Determination of the Purity of a Sample of Potassium Pyrosulfate by Conductimetric Titration.

Background:

The electrical conductance in electrolytic solutions depends on the concentration of the ions present in a solution and the inherent ability of each kind ion to conduct electricity (equivalent conductance). Any reaction that changes the concentration of ions will show changes in conductivity.

Therefore conductance method can be used for the determination of end points in precipitation titrations and/or in neutralization titrations. Dependent on the titrant and the titre, the reaction solution conductivity, will either increase or decrease. The rates of change of conductivity are different before and after the end point. Thus plots of *specific conductivity vs. volume of the titrant* will result in straight lines (arms). The location of the point at which these conductivity rate changes occur (arms intersect) amounts to the location of the end point of the titration. The equilibria at the end points tend to change the sharpness of the plot near end points. Therefore the end points are found by extrapolating the lines generated in the *conductivity vs. volume of the titrant plots*.

In this experiment the purity of a potassium pyrosulfate (molecular mass = 254.31) sample is determined using a soluble sulfate solution as an 'internal standard'. The reactions involved are;

Hydrolysis of pyrosulfate: $K_2S_2O_7$ (aq) + H_2O = $2KHSO_4$ (aq)

Titration Reactions: $2KHSO_4$ (aq) + $Ba(OH)_2$ (aq) = $BaSO_4$(s) + K_2SO_4 (aq) + $2H_2O$

K_2SO_4 (aq) + $Ba(OH)_2$ (aq) = $BaSO_4$ (s) + 2KOH (aq)

Apparatus:	Conductivity bridge, conductivity cell, pipette 25 ml, volumetric flasks 250ml (3), graduated cylinder 25 ml, tall beaker 150 ml or larger, magnetic stirrer, pipette 10ml (2), burette.
Chemicals:	Barium hydroxide, unknown (solid potassium pyrosulfate), potassium sulfate, phenopthalein.

Procedure:

Prepare a ~0.1M solution of barium hydroxide (in carbon dioxide free water) just before the experiment. Barium hydroxide reacts with carbon dioxide from the atmosphere forming barium carbonate (solid). Filter the solution as quickly as possible, if needed.

Crush a sample of solid potassium pyrosulfate nearly to a powder. Weigh *accurately* about 0.3g of potassium pyrosulfate, transfer quantitatively into the tall beaker.

Prepare a ~0.1M *standard solution* of potassium sulfate in a 250ml volumetric flask.

Set up the conductivity cell in the beaker on a magnetic stirrer. Pipette 10.00 ml of standard potassium sulfate into the tall beaker and add distilled, carbon dioxide free water until the electrodes in the conductivity cell are completely immersed and about 0.5cm below the level of the solution. Titrate this solution vs. the solution of barium hydroxide while stirring at a *constant* rate. Measure the conductance of the solution in the cell after each addition of barium hydroxide. *Allow sufficient time between additions of barium hydroxide to complete the reaction after each addition of barium hydroxide.* Barium sulfate precipitation is not instantaneous, especially closer to the end point of the titration. Continue the titration until you clearly observe three slopes (segments) on the conductimetric plot.

Treatment of data:

1. Plot the conductance vs. the volume of titrant.
2. Using the end points calculate the volume of barium hydroxide solution required to titrate the added potassium sulfate standard.
3. Calculate the concentration of barium hydroxide solution.
4. Calculate the amount of HSO_4^- produced by potassium pyrosulfate.
5. Calculate the amount of 'potassium pyrosulfate’ via the hydrolysis reaction equation.
6. Calculate the percentage purity of the solid potassium pyrosulfate.