

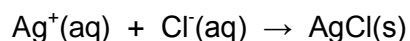
Tutorial – Titrations

1. A method for determining chloride (and bromide and iodide) in solution is the Volhard method. A measured excess of Ag^+ is added to the sample, causing the precipitation of AgCl . The excess Ag^+ is determined by titration with a standard thiocyanate (SCN^-) solution. Fe^{3+} is used as indicator, when all the Ag^+ has been used up, the SCN^- then forms the red FeSCN^{2+} .

a) In one version of Volhard's method, the precipitated AgCl is removed by filtration before back-titrating the filtrate with the SCN^- solution. Why is the AgCl removed? What could happen (in the presence of excess SCN^-) if the AgCl was not removed? Would the problem be better or worse if iodide was being determined?

b) 0.5342 g of a solid sample containing sodium iodide and sodium nitrate was dissolved in water and diluted to a final volume of 250.0 mL. A 25.00 mL aliquot of this solution was taken and treated with 25.00 mL of 0.0504 M AgNO_3 solution. The excess Ag^+ was then back-titrated with a standard (0.0498 M) NaSCN solution. The titration volume was 19.32 mL. Calculate the percentage of NaI in the solid mixture.

2. The chloride concentration in a solution was determined by gravimetric analysis. A 25.00 mL aliquot of the sample was pipetted into a beaker, some nitric acid was added and the mixture was diluted to about 100 mL with distilled water. Dilute silver nitrate was then added to precipitate silver chloride:



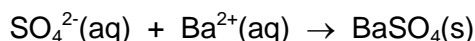
(The solubility product (K_{sp}) for silver chloride is 1.82×10^{-10})

A slight excess of AgNO_3 was added to make sure that all of the chloride had been precipitated. After all the AgNO_3 had been added, the final volume of solution was 120 mL and the excess Ag^+ concentration was $0.005 \text{ mol dm}^{-3}$. The mixture was heated almost to boiling for about 10 minutes. The beaker was covered and stored in the dark for at least one hour. The precipitate was then filtered, washed with very dilute nitric acid and dried; the mass of the precipitate was found to be 0.4258 g.

a) Use the mass of silver chloride formed to calculate the mass of chloride in the original sample solution.

- b) What was the chloride concentration (g/L) in the original sample solution?
 - c) Why was the mixture “diluted to about 100 ml with distilled water”?
 - d) Why was the mixture “heated almost to boiling for about 10 minutes”?
 - e) Why was the beaker “covered and stored in the dark for at least one hour”?
 - f) Why was a solution of nitric acid used for washing the precipitate and not pure water?
3. A sample (1.4502 g) of a mixture of potassium dihydrogen phosphate (KH_2PO_4) and potassium chloride was dissolved in water and the phosphate was precipitated as magnesium ammonium phosphate hexahydrate $\text{Mg}(\text{NH}_4)\text{PO}_4 \cdot 6\text{H}_2\text{O}$. The precipitate was filtered washed and heated strongly to convert the phosphate to magnesium pyrophosphate, $\text{Mg}_2\text{P}_2\text{O}_7$. The final mass of magnesium pyrophosphate was found to be 0.7852 g.
- a) Calculate the percentage of potassium dihydrogen phosphate in the sample.
 - b) Why was the $\text{Mg}(\text{NH}_4)\text{PO}_4 \cdot 6\text{H}_2\text{O}$ converted to $\text{Mg}_2\text{P}_2\text{O}_7$ before weighing?
Would there have been any problems if the $\text{Mg}(\text{NH}_4)\text{PO}_4 \cdot 6\text{H}_2\text{O}$ had been used as the weighing form?

4. You have to determine the sulfate in a series of samples by precipitation as barium sulfate.



You know that the samples contain about 40%(m/m) sulfate and you want to obtain at least 0.3 g of the precipitate.

- a) What mass of sulfate do you need to get 0.3 g of the precipitate?
- b) What mass of sample should you take to make sure the mass of precipitate is not less than 0.3 g?
- c) Why would you want to obtain “at least 0.3 g of the precipitate”? What is wrong with obtaining a mass of about, say, 0.003 g?