

**Supplementary Material:**

## **Oil Spill Source Identification using Colorimetric Detection**

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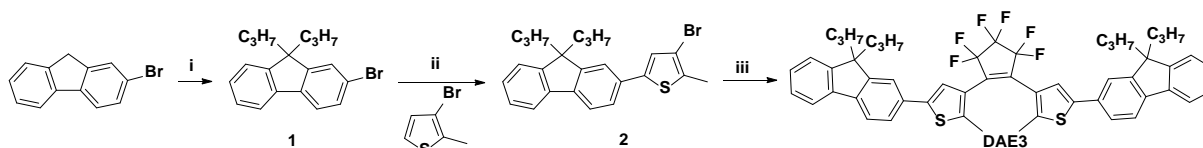
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## S1. Synthesis of DAE3:



Scheme S1: Synthetic route to DAE3. (i) 25 M sodium hydroxide (aq) then 1-bromopropane, 65 °C, N<sub>2</sub>, 16 h, (ii) pivalic acid, dimethylacetamide, potassium carbonate, palladium(II) acetate, 100 °C, N<sub>2</sub>, 16 h, (iii) *n*-butyllithium then octafluorocyclopentene, tetrahydrofuran, -78 °C to room temperature overnight.

**S1.1: Materials.** DAE1, DAE2, **1** and **2** were prepared according to literature methods.<sup>[1-3]</sup> 1-bromopropane (Sigma Aldrich), 3-bromo-2-methylthiophene (Combi Blocks), palladium acetate (Sigma Aldrich), potassium carbonate (Univar), pivalic acid (Sigma Aldrich), dimethylacetamide (Sigma Aldrich), propandiol (Sigma Aldrich), *p*-toluenesulfonic acid (Sigma Aldrich), *n*-butyllithium (Sigma Aldrich), tetrahydrofuran (Unichrome), octafluorocyclopentene (Synquest Labs) were used as received.

**S1.2: Characterisation.** <sup>1</sup>H spectra were recorded on a Bruker Ascend™ 400 MHz spectrometer. Chemical shifts are reported in parts per million (ppm) and referenced to the residual solvent peak (chloroform, <sup>1</sup>H = 7.26 ppm). Peak multiplicities are described in the following manner: singlet (s) and multiplet (m). Low resolution mass spectral data were measured by direct infusion of the diluted sample (Cole Palmer 74900 syringe pump at a flow rate of 150 μL h<sup>-1</sup>) on a Bruker Daltonics Esquire3000plus ion trap electrospray ionisation mass spectrometer (ESI MS) with an Apollo source operating at 40 eV in positive mode. All MS data was collected using Bruker Daltonics Esquire Control v5.3 and Hystar v3.1 operating on Windows XP Professional. Melting points were measured using a Stuart Scientific Melting Point SMP1 apparatus.

X-ray diffraction structure determination: The single crystal coated with viscous hydrocarbon oil was mounted on glass fiber. Data were obtained at ambient temperature on a Bruker APEXII-CCD diffractometer with graphite-monochromated Mo-K $\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ). The data set was empirically corrected for absorption (SADABS)<sup>[4]</sup> and then merged. The structure was solved by conventional methods and refined by full-matrix least-squares on all F<sup>2</sup> data using SHELX-2014,<sup>[5]</sup> in conjunction with the X-Seed<sup>[6]</sup> or Olex2 graphical user interface. All hydrogen atoms were placed in calculated positions using the riding model.

All hydrogen atoms were placed in calculated positions using the riding model. CCDC-1541410 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

Crystal data of DAE3: C<sub>53</sub>H<sub>50</sub>F<sub>6</sub>S<sub>2</sub> ( $M = 865.05$ ), blue prism,  $0.45 \times 0.2 \times 0.1 \text{ mm}^3$ , space group P-1 (No. 2),  $V = 2329.9(10) \text{ \AA}^3$ ,  $Z = 2$ ,  $D_c = 1.233 \text{ g/cm}^3$ ,  $F_{000} = 908$ , Bruker SMART CCD, MoK $\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$ ,  $T = 298 \text{ K}$ ,  $2\theta_{\text{max}} = 50.0^\circ$ , 25305 reflections collected, 8157 unique ( $R_{\text{int}} = 0.0507$ ). Final  $Goof = 1.038$ ,  $RI = 0.0684$ ,  $wR2 = 0.1831$ ,  $R$  indices based on 5845 reflections with  $I > 2\sigma(I)$  (refinement on  $F^2$ ), 556 parameters, 24 restraints. Lp and absorption corrections applied,  $\mu = 0.173 \text{ mm}^{-1}$ .

### S1.3: Synthetic Methodology.

DAE3: *n*-Butyllithium (0.62 mL, 1.45 M) was added dropwise to a stirred solution of **2** (0.42 g, 0.99 mmol) in dry THF (4 mL) at  $-78 \text{ }^\circ\text{C}$ . The reaction was stirred for 30 minutes then octafluorocyclopentene (0.105 g, 0.495 mmol) added dropwise. This reaction mixture was stirred for a further 30 min at  $-78 \text{ }^\circ\text{C}$  prior to slowly warming to room temperature overnight. The THF was removed under vacuum and the resulting residue taken up in diethyl ether (150 mL). This ether solution was consecutively washed with water (100 mL) and brine (100 mL) prior to drying over Na<sub>2</sub>SO<sub>4</sub> and the removal of the solvent under vacuum. The product was purified by column chromatography on silica gel eluting with petroleum ether / DCM (95:5) to afford DAE3 as a blue crystalline solid (0.175 g, 41 %). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  0.67 (m, 12H), 1.97 (m, 8H), 2.06 (s, 6H), 7.30-7.36 (m, 8 H), 7.47-7.54 (m, 4H), 7.67-7.70 (m, 4H); mp  $103 \pm 1.5 \text{ }^\circ\text{C}$ ; (+)-ESI-MS  $m/z$   $[\text{M} + \text{H}]^+$  865.34, (calcd for C<sub>53</sub>H<sub>51</sub>S<sub>2</sub>F<sub>6</sub><sup>+</sup>, 865.33),  $m/z$   $[\text{M} + \text{Na}]^+$  887.41 (calcd for C<sub>53</sub>H<sub>50</sub>S<sub>2</sub>F<sub>6</sub>Na<sup>+</sup>, 887.32),  $m/z$   $[\text{M} - \text{F}]^+$  845.38 (calcd for C<sub>53</sub>H<sub>50</sub>S<sub>2</sub>F<sub>5</sub><sup>+</sup>, 845.32); (-)-ESI-MS  $m/z$   $[\text{M} - \text{H}]^-$  863.35 (calcd for C<sub>53</sub>H<sub>49</sub>S<sub>2</sub>F<sub>6</sub><sup>-</sup>, 863.33).

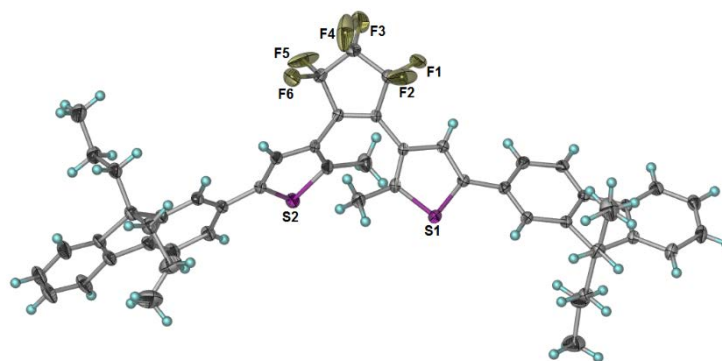


Figure S1: X-ray crystal structure of DAE3 in the opened form.

## S2. References

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