Supporting Information for:

A Naphthalimide-based fluorescent sensor for halogenated solvents

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^a Key Laboratory of Separation Science for Analytical Chemistry, Dalian Institute of Chemical Physics, Chinese Academy of Sciences, Dalian 116023, China. E-mail: <u>zcxu@dicp.ac.cn</u> ^bKey Laboratory of Pesticide & Chemical Biology, Ministry of Education, College of Chemistry, Central China Normal University, Wuhan 430079, P. R. China. E-mail: yinj@mail.ccnu.edu.cn ^c University of Chinese Academy of Sciences, Beijing 100039, China. **Materials and Instruments:** Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. ¹H-NMR and ¹³C-NMR spectra were recorded on a Bruker 400 spectrometer, using TMS as an internal standard. Chemical shifts were given in ppm and coupling constants (*J*) in Hz. Mass spectrometry data were obtained with a HP1100LC/MSD mass spectrometer and a LC/Q-TOF MS spectrometer. UV—vis absorption spectra were collected on an Agilent Cary 60 UV-Vis Spectrophotometer. Fluorescence measurements were performed on a VAEIAN CARY Eclipse fluorescence spectrophotometer equipped with a Single Cell Peltier temperature controller. For temperature dependence studies, the cuvettes were tightly sealed with a cap to prevent solvent evaporation; the temperature was held for 20—30 min to ensure that the solution in the cuvettes reached thermal equilibrium before data collection.

Synthesis:



A suspension of compound **2** (3.89 g, 10.00 mmol), Pd(PPh₃)₂Cl₂ (36.79 mg, 0.03 mmol), and CuI (7.62 mg, 0.0,4 mmol) in a mixture of THF and Et₃N (50 mL each) was degassed with nitrogen for 15 min at room temperature. 4-ethynylaniline (1.18 g, 10.10 mmol) in THF(50 mL) that was degassed with nitrogen was added to the mixture with stirring. The mixture was stirred at reflux for 12 h, and then the versatile solvents were removed under reduced pressure. The residue was purified by column chromatography packed with silica gel using dichloromethane/petroleum ether (10 : 1, v/v) as eluent to afford pure product **AMN** as an orange solid. Yield: 4.04 g, 95%. ¹H NMR(400 MHz, CDCl₃): δ_H = 8.74-8.76 (d, *J* = 7.80 Hz, 1H), 8.62-8.64 (d, *J* = 6.60 Hz, 1H), 8.52-8.54 (d, *J* = 7.20 Hz, 1H), 7.89-7.91 (d, *J* = 7.20 Hz, 1H), 7.82 (t, *J* = 7.20 Hz, 1H), 7.48-7.50 (d, *J* = 8.40 Hz, 2H), 6.70-6.72 (d, *J* = 8.40 Hz, 2H), 4.35 (t, *J* = 6.90 Hz, 2H), 3.99 (b, 2H), 3.70 (b, 4H), 2.72 (t, *J* = 6.90 Hz, 2H), 2.61 (b, 4H); ¹³C NMR (100 MHz, CDCl₃) δ ppm = 37.4, 53.9, 56.2, 67.1, 84.9, 100.9, 114.6, 122.7, 127.0, 128.5, 129.9, 130.4, 131.3, 132.5, 133.4, 147.6, 163.6, 163.9. MS (EI) m/z: calcd for C₂₆H₂₃N₃O₃ 425.17, found 425.19. Anal. Calcd for C₂₆H₂₃N₃O₃: C, 73.39; H, 5.45; N, 9.88. Found: C, 73.28; H, 5.39; N, 9.78.



Figure S1. The UV-Vis absorption spectra (A) and Fluorescence emission spectra (B) of **AMN** $(1 \times 10^{-5} \text{ M})$ in different non-halogenated solvents ($\lambda_{ex} = 420 \text{ nm}$).



Fig S2. ¹H-NMR spectra of **AMN** in CDCl₃.





Figure S4. MS spectrum of AMN.