Water analysis

Chloride content in water

**Aim:** To determine the chloride content of different samples of water.

**Background:** Sea water is salty, mainly due to the presence of sodium chloride. Fresh water contains a far smaller concentration of chloride ions than sea water. Salinity (how salty something is) is important in agriculture, as increasing the salinity of the soil has implications for loss of production. The analysis of chloride is done by titrating with silver salts.

**You will need:**

|  |  |
| --- | --- |
| 25cm3 0.05 moll-1 silver nitrate solution | 3 x empty bijoux |
| Bijou containing 0.1% fluorescein solution | 2 decimal place balance |
| Bijou containing 0.25% salt solution | Clamp stand |
| 3 x 1cm3 pipettes | 100cm3 Beaker |

**To do**

**Water sample preparation**

A 0.25% salt solution has been provided which is ready to analyse.

**Calibrating the pipette**

1. Fill the pipette with water and put into the jaws of the clamp.
2. Place the beaker on the balance, positioned underneath the pipette, and zero it.
3. Slowly tighten the clamp, counting the number of drops until the balance reads **exactly 1.00**. Repeat a couple of times and calculate the average.

**As 1cm3 of water weighs exactly 1g, this allows you to work out the mass of one drop.**

The pipette can now be filled with the silver nitrate solution.

 **Titration**

1. Add 1cm3 of the salt solution to an empty bijou.
2. Add 2 drops of fluorescein.
3. Place the pipette filled with silver nitrate into the jaws of the clamp. Tighten until the first drop falls.
4. Place the bijou under the tip of the pipette, slowly tighten the clamp, counting drops as they go. **Initially it is best to shake after every 5-10 drops, but as the end pint nears, shake after each drop.**
5. When the end point has been reached, record the number of drops in the table.



1. Repeat until you have concordant results.

**Safety**

0.05 moll-1 silver nitrate and 0.1% fluorescein solutions are both low hazard, so there is no need for eye protection for this experiment.

Recycling: It is common, where possible, to collect silver residues to reclaim.

**Results**

Average number of drops in 1cm3: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

|  |  |  |
| --- | --- | --- |
| Titre | Number of drops of silver nitrate | Volume of silver nitrate (number of drops/number of drops in 1cm3) (cm3) |
| 1 |   |   |
| 2 |   |   |
| 3 |   |   |

**Calculation**

As the silver ions react in a 1:1 ratio with the chloride ions, the concentration of chloride ions can be calculated using this method.

Average volume of silver nitrate used: \_\_\_\_\_\_\_\_\_\_\_cm3

Number of moles of silver nitrate reacting:



Concentration of chloride ions:



Phosphate content in water

**Aim:** To determine the phosphate content of different samples of water.

**Background:** Organic phosphates are important in nature. Their occurrence may result form the breakdown of organic pesticides which contain phosphates. They may exist in solution, as particles, loose fragments, or in the bodies of aquatic organisms.

**You will need:**

|  |  |
| --- | --- |
| Potassium antimony tartrate solution# | Distilled water |
| Ammonium molybdate solution# | 3cm3 pipette |
| Ascorbic acid solution# | 100cm3 beaker |
| 5 bijoux containing 5cm3 1ppm, 2ppm, 3ppm, 4ppm and 5ppm standard phosphate solutions. | 10cm3 Measuring cylinder |
| 1 x bijou containing 5cm3 water sample | Cuvettes |
| 2.5 moll-1 sulfuric acid | Colorimeter |

# see technician notes at the end for preparation.

**To do**

1. **Make up the combined reagent:** Mix the reagents in a beaker in the following proportions (and order) for 20cm3 of the combined reagent.
	1. 10cm3 2.5 moll-1 sulfuric acid.
	2. 1cm3 potassium antimony tartrate solution.
	3. 3cm3 ammonium molybdate solution.
	4. 6cm3 ascorbic acid solution.

If turbidity forms in the combined reagent, shake, and let stand for a few minutes. This reagent is stable for **four hours.**

1. Add 1cm3 of combined reagent to the bijou containing 1ppm phosphate standard solution. Replace lid and shake.
2. Repeat for the other four standard solutions, and the water sample provided.
3. Leave the solution for 15 minutes.
4. Place a cuvette containing deionised water into the colorimeter and calibrate.
5. Transfer some of the coloured solution to a cuvette and read the absorbance at 590nm (or red filter).
6. Repeat with the other reference samples, and the test sample.
7. Using the readings from the reference samples, plot the figures on the calibration graph.
8. Using the reading for the test sample, read the absorbance off the graph to determine the concentration.

**Safety**

0.00844 mol-1potassium antimony tartrate solution, ammonium molybdate, phosphate and ascorbic acid have no significant hazards.

2.5 moll-1 sulfuric acid is corrosive, and the combined reagent is a skin/eye irritant, so eye protection should be worn.

**Results**

|  |  |
| --- | --- |
| Standard phosphate solution concentration (ppm) | Absorbance |
| 0 | 0.00 |
| 1 |   |
| 2 |   |
| 3 |   |
| 4 |   |
| 5 |   |

Test sample absorbance:\_\_\_\_\_\_\_\_\_\_\_\_\_\_



Test sample concentration:\_\_\_\_\_\_\_\_ppm

Example calibration graph.



**Technician Notes**

**Equipment and materials required for each group**

|  |  |
| --- | --- |
| 50cm3 2.5M sulphuric acid | Colorimeter and Cuvettes |
| 5cm3 potassium antimony tartrate | Distilled water |
| 15cm3 ammonium molybdate solution | Beaker/bottle for combined reagent |
| 30cm3 ascorbic acid  | Test tubes |
| 15cm3 10ppm (PO43-) Phosphate standard solution | Measuring cylinder |

 **A standard stock 10ppm phosphate solution:**

A 10ppm solution of phosphate = 14.3ppm solution of potassium dihydrogen phosphate.

(10ppm = 10mg/l)

To ensure accuracy it is better to make a more concentrated solution and then dilute it.

1000ppm (PO43-) phosphate stock solution: Dissolve 1.43g of potassium dihydrogen phosphate in distilled water in a 1000cm3 volumetric flask and make up to the mark.

10ppm (PO43-) phosphate solution: Transfer 10cm3 of 1000ppm stock solution to a 1000cm3volumetric flask and make up to the mark.

**Potassium antimony tartrate**: Dissolve 1.371g K(SbO)C4H4O6. ½H2O in 400cm3 distilled water in a 500cm3 volumetric flask and dilute to volume.

**Ammonium molybdate solution**:Dissolve 20g (NH4)6Mo7O24. 4H2O in 500cm3 distilled water.

**0.1M Ascorbic Acid**:Dissolve 1.76g ascorbic acid in 100cm3 distilled water