

<Electronic Supplementary Information (ESI) available>

**Stable suprachannels via columnar cyclodimeric ensemble:  
exchange and matrix of various liquid guests in SCSC fashion**

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**Experimental**

**Materials and measurements.** All chemicals including Zinc(II) iodide, isophthaloyl chloride and 5-hydroxyisoquinoline were purchased from Sigma-Aldrich and used without further purification.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Varian Mercury Plus 300 operating at 300.00 MHz and 75.00 MHz, respectively, and the chemical shifts were related to the internal  $\text{Me}_4\text{Si}$ . Infrared spectra were obtained on a Nicolet 380 FT-IR spectrophotometer, samples having been prepared as KBr pellets. Thermal analyses were carried out under a dinitrogen atmosphere at a scan rate of 10 °C/min using a Labsys TGA-DSC 1600. The absorption spectra were recorded on an S-3150 UV-vis spectrophotometer.

**Synthesis of di(isoquinolin-5-yl)isophthalate (L).** Triethylamine (25.0 mmol, 3.48 ml) was added to a stirred mixture of 5-hydroxyisoquinoline (10.3 mmol, 1.48 g) and isophthaloyl chloride (5.0 mmol, 1.01 g) in tetrahydrofuran (50 mL) at room temperature. The reaction mixture was stirred overnight. The solution was filtered, and then the filtrate was washed several times with 0.5 N  $\text{NaHCO}_3$  aqueous solution. The chloroform layer was dried using sodium sulfate and filtered. Evaporation of the chloroform yielded a pale yellow

product. The crude product was then purified by column chromatography using chloroform and methanol as the eluent. The product finally was purified in a solvent pair of chloroform and *n*-hexane, affording di(isoquinolin-5-yl)isophthalate (L) as white solids in 91.5% yields. Anal. Calcd for C<sub>26</sub>H<sub>16</sub>N<sub>2</sub>O<sub>4</sub>: C, 74.28; H, 3.84; N, 6.66%. Found: C, 74.10; H, 3.81; N, 6.58%. IR (KBr, cm<sup>-1</sup>): 1731 (s, C=O), 1589(m), 1492(w), 1376(m), 1303 (s), 1272(s), 1222 (s), 1211(s), 1160(s), 1095(s), 1060(m), 998(w), 829(m), 759(m), 721 (m), 640 (w), 609(w). <sup>1</sup>H NMR (Me<sub>2</sub>SO-*d*<sub>6</sub>, δ): 9.46 (s, 2H), 9.02 (s, 1H), 8.68 (d, *J* = 7.63 Hz, 2H), 8.58 (d, *J* = 7.04 Hz, 1H), 8.17 (d, *J* = 8.22 Hz, 2H), 8.00 (t, *J* = 15.85 Hz, 1H), 7.84 (d, *J* = 7.63 Hz, 2H), 7.81(m, *J* = 15.85 Hz, 4H). <sup>13</sup>C NMR (Me<sub>2</sub>SO-*d*<sub>6</sub>, δ): 164.23, 152.96, 145.62, 144.14, 135.88, 127.95, 123.61, 114.42, 110.00, 39.99.

**3.5CH<sub>3</sub>CN·0.7THF@[Zn<sub>2</sub>I<sub>4</sub>L<sub>2</sub>].** An acetonitrile solution (2 mL) of ZnI<sub>2</sub> (3.2 mg, 0.01 mmol) was slowly diffused into a tetrahydrofuran solution (2 mL) of L (4.2 mg, 0.01 mmol). Colorless crystals of 3.5CH<sub>3</sub>CN·0.7THF@[Zn<sub>2</sub>I<sub>4</sub>L<sub>2</sub>] formed at the interface and were obtained in 3 days in a 67% yield. m.p. 344.6 °C (dec.). Anal. Calcd C<sub>61.8</sub>H<sub>48.1</sub>I<sub>4</sub>N<sub>7.5</sub>O<sub>8.7</sub>Zn<sub>2</sub>: C, 44.50; H, 2.89; N, 5.86%. Found: C, 43.94; H, 2.76; N, 5.49%. IR (KBr, cm<sup>-1</sup>): 1751(s, C=O), 850 (s), 1203(s), 1095(m), 1033 (m), 721 (m). <sup>1</sup>H NMR (Me<sub>2</sub>SO-*d*<sub>6</sub>, δ, dissociated): 9.46 (s, 2H), 9.02(s, 2H), 8.68(d, *J* = 8.22 Hz, 2H), 8.58 (d, *J* = 5.87 Hz, 2H), 8.17 (d, *J* = 8.22 Hz, 2H), 8.00 (t, *J* = 15.85 Hz, 2H), 7.84 (d, *J* = 7.63 Hz, 2H), 7.81(m, *J* = 15.85 Hz, 4H).

**SCSC solvate exchange.** Single crystals of 3.5CH<sub>3</sub>CN·0.7THF@[Zn<sub>2</sub>I<sub>4</sub>L<sub>2</sub>] were left a mixture of CH<sub>2</sub>I<sub>2</sub> and benzene (*v/v* = 1 : 1) media at room temperature for 12 h, resulting in the transformation of single crystals of 2CH<sub>2</sub>I<sub>2</sub>·C<sub>6</sub>H<sub>6</sub>@[Zn<sub>2</sub>I<sub>4</sub>L<sub>2</sub>]. m.p. 326.2 °C (dec.). Anal. Calcd for C<sub>60</sub>H<sub>42</sub>I<sub>8</sub>N<sub>4</sub>O<sub>8</sub>Zn<sub>2</sub>: C, 34.43; H, 2.02; N, 2.68 %. Found: C, 34.20; H, 2.00; N, 2.55%. Single crystals of 3.5CH<sub>3</sub>CN·0.7THF@[Zn<sub>2</sub>I<sub>4</sub>L<sub>2</sub>] were left in phenyl acetylene media

at 25 °C for 12 h, resulting in the transformation of single crystals of  $2.2\text{C}_8\text{H}_6@[\text{Zn}_2\text{I}_4\text{L}_2]$  suitable for single-crystal X-ray diffraction. m.p. 356.3 °C (dec.). Anal. Calcd for  $\text{C}_{69.6}\text{H}_{45.2}\text{I}_4\text{N}_4\text{O}_8\text{Zn}_2$ : C, 49.86 H, 2.73; N, 3.23%. Found: C, 48.23; H, 2.56; N, 3.12%. The other (guest) $@1$  species, namely  $\text{C}_6\text{H}_6@[\text{Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_7\text{H}_8@[\text{Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_6\text{H}_8@[\text{Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_8\text{H}_{18}\text{O}@[\text{Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_{10}\text{H}_{20}@[\text{Zn}_2\text{I}_4\text{L}_2]$ , and  $\text{C}_{12}\text{H}_{22}@[\text{Zn}_2\text{I}_4\text{L}_2]$ , were obtained in benzene, toluene, 1,4-cyclohexadiene, 1-octanol, 1-decene, and 1-dodecyne, respectively.

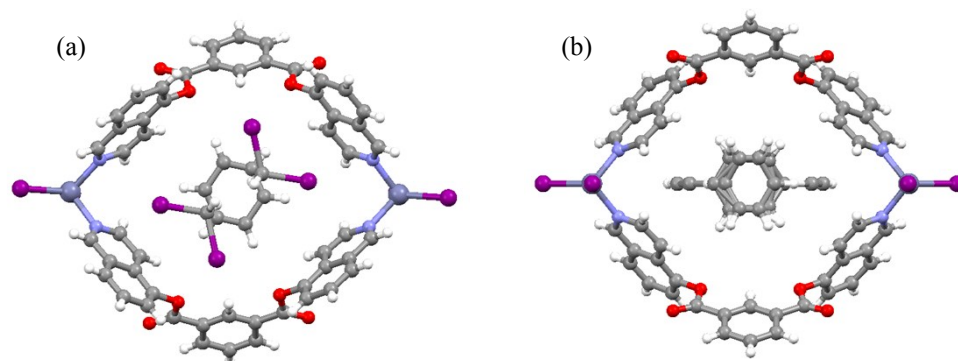
All guest solvents are reversible in excess liquid state (Fig. S5).

**Crystal-structure determination.** X-ray data were collected on a Bruker SMART automatic diffractometer with graphite-monochromated Mo  $K\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ). Thirty-six (36) frames of 2D diffraction images were collected and processed to obtain the cell parameters and orientation matrix. The data were corrected for Lorentz and polarization effects. The absorption effects were corrected using the multi-scan method (SADABS).<sup>26</sup> The diffraction data of the single crystal were measured at 120 K, respectively, with synchrotron radiation ( $\lambda = 0.6300$  and  $0.6500 \text{ \AA}$ , respectively) on a Rayonix MX225HS detector at 2D SMC with a silicon (111) double crystal monochromator (DCM) at the Pohang Accelerator Laboratory, Korea. The PAL BL2D-SMDC program<sup>27</sup> was used for data collection (detector distance was 66 mm, omega scan;  $\Delta\omega = 1^\circ$ , exposure time was 1 s per frame) and HKL3000sm (ver. 703r)<sup>28</sup> was used for cell refinement, reduction, and absorption correction. The structures were solved using the direct method (SHELXS) and refined by full-matrix least squares techniques (SHELXL 2018/3).<sup>29</sup> The non-hydrogen atoms were refined anisotropically, and the hydrogen atoms were placed in calculated positions and refined only for the isotropic thermal factors. The crystal parameters and procedural information corresponding to the data collection and structure refinement are listed in Table 1.

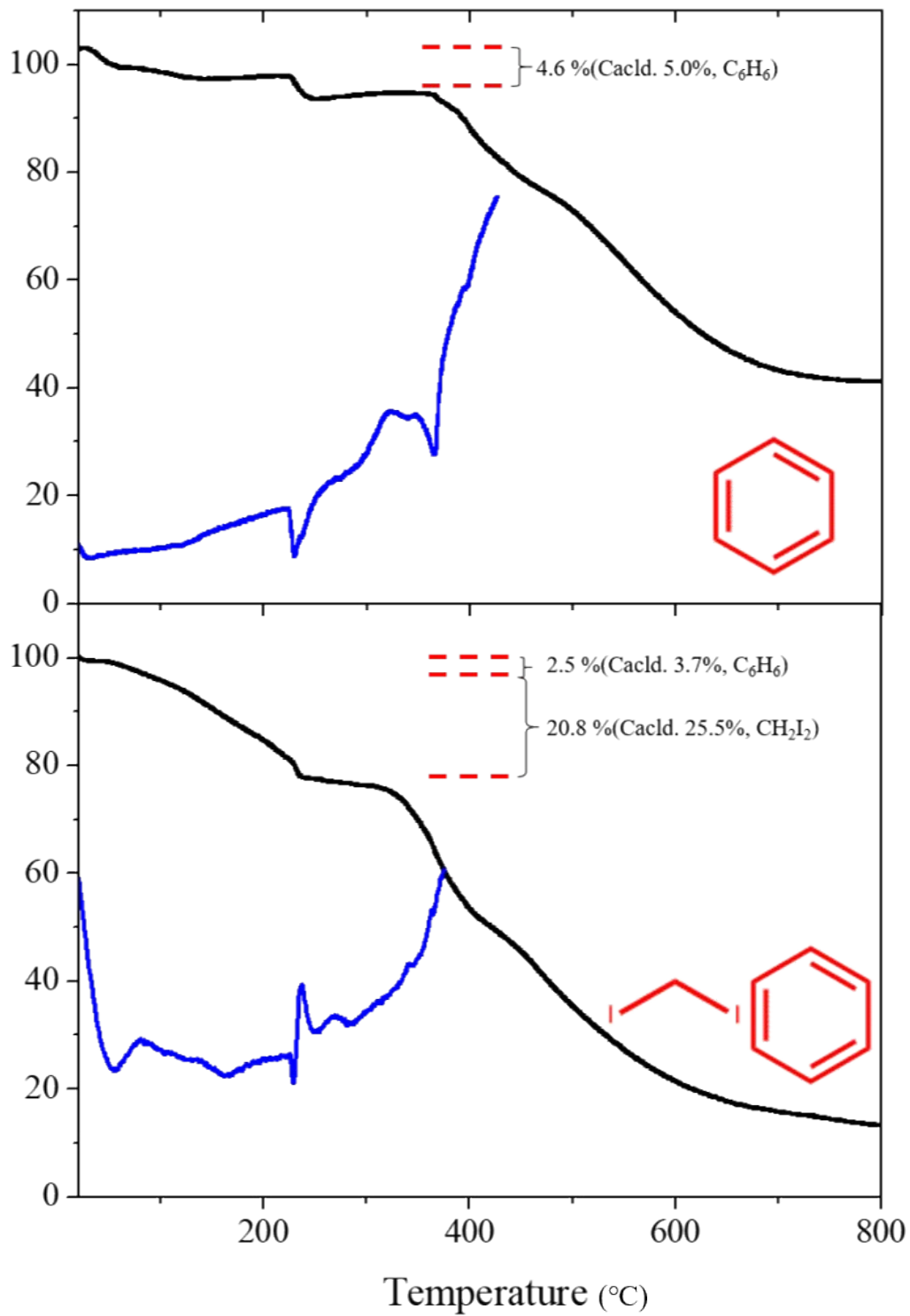
## Reference

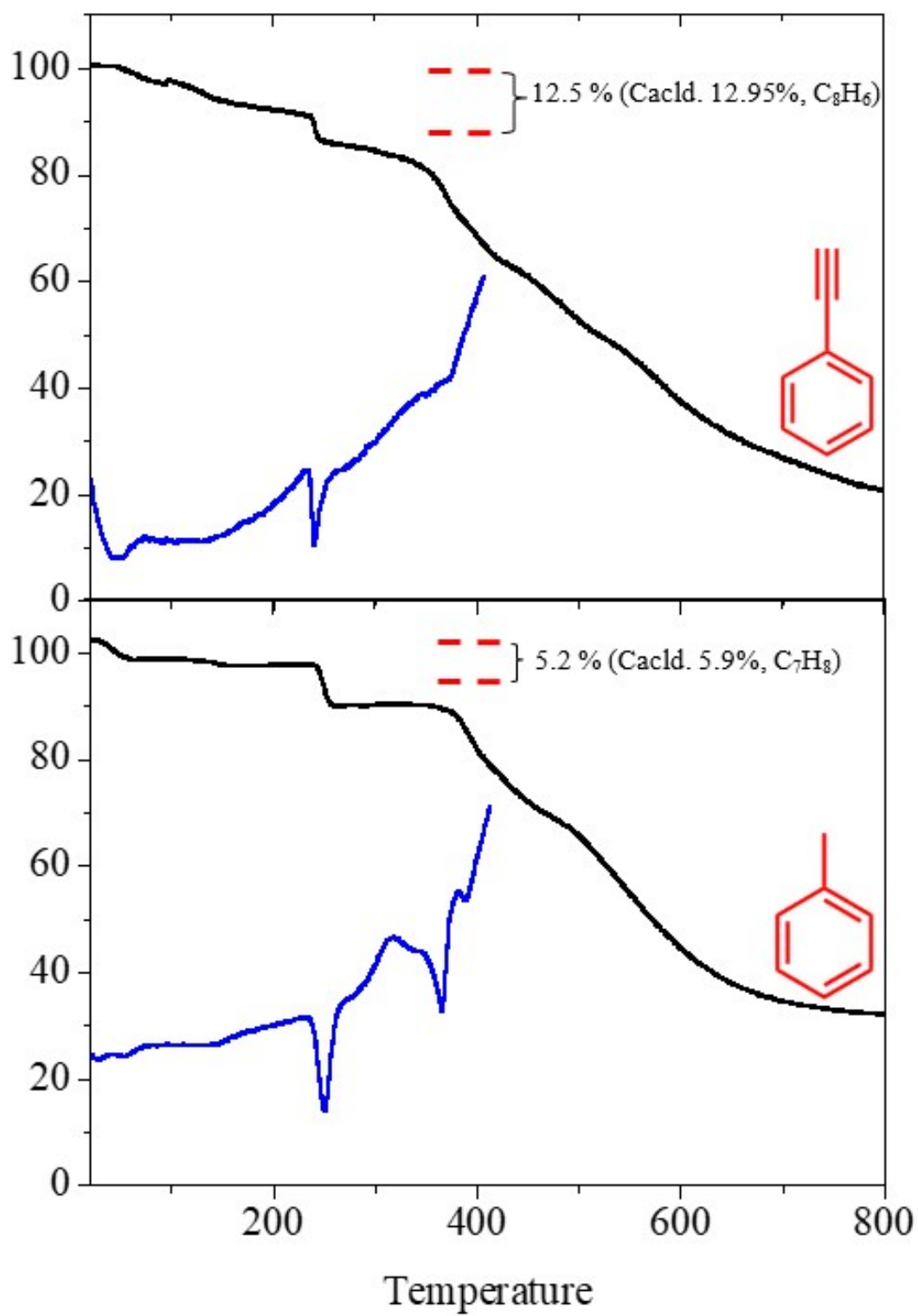
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27. J. W. Shin, K. Eom and D. Moon, *J. Synchrotron Radiat.* 2016, **23**, 369–373.
28. Z. Otwinowski and W. Minor, *Methods in Enzymology*, ed. C. W. Carter Jr. and R. M. Sweet, Academic Press, New York, 1997, **276(part A)**, 307.
29. Sheldrick, G. M. *Acta Crystallogr. Sect. C-Struct. Chem.* 2015, **71**, 3-8.

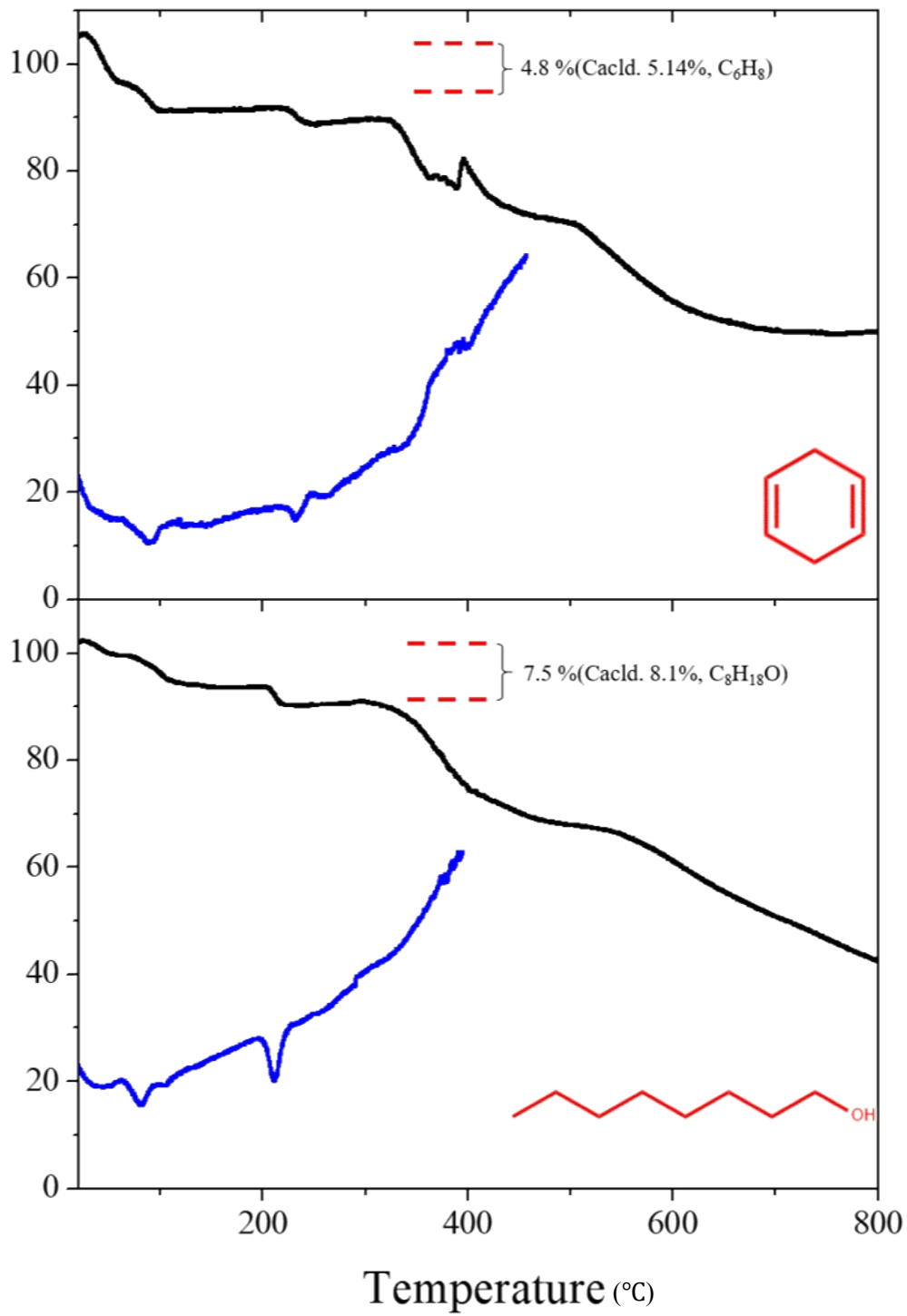
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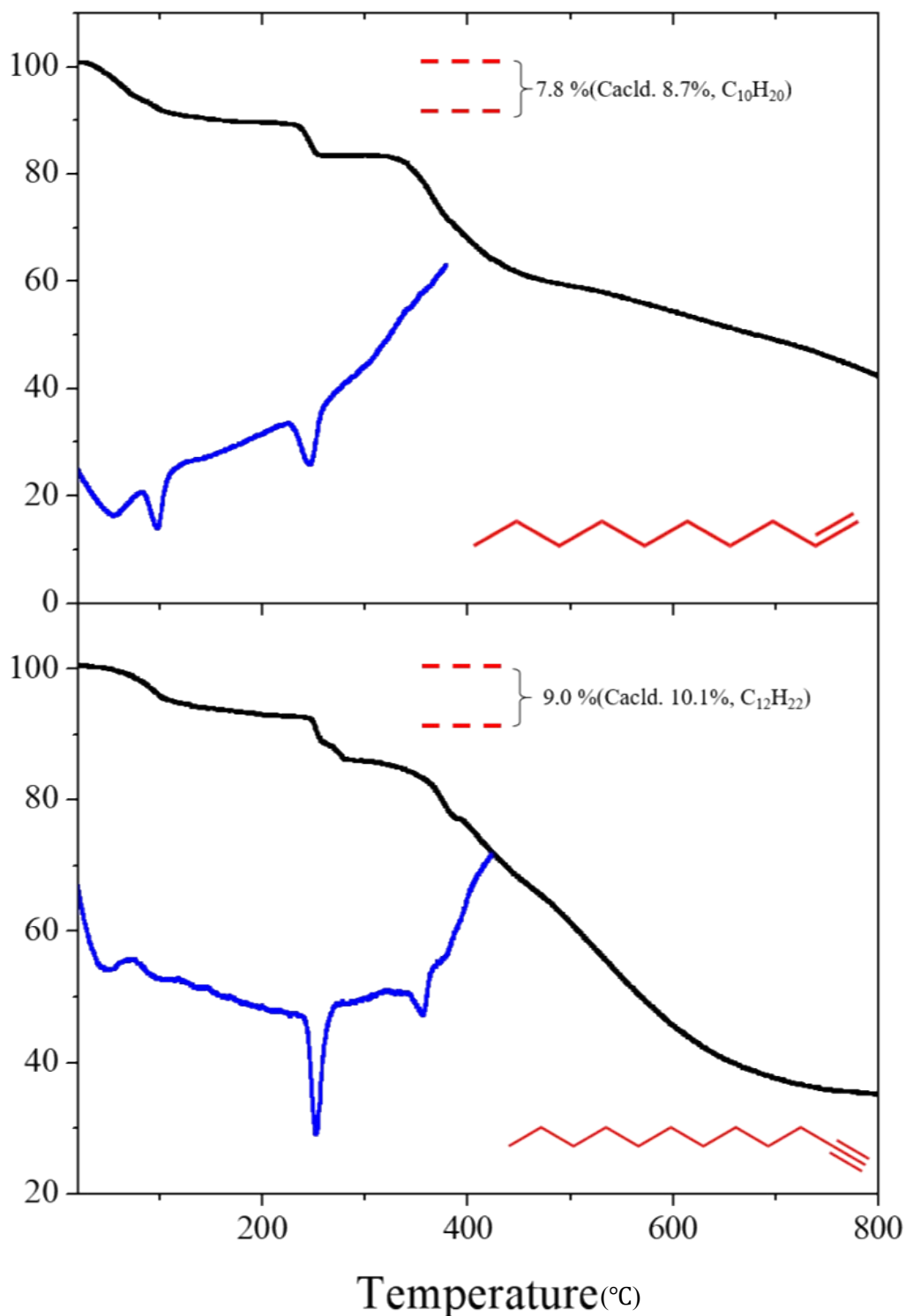
**Fig. S1.** X-ray structures of solvate exchanged species  $2\text{CH}_2\text{I}_2 \cdot \text{C}_6\text{H}_6 @ [\text{Zn}_2\text{I}_4\text{L}_2]$  and  $2.2\text{C}_8\text{H}_6 @ [\text{Zn}_2\text{I}_4\text{L}_2]$ .





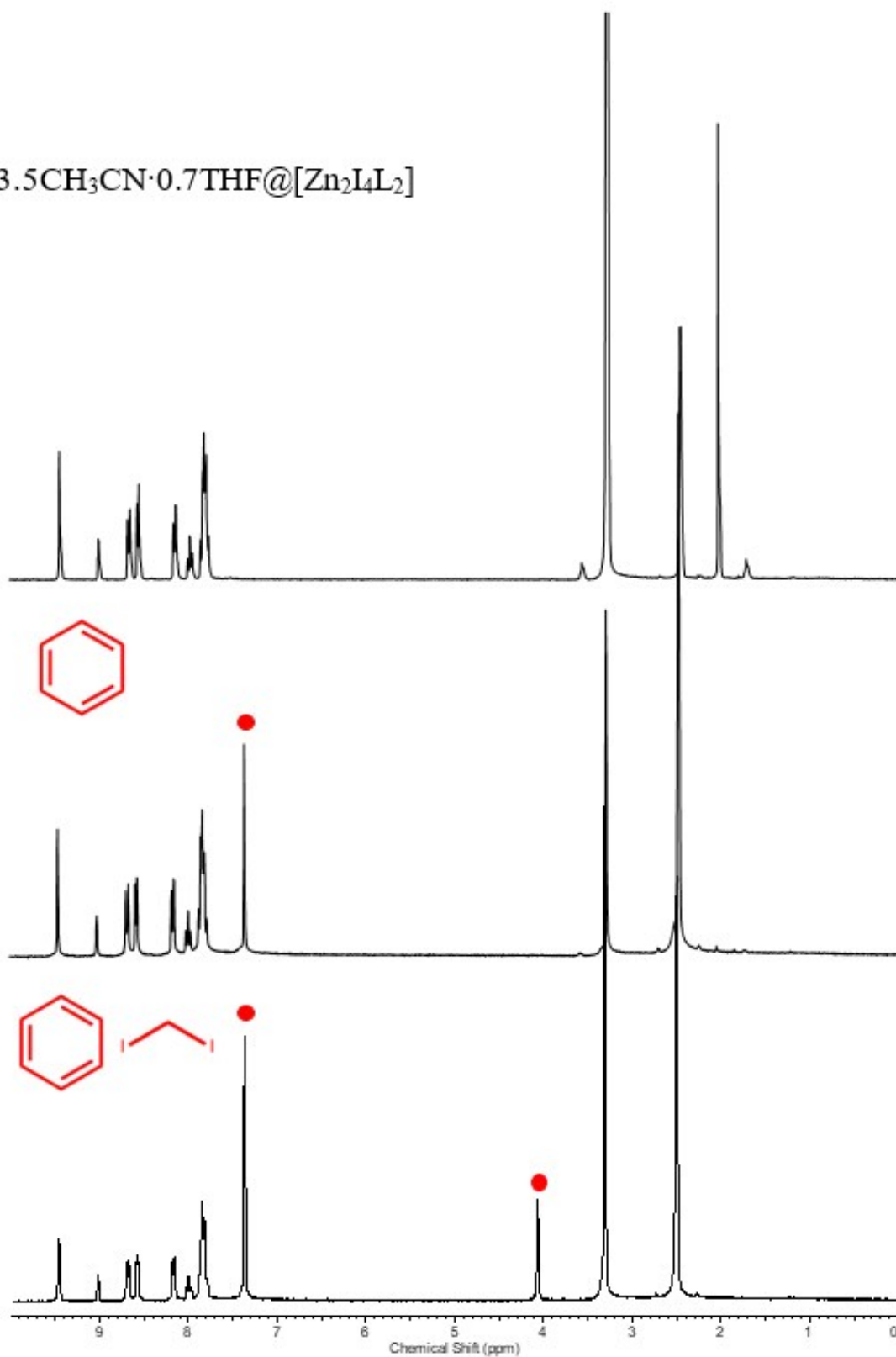


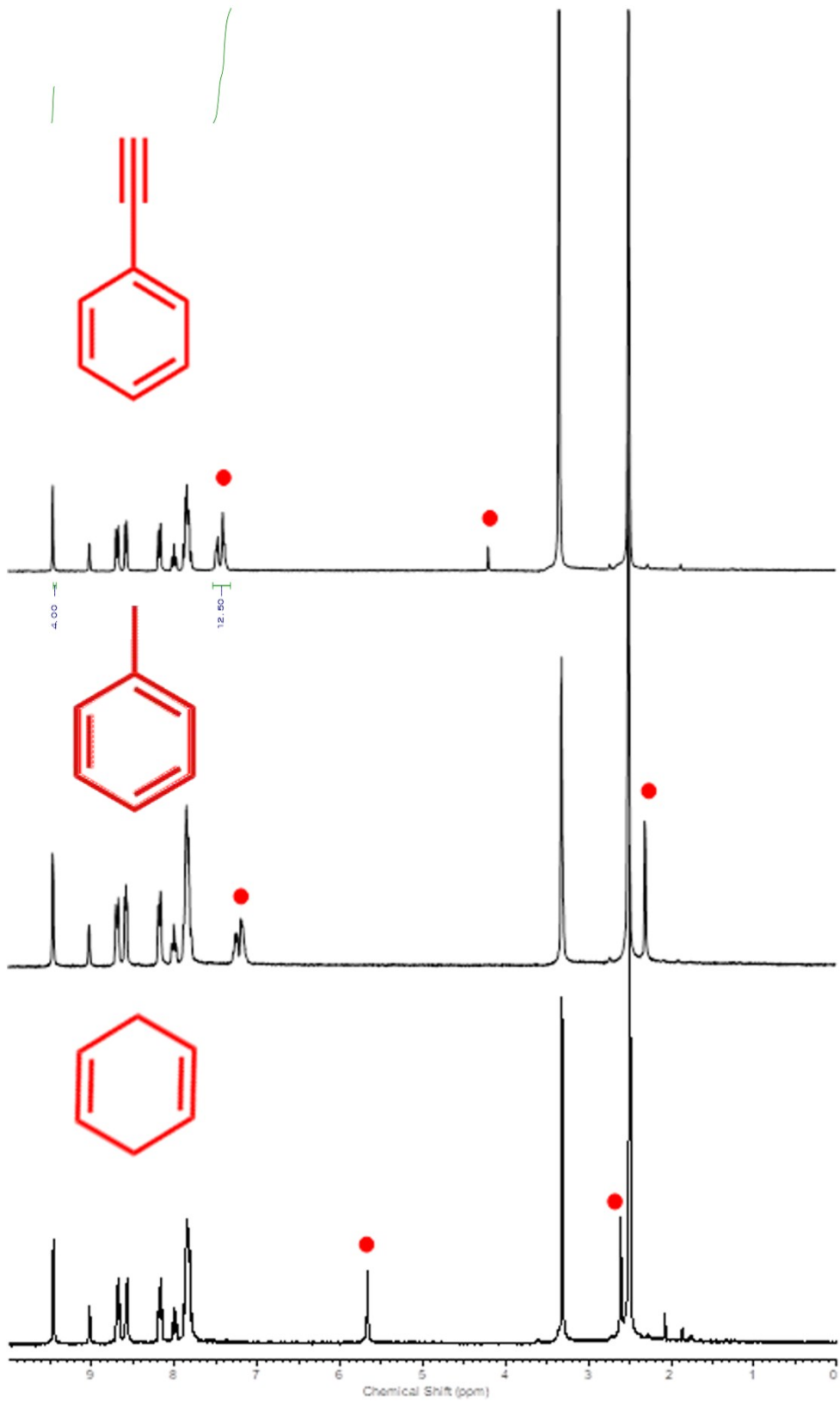


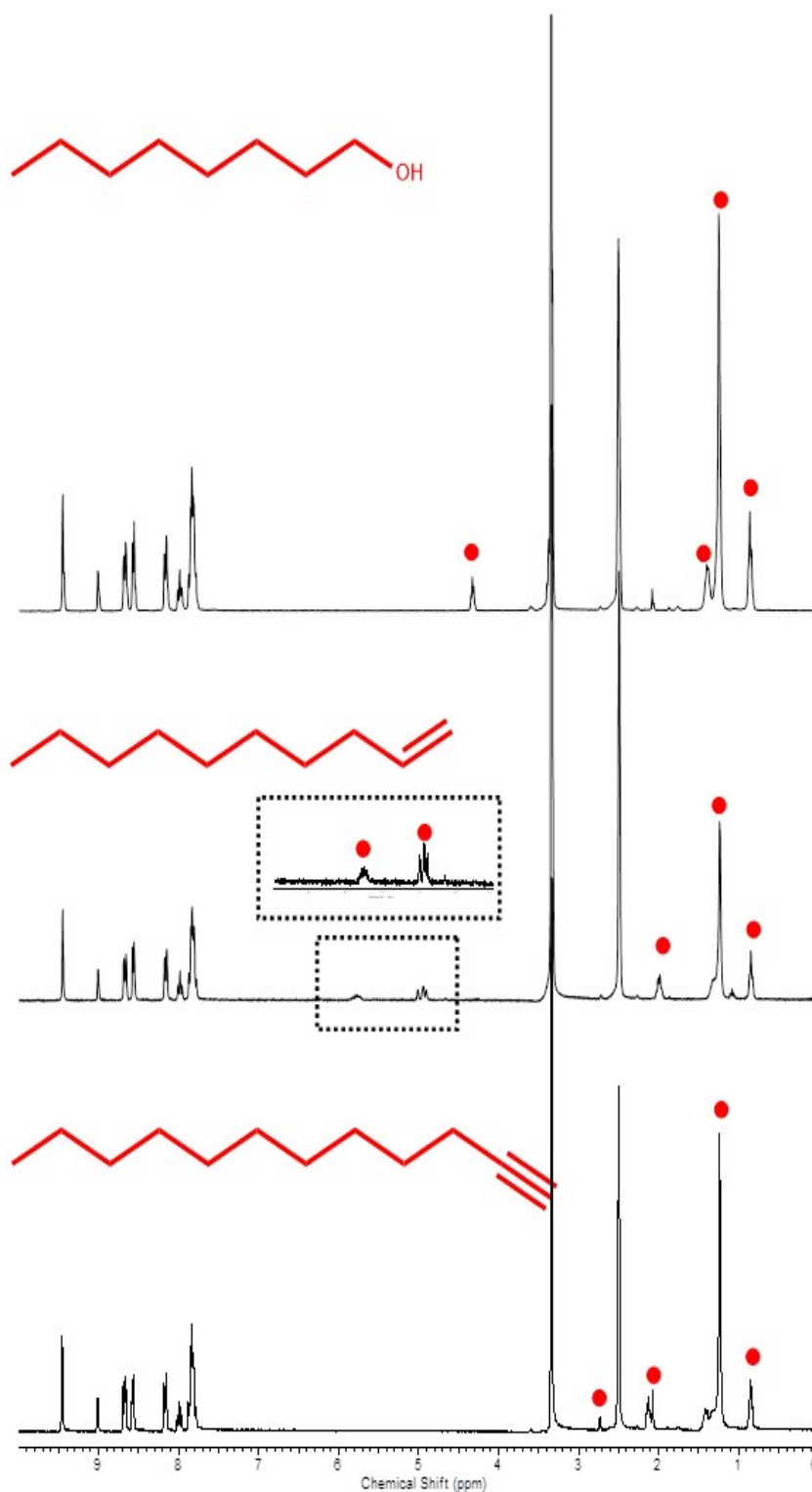


**Fig. S2.** TGA and DSC of  $C_6H_6@[Zn_2I_4L_2]$ ,  $2CH_2I_2 \cdot C_6H_6@[Zn_2I_4L_2]$ ,  $2.2C_8H_6@[Zn_2I_4L_2]$ ,  $C_7H_8@[Zn_2I_4L_2]$ ,  $C_6H_8@[Zn_2I_4L_2]$ ,  $C_8H_{18}O@[Zn_2I_4L_2]$ ,  $C_{10}H_{20}@[Zn_2I_4L_2]$ ,  $C_{12}H_{22}@[Zn_2I_4L_2]$ .

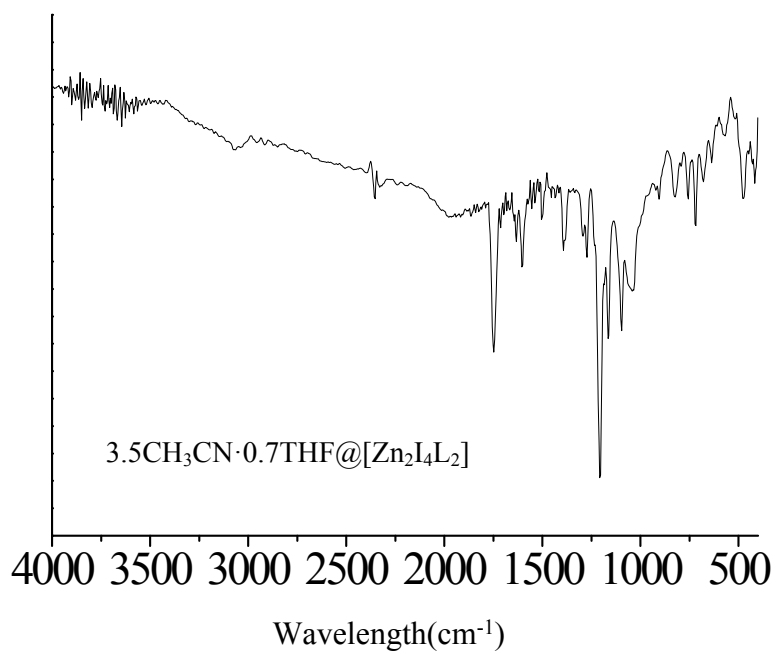
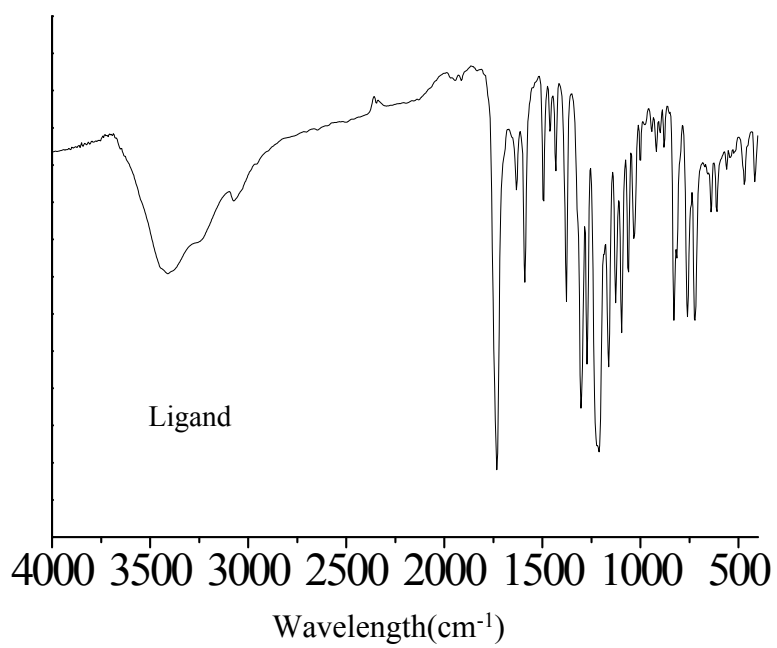
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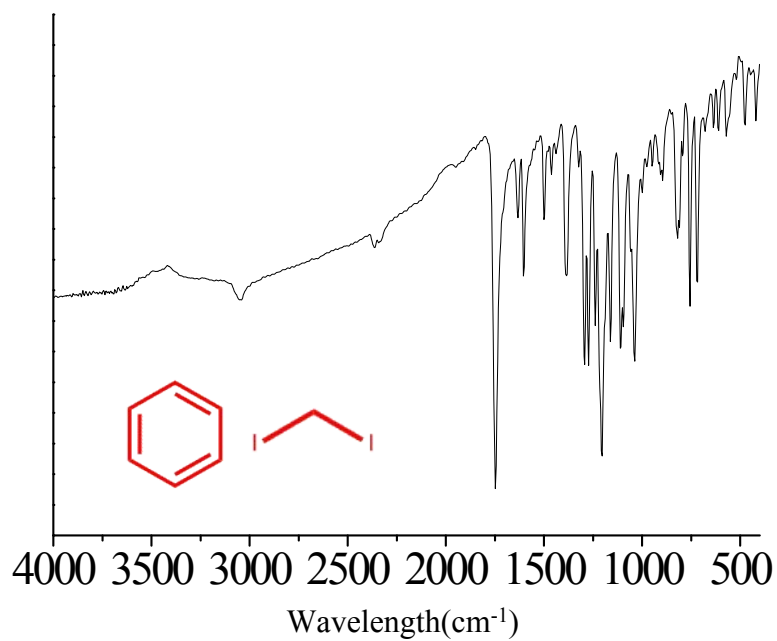
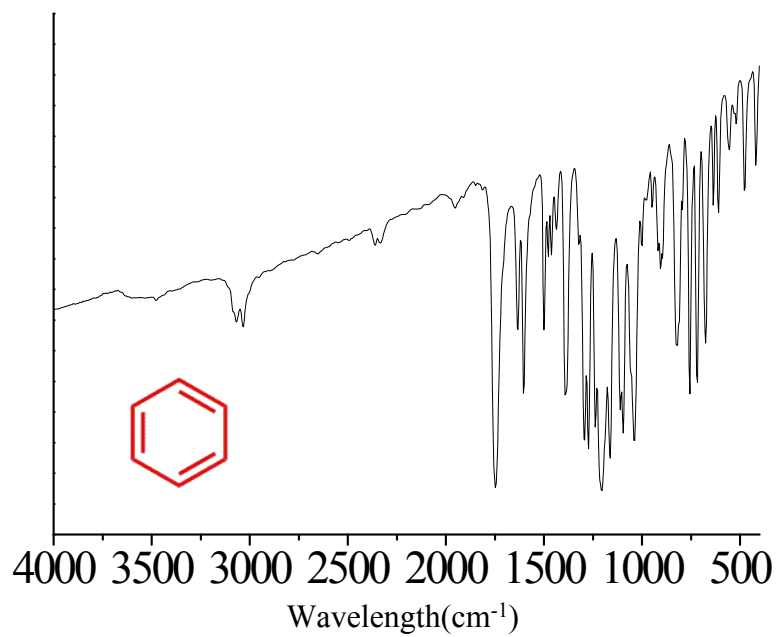


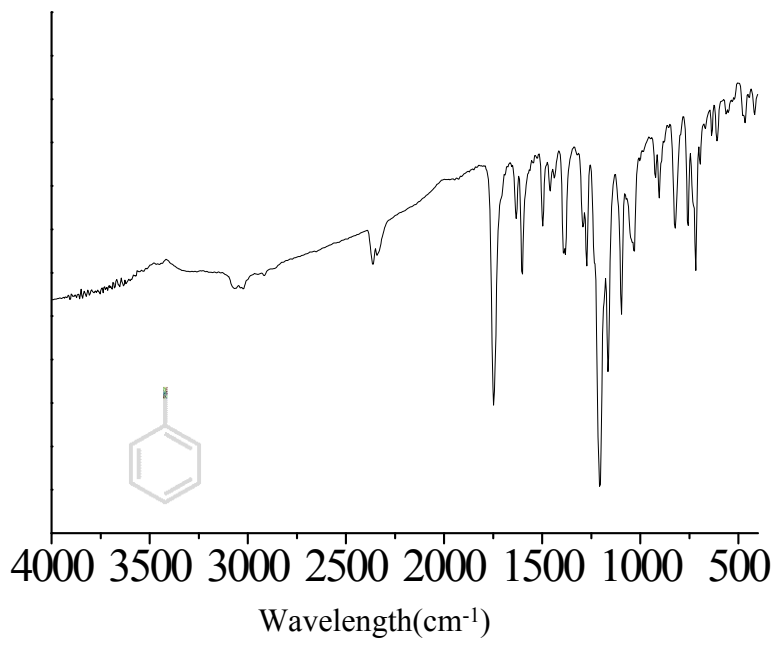
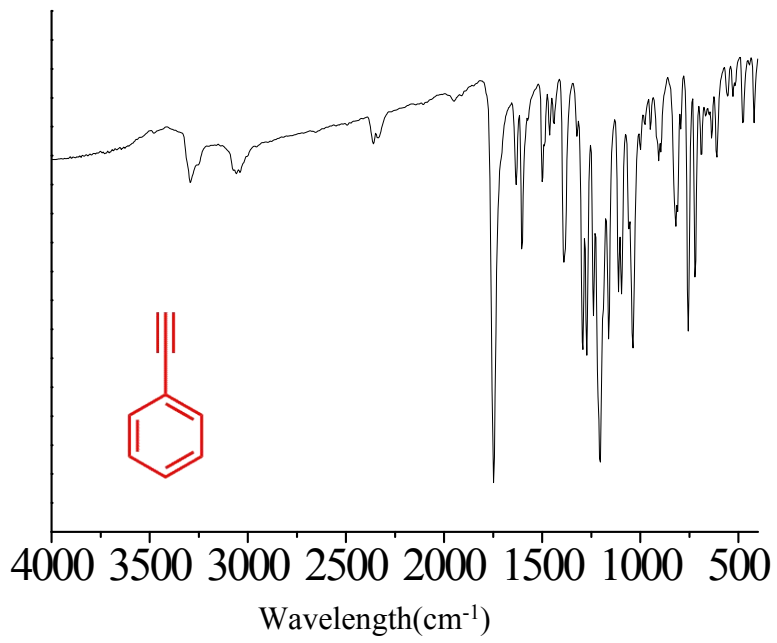


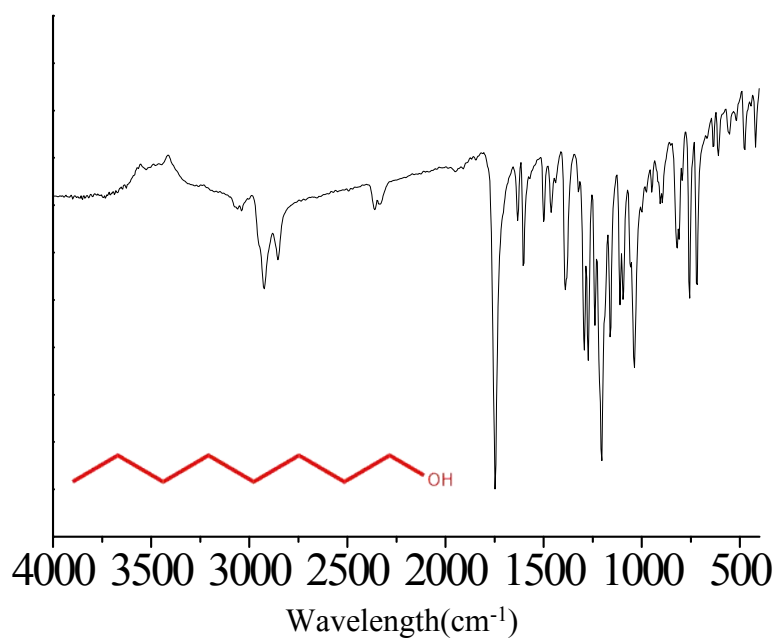
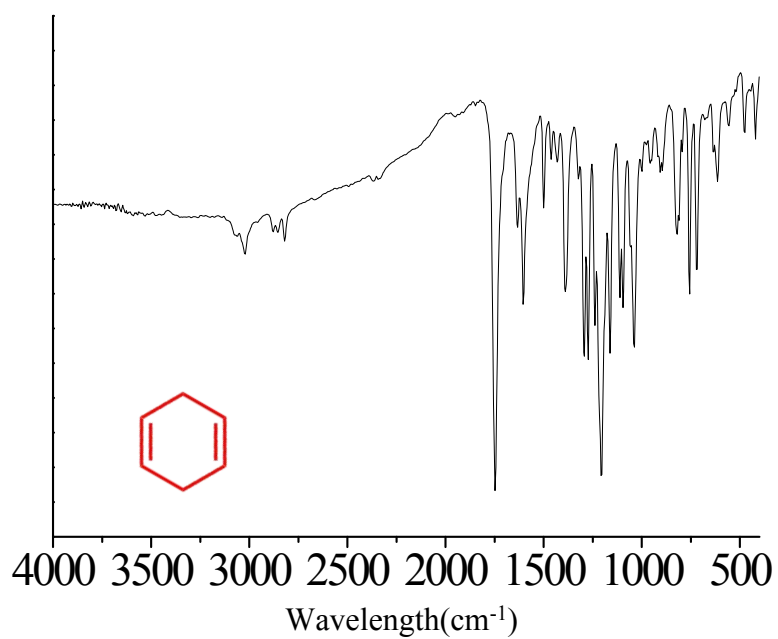


**Fig. S3.**  $^1\text{H}$  NMR spectra of  $3.5\text{CH}_3\text{CN}\cdot 0.7\text{THF}@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_6\text{H}_6@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $2\text{CH}_2\text{I}_2\cdot\text{C}_6\text{H}_6@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $2.2\text{C}_8\text{H}_6@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_7\text{H}_8@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_6\text{H}_8@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_8\text{H}_{18}\text{O}@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_{10}\text{H}_{20}@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_{12}\text{H}_{22}@\text{[Zn}_2\text{I}_4\text{L}_2]$ .

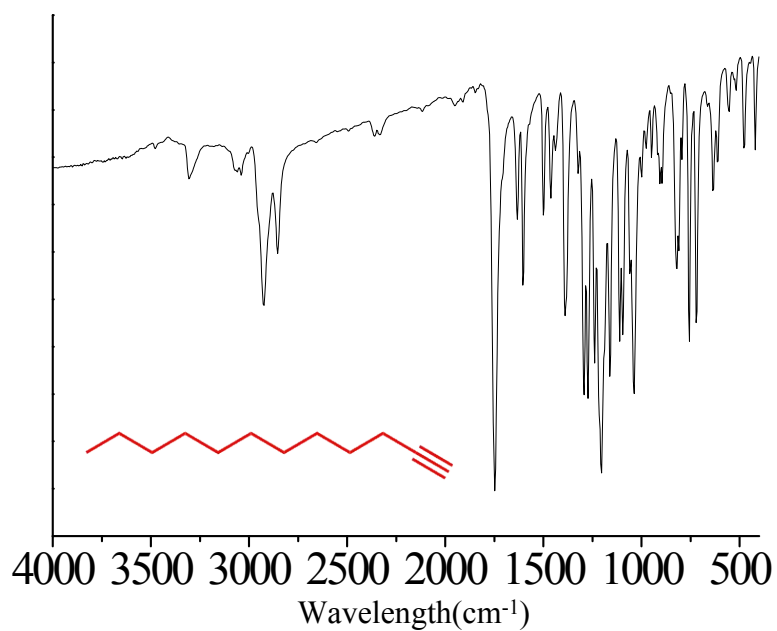
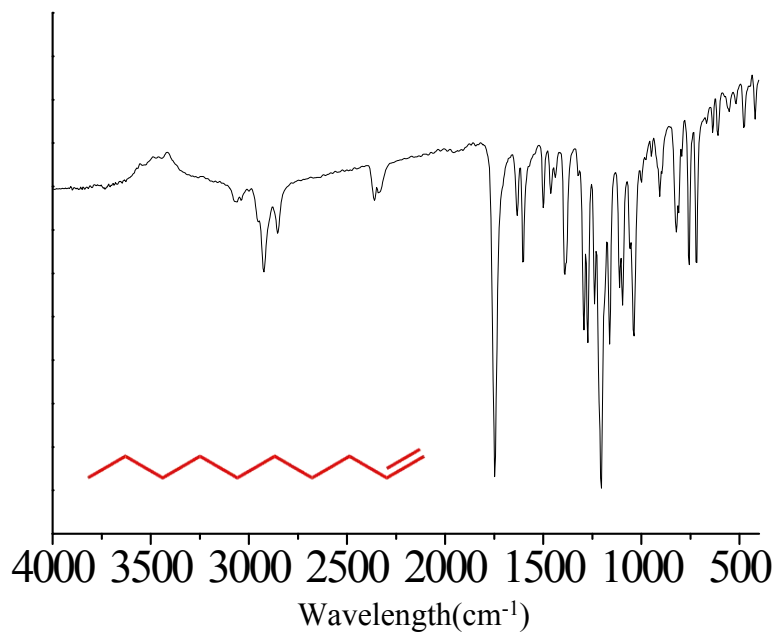




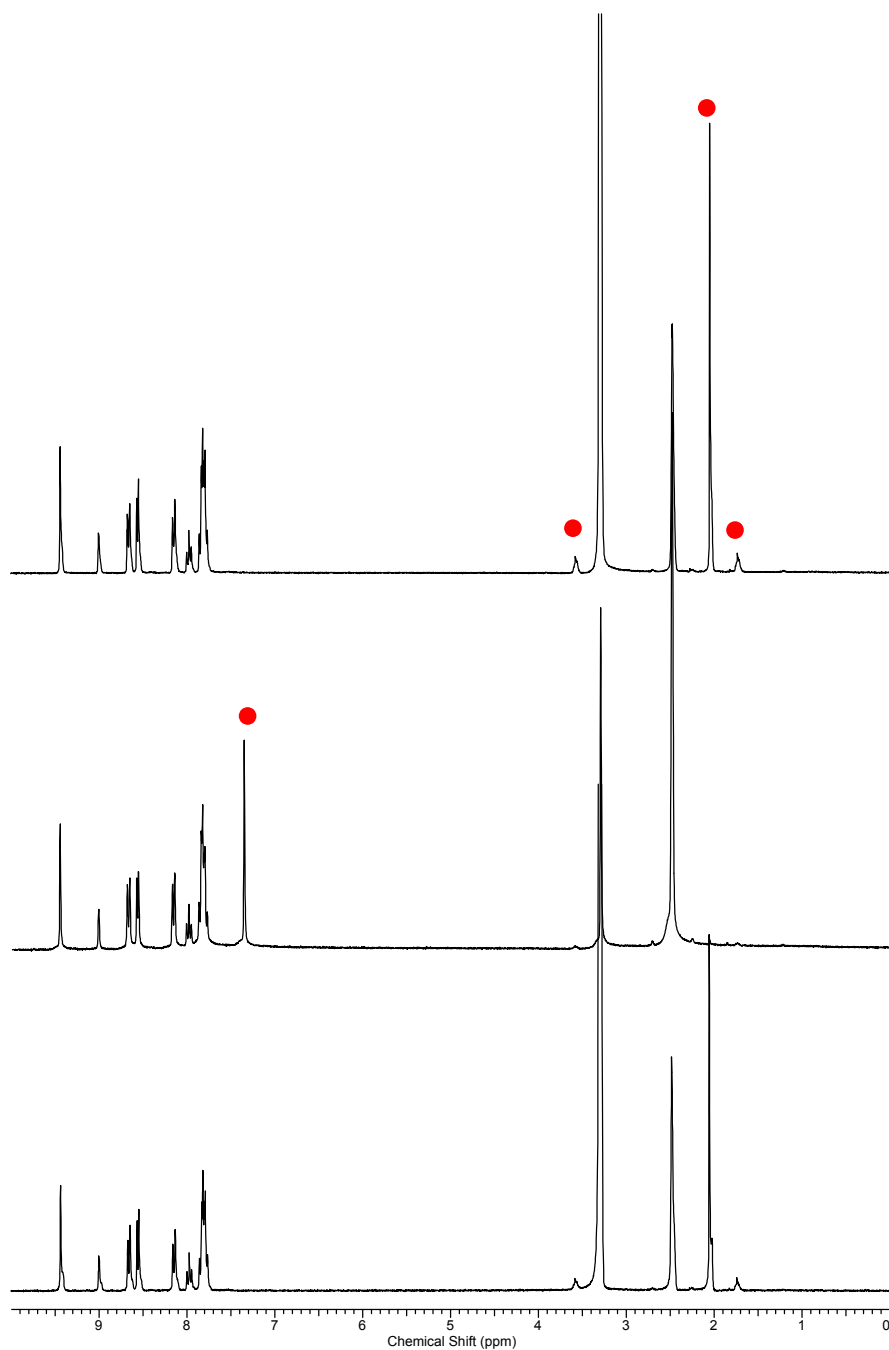
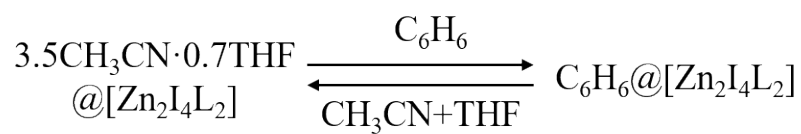








**Fig. S4.** IR spectra of Ligand,  $3.5\text{CH}_3\text{CN}\cdot 0.7\text{THF}@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_6\text{H}_6@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $2\text{CH}_2\text{I}_2\cdot\text{C}_6\text{H}_6@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $2.2\text{C}_8\text{H}_6@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_7\text{H}_8@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_6\text{H}_8@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_8\text{H}_{18}\text{O}@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_{10}\text{H}_{20}@\text{[Zn}_2\text{I}_4\text{L}_2]$ ,  $\text{C}_{12}\text{H}_{22}@\text{[Zn}_2\text{I}_4\text{L}_2]$ .



**Fig. S5.**  $3.5\text{CH}_3\text{CN}\cdot 0.7\text{THF}@[\text{Zn}_2\text{I}_4\text{L}_2]$  (a), Single crystals of  $3.5\text{CH}_3\text{CN}\cdot 0.7\text{THF}@[\text{Zn}_2\text{I}_4\text{L}_2]$  were left benzene media at room temperature for 12 h (b), Single crystals of  $\text{C}_6\text{H}_6@[\text{Zn}_2\text{I}_4\text{L}_2]$  were left a mixture of  $\text{CH}_3\text{CN}$  and  $\text{THF}$  ( $v/v = 1 : 1$ ) media at room temperature for 12 h (c).