

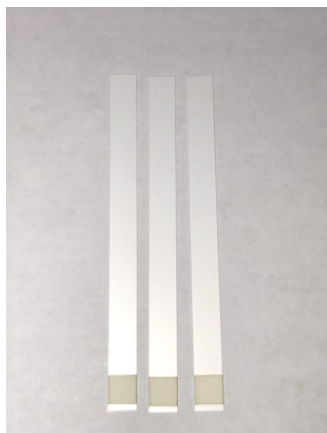
## Supporting Information

### **Mechanistic studies of visible light-induced CO release from a 3-hydroxybenzo[*g*]quinolone**

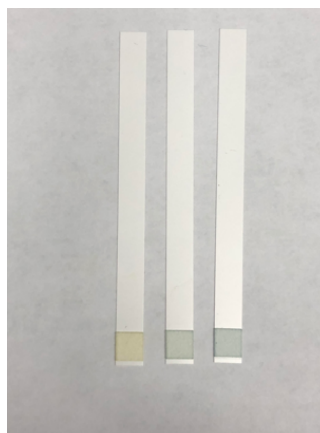
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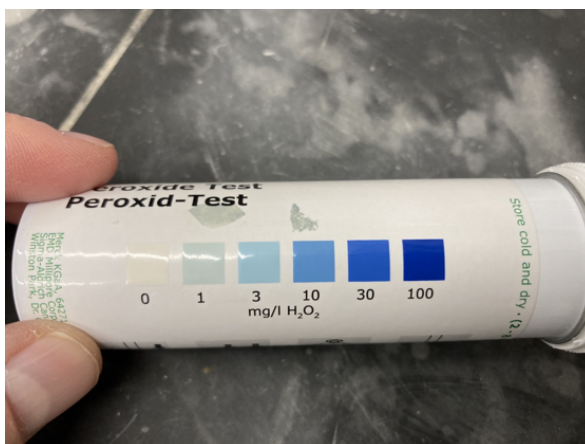
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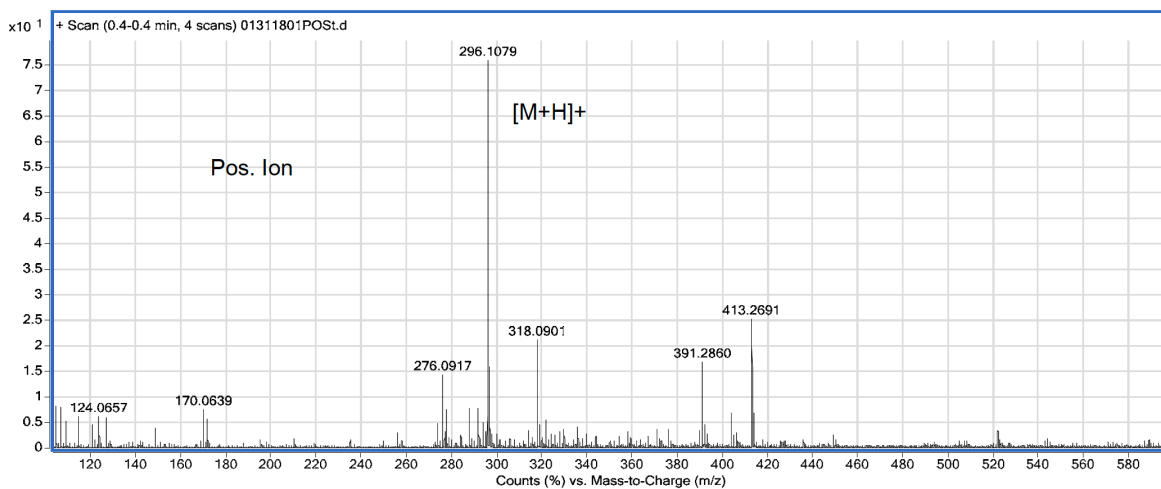
**4** +  $h\nu$  in  $\text{CH}_3\text{CN}$   
No change



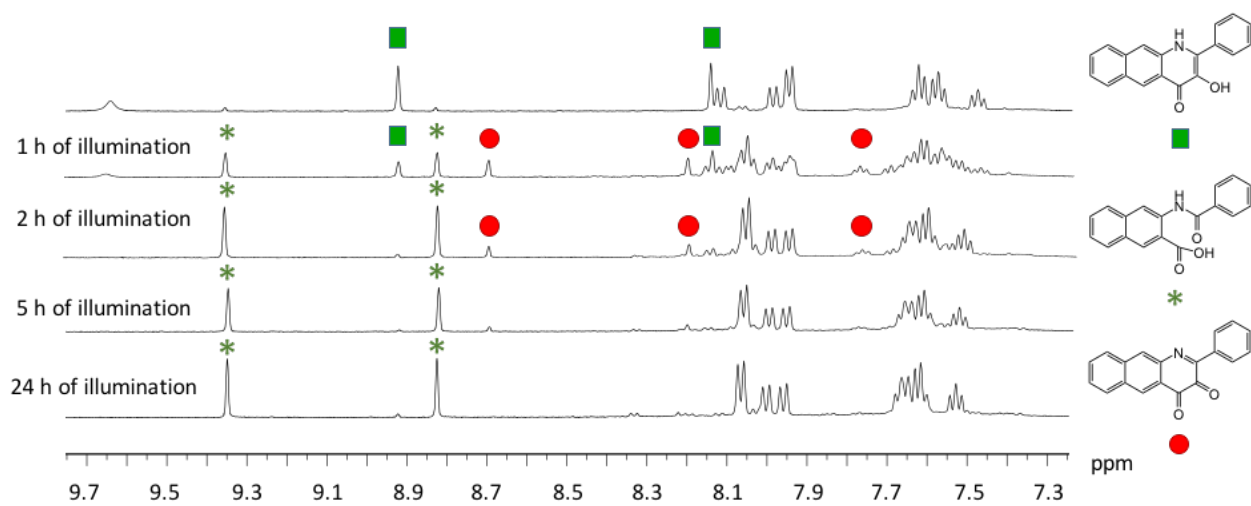
**5** +  $h\nu$  in  $\text{CH}_3\text{CN}$   
Increasing blue color



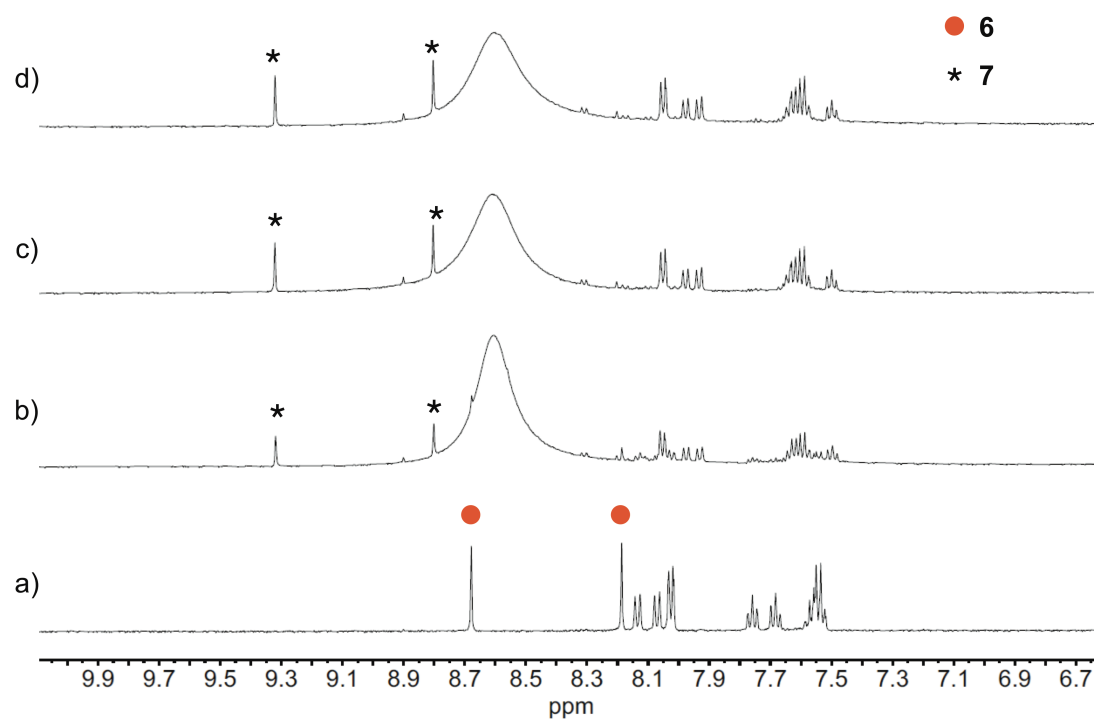
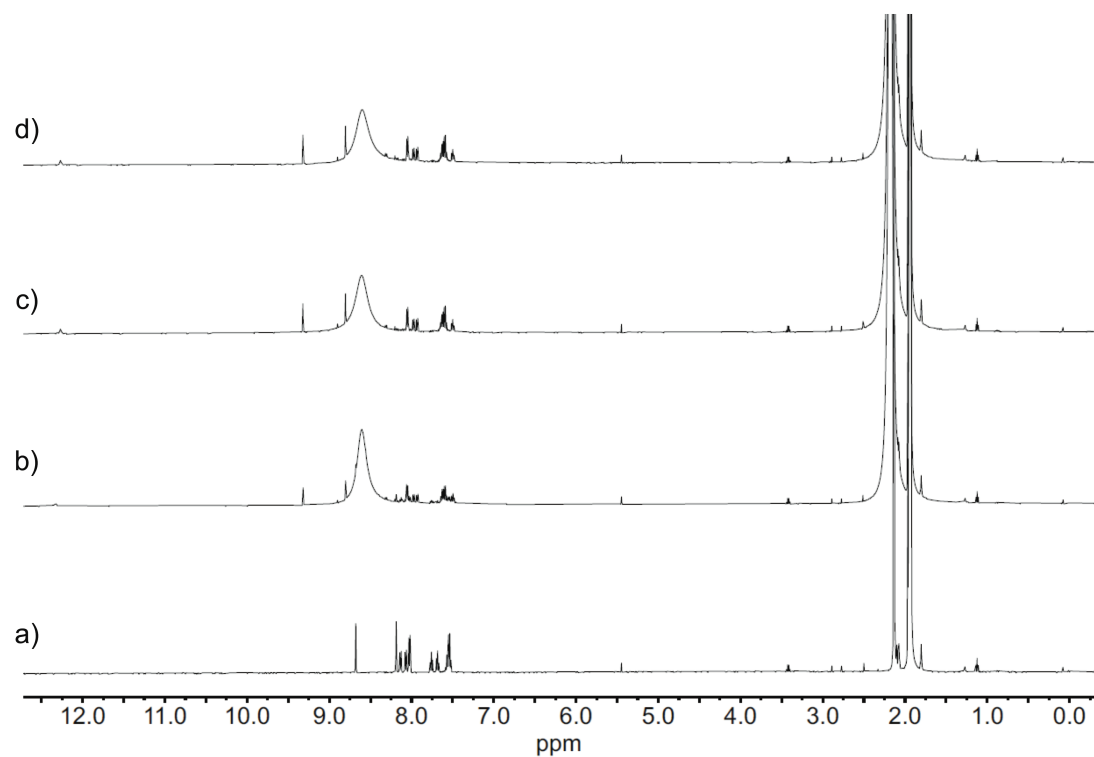
**Fig S1** (top) Photo of  $\text{H}_2\text{O}_2$  test strips which indicate formation of  $\text{H}_2\text{O}_2$  in the reaction mixtures of **4** and **5** ( $6.6 \times 10^{-4}$  M) in  $\text{CH}_3\text{CN}$  upon exposure to visible light (419 nm). For each set the left strip is the compound in  $\text{CH}_3\text{CN}$  prior to illumination. The center are right strips are following illumination for 12 and 24 min, respectively. The appearance of the blue color indicates  $\text{H}_2\text{O}_2$  formation in the reaction of **5**.



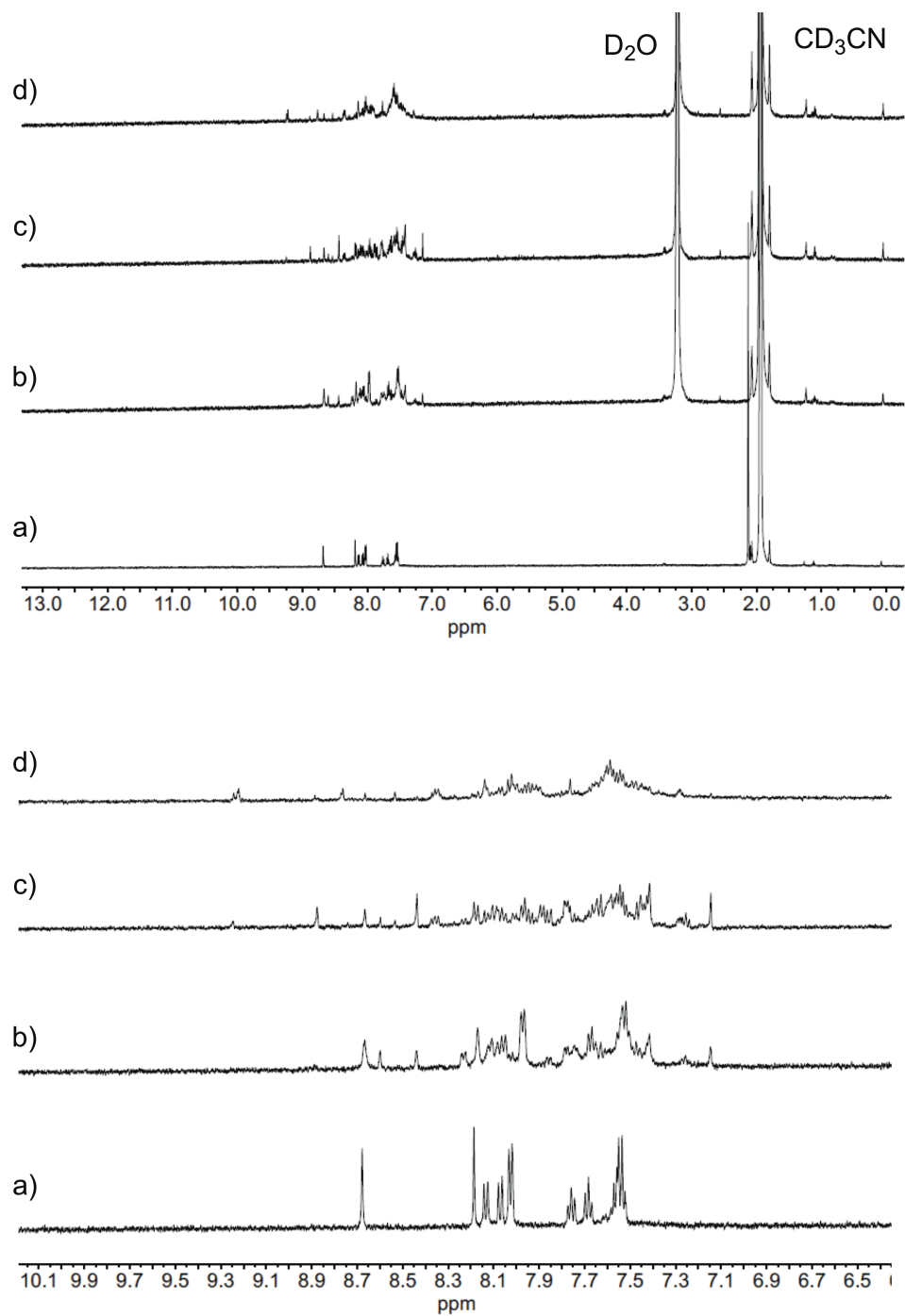
**Fig S2** ESI-MS of doubly <sup>18</sup>O labeled **7** ( $[M+H]^+ = m/z$  296) produced upon illumination of **5** with 419 nm light in the presence of <sup>18</sup>O<sub>2</sub>.



**Fig S3**  $^1\text{H}$  NMR features of a solution of **5** in wet  $\text{CD}_3\text{CN}$  illuminated (465 nm) at  $\sim 35^\circ\text{C}$  under  $\text{O}_2$ .



**Fig S4**  $^1\text{H}$  NMR spectra of **6** ( $9 \times 10^{-4}$  M) in  $\text{CD}_3\text{CN}$  upon addition of  $\text{H}_2\text{O}_2$  (10 eq, 30% solution in water) and illumination at 419 nm. (top) Full  $^1\text{H}$  NMR spectra; (bottom) features in the aromatic region of each spectrum. (a) **6** in  $\text{CD}_3\text{CN}$ ; (b) **6** +  $\text{H}_2\text{O}_2$ , 15 min illumination; (c) **6** +  $\text{H}_2\text{O}_2$ , 30 min illumination; (d) **6** +  $\text{H}_2\text{O}_2$ , 45 min illumination. The broad resonance centered at  $\sim 8.6$  ppm is due to the presence of  $\text{H}_2\text{O}_2$ .



**Fig S5** (Top) <sup>1</sup>H NMR spectra of **6** in CD<sub>3</sub>CN ( $7.7 \times 10^{-4}$  M (a)) and upon treatment with D<sub>2</sub>O (10 eq, (b)) at room temperature. Spectra (c) and (d) show changes that occur due to (c) the solution remaining at room temperature for 24 h under air, and (d) upon subsequent illumination of the solution for 15 min (419 nm)). (Bottom) Expanded views of the aromatic region of (a) – (d).