Supplementary Materials:

## **CF3-Bis-TEMPO-Vis: New Visible Light Active Bis-Benzimidazolequinone Alkoxyamine**

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## <sup>1</sup>H NMR (500 MHz) of **CF<sub>3</sub>-Bis-TEMPO-Vis** in CDCl<sub>3</sub>



<sup>13</sup>C NMR (125 MHz) of CF<sub>3</sub>-Bis-TEMPO-Vis in CDCl<sub>3</sub>



## <sup>19</sup>F NMR (470 MHz) of **CF<sub>3</sub>-Bis-TEMPO-Vis** in CDCl<sub>3</sub>



30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -2(

**Figure S1.** TEMPO release (mol%) versus time (min) from **CF<sub>3</sub>-Bis-TEMPO-Vis**, **1** and **Bis-TEMPO-Vis** at room temperature using green-LED (2 X 9 W bulbs) with [Bis-alkoxyamine]<sub>0</sub> = 5 mM (corresponding to Figure 3(A)).



**Figure S2.** TEMPO release (mol%) versus time (min) from **CF**<sub>3</sub>-**Bis-TEMPO-Vis**, **1** and **Bis-TEMPO-Vis** at room temperature using green-LED (2 X 9 W bulbs) with [Bis-alkoxyamine]<sub>0</sub> = 0.25 mM (corresponding to Figure 3(B)).



One extra data point measured for Bis-TEMPO-Vis at 25 min to demonstrate tendency towards the plateau. This extra data point measured once, whereas all other points measured in triplicate.

Figure S3. Photograph of the photoreactor.

