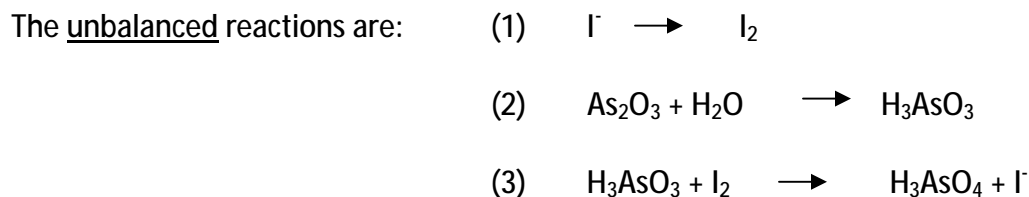


Physical Chemistry Lab

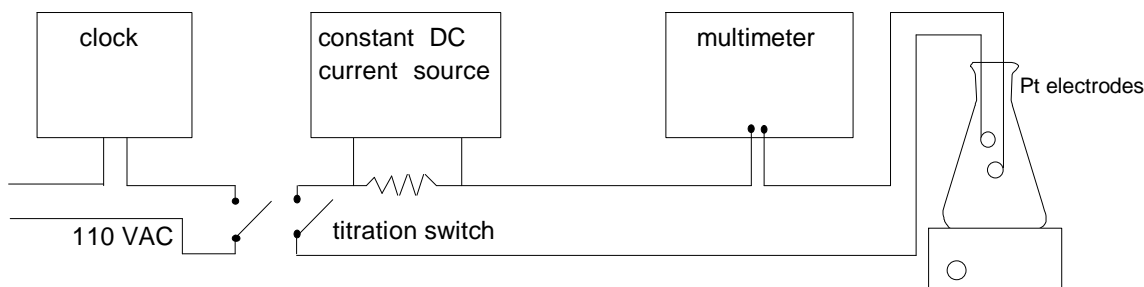
Coulometric Titration

I. Introduction

Redox reactions may be separated into half reactions involving either the gain or loss of electrons at electrodes. In this titration to determine the concentration of a $\text{As}_2\text{O}_3(\text{aq})$ solution, I_2 is formed from I^- by an oxidation half reaction. The I_2 then reacts with the $\text{As}_2\text{O}_3(\text{aq})$. The moles of I_2 formed is readily determined by measuring the current and the time and recalling that one mole of electrons equals 96485 coulombs (amp seconds). One particular advantage of this method is that I_2 is formed only as needed and hence all the disadvantages of trying to store an I_2 solution are circumvented. The purpose of this experiment is to determine the concentration of an unknown solution of $\text{As}_2\text{O}_3(\text{aq})$.



II. Apparatus



Note: A resistance of approximately 2 ohms prevents the power supply from generating a high voltage when the titration switch is off.

III. Experimental

Set up the apparatus as shown on the previous page. Using a solution containing 0.8 g KI, 3.0 g NaHCO₃, and 15 mL starch indicator diluted to 500 mL, fill a 250 mL narrow mouth Erlenmeyer flask sufficiently to cover the electrodes when inserted.

Place the flask on a magnetic stirrer and insert a stirring bar. Turn the stirrer on to insure that it stirs smoothly. (If the magnetic stirrer has a separate on/off switch, leave the switch on and use the speed control knob for adjustment of speed. Do not ever start the stirrer at high speed using the on/off switch since the stirring bar will sometimes spin around rather than spin smoothly.)

Set the multimeter to 20 ma and ma DC. Set the power supply initially on 0-15 ma upper knob) and 0-30 ma constant current (lower knob). With the titration switch off adjust the current to about 10 ma. Reposition the upper knob to 0-150 volts. (The current through the cell will be read by the multimeter.)

Lower the electrodes carefully into the flask. Adjust the stirrer speed. Do not stir extremely rapid. Turn the titration switch on and examine the blue color appearing at one of the electrodes. After about 15 seconds stop and let the solution clear. If the solution is bluer than desired add a few drops of the As₂O₃ solution until the solution clears up and then readjust the color. When satisfied with the color, set the timer on zero. (If the timer used is a 'count down' type, there will possibly be a 'count up' set of numbers.)

Pipette 1 mL (check with instructor to see what is recommended for the sample to be analyzed) of the unknown As₂O_{3(aq)} solution into the Erlenmeyer flask. Turn the titration switch on. Record the ma (which should be between 7-10 ma) every 30 seconds. Adjust the power supply as necessary to maintain this current. Every 2 minutes turn the titration switch off and let the iodine diffuse off the electrode. When the entire solution appears to be turning a permanent light blue, turn the titration switch off and see if the color persists and matches the initial color. The titration switch is equivalent to the stopcock of a buret. If the color does not persist or does not match, turn the switch on until the appropriate persistent color is achieved. Record the total time.

Calculate the concentration of the As₂O_{3(aq)} solution. Repeat the procedure at least twice more. There is no need to replace the KI solution although it might be necessary to discard some of it if the Erlenmeyer fills up.

Write-up for Coulometric Titration Experiment

I. Raw Data Tables

II. Calculations and Discussions

A. Identify the possible redox reactions in the electrolysis at each electrode and predict the actual reactions. Balance the redox equation.

B. Write the balanced equation of As_2O_3 with water to form arsenious acid.

C. Write the balanced redox reaction of arsenious acid with iodine to form arsenic acid from the balanced half cell reactions.

D. Calculate the molarity of the As_2O_3 solution for each trial. Explain the calculation. It is probably best done as a stoichiometry problem with factor label conversions.

E. Convert your stoichiometry problem into an equation involving current, time, volume of sample and other constants. Then determine the propagated error in the molarity for each trial and the best value for the molarity in each trial.

F. Determine the scatter error for the three determinations.

G. Determine the best value obtained for the molarity taking into account both the propagated and scatter error.

H. Discuss the general usefulness of the method. Comment on sources of error. What other methods might be used to determine the concentration?