## **Supplementary Information**

## Azocalix[4]arene-based Chromogenic Anion Probes

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Figure S1. (a) UV-vis titrations of 1 (10<sup>-5</sup> M) with F<sup>-</sup> in MeCN, [F<sup>-</sup>]: from  $a \rightarrow i 0 - 8$  eq. of 1; (b) the plot of A<sub>0</sub>/(A-A<sub>0</sub>) versus [F<sup>-</sup>]<sup>-1</sup>.



Figure S2. UV-vis titrations of 2 (10<sup>-5</sup> M) with F<sup>-</sup> in MeCN, [F<sup>-</sup>]: from  $a \rightarrow f$ : 0 - 6eq. of 2; (b) the plot of A<sub>0</sub>/(A-A<sub>0</sub>) versus [F<sup>-</sup>]<sup>-1</sup>.



**Figure S3.** (a) UV-vis titrations of **2** (10<sup>-5</sup> M) with AcO<sup>-</sup> in MeCN, [AcO<sup>-</sup>]: from  $a \rightarrow f : 0 - 1.0$ eq. of **2**; (b) the plot of A<sub>0</sub>/(A-A<sub>0</sub>) versus [AcO<sup>-</sup>]<sup>-1</sup>.

![](_page_4_Figure_1.jpeg)

**Figure S4.** (a) UV-vis titrations of **3** (10<sup>-5</sup> M) with F<sup>-</sup> in MeCN, [F<sup>-</sup>]: from a  $\rightarrow$ g: 0 - 6eq. of **3**; (b) the plot of A<sub>0</sub>/(A-A<sub>0</sub>) versus [F<sup>-</sup>]<sup>-2</sup>.

![](_page_5_Figure_1.jpeg)

**Figure S5.** (a) UV-vis titrations of **3** (10<sup>-5</sup> M) with AcO<sup>-</sup> in MeCN, [AcO<sup>-</sup>]: from a  $\rightarrow$  f : 0 – 5.0eq. of **3**; (b) the plot of A<sub>0</sub>/(A-A<sub>0</sub>) versus [AcO<sup>-</sup>]<sup>-1</sup>.

![](_page_6_Figure_1.jpeg)

**Figure S6.** (a) UV-vis titrations of **3** (10<sup>-5</sup> M) with H<sub>2</sub>PO<sub>4</sub><sup>-</sup> in MeCN, [H<sub>2</sub>PO<sub>4</sub><sup>-</sup>]: from a  $\rightarrow$  f: 0 – 5.0eq. of **3**; (b) the plot of A<sub>0</sub>/(A-A<sub>0</sub>) versus [H<sub>2</sub>PO<sub>4</sub><sup>-</sup>]<sup>-1</sup>.

![](_page_7_Figure_1.jpeg)

**Figure S7.** (a) UV-vis titrations of 4 (10<sup>-5</sup> M) with F<sup>-</sup> in MeCN, [F<sup>-</sup>]: from a  $\rightarrow$ g 0 – 2.46eq. of 4; (b) the plot of A<sub>0</sub>/(A-A<sub>0</sub>) versus [F<sup>-</sup>]<sup>-2</sup>.

![](_page_8_Figure_1.jpeg)

**Figure S8.** (a) UV-vis titrations of 4 (10<sup>-5</sup> M) with AcO<sup>-</sup> in MeCN, [AcO<sup>-</sup>]: from  $a \rightarrow e: 0 - 0.6eq$ . of 4; (b) the plot of A<sub>0</sub>/(A-A<sub>0</sub>) versus [AcO<sup>-</sup>]<sup>-1</sup>.

![](_page_9_Figure_1.jpeg)

**Figure S9.** (a) UV-vis titrations of **5** (10<sup>-5</sup> M) with F<sup>-</sup> in MeCN. [F<sup>-</sup>]: from a  $\rightarrow$  f: 0 – 2.4eq. of **5**; (b) the plot of A<sub>0</sub>/(A-A<sub>0</sub>) versus [F<sup>-</sup>]<sup>-2</sup>.

![](_page_10_Figure_1.jpeg)

**Figure S10.** (a) UV-vis titrations of **5** (10<sup>-5</sup> M) with AcO<sup>-</sup> in MeCN, [AcO<sup>-</sup>]: from a  $\rightarrow$  f: 0 – 2.4 eq. of **5**; (b) the plot of A<sub>0</sub>/(A-A<sub>0</sub>) versus [AcO<sup>-</sup>]<sup>-1.5</sup>.

![](_page_11_Figure_1.jpeg)

**Figure S11.** (a) UV-vis titrations of **5** (10<sup>-5</sup> M) with H<sub>2</sub>PO<sub>4</sub><sup>-</sup> in MeCN, [H<sub>2</sub>PO<sub>4</sub><sup>-</sup>]: from a  $\rightarrow$ h: 0 – 3.2 eq. of **5**; (b) the plot of A<sub>0</sub>/(A-A<sub>0</sub>) versus [H<sub>2</sub>PO<sub>4</sub><sup>-</sup>]<sup>-1</sup>.