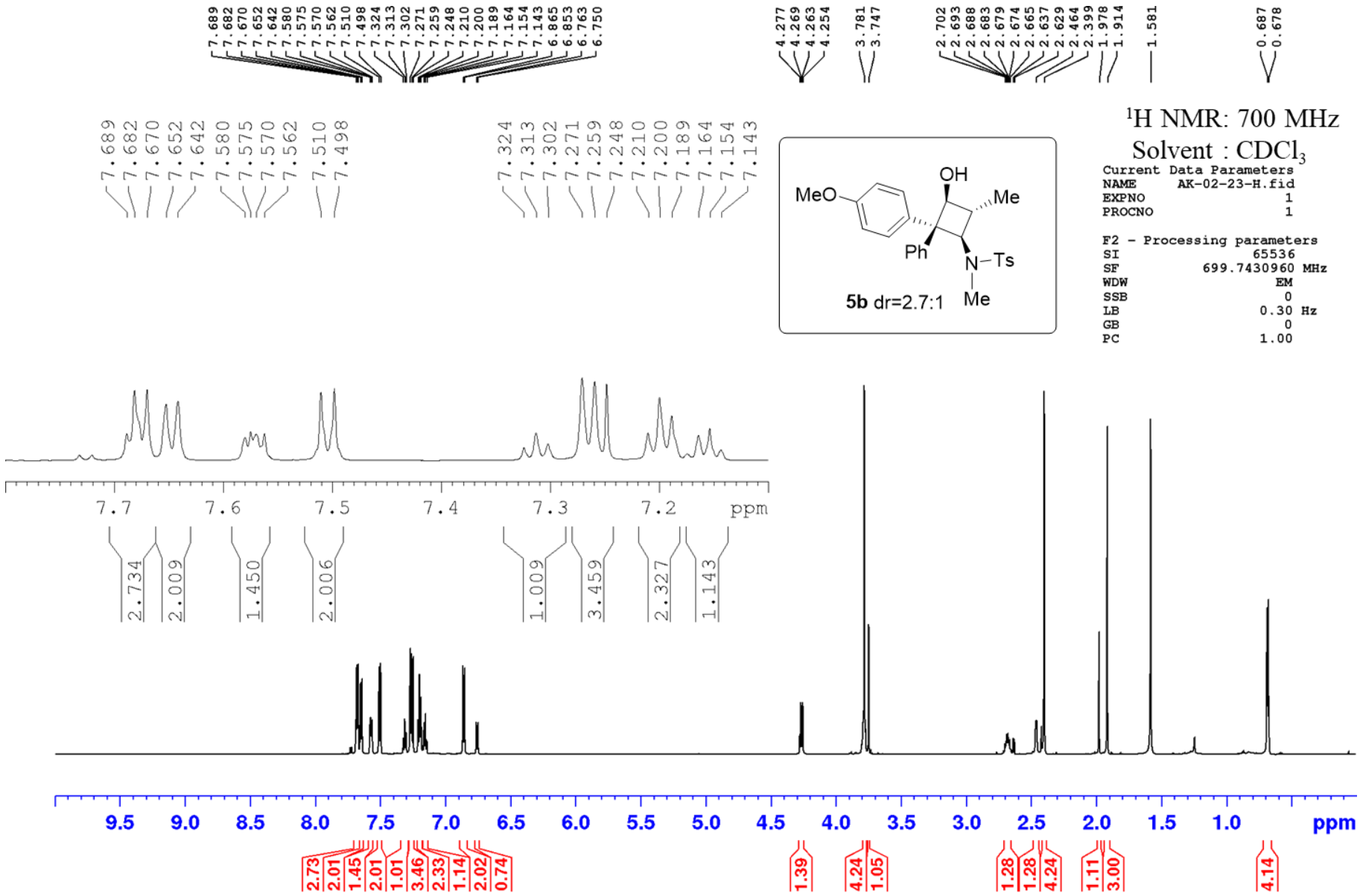
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 27 2023

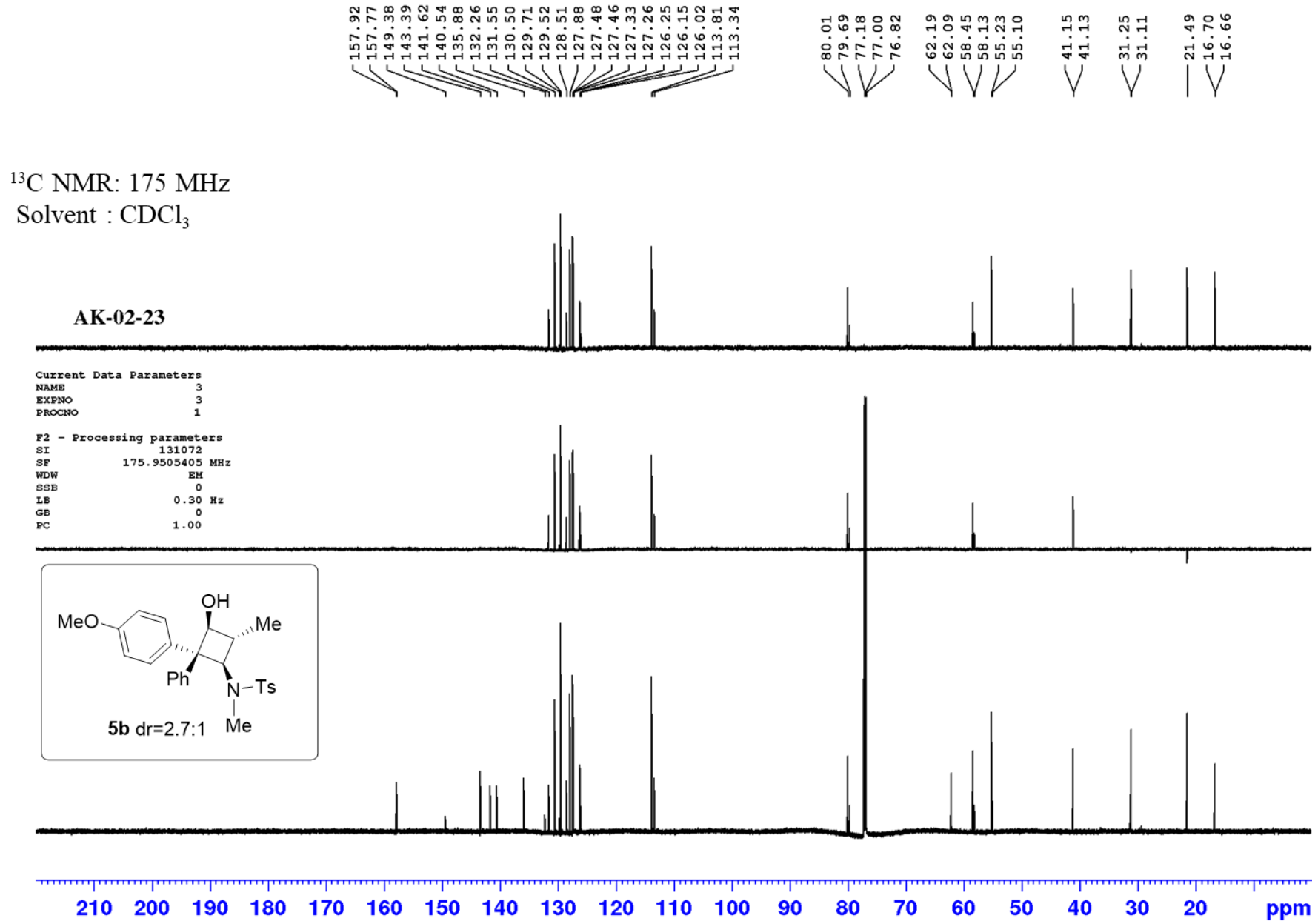



AK-02-205
Sample Name:
AK-02-205
Data Collected on:
Varian-NMR-vnmrs700
Archive directory:
Sample directory:
FidFile: AK-02-205-H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 27 2023
Hc
affected
affected
Hd
Hc
O
Hb
Me
MeO
Ph
Ha
N
Ts
5a
Hd
Hc
affected
Irradiated
Ha
Hb
8
7
6
5
4
3
2
1
ppm
1.72
1.82
4.18
100.00
0.63
0.50
2.04
0.34


AK-02-205
Sample Name:
AK-02-205
Data Collected on:
Varian-NMR-vnmrs700
Archive directory:
Sample directory:
FidFile: AK-02-205-H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 27 2023
i-pr group affected
Irradiated
affected
affected
Hd
HC
O
Hb
Me
MeO
Ph
Ha
N
Ts
5a
Ha
Me
8
7
6
5
4
3
2
1
ppm
0.29
0.63
0.35
0.77
100.00
1.60
4.04
2.66
2.92


AK-02-205
Sample Name:
AK-02-205
Data Collected on:
Varian-NMR-vnmrs700
Archive directory:
Sample directory:
FidFile: AK-02-205-H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jul 27 2023
Irradiated
affected
Hd
Hc
O
Hb
Me
MeO
Ph
Ha
N
Ts
5a
affected
affected
Irradiated
Hd
Ha
7
6
5
4
3
2
1
0.47
100.00
0.89
3.86
2.94


7.689
7.682
7.670
7.652
7.642
7.580
7.575
7.570
7.562
7.510
7.498
7.324
7.313
7.302
7.271
7.259
7.248
7.210
7.200
7.189
7.164
7.154
7.143
6.865
6.853
6.763
6.750
4.277
4.269
4.263
4.254
3.781
3.747
2.702
2.693
2.688
2.683
2.679
2.674
2.665
2.637
2.629
2.464
2.399
1.978
1.914
1.581
0.687
0.678
MeO
OH
Me
Ph
N–Ts
Me
5b dr=2.7:1
1H NMR: 700 MHz
Solvent : CDCl3
Current Data Parameters
NAME AK-02-23-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 65536
SF 699.7430960 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.7
7.6
7.5
7.4
7.3
7.2
ppm
2.734
2.009
1.450
2.006
1.009
3.459
2.327
1.143
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.73
2.01
1.45
2.01
1.01
3.46
2.33
1.14
2.02
0.74
1.39
4.24
1.05
1.28
1.28
4.24
1.11
3.00
4.14


157.92
157.77
149.38
143.39
141.62
140.54
135.88
132.26
131.55
130.50
129.71
129.52
128.51
127.88
127.48
127.46
127.33
127.26
126.25
126.15
126.02
113.81
113.34
80.01
79.69
77.18
77.00
76.82
62.19
62.09
58.45
58.13
55.23
55.10
41.15
41.13
31.25
31.11
21.49
16.70
16.66
13C NMR: 175 MHz
Solvent : CDCl3
AK-02-23
Current Data Parameters
NAME 3
EXPNO 3
PROCNO 1
F2 - Processing parameters
SI 131072
SF 175.9505405 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
OH
Me
Ph
N–Ts
Me
5b dr=2.7:1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

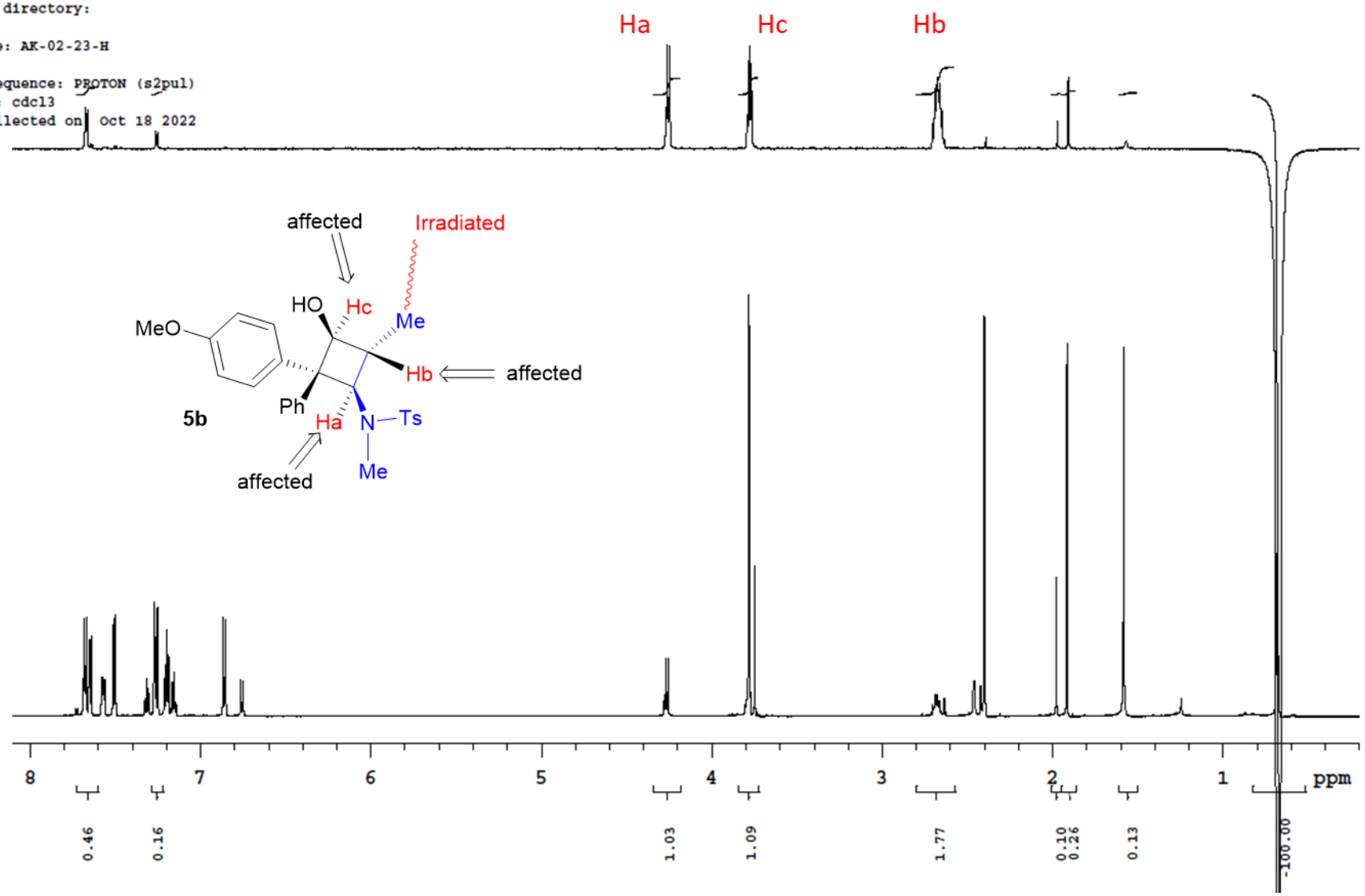
AK-02-23

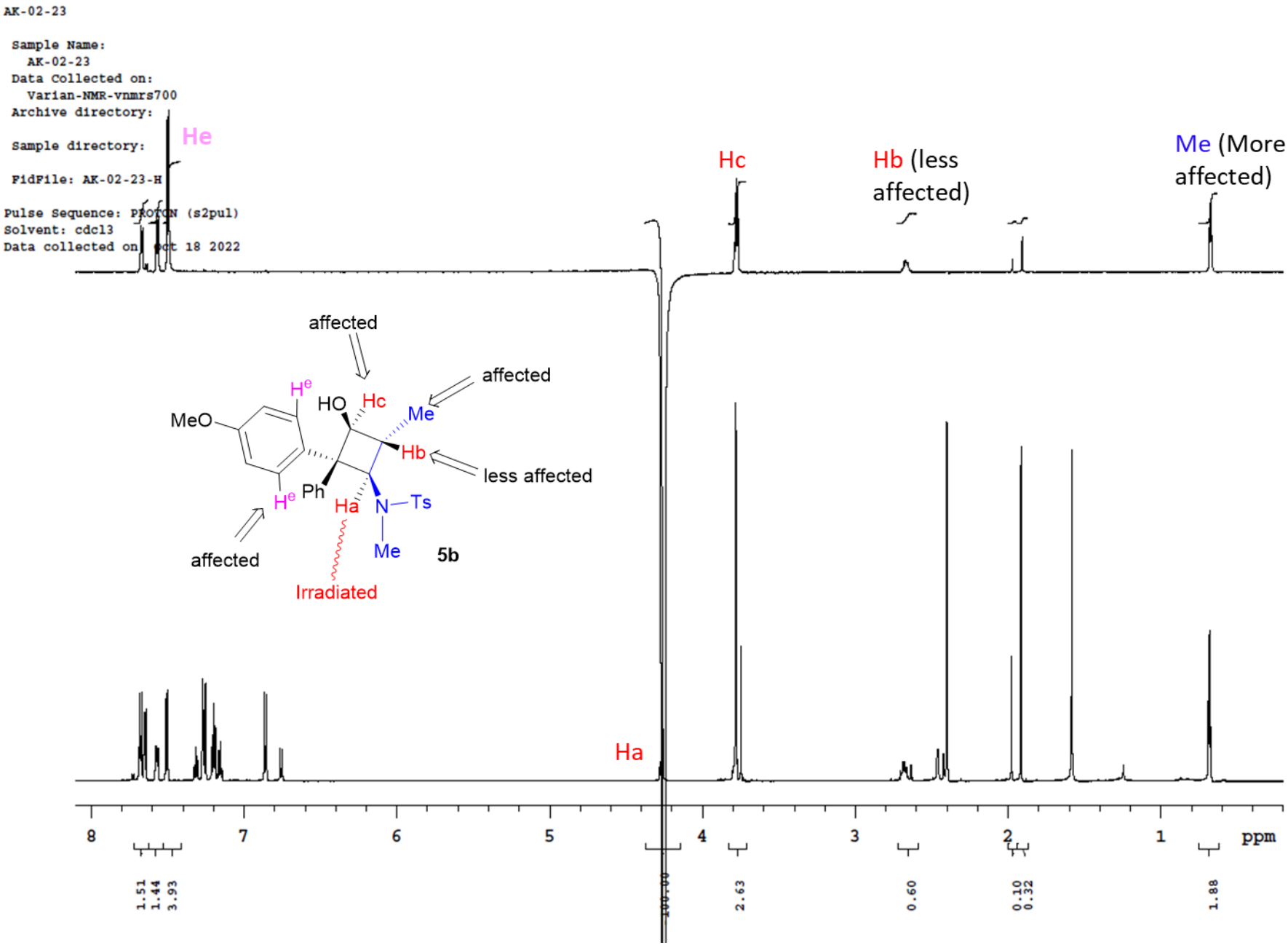
Sample Name:
AK-02-23
Data Collected on:
Varian-NMR-vnmrs700
Archive directory:

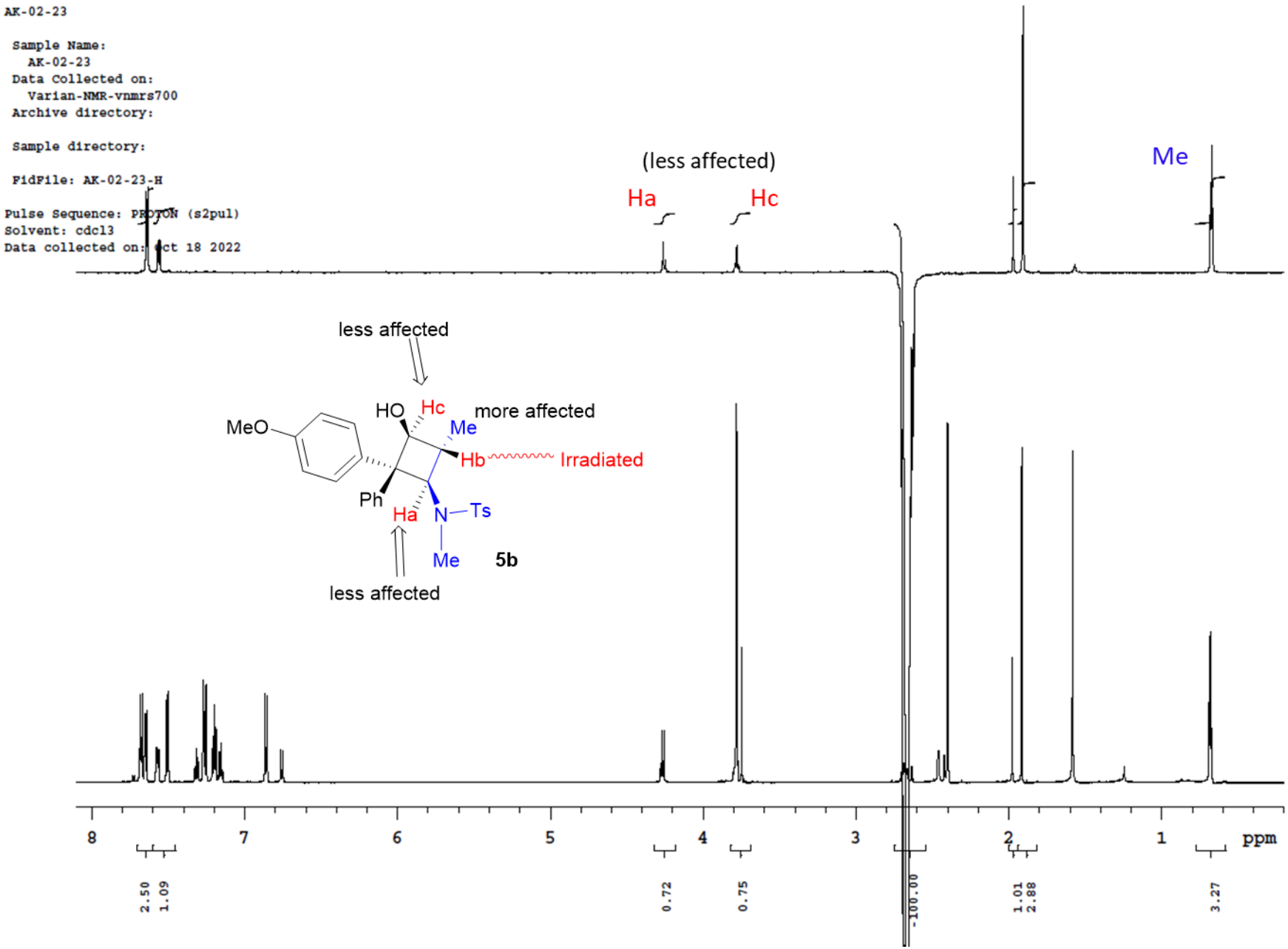
Sample directory:

FidFile: AK-02-23-H

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 18 2022




AK-02-23
Sample Name:
AK-02-23
Data Collected on:
Varian-NMR-vnmrs700
Archive directory:
Sample directory:
FidFile: AK-02-23-H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 18 2022
He
Hc
Hb (less affected)
Me (More affected)
affected
affected
affected
less affected
MeO
HO
Hc
Me
Hb
Ph
Ha
N
Ts
Me
Irradiated
5b
Ha
8
7
6
5
4
3
2
1
ppm
1.51
1.44
3.93
100.00
2.63
0.60
0.10
0.32
1.88


AK-02-23
Sample Name:
AK-02-23
Data Collected on:
Varian-NMR-vnmrs700
Archive directory:
Sample directory:
FidFile: AK-02-23-H
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 18 2022
(less affected)
Ha
Hc
Me
less affected
HO
Hc
more affected
Me
MeO
Hb
Irradiated
Ph
Ha
N
Ts
Me
5b
less affected
8
7
6
5
4
3
2
1
ppm
2.50
1.09
0.72
0.75
-100.00
1.01
2.88
3.27