

Electronic Supplementary Information for Dalton Transactions
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**Structural and photophysical studies of phenanthrene adducts
involving C_6F_5HgCl and $[o-C_6F_4Hg]_3$**

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Supporting Information

- Experimental details
- Fluorescence quenching experiments

I. Experimental details

General. Due to the toxicity of the mercury compounds discussed, extra care was taken at all times to avoid contact with solid, solution, and air-borne particulate mercury compounds. The studies herein were carried out in a well aerated fume hood. Atlantic Microlab, Inc., Norcross, GA, performed the elemental analyses. All commercially available starting materials and solvents were purchased from Aldrich Chemical and VWR, Inc. and used as provided. Compound **1** and **2** were prepared according to published procedures (see ref 2 and 10 of the main text).

Synthesis of adducts. All compounds were prepared by mixing compound **1** or **2** with the corresponding arene in THF or CHCl₃ (5–10 mL). Crystals formed upon slow evaporation of the solvent. Reagent quantities, yields, elemental analysis results, and melting points are provided for each adduct hereafter.

[**1**•phenanthrene]: **1** (0.020 g, 0.019 mmol); phenanthrene (0.0039 g, 0.019 mmol) in THF. Yield: 0.020 g, 87%. Anal. Calcd for C₃₂H₁₀F₁₂Hg₃: C, 31.27; H, 0.82. Found: C, 30.88; H 0.84. mp 321 °C (decomp).

[**2**•phenanthrene]: **2** (0.014 g, 0.035 mmol), phenanthrene (0.0061 g, 0.035 mmol) in CHCl₃. Yield: 0.0187 g, 92%. Anal. Calcd for C₂₀H₁₀F₅HgCl: C, 41.32; H, 1.73. Found: C, 41.37; H, 1.74. mp 230 °C.

[**2**•fluorene]: **2** (0.015 g, 0.037 mmol), fluorene (0.0061 g, 0.037 mmol) in CHCl₃. Yield: 0.0183 g, 87%. Anal. Calcd for C₁₉H₁₀F₅HgCl: C, 40.08; H, 1.77. Found: C, 40.21; H, 1.85. mp 219 °C. The structure of this adduct has also been determined.

Selected crystallographic data for [**2**•fluorene]: $M = 569.31$, $T = 110(2)$ K, monoclinic, space group $C2/c$ (No.15), $a = 6.770(3)$, $b = 35.555(16)$, $c = 7.066(3)$ Å, $\beta = 102.402(8)^\circ$, $V = 1661.3(13)$ Å³, $Z = 4$, $\rho_{\text{calc}} = 2.276$ gcm⁻³, μ (Mo-K α) = 9.477 mm⁻¹, independent reflections = 1837 ($R_{\text{int}} = 0.0946$), $R1$ [for 1297 reflections with $I > 2\sigma(I)$] = 0.0684, $wR2$ (all data) = 0.1575.

[2•pyrene]: **2** (0.015 g, 0.037 mmol), pyrene (0.0075 g, 0.037 mmol) in CHCl₃. Yield: 0.0200 g, 89%. Anal. Calcd for C₂₂H₁₀F₅HgCl: C, 43.65; H, 1.67. Found: C, 43.39; H, 1.67. mp 239 °C (decomp).

Crystal Structure Determinations. X-ray data for [1•phenanthrene], [2•phenanthrene], and [2•fluorene] were collected on a Bruker SMART-CCD diffractometer using graphite-monochromated Mo K α radiation ($\lambda = 0.71073$ Å). Specimens of suitable size and quality were selected and glued onto a glass fiber with freshly prepared epoxy resin. The structure was solved by direct methods, which successfully located most of the non-hydrogen atoms. Subsequent refinement on F^2 using the SHELXTL/PC package (version 6.1) allowed location of the remaining non-hydrogen atoms. Details of the crystal structures can be found in the CIF files.

Luminescence Titrations. The solution luminescence spectra were recorded with a SLM/AMINCO, Model 8100 spectrofluorometer equipped with a xenon lamp. Titration experiments with **1** were performed by adding aliquots of a CH₂Cl₂ solution of **1** (100 μ L 6.9×10^{-3} M) to a quartz cuvette containing 3.00 mL of a 1.6×10^{-4} M solution of phenanthrene. Experiments with **2** were performed in a similar fashion by titrating a CHCl₃ solution of **2** (25 μ L of 1.1×10^{-2} M) into a cuvette containing 3.00 mL of a 1.7×10^{-5} M (phenanthrene); or 8.8×10^{-6} M (fluorene) CHCl₃ solution of the arene. The samples were excited at $\lambda_{\text{excited}} = 295$ and 299 nm (phenanthrene with **1** and **2** respectively) and 274 nm (fluorene) and the emission intensities at $\lambda_{\text{max}} = 349$ nm (phenanthrene), and 306 nm (fluorene) were monitored during the titration.

The solid luminescence spectra were recorded on a SLM/AMINCO, Model 8100 spectrofluorometer equipped with a xenon lamp. Low-temperature measurements were made in a cryogenic device of local design. Collodion was used to attach the powder samples to the holder. The collodion was scanned for baseline subtraction. Liquid nitrogen was used to obtain the 77 K measurements.

II. Fluorescence quenching experiments

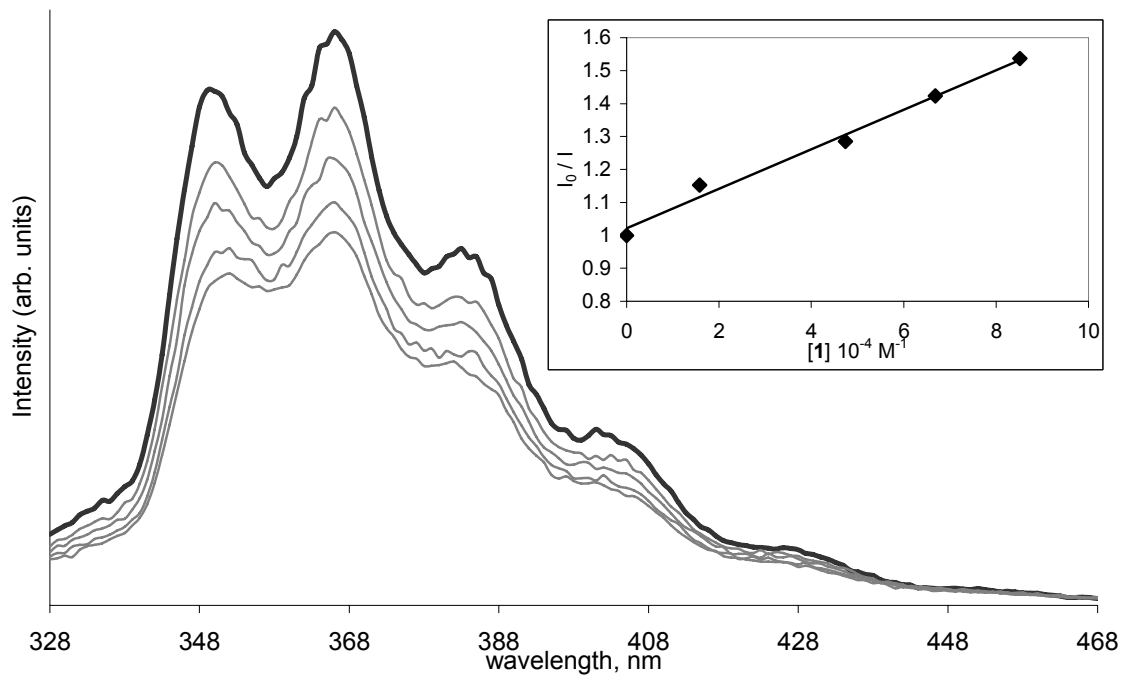


Figure 1. Fluorescence quenching of phenanthrene by 1. Inset: Stern Volmer plot.

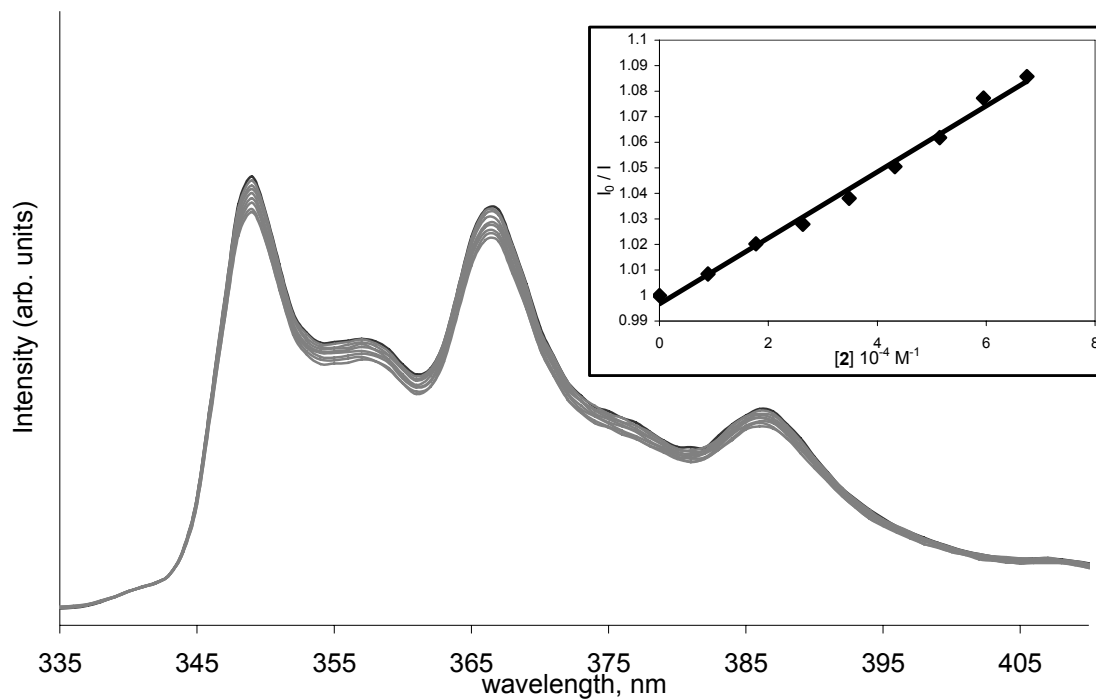


Figure 2. Fluorescence quenching of phenanthrene by 2. Inset: Stern Volmer plot.