

Preparation of Inorganic Pharmaceutical Compounds

Experiment 6 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

1. Boric Acid

Aim

To study the specified organic preparation, isolate the assigned product and calculate its yield.

Principle

An assigned acidification and crystallisation route yields boric acid from the prescribed borate source.

Requirements

- Instructor-approved reaction scheme, reagent quantities and laboratory procedure for the selected route.
- Reaction glassware, suitable ventilation, separation apparatus, balance and melting-point apparatus.

Procedure

1. Plan the selected route

Write the balanced reaction and identify the functional-group change, limiting reagent, expected product and hazards. Use the assigned supervised method for the selected option.

2. Carry out the assigned reaction

Measure the prescribed reagents and follow the authorised addition sequence and temperature controls. Record actual reaction observations and the method used.

3. Isolate the product

Cool the assigned solution to crystallise, collect, wash and dry as specified.

4. Purify and dry

Apply the assigned recrystallisation or purification method. Record solvent, observations, dry mass and melting range where appropriate.

5. Assess the product

Calculate theoretical and percentage yield. Compare the measured physical properties and assigned identity test; explain that yield alone does not prove purity.

Preparations and Reaction Scheme

Route / product	Starting material and change
Boric acid	Assigned borate-to-boric-acid route.

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Observations and Record

Record	Student entry
Selected route / balanced equation	_____
Limiting reagent and molar mass	_____
Theoretical product mass	_____
Dry product mass / melting range	_____
Percentage yield / identity finding	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- Theoretical product mass = moles of limiting reagent $\times$ stoichiometric product ratio $\times$ product molar mass.
- Percentage yield = dry product mass / theoretical product mass $\times$ 100.
- Illustration: 0.020 mol of a limiting reagent gives 0.020 mol of a 150 g/mol product. The theoretical mass is 3.00 g; 2.40 g isolated product gives 80.0% yield.

Result

Product: _____. Route: _____. Dry mass: _____. Yield: _____. Physical property / identity evidence: _____.

Precautions

- Use the specific supervised procedure and facilities for corrosive, toxic, oxidising or flammable reagents.
- Record the selected preparation option rather than combining different reaction routes.
- Use dry product mass for yield and the stated test conditions for comparison.

Viva Questions and Answers

1. Why use crystallisation?

Different solubility behaviour permits separation of the product from soluble impurities.

2. Preparation of Potash Alum

Aim

To prepare potash alum crystals and calculate yield using the stated hydrated formula.

Principle

Potash alum is $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$. It can crystallise from a suitable mixture of potassium sulphate and aluminium sulphate solutions. Solubility differences with temperature allow purification by crystallisation. Hydrated crystal mass includes water of crystallisation.

Requirements

- Prescribed potassium sulphate and aluminium sulphate with stated hydrate form.

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- Water, beakers, controlled heating, filtration apparatus and balance.

Procedure

1. Calculate reactants

Use the correct hydrate molecular mass. Identify the limiting reactant and the theoretical alum amount.

2. Dissolve

Prepare the required warm solutions using the prescribed water amounts. Combine in the assigned proportions.

3. Clarify

Filter the warm solution if required to remove insoluble matter, maintaining the specified conditions.

4. Crystallise

Concentrate or cool as directed until crystals form. Avoid uncontrolled rapid evaporation or heating that changes the hydrate.

5. Collect

Separate crystals, use the prescribed small wash and dry under conditions that preserve the intended hydrate.

6. Evaluate

Record crystal appearance and dry surface condition, weigh and calculate yield on the dodecahydrate basis.

Observations and Record

Item	Basis	Record
Reactants	Mass and hydrate form	_____
Crystals	Appearance / recovered mass	___ g
Yield	Actual ÷ theoretical × 100	___%

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- Reaction basis: $\text{K}_2\text{SO}_4 + \text{Al}_2(\text{SO}_4)_3 + 24 \text{H}_2\text{O} \rightarrow 2 \text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.
- Illustrative equimolar amounts: 1.743 g $\text{K}_2\text{SO}_4 \approx 0.010$ mol and 6.664 g $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O} \approx 0.010$ mol.
Theoretical alum = $0.020 \times 474.39 \approx 9.49$ g.
- If 9.10 g crystals are recovered on the intended hydrate basis, yield = $9.10 \div 9.49 \times 100 \approx 95.9\%$.

Result

Potash alum crystals were obtained with appearance _____. Recovered mass ____ g; hydrate-basis yield ____%.

Precautions

- Use hydrate-corrected reactant masses.

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- Do not strongly heat the final crystals when the calculation assumes the dodecahydrate.
- Excessive wash water can dissolve product and reduce yield.

Viva Questions and Answers

1. Why can cooling give crystals?

The solution can become supersaturated as solubility decreases.

2. What is the formula of potash alum?

$KAl(SO_4)_2 \cdot 12H_2O$.

3. Does a high yield prove purity?

No; retained mother liquor or impurities can inflate the mass.

3. Calcium Lactate

Aim

To study the specified organic preparation, isolate the assigned product and calculate its yield.

Principle

The assigned neutralisation or salt-forming route produces a calcium salt of lactic acid. Hydration state affects mass calculations.

Requirements

- Instructor-approved reaction scheme, reagent quantities and laboratory procedure for the selected route.
- Reaction glassware, suitable ventilation, separation apparatus, balance and melting-point apparatus.

Procedure

1. Plan the selected route

Write the balanced reaction and identify the functional-group change, limiting reagent, expected product and hazards. Use the assigned supervised method for the selected option.

2. Carry out the assigned reaction

Measure the prescribed reagents and follow the authorised addition sequence and temperature controls. Record actual reaction observations and the method used.

3. Isolate the product

Apply the prescribed concentration and crystallisation stage, collect the solid and record its specified hydration basis.

4. Purify and dry

Apply the assigned recrystallisation or purification method. Record solvent, observations, dry mass and melting range where appropriate.

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5. Assess the product

Calculate theoretical and percentage yield. Compare the measured physical properties and assigned identity test; explain that yield alone does not prove purity.

Preparations and Reaction Scheme

Route / product	Starting material and change
Calcium lactate	Assigned calcium source and lactic acid salt-forming route.

Observations and Record

Record	Student entry
Selected route / balanced equation	_____
Limiting reagent and molar mass	_____
Theoretical product mass	_____
Dry product mass / melting range	_____
Percentage yield / identity finding	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- Theoretical product mass = moles of limiting reagent $\times$ stoichiometric product ratio $\times$ product molar mass.
- Percentage yield = dry product mass / theoretical product mass $\times$ 100.
- Illustration: 0.020 mol of a limiting reagent gives 0.020 mol of a 150 g/mol product. The theoretical mass is 3.00 g; 2.40 g isolated product gives 80.0% yield.

Result

Product: _____. Route: _____. Dry mass: _____. Yield: _____. Physical property / identity evidence: _____.

Precautions

- Use the specific supervised procedure and facilities for corrosive, toxic, oxidising or flammable reagents.
- Record the selected preparation option rather than combining different reaction routes.
- Use dry product mass for yield and the stated test conditions for comparison.

Viva Questions and Answers

1. Why state hydration?

Water of crystallisation changes formula mass and calculated yield.

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4. Preparation of Magnesium Sulphate

Aim

To prepare magnesium sulphate by either permitted starting-material route and compare their reaction behaviour.

Principle

Magnesium hydroxide or carbonate reacts with sulphuric acid to form magnesium sulphate. The carbonate route also releases carbon dioxide. The product hydrate and starting-material identity determine theoretical mass; basic hydrated magnesium carbonate is not automatically identical to pure MgCO_3 .

Requirements

- Prescribed magnesium hydroxide or carbonate with known composition.
- Dilute sulphuric acid, water, balance, stirring vessels, filtration and crystallisation equipment.

Procedure

1. Choose and identify the route

Record whether the starting solid is $\text{Mg}(\text{OH})_2$, MgCO_3 or a specified basic carbonate. Use its stated composition in calculations.

2. React gradually

Use the prescribed dilute-acid addition with stirring and temperature control. For carbonate, allow gas evolution to subside before the next addition; keep the vessel unsealed.

3. Check completion

Follow the prescribed endpoint and handle any excess undissolved material as directed. A single pH reading does not itself prove product purity.

4. Clarify and crystallise

Filter if required, then concentrate and cool under the specified conditions.

5. Collect and record

Separate, wash and surface-dry the intended hydrate. Record appearance and hydrate-basis yield.

Observations and Record

Route	Reaction observation	Product record
Hydroxide	_____	_____
Carbonate	_____	_____
Crystals / hydrate	_____	___ g

Worked Example

The numbers below are illustrative; enter your own observations in the record.

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- Hydroxide: $\text{Mg}(\text{OH})_2 + \text{H}_2\text{SO}_4 \rightarrow \text{MgSO}_4 + 2 \text{H}_2\text{O}$. Carbonate: $\text{MgCO}_3 + \text{H}_2\text{SO}_4 \rightarrow \text{MgSO}_4 + \text{CO}_2 + \text{H}_2\text{O}$.
- For an ideal 2.00 g $\text{Mg}(\text{OH})_2$ portion, theoretical $\text{MgSO}_4 \cdot 7\text{H}_2\text{O} = 2.00 \times 246.47/58.32 \approx 8.45$ g.
- For 2.00 g pure MgCO_3 , theoretical heptahydrate = $2.00 \times 246.47/84.31 \approx 5.85$ g. A basic hydrated carbonate needs its own composition calculation.

Result

Starting material: _____. Product hydrate: _____. Recovered mass ____ g; theoretical mass ____ g; yield ____%.

Precautions

- Do not confuse carbonate grades or ignore water of crystallisation.
- Control gas evolution and avoid acid splashes.
- Do not strongly dry a hydrate while retaining its hydrated molecular mass in the yield calculation.

Viva Questions and Answers

1. Which route releases gas?

The carbonate route releases carbon dioxide.

2. Why do equal starting masses give different theoretical yields?

The two starting compounds have different molar masses.

3. What must be stated for yield?

The limiting reactant and the product's hydrate basis.

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