LABORATORY EXPERIMENT 4

Alkalimetric Titration of an Acid Mixture

In this experiment the quantitative composition of a solution, which is a mixture of a monoprotic strong acid (HCl) and a weaker triprotic acid (H_3PO_4) will be determined by pH-potentiometric methods. This experiment will introduce you to semi-automatic volumetric analysis and potentiometric titrations.

Phosphoric Acid, ***H_3PO_4*** (pK_1= 2.16, pK_2 = 7.16, pK_3=12.3). Phosphoric acid can be titrated as a *monobasic* acid:

$$H_3PO_4 + NaOH \rightarrow NaH_2PO_4 + H_2O$$

The pH of a solution of resulting dihydrogen phosphate is:

$$pH = \frac{pK_1 + pK_2}{2} = 4.66$$

The change of the pH near equivalence point is not very pronounced.

Phosphoric acid can also be titrated as a *dibasic* acid, with the second step

$$NaH_2PO_4 + NaOH \rightarrow Na_2HPO_4 + H_2O$$

The pH of solution of monohydrogen phosphate is

$$pH = \frac{pK_2 + pK_3}{2} = 9.7$$

At this pH, phenolphthalein (transition range 8-9.6) exists in its alkaline form; thymolphtalein (pH transition range 8.3-10.5) can be a better choice. Nevertheless, direct measurement of pH values with pH sensitive electrode is more reliable in both cases.

Hydrochloric acid ***HCl*** is very strong and it is completely titrated at the pH of the first equivalence point of phosphoric acid.

Procedure:

The unknown solution containing a mixture of HCl and H_3PO_4 is placed in a 100 or 200 mL volumetric flask and made to the mark with deionized water.

A. The burette is filled with the standard NaOH solution of known concentration. An exact aliquot of 25 mL is placed in a titration beaker, and appropriate electrode is inserted. The stirrer is turned on.

Using pH-meter as an indicator, you titrate first to a first equivalent point (pH 4.66) and record the volume V_1. At this point , all HCl is titrated and phosphoric acid is completely converted in dihydrgen phosphate:

$H_3PO_4 + NaOH \rightarrow NaH_2PO_4 + H_2O$

Continue titration to the second equivalent point (pH 9.7) and record the volume V_2.

At this point all dihydrogen phosphate is converted in monohydrgen phosphate ion:

$NaH_2PO_4 + NaOH \rightarrow Na_2HPO_4 + H_2O$

Amount of phosphoric acid in the aliquot is calculated using the formula:

$$m_{H3PO4} = \frac{FW_{H3PO4} \times C_{NaOH} \times (V_2 - V_1)}{1000}\ (g)$$

Amount of hydrocloric acid in aliquot is calculated as

$$m_{HCl} = \frac{FW_{HCl} \times C_{NaOH} \times (2V_1 - V_2)}{1000}\ (g)$$

Total amount of phosphoric aci and of hydrochloric acid in your unknown (your starting flask) is

$$m_{totalH3PO4} = \frac{m_{H3PO4} \times V_{total}}{V_{aliquot}}\ (g)$$

$$m_{totalHCl} = \frac{m_{HCl} \times V_{total}}{V_{aliquot}}\ (g)$$

Where V_{total} is volume of your flask and $V_{aliquot}$ is volume of your pipette.

B. The automatic burette is filled with the standard NaOH solution of known concentration. An exact aliquot of 25 mL is placed in a titration beaker, and appropriate electrode is inserted. The stirrer is turned on.

Start the data collection by clicking the collect button on computer screen: then turn on the titration unit. Collect the data until you see both end points on data preview screen.

Copy the data collected, and paste them into an appropriate Excel file for further processing.

Determine the exact volumes of NaOH used to achieve first and second ending point.

Two breaks will occur in the titration curves, the first corresponding to the titration of hydrogen ions from the HCl and the first hydrogen ion from the H_3PO_4. The second break corresponds to the titration of $H_2PO_4^-$ that resulted from the H_3PO_4.

Report total amount of HCl and and total amount of H_3PO_4 in your unknown. Recall that you titrate 25-mL portions of a 200-mL (or 100-mL) total sample.

Attach a spreadsheet plot of one full titration curve and the corresponding plots.