Supporting Information for

Dual-Ionomer-Based Device: Acetylcholine Transport and Nonenzymatic Sensing

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Figure S1. Time-dependent zeta potential responses obtained from ACh⁺ and H⁺ forms of BPS.





Figure S2. A. Chemical structure of salt form BPS. Individual comparison of FTIR spectra of salt and ACh⁺ forms of BPS obtained from Nano-FTIR at **B.** 950 cm⁻¹ and **C.** 1750 cm⁻¹ wavenumbers. **D.** AFM phase images of salt, proton, and ACh⁺ forms of membranes, respectively.



Figure S3. A. Overall IR spectra of the salt, proton, and ACh⁺ forms of membranes from 650-4000 cm⁻, **B.** Detailed investigation of IR spectra of AChCl with labeled characteristic peaks. Individual XPS peaks of **C.** O1s, **D.** O KLL and **E.** C1s obtained from three different cation loaded membranes and pure AChCl.

Table S1. FWHM values calculated from the corresponding characteristic BPS IR peaksshowing the changes after acetylcholination step

Wavenumber (cm ⁻¹)	Cation	FWHM
1030	Na ⁺	15.06
1030	ACh ⁺	11.94
1094	Na ⁺	8.30
1094	ACh ⁺	5.82
1204	Na ⁺	31.98
1204	ACh⁺	28.72



Figure S4. Current vs. time plots obtained from potentiostatic analyses conducted on **A.** Concentration cell and ACh⁺ Pen under $1V_{bias}$ and **B.** miniaturized ACh⁺ Pen under 55 mV_{bias}.



Figure S5. Nyquist plots obtained at different AChCl solution concentrations at 1, 3, and 24 hours for the primary equilibration step.



Figure S6. Post-equilibration data (i.e., Nyquist plots and conductivity vs time column graphs) obtained at different ACh⁺ concentration values during 1, 5, and 10 minutes of the analysis period.

$$\sigma = \frac{l}{Z' \times t \, x \, w}$$

Equation S1





Figure S7. Calibration curves obtained from conductivity values of ACh⁺-BPS measured at different solution concentrations and the corresponding fittings.



Figure S8. Dependence of conductivity on solution temperature due to the reptation of polymeric chains.



Figure S9. Equilibration of the membrane inside a 100 nM KCl solution with varying ACh⁺ concentration in the range of 1×10^{-5} M and 0.05 M.



Figure S10. A. Effect of KCl interference at different concentrations to the conductivity of ACh⁺-BPS sensor. Comparison between calibration curves obtained from pure and 100 nM KCl interfered AChCl solutions at **B.** 1, **C.** 5, and **D.** 10 minutes of analysis periods.



Figure S11. A. Stability of dual transporter-sensor over 15 cycles showing approximate values of conductivity and amount of transported ACh⁺. **B.** Conductivity measurements obtained from regenerated BPS showing an equilibration period of ~24 hours.



Figure S12. A. schematic representation of fluorometric acetylcholine assay analysis performed in PBS environment at 4 different time intervals (15, 60, 300 and 1200 sec). **B.** enzymatic reaction cycle of the acetylcholine quantification via assay kit.