Experiment 01 - Preparation and properties of tin compounds

Chemical Hazards

The following reagents are used in this experiment:

Bromine water 

Dichloromethane 

Iodine 

Mercury(II) chloride (4% in water) 

Tin (granulated)

Tin(II) chloride (10% solution in HCl) 

Zinc (granulated)

The following compounds are formed in this experiment:

Tin(IV) iodide 

Reaction mixture from test 1 

Reaction mixture from test 2 

Reaction mixture from test 3 

Experimental

The experiment consists of three parts:

1. The preparation of tin(IV) iodide.
2. The purification of tin(IV) iodide.
3. Some tests on the reducing ability of tin(II) chloride.

You should start Part 1 straight away and carry out the tests in Part 3 whilst the reaction in Part 1 is taking place.

Throughout the experiment you will need to keep appropriate notes in your lab book.

Part 1 - Preparation of tin(IV) iodide

Weigh about 2.5 g of granulated tin and 2.5 g of iodine into a clean, dry 100 ml round-bottomed (RB) flask. Add 20 ml of dichloromethane (DCM), add a stirrer bar and fit a reflux condenser.

Reflux the reaction mixture - only gentle heating is necessary as DCM has a very low boiling point (40˚C) - for about 45 min. The solution should develop an obvious orange colour. Dispose of any residual tin in the tin waste container.

The following video shows you how to set up and run the experiment.

Part 2 - Purification of tin(IV) iodide

Filter the hot solution into a conical flask through a small plug of glass wool in the neck of a filter funnel. Add 10 ml of DCM to the RB flask containing the remaining tin, and use this to rinse the remaining product in the reaction flask and funnel into the conical flask. Put any residual tin into the waste container.

Reduce the volume of the combined filtrate to about 10 ml by evaporation on a hotplate. Keep an eye on your solution; it can evaporate to dryness very quickly.

Cool the concentrated solution in an ice bath and collect the resulting crystals by suction filtration using a clean, dry Büchner funnel and filter paper.

You may find that a second crop of crystals appear in the residual orange solution in the receiving flask; if so, simply pass the suspension through a new filter and collect the additional material. Keep it separate from the first set of crystals.

Air-dry your product and measure its melting point.

HAND YOUR SAMPLE IN TO THE PREP ROOM. Label the sample vial with your name, date, group and name or formula of the compound.

Make sure the demonstrator has a record of your yield (in g) and your melting point.

Part 3 - Tests on the reducing ability of tin(II) chloride

You are provided with a solution containing 10 g of $SnCl_2$ in 100 ml of dilute hydrochloric acid. Carry out the following tests with this solution:

- To 1 ml of mercury(II) chloride ($HgCl_2$) solution add a few drops of the tin(II) chloride solution and record your observations. Add a further 1 ml of the tin solution.
- Add a few pieces of granulated zinc metal to a test tube containing a few ml of the tin(II) chloride solution.
- Test a portion of the tin(II) chloride solution with a few drops of bromine water.

Make a note of your observations and interpretations of the reactions. As far as you can, give equations for the reactions occurring and identify which reactants are oxidised and which are reduced.

Clearing up

When you have finished make sure all your glassware has been washed, dried and put away.

Assuming your glassware is contaminated with product/DCM or tin/product/DCM wash as follows:

- rinse with a small amount of DCM (waste to chlorinated waste container)
- wash with water/detergent/bottle brush (waste to sink)
- rinse with water (waste to sink)
- final rinse with acetone (waste to non-chlorinated waste container)
- dry outside of glassware
- replace in drawer

The glassware for the tests can be washed as follows:

- water/detergent/bottle brush (waste to sink)
- rinse with water (waste to sink)
- final rinse with acetone (waste to non-chlorinated waste container)
- dry outside of glassware
- replace in drawer

Make sure the demonstrator has a record of your yield (in g) and your melting point before you leave the lab.

Report

In the *Student Comments* box on the DLM please supply a literature value for the mp of your sample with a reference **(internet references e.g. Wikipedia are not acceptable)** along with your percentage yield.