Brooklyn College
Department of Chemistry

Instrumental Analysis (CHEM 42/790G) ***Potentiometry/Ion Selective Electrodes***

<u>pH Measurements</u>

A. Dissolve each of the tablets provided in 100 mL of deionized water to prepare three solutions having pH values of 4, 7, and 10 (use 100 mL volumetric flasks). These will be used for several exercises in this experiment - do not contaminate them or discard them until you are finished.

B. Calibrate the pH meter using a combination pH electrode with temperature compensation (autocalibration function). Follow the instructions provided. Rinse the electrode with deionized water after each measurement and lightly dry it with a Kim-Wipe before placing it in a different solution. Be very careful not to touch the bulb. Determine the electrode slope at 25 °C (or room temperature) by measuring the electrode potential in mV *vs.* pH from all three buffers. (Use 50 mL beakers, and 40 mL of each buffer.)

C. Use a hot plate with a stirrer to heat the samples to approximately 50 °C and remeasure the slope (potential in mV *vs.* pH) and the pH values. Gently stir the solution and do not let the stir bar come near the electrode bulb. Do not recalibrate the meter. Wait for the electrode to reach equilibrium before taking final measurements.

D. Using a 50 mL graduated cylinder, dilute 25 mL of each buffer with an equal volume of water and measure the pH and the electrode potential in mV at 25 °C(or room temperature).

E. Dilute 25 mL of each buffer with an equal volume of methanol and measure the pH and the electrode potential in mV at 25 °C (or room temperature).

F. This meter/electrode combination will be used in the next part of the experiment (to adjust pH of NaCl solutions).

Single Ion Electrode Measurement

The sodium ion-selective electrode is similar structurally to a typical glass electrode used for pH measurements. Here the glass membrane is made with a glass having a high selectivity towards sodium ions. In NaCl solutions of the proper pH the electrode potential will follow the Nernst equation very closely:

$$E = E^0 + (2.303\ RT/F) \log a_{Na^+}$$

So a tenfold change in sodium ion concentration will lead to a cell potential change of about 59.2 mV. These electrodes are susceptible to interferences fro other cations and, especially, for high hydrogen ion concentration. To minimize these interferences, calibration solutions similar in ionic character and strength to the sample solution are used. The pH is kept at least 2 units higher than the pNa to minimize pH interference.

A. Prepare a calibration curve for the Na^+ ion electrode using a calomel electrode as a reference. Use the meter provided for this purpose. Prepare 100 mL of a 1 M NaCl solution. Also prepare three ten-fold dilutions down to $1 \cdot 10^{-3}$ M NaCl (you should have 4 solutions of NaCl: $1 \cdot 10^{-3}$, $1 \cdot 10^{-2}$, 0.1 and 1 M). Adjust the pH of each solution to 4.0 using potassium biphthalate and pH-electrode.

B. Measure cell potential for all solutions (in mV)

C. Plot cell potential (mV) *vs.* sodium concentration (pNa).

C. Adjust the pH of the unknown to 4.0 and determine its sodium concentration using this calibration curve.

D. Store the electrodes in 0.1 M NaCl after the experiment.

Report

Provide a tabular summary (or graph, if appropriate) and a brief discussion of the results of each experiment.