

25. Planning the preparation and purification of *N*-phenylethanamide

Student Sheet

In this experiment you will use your knowledge of previous organic syntheses, and the information given below, to plan the preparation of a sample of *N*-phenylethanamide, $\text{C}_6\text{H}_5\text{NHCOCH}_3$ starting from phenylammonium chloride, $\text{C}_6\text{H}_5\text{NH}_3\text{Cl}$, which is a salt of phenylamine.

Intended lesson outcomes

By the end of this exercise you should be able to:

- produce a detailed plan whereby a specified quantity of purified product may be prepared
- decide how the yield of product will be calculated
- decide how the purity of the product will be assessed
- assess the risks involved and suggest appropriate safety precautions

Background information

The preparation of *N*-phenylethanamide from phenylamine is termed **acylation**. Both ethanoyl chloride, CH_3COCl , and ethanoic anhydride, $(\text{CH}_3\text{CO})_2\text{O}$, may be used as acylating agents. The reactivity of ethanoic anhydride is, however, lower than that of ethanoyl chloride and allows the reaction rate to be more easily controlled.

Phenylamine is most conveniently used in the form of the salt phenyl ammonium chloride.

The reaction is performed in two stages.

- 1 A solution of sodium ethanoate is treated with phenylammonium chloride forming phenylamine, ethanoic acid and sodium chloride.
- 2 Phenylamine then reacts with ethanoic anhydride to form *N*-phenylethanamide as a white solid, together with ethanoic acid.

An aqueous solution of phenylammonium chloride (5.0 g in 150 cm^3) is mixed with ethanoic anhydride (10 cm^3). To this mixture is added a second aqueous solution containing sodium ethanoate (30 g in 125 cm^3). The sodium ethanoate causes the reaction in step 1 to occur. Once phenylamine is released, it reacts readily with ethanoic anhydride to form *N*-phenylethanamide as a white solid. The crude solid product is isolated and recrystallised from water. Typically a yield of around 70% of the theoretical maximum is obtained.

Note

An excess of ethanoic anhydride is used in order to ensure a good yield.

The task

Give **full details** of how a 2.0 g sample of **pure** crystalline *N*-phenylethanamide could be prepared using the above solutions, without preparing an excess of product. When calculating the quantities of materials to be used, you should assume a **maximum overall yield** for the preparation of **70%**.

Give details of a physical test you would perform to confirm the purity of the crystals.

Your plan should include:

1. A list of the essential apparatus you would use
2. Equations for the reactions taking place

Appendix 2

3. The relative formula masses, M_r values, of phenyl ammonium chloride and *N*-phenylethanamide
4. The mass of phenylammonium chloride needed to make 2.0 g of *N*-phenylethanamide assuming a 100% yield
5. The quantities of phenylammonium chloride, ethanoic anhydride and sodium ethanoate needed to make 2.0 g of *N*-phenylethanamide assuming a 70% yield

Note: The workings for all calculations should be shown.

6. Full details of procedures by which 2.0 g of **pure, dry** *N*-phenylethanamide could be prepared
7. A physical test that you would do in order to assess the purity of your product
8. A full description of how you would perform the physical test for purity, and how you would interpret its results
9. Details of any potential risks in the procedure, and appropriate safety precautions to be taken

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Teachers' Notes

Planning exercises work best when students have sufficient knowledge of the topic not to need the support of books or notes. As planning may be a new skill to many students it is recommended that at the beginning of the course, group work is used so students can pool ideas and learn from each other. As students become more confident, they can progress to individual work, and finally to practising planning exercises under exam conditions.

From the student's point of view, often the most difficult part of a planning exercise is getting started. Be prepared to give help early on in the learning process, but encourage students to be more independent as the course progresses.

The main questions you might wish to consider when assessing the plan are:

- 1 is the method used clearly described?
- 2 have appropriate techniques, reagents and pieces of apparatus been selected?
- 3 is there an appreciation of scale and precision (including relevant calculations)?
- 4 have all the appropriate safety factors been properly considered?

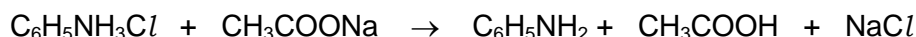
When deciding on a marking scheme for this plan, you could award one mark for each of the points below. However, this would place a greater emphasis on the *assessing the purity* section than on the *scale and precision* section. A second possibility is to mark each section to a maximum for that section. One way of doing this is illustrated below. The figure in brackets after each heading shows the maximum mark for that section, with a breakdown of how those marks are awarded, giving a maximum mark of 21 for the exercise. To change the emphasis of the plan, all that is needed is to change the mark allocation to each section.

Marking the plan

There are five main areas to consider.

1 Scale and precision: 1 mark for each (maximum mark = 6)

1. The correct equation for the reaction between phenyl ammonium chloride and sodium ethanoate.



2. The correct equation for the reaction between phenylamine and ethanoic anhydride.



3. M_r values: for phenylammonium chloride = 129.5; for *N*-phenylethanamide = 135
4. Theoretical amount of phenyl ammonium chloride needed for 2.0 g yield = 1.92 g
5. Actual amount for phenyl ammonium chloride 2.0 g yield = 2.74 g
6. Sensible quantities of the other reagents

2 Method, including apparatus: 6/7 correct = 4 marks; 4/5 correct = 3 marks; 3/4 correct = 2 marks; 1/2 correct = 1 mark (maximum mark = 4)

1. measuring cylinders
2. conical flask or other suitable vessel
3. access to a balance
4. filtering apparatus, e.g. Buchner apparatus

Appendix 2

5. mixes solution and stirs
6. removes crude product by filtration
7. washes crude product with cold water

3 Purification of the crude product: 4 correct = 3 marks; 3 correct = 2 marks; 1/2 correct = 1 mark (maximum mark = 3)

1. dissolves in the minimum quantity of hot water
2. filters while hot
3. cools hot solution, filters crystals, dries crystals
4. weighs dry sample

4 Assessing the purity of the recrystallised product: 6 correct = 4 marks; 4/5 correct = 3 marks; 3/4 correct = 2 marks; 1/2 correct = 1 mark (maximum mark = 4)

1. appropriate method chosen e.g. melting point (possibly mixed melting point or the use of spectral analysis)

outcome – e.g.

2. product melts sharply
 3. melting point agrees with data book value
- basic technique used – e.g.*
4. sample placed in melting point tube
 5. states type of / describes melting point apparatus
 6. heats slowly near melting point

5 Safety: 6/7 correct = 4 marks; 5/4 correct = 3 marks; 3/4 correct = 2 marks; 1/2 correct = 1 mark (maximum mark = 4)

1. phenyl ammonium chloride and *N*-phenylethanamide are both **toxic**
2. ethanoic anhydride is **corrosive**
3. there are potential **fire hazard** with organics
4. avoid skin contact
5. wash areas affected by spillage with water
6. wear gloves
7. wear eye protection

Technical information

- Access to hazard cards or equivalent
- When candidates have some experience in planning this exercise could be carried out in exam-style conditions with a time limit of around 1 hour