

∞ Lab Report

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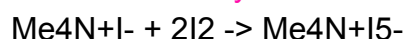
Introduction:

One of the most important things in chemistry is to know how each substance combining together to form a compound. Compounds are the pure substances which are made from two or more elements combined in a fixed composition. When 2 or more elements combined, the new compound are created. But before the new compound is completely formed, there were many processes happened when the compound are formed such as process of filtration and crystalization. Furthermore, filtration is the process of separating solid matter from a liquid, by letting the smaller particles to pass through, called a filter. The liquid which has passed through the filter is called the filtrate. The filter may be paper, cloth, sand, or other porous material. Crystalization is a way to purifying the compound by adding a hot solvent in the solution to completely dissolved the solute. As when it cools, crystal are formed.

Objective:

The purpose of this experiment is to form the combining substance named 'Tetramethylammonium iodine' and 'Iodine' to synthesizing the new compound named 'Tetramethylammonium pentaiodide' by dissolving with Ethanol. This experiment also demonstrates the process of crystalization and filtration.

Synthesis of tetramethylammonium pentaiodide:



Procedure:

1. Add to a 50 mL beaker tetramethylammonium iodide (0.5 g). Use a stirring rod to break up clumps of $\text{Me}_4\text{N}^+\text{I}^-$.
2. Add iodine (1.3 g) and 95% ethanol (12 mL).
3. In a fumehood, gently heat the beaker, with stirring, on a hot-plate for about 10 minutes until tetramethylammonium iodide has dissolved.

4. Heat at a low temperature to prevent premature crystallization.
5. Remove from the heat and allow the beaker to cool undisturbed. (Agitating the solution can cause smaller crystals to form which are more difficult to isolate and tend to occlude impurities)
6. The crystals should form during this time.
7. Once at room temperature, further cooling can be achieved by placing the beaker in an ice bath for about 10 minutes. Only put the solution on ice if necessary. During this time, set up the apparatus for suction filtration.
8. Using vacuum filtration, collect the crystals and wash them with hexanes (5 mL portions until filtrate dropping from funnel is colorless, max. 5 washings).
9. Air-dry the product with weigh.
10. Hand in a sample, in a capped vial, labelled with your name, the name of the compound and the date to the lab instructor.

Materials:

- ∞ Gloves
- ∞ Tube
- ∞ Beaker
- ∞ Flask
- ∞ Sterring glass
- ∞ Goggles
- ∞ Iodine
- ∞ Lab coat
- ∞ Filter paper
- ∞ Vacuum filtration
- ∞ Ethanol
- ∞ Tetramethylammonium iodine
- ∞ Weighing apparatus
- ∞ Watch glass

Data:

Before crystallization	After crystallization
<ul style="list-style-type: none"> ∞ Tetramethylammonium iodine 0.5g ∞ Iodine 0.59g ∞ Ethanol 12 ml 	<ul style="list-style-type: none"> ∞ $\text{Me}_4\text{N}^+\text{I}_5^-$ of 0.76g ∞ Small crystal

Discussion Questions:

- ∞ Calculate number of mole of each compound.

∞ Mass of "Me" = 15.0345 g 1.0023 mol

∞ Mass of "I" = 126.90447 g 1 mol

∞ Mass of "N" = 14.00674 g 1 mol

tetramethylammonium iodide (0.5 g) / $\text{Me}_4\text{N} + \text{I}$

$0.5/201.04921 = 0.0024$ mol

∞ Calculate percent yield. (actual yield / theoretical yield *100)

$$1.62\text{g}/1.77\text{g}\times 100 = 91.53\%$$

∞ Why do we use vacuum filtration not gravity filtration?

We use vacuum filtration to filtering the crystal because this vacuum filtration have less pressure comparing to the gravity filtration. Using the vacuum filtration would get the result faster and it filtrate thoroughly better.

- ∞ tetramethylammonium iodide 0.5g
- ∞ iodine 0.59g
- ∞ ethanol 12 ml
- ∞ Then, we cool it with ice
- ∞ Crystalization-form crystal very hard
- ∞ Outcome- small crystal
- ∞ Outcome- 0.76g

Analyzing results:

In this experiment, we synthesized 'tetramethylammonium pentaiodide' by using 'Tetramethylammonium iodide' of 0.5g and 'iodine' 0.59g. Then, we combined these two substances with 'ethanol' of 12 ml. We heated up these substance until the crystalization process happen. After we got the crystals, we need to washed the crystals with hexane and filter it with the vacuum filtration.

In the experiment, we waited for the crystal to be formed for a long time because we used the less amount of iodine. It meaned that less amount of iodine effect the rate when crystal were formed. The amount of iodine being used in the experiment also determined the size of the crystal. According to the amount we have used, it make a smaller crystal. The crystal we had was purple and brownish color.

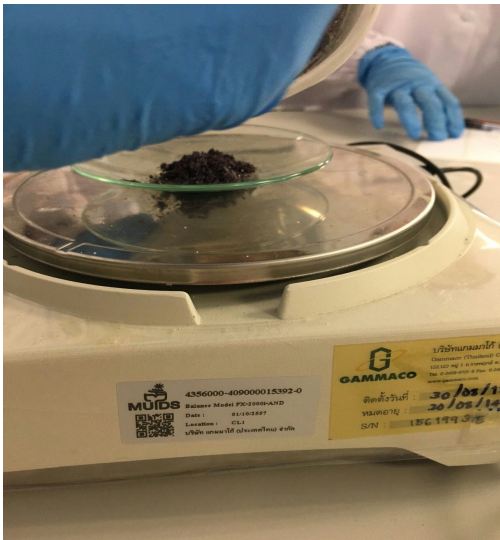
Although, we have been successful in our experiment but we had some difficulties during the experiment. The difficulies are that the crystal were hard to formed due to the amount of iodine we used. We had to cool it with ice to make the crystal formed clearer.

Conclusion:

According to this experiment our group use many substance to form the crystal. For example, tetramethylammonium iodide, iodine, ethanol and hexanes to run the process. So we know the way how to calculate the mole of each compound and bring it to calculate the percent yield. This experiment helps us know that actual

yield and theoretical yield are related to each other. Moreover, we know the process how to form the crystallization by using the synthesis of tetramethylammonium pentafluoroborate.

Pictures from the experiment:



References:

Crystallization. (n.d.). Retrieved from <http://orgchem.colorado.edu/Technique/Procedures/Crystallization/Crystallization.html>

Filtration. (2009). Retrieved from <http://www.lenntech.com/chemistry/filtration.htm>