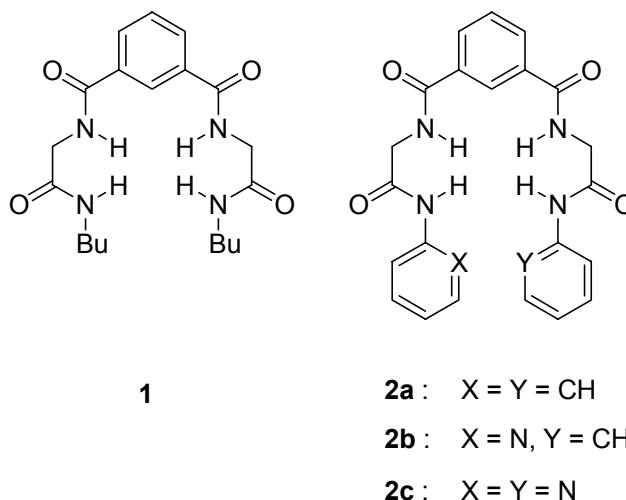


Electronic Supplemental Information

Experimental

All reagents used were of analytical grade. ^1H NMR spectra were measured on the JEOL AL-300 (300 MHz) spectrometer. Electron spray ionization mass spectra (ESI-MS) were recorded on the Applied Biosystems/MDS-Sciex API-100 spectrometer. Column chromatography was performed by using Wakogel C-200 (silica gel, 70-250 μm , Wako Chemical Co. Ltd). Melting points are uncorrected. Elemental analyses were performed at the Center of Instrumental Analysis of Gunma University.



Synthesis of the receptors

Receptor 1

Into a solution of 2-amino-*N*-butyl-acetamide (0.70 g, 5.4 mmol) and pyridine (0.42 g, 5.4 mmol) in DMF (40 ml), was slowly added isophthaloyl dichloride (0.50 g, 2.45 mmol) for 1 h at 0 °C under nitrogen atmosphere. The mixture was stirred at 0 °C for 1.5 h followed by at r.t. for 15 h. After evaporation under reduced pressure, the residue was recrystallized from MeOH-chloroform to give the desired product as colorless powder. Yield 0.115 g, 12%. Mp 234-236 °C.

^1H NMR (DMSO- d_6) δ 0.86 (t, 6H, J = 7.3 Hz), 1.26 (six, 4H, J = 7.3 Hz), 1.38 (quint, 4H, J = 6.8 Hz), 3.06 (q, 4H, J = 6.8 Hz), 3.84 (d, 4H, J = 5.9 Hz), 7.57 (t, 2H, J = 7.9 Hz), 7.88 (t, 4H, J = 5.7 Hz), 8.01 (dd, 2H, J_1 = 7.9, J_2 = 1.7 Hz), 8.38 (s, 1H), 8.77 (t, 2H, J = 5.7 Hz). Anal. Calcd for $\text{C}_{20}\text{H}_{30}\text{N}_4\text{O}_4$: C, 61.52; H, 7.74; N, 14.35. Found C, 61.39; H, 7.73; N, 14.34.

Receptor 2a

Into a solution of 2-amino-*N*-phenyl acetamide (**3**, 0.57 g, 3.8 mmol), isophthalic acid (0.27 g, 1.65 mmol), and 1-hydroxybenzotriazole (HOBt, 0.51 g, 3.8 mmol) in DMF (35 ml), was added dicyclohexylcarbodiimide (DCC, 0.8 g, 3.88 mmol) in DMF (25 ml) at 0 °C under nitrogen

atmosphere. The mixture was stirred at 0 °C for 1.5 h followed by at r.t. for 15 h. After filtration to remove dicyclohexylurea, the filtrate was evaporated under reduced pressure. The residue was washed with ethyl acetate and methanol, and recrystallized from DMF-MeOH to give the product as white solid. Yield 0.55 g, 78%. Mp > 280 °C.

¹H NMR (DMSO-*d*₆) δ 4.09 (d, 4H, *J* = 5.9 Hz), 7.04 (t, 2H, *J* = 7.3 Hz), 7.30 (t, 4H, *J* = 7.5 Hz), 7.60 (d, 4H, *J* = 8.3 Hz), 7.60 (t, 1H, *J* = 7.7 Hz), 8.05 (d, 2H, *J* = 7.7 Hz), 8.43 (s, 1H), 8.94 (t, 2H, *J* = 5.9 Hz), 10.46 (s, 2H). Anal. Calcd for C₂₄H₂₂N₄O₄: C, 66.97; H, 5.15; N, 13.02. Found C, 66.84; H, 5.18; N, 12.92. ESI-MS (negative ion mode): for [C₂₄H₂₂N₄O₄-H⁺]⁻ *m/z* = 429.16. Found *m/z* = 429.2.

Half amide 5

Into a solution of isophthalic acid (0.41 g, 2.5 mmol) and HOBT (0.31 g, 2.26 mmol) in DMF (25 ml), a solution of **3** (0.42 g, 2.26 mmol) in DMF (20 ml) and a solution of DCC (0.56 g, 2.71 mmol) were dropwised at once for 30 min at 0 °C under nitrogen atmosphere. The mixture was stirred at 0 °C for 1.5 h and followed by at r.t. for 6 h. After filtration to remove dicyclohexylurea, the filtrate was evaporated under reduced pressure. The residue was redissolved in ethyl acetate (100 ml) and filtered. The filtrate was stand overnight giving the precipitate. The precipitate was collected as white solid. Yield 0.28 g, 41%. Mp 262 °C (dec). ¹H NMR (DMSO-*d*₆) δ 4.07 (d, 2H, *J* = 6.1 Hz), 7.04 (t, 1H, *J* = 7.5 Hz), 7.30 (t, 2H, *J* = 7.5 Hz), 7.59 (d, 2H, *J* = 7.7 Hz), 8.10 (d, 1H, *J* = 8.6 Hz), 8.12 (d, 1H, *J* = 7.7 Hz), 8.49 (s, 1H), 9.04 (t, 1H, *J* = 6.1 Hz), 10.05 (s, 1H), 13.19 (bs, 1H).

Receptor 2b

Into a solution of **5** (0.38 g, 1.27 mmol), HOBT (0.18 g, 1.3 mmol), and 2-amino-*N*-(2-pyridyl)acetamide¹ (**4**, 0.23 g, 1.52 mmol) in DMF (30 ml), was dropwised DCC (0.31 g, 1.52 mmol) in DMF (25 ml) at 0 °C under nitrogen atmosphere. The mixture was stirred at 0 °C for 1.5 h and followed by at r.t. for 14 h. After filtration to remove dicyclohexylurea, the filtrate was evaporated under reduced pressure. The residue was washed with methanol and recrystallized from DMF-MeOH to give the product as white solid. Yield 0.34 g, 62%. Mp 255-257 °C.

¹H NMR (300 MHz, DMSO-*d*₆) δ 4.08 (d, 2H, *J* = 5.9 Hz), 4.15 (d, 2H, *J* = 5.9 Hz), 7.04 (t, 1H, *J* = 7.3 Hz), 7.09 (ddd, 1H, *J*₁ = 7.3, *J*₂ = 4.8, *J*₃ = 0.9 Hz), 7.30 (t, 2H, *J* = 7.7 Hz), 7.59 (d, 2H, *J* = 8.6 Hz), 7.61 (t, 1H, *J* = 7.7 Hz), 8.05 (d, 2H, *J* = 8.1 Hz), 8.05 (d, 1H, *J* = 8.1 Hz), 8.30-8.32 (m, 1H), 8.43 (s, 1H), 8.94 (bs, 1H), 8.95 (bs, 1H), 10.06 (s, 1H), 10.06 (s, 1H). Anal. Calcd for C₂₃H₂₁N₅O₄·3/4H₂O: C, 62.08; H, 5.10; N, 15.74. Found C, 61.94; H, 5.14; N, 15.64. ESI-MS (negative ion mode): Calcd for [C₂₃H₂₀N₅O₄-H⁺]⁻ *m/z* = 430.15. Found *m/z* = 430.3.

Receptor 2c

Into a solution of **4** (0.57 g, 3.8 mmol), isophthalic acid (0.28 g, 1.69 mmol), HOBT (0.51 g, 3.8 mmol)

in DMF (30 ml), was added DCC (0.78 g, 3.8 mmol) in DMF (20 ml) at 0 °C under nitrogen atmosphere. The mixture was stirred at 0 °C for 1.5 h and followed by at r.t. for 12 h. After filtration to remove dicyclohexylurea, the filtrate was evaporated under reduced pressure. The residue was washed with ethyl acetate, and methanol, and recrystallized from DMF-MeOH to give the product as white solid. Yield 0.45 g, 61%. Mp > 246.2-247.0 °C. ¹H NMR (DMSO-*d*₆) δ 4.15 (d, 4H, *J* = 5.9 Hz), 7.09 (dd, 2H, *J*₁ = 7.3, *J*₂ = 5.2 Hz), 7.67 (t, 2H, *J* = 7.7 Hz), 7.77 (dt, *J*₁ = 7.7, *J*₂ = 1.8 Hz), 8.04 (d, 2H, *J* = 7.9 Hz), 8.31 (d, 2H, *J* = 4.8 Hz), 8.41 (s, 1H), 8.94 (t, 2H, *J* = 5.9 Hz), 10.56 (s, 2H). Anal. Calcd for C₂₂H₂₀N₆O₄·H₂O: C, 58.66; H, 4.92; N, 18.66. Found C, 58.50; H, 5.05; N, 18.43. ESI-MS (negative ion mode): Calcd for [C₂₂H₂₀N₆O₄-H⁺]⁻ *m/z* = 431.15. Found *m/z* = 431.5.

1. Cusack, N. J.; Shaw, G.; Logemann, F. I. *J. Chem. Soc., Perkin Trans. 1* **1980**, 2316.

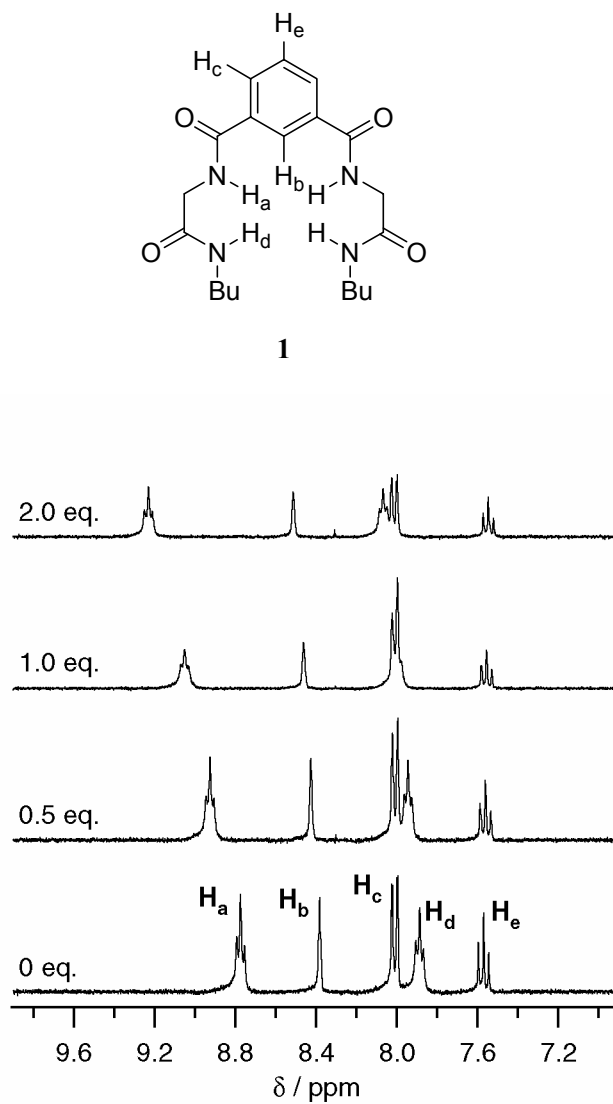


Fig. S1. Partial ^1H NMR spectra of **1** upon the addition of AcO^- in $\text{DMSO}-d_6$ at 298 K [**1**] = 5.0×10^{-3} mol dm^{-3} .

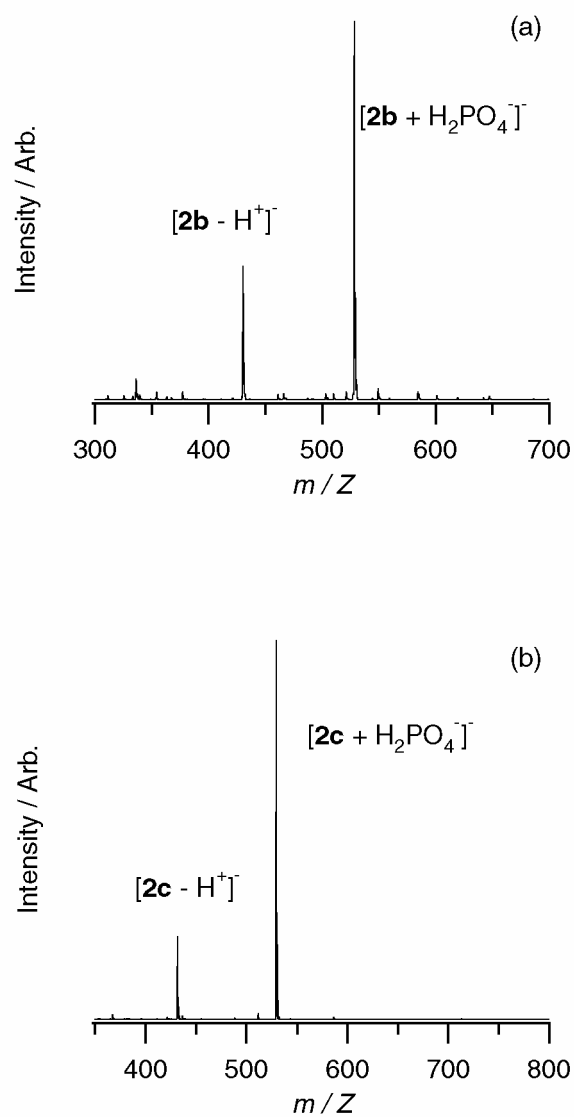


Fig. S2. ESI-MS (negative ion mode) of $2b \cdot H_2PO_4^-$ (a) and $2c \cdot H_2PO_4^-$ (b) in MeCN.

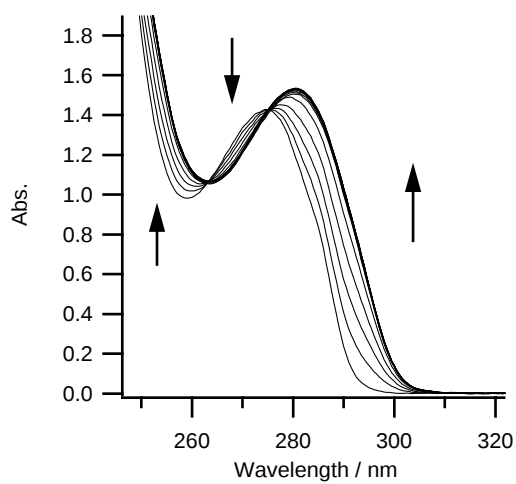


Fig. S3. UV-vis spectral changes of **2c** upon the addition of H_2PO_4^- in 0.5% DMSO-MeCN (v/v) at 298 K. $[\mathbf{2c}] = 1.0 \times 10^{-4} \text{ mol dm}^{-3}$, $[\text{H}_2\text{PO}_4^-] = 0 - 6.1 \times 10^{-4} \text{ mol dm}^{-3}$.

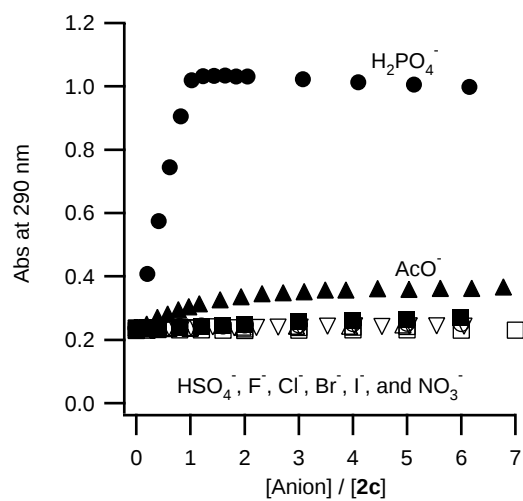


Fig. S4. The plot of absorbance at 290 nm vs. [Anion]/[2c]. [2c] = 1.0×10^{-4} mol dm⁻³ in 0.5% DMSO-MeCN (v/v) at 298 K.