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Supplemental Information

Selected data for *R*,*R*-**2**: ¹H NMR (300 MHz,CDCl₃) δ 7.85–7.16 (m, 30H), δ 4.44–4.67 (m, 4H), δ 4.13 (br, 2H), δ 3.77 (br, 4H), 1.73 (d, 6H, *J* = 6.0 Hz) ppm; FAB-LRMS (C₈₂H₇₀B₂N₆O₁₂, 3-nitrobenzylalcohol adduct) calcd 1352.5, found 1352.9. Anal Calcd for C₅₄H₅₀B₂N₂O₄: C, 79.8; H, 6.20; N: 3.45. Found: C, 79.4, H, 6.21; N, 3.45. [α]²²_D = -100±2° (*c* = 0.5, dichloromethane).

Selected data for *S*,*S*-**2**: ¹H NMR (300 MHz,CDCl₃) δ 7.83–7.13 (m, 30H), δ 4.43–4.65 (m, 4H), δ 4.09 (br, 2H), δ 3.77 (br, 4H), 1.71 (d, 6H, *J* = 6.0 Hz) ppm; FAB-LRMS (C₈₂H₇₀B₂N₆O₁₂, 3-nitrobenzylalcohol adduct) calcd 1352.5, found 1353.0. Anal Calcd for C₅₄H₅₀B₂N₂O₄·0.1CH₂Cl₂: C, 79.1; H, 6.16; N: 3.41. Found: C, 79.0, H, 6.11; N, 3.36. [α]²²_D = +92±2° (*c* = 0.5, dichloromethane).

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Figure 1. Emission spectra of *S*,*S*-**2** in the presence of tartaric acid. a), *S*,*S*-**2** with D-tartaric acid; b), *S*,*S*-**2** with L-tartaric acid, λ ex at 370 nm, pH 2.5. 3.0×10^{-6} mol dm⁻³ of sensors in 5.0×10^{-2} mol dm⁻³ NaCl solution (52.1% methanol in water). 18°C.



Figure 2. Emission spectra of *S*,*S*-**2** in the presence of tartaric acid. a), *S*,*S*-**2** with D-tartaric acid; b), *S*,*S*-**2** with L-tartaric acid, λ ex at 275 nm, pH 2.5. 1.0×10^{-6} mol dm⁻³ of sensors in 5.0×10^{-2} mol dm⁻³ NaCl ionic buffer (52.1% methanol in water). 23°C.

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Figure 3. Fluorescence intensity-pH profile of the *S*,*S*-**2** with D- and L-tartaric acid; 3.0×10^{-6} mol dm⁻³ of sensor in 0.05 mol dm⁻³ sodium chloride ionic buffer (52.1% methanol), [L- and D-Tartaric acid] = 0.05 M. λ ex 370 nm, λ em 429 nm. 22°C.

S-sensor, apparent p*K*a = 5.72 \pm 0.07, r^2 = 0.98 L-*acid*, apparent p*K*a = 7.42 \pm 0.04, r^2 = 0.998 D-*acid*, apparent p*K*a = 8.65 \pm 0.04, r^2 = 0.995



Figure 4. Fluorescence intensity changes of *S*,*S*-**2**- *vs* enantiomeric composition of D-Tartaric acid. 3.0×10^{-6} mol dm⁻³ of *S*-sensor in 5.0×10^{-2} mol dm⁻³ M NaCl buffer (52.1% methanol in water), $\lambda ex 370$ nm, $\lambda em 429$ nm, pH 2.5, 20°C. [D-Tartaric acid] + [L-Tartaric acid] = 6.0×10^{-2} mol dm⁻³. The variation of the e.e. value of the D-tartaric acid was achieved with mixing the D-Tartaric acid and the L-Tartaric acid.

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Figure 5. Fluorescence intensity of the *R*,*R*-2 and *S*,*S*-2 *vs*. concentration of D- or L-Tartaric acid. 3.0×10^{-6} mol dm⁻³ of sensor in 0.05 mol dm⁻³ sodium chloride ionic buffer (52.1% methanol), [D- or L-Tartaric acid] = 0-0.01 M. The solid lines are the 1:1 binding regression results. λ ex 370 nm, λ em 429 nm. 22°C. pH 2.5, 5.6, 7.0, 8.3.