NEATH PORT TALBOT COLLEGE COLEG CASTELL NEDD PORT TALBOT

School of Maths & Science Science Practical

Preparation of Iodoform

🔶 Aim

At the end of the experiment you should be able to:

- 1. Prepare and isolate a sample of iodoform
- 2. Purify it by recrystallisation from ethanol
- 3. Assess its purity by recording the melting point.

Introduction

Iodoform, CHI_3 , is the heavier iodine analogue of the well known chloroform $CHCl_3$. It can be prepared by the haloform reaction which involves treating a methyl alcohol or a methyl ketone with iodine in the presence of a base. Iodoform is a yellow crystalline solid which has a clinical smell. In former times it was used as an antiseptic.

♦ Safety



Control Measures

- The wearing of safety glasses, gloves and a laboratory coat at all times will be sufficient to take account of most hazards and significant risks.
- Keep stoppers on bottles as much as is possible.
- Keep flammable liquids away from flames.
- All waste is to be placed in the labelled container immediately after use.
- You are reminded of the need of good laboratory practise in order to maintain a safe working environment.



Hazards

Harmful/Irritant

Copper (II) Sulfate Solution Zinc Powder



Procedure

Preparation

- 1. Place about 2cm³ of acetone (propanone) in a 250cm³ conical flask and add 15cm³ of 2M sodium hydroxide. Swirl the mixture thoroughly.
- 2. In a measuring cylinder, measure out about 40 cm^3 of iodine solution.
- 3. Pour the iodine solution into the acetone / sodium hydroxide solution carefully and gradually, making sure you swirl the flask continuously.
- 4. After complete addition, mix the contents thoroughly and then leave the flask to stand for five minutes.

Isolation

- 1. Collect the yellow precipitate of iodoform using a suction filtration apparatus. The use of this will be demonstrated to you. Make sure you clamp the Buchner flask which will be top heavy with the funnel in it.
- 2. Wash the solid with a little cold water and then place the precipitate and filter paper onto a clean watch glass. Dry in the oven for ten minutes.

Purification

- 1. Place the solid in a boiling tube and add hot ethanol **dropwise** until the solid **just dissolves**. The ethanol can be pre-heated by placing some in a boiling tube and heating in a hot water bath.
- 2. Allow the solution to cool. The iodoform should crystallise out as a fine yellow precipitate. You can cool it in ice if the crystallisation is troublesome but be patient. You will get better quality crystals from a slow crystallisation.
- 3. Filter the solid again, dry in the oven and record a melting point.