Electronic Supplementary Material (ESI) for Chemical Science.

Supporting Information

Light-Enabled Metal-Free Pinacol Coupling by Hydrazine

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Contents

1. General experimental information

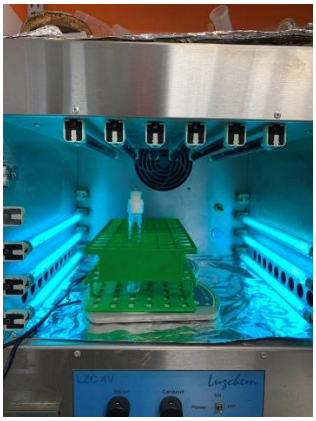
All reactions were carried out under an atmosphere of argon, unless otherwise stated. Solvents and reagents were purchased from Sigma-Aldrich chemical company and Fisher Scientific and were used without further purification unless otherwise specified. CH_3CN was dried over freshly activated 4 Å molecular sieves prior to use. 4 Å molecular sieves were purchased from Sigma-Aldrich chemical company and were freshly activated in the oven for 12 h at 380 ^oC prior to use. Product purifications were performed either with preparative chromatography on a Biotage Isolera One automated chromatography system on silica gel or with preparative analytical thin-layer chromatography (TLC) using E. Merck silica gel 60 F_{254} pre-coated plates (0.25 mm).

NMR Spectroscopy: Nuclear magnetic resonance (^1H, ^{13}C, ^{19}F) spectra were recorded on a Bruker AV500 equipped with a 60-position Sample Xpress sample changer (^1H, 500 MHz; ^{13}C, 125 MHz, ^{19}F 471 MHz). Chemical shifts are expressed in parts per million (ppm) units downfield from TMS, with the solvent residue peak as the chemical shift standard ($CDCl_3$: δ 7.26 ppm in ^1H NMR, δ 77.16 ppm in ^{13}C NMR; DMSO-d_6 δ 2.50 ppm in ^1H NMR, δ 39.52 ppm in ^{13}C NMR; acetone-d_6 δ 2.09 ppm in ^1H NMR, δ 205.87 ppm in ^{13}C NMR; CH_3CN δ 1.96 ppm in ^1H NMR, δ 118.26 ppm in ^{13}C NMR). Data are reported as following: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, td = triplet of doublets, q = quartet, quint = quintet, sext = sextet, sept = septet, m = multiplet, br = broad singlet), coupling constants *J* (Hz), and integration.

Mass Spectrometry: EI-MS was obtained from the Agilent GC-MS system. High Resolution Mass (HRMS) spectra was performed by the McGill Chemistry Department Mass Spectrometry Facility and was recorded using electrospray ionization (ESI+) and/or atmospheric pressure chemical ionization APCI(+/-), performed either on "Exactive Plus Orbitrap" a ThermoScientific high resolution accurate mass (HR/AM) FT mass spectrometer, or a Bruker Daltonics Maxis Impact quadrupole-time of flight (QTOF) mass spectrometer. Protonated molecular ions $(M+H)^+$, $(M-H)^-$ or sodium adducts $(M+Na)^+$, were used for empirical formula confirmation.

Caution*: hydrazine monohydrate is a toxic and volatile reagent, therefore appropriate personal protections should be performed running this transformation.*

2. General experimental procedure



General procedures: The 10 mL quartz tube, charged with a magnetic stir bar, was vacuumed and backfilled with argon for three times. Ketones (0.2 mmol, 1 equiv), N_2H_4•H_2O (0.15 mmol, 0.75 equiv, 7.4 µL) and CH_3CN (1 mL) were added into the tube sequentially under the protection of Argon. Then the tube was sealed and stirred at room temperature (ca. 25 ^oC) under UV irradiation (254 nm) by using a standard LZC-4V photoreactor from Luzchem company for 24 h. After completion, the reaction mixture was diluted with EtOAc and then concentrated *in vacuo*, and the resulting residue was purified by column chromatography on silica gel or preparative TLC (EtOAc/hexanes, *etc*.) to afford the corresponding products.

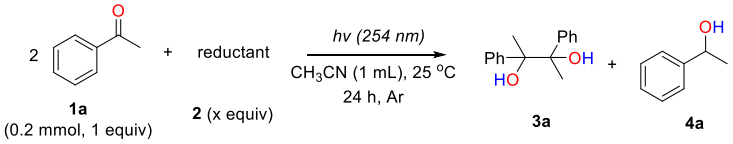
3. Procedure for gram scale reaction

Caution: gas was released during the reaction, so the oil bubbler was needed to release the in situ generated gases.

The 50 mL quartz round bottom flask, charged with a magnetic stir bar, was vacuumed and backfilled with argon for three times. Benzophenone (6 mmol, 1 equiv, 1.0933 g), N_2H_4•H_2O (4.5 mmol, 0.75 equiv, 222 µL) and CH_3CN (30 mL) were added into flask sequentially under the protection of Argon. Then the flask was sealed and connected to oil bubbler to release the gas generated during the reaction and stirred at room temperature (ca. 25 ^oC) under UV irradiation (254 nm) by using a standard LZC-4V photoreactor from Luzchem company for 72 h. After completion, the reaction mixture was diluted with EtOAc and then concentrated *in vacuo*, and the resulting residue was purified by column chromatography on silica gel (eleuent: hexanes: EtOAc = 10:1) to afford the Benzopinacol product (0.9845g, 89% yield).

4. Reaction optimization

Table S1. Optimization of the reaction conditions[a]



1a (0.2 mmol, 1 equiv) **2** (x equiv) **3a** **4a**

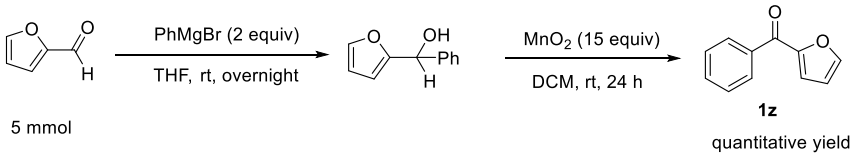
entry	N_2H_4 derivatives (x equiv)	conv. (%)	yield (%) **3a**	**4a**
1	N_2H_4 (0.75 equiv)	98	80	4
2	N_2H_4 (1 equiv)	99	81	4
3	BocNH-NHBoc (1 equiv.)	-	-	-
4	BocNH-NHTf (1 equiv.)	-	-	-
5	PhNH-NHPh (1 equiv.)	-	-	-
6[b]	MeNH-NHMe (1 equiv.)	42	27	2
7	*i*PrOH (0.75 equiv)	42	32	1

[a] Reaction conditions: acetophenone (0.2 mmol, 1 equiv), reducants (x equiv), in CH_3CN (1 mL) were stirred under argon for 24 h under UV light (254 nm) irradiation at 25 °C; starting material conversion and NMR yields were given with 1,3,5-trimethoxybenzene as the internal standard, yields calculated based on acetophenone; [b] the MeNH-NHMe was generated in situ by deprotonation of 1 equiv MeNH-NHMe•2HCl with 2 equiv K_3PO_4.

5. General synthetic methods of heteroaromatic ketones

The general procedures are demonstrated by the synthesis of furan-2-yl(phenyl)methanone, and other heteroaromatic ketones are synthesized in similar methods.

5.1 Synthesis of furan-2-yl(phenyl)methanone (**1z**):



5 mmol

1z

quantitative yield

The methods used were according to the previous report literature.[1]

To a 50 mL round-bottom flask charged with a magnetic stirring bar, furfural (5mmol, 414.2 µL) and 10 mL dry THF were charged. The phenyl magnesium bromide (10.5 mmol) (3.5 mL, 3 M solution in Et_2O) was added dropwise and the mixture was continued to stir at room temperature overnight. After completion of reaction (detected by TLC and GC-MS), the mixture was quenched by adding sat. NH_4Cl solution. The product was extracted with EtOAc (25 mL x 3) and the combined extracts were dried over Na_2SO_4, filtered, and concentrated in vacuum. The crude product (furan-2-yl(phenyl)methanol) obtained was used without further purification into the next MnO_2 oxidation step.

To a 50 mL round-bottom flask charged with a magnetic stirring bar, the crude product (furan-2-yl(phenyl)methanol) obtained in previous step,10 mL DCM and MnO_2 (6.5 g, 75 mmol, 15 equiv) were charged. After completion of reaction (around 24 h, detected by TLC and GC-MS), the mixture was filtered by a pad of celite and concentrated in vacuum. After column chromatography on silica gel (eluent: hexanes: EtOAc = 10:1), the furan-2-yl(phenyl)methanone (**1z**) was isolated as a yellow oil in quantitative yield.

6. Examine the origin of proton

The 10 mL quartz tube, charged with a magnetic stir bar, was vacuumed and backfilled with argon for three times. Benzophenone (0.2 mmol, 1 equiv), N_2H_4•H_2O (0.15 mmol, 0.75 equiv, 7.4 μL) and CD_3CN (1 mL) were added into the tube sequentially under the protection of Argon. Then the tube was sealed and stirred at room temperature (ca. 25 ^oC) under UV irradiation (254 nm) by using a standard LZC-4V photoreactor from Luzchem company for 24 h. After completion, the resulting residue was diluted by EtOAc and then the solvents were removed under vacuum. Next, the sample was directly dissolved in the $CDCl_3$ and was transferred to NMR tube under the protection of argon to test the ^1H NMR and ^2D NMR. The spectrums were shown in Figure 1a in the manuscript.

7. Procedure for fluorescence quenching studies between benzophenone and hydrazine

The procedure followed previous report.[2] The UV-Vis spectrum was performed on Agilent Cary 5000 series UV-Vis-NIR spectrometer under the conditions as below.

A stock solution of benzophenone (10 mM in HPLC grade CH_3CN) was prepared in a volumetric flask and diluted by HPLC grade CH_3CN to 0.10 mM for the UV-Vis experiment. A quartz cuvette (1 cm x 1 cm x 3 cm) was filled with the above-mentioned 0.10 mM benzophenone solution and its spectrum was recorded from 220 nm to 600 nm in the spectrometer and UV-Vis spectrum (Fig. S1).

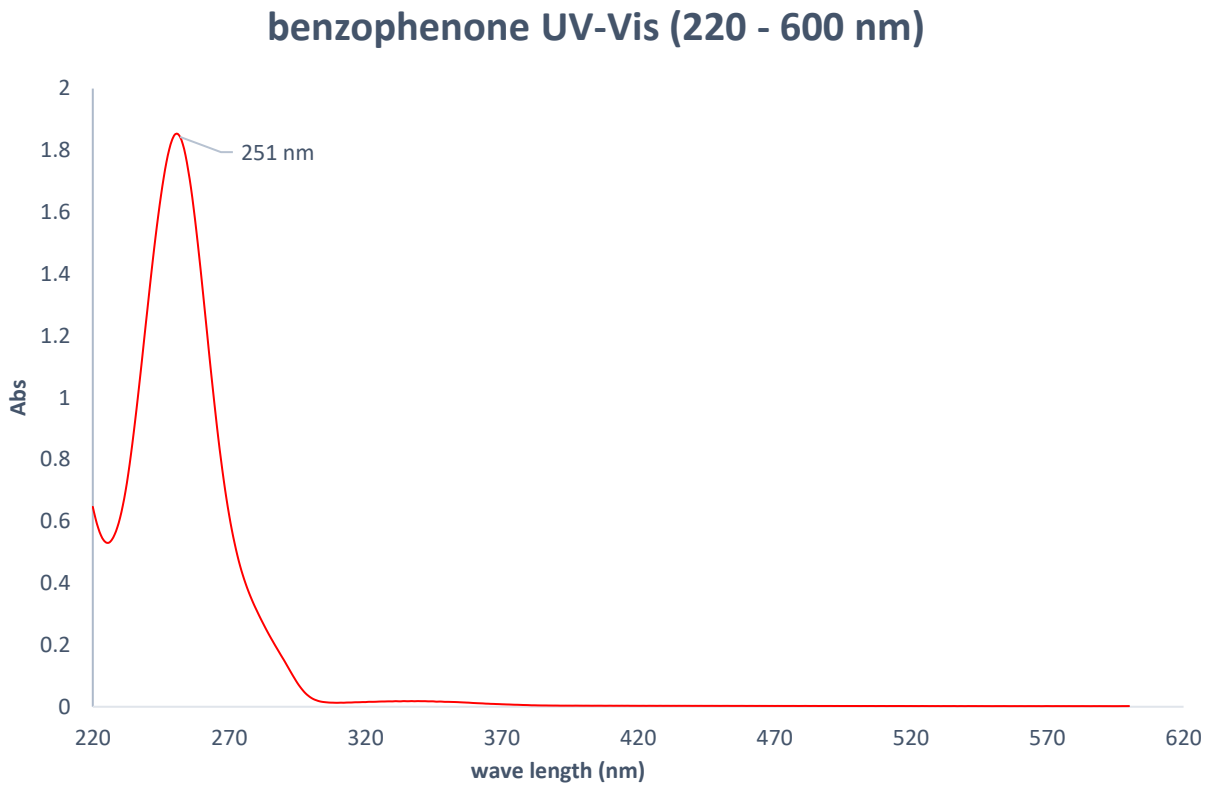


Fig. S1 Benzophenone UV-Vis spectrum (220 – 600 nm, 0.1 mM).

The quenching experiment for the fluorescence of benzophenone by hydrazine was performed on VARIAN CARY Eclipse fluorescence spectrophotometer.

A stock solution of benzophenone (100 µM, in HPLC grade CH_3CN) was prepared in a volumetric flask and diluted by HPLC grade CH_3CN to 1 µM for the quenching experiment. A quartz cuvette (1 cm × 1 cm × 3 cm) was filled with the abovementioned 1 µM benzophenone solution and its fluorescence (maximum emission at 507 nm) was recorded with excitation at 254 nm in the spectrometer. Quenching experiments were performed under duplicate conditions with the injection of 5 µL, 10 µL, 15 µL, 20 µL, 25

μL and 30 μL hydrazine monohydrate, respectively by auto-pipette. The results were averaged by three parallel experiments and shown in Fig. S2 and Fig. S3.

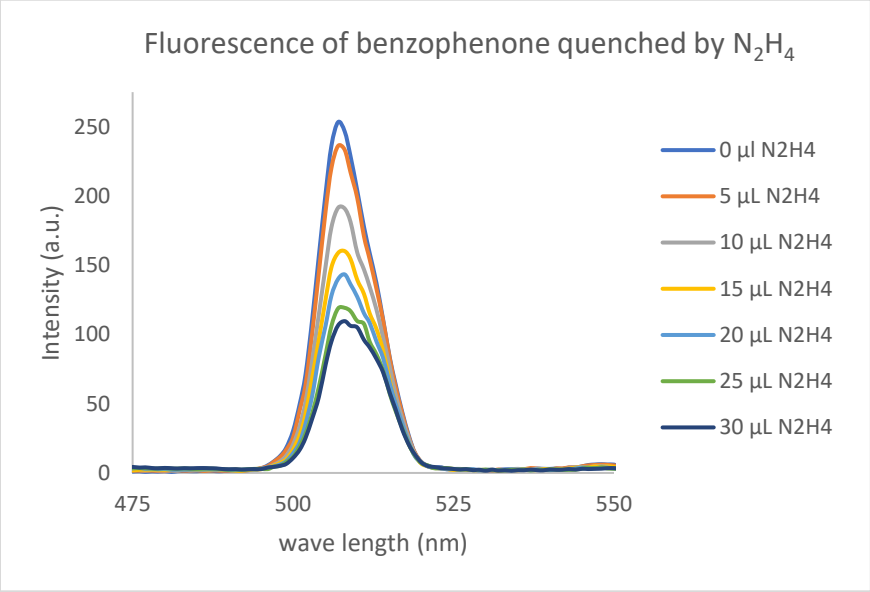


Fig. S2 Fluorescence of benzophenone quenched by N_2H_4.

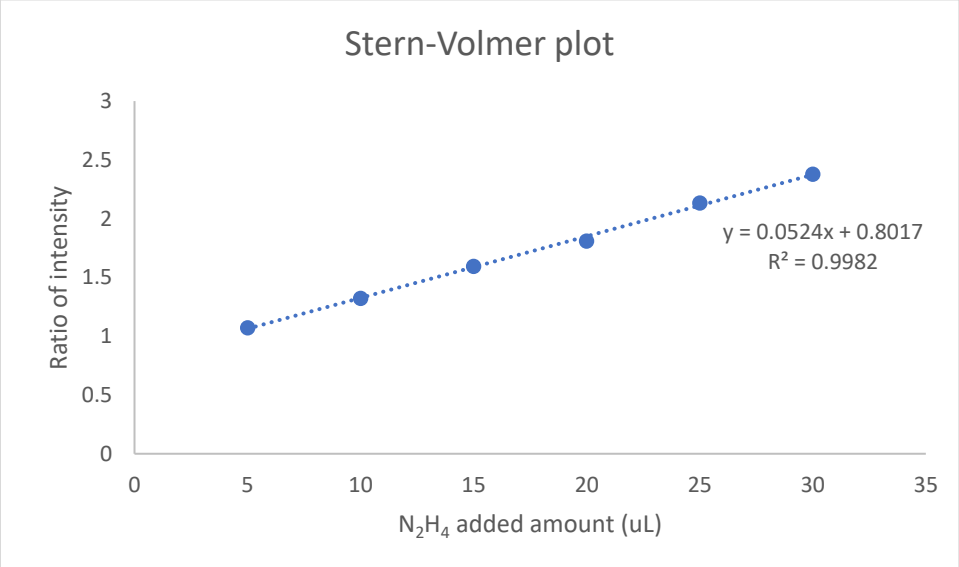


Fig. S3 Stern-Volmer plot of fluorescence of benzophenone quenched by N_2H_4.

8. Examine stability of N_2H_4 under the standard photochemical conditions

The Wilmad® quartz NMR tube (diam. 5mm, L 7 in.) was vacuumed and refilled in argon with three cycles, then N_2H_4•H_2O (0.15 mmol, 7.4 μL), internal standard 1,3,5-trimethoxybenzene (11.2 mg), and CD_3CN (1 mL) were added into the tube sequentially under the protection of Argon. Then the tube was capped, sealed and was irradiated at room temperature (ca. 25 °C) under UV light (254 nm) by using a standard LZC-4V photoreactor from Luzchem company for 24 h. After completion, the sample was direct to test the ^1H NMR. The spectrums indicated the N_2H_4 was stable under the standard conditions (Fig. S4)

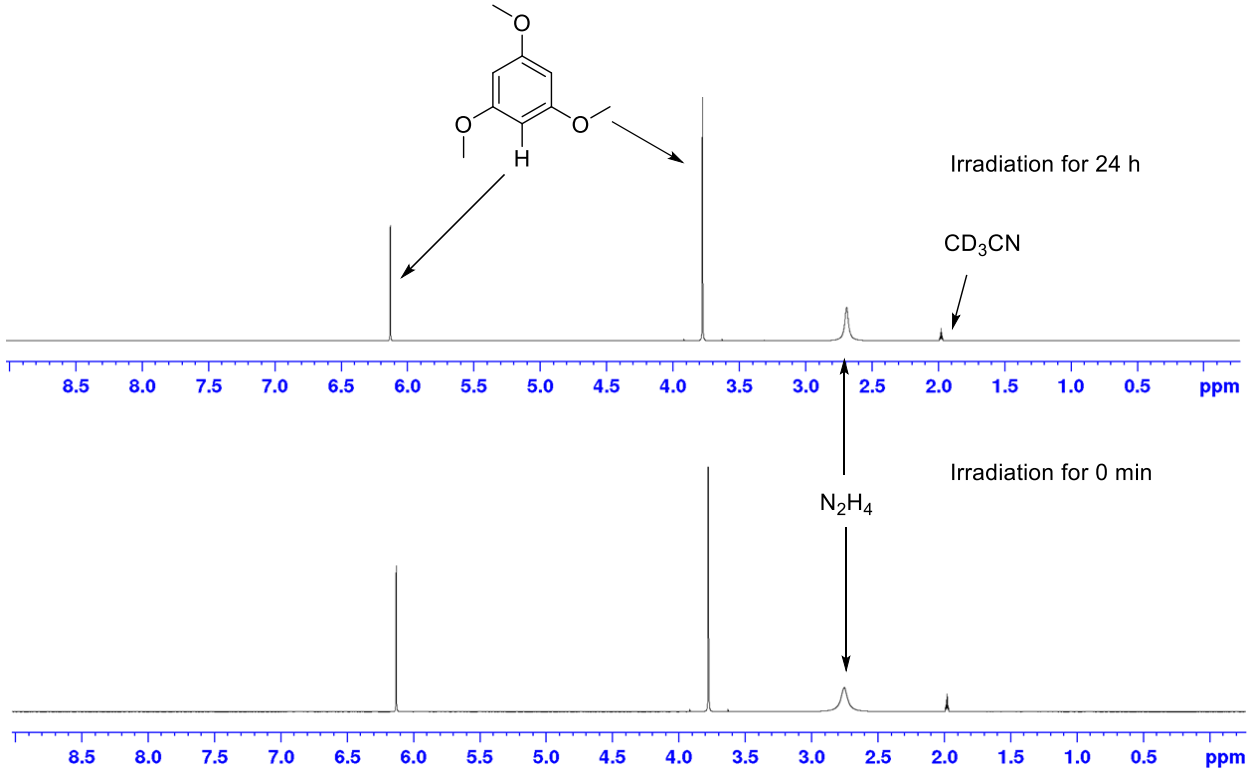


Fig. S4 in situ NMR studies of N_2H_4 stability.

Moreover, using PhCHO to examine the N_2H_4 amount in the NMR tube after irradiation showed the quantitively recovery of N_2H_4. The two evidence both strongly demonstrated the stability of N_2H_4 under the standard photochemical conditions.

9. Determine the number of hydrogens reacted in N_2H_4

The Wilmad® quartz NMR tube (diam. 5mm, L 7 in.) was vacuumed and refilled in argon with three cycles, then benzophenone (0.2 mmol, 36.4 mg, 1 equiv), N_2H_4•H_2O (x equiv, indicated in Scheme 2 in the manuscript), internal standard 1,3,5-trimethoxybenzene (11.2 mg), and CD_3CN (1 mL) were added into the tube sequentially under the protection of Argon. Then the tube was capped, sealed and was irradiated at room temperature (ca. 25 °C) under UV light (254 nm) by using a standard LZC-4V photoreactor from Luzchem company for 24 h. After completion, the sample was direct to test the ^1H NMR, the NMR yields and conversions were calculated by the internal standard 1,3,5-trimethoxybenzene.

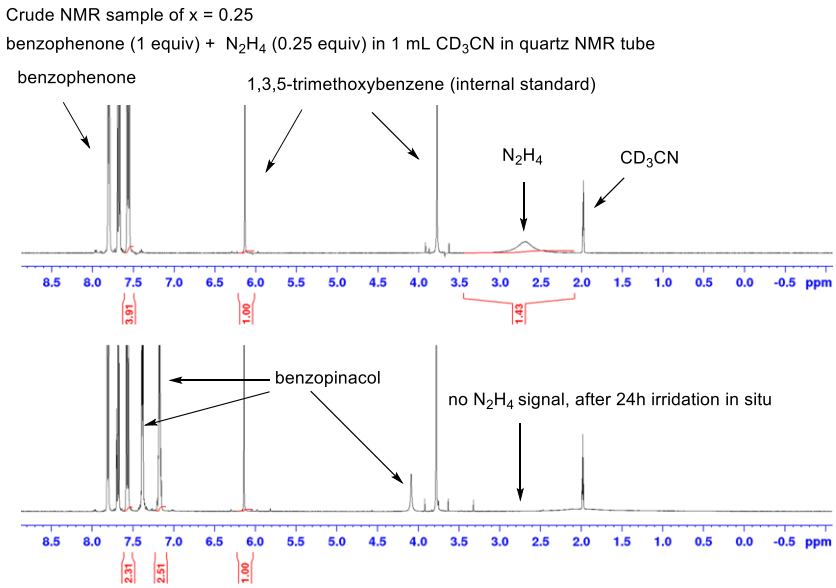


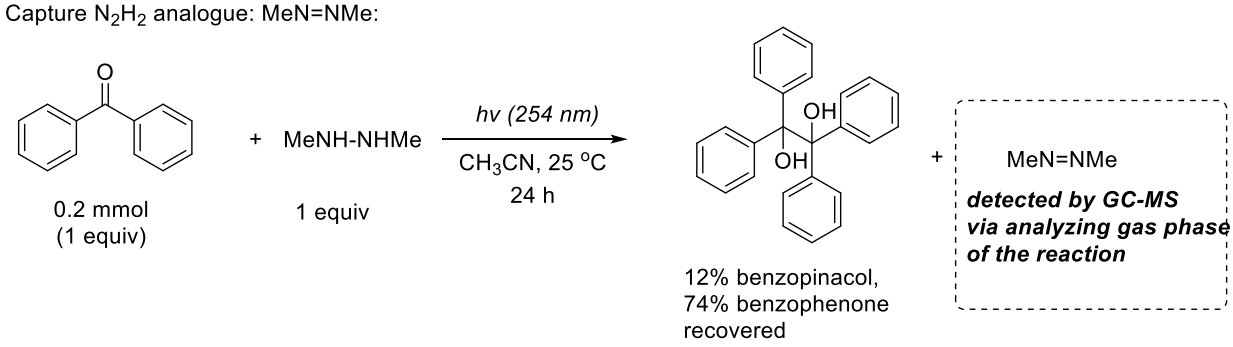
Fig. S5 Crude NMR sample of x = 0.25

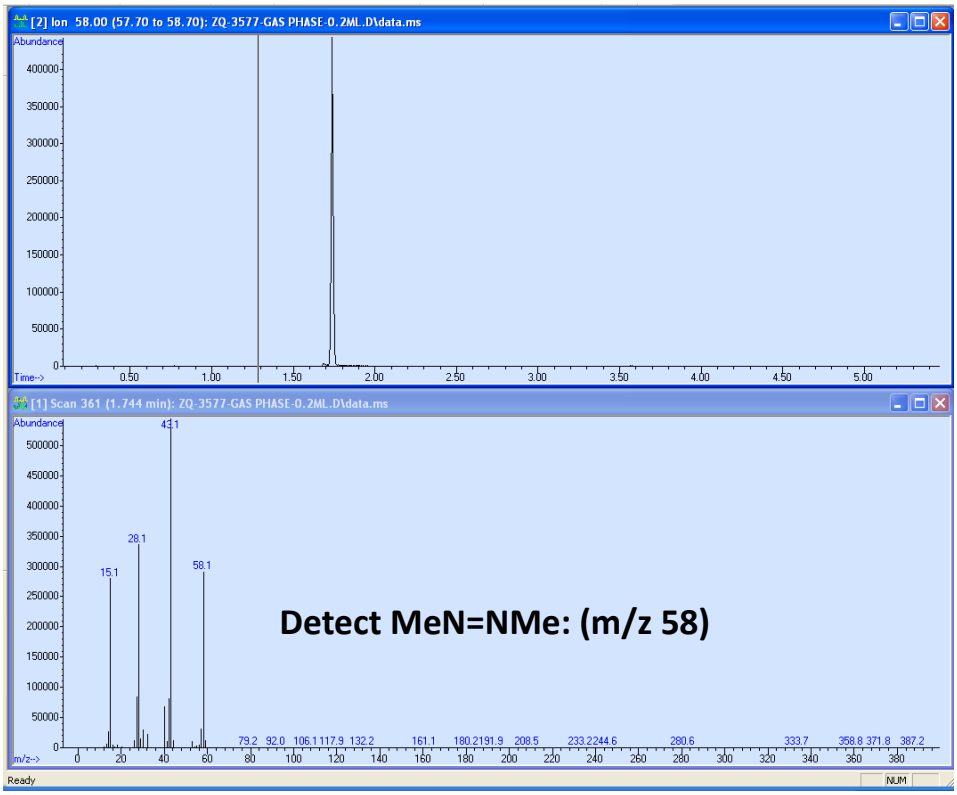
the N_2H_4 was totally used up by analyzing the crude NMR (Fig. S5), which was further confirmed by no hydrazone or azine formed when benzaldehyde was added into the NMR tube after the reaction. Combined with the results shown in Scheme 2 in the manuscript, it clearly showed that only two hydrogens in N_2H_4 were involved in this transformation, for example, when 0.25 equiv N_2H_4 was used, only around half conversion was obtained (42%) with around 42% product yield, in the presence of total 1 equiv hydrogen atoms (4 x 0.25 equiv).

10. Detection of N_2H_2 analogue: MeN=NMe

The 10 mL quartz tube, charged with a magnetic stir bar, was vacuumed and backfilled with argon for three times. Benzophenone (0.2 mmol, 1 equiv, 36.4 mg), MeNH-NHMe•2HCl (0.2 mmol, 1 equiv, 26.6 mg), K_3PO_4 (0.4 mmol, 2 equiv, 85 mg) and CH_3CN (1 mL) were added into the tube sequentially under the protection of Argon. Then the tube was sealed and stirred at room temperature (ca. 25 °C) under UV irradiation (254 nm) by using a standard LZC-4V photoreactor from Luzchem company for 24 h. After completion, the 0.2 mL of gas phase of the reaction was transferred out by gas-tight syringe and then injected into the GC-MS to analyze.

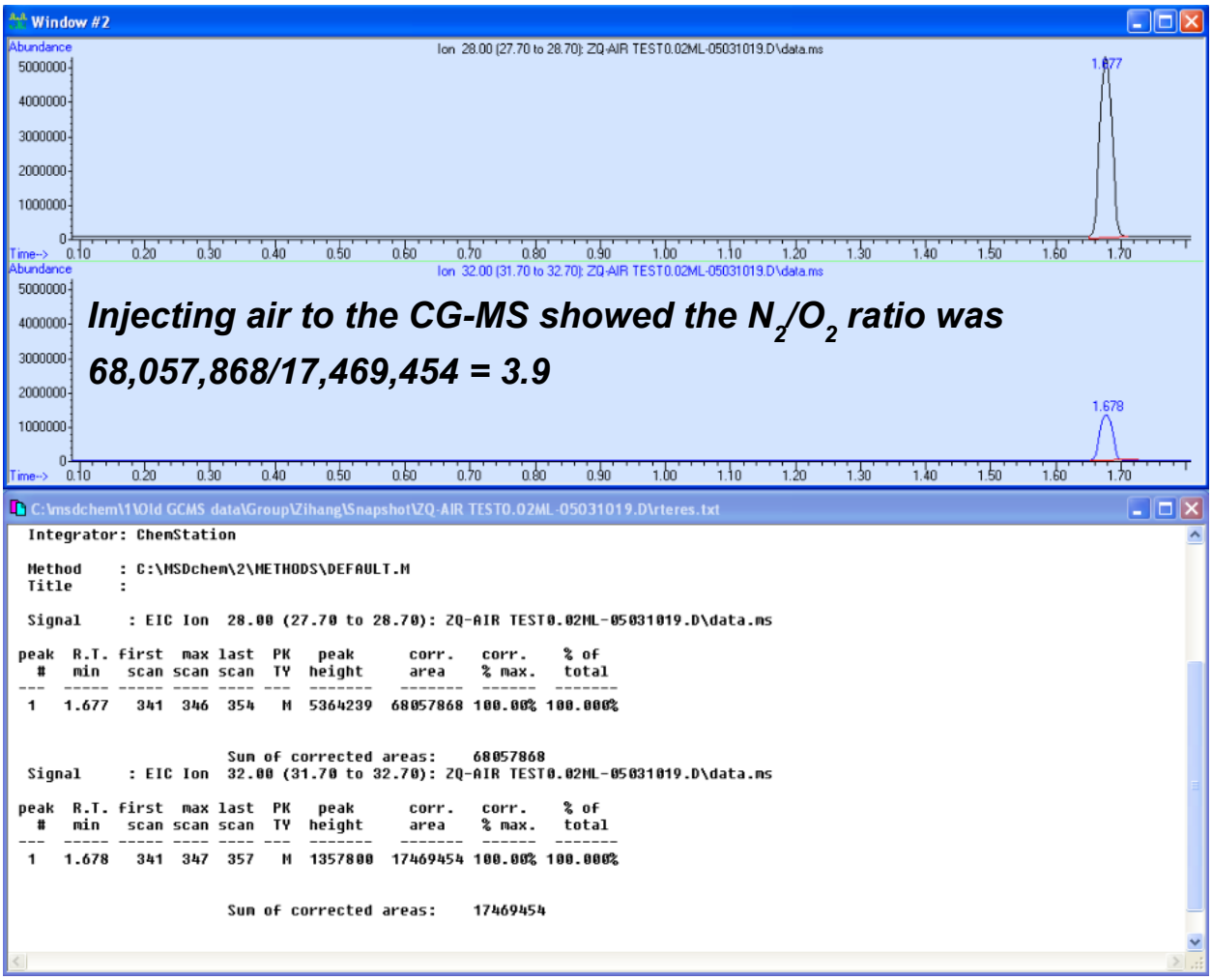
Capture N_2H_2 analogue: MeN=NMe:





11. Detection of N_2

Firstly, due to the air containing N_2 and O_2 (theoretical ratio around 4:1), the air could be used to calibrate the GC-MS. Transferring 0.02 mL air by the gas-tight syringe and injecting into the GC-MS indicated that N_2/O_2 = 3.9 very near the theoretical ratio.



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 Integrator: ChemStation

 Method    : C:\MSDchem\2\METHODS\DEFAULT.M
 Title     :

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peak  R.T. first  max last  PK   peak      corr.   corr.    % of
  #   min   scan scan scan  TY  height     area    % max.   total
---  ----- ----- ---- ---- ---  -------   -------  ------  -------
 1   1.677   341  346  354   M  5364239  68057868 100.00% 100.000%


                     Sum of corrected areas:    68057868
 Signal     : EIC Ion  32.00 (31.70 to 32.70): ZQ-AIR TEST0.02ML-05031019.D\data.ms

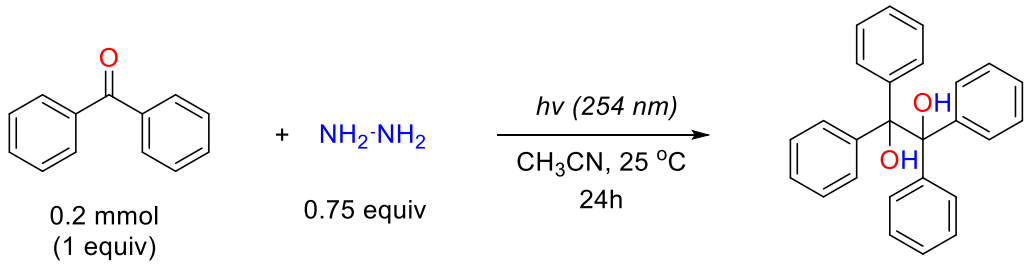
peak  R.T. first  max last  PK   peak      corr.   corr.    % of
  #   min   scan scan scan  TY  height     area    % max.   total
---  ----- ----- ---- ---- ---  -------   -------  ------  -------
 1   1.678   341  347  357   M  1357800  17469454 100.00% 100.000%

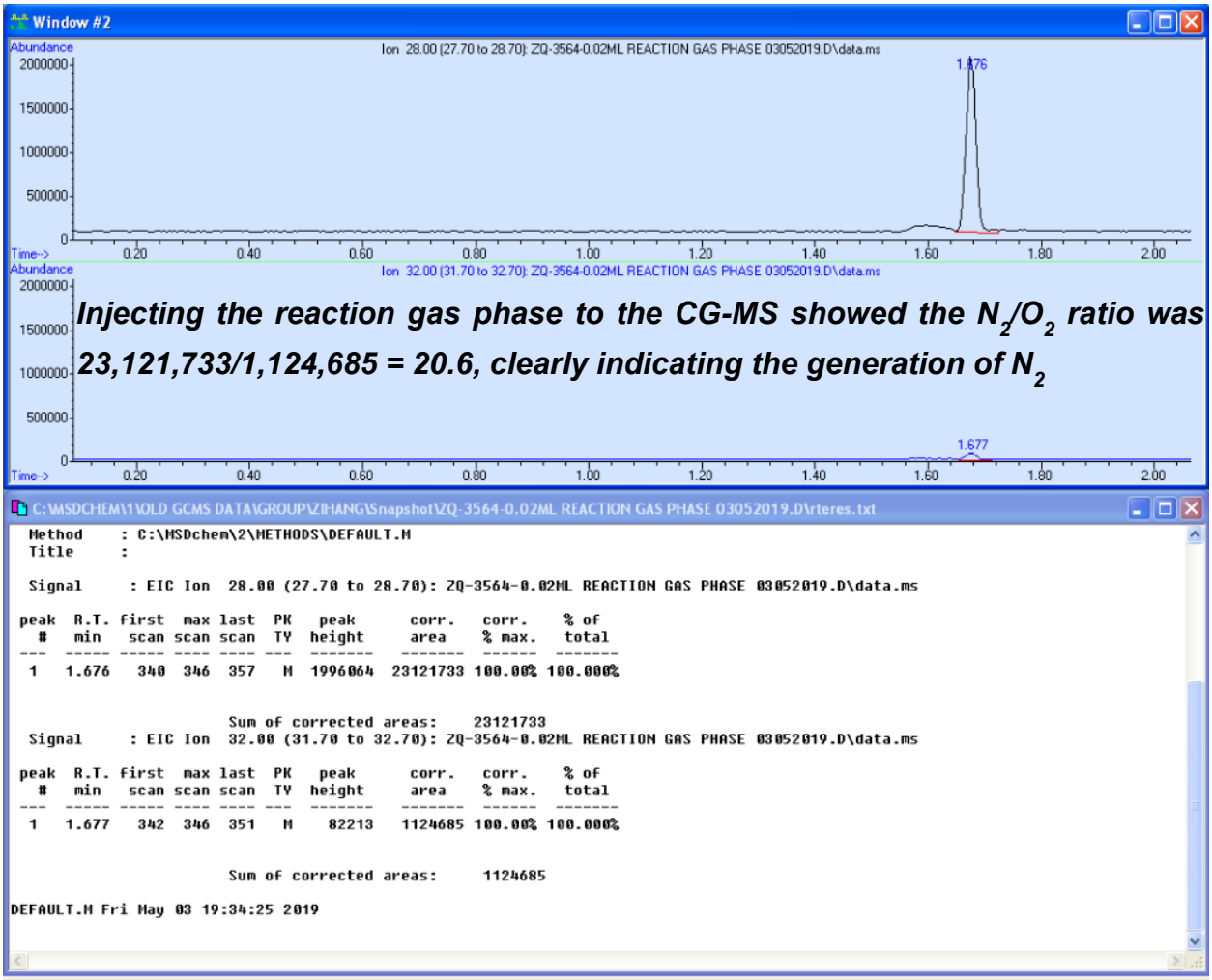

                     Sum of corrected areas:    17469454
```

Next, due to the gas-tight syringe needle will always contain trace amount of air, if the gas sample obtained from the gas phase of the reaction and N_2/O_2 ratio is significantly larger than 3.9 by GC-MS analysis, then it will tell the N_2 is generated during the reaction. The standard reaction was run as follows:

The 10 mL quartz tube, charged with a magnetic stir bar, was vacuumed and backfilled with argon for three times. Benzophenone (0.2 mmol, 1 equiv, 36.4 mg), N_2H_4•H_2O (0.15 mmol, 0.75 equiv, 7.4 µL) and CH_3CN (1 mL) were added into the tube sequentially under the protection of Argon. Then the tube was sealed and stirred at room temperature (ca. 25 ^oC) under UV irradiation (254 nm) by using a standard LZC-4V photoreactor from Luzchem company for 24 h. After completion, the 0.02 mL of gas phase of the reaction

was transferred out by gas-tight syringe and then injected into the GC-MS to analyze. The results were shown below:



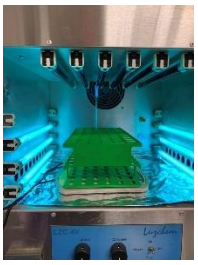


Finally, the NH_3 was not detected by analyzing the reaction gas phase in the GC-MS.

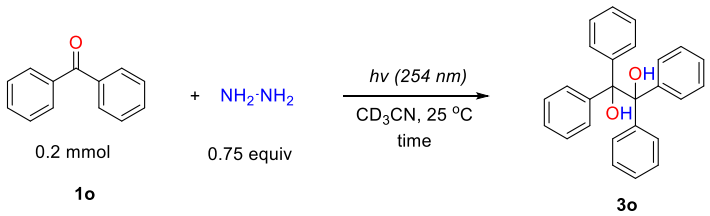
12. Identify rate determining step (RDS)

12.1 Identify whether HAT was not RDS
12.1.1Kinetic profile of the standard reaction:

The kinetic profile of the standard reaction was monitored by the ^{1}H NMR via the reaction run in situ in quartz NMR tube. The procedure was as follows:



The Wilmad® quartz NMR tube (diam. 5mm, L 7 in.) was vacuumed and refilled in argon with three cycles, then benzophenone (0.2 mmol, 36.4 mg, 1 equiv), N_2H_4•H_2O (0.75 equiv, 7.4 μL), internal standard 1,3,5-trimethoxybenzene (11.2 mg), and CD_3CN (1 mL) were added into the tube sequentially under the protection of Argon. Then the tube was capped, sealed and was irradiated at room temperature (ca. 25 °C) under UV light (254 nm) by using a standard LZC-4V photoreactor from Luzchem company. Every 30 mins, the reaction was monitored by ^{1}H NMR, the NMR yields were calculated by the internal standard 1,3,5-trimethoxybenzene. The results were summarized below (Fig. S6):



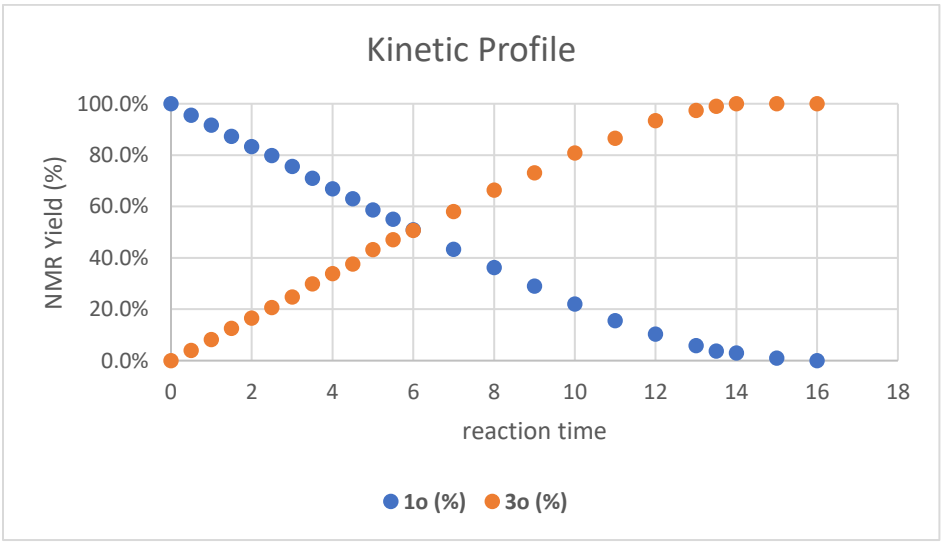
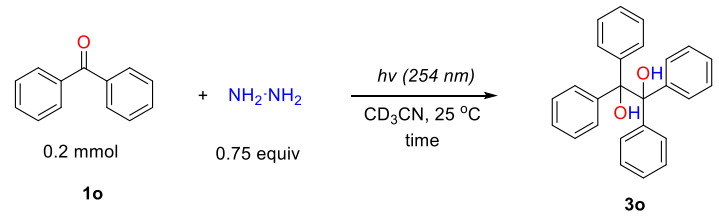


Fig. S6 Kinetic profile.

The results in Fig. S6 showed that the reaction under the standard conditions with benzophenone as the substrate would finish after 16 h. As shown in Fig. 6, the linear relationship of product against time was well fitted with the conversion of benzophenone lower than 30%. Therefore, the initial observed reaction rate (v_{obs}) was calculated within the benzophenone conversion < 30%. The calculation method and results are shown below in details:



0.2 mmol

0.75 equiv

1o

3o

time (h)	**3o** (%)	**3o** (M)
0	0.0%	0
0.5	4.3%	0.0043
1	8.8%	0.0088
1.5	13.5%	0.0135
2	17.7%	0.0177

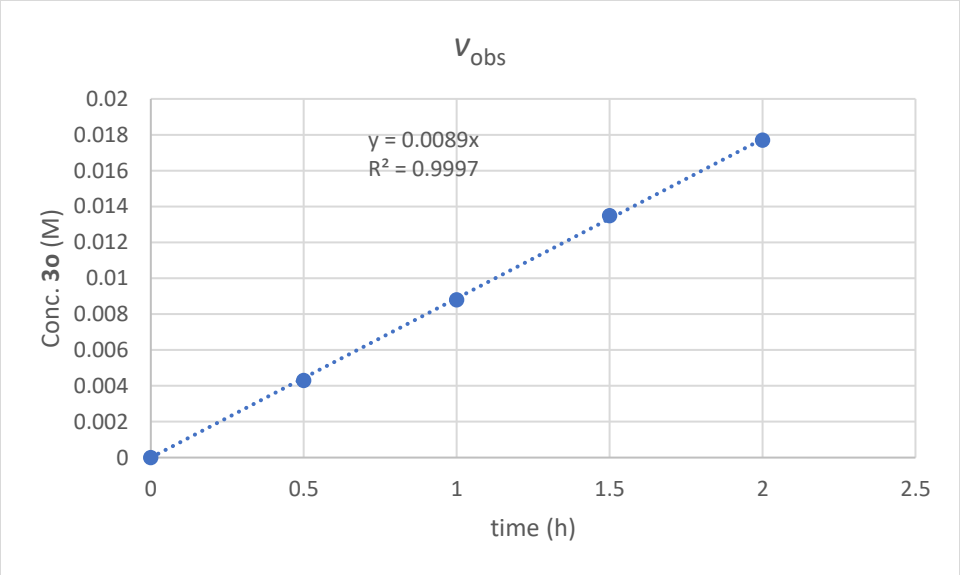
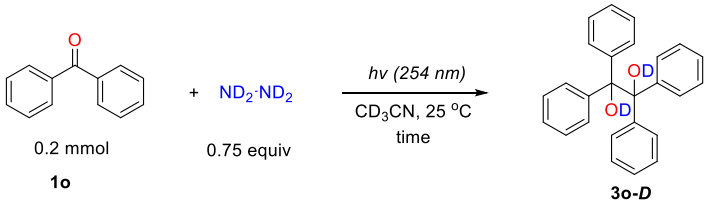


Fig. S7 Observed reaction rate.

From the results shown in Fig. S7, a very good linear fitting was obtained (R^2 = 0.9997), v_{obs} = 0.0089 M/h

12.1.2 Use N_2D_4 to detect v_{obs} for KIE studies

The procedure was similar as above mentioned by only changing the N_2H_4•H_2O to N_2D_4•D_2O. The results were summarized below (Fig. S8):



time (h)	**3o-D** (%)	**3o-D** (M)
0	0.0%	0
0.25	2.1%	0.0021
0.5	4.3%	0.0043
0.75	6.5%	0.0065
1	8.7%	0.0087

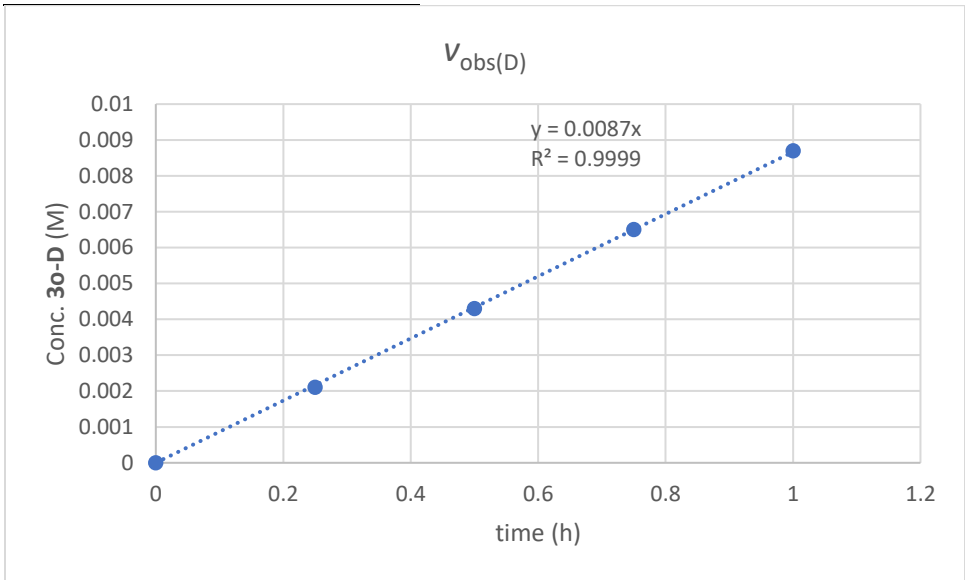
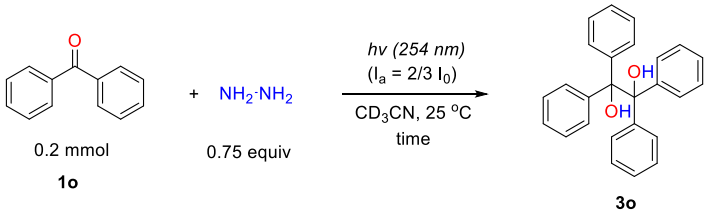


Fig. S8 Observed reaction rate for N_2D_4.

From the results shown in Fig. S8, a very good linear fitting was obtained (R^2 = 0.9999), $v_{obs(D)}$ = 0.0087 M/h. Due to KIE = k_H/k_D = $v_{obs(H)}$/ $v_{obs(D)}$ = 0.0089/0.0087 ≈ 1, there was no KIE effect, indicating that the HAT process was not RDS but a fast step.

12.2 Identify photoexcitation step was RDS

The procedure was similar with the standard conditions shown in 12.1 except for only reducing the UV light intensity to 2/3 of its original intensity (I_a = 2/3 I_0). The results were summarized below (Fig. S9):



time (h)	**3o** (%)	**3o** (M)
0	0.0%	0
0.5	3.0%	0.003
1	5.8%	0.0058
1.5	8.8%	0.0088
2	11.9%	0.0119

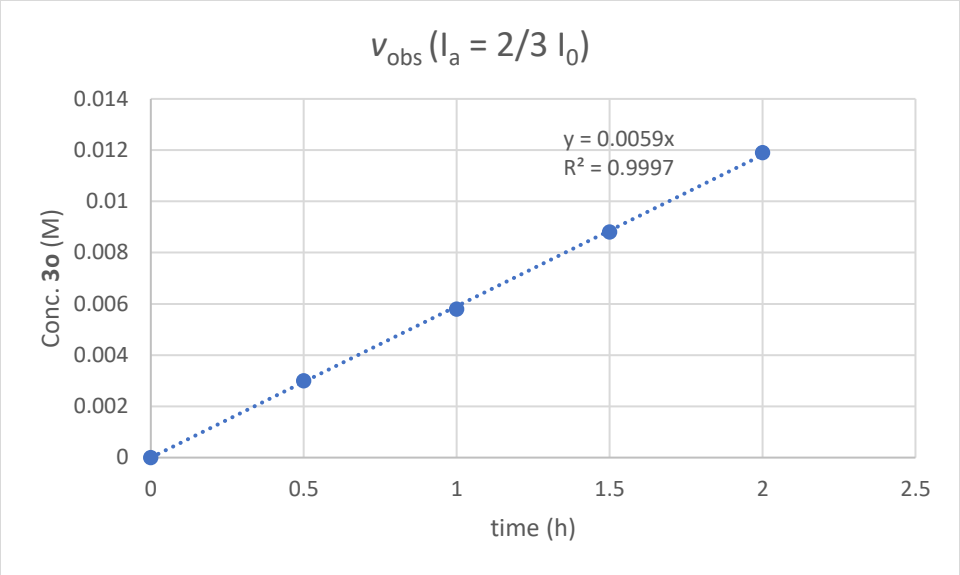


Fig. S9 Observed reaction rate for I_a = 2/3 I_0.

From the results shown in Fig. S9, a very good linear fitting was obtained (R^2 = 0.9997), $v_{obs}(I_a)$ = 0.0059 M/h. $v_{obs}(I_a)$/ $v_{obs}(I_0)$ = 0.0059/0.0089 = 0.663 =1.99/3 ≈ 2/3. It was found that the reaction rate was accordingly reduced when the light intensity was reduced, suggesting that the photoexcitation step was RDS.

13. Identify C-C bond formation mechanism

13.1 If it was radical homo-dimerization mechanism, then the simplified mechanism was shown below (Fig. S10):

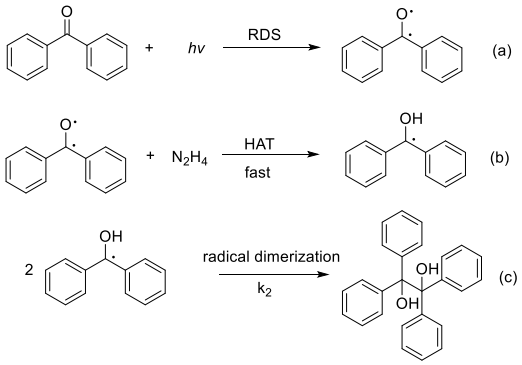


Fig. S10 Simplified mechanism of radical homo-dimerization.

The photoexcitation step could be described as the simplified equation proposed by Toby:[3]

$$A + hv \rightarrow A^*$$

$$-\frac{d[A]}{dt} = \frac{d[A^*]}{dt} = I_0 - I_1$$

where A represented benzophenone, A* represented the excited state of benzophenone, I_0 was the initial light intensity, I_1 was the light intensity after it passes through the sample.

According to the Beer-Lambert law:

$$\log\frac{I_0}{I_1} = \varepsilon l c$$

So, $I_1 = I_0 10^{-\varepsilon l c}$

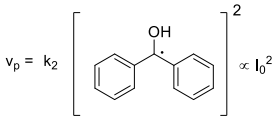
Where, ε was the molar absorptivity, l was the length of the light path, c was the concentration of the solution of the sample used in the experiment.

Then,

$$-\frac{d[A]}{dt} = \frac{d[A^*]}{dt} = I_0 - I_1 = I_0\left(1 - 10^{-\varepsilon l c}\right) = k_1 I_0 \propto I_0$$

As the ε, l were constant for the given reaction conditions, and when the conversion was low, the c was not changed greatly, so the $\left(1 - 10^{-\varepsilon l c}\right)$ could be treated as the constant (k_1) in the initial stage of reaction. Therefore, the $\frac{d[A^*]}{dt}$ would be directly proportional to the I_0.

As the mechanistic studies were shown previously, the photoexcitation step was RDS (Fig. S10, a) and the HAT process was the fast step (Fig. S10, b). Therefore, the reaction rate of the generation of the product could be simplified to be described as (Fig. S10, c):



Where v_p was the rate of the generation of the product, k_2 was the radical dimerization rate constant. Therefore, If the mechanism for the C-C bond formation was radical homo-dimerization then v_p would be directly proportional to the I_0^2 (the light intensity square), but this was not consistent of the kinetic observation of this transformation (v_p was directly proportional to the I_0). Therefore, the radical homo-dimerization pathway could not be the main pathway for the C-C bond formation step.

13.2 Instead, if it was radical addition to C=O mechanism, then the simplified mechanism was shown below (Fig. S11):

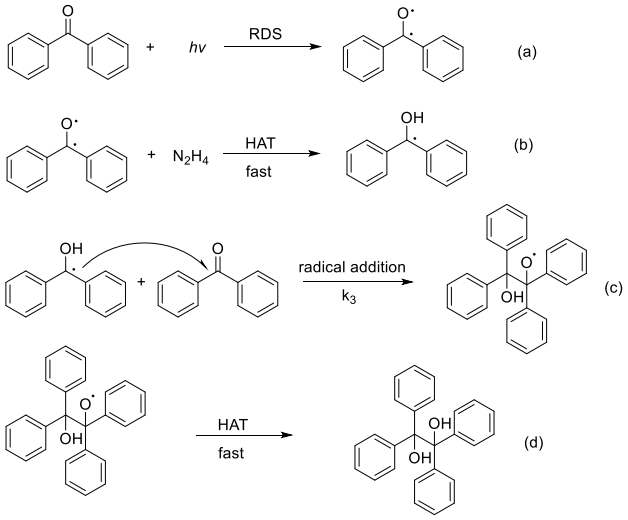
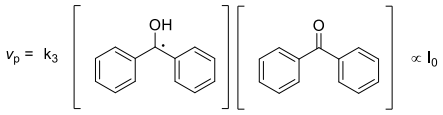


Fig. S11 Simplified mechanism of radical addition to C=O.

As the analysis shown in 13.1 with the photoexcitation step was RDS (Fig. S11, a), and HAT were fast steps (Fig. S11, b and d), the reaction rate of the generation of the product could be simplified to be described as (Fig S11, c and d):



Where k_3 was the rate constant of radical addition to the C=O. Because the concentration of benzophenone was significantly larger than the ketyl radical concentration and the benzophenone concentration would be kept nearly constant under low conversion, it could be treated as a constant. Therefore, v_p would be directly proportional to the I_0, in agreement with the kinetic observation of this transformation.

14. DFT study of the mechanism

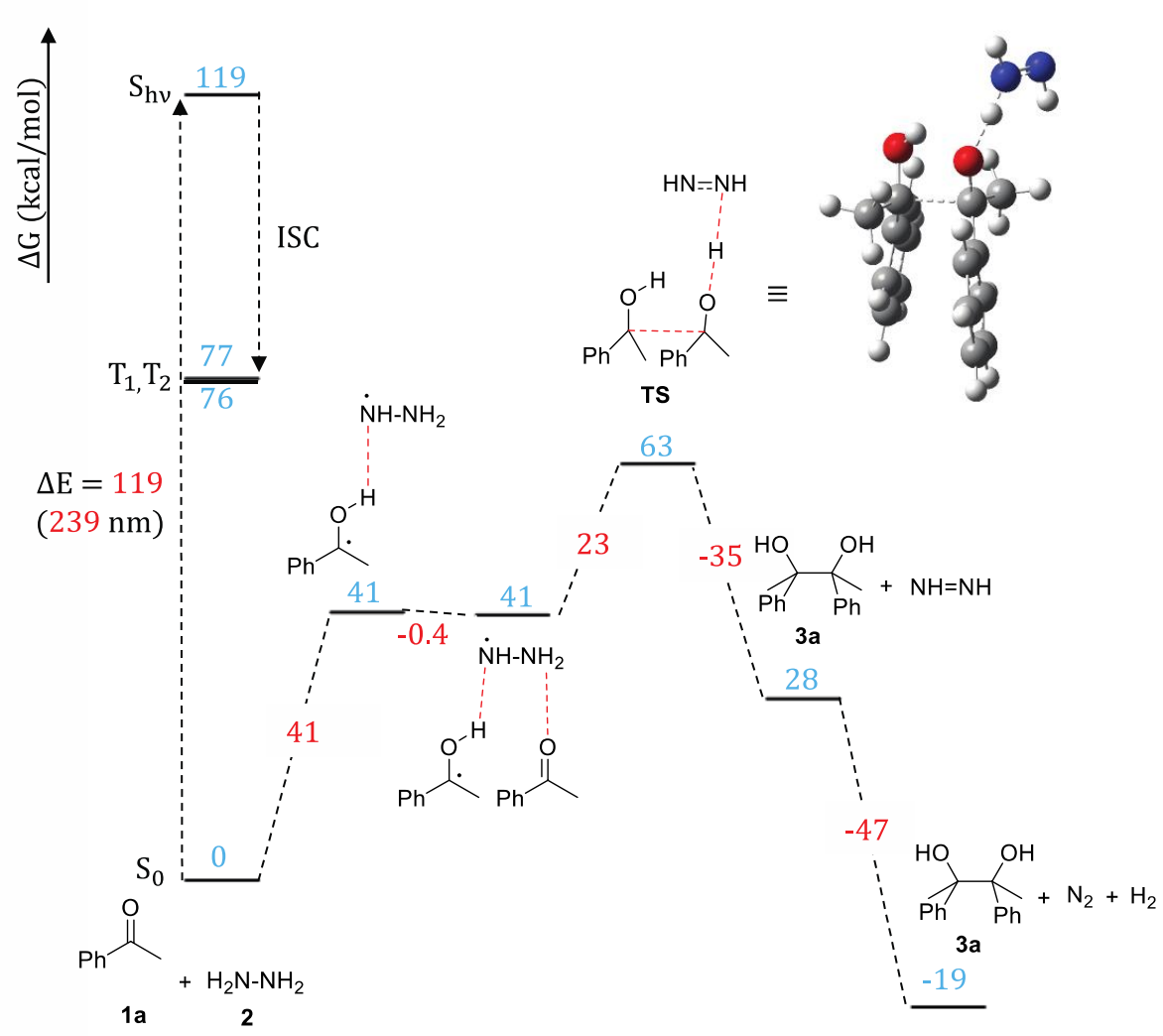


Fig. S12 The B3LYP/6-31G(d,p) free energy profile of the reaction. [a] The vertical excitation energies (as opposed to free energies) are plotted relative to the electronic ground state.

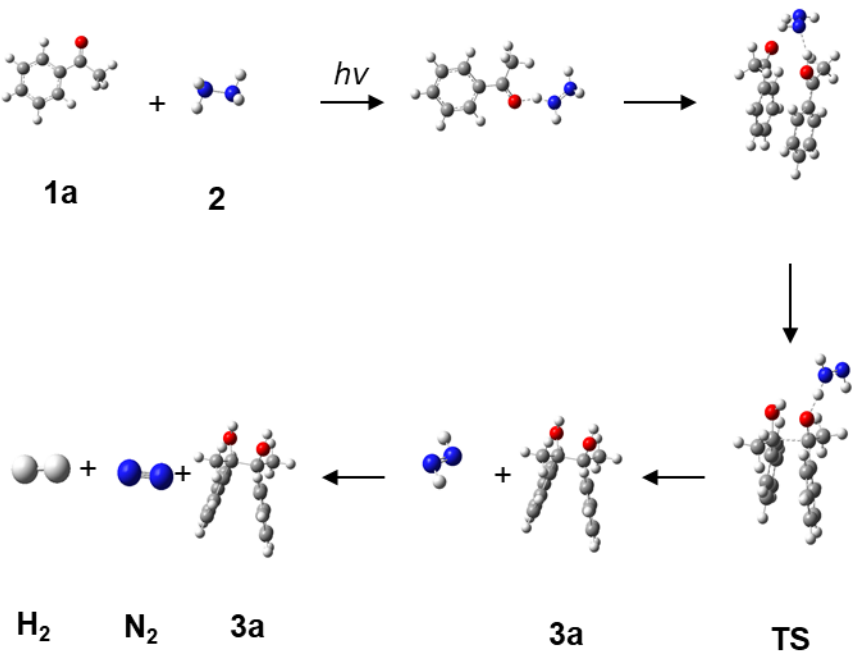


Fig. S13 Structures of intermediates and transition states along the reaction pathway.

15. Attempts of cross-pinacol coupling

Initially, we examined the feasibility of cross-pinacol coupling by mixing acetophenone, 4-CF_3 and 4-OMe acetophenones and benzophenone (the yields shown below are NMR yields):

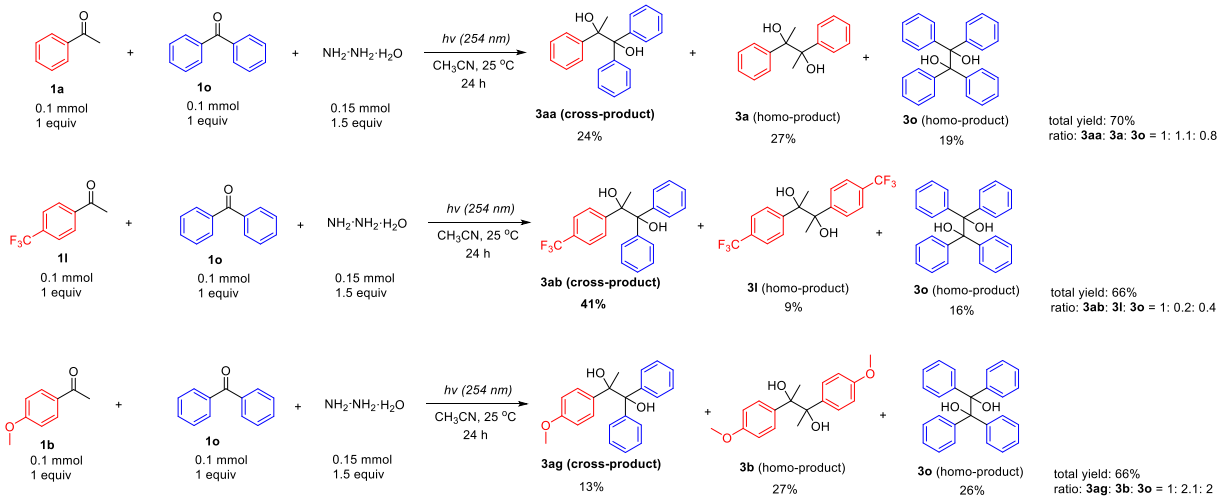
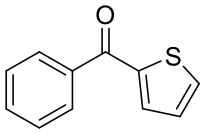


Fig. S14 Initial examination of the cross-pinacol coupling feasibility.

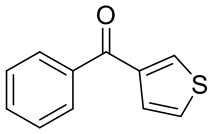
We found that acetophenone attached with electron-withdrawing group such as 4-CF_3 acetophenone could afford better cross-pinacol product selectivity and yield (**3ab**, 41% NMR yield) while mixing with benzophenone, compared with acetophenone and 4-OMe acetophenone. Next, with this reaction information in hand, we examined the preliminary scope and selectivity of the cross-pinacol reaction, and the results are summarized in Table 3 in the manuscript.

16. Spectroscopic data of synthesized heteroaromatic ketones



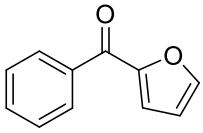
Phenyl(thiophen-2-yl)methanone (1x) (CAS: 135-00-2):[4]

^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.87 (dd, *J* = 8.3 Hz, 1.3 Hz, 2H), 7.73 (dd, *J* = 6.1 Hz, 2.0 Hz, 1H), 7.65 (dd, *J* = 3.8 Hz, 1.1 Hz, 1H), 7.60 (tt, *J* = 7.5 Hz, 1.3 Hz, 1H), 7.50 (t, *J* = 7.8 Hz, 2H), 7.17 (dd, *J* = 4.9 Hz, 3.8 Hz, 1H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 188.4, 143.8, 138.3, 135.0, 134.4, 132.4, 129.3



Phenyl(thiophen-3-yl)methanone (1y) (CAS: 6453-99-2):[5]

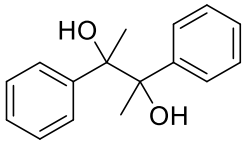
^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.94 (dd, *J* = 2.9 Hz, 1.2 Hz, 1H), 7.85 (d, *J* = 7.0 Hz, 2H), 7.61 – 7.57 (m, 2H), 7.49 (t, *J* = 8.4 Hz, 2H), 7.39 (dd, *J* = 6.1 Hz, 2.9 Hz, 1H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 190.0, 141.3, 138.6, 133.9, 132.3, 129.4, 128.6, 128.4, 126.2



Furan-2-yl(phenyl)methanone (1z) (CAS: 2689-59-0):[5]

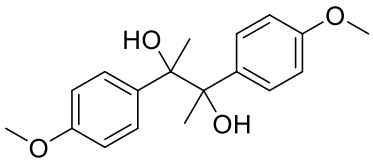
^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.94 (d, *J* = 8.2 Hz, 2H), 7.71 (dd, *J* = 1.7 Hz, 0.7 Hz, 1H), 7.60 (tt, *J* = 7.4 Hz, 1.3 Hz, 1H), 7.50 (t, *J* = 7.9 Hz, 2H), 7.24 (dd, *J* = 3.6 Hz, 0.7 Hz, 1H), 6.60 (dd, *J* = 3.6 Hz, 1.7 Hz, 1H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 182.6, 152.3, 147.1, 137.3, 132.6, 129.3, 128.4, 120.6, 112.2

17. Spectroscopic data of products



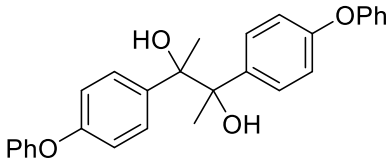
2,3-Diphenylbutane-2,3-diol (3a) (CAS: 1636-34-6):[6]

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 5:1), the product was isolated as a white solid (19.5 mg, 80% yield). *dl : meso* = 1.1: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.29 – 7.22 (m, 10H), 2.61 (br, 1H), 2.31 (br, 1H), 1.61 (s, 3H, *meso*), 1.54 (s, 3.3H, *dl*); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 143.9, 143.6, 127.5, 127.4, 127.3, 127.2, 127.1, 127.0, 79.0, 78.7, 25.3, 25.1; HRMS: (APCI, m/z) calcd for $C_{16}H_{18}O_2Na$ $[M+Na]^+$ 265.1199, found: 265.1201



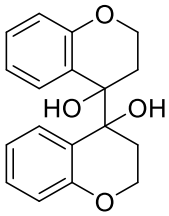
2,3-Bis(4-methoxyphenyl)butane-2,3-diol (3b) (CAS: 21985-99-9):[7]

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 3:1), the product was isolated as a white solid (24.5 mg, 81% yield). *dl : meso* = 1.3: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.15 – 7.10 (m, 4H), 6.79 – 6.76 (m, 4H), 3.81 – 3.79 (s, 6H), 2.53 (br, 1.1H), 2.25 (br, 0.9H), 1.55 (s, 2.7H, *meso*), 1.47 (s, 3.6H, *dl*); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 158.7, 158.6, 136.2, 135.8, 128.7, 128.2, 112.7, 112.5, 78.8, 78.6, 55.3, 25.3, 25.1; HRMS: (ESI, m/z) calcd for $C_{18}H_{22}O_4Na$ $[M+Na]^+$ 325.1410, found: 325.1409



2,3-Bis(4-phenoxyphenyl)butane-2,3-diol (3c):

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 3:1), the product was isolated as a white solid (30.2 mg, 71% yield). *dl : meso* = 1.3: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.35 – 7.30 (m, 4H), 7.21 – 7.16 (m, 4H), 7.12 – 7.08 (m, 2H), 7.02 – 6.97 (m, 4H), 7.90 – 6.87 (m, 4H), 2.51 (br, 1H), 2.32 (br, 1H), 1.62 (s, 2.6H, *meso*), 1.53 (s, 3.6H, *dl*); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 157.4, 157.2, 156.4, 156.2, 138.9, 138.4, 129.88, 129.86, 128.9, 128.5, 123.4, 123.3, 119.0, 118.8, 117.8, 117.5, 78.9, 78.6, 25.3, 25.2; HRMS: (ESI, m/z) calcd for $C_{28}H_{26}O_4Na$ $[M+Na]^+$ 449.1723, found: 449.1711

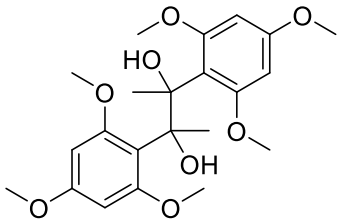


[4,4'-Bichromane]-4,4'-diol (3d)(CAS: 59214-62-9)[8]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After column chromatography on silica gel (eluent: hexanes: EtOAc = 20:1 to 1:1). *dl : meso* = 1:1, the *dl/meso* products total yield (24.4 mg, 82%).

The *dl* product was isolated as a white solid (12.3 mg, 41% yield). ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 8.14 (d, *J* = 8.0 Hz, 2H), 7.22 (t, *J* = 7.3 Hz, 2H), 6.97 (t, *J* = 8.2 Hz, 2H), 6.88 (d, *J* = 8.2 Hz, 2H), 4.11 – 4.01 (m, 2H), 4.02 – 3.97 (m, 2H), 3.22 (br, 2H), 1.75 – 1.68 (m, 2H), 1.62 – 1.58 (m, 2H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 155.7, 129.6, 129.3, 126.8, 121.2, 117.9, 73.4, 63.2, 35.2; HRMS: (ESI, m/z) calcd for $C_{18}H_{18}O_4Na$ $[M+Na]^+$ 321.1097, found: 321.1101

The *meso* product was isolated as a white solid (12.1 mg, 41% yield). ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.20 (t, *J* = 8.5 Hz, 2H), 7.13 (d, *J* = 7.7 Hz, 2H), 6.85 (d, *J* = 8.2 Hz, 2H), 6.80 (t, *J* = 8.1 Hz, 2H), 4.03 – 4.00 (m, 4H), 2.62 (br, 2H), 2.36 – 2.30 (m, 2H), 2.17 – 2.12 (m, 2H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 156.2, 129.8, 128.4, 125.7, 120.9, 117.6, 74.2, 63.7, 34.3; HRMS: (ESI, m/z) calcd for $C_{18}H_{18}O_4Na$ $[M+Na]^+$ 321.1097, found: 321.1087



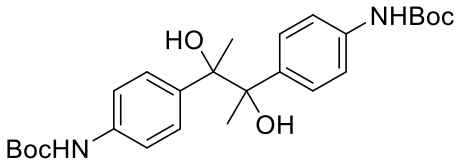
2,3-Bis(2,4,6-trimethoxyphenyl)butane-2,3-diol (3e):

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: pure EtOAc), *d.r.* = 1:1.4, the products total yield (34.2 mg, 81%).

Isomer 1 was isolated as a white solid (13.8 mg, 33% yield). ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 6.17 (d, *J* = 2.3 Hz, 2H), 6.00 (d, *J* = 2.3 Hz, 2H), 5.86 (s, 2H), 3.76 (s, 12H), 3.48 (s, 6H), 1.73 (s, 6H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 160.3, 160.1, 159.4, 116.0, 92.8, 92.2, 84.2, 56.14, 56.12, 55.4, 25.8; HRMS: (ESI, m/z) calcd for $C_{22}H_{30}O_8Na$ $[M+Na]^+$ 445.1833, found: 445.1828

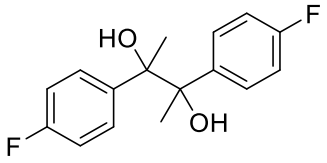
Isomer 2 was isolated as a colorless oil (20.4 mg, 48% yield). ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 6.01 (s, 4H), 5.92 (s, 2H), 6.85 (d, *J* = 8.2 Hz, 2H), 3.75 (s, 6H), 3.53 (s, 6H), 3.44

(s, 6H), 1.74 (s, 6H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 160.4, 160.0, 159.4, 115.8, 92.1, 91.9, 83.6, 56.3, 55.3, 25.7; HRMS: (ESI, m/z) calcd for $C_{22}H_{30}O_8Na$ $[M+Na]^+$ 445.1833, found: 445.1841



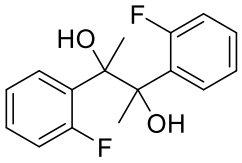
Di-tert-butyl ((2,3-dihydroxybutane-2,3-diyl)bis(4,1-phenylene))dicarbamate (3f):

Following the general procedure, the reaction mixture was diluted by the EtOAc. After column chromatography on silica gel (eluent: hexanes: EtOAc = 3:1 to 1:1), the product was isolated as a white solid (30.4 mg, 64% yield). *dl : meso* = 1: 1; ^{1}H NMR: (500 MHz, acetone-d_6, ppm): δ 8.28 (m, 2H), 7.38 – 7.35 (m, 4H), 7.12 – 7.08 (m, 2H), 7.30 (d, *J* = 8.7 Hz, 2H), 7.09 (d, *J* = 8.6 Hz, 2H), 4.10 (s, 1H), 3.86 (s, 1H), 1.46 – 1.45 (m, 24H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 153.90, 153.86, 140.90, 139.95, 139.0, 138.8, 129.0, 128.8, 117.4, 117.3, 79.9, 79.8, 79.0, 78.6, 28.7, 25.8, 25.3; HRMS: (ESI, m/z) calcd for $C_{26}H_{36}N_2O_6Na$ $[M+Na]^+$ 495.2466, found: 495.2456



2,3-Bis(4-fluorophenyl)butane-2,3-diol (3g) (CAS: 87054-23-7)[9]:

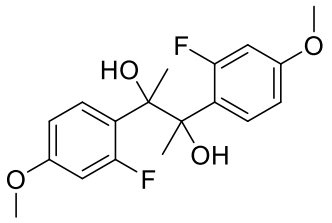
Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 3:1), the product was isolated as a white solid (17.4 mg, 63% yield). *dl : meso* = 1.1: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.20 – 7.17 (m, 2H), 7.13 – 7.11 (m, 2H), 6.94 – 6.89 (m, 4H), 2.52 (br, 1H), 2.28 (br, 1H), 1.57 (s, 3.3H), 1.49 (s, 3.7H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 163.1, 163.0, 161.2, 161.1, 139.59, 139.57, 139.22, 139.19, 129.24, 129.17, 128.84, 128.78, 114.2, 114.1, 114.0, 113.9, 78.7, 78.5, 25.2, 25.0; ^{19}F NMR: (471 MHz, $CDCl_3$, ppm): δ -115.6, -116.2; HRMS: (ESI, m/z) calcd for $C_{16}H_{15}O_6F_2$ $[M-H]^-$ 277.1046, found: 277.1054



2,3-Bis(2-fluorophenyl)butane-2,3-diol (3h) (CAS: 1798248-13-1)[10]:

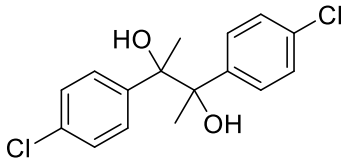
Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 3:1), the product was isolated as a white solid (18.0 mg, 65% yield). *dl : meso* = 1: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.50 – 7.46 (m, 1H), 7.31 – 7.27 (m, 1H), 7.24 – 7.12 (m, 3H), 7.01 – 6.96 (m, 2H), 6.91

– 6.87 (m, 1H), 3.19 – 3.09 (br, 2H), 1.80 (s, 3H, *meso*), 1.68 (s, 3H, *dl*); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 161.7, 161.6, 159.8, 159.7, 130.34, 130.3, 130.09, 130.06, 129.54, 129.47, 129.4, 129.3, 123.7, 123.7, 123.4, 123.3, 116.4, 116.22, 166.18, 116.0, 79.9, 79.8, 79.74, 79.71, 24.6, 24.5, 24.2, 24.1; ^{19}F NMR: (471 MHz, $CDCl_3$, ppm): δ -109.0, -109.7; HRMS: (ESI, m/z) calcd for $C_{16}H_{16}O_6F_2Na$ $[M+Na]^+$ 301.1011, found: 301.1020



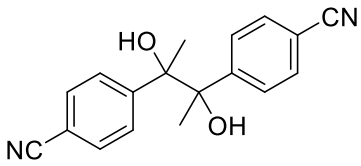
2,3-Bis(2-fluoro-4-methoxyphenyl)butane-2,3-diol (3i):

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 3:1), the product was isolated as a white solid (29.6 mg, 88% yield). *dl : meso* = 1.1: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.33 (t, *J* = 9.2 Hz, 1H), 7.01 (t, *J* = 9.2 Hz, 1H), 6.66 (dd, *J* = 8.5 Hz, 2.6 Hz, 1H), 6.53 – 6.50 (m, 2H), 6.43 (dd, *J* = 14.6 Hz, 2.6 Hz, 1H), 3.79 (s, 3H), 3.76 (s, 3H), 3.06 – 2.98 (br, 2H), 1.71 (s, 2.7 H, *meso*), 1.60 (s, 2.9 H, *dl*); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 162.1, 162.0, 160.4, 160.3, 160.2, 160.1, 130.8, 130.8, 130.6, 122.3, 122.2, 109.4, 109.1, 102.1, 101.9, 101.6, 79.6, 79.5, 55.6, 24.6, 24.2; ^{19}F NMR: (471 MHz, $CDCl_3$, ppm): δ -106.9, -107.6; HRMS: (ESI, m/z) calcd for $C_{18}H_{20}O_4F_2Na$ $[M+Na]^+$ 361.1222, found: 361.1229



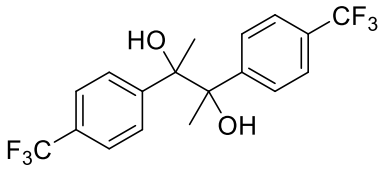
2,3-Bis(4-chlorophenyl)butane-2,3-diol (3j) (CAS: 21985-98-8)[11]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 3:1), the product was isolated as a white solid (20.7 mg, 67% yield). *dl : meso* = 1.1: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.16 – 6.98 (m, 8H), 2.43 (br, 1H), 2.14 (br, 1H), 1.45 (s, 3H, *meso*), 1.38 (s, 3.4 H, *dl*); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 142.3, 141.9, 133.3, 133.2, 129.0, 128.6, 127.54, 127.46, 78.7, 78.4, 25.2, 24.9; HRMS: (ESI, m/z) calcd for $C_{16}H_{15}O_2Cl_2$ $[M-H]^-$ 309.0455, found: 309.0450



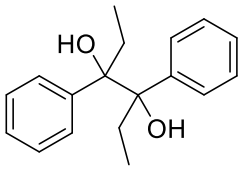
4,4'-(2,3-Dihydroxybutane-2,3-diyl)dibenzonitrile (3k) (CAS: 82491-64-3)[12]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: Acetone= 3:1), the product was isolated as a white solid (20.6 mg, 70% yield). *dl : meso* = 1: 1.7; ^{1}H NMR: (500 MHz, acetone-d_6, ppm): *dl*: δ 7.78 (d, *J* = 8.6 Hz, 4H), 7.70 (d, *J* = 8.6 Hz, 4H), 4.61 (br, 2H), 1.50 (s, 6H); *meso:* 7.52 (d, *J* = 8.7 Hz, 4H), 7.43 (d, *J* = 8.6 Hz, 4H), 4.79 (br, 2H), 1.77 (s, 6H); ^{13}C NMR: (125 MHz, acetone-d_6, ppm): δ 152.1, 151.9, 131.2, 130.8, 129.2, 128.8, 119.33, 119.31, 110.7, 110.4, 78.3, 78.0, 24.9, 24.5; HRMS: (ESI, m/z) calcd for $C_{18}H_{16}N_2O_2Na$ $[M+Na]^+$ 315.1104, found: 315.1097



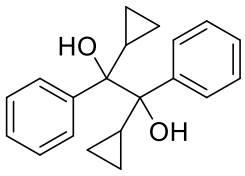
2,3-Bis(4-(trifluoromethyl)phenyl)butane-2,3-diol (3l)[13]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc= 3:1), the product was isolated as a colorless oil (26.0 mg, 69% yield). *dl : meso* = 1.1: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.52 – 7.50 (m, 4H), 7.44 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 2.61 (br, 1H), 2.22 (br, 1H), 1.57 (s, 3H, *meso*), 1.54 (s, 3.3H, *dl*); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 147.9, 147.3, 129.8, 129.6, 129.4, 127.9, 127.6, 124.4, 124.3, 78.7, 78.4, 25.4, 25.0; ^{19}F NMR: (471 MHz, $CDCl_3$, ppm): δ -62.5; HRMS: (ESI, m/z) calcd for $C_{18}H_{16}F_6O_2Na$ $[M+Na]^+$ 401.0947, found: 401.0951



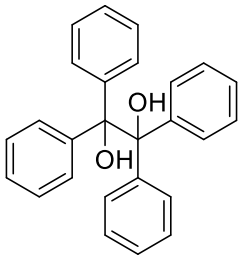
3,4-Diphenylhexane-3,4-diol (3m) (CAS: 10442-33-8)[14]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc= 5:1), the product was isolated as a white solid (15.1 mg, 63% yield). *dl : meso* = 1: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.30 – 7.18 (m, 10H), 2.59 (br, 1H), 2.22 (br, 1H), 2.42 – 2.35 (m, 1H), 2.14 – 2.16 (m, 2H), 1.76 – 1.70 (m, 1H, *dl*), 1.64 – 1.57 (m, 1H, *meso*); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 141.4, 140.4, 128.4, 127.8, 127.5, 127.3, 127.0, 126.8, 82.04, 81.96, 28.3, 27.8, 7.9, 7.7; HRMS: (ESI, m/z) calcd for $C_{18}H_{16}O_2Na$ $[M+Na]^+$ 293.1512, found: 293.1519



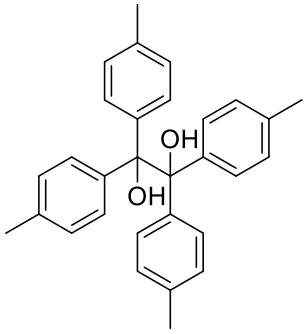
1,2-Dicyclopropyl-1,2-diphenylethane-1,2-diol (3n) (CAS: 60079-97-2)[15]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc= 10:1), the product was isolated as a white solid (21.3 mg, 72% yield). *dl : meso* = 1.1: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.35 – 7.18 (m, 10H), 2.47 (br, 1H), 2.28 (br, 1H), 1.96 – 1.90 (m, 0.9H, *meso*), 1.50 – 1.45 (m, 1H, *dl*), 0.71 – 0.60 (m, 3H), 0.58 – 0.52 (m, 1H), 0.41 – 0.35 (m, 1H), 0.30 – 0.25 (m, 1H), 0.17 – 0.07 (m, 1H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 143.8, 143.4, 127.8, 127.6, 127.2, 127.13, 127.05, 127.0, 79.6, 16.8, 16.7, 3.3, 0.2, 0.0; HRMS: (ESI, m/z) calcd for $C_{20}H_{22}O_2Na$ $[M+Na]^+$ 317.1512, found: 317.1518



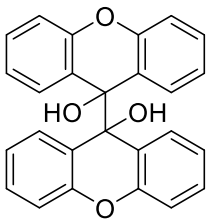
1,1,2,2-Tetraphenylethane-1,2-diol (3o) (CAS: 464-72-2)[16]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc= 10:1), the product was isolated as a white solid (30.8 mg, 84% yield); ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.31 – 7.29 (m, 8H), 7.20 –7.15 (m, 12H), 3.03 (br, 2H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 144.3, 128.7, 127.4, 127.1, 83.2; HRMS: (ESI, m/z) calcd for $C_{26}H_{22}O_2Na$ $[M+Na]^+$389.1512, found: 317.1512



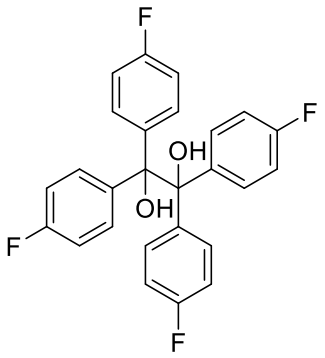
1,1,2,2-Tetra-*p*-tolylethane-1,2-diol (3p) (CAS: 913-86-0)[17]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: DCM), the product was isolated as a white solid (25.6 mg, 61% yield); ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.16 (d, *J* = 8.3 Hz, 8H), 6.98 (d, *J* = 8.2 Hz, 8H), 2.96 (br, 2H), 2.29 (s, 12H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 141.6, 136.4, 128.6, 128.1, 82.9, 21.1; HRMS: (ESI, m/z) calcd for $C_{30}H_{30}O_2Na$ $[M+Na]^+$ 445.2138, found: 445.2153



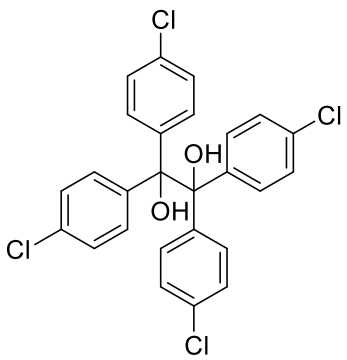
9H,9'H-[9,9'-bixanthene]-9,9'-diol (3q) (CAS: 6272-59-9)[18]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After column chromatography on silica gel (eluent: hexanes: EtOAc = 20:1 to 10:1), the product was isolated as a white solid (22.9 mg, 58% yield); ^{1}H NMR: (500 MHz, acetone-d_6, ppm): δ 7.28 (td, *J* = 7.2 Hz, 1.65 Hz, 4H), 7.23 (d, *J* = 7.2 Hz, 4H), 6.95 (t, *J* = 7.1 Hz, 4H), 6.79 (dd, J = 8.2 Hz, 1.0 Hz, 4H), 5.4 (br, 2H); ^{13}C NMR: (125 MHz, acetone-d_6, ppm): δ 152.6, 129.20, 129.17, 124.7, 122., 115.2, 75.7; HRMS: (ESI, m/z) calcd for $C_{26}H_{18}O_4Na$ $[M+Na]^+$ 417.1097, found: 417.1088



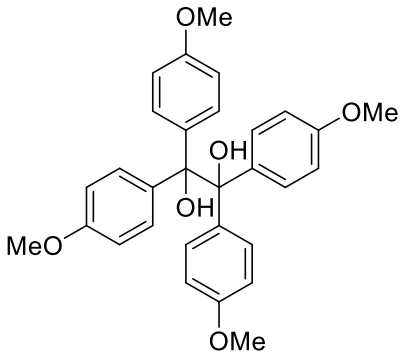
1,1,2,2-Tetrakis(4-fluorophenyl)ethane-1,2-diol (3r) (CAS: 424-82-8)[19]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 5: 1), the product was isolated as a white solid (33.5 mg, 76% yield); ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.29 – 7.26 (m, 8H), 6.92 – 6.88 (m, 8H), 2.89 (br, 2H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 161.9 (J_{C-F} = 247.3 Hz), 139.8 (J_{C-F} = 3.2 Hz), 130.4 (J_{C-F} = 8.0 Hz), 114.4 (J_{C-F} = 21.2 Hz), 82.7; ^{19}F NMR: (471 MHz, $CDCl_3$, ppm): δ -115.0; HRMS: (ESI, m/z) calcd for $C_{26}H_{18}F_4O_2Na$ $[M+Na]^+$ 461.1135, found: 461.1132



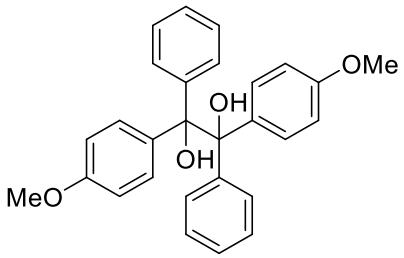
1,1,2,2-Tetrakis(4-chlorophenyl)ethane-1,2-diol (3s) (CAS: 5418-23-5)[20]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 10: 1), the product was isolated as a white solid (37.8 mg, 75% yield); ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.21 (d, *J* = 8.6 Hz, 8H), 7.16 (d, *J* = 8.9 Hz, 8H), 2.86 (br, 2H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 142.1, 133.6, 130.0, 127.9, 82.6; HRMS: (ESI, m/z) calcd for $C_{26}H_{18}Cl_4O_2Na$ $[M+Na]^+$ 524.9953, found: 524.9928



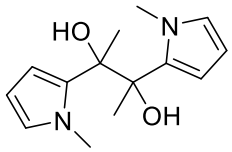
1,1,2,2-Tetrakis(4-methoxyphenyl)ethane-1,2-diol (3t) (CAS: 19920-00-4)[21]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After column chromatography on silica gel (eluent: hexanes: EtOAc = 10:1 to 5:1), the product was isolated as a colorless oil (41.0 mg, 84% yield); ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.19 (d, *J* = 8.9 Hz, 8H), 6.70 (d, *J* = 8.9 Hz, 8H), 3.76 (s, 12H), 2.91 (br, 2H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 158.3, 136.8, 130.0, 112.6, 82.8, 55.2; HRMS: (ESI, m/z) calcd for $C_{30}H_{30}O_6Na$ $[M+Na]^+$ 509.1935, found: 509.1946



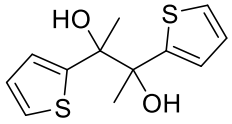
1,2-Bis(4-methoxyphenyl)-1,2-diphenylethane-1,2-diol (3u) (CAS: 2443-45-0)[22]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After column chromatography on silica gel (eluent: hexanes: EtOAc = 10:1 to 5:1), the product was isolated as a colorless oil (37.4 mg, 84% yield), d.r. = 1: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.34 – 7,30 (m, 4H), 7.20 – 7.16 (m, 10H), 6.71 (t, *J* = 9.3 Hz, 4H), 3.77 (s, 3H), 3.75 (s, 3H), 2.98 (br, 2H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 158.5, 158.4, 144.6, 136.5, 130.04, 129.97, 128.73, 128.66, 127.38, 127.37, 127.0, 126.9, 83.0, 55.3, 55.2; HRMS: (ESI, m/z) calcd for $C_{28}H_{26}O_4Na$ $[M+Na]^+$ 449.1723, found: 449.1736



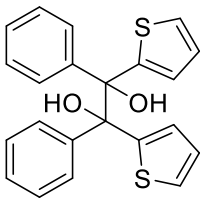
2,3-Bis(1-methyl-1*H*-pyrrol-2-yl)butane-2,3-diol (3v):

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 5: 1), the product was isolated as a white solid (7.4 mg, 30% yield), *dl : meso* = 1: 1.2; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 6.48 (t, *J* = 2.9 Hz, 1H), 6.45 (t, *J* = 2.3 Hz, 1H), 6.05 – 6.00 (m, 4H), 3.22 (s, 3.6H), 3.12 (s, 3.1H), 2.58 (br, 1H), 2.27 (br, 1.2H), 1.66 (s, 3.7H, *meso*), 1.64 (s, 3.3H, *dl*); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 133.7, 133.5, 125.1, 124.8, 109.7, 109.4, 106.4, 106.3, 78.6, 78.4, 36.6, 36.3, 26.4, 26.1; HRMS: (ESI, m/z) calcd for $C_{14}H_{20}O_2N_2Na$ $[M+Na]^+$ 271.1417, found: 271.1417



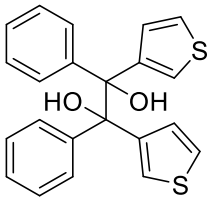
2,3-Di(thiophen-2-yl)butane-2,3-diol (3w) (CAS: 13196-16-2)[23]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 10: 1), the product was isolated as a colorless solid (5.7 mg, 20% yield), *dl : meso* = 1.6: 1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.24 – 7.19 (m, 2H), 6.96 – 6.92 (m, 2H), 6.85 – 6.81 (m, 2H), 2.81 (br, 1.2H), 2.67 (br, 0.8H), 1.69 (s, 2.3H, *meso*), 1.64 (s, 3.7H, *dl*); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 148.7, 148.3, 126.5, 126.4, 125.5, 125.0, 124.8, 124.8, 78.8, 78.6, 26.4, 26.2; HRMS: (ESI, m/z) calcd for $C_{12}H_{14}O_2S_2Na$ $[M+Na]^+$ 277.0327, found: 277.0329



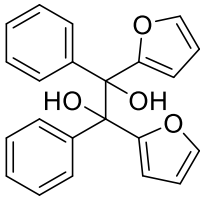
1,2-Diphenyl-1,2-di(thiophen-2-yl)ethane-1,2-diol (3x) (CAS: 54364-08-8)[24]:

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 10: 1), the product was isolated as a white solid (8.7 mg, 23% yield), d.r. = 1: 1.3; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.40 – 7.36 (m, 4H), 7.04 – 6.92 (m, 4H), 6.85 – 6.81 (m, 2H), 3.43 (br, 0.9H), 3.30 (br, 1.1H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 148.5, 142.0, 128.4, 128.0, 127.9, 127.8, 127.6, 127.4, 127.3, 126.7, 126.5, 126.1, 125.8, 83.5, 83.2; HRMS: (ESI, m/z) calcd for $C_{22}H_{18}O_2S_2Na$ $[M+Na]^+$ 401.0651, found: 401.0639



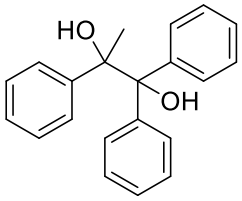
1,2-Diphenyl-1,2-di(thiophen-3-yl)ethane-1,2-diol (3y):

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 20: 1), the product was isolated as a white solid (15.5 mg, 41% yield), d.r. = 1: 1.1; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.34 – 7.32 (m, 2H), 7.22 – 7.16 (m, 11H), 7.09 – 7.00 (m, 3H), 3.04 (br, 0.9H), 3.30 (br, 1H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 145.5, 142.9, 128.7, 128.6, 128.3, 128.0, 127.46, 127.43, 127.4, 124.7, 124.6, 124.1, 123.7, 82.8, 82.6; HRMS: (ESI, m/z) calcd for $C_{22}H_{18}O_2S_2Na$ $[M+Na]^+$ 401.0651, found: 401.0643



1,2-Di(furan-2-yl)-1,2-diphenylethane-1,2-diol (3z) (CAS: 52056-41-4)[25]:

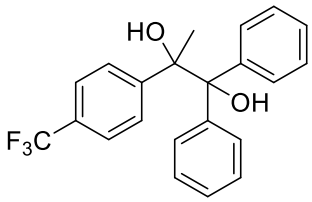
Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 20: 1), the product was isolated as a white solid (19.6 mg, 57% yield), d.r. = 1: 1.3; ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.37 – 7.35 (m, 2H), 7.25 – 7.13 (m, 10H), 6.37 – 6.30 (m, 3H), 6.14 (d, *J* = 3.3 Hz, 1H), 3.81 (br, 0.9H), 3.71 (br, 1.2H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 157.0, 155.8, 141.9, 141.7, 140.1, 139.8, 128.0, 127.9, 127.8, 127.6, 127.2, 127.0, 110.63, 110.59, 110.1, 108.8, 80.60, 80.58; HRMS: (ESI, m/z) calcd for $C_{22}H_{18}O_4Na$ $[M+Na]^+$ 369.1097, found: 369.1089



1,1,2-Triphenylpropane-1,2-diol (3aa) (CAS: 3784-22-3)[26]:

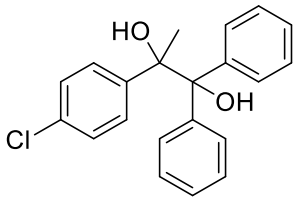
Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 20: 1), the product was isolated as a colorless oil (5.1 mg, 17% yield); ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.67 (d, *J* = 7.4 Hz, 2H), 7.55 – 7.53 (m, 2H), 7.30 – 7.18 (m, 11H), 2.83 (br, 1H), 2.43 (br, 1H), 1.72 (s, 3H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 144.6, 144.0, 143.8, 128.31, 128.26, 127.7,

127.52, 127.47, 127.18, 127.17, 127.13, 82.1, 80.5, 27.0; HRMS: (ESI, m/z) calcd for $C_{21}H_{20}O_2Na$ $[M+Na]^+$ 327.1356, found: 327.1365



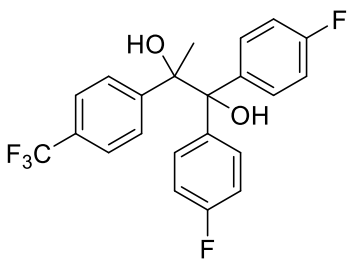
1,1-Diphenyl-2-(4-(trifluoromethyl)phenyl)propane-1,2-diol (3ab):

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 20: 1), the product was isolated as a colorless oil (14.2 mg, 38% yield); ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.62 (d, *J* = 7.2 Hz, 2H), 7.53 – 7.51 (m, 2H), 7.44 – 7.43 (m, 4H), 7.32 – 7.27 (m, 3H), 7.23 – 7.18 (m, 3H), 2.80 (br, 1H), 2.53 (br, 1H), 1.72 (s, 3H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 149.1, 143.8, 143.5, 129.0 (q, *J* = 32.4 Hz), 128.30, 128.27, 127.9, 127.84, 127.75, 127.5, 127.4, 124.3 (q, *J* = 271.4 Hz), 124.2 (q, *J* = 3.7 Hz), 82.5, 80.2, 27.2; ^{19}F NMR: (471 MHz, $CDCl_3$, ppm): δ -62.4; HRMS: (ESI, m/z) calcd for $C_{22}H_{19}O_2F_3Na$ $[M+Na]^+$ 395.1229, found: 395.1234



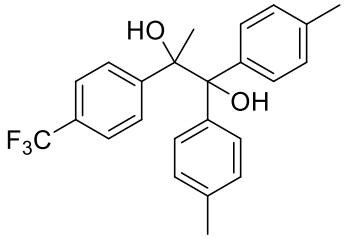
2-(4-Chlorophenyl)-1,1-diphenylpropane-1,2-diol (3ac):

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 20: 1), the product was isolated as a colorless oil (11.7 mg, 35% yield); ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.63 (d, *J* = 7.4 Hz, 2H), 7.53 – 7.51 (m, 2H), 7.31 – 7.13 (m, 10H), 2.76 (br, 1H), 2.44 (br, 1H), 1.69 (s, 3H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 143.9, 143.6, 143.4, 133.0, 129.0, 128.3, 127.8, 127.7, 127.44, 127.39, 127.3, 82.3, 80.1, 27.1; HRMS: (ESI, m/z) calcd for $C_{21}H_{19}O_2ClNa$ $[M+Na]^+$361.0966, found: 361.0971



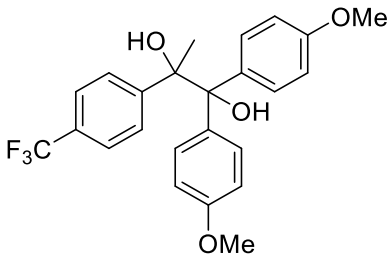
1,1-Bis(4-fluorophenyl)-2-(4-(trifluoromethyl)phenyl)propane-1,2-diol (3ad):

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 20: 1), the product was isolated as a colorless oil (14.1 mg, 36% yield); ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.62 – 7.60 (m, 2H), 7.49 – 7.39 (m, 6H), 6.99 (t, *J* = 8.7 Hz, 2H), 6.89 (t, *J* = 8.7 Hz, 2H), 2.72 (br, 1H), 2.38 (br, 1H), 1.70 (s, 3H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 162.1 (d, *J* = 247.3 Hz), 162.0 (d, *J* = 247.3 Hz), 148.7, 139.5 (d, *J* = 3.1 Hz), 139.3 (d, *J* = 3.3 Hz), 130.1, 130.0, 129.4 (q, *J* = 32.6 Hz), 127.9, 124. 4 (q, *J* = 3.7 Hz), 124.2 (q, *J* = 271.9 Hz), 114.7 (d, *J* = 17.3 Hz), 114.5 (d, *J* = 17.3 Hz), 81.7, 80.3, 27.1; ^{19}F NMR: (471 MHz, $CDCl_3$, ppm): δ -62.5, -115.1, -115.2; HRMS: (ESI, m/z) calcd for $C_{22}H_{17}O_2F_5Na$ $[M+Na]^+$431.1041, found: 431.1056



1,1-Di-*p*-tolyl-2-(4-(trifluoromethyl)phenyl)propane-1,2-diol (3ae):

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 20: 1), the product was isolated as a colorless oil (15.0 mg, 39% yield); ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.46 – 7.42 (m, 6H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.10 (d, *J* = 8.1 Hz, 2H), 7.00 (d, *J* = 8.1 Hz, 2H), 2.72 (br, 1H), 2.52 (br, 1H), 2.33 (s, 3H), 2.28 (s, 3H), 1.69 (s, 3H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 149.3, 141.0, 140.7, 137.1, 137.0, 128.9 (q, *J* = 32.4 Hz), 128.8, 128.5, 128.4, 128.2, 127.9, 127.6, 124.4 (q, *J* = 271.9 Hz), 124.1 (q, *J* = 3.7 Hz), 82.4, 80.1, 27.3, 21.09, 21.05; ^{19}F NMR: (471 MHz, $CDCl_3$, ppm): δ -62.4; HRMS: (ESI, m/z) calcd for $C_{24}H_{23}O_2F_3Na$ $[M+Na]^+$423.1542, found: 423.1556



1-(4-Hydroxyphenyl)-1-(4-methoxyphenyl)-2-(4-(trifluoromethyl)phenyl)propane-1,2-diol (3af):

Following the general procedure, the reaction mixture was diluted by the EtOAc. After preparative TLC isolation (eluent: hexanes: EtOAc = 10: 1), the product was isolated as a colorless oil (8.9 mg, 20% yield); ^{1}H NMR: (500 MHz, $CDCl_3$, ppm): δ 7.48 – 7.38 (m, 8H), 6.81 (d, *J* = 9.0 Hz, 2H), 6.73 (d, *J* = 9.0 Hz, 2H), 3.80 (s, 3H), 3.76 (s, 3H), 2.68 (br, 1H), 2.53 (br, 1H), 1.69 (s, 3H); ^{13}C NMR: (125 MHz, $CDCl_3$, ppm): δ 158.8, 158.7, 149.3,

136.1, 135.9, 129.6, 129.5, 129.0 (q, J = 32.1 Hz), 127.9, 124.4 (q, J = 271.9 Hz), 124.1 (q, J = 3.7 Hz), 113.1, 113.0, 82.2, 80.3, 55.4, 55.3, 27.1; ^{19}F NMR: (471 MHz, $CDCl_3$, ppm): δ -62.4; HRMS: (ESI, m/z) calcd for $C_{24}H_{23}O_4F_3Na$ $[M+Na]^+$455.1441, found: 455.1460

18. References

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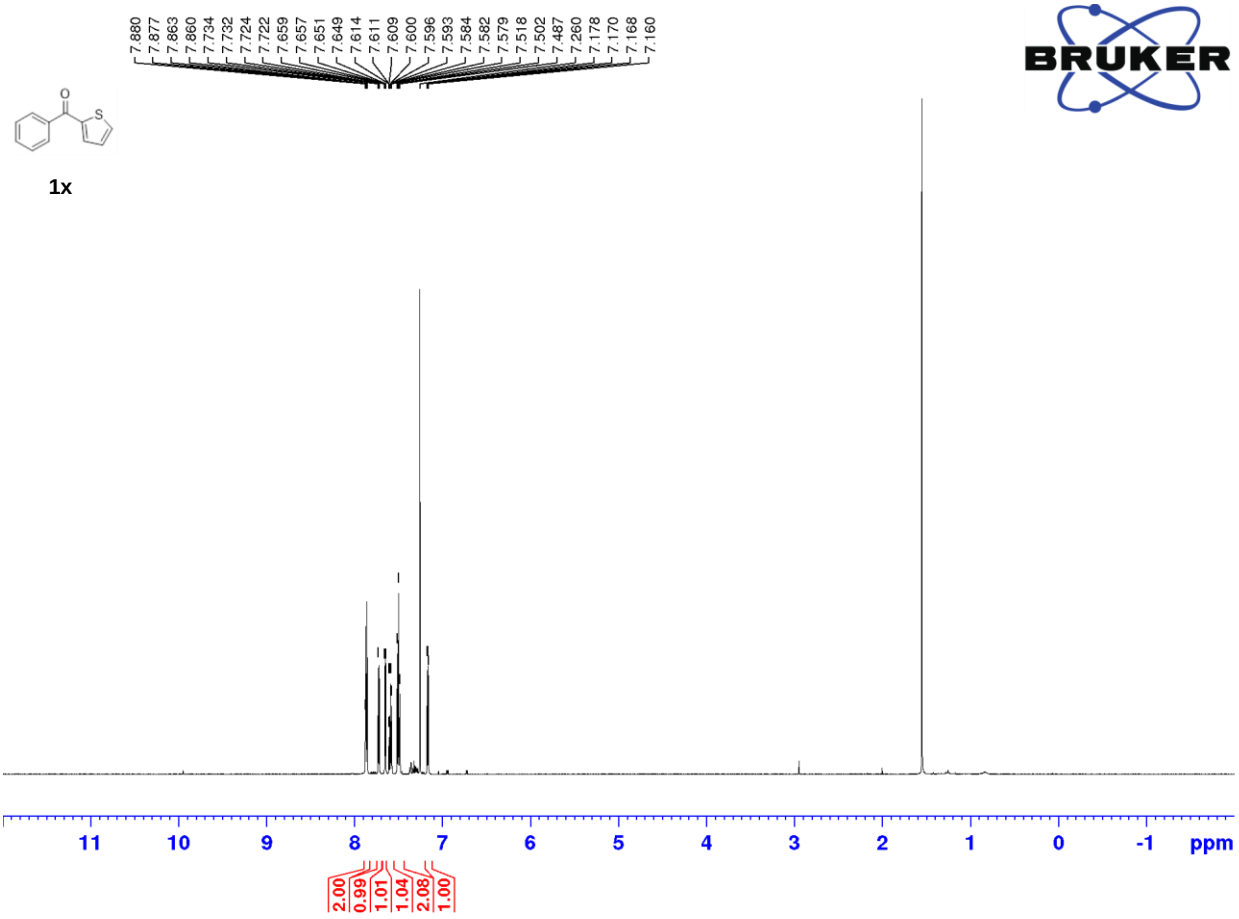
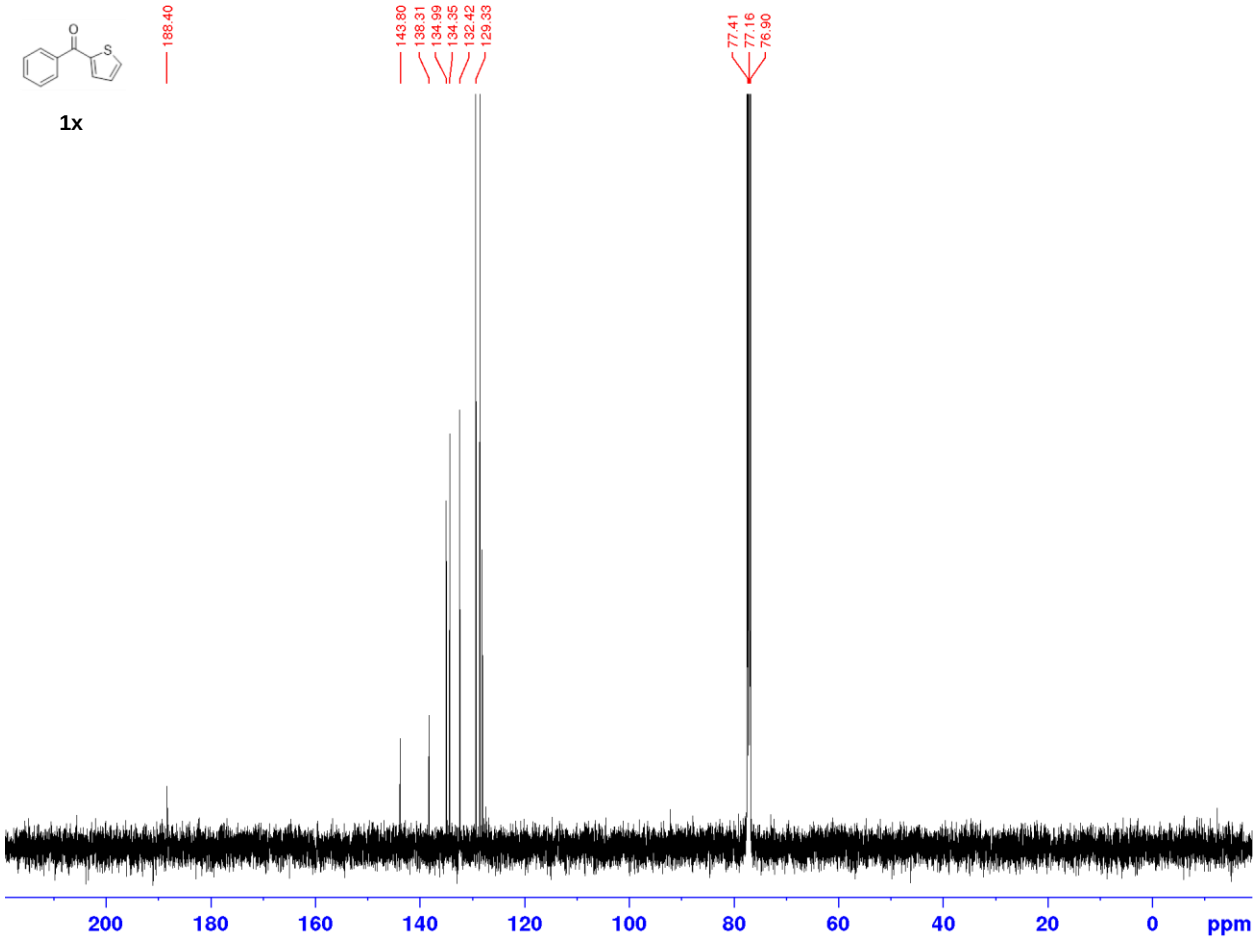
19. NMR spectra of synthesized heteroaromatic ketones

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140
120
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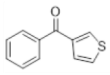
ppm



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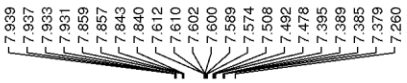
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7.937
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7.859
7.857
7.843
7.840
7.612
7.610
7.602
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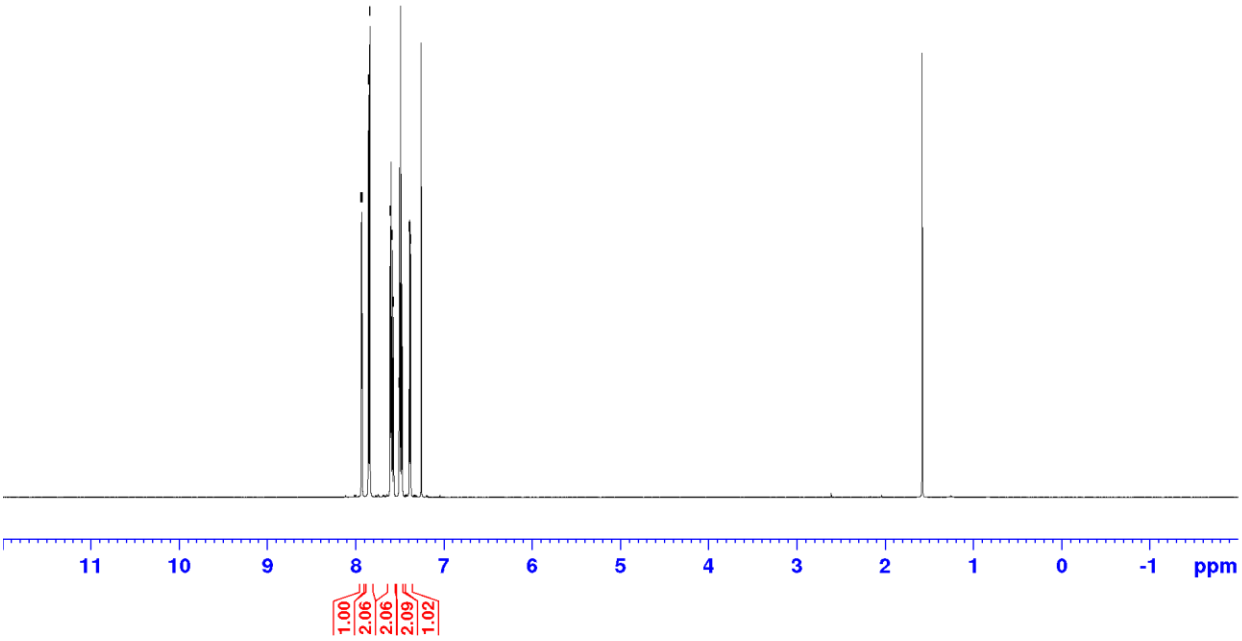
7.478
7.395
7.389
7.385
7.379
7.260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ppm
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2.06
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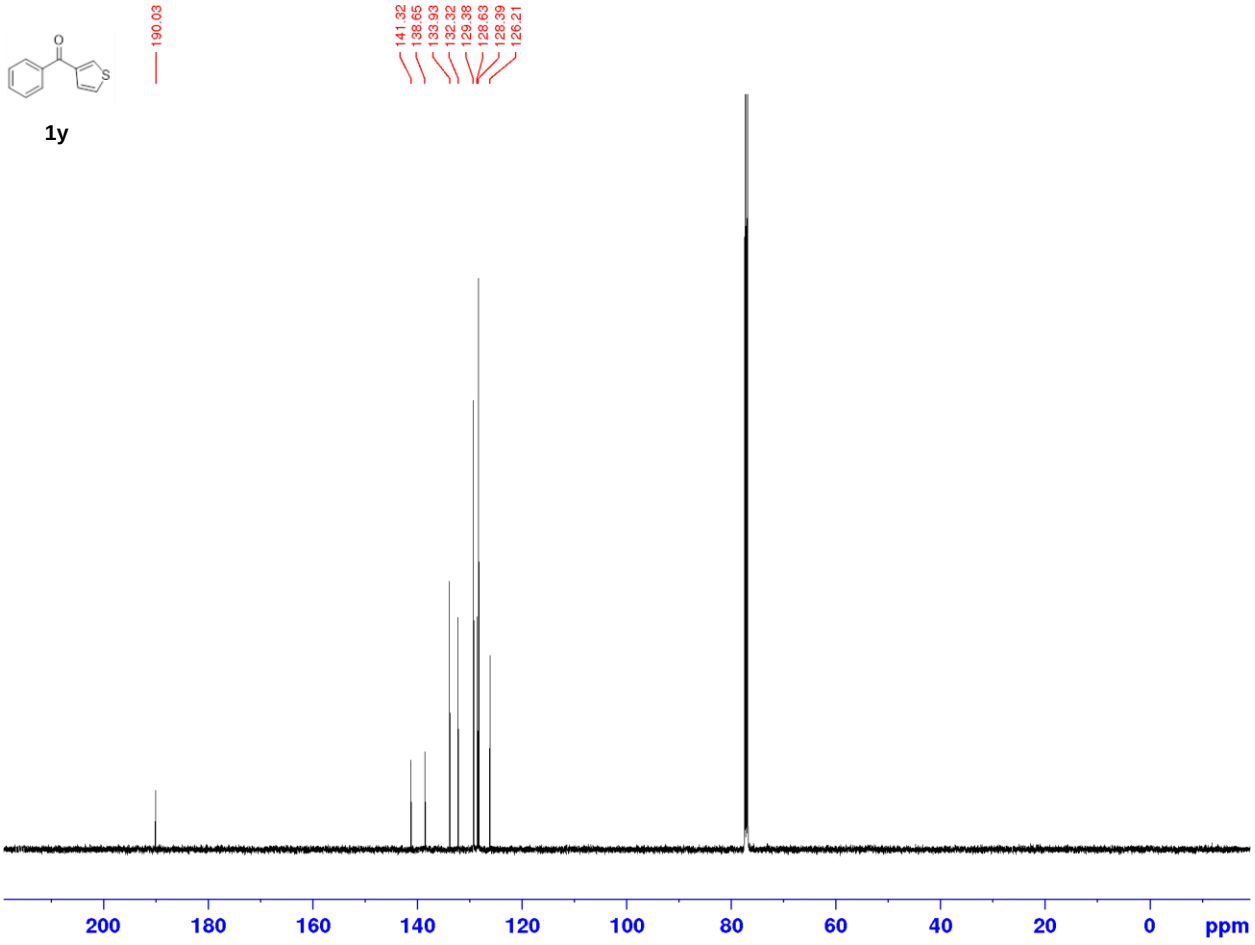
1.02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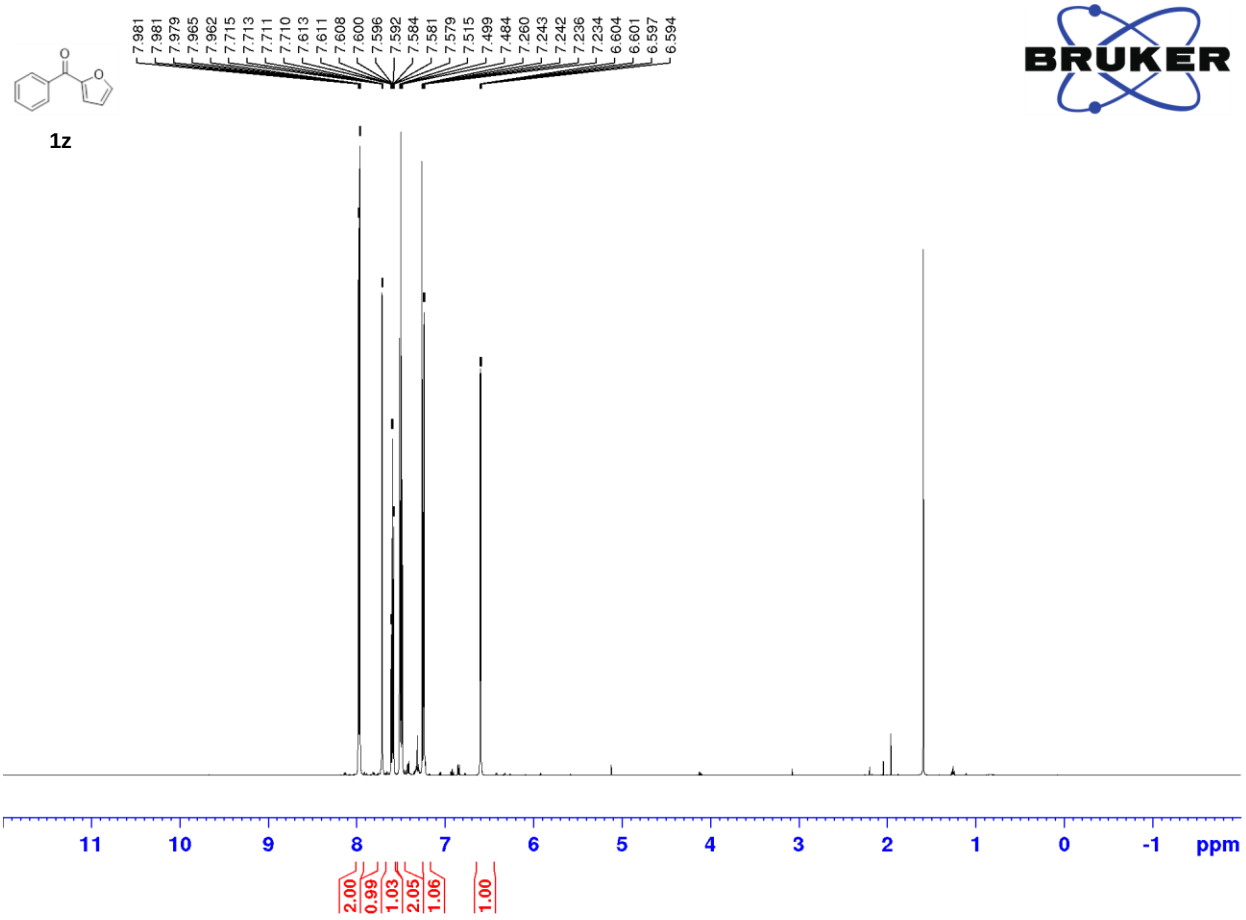
ppm

1y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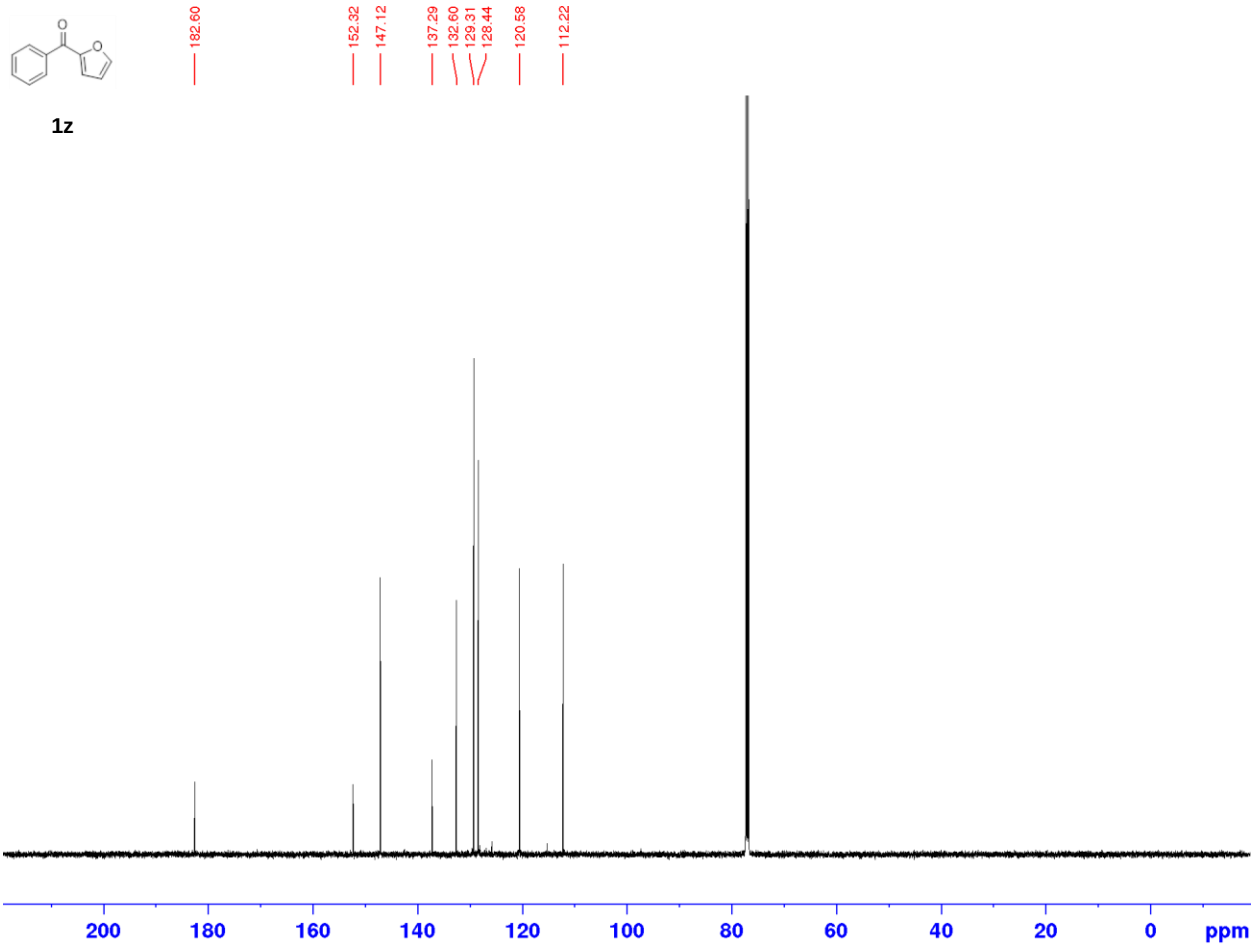
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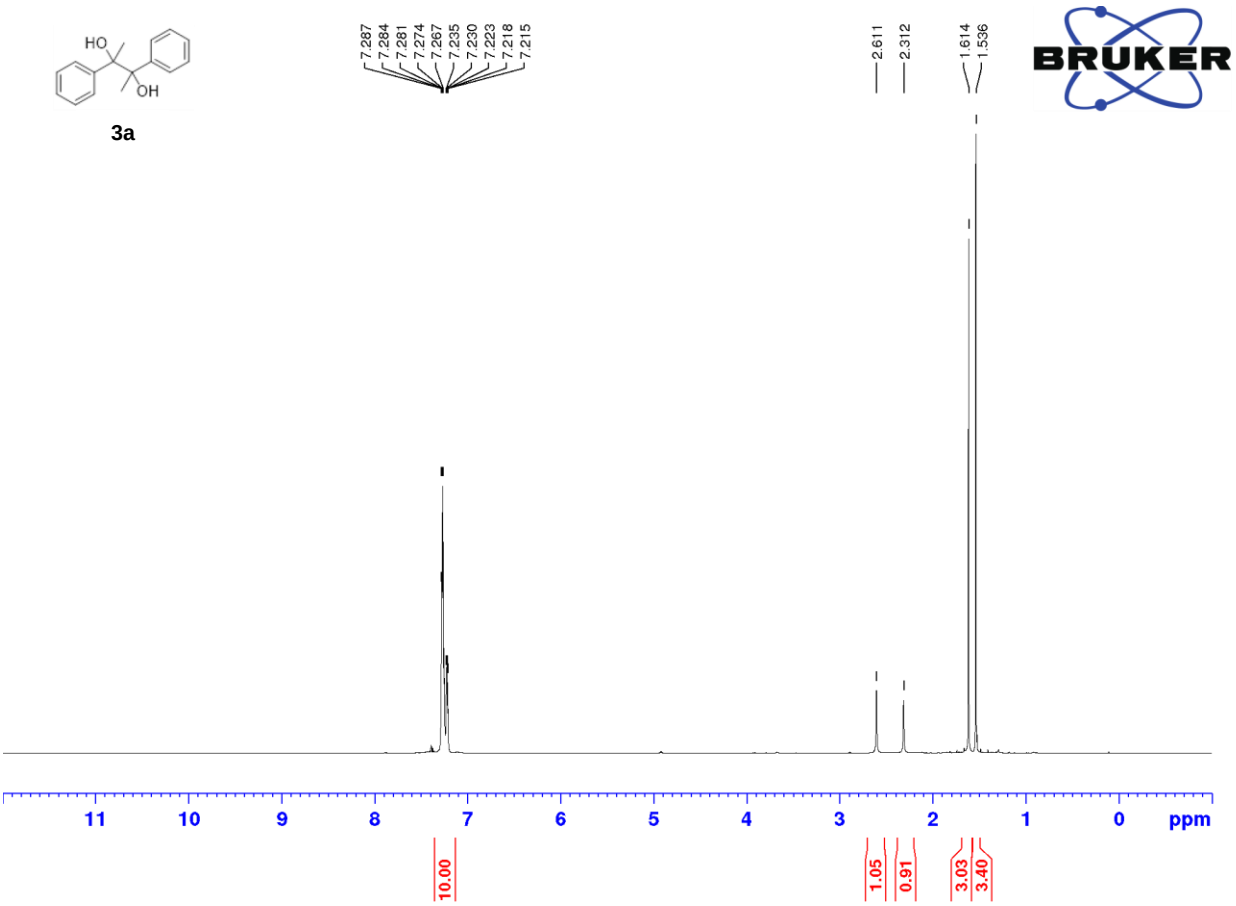
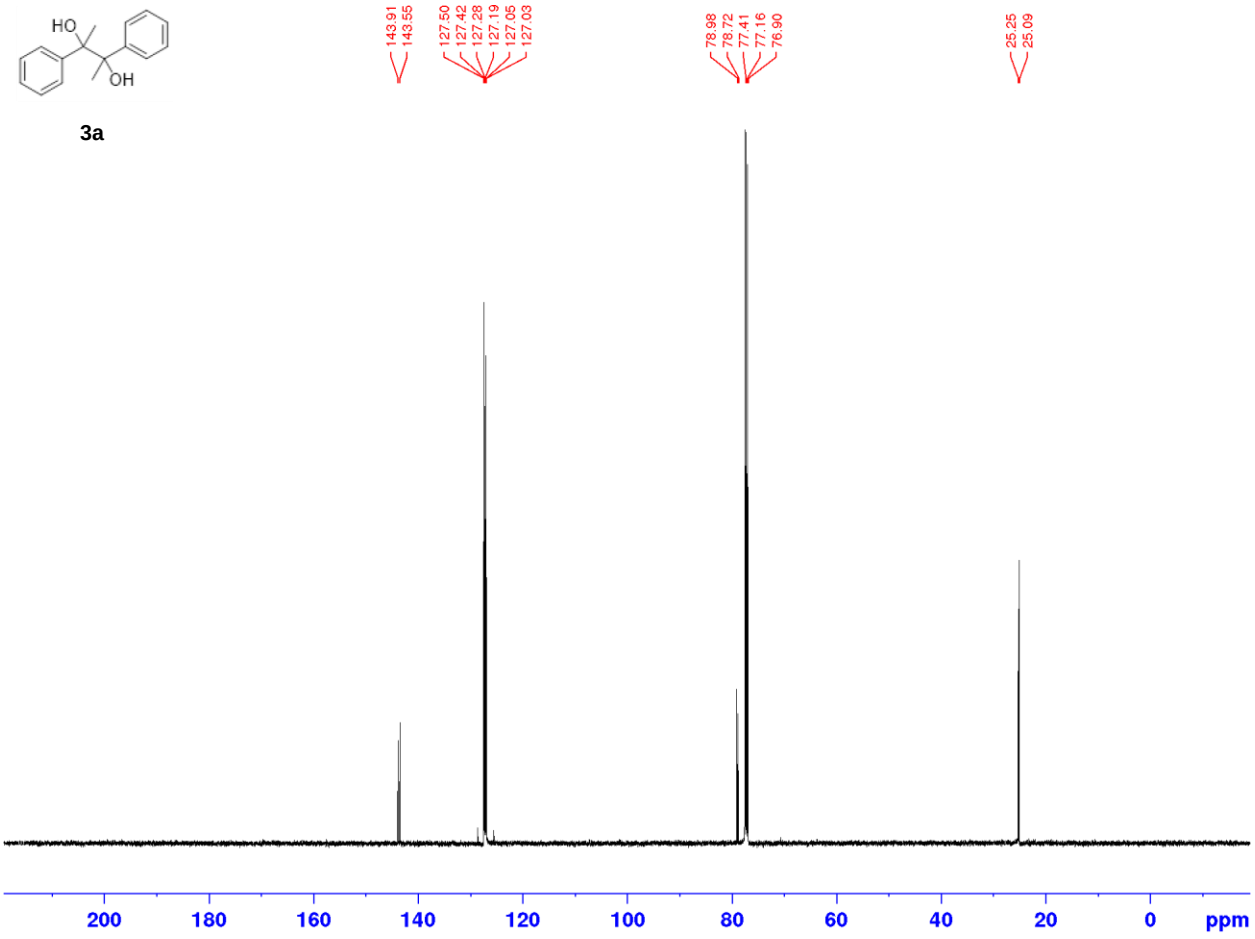
0
ppm

20. NMR spectra of products

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25.09
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0
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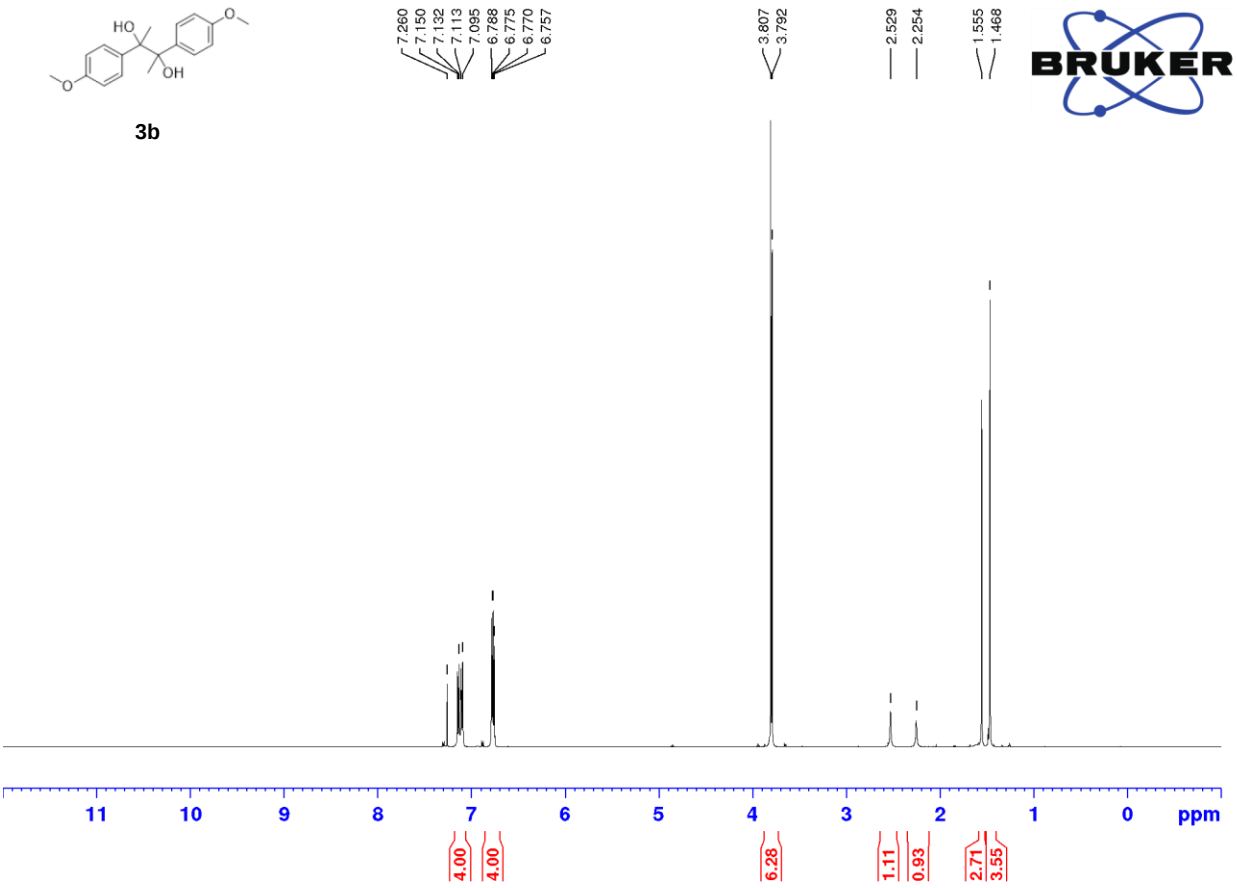
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3.792
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1.468
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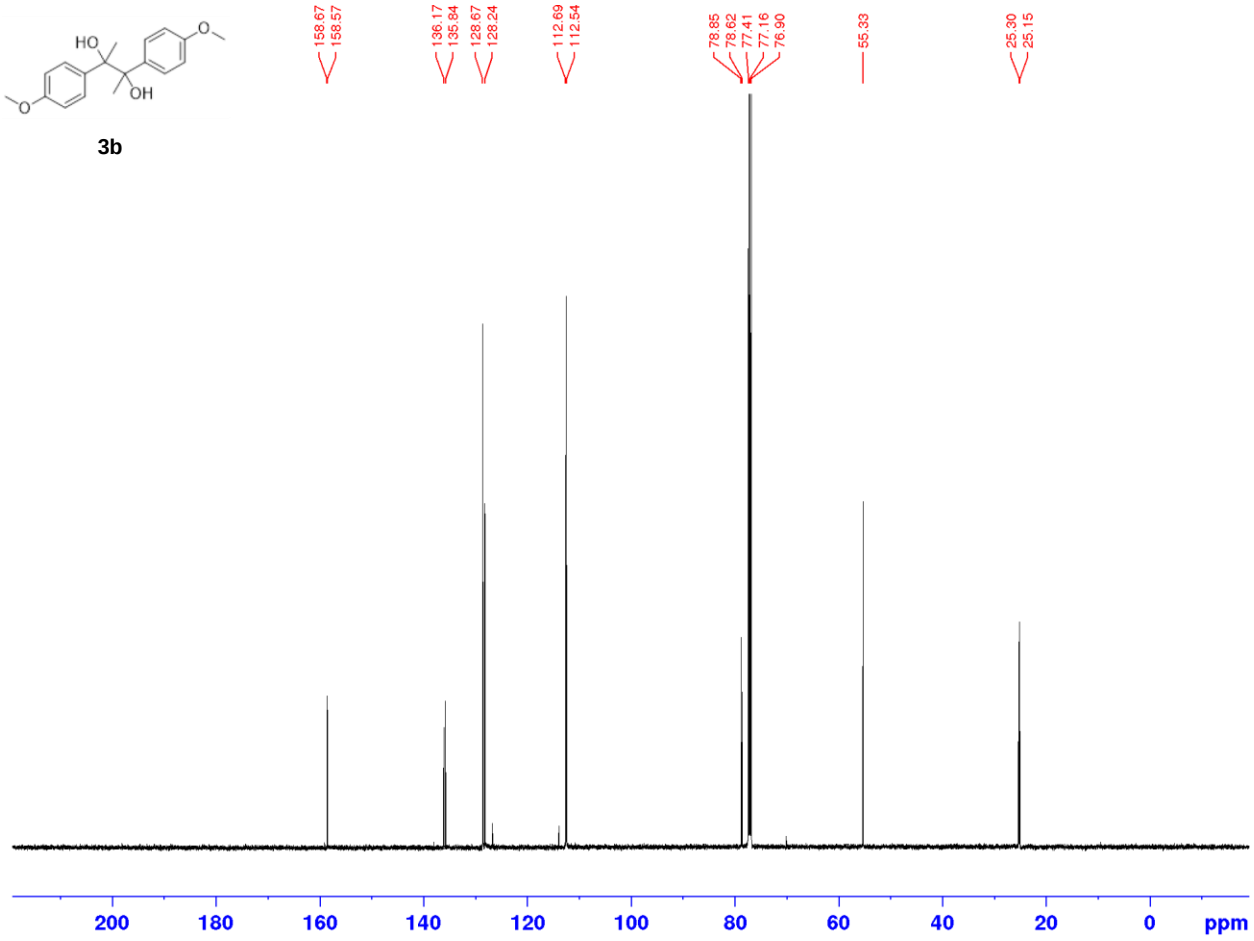
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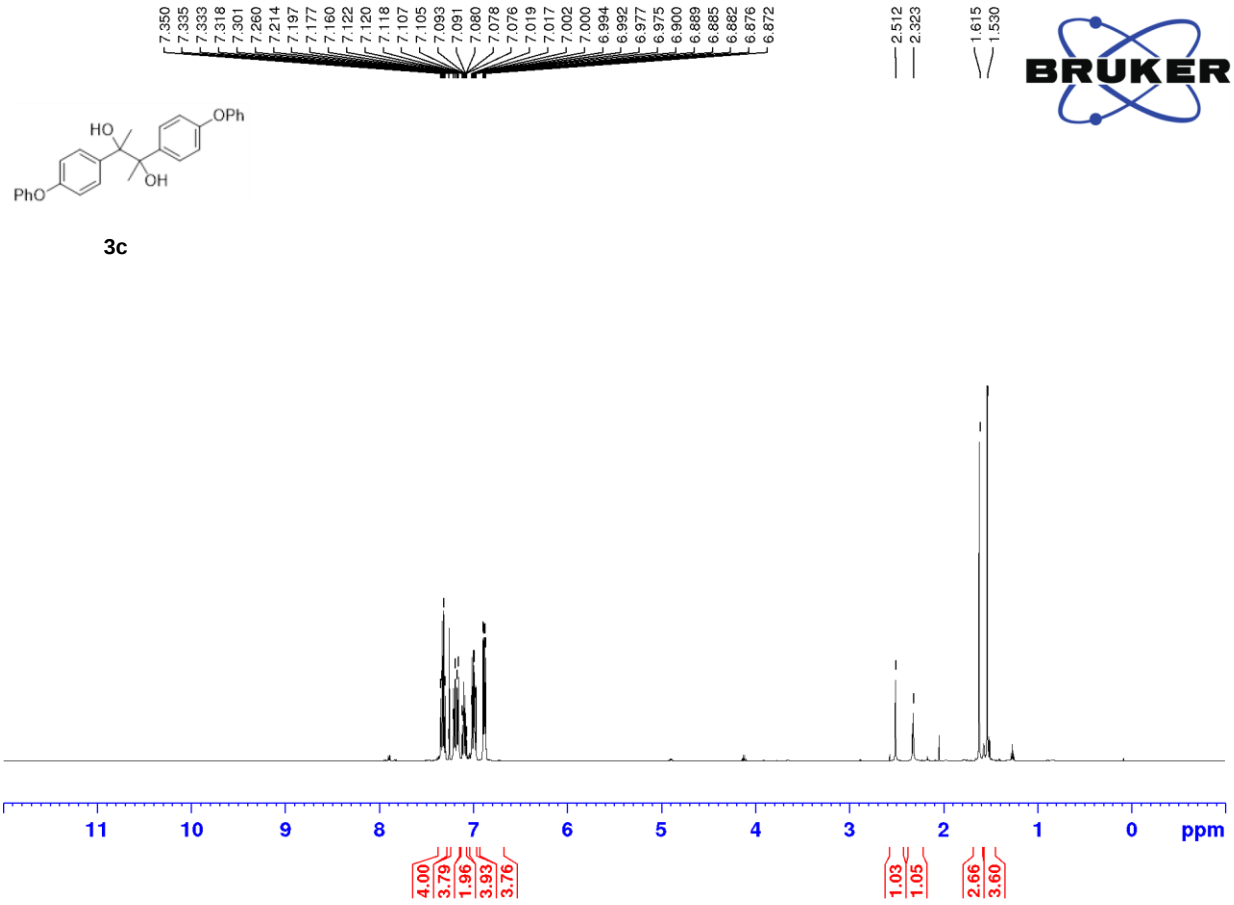
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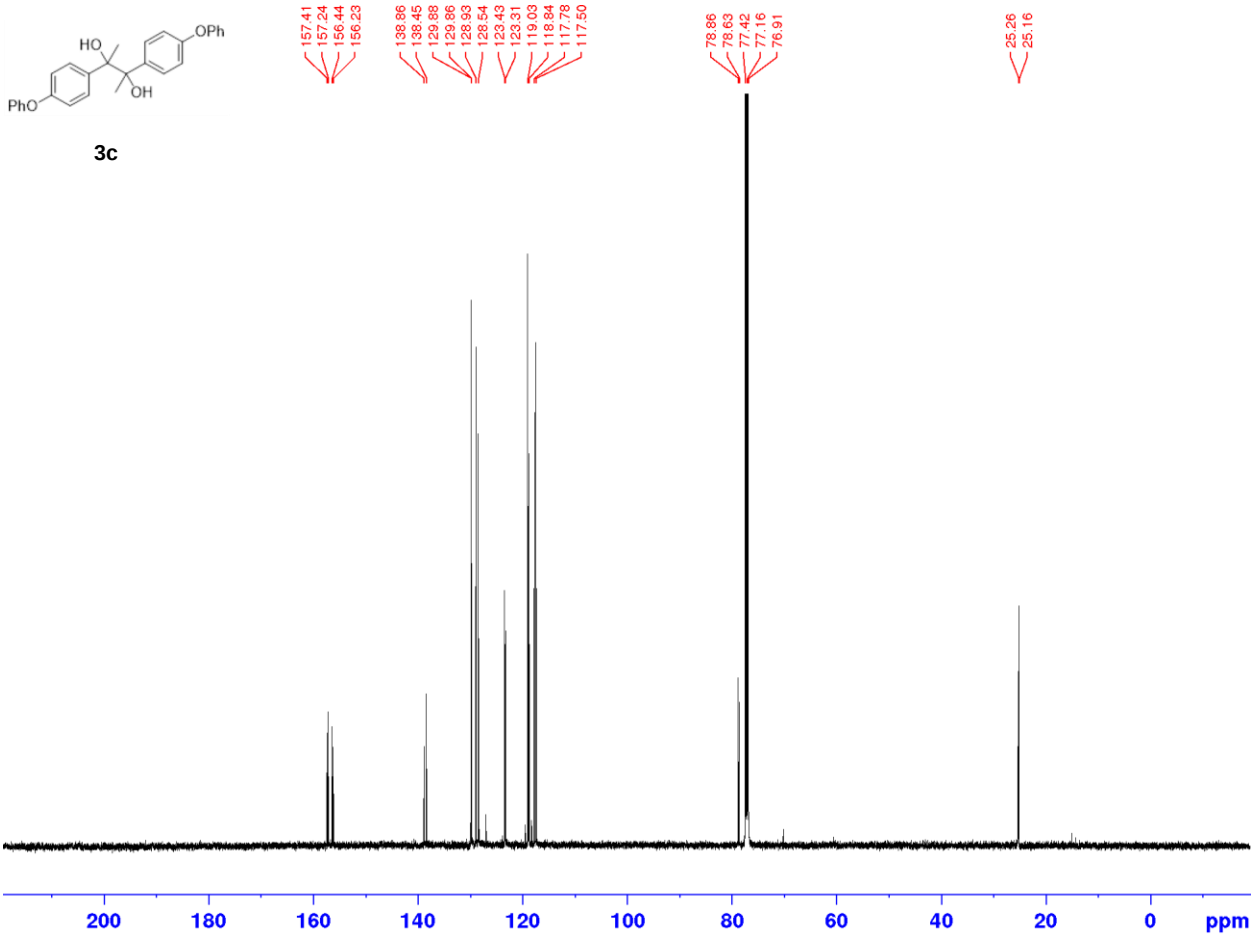
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PhO
OH
157.41
157.24
156.44
156.23
138.86
138.45
129.88
129.86
128.93
128.54
123.43
123.31
119.03
118.84
117.78
117.50
78.86
78.63
77.42
77.16
76.91
25.26
25.16
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180
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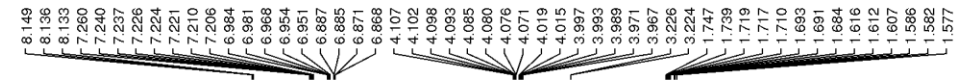
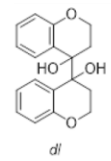
140
120
100
80
60
40
20
0
ppm

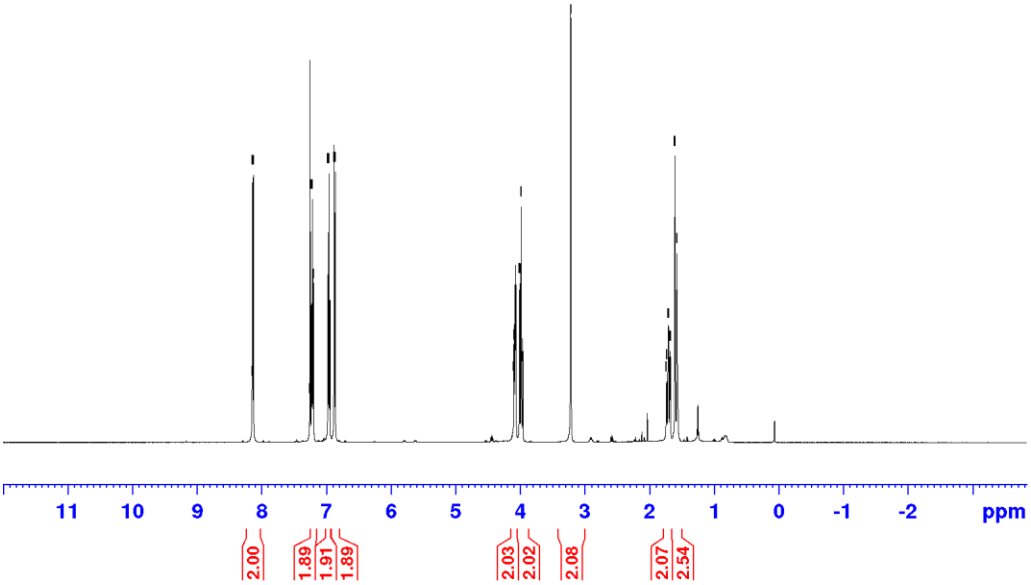
3c

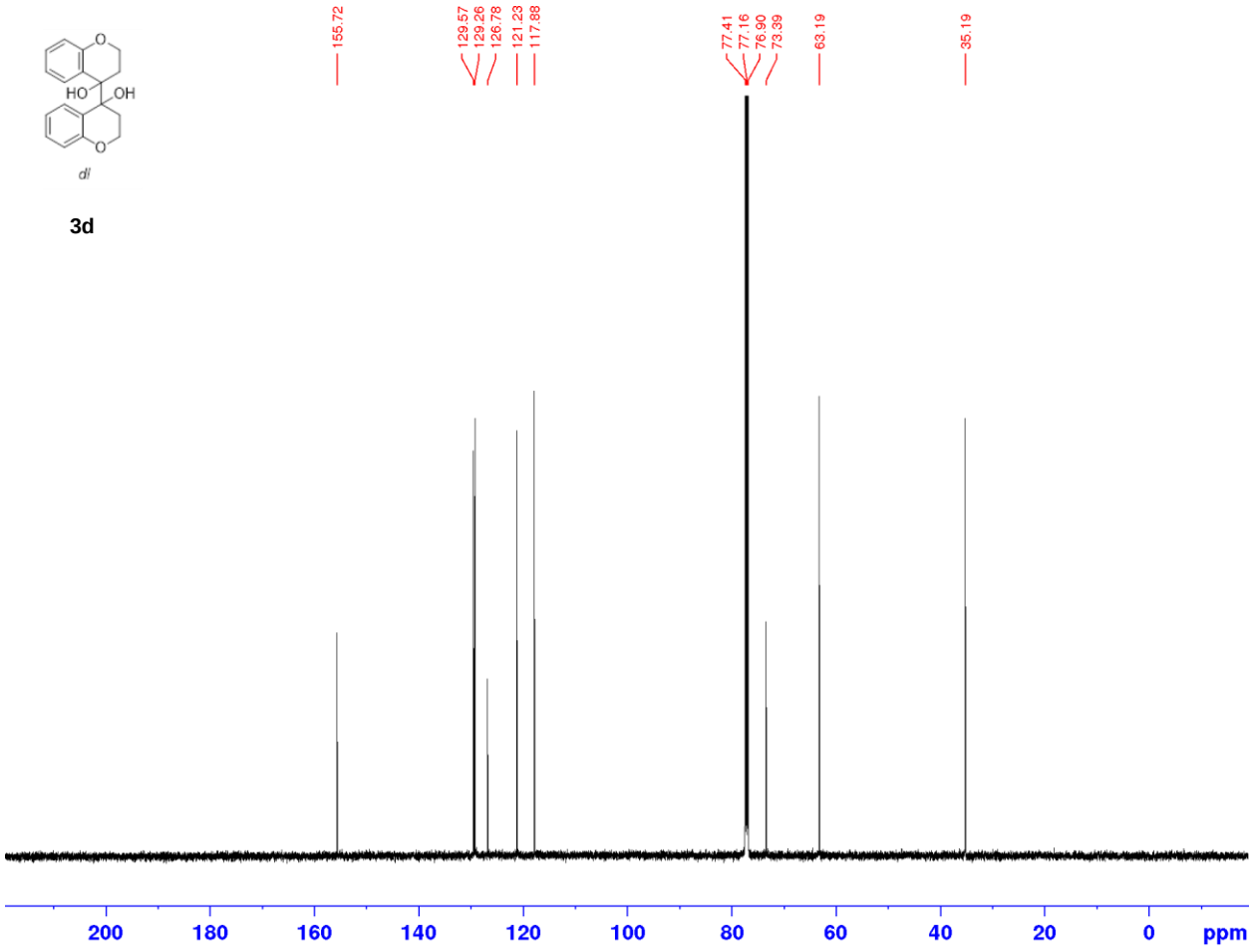

8.149
8.136
8.133
7.260
7.240
7.237
7.226
7.224
7.221
7.210
7.206
6.984
6.981
6.968
6.954
6.951
6.887
6.885
6.871
6.868
4.107
4.102
4.098
4.093
4.085
4.080
4.076
4.071
4.019
4.015
3.997
3.993
3.989
3.971
3.967
3.226
3.224
1.747
1.739
1.719
1.717
1.710
1.693
1.691
1.684
1.616
1.612
1.607
1.586
1.582
1.577

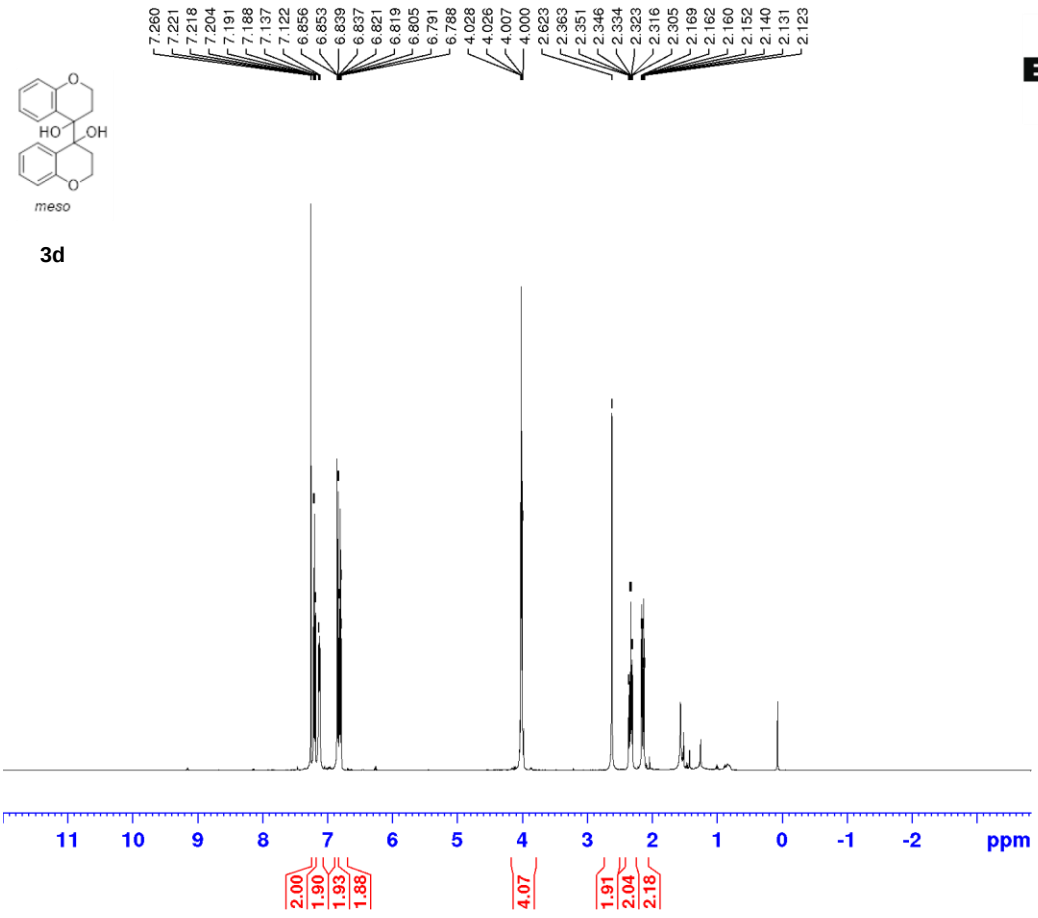

HO
OH
O
O

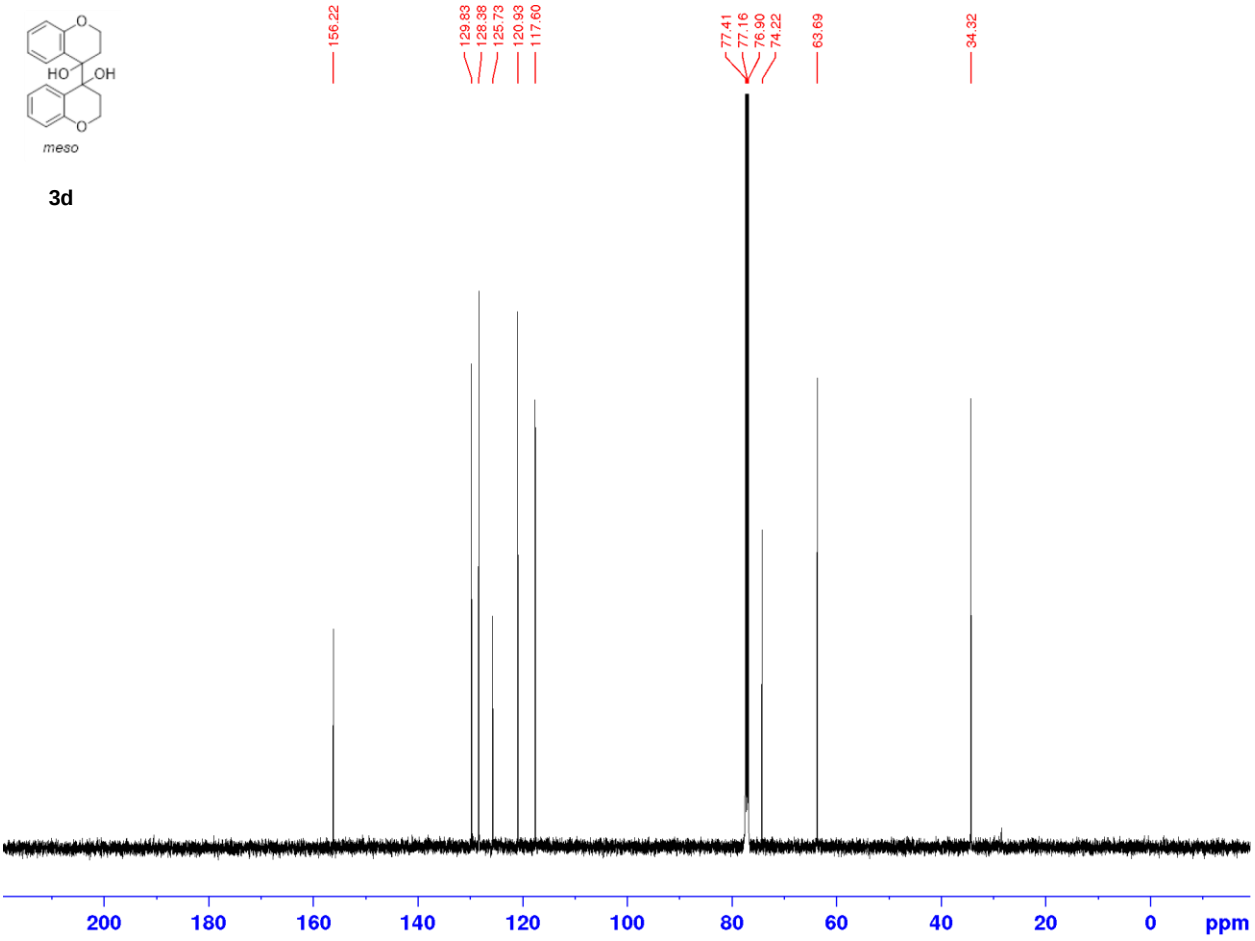
di

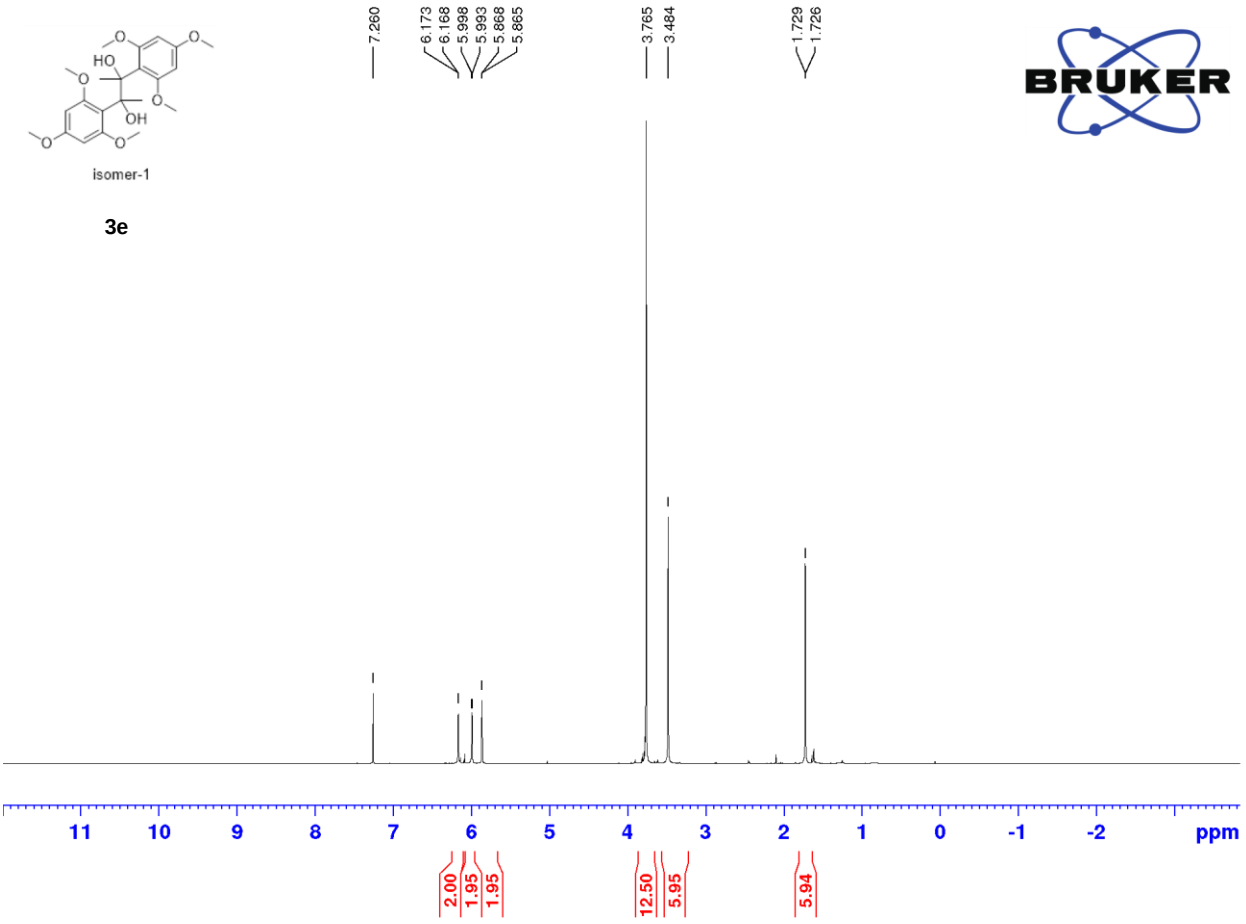
3d

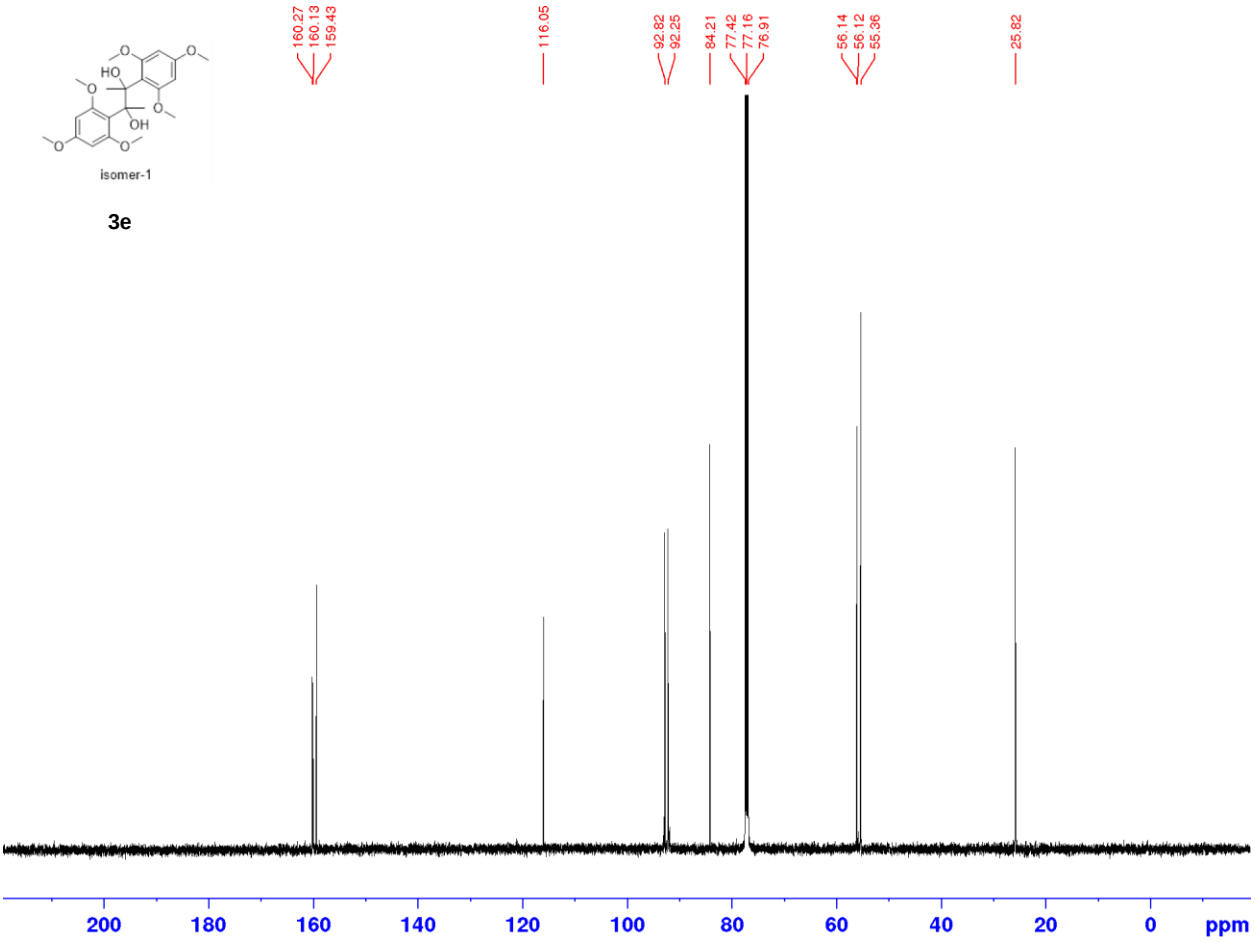

11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
2.00
1.89
1.91
1.89
2.03
2.02
2.08
2.07
2.54


155.72
129.57
129.26
126.78
121.23
117.88
77.41
77.16
76.90
73.39
63.19
35.19
HO
OH
O
O
dl
3d
200
180
160
140
120
100
80
60
40
20
0
ppm

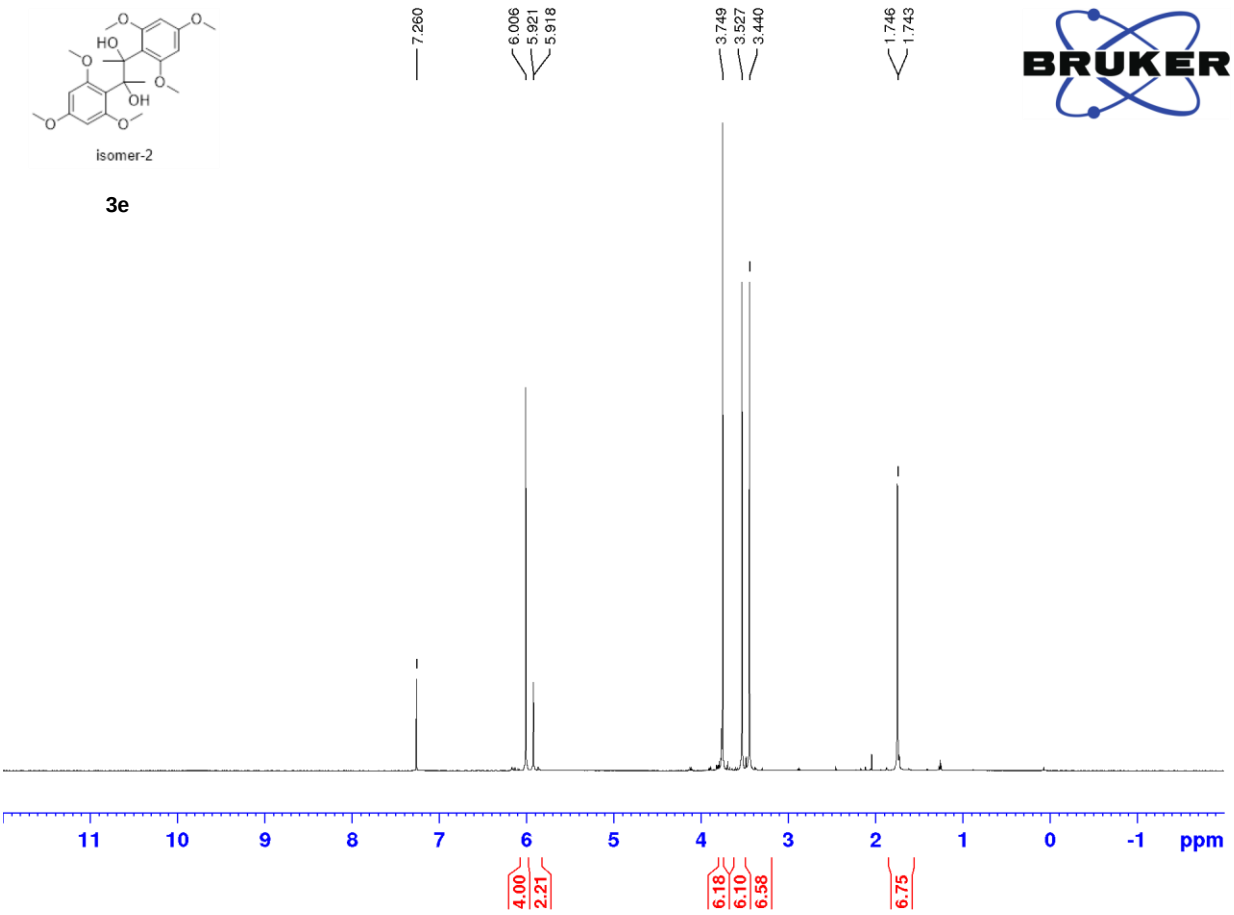

7.260
7.221
7.218
7.204
7.191
7.188
7.137
7.122
6.856
6.853
6.839
6.837
6.821
6.819
6.805
6.791
6.788
4.028
4.026
4.007
4.000
2.623
2.363
2.351
2.346
2.334
2.323
2.316
2.305
2.169
2.162
2.160
2.152
2.140
2.131
2.123
HO
OH
O
O
meso
3d
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
2.00
1.90
1.93
1.88
4.07
1.91
2.04
2.18


meso
3d
156.22
129.83
128.38
125.73
120.93
117.60
77.41
77.16
76.90
74.22
63.69
34.32
200
180
160
140
120
100
80
60
40
20
0
ppm

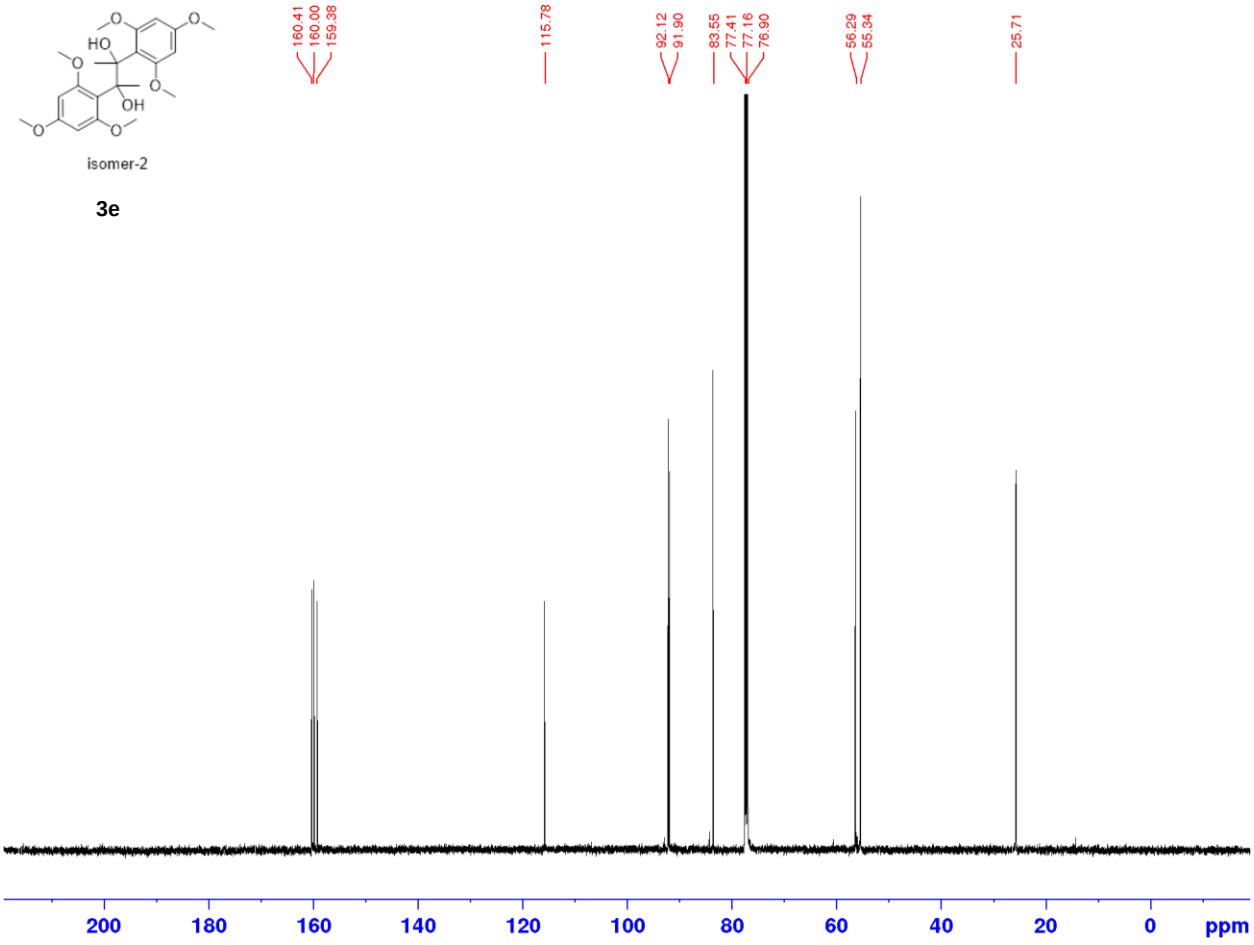

isomer-1
3e
7.260
6.173
6.168
5.998
5.993
5.868
5.865
3.765
3.484
1.729
1.726
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
2.00
1.95
1.95
12.50
5.95
5.94


isomer-1
160.27
160.13
159.43
116.05
92.82
92.25
84.21
77.42
77.16
76.91
56.14
56.12
55.36
25.82
200
180
160
140
120
100
80
60
40
20
0
ppm

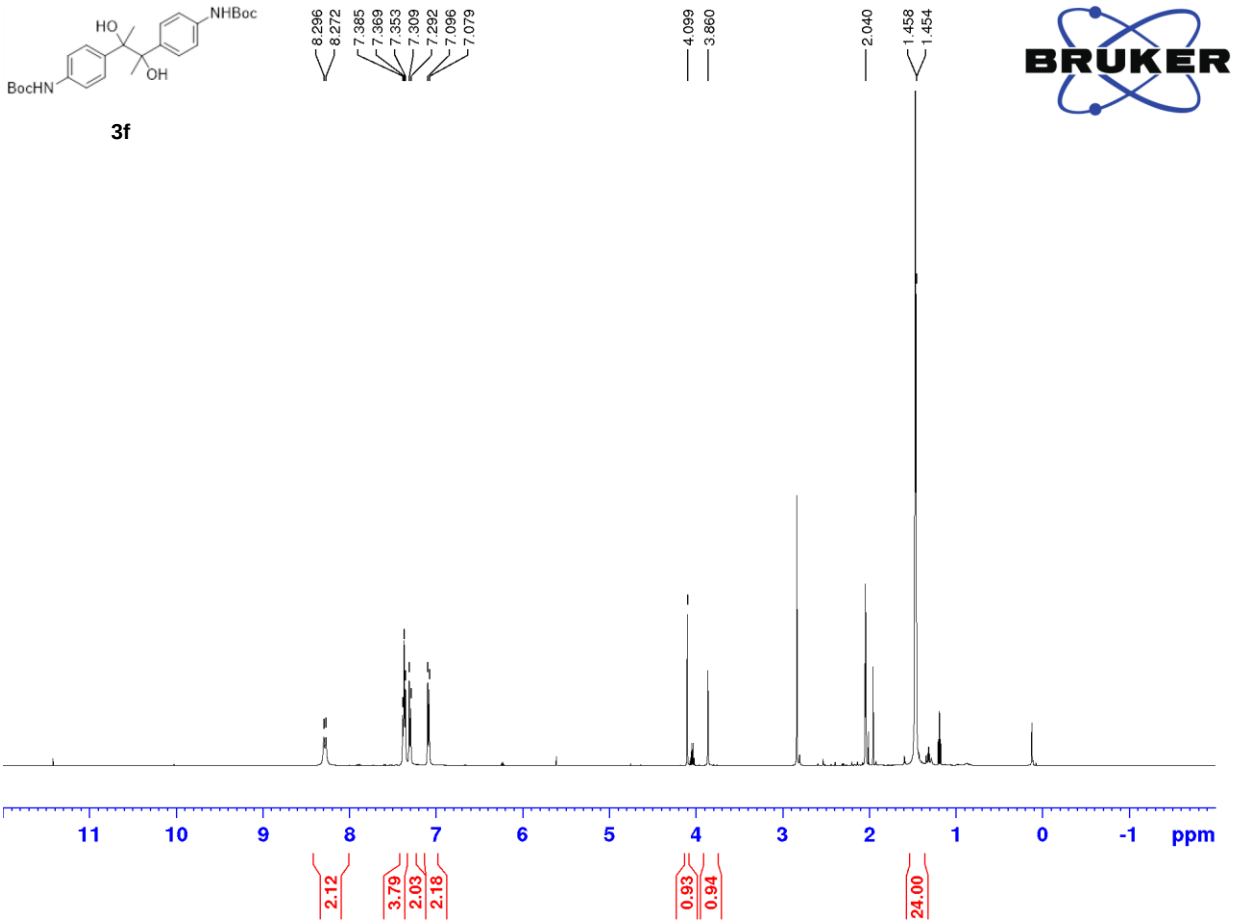
3e


HO
OH
isomer-2
7.260
6.006
5.921
5.918
3.749
3.527
3.440
1.746
1.743
BRUKER
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.00
2.21
6.18
6.10
6.58
6.75

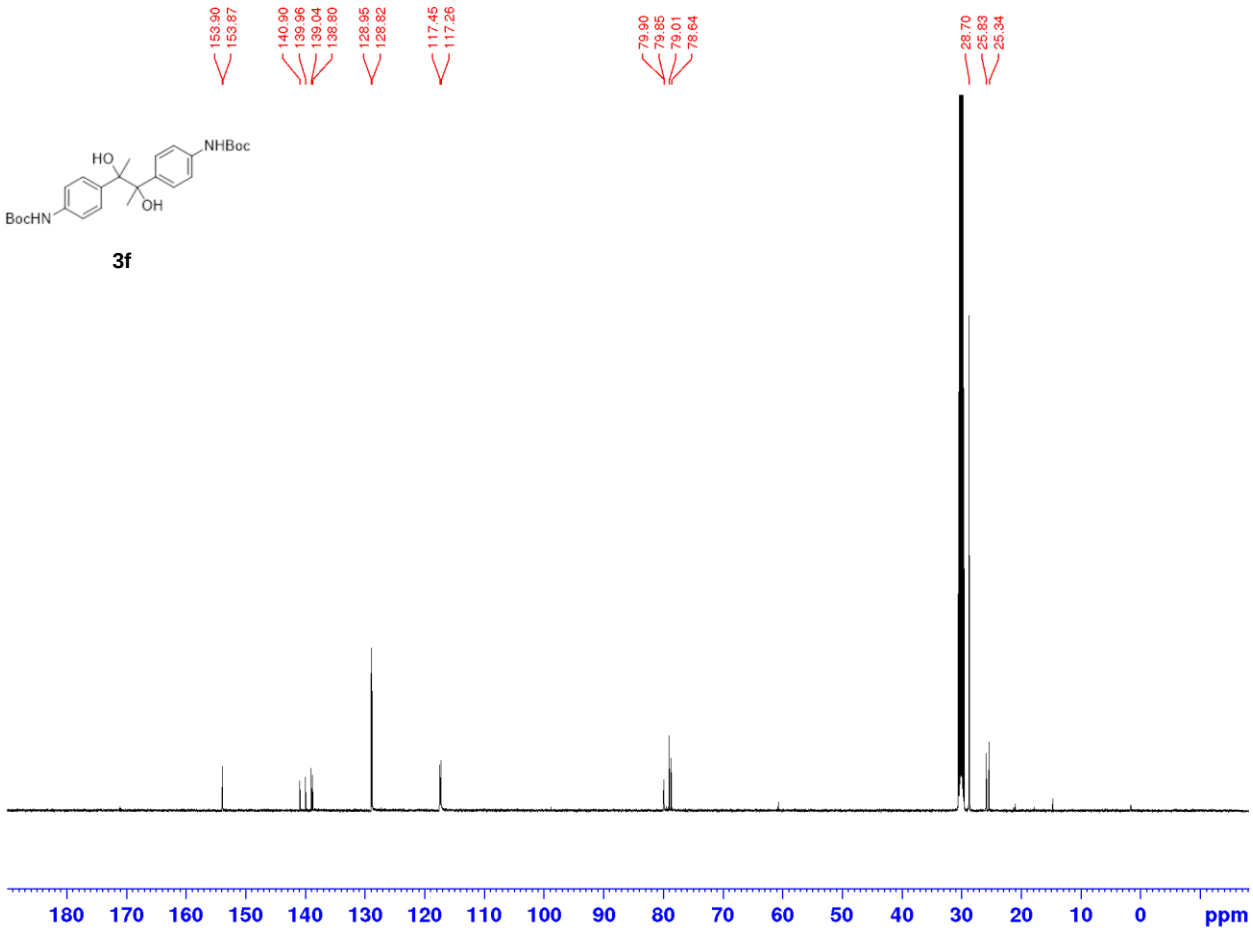
3e


HO
OH
isomer-2
160.41
160.00
159.38
115.78
92.12
91.90
83.55
77.41
77.16
76.90
56.29
55.34
25.71
200
180
160
140
120
100
80
60
40
20
0
ppm

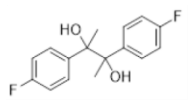
3e

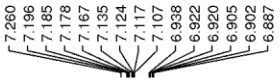

HO
NHBoc
BocHN
OH
8.296
8.272
7.385
7.369
7.353
7.309
7.292
7.096
7.079
4.099
3.860
2.040
1.458
1.454
BRUKER
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.12
3.79
2.03
2.18
0.93
0.94
24.00

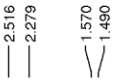
3f


153.90
153.87
140.90
139.96
139.04
138.80
128.95
128.82
117.45
117.26
79.90
79.85
79.01
78.64
28.70
25.83
25.34
NHBoc
HO
OH
BocHN
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

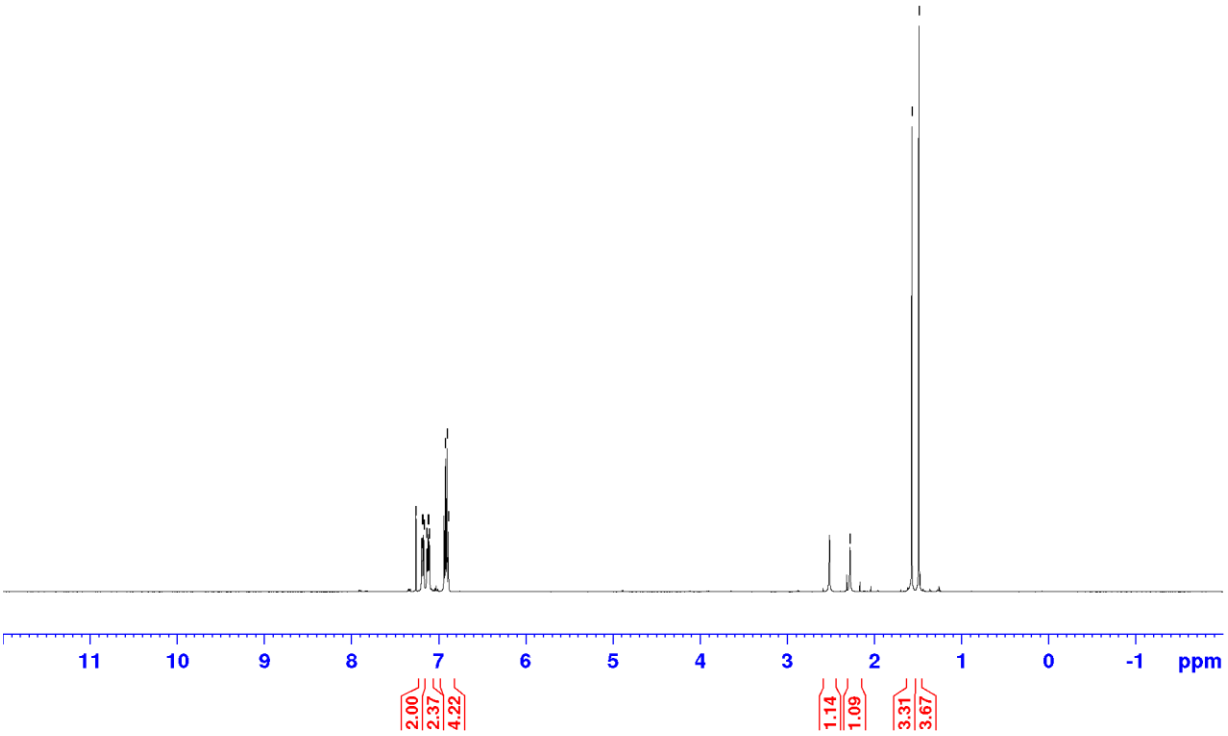
3f

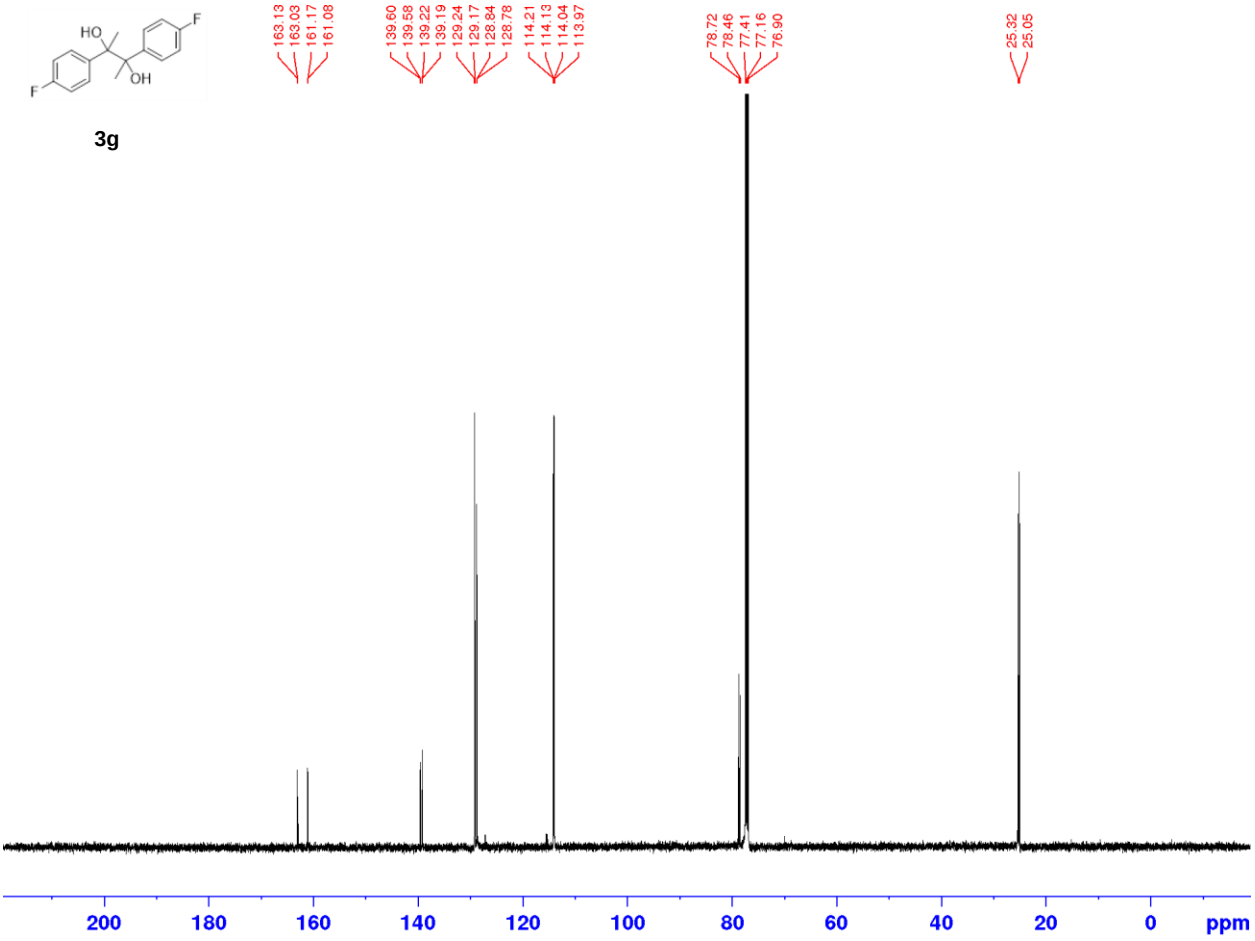

HO
F
F
OH

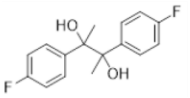

7.260
7.196
7.185
7.178
7.167
7.135
7.124
7.117
7.107
6.938
6.922
6.920
6.905
6.902
6.887


2.516
2.279
1.570
1.490

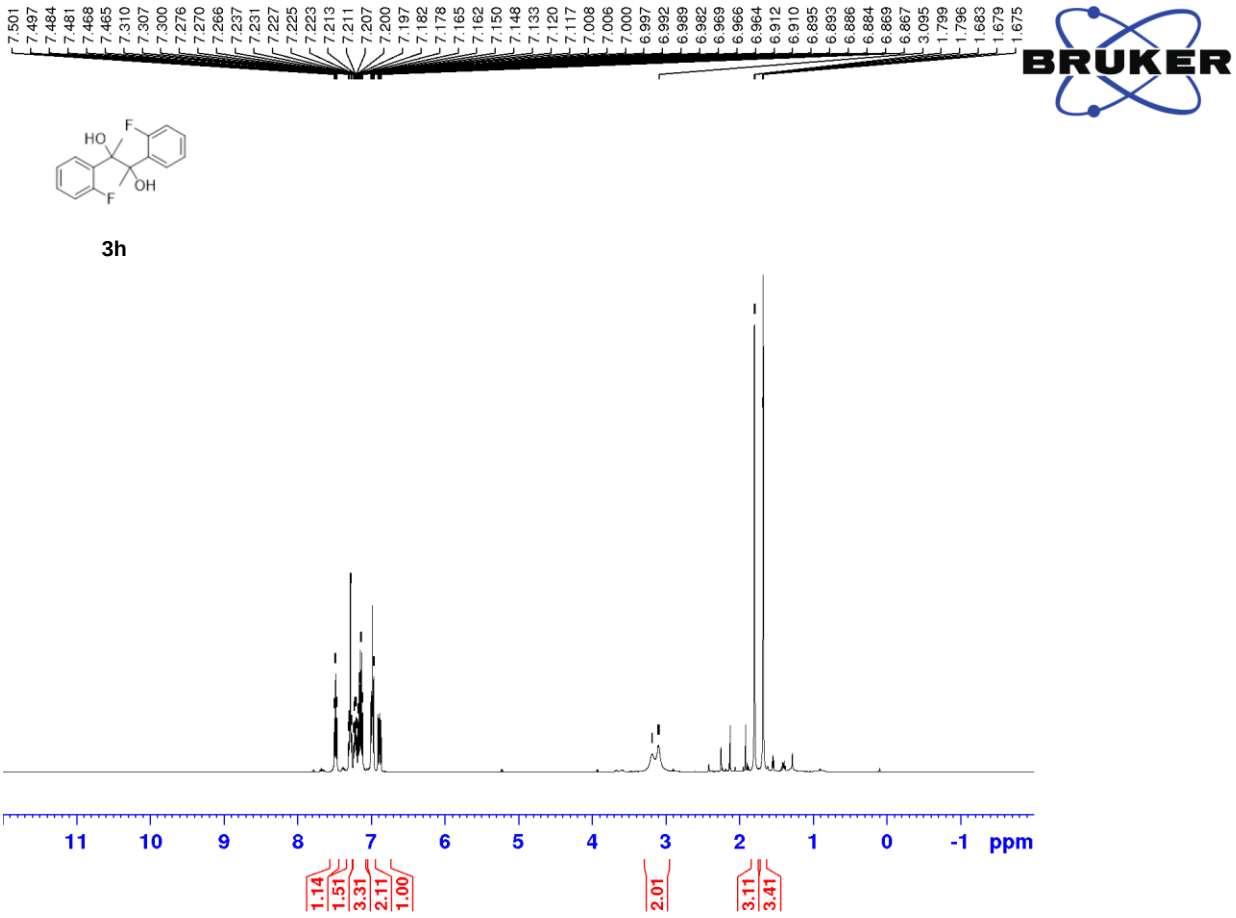
3g


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
2.37
4.22
1.14
1.09
3.31
3.67


HO
F
F
OH
3g
163.13
163.03
161.17
161.08
139.60
139.58
139.22
139.19
129.24
129.17
128.84
128.78
114.21
114.13
114.04
113.97
78.72
78.46
77.41
77.16
76.90
25.32
25.05
200
180
160
140
120
100
80
60
40
20
0
ppm



3g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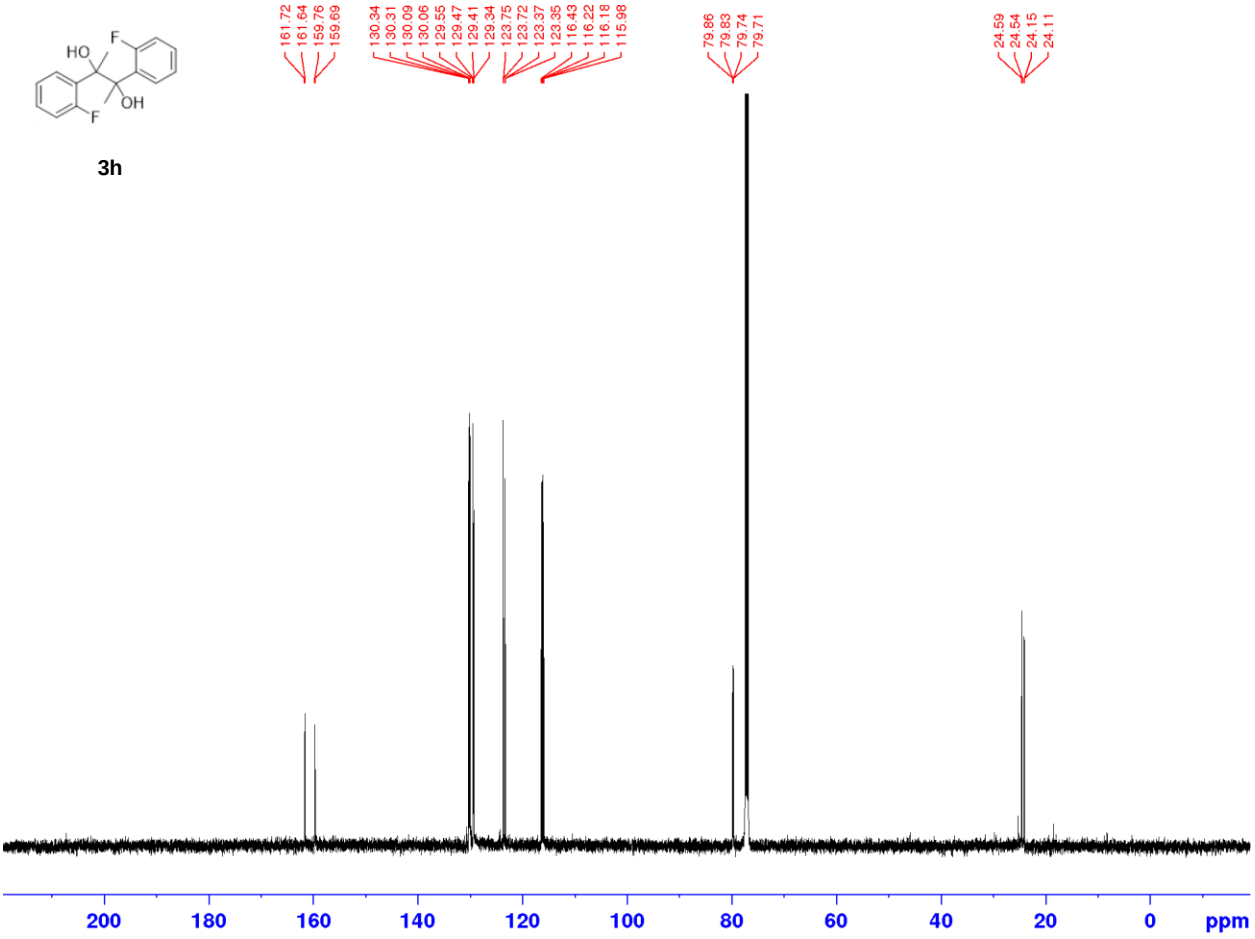
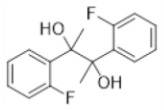
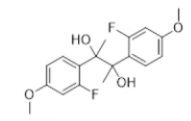
7.497
7.484
7.481
7.468
7.465
7.310
7.307
7.300
7.276
7.270
7.266
7.237
7.231
7.227
7.225
7.223
7.213
7.211
7.207
7.200
7.197
7.182
7.178
7.165
7.162
7.150
7.148
7.133
7.120
7.117
7.008
7.006
7.000
6.997
6.992
6.989
6.982
6.969
6.966
6.964
6.912
6.910
6.895
6.893
6.886
6.884
6.869
6.867
3.095
1.799
1.796
1.683
1.679
1.675
HO
F
OH
F
3h
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.14
1.51
3.31
2.11
1.00
2.01
3.11
3.41

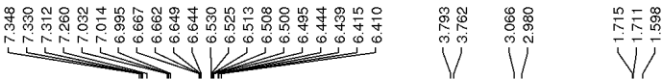

HO
F
OH
F
3h
161.72
161.64
159.76
159.69
130.34
130.31
130.09
130.06
129.55
129.47
129.41
129.34
123.75
123.72
123.37
123.35
116.43
116.22
116.18
115.98
79.86
79.83
79.74
79.71
24.59
24.54
24.15
24.11
200
180
160
140
120
100
80
60
40
20
0
ppm

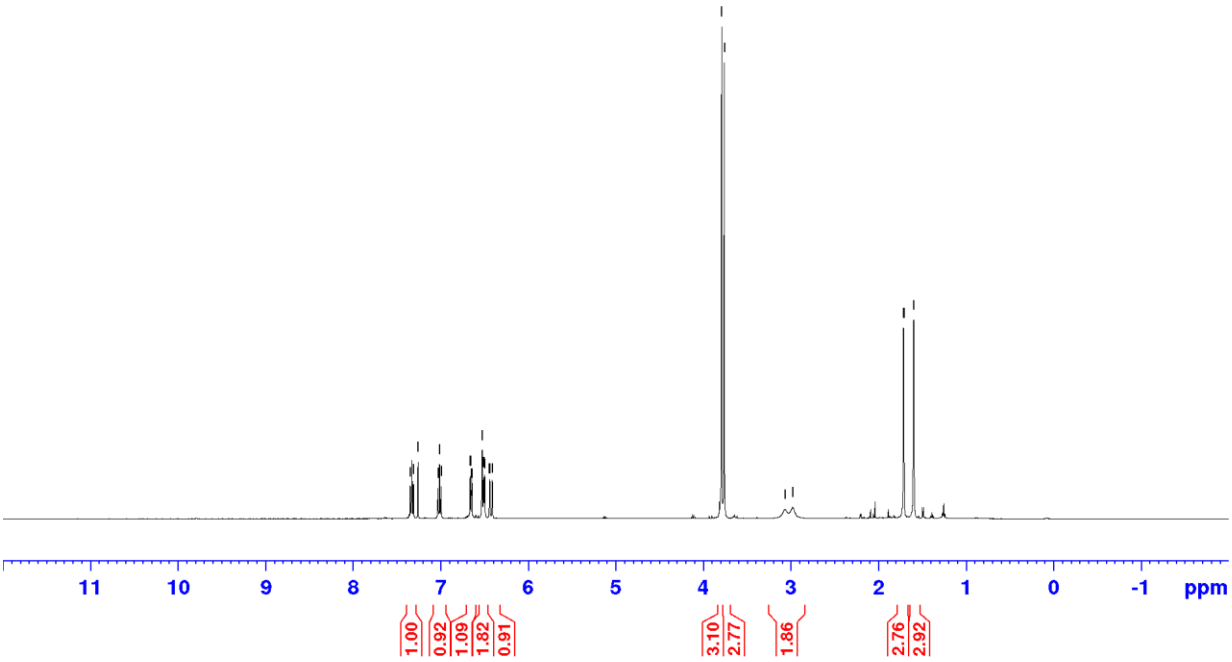


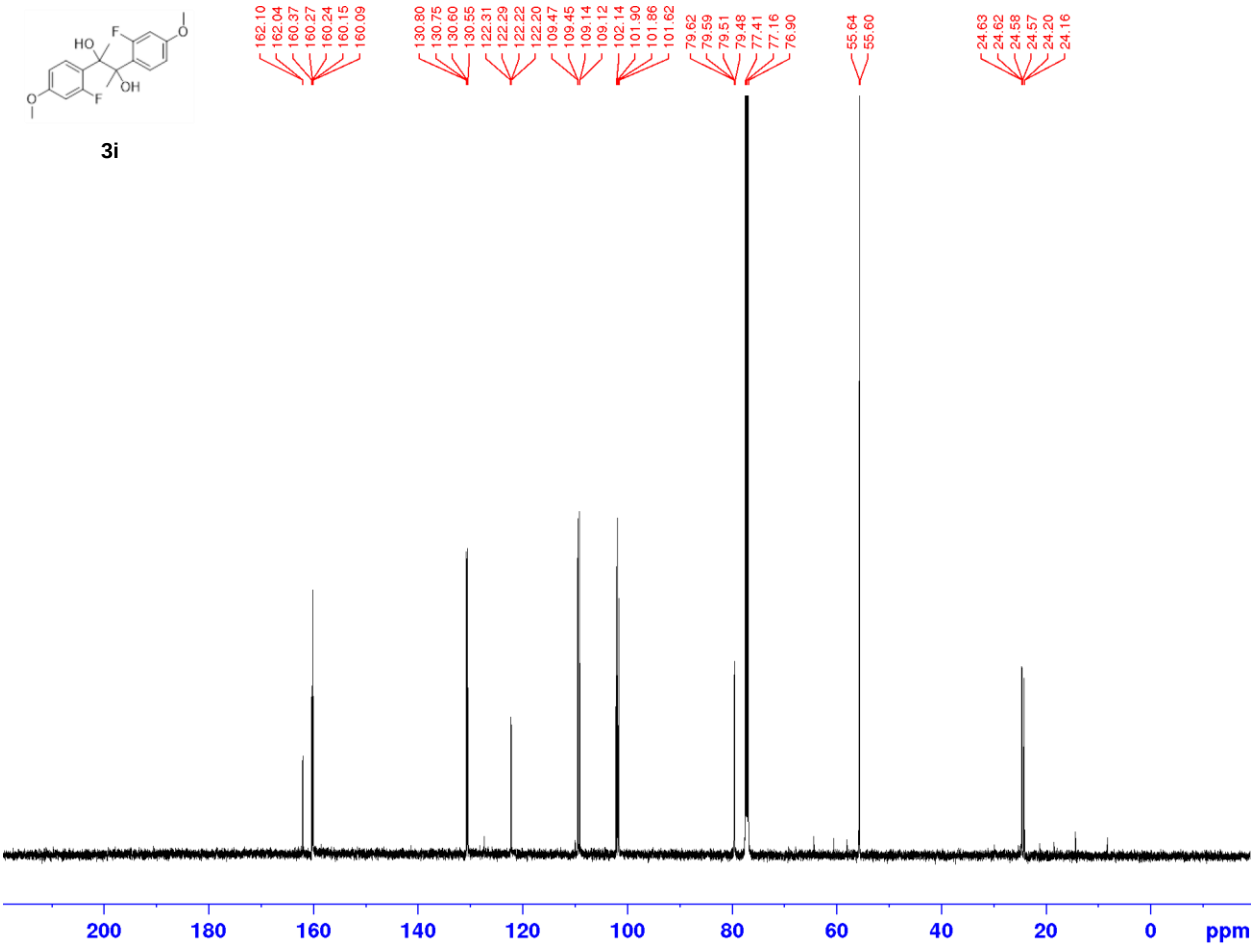
3h

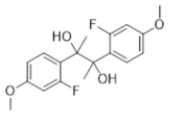

HO
F
O
O
F
OH

3i

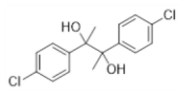

7.348
7.330
7.312
7.260
7.032
7.014
6.995
6.667
6.662
6.649
6.644
6.530
6.525
6.513
6.508
6.500
6.495
6.444
6.439
6.415
6.410
3.793
3.762
3.066
2.980
1.715
1.711
1.598


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
0.92
1.09
1.82
0.91
3.10
2.77
1.86
2.76
2.92

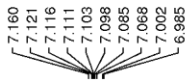

HO
F
O
O
F
OH
3i
162.10
162.04
160.37
160.27
160.24
160.15
160.09
130.80
130.75
130.60
130.55
122.31
122.29
122.22
122.20
109.47
109.45
109.14
109.12
102.14
101.90
101.86
101.62
79.62
79.59
79.51
79.48
77.41
77.16
76.90
55.64
55.60
24.63
24.62
24.58
24.57
24.20
24.16
200
180
160
140
120
100
80
60
40
20
0
ppm

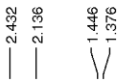


3i

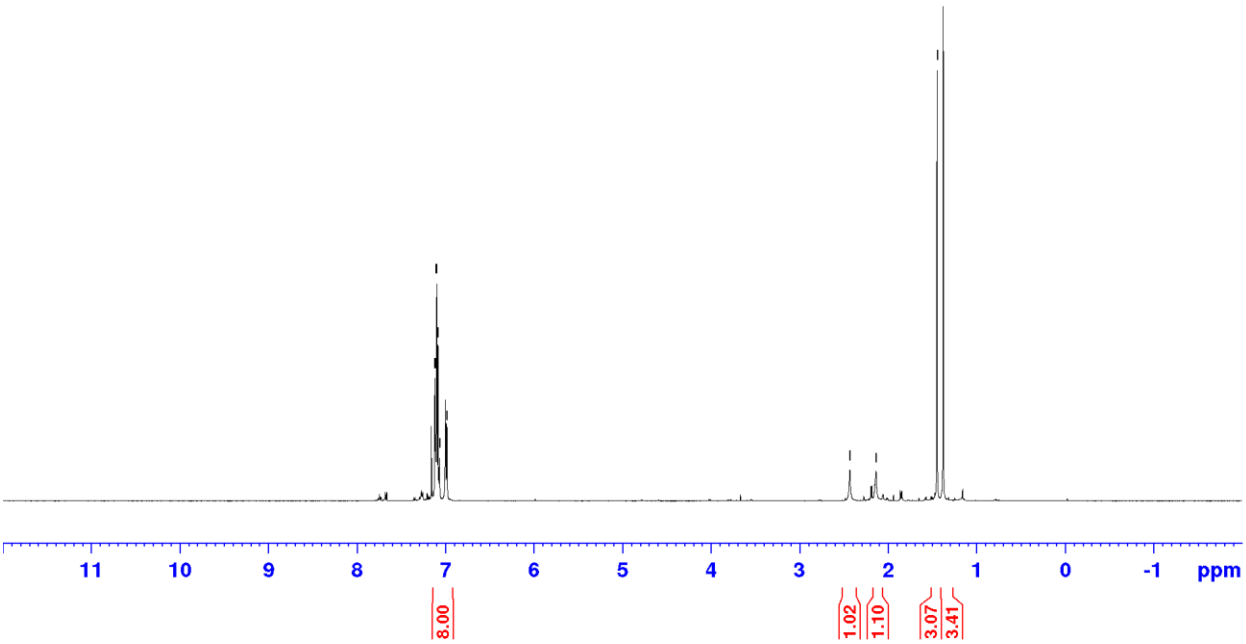

HO
Cl
Cl
OH

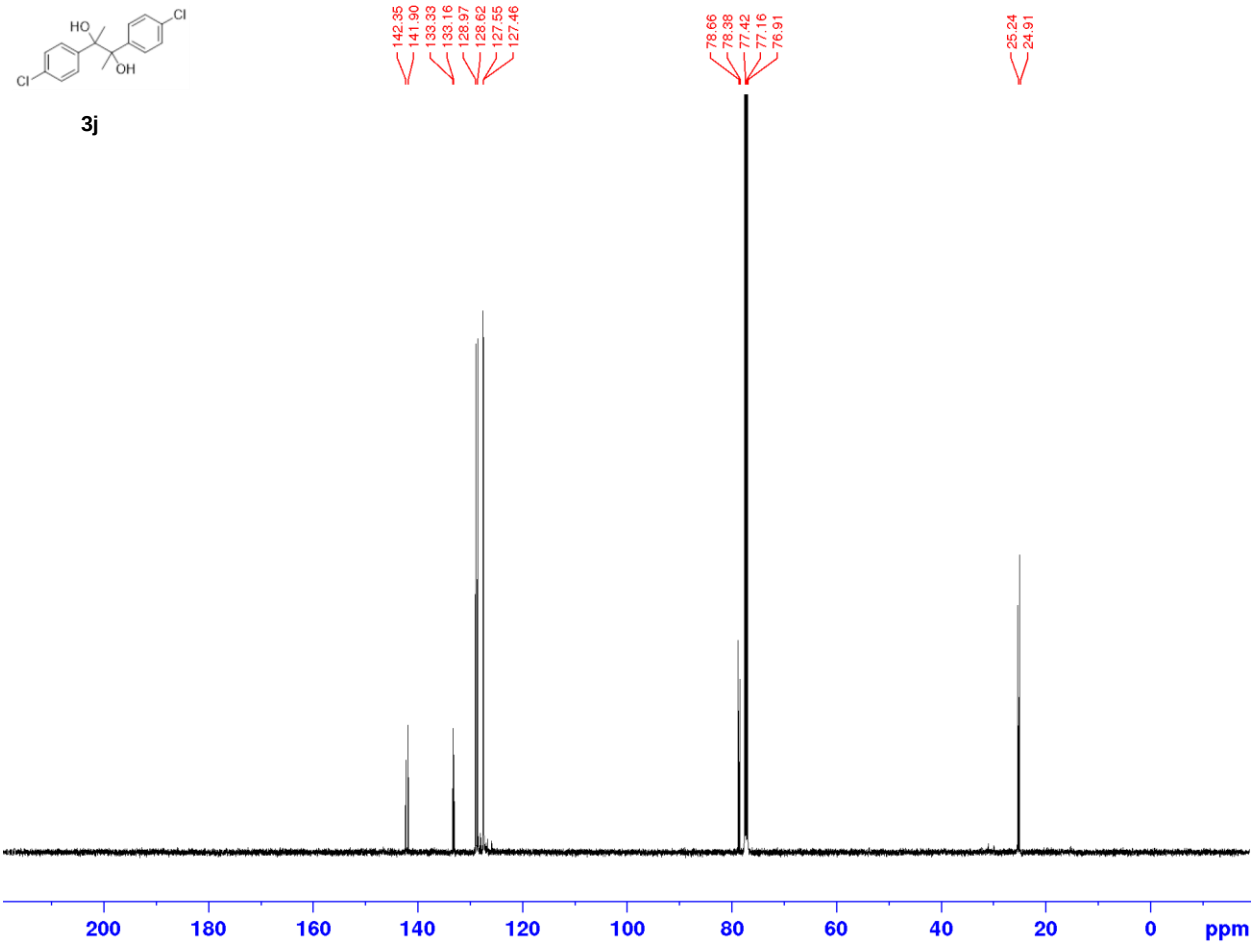
3j

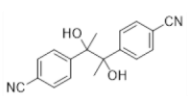

7.160
7.121
7.116
7.111
7.103
7.098
7.085
7.068
7.002
6.985


2.432
2.136
1.446
1.376


BRUKER


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
8.00
1.02
1.10
3.07
3.41


HO
Cl
Cl
OH
3j
142.35
141.90
133.33
133.16
128.97
128.62
127.55
127.46
78.66
78.38
77.42
77.16
76.91
25.24
24.91
200
180
160
140
120
100
80
60
40
20
0
ppm


HO
CN
NC
OH

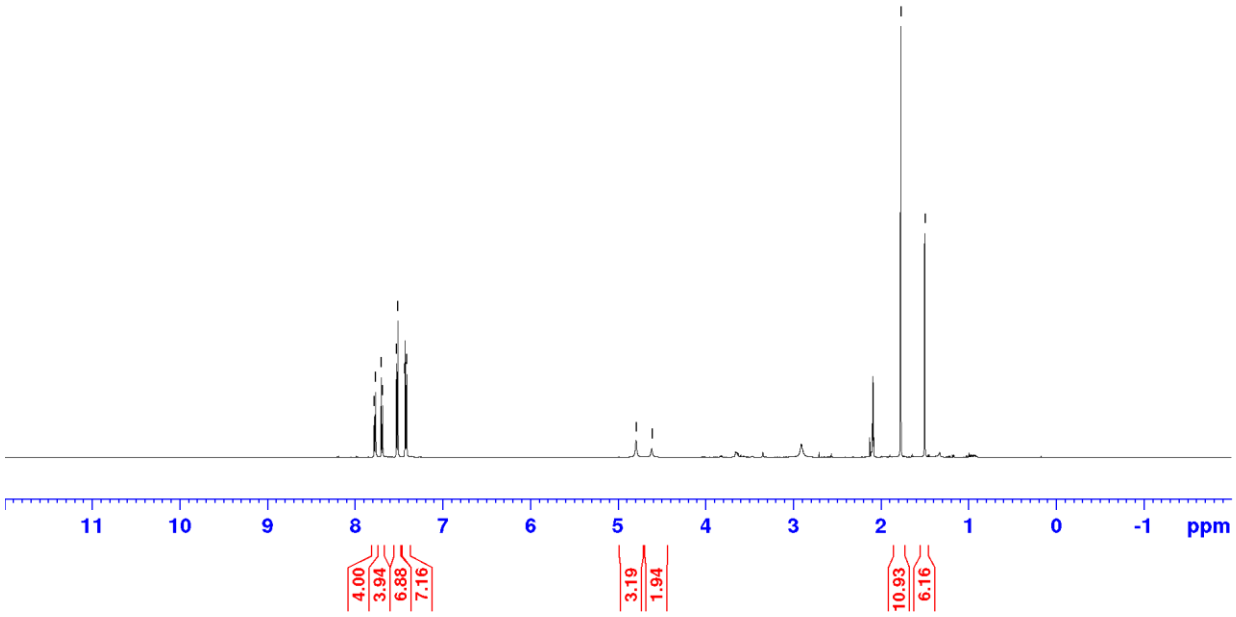
3k

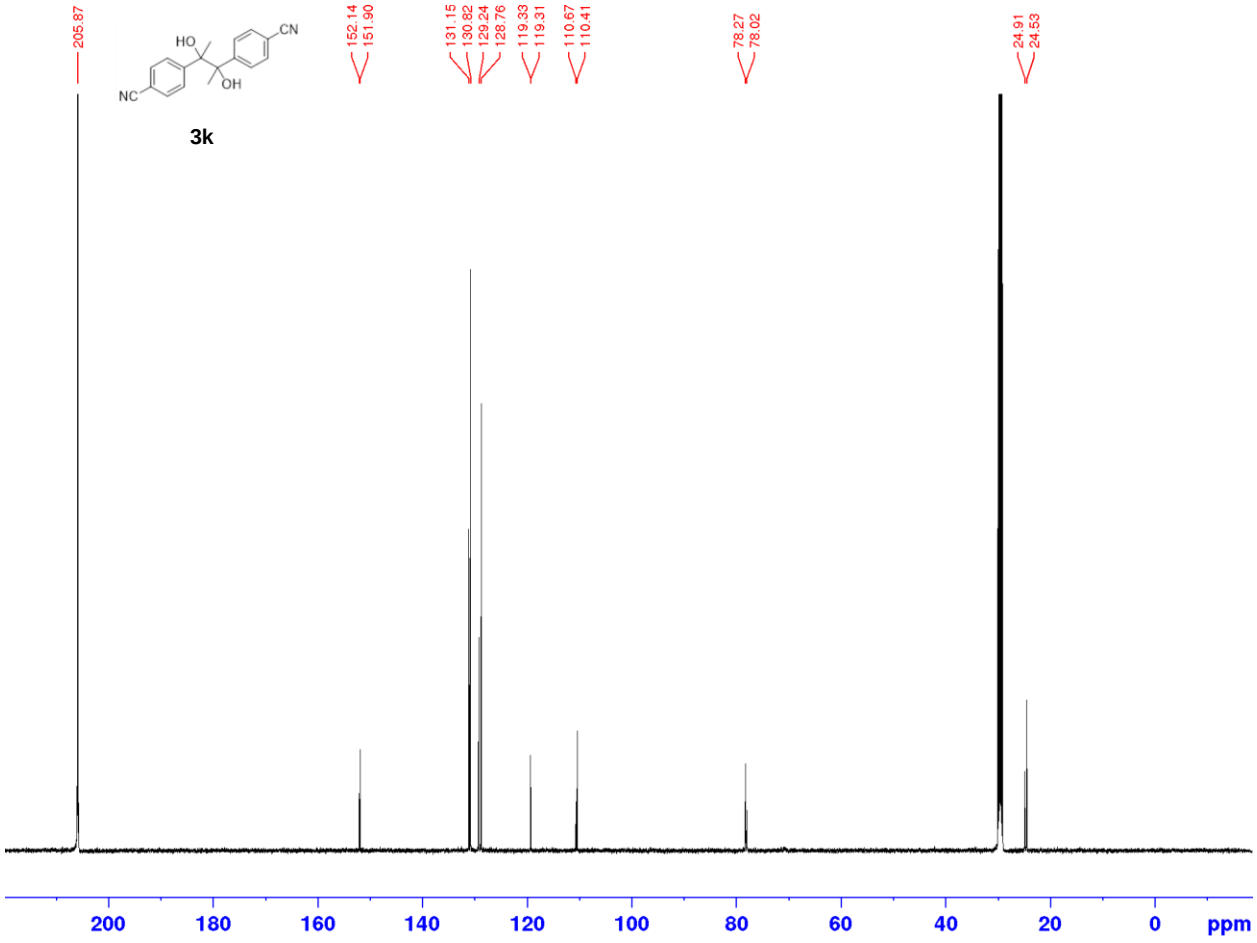

7.787
7.770
7.707
7.703
7.690
7.534
7.516
7.434
7.417

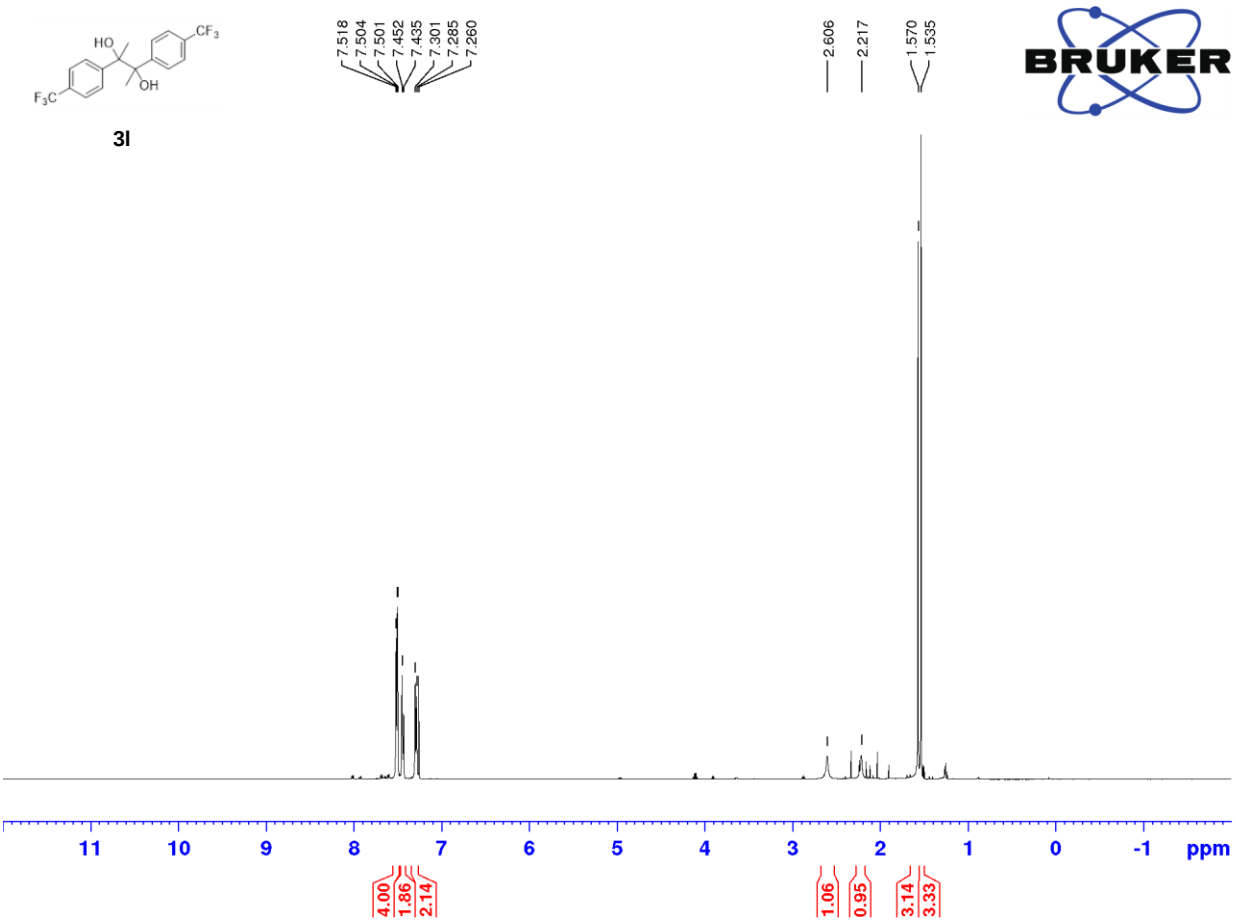

4.793
4.614

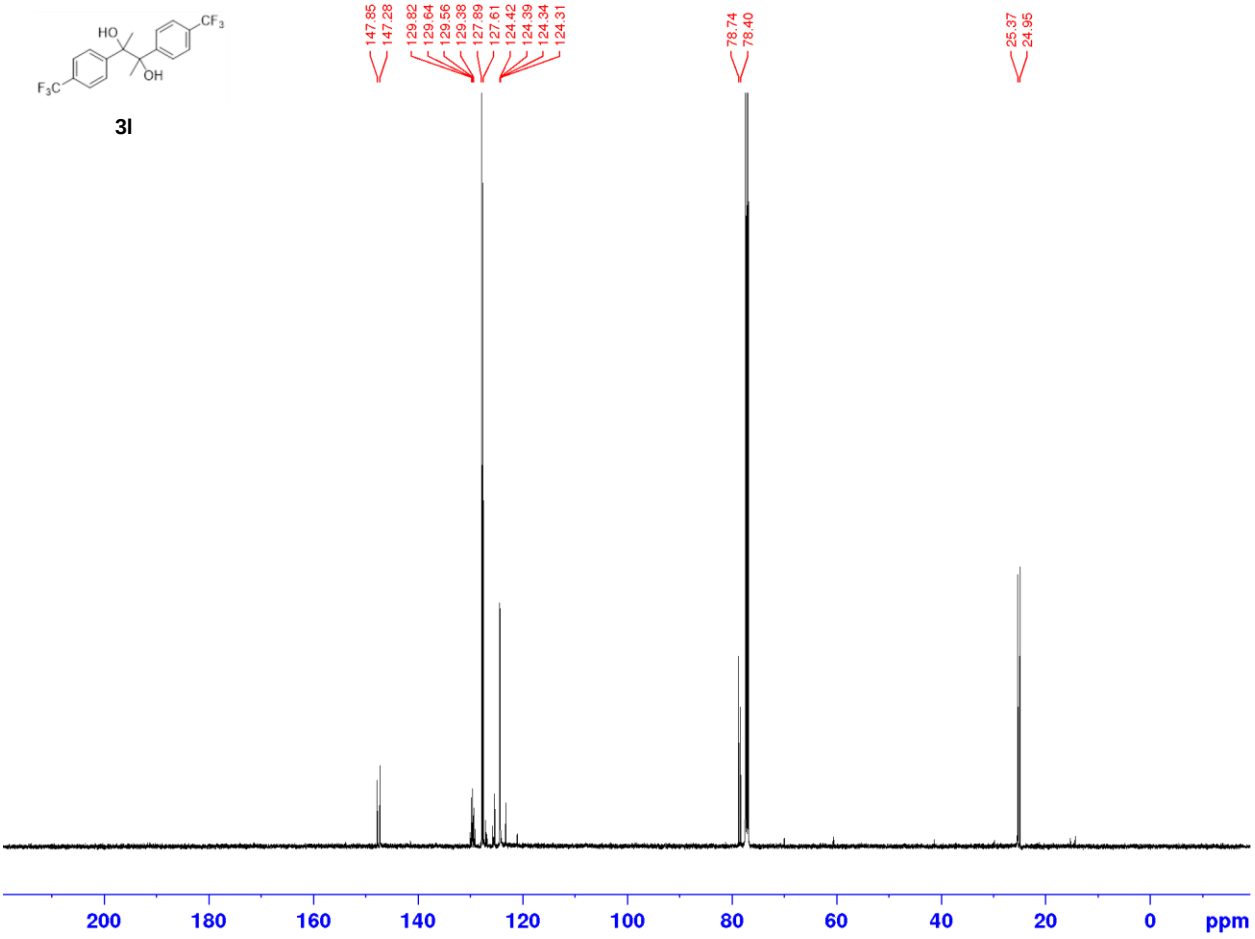

2.090
1.773
1.500

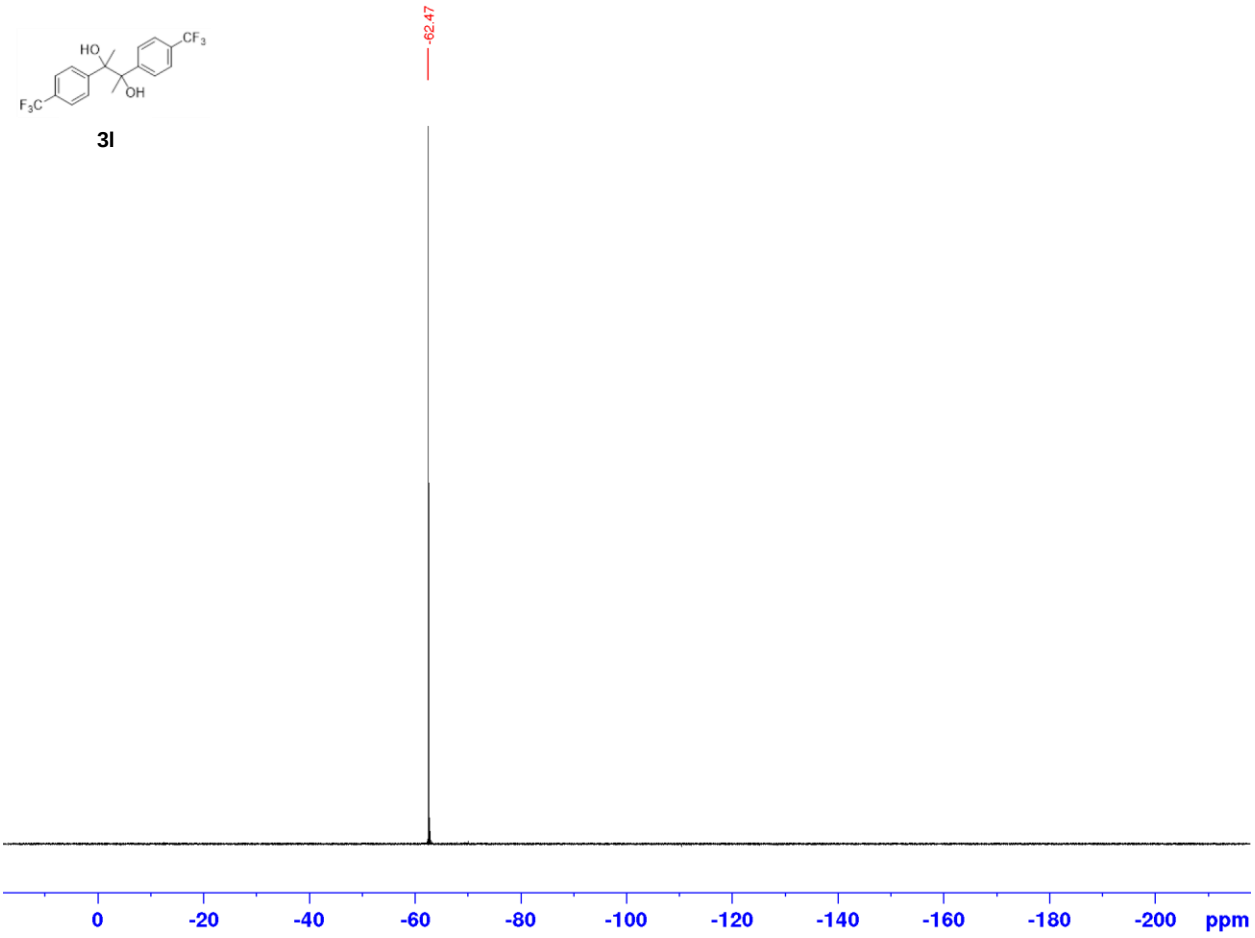

BRUKER


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.00
3.94
6.88
7.16
3.19
1.94
10.93
6.16


205.87
152.14
151.90
131.15
130.82
129.24
128.76
119.33
119.31
110.67
110.41
78.27
78.02
24.91
24.53
HO
CN
NC
OH
3k
200
180
160
140
120
100
80
60
40
20
0
ppm


HO
CF3
F3C
OH
3l
7.518
7.504
7.501
7.452
7.435
7.301
7.285
7.260
2.606
2.217
1.570
1.535
BRUKER
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.00
1.86
2.14
1.06
0.95
3.14
3.33


HO
CF3
F3C
OH
3l
147.85
147.28
129.82
129.64
129.56
129.38
127.89
127.61
124.42
124.39
124.34
124.31
78.74
78.40
25.37
24.95
200
180
160
140
120
100
80
60
40
20
0
ppm


HO
CF3
F3C
OH
3l
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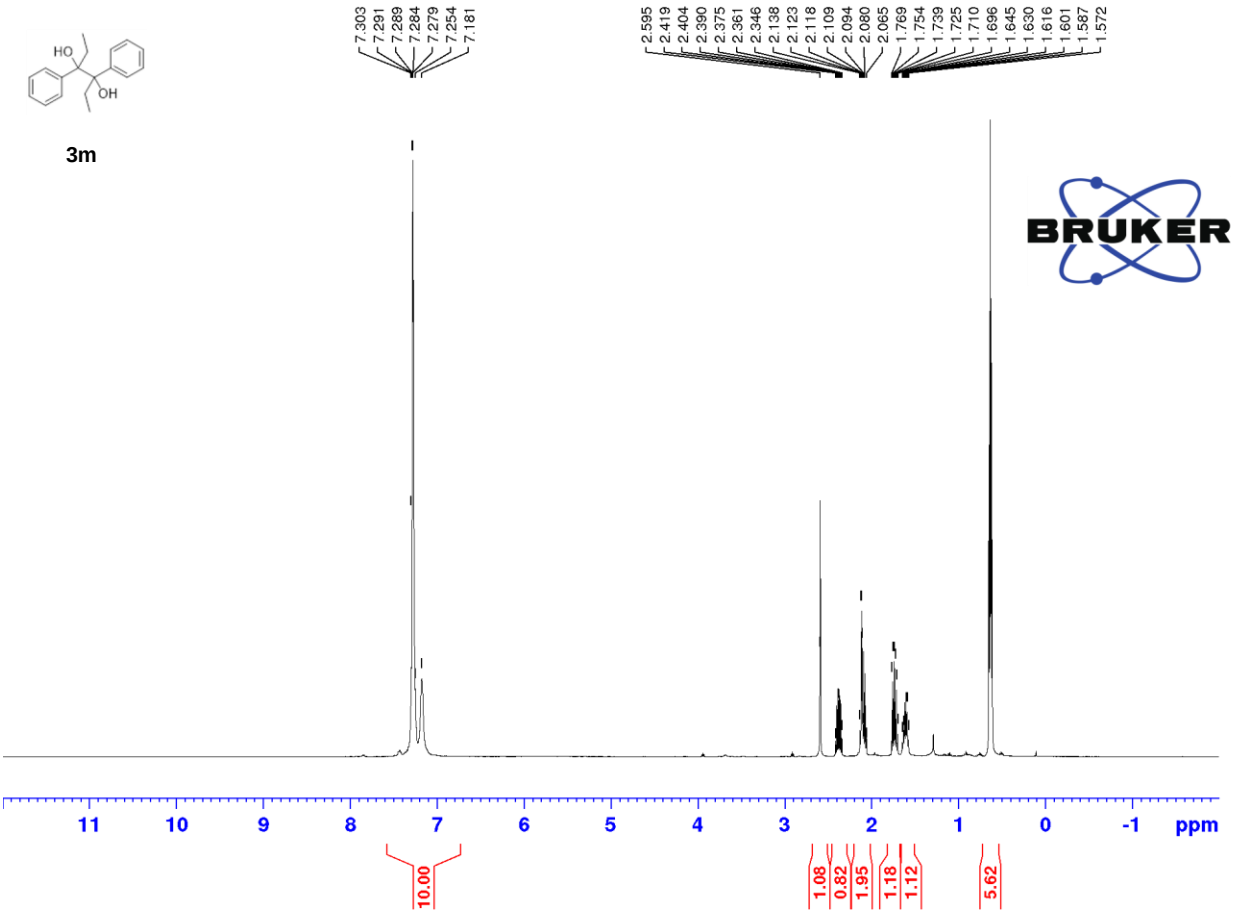
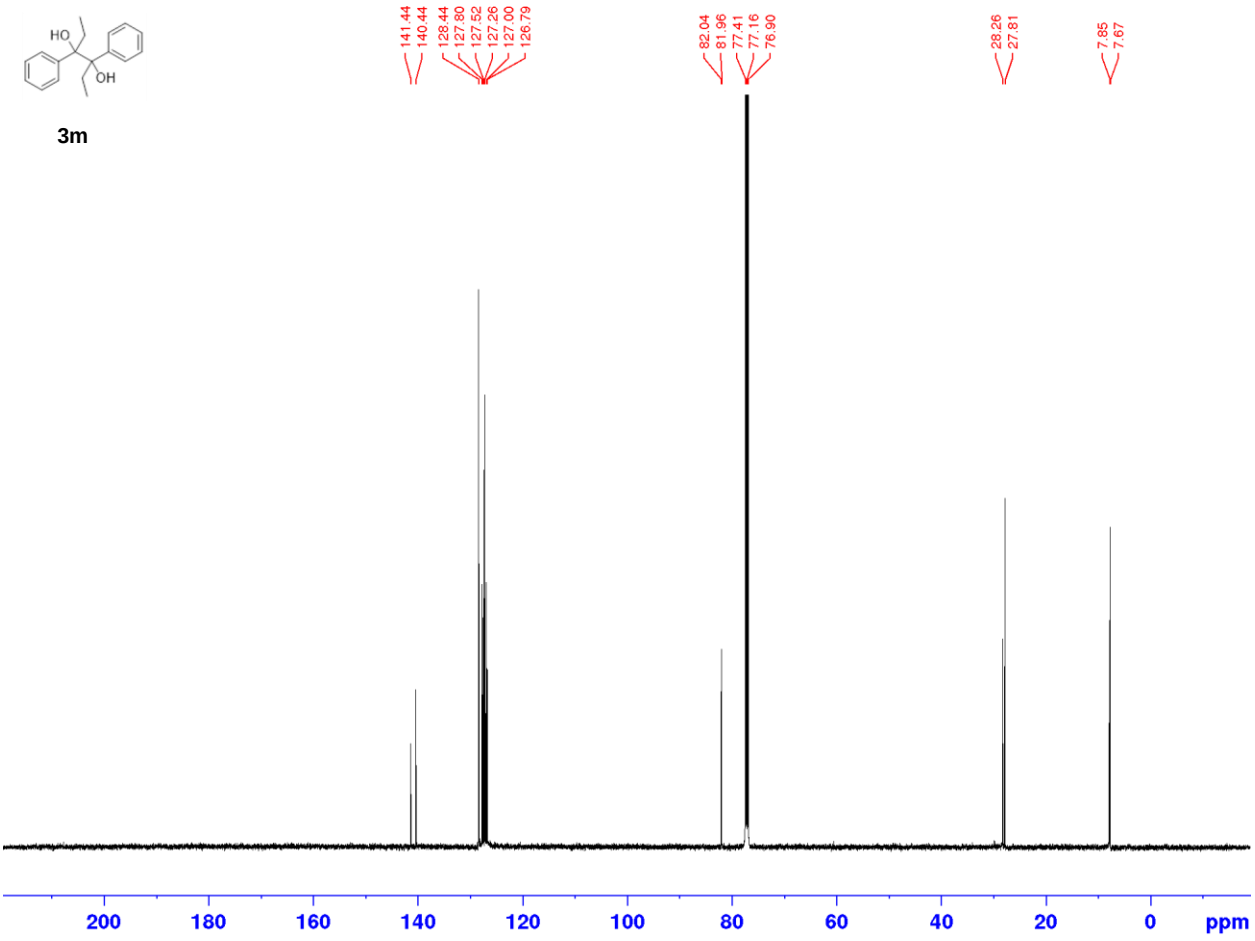
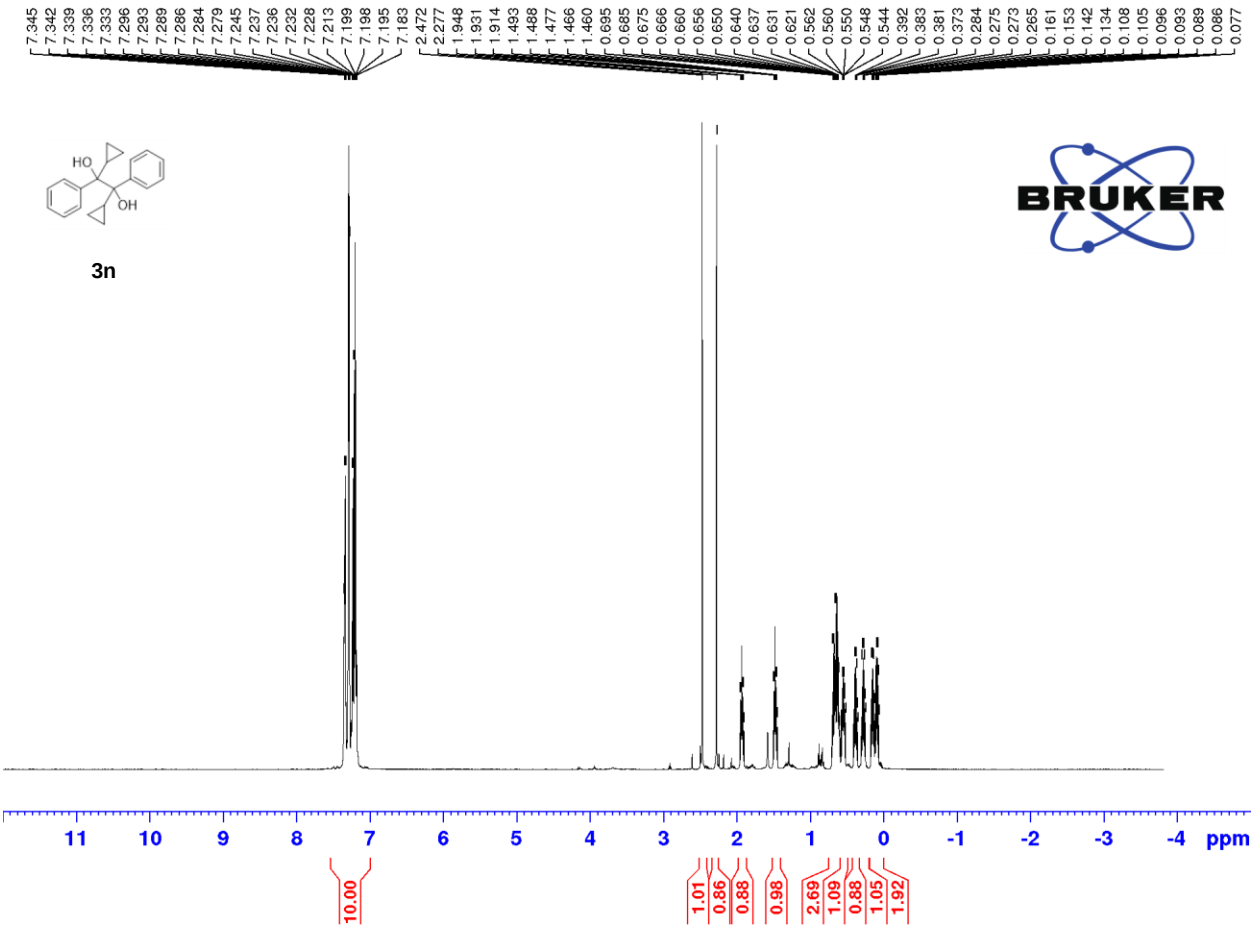
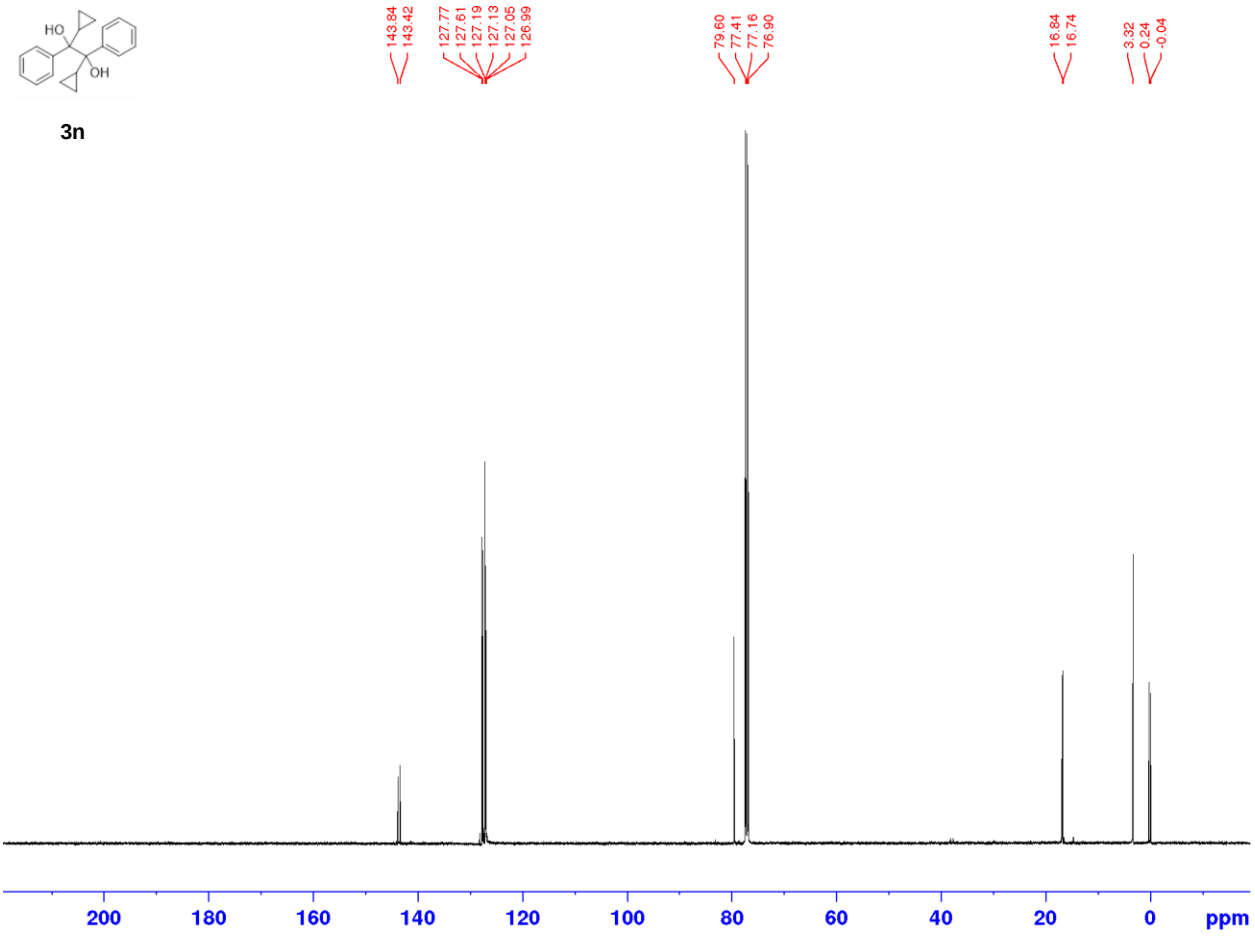
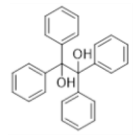
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm

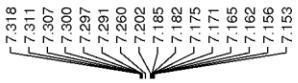

HO
OH
3m
7.303
7.291
7.289
7.284
7.279
7.254
7.181
2.595
2.419
2.404
2.390
2.375
2.361
2.346
2.138
2.123
2.118
2.109
2.094
2.080
2.065
1.769
1.754
1.739
1.725
1.710
1.696
1.645
1.630
1.616
1.601
1.587
1.572
BRUKER
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
10.00
1.08
0.82
1.95
1.18
1.12
5.62


HO
OH
3m
141.44
140.44
128.44
127.80
127.52
127.26
127.00
126.79
82.04
81.96
77.41
77.16
76.90
28.26
27.81
7.85
7.67
200
180
160
140
120
100
80
60
40
20
0
ppm

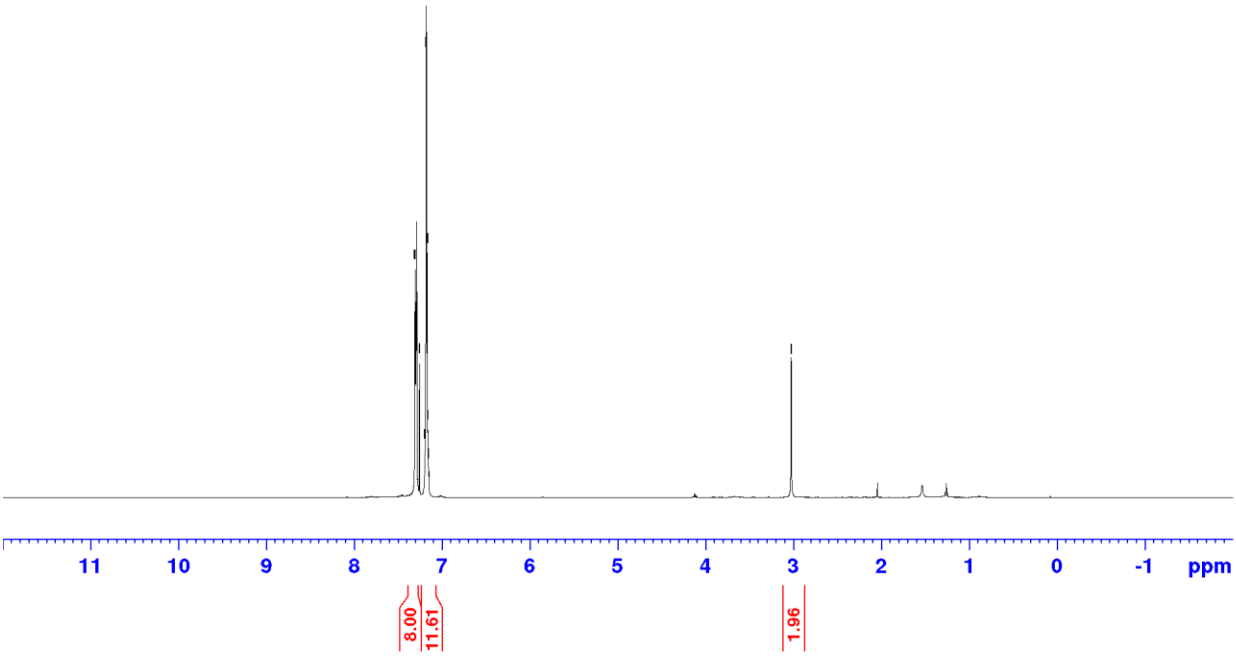

7.345
7.342
7.339
7.336
7.333
7.296
7.293
7.289
7.286
7.284
7.279
7.245
7.237
7.236
7.232
7.228
7.213
7.199
7.198
7.195
7.183
2.472
2.277
1.948
1.931
1.914
1.493
1.488
1.477
1.466
1.460
0.695
0.685
0.675
0.666
0.660
0.656
0.650
0.640
0.637
0.631
0.621
0.562
0.560
0.550
0.548
0.544
0.392
0.383
0.381
0.373
0.284
0.275
0.273
0.265
0.161
0.153
0.142
0.134
0.108
0.105
0.096
0.093
0.089
0.086
0.077
HO
OH
3n
BRUKER
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
-4
ppm
10.00
1.01
0.86
0.88
0.98
2.69
1.09
0.88
1.05
1.92

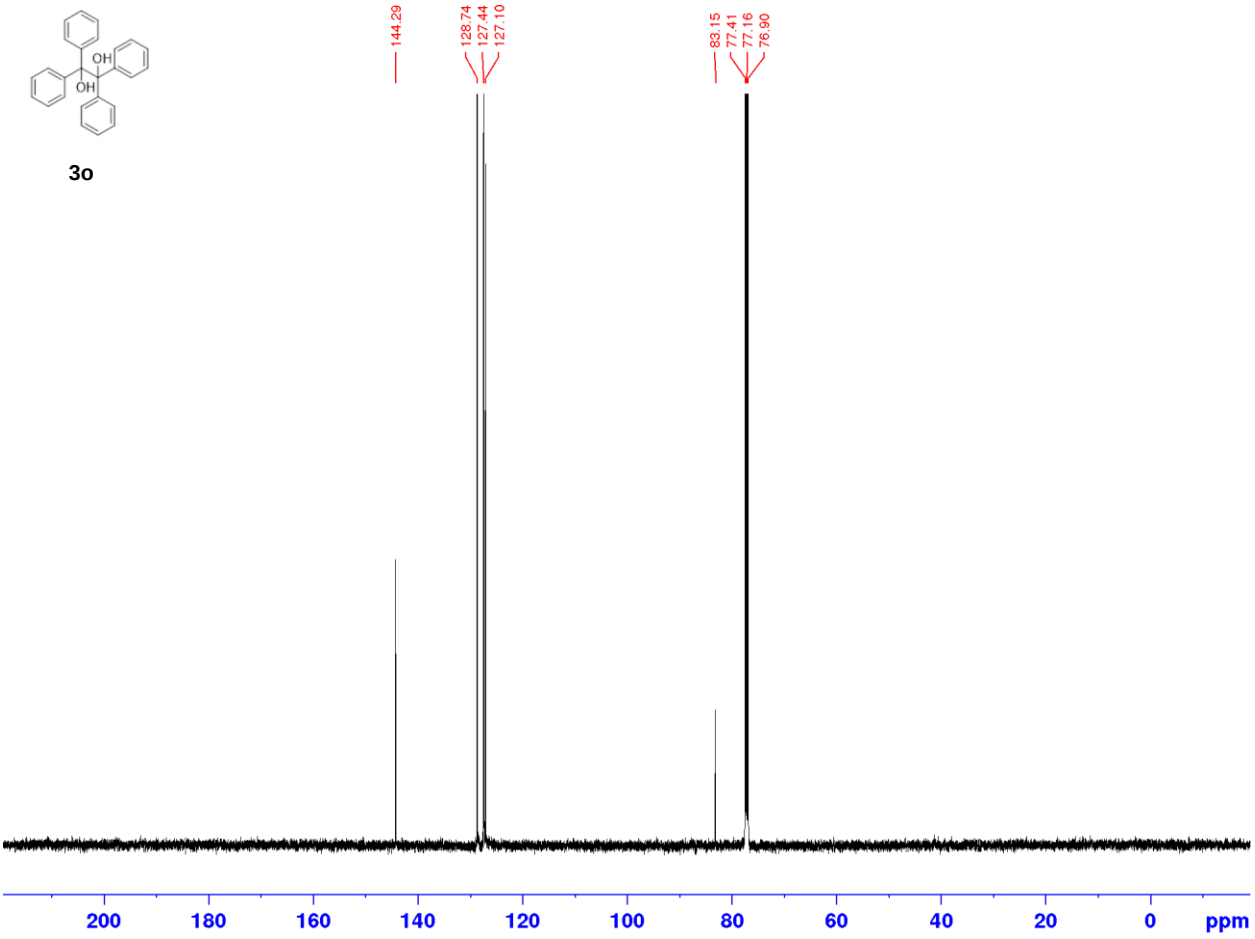

HO
OH
3n
143.84
143.42
127.77
127.61
127.19
127.13
127.05
126.99
79.60
77.41
77.16
76.90
16.84
16.74
3.32
0.24
-0.04
200
180
160
140
120
100
80
60
40
20
0
ppm

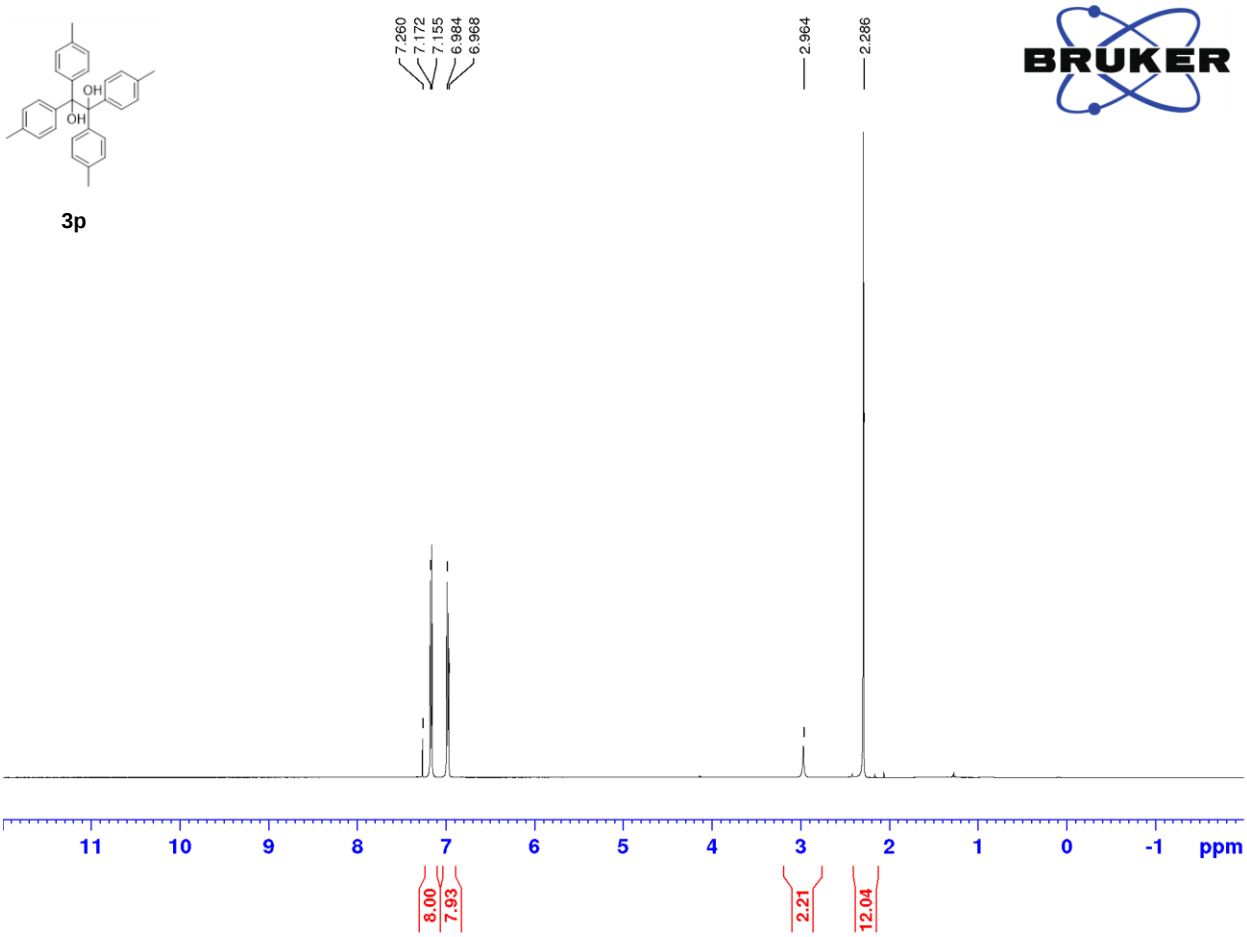

OH
OH

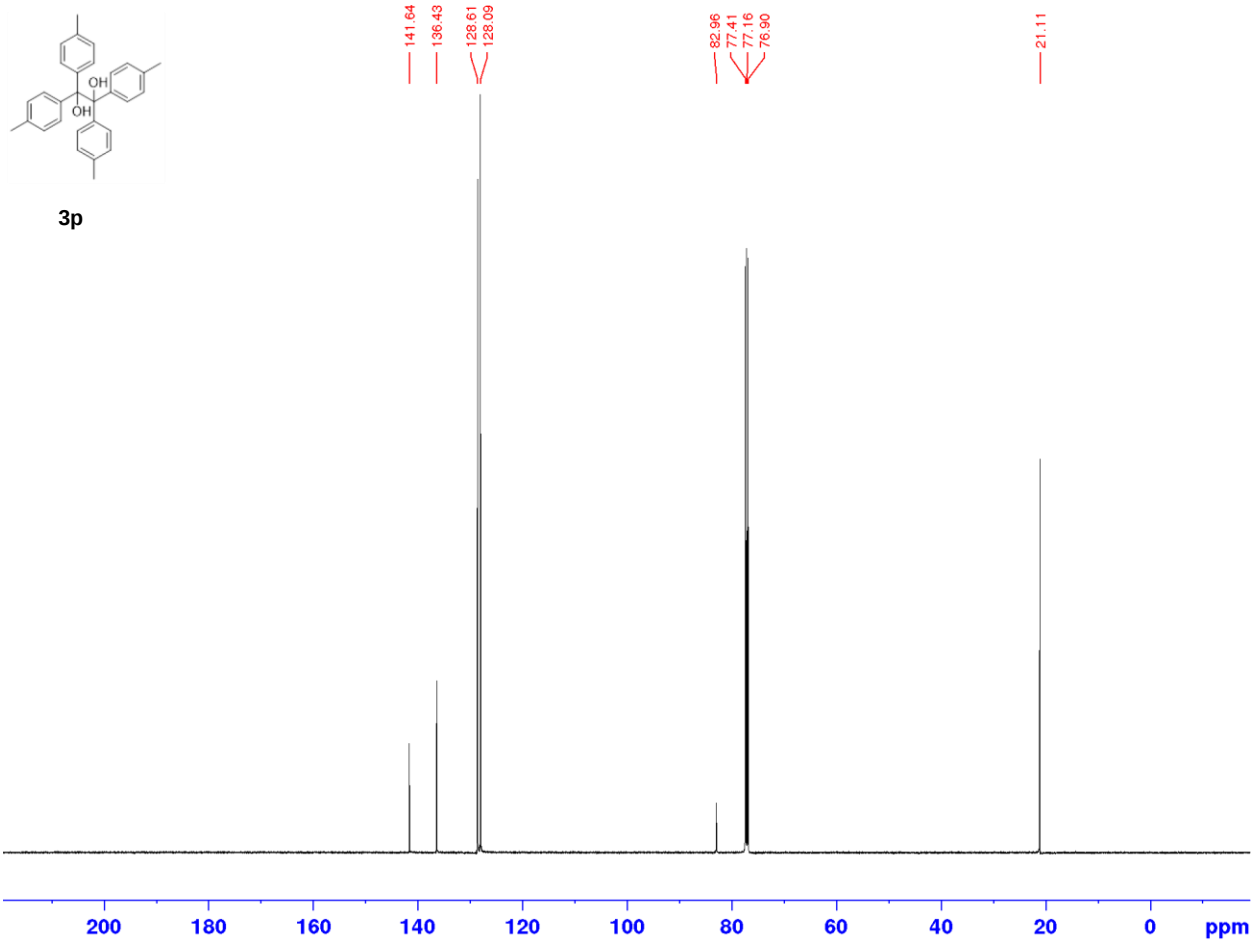

7.318
7.311
7.307
7.300
7.297
7.291
7.260
7.202
7.185
7.182
7.175
7.171
7.165
7.162
7.156
7.153
3.029

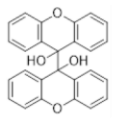
3o


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
8.00
11.61
1.96


OH
OH
3o
144.29
128.74
127.44
127.10
83.15
77.41
77.16
76.90
200
180
160
140
120
100
80
60
40
20
0
ppm

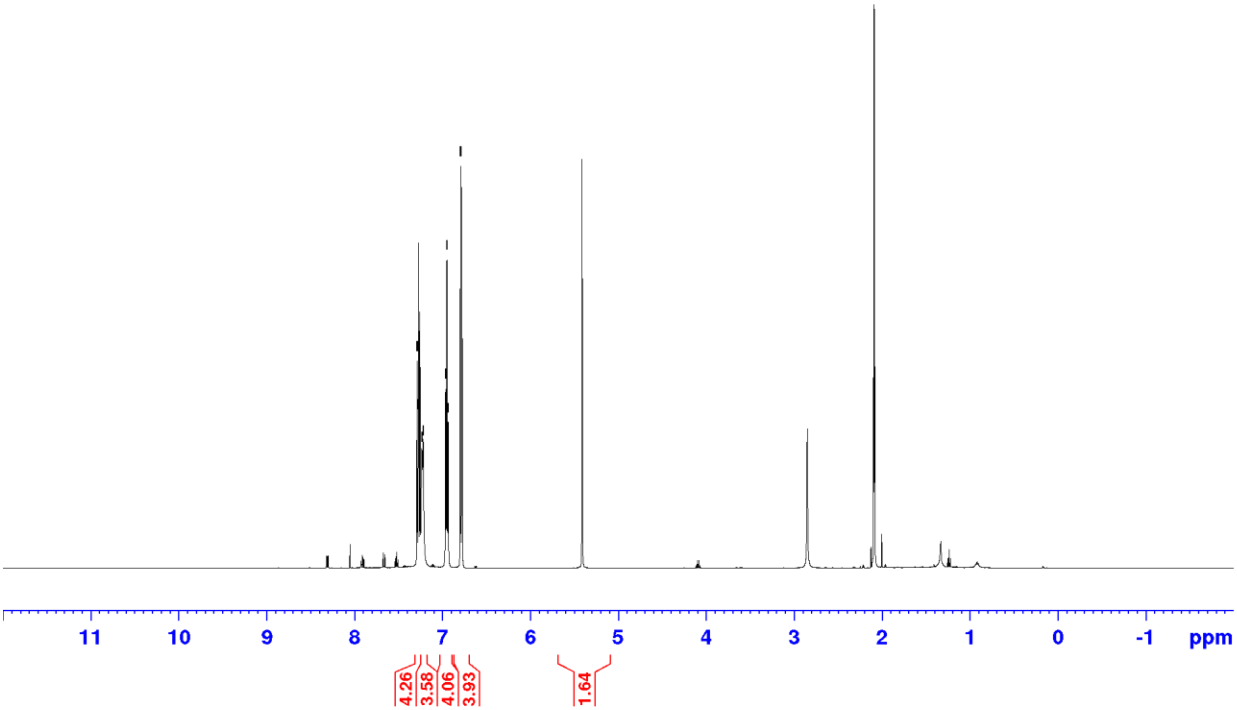

OH
OH
3p
7.260
7.172
7.155
6.984
6.968
2.964
2.286
BRUKER
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
8.00
7.93
2.21
12.04

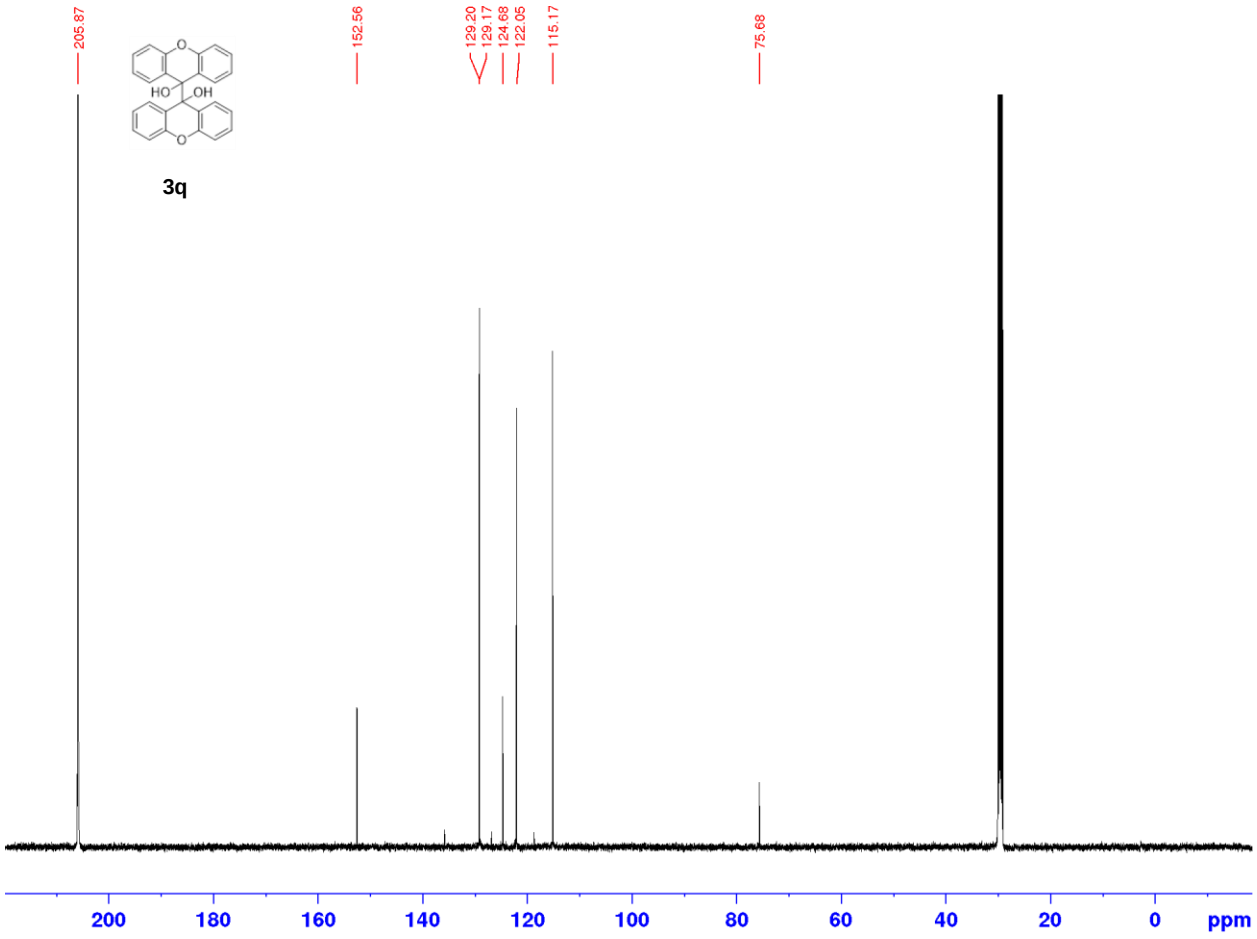

OH
OH
3p
141.64
136.43
128.61
128.09
82.96
77.41
77.16
76.90
21.11
200
180
160
140
120
100
80
60
40
20
0
ppm

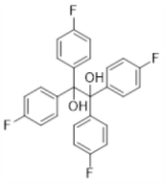

HO
OH
O
O


7.291
7.287
7.276
7.274
7.273
7.271
7.260
7.257
7.236
7.221
6.966
6.965
6.951
6.937
6.935
6.797
6.795
6.780
6.778
5.412
2.090

3q


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.26
3.58
4.06
3.93
1.64


205.87
152.56
129.20
129.17
124.68
122.05
115.17
75.68
HO
OH
O
O
3q
200
180
160
140
120
100
80
60
40
20
0
ppm

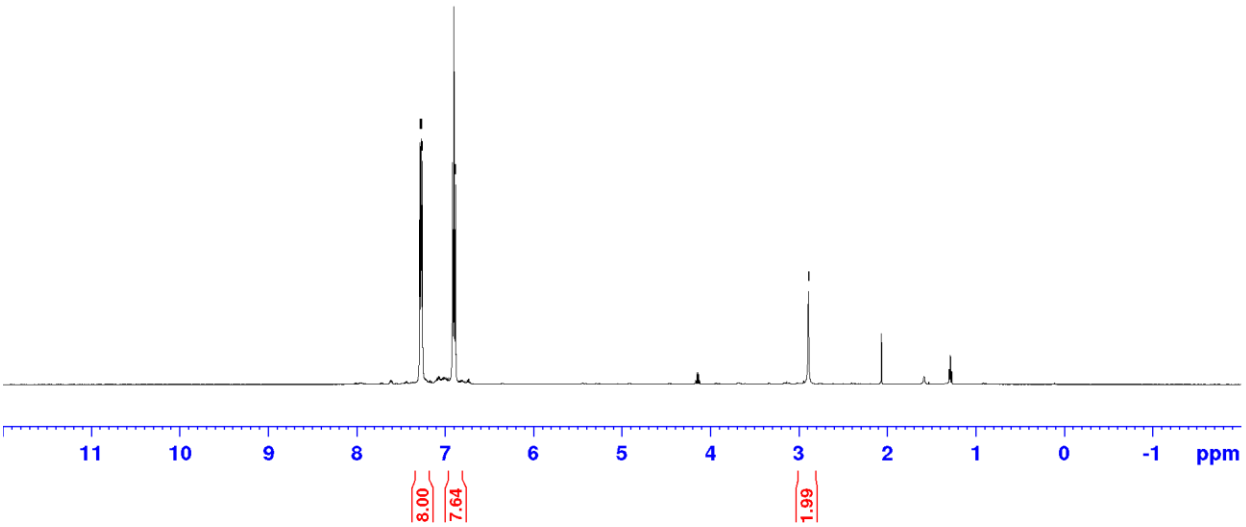

F
F
OH
OH
F

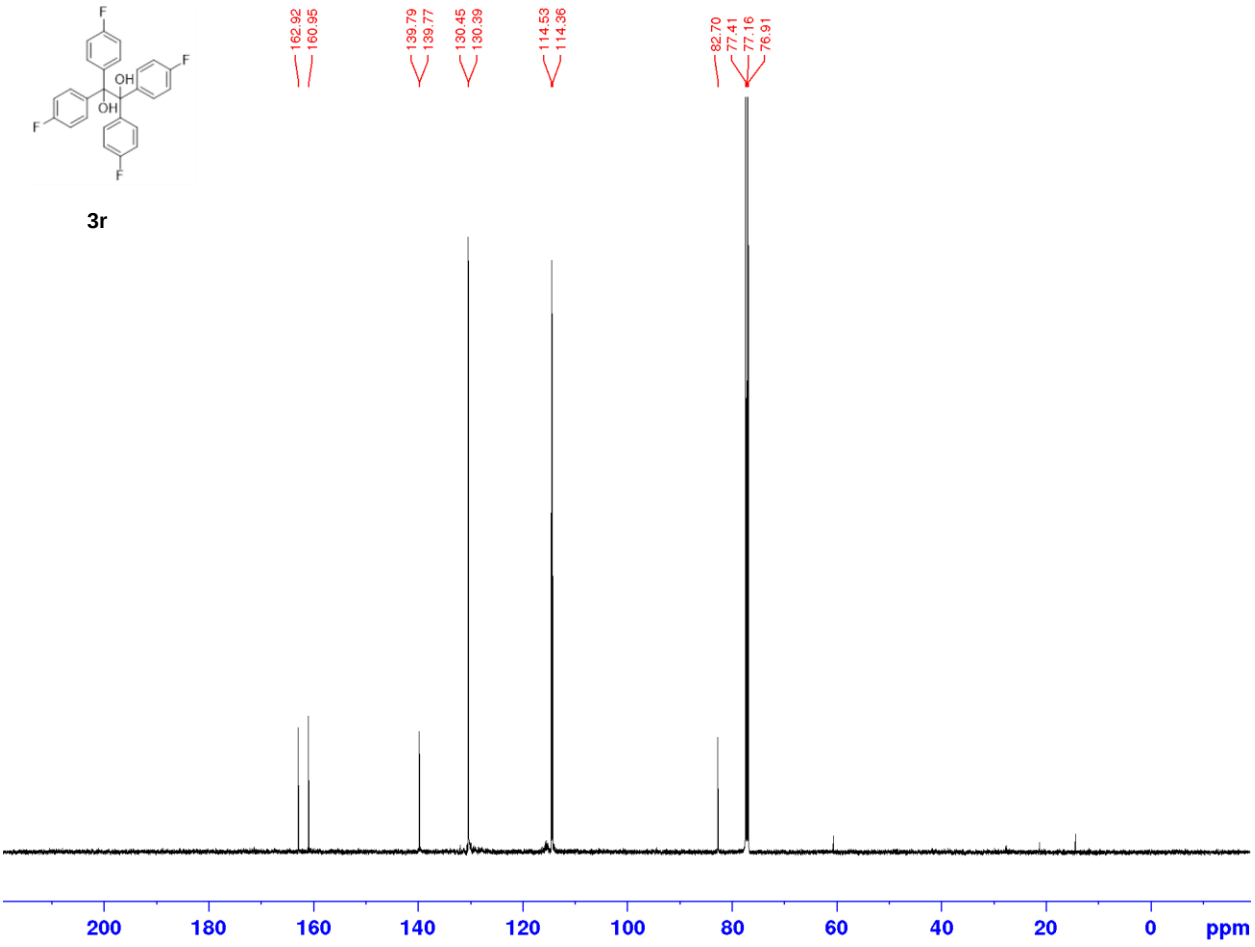
3r

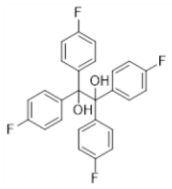

7.285
7.275
7.268
7.257
6.917
6.899
6.882


2.890

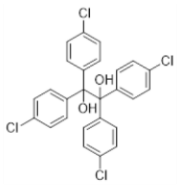

BRUKER


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
8.00
7.64
1.99


F
F
OH
OH
F
F
3r
162.92
160.95
139.79
139.77
130.45
130.39
114.53
114.36
82.70
77.41
77.16
76.91
200
180
160
140
120
100
80
60
40
20
0
ppm



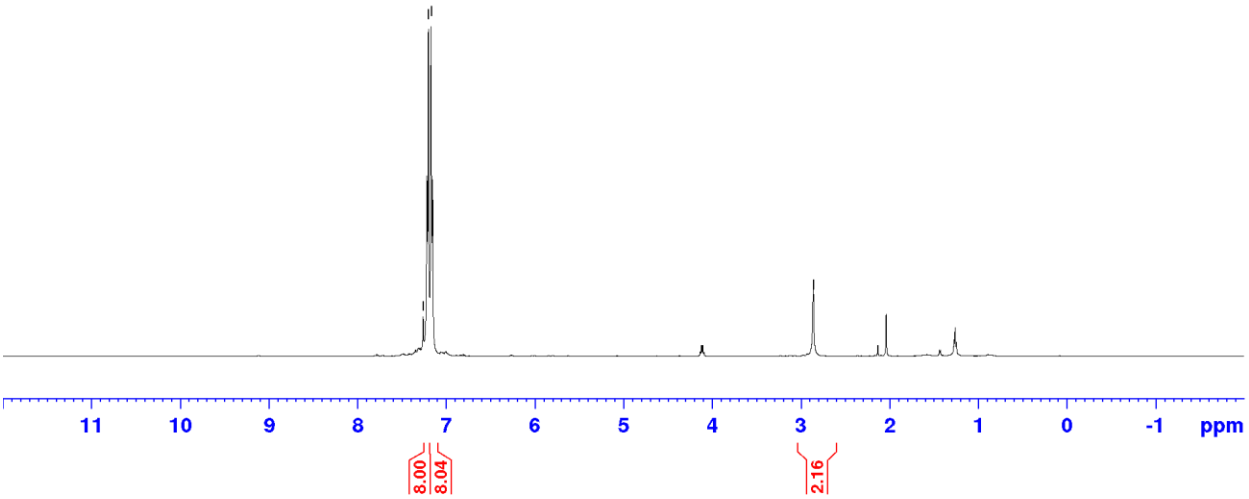
3r

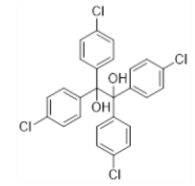

Cl
Cl
OH
OH
Cl
Cl


7.260
7.219
7.201
7.171
7.154


2.859

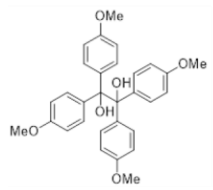
3s


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
8.00
8.04
2.16


Cl
Cl
OH
OH
Cl
Cl

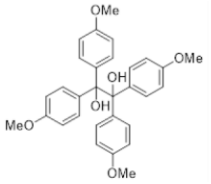
3s

142.13
133.63
129.98
127.87
82.63
77.41
77.16
76.91
200
180
160
140
120
100
80
60
40
20
0
ppm


OMe
OMe
OH
OH
MeO
OMe

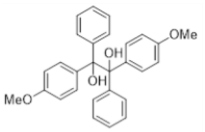
3t

7.260
7.196
7.178
6.712
6.694
3.759
2.905
11
10
9
8
7
6
5
4
3
2
1
0
ppm
8.00
7.97
12.28
1.98

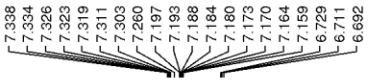

OMe
OMe
OH
OH
MeO
OMe

3t

158.33
136.84
129.96
112.62
82.85
77.41
77.16
76.90
55.24
200
180
160
140
120
100
80
60
40
20
0
ppm

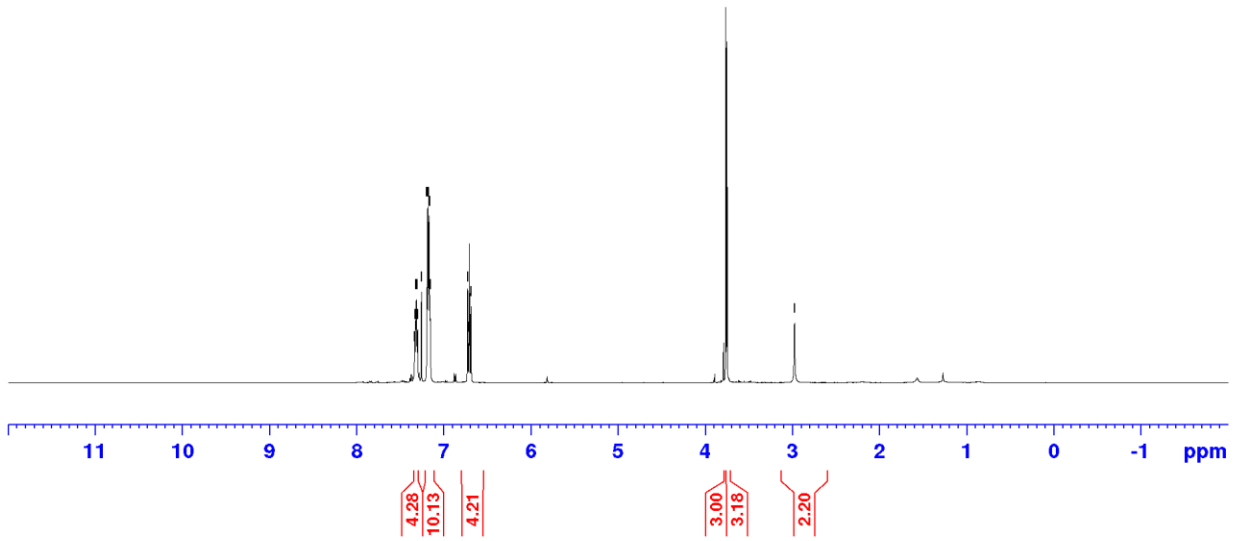

OMe
OH
OH
MeO

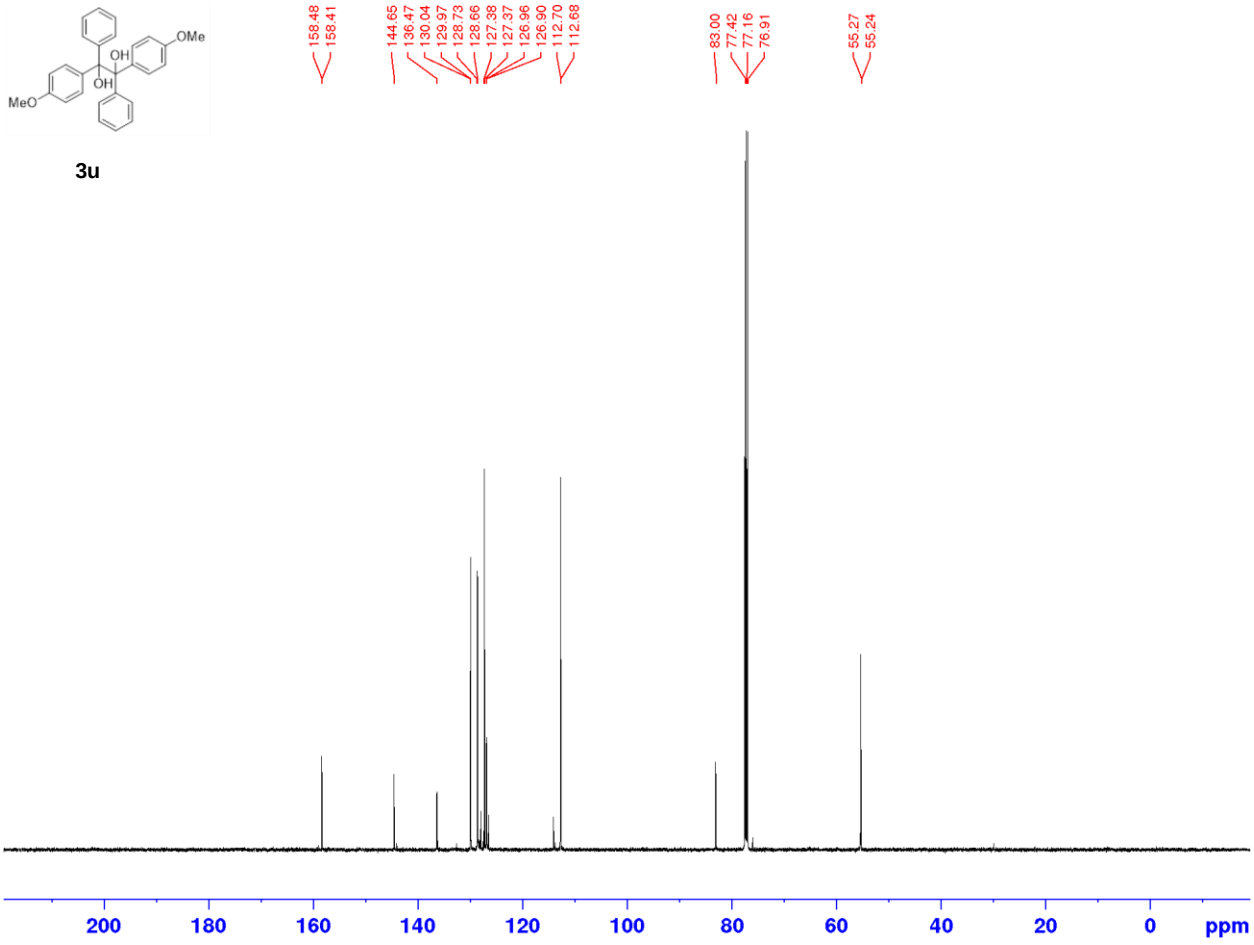
3u


7.338
7.334
7.326
7.323
7.319
7.311
7.303
7.260
7.197
7.193
7.188
7.184
7.180
7.173
7.170
7.164
7.159
6.729
6.711
6.692

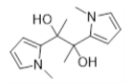

3.767
3.755
2.979


BRUKER

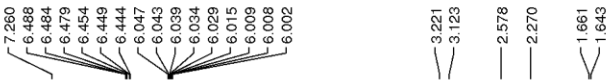

11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.28
10.13
4.21
3.00
3.18
2.20


OMe
OH
OH
MeO
158.48
158.41
144.65
136.47
130.04
129.97
128.73
128.66
127.38
127.37
126.96
126.90
112.70
112.68
83.00
77.42
77.16
76.91
55.27
55.24
200
180
160
140
120
100
80
60
40
20
0
ppm

3u


HO
N
N
OH

3v


7.260
6.488
6.484
6.479
6.454
6.449
6.444
6.047
6.043
6.039
6.034
6.029
6.015
6.009
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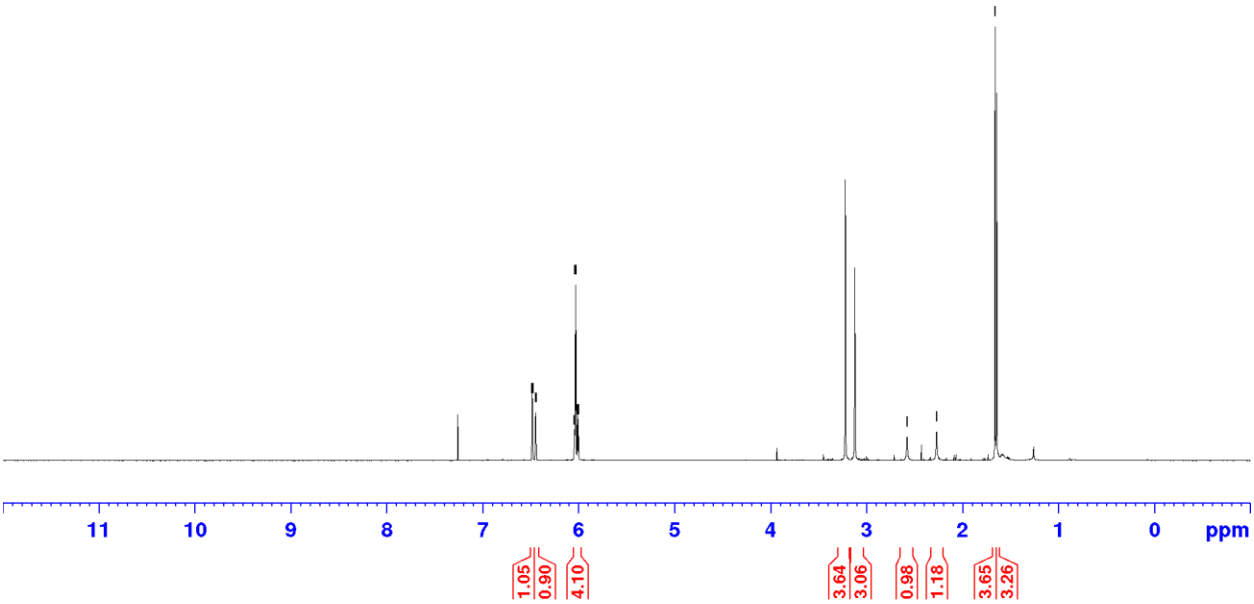
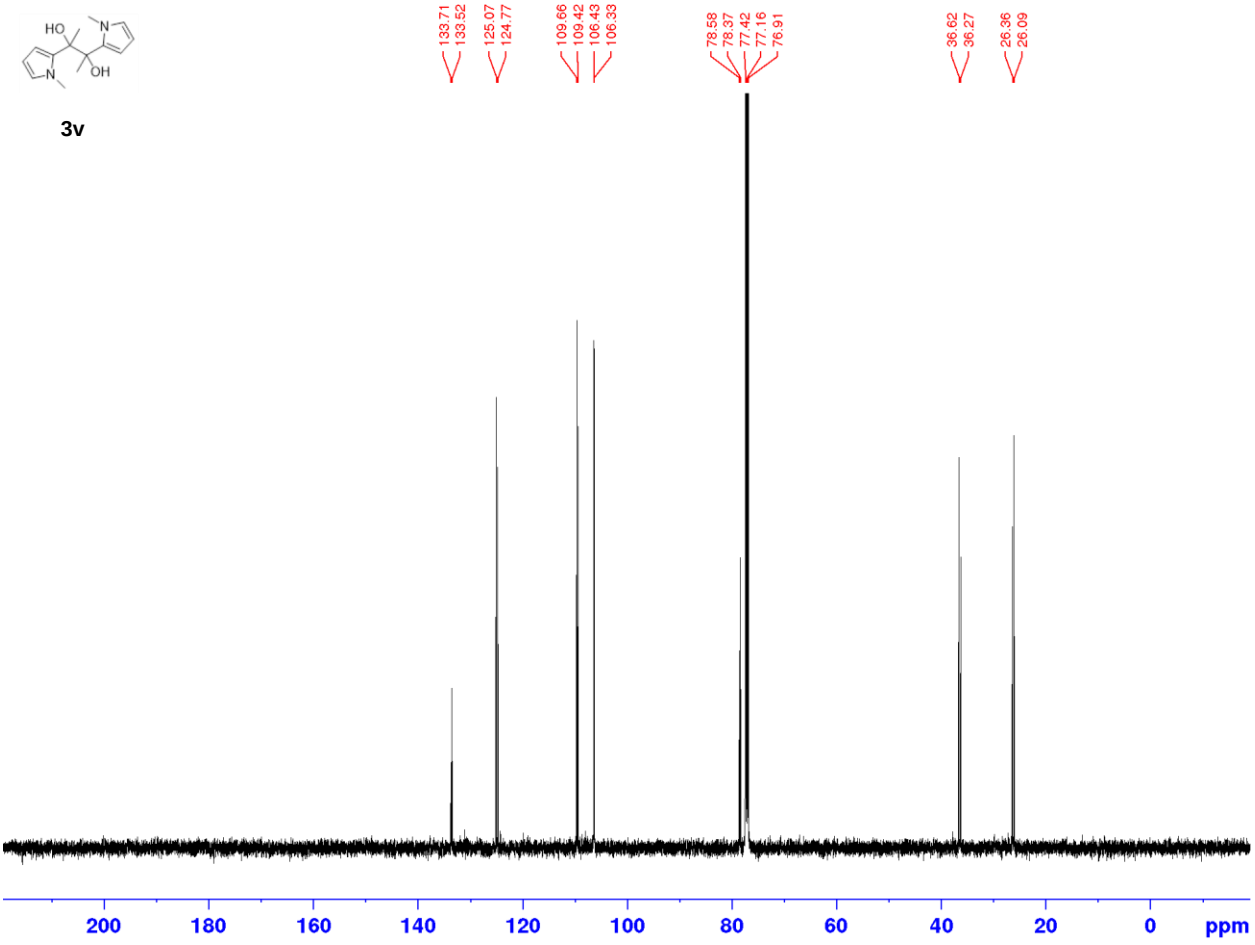
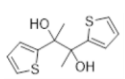
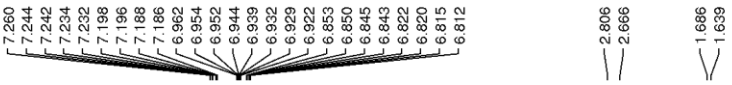
6.002
3.221
3.123
2.578
2.270
1.661
1.643


11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.05
0.90
4.10
3.64
3.06
0.98
1.18
3.65
3.26


HO
N
N
OH
3v
133.71
133.52
125.07
124.77
109.66
109.42
106.43
106.33
78.58
78.37
77.42
77.16
76.91
36.62
36.27
26.36
26.09
200
180
160
140
120
100
80
60
40
20
0
ppm


HO
S
S
OH

3w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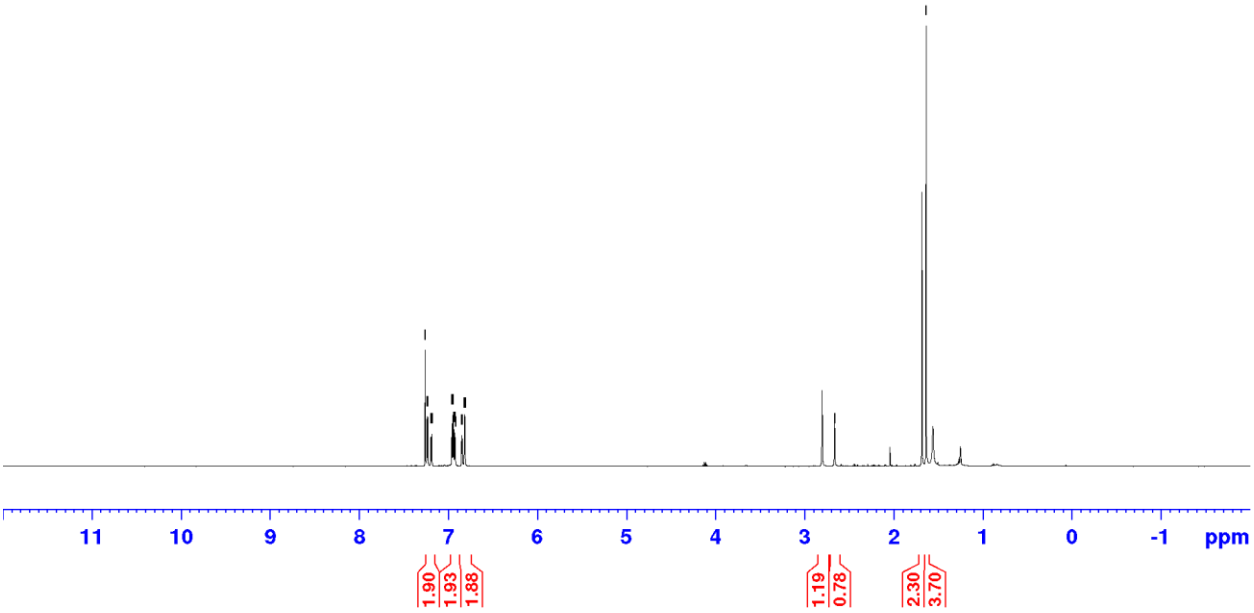
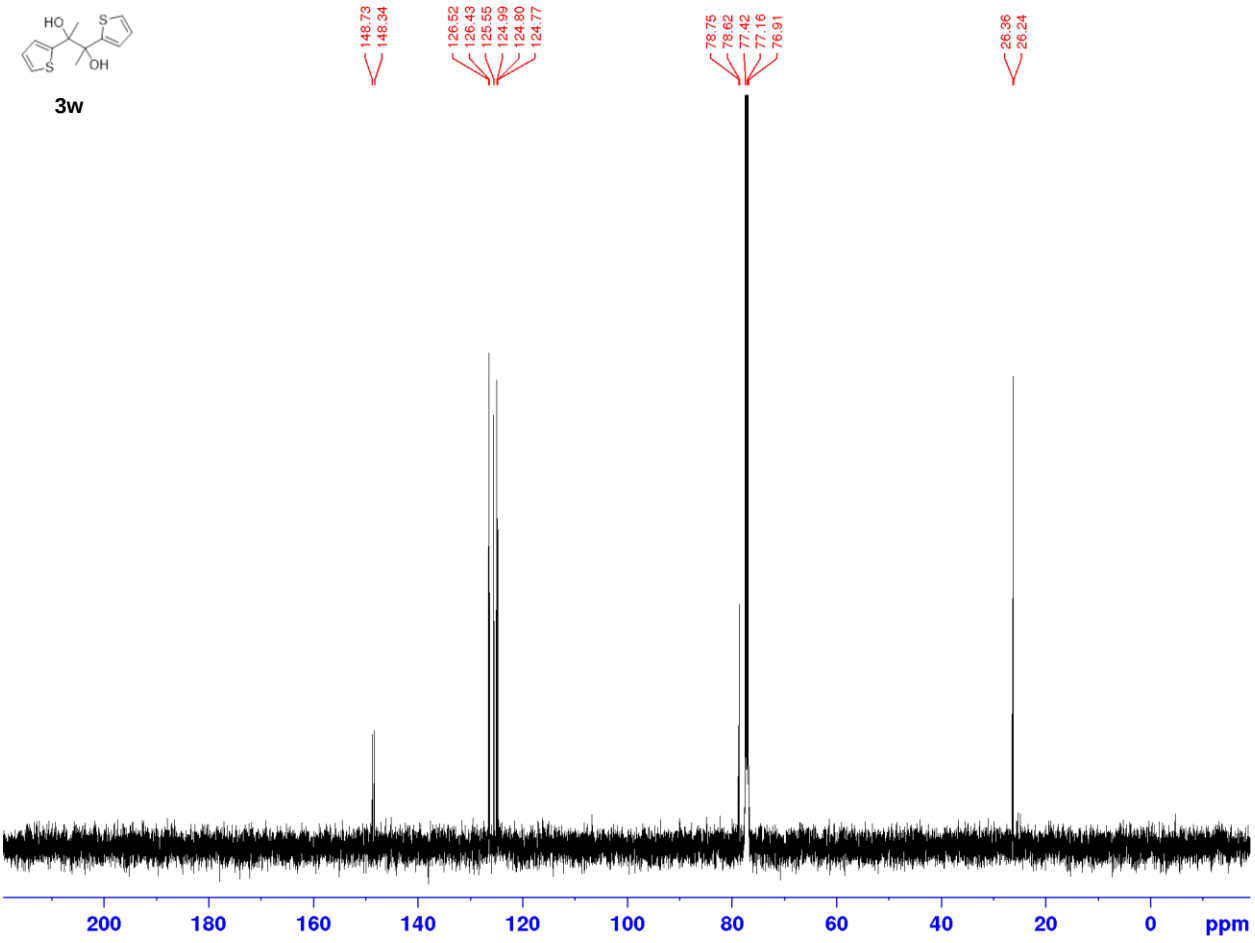
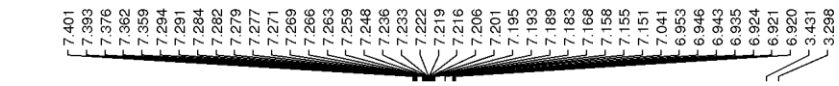
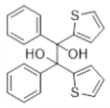
7.244
7.242
7.234
7.232
7.198
7.196
7.188
7.186
6.962
6.954
6.952
6.944
6.939
6.932
6.929
6.922
6.853
6.850
6.845
6.843
6.822
6.820
6.815
6.812
2.806
2.666
1.686
1.639

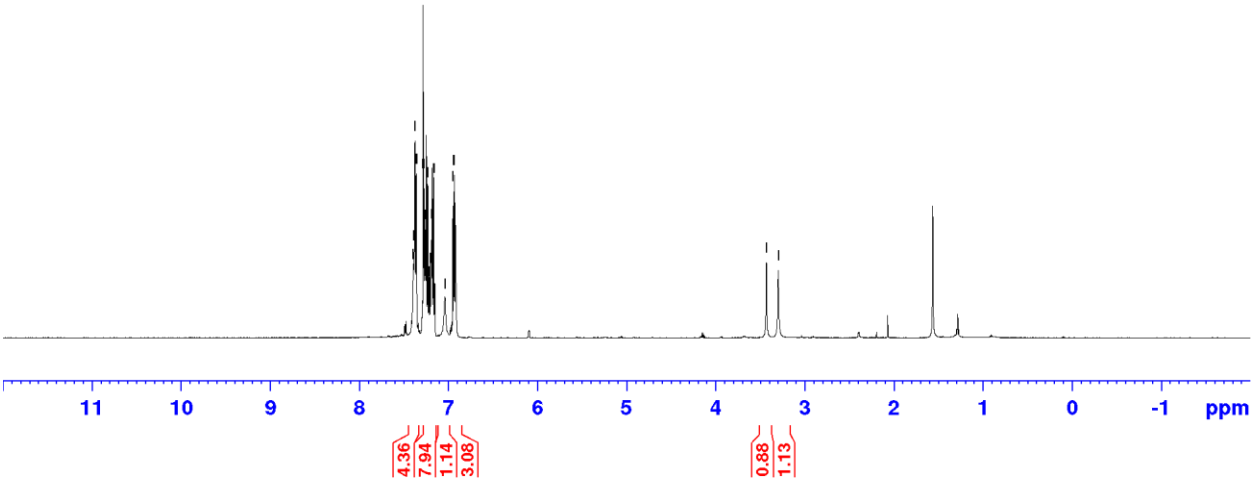

11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.90
1.93
1.88
1.19
0.78
2.30
3.70

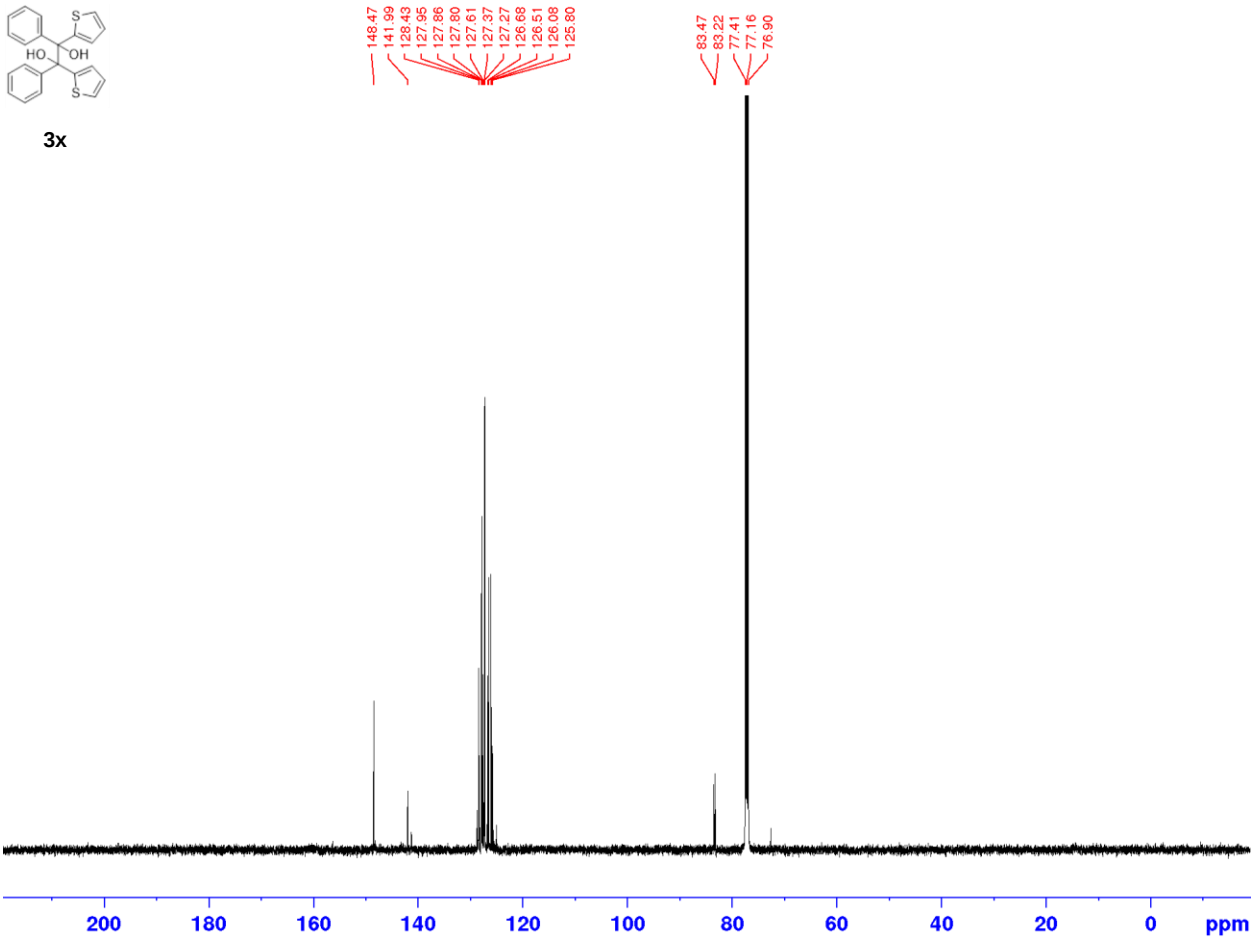

HO
S
S
OH
3w
148.73
148.34
126.52
126.43
125.55
124.99
124.80
124.77
78.75
78.62
77.42
77.16
76.91
26.36
26.24
200
180
160
140
120
100
80
60
40
20
0
ppm

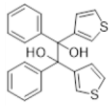

7.401
7.393
7.376
7.362
7.359
7.294
7.291
7.284
7.282
7.279
7.277
7.271
7.269
7.266
7.263
7.259
7.248
7.236
7.233
7.222
7.219
7.216
7.206
7.201
7.195
7.193
7.189
7.183
7.168
7.158
7.155
7.151
7.041
6.953
6.946
6.943
6.935
6.924
6.921
6.920
3.431
3.298


HO
OH
S
S

3x

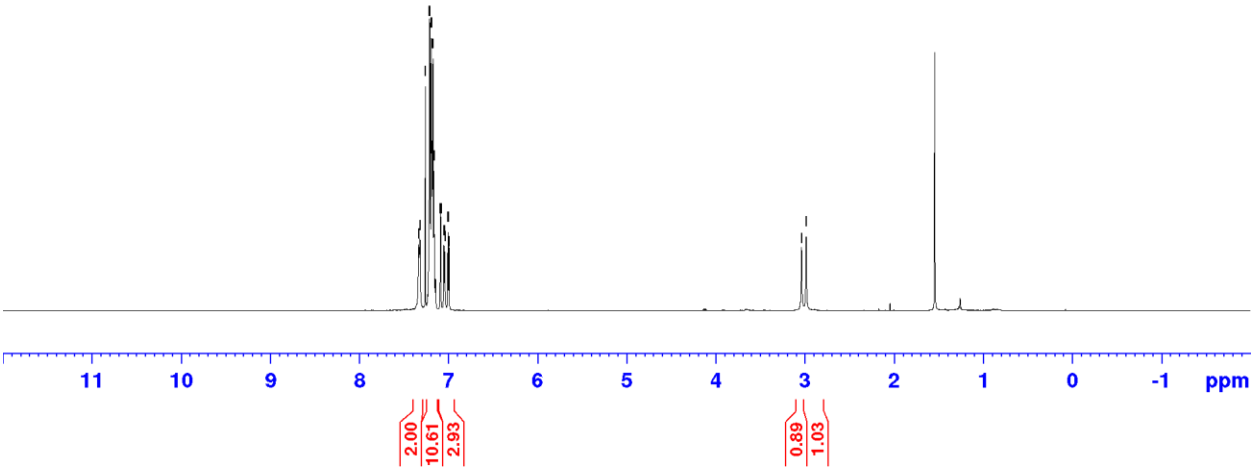

11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.36
7.94
1.14
3.08
0.88
1.13

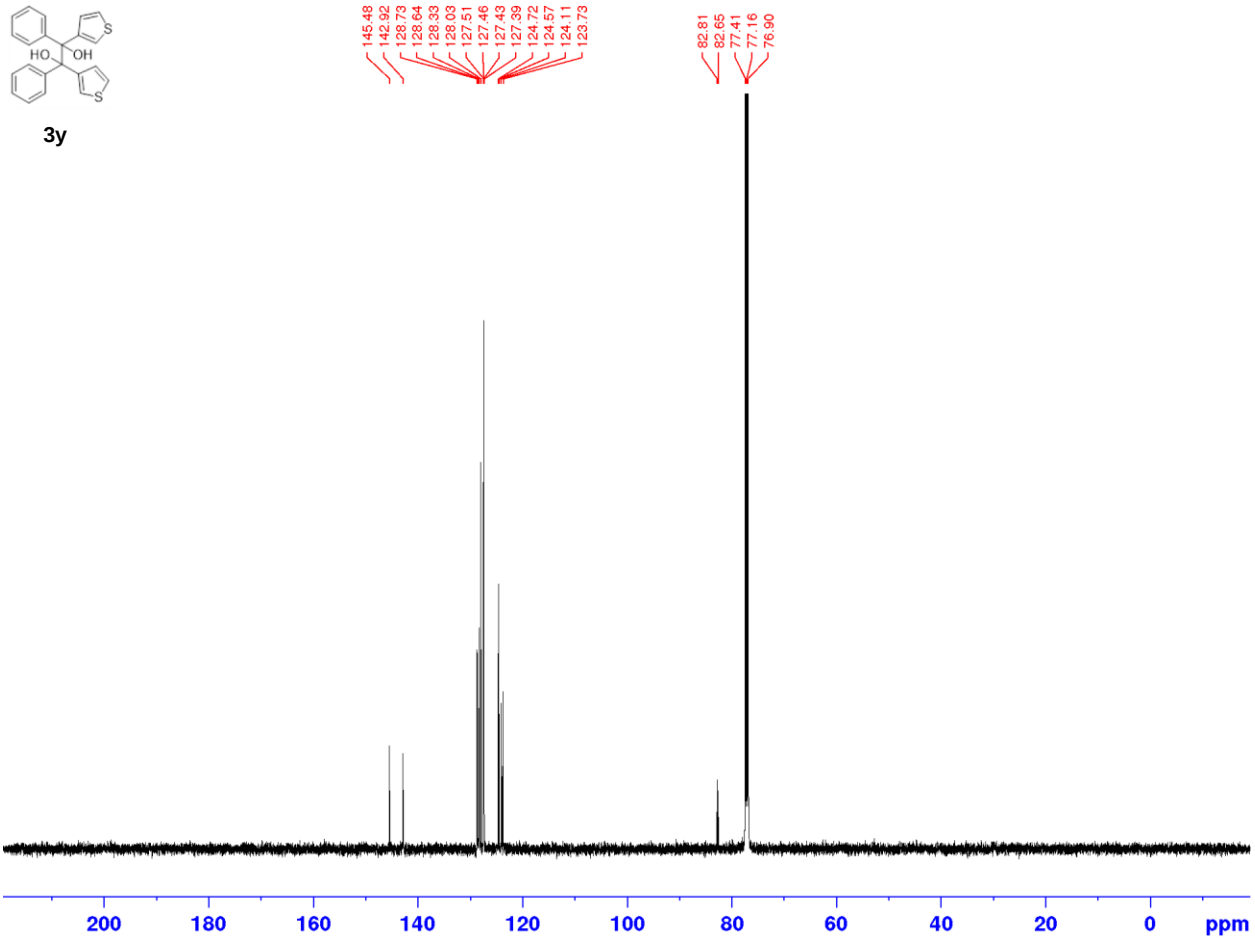

HO
OH
S
S
3x
148.47
141.99
128.43
127.95
127.86
127.80
127.61
127.37
127.27
126.68
126.51
126.08
125.80
83.47
83.22
77.41
77.16
76.90
200
180
160
140
120
100
80
60
40
20
0
ppm


HO
OH
S
S

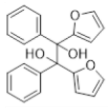
3y


7.336
7.321
7.260
7.220
7.212
7.197
7.194
7.189
7.187
7.183
7.179
7.174
7.164
7.094
7.091
7.088
7.085
7.053
7.051
7.043
7.041
7.007
7.005
6.997
6.995
3.039
2.986

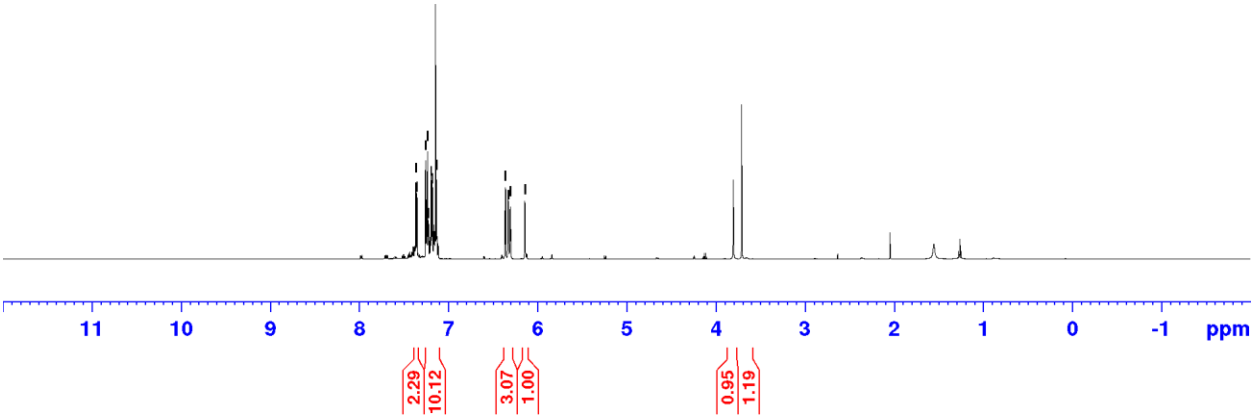

11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
10.61
2.93
0.89
1.03

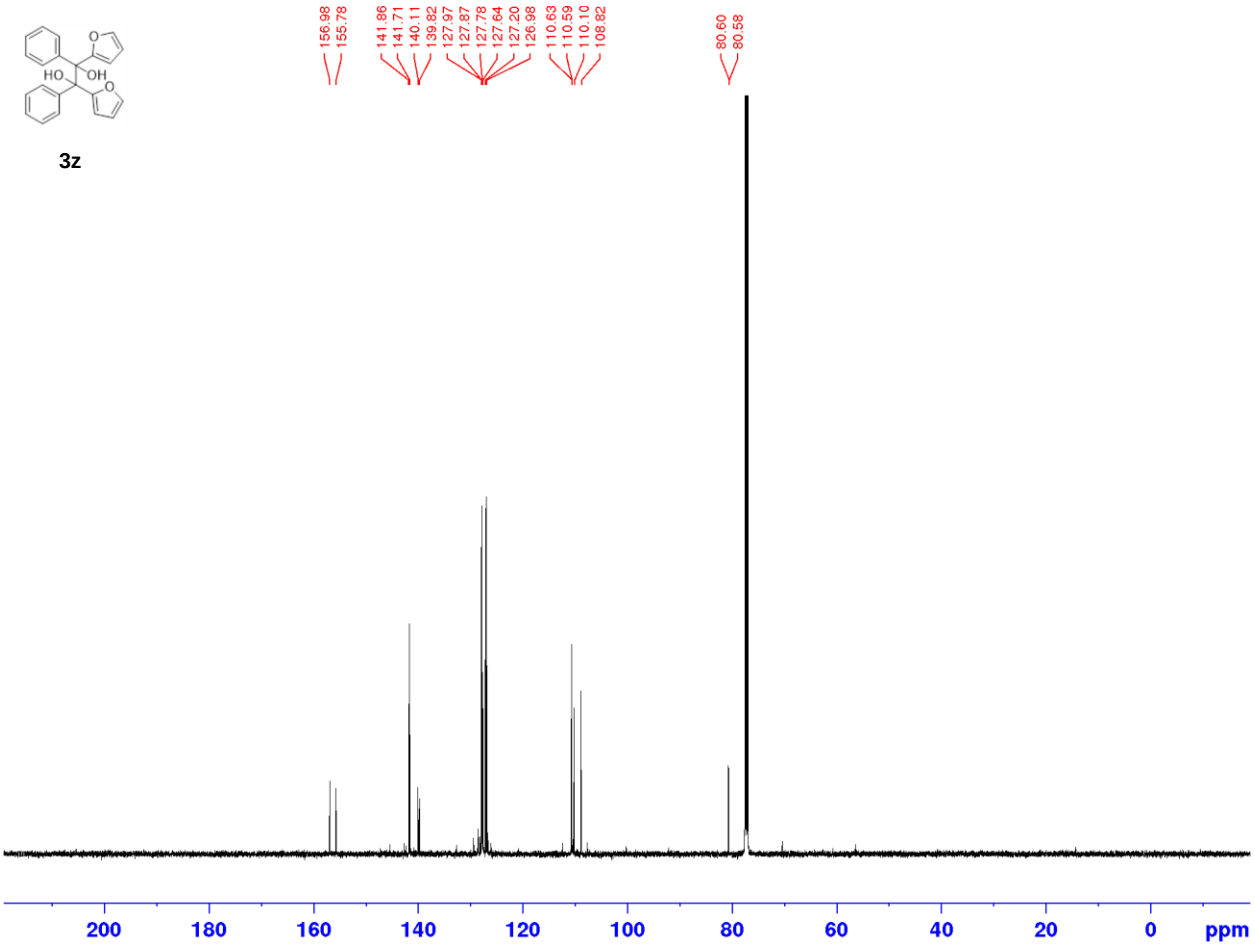

HO
OH
S
S
3y
145.48
142.92
128.73
128.64
128.33
128.03
127.51
127.46
127.43
127.39
124.72
124.57
124.11
123.73
82.81
82.65
77.41
77.16
76.90
200
180
160
140
120
100
80
60
40
20
0
ppm

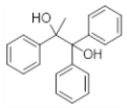

7.371
7.369
7.367
7.366
7.358
7.357
7.355
7.353
7.260
7.252
7.240
7.236
7.226
7.224
7.197
7.196
7.185
7.182
7.147
7.143
7.134
6.367
6.366
6.361
6.359
6.335
6.332
6.329
6.325
6.313
6.309
6.306
6.303
6.147
6.146
6.141
6.139
3.805
3.711

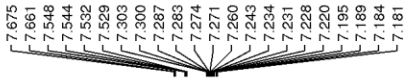

HO
OH
O
O

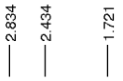
3z


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.29
10.12
3.07
1.00
0.95
1.19

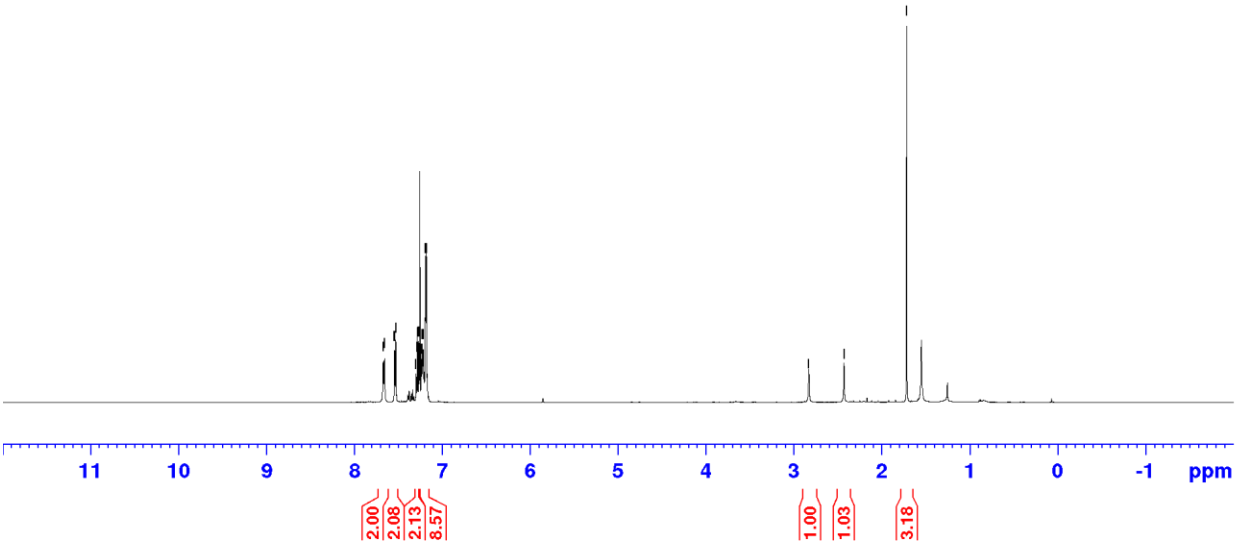

HO
OH
O
O
3z
156.98
155.78
141.86
141.71
140.11
139.82
127.97
127.87
127.78
127.64
127.20
126.98
110.63
110.59
110.10
108.82
80.60
80.58
200
180
160
140
120
100
80
60
40
20
0
ppm

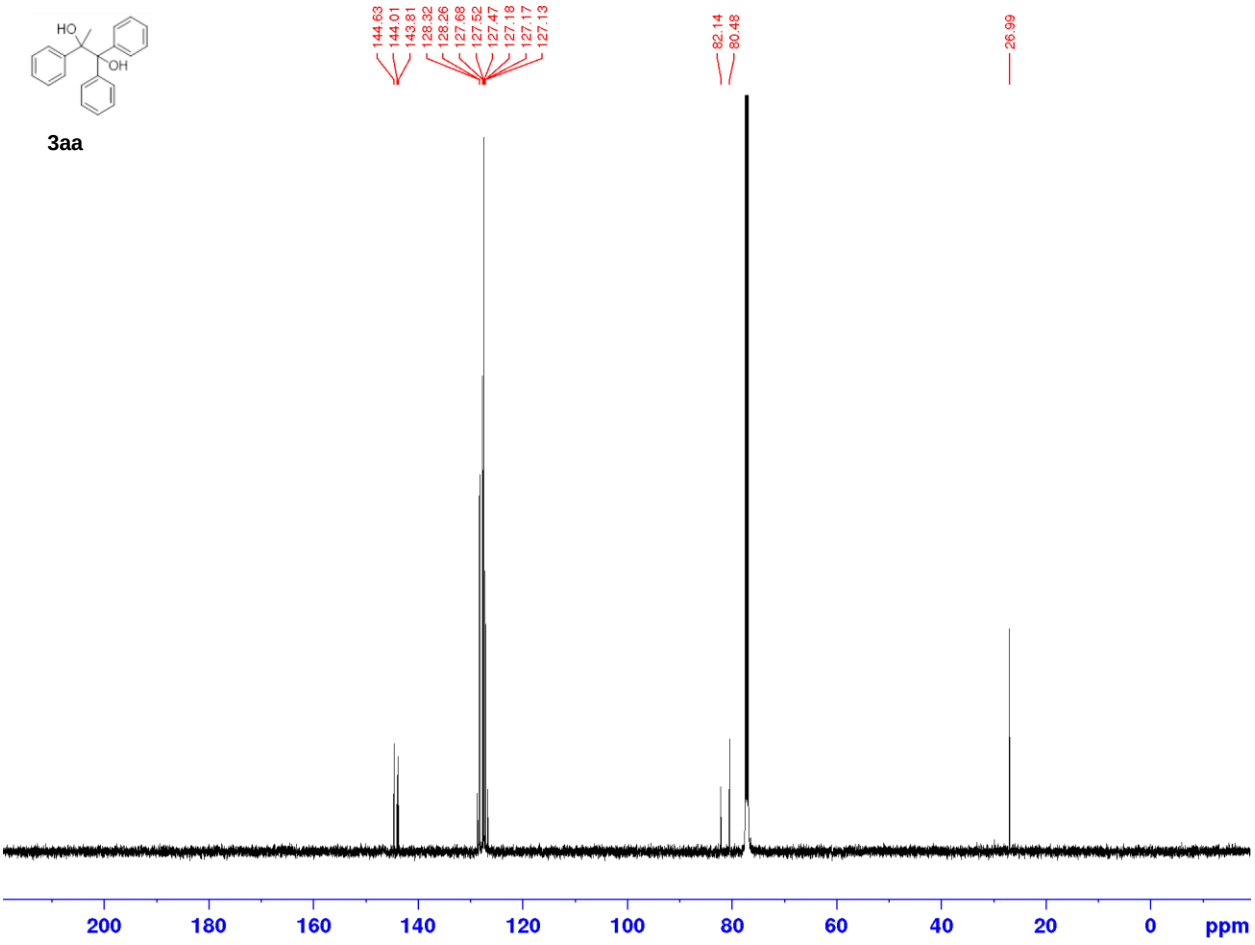

HO
OH

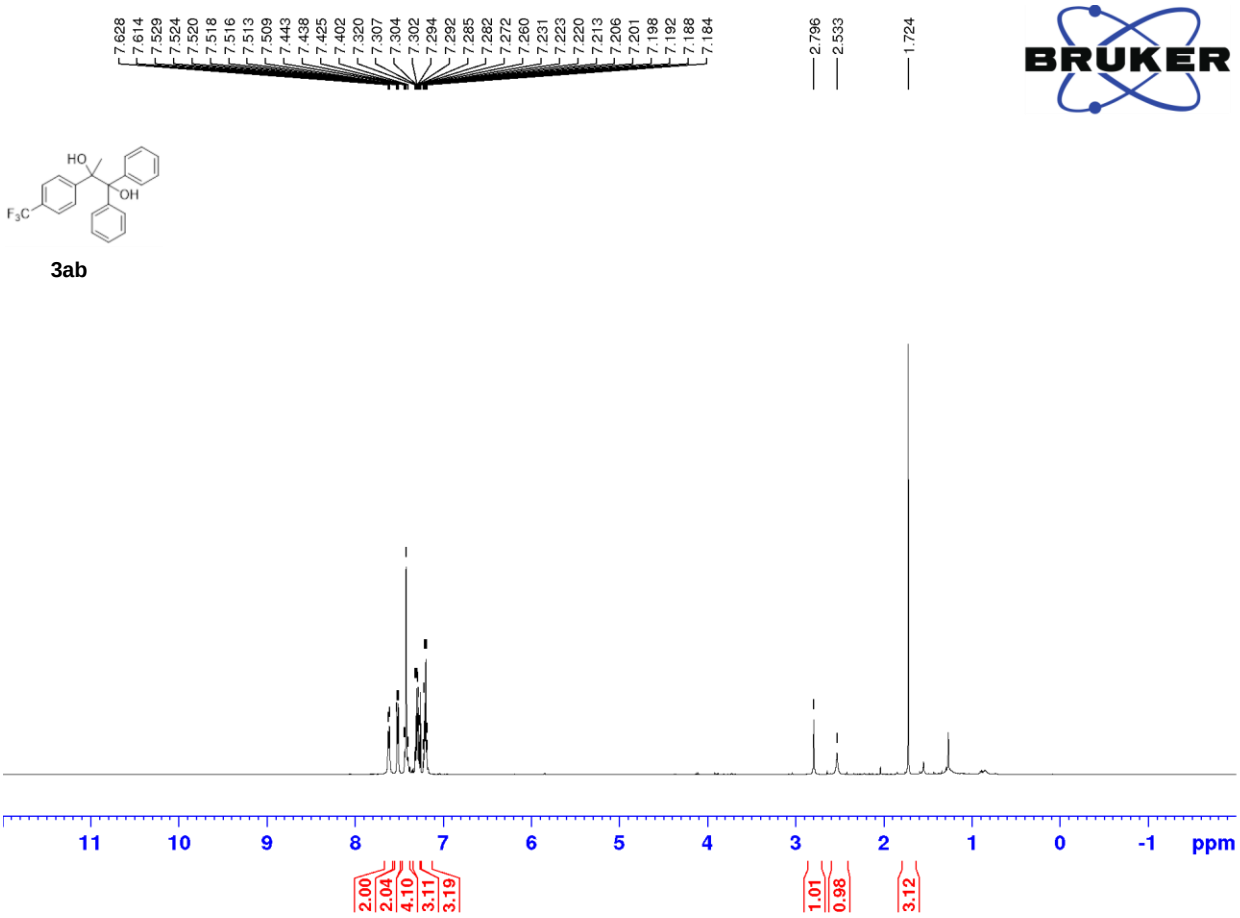

7.675
7.661
7.548
7.544
7.532
7.529
7.303
7.300
7.287
7.283
7.274
7.271
7.260
7.243
7.234
7.231
7.228
7.220
7.195
7.189
7.184
7.181


2.834
2.434
1.721

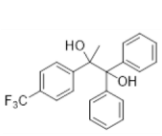
3aa

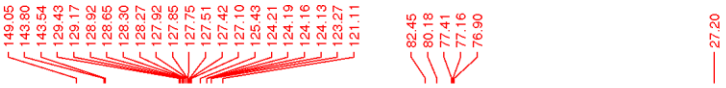

11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
2.08
2.13
8.57
1.00
1.03
3.18


HO
OH
3aa
144.63
144.01
143.81
128.32
128.26
127.68
127.52
127.47
127.18
127.17
127.13
82.14
80.48
26.99
200
180
160
140
120
100
80
60
40
20
0
ppm

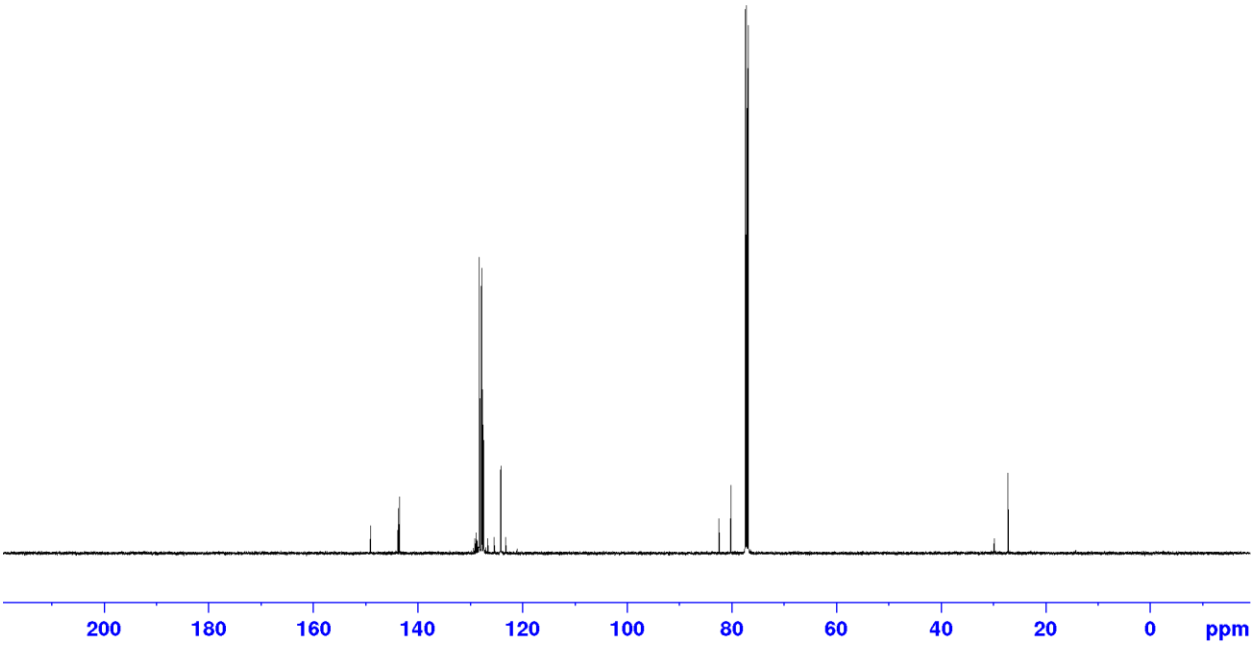

7.628
7.614
7.529
7.524
7.520
7.518
7.516
7.513
7.509
7.443
7.438
7.425
7.402
7.320
7.307
7.304
7.302
7.294
7.292
7.285
7.282
7.272
7.260
7.231
7.223
7.220
7.213
7.206
7.201
7.198
7.192
7.188
7.184
2.796
2.533
1.724
HO
OH
F3C
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
2.04
4.10
3.11
3.19
1.01
0.98
3.12

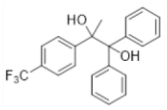
3ab


HO
OH
F3C

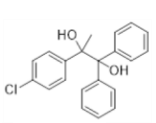

149.05
143.80
143.54
129.43
129.17
128.92
128.65
128.30
128.27
127.92
127.85
127.75
127.51
127.42
127.10
125.43
124.21
124.19
124.16
124.13
123.27
121.11
82.45
80.18
77.41
77.16
76.90
27.20

3ab


200
180
160
140
120
100
80
60
40
20
0
ppm

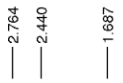


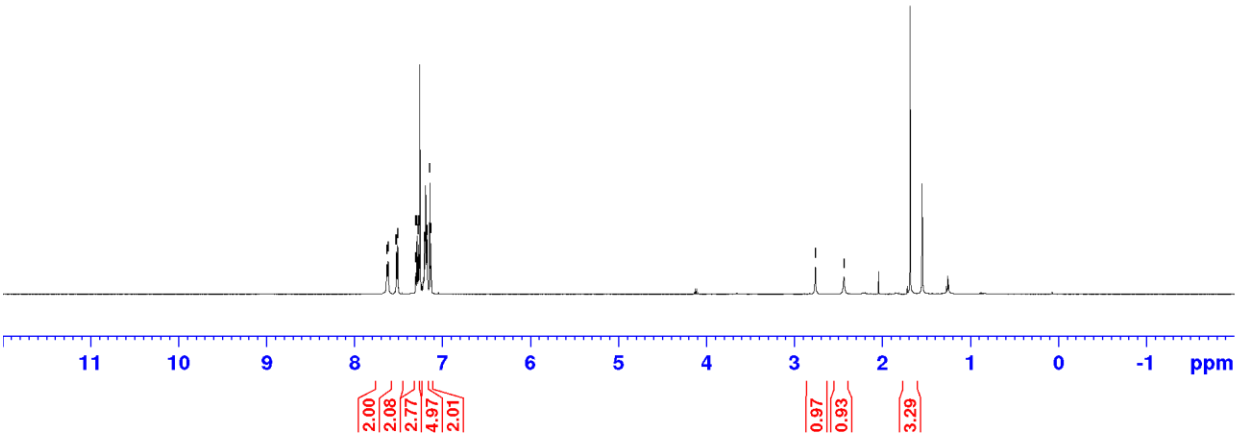
3ab

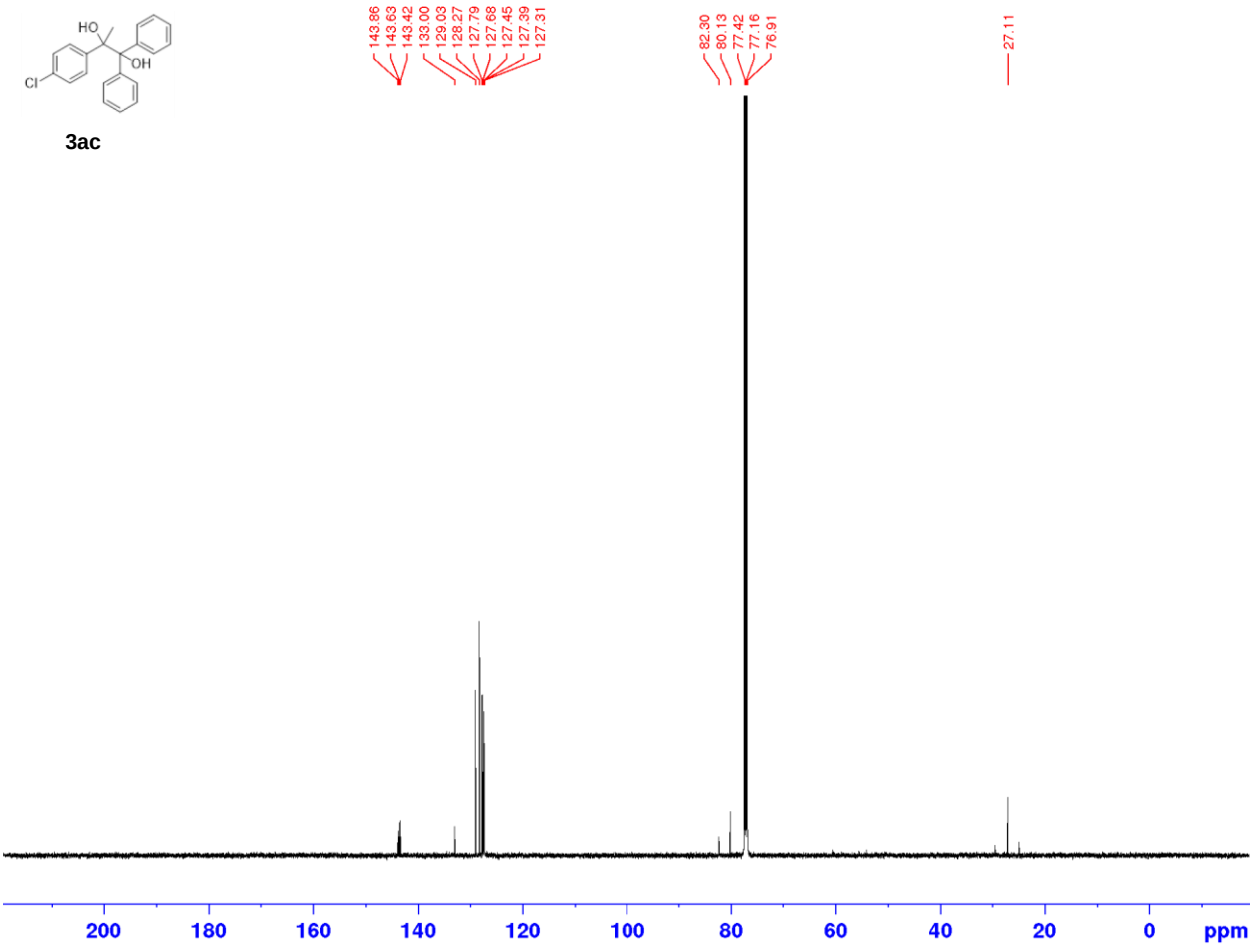

HO
OH
Cl

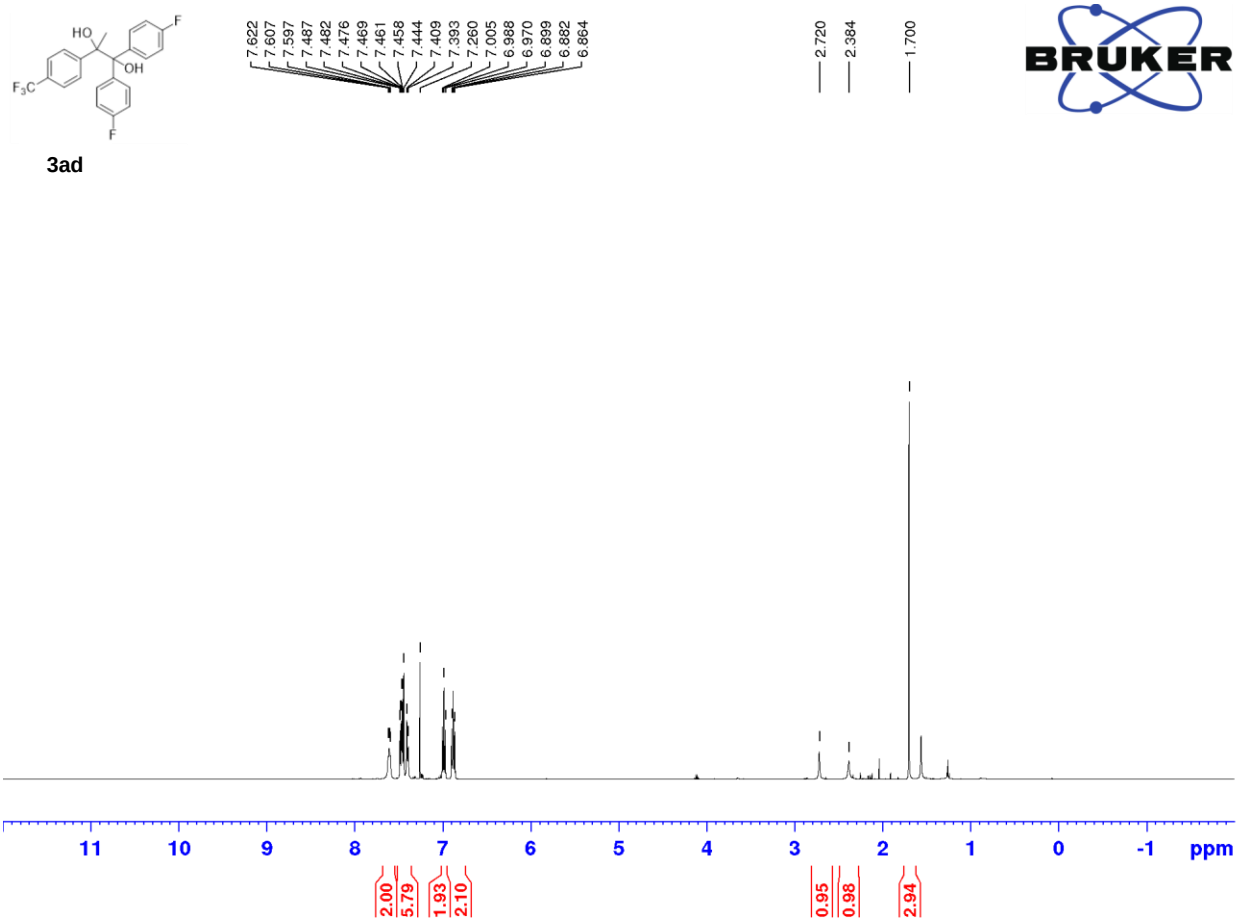
3ac


7.636
7.621
7.529
7.525
7.512
7.509
7.311
7.307
7.304
7.294
7.279
7.271
7.268
7.265
7.254
7.209
7.195
7.181
7.148
7.130

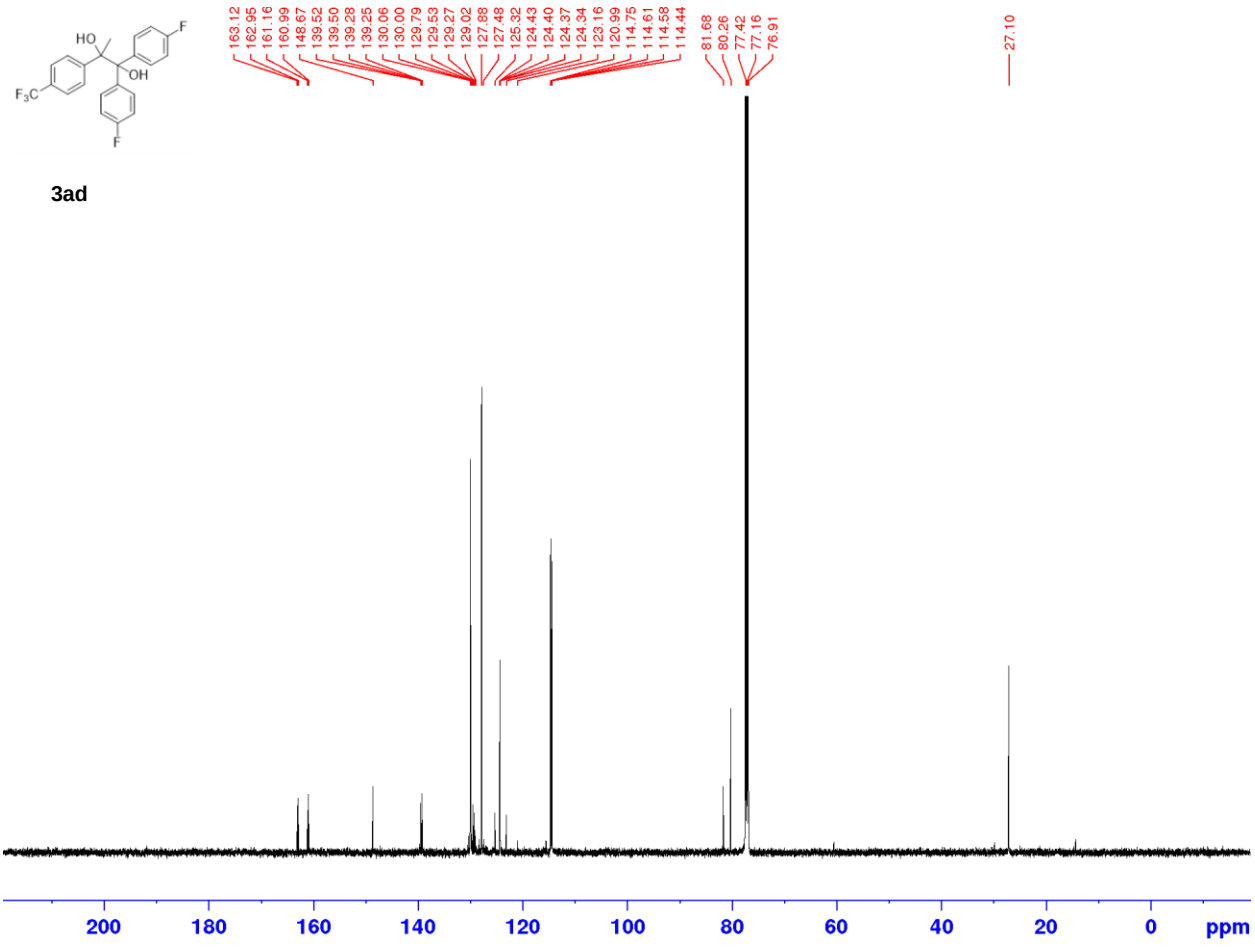

2.764
2.440
1.687


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
2.08
2.77
4.97
2.01
0.97
0.93
3.29

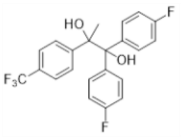

HO
OH
Cl
3ac
143.86
143.63
143.42
133.00
129.03
128.27
127.79
127.68
127.45
127.39
127.31
82.30
80.13
77.42
77.16
76.91
27.11
200
180
160
140
120
100
80
60
40
20
0
ppm


HO
OH
F
F
F3C
7.622
7.607
7.597
7.487
7.482
7.476
7.469
7.461
7.458
7.444
7.409
7.393
7.260
7.005
6.988
6.970
6.899
6.882
6.864
2.720
2.384
1.700
BRUKER
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
5.79
1.93
2.10
0.95
0.98
2.94

3ad

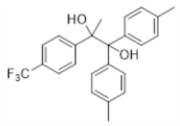

HO
OH
F
F
F3C
163.12
162.95
161.16
160.99
148.67
139.52
139.50
139.28
139.25
130.06
130.00
129.79
129.53
129.27
129.02
127.88
127.48
125.32
124.43
124.40
124.37
124.34
123.16
120.99
114.75
114.61
114.58
114.44
81.68
80.26
77.42
77.16
76.91
27.10
200
180
160
140
120
100
80
60
40
20
0
ppm

3ad


HO
OH
F
F3C
F

3ad

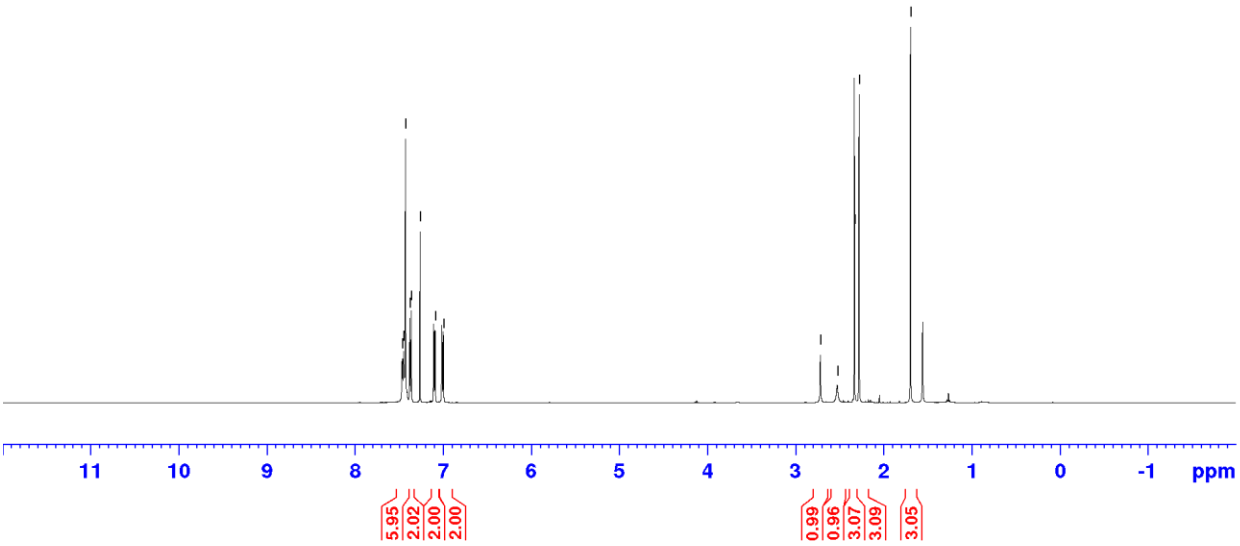
-62.50
-115.10
-115.17
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm

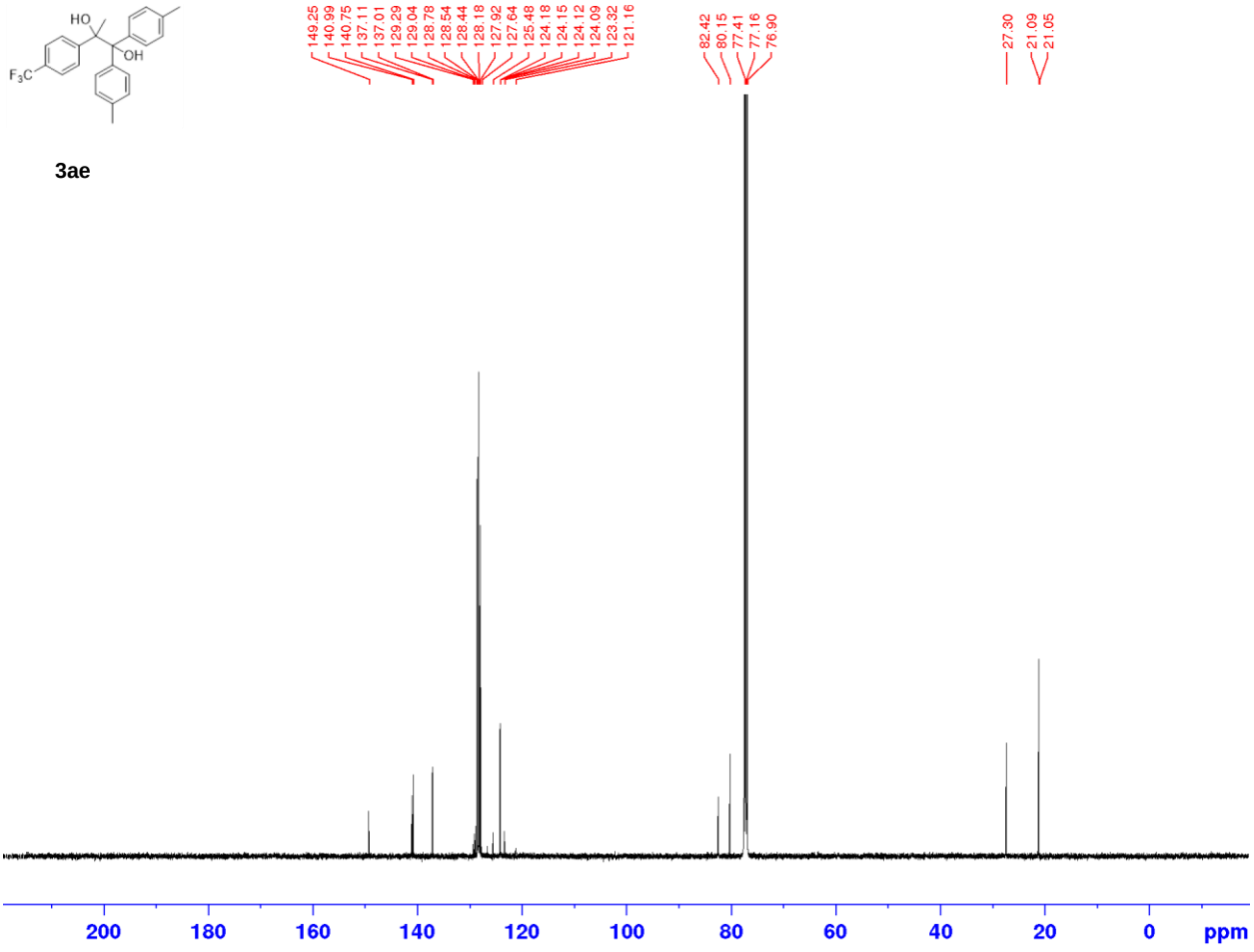

HO
OH
F3C


7.462
7.445
7.425
7.375
7.358
7.260
7.105
7.088
7.012
6.995

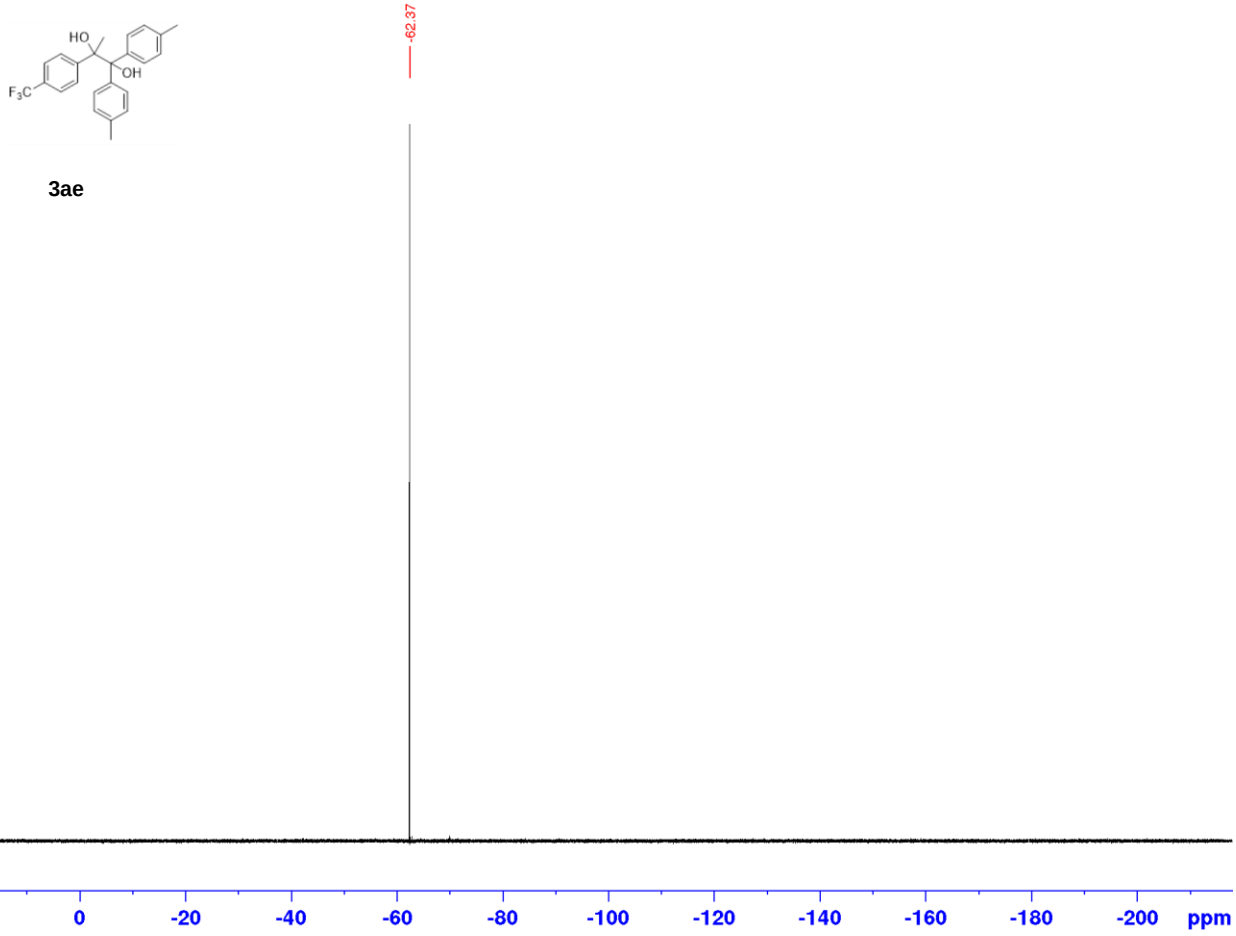

2.715
2.524
2.330
2.276
1.693

3ae

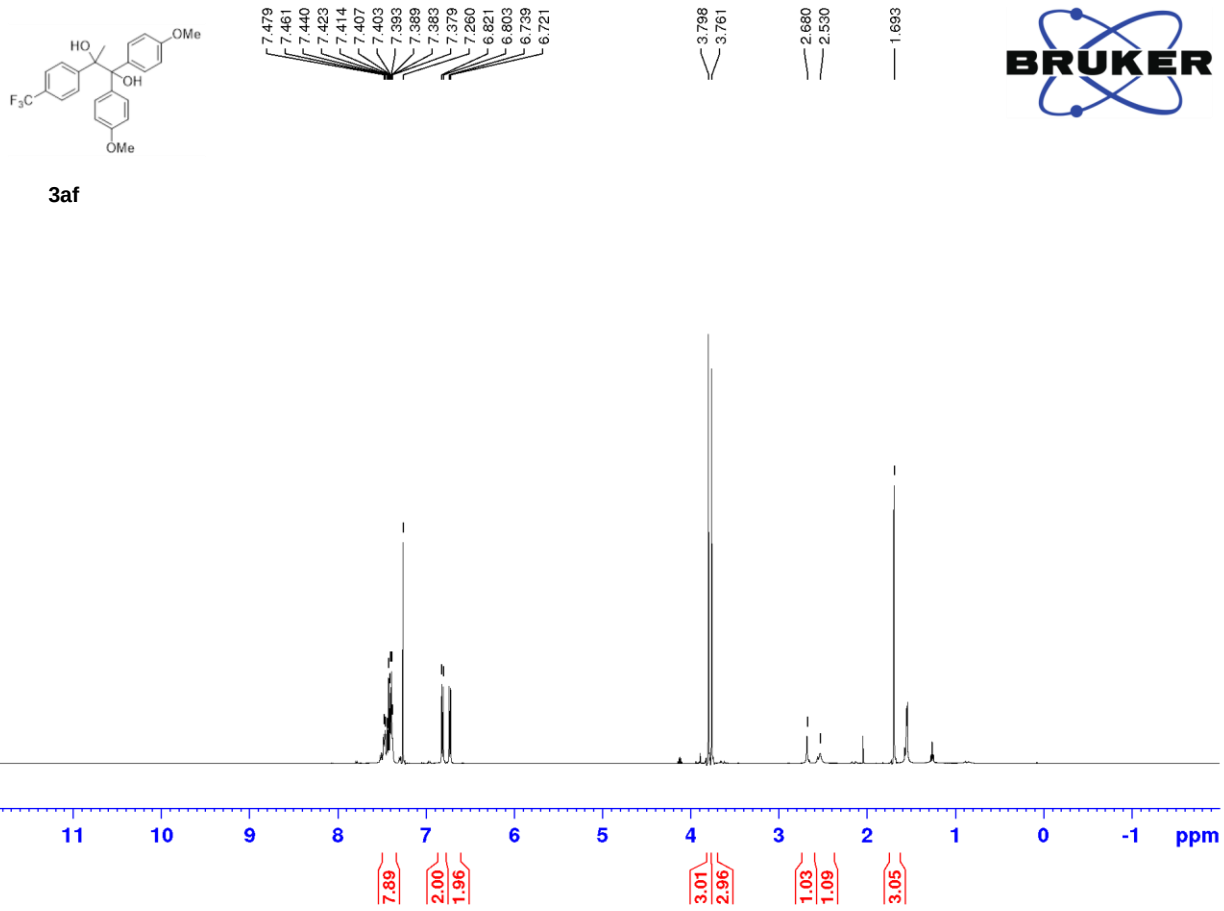

11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
5.95
2.02
2.00
2.00
0.99
0.96
3.07
3.09
3.05


HO
OH
F3C
149.25
140.99
140.75
137.11
137.01
129.29
129.04
128.78
128.54
128.44
128.18
127.92
127.64
125.48
124.18
124.15
124.12
124.09
123.32
121.16
82.42
80.15
77.41
77.16
76.90
27.30
21.09
21.05
200
180
160
140
120
100
80
60
40
20
0
ppm

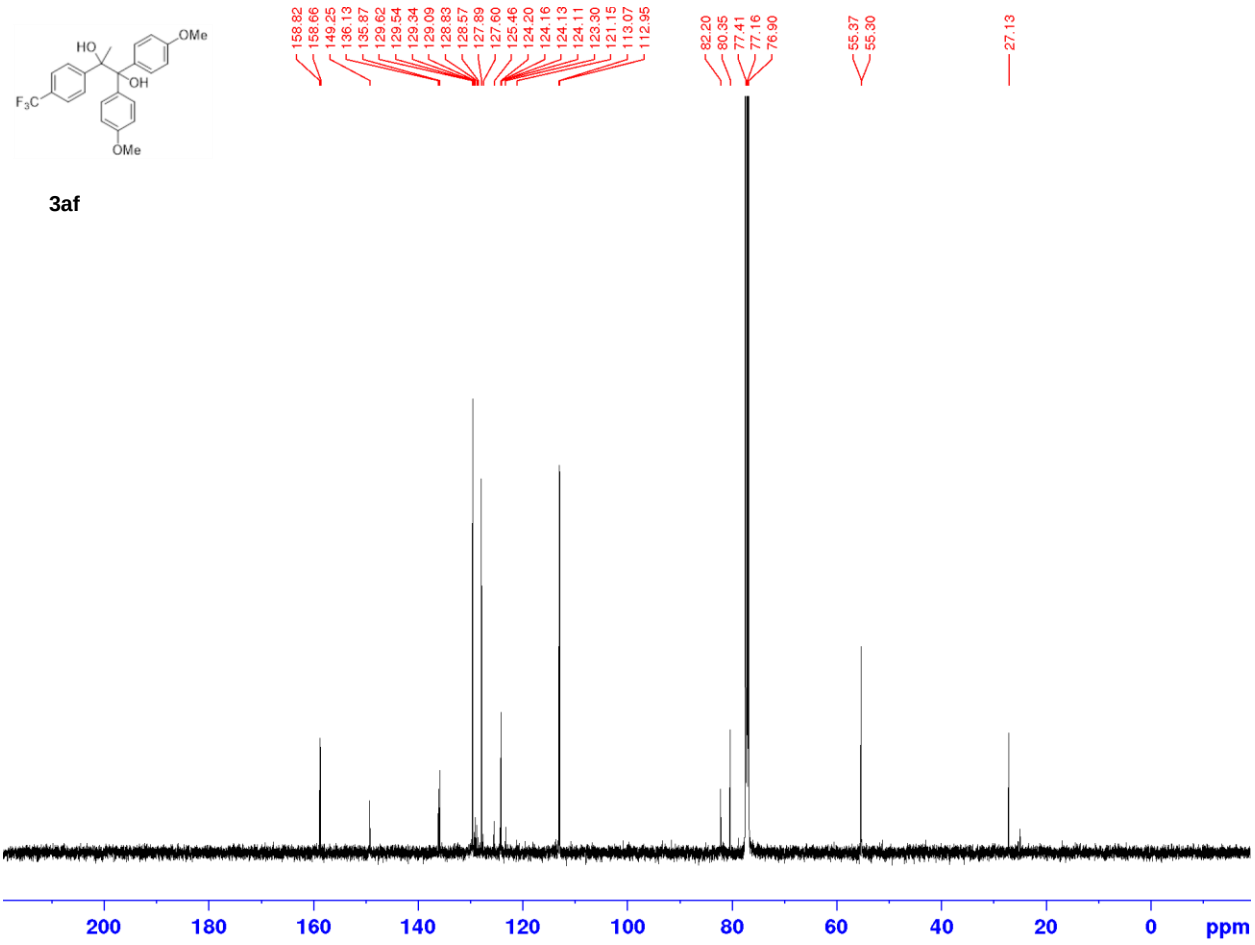
3ae



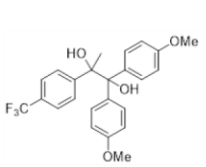
3ae


HO
OMe
OH
F3C
OMe
7.479
7.461
7.440
7.423
7.414
7.407
7.403
7.393
7.389
7.383
7.379
7.260
6.821
6.803
6.739
6.721
3.798
3.761
2.680
2.530
1.693
BRUKER
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
7.89
2.00
1.96
3.01
2.96
1.03
1.09
3.05

3af


HO
OMe
OH
F3C
OMe
158.82
158.66
149.25
136.13
135.87
129.62
129.54
129.34
129.09
128.83
128.57
127.89
127.60
125.46
124.20
124.16
124.13
124.11
123.30
121.15
113.07
112.95
82.20
80.35
77.41
77.16
76.90
55.37
55.30
27.13
200
180
160
140
120
100
80
60
40
20
0
ppm

3af



3af