College of Science

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Te Whare Wānanga o Waitaha
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Determination of Chloride Ion Concentration by Gravimetry

Safety

Lab coats, safety glasses and enclosed footwear must be worn at all times in the laboratory.
Silver nitrate solution causes staining of skin and fabric (chemical burns). Any spills should be rinsed with water immediately.
Concentrated nitric acid is very corrosive, take great care using the 6 molL^{-1} solution.

Introduction

This method determines the chloride ion concentration of a solution by gravimetric analysis. A precipitate of silver chloride is formed by adding a solution of silver nitrate to the aqueous solution of chloride ions. The precipitate is collected by careful filtration and weighed.

$$Ag+_{(aq)} + Cl-_{(aq)} \rightarrow AgCl_{(s)}$$

The precipitate can be collected more easily if the reaction solution is heated before filtering. This causes the solid silver chloride particles to coagulate. The precipitation is carried out under acidic conditions to avoid possible errors due to the presence of carbonate and phosphate ions which, under basic conditions, would also precipitate with the silver ions.

As the method requires very careful weighing of the samples, it is best to use it on solutions that are known to contain a fairly significant concentration of chloride ions, such as seawater which is used as the example here. For this reason it should not be used for stream or river water.

Equipment Needed

100 mL volumetric flask

pipettes

250 mL conical flask

burette and stand

Bunsen burner, tripod and gauze

measuring cylinders

Buchner funnel, filter paper and a sidearm filtering flask

Solutions Needed

Silver nitrate solution: (0.1 mol L^{-1}) If possible, dry 5 g of $AgNO_3$ for 2 hours at 100°C and allow to cool. Accurately weigh about 4.25 g of solid $AgNO_3$ and dissolve it in 250 mL of distilled water in a conical flask. Store the solution in a brown bottle.

Nitric acid solutions: A 6 mol L^{-1} solution of nitric acid is needed. **(See safety notes)**. In addition, for washing the precipitate, the following dilute nitric acid solution is needed. Add 1 mL of 6 molL^{-1} HNO3 solution to about 500 mL of distilled water.

Methyl orange indicator

Method

Sample Preparation

If the seawater contains traces of solid matter such as sand or seaweed, it must be filtered before use. Otherwise they will end up being weighed along with the silver chloride precipitate.

Titration

1. Dilute seawater by pipetting a 50 mL sample into a 100 mL volumetric flask and making it up to the mark with distilled water.
2. Pipette a 20 mL sample of diluted seawater into a conical flask and add about 115 mL distilled water and 1 drop of methyl orange indicator. Add dilute nitric acid dropwise until the indicator turns pink, then add 1 mL of 6 molL^{-1} nitric acid. **(See safety notes)**

3. Add dropwise from a burette 55 mL of 0.1 mol L^{-1} silver nitrate solution. Allow the solution to stand for a minute and then test to see if it is completely precipitated by adding one drop of 0.1 mol L^{-1} silver nitrate solution. If more of the silver chloride precipitate forms add an additional 5 mL of the silver nitrate solution and retest for complete precipitation.
4. Heat the solution to boiling. Remove from heat and let stand in the dark for at least 1 hour until the precipitate coagulates (figure 1).
5. Before filtering with the Buchner funnel and flask, weigh the filter paper. Then use the equipment to filter the supernatant liquid (the liquid above the precipitate).
6. Wash the precipitate in the conical flask three times with a few mL of the very dilute nitric acid solution, pouring each washing through the Buchner funnel.
7. Finally transfer the precipitate itself to the Buchner funnel washing any loose particles from the conical flask with a little distilled water.
8. Wash the precipitate on the Buchner funnel three times with a few mL of the very dilute nitric acid solution. Then wash the precipitate three times with a few mL of distilled water.
9. Carefully place the filter paper containing the precipitate on a watch glass and dry overnight. Weigh the dried filter paper and precipitate and calculate the weight of the dried precipitate using the known weight of the filter paper.

Figure 1: Left flask: a cloudy white precipitate of silver chloride forms upon addition of silver nitrate to the chloride sample solution. Right flask: the result of heating and standing. The silver chloride precipitate is seen to coagulate into large clumps, leaving a clear solution. NB: exposure to sunlight may result in some decomposition to form elemental silver, giving the precipitate a slight purple colour as seen here. Try to avoid exposing the precipitate to sunlight any longer than is absolutely necessary.

Result Calculations

1. Use the mass (in grams) of silver chloride in the dried precipitate (step 9 of the method) with the equation of the method to determine the number of moles of chloride ions in your sample.

$$Ag+_{(aq)} + Cl-_{(aq)} \rightarrow AgCl_{(s)}$$

2. Calculate the concentration of chloride ions in the diluted seawater.
3. Calculate the concentration of chloride ions in the original (undiluted) seawater.
4. Calculate the concentration of sodium chloride in the seawater in $molL^{-1}$, gL^{-1} and g/100 mL (%).

Additional Notes

1. Residues containing silver ions are usually saved for later recovery of silver metal. Check this with your teacher, or the laboratory supervisor.

Contact Us

If you have any questions or comments relating to this experiment, please contact us. Please note that this service is for senior school chemistry students in New Zealand only. We regret we are unable to respond to queries from overseas.

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