

Supporting Information

Oxidative Deprotection of *p*-Methoxybenzyl Ethers via Metal-Free Photoredox Catalysis

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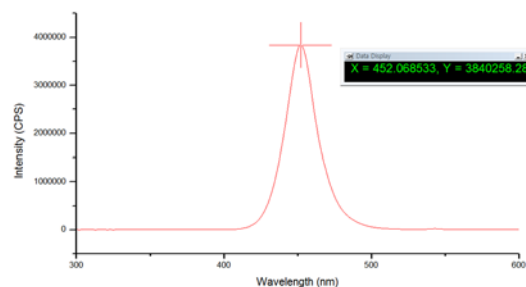
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I. Reaction setup

Irradiation of photochemical reactions was carried out using two MR16 5W blue LED spotlight lamp for milligram scale reaction while Kessil 40W (Model No: A160WE Tuna Blue) spotlight lamp was used for gram scale reaction. The pictures of two utilized spotlight lamps and their description are given below:



MR16 5W blue LED spotlight lamp



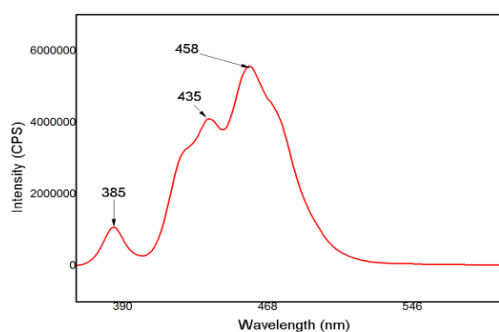
Emission spectrum of 5W blue LED

	Specification
Power	5 W
Voltage	12 V
Wavelength	452 nm

Figure S1A. Description for 5 W blue LED spotlight lamp



Kessil 40W Spotlight lamp



Emission spectrum of Kessil (40 W) source

	Specification
Power	40 W (max)
Voltage	12 V
Wavelength	385, 435, 458 nm

Figure S1B. Description for Kessil (40 W) spotlight lamp

To milligram scale, two MR16 5W blue LEDs spotlight lamp are positioned 2cm away from the reaction vial using customized reactor that was made by acrylic plate.

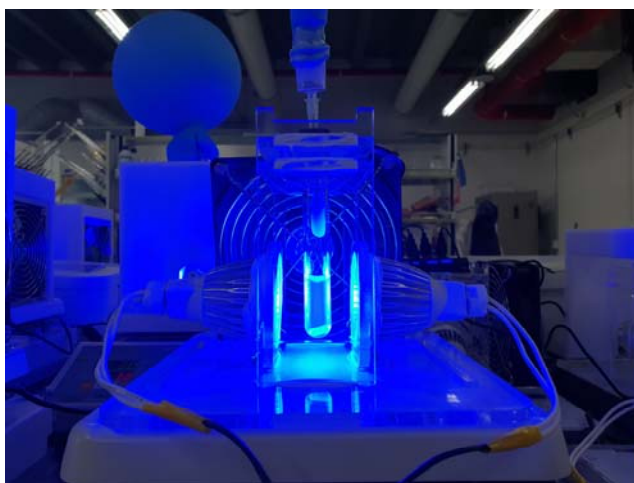


Figure S2. Milligram scale reaction set up

For gram scale reaction, one Kessil spotlight lamp (40 W) is positioned 5cm away from the reaction flask as shown in figure S3.

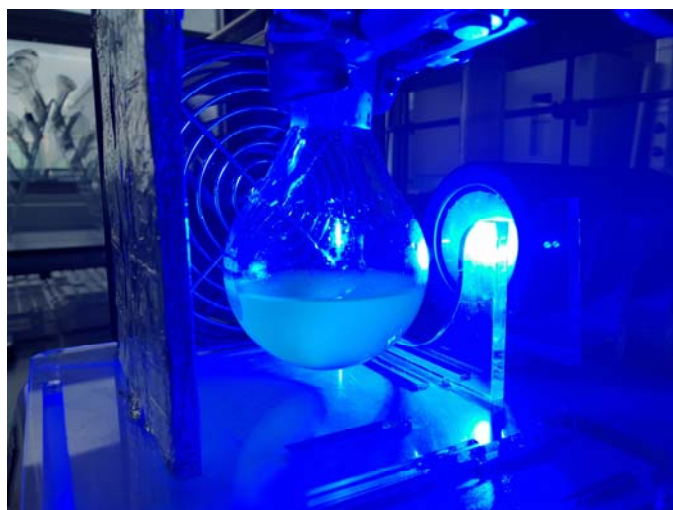


Figure S3. Gram scale reaction set up

In the optimized reaction conditions, the reaction is not significantly affected by temperature. The fan was used or not used according to the external temperature to maintain 20 ~ 30 °C in reactor.

II. Mechanistic studies

A. UV-Visible study

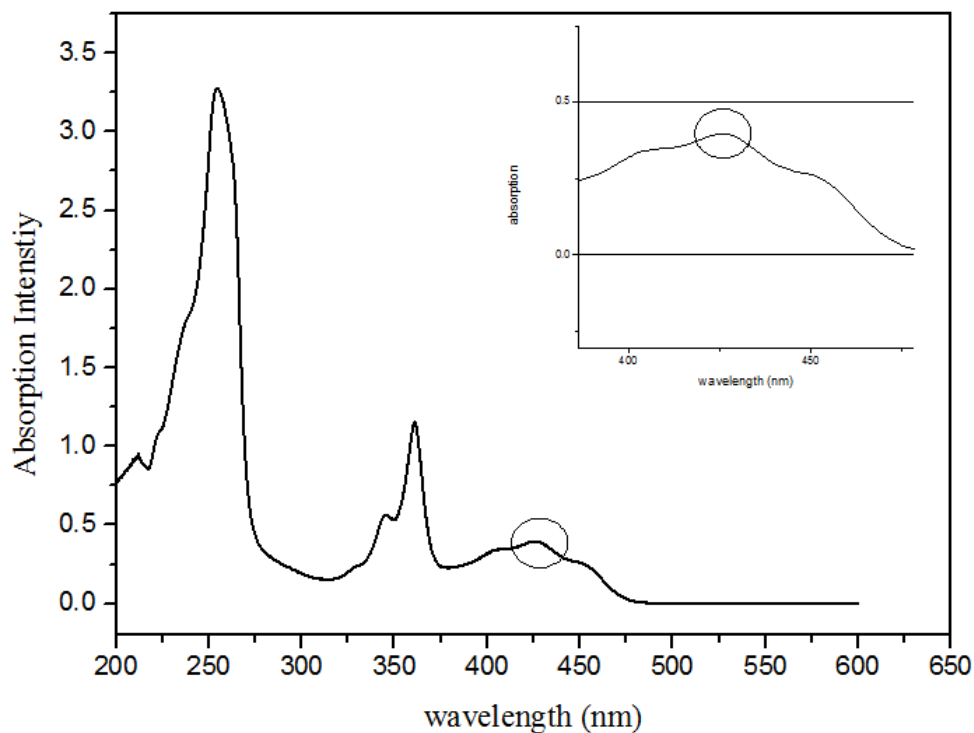


Figure S4. UV-Visible absorption spectra of 9-mesityl-10-methylacridinium perchlorate

A solution of 1.0×10^{-5} M in CH_3CN of the catalyst 9-mesityl-10-methylacridinium perchlorate ($\text{Acr}^+\text{-Mes}$) was prepared and UV-Visible absorption spectrum was measured. This graph showed that 9-mesityl-10-methylacridinium perchlorate has absorption at 430~455 wavelength (blue LED).

B. Luminescence quenching Study

$\text{Acr}^+\text{-Mes}$ was excited at 435 nm in $\text{CH}_3\text{CN}/\text{H}_2\text{O}$ solution (5:1) and the emission intensity was observed at ~ 499 nm. Both CH_3CN and H_2O were degassed with a stream of argon gas for 30 min. In a typical experiment, the emission spectrum of a 1.0×10^{-3} (M) solution of $\text{Acr}^+\text{-Mes}$ in $\text{CH}_3\text{CN}/\text{H}_2\text{O}$ solution (5:1) was collected. Then, **1a** was added to the measured solution in a quartz cuvette and the emission spectrum of the sample was collected. I_0 and I signify the intensities of the emission in the absence and presence of the quencher at ~ 499 nm. All the emission spectra were collected using Horiba Fluoromax-4P spectrophotometer.

The steady decrease of the emission intensity of the $\text{Acr}^+\text{-Mes}$ catalyst solution with the gradual increase of the amount of **1a** as presented in Figure S5 and Figure S6 supports that reaction mechanism occurring through reductive quenching cycle.

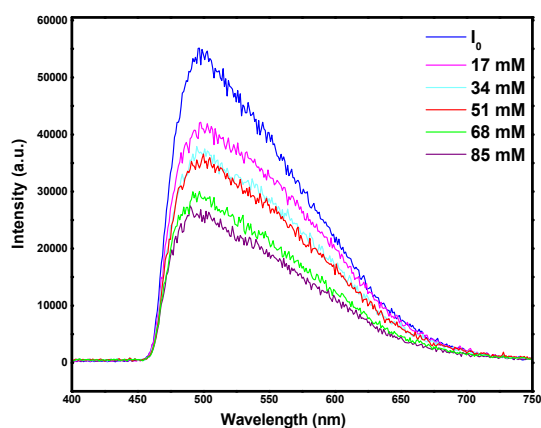


Figure S5. Luminescence quenching by **1a**

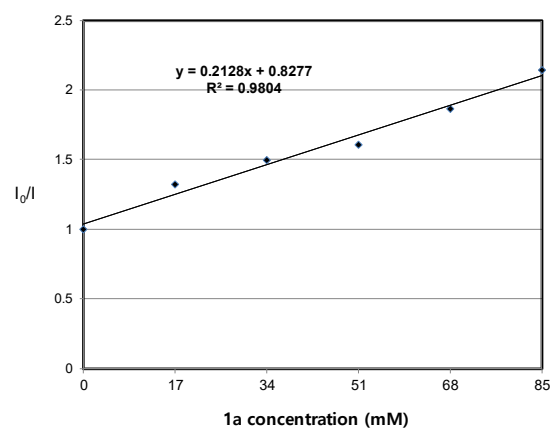
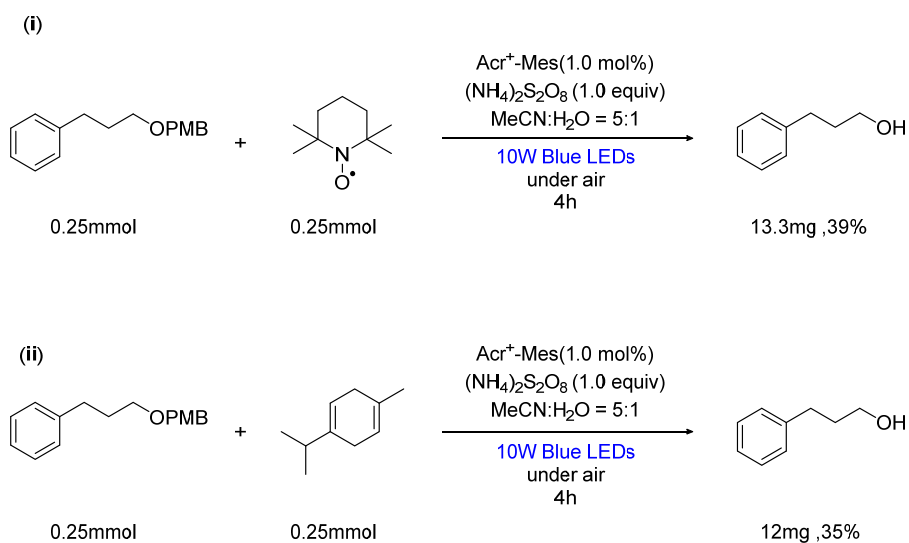


Figure S6. Stern-Volmer equation for **1a**

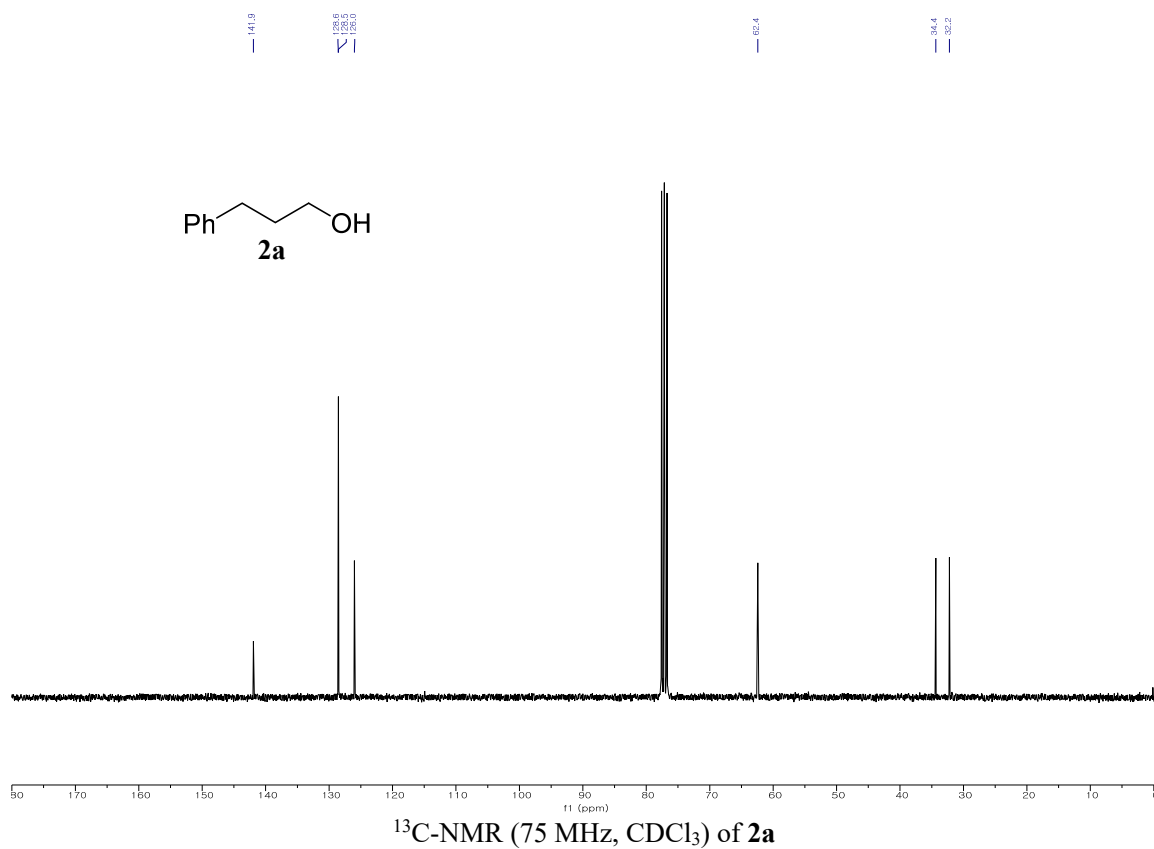
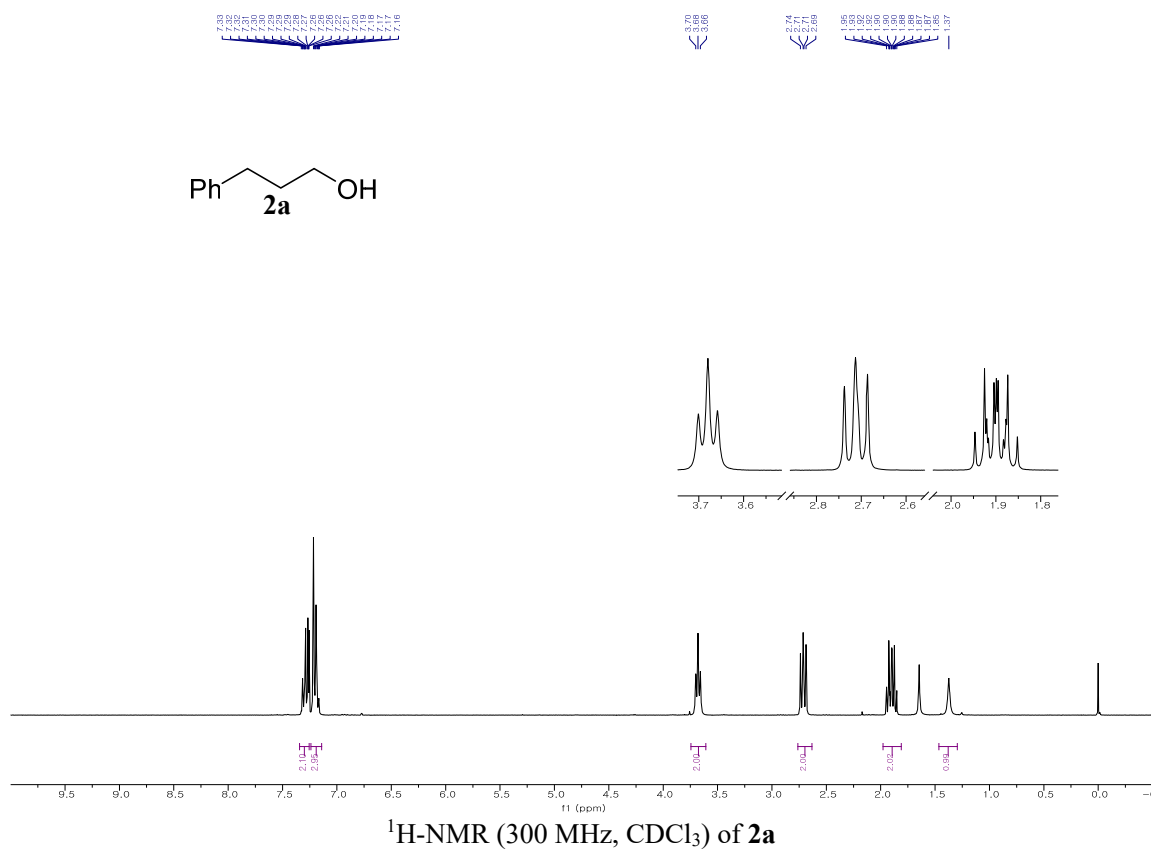
C. Radical trapping experiments

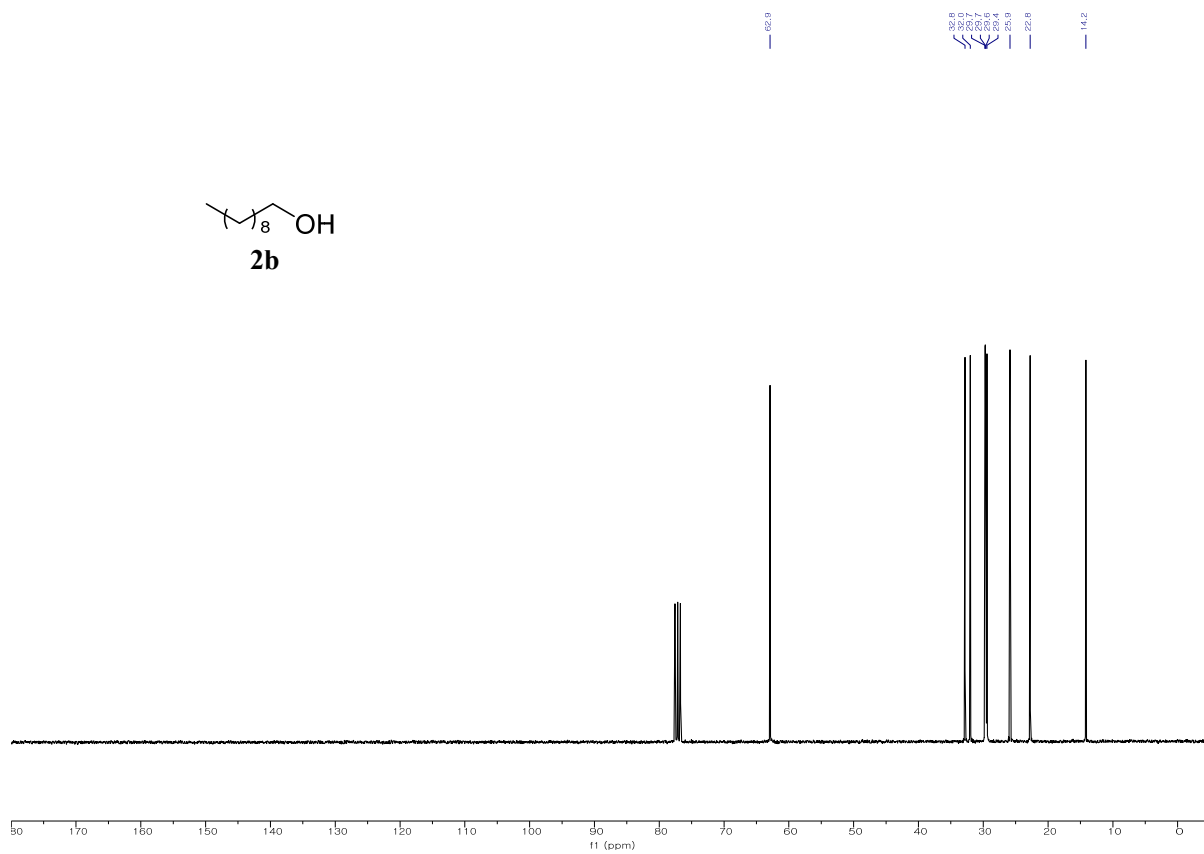
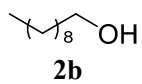
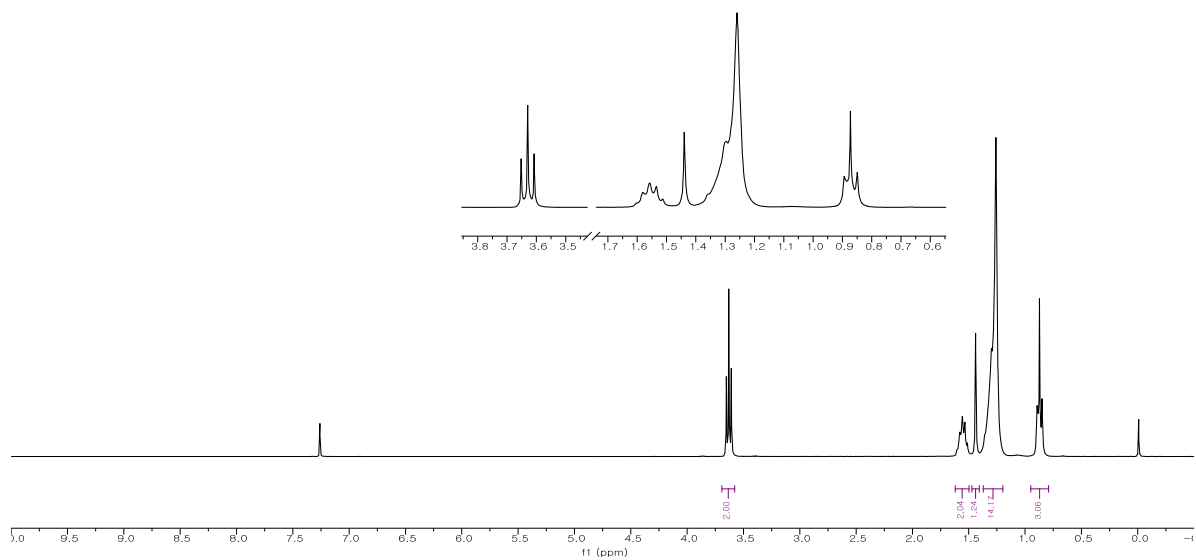
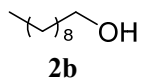
To get some mechanistic insight into the reaction mechanism, a radical trapping experiments were performed. The visible-light photoredox catalyzed oxidative cleavage of PMB ether was suppressed by the addition of 2,2,6,6-tetramethylpiperidine 1-oxyl (TEMPO) and γ -terpinene as a radical trapping reagent. This result indicated that the reaction involves a radical process (Scheme S1).

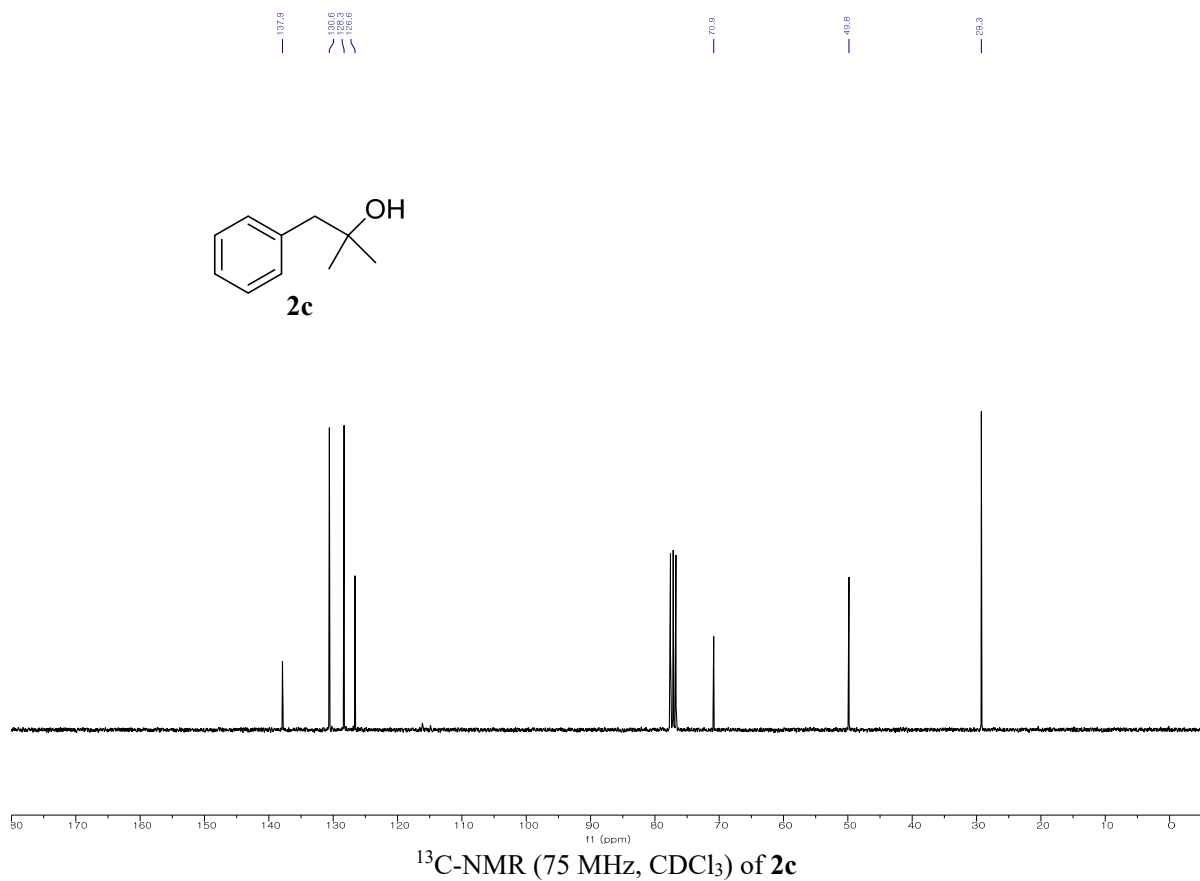
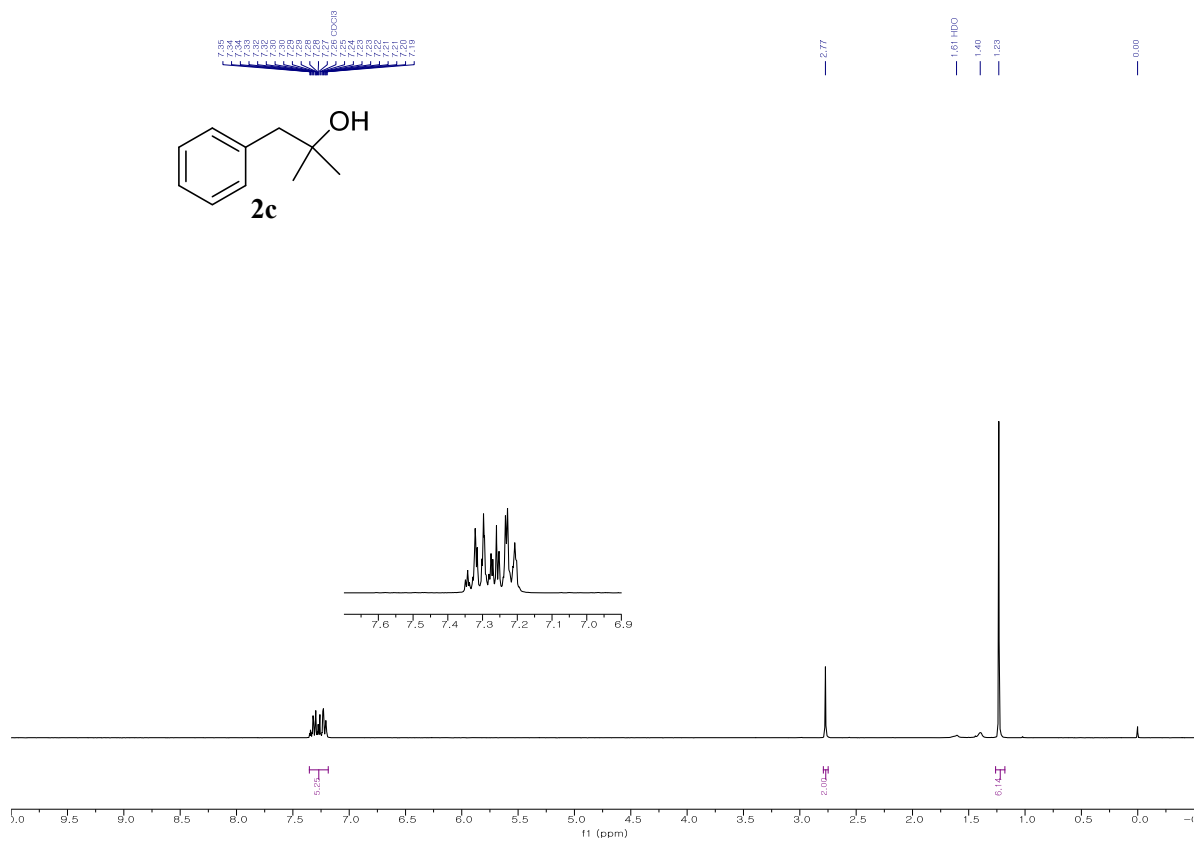


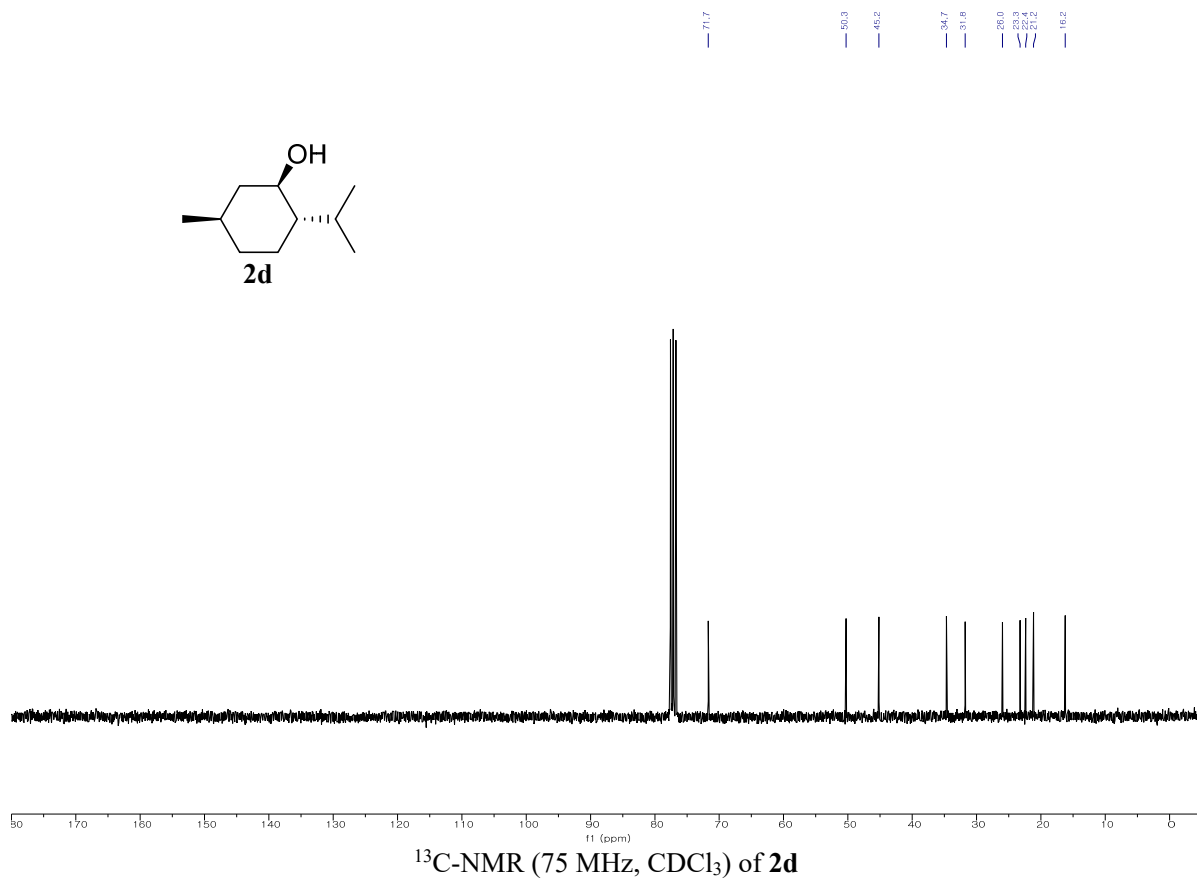
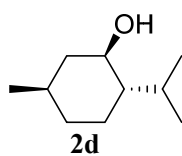
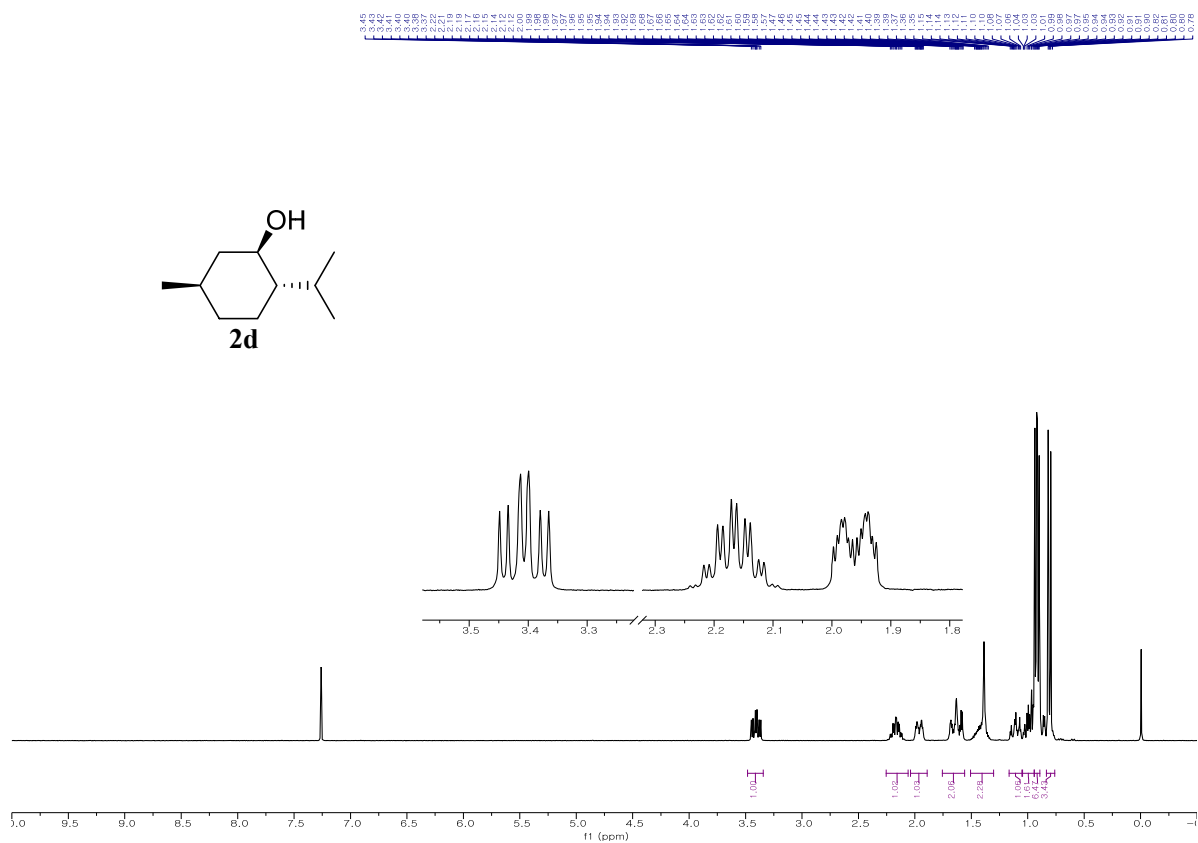
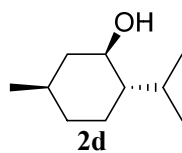
Scheme S1. Radical trapping experiments

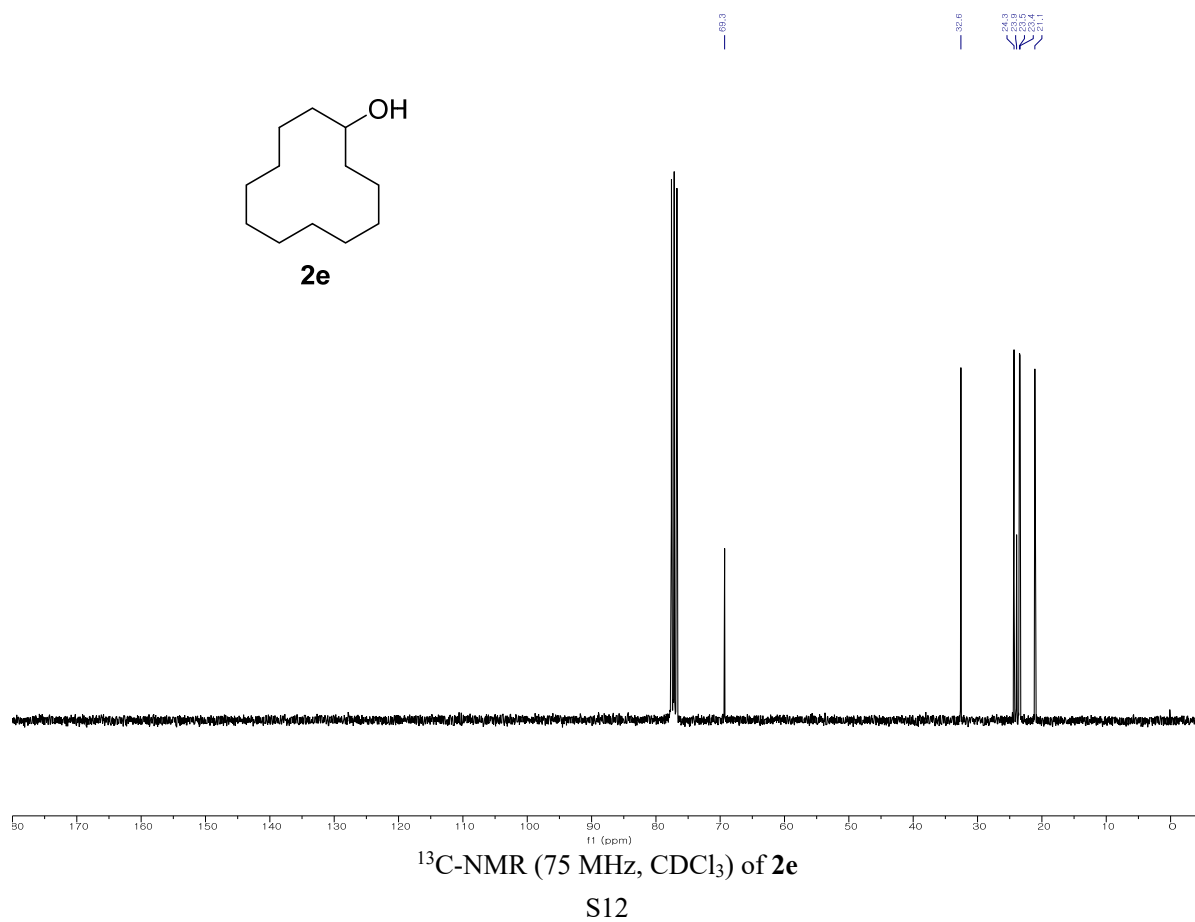
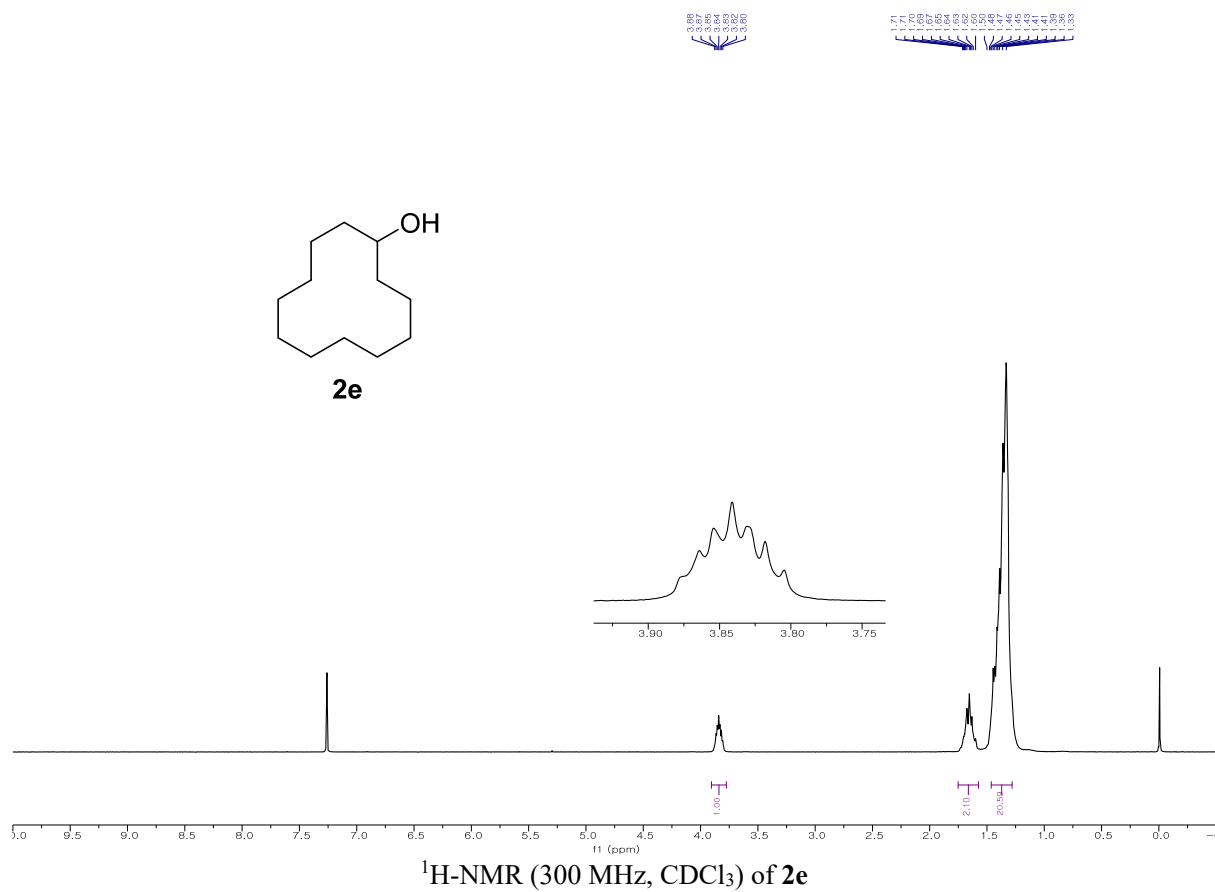
II. ^1H and ^{13}C NMR Spectra

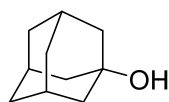




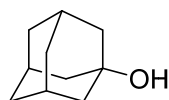








2f



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