## SUPPLEMENTARY MATERIAL

## Simultaneous Observation of Triplet and Singlet Cyclopentane-1,3-diyl Diradicals in the Intersystem Crossing Process

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## <sup>1</sup>H and <sup>13</sup>C NMR spectra

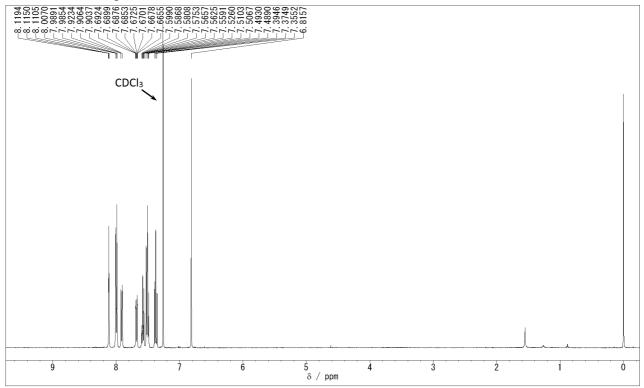


Figure S1. <sup>1</sup>H NMR spectrum of compound **3** (400 MHz, CDCl<sub>3</sub>)

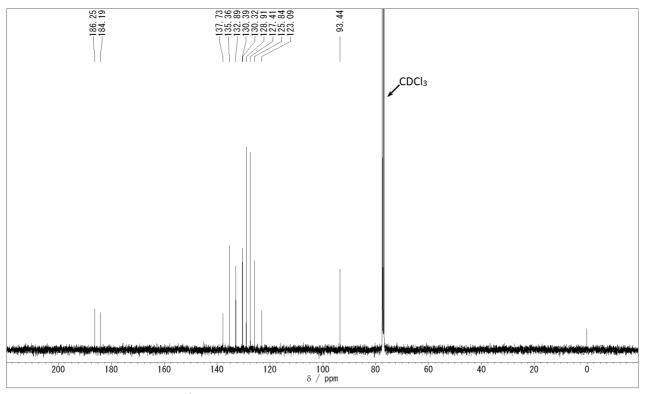


Figure S2. <sup>13</sup>C NMR spectrum of compound **3** (100 MHz, CDCl<sub>3</sub>)

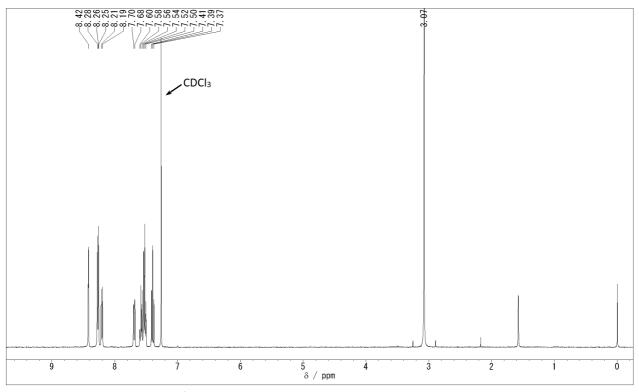


Figure S3. <sup>1</sup>H NMR spectrum of compound **5** (400 MHz, CDCl<sub>3</sub>)

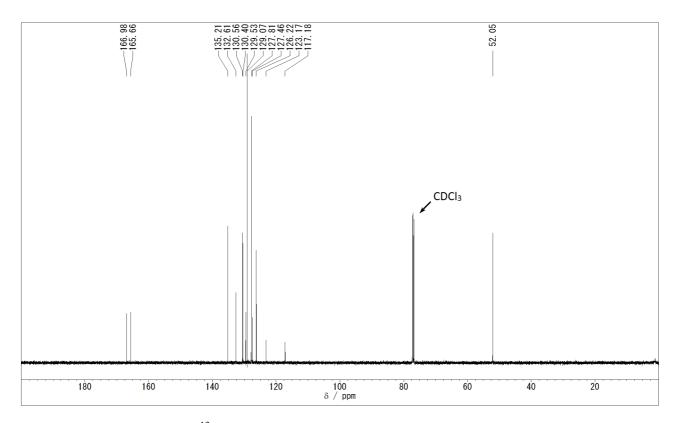


Figure S4. <sup>13</sup>C NMR spectrum of compound **5** (100 MHz, CDCl<sub>3</sub>)

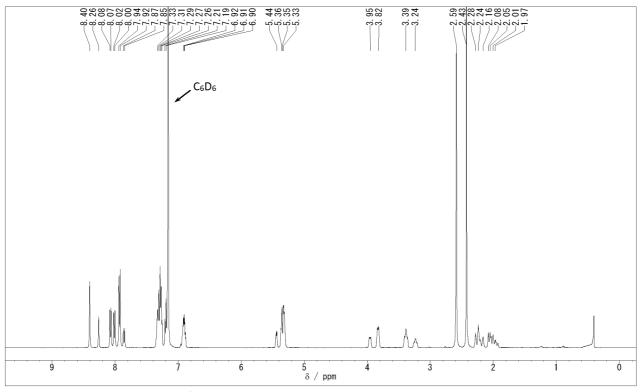


Figure S5. <sup>1</sup>H NMR spectrum of compound **6** (400 MHz, C<sub>6</sub>D<sub>6</sub>)

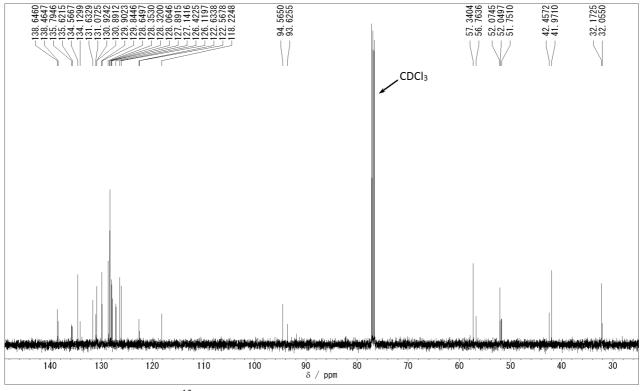


Figure S6. <sup>13</sup>C NMR spectrum of compound **6** (100 MHz, CDCl<sub>3</sub>)

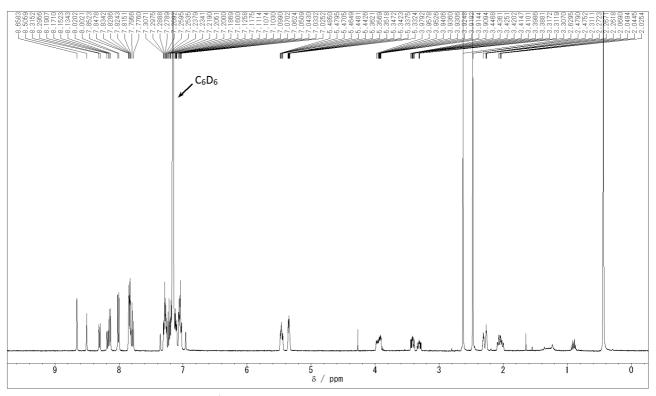


Figure S7. <sup>1</sup>H NMR spectrum of **AZ3'** (400 MHz, C<sub>6</sub>D<sub>6</sub>)

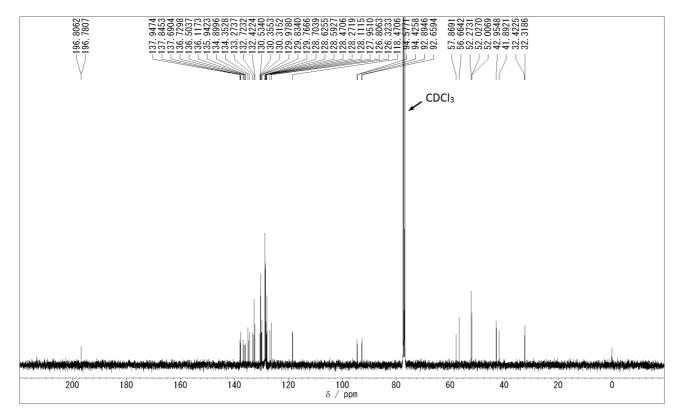


Figure S8. <sup>13</sup>C NMR spectrum of **AZ3'** (100 MHz, CDCl<sub>3</sub>)

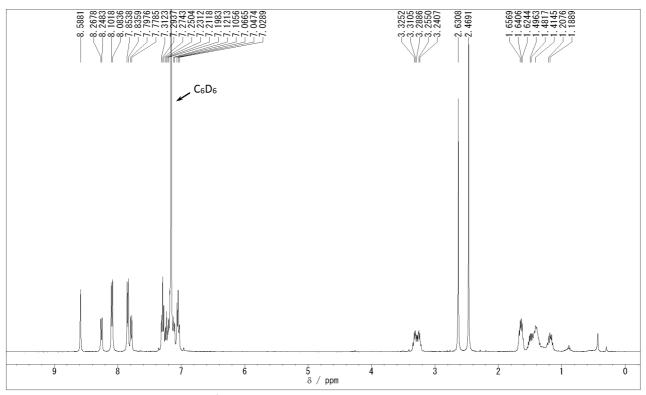


Figure S9. <sup>1</sup>H NMR spectrum of **AZ3** (400 MHz, C<sub>6</sub>D<sub>6</sub>)

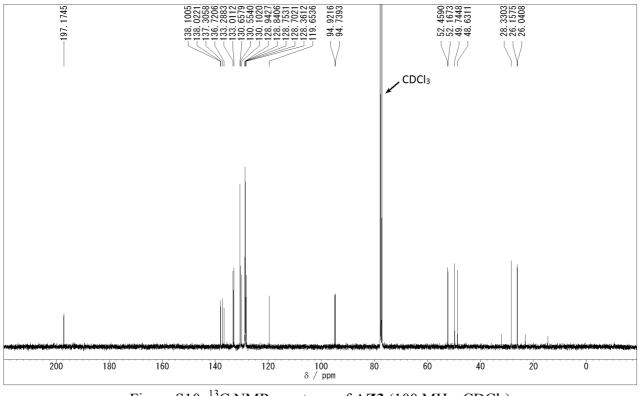


Figure S10. <sup>13</sup>C NMR spectrum of AZ3 (100 MHz, CDCl<sub>3</sub>)

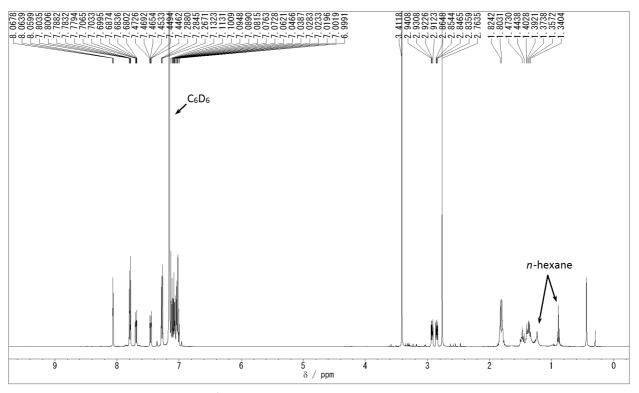


Figure S11. <sup>1</sup>H NMR spectrum of **CP3** (400 MHz,  $C_6D_6$ )

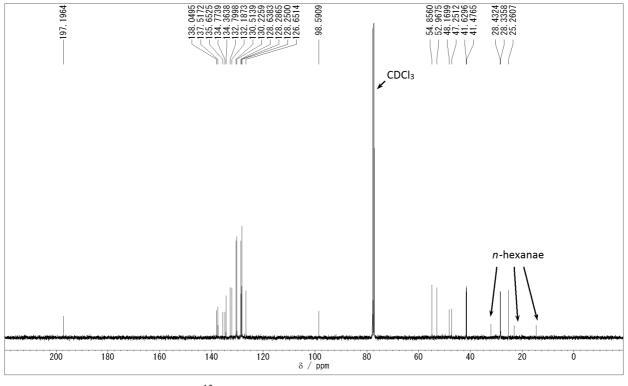
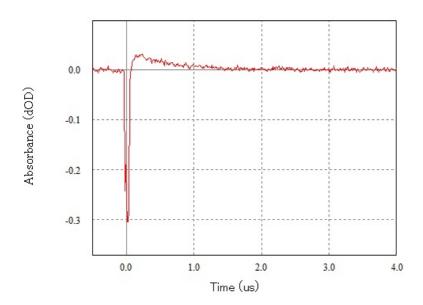


Figure S12. <sup>13</sup>C NMR spectrum of CP3 (100 MHz, CDCl<sub>3</sub>)

Time Profile at 420 nm in Figure 2a.



Time profile at 420 nm in Figure 3a

