Extraction and complexation of alkali and alkaline earth metal cations by lower-rim calix[4]arene diethylene glycol amide derivatives

ELECTRONIC SUPPLEMENTARY INFORMATION

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Mass spectra were obtained on Agilent 6420 Triple Quadrupole Mass Spectrometer. Solutions were introduced directly via Agilent 1200 HPLC system. For ionization enhancement formic acid (w = 0.1 %, p.a. Kemika) was used. The ESI MS spectra were recorded in the range of m/z 100 to m/z 2000 in positive ion mode. The following parameters were used: capillary potential 4 kV, fragmentor voltage 135 V, gas flow rate 0.6 dm^{-3} min^{-1} and gas temperature 300 °C. Tandem mass spectrometry of protonated molecules was carried out using collision energies of 15–60 eV and nitrogen as a collision gas.

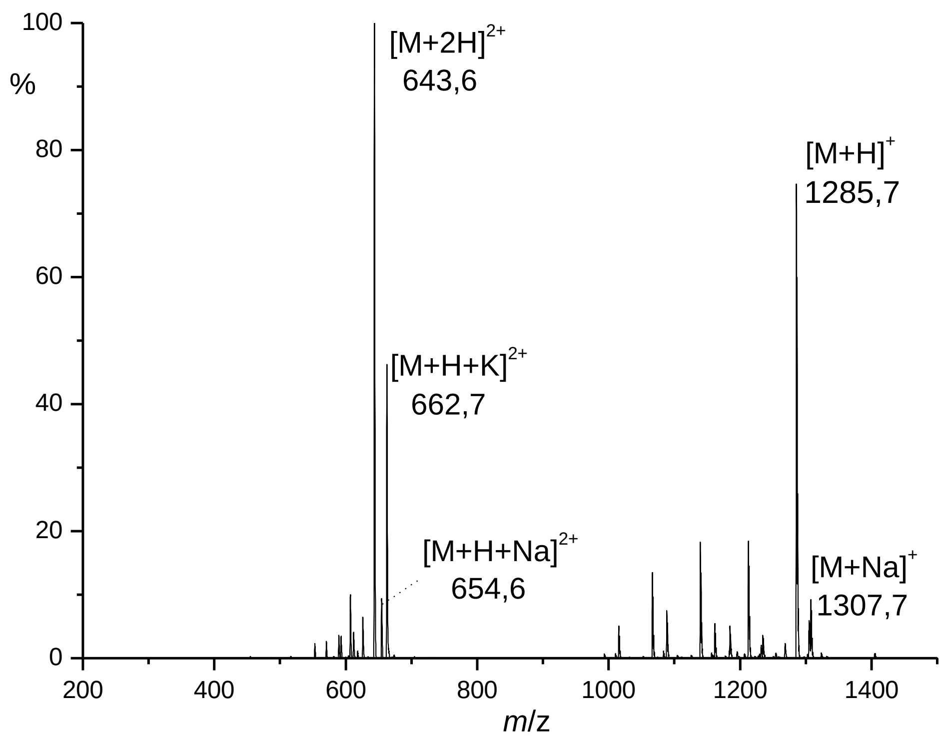


Figure S1. ESI mass spectrum of **1** in acetonitrile; $c(\mathbf{1}) = 2 \times 10^{-3}$ mol dm^{-3}.

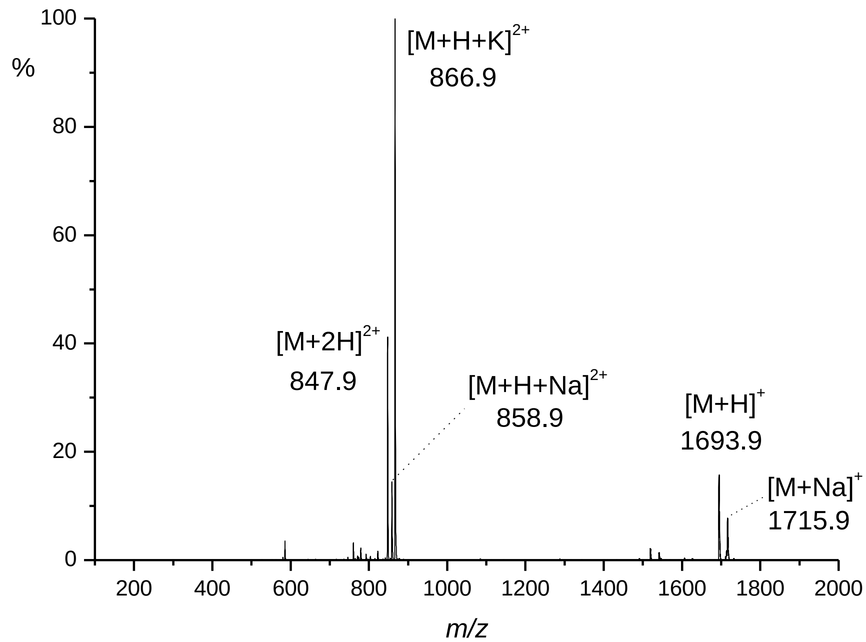


Figure S2. ESI mass spectrum of **2** in acetonitrile; $c(\mathbf{2}) = 2 \times 10^{-3}$ mol dm^{-3}.

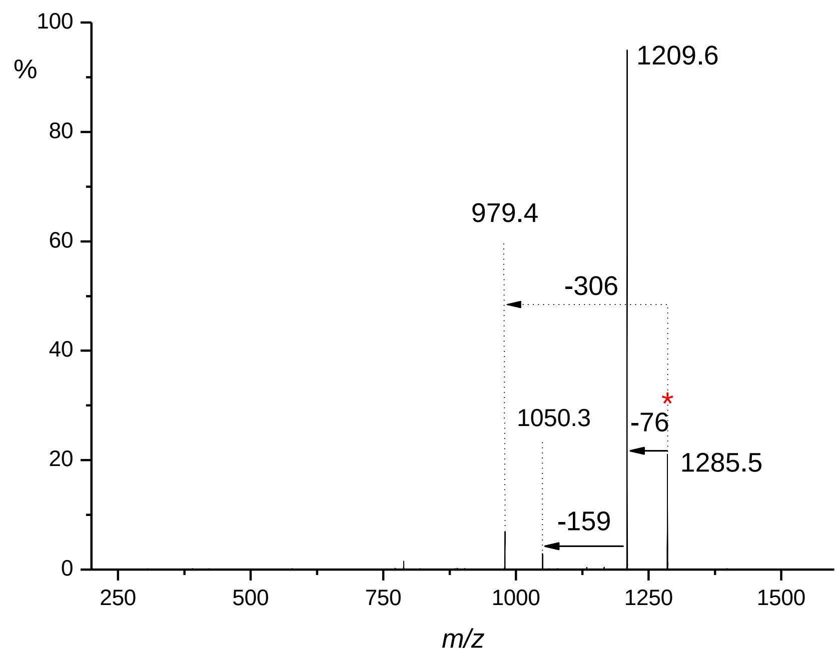


Figure S3. MS/MS spectrum of $[\mathbf{1}+H]^+$. Collision energy = 45 eV.

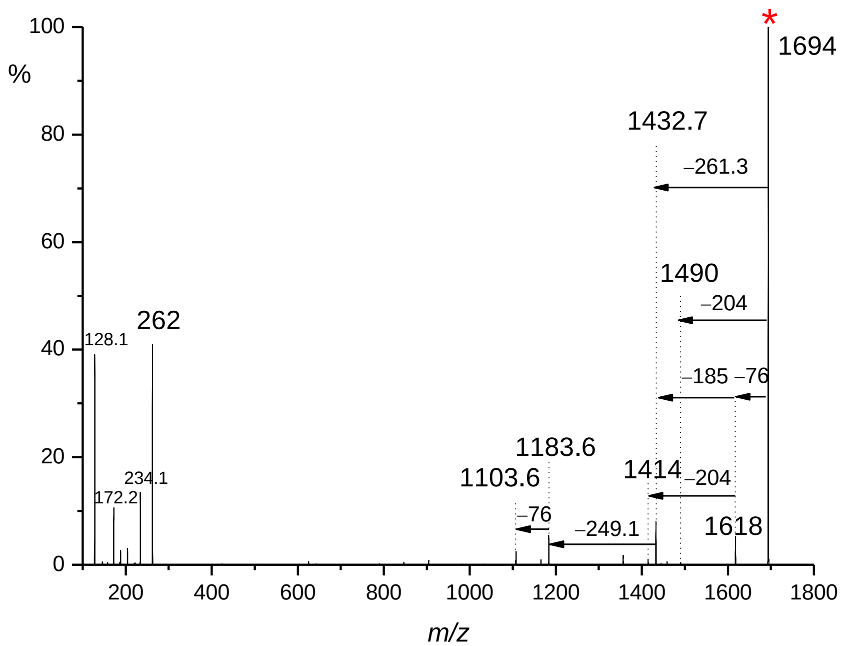
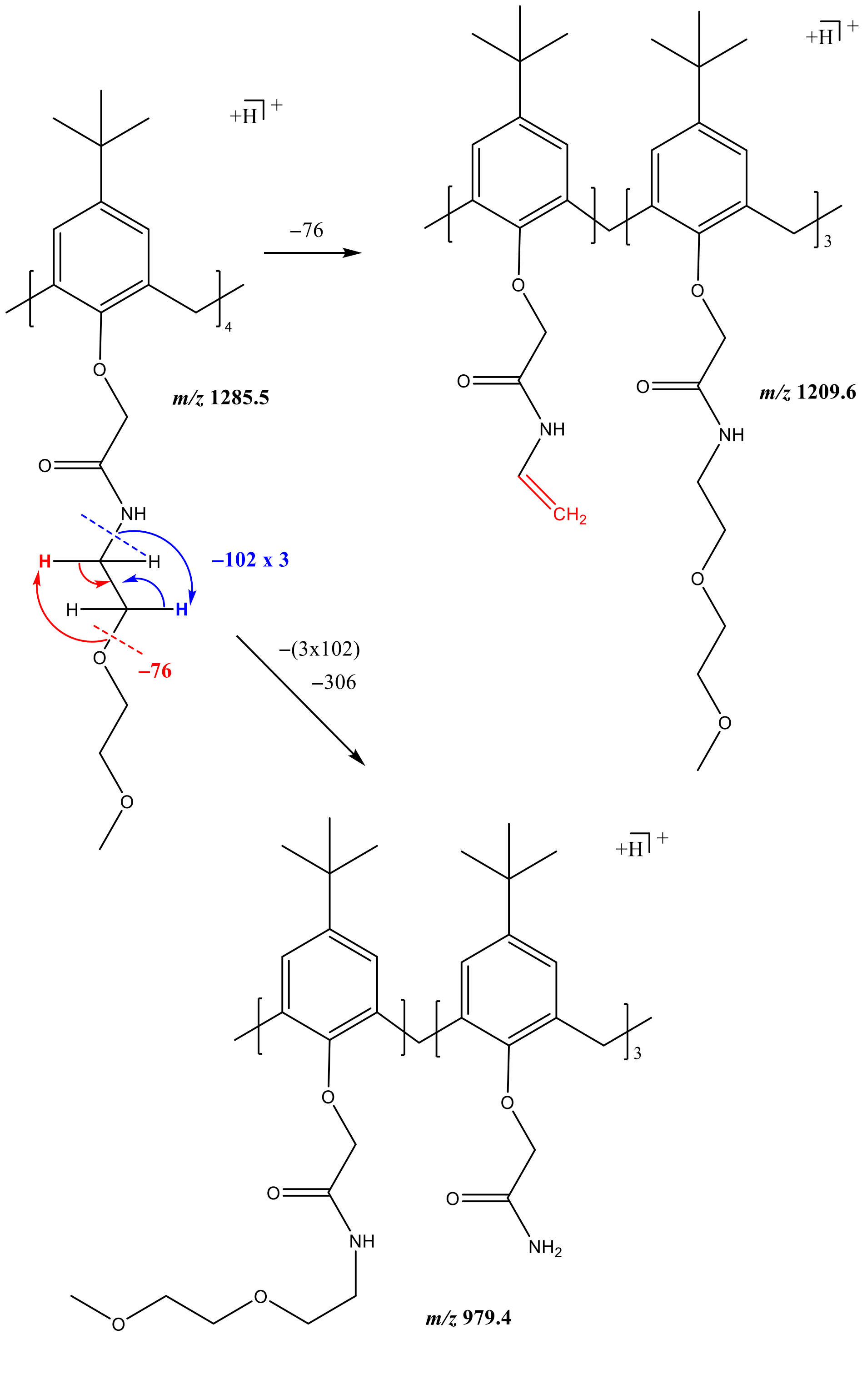
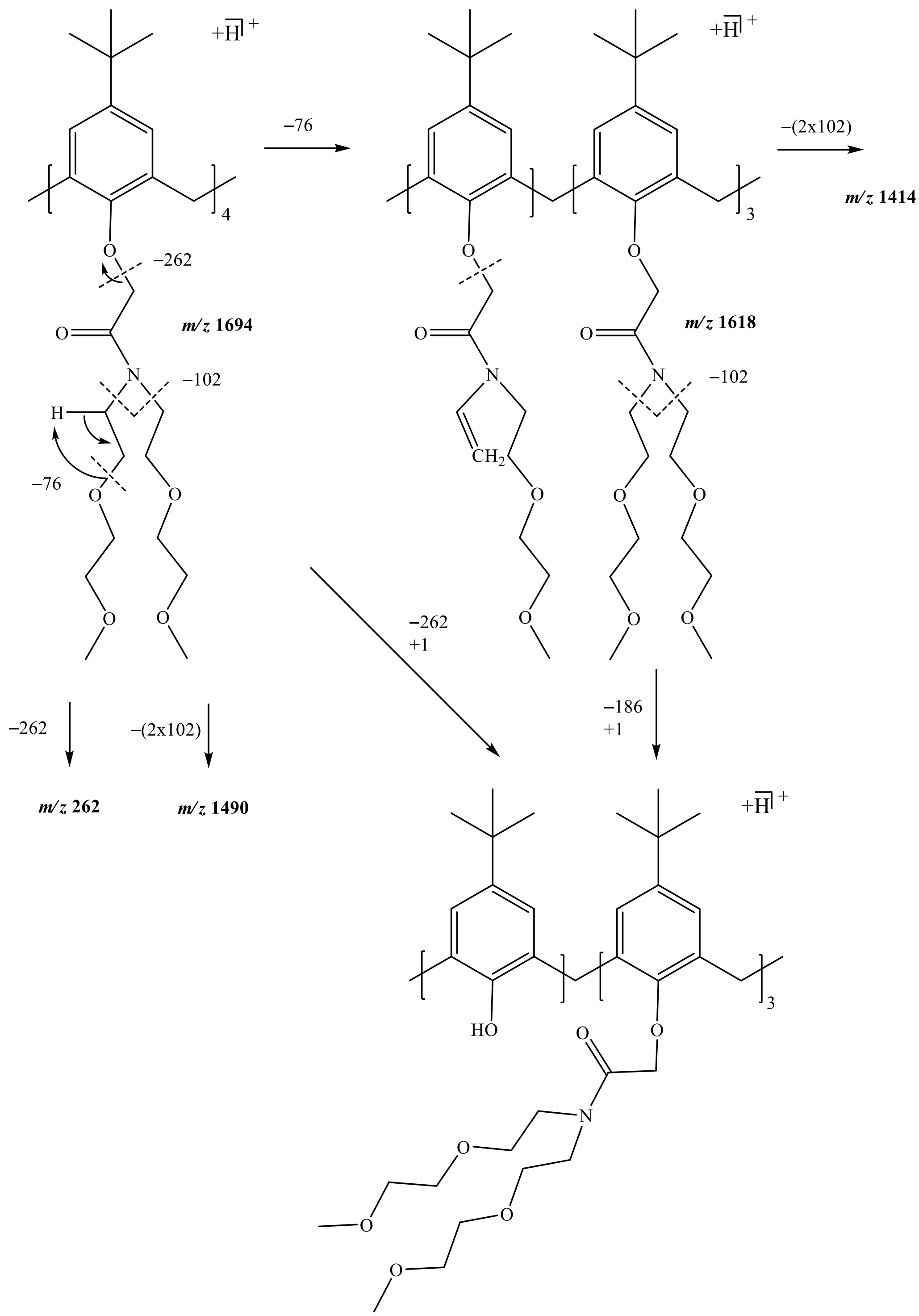


Figure S4. MS/MS spectrum of [**2**+H]$^+$. Collision energy = 55 eV.



Scheme S1. The proposed fragmentation pathway of protonated molecular ion [**1**+H]$^+$.



Scheme S2. The proposed fragmentation pathway of protonated molecular ion [**2**+H]$^{+}$.

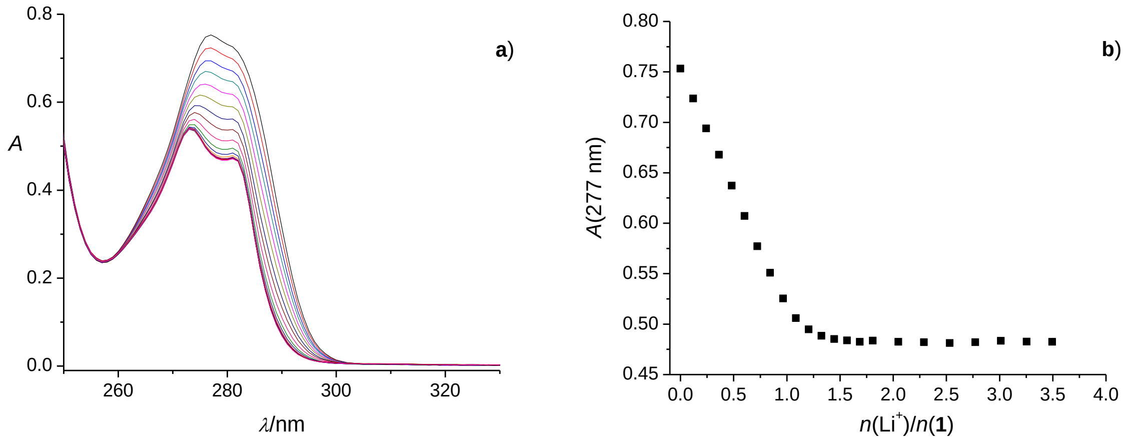


Figure S5. (a) Spectrophotometric titration of **1** (c = 1.89 × 10^{-4} mol dm^{-3}) with $LiClO_4$ (c = 2.30 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Li^+)$ / n(**1**) = 0 (top curve) – 3.49 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 277 nm on $n(Li^+)$ / n(**1**) ratio.

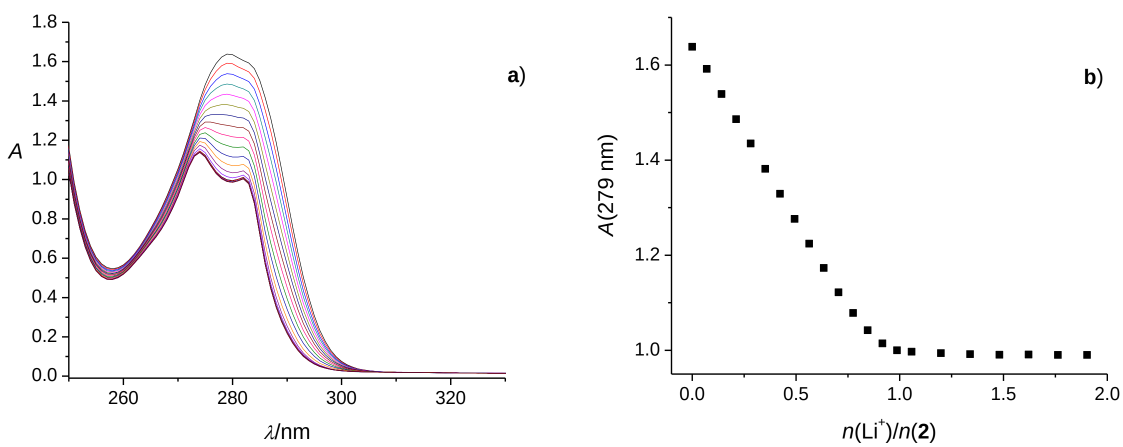


Figure S6. (a) Spectrophotometric titration of **2** (c = 3.24 × 10^{-4} mol dm^{-3}) with $LiClO_4$ (c = 2.28 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Li^+)$ / n(**2**) = 0 (top curve) – 1.90 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 279 nm on $n(Li^+)$ / n(**2**) ratio.

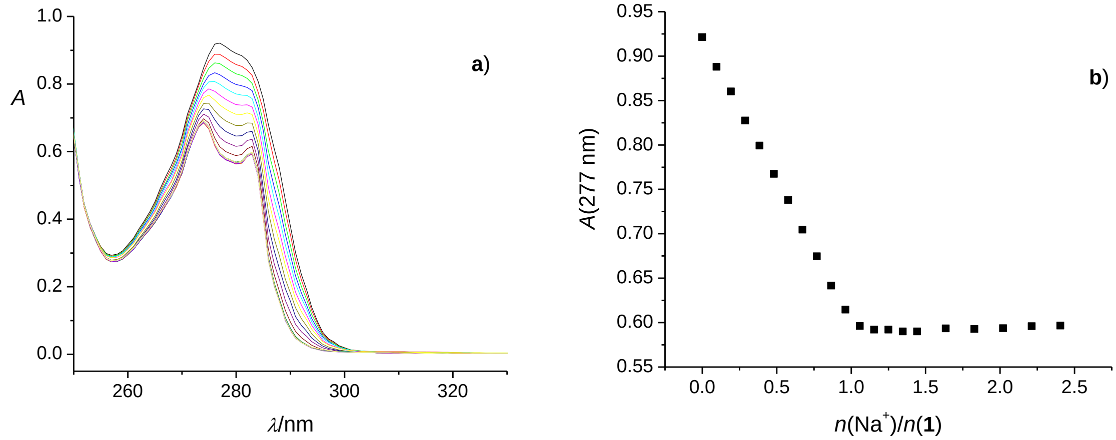


Figure S7. (a) Spectrophotometric titration of **1** (c = 2.30 × 10^{-4} mol dm^{-3}) with $NaClO_4$ (c = 2.15 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Na^+)$ / n(**1**) = 0 (top curve) – 2.-40 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 277 nm on $n(Na^+)$ / n(**1**) ratio.

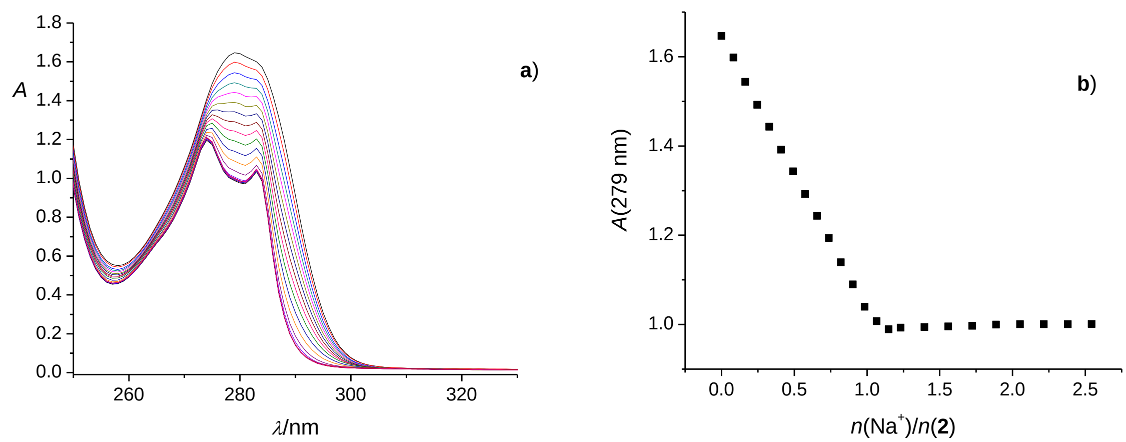


Figure S8. (a) Spectrophotometric titration of **2** (c = 2.70 × 10^{-4} mol dm^{-3}) with $NaClO_4$ (c = 2.2 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Na^+)$ / n(**2**) = 0 (top curve) – 2.54 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 279 nm on $n(Na^+)$ / n(**2**) ratio.

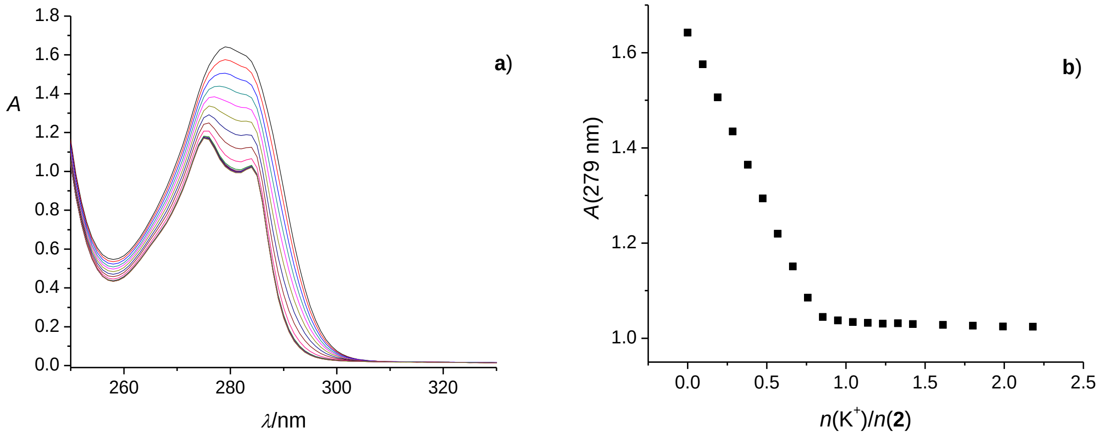


Figure S9. (a) Spectrophotometric titration of **2** (c = 2.26 × 10^{-4} mol dm^{-3}) with $KClO_4$ (c = 2.34 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(K^+)$ / n(**2**) = 0 (top curve) – 2.18 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 279 nm on $n(K^+)$ / n(**2**) ratio.

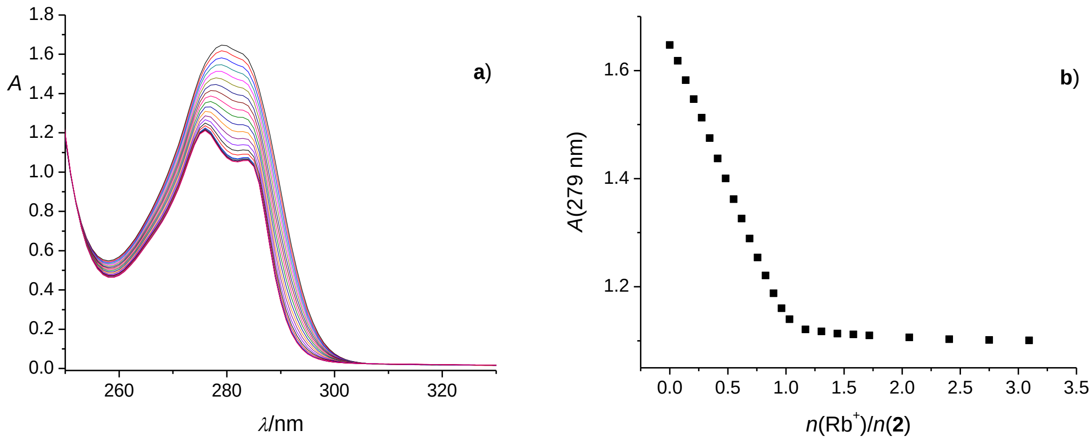


Figure S10. (a) Spectrophotometric titration of **2** (c = 2.70 × 10^{-4} mol dm^{-3}) with $RbNO_3$ (c = 1.86 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Rb^+)$ / n(**2**) = 0 (top curve) – 3.09 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 279 nm on $n(Rb^+)$ / n(**2**) ratio.

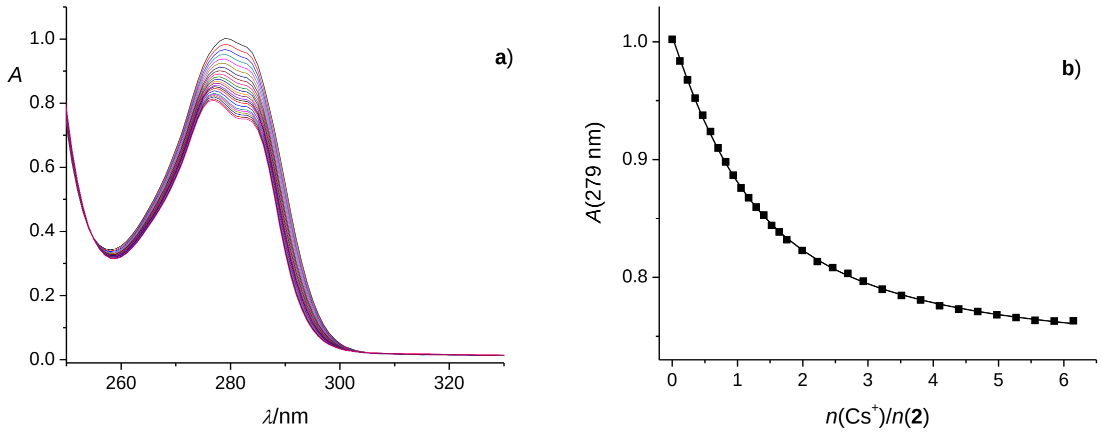


Figure S11. (a) Spectrophotometric titration of **2** (c = 1.62 × 10^{-4} mol dm^{-3}) with $CsNO_3$ (c = 1.90 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Cs^+)$ / n(**2**) = 0 (top curve) – 6.15 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 279 nm on $n(Cs^+)$ / n(**2**) ratio. ■ experimental; – calculated.

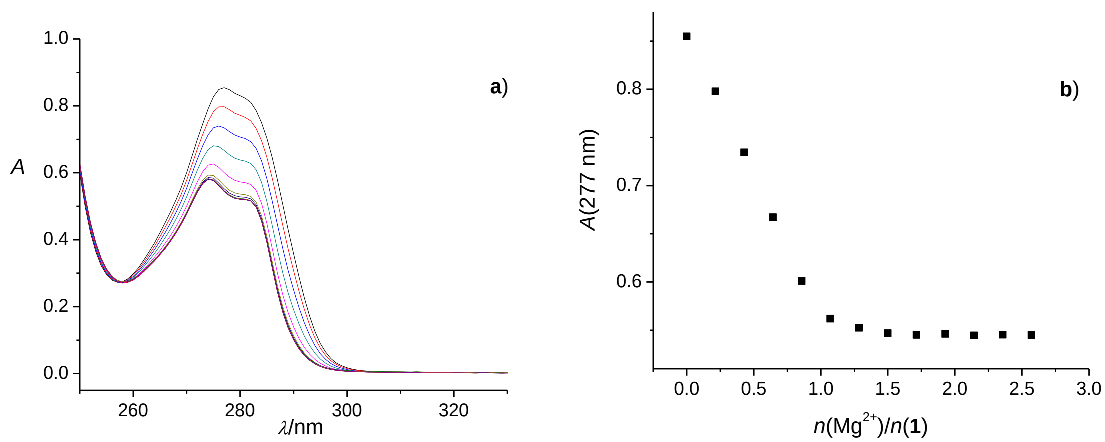


Figure S12. (a) Spectrophotometric titration of **1** (c = 2.17 × 10^{-4} mol dm^{-3}) with $Mg(ClO_4)_2$ (c = 1.86 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Mg^{2+})$ / n(**1**) = 0 (top curve) – 2.57 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 277 nm on $n(Mg^{2+})$ / n(**1**) ratio.

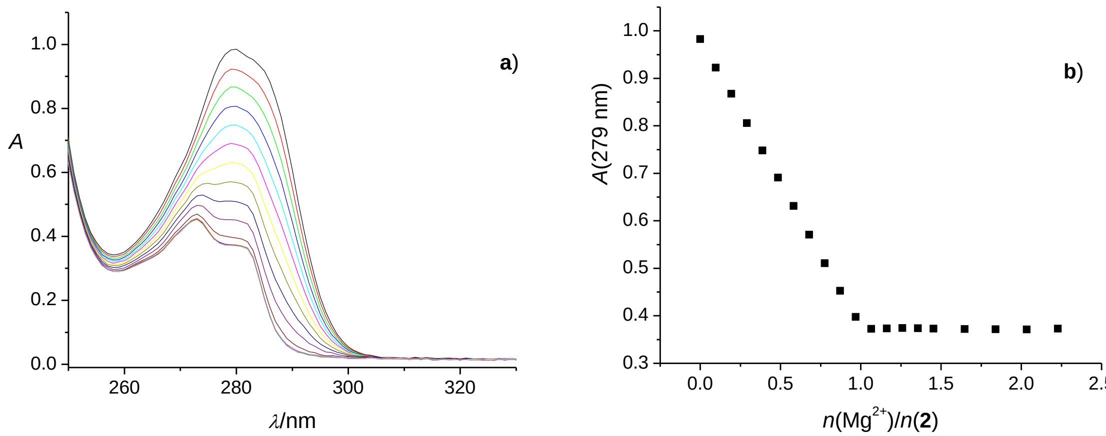


Figure S13. (a) Spectrophotometric titration of **2** (c = 2.06 × 10^{-4} mol dm^{-3}) with $Mg(ClO_4)_2$ (c = 1.99 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Mg^{2+})$ / n(**2**) = 0 (top curve) – 2.23 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 279 nm on $n(Mg^{2+})$ / n(**2**) ratio.

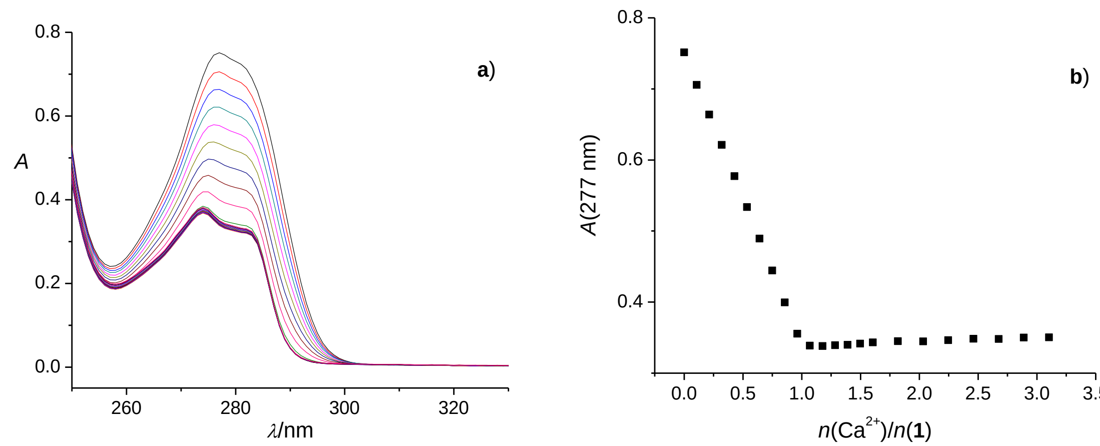


Figure S14. (a) Spectrophotometric titration of **1** (c = 1.89 × 10^{-4} mol dm^{-3}) with $Ca(ClO_4)_2$ (c = 2.03 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Li^{+})$ / n(**1**) = 0 (top curve) – 3.10 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 277 nm on $n(Ca^{2+})$ / n(**1**) ratio.

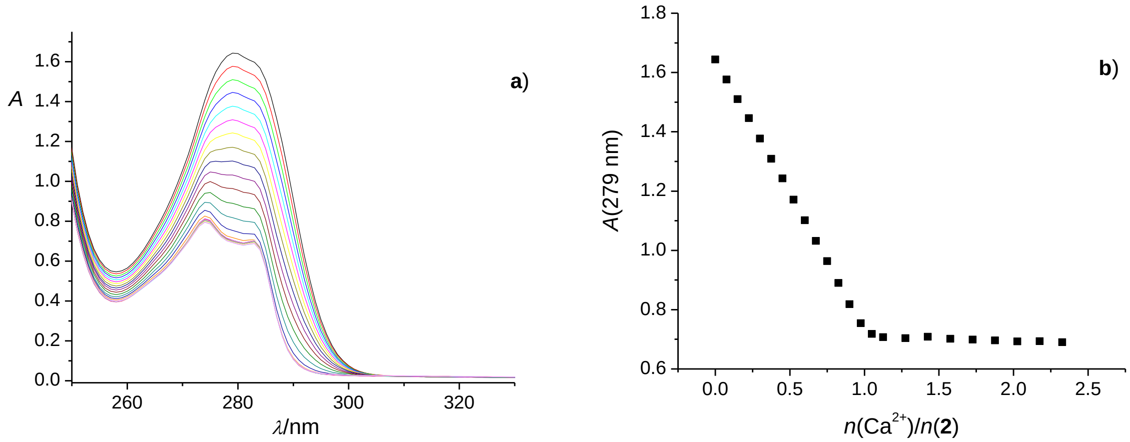


Figure S15. (a) Spectrophotometric titration of **2** (c = 2.70 × 10^{-4} mol dm^{-3}) with $Ca(ClO_4)_2$ (c = 2.06 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Ca^{2+})$ / n(**2**) = 0 (top curve) – 2.33 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 279 nm on $n(Ca^{2+})$ / n(**2**) ratio.

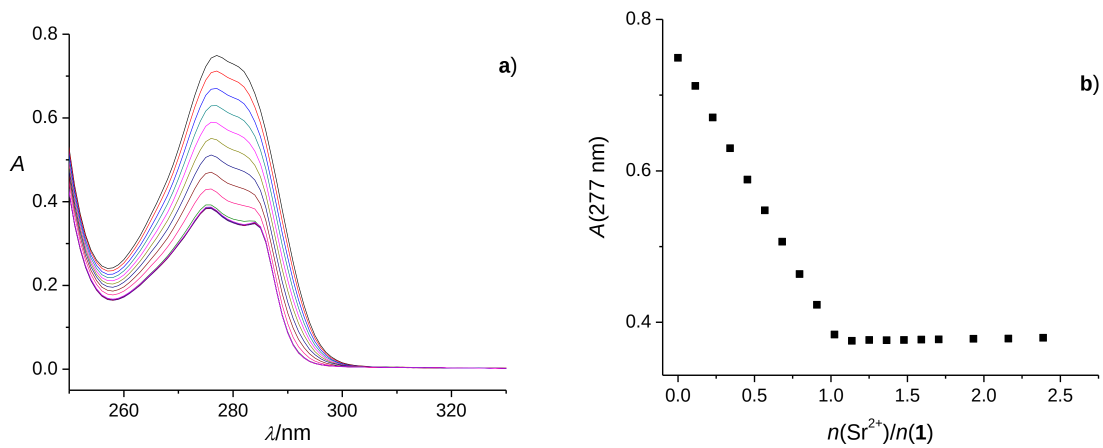


Figure S16. (a) Spectrophotometric titration of **1** (c = 1.89 × 10^{-4} mol dm^{-3}) with $Sr(ClO_4)_2$ (c = 2.15 × 10^{-3} mol dm^{-3}) in acetonitrile. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Sr^{2+})$ / n(**1**) = 0 (top curve) – 2.39 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 277 nm on $n(Sr^{2+})$ / n(**1**) ratio.

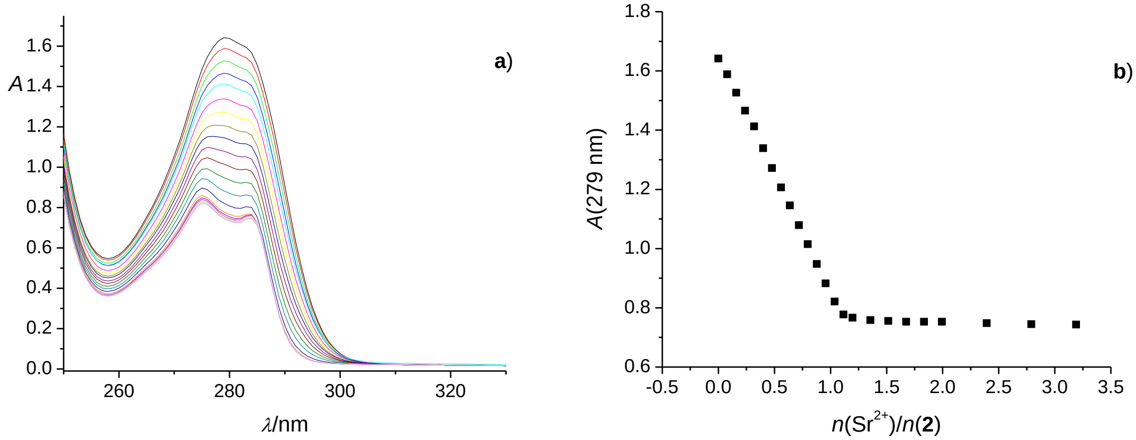


Figure S17. (a) Spectrophotometric titration of **2** ($c = 2.70 \times 10^{-4}$ mol dm^{-3}) with $Sr(ClO_4)_2$ ($c = 2.15 \times 10^{-3}$ mol dm^{-3}) in acetonitrile. $l = 1$ cm; $t = (25.0 \pm 0.1)$ °C; $n(Sr^{2+}) / n(\mathbf{2}) = 0$ (top curve) – 3.19 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 279 nm on $n(Sr^{2+}) / n(\mathbf{2})$ ratio.

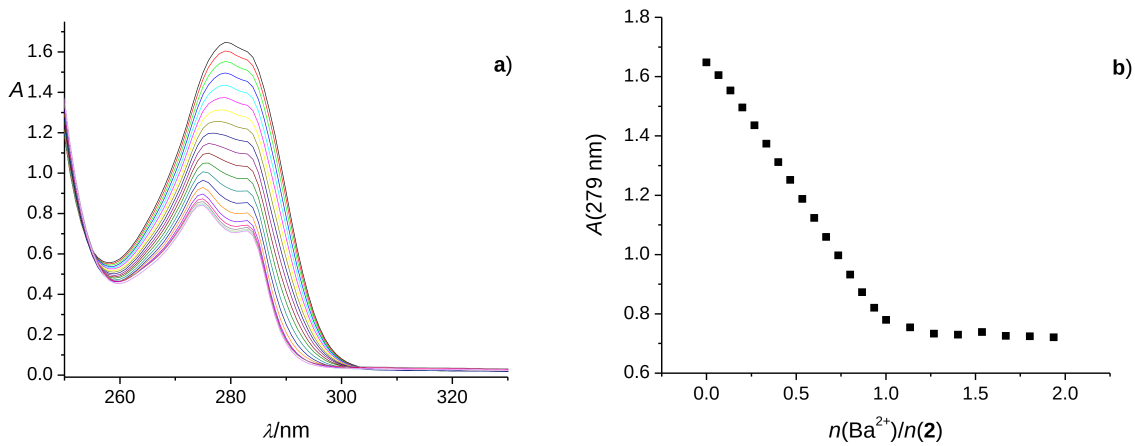


Figure S18. (a) Spectrophotometric titration of **2** ($c = 3.24 \times 10^{-4}$ mol dm^{-3}) with $Ba(ClO_4)_2$ ($c = 1.80 \times 10^{-3}$ mol dm^{-3}) in acetonitrile. $l = 1$ cm; $t = (25.0 \pm 0.1)$ °C; $n(Ba^{2+}) / n(\mathbf{2}) = 0$ (top curve) – 1.94 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 279 nm on $n(Ba^{2+}) / n(\mathbf{2})$ ratio.

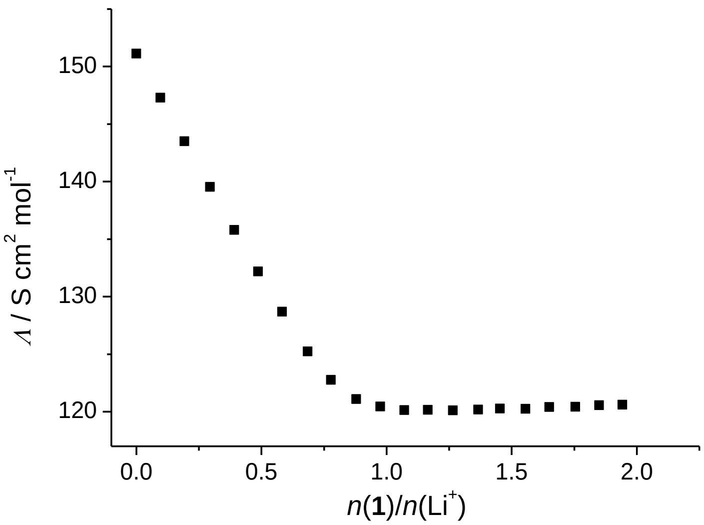


Figure S19. Conductometric titration of $LiClO_4$ (c = 1.78 × 10^{-4} mol dm^{-3}) with **1** (c = 1.90 × 10^{-3} mol dm^{-3}) in acetonitrile; t = (25.0 ± 0.1) °C.

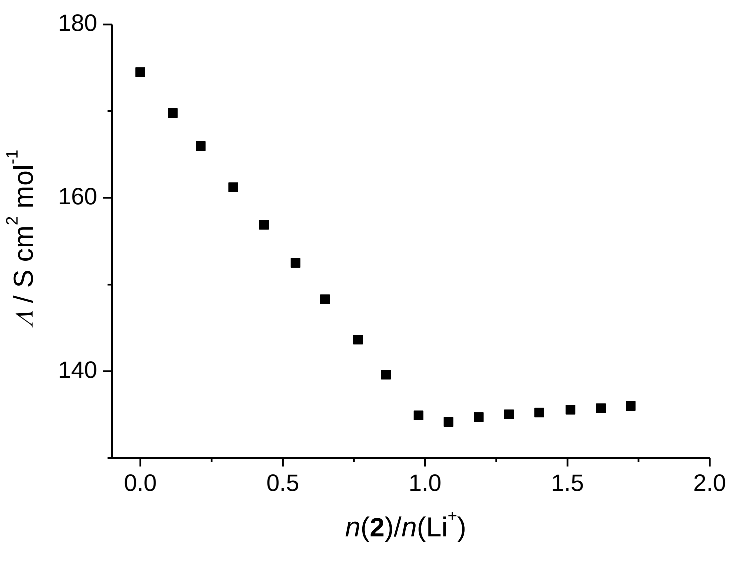


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Figure S20. Conductometric titration of $LiClO_4$ (c = 1.09 × 10^{-4} mol dm^{-3}) with **2** (c = 1.23 × 10^{-3} mol dm^{-3}) in acetonitrile; t = (25.0 ± 0.1) °C.

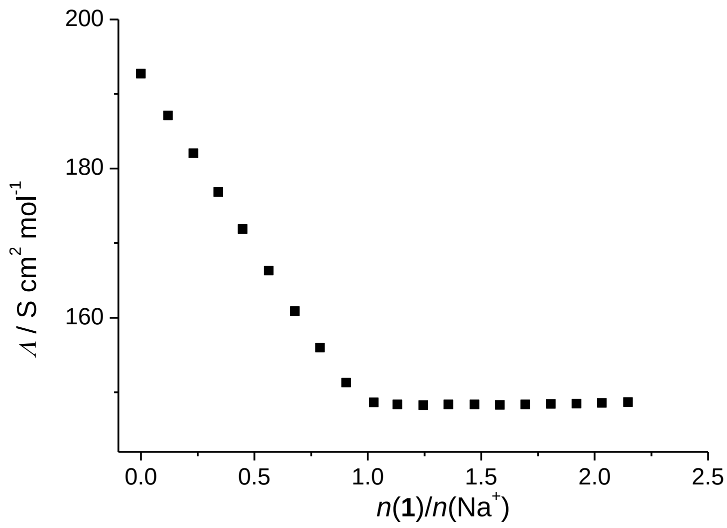

Figure S21. Conductometric titration of $NaClO_4$ ($c = 1.53 \times 10^{-4}$ mol dm^{-3}) with **1** ($c = 1.90 \times 10^{-3}$ mol dm^{-3}) in acetonitrile; $t = (25.0 \pm 0.1)$ °C.

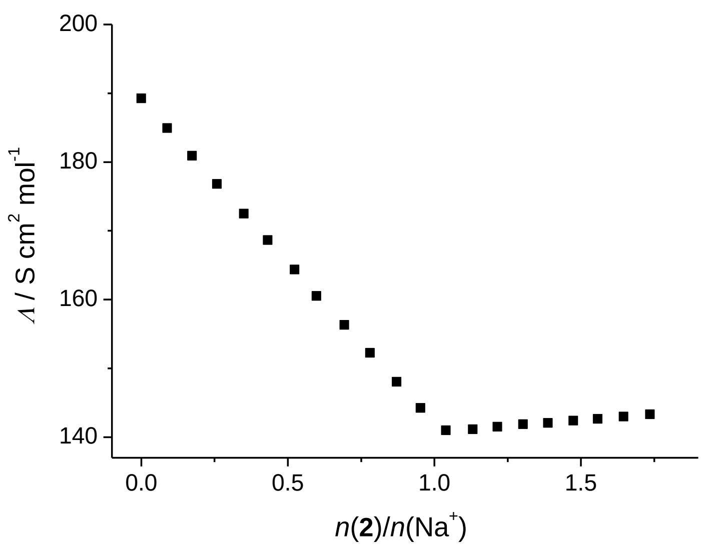

Figure S22. Conductometric titration of $NaClO_4$ ($c = 6.33 \times 10^{-5}$ mol dm^{-3}) with **2** ($c = 9.96 \times 10^{-4}$ mol dm^{-3}) in acetonitrile; $t = (25.0 \pm 0.1)$ °C.

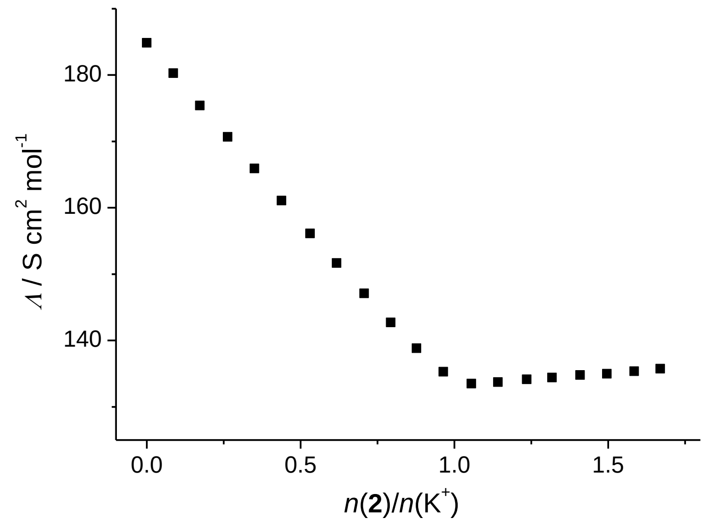

Figure S23. Conductometric titration of $KClO_4$ (c = 1.03 × 10^{-4} mol dm^{-3}) with **2** (c = 9.57 × 10^{-4} mol dm^{-3}) in acetonitrile; t = (25.0 ± 0.1) °C.

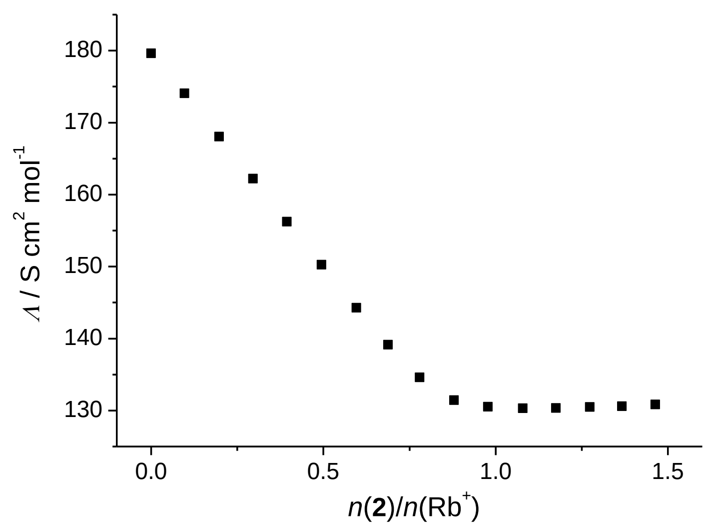

Figure S24. Conductometric titration of $RbClO_4$ (c = 9.43 × 10^{-5} mol dm^{-3}) with **2** (c = 9.69 × 10^{-4} mol dm^{-3}) in acetonitrile; t = (25.0 ± 0.1) °C.

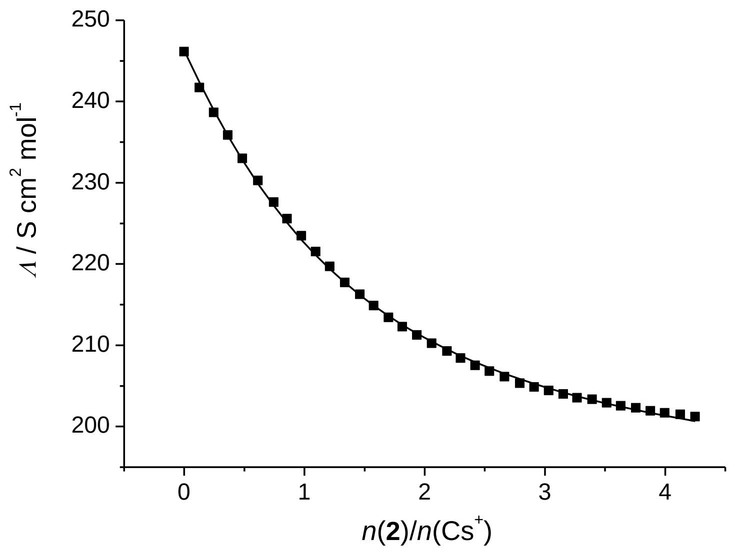

Figure S25. Conductometric titration of $CsNO_3$ (c = 9.59 × 10^{-5} mol dm^{-3}) with **2** (c = 1.39 × 10^{-3} mol dm^{-3}) in acetonitrile; t = (25.0 ± 0.1) °C. ■ experimental; – calculated.

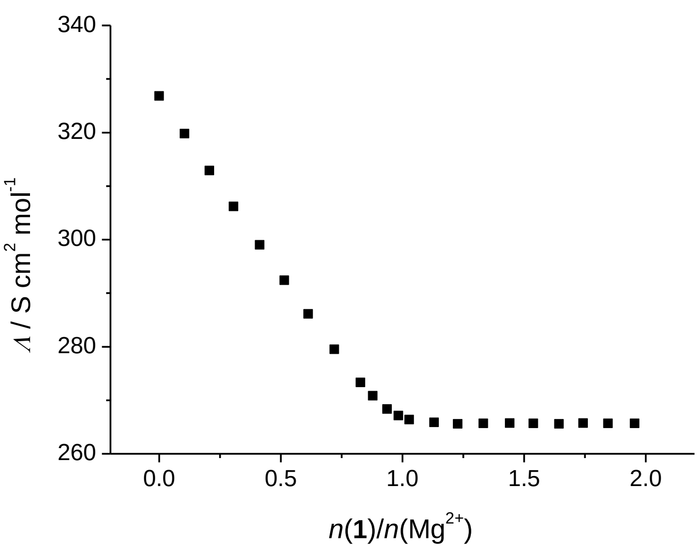

Figure S26. Conductometric titration of $Mg(ClO_4)_2$ (c = 1.81 × 10^{-4} mol dm^{-3}) with **1** (c = 1.90 × 10^{-3} mol dm^{-3}) in acetonitrile; t = (25.0 ± 0.1) °C.

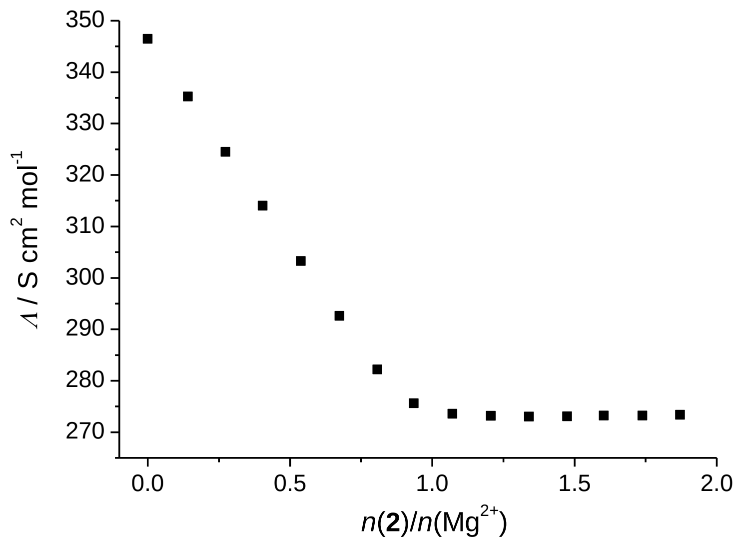

Figure S27. Conductometric titration of $Mg(ClO_4)_2$ (c = 9.58 × 10^{-5} mol dm^{-3}) with **2** (c = 1.33 × 10^{-3} mol dm^{-3}) in acetonitrile; t = (25.0 ± 0.1) °C.

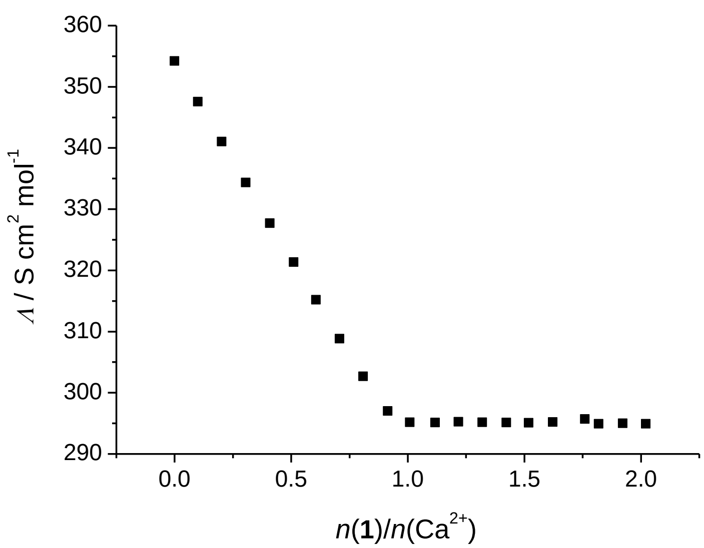

Figure S28. Conductometric titration of $Ca(ClO_4)_2$ (c = 1.81 × 10^{-4} mol dm^{-3}) with **1** (c = 1.90 × 10^{-3} mol dm^{-3}) in acetonitrile; t = (25.0 ± 0.1) °C.

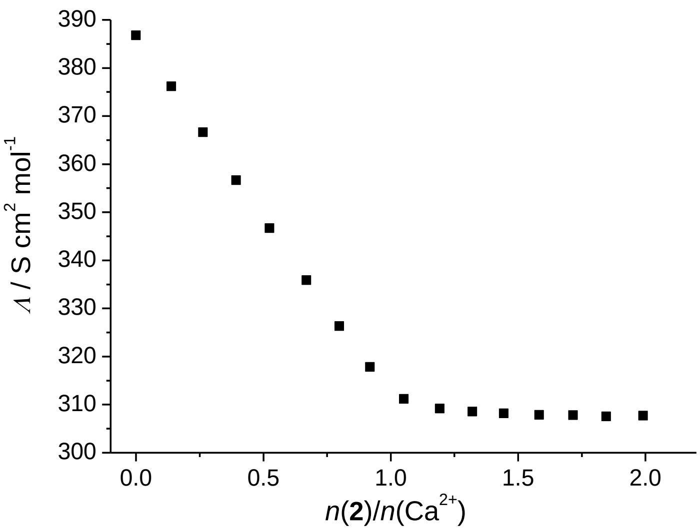

Figure S29. Conductometric titration of $Ca(ClO_4)_2$ (c = 9.62 × 10^{-5} mol dm^{-3}) with **2** (c = 1.33 × 10^{-3} mol dm^{-3}) in acetonitrile; t = (25.0 ± 0.1) °C.

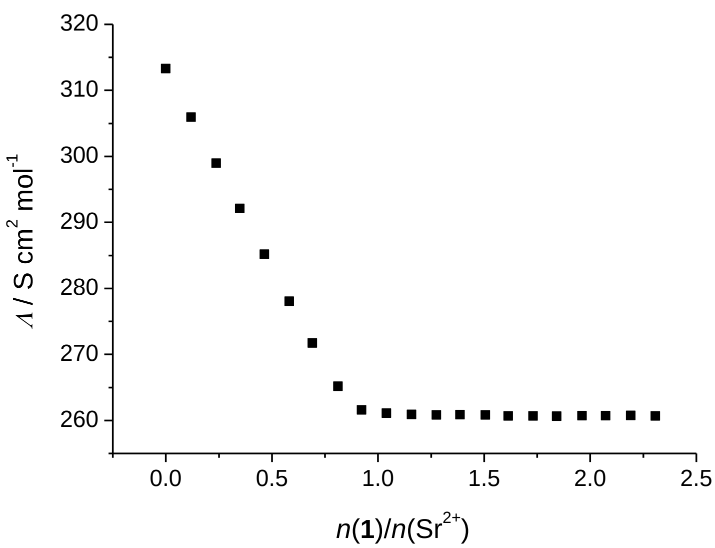

Figure S30. Conductometric titration of $Sr(ClO_4)_2$ (c = 1.61 × 10^{-4} mol dm^{-3}) with **1** (c = 1.90 × 10^{-3} mol dm^{-3}) in acetonitrile; t = (25.0 ± 0.1) °C.

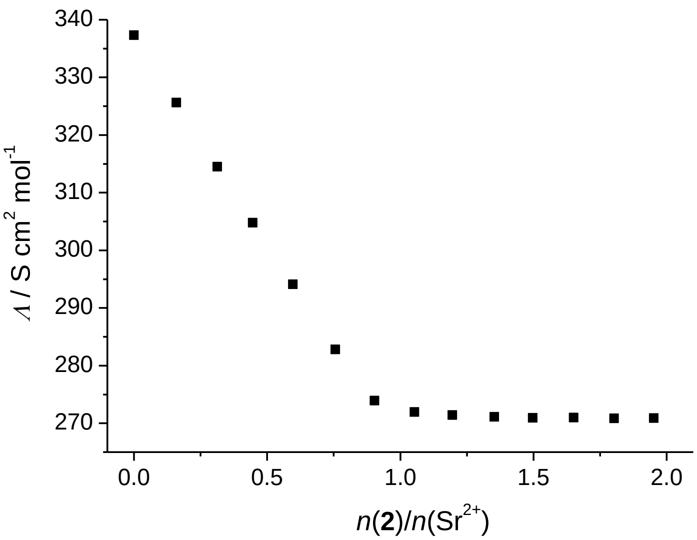

Figure S31. Conductometric titration of $Sr(ClO_4)_2$ ($c = 8.43 \times 10^{-5}$ mol dm^{-3}) with **2** ($c = 1.33 \times 10^{-3}$ mol dm^{-3}) in acetonitrile; $t = (25.0 \pm 0.1)$ °C.

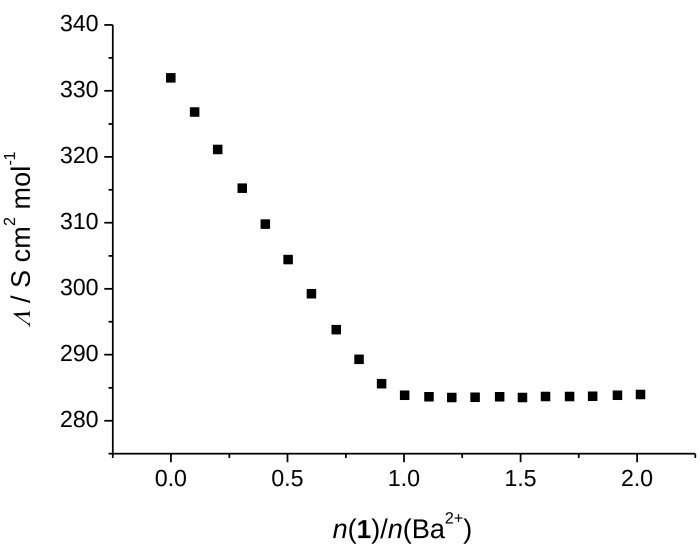

Figure S32. Conductometric titration of $Ba(ClO_4)_2$ ($c = 1.72 \times 10^{-4}$ mol dm^{-3}) with **1** ($c = 1.90 \times 10^{-3}$ mol dm^{-3}) in acetonitrile; $t = (25.0 \pm 0.1)$ °C.

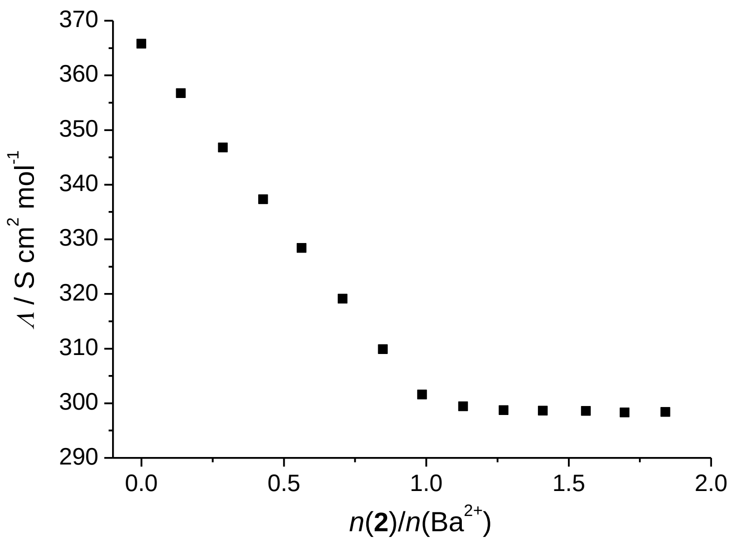

Figure S33. Conductometric titration of $Ba(ClO_4)_2$ (c = 8.99 × 10^{-5} mol dm^{-3}) with **2** (c = 1.33 × 10^{-3} mol dm^{-3}) in acetonitrile; t = (25.0 ± 0.1) °C.

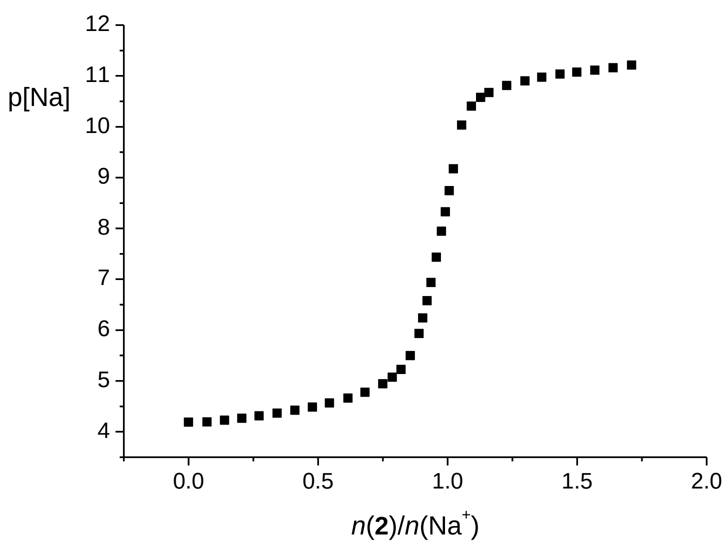

Figure S34. Potentiometric titration of $NaClO_4$ (c = 6.89 × 10^{-5} mol dm^{-3}) with **2** (c = 7.08 × 10^{-4} mol dm^{-3}) in acetonitrile. $V_0(NaClO_4)$ = 30.3 cm^3 I_c = 0.01 mol dm^{-3} ($(C_2H_5)_4NClO_4$); t = (25.0 ± 0.1) °C.

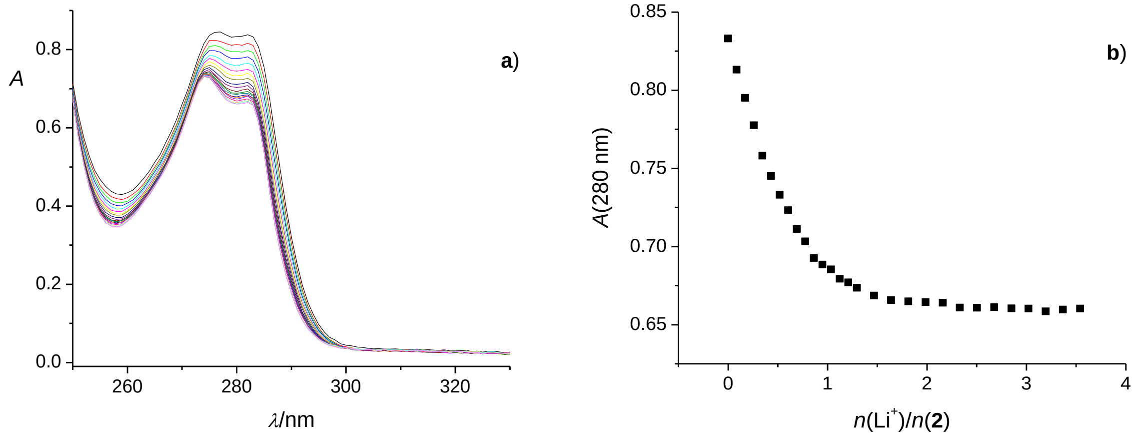

Figure S35. (a) Spectrophotometric titration of **2** (c = 2.28 × 10^{-4} mol dm^{-3}) with $LiClO_4$ (c = 1.97 × 10^{-3} mol dm^{-3}) in methanol. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Li^+)$ / n(**2**) = 0 (top curve) – 2.68 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 280 nm on $n(Li^+)$ / n(**2**) ratio.

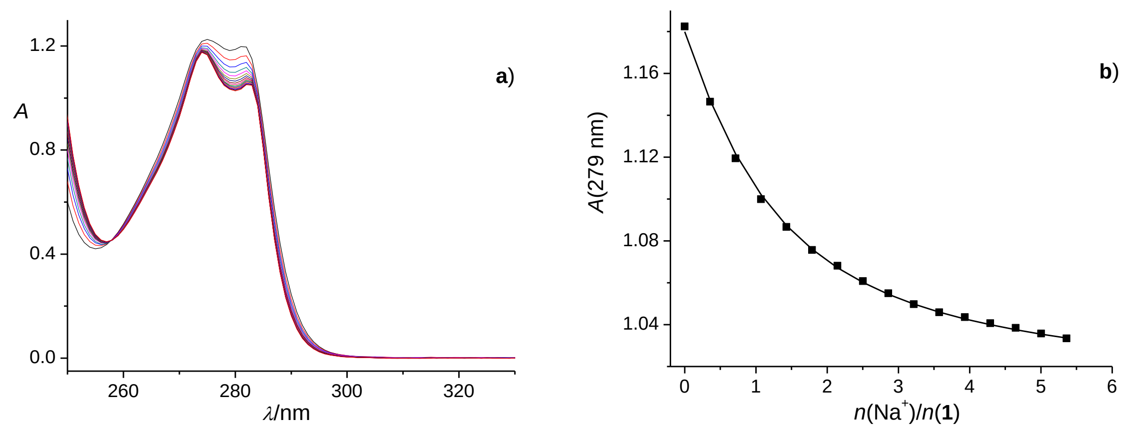

Figure S36. (a) Spectrophotometric titration of **1** (c = 2.22 × 10^{-4} mol dm^{-3}) with $NaClO_4$ (c = 3.17 × 10^{-3} mol dm^{-3}) in methanol. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Na^+)$ / n(**1**) = 0 (top curve) – 5.36 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 279 nm on $n(Na^+)$ / n(**1**) ratio. ■ experimental; – calculated.

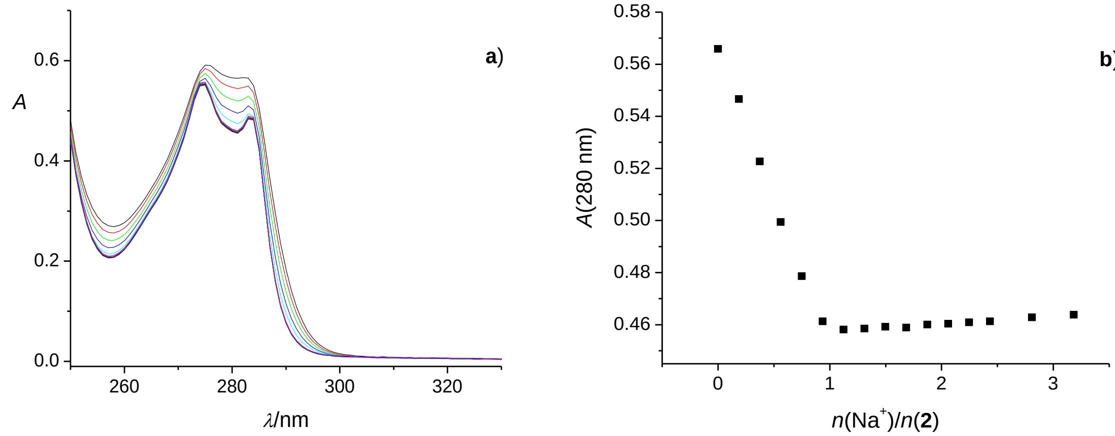

Figure S37. (a) Spectrophotometric titration of **2** ($c = 1.28 \times 10^{-4}$ mol dm^{-3}) with $NaClO_4$ ($c = 2.39 \times 10^{-3}$ mol dm^{-3}) in methanol. $l = 1$ cm; $t = (25.0 \pm 0.1)$ °C; $n(Na^+)$ / n(**2**) = 0 (top curve) – 3.18 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 280 nm on $n(Na^+)$ / n(**2**) ratio.

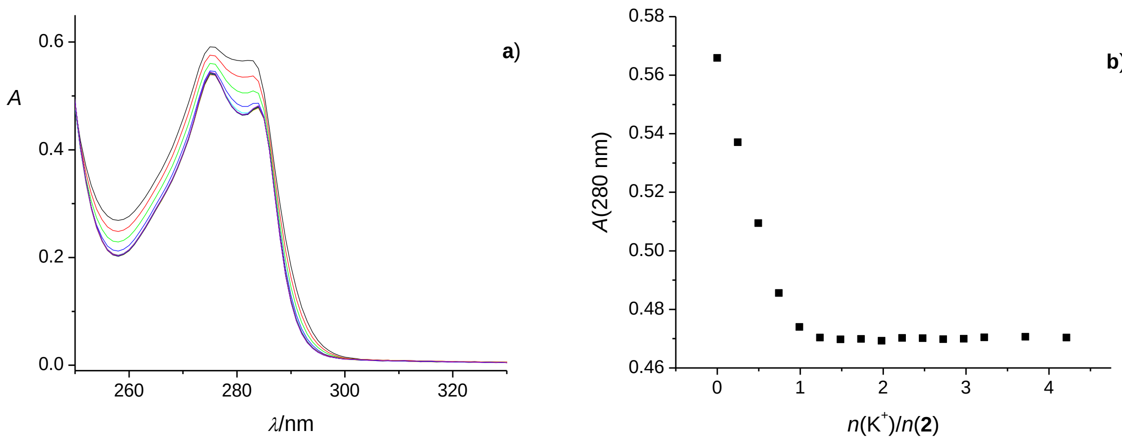

Figure S38. (a) Spectrophotometric titration of **2** ($c = 1.28 \times 10^{-4}$ mol dm^{-3}) with $KClO_4$ ($c = 3.16 \times 10^{-3}$ mol dm^{-3}) in methanol. $l = 1$ cm; $t = (25.0 \pm 0.1)$ °C; $n(K^+)$ / n(**2**) = 0 (top curve) – 4.21 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 280 nm on $n(K^+)$ / n(**2**) ratio.

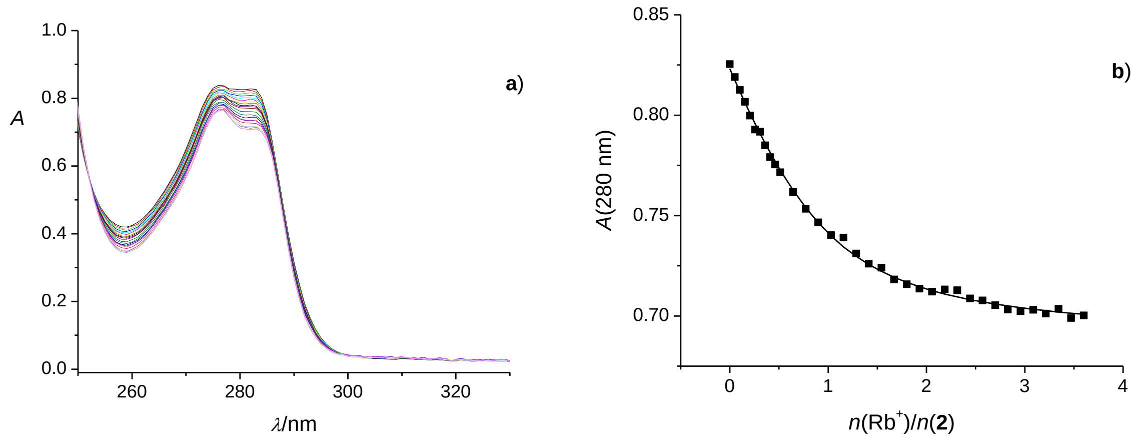

Figure S39. (a) Spectrophotometric titration of **2** (c = 2.28 × 10^{-4} mol dm^{-3}) with $RbNO_3$ (c = 1.17 × 10^{-3} mol dm^{-3}) in methanol. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Rb^+)$ / n(**2**) = 0 (top curve) – 3.60 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 280 nm on $n(Rb^+)$ / n(**2**) ratio. ■ experimental; – calculated.

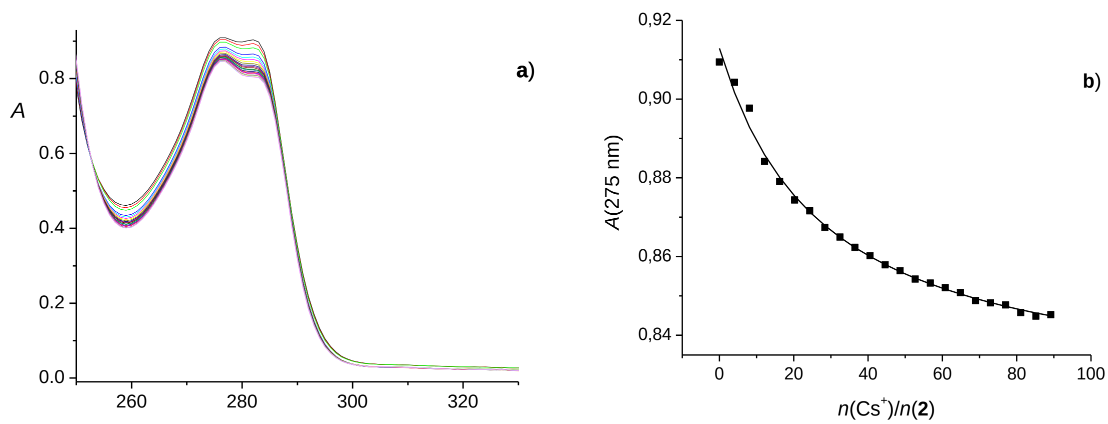

Figure S40. (a) Spectrophotometric titration of **2** (c = 2.44 × 10^{-4} mol dm^{-3}) with $CsNO_3$ (c = 3.95 × 10^{-2} mol dm^{-3}) in methanol. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Rb^+)$ / n(**2**) = 0 (top curve) – 89.2 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 275 nm on $n(Cs^+)$ / n(**2**) ratio. ■ experimental; – calculated.

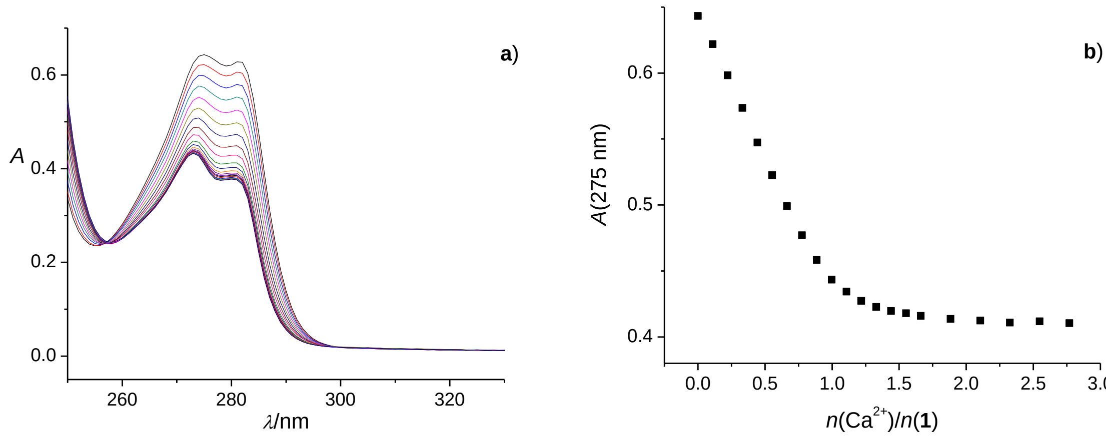

Figure S41. (a) Spectrophotometric titration of **1** ($c = 1.95 \times 10^{-4}$ mol dm^{-3}) with $Ca(ClO_4)_2$ ($c = 2.16 \times 10^{-3}$ mol dm^{-3}) in methanol. $l = 1$ cm; $t = (25.0 \pm 0.1)$ °C; $n(Ca^{2+})$ / n(**1**) = 0 (top curve) – 2.77 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 275 nm on $n(Ca^{2+})$ / n(**1**) ratio.

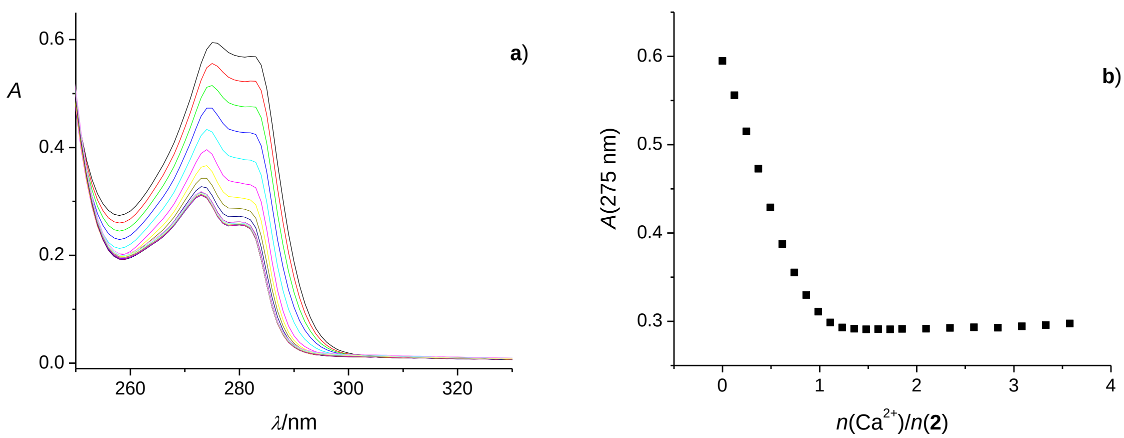

Figure S42. (a) Spectrophotometric titration of **2** ($c = 1.75 \times 10^{-4}$ mol dm^{-3}) with $Ca(ClO_4)_2$ ($c = 2.16 \times 10^{-3}$ mol dm^{-3}) in methanol. $l = 1$ cm; $t = (25.0 \pm 0.1)$ °C; $n(Ca^{2+})$ / n(**2**) = 0 (top curve) – 3.58 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 275 nm on $n(Ca^{2+})$ / n(**2**) ratio.

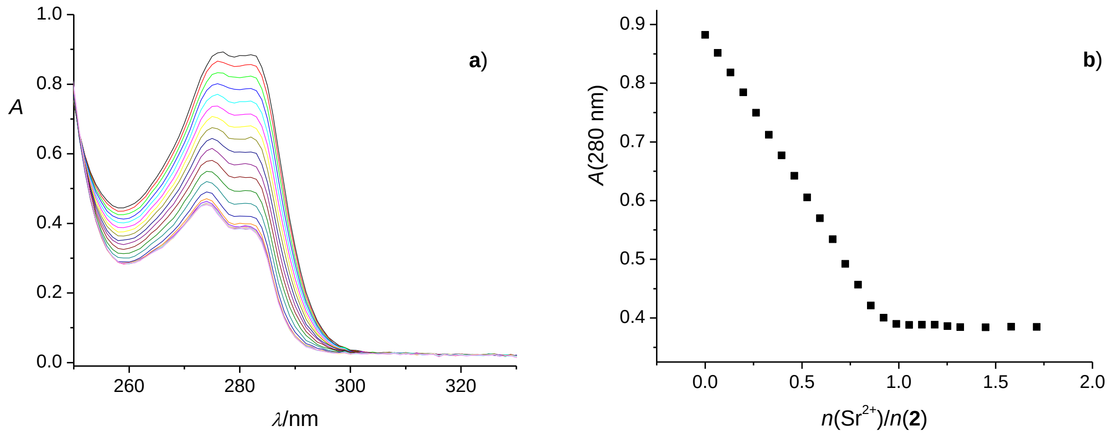

Figure S43. (a) Spectrophotometric titration of **2** (c = 1.78 × 10^{-4} mol dm^{-3}) with $Sr(ClO_4)_2$ (c = 2.20 × 10^{-3} mol dm^{-3}) in methanol. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Sr^{2+})$ / n(**2**) = 0 (top curve) – 1.71 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 280 nm on $n(Sr^{2+})$ / n(**2**) ratio.

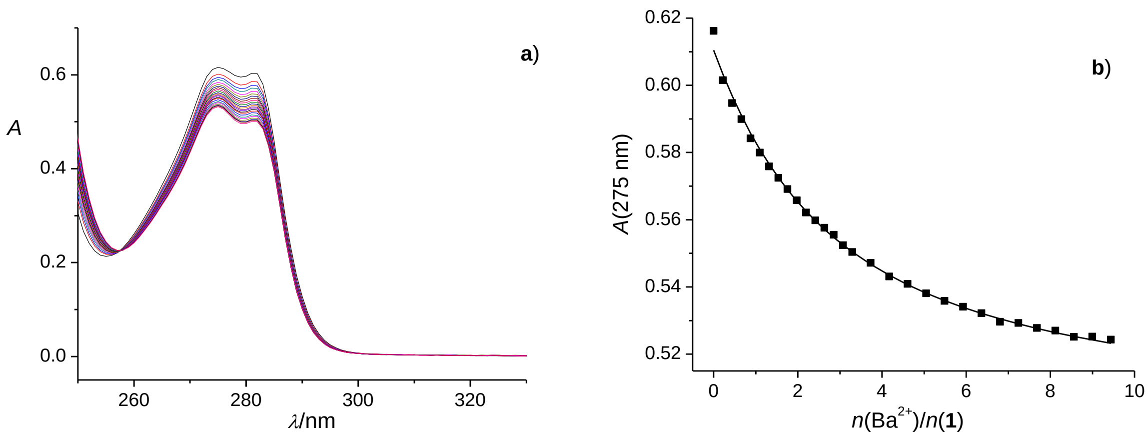

Figure S44. (a) Spectrophotometric titration of **1** (c = 2.06 × 10^{-4} mol dm^{-3}) with $Ba(ClO_4)_2$ (c = 4.52 × 10^{-3} mol dm^{-3}) in methanol. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Ba^{2+})$ / n(**1**) = 0 (top curve) – 9.44 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 275 nm on $n(Ba^{2+})$ / n(**1**) ratio. ■ experimental; – calculated.

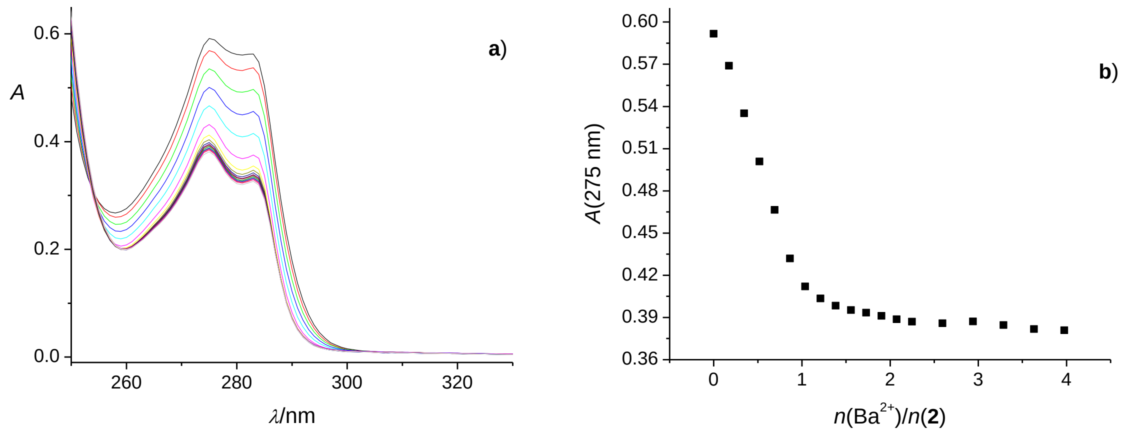

Figure S45. (a) Spectrophotometric titration of **2** (c = 1.28 × 10^{-4} mol dm^{-3}) with $Ba(ClO_4)_2$ (c = 2.21 × 10^{-3} mol dm^{-3}) in methanol. l = 1 cm; t = (25.0 ± 0.1) °C; $n(Ba^{2+})$ / n(**2**) = 0 (top curve) – 3.98 (bottom curve); the spectra are corrected for dilution. (b) Dependence of absorbance at 275 nm on $n(Ba^{2+})$ / n(**2**) ratio.

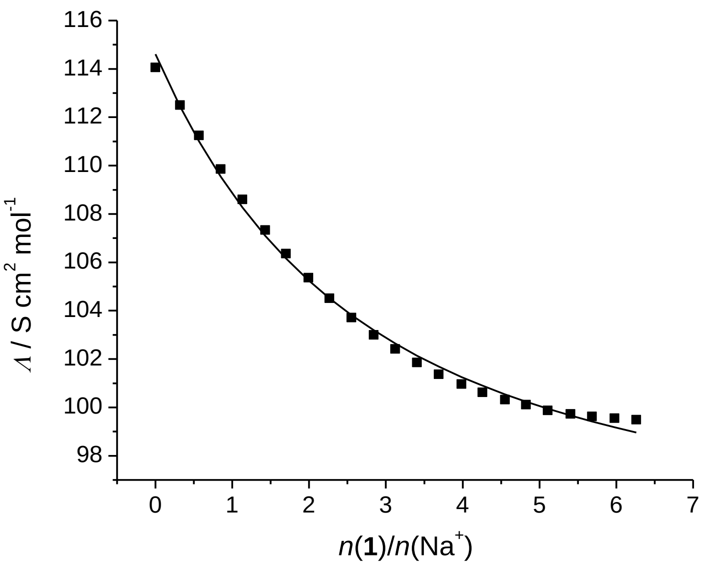

Figure S46. Conductometric titration of $NaClO_4$ (c = 7.82 × 10^{-5} mol dm^{-3}) with **1** (c = 2.22 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C. ■ experimental; – calculated.

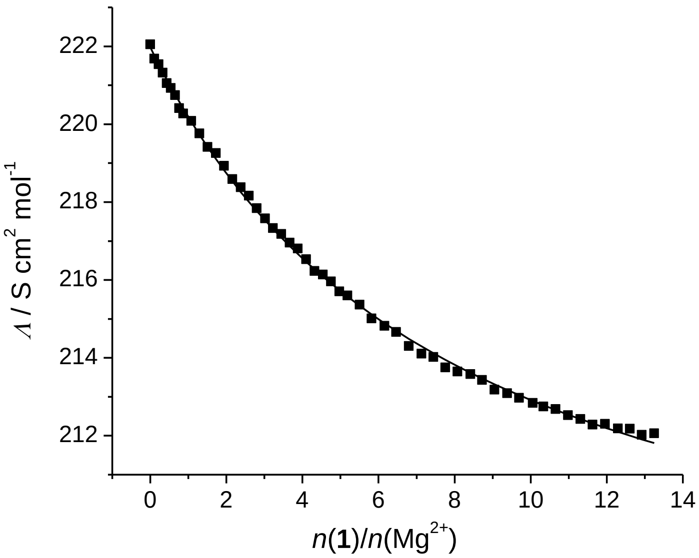

Figure S47. Conductometric titration of $Mg(ClO_4)_2$ (c = 9.11 × 10^{-5} mol dm^{-3}) with **1** (c = 2.03 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C. ■ experimental; – calculated.

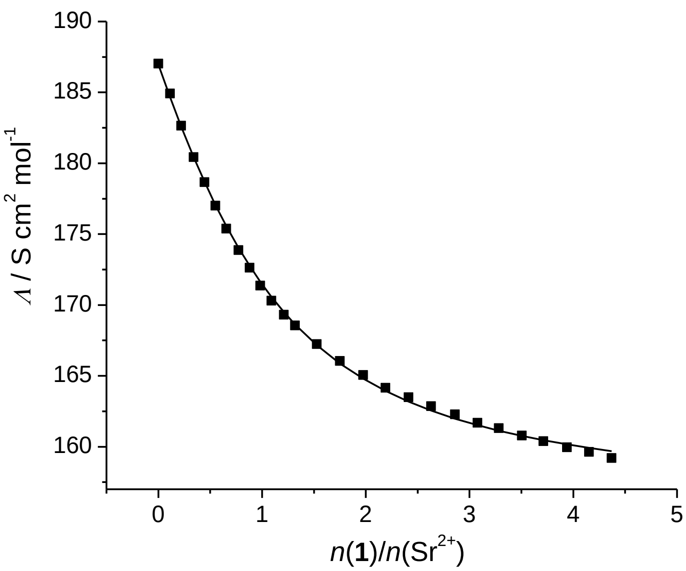

Figure S48. Conductometric titration of $Sr(ClO_4)_2$ (c = 1.35 × 10^{-4} mol dm^{-3}) with **1** (c = 1.92 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C. ■ experimental; – calculated.

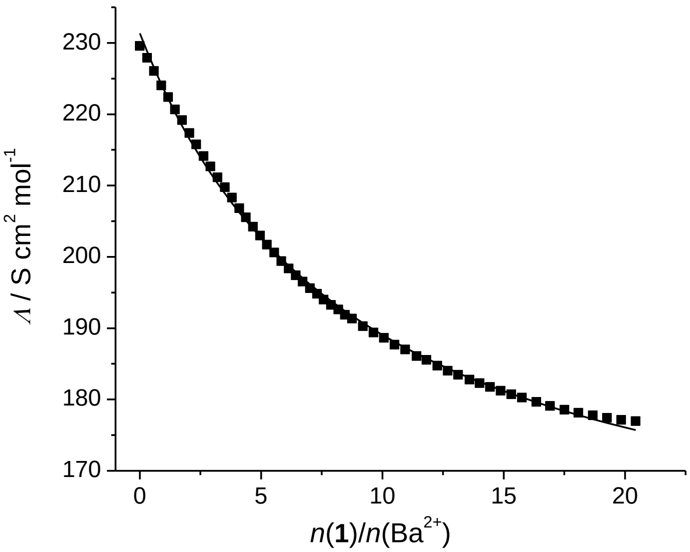

Figure S49. Conductometric titration of $Ba(ClO_4)_2$ (c = 1.41 × 10^{-4} mol dm^{-3}) with **1** (c = 1.92 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C. ■ experimental; – calculated.

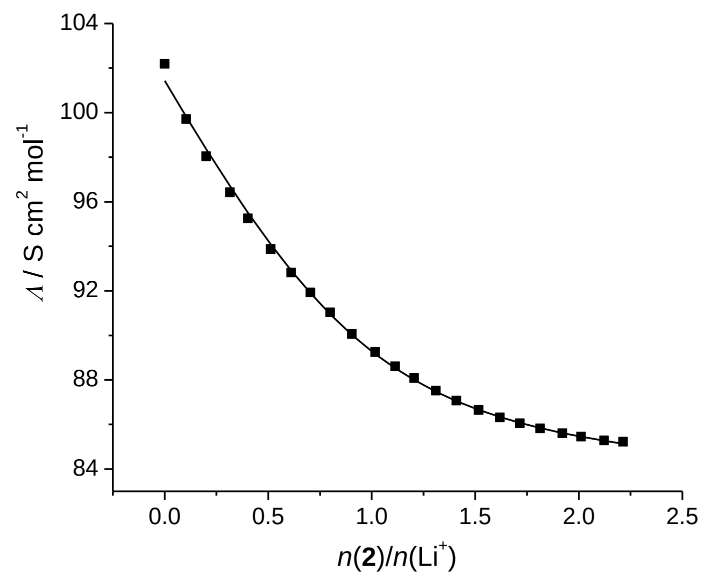

Figure S50. Conductometric titration of $LiClO_4$ (c = 1.05 × 10^{-4} mol dm^{-3}) with **2** (c = 2.33 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C. ■ experimental; – calculated.

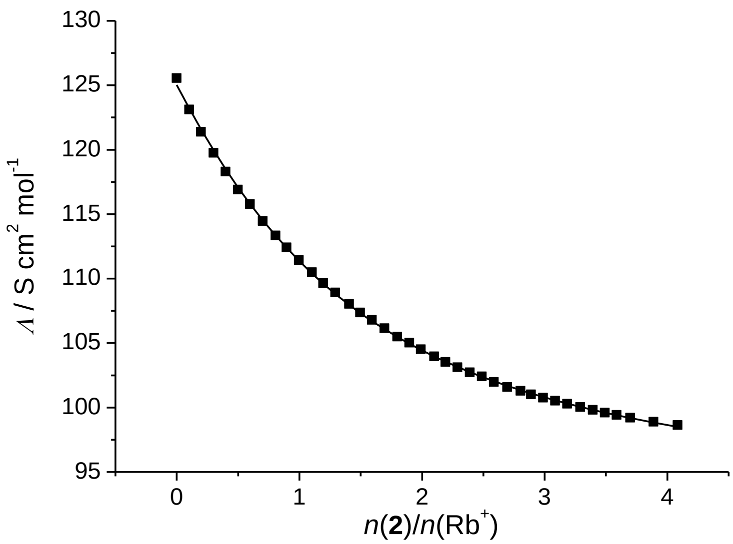

Figure S51. Conductometric titration of $RbNO_3$ (c = 1.07 × 10^{-4} mol dm^{-3}) with **2** (c = 2.33 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C. ■ experimental; – calculated.

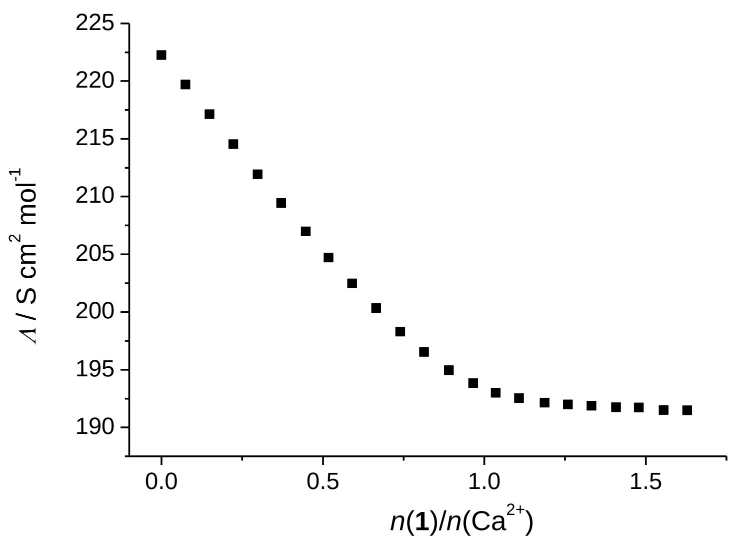

Figure S52. Conductometric titration of $Ca(ClO_4)_2$ (c = 1.96 × 10^{-4} mol dm^{-3}) with **1** (c = 1.92 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C.

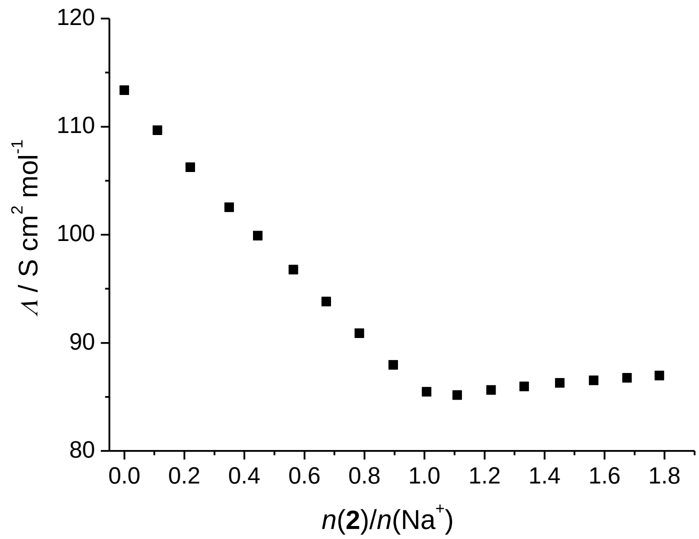

Figure S53. Conductometric titration of $NaClO_4$ (c = 9.44 × 10^{-5} mol dm^{-3}) with **2** (c = 2.12 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C.

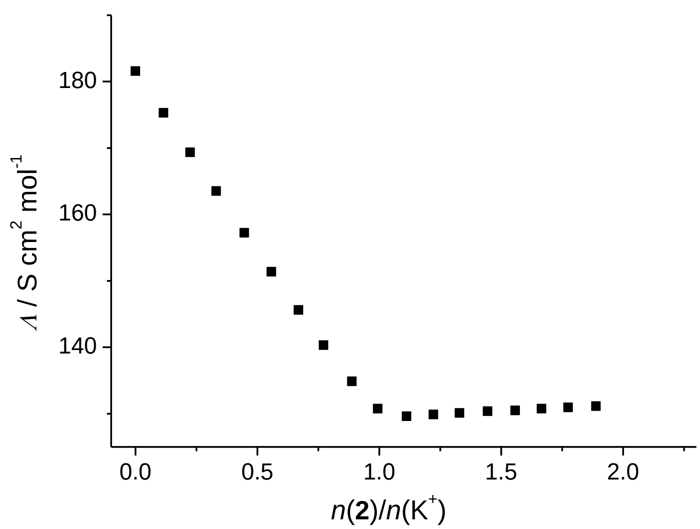

Figure S54. Conductometric titration of $KClO_4$ (c = 1.09 × 10^{-4} mol dm^{-3}) with **2** (c = 1.21 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C.

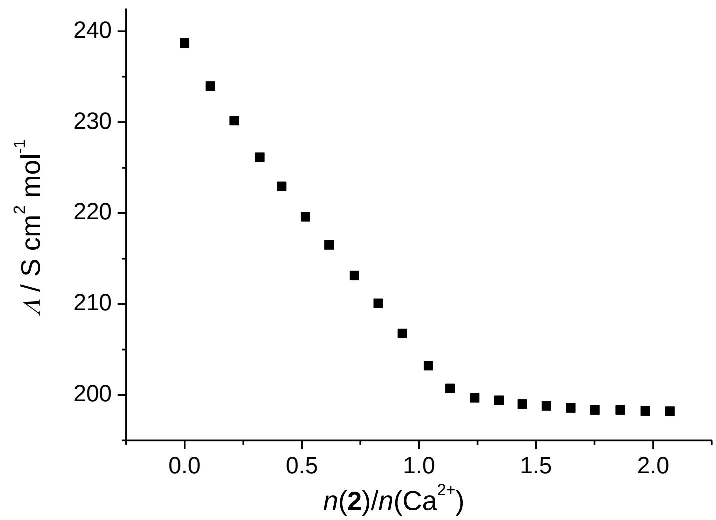

Figure S55. Conductometric titration of $Ca(ClO_4)_2$ (c = 1.08 × 10^{-4} mol dm^{-3}) with **2** (c = 2.33 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C.

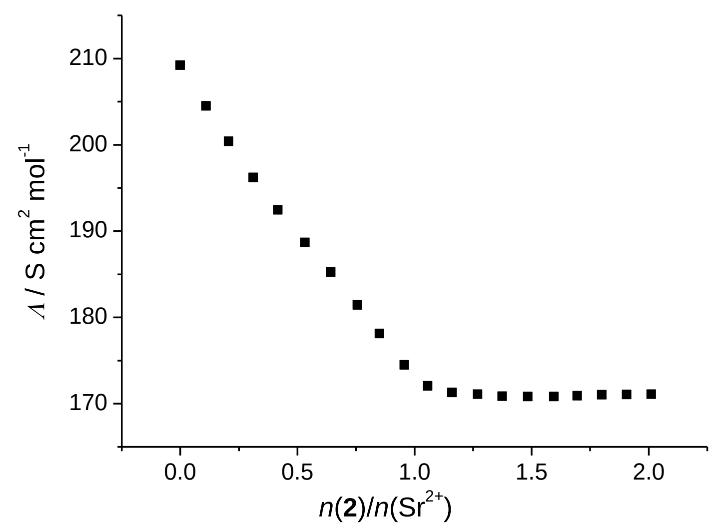

Figure S56. Conductometric titration of $Sr(ClO_4)_2$ (c = 1.86 × 10^{-4} mol dm^{-3}) with **2** (c = 2.33 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C.

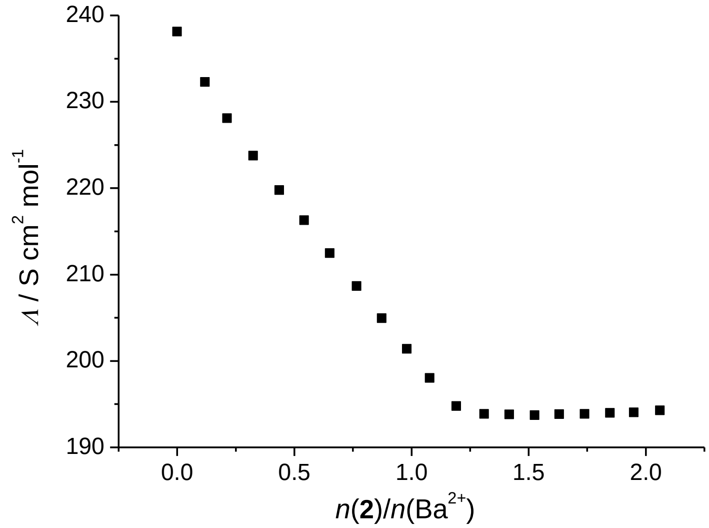

Figure S57. Conductometric titration of $Ba(ClO_4)_2$ (c = 1.02 × 10^{-4} mol dm^{-3}) with **2** (c = 2.33 × 10^{-3} mol dm^{-3}) in methanol; t = (25.0 ± 0.1) °C.