Electronic Supplementary Material (ESI) for Chemical Science.

Supplementary Information

Electrochemically Driven Stereoselective Approach to *syn*-1,2-Diol Derivatives from Vinylarenes and DMF

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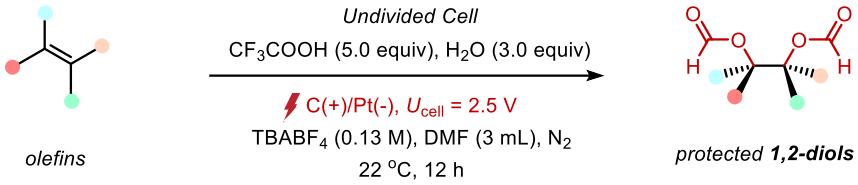
Section 1. General Information

All reactions were performed in oven-dried two-neck glass tubes unless otherwise noted. The tubes were fitted with a rubber septum and a threaded Teflon cap with airtight, electrical feed-throughs. The reactions were conducted under a nitrogen atmosphere. Flash chromatography was performed using silica gel 60 (230-400 mesh) from SiliCycle. Commercial reagents were purchased from Sigma Aldrich, Alfa Aesar, Acros, and TCI and used as received. (8*R*,9*S*,13*S*,14*S*)-13-methyl-3-vinyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[a]phenanthren-17-one,[1] ethyl 2-benzamido-3-(4-vinylphenyl)propanoate[2], *N*-cinnamyl-4-methylbenzenesulfonamide[3] (starting materials for **16**, **17**, and **23** respectivly) and **29**[4] were synthesized by the previously reported procedures. Proton nuclear magnetic resonance (^{1}H NMR) spectra was recorded on 300 MHz or 600 MHz, carbon nuclear magnetic resonance (^{13}C NMR) spectra was recorded on 75 MHz or 100 MHz and fluorine nuclear magnetic resonance (^{19}F NMR) was recorded on 282 MHz. Chemical shifts for protons are reported in parts per million downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent ($CHCl_3$ = δ 7.26). Chemical shifts for carbon are reported in parts per million downfield from tetramethylsilane and are referenced to the carbon resonances of the solvent ($CDCl_3$ = δ 77.0). Data are represented as follows: chemical shift, multiplicity (br. s = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz), integration. Infrared (IR) spectra of the newly synthesized compounds were obtained using a Bruker Alpha FT-IR spectrometer. Cyclic voltammetry data were measured with a Biologics SP-50 potentiostat. High resolution mass spectra was obtained from Korea Basic Science Institute (Daegu) by electron ionization (EI) and fast atom bombardment (FAB) method or from KAIST Research Analysis Center by using electrospray ionization (ESI) method.

Electrolysis experiments were performed using a Biologics SP-50 potentiostat/galvanostat or a DC power supply. Carbon Felt was purchased from Fuel Cell Store. The carbon was cut into 1 x 0.5 x 0.6 cm^3 pieces before use, and was connected to electrical feed-through on the Teflon cap of the electrochemical cell via a piece of graphite (2B pencil lead, 2 mm in diameter). The platinum plate was cut into 1 x 0.5 x 0.02 cm^3 and was connected to electrical feed-through on the Teflon cap of the electrochemical cell via a piece of graphite (2B pencil lead, 2 mm in diameter). Saturated calomel electrode (SCE) reference electrodes were obtained from CH Instruments.

Abbreviations: ^{t}Bu—*tert*-butyl, Me—methyl, Ac—acetyl, Ph—phenyl, Bz—benzoyl, DCM—dichloromethane, MeCN—acetonitrile, THF—tetrahydrofuran, Tf—trifluoromethansulfonyl, TFA—Trifluoroacetic acid, TBA—tetrabutylammonium, DMF—*N*,*N*-dimethylformamide.

Section 2. General Procedures for Electrochemically Driven *syn*-Dioxygenation of Olefins



An oven-dried, 10 mL two-neck glass tube was equipped with a magnetic stir bar, a rubber septum, a threaded Teflon cap fitted with electrical feed-throughs, a carbon felt anode (1.0 *0.5 cm^2) (connected to the electrical feedthrough via a 9 cm in length, 2 mm in diameter graphite rod), and a platinum plate cathode (0.5 * 1.0 cm^2). To this reaction vessel, $TBABF_4$ (131.6 mg, 0.4 mmol) was added. The cell was sealed and flushed with nitrogen gas for 5 minutes, followed by the sequential addition via syringe of olefin substrate (0.2 mmol, 1.0 equiv) in 3 mL of DMF and trifluoroacetic acid (1.0 mmol, 77 μL) and water (0.6 mmol, 11 μL). A nitrogen-filled balloon was adapted through the septum to sustain a nitrogen atmosphere. Electrolysis was initiated at a constant voltage of 2.5 mA at room temperature (22 °C) for 12 h. The mixture was then diluted with ethyl acetate (60 mL) and then washed with water, brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The residue was subjected to flash column chromatography on silica gel (eluted with hexanes/ethyl acetate) to yield the desired product.

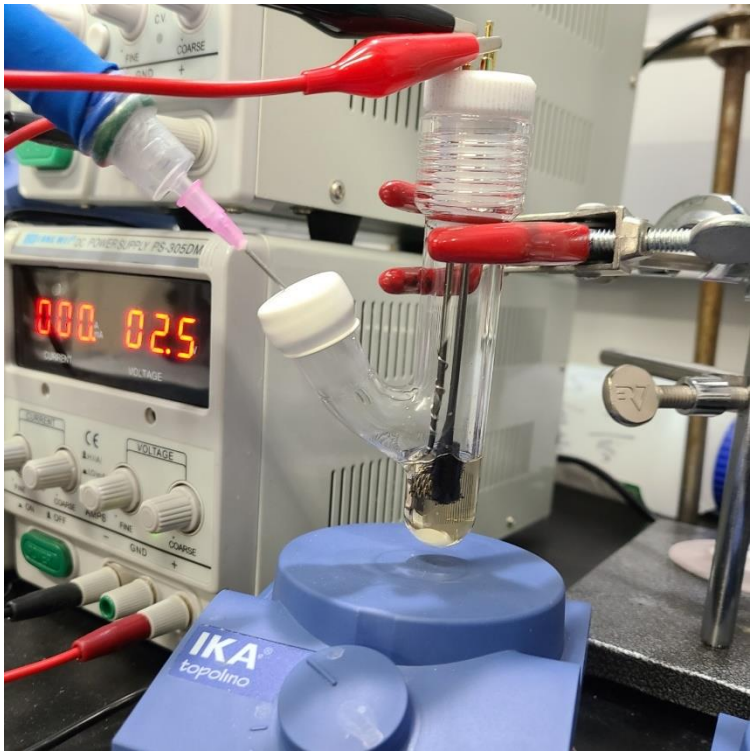
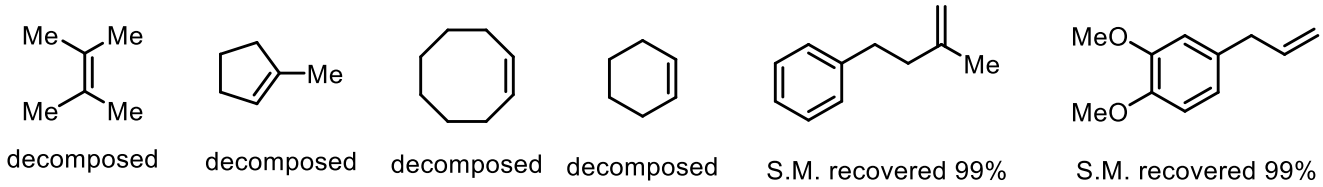


Figure S1. Setup for Electrochemical *syn*-Dioxygenation of Olefins

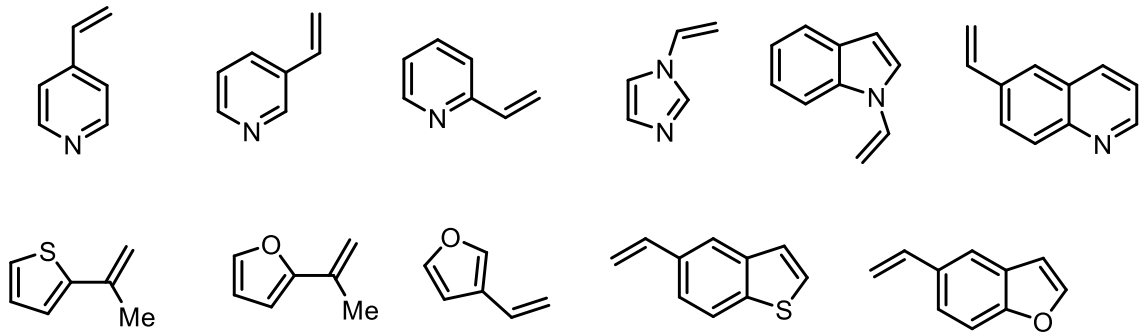
Unsuccessful Substrates

Unactivated Simple Olefins



Scheme S1. Unsuccessful Substrate Scopes: Unactivated Simple Olefins

Unsuccessful Vinyl Heteroarene Scopes



Scheme S2. Unsuccessful Vinyl Heteroarene Scopes

Section 3. Quantum Chemical Simulations

Oxidation potential of **C** (Figure 2)

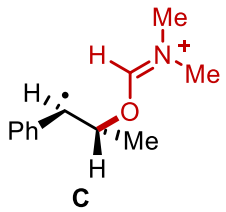
All DFT calculations were carried out using density functional theory[5] with Gaussian 09 program.[6] Geometry optimizations to the stationary points were performed with B3LYP[7–11] levels of theory and the 6-31G** basis set.[12-14] The electronic energies of optimized sturctures were reevaluated by single point calculations of M06 functionals[15] with 6-311++G** basis set.[16] Thermodynamic parameters including Gibbs free energies at 298.15 K were obtained by frequency calculations. Vibrational frequencies were carried out at the same level of theory as the geometry optimizations. Then, solvation correction energies were calculated by using the optimized gas-phase geometries at the same level of theory as the single point calculations. Solvation of DMF (ε = 37.51) was considered by single point calculation with integral equation formalism PCM model.

We performed quantum chemical simulations to predict electrochemical property of the proposed intermediate **C** (Figure 2). Oxidation potential (*E*) could be estimated from the Gibbs free energy change during the reduction:

$$E(\text{V vs. SCE}) = -\frac{\Delta G}{nF} - E(\text{SCE})$$

where Δ*G* is the Gibbs free energy difference between cation **C** and dication **D**, where **D** is formed by an electrochemical oxidation of **C**. n is the number of electrons involved (n=1 in this study), and F is Faraday constant of 23.06 kcal/mol. The calculated oxidation potential was referenced to the absolute reduction potential of NHE—4.43 V.[17,18] For the calculations of oxidation potential, electronic energies were further corrected by single point calculations of M06-D3 and M06-2X functionals.[19] We chose an oxidation potential calculated from M06 functional, because the results from three different functions showed less than 0.02 V of deviations.

Table S1. Cartesian Coodinates of DFT-optimized Stucture of **C**



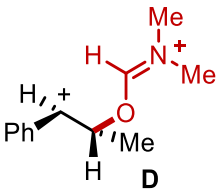
C

C12NH17O

C	2.870356000000	-0.938036000000	-1.398648000000
C	1.799839000000	-0.115625000000	-1.089933000000
C	1.657646000000	0.438056000000	0.218521000000
C	2.651915000000	0.106265000000	1.188369000000
C	3.718034000000	-0.715579000000	0.866908000000
C	3.835820000000	-1.243149000000	-0.427268000000
H	2.965335000000	-1.345072000000	-2.400259000000
H	1.067980000000	0.109581000000	-1.859633000000
H	2.566222000000	0.521035000000	2.188666000000
H	4.466411000000	-0.948581000000	1.617373000000
H	4.674303000000	-1.884241000000	-0.678901000000
C	0.590548000000	1.292824000000	0.595838000000
H	0.560868000000	1.640125000000	1.626264000000
C	-1.051995000000	3.139420000000	0.097560000000
H	-0.285352000000	3.912917000000	-0.008650000000
H	-1.892589000000	3.400972000000	-0.548428000000

H	-1.386927000000	3.130501000000	1.138362000000
O	-1.748881000000	0.846408000000	-0.363860000000
C	-1.734873000000	-0.312577000000	0.180090000000
H	-0.841782000000	-0.654866000000	0.703282000000
N	-2.770010000000	-1.114032000000	0.131867000000
C	-4.021812000000	-0.742930000000	-0.545179000000
H	-3.927733000000	0.252875000000	-0.971673000000
H	-4.228730000000	-1.468159000000	-1.336237000000
H	-4.839124000000	-0.760867000000	0.180323000000
C	-2.726112000000	-2.439956000000	0.763965000000
H	-1.754848000000	-2.600709000000	1.232777000000
H	-3.509556000000	-2.507260000000	1.523003000000
H	-2.893493000000	-3.209845000000	0.006518000000
C	-0.453272000000	1.807508000000	-0.296121000000
H	-0.176429000000	1.781232000000	-1.349580000000

Table S2. Cartesian Coodinates of DFT-optimized Stucture of **D**



D

C12NH17O

C	-3.920388000000	1.121018000000	0.683402000000
C	-2.579546000000	1.011267000000	0.409372000000
C	-2.035122000000	-0.252658000000	-0.033573000000
C	-2.924767000000	-1.387292000000	-0.164452000000
C	-4.266012000000	-1.257683000000	0.116344000000
C	-4.761936000000	-0.008589000000	0.537456000000
H	-4.341072000000	2.063437000000	1.016849000000
H	-1.933070000000	1.873954000000	0.531324000000
H	-2.519321000000	-2.339609000000	-0.493489000000
H	-4.937412000000	-2.103522000000	0.016781000000
H	-5.820597000000	0.092270000000	0.762292000000
C	-0.708970000000	-0.442054000000	-0.351866000000
H	-0.397316000000	-1.438313000000	-0.665188000000
C	0.397567000000	1.308355000000	-1.757527000000
H	-0.527179000000	1.873385000000	-1.895558000000
H	1.234523000000	2.012010000000	-1.787801000000
H	0.502354000000	0.595591000000	-2.578610000000
O	1.628680000000	-0.175025000000	-0.234789000000
C	2.671001000000	0.422781000000	0.293043000000
H	2.565211000000	1.427840000000	0.699196000000
N	3.824517000000	-0.159874000000	0.355178000000
C	4.077138000000	-1.515298000000	-0.178392000000
H	3.178705000000	-1.911490000000	-0.644017000000
H	4.883697000000	-1.450652000000	-0.912217000000
H	4.390386000000	-2.159406000000	0.646543000000
C	4.977772000000	0.520986000000	0.987903000000
H	4.685095000000	1.507949000000	1.346349000000
H	5.327286000000	-0.087081000000	1.824937000000
H	5.775328000000	0.617881000000	0.248290000000
C	0.378293000000	0.586677000000	-0.400192000000
H	0.302137000000	1.300010000000	0.427278000000

Table S3. Vibrational Frequencies of Optimized Geometries

The vibrational frequencies of optimized geometries are given in below (units: cm–1).

C

16.5092	29.4367	51.5236
89.4316	100.5585	131.5983
141.3342	160.5697	190.8940
231.5333	253.7423	265.9705
283.3787	371.9902	401.0506
406.0083	409.6778	414.5405
459.0018	482.6692	534.9034
614.7477	621.1488	680.7811
689.2711	746.0455	790.6258
835.3893	837.1480	875.6887
926.1160	942.5387	983.5881
996.5558	1011.0963	1022.4235
1027.7776	1044.7560	1070.5122
1101.8325	1125.7914	1135.5814
1150.7645	1177.4477	1179.6152
1191.2381	1209.3991	1247.9095
1263.0436	1270.0378	1337.8350
1358.0273	1369.6581	1378.3943
1428.1401	1456.8214	1458.2410
1469.5943	1476.5402	1485.1481
1493.0423	1502.5380	1503.3739
1505.5942	1508.1434	1526.7218
1536.5815	1589.5363	1615.7298
1730.5587	3063.7211	3068.2909
3074.7412	3135.1215	3145.8162
3146.5509	3146.6543	3157.0665
3165.5927	3174.3395	3175.5499
3190.5200	3192.4315	3205.5361
3206.7727	3212.8751	3223.0329

D

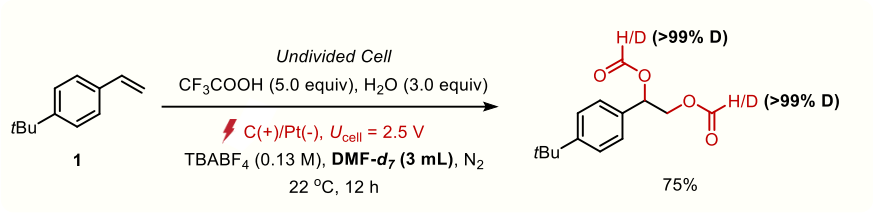
28.0620	31.6703	56.3303
79.1857	93.5247	102.5728
160.5034	166.5480	180.7970
228.5289	259.0968	275.9886
316.9544	341.0859	377.8783
409.4189	431.5030	433.5552
474.2384	517.3516	558.6833
605.1076	636.9188	648.8749
797.6734	825.6086	827.5683
839.5481	885.9098	927.4206
948.7817	984.5515	998.6774
1000.1036	1017.7494	1024.2644
1028.8098	1051.8083	1055.4946
1099.6098	1117.4051	1119.9743
1153.0059	1169.8935	1176.2748
1207.1140	1222.2174	1244.9268
1272.1769	1297.6814	1332.9551
1361.6264	1381.2718	1420.9901
1428.8875	1446.8583	1457.0437
1469.9673	1475.8629	1481.2393
1488.7754	1491.1160	1495.4720
1500.7443	1502.5885	1524.2877
1576.7384	1602.4902	1663.8219
1761.3301	3067.0071	3078.1595
3082.3241	3084.2074	3150.9894
3162.2019	3165.4571	3167.8684
3177.7064	3183.4308	3188.1334
3209.8027	3212.5289	3217.5708
3217.7599	3232.5649	3235.0889

Section 4. Procedures of Mechanistic Studies

1. Deuterium Labeling Experiments

1.1. Reaction under DMF-d_7 as a solvent

An identical procedure was followed as described in **Section 2** with the exception that the reaction was carried out in 3 mL of DMF-d_7 as a solvent. When the reaction was finished, the mixture was then diluted with ethyl acetate (60 mL) and then washed with water, brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The ^1H NMR yield and the ratio of proton/deuterium (H/D) of desired product was determined by integration using an internal standard (1,2-dimethoxyethane).



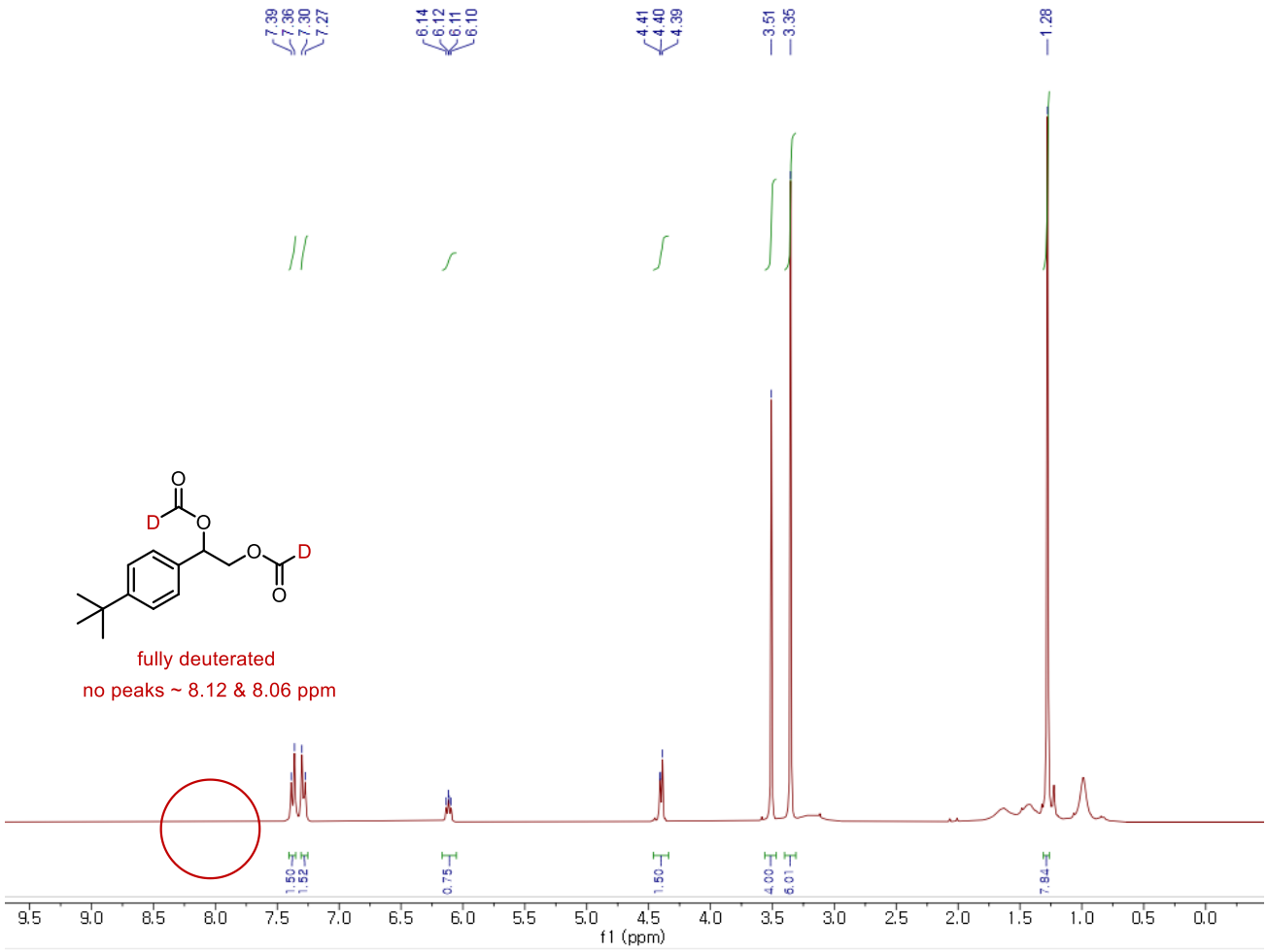
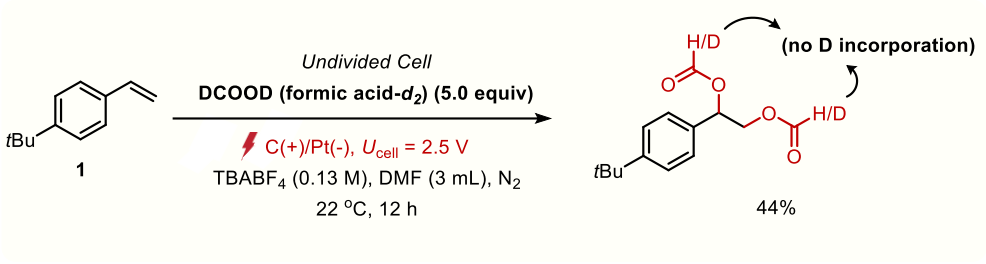


Figure S2. 1H NMR of Deuterium Scrambling Experiment Using DMF-d_7

1.2. Reaction under formic acid-d_2 as an acid additive

An identical procedure was followed as described in **Section 2** with the exception that the reaction was carried out with 5.0 equiv of formic acid-d_2 (1.0 mmol, 38 μL) instead of trifluoroacetic acid. When the reaction was finished, the mixture was then diluted with ethyl acetate (60 mL) and then washed with water, brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The ^{1}H NMR yield and the ratio of proton/deuterium (H/D) of desired product was determined by integration using an internal standard (1,2-dimethoxyethane).



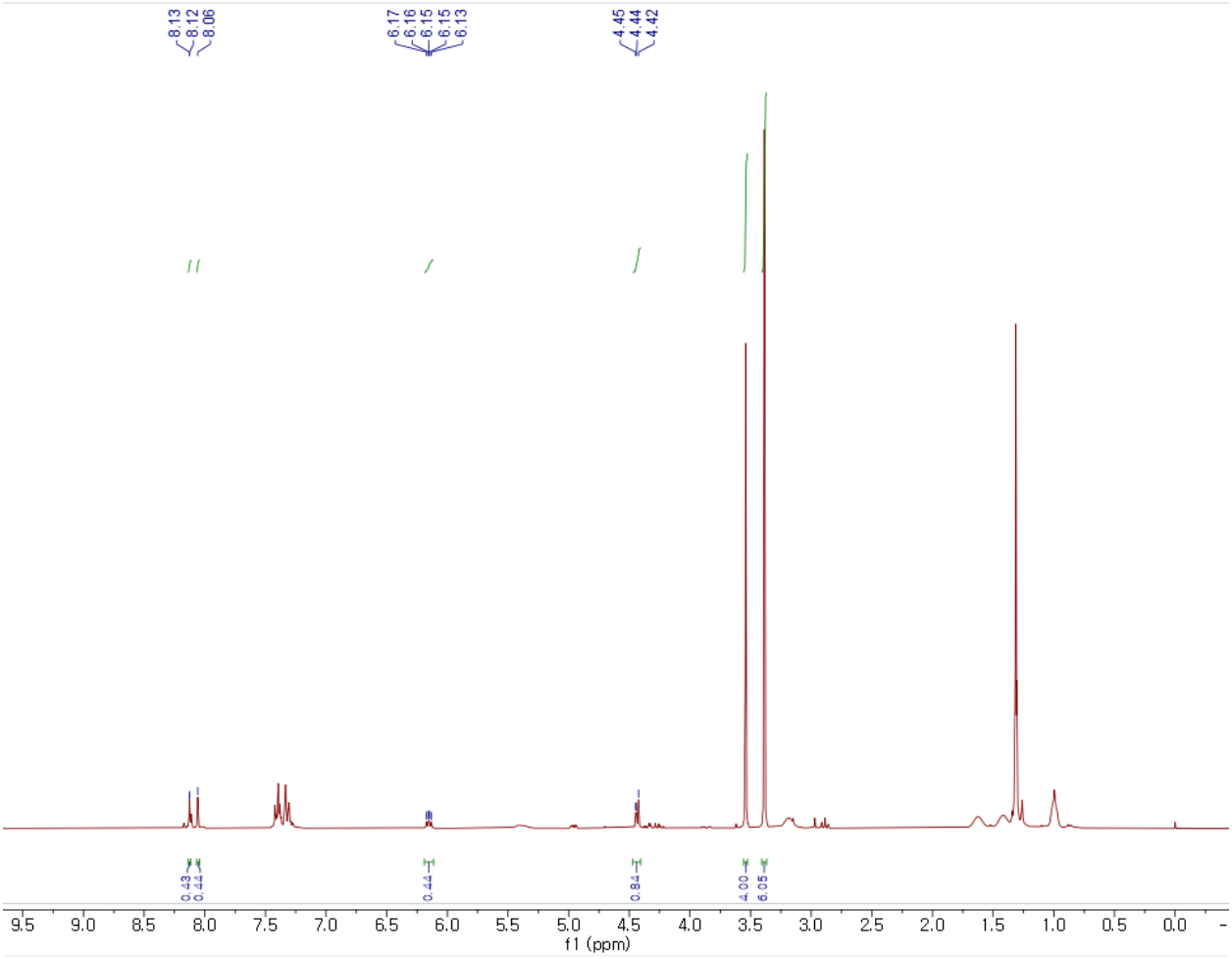
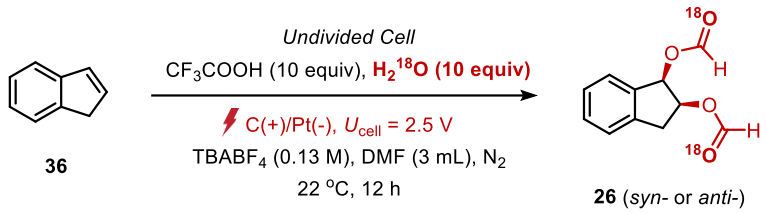


Figure S3. 1H NMR of Deuterium Scrambling Experiment Using Formic acid-d_2

2. ^{18}O-Oxygen labeling experiments



calculated of ESI-MS	m/z $[M+Na]^+$	*syn*-**26**	*anti*-**26**
unlabeled	229.0471	4.0%	3.2%
^{18}O-labeled	231.0514	26.7%	26.9%
doubly ^{18}O-labeled	233.0556	69.3%	69.9%

abundance was calculated by integration of peak area

An identical procedure was followed as described in **Section 2** with the exception that the reaction was carried out with 10.0 equiv of $H_2{}^{18}O$ (97 % ^{18}O-enriched, 2 mmol, 36 µL) instead of water. When the reaction was finished, the mixture was then diluted with ethyl acetate (60 mL) and then washed with water, brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The residue was subjected to flash column chromatography on silica gel (eluted with hexanes/ethyl acetate) to separate each isomer (*syn*-**26** (40%) and *anti*-**26** (9%)). High-resolution mass spectrum (ESI) showed the following isotope pattern:

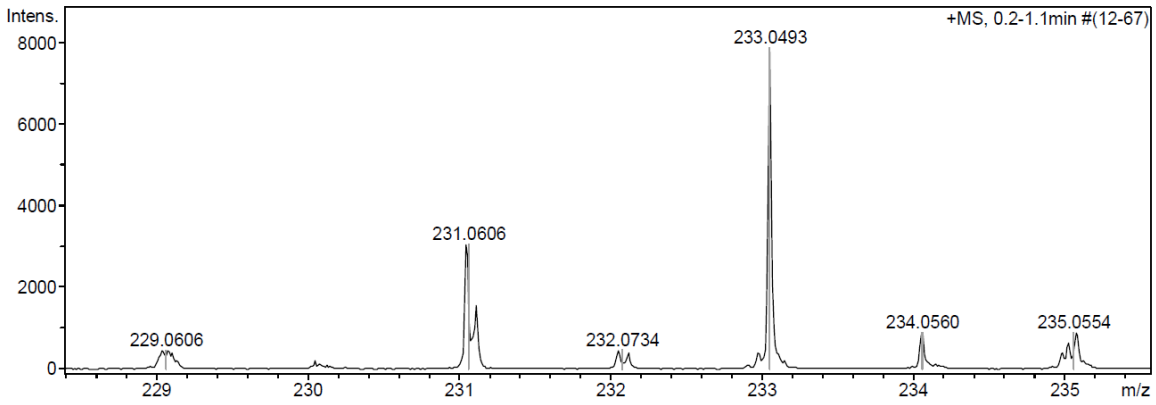


Figure S4. ESI-MS obtained from the isolated *syn*-**26**

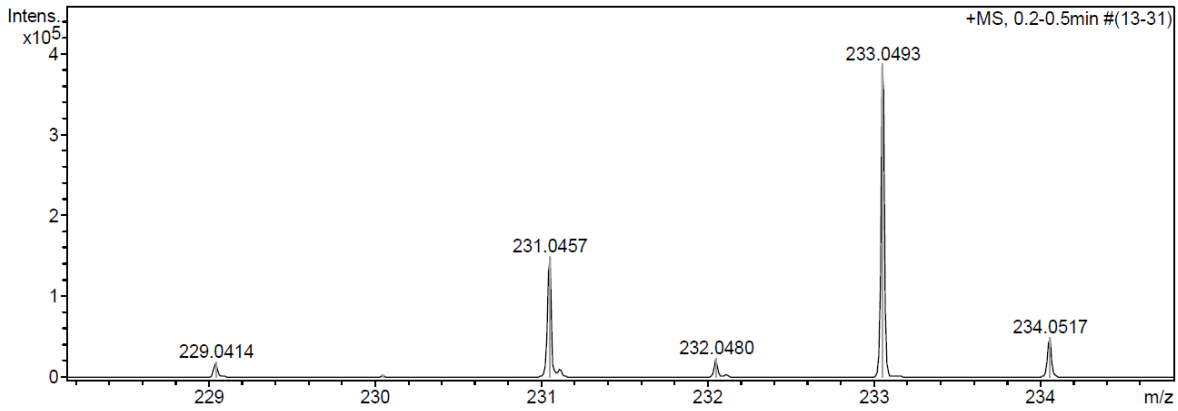
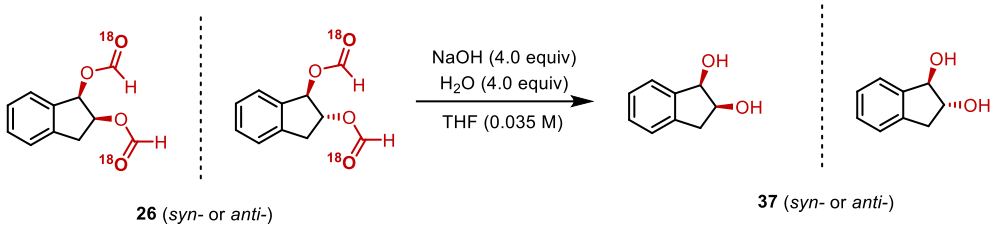


Figure S5. ESI-MS obtained from the isolated *anti*-**26**



26 (*syn*- or *anti*-) **37** (*syn*- or *anti*-)

calculated of ESI-MS	m/z $[M+Na]^+$	*syn*-**37**	*anti*-**37**
unlabeled	173.0573	99.0%	100.0%
^{18}O-labeled	175.0615	1.0%	not detected
doubly ^{18}O-labeled	177.0658	not detected	not detected

abundance was calculated by integration of peak area

A solution of the *syn*- or *anti*-**26** obtained in the ^{18}O-labeling experiment (5.2 mg, 0.035 mmol, in 1 mL of THF) was stirred with NaOH (5.6 mg, 0.14 mmol, 4.0 equiv.) and H_2O (2.5 μL, 0.14 mmol, 4.0 equiv.) at room temperature for 6 hours. The resulting mixture was filtered through Na_2SO_4 and concentrated on a rotary evaporator. The desired product (*syn*-**37** or *anti*-**37**) was obtained by column chromatography in quantative yields. High-resolution mass spectrum (ESI) of each isomers of **37** showed the following isotope pattern:

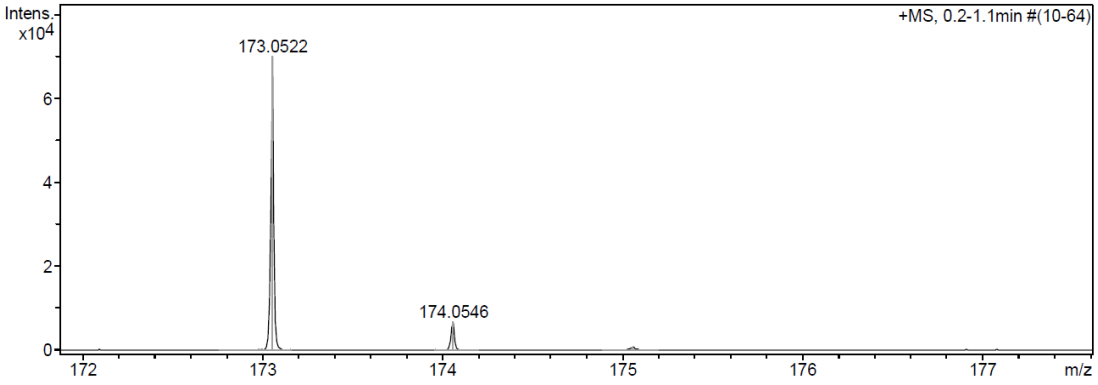


Figure S6. ESI-MS obtained from the isolated *syn*-**37**

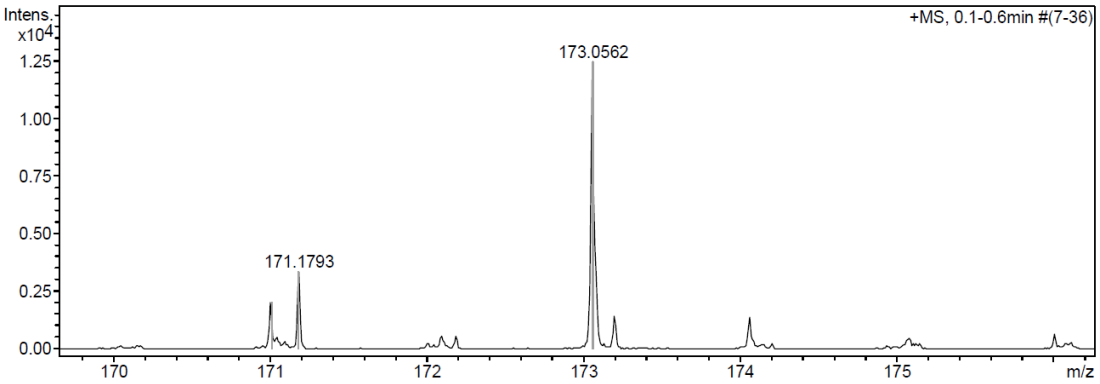


Figure S7. ESI-MS obtained from the isolated *anti*-**37**

3. Voltammetric studies

General information**:** Cyclic voltammetry (CV) was conducted in a 10 mL glass vial fitted with a glassy carbon working electrode (3 mm in diameter), SCE reference electrode, and a platinum wire counter electrode. The solution of interest was sparged with nitrogen for 3-5 minutes before data collection.

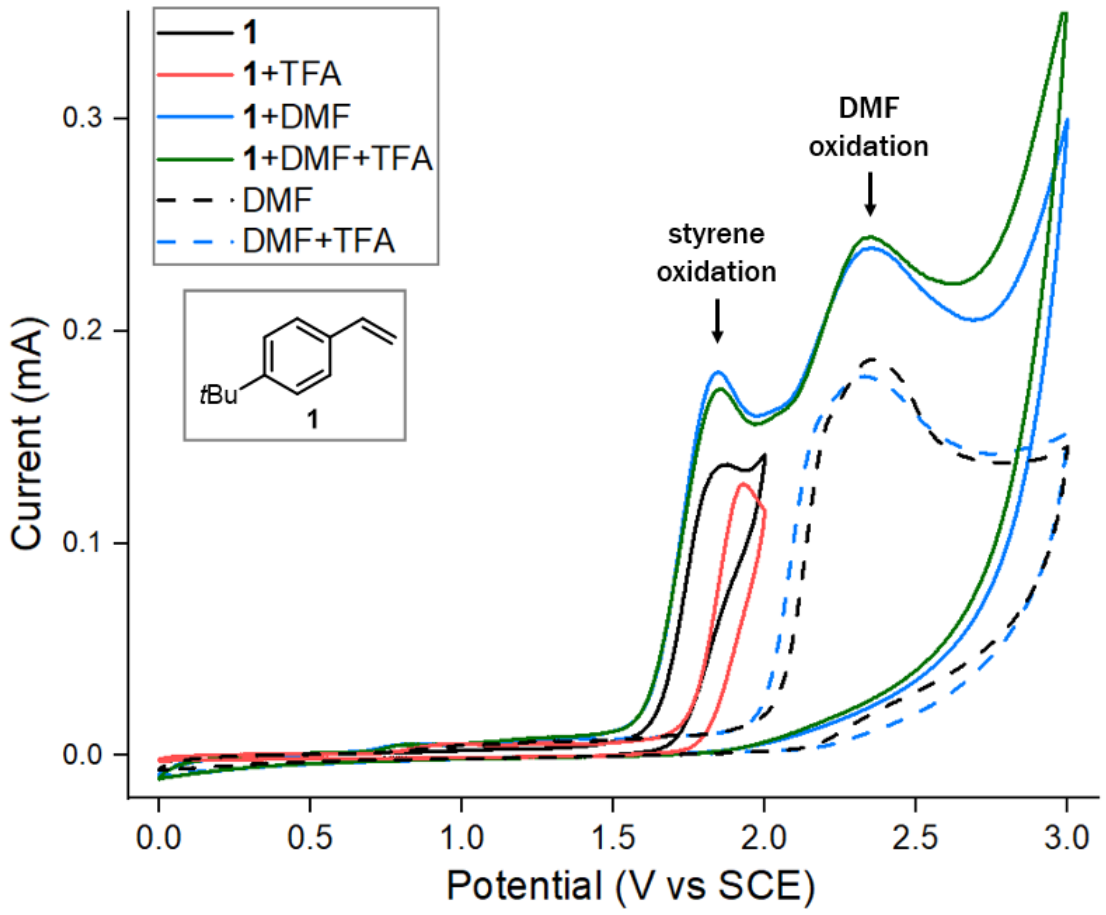
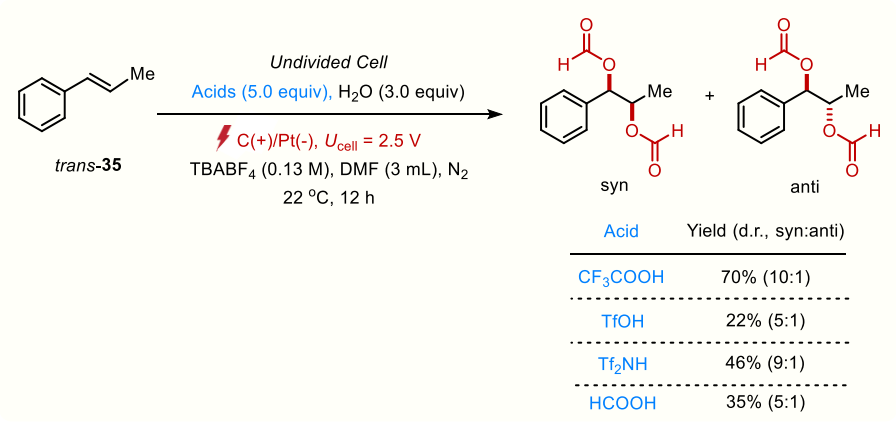
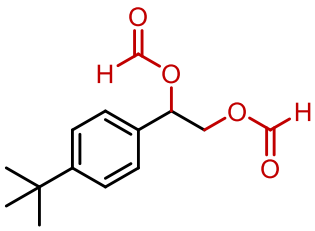


Figure S8. Cyclic voltammogram of **1**, DMF, TFA and their mixtures in MeCN. Conditions: $TBABF_4$ (0.10 M), **1** (5 mM), DMF (5 mM), TFA (5 mM). Scan rate: 100 mV/s.

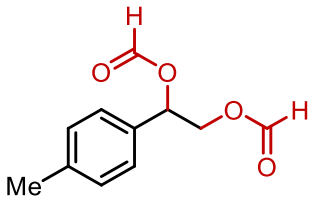
4. Evaluation of Diastereoselectivity by Control Experiments with Different Acids



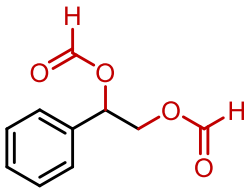
Section 5. Spectral Data for Products



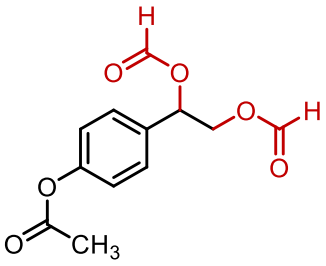
1-[4-(tert-Butyl)phenyl]ethane-1,2-diyl diformate (2). Purified using silica gel chromatography to give 45.0 mg (90% yield) of **2** as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.14 (s, 1H), 8.07 (s, 1H), 7.41 (d, *J* = 8.7 Hz, 2H), 7.32 (d, *J* = 8.2 Hz, 2H), 6.16 (dd, *J* = 7.3, 4.9 Hz, 1H), 4.49 – 4.37 (m, 2H), 1.31 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.5, 160.1, 152.3, 132.4, 126.7, 125.9, 72.7, 65.3, 34.8, 31.4; IR (Film): 2959, 2906, 2859, 1722, 1512, 1463, 1365, 1269, 1151, 1111 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{14}H_{18}O_4^+$]: 250.1200, found 250.1205.



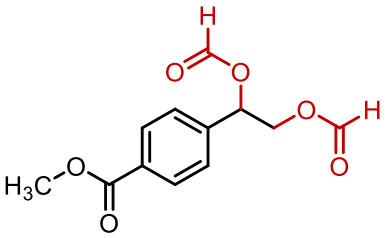
1-(*p*-Tolyl)ethane-1,2-diyl diformate (3). Purified using silica gel chromatography to give 20.8 mg (50% yield) of **3** as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (s, 1H), 8.09 (s, 1H), 7.31 (d, *J* = 8.2 Hz, 2H), 7.22 (d, *J* = 7.8 Hz, 2H), 6.16 (dd, *J* = 7.6, 4.6 Hz, 1H), 4.63 – 4.32 (m, 2H), 2.38 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.3, 159.9, 139.0, 132.4, 129.5, 126.7, 72.7, 65.1, 21.2. IR (Film): 2952, 2919, 2850, 1721, 1516, 1454, 1377, 1313, 1285, 1153, 1037, cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{11}H_{12}O_4^+$]: 208.0736, found 208.0734.



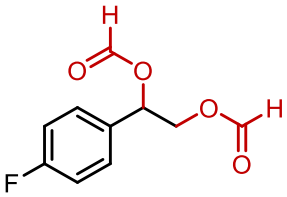
1-Phenylethane-1,2-diyl diformate (4). Purified using silica gel chromatography to give 28.0 mg (72% yield) of **4** as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.17 (s, 1H), 8.08 (s, 1H), 7.42 (d, *J* = 3.2 Hz, 5H), 6.19 (t, *J* = 6.5 Hz, 1H), 4.53 – 4.40 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.3, 159.8, 135.3, 129.0, 128.8, 126.7, 72.7, 65.1; IR (Film): 3055, 2984, 2937, 1726, 1606, 1496, 1454, 1374, 1265, 1156, 1027 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{10}H_{10}O_4^+$]: 194.0579, found 194.0581.



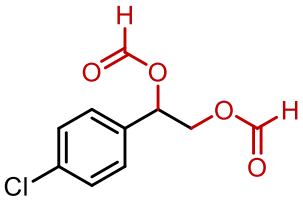
4-[1,2-Bis(formyloxy)ethyl]phenyl acetate (5). Purified using silica gel chromatography to give 25.2 mg (50% yield) of **5** as a white solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.15 (s, 1H), 8.08 (s, 1H), 7.44 (d, *J* = 8.6 Hz, 2H), 7.15 (d, *J* = 8.6 Hz, 2H), 6.20 (t, *J* = 6.0 Hz, 1H), 4.45 (d, *J* = 6.0 Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.2, 160.3, 159.7, 151.1, 132.9, 128.0, 122.0, 72.1, 64.9, 21.1; IR (Film): 2925, 2852, 1753, 1719, 1608, 1509, 1428, 1370, 1182, 1157, 1042 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{12}H_{12}O_6^+$]: 252.0634, found 252.0634.



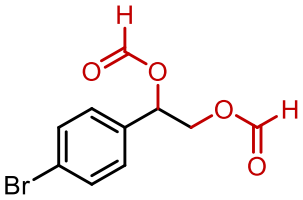
Methyl 4-(1,2-bis(formyloxy)ethyl)benzoate (6). Purified using silica gel chromatography to give 24.2 mg (48% yield) of **6** as a white solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.18 (s, 1H), 8.14 – 8.01 (m, 3H), 7.50 (d, J = 8.3 Hz, 2H), 6.23 (dd, J = 7.1, 4.5 Hz, 1H), 4.56 – 4.42 (m, 2H), 3.95 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.4, 160.2, 159.6, 140.2, 130.8, 130.1, 126.7, 72.2, 64.8, 52.3; IR (Film): 2953, 2923, 2851, 1718, 1613, 1436, 1280, 1151, 1112, 1019 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{12}H_{12}O_6^+$]: 252.0634, found 252.0634.



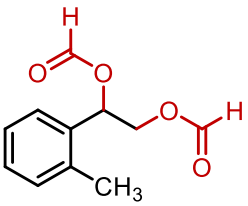
1-(4-Fluorophenyl)ethane-1,2-diyl diformate (7). Purified using silica gel chromatography to give 31.8 mg (75% yield) of **7** as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.15 (s, 1H), 8.08 (s, 1H), 7.41 (dd, *J* = 8.6, 5.3 Hz, 2H), 7.10 (t, *J* = 8.6 Hz, 2H), 6.16 (t, *J* = 6.0 Hz, 1H), 4.65 – 4.32 (m, 2H); δ ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.0 (d, *J* = 246.8 Hz), 160.3, 159.8, 131.3 (d, *J* = 3.5 Hz), 128.7 (d, *J* = 8.4 Hz), 115.9 (d, *J* = 21.7 Hz), 72.1, 64.9; ^{19}F NMR (282 MHz, $CDCl_3$) δ -112.14 (dtt, *J* = 10.7, 6.5, 2.6 Hz); IR (Film): 3032, 2923, 2850, 1718, 1495, 1454, 1374, 1333, 1266, 1155, 1026 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{10}H_9FO_4^+$]: 212.0485, found 212.0487.



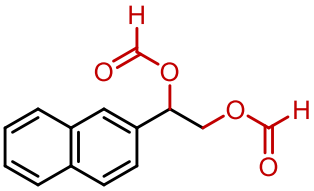
1-(4-Chlorophenyl)ethane-1,2-diyl diformate (8). Purified using silica gel chromatography to give 27.4 mg (60% yield) of **8** as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.12 (s, 1H), 8.05 (s, 1H), 7.41 – 7.29 (m, 4H), 6.12 (t, *J* = 5.9 Hz, 1H), 4.42 (d, *J* = 6.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.2, 159.7, 135.1, 133.9, 129.1, 128.2, 72.0, 64.8; IR (Film): 2922, 2851, 1720, 1600, 1493, 1376, 1345, 1149, 1092, 1014 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{10}H_9ClO_4^+$]: 228.0189, found 228.0188.



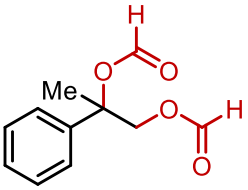
1-(4-Bromophenyl)ethane-1,2-diyl diformate (9). Purified using silica gel chromatography to give 60% yield of **9**. The yield was determined by ^{1}H NMR using 1,2-dimethoxyethane as an internal standard for this case because of an inseparable impurity; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.15 (s, 1H), 8.07 (s, 1H), 7.55 (d, *J* = 8.5 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 6.13 (t, *J* = 5.9 Hz, 1H), 4.52 – 4.29 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.2, 159.7, 134.4, 132.0, 128.4, 123.1, 72.0, 64.7; IR (Film): 2924, 2850, 1720, 1593, 1489, 1407, 1376, 1345, 1152, 1073 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{10}H_9BrO_4^+$]: 271.9684, found 271.9687.



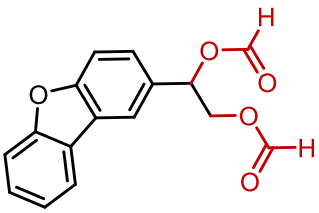
1-(*o*-Tolyl)ethane-1,2-diyl diformate (10). Purified using silica gel chromatography to give 29.1 mg (70% yield) of **10** as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (s, 1H), 8.10 (s, 1H), 7.44 – 7.38 (m, 1H), 7.30 – 7.19 (m, 3H), 6.41 (dd, *J* = 7.3, 4.8 Hz, 1H), 4.60 – 4.14 (m, 2H), 2.49 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.4, 159.9, 135.5, 133.7, 130.7, 128.8, 126.4, 126.2, 69.8, 64.5, 19.1; IR (Film): 2923, 2852, 1719, 1491, 1462, 1376, 1358, 1287, 1264, 1148 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{11}H_{12}O_4^+$]: 208.0736, found 208.0737.



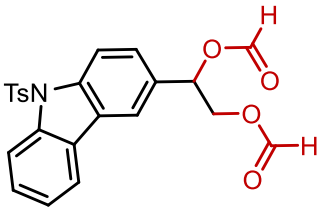
1-(Naphthalen-2-yl)ethane-1,2-diyl diformate (11). Purified using silica gel chromatography to give 31.7 mg (65% yield) of **11** as a white solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.20 (s, 1H), 8.08 (s, 1H), 8.00 – 7.73 (m, 4H), 7.61 – 7.39 (m, 3H), 6.34 (dd, *J* = 7.2, 4.8 Hz, 1H), 4.64 – 4.44 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.3, 159.9, 133.4, 133.0, 132.7, 128.8, 128.1, 127.7, 126.7, 126.6, 126.4, 123.9, 72.9, 65.1; IR (Film): 3055, 2927, 2851, 1717, 1495, 1447, 1376, 1286, 1152, 1070 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{14}H_{12}O_4^+$]: 244.0736, found 244.0734.



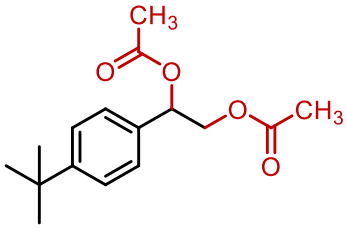
2-Phenylpropane-1,2-diyl diformate (12). Purified using silica gel chromatography to give 27.0 mg (65% yield) of **12** as a white solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10 (s, 2H), 7.54 – 7.33 (m, 5H), 4.70 – 4.26 (m, 2H), 1.99 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.2, 159.5, 139.9, 128.7, 128.3, 125.1, 82.3, 69.0, 22.1; IR (Film): 2979, 2922, 2851, 1717, 1495, 1447, 1376, 1286, 1152, 1070 cm^{-1}; HRMS (EI) exact mass calculated for [M-H^+, $C_{11}H_{12}O_4^+$]: 207.0652, found 207.0659.



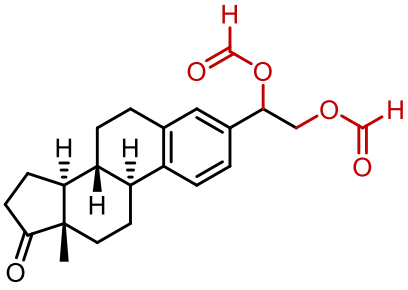
1-(Dibenzo[b,d]furan-2-yl)ethane-1,2-diyl diformate (13). Purified using silica gel chromatography to give 31.2 mg (55% yield) of **13** as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.21 (s, 1H), 8.12 (s, 1H), 8.07 – 7.92 (m, 2H), 7.66 – 7.58 (m, 2H), 7.52 (ddd, *J* = 8.3, 4.4, 2.4 Hz, 2H), 7.40 (td, *J* = 7.5, 1.1 Hz, 1H), 6.36 (dd, *J* = 8.1, 4.1 Hz, 1H), 4.68 – 4.45 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.3, 159.9, 156.6, 156.3, 130.0, 127.7, 125.8, 124.7, 123.6, 123.0, 120.8, 119.4, 112.1, 111.8, 72.8, 65.2; IR (Film): 2939, 1719, 1603, 1589, 1481, 1450, 1377, 1339, 1322, 1245, 1153 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{16}H_{12}O_5^+$]: 284.0685, found 284.0687.



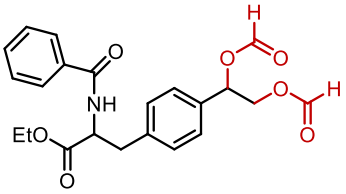
1-(9-Tosyl-9*H*-carbazol-3-yl)ethane-1,2-diyl diformate (14). Purified using silica gel chromatography to give 48.1 mg (55% yield) of **14** as a pale yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.33 (dd, *J* = 8.5, 5.0 Hz, 2H), 8.18 (s, 1H), 8.08 (s, 1H), 7.97 – 7.87 (m, 2H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.59 – 7.47 (m, 2H), 7.38 (td, *J* = 7.5, 1.0 Hz, 1H), 7.11 (d, *J* = 8.1 Hz, 2H), 6.31 (dd, *J* = 7.7, 4.4 Hz, 1H), 4.63 – 4.41 (m, 2H), 2.26 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.4, 159.9, 145.2, 138.7, 138.6, 134.9, 131.1, 129.8, 127.9, 126.7, 126.5, 125.9, 125.7, 124.1, 120.2, 118.6, 115.4, 115.1, 72.7, 65.2, 21.6; IR (Film): 2946, 1724, 1598, 1485, 1446, 1369, 1231, 1172, 1153, 1090 cm^{-1}; HRMS (ESI) exact mass calculated for [M+Na^+, $C_{23}H_{19}NNaO_6S^+$]: 460.0825, found 460.0855.



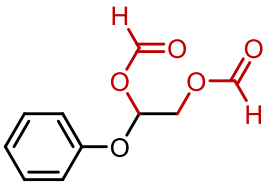
1-[4-(*tert*-butyl)phenyl]ethane-1,2-diyl diacetate (15). Purified using silica gel chromatography to give 17.2 mg (31% yield) of **15** as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.5 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 6.01 (dd, *J* = 7.8, 4.2 Hz, 1H), 4.41 – 4.19 (m, 2H), 2.11 (s, 3H), 2.06 (s, 3H), 1.31 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.7, 170.1, 151.6, 133.4, 126.5, 125.5, 73.1, 66.1, 34.6, 31.2, 21.1, 20.8; IR (Film): 2959, 2868, 1741, 1512, 1462, 1435, 1366, 1219, 1107, 1042, 1018 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{16}H_{22}O_4^+$]: 278.1518, found 278.1518.



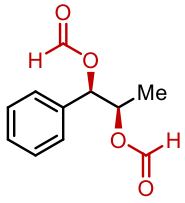
1-((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl)ethane-1,2-diyl diformate (16). Isolated as a 1:1 mixture of diastereomers. Purified using silica gel chromatography to give 46.6 mg (63% yield) of **16** as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (s, 1H), 8.09 (s, 1H), 7.34 (d, *J* = 8.1 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 1H), 7.14 (s, 1H), 6.13 (dd, *J* = 7.5, 4.7 Hz, 1H), 4.54 – 4.37 (m, 2H), 2.97 – 2.93 (m, 2H), 2.62 – 2.41 (m, 2H), 2.32 (td, *J* = 10.4, 4.0 Hz, 1H), 2.25 – 1.93 (m, 4H), 1.80 – 1.31 (m, 6H), 0.93 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 220.7, 160.4, 159.9, 140.8, 137.1, 132.8, 127.4 (2C), 125.9, 124.1 (2C), 72.6 (2C), 65.1, 50.4, 47.9, 44.3, 37.9, 35.8, 31.5, 29.3 (2C), 26.3, 25.6, 21.5, 13.8; IR (Film): x cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{22}H_{26}O_5^+$]: 370.1780, found 370.1780.



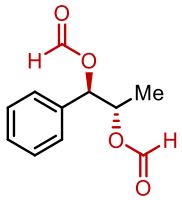
Ethyl 2-benzamido-3-{4-[1,2-bis(formyloxy)ethyl]phenyl}propanoate (17). Purified using silica gel chromatography to give 53.7 mg (65% yield) of **17** as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.12 (s, 1H), 8.04 (s, 1H), 7.73 (d, *J* = 8.3 Hz, 2H), 7.51 (t, *J* = 6.6 Hz, 1H), 7.46 – 7.37 (m, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 7.18 (d, *J* = 8.2 Hz, 2H), 6.64 (d, *J* = 7.6 Hz, 1H), 6.14 (t, *J* = 6.0 Hz, 1H), 5.12 – 4.99 (m, 1H), 4.41 (d, *J* = 6.0 Hz, 2H), 4.21 (q, *J* = 7.1 Hz, 2H), 3.38 – 3.15 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.4, 166.8, 160.3, 159.8, 137.1, 134.1, 133.8, 131.8, 129.9, 128.6, 127.0, 126.9, 72.4, 65.0, 61.7, 53.4, 37.6, 14.1; IR (Film): 3341, 2979, 2933, 1723, 1646, 1525, 1487, 1446, 1374, 1153 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{22}H_{23}NO_7^+$]: 413.1475, found 413.1476.



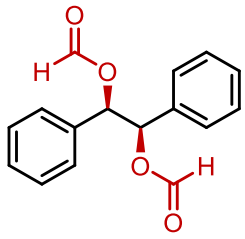
1-Phenoxyethane-1,2-diyl diformate (18). Purified using silica gel chromatography to give 15.5 mg (37% yield) of **18** as a colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10 (d, *J* = 4.0 Hz, 2H), 7.38 – 7.29 (m, 2H), 7.10 (t, *J* = 7.3 Hz, 1H), 7.01 (d, *J* = 7.6 Hz, 3H), 6.78 (t, *J* = 5.0 Hz, 1H), 4.50 (ddd, *J* = 58.8, 11.5, 5.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.0, 159.3, 155.3, 129.8, 123.8, 116.9, 91.4, 62.5; IR (Film): 2920, 2850, 1723, 1495, 1449, 1377, 1151, 1096, 1062, 1030 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{10}H_{10}O_5^+$]: 210.0528, found 210.0528.



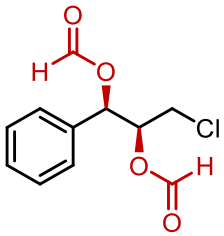
1-Phenylpropane-1,2-diyl diformate (19). ^{1}H NMR analysis [integration of formyl resonances at 8.11 (major) and 8.17 (minor) ppm] or the unpurified reaction indicated a 10:1 d.r.; **18** was obtained as a major diastereomer from the reaction using *trans*-**34** (*trans*-β-methylstyrene) (Figure 4C) as a starting material. Purified using silica gel chromatography to give 26.6 mg (64% yield) of **18** as a colorless oil. Relative configuration was determined after hydrolysis of **18** using the same method provided in page S10 and compared with the spectral data known in the precedent literature[20]; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.11 (s, 1H), 8.06 (s, 1H), 7.42 – 7.33 (m, 5H), 5.88 (d, *J* = 7.5 Hz, 1H), 5.43 (p, *J* = 6.6 Hz, 1H), 1.15 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.2, 159.8, 135.8, 129.0, 128.8, 127.4, 76.8, 71.1, 16.6.



1-Phenylpropane-1,2-diyl diformate (19'). ^{1}H NMR analysis [integration of formyl resonances at 8.17 (major) and 8.11 (minor) ppm] or the unpurified reaction indicated a 3:1 d.r.; **18'** was obtained as a major diastereomer from the reaction using *cis*-**34** (*cis*-β-methylstyrene) (Figure 4C) as a starting material. Purified using silica gel chromatography to give 20.4 mg (49% yield) of **18'** as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.20 (s, 1H), 8.04 (s, 1H), 7.41 – 7.37 (m, 5H), 6.08 (d, *J* = 5.2 Hz, 1H), 5.54 – 5.28 (m, 1H), 1.27 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.3, 159.9, 135.6, 128.8, 128.7, 127.2, 75.8, 71.4, 14.8; Properties for mixture of diastereomers: IR (Film): 2987, 2934, 1718, 1495, 1454, 1381, 1263, 1155, 1079, 1051 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{11}H_{12}O_4^+$]: 208.0736, found 208.0736.

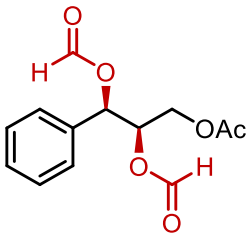


1,2-Diphenylethane-1,2-diyl diformate (20). Purified using silica gel chromatography to give 9.2 mg (17% yield) of **20** was obtained as a sole diastereomer as a white solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.13 (s, 2H), 7.25 – 7.10 (m, 10H), 6.19 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.7, 135.1, 128.8, 128.4, 127.6, 76.6; IR (Film): 3034, 2932, 1720, 1494, 1455, 1329, 1282, 1254, 1145, 1076 cm^{-1}; HRMS (ESI) exact mass calculated for [M+Na^+, $C_{16}H_{14}O_4Na^+$]: 293.0784, found 293.0785.



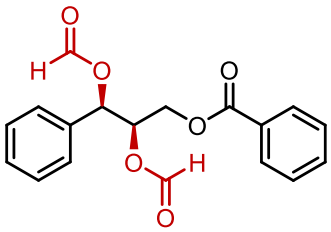
3-Chloro-1-phenylpropane-1,2-diyl diformate (21). ^{1}H NMR analysis [integration of formyl resonances at 8.09 (major) and 8.03 (minor) ppm] or the unpurified reaction indicated a 10:1 d.r. Purified using silica gel chromatography to afford the title as a colorless oil (24.2 mg , 50% yield).

Major diastereomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.13 (s, 1H), 8.10 (s, 1H), 7.46 – 7.35 (m, 8H), 6.18 (d, *J* = 7.4 Hz, 1H), 5.56 (dt, *J* = 8.3, 4.6 Hz, 1H), 3.64 (dd, *J* = 12.3, 4.0 Hz, 1H), 3.34 (dd, *J* = 12.3, 4.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.7, 159.5, 134.8, 129.5, 129.1, 127.4, 73.7, 73.0, 42.3; Properties for mixture of diastereomers: IR (Film): 2925, 2852, 1722, 1587, 1495, 1455, 1432, 1374, 1330, 1143, 1079 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{11}H_{11}ClO_4^+$]: 242.0346, found 242.0348.



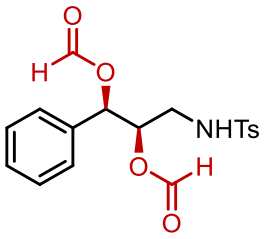
2,3-bis(formyloxy)-3-phenylpropyl acetate (22). ^{1}H NMR analysis [integration of benzyl resonances at 6.10 (major) and 5.90 (minor) ppm] or the unpurified reaction indicated a 10:1 d.r. Purified using silica gel chromatography to afford the title as a colorless oil (31.9 mg , 56% yield).

Major diastereomer: ^{1}H NMR (600 MHz, $cdcl_3$) δ 8.09 (s, 2H), 7.43 – 7.32 (m, 5H), 6.10 (d, *J* = 7.7 Hz, 1H), 5.65 – 5.53 (m, 1H), 4.27 (dd, *J* = 12.4, 3.3 Hz, 1H), 3.83 (dd, *J* = 12.3, 5.6 Hz, 1H), 2.06 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.3, 159.8, 159.5, 134.8, 129.4, 129.0, 127.2, 73.4, 71.6, 61.9, 20.6; Properties for mixture of diastereomers: IR (Film): 2926, 2853, 1722, 1666, 1496, 1454, 1370, 1229, 1151, 1047 cm^{-1}; HRMS (ESI) exact mass calculated for [$M+Na^+$, $C_{13}H_{14}O_6Na^+$]: 289.0683, found 289.0732.



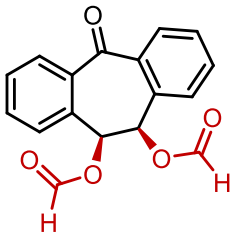
2,3-Bis(formyloxy)-3-phenylpropyl benzoate (23). ^{1}H NMR analysis [integration of formyl resonances at 8.13 (major) and 8.17 (minor) ppm] or the unpurified reaction indicated a 16:1 d.r. Purified using silica gel chromatography to afford the title as a colorless oil (38.7 mg , 59% yield);

Major diastereomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.12 (d, *J* = 4.4 Hz, 2H), 8.06 – 7.96 (m, 2H), 7.63 – 7.54 (m, 1H), 7.51 – 7.33 (m, 7H), 6.20 (d, *J* = 8.0 Hz, 1H), 5.82 – 5.69 (m, 1H), 4.46 (dd, *J* = 12.3, 3.3 Hz, 1H), 4.12 (dd, *J* = 12.3, 5.6 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.8, 159.8, 159.5, 134.8, 133.4, 129.7, 129.4, 129.2, 129.0, 128.5, 127.3, 73.6, 71.7, 62.5; Properties for mixture of diastereomers: IR (Film): 2935, 1720, 1601, 1584, 1494, 1452, 1315, 1217, 1150, 1115, 1071 cm^{-1}; HRMS (ESI) exact mass calculated for [M+Na^+, $C_{18}H_{16}O_6Na^+$]: 351.0839, found 351.0895.



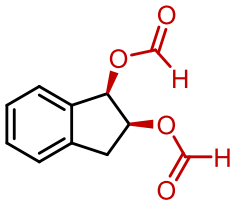
3-[(4-Methylphenyl)sulfonamide]-1-phenylpropane-1,2-diyl diformate (24). ^{1}H NMR analysis [integration of aromatic resonances at 7.66 (major) and 7.81 (minor) ppm] or the unpurified reaction indicated a 7:1 d.r. Purified using silica gel chromatography to afford the title as a colorless oil (32.4 mg , 43% yield).

Major diastereomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.93 (s, 1H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.37 – 7.19 (m, 8H), 6.02 (d, *J* = 6.9 Hz, 1H), 5.36 (q, *J* = 5.8 Hz, 1H), 5.19 (t, *J* = 6.6 Hz, 1H), 3.05 (t, *J* = 5.5 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.8, 159.6, 143.8, 136.3, 134.7, 129.8, 129.2, 128.9, 127.2, 127.1, 73.5, 72.6, 42.7, 21.5; Properties for mixture of diastereomers: IR (Film): 3036, 2928, 1724, 1597, 1494, 1432, 1376, 1330, 1267, 1155, 1093 cm^{-1} HRMS (ESI) exact mass calculated for [M+Na^+, $C_{18}H_{16}O_6Na^+$]: 400.0825, found 400.0860.



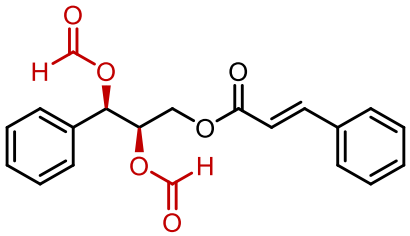
5-Oxo-10,11-dihydro-5H-dibenzo[*a,d*][7]annulene-10,11-diyl diformate (25). ^{1}H NMR analysis [integration of benzyl resonances at 6.57 (major) and 6.47 (minor) ppm] or the unpurified reaction indicated a 8:1 d.r. Purified using silica gel chromatography to afford the title as a white solid (35.5 mg , 60% yield).

Major diastereomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (s, 2H), 8.01 (dd, *J* = 8.1, 1.6 Hz, 2H), 7.63 – 7.55 (m, 2H), 7.50 (t, *J* = 6.9 Hz, 4H), 6.57 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 193.5, 159.8, 137.5, 132.8 (2C), 130.4, 129.3, 128.5, 73.9; Properties for mixture of diastereomers: IR (Film): 2929, 1723, 1655, 1597, 1451, 1344, 1294, 1240, 1143, 1076 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{17}H_{12}O_5^+$]: 296.0685, found 296.0683.

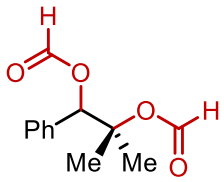


2,3-Dihydro-1*H*-indene-1,2-diyl diformate (26). ^{1}H NMR analysis [integration of benzyl resonances at 6.36 (major) and 6.43 (minor) ppm] or the unpurified reaction indicated a 4:1 d.r. Purified using silica gel chromatography to afford the title as a white solid (22.4 mg , 49% yield).

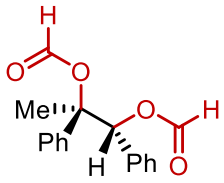
Major diastereomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.14 (s, 1H), 8.09 (s, 1H), 7.48 – 7.39 (m, 1H), 7.43 – 7.31 (m, 1H), 7.35 – 7.26 (m, 2H), 6.36 (d, *J* = 5.2 Hz, 1H), 5.66 (q, *J* = 5.5 Hz, 1H), 3.38 – 3.08 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.2 (2C), 130.1, 127.7, 126.0, 125.1, 74.6, 72.8, 35.8; Properties for mixture of diastereomers: IR (Film): 2924, 2850, 1720, 1593, 1489, 1407, 1376, 1345, 1152, 1073 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{11}H_{10}O_4^+$]: 206.0579, found 206.0582.



2,3-Bis(formyloxy)-3-phenylpropyl cinnamate (27). Purified using silica gel chromatography to afford the title as a white solid (62.3 mg, 88% yield); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.14 (d, *J* = 4.3 Hz, 2H), 7.72 (d, *J* = 16.0 Hz, 1H), 7.61 – 7.52 (m, 2H), 7.49 – 7.37 (m, 8H), 6.47 (d, *J* = 16.0 Hz, 1H), 6.21 (d, *J* = 7.9 Hz, 1H), 5.77 – 5.65 (m, 1H), 4.39 (dd, *J* = 12.3, 3.3 Hz, 1H), 4.04 (dd, *J* = 12.3, 5.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 159.8, 159.5, 146.0, 134.9, 134.0, 130.6, 129.4, 129.0, 128.9, 128.2, 127.3, 116.8, 73.5, 71.8, 62.0; IR (Film): 2942, 1718, 1635, 1578, 1495, 1451, 1310, 1270, 1202, 1148 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{20}H_{18}O_6^+$]: 354.1103, found 354.1102.

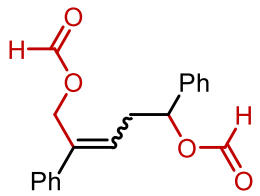


2-Methyl-1-phenylpropane-1,2-diyl diformate (28). Purified using silica gel chromatography to afford the title as a white solid (19.1 mg, 43% yield); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16 (d, *J* = 1.0 Hz, 1H), 8.01 (s, 1H), 7.45 – 7.30 (m, 5H), 6.08 (s, 1H), 1.54 (d, *J* = 2.7 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.07, 159.70, 135.51, 128.68, 128.25, 128.17, 83.34, 78.94, 22.97, 22.09; IR (Film): 2988, 2925, 2853, 1718, 1495, 1454, 1386, 1370, 1285, 1142 cm^{-1}; HRMS (ESI) exact mass calculated for [M+Na^+, $C_{12}H_{14}O_4Na^+$]: 245.0784, found 245.0824.



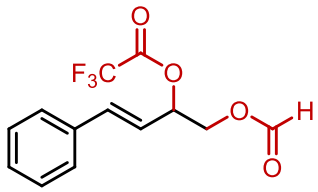
1,2-Diphenylpropane-1,2-diyl diformate (29). ^{1}H NMR analysis [integration of formyl resonances at 8.08 (major) and 8.02 (minor) ppm] or the unpurified reaction indicated a 10:1 d.r. Purified using silica gel chromatography to afford the title as a colorless oil (27.8 mg , 49% yield).

Major diastereomer: ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.14 (s, 1H), 8.08 (s, 1H), 7.31 – 7.26 (m, 4H), 7.20 – 7.11 (m, 4H), 6.91 (d, *J* = 7.6 Hz, 2H), 6.15 (s, 1H), 1.95 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.5 (2C), 139.4, 134.3, 128.5, 128.3 (2C), 128.2, 127.7, 126.2, 85.4, 79.9, 19.0; Properties for mixture of diastereomers: IR (Film): 2929, 2853, 1720, 1606, 1512, 1452, 1422, 1376, 1347, 1153 cm^{-1}; HRMS (ESI) exact mass calculated for [M+Na^+, $C_{17}H_{16}O_4Na^+$]: 307.0941, found 307.0979.

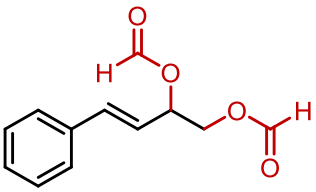


2,5-Diphenylpent-2-ene-1,5-diyl diformate (31). Purified using silica gel chromatography to afford **30** as a mixture of diastereomers (white solid, 30.4 mg, 49% yield).

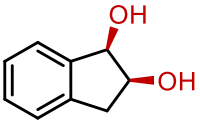
Major diastereomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.15 (s, 1H), 8.02 (s, 1H), 7.45 – 7.36 (m, 4H), 7.40 – 7.29 (m, 6H), 6.04 (t, *J* = 6.7 Hz, 1H), 5.97 (t, *J* = 7.5 Hz, 1H), 5.07 - 4.98 (m, 2H), 3.22 – 2.81 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.8, 160.2, 140.0, 139.0, 137.1, 128.8, 128.7, 128.5 (2C), 127.7, 126.5, 126.3, 75.0, 60.3, 35.6; Properties for mixture of diastereomers: IR (Film): 3032, 2922, 2848, 1718, 1494, 1454, 1374, 1333, 1158, 1026 cm^{-1}; HRMS (EI) exact mass calculated for [M^+, $C_{19}H_{18}O_4^+$]: 310.1205, found 310.1207.



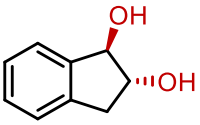
(*E*)-1-(Formyloxy)-4-phenylbut-3-en-2-yl 2,2,2-trifluoroacetate (33). Purified using silica gel chromatography to afford **32** as a colorless oil (25.9 mg, 45% yield); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.42 – 7.19 (m, 5H), 6.77 (d, *J* = 15.8 Hz, 1H), 6.07 (dd, *J* = 15.9, 7.9 Hz, 1H), 5.77 (td, *J* = 7.8, 3.5 Hz, 1H), 4.45 (ddd, *J* = 12.2, 3.5, 0.9 Hz, 1H), 4.31 (ddd, *J* = 12.2, 7.7, 0.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.1, 156.6 (q, *J* = 32.3 Hz), 137.5, 134.9, 129.1, 128.8, 127.0, 119.6, 114.4 (q, *J* = 285.7 Hz), 76.5, 63.5; ^{19}F NMR (282 MHz, $CDCl_3$) δ -75.0; IR (Film): 2921, 2851, 1786, 1728, 1494, 1451, 1378, 1222, 1148, 1026 cm^{-1}; HRMS (EI) exact mass calculated for [$C_{13}H_{11}F_3O_4^+$]: 288.0609, found 288.0609.



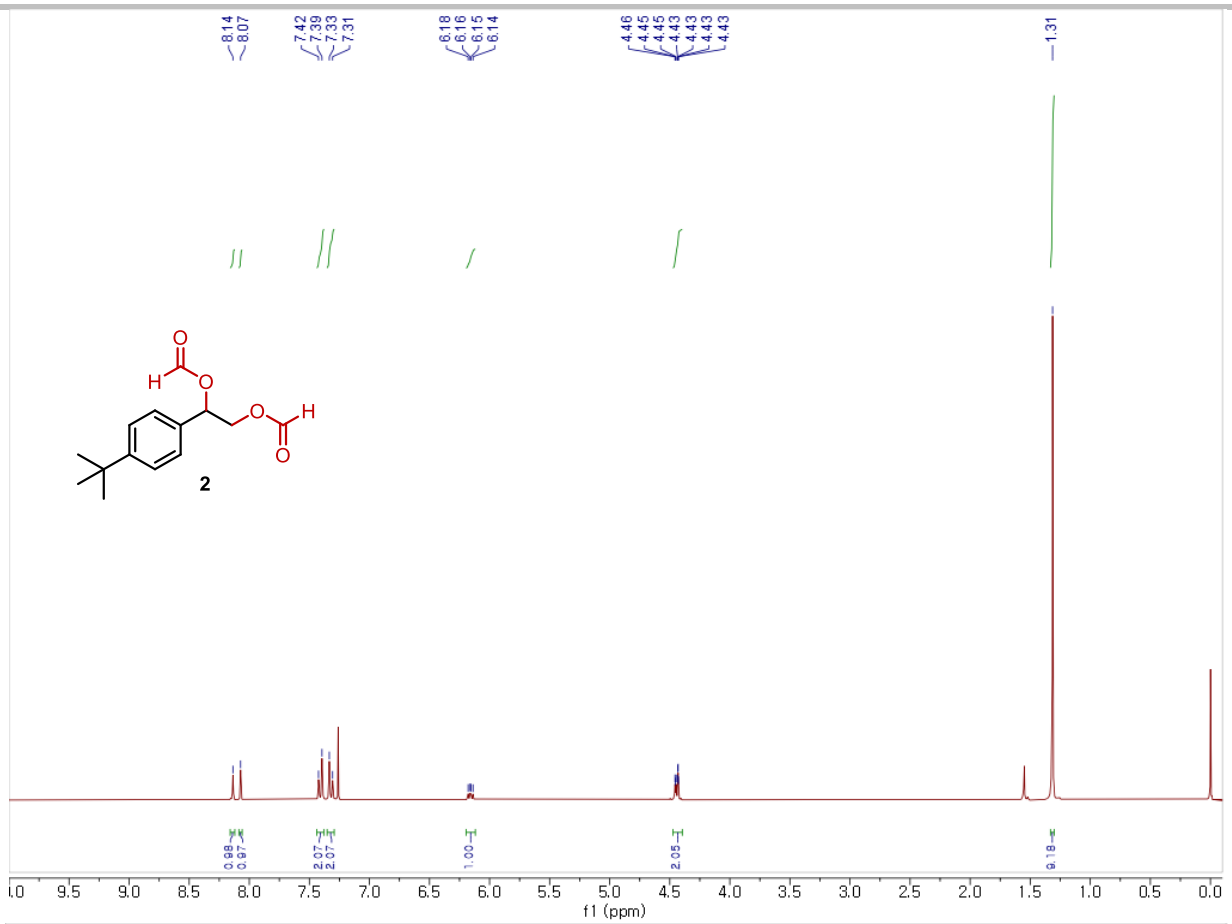
(*E*)-4-Phenylbut-3-ene-1,2-diyl diformate (34). Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.14 (s, 1H), 8.09 (s, 1H), 7.44 – 7.28 (m, 5H), 6.77 (d, *J* = 16.2 Hz, 1H), 6.14 (dd, *J* = 16.0, 7.3 Hz, 1H), 5.83 (td, *J* = 7.2, 3.7 Hz, 1H), 4.45 (dd, *J* = 11.9, 3.7 Hz, 1H), 4.34 (dd, *J* = 11.9, 7.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.4, 159.9, 135.5, 135.4, 128.7, 128.6, 126.8, 121.8, 71.6, 64.1; IR (Film): 2924, 2849, 1787, 1721, 1493, 1450, 1376, 1352, 1154, 1029 cm^{-1}; HRMS (EI) exact mass calculated for [$C_{12}H_{12}O_4^+$]: 220.0736, found 220.0736.



2,3-Dihydro-1H-indene-1,2-diol (*syn*-**37**). Relative configuration of **36** was determined by comparing with the spectral data known in the precedent literature.[20] White Solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 – 7.40 (m, 1H), 7.34 – 7.14 (m, 4H), 4.97 (d, *J* = 4.9 Hz, 1H), 4.46 (q, *J* = 5.5 Hz, 1H), 3.11 (dd, *J* = 16.3, 5.7 Hz, 1H), 2.94 (dd, *J* = 16.2, 3.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.9, 140.1, 128.8, 127.1, 125.3, 125.0, 75.9, 73.4, 38.5; HRMS (EI) exact mass calculated for [$C_9H_{10}O_2^+$]: 150.0681, found 150.0679.



2,3-Dihydro-1H-indene-1,2-diol (*anti*-**37**). White Solid. ^{1}H NMR (300 MHz, MeOD) δ 7.39 – 7.32 (m, 1H), 7.28 – 7.15 (m, 4H), 4.88 (d, *J* = 4.4 Hz, 1H), 4.26 (td, *J* = 6.9, 5.3 Hz, 1H), 3.24 (dd, *J* = 15.7, 7.1 Hz, 1H), 2.74 (dd, *J* = 15.7, 6.7 Hz, 1H); ^{13}C NMR (75 MHz, MeOD) δ 142.5, 139.3, 127.9, 126.5, 124.4, 124.0, 81.1, 80.2, 37.6; HRMS (EI) exact mass calculated for [$C_9H_{10}O_2^+$]: 150.0681, found 150.0680.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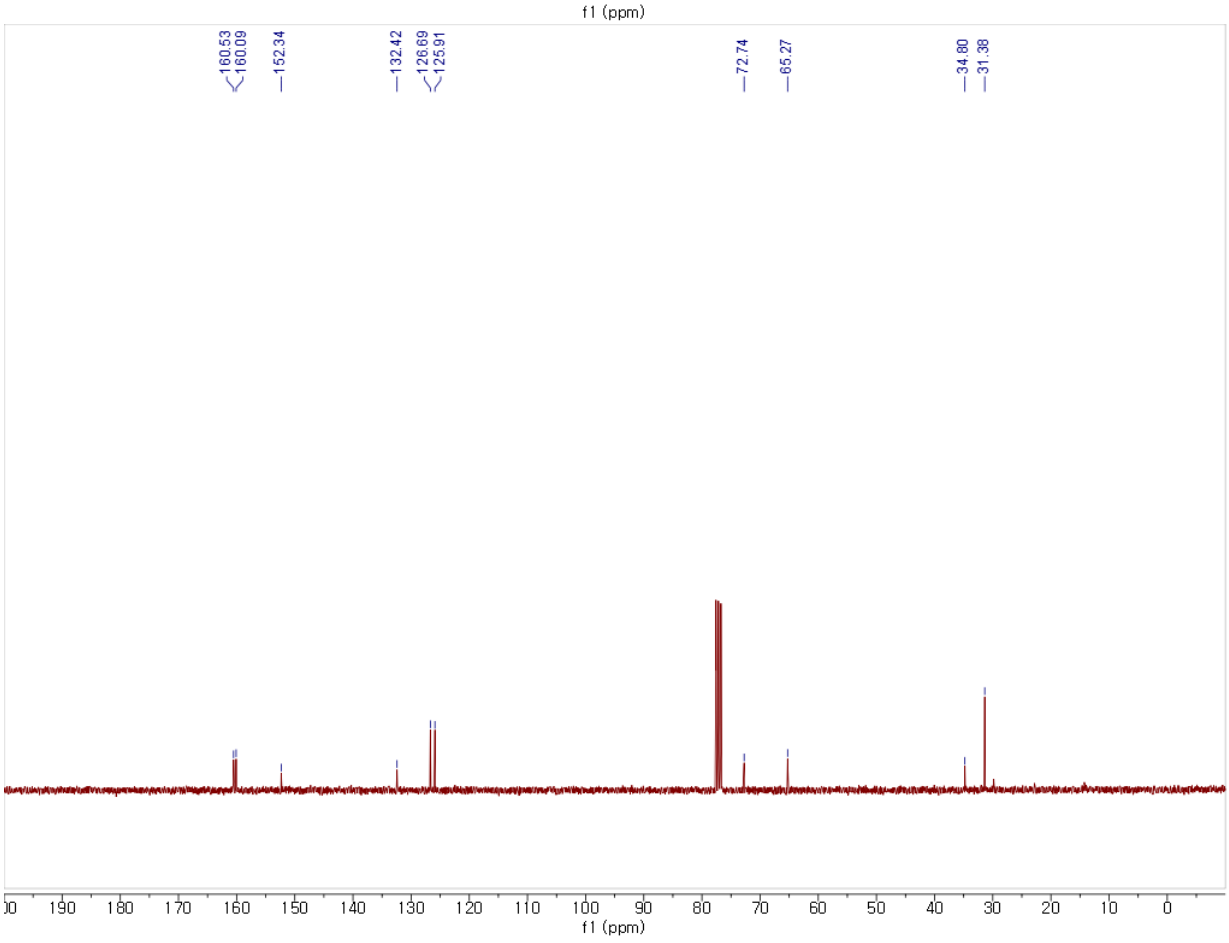
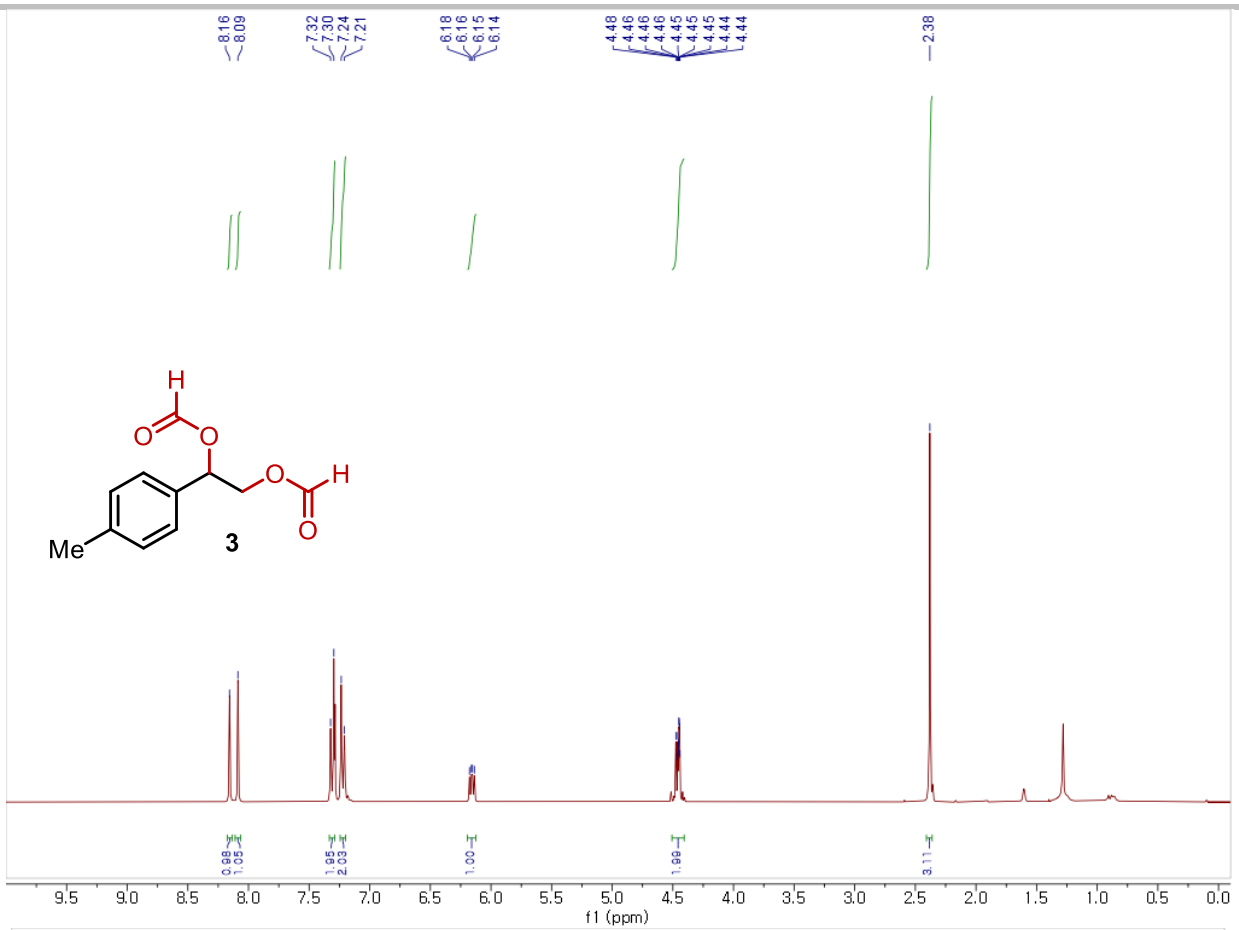
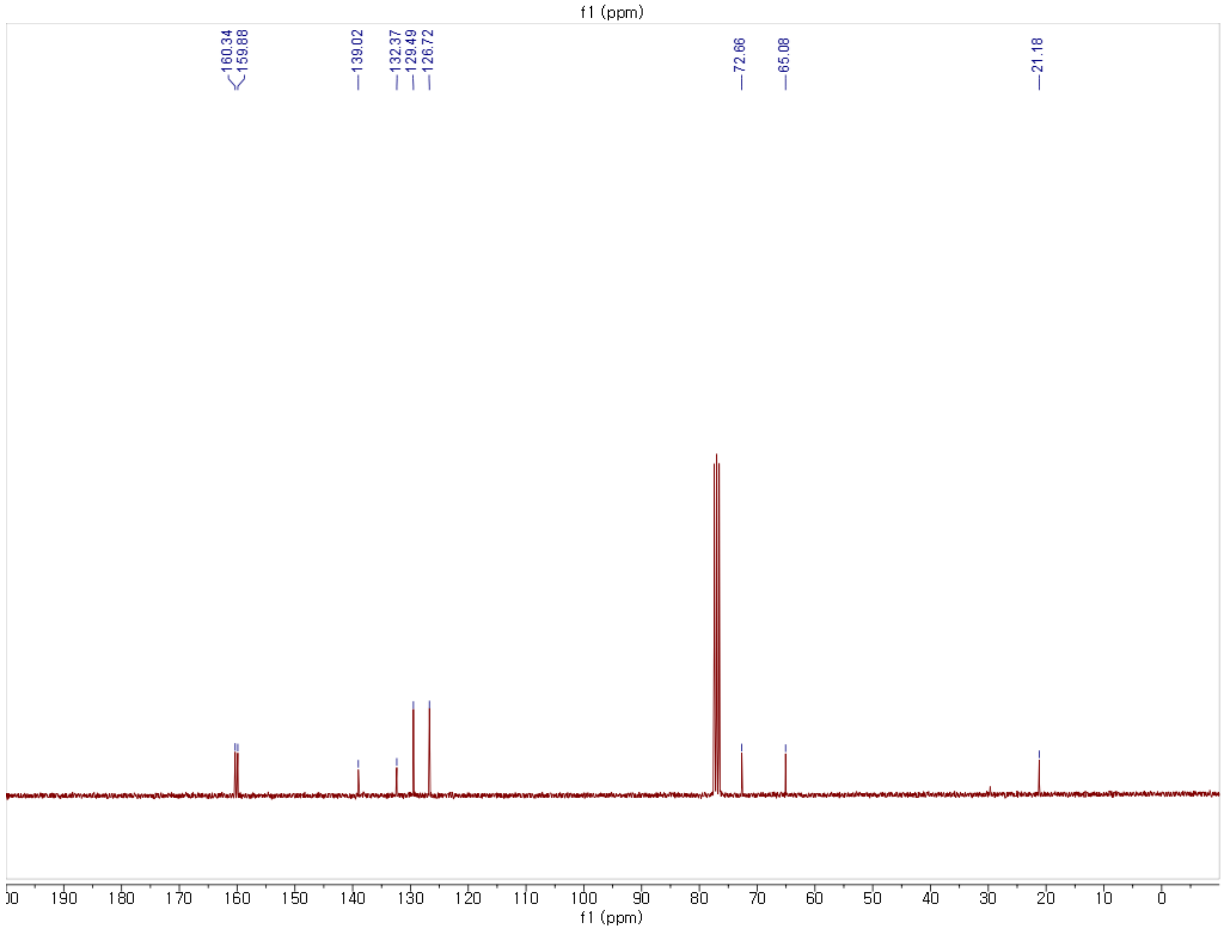
8.07
7.42
7.39
7.33
7.31
6.18
6.16
6.15
6.14
4.46
4.45
4.45
4.43
4.43
4.43
4.43
1.31
2
0.98
0.97
2.07
2.07
1.00
2.05
9.18
f1 (ppm)


160.53
160.09
152.34
132.42
126.69
125.91
72.74
65.27
34.80
31.38
f1 (ppm)


8.16
8.09
7.32
7.30
7.24
7.21
6.18
6.16
6.15
6.14
4.48
4.46
4.46
4.46
4.45
4.45
4.45
4.44
4.44
2.38
H
O
O
O
H
O
Me
3
0.98
1.05
1.95
2.93
1.00
1.99
3.11
f1 (ppm)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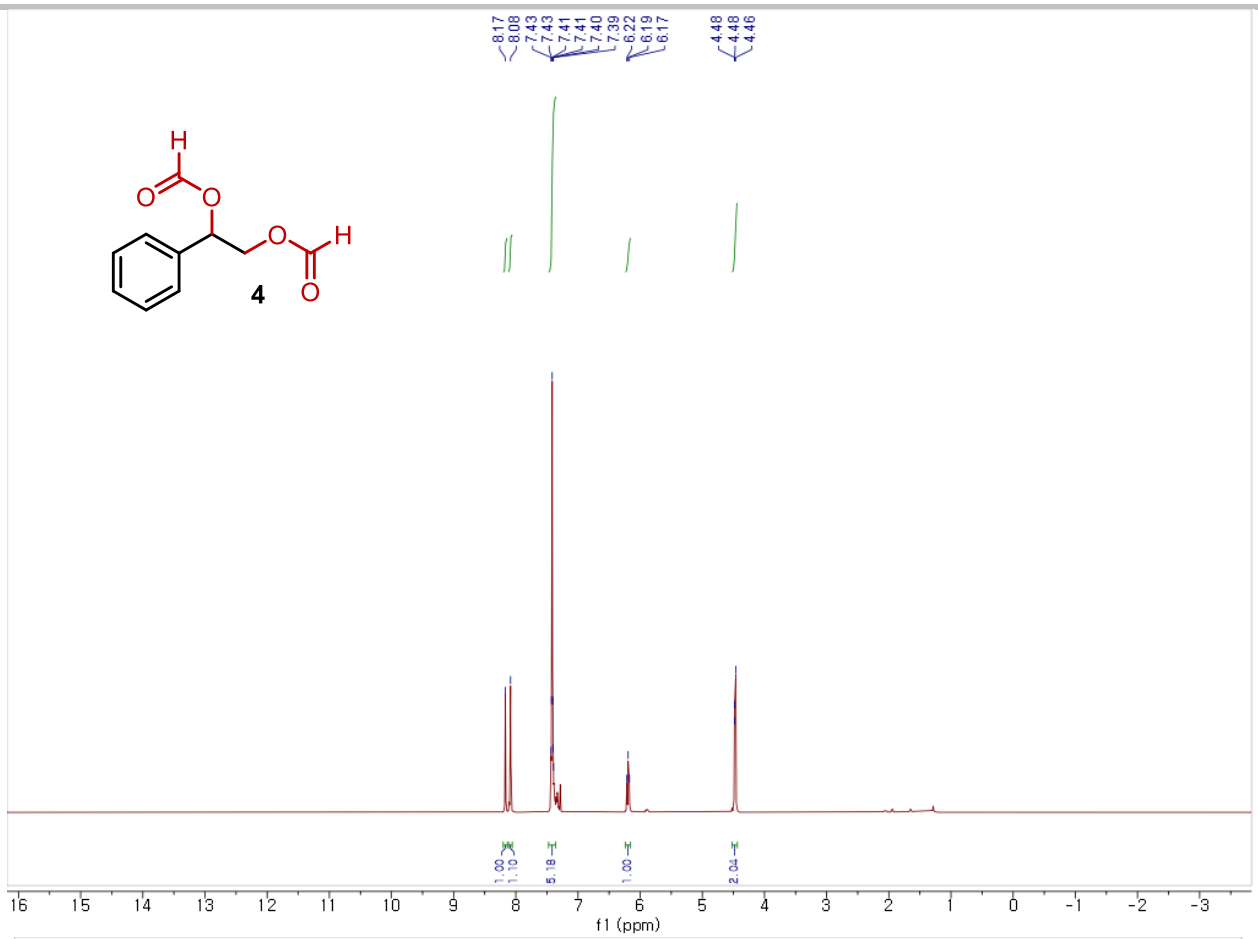
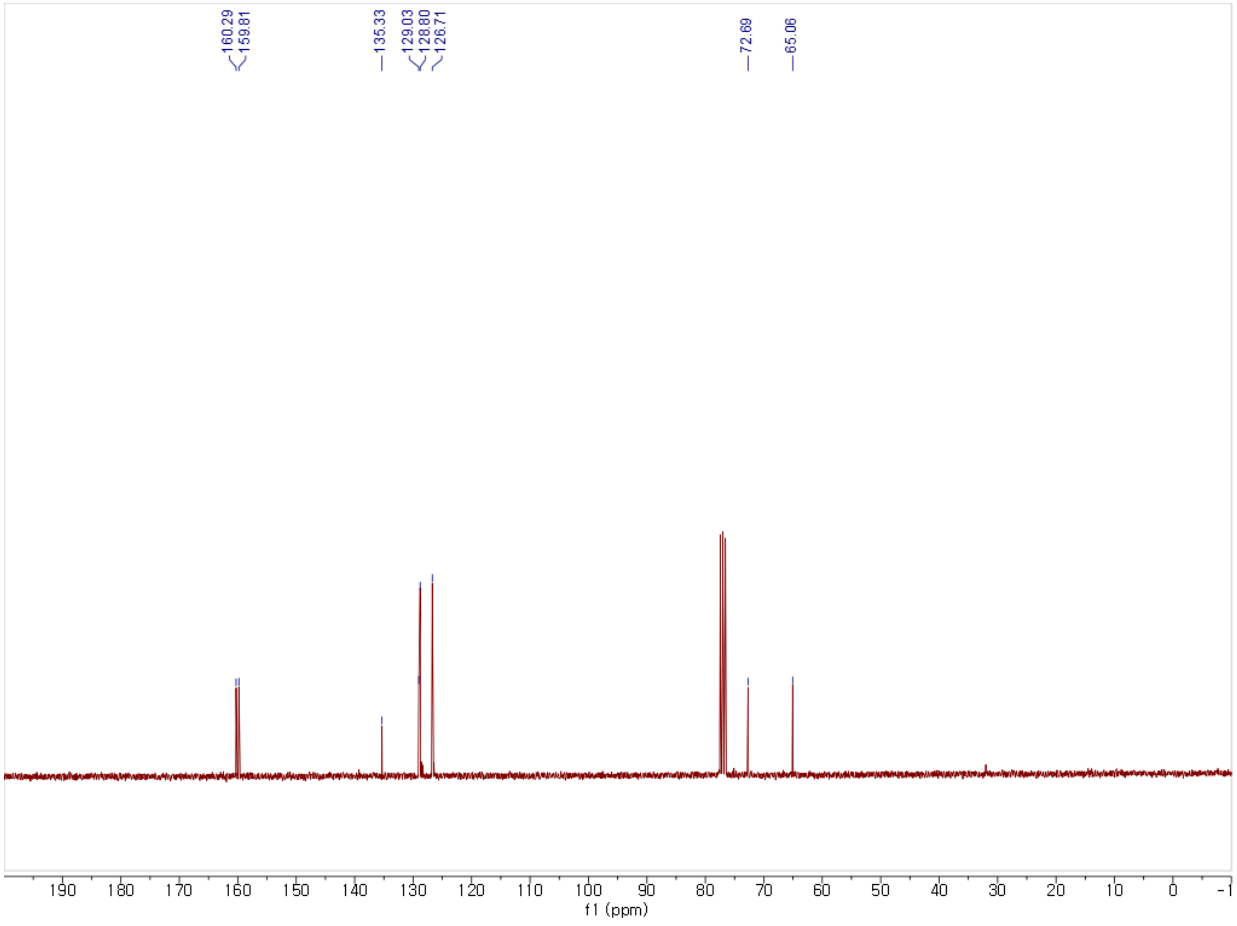
159.88
139.02
132.37
129.49
126.72
72.66
65.08
21.18
f1 (ppm)


8.17
8.08
7.43
7.43
7.41
7.41
7.40
7.39
6.22
6.19
6.17
4.48
4.48
4.46
1.00
1.10
5.18
1.00
2.04
f1 (ppm)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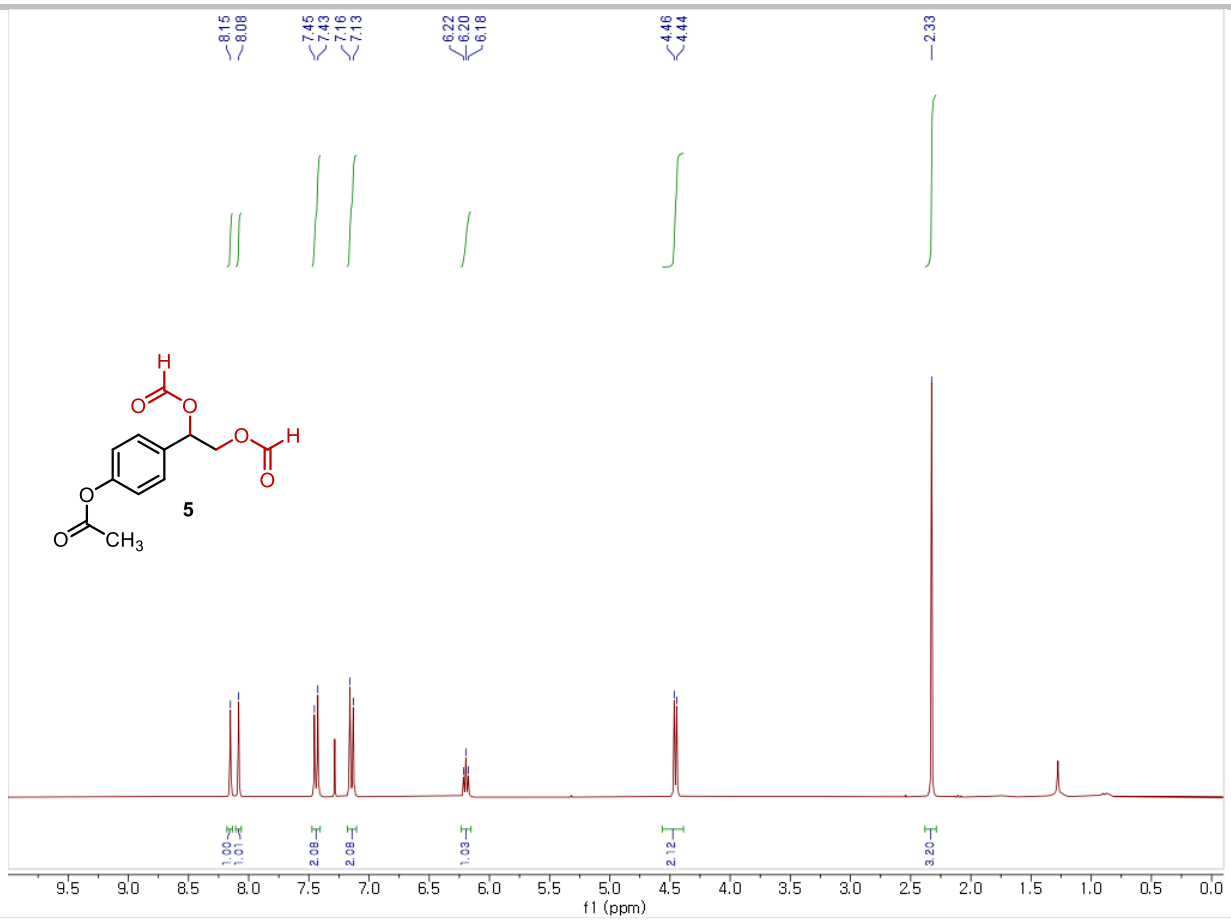
159.81
135.33
129.03
128.80
126.71
72.69
65.06
f1 (ppm)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8.08
7.45
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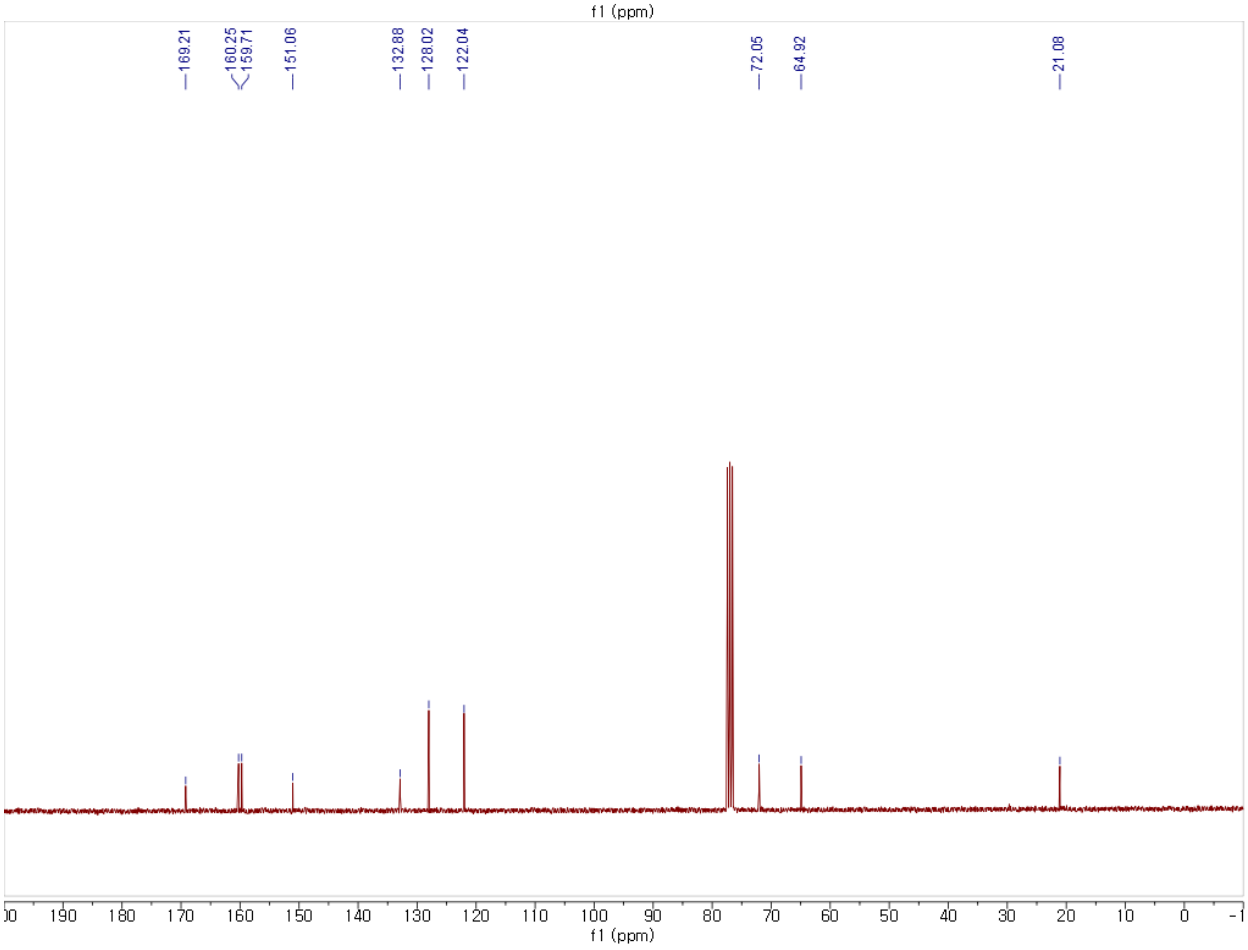
7.16
7.13
6.22
6.20
6.18
4.46
4.44
2.33
5
f1 (ppm)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160.25
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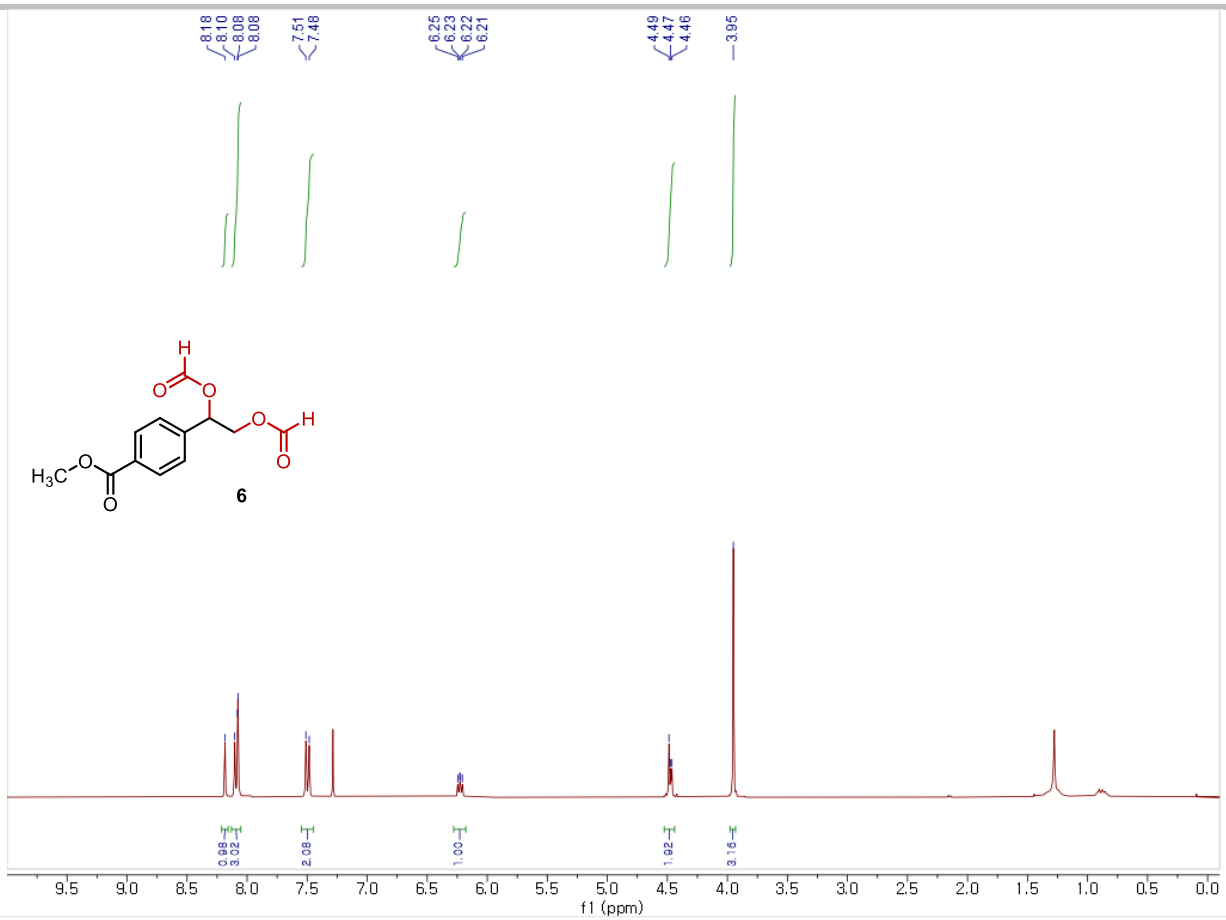
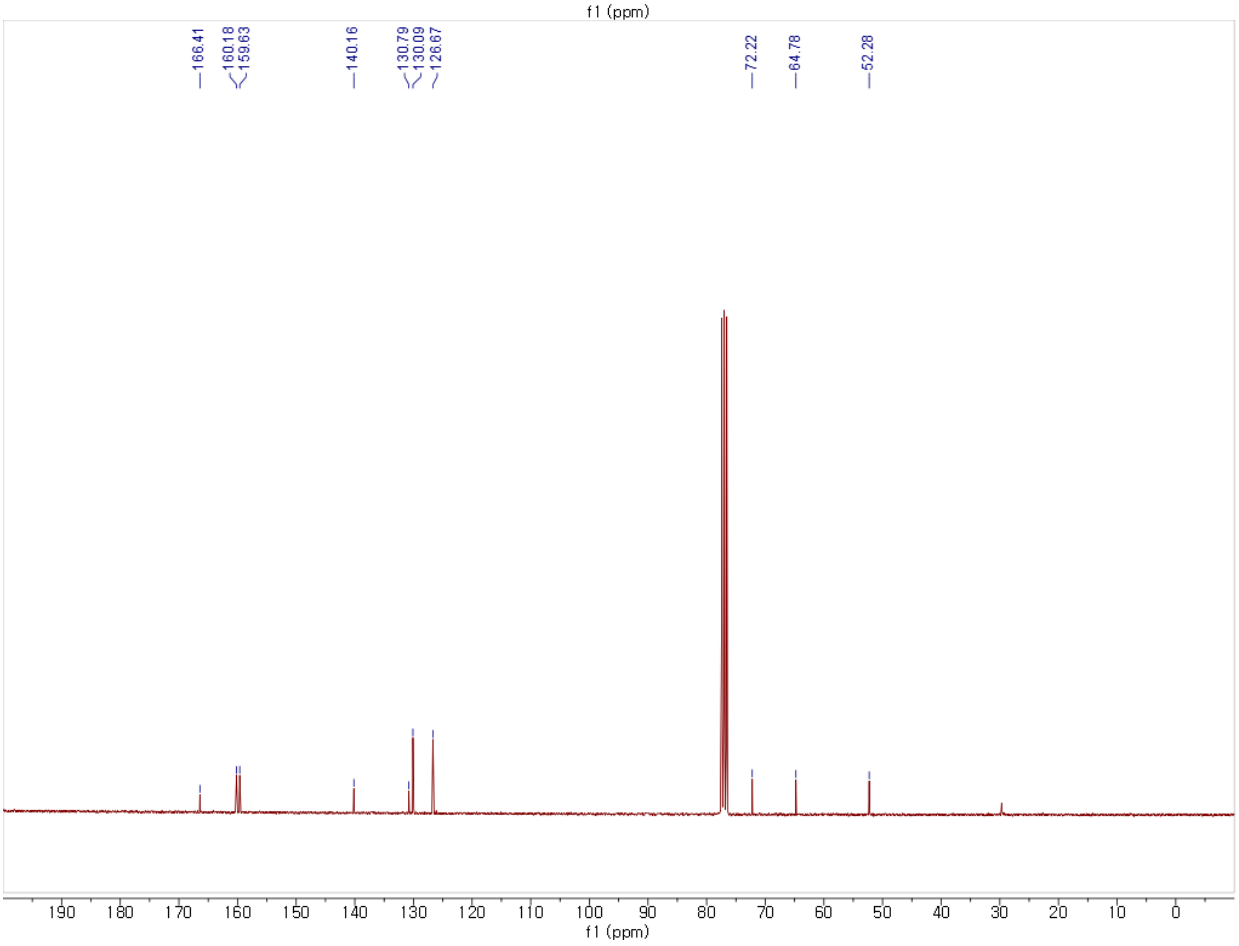
132.88
128.02
122.04
72.05
64.92
21.08
f1 (ppm)


8.18
8.10
8.08
8.08
7.51
7.48
6.25
6.23
6.22
6.21
4.49
4.47
4.46
3.95
H
O
O
O
H
O
H3C
O
O
6
0.98
3.02
2.08
1.00
1.92
3.16
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
f1 (ppm)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160.18
159.63
140.16
130.79
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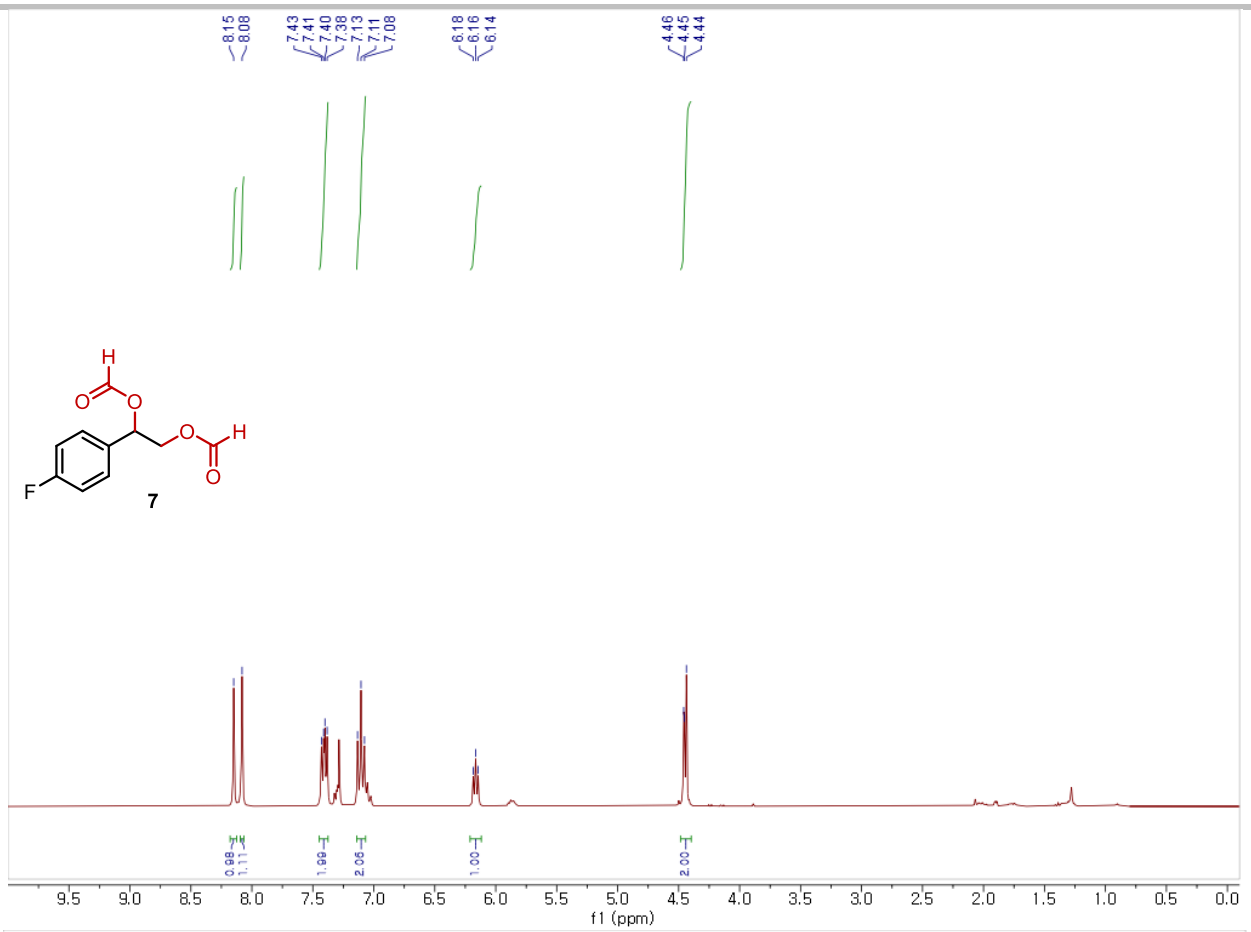
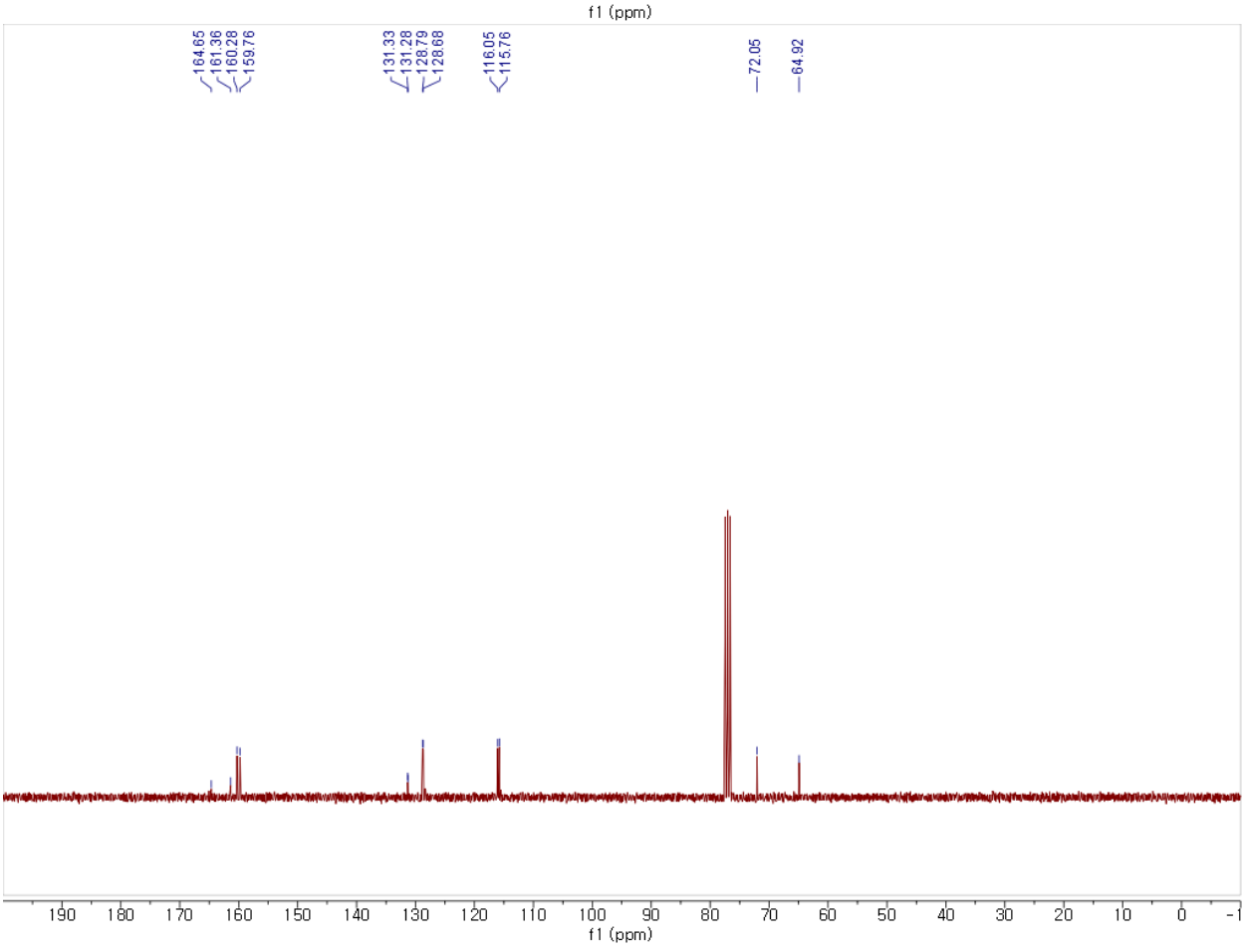
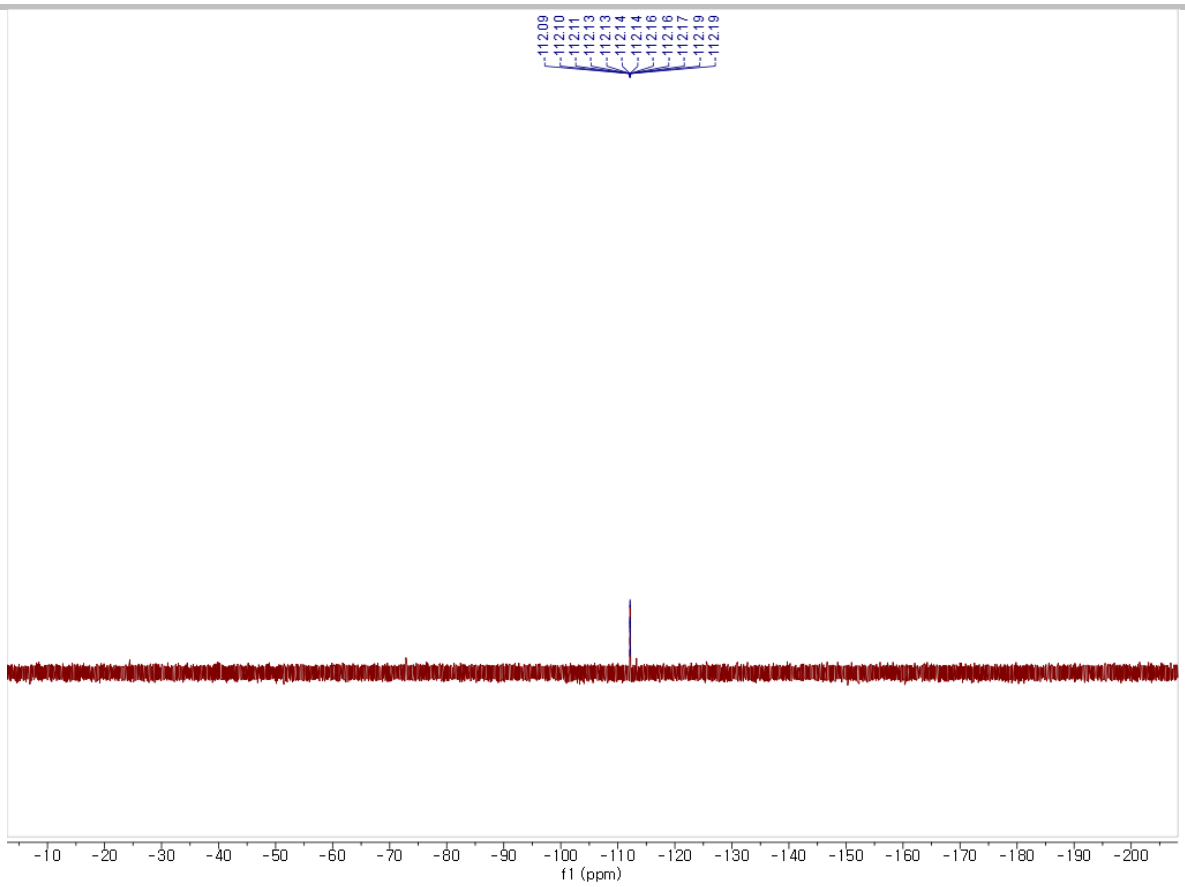
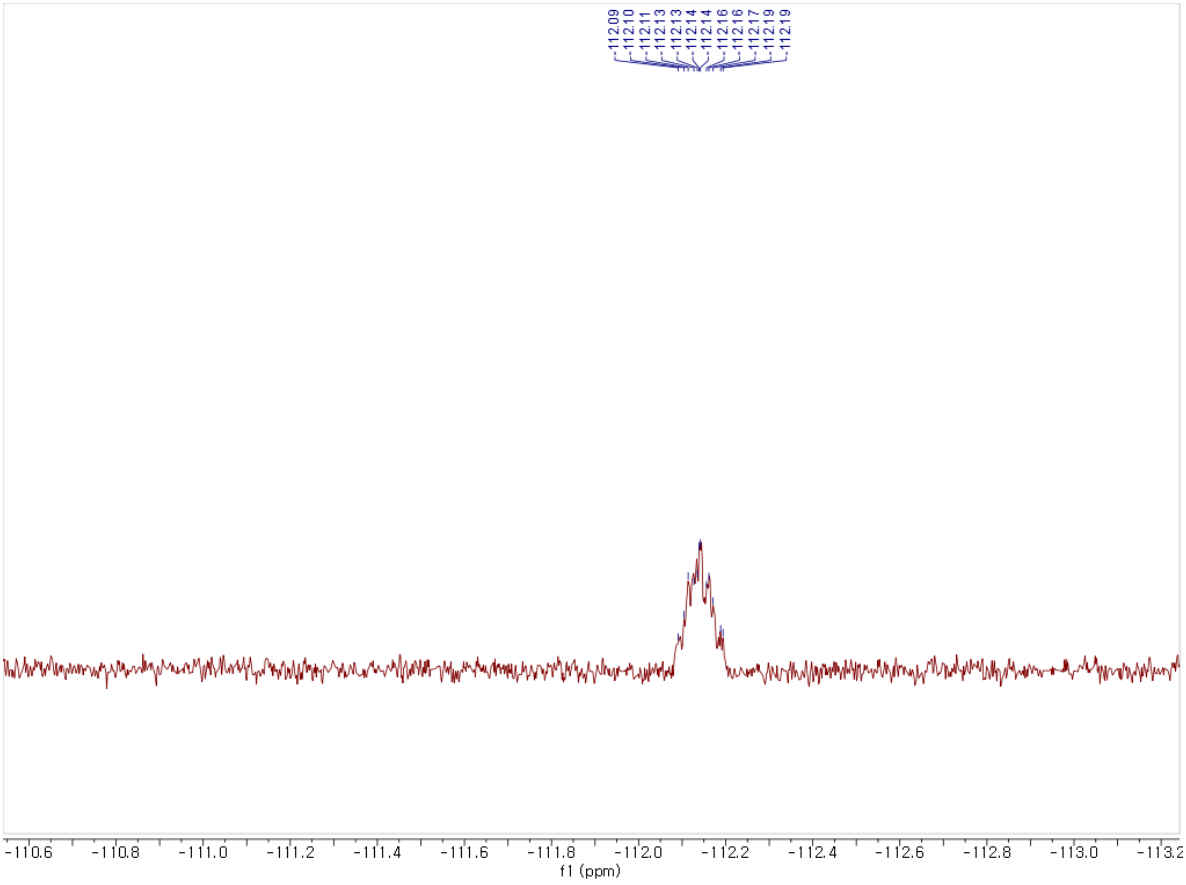
126.67
72.22
64.78
52.28
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.15
8.08
7.43
7.41
7.40
7.38
7.13
7.11
7.08
6.18
6.16
6.14
4.46
4.45
4.44
H
O
O
O
H
O
F
7
0.98
1.11
1.99
2.06
1.00
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
f1 (ppm)


164.65
161.36
160.28
159.76
131.33
131.28
128.79
128.68
116.05
115.76
72.05
64.92
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)


-112.09
-112.10
-112.11
-112.13
-112.13
-112.14
-112.16
-112.16
-112.17
-112.19
-112.19
-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)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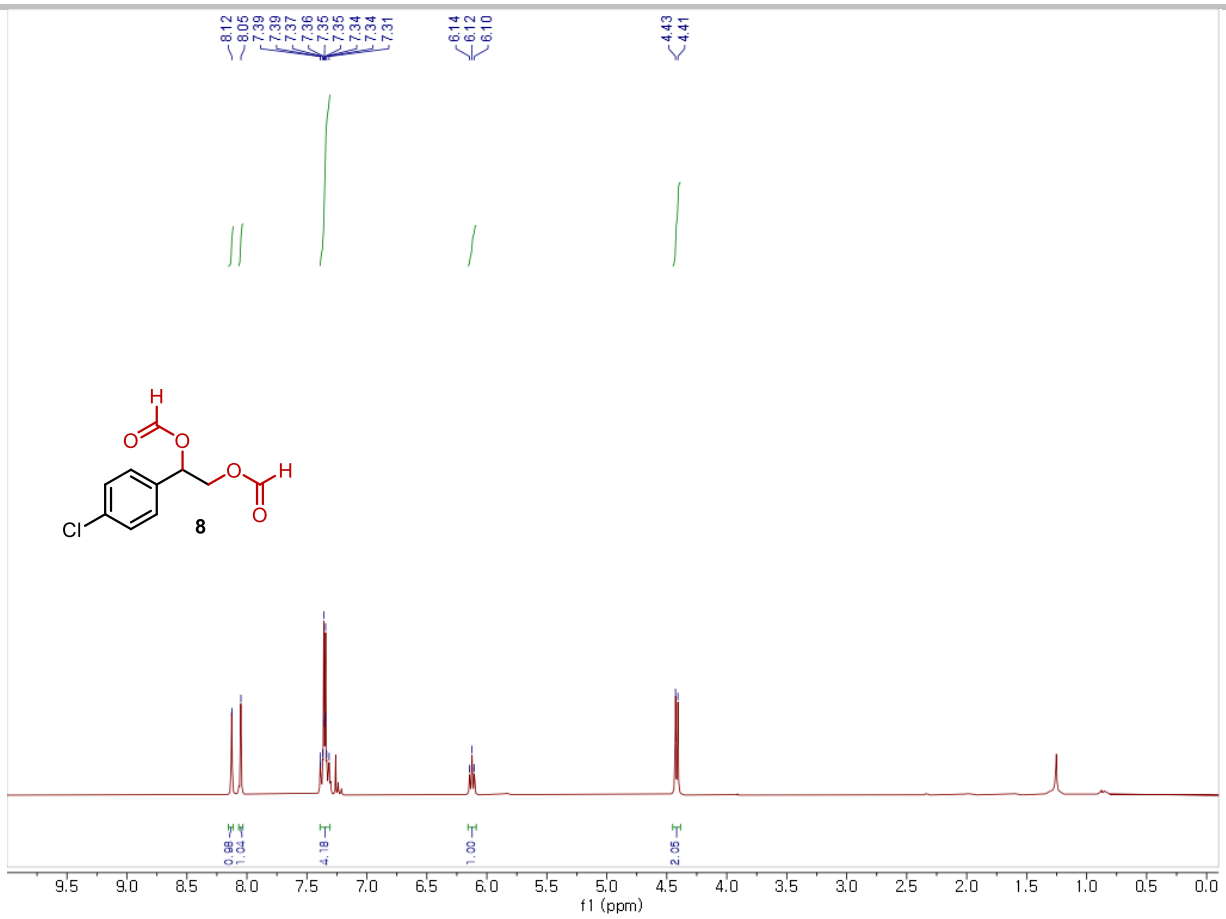
-112.10
-112.11
-112.13
-112.13
-112.14
-112.16
-112.16
-112.17
-112.19
-112.19
-110.6 -110.8 -111.0 -111.2 -111.4 -111.6 -111.8 -112.0 -112.2 -112.4 -112.6 -112.8 -113.0 -113.2
f1 (ppm)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7.35
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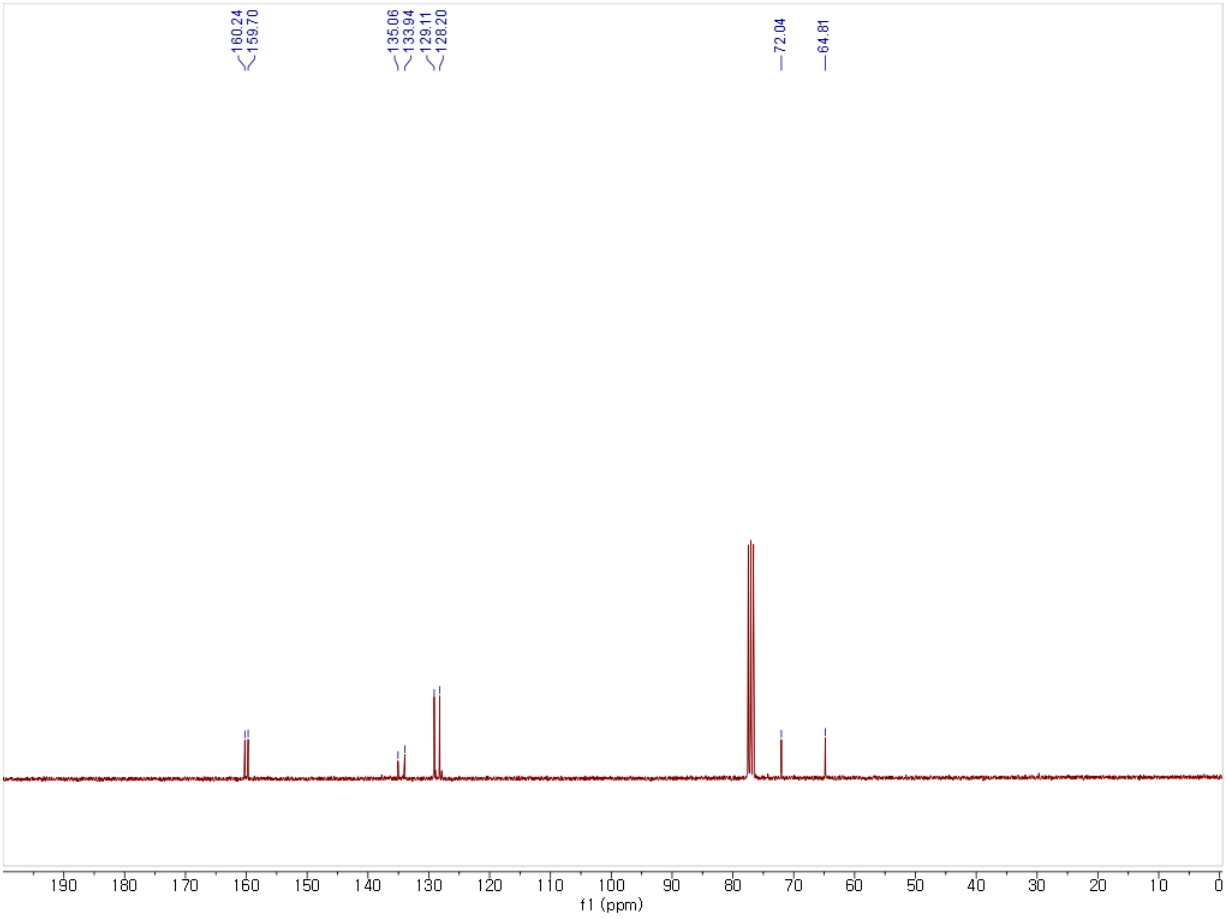
7.34
7.31
6.14
6.12
6.10
4.43
4.41
H
O
O
O
H
O
Cl
8
0.88
1.04
4.18
1.00
2.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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159.70
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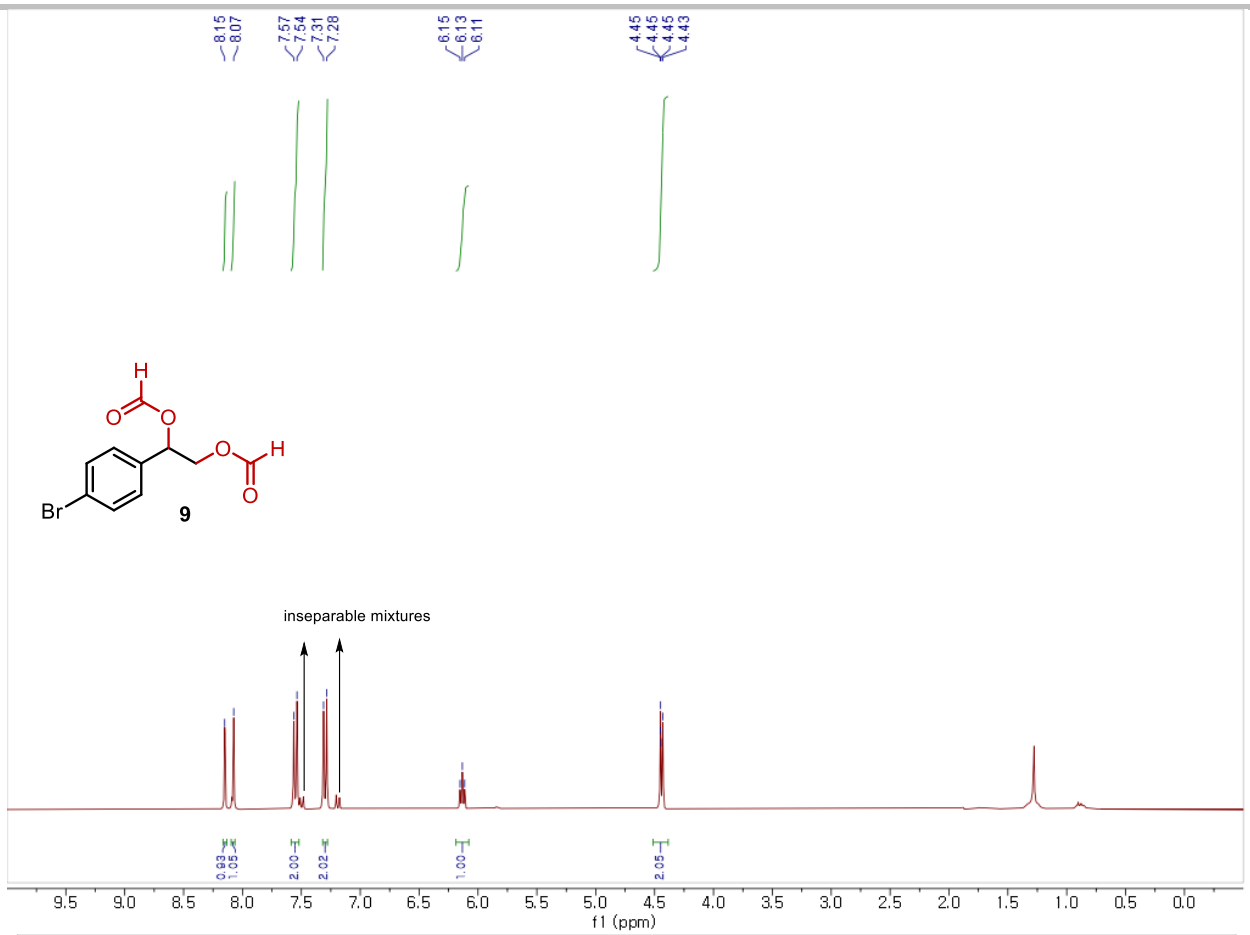
129.11
128.20
72.04
64.81
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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8.07
7.57
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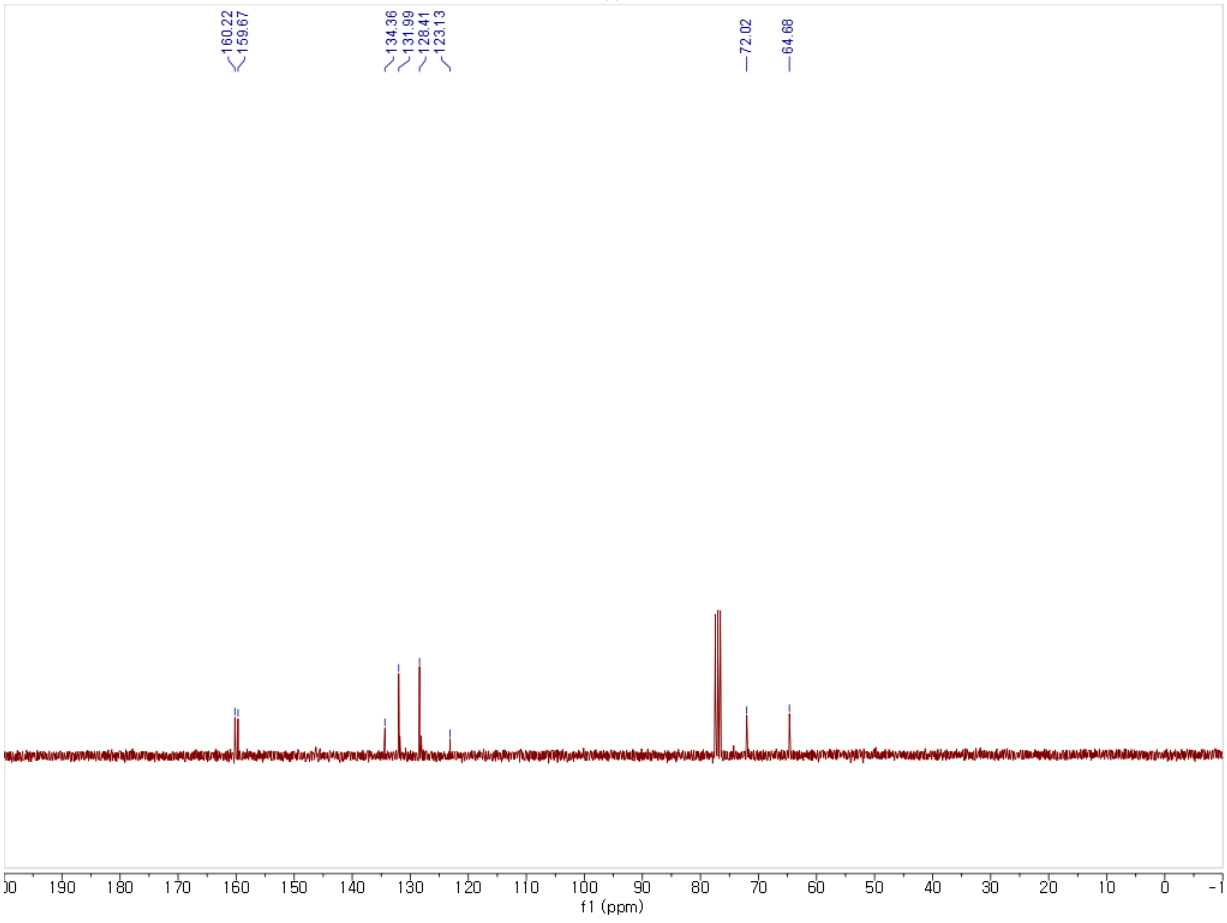
7.31
7.28
6.15
6.13
6.11
4.45
4.45
4.45
4.43
H
O
O
O
H
O
Br
9
inseparable mixtures
0.83
1.05
2.00
2.02
1.00
2.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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159.67
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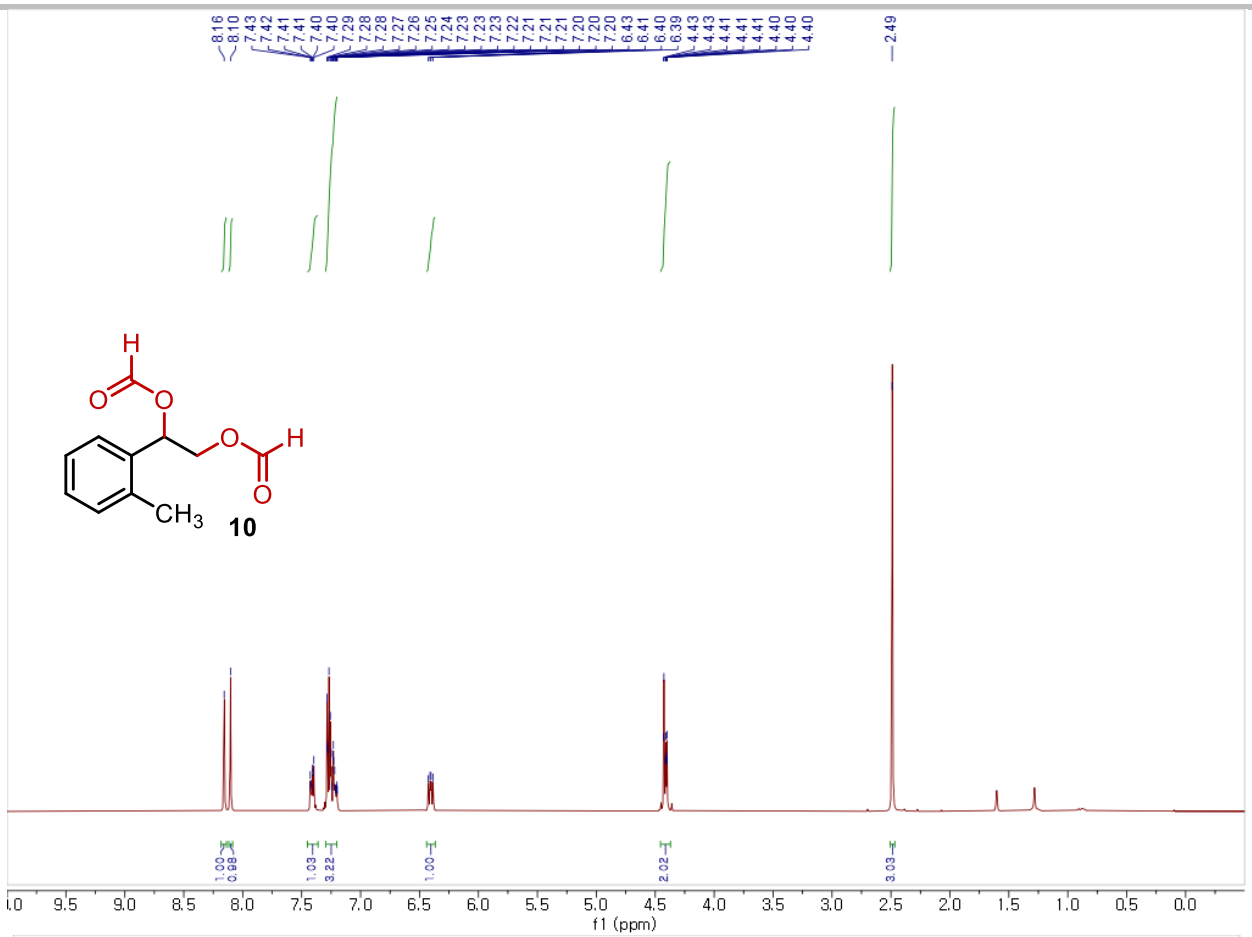
128.41
123.13
72.02
64.68
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)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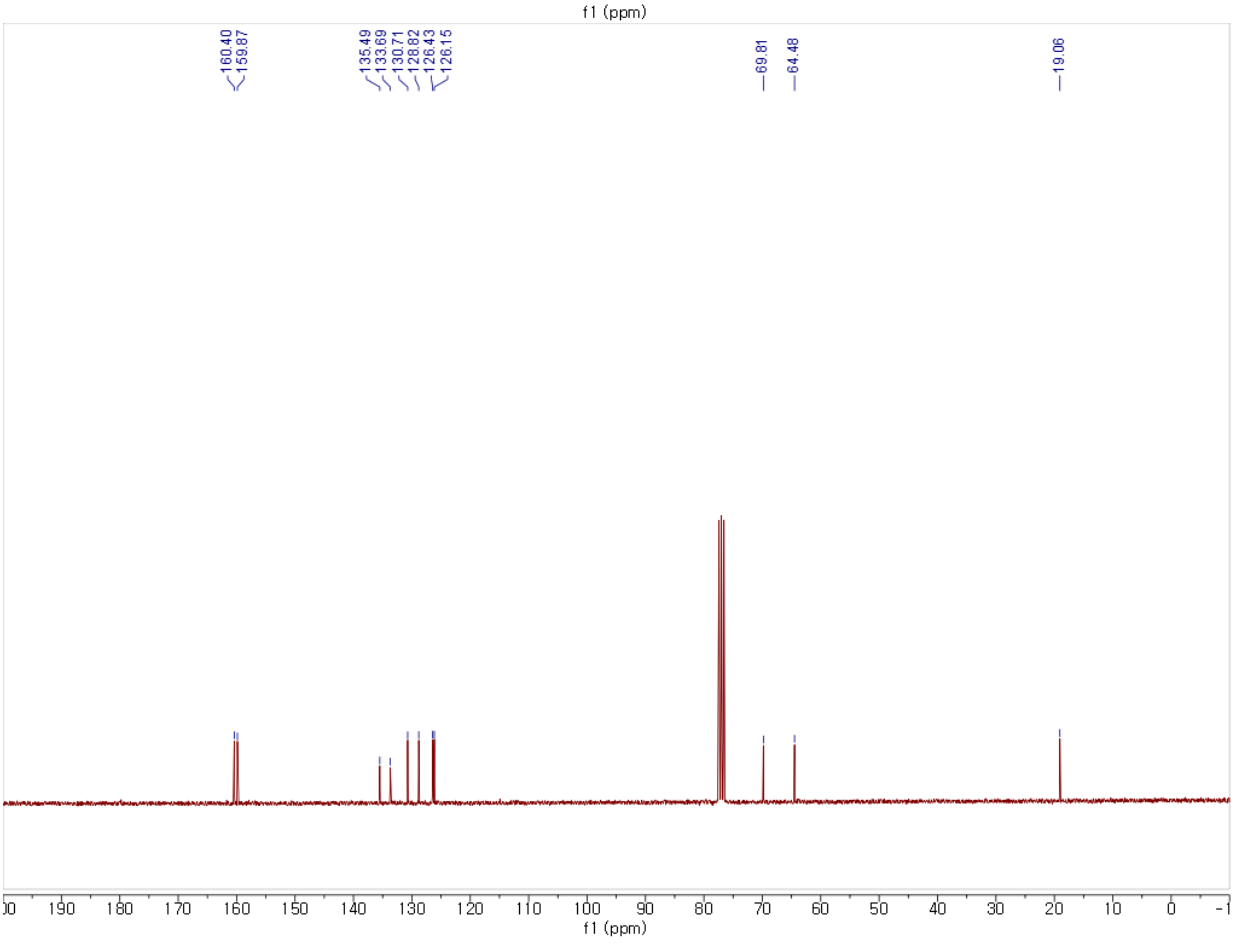
7.26
7.25
7.24
7.23
7.23
7.22
7.21
7.21
7.20
7.20
6.43
6.41
6.40
6.39
4.43
4.41
4.41
4.40
4.40
4.40
2.49
H
O
O
O
H
O
CH3
10
1.00
0.98
1.03
3.22
1.00
2.02
3.03
f1 (ppm)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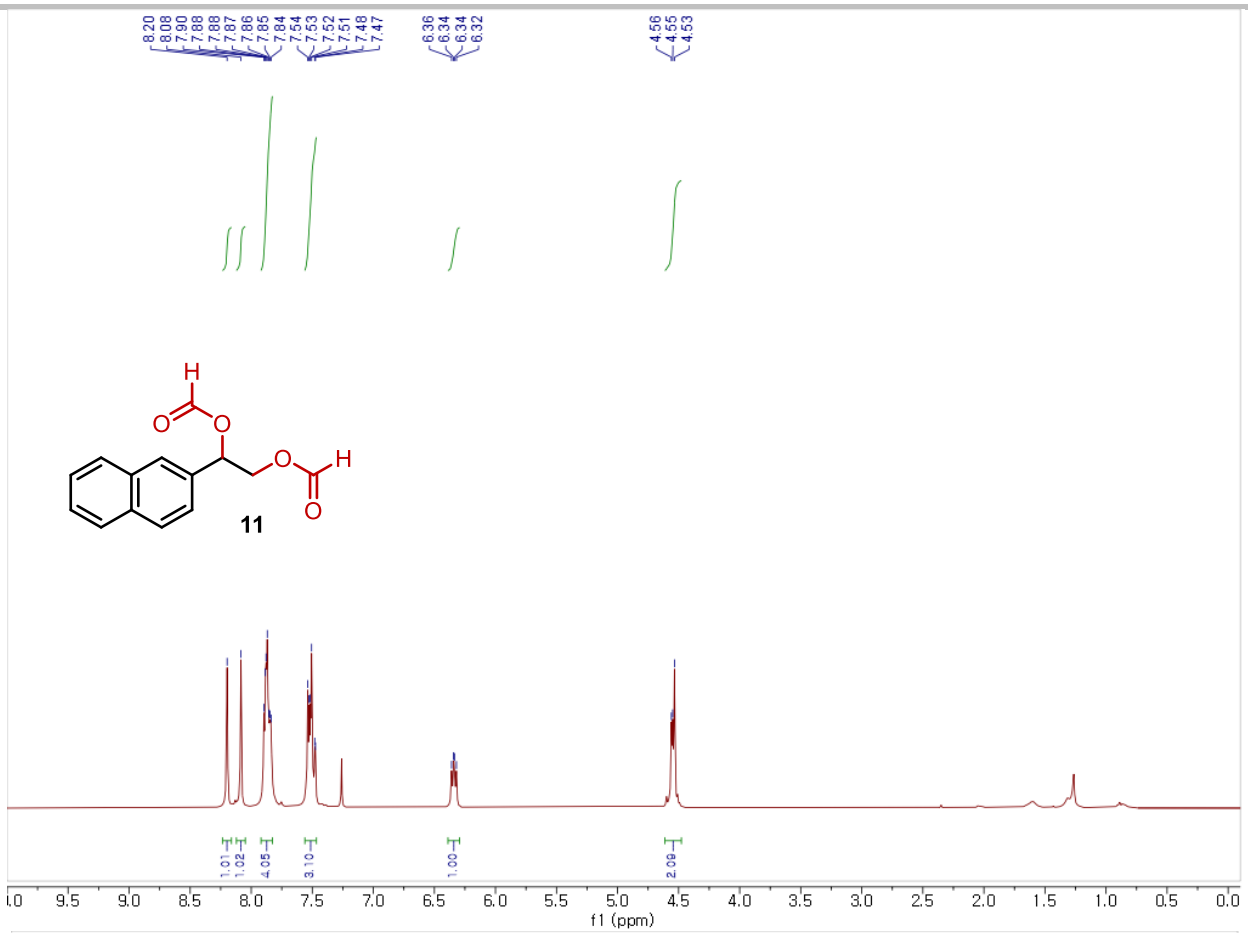
64.48
19.06
f1 (ppm)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7.52
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7.48
7.47
6.36
6.34
6.34
6.32
4.56
4.55
4.53
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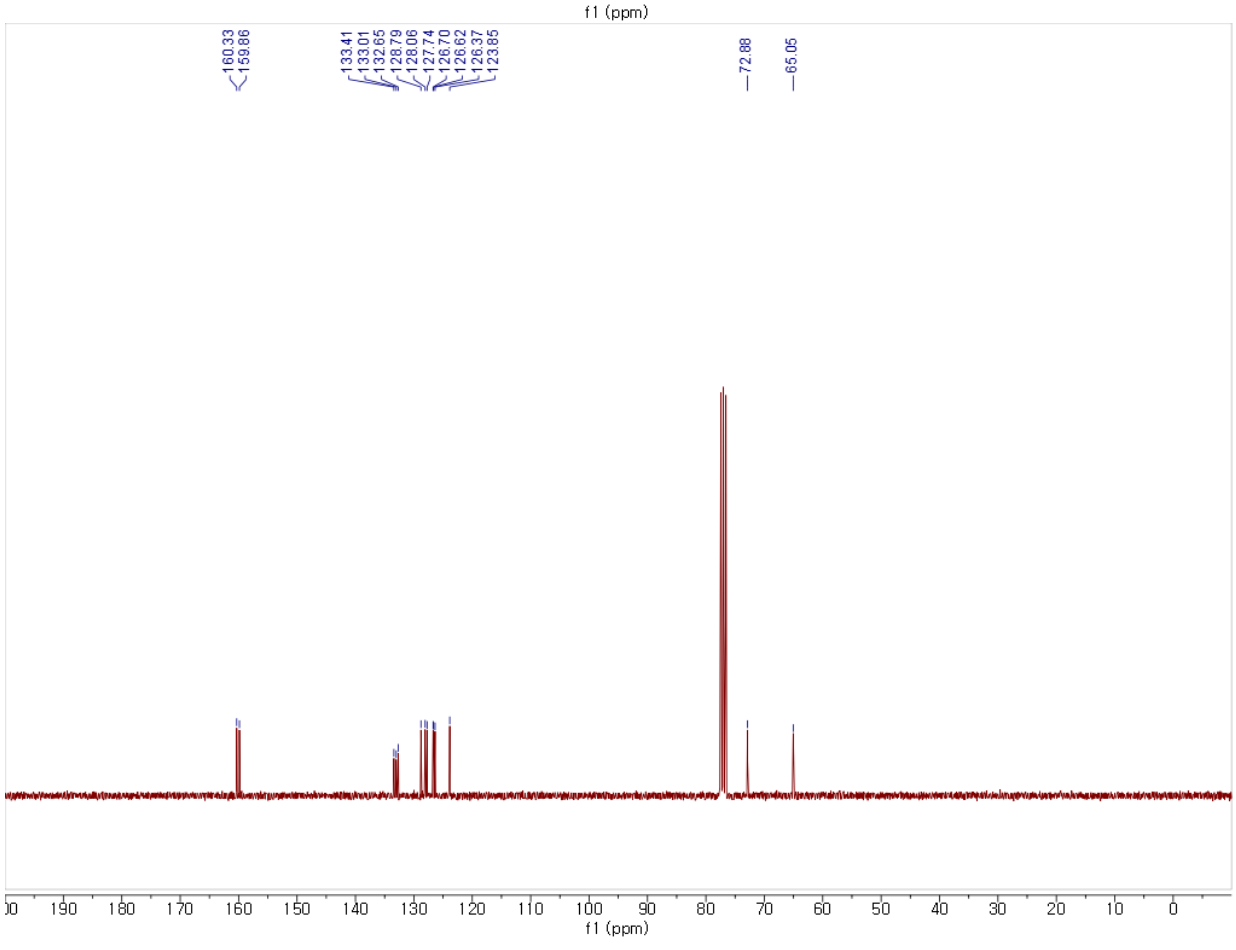
O
O
O
H
11
1.01
1.02
4.05
3.10
1.00
2.09
f1 (ppm)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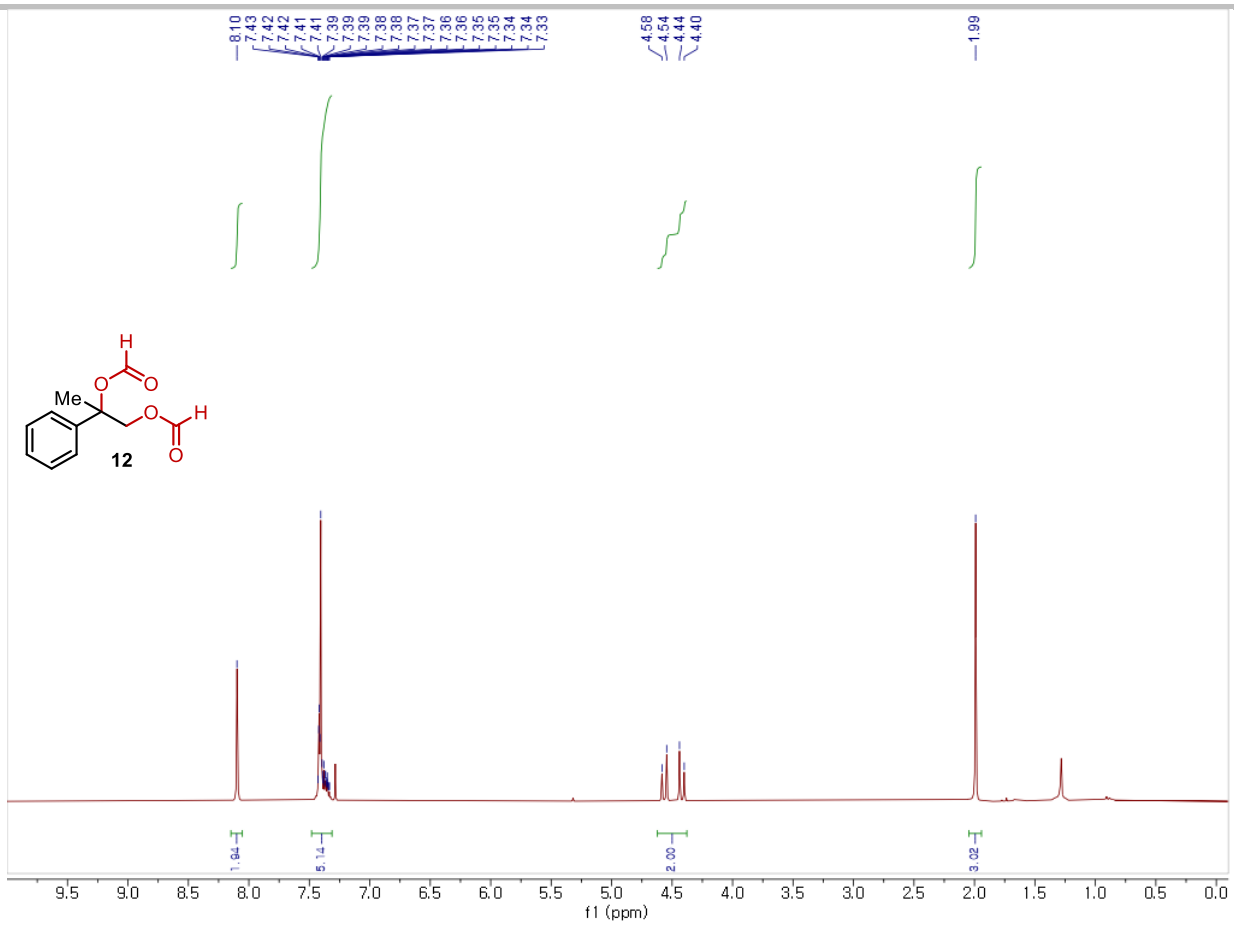
65.05
f1 (ppm)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7.37
7.36
7.36
7.35
7.35
7.34
7.34
7.33
4.58
4.54
4.44
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O
O
Me
O
O
H
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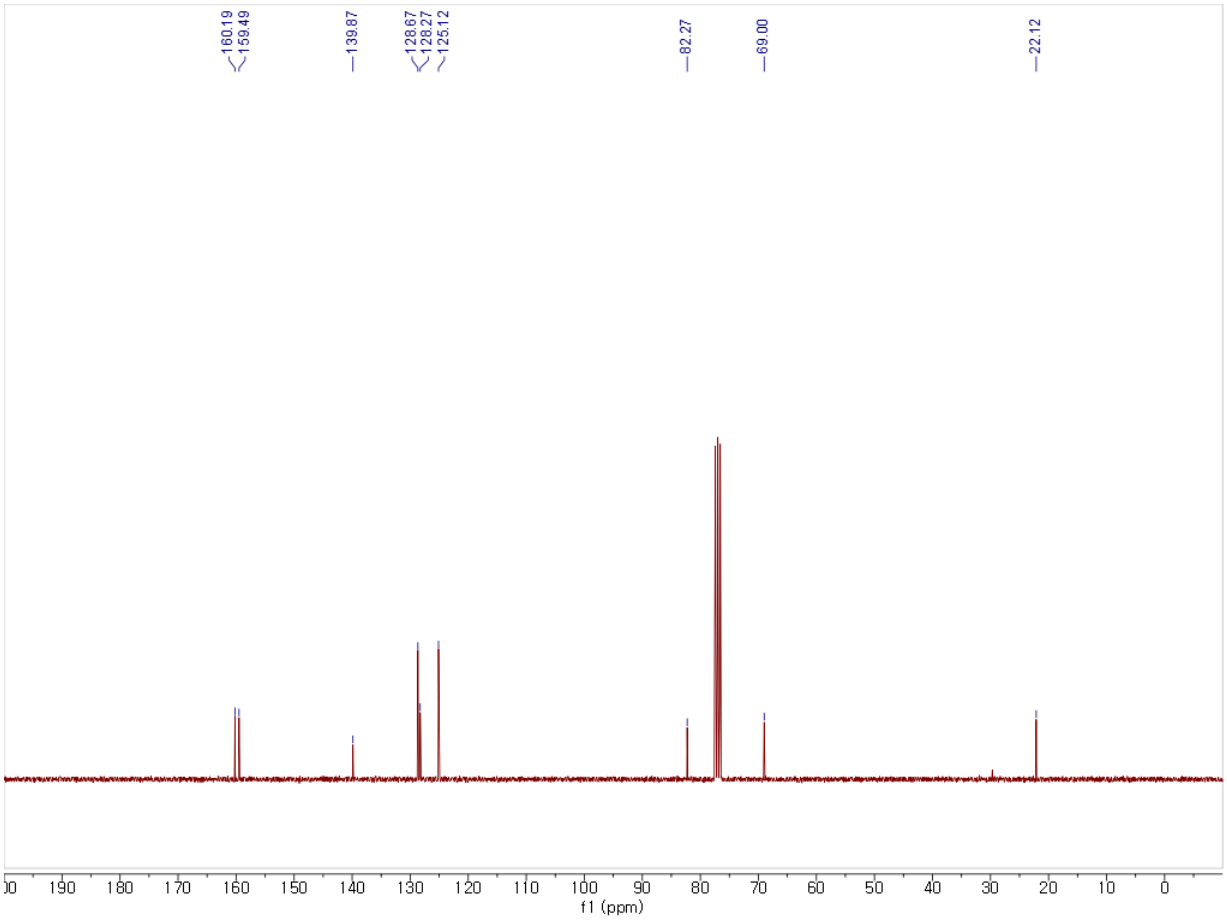
5.14
2.00
3.02
f1 (ppm)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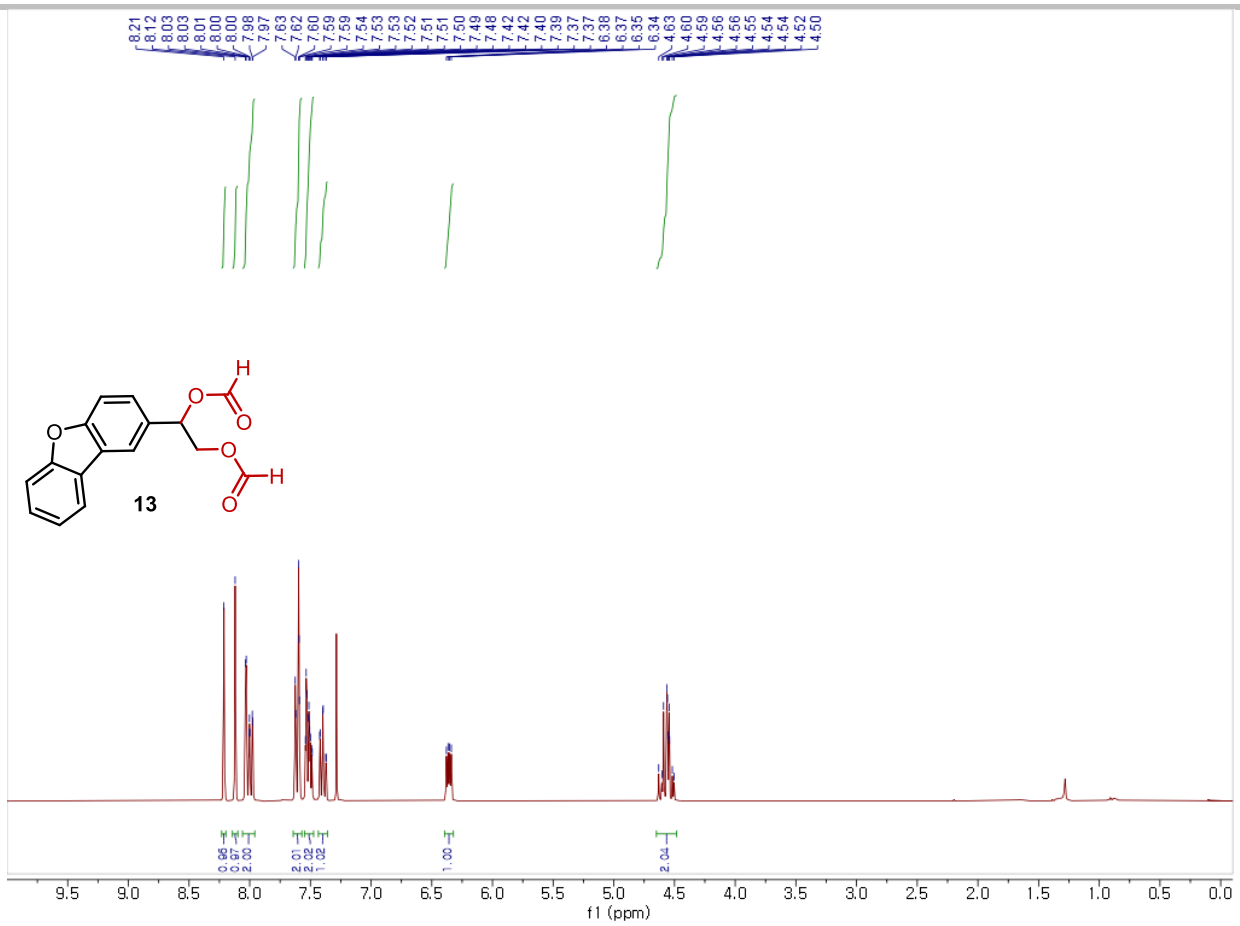
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8.03
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7.98
7.97
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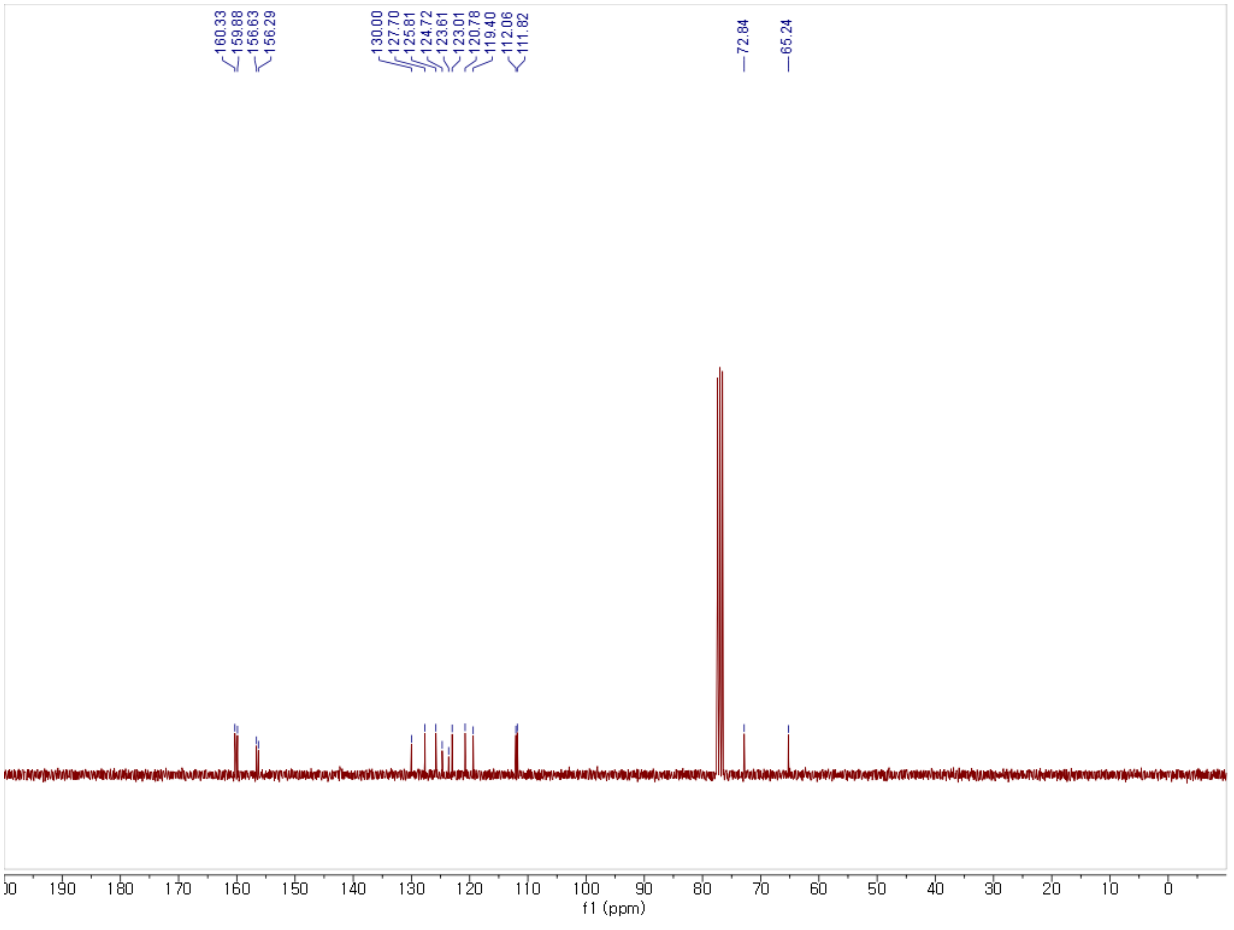
7.59
7.59
7.54
7.53
7.53
7.52
7.51
7.51
7.50
7.49
7.48
7.42
7.42
7.40
7.39
7.37
7.37
6.38
6.37
6.35
6.34
4.63
4.60
4.59
4.56
4.56
4.55
4.54
4.54
4.52
4.50
13
0.95
0.97
2.00
2.01
2.02
1.02
1.00
2.04
f1 (ppm)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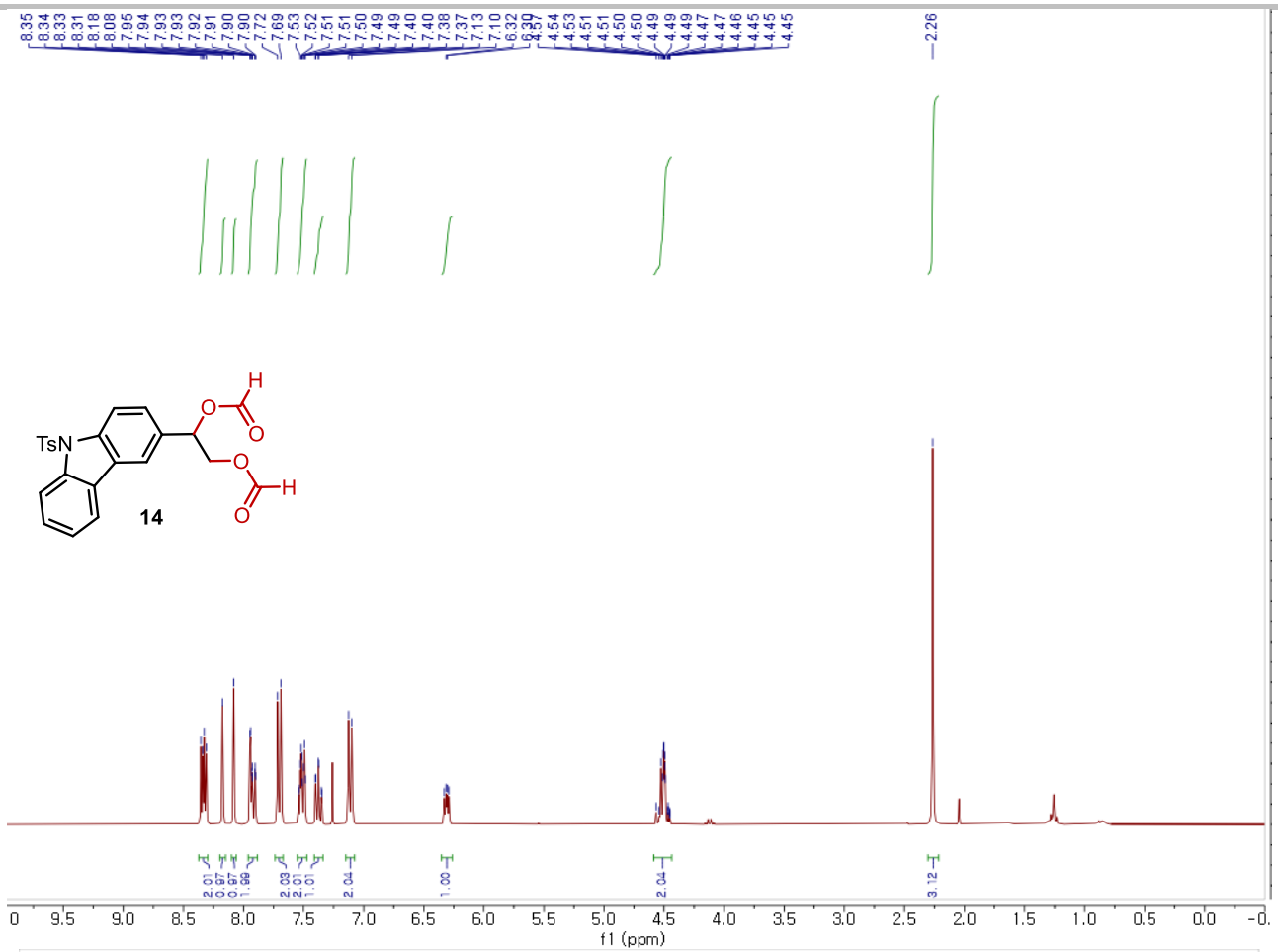
123.01
120.78
119.40
112.06
111.82
72.84
65.24
f1 (ppm)


TsN
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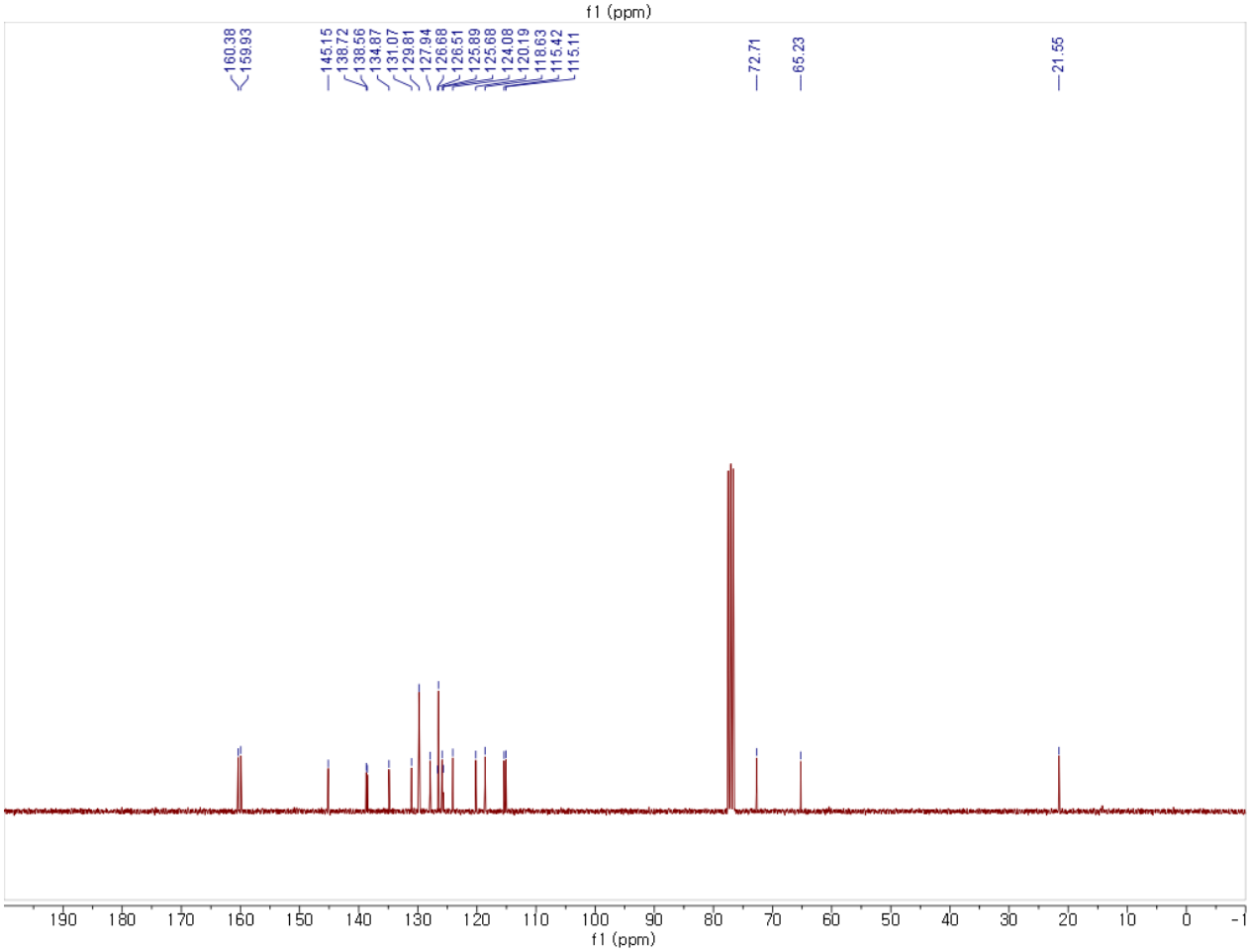
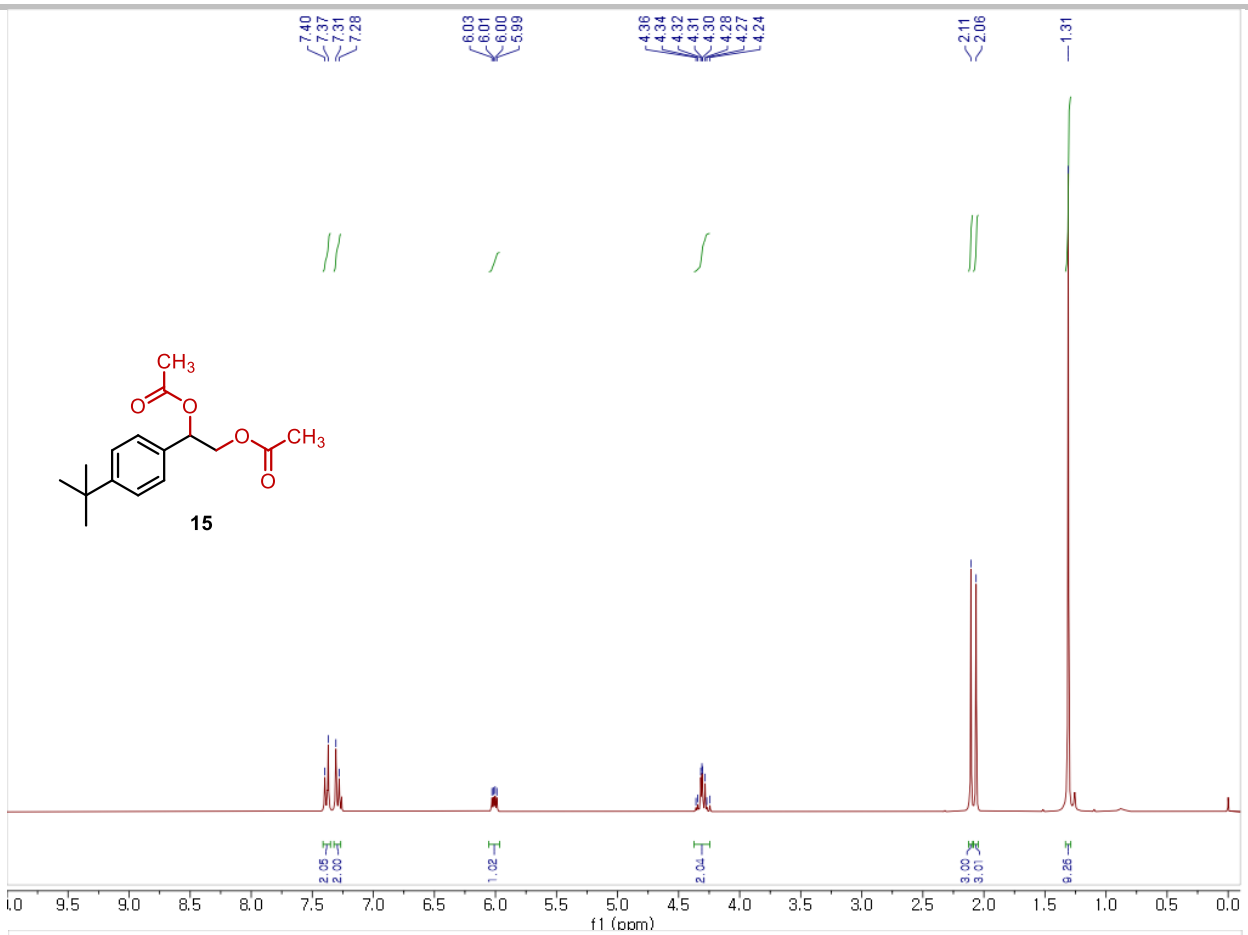

f1 (ppm)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6.01
6.00
5.99
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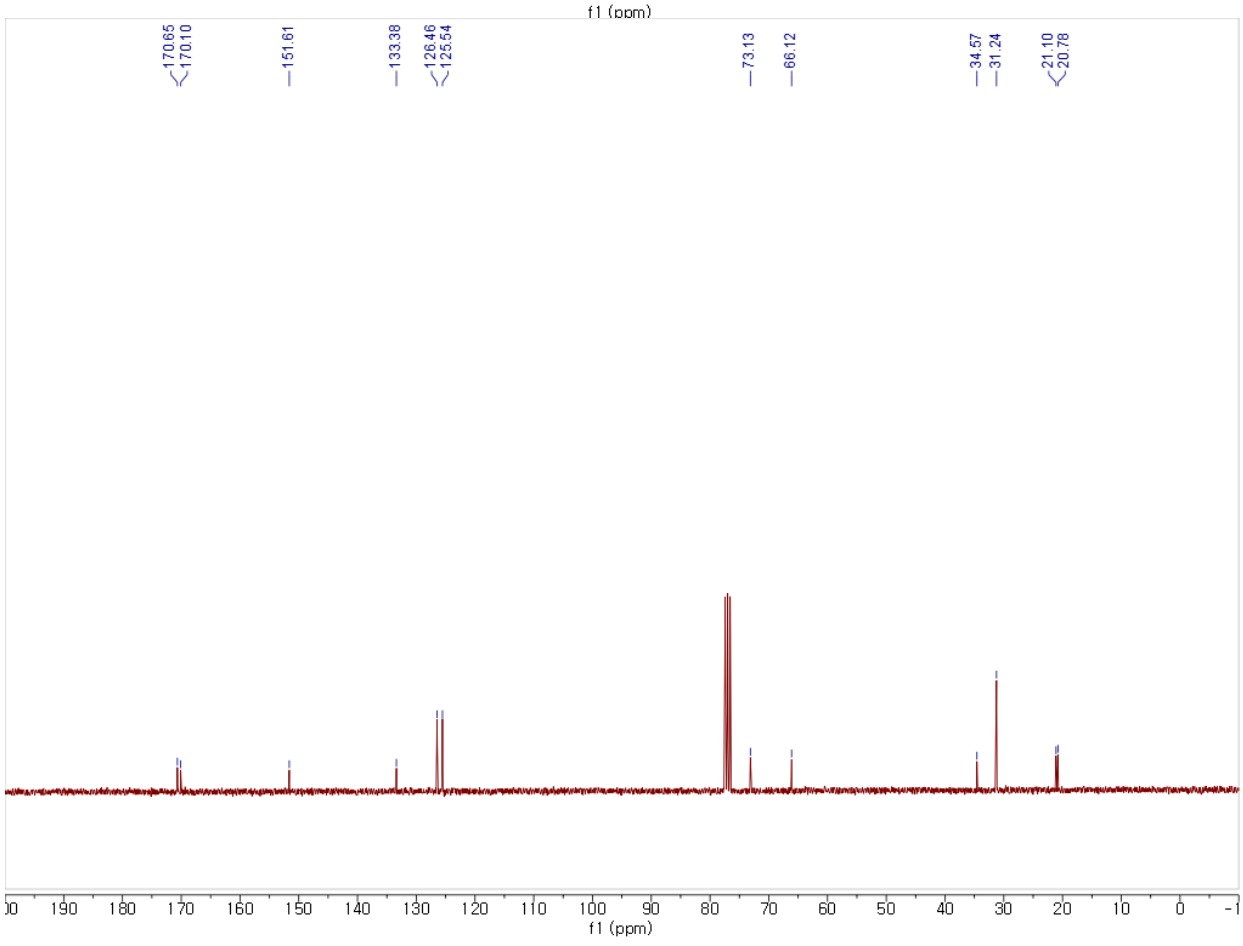
4.34
4.32
4.31
4.30
4.28
4.27
4.24
2.11
2.06
1.31
2.05
2.00
1.02
2.04
3.00
3.01
9.26
f1 (ppm)

15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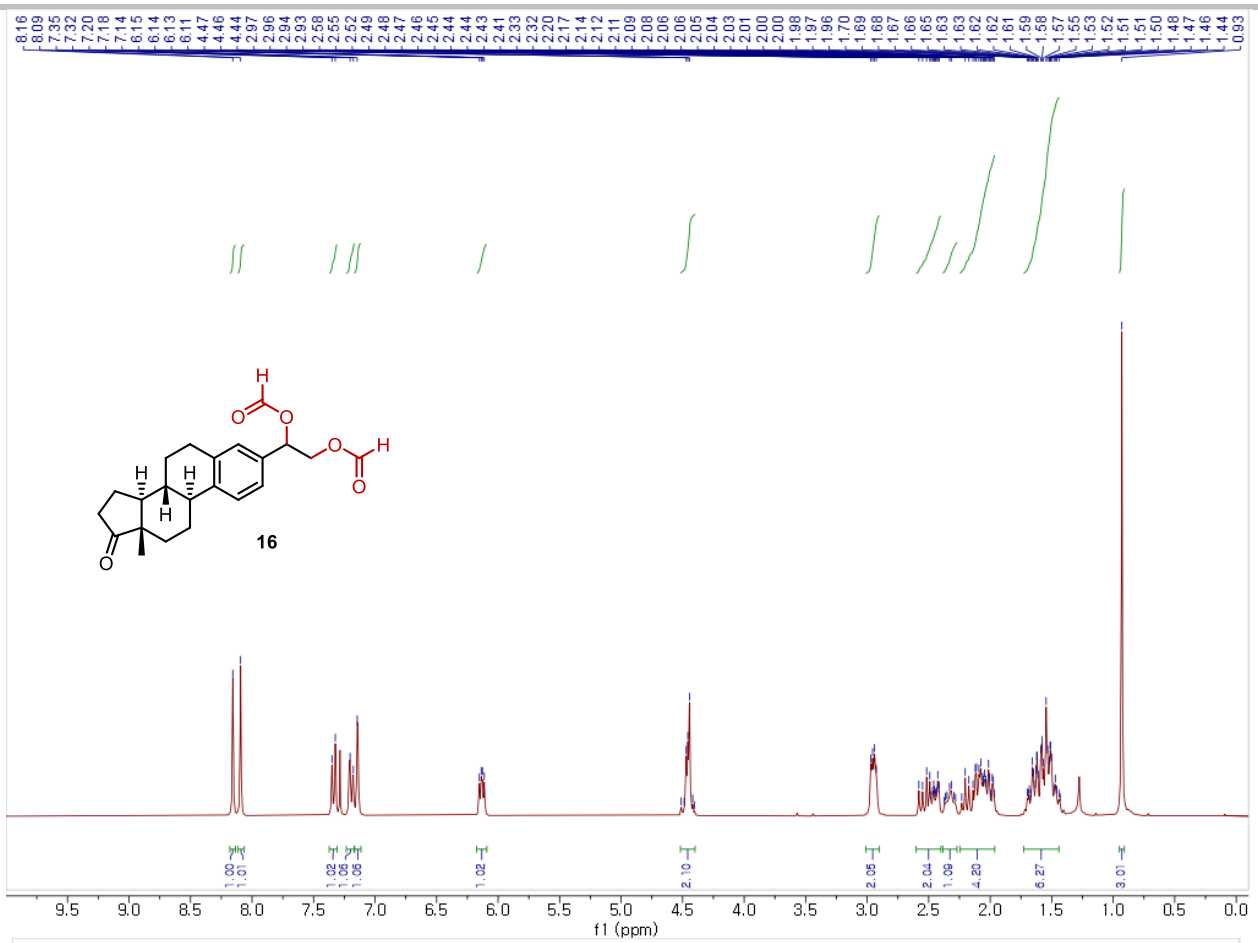
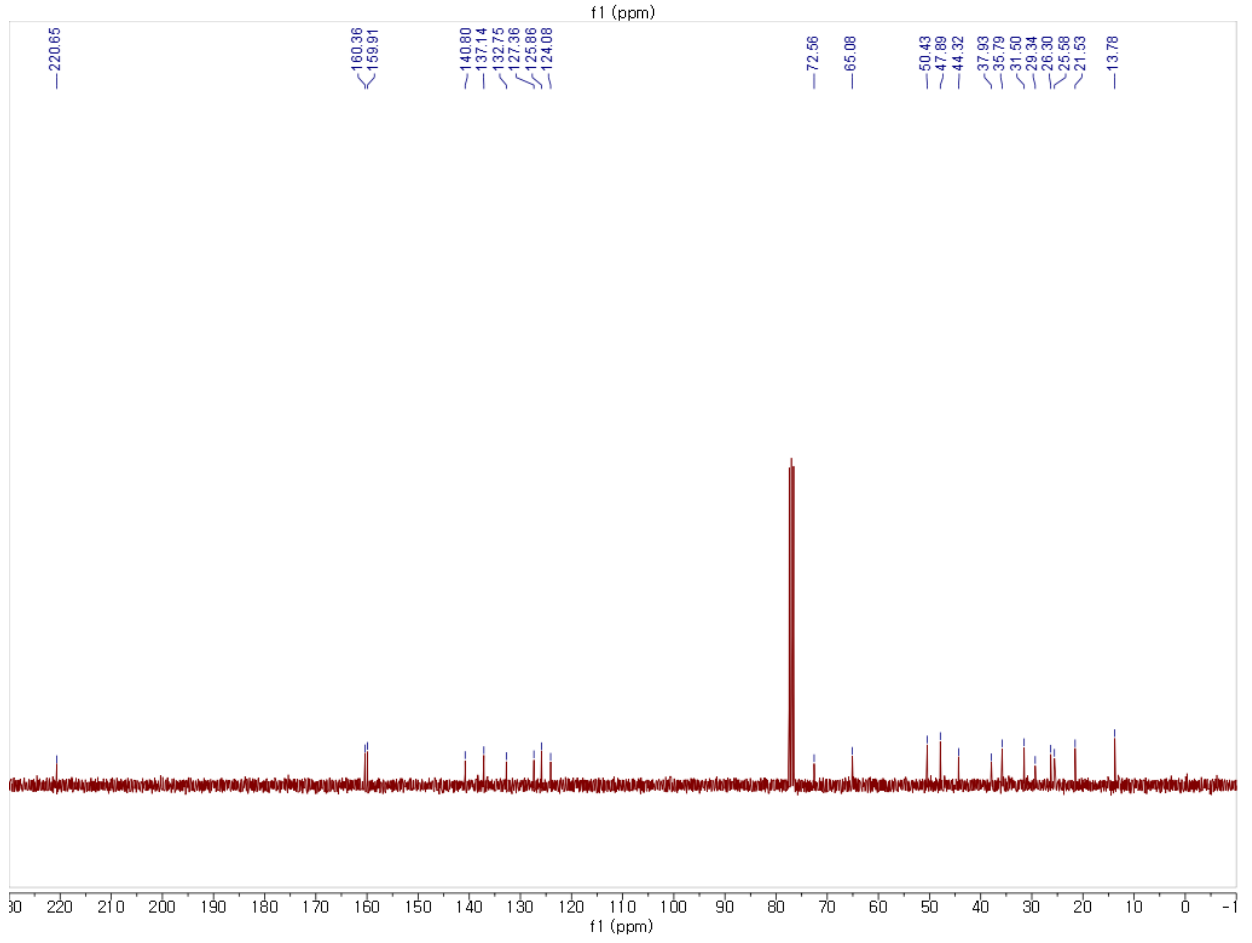
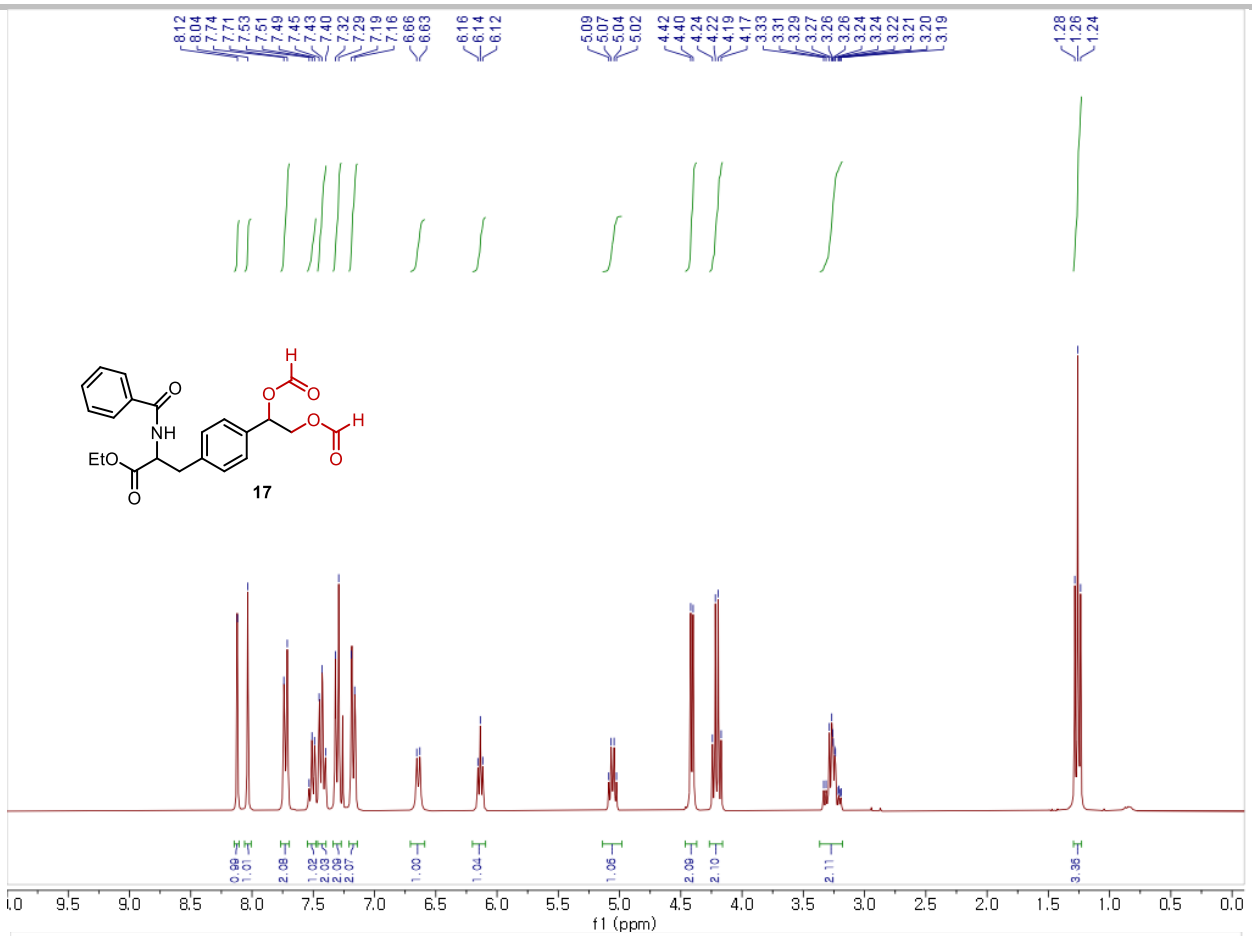
f1 (ppm)


16
f1 (ppm)


f1 (ppm)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7.45
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7.40
7.32
7.29
7.19
7.16
6.66
6.63
6.16
6.14
6.12
5.09
5.07
5.04
5.02
4.42
4.40
4.24
4.22
4.19
4.17
3.33
3.31
3.29
3.27
3.26
3.24
3.24
3.22
3.21
3.20
3.19
1.28
1.26
1.24
H
O
O
O
O
H
O
NH
EtO
O
17
0.99
1.01
2.98
1.02
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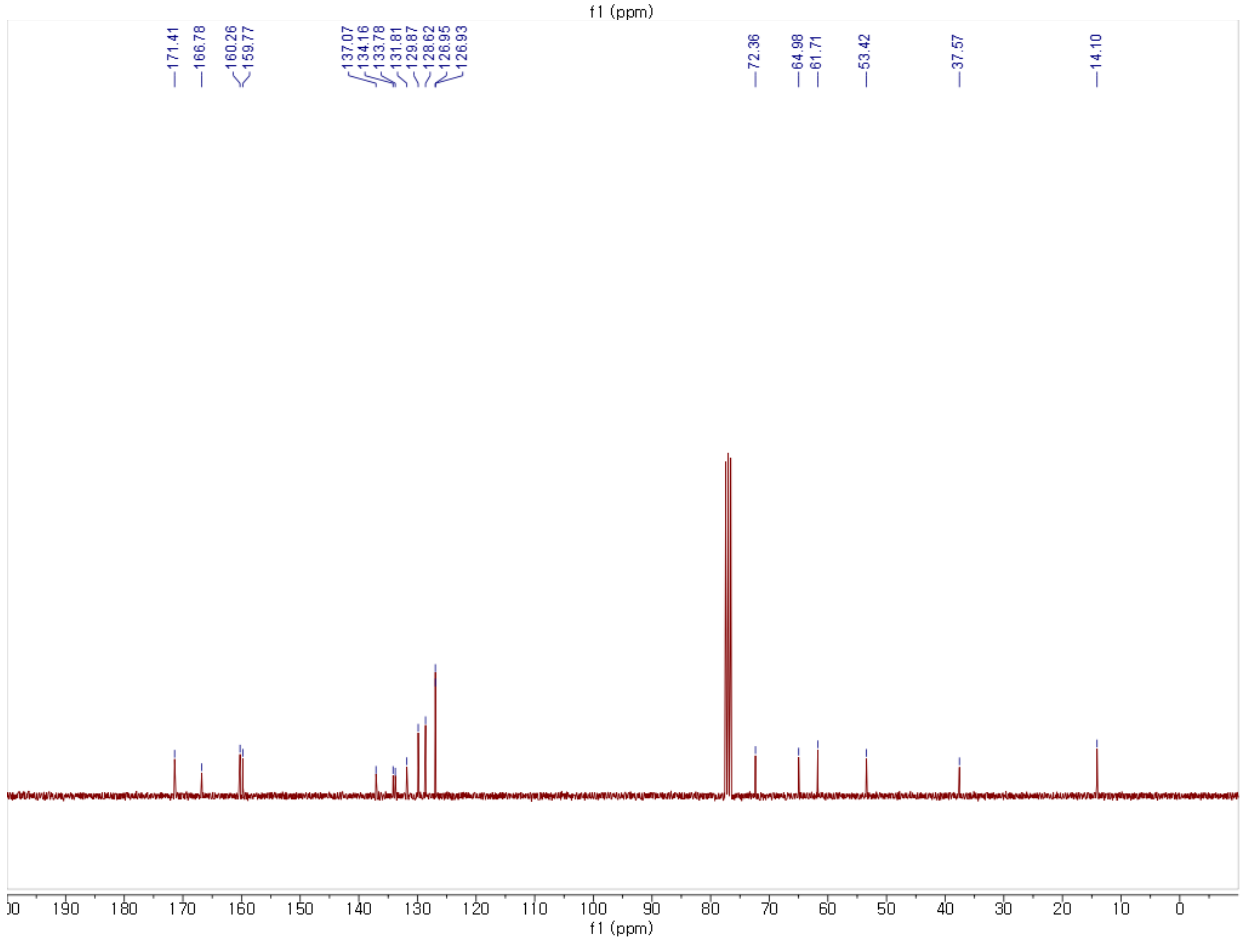
1.04
1.06
2.09
2.10
2.11
3.35
f1 (ppm)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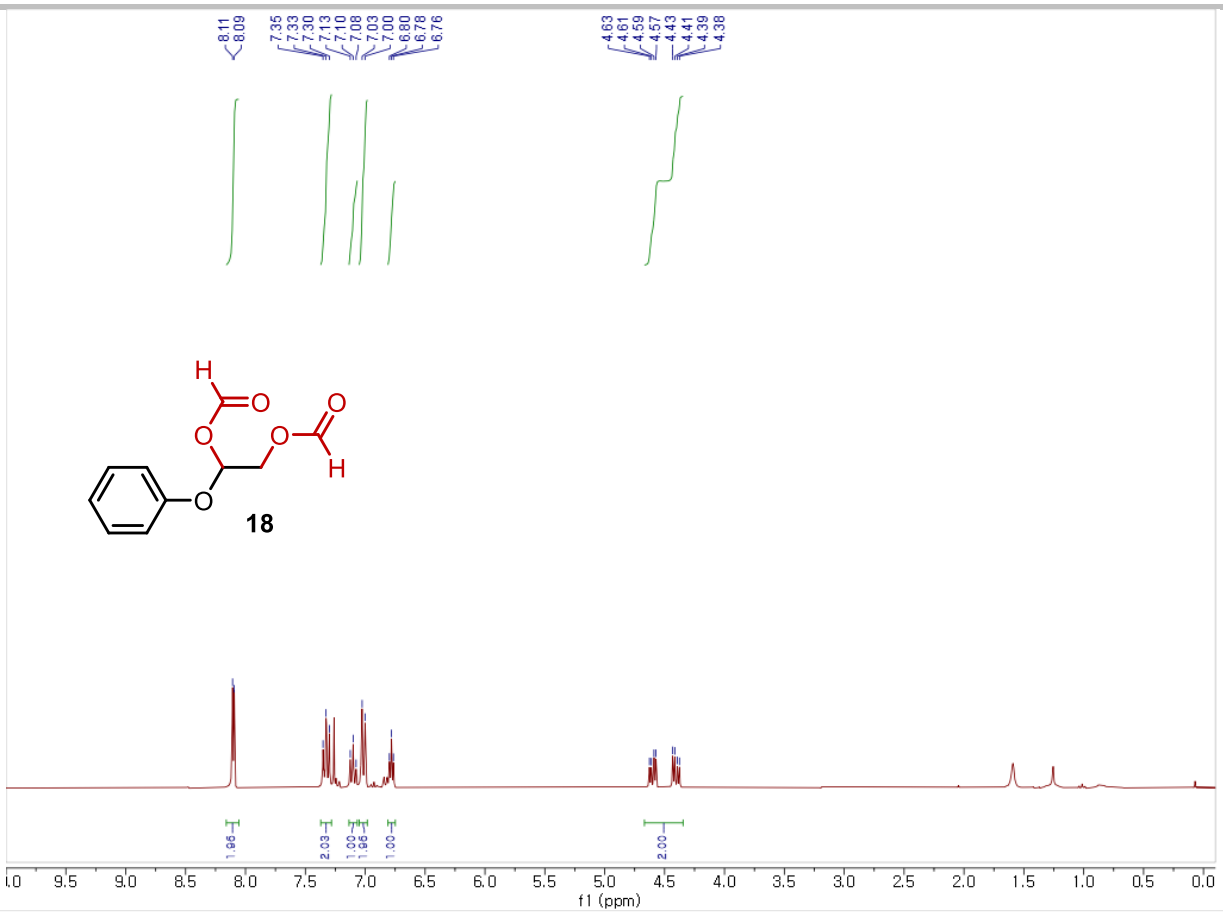
64.98
61.71
53.42
37.57
14.10
f1 (ppm)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7.08
7.03
7.00
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6.78
6.76
4.63
4.61
4.59
4.57
4.43
4.41
4.39
4.38
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H
O
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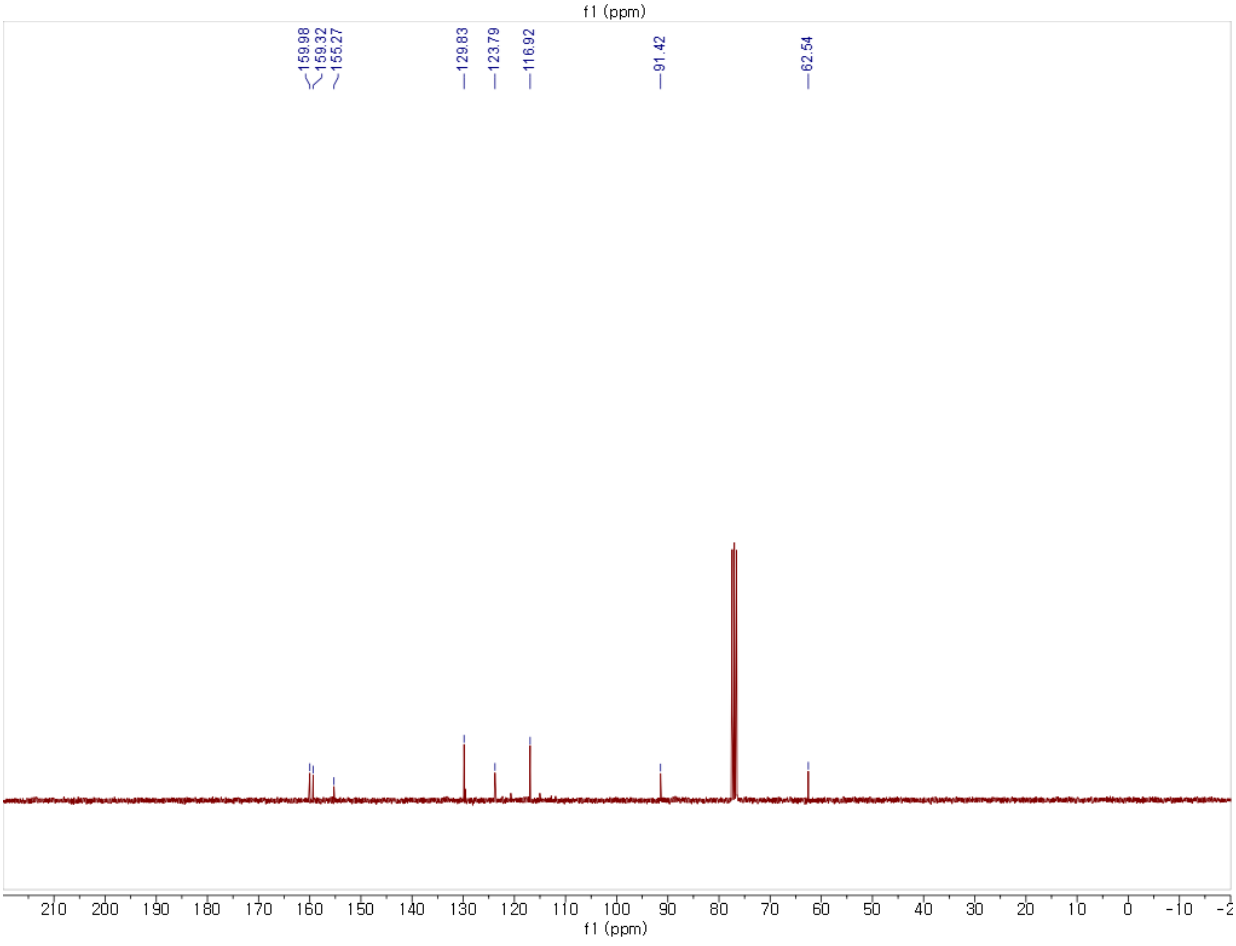
1.03
1.96
1.00
2.00
f1 (ppm)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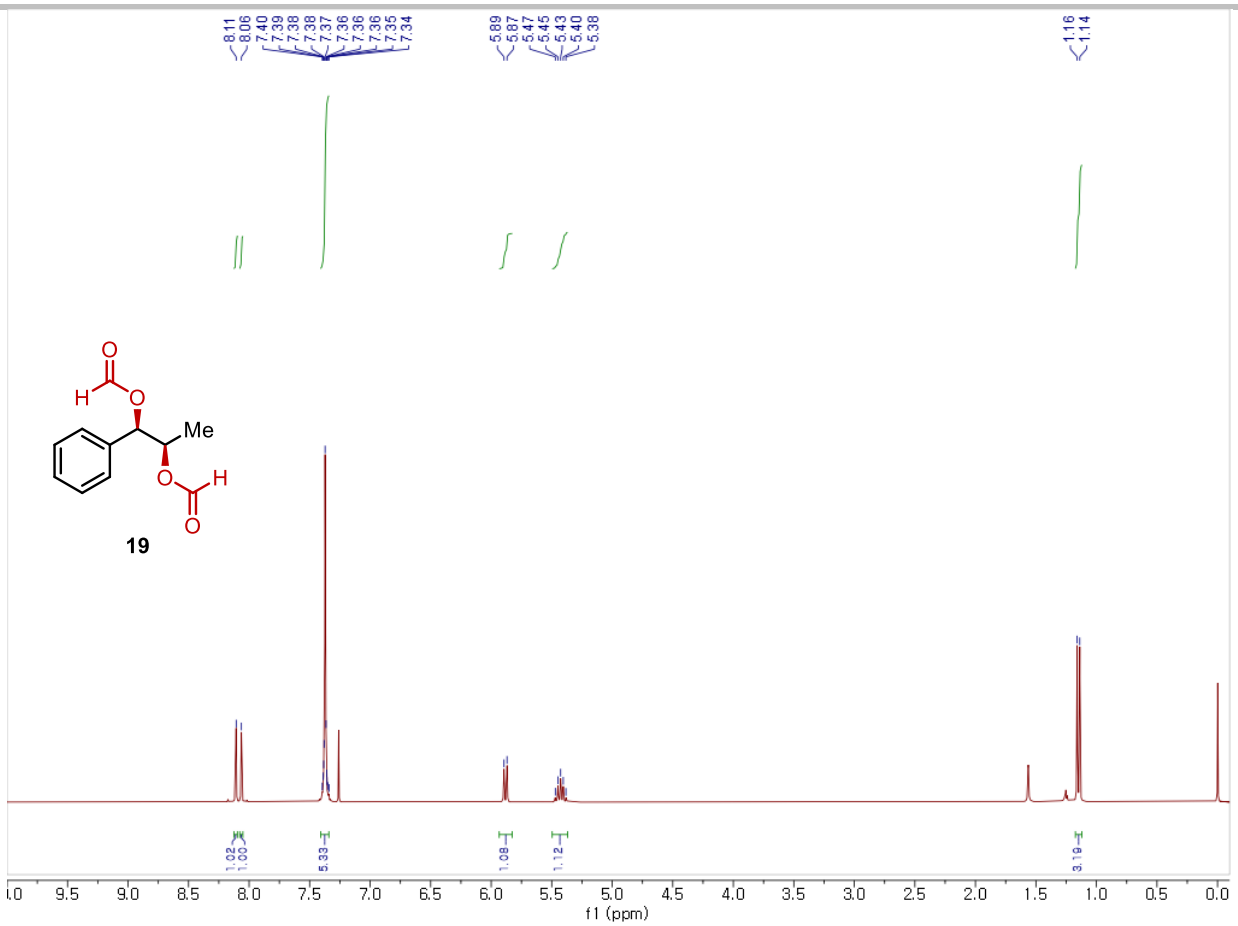
62.54
f1 (ppm)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7.38
7.37
7.36
7.36
7.36
7.35
7.34
5.89
5.87
5.47
5.45
5.43
5.40
5.38
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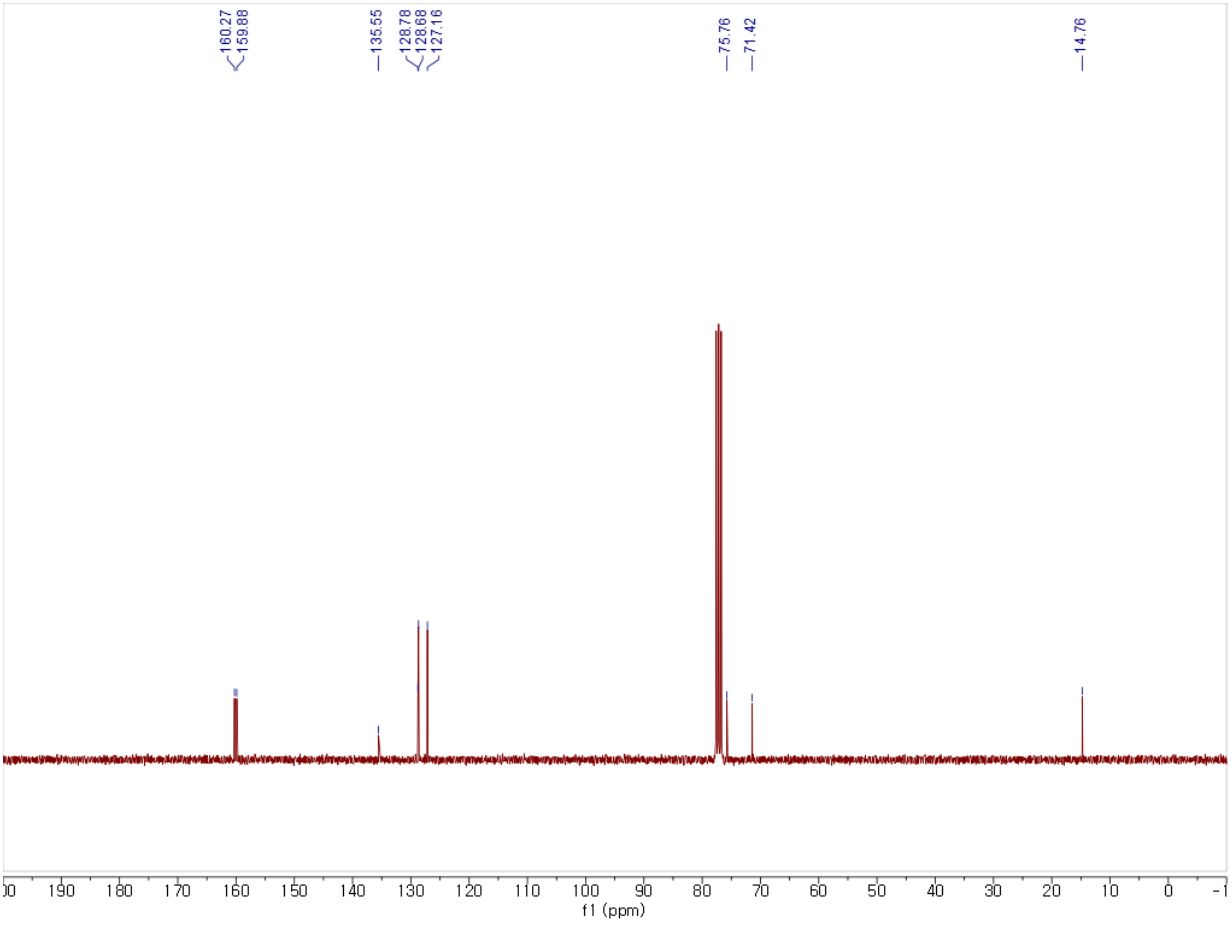
1.14
Me
19
1.02
1.00
5.33
1.08
1.12
3.19
f1 (ppm)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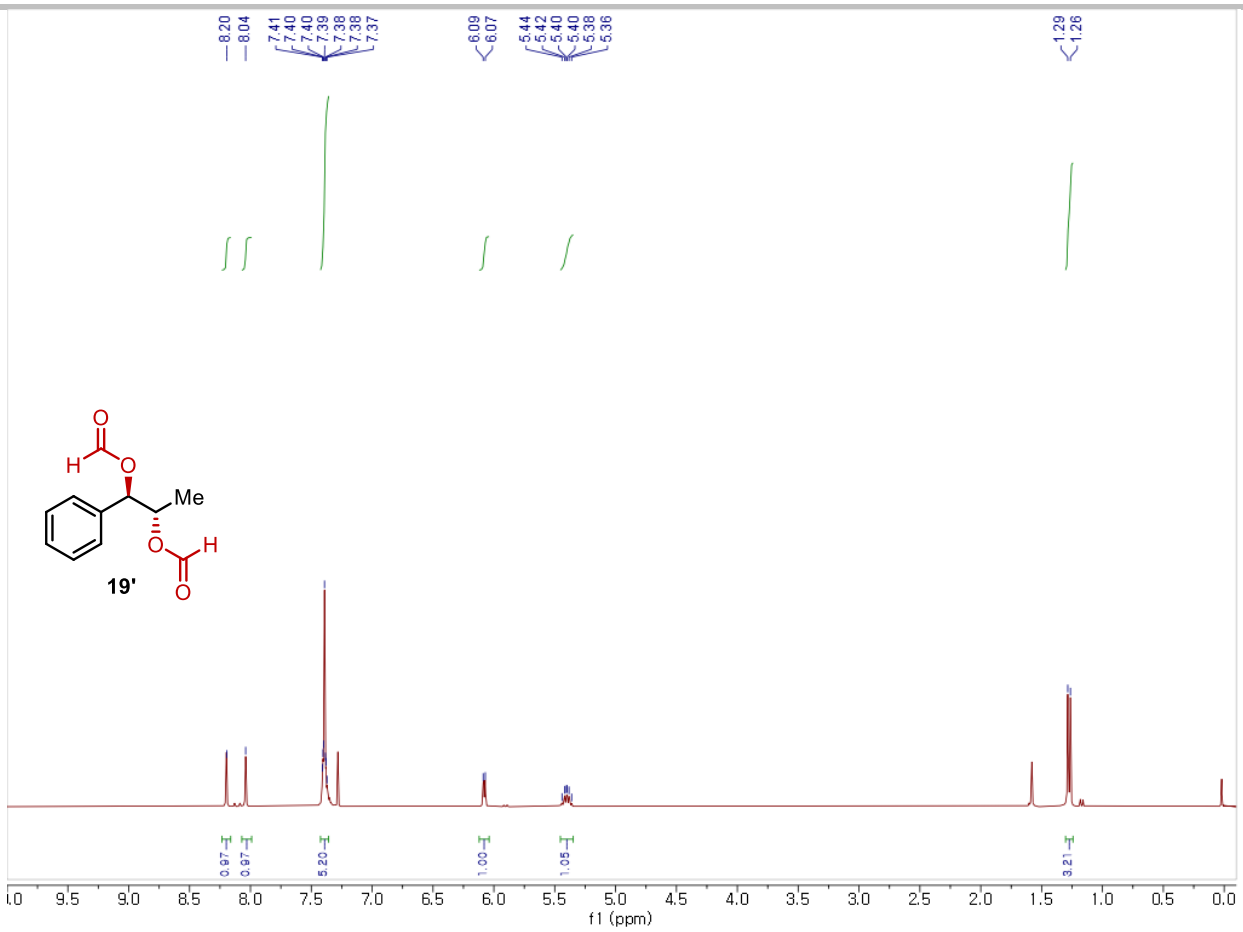
75.76
71.42
14.76
f1 (ppm)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7.40
7.39
7.38
7.38
7.37
6.09
6.07
5.44
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5.40
5.40
5.38
5.36
1.29
1.26
O
H
O
Me
O
H
O
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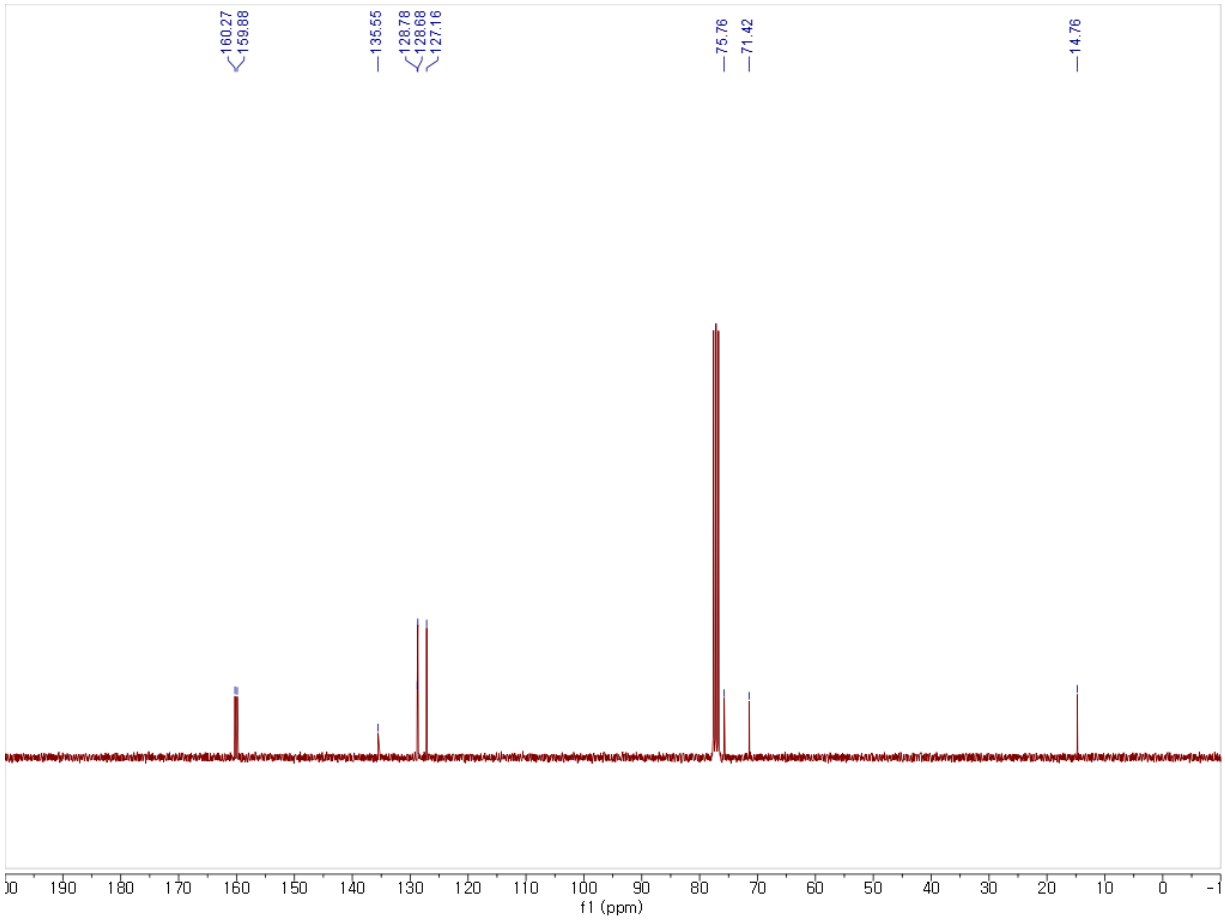
0.97
0.97
5.20
1.00
1.05
3.21
f1 (ppm)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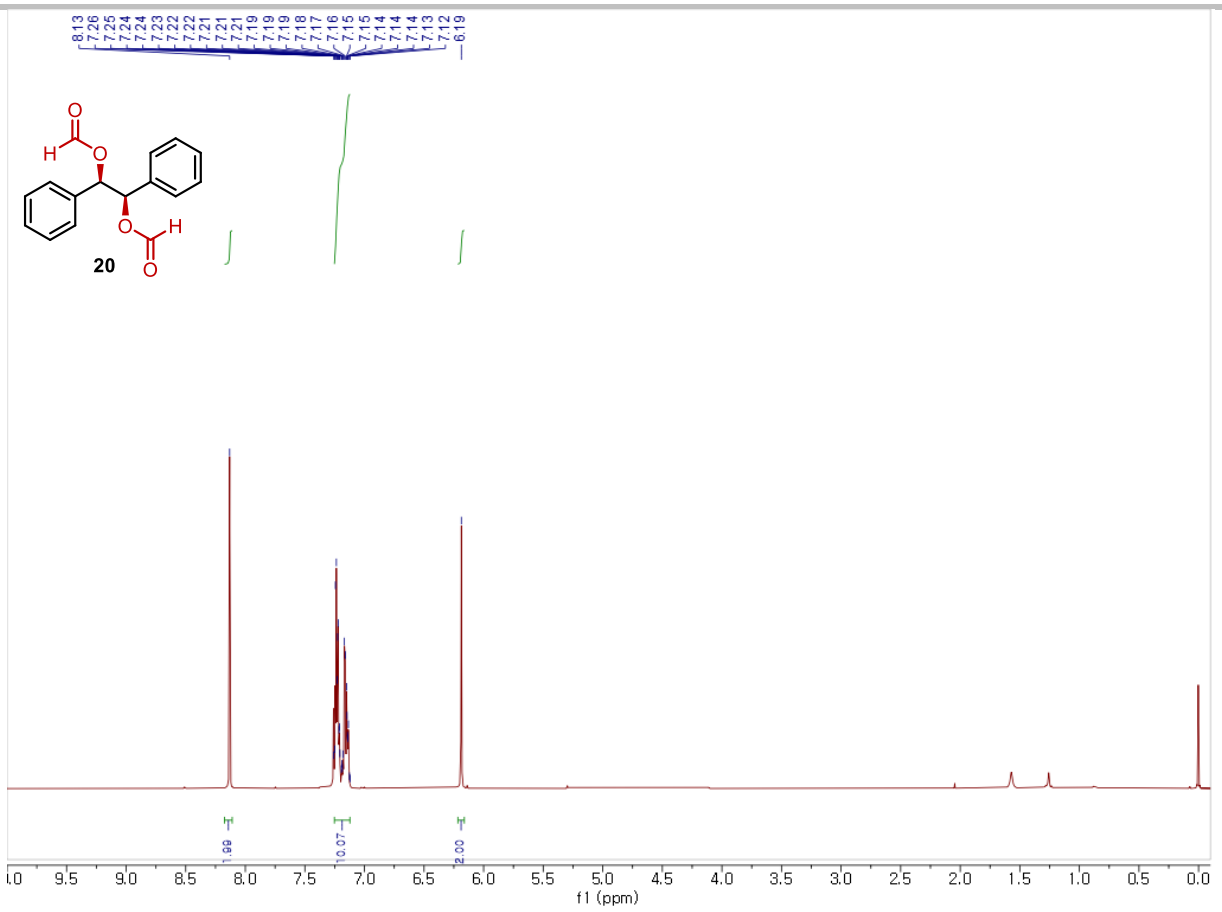
71.42
14.76
f1 (ppm)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7.19
7.19
7.19
7.18
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7.16
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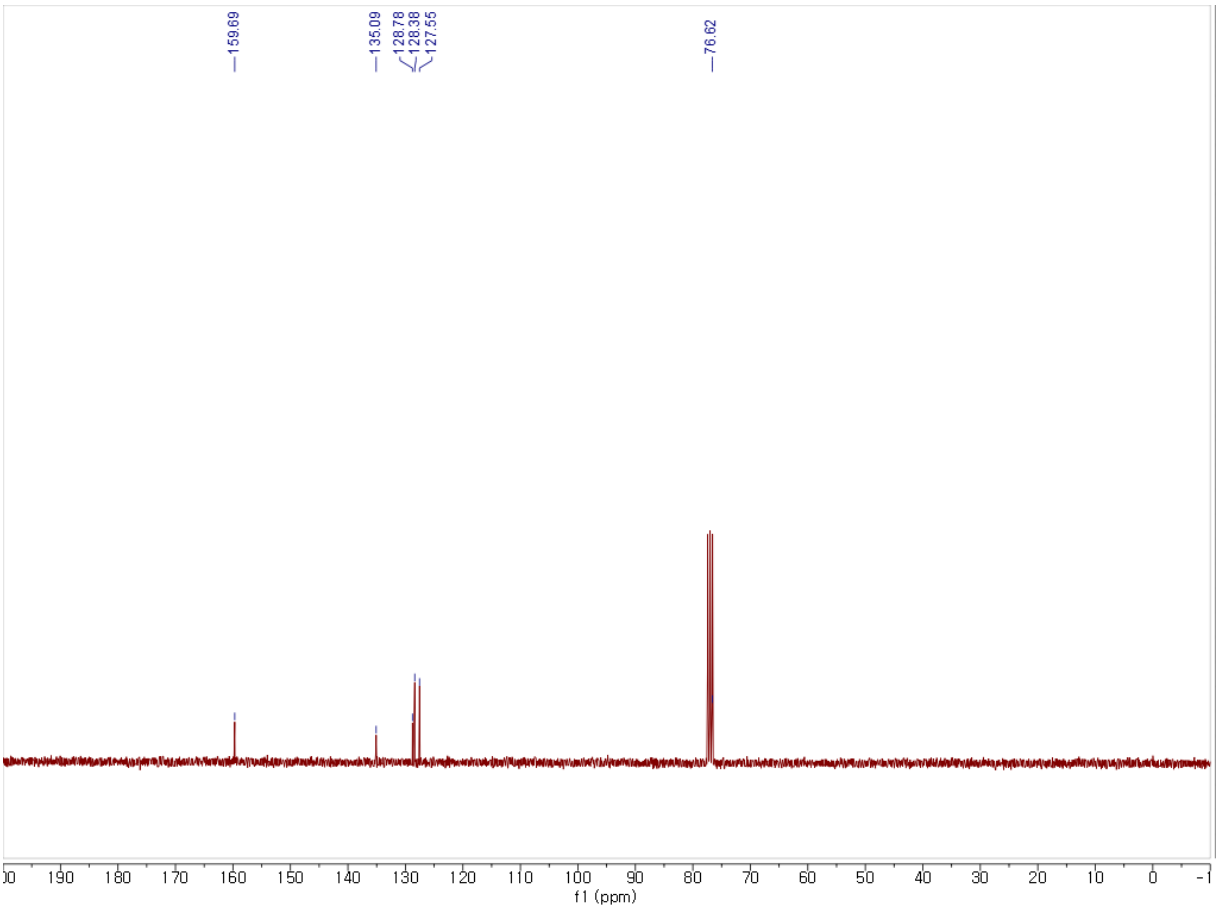
6.19
20
1.99
10.07
2.00
f1 (ppm)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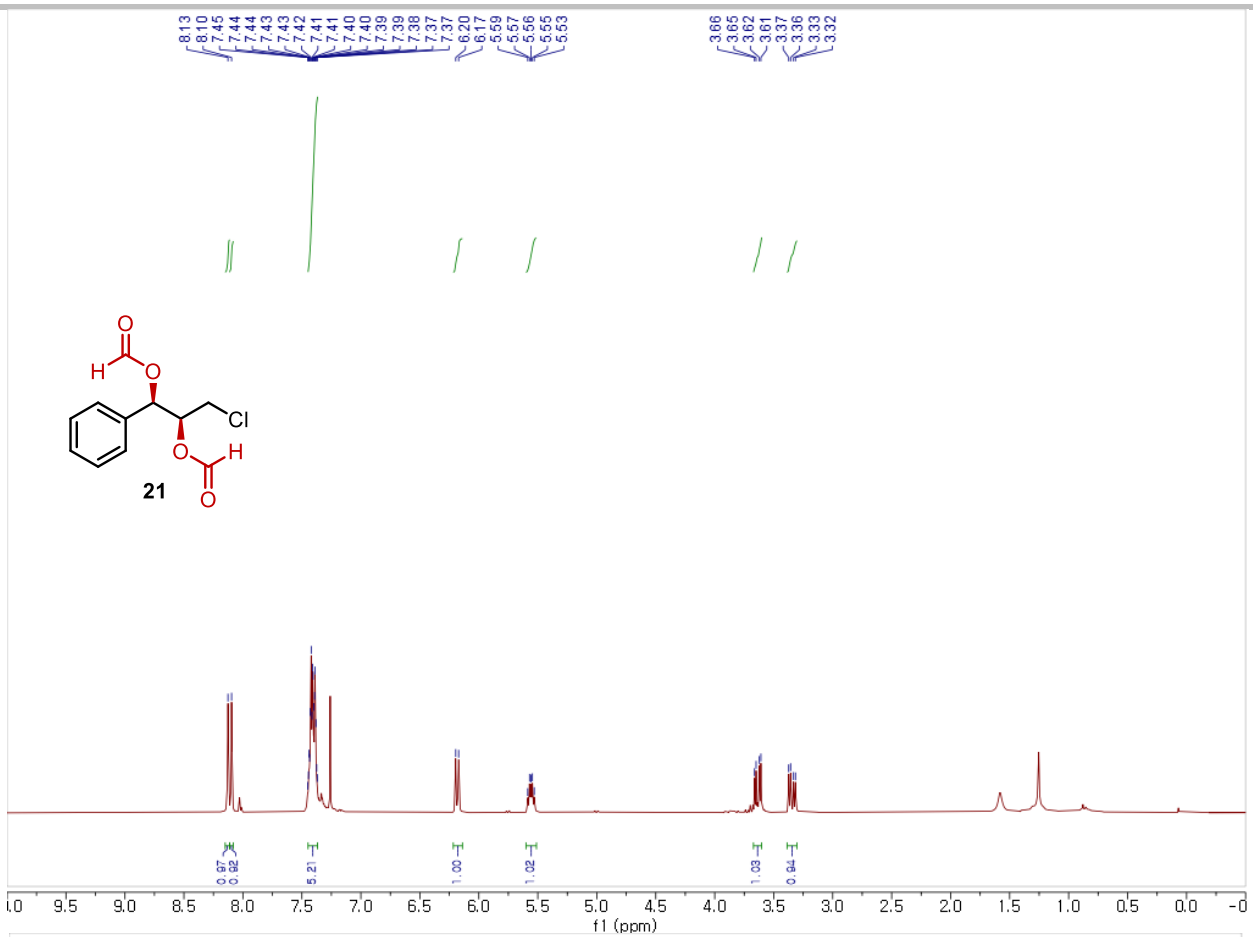
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7.41
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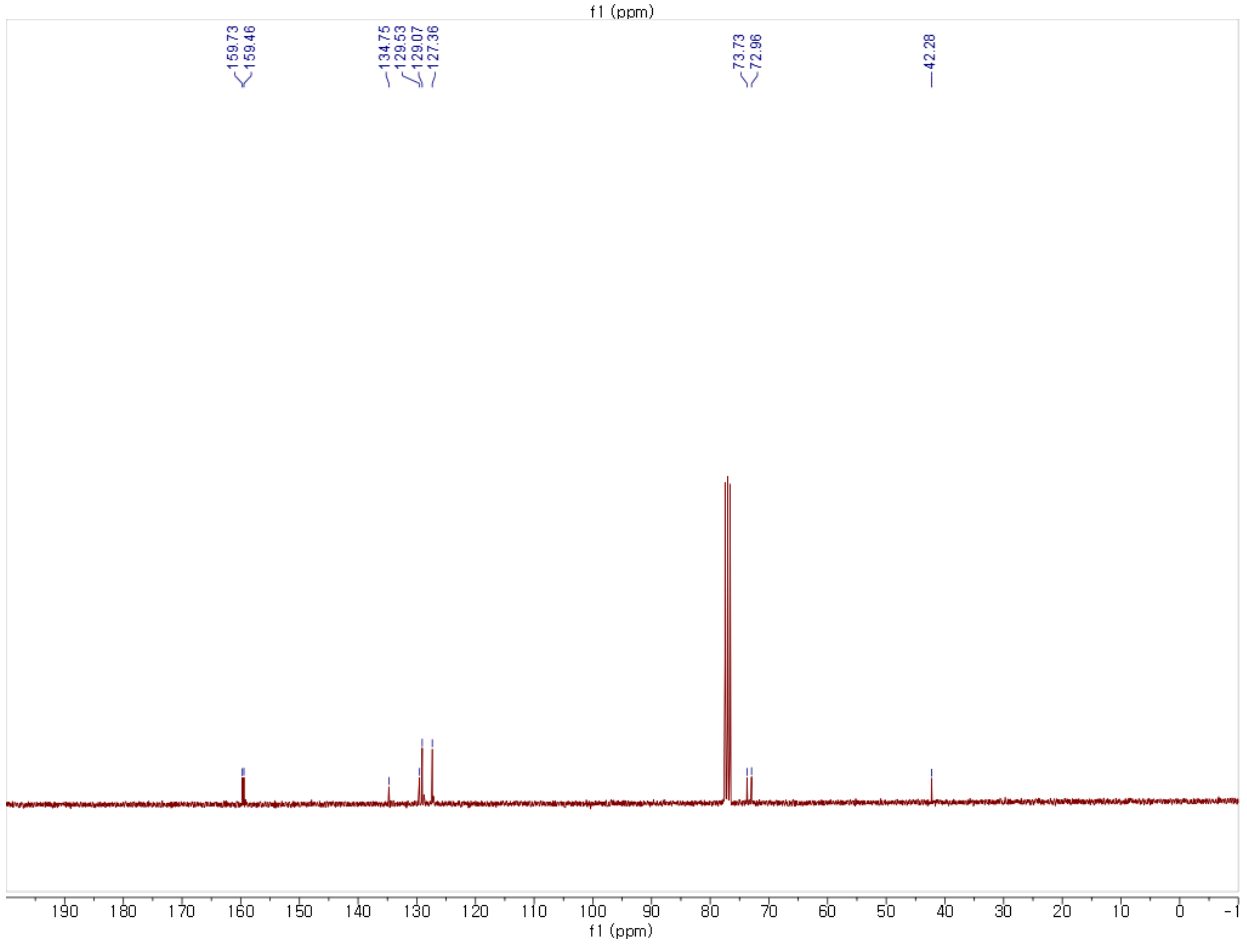
7.40
7.39
7.39
7.38
7.37
7.37
6.20
6.17
5.59
5.57
5.56
5.55
5.53
3.66
3.65
3.62
3.61
3.37
3.36
3.33
3.32
O
H
O
Cl
O
H
O
21
0.97
0.92
5.21
1.00
1.02
1.03
0.94
f1 (ppm)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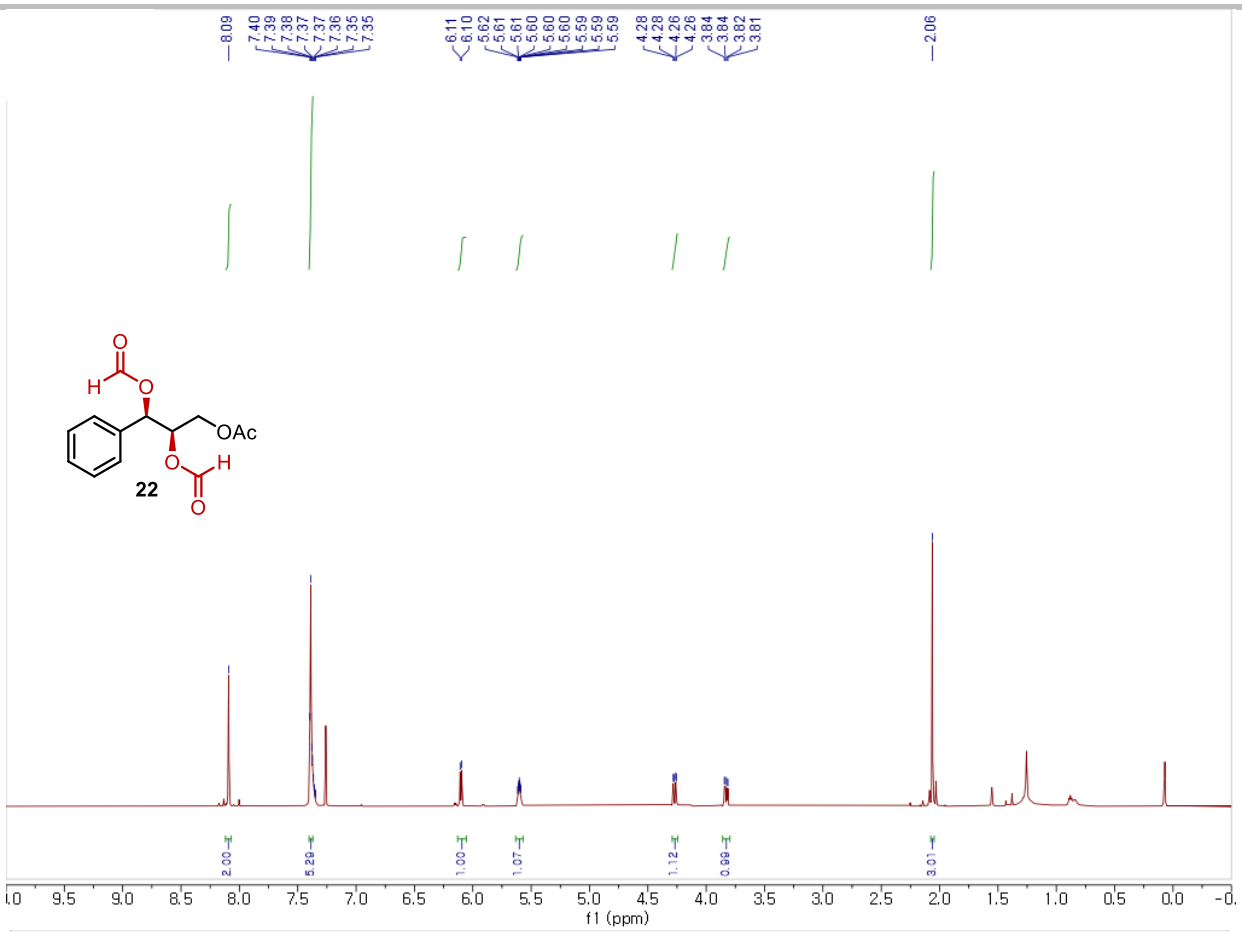
127.36
73.73
72.96
42.28
f1 (ppm)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7.38
7.37
7.37
7.36
7.35
7.35
6.11
6.10
5.62
5.61
5.61
5.60
5.60
5.60
5.59
5.59
5.59
4.28
4.28
4.26
4.26
3.84
3.84
3.82
3.81
2.06
O
H
O
OAc
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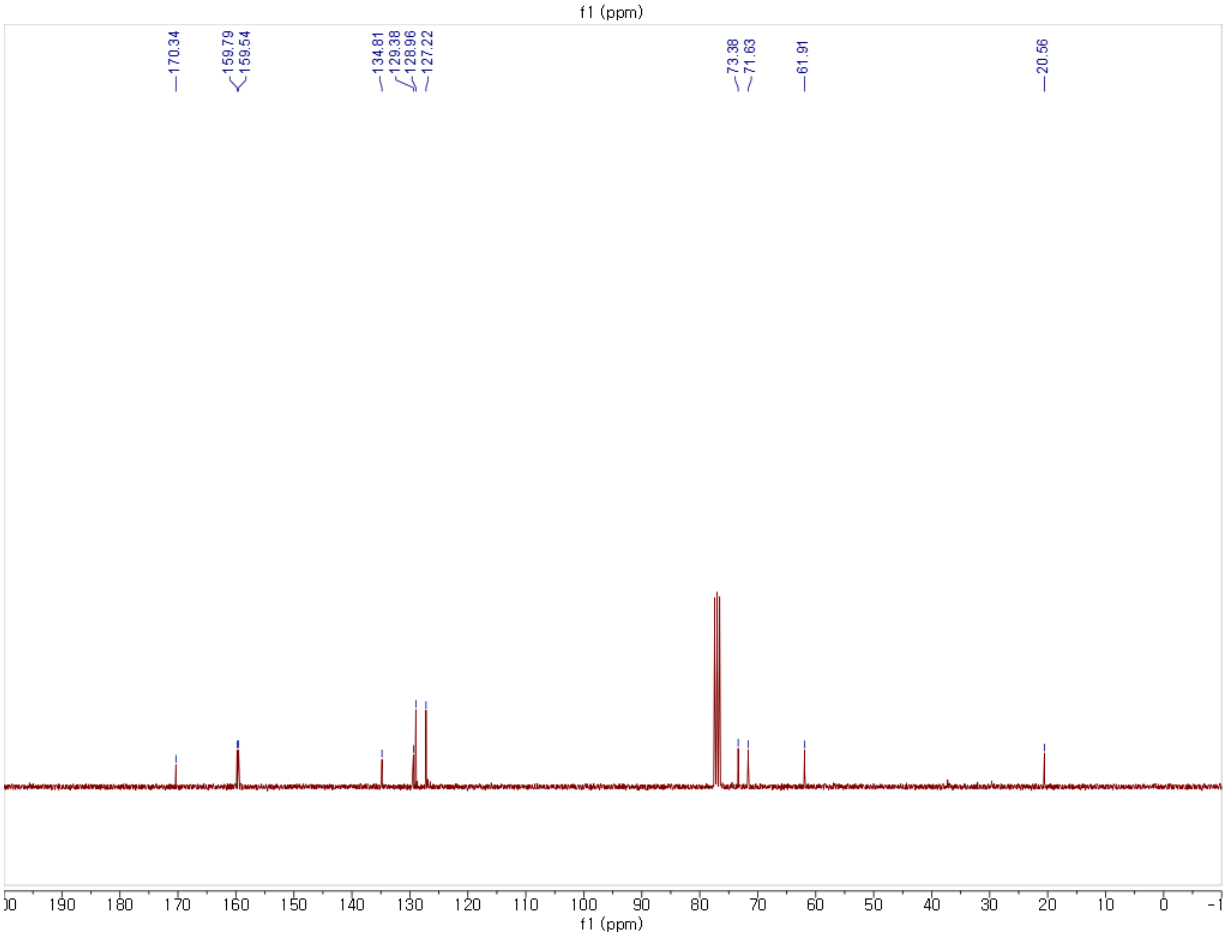
H
O
22
2.00
5.29
1.00
1.07
1.12
0.99
3.01
f1 (ppm)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73.38
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20.56
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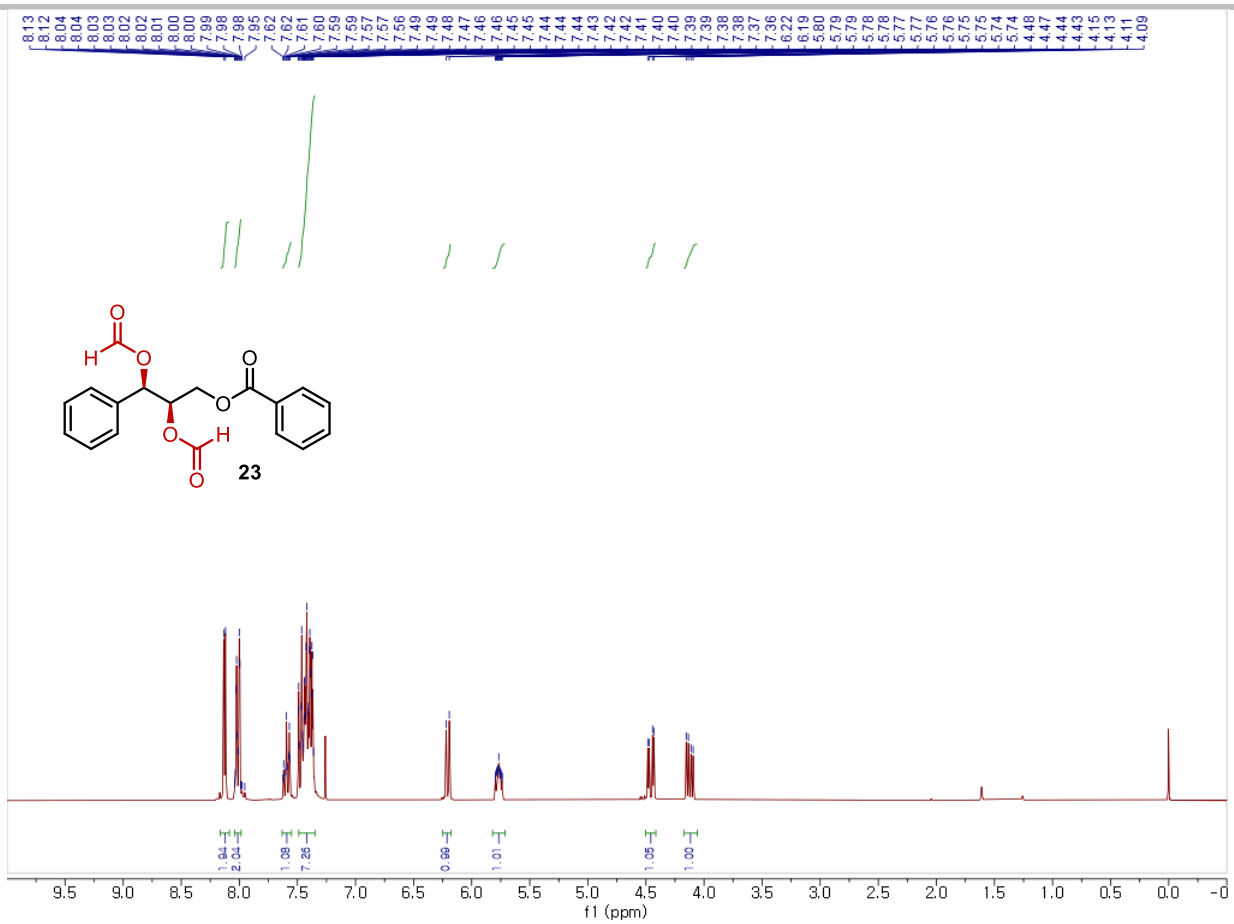
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8.04
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8.03
8.02
8.02
8.01
8.00
8.00
7.99
7.98
7.98
7.95
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7.62
7.61
7.61
7.60
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7.57
7.57
7.56
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7.49
7.48
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7.44
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7.43
7.42
7.42
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7.40
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7.39
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7.37
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5.79
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5.78
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5.77
5.76
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4.44
4.43
4.15
4.13
4.11
4.09
O
H
O
O
O
O
H
O
23
1.94
2.04
1.08
7.26
0.99
1.01
1.05
1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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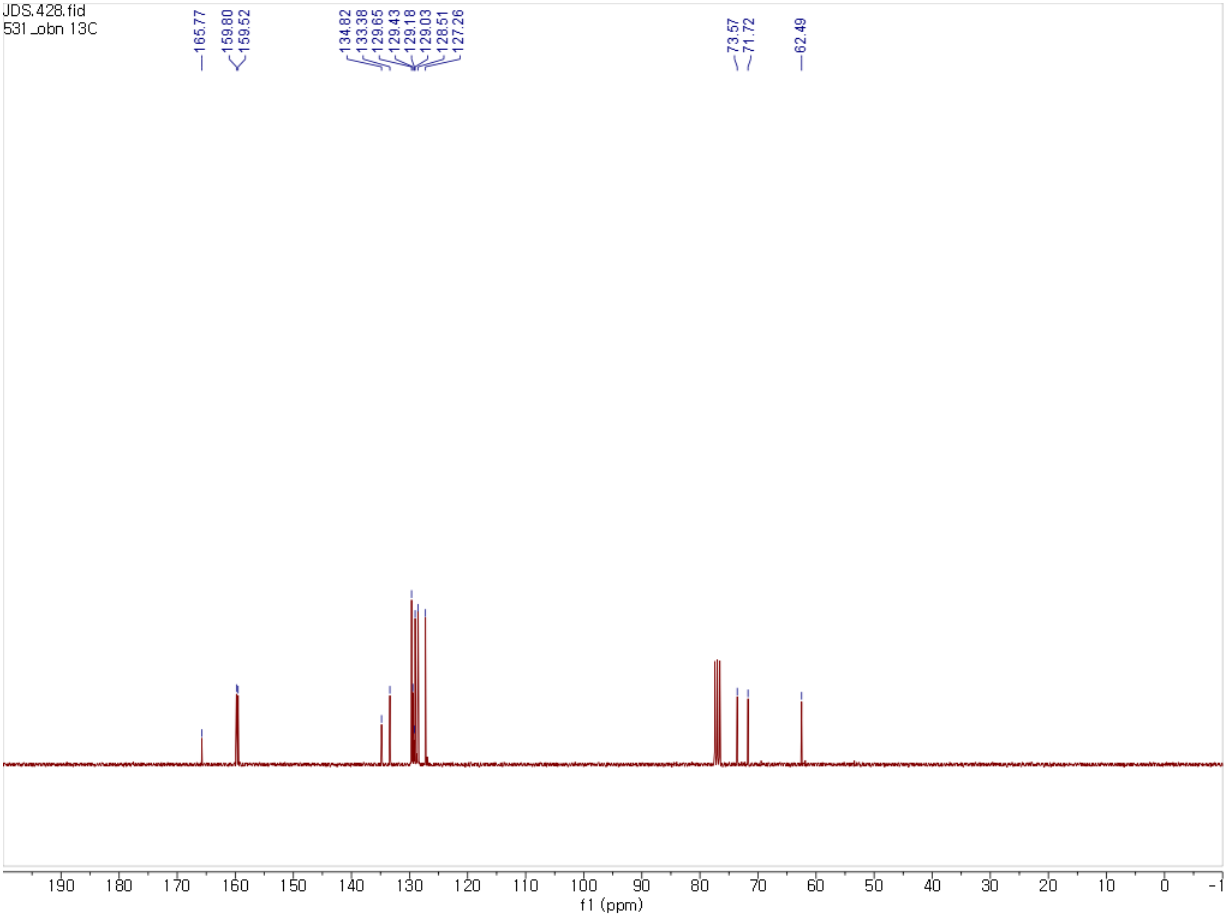
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)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531_obn 13C
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133.38
129.65
129.43
129.18
129.03
128.51
127.26
73.57
71.72
62.49
190
180
170
160
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140
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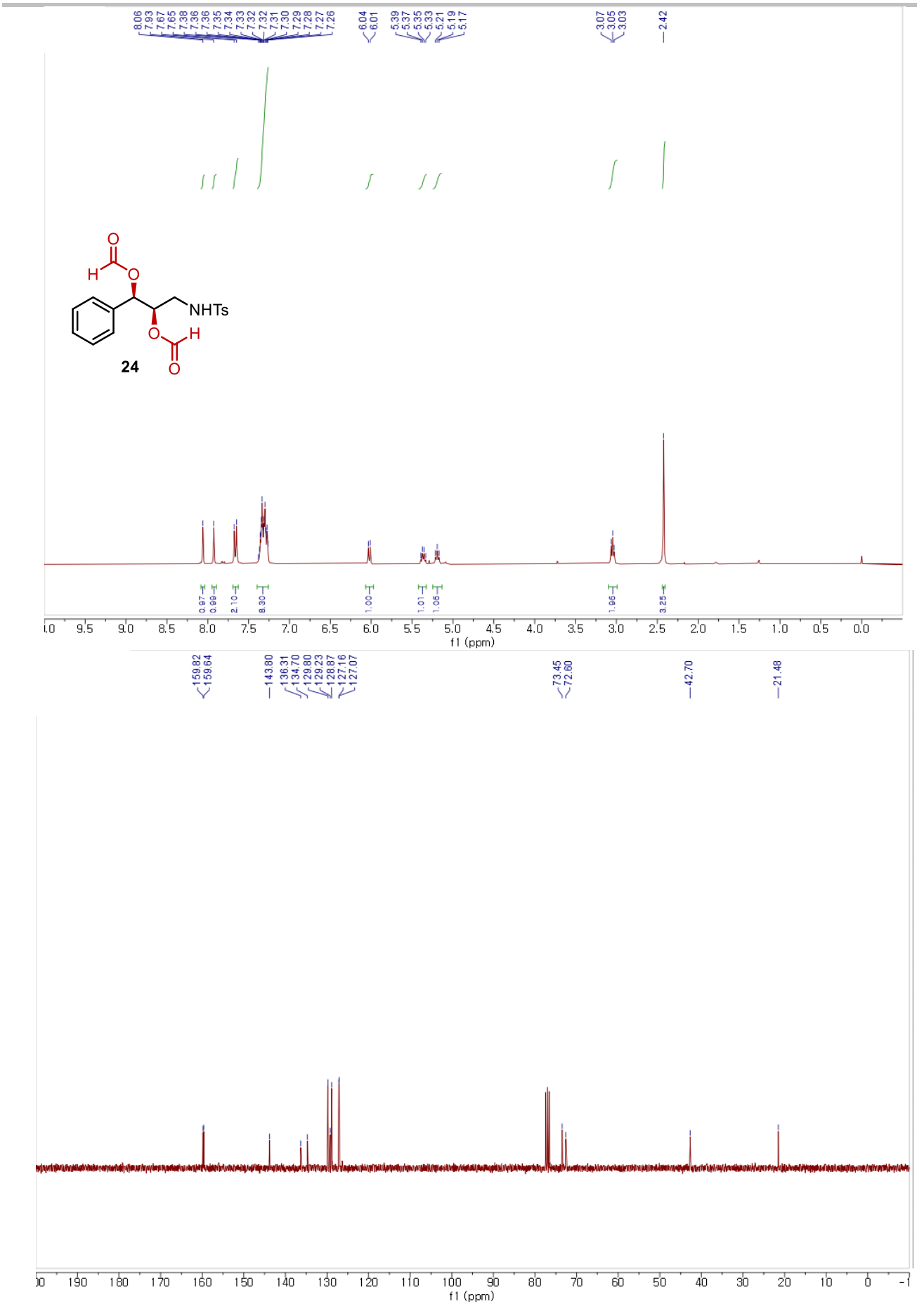
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)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7.33
7.32
7.32
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7.28
7.27
7.26
6.04
6.01
5.39
5.37
5.35
5.33
5.21
5.19
5.17
3.07
3.05
3.03
2.42
NHTs
24
0.97
0.99
2.10
8.30
1.00
1.01
1.06
1.96
3.25
f1 (ppm)
159.82
159.64
143.80
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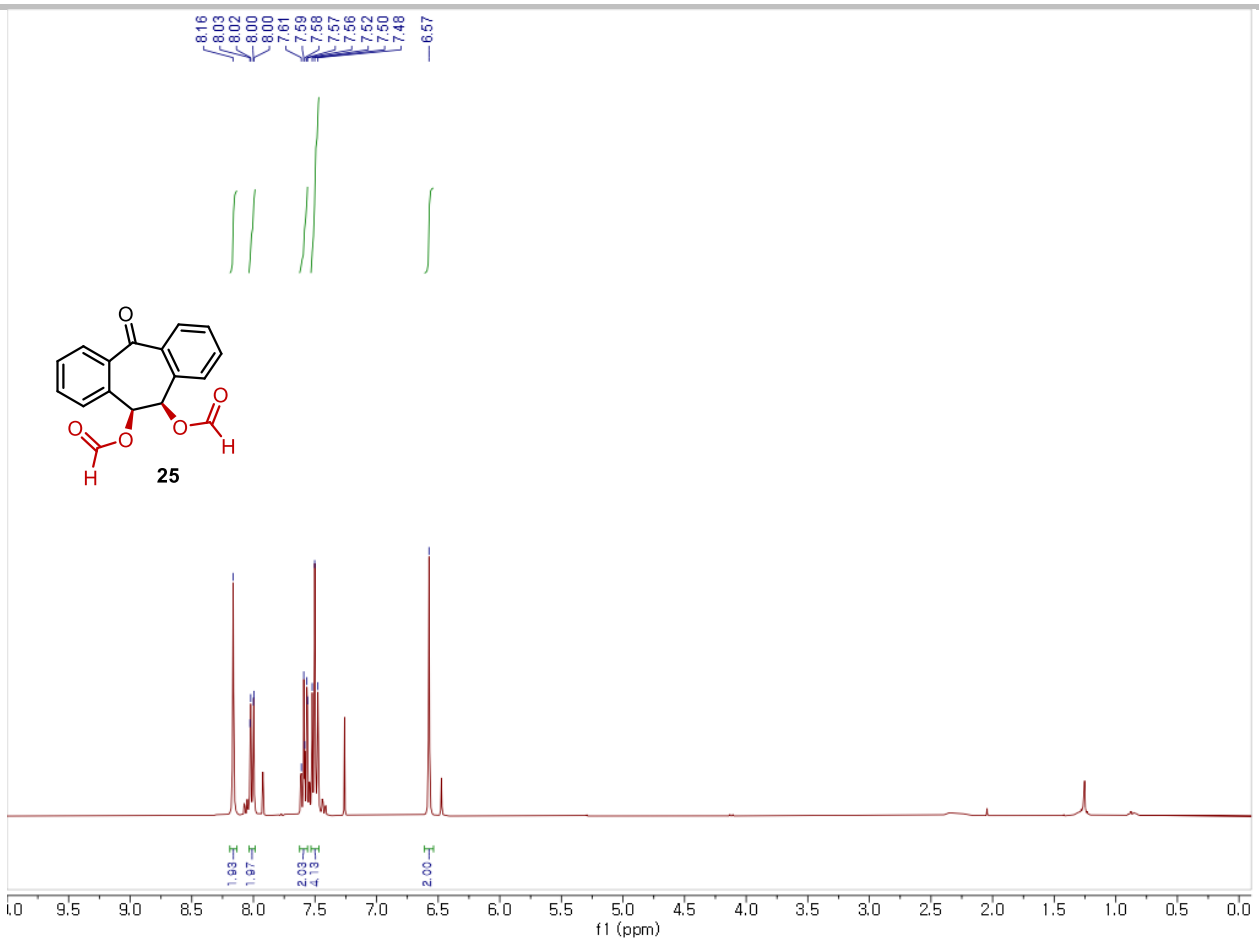
129.23
128.87
127.16
127.07
73.45
72.60
42.70
21.48
f1 (ppm)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8.00
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7.58
7.57
7.56
7.52
7.50
7.48
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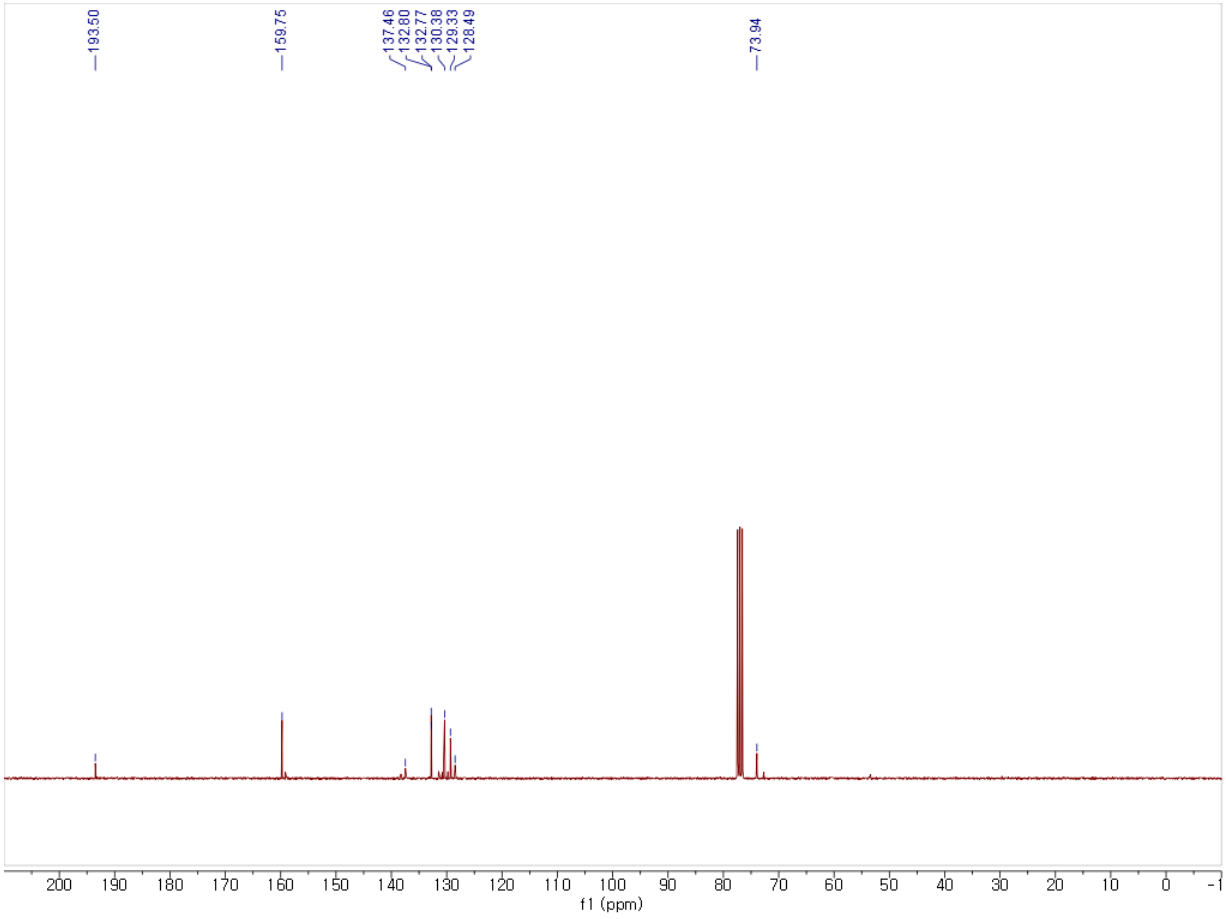
1.97
2.03
4.13
2.00
f1 (ppm)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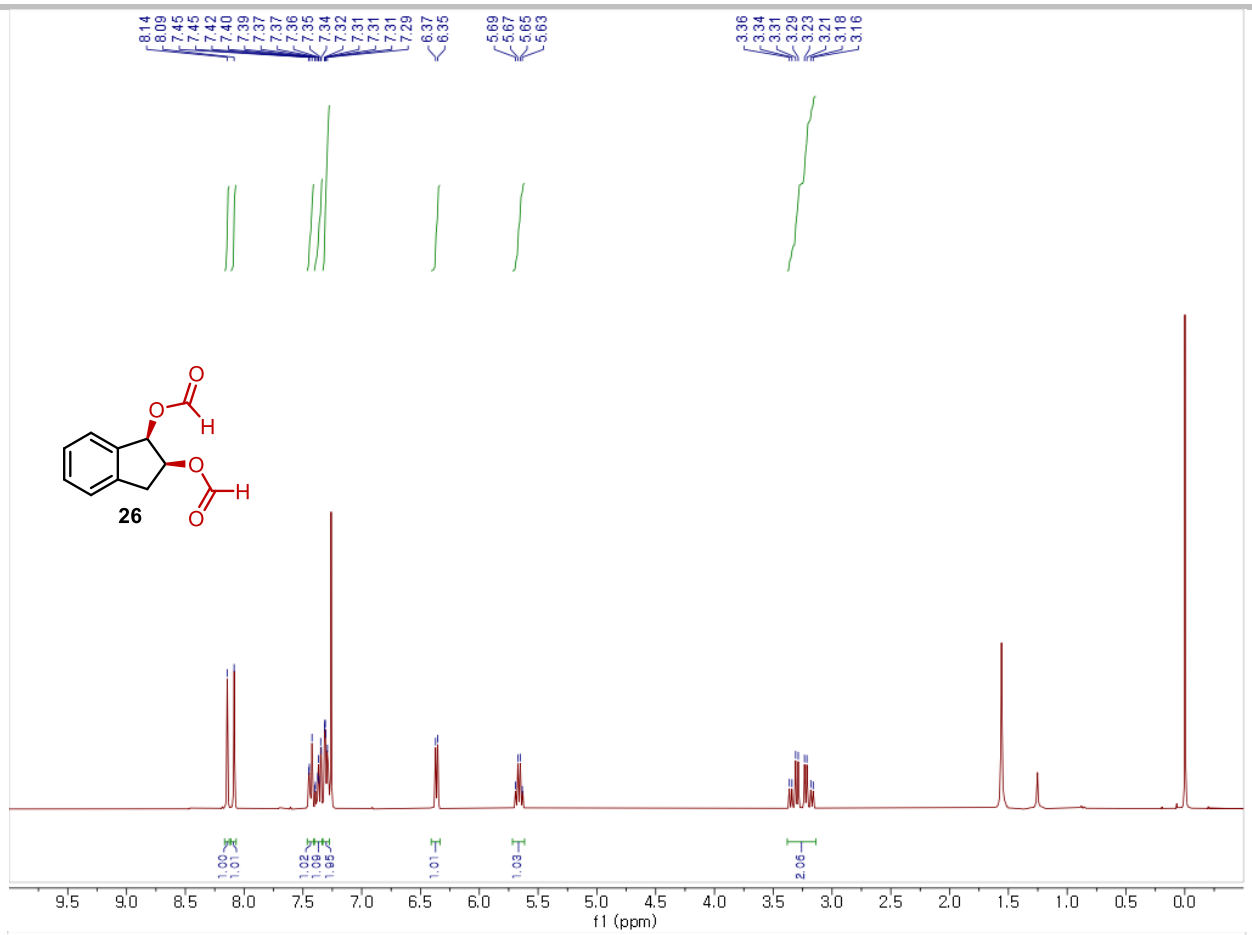
73.94
f1 (ppm)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7.36
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7.32
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7.31
7.31
7.29
6.37
6.35
5.69
5.67
5.65
5.63
3.36
3.34
3.31
3.29
3.23
3.21
3.18
3.16
O
O
H
O
O
H
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1.01
1.02
1.09
1.85
1.01
1.03
2.06
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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2.0
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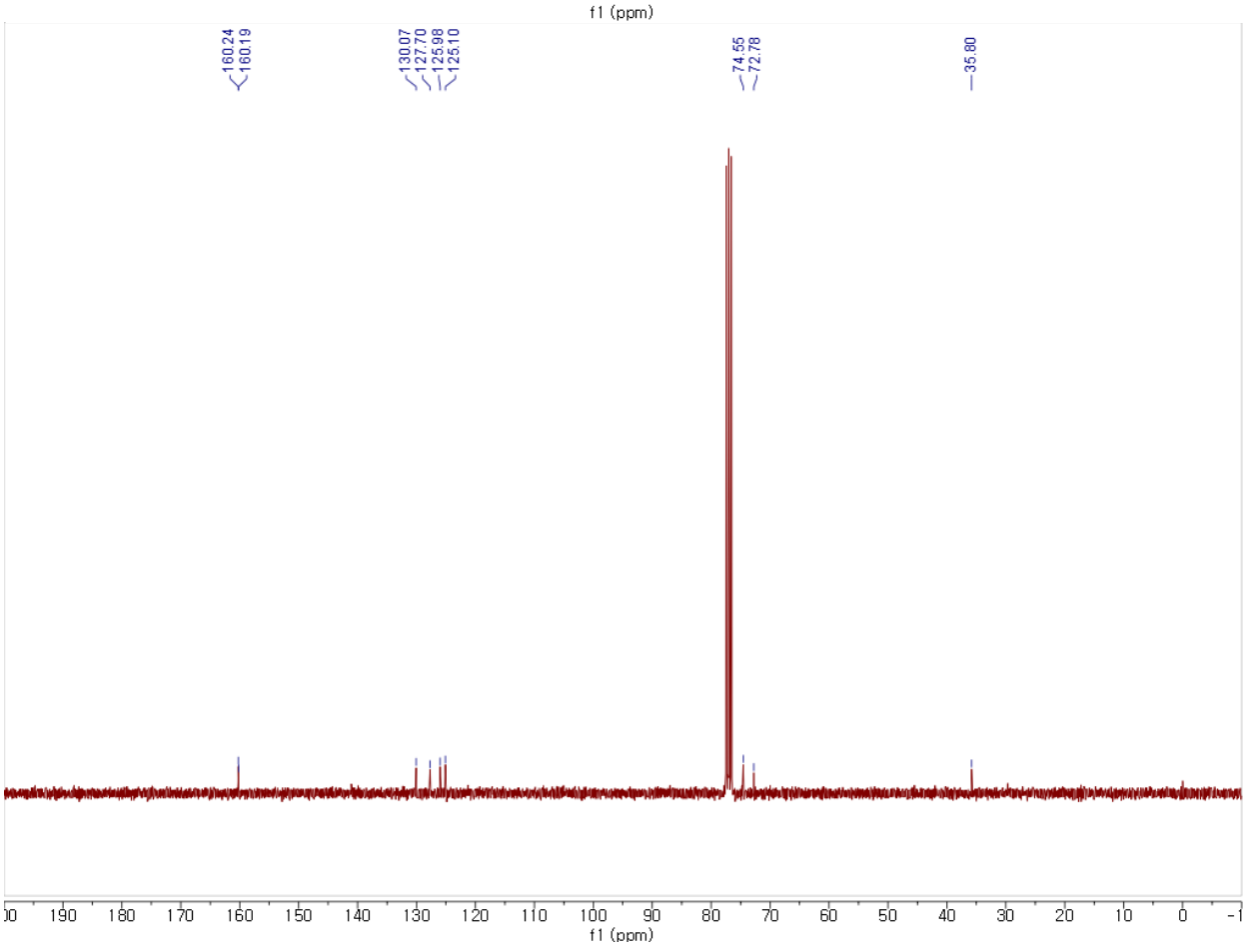
0.0
f1 (ppm)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190
180
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130
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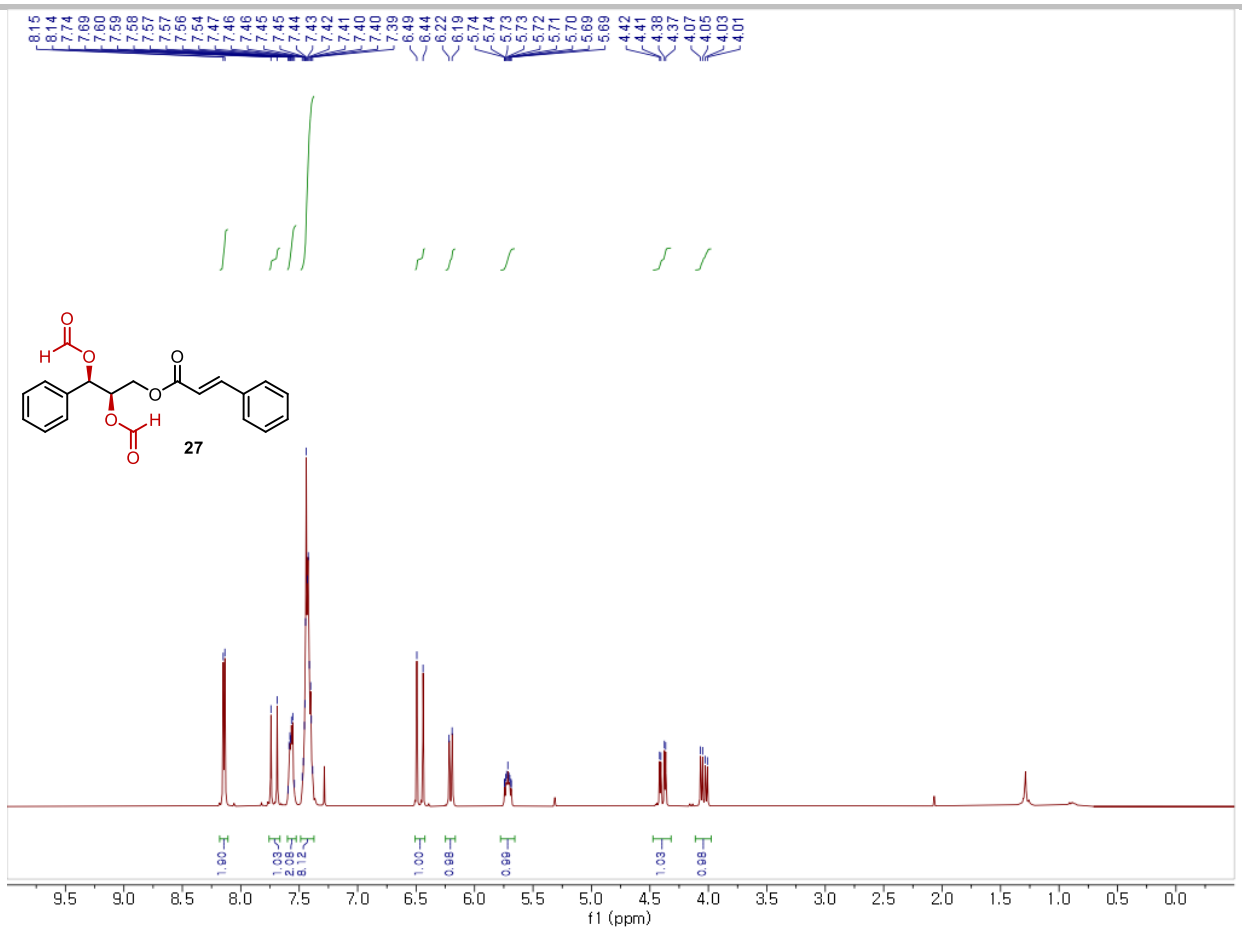
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.46
7.45
7.45
7.44
7.43
7.42
7.41
7.40
7.40
7.39
6.49
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6.22
6.19
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5.74
5.73
5.73
5.72
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5.70
5.69
5.69
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4.41
4.38
4.37
4.07
4.05
4.03
4.01
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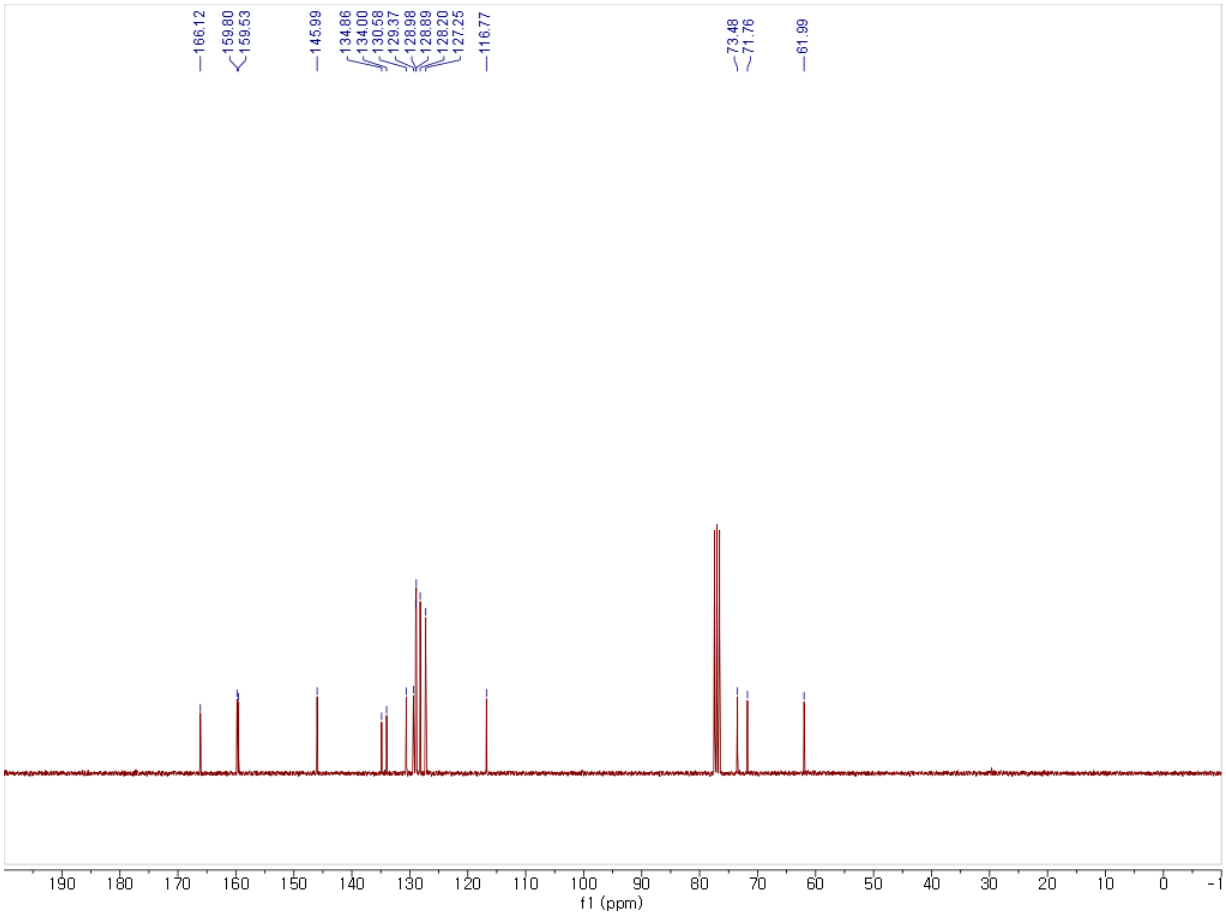
1.03
2.08
8.12
1.00
0.98
0.99
1.03
0.98
f1 (ppm)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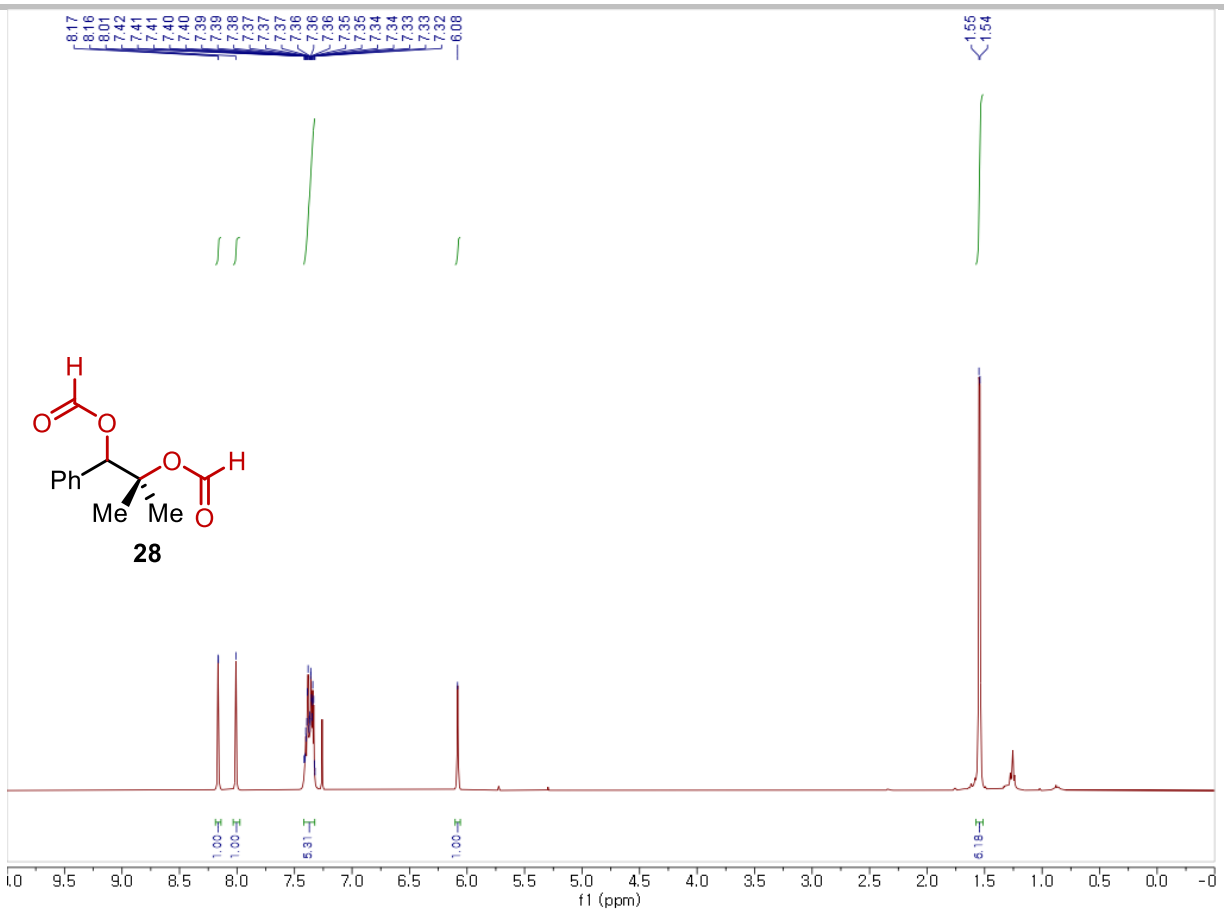
128.89
128.20
127.25
116.77
73.48
71.76
61.99
f1 (ppm)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7.41
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7.37
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7.36
7.35
7.35
7.34
7.34
7.33
7.33
7.32
6.08
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1.54
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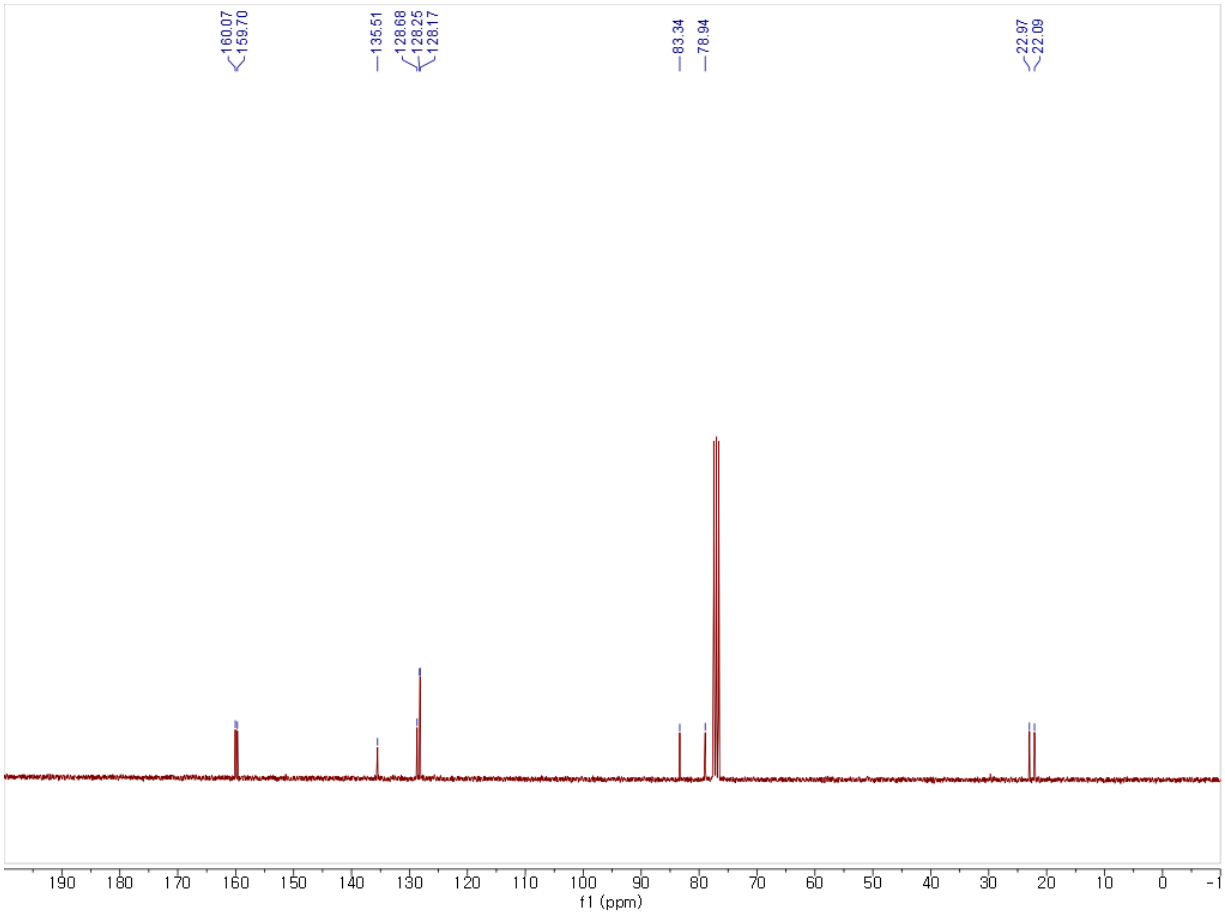
H
Me
Me
O
28
1.00
1.00
5.31
1.00
6.18
f1 (ppm)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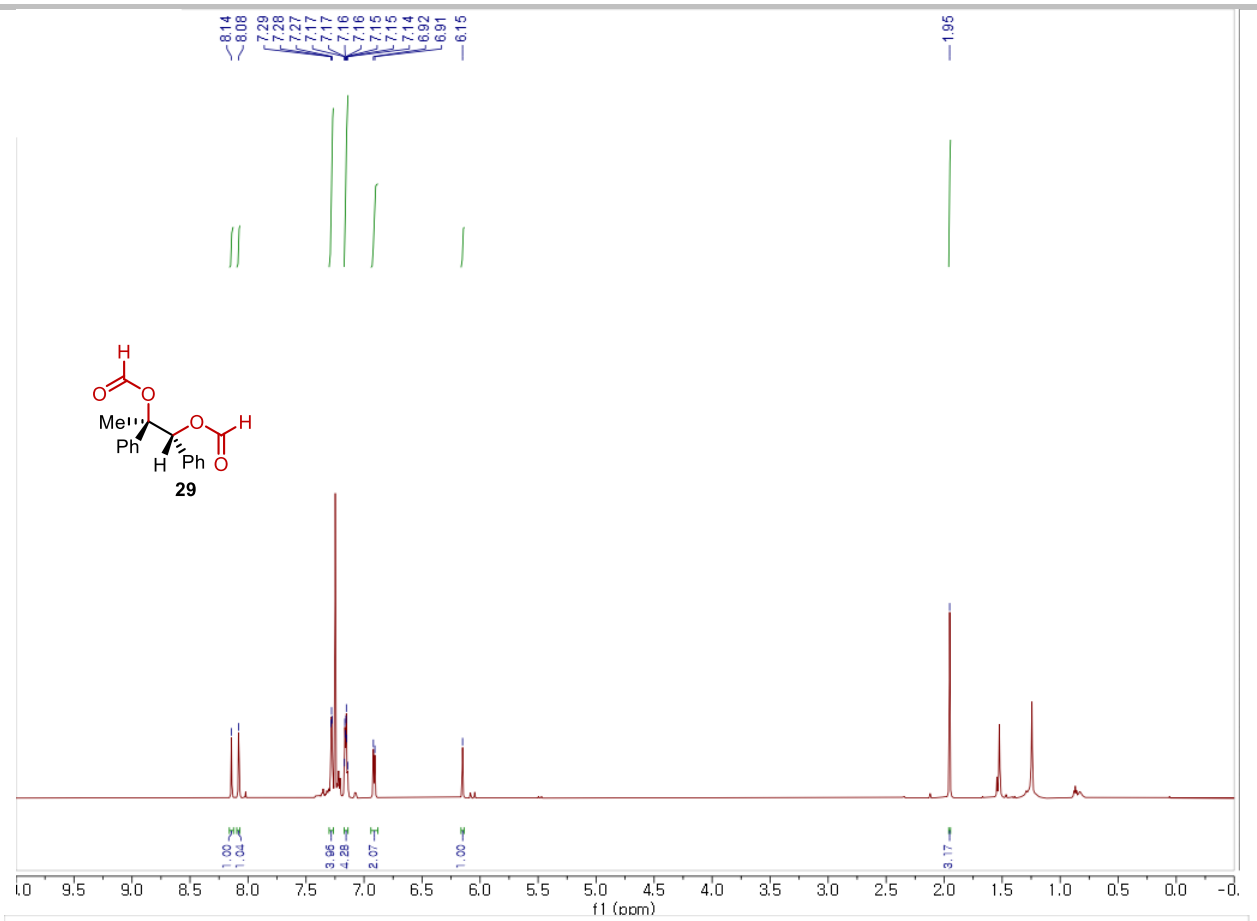
128.25
128.17
83.34
78.94
22.97
22.09
f1 (ppm)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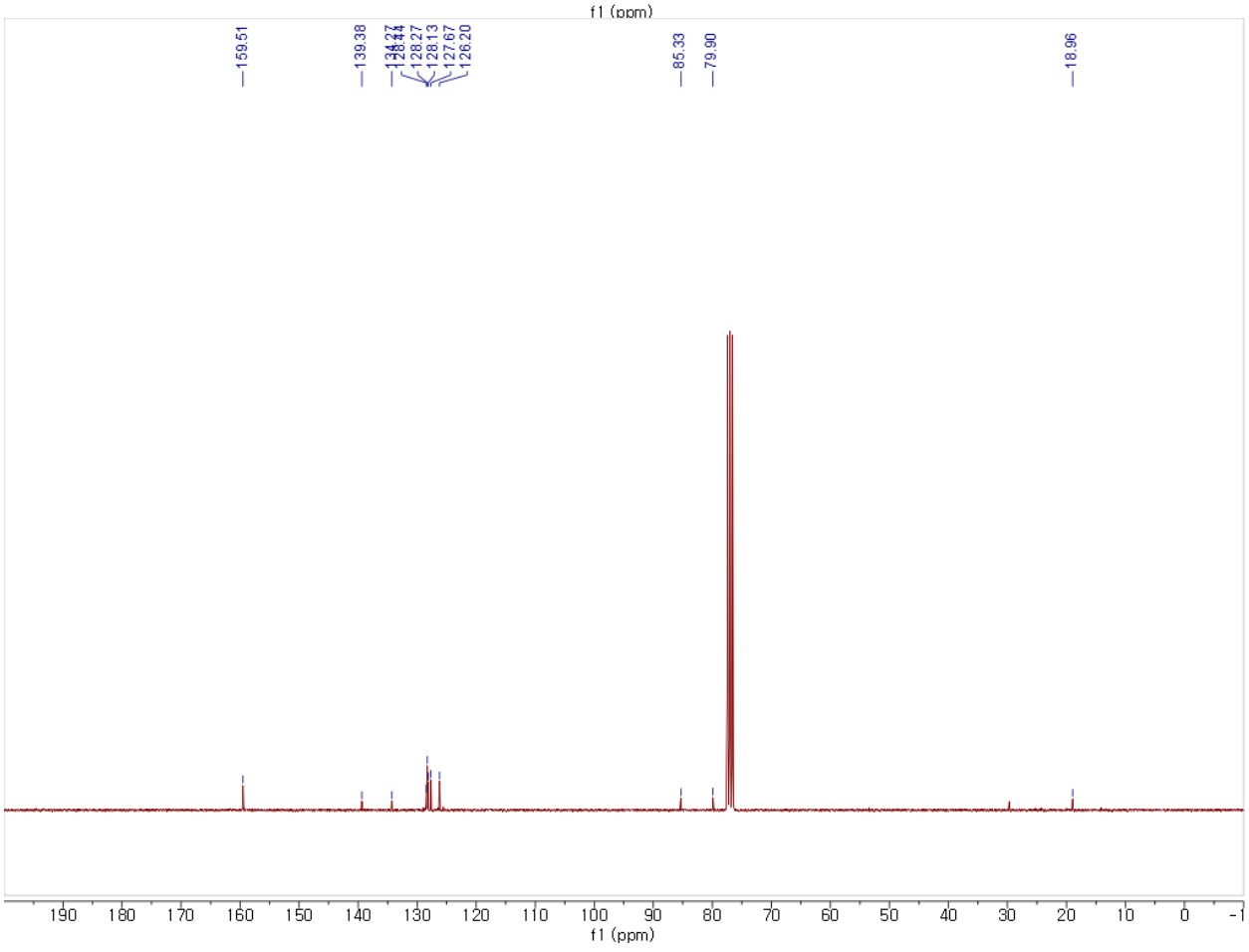
7.28
7.27
7.17
7.17
7.16
7.16
7.15
7.15
7.14
6.92
6.91
6.15
1.95
H
O
O
Me
Ph
O
H
H
Ph
O
29
1.00
1.04
3.96
4.28
2.07
1.00
3.17
f1 (ppm)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85.33
79.90
18.96
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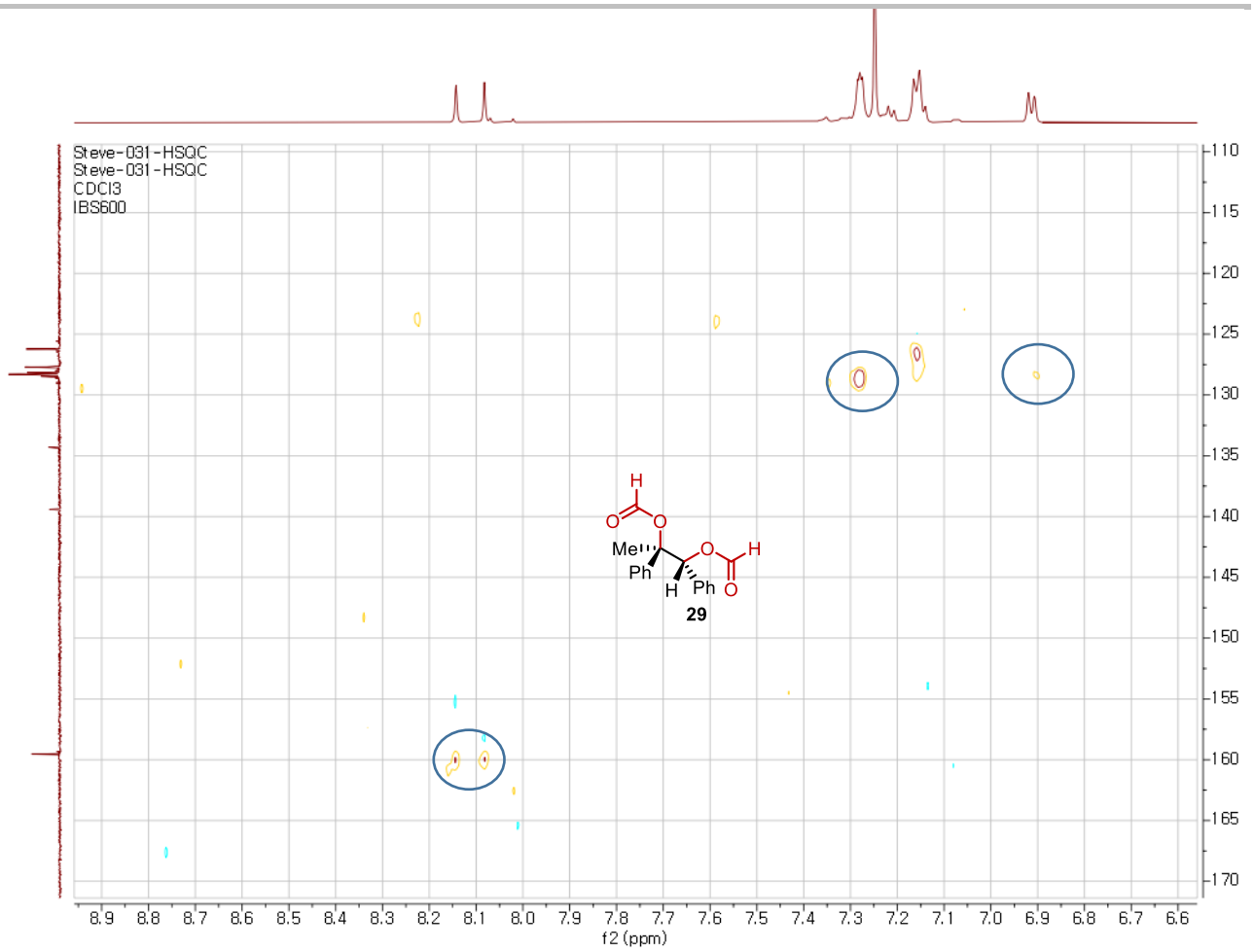
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Steve-031-HSQC
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IBS600
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O
Me
Ph
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H
H
Ph
O
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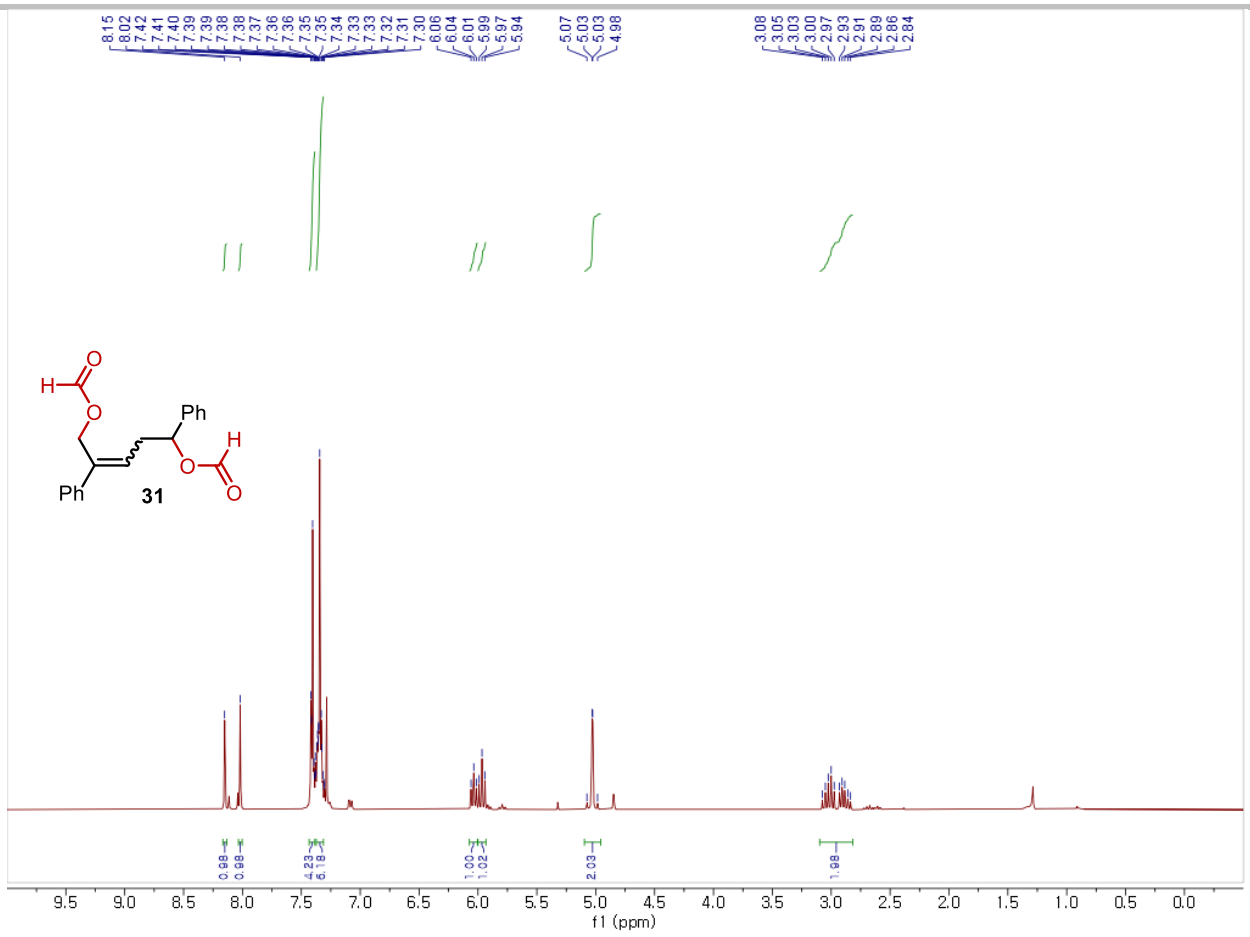
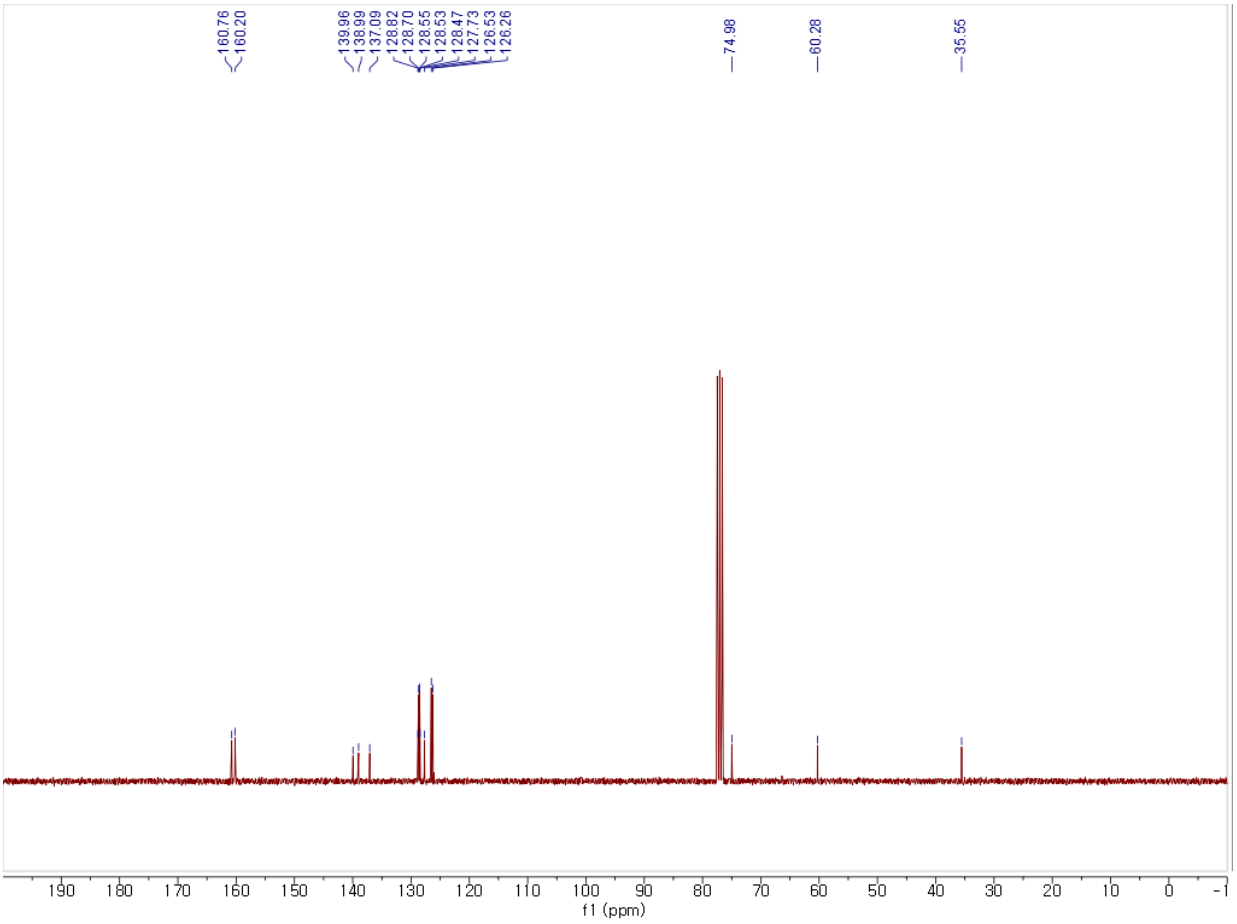
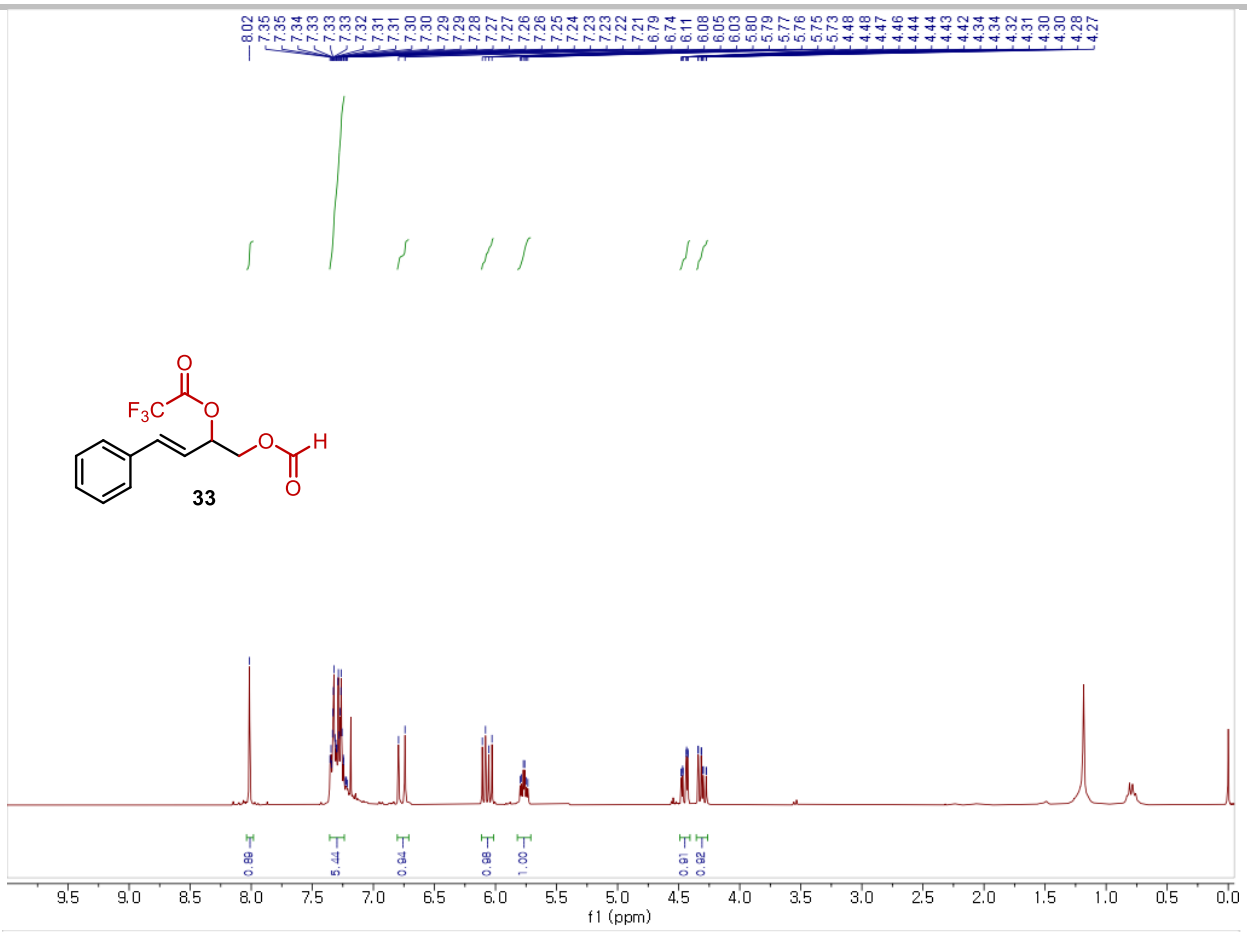
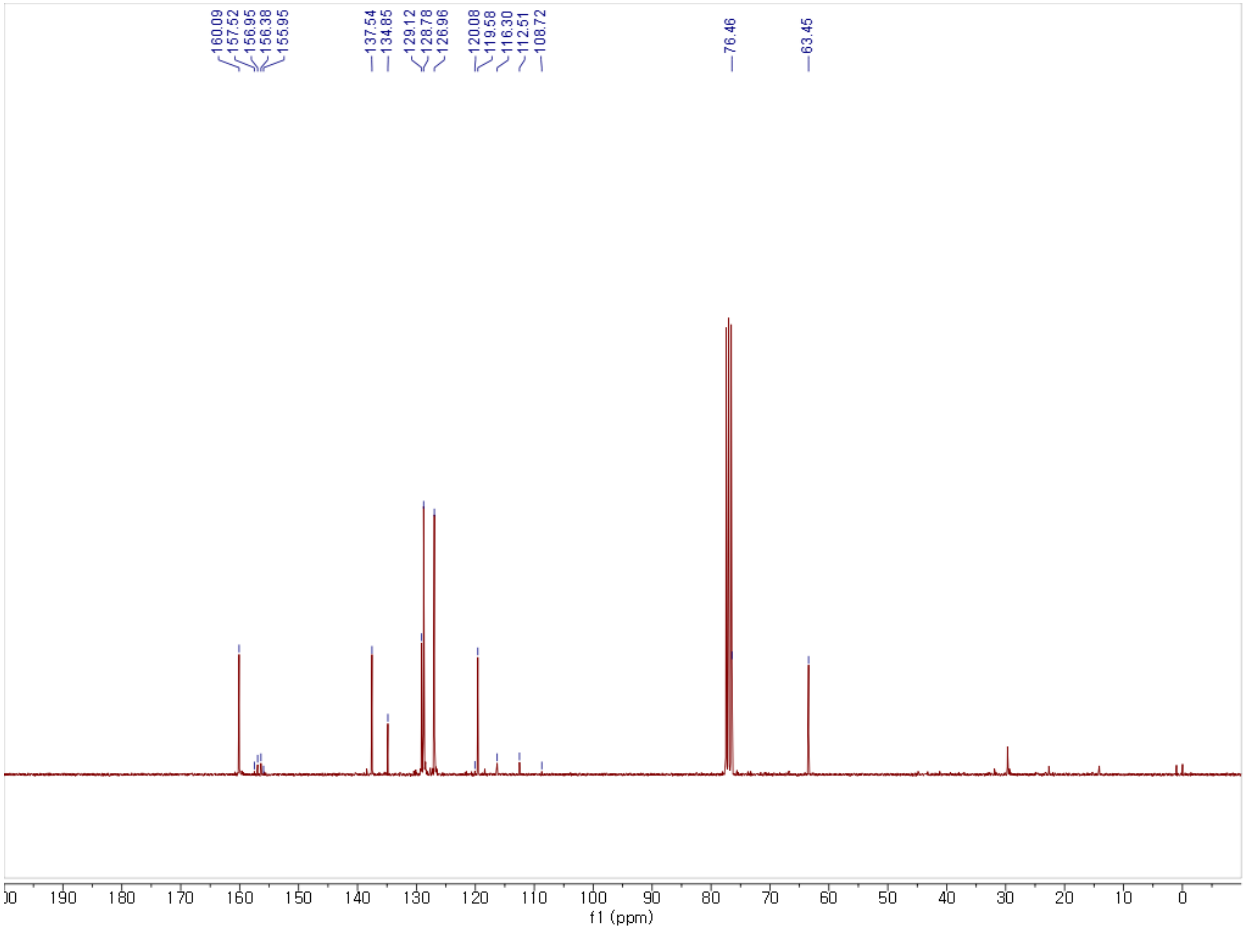
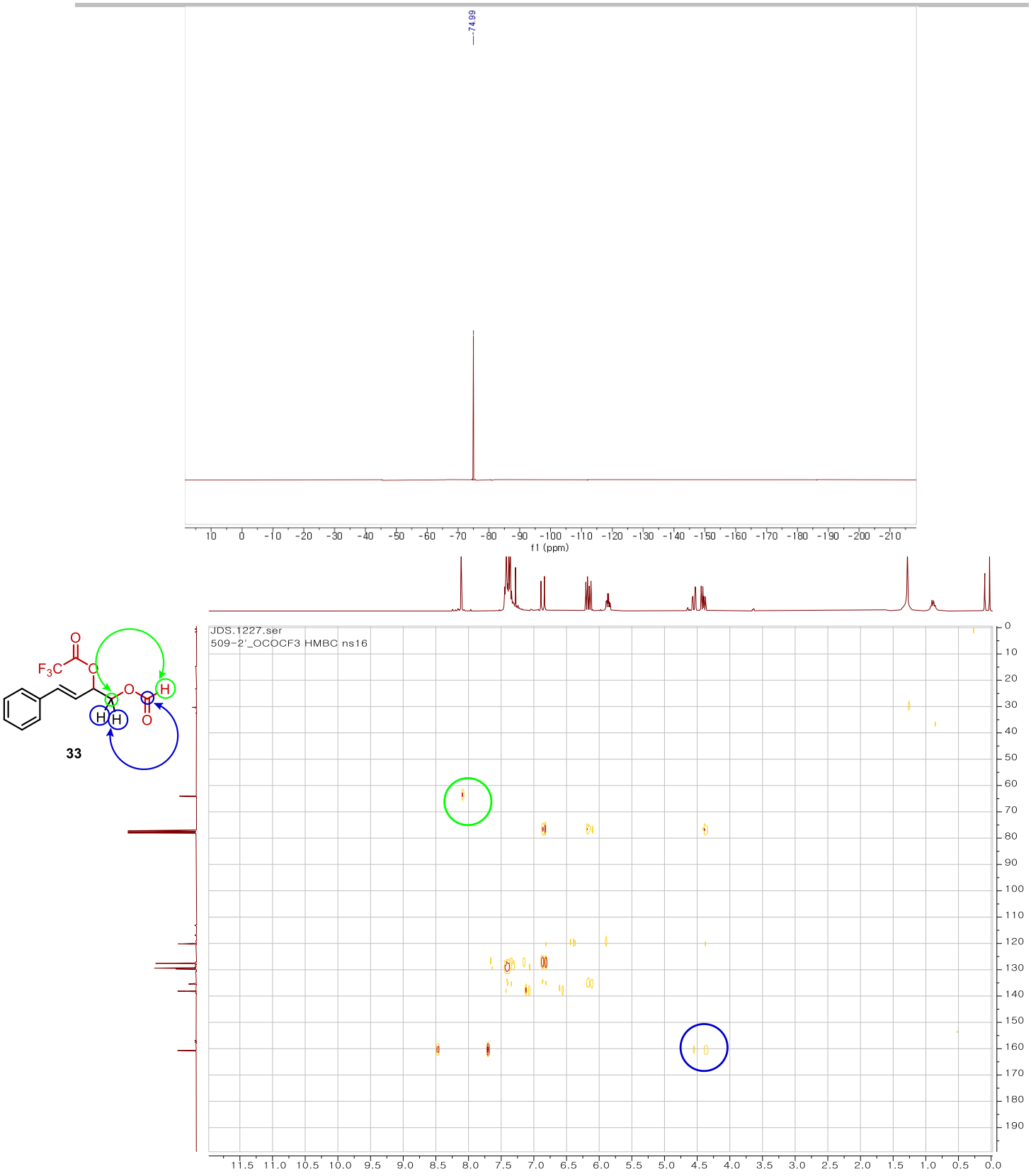
115
120
125
130
135
140
145
150
155
160
165
170
8.9 8.8 8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6
f2 (ppm)


8.15
8.02
7.42
7.41
7.40
7.39
7.39
7.38
7.38
7.37
7.36
7.36
7.35
7.35
7.34
7.33
7.33
7.32
7.31
7.30
6.06
6.04
6.01
5.99
5.97
5.94
5.07
5.03
5.03
4.98
3.08
3.05
3.03
3.00
2.97
2.93
2.91
2.89
2.86
2.84
H
O
O
Ph
H
O
Ph
O
31
0.88
0.88
4.23
4.18
1.00
1.02
2.03
1.98
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
f1 (ppm)

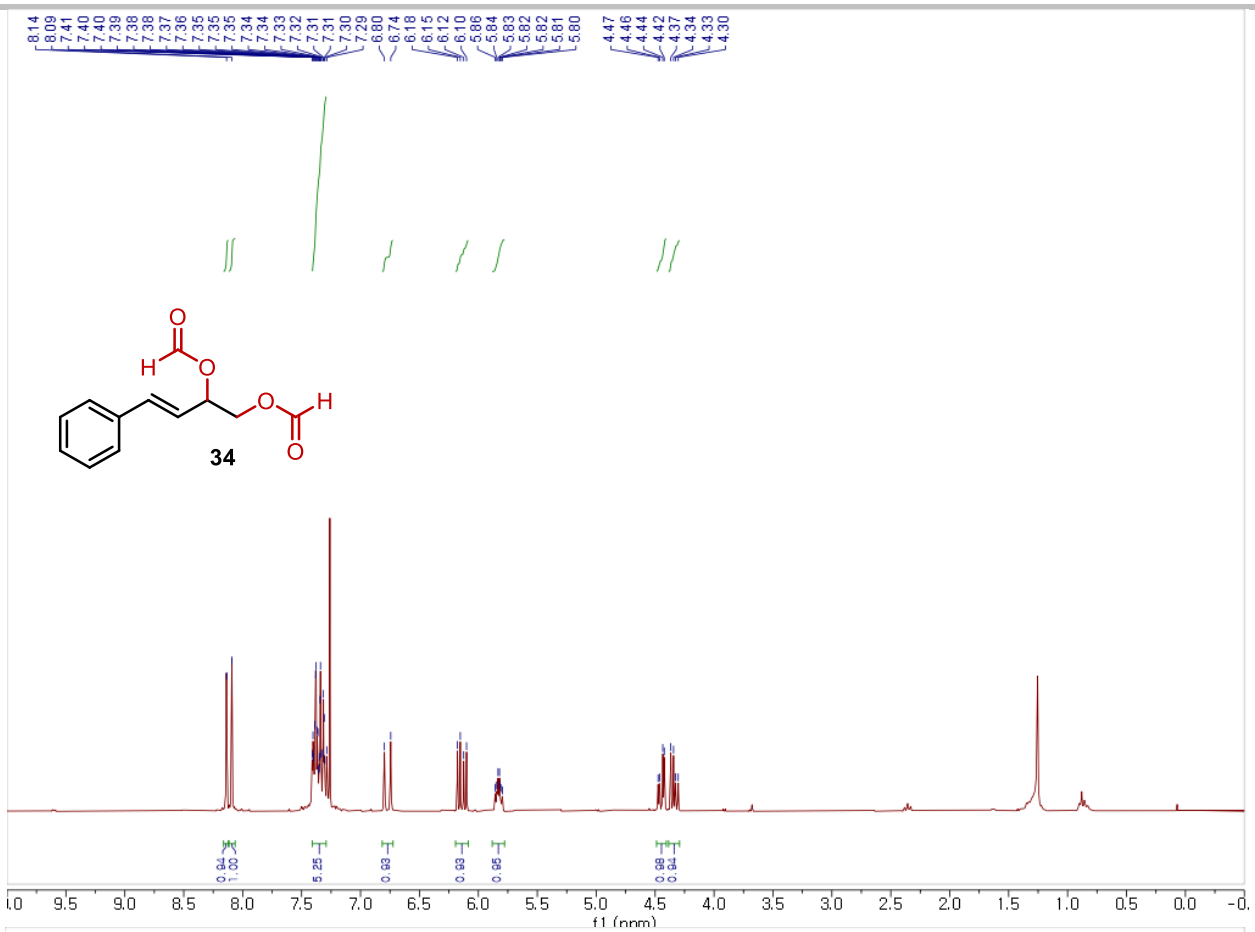

160.76
160.20
139.96
138.99
137.09
128.82
128.70
128.55
128.53
128.47
127.73
126.53
126.26
74.98
60.28
35.55
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

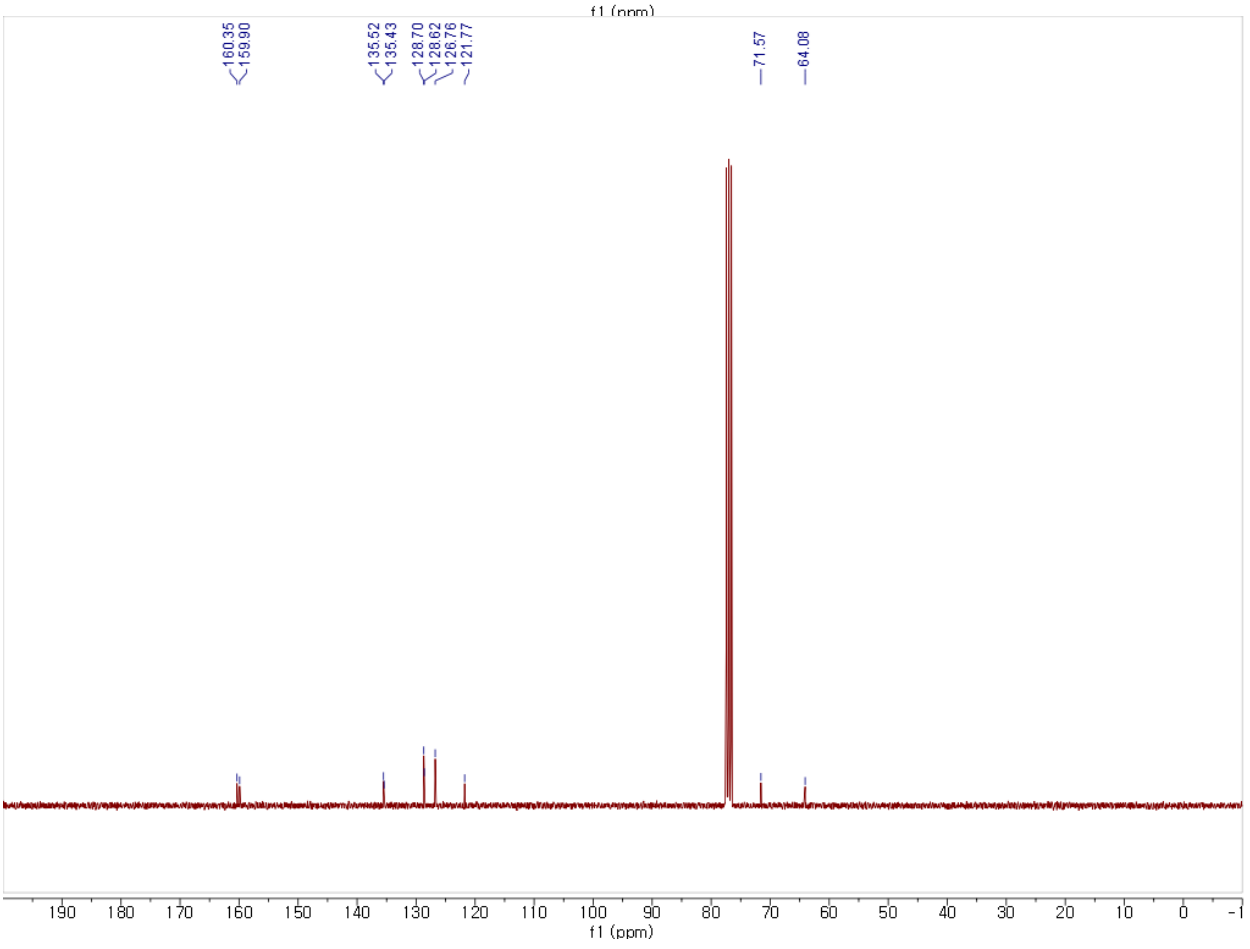

8.02
7.35
7.34
7.33
7.32
7.31
7.30
7.29
7.28
7.27
7.26
7.25
7.24
7.23
7.22
7.21
6.79
6.74
6.11
6.08
6.05
6.03
5.80
5.79
5.77
5.76
5.75
5.73
4.48
4.47
4.46
4.44
4.43
4.42
4.34
4.32
4.31
4.30
4.28
4.27
O
F3C
O
O
O
H
33
0.89
5.44
0.94
0.98
1.00
0.91
0.82
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

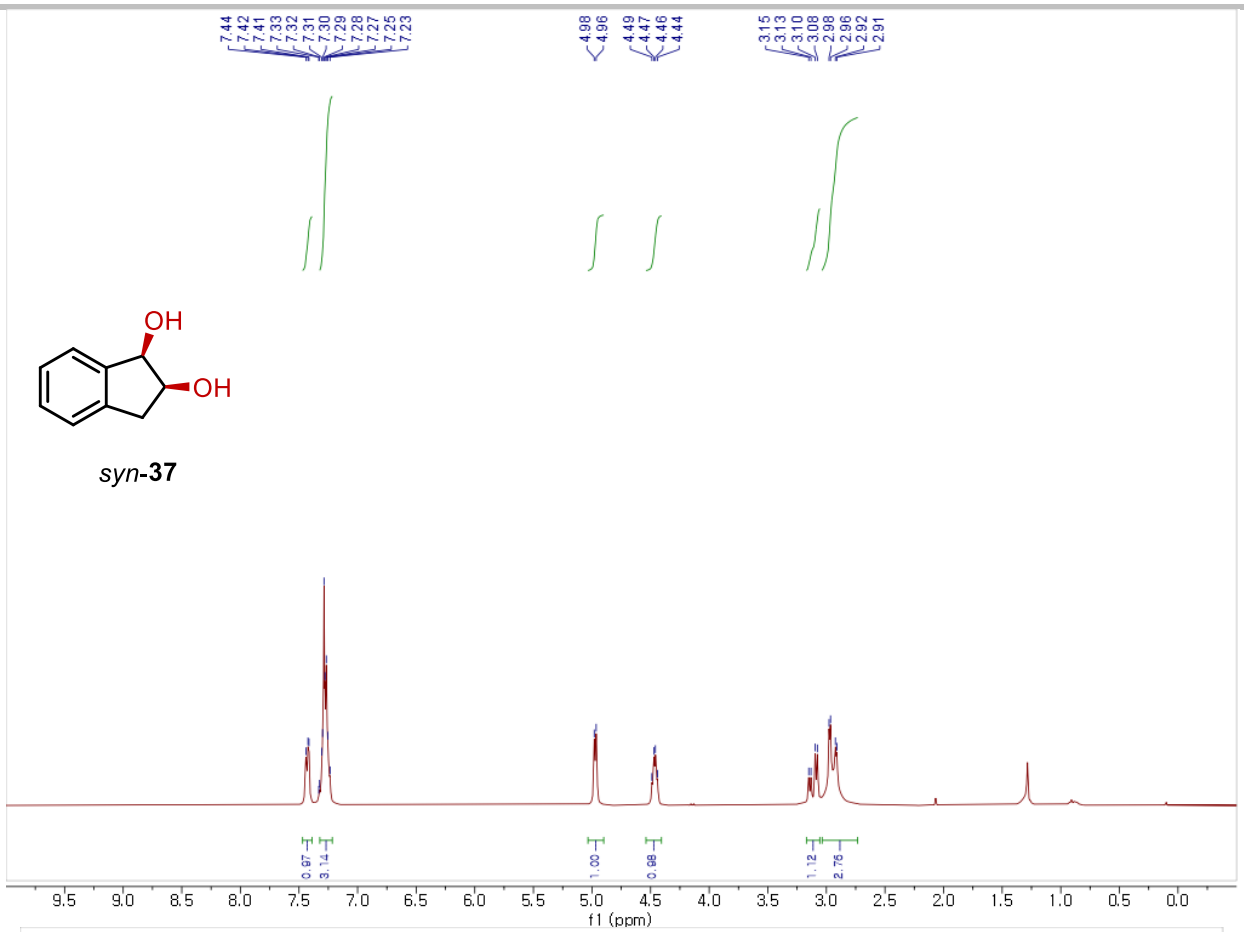

160.09
157.52
156.95
156.38
155.95
137.54
134.85
129.12
128.78
126.96
120.08
119.58
116.30
112.51
108.72
76.46
63.45
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-74.99
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)
JDS.1227.ser
509-2'_OCOCF3 HMBC ns16
11.5 11.0 10.5 10.0 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
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190

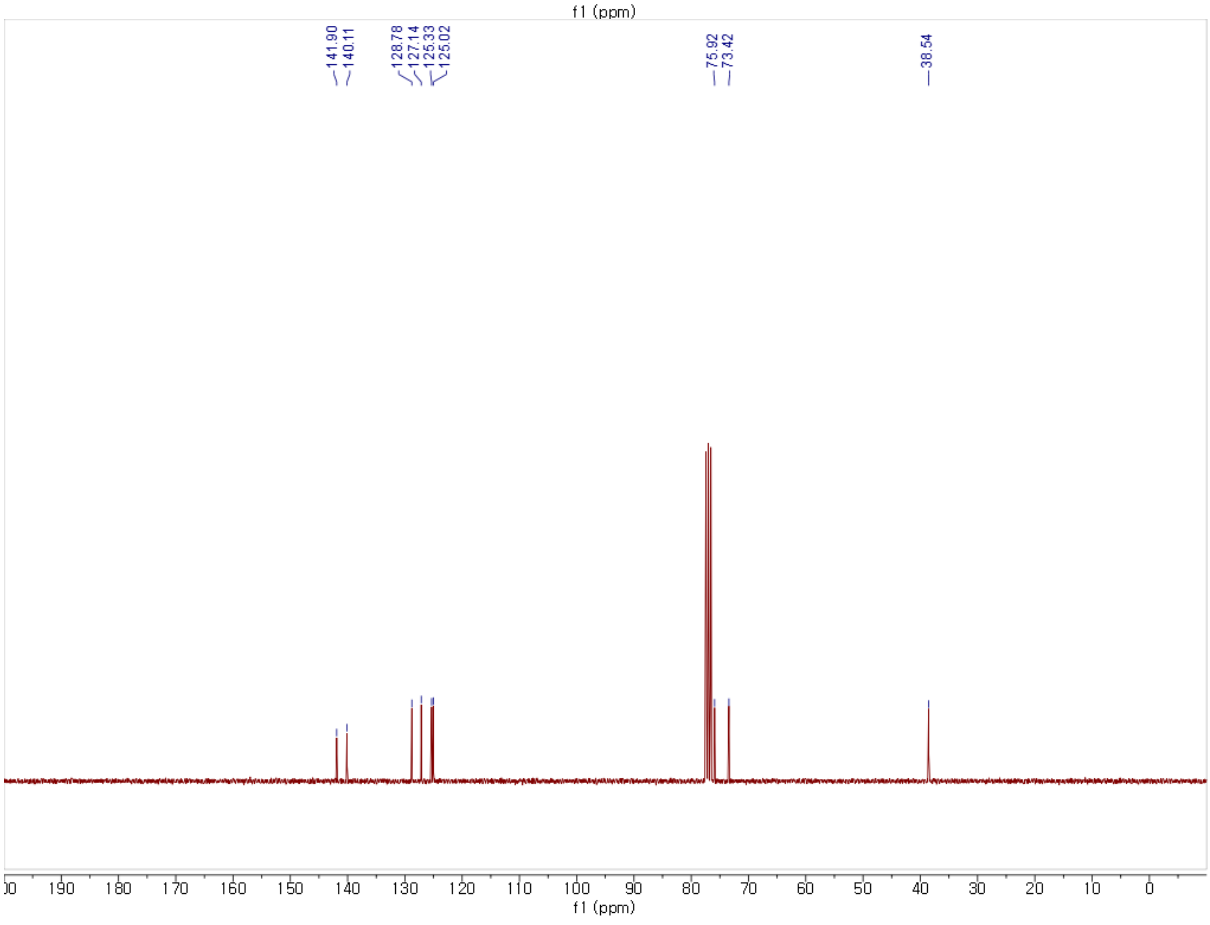
33

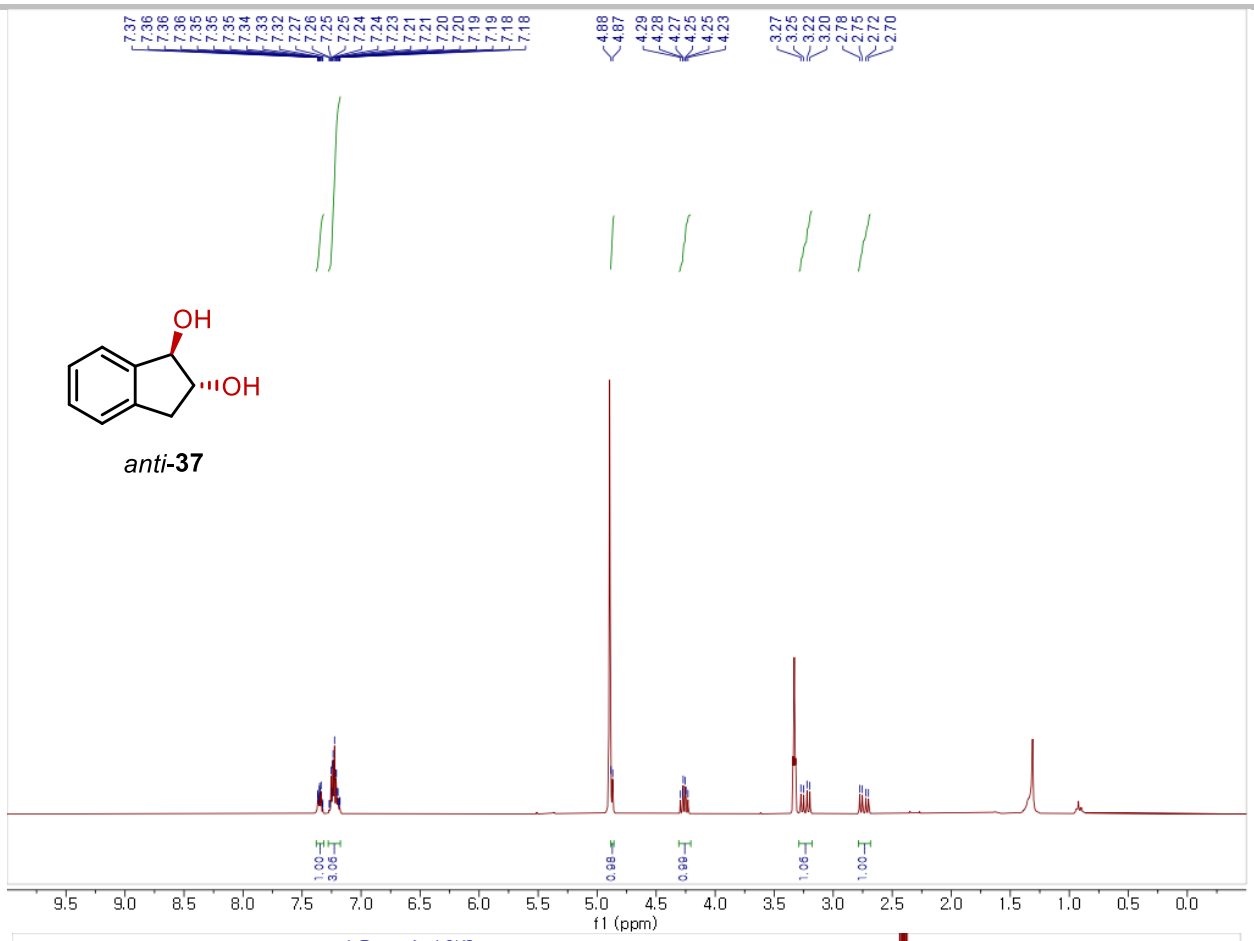

34
f1 (ppm)

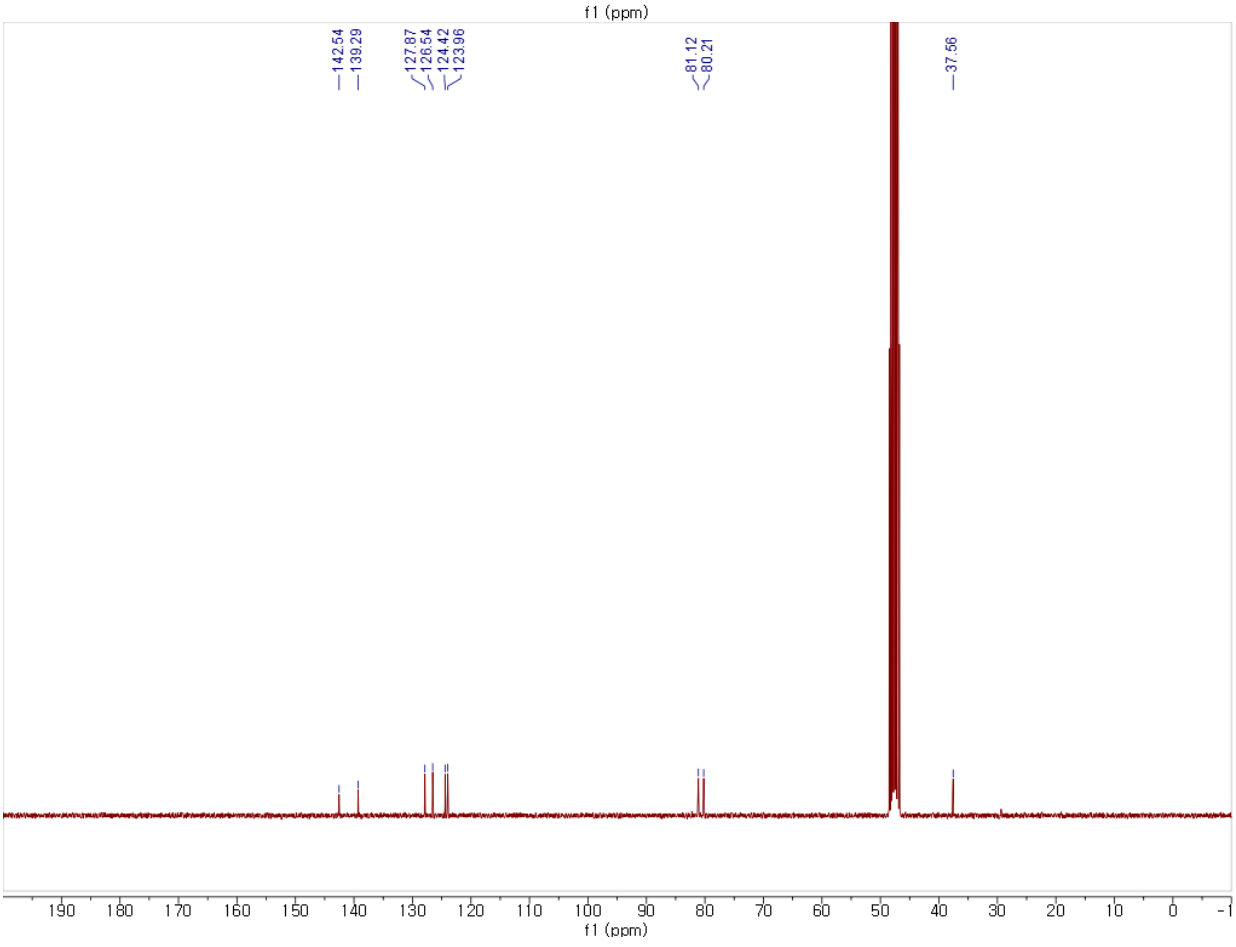

f1 (ppm)


7.44
7.42
7.41
7.33
7.32
7.31
7.30
7.29
7.28
7.27
7.25
7.23
4.98
4.96
4.49
4.47
4.46
4.44
3.15
3.13
3.10
3.08
2.98
2.96
2.92
2.91
OH
OH
0.97
3.14
1.00
0.98
1.12
2.78
f1 (ppm)

syn-**37**


141.90
140.11
128.78
127.14
125.33
125.02
75.92
73.42
38.54
f1 (ppm)


OH
OH
anti-37
f1 (ppm)


f1 (ppm)

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