Supporting Information for

Programmable divergent electrochemical ring-opening multifunctionalization of strained rings

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1. General Information

1.1 Analytic methods

^{1}H NMR, ^{13}C NMR, ^{19}F NMR spectra were recorded on Bruker Avance NEO 400 MHz NMR spectrometer and Bruker Avance NEO 500 MHz NMR spectrometer, with $CDCl_3$, MeOD-d_4 or DMSO-d_6 as solvent. ^{1}H NMR chemical shifts (in ppm) were referenced to $CHCl_3$ (δ = 7.26 ppm) in $CDCl_3$, DMSO (δ = 2.51 ppm) in DMSO-d_6, MeOH (δ = 3.31 ppm) in CD_3OD, or Me_4Si (δ = 0 ppm) as an internal standard. The data of ^{1}H NMR were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and br = broad), coupling constant (*J* values) in Hz and integration. ^{13}C NMR spectra were obtained by the same NMR spectrometers and were calibrated with $CDCl_3$ (δ = 77.00 ppm), DMSO-d_6 (δ = 39.52 ppm), CD_3OD (δ = 49.00 ppm). High-resolution mass spectra (HRMS) were recorded on a Waters G2-XS/APGC mass spectrometer. Gas Chromatography-Mass Spectrometry (GC-MS) was performed on a SHIMADZU GCMS-QP2010 SE system with SH-5MS capillary column (30 m × 0.25 mm × 0.25 μm). Flash column chromatography was performed with silica gel (200-300 mesh) supplied by Yantai Xin Nuo Chemical Co., LTD, eluting with ethyl acetate/petroleum ether. Thin-layer chromatography (TLC) were performed on Xin Nuo GF254 silica gel pre-coated plates (0.20-0.25 mm, purchased from Yantai Xin Nuo Chemical Co., LTD) and visualized with UV and phosphomolybdic acid unless otherwise noted.

1.2 Reagents

All commercially available compounds were purchased from Energy Chemical, Innochem, TCI, Adamas, Alfa-Aesar, Aladdin, Bidepharm, Energy Chemical, Leyan, Macklin, Meryer and Thermo Fisher Scientific. All the solvents and all the other reagents were directly used from purchased without any further purification unless otherwise specified.

2. Experimental Section

2.1 Reaction set-up and experimental procedures

2.1.1 Reaction set-up

Platinum plate (99.99%, 15 mm × 15 mm × 0.3 mm), other electrode materials (purchased from Cetech Co., Ltd or Wuhu Eryi Materials Technology Co., Ltd.) and electrode clamp (purchased from Gaoss Union or Xuzhou Xinke Instrument Co., Ltd.) were used for electrolysis experiments. Direct current (DC) electrolysis experiments were performed using the DC power supply (purchased from Beijing Hansheng Puyuan Technology Co., Ltd. or Shenzhen Maisi Taike Electronics Co., Ltd.). Alternating Current (AC) electrolysis experiments were performed using the Waveform Amplifier (purchased from Accel Instrument or Xi'an Antai Electronic Technology Co., Ltd.), 70 MHz Digital Oscilloscope (purchased from Beijing Hansheng Puyuan Technology Co., Ltd.) and DG811 Function/Arbitrary Waveform Generator (purchased from Beijing Hansheng Puyuan Technology Co., Ltd.).

The photoelectrochemical reactions were performed on Blue LEDs photoreactor. Blue LEDs photoreactor was consisted of a Blue LEDs strip (390-400 nm, 40 W Blue LEDs) and a bulb (390-400 nm, 40 W Blue LEDs) was purchased from Xuzhou Aijia Electronic.

The specific reaction set-up is illustrated in the diagram below (Figure **S1 - S4**).

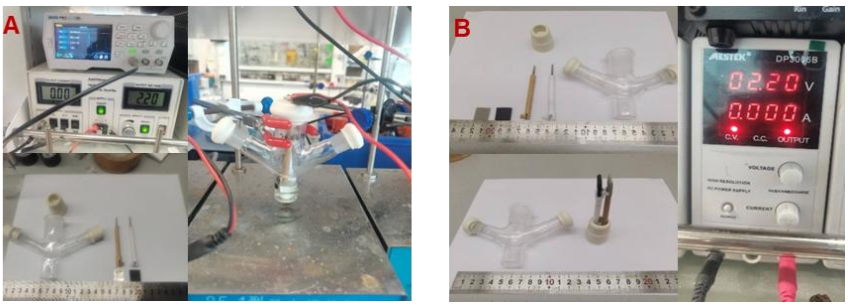


Figure S1. Reaction set-up. (A) Ring-opening trioxygenation of aminocyclopropanes (AC) (B) Ring-opening trioxygenation of arylcyclopropanes (DC)

Figure S2. Reaction set-up of ring-opening tetraoxygenation of aminocyclopropanes

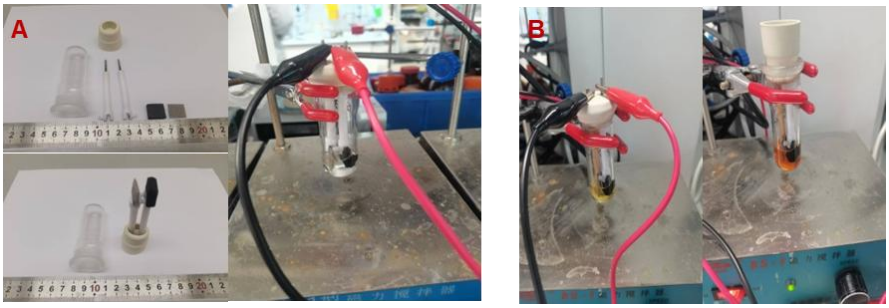
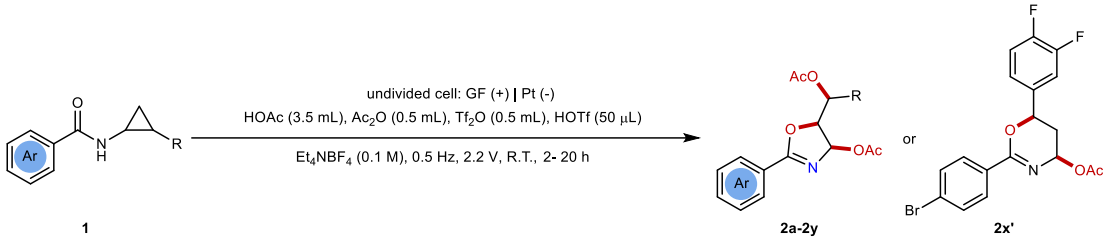


Figure S3. Reaction set-up. (A) Ring-opening trichlorohydroxylation of aminocyclopropanes (B) Ring-opening tribromohydroxylation of aminocyclopropanes

Figure S4. Reaction set-up of ring-opening tetraoxygenation of aminocyclobutanes (AC)

2.1.2 General experimental procedures

Reactions of site-selective ring-opening trioxygenation of aminocyclopropanes



General procedure A (**GP A**): For aminocyclopropanes, in a 10 mL oven-dried three-necked undivided cell equipped with a stir bar. To the cell was added aminocyclopropanes **1** (0.1 mmol), Et_4NBF_4 (98.8 mg, 0.1 M), HOAc (3.5 mL), Ac_2O (0.5 mL) and Tf_2O (0.5 mL). The mixture was stirred for 1 min, and then HOTf (50 μL) was carefully added. The undivided cell was equipped with a graphite felt anode (15 mm × 15 mm × 3 mm) in the positive half-period and platinum cathode (15 mm × 15 mm × 0.3 mm) in the negative half-period. Mixtures were stirred at room temperature and electrolyzed at an alternating voltage of 2.2 V (square wave, 0.5 Hz, duty ratio (D) = 80%, OFFSET = 0.8 V) for time. After completion of the reaction as monitored by TLC (usually 4 h), the reaction mixture was poured into a saturated sodium carbonate solution (ca. 20 mL). The graphite felt anode was washed with EtOAc (3×5 mL) and these washes were added to the reaction mixture. The aqueous layer was separated and extracted with EtOAc (3×15 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration in vacuo, the crude product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 5:1 - 2:1) to afford target product **2**.

When testing various anode materials (Figure S5), it was observed that product yields under alternating current (AC) conditions consistently exceeded those under direct current (DC). Experimental observations revealed varying degrees of anode degradation in DC configurations (especially 0.5 mm graphite, Figure S6), demonstrating that alternating current plays a critical role in this reaction.

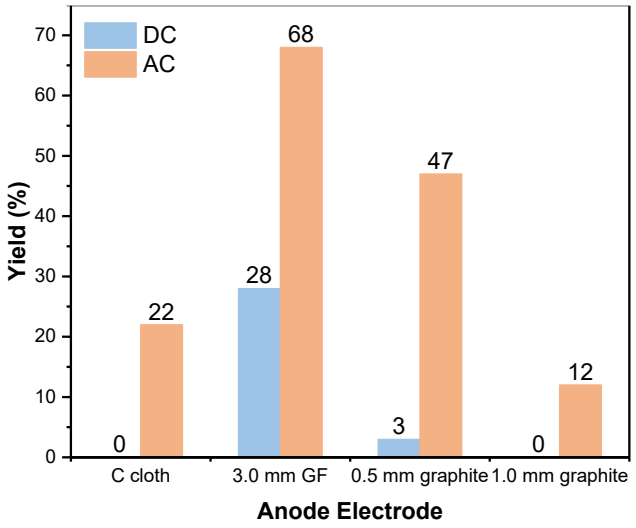


Figure S5. The impact of different anode electrodes on product yield

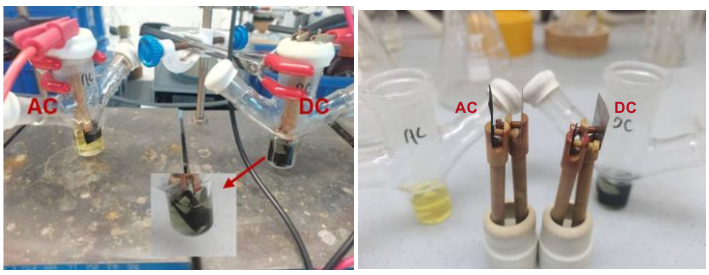
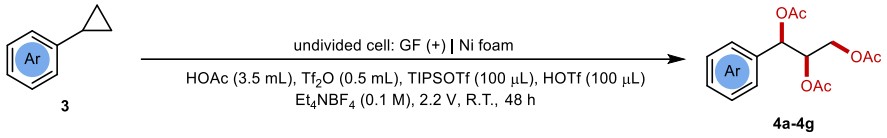


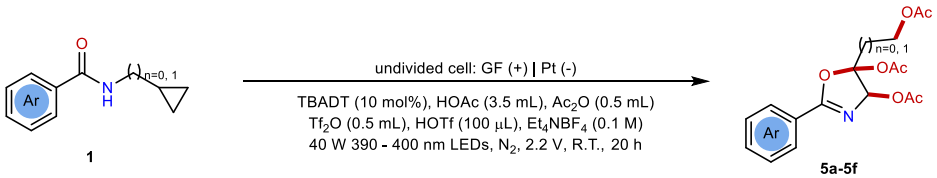
Figure S6. The anode graphite electrode detached under DC conditions (2 h)

Reactions of site-selective ring-opening trioxygenation of arylcyclopropanes



General procedure B (**GP B**): For arylcyclopropanes, in a 10 mL oven-dried three-necked undivided cell equipped with a stir bar. The undivided cell was equipped with a graphite felt anode (15 mm × 15 mm × 3 mm) and Ni foam cathode (15 mm × 15 mm × 3 mm). To the cell was added arylcyclopropanes **3** (0.3 mmol), Et_4NBF_4 (91.1 mg, 0.1 M), HOAc (3.5 mL), Tf_2O (0.5 mL) and TIPSOTf (100 μL). The mixture was stirred for 1 min, and then HOTf (100 μL) was carefully added. The cell was sealed using a rubber septum and parafilm. Mixtures were stirred at room temperature and electrolyzed at a controlled voltage of 2.2 V for 48 h under air. After completion of the reaction as monitored by GC-MS (usually 48 h), the reaction mixture was poured into a saturated sodium carbonate solution (ca. 20 mL). The graphite felt anode was washed with EtOAc (3×5 mL) and these washes were added to the reaction mixture. The aqueous layer was separated and extracted with EtOAc (3×15 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration in vacuo, the crude product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 6:1) to afford target product **4**.

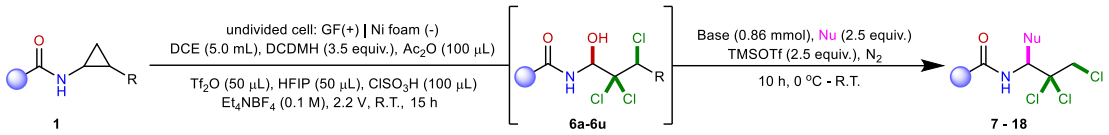
Reactions of site-selective ring-opening tetraoxygenation of aminocyclopropanes



General procedure C (**GP C**): For aminocyclopropanes, in a 20 mL oven-dried undivided cell equipped with a stir bar. The undivided tube was equipped with a graphite felt anode (15 mm × 15 mm × 3 mm) and platinum cathode (15 mm × 15 mm × 0.3 mm). To the cell was added aminocyclopropanes **1** (0.1 mmol), TBADT (10 mol%), Et_4NBF_4 (99.8 mg, 0.1 M), HOAc (3.5 mL), Ac_2O (0.5 mL) and Tf_2O (0.5 mL). The mixture was stirred for 1 min, and then HOTf (100 μL) was carefully added. The cell was sealed using a rubber septum and parafilm, and was then flushed with

nitrogen gas for 5 min. The reaction tube was suspended 2.0 cm above the 390 - 400 nm, 40 W LEDs. Mixtures were stirred at room temperature and electrolyzed at a controlled voltage of 2.2 V for 20 h. After completion of the reaction as monitored by GC-MS (usually 20 h), the reaction mixture was poured into a saturated sodium carbonate solution (ca. 20 mL). The graphite felt anode was washed with EtOAc (3×5 mL) and these washes were added to the reaction mixture. The aqueous layer was separated and extracted with EtOAc (3×15 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration in vacuo, the crude product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 4:1) to afford target product **5**.

Reactions of site-selective ring-opening trichlorohydroxylation of aminocyclopropanes



General procedure D (**GP D**): For aminocyclopropanes, in a 20 mL oven-dried undivided cell equipped with a stir bar. The undivided tube was equipped with a graphite felt anode (15 mm × 15 mm × 6.5 mm) and Ni foam cathode (15 mm × 15 mm × 3 mm). To the cell was added aminocyclopropanes **1** (0.1 mmol), Et_4NBF_4 (115.7 mg, 0.1 M), 1,3-dichloro-5,5-dimethylimidazolidine-2,4-dione (DCDMH, 3.5 equiv.), DCE (5.0 mL), Ac_2O (100 μL), Tf_2O (50 μL) and HFIP (50 μL). The mixture was stirred for 1 min, and then $ClSO_3H$ (100 μL) was carefully added. Mixtures were stirred at room temperature and electrolyzed at a controlled voltage of 2.2 V for 15 h. After completion of the reaction as monitored by TLC (usually 15 h), the reaction mixture was poured into a saturated sodium carbonate solution (ca. 20 mL). The graphite felt anode was washed with EtOAc (3×5 mL) and these washes were added to the reaction mixture. The aqueous layer was separated and extracted with EtOAc (3×15 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration in vacuo, the crude product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 4:1 - 3:1) to afford target product **6**.

General procedure E (**GP E**): For aminocyclopropanes, in a 20 mL oven-dried undivided cell equipped with a stir bar. The undivided tube was equipped with a graphite felt anode (15 mm × 15 mm × 6.5 mm) and Ni foam cathode (15 mm × 15 mm × 3 mm). To the cell was added aminocyclopropanes **1** (0.1 mmol), Et_4NBF_4 (115.7 mg, 0.1 M), DCDMH (3.5 equiv.), DCE (5.0 mL), Ac_2O (100 μL), Tf_2O (50 μL) and HFIP (50 μL). The mixture was stirred for 1 min, and then $ClSO_3H$ (100 μL) was carefully added. Mixtures were stirred at room temperature and electrolyzed at a controlled voltage of 2.2 V for 15 h.

After completion of the reaction, the reaction mixture was cooled to 0 °C. To the solution was slowly added base (0.86 mmol), TMSOTf (2.5 equiv.) followed by the coupling partner (2.5 equiv.). The reaction mixture was gradually warmed to room temperature and further stirred at room temperature for another 10 h without electricity. After completion of the reaction as monitored by TLC (usually 10 h), the reaction mixture was poured into a saturated sodium carbonate solution (ca. 20 mL). The graphite felt anode was washed with EtOAc (3×5 mL) and these washes were added to the reaction mixture. The aqueous layer was separated and extracted with EtOAc (3×15 mL), and the combined

organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration in vacuo, the crude product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 5:1 - 1:1) to afford target product **7** - **18**.

Gram-scale reaction for ring-opening trichlorohydroxylation of aminocyclopropanes

In a 120 mL oven-dried undivided cell (Figure S7) equipped with a stir bar. The undivided tube was equipped with a graphite felt anode (40 mm × 35 mm × 6.5 mm) and Ni foam cathode (40 mm × 35 mm × 3 mm). To the cell was added *N*-cyclopropylbenzamide **1d** (1.8 mmol), Et_4NBF_4 (1.7 g, 0.1 M), DCDMH (1.24 g, 3.5 equiv.), DCE (75.0 mL), Ac_2O (0.18 mL), Tf_2O (0.9 mL) and HFIP (0.9 mL). The mixture was stirred for 1 min, and then $ClSO_3H$ (1.8 mL) was carefully added. Mixtures were stirred at room temperature and electrolyzed at a controlled voltage of 2.2 V for 20 h. After completion of the reaction as monitored by TLC, the reaction mixture was poured into a saturated sodium carbonate solution (ca. 100 mL). The graphite felt anode was washed with DCM (3×25 mL) and these washes were added to the reaction mixture. The aqueous layer was separated and extracted with DCM (3×25 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Upon completion, the mixture was filtered and concentrated in vacuo. **6d** was obtained by flash silica column chromatography (petroleum ether/ethyl acetate = 4:1) as a white solid (432.8 mg, 86% yield).

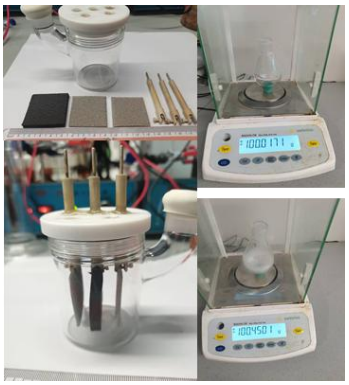
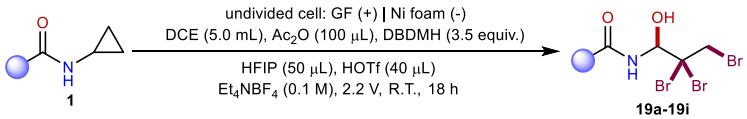


Figure S7. Reaction set-up of gram-scale reaction

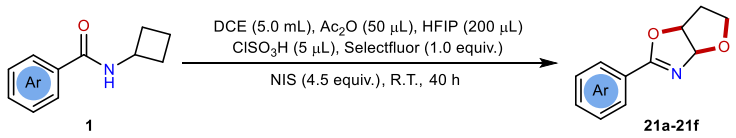
Gram-scale reaction for ring-opening tribromohydroxylation of aminocyclopropanes



General procedure F (**GP F**): For aminocyclopropanes, in a 20 mL oven-dried undivided cell equipped with a stir bar. The undivided tube was equipped with a graphite felt anode (15 mm × 15 mm × 6.5 mm) and Ni foam cathode (15 mm × 15 mm × 3 mm). To the cell was added Et_4NBF_4 (112.6 mg, 0.1 M), 1,3-dibromo-5,5-dimethylimidazolidine-2,4-dione (DBDMH, 3.5 equiv.), DCE (5.0 mL), Ac_2O (100 μL), HFIP (50 μL) and then HOTf (40 μL) was carefully added. Mixtures were stirred at room temperature and electrolyzed at a controlled voltage of 2.2 V for 3 h. To the solution

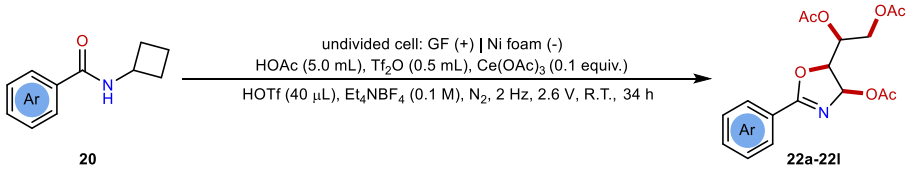
was slowly added aminocyclopropanes **1** (0.1 mmol) and further stirred at room temperature for another 20 h without electricity. After completion of the reaction as monitored by TLC (usually 20 h), the reaction mixture was poured into a saturated sodium carbonate solution (ca. 20 mL). The graphite felt anode was washed with EtOAc (3×5 mL) and these washes were added to the reaction mixture. The aqueous layer was separated and extracted with EtOAc (3×15 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration in vacuo, the crude product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 4:1 - 3:1) to afford target product **19**.

Reactions of site-selective ring-opening trioxygenation of aminocyclobutanes



General procedure G (**GP G**): For aminocyclobutanes, in a 20 mL oven-dried tube equipped with a stir bar. To the cell was added aminocyclobutanes **1** (0.05 mmol), DCE (5.0 mL), Ac_2O (50 μL), HFIP (200 μL) and $ClSO_3H$ (5 μL). NIS (4.5 equiv.) and Selectfluor (35.4 mg, 1.0 equiv.) were carefully added in 2 times interval of 20 h. Mixtures were stirred at room temperature for 40 h. After completion of the reaction as monitored by TLC (usually 40 h), the reaction mixture was poured into a saturated sodium thiosulfate solution (ca. 20 mL). The aqueous layer was separated and extracted with EtOAc (3×15 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration in vacuo, the crude product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 2:1) to afford target product **21**.

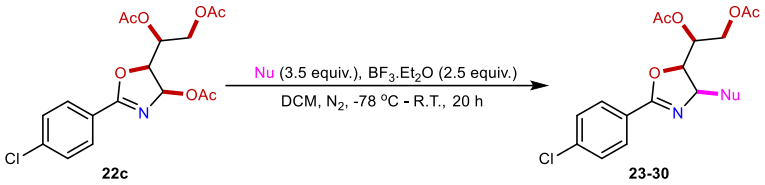
Reactions of site-selective ring-opening tetraoxygenation of aminocyclobutanes



General procedure H (**GP H**): For aminocyclobutanes, in a 10 mL oven-dried undivided cell equipped with a stir bar. To the cell was added aminocyclobutanes **20** (0.2 mmol), Et_4NBF_4 (120.2 mg, 0.1 M), HOAc (5.0 mL), Tf_2O (0.5 mL) and $Ce(OAc)_3$ (0.1 equiv.). The mixture was stirred for 1 min, and then HOTf (40 μL) was carefully added. The undivided tube was equipped with a graphite felt anode (15 mm × 15 mm × 6.5 mm) in the positive half-period and Ni foam (15 mm × 15 mm × 3 mm) in the negative half-period. The cell was sealed using a rubber septum and parafilm and was then flushed with nitrogen gas for 5 min. Mixtures were stirred at room temperature and electrolyzed at an alternating voltage of 2.6 V (square wave, 2 Hz, duty ratio (D) = 80%, OFFSET = 0.8 V) for 34 h. After completion of the reaction as monitored by TLC (usually 34 h), the reaction mixture was poured into a saturated sodium carbonate solution (ca. 20 mL). The graphite felt anode

was washed with EtOAc (3×5 mL) and these washes were added to the reaction mixture. The aqueous layer was separated and extracted with EtOAc (3×15 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration in vacuo, the crude product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1 - 2:1) to afford target product **22**.

*Derivatization of **22c***



General procedure I (**GP I**): In a 20 mL oven-dried tube equipped with a stir bar. To the cell was added tetraoxygenation product **22c** (0.1 mmol), DCM (2.0 mL). The cell was sealed using a rubber septum and parafilm and was then flushed with nitrogen gas for 5 min. The reaction mixture was cooled to -78 °C. To the solution was slowly added $BF_3 \cdot Et_2O$ (2.5 equiv.) followed by the coupling partner (3.5 equiv.). The reaction mixture was gradually warmed to room temperature and further stirred at room temperature for 20 h. After completion of the reaction as monitored by TLC (usually 20 h), the reaction mixture was poured into a saturated brine solution (ca. 20 mL). The aqueous layer was separated and extracted with DCM (3×15 mL), and the combined organic layers dried over anhydrous Na_2SO_4. Following concentration in vacuo, the crude product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 5:1 - 2:1) to afford target product **23** - **30**.

2.2 Scope of substrates

Substrate preparation: Most of substrates used were known compounds[1-12] and new substrates were synthesized according to previous literature[1].

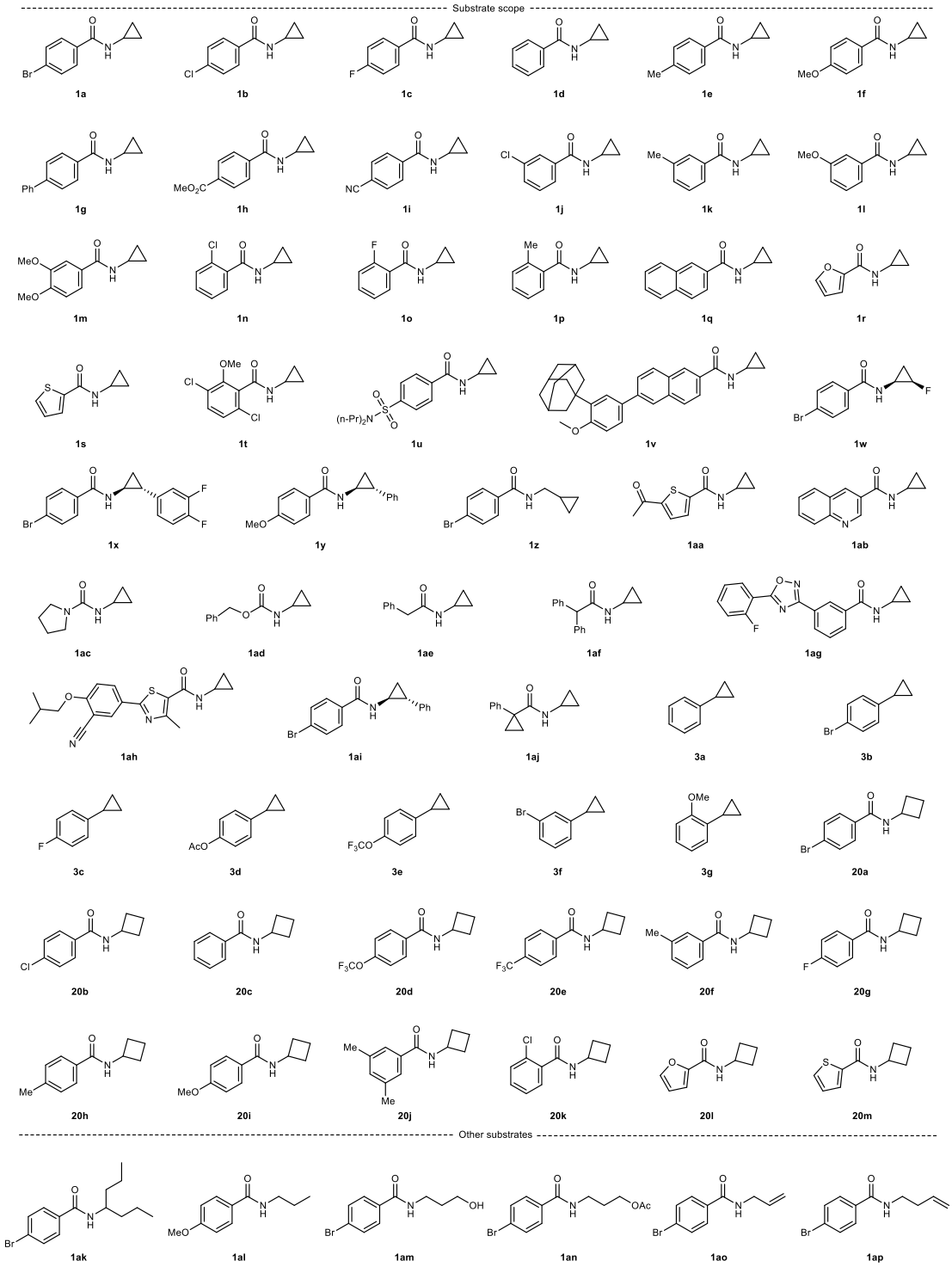
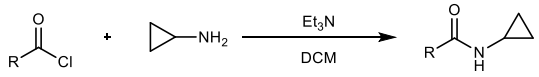
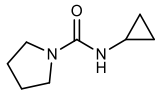


Figure S8. Substituted cyclopropanes/cyclobutanes or *N*-alkylamides



General procedure J (GP J): Following the reported procedure[1], to a solution of cyclopropylamine (0.76 mL, 11.0 mmol, 1.1 equiv.) and triethylamine (1.5 mL, 11.0 mmol, 1.1 equiv.) in DCM (10.0 mL) was slowly added a solution of acyl chloride (10.0 mmol, 1.0 equiv.) in DCM (10.0 mL) at 0 °C. The reaction mixture was stirred at room temperature for 24 h. Upon completion, the mixture was quenched by the addition of 1 M HCl (10 mL). The aqueous layer was then extracted with dichloromethane. The organic extract was washed with 1 M NaOH (10 mL) and brine (10 mL), dried over Na_2SO_4, filtered and concentrated in vacuo. In most cases, the crude product was pure enough to be used as such, without further purification. Compounds **1a-1s**, **1ac-1af** were prepared following **GP J**.



1ac

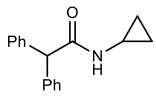
***N*-cyclopropylpyrrolidine-1-carboxamide (1ac):**

Following **GP J**, using pyrrolidine-1-carbonyl chloride (1.3 g, 10.0 mmol) and cyclopropylamine (0.76 mL, 11.0 mmol), **1ac** was obtained as a white solid (1.4 g, 88% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 4.47 (s, 1H), 3.30 – 3.23 (m, 4H), 2.65 – 2.58 (m, 1H), 1.91 – 1.81 (m, 4H), 0.71 – 0.65 (m, 2H), 0.47 – 0.42 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 157.6, 45.4, 25.5, 23.2, 6.8.

HRMS: calc. for $C_8H_{15}N_2O^+$ $(M+H)^+$, 155.1179, found, 155.1191.



1af

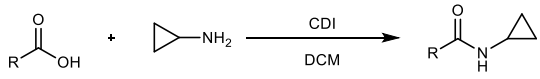
***N*-cyclopropyl-2,2-diphenylacetamide (1af):**

Following **GP J**, using 2,2-diphenylacetyl chloride (2.3 g, 10.0 mmol) and cyclopropylamine (0.76 mL, 11.0 mmol), **1af** was obtained as a white solid (2.1 g, 85% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.33 – 7.28 (m, 5H), 7.25 – 7.21 (m, 5H), 5.72 (s, 1H), 4.86 (s, 1H), 2.78 – 2.70 (m, 1H), 0.77 – 0.71 (m, 2H), 0.46 – 0.40 (m, 2H).

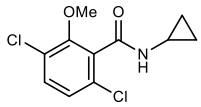
^{13}C NMR (126 MHz, Chloroform-*d*) δ 173.2, 139.4, 128.8, 128.7, 127.2, 58.9, 22.9, 6.7.

HRMS: calc. for $C_{17}H_{17}NNaO^+$ $(M+Na)^+$, 274.1202, found, 274.1203.



General procedure K (GP K): Following the reported procedure[1], carboxylic acid (10.0 mmol, 1.0 equiv.) and CDI (1.6, 10.0 mmol, 1.0 equiv.) were suspended in DCM (50.0 mL). The resulting mixture was stirred for 2 h before adding cyclopropylamine (0.76 mL, 11.0 mmol, 1.1 equiv.). The reaction mixture was stirred at room temperature for another 24 h. Upon completion, the mixture was quenched by addition of 1 M NaOH (2×25 mL). The aqueous layer was then extracted with dichloromethane. The organic layer was washed with 1 M HCl (50 mL). The combined organic layers were washed with brine (50 mL), dried over Na_2SO_4, filtered and concentrated in vacuo. The

crude product was purified by flash column chromatography on silica gel to afford the desired product. Compounds **1t - 1v**, **1aa**, **1ab**, **1ag**, **1ah, 1aj** were prepared following **GP K**.



1t

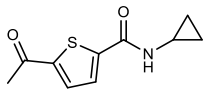
3,6-dichloro-*N*-cyclopropyl-2-methoxybenzamide (1t):

Following **GP K**, using 3,6-dichloro-2-methoxybenzoic acid (2.2 g, 10.0 mmol) and cyclopropylamine (0.76 mL, 11.0 mmol), **1t** was obtained as a white solid (1.3 g, 50% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.27 (d, *J* = 8.6 Hz, 1H), 7.05 (d, *J* = 8.6 Hz, 1H), 6.09 (s, 1H), 3.88 (s, 3H), 2.91 – 2.84 (m, 1H), 0.91 – 0.82 (m, 2H), 0.68 – 0.60 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 165.3, 153.6, 132.9, 131.3, 130.0, 126.7, 126.0, 62.6, 22.9, 6.7.

HRMS: calc. for $C_{11}H_{12}Cl_2NO_2^+$ $(M+H)^+$, 260.0240, found, 260.0251.



1aa

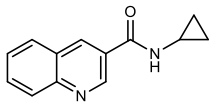
5-acetyl-*N*-cyclopropylthiophene-2-carboxamide (1aa):

Following **GP K**, using 5-acetylthiophene-2-carboxylic acid (1.7 g, 10.0 mmol) and cyclopropylamine (0.76 mL, 11.0 mmol), **1aa** was obtained as a white solid (1.2 g, 57% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.60 (d, *J* = 3.9 Hz, 1H), 7.50 (d, *J* = 4.0 Hz, 1H), 6.46 (s, 1H), 2.90 – 2.84 (m, 1H), 2.56 (s, 3H), 0.90 – 0.82 (m, 2H), 0.69 – 0.60 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 190.7, 162.4, 146.6, 144.9, 132.0, 128.8, 26.9, 23.3, 6.8.

HRMS: calc. for $C_{10}H_{11}NNaO_2S^+$ $(M+Na)^+$, 232.0403, found, 232.0412.



1ab

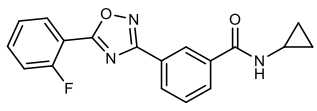
***N*-cyclopropylquinoline-3-carboxamide (1ab):**

Following **GP K**, using quinoline-3-carboxylic acid (1.7 g, 10.0 mmol) and cyclopropylamine (0.76 mL, 11.0 mmol), **1ab** was obtained as a white solid (1.1 g, 53% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 9.22 (d, *J* = 2.2 Hz, 1H), 8.55 (d, *J* = 2.2 Hz, 1H), 8.11 (d, *J* = 8.5 Hz, 1H), 7.87 – 7.82 (m, 1H), 7.80 – 7.75 (m, 1H), 7.61 – 7.55 (m, 1H), 6.71 (s, 1H), 3.01 – 2.92 (m, 1H), 0.95 – 0.87 (m, 2H), 0.73 – 0.66 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.1, 149.2, 148.0, 135.5, 131.2, 129.3, 128.7, 127.5, 126.9, 126.8, 23.3, 6.8.

HRMS: calc. for $C_{13}H_{12}N_2NaO^+$ $(M+Na)^+$, 235.0842, found, 235.0843.

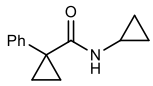


1ag

***N*-cyclopropyl-3-(5-(2-fluorophenyl)-1,2,4-oxadiazol-3-yl)benzamide (1ag):**

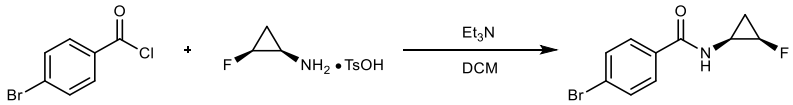
Following **GP K**, using 3-(5-(2-fluorophenyl)-1,2,4-oxadiazol-3-yl)benzoic acid (2.8 g, 10.0 mmol)

and cyclopropylamine (0.76 mL, 11.0 mmol), **1ag** was obtained as a white solid (2.2 g, 68% yield).
^{1}H NMR (500 MHz, DMSO-d_6) δ 8.71 (d, J = 4.2 Hz, 1H), 8.53 (s, 1H), 8.28– 8.23 (m, 1H), 8.23 – 8.20 (m, 1H), 8.07 – 8.03 (m, 1H), 7.84 – 7.76 (m, 1H), 7.68 (t, J = 7.7 Hz, 1H), 7.59 – 7.53 (m, 1H), 7.52 – 7.48 (m, 1H), 2.94 – 2.85 (m, 1H), 0.75 – 0.70 (m, 2H), 0.64 – 0.59 (m, 2H).
^{13}C NMR (126 MHz, DMSO-d_6) δ 172.8 (d, J_{CF} = 4.1 Hz), 167.8, 166.7, 160.1 (d, J_{CF} = 257.8 Hz), 135.9 (d, J_{CF} = 8.9 Hz), 135.5, 131.0, 130.4, 129.7, 129.4, 126.12 (d, J_{CF} = 10.9 Hz), 126.07, 125.6 (d, J_{CF} = 3.6 Hz), 117.4 (d, J_{CF} = 20.6 Hz), 111.8 (d, J_{CF} = 11.4 Hz), 23.3, 5.8.
^{19}F NMR (471 MHz, DMSO-d_6) δ -109.4.
HRMS: calc. for $C_{18}H_{14}FN_3NaO_2^+$ $(M+Na)^+$, 346.0962, found, 346.0967.

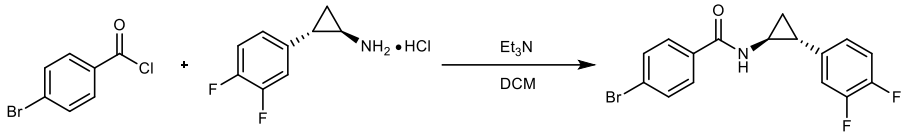


1aj

***N*-cyclopropyl-1-phenylcyclopropane-1-carboxamide (1aj):**
Following **GP K**, using 1-phenylcyclopropane-1-carboxylic acid (1.6 g, 10.0 mmol) and cyclopropylamine (0.76 mL, 11.0 mmol), **1aj** was obtained as a white solid (1.6 g, 80% yield).
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.37 – 7.32 (m, 4H), 7.31 – 7.27 (m, 1H), 5.34 (s, 1H), 2.65 – 2.57 (m, 1H), 1.59 (q, J = 3.6 Hz, 2H), 1.01 (q, J = 3.7 Hz, 2H), 0.68 – 0.63 (m, 2H), 0.33 – 0.28 (m, 2H).
^{13}C NMR (126 MHz, Chloroform-*d*) δ 175.2, 139.8, 130.9, 128.9, 127.8, 30.3, 23.0, 15.5, 6.5.
HRMS: calc. for $C_{13}H_{15}NNaO^+$ $(M+Na)^+$, 224.1046, found, 224.1058.



4-bromo-*N*-(2-fluorocyclopropyl)benzamide (1w):
Following the **GP J**, using 4-bromobenzoyl chloride (2.2 g, 10.0 mmol) and (1*R*,2*S*)-2-fluorocyclopropanamine 4-methylbenzenesulfonate (3.7 g, 15.0 mmol), **1w** was obtained as a white solid (1.77 g, 69% yield).
To a solution of (1*R*,2*S*)-2-fluorocyclopropanamine 4-methylbenzenesulfonate (3.7 g, 15.0 mmol) and triethylamine (1.5 mL, 11.0 mmol, 1.1 equiv.) in DCM (10.0 mL) was slowly added a solution of 4-bromobenzoyl chloride (2.2 g, 10.0 mmol) in DCM (10.0 mL) at 0 °C. The reaction mixture was stirred at room temperature for 24 h. Upon completion, the mixture was quenched by the addition of 1 M HCl (10 mL). The aqueous layer was then extracted with dichloromethane. The organic extract was washed with 1 M NaOH (10 mL) and brine (10 mL), dried over Na_2SO_4, filtered and concentrated in vacuo. **1w** was obtained by flash silica column chromatography (petroleum ether/ethyl acetate = 5:1) as a white solid.
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.64 (d, J = 8.5 Hz, 2H), 7.55 (d, J = 8.6 Hz, 2H), 6.40 (s, 1H), 4.84 – 4.64 (m, 1H), 3.04 – 2.96 (m, 1H), 1.28 – 1.19 (m, 1H), 1.08 – 0.96 (m, 1H).
^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.9, 132.7, 131.8, 128.6, 126.4, 70.0 (d, J_{CF} = 221.1 Hz), 25.8 (d, J_{CF} = 9.3 Hz), 13.3 (d, J_{CF} = 9.6 Hz).
^{19}F NMR (471 MHz, Chloroform-*d*) δ -210.0.
HRMS: calc. for $C_{10}H_9BrFNNaO^+$ $(M+Na)^+$, 279.9744, found, 279.9741.



4-bromo-*N*-(2-(3,4-difluorophenyl)cyclopropyl)benzamide (1x):

Following the **GP J**, using 4-bromobenzoyl chloride (2.2 g, 10.0 mmol) and (1*R*,2*S*)-2-(3,4-difluorophenyl)cyclopropan-1-amine hydrochloride (2.3 g, 11.0 mmol), **1x** was obtained as a white solid (2.5 g, 70% yield).

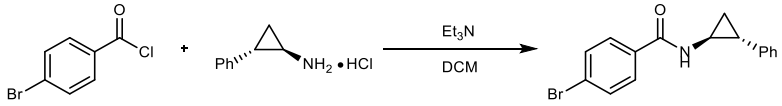
To a solution of (1*R*,2*S*)-2-(3,4-difluorophenyl)cyclopropan-1-amine hydrochloride (2.3 g, 11.0 mmol) and triethylamine (1.5 mL, 11.0 mmol, 1.1 equiv.) in DCM (10.0 mL) was slowly added a solution of 4-bromobenzoyl chloride (2.2 g, 10.0 mmol) in DCM (10.0 mL) at 0 °C. The reaction mixture was stirred at room temperature for 24 h. Upon completion, the mixture was quenched by the addition of 1 M HCl (10 mL). The aqueous layer was then extracted with dichloromethane. The organic extract was washed with 1 M NaOH (10 mL) and brine (10 mL), dried over Na_2SO_4, filtered and concentrated in vacuo. **1x** was obtained by flash silica column chromatography (petroleum ether/ethyl acetate = 5:1) as a white solid.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.63 (d, *J* = 8.3 Hz, 2H), 7.56 (d, *J* = 8.4 Hz, 2H), 7.10 – 7.00 (m, 2H), 6.98 – 6.94 (m, 1H), 6.45 (s, 1H), 2.99 – 2.91 (m, 1H), 2.17 – 2.08 (m, 1H), 1.29 – 1.24 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.7, 150.2 (dd, J_{CF} = 248.1, 12.7 Hz), 149.1 (dd, J_{CF} = 246.4, 12.5 Hz), 137.2 (dd, J_{CF} = 5.4, 3.8 Hz), 132.8, 131.9, 128.5, 126.5, 122.9 (dd, J_{CF} = 6.0, 3.4 Hz), 117.1 (d, J_{CF} = 17.2 Hz), 115.9 (d, J_{CF} = 17.4 Hz), 32.3, 24.5, 15.5.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -138.0 (d, *J* = 21.0 Hz), -141.3 (d, *J* = 21.0 Hz).

HRMS: calc. for $C_{16}H_{13}BrF_2NO^+$ $(M+H)^+$, 352.0143, found, 352.0145.



4-bromo-*N*-(2-phenylcyclopropyl)benzamide (1ai):

Following the **GP J**, using 4-bromobenzoyl chloride (2.2 g, 10.0 mmol) and (1*R*,2*S*)-2-phenylcyclopropan-1-amine hydrochloride (1.9 g, 11.0 mmol), **1ai** was obtained as a white solid (2.4 g, 76% yield).

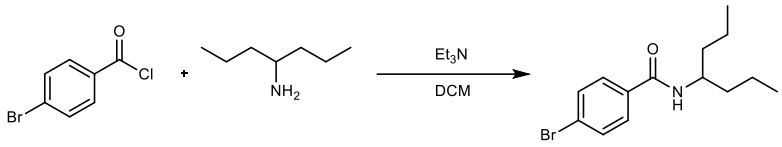
To a solution of (1*R*,2*S*)-2-phenylcyclopropan-1-amine hydrochloride (1.9 g, 11.0 mmol) and triethylamine (1.5 mL, 11.0 mmol, 1.1 equiv.) in DCM (10.0 mL) was slowly added a solution of 4–bromobenzoyl chloride (2.2 g, 10.0 mmol) in DCM (10.0 mL) at 0 °C. The reaction mixture was stirred at room temperature for 24 h. Upon completion, the mixture was quenched by the addition of 1 M HCl (10 mL). The aqueous layer was then extracted with dichloromethane. The organic extract was washed with 1 M NaOH (10 mL) and brine (10 mL), dried over Na_2SO_4, filtered and concentrated in vacuo. **1ai** was obtained by flash silica column chromatography (petroleum ether/ethyl acetate = 5:1) as a white solid.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.63 (d, *J* = 8.6 Hz, 2H), 7.54 (d, *J* = 8.5 Hz, 2H), 7.28 (t, *J* = 7.6 Hz, 2H), 7.21 – 7.15 (m, 3H), 6.58 (s, 1H), 3.11 – 3.01 (m, 1H), 2.20 – 2.13 (m, 1H), 1.35 – 1.29 (m, 1H), 1.29 – 1.23 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.6, 140.2, 133.0, 131.8, 128.5, 128.4, 126.5, 126.3, 126.2,

32.5, 24.9, 16.2.

HRMS: calc. for $C_{16}H_{15}BrNO^+$ $(M+H)^+$, 316.0332, found, 316.0333.



4-bromo-*N*-(heptan-4-yl)benzamide (1ak):

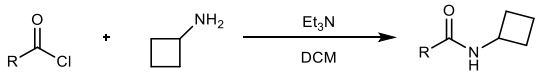
Following the **GP J**, using 4-bromobenzoyl chloride (2.2 g, 10.0 mmol) and heptan-4-amine (1.65 mL, 11.0 mmol, 1.1 equiv.), **1ak** was obtained as a white solid (2.6 g, 88% yield).

To a solution of heptan-4-amine (1.65 mL, 11.0 mmol, 1.1 equiv.) and triethylamine (1.5 mL, 11.0 mmol, 1.1 equiv.) in DCM (10.0 mL) was slowly added a solution of 4-bromobenzoyl chloride (2.2 g, 10.0 mmol) in DCM (10.0 mL) at 0 °C. The reaction mixture was stirred at room temperature for 24 h. Upon completion, the mixture was quenched by the addition of 1 M HCl (10 mL). The aqueous layer was then extracted with dichloromethane. The organic extract was washed with 1 M NaOH (10 mL) and brine (10 mL), dried over Na_2SO_4, filtered and concentrated in vacuo. **1ak** was obtained by flash silica column chromatography (petroleum ether/ethyl acetate = 5:1) as a white solid.

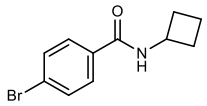
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.61 (d, *J* = 8.1 Hz, 2H), 7.53 (d, *J* = 8.1 Hz, 2H), 5.89 (d, *J* = 9.2 Hz, 1H), 4.28 – 3.91 (m, 1H), 1.60 – 1.51 (m, 2H), 1.60 – 1.50 (m, 6H), 0.91 (t, *J* = 7.2 Hz, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 166.1, 133.9, 131.7, 128.4, 125.8, 49.5, 37.5, 19.2, 14.0.

HRMS: calc. for $C_{14}H_{21}BrNO^+$ $(M+H)^+$, 298.0801, found, 298.0808.



General procedure L (GP L): Following the reported procedure[1], to a solution of cyclobutanamine (0.94 mL, 11.0 mmol, 1.1 equiv.) and triethylamine (1.5 mL, 11.0 mmol, 1.1 equiv.) in DCM (10.0 mL) was slowly added a solution of acyl chloride (10.0 mmol, 1.0 equiv.) in DCM (10.0 mL) at 0 °C. The reaction mixture was stirred at room temperature for 24 h. Upon completion, the mixture was quenched by the addition of 1 M HCl (10 mL). The aqueous layer was then extracted with dichloromethane. The organic extract was washed with 1 M NaOH (10 mL) and brine (10 mL), dried over Na_2SO_4, filtered and concentrated in vacuo. In most cases, the crude product was pure enough to be used as such, without further purification. Compounds **20a - 20m** were prepared following **GP L**.



20a

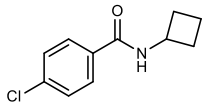
4-bromo-*N*-cyclobutylbenzamide (20a):

Following **GP L**, using 4-bromobenzoyl chloride (2.2 g, 10.0 mmol) and cyclobutanamine (0.94 mL, 11.0 mmol), **20a** was obtained as a white solid (2.4 g, 93% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.64 – 7.60 (m, 2H), 7.56 – 7.53 (m, 2H), 6.30 (s, 1H), 4.63 – 4.49 (m, 1H), 2.46 – 2.37 (m, 2H), 2.01 – 1.90 (m, 2H), 1.80 – 1.72 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 165.6, 133.4, 131.7, 128.5, 126.0, 45.3, 31.2, 15.2.

HRMS: calc. for $C_{11}H_{13}BrNO^+$ $(M+H)^+$, 254.0175, found, 254.0181.



20b

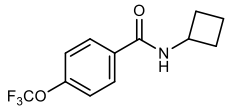
4-chloro-*N*-cyclobutylbenzamide (20b):

Following **GP L**, using 4-chlorobenzoyl chloride (1.7 g, 10.0 mmol) and cyclobutanamine (0.94 mL, 11.0 mmol), **20b** was obtained as a white solid (1.9 g, 90% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.69 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H), 6.38 (d, *J* = 7.4 Hz, 1H), 4.61 – 4.50 (m, 1H), 2.46 – 2.35 (m, 2H), 2.00 – 1.90 (m, 2H), 1.79 – 1.71 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 165.5, 137.5, 132.9, 128.7, 128.3, 45.2, 31.2, 15.2.

HRMS: calc. for $C_{11}H_{13}ClNO^+$ $(M+H)^+$, 210.0680, found, 210.0691.



20d

***N*-cyclobutyl-4-(trifluoromethoxy)benzamide (20d):**

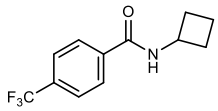
Following **GP L**, using 4-(trifluoromethoxy)benzoyl chloride (2.2 g, 10.0 mmol) and cyclobutanamine (0.94 mL, 11.0 mmol), **20d** was obtained as a white solid (2.4 g, 92% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.79 (d, *J* = 8.7 Hz, 2H), 7.23 (d, *J* = 8.3 Hz, 2H), 6.39 (d, *J* = 6.4 Hz, 1H), 4.62 – 4.50 (m, 1H), 2.47 – 2.37 (m, 2H), 2.01 – 1.92 (m, 2H), 1.80 – 1.72 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 165.3, 151.3 (q, J_{CF} = 2.0 Hz), 133.1, 128.8, 120.6, 120.3 (q, J_{CF} = 258.3 Hz), 45.3, 31.2, 15.2.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -57.8.

HRMS: calc. for $C_{12}H_{12}F_3NNaO_2^+$ $(M+Na)^+$, 282.0712, found, 282.0718.



20e

***N*-cyclobutyl-4-(trifluoromethyl)benzamide (20e):**

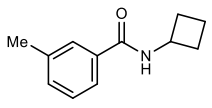
Following **GP L**, using 4-(trifluoromethyl)benzoyl chloride (2.1 g, 10.0 mmol) and cyclobutanamine (0.94 mL, 11.0 mmol), **20e** was obtained as a white solid (2.0 g, 81% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.86 (d, *J* = 8.0 Hz, 2H), 7.68 (d, *J* = 8.1 Hz, 2H), 6.32 (s, 1H), 4.65 – 4.54 (m, 1H), 2.51 – 2.40 (m, 2H), 2.03 – 1.93 (m, 2H), 1.83 – 1.74 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 165.3, 137.9, 133.1 (q, J_{CF} = 32.7 Hz), 127.3, 125.6 (q, J_{CF} = 3.8 Hz), 123.7 (q, J_{CF} = 272.5 Hz), 45.4, 31.2, 15.2.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -62.9.

HRMS: calc. for $C_{12}H_{12}F_3NNaO^+$ $(M+Na)^+$, 266.0763, found, 266.0762.



20f

***N*-cyclobutyl-3-methylbenzamide (20f):**

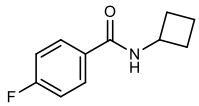
Following **GP L**, using 3-methylbenzoyl chloride (1.5 g, 10.0 mmol) and cyclobutanamine (0.94

mL, 11.0 mmol), **20f** was obtained as a white solid (1.8 g, 94% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.58 (s, 1H), 7.55 – 7.51 (m, 1H), 7.28 – 7.25 (m, 2H), 6.52 (d, *J* = 7.8 Hz, 1H), 4.62 – 4.52 (m, 1H), 2.43 – 2.36 (m, 2H), 2.35 (s, 3H), 2.01 – 1.91 (m, 2H), 1.77 – 1.68 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 166.7, 138.2, 134.5, 131.9, 128.2, 127.6, 123.8, 45.1, 31.2, 21.2, 15.1.

HRMS: calc. for $C_{12}H_{16}NO^+$ $(M+H)^+$, 190.1226, found, 190.1238.



20g

***N*-cyclobutyl-4-fluorobenzamide (20g):**

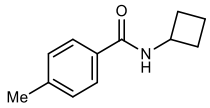
Following **GP L**, using 4-fluorobenzoyl chloride (1.9 g, 10.0 mmol) and cyclobutanamine (0.94 mL, 11.0 mmol), **20g** was obtained as a white solid (1.8 g, 92% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.79 – 7.73 (m, 2H), 7.10 – 7.04 (m, 2H), 6.36 (s, 1H), 4.56 (hept, *J* = 8.1 Hz, 1H), 2.46 – 2.36 (m, 2H), 2.05 – 1.90 (m, 2H), 1.82 – 1.67 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 165.5, 164.6 (d, J_{CF} = 251.6 Hz), 130.7 (d, J_{CF} = 3.2 Hz), 129.2 (d, J_{CF} = 8.8 Hz), 115.5 (d, J_{CF} = 21.8 Hz), 45.2, 31.2, 15.6.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -108.5.

HRMS: calc. for $C_{11}H_{13}FNO^+$ $(M+H)^+$, 194.0976, found, 194.0988.



20h

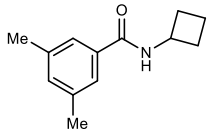
***N*-cyclobutyl-4-methylbenzamide (20h):**

Following **GP L**, using 4-methylbenzoyl chloride (1.5 g, 10.0 mmol) and cyclobutanamine (0.94 mL, 11.0 mmol), **20h** was obtained as a white solid (1.6 g, 85% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.65 (d, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 7.8 Hz, 2H), 6.34 (d, *J* = 7.7 Hz, 1H), 4.58 (hept, *J* = 8.1 Hz, 1H), 2.46 – 2.39 (m, 2H), 2.38 (s, 3H), 1.99 – 1.90 (m, 2H), 1.78 – 1.71 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 166.5, 141.7, 131.7, 129.1, 126.8, 45.1, 31.3, 21.4, 15.1.

HRMS: calc. for $C_{12}H_{16}NO^+$ $(M+H)^+$, 190.1226, found, 190.1238.



20j

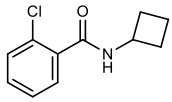
***N*-cyclobutyl-3,5-dimethylbenzamide (20j):**

Following **GP L**, using 3,5-dimethylbenzoyl chloride (1.7 g, 10.0 mmol) and cyclobutanamine (0.94 mL, 11.0 mmol), **20j** was obtained as a white solid (1.8 g, 87% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.35 (s, 2H), 7.11 (s, 1H), 6.24 (s, 1H), 4.58 (hept, *J* = 8.2 Hz, 1H), 2.46 – 2.39 (m, 2H), 2.34 (s, 6H), 1.99 – 1.90 (m, 2H), 1.79 – 1.72 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 166.9, 138.2, 134.6, 132.9, 124.6, 45.1, 31.4, 21.2, 15.1.

HRMS: calc. for $C_{13}H_{17}NNaO^+$ $(M+Na)^+$, 226.1202, found, 226.1213.



20k

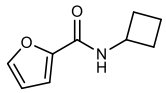
2-chloro-*N*-cyclobutylbenzamide (20k):

Following **GP L**, using 2-chlorobenzoyl chloride (1.7 g, 10.0 mmol) and cyclobutanamine (0.94 mL, 11.0 mmol), **20k** was obtained as a white solid (1.7 g, 83% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.66 – 7.60 (m, 1H), 7.40 – 7.36 (m, 1H), 7.35 – 7.28 (m, 2H), 6.36 (s, 1H), 4.58 (hept, *J* = 8.1 Hz, 1H), 2.48 – 2.40 (m, 2H), 2.03 – 1.97 (m, 2H), 1.83 – 1.73 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 165.4, 135.1, 131.2, 130.5, 130.2, 130.1, 127.0, 45.2, 31.1, 15.3.

HRMS: calc. for $C_{11}H_{13}ClNO^+$ $(M+H)^+$, 210.0680, found, 210.0692.



20l

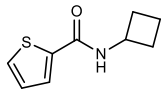
***N*-cyclobutylfuran-2-carboxamide (20l):**

Following **GP L**, using furan-2-carbonyl chloride (1.3 g, 10.0 mmol) and cyclobutanamine (0.94 mL, 11.0 mmol), **20l** was obtained as a white solid (1.3 g, 80% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.42 (d, *J* = 1.3 Hz, 1H), 7.08 (d, *J* = 3.5 Hz, 1H), 6.53 – 6.43 (m, 2H), 4.56 (hept, *J* = 8.2 Hz, 1H), 2.45 – 2.37 (m, 2H), 2.02 – 1.92 (m, 2H), 1.80 – 1.71 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 157.3, 148.0, 143.6, 114.1, 112.1, 44.3, 31.3, 15.1.

HRMS: calc. for $C_9H_{11}NNaO_2^+$ $(M+Na)^+$, 188.0682, found, 188.0679.



20m

***N*-cyclobutylthiophene-2-carboxamide (20m):**

Following **GP L**, using thiophene-2-carbonyl chloride (1.5 g, 10.0 mmol) and cyclobutanamine (0.94 mL, 11.0 mmol), **20m** was obtained as a white solid (1.4 g, 76% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.55 – 7.52 (m, 1H), 7.44 – 7.41 (m, 1H), 7.06 – 7.00 (m, 1H), 6.49 (d, *J* = 7.9 Hz, 1H), 4.54 (hept, *J* = 8.2 Hz, 1H), 2.43 – 2.35 (m, 2H), 2.02 – 1.92 (m, 2H), 1.76 – 1.63 (m, 2H).

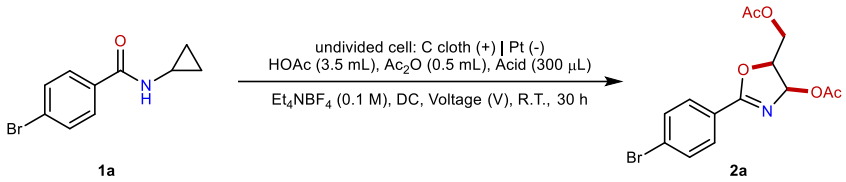
^{13}C NMR (126 MHz, Chloroform-*d*) δ 161.0, 139.1, 129.8, 127.9, 127.5, 45.1, 31.1, 15.1.

HRMS: calc. for $C_9H_{11}NNaOS^+$ $(M+Na)^+$, 204.0454, found, 204.0453.

2.3 Optimization of reaction conditions

*Screening of site-selective ring-opening trioxygenation of **1a** (Tables S1-S4)*

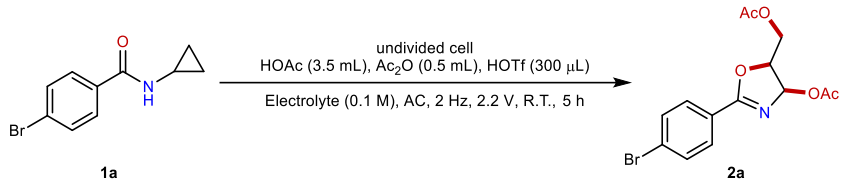
Table S1. Optimization of reaction conditions-Part I



Entry	Voltage (V)	Acid (300 μL)	**Conversion** (%)[a]	**2a** (%)[a]
1	2.0	HOTf	0	0
2	2.1	HOTf	8	4
3	2.2	HOTf	22	11
4	2.3	HOTf	30	7
5	2.4	HOTf	42	5
6	5 mA	HOTf	49	0
7[c]	2.2	HOTf	37	28 (27)[b]
8[d]	2.2	HOTf	45	26 (25)[b]
9[e]	2.2	HOTf	48	24 (21)[b]
10	2.2	TFA	11	5
11	2.2	$ClSO_3H$	73	0
12	2.2	$MeSO_3H$	26	6
13	2.2	H_2SO_4	100	0
14	2.2	HNO_3	100	0
15	2.2	HCOOH	4	0
16	2.2	HFIP	7	0
17	2.2	TFE	15	0
18	2.2	$BF_3{\cdot}Et_2O$	5	0

Standard conditions: Reaction with 0.3 mmol of **1a**, HOAc (3.5 mL), Ac_2O (0.5 mL), Acid (300 μL) and Et_4NBF_4 (0.1 M) in an undivided cell with a constant voltage of 2.2 V at room temperature for 30 h, C cloth anode (15 mm × 15 mm × 0.3 mm), platinum cathode (15 mm × 15 mm × 0.3 mm). [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Isolated yield. [c] Graphite felt (15 mm × 15 mm × 3 mm) instead of C cloth (15 mm × 15 mm × 0.3 mm). [d] Wash C cloth with HOAc (1.0 mL) every 10 h. [e] Replace new C cloth every 10 h.

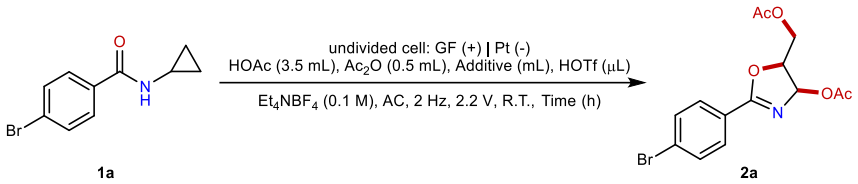
Table S2. Optimization of reaction conditions-Part II



Entry	Electrolyte	Electrode	**Conversion** (%)[a]	**2a** (%)[a]
1	$LiClO_4$	GF (+) | Pt (-)	71	25
2	Et_4NBF_4	GF (+) | Pt (-)	79	37
3	Et_4NOAc	GF (+) | Pt (-)	100	10
4	Bu_4NBF_4	GF (+) | Pt (-)	100	13
5	Bu_4NPF_6	GF (+) | Pt (-)	100	6
6	Bu_4NOAc	GF (+) | Pt (-)	100	3
7	Bu_4NClO_4	GF (+) | Pt (-)	100	14
8	Et_4NBF_4	Graphite (+) | Pt (-)	75	32
9[b]	Et_4NBF_4	GF (+) | Pt (-)	90	22
10	Et_4NBF_4	GF (+) | GF (-)	60	16
11[c]	Et_4NBF_4	Pt (+) | Pt (-)	82	12
12	Et_4NBF_4	GF (+) | Ni (-)	100	0
13	Et_4NBF_4	GF (+) | Ni foam (-)	83	15
14	Et_4NBF_4	GF (+) | Cu (-)	10	0
15	Et_4NBF_4	GF (+) | Fe (-)	100	0
16[d]	Et_4NBF_4	GF (+) | Pt (-)	100	47
17[e]	**Et_4NBF_4**	**GF (+) | Pt (-)**	**100**	**55**
18[f]	Et_4NBF_4	GF (+) | Pt (-)	76	35

Standard conditions: Reaction with 0.3 mmol of **1a**, HOAc (3.5 mL), Ac_2O (0.5 mL), HOTf (300 μL) and Electrolyte (0.1 M) in an undivided cell with an alternating voltage of 2.2 V at room temperature for 5 h, square wave, 2 Hz, duty ratio (D) = 80%, OFFSET = 0.8 V, anode (15 mm × 15 mm × 3 mm) in the positive half-period and cathode (15 mm × 15 mm × 0.3 mm) in the negative half-period. [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] anode (15 mm × 15 mm × 6.5 mm) instead of anode (15 mm × 15 mm × 3 mm). [c] anode (15 mm × 15 mm × 0.3 mm) instead of anode (15 mm × 15 mm × 3 mm). [d] 0.2 mmol of **1a** was used. [e] 0.1 mmol of **1a** was used. [f] 0.4 mmol of **1a** was used.

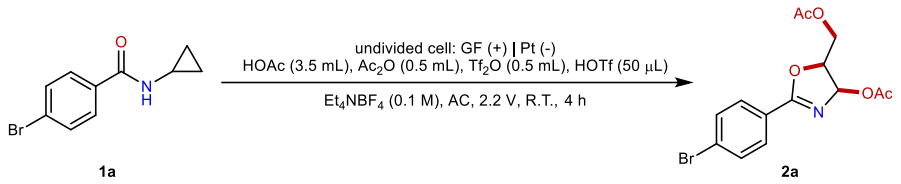
Table S3. Optimization of reaction conditions-Part III



Entry	Additive	HOTf (μL)	Time (h)	**Conversion** (%)[a]	**2a** (%)[a]
1	$BF_3{\cdot}Et_2O$ (0.2 mL)	300	5	88	48
2	TFE (0.2 mL)	300	5	84	29
3	TMSOTf (0.2 mL)	300	5	94	50
4	TMSCN (0.2 mL)	300	5	83	19
5	$TMSCF_3$ (0.2 mL)	300	5	100	0
6	$B(OEt)_3$ (0.2 mL)	300	5	79	23
7	HCOOH (0.2 mL)	300	5	100	9
8	TFAA (0.2 mL)	300	5	100	0
9	Tf_2O (0.2 mL)	300	5	100	56
10	H_2SO_4 (0.2 mL)	300	5	100	7
11	$NaHSO_3$ (0.1 equiv.)	300	5	100	0
12	$NaHSO_4$ (0.1 equiv.)	300	5	100	32
13	Tf_2O (0.1 mL)	300	5	100	52
14	Tf_2O (0.3 mL)	300	5	100	58
15	Tf_2O (0.5 mL)	300	5	100	65
16	Tf_2O (0.5 mL)	20	5	100	50
17	Tf_2O (0.5 mL)	50	5	100	67 (65%, dr = 1.6:1)[b]
18	Tf_2O (0.5 mL)	100	5	100	67 (64%, dr = 1.4:1)[b]
19	Tf_2O (0.5 mL)	200	5	100	65
20	Tf_2O (0.5 mL)	50	3	91	61
21	**Tf_2O (0.5 mL)**	**50**	**4**	**100**	**67 (65%, dr = 1.6:1)[b]**
22	Tf_2O (0.5 mL)	50	6	100	66
23	Tf_2O (0.5 mL)	50	8	100	59
24	Tf_2O (0.5 mL)	50	10	100	56

Standard conditions: Reaction with 0.1 mmol of **1a**, HOAc (3.5 mL), Ac_2O (0.5 mL), HOTf (μL), Additive and Et_4NBF_4 (0.1 M) in an undivided cell with an alternating voltage of 2.2 V at room temperature for 5 h, square wave, 2 Hz, duty ratio (D) = 80%, OFFSET = 0.8 V, graphite felt anode (15 mm × 15 mm × 3 mm) in the positive half-period and Pt cathode (15 mm × 15 mm × 0.3 mm) in the negative half-period. [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Isolated yield.

Table S4. Optimization of reaction conditions-Part IV

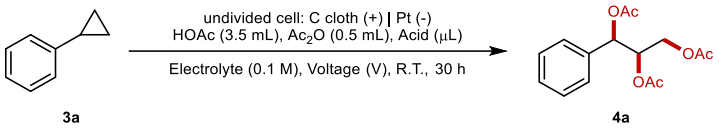


Entry	Frequency (Hz)	Duty ratio (%)	OFFSET (V)	**1a** (%)[a]	**2a** (%)[a]
1	0.1	80	0.8	0	50
2	**0.5**	**80**	**0.8**	**0**	**69 (68%, dr = 1.5:1)[b]**
3	1	80	0.8	0	67
4	5	80	0.8	0	37
5	0.5	80	0.9	14	31
6	0.5	80	0.7	0	64
7	0.5	70	0.8	0	67
8	0.5	90	0.8	0	60
9	0.5	70	0.7	0	65
10[c]	0.5	80	0.8	0	22
11[d]	0.5	80	0.8	0	50
12[e]	0.5	80	0.8	4	62
13[f]	0.5	80	0.8	0	60
13[g]	0.5	80	0.8	0	67
14[h]	0.5	80	0.8	0	65

Standard conditions: Reaction with 0.1 mmol of **1a**, HOAc (3.5 mL), Ac_2O (0.5 mL), Tf_2O (0.5 mL), HOTf (50 μL) and Et_4NBF_4 (0.1 M) in an undivided cell with an alternating voltage of 2.2 V at room temperature for 4 h, GF anode (15 mm × 15 mm × 3 mm) in the positive half-period and Pt cathode (15 mm × 15 mm × 0.3 mm) in the negative half-period. [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Isolated yield. [c] Sine wave instead of square wave. [d] Pulse wave instead of square wave. [e] HOAc (4.0 mL) instead of HOAc (3.5 mL). [f] HOAc (3.0 mL) instead of HOAc (3.5 mL). [g] Ac_2O (0.4 mL) instead of Ac_2O (0.5 mL). [h] Ac_2O (0.6 mL) instead of Ac_2O (0.5 mL).

*Screening of site-selective ring-opening trioxygenation of **3a** (Tables S5-S7)*

Table S5. Optimization of reaction conditions-Part I

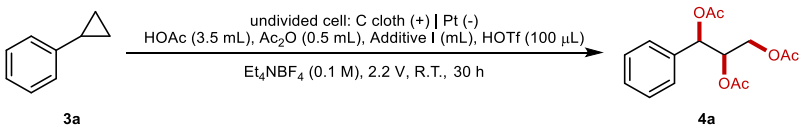


3a **4a**

Entry	Electrolyte	Voltage (V)	Acid (μL)	**4a** (%)[a]
1	$LiClO_4$	2.2	HOTf (200)	11
2	Et_4NBF_4	2.2	HOTf (200)	13
3	Et_4NOAc	2.2	HOTf (200)	4
4	Bu_4NBF_4	2.2	HOTf (200)	0
5	Bu_4NPF_6	2.2	HOTf (200)	0
6	Bu_4NOAc	2.2	HOTf (200)	0
7	Bu_4NClO_4	2.2	HOTf (200)	0
8	Et_4NBF_4	1.5	HOTf (200)	0
9	Et_4NBF_4	1.8	HOTf (200)	0
10	Et_4NBF_4	2.1	HOTf (200)	9
11	Et_4NBF_4	2.3	HOTf (200)	11
12	Et_4NBF_4	2.5	HOTf (200)	10
13	**Et_4NBF_4**	**2.2**	**HOTf (100)**	**15**
14	Et_4NBF_4	2.2	HOTf (300)	10
15	Et_4NBF_4	5 mA	HOTf (200)	0
16	Et_4NBF_4	10 mA	HOTf (200)	0
17	Et_4NBF_4	2.2	TFA (200)	3
18	Et_4NBF_4	2.2	$ClSO_3H$ (200)	0
19	Et_4NBF_4	2.2	$MeSO_3H$ (200)	0
20	Et_4NBF_4	2.2	H_2SO_4 (200)	0
21	Et_4NBF_4	2.2	HNO_3 (200)	0
22	Et_4NBF_4	2.2	$BF_3{\cdot}Et_2O$ (200)	0

Standard conditions: Reaction with 0.3 mmol of **3a**, HOAc (3.5 mL), Ac_2O (0.5 mL), Acid (μL) and Electrolyte (0.1 M) in an undivided cell with a constant voltage at room temperature for 30 h, C cloth anode (15 mm × 15 mm × 0.3 mm), Pt cathode (15 mm × 15 mm × 0.3 mm). [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard.

Table S6. Optimization of reaction conditions-Part II

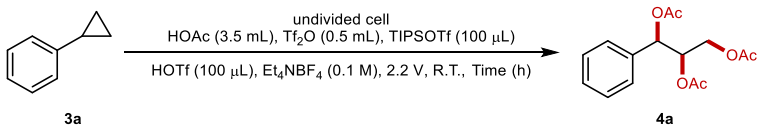


3a **4a**

Entry	Additive I (mL)	**4** (%)[a]
1	Tf_2O (0.1)	14
2	Tf_2O (0.3)	18
3	Tf_2O (0.5)	21
4[b]	Tf_2O (0.5)	23
5	Tf_2O (0.7)	22
6	$BF_3{\cdot}Et_2O$ (0.2)	11
7	TFE (0.2)	0
8	TMSOTf (0.2)	17
9	TBSOTf (0.2)	17
10	TESOTf (0.2)	20
11	TIPSOTf (0.2)	20
12	Et_3SiH (0.2)	16
13	TFAA (0.2)	0
14[c]	Tf_2O (0.5)	23
15[d]	**Tf_2O (0.5)**	**25**
16[e]	Tf_2O (0.5)	22

Standard conditions: Reaction with 0.3 mmol of **3a**, HOAc (3.5 mL), Ac_2O (0.5 mL), HOTf (100 μL), Additive I (mL) and Et_4NBF_4 (0.1 M) in an undivided cell with a constant voltage of 2.2 V at room temperature for 30 h, C cloth anode (15 mm × 15 mm × 0.3 mm), Pt cathode (15 mm × 15 mm × 0.3 mm). [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Without Ac_2O. [c] With TIPSOTf (0.2 mL). [d] With TIPSOTf (0.1 mL). [e] With TIPSOTf (0.3 mL).

Table S7. Optimization of reaction conditions-Part III



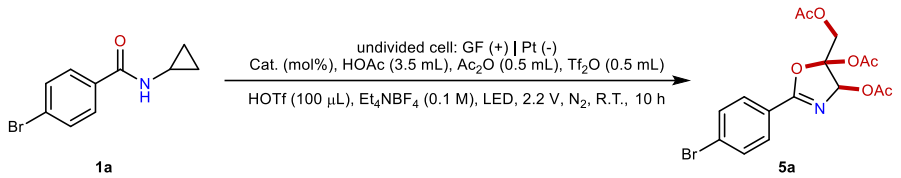
3a 4a

Entry	Electrode	Time (h)	**4** (%)[a]
1	Graphite (+) | Pt (-)[d]	30	21
2	GF (+) | Pt (-)[d]	30	29
3[c]	GF (+) | Pt (-)[d]	30	14
4	GF (+) | SS (-)[d]	30	0
5	GF (+) | Ni (-)[d]	30	35
6	GF (+) | Ni foam (-)	30	45
7	GF (+) | Cu (-)[d]	30	23
8	GF (+) | Fe (-)[d]	30	17
9	GF (+) | Ti (-)[d]	30	9
10	GF (+) | Al (-)[d]	30	0
11	GF (+) | Sn (-)[d]	30	40
12	GF (+) | Zn (-)[d]	30	14
13	GF (+) | GF (-)	30	0
14[d,e]	Pt (+) | Pt (-)	30	0
15	GF (+) | Ni foam (-)	23	36
16	GF (+) | Ni foam (-)	36	48
17	**GF (+) | Ni foam (-)**	**48**	**57 (55%, dr = 1.9:1)[b]**
18	GF (+) | Ni foam (-)	58	57 (54%, dr = 1.7:1)[b]

Standard conditions: Reaction with 0.3 mmol of **3a**, HOAc (3.5 mL), Tf_2O (0.5 mL), TIPSOTf (100 μL), HOTf (100 μL) and Et_4NBF_4 (0.1 M) in an undivided cell with a constant voltage of 2.2 V at room temperature for time, anode (15 mm × 15 mm × 3 mm), cathode (15 mm × 15 mm × 3 mm). [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Isolated yield. [c] Graphite felt (15 mm × 15 mm × 6.5 mm). [d] Cathode (15 mm × 15 mm × 0.3 mm). [e] Anode (15 mm × 15 mm × 0.3 mm).

*Screening of site-selective ring-opening tetraoxygenation of **1a** (Tables S8-S9)*

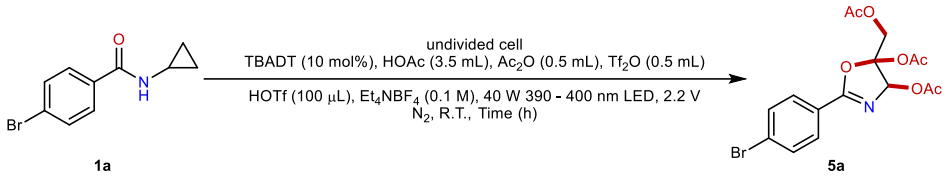
Table S8. Optimization of reaction conditions-Part I



Entry	Cat. (mol%)	LED	**5a** (%)[a]
1	$FeCl_3$ (5)	40 W 390 - 400 nm	27
2	$FeCl_3$ (5)	50 W 395 - 400 nm	10
3	$FeCl_3$ (5)	40 W 455 - 460 nm	0
4	$FeCl_3$ (5)	3 W 395 - 400 nm	0
5	$FeCl_3·6H_2O$ (5)	40 W 390 - 400 nm	7
6	$CeCl_3$ (5)	40 W 390 - 400 nm	0
7	$Ce(OAc)_3$ (5)	40 W 390 - 400 nm	0
8	TBADT (5)	40 W 390 - 400 nm	30
9	**TBADT (10)**	**40 W 390 - 400 nm**	**34**
10	TBADT (15)	40 W 390 - 400 nm	34
11	TBADT (20)	40 W 390 - 400 nm	33
12	NaDT (5)	40 W 390 - 400 nm	14

Standard conditions: Reaction with 0.1 mmol of **1a**, Cat. (mol%), HOAc (3.5 mL), Ac_2O (0.5 mL), Tf_2O (0.5 mL), HOTf (100 μL) and Et_4NBF_4 (0.1 M) in an undivided cell with a constant voltage of 2.2 V under LEDs at room temperature for 10 h, N_2, graphite felt anode (15 mm × 15 mm × 3 mm), Pt cathode (15 mm × 15 mm × 0.3 mm). [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard.

Table S9. Optimization of reaction conditions-Part II

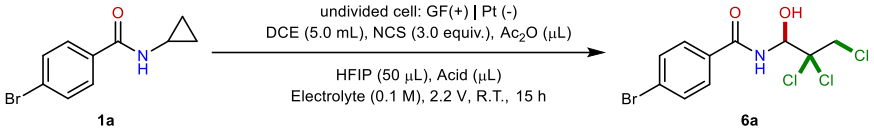


Entry	Electrode	Time (h)	Additive (mL)	**5a** (%)[a]
1	Graphite (+) | Pt (-)	10		24
2	C cloth (+) | Pt (-)	10		5
3	GF (+) | Ni (-)	10		3
4[c]	GF (+) | Ni foam (-)	10		25
5[c]	GF (+) | GF (-)	10		7
6[d]	Pt (+) | Pt (-)	10		0
7	GF (+) | Pt (-)	5		15
8	GF (+) | Pt (-)	8		24
9	GF (+) | Pt (-)	15		34
10	**GF (+) | Pt (-)**	**20**		**40 (41%, dr = 2.3:1)[b]**
11	GF (+) | Pt (-)	25		39
12	GF (+) | Pt (-)	30		33
13	GF (+) | Pt (-)	20	$BF_3{\cdot}Et_2O$ (0.2)	38
14	GF (+) | Pt (-)	20	TFE (0.2)	12
15	GF (+) | Pt (-)	20	TMSOTf (0.2)	35
16	GF (+) | Pt (-)	20	TIPSOTf (0.2)	30
17	GF (+) | Pt (-)	20	TFAA (0.2)	10

Standard conditions: Reaction with 0.1 mmol of **1a**, TBADT (10 mol%), HOAc (3.5 mL), Ac_2O (0.5 mL), Tf_2O (0.5 mL), HOTf (100 μL), Additive and Et_4NBF_4 (0.1 M) in an undivided cell with a constant voltage of 2.2 V under 40 W 390 - 400 nm LEDs at room temperature for time, anode (15 mm × 15 mm × 3 mm), cathode (15 mm × 15 mm × 0.3 mm), N_2. [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Isolated yield. [c] Cathode (15 mm × 15 mm × 3 mm). [d] Pt (15 mm × 15 mm × 0.3 mm).

*Screening of site-selective ring-opening trichlorohydroxylation of **1a** (Tables S10-S11)*

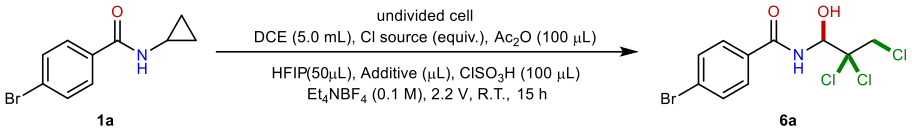
Table S10. Optimization of reaction conditions-Part I



Entry	Electrolyte	Ac_2O (μL)	Acid (μL)	**6a** (%)[a]
1	TBACl	500	$ClSO_3H$ (100)	19
2	TBACl	100	$ClSO_3H$ (100)	0
3	TBAOAc	500	$ClSO_3H$ (100)	0
4	TBAOAc	100	$ClSO_3H$ (100)	16
5	Et_4NBF_4	500	$ClSO_3H$ (100)	0
6	**Et_4NBF_4**	**100**	**$ClSO_3H$ (100)**	**40**
7	Et_4NBF_4	50	$ClSO_3H$ (100)	36
8	Et_4NBF_4		$ClSO_3H$ (100)	31
9	Et_4NBF_4	100	$ClSO_3H$ (60)	33
10	Et_4NBF_4	100	$ClSO_3H$ (150)	40
11[b]	Et_4NBF_4	100	$ClSO_3H$ (100)	31
12[c]	Et_4NBF_4	100	$ClSO_3H$ (100)	24
13	Et_4NBF_4	100	TFA (100)	0
14	Bu_4NClO_4	100	$MeSO_3H$ (100)	30
15	Et_4NBF_4	100	HOTf (100)	11

Standard conditions: Reaction with 0.2 mmol of **1a**, DCE (5.0 mL), NCS (3.0 equiv.), Ac_2O (μL), HFIP (50 μL), Acid (μL) and Electrolyte (0.1 M) in an undivided cell with a constant voltage of 2.2 V at room temperature for 15 h, graphite felt anode (15 mm × 15 mm × 6.5 mm), Pt cathode (15 mm × 15 mm × 0.3 mm). [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Without HFIP. [c] HFIP (100 μL) instead of HFIP (50 μL).

Table S11. Optimization of reaction conditions-Part II

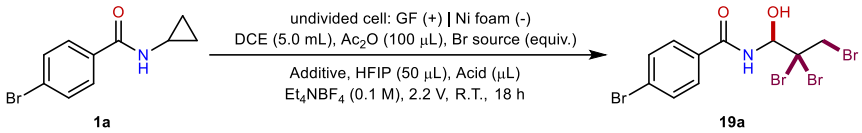


Entry	Additive (μL)	Electrode	Cl source (equiv.)	**6a** (%)[a]
1	Tf_2O (30)	GF (+) | Pt (-)[c]	NCS (3.0)	45
2	Tf_2O (50)	GF (+) | Pt (-)[c]	NCS (3.0)	47
3	Tf_2O (80)	GF (+) | Pt (-)[c]	NCS (3.0)	44
4	Tf_2O (100)	GF (+) | Pt (-)[c]	NCS (3.0)	45
5	Tf_2O (150)	GF (+) | Pt (-)[c]	NCS (3.0)	41
6	TFE (100)	GF (+) | Pt (-)[c]	NCS (3.0)	32
7	TFAA (100)	GF (+) | Pt (-)[c]	NCS (3.0)	38
8[d]	Tf_2O (50)	GF (+) | Pt (-)[c]	NCS (3.0)	42
9	Tf_2O (50)	Graphite (+) | Pt (-)[c]	NCS (3.0)	19
10[e]	Tf_2O (50)	C cloth (+) | Pt (-)[c]	NCS (3.0)	27
11	Tf_2O (50)	GF (+) | Ni (-)	NCS (3.0)	15
12	Tf_2O (50)	GF (+) | Ni foam (-)	NCS (3.0)	48
13	Tf_2O (50)	GF (+) | GF (-)	NCS (3.0)	23
14[c]	Tf_2O (50)	Pt (+) | Pt (-)	NCS (3.0)	41
15	Tf_2O (50)	GF (+) | Ni foam (-)	NCS (3.5)	50
16	Tf_2O (50)	GF (+) | Ni foam (-)	NCS (4.0)	42
17	Tf_2O (50)	GF (+) | Ni foam (-)	LiCl (3.5)	29
18	Tf_2O (50)	GF (+) | Ni foam (-)	DCDMH (2.0)	55
19	Tf_2O (50)	GF (+) | Ni foam (-)	DCDMH (3.5)	68
20	Tf_2O (50)	GF (+) | Ni foam (-)	DCDMH (4.0)	60
21	Tf_2O (50)	GF (+) | Ni foam (-)	DCDMH (5.0)	68
22[f]	**Tf_2O (50)**	**GF (+) | Ni foam (-)**	**DCDMH (3.5)**	**80 (78%)[b]**
23[g]	Tf_2O (50)	GF (+) | Ni foam (-)	DCDMH (3.5)	62
24[h]	Tf_2O (50)	GF (+) | Ni foam (-)	DCDMH (3.5)	42
25[i]	Tf_2O (50)	GF (+) | Ni foam (-)	DCDMH (3.5)	0
26[j]	Tf_2O (50)	GF (+) | Ni foam (-)	DCDMH (3.5)	0
27[k]	Tf_2O (50)	GF (+) | Ni foam (-)	DCDMH (3.5)	0

Standard conditions: Reaction with 0.2 mmol of **1a**, DCE (5.0 mL), Cl source (equiv.), Ac_2O (100 μL), HFIP (50 μL), Additive (μL), $ClSO_3H$ (100 μL) and Et_4NBF_4 (0.1 M) in an undivided cell with a constant voltage of 2.2 V at room temperature for 15 h, anode (15 mm × 15 mm × 6.5 mm), cathode (15 mm × 15 mm × 3 mm). [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Isolated yield. [c] Pt (15 mm × 15 mm × 0.3 mm). [d] Graphite felt anode (15 mm × 15 mm × 3 mm). [e] C cloth anode (15 mm × 15 mm × 0.3 mm). [f] 0.1 mmol of **1a** was used. [g] 0.3 mmol of **1a** was used. [h] DCM instead of DCE. [i] Toluene instead of DCE. [j] $CHCl_3$ instead of DCE. [k] DME instead of DCE.

*Screening of site-selective ring-opening tribromohydroxylation of **1a** (Tables S12-S13)*

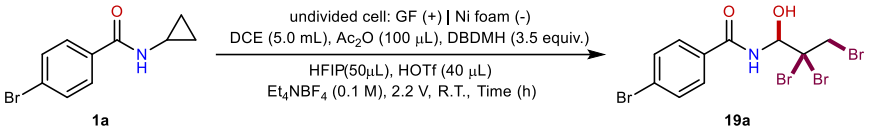
Table S12. Optimization of reaction conditions-Part I



Entry	Br source (equiv.)	Additive	Acid (μL)	**19a** (%)[a]
1	NBS (2.0)		$ClSO_3H$ (100)	17[b]
2	NBS (3.5)		$ClSO_3H$ (100)	23[b]
3	NBS (4.0)		$ClSO_3H$ (100)	22[b]
4	DBDMH (2.0)		$ClSO_3H$ (100)	29[b]
5	DBDMH (3.5)		$ClSO_3H$ (100)	37[b]
6	DBDMH (3.5)	Na_2HPO_4 (0.2 equiv.)	$ClSO_3H$ (100)	24[b]
7	DBDMH (3.5)	Na_2HPO_4 (0.5 equiv.)	$ClSO_3H$ (100)	23[b]
8	DBDMH (3.5)	Na_2HPO_4 (1.0 equiv.)	$ClSO_3H$ (100)	29[b]
9	DBDMH (4.0)	Tf_2O (0.02 mL)	$ClSO_3H$ (100)	35[b]
10	DBDMH (3.5)	Tf_2O (0.05 mL)	$ClSO_3H$ (100)	32[b]
11	DBDMH (3.5)	Tf_2O (0.1 mL)	$ClSO_3H$ (100)	33[b]
12	DBDMH (3.5)	$BF_3{\cdot}Et_2O$ (0.2 mL)	$ClSO_3H$ (100)	21[b]
13	DBDMH (3.5)	TFE (0.2 mL)	$ClSO_3H$ (100)	18[b]
14	DBDMH (3.5)	TMSOTf (0.2 mL)	$ClSO_3H$ (100)	33[b]
15	DBDMH (3.5)		TFA (100)	0
16	DBDMH (3.5)		$MeSO_3H$ (100)	5
17	DBDMH (3.5)		HOTf (20)	24
18	DBDMH (3.5)		HOTf (40)	25
19	DBDMH (3.5)		HOTf (60)	24
20	DBDMH (3.5)		HOTf (80)	13
21	DBDMH (3.5)		HOTf (100)	7

Standard conditions: Reaction with 0.1 mmol of **1a**, DCE (5.0 mL), Br source (equiv.), Ac_2O (100 μL), Acid (μL), Additive and Et_4NBF_4 (0.1 M) in an undivided cell with a constant voltage of 2.2 V at room temperature for 18 h, graphite felt anode (15 mm × 15 mm × 6.5 mm), Ni foam cathode (15 × mm × 15 mm × 3 mm). [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Mixture with 4-bromo-*N*-(2,2-dibromo-3-chloro-1-hydroxypropyl)benzamide.

Table S13. Optimization of reaction conditions-Part II

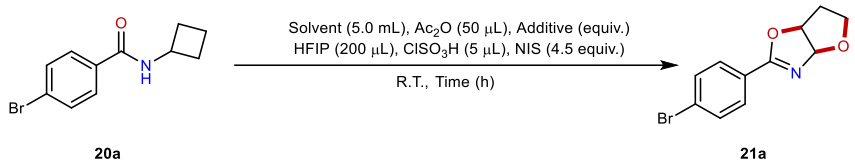


Entry	Step 1	Step 2	**19a** (%)[a]
1	2 h, with **1a** (0.1 mmol)	without electrolysis, 20 h	5
2	2 h, with **1a** (0.1 mmol)	with electrolysis, 20 h	20
3	2 h	add **1a** (0.1 mmol), without electrolysis, 20 h	61
4	2 h	add **1a** (0.1 mmol), with electrolysis, 20 h	24
5	1 h	add **1a** (0.1 mmol), without electrolysis, 20 h	54
6	**3 h**	**add 1a (0.1 mmol), without electrolysis, 20 h**	**69 (67)[b]**
7	4 h	add **1a** (0.1 mmol), without electrolysis, 20 h	67
8	6 h	add **1a** (0.1 mmol), without electrolysis, 20 h	56
9	3 h	add **1a** (0.1 mmol), without electrolysis, 10 h	64
10	3 h	add **1a** (0.1 mmol), without electrolysis, 15 h	68
11	3 h	add **1a** (0.1 mmol), without electrolysis, 25 h	67
12[c]	3 h	add **1a** (0.1 mmol), without electrolysis, 20 h	0
13[d]	3 h	add **1a** (0.1 mmol), without electrolysis, 20 h	10
14[e]	3 h	add **1a** (0.1 mmol), without electrolysis, 20 h	28

Standard conditions: Reaction with DCE (5.0 mL), DBDMH (3.5 equiv.), Ac_2O (100 μL), HFIP (50 μL), HOTf (40 μL) and Et_4NBF_4 (0.1 M) in an undivided cell with a constant voltage of 2.2 V at room temperature for time, graphite felt anode (15 mm × 15 mm × 6.5 mm), Ni foam cathode (15 mm × 15 mm × 3 mm). [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Isolated yield. [c] TFA instead of HOTf. [d] $MeSO_3H$ instead of HOTf. [e] $ClSO_3H$ instead of HOTf.

*Screening of site-selective ring-opening trioxygenation of **20a** (Tables S14-S15)*

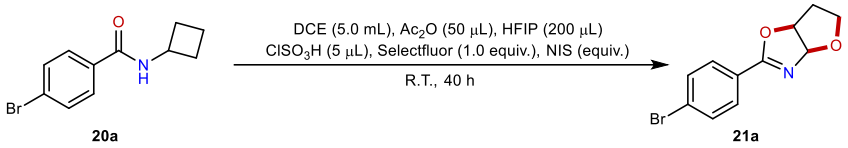
Table S14. Optimization of reaction conditions-Part I



Entry	Solvent (5.0 mL)	Additive	Time (h)	**Conversion** (%)[a]	**21a** (%)[a]
1	DCE		15	31	10
2[b]	DCE		15	59	5
3[c]	DCE		15	2	0
4	DCM		15	95	0
5	$CHCl_3$		15	70	0
6	MeCN		15	5	0
7	THF		15	75	0
8	Toluene		15	3	0
9	MeOH		15	0	0
10	DMSO		15	72	5
11	NMP		15	0	0
12	Acetone		15	1	0
13	DCE	$PhI(OAc)_2$ (0.1 equiv.)	15	42	7
14	DCE	$PhI(OAc)_2$ (1.0 equiv.)	15	51	9
15	DCE	Selectfluor (0.1 equiv.)	15	48	8
16	DCE	Selectfluor (1.0 equiv.)	15	57	16
17	DCE	Selectfluor (2.0 equiv.)	15	51	11
18	DCE	PIFA (0.1 equiv.)	15	55	11
19	DCE	PIFA (1.0 equiv.)	15	67	17
20	DCE	NFSI (0.1 equiv.)	15	48	8
21	DCE	NFSI (1.0 equiv.)	15	50	7
22	DCE	*m*-CPBA (1.0 equiv.)	15	60	6
23	DCE	TFE (0.1 mL)	15	60	11
24	DCE	$CeCO_3$ (1.0 equiv.)	15	56	13
25	DCE	$FeCl_3$ (1.0 equiv.)	15	46	0
26	DCE	LiCl (1.0 equiv.)	15	24	0
27	DCE	Selectfluor (1.0 equiv.)	40	51	24
28	DCE		72	82	30
29	**DCE**	**Selectfluor (1.0 equiv.)**	**72**	**87**	**33**
30	DCE	Selectfluor (1.0 equiv.)	96	96	31

Standard conditions: Reaction with 0.1 mmol of **20a,** Solvent (5.0 mL), Ac_2O (50 μL), HFIP (200 μL), $ClSO_3H$ (5 μL), Additive and NIS (4.5 equiv.) in a tube at room temperature for time. [a] The conversions and yields were determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Without Ac_2O. [c] Without HFIP.

Table S15. Optimization of reaction conditions-Part II

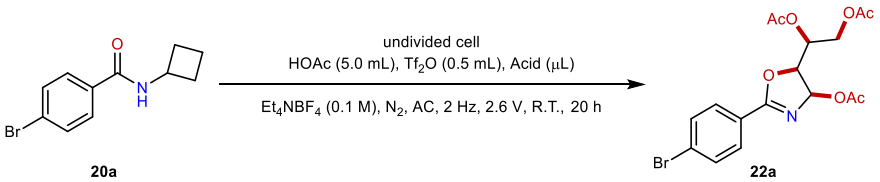


Entry	NIS (equiv.)	**Conversion** (%)[a]	**21a** (%)[a]
1	1.0	17	5
2	2.0	23	11
3	3.0	31	18
4	4.0	43	22
5[c]	4.5	47	27
6	5.0	70	27
7[d]	4.5	76	35
8[c,d]	**4.5**	**96**	**41 (40)**[b]
7[e]	4.5	49	14
8[f]	4.5	29	17

Standard conditions: Reaction with 0.1 mmol of **20a,** DCE (5.0 mL), Ac_2O (50 μL), HFIP (200 μL), $ClSO_3H$ (5 μL), Selectfluor (1.0 equiv.) and NIS (equiv.) in a tube at room temperature for 40 h. [a] The conversions and yields were determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Isolated yield. [c] NIS and Selectfluor was carefully added in 2 times interval of 20 h. [d] 0.05 mmol was used. [e] 0.2 mmol was used. [f] 0.3 mmol was used.

*Screening of site-selective ring-opening tetraoxygenation of **20a** (Tables S16-S18)*

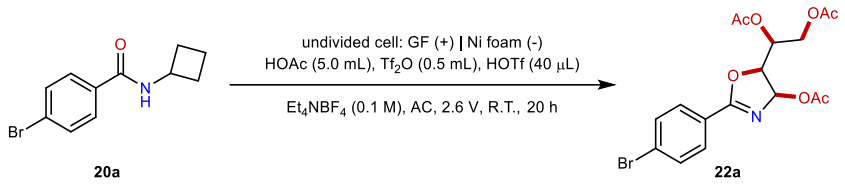
Table S16. Optimization of reaction conditions-Part I



Entry	Electrode	Acid (μL)	**22a** (%)[a]
1	1.0 mm Graphite (+) | 0.3 mm Pt (-)	HOTf (200)	4
2	0.3 mm C cloth (+) | 0.3 mm Pt (-)	HOTf (200)	5
3	3.0 mm GF (+) | 0.3 mm Pt (-)	HOTf (200)	3
4	0.3 mm Pt (+) | 0.3 mm Pt (-)	HOTf (200)	7
5	1.0 mm Graphite (+) | 3.0 mm Ni foam (-)	HOTf (200)	16
6	2.0 mm Graphite (+) | 3.0 mm Ni foam (-)	HOTf (200)	17
7	2.0 mm Graphite (+) | 2.0 mm Graphite (-)	HOTf (200)	17
8	3.0 mm Graphite (+) | 3.0 mm Ni foam (-)	HOTf (200)	17
9	0.3 mm C cloth (+) | 3.0 mm Ni foam (-)	HOTf (200)	10
10	6.5 mm GF (+) | 3.0 mm Ni foam (-)	HOTf (200)	26
11	6.5 mm GF (+) | 3.0 mm Ni foam (-)	HOTf (200)	22
12	**6.5 mm GF (+) | 3.0 mm Ni foam (-)**	**HOTf (40)**	**29**
13[b]	6.5 mm GF (+) | 3.0 mm Ni foam (-)	HOTf (40)	24
14[c]	6.5 mm GF (+) | 3.0 mm Ni foam (-)	HOTf (40)	20
13	6.5 mm GF (+) | 3.0 mm Ni foam (-)	HOTf (60)	22
14	6.5 mm GF (+) | 3.0 mm Ni foam (-)	HOTf (100)	24
15	6.5 mm GF (+) | 3.0 mm Ni foam (-)	HOTf 300)	25
16	3.0 mm GF (+) | 3.0 mm Ni foam (-)	HOTf (200)	15
17	6.5 mm GF (+) | 6.5 mm GF (-)	HOTf (200)	20
18	6.5 mm GF (+) | 0.3 mm SS (-)	HOTf (200)	23
19	6.5 mm GF (+) | 0.3 mm Cu foam (-)	HOTf (200)	23
20	6.5 mm GF (+) | 0.3 mm Al (-)	HOTf (200)	6
21	6.5 mm GF (+) | 3.0 mm Ni foam (-)	TFA (200)	2
22	6.5 mm GF (+) | 3.0 mm Ni foam (-)	$MeSO_3H$ (200)	6
23	6.5 mm GF (+) | 3.0 mm Ni foam (-)	$ClSO_3H$ (200)	0
24	6.5 mm GF (+) | 3.0 mm Ni foam (-)	HCl (200)	0
25	6.5 mm GF (+) | 3.0 mm Ni foam (-)	H_2SO_4 (200)	0
26	6.5 mm GF (+) | 3.0 mm Ni foam (-)	$BF_3{\cdot}Et_2O$ (200)	3

Standard conditions: Reaction with 0.2 mmol of **20a**, HOAc (5.0 mL), Tf_2O (0.5 mL), Acid (μL) and Et_4NBF_4 (0.1 M) in an undivided cell with an alternating voltage of 2.6 V at room temperature for 20 h, square wave, 2 Hz, duty ratio (D) = 80%, OFFSET = 0.8 V, anode (15 mm × 15 mm × x mm) in the positive half-period and cathode (15 mm × 15 mm × x mm) in the negative half-period. [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] 2.5 V. [c] 2.7 V.

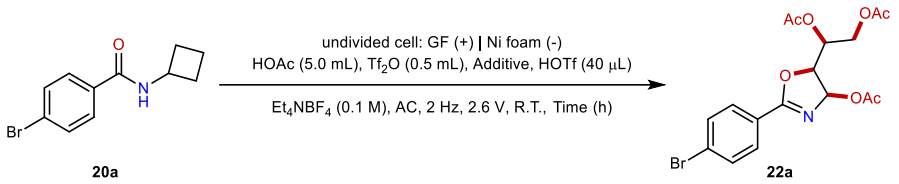
Table S17. Optimization of reaction conditions-Part II



Entry	Frequency (Hz)	Duty ratio (%)	OFFSET (V)	**22a** (%)[a]
1	0.1	80	0.8	5
2	0.5	80	0.8	11
3	1	80	0.8	20
4	5	80	0.8	8
5	2	80	0.9	27
6	2	80	0.7	23
7	2	80	0.6	16
8	2	80	0.4	7
9	2	90	0.8	25
10	2	70	0.8	24
11	2	60	0.8	0
12	2	50	0.8	0

Standard conditions: Reaction with 0.2 mmol of **20a**, HOAc (5.0 mL), Tf_2O (0.5 mL), HOTf (40 μL) and Et_4NBF_4 (0.1 M) in an undivided cell with an alternating voltage of 2.6 V at room temperature for 20 h, square wave, frequency, duty ratio, OFFSET, graphite felt anode (15 mm × 15 mm × 6.5 mm) in the positive half-period and Ni foam cathode (15 mm × 15 mm × 3 mm) in the negative half-period. [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard.

Table S18. Optimization of reaction conditions-Part III



Entry	Additive	Time (h)	**22a** (%)[a]
1	LiOTf (0.5 equiv.)	20	15
2	$Y(OTf)_3$ (0.5 equiv.)	20	31
3	$Cu(OTf)_3$ (0.5 equiv.)	20	18
4	$Fe(OTf)_3$ (0.5 equiv.)	20	0
5	$Fe(OTf)_2$ (0.5 equiv.)	20	0
6	$Bi(OTf)_3$ (0.5 equiv.)	20	24
7	$Ce(OTf)_3$ (0.1 equiv.)	20	40
8	$Ce(OTf)_3$ (0.1 equiv.)	34	4
9	$Ce(OTf)_3$ (0.3 equiv.)	20	8
10	$Ce_2(SO_4)_3$ (0.1 equiv.)	20	24
11	CAN (0.1 equiv.)	20	38
12	CAN (0.1 equiv.)	34	42
13	CAN (0.2 equiv.)	20	15
14	$Ce(CO_3)_3$ (0.1 equiv.)	20	29
15	CeO_2 (0.1 equiv.)	20	13
16	$Ce(OAc)_3$ (0.1 equiv.)	20	42
17	$Ce(OAc)_3$ (0.1 equiv.)	34	44
18[c]	**$Ce(OAc)_3$ (0.1 equiv.)**	**34**	**46 (46%, dr = 3:1)**[b]
19	$Ce(OAc)_3$ (0.1 equiv.)	48	45
20	$Ce(OAc)_3$ (0.5 equiv.)	20	31
21	$Mn(OAc)_2$ (0.5 equiv.)	20	5
22	$Cu(OAc)_2$ (0.5 equiv.)	20	20
23	NaOAc (0.5 equiv.)	20	7
24	$Pd(OAc)_2$ (0.5 equiv.)	20	18
25	$Mn(OAc)_2$ (0.5 equiv.)	20	5
26	CF_2COOH (0.5 equiv.)	20	9
27	CF_2COONa (0.5 equiv.)	20	24
28	Et_3SiH (0.5 equiv.)	20	25
29	$K_2S_2O_8$ (0.5 equiv.)	20	6
30	TsOH (0.5 equiv.)	20	0
31	MsONa (0.5 equiv.)	20	11
32	DIPEA (0.5 equiv.)	20	12
33	Pyridine (0.5 equiv.)	20	18
34	TEAA (0.5 equiv.)	20	27
35	CsF (0.5 equiv.)	20	27
36	AgF_2 (0.5 equiv.)	20	26

37	FeF_3 (0.5 equiv.)	20	3
38	$FeSO_4$ (0.5 equiv.)	20	3
39	PPh_3 (0.5 equiv.)	20	18
40	$NiBr_2$ (0.5 equiv.)	20	3
41	Mn (0.5 equiv.)	20	13
42	$MnCl_2$ (0.5 equiv.)	20	0
43	$CeCl_3$ (0.5 equiv.)	20	5
44	$InCl_3$ (0.5 equiv.)	20	0
45	$BF_3{\cdot}Et_2O$ (0.2 mL)	20	2
46	TIPSOTf (0.2 mL)	20	24
47	TMSOTf (0.2 mL)	20	18
48	TBSOTf (0.2 mL)	20	26
49	TFAA (0.2 mL)	20	0
50	HCOOH (0.2 mL)	20	0
51	MeOTf (0.2 mL)	20	8
52	TMSCN (0.2 mL)	20	14
53	$TMSN_3$ (0.2 mL)	20	18

Standard conditions: Reaction with 0.2 mmol of **20a**, HOAc (5.0 mL), Tf_2O (0.5 mL), Additive, HOTf (40 μL) and Et_4NBF_4 (0.1 M) in an undivided cell with an alternating voltage of 2.6 V at room temperature for time, square wave, 2 Hz, duty ratio (D) = 80%, OFFSET = 0.8 V, graphite felt anode (15 mm × 15 mm × 6.5 mm) in the positive half-period and Ni foam cathode (15 mm × 15 mm × 3 mm) in the negative half-period. [a] Yields determined by ^1H NMR analysis using triphenylmethane as the internal standard. [b] Isolated yield. [c] Under N_2.

2.4 Mechanistic studies

2.4.1 Cyclic voltammetry measurement

Cyclic voltammetry (CV) experiments were conducted in a 10 mL glass vial fitted with a glassy carbon working electrode (3.0 mm in diameter), a platinum wire auxiliary electrode and SCE reference electrode. The scan rate was 0.1 V/s. Current was reported in A, while all potentials were reported in V. $E_{p/2}$ was measured. Samples were prepared with 1 mM of substrates respectively, with 0.1 M Et_4NBF_4 in 3.5 mL of anhydrous acetonitrile. We measured the redox potential of selected substrates and possible intermediates (Figure S9-11) in CH_3CN. Comparative analysis (Figure S12) of redox potential diagrams (**1a** vs **1ak**, **1f** vs **1al**, **3a** vs **3i**, and **20a** vs **1ak**) revealed oxidation potentials of $E_{p/2}$ = 1.52 V vs. SCE for the cyclopropaneamide (cyclopropane), $E_{p/2}$ = 1.66 V vs. SCE for the arylcyclopropane (cyclopropane), and $E_{p/2}$ = 1.72 V vs. SCE for the cyclobutaneamide (cyclobutane). Subsequent CV measurements of **1a**, **3a**, and **20a** in the reaction system (Figure S13-15)[13] showed that adding HOTf or/and Tf_2O eliminated the oxidation peaks of cyclopropane/butane, suggesting multioxidation of amidecyclopropanes/butanes initiates via amide nitrogen oxidation.

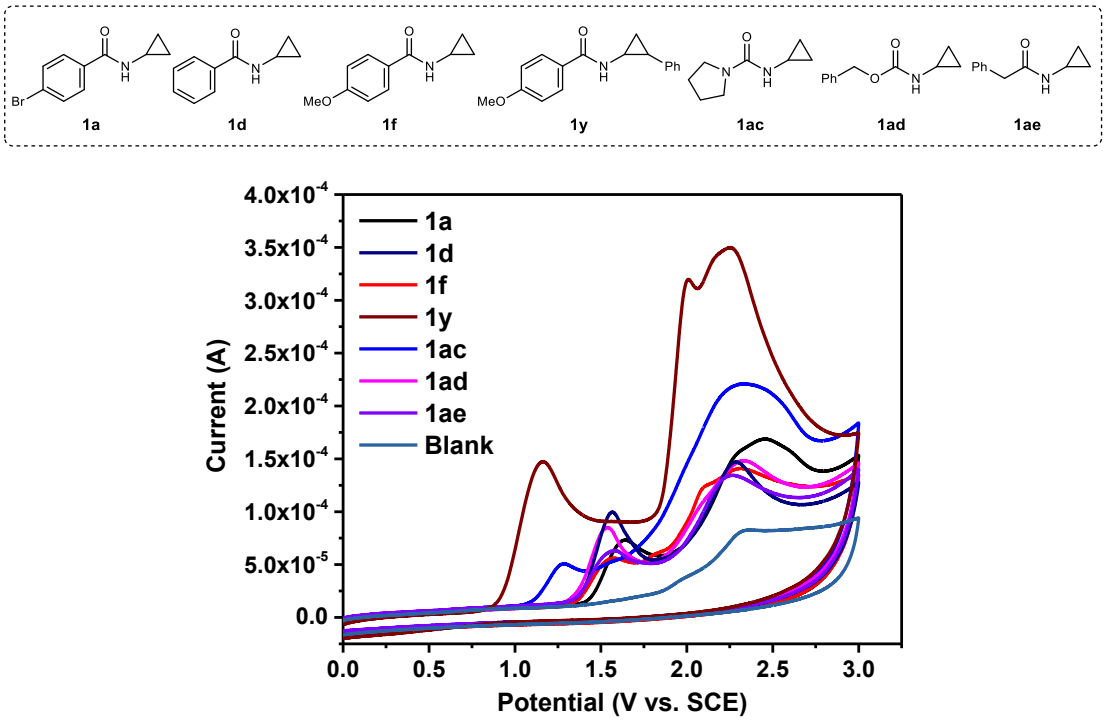


Figure S9. Cyclic voltammogram of selected aminocyclopropanes **[1 mM]** in MeCN

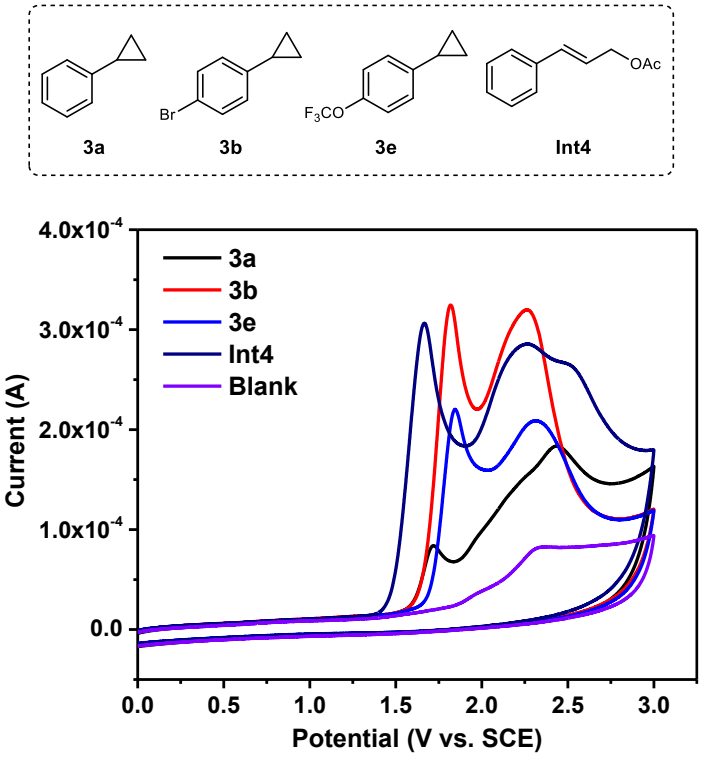


Figure S10. Cyclic voltammogram of selected arylcyclopropanes **[1 mM]** in MeCN

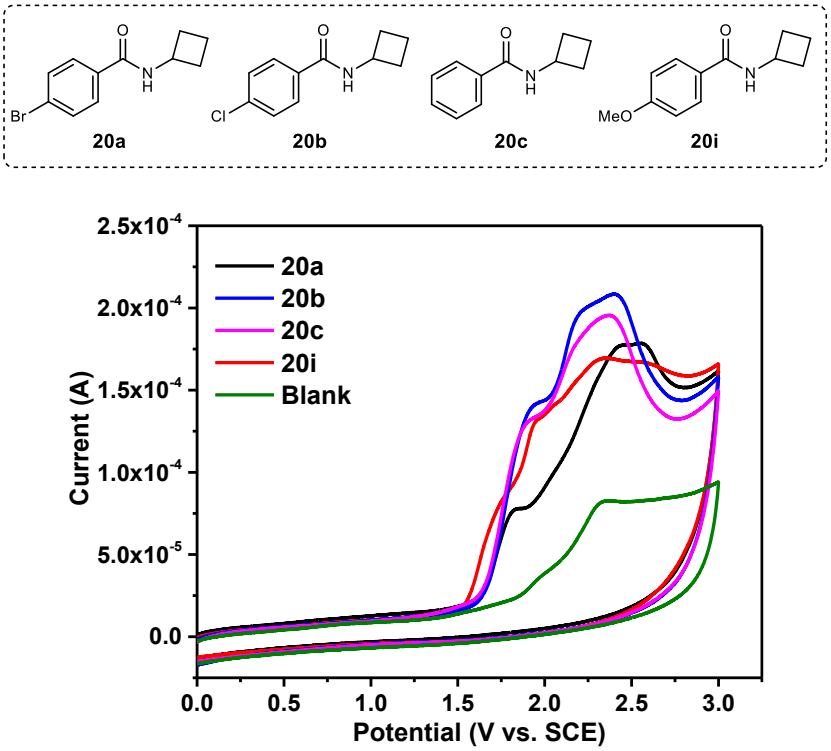
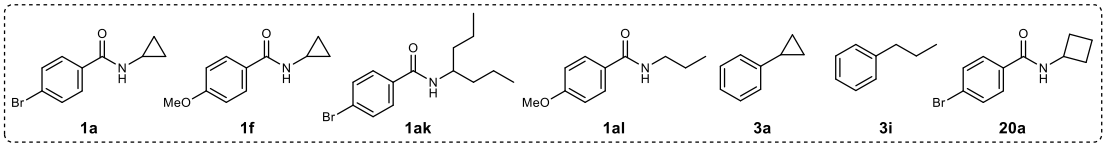


Figure S11. Cyclic voltammogram of selected aminocyclobutanes **[1 mM]** in MeCN



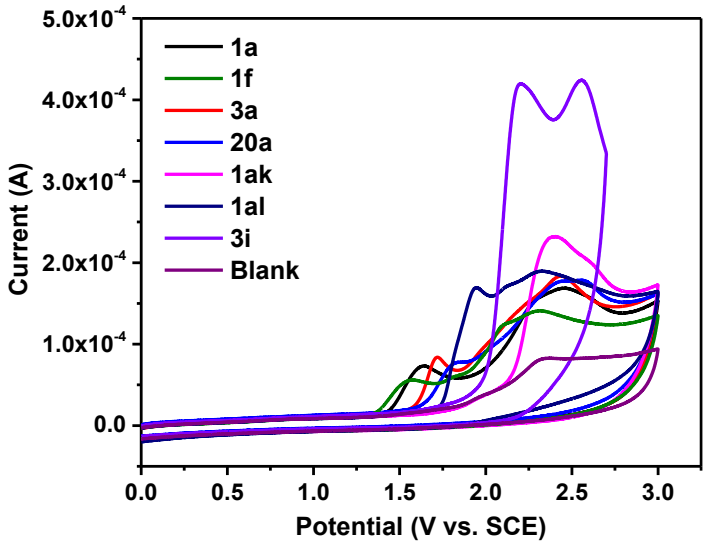


Figure S12. Cyclic voltammogram of selected substrates **[1 mM]** in MeCN

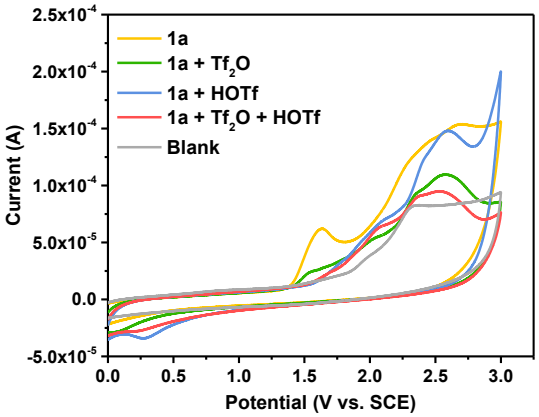


Figure S13. Cyclic voltammogram of **1a [1 mM]** in MeCN

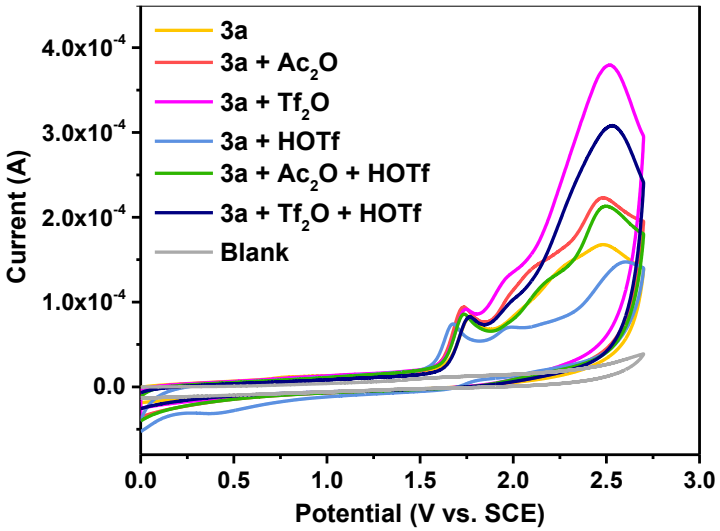


Figure S14. Cyclic voltammogram of **3a [1 mM]** in MeCN

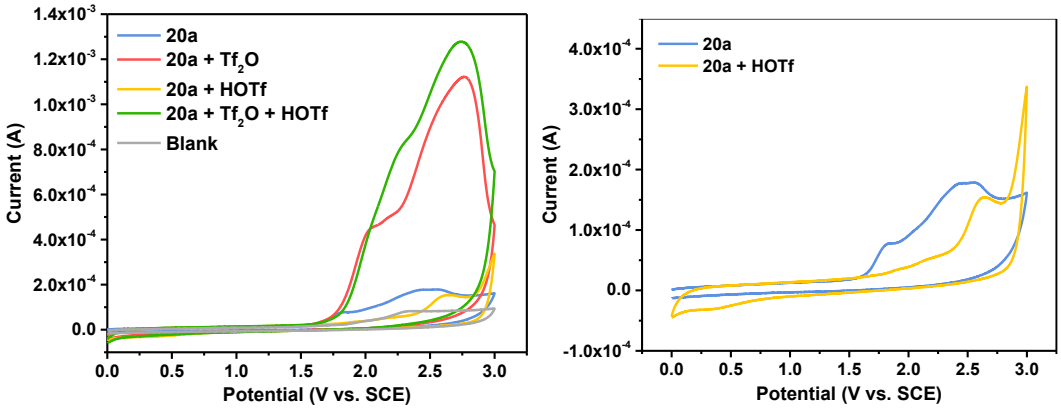
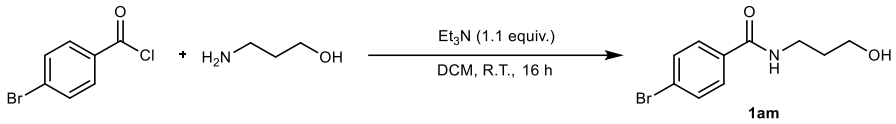


Figure S15. Cyclic voltammogram of **20a [1 mM]** in MeCN

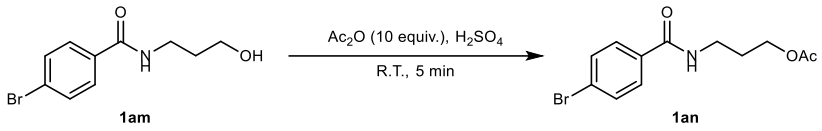
2.4.2 Verification of possible intermediates

Synthesis of possible intermediates



4-bromo-*N*-(3-hydroxypropyl)benzamide (1am):

Following **GP A**, to a solution of 3-aminopropan-1-ol (0.84 mL, 11.0 mmol, 1.1 equiv.) and triethylamine (1.5 mL, 11.0 mmol, 1.1 equiv.) in DCM (10.0 mL) was slowly added a solution of 4-bromobenzoyl chloride (2.2 g, 10.0 mmol) in DCM (10.0 mL) at 0 °C. The reaction mixture was stirred at room temperature for 16 h. Upon completion, the mixture was quenched by the addition of 1 M HCl (10 mL). The aqueous layer was then extracted with dichloromethane. The organic extract was washed with 1 M NaOH (10 mL) and brine (10 mL), dried over Na_2SO_4, filtered and concentrated in vacuo. **1am** was obtained by flash silica column chromatography (petroleum ether/ethyl acetate = 5:1) as a white solid (2.3 g, 88% yield).



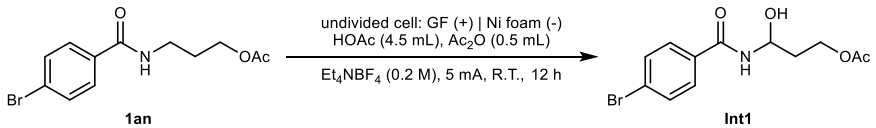
3-(4-bromobenzamido)propyl acetate (1an):

To a solution of **1am** (1.3 g, 5.0 mmol, 1.0 equiv.) and Ac_2O (4.7 mL, 50.0 mmol, 10.0 equiv.) was slowly added H_2SO_4 (0.01 mL). The reaction mixture was stirred at room temperature for 5 min. Upon completion, the mixture was filtered and concentrated in vacuo. **1an** was obtained by flash silica column chromatography (petroleum ether/ethyl acetate = 5 : 1) as a white solid (0.3 g, 22% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.63 (d, *J* = 8.5 Hz, 2H), 7.51 (d, *J* = 8.5 Hz, 2H), 6.86 (t, *J* = 6.1 Hz, 1H), 4.17 (t, *J* = 6.1 Hz, 2H), 3.46 (q, *J* = 6.4 Hz, 2H), 2.04 (s, 3H), 1.90 (p, *J* = 6.3 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 171.5, 166.5, 133.2, 131.7, 128.5, 126.0, 61.8, 36.6, 28.6, 20.9.

HRMS: calc. for $C_{12}H_{14}BrNNaO_3^+$ $(M+Na)^+$, 322.0049, found, 322.0051.



3-(4-bromobenzamido)-3-hydroxypropyl acetate (Int1):

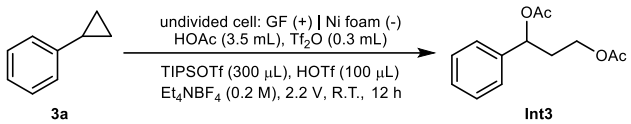
In a 10 mL oven-dried three-necked undivided cell equipped with a stir bar. To the cell was added **1an** (89.7 mg, 0.3 mmol), Et_4NBF_4 (0.2 M), HOAc (4.5 mL), Ac_2O (0.5 mL). The undivided cell was equipped with a graphite felt anode (15 mm × 15 mm × 6.5 mm) and Ni foam cathode (15 mm × 15 mm × 3 mm). Mixtures were stirred and electrolyzed at constant current of 5 mA at room temperature for 12 h. After completion of the reaction as monitored by TLC, the reaction mixture was poured into a saturated sodium carbonate solution (ca. 20 mL). The graphite felt anode was

washed with EtOAc (3×5 mL) and these washes were added to the reaction mixture. The aqueous layer was separated and extracted with EtOAc (3×15 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration in vacuo, the crude product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford target product **Int1** (53 mg, 57% yield).

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.64 (d, *J* = 8.6 Hz, 2H), 7.55 (d, *J* = 8.2 Hz, 2H), 7.36 (d, *J* = 7.5 Hz, 1H), 5.64 (d, *J* = 6.7 Hz, 1H), 4.41 (s, 1H), 4.33 – 4.23 (m, 2H), 2.09 – 2.07 (m, 2H), 2.06 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 171.0, 167.3, 132.2, 131.9, 128.6, 126.9, 72.7, 60.4, 34.0, 21.0.

HRMS: calc. for $C_{12}H_{14}BrNNaO_4^+$ $(M+Na)^+$, 337.9998, found, 337.9995.



1-phenylpropane-1,3-diyl diacetate (Int3):

In a 10 mL oven-dried three-necked undivided cell equipped with a stir bar. The undivided cell was equipped with a graphite felt anode (15 mm × 15 mm × 3 mm) and Ni foam cathode (15 mm × 15 mm × 3 mm). To the cell was added **3a** (0.6 mmol), Et_4NBF_4 (0.2 M), HOAc (3.5 mL), Tf_2O (0.3 mL), TIPSOTf (300 μL). The mixture was stirred for 1 min, and then HOTf (100 μL) was carefully added. The cell was sealed using a rubber septum and parafilm and was backfilled with N_2 atmosphere. Mixtures were stirred and electrolyzed at a controlled voltage of 2.2 V at room temperature for 12 h. After completion of the reaction as monitored by GC-MS, the reaction mixture was poured into a saturated sodium carbonate solution (ca. 20 mL). The graphite felt anode was washed with EtOAc (3×5 mL) and these washes were added to the reaction mixture. The aqueous layer was separated and extracted with EtOAc (3×15 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration in vacuo, the crude product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether : ethyl acetate = 6 : 1) to afford target product **Int3** (55.3 mg, 39% yield).

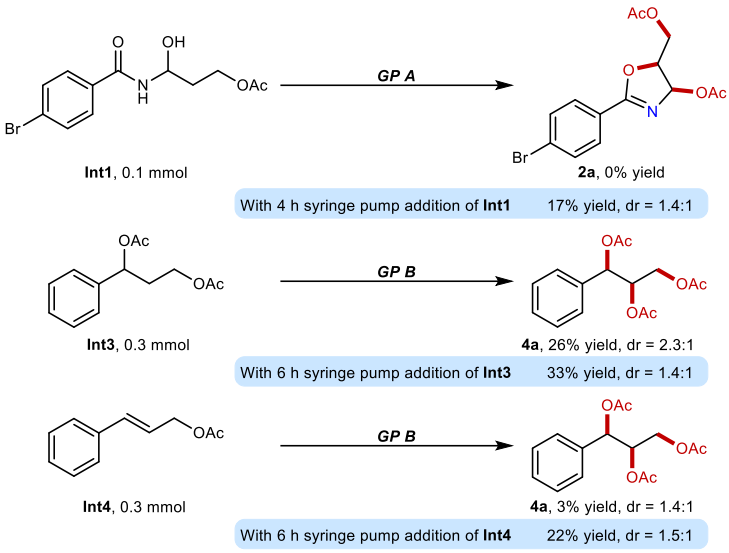
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.35 – 7.32 (m, 4H), 7.30 – 7.26 (m, 1H), 5.88 – 5.84 (m, 1H), 4.19 – 4.12 (m, 1H), 4.05 – 3.98 (m, 1H), 2.28 – 2.20 (m, 1H), 2.16 – 2.08 (m, 1H), 2.06 (s, 3H), 2.02 (s, 3H).

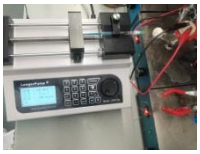
^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.8, 170.0, 139.8, 128.5, 128.1, 126.3, 72.7, 60.6, 35.1, 21.0, 20.7.

HRMS: calc. for $C_{13}H_{16}NaO_4^+$ $(M+Na)^+$, 259.0941, found, 259.0943.

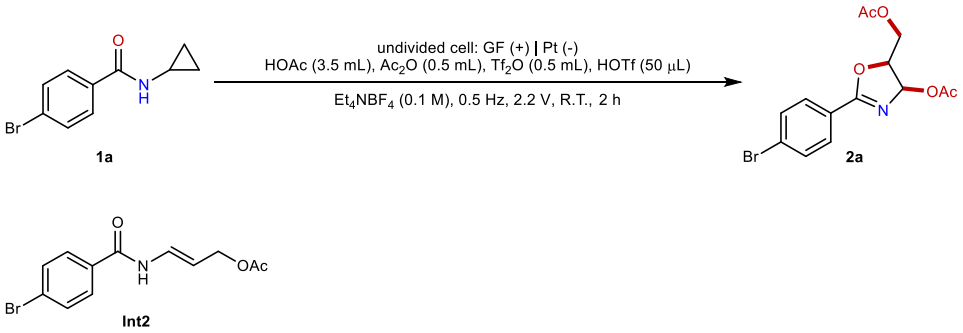
Some possible intermediates were tested

Site-selective ring-opening trioxygenation of substituted cyclopropanes





HR-MS analysis:



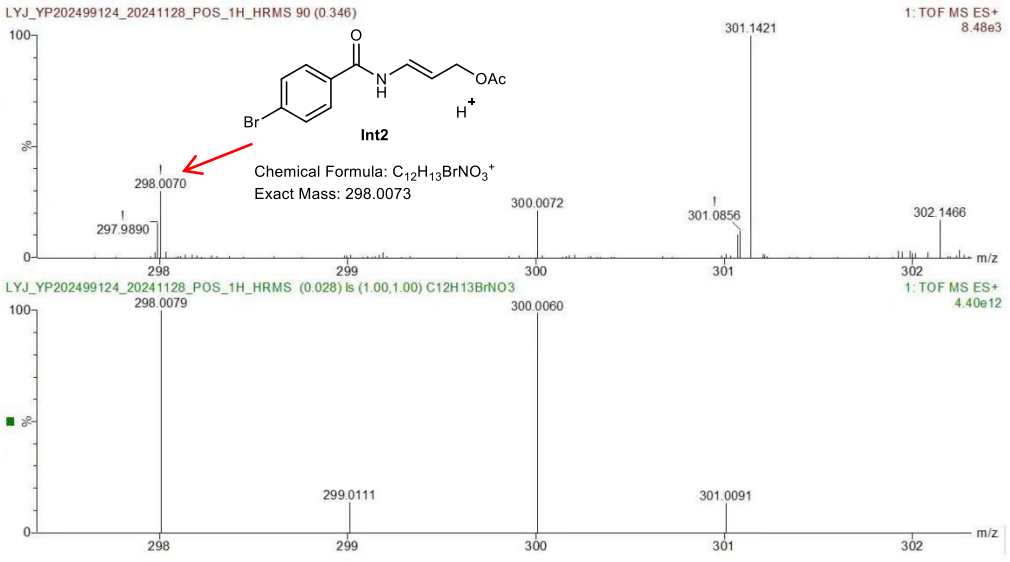
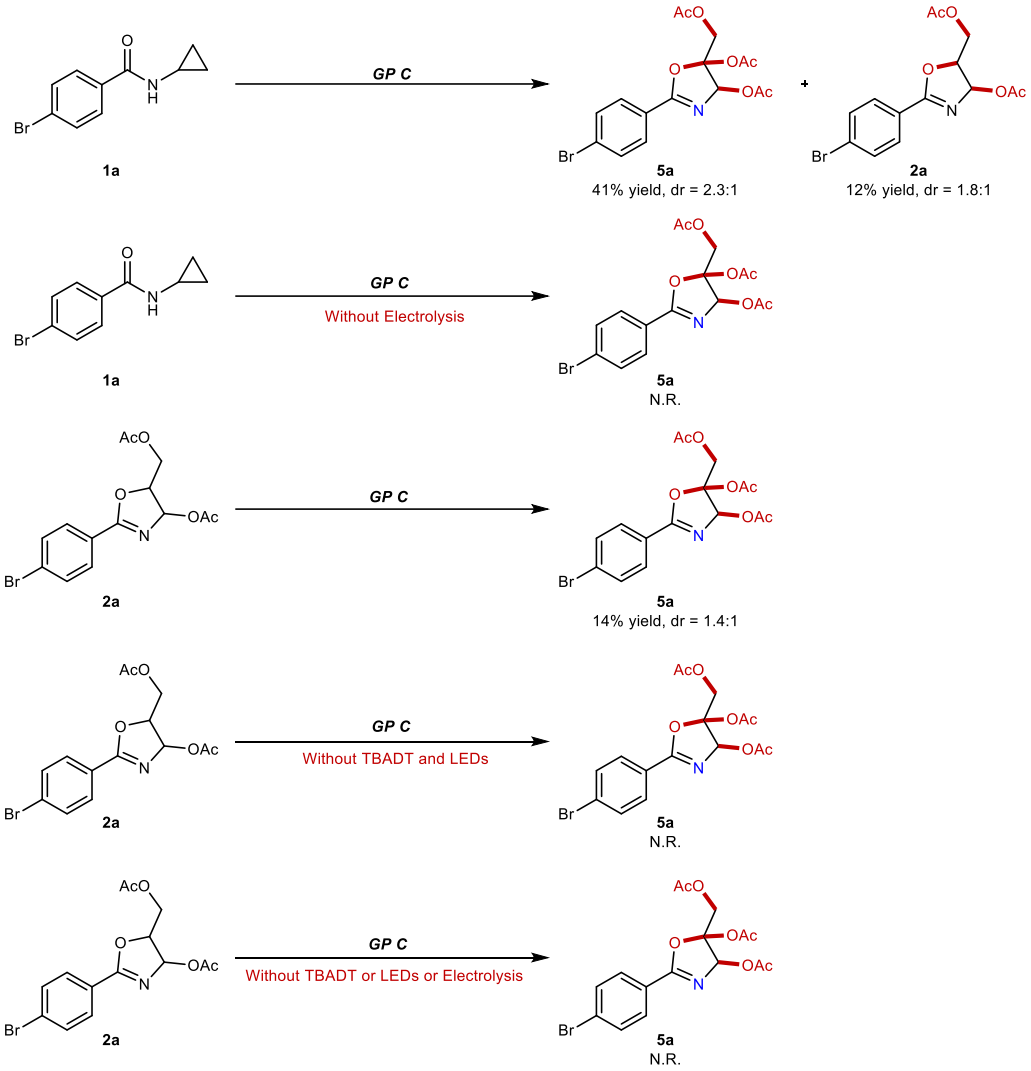
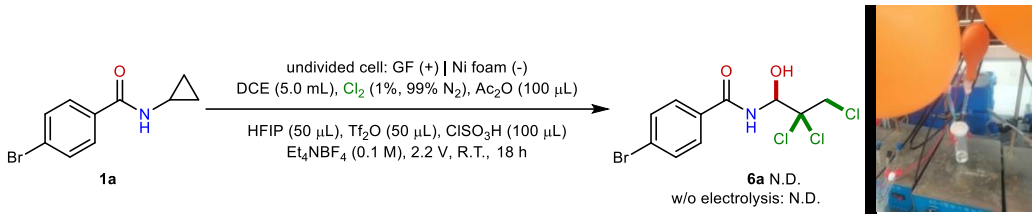


Figure S16. HR-MS analysis of **Int2**

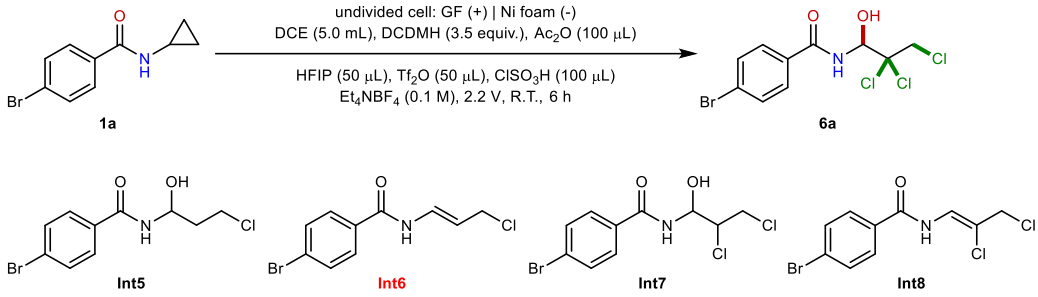
Site-selective ring-opening tetraoxygenation of substituted cyclopropanes



Site-selective ring-opening trichlorohydroxylation of substituted cyclopropanes



HR-MS analysis



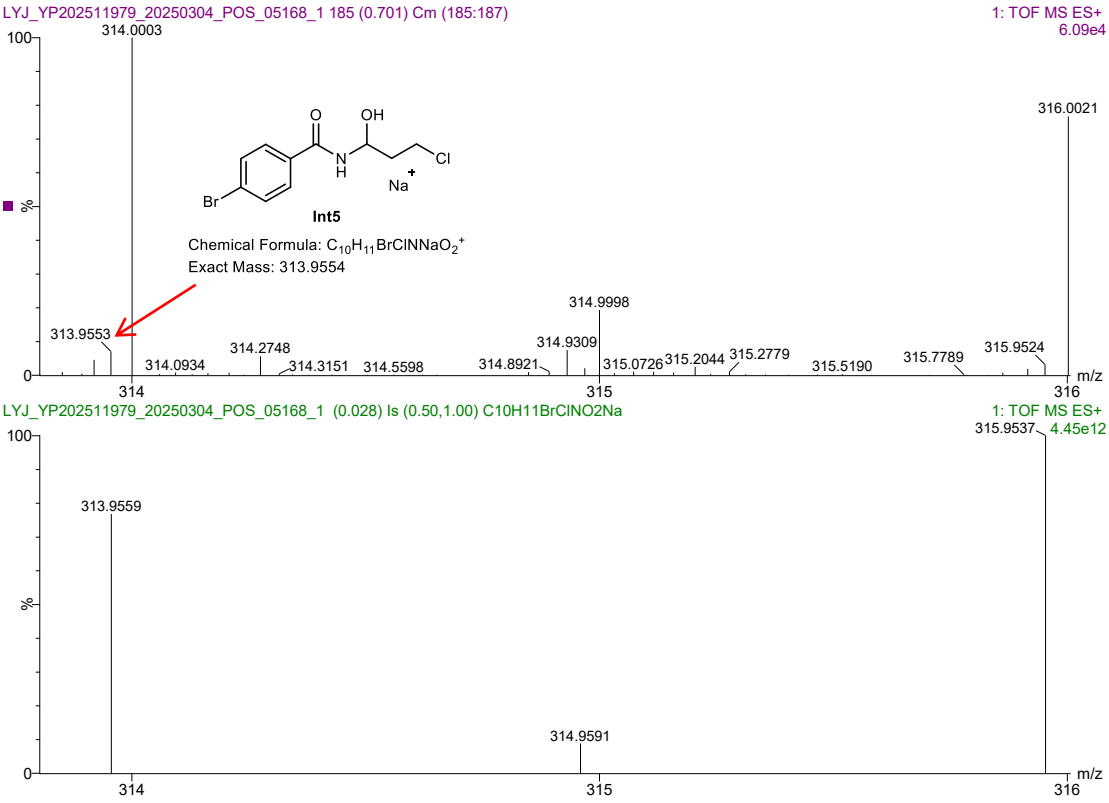


Figure S17. HR-MS analysis of **Int5**

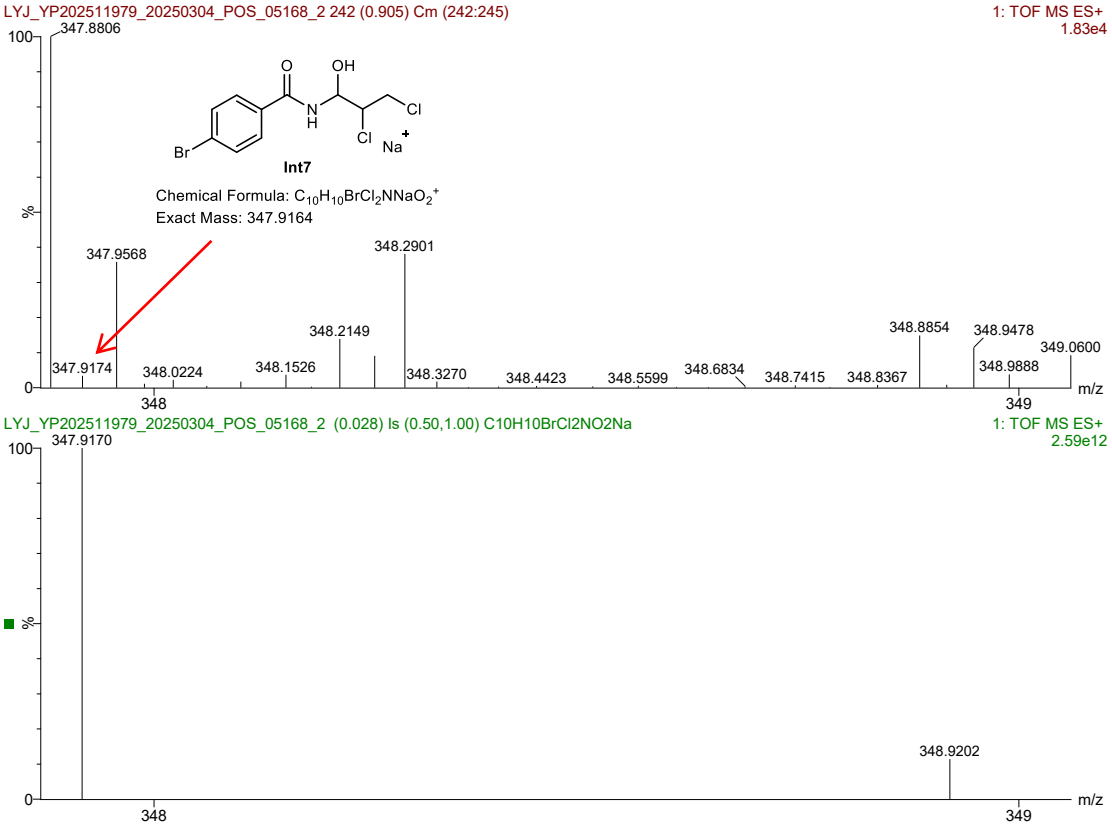


Figure S18. HR-MS analysis of **Int7**

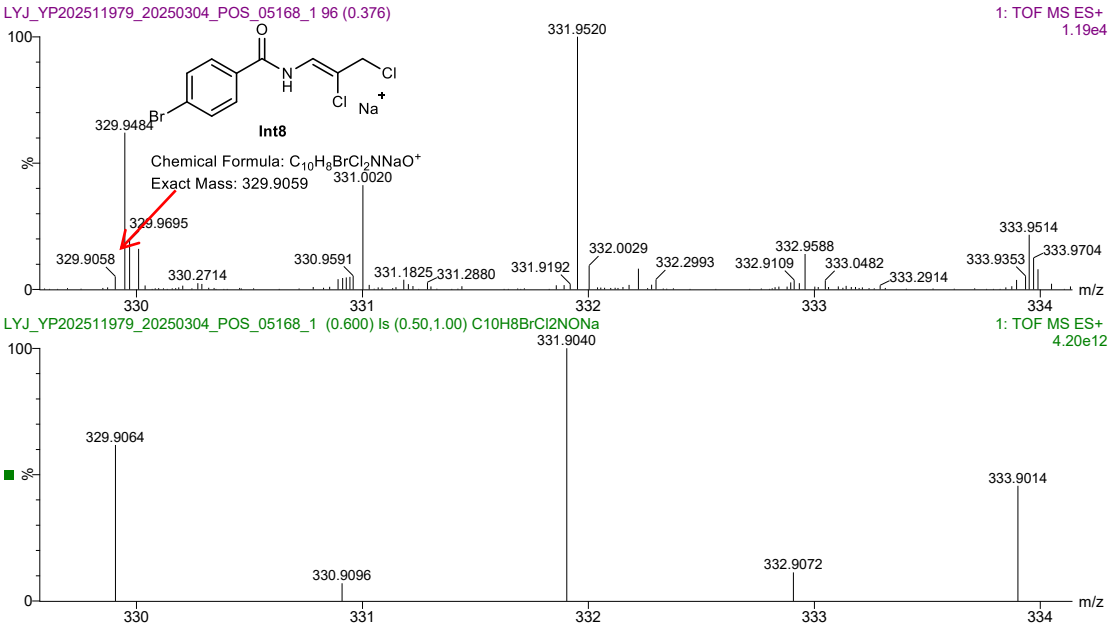
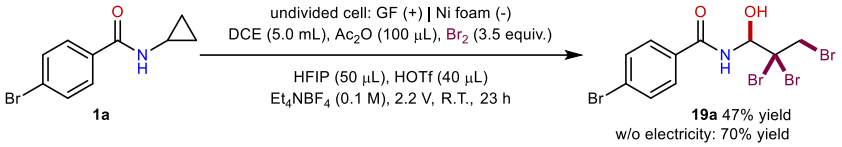
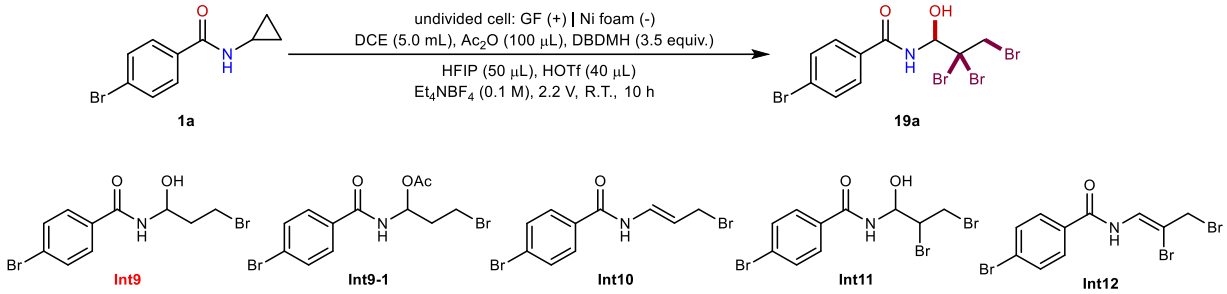


Figure S19. HR-MS analysis of **Int8**.

Site-selective ring-opening tribromohydroxylation of substituted cyclopropanes



HR-MS analysis



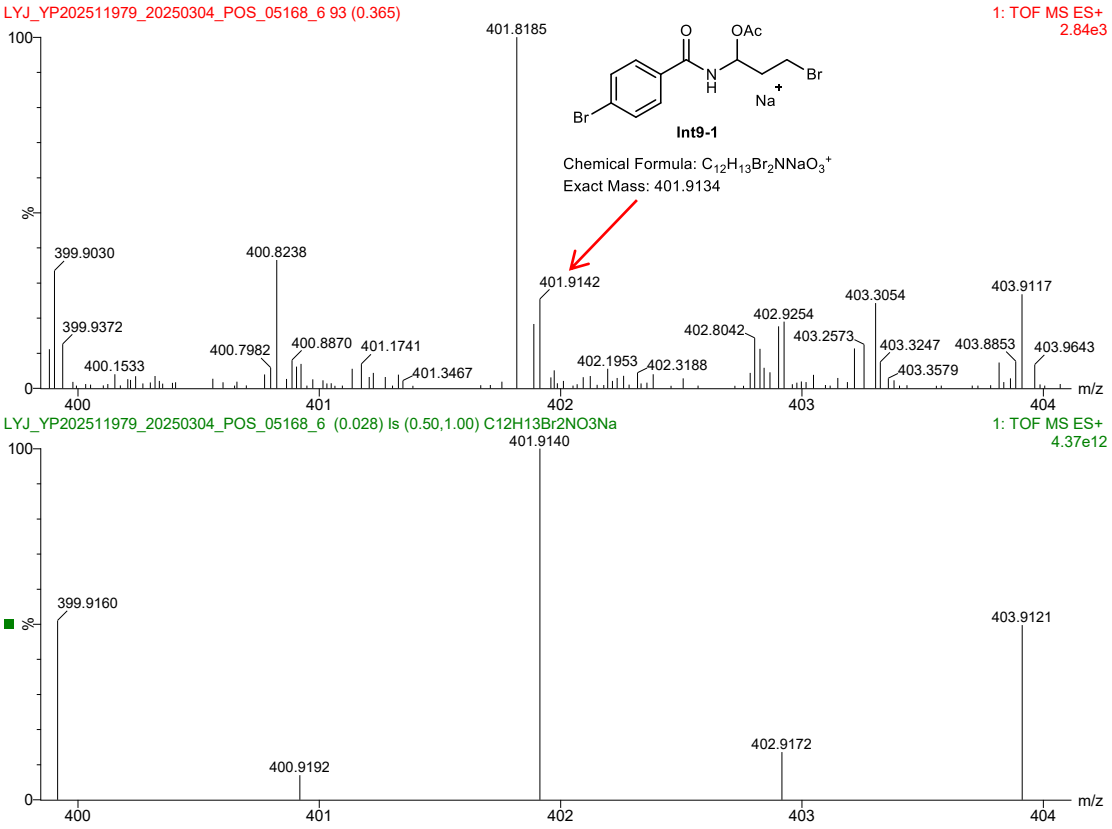


Figure S20. HR-MS analysis of **Int9-1**.

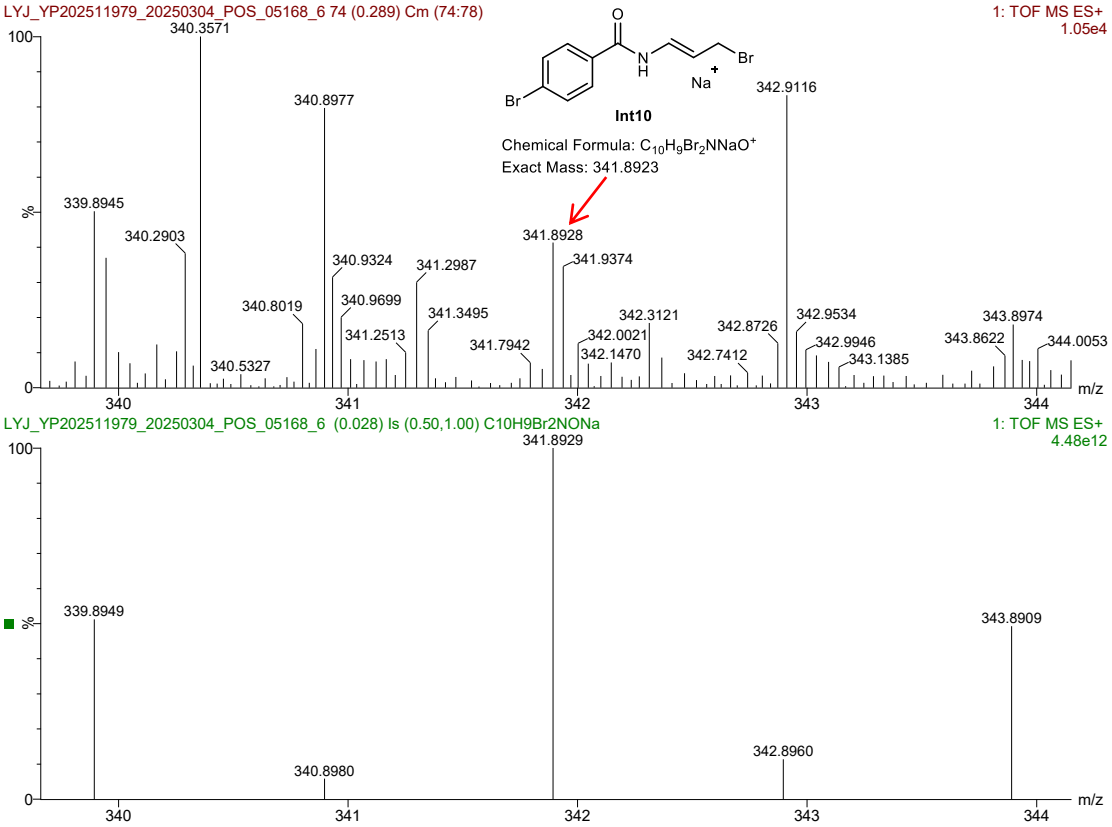


Figure S21. HR-MS analysis of **Int10**

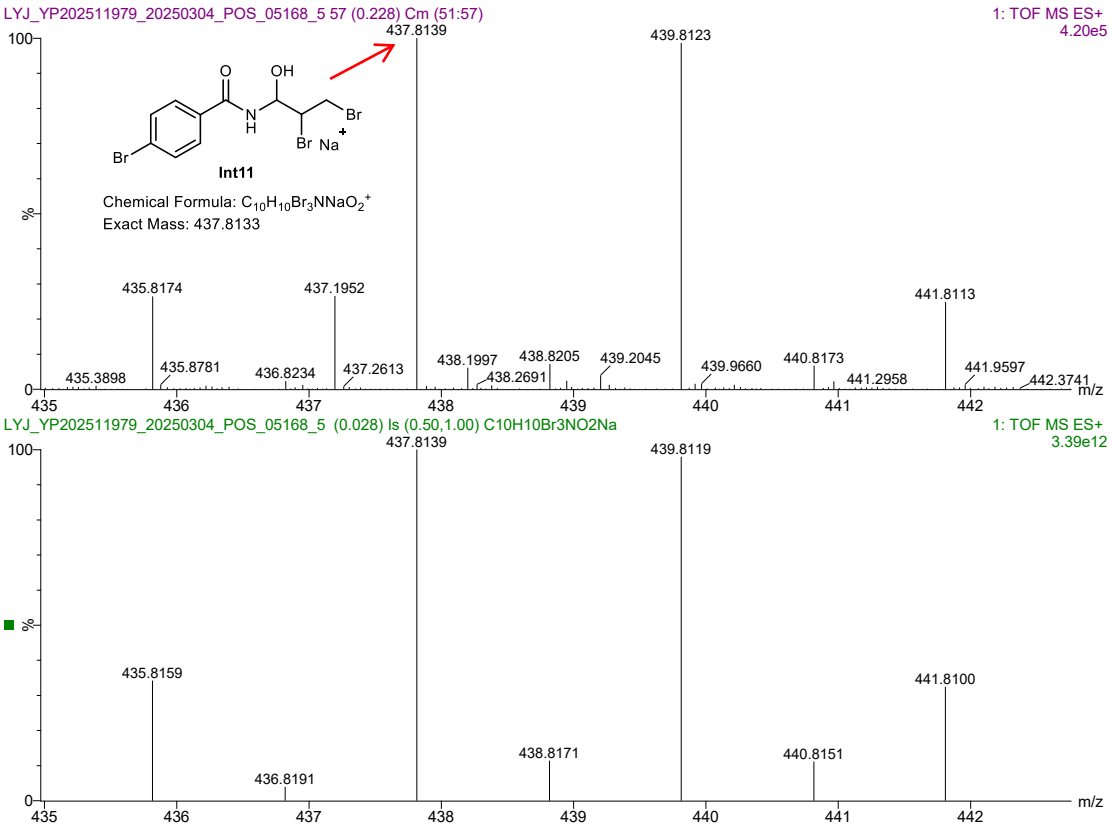


Figure S22. HR-MS analysis of **Int11**

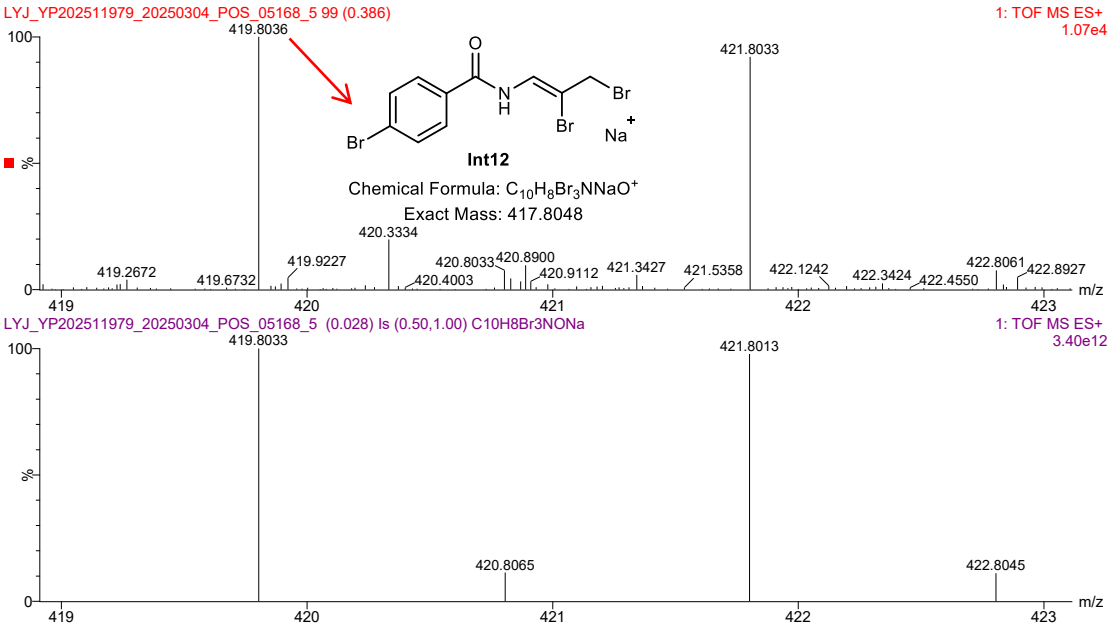
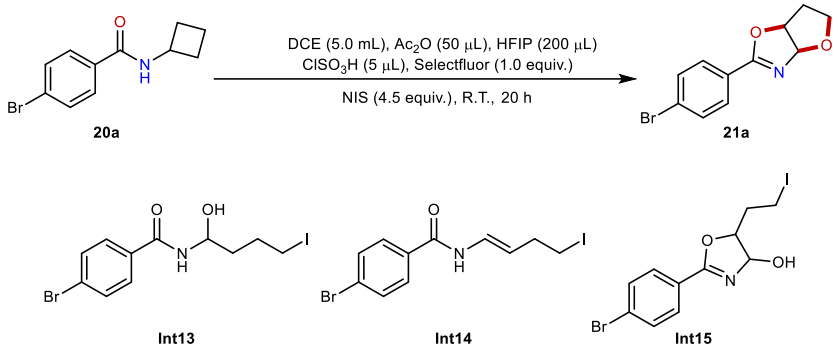


Figure S23. HR-MS analysis of **Int12**

Site-selective ring-opening trioxygenation of substituted cyclobutanes



HR-MS analysis

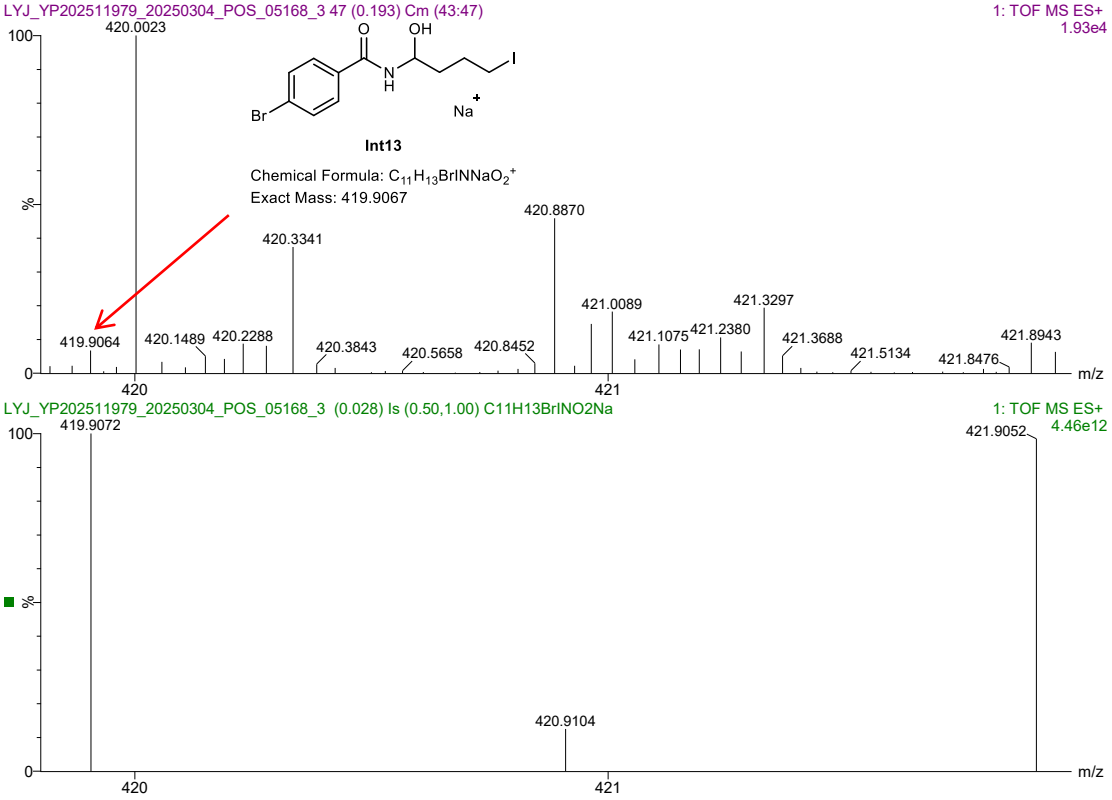


Figure S24. HR-MS analysis of **Int13**

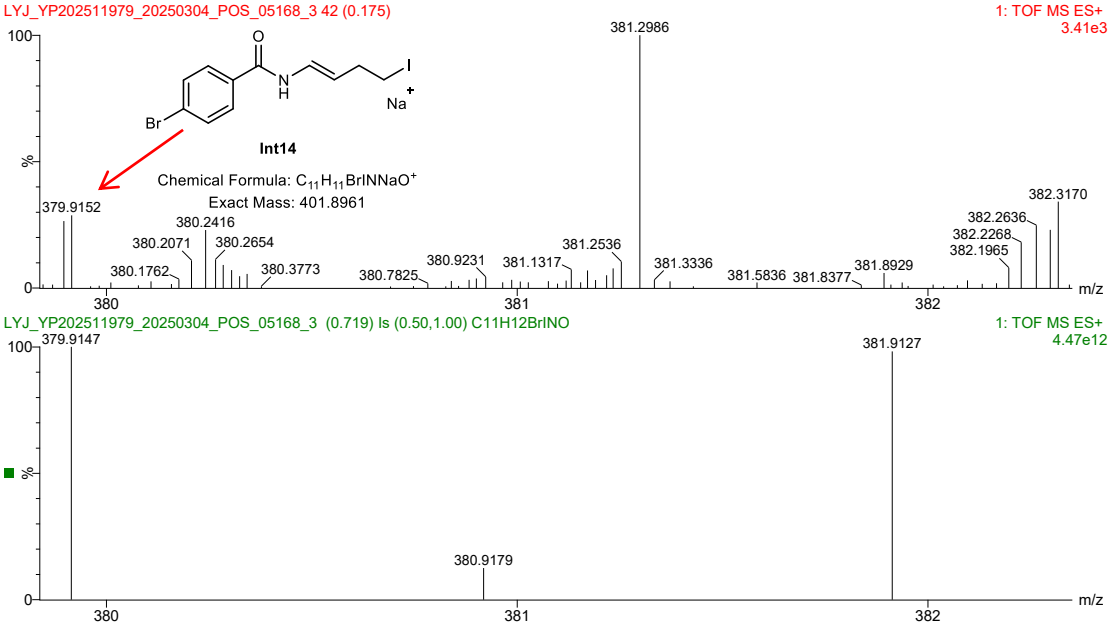


Figure S25. HR-MS analysis of **Int14**

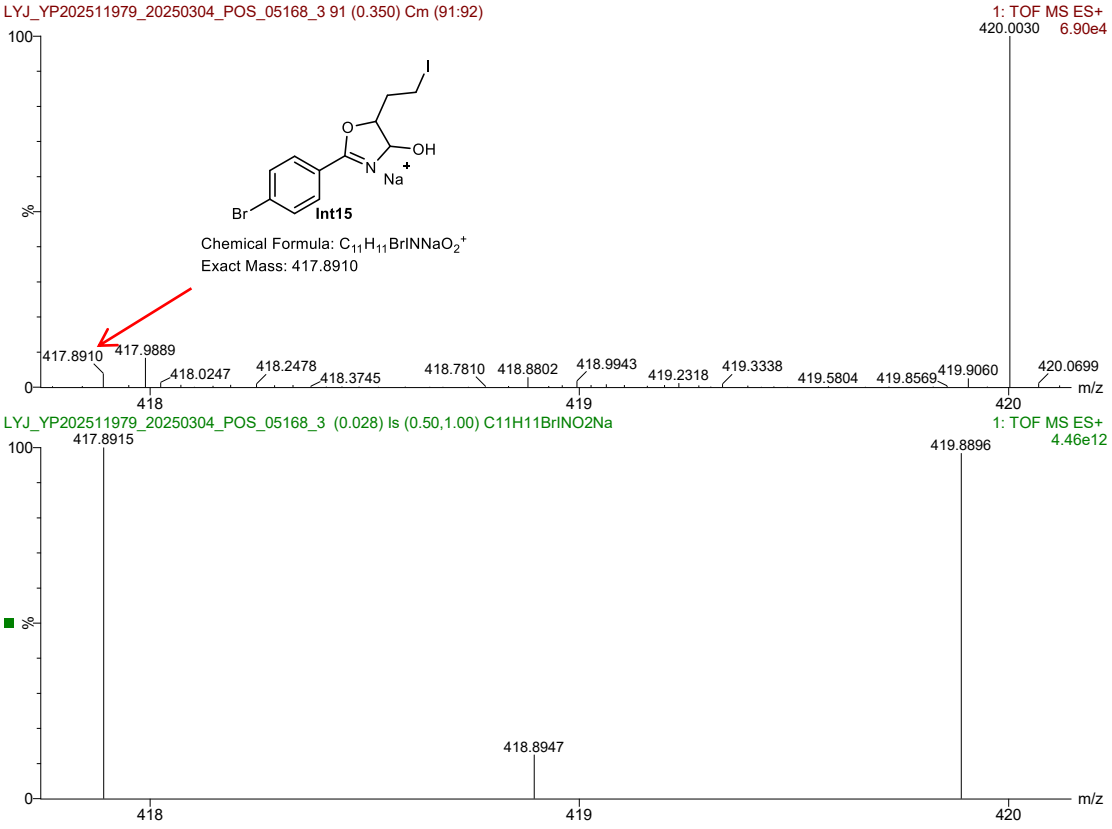


Figure S26. HR-MS analysis of **Int15**

2.4.3 Monitoring reaction processes using NMR

In situ monitoring of the reaction progress via NMR spectroscopy (performed without workup procedures by directly drying the reaction mixture followed by ^{13}C NMR measurement) revealed a discernible chemical shift variation in the carbonyl carbon resonance of the **1a**/**2a** system (Figure S27-29). This observation suggests a potential interaction between the **1a**/**2a** and the Tf_2O/HOTf reagent system, possibly through Lewis acid-base coordination or transient complex formation[13], which may contribute to stabilizing the reactive intermediates or products throughout the reaction process.

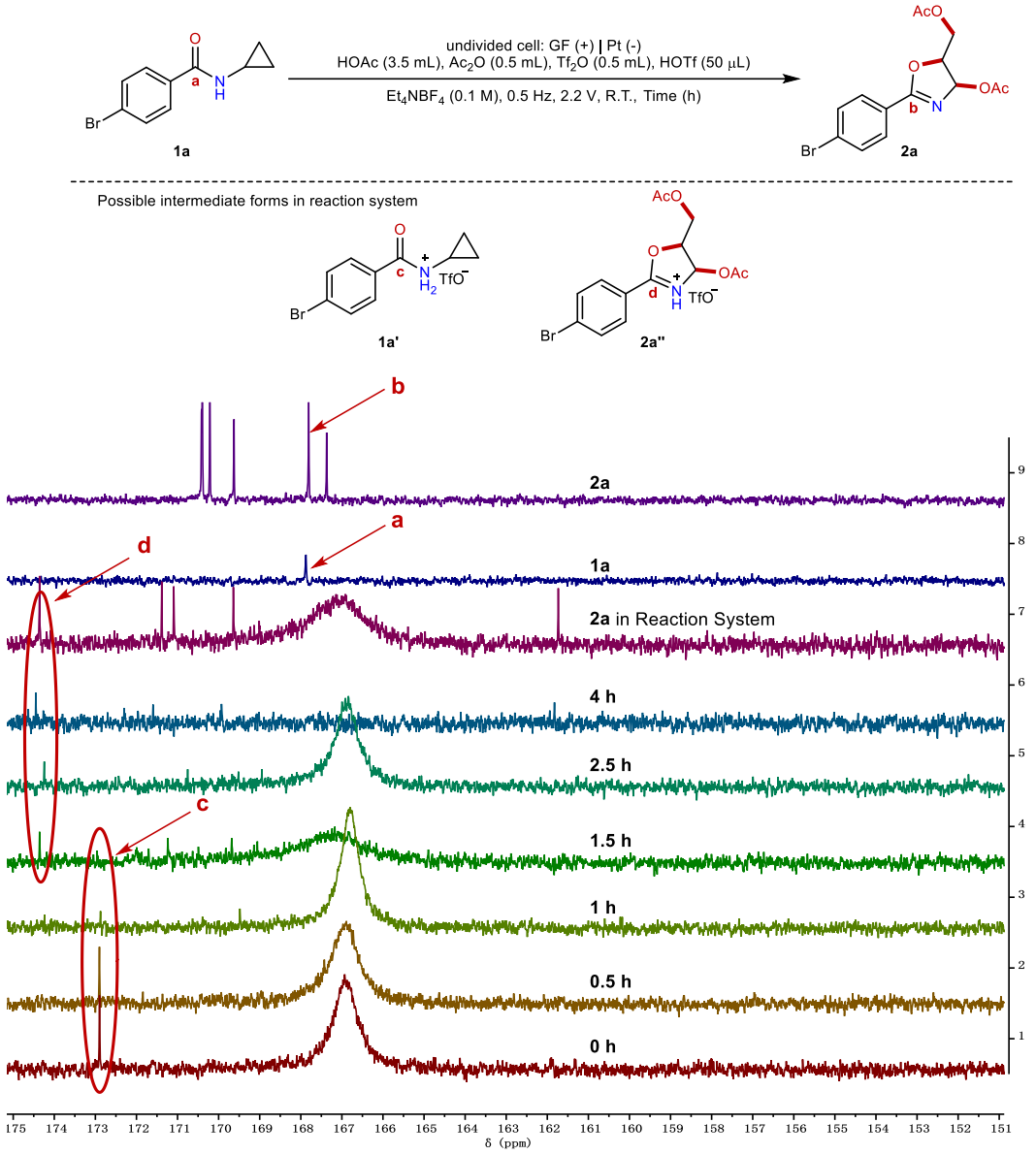


Figure S27. Monitoring reaction process by ^{13}C NMR

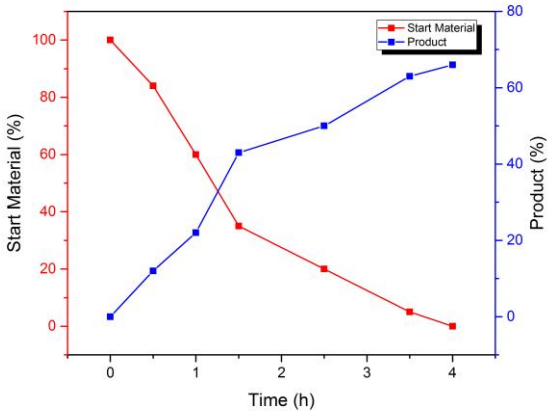


Figure S28. The line graph illustrates the changes in substrate consumption and product yield over time

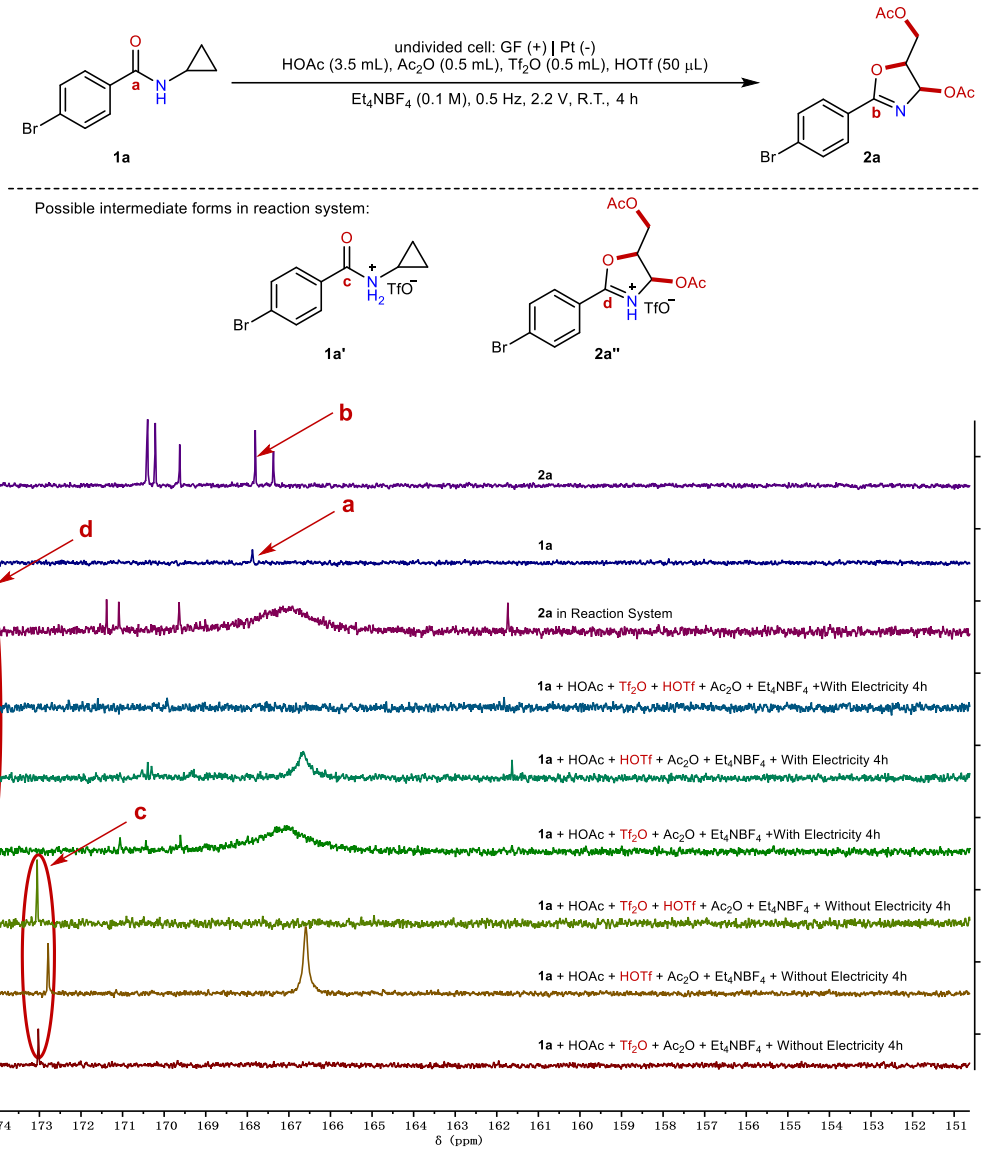
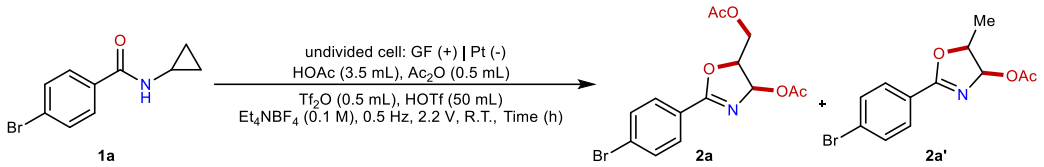


Figure S29. In situ ^{13}C NMR monitoring of the reaction progress with or without Tf_2O/HOTf

2.4.4 By-product analysis

GC-MS analysis combined with validation experiments revealed that compounds **2a'** and **4a'** were reaction by-products.



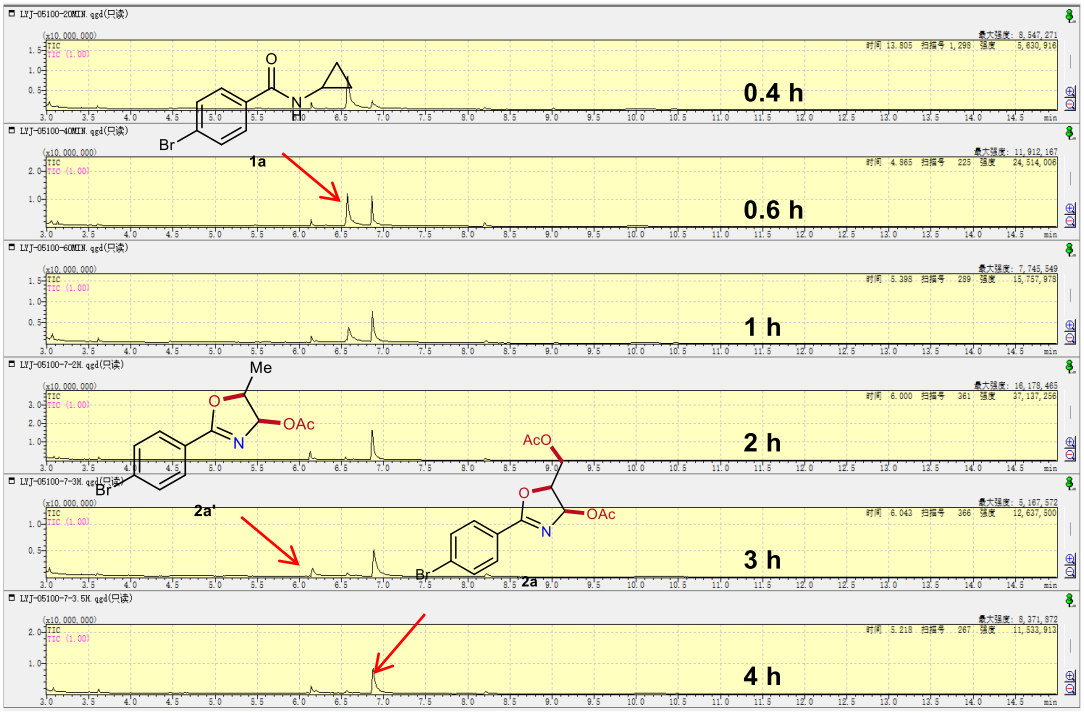
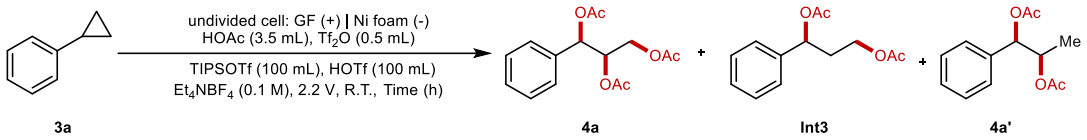


Figure S30. Mass spectrum of **1a**, **2a** and **2a'** peak from GCMS



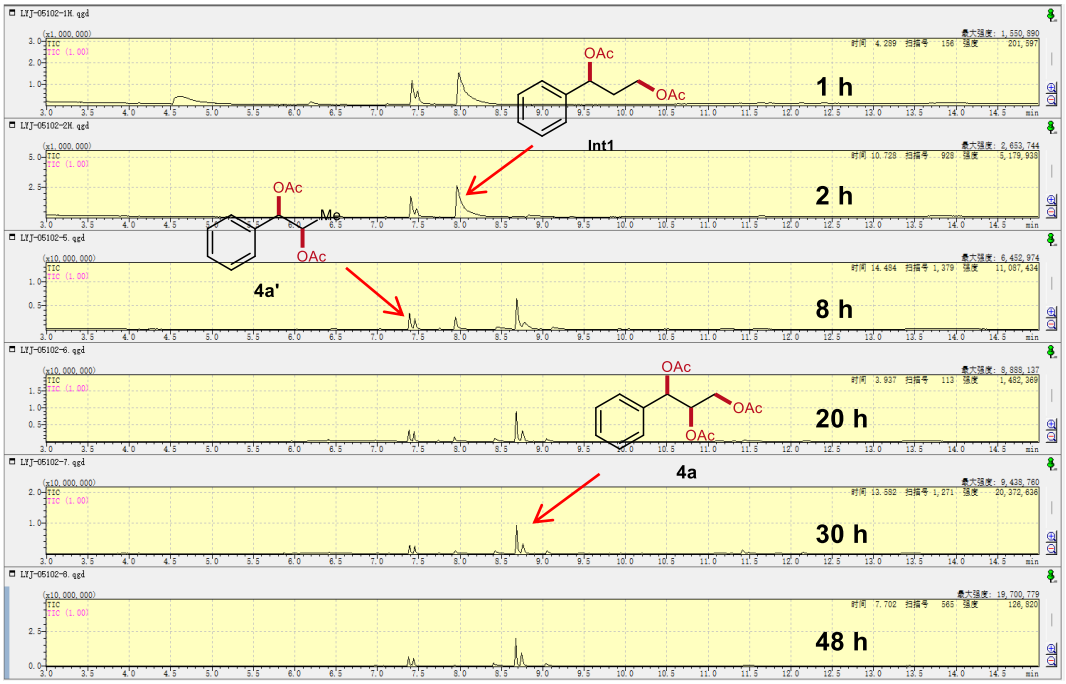
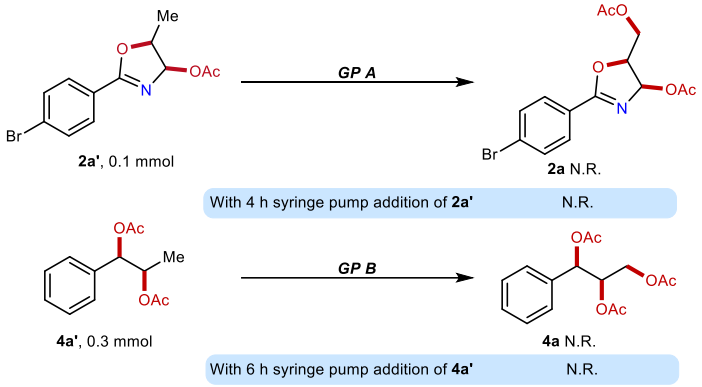
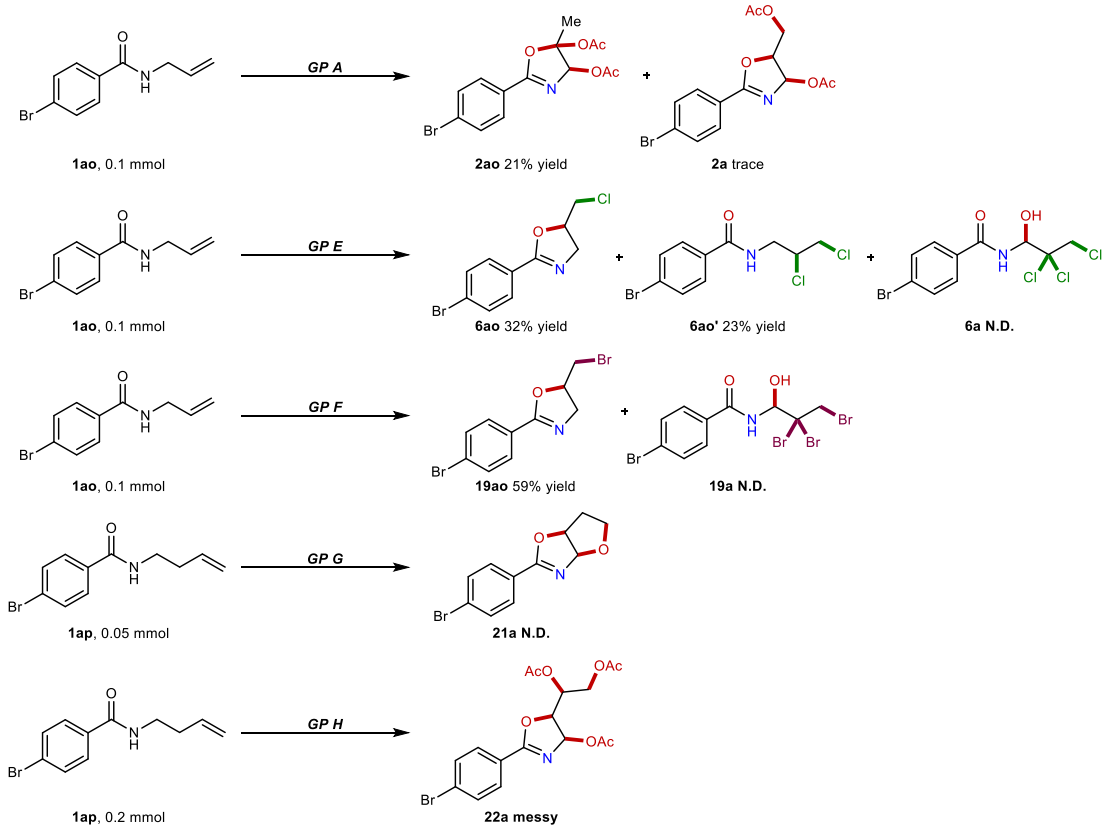


Figure S31. Mass spectrum of **4a**, **4a'** and **Int1** peak from GCMS



2.4.5 Other validation experiments



2.4.6 Proposed mechanisms

Site-selective ring-opening trichlorohydroxylation of substituted cyclopropanes

Based on mechanistic studies and literature precedents[14-16], a plausible pathway for the electrochemical trichlorohydroxylation of substituted cyclopropanes involves initial single-electron oxidation of the cyclopropane under electrocatalysis to generate radical cation intermediate **L**, which undergoes C–C bond cleavage in the presence of H_2O to form ring-opened intermediate **M**. Subsequent reaction of **M** with DCDMH (**N**) produces intermediate **O** and intermediate **5** (**Int5**), the latter undergoing reversible E1 elimination under strong acid catalysis to yield imine intermediate **P** and olefin intermediate **6** (**Int6**), collectively establishing an olefin release pool with **Int5**. Concurrently, N–Cl bond cleavage of DCDMH generates intermediate **Q** and chloronium species, which attack **Int6** to form chloronium intermediate **R**. Under the action of a nucleophile formed intermediate **7** (**Int7**), which undergoes reversible E1 elimination to create intermediate **8** (**Int8**) and intermediate **S**, forming a second olefin release pool. Finally, **Int8** undergoes electro-oxidative functionalization via chloronium ion attack to afford the trichlorohydroxylated product **6**.

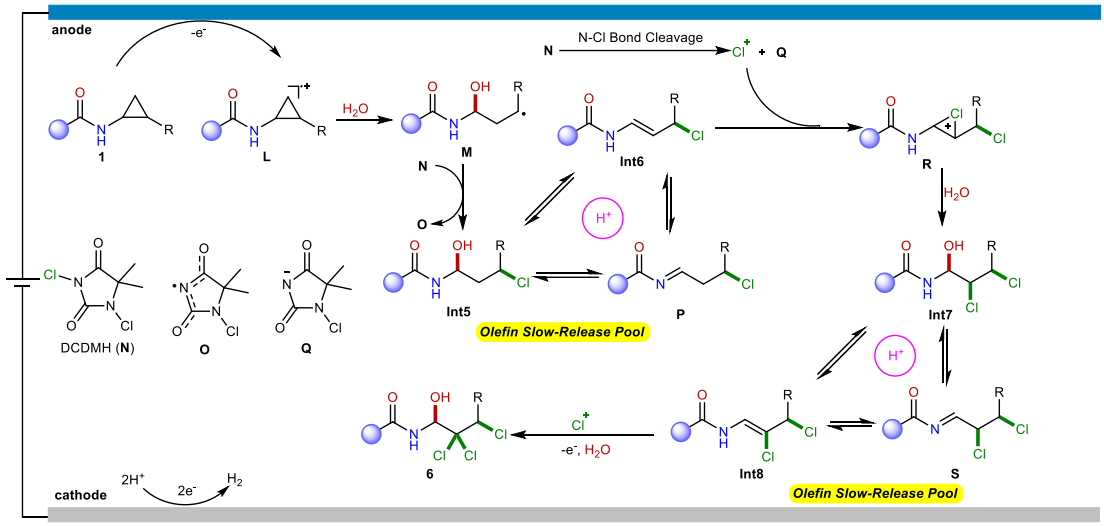


Figure S32. Possible mechanisms for trichlorohydroxylation of substituted cyclopropanes

Site-selective ring-opening tribromohydroxylation of substituted cyclopropanes

Based on mechanistic studies and literature precedents[17-20], a plausible pathway for the electrochemical tribromohydroxylation of substituted cyclopropanes involves the cleavage of the N–Br bond in DBDMH (**T**) to generate intermediate **U** and bromine cations, which are subsequently reduced to liquid bromine (Br_2) at the cathode. Under the influence of Br_2, intermediate **V** forms and undergoes N–Br bond cleavage, triggering the opening of the cyclopropane ring to yield an imine intermediate **W**. Tautomerization between enamines and imines then generates olefin intermediate **9** (**Int9**), which undergoes oxidative functionalization to form intermediate **8** (**Int8**). Collectively, **W**, **Int8**, and **Int9** constitute an olefin release pool. The **Int9** reacts with bromine to form a bromonium intermediate **X**, which generate intermediate **10** (**Int10**) in the presence of H_2O. This intermediate can reversibly eliminate via an E1 mechanism to produce intermediates **Y** and **11** (**Int11**), forming a second olefin release pool. **Int11** undergoes bromonium ion formation through bromine attack, followed by oxidative functionalization to yield the final product **19**. Alternatively, **int10** can be directly converted to product **19** in the presence of liquid bromine, completing the reaction network.

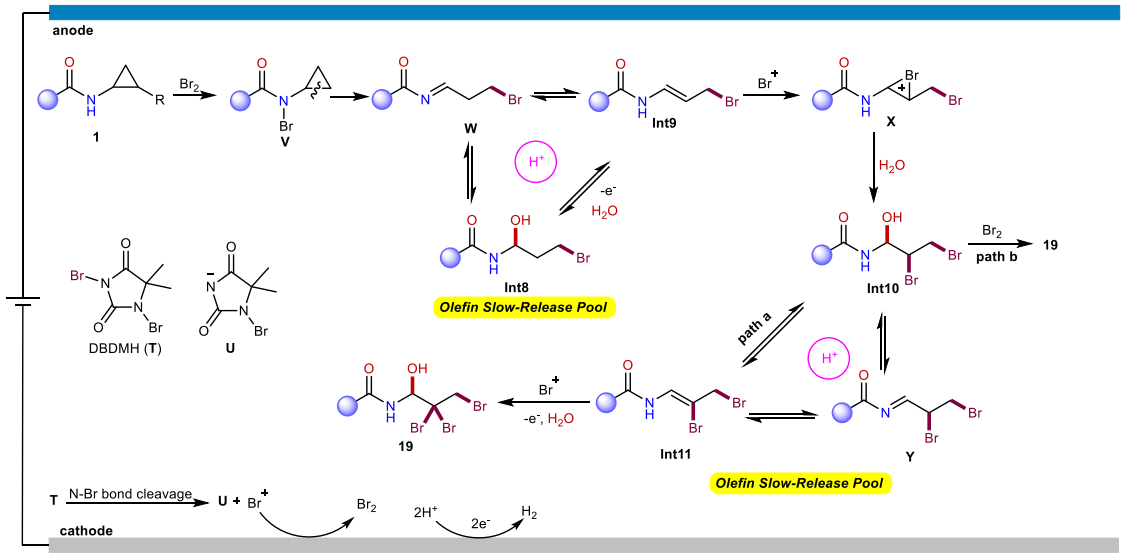


Figure S33. Possible mechanisms for tribromohydroxylation of substituted cyclopropanes

Site-selective ring-opening trioxygenation of substituted cyclobutanes

Based on mechanistic studies and literature precedents[20,21], a plausible pathway for the trioxygenation of substituted cyclobutanes is proposed. In the presence of NIS, intermediate **Z** forms and undergoes N–I bond cleavage, triggering the opening of the cyclopropane ring to yield an imine intermediate **AA**. Subsequent tautomerization between enamines and imines generates olefin intermediate **14**, which is converted to intermediate **13** (**Int13**) in the presence of H_2O. Collectively, **AA**, **Int13**, and **Int14** constitute an olefin slow-release pool. N–I bond cleavage of **AB** generates intermediate **AC** and iodine cation, which attack **Int14** to form iodonium intermediate **AD**. Nucleophilic attack on **AD** leads to intermediate **15** (**Int15**). Finally, **Int15** undergoes an SN2 reaction to afford the desired product.

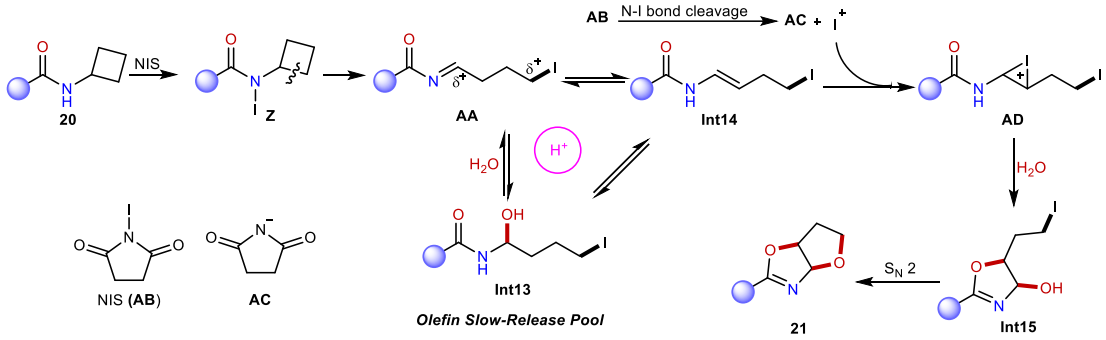


Figure S34. Possible mechanisms for trioxygenation of substituted cyclobutanes

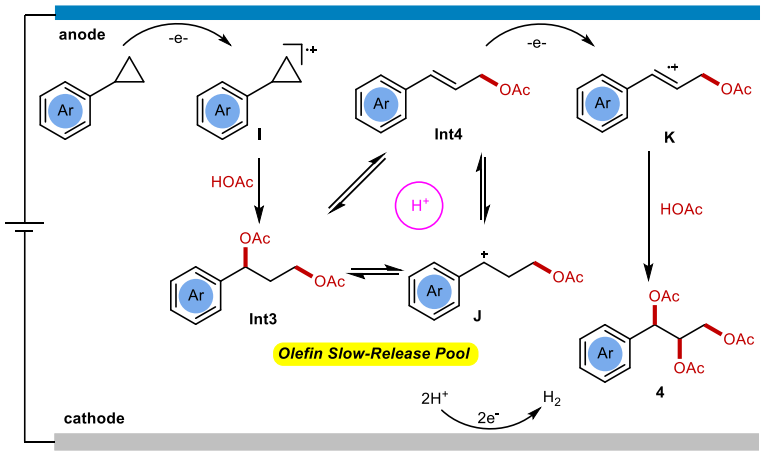
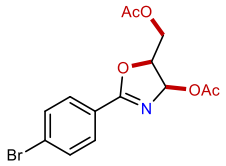


Figure S35. Possible mechanisms for trioxygenation of arylcyclopropanes

2.5 Characterization of new compounds



2a

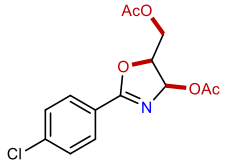
(4-acetoxy-2-(4-bromophenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2a):

Following **GP A**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 24.2 mg (68%, dr = 1.5:1) of **2a** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.88 – 7.85 (m, 2H), 7.58 – 7.55 (m, 2H), 6.81 (d, *J* = 7.4 Hz, 0.38H), 6.44 (d, *J* = 3.9 Hz, 0.58H), 4.91 – 4.83 (m, 0.39H), 4.76 – 4.67 (m, 0.62H), 4.52 (dd, *J* = 12.2, 3.5 Hz, 0.63H), 4.39 – 4.27 (m, 1.41H), 2.12 (s, 2H), 2.10 (s, 2H), 2.02 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.42, 170.40, 170.2, 169.6, 167.8, 167.4, 131.78, 131.76, 130.4 (major, minor), 127.54, 127.51, 125.1 (major, minor), 93.5, 89.8, 82.7, 79.1, 62.7, 60.9, 20.9, 20.8, 20.7, 20.6.

HRMS: calc. for $C_{14}H_{14}BrNNaO_5^+$ $(M+Na)^+$, 377.9948, found, 377.9943.



2b

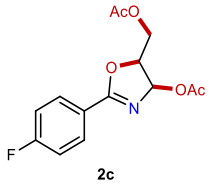
(4-acetoxy-2-(4-chlorophenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2b):

Following **GP A**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 19.0 mg (61%, dr = 1.5:1) of **2b** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.98 – 7,93 (m, 2H), 7.44 – 7.39 (m, 2H), 6.82 (d, *J* = 7.4 Hz, 0.38H), 6.45 (d, *J* = 3.9 Hz, 0.58H), 4.91 – 4.84 (m, 0.41H), 4.76 – 4.70 (m, 0.63H), 4.56 – 4.50 (m, 0.63H), 4.41 – 4.28 (m, 1.41H), 2.13 (s, 2H), 2.12 (s, 1H), 2.11 (s, 1H), 2.04 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.47, 170.45, 170.3, 169.7, 167.8, 167.3, 139.00, 138.98, 130.28, 130.27, 128.85, 128.83, 124.7 (major, minor), 93.5, 89.8, 82.8, 79.1, 62.7, 61.0, 21.0, 20.8, 20.7, 20.6.

HRMS: calc. for $C_{14}H_{14}ClNNaO_5^+$ $(M+Na)^+$, 334.0453, found, 334.0445.



2c

(4-acetoxy-2-(4-fluorophenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2c):

Following **GP A**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 17.4 mg (59%, dr = 1.5:1) of **2c** as a colorless oil.

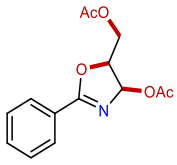
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.11 – 7.99 (m, 2H), 7.16 – 7.08 (m, 2H), 6.82 (d, *J* = 7.3 Hz,

0.37H), 6.45 (d, J = 3.9 Hz, 0.57H), 4.92 – 4.85 (m, 0.37H), 4.77 – 4.70 (m, 0.61H), 4.53 (dd, J = 12.2, 3.6 Hz, 0.61H), 4.40 – 4.25 (m, 1.33H), 2.13 (s, 2H), 2.12 (s, 2H), 2.04 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.48, 170.47, 170.3, 169.7, 167.7, 167.3, 165.5 (d, J_{CF} = 254.0 Hz, major, minor), 131.40 (d, J_{CF} = 9.3 Hz), 131.38 (d, J_{CF} = 9.3 Hz), 122.5 (d, J_{CF} = 3.2 Hz, major, minor), 115.74 (d, J_{CF} = 22.0 Hz), 115.72 (d, J_{CF} = 22.0 Hz), 93.6, 89.9, 82.8, 79.1, 62.8, 61.0, 21.0, 20.8, 20.7, 20.6.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -105.7, -105.8.

HRMS: calc. for $C_{14}H_{14}FNNaO_5^+$ $(M+Na)^+$, 318.0748, found, 318.0745.



2d

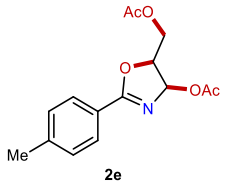
(4-acetoxy-2-phenyl-4,5-dihydrooxazol-5-yl)methyl acetate (2d):

Following **GP A**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 15.0 mg (54%, dr = 1.5:1) of **2d** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.06 – 7.99 (m, 2H), 7.58 – 7.51 (m, 1H), 7.47 – 7.41 (m, 2H), 6.84 (d, J = 7.4 Hz, 0.37H), 6.47 (d, J = 3.9 Hz, 0.57H), 4.91 – 4.84 (m, 0.39H), 4.77 – 4.71 (m, 0.60H), 4.54 (dd, J = 12.2, 3.6 Hz, 0.61H), 4.43 – 4.26 (m, 1.38H), 2.13 (s, 2H), 2.12 (s, 1H), 2.12 (s, 1H), 2.04 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5 (major, minor), 170.3, 169.8, 168.7, 168.2, 132.62, 132.61, 129.0, 128.9, 128.47, 128.46, 126.3, 126.2, 93.7, 90.0, 82.6, 78.9, 62.8, 61.0, 21.0, 20.9, 20.8, 20.6.

HRMS: calc. for $C_{14}H_{15}NNaO_5^+$ $(M+Na)^+$, 300.0842, found, 300.0842.



2e

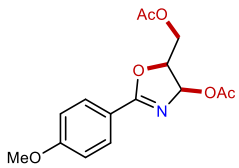
(4-acetoxy-2-(p-tolyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2e):

Following **GP A** with a reaction time of 5.5 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 17.2 mg (59%, dr = 1.4:1) of **2e** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.91 (d, J = 7.9 Hz, 2H), 7.24 (d, J = 8.0 Hz, 2H), 6.82 (d, J = 7.3 Hz, 0.41H), 6.45 (d, J = 3.2 Hz, 0.57H), 4.91 – 4.81 (m, 0.44H), 4.76 – 4.68 (m, 0.60H), 4.57 – 4.50 (m, 0.61H), 4.42 – 4.21 (m, 1.45H), 2.40 (s, 3H), 2.12 (s, 2H), 2.11 (s, 2H), 2.03 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.5 (major, minor), 170.4, 169.8, 168.8, 168.3, 143.28, 143.26, 129.19, 129.18, 128.94, 128.91, 123.5 (major, minor), 93.8, 90.0, 82.5, 78.8, 62.8, 61.1, 21.6 (major, minor), 21.0, 20.9, 20.7, 20.6.

HRMS: calc. for $C_{15}H_{17}NNaO_5^+$ $(M+Na)^+$, 314.0999, found, 314.1000.



2f

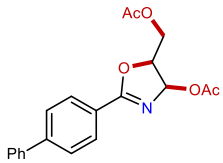
(4-acetoxy-2-(4-methoxyphenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2f):

Following **GP A** with a reaction time of 3 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 2:1) to afford 18.4 mg (60%, dr = 1.9:1) of **2f** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 8.8 Hz, 2H), 6.96 – 6.90 (m, 2H), 6.81 (d, *J* = 7.2 Hz, 0.35H), 6.43 (d, *J* = 3.8 Hz, 0.65H), 4.90 – 4.79 (m, 0.37H), 4.73 – 4.67 (m, 0.66H), 4.56 – 4.50 (m, 0.69H), 4.41 – 4.24 (m, 1.40H), 3.85 (s, 3H), 2.12 (s, 2H), 2.11 (s, 2H), 2.03 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.52, 170.51, 170.4, 169.8, 168.6, 168.1, 163.11, 163.10, 130.9, 130.8, 118.5 (major, minor), 113.83, 113.81, 93.8, 90.0, 82.5, 78.8, 62.9, 61.1, 55.4 (major, minor), 21.0, 20.9, 20.7, 20.6.

HRMS: calc. for $C_{15}H_{17}NNaO_6^+$ $(M+Na)^+$, 330.0948, found, 330.0952.



2g

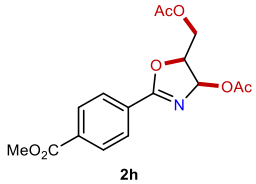
(2-([1,1'-biphenyl]-4-yl)-4-acetoxy-4,5-dihydrooxazol-5-yl)methyl acetate (2g):

Following **GP A** with a reaction time of 10 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 16.6 mg (47%, dr = 4.5:1) of **2g** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.10 (d, *J* = 8.2 Hz, 2H), 7.70 – 7.67 (m, 2H), 7.63 (d, *J* = 7.6 Hz, 2H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.40 (t, *J* = 7.3 Hz, 1H), 6.87 (d, *J* = 7.4 Hz, 0.18H), 6.50 (d, *J* = 3.8 Hz, 0.81H), 4.94 – 4.85 (m, 0.26H), 4.79 – 4.74 (m, 0.80H), 4.56 (dd, *J* = 12.2, 3.6 Hz, 0.85H), 4.45 – 4.27 (m, 1.13H), 2.15 (s, 2.50H), 2.14 (s, 1H), 2.06 (s, 2.50H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.6, 170.4, 168.6, 145.4, 139.9, 129.5, 128.9, 128.2, 127.2, 127.1, 125.0, 93.7, 82.6, 62.9, 21.1, 20.7.

HRMS: calc. for $C_{20}H_{19}NNaO_5^+$ $(M+Na)^+$, 376.1155, found, 376.1154.



2h

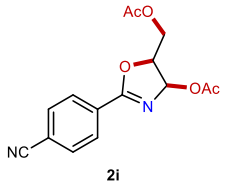
Methyl 4-(4-acetoxy-5-(acetoxymethyl)-4,5-dihydrooxazol-2-yl)benzoate (2h):

Following **GP A** with a reaction time of 10 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 18.4 mg (55%, dr = 1.5:1) of **2h** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.10 (s, 4H), 6.85 (d, *J* = 7.4 Hz, 0.39H), 6.49 (d, *J* = 4.0 Hz, 0.58H), 4.95 – 4.86 (m, 0.41H), 4.80 – 4.72 (m, 0.59H), 4.58 – 4.51 (m, 0.60H), 4.43 – 4.29 (m, 1.40H), 3.94 (s, 3H), 2.14 (s, 1.90H), 2.12 (s, 1.15H), 2.12 (s, 1.16H), 2.04 (s, 1.78H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.48, 170.45, 170.2, 169.6, 167.8, 167.4, 166.2 (major, minor), 133.64, 133.61, 130.2 (major, minor), 129.62, 129.60, 129.0, 128.9, 93.5, 89.8, 82.8, 79.2, 62.7, 60.9, 52.4 (major, minor), 21.0, 20.8, 20.7, 20.6.

HRMS: calc. for $C_{16}H_{17}NNaO_7^+$ $(M+Na)^+$, 358.0897, found, 358.0898.



2i

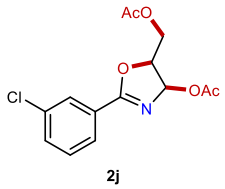
(4-acetoxy-2-(4-cyanophenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2i):

Following **GP A** with a reaction time of 15 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 12.4 mg (41%, dr = 1.8:1) of **2i** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.17 – 8.02 (m, 2H), 7.78 – 7.70 (m, 2H), 6.86 (d, *J* = 7.5 Hz, 0.35H), 6.49 (d, *J* = 4.0 Hz, 0.64H), 4.97 – 4.89 (m, 0.35H), 4.81 – 4.75 (m, 0.64H), 4.57– 4.51 (m, 0.66H), 4.42 – 4.29 (m, 1.34H), 2.14 (s, 2H), 2.13 (s, 1H), 2.12 (s, 1H), 2.04 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.44, 170.39, 170.1, 169.6, 166.9, 166.5, 132.3, 132.2, 130.34, 130.32, 129.48, 129.47, 117.9 (major, minor), 116.1, 116.0, 93.2, 89.6, 83.1, 79.4, 62.6, 60.8, 20.9, 20.8, 20.7, 20.6.

HRMS: calc. for $C_{15}H_{14}N_2NaO_5^+$ $(M+Na)^+$, 325.0795, found, 325.0796.



2j

(4-acetoxy-2-(3-chlorophenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2j):

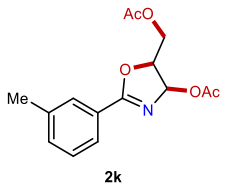
Following **GP A**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 16.2 mg (52%, dr = 1.5:1) of **2j** as a white solid.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.02 (t, *J* = 1.9 Hz, 1H), 7.93 – 7.88 (m, 1H), 7.55 – 7.47 (m, 1H), 7.41 – 7.36 (m, 1H), 6.83 (d, *J* = 7.4 Hz, 0.42H), 6.47 (d, *J* = 4.0 Hz, 0.63H), 4.92 – 4.86 (m, 0.39H), 4.76 – 4.69 (m, 0.65H), 4.53 (dd, *J* = 12.3, 3.6 Hz, 0.63H), 4.41 – 4.25 (m, 1.45H), 2.13 (s, 2H), 2.12 (s, 2H), 2.05 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.48, 170.45, 170.2, 169.7, 167.4, 167.0, 134.62, 134.61, 132.64, 132.62, 129.82, 129.80, 129.00, 128.96, 128.0 (major, minor), 127.0 (major, minor), 93.4, 89.7, 82.8, 79.2, 62.7, 60.9, 21.0, 20.8, 20.7, 20.6.

HRMS: calc. for $C_{14}H_{14}ClNNaO_5^+$ $(M+Na)^+$, 334.0453, found, 334.0450.

M.p.: 113.5 - 114.2 °C



2k

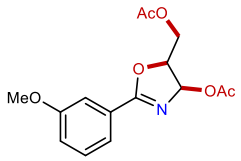
(4-acetoxy-2-(m-tolyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2k):

Following **GP A**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 16.3 mg (56%, dr = 2.2:1) of **2k** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.87 (s, 1H), 7.81 (d, *J* = 7.3 Hz, 1H), 7.39 – 7.29 (m, 2H), 6.84 (d, *J* = 7.3 Hz, 0.31H), 6.47 (d, *J* = 3.9 Hz, 0.68H), 4.90 – 4.84 (m, 0.34H), 4.75 – 4.70 (m, 0.72H), 4.56 – 4.51 (m, 0.70H), 4.41 – 4.29 (m, 1.40H), 2.39 (s, 3H), 2.13 (s, 2H), 2.12 (s, 1H), 2.12 (s, 1H), 2.05 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.6 (major, minor), 170.4, 169.8, 168.9, 168.5, 138.3 (major, minor), 133.4 (major, minor), 129.52, 129.49, 128.39, 128.38, 126.10, 126.09, 126.06, 126.0, 93.6, 89.9, 82.5, 78.8, 62.9, 61.1, 21.2 (major, minor), 21.1, 20.9, 20.8, 20.7.

HRMS: calc. for $C_{15}H_{17}NNaO_5^+$ $(M+Na)^+$, 314.0999, found, 314.1003.



2l

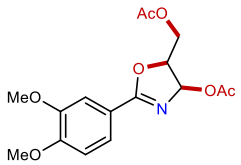
(4-acetoxy-2-(3-methoxyphenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2l):

Following **GP A** with a reaction time of 2 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 2:1) to afford 12.0 mg (39%, dr = 1.4:1) of **2l** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.63 – 7.59 (m, 1H), 7.55 (s, 1H), 7.34 (t, *J* = 7.9 Hz, 1H), 7.10 (d, *J* = 2.8 Hz, 1H), 6.84 (d, *J* = 7.3 Hz, 0.42H), 6.47 (d, *J* = 3.9 Hz, 0.58H), 4.92 – 4.84 (m, 0.44H), 4.75 – 4.70 (m, 0.58H), 4.54 (dd, *J* = 12.2, 3.6 Hz, 0.61H), 4.42 – 4.28 (m, 1.47H), 3.85 (s, 3H), 2.14 (s, 2H), 2.12 (s, 1H), 2.12 (s, 1H), 2.05 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.5 (major, minor), 170.3, 169.8, 168.7, 168.2, 159.5 (major, minor), 129.6, 129.5, 127.5 (major, minor), 121.40, 121.39, 119.5, 119.4, 113.21, 113.17, 93.7, 90.0, 82.6, 78.9, 62.8, 61.0, 55.48, 55.46, 21.0, 20.9, 20.8, 20.6.

HRMS: calc. for $C_{15}H_{17}NNaO_6^+$ $(M+Na)^+$, 330.0948, found, 330.0954.



2m

(4-acetoxy-2-(3,4-dimethoxyphenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2m):

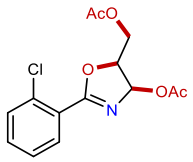
Following **GP A** at a controlled voltage of 2.0 V for 2 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 2:1) to afford 15.5 mg (46%, dr = 1.4:1) of **2m** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 9.1 Hz, 1H), 7.52 (s, 1H), 6.86 (d, *J* = 8.7 Hz, 1H), 6.79 (d, *J* = 7.2 Hz, 0.40H), 6.42 (d, *J* = 3.7 Hz, 0.55H), 4.89 – 4.80 (m, 0.45H), 4.72 – 4.65 (m, 0.57H), 4.51 (dd, *J* = 12.1, 3.6 Hz, 0.61H), 4.40 – 4.22 (m, 1.46H), 3.90 (s, 6H), 2.10 (s, 2H), 2.09 (s, 2H), 2.01 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.44, 170.41, 170.3, 169.8, 168.5, 168.0, 152.7 (major, minor), 148.7 (major, minor), 122.7 (major, minor), 118.6 (major, minor), 111.2 (major, minor),

110.33, 110.30, 93.7, 90.0, 82.5, 78.8, 62.7, 61.0, 55.99, 55.96, 55.9 (major, minor), 21.0, 20.8, 20.7, 20.6.

HRMS: calc. for $C_{16}H_{19}NNaO_7^+$ $(M+Na)^+$, 360.1054, found, 360.1059.



2n

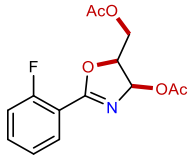
(4-acetoxy-2-(2-chlorophenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2n):

Following **GP A**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 15.9 mg (51%, dr = 1.9:1) of **2n** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.84 (d, *J* = 7.8 Hz, 1H), 7.50 – 7.39 (m, 2H), 7.33 (t, *J* = 7.6 Hz, 1H), 6.88 (d, *J* = 7.6 Hz, 0.35H), 6.51 (d, *J* = 3.6 Hz, 0.65H), 4.94 – 4.87 (m, 0.36H), 4.76 – 4.71 (m, 0.66H), 4.55 (dd, *J* = 12.3, 3.3 Hz, 0.69H), 4.47 – 4.29 (m, 1.61H), 2.14 (s, 2H), 2.13 (s, 1H), 2.10 (s, 1H), 2.07 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 170.5 (major, minor), 170.2, 169.7, 167.6, 167.0, 133.9 (major, minor), 132.59, 132.57, 131.8, 131.7, 131.0, 130.9, 126.63, 126.60, 126.02, 125.99, 93.4, 89.9, 82.5, 78.8, 62.7, 60.8, 21.0, 20.8, 20.73, 20.65.

HRMS: calc. for $C_{14}H_{14}ClNNaO_5^+$ $(M+Na)^+$, 334.0453, found, 334.0452.



2o

(4-acetoxy-2-(2-fluorophenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2o):

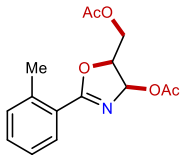
Following **GP A**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 12.7 mg (43%, dr = 1.6:1) of **2o** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.01 – 7.91 (m, 1H), 7.57 – 7.47 (m, 1H), 7.25 – 7.13 (m, 2H), 6.88 (d, *J* = 7.5 Hz, 0.38H), 6.50 (d, *J* = 3.9 Hz, 0.60H), 4.91 – 4.84 (m, 0.38H), 4.75 – 4.69 (m, 0.66H), 4.54 (dd, *J* = 12.2, 3.6 Hz, 0.66H), 4.43 – 4.32 (m, 1.38H), 2.13 (s, 2H), 2.12 (s, 1H), 2.11 (s, 1H), 2.06 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5 (major, minor), 170.3, 169.7, 165.7 (d, J_{CF} = 5.3 Hz), 165.2 (d, J_{CF} = 5.3 Hz), 161.6 (d, J_{CF} = 260.5 Hz), 161.5 (d, J_{CF} = 260.5 Hz), 134.3 (d, J_{CF} = 8.9 Hz, major, minor), 131.5 (major, minor), 124.11 (d, J_{CF} = 3.7 Hz), 124.08 (d, J_{CF} = 3.7 Hz), 116.9 (d, J_{CF} = 21.6 Hz, major, minor), 114.71 (d, J_{CF} = 10.0 Hz), 114.69 (d, J_{CF} = 10.0 Hz), 93.4, 89.8, 82.2, 78.4, 62.7, 60.8, 21.0, 20.8, 20.7, 20.6.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -107.5, -107.6.

HRMS: calc. for $C_{14}H_{14}FNNaO_5^+$ $(M+Na)^+$, 318.0748, found, 318.0752.



2p

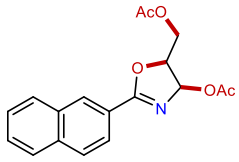
(4-acetoxy-2-(o-tolyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2p):

Following **GP A** with a reaction time of 4.5 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 14.6 mg (50%, dr = 1.6:1) of **2p** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.90 – 7.85 (m, 1H), 7.42 – 7.34 (m, 1H), 7.28 – 7.24 (m, 2H), 6.86 (d, J = 7.4 Hz, 0.38H), 6.48 (d, J = 3.8 Hz, 0.60H), 4.87 – 4.80 (m, 0.37H), 4.70 – 4.65 (m, 0.62H), 4.52 (dd, J = 12.1, 3.6 Hz, 0.64H), 4.44 – 4.27 (m, 1.41H), 2.61 (s, 3H), 2.14 (s, 2H), 2.12 (s, 1H), 2.11 (s, 1H), 2.06 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5 (major, minor), 170.4, 169.8, 169.4, 168.8, 139.64, 139.61, 131.7 (major, minor), 131.47, 131.46, 130.4 (major, minor), 125.71, 125.68, 125.65, 125.6, 93.9, 90.3, 81.7, 78.0, 62.9, 61.0, 21.9, 21.8, 21.1, 20.9, 20.75, 20.65.

HRMS: calc. for $C_{15}H_{17}NNaO_5^+$ $(M+Na)^+$, 314.0999, found, 314.1000.



2q

(4-acetoxy-2-(naphthalen-2-yl)-4,5-dihydrooxazol-5-yl)methyl acetate (2q):

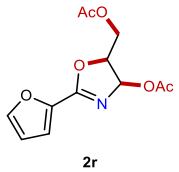
Following **GP A** at a controlled voltage of 1.8 V for 19 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 9.2 mg (28%, dr = 2.7:1) of **2q** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.56 (s, 1H), 8.08 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.93 (d, *J* = 8.1 Hz, 1H), 7.89 (t, *J* = 8.8 Hz, 2H), 7.61 – 7.52 (m, 2H), 6.90 (d, *J* = 7.3 Hz, 0.27H), 6.54 (d, *J* = 3.9 Hz, 0.73H), 4.97 – 4.91 (m, 0.29H), 4.82 – 4.78 (m, 0.76H), 4.60 (dd, *J* = 12.2, 3.6 Hz, 0.79H), 4.47 – 4.32 (m, 1.27H), 2.16 (s, 2H), 2.14 (s, 2H), 2.05 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.6 (major, minor), 170.4, 169.8, 168.8, 168.4, 135.3 (major, minor), 132.5 (major, minor), 130.13, 130.09, 129.12, 129.11, 128.3 (major, minor), 128.20, 128.18, 127.8 (major, minor), 126.8 (major, minor), 124.8 (major, minor), 123.5 (major, minor), 93.8, 90.0, 82.7, 79.0, 62.9, 61.1, 21.1, 20.9, 20.8, 20.7.

HRMS: calc. for $C_{18}H_{17}NNaO_5^+$ $(M+Na)^+$, 350.0999, found, 350.1002.

M.p.: 117.1 - 118.4 °C



2r

(4-acetoxy-2-(furan-2-yl)-4,5-dihydrooxazol-5-yl)methyl acetate (2r):

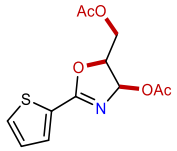
Following **GP A** at a controlled voltage of 2.0 V for 2 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to

afford 9.6 mg (36%, dr = 3.0:1) of **2r** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.61 (s, 1H), 7.15 (d, *J* = 3.8 Hz, 1H), 6.84 (d, *J* = 7.2 Hz, 0.27H), 6.54 – 6.51 (m, 1H), 6.47 (d, *J* = 3.9 Hz, 0.80H), 4.90 – 4.82 (m, 0.28H), 4.75 – 4.68 (m, 0.77H), 4.50 (dd, *J* = 12.3, 3.6 Hz, 0.68H), 4.42 – 4.22 (m, 1.39H), 2.12 (s, 2H), 2.12 (s, 1H), 2.10 (s, 1H), 2.06 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5 (major, minor), 170.2, 169.5, 160.5, 160.2, 146.5 (major, minor), 141.6 (major, minor), 117.1 (major, minor), 112.0 (major, minor), 93.2, 89.4, 82.7, 79.1, 62.7, 60.9, 21.0, 20.82, 20.75, 20.7.

HRMS: calc. for $C_{12}H_{13}NNaO_6^+$ $(M+Na)^+$, 290.0635, found, 290.0636.



2s

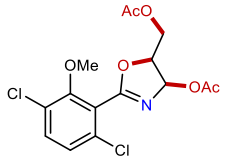
(4-acetoxy-2-(thiophen-2-yl)-4,5-dihydrooxazol-5-yl)methyl acetate (2s):

Following **GP A** at a controlled voltage of 2.2 V for 5 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 11.3 mg (40%, dr = 3.5:1) of **2s** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.80 – 7.67 (d, *J* = 3.7 Hz, 1H), 7.59 – 7.42 (m, 1H), 7.17 – 7.09 (m, 1H), 6.85 – 6.77 (m, 0.22H), 6.47 – 6.39 (m, 0.77H), 4.91 – 4.83 (m, 0.22H), 4.77 – 4.69 (m, 0.76H), 4.58 – 4.47 (m, 0.81H), 4.41– 4.27 (m, 1.23H), 2.17 – 2.10 (m, 3.77H), 2.07 – 2.03 (m, 2.29H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5 (major, minor), 170.4 (major, minor), 164.3 (major, minor), 132.4 (major, minor), 131.9 (major, minor), 128.6 (major, minor), 127.9 (major, minor), 93.6, 89.8, 83.0, 79.3, 62.7, 60.9, 21.0, 20.9, 20.8, 20.7.

HRMS: calc. for $C_{12}H_{13}NNaO_5S^+$ $(M+Na)^+$, 306.0407, found, 306.0413.



2t

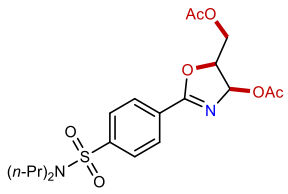
(4-acetoxy-2-(3,6-dichloro-2-methoxyphenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2t):

Following **GP A**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 21.1 mg (56%, dr = 3.2:1) of **2t** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.43 – 7.39 (m, 1H), 7.18 – 7.13 (m, 1H), 6.89 (d, *J* = 7.5 Hz, 0.23H), 6.53 (d, *J* = 3.9 Hz, 0.73H), 4.98 – 4.90 (m, 0.24H), 4.81 – 4.73 (m, 0.76H), 4.51 (dd, *J* = 12.3, 3.6 Hz, 0.78H), 4.43 – 4.29 (m, 1.27H), 3.92 (s, 3H), 2.15 (s, 2.24H), 2.14 (s, 0.81H), 2.11 (s, 2.25H), 2.09 (s, 0.82H).

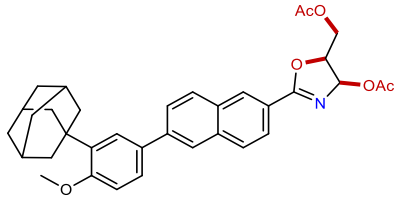
^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5 (major, minor), 170.1, 169.5, 164.7, 164.3, 155.6, 155.4, 132.81, 132.78, 132.0 (major, minor), 126.93, 126.86, 125.90, 125.85, 124.59, 124.56, 92.9, 89.6, 82.6, 79.1, 62.6, 62.4 (major, minor), 60.9, 20.9, 20.74, 20.70, 20.65.

HRMS: calc. for $C_{15}H_{15}Cl_2NNaO_6^+$ $(M+Na)^+$, 398.0169, found, 398.0168.



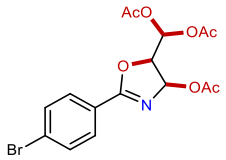
2u

(4-acetoxy-2-(4-(*N*,*N*-dipropylsulfamoyl)phenyl)-4,5-dihydrooxazol-5-yl)methyl acetate (2u):
Following **GP A** with a reaction time of 15 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 28.6 mg (65%, dr = 1.2:1) of **2u** as a colorless oil.
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.16 – 8.13 (m, 2H), 7.87 – 7.85 (m, 2H), 6.85 (d, *J* = 7.5 Hz, 0.44H), 6.48 (d, *J* = 3.7 Hz, 0.53H), 4.95 – 4.87 (m, 0.45H), 4.80 – 4.73 (m, 0.55H), 4.53 (dd, *J* = 12.3, 3.5 Hz, 0.60H), 4.42 – 4.28 (m, 1.43H), 3.11 – 3.06 (m, 4H), 2.13 (s, 1.71H), 2.12 (s, 2.68H), 2.04 (s, 1.65H), 1.61 – 1.48 (m, 4H), 0.85 (t, *J* = 7.4 Hz, 6H).
^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.44, 170.40, 170.1, 169.6, 167.2, 166.8, 143.98, 143.95, 129.70, 129.69, 129.5 (major, minor), 127.04, 127.02, 93.3, 89.7, 82.9, 79.3, 62.7, 60.8, 49.81, 49.78, 21.83, 21.80, 20.9, 20.8, 20.7, 20.6, 11.1 (major, minor).
HRMS: calc. for $C_{20}H_{28}N_2NaO_7S^+$ $(M+Na)^+$, 463.1509, found, 463.1511.



2v

(4-acetoxy-2-(6-(3-(adamantan-1-yl)-4-methoxyphenyl)naphthalen-2-yl)-4,5-dihydrooxazol-5-yl)methyl acetate (2v):
Following **GP A** at a controlled voltage of 1.4 V for 5 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 20.4 mg (36%, dr = 2.9:1) of **2v** as a colorless oil.
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.56 (s, 1H), 8.09 (d, *J* = 8.6 Hz, 1H), 8.01 (s, 1H), 7.95 (dd, *J* = 18.9, 8.5 Hz, 2H), 7.80 (d, *J* = 8.4 Hz, 1H), 7.60 (s, 1H), 7.54 (d, *J* = 8.4 Hz, 1H), 7.00 (d, *J* = 8.4 Hz, 1H), 6.91 (d, *J* = 7.4 Hz, 0.26H), 6.55 (d, *J* = 3.9 Hz, 0.75H), 4.99 – 4.92 (m, 0.27H), 4.84 – 4.77 (m, 0.80H), 4.64 – 4.58 (m, 0.81H), 4.47 – 4.33 (m, 1.24H), 3.91 (s, 3H), 2.19 (d, *J* = 2.7 Hz, 6H), 2.17 (s, 2H), 2.16 (s, 2H), 2.10 (s, 3H), 2.07 (s, 2H), 1.80 (s, 6H).
^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.6 (major, minor), 170.4, 169.8, 168.9, 168.5, 158.9 (major, minor), 141.4 (major, minor), 139.0 (major, minor), 135.7 (major, minor), 132.5 (major, minor), 131.2 (major, minor), 129.9, 129.8, 129.5 (major, minor), 128.4 (major, minor), 126.6 (major, minor), 126.0 (major, minor), 125.7 (major, minor), 125.2 (major, minor), 124.8 (major, minor), 123.0 (major, minor), 112.1 (major, minor), 93.8, 90.1, 82.7, 79.0, 62.9, 61.2, 55.1 (major, minor), 40.6 (major, minor), 37.2 (major, minor), 37.1 (major, minor), 29.1 (major, minor), 21.1, 20.9, 20.8, 20.7.
HRMS: calc. for $C_{35}H_{37}NNaO_6^+$ $(M+Na)^+$, 590.2513, found, 590.2516.



2w

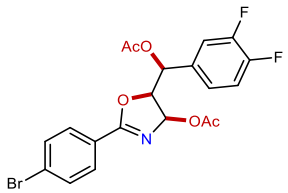
(4-acetoxy-2-(4-bromophenyl)-4,5-dihydrooxazol-5-yl)methylene diacetate (2w):

Following **GP A** at a controlled voltage of 1.5 V for 12 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 21.1 mg (51%, dr = 1.9:1) of **2w** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.86 (t, *J* = 8.6 Hz, 2H), 7.60 – 7.57 (m, 2H), 7.22 – 7.20 (m, 0.35H), 7.08 (d, *J* = 3.6 Hz, 0.65H), 6.87 (d, *J* = 7.4 Hz, 0.38H), 6.69 (d, *J* = 3.5 Hz, 0.64H), 4.81 (t, *J* = 6.8 Hz, 0.36H), 4.74 – 4.71 (m, 0.64H), 2.13 (s, 5H), 2.11 (s, 1H), 2.08 (s, 1H), 2.03 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.54, 169.45, 168.43, 168.42, 167.93, 167.90, 167.6, 167.2, 131.91, 131.88, 130.5, 130.4, 127.8, 127.7, 124.9, 124.8, 91.4, 88.9, 86.2, 84.9, 82.3, 79.5, 21.0, 20.8, 20.7, 20.6 (major, minor), 20.5.

HRMS: calc. for $C_{16}H_{16}BrNNaO_7^+$ $(M+Na)^+$, 436.0002, found, 436.0005.



2x

5-(acetoxy(3,4-difluorophenyl)methyl)-2-(4-bromophenyl)-4,5-dihydrooxazol-4-yl acetate (2x):

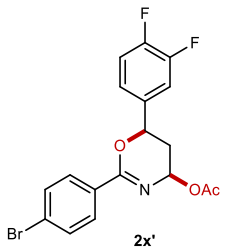
Following **GP A** at a controlled voltage of 1.4 V for 12 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 15.0 mg (32%, dr > 20:1) of **2x** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.81 (d, *J* = 8.5 Hz, 2H), 7.58 (d, *J* = 8.1 Hz, 2H), 7.25 – 7.20 (m, 1H), 7.16 – 7.11 (m, 2H), 6.54 (d, *J* = 4.0 Hz, 1H), 6.02 (d, *J* = 4.9 Hz, 1H), 4.79 (t, *J* = 4.5 Hz, 1H), 2.11 (s, 3H), 2.10 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.7, 169.4, 167.4, 150.5 (dd, J_{CF} = 249.9, 28.7 Hz), 150.4 (dd, J_{CF} = 249.9, 27.4 Hz), 132.0, 131.7 (dd, J_{CF} = 5.1, 4.0 Hz), 130.3, 127.8, 124.8, 123.5 (dd, J_{CF} = 6.5, 3.8 Hz), 117.7 (d, J_{CF} = 17.5 Hz), 116.5 (d, J_{CF} = 18.3 Hz), 92.2, 85.5, 72.5, 21.0, 20.8.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -136.1 (d, *J* = 20.8 Hz), -136.6 (d, *J* = 21.5 Hz).

HRMS: calc. for $C_{20}H_{16}BrF_2NNaO_5^+$ $(M+Na)^+$, 490.0072, found, 490.0077.



2x'

2-(4-bromophenyl)-6-(3,4-difluorophenyl)-5,6-dihydro-4H-1,3-oxazin-4-yl acetate (2x'):

Following **GP A** at a controlled voltage of 1.5 V for 10 h: the desired pure product was purified by

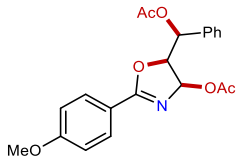
preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 11.9 mg (29%, dr = 1.2:1) of **2x'** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 8.6 Hz, 1H), 7.86 (d, *J* = 8.6 Hz, 1H), 7.56 - 7.50 (m, 2H), 7.25 - 7.18 (m, 2H), 7.15 - 7.09 (m, 1H), 6.40 - 6.34 (m, 0.54H), 6.14 (t, *J* = 3.8 Hz, 0.46H), 5.42 - 5.33 (m, 1H), 2.64 - 2.59 (m, 0.44H), 2.32 - 2.27 (m, 0.42H), 2.26 - 2.19 (m, 0.61H), 2.17 (s, 1.51H), 2.15 (s, 1.48H), 1.95 - 1.84 (m, 0.61H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.3, 169.7, 157.7, 155.8, 150.6 (dd, J_{CF} = 249.4, 23.1 Hz) (major, mino), 150.5 (dd, J_{CF} = 249.5, 23.4 Hz) (major, mino), 136.1 (dd, J_{CF} = 5.4, 3.7 Hz), 135.7 (dd, J_{CF} = 5.3, 3.5 Hz), 131.5, 131.4, 131.2, 131.0, 129.4, 129.2, 126.5, 126.2, 122.0 (dd, J_{CF} = 6.4, 3.6 Hz), 121.8 (dd, J_{CF}= 6.6, 3.7 Hz), 117.81 (d, J_{CF} = 17.4 Hz), 117.80 (d, J_{CF} = 17.6 Hz), 115.1 (d, J_{CF} = 18.4 Hz), 115.0 (d, J_{CF} = 18.3 Hz), 79.5, 75.1, 74.8, 72.2, 34.0, 33.6, 21.3, 21.3.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -136.1 (dd, *J* = 21.2, 5.6 Hz), -137.4 (dd, *J* = 179.6, 20.9 Hz).

HRMS: calc. for $C_{18}H_{15}BrF_2NO_3^+$ $(M+H)^+$, 410.0198, found, 410.0196.



2y

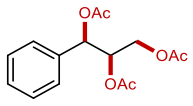
5-(acetoxy(phenyl)methyl)-2-(4-methoxyphenyl)-4,5-dihydrooxazol-4-yl acetate (2y):

Following **GP A** at a controlled voltage of 1.0 V for 5 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 13.8 mg (36%, dr > 20:1) of **2y** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.93 – 7.88 (m, 2H), 7.40 (d, *J* = 7.5 Hz, 2H), 7.36 – 7.29 (m, 3H), 6.92 – 6.90 (m, 2H), 6.62 (s, 1H), 6.12 (s, 1H), 4.80 (s, 1H), 3.85 (s, 3H), 2.08 (s, 3H), 2.04 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.69, 169.67, 168.1, 163.0, 134.8, 130.7, 128.8, 128.6, 127.1, 118.5, 113.8, 92.2, 85.4, 73.6, 55.4, 21.0, 20.9.

HRMS: calc. for $C_{21}H_{22}NO_6^+$ $(M+H)^+$, 384.1442, found, 384.1447.



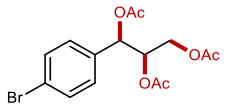
4a

1-phenylpropane-1,2,3-triyl triacetate (4a)[15]:

Following **GP B**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 5:1) to afford 48.6 mg (55%, dr = 1.9:1) of **4a** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.39 – 7.35 (m, 3.59H), 7.35 – 7.29 (m, 1.40H), 6.01 (d, *J* = 5.6 Hz, 0.66H), 5.97 (d, *J* = 7.2 Hz, 0.34H), 5.46 – 5.37 (m, 1H), 4.29 – 4.20 (m, 1.66H), 3.81 (dd, *J* = 12.1, 5.7 Hz, 0.35H), 2.13 (s, 2H), 2.08 (s, 1H), 2.05 (s, 1H), 2.05 (s, 1H), 2.02 (s, 2H), 1.99 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 170.3, 169.93, 169.86, 169.6, 169.5, 135.9, 135.8, 128.8, 128.7, 128.5, 128.4, 127.1, 126.8, 73.8, 73.1, 72.4, 72.2, 62.1, 61.6, 20.9 (major, minor), 20.7 (major, minor), 20.6 (major, minor).



4b

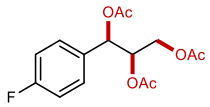
1-(4-bromophenyl)propane-1,2,3-triyl triacetate (4b):

Following **GP B**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 5:1) to afford 52.6 mg (47%, dr > 20:1) of **4b** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.48 – 7.46 (m, 2H), 7.24 – 7.21 (m, 2H), 5.91 (d, *J* = 6.1 Hz, 1H), 5.34 (q, *J* = 5.7 Hz, 1H), 4.20 (d, *J* = 4.9 Hz, 2H), 2.10 (s, 3H), 2.01 (s, 3H), 1.97 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 169.8, 169.3, 135.0, 131.6, 128.6, 122.6, 72.5, 72.1, 61.5, 20.8, 20.7, 20.6.

HRMS: calc. for $C_{15}H_{17}BrNaO_6^+$ $(M+Na)^+$, 395.0101, found, 395.0102.



4c

1-(4-fluorophenyl)propane-1,2,3-triyl triacetate (4c):

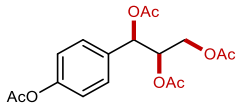
Following **GP B**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 56.2 mg (60%, dr = 9:1) of **4c** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.33 (dd, *J* = 8.3, 5.3 Hz, 2H), 7.07 – 6.94 (m, 2H), 5.93 (d, *J* = 5.9 Hz, 1H), 5.39 – 5.31 (m, 1H), 4.20 (d, *J* = 4.9 Hz, 1.80H), 3.78 (dd, *J* = 12.2, 5.7 Hz, 0.20H), 2.09 (s, 2.30H), 2.06 (s, 0.71H), 2.03 (s, 0.69H), 2.03 (s, 0.71H), 2.01 (s, 2.27H), 1.96 (s, 2.31H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 169.7, 169.4, 162.6 (d, J_{CF} = 247.4 Hz), 131.8 (d, J_{CF} = 3.3 Hz), 128.8 (d, J_{CF} = 8.3 Hz), 115.4 (d, J_{CF} = 21.6 Hz), 72.4, 72.2, 61.6, 20.8, 20.61, 20.57.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -112.4, -113.0.

HRMS: calc. for $C_{15}H_{17}FNaO_6^+$ $(M+Na)^+$, 335.0901, found, 335.0902.



4d

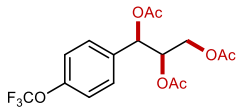
1-(4-acetoxyphenyl)propane-1,2,3-triyl triacetate (4d):

Following **GP B**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 49.7 mg (47%, dr = 4.4:1) of **4d** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.39 – 7.36 (m, 2H), 7.25 – 7.06 (m, 2H), 6.00 (d, *J* = 5.8 Hz, 0.18H), 5.97 (dd, *J* = 7.3, 2.1 Hz, 0.79H), 5.42 – 5.37 (m, 1H), 4.30 – 4.20 (m, 1.21H), 3.83 – 3.75 (m, 0.83H), 2.32 (s, 0.37H), 2.28 (s, 2.68H), 2.11 (s, 0.51H), 2.07 (s, 2.53H), 2.05 (s, 2.42H), 2.04 (s, 2.59H), 2.02 (s, 0.51H), 1.99 (s, 0.39H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.4, 170.0, 169.6, 169.1, 151.0, 133.4, 128.4, 121.9, 73.2, 72.1, 62.0, 21.1, 20.9, 20.7, 20.6.

HRMS: calc. for $C_{17}H_{20}NaO_8^+$ $(M+Na)^+$, 375.1050, found, 375.1055.



4e

1-(4-(trifluoromethoxy)phenyl)propane-1,2,3-triyl triacetate (4e):

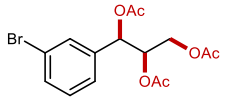
Following **GP B**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 60.1 mg (53%, dr > 20:1) of **4e** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.39 (d, *J* = 8.6 Hz, 2H), 7.19 (d, *J* = 8.3 Hz, 2H), 5.97 (d, *J* = 6.0 Hz, 1H), 5.40 – 5.33 (m, 1H), 4.22 – 4.19 (m, 2H), 2.11 (s, 2.79H), 2.08 (s, 0.29H), 2.04 (s, 0.53H), 2.01 (s, 2.86H), 1.97 (s, 2.70H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 169.8, 169.4, 149.2, 134.6, 128.5, 120.9, 120.3 (q, J_{CF} = 257.6 Hz), 72.3, 72.1, 61.5, 20.8, 20.61, 20.55.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -57.9.

HRMS: calc. for $C_{16}H_{17}F_3NaO_7^+$ $(M+Na)^+$, 401.0819, found, 401.0819.



4f

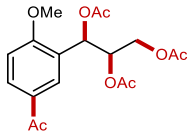
1-(3-bromophenyl)propane-1,2,3-triyl triacetate (4f):

Following **GP B**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 39.2 mg (35%, dr = 1:1) of **4f** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.50 (s, 1H), 7.45 (t, *J* = 7.0 Hz, 1H), 7.28 (d, *J* = 7.9 Hz, 1H), 7.22 (t, *J* = 7.8 Hz, 1H), 5.94 (d, *J* = 5.8 Hz, 0.49H), 5.90 (d, *J* = 6.7 Hz, 0.49H), 5.41 – 5.31 (m, 1H), 4.31 – 4.17 (m, 1.59H), 3.82 (dd, *J* = 12.1, 5.7 Hz, 0.48H), 2.13 (s, 1.51H), 2.10 (s, 1.54H), 2.05 (s, 3H), 2.02 (s, 1.59H), 2.00 (s, 1.44H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 170.3, 169.84, 169.81, 169.6, 169.4, 138.25, 138.22, 132.0, 131.7, 130.3, 130.14, 130.09, 129.9, 125.8, 125.4, 122.7, 122.6, 73.0, 72.31, 72.26, 71.9, 61.9, 61.4, 20.88, 20.87, 20.70, 20.67, 20.6 (major, mino).

HRMS: calc. for $C_{15}H_{17}BrNaO_6^+$ $(M+Na)^+$, 395.0101, found, 395.0101.



4g

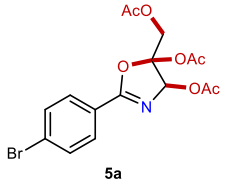
1-(5-acetoxy-2-methoxyphenyl)propane-1,2,3-triyl triacetate (4g):

Following **GP B** at a controlled voltage of 2.0 V: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 56.2 mg (49%, dr = 1.1:1) of **4g** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.95 – 7.85 (m, 2H), 6.92 – 6.82 (m, 1H), 6.33 (dd, *J* = 6.2, 4.3 Hz, 1H), 5.57 – 5.45 (m, 1H), 4.26 – 4.17 (m, 1H), 4.09 – 4.03 (m, 1H), 3.91 (s, 3H), 2.53 (s, 1.43H), 2.52 (s, 1.55H), 2.13 (s, 1.46H), 2.12 (s, 1.51H), 2.02 (s, 1.45H), 2.01 (s, 1.52H), 1.94 (s, 1.45H), 1.91 (s, 1.55H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 196.3 (major, minor), 170.44, 170.37, 169.8 (major, minor), 169.6, 169.5, 160.1, 159.9, 130.9, 130.8, 130.1, 130.0, 128.0, 127.7, 125.0, 124.3, 110.01, 109.98, 70.9, 70.8, 68.5, 68.2, 62.3, 61.9, 55.95, 55.89, 26.2 (major, minor), 20.9, 20.8, 20.7, 20.6, 20.52, 20.48.

HRMS: calc. for $C_{18}H_{22}NaO_8^+$ $(M+Na)^+$, 389.1207, found, 389.1209.



5a

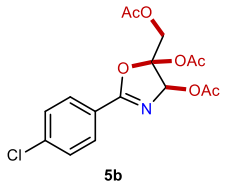
5-(acetoxymethyl)-2-(4-bromophenyl)-4,5-dihydrooxazole-4,5-diyl diacetate (5a):

Following **GP C**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 17.0 mg (41%, dr = 2.3:1) of **5a** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.91– 7.82 (m, 2H), 7.61 – 7.55 (m, 2H), 6.83 (s, 0.70H), 6.67 (s, 0.30H), 4.89 – 4.79 (m, 1H), 4.57 – 4.48 (m, 1H), 2.14 (s, 1H), 2.13 (s, 2H), 2.11 (s, 2H), 2.11 (s, 1H), 2.09 (s, 2H), 2.04 (s, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.9, 169.8, 169.3, 168.8, 168.3, 167.4, 166.7, 165.4, 133.0, 132.8, 129.0, 128.9, 128.54, 128.53, 125.7, 125.6, 107.6, 105.3, 94.0, 90.6, 63.2, 60.6, 21.5, 21.2, 20.8, 20.59, 20.56 (major, minor).

HRMS: calc. for $C_{16}H_{16}BrNNaO_7^+$ $(M+Na)^+$, 436.0002, found, 436.0004.



5b

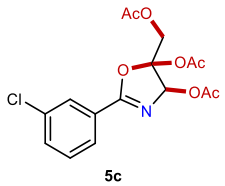
5-(acetoxymethyl)-2-(4-chlorophenyl)-4,5-dihydrooxazole-4,5-diyl diacetate (5b):

Following **GP C**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 17.4 mg (47%, dr = 1.5:1) of **5b** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.94 (t, *J* = 8.0 Hz, 2H), 7.41 (d, *J* = 8.1 Hz, 2H), 6.83 (s, 0.60H), 6.68 (s, 0.41H), 4.89 - 4.79 (m, 1H), 4.57 - 4.76 (m, 1H), 2.14 (s, 1H), 2.13 (s, 2H), 2.11 (s, 2H), 2.10 (s, 1H), 2.09 (s, 2H), 2.04 (s, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.8, 169.7, 169.2, 168.7, 168.2, 167.3, 165.7, 164.5, 139.4, 139.2, 130.3, 130.2, 128.9 (major, minor), 124.13, 124.05, 107.7, 105.4, 93.9, 90.5, 63.1, 60.6, 21.4, 21.2, 20.7, 20.6, 20.5 (major, minor).

HRMS: calc. for $C_{16}H_{16}ClNNaO_7^+$ $(M+Na)^+$, 392.0508, found, 392.0508.



5c

5-(acetoxymethyl)-2-(3-chlorophenyl)-4,5-dihydrooxazole-4,5-diyl diacetate (5c):

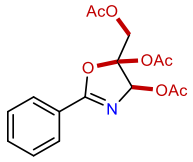
Following **GP C**: the desired pure product was purified by preparative thin-layer chromatography

(PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 14.8 mg (40%, dr = 1.9:1) of **5c** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.4 Hz, 1H), 7.89 (t, *J* = 7.7 Hz, 1H), 7.52 (d, *J* = 8.1 Hz, 1H), 7.38 (t, *J* = 8.1 Hz, 1H), 6.85 (s, 0.65H), 6.69 (s, 0.35H), 4.91 - 4.74 (m, 1H), 4.58 - 4.45 (m, 1H), 2.14 (s, 3H), 2.12 (s, 3H), 2.10 (s, 2H), 2.06 (s, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.9, 169.7, 169.2, 168.7, 168.2, 167.3, 165.4, 164.2, 134.72, 134.70, 133.0, 132.8, 129.9 (major, minor), 129.0, 128.8, 127.4, 127.3, 127.1, 126.9, 107.8, 105.4, 93.8, 90.4, 63.2, 60.6, 21.4, 21.2, 20.7, 20.6, 20.54, 20.51.

HRMS: calc. for $C_{16}H_{16}ClNNaO_7^+$ $(M+Na)^+$, 392.0508, found, 392.0512.



5d

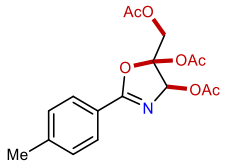
5-(acetoxymethyl)-2-phenyl-4,5-dihydrooxazole-4,5-diyl diacetate (5d):

Following **GP C**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 12.4 mg (37%, dr = 1.6:1) of **5d** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.01 (t, *J* = 7.9 Hz, 2H), 7.59 – 7.51 (m, 1H), 7.44 (t, *J* = 7.9 Hz, 2H), 6.86 (s, 0.64H), 6.70 (s, 0.40H), 4.92 – 4.82 (m, 1H), 4.59 – 4.50 (m, 1H), 2.14 (s, 1H), 2.13 (s, 2H), 2.11 (s, 3H), 2.09 (s, 2H), 2.05 (s, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.9, 169.7, 169.2, 168.7, 168.3, 167.3, 165.8, 164.6, 131.9 (major, minor), 130.4, 130.3, 128.0, 127.8, 124.6, 124.5, 107.8, 105.4, 93.9, 90.5, 63.1, 60.6, 21.4, 21.2, 20.7, 20.6, 20.5 (major, minor).

HRMS: calc. for $C_{16}H_{17}NNaO_7^+$ $(M+Na)^+$, 358.0897, found, 358.0900.



5e

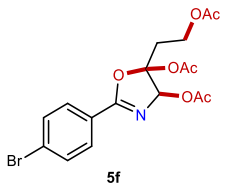
5-(acetoxymethyl)-2-(p-tolyl)-4,5-dihydrooxazole-4,5-diyl diacetate (5e):

Following **GP C**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 11.5 mg (33%, dr = 1.2:1) of **5e** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.89 (t, *J* = 7.7 Hz, 2H), 7.23 (d, *J* = 7.8 Hz, 2H), 6.83 (s, 0.54H), 6.68 (s, 0.45H), 4.91 – 4.81 (m, 1H), 4.58 – 4.51 (m, 1H), 2.39 (s, 3H), 2.13 (s, 1H), 2.12 (s, 2H), 2.10 (s, 3H), 2.08 (s, 2H), 2.03 (s, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.9, 169.8, 169.3, 168.8, 168.3, 167.3, 166.8, 165.5, 143.7, 143.5, 129.24, 129.22, 129.0, 128.8, 122.8, 122.7, 107.5, 105.2, 94.0, 90.7, 63.1, 60.6, 21.7, 21.6, 21.5, 21.2, 20.8, 20.6, 20.55, 20.52.

HRMS: calc. for $C_{17}H_{19}NNaO_7^+$ $(M+Na)^+$, 372.1054, found, 372.1055.



5f

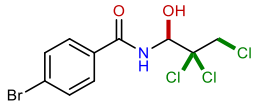
5-(2-acetoxyethyl)-2-(4-bromophenyl)-4,5-dihydrooxazole-4,5-diyl diacetate (5f):

Following **GP C**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 11.6 mg (27%, dr > 20:1) of **5f** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 8.5 Hz, 2H), 7.58 (d, *J* = 8.6 Hz, 2H), 6.73 (s, 1H), 4.35 – 4.28 (m, 2H), 2.61 – 2.54 (m, 2H), 2.17 (s, 3H), 2.10 (s, 3H), 2.06 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.7, 168.9, 168.5, 165.7, 131.9, 130.4, 127.9, 124.7, 110.3, 94.0, 59.3, 30.7, 21.5, 20.9, 20.9.

HRMS: calc. for $C_{17}H_{18}BrNNaO_7^+$ $(M+Na)^+$, 450.0159, found, 450.0163.



6a

4-bromo-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6a):

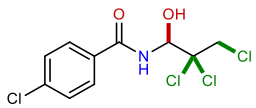
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 28.2 mg (78%) of **6a** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.77 (d, *J* = 8.6 Hz, 2H), 7.66 (d, *J* = 8.6 Hz, 2H), 6.17 (s, 1H), 4.33 (d, *J* = 11.9 Hz, 1H), 4.20 (d, *J* = 11.9 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 169.2, 134.3, 132.8, 130.6, 127.6, 93.5, 77.6, 52.7.

HRMS: calc. for $C_{10}H_9BrCl_3NNaO_2^+$ $(M+Na)^+$, 381.8774, found, 381.8768.

M.p.: 135.1 - 136.0 °C



6b

4-chloro-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6b):

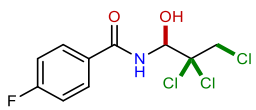
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 23.8 mg (75%) of **6b** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.84 (d, *J* = 8.5 Hz, 2H), 7.50 (d, *J* = 8.6 Hz, 2H), 6.17 (s, 1H), 4.33 (d, *J* = 12.0 Hz, 1H), 4.20 (d, *J* = 12.0 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 169.0, 139.2, 133.8, 130.4, 129.8, 93.5, 77.6, 52.7.

HRMS: calc. for $C_{10}H_9Cl_4NNaO_2^+$ $(M+Na)^+$, 337.9280, found, 337.9277.

M.p.: 113.8 - 114.3 °C

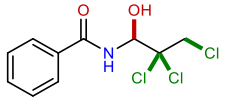


6c

4-fluoro-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6c):

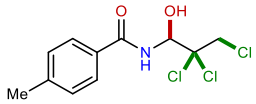
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography

(PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 27.3 mg (91%) of **6c** as a white solid.
^{1}H NMR (500 MHz, Methanol-d_4) δ 7.96 – 7.88 (m, 2H), 7.25 – 7.15 (m, 2H), 6.17 (s, 1H), 4.33 (d, J = 12.0 Hz, 1H), 4.20 (d, J = 11.9 Hz, 1H).
^{13}C NMR (126 MHz, Methanol-d_4) δ 169.0, 166.5 (d, J_{CF} = 250.9 Hz), 131.5 (d, J_{CF} = 3.1 Hz), 131.4 (d, J_{CF} = 9.2 Hz), 116.5 (d, J_{CF} = 22.3 Hz), 93.6, 77.6, 52.7.
^{19}F NMR (471 MHz, Methanol-d_4) δ -109.8.
HRMS: calc. for $C_{10}H_9Cl_3FNNaO_2^+$ $(M+Na)^+$, 321.9575, found, 321.9575.
M.p.: 123.1 - 124.1 °C



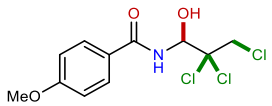
6d

***N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6d):**
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 24.6 mg (87%) of **6d** as a white solid.
^{1}H NMR (500 MHz, Methanol-d_4) δ 7.85 (d, J = 7.6 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.49 (t, J = 7.6 Hz, 2H), 6.18 (s, 1H), 4.33 (d, J = 12.0 Hz, 1H), 4.20 (d, J = 12.0 Hz, 1H).
^{13}C NMR (126 MHz, Methanol-d_4) δ 170.1, 135.2, 133.2, 129.6, 128.7, 93.6, 77.6, 52.7.
HRMS: calc. for $C_{10}H_{10}Cl_3NNaO_2^+$ $(M+Na)^+$, 303.9669, found, 303.9670.
M.p.: 124.3 - 125.1 °C



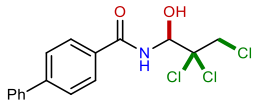
6e

4-methyl-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6e):
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 21.7 mg (73%) of **6e** as a white solid.
^{1}H NMR (500 MHz, Methanol-d_4) δ 7.75 (d, J = 8.3 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H), 6.16 (s, 1H), 4.32 (d, J = 12.0 Hz, 1H), 4.20 (d, J = 12.0 Hz, 1H), 2.41 (s, 3H).
^{13}C NMR (126 MHz, Methanol-d_4) δ 170.0, 144.0, 132.2, 130.2, 128.7, 93.7, 77.5, 52.7, 21.5.
HRMS: calc. for $C_{11}H_{12}Cl_3NNaO_2^+$ $(M+Na)^+$, 317.9826, found, 317.9825.
M.p.: 164.4 - 165.3 °C



6f

4-methoxy-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6f):
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 20.3 mg (65%) of **6f** as a white solid.
^{1}H NMR (500 MHz, Methanol-d_4) δ 7.84 (d, J = 8.9 Hz, 2H), 7.04 – 6.99 (m, 2H), 6.16 (s, 1H), 4.33 (d, J = 12.0 Hz, 1H), 4.20 (d, J = 11.9 Hz, 1H), 3.86 (s, 3H).
^{13}C NMR (126 MHz, Methanol-d_4) δ 169.5, 164.4, 130.6, 127.1, 114.8, 93.7, 77.5, 56.0, 52.7.
HRMS: calc. for $C_{11}H_{12}Cl_3NNaO_3^+$ $(M+Na)^+$, 333.9775, found, 333.9781.
M.p.: 137.4 - 138.2 °C



6g

***N*-(2,2,3-trichloro-1-hydroxypropyl)-[1,1'-biphenyl]-4-carboxamide (6g):**

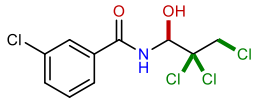
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 21.5 mg (60%) of **6g** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.95 (d, J = 8.4 Hz, 2H), 7.76 (d, J = 8.4 Hz, 2H), 7.69 – 7.67 (m, 2H), 7.49 – 7.44 (m, 2H), 7.41 – 7.36 (m, 1H), 6.20 (s, 1H), 4.35 (d, J = 12.0 Hz, 1H), 4.22 (d, J = 12.0 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 169.8, 146.2, 141.2, 133.8, 130.0, 129.3, 129.2, 128.2, 128.1, 93.7, 77.6, 52.7.

HRMS: calc. for $C_{16}H_{14}Cl_3NNaO_2^+$ $(M+Na)^+$, 379.9982, found, 379.9983.

M.p.: 213.7 - 214.3 °C



6h

3-chloro-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6h):

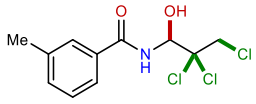
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 23.5 mg (74%) of **6h** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.85 (s, 1H), 7.78 – 7.76 (m, 1H), 7.59 – 7.57 (m, 1H), 7.48 (t, J = 7.9 Hz, 1H), 6.17 (s, 1H), 4.33 (d, J = 12.0 Hz, 1H), 4.20 (d, J = 12.0 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 168.7, 137.2, 135.6, 133.0, 131.2, 128.9, 127.1, 93.5, 77.7, 52.7.

HRMS: calc. for $C_{10}H_9Cl_4NNaO_2^+$ $(M+Na)^+$, 337.9280, found, 337.9286.

M.p.: 131.3 - 132.1 °C



6i

3-methyl-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6i):

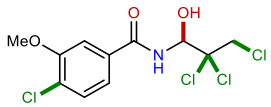
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 22.8 mg (77%) of **6i** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.67 (s, 1H), 7.64 (d, J = 7.4 Hz, 1H), 7.42 – 7.34 (m, 2H), 6.17 (s, 1H), 4.33 (d, J = 12.0 Hz, 1H), 4.20 (d, J = 12.0 Hz, 1H), 2.41 (s, 3H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 170.3, 139.7, 135.2, 133.8, 129.5, 129.1, 125.8, 93.6, 77.5, 52.7, 21.4.

HRMS: calc. for $C_{11}H_{12}Cl_3NNaO_2^+$ $(M+Na)^+$, 317.9826, found, 317.9832.

M.p.: 118.8 - 119.7 °C



6j

4-chloro-3-methoxy-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6j):

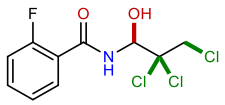
Following **GP D** at a controlled voltage of 2.0 V: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 17.4 mg (50%) of **6j** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.37 (d, J = 8.7 Hz, 1H), 7.05 (d, J = 3.0 Hz, 1H), 7.04 – 6.99 (m, 1H), 6.12 (s, 1H), 4.32 (d, J = 12.0 Hz, 1H), 4.19 (d, J = 12.0 Hz, 1H), 3.83 (s, 3H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 169.4, 159.8, 137.5, 132.1, 123.1, 118.1, 115.5, 93.2, 77.3, 56.2, 52.7.

HRMS: calc. for $C_{11}H_{11}Cl_4NNaO_3^+$ $(M+Na)^+$, 367.9385, found, 367.9392.

M.p.: 118.7 - 119.6 ºC



6k

2-fluoro-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6k):

Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 21.0 mg (70%) of **6k** as a white solid.

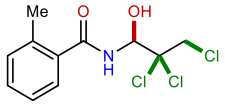
^{1}H NMR (500 MHz, Methanol-d_4) δ 7.87– 7.81 (m, 1H), 7.61 – 7.51 (m, 1H), 7.34 – 7.30 (m, 1H), 7.28 – 7.22 (m, 1H), 6.16 (s, 1H), 4.34 (d, J = 11.9 Hz, 1H), 4.20 (d, J = 11.9 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 166.1 (d, J_{CF} = 2.0 Hz), 161.8 (d, J_{CF} = 249.1 Hz), 134.9 (d, J_{CF} = 9.2 Hz), 132.0 (d, J_{CF} = 2.3 Hz), 125.9 (d, J_{CF} = 3.6 Hz), 123.0 (d, J_{CF} = 12.6 Hz), 117.4 (d, J_{CF} = 23.5 Hz), 93.3, 77.2, 52.6.

^{19}F NMR (471 MHz, Methanol-d_4) δ -115.5.

HRMS: calc. for $C_{10}H_9Cl_3FNNaO_2^+$ $(M+Na)^+$, 321.9575, found, 321.9573.

M.p.: 113.0 - 113.9 ºC



6l

2-methyl-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6l):

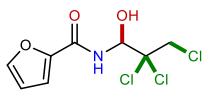
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 18.1 mg (61%) of **6l** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.42 – 7.38 (m, 1H), 7.38 – 7.33 (m, 1H), 7.28 – 7.22 (m, 2H), 6.15 (s, 1H), 4.33 (d, J = 12.0 Hz, 1H), 4.19 (d, J = 12.0 Hz, 1H), 2.45 (s, 3H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 172.8, 137.4, 136.9, 131.8, 131.2, 128.1, 126.8, 93.4, 77.1, 52.7, 19.8.

HRMS: calc. for $C_{11}H_{12}Cl_3NNaO_2^+$ $(M+Na)^+$, 317.9826, found, 317.9830.

M.p.: 134.2 - 135.0 ºC



6m

***N*-(2,2,3-trichloro-1-hydroxypropyl)furan-2-carboxamide (6m):**

Following **GP D** at a controlled voltage of 1.8 V: the desired pure product was purified by

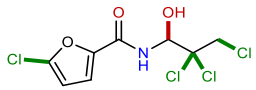
preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 10.4 mg (38%) of **6m** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.75 – 7.70 (m, 1H), 7.25 (d, J = 3.5 Hz, 1H), 6.63 (dd, J = 3.6, 1.8 Hz, 1H), 6.10 (s, 1H), 4.33 (d, J = 11.9 Hz, 1H), 4.20 (d, J = 12.0 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 159.9, 148.1, 147.0, 116.8, 113.3, 93.4, 76.8, 52.6.

HRMS: calc. for $C_8H_8Cl_3NNaO_3^+$ $(M+Na)^+$, 293.9462, found, 293.9470.

M.p.: 127.8 - 128.6 °C



6m'

5-chloro-*N*-(2,2,3-trichloro-1-hydroxypropyl)furan-2-carboxamide (6m'):

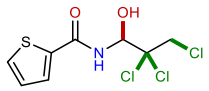
Following **GP D** at a controlled voltage of 1.8 V: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 12.3 mg (40%) of **6m'** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.29 (d, J = 3.6 Hz, 1H), 6.52 (d, J = 3.6 Hz, 1H), 6.09 (s, 1H), 4.32 (d, J = 12.0 Hz, 1H), 4.19 (d, J = 12.0 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 158.8, 147.6, 141.0, 118.9, 110.5, 93.3, 76.9, 52.6.

HRMS: calc. for $C_8H_7Cl_4NNaO_3^+$ $(M+Na)^+$, 327.9072, found, 327.9070.

M.p.: 144.6 - 145.4 °C



6n

***N*-(2,2,3-trichloro-1-hydroxypropyl)thiophene-2-carboxamide (6n):**

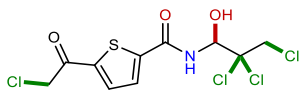
Following **GP D** at a controlled voltage of 2.0 V: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 10.1 mg (35%) of **6n** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.85 (d, J = 3.8 Hz, 1H), 7.72 (d, J = 5.0 Hz, 1H), 7.16 (t, J = 4.4 Hz, 1H), 6.13 (s, 1H), 4.32 (d, J = 12.0 Hz, 1H), 4.19 (d, J = 12.0 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 164.0, 139.3, 132.8, 130.7, 128.9, 93.5, 77.5, 52.7.

HRMS: calc. for $C_8H_8Cl_3NNaO_2S^+$ $(M+Na)^+$, 309.9234, found, 309.9237.

M.p.: 143.1 - 143.7 °C



6o

5-(2-chloroacetyl)-*N*-(2,2,3-trichloro-1-hydroxypropyl)thiophene-2-carboxamide (6o):

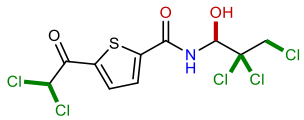
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 18.6 mg (51%) of **6o** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.91 (d, J = 4.1 Hz, 1H), 7.88 (d, J = 4.1 Hz, 1H), 6.14 (s, 1H), 4.87 (s, 2H), 4.32 (d, J = 11.9 Hz, 1H), 4.19 (d, J = 11.9 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 186.8, 163.0, 147.0, 145.5, 134.6, 131.0, 93.3, 77.7, 52.7, 46.7.

HRMS: calc. for $C_{10}H_9Cl_4NNaO_3S^+$ $(M+Na)^+$, 385.8949, found, 385.8958.

M.p.: 194.2 - 194.9 °C



6o'

5-(2,2-dichloroacetyl)-*N*-(2,2,3-trichloro-1-hydroxypropyl)thiophene-2-carboxamide (6o'):

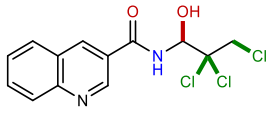
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 12.0 mg (30%) of **6o'** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 8.04 (d, J = 4.2 Hz, 1H), 7.91 (d, J = 4.1 Hz, 1H), 7.26 (s, 1H), 6.15 (s, 1H), 4.32 (d, J = 12.0 Hz, 1H), 4.19 (d, J = 11.9 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 181.6, 162.7, 148.4, 142.3, 136.1, 130.9, 93.2, 77.7, 69.0, 52.6.

HRMS: calc. for $C_{10}H_8Cl_5NNaO_3S^+$ $(M+Na)^+$, 419.8560, found, 419.8557.

M.p.: 207.4 - 208.2 °C



6p

***N*-(2,2,3-trichloro-1-hydroxypropyl)quinoline-3-carboxamide (6p):**

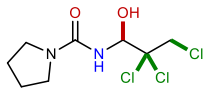
Following **GP D** with Tf_2O (80 μL) and $ClSO_3H$ (80 μL): the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 14.7 mg (44%) of **6p** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 9.24 (d, J = 2.2 Hz, 1H), 8.84 (d, J = 2.2 Hz, 1H), 8.15 – 8.06 (m, 2H), 7.92 – 7.87 (m, 1H), 7.74 – 7.69 (m, 1H), 6.27 (s, 1H), 4.37 (d, J = 12.0 Hz, 1H), 4.24 (d, J = 11.9 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 168.1, 149.94, 149.90, 138.2, 133.1, 130.4, 129.2, 129.0, 128.4, 128.3, 93.5, 77.7, 52.7.

HRMS: calc. for $C_{13}H_{11}Cl_3N_2NaO_2^+$ $(M+Na)^+$, 354.9778, found, 354.9785.

M.p.: 174.9 - 175.6 °C



6q

***N*-(2,2,3-trichloro-1-hydroxypropyl)pyrrolidine-1-carboxamide (6q):**

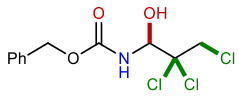
Following **GP D** at a controlled voltage of 1.8 V: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 19.3 mg (70%) of **6q** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 5.82 (d, J = 9.5 Hz, 1H), 4.26 (d, J = 11.9 Hz, 1H), 4.16 (d, J = 11.9 Hz, 1H), 3.48 – 3.33 (m, 4H), 1.98 – 1.89 (m, 4H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 157.3, 94.2, 78.7, 52.9, 46.7, 26.4.

HRMS: calc. for $C_8H_{13}Cl_3N_2NaO_2^+$ $(M+Na)^+$, 296.9935, found, 296.9944.

M.p.: 101.2 - 101.6 °C



6r

Benzyl (2,2,3-trichloro-1-hydroxypropyl)carbamate (6r):

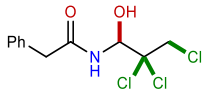
Following **GP D** with Tf_2O (40 μL) and $ClSO_3H$ (50 μL) at a controlled voltage of 1.8 V for 24 h: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 14.1 mg (45%) of **6r** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.39 (d, J = 7.2 Hz, 2H), 7.35 (t, J = 7.3 Hz, 2H), 7.31 (d, J = 7.1 Hz, 1H), 5.70 (s, 1H), 5.14 (d, J = 5.8 Hz, 2H), 4.25 (d, J = 12.0 Hz, 1H), 4.12 (d, J = 12.0 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 157.8, 138.0, 129.5, 129.1, 129.0, 93.2, 79.6, 67.9, 52.7.

HRMS: calc. for $C_{11}H_{12}Cl_3NNaO^+$ $(M+Na)^+$, 333.9775, found, 333.9780.

M.p.: 104.7 - 105.3 °C



6s

2-phenyl-*N*-(2,2,3-trichloro-1-hydroxypropyl)acetamide (6s):

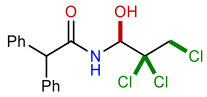
Following **GP D** with Tf_2O (40 μL) and $ClSO_3H$ (50 μL) at a controlled voltage of 2.3 V: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 19.0 mg (64%) of **6s** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.34 – 7.29 (m, 4H), 7.27 – 7.22 (m, 1H), 5.92 (s, 1H), 4.23 (d, J = 12.0 Hz, 1H), 4.09 (d, J = 12.0 Hz, 1H), 3.70 – 3.56 (m, 2H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 173.9, 136.5, 130.3, 129.6, 128.0, 93.3, 76.8, 52.7, 43.6.

HRMS: calc. for $C_{11}H_{12}Cl_3NNaO_2^+$ $(M+Na)^+$, 317.9826, found, 317.9832.

M.p.: 114.4 - 115.1 °C



6t

2,2-diphenyl-*N*-(2,2,3-trichloro-1-hydroxypropyl)acetamide (6t):

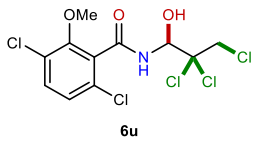
Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 18.3 mg (49%) of **6t** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.36 – 7.28 (m, 8H), 7.27 – 7.21 (m, 2H), 5.98 (s, 1H), 5.19 (s, 1H), 4.22 (d, J = 12.0 Hz, 1H), 4.07 (d, J = 11.9 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 174.5, 140.9, 140.7, 130.2, 129.9, 129.5, 129.4, 128.1, 128.1, 93.3, 76.8, 58.7, 52.6.

HRMS: calc. for $C_{17}H_{16}Cl_3NNaO_2^+$ $(M+Na)^+$, 394.0139, found, 394.0140.

M.p.: 124.5 - 125.2 °C



6u

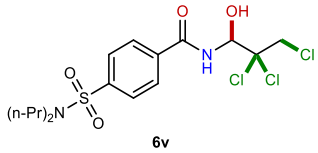
3,6-dichloro-2-methoxy-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6u):

Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 19.1 mg (50%) of **6u** as a colorless oil.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.47 (d, J = 8.7 Hz, 2H), 7.23 (d, J = 8.6 Hz, 2H), 6.14 (s, 1H), 4.32 (d, J = 11.9 Hz, 1H), 4.19 (d, J = 11.9 Hz, 1H), 3.92 (s, 3H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 166.6, 155.1, 134.2, 132.7, 131.0, 127.8, 127.1, 93.1, 77.2, 62.9, 52.8.

HRMS: calc. for $C_{11}H_{10}Cl_5NNaO_3^+$ $(M+Na)^+$, 401.8996, found, 401.8995.



6v

4-(*N*,*N*-dipropylsulfamoyl)-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6v):

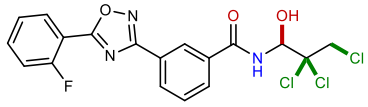
Following **GP D** at a controlled voltage of 2.4 V: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 29.0 mg (65%) of **6v** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 8.01 (d, J = 8.4 Hz, 2H), 7.92 (d, J = 8.4 Hz, 2H), 6.19 (s, 1H), 4.34 (d, J = 12.0 Hz, 1H), 4.21 (d, J = 12.0 Hz, 1H), 3.15 – 3.08 (m, 4H), 1.59 – 1.52 (m, 4H), 0.87 (t, J = 7.4 Hz, 6H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 168.7, 144.3, 138.9, 129.6, 128.2, 93.4, 77.6, 52.6, 51.2, 23.0, 11.4.

HRMS: calc. for $C_{16}H_{23}Cl_3N_2NaO_4S^+$ $(M+Na)^+$, 467.0336, found, 467.0339.

M.p.: 107.7 - 108.9 °C



6w

3-(5-(2-fluorophenyl)-1,2,4-oxadiazol-3-yl)-*N*-(2,2,3-trichloro-1-hydroxypropyl)benzamide (6w):

Following **GP D**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 26.7 mg (60%) of **6w** as a white solid.

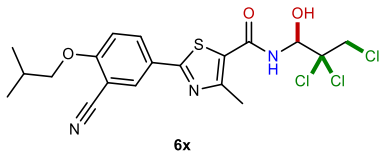
^{1}H NMR (500 MHz, Methanol-d_4) δ 8.63 (t, J = 1.8 Hz, 1H), 8.37 – 8.33 (m, 1H), 8.29 – 8.24 (m, 1H), 8.07 – 8.02 (m, 1H), 7.77 – 7.65 (m, 2H), 7.47 – 7.36 (m, 2H), 6.23 (s, 1H), 4.36 (d, J = 12.0 Hz, 1H), 4.23 (d, J = 12.0 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 174.7 (d, J_{CF} = 4.1 Hz), 169.34, 169.32, 162.2 (d, J_{CF} = 259.2 Hz), 136.5 (d, J_{CF} = 8.7 Hz), 136.3, 132.1, 131.7, 131.5, 130.5, 128.5, 127.8, 126.2 (d, J_{CF} = 3.7 Hz), 118.2 (d, J_{CF} = 21.1 Hz), 113.6 (d, J_{CF} = 11.6 Hz), 93.5, 77.8, 52.7.

^{19}F NMR (471 MHz, Methanol-d_4) δ -110.7.

HRMS: calc. for $C_{18}H_{13}Cl_3FN_3NaO_3^+$ $(M+H)^+$, 465.9899, found, 465.9900.

M.p.: 231.2 - 232.0 °C



6x

2-(3-cyano-4-isobutoxyphenyl)-4-methyl-*N*-(2,2,3-trichloro-1-hydroxypropyl)thiazole-5-carboxamide (6x):

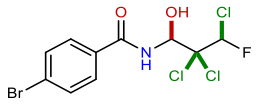
Following **GP D** with Tf_2O (40 μL) and $ClSO_3H$ (60 μL): the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 23.8 mg (50%) of **6x** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 8.22 (d, J = 2.3 Hz, 1H), 8.18 (dd, J = 8.9, 2.4 Hz, 1H), 7.28 (d, J = 8.9 Hz, 1H), 6.12 (s, 1H), 4.34 (d, J = 11.9 Hz, 1H), 4.20 (d, J = 12.0 Hz, 1H), 3.99 (d, J = 6.4 Hz, 2H), 2.70 (s, 3H), 2.26 – 2.09 (m, 1H), 1.10 (d, J = 6.7 Hz, 6H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 167.3, 163.9, 163.7, 157.6, 134.1, 132.8, 127.12, 127. 05, 116.4, 114.5, 103.5, 93.5, 77.4, 76.9, 52.6, 29.4, 19.3, 17.4.

HRMS: calc. for $C_{19}H_{20}Cl_3N_3NaO_3S^+$ $(M+Na)^+$, 498.0183, found, 498.0187.

M.p.: 245.5 - 246.4 ºC



6y

4-bromo-*N*-(2,2,3-trichloro-3-fluoro-1-hydroxypropyl)benzamide (6y):

Following **GP D** with Tf_2O (20 μL) and $ClSO_3H$ (40 μL) at a controlled voltage of 1.5 V: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 15.6 mg (41%, dr > 20:1) of **6y** as a white solid.

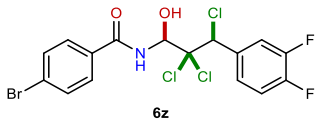
^{1}H NMR (500 MHz, Methanol-d_4) δ 7.79 – 7.76 (m, 2H), 7.66 (d, J = 8.3 Hz, 2H), 6.83 – 6.49 (m, 1H), 6.29 – 6.03 (m, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 169.13, 169.10, 134.18, 134.16, 132.8 (major, mino), 130.6 (major, mino), 127.7 (major, mino), 103.5 (d, J_{CF} = 250.7 Hz), 102.7 (d, J_{CF} = 252.3 Hz), 94.0 (d, J_{CF} = 24.1 Hz), 93.8 (d, J_{CF} = 18.3 Hz), 78.2, 77.5 (d, J_{CF} = 3.4 Hz).

^{19}F NMR (471 MHz, Methanol-d_4) δ -137.9.

HRMS: calc. for $C_{10}H_8BrCl_3FNNaO_2^+$ $(M+Na)^+$, 399.8680, found, 399.8684.

M.p.: 111.3 - 112.0 ºC



6z

4-bromo-*N*-(2,2,3-trichloro-3-(3,4-difluorophenyl)-1-hydroxypropyl)benzamide (6z):

Following **GP D** with Tf_2O (20 μL) and $ClSO_3H$ (50 μL) at a controlled voltage of 1.5 V: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 17.5 mg (37%, dr = 2.9:1) of **6z** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.80 – 7.71 (m, 2H), 7.67 – 7.59 (m, 3H), 7.55 – 7.43 (m, 1H), 7.34 – 7.27 (m, 1H), 6.42 (s, 0.70H), 5.75 (s, 0.74H), 5.73 (s, 0.25H), 5.55 (s, 0.24H).

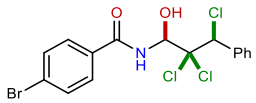
^{13}C NMR (126 MHz, Methanol-d_4) δ 169.1, 168.9, 152.2 (dd, J_{CF} = 249.7, 12.8 Hz), 152.1 (dd, J_{CF} = 249.6, 12.5 Hz), 150.9 (dd, J_{CF} = 247.2, 12.7 Hz), 150.7 (dd, J_{CF} = 246.6, 12.9 Hz), 134.4 (dd, J_{CF}

= 6.0, 4.0 Hz), 134.3, 134.13, 134.12 (dd, J_{CF} = 7.3, 4.1 Hz), 132.84, 132.81, 130.6, 130.5, 128.6 (dd, J_{CF} = 6.9, 3.6 Hz), 127.8 (dd, J_{CF} = 6.7, 3.8 Hz), 127.7, 127.6, 120.7 (d, J_{CF} = 19.0 Hz), 120.1 (d, J_{CF} = 19.1 Hz), 118.0 (d, J_{CF} = 17.8 Hz), 117.5 (d, J_{CF} = 17.9 Hz), 97.8, 96.8, 78.35, 78.30, 68.4, 65.3.

^{19}F NMR (471 MHz, Methanol-d_4) δ -139.3 (d, J = 20.3 Hz), -140.4 (d, J = 20.8 Hz).

HRMS: calc. for $C_{16}H_{11}BrCl_3F_2NNaO_2^+$ $(M+Na)^+$, 493.8899, found, 493.8902.

M.p.: 177.8 - 178.6 °C



6aa

4-bromo-*N*-(2,2,3-trichloro-1-hydroxy-3-phenylpropyl)benzamide (6aa):

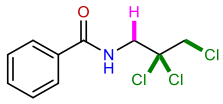
Following **GP D** with Tf_2O (20 μL) and $ClSO_3H$ (50 μL) at a controlled voltage of 1.5 V: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 22.8 mg (52%, dr = 1.3:1) of **6aa** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.77 – 7.74 (m, 1H), 7.72 – 7.69 (m, 2H), 7.67 – 7.62 (m, 3H), 7.43 – 7.39 (m, 3H), 6.43 (s, 0.43H), 5.71 (s, 0.45H), 5.70 (s, 0.56H), 5.54 (s, 0.54H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 169.1, 168.8, 137.0, 136.8, 134.3, 134.2, 132.82, 132.80, 131.7, 130.9, 130.6, 130.52, 130.49, 130.3, 129.2, 128.7, 127.6 (major, minor), 98.3, 97.2, 78.43, 78.37, 69.9, 66.8.

HRMS: calc. for $C_{16}H_{13}BrCl_3NNaO_2^+$ $(M+Na)^+$, 457.9087, found, 457.9089.

M.p.: 151.2 - 151.6 °C



7

***N*-(2,2,3-trichloropropyl)benzamide (7):**

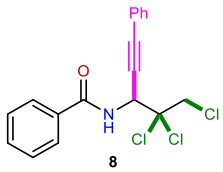
Following **GP E**, using triethylsilane (Et_3SiH) as nucleophile, triethylamine (Et_3N) as base: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 15.2 mg (57%) of **7** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.89 – 7.83 (m, 2H), 7.59 – 7.54 (m, 1H), 7.51 – 7.46 (m, 2H), 4.22 (s, 2H), 4.19 (s, 2H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 170.8, 135.2, 133.1, 129.6, 128.6, 91.2, 53.6, 51.1.

HRMS: calc. for $C_{10}H_{10}Cl_3NNaO^+$ $(M+Na)^+$, 287.9720, found, 287.9726.

M.p.: 122.0 - 122.9 °C



8

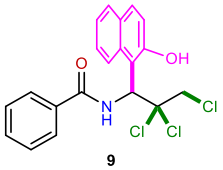
***N*-(4,4,5-trichloro-1-phenylpent-1-yn-3-yl)benzamide (8):**

Following **GP E**, using trimethyl(phenylethynyl)silane as nucleophile, $NaHCO_3$ as base: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 13.6 mg (37%) of **8** as a colorless oil.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.93 – 7.88 (m, 2H), 7.60 – 7.57 (m, 1H), 7.52 – 7.48 (m, 4H), 7.40 – 7.34 (m, 3H), 6.15 (s, 1H), 4.44 (d, J = 12.4 Hz, 1H), 4.35 (d, J = 12.3 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 170.1, 134.8, 133.3, 132.9, 130.3, 129.6, 129.6, 129.0, 123.0, 93.2, 87.0, 83.5, 53.5, 52.7.

HRMS: calc. for $C_{18}H_{14}Cl_3NNaO^+$ $(M+Na)^+$, 388.0033, found, 388.0034.



9

***N*-(2,2,3-trichloro-1-(2-hydroxynaphthalen-1-yl)propyl)benzamide (9):**

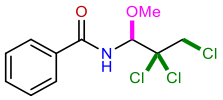
Following **GP E**, using 2-naphthol as nucleophile, $NaHCO_3$ as base: the desired pure prod uct was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 14.3 mg (35%) of **9** as a white solid.

^{1}H NMR (500 MHz, DMSO-d_6) δ 11.05 (s, 1H), 9.15 (d, J = 9.8 Hz, 1H), 8.26 (d, J = 8.8 Hz, 1H), 7.88 – 7.84 (m, 3H), 7.82 (s, 1H), 7.64 – 7.56 (m, 2H), 7.51 (t, J = 7.6 Hz, 2H), 7.35 (t, J = 7.4 Hz, 1H), 7.22 (d, J = 8.9 Hz, 1H), 7.10 (d, J = 9.8 Hz, 1H), 5.06 (d, J = 12.7 Hz, 1H), 4.22 (d, J = 12.7 Hz, 1H).

^{13}C NMR (126 MHz, DMSO-d_6) δ 165.7, 154.1, 133.7, 133.6, 131.9, 131.1, 128.6, 128.5, 128.2, 127.3, 126.9, 123.3, 122.9, 118.8, 112.8, 96.0, 56.2, 54.1.

HRMS: calc. for $C_{20}H_{16}Cl_3NNaO_2^+$ $(M+Na)^+$, 430.0139, found, 430.0146.

M.p.: 178.9 - 179.7 °C



10

***N*-(2,2,3-trichloro-1-methoxypropyl)benzamide (10):**

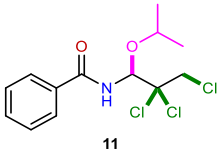
Following **GP E**, using dimthyl malonate as nucleophile, $NaHCO_3$ as base: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 13.6 mg (46%) of **10** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.91 – 7.86 (m, 2H), 7.63 – 7.56 (m, 1H), 7.54 – 7.49 (m, 2H), 5.91 (s, 1H), 4.35 (d, J = 12.0 Hz, 1H), 4.21 (d, J = 12.0 Hz, 1H), 3.48 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-d) δ 171.4, 134.8, 133.4, 129.7, 128.8, 91.9, 84.8, 57.0, 52.7.

HRMS: calc. for $C_{11}H_{12}Cl_3NNaO_2^+$ $(M+Na)^+$, 317.9826, found, 317.9834.

M.p.: 107.0 - 107.9 °C



11

***N*-(2,2,3-trichloro-1-isopropoxypropyl)benzamide (11):**

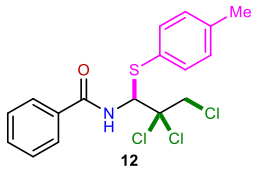
Following **GP E**, using allyltrimethylsilane as nucleophile, $NaHCO_3$ as base: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 16.2 mg (50%) of **11** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.92 – 7.80 (m, 2H), 7.64 – 7.56 (m, 1H), 7.50 (t, J = 7.7 Hz, 2H), 6.07 (s, 1H), 4.34 (d, J = 11.9 Hz, 1H), 4.20 (d, J = 11.9 Hz, 1H), 4.00 – 3.90 (m, 1H), 1.28 (d, J = 6.1 Hz, 3H), 1.20 (d, J = 6.2 Hz, 3H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 171.1, 135.0, 133.3, 129.6, 128.8, 92.5, 81.6, 72.4, 52.5, 23.3, 21.8.

HRMS: calc. for $C_{13}H_{16}Cl_3NNaO_2^+$ $(M+Na)^+$, 346.0139, found, 346.0143.

M.p.: 116.6 - 117.3 °C



12

***N*-(2,2,3-trichloro-1-(p-tolylthio)propyl)benzamide (12):**

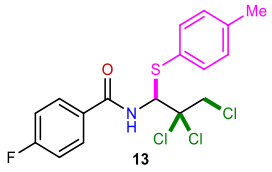
Following **GP E**, using p-toluenethiol as nucleophile, $NaHCO_3$ as base: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 15.2 mg (39%) of **12** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.78 – 7.62 (m, 2H), 7.55 (t, J = 7.4 Hz, 1H), 7.51 – 7.42 (m, 4H), 7.14 (d, J = 7.7 Hz, 2H), 6.35 – 6.10 (m, 1H), 4.59 (d, J = 12.2 Hz, 1H), 4.38 (d, J = 12.2 Hz, 1H), 2.31 (s, 3H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 170.1, 140.6, 135.6, 135.0, 133.1, 131.1, 129.5, 129.1, 128.7, 93.6, 68.1, 54.1, 21.2.

HRMS: calc. for $C_{17}H_{16}Cl_3NNaOS^+$ $(M+Na)^+$, 409.9910, found, 409.9912.

M.p.: 135.6 - 136.2 °C



13

4-fluoro-*N*-(2,2,3-trichloro-1-(p-tolylthio)propyl)benzamide (13):

Following **GP E**, using p-toluenethiol as nucleophile, $NaHCO_3$ as base: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 20.3 mg (50%) of **13** as a white solid.

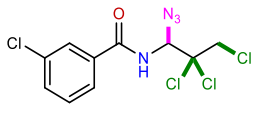
^{1}H NMR (500 MHz, Methanol-d_4) δ 7.80 – 7.74 (m, 2H), 7.47 (d, J = 8.1 Hz, 2H), 7.21 – 7.16 (m, 2H), 7.14 (d, J = 7.9 Hz, 2H), 6.23 (s, 1H), 4.59 (d, J = 12.2 Hz, 1H), 4.38 (d, J = 12.3 Hz, 1H), 2.31 (s, 3H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 168.9, 166.4 (d, J = 250.9 Hz), 140.6, 135.6, 131.4 (d, J = 9.2 Hz), 131.3 (d, J = 3.1 Hz), 131.1, 129.1, 116.4 (d, J = 22.4 Hz), 93.5, 68.1, 54.0, 21.2.

^{19}F NMR (471 MHz, Methanol-d_4) δ -109.8.

HRMS: calc. for $C_{17}H_{15}Cl_3FNNaOS^+$ $(M+Na)^+$, 427.9816, found, 427.9821.

M.p.: 134.3 - 135.0 °C



14

***N*-(1-azido-2,2,3-trichloropropyl)-3-chlorobenzamide (14):**

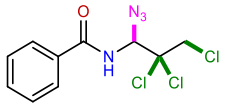
Following **GP E**, using azidotrimethylsilane ($TMSN_3$) as nucleophile, Et_3N as base: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 26.3 mg (77%) of **14** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.89 (t, J = 1.9 Hz, 1H), 7.84 – 7.80 (m, 1H), 7.64 – 7.60 (m, 1H), 7.51 (t, J = 7.9 Hz, 1H), 6.39 (s, 1H), 4.32 (d, J = 12.3 Hz, 1H), 4.27 (d, J = 12.4 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 169.5, 136.2, 135.7, 133.5, 131.3, 129.1, 127.5, 91.3, 72.0, 52.6

HRMS: calc. for $C_{10}H_8Cl_4N_4NaO^+$ $(M+Na)^+$, 362.9344, found, 362.9336.

M.p.: 103.0 - 103.5 °C



15

***N*-(1-azido-2,2,3-trichloropropyl)benzamide (15):**

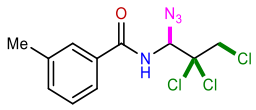
Following **GP E**, using $TMSN_3$ as nucleophile, Et_3N as base: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 24.3 mg (79%) of **15** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.93 – 7.86 (m, 2H), 7.64 – 7.58 (m, 1H), 7.55 – 7.49 (m, 2H), 6.40 (s, 1H), 4.32 (d, J = 12.3 Hz, 1H), 4.27 (d, J = 12.4 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 171.0, 134.2, 133.6, 129.7, 129.1, 91.4, 72.0, 52.6.

HRMS: calc. for $C_{10}H_9Cl_3N_4NaO^+$ $(M+Na)^+$, 328.9734, found, 328.9740.

M.p.: 108.8 - 109.5 °C



16

***N*-(1-azido-2,2,3-trichloropropyl)-3-methylbenzamide (16):**

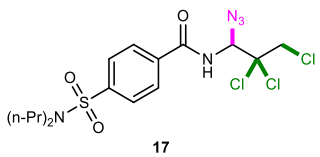
Following **GP E**, using $TMSN_3$ as nucleophile, Et_3N as base: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 20.6 mg (64%) of **16** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.71 (s, 1H), 7.68 (d, J = 7.7 Hz, 1H), 7.43 (d, J = 7.6 Hz, 1H), 7.39 (t, J = 7.5 Hz, 1H), 6.40 (s, 1H), 4.32 (d, J = 12.4 Hz, 1H), 4.27 (d, J = 12.4 Hz, 1H), 2.43 (s, 3H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 171.2, 139.8, 134.3, 134.2, 129.6, 129.5, 126.2, 91.4, 72.0, 52.6, 21.3.

HRMS: calc. for $C_{11}H_{11}Cl_3N_4NaO^+$ $(M+Na)^+$, 342.9891, found, 342.9894.

M.p.: 84.1 - 84.5 °C



17

***N*-(1-azido-2,2,3-trichloropropyl)-4-(*N*,*N*-dipropylsulfamoyl)benzamide (17):**

Following **GP E**, using $TMSN_3$ as nucleophile, Et_3N as base: the desired pure product was purified

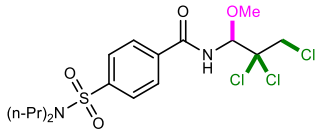
by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 33.9 mg (72%) of **17** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 8.05 (d, J = 8.5 Hz, 2H), 7.95 (d, J = 8.5 Hz, 2H), 6.40 (s, 1H), 4.33 (d, J = 12.4 Hz, 1H), 4.29 (d, J = 12.3 Hz, 1H), 3.16 – 3.10 (m, 4H), 1.60 – 1.52 (m, 4H), 0.89 (t, J = 7.4 Hz, 6H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 169.6, 144.9, 138.0, 130.0, 128.3, 91.3, 72.0, 52.6, 51.3, 23.1, 11.4.

HRMS: calc. for $C_{16}H_{22}Cl_3N_5NaO_3S^+$ $(M+Na)^+$, 492.0401, found, 492.0404.

M.p.: 107.6 - 108.5 °C



18

4-(*N*,*N*-dipropylsulfamoyl)-*N*-(2,2,3-trichloro-1-methoxypropyl)benzamide (18):

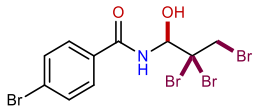
Following **GP E**, using dimthyl malonate as nucleophile, $NaHCO_3$ as base: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 22.5 mg (49%) of **18** as as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 8.04 (d, J = 8.5 Hz, 2H), 7.94 (d, J = 8.5 Hz, 2H), 5.91 (s, 1H), 4.35 (d, J = 12.0 Hz, 1H), 4.22 (d, J = 12.0 Hz, 1H), 3.49 (s, 3H), 3.17 – 3.09 (m, 4H), 1.60 – 1.52 (m, 4H), 0.89 (t, J = 7.4 Hz, 6H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 170.0, 144.6, 138.6, 129.8, 128.3, 91.8, 84.9, 57.1, 52.7, 51.3, 23.1, 11.4.

HRMS: calc. for $C_{17}H_{25}Cl_3N_2NaO_4S^+$ $(M+Na)^+$, 481.0493, found, 481.0498.

M.p.: 107.9 - 108.4 °C



19a

4-bromo-*N*-(2,2,3-tribromo-1-hydroxypropyl)benzamide (19a):

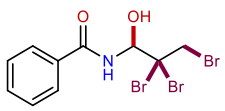
Following **GP F**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 33.2 mg (67%) of **19a** as a white solid.

^{1}H NMR (400 MHz, Methanol-d_4) δ 7.81 – 7.72 (m, 2H), 7.69 – 7.63 (m, 2H), 6.03 (s, 1H), 4.49 (d, J = 11.3 Hz, 1H), 4.30 (d, J = 11.2 Hz, 1H).

^{13}C NMR (101 MHz, Methanol-d_4) δ 168.9, 134.3, 132.9, 130.5, 127.6, 78.3, 73.7, 42.9.

HRMS: calc. for $C_{10}H_9Br_4NNaO_2^+$ $(M+Na)^+$, 513.7259, found, 513.7252.

M.p.: 134.2 - 134.8 °C



19b

***N*-(2,2,3-tribromo-1-hydroxypropyl)benzamide (19b):**

Following **GP F**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 29.1 mg (70%) of **19b** as a white

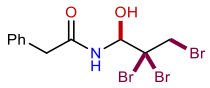
solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.87 – 7.83 (m, 2H), 7.61 – 7.56 (m, 1H), 7.53 – 7.48 (m, 2H), 6.04 (s, 1H), 4.50 (d, J = 11.2 Hz, 1H), 4.31 (d, J = 11.2 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 169.8, 135.2, 133.2, 129.7, 128.6, 78.2, 73.9, 42.9.

HRMS: calc. for $C_{10}H_{10}Br_3NNaO_2^+$ $(M+Na)^+$, 437.8133, found, 437.8137.

M.p.: 119.7 - 120.5 °C



19c

2-phenyl-*N*-(2,2,3-tribromo-1-hydroxypropyl)acetamide (19c):

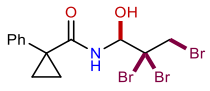
Following **GP F**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 14.2 mg (33%) of **19c** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.36 – 7.29 (m, 4H), 7.27 – 7.21 (m, 1H), 5.78 (s, 1H), 4.39 (d, J = 11.3 Hz, 1H), 4.18 (d, J = 11.3 Hz, 1H), 3.64 (q, J = 14.7 Hz, 2H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 173.6, 136.3, 130.4, 129.6, 128.0, 77.6, 73.3, 43.6, 42.9.

HRMS: calc. for $C_{11}H_{12}Br_3NNaO_2^+$ $(M+Na)^+$, 449.8310, found, 449.8317.

M.p.: 121.0 - 121.7 °C



19d

1-phenyl-*N*-(2,2,3-tribromo-1-hydroxypropyl)cyclopropane-1-carboxamide (19d):

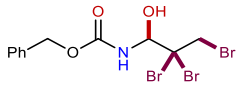
Following **GP F**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 20.5 mg (45%) of **19d** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.50 – 7.47 (m, 2H), 7.43 (t, J = 7.6 Hz, 2H), 7.38 – 7.34 (m, 1H), 5.65 – 5.56 (m, 1H), 4.33 (d, J = 11.2 Hz, 1H), 4.13 (d, J = 11.3 Hz, 1H), 1.64 – 1.58 (m, 1H), 1.57 – 1.52 (m, 1H), 1.26 – 1.21 (m, 1H), 1.15 – 1.10 (m, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 175.7, 139.8, 132.3, 130.3, 129.5, 77.7, 73.8, 42.4, 31.7, 16.9, 16.2.

HRMS: calc. for $C_{13}H_{14}Br_3NNaO_2^+$ $(M+Na)^+$, 475.8467, found, 475.8467.

M.p.: 124.2 - 125.1 °C



19e

Benzyl (2,2,3-tribromo-1-hydroxypropyl)carbamate (19e):

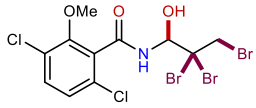
Following **GP F**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 28.5 mg (64%) of **19e** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.39 (d, J = 6.9 Hz, 2H), 7.36 – 7.29 (m, 3H), 5.57 (s, 1H), 5.15 (s, 2H), 4.41 (d, J = 11.2 Hz, 1H), 4.23 (d, J = 11.2 Hz, 1H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 157.6, 137.9, 129.5, 129.11, 128.99, 80.4, 73.4, 67.9, 43.1.

HRMS: calc. for $C_{11}H_{12}Br_3NNaO_3^+$ $(M+Na)^+$, 465.8260, found, 465.8267.

M.p.: 90.5 - 91.3 °C



19f

3,6-dichloro-2-methoxy-*N*-(2,2,3-tribromo-1-hydroxypropyl)benzamide (19f):

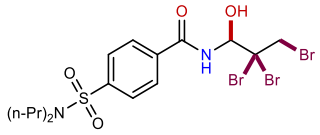
Following **GP F**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 33.5 mg (65%) of **19f** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 7.47 (d, J = 8.7 Hz, 1H), 7.23 (d, J = 8.7 Hz, 1H), 6.03 (s, 1H), 4.47 (d, J = 11.2 Hz, 1H), 4.30 (d, J = 11.2 Hz, 1H), 3.94 (s, 3H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 166.5, 155.1, 134.3, 132.7, 131.1, 127.8, 127.1, 78.2, 72.6, 63.0, 43.2.

HRMS: calc. for $C_{11}H_{10}Br_3Cl_2NNaO_3^+$ $(M+Na)^+$, 533.7480, found, 533.7490.

M.p.: 102.2 - 102.8 °C



19g

4-(*N*,*N*-dipropylsulfamoyl)-*N*-(2,2,3-tribromo-1-hydroxypropyl)benzamide (19g):

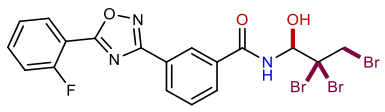
Following **GP F**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 35.9 mg (62%) of **19g** as a white solid.

^{1}H NMR (500 MHz, Methanol-d_4) δ 8.01 (d, J = 8.6 Hz, 2H), 7.93 (d, J = 8.5 Hz, 2H), 6.06 (s, 1H), 4.50 (d, J = 11.2 Hz, 1H), 4.31 (d, J = 11.2 Hz, 1H), 3.16 – 3.08 (m, 4H), 1.61 – 1.52 (m, 4H), 0.88 (t, J = 7.4 Hz, 6H).

^{13}C NMR (126 MHz, Methanol-d_4) δ 168.6, 144.4, 139.1, 129.6, 128.3, 78.4, 73.5, 51.3, 42.9, 23.1, 11.4.

HRMS: calc. for $C_{16}H_{23}Br_3N_2NaO_4S^+$ $(M+Na)^+$, 598.8821, found, 598.8819.

M.p.: 122.4 - 123.3 °C



19h

3-(5-(2-fluorophenyl)-1,2,4-oxadiazol-3-yl)-*N*-(2,2,3-tribromo-1-hydroxypropyl)benzamide (19h):

Following **GP F**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 31.8 mg (55%) of **19h** as a white solid.

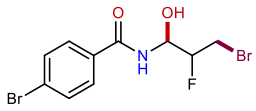
^{1}H NMR (500 MHz, DMSO-d_6) δ 8.94 (d, J = 8.9 Hz, 1H), 8.57 (t, J = 1.8 Hz, 1H), 8.32 – 8.23 (m, 2H), 8.16 – 8.10 (m, 1H), 7.86 – 7.79 (m, 1H), 7.74 (t, J = 7.8 Hz, 1H), 7.60 – 7.50 (m, 2H), 7.40 (d, J = 5.2 Hz, 1H), 6.05 – 5.99 (m, 1H), 4.52 – 4.41 (m, 2H).

^{13}C NMR (126 MHz, DMSO-d_6) δ 172.7 (d, J = 4.2 Hz), 167.6, 166.0, 160.0 (d, J_{CF} = 257.9 Hz), 135.8 (d, J_{CF} = 9.0 Hz), 134.9, 130.94, 130.90, 130.1, 129.4, 126.7, 126.1, 125.6 (d, J_{CF} = 3.5 Hz), 117.4 (d, J_{CF} = 20.5 Hz), 111.7 (d, J_{CF} = 11.2 Hz), 77.3, 74.1, 43.1.

^{19}F NMR (471 MHz, DMSO-d_6) δ -109.4.

HRMS: calc. for $C_{18}H_{13}Br_3FN_3NaO_3^+$ $(M+Na)^+$, 597.8383, found, 597.8384.

M.p.: 169.2 - 170.1 °C



19i

4-bromo-*N*-(3-bromo-2-fluoro-1-hydroxypropyl)benzamide (19i):

Following **GP F**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 18.5 mg (52%, dr = 1.2:1) of **19i** as a white solid.

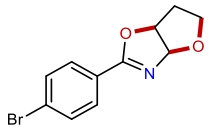
^{1}H NMR (500 MHz, DMSO-d_6) δ 9.09 (d, J = 8.7 Hz, 0.53H), 8.94 (d, J = 8.6 Hz, 0.43H), 7.84 (d, J = 8.2 Hz, 2H), 7.70 (t, J = 7.7 Hz, 2H), 5.66 – 5.55 (m, 1H), 4.81 – 4.61 (m, 1H), 3.97 – 3.60 (m, 3H).

^{13}C NMR (126 MHz, DMSO-d_6) δ 165.4, 165.2, 133.05, 132.98, 131.4, 131.3, 129.7, 129.6, 125.41, 125.40, 92.2 (d, J_{CF} = 179.7 Hz), 90.9 (d, J_{CF} = 178.6 Hz), 72.9 (d, J_{CF} = 24.8 Hz), 72.7 (d, J_{CF} = 26.2 Hz), 33.0 (d, J_{CF} = 20.9 Hz), 31.7 (d, J_{CF} = 22.1 Hz).

^{19}F NMR (471 MHz, DMSO-d_6) δ -189.0, -190.2.

HRMS: calc. for $C_{10}H_{10}Br_2FNNaO_2^+$ $(M+Na)^+$, 377.8934, found, 377.8940.

M.p.: 105.3 - 105.9 °C



21a

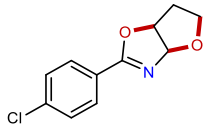
2-(4-bromophenyl)-3a,5,6,6a-tetrahydrofuro[2,3-*d*]oxazole (21a):

Following **GP G**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 5.3 mg (40%) of **21a** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-d) δ 7.87 (d, J = 8.5 Hz, 2H), 7.57 (d, J = 8.6 Hz, 2H), 6.21 (d, J = 5.4 Hz, 1H), 5.15 (t, J = 5.7 Hz, 1H), 4.06 – 4.00 (m, 1H), 3.56 – 3.48 (m, 1H), 2.22 (dd, J = 13.8, 4.8 Hz, 1H), 2.12 – 2.03 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-d) δ 166.3, 131.7, 130.3, 126.9, 125.7, 101.3, 82.6, 64.3, 33.6.

HRMS: calc. for $C_{11}H_{10}BrNNaO_2^+$ $(M+H)^+$, 289.9787, found, 289.9792.



21b

2-(4-chlorophenyl)-3a,5,6,6a-tetrahydrofuro[2,3-*d*]oxazole (21b):

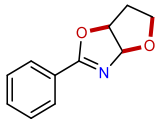
Following **GP G**: the desired pure product was purified by preparative thin-layer chromatography

(PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 4.1 mg (37%) of **21b** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 8.3 Hz, 2H), 7.40 (d, *J* = 8.3 Hz, 2H), 6.22 (d, *J* = 5.5 Hz, 1H), 5.15 (t, *J* = 5.7 Hz, 1H), 4.03 (t, *J* = 8.5 Hz, 1H), 3.59 – 3.47 (m, 1H), 2.22 (dd, *J* = 13.8, 4.8 Hz, 1H), 2.13 – 2.03 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 166.2, 138.4, 130.1, 128.7, 125.2, 101.3, 82.6, 64.3, 33.6.

HRMS: calc. for $C_{11}H_{11}ClNO_2^+$ $(M+H)^+$, 224.0473, found, 224.0476.



21c

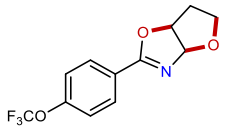
2-phenyl-3a,5,6,6a-tetrahydrofuro[2,3-*d*]oxazole (21c):

Following **GP G**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 3.6 mg (38%) of **21c** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.02 – 7.99 (m, 2H), 7.52 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 6.23 (d, *J* = 5.4 Hz, 1H), 5.15 (t, *J* = 5.7 Hz, 1H), 4.02 (t, *J* = 8.5 Hz, 1H), 3.58 – 3.49 (m, 1H), 2.23 (dd, *J* = 13.7, 4.8 Hz, 1H), 2.12 – 2.03 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.1, 132.1, 128.8, 128.4, 126.8, 101.3, 82.3, 64.2, 33.7.

HRMS: calc. for $C_{11}H_{11}NNaO_2^+$ $(M+Na)^+$, 212.0682, found, 212.0686.



21d

2-(4-(trifluoromethoxy)phenyl)-3a,5,6,6a-tetrahydrofuro[2,3-*d*]oxazole (21d):

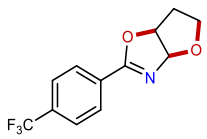
Following **GP G**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 6.0 mg (44%) of **21d** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.05 (d, *J* = 8.8 Hz, 2H), 7.29 – 7.25 (m, 2H), 6.23 (d, *J* = 5.5 Hz, 1H), 5.16 (t, *J* = 5.7 Hz, 1H), 4.07 – 4.00 (m, 1H), 3.57 – 3.48 (m, 1H), 2.27 – 2.19 (m, 1H), 2.14 – 2.03 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 165.9, 151.9, 130.7, 125.3, 120.4, 120.3 (q, J_{CF} = 258.5 Hz), 101.4, 82.6, 64.3, 33.6.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -57.7.

HRMS: calc. for $C_{12}H_{11}F_3NO_3^+$ $(M+H)^+$, 274.0686, found, 274.0689.



21e

2-(4-(trifluoromethyl)phenyl)-3a,5,6,6a-tetrahydrofuro[2,3-*d*]oxazole (21e):

Following **GP G**: the desired pure product was purified by preparative thin-layer chromatography

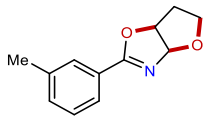
(PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 4.5 mg (35%) of **21e** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.12 (d, *J* = 8.0 Hz, 2H), 7.69 (d, *J* = 8.0 Hz, 2H), 6.25 (d, *J* = 5.4 Hz, 1H), 5.19 (t, *J* = 5.8 Hz, 1H), 4.05 (t, *J* = 8.5 Hz, 1H), 3.56 – 3.49 (m, 1H), 2.25 (dd, *J* = 13.9, 4.7 Hz, 1H), 2.15 – 2.05 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 165.8, 133.7 (q, *J* = 32.7 Hz), 130.1, 129.2, 125.4 (q, *J* = 3.8 Hz), 123.7 (q, *J* = 272.7 Hz), 101.3, 82.7, 64.3, 33.6.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -63.0.

HRMS: calc. for $C_{12}H_{11}F_3NO_2^+$ $(M+H)^+$, 258.0736, found, 258.0742.



21f

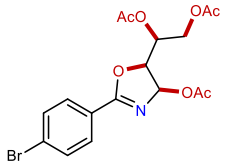
2-(m-tolyl)-3a,5,6,6a-tetrahydrofuro[2,3-*d*]oxazole (21f):

Following **GP G**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 4.2 mg (41%) of **21f** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.85 (s, 1H), 7.79 (d, *J* = 6.5 Hz, 1H), 7.34 – 7.29 (m, 2H), 6.22 (d, *J* = 5.4 Hz, 1H), 5.14 (t, *J* = 5.7 Hz, 1H), 4.02 (t, *J* = 8.4 Hz, 1H), 3.59 – 3.48 (m, 1H), 2.38 (s, 3H), 2.22 (dd, *J* = 13.7, 4.8 Hz, 1H), 2.11 – 2.02 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 167.3, 138.2, 132.8, 129.4, 128.3, 126.6, 125.9, 101.3, 82.2, 64.2, 33.7, 21.2.

HRMS: calc. for $C_{12}H_{14}NO_2^+$ $(M+H)^+$, 204.1019, found, 204.1023.



22a

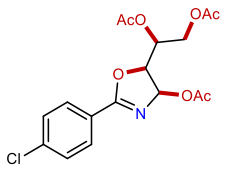
1-(4-acetoxy-2-(4-bromophenyl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22a):

Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 39.4 mg (46%, dr = 3:1) of **22a** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 8.2 Hz, 2H), 6.85 (d, *J* = 7.0 Hz, 0.25H), 6.67 (d, *J* = 3.7 Hz, 0.75H), 5.48 – 5.40 (m, 0.25H), 5.37 – 5.32 (m, 0.75H), 4.80 – 4.73 (m, 0.29H), 4.70 – 4.66 (m, 0.74H), 4.48 – 4.42 (m, 0.74H), 4.28 – 4.19 (m, 1.29H), 2.12 (s, 3H), 2.06 (s, 2.12H), 2.05 (s, 1.76H), 2.03 (s, 2.14H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 170.3, 169.8, 169.6, 169.4, 169.3, 167.4, 167.3, 131.9, 131.8, 130.3 (major, minor), 127.72, 127.68, 125.4, 124.9, 92.2, 88.4, 82.8, 78.4, 69.4, 67.3, 62.9, 61.8, 21.03, 21.01, 20.70, 20.69, 20.64, 20.58.

HRMS: calc. for $C_{17}H_{18}BrNNaO_7^+$ $(M+Na)^+$, 450.0159, found, 450.0154.



22b

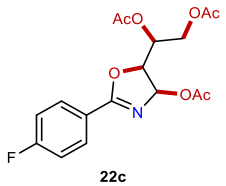
1-(4-acetoxy-2-(4-chlorophenyl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22b):

Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 39.9 mg (52%, dr = 5.6:1) of **22b** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.94 – 7.91 (m, 2H), 7.43 – 7.38 (m, 2H), 6.85 (d, *J* = 7.0 Hz, 0.15H), 6.68 (d, *J* = 3.7 Hz, 0.84H), 5.46 – 5.40 (m, 0.15H), 5.37 – 5.33 (m, 0.85H), 4.80 – 4.74 (m, 0.15H), 4.71 – 4.67 (m, 0.79H), 4.48 – 4.43 (m, 0.86H), 4.28 – 4.19 (m, 1.21H), 2.12 (s, 3H), 2.07 (s, 2.52H), 2.06 (s, 0.45H), 2.05 (s, 0.42H), 2.03 (s, 2.57H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.3, 169.9, 169.6, 167.3, 139.1, 130.2, 128.9, 124.4, 92.2, 82.8, 69.4, 61.9, 21.0, 20.7, 20.6.

HRMS: calc. for $C_{17}H_{18}ClNNaO_7^+$ $(M+Na)^+$, 406.0664, found, 406.0667.



22c

1-(4-acetoxy-2-(4-fluorophenyl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22c):

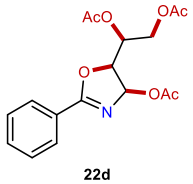
Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 32.3 mg (44%, dr = 4.2:1) of **22c** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.07 – 7.95 (m, 2H), 7.15 – 7.10 (m, 2H), 6.87 – 6.84 (m, 0.19H), 6.70 – 6.63 (m, 0.80H), 5.48 – 5.41 (m, 0.20H), 5.39 – 5.31 (m, 0.80H), 4.81 – 4.74 (m, 0.21H), 4.71 – 4.62 (m, 0.79H), 4.50 – 4.42 (m, 0.76H), 4.32 – 4.21 (m, 1.26H), 2.13 (s, 3H), 2.10 (s, 0.73H), 2.07 (s, 3H), 2.04 (s, 2.31H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.4, 169.9, 169.7, 167.3, 165.5 (d, J_{CF} = 254.0 Hz), 131.4 (d, J_{CF} = 9.2 Hz), 122.2 (d, J_{CF} = 3.3 Hz), 115.8 (d, J_{CF} = 22.1 Hz), 92.3, 82.8, 69.4, 61.9, 21.1, 20.7, 20.6.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -105.4, -105.5.

HRMS: calc. for $C_{17}H_{18}FNNaO_7^+$ $(M+Na)^+$, 390.0960, found, 390.0965.

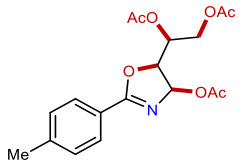


22d

1-(4-acetoxy-2-phenyl-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22d):

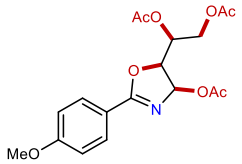
Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 30.0 mg (43%, dr = 3.6:1) of **22d** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.00 – 7.96 (m, 2H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.42 (t, *J* = 7.7 Hz, 2H), 6.86 (d, *J* = 7.0 Hz, 0.21H), 6.68 (d, *J* = 3.6 Hz, 0.75H), 5.47 – 5.41 (m, 0.22H), 5.38 – 5.30 (m, 0.78H), 4.79 – 4.73 (m, 0.25H), 4.69 – 4.65 (m, 0.78H), 4.49 – 4.42 (m, 0.77H), 4.30 – 4.19 (m, 1.24H), 2.11 (s, 3H), 2.08 (s, 0.77H), 2.06 (s, 2H), 2.05 (s, 1H), 2.03 (s, 2.25H).
^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 170.3, 169.9, 169.6, 169.5, 169.3, 168.2, 168.1, 132.71, 132.66, 128.9 (major, minor), 128.5 (major, minor), 126.0 (major, minor), 92.3, 88.6, 82.6, 78.2, 69.4, 67.4, 62.9, 61.9, 21.0, 20.8, 20.69, 20.66, 20.63, 20.56.
HRMS: calc. for $C_{17}H_{19}NNaO_7^+$ $(M+Na)^+$, 372.1054, found, 372.1055.



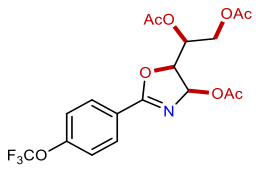
22e

1-(4-acetoxy-2-(p-tolyl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22e):
Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 29.8 mg (41%, dr = 2.9:1) of **22e** as a colorless oil.
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 8.3 Hz, 2H), 6.85 (d, *J* = 6.9 Hz, 0.25H), 6.66 (d, *J* = 3.5 Hz, 0.73H), 5.49 – 5.38 (m, 0.26H), 5.37 – 5.30 (m, 0.72H), 4.77–4.71 (m, 0.27H), 4.69 – 4.59 (m, 0.74H), 4.48 – 4.40 (m, 0.74H), 4.31 – 4.20 (m, 1.27H), 2.39 (s, 3H), 2.11 (s, 3H), 2.08 (s, 0.85H), 2.06 (s, 1.93H), 2.05 (d, *J* = 1.4 Hz, 1.10H), 2.03 (s, 2.13H).
^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 170.3, 169.9, 169.7, 169.5, 169.3, 168.3, 168.2, 143.4, 143.3, 129.2 (major, minor), 128.85, 128.84, 123.1 (major, minor), 92.4, 88.6, 82.5, 78.1, 69.4, 67.4, 62.9, 61.9, 21.6 (major, mino), 21.0, 20.8, 20.68, 20.65, 20.62, 20.56.
HRMS: calc. for $C_{18}H_{21}NNaO_7^+$ $(M+Na)^+$, 386.1210, found, 386.1214.



22f

1-(4-acetoxy-2-(4-methoxyphenyl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22f):
Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 26.6 mg (35%, dr = 3.9:1) of **22f** as a colorless oil.
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.97 – 7.91 (m, 2H), 6.94 – 6.89 (m, 2H), 6.84 (d, *J* = 6.8 Hz, 0.22H), 6.65 (d, *J* = 3.5 Hz, 0.74H), 5.47 – 5.39 (m, 0.20H), 5.35 – 5.33 (m, 0.77H), 4.76 – 4.71 (m, 0.28H), 4.68 – 4.62 (m, 0.75H), 4.48 – 4.43 (m, 0.78H), 4.30 – 4.20 (m, 1.30H), 3.85 (s, 3H), 2.12 (s, 3H), 2.10 (s, 0.85H), 2.07 (s, 2.15H), 2.06 (s, 1.66H), 2.04 (s, 2.32H).
^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.4, 170.0, 169.8, 168.1, 163.2, 130.9, 118.2, 113.9, 92.5, 82.5, 69.5, 62.0, 55.4, 21.1, 20.7, 20.6.
HRMS: calc. for $C_{18}H_{21}NNaO_8^+$ $(M+Na)^+$, 402.1159, found, 402.1159.



22g

1-(4-acetoxy-2-(4-(trifluoromethoxy)phenyl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22g):

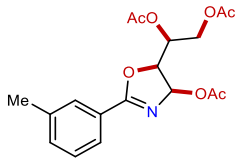
Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 34.7 mg (40%, dr = 2.7:1) of **22g** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.07 – 8.03 (m, 2H), 7.29 – 7.26 (m, 2H), 6.86 (d, *J* = 7.0 Hz, 0.26H), 6.69 (d, *J* = 3.7 Hz, 0.71H), 5.48 – 5.41 (m, 0.26H), 5.39 – 5.33 (m, 0.72H), 4.82 – 4.76 (m, 0.26H), 4.72 – 4.67 (m, 0.73H), 4.49 – 4.41 (m, 0.77H), 4.31 – 4.18 (m, 1.21H), 2.13 (s, 2.18H), 2.10 (s, 0.89H), 2.07 (s, 2.07H), 2.06 (s, 1.79H), 2.04 (s, 2.16H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.3, 169.9, 169.6, 167.0, 152.4, 130.8, 124.4, 120.5, 120.2 (q, J_{CF} = 258.8 Hz), 92.2, 82.9, 69.4, 61.9, 21.0, 20.7, 20.6.

^{19}F NMR (471 MHz, Chloroform-*d*) δ -57.7.

HRMS: calc. for $C_{18}H_{18}F_3NNaO_8^+$ $(M+Na)^+$, 456.0877, found, 456.0875.



22h

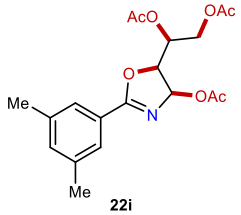
1-(4-acetoxy-2-(m-tolyl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22h):

Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 35.6 mg (49%, dr = 2.3:1) of **22h** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.83 (s, 1H), 7.77 (d, *J* = 7.5 Hz, 1H), 7.36 – 7.29 (m, 2H), 6.86 (d, *J* = 6.9 Hz, 0.30H), 6.68 (d, *J* = 3.6 Hz, 0.70H), 5.46 – 5.42 (m, 0.31H), 5.38 – 5.29 (m, 0.71H), 4.78 – 4.73 (m, 0.32H), 4.70 – 4.64 (m, 0.70H), 4.49 – 4.43 (m, 0.69H), 4.32 – 4.17 (m, 1.33H), 2.37 (s, 3H), 2.12 (s, 2H), 2.09 (s, 1H), 2.07 (s, 2H), 2.06 (s, 2H), 2.04 (s, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 170.4, 169.9, 169.7, 169.5, 169.3, 168.4, 168.3, 138.35, 138.34, 133.54, 133.49, 129.42, 129.40, 128.4 (major, minor), 126.0 (major, minor), 125.8 (major, minor), 92.3, 88.5, 82.5, 78.1, 69.4, 67.4, 63.0, 61.9, 21.1 (major, minor), 21.0, 20.8, 20.71, 20.69, 20.66, 20.6.

HRMS: calc. for $C_{18}H_{21}NNaO_7^+$ $(M+Na)^+$, 386.1210, found, 386.1212.



22i

1-(4-acetoxy-2-(3,5-dimethylphenyl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22i):

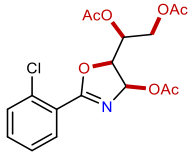
Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography

(PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 40.8 mg (54%, dr = 8.3:1) of **22i** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.61 (s, 2H), 7.16 (s, 1H), 6.67 (d, *J* = 3.6 Hz, 1H), 5.38 – 5.31 (m, 1H), 4.69 – 4.64 (dd, *J* = 5.5, 3.6 Hz, 1H), 4.49 – 4.43 (m, 1H), 4.27 – 4.21 (m, 1H), 2.34 (s, 6H), 2.12 (s, 3H), 2.07 (s, 3H), 2.05 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.4, 169.9, 169.7, 168.6, 138.2, 134.4, 126.6, 125.7, 92.3, 82.5, 69.5, 61.9, 21.1, 21.1, 20.7, 20.6.

HRMS: calc. for $C_{19}H_{23}NNaO_7^+$ $(M+Na)^+$, 400.1367, found, 400.1370.



22j

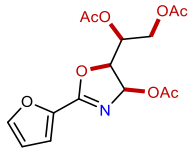
1-(4-acetoxy-2-(2-chlorophenyl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22j):

Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 33.0 mg (43%, dr = 3.5:1) of **22j** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.87 – 7.79 (m, 1H), 7.49 – 7.42 (m, 2H), 7.36 – 7.28 (m, 1H), 6.90 (dd, *J* = 7.4, 2.9 Hz, 0.20H), 6.73 (d, *J* = 3.6 Hz, 0.77H), 5.49 – 5.42 (m, 0.26H), 5.41 – 5.31 (m, 0.74H), 4.84 – 4.76 (m, 0.26H), 4.73 – 4.66 (m, 0.80H), 4.52 – 4.46 (m, 0.70H), 4.37 – 4.19 (m, 1.35H), 2.13 (s, 3H), 2.07 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 170.3, 169.9, 169.5, 169.3, 169.2, 167.2 (major, minor), 133.83, 133.78, 132.8, 132.7, 131.8 (major, minor), 131.0, 130.9, 126.7 (major, minor), 125.7 (major, minor), 92.0, 88.3, 82.6, 78.2, 69.4, 67.3, 63.0, 62.0, 21.0, 20.9, 20.8, 20.74, 20.68, 20.6.

HRMS: calc. for $C_{17}H_{18}ClNNaO_7^+$ $(M+Na)^+$, 406.0664, found, 406.0662.



22k

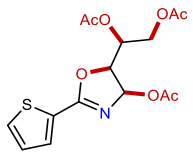
1-(4-acetoxy-2-(furan-2-yl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22k):

Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 18.3 mg (27%, dr = 3.7:1) of **22k** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.60 (s, 1H), 7.12 (d, *J* = 3.5 Hz, 1H), 6.86 (d, *J* = 6.7 Hz, 0.21H), 6.70 (d, *J* = 3.6 Hz, 0.78H), 6.55 – 6.51 (m, 1H), 5.45 – 5.38 (m, 0.21H), 5.35 – 5.29 (m, 0.76H), 4.77 – 4.71 (m, 0.21H), 4.68 – 4.64 (m, 0.83H), 4.47 – 4.42 (m, 0.72H), 4.38 – 4.17 (m, 1.24H), 2.11 (s, 2.22H), 2.09 (s, 0.77H), 2.06 (s, 2.16H), 2.06 – 2.03 (m, 3.81H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 170.3, 169.9, 169.5, 169.51, 169.46, 160.1, 160.0, 146.7, 146.6, 141.4, 141.3, 117.3, 117.2, 112.00, 111.98, 91.9, 88.1, 82.7, 78.3, 69.3, 67.3, 62.8, 61.8, 21.0, 20.8, 20.70 (major, minor), 20.68, 20.6.

HRMS: calc. for $C_{15}H_{17}NNaO_8^+$ $(M+Na)^+$, 362.0846, found, 362.0847.



22l

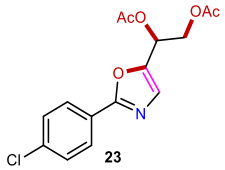
1-(4-acetoxy-2-(thiophen-2-yl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (22l):

Following **GP H**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 21.3 mg (30%, dr = 11:1) of **22l** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.74 – 7.72 (m, 1H), 7.58 – 7.53 (m, 1H), 7.12 – 7.09 (m, 1H), 6.73 – 6.63 (m, 1H), 5.48 – 5.29 (m, 1H), 4.71 – 4.64 (m, 1H), 4.51 – 4.42 (m, 1H), 4.28 – 4.16 (m, 1H), 2.12 (s, 3H), 2.07 (s, 3H), 2.05 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.4, 169.9, 169.7, 163.9, 132.5, 132.1, 128.2, 127.9, 92.3, 83.1, 69.4, 61.9, 21.1, 20.7, 20.6.

HRMS: calc. for $C_{15}H_{17}NNaO_7S^+$ $(M+Na)^+$, 378.0618, found, 378.0618.



23

1-(2-(4-chlorophenyl)oxazol-5-yl)ethane-1,2-diyl diacetate (23):

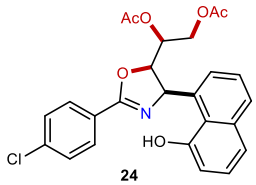
Following **GP I**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 21.4 mg (66%) of **23** as a white solid.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.2 Hz, 2H), 7.44 (d, *J* = 8.1 Hz, 2H), 7.21 (s, 1H), 6.22 – 6.17 (m, 1H), 4.57 – 4.51 (m, 1H), 4.50 – 4.44 (m, 1H), 2.12 (s, 3H), 2.08 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.4, 169.7, 161.3, 146.8, 136.9, 129.1, 127.9, 127.8, 125.5, 64.4, 62.8, 20.8, 20.7.

HRMS: calc. for $C_{15}H_{14}ClNNaO_5^+$ $(M+Na)^+$, 346.0453, found, 346.0451.

M.p.: 69.2 - 70.0 °C



24

1-(2-(4-chlorophenyl)-4-(8-hydroxynaphthalen-1-yl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (24):

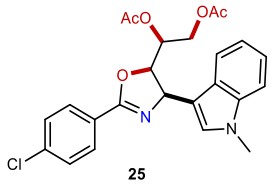
Following **GP I**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 19.7 mg (42%) of **24** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.97 – 7.94 (m, 2H), 7.77 (d, *J* = 8.6 Hz, 1H), 7.69 (d, *J* = 8.1 Hz, 1H), 7.42 (d, *J* = 8.2 Hz, 3H), 7.34 – 7.27 (m, 2H), 6.67 (d, *J* = 8.7 Hz, 1H), 6.20 (d, *J* = 7.9 Hz, 1H), 5.57 – 5.50 (m, 1H), 5.12 – 5.05 (m, 1H), 4.49 – 4.42 (m, 1H), 4.25 – 4.18 (m, 1H), 1.95 (s, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.6, 170.2, 164.0, 153.6, 138.6, 132.5, 130.2, 129.9, 129.4,

129.1, 129.0, 126.9, 124.8, 123.0, 121.6, 119.1, 115.9, 82.9, 71.5, 65.5, 62.1, 20.8, 20.6.

HRMS: calc. for $C_{25}H_{22}ClNNaO_6^+$ $(M+Na)^+$, 490.1028, found, 490.1031.



25

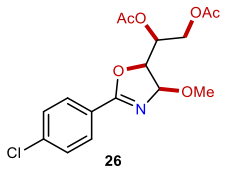
1-(2-(4-chlorophenyl)-4-(1-methyl-1H-indol-3-yl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (25):

Following **GP I**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 20.5 mg (45%) of **25** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.42 (d, *J* = 8.6 Hz, 2H), 7.32 (d, *J* = 8.2 Hz, 1H), 7.25 – 7.21 (m, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 7.04 (s, 1H), 5.49 (d, *J* = 6.3 Hz, 1H), 5.44 – 5.38 (m, 1H), 4.93 (t, *J* = 6.1 Hz, 1H), 4.48 (dd, *J* = 12.2, 3.3 Hz, 1H), 4.23 (dd, *J* = 12.2, 6.1 Hz, 1H), 3.76 (s, 3H), 2.08 (s, 3H), 2.01 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.6, 170.1, 162.0, 138.0, 137.7, 129.8, 128.8, 126.8, 125.8, 125.6, 122.1, 119.5, 118.8, 114.2, 109.7, 83.1, 71.5, 65.8, 62.0, 32.8, 20.9, 20.7.

HRMS: calc. for $C_{24}H_{23}ClN_2NaO_5^+$ $(M+Na)^+$, 477.1188, found, 477.1190.



26

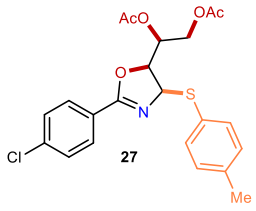
1-(2-(4-chlorophenyl)-4-methoxy-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (26):

Following **GP I**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 18.5 mg (52%) of **26** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.91 (d, *J* = 8.5 Hz, 2H), 7.41 (d, *J* = 8.6 Hz, 2H), 5.37 (d, *J* = 4.3 Hz, 1H), 5.30 (dd, *J* = 5.4, 1.8 Hz, 1H), 4.57 – 4.53 (m, 1H), 4.45 – 4.39 (m, 1H), 4.29 – 4.15 (m, 1H), 3.56 (s, 3H), 2.09 (s, 3H), 2.06 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.4, 169.9, 165.2, 138.6, 130.1, 128.8, 125.2, 99.9, 82.6, 69.8, 61.9, 56.0, 20.72, 20.69.

HRMS: calc. for $C_{16}H_{18}ClNNaO_6^+$ $(M+Na)^+$, 378.0715, found, 378.0719.

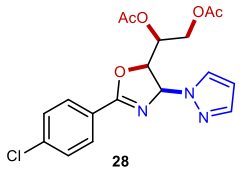


27

1-(2-(4-chlorophenyl)-4-(p-tolylthio)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (27):

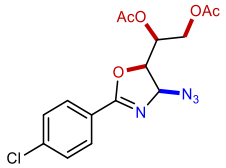
Following **GP I**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 25.5 mg (57%) of **27** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 8.3 Hz, 2H), 7.42 (d, *J* = 7.8 Hz, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 7.08 (d, *J* = 7.9 Hz, 2H), 5.39 (d, *J* = 5.3 Hz, 1H), 5.30 – 5.23 (m, 1H), 4.72 (t, *J* = 5.2 Hz, 1H), 4.34 – 4.30 (m, 1H), 4.19 – 4.11 (m, 1H), 2.30 (s, 3H), 2.06 (s, 3H), 1.97 (s, 3H).
^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.4, 169.9, 163.7, 139.0, 138.4, 134.7, 129.83, 129.79, 128.8, 127.3, 124.9, 83.1, 75.1, 70.5, 61.7, 21.1, 20.7, 20.6.
HRMS: calc. for $C_{22}H_{22}ClNNaO_5S^+$ $(M+Na)^+$, 470.0799, found, 470.0799.



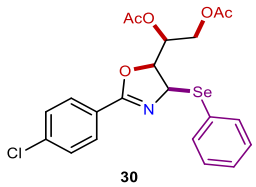
28

1-(2-(4-chlorophenyl)-4-(1*H*-pyrazol-1-yl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (28):
Following **GP I**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 1:1) to afford 20.8 mg (53%) of **28** as a colorless oil.
^{1}H NMR (500 MHz, DMSO-d_6) δ 7.92 – 7.86 (m, 3H), 7.63 – 7.58 (m, 2H), 7.55 – 7.51 (m, 1H), 6.50 (d, *J* = 6.8 Hz, 1H), 5.60 (t, *J* = 5.4 Hz, 1H), 5.23 – 5.18 (m, 1H), 4.48 (t, *J* = 4.9 Hz, 1H), 4.34 – 4.28 (m, 1H), 4.25 – 4.20 (m, 1H), 2.03 (s, 3H), 1.99 (s, 3H).
^{13}C NMR (126 MHz, DMSO-d_6) δ 170.1, 169.7, 162.5, 136.9, 136.1, 129.9, 129.4, 129.0, 128.3, 125.7, 91.4, 83.9, 69.7, 61.5, 20.6, 20.5.
HRMS: calc. for $C_{18}H_{18}ClN_3NaO_5^+$ $(M+Na)^+$, 414.0827, found, 414.0827.



29

1-(4-azido-2-(4-chlorophenyl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (29):
Following **GP I**: the desired pure product was purified by preparative thin-layer chromatography (PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 18.3 mg (50%, dr = 4:1) of **29** as a colorless oil.
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.91 (d, *J* = 8.6 Hz, 2H), 7.42 (d, *J* = 8.6 Hz, 2H), 5.64 (d, *J* = 4.8 Hz, 1H), 5.35 (d, *J* = 4.5 Hz, 0.21H), 5.30 – 5.25 (m, 0.85H), 4.60 – 4.53 (m, 1H), 4.46 – 4.41 (m, 0.87H), 4.27 – 4.20 (m, 1.21H), 2.11 (s, 2.61H), 2.08 (s, 0.80H), 2.06 (s, 2.65H).
^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.3, 169.8, 165.8, 139.1, 130.2, 129.0, 124.3, 83.8, 83.0, 69.7, 61.8, 20.7, 20.6.
HRMS: calc. for $C_{15}H_{15}ClN_4NaO_5^+$ $(M+Na)^+$, 389.0623, found, 389.0629.



30

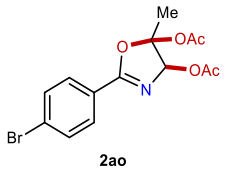
1-(2-(4-chlorophenyl)-4-(phenylselanyl)-4,5-dihydrooxazol-5-yl)ethane-1,2-diyl diacetate (30):
Following **GP I**: the desired pure product was purified by preparative thin-layer chromatography

(PTLC) (eluent: petroleum ether: ethyl acetate = 3:1) to afford 15.4 mg (32%) of **30** as a colorless oil.

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.79 (d, *J* = 8.7 Hz, 2H), 7.61 (d, *J* = 7.4 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H), 7.30 (t, *J* = 7.6 Hz, 1H), 7.22 (t, *J* = 7.5 Hz, 2H), 5.58 (d, *J* = 4.6 Hz, 1H), 5.22 – 5.12 (m, 1H), 4.91 (t, *J* = 5.0 Hz, 1H), 4.30 (dd, *J* = 12.3, 3.7 Hz, 1H), 4.10 (dd, *J* = 12.2, 5.7 Hz, 1H), 2.05 (s, 3H), 1.94 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.3, 169.8, 163.5, 138.3, 136.3, 129.7, 129.1, 128.80, 128.78, 126.4, 124.9, 84.6, 70.6, 68.5, 61.7, 20.7, 20.6.

HRMS: calc. for $C_{21}H_{20}ClNNaO_5Se^+$ $(M+Na)^+$, 504.0087, found, 504.0087.

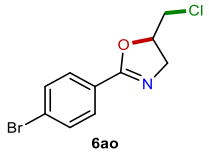


2ao

2-(4-bromophenyl)-5-methyl-4,5-dihydrooxazole-4,5-diyl diacetate (2ao):

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.6 Hz, 2H), 7.58 (d, *J* = 8.5 Hz, 2H), 6.68 (s, 1H), 2.17 (s, 3H), 2.08 (s, 3H), 1.83 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 169.2, 168.6, 166.2, 131.9, 130.4, 127.7, 125.0, 110.6, 94.4, 21.7, 20.8, 18.5.

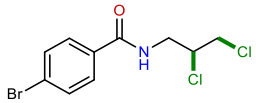


6ao

2-(4-bromophenyl)-5-(chloromethyl)-4,5-dihydrooxazole (6ao):

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.80 (d, *J* = 8.5 Hz, 2H), 7.55 (d, *J* = 8.6 Hz, 2H), 4.98 – 4.88 (m, 1H), 4.17 (dd, *J* = 15.2, 9.7 Hz, 1H), 3.93 (dd, *J* = 15.2, 6.7 Hz, 1H), 3.68 (d, *J* = 5.3 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 163.0, 131.7, 129.7, 126.22, 126.19, 78.3, 58.3, 45.4.

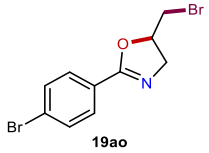


6ao'

4-bromo-*N*-(2,3-dichloropropyl)benzamide (6ao'):

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.66 (d, *J* = 8.6 Hz, 2H), 7.59 (d, *J* = 8.5 Hz, 2H), 4.40 – 4.33 (m, 1H), 4.07 (dd, *J* = 14.3, 4.2 Hz, 1H), 3.85 – 3.75 (m, 2H), 3.69 (dd, *J* = 14.3, 7.5 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 166.7, 132.5, 132.0, 128.6, 126.7, 59.6, 46.0, 43.6.

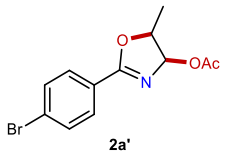


19ao

5-(bromomethyl)-2-(4-bromophenyl)-4,5-dihydrooxazole (19ao):

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.79 (d, *J* = 8.6 Hz, 2H), 7.55 (d, *J* = 8.6 Hz, 2H), 4.97 – 4.89 (m, 1H), 4.17 (dd, *J* = 15.2, 9.7 Hz, 1H), 3.90 (dd, *J* = 15.2, 6.7 Hz, 1H), 3.58 – 3.48 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 162.8, 131.6, 129.7, 126.2, 126.2, 78.0, 59.3, 33.5.

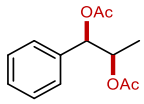


2a'

2-(4-bromophenyl)-5-methyl-4,5-dihydrooxazol-4-yl acetate (2a'):

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 8.5 Hz, 2H), 7.58 (d, *J* = 8.5 Hz, 2H), 6.23 (d, *J* = 3.5 Hz, 1H), 4.71 – 4.62 (m, 1H), 2.12 (s, 3H), 1.49 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.5, 167.8, 131.8, 130.4, 127.3, 125.8, 97.3, 82.2, 21.1, 18.2.



4a'

1-phenylpropane-1,2-diyl diacetate (4a'):

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.37 – 7.33 (m, 4H), 7.31 (q, *J* = 4.1 Hz, 1H), 5.92 (d, *J* = 4.3 Hz, 1H), 5.22 (p, *J* = 6.0 Hz, 1H), 2.14 (s, 3H), 2.01 (s, 3H), 1.18 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 170.3, 169.9, 136.6, 128.3, 128.2, 126.9, 76.1, 71.6, 21.08, 21.05, 14.6.

2.6 References

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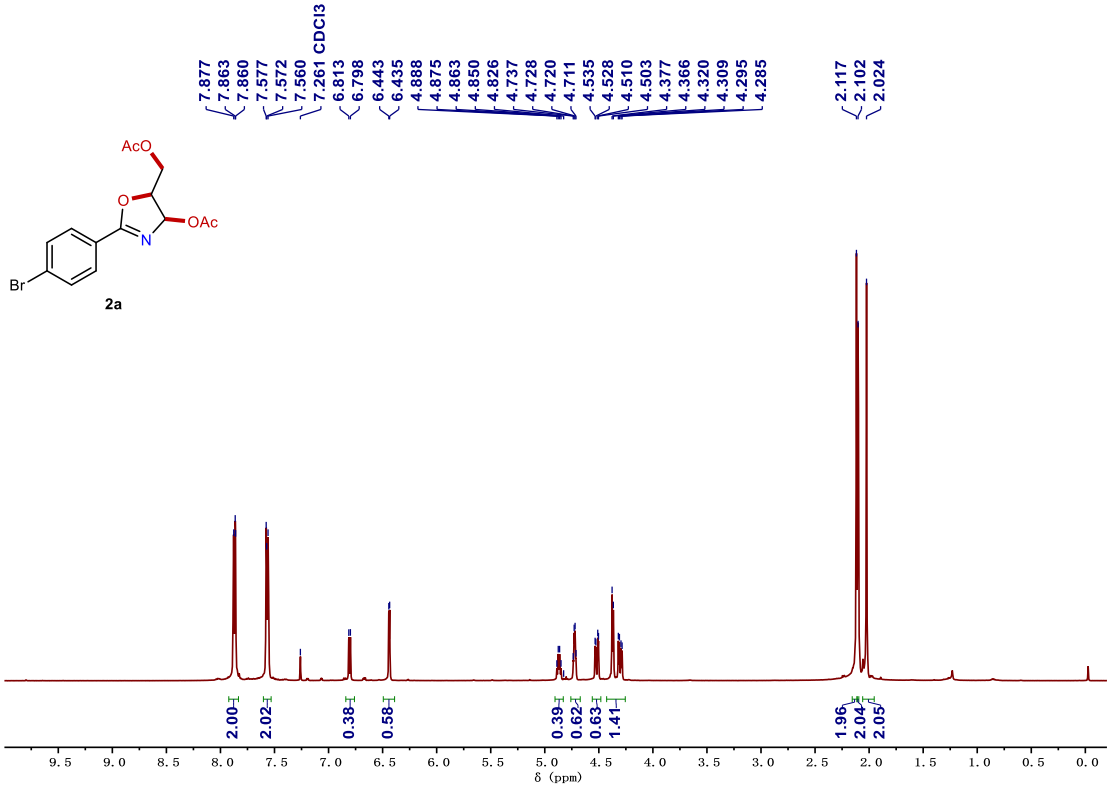
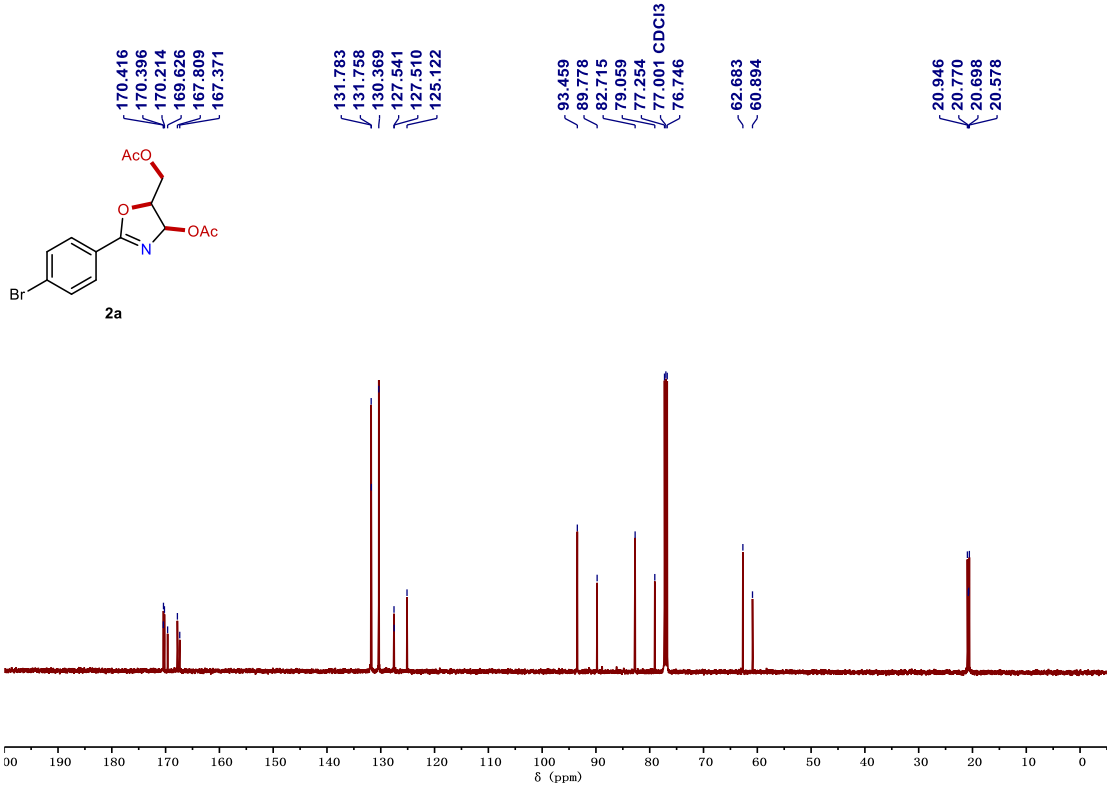
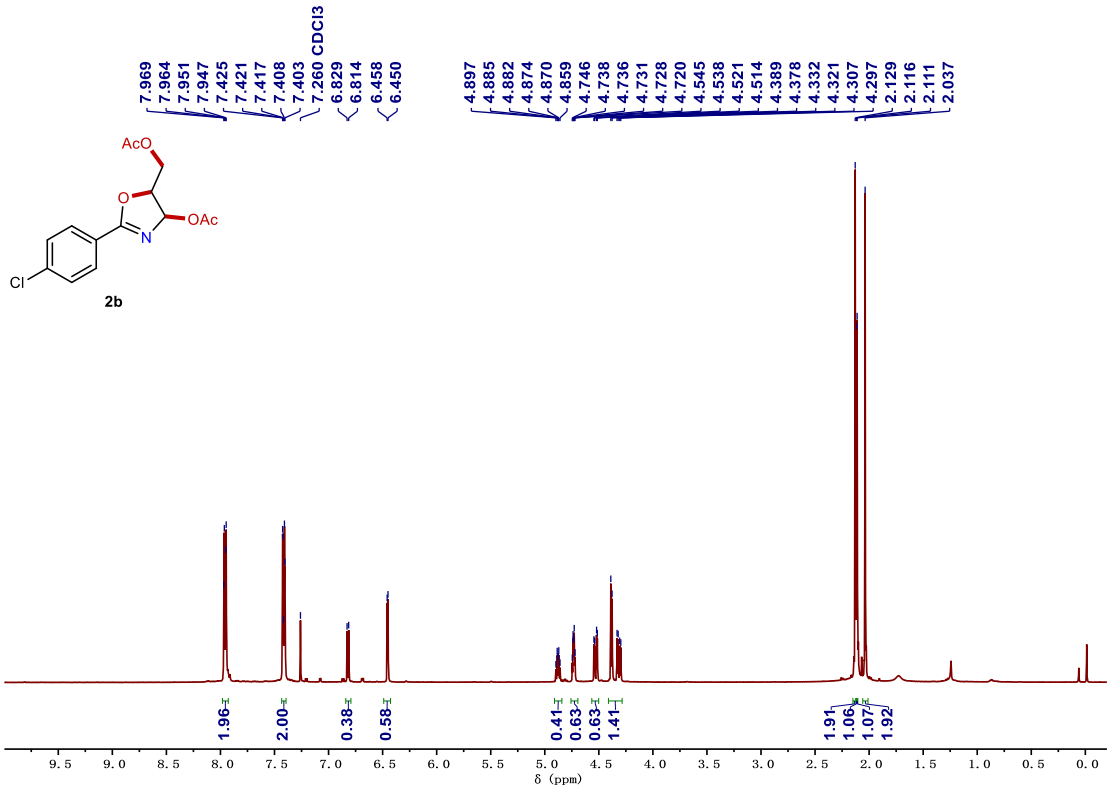
3. NMR Spectra



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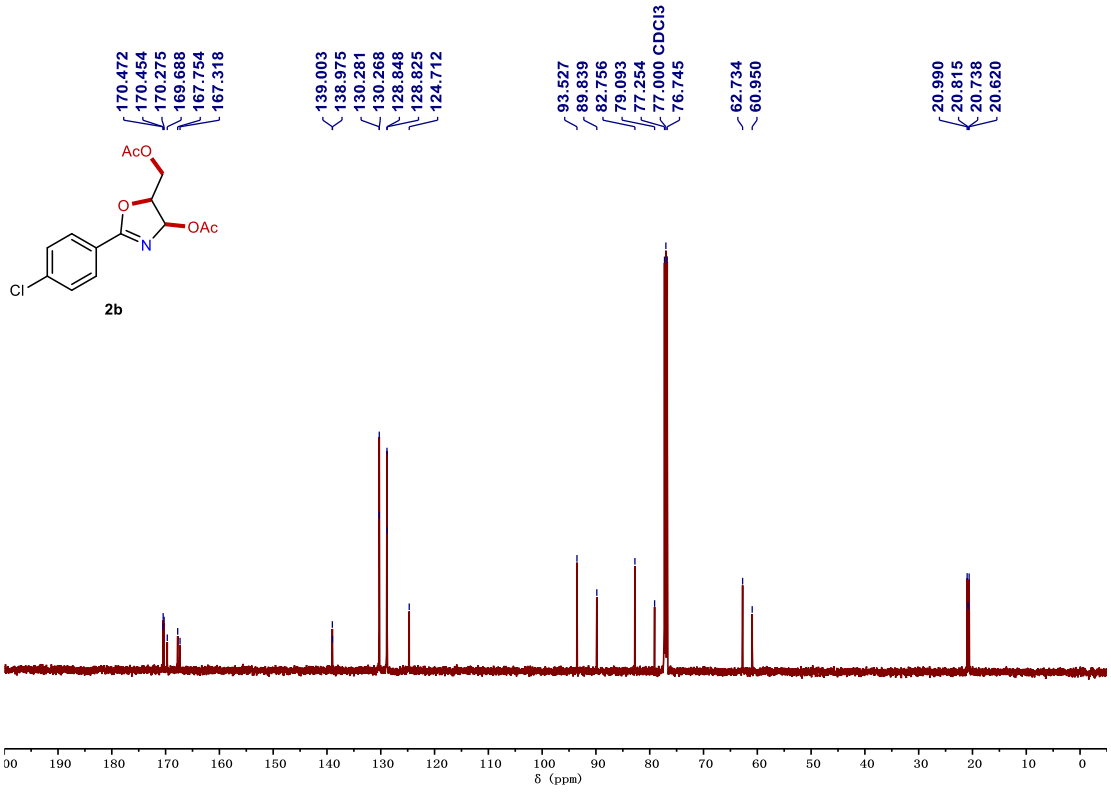
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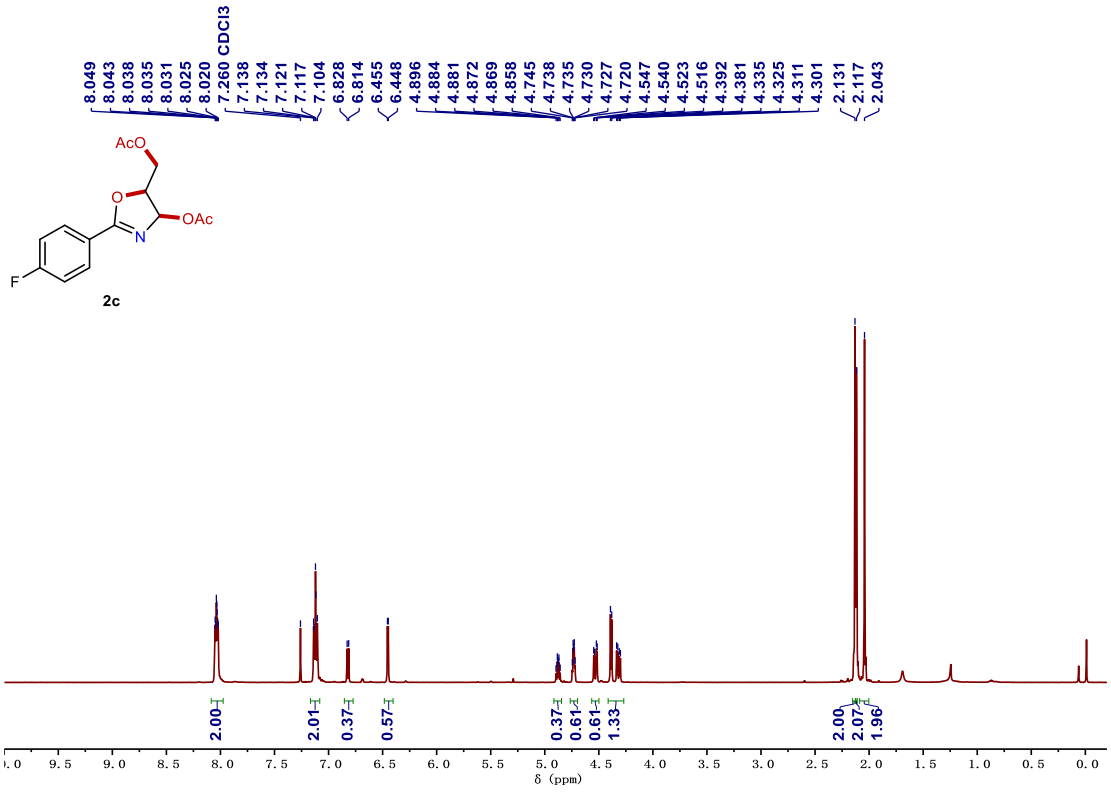
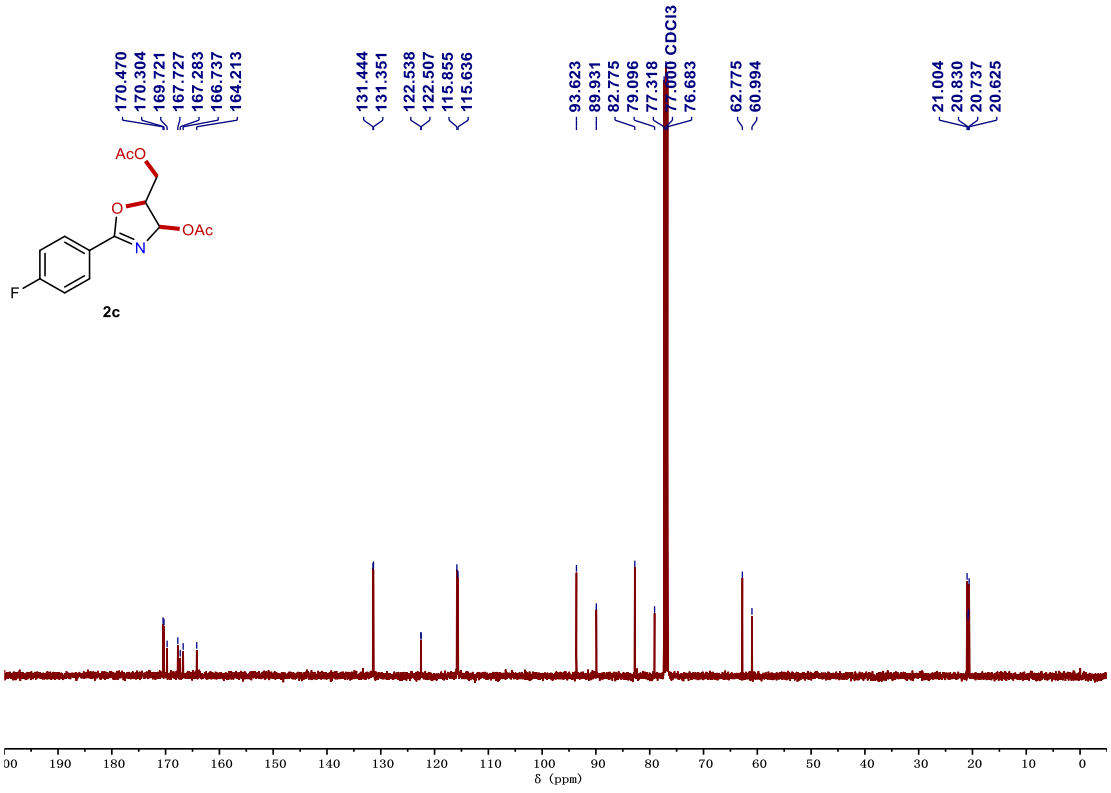
60.950
20.990
20.815
20.738
20.620
AcO
O
OAc
N
Cl
2b
δ (ppm)


8.049
8.043
8.038
8.035
8.031
8.025
8.020
7.260 CDCl3
7.138
7.134
7.121
7.117
7.104
6.828
6.814
6.455
6.448
4.896
4.884
4.881
4.872
4.869
4.858
4.745
4.738
4.735
4.730
4.727
4.720
4.547
4.540
4.523
4.516
4.392
4.381
4.335
4.325
4.311
4.301
2.131
2.117
2.043
AcO
O
OAc
N
F
2c
2.00
2.01
0.37
0.57
0.37
0.61
0.61
1.33
2.00
2.07
1.96
δ (ppm)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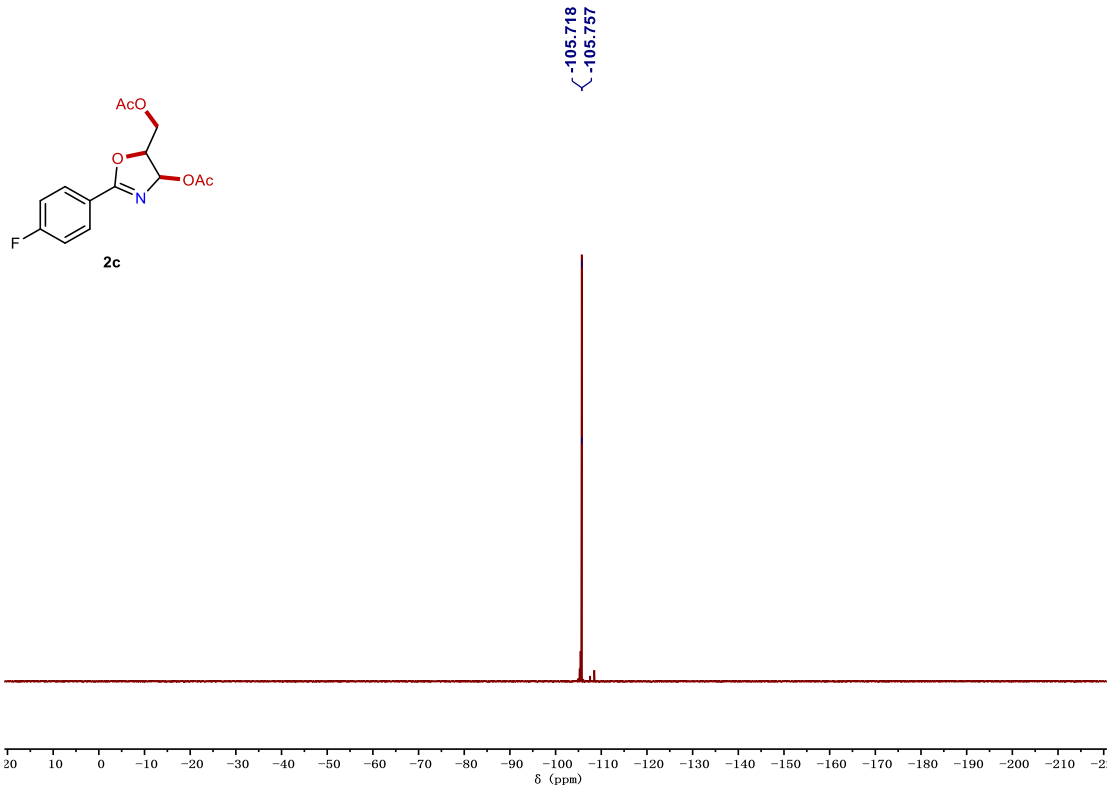
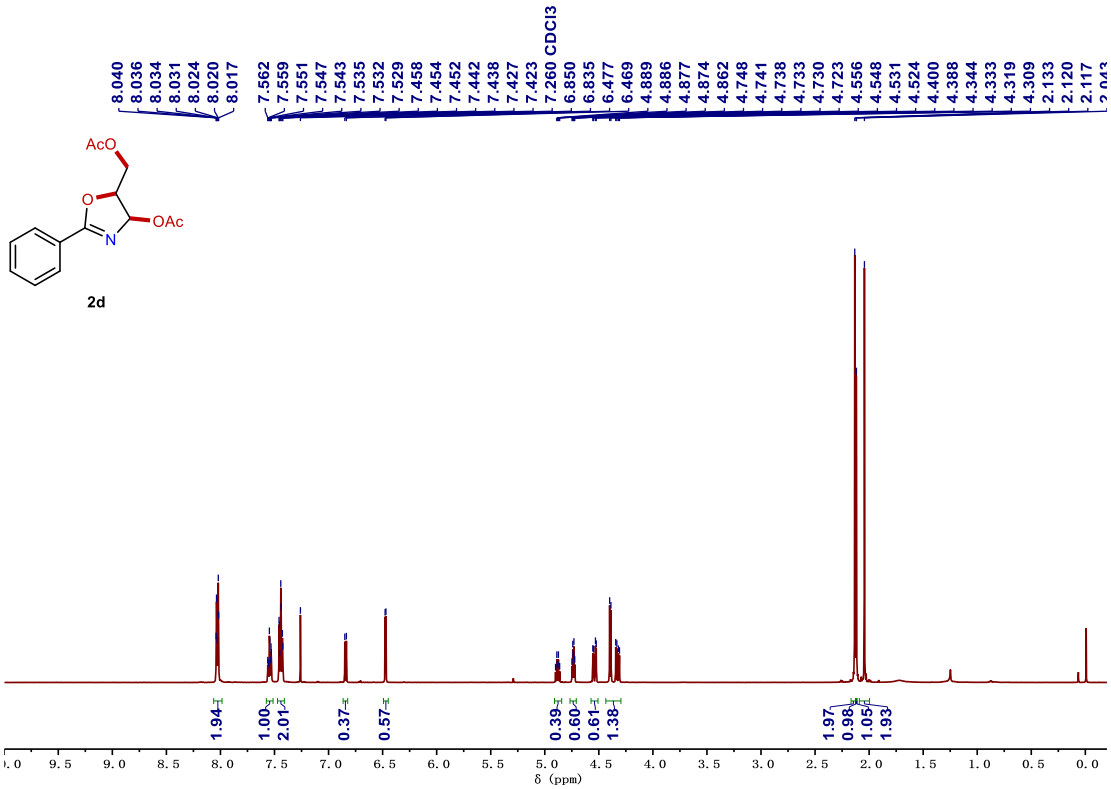
170.304
169.721
167.727
167.283
166.737
164.213
131.444
131.351
122.538
122.507
115.855
115.636
93.623
89.931
82.775
79.096
77.318
77.000 CDCl3
76.683
62.775
60.994
21.004
20.830
20.737
20.625
AcO
O
OAc
N
F
2c
δ (ppm)


-105.718
-105.757
AcO
O
OAc
N
F
2c
20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
δ (ppm)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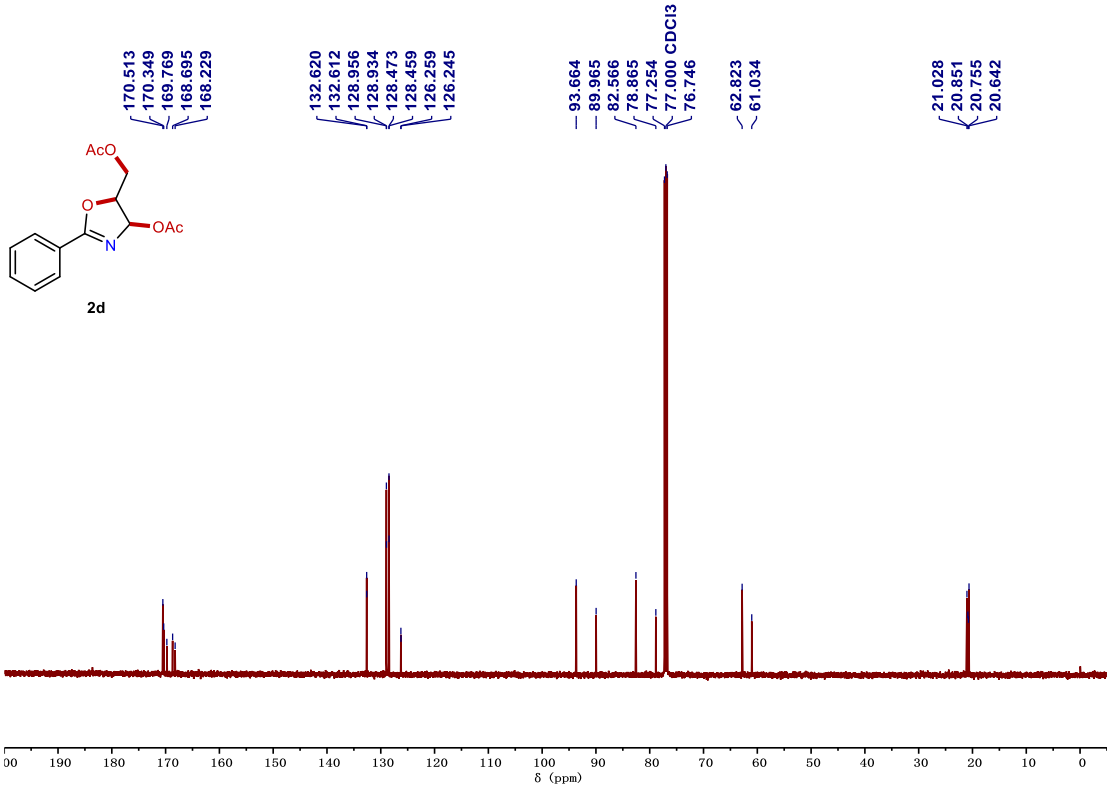
8.036
8.034
8.031
8.024
8.020
8.017
7.562
7.559
7.551
7.547
7.543
7.535
7.532
7.529
7.458
7.454
7.452
7.442
7.438
7.427
7.423
7.260 CDCl3
6.850
6.835
6.477
6.469
4.889
4.886
4.877
4.874
4.862
4.748
4.741
4.738
4.733
4.730
4.723
4.556
4.548
4.531
4.524
4.400
4.388
4.344
4.333
4.319
4.309
2.133
2.120
2.117
AcO
O
OAc
N
2d
1.94
1.00
2.01
0.37
0.57
0.39
0.60
0.61
1.38
1.97
0.98
1.05
1.93
δ (ppm)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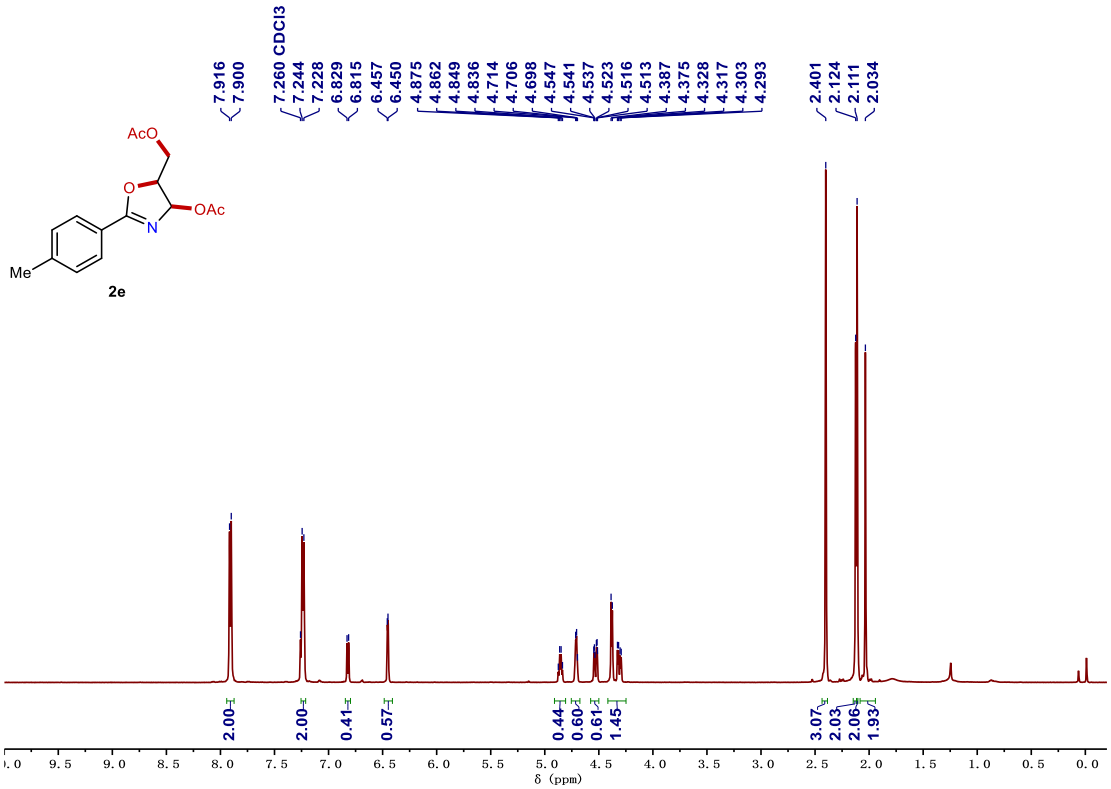
170.349
169.769
168.695
168.229
132.620
132.612
128.956
128.934
128.473
128.459
126.259
126.245
93.664
89.965
82.566
78.865
77.254
77.000 CDCl3
76.746
62.823
61.034
21.028
20.851
20.755
20.642
AcO
O
OAc
N
2d
δ (ppm)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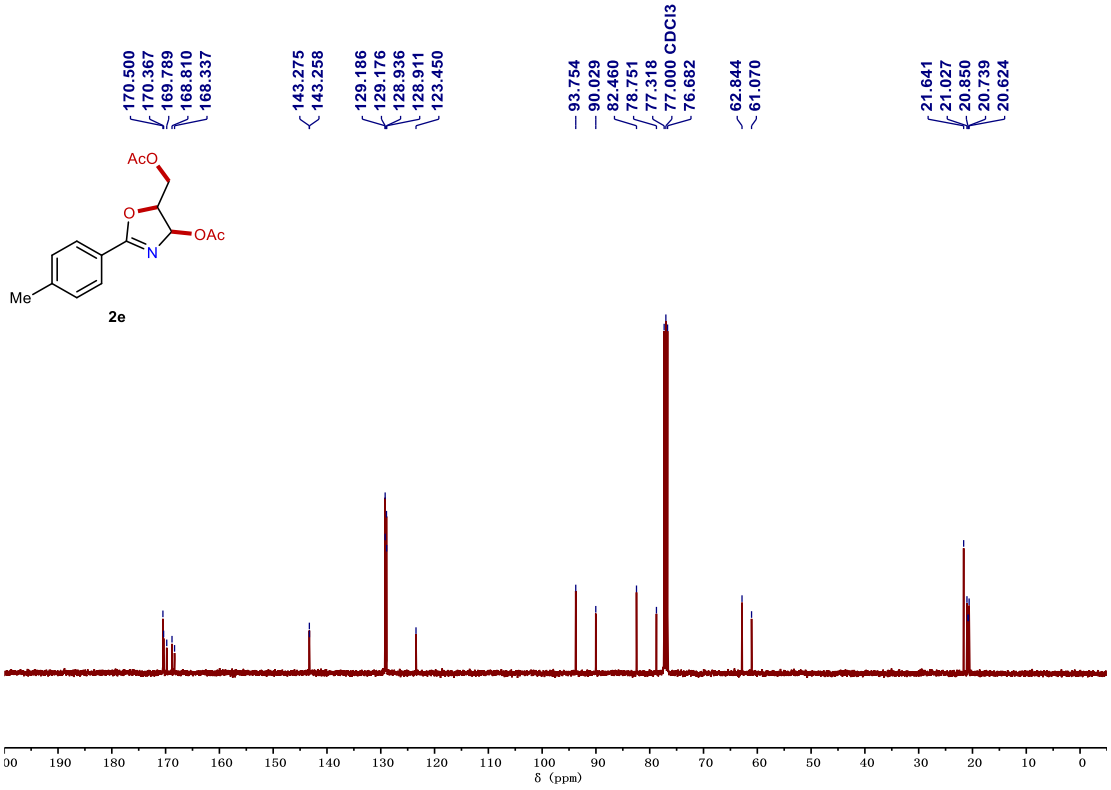
7.900
7.260 CDCl3
7.244
7.228
6.829
6.815
6.457
6.450
4.875
4.862
4.849
4.836
4.714
4.706
4.698
4.547
4.541
4.537
4.523
4.516
4.513
4.387
4.375
4.328
4.317
4.303
4.293
2.401
2.124
2.111
2.034
AcO
O
OAc
N
Me
2e
2.00
2.00
0.41
0.57
0.44
0.60
0.61
1.45
3.07
2.03
2.06
1.93
δ (ppm)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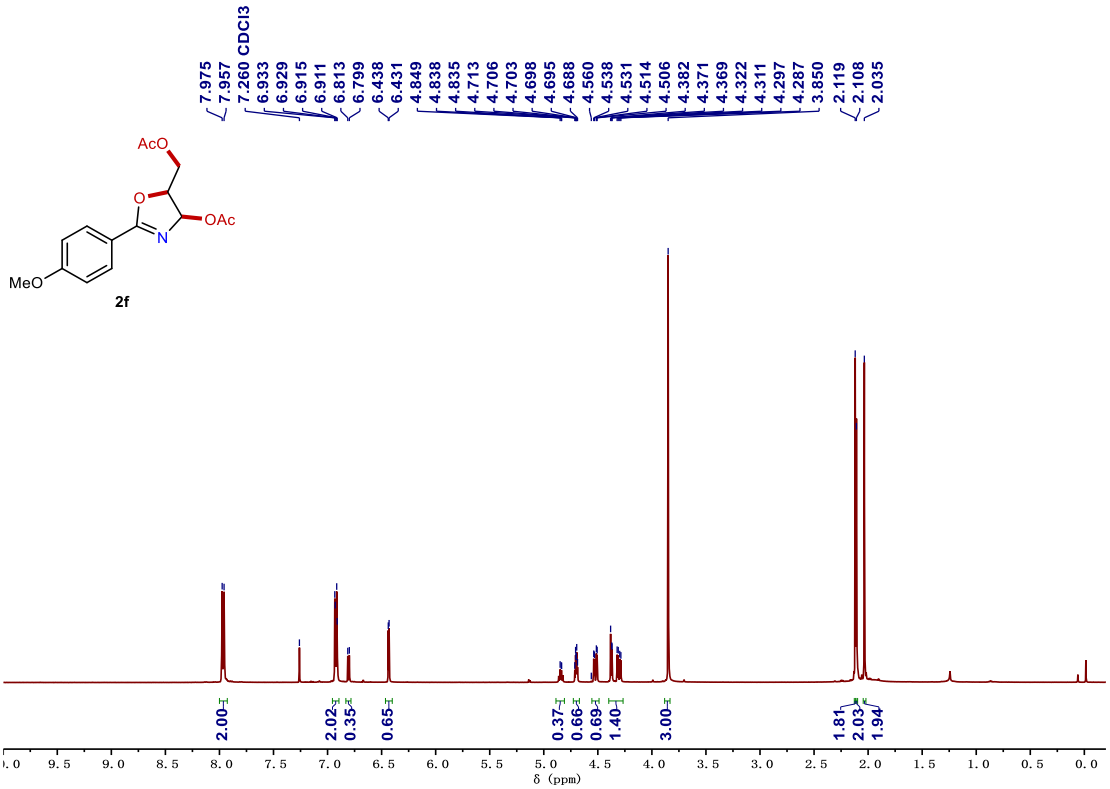
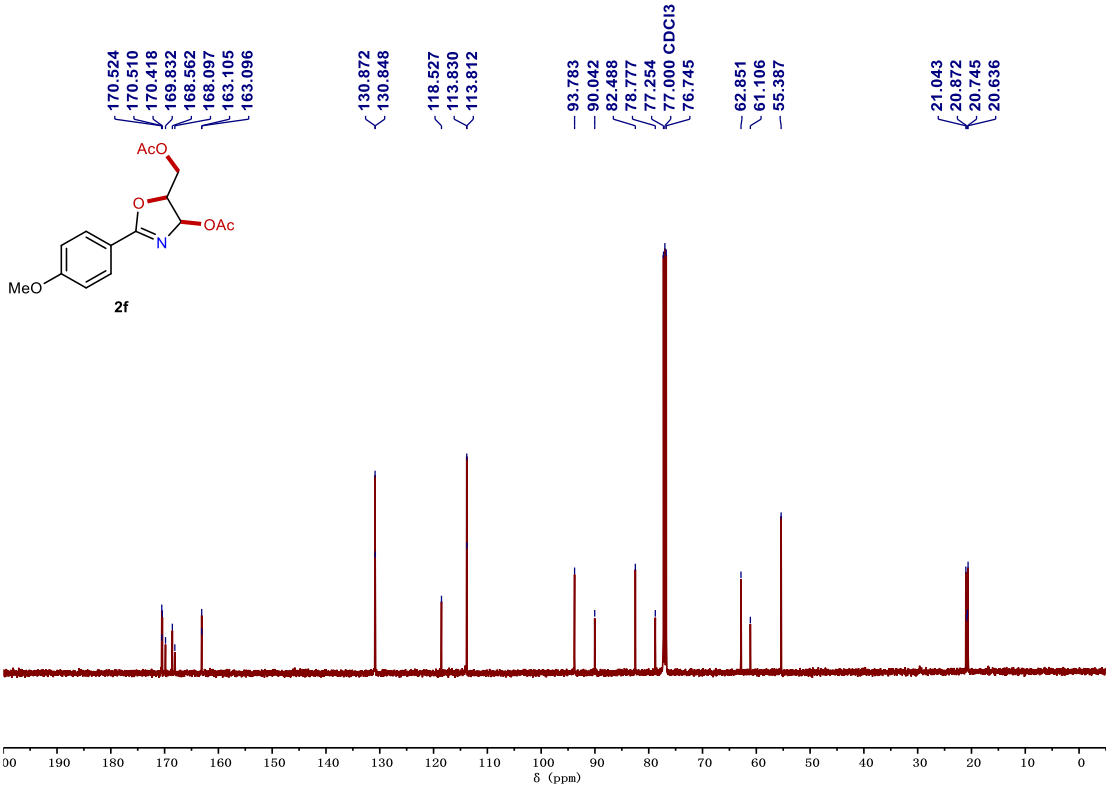
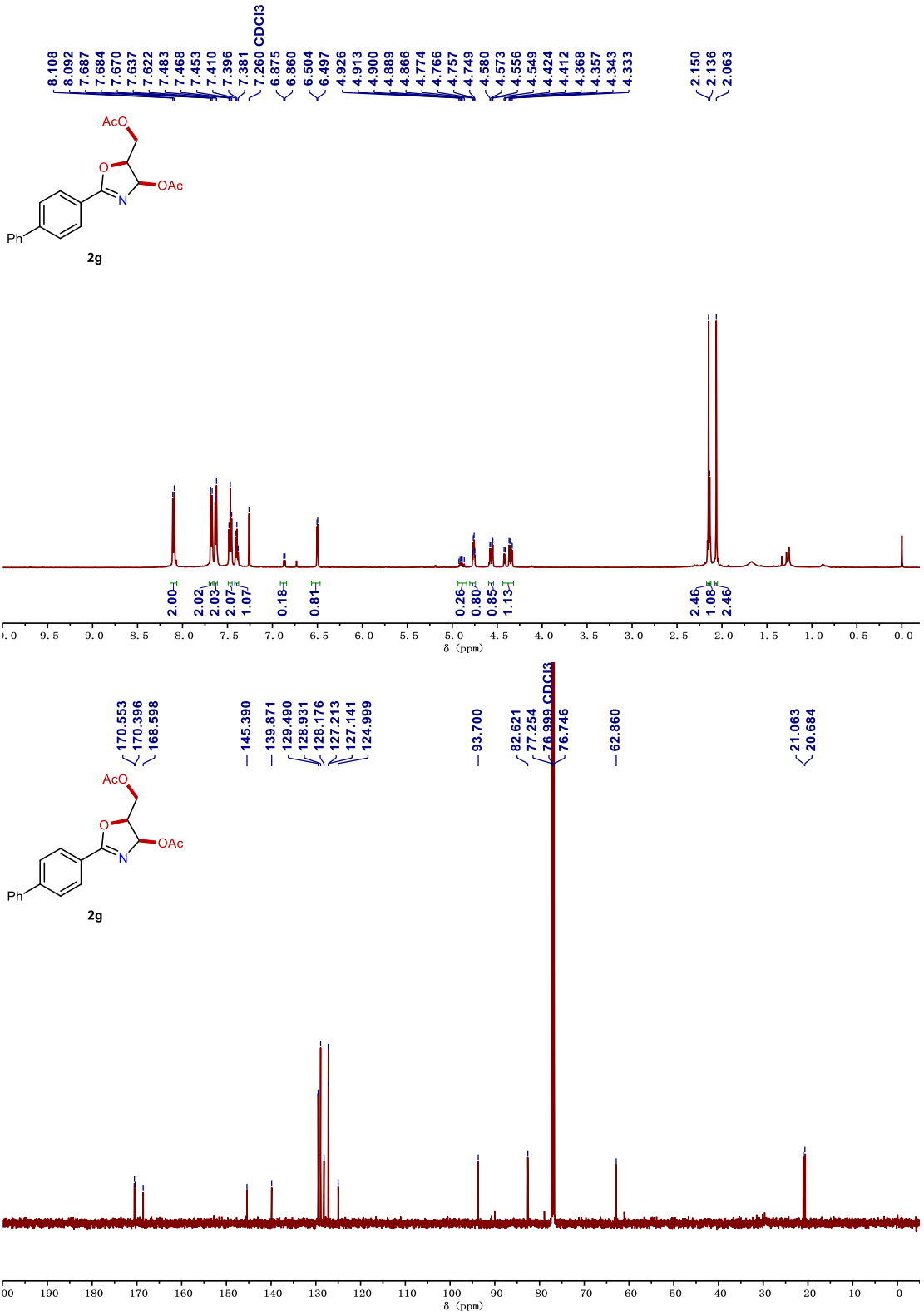
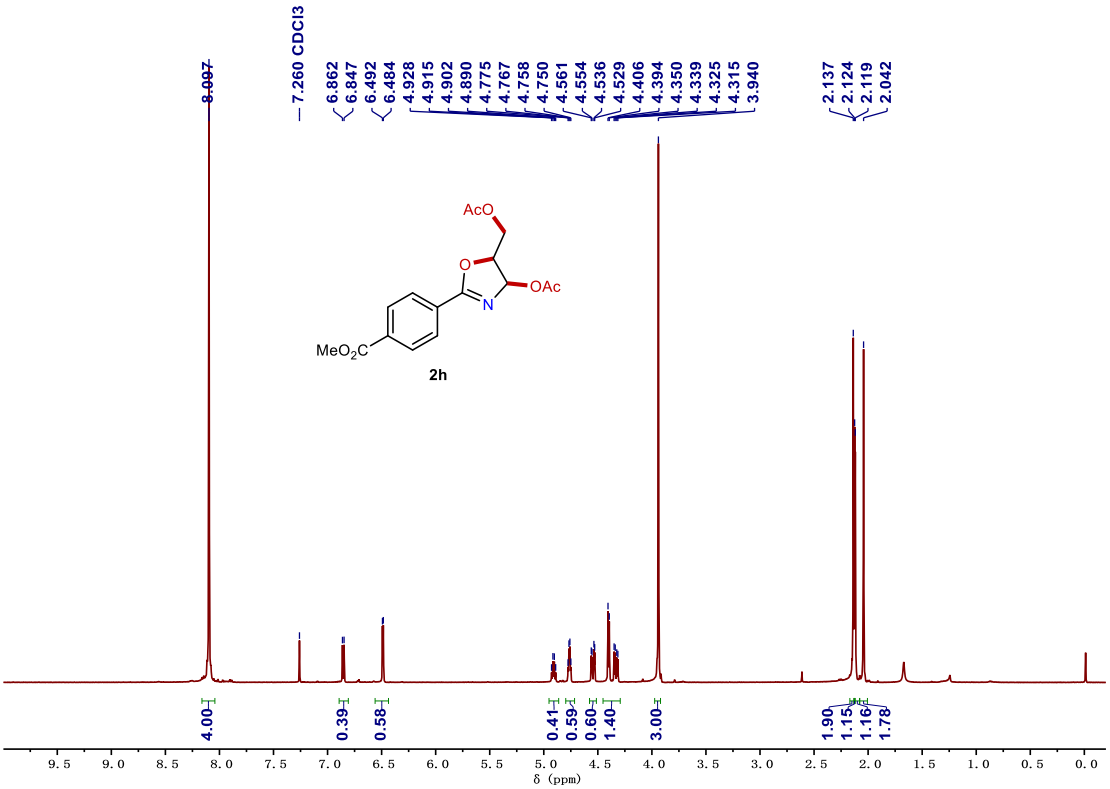
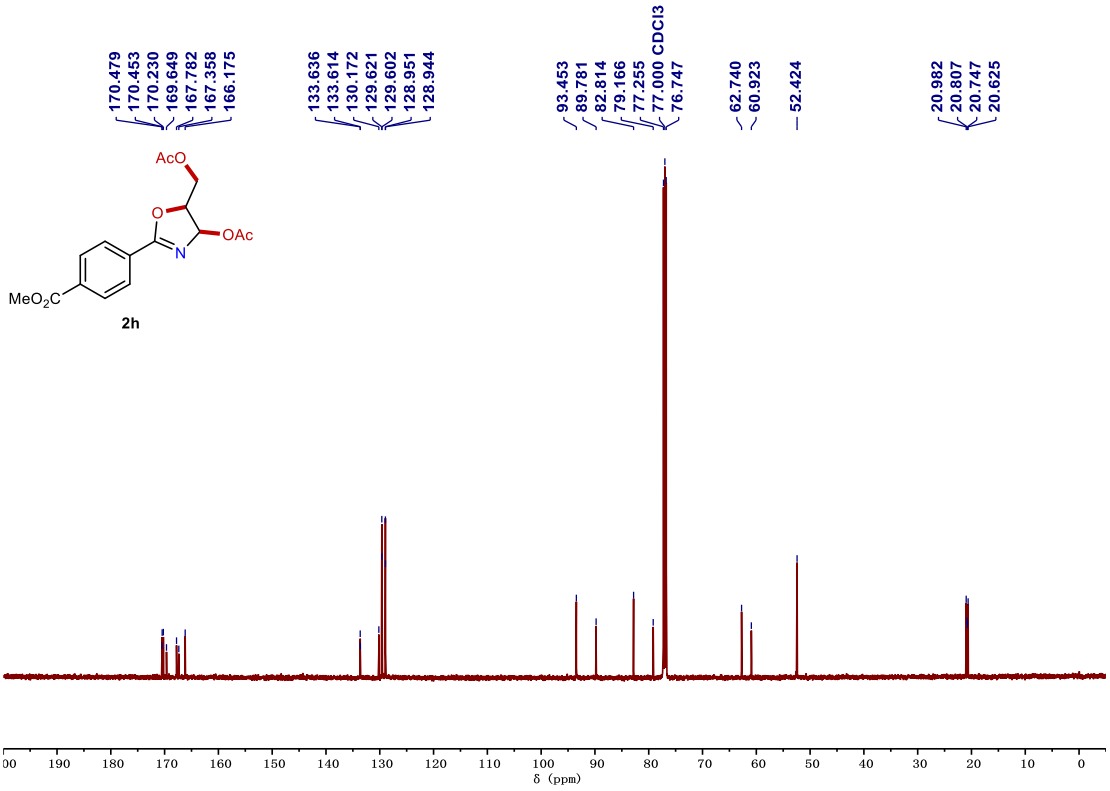
170.367
169.789
168.810
168.337
143.275
143.258
129.186
129.176
128.936
128.911
123.450
93.754
90.029
82.460
78.751
77.318
77.000 CDCl3
76.682
62.844
61.070
21.641
21.027
20.850
20.739
20.624
AcO
O
OAc
N
Me
2e
δ (ppm)


7.975
7.957
7.260 CDCl3
6.933
6.929
6.915
6.911
6.813
6.799
6.438
6.431
4.849
4.838
4.835
4.713
4.706
4.703
4.698
4.695
4.688
4.560
4.538
4.531
4.514
4.506
4.382
4.371
4.369
4.322
4.311
4.297
4.287
3.850
2.119
2.108
2.035
AcO
O
OAc
N
MeO
2f
2.00
2.02
0.35
0.65
0.37
0.66
0.69
1.40
3.00
1.81
2.03
1.94
δ (ppm)


170.524
170.510
170.418
169.832
168.562
168.097
163.105
163.096
130.872
130.848
118.527
113.830
113.812
93.783
90.042
82.488
78.777
77.254
77.000 CDCl3
76.745
62.851
61.106
55.387
21.043
20.872
20.745
20.636
AcO
O
OAc
N
MeO
2f
δ (ppm)


8.108
8.092
7.687
7.684
7.670
7.637
7.622
7.483
7.468
7.453
7.410
7.396
7.381
7.260 CDCl3
6.875
6.860
6.504
6.497
4.926
4.913
4.900
4.889
4.866
4.774
4.766
4.757
4.749
4.580
4.573
4.556
4.549
4.424
4.412
4.368
4.357
4.343
4.333
2.150
2.136
2.063
AcO
O
OAc
N
Ph
2g
2.00
2.02
2.03
2.07
1.07
0.18
0.81
0.26
0.80
0.85
1.13
2.46
1.08
2.46
δ (ppm)
170.553
170.396
168.598
145.390
139.871
129.490
128.931
128.176
127.213
127.141
124.999
93.700
82.621
77.254
76.999 CDCl3
76.746
62.860
21.063
20.684
δ (ppm)


8.097
7.260 CDCl3
6.862
6.847
6.492
6.484
4.928
4.915
4.902
4.890
4.775
4.767
4.758
4.750
4.561
4.554
4.536
4.529
4.406
4.394
4.350
4.339
4.325
4.315
3.940
2.137
2.124
2.119
2.042
AcO
O
OAc
N
MeO2C
2h
4.00
0.39
0.58
0.41
0.59
0.60
1.40
3.00
1.90
1.15
1.16
1.78
δ (ppm)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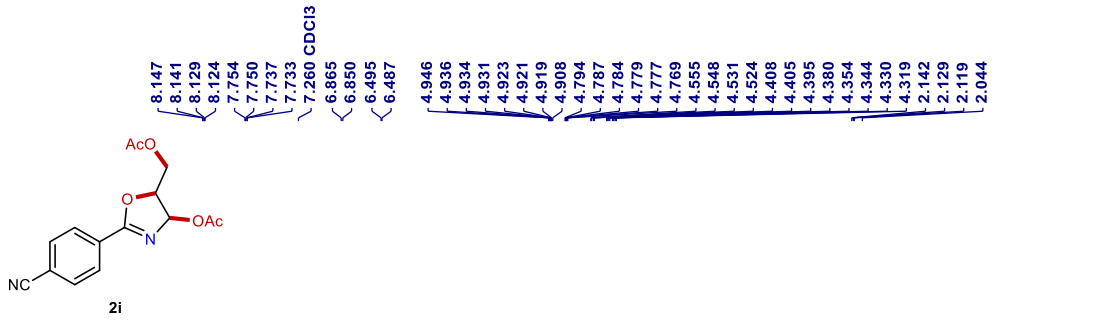
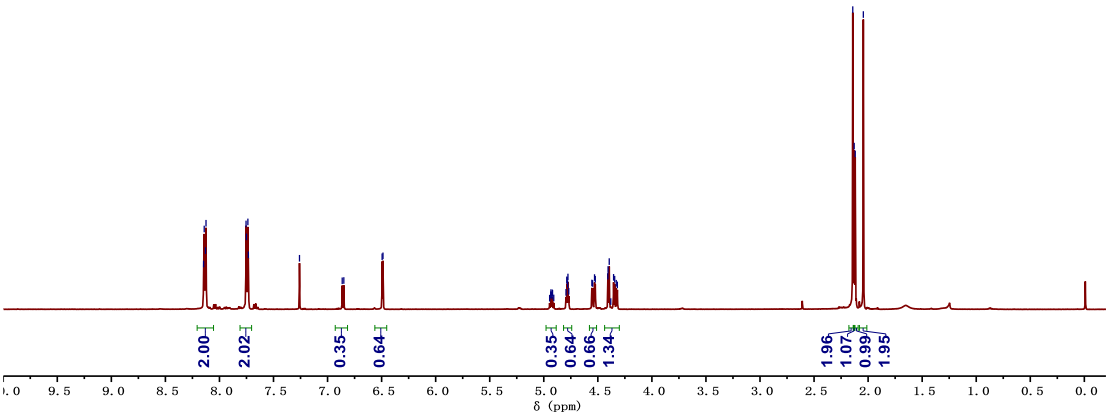
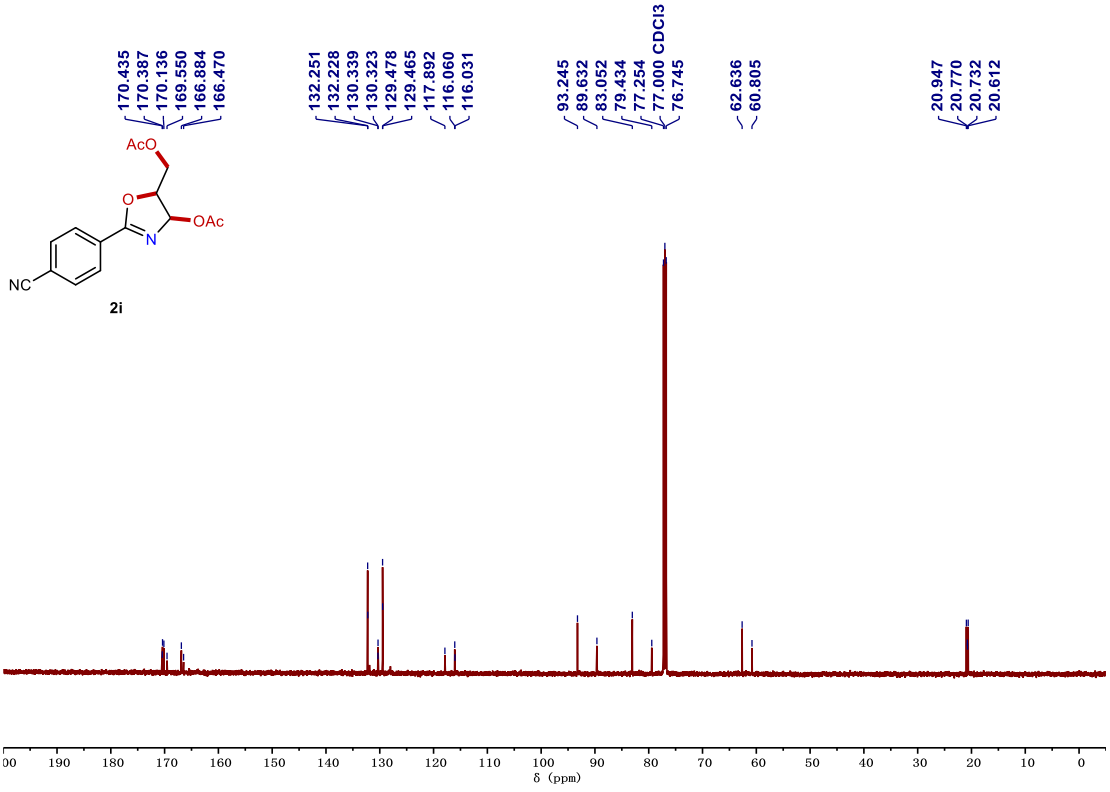
170.453
170.230
169.649
167.782
167.358
166.175
133.636
133.614
130.172
129.621
129.602
128.951
128.944
93.453
89.781
82.814
79.166
77.255
77.000 CDCl3
76.747
62.740
60.923
52.424
20.982
20.807
20.747
20.625
AcO
O
OAc
N
MeO2C
2h
δ (ppm)

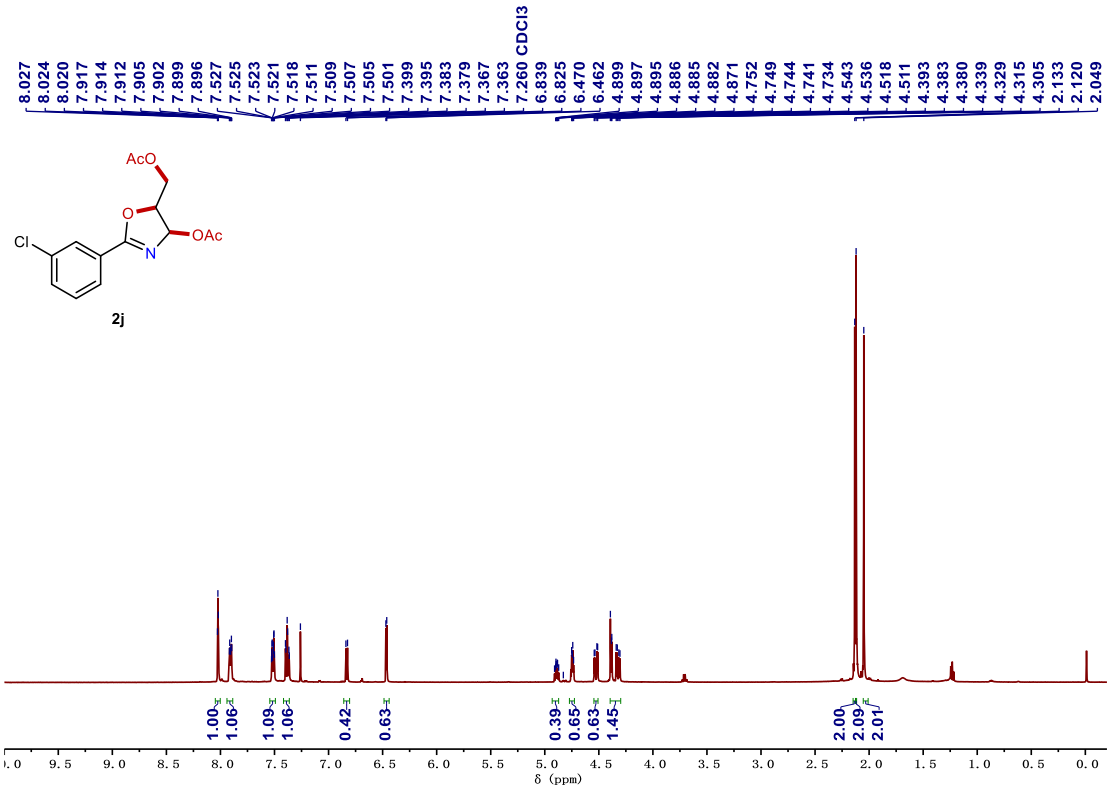

8.147
8.141
8.129
8.124
7.754
7.750
7.737
7.733
7.260 CDCl3
6.865
6.850
6.495
6.487
4.946
4.936
4.934
4.931
4.923
4.921
4.919
4.908
4.794
4.787
4.784
4.779
4.777
4.769
4.555
4.548
4.531
4.524
4.408
4.405
4.395
4.380
4.354
4.344
4.330
4.319
2.142
2.129
2.119
2.044
AcO
O
OAc
N
NC

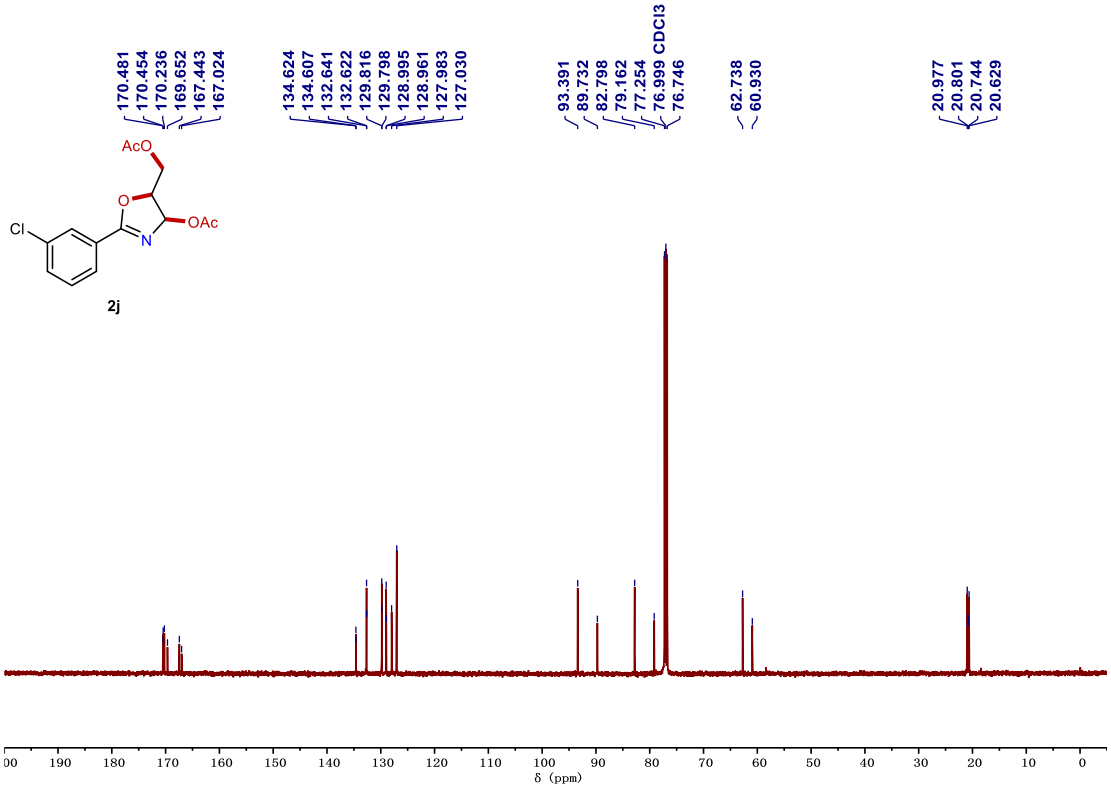
2i


2.00
2.02
0.35
0.64
0.35
0.64
0.66
1.34
1.96
1.07
0.99
1.95
δ (ppm)


170.435
170.387
170.136
169.550
166.884
166.470
132.251
132.228
130.339
130.323
129.478
129.465
117.892
116.060
116.031
93.245
89.632
83.052
79.434
77.254
77.000 CDCl3
76.745
62.636
60.805
20.947
20.770
20.732
20.612
AcO
O
OAc
N
NC
δ (ppm)

2i


8.027
8.024
8.020
7.917
7.914
7.912
7.905
7.902
7.899
7.896
7.527
7.525
7.523
7.521
7.518
7.511
7.509
7.507
7.505
7.501
7.399
7.395
7.383
7.379
7.367
7.363
7.260 CDCl3
6.839
6.825
6.470
6.462
4.899
4.897
4.895
4.886
4.885
4.882
4.871
4.752
4.749
4.744
4.741
4.734
4.543
4.536
4.518
4.511
4.393
4.383
4.380
4.339
4.329
4.315
4.305
2.133
2.120
2.049
AcO
O
OAc
N
Cl
2j
1.00
1.06
1.09
1.06
0.42
0.63
0.39
0.65
0.63
1.45
2.00
2.09
2.01
δ (ppm)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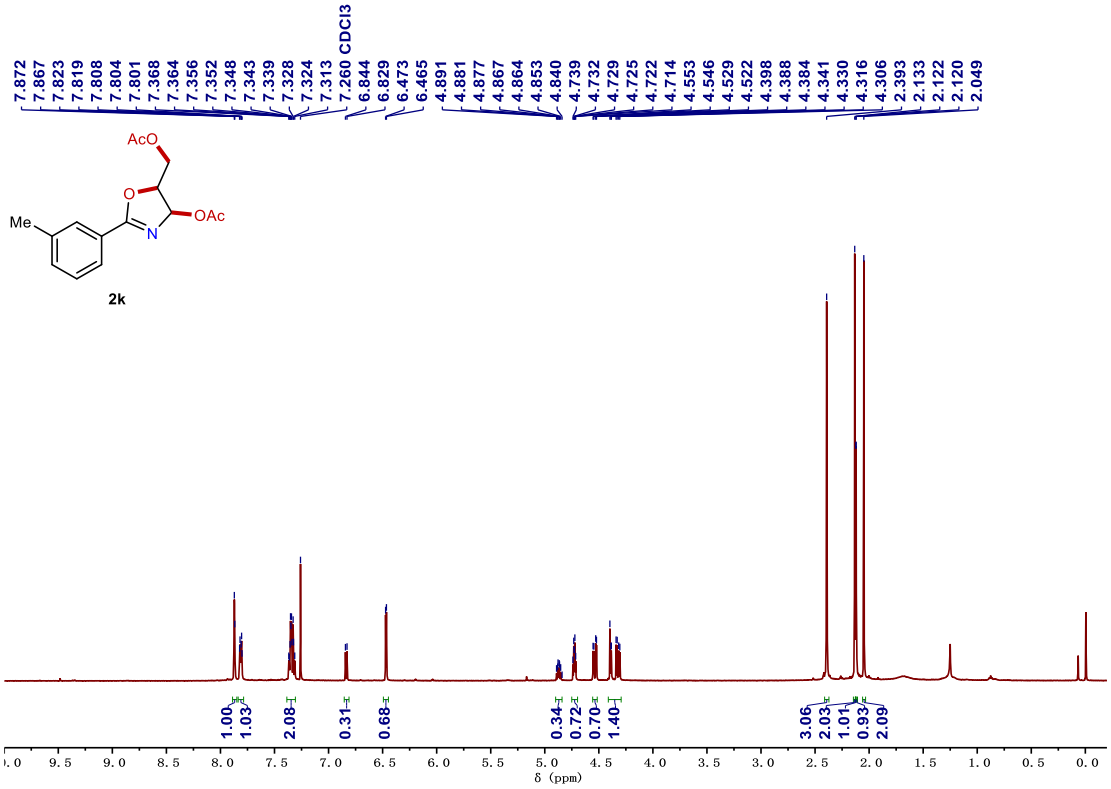
170.454
170.236
169.652
167.443
167.024
134.624
134.607
132.641
132.622
129.816
129.798
128.995
128.961
127.983
127.030
93.391
89.732
82.798
79.162
77.254
76.999 CDCl3
76.746
62.738
60.930
20.977
20.801
20.744
20.629
AcO
O
OAc
N
Cl
2j
δ (ppm)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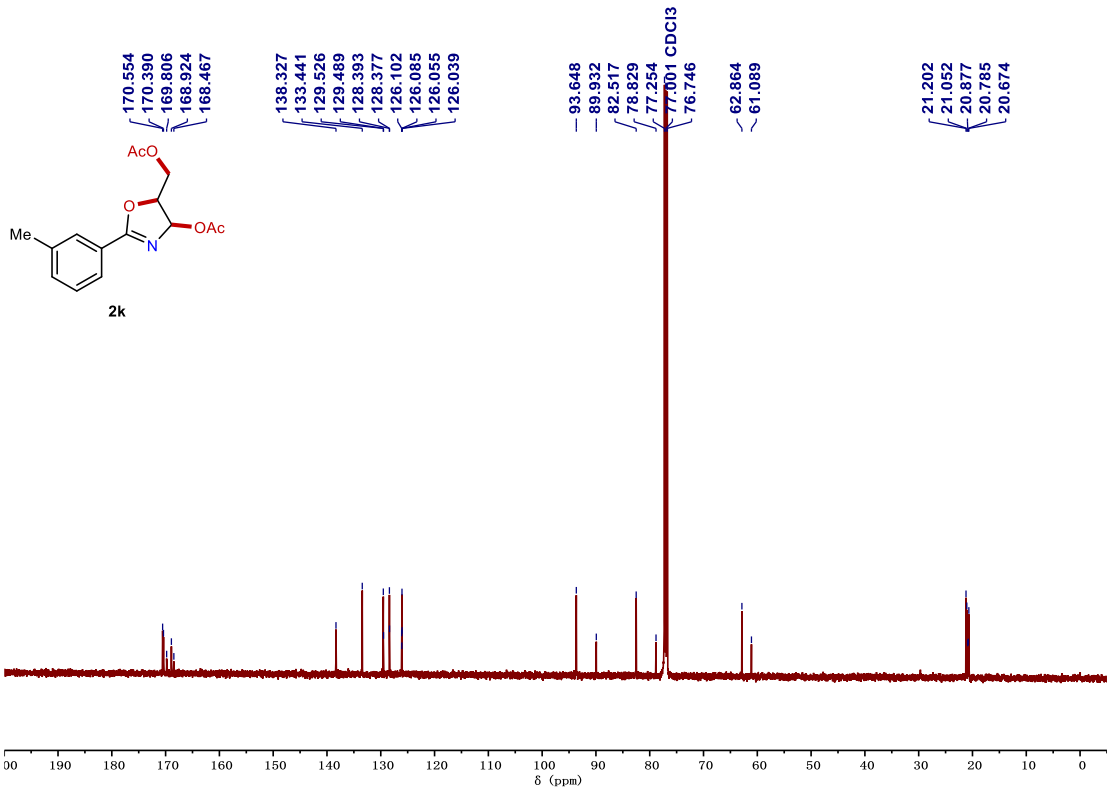
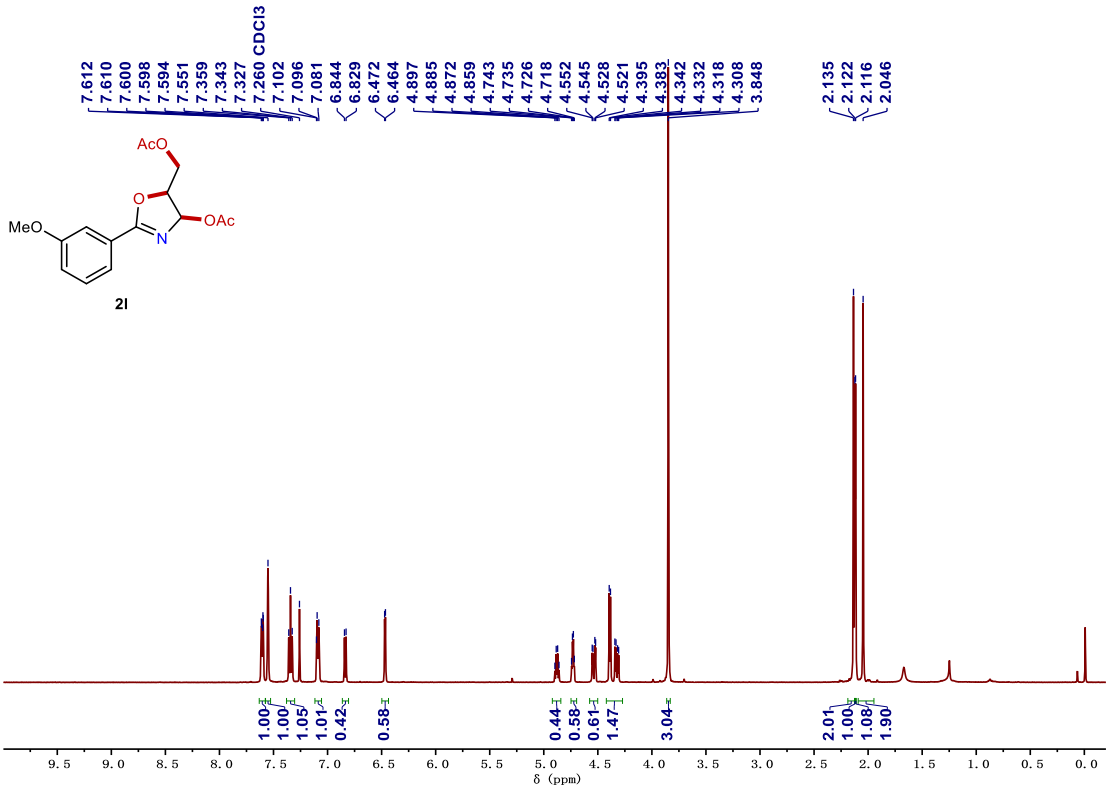
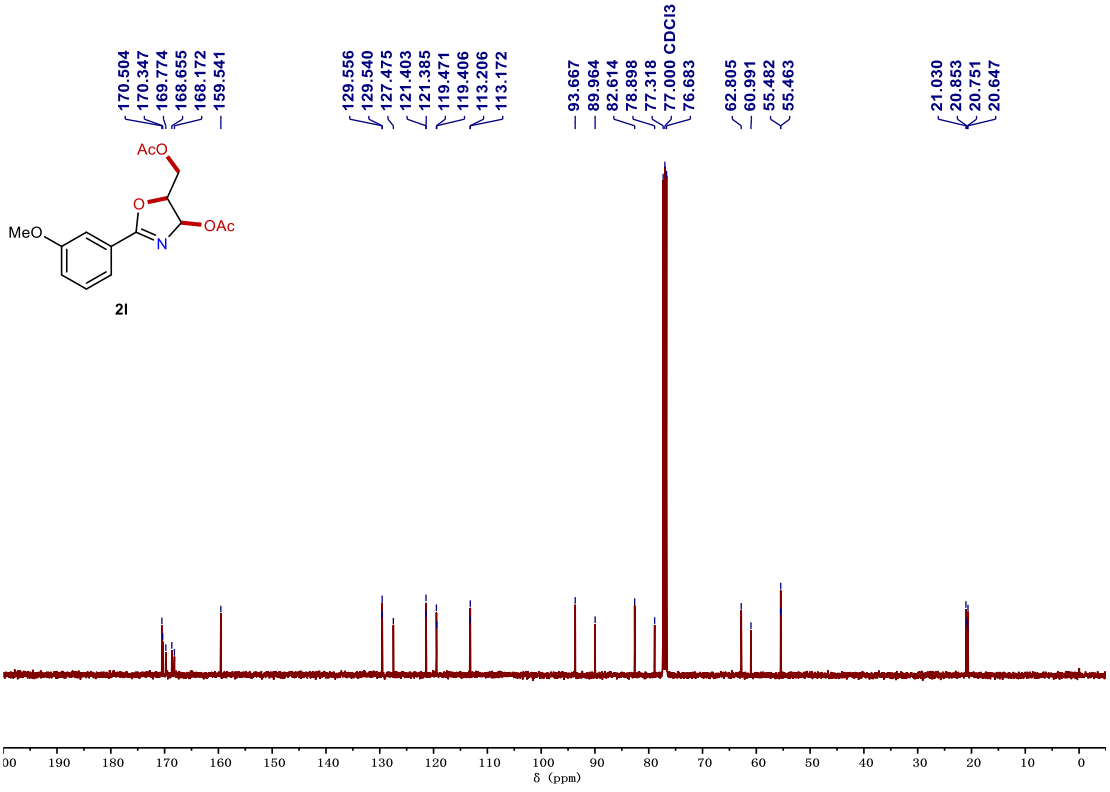
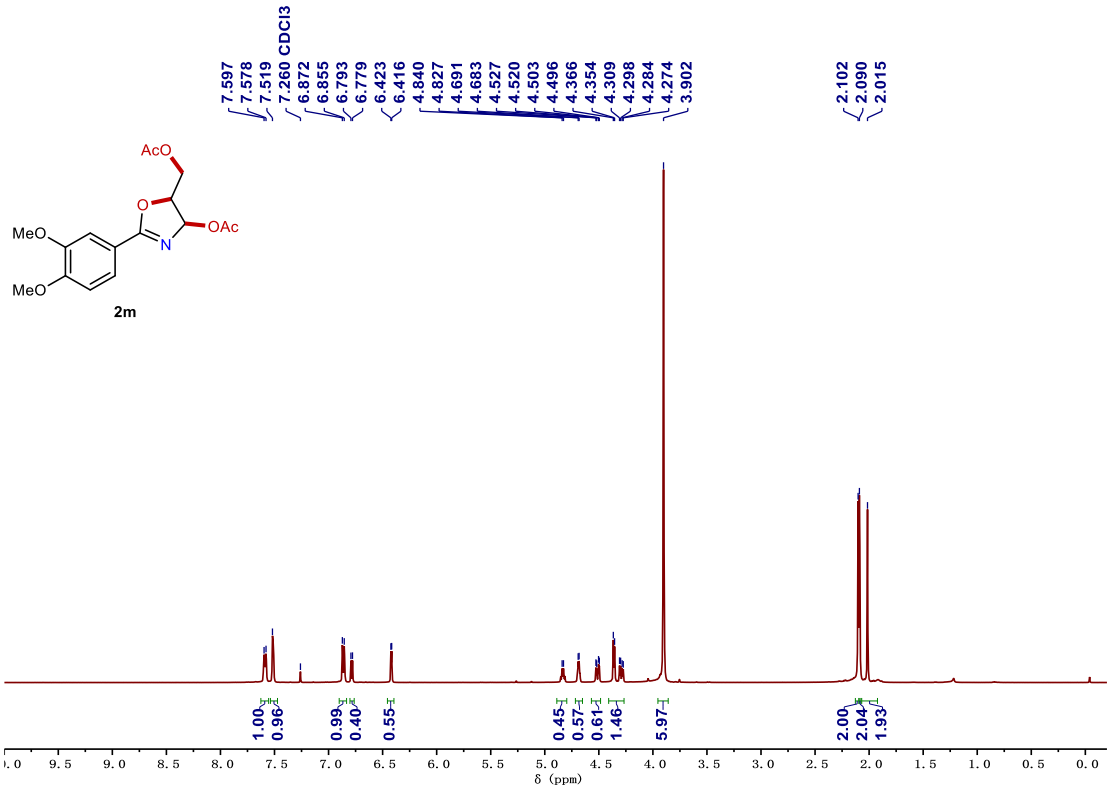
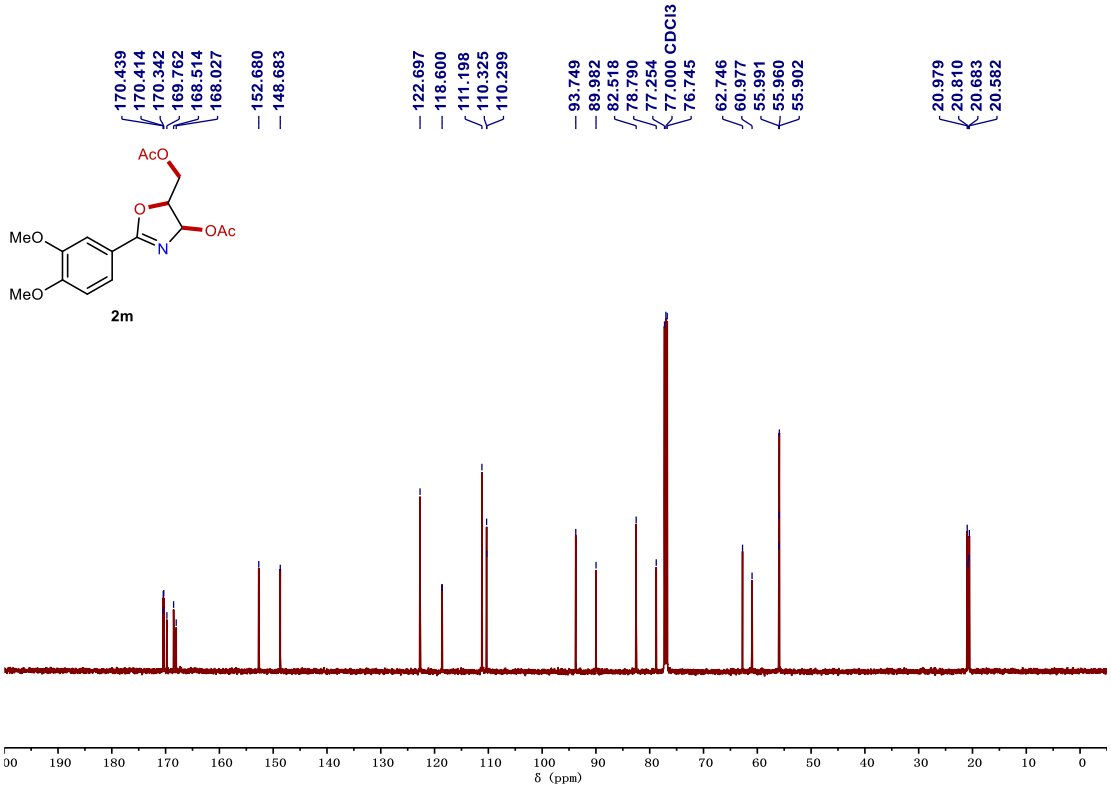
7.867
7.823
7.819
7.808
7.804
7.801
7.368
7.364
7.356
7.352
7.348
7.343
7.339
7.328
7.324
7.313
7.260 CDCl3
6.844
6.829
6.473
6.465
4.891
4.881
4.877
4.867
4.864
4.853
4.840
4.739
4.732
4.729
4.725
4.722
4.714
4.553
4.546
4.529
4.522
4.398
4.388
4.384
4.341
4.330
4.316
4.306
2.393
2.133
2.122
2.120
2.049
AcO
O
N
OAc
Me
2k
1.00
1.03
2.08
0.31
0.68
0.34
0.72
0.70
1.40
3.06
2.03
1.01
0.93
2.09
δ (ppm)


170.554
170.390
169.806
168.924
168.467
138.327
133.441
129.526
129.489
128.393
128.377
126.102
126.085
126.055
126.039
93.648
89.932
82.517
78.829
77.254
77.001 CDCl3
76.746
62.864
61.089
21.202
21.052
20.877
20.785
20.674
AcO
O
N
OAc
Me
2k
δ (ppm)


7.612
7.610
7.600
7.598
7.594
7.551
7.359
7.343
7.327
7.260 CDCl3
7.102
7.096
7.081
6.844
6.829
6.472
6.464
4.897
4.885
4.872
4.859
4.743
4.735
4.726
4.718
4.552
4.545
4.528
4.521
4.395
4.383
4.342
4.332
4.318
4.308
3.848
2.135
2.122
2.116
2.046
AcO
O
OAc
MeO
N
2l
1.00
1.00
1.05
1.01
0.42
0.58
0.44
0.58
0.61
1.47
3.04
2.01
1.00
1.08
1.90
δ (ppm)


170.504
170.347
169.774
168.655
168.172
159.541
129.556
129.540
127.475
121.403
121.385
119.471
119.406
113.206
113.172
93.667
89.964
82.614
78.898
77.318
77.000 CDCl3
76.683
62.805
60.991
55.482
55.463
21.030
20.853
20.751
20.647
AcO
O
OAc
MeO
N
2l
δ (ppm)


7.597
7.578
7.519
7.260 CDCl3
6.872
6.855
6.793
6.779
6.423
6.416
4.840
4.827
4.691
4.683
4.527
4.520
4.503
4.496
4.366
4.354
4.309
4.298
4.284
4.274
3.902
2.102
2.090
2.015
AcO
O
OAc
N
MeO
MeO
2m
1.00
0.96
0.99
0.40
0.55
0.45
0.57
0.61
1.46
5.97
2.00
2.04
1.93
δ (ppm)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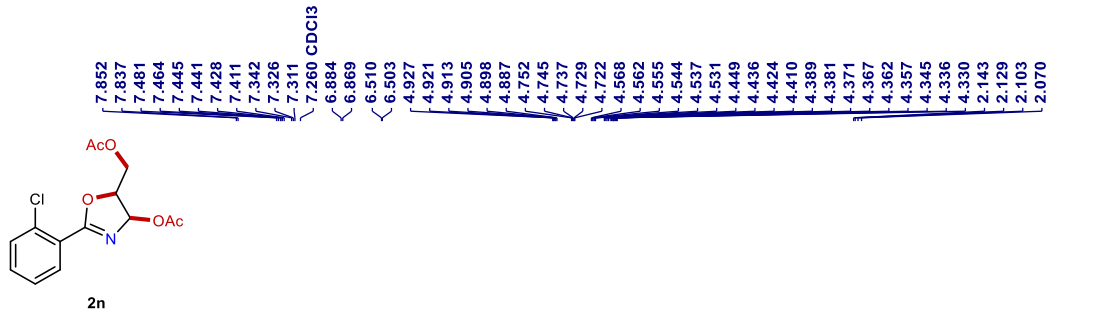
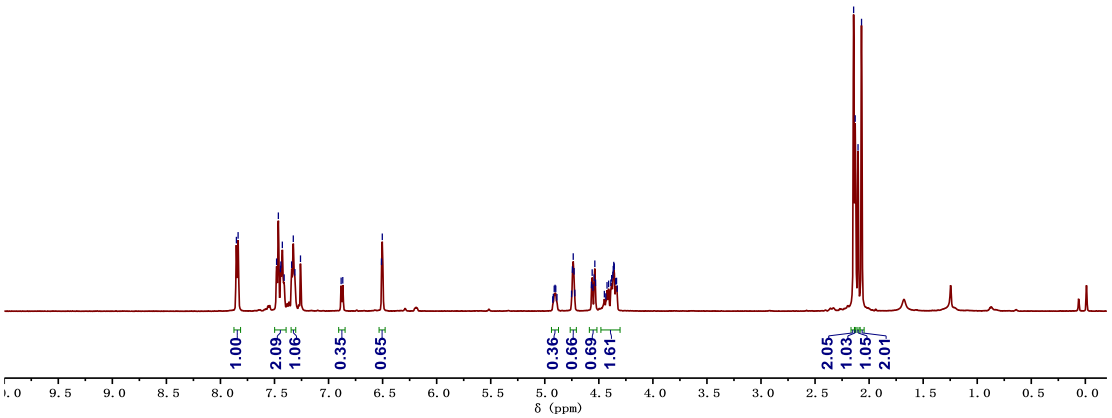
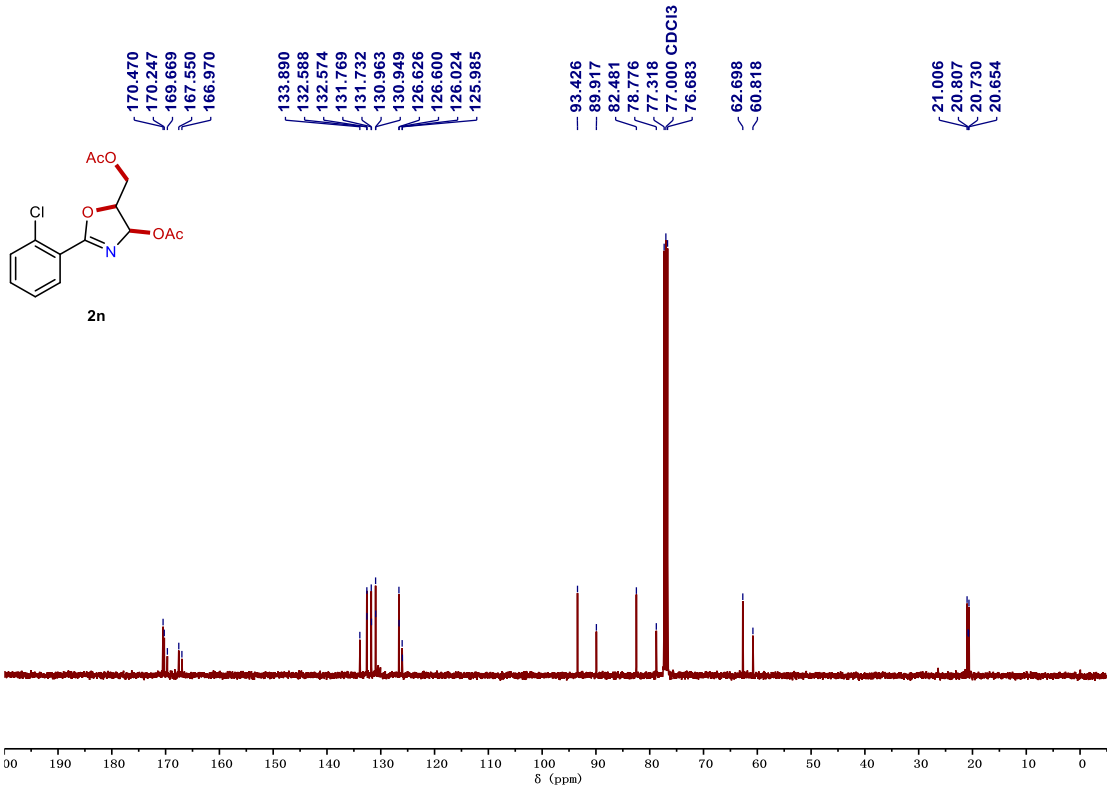
170.414
170.342
169.762
168.514
168.027
152.680
148.683
122.697
118.600
111.198
110.325
110.299
93.749
89.982
82.518
78.790
77.254
77.000 CDCl3
76.745
62.746
60.977
55.991
55.960
55.902
20.979
20.810
20.683
20.582
AcO
O
OAc
N
MeO
MeO
2m
δ (ppm)


7.852
7.837
7.481
7.464
7.445
7.441
7.428
7.411
7.342
7.326
7.311
7.260 CDCl3
6.884
6.869
6.510
6.503
4.927
4.921
4.913
4.905
4.898
4.887
4.752
4.745
4.737
4.729
4.722
4.568
4.562
4.555
4.544
4.537
4.531
4.449
4.436
4.424
4.410
4.389
4.381
4.371
4.367
4.362
4.357
4.345
4.336
4.330
2.143
2.129
2.103
2.070

AcO
Cl
O
OAc
N

2n


1.00
2.09
1.06
0.35
0.65
0.36
0.66
0.69
1.61
2.05
1.03
1.05
2.01
δ (ppm)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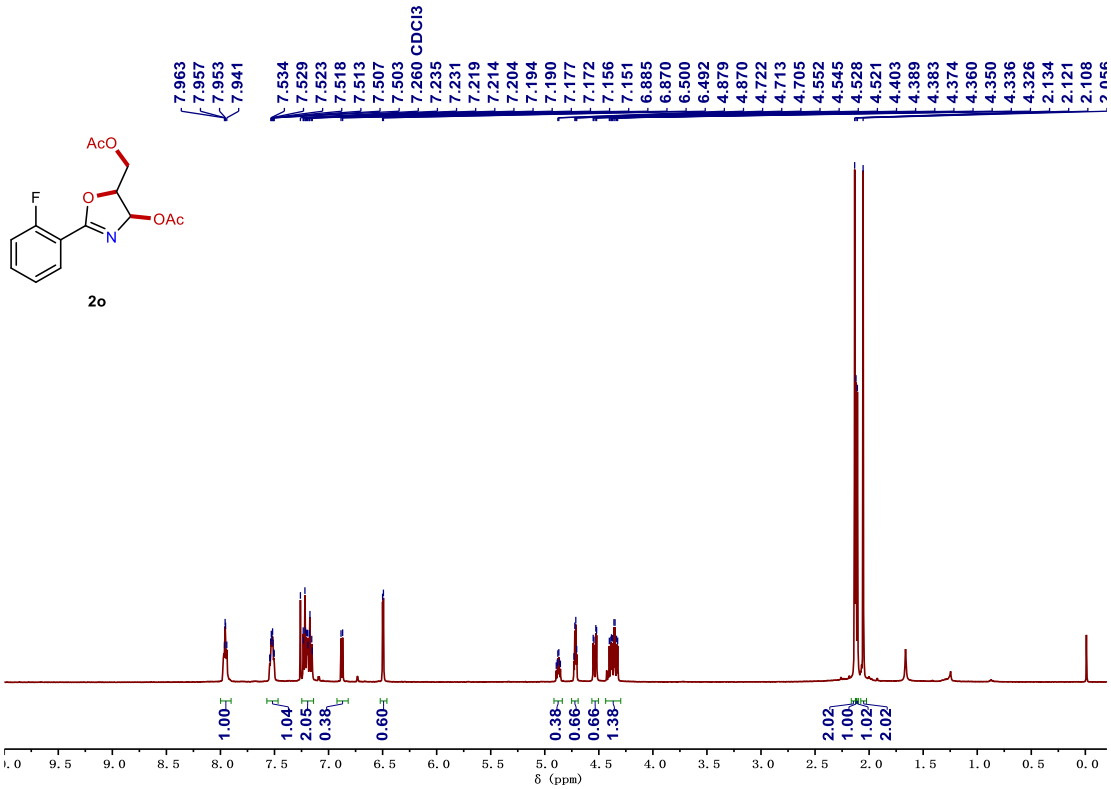
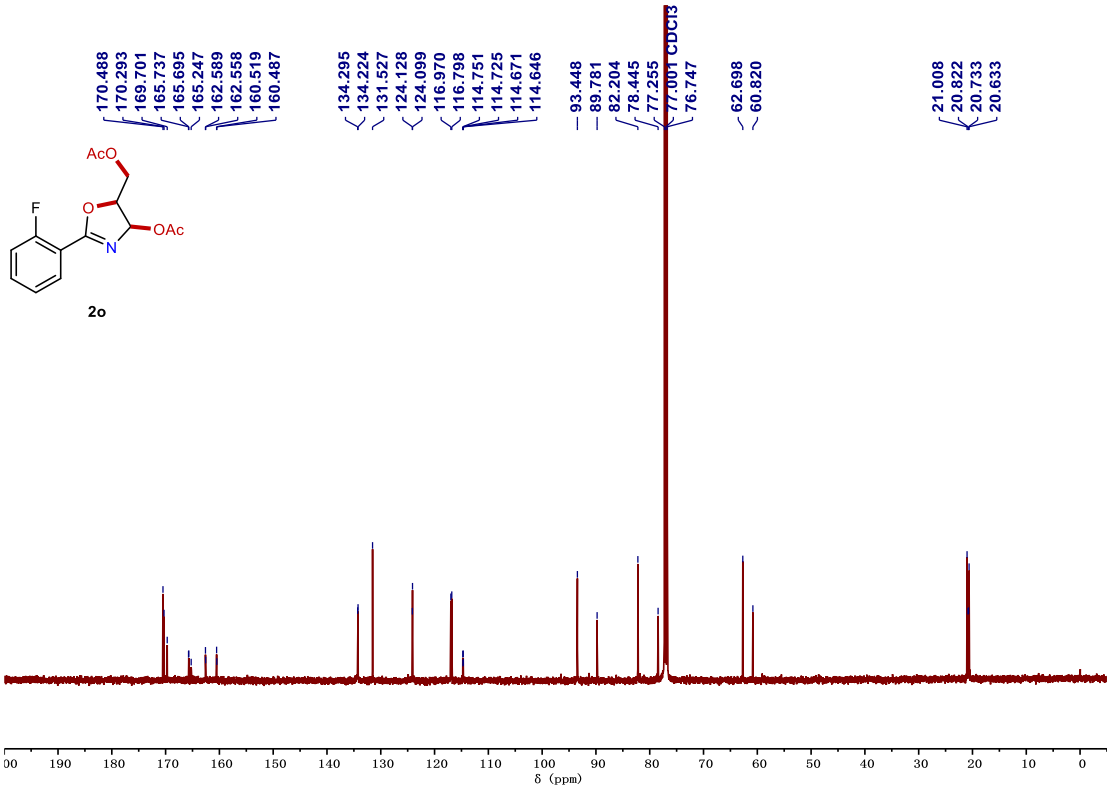
170.247
169.669
167.550
166.970
133.890
132.588
132.574
131.769
131.732
130.963
130.949
126.626
126.600
126.024
125.985
93.426
89.917
82.481
78.776
77.318
77.000 CDCl3
76.683
62.698
60.818
21.006
20.807
20.730
20.654
δ (ppm)

AcO
Cl
O
OAc
N

2n


7.963
7.957
7.953
7.941
7.534
7.529
7.523
7.518
7.513
7.507
7.503
7.260 CDCl3
7.235
7.231
7.219
7.214
7.204
7.194
7.190
7.177
7.172
7.156
7.151
6.885
6.870
6.500
6.492
4.879
4.870
4.722
4.713
4.705
4.552
4.545
4.528
4.521
4.403
4.389
4.383
4.374
4.360
4.350
4.336
4.326
2.134
2.121
2.108
AcO
F
O
OAc
N
2o
1.00
1.04
2.05
0.38
0.60
0.38
0.66
0.66
1.38
2.02
1.00
1.02
2.02
δ (ppm)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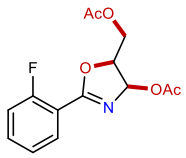
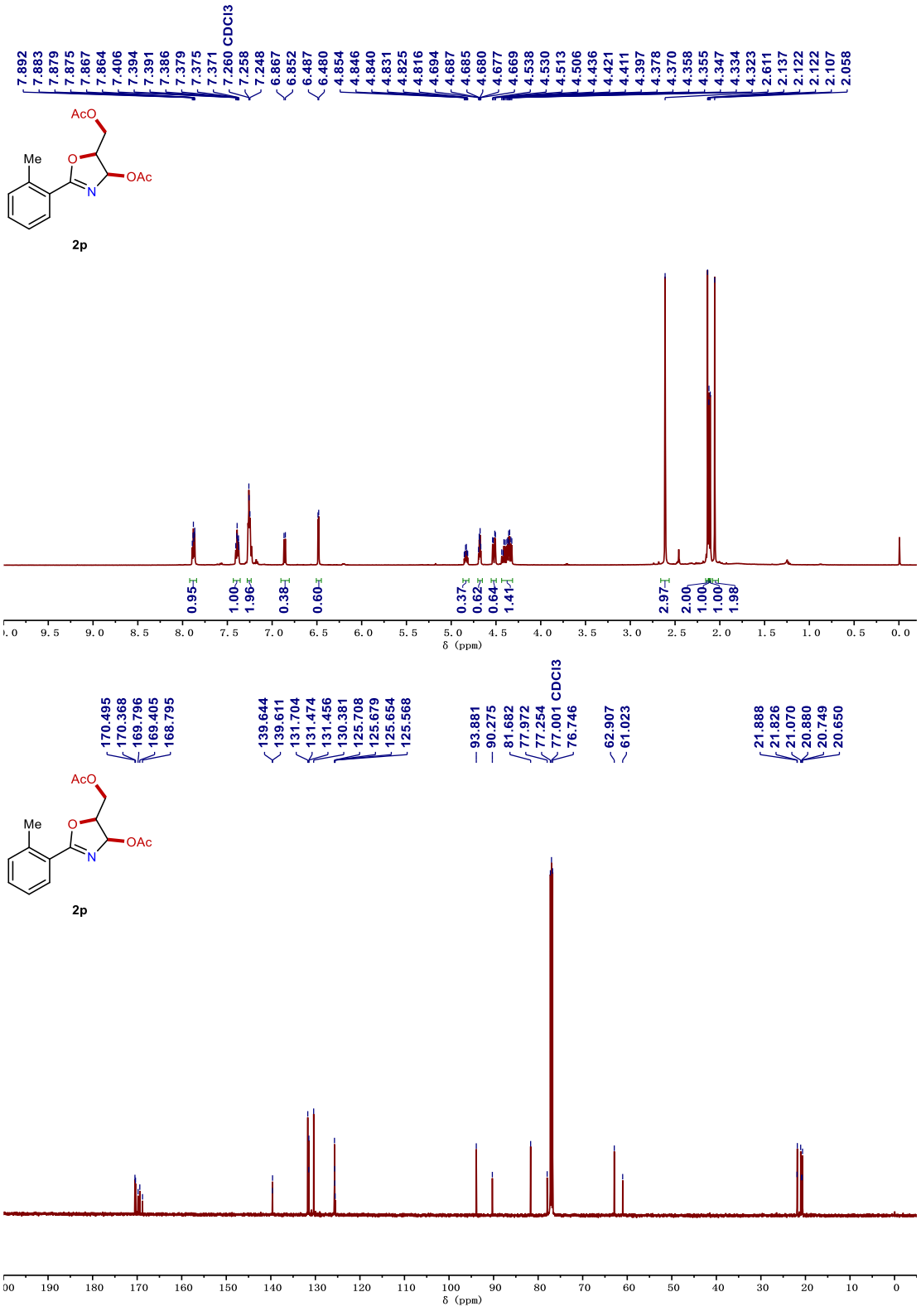
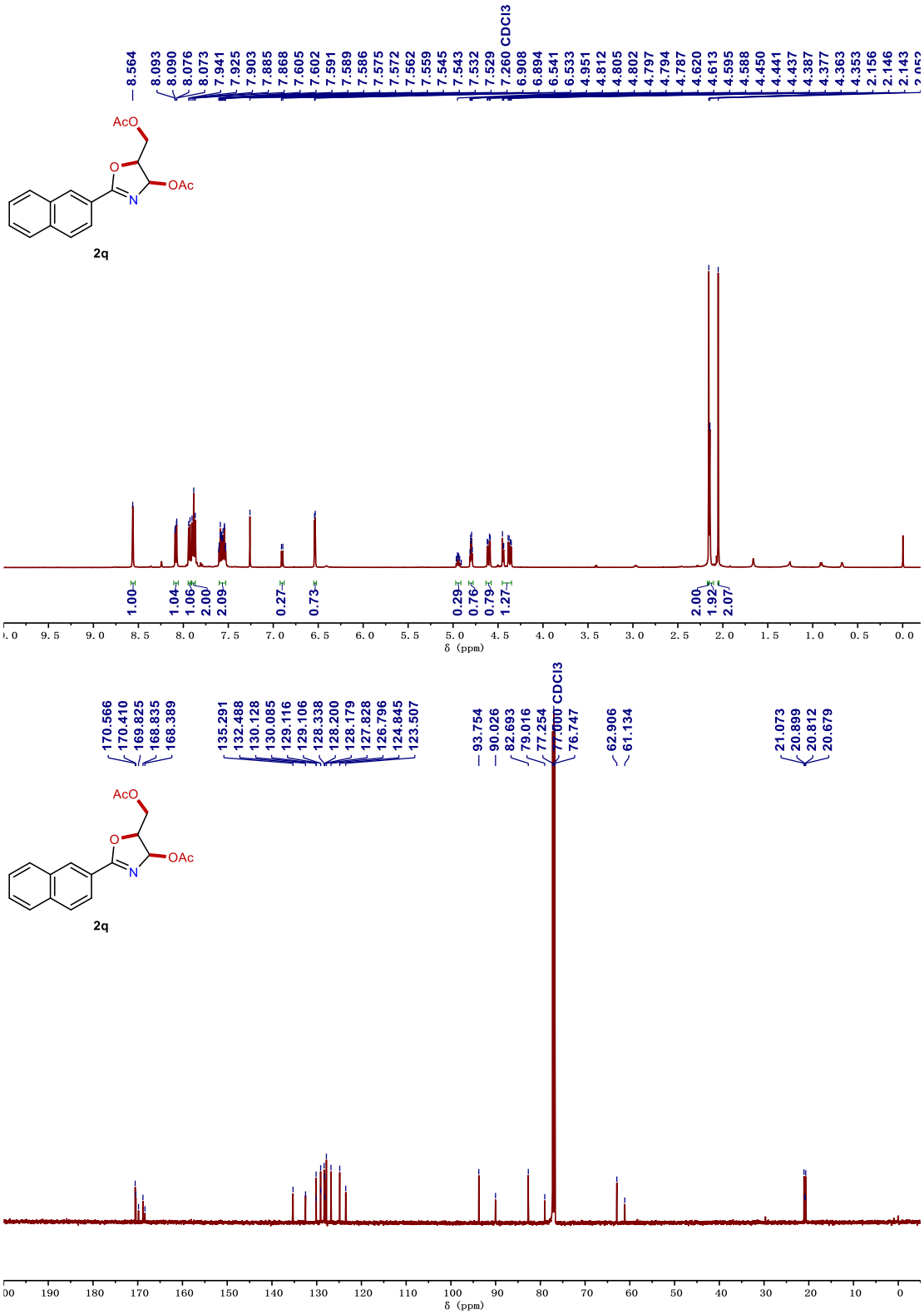
170.293
169.701
165.737
165.695
165.247
162.589
162.558
160.519
160.487
134.295
134.224
131.527
124.128
124.099
116.970
116.798
114.751
114.725
114.671
114.646
93.448
89.781
82.204
78.445
77.255
77.001 CDCl3
76.747
62.698
60.820
21.008
20.822
20.733
20.633
AcO
F
O
OAc
N
2o
δ (ppm)

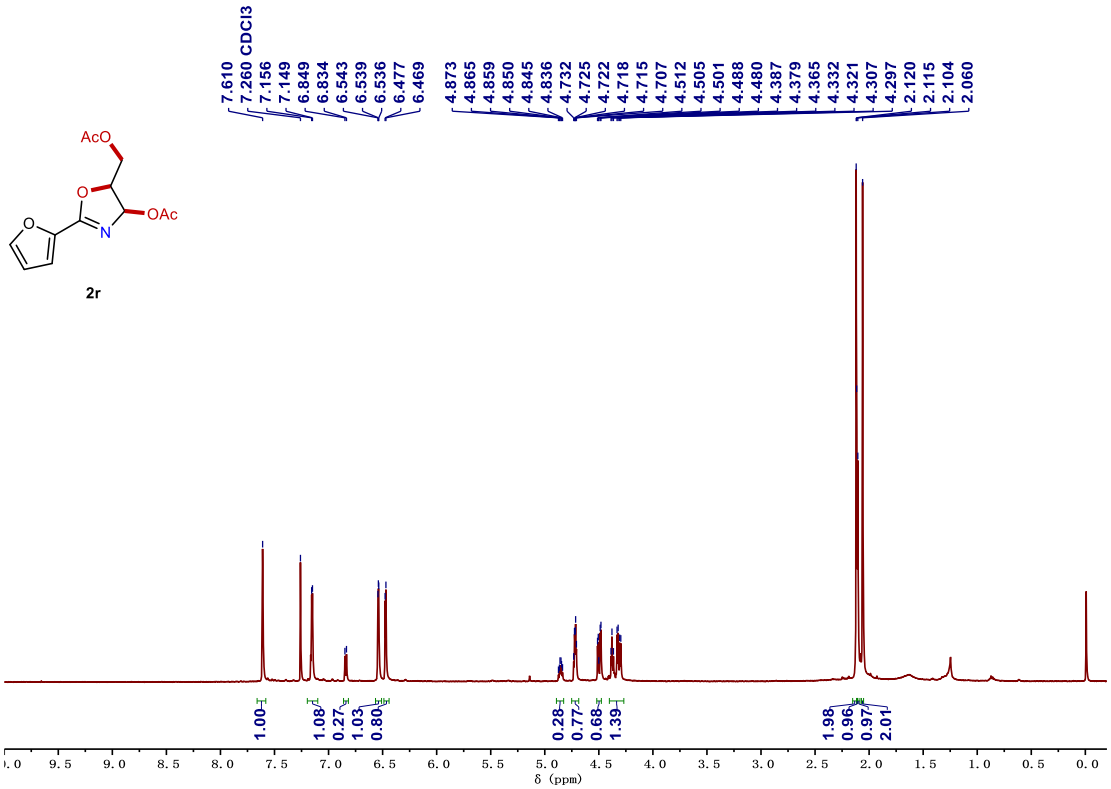

AcO
F
O
OAc
N

2o

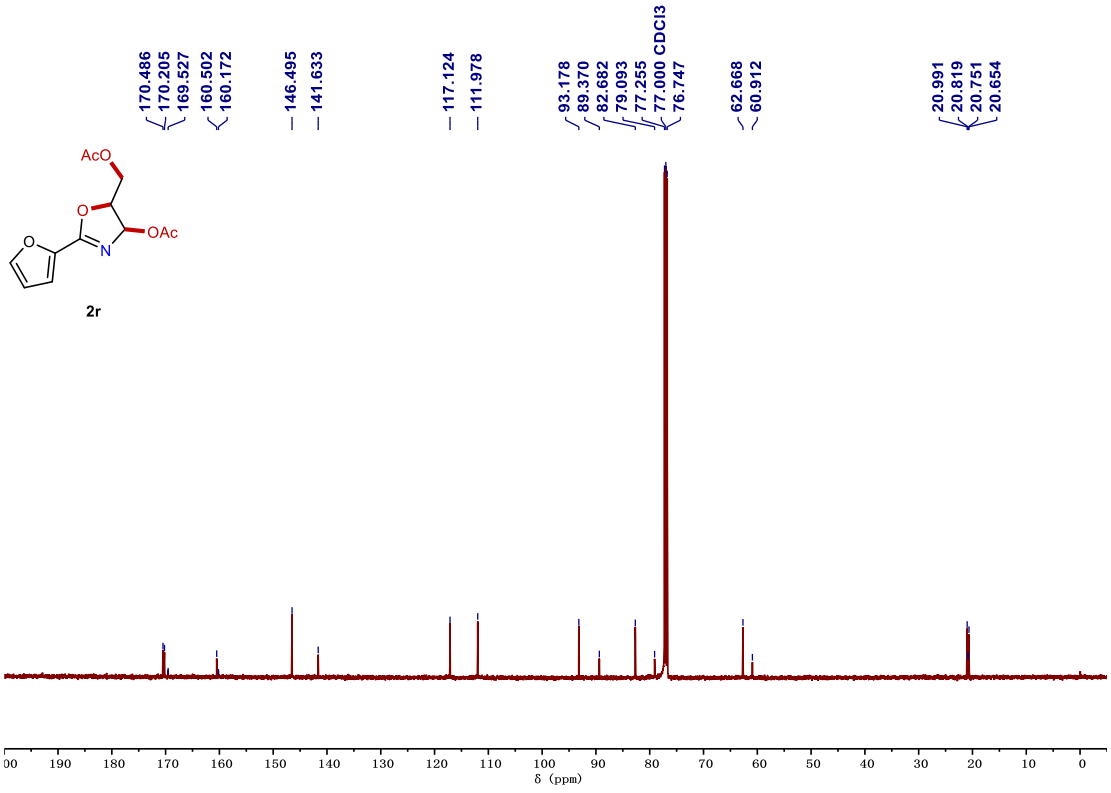
-107.542
-107.648
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)


7.892
7.883
7.879
7.875
7.867
7.864
7.406
7.394
7.391
7.386
7.379
7.375
7.371
CDCl3
7.258
7.248
6.867
6.852
6.487
6.480
4.854
4.846
4.840
4.831
4.825
4.816
4.694
4.687
4.685
4.680
4.677
4.669
4.538
4.530
4.513
4.506
4.436
4.421
4.411
4.397
4.378
4.370
4.358
4.355
4.347
4.334
4.323
2.611
2.137
2.122
2.122
2.107
2.058
AcO
Me
O
OAc
N
2p
0.95
1.00
1.96
0.38
0.60
0.37
0.62
0.64
1.41
2.97
2.00
1.00
1.00
1.98
δ (ppm)
170.495
170.368
169.796
169.405
168.795
139.644
139.611
131.704
131.474
131.456
130.381
125.708
125.679
125.654
125.568
93.881
90.275
81.682
77.972
77.254
77.001
CDCl3
76.746
62.907
61.023
21.888
21.826
21.070
20.880
20.749
20.650
AcO
Me
O
OAc
N
2p
δ (ppm)

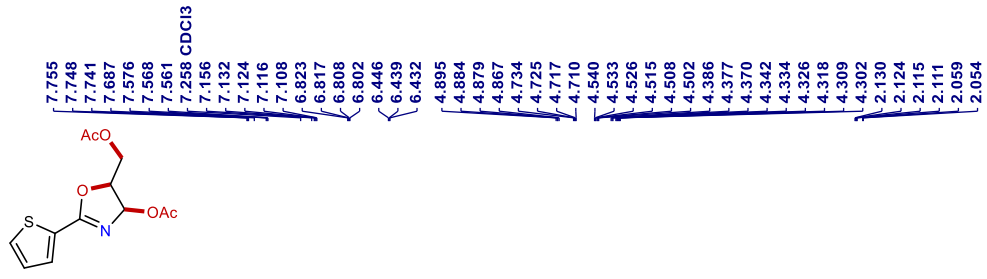

8.564
8.093
8.090
8.076
8.073
7.941
7.925
7.903
7.885
7.868
7.605
7.602
7.591
7.589
7.586
7.575
7.572
7.562
7.559
7.545
7.543
7.532
7.529
7.260 CDCl3
6.908
6.894
6.541
6.533
4.951
4.812
4.805
4.802
4.797
4.794
4.787
4.620
4.613
4.595
4.588
4.450
4.441
4.437
4.387
4.377
4.363
4.353
2.156
2.146
2.143
AcO
O
OAc
N
2q
1.00
1.04
1.06
2.00
2.09
0.27
0.73
0.29
0.76
0.79
1.27
2.00
1.92
2.07
δ (ppm)
170.566
170.410
169.825
168.835
168.389
135.291
132.488
130.128
130.085
129.116
129.106
128.338
128.200
128.179
127.828
126.796
124.845
123.507
93.754
90.026
82.693
79.016
77.254
77.000 CDCl3
76.747
62.906
61.134
21.073
20.899
20.812
20.679
AcO
O
OAc
N
2q
δ (ppm)


7.610
7.260 CDCl3
7.156
7.149
6.849
6.834
6.543
6.539
6.536
6.477
6.469
4.873
4.865
4.859
4.850
4.845
4.836
4.732
4.725
4.722
4.718
4.715
4.707
4.512
4.505
4.501
4.488
4.480
4.387
4.379
4.365
4.332
4.321
4.307
4.297
2.120
2.115
2.104
2.060
AcO
OAc
O
N
1.00
1.08
0.27
1.03
0.80
0.28
0.77
0.68
1.39
1.98
0.96
0.97
2.01
δ (ppm)

2r

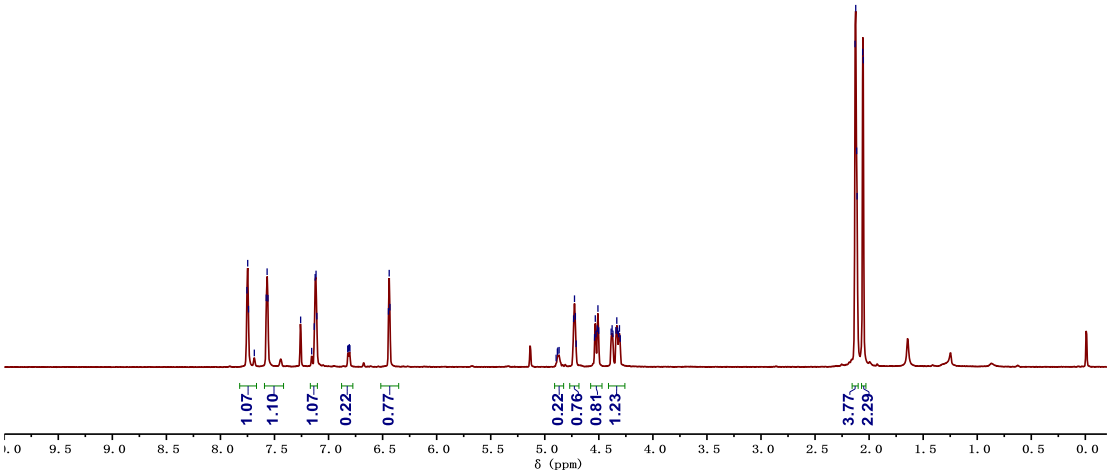

170.486
170.205
169.527
160.502
160.172
146.495
141.633
117.124
111.978
93.178
89.370
82.682
79.093
77.255
77.000 CDCl3
76.747
62.668
60.912
20.991
20.819
20.751
20.654
AcO
OAc
O
N
δ (ppm)

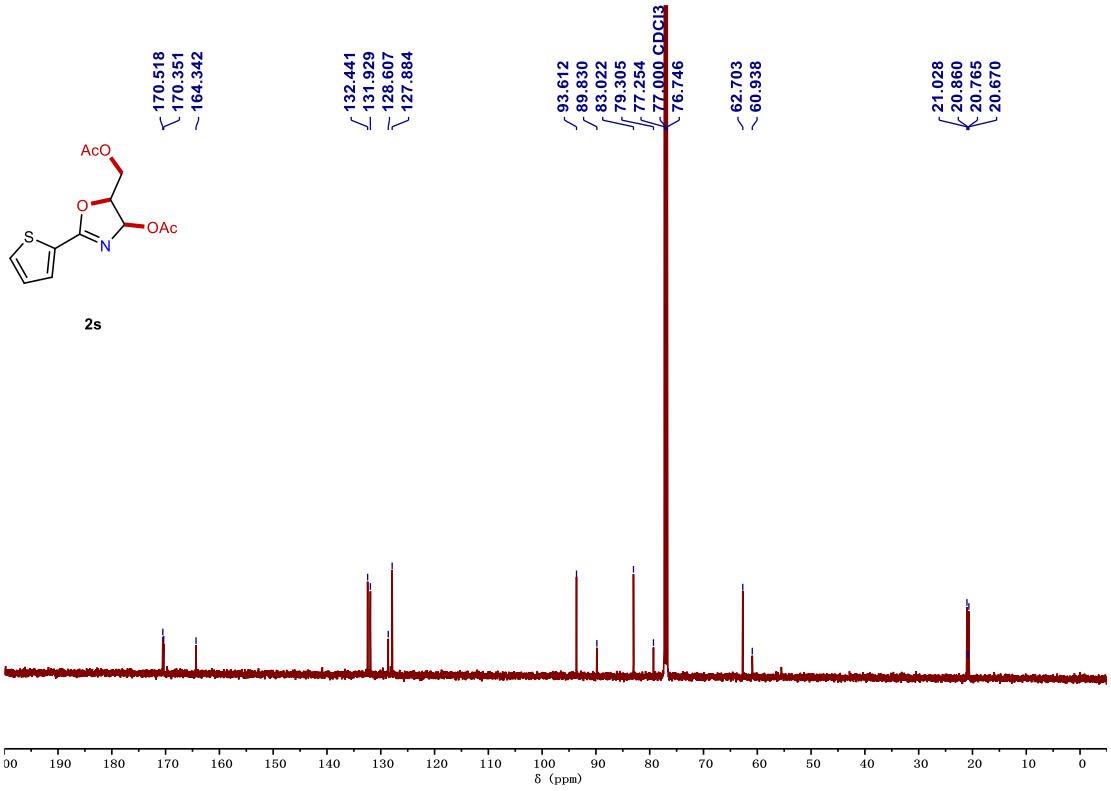
2r


7.755
7.748
7.741
7.687
7.576
7.568
7.561
7.258 CDCl3
7.156
7.132
7.124
7.116
7.108
6.823
6.817
6.808
6.802
6.446
6.439
6.432
4.895
4.884
4.879
4.867
4.734
4.725
4.717
4.710
4.540
4.533
4.526
4.515
4.508
4.502
4.386
4.377
4.370
4.342
4.334
4.326
4.318
4.309
4.302
2.130
2.124
2.115
2.111
2.059
2.054

AcO
O
OAc
S
N

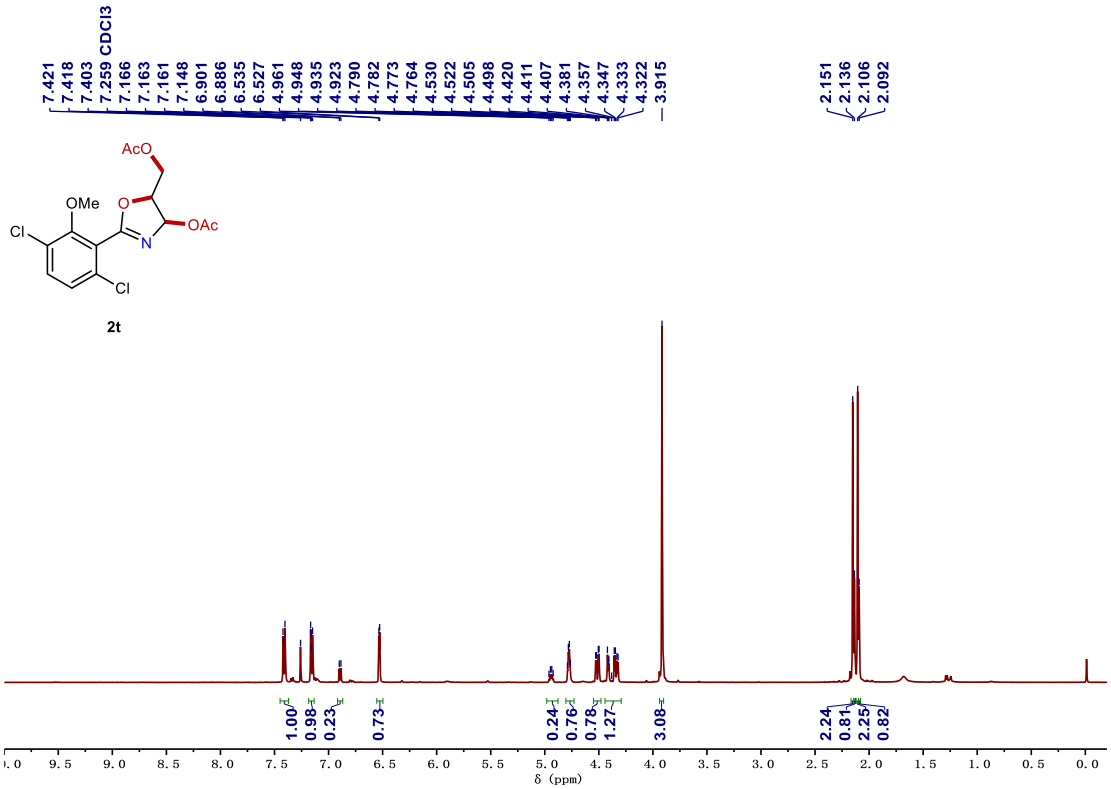
2s

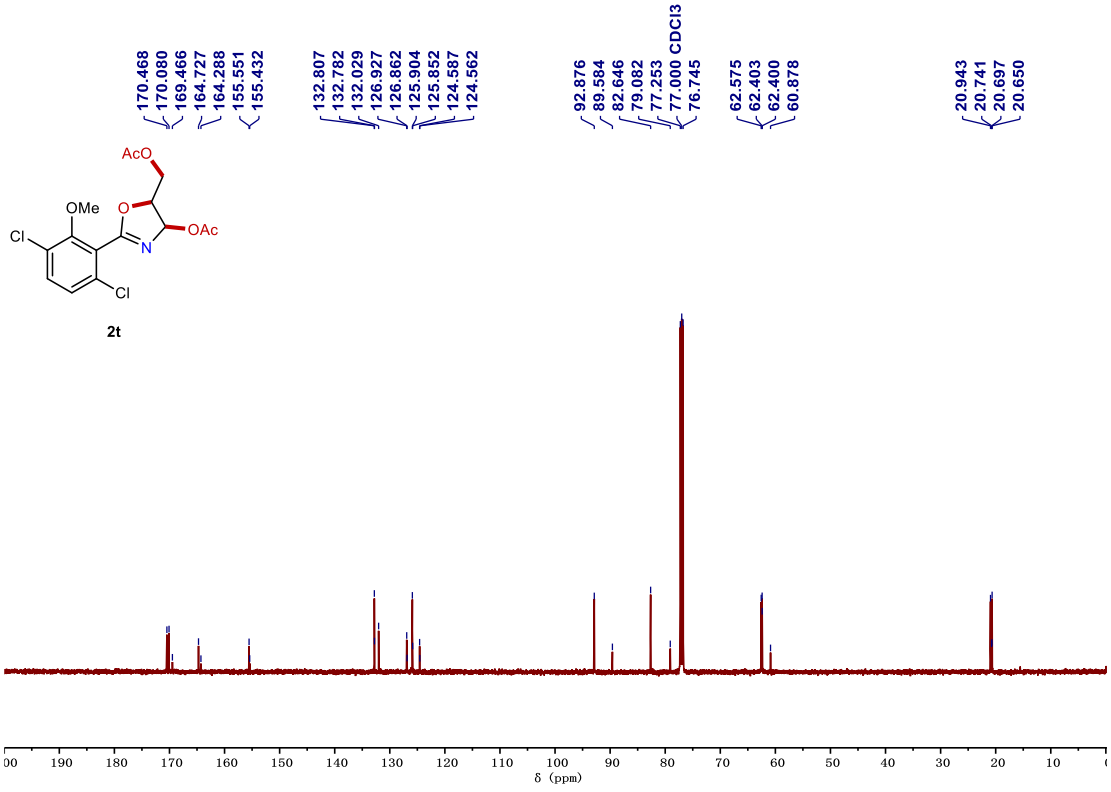

1.07
1.10
1.07
0.22
0.77
0.22
0.76
0.81
1.23
3.77
2.29
δ (ppm)

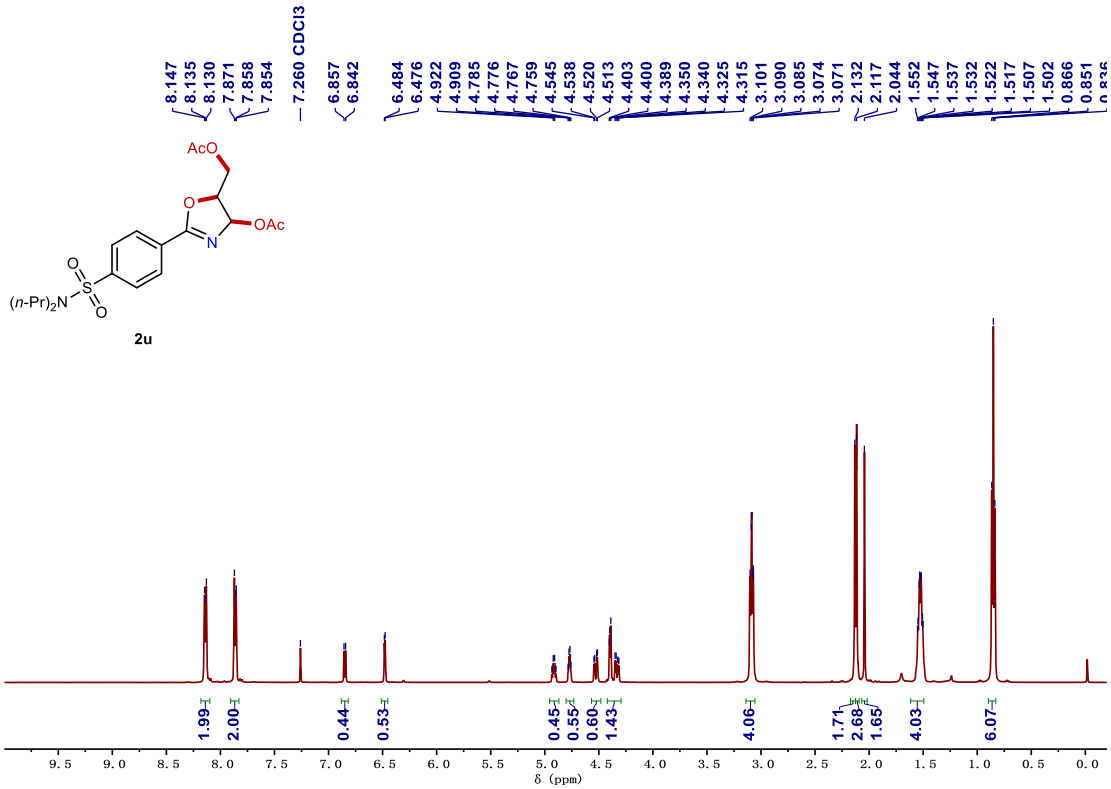

170.518
170.351
164.342
132.441
131.929
128.607
127.884
93.612
89.830
83.022
79.305
77.254
77.000 CDCl3
76.746
62.703
60.938
21.028
20.860
20.765
20.670
δ (ppm)

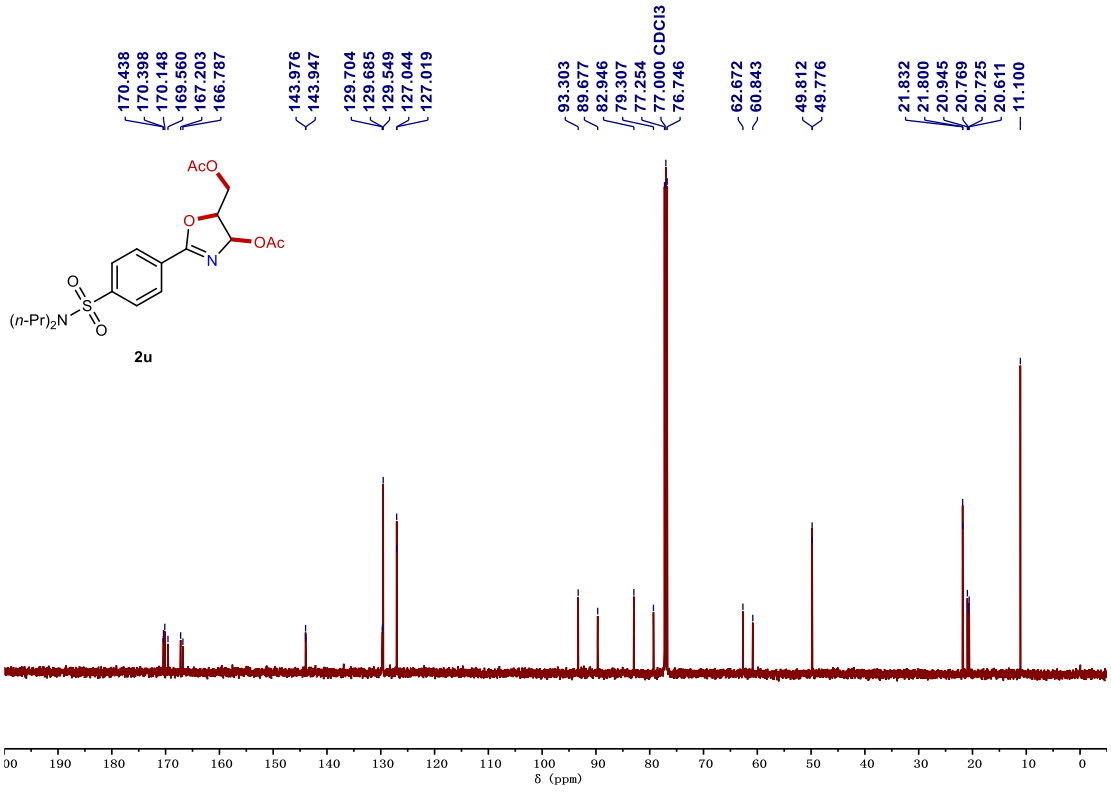
AcO
O
OAc
S
N

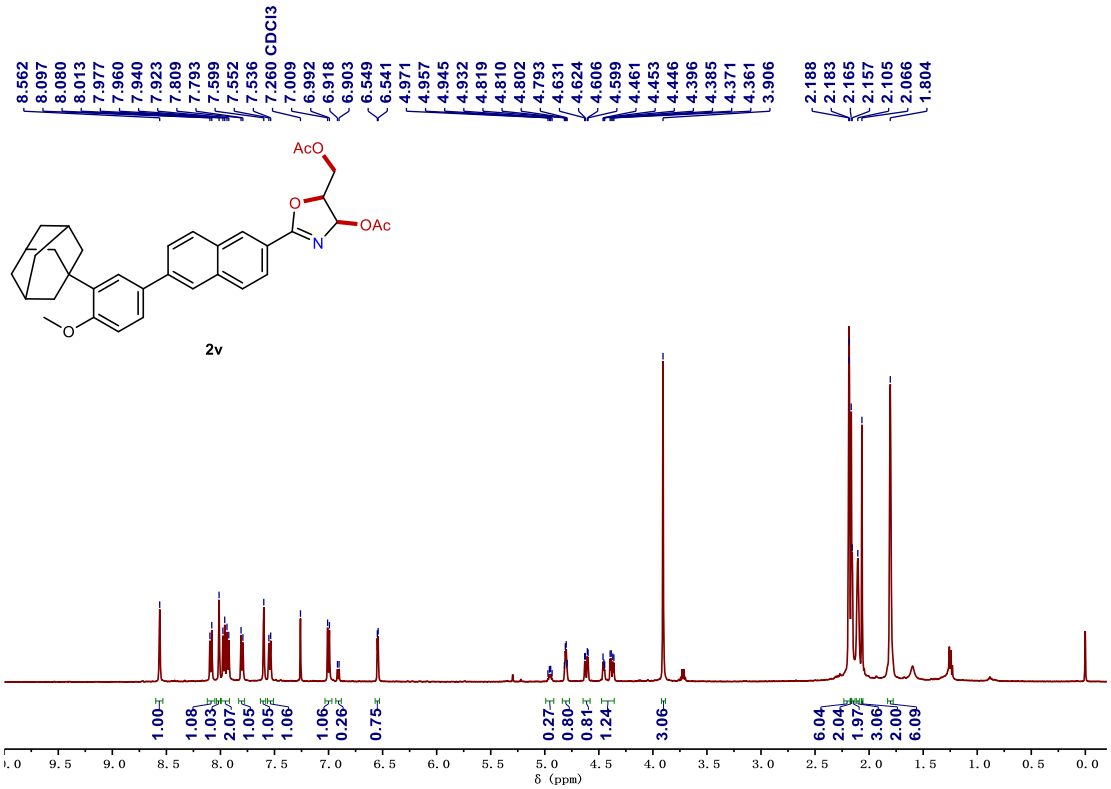
2s

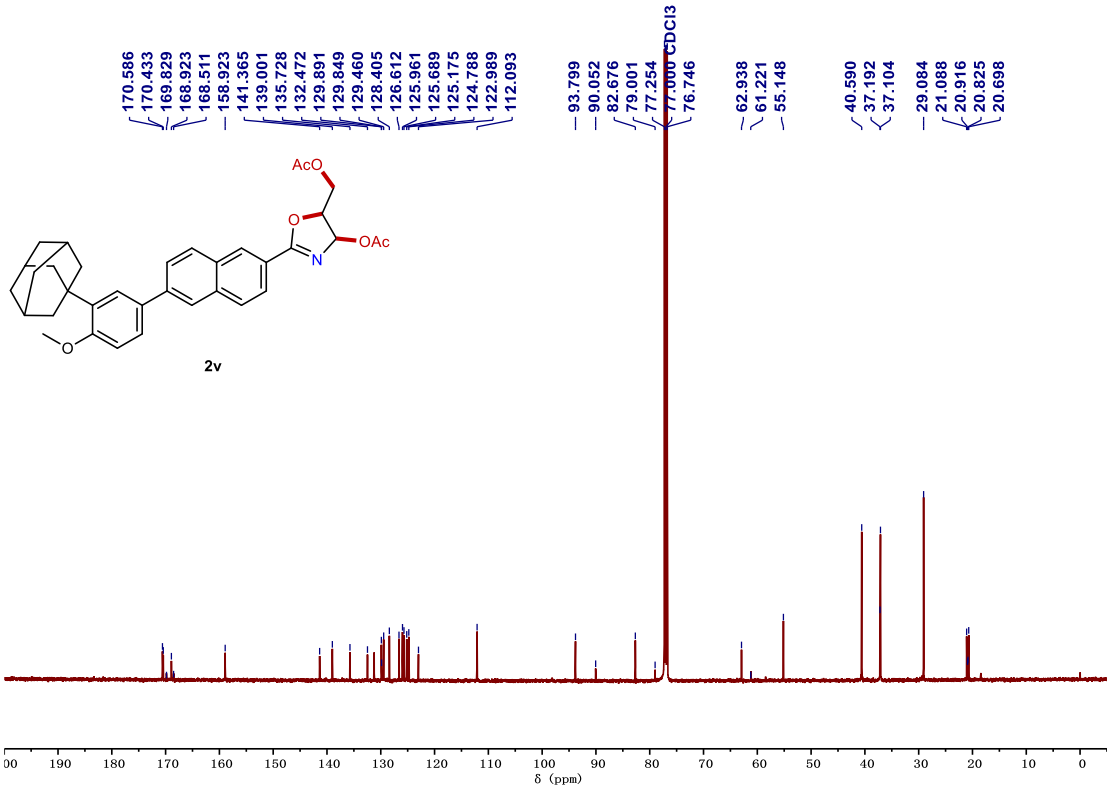

7.421
7.418
7.403
CDCl3
7.259
7.166
7.163
7.161
7.148
6.901
6.886
6.535
6.527
4.961
4.948
4.935
4.923
4.790
4.782
4.773
4.764
4.530
4.522
4.505
4.498
4.420
4.411
4.407
4.381
4.357
4.347
4.333
4.322
3.915
2.151
2.136
2.106
2.092
AcO
OMe
O
OAc
N
Cl
Cl
2t
1.00
0.98
0.23
0.73
0.24
0.76
0.78
1.27
3.08
2.24
0.81
2.25
0.82
δ (ppm)

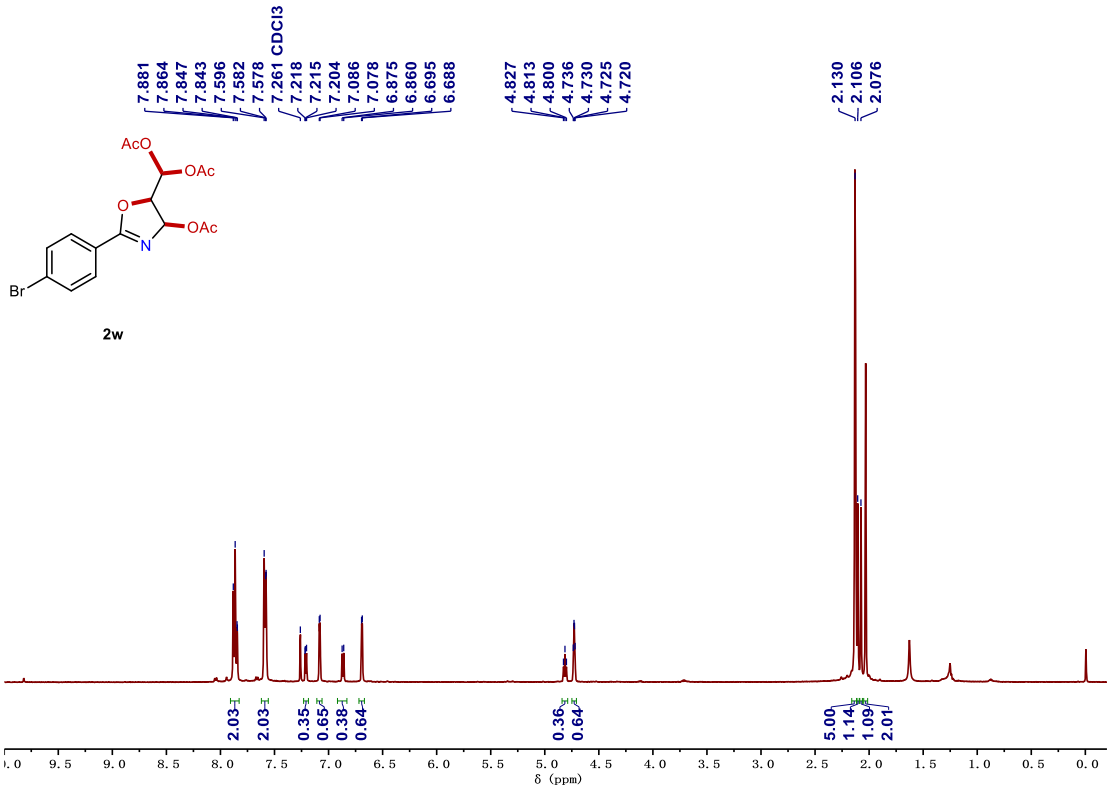

170.468
170.080
169.466
164.727
164.288
155.551
155.432
132.807
132.782
132.029
126.927
126.862
125.904
125.852
124.587
124.562
92.876
89.584
82.646
79.082
77.253
77.000
CDCl3
76.745
62.575
62.403
62.400
60.878
20.943
20.741
20.697
20.650
AcO
OMe
O
OAc
N
Cl
Cl
2t
δ (ppm)

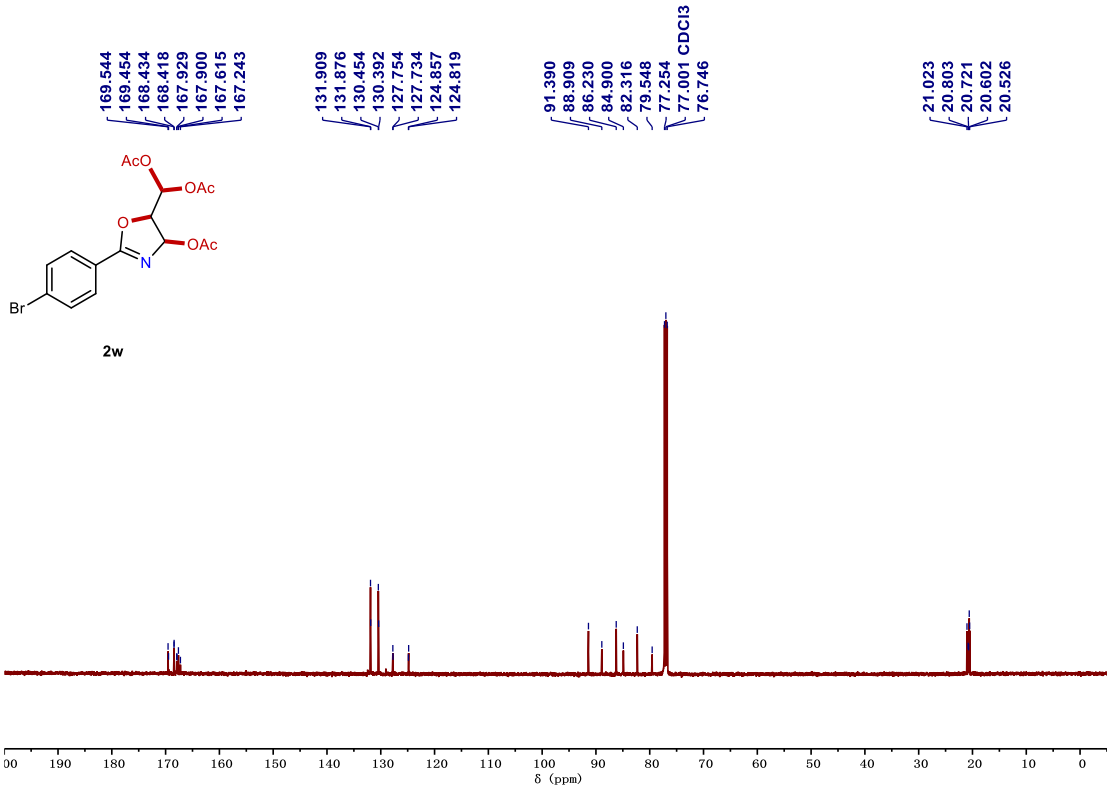

8.147
8.135
8.130
7.871
7.858
7.854
7.260 CDCl3
6.857
6.842
6.484
6.476
4.922
4.909
4.785
4.776
4.767
4.759
4.545
4.538
4.520
4.513
4.403
4.400
4.389
4.350
4.340
4.325
4.315
3.101
3.090
3.085
3.074
3.071
2.132
2.117
2.044
1.552
1.547
1.537
1.532
1.522
1.517
1.507
1.502
0.866
0.851
AcO
O
OAc
N
O
S
O
(n-Pr)2N
2u
1.99
2.00
0.44
0.53
0.45
0.55
0.60
1.43
4.06
1.71
2.68
1.65
4.03
6.07
δ (ppm)

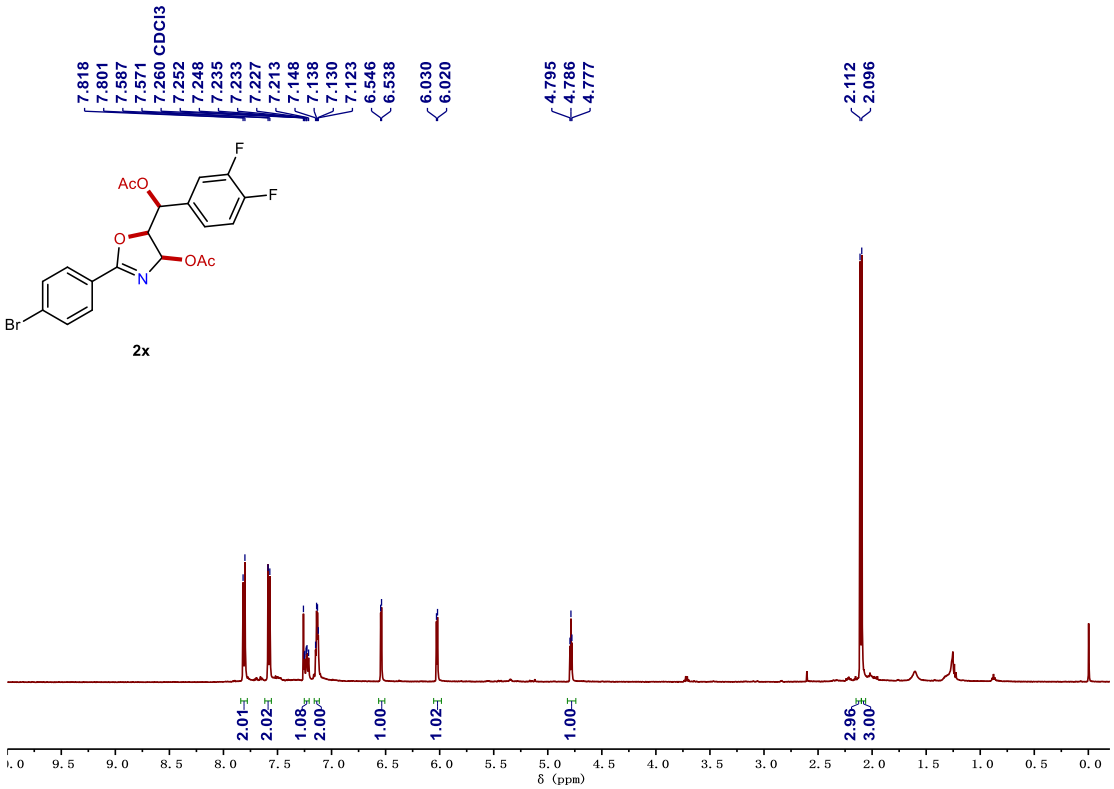

170.438
170.398
170.148
169.560
167.203
166.787
143.976
143.947
129.704
129.685
129.549
127.044
127.019
93.303
89.677
82.946
79.307
77.254
77.000 CDCl3
76.746
62.672
60.843
49.812
49.776
21.832
21.800
20.945
20.769
20.725
20.611
11.100
AcO
O
OAc
N
O
S
O
(n-Pr)2N
2u
δ (ppm)

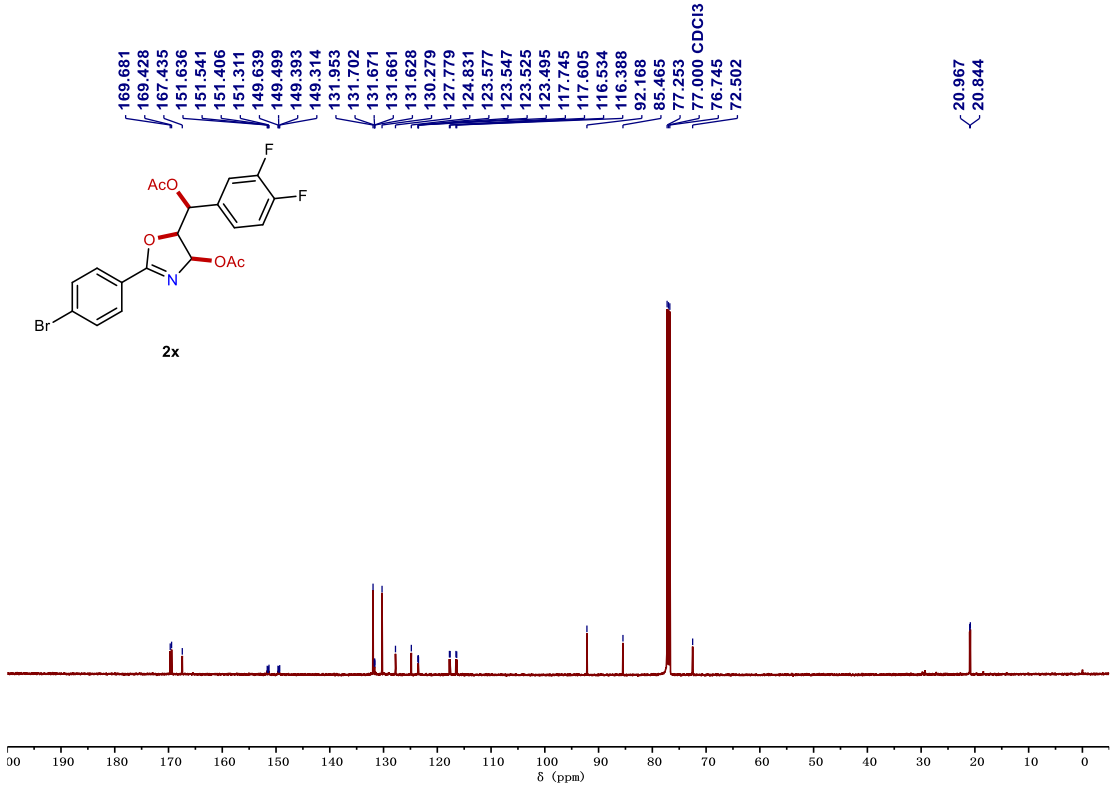

8.562
8.097
8.080
8.013
7.977
7.960
7.940
7.923
7.809
7.793
7.599
7.552
7.536
7.260 CDCl3
7.009
6.992
6.918
6.903
6.549
6.541
4.971
4.957
4.945
4.932
4.819
4.810
4.802
4.793
4.631
4.624
4.606
4.599
4.461
4.453
4.446
4.396
4.385
4.371
4.361
3.906
2.188
2.183
2.165
2.157
2.105
2.066
1.804
AcO
O
OAc
N
O
2v
1.00
1.08
1.03
2.07
1.05
1.05
1.06
1.06
0.26
0.75
0.27
0.80
0.81
1.24
3.06
6.04
2.04
1.97
3.06
2.00
6.09
δ (ppm)


170.586
170.433
169.829
168.923
168.511
158.923
141.365
139.001
135.728
132.472
129.891
129.849
129.460
128.405
126.612
125.961
125.689
125.175
124.788
122.989
112.093
93.799
90.052
82.676
79.001
77.254
77.000 CDCl3
76.746
62.938
61.221
55.148
40.590
37.192
37.104
29.084
21.088
20.916
20.825
20.698
AcO
O
OAc
N
O
2v
δ (ppm)


7.881
7.864
7.847
7.843
7.596
7.582
7.578
7.261 CDCl3
7.218
7.215
7.204
7.086
7.078
6.875
6.860
6.695
6.688
4.827
4.813
4.800
4.736
4.730
4.725
4.720
2.130
2.106
2.076
AcO
OAc
OAc
O
N
Br
2w
2.03
2.03
0.35
0.65
0.38
0.64
0.36
0.64
5.00
1.14
1.09
2.01
δ (ppm)


169.544
169.454
168.434
168.418
167.929
167.900
167.615
167.243
131.909
131.876
130.454
130.392
127.754
127.734
124.857
124.819
91.390
88.909
86.230
84.900
82.316
79.548
77.254
77.001 CDCl3
76.746
21.023
20.803
20.721
20.602
20.526
AcO
OAc
OAc
O
N
Br
2w
δ (ppm)


7.818
7.801
7.587
7.571
7.260 CDCl3
7.252
7.248
7.235
7.233
7.227
7.213
7.148
7.138
7.130
7.123
6.546
6.538
6.030
6.020
4.795
4.786
4.777
2.112
2.096
F
F
AcO
O
N
OAc
Br
2x
2.01
2.02
1.08
2.00
1.00
1.02
1.00
2.96
3.00
δ (ppm)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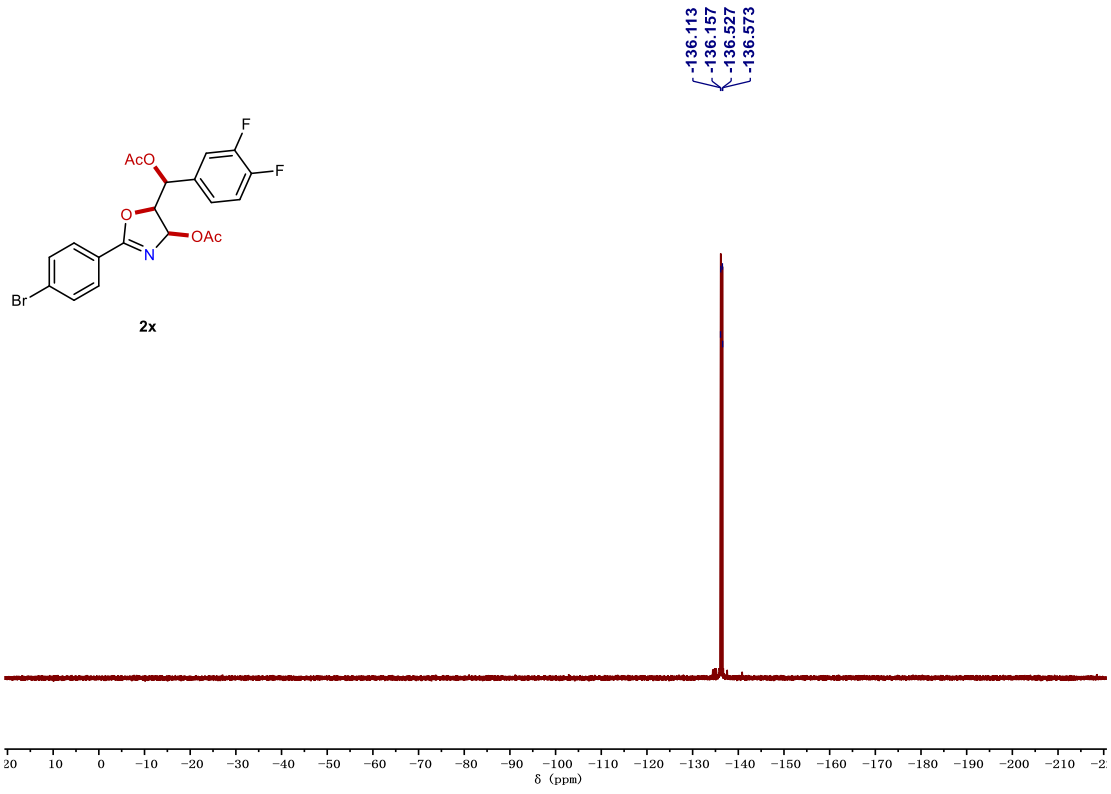
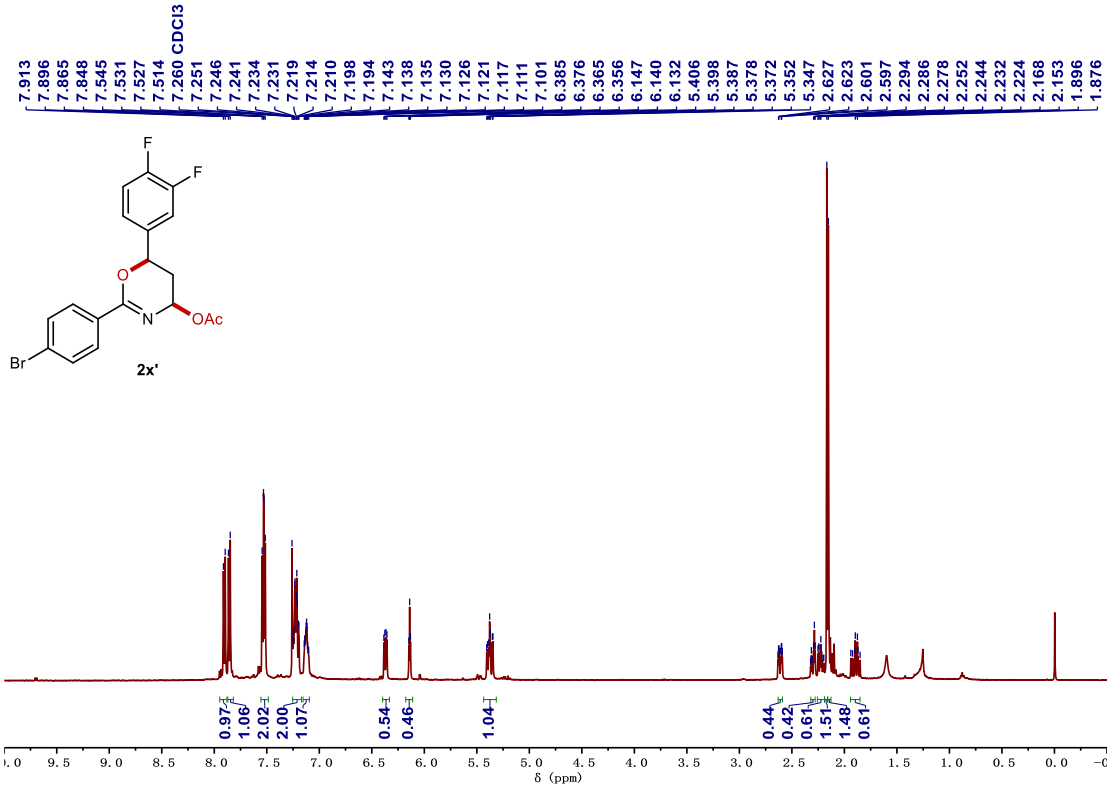
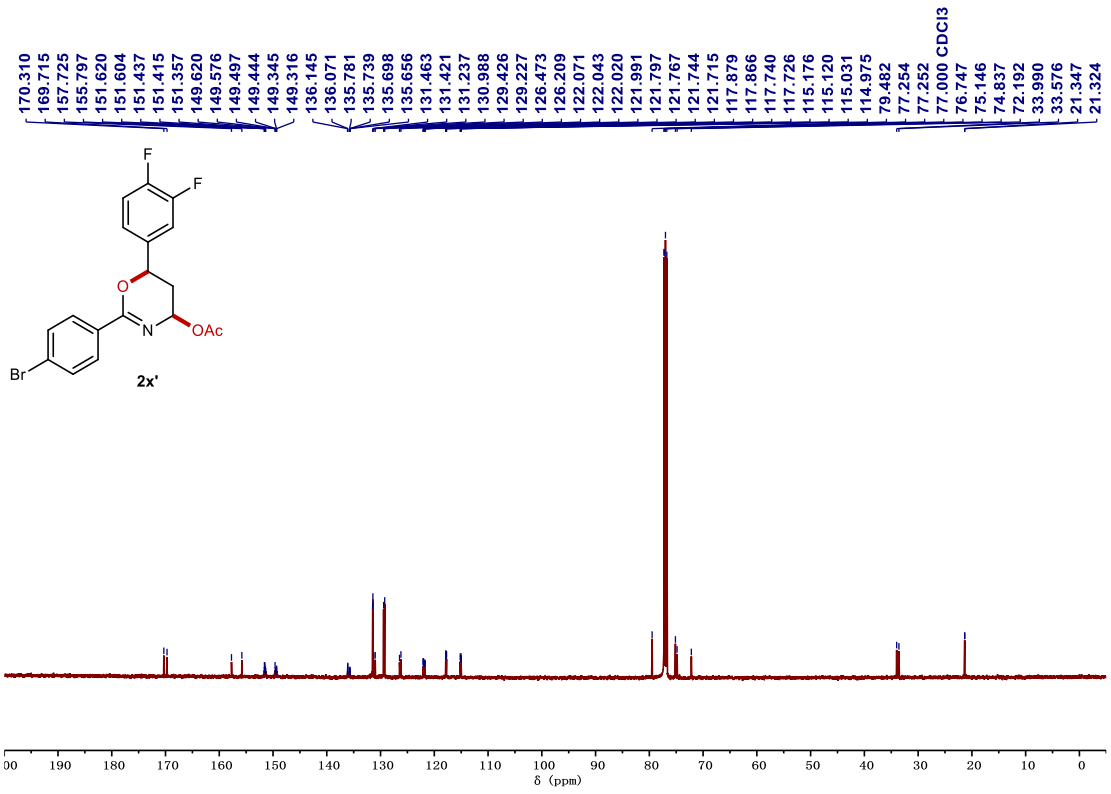
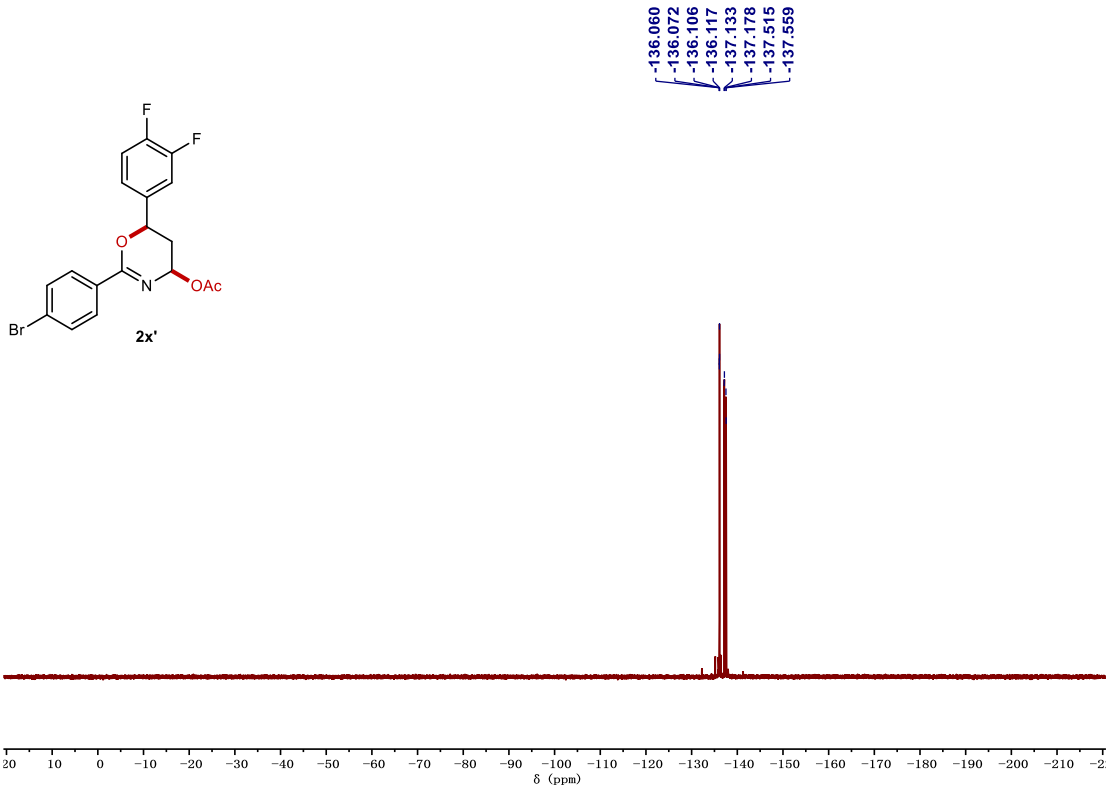
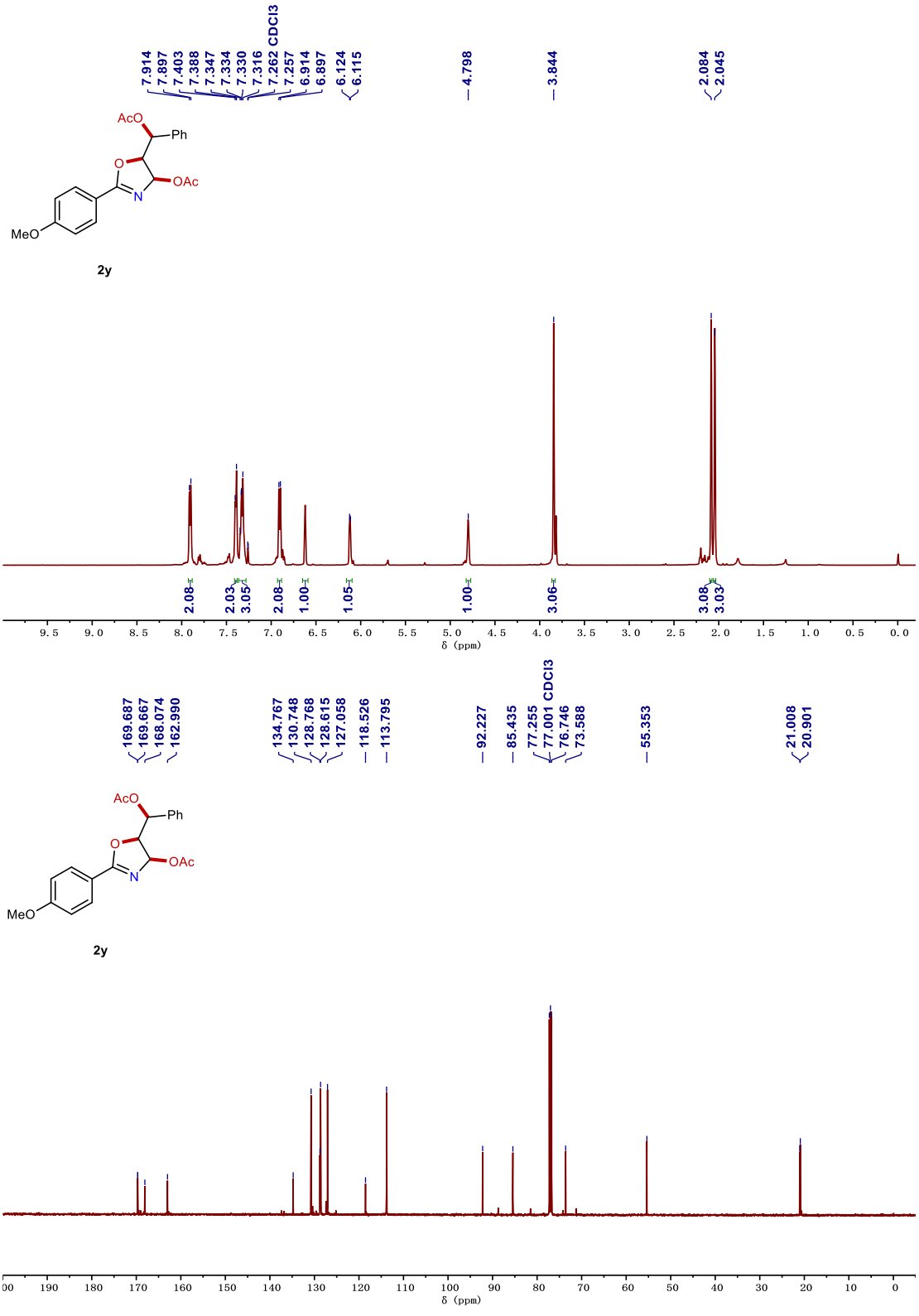
169.428
167.435
151.636
151.541
151.406
151.311
149.639
149.499
149.393
149.314
131.953
131.702
131.671
131.661
131.628
130.279
127.779
124.831
123.577
123.547
123.525
123.495
117.745
117.605
116.534
116.388
92.168
85.465
77.253
77.000 CDCl3
76.745
72.502
20.967
20.844
F
F
AcO
O
N
OAc
Br
2x
δ (ppm)

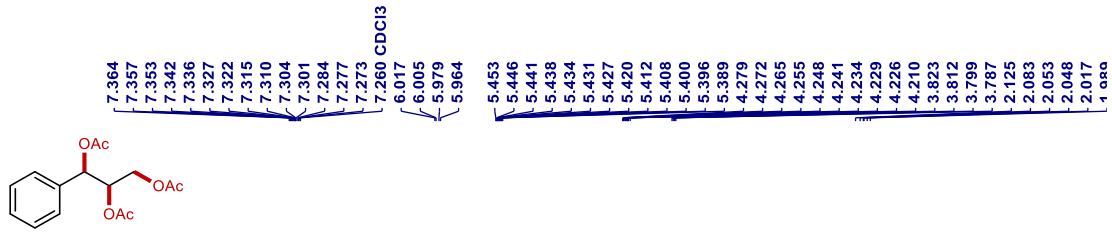

-136.113
-136.157
-136.527
-136.573
F
AcO
F
O
OAc
N
Br
2x
δ (ppm)


7.913
7.896
7.865
7.848
7.545
7.531
7.527
7.514
7.260 CDCl3
7.251
7.246
7.241
7.234
7.231
7.219
7.214
7.210
7.198
7.194
7.143
7.138
7.135
7.130
7.126
7.121
7.117
7.111
7.101
6.385
6.376
6.365
6.356
6.147
6.140
6.132
5.406
5.398
5.387
5.378
5.372
5.352
5.347
2.627
2.623
2.601
2.597
2.294
2.286
2.278
2.252
2.244
2.232
2.224
2.168
2.153
1.896
1.876
F
F
O
N
OAc
Br
2x'
0.97
1.06
2.02
2.00
1.07
0.54
0.46
1.04
0.44
0.42
0.61
1.51
1.48
0.61
δ (ppm)

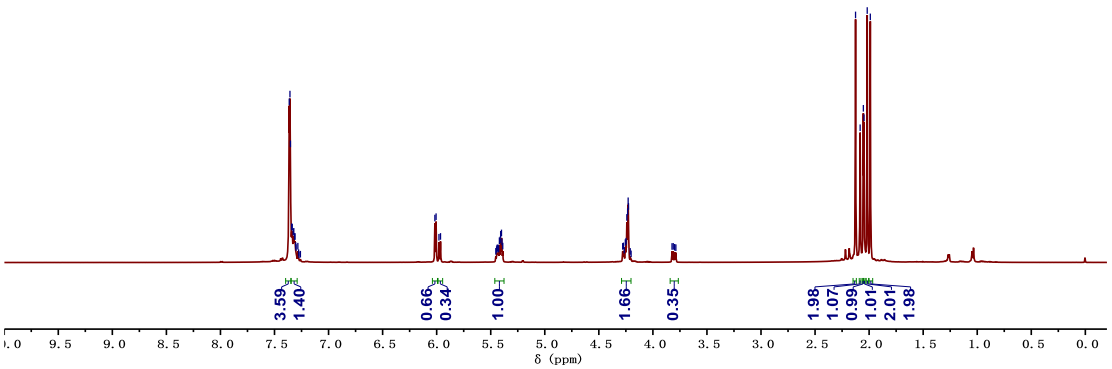

170.310
169.715
157.725
155.797
151.620
151.604
151.437
151.415
151.357
149.620
149.576
149.497
149.444
149.345
149.316
136.145
136.071
135.781
135.739
135.698
135.656
131.463
131.421
131.237
130.988
129.426
129.227
126.473
126.209
122.071
122.043
122.020
121.991
121.797
121.767
121.744
121.715
117.879
117.866
117.740
117.726
115.176
115.120
115.031
114.975
79.482
77.254
77.252
77.000 CDCl3
76.747
75.146
74.837
72.192
33.990
33.576
21.347
21.324
F
F
O
N
OAc
Br
2x'
δ (ppm)

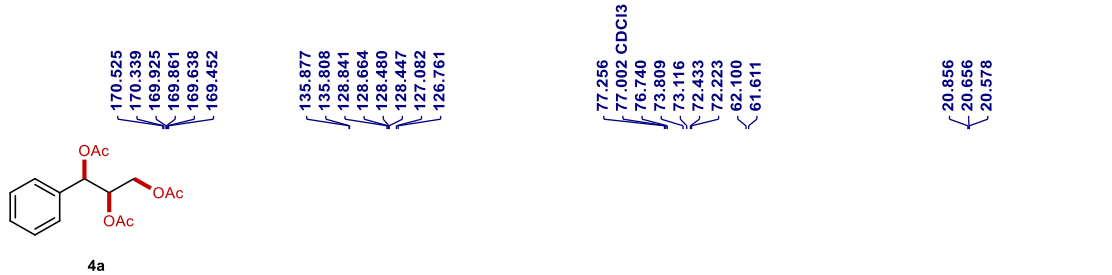

-136.060
-136.072
-136.106
-136.117
-137.133
-137.178
-137.515
-137.559
F
F
O
N
OAc
Br
2x'
δ (ppm)


7.914
7.897
7.403
7.388
7.347
7.334
7.330
7.316
7.262 CDCl3
7.257
6.914
6.897
6.124
6.115
4.798
3.844
2.084
2.045
AcO
Ph
O
OAc
N
MeO
2y
2.08
2.03
3.05
2.08
1.00
1.05
1.00
3.06
3.08
3.03
δ (ppm)
169.687
169.667
168.074
162.990
134.767
130.748
128.768
128.615
127.058
118.526
113.795
92.227
85.435
77.255
77.001 CDCl3
76.746
73.588
55.353
21.008
20.901
δ (ppm)

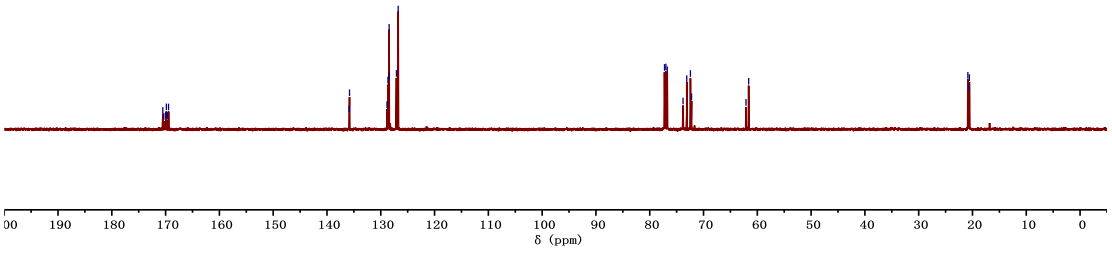

7.364
7.357
7.353
7.342
7.336
7.327
7.322
7.315
7.310
7.304
7.301
7.284
7.277
7.273
7.260 CDCl3
6.017
6.005
5.979
5.964
5.453
5.446
5.441
5.438
5.434
5.431
5.427
5.420
5.412
5.408
5.400
5.396
5.389
4.279
4.272
4.265
4.255
4.248
4.241
4.234
4.229
4.226
4.210
3.823
3.812
3.799
3.787
2.125
2.083
2.053
2.048
2.017
OAc
OAc
OAc

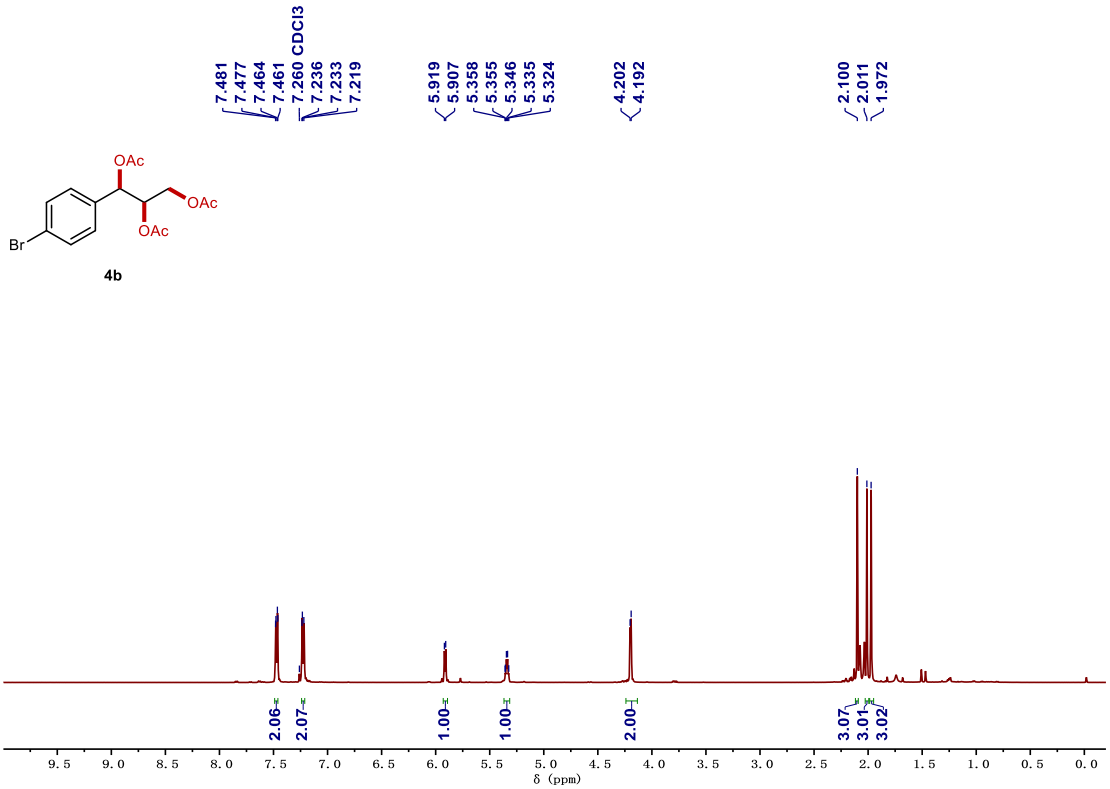
4a

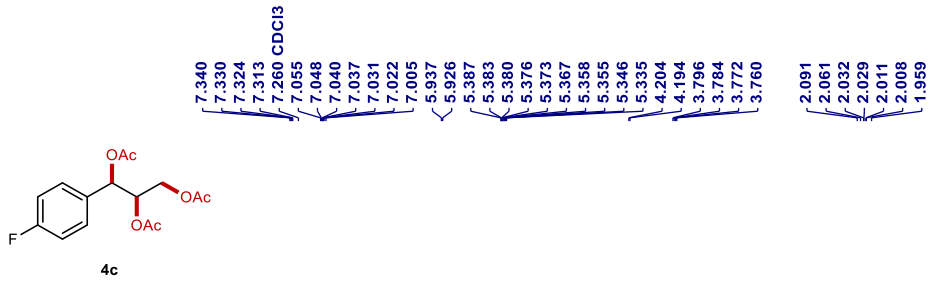

3.59
1.40
0.66
0.34
1.00
1.66
0.35
1.98
1.07
0.99
1.01
2.01
1.98
δ (ppm)


170.525
170.339
169.925
169.861
169.638
169.452
135.877
135.808
128.841
128.664
128.480
128.447
127.082
126.761
77.256
77.002 CDCl3
76.740
73.809
73.116
72.433
72.223
62.100
61.611
20.856
20.656
20.578
OAc
OAc
OAc

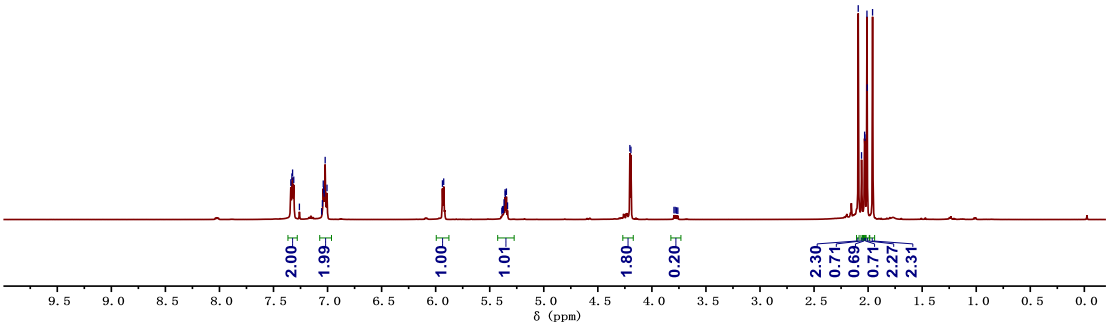
4a

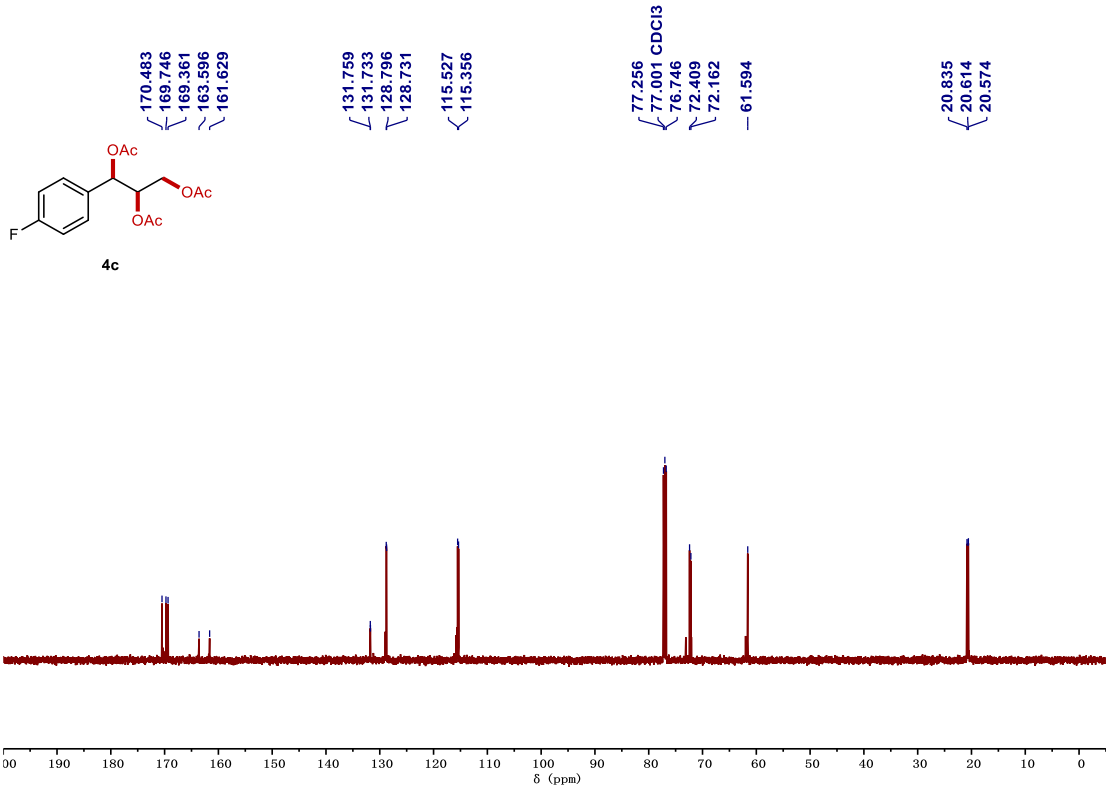

δ (ppm)


7.481
7.477
7.464
7.461
7.260 CDCl3
7.236
7.233
7.219
5.919
5.907
5.358
5.355
5.346
5.335
5.324
4.202
4.192
2.100
2.011
1.972
OAc
OAc
OAc
Br
4b
2.06
2.07
1.00
1.00
2.00
3.07
3.01
3.02
δ (ppm)
170.472
169.760
169.332
134.963
131.644
128.593
122.608
77.255
77.000 CDCl3
76.746
72.465
72.071
61.506
20.827
20.657
20.587
OAc
OAc
OAc
Br
4b
δ (ppm)

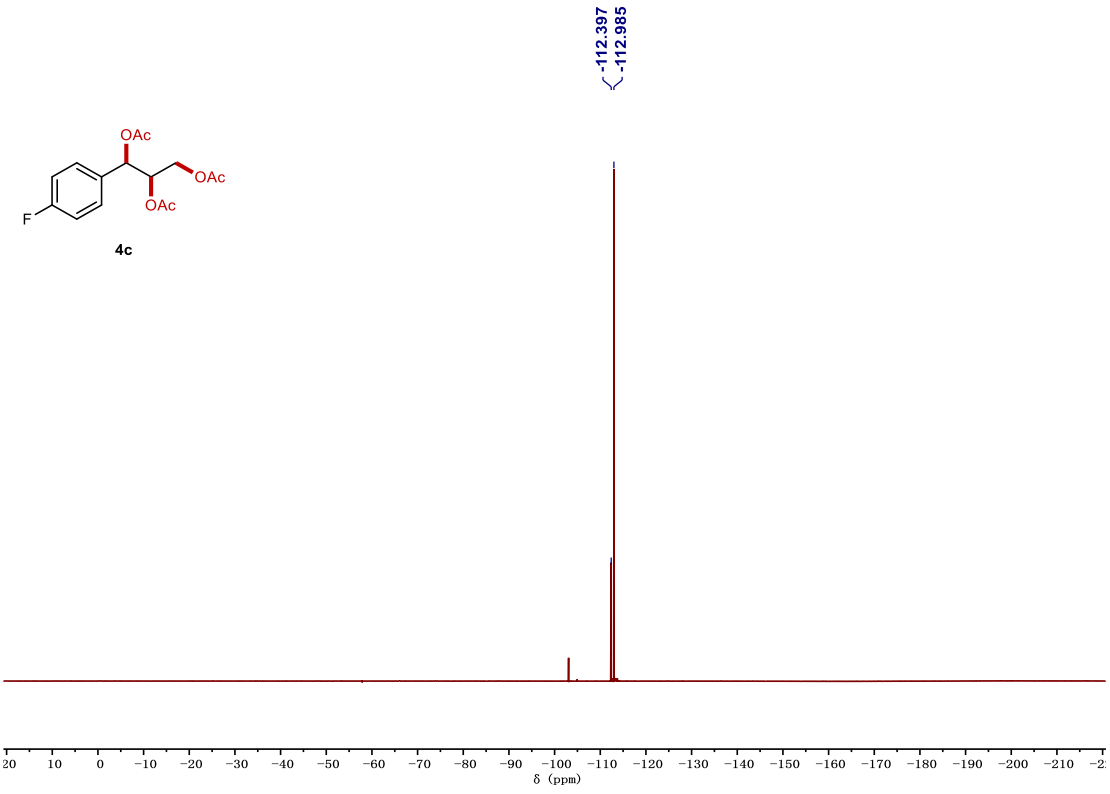

7.340
7.330
7.324
7.313
7.260 CDCl3
7.055
7.048
7.040
7.037
7.031
7.022
7.005
5.937
5.926
5.387
5.383
5.380
5.376
5.373
5.367
5.358
5.355
5.346
5.335
4.204
4.194
3.796
3.784
3.772
3.760
2.091
2.061
2.032
2.029
2.011
2.008
1.959
OAc
OAc
OAc
F

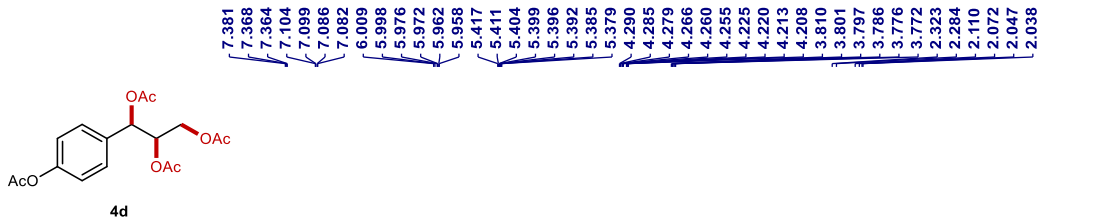
4c


2.00
1.99
1.00
1.01
1.80
0.20
2.30
0.71
0.69
0.71
2.27
2.31
δ (ppm)


170.483
169.746
169.361
163.596
161.629
131.759
131.733
128.796
128.731
115.527
115.356
77.256
77.001 CDCl3
76.746
72.409
72.162
61.594
20.835
20.614
20.574
OAc
OAc
OAc
F
δ (ppm)

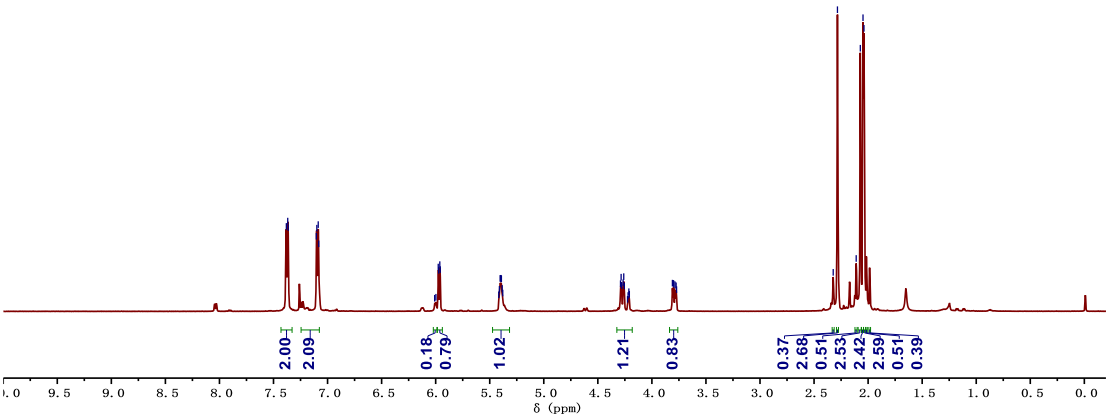
4c

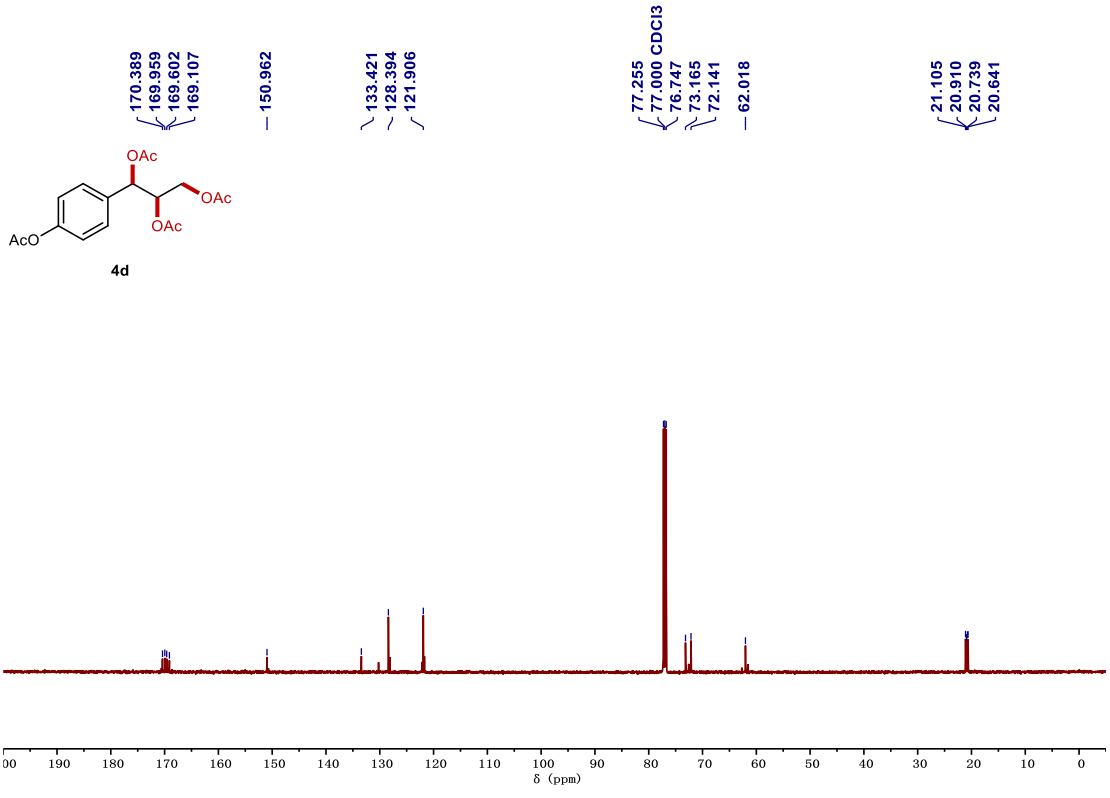

-112.397
-112.985
OAc
OAc
OAc
F
4c
20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
δ (ppm)


7.381
7.368
7.364
7.104
7.099
7.086
7.082
6.009
5.998
5.976
5.972
5.962
5.958
5.417
5.411
5.404
5.399
5.396
5.392
5.385
5.379
4.290
4.285
4.279
4.266
4.260
4.255
4.225
4.220
4.213
4.208
3.810
3.801
3.797
3.786
3.776
3.772
2.323
2.284
2.110
2.072
2.047
2.038

OAc
OAc
OAc
AcO

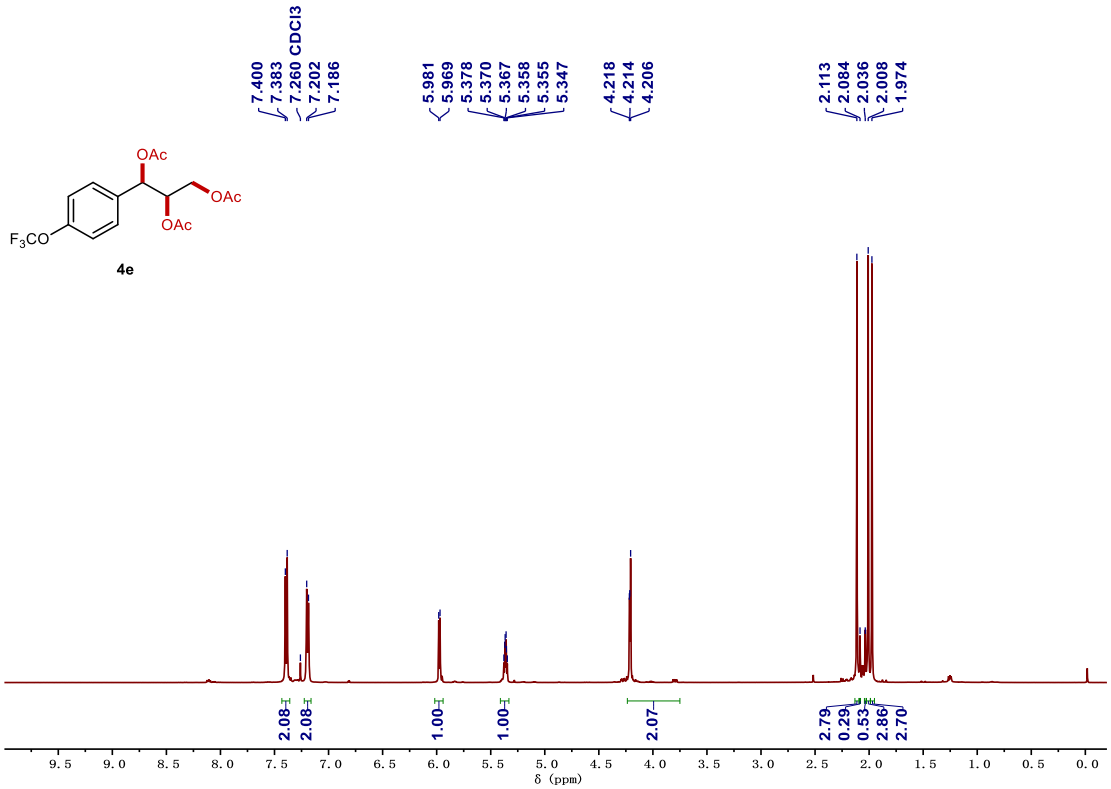
4d

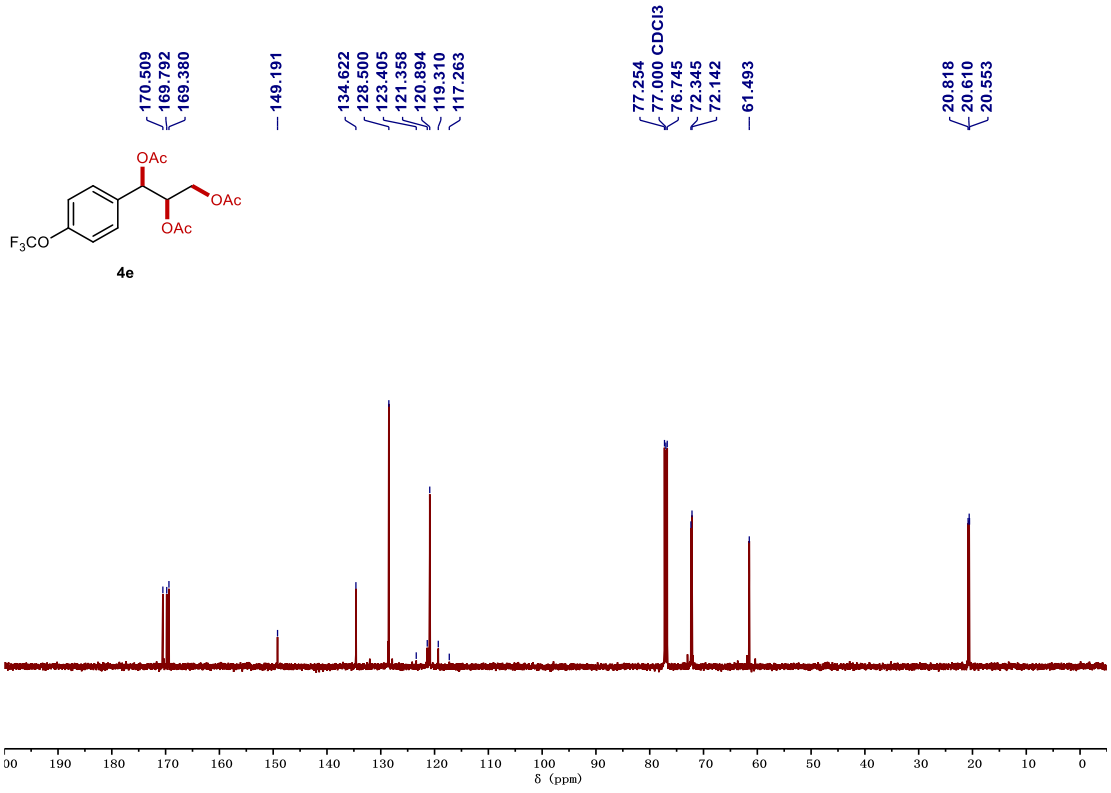

2.00
2.09
0.18
0.79
1.02
1.21
0.83
0.37
2.68
0.51
2.53
2.42
2.59
0.51
0.39
δ (ppm)

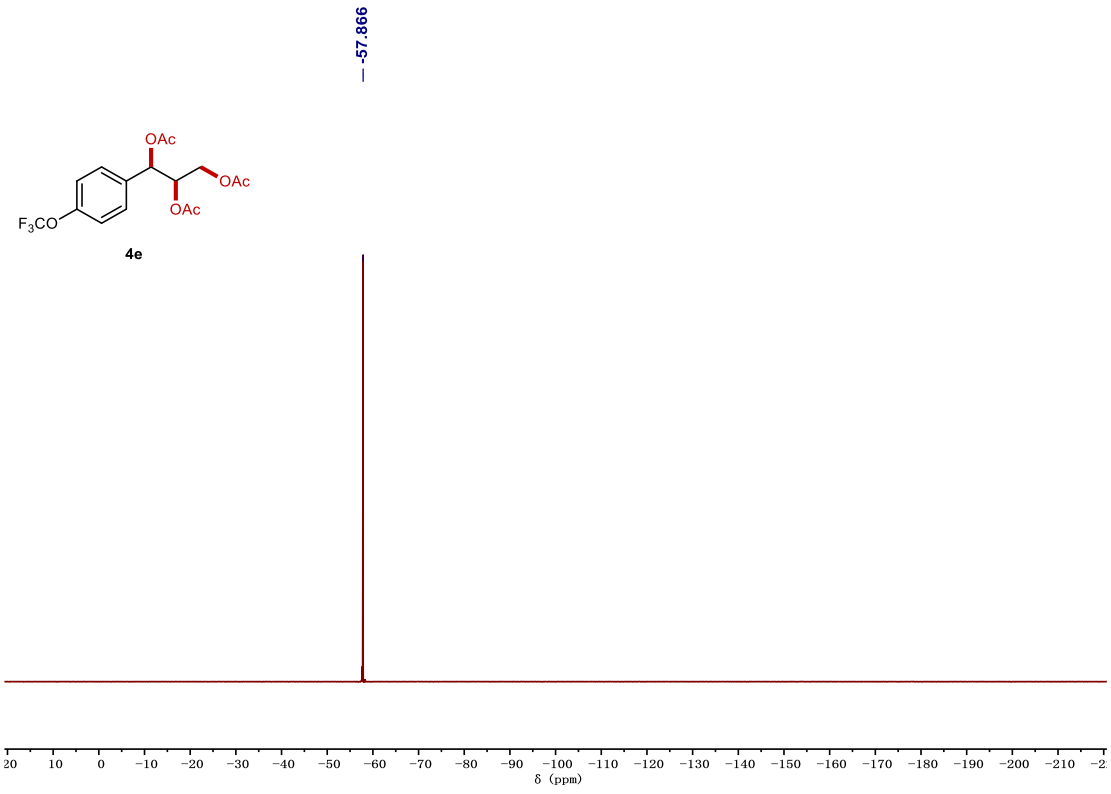

170.389
169.959
169.602
169.107
150.962
133.421
128.394
121.906
77.255
77.000 CDCl3
76.747
73.165
72.141
62.018
21.105
20.910
20.739
20.641
δ (ppm)

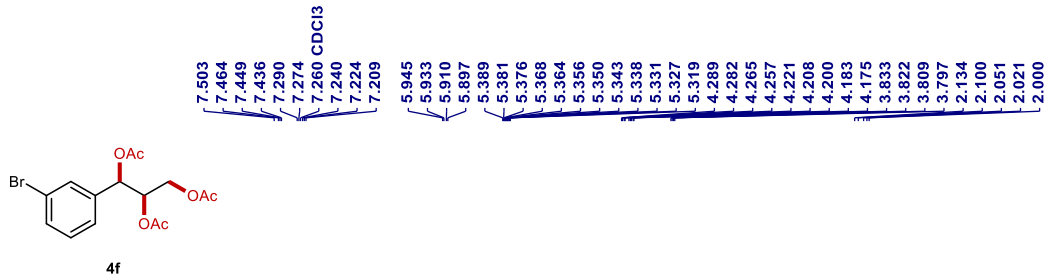
OAc
OAc
OAc
AcO

4d


7.400
7.383
7.260 CDCl3
7.202
7.186
5.981
5.969
5.378
5.370
5.367
5.358
5.355
5.347
4.218
4.214
4.206
2.113
2.084
2.036
2.008
1.974
OAc
OAc
OAc
F3CO
4e
2.08
2.08
1.00
1.00
2.07
2.79
0.29
0.53
2.86
2.70
δ (ppm)

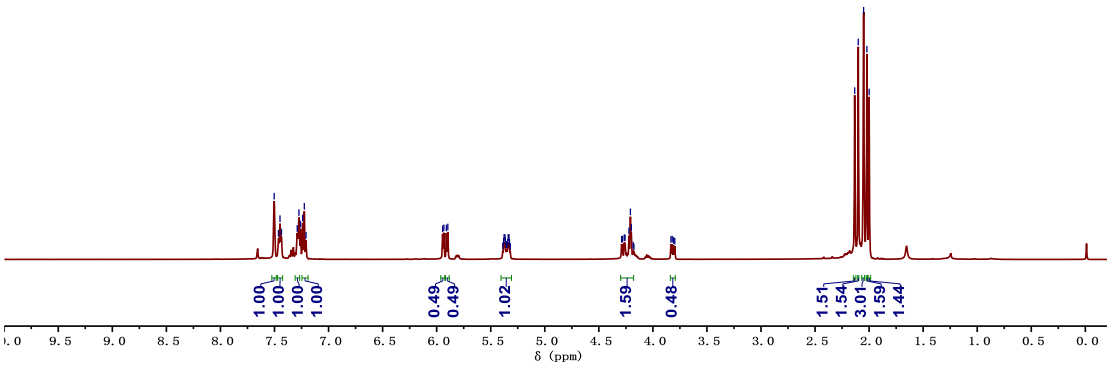

170.509
169.792
169.380
149.191
134.622
128.500
123.405
121.358
120.894
119.310
117.263
77.254
77.000 CDCl3
76.745
72.345
72.142
61.493
20.818
20.610
20.553
OAc
OAc
OAc
F3CO
4e
δ (ppm)


-57.866
OAc
OAc
OAc
F3CO
4e
20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
δ (ppm)

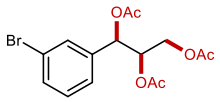

7.503
7.464
7.449
7.436
7.290
7.274
7.260 CDCl3
7.240
7.224
7.209
5.945
5.933
5.910
5.897
5.389
5.381
5.376
5.368
5.364
5.356
5.350
5.343
5.338
5.331
5.327
5.319
4.289
4.282
4.265
4.257
4.221
4.208
4.200
4.183
4.175
3.833
3.822
3.809
3.797
2.134
2.100
2.051
2.021
2.000

OAc
Br
OAc
OAc

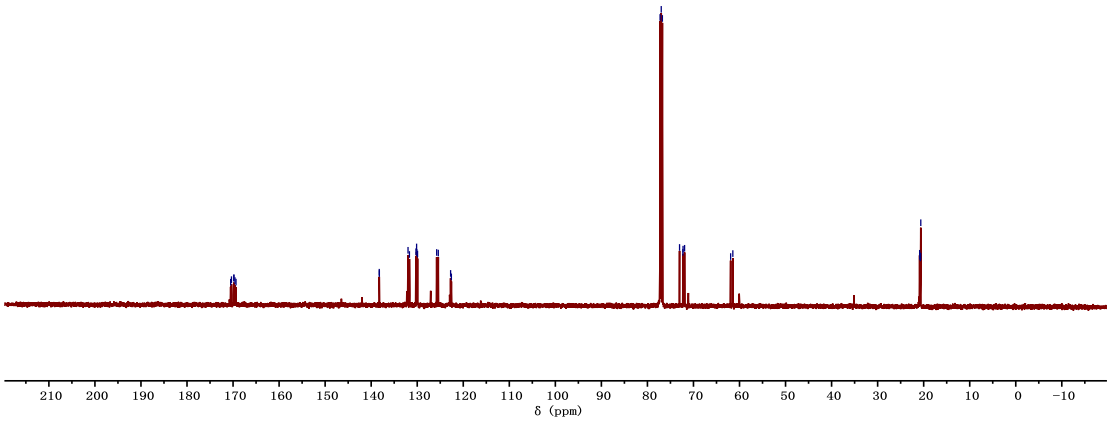
4f

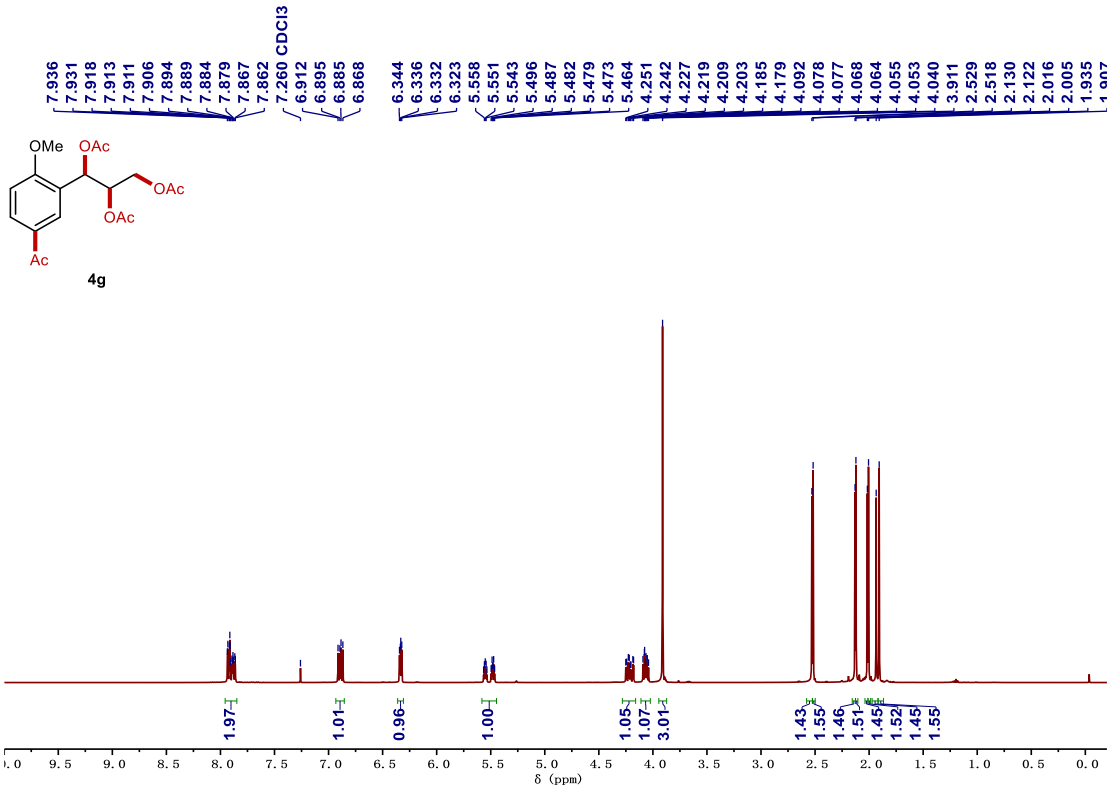

1.00
1.00
1.00
1.00
0.49
0.49
1.02
1.59
0.48
1.51
1.54
3.01
1.59
1.44
δ (ppm)

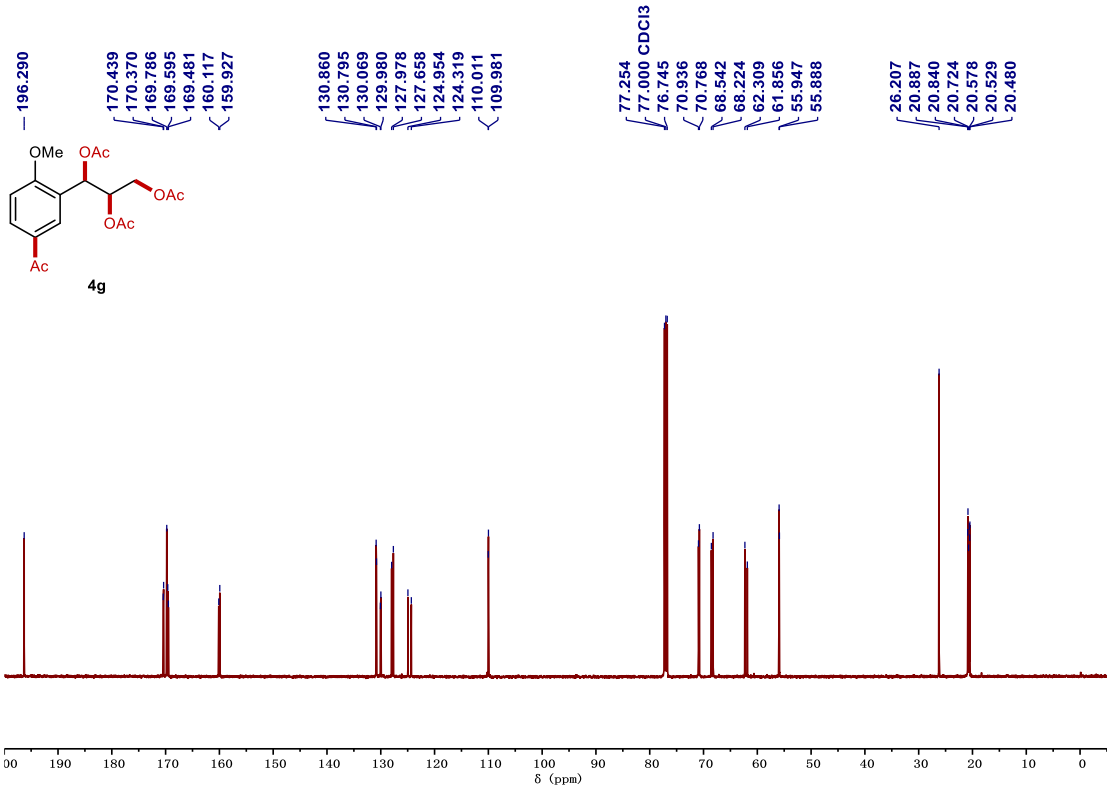

170.521
170.345
169.836
169.814
169.581
169.357
138.245
138.220
131.995
131.692
130.274
130.135
130.087
129.927
125.775
125.424
122.749
122.582
77.255
77.000 CDCl3
76.747
73.023
72.305
72.262
71.931
61.926
61.449
20.877
20.873
20.704
20.671
20.627


OAc
Br
OAc
OAc

4f


δ (ppm)


7.936
7.931
7.918
7.913
7.911
7.906
7.894
7.889
7.884
7.879
7.867
7.862
7.260 CDCl3
6.912
6.895
6.885
6.868
6.344
6.336
6.332
6.323
5.558
5.551
5.543
5.496
5.487
5.482
5.479
5.473
5.464
4.251
4.242
4.227
4.219
4.209
4.203
4.185
4.179
4.092
4.078
4.077
4.068
4.064
4.055
4.053
4.040
3.911
2.529
2.518
2.130
2.122
2.016
2.005
1.935
OMe
OAc
OAc
OAc
Ac
4g
1.97
1.01
0.96
1.00
1.05
1.07
3.01
1.43
1.55
1.46
1.51
1.45
1.52
1.45
1.55
δ (ppm)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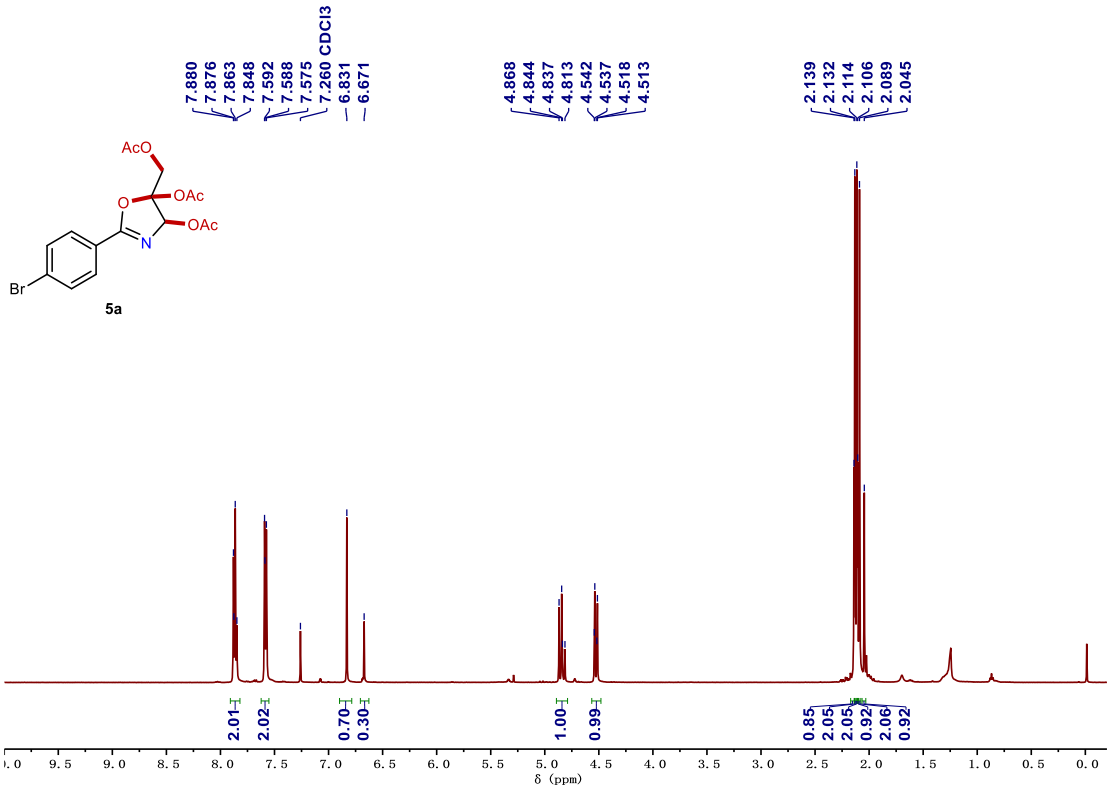
170.439
170.370
169.786
169.595
169.481
160.117
159.927
130.860
130.795
130.069
129.980
127.978
127.658
124.954
124.319
110.011
109.981
77.254
77.000 CDCl3
76.745
70.936
70.768
68.542
68.224
62.309
61.856
55.947
55.888
26.207
20.887
20.840
20.724
20.578
20.529
20.480
OMe
OAc
OAc
OAc
Ac
4g
δ (ppm)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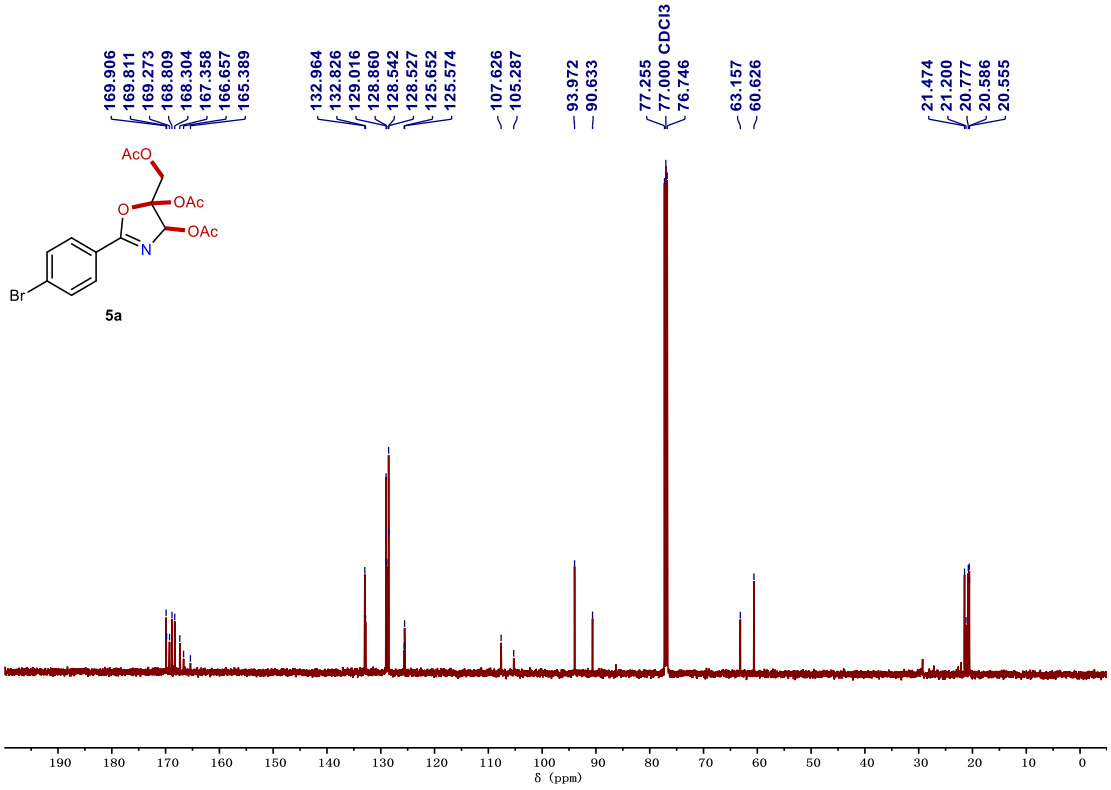
7.876
7.863
7.848
7.592
7.588
7.575
7.260 CDCl3
6.831
6.671
4.868
4.844
4.837
4.813
4.542
4.537
4.518
4.513
2.139
2.132
2.114
2.106
2.089
2.045
AcO
OAc
OAc
O
N
Br
5a
2.01
2.02
0.70
0.30
1.00
0.99
0.85
2.05
2.05
0.92
2.06
0.92
δ (ppm)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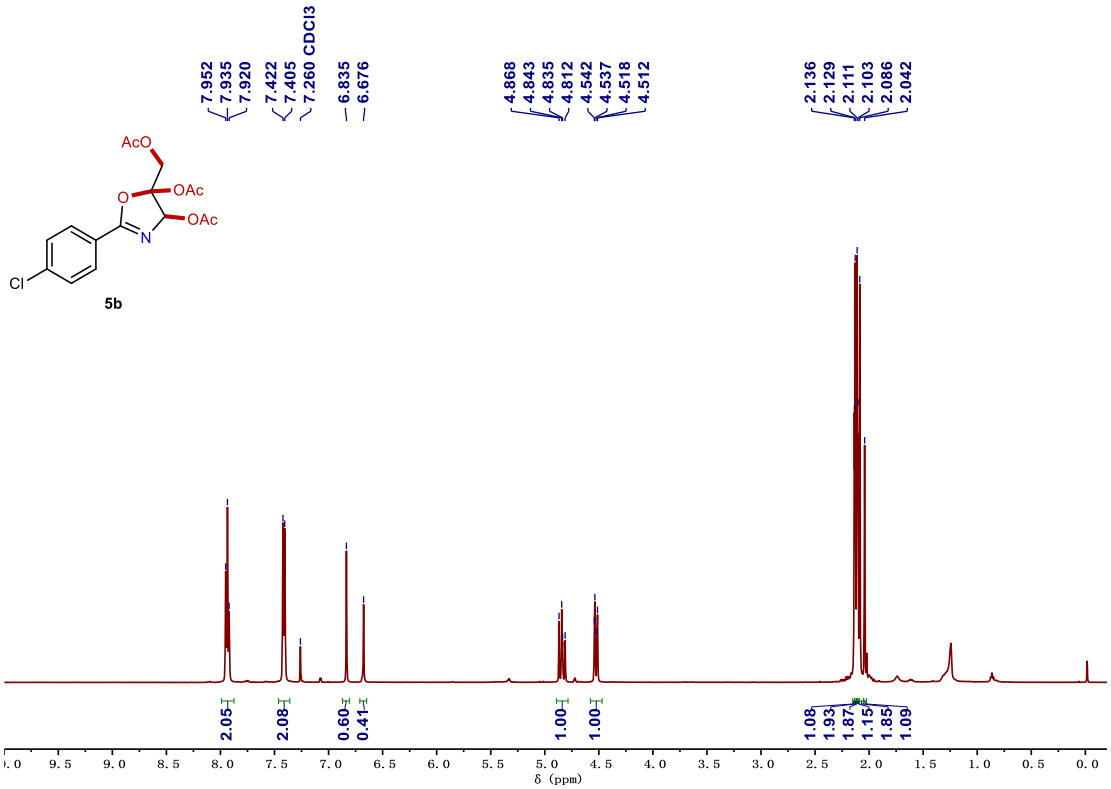
169.811
169.273
168.809
168.304
167.358
166.657
165.389
132.964
132.826
129.016
128.860
128.542
128.527
125.652
125.574
107.626
105.287
93.972
90.633
77.255
77.000 CDCl3
76.746
63.157
60.626
21.474
21.200
20.777
20.586
20.555
AcO
OAc
OAc
O
N
Br
5a
δ (ppm)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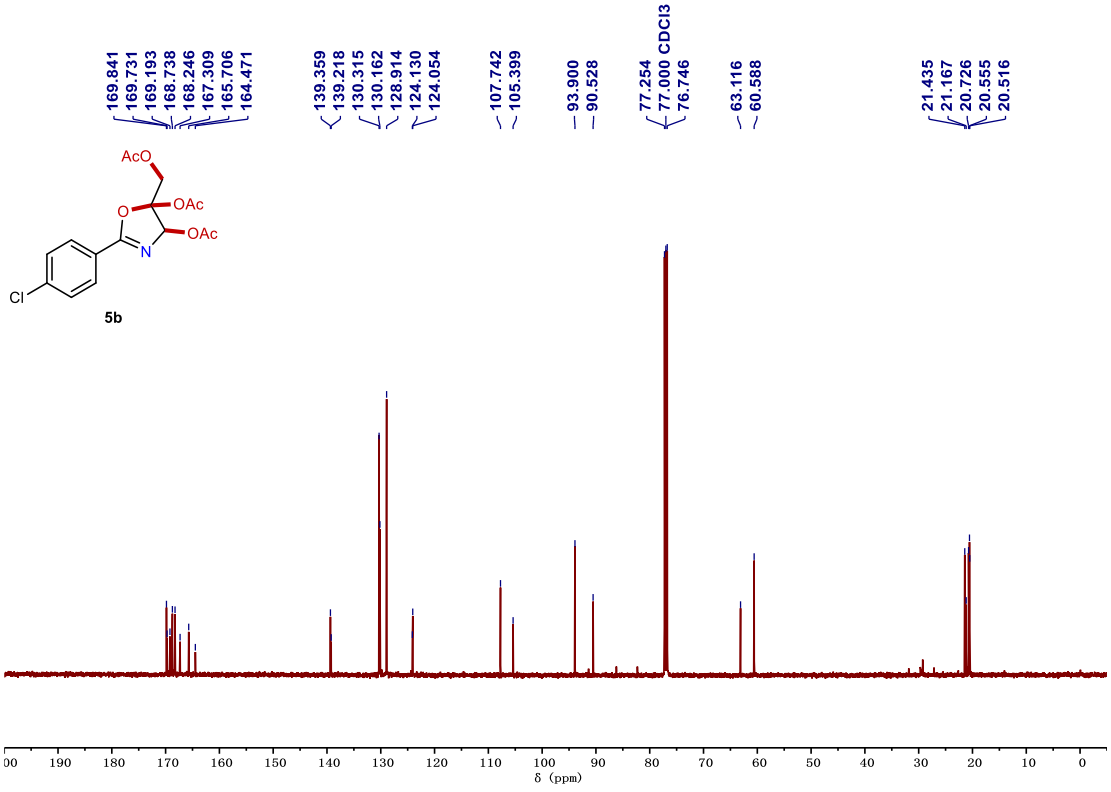
7.935
7.920
7.422
7.405
7.260 CDCl3
6.835
6.676
4.868
4.843
4.835
4.812
4.542
4.537
4.518
4.512
2.136
2.129
2.111
2.103
2.086
2.042
AcO
OAc
OAc
O
N
Cl
5b
2.05
2.08
0.60
0.41
1.00
1.00
1.08
1.93
1.87
1.15
1.85
1.09
δ (ppm)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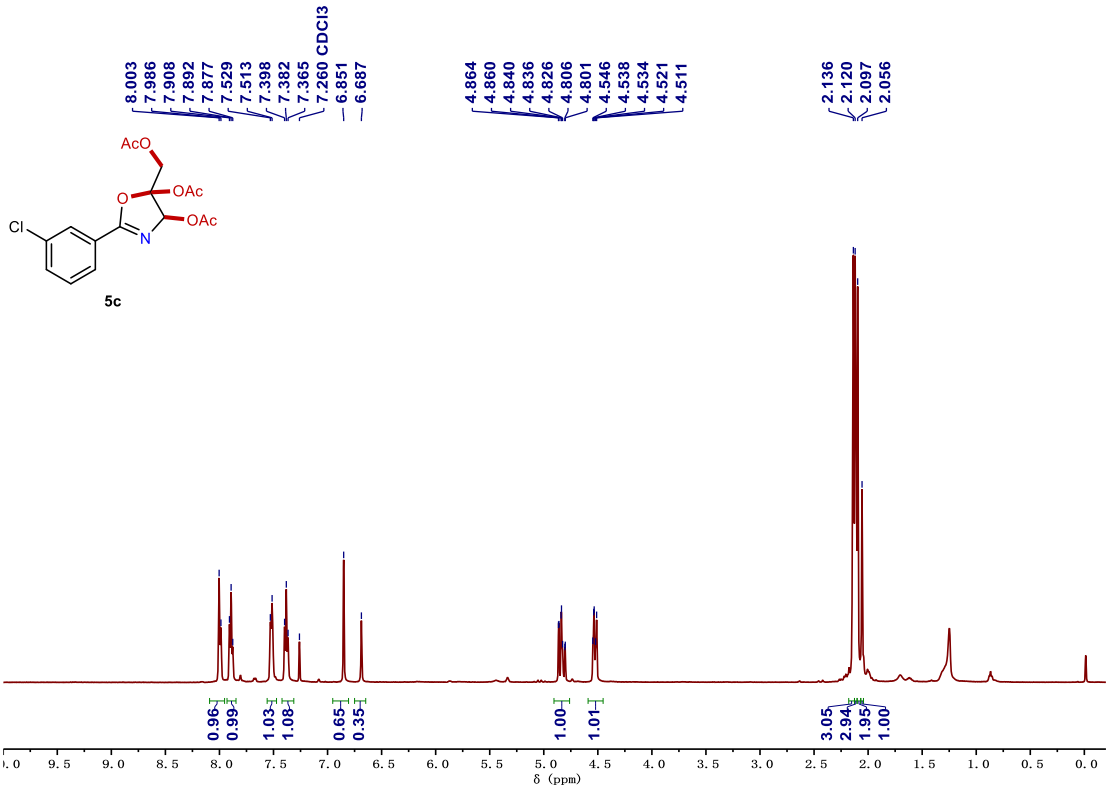
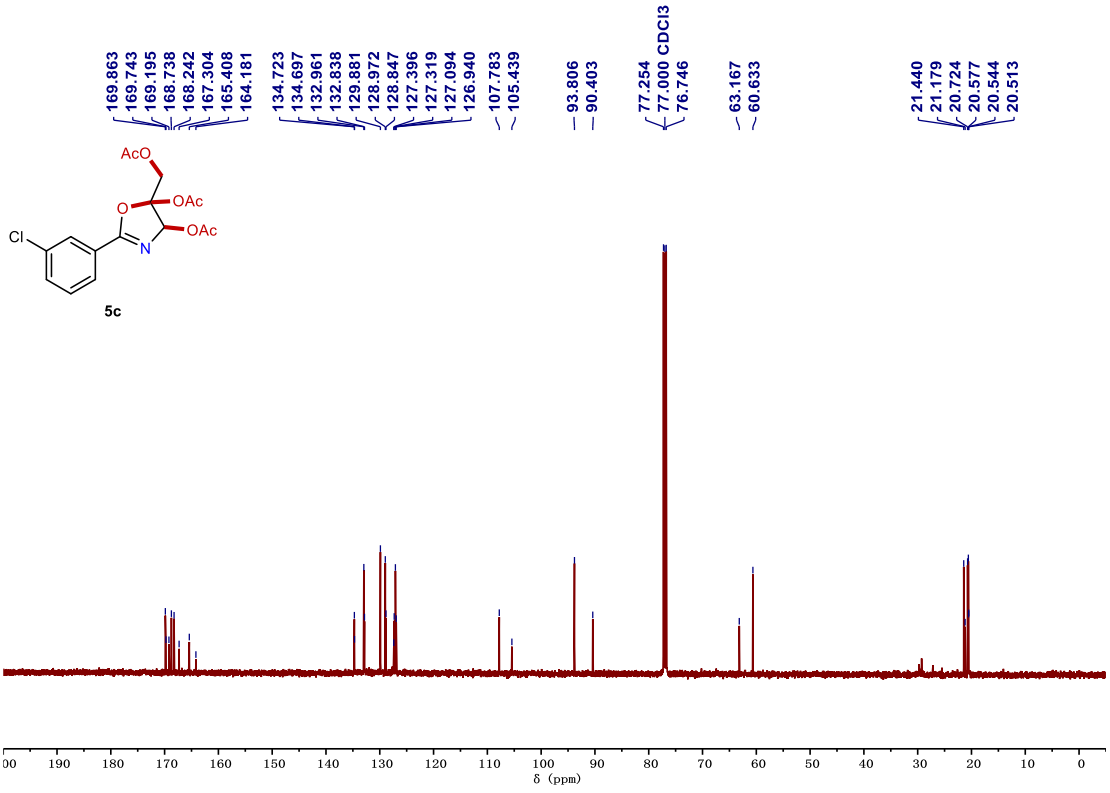
169.731
169.193
168.738
168.246
167.309
165.706
164.471
139.359
139.218
130.315
130.162
128.914
124.130
124.054
107.742
105.399
93.900
90.528
77.254
77.000 CDCl3
76.746
63.116
60.588
21.435
21.167
20.726
20.555
20.516
AcO
OAc
OAc
O
N
Cl
5b
δ (ppm)


8.003
7.986
7.908
7.892
7.877
7.529
7.513
7.398
7.382
7.365
7.260 CDCl3
6.851
6.687
4.864
4.860
4.840
4.836
4.826
4.806
4.801
4.546
4.538
4.534
4.521
4.511
2.136
2.120
2.097
2.056
AcO
OAc
OAc
Cl
O
N
5c
0.96
0.99
1.03
1.08
0.65
0.35
1.00
1.01
3.05
2.94
1.95
1.00
δ (ppm)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169.195
168.738
168.242
167.304
165.408
164.181
134.723
134.697
132.961
132.838
129.881
128.972
128.847
127.396
127.319
127.094
126.940
107.783
105.439
93.806
90.403
77.254
77.000 CDCl3
76.746
63.167
60.633
21.440
21.179
20.724
20.577
20.544
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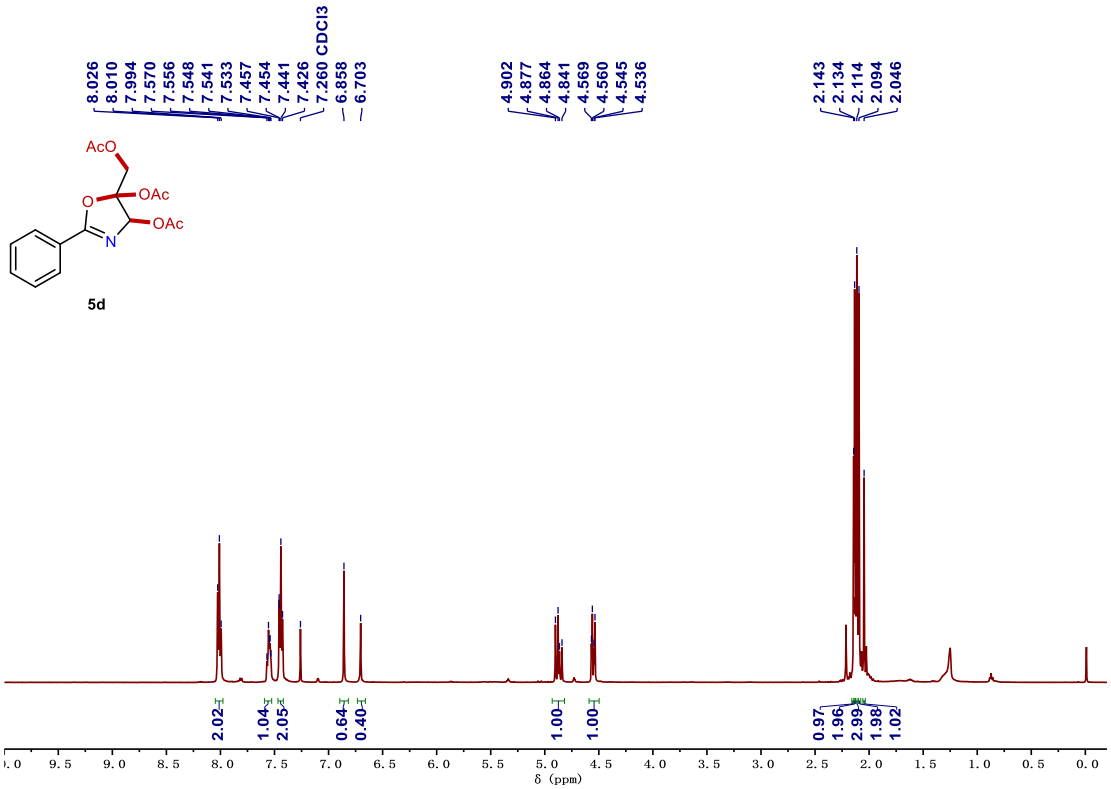
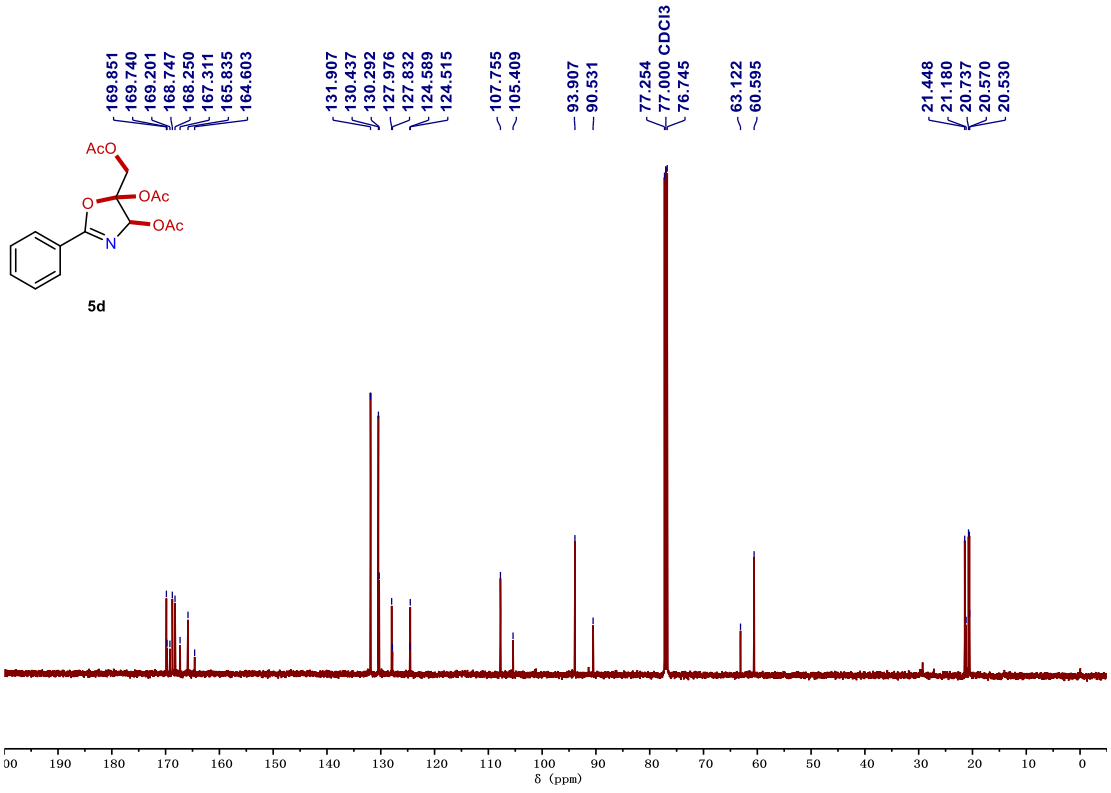
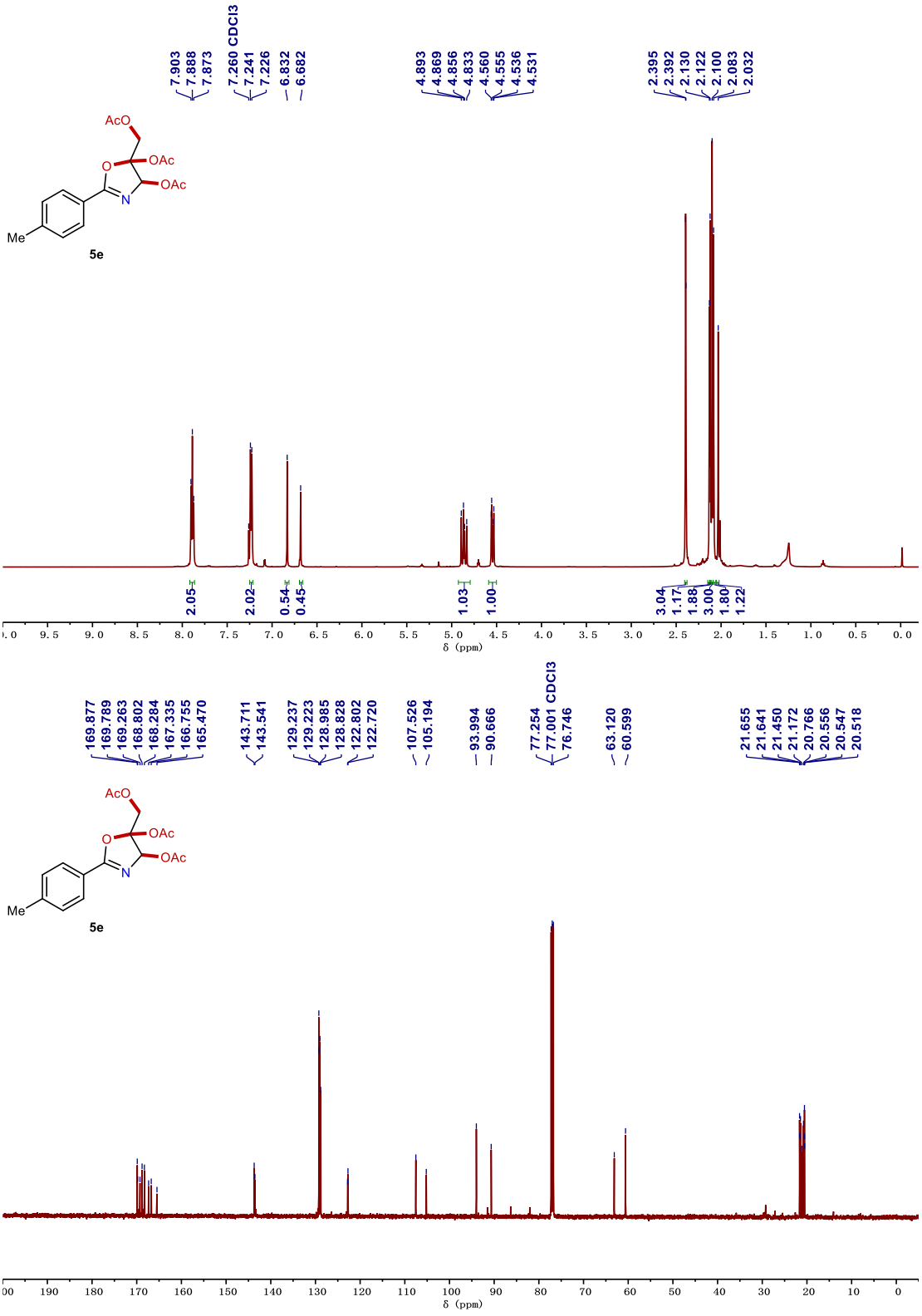
AcO
OAc
OAc
Cl
O
N
5c
δ (ppm)


8.026
8.010
7.994
7.570
7.556
7.548
7.541
7.533
7.457
7.454
7.441
7.426
7.260 CDCl3
6.858
6.703
4.902
4.877
4.864
4.841
4.569
4.560
4.545
4.536
2.143
2.134
2.114
2.094
2.046
AcO
OAc
OAc
O
N
5d
2.02
1.04
2.05
0.64
0.40
1.00
1.00
0.97
1.96
2.99
1.98
1.02
δ (ppm)


169.851
169.740
169.201
168.747
168.250
167.311
165.835
164.603
131.907
130.437
130.292
127.976
127.832
124.589
124.515
107.755
105.409
93.907
90.531
77.254
77.000 CDCl3
76.745
63.122
60.595
21.448
21.180
20.737
20.570
20.530
AcO
OAc
OAc
O
N
5d
δ (ppm)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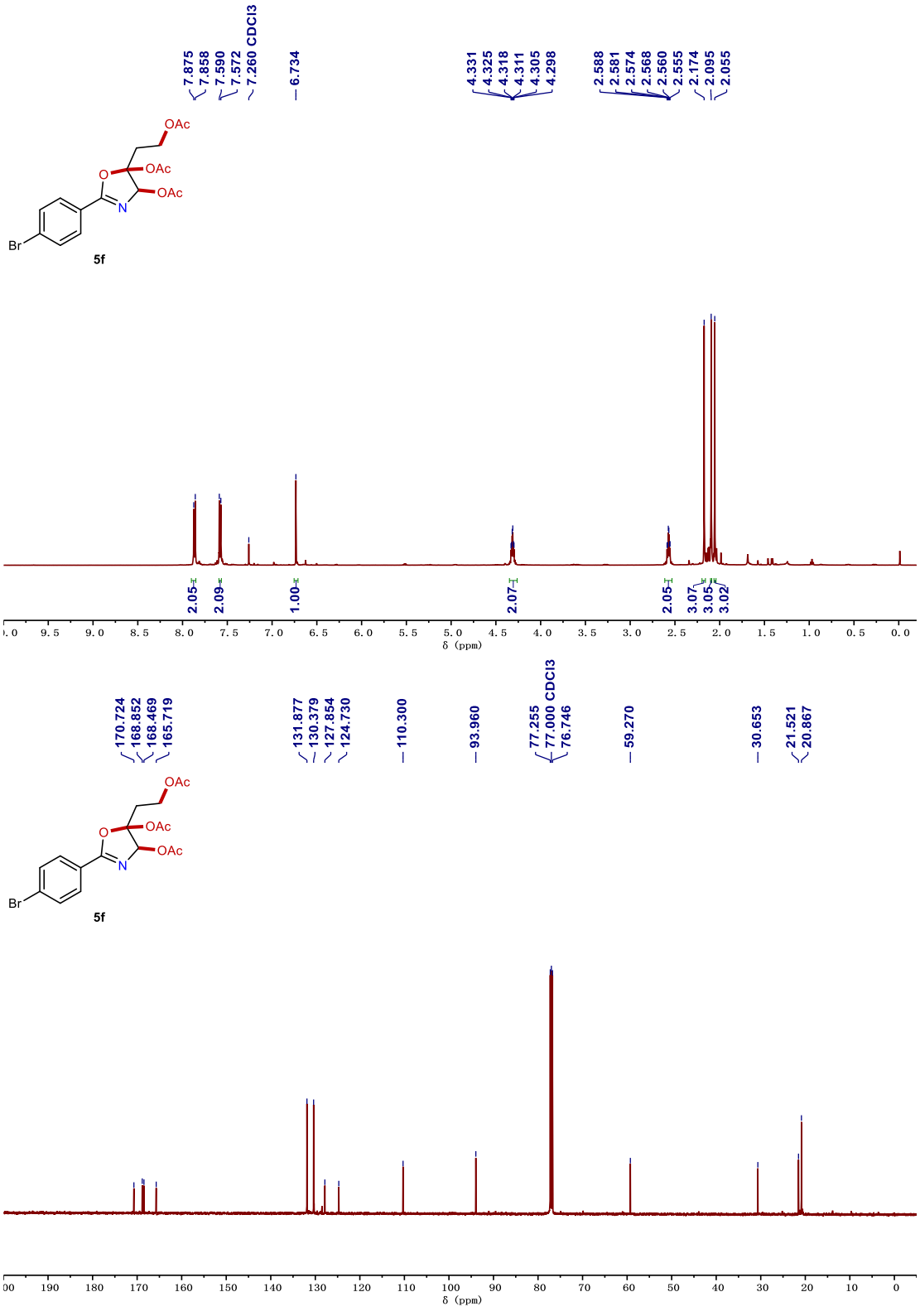
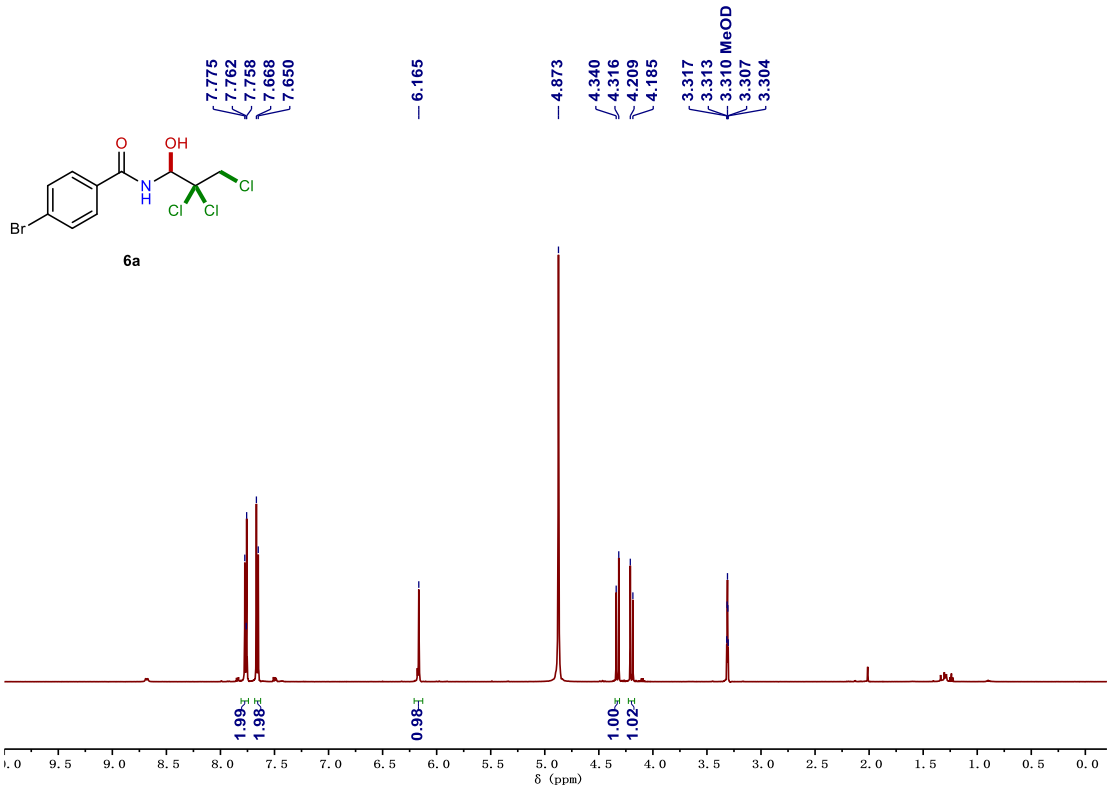
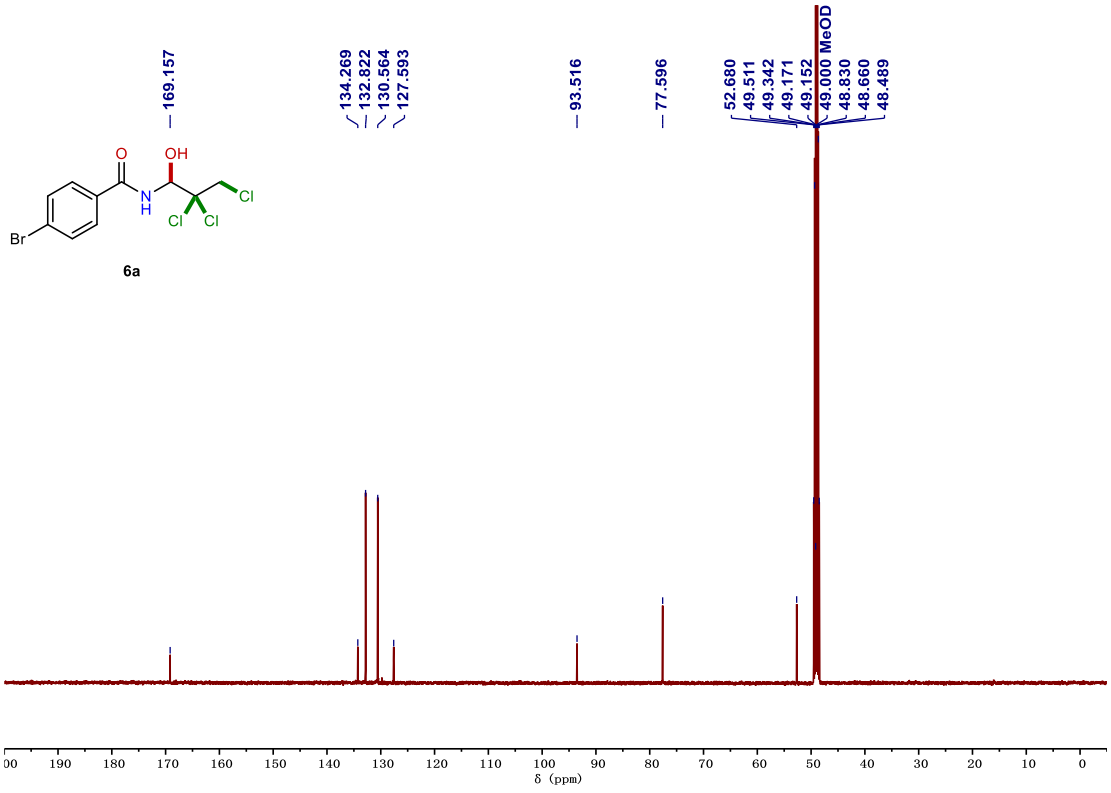
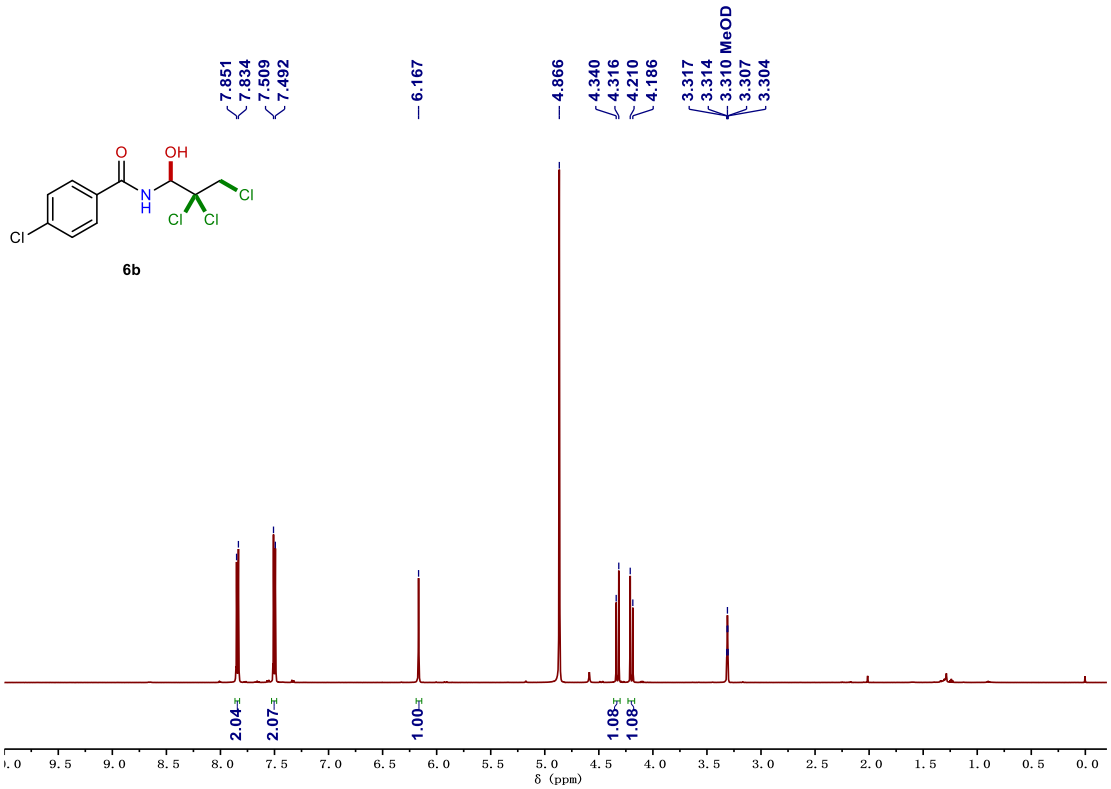
7.888
7.873
7.260 CDCl3
7.241
7.226
6.832
6.682
4.893
4.869
4.856
4.833
4.560
4.555
4.536
4.531
2.395
2.392
2.130
2.122
2.100
2.083
2.032
AcO
OAc
OAc
O
N
Me
5e
2.05
2.02
0.54
0.45
1.03
1.00
3.04
1.17
1.88
3.00
1.80
1.22
δ (ppm)
169.877
169.789
169.263
168.802
168.284
167.335
166.755
165.470
143.711
143.541
129.237
129.223
128.985
128.828
122.802
122.720
107.526
105.194
93.994
90.666
77.254
77.001 CDCl3
76.746
63.120
60.599
21.655
21.641
21.450
21.172
20.766
20.556
20.547
20.518
δ (ppm)


7.875
7.858
7.590
7.572
7.260 CDCl3
6.734
4.331
4.325
4.318
4.311
4.305
4.298
2.588
2.581
2.574
2.568
2.560
2.555
2.174
2.095
2.055
OAc
OAc
OAc
O
N
Br
5f
2.05
2.09
1.00
2.07
2.05
3.07
3.05
3.02
δ (ppm)
170.724
168.852
168.469
165.719
131.877
130.379
127.854
124.730
110.300
93.960
77.255
77.000 CDCl3
76.746
59.270
30.653
21.521
20.867
δ (ppm)


7.775
7.762
7.758
7.668
7.650
6.165
4.873
4.340
4.316
4.209
4.185
3.317
3.313
3.310 MeOD
3.307
3.304
O
OH
N
H
Cl
Cl
Cl
Br
6a
1.99
1.98
0.98
1.00
1.02
δ (ppm)


169.157
134.269
132.822
130.564
127.593
93.516
77.596
52.680
49.511
49.342
49.171
49.152
49.000 MeOD
48.830
48.660
48.489
O
OH
N
H
Cl
Cl
Cl
Br
6a
δ (ppm)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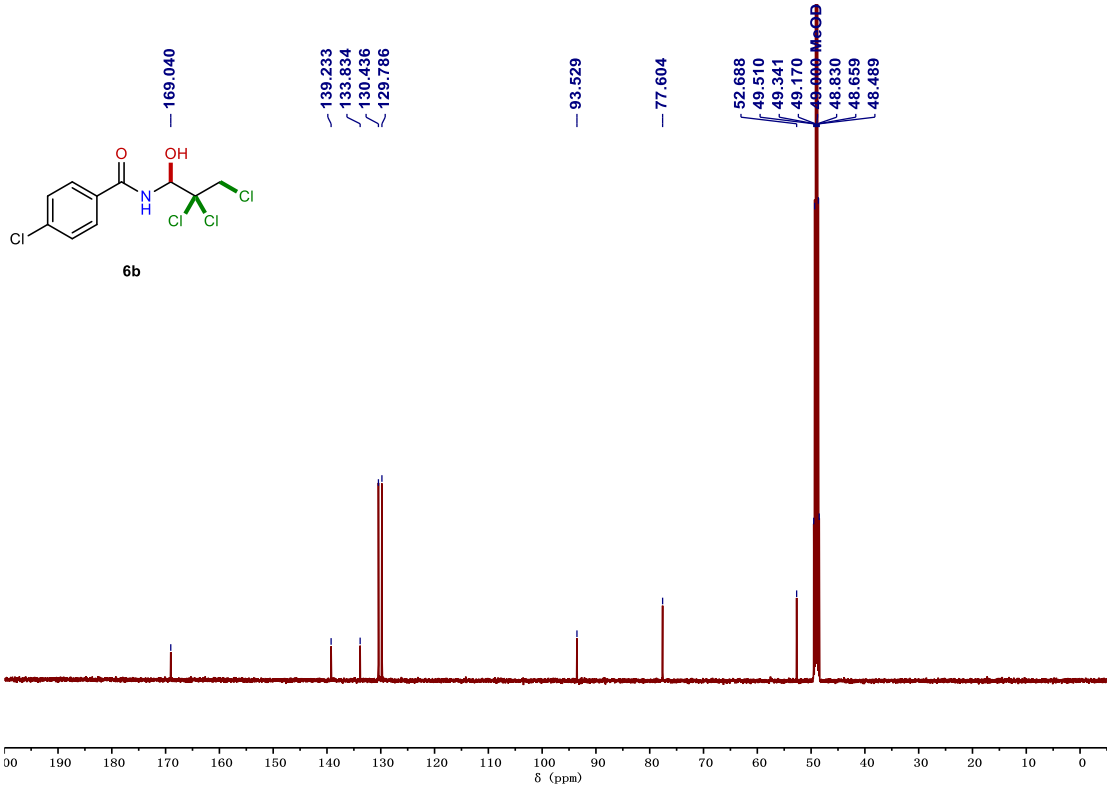
7.834
7.509
7.492
6.167
4.866
4.340
4.316
4.210
4.186
3.317
3.314
3.310 MeOD
3.307
3.304
O
OH
N
H
Cl
Cl
Cl
Cl
6b
2.04
2.07
1.00
1.08
1.08
δ (ppm)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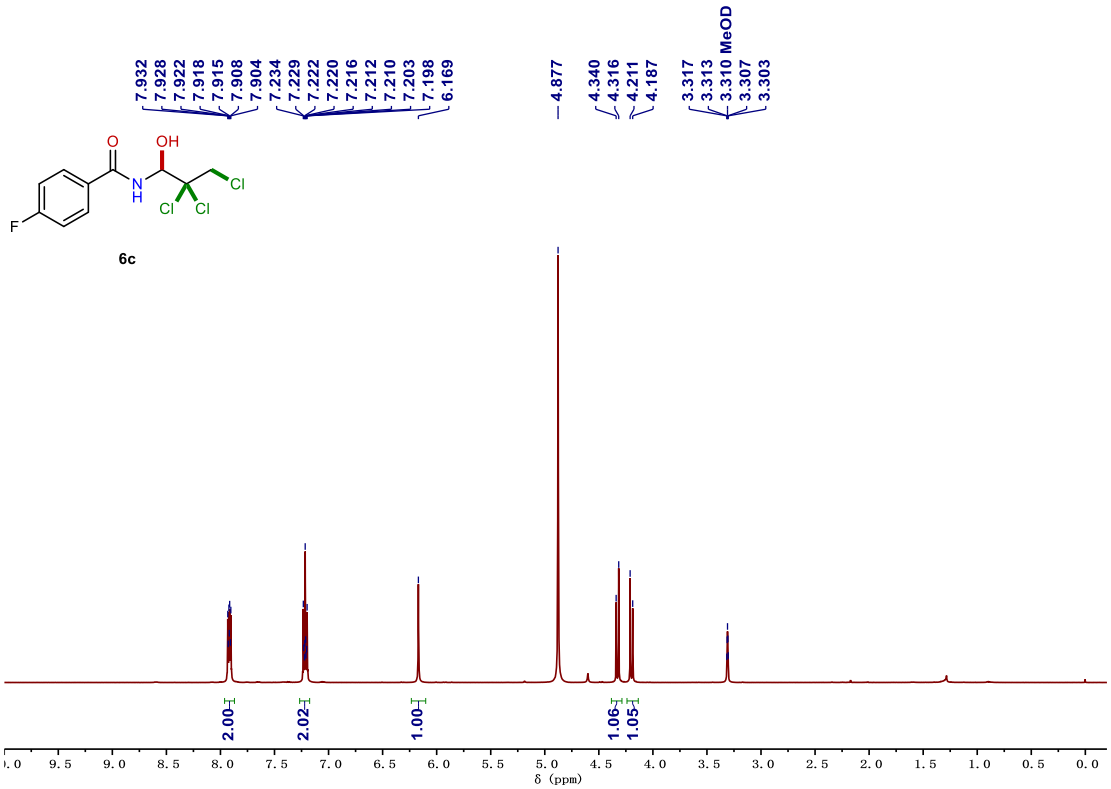
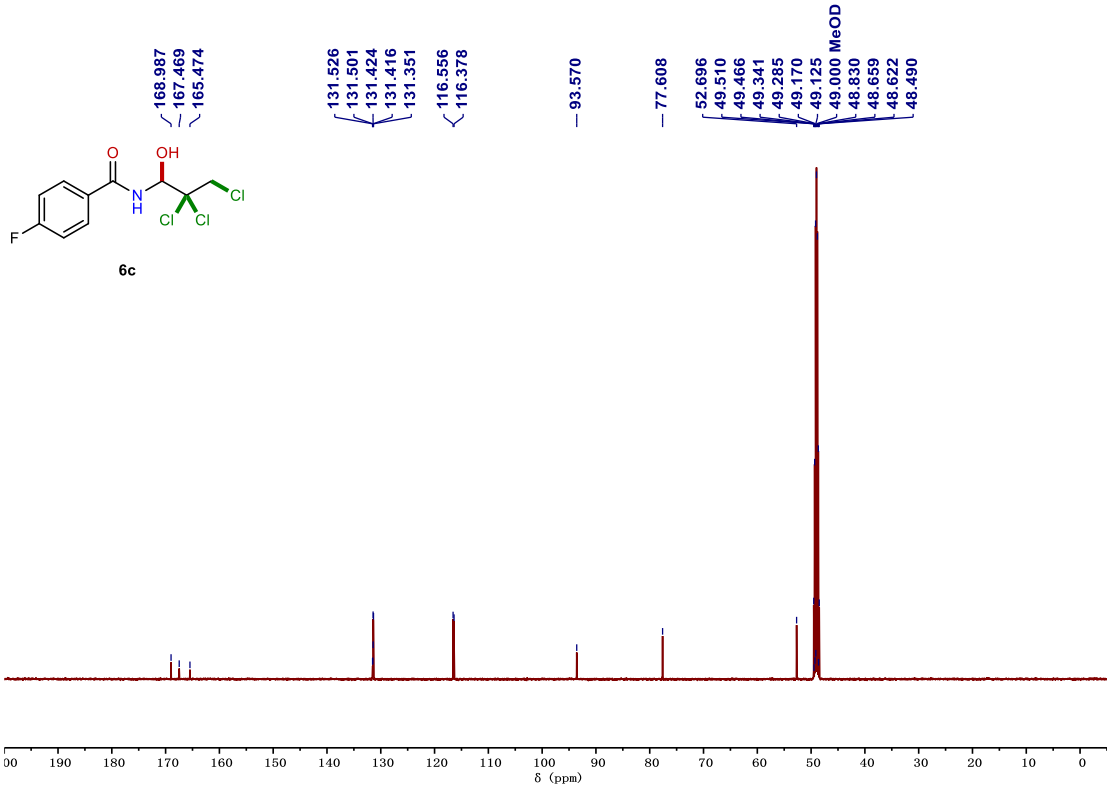
139.233
133.834
130.436
129.786
93.529
77.604
52.688
49.510
49.341
49.170
49.000 MeOD
48.830
48.659
48.489
O
OH
N
H
Cl
Cl
Cl
Cl
6b
δ (ppm)


7.932
7.928
7.922
7.918
7.915
7.908
7.904
7.234
7.229
7.222
7.220
7.216
7.212
7.210
7.203
7.198
6.169
4.877
4.340
4.316
4.211
4.187
3.317
3.313
3.310 MeOD
3.307
3.303
O
OH
N
H
Cl
Cl
Cl
F
6c
2.00
2.02
1.00
1.06
1.05
δ (ppm)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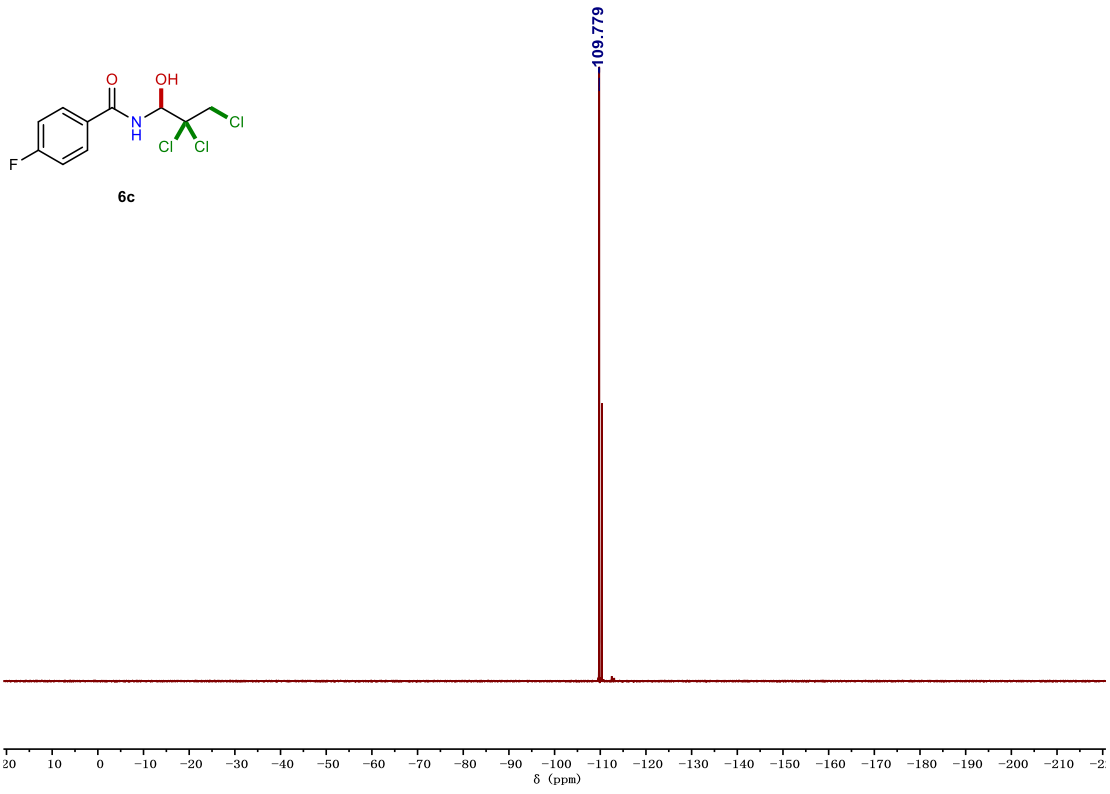
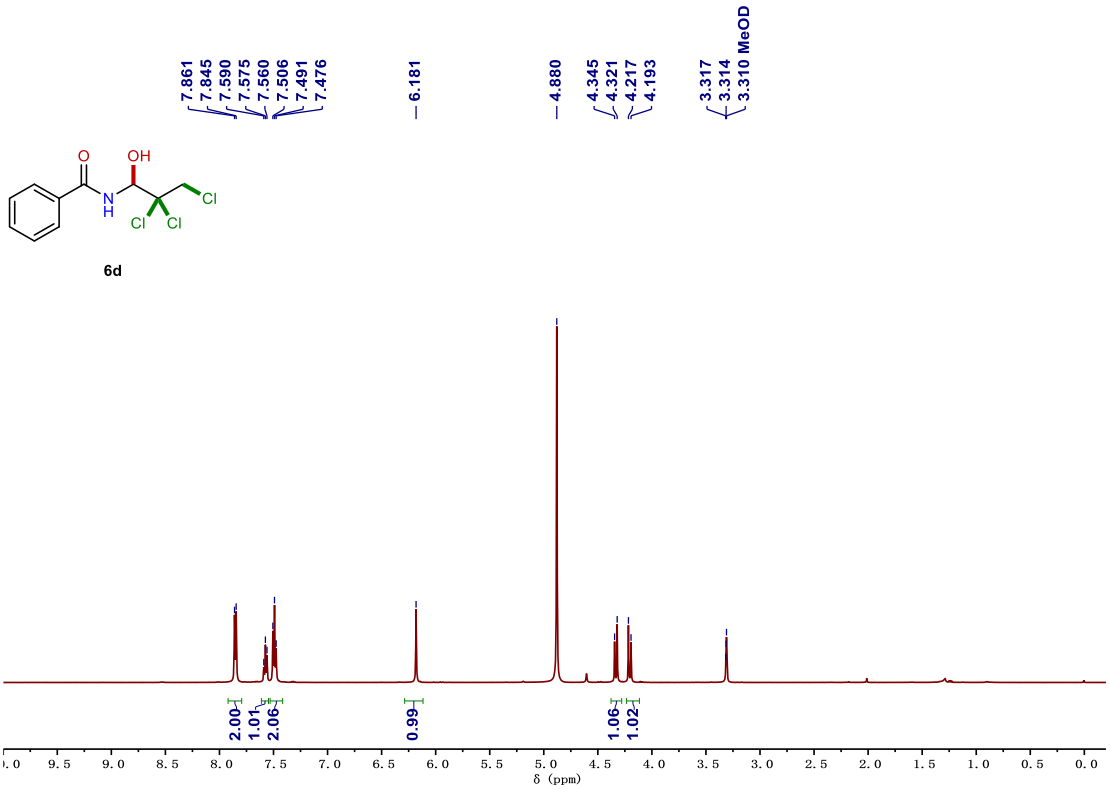
165.474
131.526
131.501
131.424
131.416
131.351
116.556
116.378
93.570
77.608
52.696
49.510
49.466
49.341
49.285
49.170
49.125
49.000 MeOD
48.830
48.659
48.622
48.490
O
OH
N
H
Cl
Cl
Cl
F
6c
δ (ppm)


O
OH
N
H
Cl
Cl
Cl
F
109.779
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)

6c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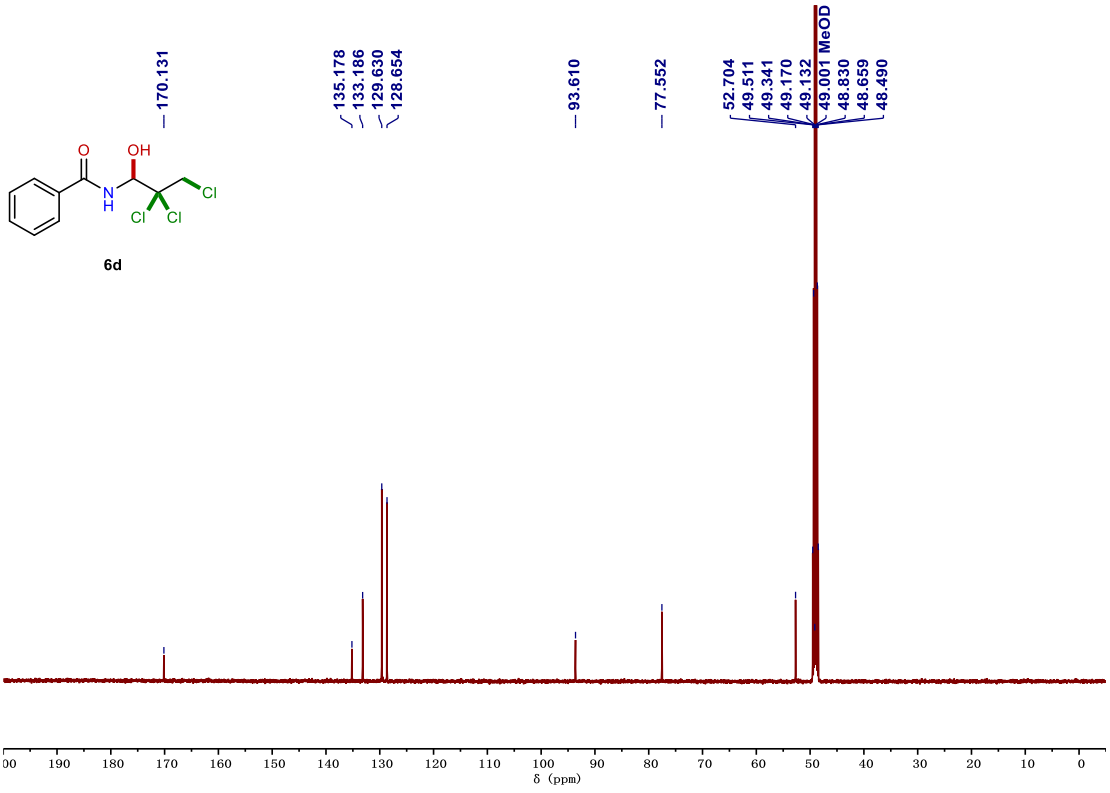
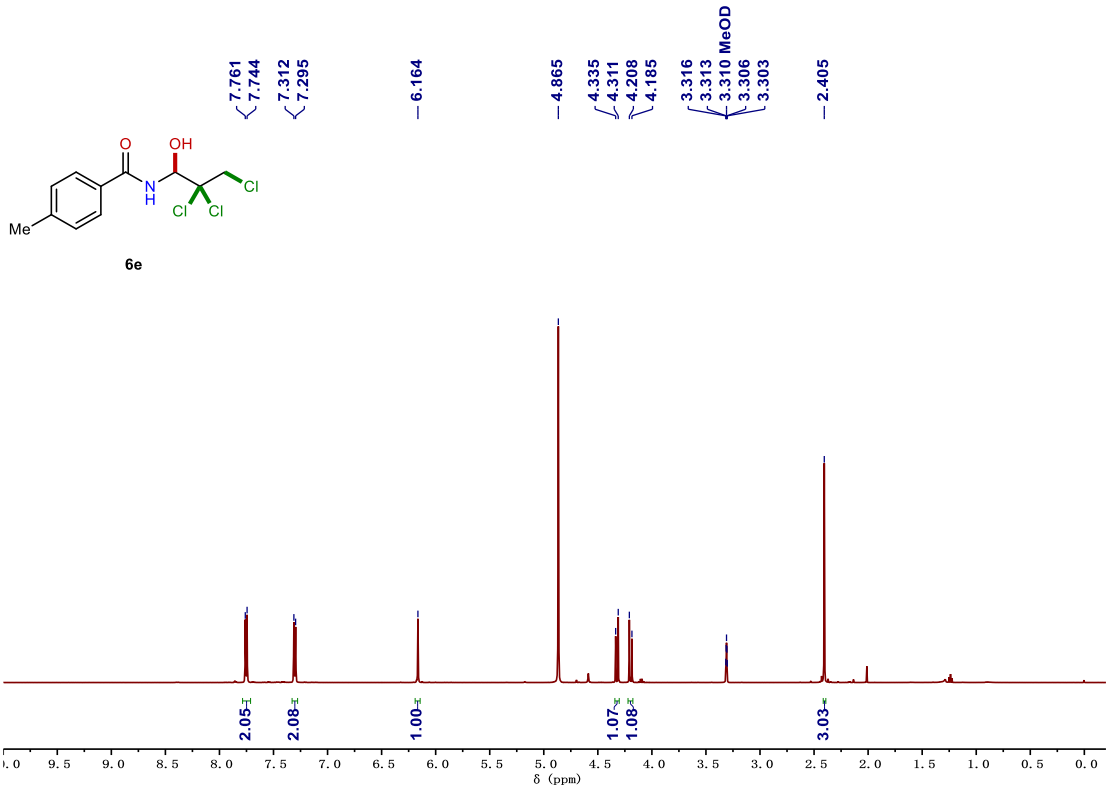
7.845
7.590
7.575
7.560
7.506
7.491
7.476
6.181
4.880
4.345
4.321
4.217
4.193
3.317
3.314
3.310 MeOD
O
OH
N
H
Cl
Cl
Cl
6d
2.00
1.01
2.06
0.99
1.06
1.02
δ (ppm)


170.131
135.178
133.186
129.630
128.654
93.610
77.552
52.704
49.511
49.341
49.170
49.132
49.001 MeOD
48.830
48.659
48.490
O
OH
N
H
Cl
Cl
Cl
6d
δ (ppm)


7.761
7.744
7.312
7.295
6.164
4.865
4.335
4.311
4.208
4.185
3.316
3.313
3.310 MeOD
3.306
3.303
2.405
O
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Me
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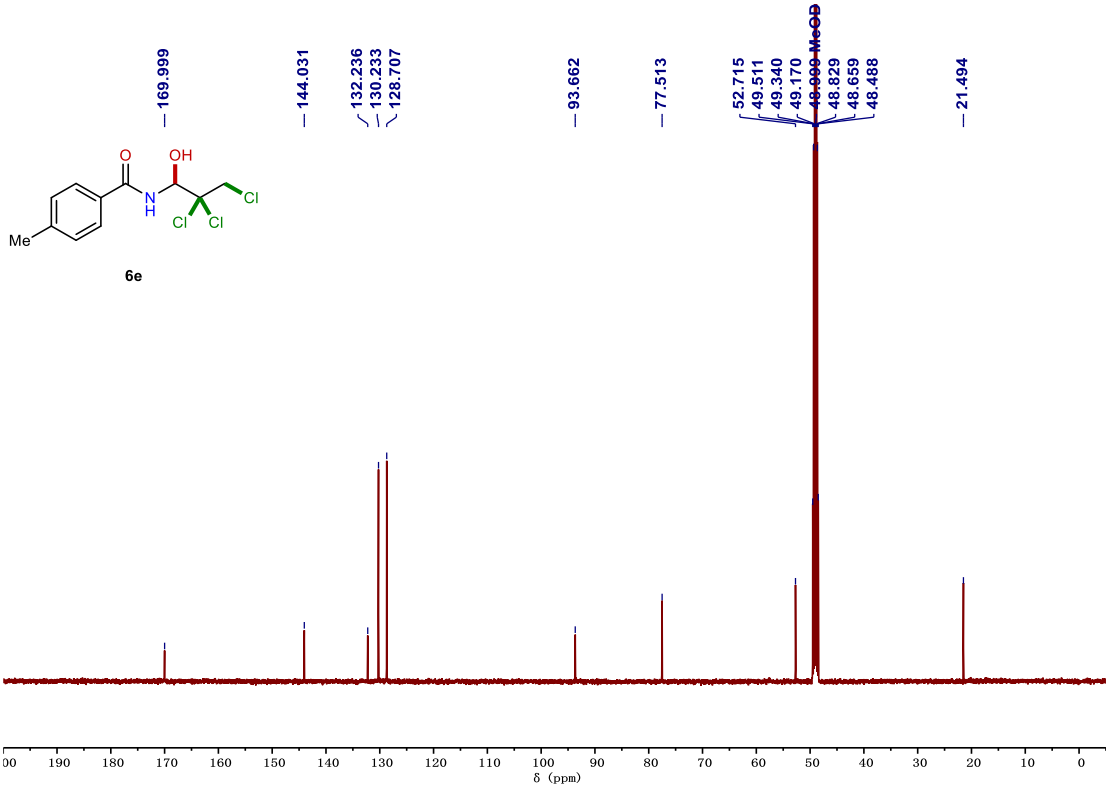
1.00
1.07
1.08
3.03
δ (ppm)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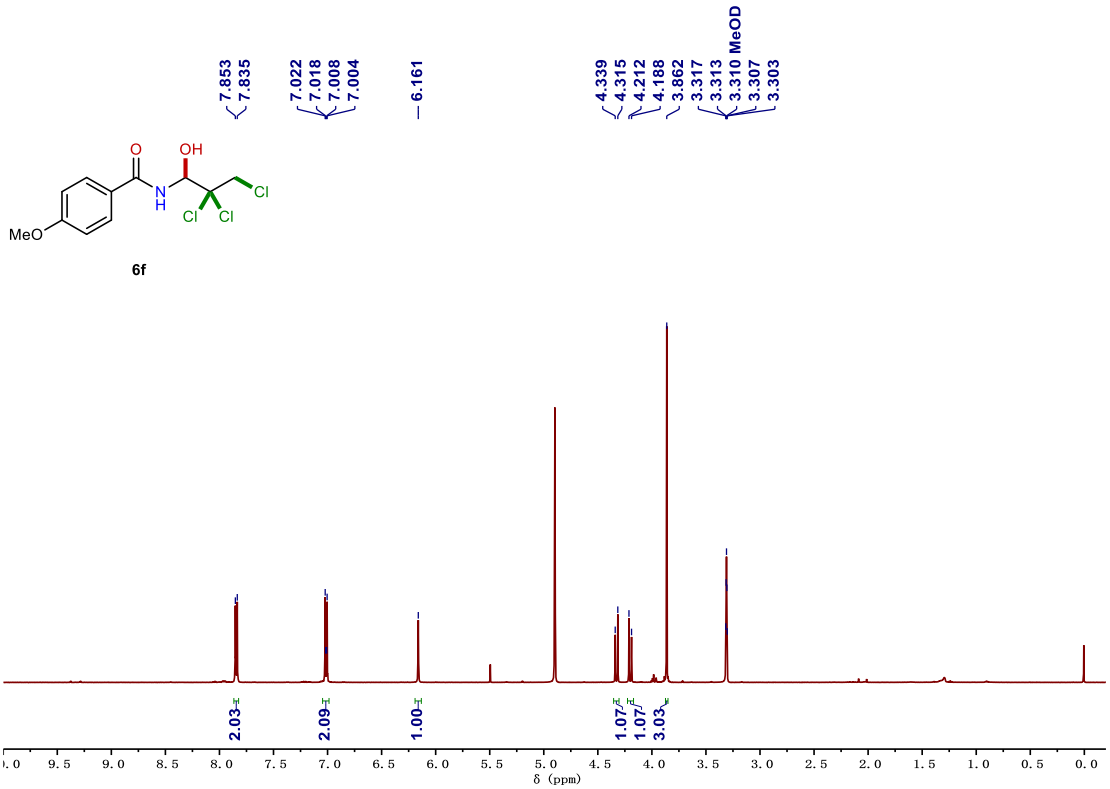
144.031
132.236
130.233
128.707
93.662
77.513
52.715
49.511
49.340
49.170
48.990 MeOD
48.829
48.659
48.488
21.494
O
OH
N
H
Cl
Cl
Cl
Me
6e
δ (ppm)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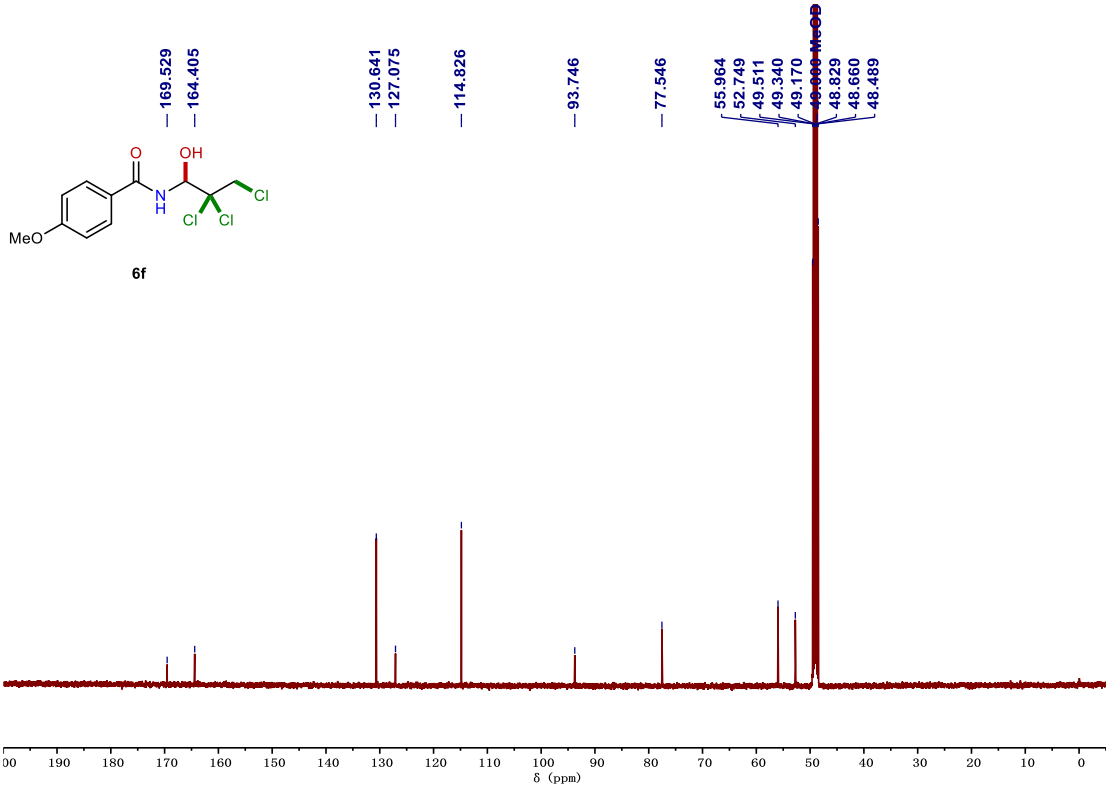
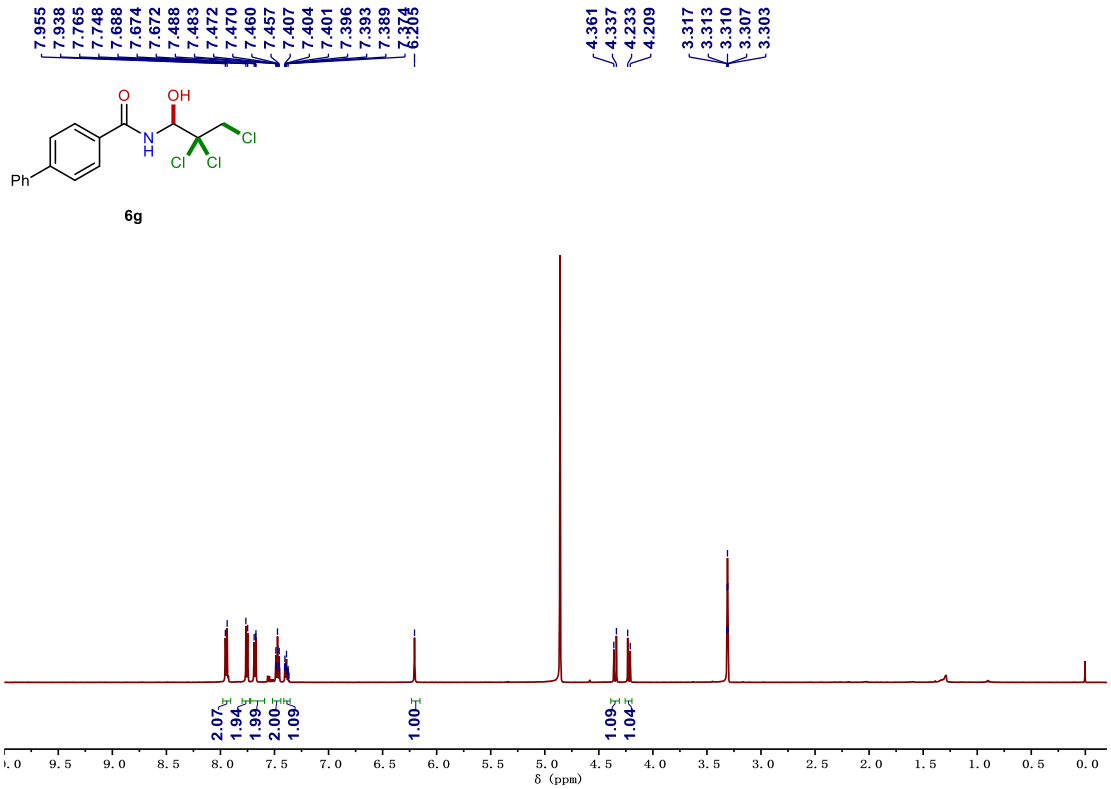
7.018
7.008
7.004
6.161
4.339
4.315
4.212
4.188
3.862
3.317
3.313
3.310 MeOD
3.307
3.303
O
OH
N
H
Cl
Cl
Cl
MeO
6f
2.03
2.09
1.00
1.07
1.07
3.03
δ (ppm)


169.529
164.405
130.641
127.075
114.826
93.746
77.546
55.964
52.749
49.511
49.340
49.170
49.000 MeOD
48.829
48.660
48.489
O
OH
N
H
Cl
Cl
Cl
MeO
6f
δ (ppm)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7.765
7.748
7.688
7.674
7.672
7.488
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7.472
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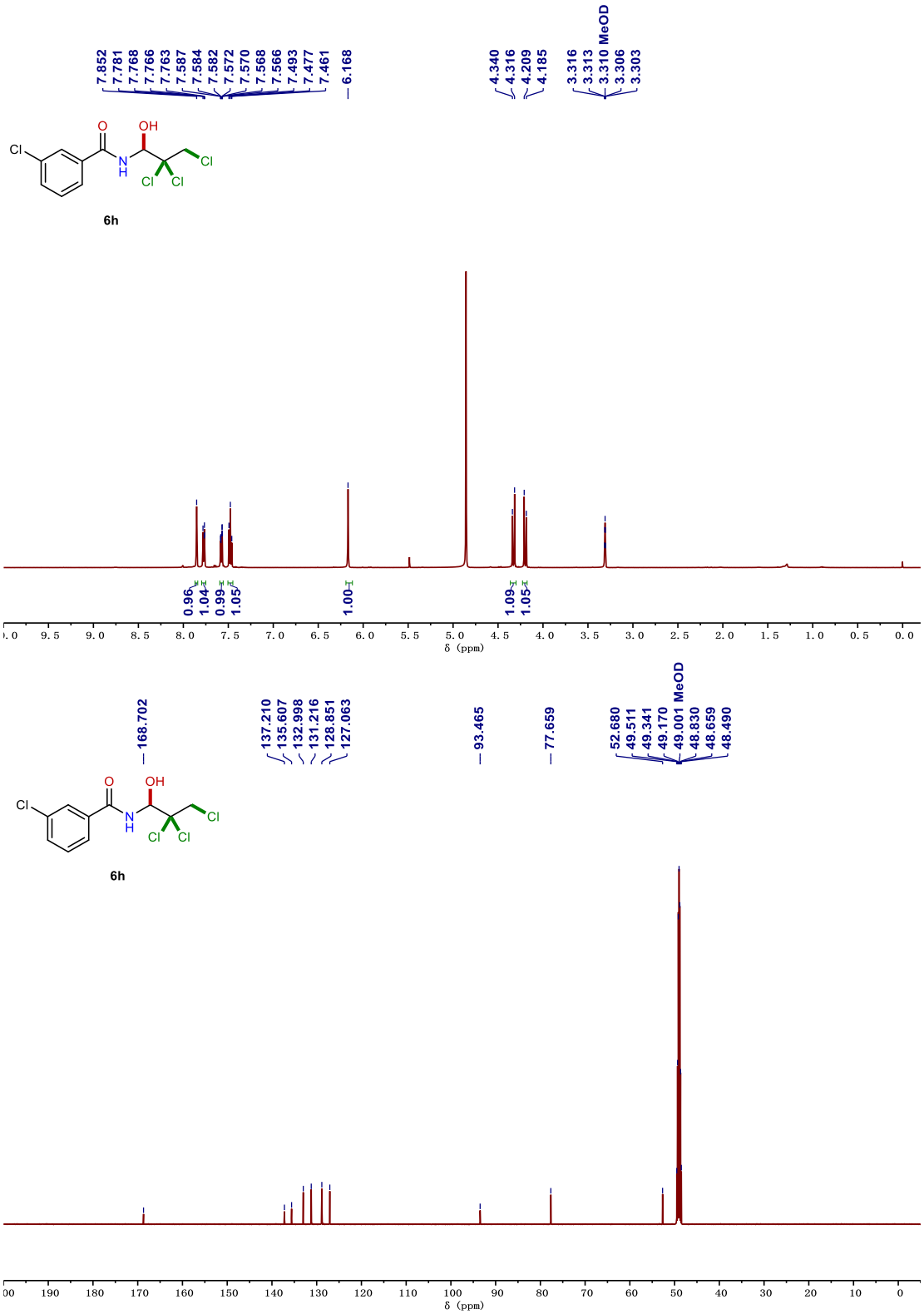
7.460
7.457
7.407
7.404
7.401
7.396
7.393
7.389
7.374
7.365
6.205
4.361
4.337
4.233
4.209
3.317
3.313
3.310
3.307
3.303
O
OH
N
H
Cl
Cl
Cl
Ph
6g
2.07
1.94
1.99
2.00
1.09
1.00
1.09
1.04
δ (ppm)
169.815
146.207
141.197
133.823
130.043
129.329
129.185
128.163
128.105
93.678
77.607
52.738
49.511
49.340
49.169
48.829
48.659
48.489
δ (ppm)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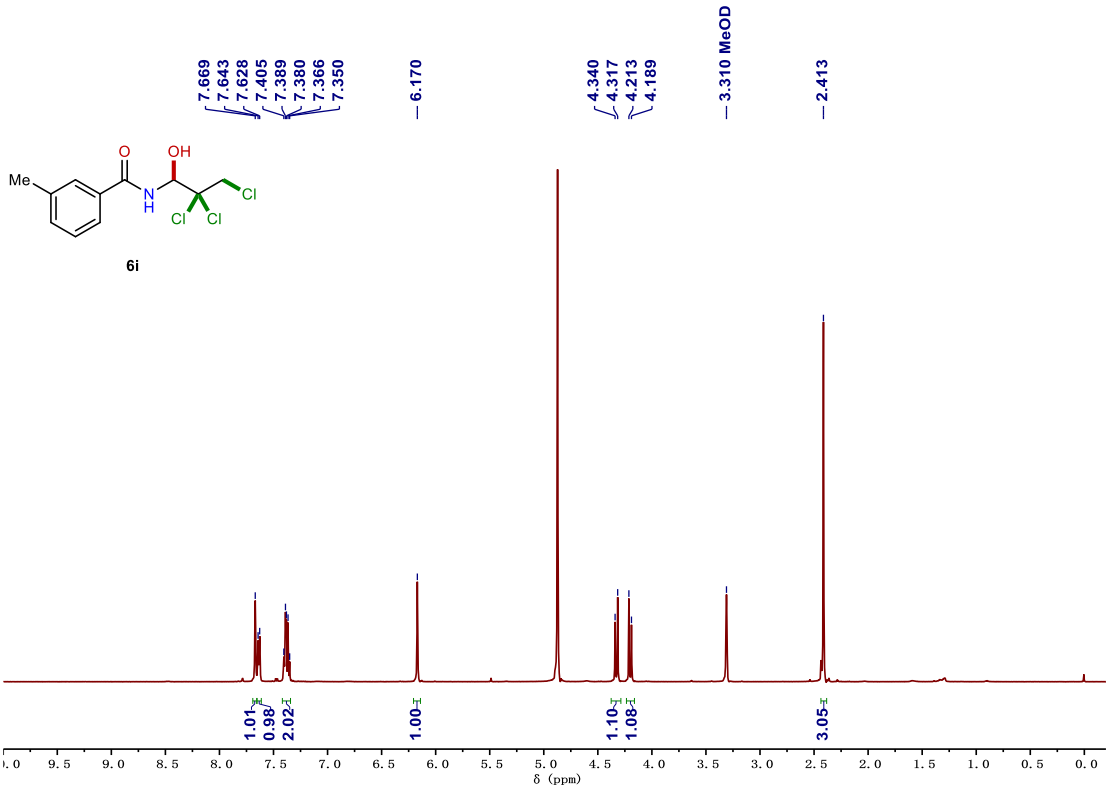
7.781
7.768
7.766
7.763
7.587
7.584
7.582
7.572
7.570
7.568
7.566
7.493
7.477
7.461
6.168
4.340
4.316
4.209
4.185
3.316
3.313
3.310 MeOD
3.306
3.303
O
OH
N
H
Cl
Cl
Cl
Cl
6h
0.96
1.04
0.99
1.05
1.00
1.09
1.05
δ (ppm)
168.702
137.210
135.607
132.998
131.216
128.851
127.063
93.465
77.659
52.680
49.511
49.341
49.170
49.001 MeOD
48.830
48.659
48.490
δ (ppm)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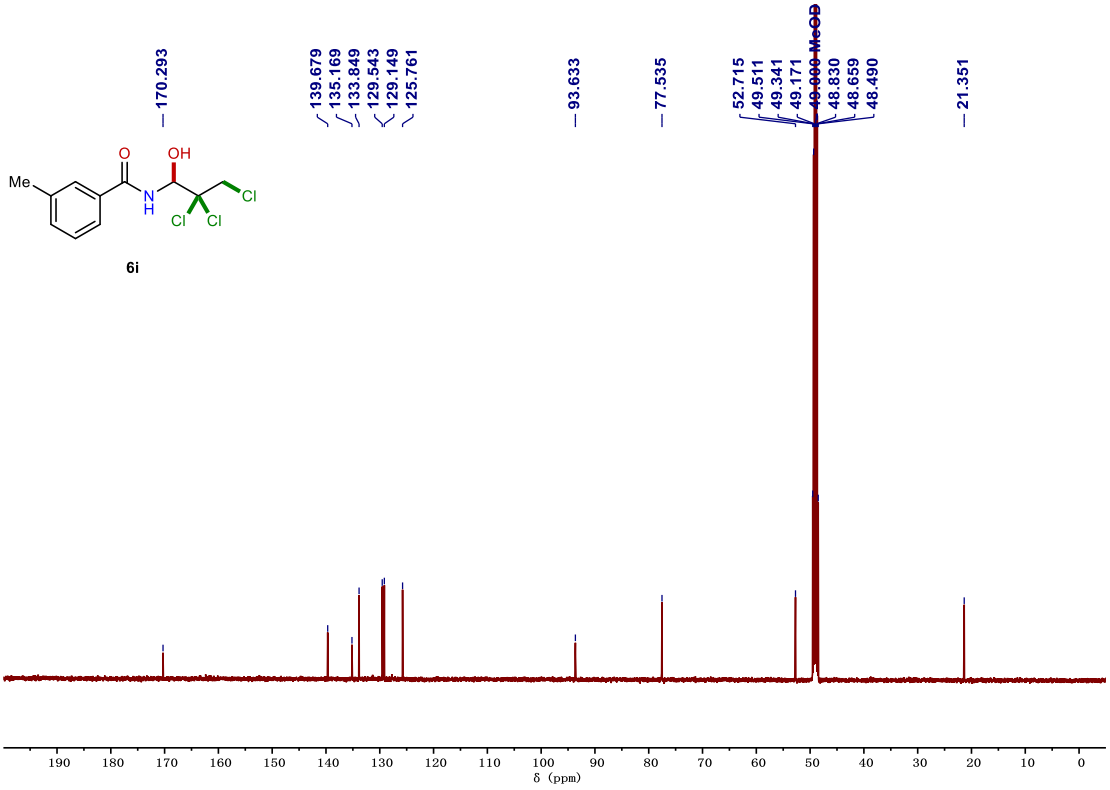
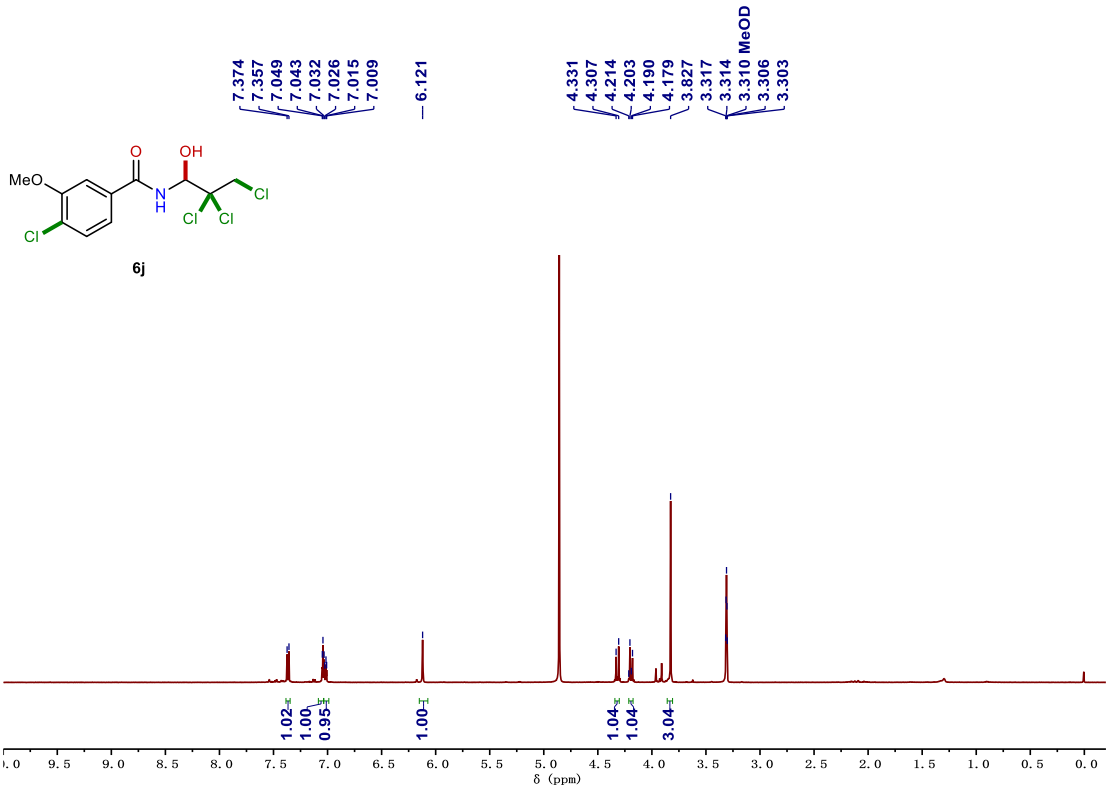
7.628
7.405
7.389
7.380
7.366
7.350
6.170
4.340
4.317
4.213
4.189
3.310 MeOD
2.413
O
OH
N
H
Me
Cl
Cl
Cl
6i
1.01
0.98
2.02
1.00
1.10
1.08
3.05
δ (ppm)


170.293
139.679
135.169
133.849
129.543
129.149
125.761
93.633
77.535
52.715
49.511
49.341
49.171
49.000 MeOD
48.830
48.659
48.490
21.351
O
OH
N
H
Me
Cl
Cl
Cl
6i
δ (ppm)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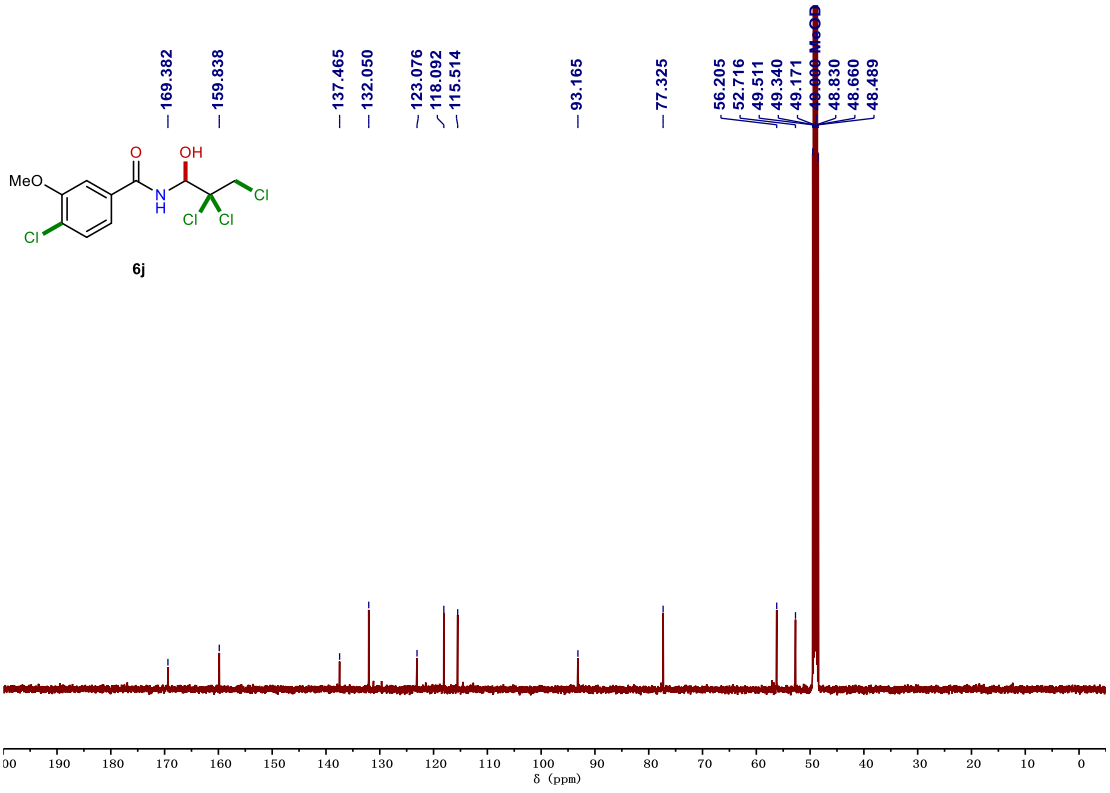
7.357
7.049
7.043
7.032
7.026
7.015
7.009
6.121
4.331
4.307
4.214
4.203
4.190
4.179
3.827
3.317
3.314
3.310 MeOD
3.306
3.303
MeO
O
OH
N
H
Cl
Cl
Cl
Cl
1.02
1.00
0.95
1.00
1.04
1.04
3.04
δ (ppm)

6j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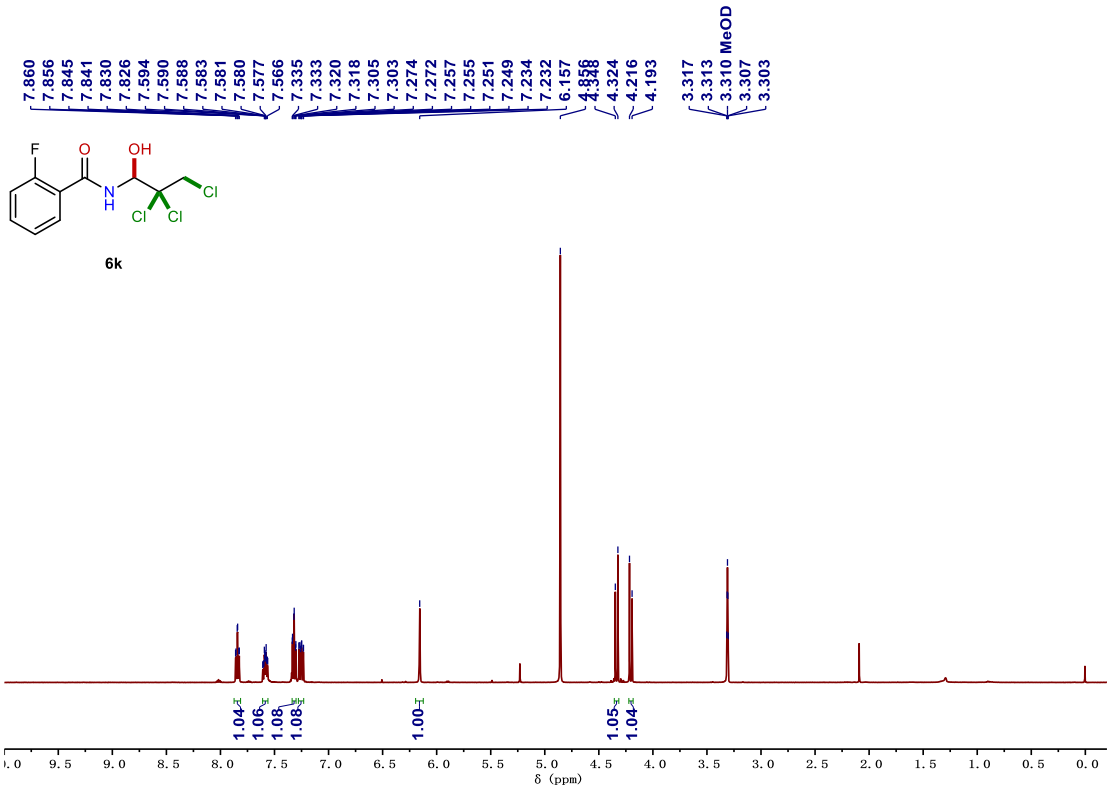
159.838
137.465
132.050
123.076
118.092
115.514
93.165
77.325
56.205
52.716
49.511
49.340
49.171
48.830
48.660
48.489
MeO
O
OH
N
H
Cl
Cl
Cl
Cl
δ (ppm)

6j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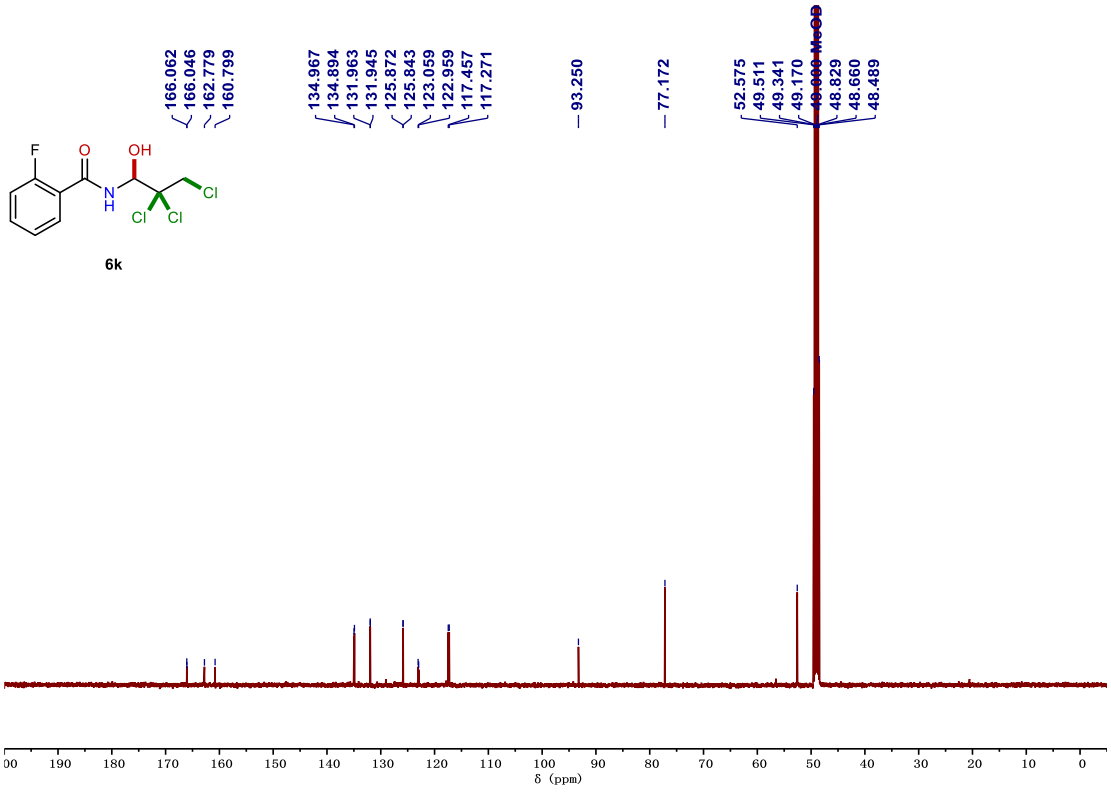
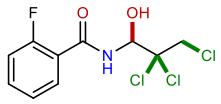
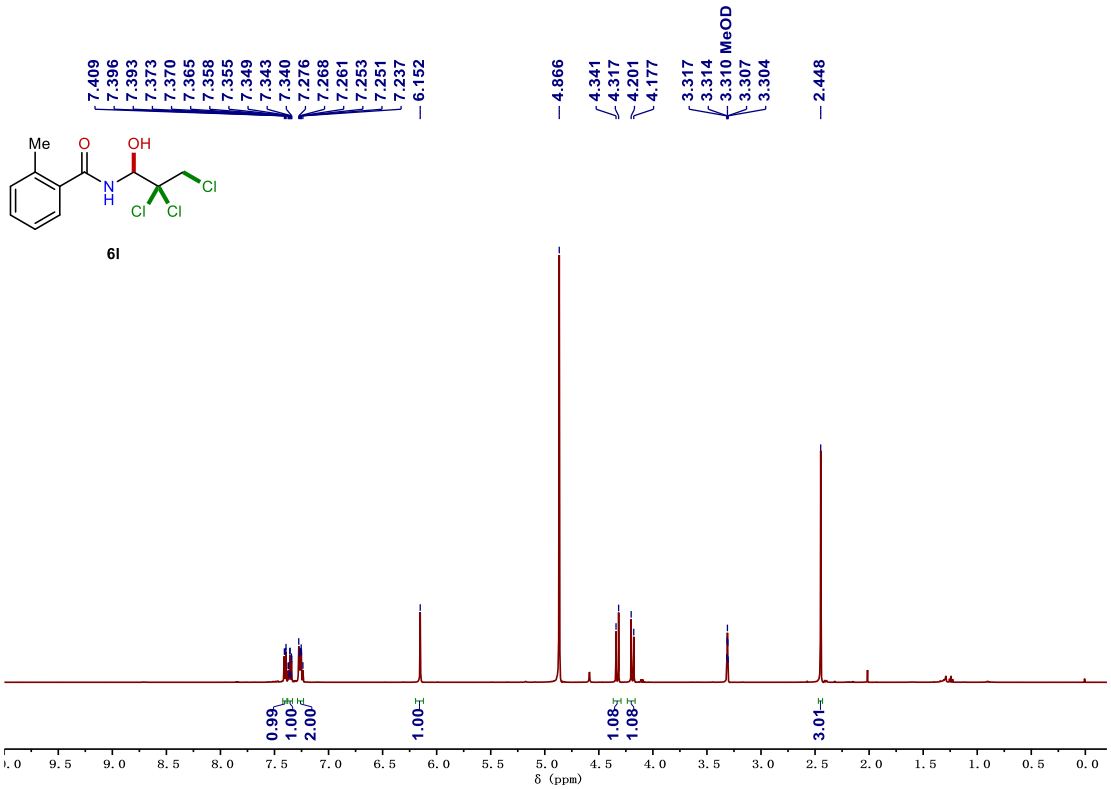
7.856
7.845
7.841
7.830
7.826
7.594
7.590
7.588
7.583
7.581
7.580
7.577
7.566
7.335
7.333
7.320
7.318
7.305
7.303
7.274
7.272
7.257
7.255
7.251
7.249
7.234
7.232
6.157
4.324
4.216
4.193
3.317
3.313
3.310 MeOD
3.307
3.303
F
O
OH
N
H
Cl
Cl
Cl
6k
1.04
1.06
1.08
1.08
1.00
1.05
1.04
δ (ppm)


166.062
166.046
162.779
160.799
134.967
134.894
131.963
131.945
125.872
125.843
123.059
122.959
117.457
117.271
93.250
77.172
52.575
49.511
49.341
49.170
49.000 MeOD
48.829
48.660
48.489
F
O
OH
N
H
Cl
Cl
Cl
6k
δ (ppm)


F
O
OH
N
H
Cl
Cl
Cl

6k

-115.518
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)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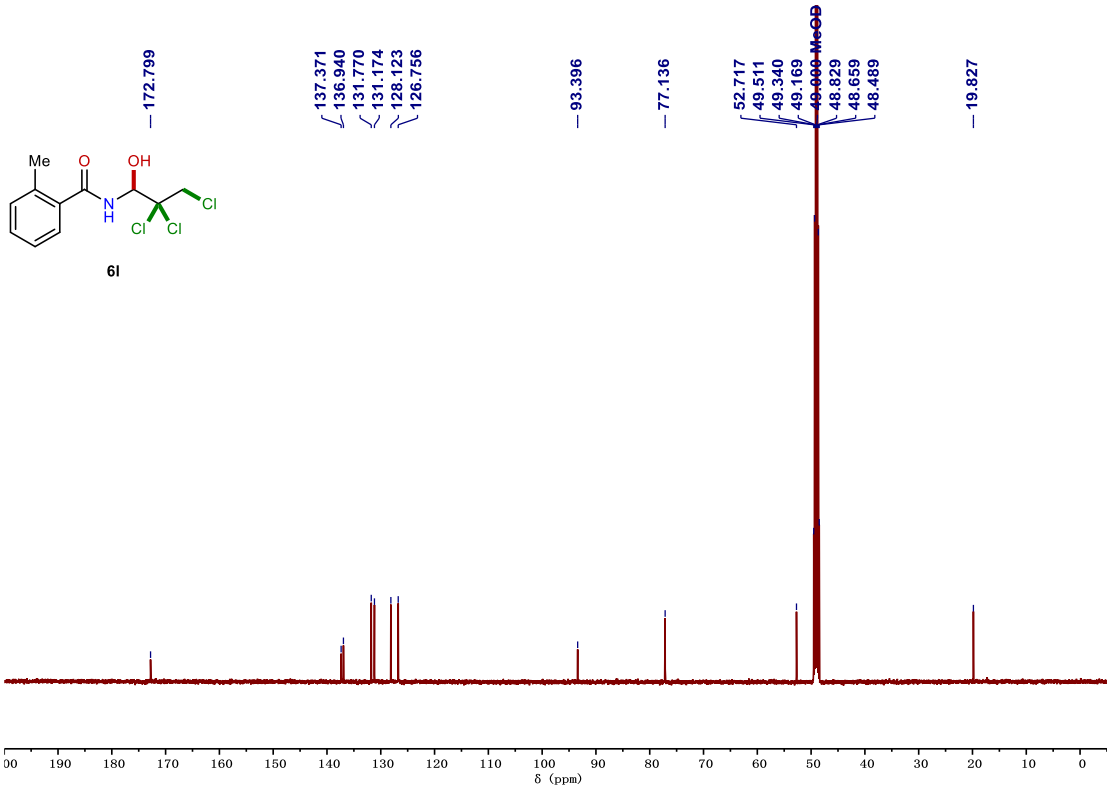
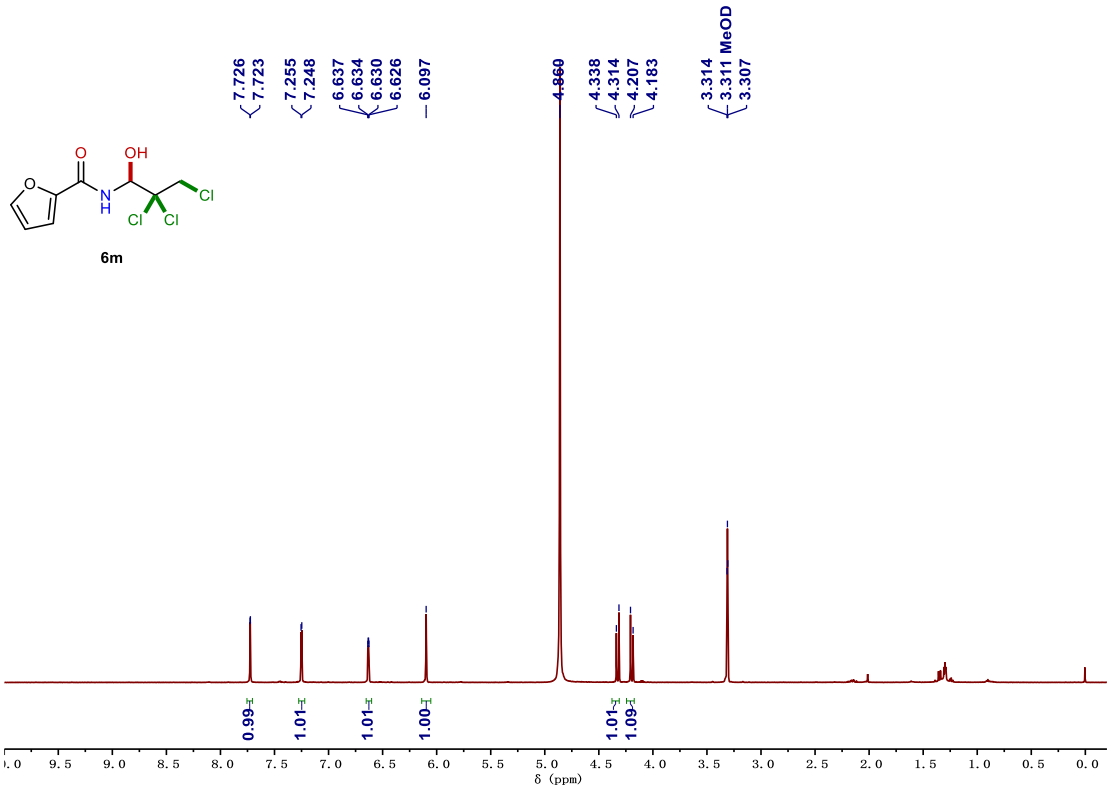
7.396
7.393
7.373
7.370
7.365
7.358
7.355
7.349
7.343
7.340
7.276
7.268
7.261
7.253
7.251
7.237
6.152
4.866
4.341
4.317
4.201
4.177
3.317
3.314
3.310 MeOD
3.307
3.304
2.448
Me
O
OH
N
H
Cl
Cl
Cl
0.99
1.00
2.00
1.00
1.08
1.08
3.01
δ (ppm)

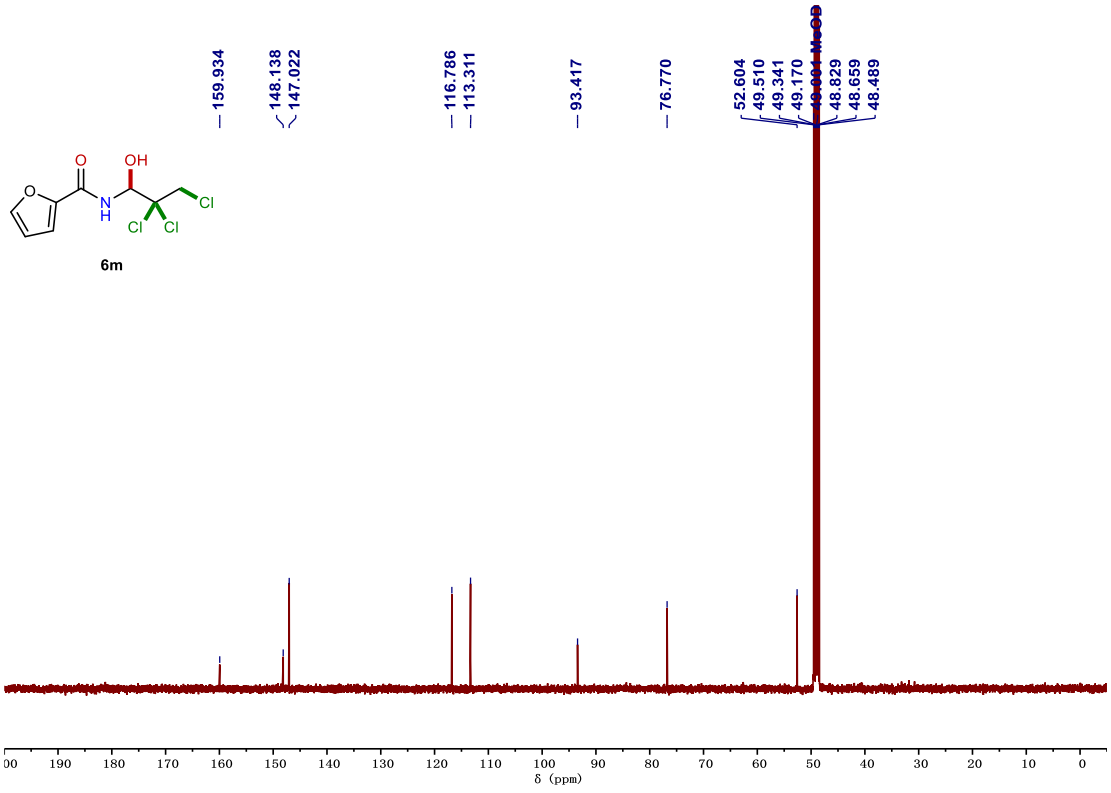
6l


172.799
137.371
136.940
131.770
131.174
128.123
126.756
93.396
77.136
52.717
49.511
49.340
49.169
49.000 MeOD
48.829
48.659
48.489
19.827
Me
O
OH
N
H
Cl
Cl
Cl
δ (ppm)

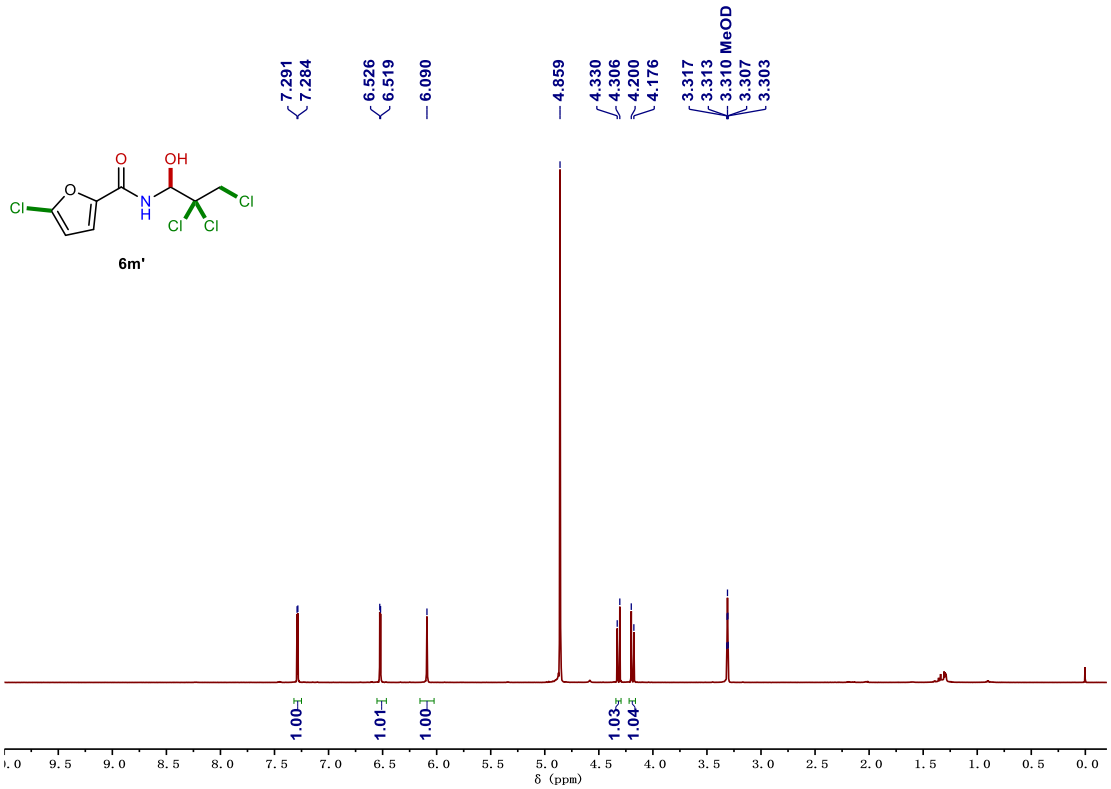
6l

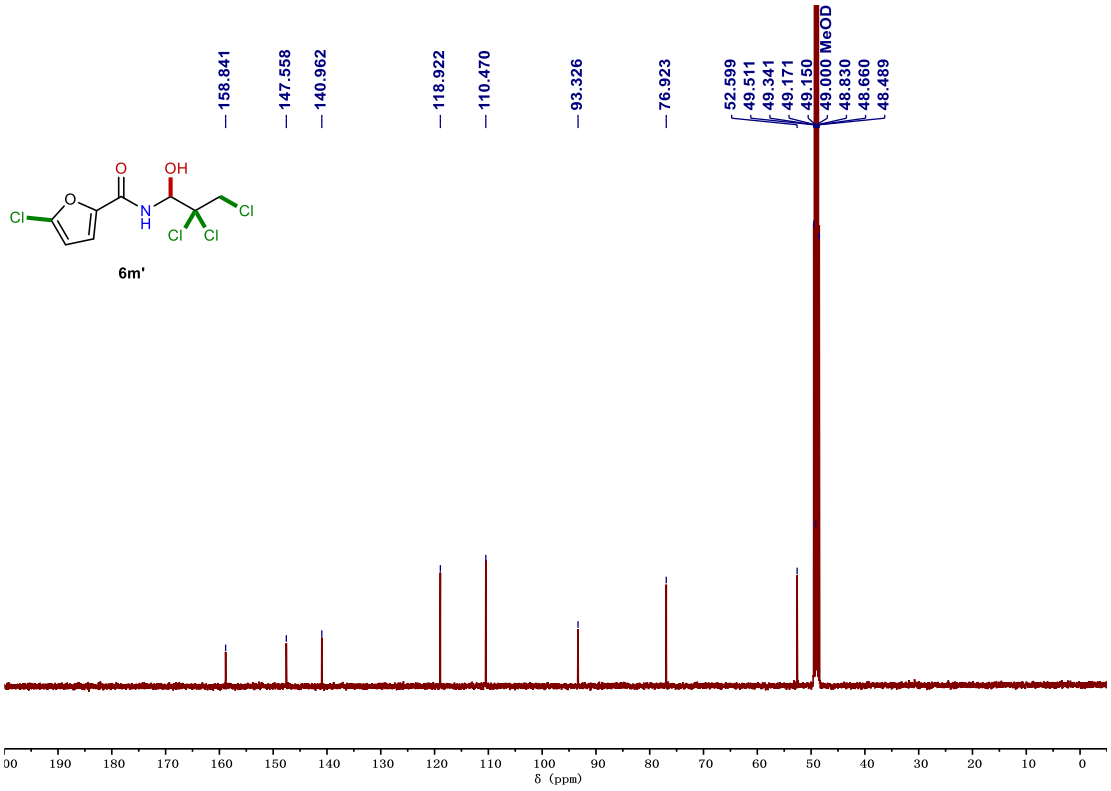

7.726
7.723
7.255
7.248
6.637
6.634
6.630
6.626
6.097
4.860
4.338
4.314
4.207
4.183
3.314
3.311 MeOD
3.307
O
OH
N
H
Cl
Cl
Cl
0.99
1.01
1.01
1.00
1.01
1.09
δ (ppm)

6m


159.934
148.138
147.022
116.786
113.311
93.417
76.770
52.604
49.510
49.341
49.170
48.829
48.659
48.489
O
OH
N
H
Cl
Cl
Cl
δ (ppm)

6m


7.291
7.284
6.526
6.519
6.090
4.859
4.330
4.306
4.200
4.176
3.317
3.313
3.310 MeOD
3.307
3.303
O
OH
N
H
Cl
Cl
Cl
Cl
6m'
1.00
1.01
1.00
1.03
1.04
δ (ppm)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140.962
118.922
110.470
93.326
76.923
52.599
49.511
49.341
49.171
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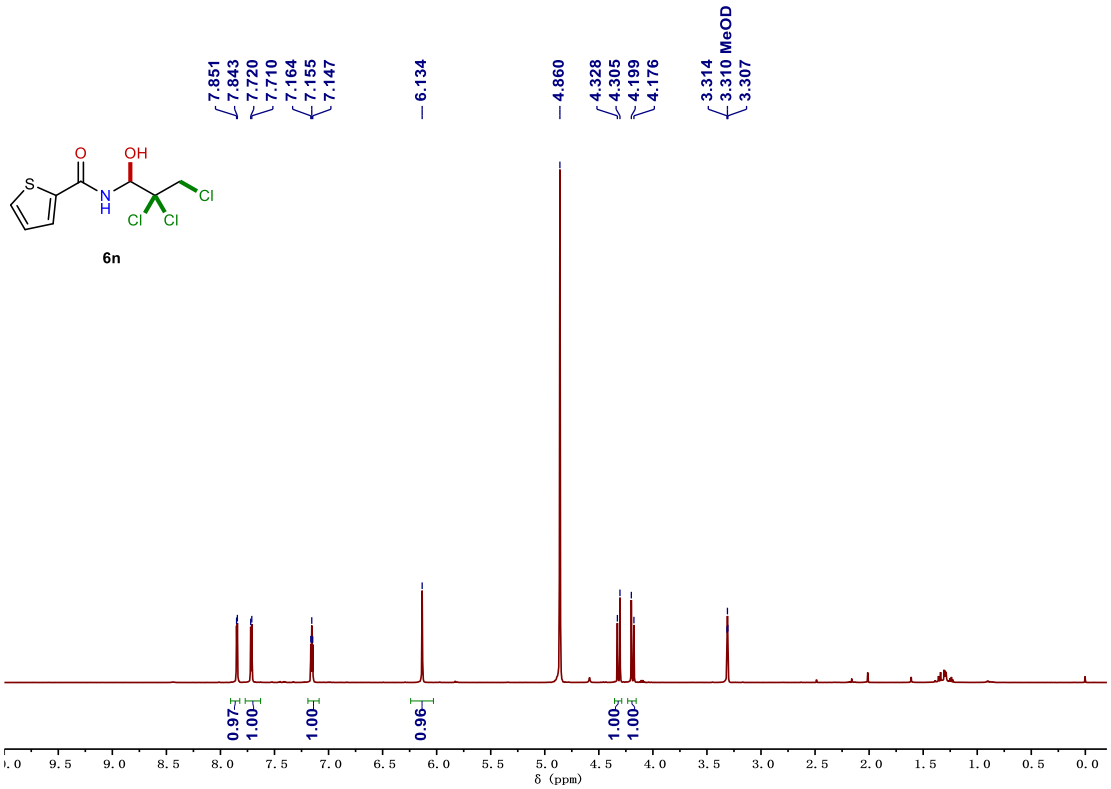
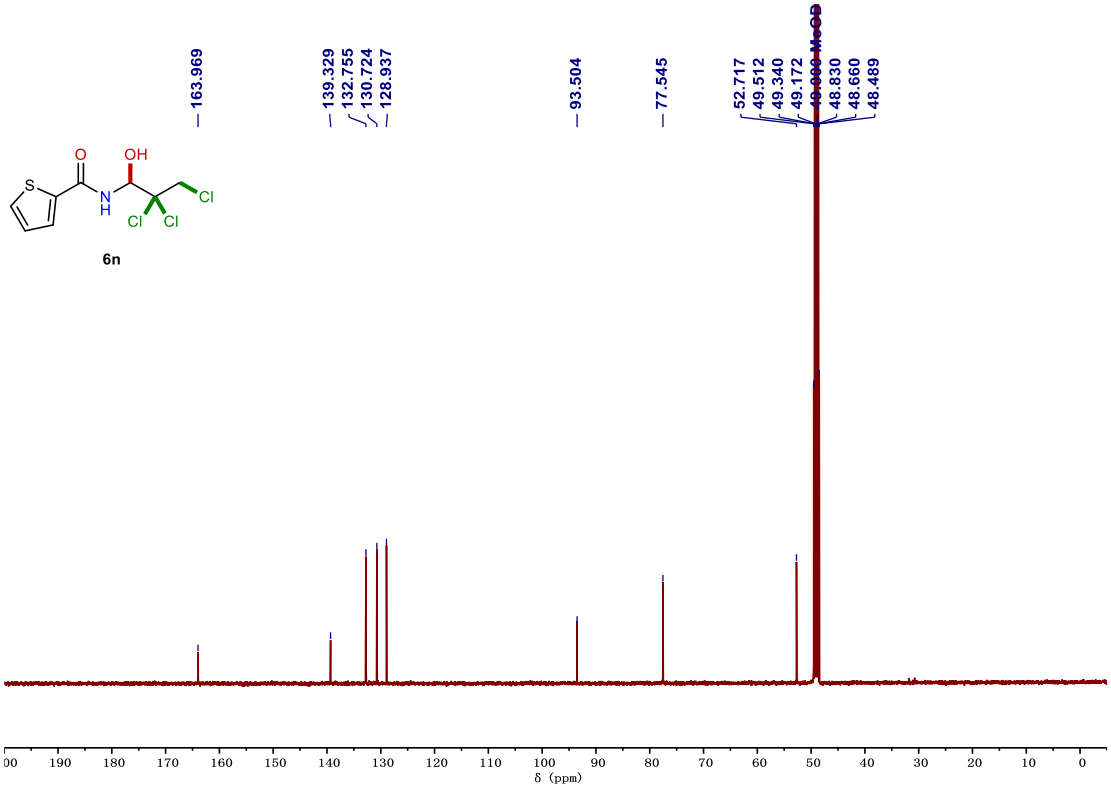
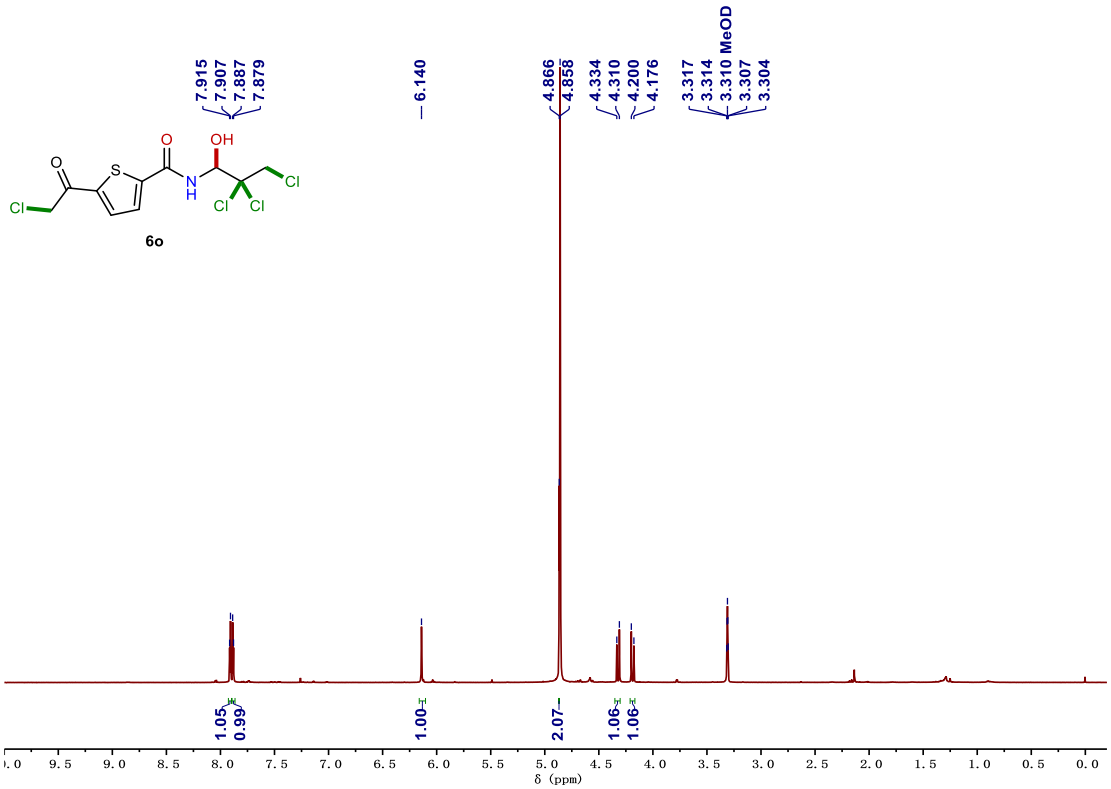
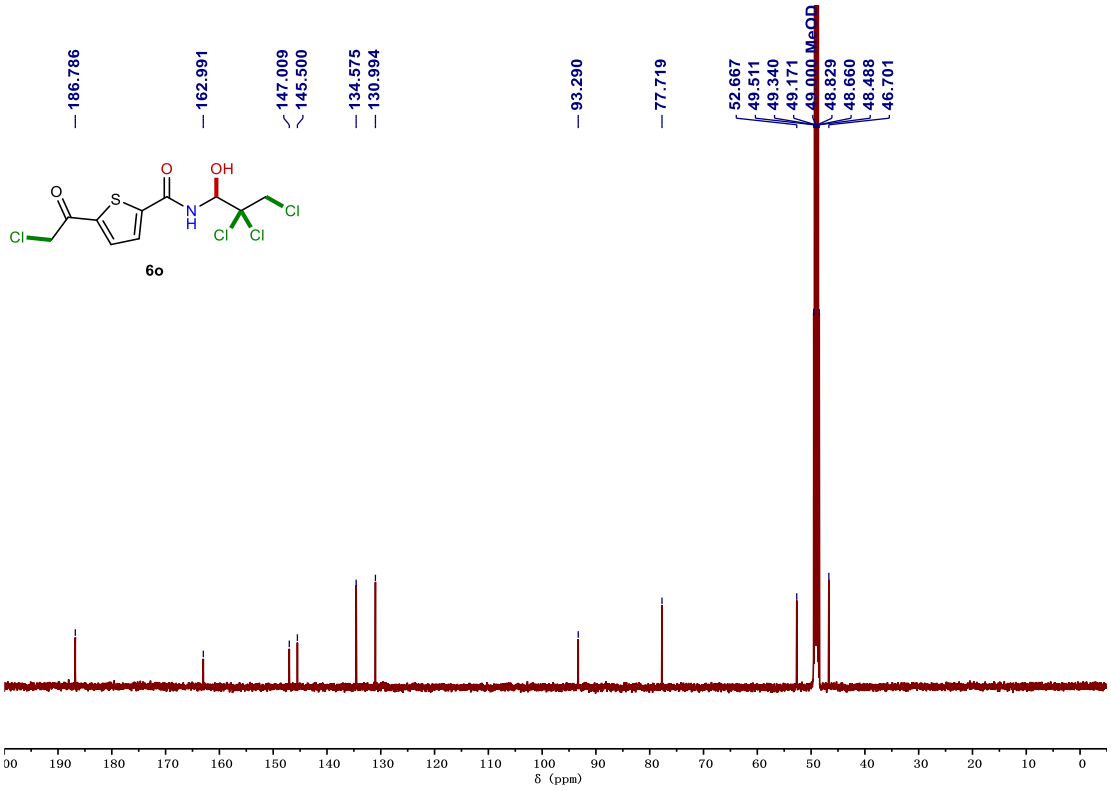
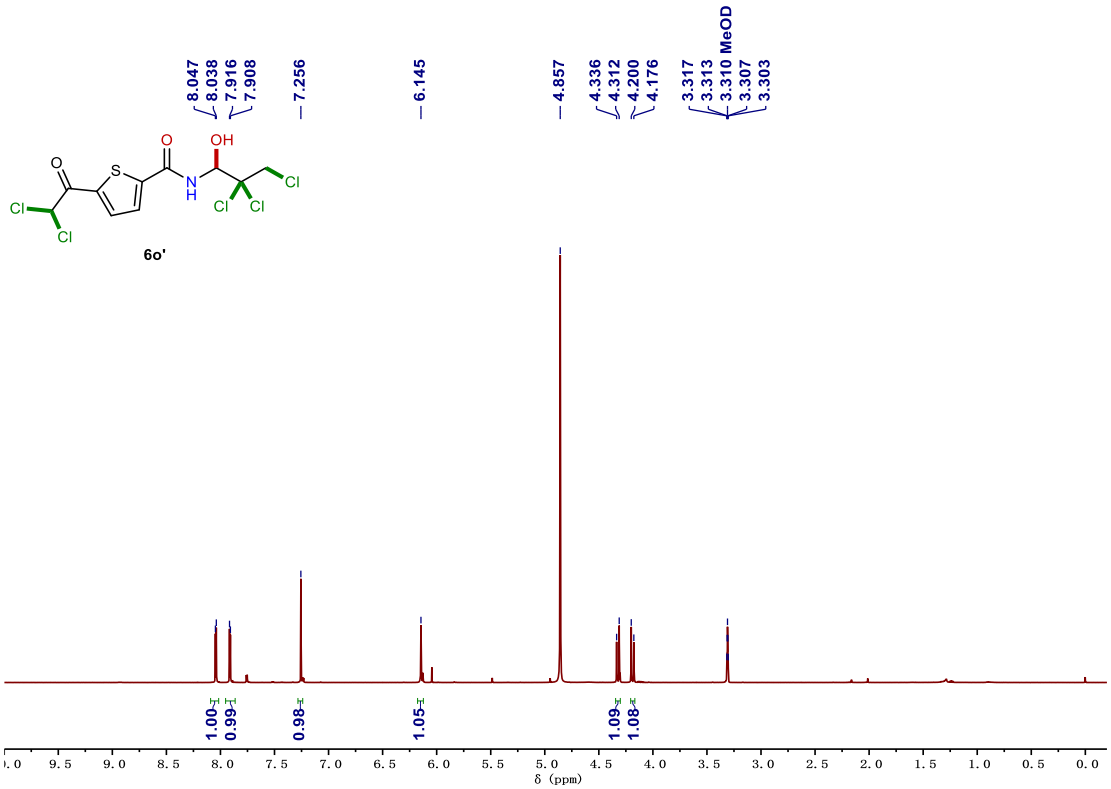
OH
N
H
Cl
Cl
Cl
Cl
6m'
δ (ppm)

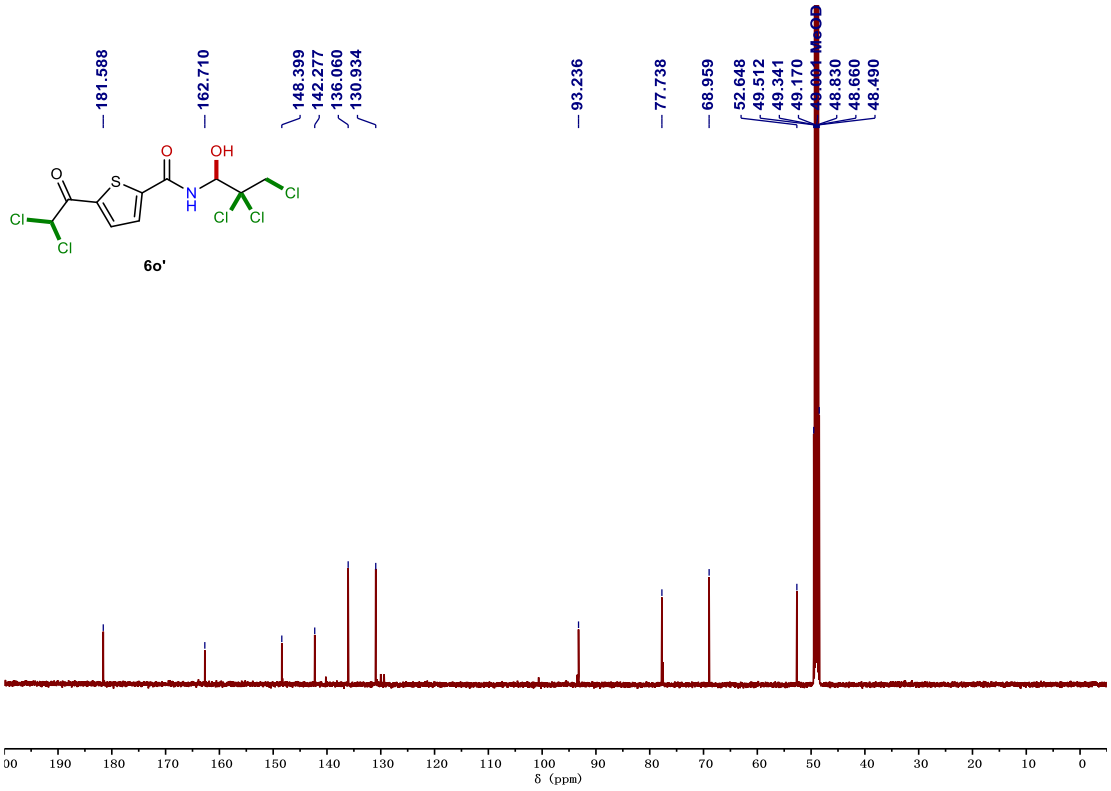

7.851
7.843
7.720
7.710
7.164
7.155
7.147
6.134
4.860
4.328
4.305
4.199
4.176
3.314
3.310 MeOD
3.307
O
OH
S
N
H
Cl
Cl
Cl
6n
0.97
1.00
1.00
0.96
1.00
1.00
δ (ppm)


163.969
139.329
132.755
130.724
128.937
93.504
77.545
52.717
49.512
49.340
49.172
49.000 MeOD
48.830
48.660
48.489
O
OH
S
N
H
Cl
Cl
Cl
6n
δ (ppm)


7.915
7.907
7.887
7.879
6.140
4.866
4.858
4.334
4.310
4.200
4.176
3.317
3.314
3.310 MeOD
3.307
3.304
O
OH
S
N
H
Cl
Cl
Cl
Cl
6o
1.05
0.99
1.00
2.07
1.06
1.06
δ (ppm)


186.786
162.991
147.009
145.500
134.575
130.994
93.290
77.719
52.667
49.511
49.340
49.171
49.000 MeOD
48.829
48.660
48.488
46.701
O
OH
S
N
H
Cl
Cl
Cl
Cl
6o
δ (ppm)


8.047
8.038
7.916
7.908
7.256
6.145
4.857
4.336
4.312
4.200
4.176
3.317
3.313
3.310 MeOD
3.307
3.303
1.00
0.99
0.98
1.05
1.09
1.08
δ (ppm)
6o'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162.710
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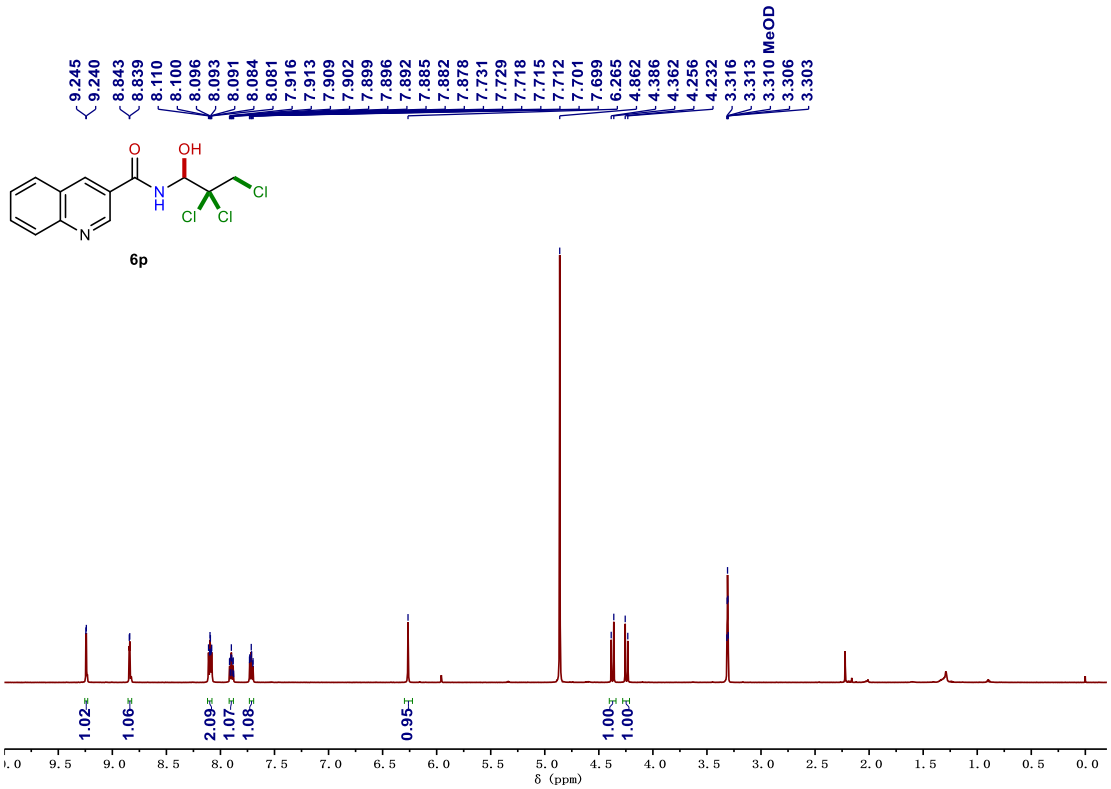
142.277
136.060
130.934
93.236
77.738
68.959
52.648
49.512
49.341
49.170
49.001 MeOD
48.830
48.660
48.490
δ (ppm)
6o'


9.245
9.240
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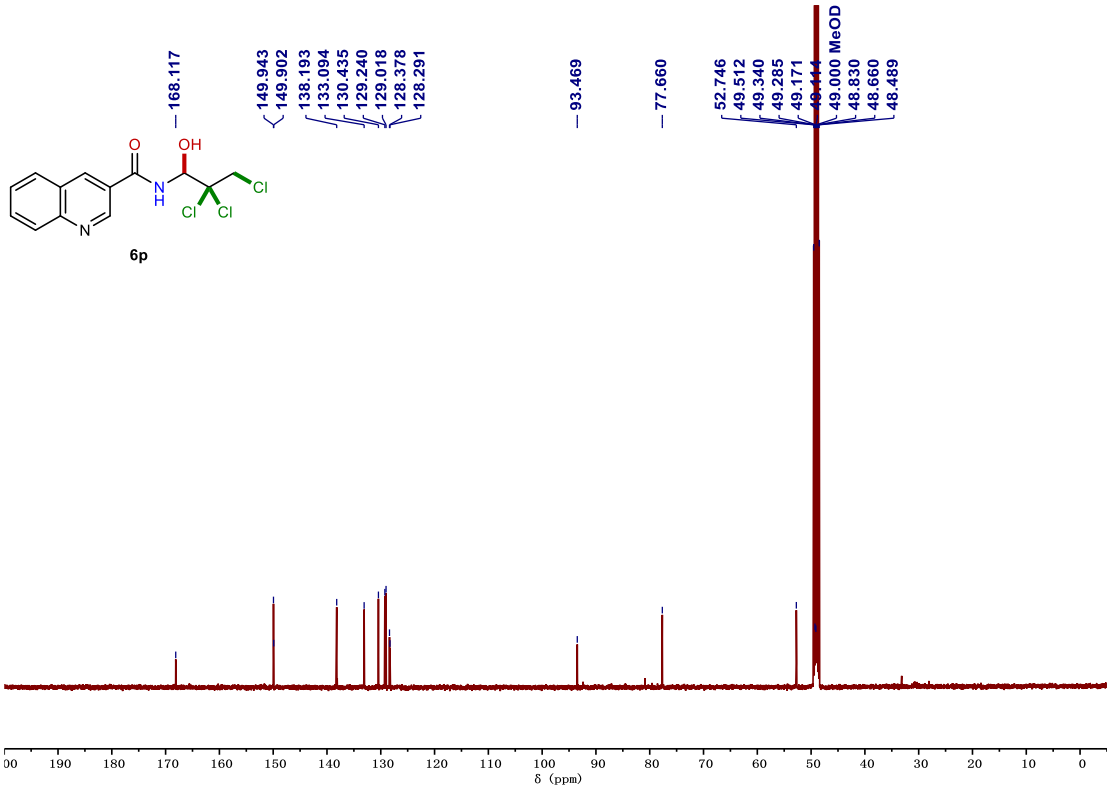
8.839
8.110
8.100
8.096
8.093
8.091
8.084
8.081
7.916
7.913
7.909
7.902
7.899
7.896
7.892
7.885
7.882
7.878
7.731
7.729
7.718
7.715
7.712
7.701
7.699
6.265
4.862
4.386
4.362
4.256
4.232
3.316
3.313
3.310 MeOD
3.306
3.303
O
OH
N
H
Cl
Cl
Cl
N
6p
1.02
1.06
2.09
1.07
1.08
0.95
1.00
1.00
δ (ppm)


168.117
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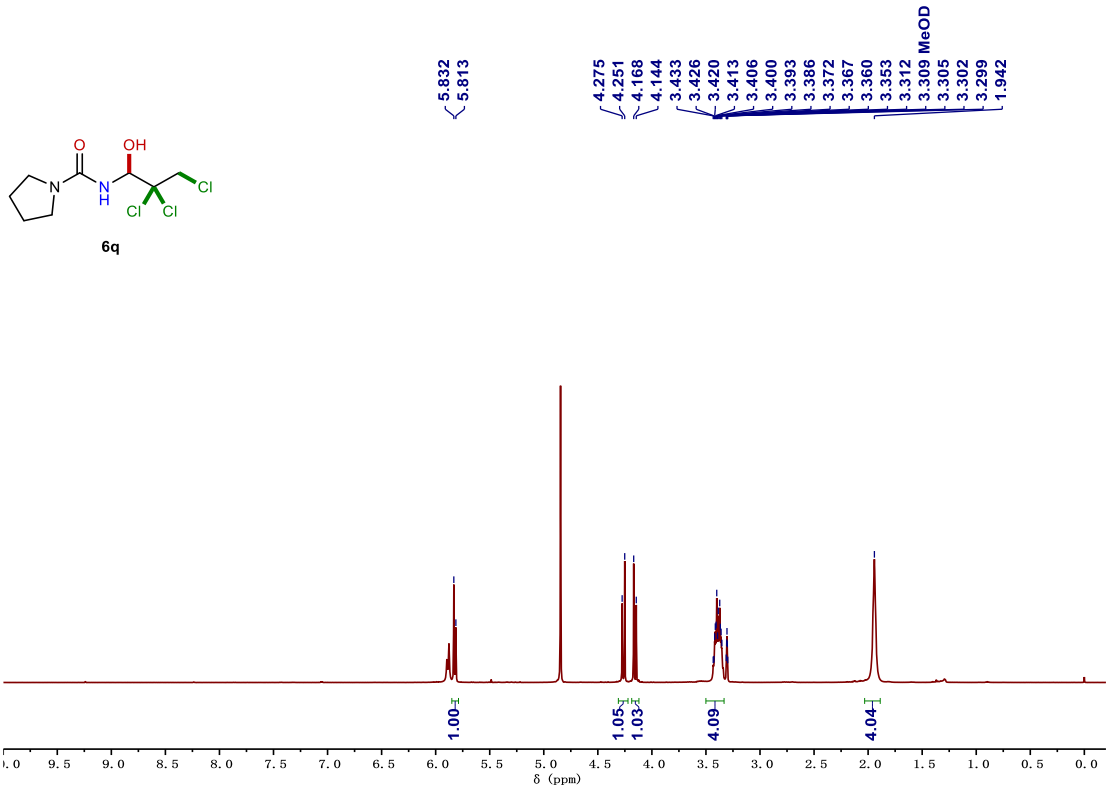
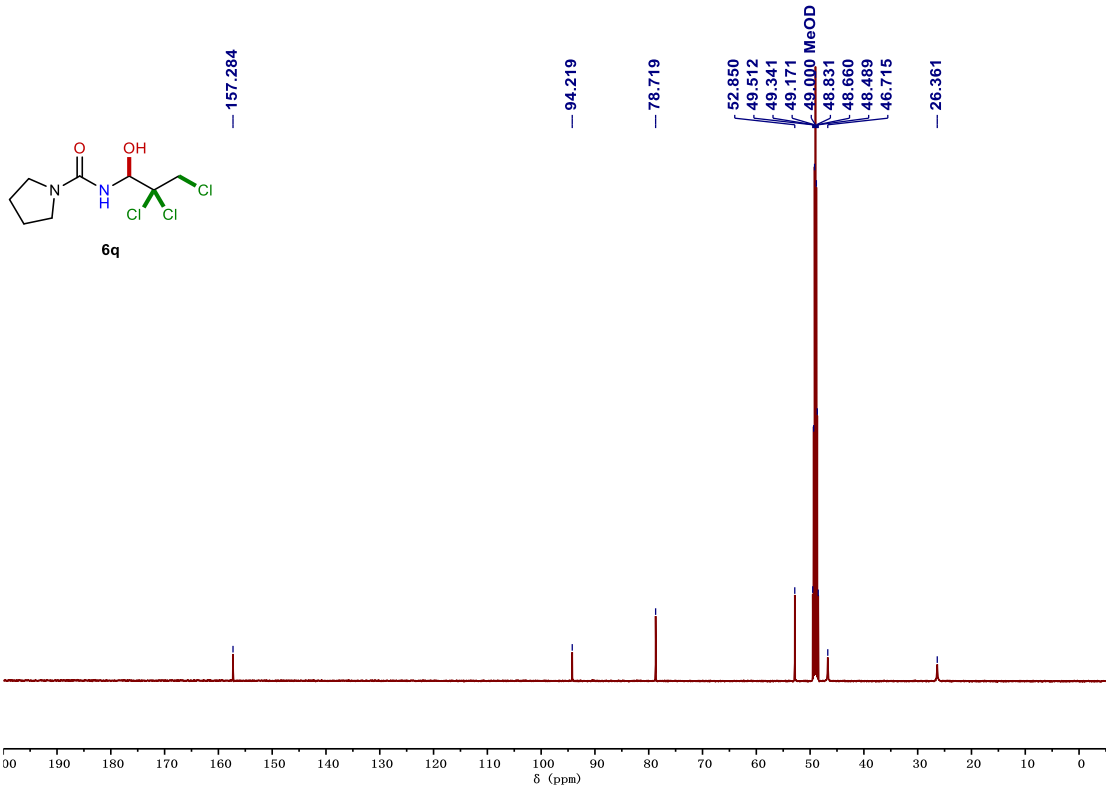
149.902
138.193
133.094
130.435
129.240
129.018
128.378
128.291
93.469
77.660
52.746
49.512
49.340
49.285
49.171
49.000 MeOD
48.830
48.660
48.489
O
OH
N
H
Cl
Cl
Cl
N
6p
δ (ppm)


5.832
5.813
4.275
4.251
4.168
4.144
3.433
3.426
3.420
3.413
3.406
3.400
3.393
3.386
3.372
3.367
3.360
3.353
3.312
3.309 MeOD
3.305
3.302
3.299
1.942
O
OH
N
H
Cl
Cl
Cl
6q
1.00
1.05
1.03
4.09
4.04
δ (ppm)


157.284
94.219
78.719
52.850
49.512
49.341
49.171
49.000 MeOD
48.831
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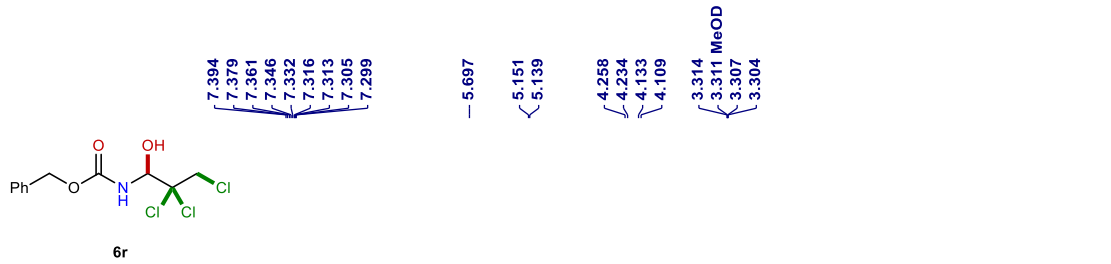
48.489
46.715
26.361
O
OH
N
H
Cl
Cl
Cl
6q
δ (ppm)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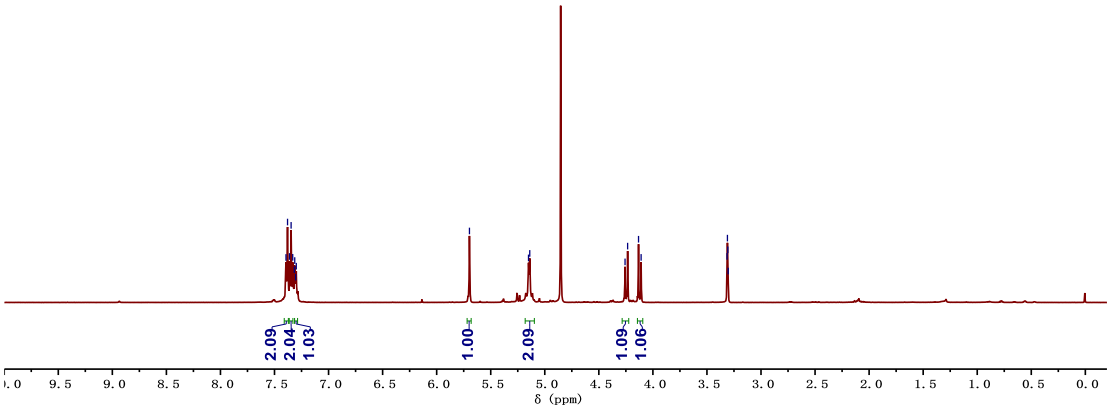
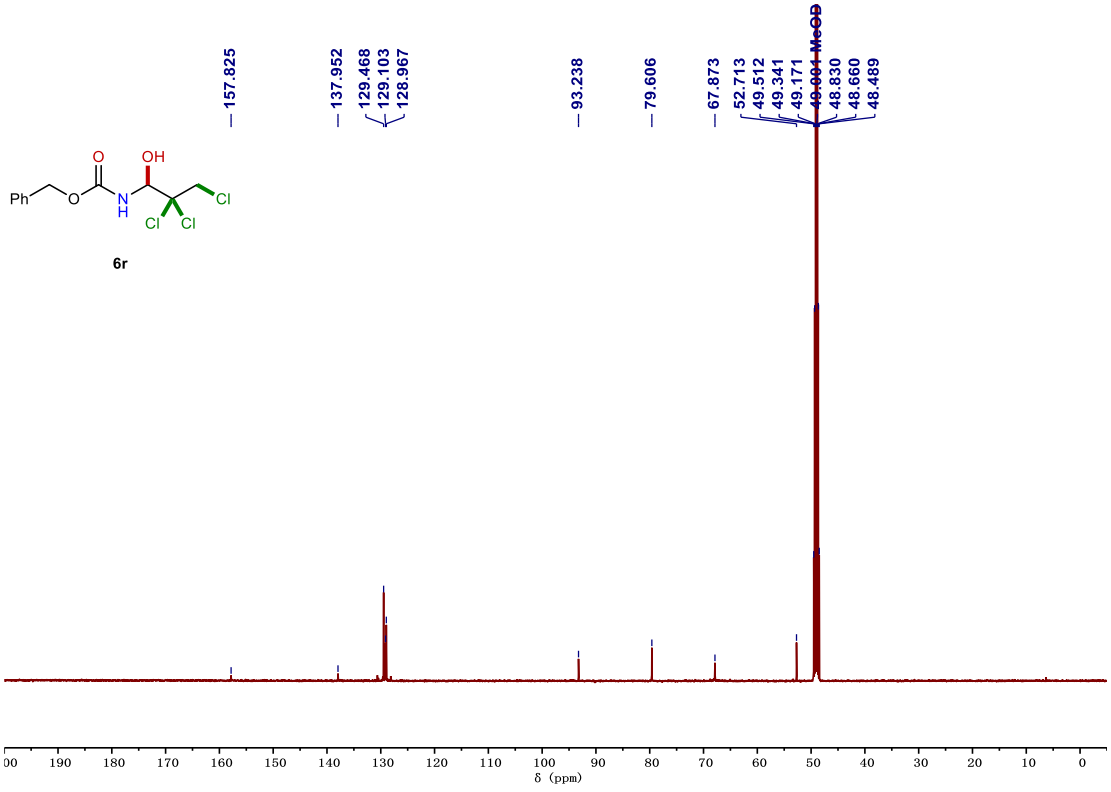
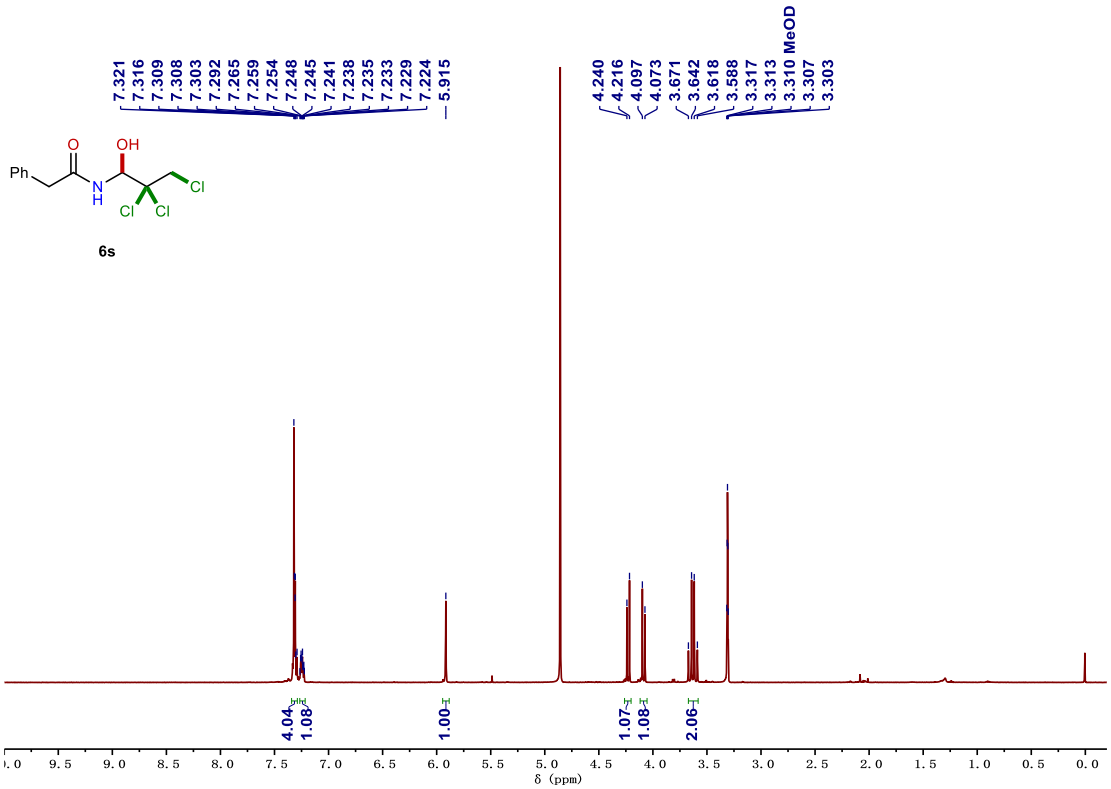
7.332
7.316
7.313
7.305
7.299
5.697
5.151
5.139
4.258
4.234
4.133
4.109
3.314
3.311 MeOD
3.307
3.304
O
OH
Ph
O
N
H
Cl
Cl
Cl

6r


2.09
2.04
1.03
1.00
2.09
1.09
1.06
δ (ppm)


157.825
137.952
129.468
129.103
128.967
93.238
79.606
67.873
52.713
49.512
49.341
49.171
49.001 MeOD
48.830
48.660
48.489
O
OH
Ph
O
N
H
Cl
Cl
Cl
δ (ppm)

6r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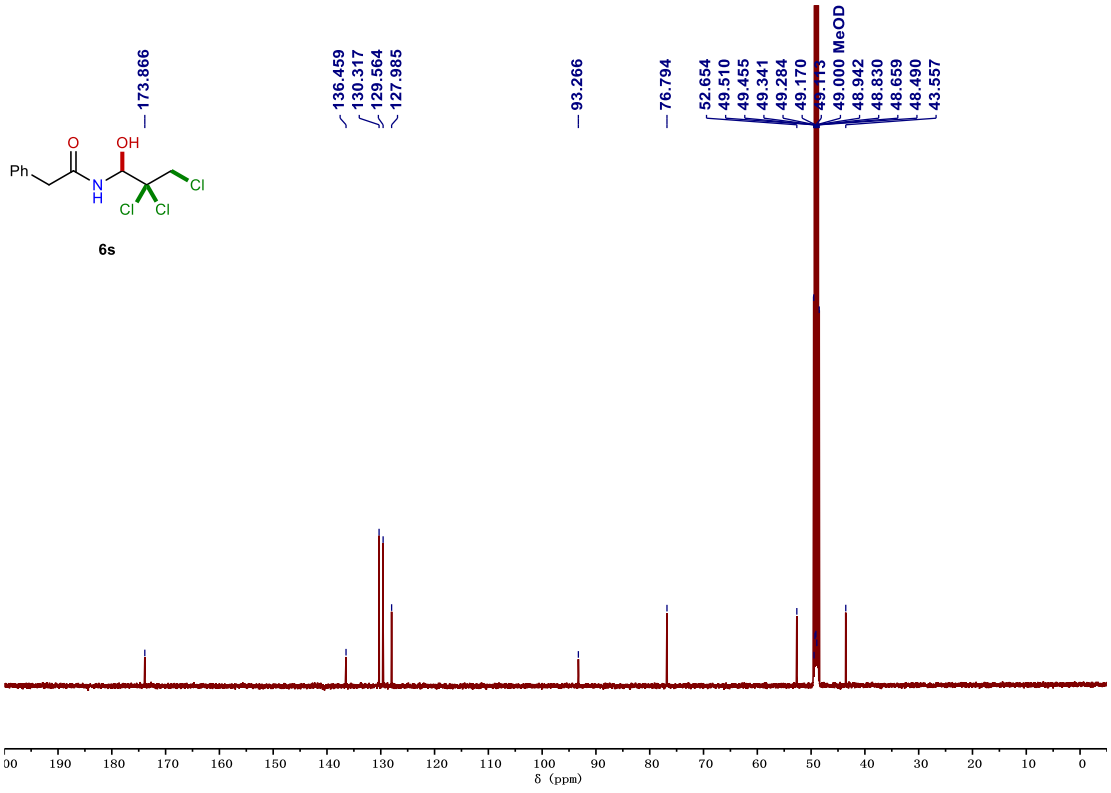
7.316
7.309
7.308
7.303
7.292
7.265
7.259
7.254
7.248
7.245
7.241
7.238
7.235
7.233
7.229
7.224
5.915
4.240
4.216
4.097
4.073
3.671
3.642
3.618
3.588
3.317
3.313
3.310 MeOD
3.307
3.303
O
OH
Ph
N
H
Cl
Cl
Cl
6s
4.04
1.08
1.00
1.07
1.08
2.06
δ (ppm)


173.866
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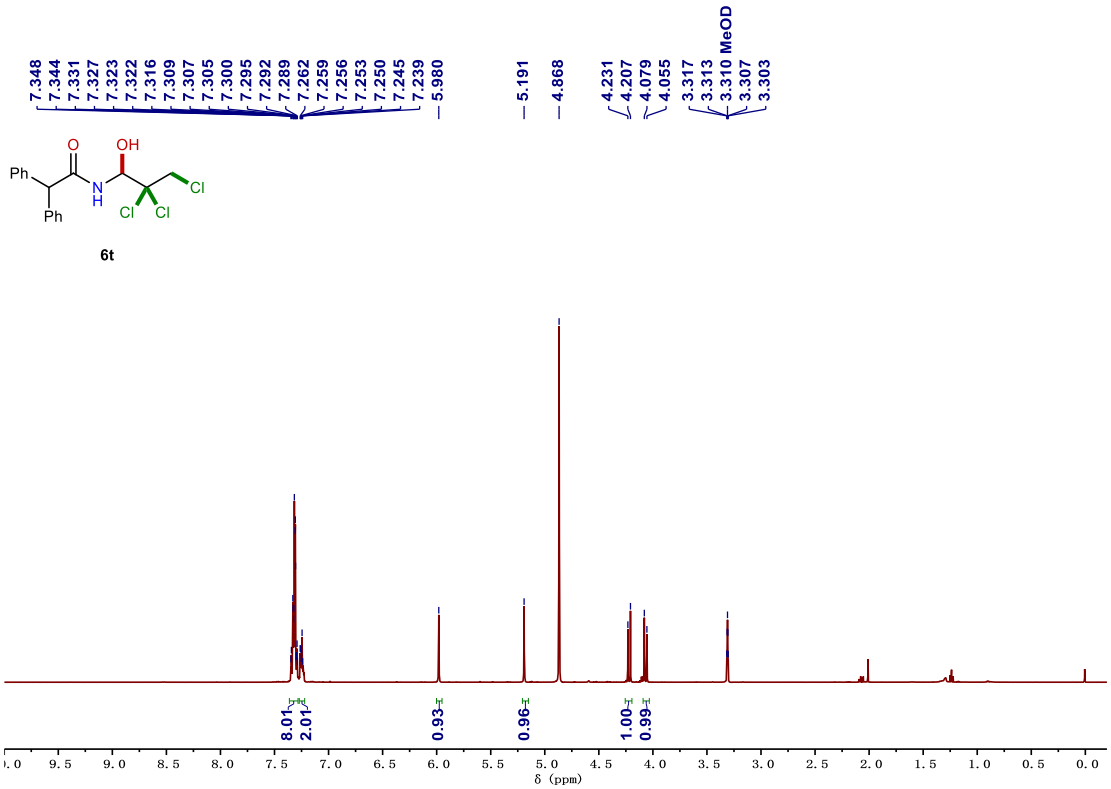
130.317
129.564
127.985
93.266
76.794
52.654
49.510
49.455
49.341
49.284
49.170
49.113
49.000 MeOD
48.942
48.830
48.659
48.490
43.557
O
OH
Ph
N
H
Cl
Cl
Cl
6s
δ (ppm)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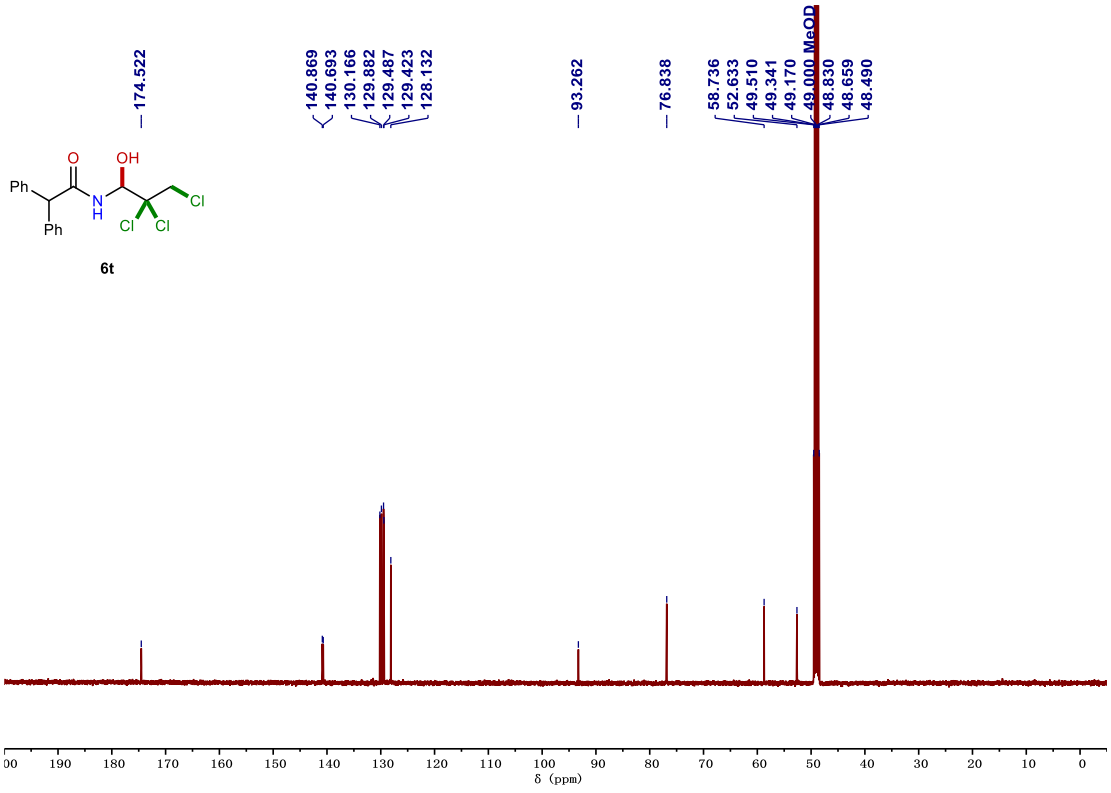
7.344
7.331
7.327
7.323
7.322
7.316
7.309
7.307
7.305
7.300
7.295
7.292
7.289
7.262
7.259
7.256
7.253
7.250
7.245
7.239
5.980
5.191
4.868
4.231
4.207
4.079
4.055
3.317
3.313
3.310 MeOD
3.307
3.303
Ph
O
OH
N
H
Cl
Cl
Cl
Ph
6t
8.01
2.01
0.93
0.96
1.00
0.99
δ (ppm)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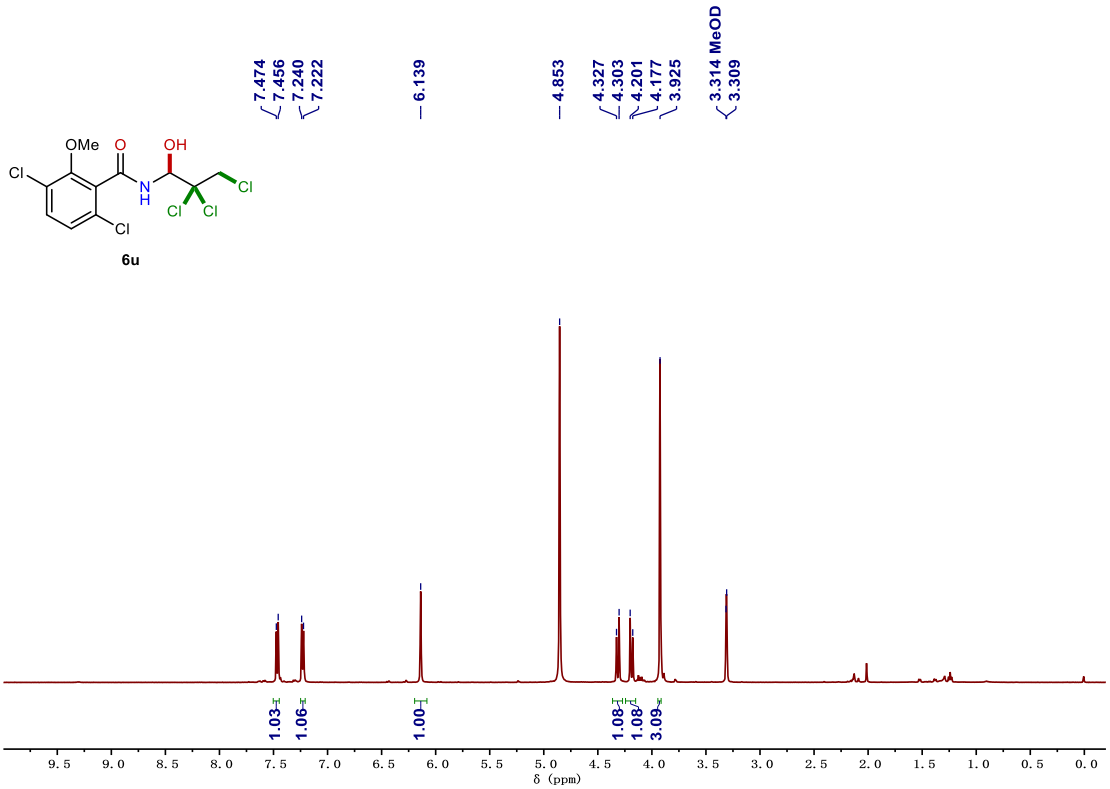
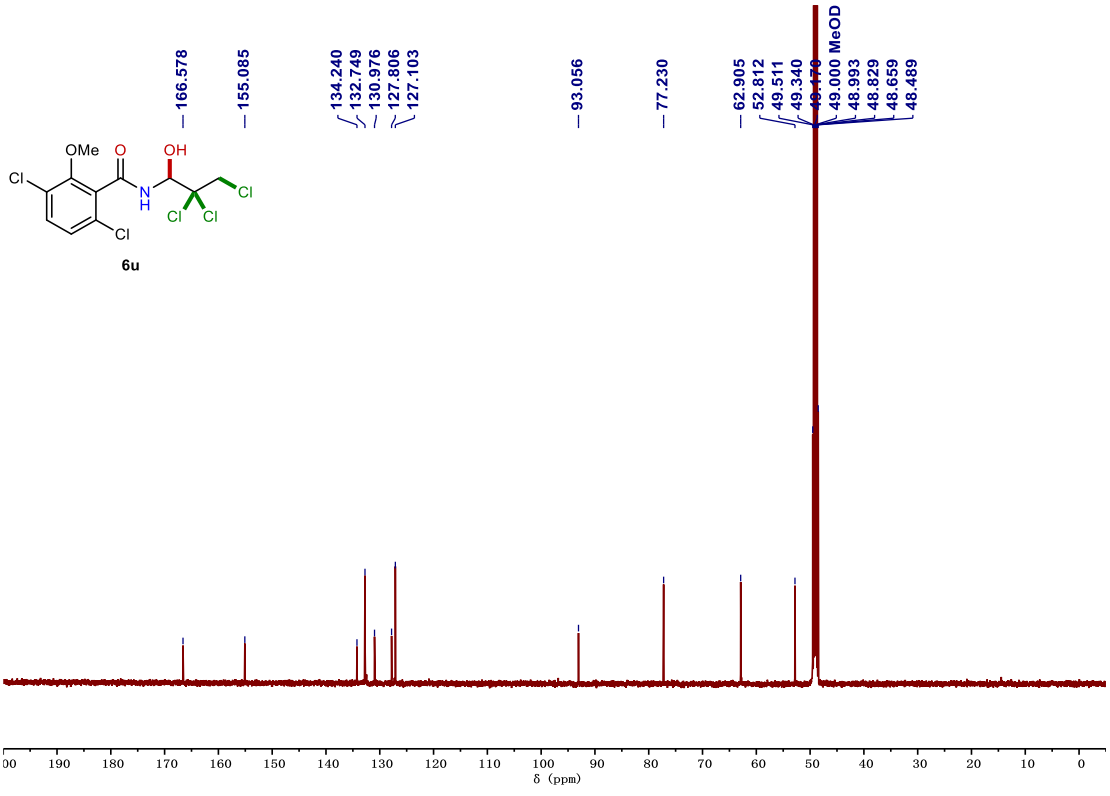
140.693
130.166
129.882
129.487
129.423
128.132
93.262
76.838
58.736
52.633
49.510
49.341
49.170
49.000 MeOD
48.830
48.659
48.490
Ph
O
OH
N
H
Cl
Cl
Cl
Ph
6t
δ (ppm)


7.474
7.456
7.240
7.222
6.139
4.853
4.327
4.303
4.201
4.177
3.925
3.314 MeOD
3.309
OMe
O
OH
Cl
N
H
Cl
Cl
Cl
Cl
6u
1.03
1.06
1.00
1.08
1.08
3.09
δ (ppm)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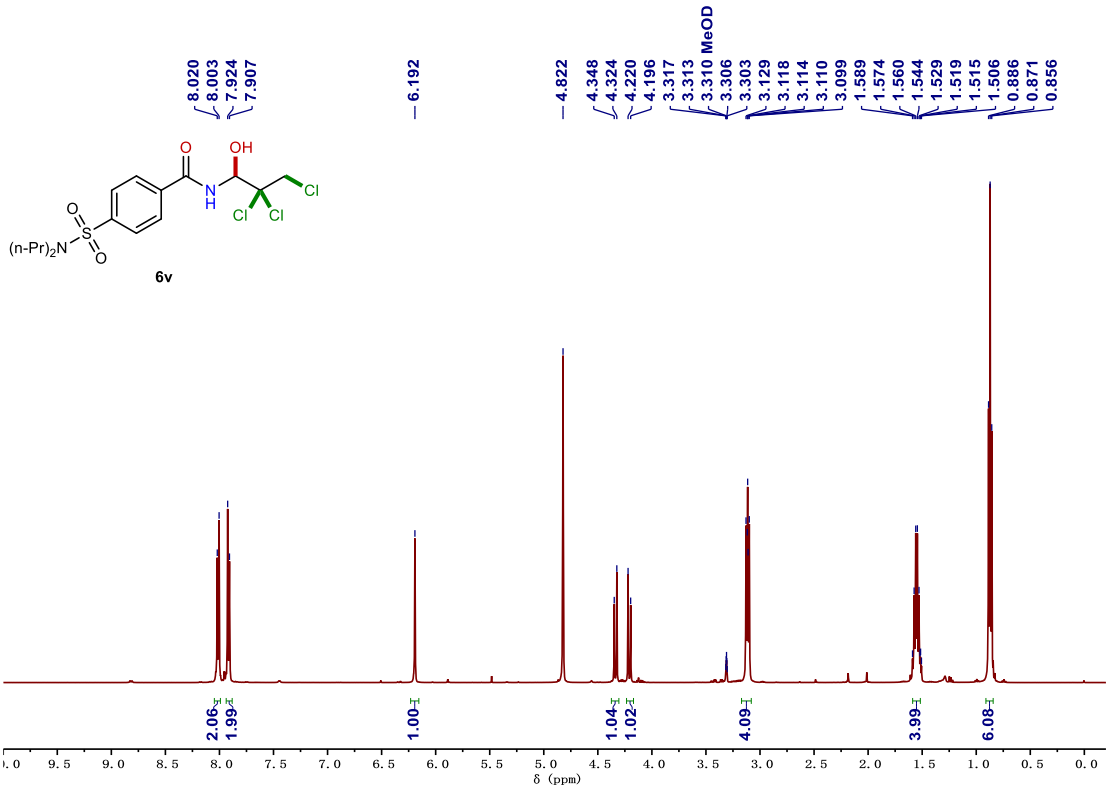
134.240
132.749
130.976
127.806
127.103
93.056
77.230
62.905
52.812
49.511
49.340
49.170
49.000 MeOD
48.993
48.829
48.659
48.489
OMe
O
OH
Cl
N
H
Cl
Cl
Cl
Cl
6u
δ (ppm)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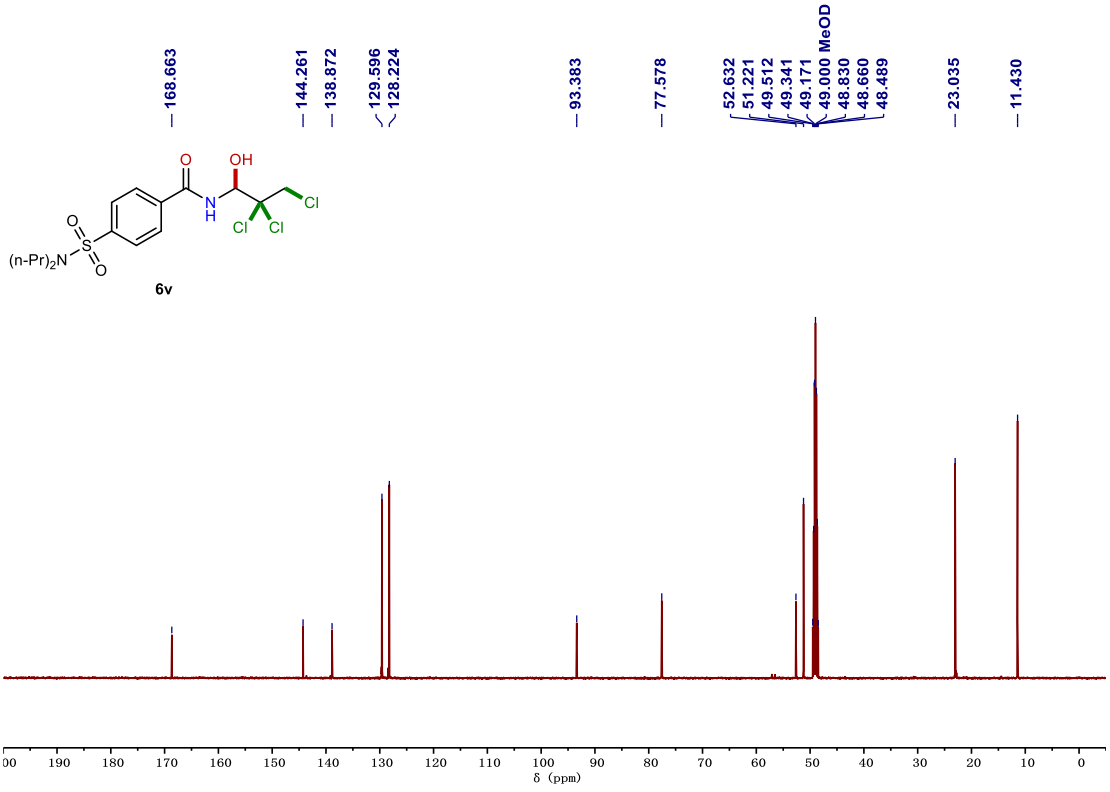
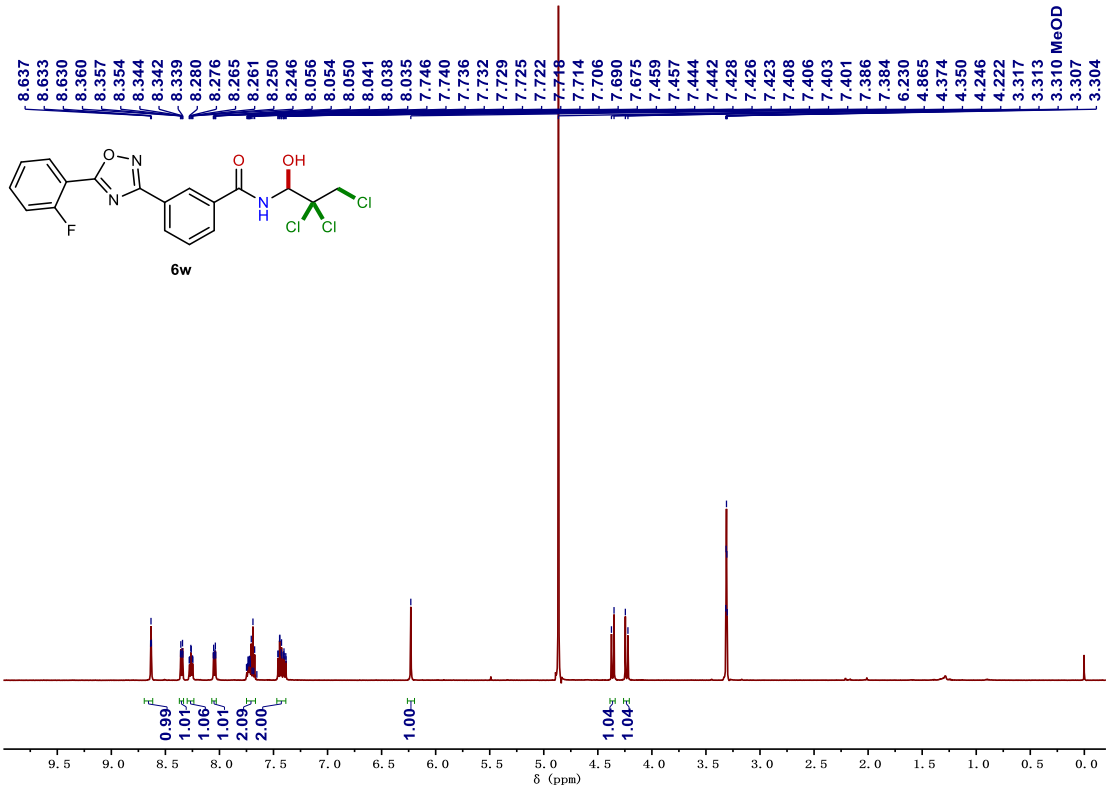
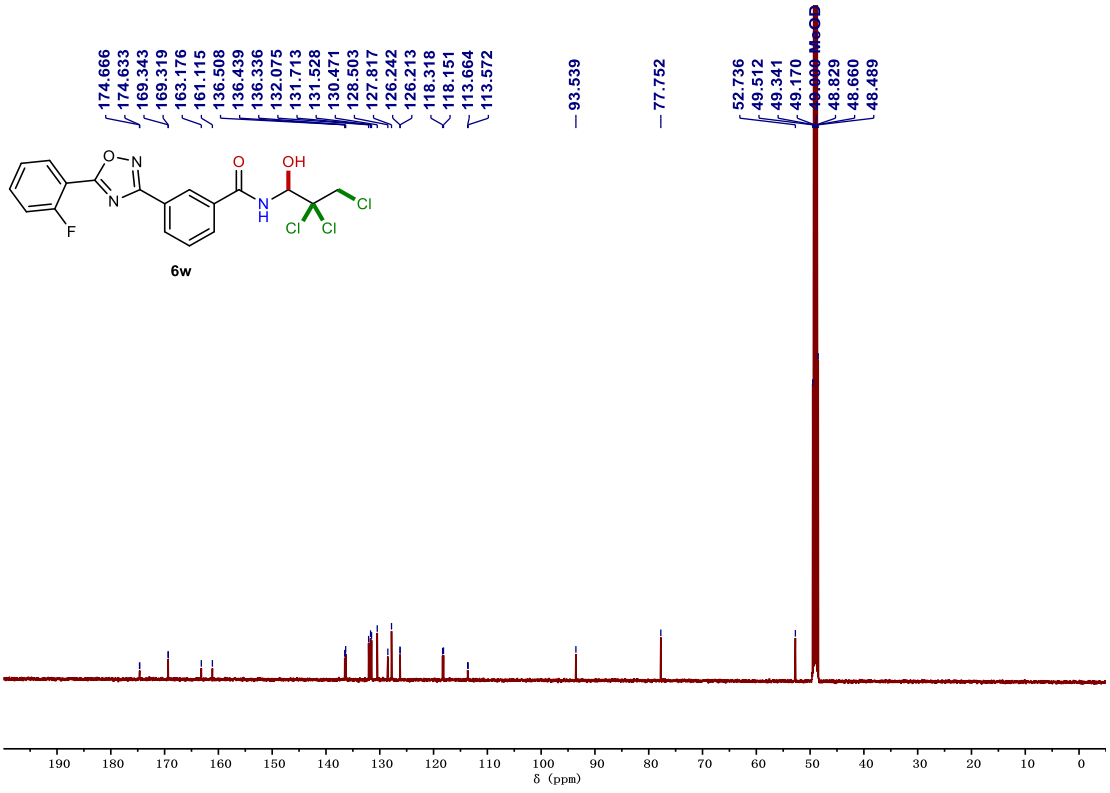
8.003
7.924
7.907
6.192
4.822
4.348
4.324
4.220
4.196
3.317
3.313
3.310 MeOD
3.306
3.303
3.129
3.118
3.114
3.110
3.099
1.589
1.574
1.560
1.544
1.529
1.519
1.515
1.506
0.886
0.871
0.856
O
OH
N
H
Cl
Cl
Cl
O
S
O
(n-Pr)2N
6v
2.06
1.99
1.00
1.04
1.02
4.09
3.99
6.08
δ (ppm)


168.663
144.261
138.872
129.596
128.224
93.383
77.578
52.632
51.221
49.512
49.341
49.171
49.000 MeOD
48.830
48.660
48.489
23.035
11.430
O
OH
N
H
Cl
Cl
Cl
O
S
O
(n-Pr)2N
6v
δ (ppm)


8.637
8.633
8.630
8.360
8.357
8.354
8.344
8.342
8.339
8.280
8.276
8.265
8.261
8.250
8.246
8.056
8.054
8.050
8.041
8.038
8.035
7.746
7.740
7.736
7.732
7.729
7.725
7.722
7.718
7.714
7.706
7.690
7.675
7.459
7.457
7.444
7.442
7.428
7.426
7.423
7.408
7.406
7.403
7.401
7.386
7.384
6.230
4.865
4.374
4.350
4.246
4.222
3.317
3.313
3.310 MeOD
3.307
3.304
O
N
N
F
O
OH
N
H
Cl
Cl
Cl
6w
0.99
1.01
1.06
1.01
2.09
2.00
1.00
1.04
1.04
δ (ppm)


174.666
174.633
169.343
169.319
163.176
161.115
136.508
136.439
136.336
132.075
131.713
131.528
130.471
128.503
127.817
126.242
126.213
118.318
118.151
113.664
113.572
93.539
77.752
52.736
49.512
49.341
49.170
49.000 MeOD
48.829
48.660
48.489
O
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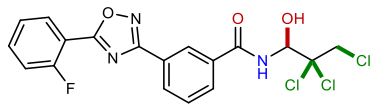
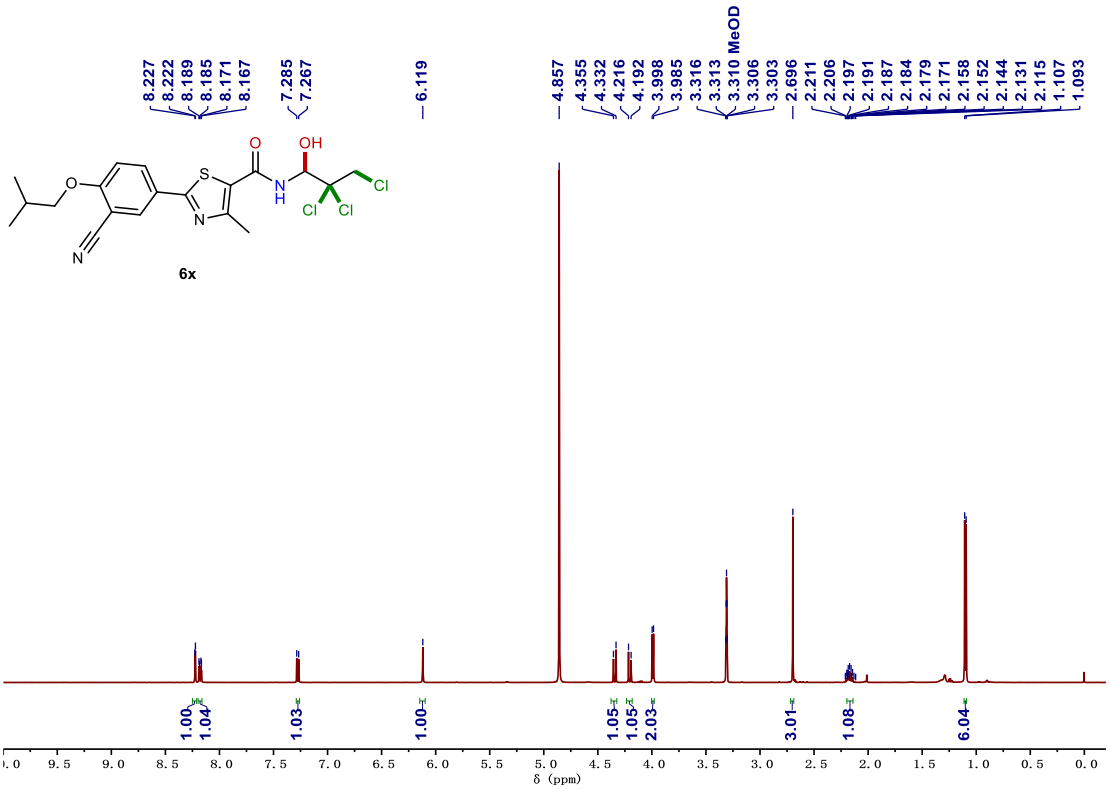
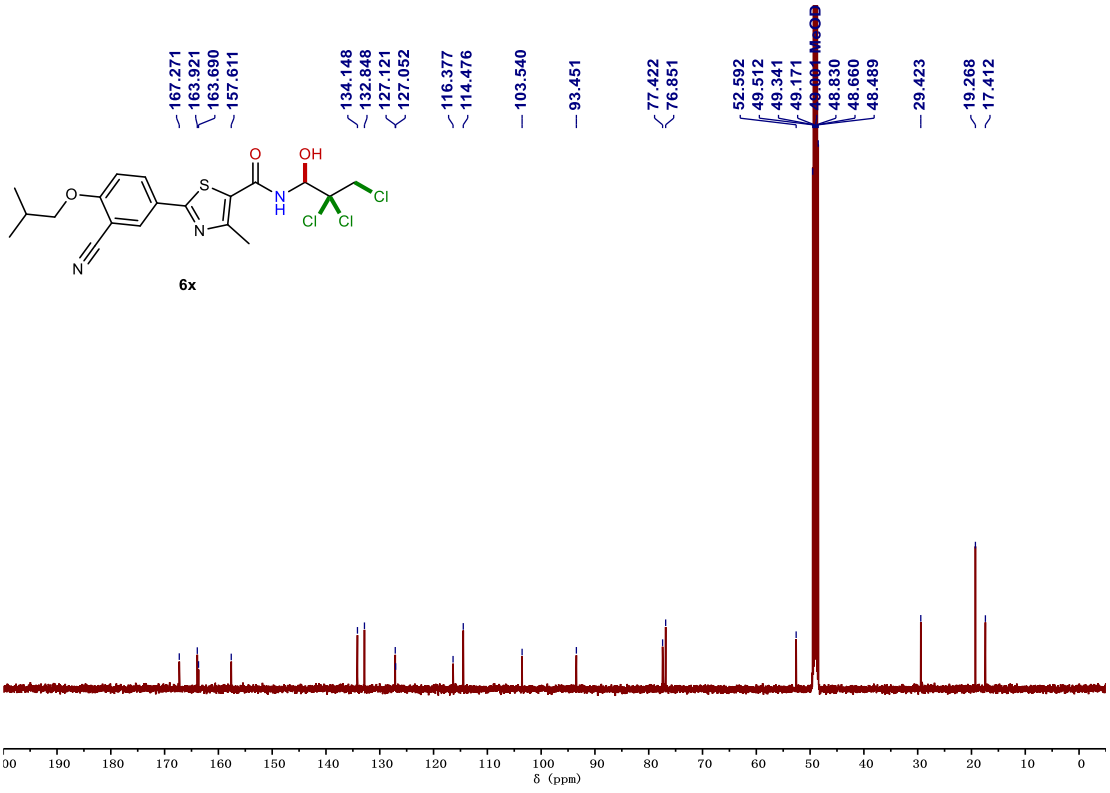
N
F
O
OH
N
H
Cl
Cl
Cl
6w
δ (ppm)

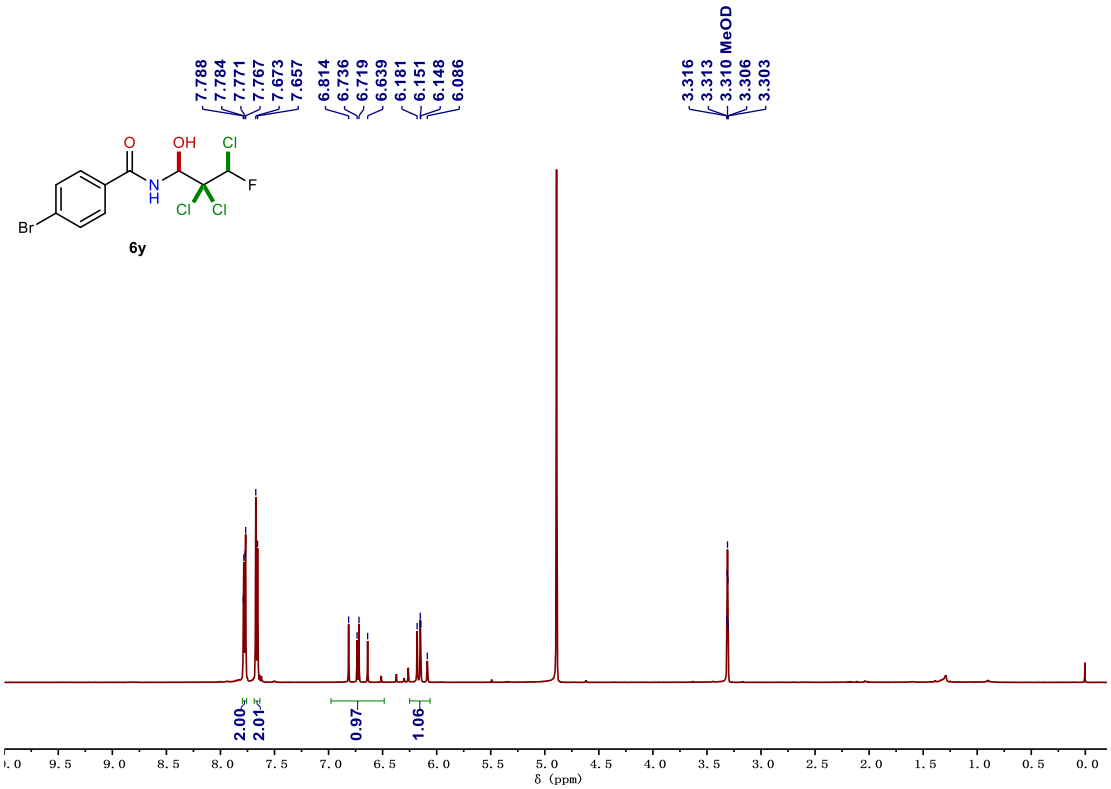

O
N
N
F
O
OH
N
H
Cl
Cl
Cl

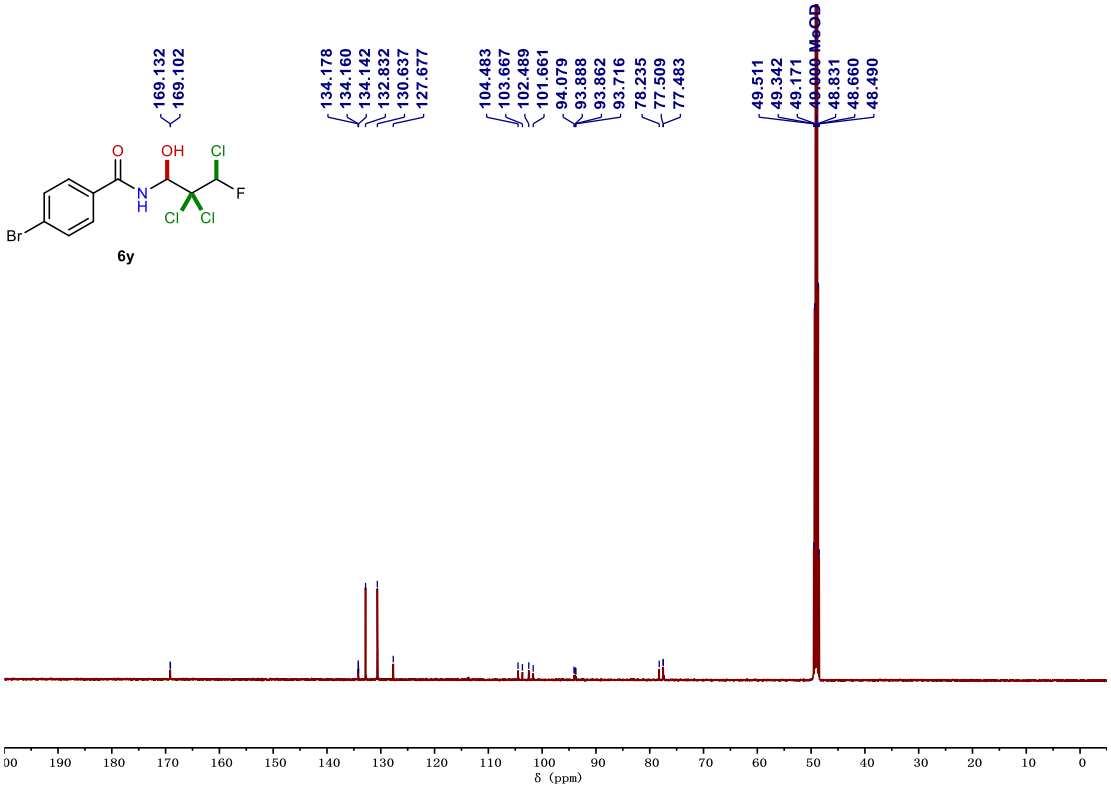
6w

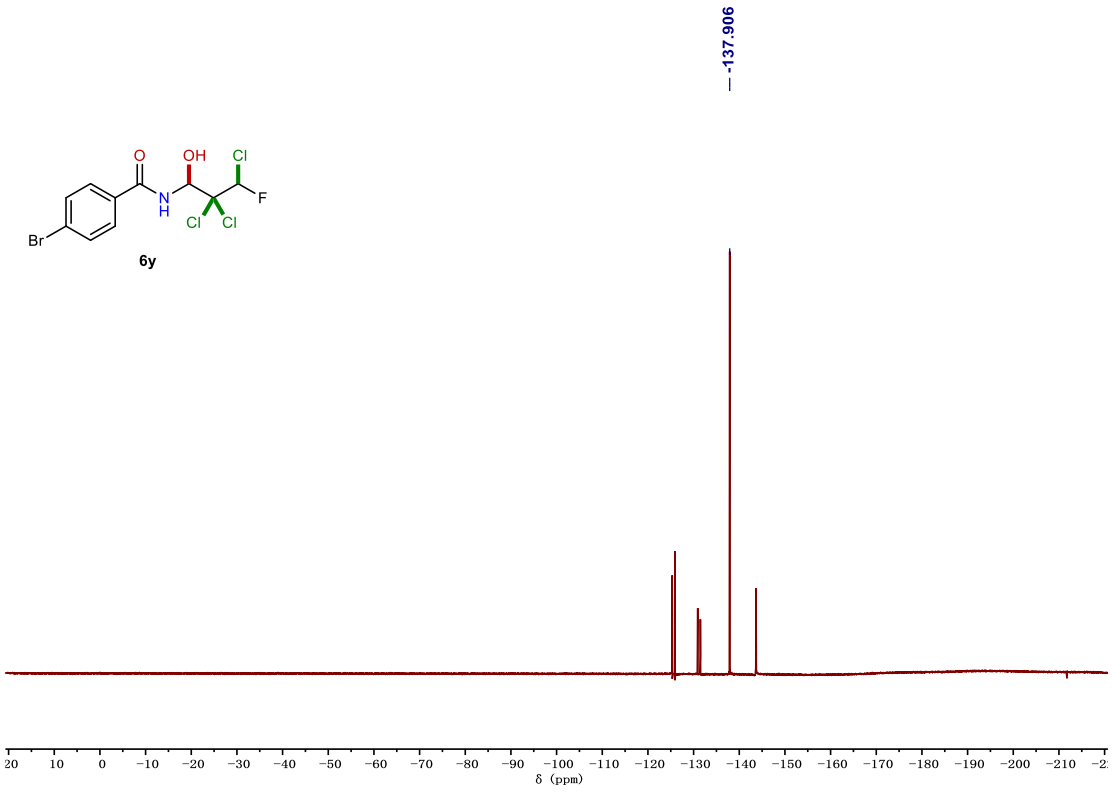
-110.735
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)

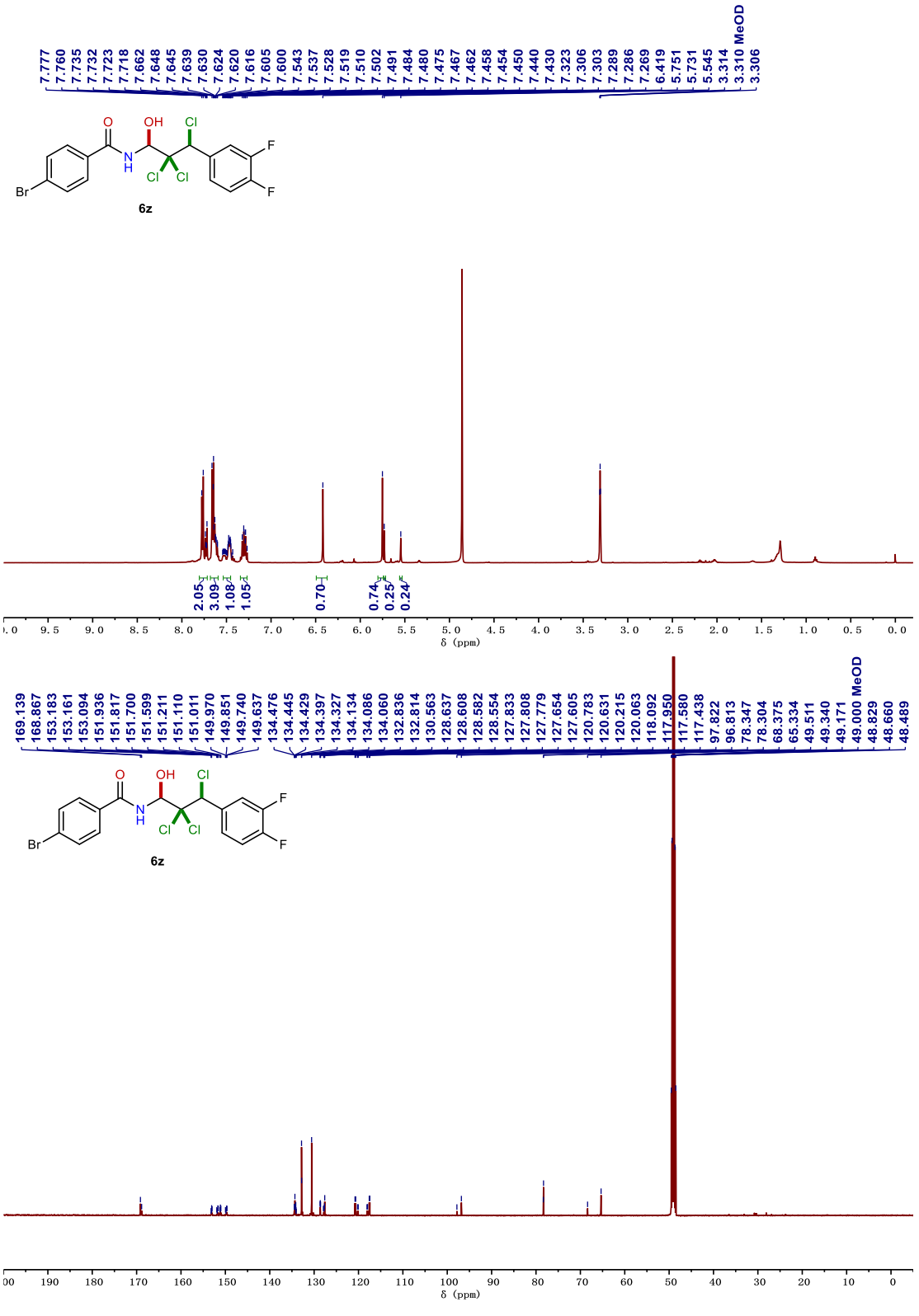

8.227
8.222
8.189
8.185
8.171
8.167
7.285
7.267
6.119
4.857
4.355
4.332
4.216
4.192
3.998
3.985
3.316
3.313
3.310 MeOD
3.306
3.303
2.696
2.211
2.206
2.197
2.191
2.187
2.184
2.179
2.171
2.158
2.152
2.144
2.131
2.115
1.107
1.093
O
OH
S
N
H
Cl
Cl
Cl
O
N
6x
1.00
1.04
1.03
1.00
1.05
1.05
2.03
3.01
1.08
6.04
δ (ppm)


167.271
163.921
163.690
157.611
134.148
132.848
127.121
127.052
116.377
114.476
103.540
93.451
77.422
76.851
52.592
49.512
49.341
49.171
48.830
48.660
48.489
29.423
19.268
17.412
O
OH
S
N
H
Cl
Cl
Cl
O
N
6x
δ (ppm)


7.788
7.784
7.771
7.767
7.673
7.657
6.814
6.736
6.719
6.639
6.181
6.151
6.148
6.086
3.316
3.313
3.310 MeOD
3.306
3.303
O
OH
Cl
N
H
F
Cl
Cl
Br
6y
2.00
2.01
0.97
1.06
δ (ppm)


169.132
169.102
134.178
134.160
134.142
132.832
130.637
127.677
104.483
103.667
102.489
101.661
94.079
93.888
93.862
93.716
78.235
77.509
77.483
49.511
49.342
49.171
49.000 MeOD
48.831
48.660
48.490
O
OH
Cl
N
H
F
Cl
Cl
Br
6y
δ (ppm)


-137.906
O
OH
Cl
N
H
F
Cl
Cl
Br
6y
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)


7.777
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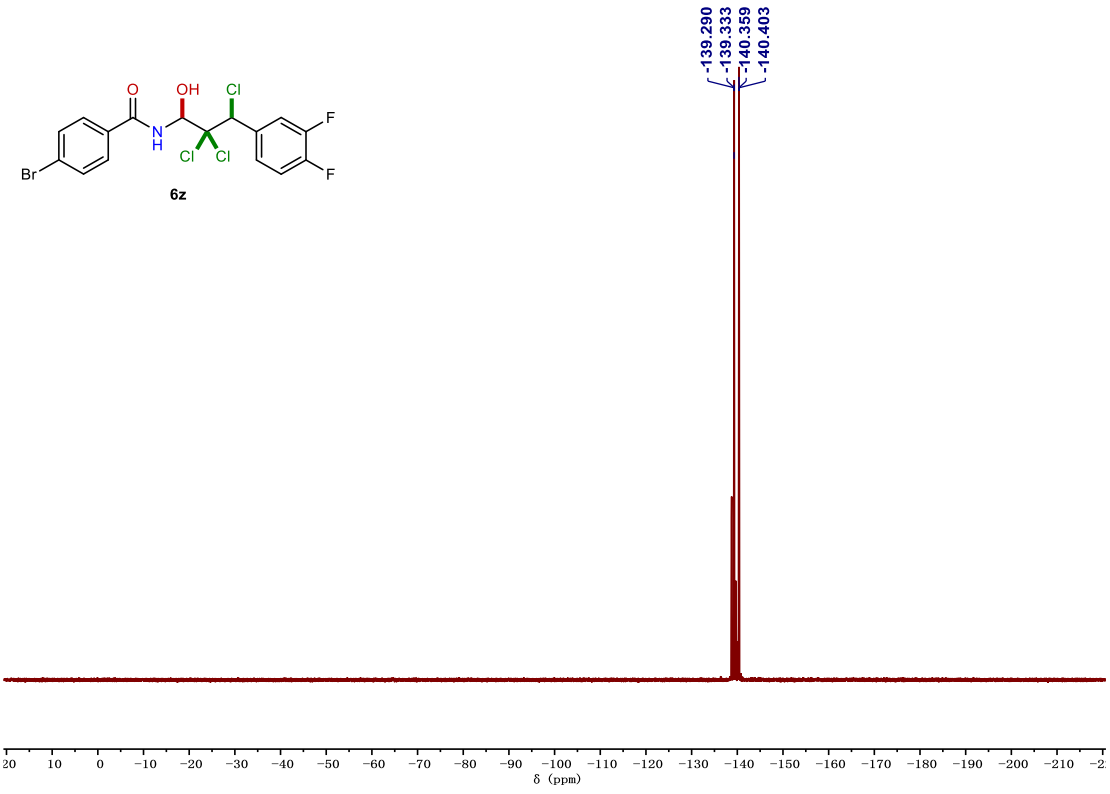
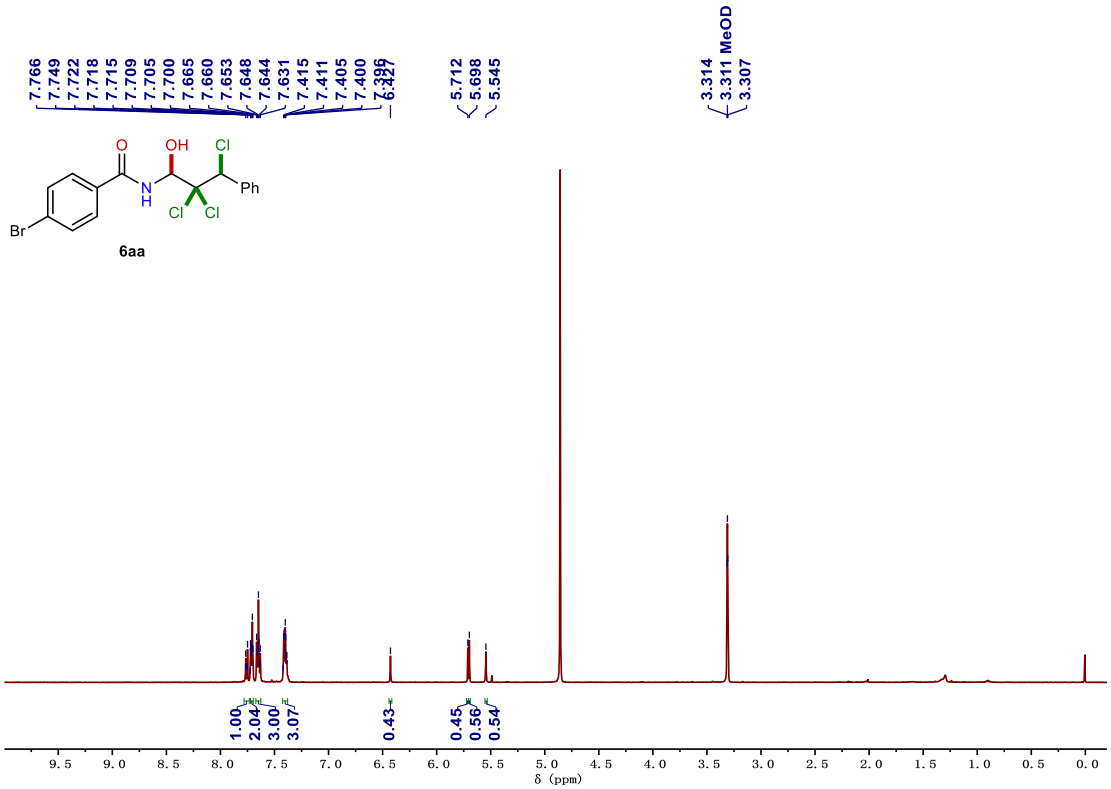
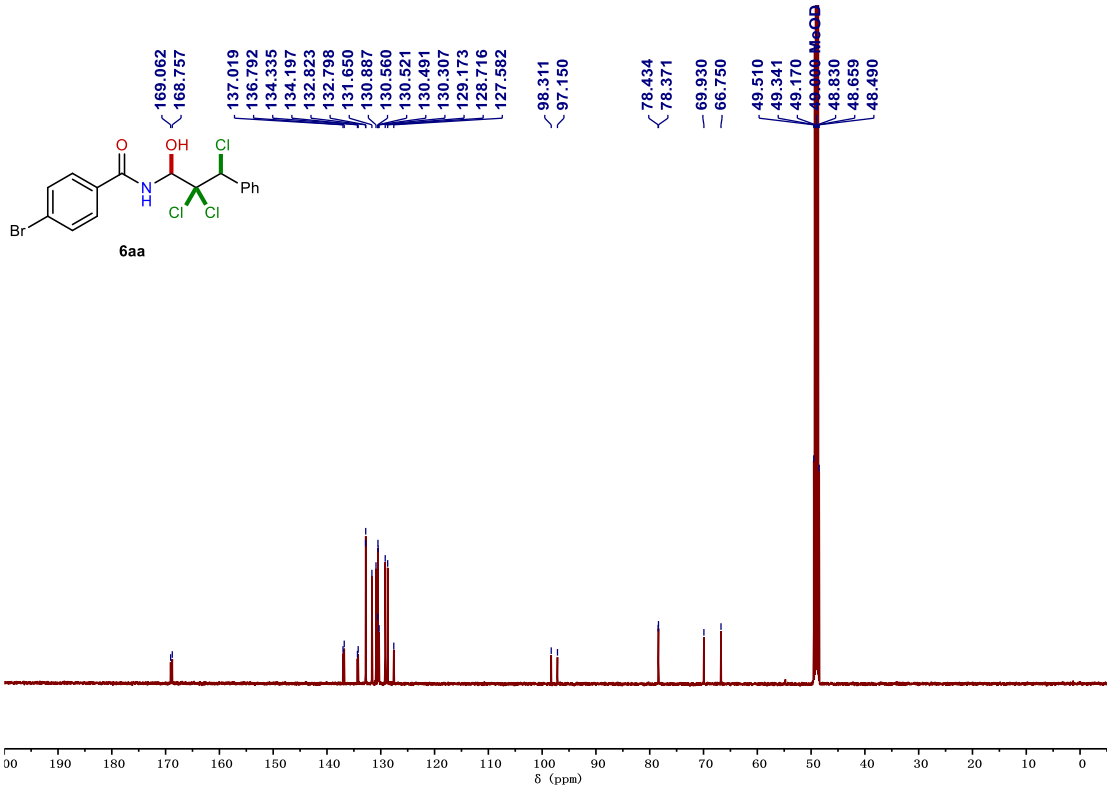
7.735
7.732
7.723
7.718
7.662
7.648
7.645
7.639
7.630
7.624
7.620
7.616
7.605
7.600
7.543
7.537
7.528
7.519
7.510
7.502
7.491
7.484
7.480
7.475
7.467
7.462
7.458
7.454
7.450
7.440
7.430
7.323
7.306
7.303
7.289
7.286
7.269
6.419
5.751
5.731
5.545
3.314
3.310 MeOD
3.306
O
OH
Cl
N
H
Cl
Cl
Br
F
F
6z
2.05
3.09
1.08
1.05
0.70
0.74
0.25
0.24
δ (ppm)
169.139
168.867
153.183
153.161
153.094
151.936
151.817
151.700
151.599
151.211
151.110
151.011
149.970
149.851
149.740
149.637
134.476
134.445
134.429
134.397
134.327
134.134
134.086
134.060
132.836
132.814
130.563
128.637
128.608
128.582
128.554
127.833
127.808
127.779
127.654
127.605
120.783
120.631
120.215
120.063
118.092
117.950
117.580
117.438
97.822
96.813
78.347
78.304
68.375
65.334
49.511
49.340
49.171
49.000 MeOD
48.829
48.660
48.489
O
OH
Cl
N
H
Cl
Cl
Br
F
F
6z
δ (ppm)


-139.290
-139.333
-140.359
-140.403
O
OH
Cl
N
H
Cl
Cl
Br
F
F
6z
δ (ppm)


7.766
7.749
7.722
7.718
7.715
7.709
7.705
7.700
7.665
7.660
7.653
7.648
7.644
7.631
7.415
7.411
7.405
7.400
5.712
5.698
5.545
3.314
3.311 MeOD
3.307
1.00
2.04
3.00
3.07
0.43
0.45
0.56
0.54
δ (ppm)

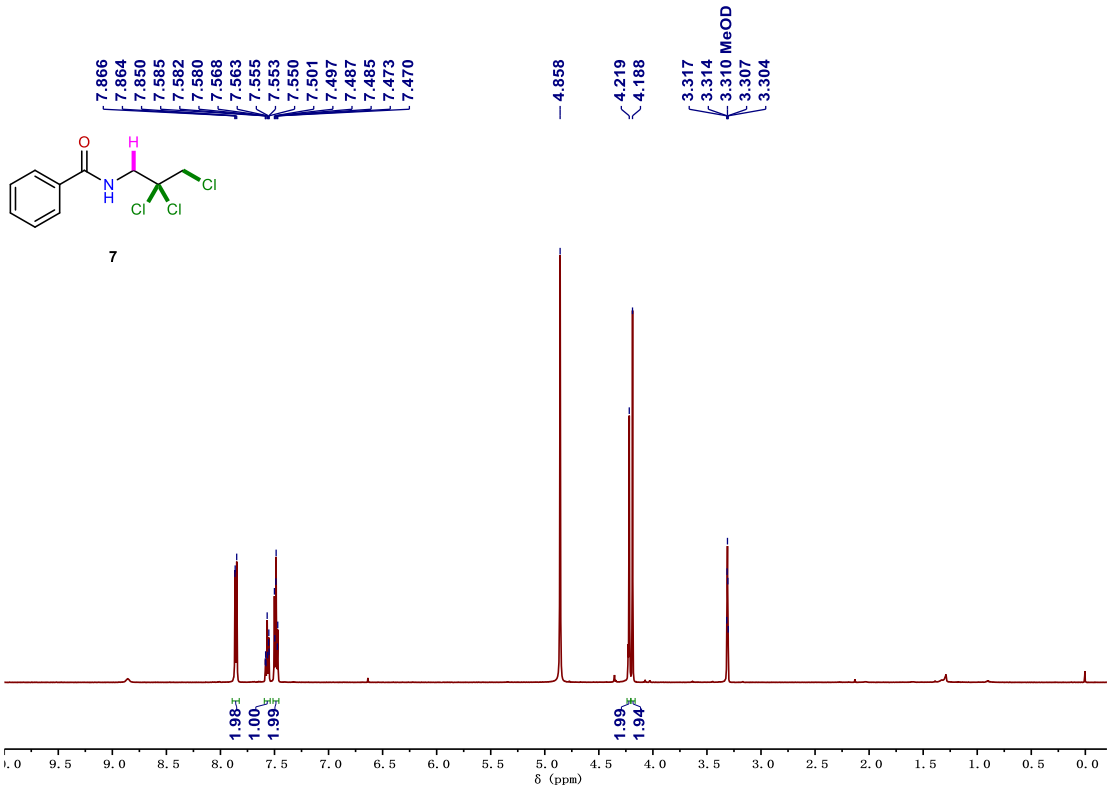

169.062
168.757
137.019
136.792
134.335
134.197
132.823
132.798
131.650
130.887
130.560
130.521
130.491
130.307
129.173
128.716
127.582
98.311
97.150
78.434
78.371
69.930
66.750
49.510
49.341
49.170
49.000 MeOD
48.830
48.659
48.490
δ (ppm)

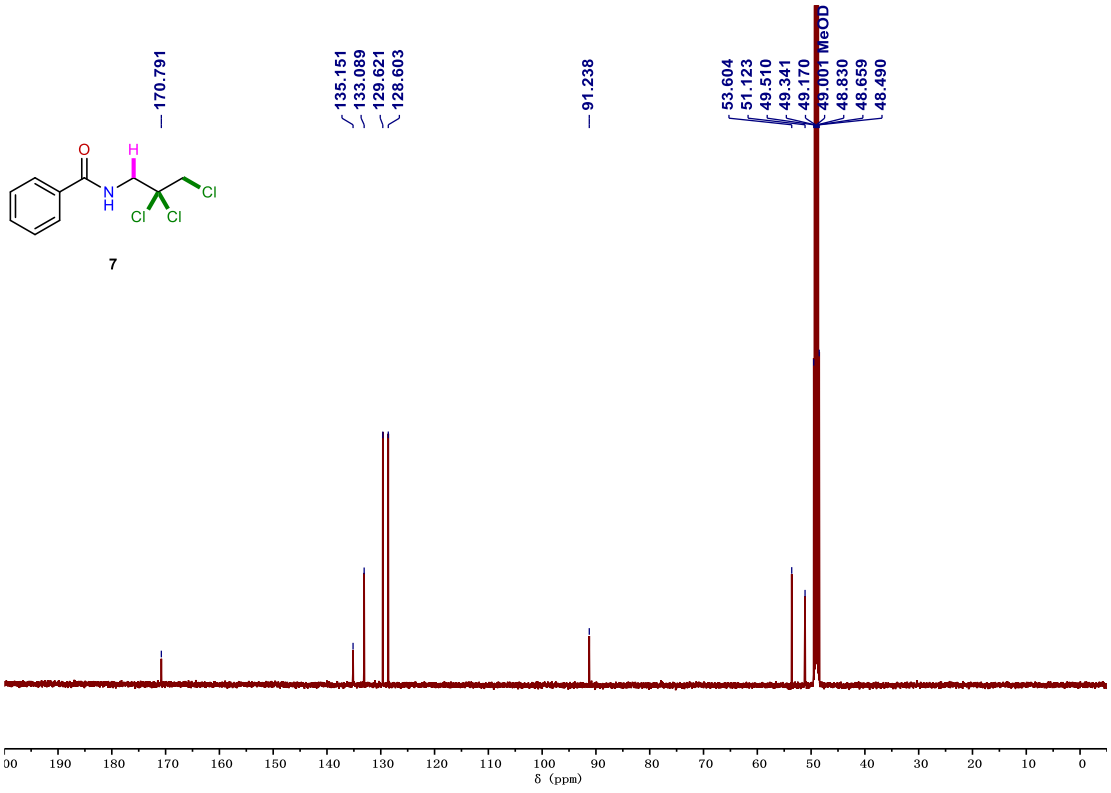
O
OH
Cl
N
H
Ph
Cl
Cl
Br

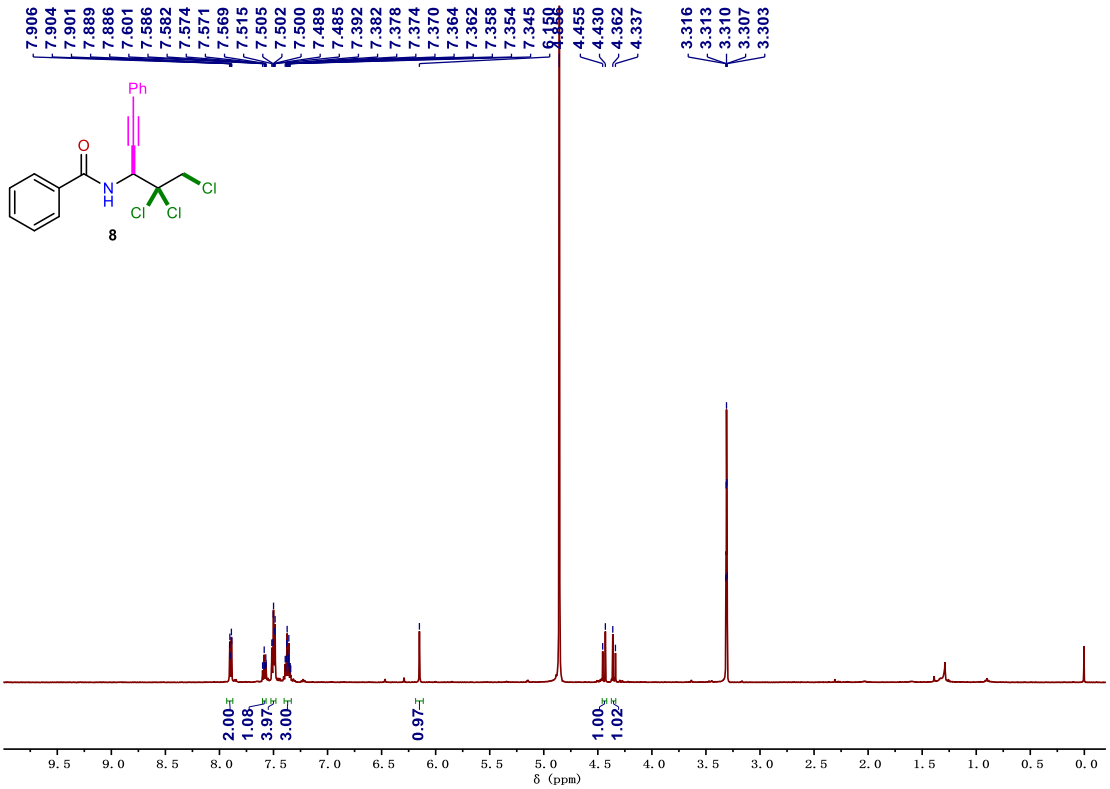
6aa

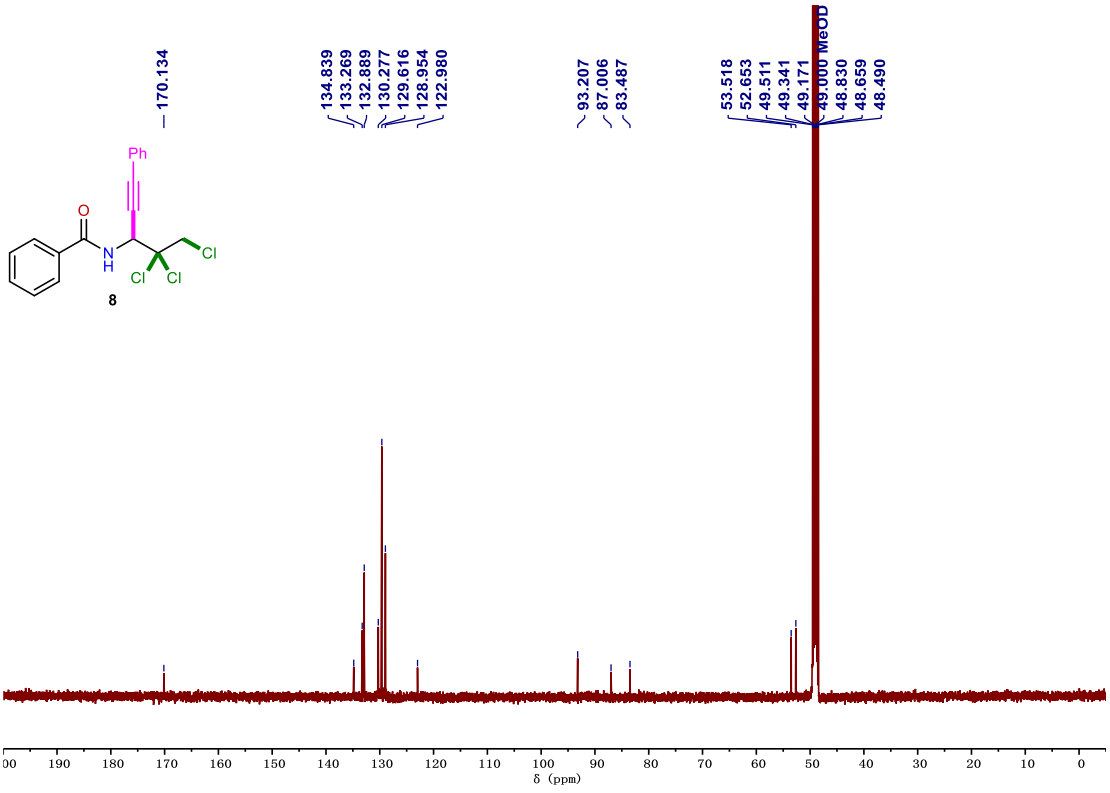
O
OH
Cl
N
H
Ph
Cl
Cl
Br

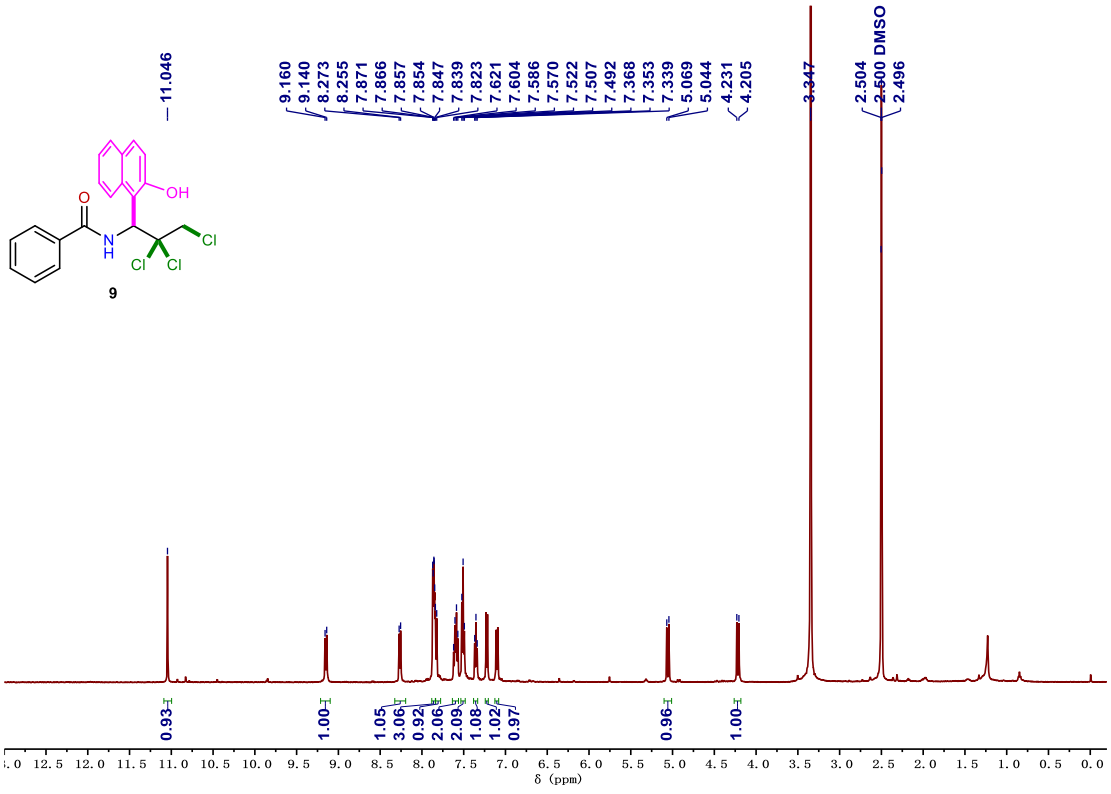
6aa

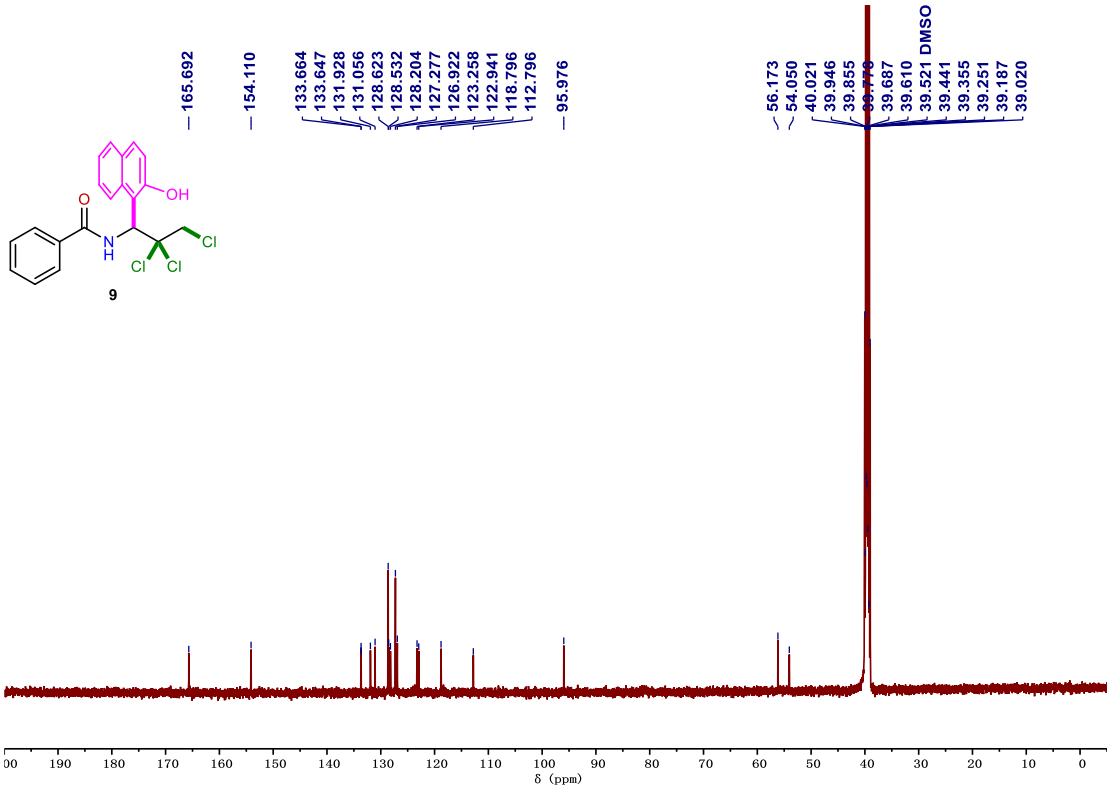

7.866
7.864
7.850
7.585
7.582
7.580
7.568
7.563
7.555
7.553
7.550
7.501
7.497
7.487
7.485
7.473
7.470
4.858
4.219
4.188
3.317
3.314
3.310 MeOD
3.307
3.304
O
H
N
H
Cl
Cl
Cl
7
1.98
1.00
1.99
1.99
1.94
δ (ppm)

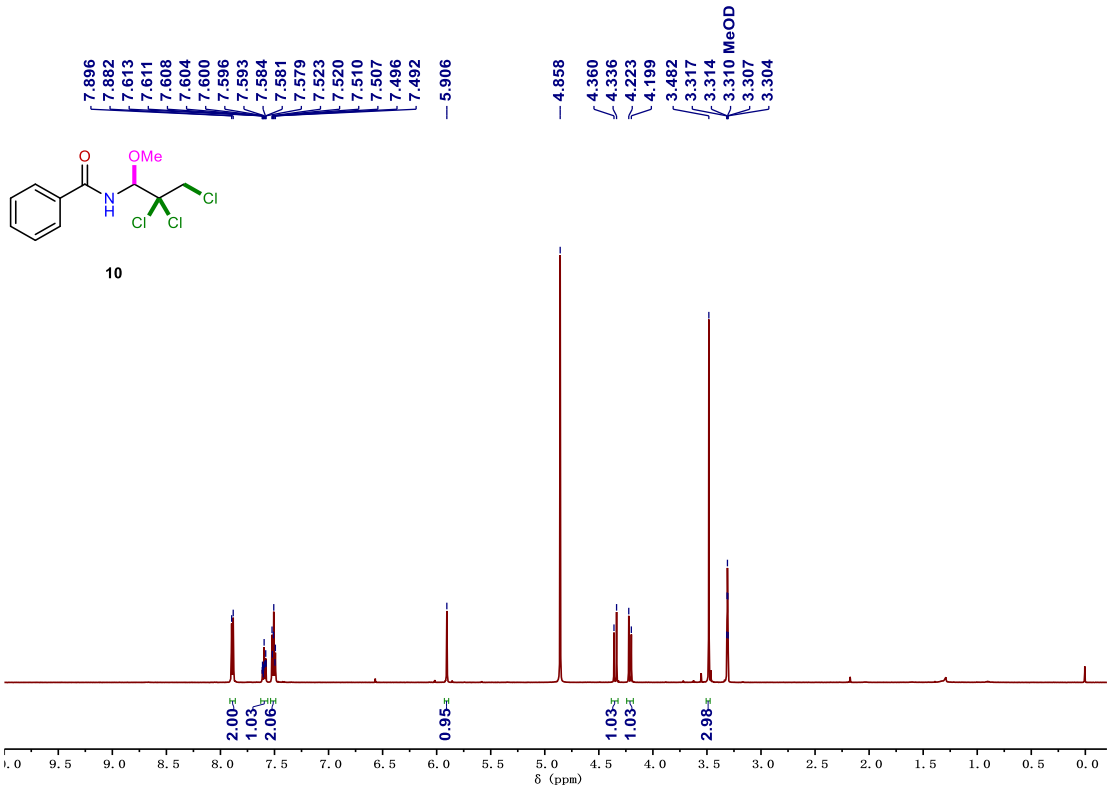

170.791
135.151
133.089
129.621
128.603
91.238
53.604
51.123
49.510
49.341
49.170
49.001 MeOD
48.830
48.659
48.490
O
H
N
H
Cl
Cl
Cl
7
δ (ppm)

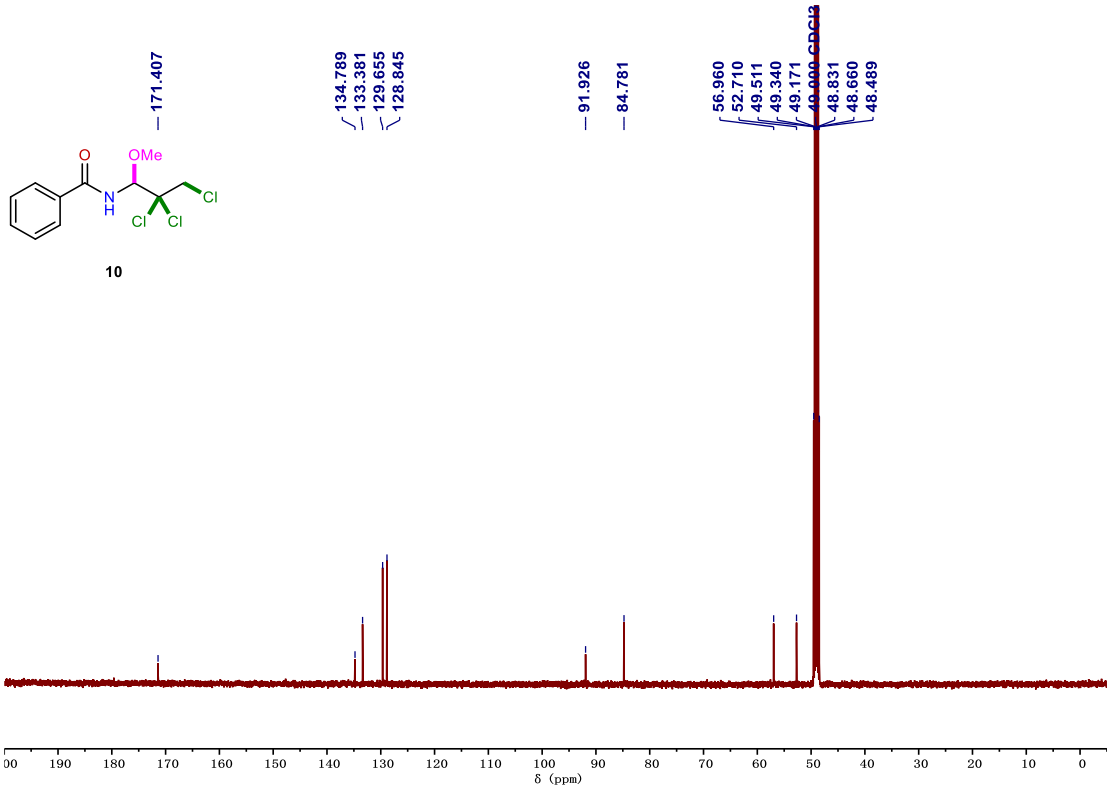

7.906
7.904
7.901
7.889
7.886
7.601
7.586
7.582
7.574
7.571
7.569
7.515
7.505
7.502
7.500
7.489
7.485
7.392
7.382
7.378
7.374
7.370
7.364
7.362
7.358
7.354
7.345
4.455
4.430
4.362
4.337
3.316
3.313
3.310
3.307
3.303
Ph
O
N
H
Cl
Cl
Cl
8
2.00
1.08
3.97
3.00
0.97
1.00
1.02
δ (ppm)

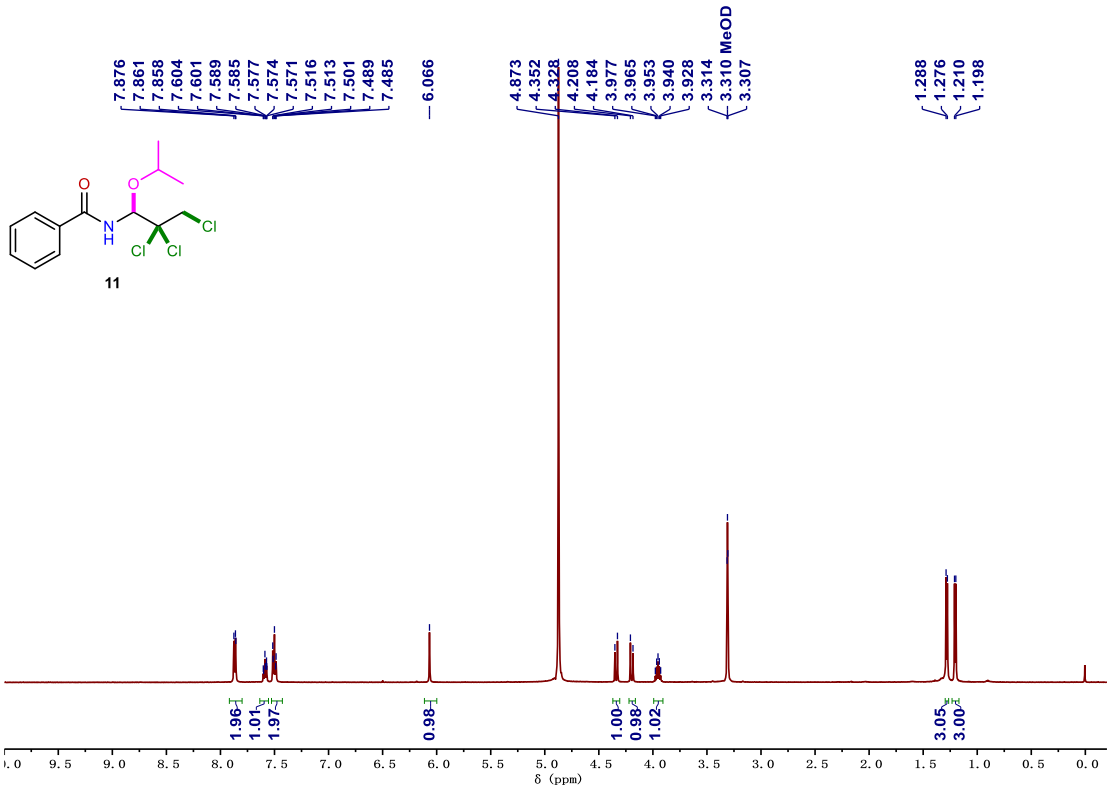

170.134
134.839
133.269
132.889
130.277
129.616
128.954
122.980
93.207
87.006
83.487
53.518
52.653
49.511
49.341
49.171
49.000 MeOD
48.830
48.659
48.490
Ph
O
N
H
Cl
Cl
Cl
8
δ (ppm)


11.046
9.160
9.140
8.273
8.255
7.871
7.866
7.857
7.854
7.847
7.839
7.823
7.621
7.604
7.586
7.570
7.522
7.507
7.492
7.368
7.353
7.339
5.069
5.044
4.231
4.205
3.347
2.504
2.500 DMSO
2.496
OH
O
N
H
Cl
Cl
Cl
9
0.93
1.00
1.05
3.06
0.92
2.06
2.09
1.08
1.02
0.97
0.96
1.00
δ (ppm)


165.692
154.110
133.664
133.647
131.928
131.056
128.623
128.532
128.204
127.277
126.922
123.258
122.941
118.796
112.796
95.976
56.173
54.050
40.021
39.946
39.855
39.687
39.610
39.521 DMSO
39.441
39.355
39.251
39.187
39.020
OH
O
N
H
Cl
Cl
Cl
9
δ (ppm)


7.896
7.882
7.613
7.611
7.608
7.604
7.600
7.596
7.593
7.584
7.581
7.579
7.523
7.520
7.510
7.507
7.496
7.492
5.906
4.858
4.360
4.336
4.223
4.199
3.482
3.317
3.314
3.310 MeOD
3.307
3.304
O
OMe
N
H
Cl
Cl
Cl
10
2.00
1.03
2.06
0.95
1.03
1.03
2.98
δ (ppm)


171.407
134.789
133.381
129.655
128.845
91.926
84.781
56.960
52.710
49.511
49.340
49.171
49.000 CDCl3
48.831
48.660
48.489
O
OMe
N
H
Cl
Cl
Cl
10
δ (ppm)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7.858
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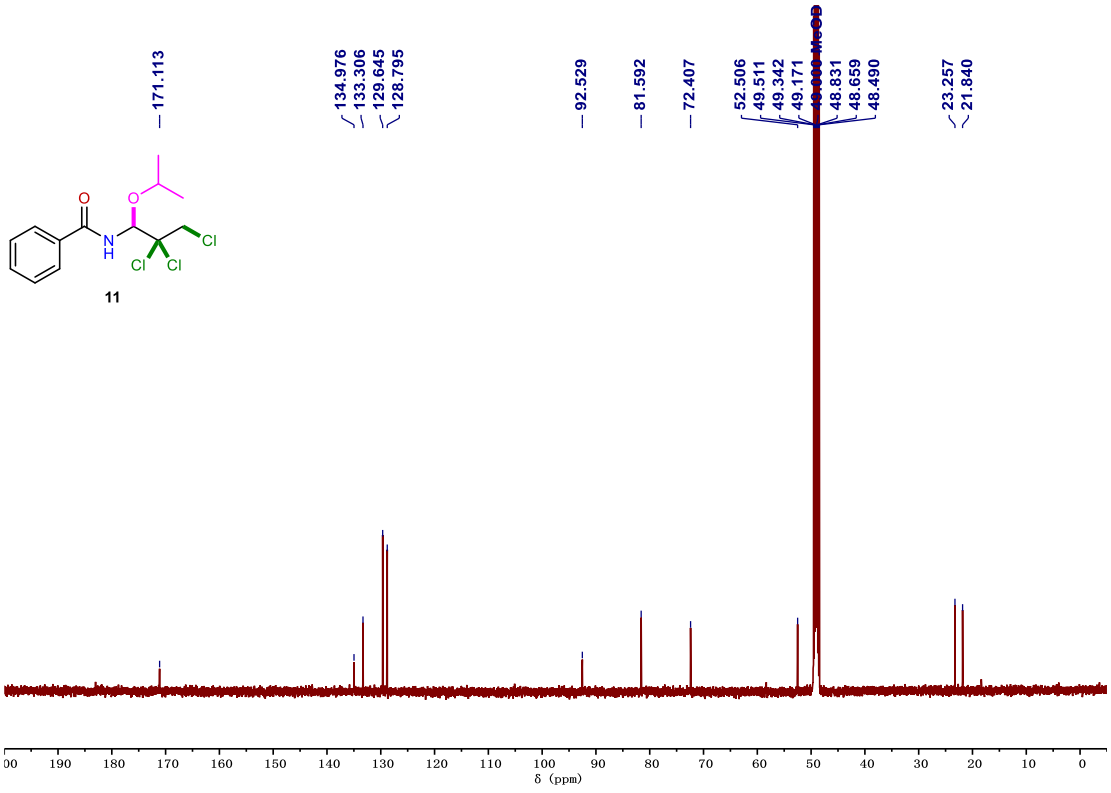
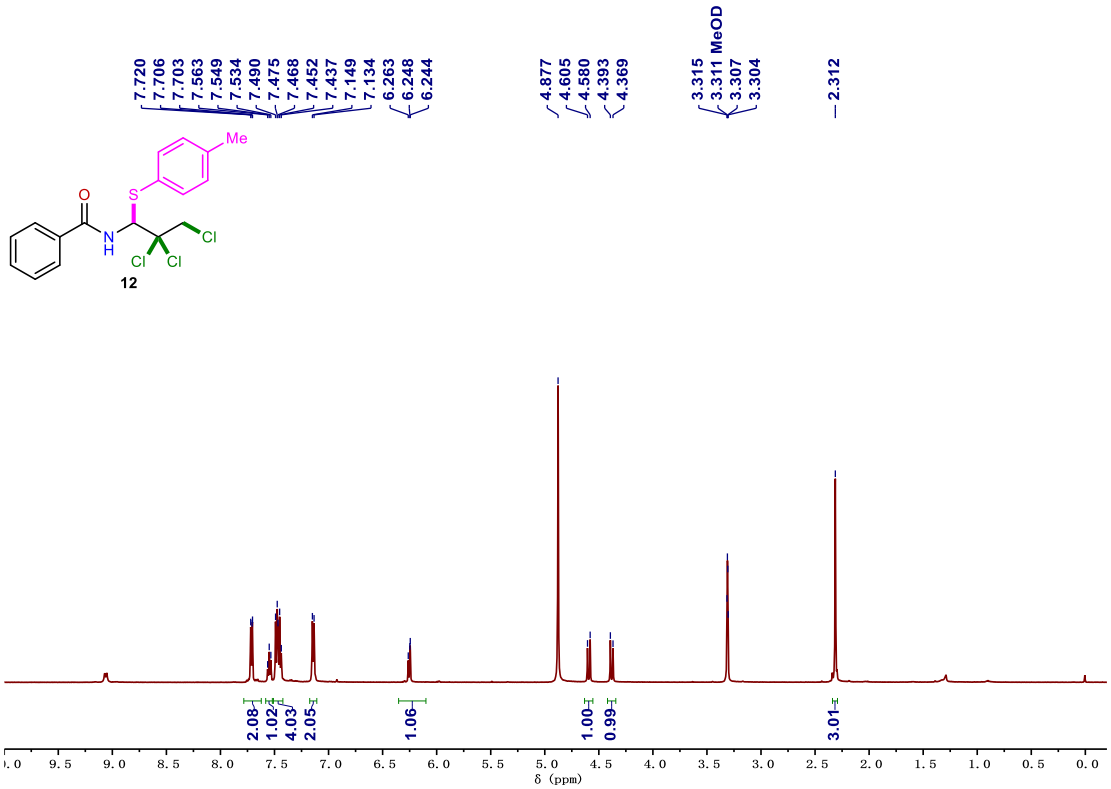
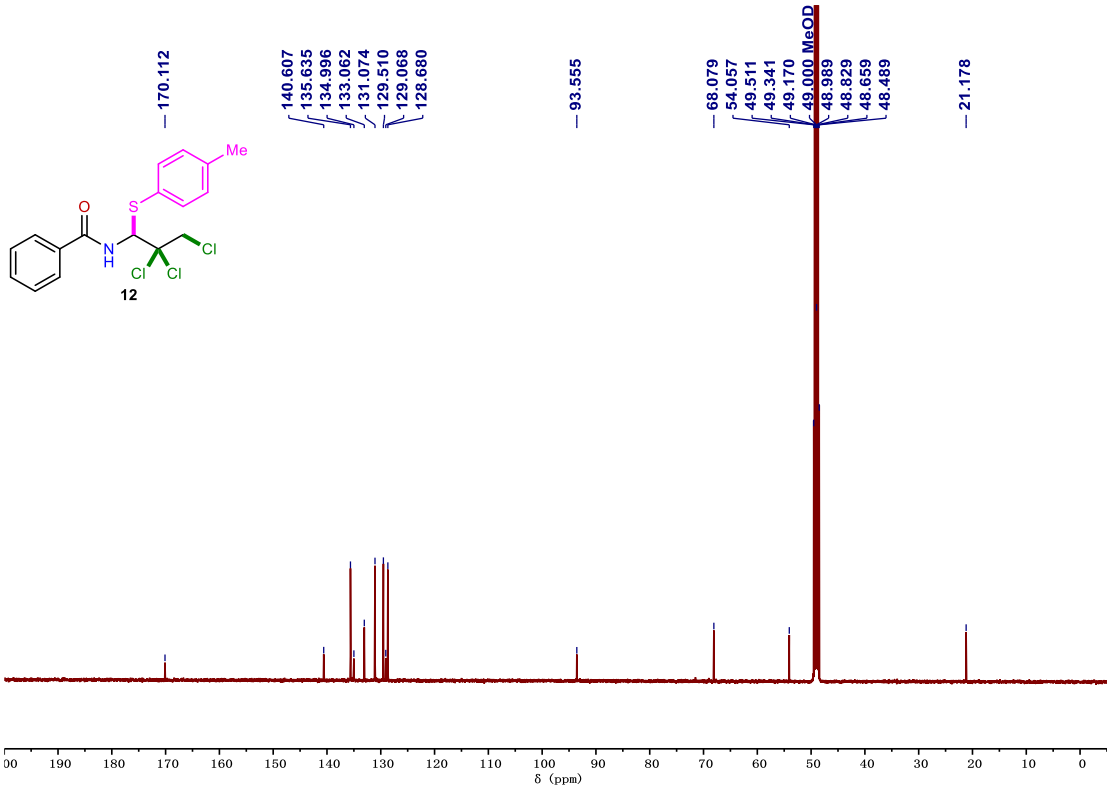
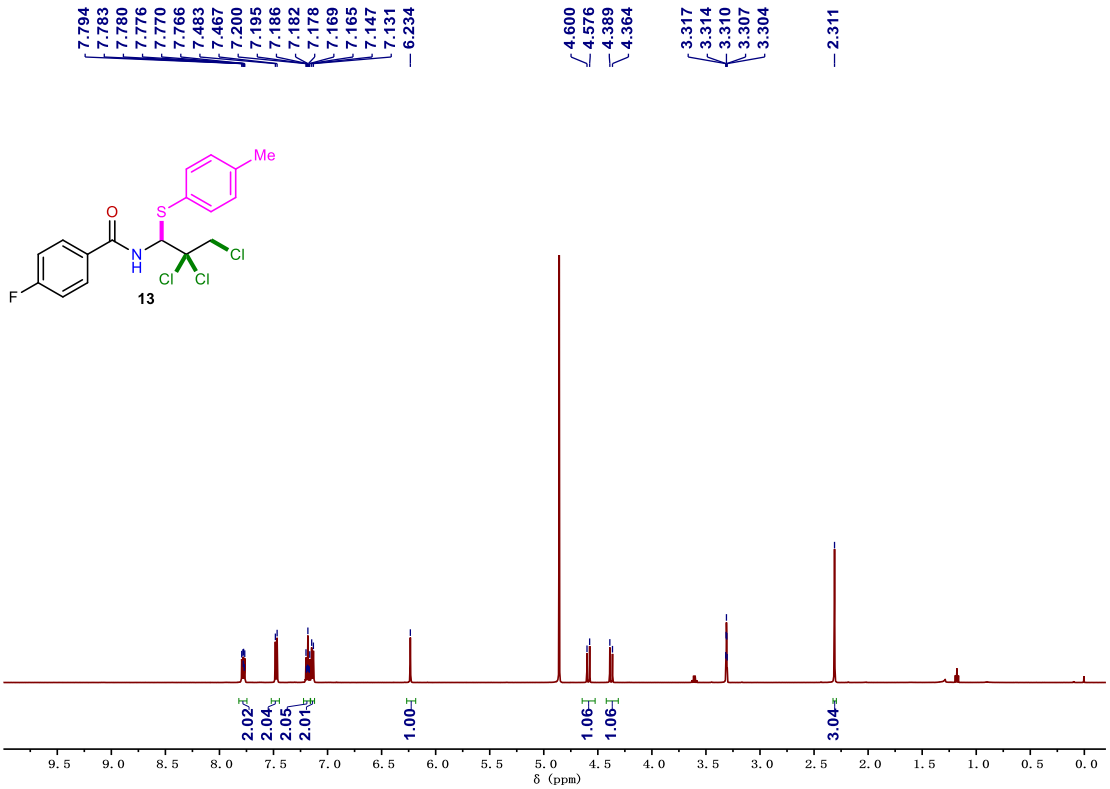
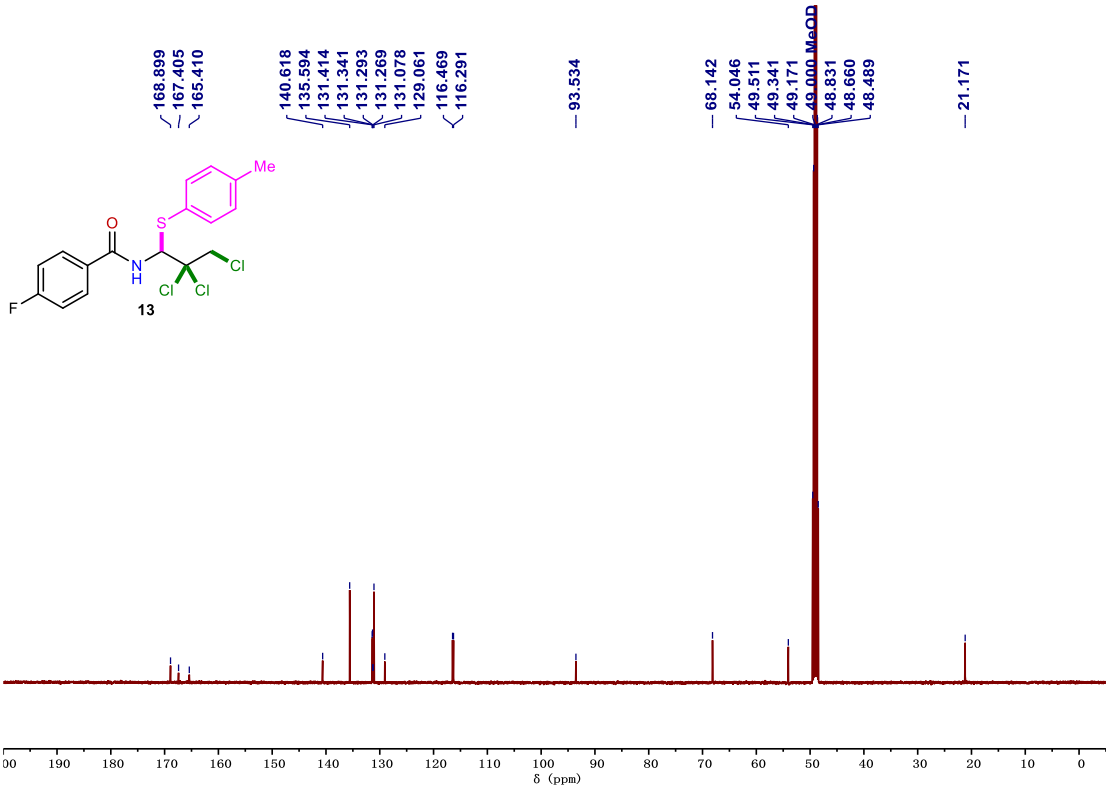
7.601
7.589
7.585
7.577
7.574
7.571
7.516
7.513
7.501
7.489
7.485
6.066
4.873
4.352
4.328
4.208
4.184
3.977
3.965
3.953
3.940
3.928
3.314
3.310 MeOD
3.307
1.288
1.276
1.210
1.198
1.96
1.01
1.97
0.98
1.00
0.98
1.02
3.05
3.00
δ (ppm)
O
O
N
H
Cl
Cl
Cl
11

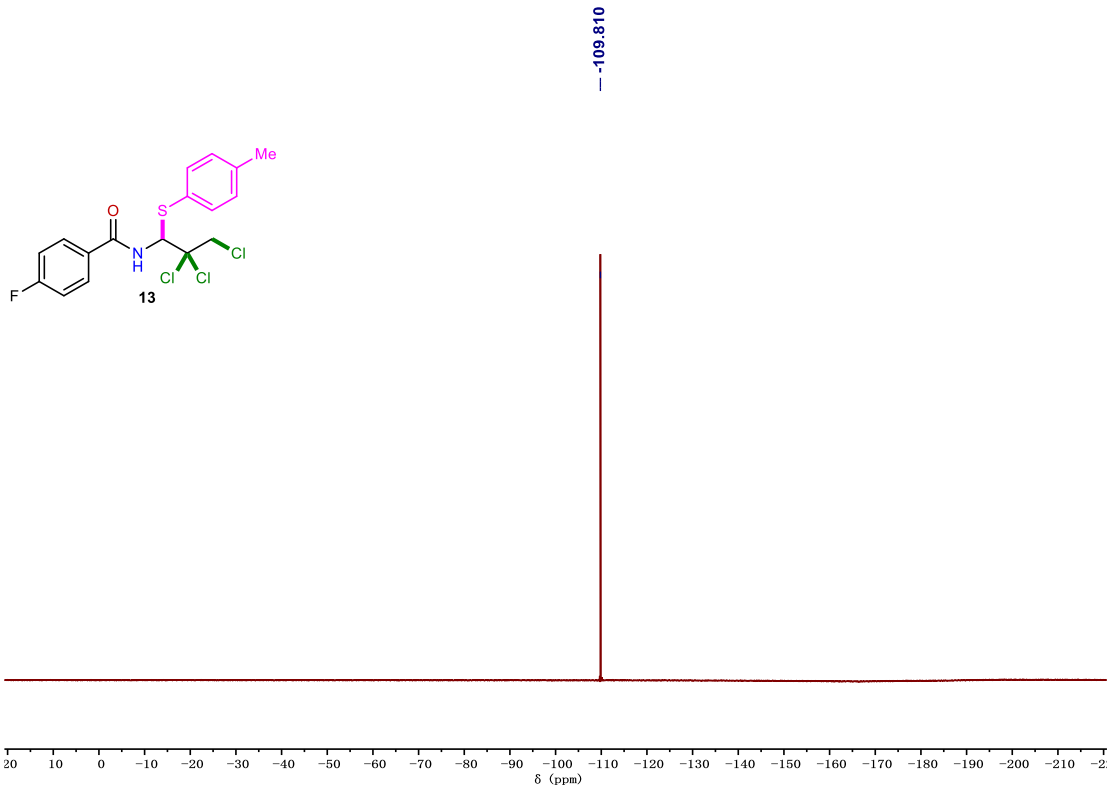

171.113
134.976
133.306
129.645
128.795
92.529
81.592
72.407
52.506
49.511
49.342
49.171
48.831
48.659
48.490
23.257
21.840
δ (ppm)
O
O
N
H
Cl
Cl
Cl
11

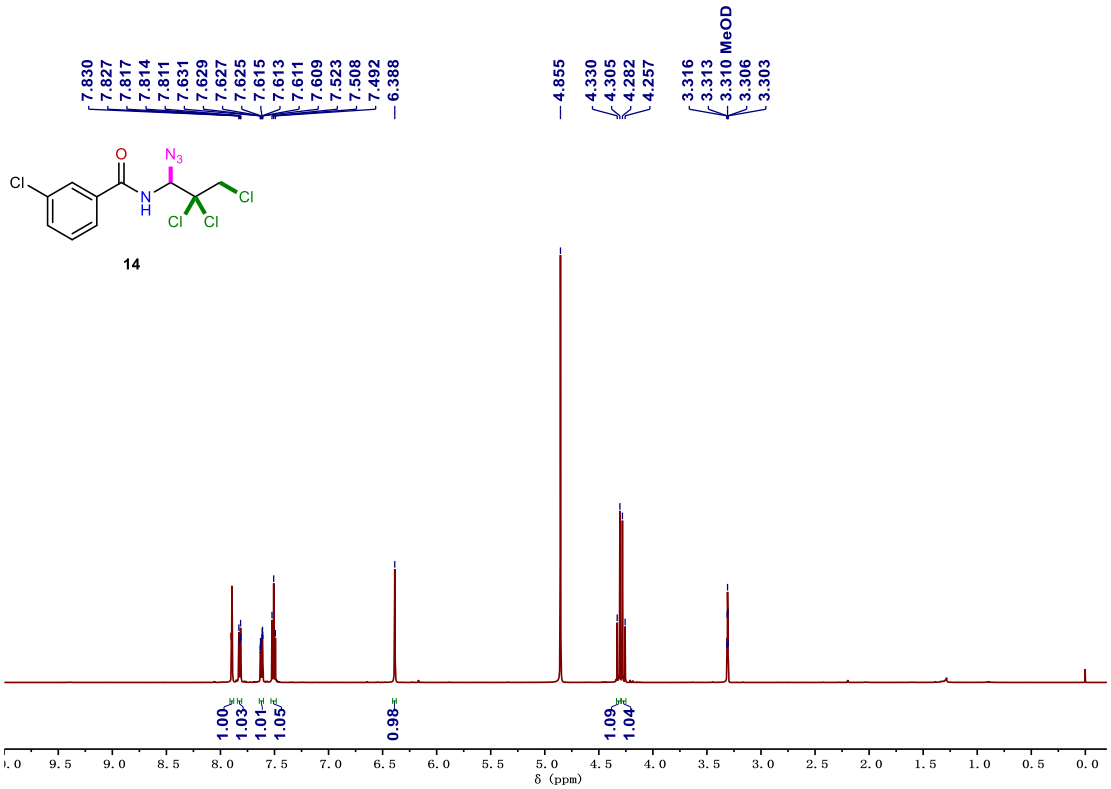

7.720
7.706
7.703
7.563
7.549
7.534
7.490
7.475
7.468
7.452
7.437
7.149
7.134
6.263
6.248
6.244
4.877
4.605
4.580
4.393
4.369
3.315
3.311 MeOD
3.307
3.304
2.312
Me
S
O
N
H
Cl
Cl
Cl
12
2.08
1.02
4.03
2.05
1.06
1.00
0.99
3.01
δ (ppm)

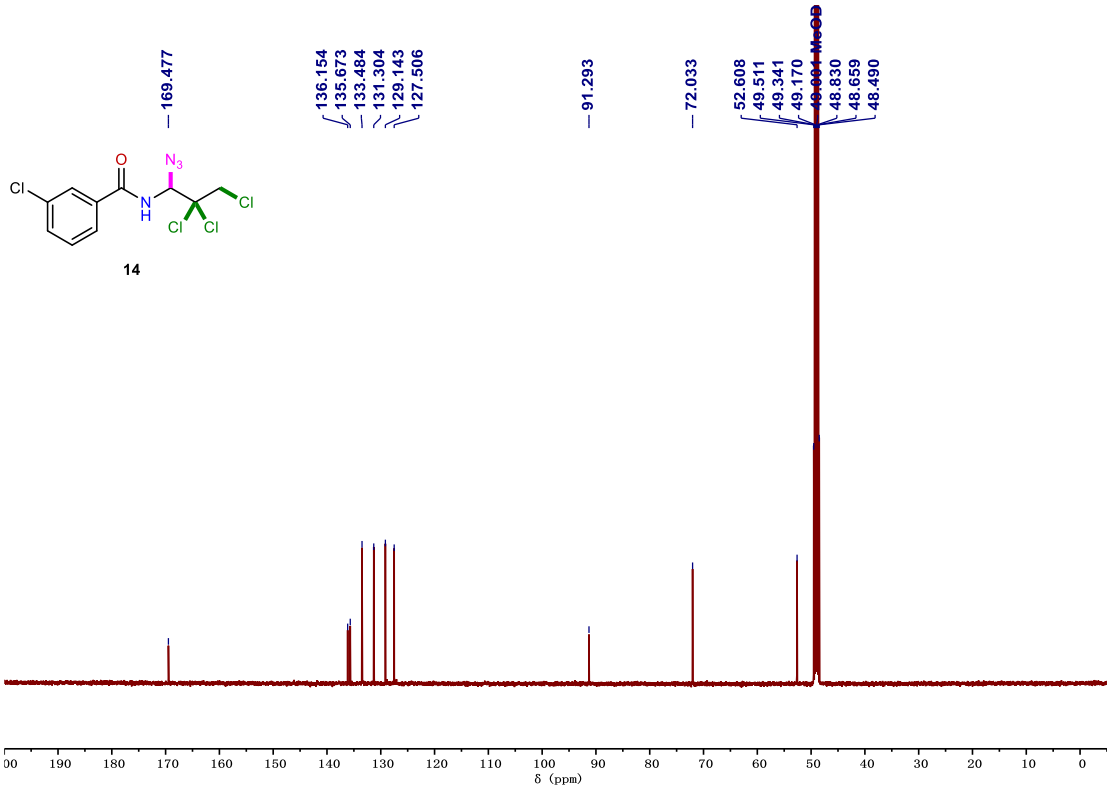

170.112
140.607
135.635
134.996
133.062
131.074
129.510
129.068
128.680
93.555
68.079
54.057
49.511
49.341
49.170
49.000 MeOD
48.989
48.829
48.659
48.489
21.178
Me
S
O
N
H
Cl
Cl
Cl
12
δ (ppm)

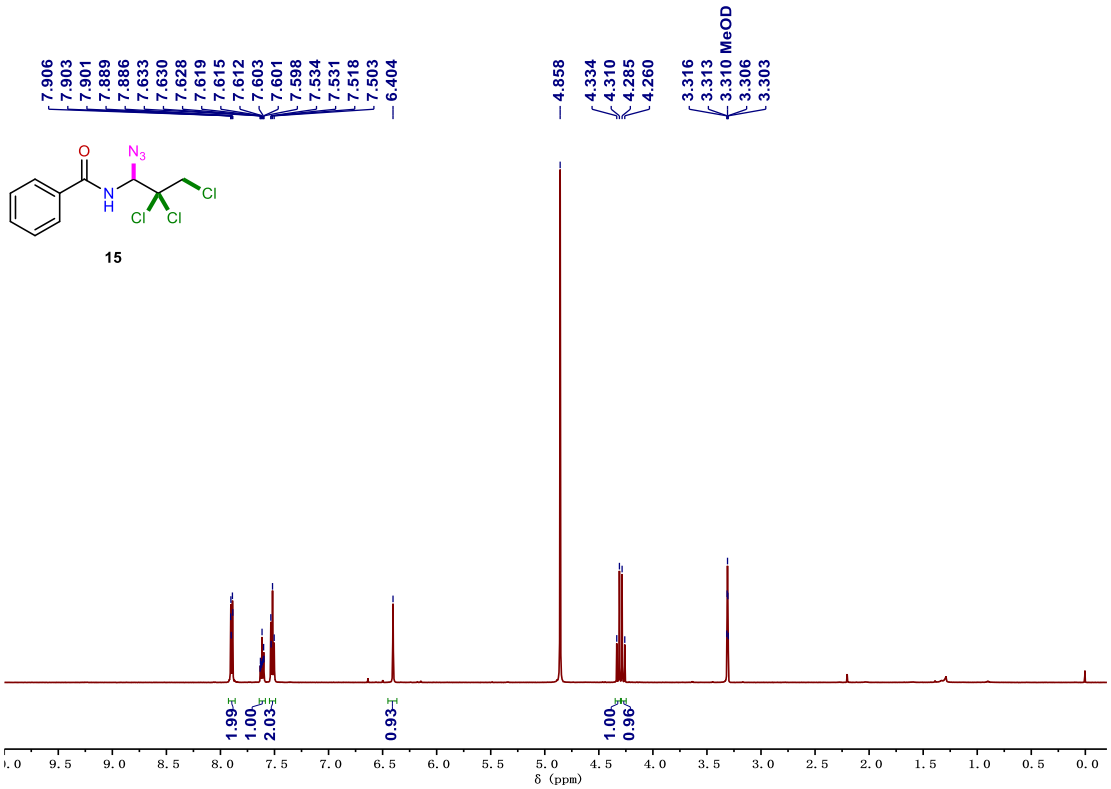

7.794
7.783
7.780
7.776
7.770
7.766
7.483
7.467
7.200
7.195
7.186
7.182
7.178
7.169
7.165
7.147
7.131
6.234
4.600
4.576
4.389
4.364
3.317
3.314
3.310
3.307
3.304
2.311
Me
S
O
N
H
Cl
Cl
Cl
F
13
2.02
2.04
2.05
2.01
1.00
1.06
1.06
3.04
δ (ppm)

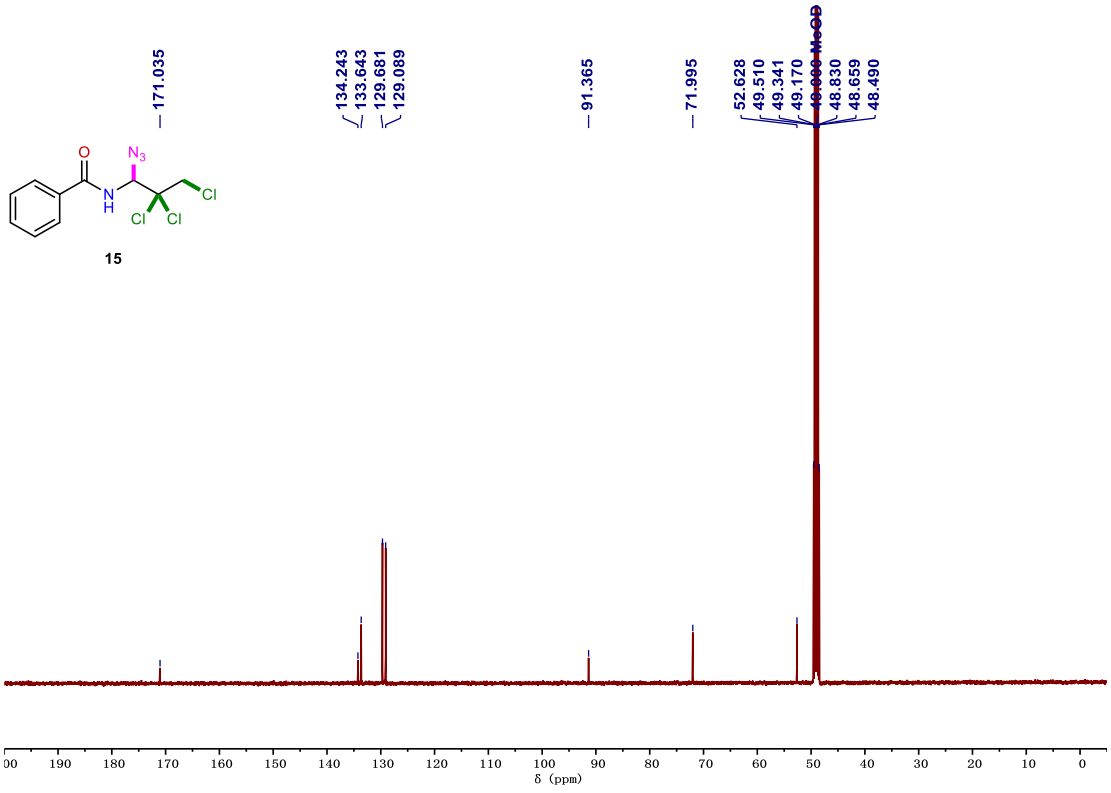

168.899
167.405
165.410
140.618
135.594
131.414
131.341
131.293
131.269
131.078
129.061
116.469
116.291
93.534
68.142
54.046
49.511
49.341
49.171
49.000 MeOD
48.831
48.660
48.489
21.171
Me
S
O
N
H
Cl
Cl
Cl
F
13
δ (ppm)

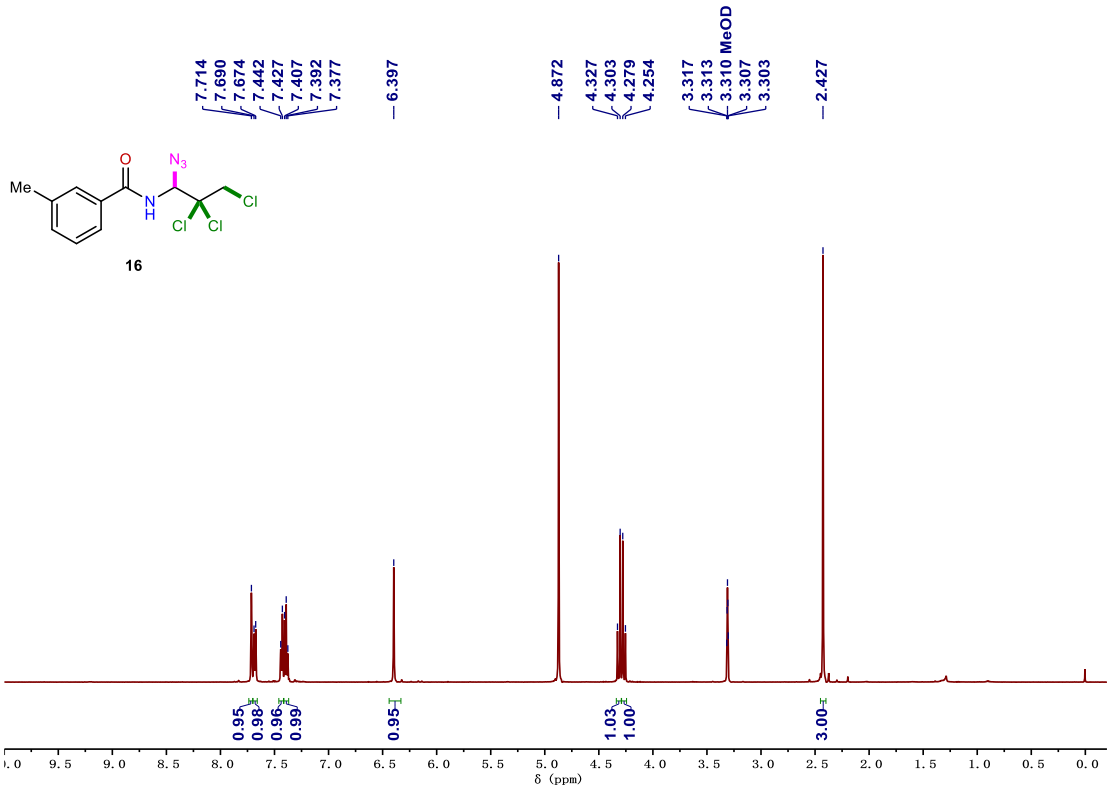

-109.810
Me
S
O
N
H
Cl
Cl
Cl
F
13
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)

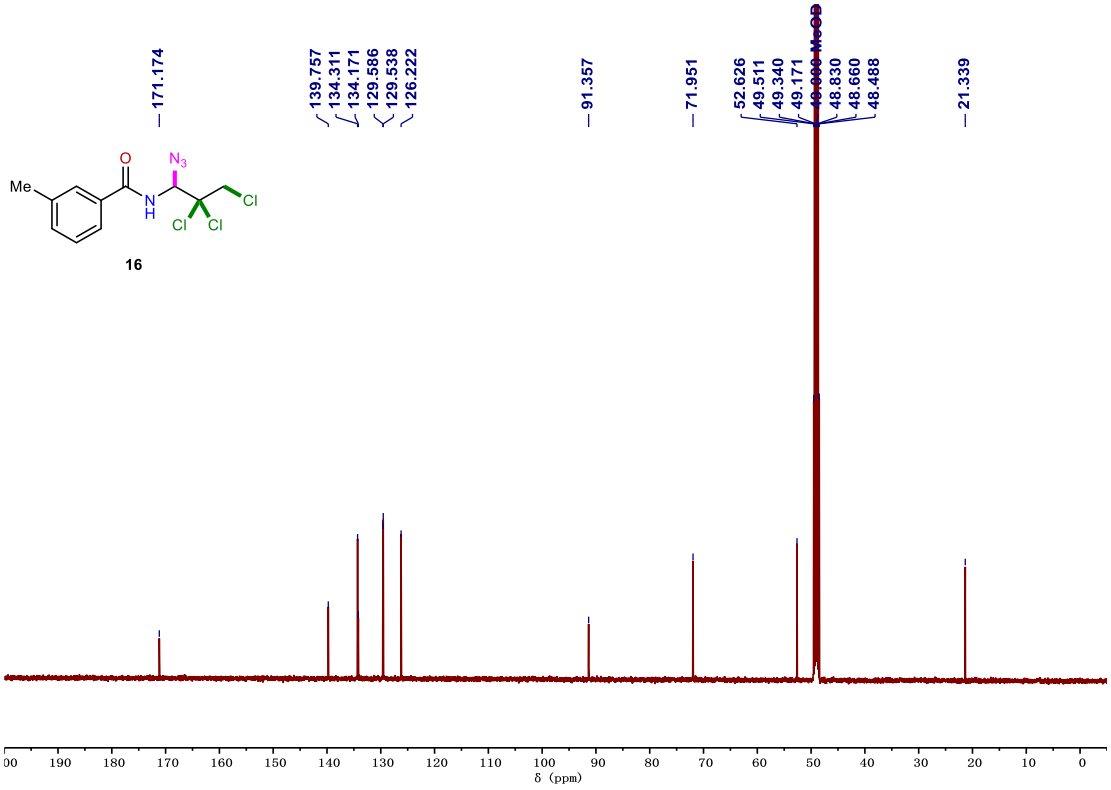

7.830
7.827
7.817
7.814
7.811
7.631
7.629
7.627
7.625
7.615
7.613
7.611
7.609
7.523
7.508
7.492
6.388
4.855
4.330
4.305
4.282
4.257
3.316
3.313
3.310 MeOD
3.306
3.303
O
N3
N
H
Cl
Cl
Cl
Cl
14
1.00
1.03
1.01
1.05
0.98
1.09
1.04
δ (ppm)

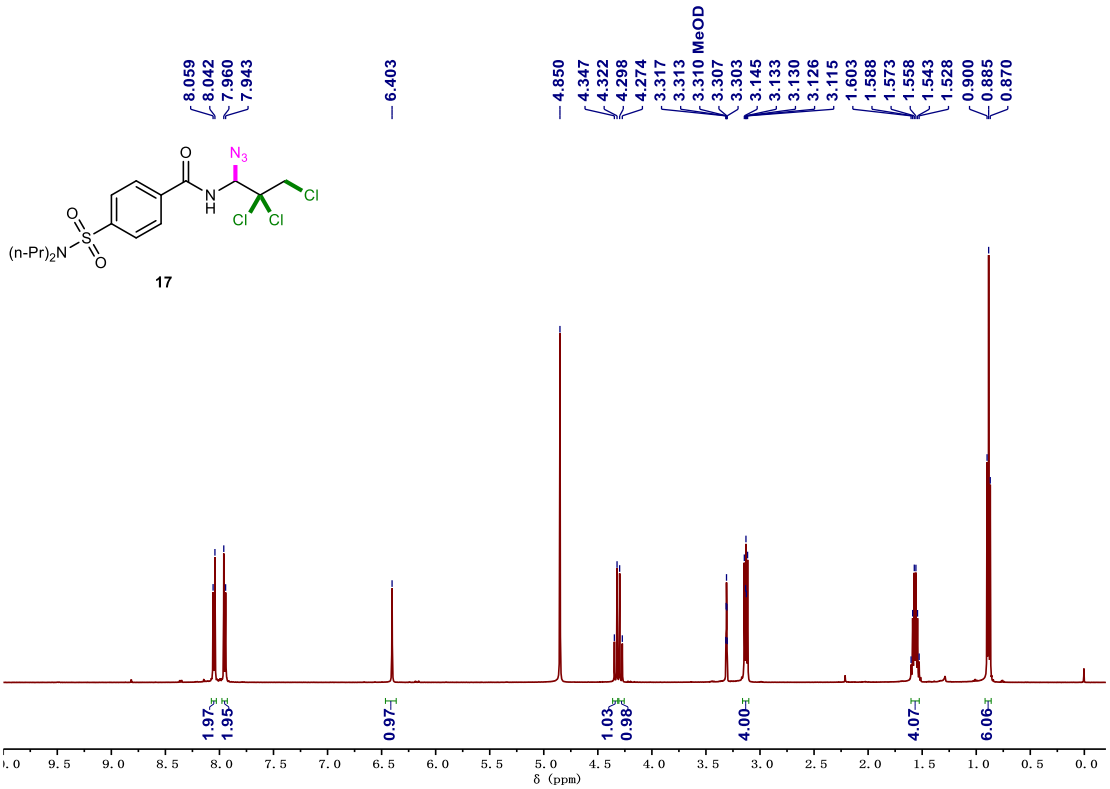

169.477
136.154
135.673
133.484
131.304
129.143
127.506
91.293
72.033
52.608
49.511
49.341
49.170
49.001 MeOD
48.830
48.659
48.490
O
N3
N
H
Cl
Cl
Cl
Cl
14
δ (ppm)

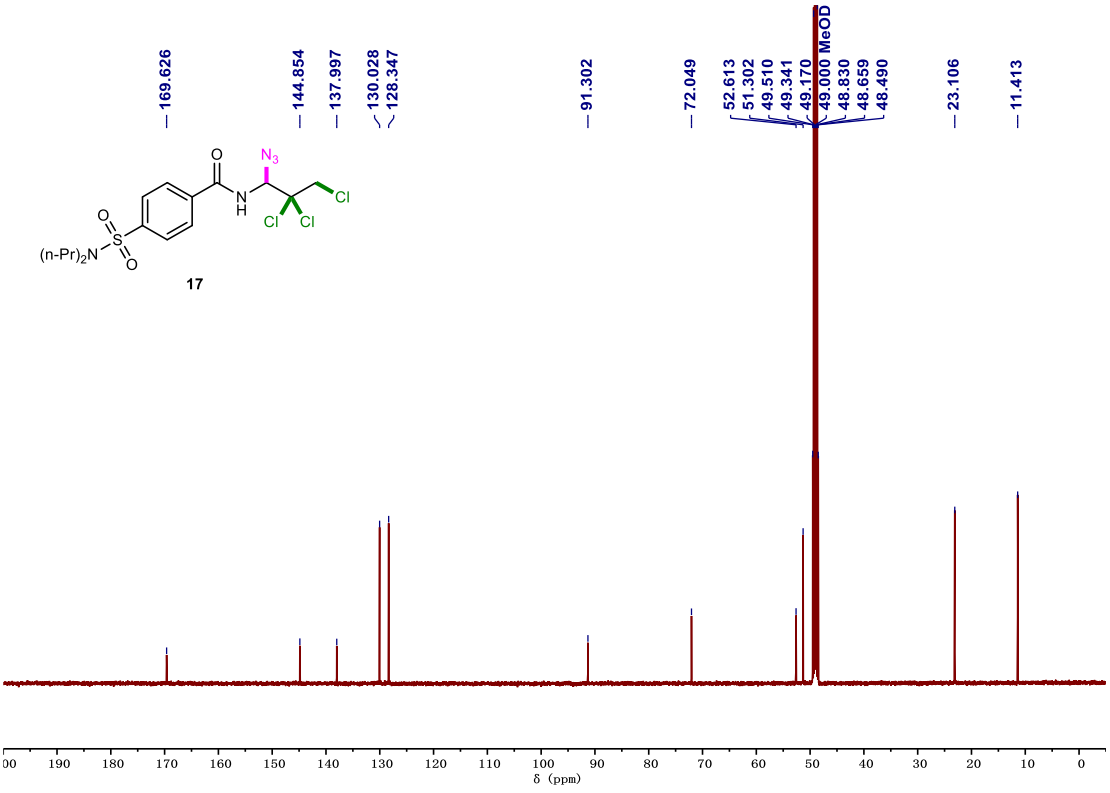

7.906
7.903
7.901
7.889
7.886
7.633
7.630
7.628
7.619
7.615
7.612
7.603
7.601
7.598
7.534
7.531
7.518
7.503
6.404
4.858
4.334
4.310
4.285
4.260
3.316
3.313
3.310 MeOD
3.306
3.303
O
N3
N
H
Cl
Cl
Cl
15
1.99
1.00
2.03
0.93
1.00
0.96
δ (ppm)

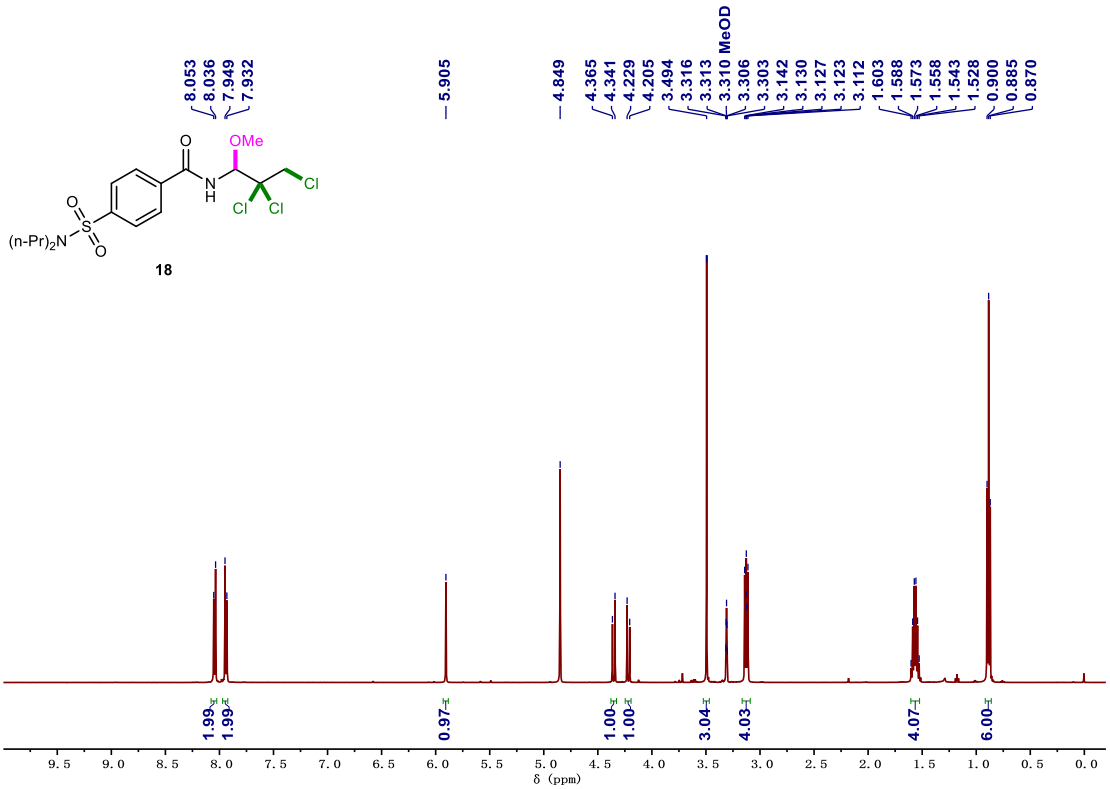

171.035
134.243
133.643
129.681
129.089
91.365
71.995
52.628
49.510
49.341
49.170
49.000 MeOD
48.830
48.659
48.490
O
N3
N
H
Cl
Cl
Cl
15
δ (ppm)

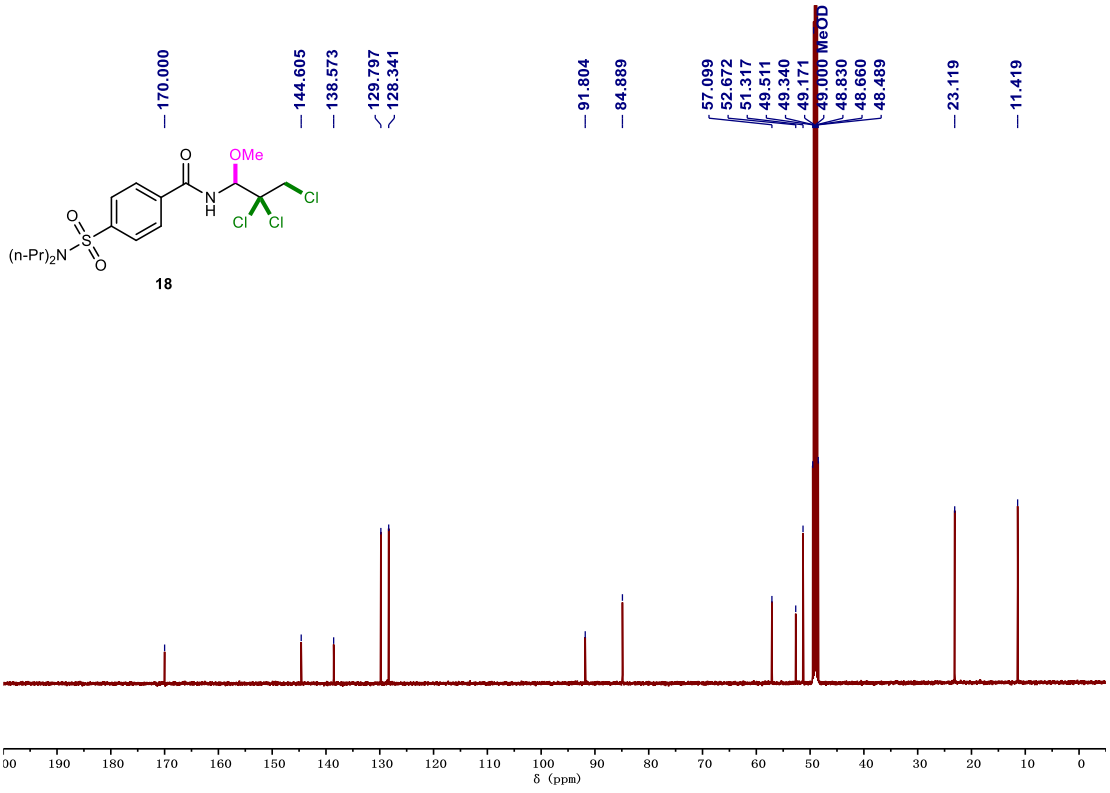

7.714
7.690
7.674
7.442
7.427
7.407
7.392
7.377
6.397
4.872
4.327
4.303
4.279
4.254
3.317
3.313
3.310 MeOD
3.307
3.303
2.427
Me
O
N3
N
H
Cl
Cl
Cl
16
0.95
0.98
0.96
0.99
0.95
1.03
1.00
3.00
δ (ppm)

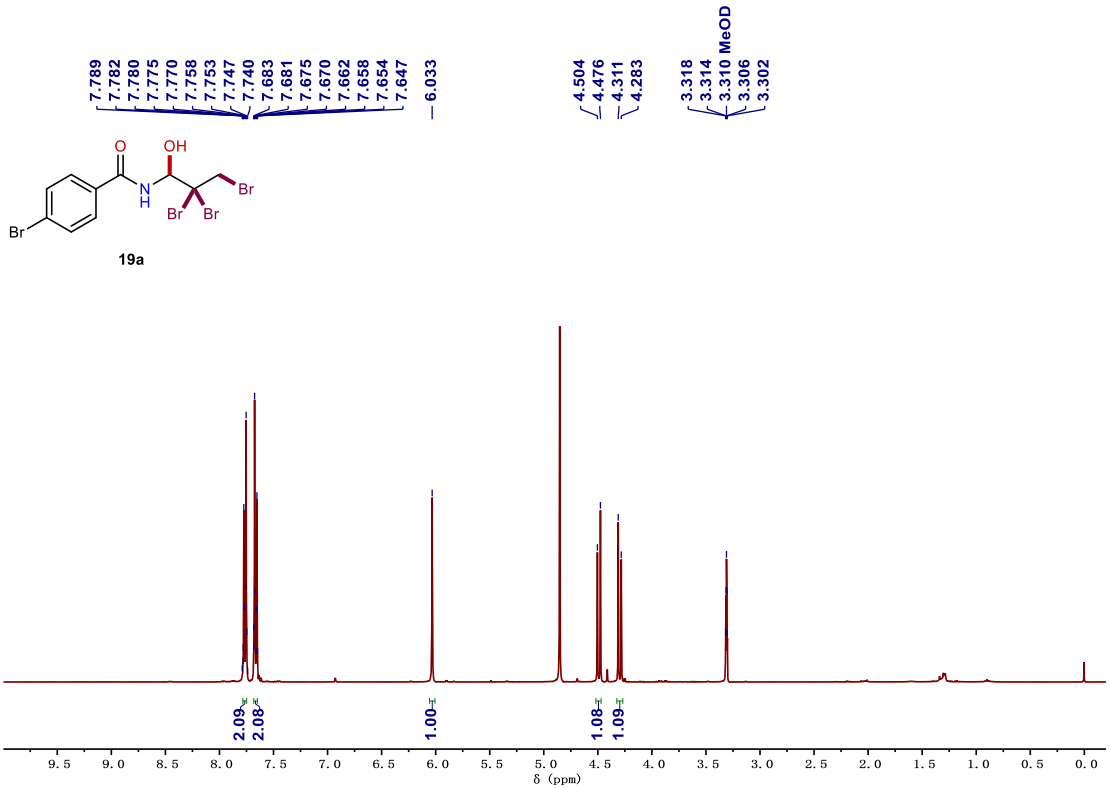

171.174
139.757
134.311
134.171
129.586
129.538
126.222
91.357
71.951
52.626
49.511
49.340
49.171
49.000 MeOD
48.830
48.660
48.488
21.339
Me
O
N3
N
H
Cl
Cl
Cl
16
δ (ppm)


8.059
8.042
7.960
7.943
6.403
4.850
4.347
4.322
4.298
4.274
3.317
3.313
3.310 MeOD
3.307
3.303
3.145
3.133
3.130
3.126
3.115
1.603
1.588
1.573
1.558
1.543
1.528
0.900
0.885
0.870
17
1.97
1.95
0.97
1.03
0.98
4.00
4.07
6.06
δ (ppm)


169.626
144.854
137.997
130.028
128.347
91.302
72.049
52.613
51.302
49.510
49.341
49.170
49.000 MeOD
48.830
48.659
48.490
23.106
11.413
17
δ (ppm)


8.053
8.036
7.949
7.932
5.905
4.849
4.365
4.341
4.229
4.205
3.494
3.316
3.313
3.310 MeOD
3.306
3.303
3.142
3.130
3.127
3.123
3.112
1.603
1.588
1.573
1.558
1.543
1.528
0.900
0.885
0.870
OMe
Cl
Cl
Cl
O
N
H
O
S
O
(n-Pr)2N
18
1.99
1.99
0.97
1.00
1.00
3.04
4.03
4.07
6.00
δ (ppm)


170.000
144.605
138.573
129.797
128.341
91.804
84.889
57.099
52.672
51.317
49.511
49.340
49.171
49.000 MeOD
48.830
48.660
48.489
23.119
11.419
OMe
Cl
Cl
Cl
O
N
H
O
S
O
(n-Pr)2N
18
δ (ppm)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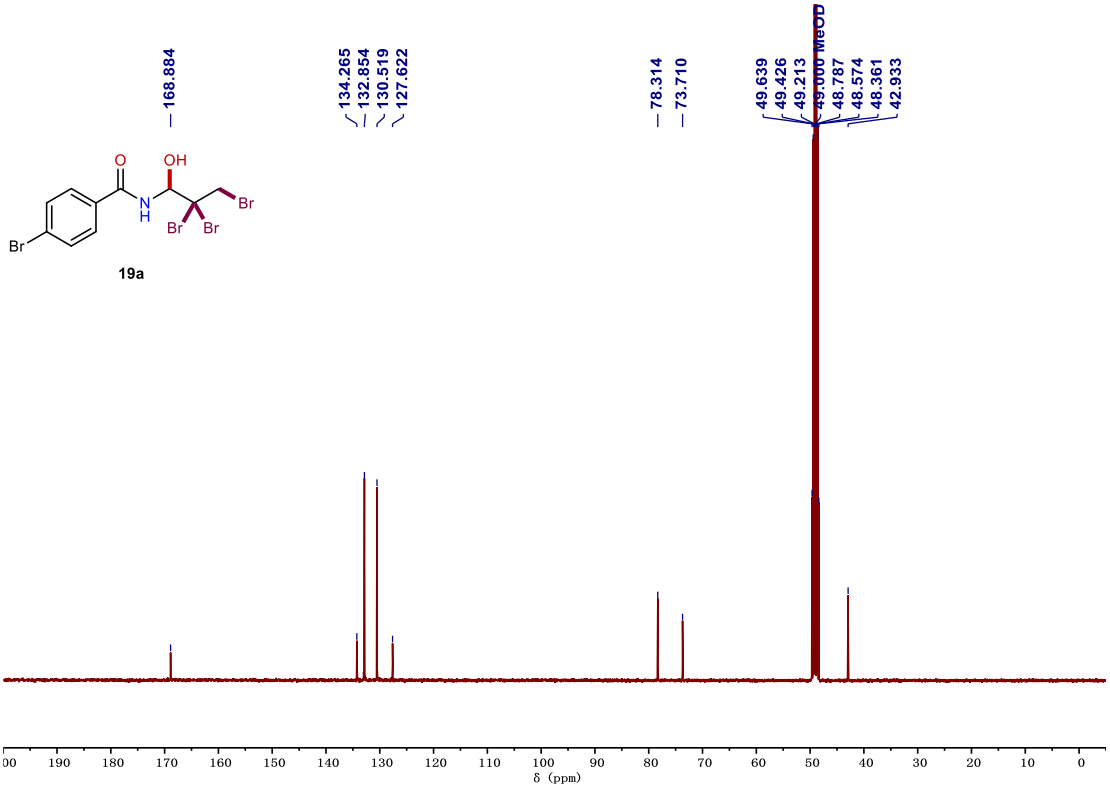
7.782
7.780
7.775
7.770
7.758
7.753
7.747
7.740
7.683
7.681
7.675
7.670
7.662
7.658
7.654
7.647
6.033
4.504
4.476
4.311
4.283
3.318
3.314
3.310 MeOD
3.306
3.302
O
OH
N
H
Br
Br
Br
Br
19a
2.09
2.08
1.00
1.08
1.09
δ (ppm)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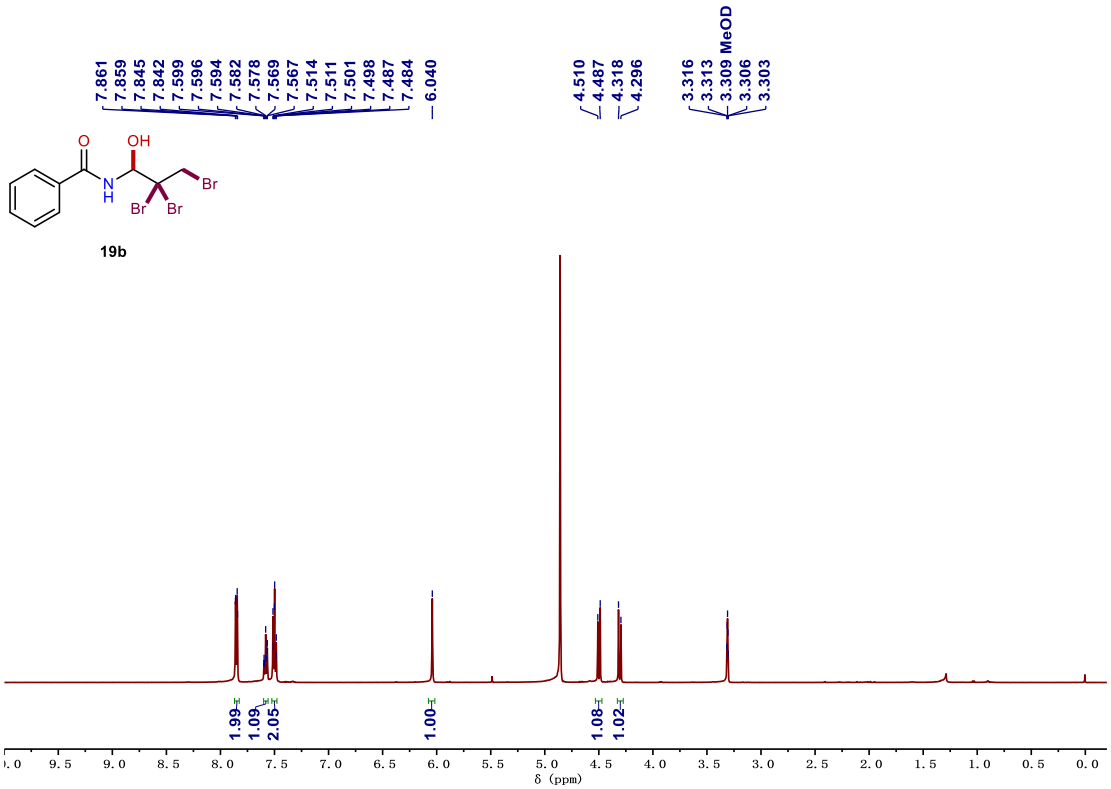
134.265
132.854
130.519
127.622
78.314
73.710
49.639
49.426
49.213
49.000 MeOD
48.787
48.574
48.361
42.933
O
OH
N
H
Br
Br
Br
Br
19a
δ (ppm)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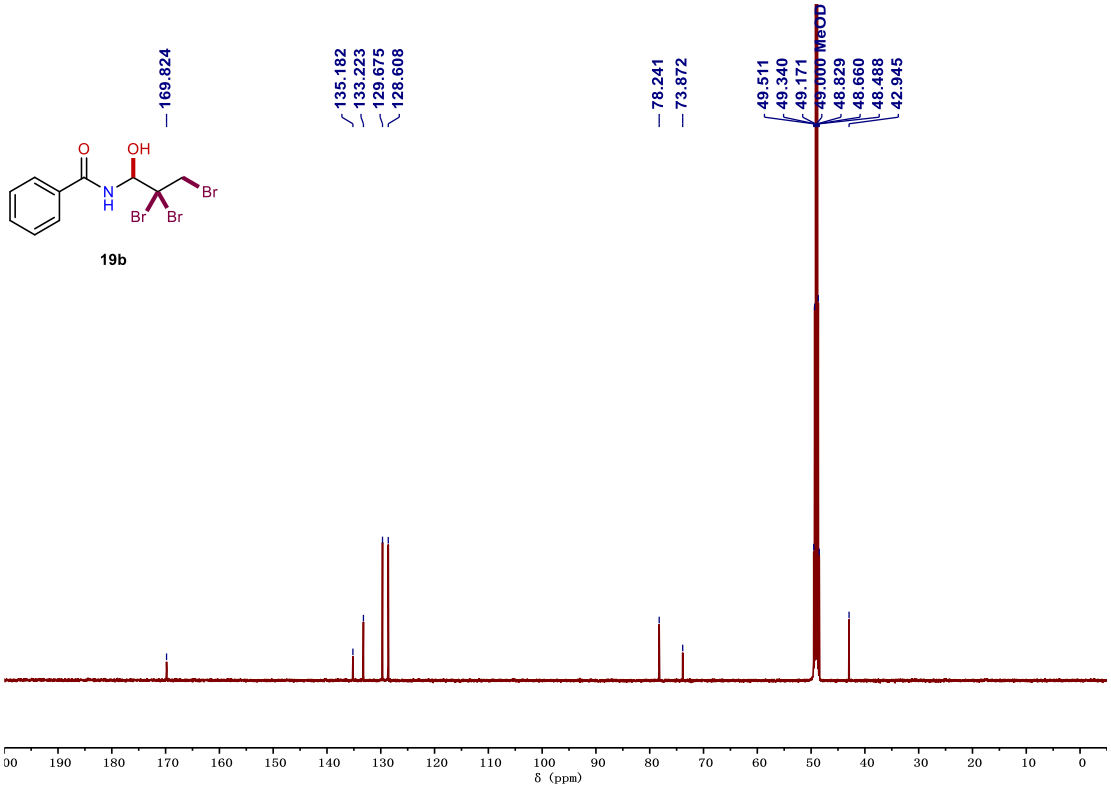
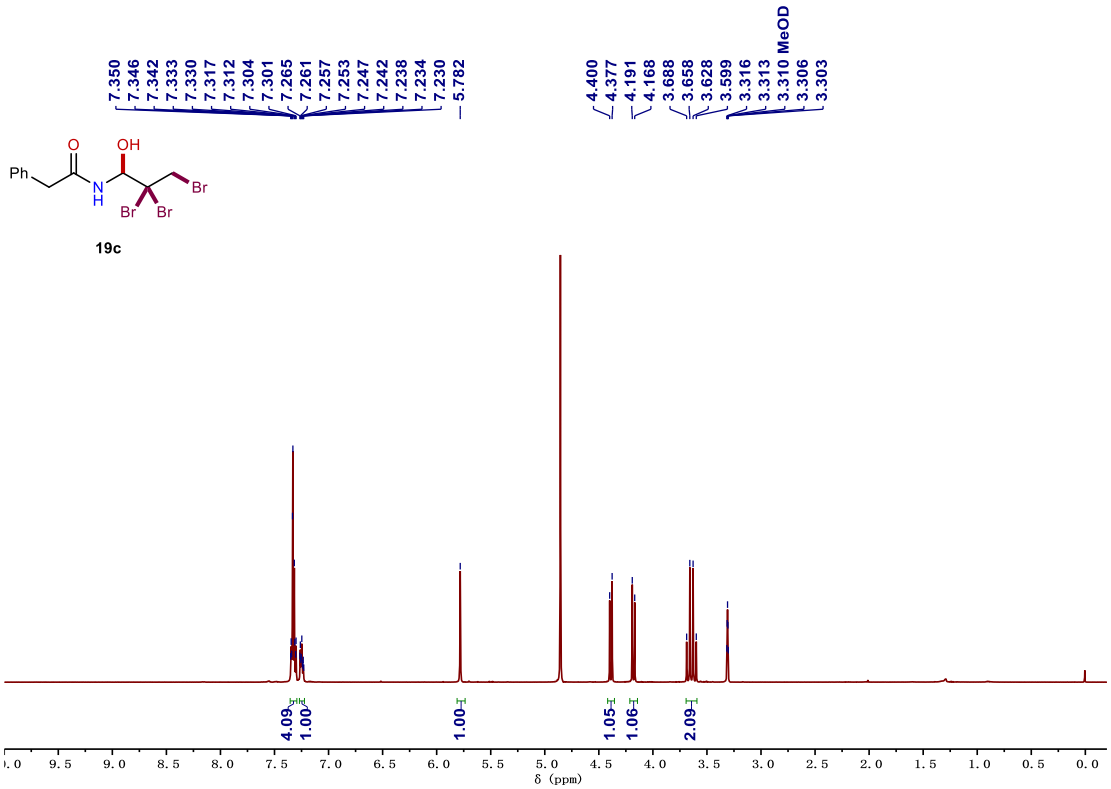
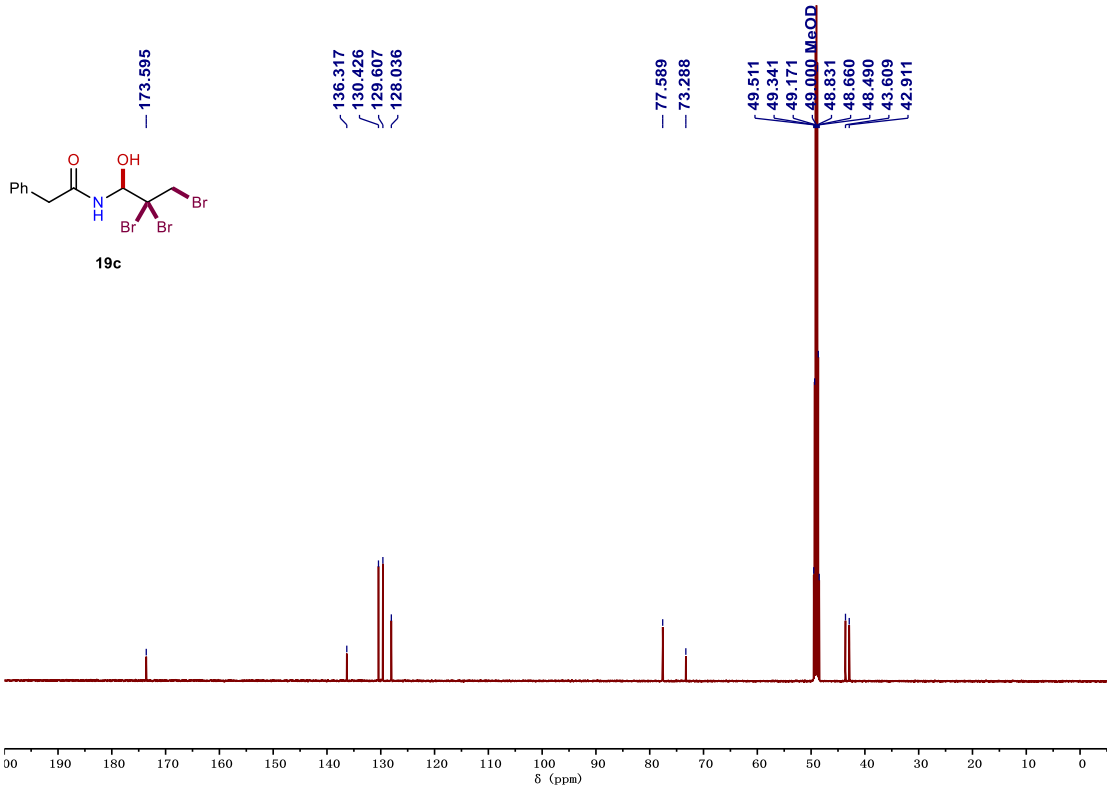
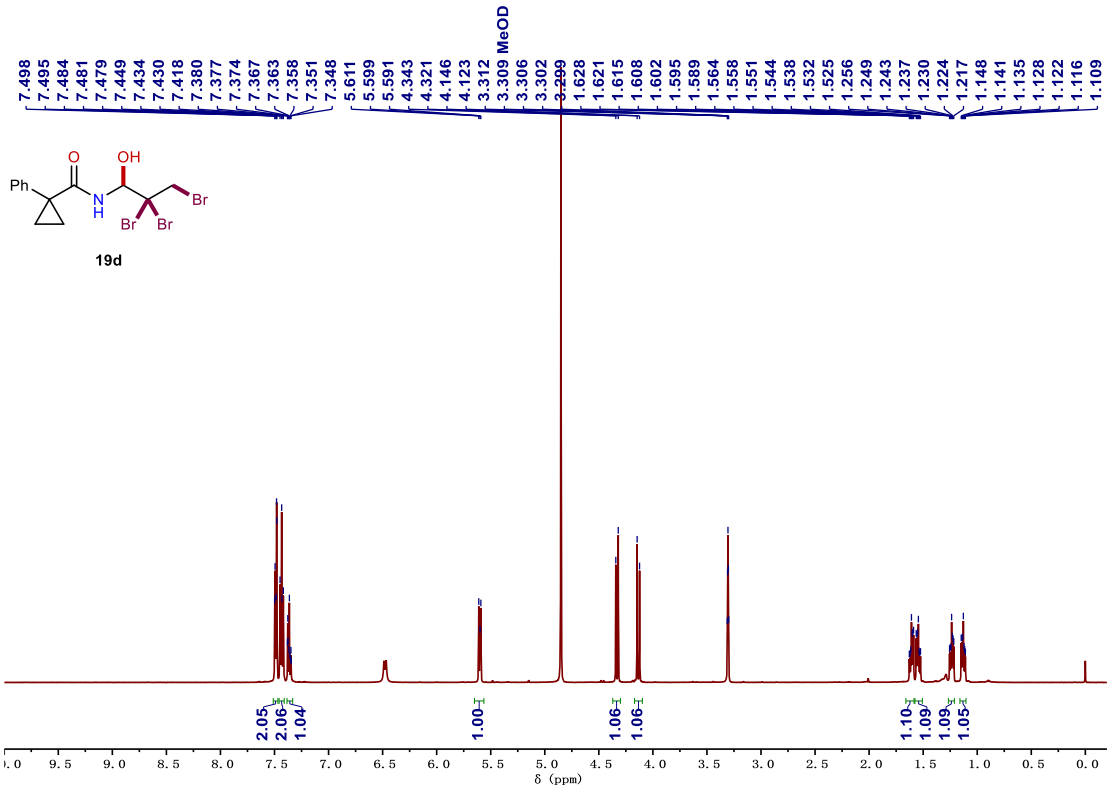
7.859
7.845
7.842
7.599
7.596
7.594
7.582
7.578
7.569
7.567
7.514
7.511
7.501
7.498
7.487
7.484
6.040
4.510
4.487
4.318
4.296
3.316
3.313
3.309 MeOD
3.306
3.303
O
OH
N
H
Br
Br
Br
19b
1.99
1.09
2.05
1.00
1.08
1.02
δ (ppm)


169.824
135.182
133.223
129.675
128.608
78.241
73.872
49.511
49.340
49.171
49.000 MeOD
48.829
48.660
48.488
42.945
O
OH
N
H
Br
Br
Br
19b
δ (ppm)


7.350
7.346
7.342
7.333
7.330
7.317
7.312
7.304
7.301
7.265
7.261
7.257
7.253
7.247
7.242
7.238
7.234
7.230
5.782
4.400
4.377
4.191
4.168
3.688
3.658
3.628
3.599
3.316
3.313
3.310 MeOD
3.306
3.303
O
OH
Ph
N
H
Br
Br
Br
19c
4.09
1.00
1.00
1.05
1.06
2.09
δ (ppm)


173.595
136.317
130.426
129.607
128.036
77.589
73.288
49.511
49.341
49.171
49.000 MeOD
48.831
48.660
48.490
43.609
42.911
O
OH
Ph
N
H
Br
Br
Br
19c
δ (ppm)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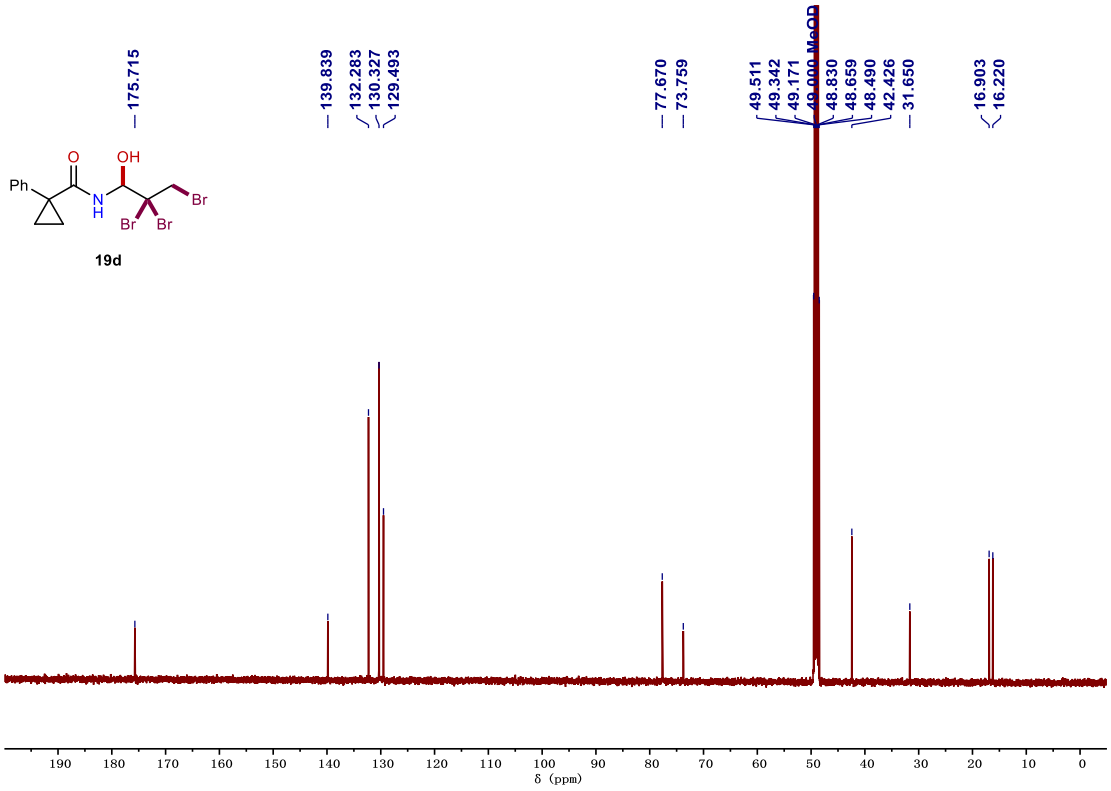
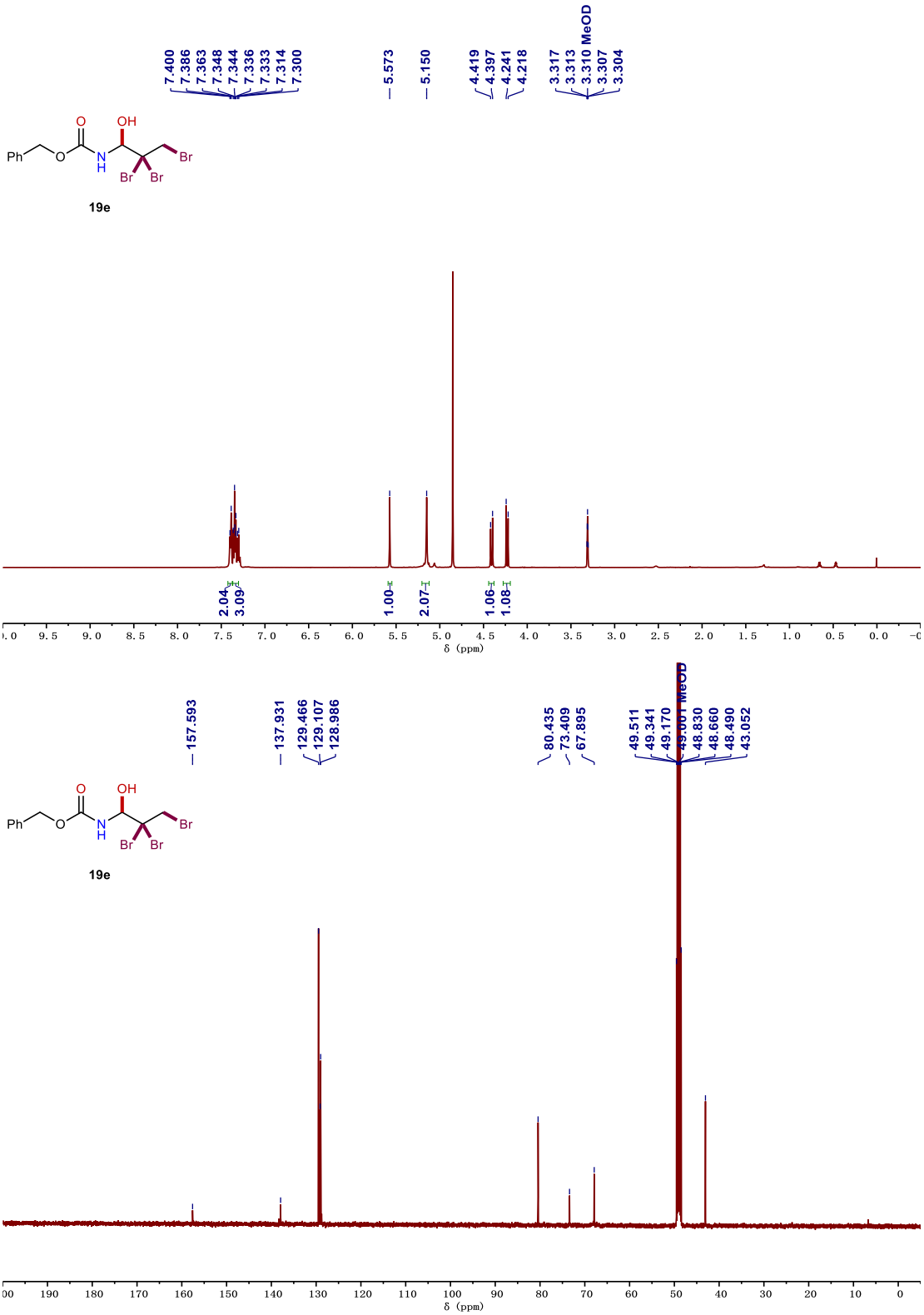
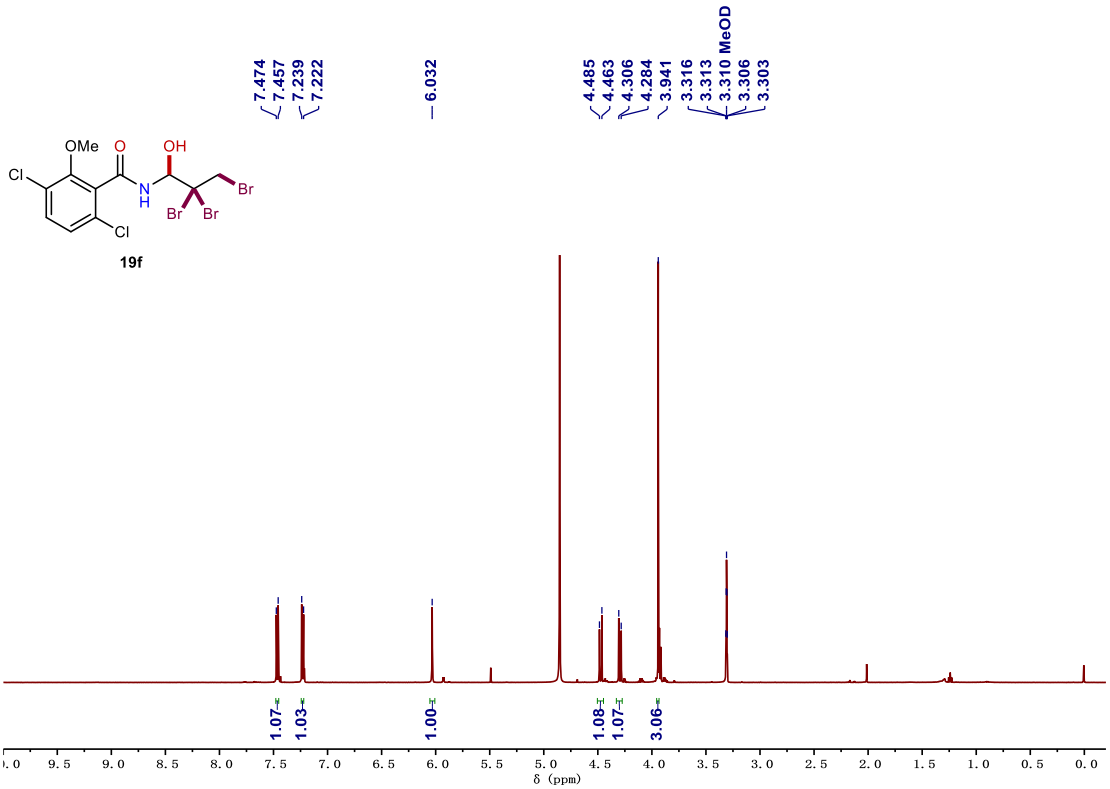
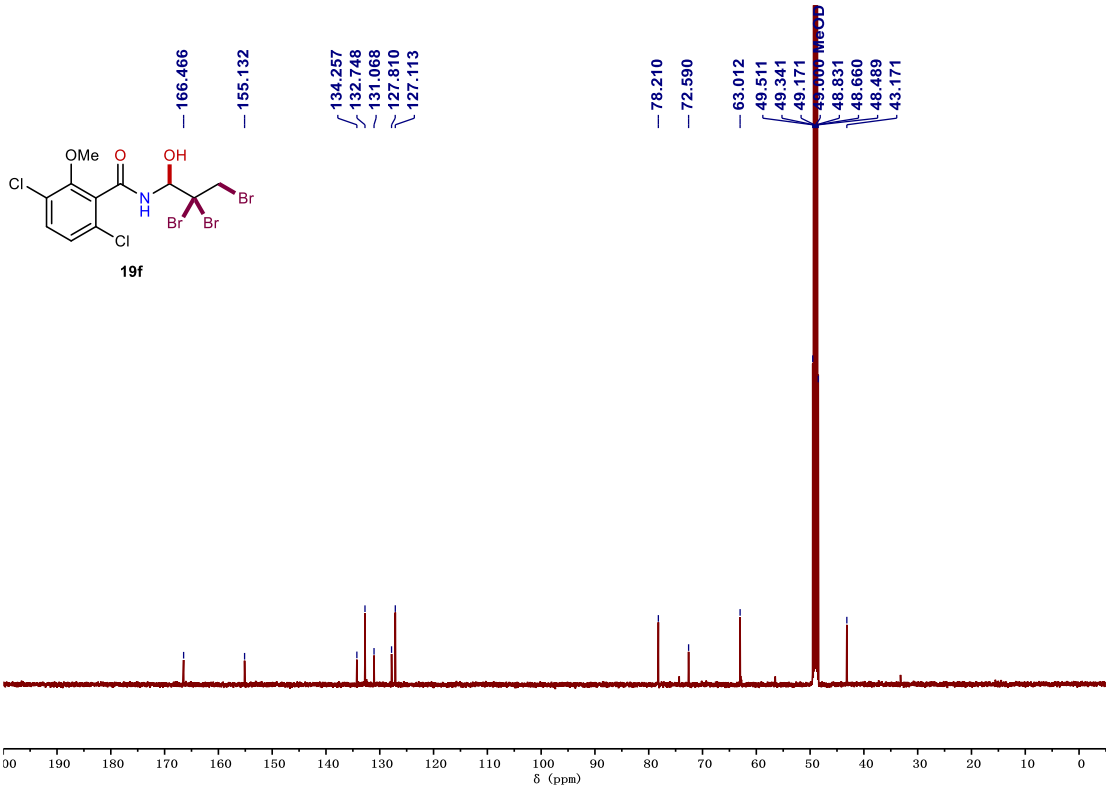
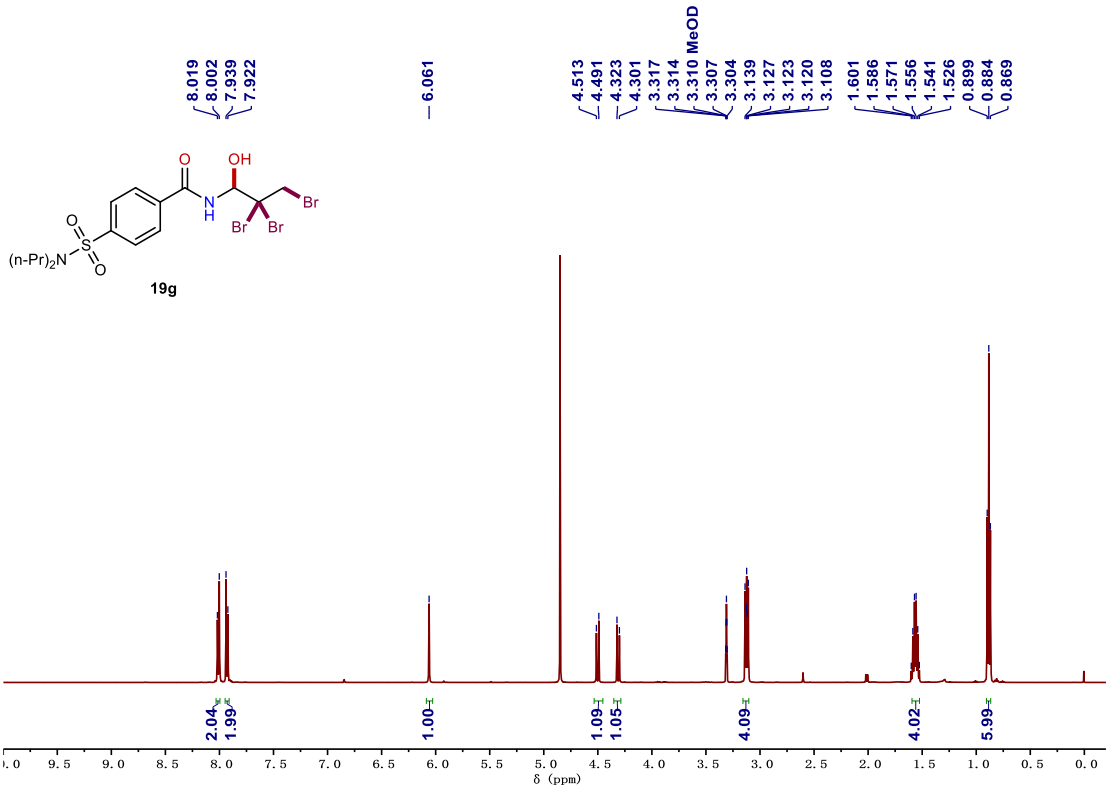
7.495
7.484
7.481
7.479
7.449
7.434
7.430
7.418
7.380
7.377
7.374
7.367
7.363
7.358
7.351
7.348
5.611
5.599
5.591
4.343
4.321
4.146
4.123
3.312
3.309 MeOD
3.306
3.302
3.299
1.628
1.621
1.615
1.608
1.602
1.595
1.589
1.564
1.558
1.551
1.544
1.538
1.532
1.525
1.256
1.249
1.243
1.237
1.230
1.224
1.217
1.148
1.141
1.135
1.128
1.122
1.116
1.109
O
OH
Ph
N
H
Br
Br
Br
19d
2.05
2.06
1.04
1.00
1.06
1.06
1.10
1.09
1.09
1.05
δ (ppm)

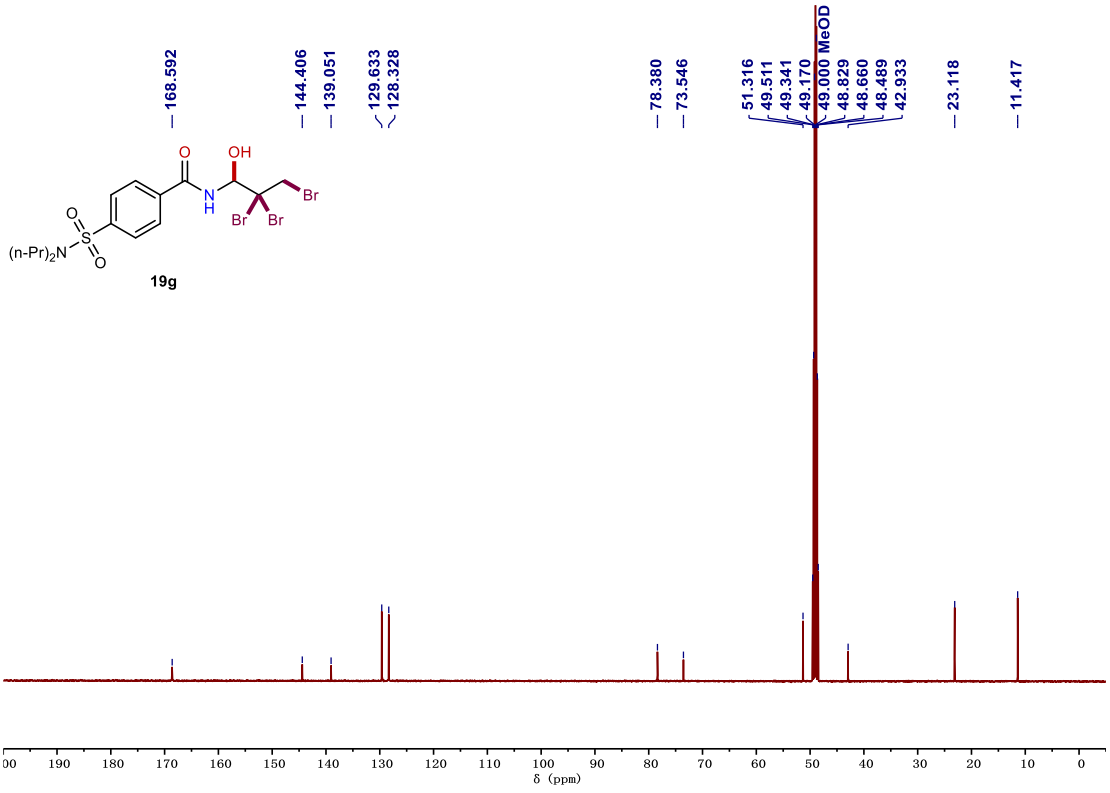

175.715
139.839
132.283
130.327
129.493
77.670
73.759
49.511
49.342
49.171
49.000 MeOD
48.830
48.659
48.490
42.426
31.650
16.903
16.220
O
OH
Ph
N
H
Br
Br
Br
19d
δ (ppm)

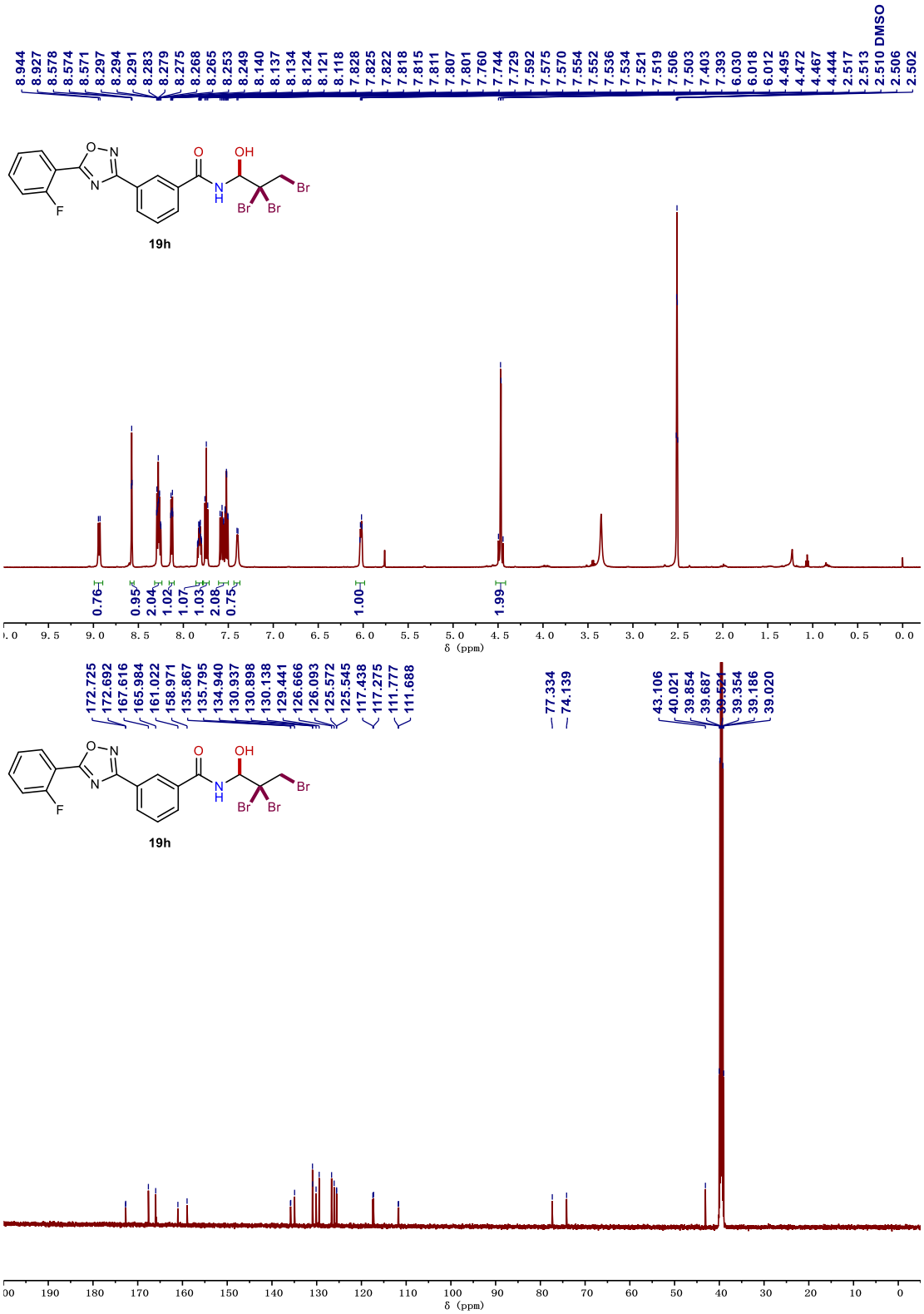

7.400
7.386
7.363
7.348
7.344
7.336
7.333
7.314
7.300
5.573
5.150
4.419
4.397
4.241
4.218
3.317
3.313
3.310 MeOD
3.307
3.304
O
OH
Ph
O
N
H
Br
Br
Br
19e
2.04
3.09
1.00
2.07
1.06
1.08
δ (ppm)
157.593
137.931
129.466
129.107
128.986
80.435
73.409
67.895
49.511
49.341
49.170
49.001 MeOD
48.830
48.660
48.490
43.052
δ (ppm)

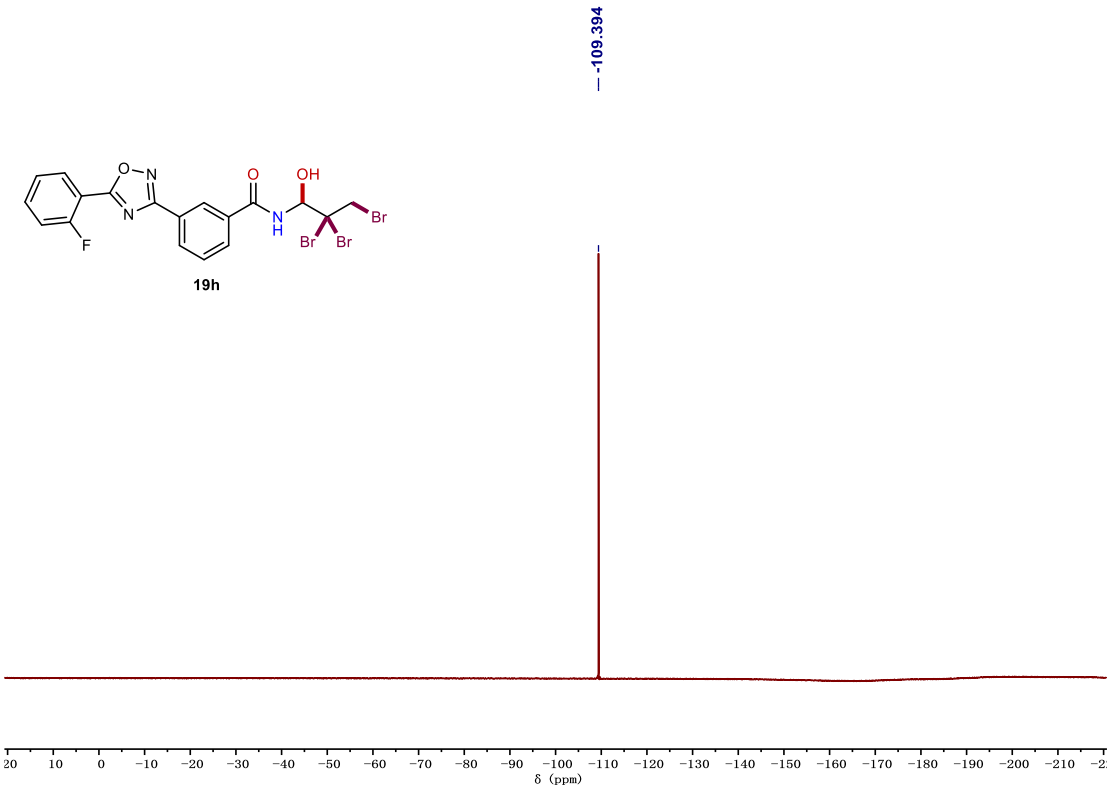

7.474
7.457
7.239
7.222
6.032
4.485
4.463
4.306
4.284
3.941
3.316
3.313
3.310 MeOD
3.306
3.303
OMe
O
OH
Cl
N
H
Br
Br
Br
Cl
19f
1.07
1.03
1.00
1.08
1.07
3.06
δ (ppm)

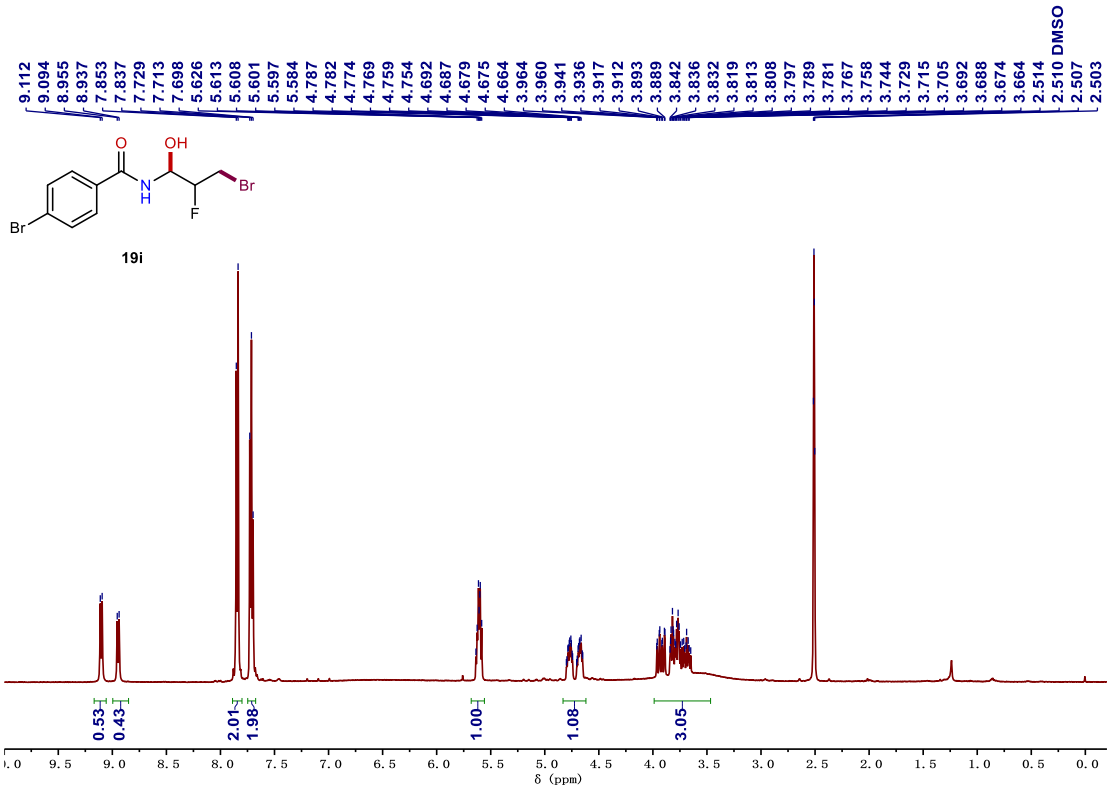

166.466
155.132
134.257
132.748
131.068
127.810
127.113
78.210
72.590
63.012
49.511
49.341
49.171
49.000 MeOD
48.831
48.660
48.489
43.171
OMe
O
OH
Cl
N
H
Br
Br
Br
Cl
19f
δ (ppm)

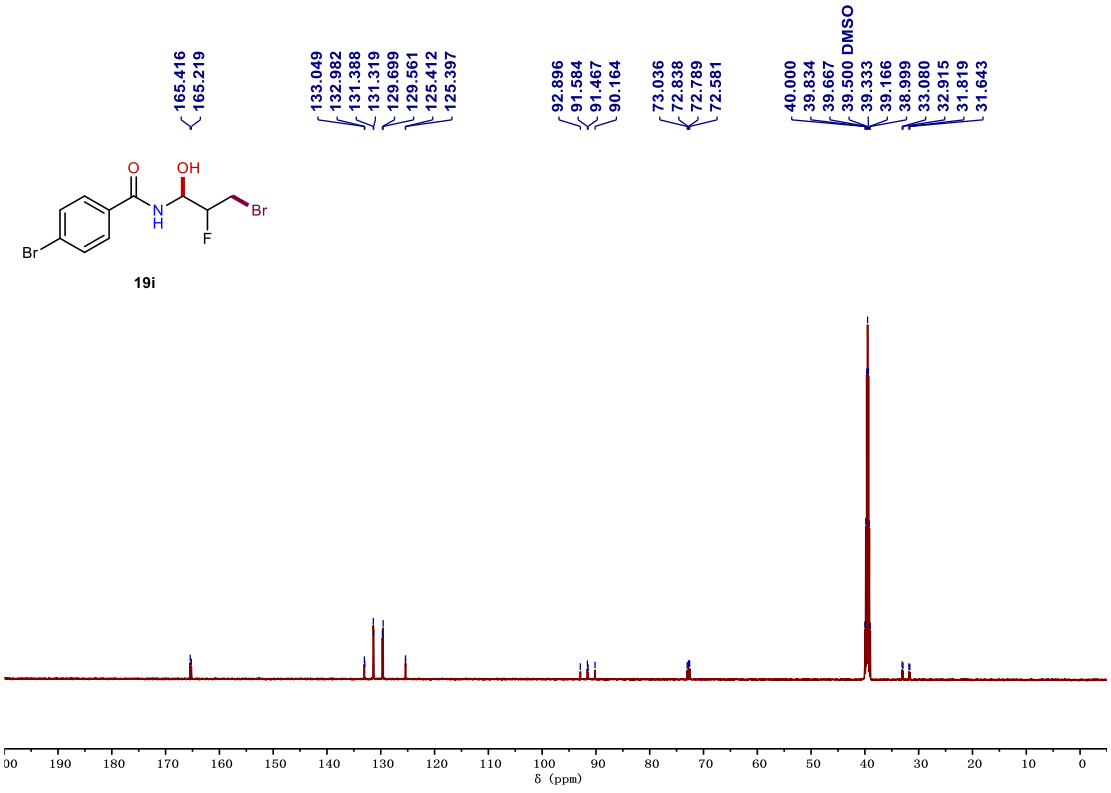

8.019
8.002
7.939
7.922
6.061
4.513
4.491
4.323
4.301
3.317
3.314
3.310 MeOD
3.307
3.304
3.139
3.127
3.123
3.120
3.108
1.601
1.586
1.571
1.556
1.541
1.526
0.899
0.884
0.869
2.04
1.99
1.00
1.09
1.05
4.09
4.02
5.99
δ (ppm)
19g

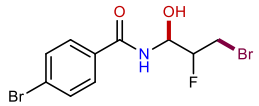

168.592
144.406
139.051
129.633
128.328
78.380
73.546
51.316
49.511
49.341
49.170
49.000 MeOD
48.829
48.660
48.489
42.933
23.118
11.417
δ (ppm)
19g


8.944
8.927
8.578
8.574
8.571
8.297
8.294
8.291
8.283
8.279
8.275
8.268
8.265
8.253
8.249
8.140
8.137
8.134
8.124
8.121
8.118
7.828
7.825
7.822
7.818
7.815
7.811
7.807
7.801
7.760
7.744
7.729
7.592
7.575
7.570
7.554
7.552
7.536
7.534
7.521
7.519
7.506
7.503
7.403
7.393
6.030
6.018
6.012
4.495
4.472
4.467
4.444
2.517
2.513
2.510 DMSO
2.506
2.502
O
N
N
F
O
OH
N
H
Br
Br
Br
19h
0.76
0.95
2.04
1.02
1.07
1.03
2.08
0.75
1.00
1.99
δ (ppm)
172.725
172.692
167.616
165.984
161.022
158.971
135.867
135.795
134.940
130.937
130.898
130.138
129.441
126.666
126.093
125.572
125.545
117.438
117.275
111.777
111.688
77.334
74.139
43.106
40.021
39.854
39.687
39.521
39.354
39.186
39.020
δ (ppm)


-109.394
O
N
N
F
O
N
H
OH
Br
Br
Br
19h
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)

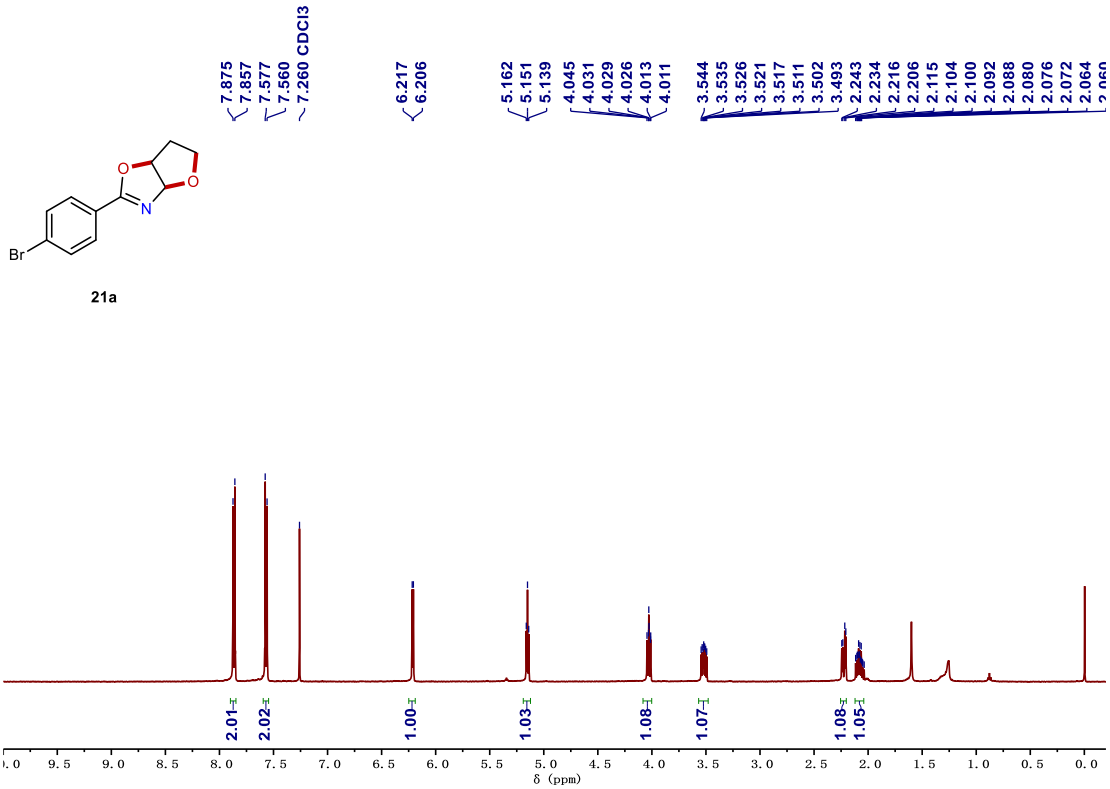

9.112
9.094
8.955
8.937
8.853
7.837
7.729
7.713
7.698
5.626
5.613
5.608
5.601
5.597
5.584
4.787
4.782
4.774
4.769
4.759
4.754
4.692
4.687
4.679
4.675
4.664
3.964
3.960
3.941
3.936
3.917
3.912
3.893
3.889
3.842
3.836
3.832
3.819
3.813
3.808
3.797
3.789
3.781
3.767
3.758
3.744
3.729
3.715
3.705
3.692
3.688
3.674
3.664
2.514
2.510 DMSO
2.507
2.503
O
OH
N
H
Br
F
Br
19i
0.53
0.43
2.01
1.98
1.00
1.08
3.05
δ (ppm)

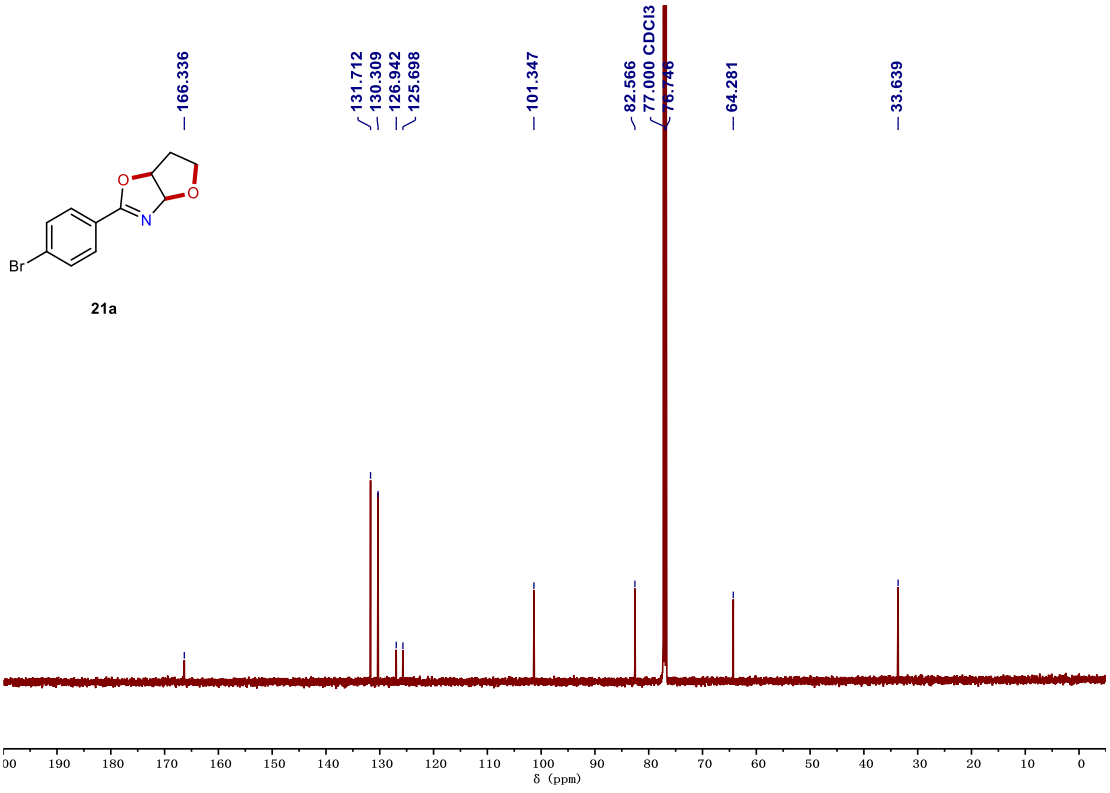

165.416
165.219
133.049
132.982
131.388
131.319
129.699
129.561
125.412
125.397
92.896
91.584
91.467
90.164
73.036
72.838
72.789
72.581
40.000
39.834
39.667
39.500 DMSO
39.333
39.166
38.999
33.080
32.915
31.819
31.643
O
OH
N
H
Br
F
Br
19i
δ (ppm)

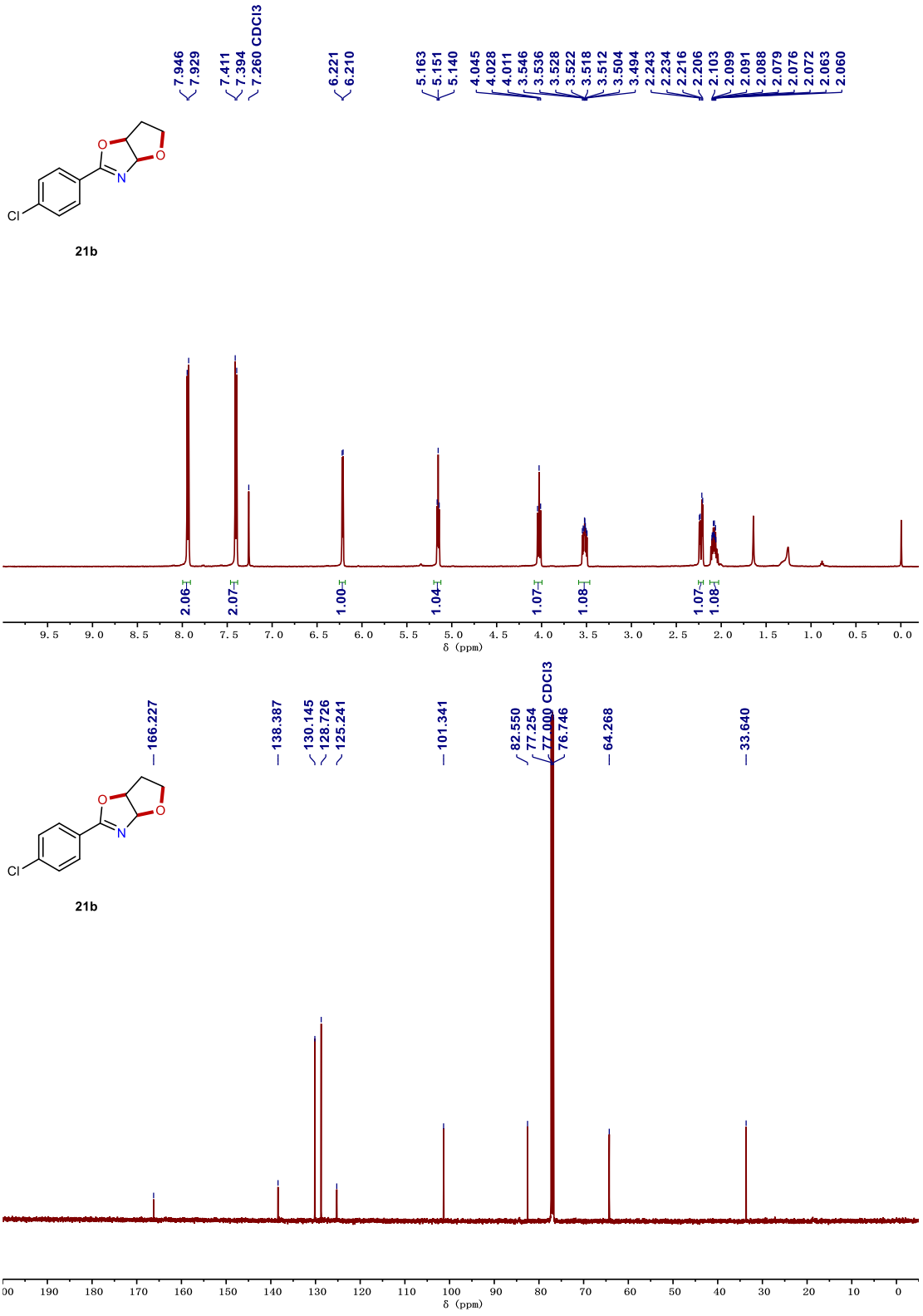

O
OH
N
H
Br
F
Br

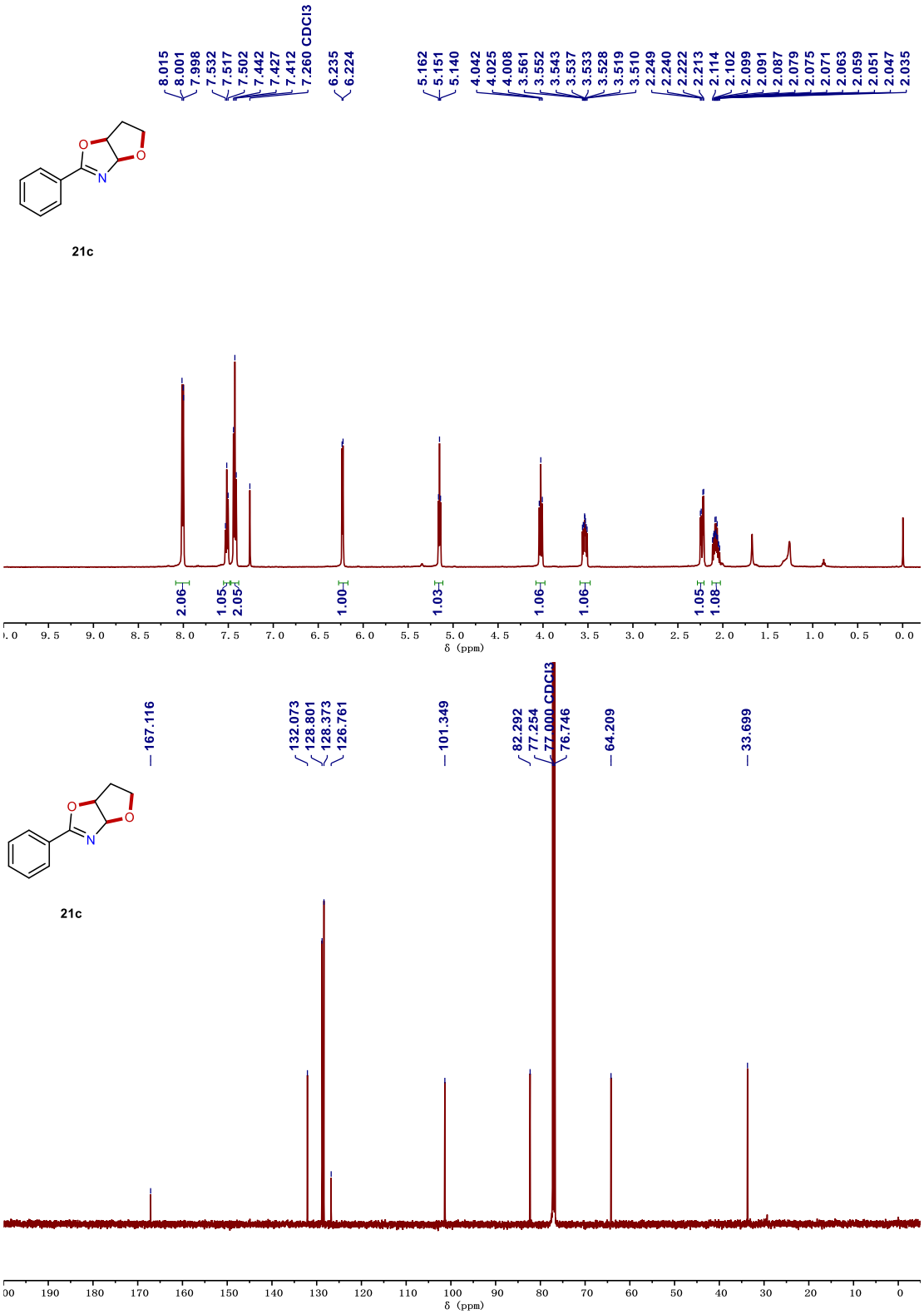
19i

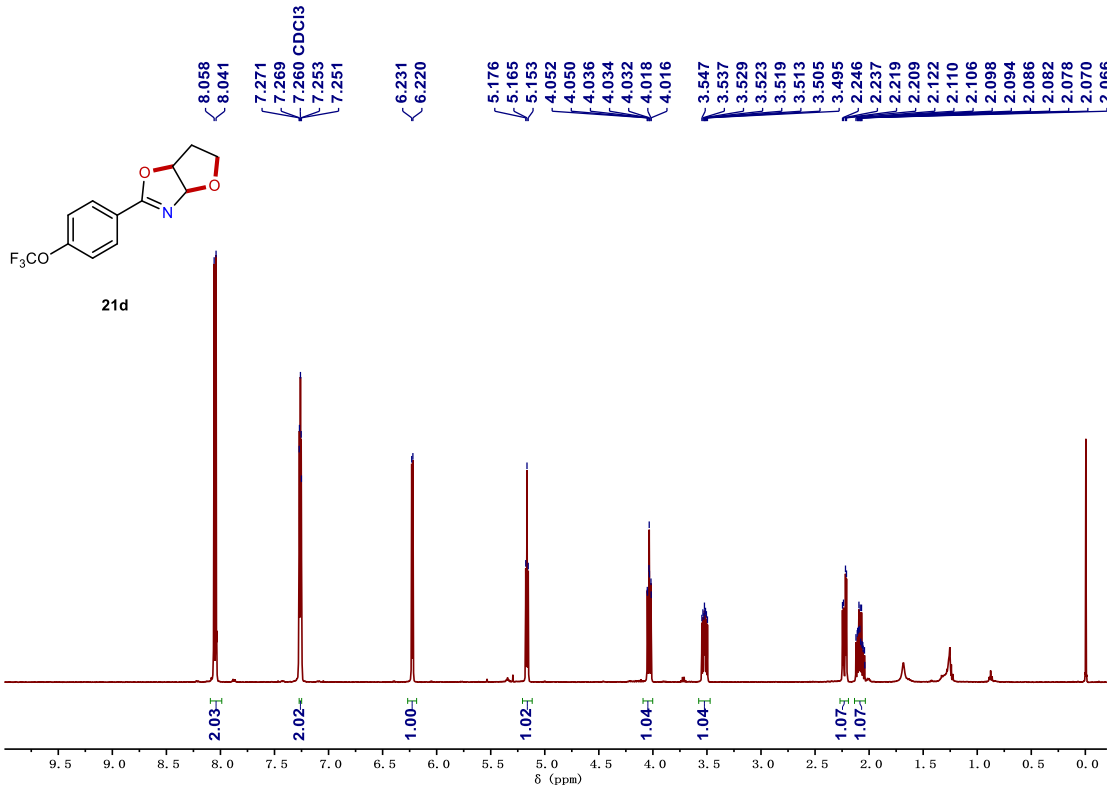
-189.034
-190.198
20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
δ (ppm)

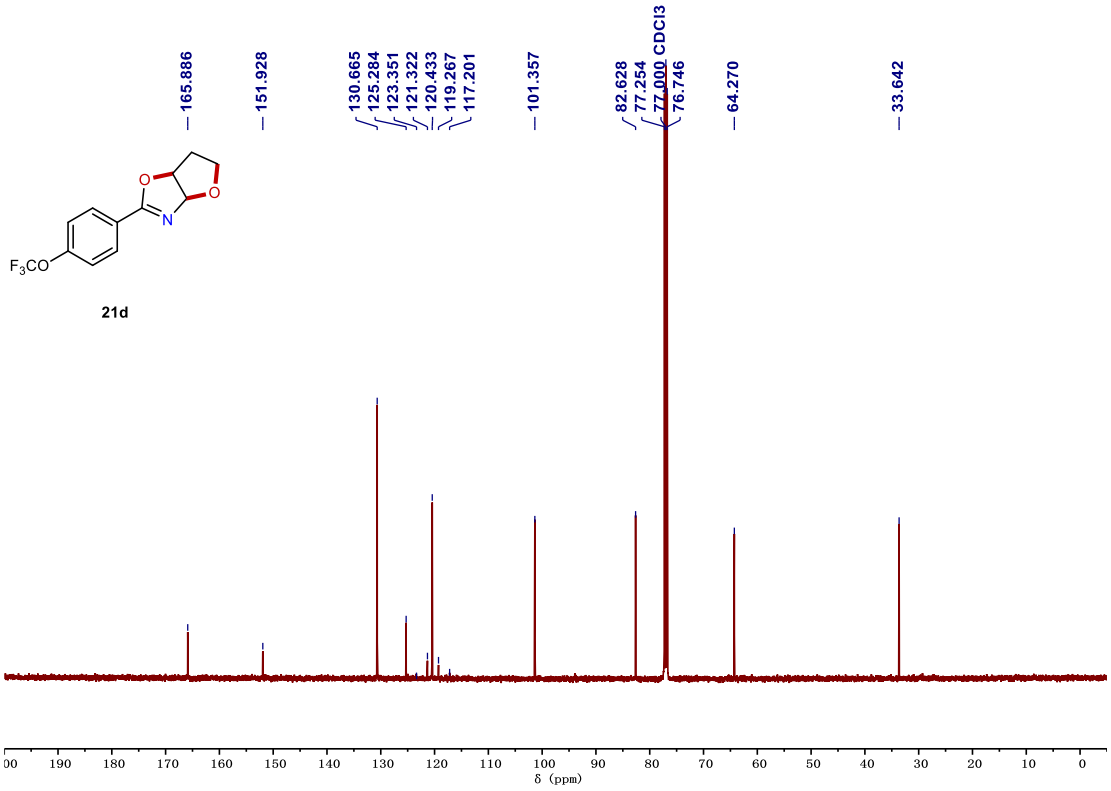

7.875
7.857
7.577
7.560
7.260 CDCl3
6.217
6.206
5.162
5.151
5.139
4.045
4.031
4.029
4.026
4.013
4.011
3.544
3.535
3.526
3.521
3.517
3.511
3.502
3.493
2.243
2.234
2.216
2.206
2.115
2.104
2.100
2.092
2.088
2.080
2.076
2.072
2.064
O
O
N
Br
21a
2.01
2.02
1.00
1.03
1.08
1.07
1.08
1.05
δ (ppm)

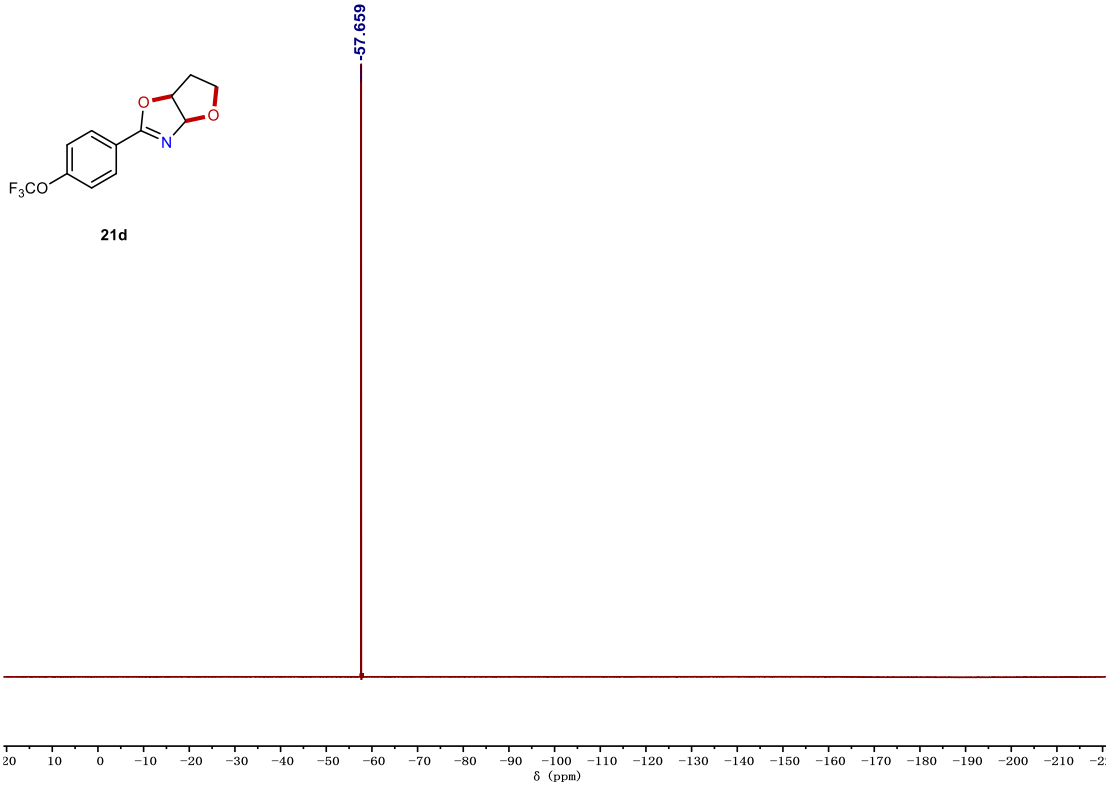

166.336
131.712
130.309
126.942
125.698
101.347
82.566
77.000 CDCl3
76.746
64.281
33.639
O
O
N
Br
21a
δ (ppm)


7.946
7.929
7.411
7.394
7.260 CDCl3
6.221
6.210
5.163
5.151
5.140
4.045
4.028
4.011
3.546
3.536
3.528
3.522
3.518
3.512
3.504
3.494
2.243
2.234
2.216
2.206
2.103
2.099
2.091
2.088
2.079
2.076
2.072
2.063
2.060
O
O
N
Cl
21b
2.06
2.07
1.00
1.04
1.07
1.08
1.07
1.08
δ (ppm)
166.227
138.387
130.145
128.726
125.241
101.341
82.550
77.254
77.000 CDCl3
76.746
64.268
33.640
O
O
N
Cl
21b
δ (ppm)

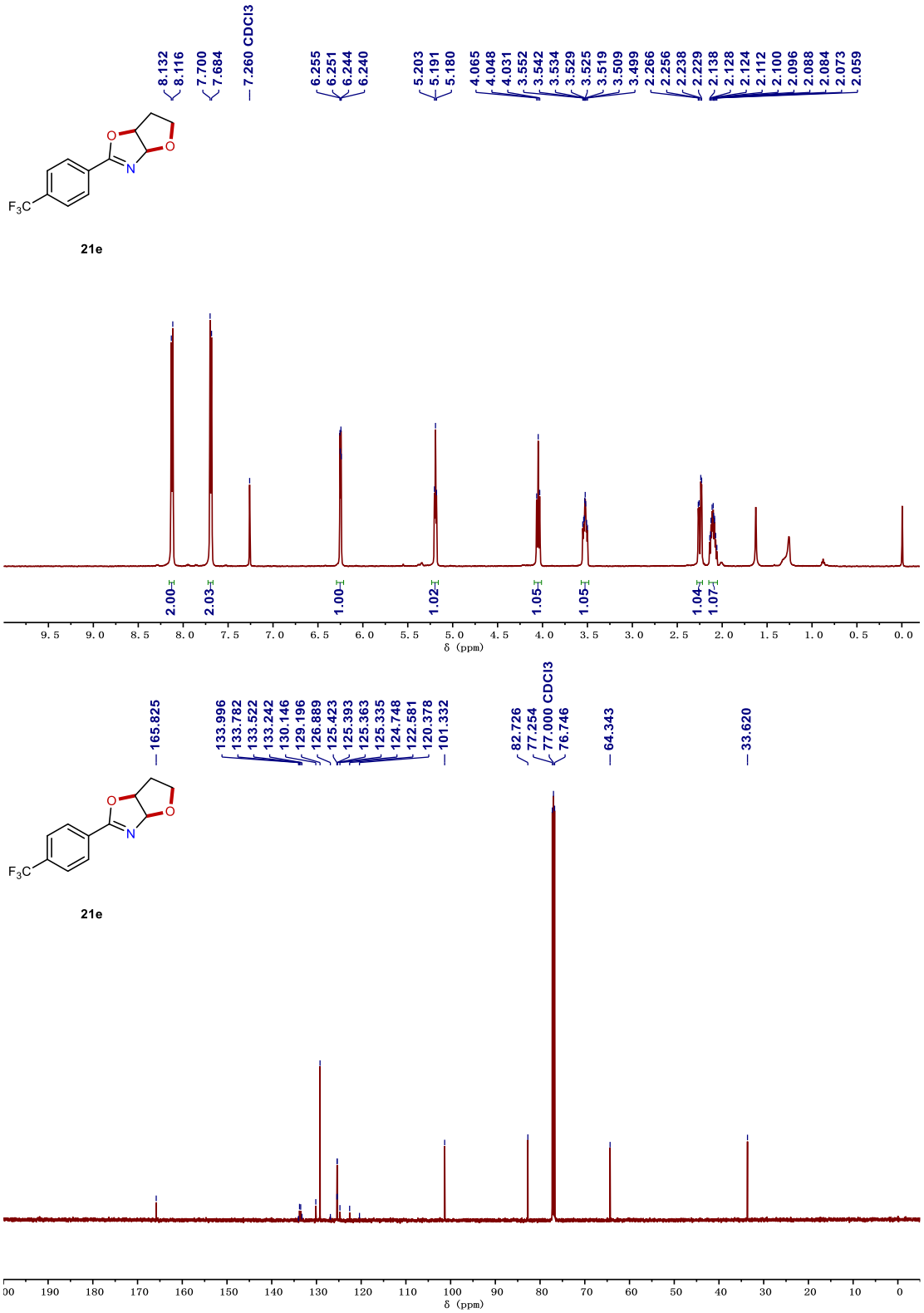

8.015
8.001
7.998
7.532
7.517
7.502
7.442
7.427
7.412
7.260 CDCl3
6.235
6.224
5.162
5.151
5.140
4.042
4.025
4.008
3.561
3.552
3.543
3.537
3.533
3.528
3.519
3.510
2.249
2.240
2.222
2.213
2.114
2.102
2.099
2.091
2.087
2.079
2.075
2.071
2.063
2.059
2.051
2.047
2.035
21c
2.06
1.05
2.05
1.00
1.03
1.06
1.06
1.05
1.08
δ (ppm)
167.116
132.073
128.801
128.373
126.761
101.349
82.292
77.254
77.000 CDCl3
76.746
64.209
33.699
21c
δ (ppm)

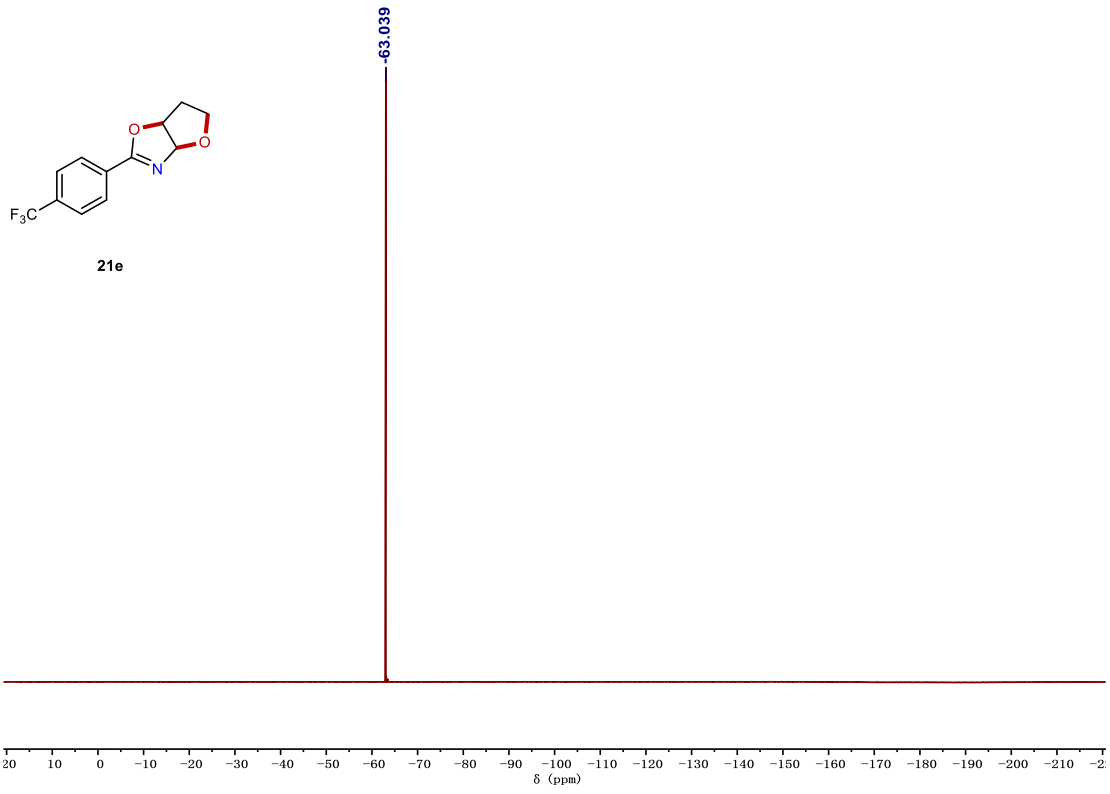

8.058
8.041
7.271
7.269
7.260 CDCl3
7.253
7.251
6.231
6.220
5.176
5.165
5.153
4.052
4.050
4.036
4.034
4.032
4.018
4.016
3.547
3.537
3.529
3.523
3.519
3.513
3.505
3.495
2.246
2.237
2.219
2.209
2.122
2.110
2.106
2.098
2.094
2.086
2.082
2.078
2.070
F3CO
21d
2.03
2.02
1.00
1.02
1.04
1.04
1.07
1.07
δ (ppm)

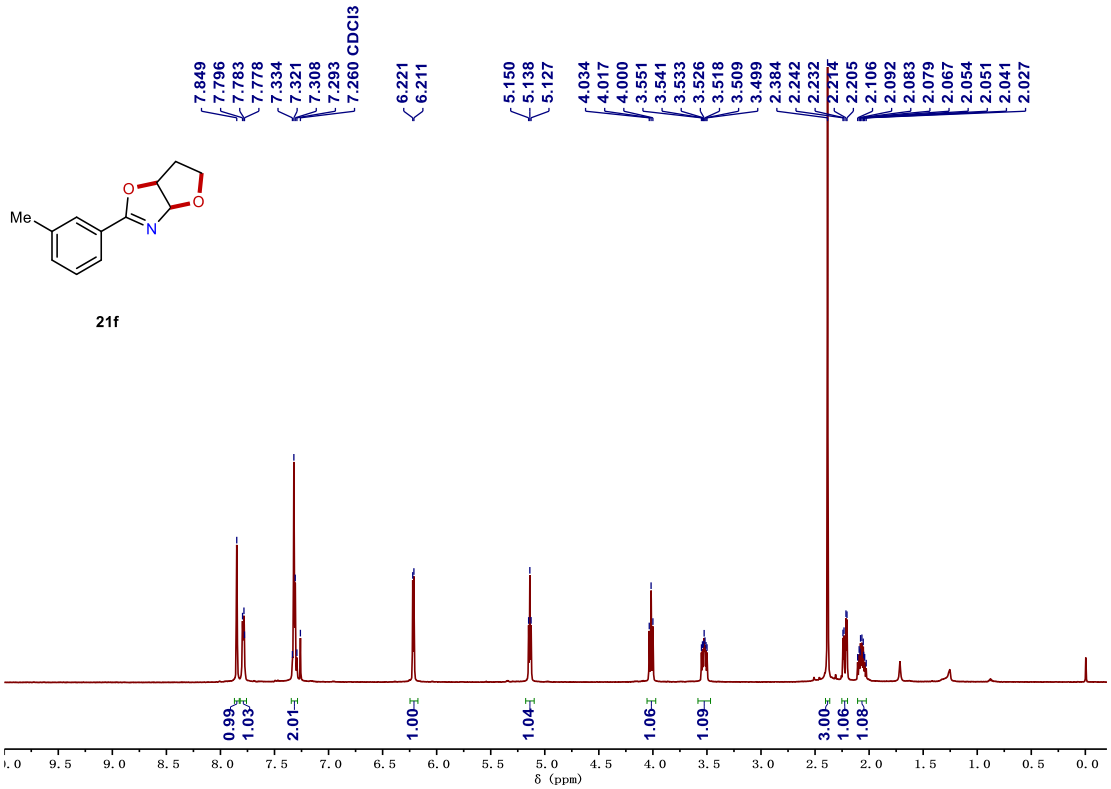

165.886
151.928
130.665
125.284
123.351
121.322
120.433
119.267
117.201
101.357
82.628
77.254
77.000 CDCl3
76.746
64.270
33.642
F3CO
21d
δ (ppm)

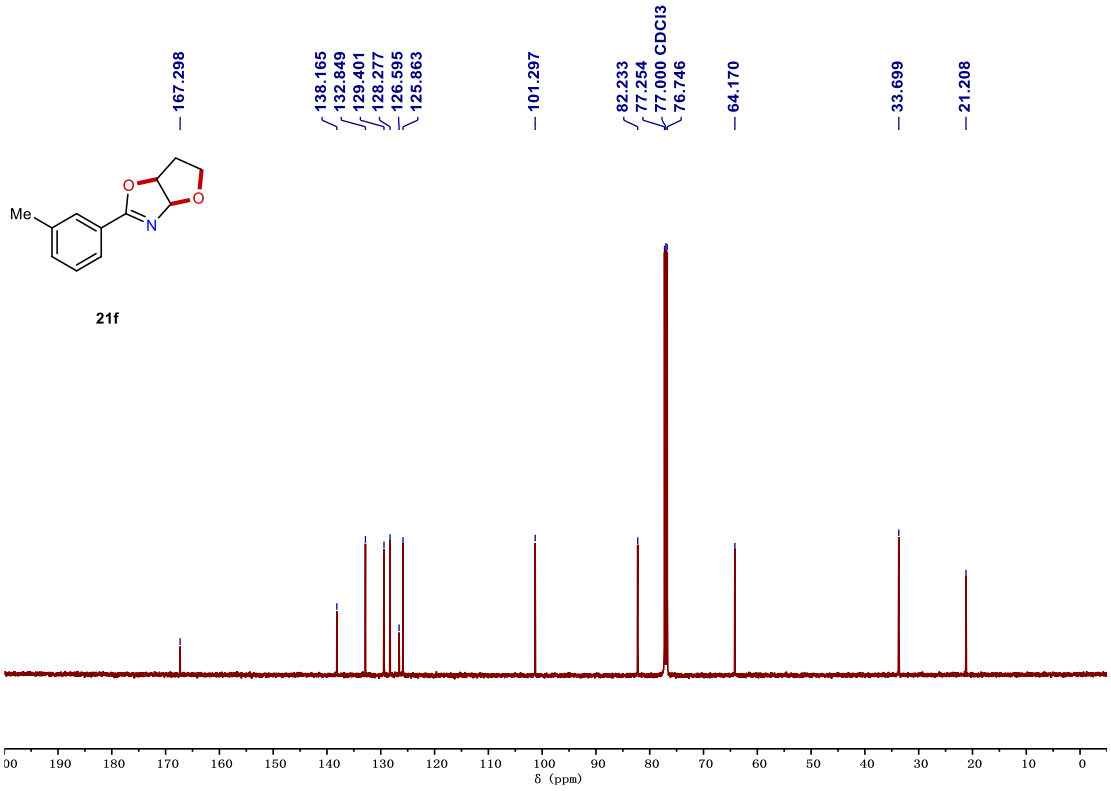

-57.659
O
O
N
F3CO
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)

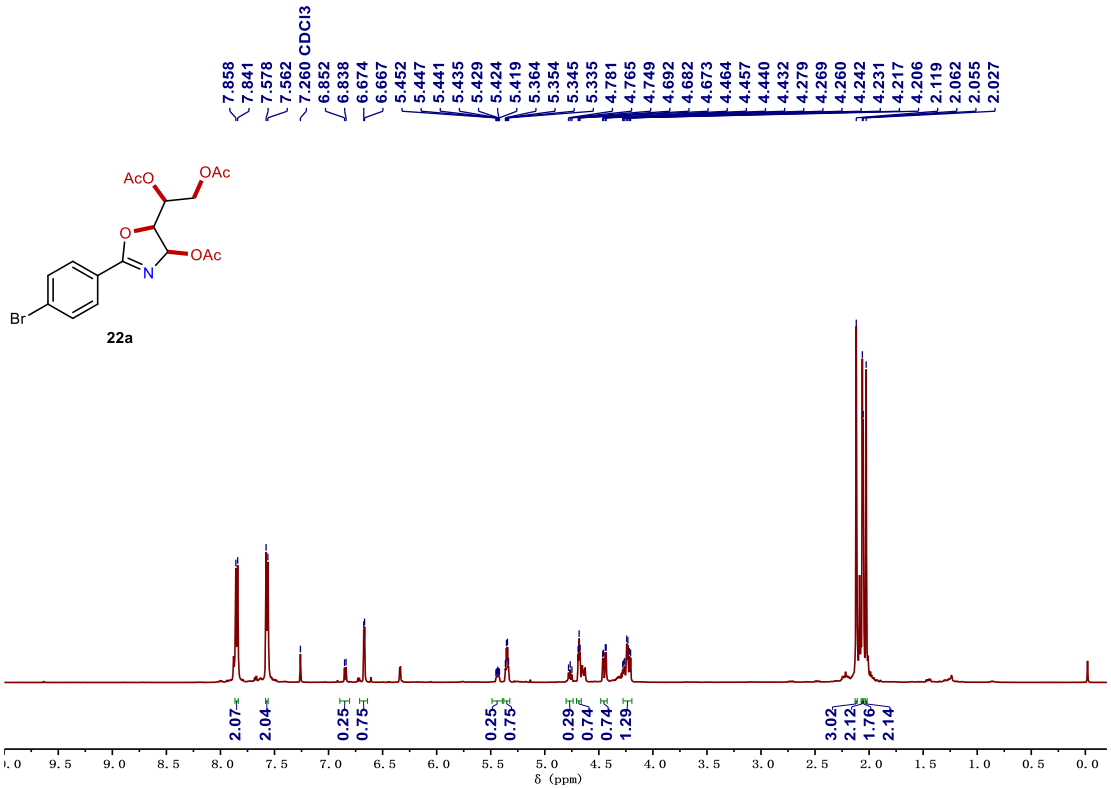
21d

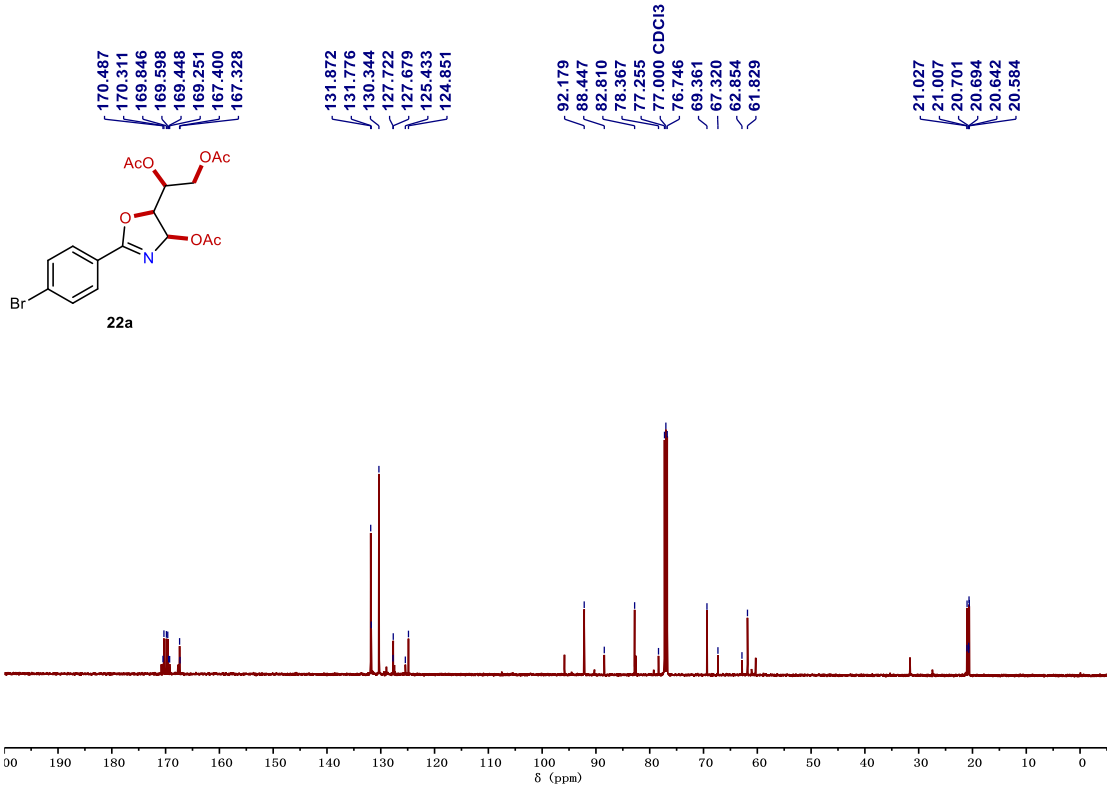

8.132
8.116
7.700
7.684
7.260 CDCl3
6.255
6.251
6.244
6.240
5.203
5.191
5.180
4.065
4.048
4.031
3.552
3.542
3.534
3.529
3.525
3.519
3.509
3.499
2.266
2.256
2.238
2.229
2.138
2.128
2.124
2.112
2.100
2.096
2.088
2.084
2.073
2.059
O
O
N
F3C
21e
2.00
2.03
1.00
1.02
1.05
1.05
1.04
1.07
δ (ppm)
165.825
133.996
133.782
133.522
133.242
130.146
129.196
126.889
125.423
125.393
125.363
125.335
124.748
122.581
120.378
101.332
82.726
77.254
77.000 CDCl3
76.746
64.343
33.620
O
O
N
F3C
21e
δ (ppm)

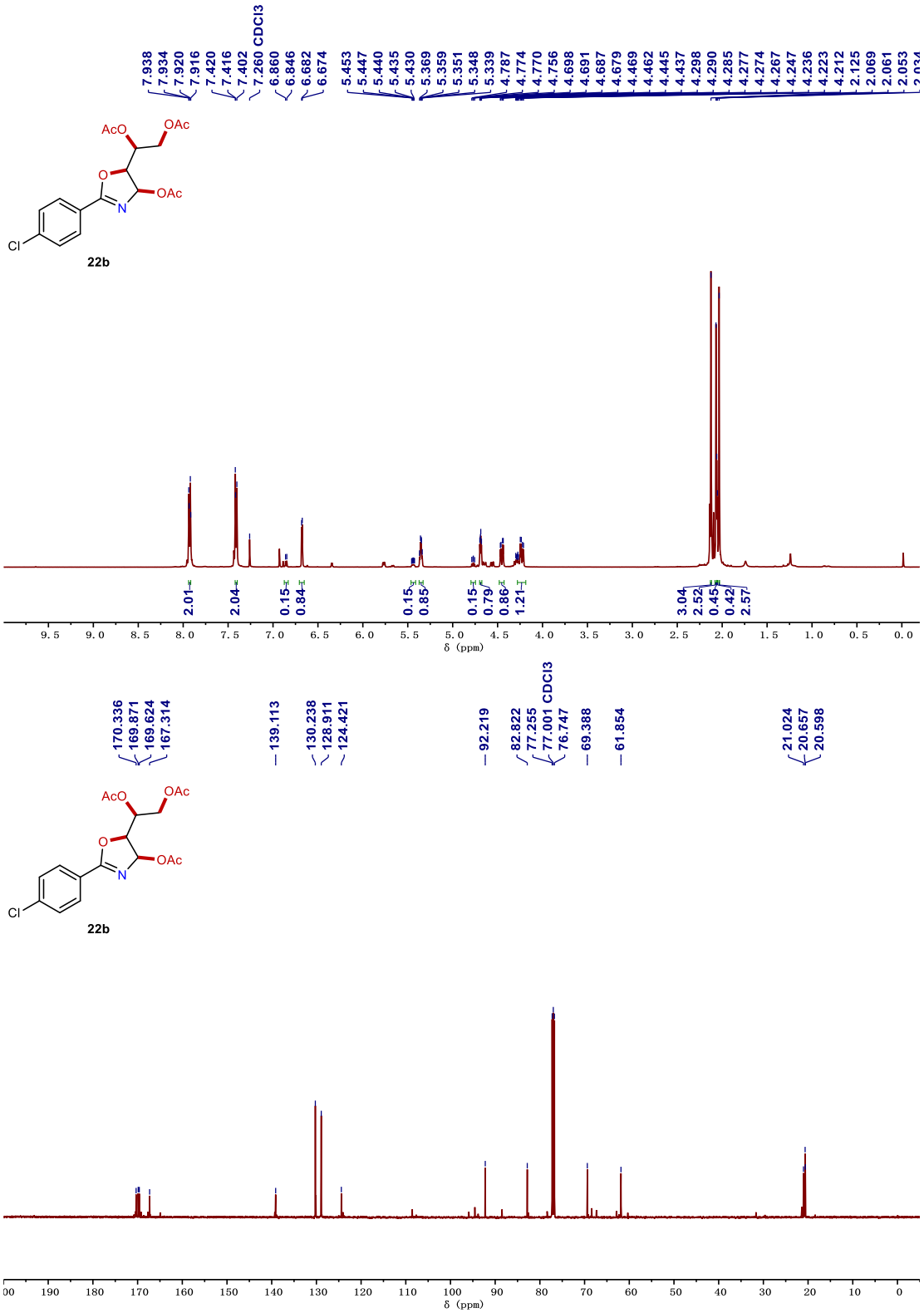

-63.039
O
O
N
F3C
21e
20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
δ (ppm)

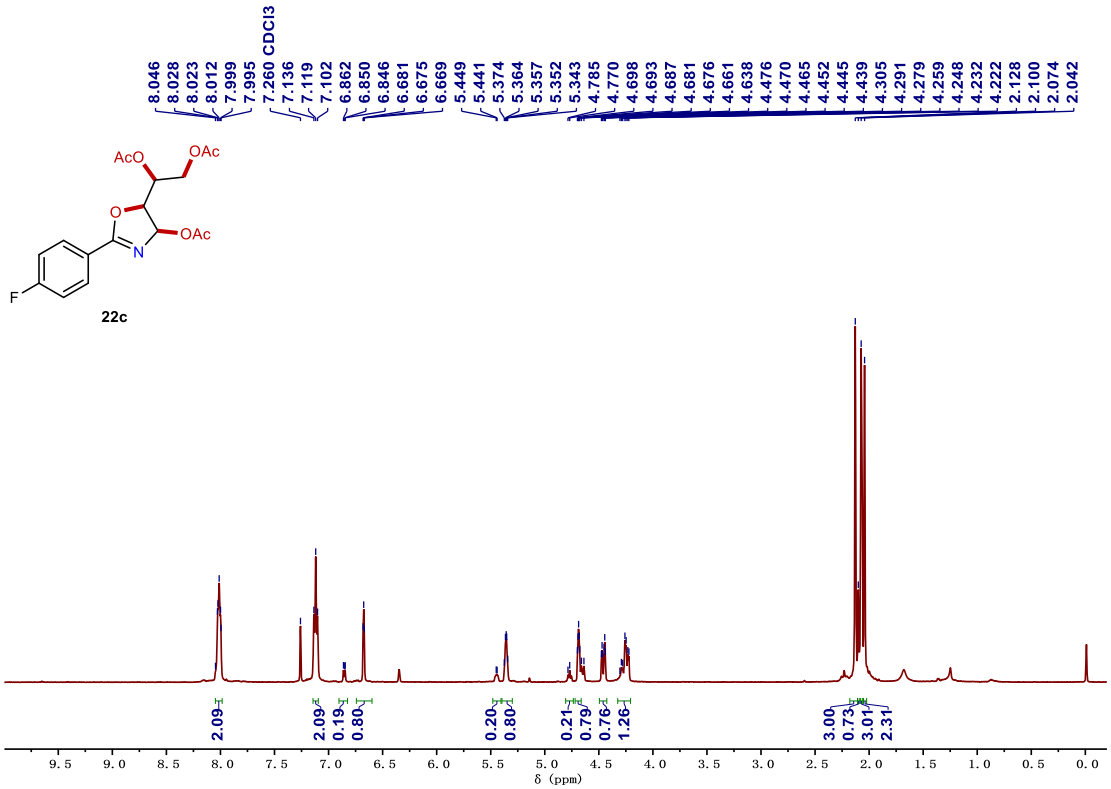

7.849
7.796
7.783
7.778
7.334
7.321
7.308
7.293
7.260 CDCl3
6.221
6.211
5.150
5.138
5.127
4.034
4.017
4.000
3.551
3.541
3.533
3.526
3.518
3.509
3.499
2.384
2.242
2.232
2.214
2.205
2.106
2.092
2.083
2.079
2.067
2.054
2.051
2.041
2.027
Me
O
O
N
21f
0.99
1.03
2.01
1.00
1.04
1.06
1.09
3.00
1.06
1.08
δ (ppm)


167.298
138.165
132.849
129.401
128.277
126.595
125.863
101.297
82.233
77.254
77.000 CDCl3
76.746
64.170
33.699
21.208
Me
O
O
N
21f
δ (ppm)


7.858
7.841
7.578
7.562
7.260 CDCl3
6.852
6.838
6.674
6.667
5.452
5.447
5.441
5.435
5.429
5.424
5.419
5.364
5.354
5.345
5.335
4.781
4.765
4.749
4.692
4.682
4.673
4.464
4.457
4.440
4.432
4.279
4.269
4.260
4.242
4.231
4.217
4.206
2.119
2.062
2.055
2.027
AcO
OAc
O
OAc
N
Br
22a
2.07
2.04
0.25
0.75
0.25
0.75
0.29
0.74
0.74
1.29
3.02
2.12
1.76
2.14
δ (ppm)


170.487
170.311
169.846
169.598
169.448
169.251
167.400
167.328
131.872
131.776
130.344
127.722
127.679
125.433
124.851
92.179
88.447
82.810
78.367
77.255
77.000 CDCl3
76.746
69.361
67.320
62.854
61.829
21.027
21.007
20.701
20.694
20.642
20.584
AcO
OAc
O
OAc
N
Br
22a
δ (ppm)


7.938
7.934
7.920
7.916
7.420
7.416
7.402
7.260 CDCl3
6.860
6.846
6.682
6.674
5.453
5.447
5.440
5.435
5.430
5.369
5.359
5.351
5.348
5.339
4.787
4.774
4.770
4.756
4.698
4.691
4.687
4.679
4.469
4.462
4.445
4.437
4.298
4.290
4.285
4.277
4.274
4.267
4.247
4.236
4.223
4.212
2.125
2.069
2.061
2.053
AcO
OAc
O
N
OAc
Cl
22b
2.01
2.04
0.15
0.84
0.15
0.85
0.15
0.79
0.86
1.21
3.04
2.52
0.45
0.42
2.57
δ (ppm)
170.336
169.871
169.624
167.314
139.113
130.238
128.911
124.421
92.219
82.822
77.255
77.001 CDCl3
76.747
69.388
61.854
21.024
20.657
20.598
AcO
OAc
O
N
OAc
Cl
22b
δ (ppm)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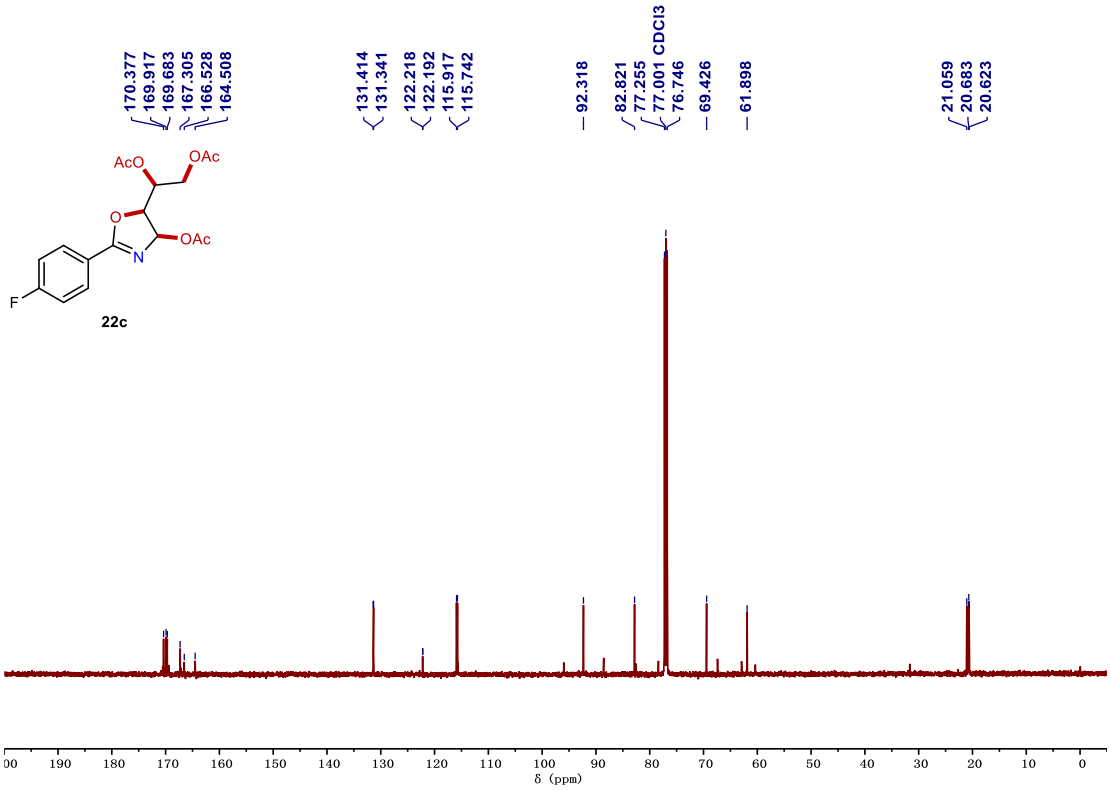
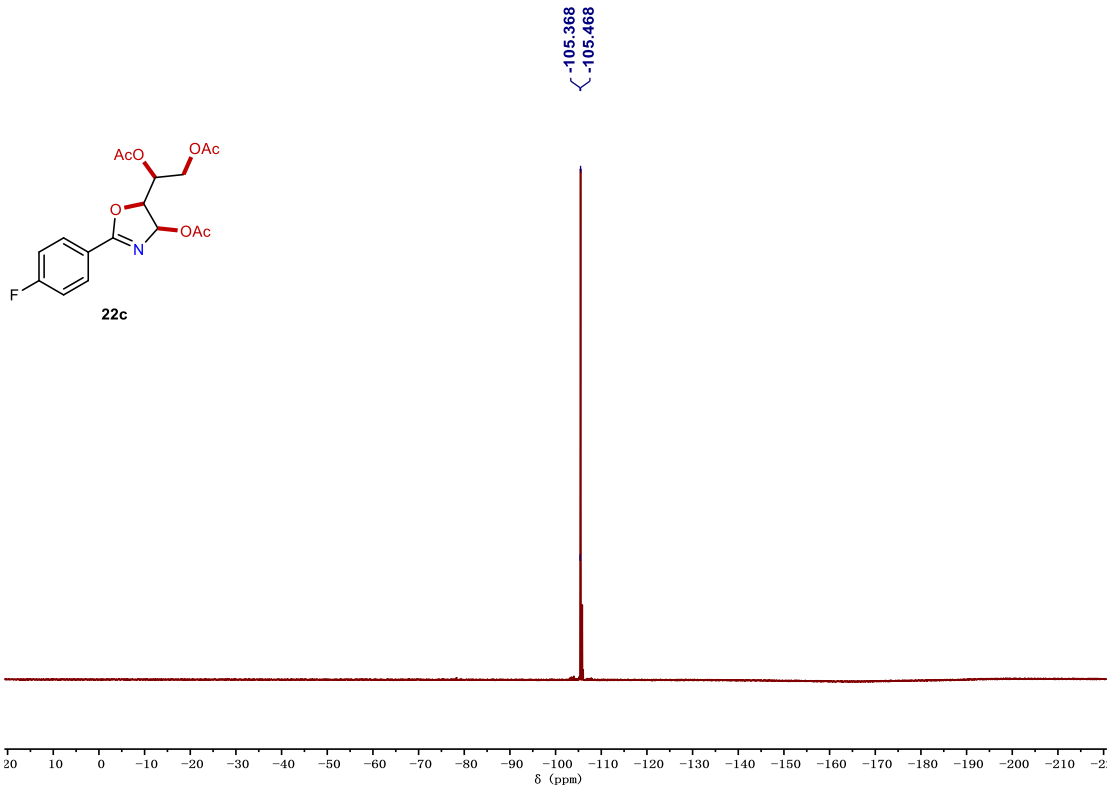
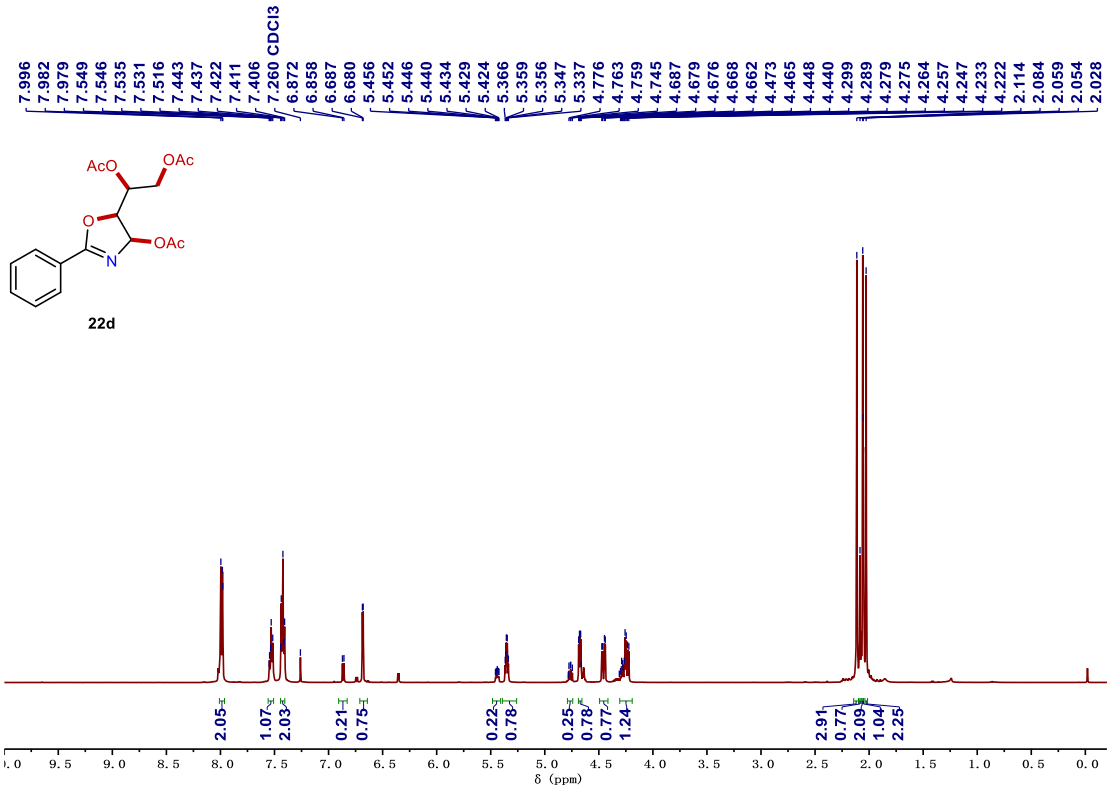
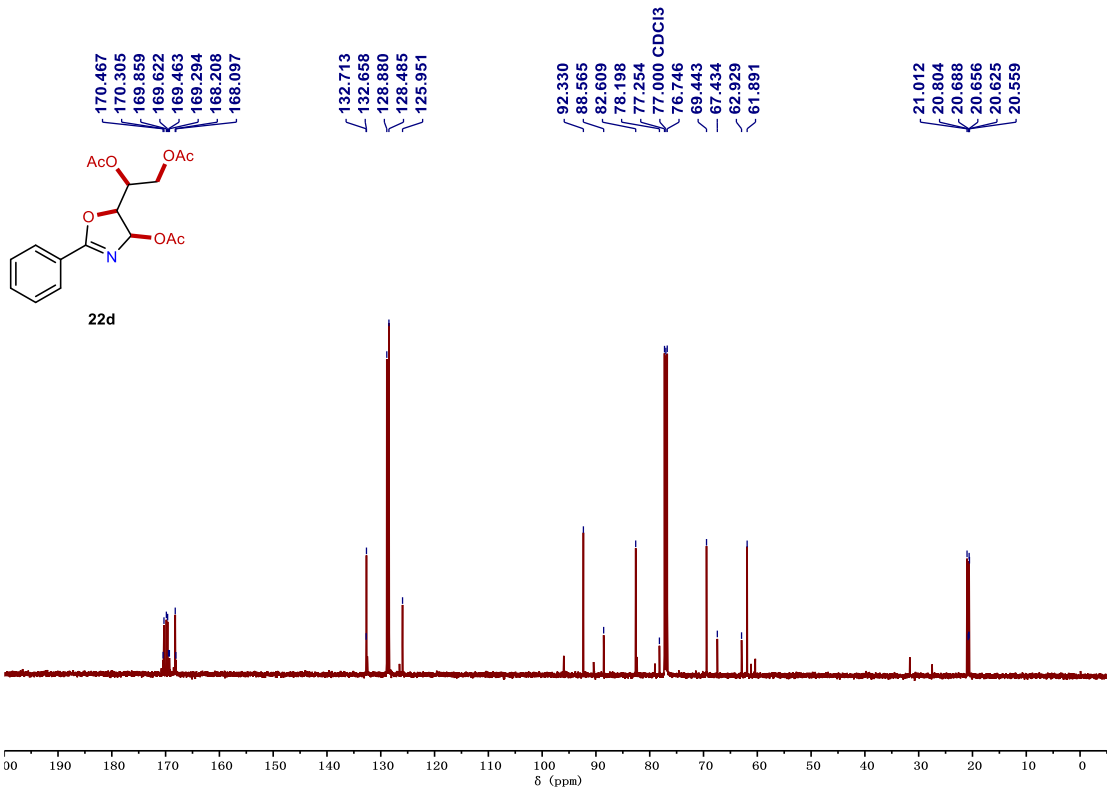
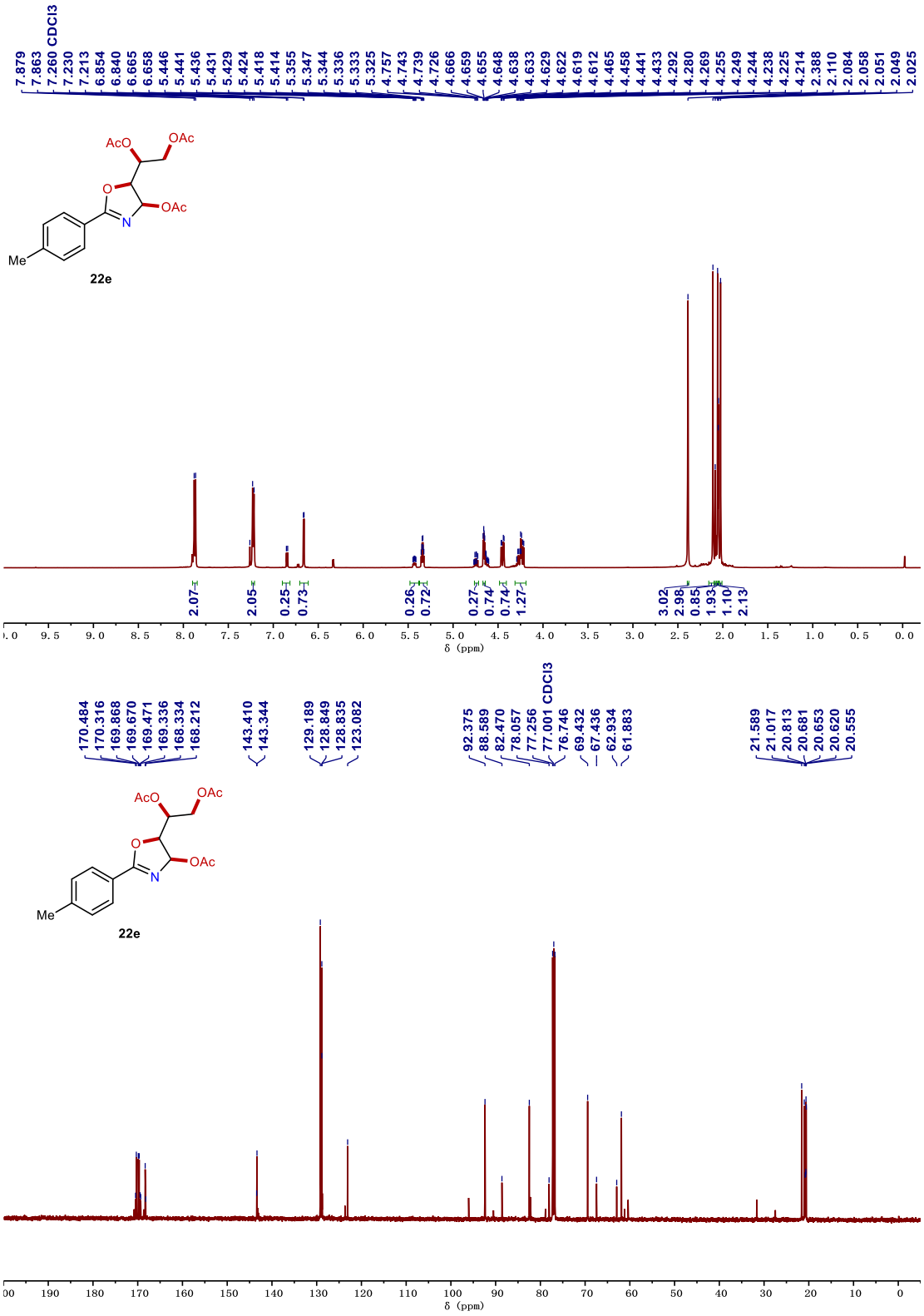
8.028
8.023
8.012
7.999
7.995
7.260 CDCl3
7.136
7.119
7.102
6.862
6.850
6.846
6.681
6.675
6.669
5.449
5.441
5.374
5.364
5.357
5.352
5.343
4.785
4.770
4.698
4.693
4.687
4.681
4.676
4.661
4.638
4.476
4.470
4.465
4.452
4.445
4.439
4.305
4.291
4.279
4.259
4.248
4.232
4.222
2.128
2.100
2.074
2.042
AcO
OAc
O
OAc
N
F
22c
2.09
2.09
0.19
0.80
0.20
0.80
0.21
0.79
0.76
1.26
3.00
0.73
3.01
2.31
δ (ppm)

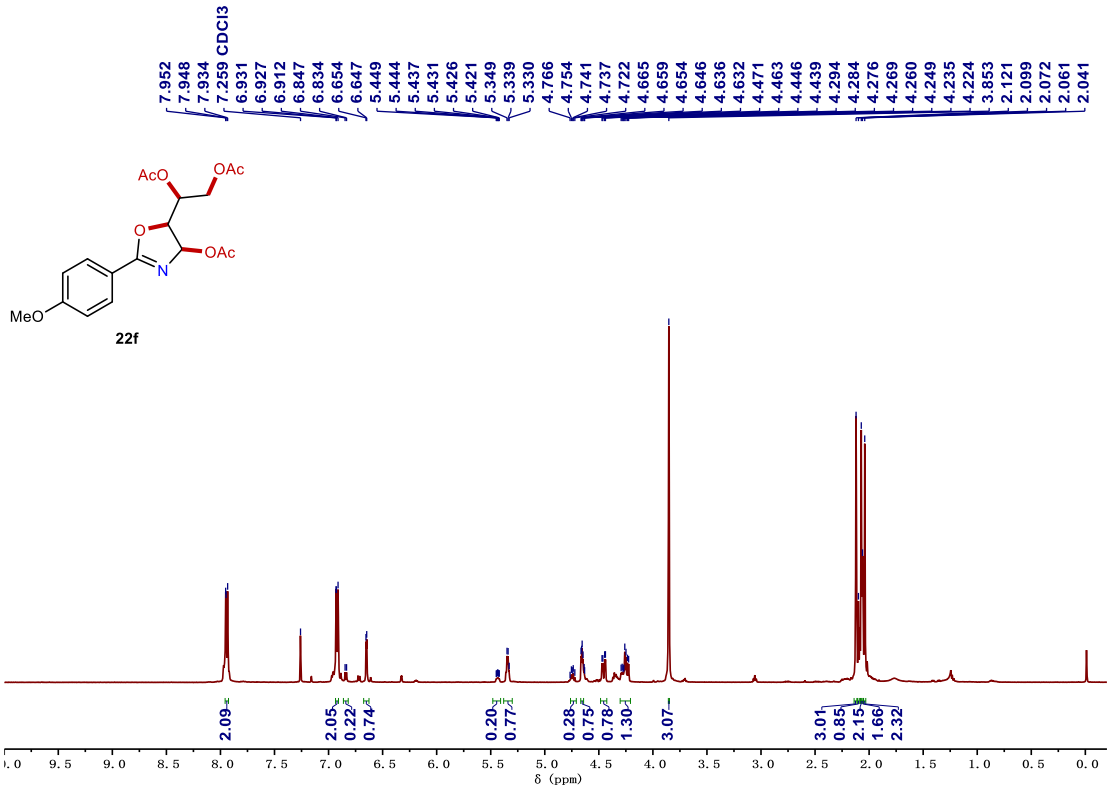

170.377
169.917
169.683
167.305
166.528
164.508
131.414
131.341
122.218
122.192
115.917
115.742
92.318
82.821
77.255
77.001 CDCl3
76.746
69.426
61.898
21.059
20.683
20.623
AcO
OAc
O
OAc
N
F
22c
δ (ppm)

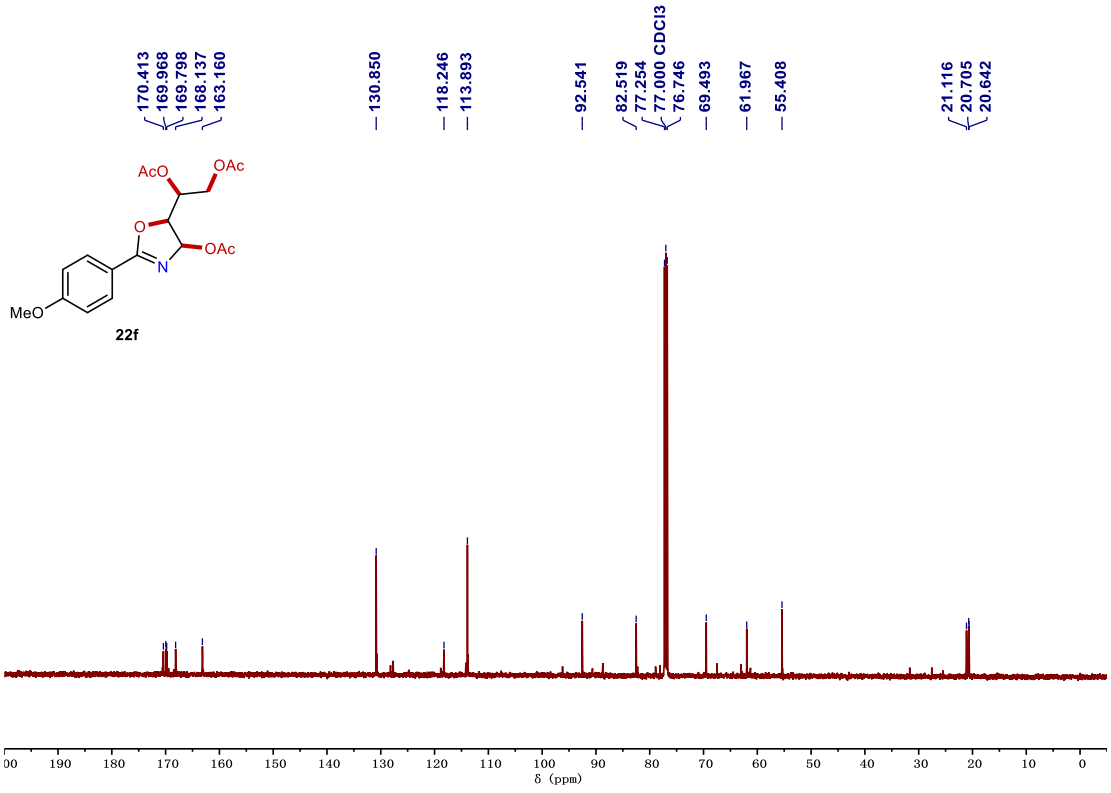

-105.368
-105.468
AcO
OAc
O
N
OAc
F
22c
20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
δ (ppm)

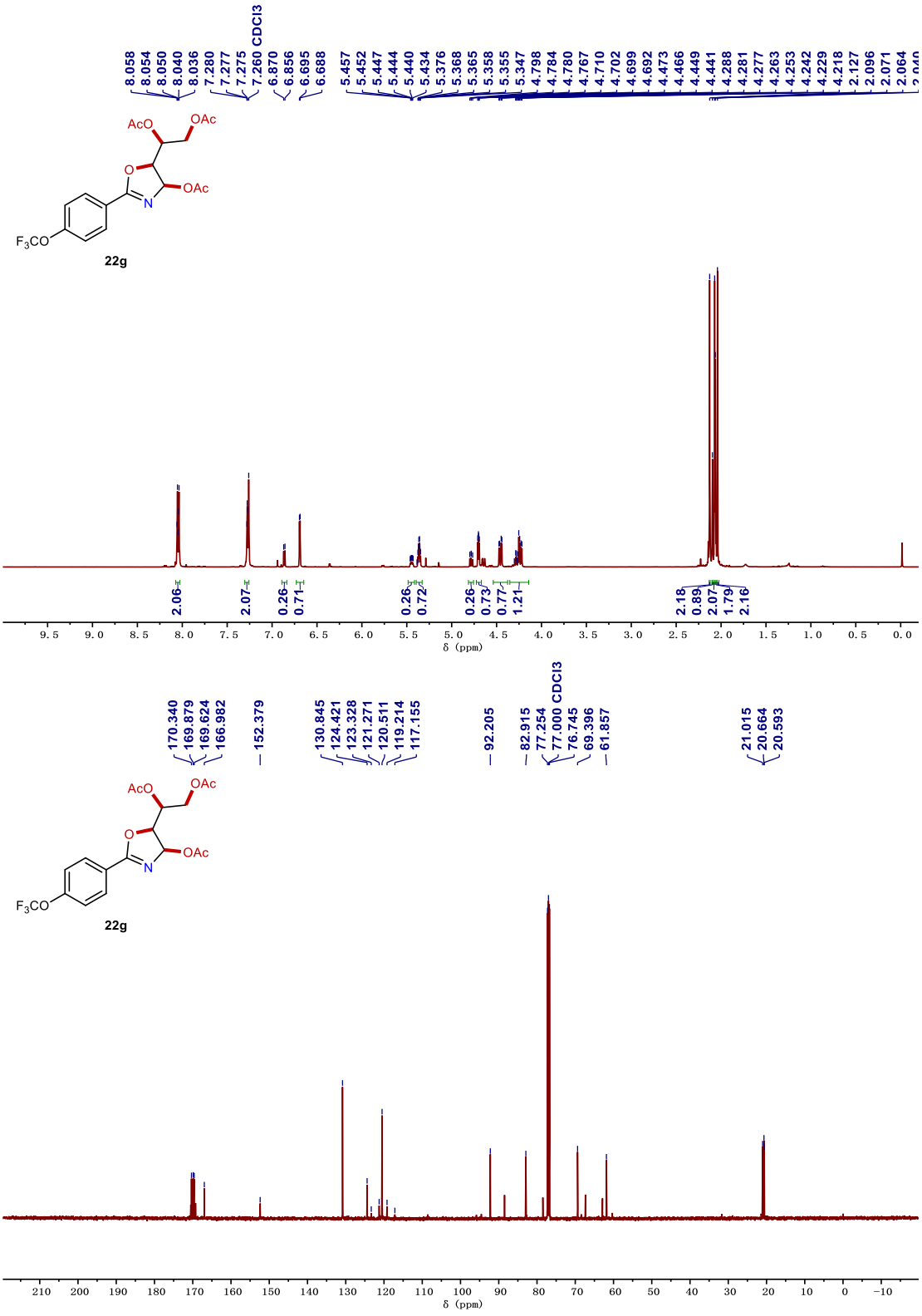

7.996
7.982
7.979
7.549
7.546
7.535
7.531
7.516
7.443
7.437
7.422
7.411
7.406
7.260 CDCl3
6.872
6.858
6.687
6.680
5.456
5.452
5.446
5.440
5.434
5.429
5.424
5.366
5.359
5.356
5.347
5.337
4.776
4.763
4.759
4.745
4.687
4.679
4.676
4.668
4.662
4.473
4.465
4.448
4.440
4.299
4.289
4.279
4.275
4.264
4.257
4.247
4.233
4.222
2.114
2.084
2.059
2.054
2.028
AcO
OAc
O
OAc
N
22d
2.05
1.07
2.03
0.21
0.75
0.22
0.78
0.25
0.78
0.77
1.24
2.91
0.77
2.09
1.04
2.25
δ (ppm)

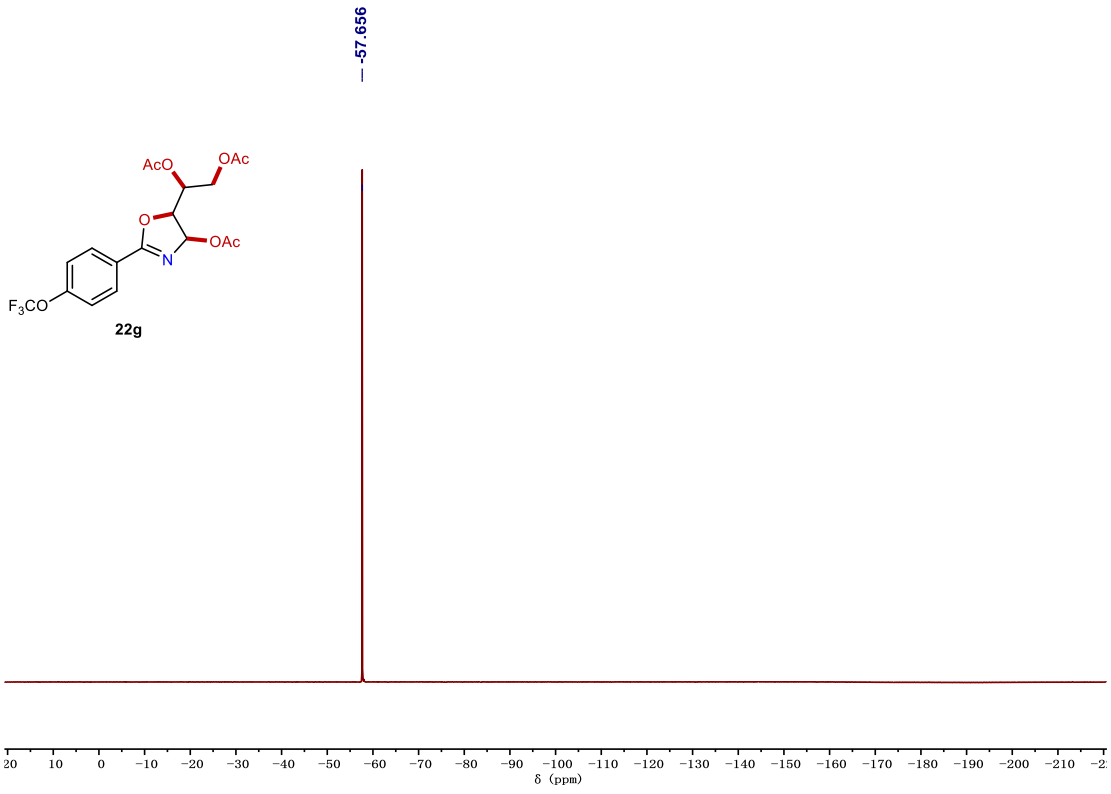

170.467
170.305
169.859
169.622
169.463
169.294
168.208
168.097
132.713
132.658
128.880
128.485
125.951
92.330
88.565
82.609
78.198
77.254
77.000 CDCl3
76.746
69.443
67.434
62.929
61.891
21.012
20.804
20.688
20.656
20.625
20.559
AcO
OAc
O
OAc
N
22d
δ (ppm)

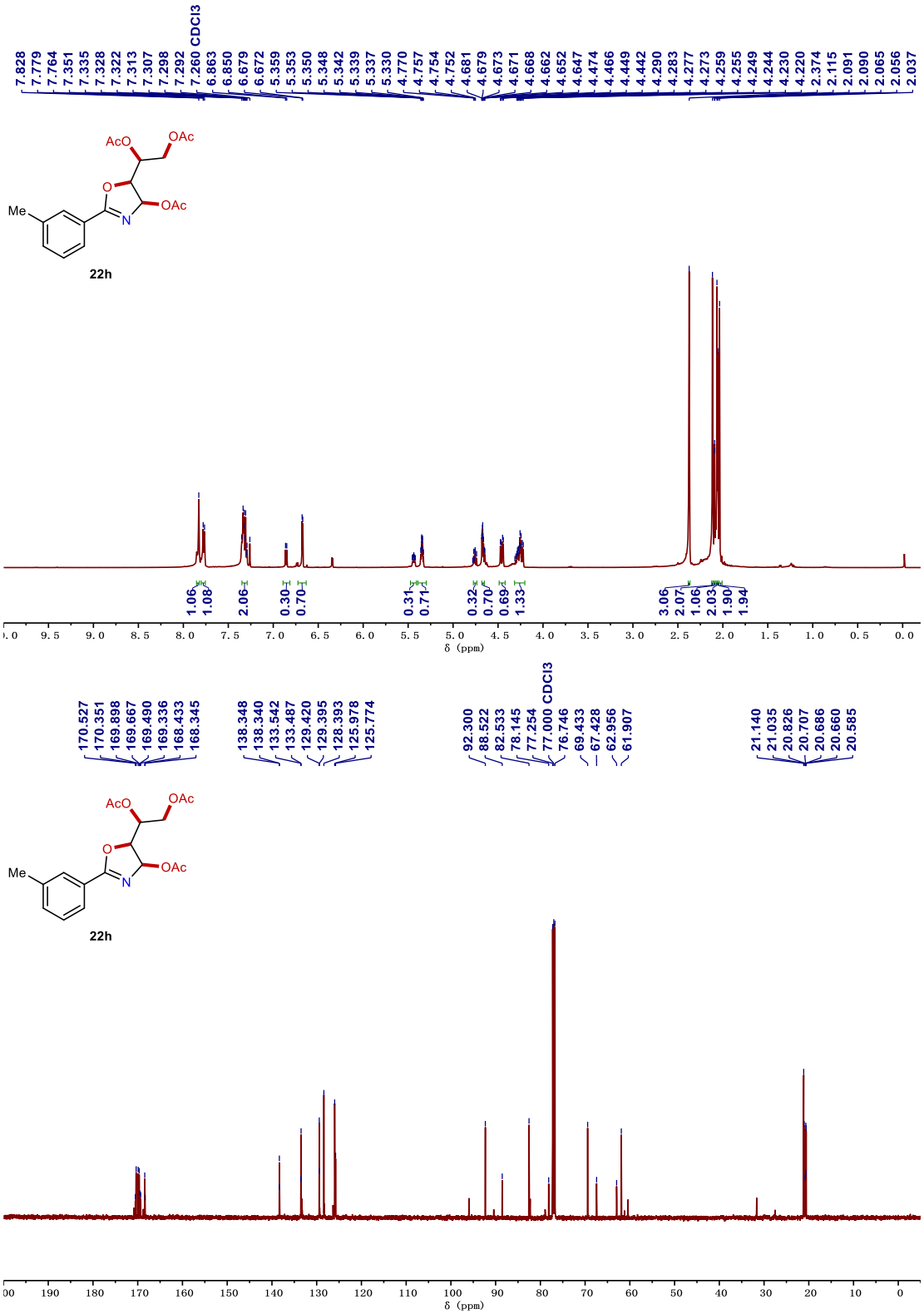

7.879
7.863
7.260 CDCl3
7.230
7.213
6.854
6.840
6.665
6.658
6.446
5.441
5.436
5.431
5.429
5.424
5.418
5.414
5.355
5.347
5.344
5.336
5.333
5.325
4.757
4.743
4.739
4.726
4.666
4.659
4.655
4.648
4.638
4.633
4.629
4.622
4.619
4.612
4.465
4.458
4.441
4.433
4.292
4.280
4.269
4.255
4.249
4.244
4.238
4.225
4.214
2.388
2.110
2.084
2.058
2.051
2.049
2.025
AcO
OAc
O
OAc
N
Me
22e
2.07
2.05
0.25
0.73
0.26
0.72
0.27
0.74
0.74
1.27
3.02
2.98
0.85
1.93
1.10
2.13
δ (ppm)
170.484
170.316
169.868
169.670
169.471
169.336
168.334
168.212
143.410
143.344
129.189
128.849
128.835
123.082
92.375
88.589
82.470
78.057
77.256
77.001 CDCl3
76.746
69.432
67.436
62.934
61.883
21.589
21.017
20.813
20.681
20.653
20.620
20.555
AcO
OAc
O
OAc
N
Me
22e
δ (ppm)

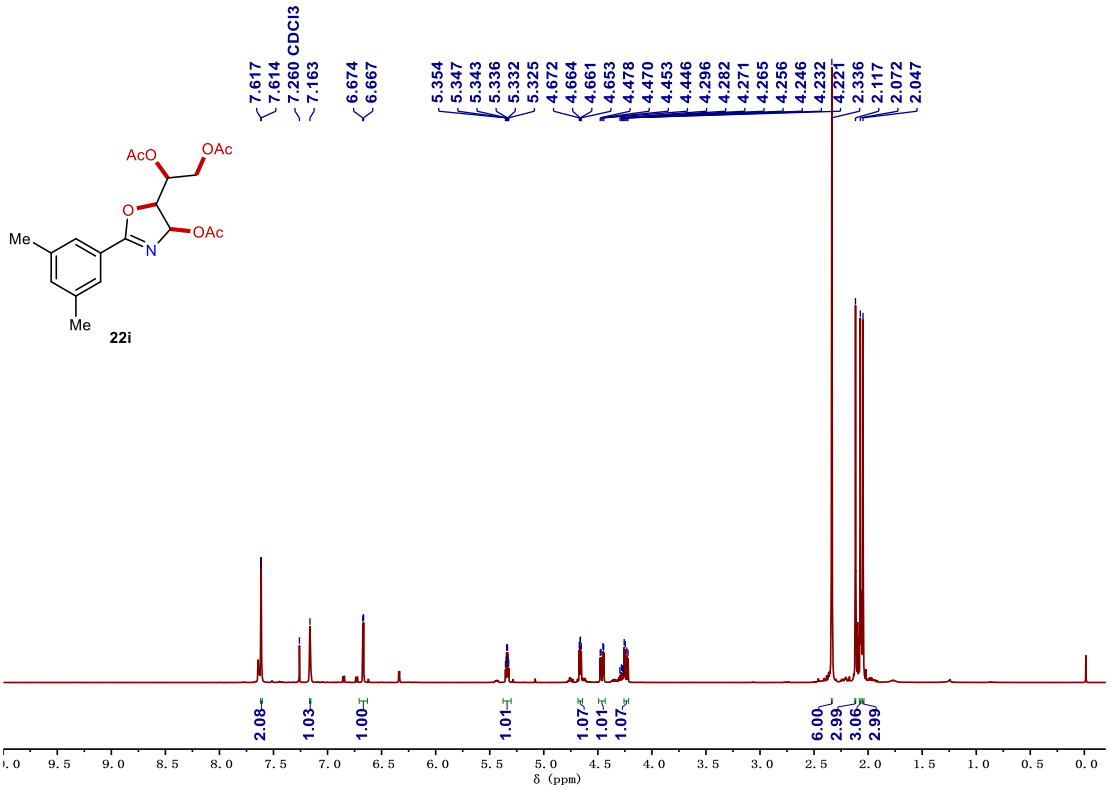

7.952
7.948
7.934
7.259 CDCl3
6.931
6.927
6.912
6.847
6.834
6.654
6.647
5.449
5.444
5.437
5.431
5.426
5.421
5.349
5.339
5.330
4.766
4.754
4.741
4.737
4.722
4.665
4.659
4.654
4.646
4.636
4.632
4.471
4.463
4.446
4.439
4.294
4.284
4.276
4.269
4.260
4.249
4.235
4.224
3.853
2.121
2.099
2.072
2.061
2.041
AcO
OAc
O
OAc
N
MeO
22f
2.09
2.05
0.22
0.74
0.20
0.77
0.28
0.75
0.78
1.30
3.07
3.01
0.85
2.15
1.66
2.32
δ (ppm)

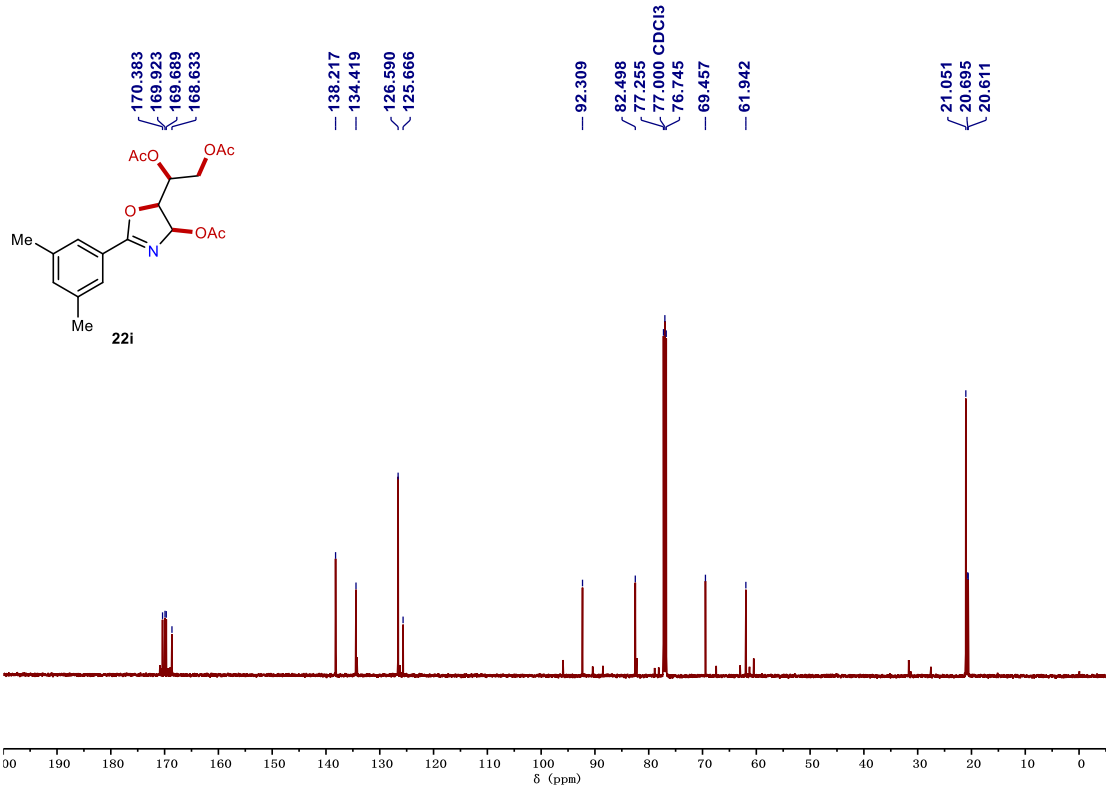

170.413
169.968
169.798
168.137
163.160
130.850
118.246
113.893
92.541
82.519
77.254
77.000 CDCl3
76.746
69.493
61.967
55.408
21.116
20.705
20.642
AcO
OAc
O
OAc
N
MeO
22f
δ (ppm)

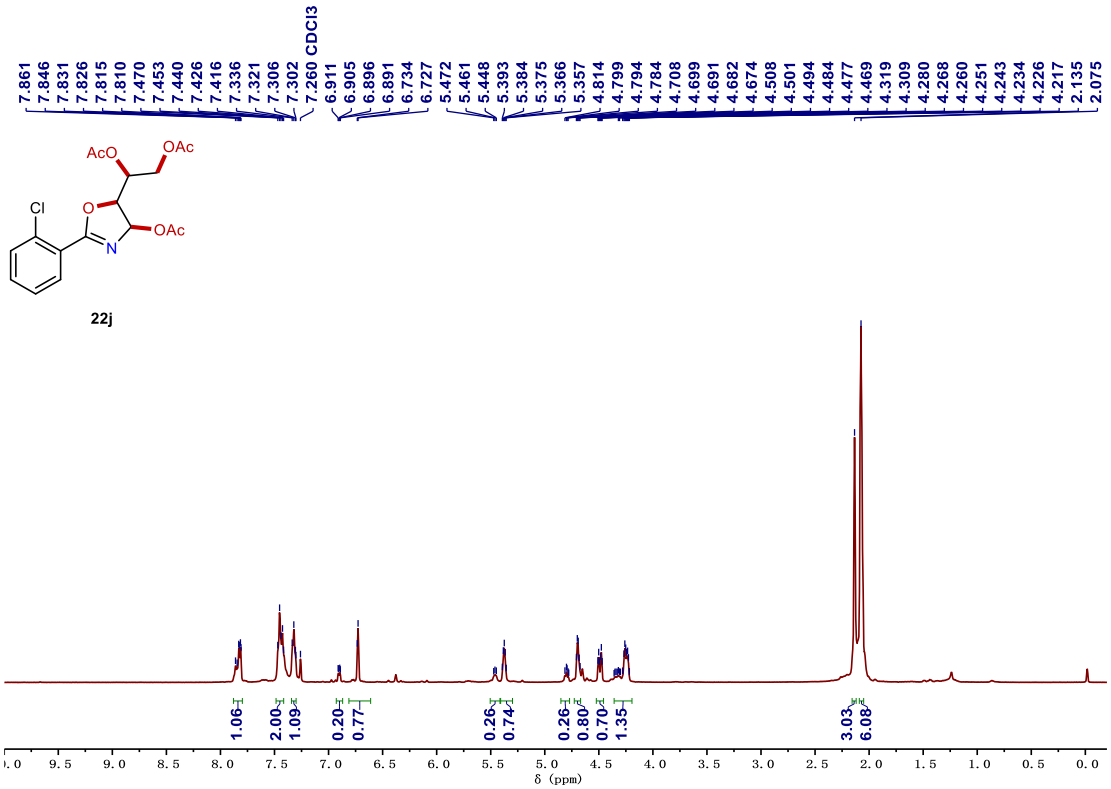

8.058
8.054
8.050
8.040
8.036
7.280
7.277
7.275
7.260 CDCl3
6.870
6.856
6.695
6.688
5.457
5.452
5.447
5.444
5.440
5.434
5.376
5.368
5.365
5.358
5.355
5.347
4.798
4.784
4.780
4.767
4.710
4.702
4.699
4.692
4.473
4.466
4.449
4.441
4.288
4.281
4.277
4.263
4.253
4.242
4.229
4.218
2.127
2.096
2.071
2.064
AcO
OAc
O
OAc
N
F3CO
22g
2.06
2.07
0.26
0.71
0.26
0.72
0.26
0.73
0.77
1.21
2.18
0.89
2.07
1.79
2.16
δ (ppm)
170.340
169.879
169.624
166.982
152.379
130.845
124.421
123.328
121.271
120.511
119.214
117.155
92.205
82.915
77.254
77.000 CDCl3
76.745
69.396
61.857
21.015
20.664
20.593
δ (ppm)


-57.656
AcO
OAc
O
OAc
N
F3CO
22g
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)

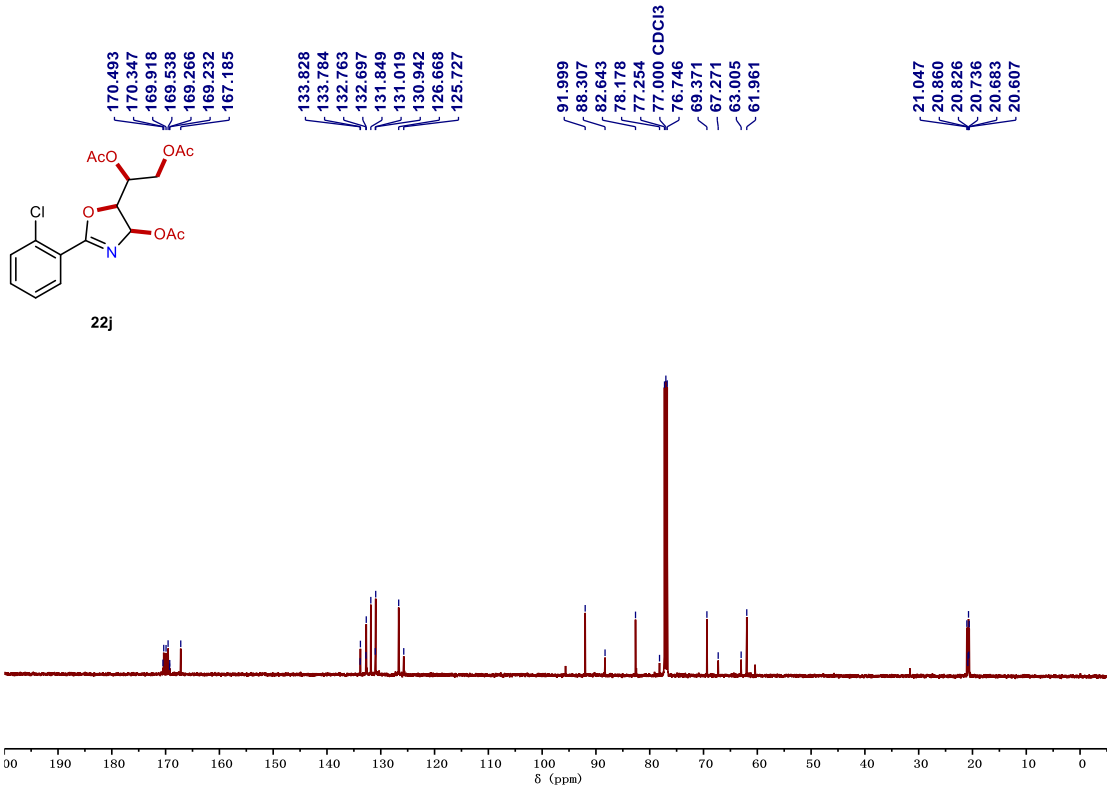

7.828
7.779
7.764
7.351
7.335
7.328
7.322
7.313
7.307
7.298
7.292
7.260 CDCl3
6.863
6.850
6.679
6.672
5.359
5.353
5.350
5.348
5.342
5.339
5.337
5.330
4.770
4.757
4.754
4.752
4.681
4.679
4.673
4.671
4.668
4.662
4.652
4.647
4.474
4.466
4.449
4.442
4.290
4.283
4.277
4.273
4.259
4.255
4.249
4.244
4.230
4.220
2.374
2.115
2.091
2.090
2.065
2.056
2.037
AcO
OAc
O
OAc
Me
N
22h
1.06
1.08
2.06
0.30
0.70
0.31
0.71
0.32
0.70
0.69
1.33
3.06
2.07
1.06
2.03
1.90
1.94
δ (ppm)
170.527
170.351
169.898
169.667
169.490
169.336
168.433
168.345
138.348
138.340
133.542
133.487
129.420
129.395
128.393
125.978
125.774
92.300
88.522
82.533
78.145
77.254
77.000 CDCl3
76.746
69.433
67.428
62.956
61.907
21.140
21.035
20.826
20.707
20.686
20.660
20.585
AcO
OAc
O
OAc
Me
N
22h
δ (ppm)


7.617
7.614
7.260 CDCl3
7.163
6.674
6.667
5.354
5.347
5.343
5.336
5.332
5.325
4.672
4.664
4.661
4.653
4.478
4.470
4.453
4.446
4.296
4.282
4.271
4.265
4.256
4.246
4.232
4.221
2.336
2.117
2.072
2.047
AcO
OAc
O
OAc
N
Me
Me
22i
2.08
1.03
1.00
1.01
1.07
1.01
1.07
6.00
2.99
3.06
2.99
δ (ppm)

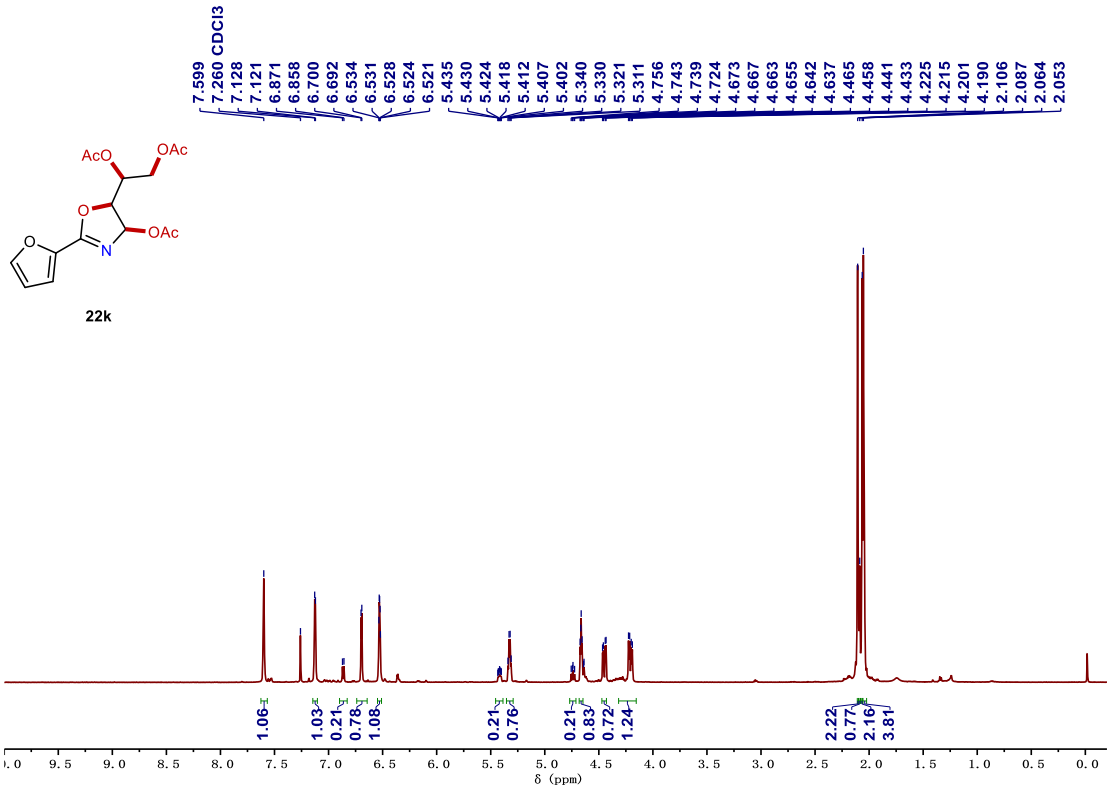

170.383
169.923
169.689
168.633
138.217
134.419
126.590
125.666
92.309
82.498
77.255
77.000 CDCl3
76.745
69.457
61.942
21.051
20.695
20.611
AcO
OAc
O
OAc
N
Me
Me
22i
δ (ppm)

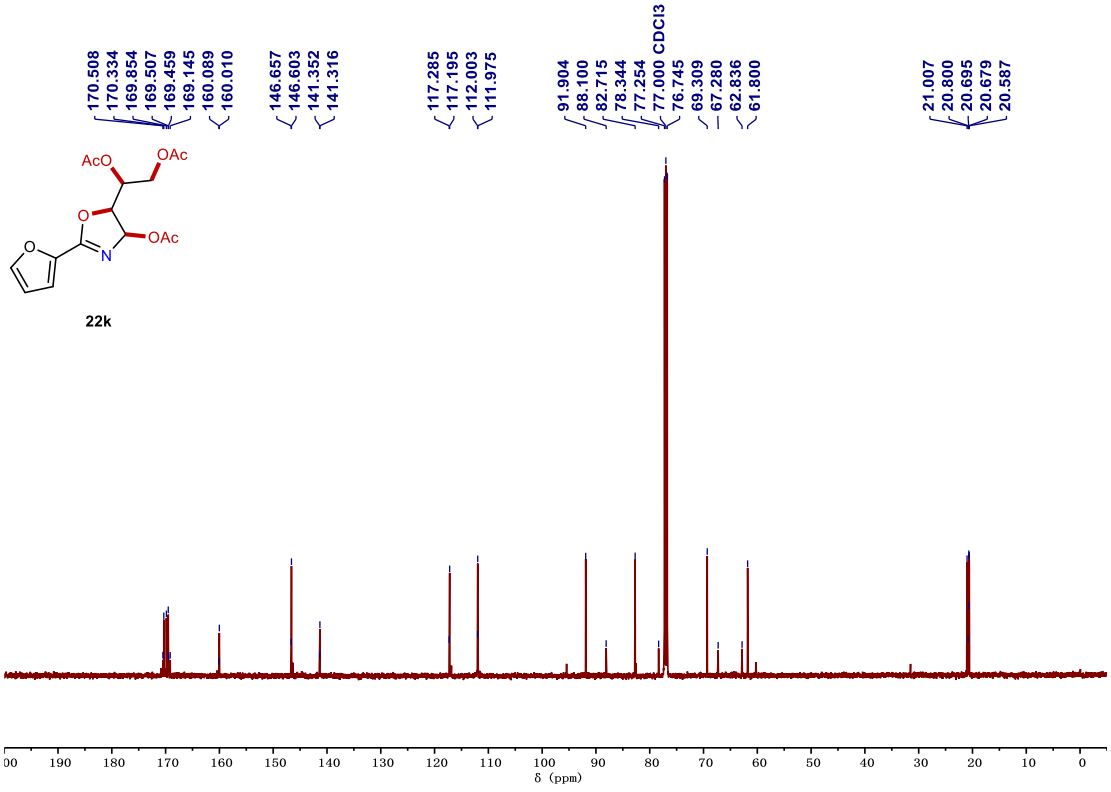

7.861
7.846
7.831
7.826
7.815
7.810
7.470
7.453
7.440
7.426
7.416
7.336
7.321
7.306
7.302
7.260 CDCl3
6.911
6.905
6.896
6.891
6.734
6.727
5.472
5.461
5.448
5.393
5.384
5.375
5.366
5.357
4.814
4.799
4.794
4.784
4.708
4.699
4.691
4.682
4.674
4.508
4.501
4.494
4.484
4.477
4.469
4.319
4.309
4.280
4.268
4.260
4.251
4.243
4.234
4.226
4.217
2.135
2.075
AcO
OAc
Cl
O
N
OAc
1.06
2.00
1.09
0.20
0.77
0.26
0.74
0.26
0.80
0.70
1.35
3.03
6.08
δ (ppm)

22j

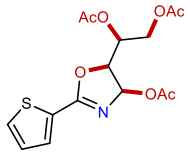

170.493
170.347
169.918
169.538
169.266
169.232
167.185
133.828
133.784
132.763
132.697
131.849
131.019
130.942
126.668
125.727
91.999
88.307
82.643
78.178
77.254
77.000 CDCl3
76.746
69.371
67.271
63.005
61.961
21.047
20.860
20.826
20.736
20.683
20.607
AcO
OAc
Cl
O
N
OAc
δ (ppm)

22j

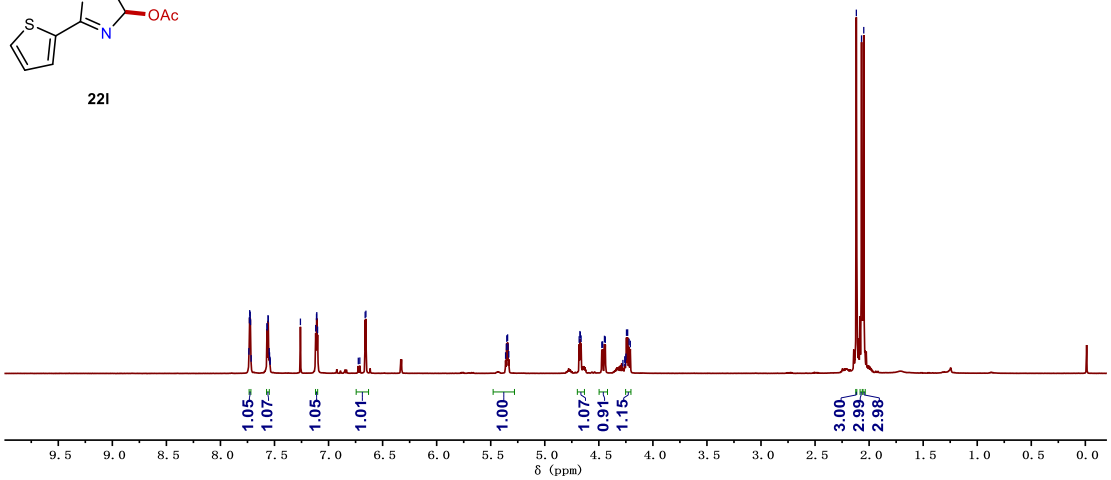

7.599
7.260 CDCl3
7.128
7.121
6.871
6.858
6.700
6.692
6.534
6.531
6.528
6.524
6.521
5.435
5.430
5.424
5.418
5.412
5.407
5.402
5.340
5.330
5.321
5.311
4.756
4.743
4.739
4.724
4.673
4.667
4.663
4.655
4.642
4.637
4.465
4.458
4.441
4.433
4.225
4.215
4.201
4.190
2.106
2.087
2.064
2.053
AcO
OAc
O
OAc
N
O
22k
1.06
1.03
0.21
0.78
1.08
0.21
0.76
0.21
0.83
0.72
1.24
2.22
0.77
2.16
3.81
δ (ppm)

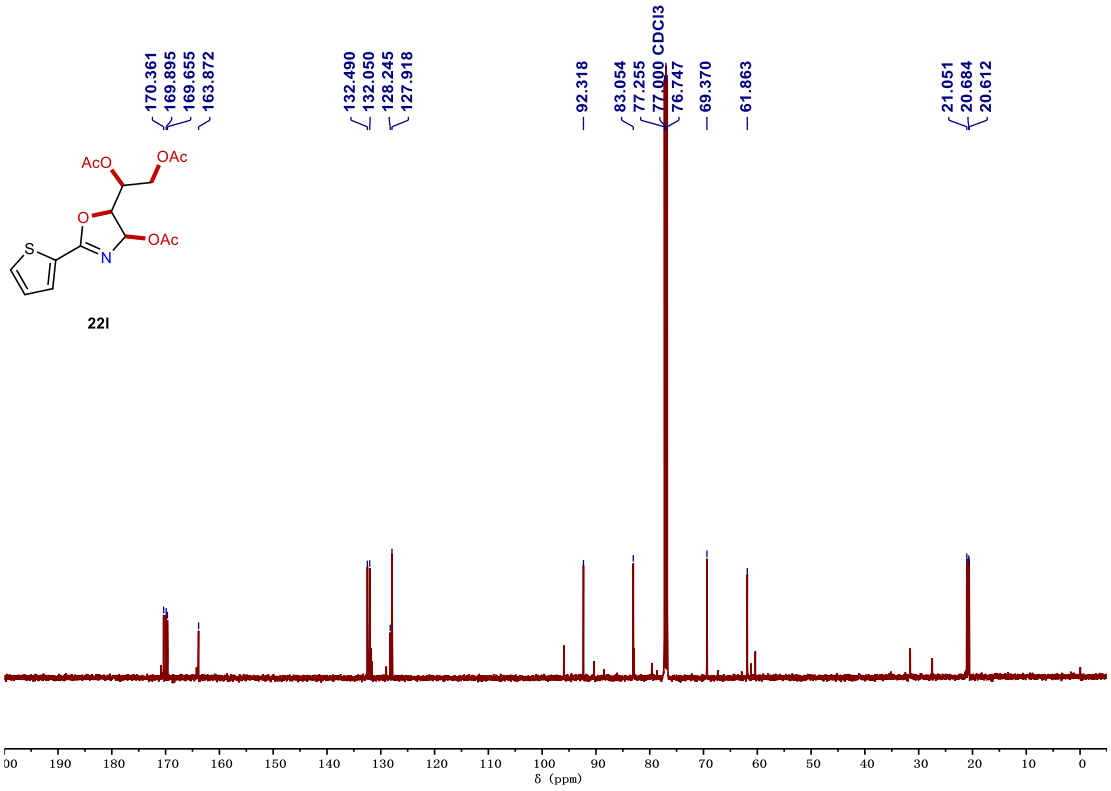

170.508
170.334
169.854
169.507
169.459
169.145
160.089
160.010
146.657
146.603
141.352
141.316
117.285
117.195
112.003
111.975
91.904
88.100
82.715
78.344
77.254
77.000 CDCl3
76.745
69.309
67.280
62.836
61.800
21.007
20.800
20.695
20.679
20.587
AcO
OAc
O
OAc
N
O
22k
δ (ppm)


7.737
7.732
7.729
7.724
7.722
7.716
7.570
7.567
7.560
7.557
7.549
7.546
7.260 CDCl3
7.119
7.112
7.109
6.711
6.662
6.654
5.363
5.355
5.352
5.344
5.341
5.333
4.682
4.675
4.671
4.664
4.473
4.465
4.448
4.440
4.277
4.260
4.256
4.250
4.244
4.233
4.220
4.209
2.118
2.070
2.050


AcO
OAc
O
OAc
S
N

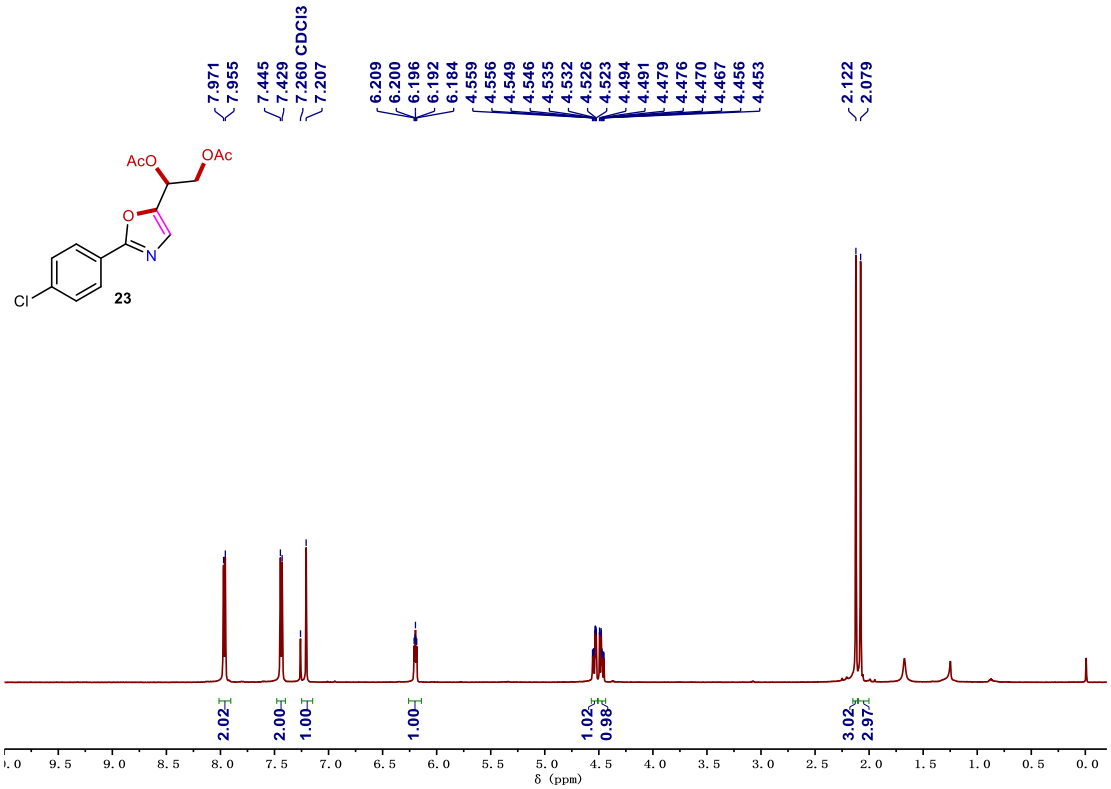
22l

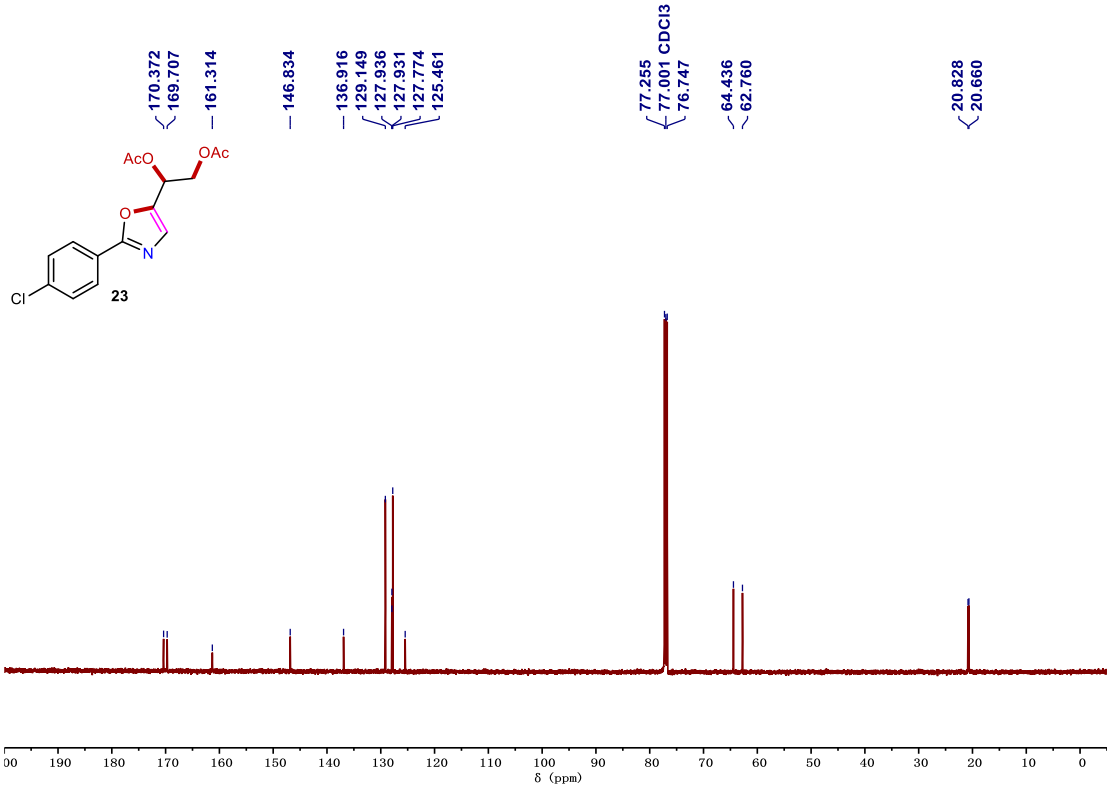

1.05
1.07
1.05
1.01
1.00
1.07
0.91
1.15
3.00
2.99
2.98
δ (ppm)

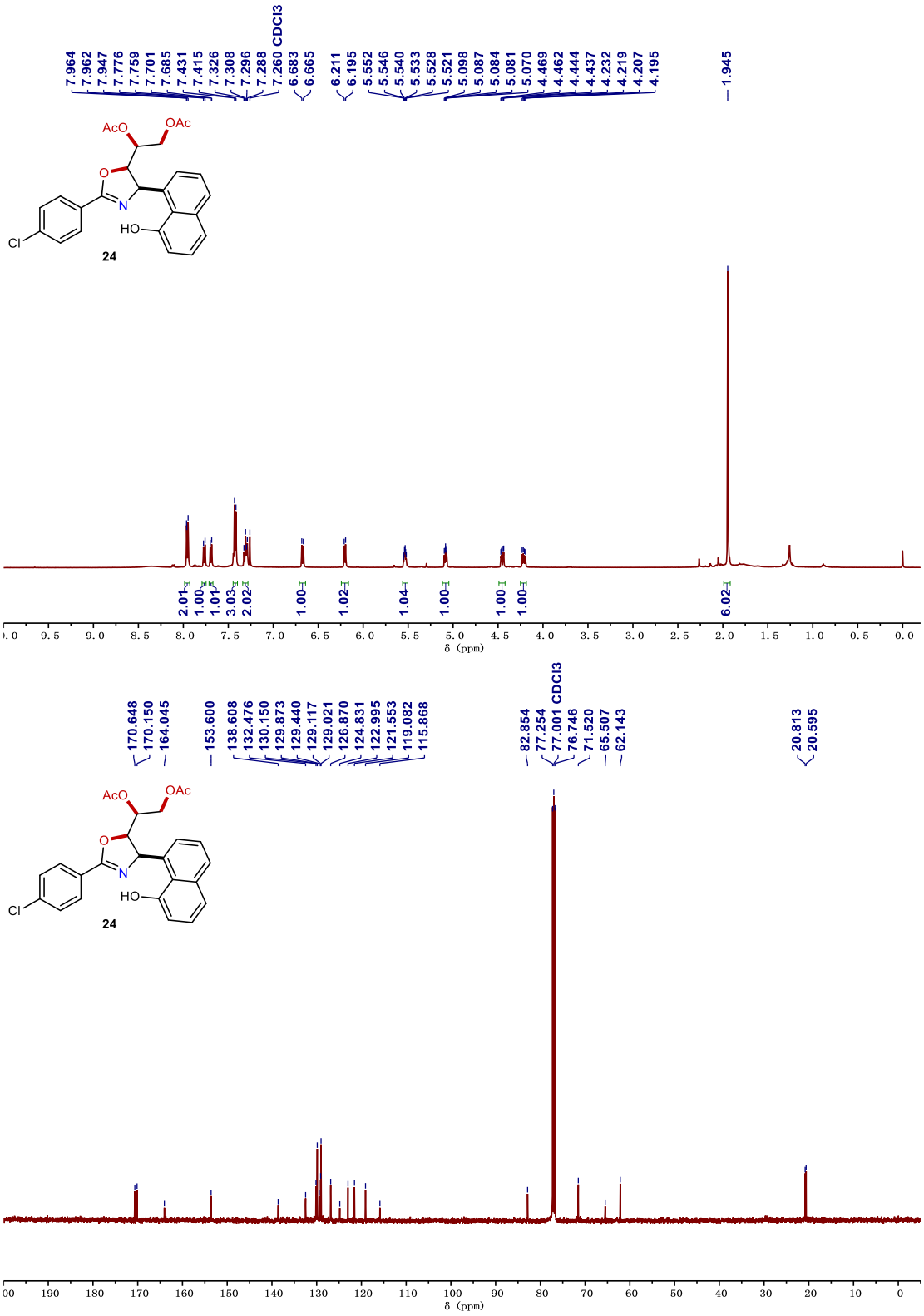

170.361
169.895
169.655
163.872
132.490
132.050
128.245
127.918
92.318
83.054
77.255
77.000 CDCl3
76.747
69.370
61.863
21.051
20.684
20.612
δ (ppm)

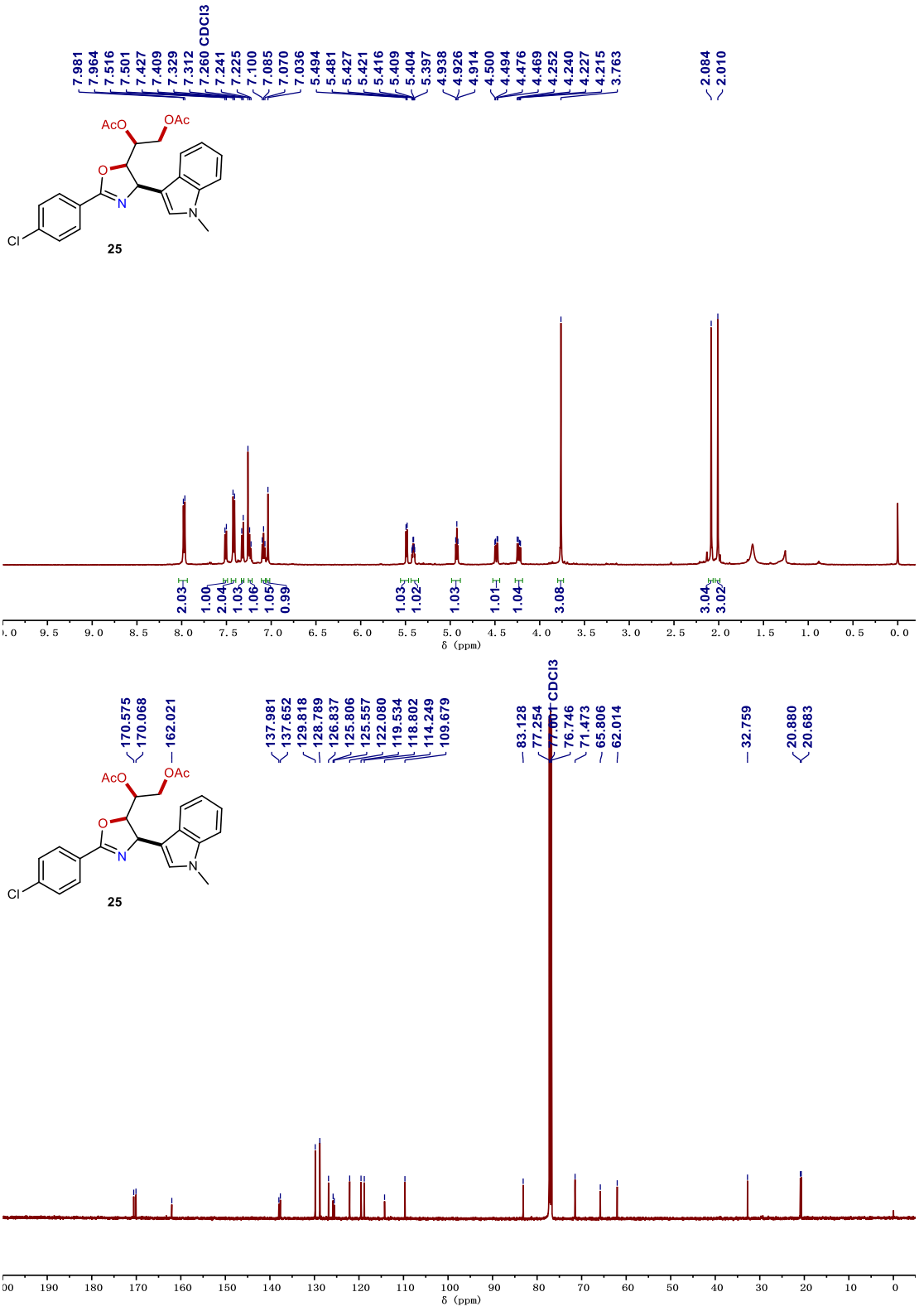
AcO
OAc
O
OAc
S
N

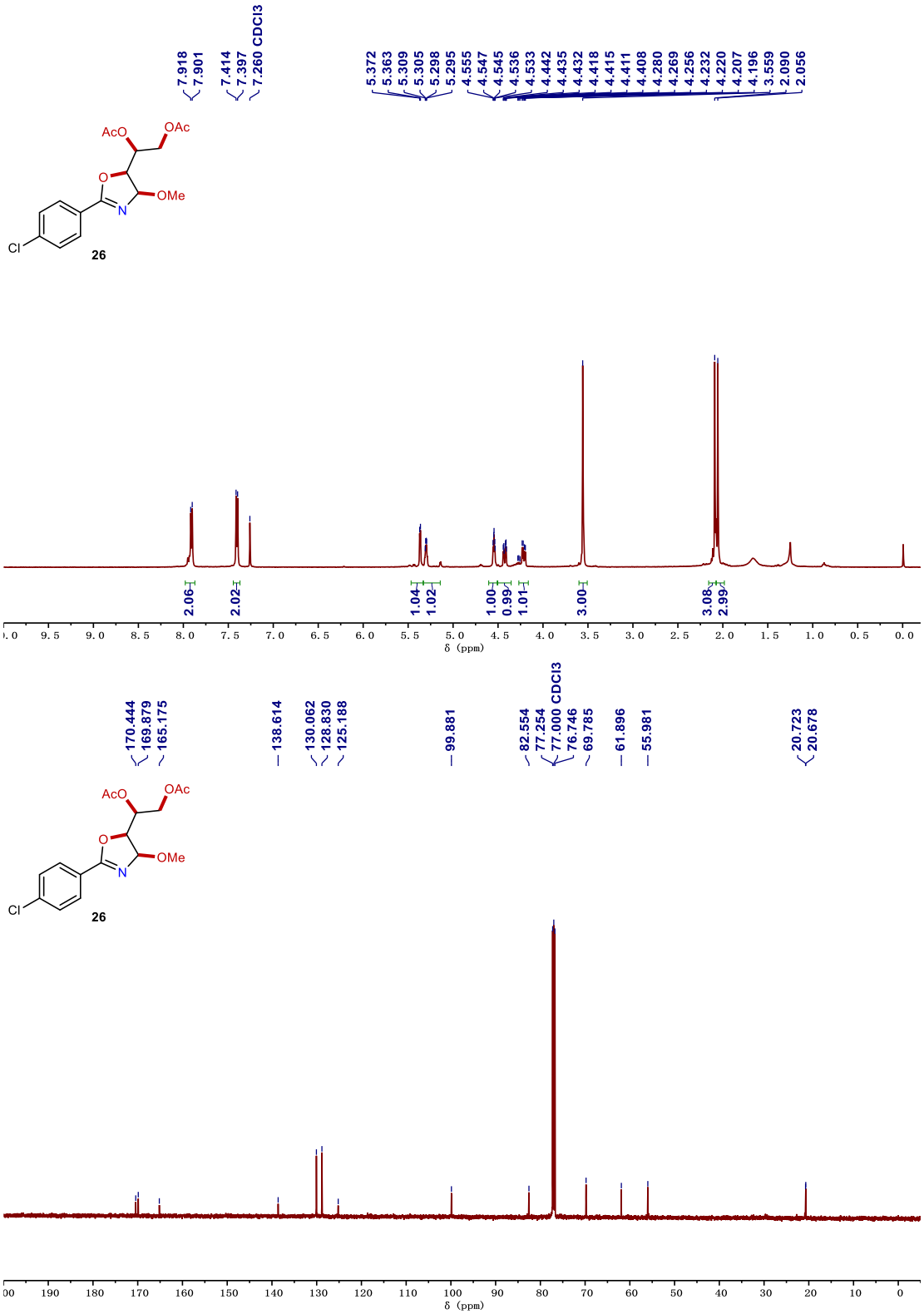
22l

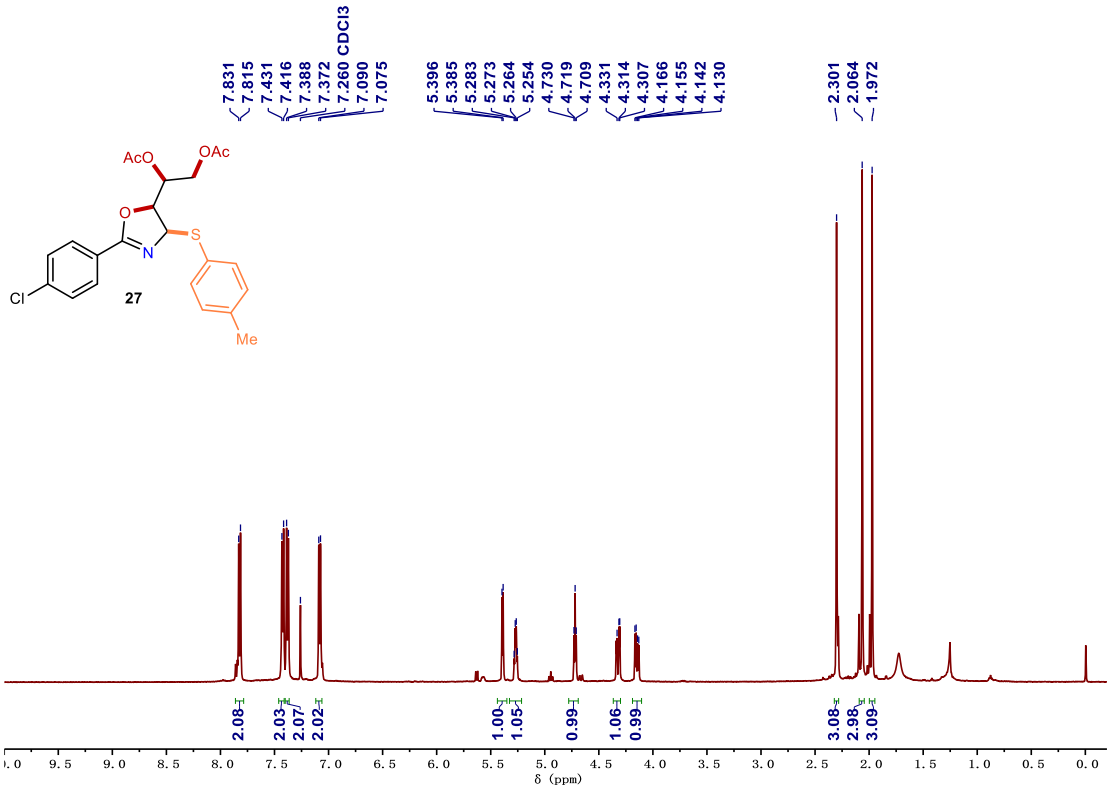

7.971
7.955
7.445
7.429
7.260 CDCl3
7.207
6.209
6.200
6.196
6.192
6.184
4.559
4.556
4.549
4.546
4.535
4.532
4.526
4.523
4.494
4.491
4.479
4.476
4.470
4.467
4.456
4.453
2.122
2.079
AcO
OAc
O
N
Cl
23
2.02
2.00
1.00
1.00
1.02
0.98
3.02
2.97
δ (ppm)

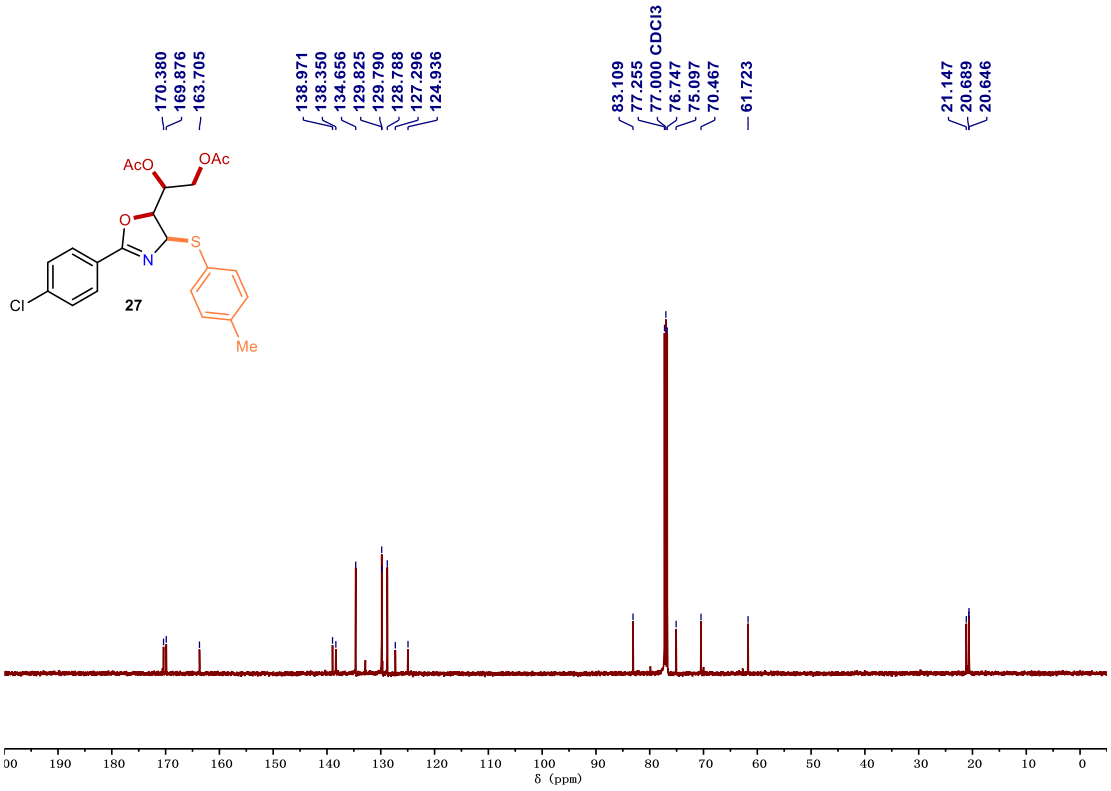

170.372
169.707
161.314
146.834
136.916
129.149
127.936
127.931
127.774
125.461
77.255
77.001 CDCl3
76.747
64.436
62.760
20.828
20.660
AcO
OAc
O
N
Cl
23
δ (ppm)

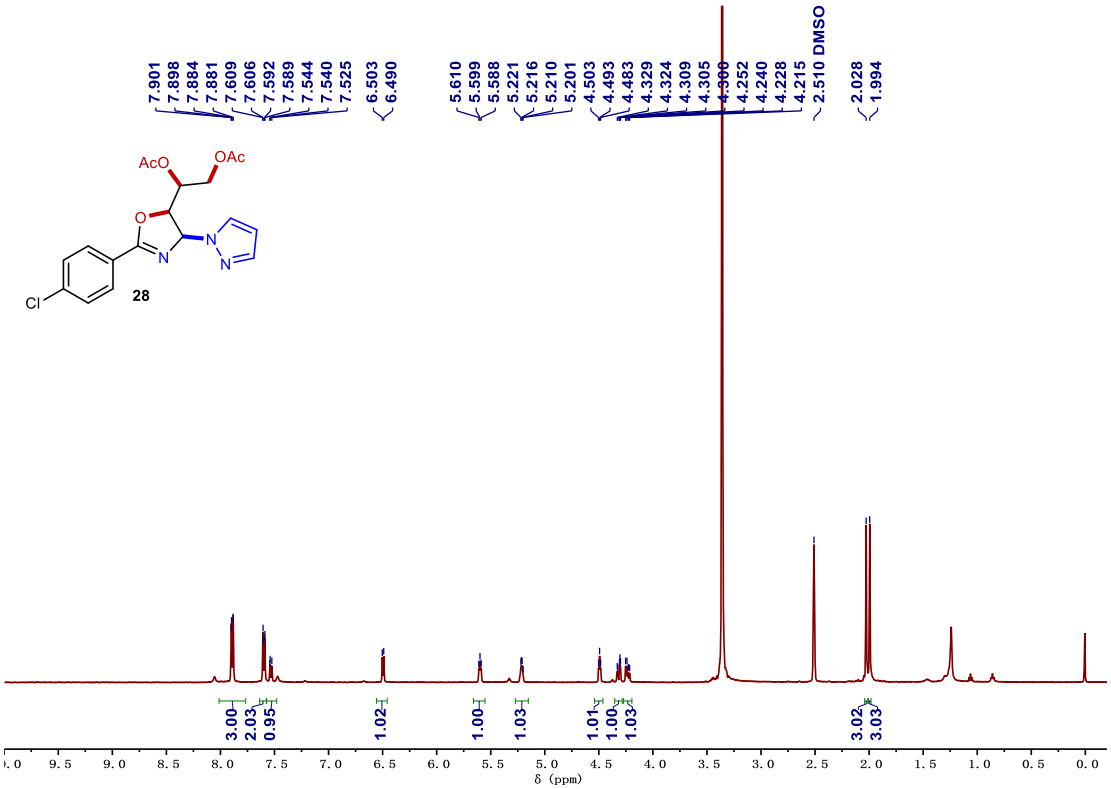

7.964
7.962
7.947
7.776
7.759
7.701
7.685
7.431
7.415
7.326
7.308
7.296
7.288
7.260 CDCl3
6.683
6.665
6.211
6.195
5.552
5.546
5.540
5.533
5.528
5.521
5.098
5.087
5.084
5.081
5.070
4.469
4.462
4.444
4.437
4.232
4.219
4.207
4.195
1.945
AcO
OAc
O
N
HO
Cl
24
2.01
1.00
1.01
3.03
2.02
1.00
1.02
1.04
1.00
1.00
1.00
6.02
δ (ppm)
170.648
170.150
164.045
153.600
138.608
132.476
130.150
129.873
129.440
129.117
129.021
126.870
124.831
122.995
121.553
119.082
115.868
82.854
77.254
77.001 CDCl3
76.746
71.520
65.507
62.143
20.813
20.595
δ (ppm)

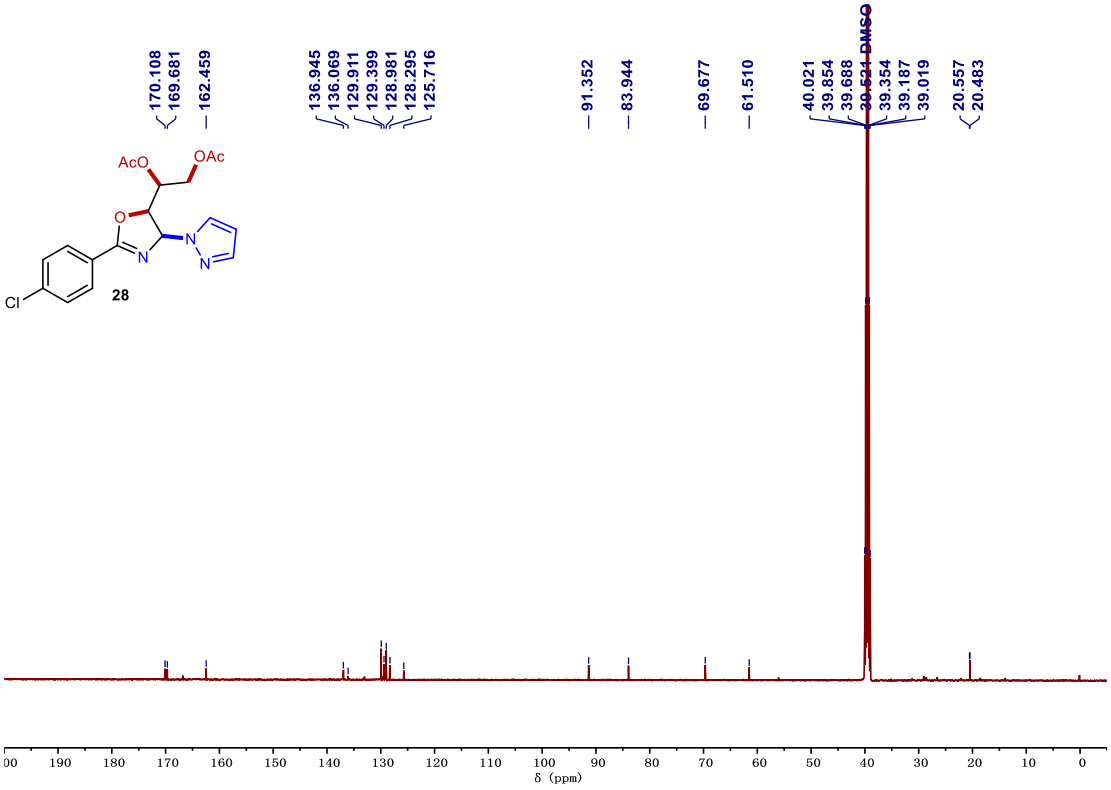

7.981
7.964
7.516
7.501
7.427
7.409
7.329
7.312
7.260 CDCl3
7.241
7.225
7.100
7.085
7.070
7.036
5.494
5.481
5.427
5.421
5.416
5.409
5.404
5.397
4.938
4.926
4.914
4.500
4.494
4.476
4.469
4.252
4.240
4.227
4.215
3.763
2.084
2.010
AcO
OAc
O
N
N
Cl
25
2.03
1.00
2.04
1.03
1.06
1.05
0.99
1.03
1.02
1.03
1.01
1.04
3.08
3.04
3.02
δ (ppm)
170.575
170.068
162.021
137.981
137.652
129.818
128.789
126.837
125.806
125.557
122.080
119.534
118.802
114.249
109.679
83.128
77.254
77.001 CDCl3
76.746
71.473
65.806
62.014
32.759
20.880
20.683
δ (ppm)

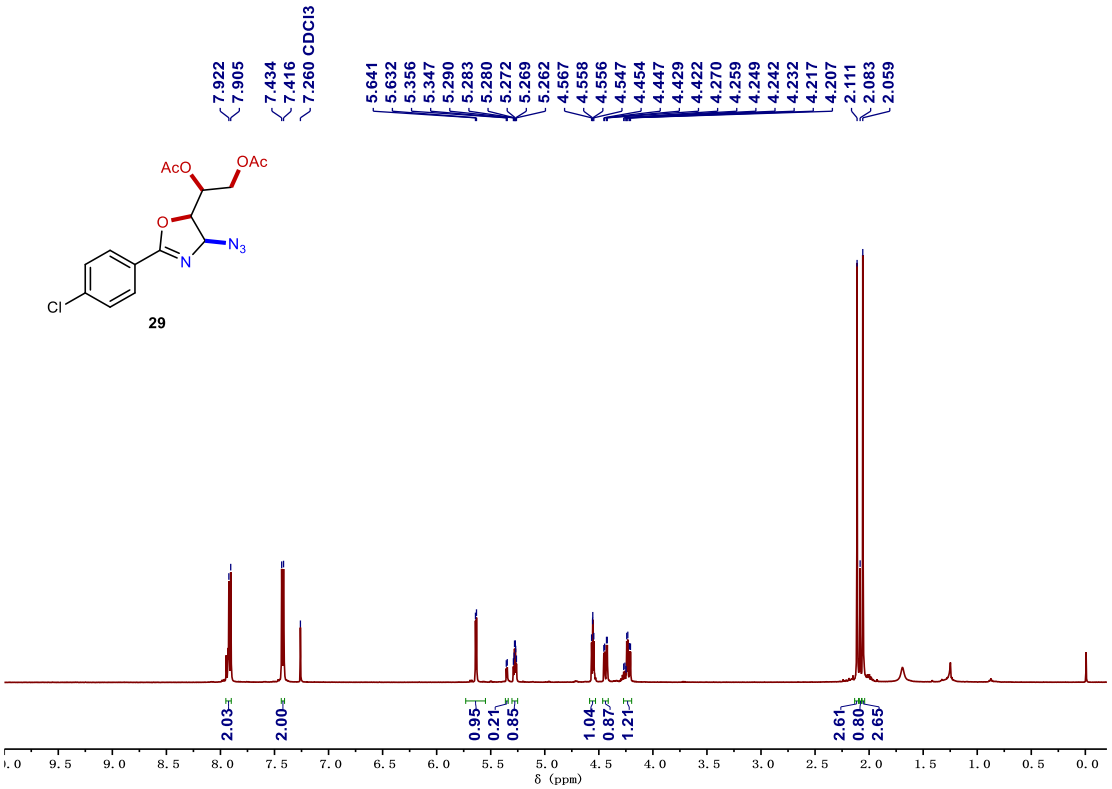

7.918
7.901
7.414
7.397
7.260 CDCl3
5.372
5.363
5.309
5.305
5.298
5.295
4.555
4.547
4.545
4.536
4.533
4.442
4.435
4.432
4.418
4.415
4.411
4.408
4.280
4.269
4.256
4.232
4.220
4.207
4.196
3.559
2.090
2.056
AcO
OAc
O
N
OMe
Cl
26
2.06
2.02
1.04
1.02
1.00
0.99
1.01
3.00
3.08
2.99
δ (ppm)
170.444
169.879
165.175
138.614
130.062
128.830
125.188
99.881
82.554
77.254
77.000 CDCl3
76.746
69.785
61.896
55.981
20.723
20.678
AcO
OAc
O
N
OMe
Cl
26
δ (ppm)

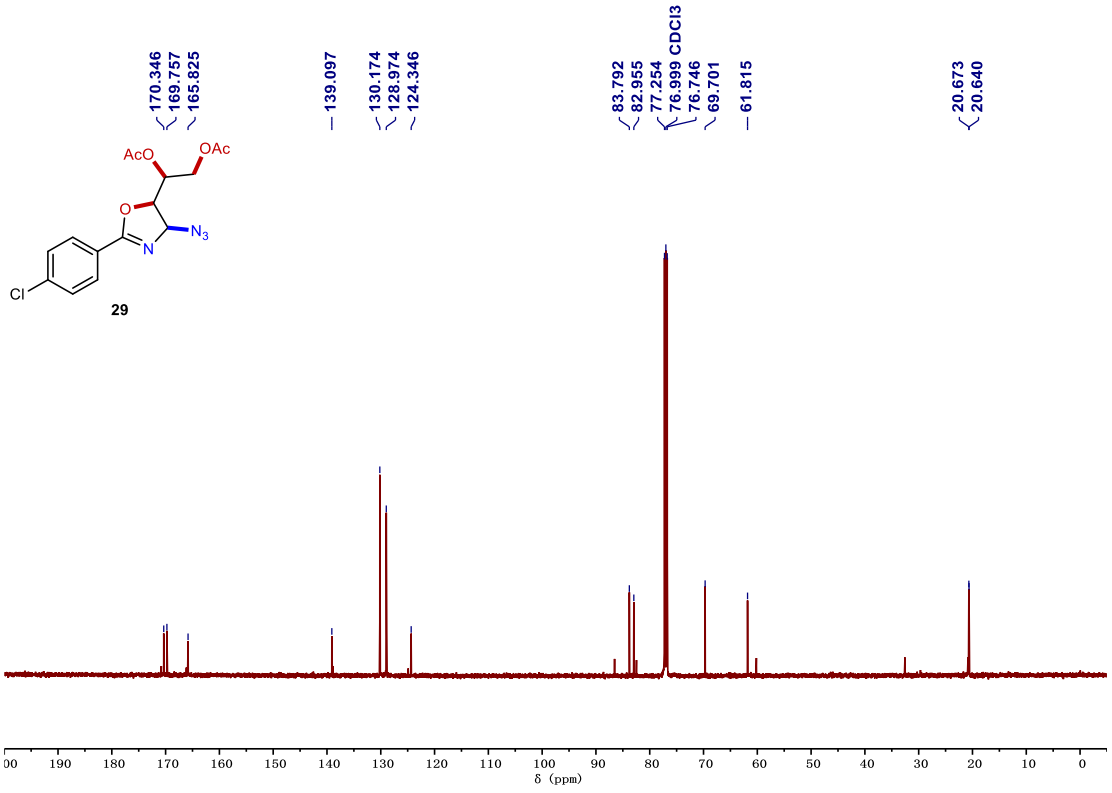

7.831
7.815
7.431
7.416
7.388
7.372
7.260 CDCl3
7.090
7.075
5.396
5.385
5.283
5.273
5.264
5.254
4.730
4.719
4.709
4.331
4.314
4.307
4.166
4.155
4.142
4.130
2.301
2.064
1.972
AcO
OAc
O
N
S
Cl
Me
27
2.08
2.03
2.07
2.02
1.00
1.05
0.99
1.06
0.99
3.08
2.98
3.09
δ (ppm)

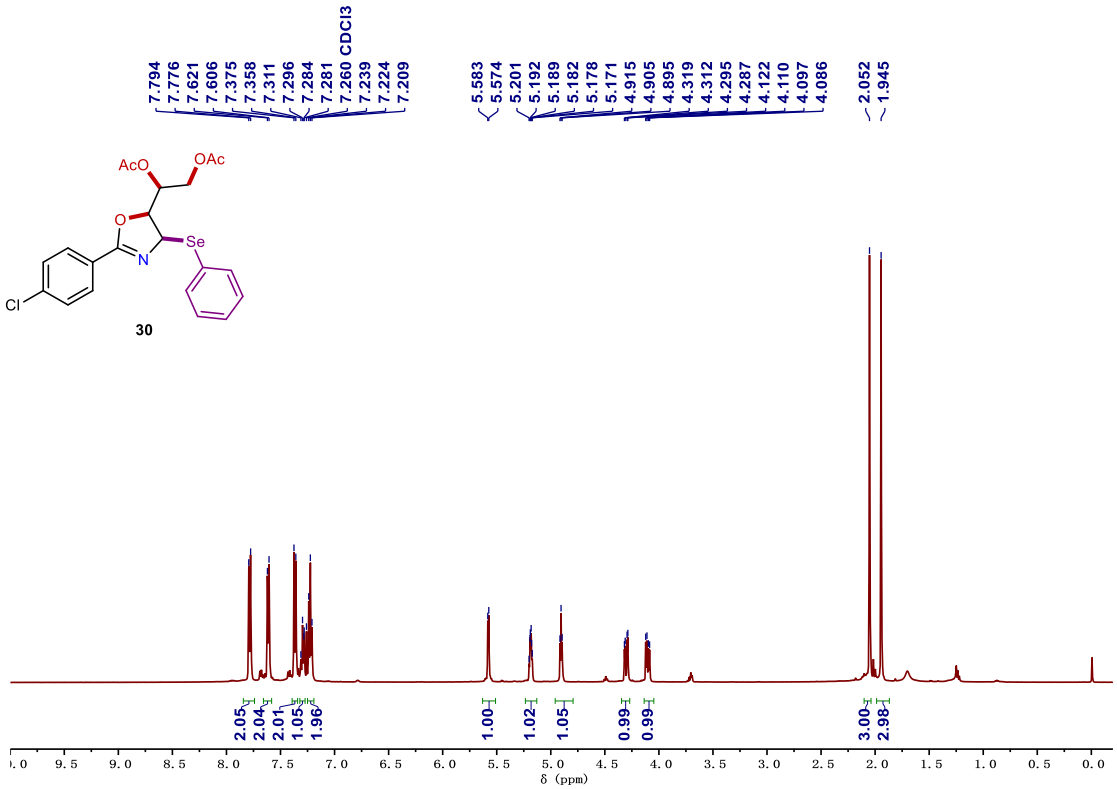

170.380
169.876
163.705
138.971
138.350
134.656
129.825
129.790
128.788
127.296
124.936
83.109
77.255
77.000 CDCl3
76.747
75.097
70.467
61.723
21.147
20.689
20.646
AcO
OAc
O
N
S
Cl
Me
27
δ (ppm)

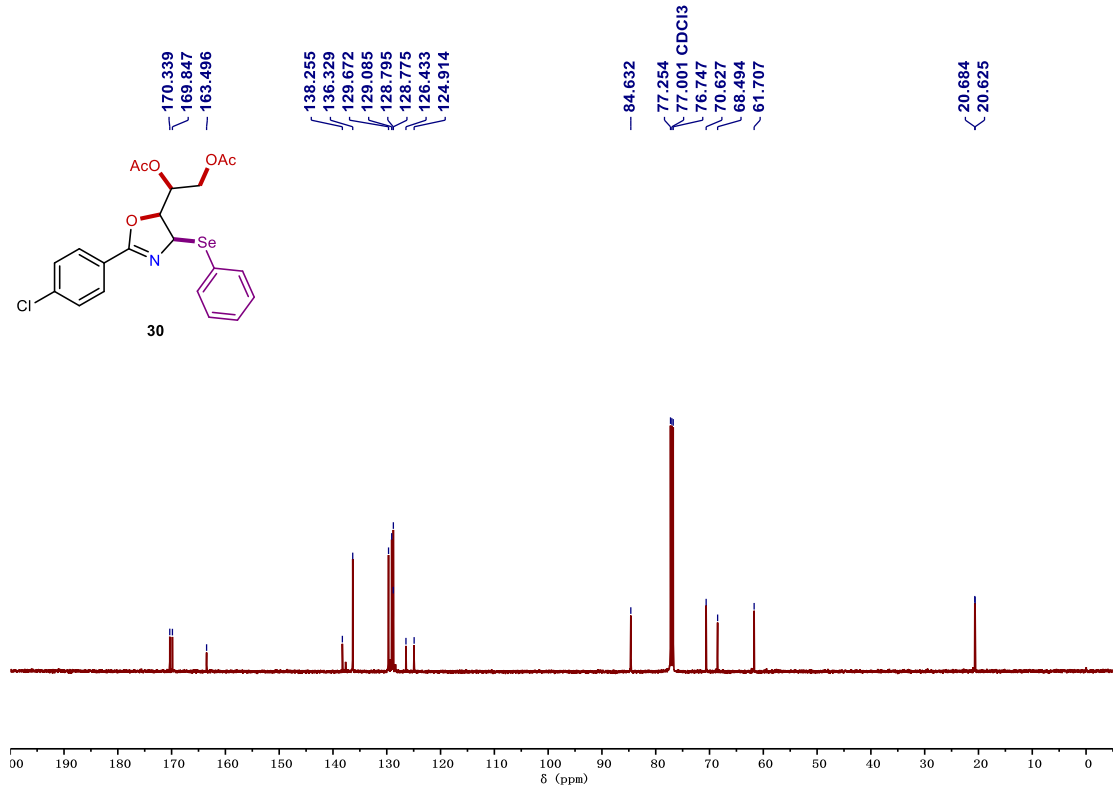

7.901
7.898
7.884
7.881
7.609
7.606
7.592
7.589
7.544
7.540
7.525
6.503
6.490
5.610
5.599
5.588
5.221
5.216
5.210
5.201
4.503
4.493
4.483
4.329
4.324
4.309
4.305
4.300
4.252
4.240
4.228
4.215
2.510 DMSO
2.028
1.994
AcO
OAc
O
N
N
Cl
28
3.00
2.03
0.95
1.02
1.00
1.03
1.01
1.00
1.03
3.02
3.03
δ (ppm)

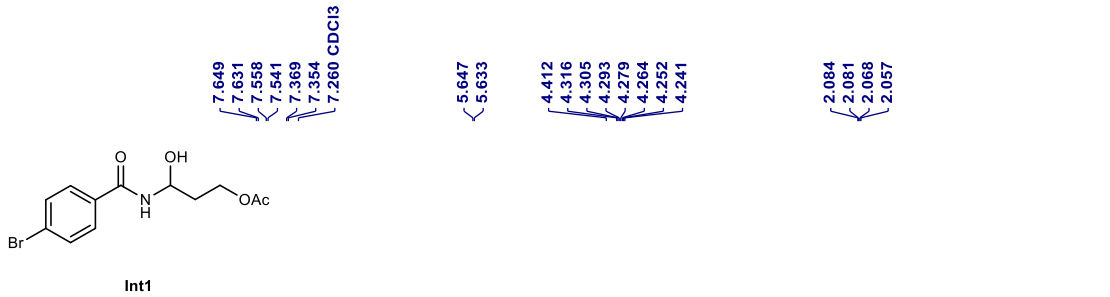

170.108
169.681
162.459
136.945
136.069
129.911
129.399
128.981
128.295
125.716
91.352
83.944
69.677
61.510
40.021
39.854
39.688
39.521 DMSO
39.354
39.187
39.019
20.557
20.483
AcO
OAc
O
N
N
Cl
28
δ (ppm)


7.922
7.905
7.434
7.416
7.260 CDCl3
5.641
5.632
5.356
5.347
5.290
5.283
5.280
5.272
5.269
5.262
4.567
4.558
4.556
4.547
4.454
4.447
4.429
4.422
4.270
4.259
4.249
4.242
4.232
4.217
4.207
2.111
2.083
2.059
AcO
OAc
O
N
N3
Cl
29
2.03
2.00
0.95
0.21
0.85
1.04
0.87
1.21
2.61
0.80
2.65
δ (ppm)

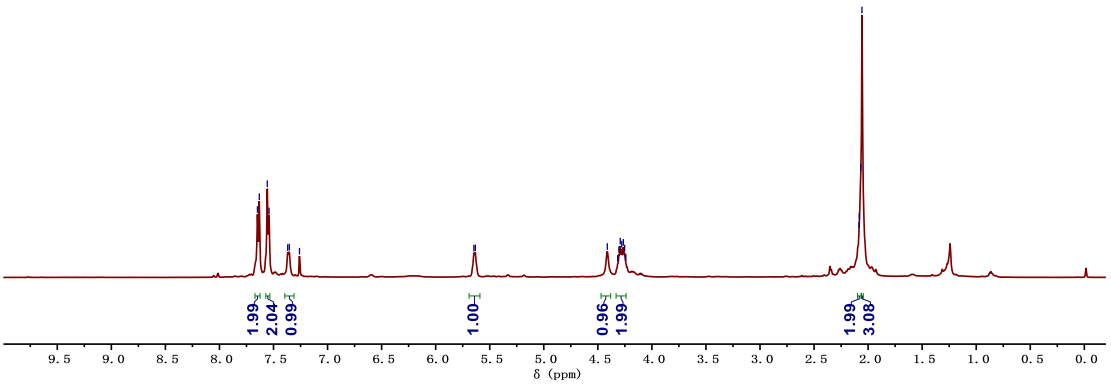

170.346
169.757
165.825
139.097
130.174
128.974
124.346
83.792
82.955
77.254
76.999 CDCl3
76.746
69.701
61.815
20.673
20.640
AcO
OAc
O
N
N3
Cl
29
δ (ppm)

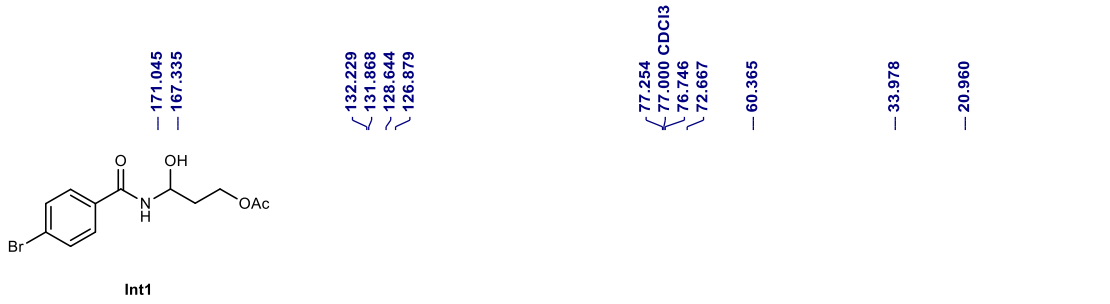

7.794
7.776
7.621
7.606
7.375
7.358
7.311
7.296
7.284
7.281
7.260 CDCl3
7.239
7.224
7.209
5.583
5.574
5.201
5.192
5.189
5.182
5.178
5.171
4.915
4.905
4.895
4.319
4.312
4.295
4.287
4.122
4.110
4.097
4.086
2.052
1.945
AcO
OAc
O
N
Se
Cl
30
2.05
2.04
2.01
1.05
1.96
1.00
1.02
1.05
0.99
0.99
3.00
2.98
δ (ppm)


170.339
169.847
163.496
138.255
136.329
129.672
129.085
128.795
128.775
126.433
124.914
84.632
77.254
77.001 CDCl3
76.747
70.627
68.494
61.707
20.684
20.625
AcO
OAc
O
N
Se
Cl
30
δ (ppm)

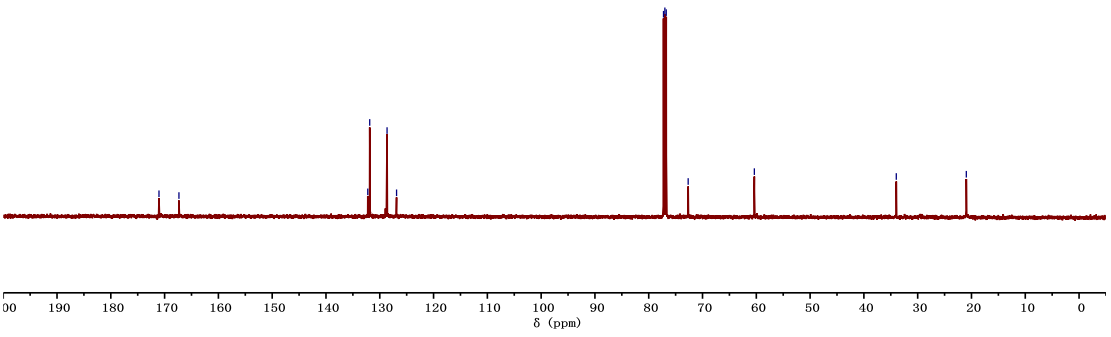

7.649
7.631
7.558
7.541
7.369
7.354
7.260 CDCl3
5.647
5.633
4.412
4.316
4.305
4.293
4.279
4.264
4.252
4.241
2.084
2.081
2.068
2.057
O
OH
N
H
OAc
Br

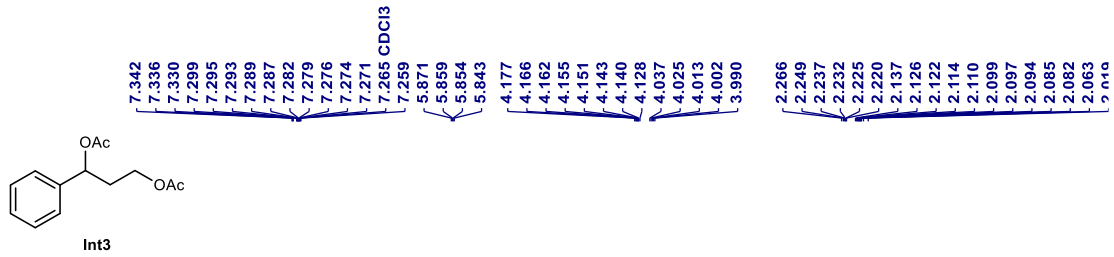
Int1


1.99
2.04
0.99
1.00
0.96
1.99
1.99
3.08
δ (ppm)

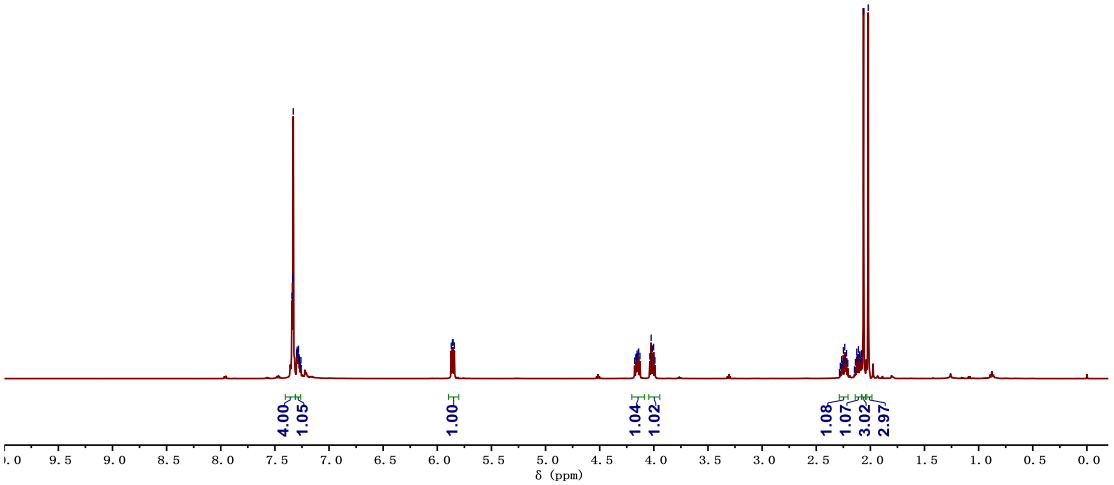

171.045
167.335
132.229
131.868
128.644
126.879
77.254
77.000 CDCl3
76.746
72.667
60.365
33.978
20.960
O
OH
N
H
OAc
Br

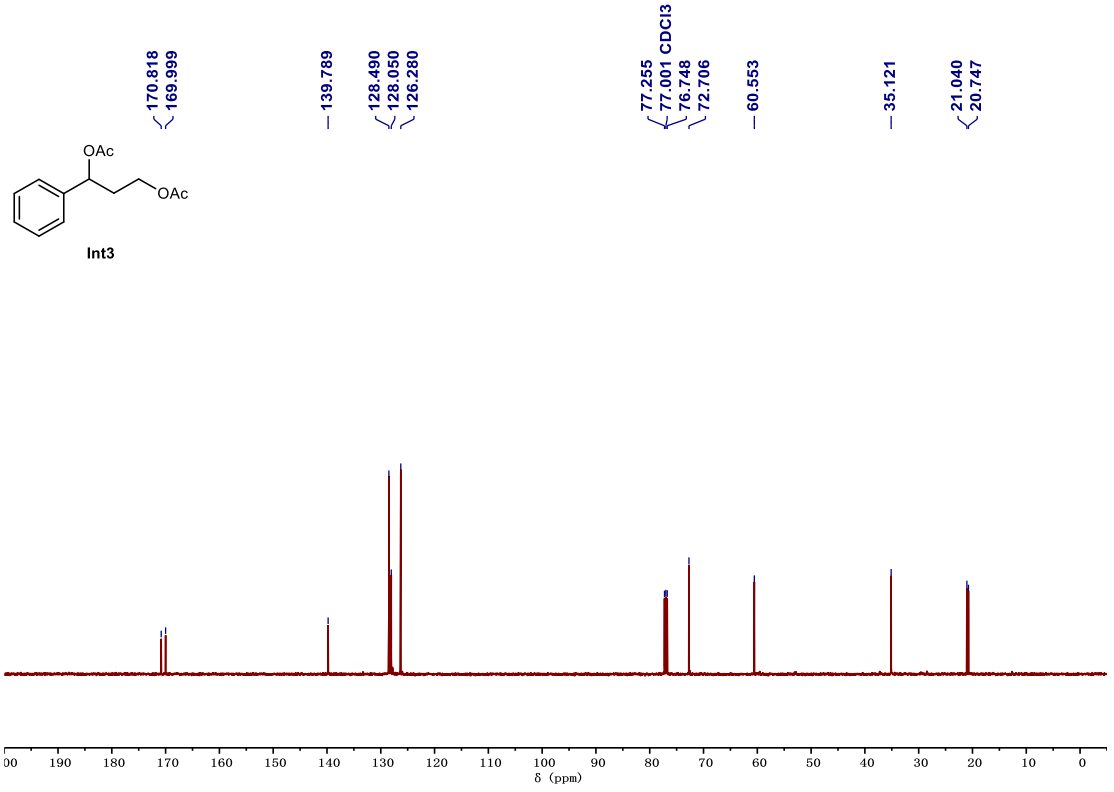
Int1


δ (ppm)

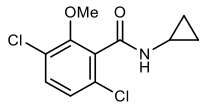

7.342
7.336
7.330
7.299
7.295
7.293
7.289
7.287
7.282
7.279
7.276
7.274
7.271
7.265 CDCl3
7.259
5.871
5.859
5.854
5.843
4.177
4.166
4.162
4.155
4.151
4.143
4.140
4.128
4.037
4.025
4.013
4.002
3.990
2.266
2.249
2.237
2.232
2.225
2.220
2.137
2.126
2.122
2.114
2.110
2.099
2.097
2.094
2.085
2.082
2.063
OAc
OAc

Int3


4.00
1.05
1.00
1.04
1.02
1.08
1.07
3.02
2.97
δ (ppm)

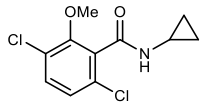

170.818
169.999
139.789
128.490
128.050
126.280
77.255
77.001 CDCl3
76.748
72.706
60.553
35.121
21.040
20.747
OAc
OAc
δ (ppm)

Int3


OMe
O
Cl
N
H
Cl

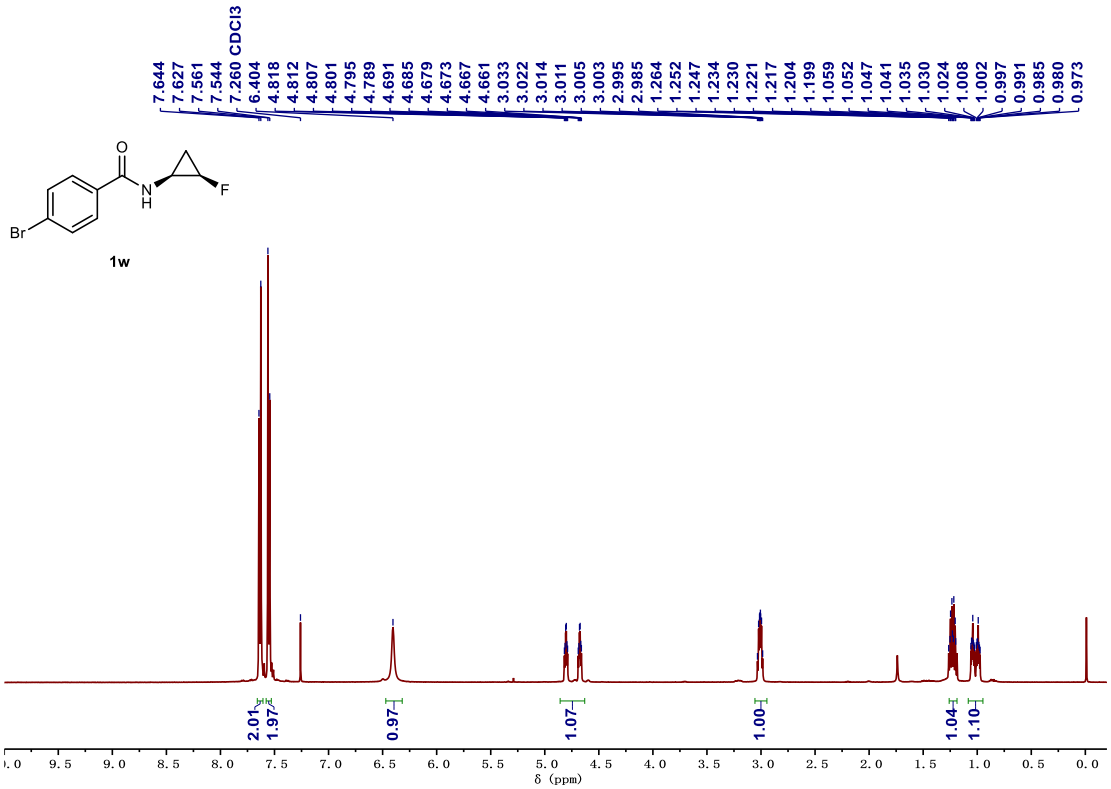
1t

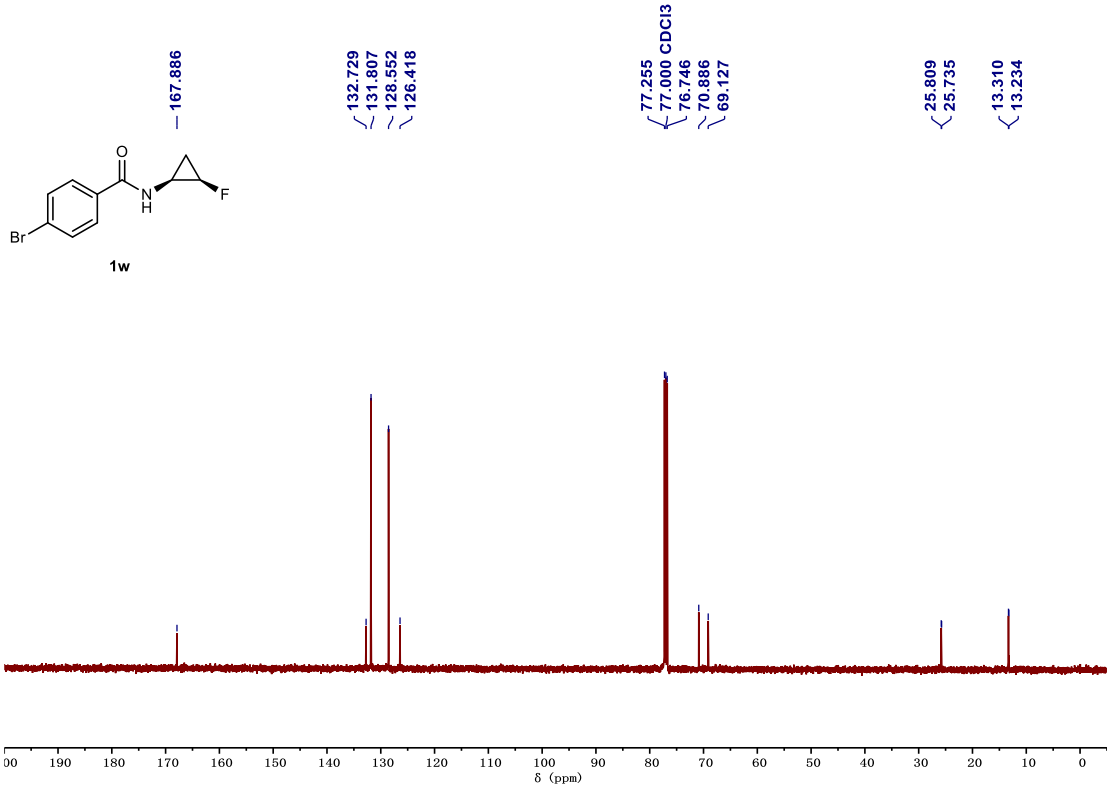
7.277
7.260 CDCl3
7.062
7.044
6.085
3.875
2.895
2.888
2.881
2.874
2.867
2.859
0.875
0.865
0.861
0.859
0.850
0.847
0.844
0.836
0.649
0.641
0.638
0.635
0.631
0.627
0.625
0.616
1.07
0.99
0.92
3.05
1.00
2.09
2.01
δ (ppm)

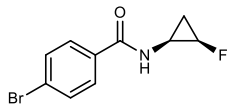

OMe
O
Cl
N
H
Cl

1t

165.342
153.609
132.861
131.251
129.982
126.749
125.956
77.255
77.001 CDCl3
76.746
62.571
22.855
6.660
δ (ppm)

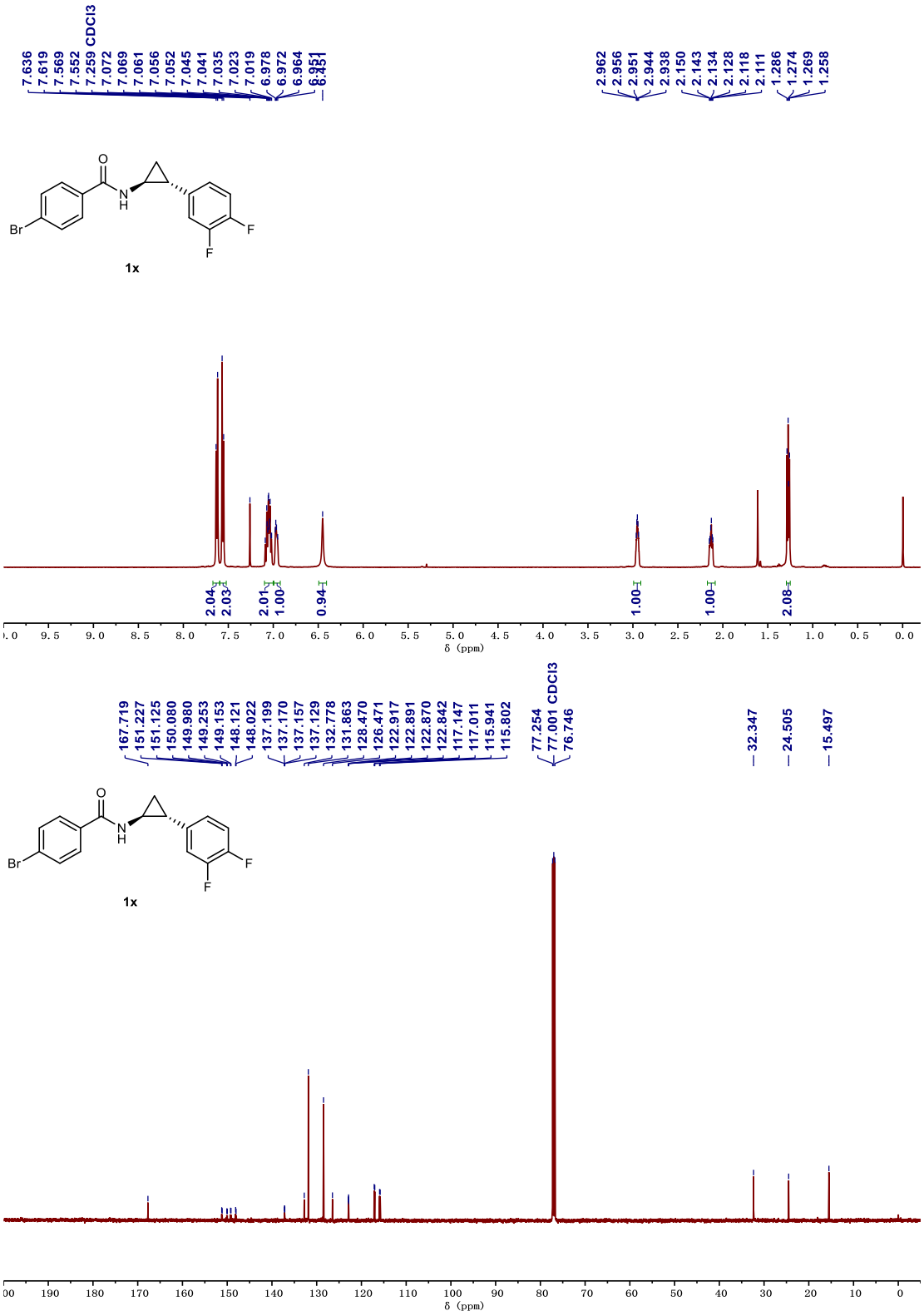

7.644
7.627
7.561
7.544
7.260 CDCl3
6.404
4.818
4.812
4.807
4.801
4.795
4.789
4.691
4.685
4.679
4.673
4.667
4.661
3.033
3.022
3.014
3.011
3.005
3.003
2.995
2.985
1.264
1.252
1.247
1.234
1.230
1.221
1.217
1.204
1.199
1.059
1.052
1.047
1.041
1.035
1.030
1.024
1.008
1.002
0.997
0.991
0.985
0.980
0.973
O
N
H
F
Br
1w
2.01
1.97
0.97
1.07
1.00
1.04
1.10
δ (ppm)

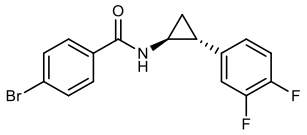

167.886
132.729
131.807
128.552
126.418
77.255
77.000 CDCl3
76.746
70.886
69.127
25.809
25.735
13.310
13.234
O
N
H
F
Br
1w
δ (ppm)


O
N
H
F
Br

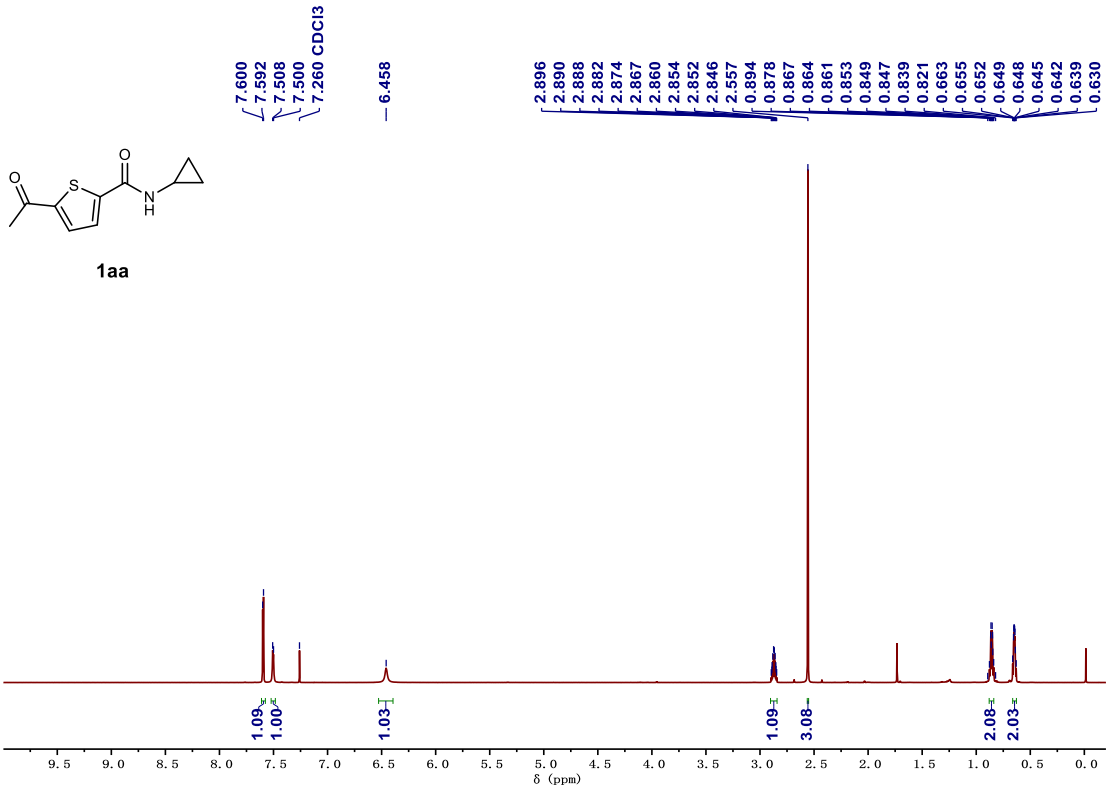
1w

-209.986
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)

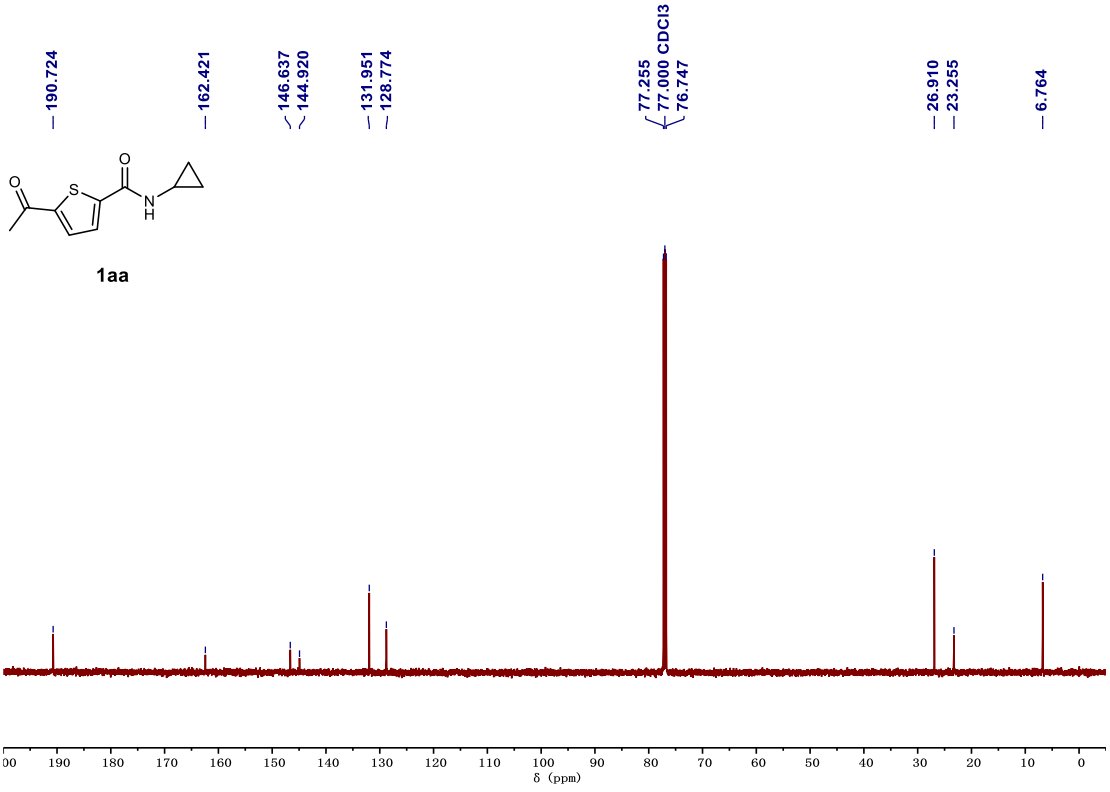

7.636
7.619
7.569
7.552
7.259 CDCl3
7.072
7.069
7.061
7.056
7.052
7.045
7.041
7.035
7.023
7.019
6.978
6.972
6.964
6.951
6.944
2.962
2.956
2.951
2.944
2.938
2.150
2.143
2.134
2.128
2.118
2.111
1.286
1.274
1.269
1.258
Br
F
F
1x
2.04
2.03
2.01
1.00
0.94
1.00
1.00
2.08
δ (ppm)
167.719
151.227
151.125
150.080
149.980
149.253
149.153
148.121
148.022
137.199
137.170
137.157
137.129
132.778
131.863
128.470
126.471
122.917
122.891
122.870
122.842
117.147
117.011
115.941
115.802
77.254
77.001 CDCl3
76.746
32.347
24.505
15.497
Br
F
F
1x
δ (ppm)



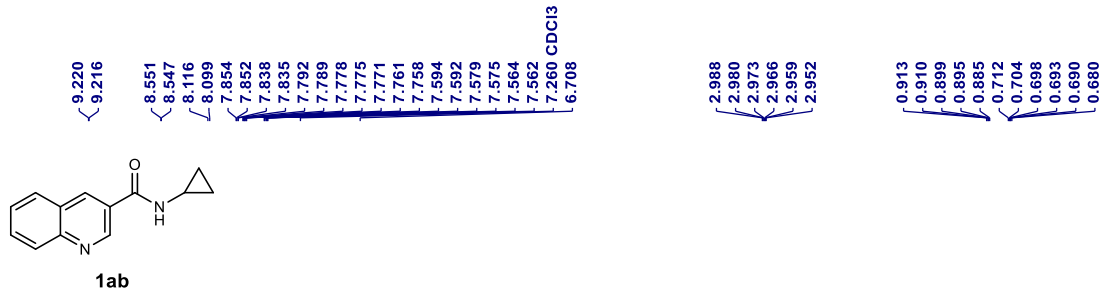
1x


7.600
7.592
7.508
7.500
7.260 CDCl3
6.458
2.896
2.890
2.888
2.882
2.874
2.867
2.860
2.854
2.852
2.846
2.557
0.894
0.878
0.867
0.864
0.861
0.853
0.849
0.847
0.839
0.821
0.663
0.655
0.652
0.649
0.648
0.645
0.642
0.639
0.630
1.09
1.00
1.03
1.09
3.08
2.08
2.03
δ (ppm)

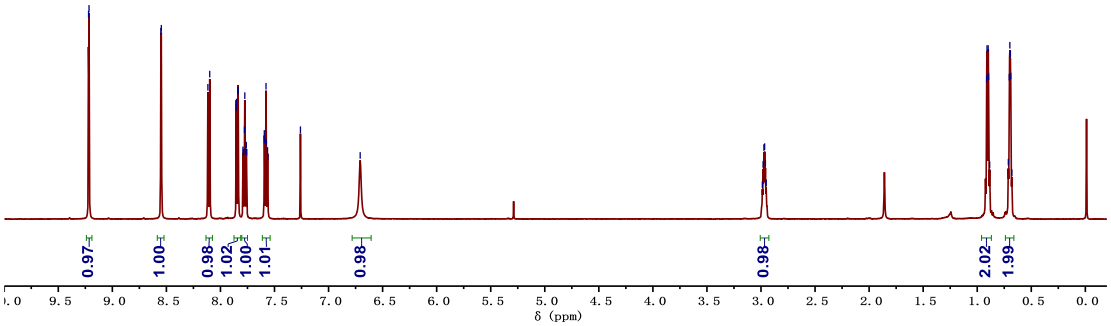
1aa

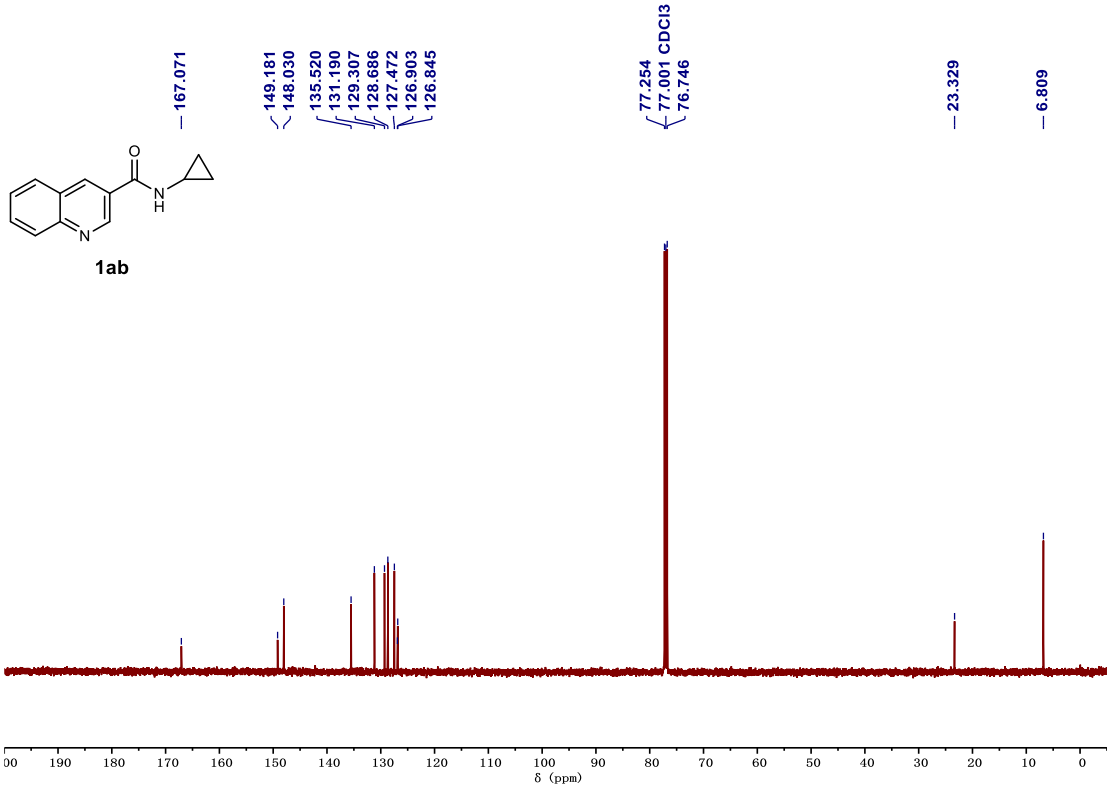

190.724
162.421
146.637
144.920
131.951
128.774
77.255
77.000 CDCl3
76.747
26.910
23.255
6.764
δ (ppm)

1aa

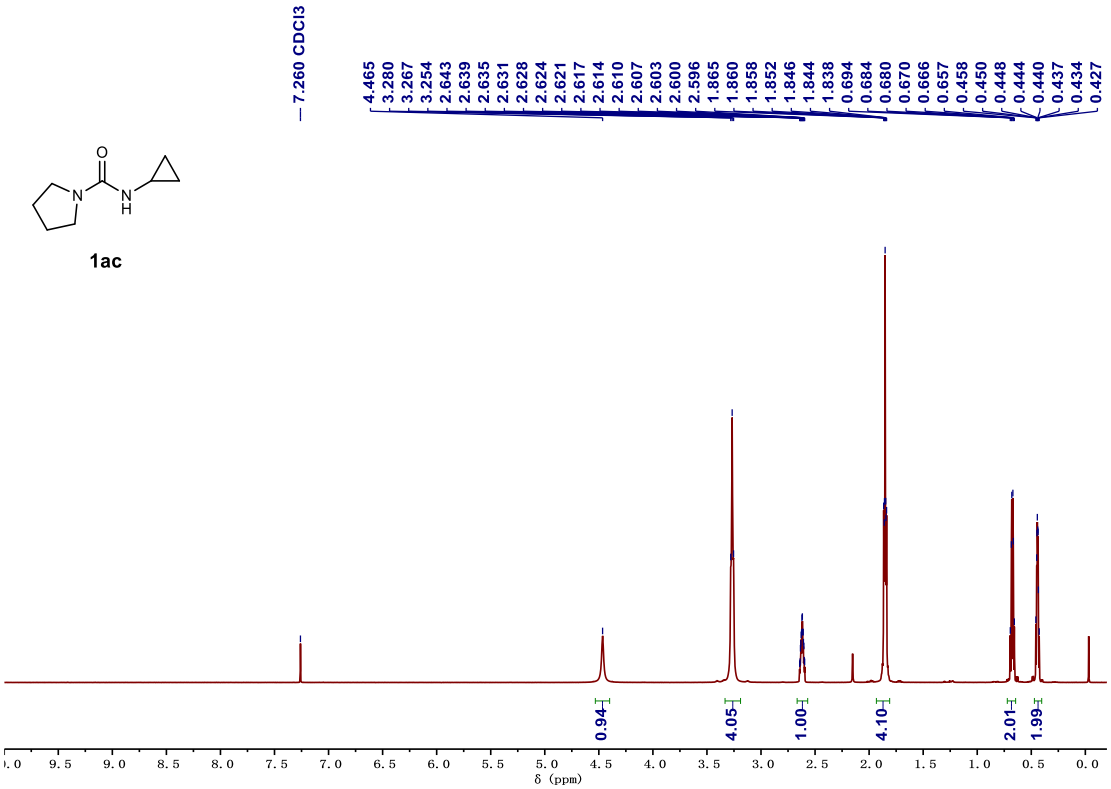

9.220
9.216
8.551
8.547
8.116
8.099
7.854
7.852
7.838
7.835
7.792
7.789
7.778
7.775
7.771
7.761
7.758
7.594
7.592
7.579
7.575
7.564
7.562
7.260 CDCl3
6.708
2.988
2.980
2.973
2.966
2.959
2.952
0.913
0.910
0.899
0.895
0.885
0.712
0.704
0.698
0.693
0.690
0.680

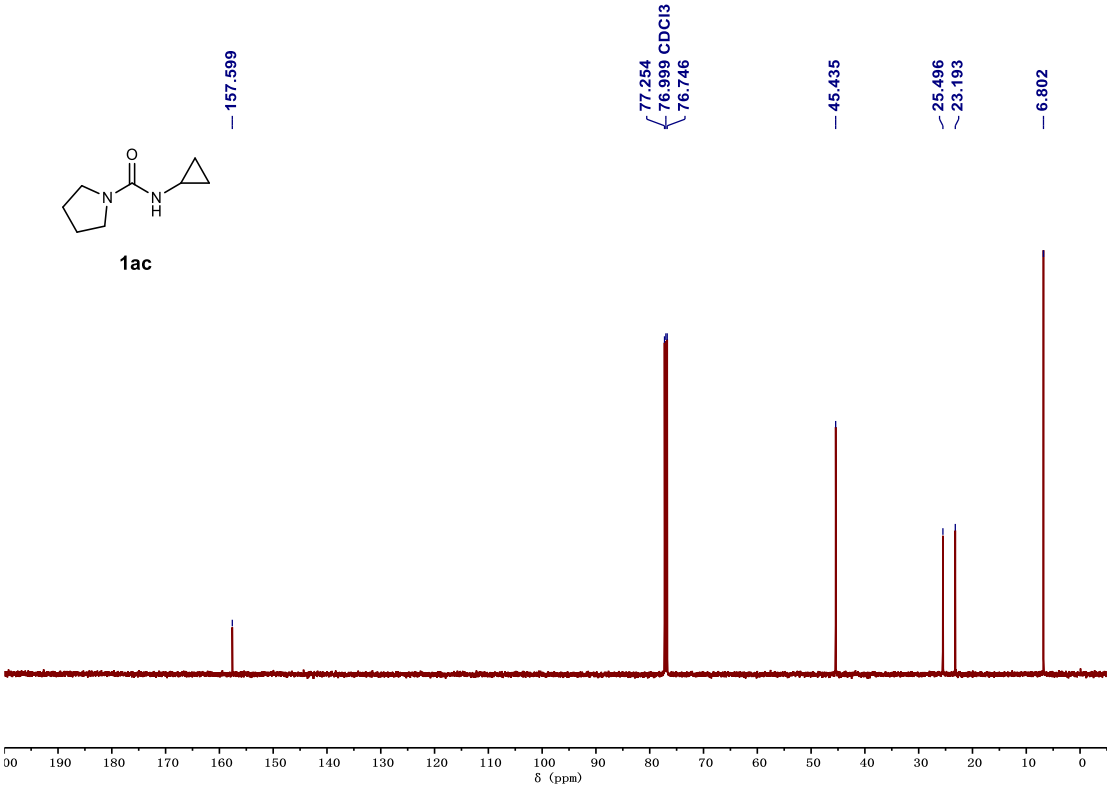
1ab


0.97
1.00
0.98
1.02
1.00
1.01
0.98
0.98
2.02
1.99
δ (ppm)

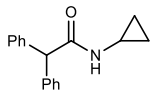

167.071
149.181
148.030
135.520
131.190
129.307
128.686
127.472
126.903
126.845
77.254
77.001 CDCl3
76.746
23.329
6.809
δ (ppm)

1ab

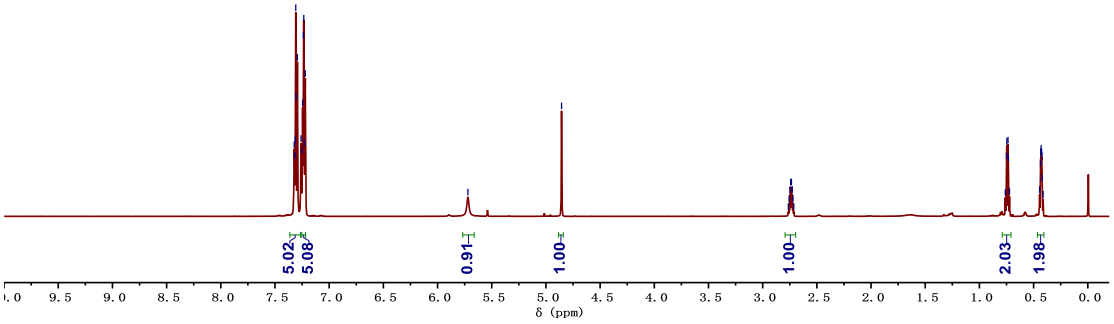

7.260 CDCl3
4.465
3.280
3.267
3.254
2.643
2.639
2.635
2.631
2.628
2.624
2.621
2.617
2.614
2.610
2.607
2.603
2.600
2.596
1.865
1.860
1.858
1.852
1.846
1.844
1.838
0.694
0.684
0.680
0.670
0.666
0.657
0.458
0.450
0.448
0.444
0.440
0.437
0.434
0.427
1ac
0.94
4.05
1.00
4.10
2.01
1.99
δ (ppm)

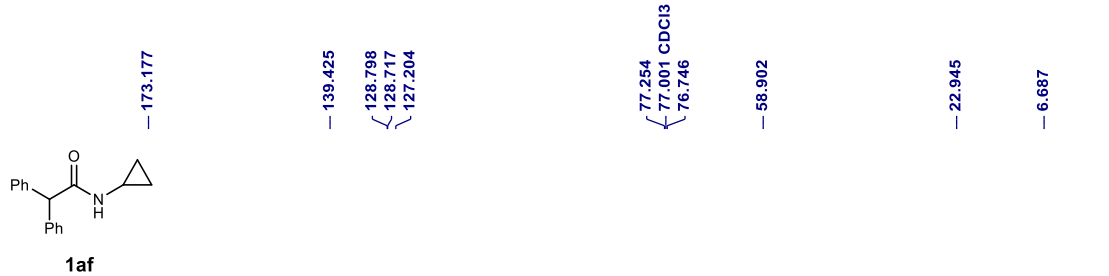

157.599
77.254
76.999 CDCl3
76.746
45.435
25.496
23.193
6.802
1ac
δ (ppm)


7.323
7.319
7.313
7.308
7.306
7.293
7.259 CDCl3
7.251
7.245
7.238
7.234
7.230
7.221
5.720
4.856
2.763
2.756
2.749
2.742
2.734
2.727
2.720
2.713
0.761
0.751
0.747
0.736
0.733
0.722
0.445
0.437
0.434
0.431
0.426
0.423
0.420
0.412


Ph
O
N
H
Ph

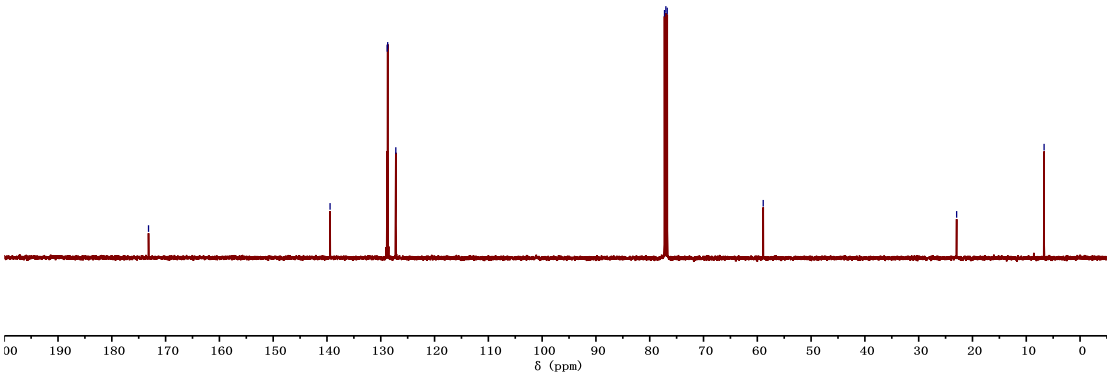
1af

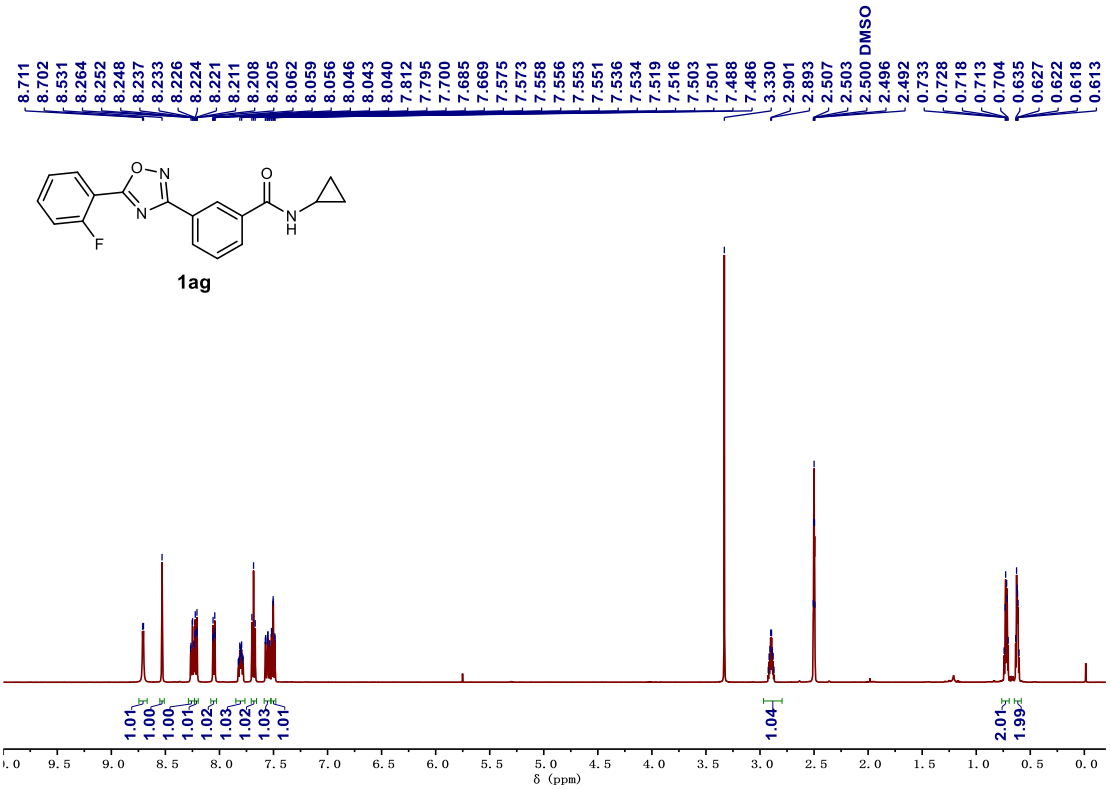

5.02
5.08
0.91
1.00
1.00
2.03
1.98
δ (ppm)


173.177
139.425
128.798
128.717
127.204
77.254
77.001 CDCl3
76.746
58.902
22.945
6.687

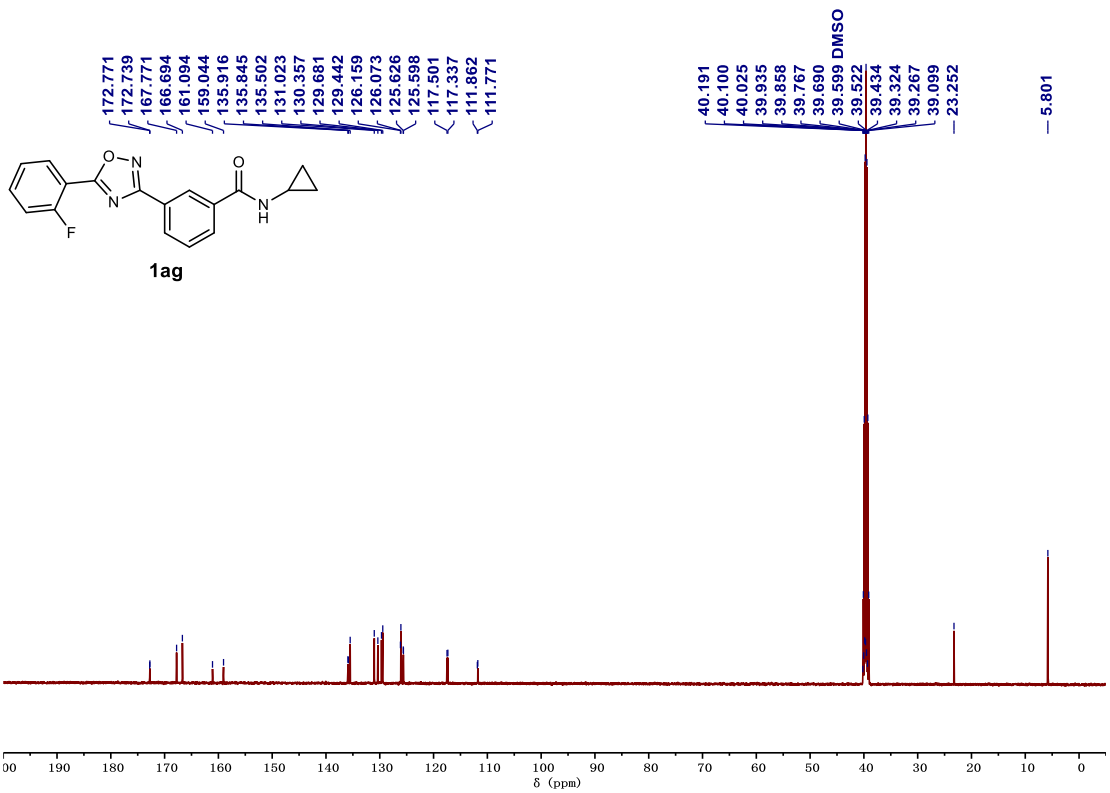
Ph
O
N
H
Ph

1af

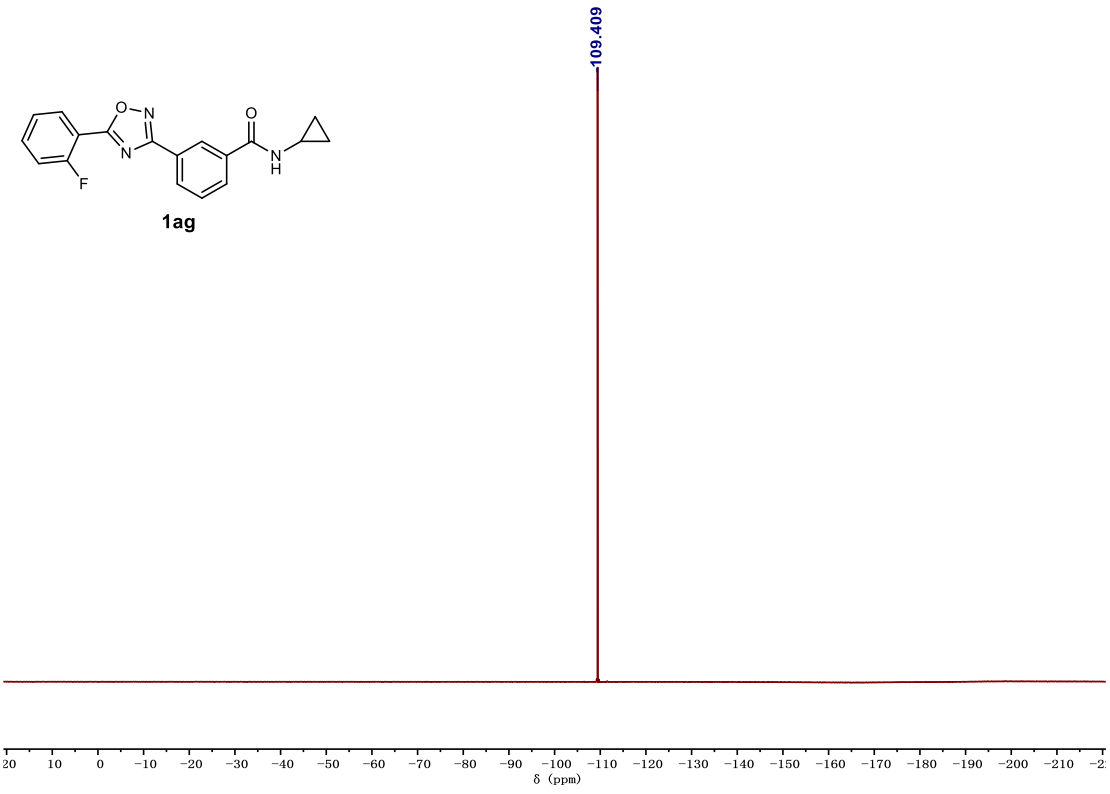

δ (ppm)


8.711
8.702
8.531
8.264
8.252
8.248
8.237
8.233
8.226
8.224
8.221
8.211
8.208
8.205
8.062
8.059
8.056
8.046
8.043
8.040
7.812
7.795
7.700
7.685
7.669
7.575
7.573
7.558
7.556
7.553
7.551
7.536
7.534
7.519
7.516
7.503
7.501
7.488
7.486
3.330
2.901
2.893
2.507
2.503
2.500 DMSO
2.496
2.492
0.733
0.728
0.718
0.713
0.704
0.635
0.627
0.622
0.618
0.613
1.01
1.00
1.00
1.01
1.02
1.03
1.02
1.03
1.01
1.04
2.01
1.99
δ (ppm)

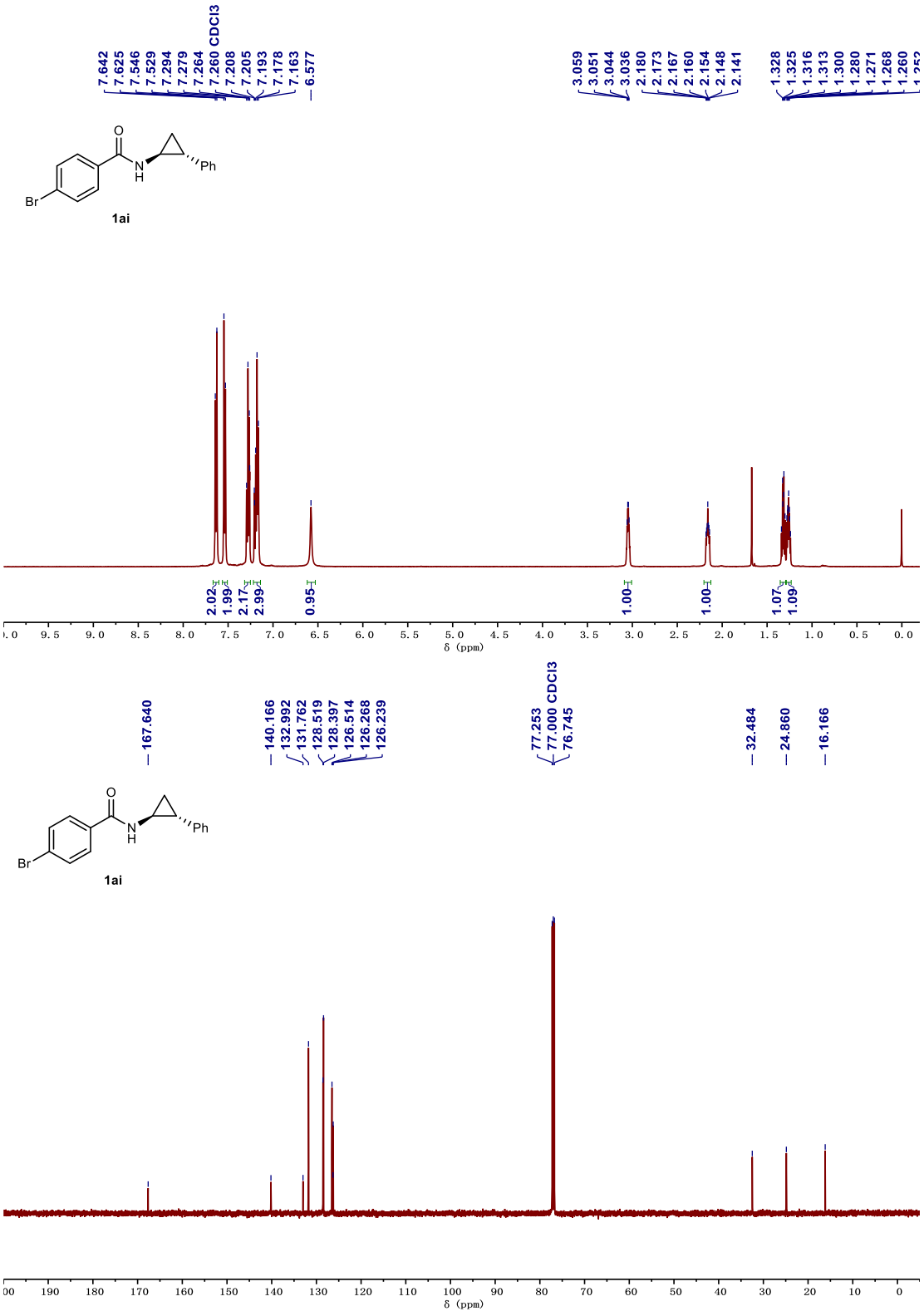
1ag

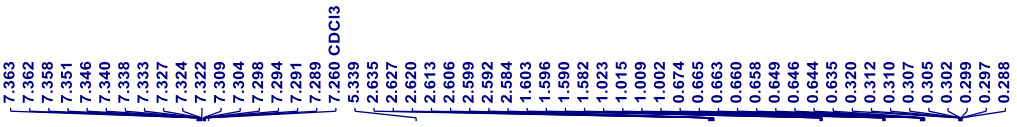

172.771
172.739
167.771
166.694
161.094
159.044
135.916
135.845
135.502
131.023
130.357
129.681
129.442
126.159
126.073
125.626
125.598
117.501
117.337
111.862
111.771
40.191
40.100
40.025
39.935
39.858
39.767
39.690
39.599 DMSO
39.522
39.434
39.324
39.267
39.099
23.252
5.801
δ (ppm)

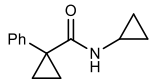
1ag


109.409
O
N
N
O
N
H
F
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)

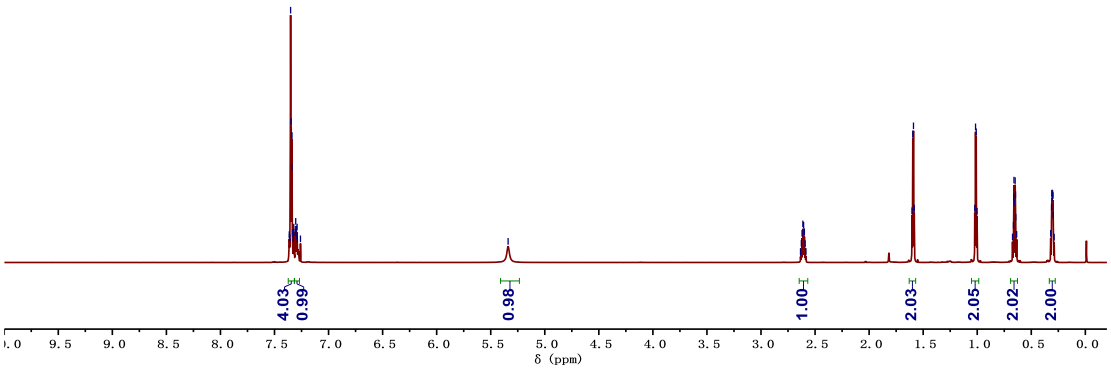
1ag


7.642
7.625
7.546
7.529
7.294
7.279
7.264
7.260 CDCl3
7.208
7.205
7.193
7.178
7.163
6.577
3.059
3.051
3.044
3.036
2.180
2.173
2.167
2.160
2.154
2.148
2.141
1.328
1.325
1.316
1.313
1.300
1.280
1.271
1.268
1.260
O
N
H
Ph
Br
1ai
2.02
1.99
2.17
2.99
0.95
1.00
1.00
1.07
1.09
δ (ppm)
167.640
140.166
132.992
131.762
128.519
128.397
126.514
126.268
126.239
77.253
77.000 CDCl3
76.745
32.484
24.860
16.166
O
N
H
Ph
Br
1ai
δ (ppm)

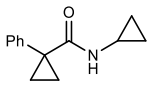

7.363
7.362
7.358
7.351
7.346
7.340
7.338
7.333
7.327
7.324
7.322
7.309
7.304
7.298
7.294
7.291
7.289
7.260 CDCl3
5.339
2.635
2.627
2.620
2.613
2.606
2.599
2.592
2.584
1.603
1.596
1.590
1.582
1.023
1.015
1.009
1.002
0.674
0.665
0.663
0.660
0.658
0.649
0.646
0.644
0.635
0.320
0.312
0.310
0.307
0.305
0.302
0.299
0.297
0.288


O
Ph
N
H

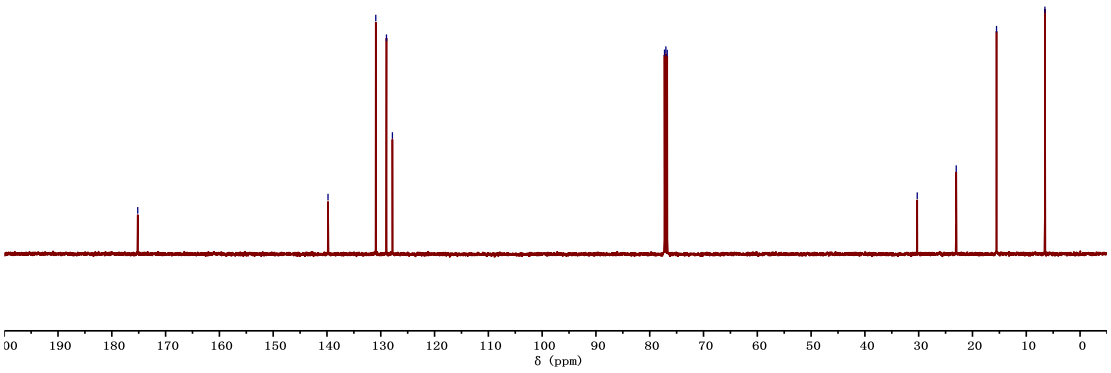
1aj

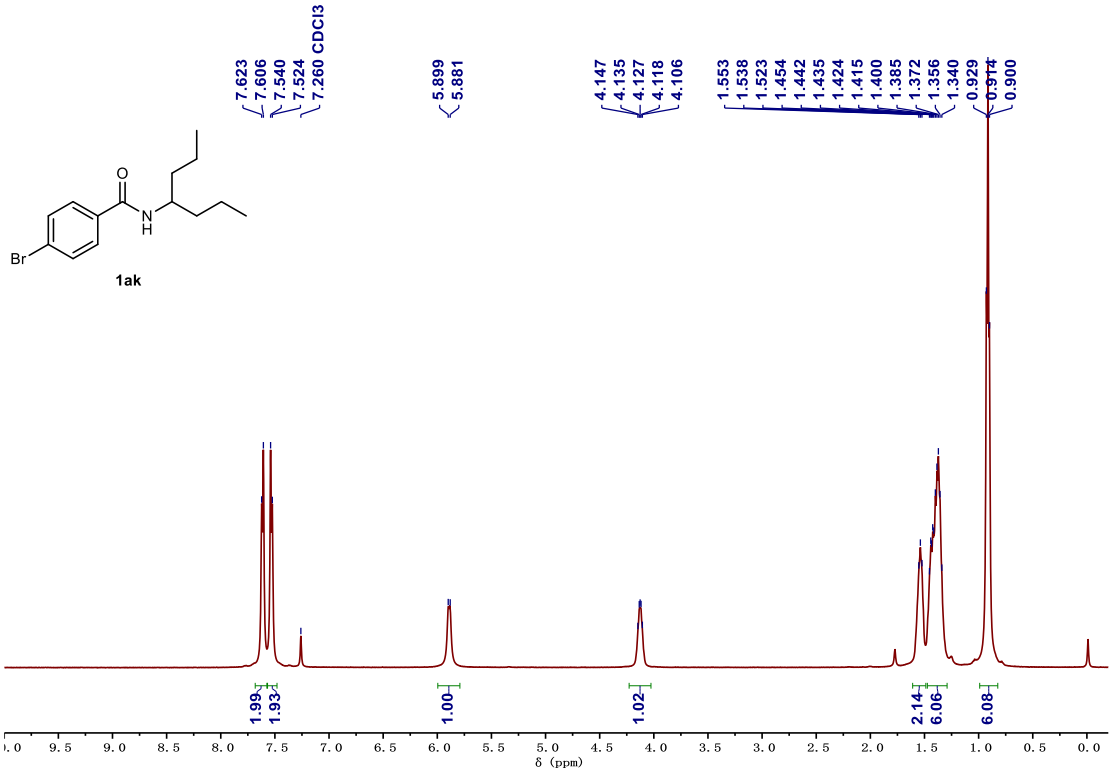

4.03
0.99
0.98
1.00
2.03
2.05
2.02
2.00
δ (ppm)

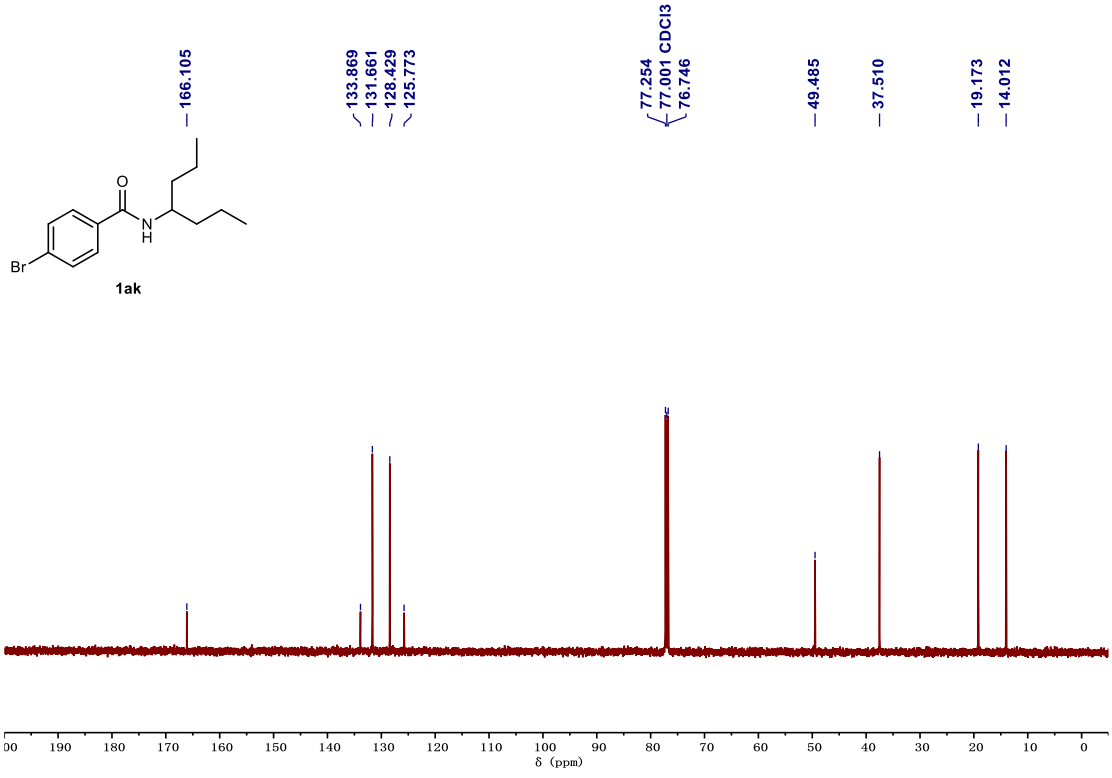

175.180
139.807
130.931
128.941
127.836
77.254
76.999 CDCl3
76.746
30.269
23.021
15.530
6.536

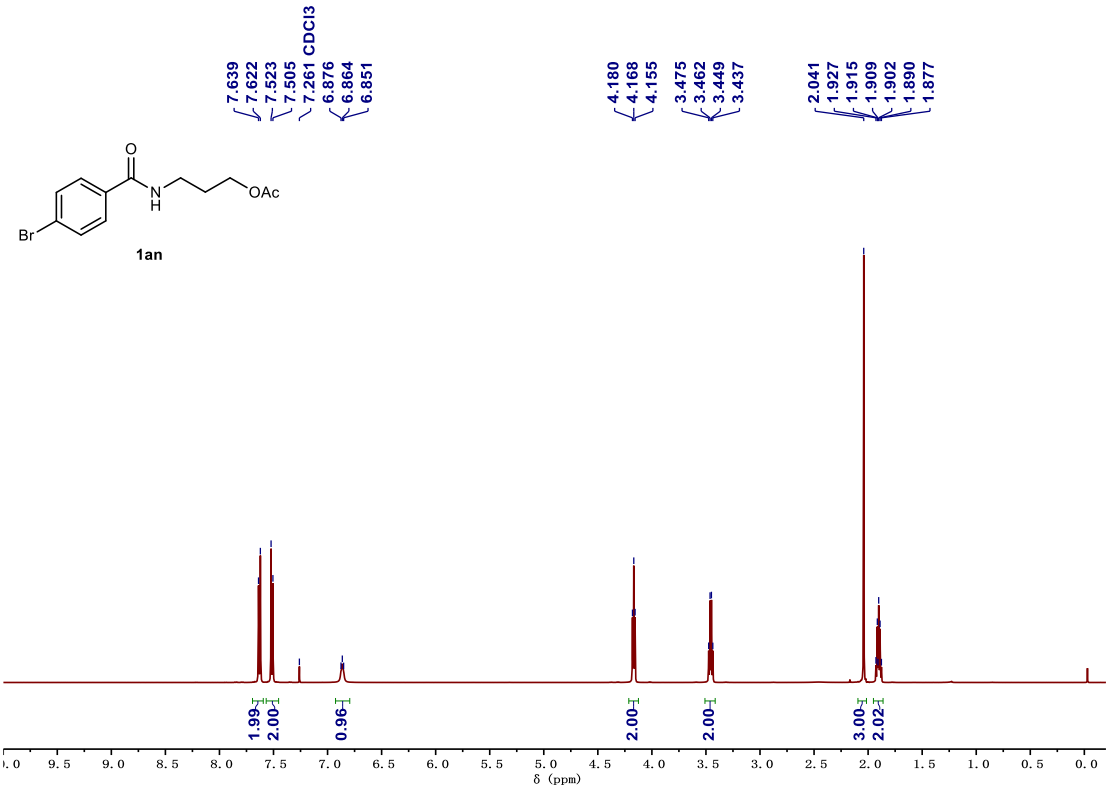

O
Ph
N
H

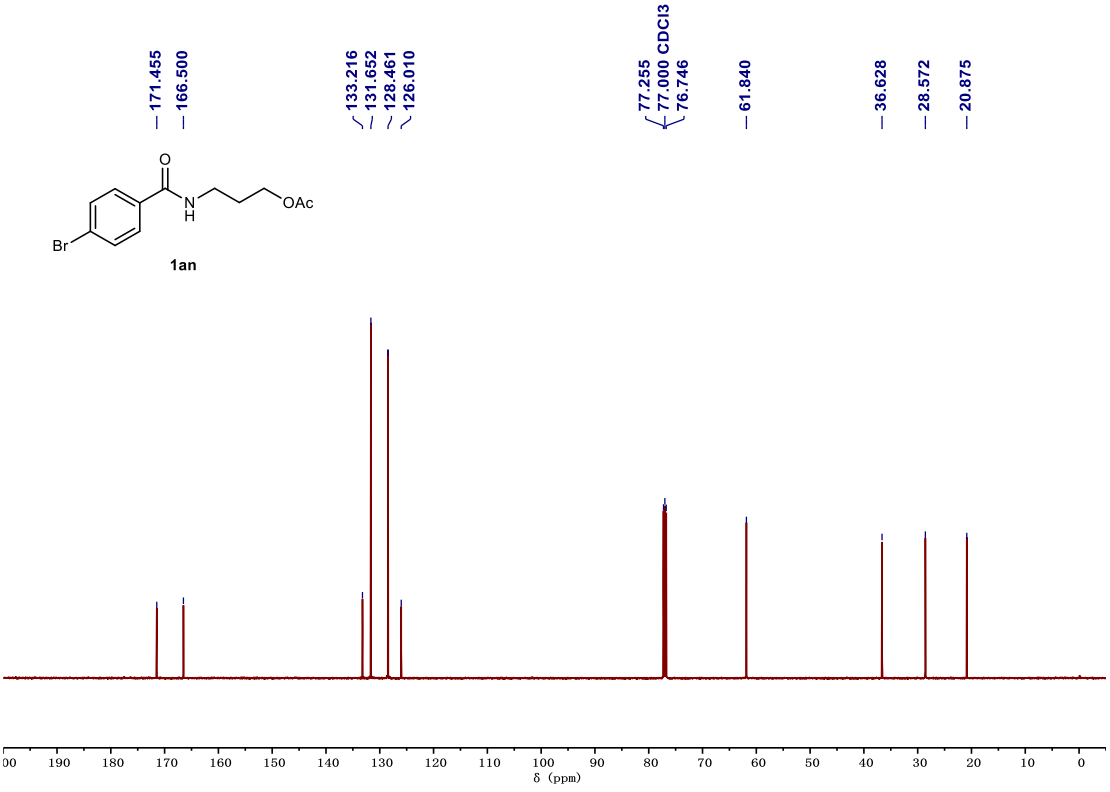
1aj

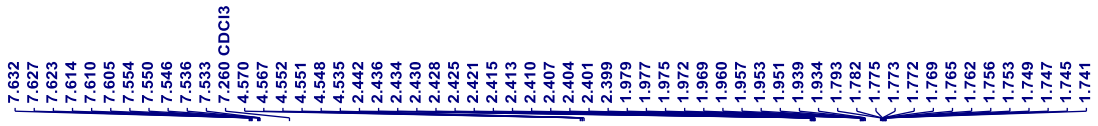

δ (ppm)

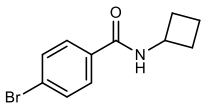

7.623
7.606
7.540
7.524
7.260 CDCl3
5.899
5.881
4.147
4.135
4.127
4.118
4.106
1.553
1.538
1.523
1.454
1.442
1.435
1.424
1.415
1.400
1.385
1.372
1.356
1.340
0.929
0.914
0.900
O
N
H
Br
1ak
1.99
1.93
1.00
1.02
2.14
6.06
6.08
δ (ppm)


166.105
133.869
131.661
128.429
125.773
77.254
77.001 CDCl3
76.746
49.485
37.510
19.173
14.012
O
N
H
Br
1ak
δ (ppm)

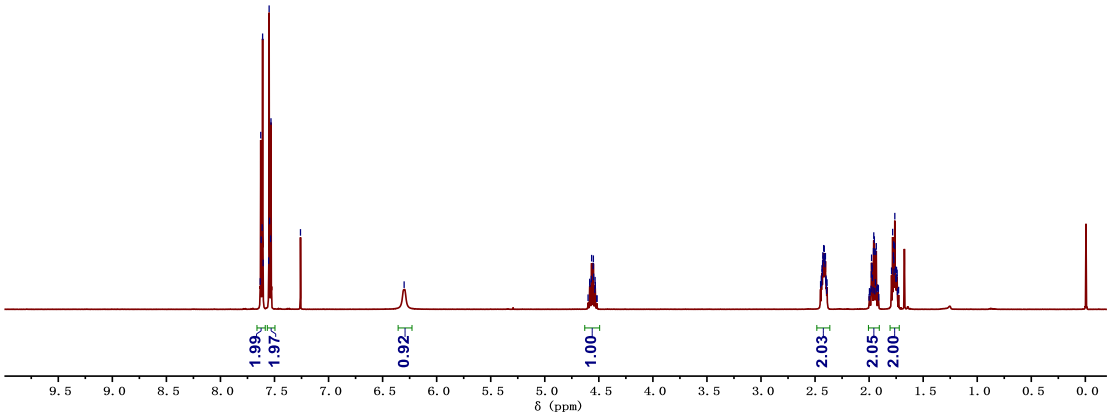

7.639
7.622
7.523
7.505
7.261 CDCl3
6.876
6.864
6.851
4.180
4.168
4.155
3.475
3.462
3.449
3.437
2.041
1.927
1.915
1.909
1.902
1.890
1.877
O
N
H
OAc
Br
1an
1.99
2.00
0.96
2.00
2.00
3.00
2.02
δ (ppm)

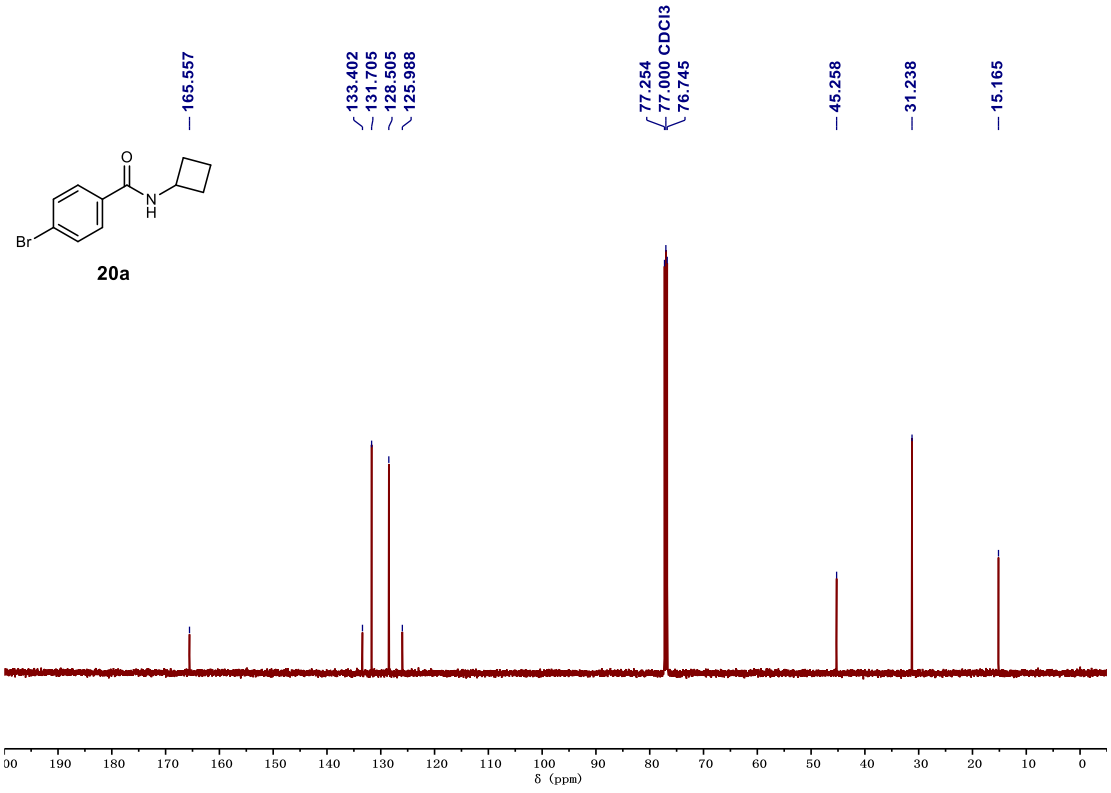

171.455
166.500
133.216
131.652
128.461
126.010
77.255
77.000 CDCl3
76.746
61.840
36.628
28.572
20.875
O
N
H
OAc
Br
1an
δ (ppm)


7.632
7.627
7.623
7.614
7.610
7.605
7.554
7.550
7.546
7.536
7.533
7.260 CDCl3
4.570
4.567
4.552
4.551
4.548
4.535
2.442
2.436
2.434
2.430
2.428
2.425
2.421
2.415
2.413
2.410
2.407
2.404
2.401
2.399
1.979
1.977
1.975
1.972
1.969
1.960
1.957
1.953
1.951
1.939
1.934
1.793
1.782
1.775
1.773
1.772
1.769
1.765
1.762
1.756
1.753
1.749
1.747
1.745
1.741


O
N
H
Br

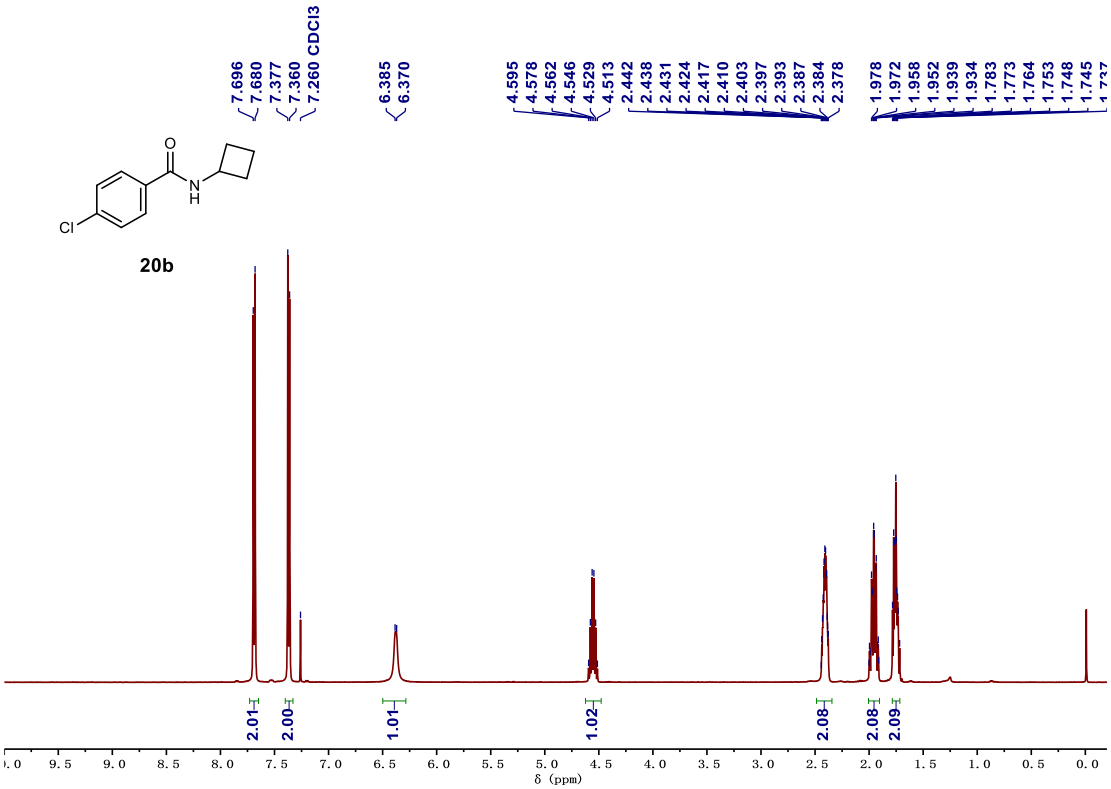
20a

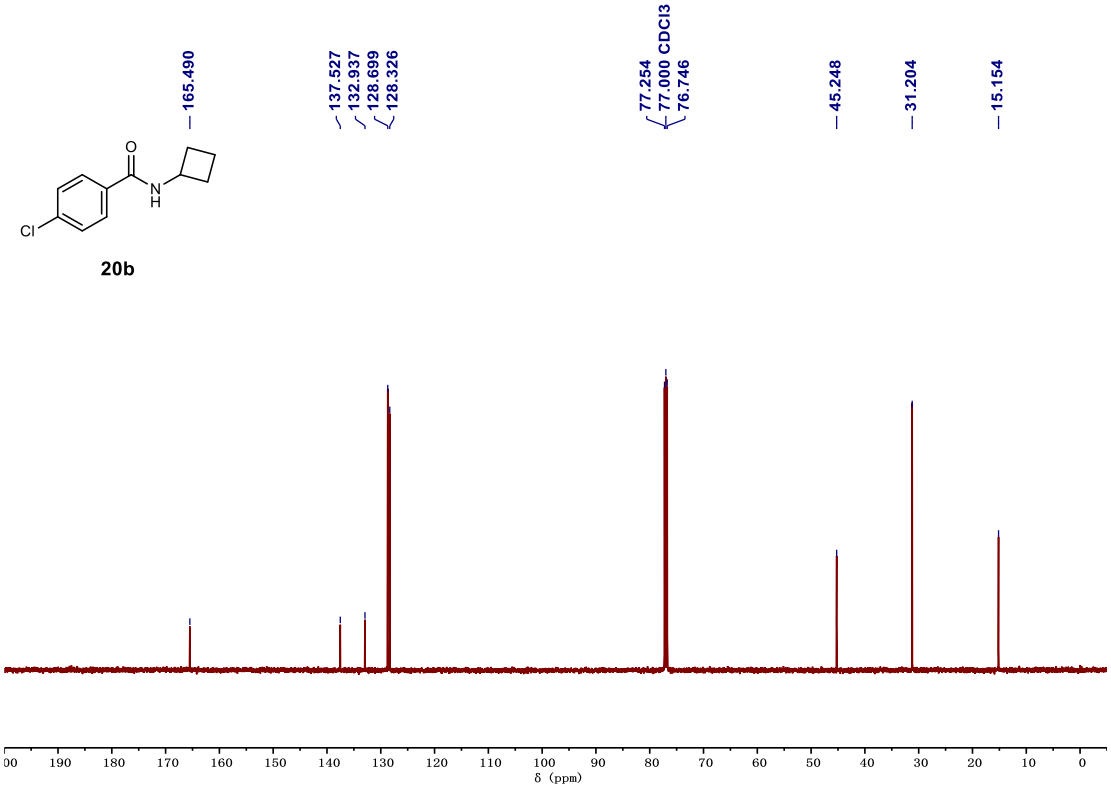

1.99
1.97
0.92
1.00
2.03
2.05
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ (ppm)

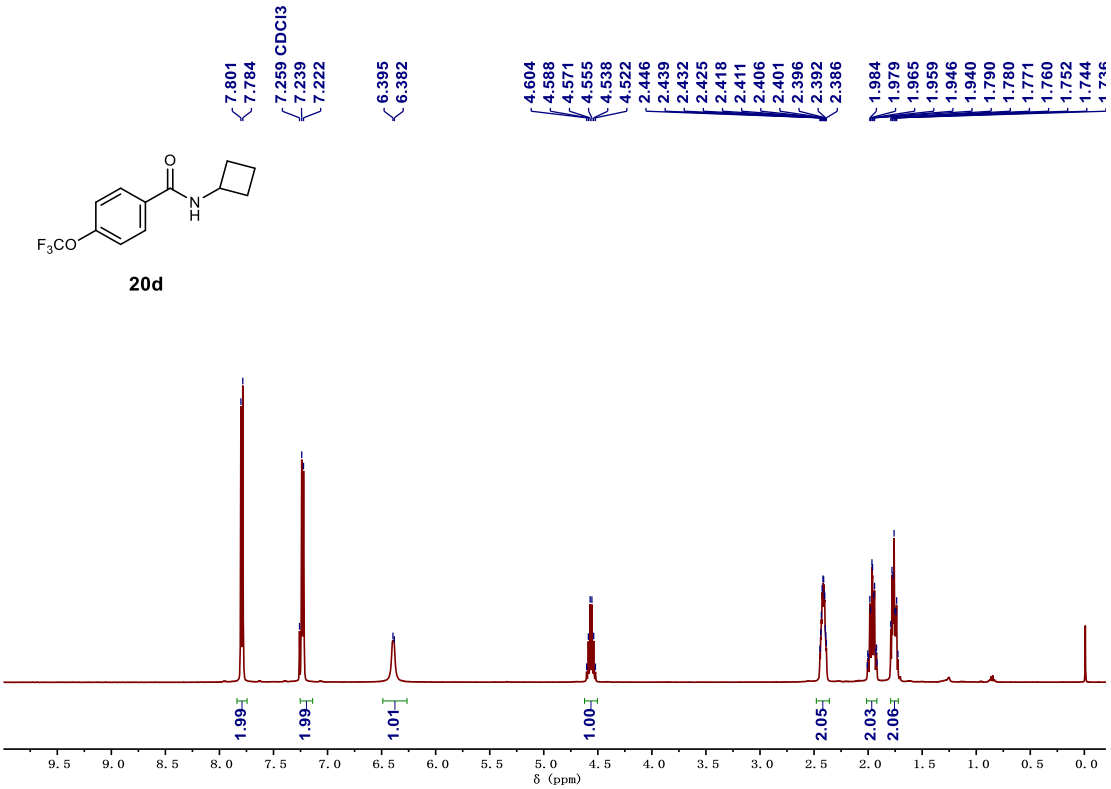

165.557
133.402
131.705
128.505
125.988
77.254
77.000 CDCl3
76.745
45.258
31.238
15.165
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

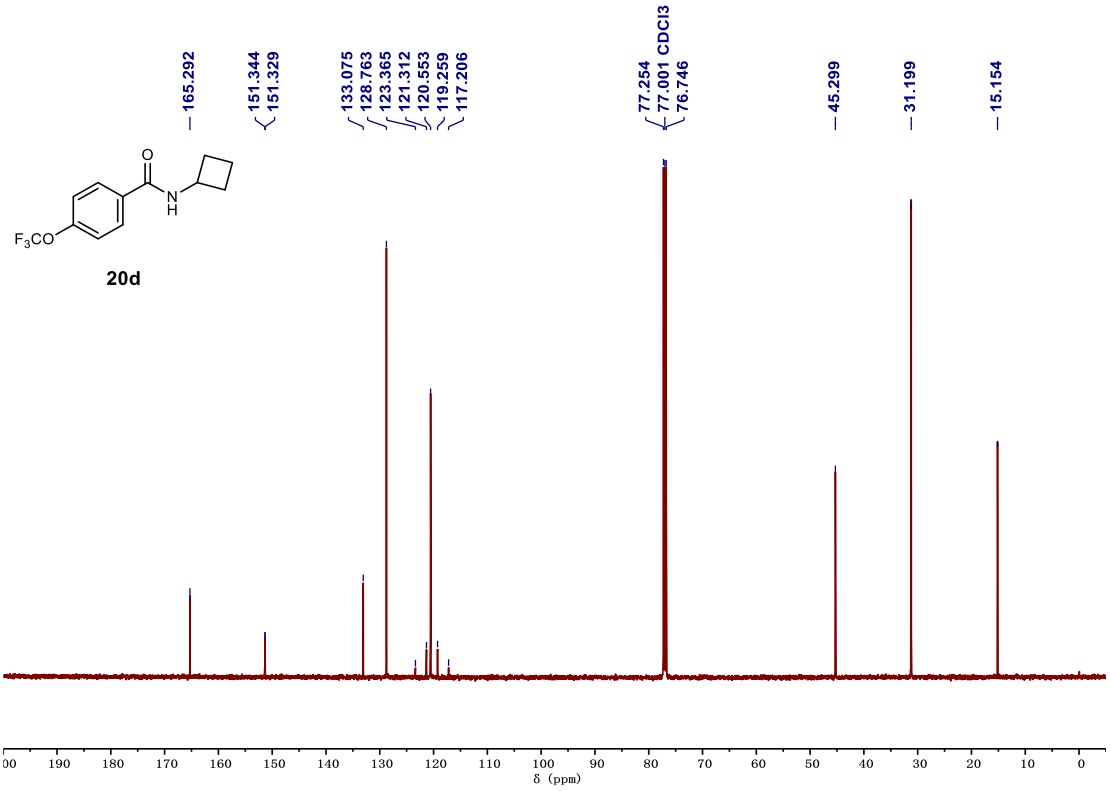
O
N
H
Br

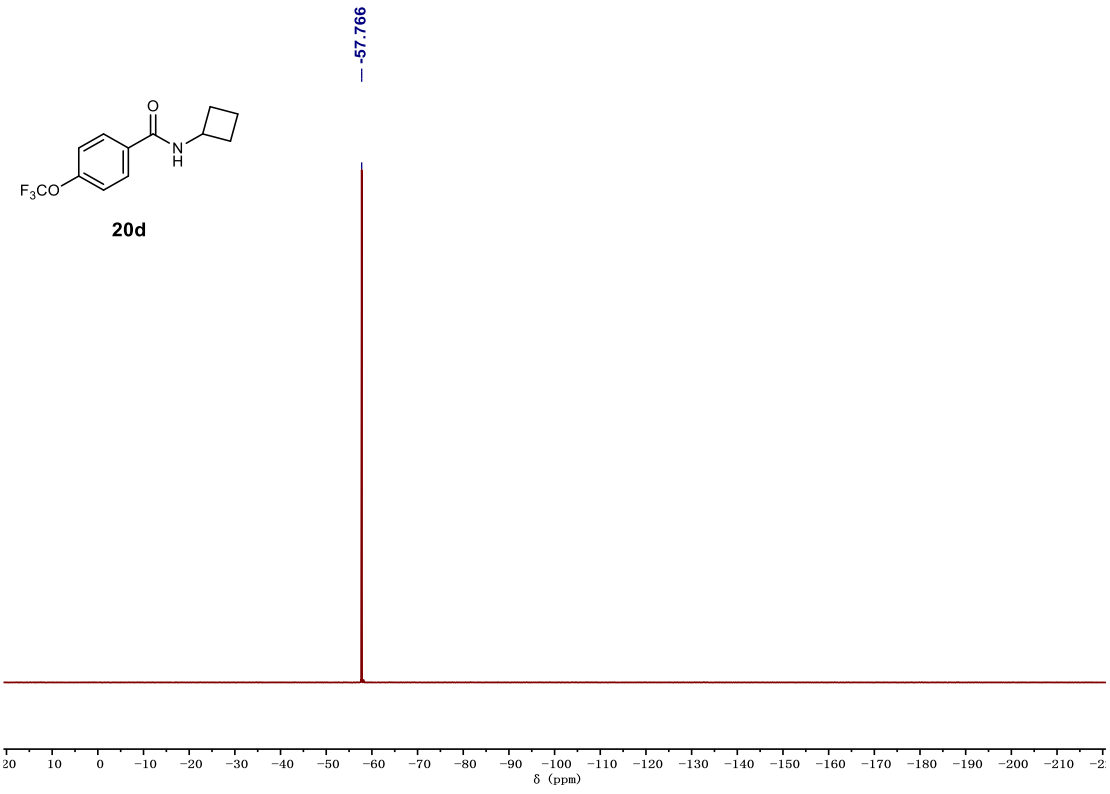
20a

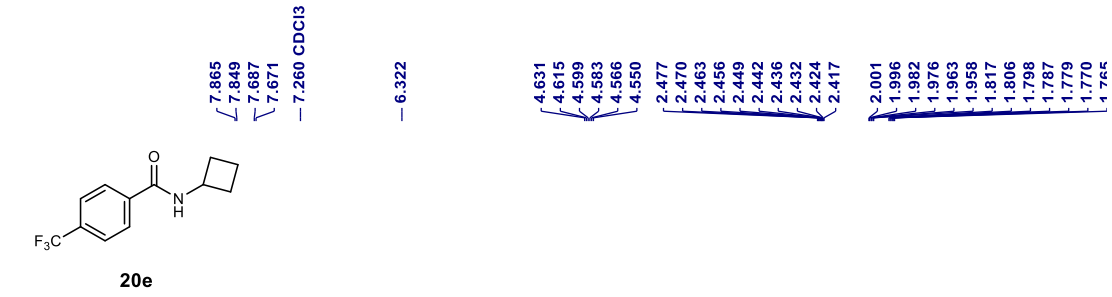

7.696
7.680
7.377
7.360
7.260 CDCl3
6.385
6.370
4.595
4.578
4.562
4.546
4.529
4.513
2.442
2.438
2.431
2.424
2.417
2.410
2.403
2.397
2.393
2.387
2.384
2.378
1.978
1.972
1.958
1.952
1.939
1.934
1.783
1.773
1.764
1.753
1.748
1.745
O
N
H
Cl
20b
2.01
2.00
1.01
1.02
2.08
2.08
2.09
δ (ppm)


165.490
137.527
132.937
128.699
128.326
77.254
77.000 CDCl3
76.746
45.248
31.204
15.154
O
N
H
Cl
20b
δ (ppm)

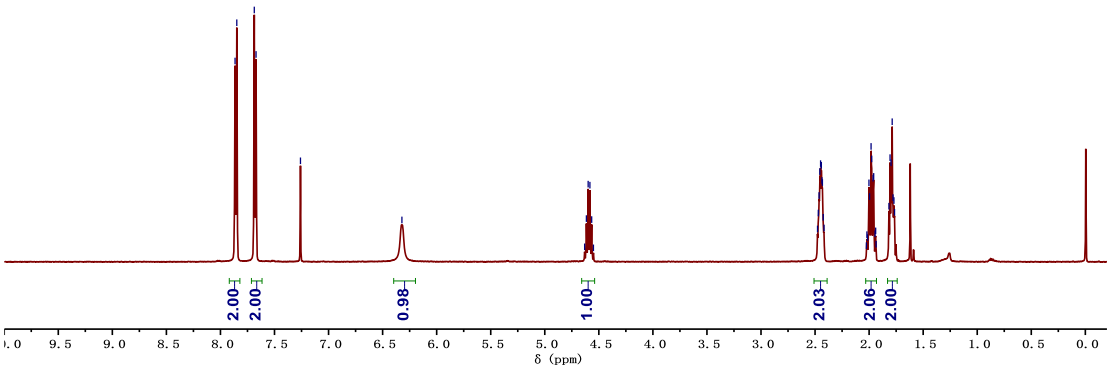

7.801
7.784
7.259 CDCl3
7.239
7.222
6.395
6.382
4.604
4.588
4.571
4.555
4.538
4.522
2.446
2.439
2.432
2.425
2.418
2.411
2.406
2.401
2.396
2.392
2.386
1.984
1.979
1.965
1.959
1.946
1.940
1.790
1.780
1.771
1.760
1.752
1.744
O
N
H
F3CO
20d
1.99
1.99
1.01
1.00
2.05
2.03
2.06
δ (ppm)

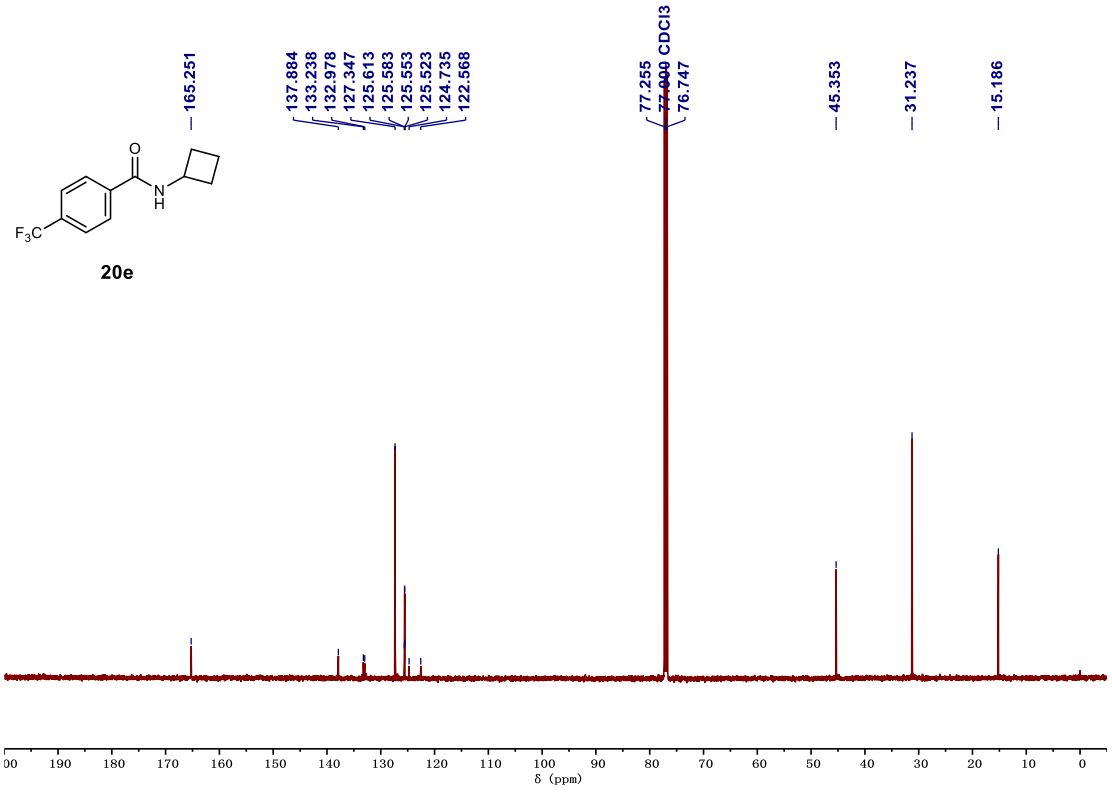

165.292
151.344
151.329
133.075
128.763
123.365
121.312
120.553
119.259
117.206
77.254
77.001 CDCl3
76.746
45.299
31.199
15.154
O
N
H
F3CO
20d
δ (ppm)


-57.766
O
N
H
F3CO
20d
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)

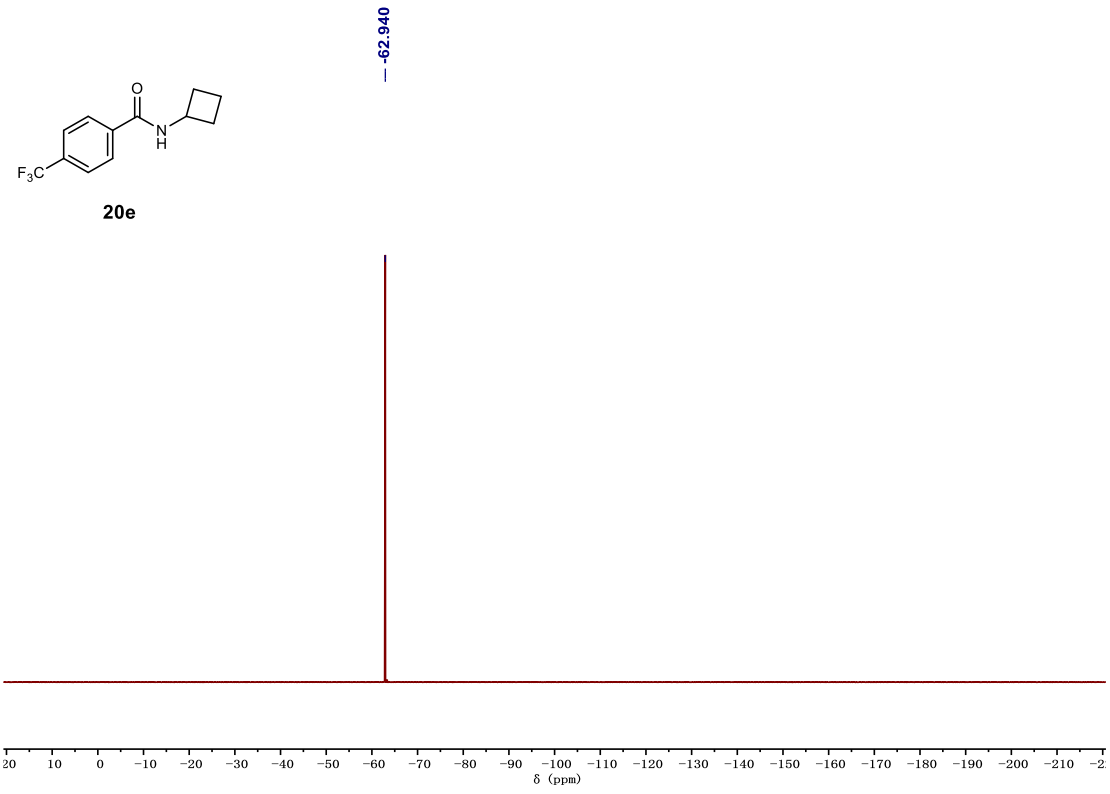

7.865
7.849
7.687
7.671
7.260 CDCl3
6.322
4.631
4.615
4.599
4.583
4.566
4.550
2.477
2.470
2.463
2.456
2.449
2.442
2.436
2.432
2.424
2.417
2.001
1.996
1.982
1.976
1.963
1.958
1.817
1.806
1.798
1.787
1.779
1.770
O
N
H
F3C

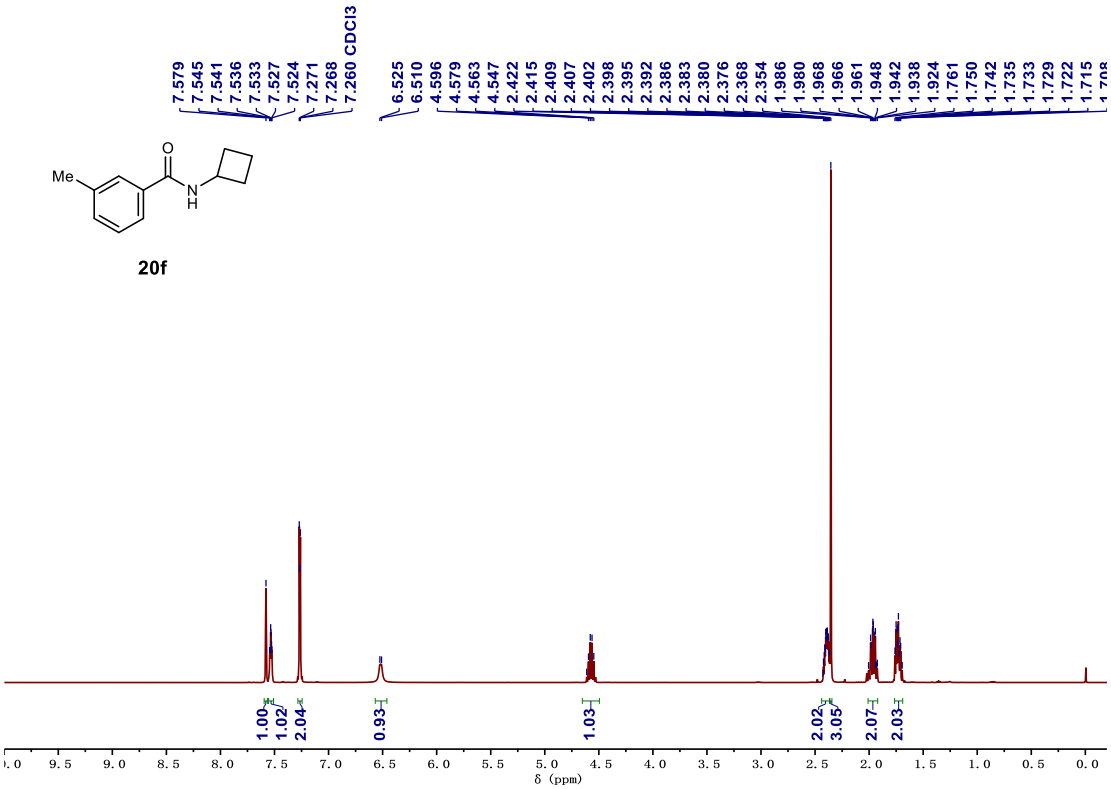
20e

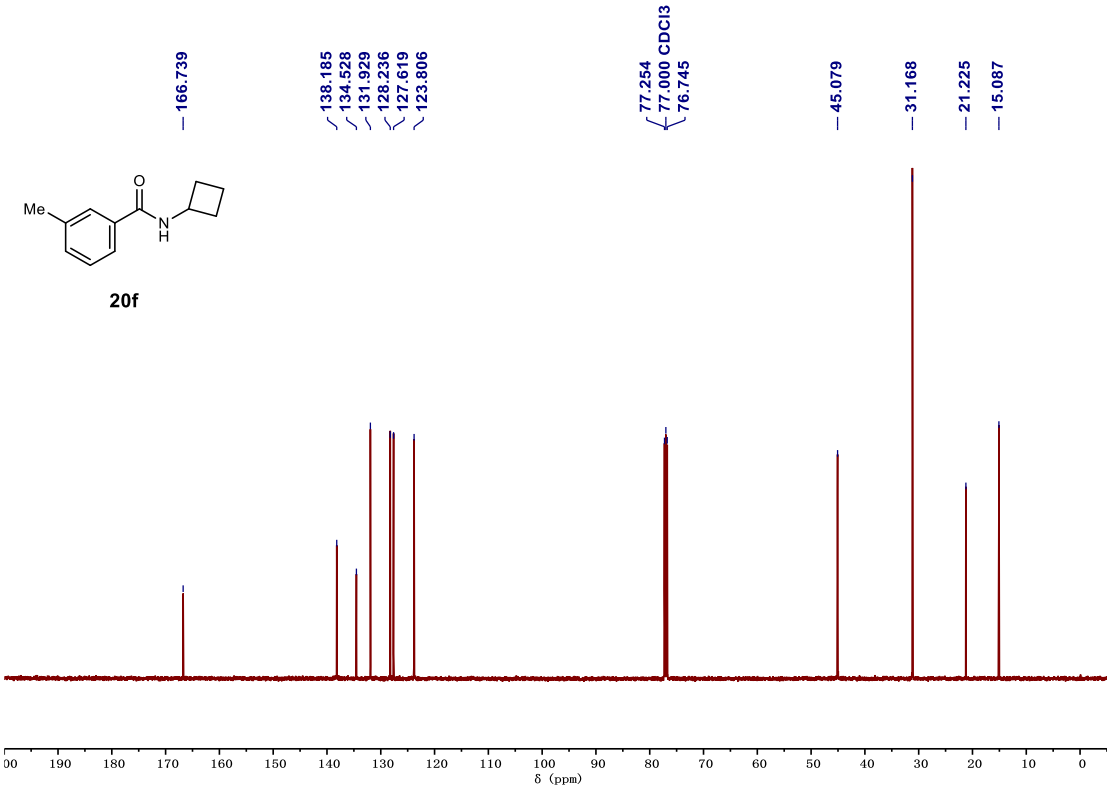

2.00
2.00
0.98
1.00
2.03
2.06
2.00
δ (ppm)

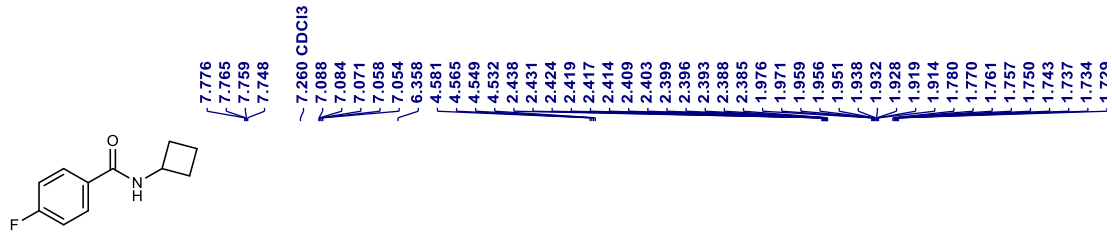

165.251
137.884
133.238
132.978
127.347
125.613
125.583
125.553
125.523
124.735
122.568
77.255
77.000 CDCl3
76.747
45.353
31.237
15.186
O
N
H
F3C
δ (ppm)

20e

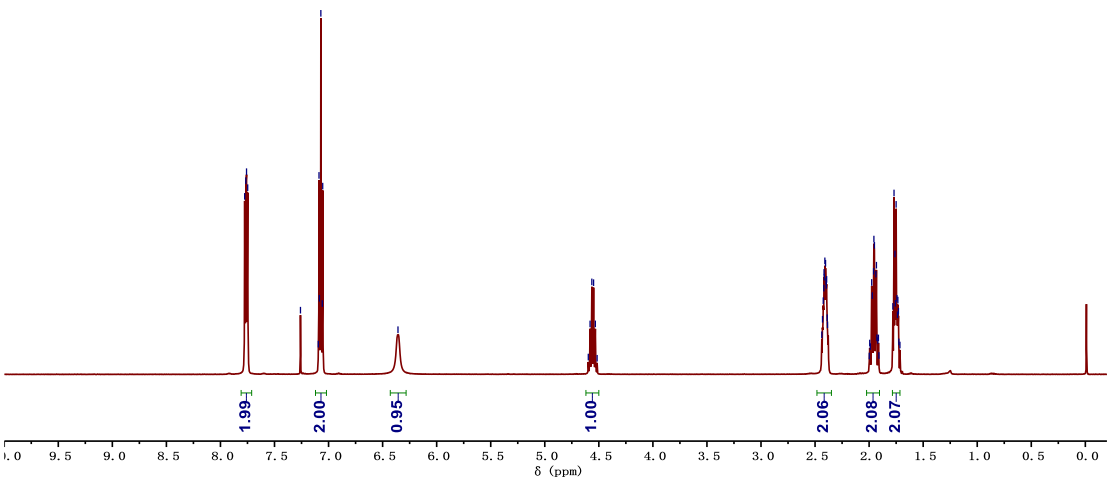

-62.940
O
N
H
F3C
20e
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
δ (ppm)


7.579
7.545
7.541
7.536
7.533
7.527
7.524
7.271
7.268
7.260 CDCl3
6.525
6.510
4.596
4.579
4.563
4.547
2.422
2.415
2.409
2.407
2.402
2.398
2.395
2.392
2.386
2.383
2.380
2.376
2.368
2.354
1.986
1.980
1.968
1.966
1.961
1.948
1.942
1.938
1.924
1.761
1.750
1.742
1.735
1.733
1.729
1.722
1.715
Me
O
N
H
20f
1.00
1.02
2.04
0.93
1.03
2.02
3.05
2.07
2.03
δ (ppm)

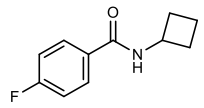

166.739
138.185
134.528
131.929
128.236
127.619
123.806
77.254
77.000 CDCl3
76.745
45.079
31.168
21.225
15.087
Me
O
N
H
20f
δ (ppm)


7.776
7.765
7.759
7.748
7.260 CDCl3
7.088
7.084
7.071
7.058
7.054
6.358
4.581
4.565
4.549
4.532
2.438
2.431
2.424
2.419
2.417
2.414
2.409
2.403
2.399
2.396
2.393
2.388
2.385
1.976
1.971
1.959
1.956
1.951
1.938
1.932
1.928
1.919
1.914
1.780
1.770
1.761
1.757
1.750
1.743
1.737
1.734
O
N
H
F

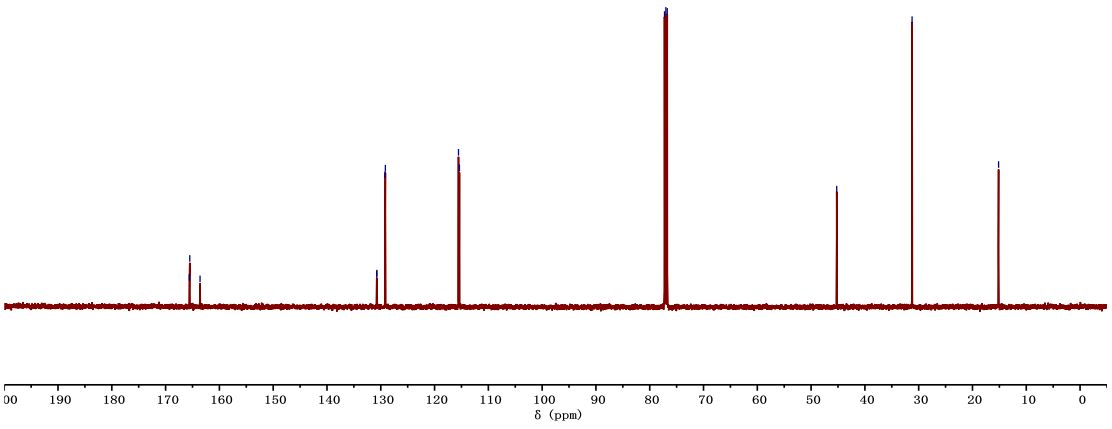
20g

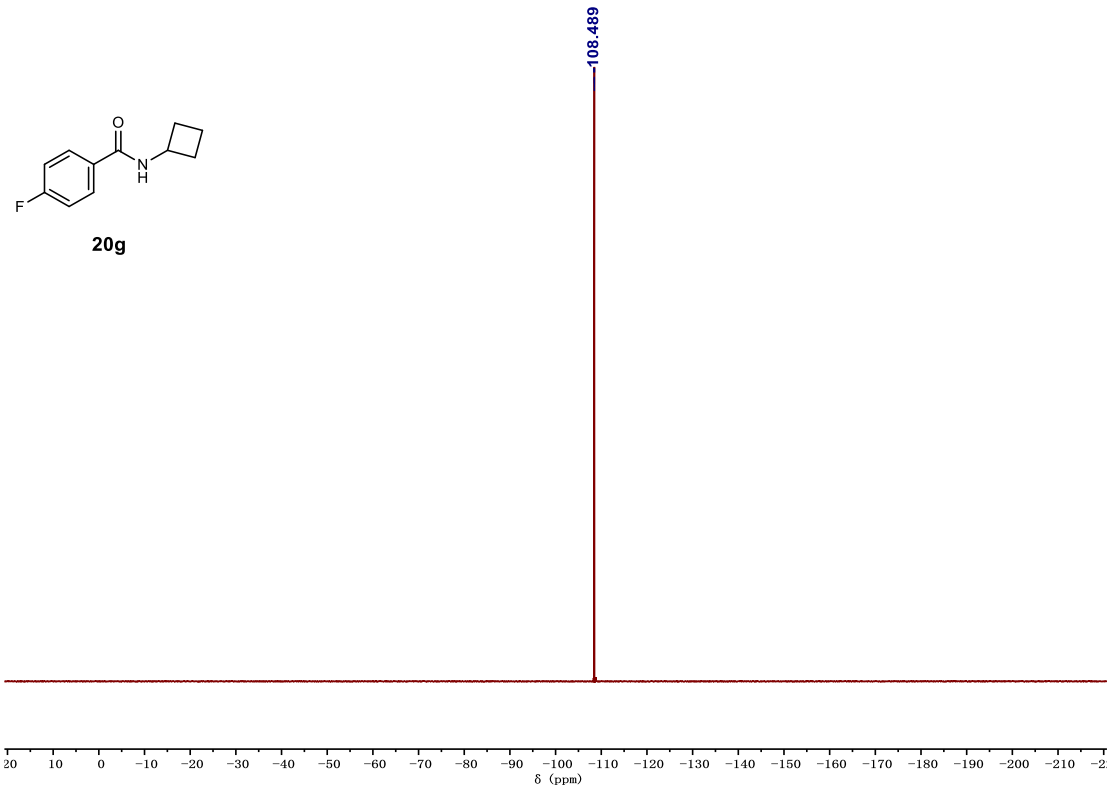

1.99
2.00
0.95
1.00
2.06
2.08
2.07
δ (ppm)

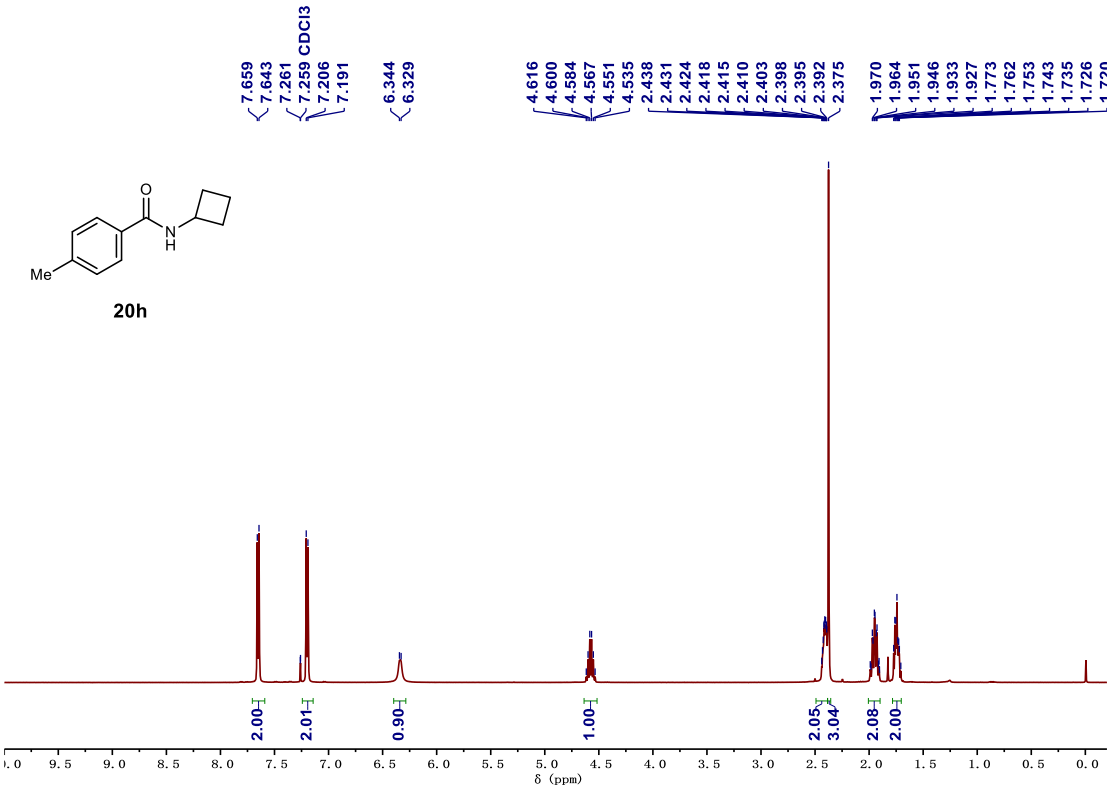

165.595
165.506
163.595
130.754
130.729
129.216
129.146
115.550
115.376
77.253
77.000 CDCl3
76.745
45.238
31.228
15.147

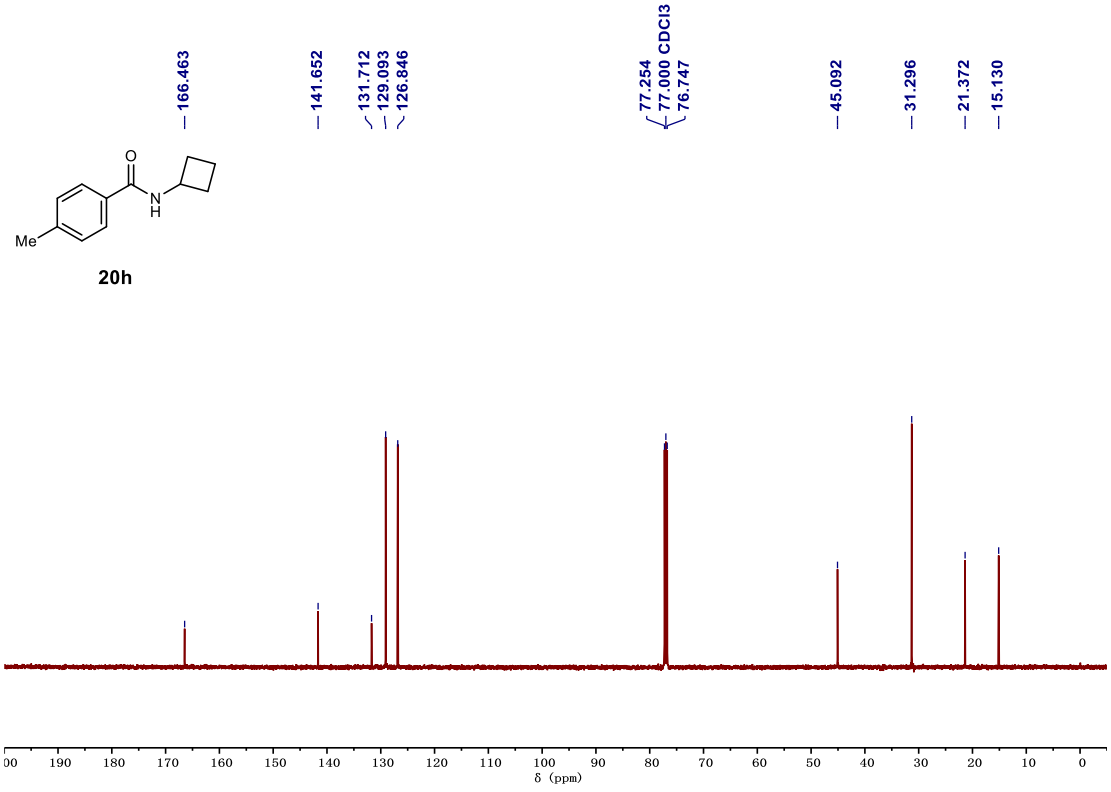

O
N
H
F

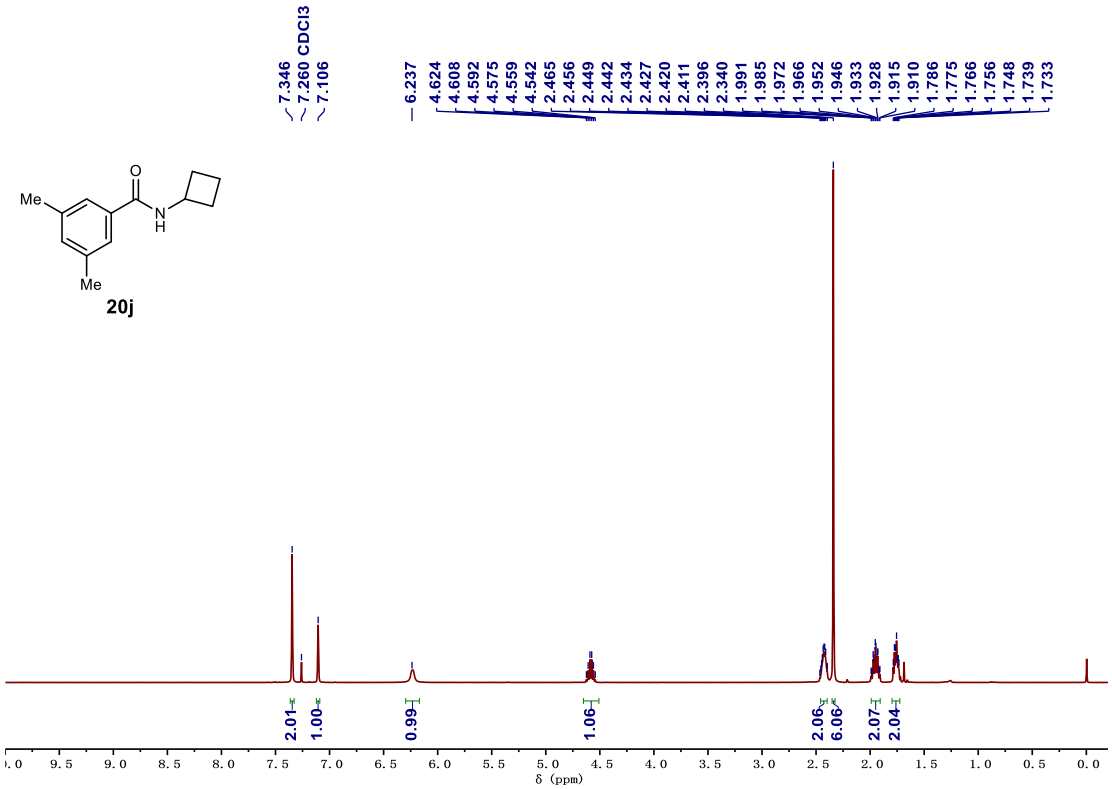
20g

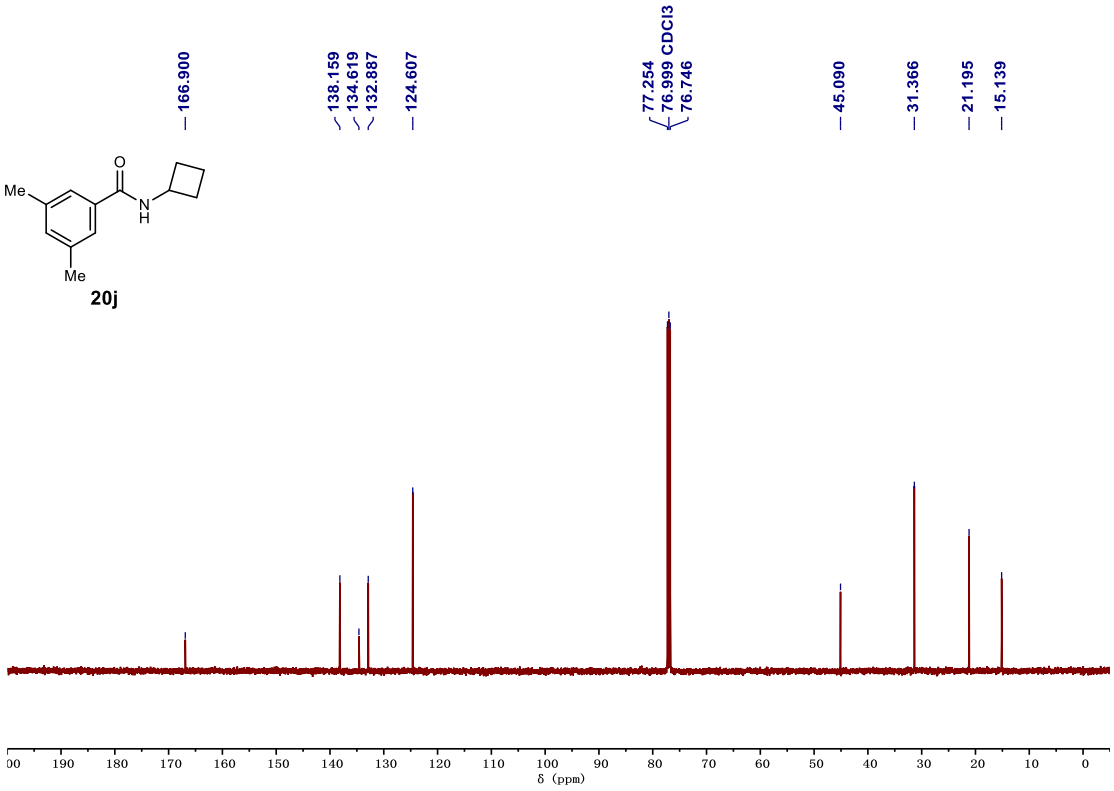

δ (ppm)


108.489
O
N
H
F
20g
20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
δ (ppm)

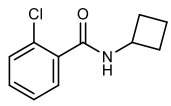

7.659
7.643
7.261
7.259 CDCl3
7.206
7.191
6.344
6.329
4.616
4.600
4.584
4.567
4.551
4.535
2.438
2.431
2.424
2.418
2.415
2.410
2.403
2.398
2.395
2.392
2.375
1.970
1.964
1.951
1.946
1.933
1.927
1.773
1.762
1.753
1.743
1.735
1.726
O
N
H
Me
20h
2.00
2.01
0.90
1.00
2.05
3.04
2.08
2.00
δ (ppm)


166.463
141.652
131.712
129.093
126.846
77.254
77.000 CDCl3
76.747
45.092
31.296
21.372
15.130
O
N
H
Me
20h
δ (ppm)

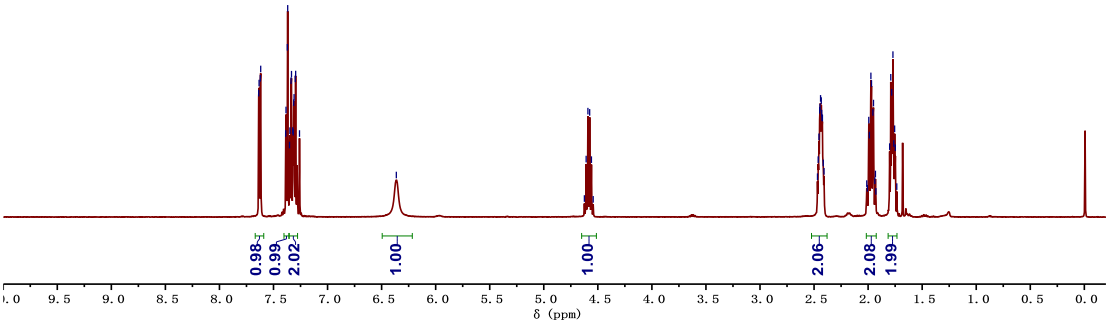

7.346
7.260 CDCl3
7.106
6.237
4.624
4.608
4.592
4.575
4.559
4.542
2.465
2.456
2.449
2.442
2.434
2.427
2.420
2.411
2.396
2.340
1.991
1.985
1.972
1.966
1.952
1.946
1.933
1.928
1.915
1.910
1.786
1.775
1.766
1.756
1.748
1.739
1.733
Me
Me
O
N
H
20j
2.01
1.00
0.99
1.06
2.06
6.06
2.07
2.04
δ (ppm)

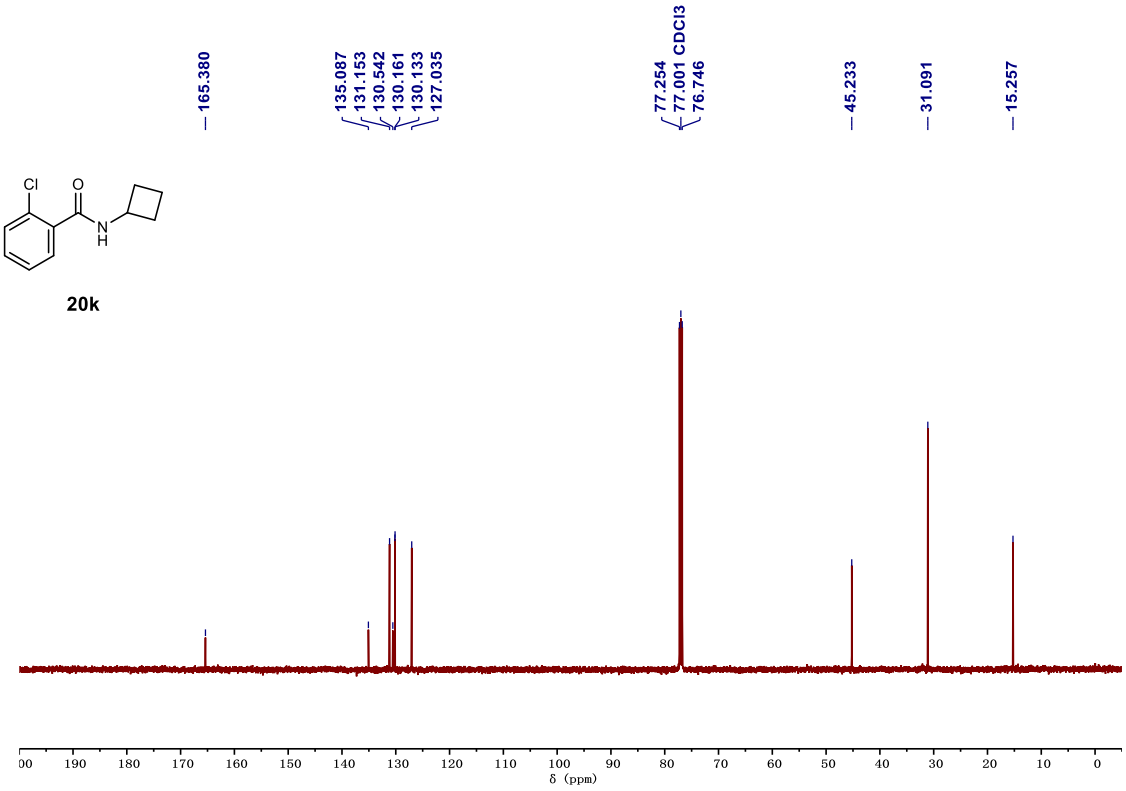

166.900
138.159
134.619
132.887
124.607
77.254
76.999 CDCl3
76.746
45.090
31.366
21.195
15.139
Me
Me
O
N
H
20j
δ (ppm)


7.637
7.633
7.622
7.618
7.388
7.384
7.372
7.369
7.352
7.348
7.337
7.334
7.322
7.318
7.313
7.310
7.298
7.295
7.260 CDCl3
6.364
4.624
4.608
4.592
4.576
4.559
4.543
2.468
2.461
2.455
2.448
2.445
2.440
2.433
2.429
2.427
2.422
2.415
2.408
1.994
1.988
1.974
1.969
1.956
1.950
1.801
1.790
1.782
1.770
1.763
1.755


Cl
O
N
H

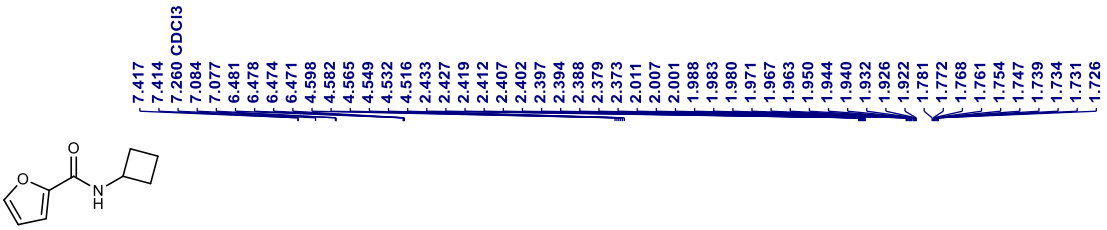
20k


0.98
0.99
2.02
1.00
1.00
2.06
2.08
1.99
δ (ppm)

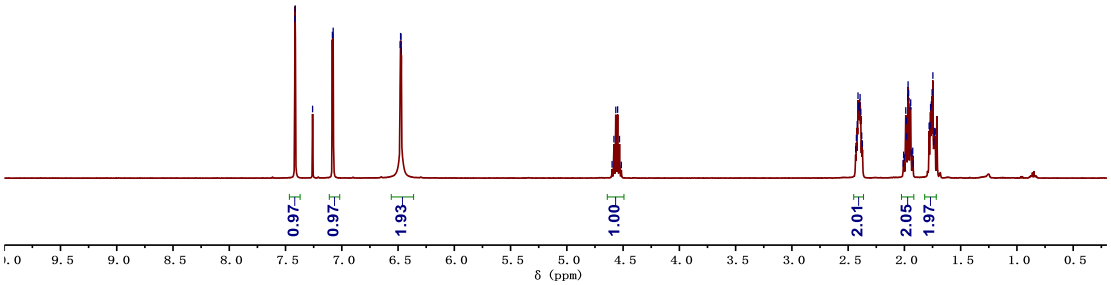

165.380
135.087
131.153
130.542
130.161
130.133
127.035
77.254
77.001 CDCl3
76.746
45.233
31.091
15.257
δ (ppm)

Cl
O
N
H

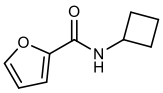
20k


7.417
7.414
7.260 CDCl3
7.084
7.077
6.481
6.478
6.474
6.471
4.598
4.582
4.565
4.549
4.532
4.516
2.433
2.427
2.419
2.412
2.407
2.402
2.397
2.394
2.388
2.379
2.373
2.011
2.007
2.001
1.988
1.983
1.980
1.971
1.967
1.963
1.950
1.944
1.940
1.932
1.926
1.922
1.781
1.772
1.768
1.761
1.754
1.747
1.739
1.734
1.731
1.726

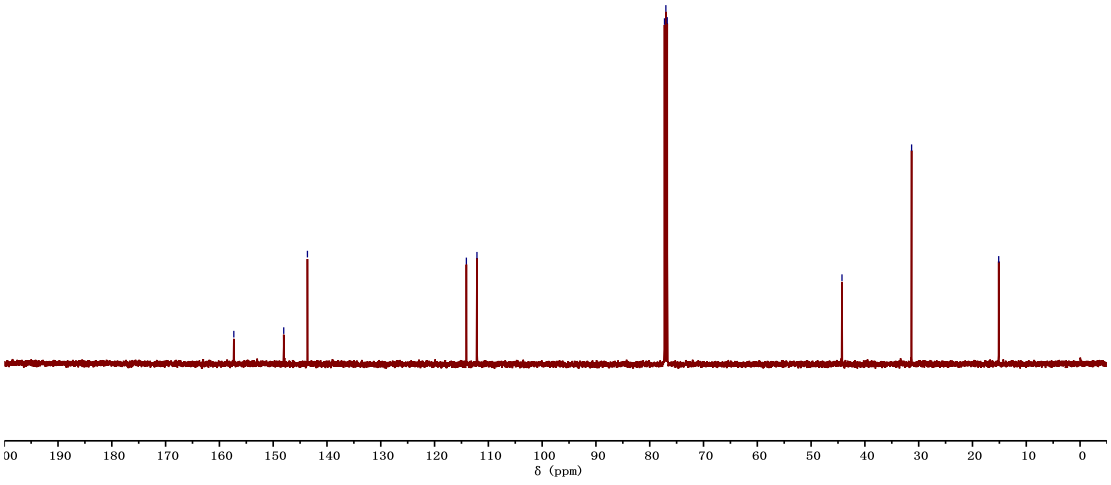
20l

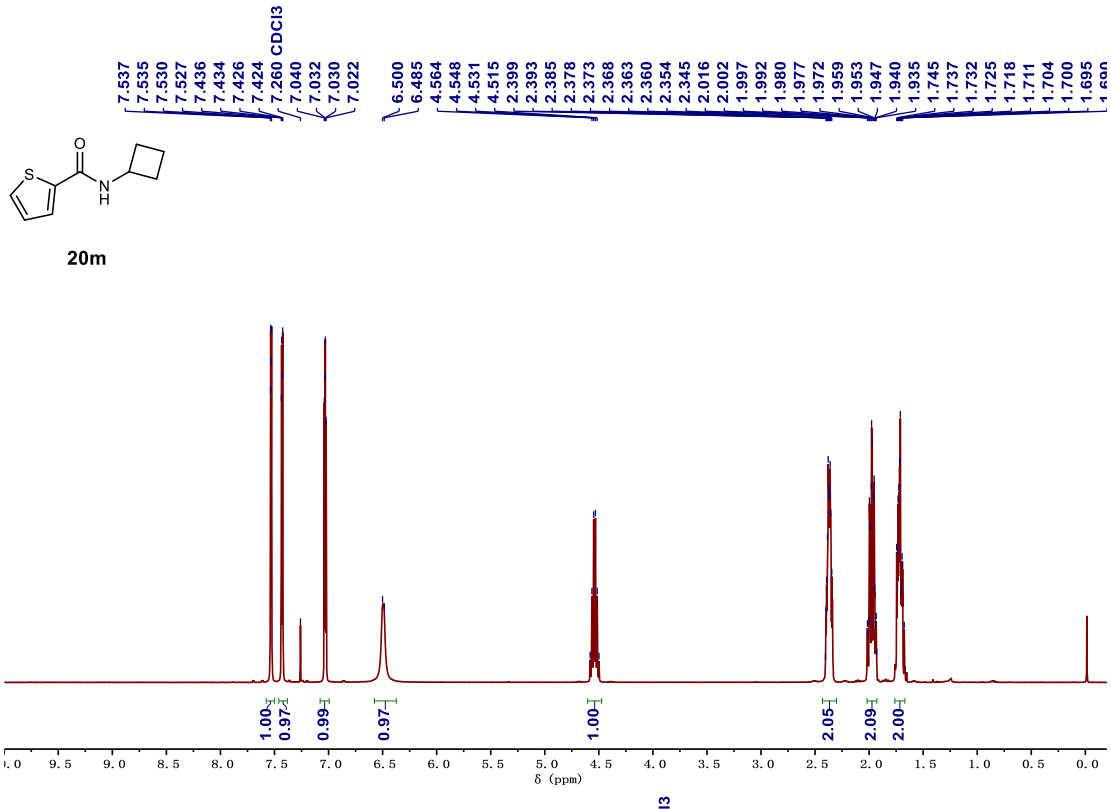

0.97
0.97
1.93
1.00
2.01
2.05
1.97
δ (ppm)

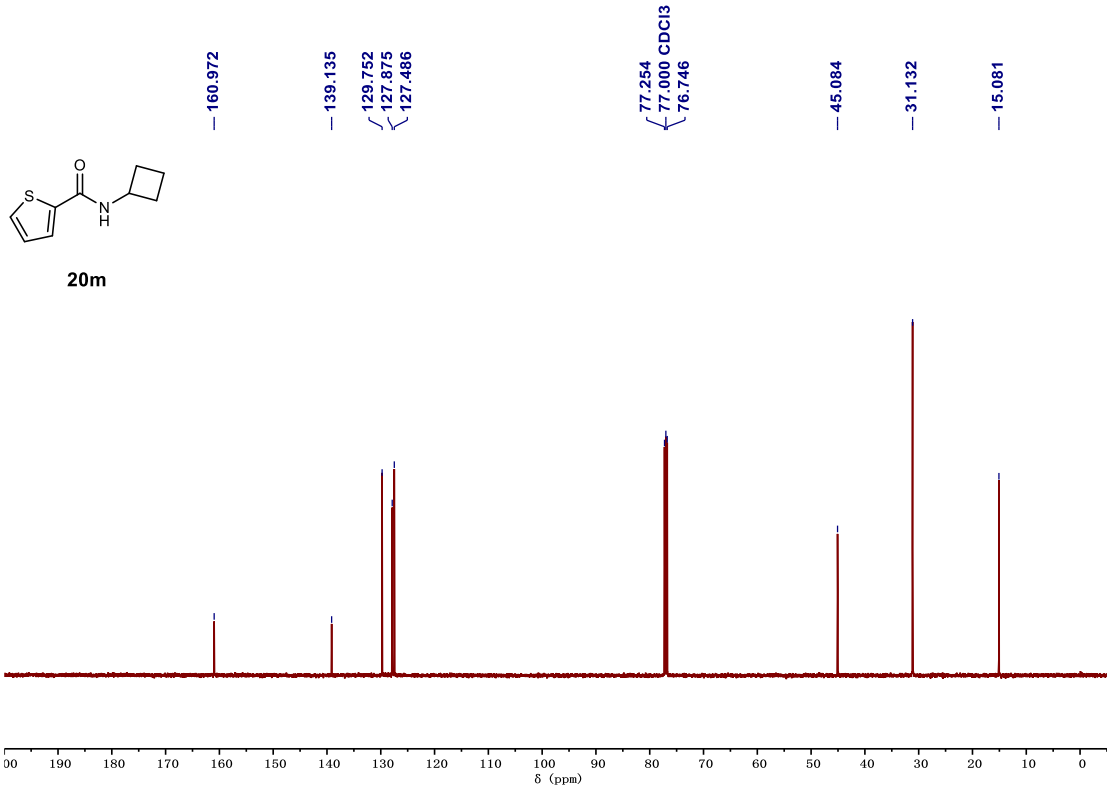

157.318
148.040
143.634
114.085
112.118
77.254
77.000 CDCl3
76.746
44.258
31.319
15.126

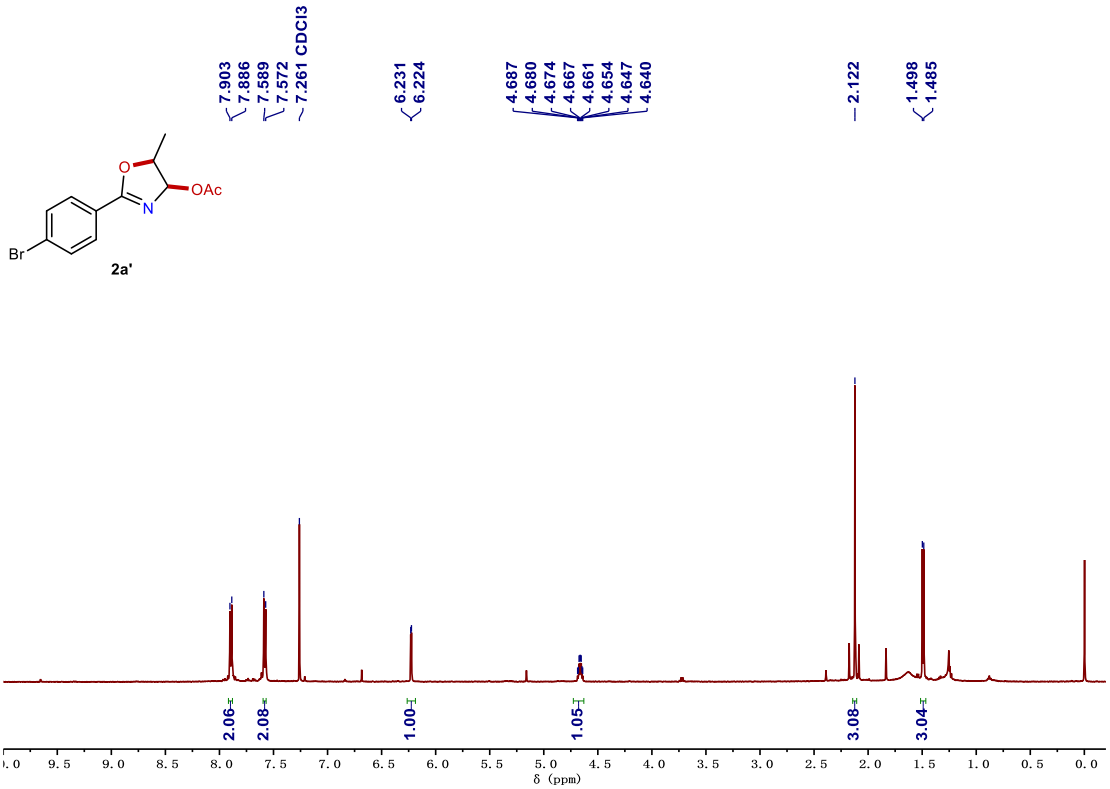


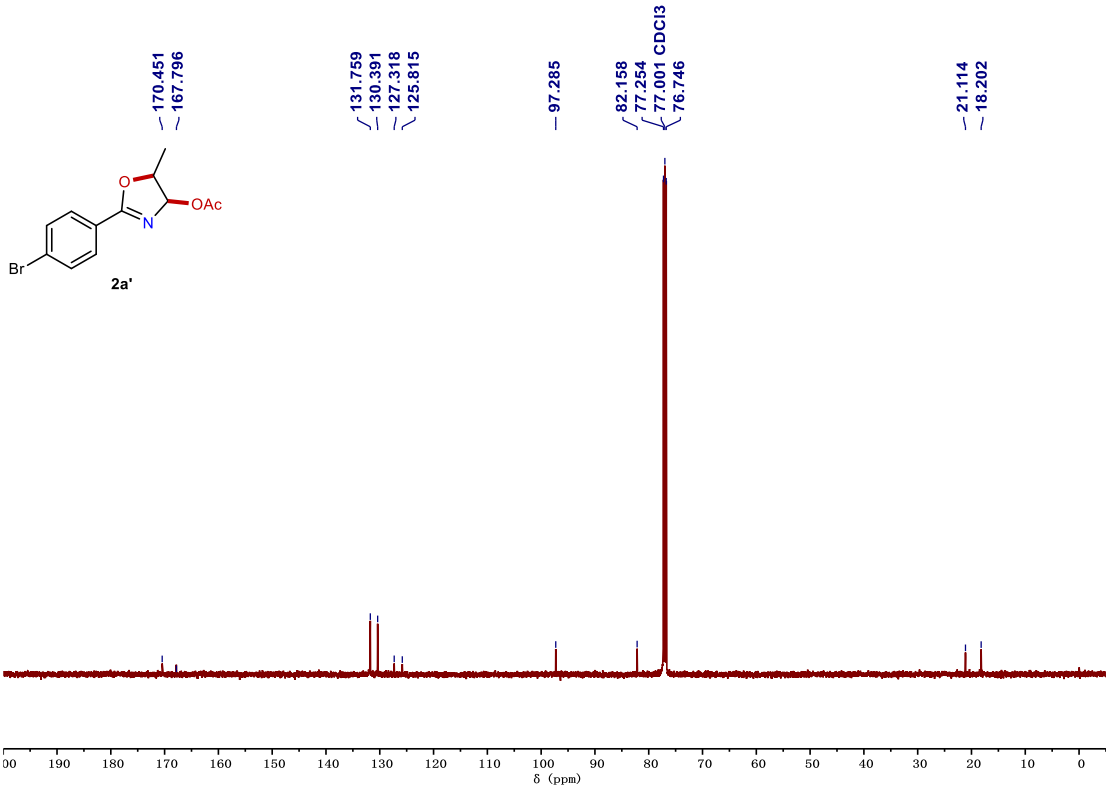
20l

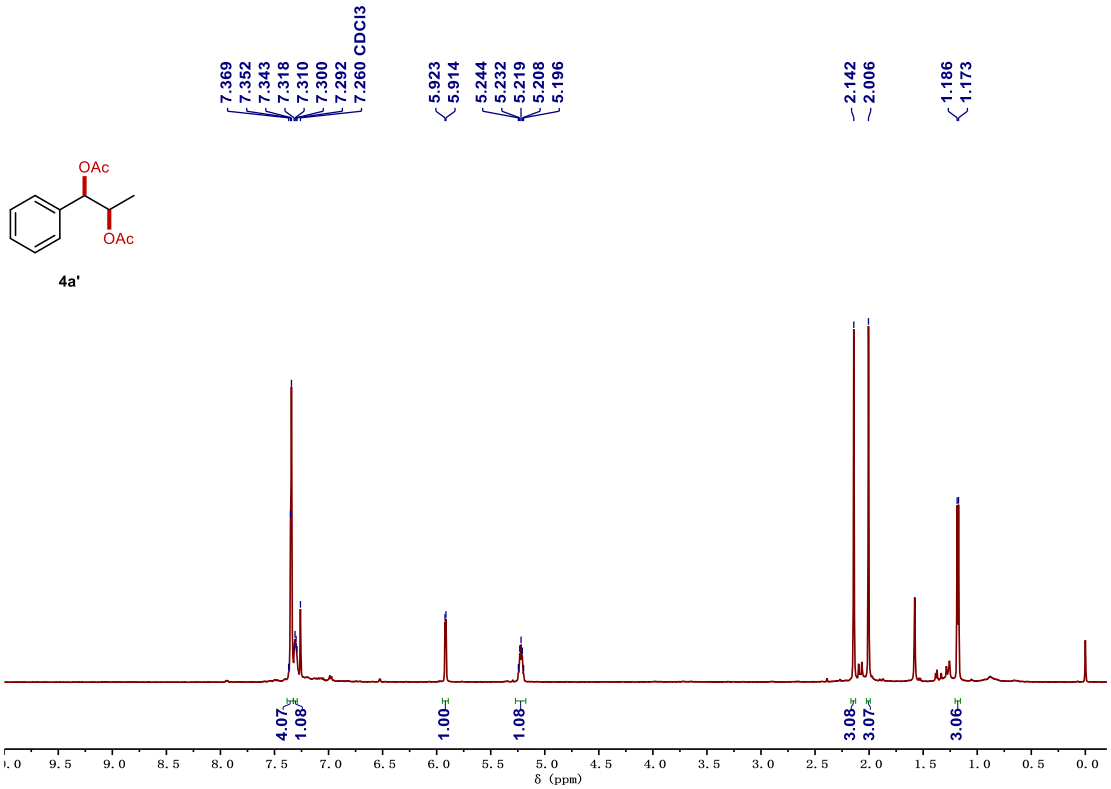

δ (ppm)

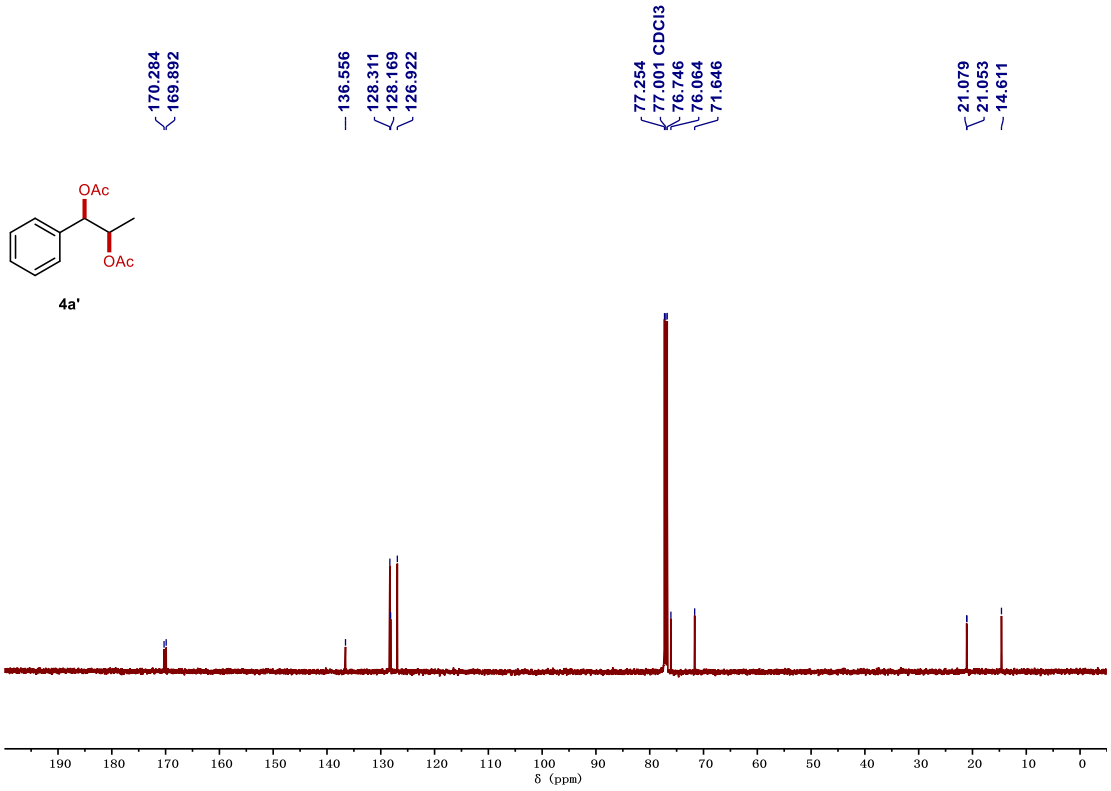

7.537
7.535
7.530
7.527
7.436
7.434
7.426
7.424
7.260 CDCl3
7.040
7.032
7.030
7.022
6.500
6.485
4.564
4.548
4.531
4.515
2.399
2.393
2.385
2.378
2.373
2.368
2.363
2.360
2.354
2.345
2.016
2.002
1.997
1.992
1.980
1.977
1.972
1.959
1.953
1.947
1.940
1.935
1.745
1.737
1.732
1.725
1.718
1.711
1.704
1.700
1.695
S
O
N
H
20m
1.00
0.97
0.99
0.97
1.00
2.05
2.09
2.00
δ (ppm)

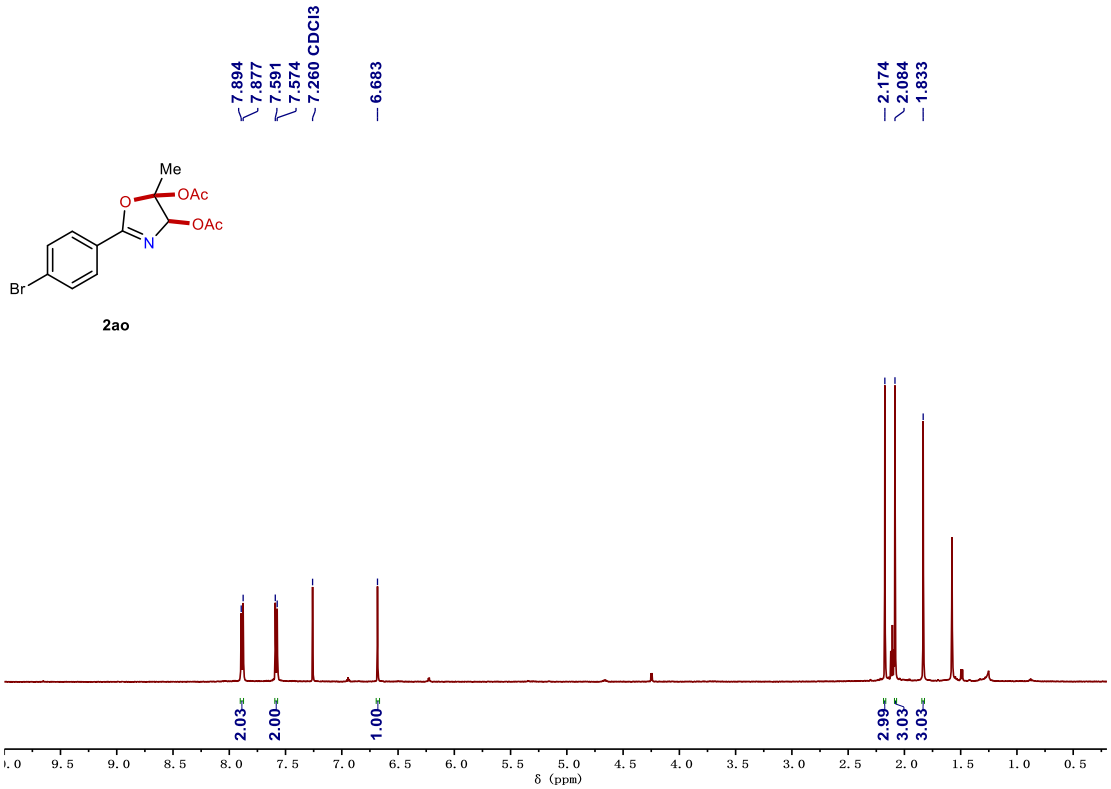

160.972
139.135
129.752
127.875
127.486
77.254
77.000 CDCl3
76.746
45.084
31.132
15.081
S
O
N
H
20m
δ (ppm)

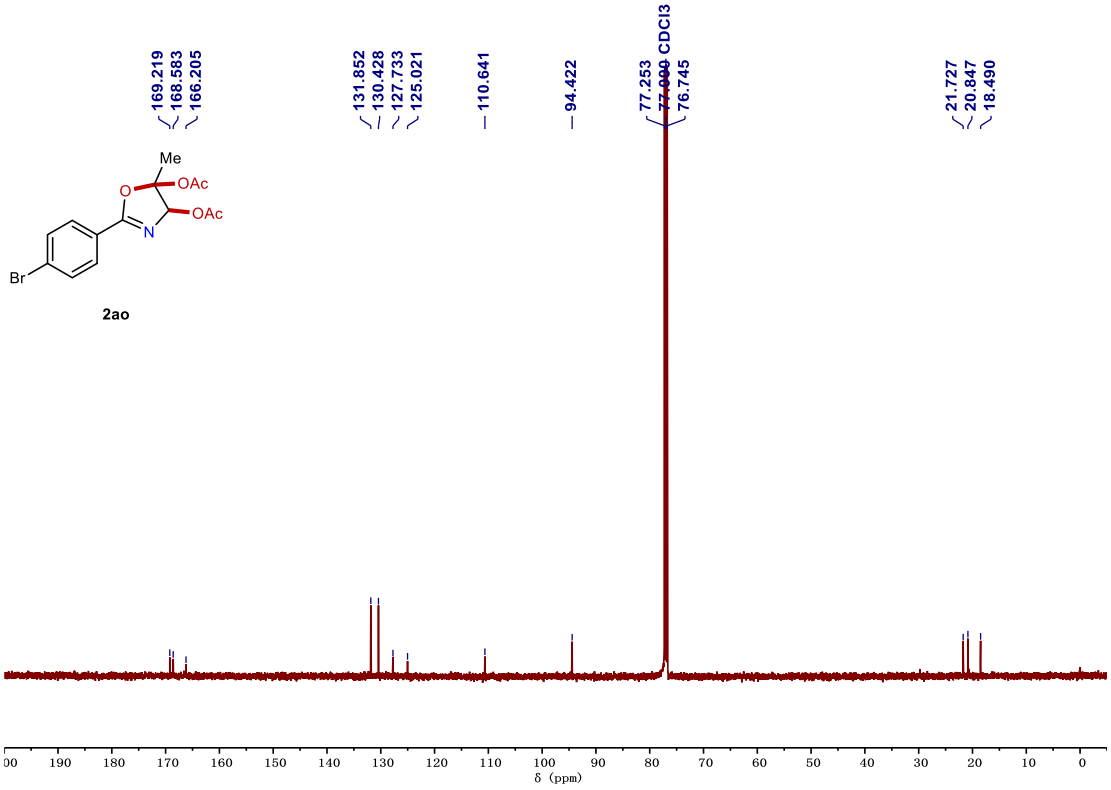

7.903
7.886
7.589
7.572
7.261 CDCl3
6.231
6.224
4.687
4.680
4.674
4.667
4.661
4.654
4.647
4.640
2.122
1.498
1.485
O
OAc
N
Br
2a'
2.06
2.08
1.00
1.05
3.08
3.04
δ (ppm)

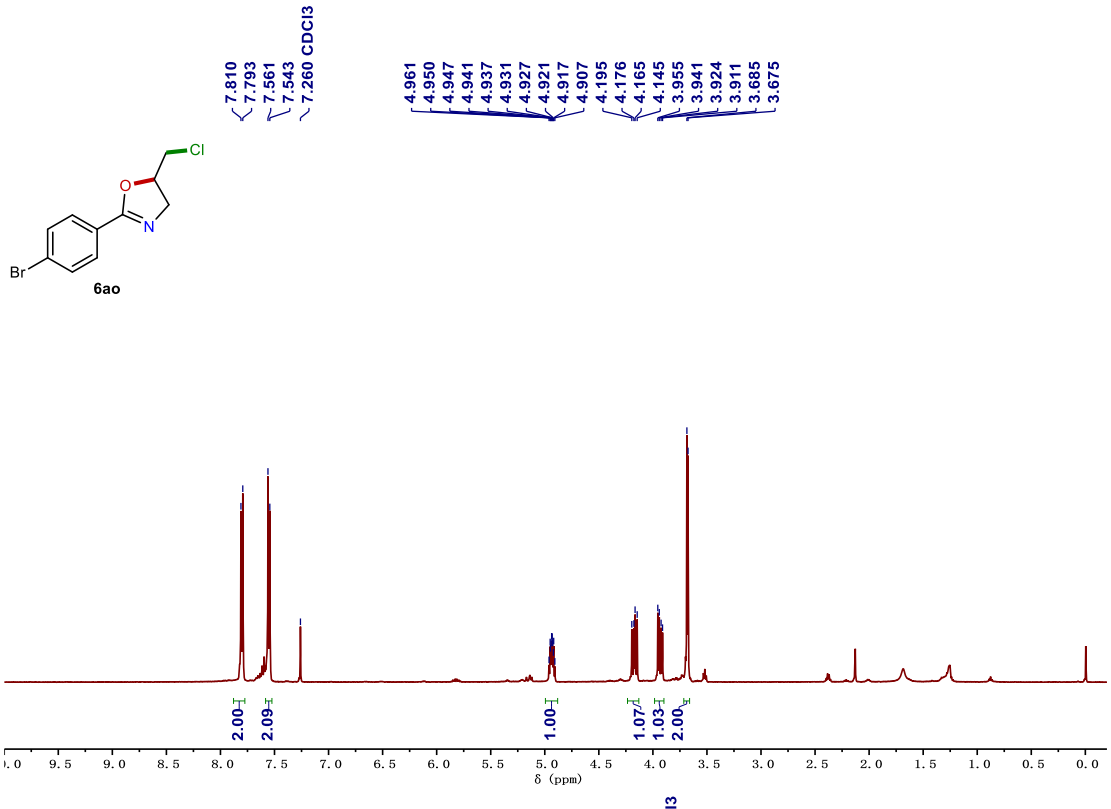

170.451
167.796
131.759
130.391
127.318
125.815
97.285
82.158
77.254
77.001 CDCl3
76.746
21.114
18.202
O
OAc
N
Br
2a'
δ (ppm)

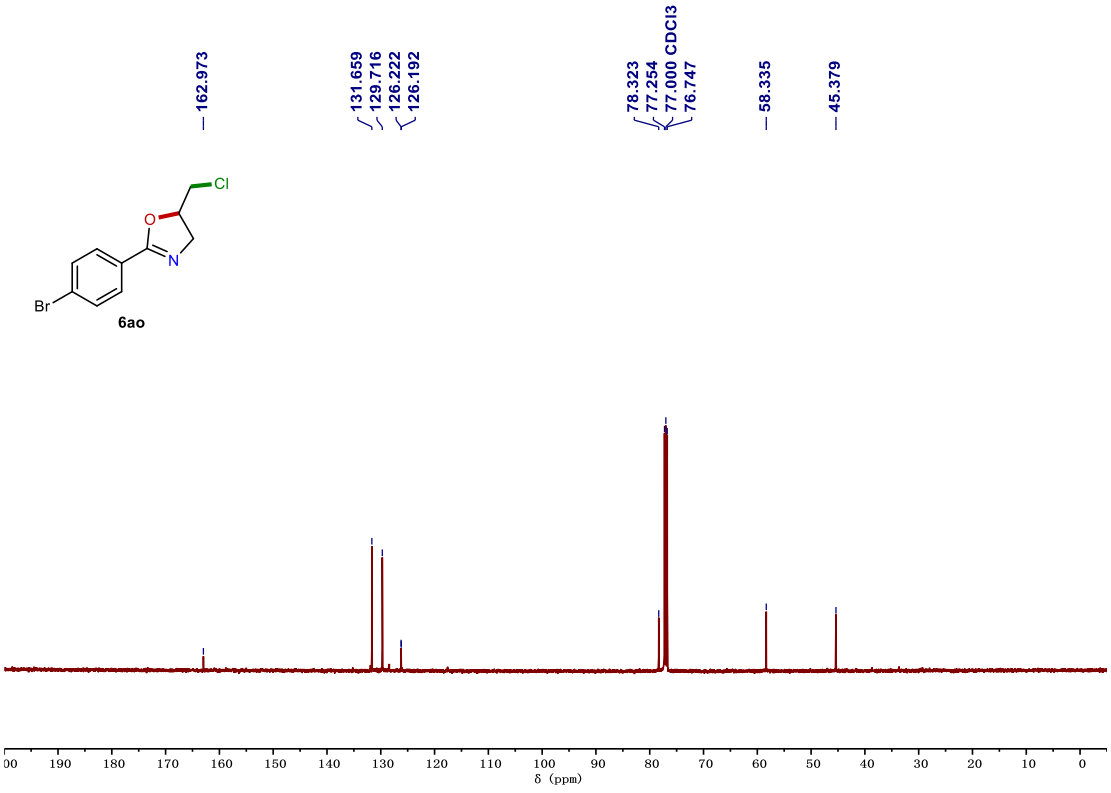

7.369
7.352
7.343
7.318
7.310
7.300
7.292
7.260 CDCl3
5.923
5.914
5.244
5.232
5.219
5.208
5.196
2.142
2.006
1.186
1.173
OAc
OAc
4a'
4.07
1.08
1.00
1.08
3.08
3.07
3.06
δ (ppm)

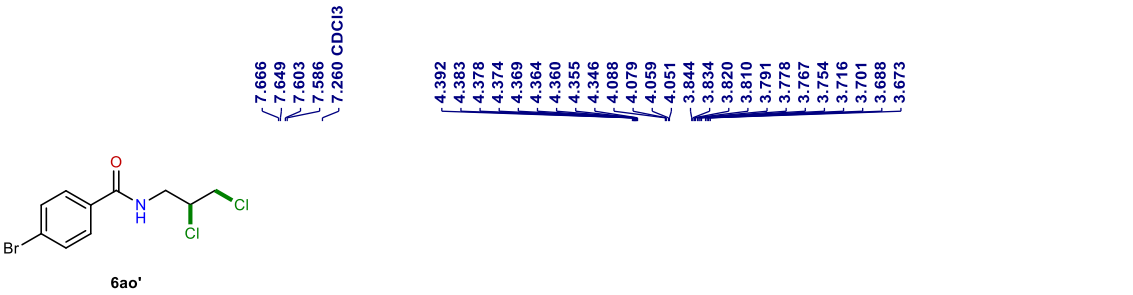

170.284
169.892
136.556
128.311
128.169
126.922
77.254
77.001 CDCl3
76.746
76.064
71.646
21.079
21.053
14.611
OAc
OAc
4a'
δ (ppm)


7.894
7.877
7.591
7.574
7.260 CDCl3
6.683
2.174
2.084
1.833
Me
OAc
OAc
O
N
Br
2ao
2.03
2.00
1.00
2.99
3.03
3.03
δ (ppm)

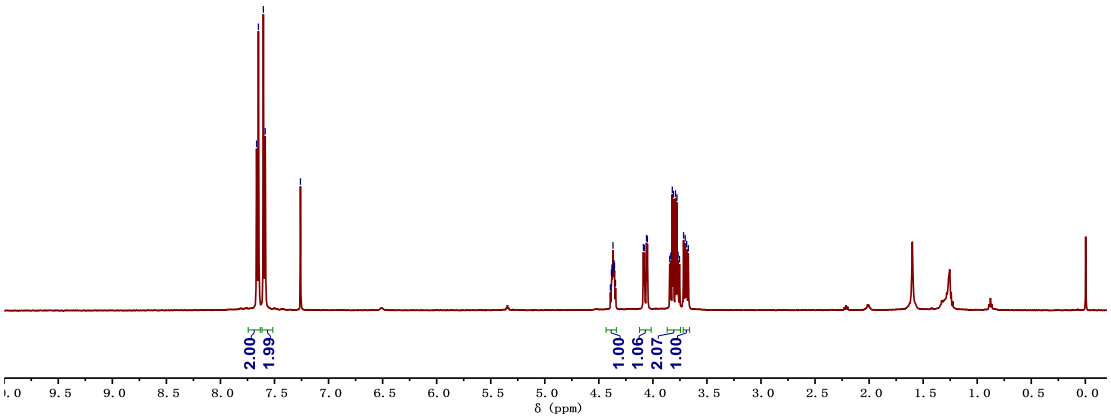

169.219
168.583
166.205
131.852
130.428
127.733
125.021
110.641
94.422
77.253
77.000 CDCl3
76.745
21.727
20.847
18.490
Me
OAc
OAc
O
N
Br
2ao
δ (ppm)

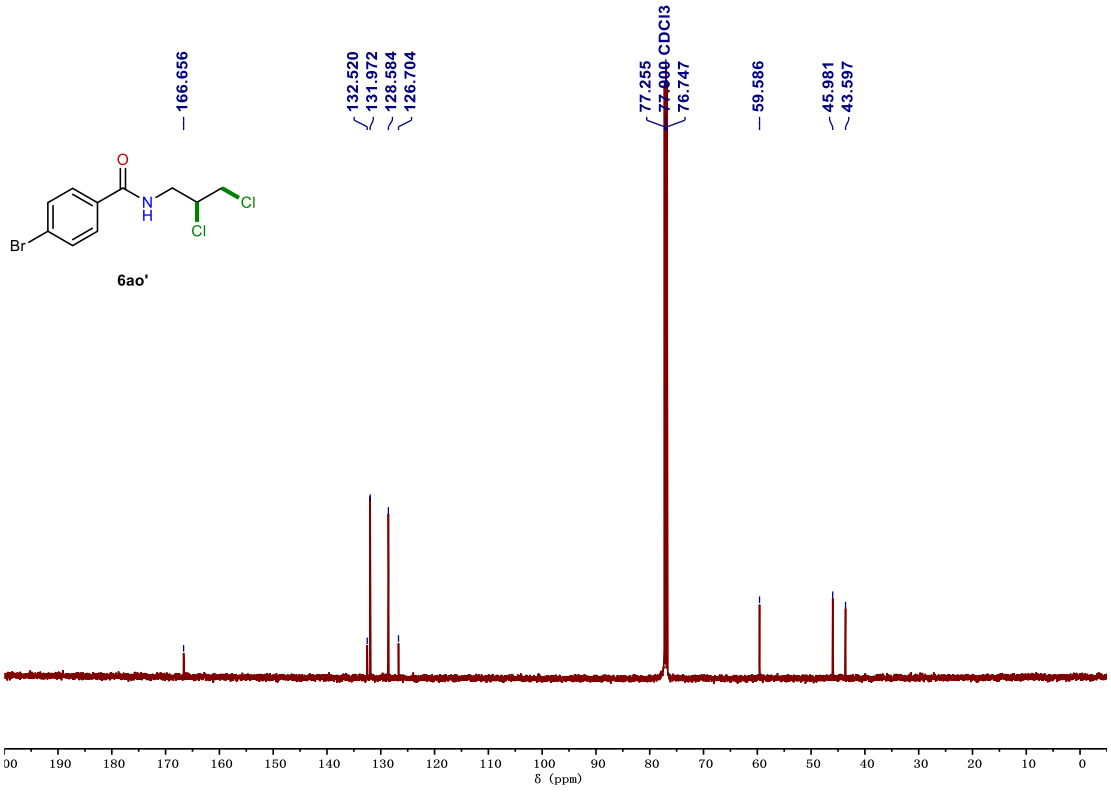

7.810
7.793
7.561
7.543
7.260 CDCl3
4.961
4.950
4.947
4.941
4.937
4.931
4.927
4.921
4.917
4.907
4.195
4.176
4.165
4.145
3.955
3.941
3.924
3.911
3.685
3.675
Cl
O
N
Br
6ao
2.00
2.09
1.00
1.07
1.03
2.00
δ (ppm)


162.973
131.659
129.716
126.222
126.192
78.323
77.254
77.000 CDCl3
76.747
58.335
45.379
Cl
O
N
Br
6ao
δ (ppm)


7.666
7.649
7.603
7.586
7.260 CDCl3
4.392
4.383
4.378
4.374
4.369
4.364
4.360
4.355
4.346
4.088
4.079
4.059
4.051
3.844
3.834
3.820
3.810
3.791
3.778
3.767
3.754
3.716
3.701
3.688
3.673
O
N
H
Cl
Cl
Br

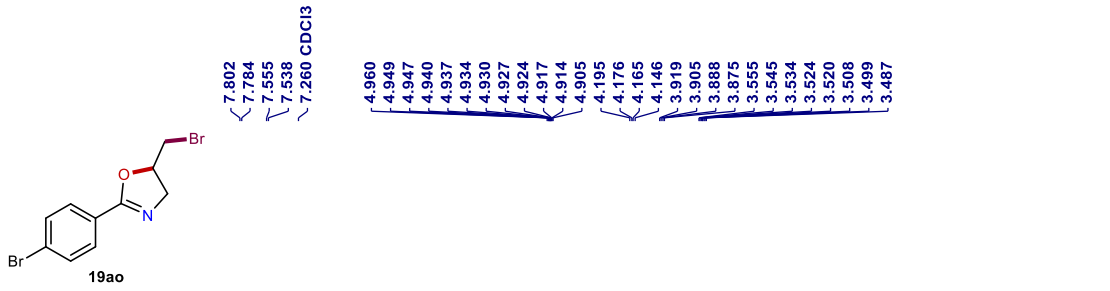
6ao'

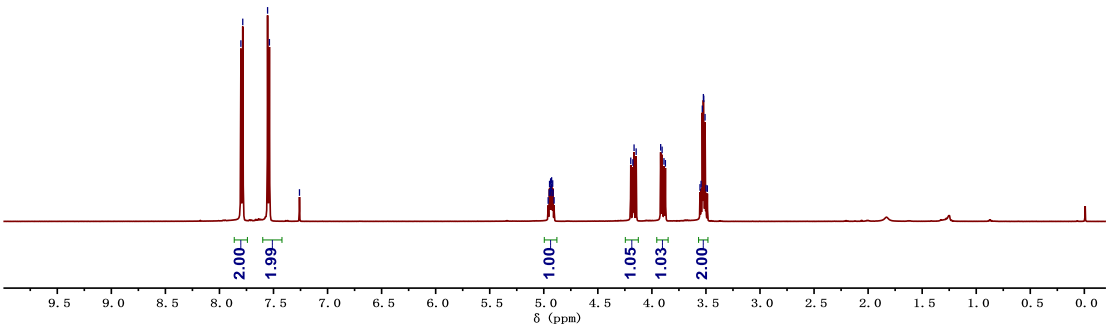

2.00
1.99
1.00
1.06
2.07
1.00
δ (ppm)

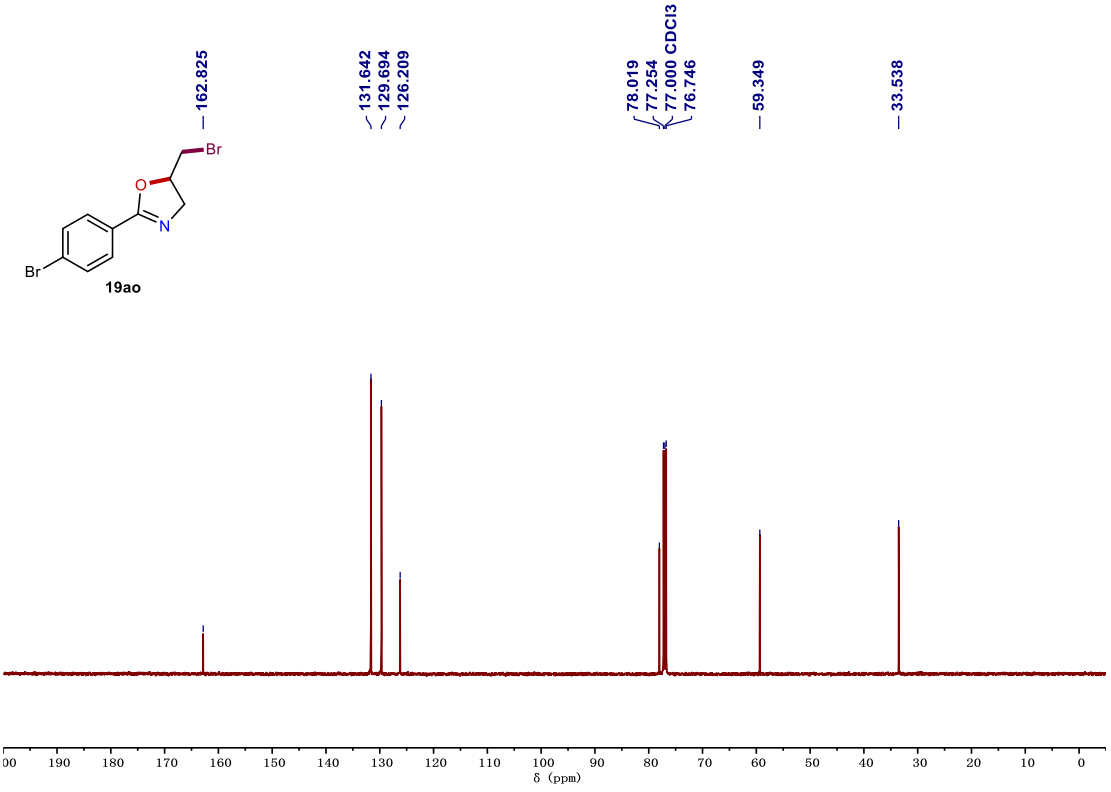

166.656
132.520
131.972
128.584
126.704
77.255
77.000 CDCl3
76.747
59.586
45.981
43.597
δ (ppm)

O
N
H
Cl
Cl
Br

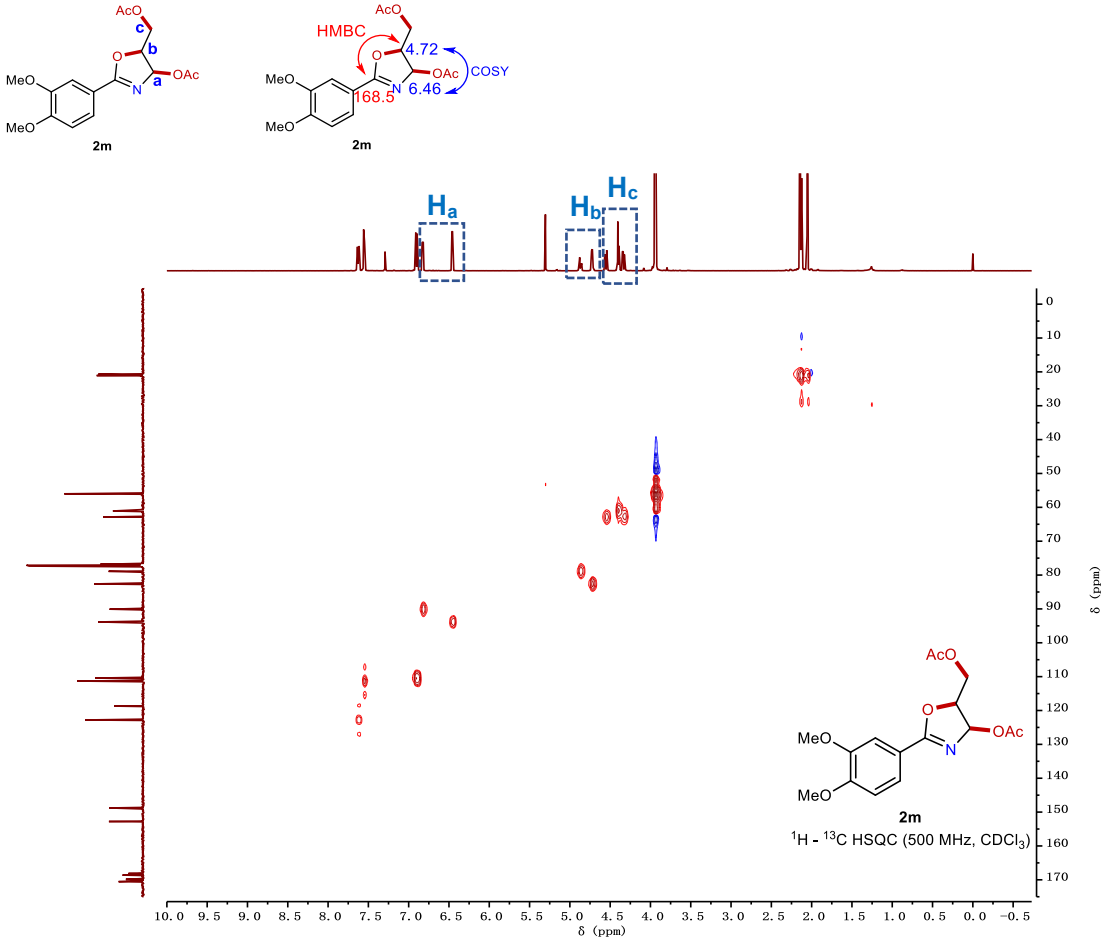
6ao'

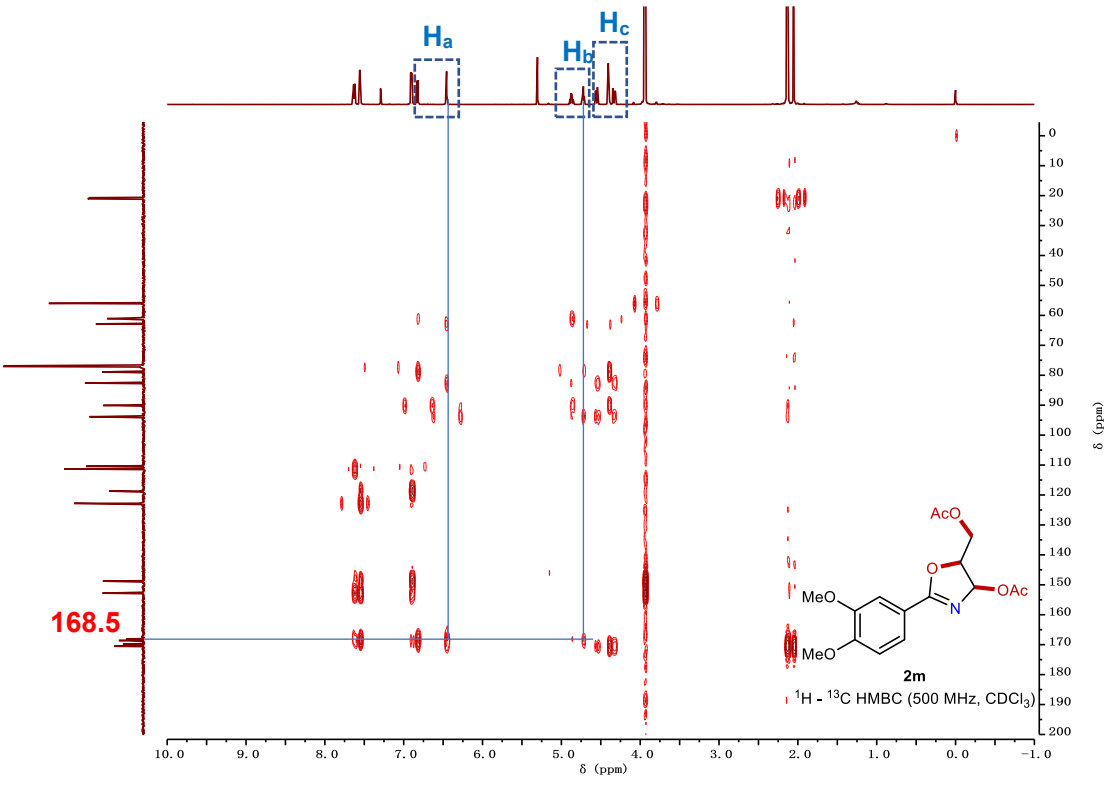

7.802
7.784
7.555
7.538
7.260 CDCl3
4.960
4.949
4.947
4.940
4.937
4.934
4.930
4.927
4.924
4.917
4.914
4.905
4.195
4.176
4.165
4.146
3.919
3.905
3.888
3.875
3.555
3.545
3.534
3.524
3.520
3.508
3.499
3.487
Br
O
N
Br
19ao


2.00
1.99
1.00
1.05
1.03
2.00
δ (ppm)


162.825
131.642
129.694
126.209
78.019
77.254
77.000 CDCl3
76.746
59.349
33.538
Br
O
N
Br
19ao
δ (ppm)

4. 2D NMR


AcO
c
b
a
OAc
MeO
MeO
2m
HMBC
4.72
COSY
6.46
168.5
Ha
Hb
Hc
δ (ppm)
2m
1H - 13C HSQC (500 MHz, CDCl3)


Ha
Hb
Hc
168.5
δ (ppm)
2m
1H - 13C HMBC (500 MHz, CDCl3)

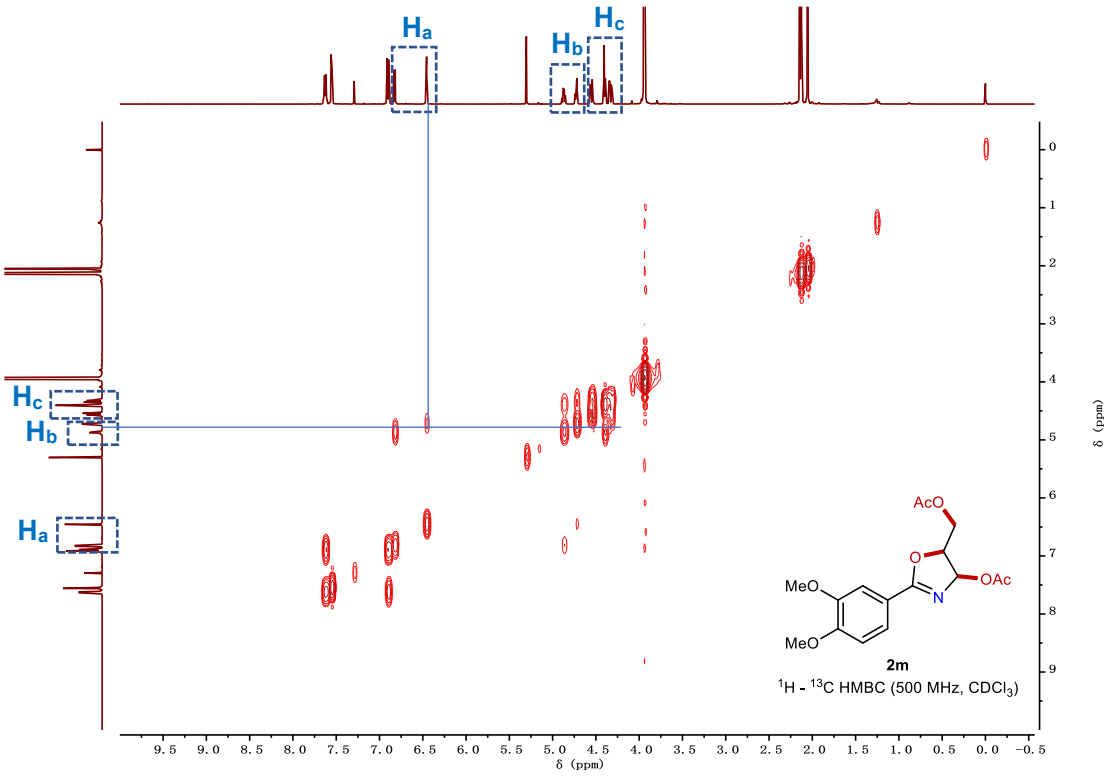
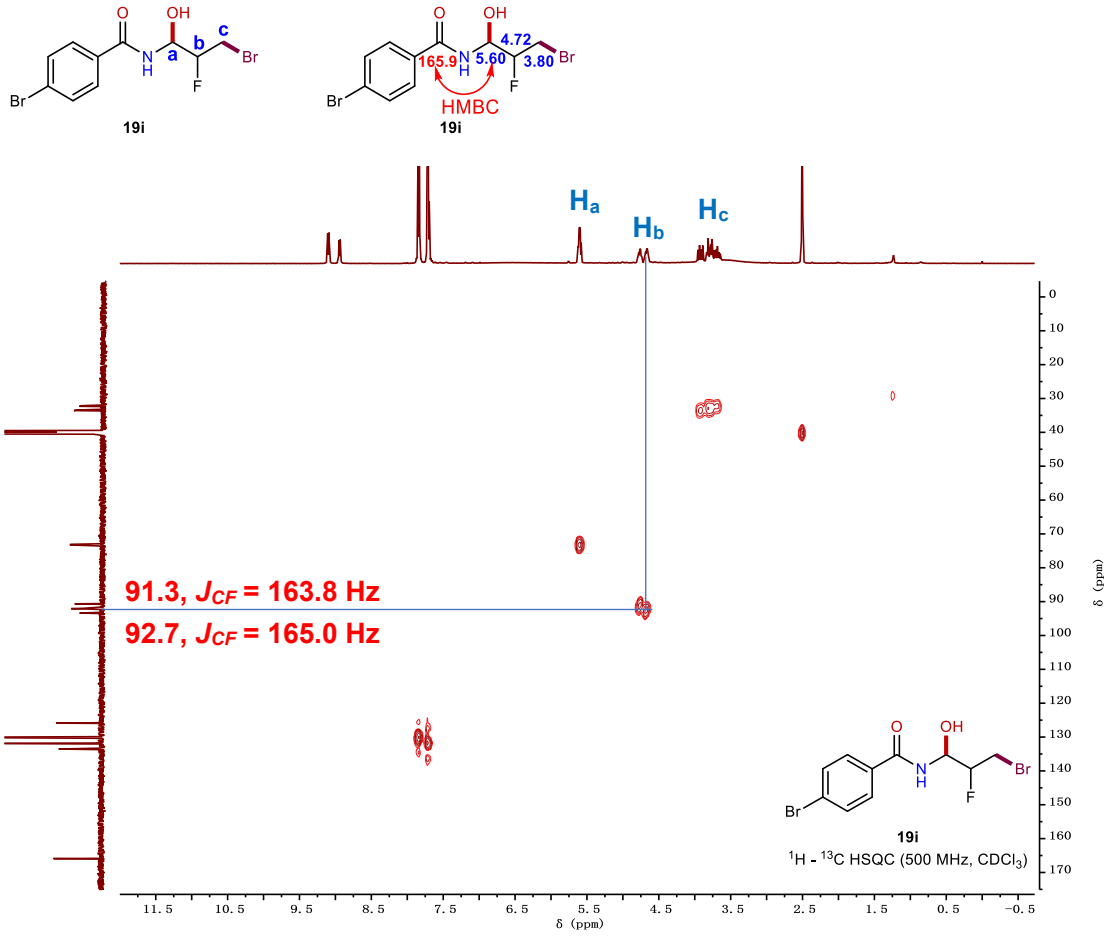
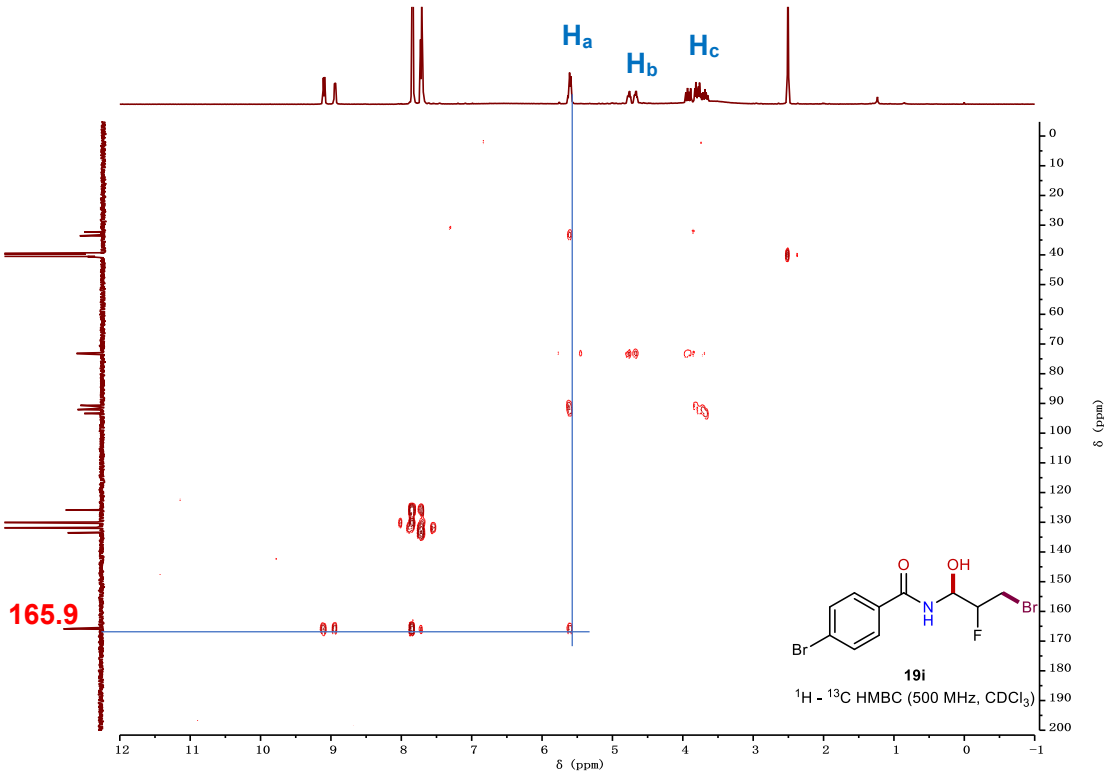


Figure S35. 2D NMR of **2m**


19i
165.9
5.60
4.72
3.80
HMBC
19i
Ha
Hb
Hc
91.3, JCF = 163.8 Hz
92.7, JCF = 165.0 Hz
19i
1H - 13C HSQC (500 MHz, CDCl3)
δ (ppm)


Ha
Hb
Hc
165.9
19i
1H - 13C HMBC (500 MHz, CDCl3)
δ (ppm)

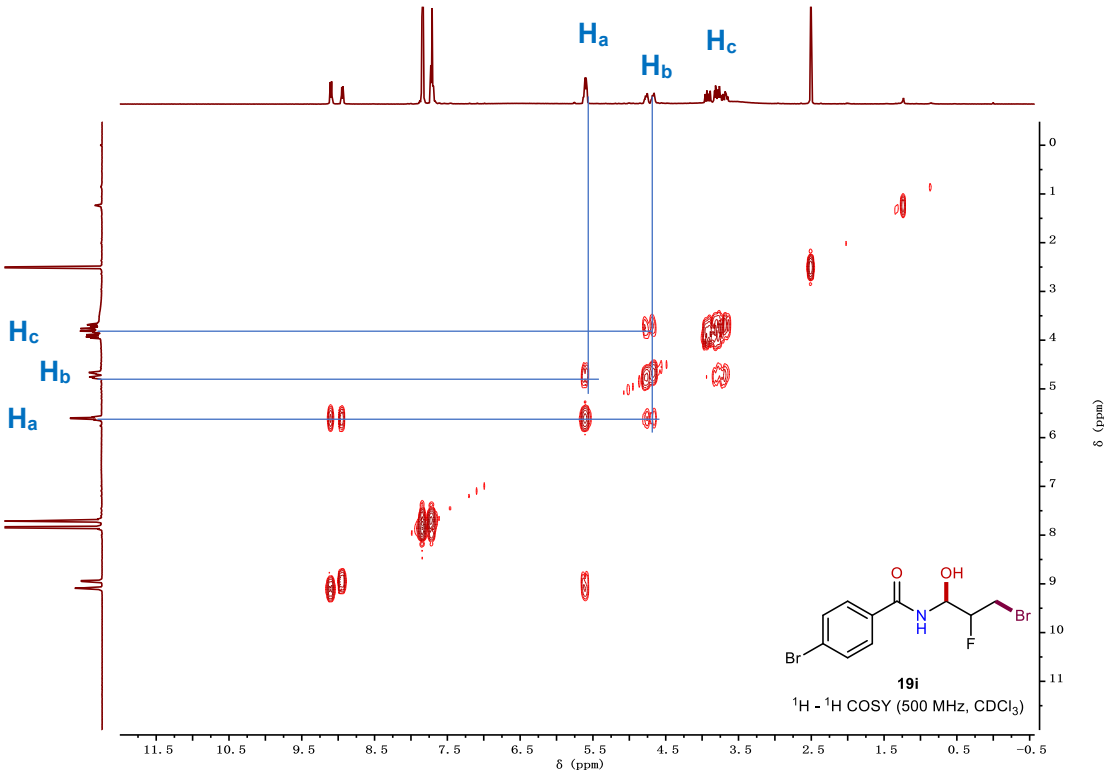
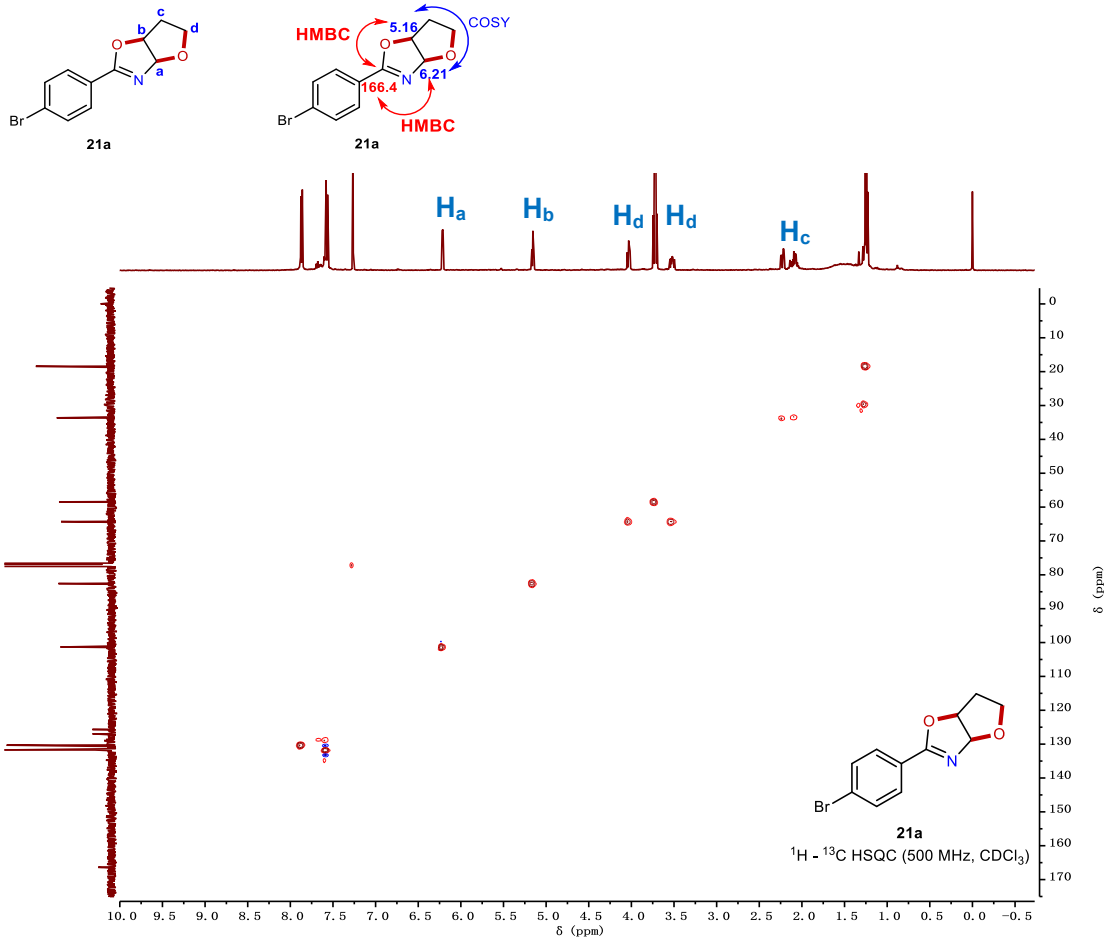
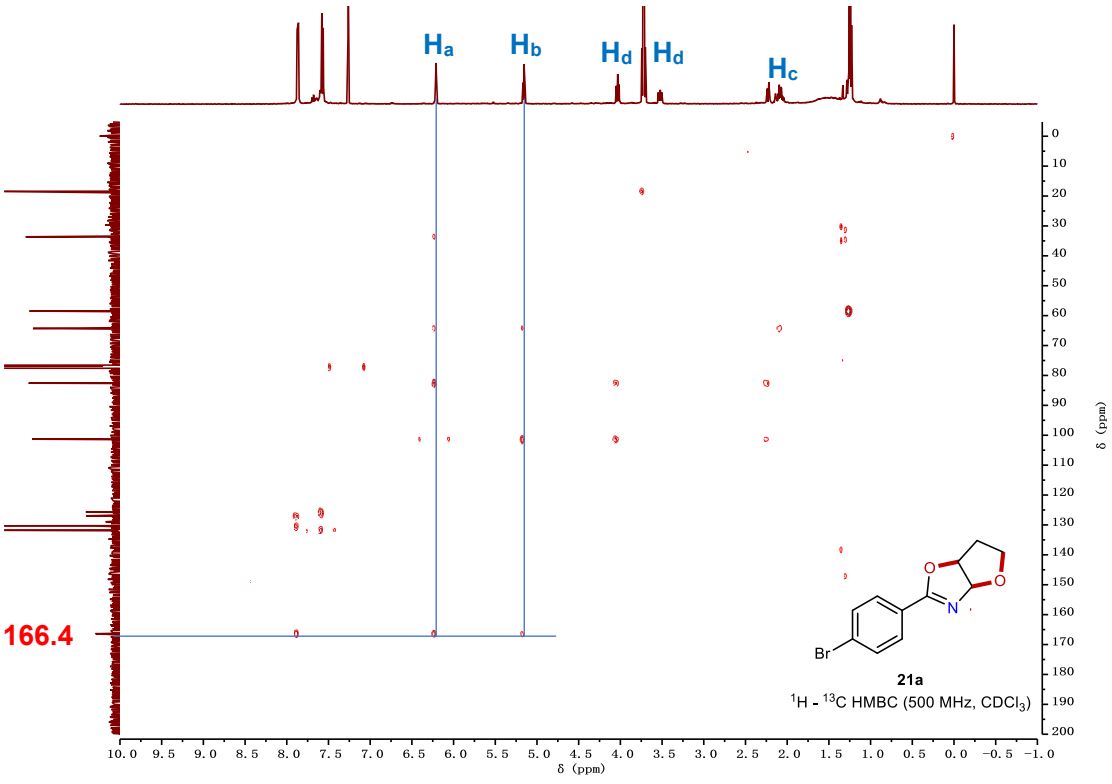


Figure S36. 2D NMR of **19i**


21a
HMBC
5.16
COSY
6.21
166.4
HMBC
21a
Ha
Hb
Hd
Hd
Hc
δ (ppm)
21a
1H - 13C HSQC (500 MHz, CDCl3)


Ha
Hb
Hd
Hd
Hc
166.4
δ (ppm)
21a
1H - 13C HMBC (500 MHz, CDCl3)

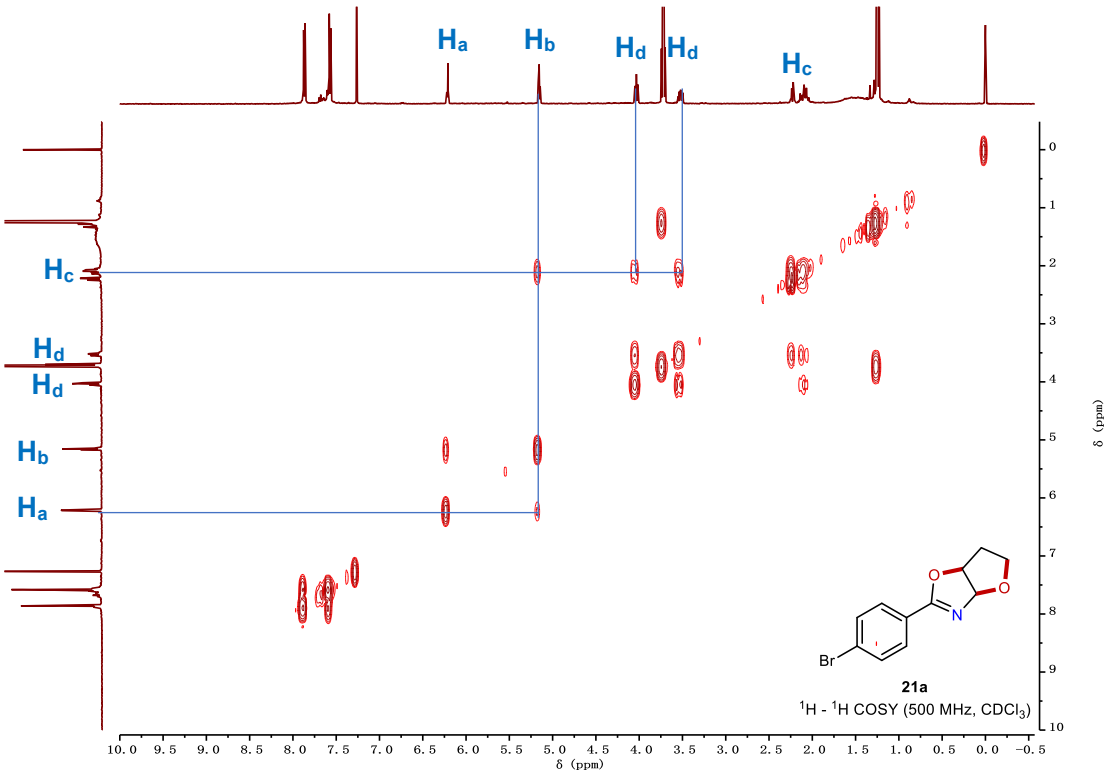
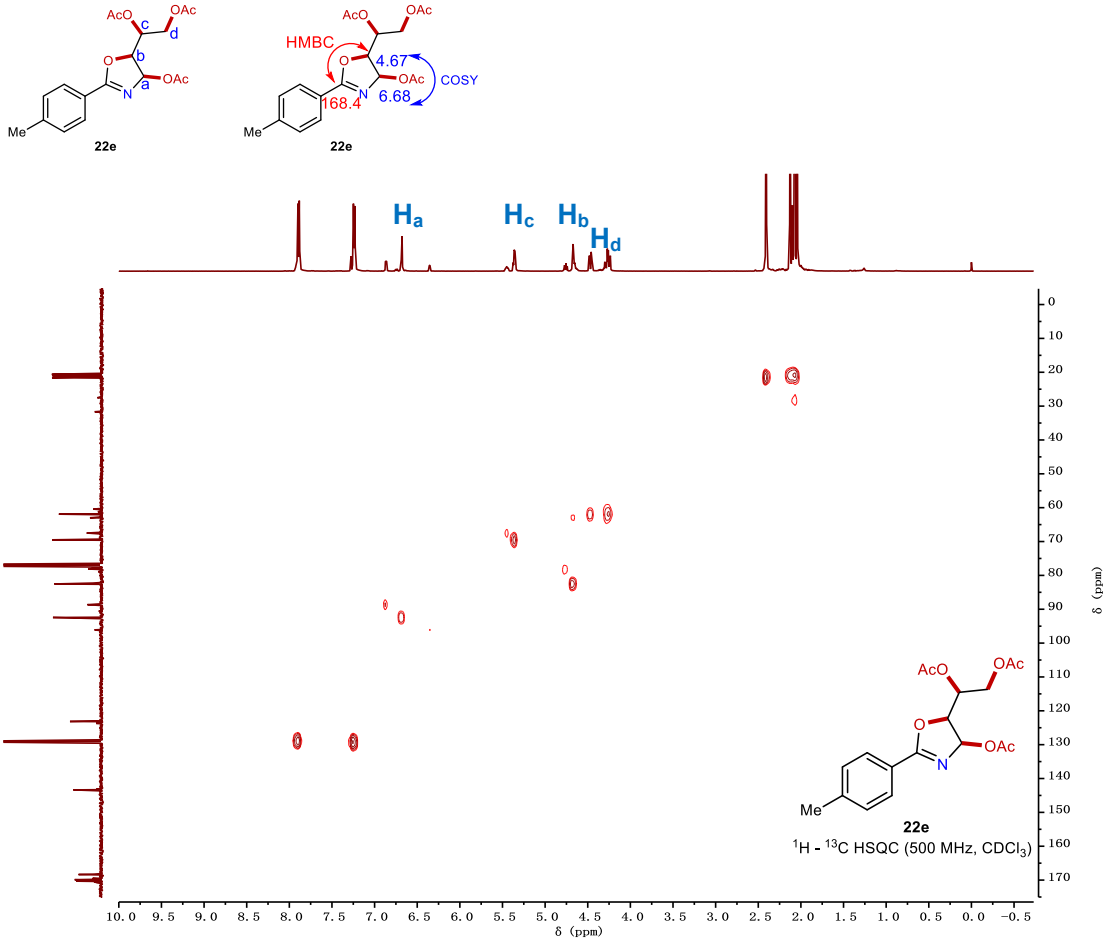
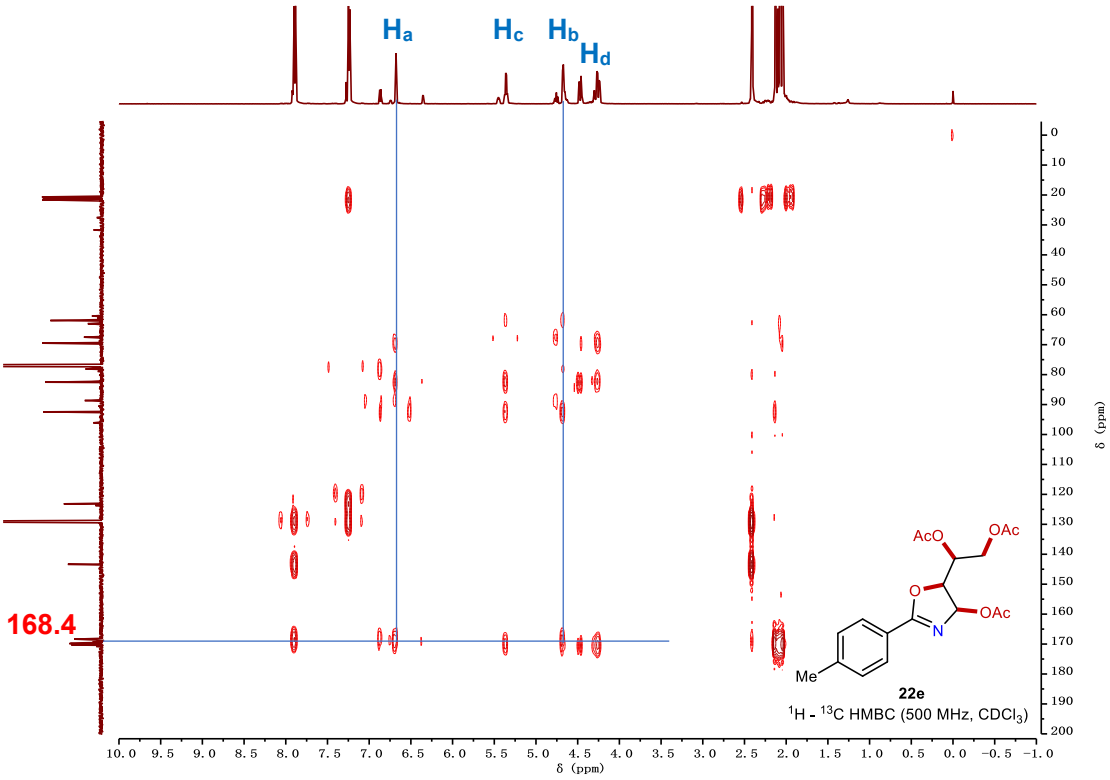


Figure S37. 2D NMR of **21a**


AcO
OAc
c
d
b
a
OAc
O
N
Me
22e
HMBC
4.67
COSY
6.68
168.4
Ha
Hc
Hb
Hd
δ (ppm)
22e
1H - 13C HSQC (500 MHz, CDCl3)


Ha
Hc
Hb
Hd
168.4
δ (ppm)
22e
1H - 13C HMBC (500 MHz, CDCl3)

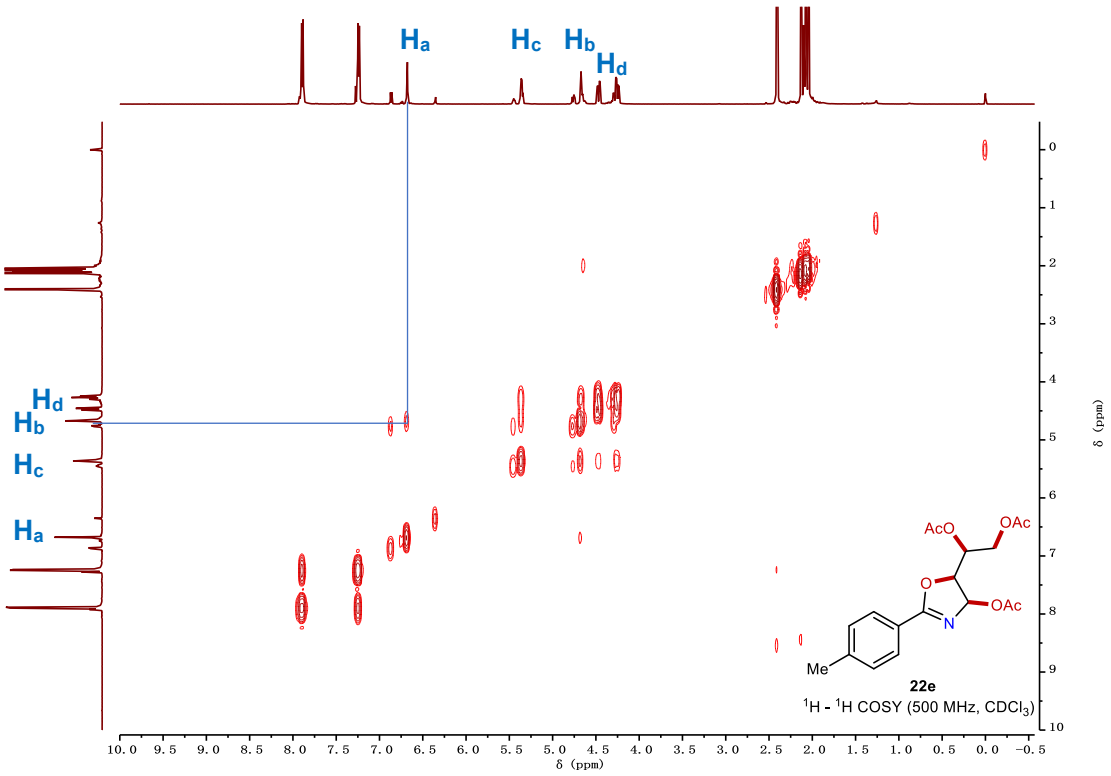


Figure S38. 2D NMR of **22e**

5. NOESY Analysis of Compound 2g

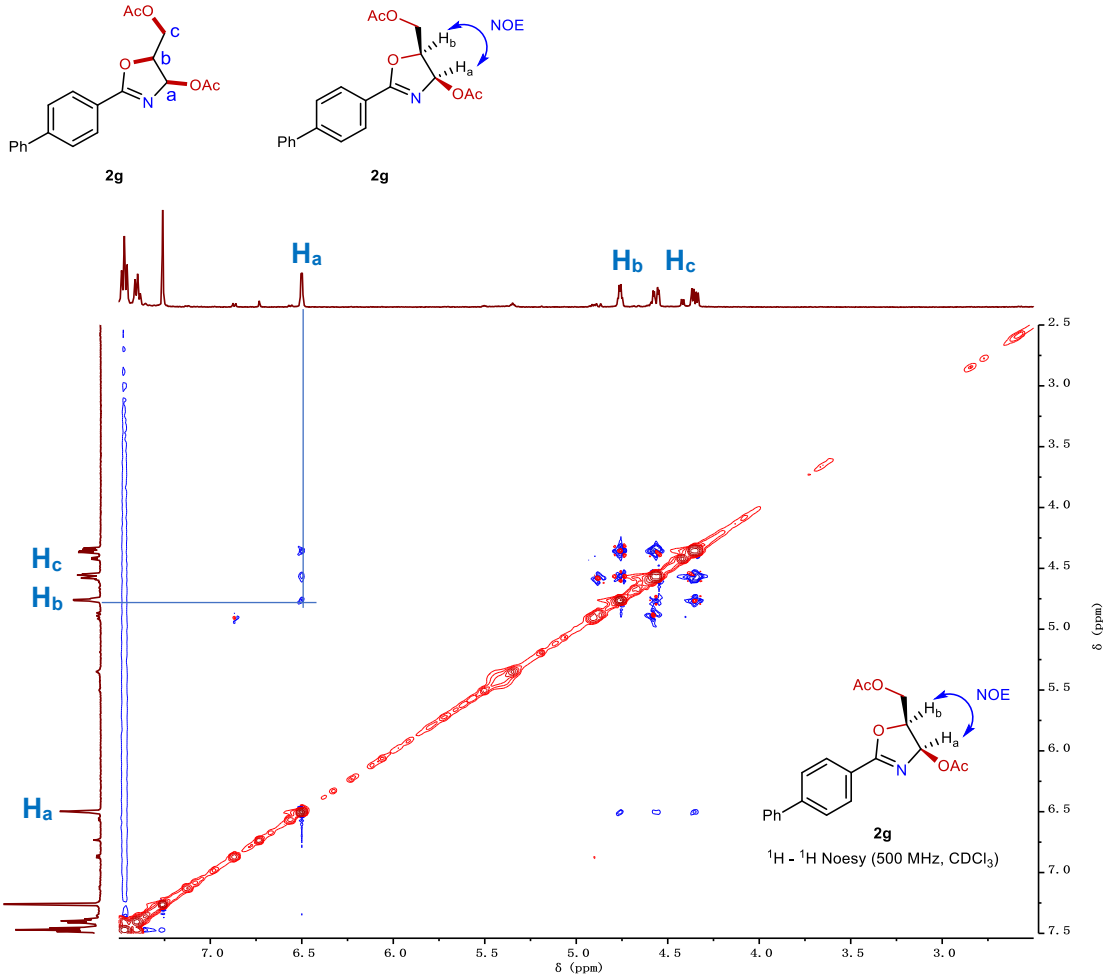


Figure S39. NOE analysis of **2g**

6. X–ray Data Analysis

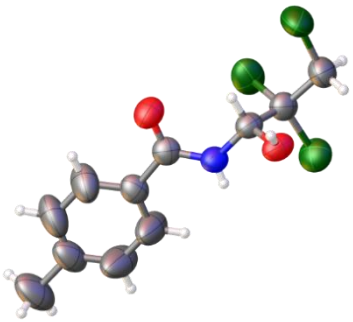


Figure 40. The X-ray structure of **6e** (CCDC: 2442144, 50% probability thermal ellipsoids)

Table S19 Crystal data and structure refinement for t.

Identification code	t
Empirical formula	$C_{11}H_{12}Cl_3O_2N$
Formula weight	296.57
Temperature/K	298(2)
Crystal system	triclinic
Space group	P–1
a/Å	6.8567(3)
b/Å	10.4133(4)
c/Å	11.2353(4)
α/°	116.575(4)
β/°	98.166(3)
γ/°	99.590(3)
Volume/Å^3	685.76(5)
Z	2
ρ_{calc}g/cm^3	1.436
μ/mm^{-1}	5.978
F(000)	304.0
Crystal size/mm^3	0.15 × 0.14 × 0.12
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.078 to 155.008
Index ranges	–8 ≤ h ≤ 7, –12 ≤ k ≤ 13, –13 ≤ l ≤ 14
Reflections collected	8303
Independent reflections	2756 [R_{int} = 0.0217, R_{sigma} = 0.0168]
Data/restraints/parameters	2756/0/160
Goodness–of–fit on F^2	1.083
Final R indexes [I>=2σ (I)]	R_1 = 0.0516, wR_2 = 0.1511
Final R indexes [all data]	R_1 = 0.0551, wR_2 = 0.1543
Largest diff. peak/hole / e Å^{-3}	0.35/–0.34

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) t

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: t

```
Bond precision:   C-C = 0.0063 A           Wavelength=1.54184

Cell:           a=6.8567(3)         b=10.4133(4)      c=11.2353(4)
                alpha=116.575(4)    beta=98.166(3)    gamma=99.590(3)
Temperature:    298 K
```

	Calculated	Reported
Volume	685.77(6)	685.76(5)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C11 H12 Cl3 N O2	C11 H12 Cl3 N O2
Sum formula	C11 H12 Cl3 N O2	C11 H12 Cl3 N O2
Mr	296.57	296.57
Dx,g cm-3	1.436	1.436
Z	2	2
Mu (mm-1)	5.978	5.978
F000	304.0	304.0
F000'	306.85	
h,k,lmax	8,13,14	8,13,14
Nref	2920	2756
Tmin,Tmax	0.469,0.488	0.396,1.000
Tmin'	0.355	

```
Correction method= # Reported T Limits: Tmin=0.396 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.944            Theta(max)= 77.504

R(reflections)= 0.0516( 2460)                    wR2(reflections)=
                                                 0.1543( 2756)
S = 1.083                  Npar= 160
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of         C2 Check
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of         C3 Check
PLAT241_ALERT_2_C High   'MainMol' Ueq as Compared to Neighbors of         C5 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C1 Check
PLAT242_ALERT_2_C Low    'MainMol' Ueq as Compared to Neighbors of         C4 Check
PLAT334_ALERT_2_C Small <C-C> Benzene Dist.   C1       -C6       .       1.37 Ang.
PLAT340_ALERT_3_C Low Bond Precision on  C-C Bonds ..............     0.0063 Ang.
PLAT352_ALERT_3_C Short  N-H (X0.87,N1.01A)  N1       - H4       .       0.73 Ang.
PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ......      2.921 Check
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600         25 Report
               -2 -8  1,  -1  1  1,   0  2  1,  -2 -8  2,  -8  0  3,  -8  1  3,
                5-10  4,  -8  0  4,  -8  1  4,  -8  2  4,   0  6  4,  -7 -1  5,
               -7  0  5,  -7 -2  6,  -7 -1  6,  -7 -4  7,  -7 -3  7,  -7 -2  7,
               -7 -1  7,  -7  0  7,  -7 -3  8,  -7 -2  8,  -7 -1  8,  -7  0  8,
                3 -9 11,
```

Alert level G

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PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          1 Report
            H1
PLAT434_ALERT_2_G Short Inter HL..HL Contact Cl1      ..Cl3      .       3.39 Ang.
                                                  -1+x,y,z  =      1_455 Check
PLAT434_ALERT_2_G Short Inter HL..HL Contact Cl2      ..Cl2      .       3.30 Ang.
                                              1-x,2-y,2-z  =      2_677 Check
PLAT793_ALERT_4_G Model has Chirality at C9          (Centro SpGr)          S Verify
PLAT883_ALERT_1_G Absent Datum for _atom_sites_solution_primary ..     Please Do !
PLAT899_ALERT_4_G SHELXL2018 is Outdated   and Succeeded by SHELXL     2019/3 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        139 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........        3.0 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........      7.728 Note
             Predicted wR2: Based on SigI**2  2.00 or SHELX Weight 14.24
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          3 Info
PLAT992_ALERT_5_G Repd & Actual _reflns_number_gt Values Differ by          3 Check
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
  10 ALERT level C = Check. Ensure it is not caused by an omission or oversight
  11 ALERT level G = General information/check it is not something unexpected

   1 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   9 ALERT type 2 Indicator that the structure model may be wrong or deficient
   5 ALERT type 3 Indicator that the structure quality may be low
   3 ALERT type 4 Improvement, methodology, query or suggestion
   3 ALERT type 5 Informative message, check
```

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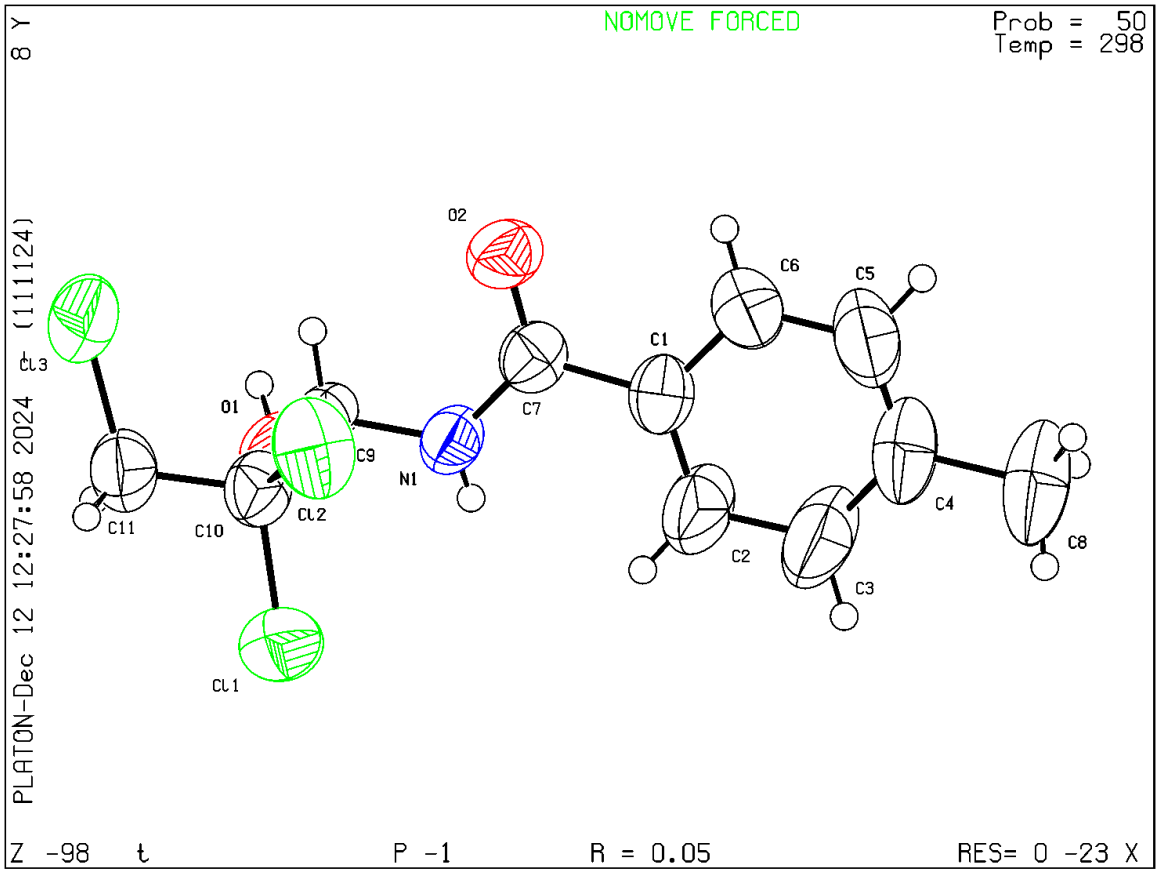
PLATON version of 11/11/2024; check.def file version of 11/11/2024

Datablock t - ellipsoid plot



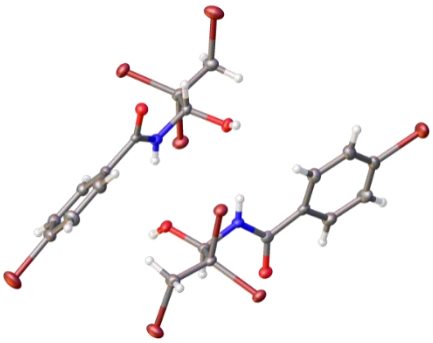


Figure 41. The X-ray structure of **19a** (CCDC: 2446154, 50% probability thermal ellipsoids)

Table S20 Crystal data and structure refinement for 20250422LYJ-31837-1_auto.

Identification code	20250422LYJ-31837-1_auto
Empirical formula	$C_{10}H_9Br_4NO_2$
Formula weight	494.82
Temperature/K	100.00(10)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	13.62980(10)
b/Å	25.7396(2)
c/Å	8.33400(10)
α/°	90
β/°	95.7650(10)
γ/°	90
Volume/Å^3	2908.99(5)
Z	8
ρ_{calc}g/cm^3	2.260
μ/mm^{-1}	13.444
F(000)	1856.0
Crystal size/mm^3	0.14 × 0.12 × 0.1
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	6.518 to 151.686
Index ranges	$-17 \le h \le 16$, $-31 \le k \le 31$, $-6 \le l \le 10$
Reflections collected	21226
Independent reflections	5742 [R_{int} = 0.0292, R_{sigma} = 0.0199]
Data/restraints/parameters	5742/0/315
Goodness–of–fit on F^2	1.049
Final R indexes [I>=2σ (I)]	R_1 = 0.0313, wR_2 = 0.0850
Final R indexes [all data]	R_1 = 0.0330, wR_2 = 0.0862
Largest diff. peak/hole / e Å^{-3}	1.59/–1.07

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 20250422lyj-31837-1_auto

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 20250422lyj-31837-1_auto

```
Bond precision:   C-C = 0.0051 A          Wavelength=1.54184

Cell:             a=13.6298(1)     b=25.7396(2)        c=8.3340(1)
                  alpha=90         beta=95.765(1)      gamma=90
Temperature:      100 K
```

	Calculated	Reported
Volume	2908.99(5)	2908.99(5)
Space group	P 21/c	P 21/c
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C10 H9 Br4 N O2	C10 H9 Br4 N O2
Sum formula	C10 H9 Br4 N O2	C10 H9 Br4 N O2
Mr	494.78	494.82
Dx,g cm-3	2.260	2.260
Z	8	8
Mu (mm-1)	13.444	13.444
F000	1856.0	1856.0
F000'	1836.99	
h,k,lmax	17,32,10	17,31,10
Nref	6064	5742
Tmin,Tmax	0.190,0.261	0.255,1.000
Tmin'	0.122	

```
Correction method= # Reported T Limits: Tmin=0.255 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.947           Theta(max)= 75.843

R(reflections)= 0.0313( 5427)                    wR2(reflections)=
                                                 0.0862( 5742)
S = 1.049                  Npar= 315
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

```
PLAT431_ALERT_2_B Short Inter HL..A Contact  Br2      ..O1       .       2.96 Ang.
                                          1-x,1-y,-z  =      3_665 Check
PLAT431_ALERT_2_B Short Inter HL..A Contact  Br3      ..O3       .       3.04 Ang.
                                          2-x,1-y,1-z  =      3_766 Check
PLAT431_ALERT_2_B Short Inter HL..A Contact  Br7      ..O3       .       3.02 Ang.
                                          2-x,1-y,2-z  =      3_767 Check
```

Alert level C

```
PLAT601_ALERT_2_C Unit Cell Contains Solvent Accessible VOIDS   <=         34 Ang**3
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L=    0.600          8 Report
             -16  1  3,   2  1  9,   3  1  9,   4  1  9,   5  1  9,   4  2  9,
               5  2  9,   5  3  9,
PLAT971_ALERT_2_C Check Calcd Resid. Dens.  1.02Ang From Br6             1.55 eA-3
```

Alert level G

```
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          2 Report
             H2    H4
PLAT083_ALERT_2_G SHELXL Second Parameter in WGHT  Unusually Large       5.81 Why ?
PLAT142_ALERT_4_G s.u. on b - Axis Small or Missing ..............    0.00020 Ang.
PLAT793_ALERT_4_G Model has Chirality at C8          (Centro SpGr)          R Verify
PLAT793_ALERT_4_G Model has Chirality at C18         (Centro SpGr)          S Verify
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600        307 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........        3.7 Low
PLAT969_ALERT_5_G The 'Henn et al.' R-Factor-gap value ...........      4.338 Note
             Predicted wR2: Based on SigI**2  1.99 or SHELX Weight  8.22
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          1 Info
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PLATON version of 02/02/2025; check.def file version of 02/02/2025

Datablock 20250422lyj-31837-1_auto - ellipsoid plot