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Cu_4I_4-cubane cluster based on tris(*p*-anisyl)arsine: synthesis, crystal structure and photophysical properties

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§1. Materials and Instrumentation

Caution! These operations, which contain organoarsenic compounds could be potential hazardous. For safety, experiments should be performed in an efficient hood.

$AsAn_3$ was synthesized following the described procedure[1]. CuI (99%, Sigma-Aldrich), *n*-BuLi (2,5 M hexane solution, Sigma) and EtCN (≥99%, Sigma-Aldrich) were used as purchased. All the solvents (CH_2Cl_2, THF, hexane) prior to use were purified by common protocols.

FT-IR spectra were collected on a Bruker Vertex 80 spectrometer. The CHN microanalysis was performed on a MICRO cube analyzer. Thermogravimetric analyses (TGA/c-DTA/DTG) were carried out in a closed Al_2O_3 pan under argon flow at 10 °C/min^{-1} heating rate using a NETZSCH STA 449 F1 Jupiter STA.

Powder X-ray diffraction (PXRD) patterns were recorded on a Shimadzu XRD-7000 diffractometer (Cu-Kα radiation, Ni – filter, 3–35° 2θ range, 0.03° 2θ step, 5s per point).

Emission and excitation spectra were recorded on a Fluorolog 3 spectrometer (Horiba Jobin Yvon) equipped with a cooled PC177CE-010 photon detection module and an R2658 photomultiplier. The absolute PLQYs were determined at 298 K using a Fluorolog 3 Quanta-phi integrating sphere. Temperature-dependent excitation and emission spectra as well as emission decays were recorded using an Optistat DN optical cryostat (Oxford Instruments) integrated with above spectrometer.

§2. Synthetic procedures and characterization data

$[Cu_4I_4(AsAn_3)_4]\cdot CH_2Cl_2$ (**1**·CH_2Cl_2).

To an EtCN (1 mL) solution of CuI (25 mg, 0.131 mmol) a solution of $AsAn_3$ (52 mg, 0.131 mmol) in CH_2Cl_2 (4 ml) was added dropwise. The resulting mixture was stirred at room temperature for 30 min. The precipitate formed was centrifuged and dried in air. White powder. Yield: 65 mg (82%). FT-IR (KBr, cm^{-1}): 413 (w), 480 (m), 521 (s), 598 (w), 633 (w), 712 (w), 750 (m), 793 (s), 822 (s), 937 (w), 961 (w), 1007 (m), 1028 (s), 1078 (s), 1105 (m), 1177 (vs), 1250 (vs), 1288 (s), 1306 (m), 1400 (m), 1439 (m), 1460 (s), 1497 (vs), 1570 (s), 1591 (s), 1888 (w), 2039 (w), 2835 (m), 2940 (m), 2955 (m), 2999 (w), 3059 (w). Anal. Calc. for $C_{85}H_{86}As_4Cu_4I_4Cl_2O_{12}$ (2431.98): C, 42.0; H, 3.6. Found: C, 41.9; H. 3.4.

§3. Powder X-ray diffraction data

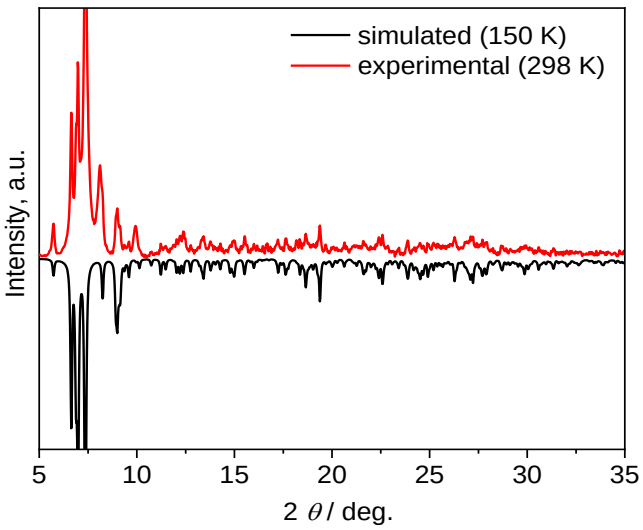

Figure S1. Experimental and simulated PXRD patterns of **1**·CH_2Cl_2.

§4. FT-IR spectra

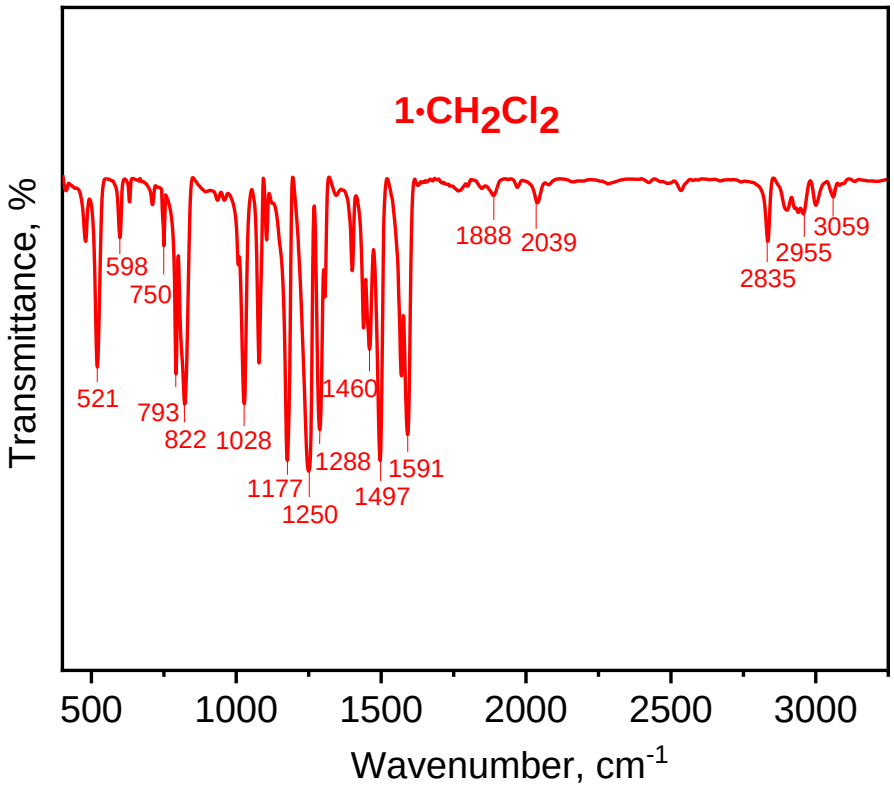

Figure S2. FT-IR spectrum for **1**·CH_2Cl_2 in the 400–3250 cm^{-1} region.

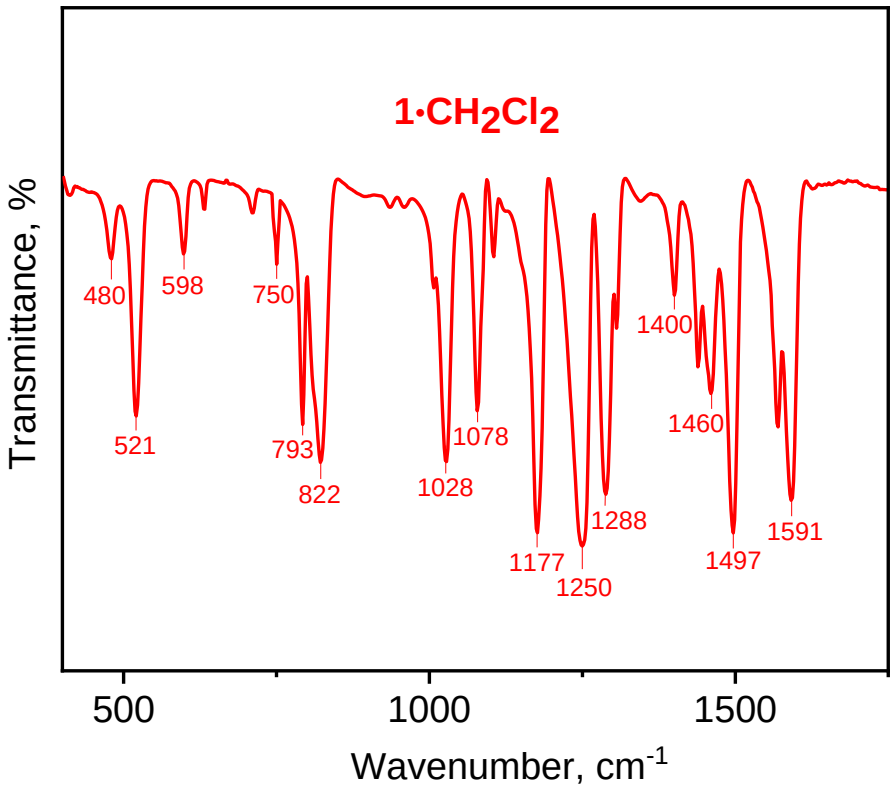

Figure S3. FT-IR spectrum for **1**·CH_2Cl_2 in the fingerprint region.

§5. TGA&DTG curves

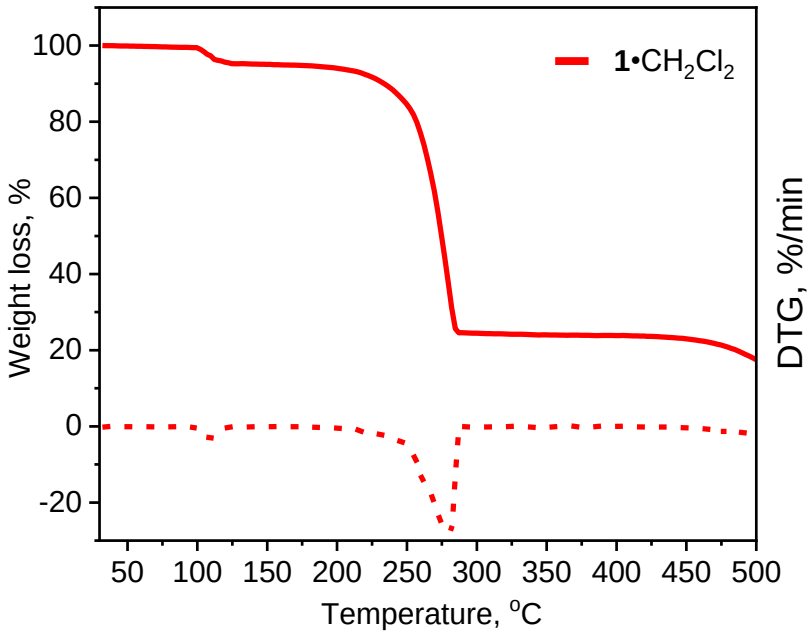

Figure S4. TGA&DTG curve for **1**·CH_2Cl_2.

§6. Temperature dependent excitation spectra of 1·CH_2Cl_2

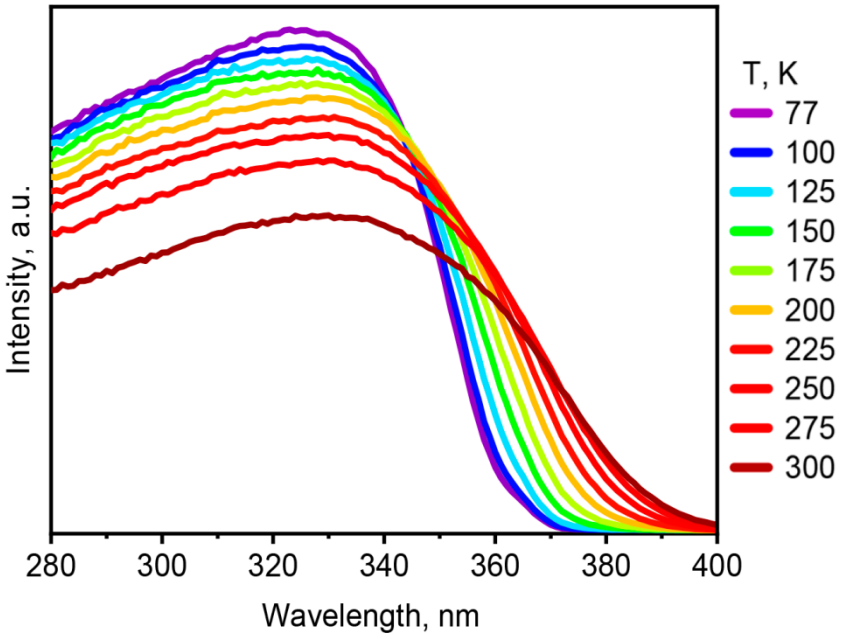

Figure S5. Temperature dependent excitation spectra of **1**·CH_2Cl_2 (λ_{em} = 575 nm).

§7. References

[1] S. Tanaka, M. Konishi, H. Imoto, Y. Nakamura, M. Ishida, H. Furuta, K. Naka, *Inorg. Chem.*, 2020, **59**, 9587.