Supporting Information

Electrochemical Oxidation of propargyl alcohol: A rapid access to unprecedented dioxo-orthoester under mild open-air condition

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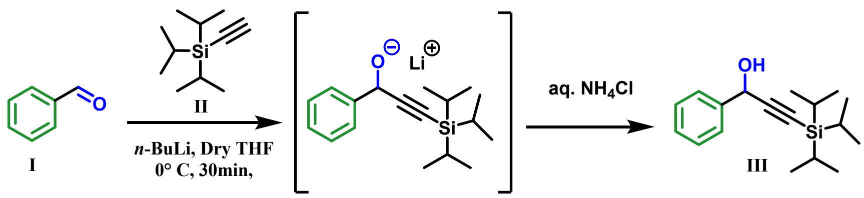
1. General Information

Electrochemical synthesis of all the 2,3-dioxo orthoester substrates 2a-2zc was performed inside a three-necked pear-shaped container (10 ml & 25 ml). Propargyl alcohols 1a-1zd are being synthesized according to the following procedures from their corresponding aryl aldehydes and (Triisopropylsilyl)acetylene. All reagents, especially aldehydes, (Triisopropylsilyl)acetylene, and n-BuLi, were bought from Sigma-Aldrich and used without any further purifications. Glass apparatuses are 4-5 h oven-dried, and solvents were dried as per their conventional drying procedures. Column chromatographic purifications were done using silica gel having a mesh size of 100-200. Constant current electrosynthesis was performed using Orgel® made by Eliteck Industries Private Ltd., an instrument that can supply constant current or constant voltage through a switch while stirring the electrolyte during the application of current or voltage with variable stirring speed (rpm). The current (0 to 100 mA) or voltage (0 to 30 V) can be controlled subtly with a knob. The isolated yield of the column-purified orthoesters have been recorded FTIR on the JASCO FT/IR-4600 instrument. ^{1}H, ^{13}C{^{1}H} & ^{19}F NMR spectra were recorded on BRUKER ASCEND 400 unit [^{1}H NMR (400 MHz), ^{13}C{1H} NMR (100 MHz) & ^{19}F NMR (376 MHz)] taking TMS (δ = 0 ppm) as internal standard and high-resolution mass spectra (HRMS) of all triazoles moieties have been recorded on WATERS XEVO G2- XS QT of mass spectrometer. Melting points were measured with a Lab X India digital melting point apparatus. The single crystal XRD data of 2zg was recorded on a BRUKER diffractometer and structure was elucidated through Apex-IV software. NMR splitting pattern singlet, doublet, triplet, quartet, heptet, doublet of doublet, doublet of triplet, doublet of quartet, quartet of doublet, doublet of doublet of triplet, doublet of doublet of doublet and multiplate have been abbreviated as s, d, t, q, hept, dd, dt, dq, qd, ddt, ddd and m respectively.

2.General synthetic procedure of Propargyl Alcohols 1a-1zc:

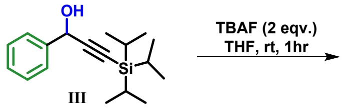
a. Typical procedure for the synthesis of silylated propargyl alcohols III.



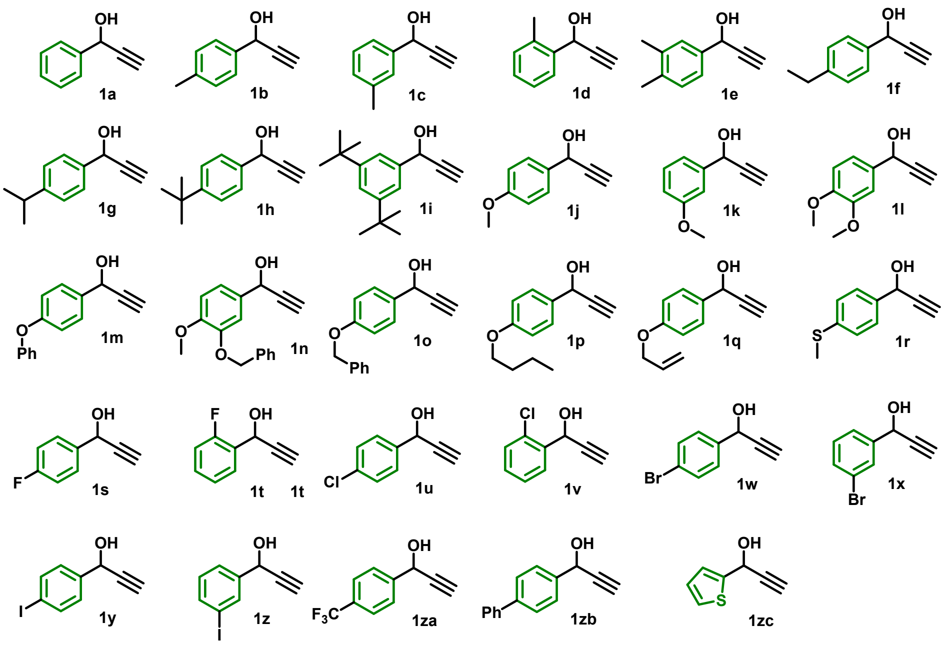
Scheme S1. Synthesis of silylated propargyl alcohol **III**

To a previously oven dried two-necked 50 ml round bottom flask 15 ml dry THF added which was then properly degassed and fitted with a N_2 balloon. Through a septa-syringe system (Triisopropylsilyl)acetylene **II** was added dropwise. The flask was cooled to 0°C, followed by n-BuLi (1.4 equivalents) was added dropwise over a span of 10 minutes. The mix was stirred for 15 min at that temp followed by the addition of the THF solution of Benzaldehyde **I** dropwise. Then the reaction mixture was stirred for another 30 min. The completion of the reaction was confirmed by T.L.C., and it was quenched using saturated ammonium chloride solution followed by the addition of ethyl acetate. The combine organic layer was collected on anhydrous sodium sulphate after the work up the reaction mixture. The concentrated organic solution was then purified through column chromatography (silica gel 100-200 mesh) using ethyl acetate: hexane (1:4) as eluent affording silylated propargyl alcohol [1] (**III**, 3.75 mmol, 80%), a yellow liquid.

b. Procedure of silyl group deprotection reaction to afford propargyl alcohol 1a.



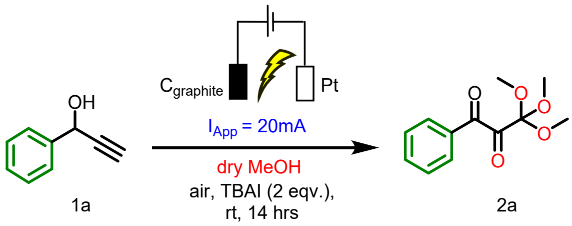
Scheme S2. Deprotection of silly group to synthesise propargyl alcohol **1a**



Silylated propargyl alcohol **III** was taken in an oven dried 50 ml round bottom flask followed by the addition of 10 ml THF. Then dropwise tetrabutyl ammoniumfluoride (TBAF, 75% in water) was added to the reaction mixture which was stirred for 1hr at rt. The completion of the reaction confirmed by T.L.C., and it was the quenched by ethyl acetate followed by water. The combine organic layer was collected on anhydrous sodium sulphate after the work up the reaction mixture. The concentrated organic solution was then purified through column chromatography (silica gel 100-200 mesh) using ethyl acetate: hexane (3:7) as eluent affording propargyl alcohol [2] (**1a**, 1.20 mmol, 76%), a light-yellow liquid.

Propargyl alcohols **1b-1zc** was synthesised using the same procedure mentioned above using corresponding aryl aldehydes and (Triisopropylsilyl)acetylene and TBAF.

3. Synthetic procedure of electrolysis of 2a.



Scheme S3. Electrochemical synthesis of Orthoester 2a

In a pear-shaped container **1** was taken followed by the addition of 2eqv. TBAI as electrolyte, which was then fitted with $C_{Graphite}$ as anode and spiral Pt wire as cathode. The electrolysis was done in dry MeOH medium at constant current flow 20mA and the mix was stirred at rt for 14hrs. The completion of reaction was confirmed by TLC and then it was quenched by ethyl acetate followed by saturated sodium thiosulphate solution. The combine organic layer was collected on anhydrous sodium sulphate after the work up the reaction mixture. The concentrated organic solution was then purified through column chromatography (silica gel 100-200 mesh) using ethyl acetate: hexane (1:9) as eluent affording 2,3-dioxoorthoester (**2a**, 1.20 mmol, 65%), a yellow liquid.

4. Control Experiment:

a) Proof for radical mechanism by Mass Spectrometry method

In a Pear-shaped container **1a** was electrolysed in MeOH along with 5eqv. of TEMPO using TBAI as electrolyte. as a result, the desire product **2a** was not obtained after the optimum reaction time and that suggests the radical mechanism. Hence it is also proved by the Mass Spectrometric detection of reaction mixture found a TEMPO adduct which is given in the fig. having protonated mass m/z = 414.2616.

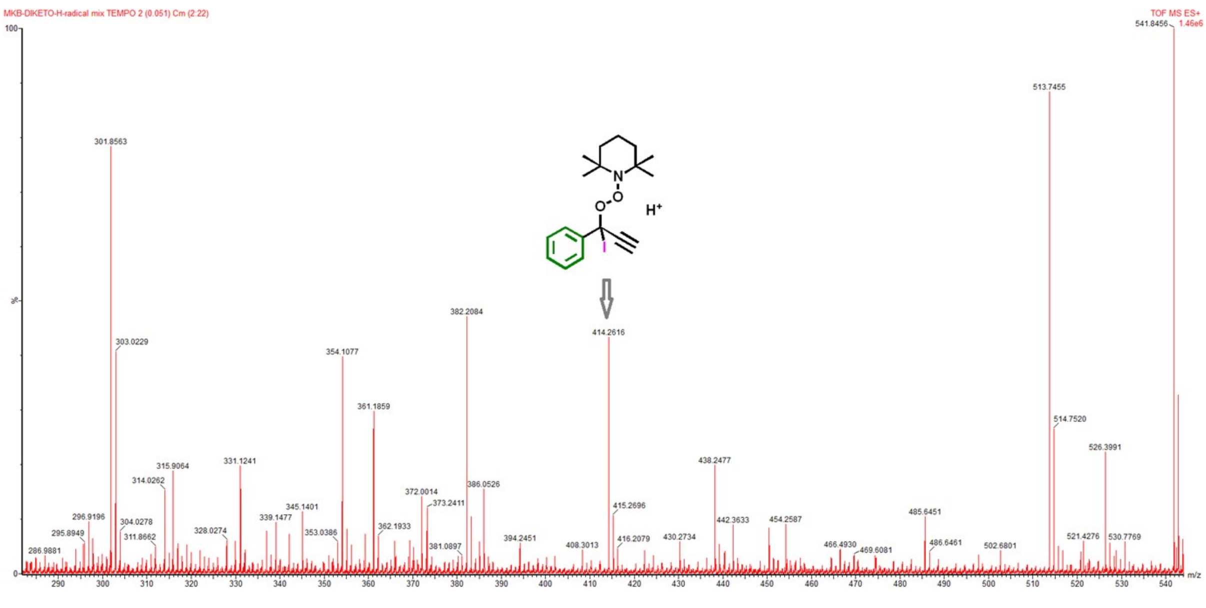


Fig1. HRMS data of TEMPO adduct with a radical intermediate.

b) HRMS data of some detected intermediates

To prove the mechanism, we have taken the HRMS data of the reaction mixture and we have found two intermediates from HRMS data. Between them 1st one is the protonated mass of the ynone intermediate (1a′) having m/z = 131.3485 (fig. 2) and another one is at m/z (M + Na) = 231.3916 of the intermediate (1ad′) shown in fig 3.

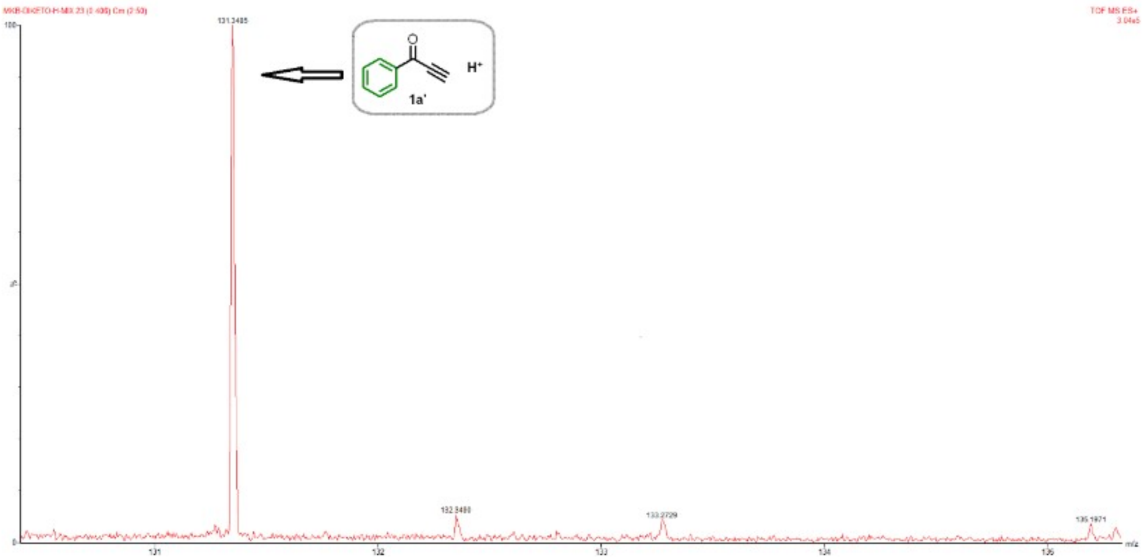


Fig2. HRMS data of intermediate **1a′**

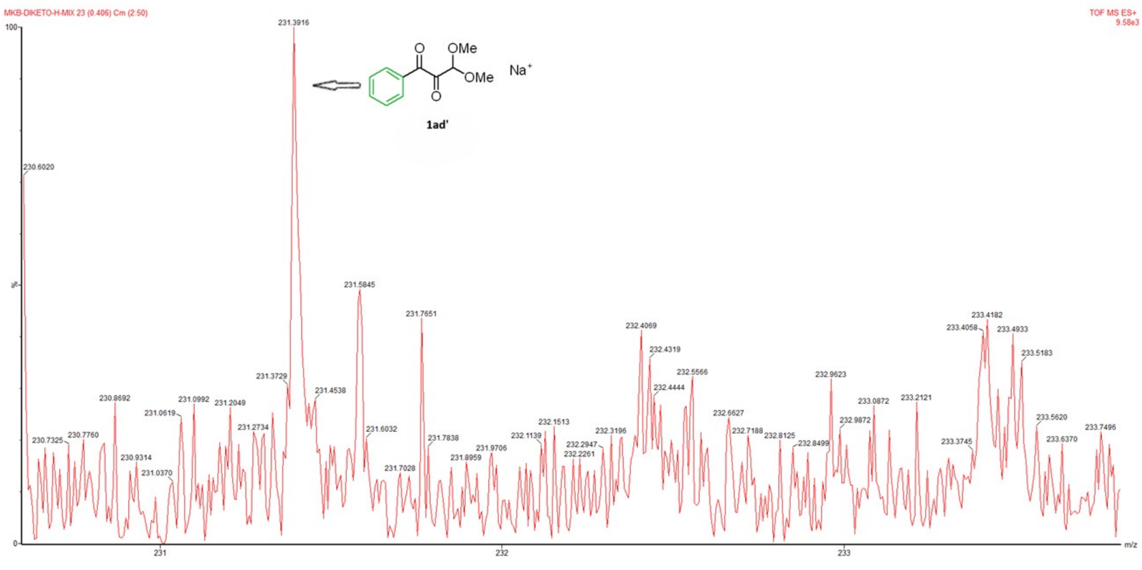
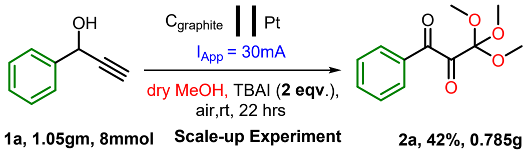


Fig3. HRMS data of intermediate **1ad′**

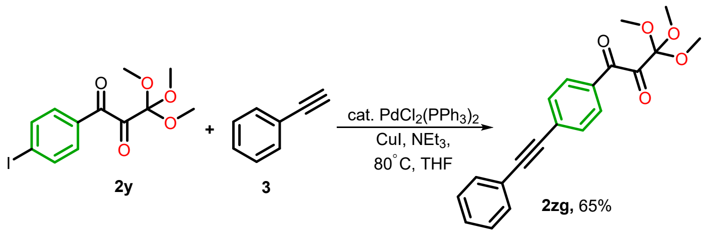
5. Gram Scale Synthesis



Scheme S4. Gram Scale synthesis of Orthoester 2a

To a 50 ml oven dried 3 neck pear-shaped container **1a** (1.05g, 8mmol) was charged followed by 2eqv. of TBAI as electrolyte. Which was then fitted with $C_{Graphite}$ as anode and Pt as cathode. The electrolysis was done in 20 ml dry MeOH medium at constant current flow 30mA and the mix was stirred at rt for 22hrs. The completion of reaction was confirmed by TLC and then it was quenched by ethyl acetate followed by saturated sodium thiosulphate solution. The combine organic layer was collected on anhydrous sodium sulphate after the work up the reaction mixture. The concentrated organic solution was then purified through column chromatography (silica gel 100-200 mesh) using ethyl acetate: hexane (1:9) as eluent affording 2,3-dioxoorthoester **2a** (0.785g, 42%), a yellow liquid.

6. Synthetic Transformation



Scheme S5. Sonogashira Coupling reaction of 2y

To an oven dried seal tube **2y** (75 mg, 0.2 mmol) was taken along with Phenyl Acetylene **3** (31mg, 0.3mmol). 10 mol% of $PdCl_2(PPh_3)_2$ used as catalyst followed by the addition of 10 mol% of CuI and stirred at 80°C in THF medium for 2hr after 2eqv. of NEt_3 addition. After getting confirmation of reaction completion from TLC profile it was quenched by EtOAc followed by water. The combine organic layer was collected on anhydrous sodium sulphate after the work up the reaction mixture. The concentrated organic solution was then purified through column chromatography (silica gel 100-200 mesh) using ethyl acetate: hexane (1:4) as eluent affording another 2,3-dioxoorthoester as Sonogashira coupling product [3] (**2zg**, 44.8mg, 0.13 mmol, 65%), a pale-yellow solid (m.p.- 76°-80°C).

7. Reference:

1. R. De; A. Savarimuthu; T. Ballav; P. Singh; J. Nanda; A. Hasija; D. Chopra; M. K. Bera, *Synlett*, 2020, **31**, 1587-1592; R. De; A. Savarimuthu; S. Chandra; M. K. Bera, *New J. Chem*. 2021, **45**, 17871-17877; R. De; A. Hasija; D. Chopra; M. K. Bera, *Tetrahedron.* 2022, 120, 132906.
2. M. Dominguez, H.-U. Reissig, *Synthesis,* 2014**, 46**, 1100-1106.
3. K. Sonogashira, Y. Tohda and N.Hagihara, *Tetrahedron Letters*, 1975, **16**, 4467–4470.

8. Cyclic Voltametric Study

The cyclic voltammetry was employed to investigate the electrochemical behaviour of 1a (starting material) in methanol using reactive electrolyte (0.1 (M) *n*-Bu_4NI) and inert supporting electrolyte (0.1 (M) *n*-Bu_4NPF_6). Measurement was conducted using glassy carbon working electrode of active area 0.07 cm^2, Pt-spring auxiliary electrode and an Ag/AgCl (in saturated KCl) as reference electrode. In presence of reactive electrolyte 0.1(M) *n*-Bu_4NI, the broad reductive peak at -0.9V attributed to the background electrolyte was shifted towards more negative -1.2V on addition of 1a (Fig. 4a), highlighting the interaction between the iodine radical and 1a as predicted in mechanism and supported by DFT. A similar broad reductive signal was observed using the inert electrolyte at -0.5V which shifted to -0.87V in presence of 1a (Fig. 4b)

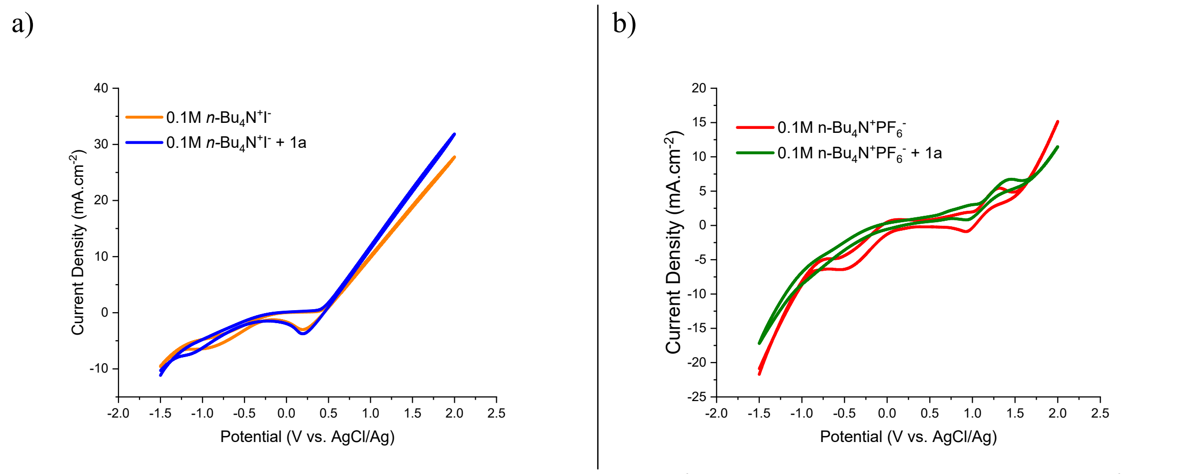


Fig. 4. a. We used AgCl/Ag as the reference electrode Glassy Carbon (0.07 cm^2) as working electrode and spiral Pt wire (1.76 cm^2) as the auxiliary electrode to perform cyclic voltametric study of 0.1 (M) *n*-Bu_4NI (orange) solution in 15 ml MeOH and compared with 10 mM 1a in 0.1 (M) *n*-Bu_4NI in 15 ml MeOH (blue). b. Cyclic voltammetry of 10 (mM) Starting Material 1a (green) in 0.1 (M) *n*-Bu_4NPF_6 electrolytic solution using 0.07 cm^2 Glassy Carbon as working electrode against AgCl/Ag as reference electrode and spiral Pt wire (1.76 cm^2) as the auxiliary electrode at a scan rate of 50 $mV.s^{-1}$, compared to a blank i.e. 0.1 (M) *n*-Bu_4NPF_6 electrolytic solution (red).

9. Computational details

All density functional theory calculations were performed using Gaussian16.[1] Geometry optimizations were performed at the wB97xD[2] level using the valence triple-ζ-type of basis setDef2TZVP[3] for all atoms except I, for which the SDD basis set (Stuttgart/Dresden ECP) was used, and this basis is augmented by an additional d-type polarization function for I with ζd = 1.050 and for Ag with ζf = 0.289.[4] The solvent effects (methanol) were evaluated implicitly by a self-consistent reaction field (SCRF) approach for all the intermediates and transition states, using the SMD continuum solvation model.[5] Unless specified otherwise, Δ*G* was used throughout the text. Frequency calculations of reactants and transition states were performed at the same level of theory. The optimized geometries were confirmed to be minima (no imaginary frequencies) or transition structures (one imaginary frequency) by frequency calculations. All transition states were optimized using the default Berny algorithm implemented in the Gaussian 16 code. Intrinsic reaction coordinate (IRC) calculations were performed to verify the expected connections of the first-order saddle points with the local minima found on the potential energy surface.[6] Hirshfeld spin distributions were computed with Multiwfn 3.8.[7] CYLview was used to create images of optimized structures.[8]

a. References.

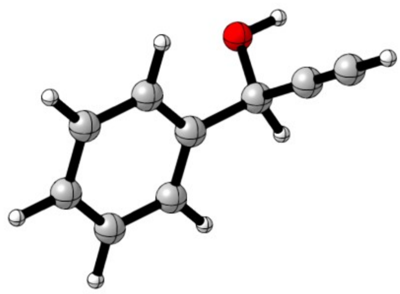
[1] Gaussian 16, Revision B.01, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, G. A. Petersson, H. Nakatsuji, X. Li, M. Caricato, A. V. Marenich, J. Bloino, B. G. Janesko, R. Gomperts, B. Mennucci, H. P. Hratchian, J. V. Ortiz, A. F. Izmaylov, J. L. Sonnenberg, D. Williams-Young, F. Ding, F. Lipparini, F. Egidi, J. Goings, B. Peng, A. Petrone, T. Henderson, D. Ranasinghe, V. G. Zakrzewski, J. Gao, N. Rega, G. Zheng, W. Liang, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, K. Throssell, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. J. Bearpark, J. J. Heyd, E. N. Brothers, K. N. Kudin, V. N. Staroverov, T. A. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. P. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, J. M. Millam, M. Klene, C. Adamo, R. Cammi, J. W. Ochterski, R. L. Martin, K. Morokuma, O. Farkas, J. B. Foresman, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2016.
[2] J. D. Chai, M. Head-Gordon, *Phys. Chem. Chem. Phys*., 2008, **10**, 6615–6620.
[3] (a) F. Weigend, R. Ahlrichs, *Phys. Chem. Chem. Phys*., 2005, **7**, 3297–3305; (b) F. Weigend, *Phys. Chem. Chem. Phys*., 2006, **8**, 1057–1065.
[4] A. HGllwarth, M. Biihme, S. Dapprich, A. W. Ehlers, A. Gobbi, V. Jonas, K. F. Kijhler, R. Stegmann, A. Veldkamp, G. Frenking, *Chem. Phys. Lett.*, 1993, **208**, 237–240.
[5] A. V. Marenich, C. J. Cramer, D. G. Truhlar, *J. Phys. Chem. B*, 2009, **113**, 6378–6396.
[6] C. Gonzalez and H. B. Schlegel, *J. Phys. Chem*., 1990, **94**, 5523–5527.
[7] (a) T. Lu, F. Chen, Multiwfn: *J. Comput. Chem*., 2012, **33**, 580-592; (b) T. Lu, J. *Chem. Phys*., 2024, **161**, 082503.
[8] C. Y. Legault, CYLview20. Canada Université de Sherbrooke 2020, http://www.cylview.org.

b. Energies and cartesian coordinates of optimized structures

1a

Zero-point correction= 0.143571 (Hartree/Particle)
Thermal correction to Energy= 0.152506
Thermal correction to Enthalpy= 0.153450
Thermal correction to Gibbs Free Energy= 0.108602
Sum of electronic and zero-point Energies= -422.799533
Sum of electronic and thermal Energies= -422.790598
Sum of electronic and thermal Enthalpies= -422.789654
Sum of electronic and thermal Free Energies= -422.834502

6	-3.908192000	21.599053000	-6.371292000
6	-2.840932000	22.483747000	-6.246692000
6	-3.718473000	20.254147000	-6.087116000
6	-1.599997000	22.025257000	-5.836716000
6	-2.472295000	19.792548000	-5.682820000
6	-1.412782000	20.677094000	-5.555493000
1	-2.982743000	23.534183000	-6.470381000
1	-4.547437000	19.563263000	-6.181269000
1	-0.774868000	22.719215000	-5.739528000
1	-2.332950000	18.741468000	-5.464646000
1	-0.441639000	20.319324000	-5.238096000
6	-5.270845000	22.103184000	-6.796447000
8	-5.713096000	23.046175000	-5.828192000
6	-5.229841000	22.701957000	-8.140795000
6	-5.211073000	23.187512000	-9.236720000
1	-5.190689000	23.619401000	-10.212313000
1	-6.611973000	23.308324000	-6.042902000
1	-5.959960000	21.254876000	-6.830949000

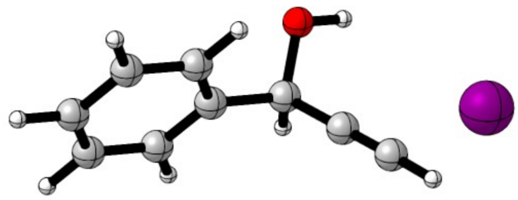


RC1

Zero-point correction= 0.144158 (Hartree/Particle)
Thermal correction to Energy= 0.155416
Thermal correction to Enthalpy= 0.156360
Thermal correction to Gibbs Free Energy= 0.102058
Sum of electronic and zero-point Energies= -434.210656
Sum of electronic and thermal Energies= -434.199398

Sum of electronic and thermal Enthalpies= -434.198454
Sum of electronic and thermal Free Energies= -434.252757

6	-3.947049000	21.690530000	-6.339314000
6	-2.836369000	22.525347000	-6.259497000
6	-3.812710000	20.339593000	-6.053115000
6	-1.604860000	22.010340000	-5.890297000
6	-2.576200000	19.822305000	-5.689172000
6	-1.472216000	20.656483000	-5.606049000
1	-2.936782000	23.579897000	-6.485395000
1	-4.676295000	19.688696000	-6.113181000
1	-0.744928000	22.664785000	-5.827644000
1	-2.479032000	18.767304000	-5.467424000
1	-0.508486000	20.254884000	-5.320117000
6	-5.295544000	22.254773000	-6.733723000
8	-5.632771000	23.295109000	-5.831910000
6	-5.264593000	22.745538000	-8.121844000
6	-5.243321000	23.145325000	-9.257295000
1	-5.174498000	23.487608000	-10.266370000
1	-6.508607000	23.623261000	-6.058624000
1	-6.039407000	21.454745000	-6.684351000
53	-8.229771000	23.829483000	-8.665386000



TS1

Zero-point correction= 0.138003 (Hartree/Particle)
Thermal correction to Energy= 0.148674
Thermal correction to Enthalpy= 0.149618
Thermal correction to Gibbs Free Energy= 0.097321
Sum of electronic and zero-point Energies= -434.171716
Sum of electronic and thermal Energies= -434.161046
Sum of electronic and thermal Enthalpies= -434.160101
Sum of electronic and thermal Free Energies= -434.212398
Frequency -968.6727

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6	-3.774115000	20.300880000	-6.059405000
6	-1.624068000	22.040684000	-5.857621000
6	-2.530054000	19.823960000	-5.666888000
6	-1.454752000	20.692733000	-5.564205000
1	-2.993514000	23.565572000	-6.484657000
1	-4.615639000	19.623019000	-6.134494000
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1	-0.485356000	20.322884000	-5.255457000
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6	-5.271801000	22.654505000	-8.163900000
6	-5.277511000	23.075512000	-9.293575000
1	-5.232614000	23.428067000	-10.300644000
1	-6.721210000	23.715426000	-6.435941000
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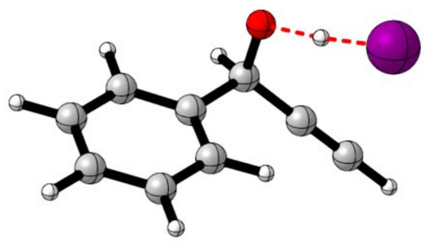
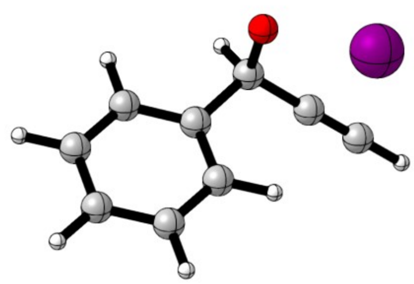


RC2

Thermal correction to Enthalpy= 0.142846
Thermal correction to Gibbs Free Energy= 0.089556
Sum of electronic and zero-point Energies= -433.540839
Sum of electronic and thermal Energies= -433.529900
Sum of electronic and thermal Enthalpies= -433.528956
Sum of electronic and thermal Free Energies= -433.582246

6	-5.437374000	21.052148000	-4.668175000
6	-6.621206000	20.788998000	-3.984315000
6	-4.218866000	20.595187000	-4.174480000

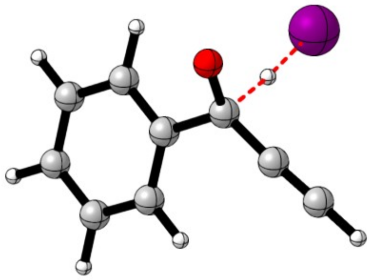


6	-6.588594000	20.024695000	-2.831424000
6	-4.194353000	19.831552000	-3.019671000
6	-5.376327000	19.546145000	-2.349215000
1	-7.559763000	21.170474000	-4.365305000
1	-3.299877000	20.820563000	-4.699520000
1	-7.508496000	19.801782000	-2.307227000
1	-3.251002000	19.461044000	-2.641125000
1	-5.353399000	18.950761000	-1.445669000
6	-5.487577000	21.921869000	-5.929962000
8	-5.283038000	23.153510000	-5.342414000
6	-4.507597000	21.567664000	-6.958321000
6	-3.727305000	21.282927000	-7.820769000
1	-3.027659000	21.031477000	-8.586512000
1	-6.497625000	21.871282000	-6.349765000
53	-2.755189000	24.284847000	-5.061379000

TS2

Zero-point correction= 0.123341 (Hartree/Particle)
Thermal correction to Energy= 0.134068
Thermal correction to Enthalpy= 0.135012
Thermal correction to Gibbs Free Energy= 0.082499
Sum of electronic and zero-point Energies= -433.526015
Sum of electronic and thermal Energies= -433.515288
Sum of electronic and thermal Enthalpies= -433.514344
Sum of electronic and thermal Free Energies= -433.566858
Frequency -554.2619

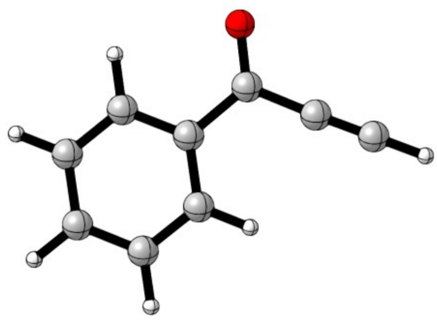
6	-5.541937000	21.116929000	-4.692214000
6	-6.619709000	21.291572000	-3.818303000
6	-4.521958000	20.215542000	-4.385108000
6	-6.672319000	20.561126000	-2.644639000
6	-4.596688000	19.479564000	-3.217908000
6	-5.665488000	19.654383000	-2.344615000
1	-7.417684000	21.975594000	-4.074146000
1	-3.684741000	20.091816000	-5.058875000
1	-7.506610000	20.691705000	-1.968553000
1	-3.815473000	18.768806000	-2.983233000
1	-5.712713000	19.077609000	-1.430242000
6	-5.508344000	21.904094000	-5.947502000
8	-5.828569000	23.169912000	-5.720472000
6	-4.533792000	21.686732000	-6.960997000
6	-3.774199000	21.485638000	-7.869530000
1	-3.085929000	21.304067000	-8.665495000
1	-6.736656000	21.302156000	-6.674442000
53	-8.223708000	20.715253000	-7.449165000



1a'

Zero-point correction= 0.120174 (Hartree/Particle)
Thermal correction to Energy= 0.128420
Thermal correction to Enthalpy= 0.129364
Thermal correction to Gibbs Free Energy= 0.086325
Sum of electronic and zero-point Energies= -421.611206
Sum of electronic and thermal Energies= -421.602960
Sum of electronic and thermal Enthalpies= -421.602016
Sum of electronic and thermal Free Energies= -421.645056

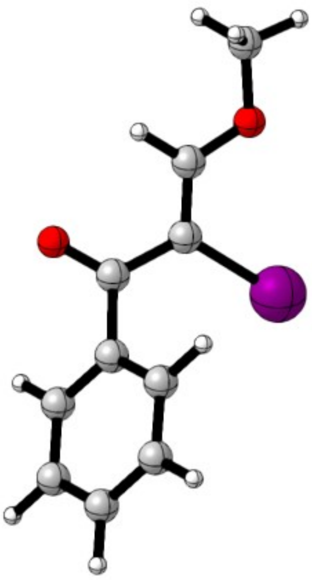
6	-3.656927000	21.967757000	-6.453824000
6	-2.936687000	22.472898000	-5.370553000
6	-3.287705000	20.751999000	-7.025685000
6	-1.860005000	21.767224000	-4.867947000
6	-2.208009000	20.047858000	-6.518695000
6	-1.495220000	20.554495000	-5.441830000
1	-3.226960000	23.417117000	-4.929922000
1	-3.842421000	20.355396000	-7.865972000



1 -1.302001000 22.159575000 -4.028104000
1 -1.922919000 19.104143000 -6.964279000
1 -0.651385000 20.003760000 -5.046333000
6 -4.805235000 22.744174000 -6.971984000
8 -5.157973000 23.806585000 -6.493883000
6 -5.536869000 22.200280000 -8.104780000
6 -6.164368000 21.783350000 -9.037070000
1 -6.723609000 21.411461000 -9.867971000

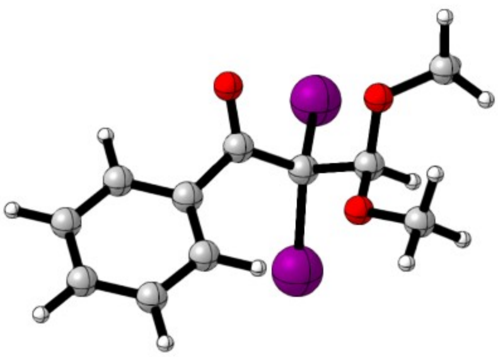
1aa’

Zero-point correction= 0.167375 (Hartree/Particle)
Thermal correction to Energy= 0.180005
Thermal correction to Enthalpy= 0.180949
Thermal correction to Gibbs Free Energy= 0.125296
Sum of electronic and zero-point Energies= -834.549539
Sum of electronic and thermal Energies= -834.536908
Sum of electronic and thermal Enthalpies= -834.535964
Sum of electronic and thermal Free Energies= -834.591618
6 -3.510317000 21.996535000 -6.598641000
6 -3.066748000 22.363551000 -5.331406000
6 -2.962640000 20.881413000 -7.227042000
6 -2.099378000 21.608491000 -4.688658000
6 -1.974882000 20.144754000 -6.593594000
6 -1.549873000 20.501487000 -5.321098000
1 -3.490904000 23.234391000 -4.848417000
1 -3.293699000 20.601135000 -8.218779000
1 -1.770048000 21.886365000 -3.695919000
1 -1.538584000 19.289178000 -7.092268000
1 -0.787254000 19.917005000 -4.822527000
6 -4.509123000 22.861429000 -7.291021000
8 -4.394224000 24.077993000 -7.226950000
6 -5.607316000 22.256692000 -8.064655000
6 -6.080418000 22.927136000 -9.127958000
1 -5.620438000 23.868917000 -9.415417000
8 -7.077891000 22.495298000 -9.875841000
6 -7.398084000 23.290860000 -11.023295000
1 -7.672296000 24.300193000 -10.716472000
1 -6.550709000 23.319094000 -11.708190000
1 -8.244473000 22.806062000 -11.500787000
53 -6.482632000 20.447530000 -7.496423000



1ab’

Zero-point correction= 0.212274 (Hartree/Particle)
Thermal correction to Energy= 0.229611
Thermal correction to Enthalpy= 0.230555
Thermal correction to Gibbs Free Energy= 0.163220
Sum of electronic and zero-point Energies= -674.714890
Sum of electronic and thermal Energies= -674.697553
Sum of electronic and thermal Enthalpies= -674.696609
Sum of electronic and thermal Free Energies= -674.763944
6 -7.303882000 24.101394000 -5.705354000
6 -7.951088000 23.732711000 -4.526498000
6 -6.788936000 25.388495000 -5.834021000
6 -8.061742000 24.632968000 -3.480231000
6 -6.929630000 26.294249000 -4.795441000
6 -7.555472000 25.917561000 -3.615501000
1 -8.355125000 22.733748000 -4.428813000
1 -6.301106000 25.687049000 -6.749119000
1 -8.550141000 24.332696000 -2.562494000
1 -6.544088000 27.299038000 -4.908521000
1 -7.650865000 26.626150000 -2.802793000
6 -7.285900000 23.073776000 -6.789558000

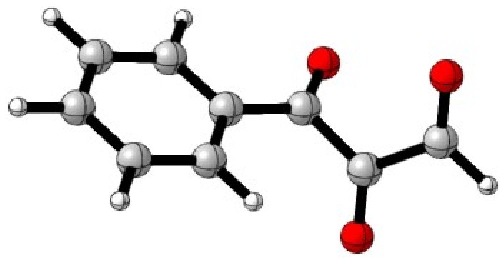


8	-8.293915000	22.449593000	-7.019296000
6	-6.020875000	22.738585000	-7.627432000
53	-5.971441000	20.579512000	-7.914216000
6	-6.081424000	23.408330000	-9.031685000
8	-6.146100000	24.777052000	-8.809637000
6	-5.990882000	25.564737000	-9.979012000
1	-5.063947000	25.305010000	-10.498504000
1	-6.838285000	25.439826000	-10.656028000
1	-5.943807000	26.601264000	-9.652432000
1	-5.173193000	23.146733000	-9.586478000
8	-7.211792000	22.976607000	-9.722424000
6	-6.975991000	22.464040000	-11.027317000
1	-6.486043000	23.205834000	-11.662067000
1	-6.367517000	21.557715000	-10.991099000
1	-7.951263000	22.222679000	-11.444533000
53	-4.145173000	23.292798000	-6.678788000

1ac'

Zero-point correction= 0.129382 (Hartree/Particle)
Thermal correction to Energy= 0.139892
Thermal correction to Enthalpy= 0.140836
Thermal correction to Gibbs Free Energy= 0.090897
Sum of electronic and zero-point Energies= -572.108648
Sum of electronic and thermal Energies= -572.098139
Sum of electronic and thermal Enthalpies= -572.097194
Sum of electronic and thermal Free Energies= -572.147133

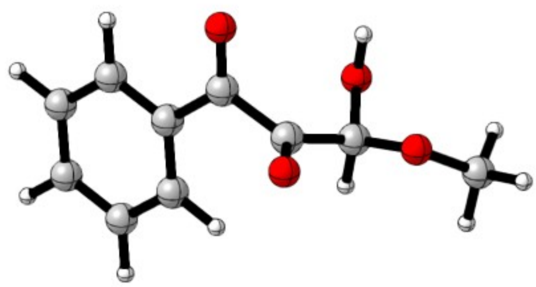
6	1.456382000	1.476982000	0.439128000
8	1.689328000	2.658003000	0.585992000
6	2.465554000	0.439213000	0.198484000
6	2.091175000	-0.879463000	-0.058462000
6	3.816167000	0.793729000	0.208241000
6	3.062770000	-1.835835000	-0.301811000
6	4.780546000	-0.165317000	-0.030782000
6	4.403396000	-1.479709000	-0.285430000
1	1.047766000	-1.166392000	-0.074782000
1	4.096770000	1.819811000	0.405423000
1	2.773930000	-2.858600000	-0.503009000
1	5.827725000	0.106017000	-0.020639000
1	5.161323000	-2.229504000	-0.472433000
6	-0.020995000	1.098703000	0.582615000
8	-0.435574000	0.474951000	1.521389000
6	-0.988898000	1.638606000	-0.472398000
8	-0.598599000	2.109009000	-1.503235000
1	-2.053578000	1.517552000	-0.217809000



1ac''

Zero-point correction= 0.186563 (Hartree/Particle)
Thermal correction to Energy= 0.199971
Thermal correction to Enthalpy= 0.200916
Thermal correction to Gibbs Free Energy= 0.144108
Sum of electronic and zero-point Energies= -687.823745
Sum of electronic and thermal Energies= -687.810336
Sum of electronic and thermal Enthalpies= -687.809392
Sum of electronic and thermal Free Energies= -687.866200

6	-6.927814000	24.999967000	-6.353666000
6	-8.124643000	25.245179000	-5.678657000
6	-5.932484000	24.231015000	-5.752762000
6	-8.320203000	24.723353000	-4.414878000
6	-6.132707000	23.711249000	-4.484766000
6	-7.324286000	23.957005000	-3.818514000
1	-8.892305000	25.841543000	-6.153599000
1	-4.997757000	24.037524000	-6.264627000

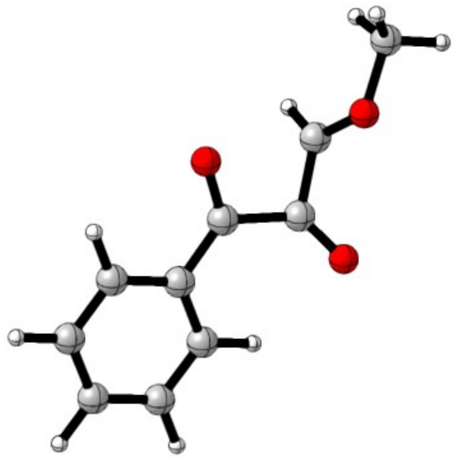


1	-9.247494000	24.910375000	-3.889836000
1	-5.359879000	23.116327000	-4.017028000
1	-7.480215000	23.549748000	-2.827797000
6	-6.733434000	25.557560000	-7.699860000
8	-7.525287000	26.278223000	-8.270936000
6	-5.433214000	25.260879000	-8.459837000
8	-4.521034000	26.041672000	-8.414063000
6	-5.393643000	23.968188000	-9.281678000
8	-4.574340000	24.186554000	-10.373021000
6	-4.139076000	22.994287000	-11.013293000
1	-3.603565000	22.353469000	-10.307283000
1	-4.979760000	22.443897000	-11.439928000
1	-3.465287000	23.296526000	-11.811707000
8	-6.678807000	23.527479000	-9.611204000
1	-4.980851000	23.193583000	-8.621028000
1	-7.101794000	24.173178000	-10.186796000

1ac’’’

Zero-point correction= 0.170607 (Hartree/Particle)
Thermal correction to Energy= 0.182856
Thermal correction to Enthalpy= 0.183800
Thermal correction to Gibbs Free Energy= 0.130220
Sum of electronic and zero-point Energies= -611.770463
Sum of electronic and thermal Energies= -611.758215
Sum of electronic and thermal Enthalpies= -611.757270
Sum of electronic and thermal Free Energies= -611.810850

6	-6.720473000	24.479108000	-6.128878000
6	-8.050915000	24.816504000	-5.856748000
6	-5.812856000	24.291978000	-5.082861000
6	-8.463739000	24.970037000	-4.549728000
6	-6.238810000	24.441631000	-3.775274000
6	-7.557787000	24.781715000	-3.510553000
1	-8.745386000	24.958397000	-6.673834000
1	-4.786196000	24.023261000	-5.285692000
1	-9.489934000	25.234574000	-4.333713000
1	-5.541799000	24.292962000	-2.961894000
1	-7.885845000	24.900360000	-2.485914000
6	-6.313589000	24.304530000	-7.508732000
8	-7.038458000	24.310710000	-8.492345000
6	-4.880679000	24.131639000	-7.998470000
8	-3.832772000	24.427939000	-7.526260000
6	-5.007028000	23.610345000	-9.434930000
8	-4.467017000	24.270574000	-10.327487000
6	-4.458648000	23.814446000	-11.718994000
1	-4.907992000	22.827301000	-11.780012000
1	-5.022875000	24.560482000	-12.269267000
1	-3.413579000	23.813905000	-12.010327000
1	-5.452904000	22.635724000	-9.636851000

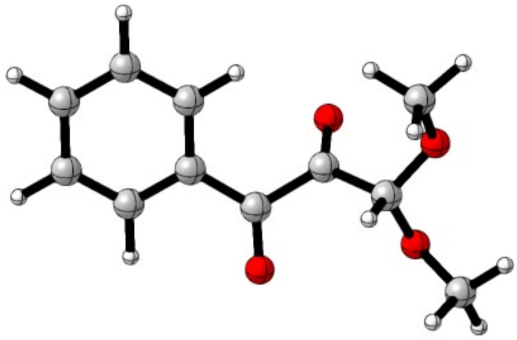


1ad’

Zero-point correction= 0.214721 (Hartree/Particle)
Thermal correction to Energy= 0.229641
Thermal correction to Enthalpy= 0.230585
Thermal correction to Gibbs Free Energy= 0.170055
Sum of electronic and zero-point Energies= -727.089169
Sum of electronic and thermal Energies= -727.074249
Sum of electronic and thermal Enthalpies= -727.073305
Sum of electronic and thermal Free Energies= -727.133835

6	-6.908035000	24.860367000	-6.094353000
6	-7.738389000	25.768073000	-5.434480000
6	-6.429375000	23.738293000	-5.417960000

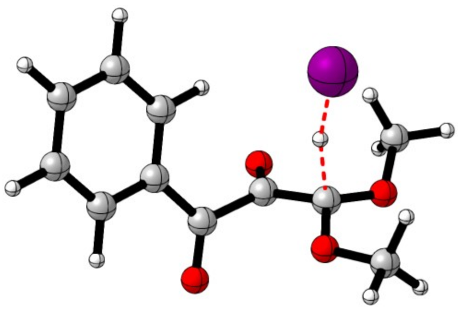
6	-8.075087000	25.561256000	-4.110587000
6	-6.782339000	23.528294000	-4.095105000
6	-7.598129000	24.440032000	-3.441210000
1	-8.106681000	26.636113000	-5.964934000
1	-5.796727000	23.018916000	-5.919327000
1	-8.709630000	26.271268000	-3.596954000
1	-6.417901000	22.653210000	-3.573850000
1	-7.865151000	24.277554000	-2.404851000
6	-6.594121000	25.096528000	-7.515602000
8	-7.201761000	25.871978000	-8.222459000
6	-5.402524000	24.367085000	-8.155832000
8	-4.336539000	24.321830000	-7.604233000
6	-5.617352000	23.793413000	-9.571013000
8	-5.355894000	24.841231000	-10.434900000
6	-5.887040000	24.652848000	-11.742256000
1	-5.458188000	23.768114000	-12.216310000
1	-6.975193000	24.558974000	-11.702071000
1	-5.619674000	25.536233000	-12.317294000
8	-4.727520000	22.752855000	-9.827175000
6	-5.129568000	21.505814000	-9.282707000
1	-5.156867000	21.534167000	-8.189749000
1	-6.114215000	21.215191000	-9.660327000
1	-4.391229000	20.772359000	-9.598232000
1	-6.653711000	23.439215000	-9.668058000



TS3

Zero-point correction= 0.208574 (Hartree/Particle)
Thermal correction to Energy= 0.225268
Thermal correction to Enthalpy= 0.226212
Thermal correction to Gibbs Free Energy= 0.160417
Sum of electronic and zero-point Energies= -738.490967
Sum of electronic and thermal Energies= -738.474272
Sum of electronic and thermal Enthalpies= -738.473328
Sum of electronic and thermal Free Energies= -738.539123
Frequency -891.2244

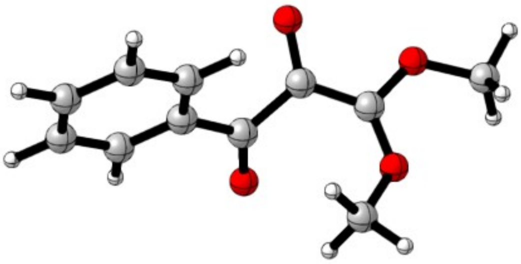
6	-6.496667000	25.051629000	-6.094459000
6	-7.108875000	26.164562000	-5.514199000
6	-6.755659000	23.775847000	-5.596186000
6	-7.973143000	25.998260000	-4.449845000
6	-7.617018000	23.615474000	-4.523634000
6	-8.225797000	24.723572000	-3.953768000
1	-6.906997000	27.150963000	-5.909952000
1	-6.283229000	22.906658000	-6.032861000
1	-8.453816000	26.858745000	-4.003855000
1	-7.814032000	22.625408000	-4.135065000
1	-8.903086000	24.596072000	-3.119062000
6	-5.586755000	25.256611000	-7.232449000
8	-5.183501000	26.338470000	-7.600411000
6	-5.030327000	24.035767000	-7.980297000
8	-4.130489000	23.390308000	-7.490971000
6	-5.628571000	23.732696000	-9.308809000
8	-6.290333000	24.763728000	-9.832753000
6	-6.885873000	24.589769000	-11.123779000
1	-6.118997000	24.575286000	-11.896224000
1	-7.465849000	23.666759000	-11.150116000
1	-7.537888000	25.447310000	-11.258439000
8	-5.017975000	22.955999000	-10.199258000
6	-4.460725000	21.693117000	-9.806160000
1	-5.054990000	21.237077000	-9.016393000
1	-4.490768000	21.072839000	-10.697901000
1	-3.433658000	21.828918000	-9.476287000



1	-6.737172000	22.866453000	-8.765684000
53	-8.202221000	21.811067000	-8.572051000

1ad''

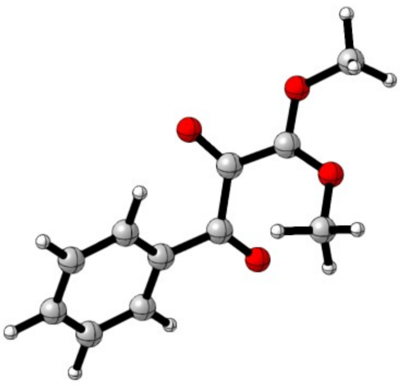
Zero-point correction= 0.203353 (Hartree/Particle)
Thermal correction to Energy= 0.217545
Thermal correction to Enthalpy= 0.218489
Thermal correction to Gibbs Free Energy= 0.160528
Sum of electronic and zero-point Energies= -726.475239
Sum of electronic and thermal Energies= -726.461046
Sum of electronic and thermal Enthalpies= -726.460102
Sum of electronic and thermal Free Energies= -726.518064



6	-7.141724000	24.651985000	-6.046590000
6	-8.254334000	24.984140000	-5.275324000
6	-5.974326000	24.215533000	-5.422481000
6	-8.196926000	24.890752000	-3.896116000
6	-5.925334000	24.107517000	-4.042340000
6	-7.032883000	24.449826000	-3.279108000
1	-9.158720000	25.322476000	-5.763757000
1	-5.108951000	23.946035000	-6.012755000
1	-9.059003000	25.159850000	-3.299956000
1	-5.021259000	23.757779000	-3.561550000
1	-6.989668000	24.372483000	-2.200210000
6	-7.250165000	24.707359000	-7.524884000
8	-8.329155000	24.668465000	-8.092196000
6	-5.994807000	24.902291000	-8.328556000
8	-5.142841000	25.742410000	-7.940117000
6	-5.845028000	24.256699000	-9.594155000
8	-4.944025000	24.765398000	-10.392192000
6	-4.599728000	24.072273000	-11.602836000
1	-4.274564000	23.058372000	-11.377549000
1	-5.449287000	24.057448000	-12.282632000
1	-3.784150000	24.645638000	-12.031594000
8	-6.468425000	23.207919000	-10.050834000
6	-7.099311000	22.234307000	-9.203797000
1	-6.603250000	22.192864000	-8.235006000
1	-8.152437000	22.473346000	-9.089999000
1	-6.979494000	21.287329000	-9.722191000

1ad'''

Zero-point correction= 0.204258 (Hartree/Particle)
Thermal correction to Energy= 0.218930
Thermal correction to Enthalpy= 0.219874
Thermal correction to Gibbs Free Energy= 0.161164
Sum of electronic and zero-point Energies= -726.308382
Sum of electronic and thermal Energies= -726.293710
Sum of electronic and thermal Enthalpies= -726.292766
Sum of electronic and thermal Free Energies= -726.351475



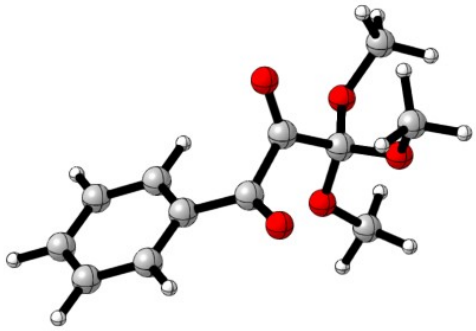
6	-6.957036000	24.671979000	-5.992982000
6	-8.019303000	25.400466000	-5.449700000
6	-6.118191000	23.927766000	-5.160637000
6	-8.230173000	25.393208000	-4.085783000
6	-6.345818000	23.915861000	-3.795480000
6	-7.393953000	24.650066000	-3.259601000
1	-8.663523000	25.973285000	-6.103138000
1	-5.306782000	23.343910000	-5.570666000
1	-9.045551000	25.963472000	-3.661630000
1	-5.704212000	23.332677000	-3.148984000
1	-7.563798000	24.643091000	-2.190671000
6	-6.786270000	24.678910000	-7.442249000
8	-7.612187000	25.033017000	-8.256987000
6	-5.461171000	24.271015000	-8.104074000

8	-4.361654000	24.306804000	-7.656387000
6	-5.659189000	23.950703000	-9.606401000
8	-5.212849000	24.823804000	-10.395281000
6	-5.293524000	24.610702000	-11.838955000
1	-4.716961000	23.724538000	-12.089668000
1	-6.338744000	24.500866000	-12.114684000
1	-4.858840000	25.505319000	-12.267108000
8	-6.152291000	22.877951000	-10.042969000
6	-6.658403000	21.840215000	-9.144596000
1	-5.948999000	21.677929000	-8.337179000
1	-7.633779000	22.154831000	-8.784585000
1	-6.736592000	20.958110000	-9.768329000

2a

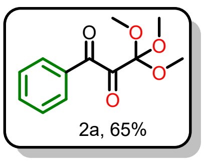
Zero-point correction= 0.246658 (Hartree/Particle)
Thermal correction to Energy= 0.264218
Thermal correction to Enthalpy= 0.265162
Thermal correction to Gibbs Free Energy= 0.198747
Sum of electronic and zero-point Energies= -841.596279
Sum of electronic and thermal Energies= -841.578719
Sum of electronic and thermal Enthalpies= -841.577775
Sum of electronic and thermal Free Energies= -841.644190

6	-7.217374000	24.056921000	-5.872127000
6	-8.293915000	23.965856000	-4.989185000
6	-6.141037000	24.892538000	-5.577756000
6	-8.288000000	24.701315000	-3.819604000
6	-6.143369000	25.632952000	-4.407614000
6	-7.213299000	25.534802000	-3.529749000
1	-9.125549000	23.315816000	-5.226681000
1	-5.304895000	24.977734000	-6.260413000
1	-9.119244000	24.628981000	-3.130898000
1	-5.310880000	26.285101000	-4.180267000
1	-7.212059000	26.111503000	-2.613720000
6	-7.246091000	23.281993000	-7.122394000
8	-8.193946000	22.635420000	-7.514277000
6	-5.973777000	23.214321000	-7.978256000
8	-5.035451000	22.557426000	-7.622790000
6	-6.000308000	23.992100000	-9.327420000
8	-6.782389000	25.089731000	-9.037728000
6	-7.053033000	25.981360000	-10.117165000
1	-6.131089000	26.306810000	-10.599090000
1	-7.712463000	25.515839000	-10.849738000
1	-7.552429000	26.839316000	-9.674428000
8	-4.723765000	24.454508000	-9.629711000
6	-3.862001000	23.656156000	-10.432086000
1	-3.565249000	22.741134000	-9.918236000
1	-4.321154000	23.415871000	-11.390880000
1	-2.978720000	24.266454000	-10.606809000
8	-6.571426000	23.269761000	-10.361408000
6	-6.488514000	21.844707000	-10.365493000
1	-5.462232000	21.490831000	-10.273960000
1	-7.100259000	21.415633000	-9.569816000
1	-6.887781000	21.532360000	-11.327144000



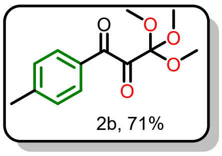
10. NMR Data of synthesised 2,3-dioxo orthoester derivatives:

3,3,3-trimethoxy-1-phenylpropane-1,2-dione (2a)



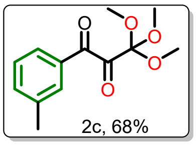
Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2a** (94 mg, 65% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 7.90-7.88 (m,2H), 7.69-7.64 (m, 1H), 7.53(t, *J* = 7.7Hz, 2H), 3.46 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 197.9, 194.3, 134.7, 132.5, 129.5, 128.9, 110.2, 51.0; **IR** (neat, cm^{-1}) = 1673, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{12}H_{14}NaO_5$: 261.0739; Found: 261.0741.

3,3,3-trimethoxy-1-(p-tolyl)propane-1,2-dione (2b)


2b, 71%

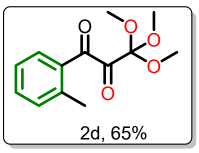
Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2b** (104 mg,71% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** =7.78 (d, *J* = 7.9 Hz, 2H), 7.32 (d, *J* =7.9 Hz, 2H), 3.45 (s, 9H), 2.45 (s, 3H);**^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** =198.0, 193.9, 145.9, 130.1, 129.6, 110.2, 50.9, 21.9; **IR** (neat, cm^{-1}) = 1673, 173; **HRMS** (ESI)m/z: (M + Na) calcd for $C_{13}H_{16}NaO_5$:275.0895; Found: 275.0898.

3,3,3-trimethoxy-1-(m-tolyl)propane-1,2-dione (2c)


2c, 68%

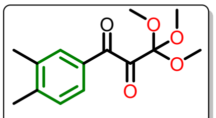
Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2c** (98 mg, 68% yield) as a pale-yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 7.71 (s, 1H), 7.66 (dd, *J* =7.7, 1.6 Hz, 1H), 7.47 (d, *J* = 7.6 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 1H), 3.46 (s, 9H), 2.43 (s, 3H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 198.0, 194.4, 138.8, 135.6, 132.4, 129.7, 128.8, 127, 110.2, 50.9, 21.4; **IR** (neat, cm^{-1}) = 1673, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{13}H_{16}NaO_5$: 275.0895; Found: 275.0900.

3,3,3-trimethoxy-1-(o-tolyl)propane-1,2-dione (2d)


2d, 65%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2d** (95 mg, 65% yield) as a deep yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 7.62 (d, *J* = 7.8 Hz, 1H), 7.50 (t, *J* =7.5 Hz, 1H), 7.33 (d, *J* = 7 Hz, 2H), 3.45 (s, 9H), 2.66 (s, 3H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 197.5,196.5, 141.2, 133.5, 132.5, 132.4, 130.9, 125.8, 110.2, 50.9, 21.6; **IR** (neat, cm^{-1}) = 1673, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{13}H_{16}NaO_5$: 275.0895; Found: 275.0898.

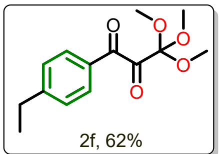
1-(3,4-dimethylphenyl)-3,3,3-trimethoxypropane-1,2-dione (2e)



2e, 60%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2e** (85 mg,60% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 7.67 (d, *J* = 1.9 Hz, 1H), 7.60 (dd, *J* =7.9, 2.0 Hz, 1H), 7.29-7.26 (m, 1H), 3.45 (s, 9H), 2.36 (s, 3H), 2.34 (s, 3H);**^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 198, 194.1, 144.8, 137.5, 130.4, 130.3, 130.1, 127.5, 110.2, 50.9, 20.3, 19.8; **IR** (neat, cm^{-1}) = 1673, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for$C_{14}H_{18}NaO_5$: 289.1052; Found: 289.1055.

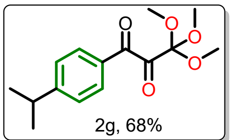
1-(4-ethylphenyl)-3,3,3-trimethoxypropane-1,2-dione (2f)



2f, 62%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2f** (88 mg,62% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 7.82-7.80 (m, 2H), 7.35 (d, *J* =8.2 Hz, 2H),3.46 (s, 9H), 2.75 (q, *J* = 7.6 Hz, 2H), 1.28(t, *J* = 7.6 Hz, 3H); **^{13}C{H}NMR** (100 MHz, $CDCl_3$) ***δ*** = 198, 193.9, 152.0, 130.2, 129.7, 128.5, 110.2, 50.9, 29.2, 15.1; **IR** (neat, cm^{-1}) = 1673, 1733**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{14}H_{18}NaO_5$: 289.1052; Found: 289.1057.

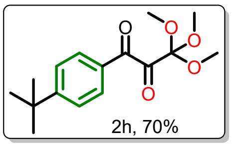
1-(4-isopropylphenyl)-3,3,3-trimethoxypropane-1,2-dione (2g)



2g, 68%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2g** (98 mg,68% yield) as a pale-yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 7.82 (d, *J* = 8.1 Hz, 2H), 7.38 (d, *J* =8.1 Hz, 2H), 3.46 (s, 9H), 3.00 (hept, *J* = 6.8 Hz, 1H), 1.29(d, *J* = 6.9 Hz, 6H;**^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 198, 193.9, 156.6, 130.4, 129.8, 127.1, 110.2, 51, 34.5, 23.6; **IR** (neat, cm^{-1}) = 1673, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{15}H_{20}NaO_5$: 303.1208; Found: 303.1211.

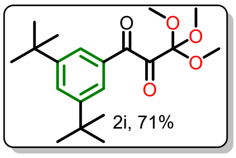
1-(4-(tert-butyl)phenyl)-3,3,3-trimethoxypropane-1,2-dione (2h)



2h, 70%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2h** (95 mg,70% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** = 7.83 (d, *J* = 8.1 Hz, 2H), 7.54 (d, *J* = 8.2Hz, 2H), 3.46 (s, 9H), 1.37 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 197.9, 193.8, 158.7, 129.9, 129.5, 125.9, 110.2, 50.9, 35.4, 31.0; **IR** (neat, cm^{-1}) = 1673, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{16}H_{22}NaO_5$: 317.1365; Found: 317.1369.

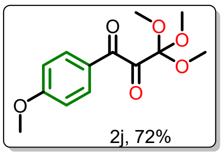
1-(3,5-di-tert-butylphenyl)-3,3,3-trimethoxypropane-1,2-dione (2i)



2i, 71%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2i** (102 mg,71% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 7.76 (d, *J* = 1.9 Hz, 2H), 7.74 (t, *J* = 1.9 Hz, 1H), 3.46 (s, 9H), 1.36 (s, 18H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** =198.5, 195.1, 151.6, 132.1, 129.2, 123.9, 110.2, 50.9, 35.0, 31.3; **IR** (neat, cm^{-1}) = 1673, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{20}H_{30}NaO_5$: 373.1991; Found: 373.1994.

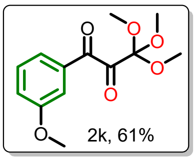
3,3,3-trimethoxy-1-(4-methoxyphenyl)propane-1,2-dione (2j)



2j, 72%

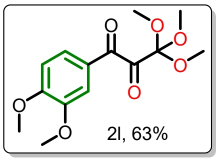
Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 17:3 as the eluent) to give **2j** (108 mg,72% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** = 7.86-7.84 (m, 2H), 7.00-6.98 (m, 2H), 3.90 (s, 3H), 3.44(s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 198.1, 192.7, 164.8, 131.9, 125.5, 114.3, 110.2, 55.7, 51.0; **IR** (neat, cm^{-1}) = 1670, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{13}H_{16}NaO_6$: 291.0845; Found: 291.0848.

3,3,3-trimethoxy-1-(3-methoxyphenyl)propane-1,2-dione (2k)



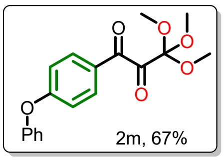
Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 17:3 as the eluent) to give **2k** (87 mg, 61% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** = 7.45-7.42 (m, 3H), 7.21 (dq, *J* = 6.4, 3.5 Hz, 1H), 3.87(s, 3H), 3.45 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 197.8, 194.2, 159.9, 133.7, 129.9, 122.7, 121.7, 112.5, 110.2, 55.5, 51.0; **IR** (neat, cm^{-1}) = 1673, 1731**;** **HRMS** (ESI) m/z: (M + Na) calcd for $C_{13}H_{16}NaO_6$: 321.1103; Found: 321.1105.

1-(3,4-dimethoxyphenyl)-3,3,3-trimethoxypropane-1,2-dione (2l)



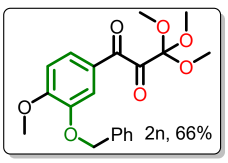
Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 4:1 as the eluent) to give **2l** (88 mg,63% yield) as a pale-yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** = 7.49 (d, *J* = 2.0 Hz, 1H), 7.42 (dd, *J* = 8.4, 2.0Hz, 1H), 6.94 (d, *J* = 8.4 Hz, 1H), 3.97 (s, 3H), 3.95 (s, 3H), 3.45 (s, 9H);**^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 198.0, 192.9, 154.7, 149.4, 125.7, 125.6, 110.3, 110.2, 110.0, 56.2, 56.0, 51.0; **IR** (neat, cm^{-1}) = 1670, 1731**;** **HRMS** (ESI) m/z: (M + Na)calcd for $C_{14}H_{18}NaO_7$: 321.0950; Found: 321.0952.

3,3,3-trimethoxy-1-(4-phenoxyphenyl)propane-1,2-dione (2m)



Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 17:3 as the eluent) to give **2m** (89 mg,67% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** = 7.87-7.84 (m, 2H), 7.45-7.40 (m, 2H),7.27-7.22 (m, 1H), 7.12-7.09 (m, 2H), 7.05-7.02 (m, 2H), 3.45 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 197.9, 192.8, 163.6, 154.8, 131.9,130.2, 126.9, 125.1, 120.6, 117.3, 110.2, 51.0; **IR** (neat, cm^{-1}) = 1671, 1731**;** **HRMS** (ESI) m/z: (M + Na) calcd for $C_{18}H_{18}NaO_6$: 353.1001; Found: 353.1006.

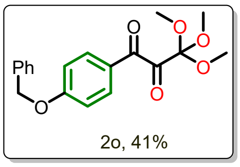
1-(3-(benzyloxy)-4-methoxyphenyl)-3,3,3-trimethoxypropane-1,2-dione (2n)



Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 4:1 as the eluent) to give

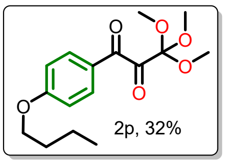
2n (96 mg,66% yield) as a pale- yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** = 7.53 (d, *J* = 2.0 Hz, 1H), 7.46 (td, *J* = 7.8, 1.9 Hz, 3H), 7.41-7.37 (m, 2H), 7.35-7.31 (m, 1H), 6.96 (d, *J* = 8.4 Hz,1H),5.19 (s, 2H), 3.96 (s, 3H), 3.42 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 197.9, 192.7, 155.3, 148.5, 136.2, 128.7, 128.2, 127.6, 127.5, 125.8, 125.5, 112.4, 110.8, 110.2, 70.9, 56.2, 50.9; **IR** (neat, cm^{-1}) = 1669, 1730**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{20}H_{22}NaO_7$: 397.1263; Found: 397.1264.

1-(4-(benzyloxy)phenyl)-3,3,3-trimethoxypropane-1,2-dione (2o)



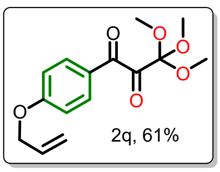
Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 17:3 as the eluent) to give **2o** (56 mg,41% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** = 7.86 (d, *J* = 8.8 Hz, 2H), 7.44-7.38 (m, 5H), 7.07 (d, *J* = 8.9 Hz, 2H), 5.17 (s, 2H), 3.46 (s, 9H); **^{13}C{H} NMR**(100 MHz, $CDCl_3$) ***δ*** = 198.0, 192.7, 163.9, 135.9, 131.9, 128.8, 128.4, 127.5, 125.7, 115.1, 110.2, 70.3, 51.0; **IR** (neat, cm^{-1}) = 1669, 1730**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{19}H_{20}NaO_6$: 367.1158; Found: 367.1160.

1-(4-butoxyphenyl)-3,3,3-trimethoxypropane-1,2-dione (2p)



Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 17:3 as the eluent) to give **2p** (42 mg, 32% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** = 7.84 (d, *J* = 8.9 Hz, 2H), 6.98 (d, *J* = 8.9 Hz, 2H), 4.06 (t, *J* = 6.5 Hz, 2H), 3.45 (s, 9H), 1.85-1.47 (m, 2H), 1.51 (qd, *J* = 8.6, 6.5 Hz, 2H), 1.00 (t, *J* = 7.4 Hz, 3H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** =198.1, 192.7, 164.5, 131.9, 125.3, 114.7, 110.2, 68.2, 50.9, 31.0, 19.2, 13.8; **IR** (neat, cm^{-1}) = 1671, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{16}H_{22}NaO_6$: 333.1314; Found: 333.1315.

1-(4-(allyloxy)phenyl)-3,3,3-trimethoxypropane-1,2-dione (2q)

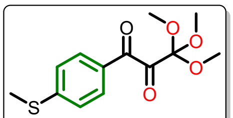


Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 17:3 as the eluent) to give **2q** (86 mg, 61% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** =

7.82 (d, J = 8.9 Hz, 2H), 6.98 (d, J = 8.9 Hz, 2H), 6.03 (ddt, J = 17.3, 10.5, 5.3 Hz, 1H), 5.41 (dq, J = 17.3, 1.6 Hz, 1H), 5.31 (dq, J = 10.5, 1.4 Hz, 1H), 4.61 (dt, J = 5.4, 1.5 Hz, 2H),3.43 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) δ = 198.0, 192.7, 163.8, 132.2, 131.9, 125.6, 118.4,

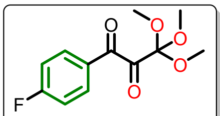
114.9, 110.2, 69.0, 50.9; **IR** (neat, cm^{-1}) = 1669, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{15}H_{18}NaO_6$: 317.1001; Found: 317.1003.

3,3,3-trimethoxy-1-(4-(methylthio)phenyl)propane-1,2-dione (2r)


2r, 35%

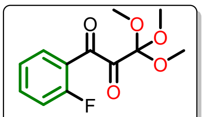
Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 22:3 as the eluent) to give **2r** (43 mg, 35% yield) as a dark-yellow semisolid; **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.78 (d, J = 8.6 Hz, 2H), 7.31 (d, J =8.6, 2H), 3.45 (s, 9H), 2.54 (s, 3H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) δ = 197.9, 193.2, 148.6, 129.8, 128.7, 124.9, 110.2, 51.0, 14.6; **IR** (neat, cm^{-1}) = 1670, 1730**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{13}H_{16}NaO_6S$: 307.0616; Found: 307.0619.

1-(4-fluorophenyl)-3,3,3-trimethoxypropane-1,2-dione (2s)


2s, 63%

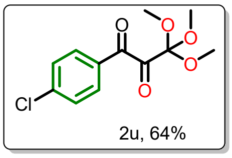
Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2s** (94 mg, 63% yield) as a pale-yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.95-7.90 (m, 2H), 7.20 (t, J = 8.6Hz, 2H), 3.46 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) δ =197.7, 192.7, 166.7 (d, J = 257.8 Hz), 132.3 (d, J = 9.8 Hz), 129.1, 116.3 (d, J = 22.3 Hz), 110.1, 51.0; **^{19}F NMR** (376 MHz, $CDCl_3$) δ = -101.7; **IR** (neat, cm^{-1}) = 1673, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{12}H_{13}FNaO_5$: 279.0645; Found: 279.0649.

1-(2-fluorophenyl)-3,3,3-trimethoxypropane-1,2-dione (2t)


2t, 57%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2t** (69 mg, 57% yield) as a pale-yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.98 (ddd, J = 7.9, 7.0, 1.9 Hz, 1H), 7.69 7.63 (m, 1H), 7.33 (td, J = 7.6, 1.1, 1H), 7.17 (ddd, J = 10.7, 8.4, 1.0 Hz, 1H), 3.45(s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** =194.4, 191.3, 162.9 (d, *J* = 258.0 Hz), 136.7 (d, *J* = 9.1 Hz), 130.4 (d, *J* = 2.1 Hz), 124.8 (d, *J* = 3.4 Hz), 121.4 (d, *J* = 11.5 Hz), 116.6 (d, *J* = 21.6 Hz), 110.5, 50.9; **^{19}F NMR** (376 MHz, $CDCl_3$) ***δ*** = -107.6; **IR** (neat, cm^{-1}) = 1673, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{12}H_{13}FNaO_5$: 279.0645; Found: 279.0649.

1-(4-chlorophenyl)-3,3,3-trimethoxypropane-1,2-dione (2u)

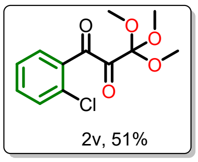


2u, 64%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2u** (92 mg, 64% yield) as a pale-yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** = 7.83 (d, *J* = 8.6 Hz, 2H), 7.51 (d, *J* = 8.6 Hz, 2H), 3.45 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 197.6, 193.1, 141.4, 130.9, 130.6, 129.4, 110.1, 51.0; **IR** (neat, cm^{-1}) = 1673, 1731**;**

HRMS (ESI) m/z: (M + Na) calcd for $C_{12}H_{13}ClNaO_5$: 295.0349; Found: 295.0354.

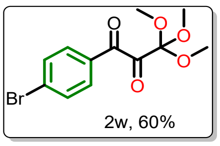
1-(2-chlorophenyl)-3,3,3-trimethoxypropane-1,2-dione (2v)



2v, 51%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2a** (53 mg, 51% yield) as a dark-yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ* =** 7.77 (dd, *J* = 7.8,1.6 Hz, 1H), 7.56-7.48 (m, 2H), 7.41 (td, *J* = 7.4, 1.5, 1H), 3.44(s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 194.7, 193.5, 138.4, 134.2, 132.1, 131.2, 128.8, 126.9,115.6, 50.9; **IR** (neat, cm^{-1}) = 1673, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{12}H_{13}ClNaO_5$: 295.0349; Found: 295.0354

1-(4-bromophenyl)-3,3,3-trimethoxypropane-1,2-dione (2w)

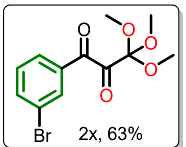


2w, 60%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2w** (74 mg, 60% yield) as a pale-yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ* =**

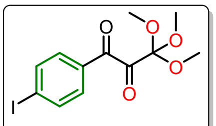
7.75 (d, J = 8.4 Hz, 2H), 7.68 (d, J = 8.5 Hz, 2H), 3.45 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 197.5, 193.4, 132.3, 131.2, 130.8, 130.3, 110.1, 51.0; **IR** (neat, cm^{-1}) = 1673, 1731**;** **HRMS** (ESI) m/z: (M + Na) calcd for $C_{12}H_{13}BrNaO_5$: 338.9844; Found: 338.9845.

1-(3-bromophenyl)-3,3,3-trimethoxypropane-1,2-dione (2x)


2x, 63%

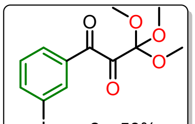
Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2x** (80 mg, 63% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 8.03 (t, J = 1.8 Hz, 1H), 7.80-7.78 (m, 2H), 7.41 (t, J = 7.9 Hz, 1H), 3.45 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** =197.2, 192.9, 137.5, 134.2, 132.0, 130.5, 128.2, 123.2, 110.1, 51.1; **IR** (neat, cm^{-1}) = 1673, 1731**;** **HRMS** (ESI) m/z: (M + Na) calcd for $C_{12}H_{13}BrNaO_5$: 338.9844; Found: 338.9848.

1-(4-iodophenyl)-3,3,3-trimethoxypropane-1,2-dione (2y)


2y, 58%

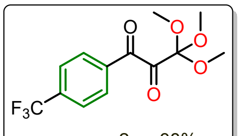
Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2y** (82 mg, 58% yield) as a white amorphous solid (m.p.- 90°-94°C); **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 7.91 (d, J = 8.5 Hz, 2H), 7.59 (d, J = 8.5 Hz, 2H), 3.44 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 197.5, 193.7, 138.3, 131.8, 130.6, 110.1, 103.38, 51.0; **IR** (neat, cm^{-1}) = 1673, 1731**;** **HRMS** (ESI) m/z: (M + Na) calcd for $C_{12}H_{13}INaO_5$: 386.9705; Found: 386.9709.

1-(3-iodophenyl)-3,3,3-trimethoxypropane-1,2-dione (2z)


2z, 58%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2z** (85 mg, 58% yield) as a pale-yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** = 8.22 (t, J = 1.7 Hz, 1H), 7.98 (dt, J = 7.9, 1.4 Hz, 1H), 7.81 (dt, J = 7.8, 1.3 Hz, 1H), 7.27 (t, J = 7.81 Hz, 1H), 3.45 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** =197.2, 192.9, 143.3, 137.9, 134.2, 130.5, 128.7, 110.1, 94.5, 51.0; **IR** (neat, cm^{-1}) = 1673, 1731**;** **HRMS** (ESI) m/z: (M + Na) calcd for $C_{12}H_{13}INaO_5$: 386.9705; Found: 386.9708.

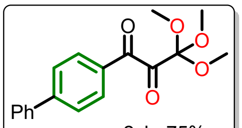
3,3,3-trimethoxy-1-(4-(trifluoromethyl)phenyl)propane-1,2-dione (2za)



2za, 38%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2za** (50 mg, 38% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 8.01 (d, J = 8.0 Hz, 2H), 7.80 (d, J = 8.0 Hz, 2H), 3.46 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 197.4, 193.4, 135.7 (d, J = 33 Hz), 135.1, 129.7, 125.9(q, J = 3.8 Hz), 124.7, 122.0, 110.1, 51.1; **^{19}F NMR** (376 MHz, $CDCl_3$) ***δ*** = -63.4; **IR** (neat, cm^{-1}) = 1673, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{13}H_{13}F_3NaO_5$: 329.0613; Found: 329.0616.

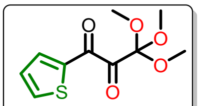
1-([1,1'-biphenyl]-4-yl)-3,3,3-trimethoxypropane-1,2-dione (2zb)



2zb, 75%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2zb** (106 mg, 75% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 7.98-7.96 (m, 2H), 7.75 (d, J = 8.3 Hz, 2H), 7.65 (d, J = 7.0 Hz, 2H), 7.50 (t, J = 7.3 Hz, 2H), 7.46-7.44 (m, 1H), 3.45 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 197.9, 193.9, 147.4, 139.6, 131.2, 130.1, 129.1, 128.6, 127.6, 127.4, 110.2, 51.0; **IR** (neat, cm^{-1}) = 1670, 1730**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{18}H_{18}NaO_5$: 337.1052; Found: 337.1055.

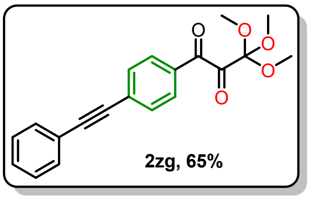
3,3,3-trimethoxy-1-(thiophen-2-yl)propane-1,2-dione (2zc)



2zc, 51%

Following the synthetic electrolysis procedure, the crude product was purified by silica gel chromatography (PE: EA = 9:1 as the eluent) to give **2zc** (69 mg, 51% yield) as a yellow oil; **^{1}H NMR** (400 MHz, $CDCl_3$) ***δ*** **=** 7.83 (dd, J = 4.9, 1.2 Hz, 1H), 7.70 (dd, J = 3.9, 1.2 Hz, 1H), 7.20 (dd, J = 4.9, 3.8 Hz, 1H), 3.44 (s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** = 195.3, 185.4, 193.3, 136.4, 136.0, 128.7, 110.4, 51.0; **IR** (neat, cm^{-1}) = 1671, 1731**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{10}H_{12}SNaO_5$: 267.0303; Found: 267.0306.

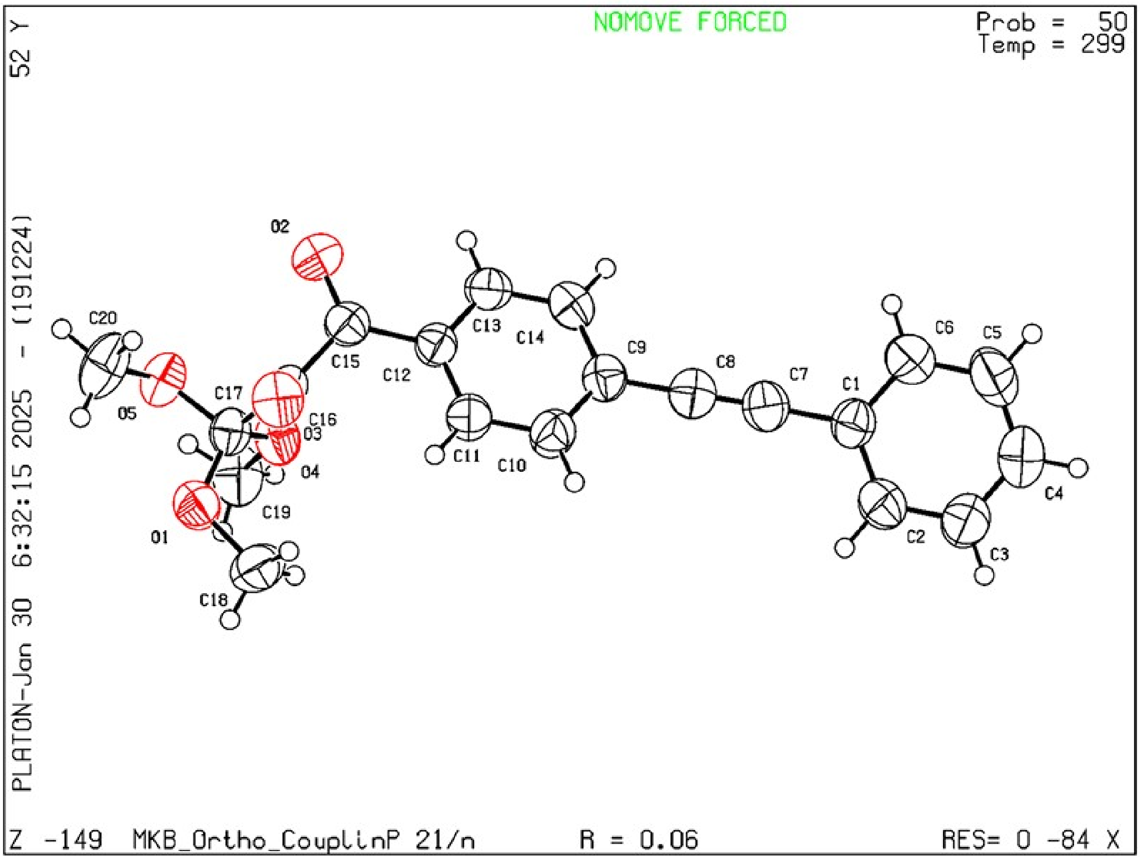
3,3,3-trimethoxy-1-(4-(phenylethynyl)phenyl)propane-1,2-dione (2zg)


2zg, 65%

^{1}H NMR (400 MHz, $CDCl_3$) ***δ* =** 7.88 (d, *J* = 8.3 Hz, 2H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.58 (dd, *J* = 6.6, 3.0 Hz, 2H), 7.41-7.39 (m, 3H), 3.47(s, 9H); **^{13}C{H} NMR** (100 MHz, $CDCl_3$) ***δ*** =197.7, 193.5, 131.9, 131.8, 131.5, 129.9, 129.4, 129.1, 128.5, 122.4, 110.2, 93.9, 88.5, 51.0**; IR** (neat, cm^{-1}) = 1673, 1731, 2214**; HRMS** (ESI) m/z: (M + Na) calcd for $C_{20}H_{18}NaO_5$: 338.1154; Found: 338.1159.

11. X-ray data for 2zg

Datablock MKB_Ortho_Coupling_0m_a - ellipsoid plot



checkCIF/PLATON report

Structure factors have been supplied for datablock(s) MKB_Ortho_Coupling_0m_a

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.

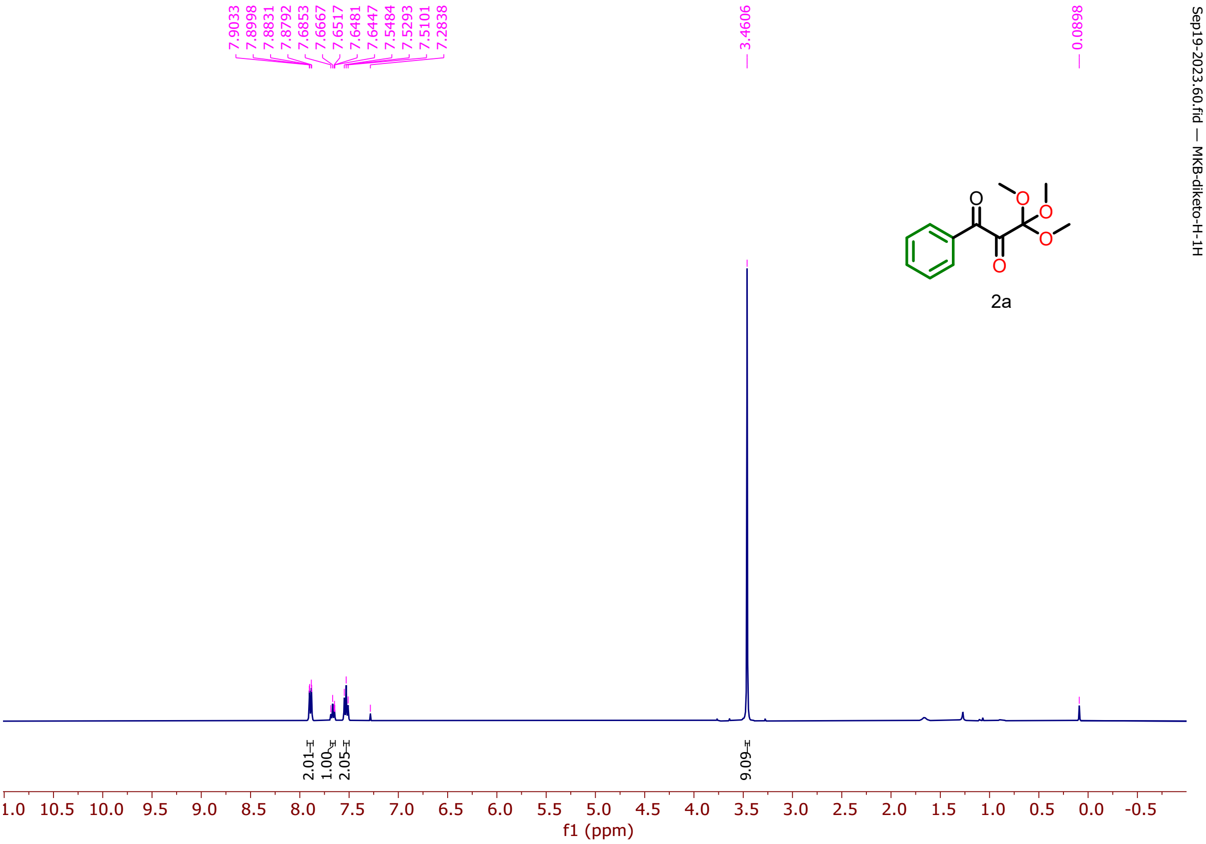
No syntax errors found. CIF dictionary Interpreting this report

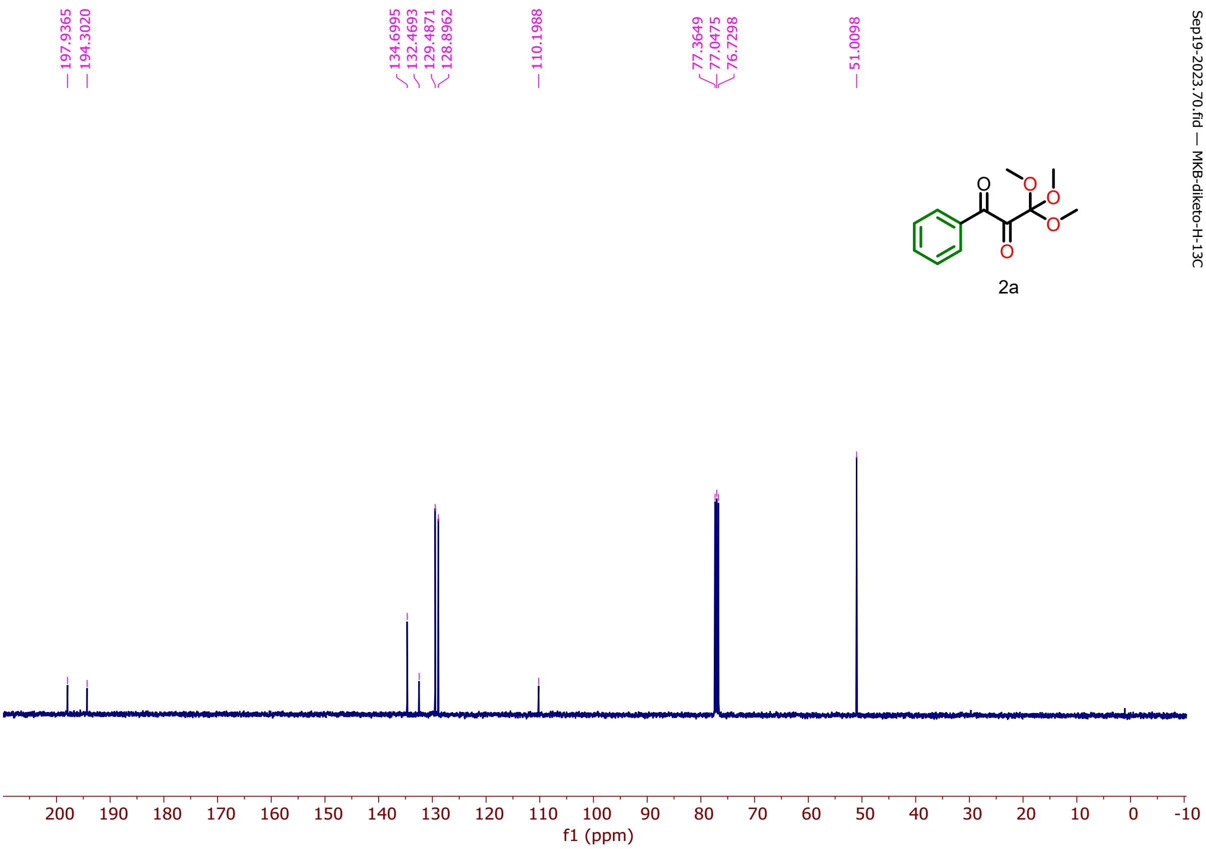
Datablock: MKB_Ortho_Coupling_0m_a

```
Bond precision:    C-C = 0.0056 A           Wavelength=0.71073

Cell:              a=13.1701(8)     b=6.1476(3)          c=22.2657(13)
                   alpha=90         beta=104.251(2)      gamma=90
Temperature:       299 K

                  Calculated                    Reported
Volume            1747.26(17)                   1747.25(17)
Space group       P 21/n                        P 21/n
Hall group        -P 2yn                        -P 2yn
Moiety formula    C20 H18 O5                    C20 H18 O5
Sum formula       C20 H18 O5                    C1.57 H1.41 O0.39
Mr                338.34                        26.54
Dx,g cm-3         1.286                         1.286
Z                 4                             51
Mu (mm-1)         0.092                         0.092
F000              712.0                         712.0
F000'             712.40
h,k,lmax          15,7,26                       15,7,26
Nref              3113                          3099
Tmin,Tmax
Tmin'

Correction method= Not given

Data completeness= 0.996            Theta(max)= 25.068

R(reflections)= 0.0619( 1779)                          wR2(reflections)=
                                                       0.1863( 3099)
S = 1.142                  Npar= 230
```

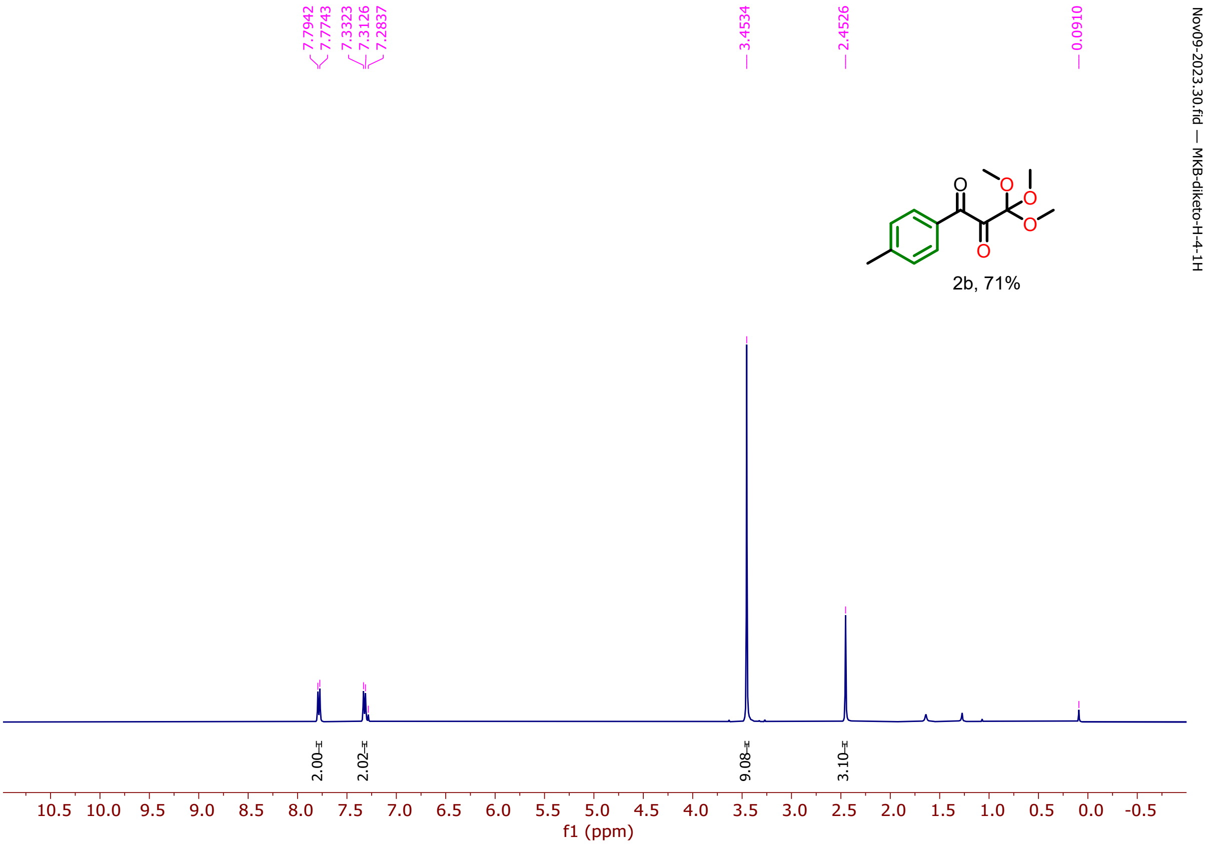
12. NMR DATA

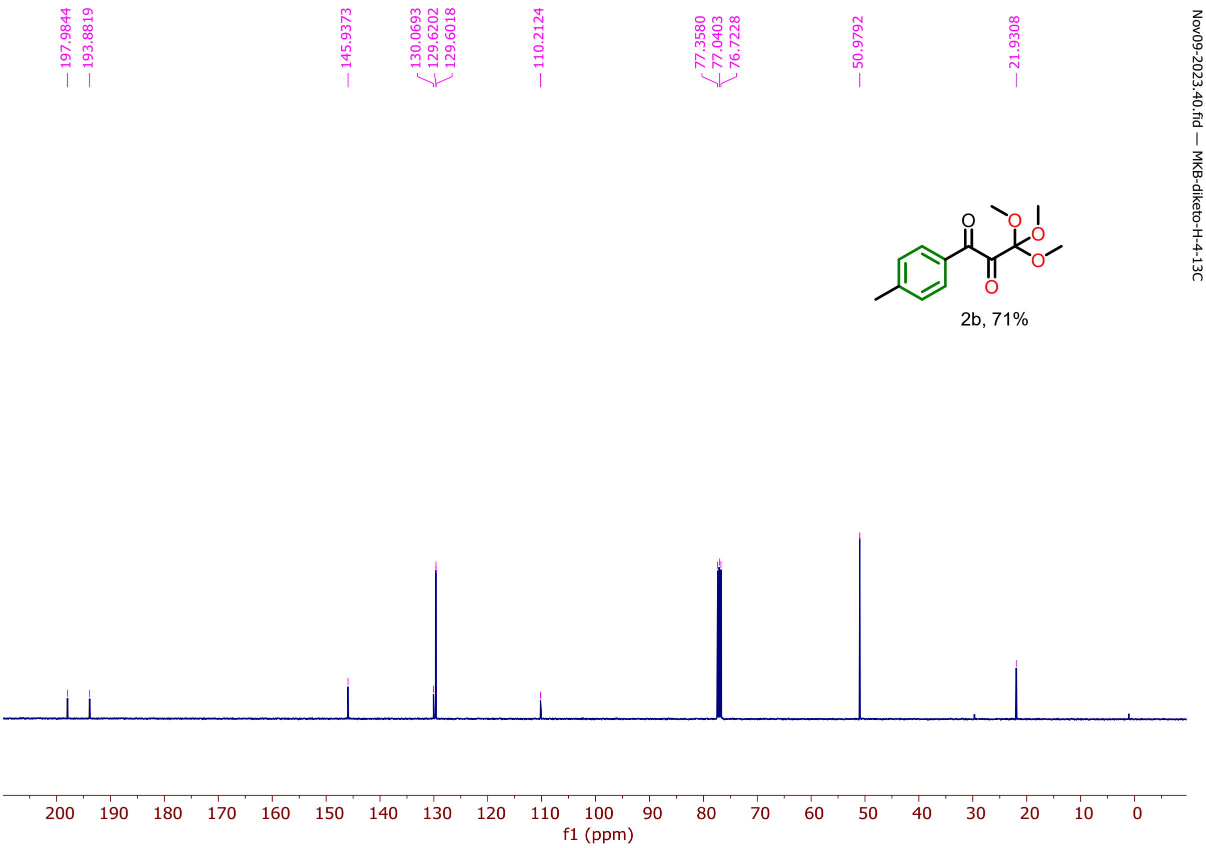
^{1}H NMR & ^{13}C NMR of 2a



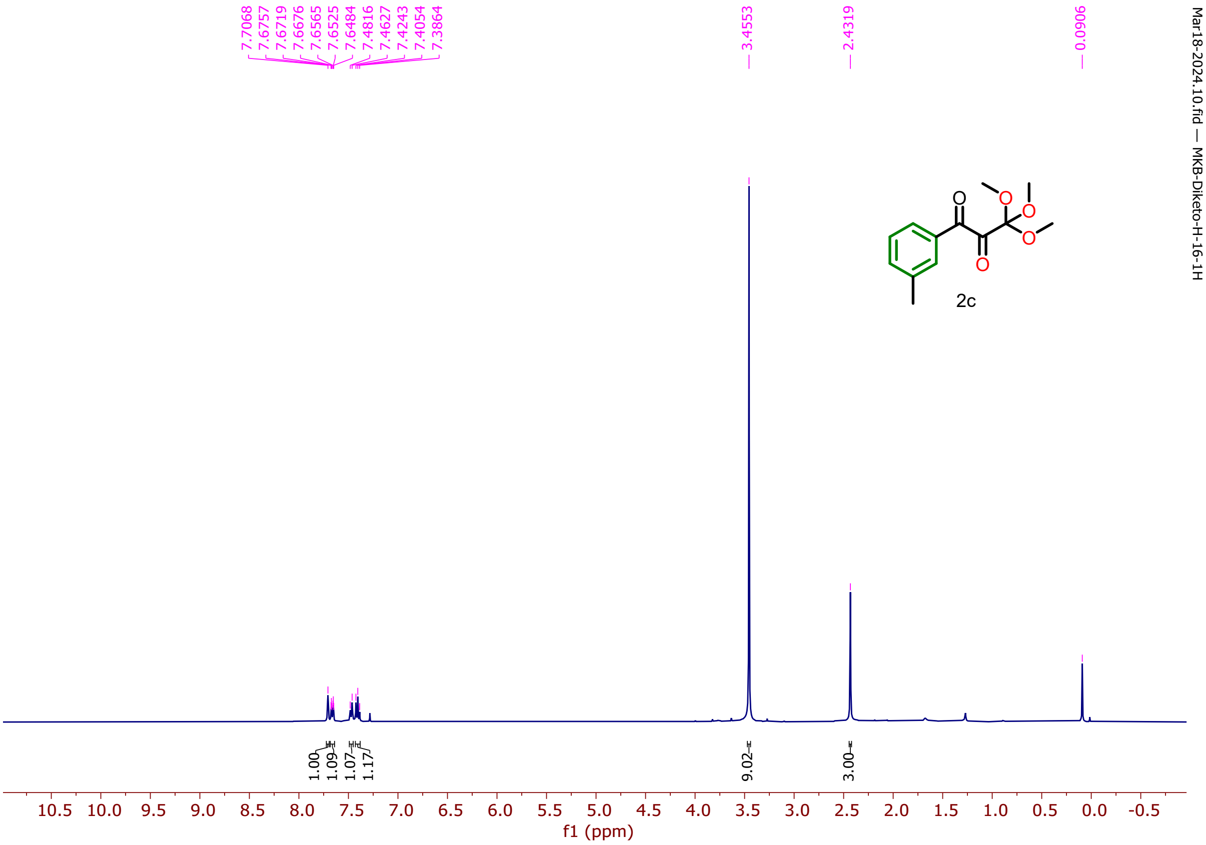


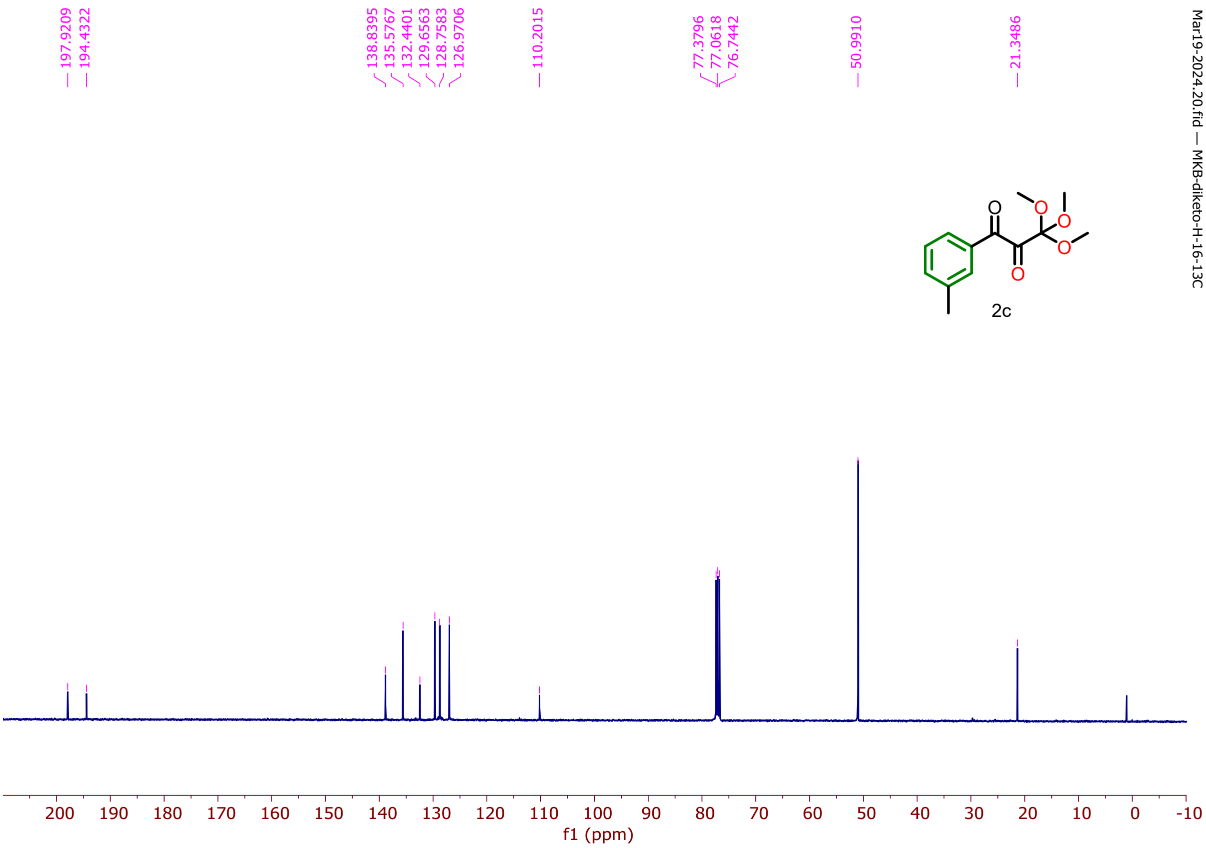
[1]H NMR & [13]C NMR of 2b



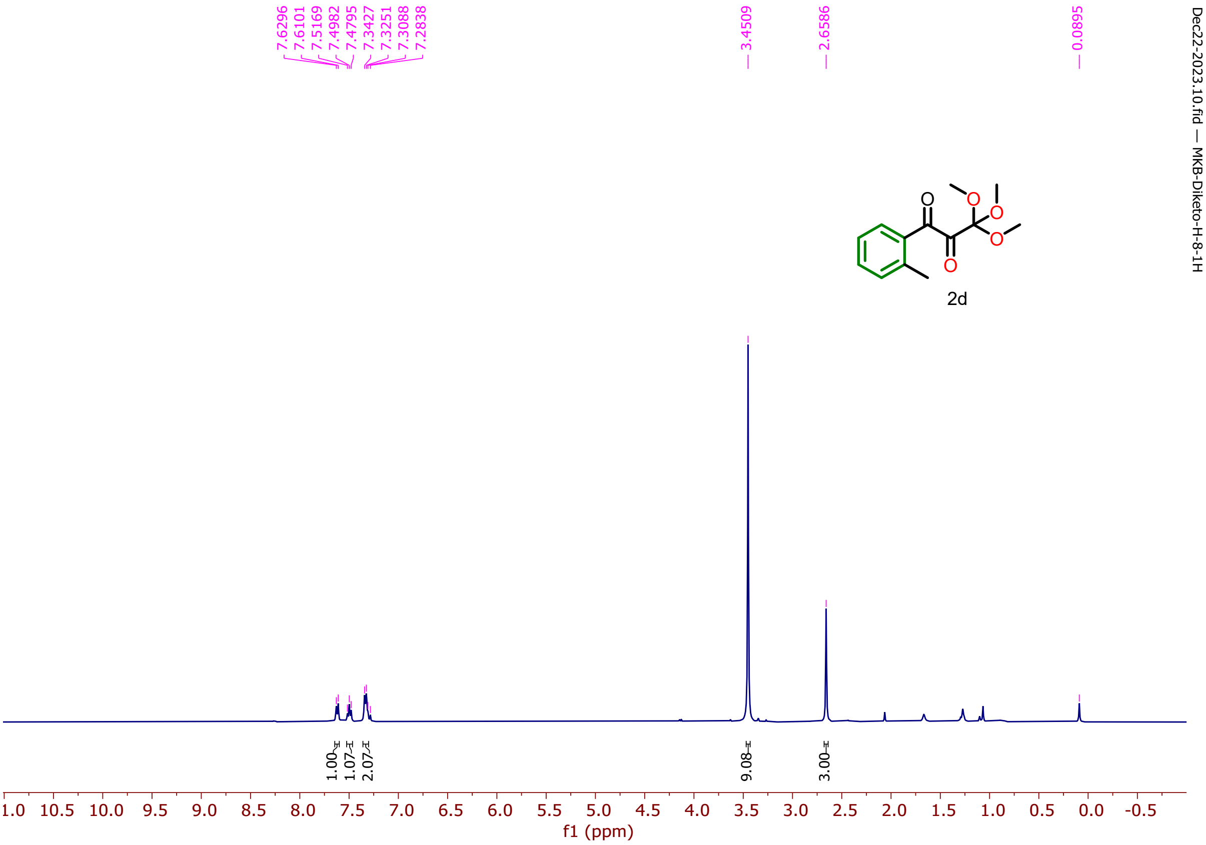


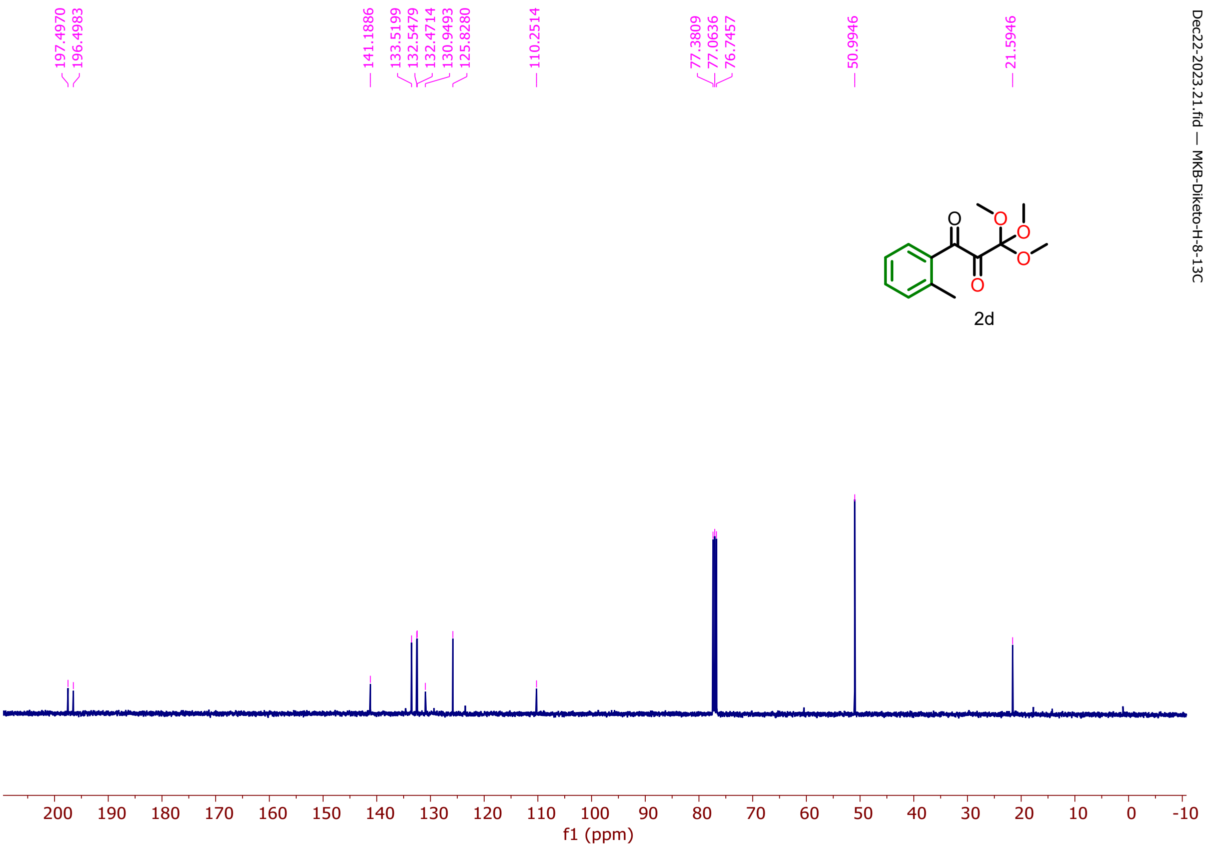
^{1}H NMR & ^{13}C NMR of 2c



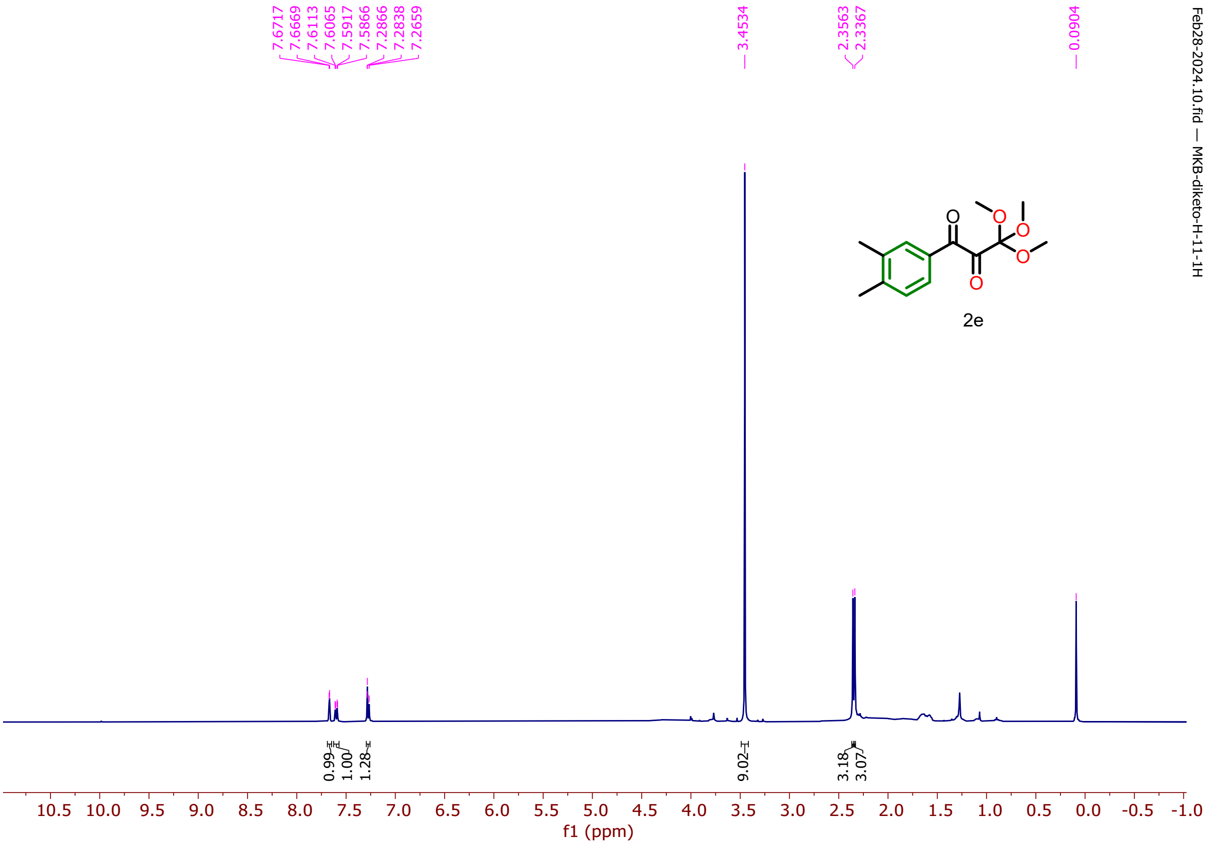


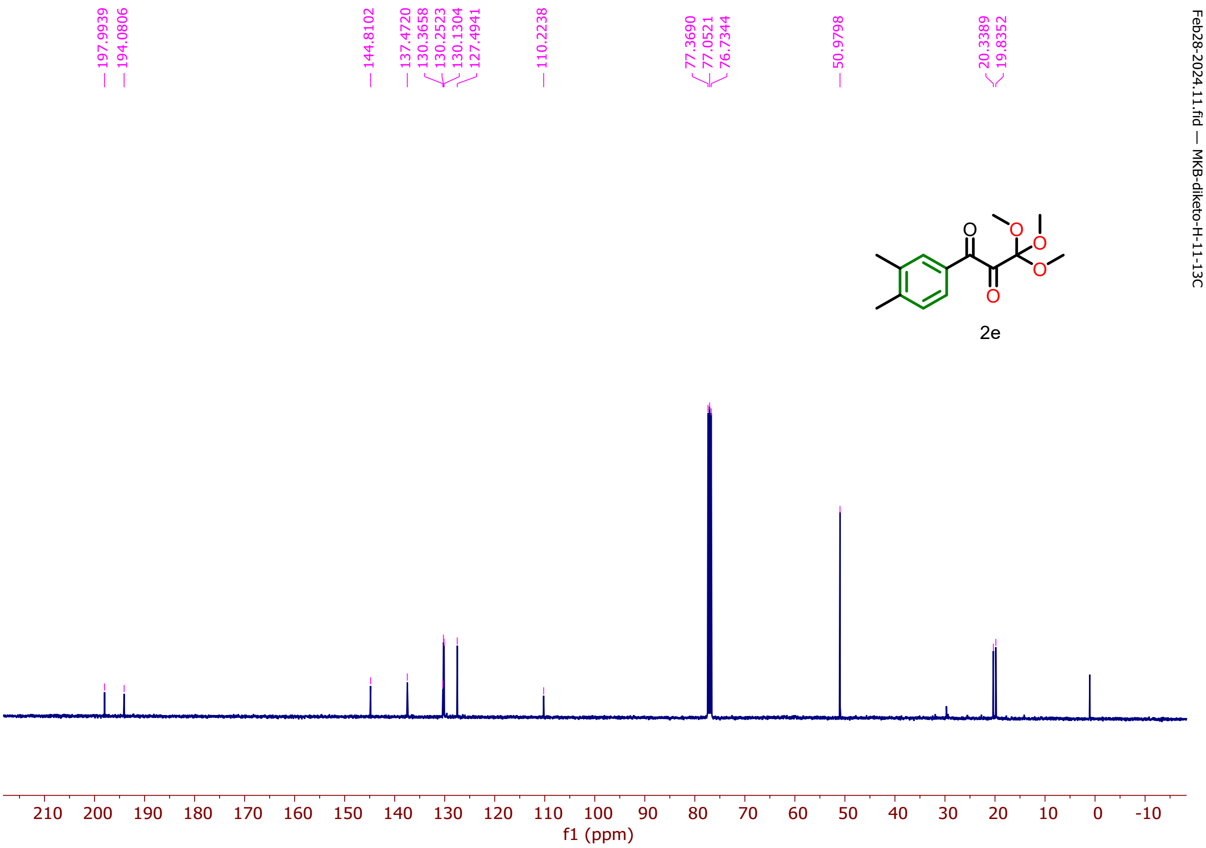
^{1}H NMR & ^{13}C NMR of 2d



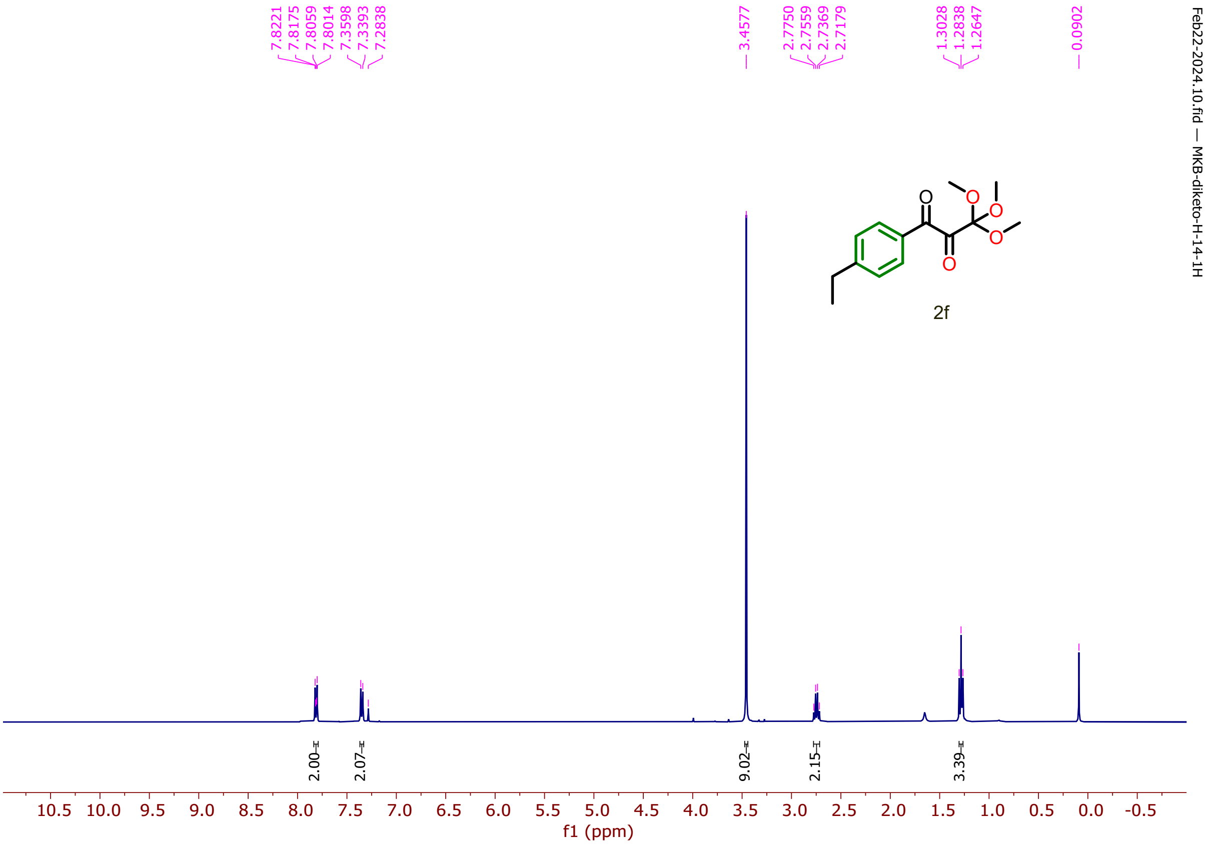


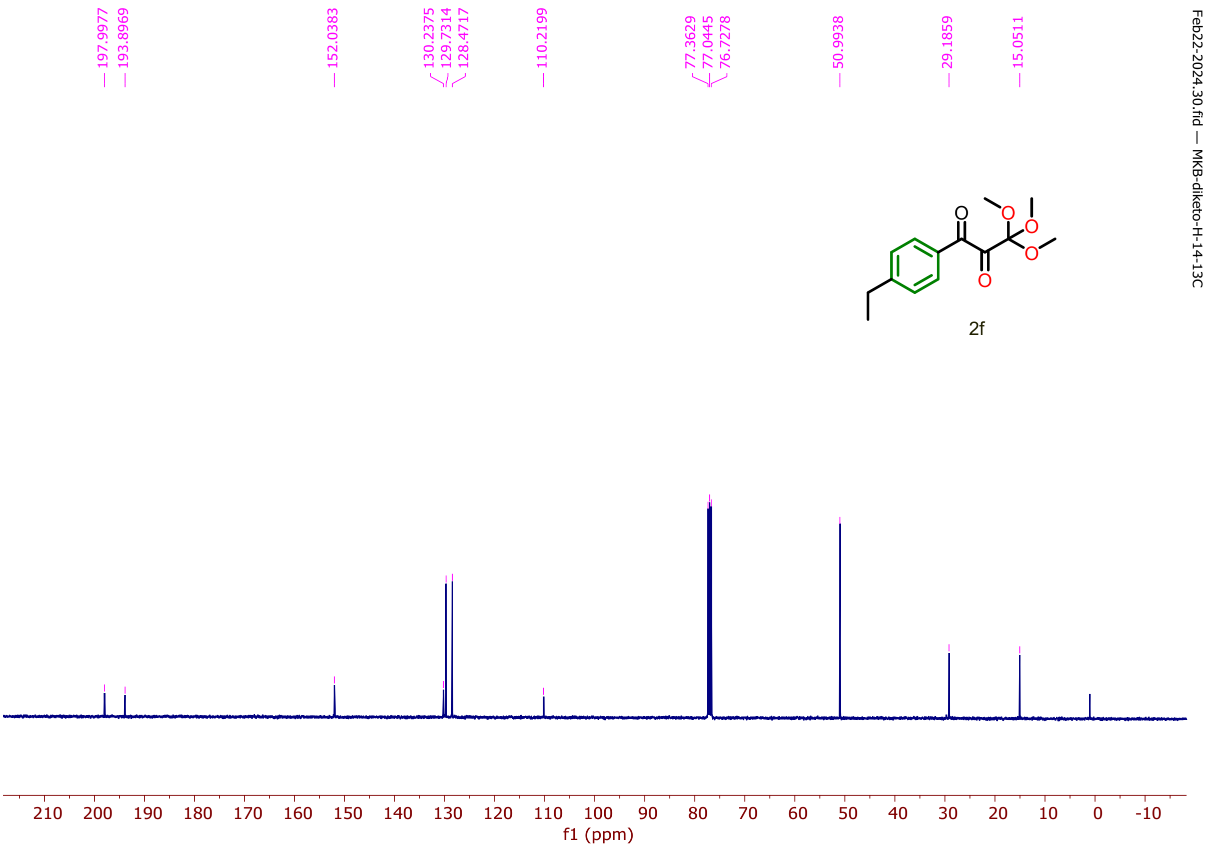
^{1}H NMR & ^{13}C NMR of 2e



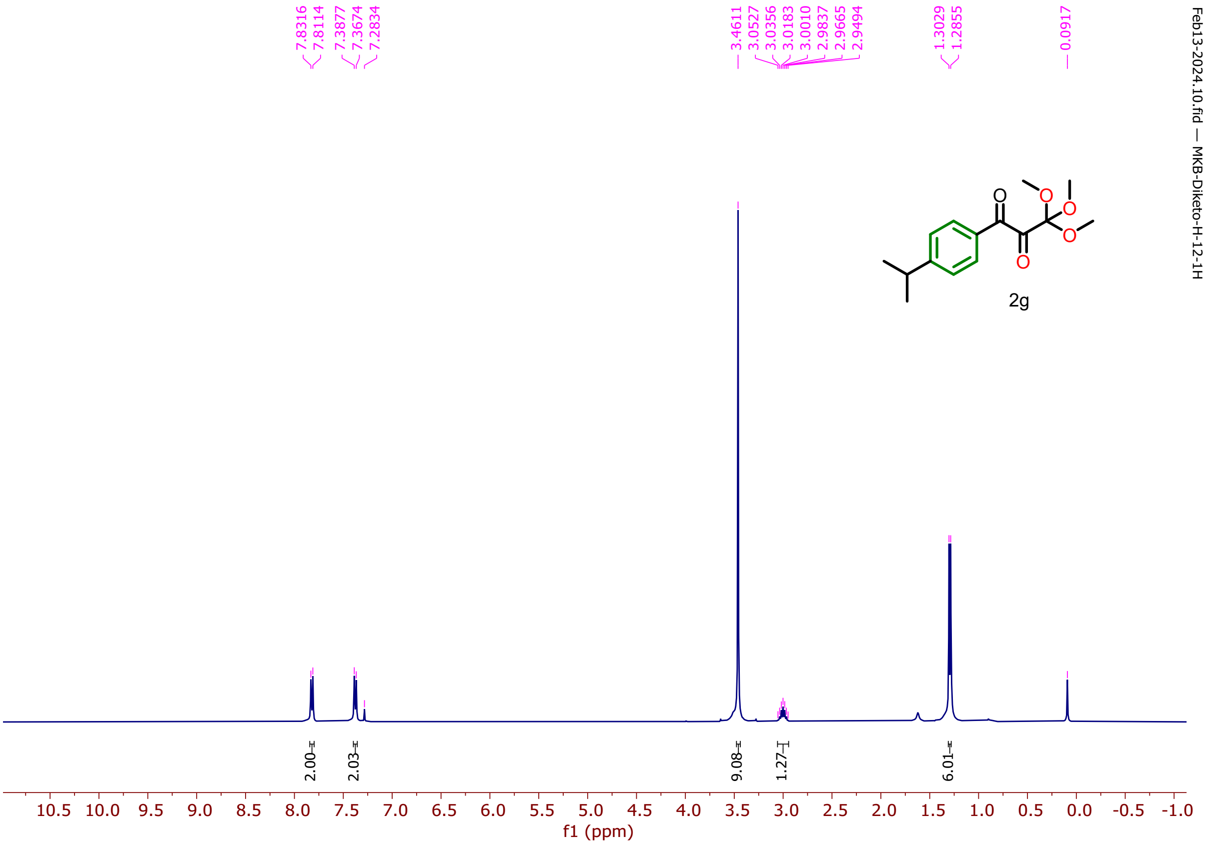


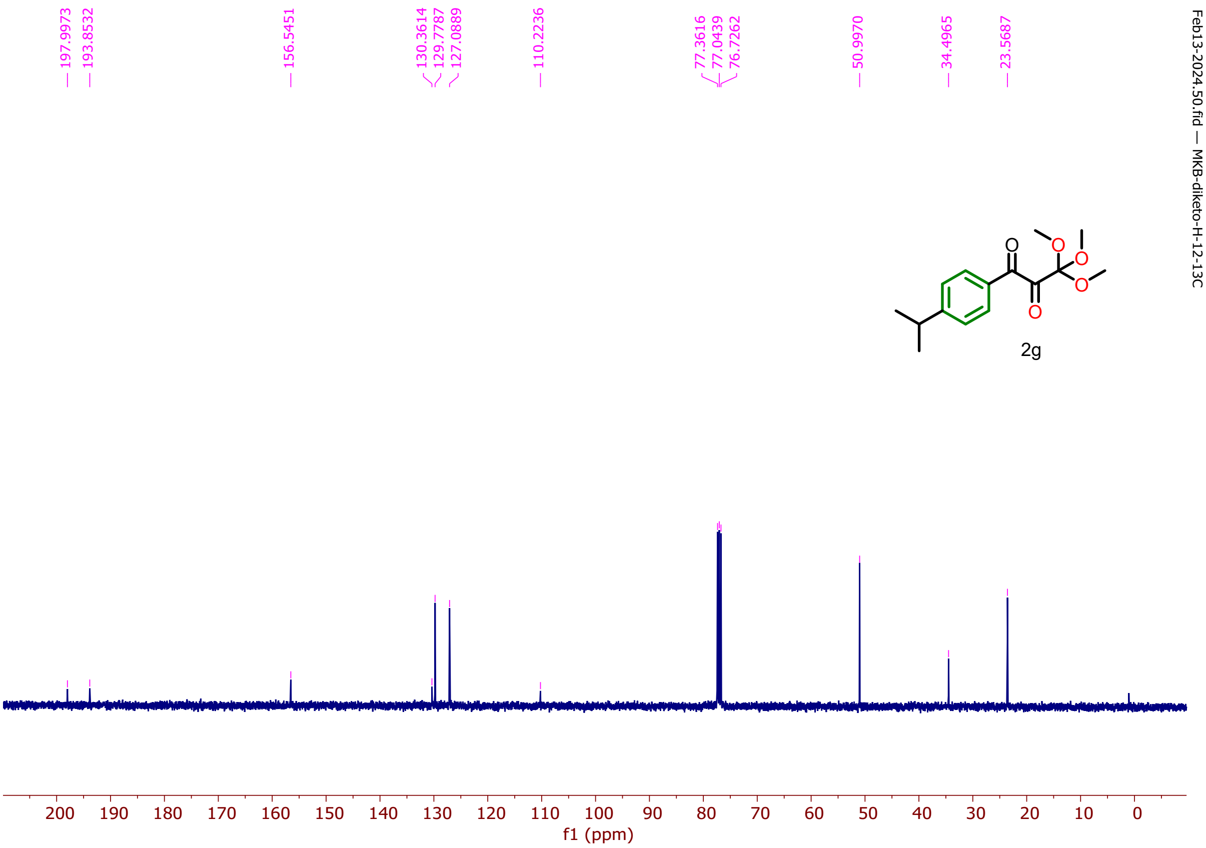
[1]H NMR & [13]C NMR of 2f



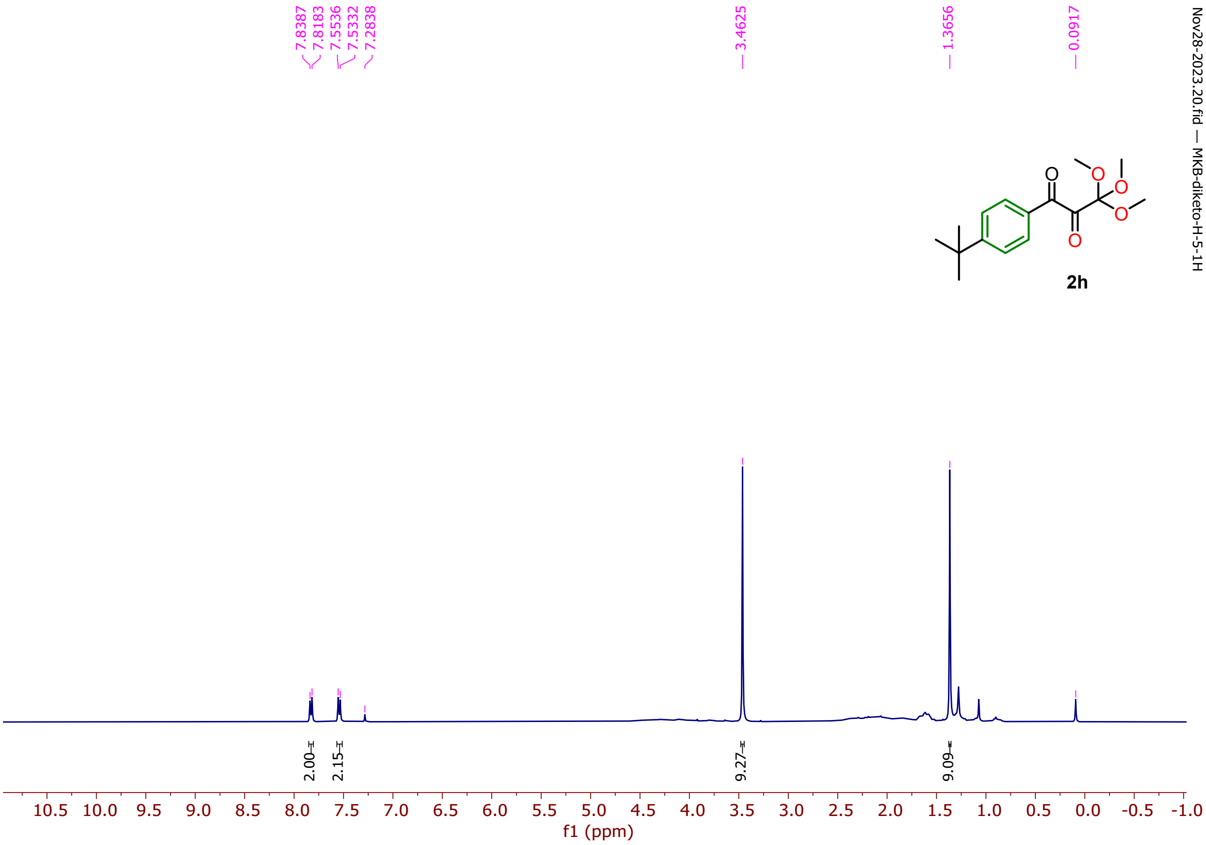


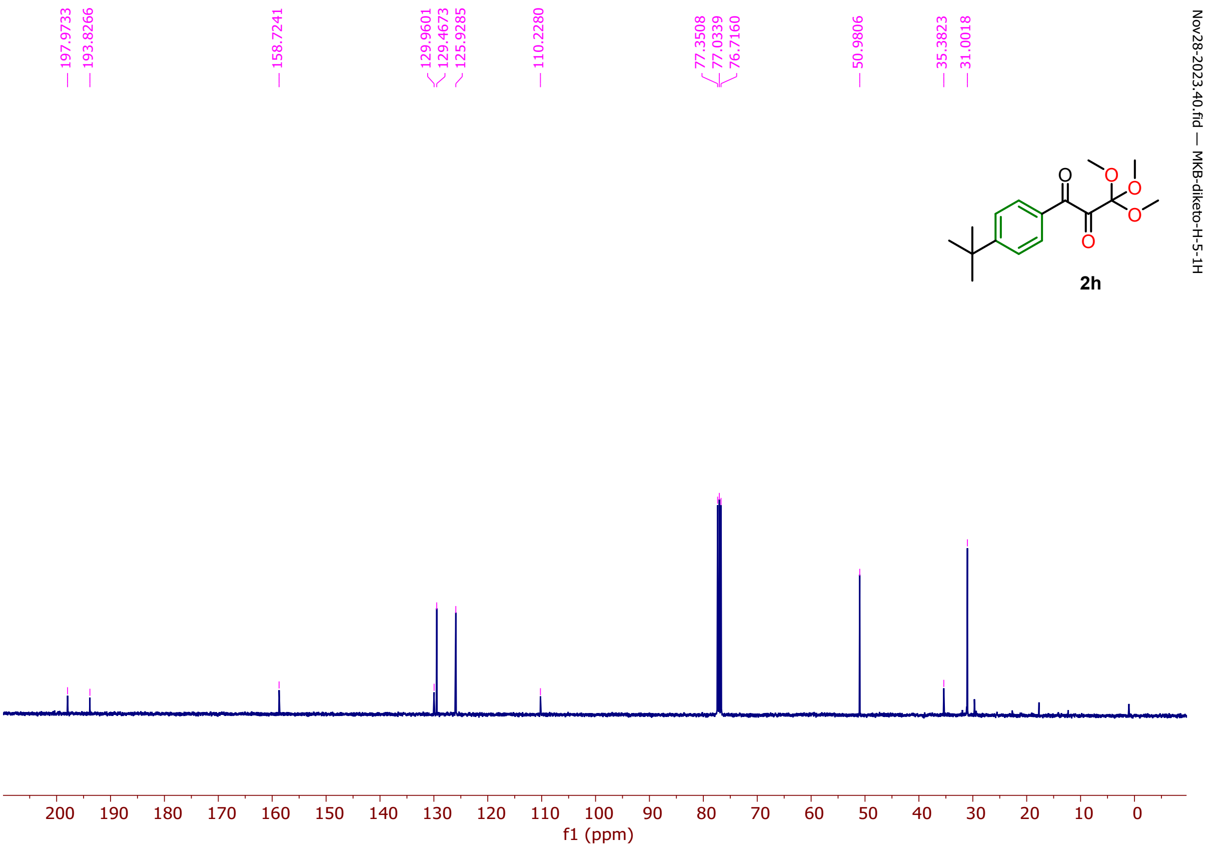
^{1}H NMR & ^{13}C NMR of 2g



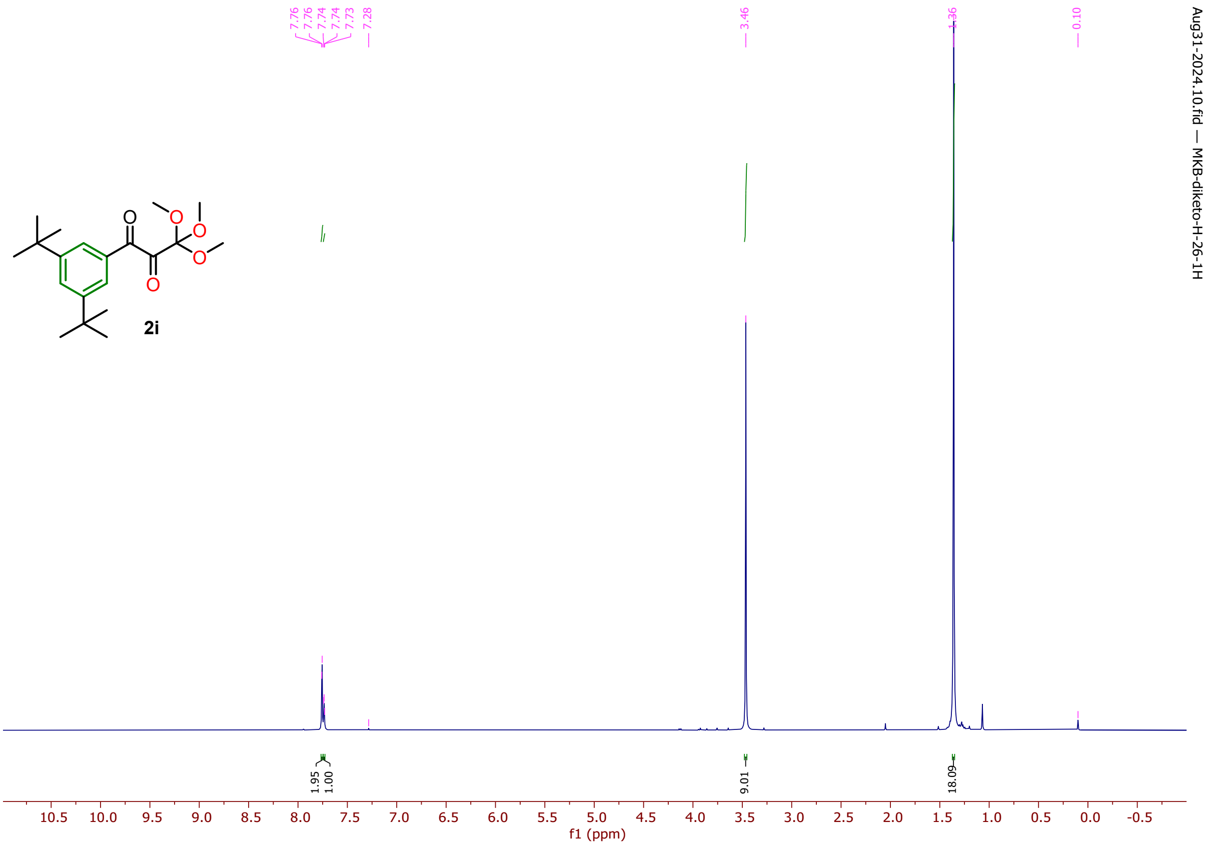


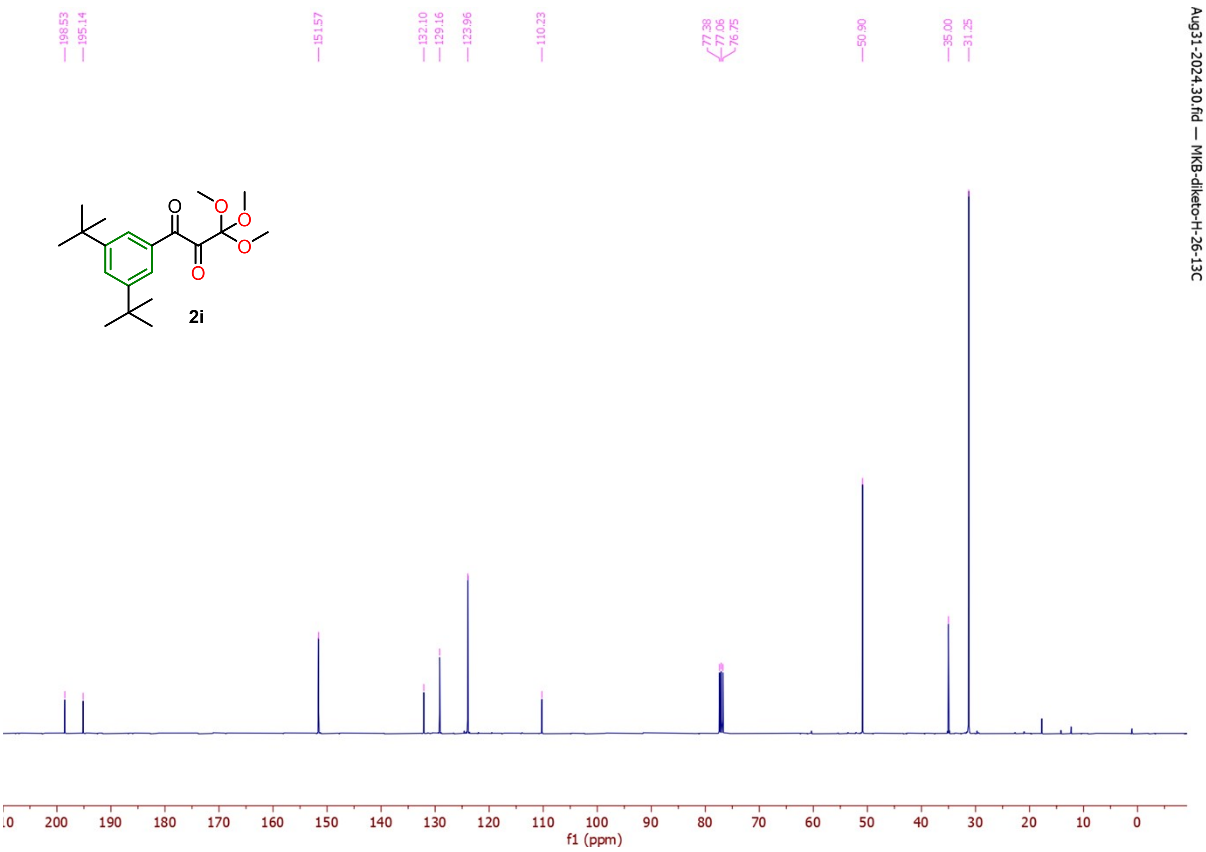
^{1}H NMR & ^{13}C NMR of 2h



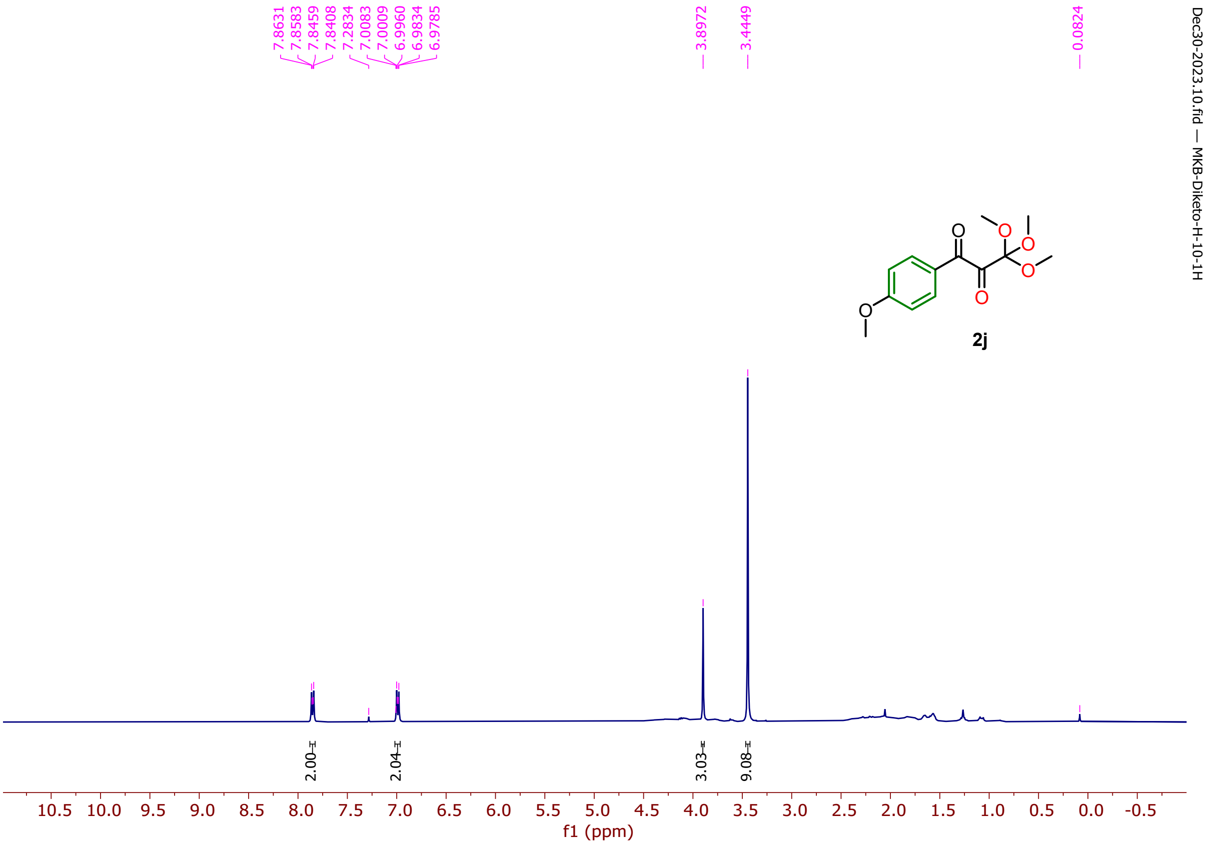


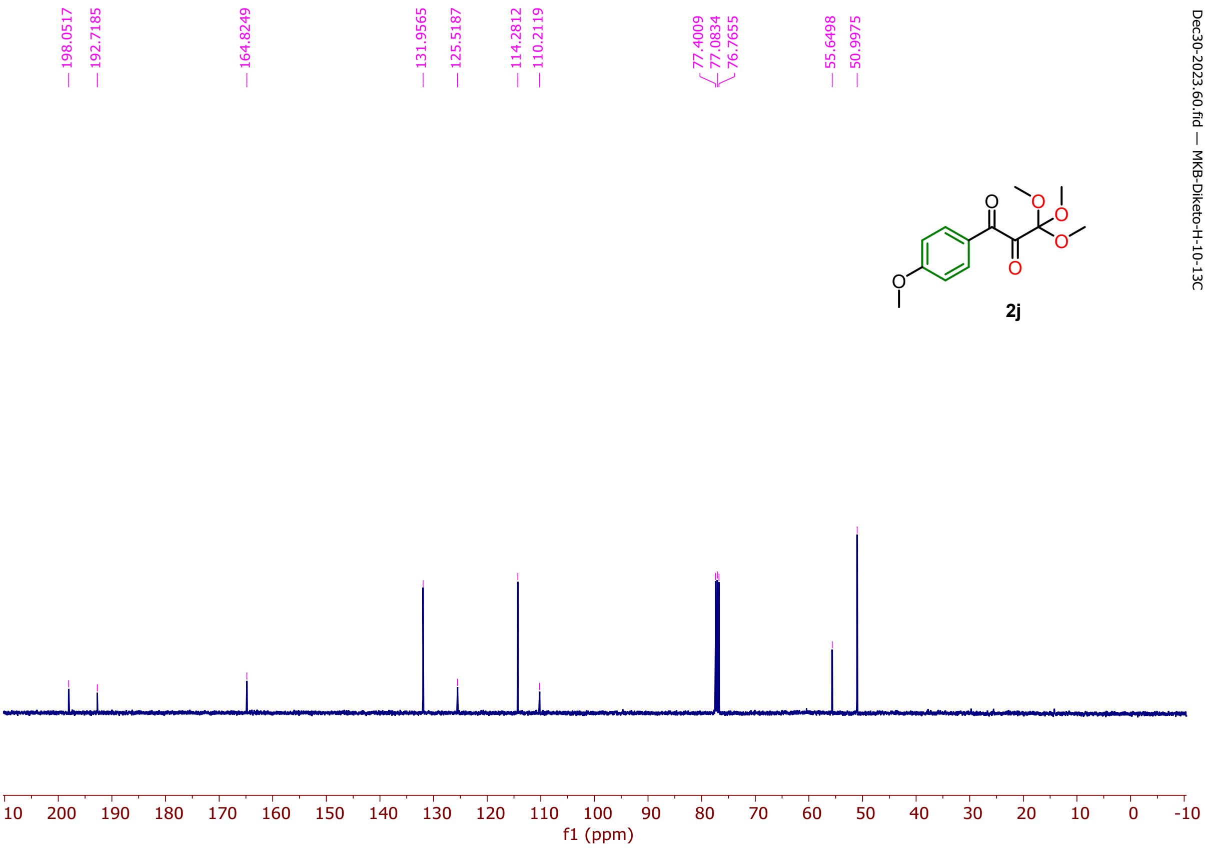
[1]H NMR & [13]C NMR of 2i



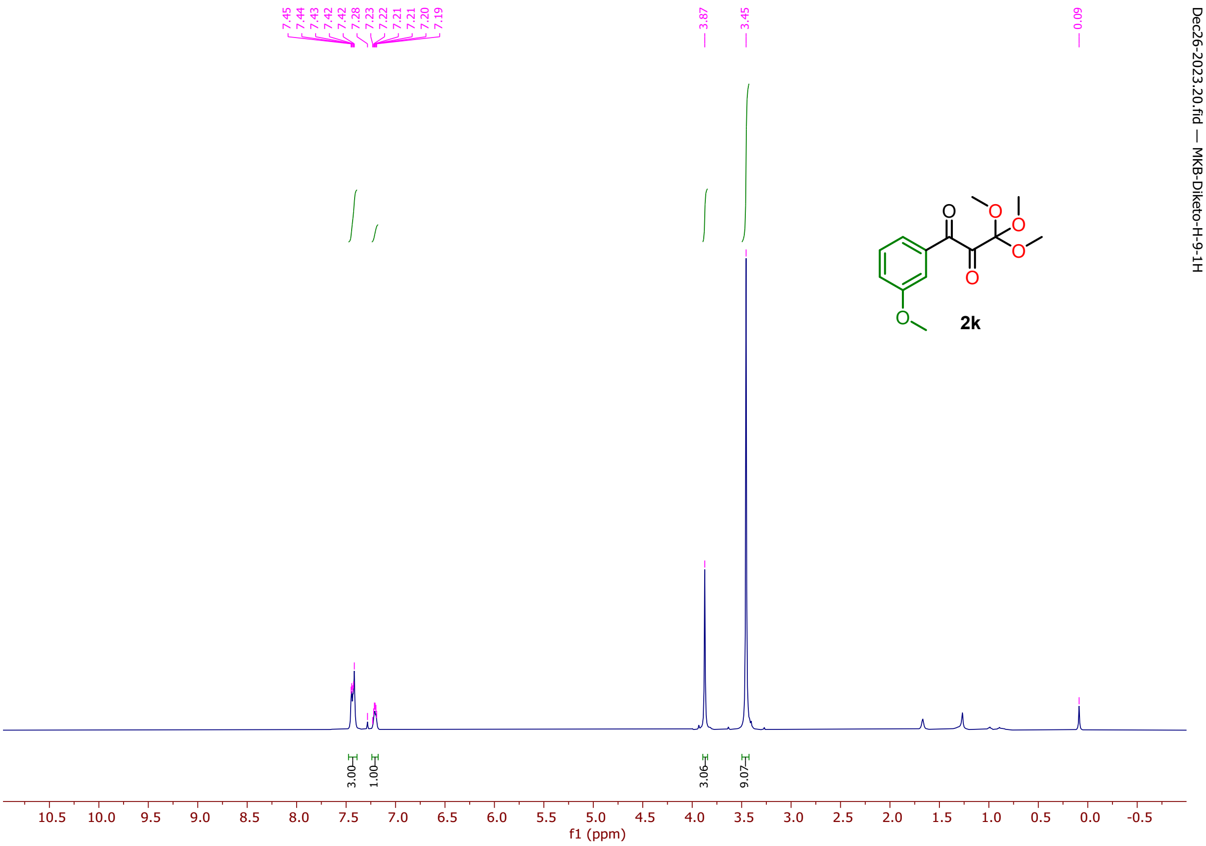


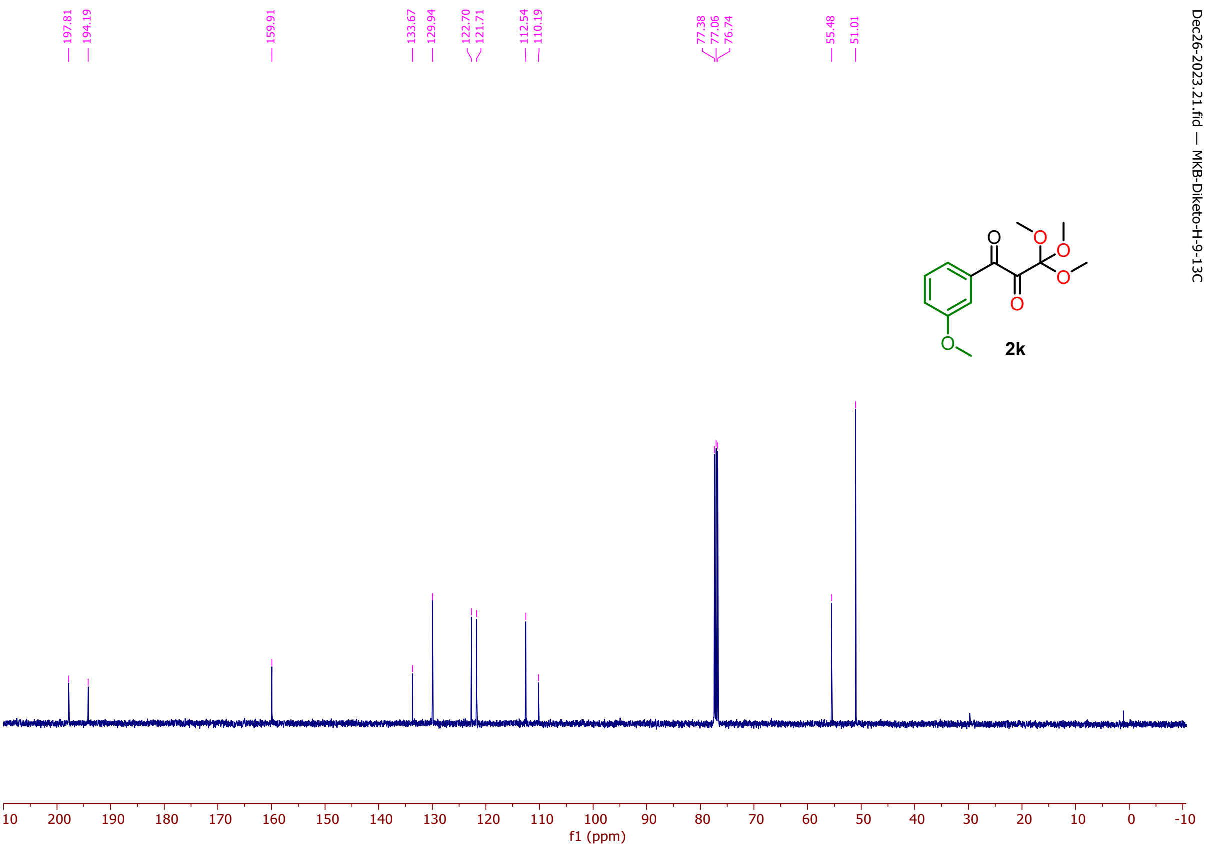
^{1}H NMR & ^{13}C NMR of 2j



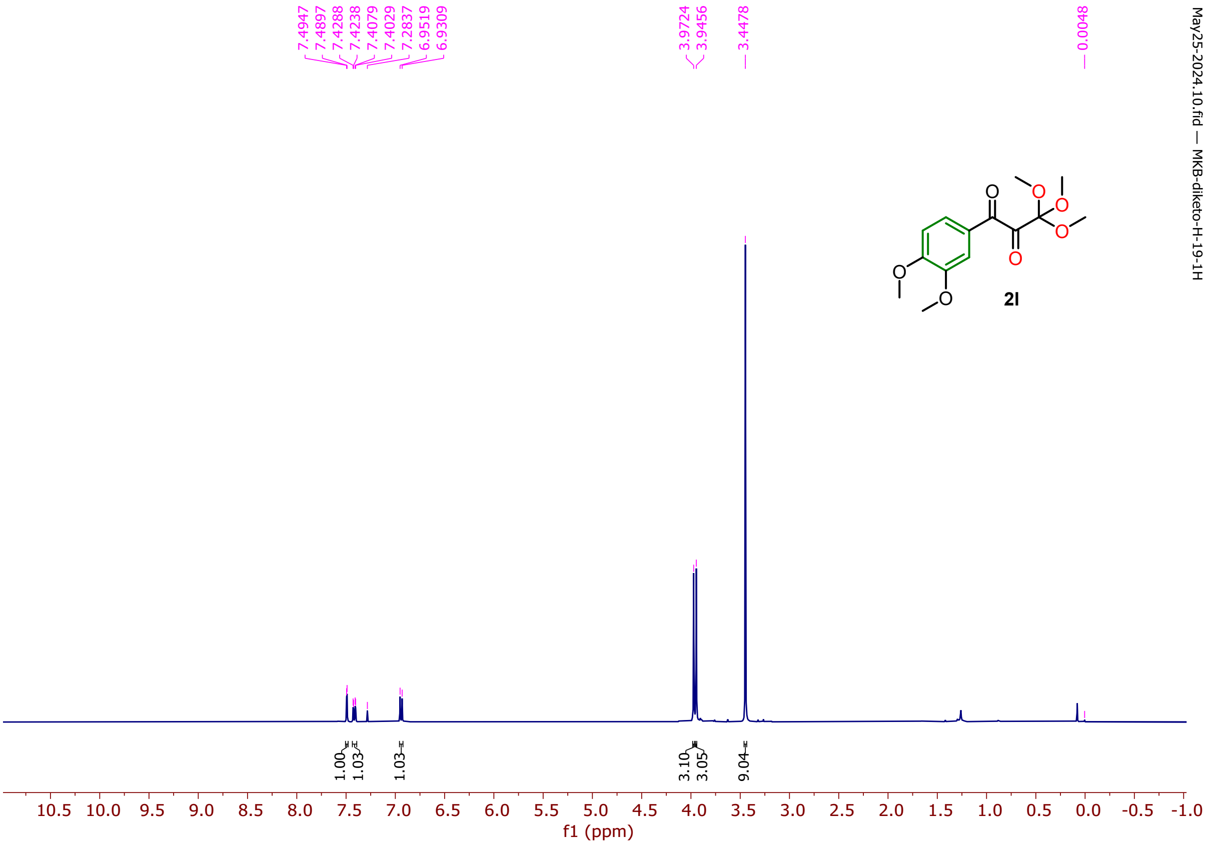


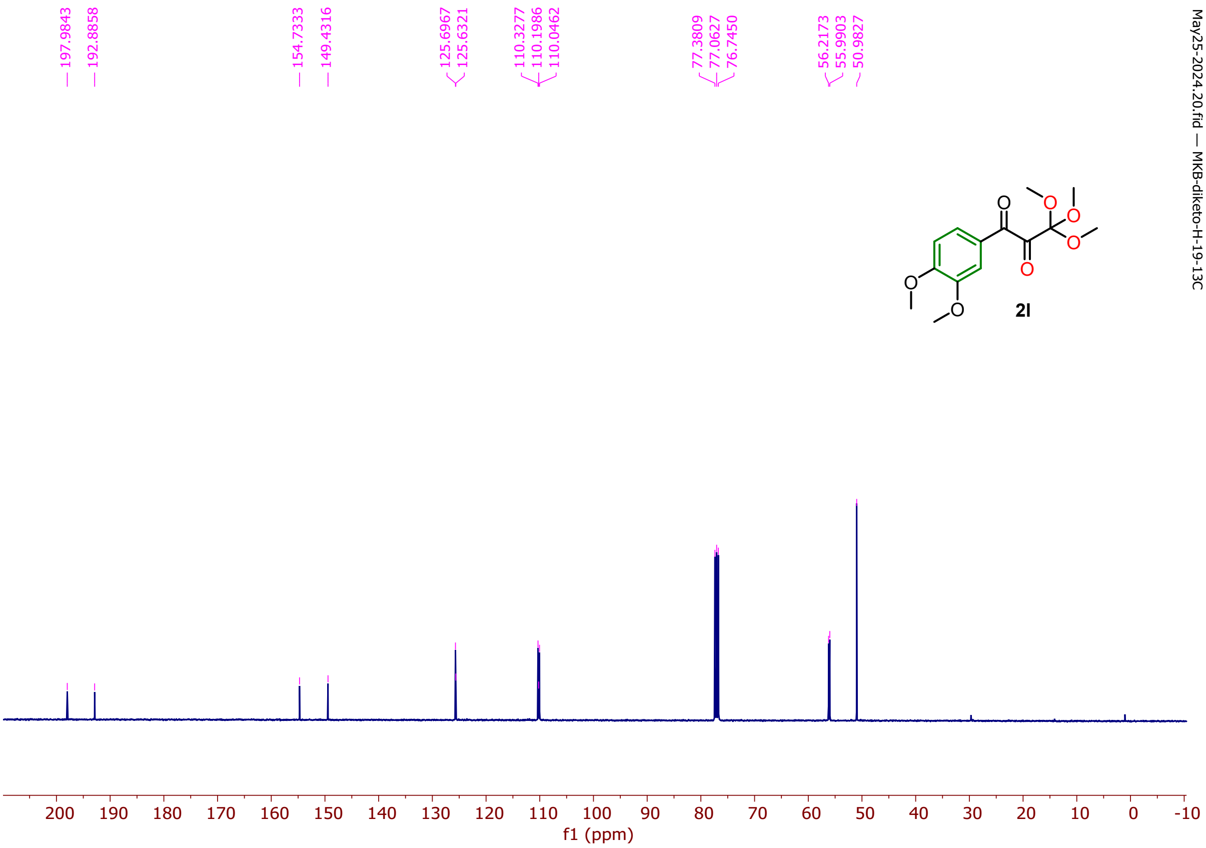
^{1}H NMR & ^{13}C NMR of 2k



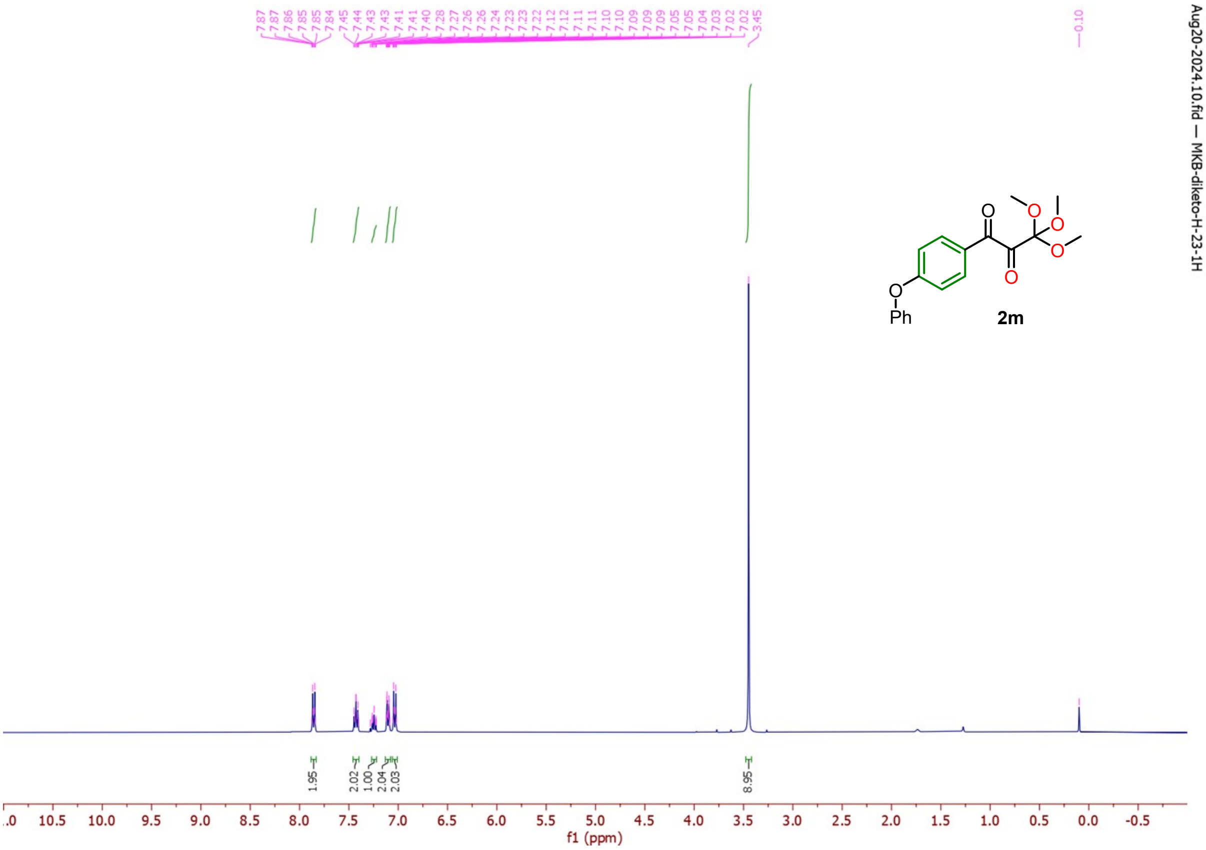


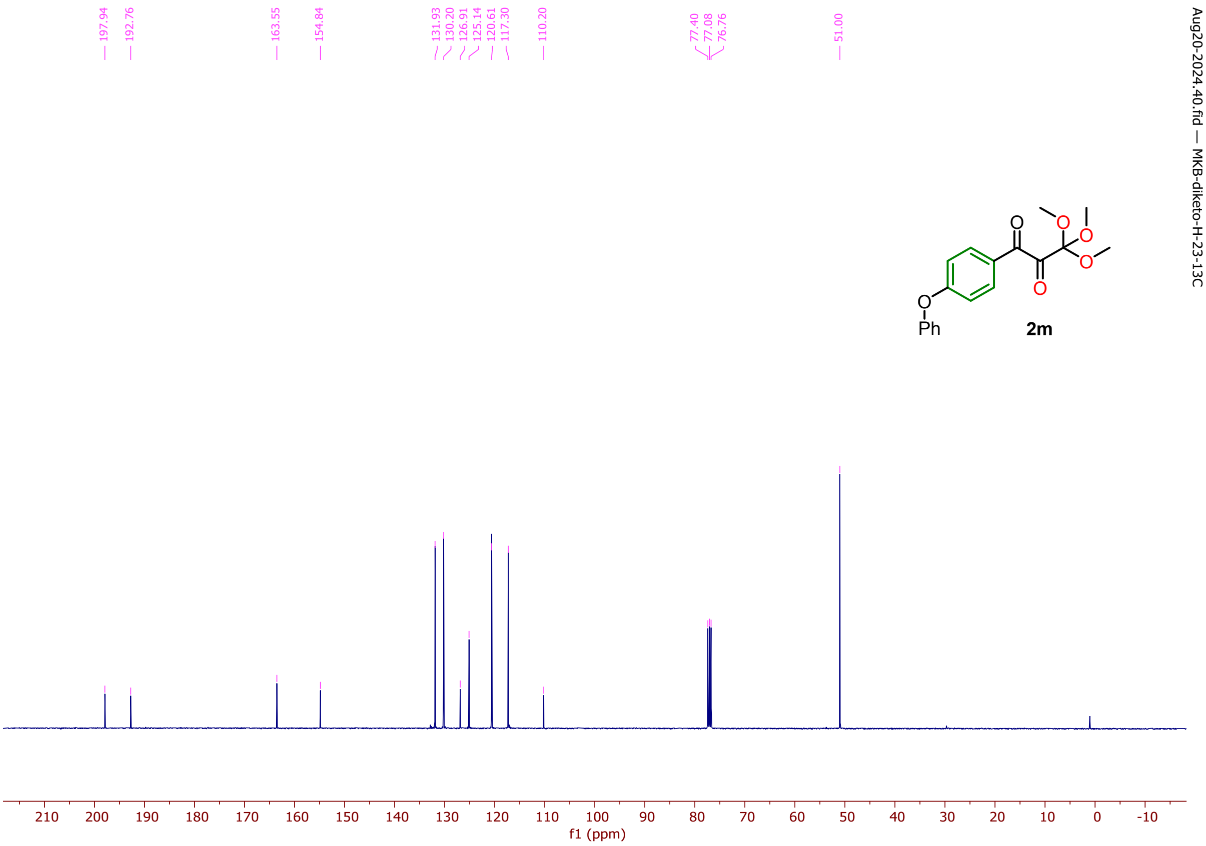
^{1}H NMR & ^{13}C NMR of 2l



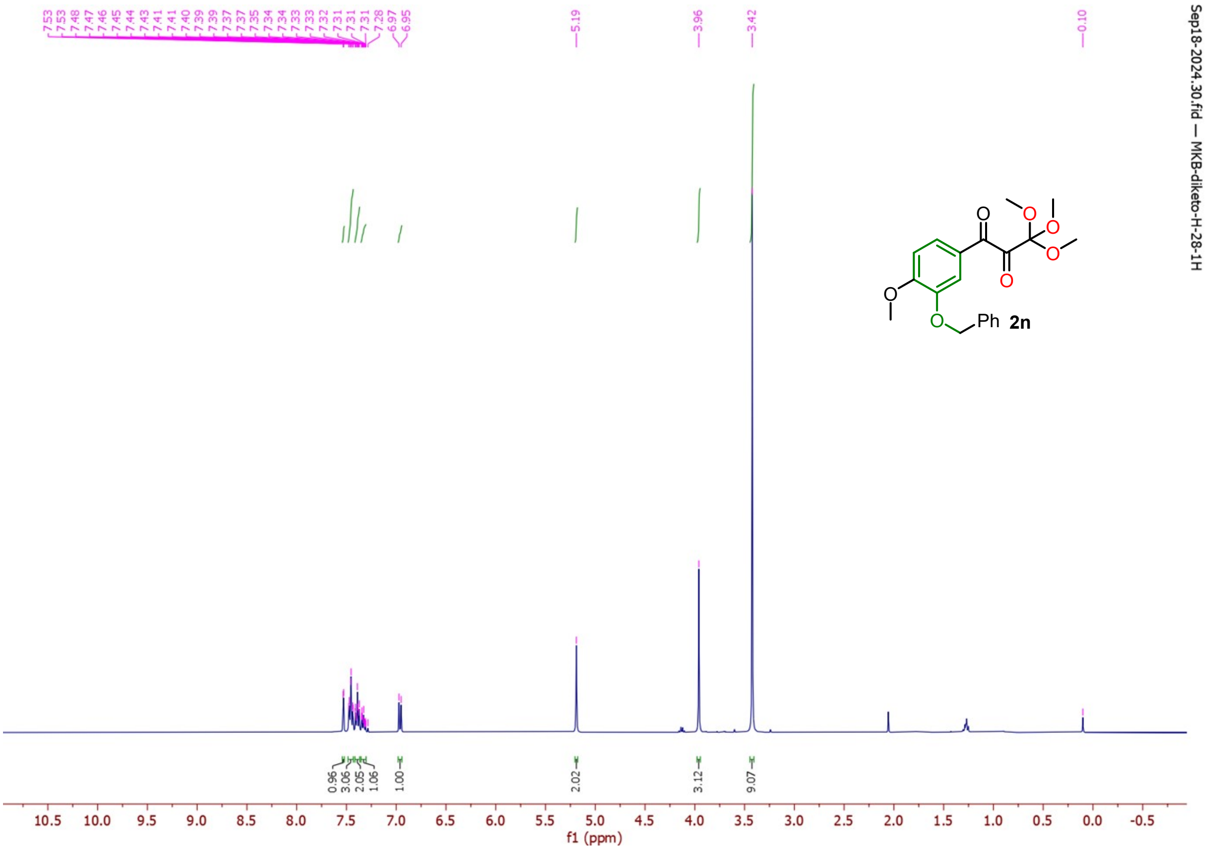


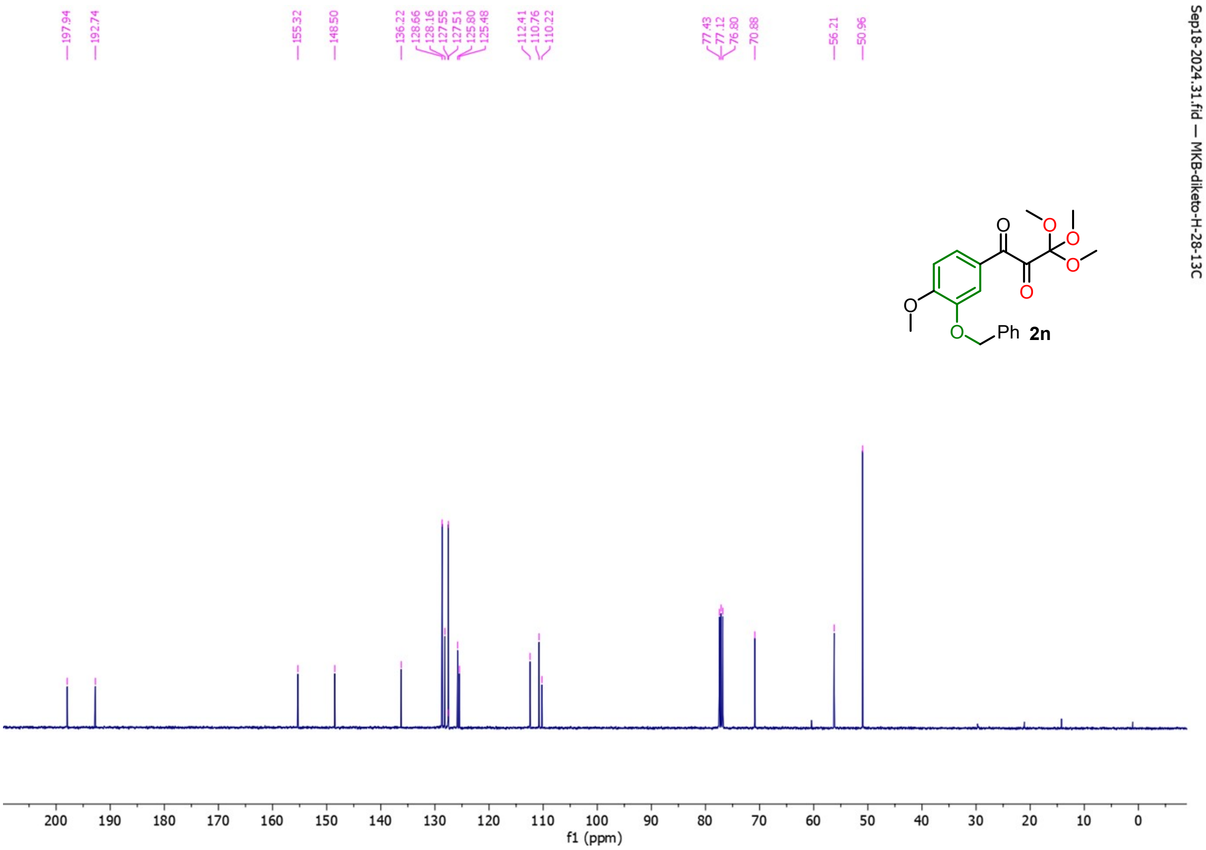
[1]H NMR & [13]C NMR of 2m



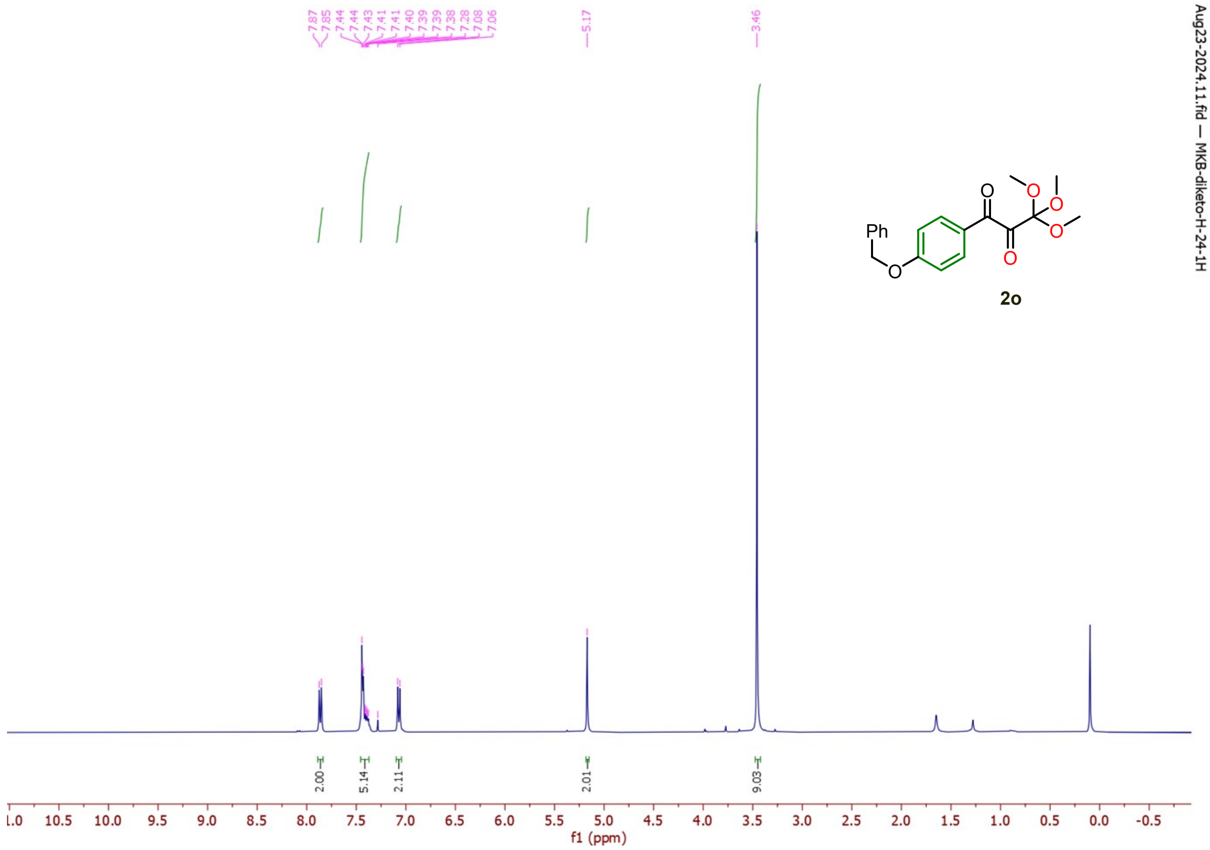


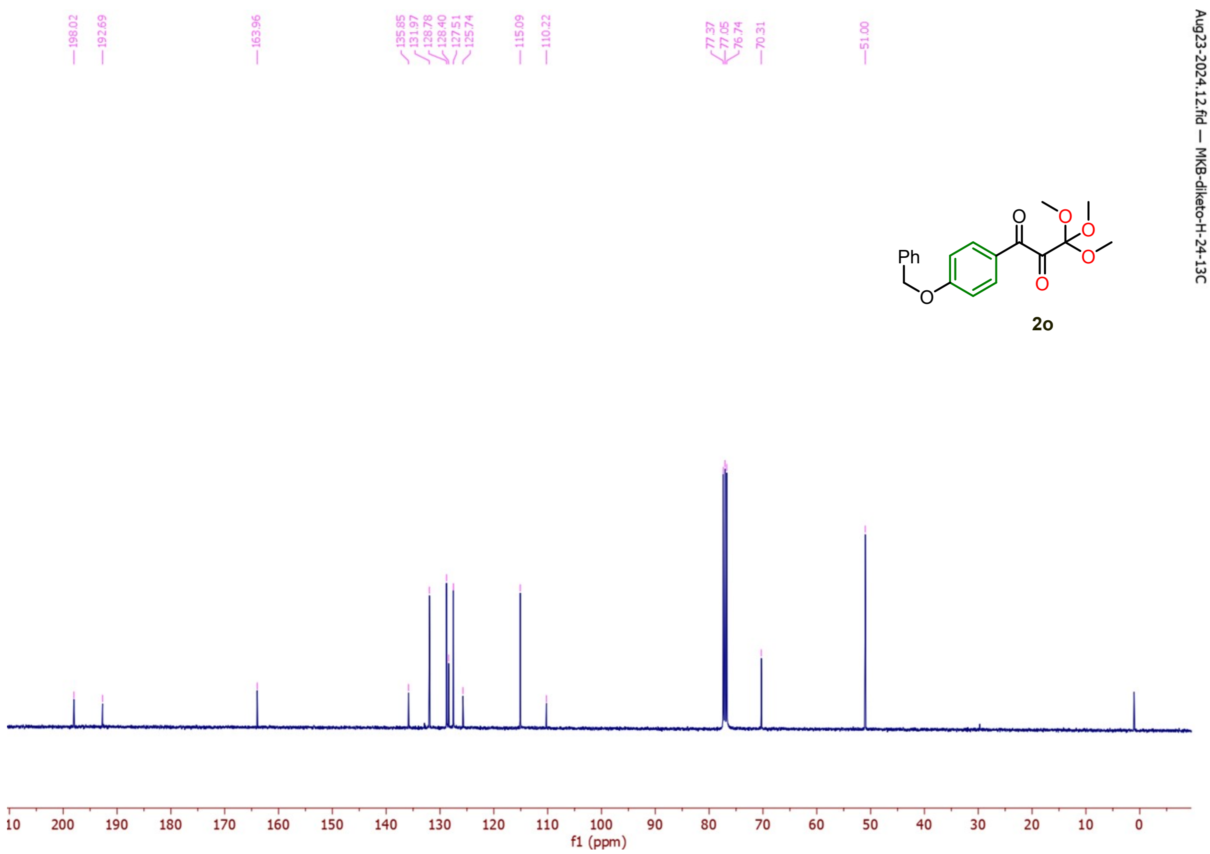
^{1}H NMR & ^{13}C NMR of 2n



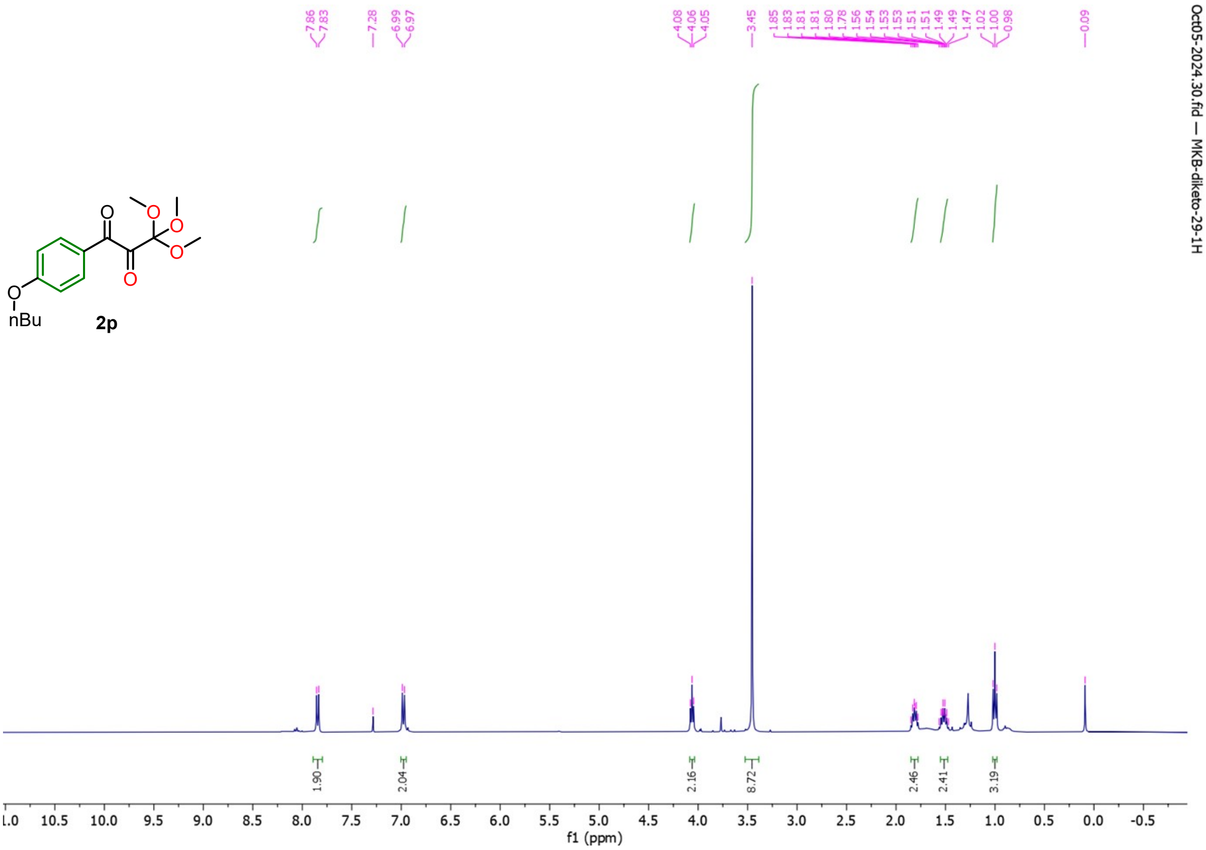


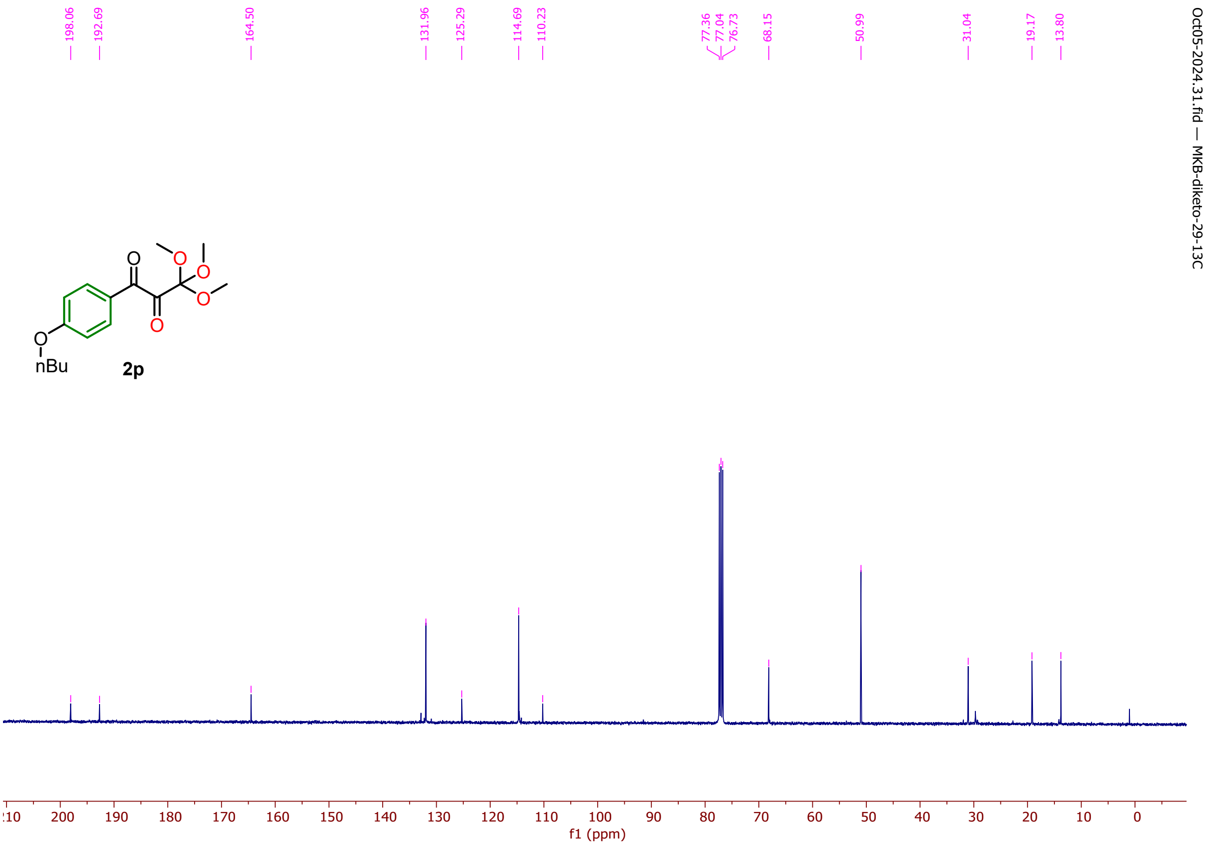
[1]H NMR & [13]C NMR of 2o



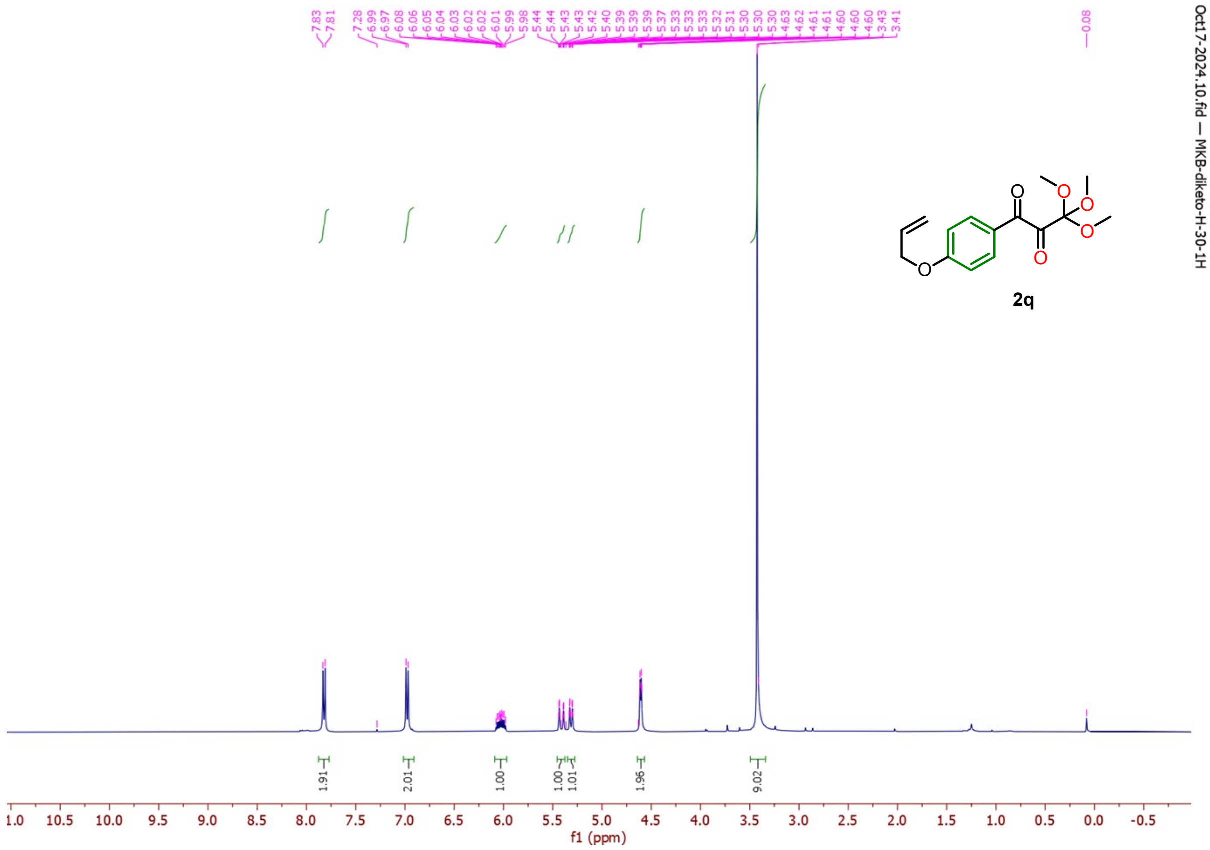


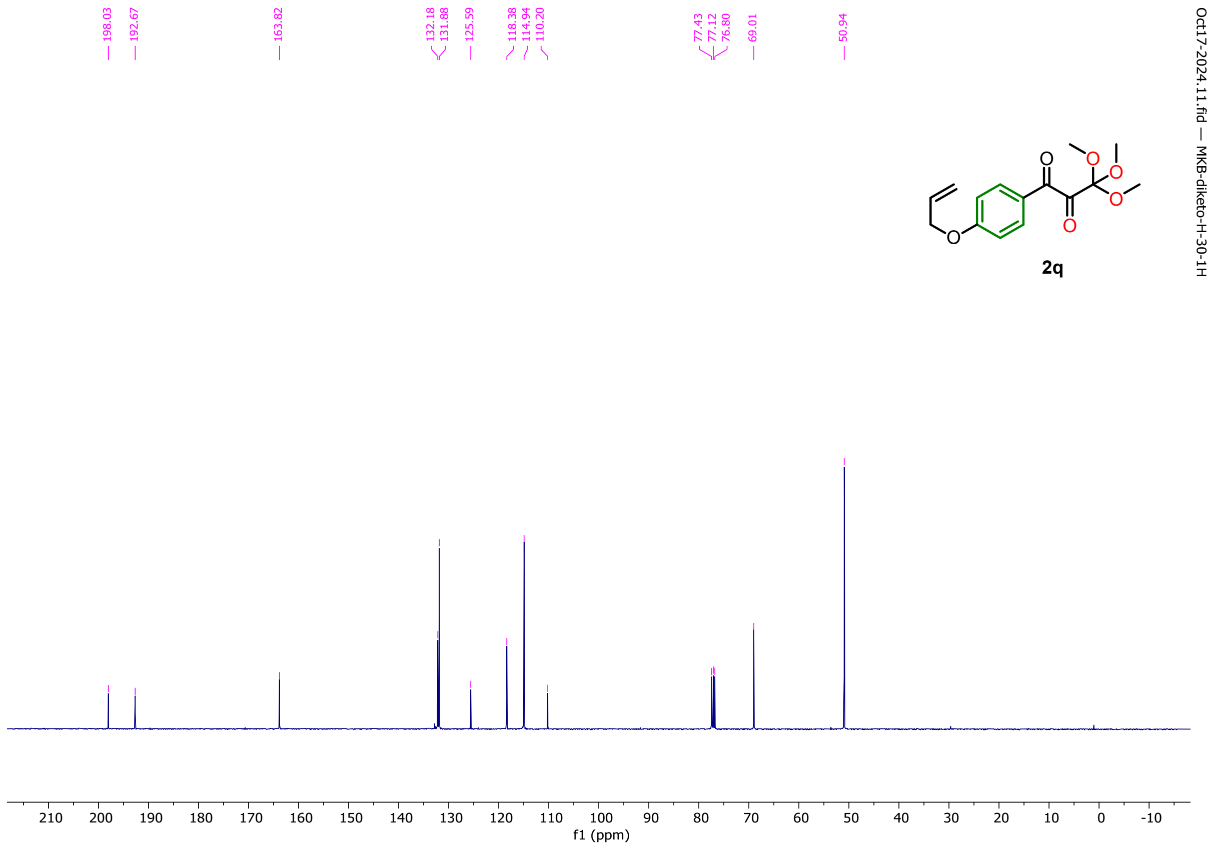
^{1}H NMR & ^{13}C NMR of 2p



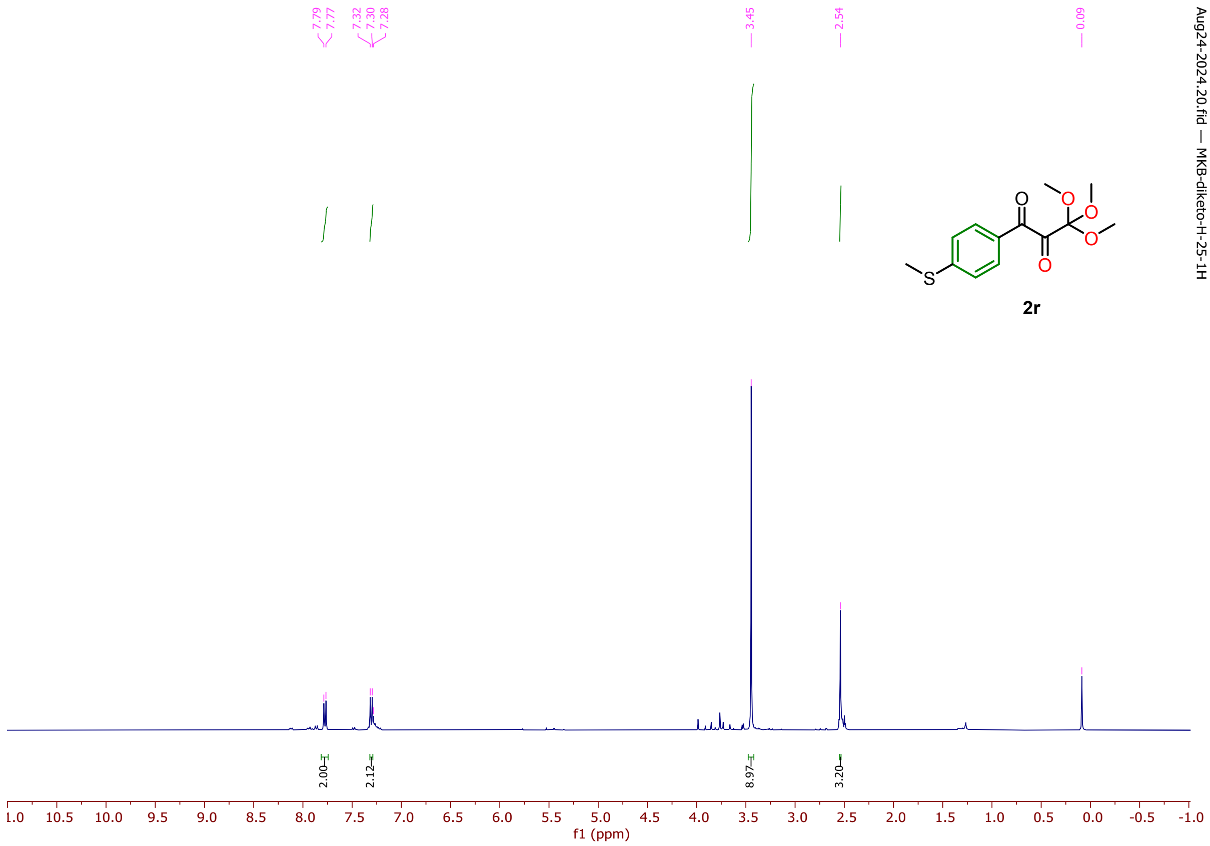


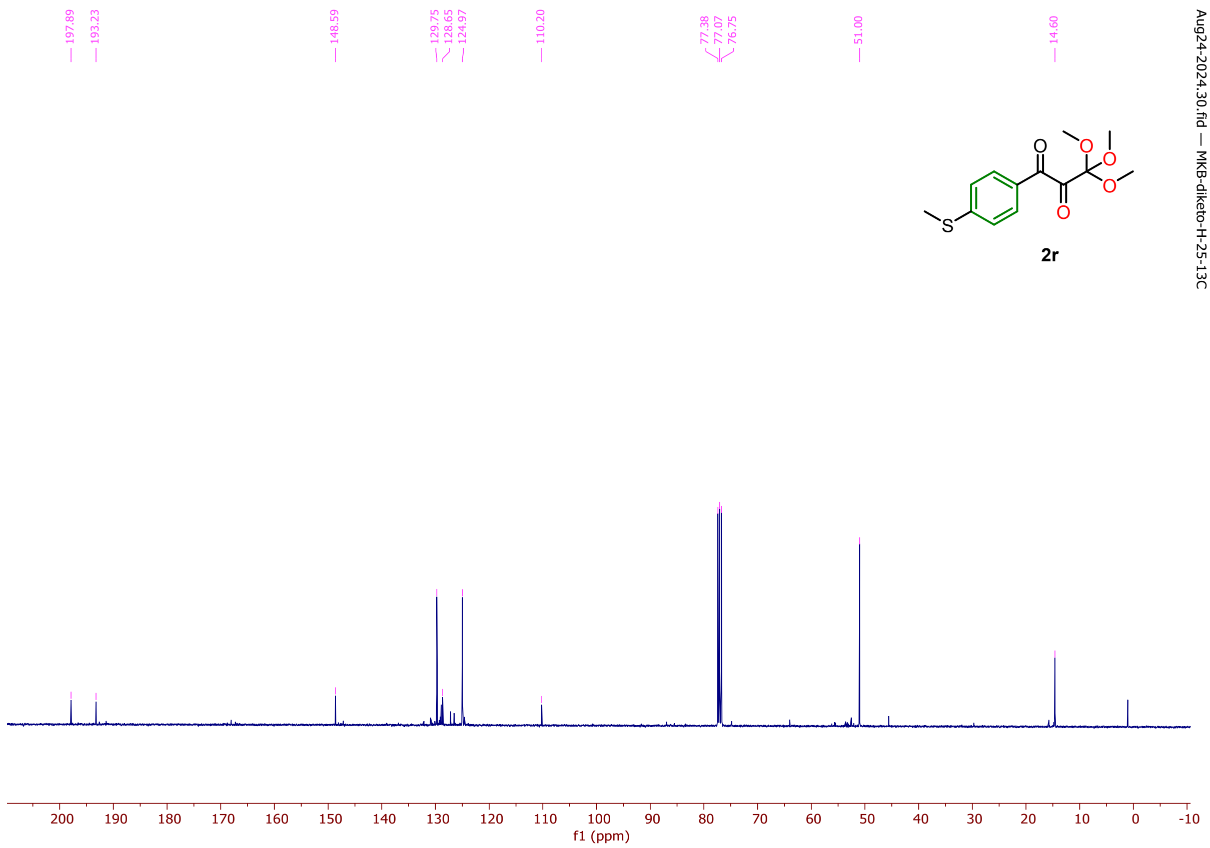
[1]H NMR & [13]C NMR of 2q



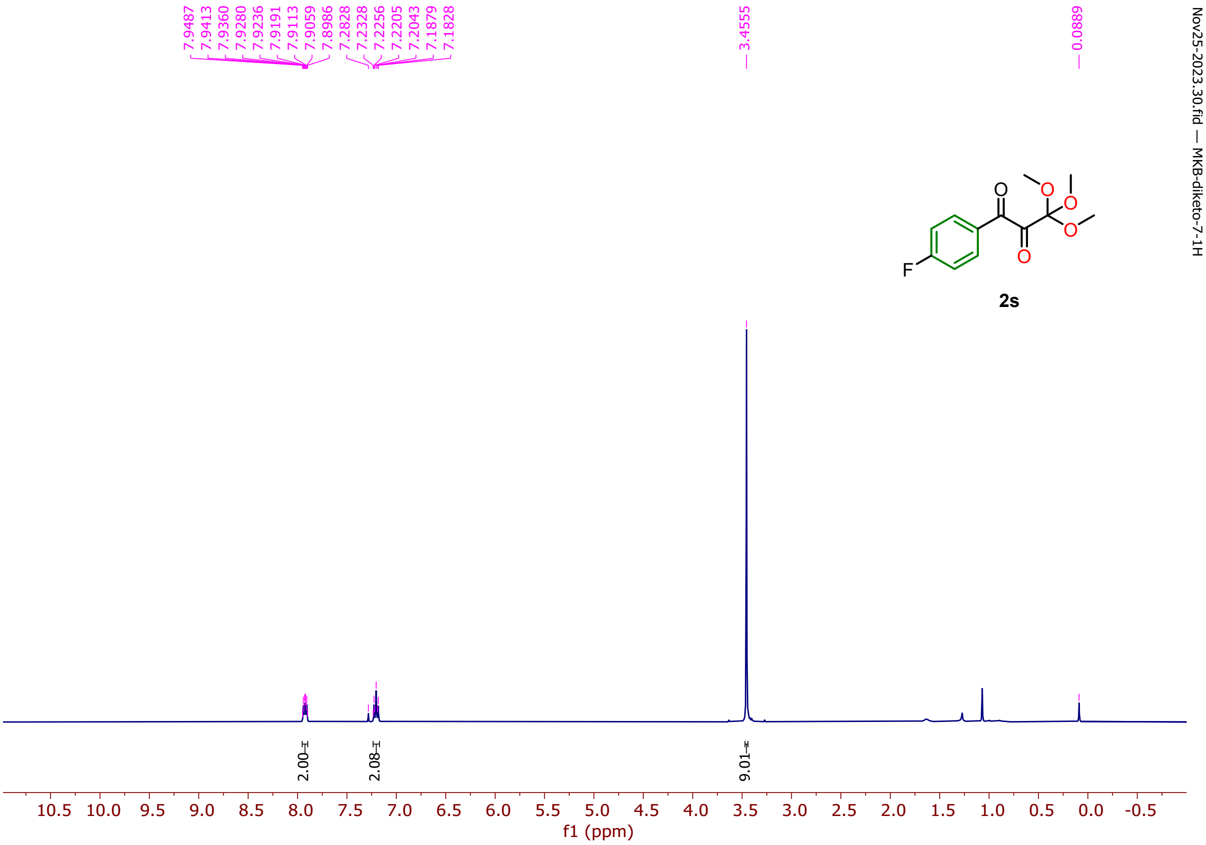


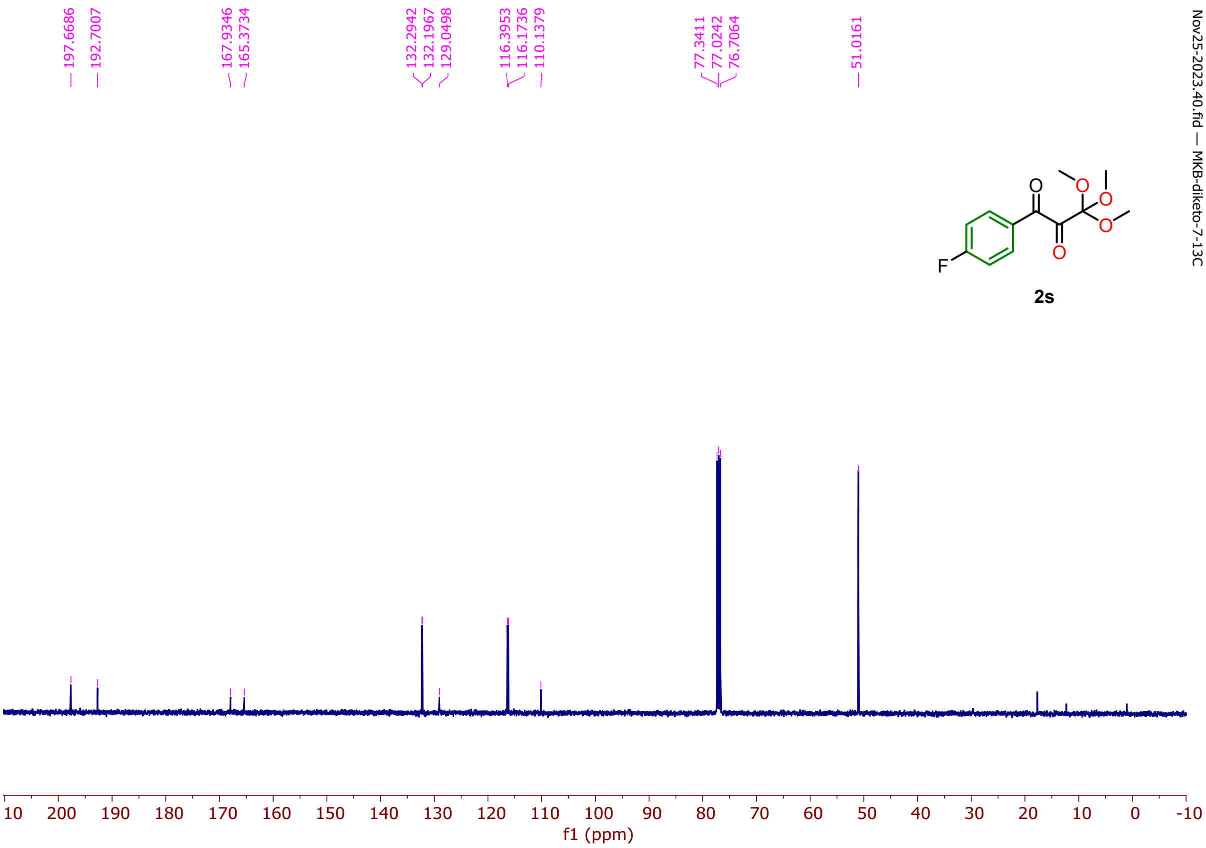
[1]H NMR & [13]C NMR of 2r

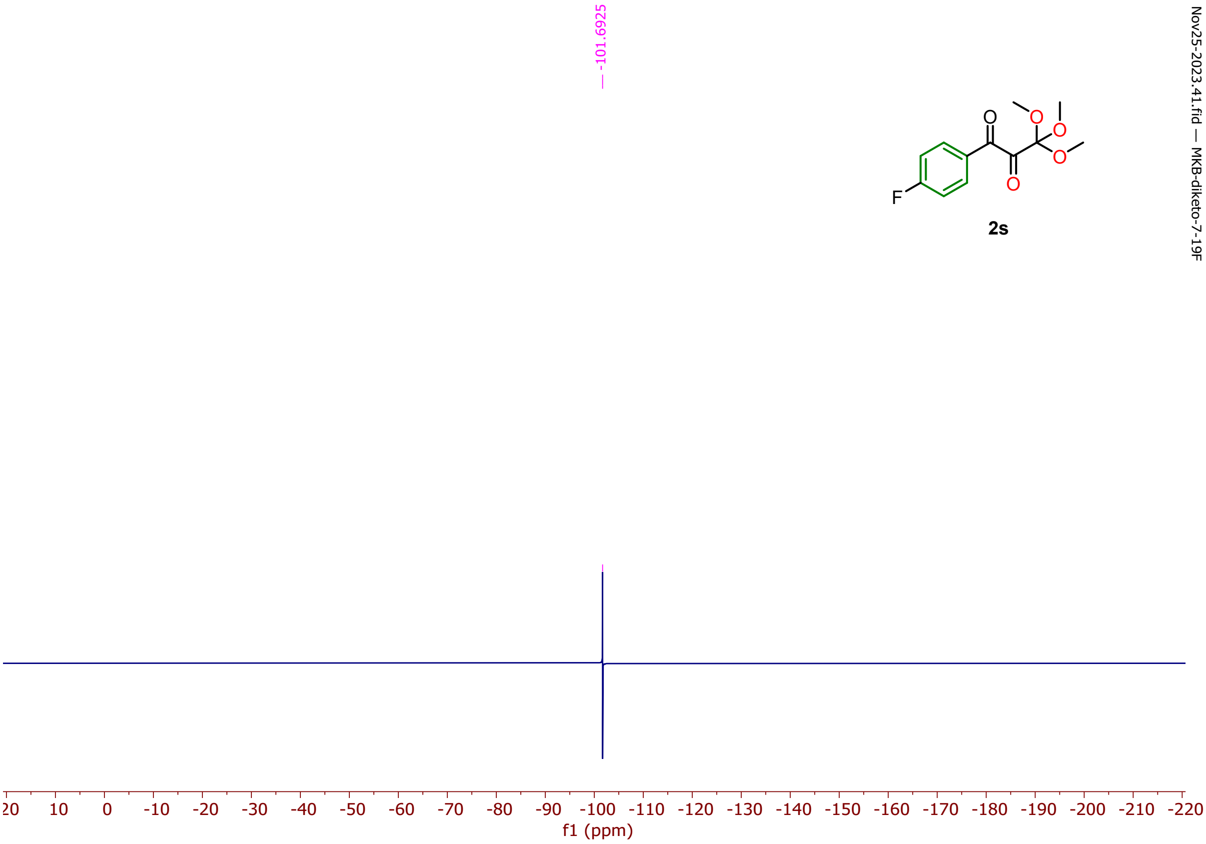




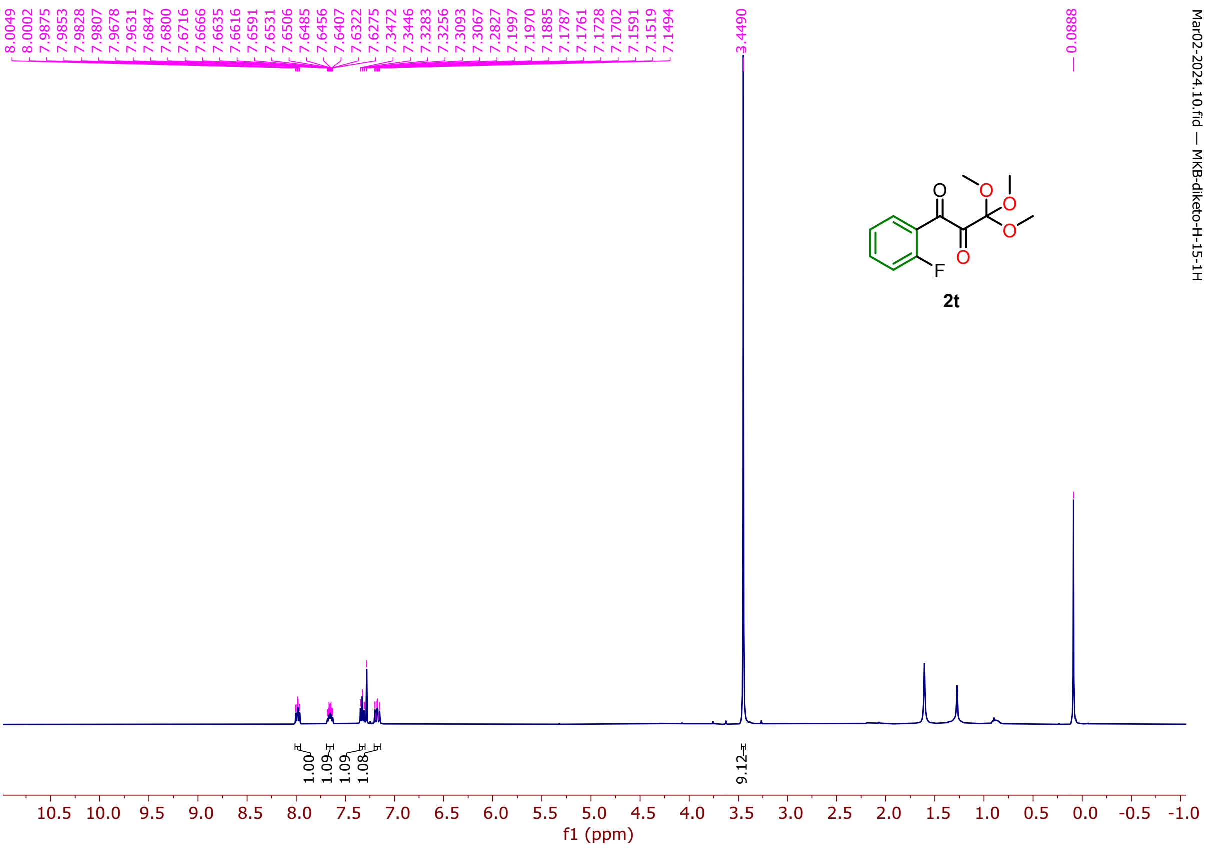
^{1}H NMR, ^{13}C NMR & ^{19}F NMR of 2s

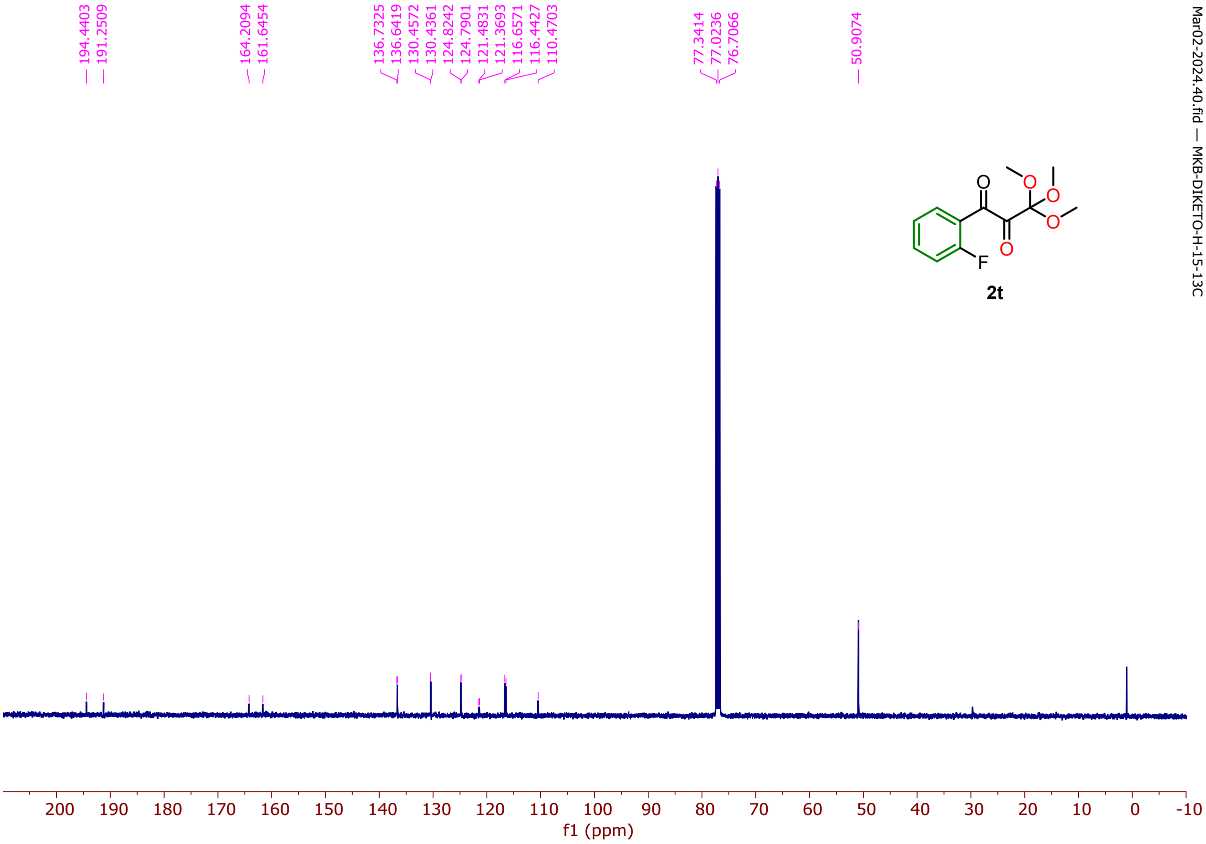






^{1}H NMR, ^{13}C NMR & ^{19}F NMR of 2t

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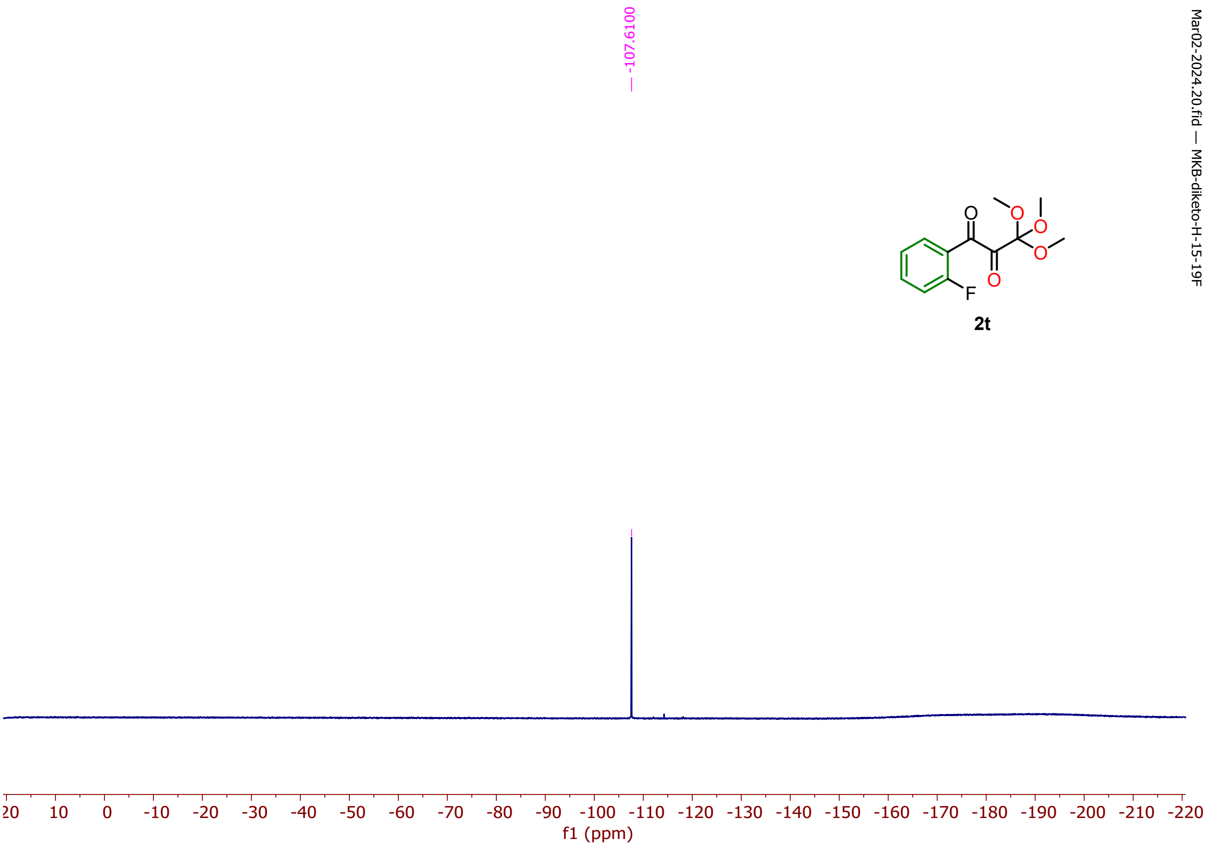
f1 (ppm)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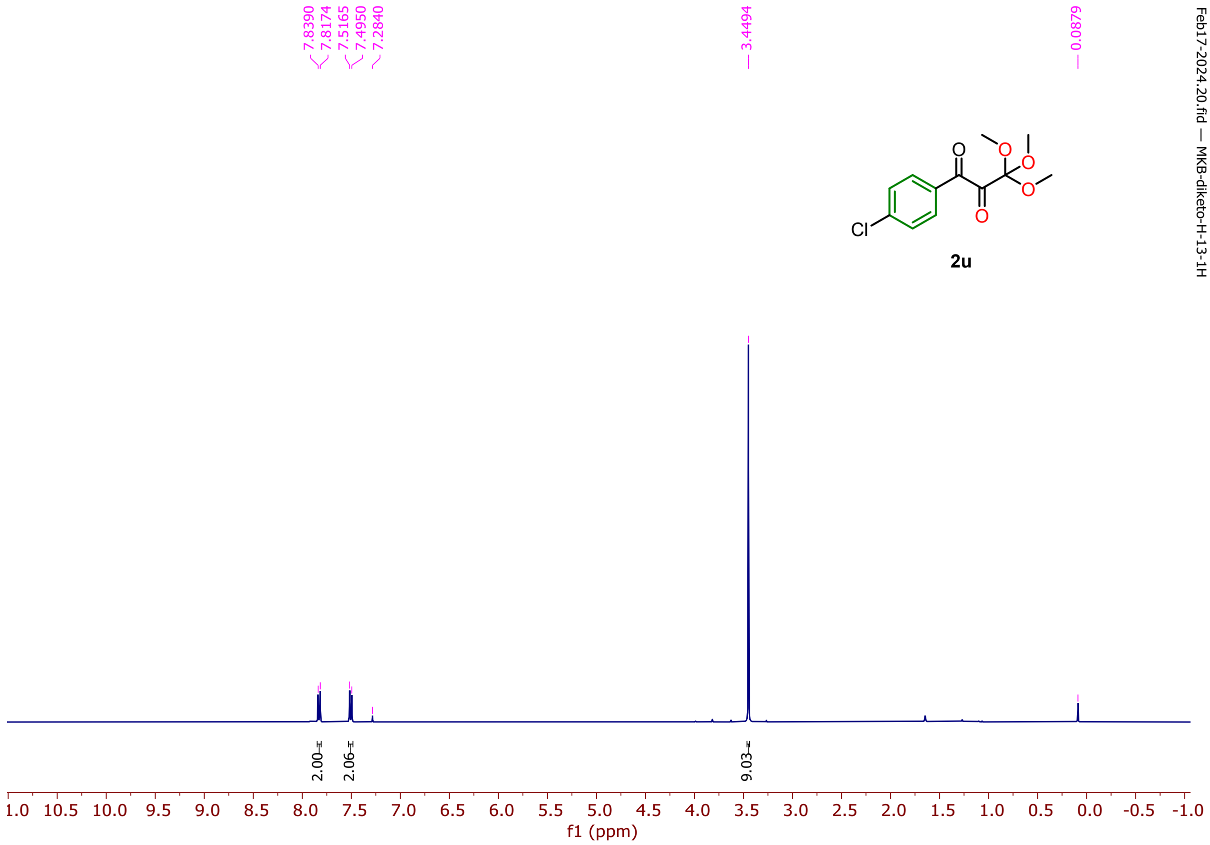
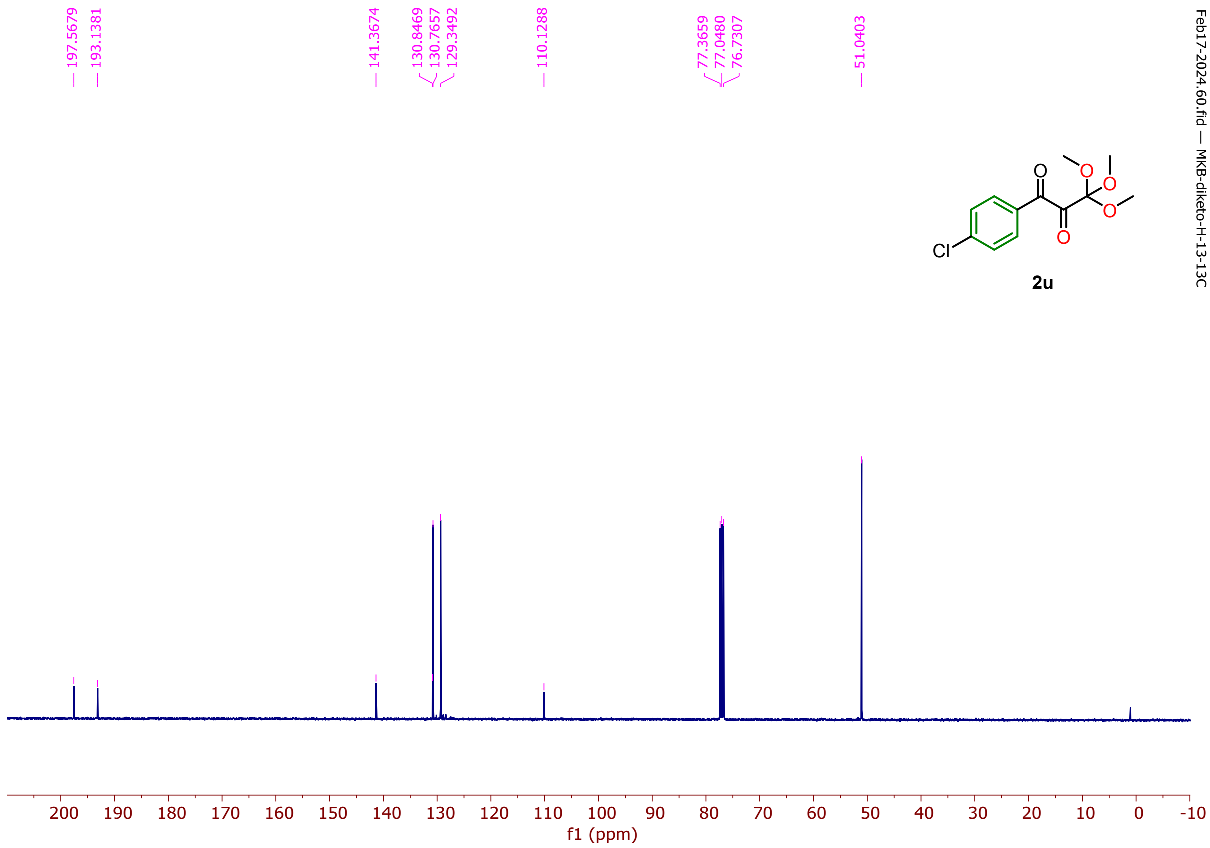
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

[1]H NMR & [13]C NMR of 2u





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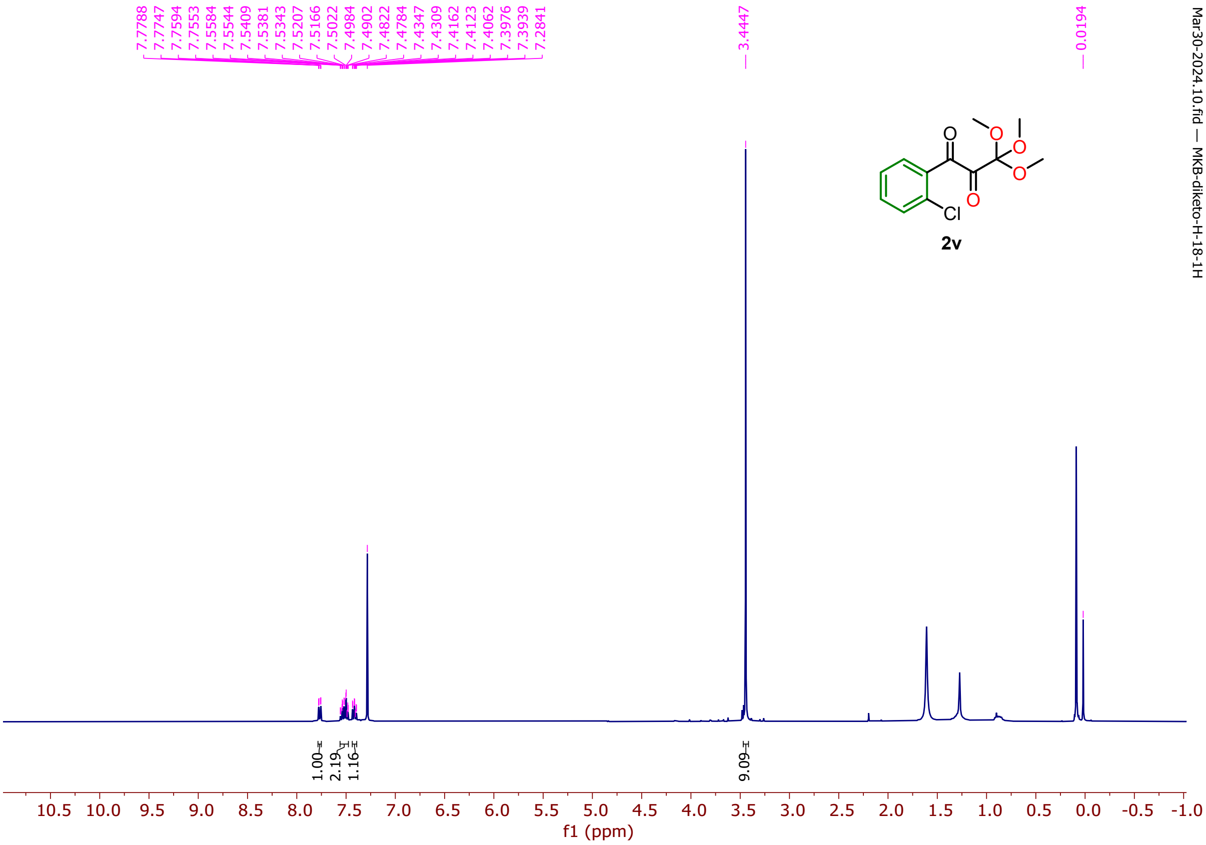
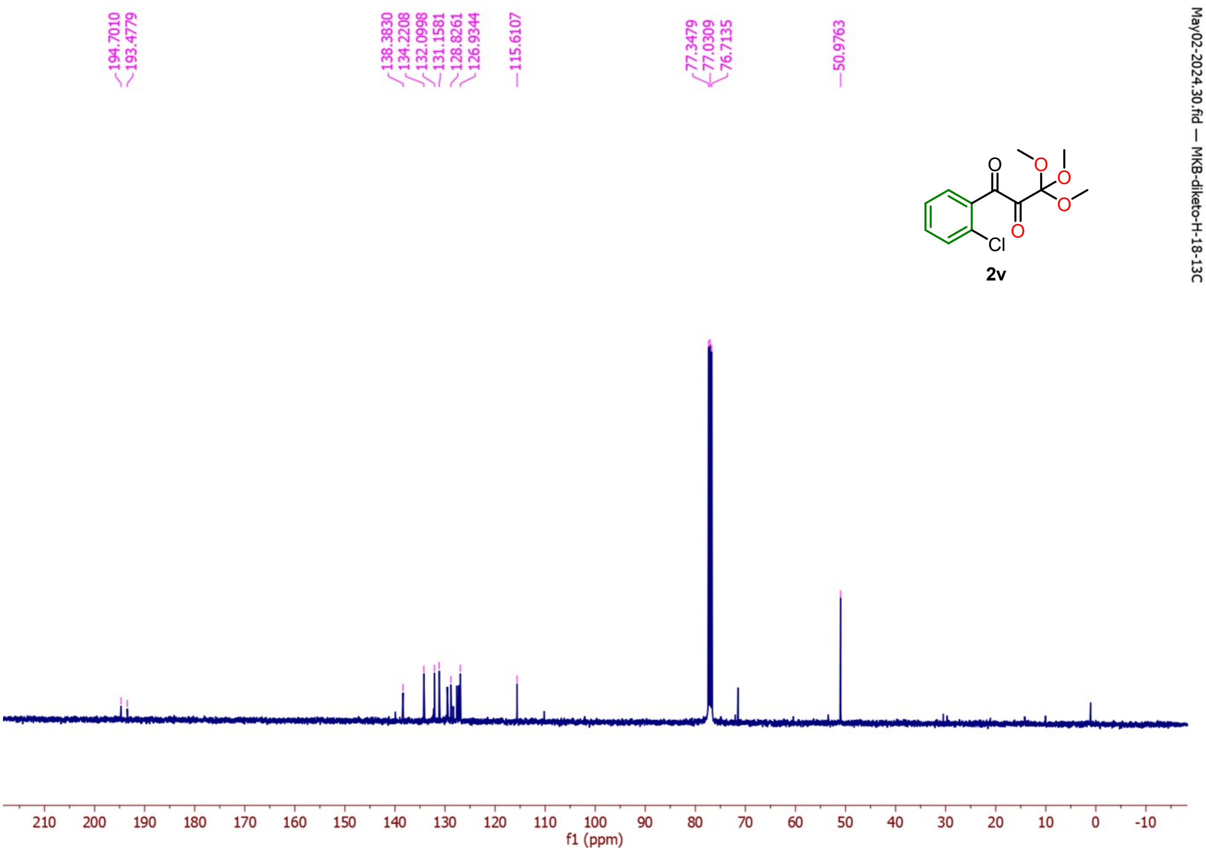
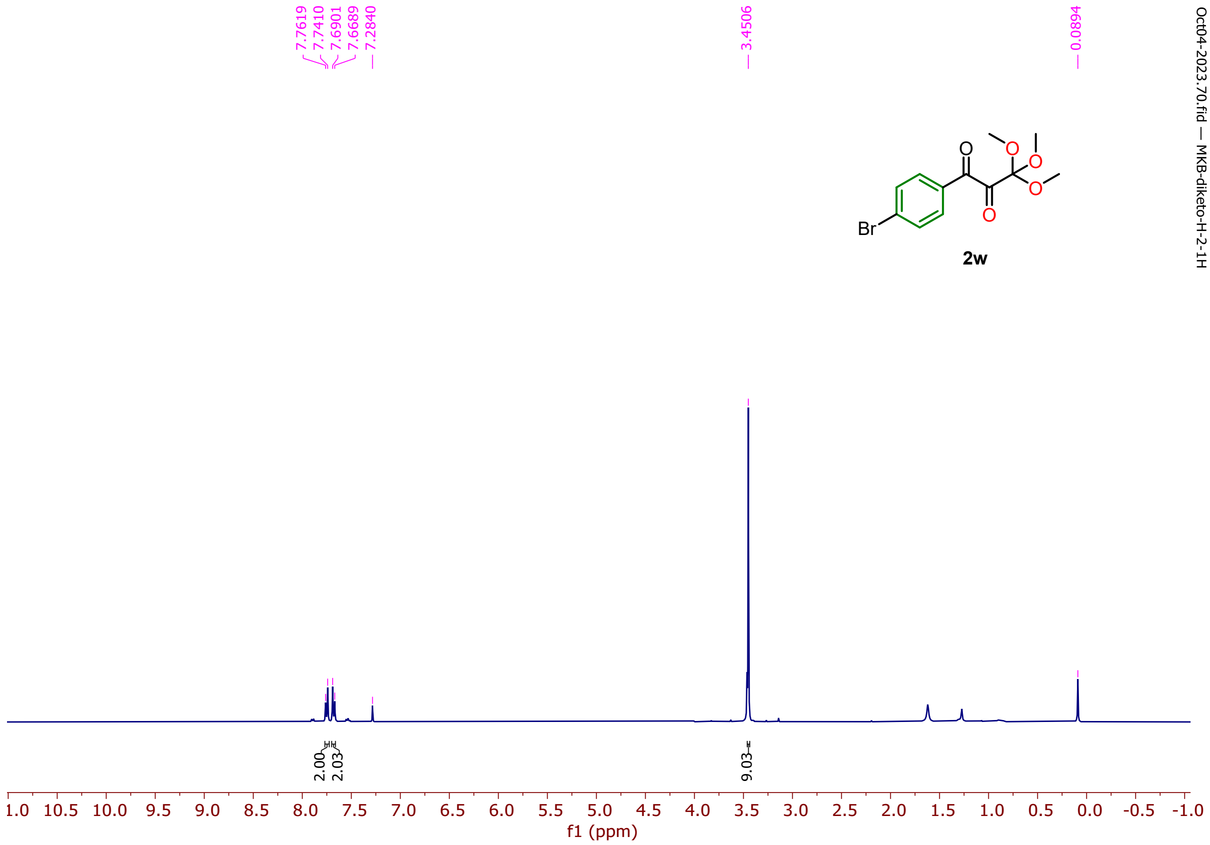
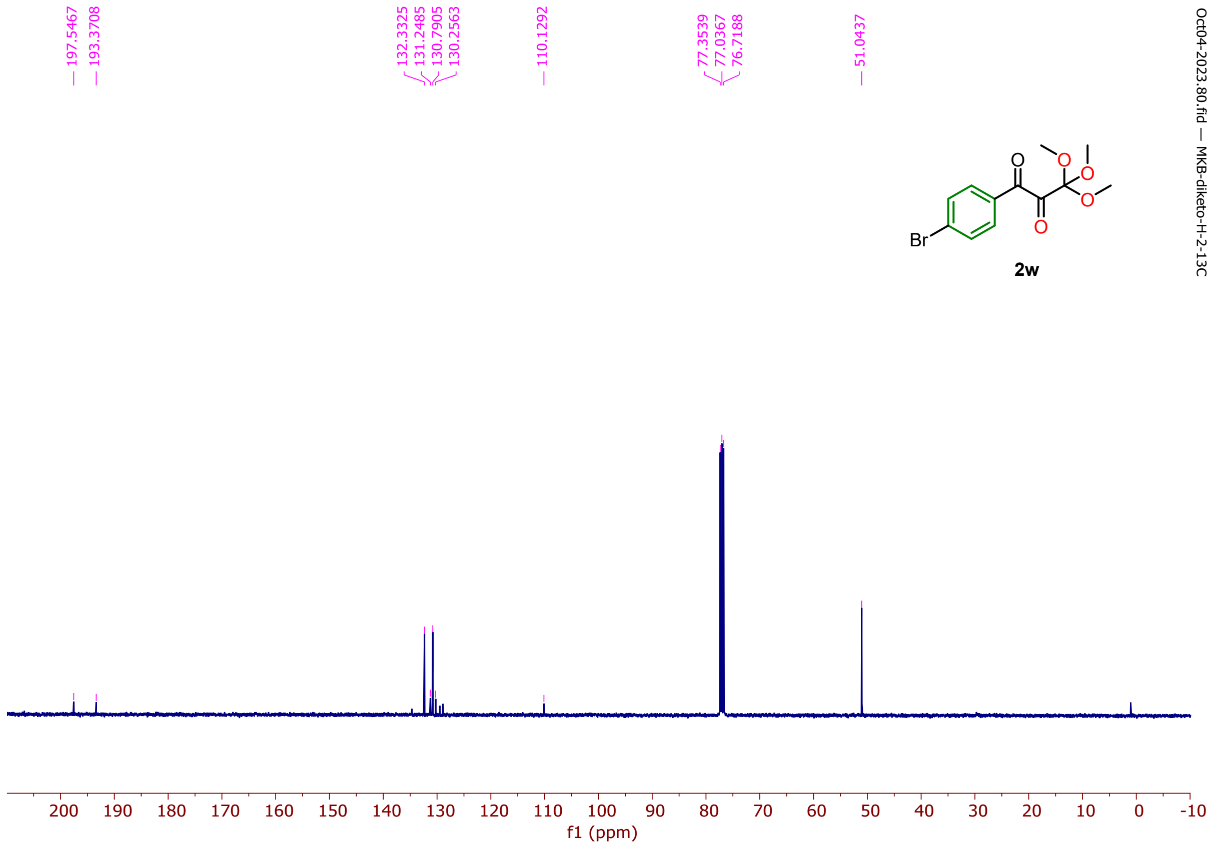


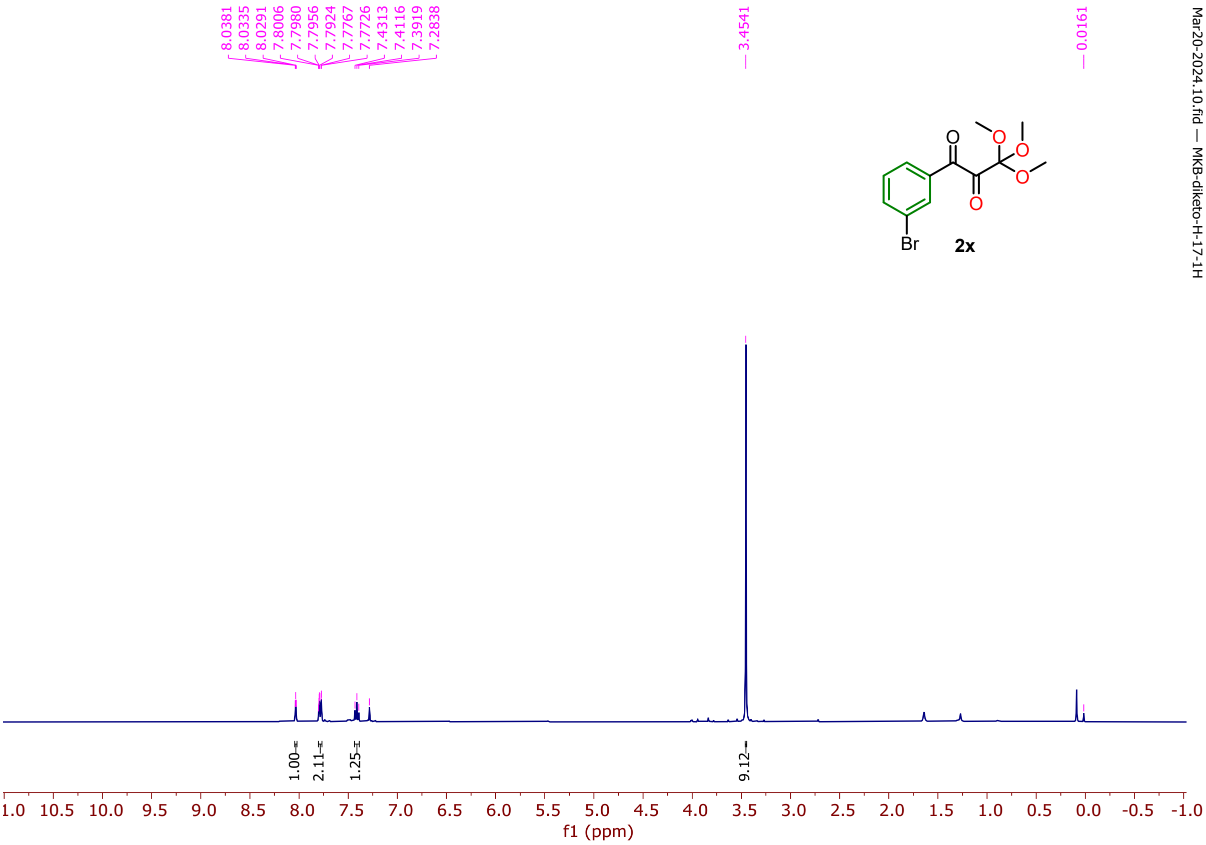


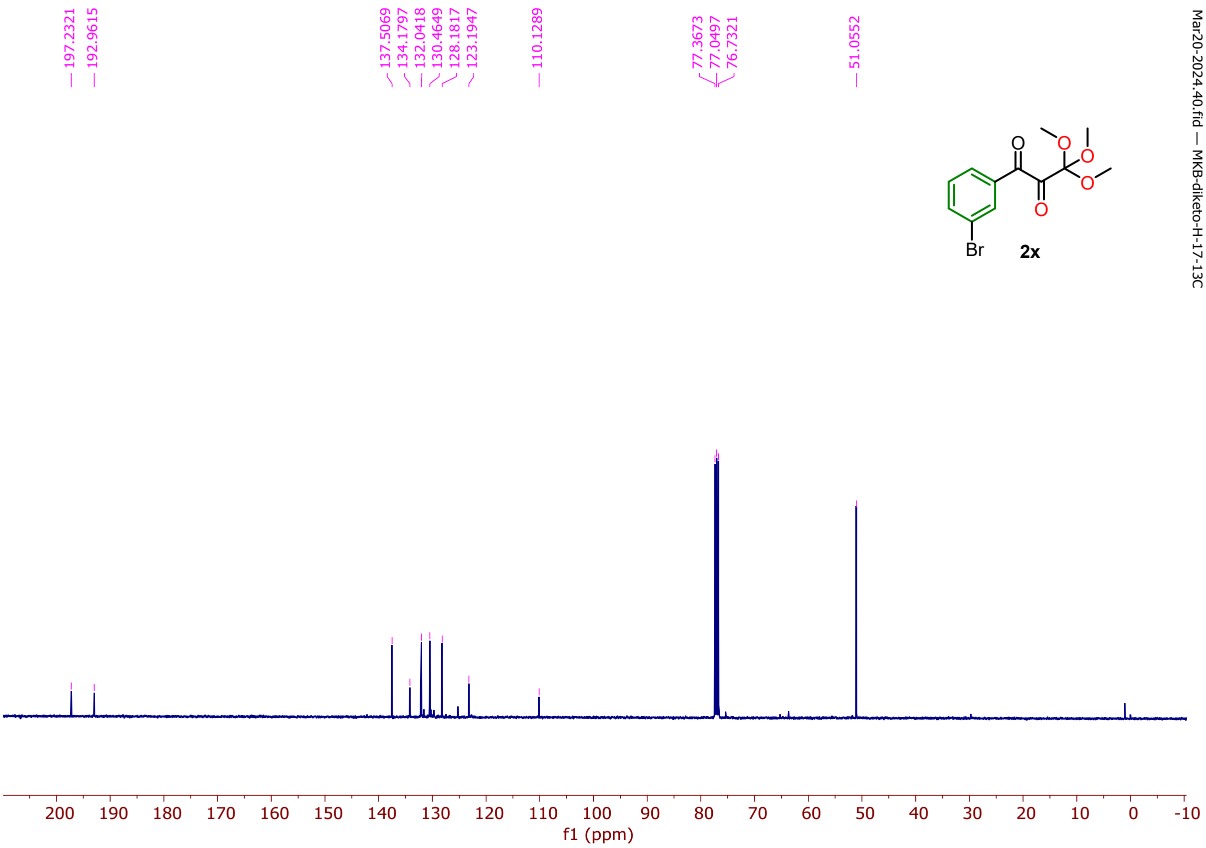
^{1}H NMR & ^{13}C NMR of 2w





[1]H NMR & [13]C NMR of 2x





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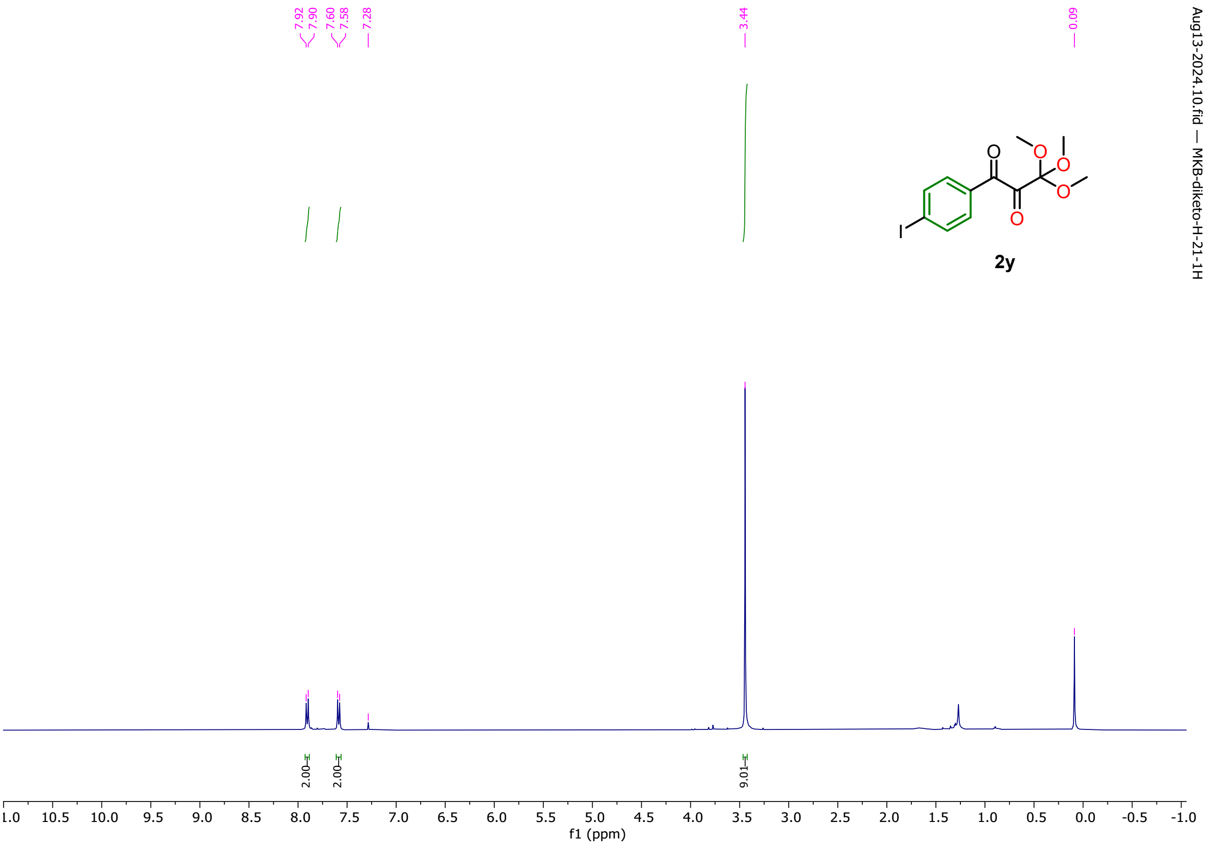
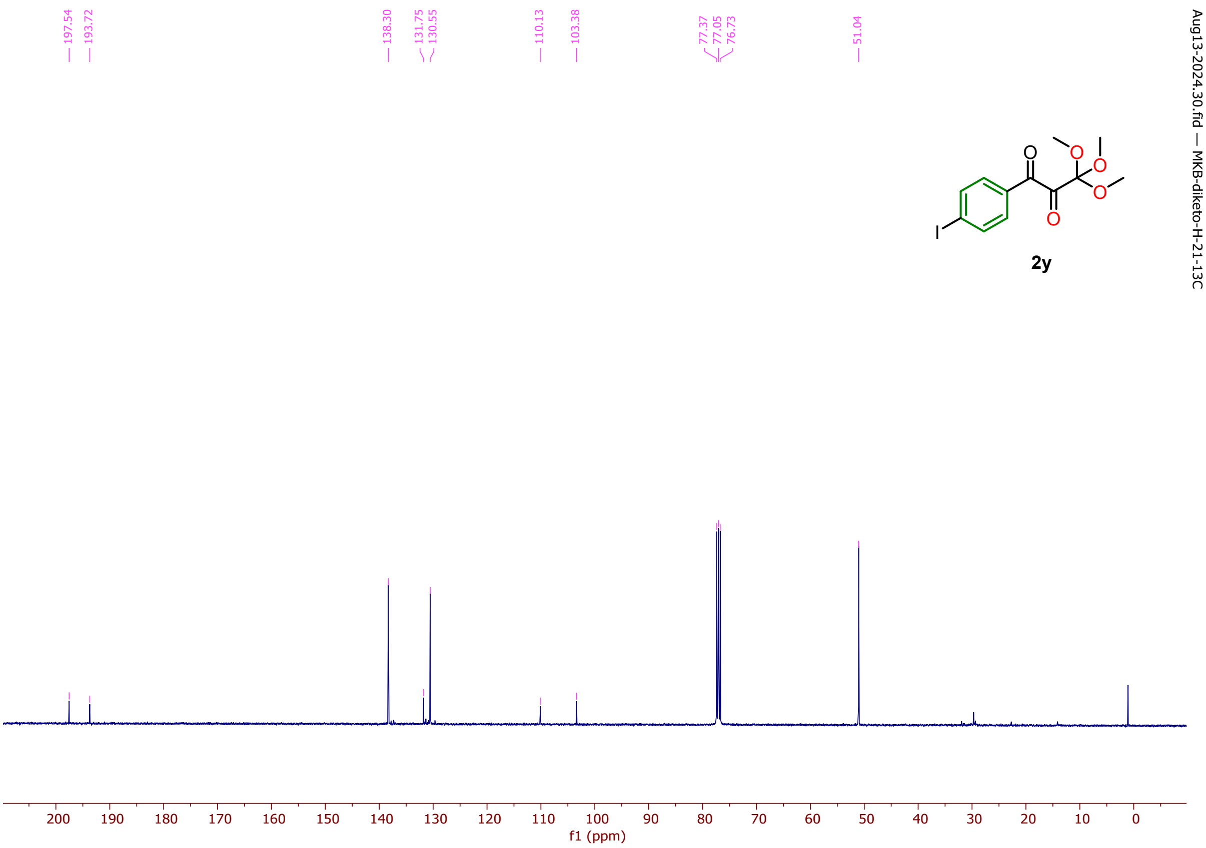
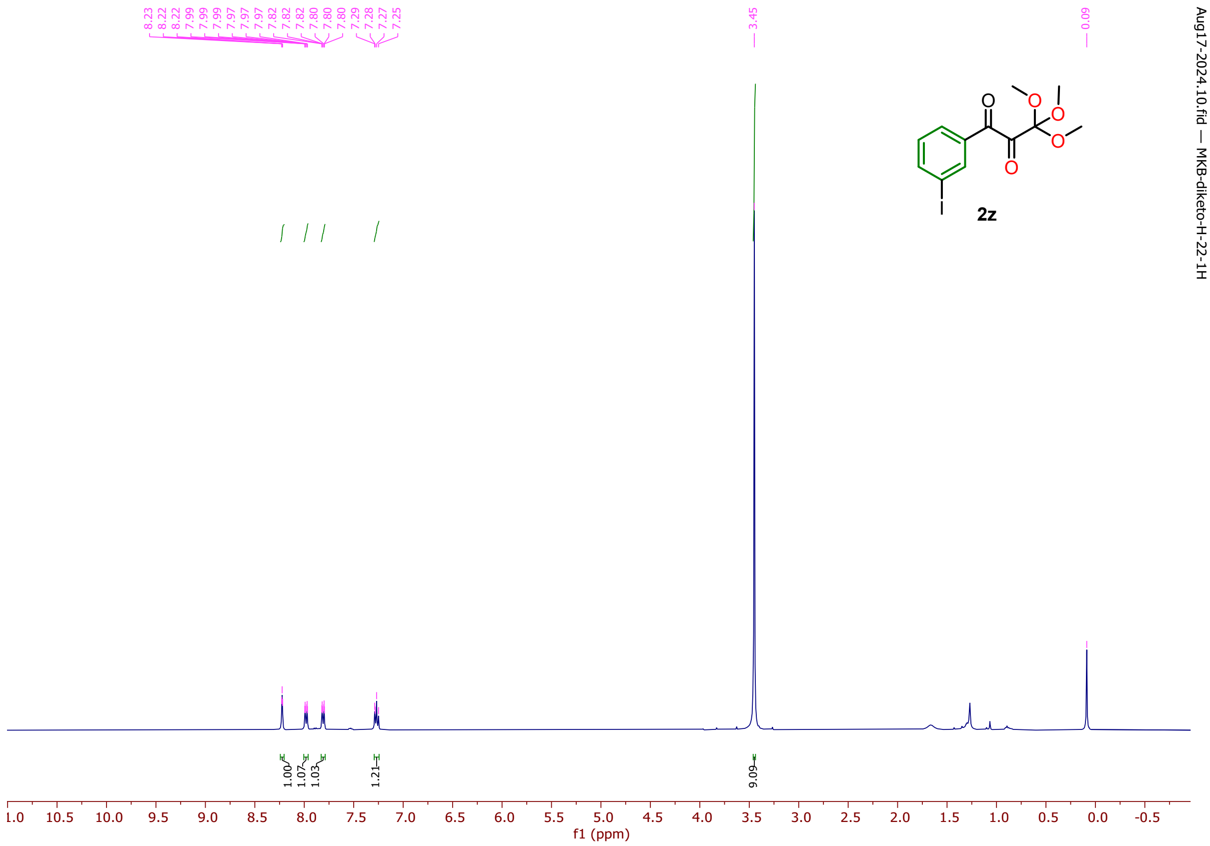
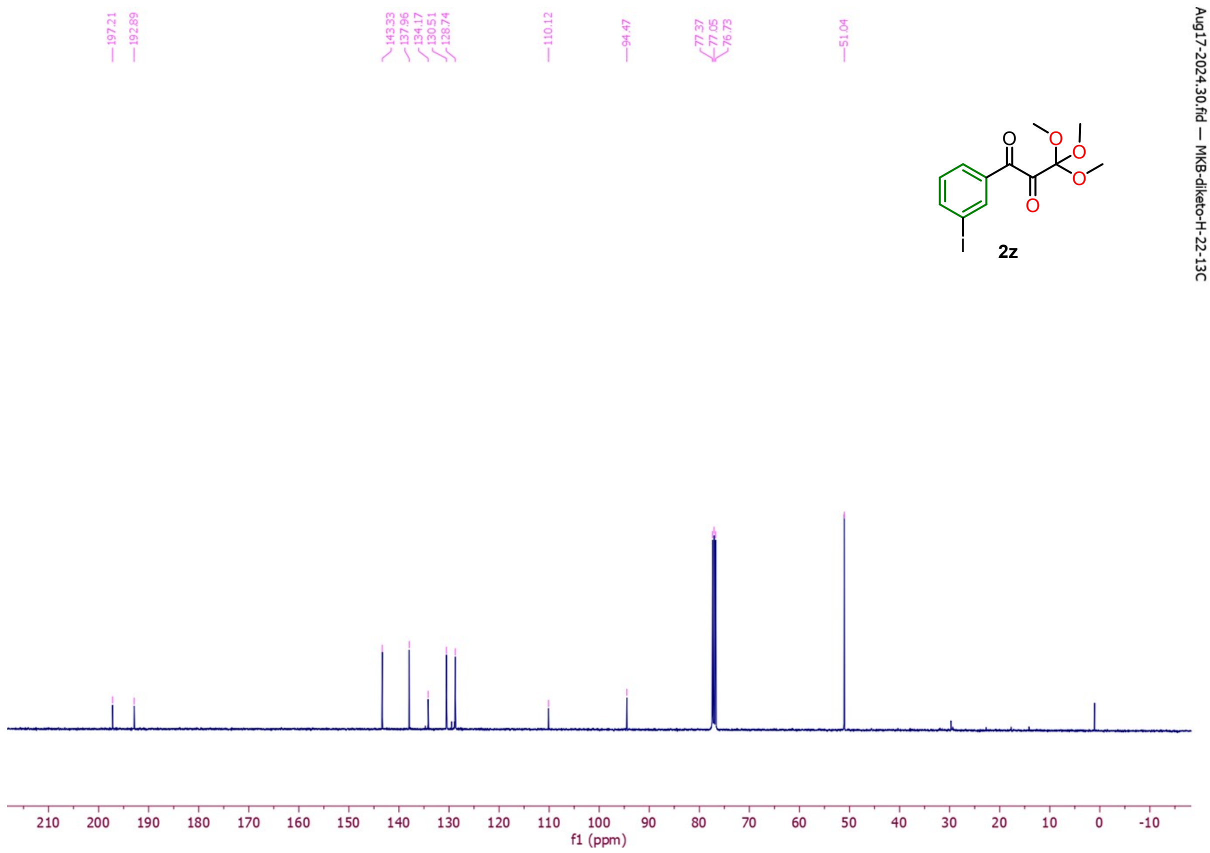


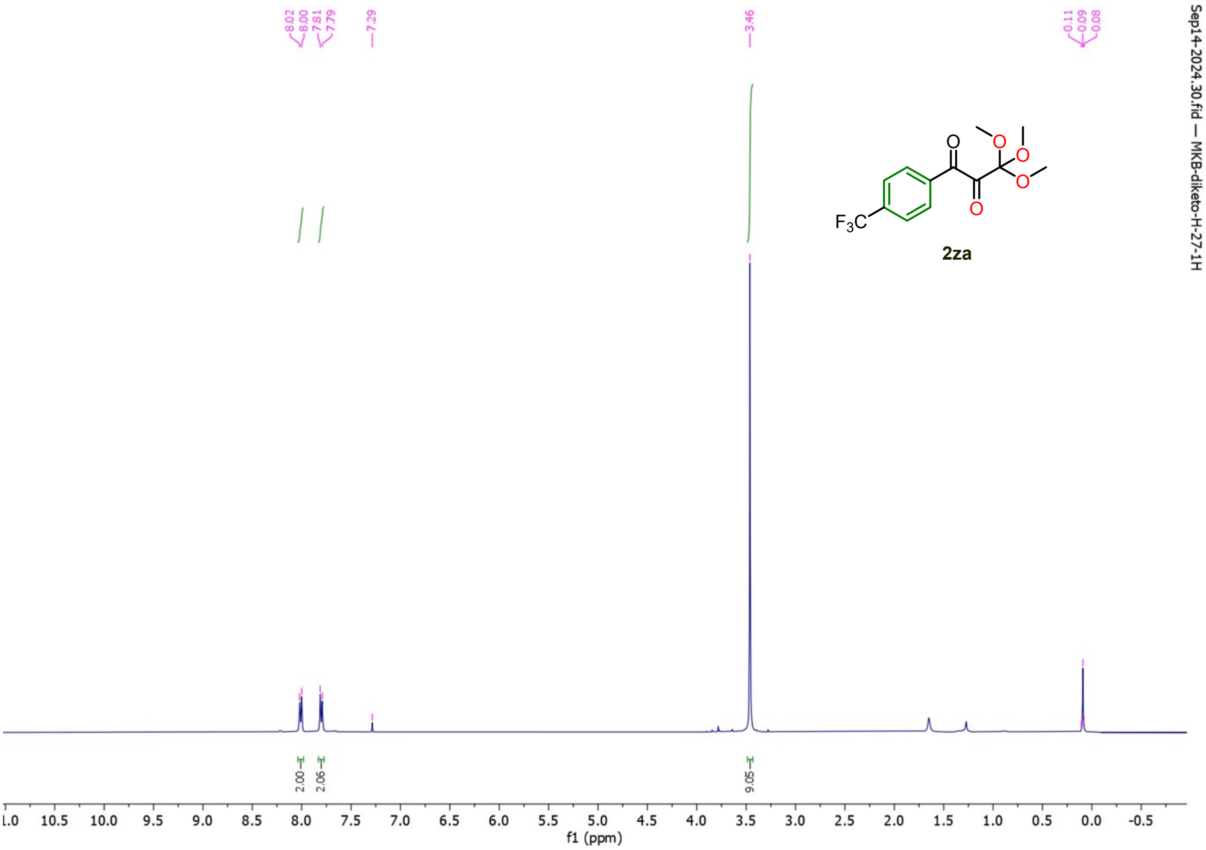


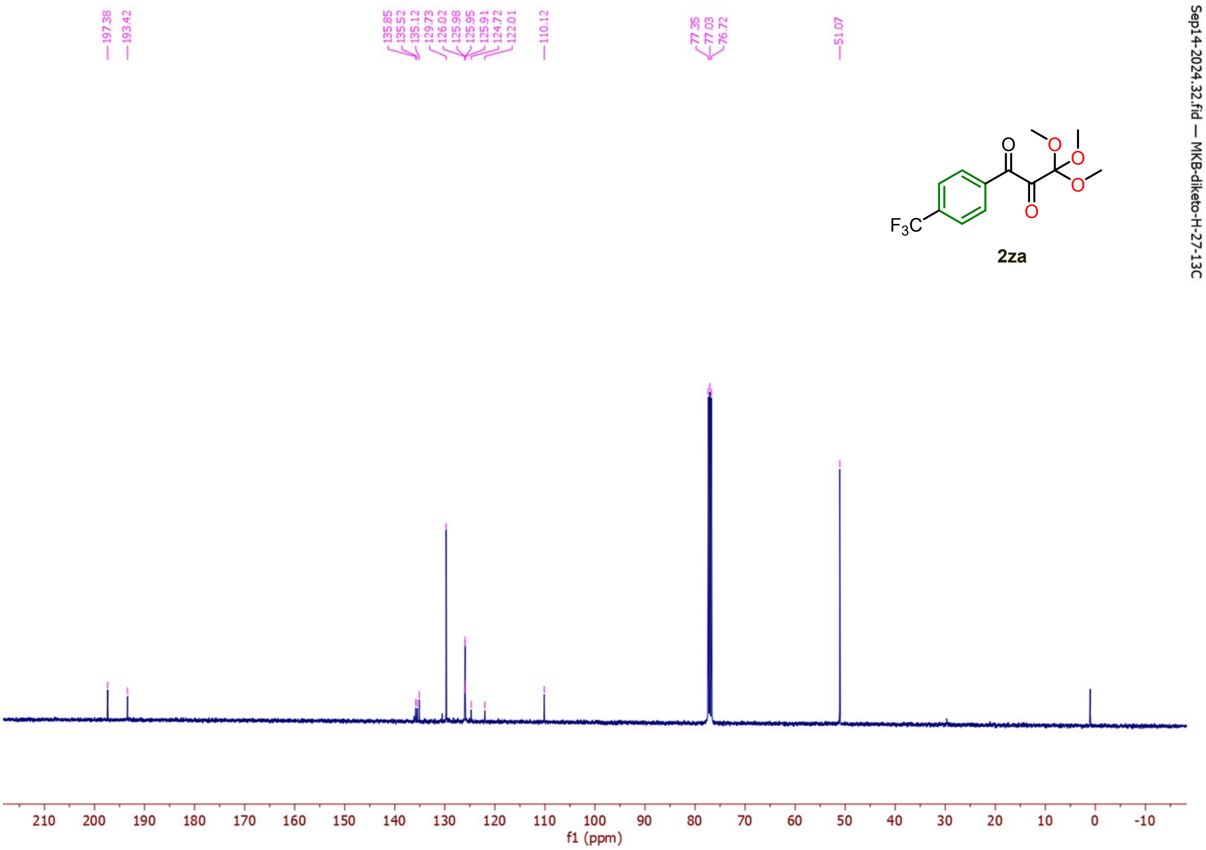
[1]H NMR & [13]C NMR of 2z

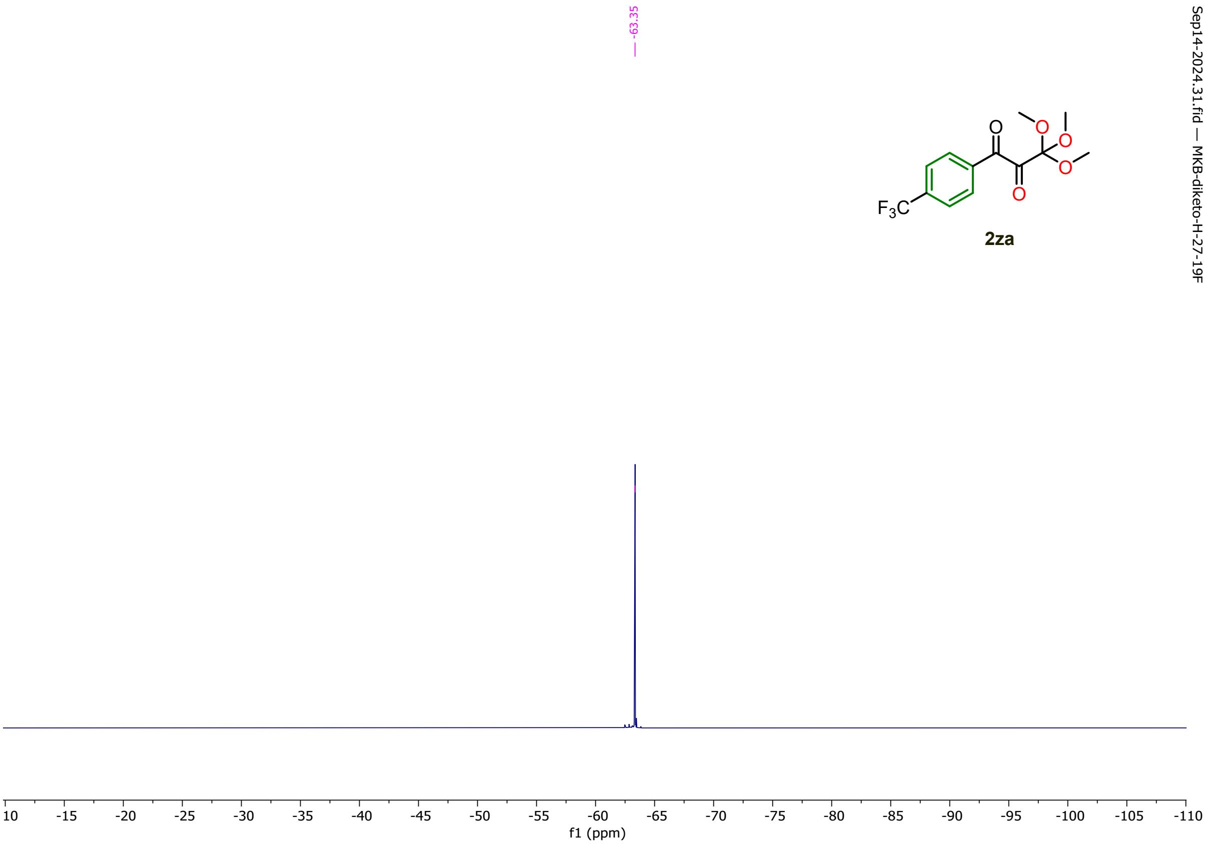




^{1}H NMR, ^{13}C NMR & ^{19}F NMR of 2za







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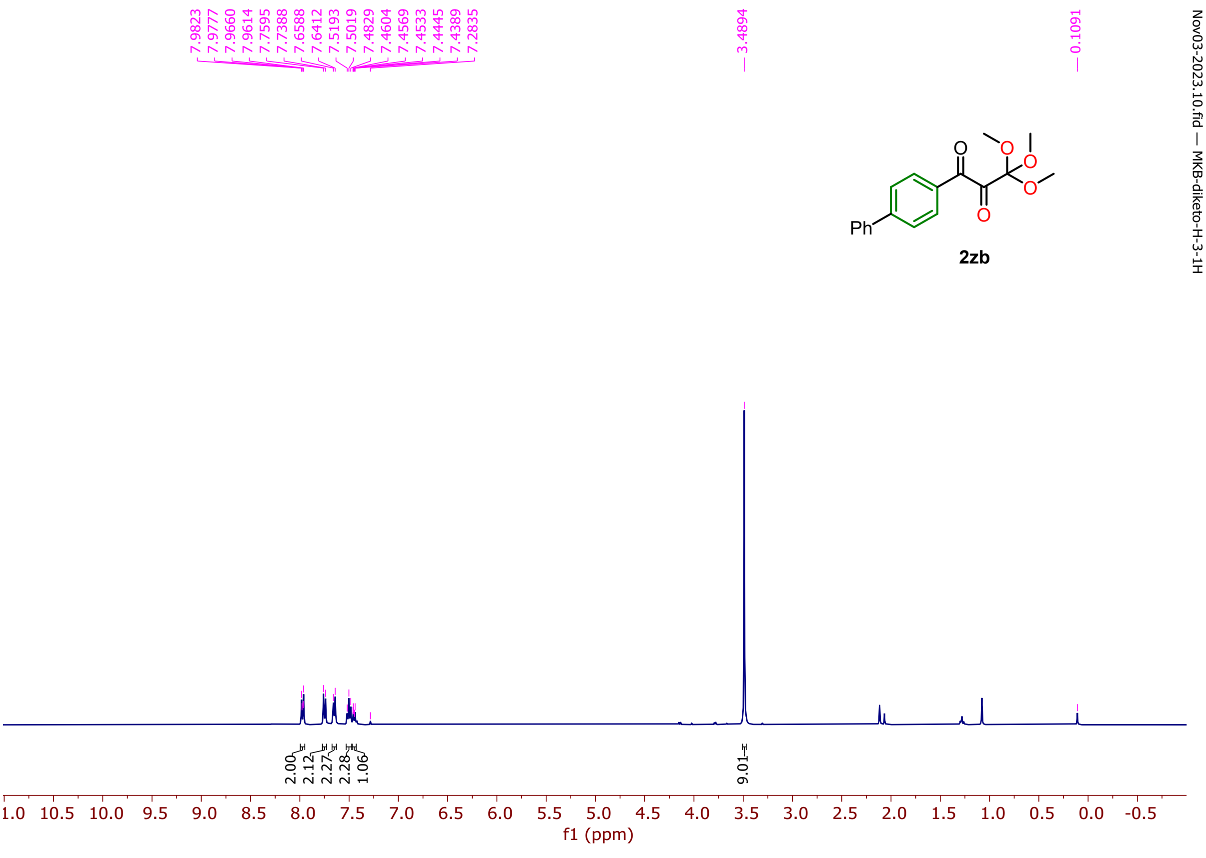

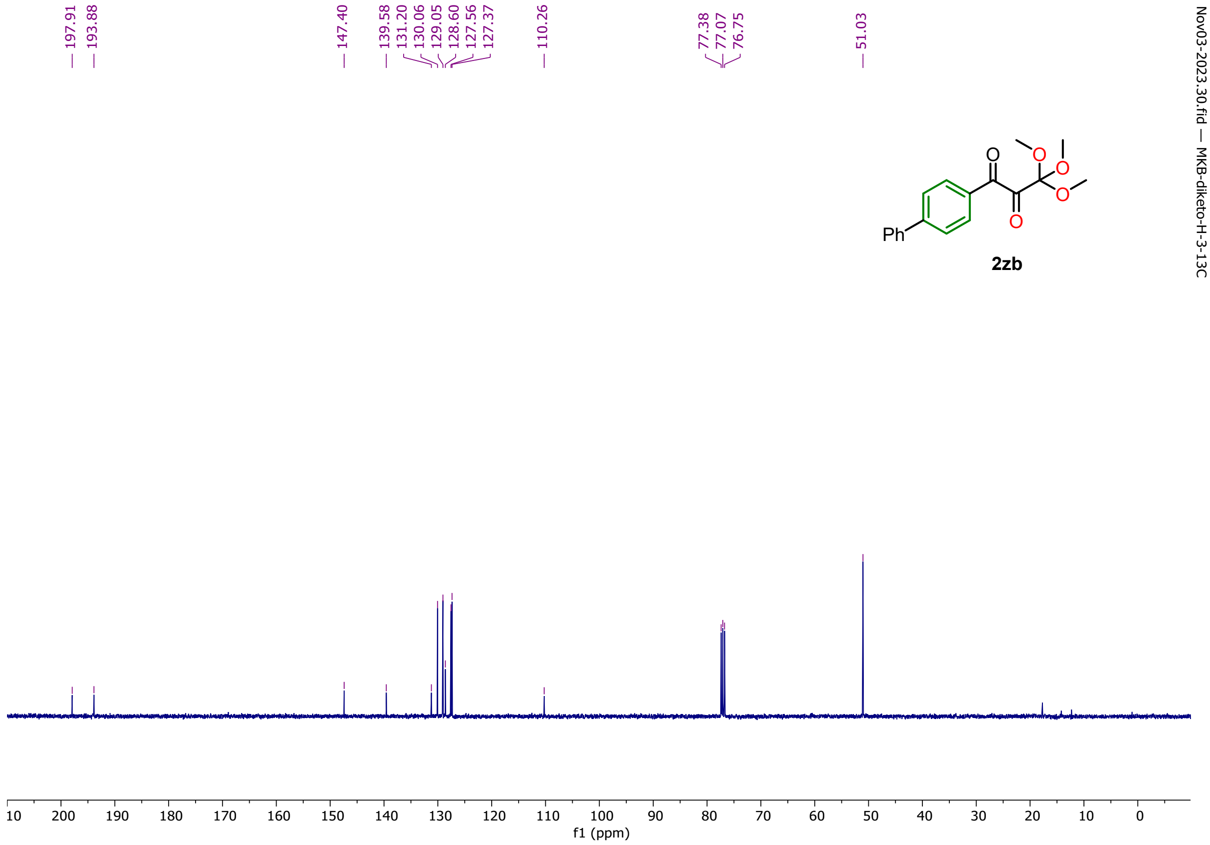

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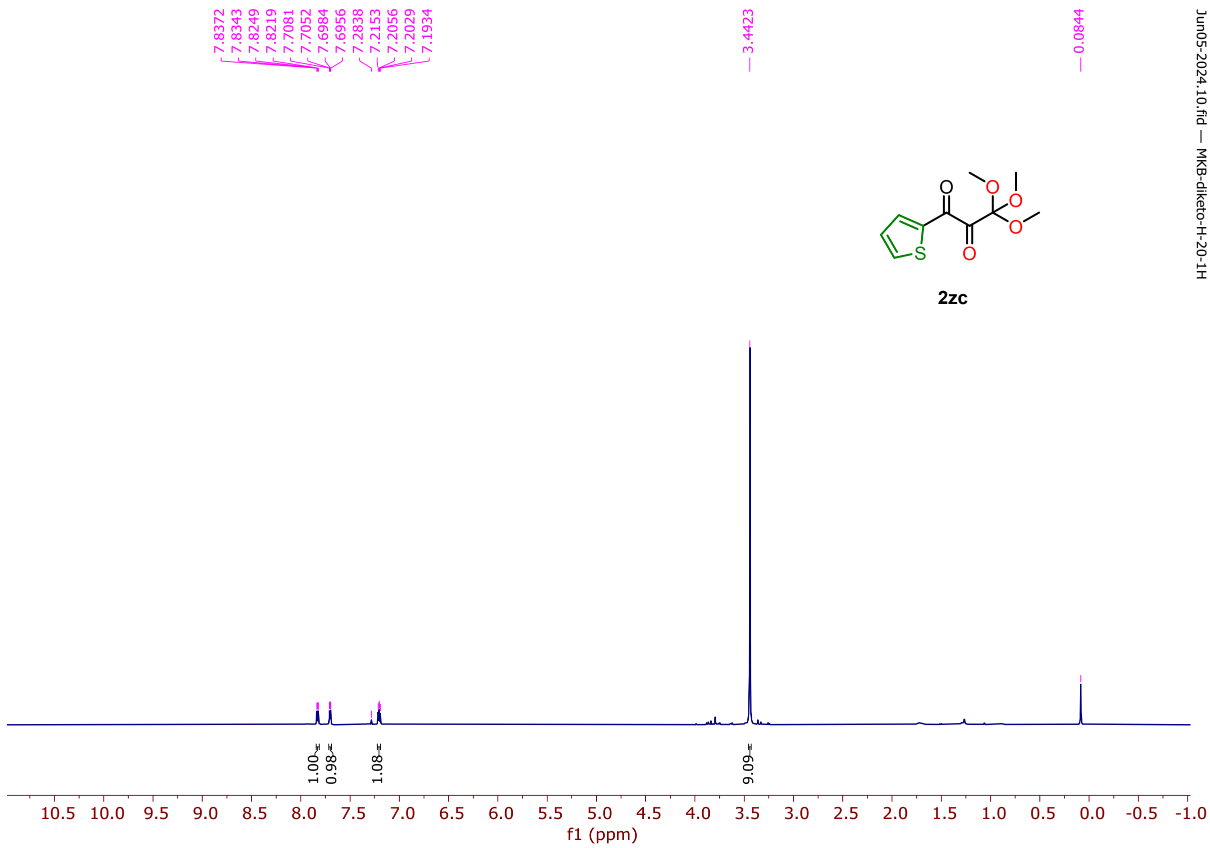
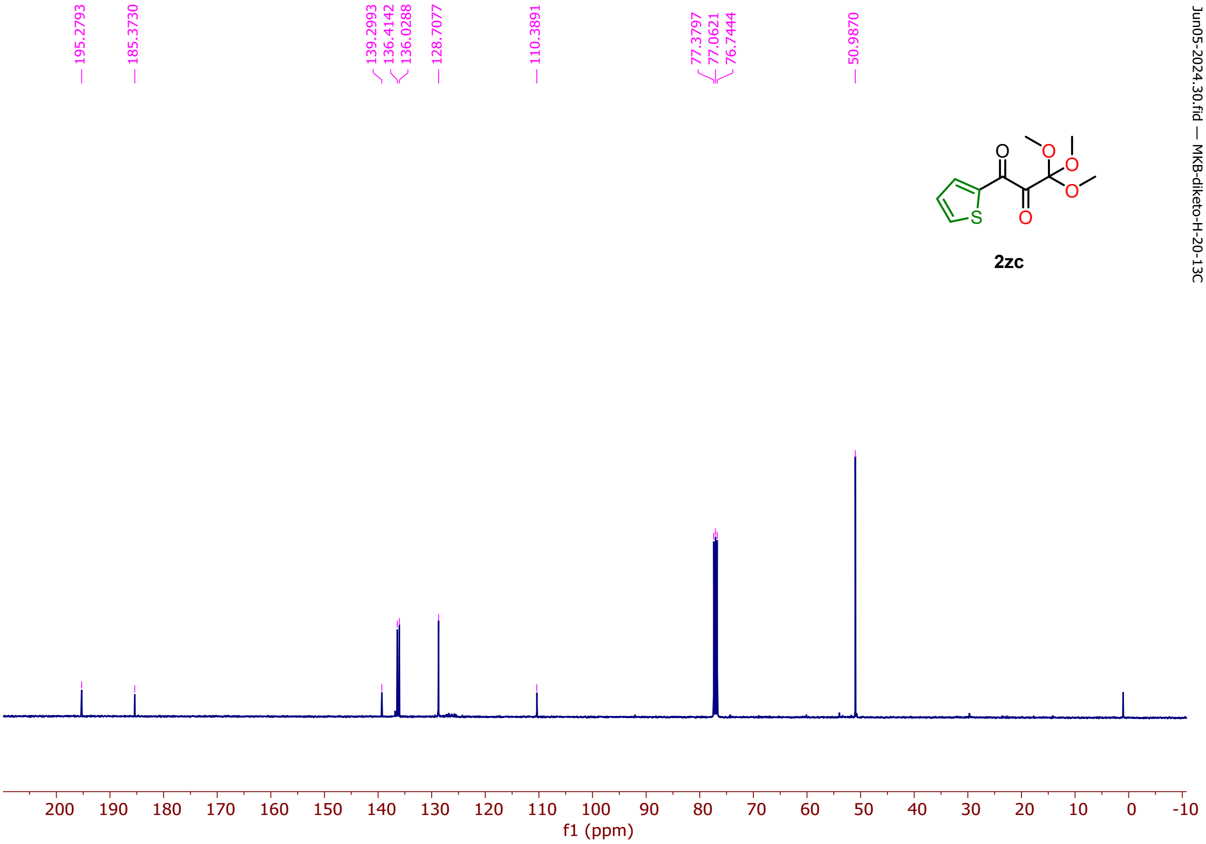
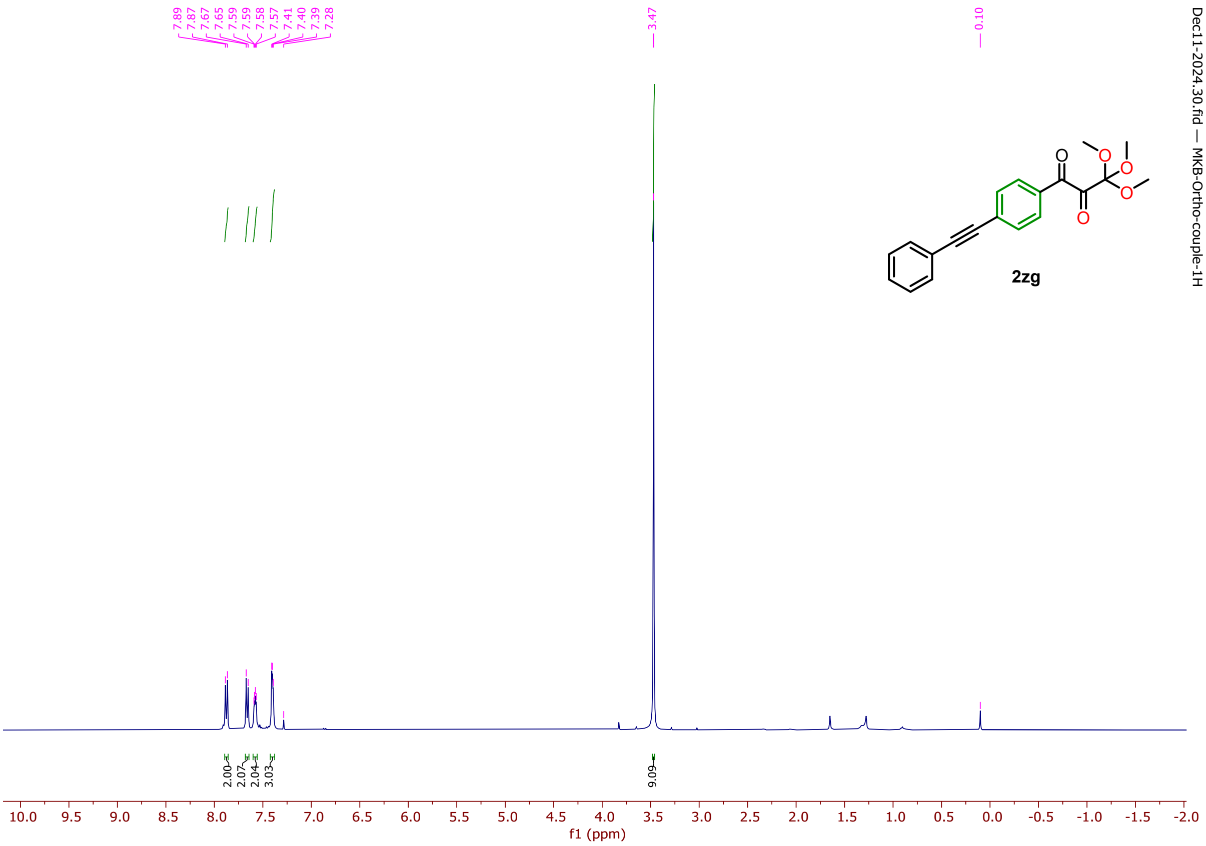
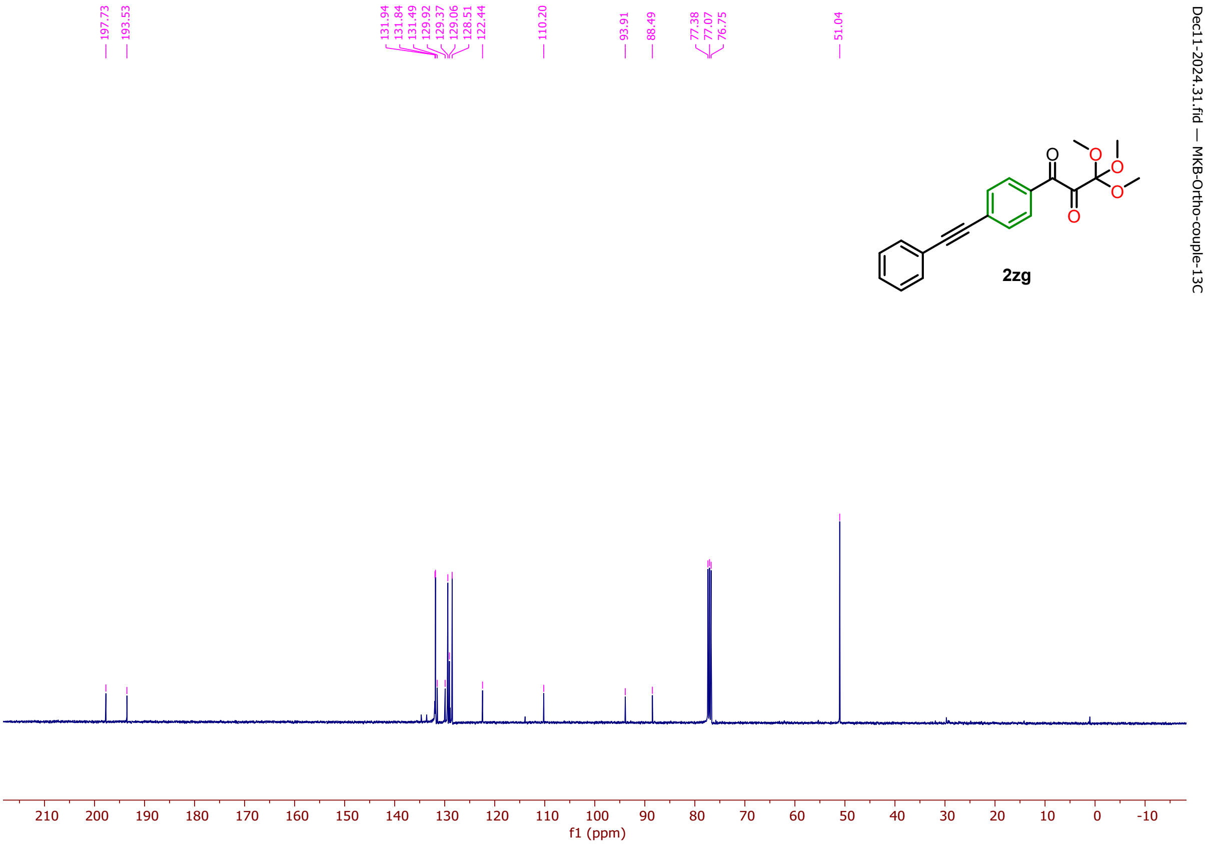




^{1}H NMR & ^{13}C NMR of 2zg

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f1 (ppm)