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Semester 1 - Practical Notes

Pharmaceutical Inorganic and Analytical Chemistry

Complete Subject Manual

19 practicals and activities. Use the PDF bookmarks to move between practicals. Page numbers restart within each practical; the page counts below exclude the promotion page.

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Chloride and Modified Chloride Limit Tests

Experiment 1 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare the impurity response of a test preparation with the prescribed standard under matched conditions.

Principle

Chloride reacts with silver ions to produce silver chloride turbidity: $Ag^+ + Cl^- \rightarrow AgCl$. The test and standard are compared after the same reagent additions and development conditions. The prescribed acidic medium helps reduce interference from ions such as carbonate.

Requirements

- Prescribed monograph for the test material, impurity standard and suitable matched comparison vessels.
- Silver nitrate test reagent, specified nitric acid and chloride-free vehicle.
- For the modified test: the material-specific approved pretreatment and recovery check.

Procedure

1. Read the monograph

Identify test mass, dissolution method, chloride-standard amount, reagent strengths and comparison time.

2. Prepare test and standard

Dissolve the test portion and prepare the standard with the stated amount of chloride. Use matched cylinders and the same final volume.

3. Develop turbidity

Add the prescribed acid and silver nitrate in the stated sequence. Mix equally and protect from inappropriate light exposure.

4. Compare

At the defined time compare turbidity under the prescribed viewing conditions; record less than, equal to or greater than the standard.

5. Perform the modified test

Use the prescribed substance-specific pretreatment, then carry out the matched comparison. Record how interference was removed and whether the impurity was retained.

Modified Test: What Changes

- The precipitation principle remains the same; preparation is adapted to the named test material.
- For a carbonate-containing matrix, the prescribed acid treatment may remove carbonate with carbon dioxide evolution before the final comparison. This is a matrix example, not a universal modified recipe.
- For coloured or reactive material, use the monograph's approved decolourisation or other pretreatment and a suitable recovery/control check. Apply the same relevant treatment to the comparison preparation when specified.

Observations and Record

Item	Required record	Observation
Test portion / standard	Mass; impurity amount in standard	_____
Conditions	Reagents, final amount and development time	_____
Comparison	Test versus standard response	_____

Worked Example

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

- Illustrative limit calculation: a 2.0 g test portion has a stated teaching limit of 20 µg chloride/g. The comparator contains 2.0 × 20 = 40 µg chloride.
- A standard containing 20 µg/mL supplies 40 µg in 2.0 mL. These numbers demonstrate comparator preparation, not a universal IP chloride limit.

Result

The test response was ____ compared with the standard. Under valid matched conditions it ____ the prescribed limit; an invalid blank, standard or preparation requires repeat assessment.

Precautions

- Use chloride-free water and clean glassware; contamination can produce a false failure.
- Match reagent additions, final volume and reading time.
- Do not improvise pretreatment with an oxidiser or reducing agent; it may introduce chloride or alter recovery.
- Use the specific substance's prescribed limit; a teaching calculation does not set the permitted impurity concentration.

Viva Questions and Answers

1. What produces turbidity?

Silver chloride particles formed from chloride and silver ions.

2. Why use matched conditions?

The visual response depends on reagent concentration, time and optical conditions.

3. Why can a modified test be necessary?

Colour, insolubility or chemical interference can obscure the ordinary comparison.

Sulphate and Modified Sulphate Limit Tests

Experiment 2 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare the impurity response of a test preparation with the prescribed standard under matched conditions.

Principle

Sulphate reacts with barium ions to form barium sulphate turbidity: $Ba^{2+} + SO_4^{2-} \rightarrow BaSO_4$. The prescribed acidic conditions and reagent system support a reproducible comparison. Carbonate and other matrix effects must not be allowed to produce unrelated turbidity.

Requirements

- Prescribed monograph for the test material, impurity standard and suitable matched comparison vessels.
- Specified barium chloride or barium-sulphate reagent system, acid and sulphate-free vehicle.
- Material-specific pretreatment for the modified test.

Procedure

1. Identify conditions

Record the material, test mass, sulphate-standard amount, reagent system and development time from the prescribed method.

2. Prepare the pair

Make test and standard to the same stated final amount in matched comparison vessels. Check that any insoluble material is handled as prescribed.

3. Develop turbidity

Add acid and the prescribed barium reagent in the specified order. Mix both equally.

4. Compare

At the fixed time compare the test turbidity with the standard under the same viewing conditions.

5. Apply the modification

Where the matrix interferes, use the approved material-specific preparation and verify that it has not removed or introduced sulphate.

Modified Test: Matrix Preparation

- A modified test keeps the sulphate-comparison objective but changes the preparation needed for a particular substance.
- Record the reason for modification: colour, poor dissolution or another specified interference.

- If carbonate is present, the approved acid-treatment stage can remove it before barium addition. Strongly coloured materials need the prescribed preparation and controls; do not infer a method solely from the material's colour.

Observations and Record

Item	Required record	Observation
Test portion / standard	Mass; impurity amount in standard	_____
Conditions	Reagents, final amount and development time	_____
Comparison	Test versus standard response	_____

Worked Example

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

- Illustrative standard: 2.0 g material at a teaching limit of 50 µg/g requires 100 µg sulphate in the comparator.
- If the standard is 50 µg/mL, use 2.0 mL to supply 100 µg. Match the prescribed final volume and reagent additions.

Result

The test response was ____ compared with the standard. Under valid matched conditions it ____ the prescribed limit; an invalid blank, standard or preparation requires repeat assessment.

Precautions

- Do not replace the prescribed barium reagent system with an arbitrary solution strength.
- Keep volumes, mixing and time the same for test and standard.
- Use sulphate-free materials and avoid carryover from earlier tests.
- Use the specific substance's prescribed limit; a teaching calculation does not set the permitted impurity concentration.

Viva Questions and Answers

1. What is the insoluble product?

Barium sulphate.

2. Why may acidification be important?

It can suppress carbonate-related interference while providing the stated reaction conditions.

3. Does a darker starting colour mean more sulphate?

No; matrix colour is an interference, not a sulphate assay.

Iron Limit Test

Experiment 3 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare the impurity response of a test preparation with the prescribed standard under matched conditions.

Principle

In the traditional teaching method, thioglycolic acid reduces iron as needed and forms a coloured iron complex in the prescribed alkaline medium. Citric acid assists the solution chemistry and limits precipitation. Colour is compared with a standard containing the allowed iron amount.

Requirements

- Prescribed monograph for the test material, impurity standard and suitable matched comparison vessels.
- Iron standard, thioglycolic-acid reagent, specified citric-acid and alkaline reagents.
- Iron-free vehicle and matched colour-comparison cylinders.

Procedure

1. Record the method

Identify the material, test portion, iron-standard amount and specified reagent strengths.

2. Prepare solutions

Dissolve or prepare the test as prescribed. Make the standard with the stated iron amount and a matched final volume.

3. Develop colour

Add the reagents in the specified order and adjust to the stated alkaline conditions. Mix and allow the same colour-development time for both.

4. Inspect controls

Check that reagent blanks and the standard behave acceptably. A precipitate or obscuring starting colour may invalidate comparison.

5. Compare

Compare the developed colour, commonly purple in this teaching method, and record the relation to the standard.

Observations and Record

Item	Required record	Observation
Test portion / standard	Mass; impurity amount in standard	_____
Conditions	Reagents, final amount and development time	_____
Comparison	Test versus standard response	_____

Worked Example

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

- Illustrative standard amount: 1.0 g test portion at 10 µg iron/g requires 10 µg iron.
- A 5 µg/mL standard supplies this amount in 2.0 mL. A matched colour comparison is still required; arithmetic alone is not a test result.

Result

The test response was ____ compared with the standard. Under valid matched conditions it ____ the prescribed limit; an invalid blank, standard or preparation requires repeat assessment.

Precautions

- Use iron-free reagents and avoid metal contamination.
- The alkaline condition must match the method; uncontrolled pH changes can affect colour or precipitation.
- Do not use the colour response as a quantitative iron assay unless the method has been validated for that purpose.
- Use the specific substance's prescribed limit; a teaching calculation does not set the permitted impurity concentration.

Viva Questions and Answers

1. What is the role of thioglycolic acid?

It supports reduction and formation of the coloured iron complex.

2. Why is citric acid included?

It assists complexation and helps prevent unwanted precipitation under the method conditions.

3. What does a limit test report?

Whether the impurity response is within the prescribed comparator limit, under valid conditions.

Lead Limit Test

Experiment 4 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare the impurity response of a test preparation with the prescribed standard under matched conditions.

Principle

A traditional lead limit test uses the coloured lead-dithizone complex under controlled extraction conditions. pH, interfering metals and the solvent phase affect the response. The test is compared with the prescribed lead standard, rather than judged simply by the presence of any colour.

Requirements

- Prescribed monograph for the test material, impurity standard and suitable matched comparison vessels.
- Lead standard and instructor-prepared dithizone reagent system.
- Specified extraction vessels, solvent and approved laboratory controls.

Procedure

1. Review the method

Identify the material, prescribed lead limit, extraction scheme and controls. The instructor provides the hazardous reagent system.

2. Prepare the pair

Prepare the test portion and lead-standard comparison with the specified matched conditions.

3. Observe extraction

Under the laboratory's controlled procedure, observe mixing, phase separation and identification of the phase containing the coloured complex.

4. Assess validity

Check the blank, standard and interference controls. Emulsions or incomplete phase separation must not be read as a valid test.

5. Compare and record

Compare the prescribed phase colour and record whether the test response exceeds the standard.

Observations and Record

Item	Required record	Observation
Test portion / standard	Mass; impurity amount in standard	_____
Conditions	Reagents, final amount and development time	_____
Comparison	Test versus standard response	_____

Worked Example

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

- Illustrative comparator: 0.5 g material at 20 µg lead/g needs 10 µg lead in the standard.
- A 10 µg/mL standard supplies 10 µg in 1.0 mL. The actual monograph determines the limit and extraction conditions.

Result

The test response was ____ compared with the standard. Under valid matched conditions it ____ the prescribed limit; an invalid blank, standard or preparation requires repeat assessment.

Precautions

- Lead and the extraction reagents require the laboratory's exposure controls and segregated waste collection.
- Use instructor-prepared hazardous reagents; do not improvise masking-reagent formulas.
- Do not discard metal-containing solvent into ordinary drains.
- Use the specific substance's prescribed limit; a teaching calculation does not set the permitted impurity concentration.

Viva Questions and Answers

1. What gives the characteristic colour?

A lead-dithizone complex in the prescribed solvent system.

2. Why control pH?

Complex formation and interfering-metal behaviour depend on it.

3. Why does phase separation matter?

The comparison must be made in the specified phase under valid extraction conditions.

Arsenic Limit Test

Experiment 5 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare the impurity response of a test preparation with the prescribed standard under matched conditions.

Principle

The classical Gutzeit principle detects arsenic through a stain produced in a controlled gas-generation apparatus. The generated arsenic-containing gas is highly toxic, so this practical is taught through a sealed authorised demonstration, supplied records or a simulation. No gas-generation recipe is part of these notes.

Requirements

- Prescribed monograph for the test material, impurity standard and suitable matched comparison vessels.
- Instructor-operated closed demonstration or approved video and comparator stain record.
- Method-specific standard information and observation sheet.

Procedure

1. Identify the principle

Label the closed reaction section, interference-control section and stain-detection section on the supplied apparatus diagram.

2. Check demonstration records

Record test-portion identity, standard arsenic amount, controlled exposure period and the instructor's validity checks.

3. Observe without intervention

Study the sealed demonstration or video. Students do not prepare gas-generating reagents, open the apparatus or handle detector material.

4. Compare stains

Use the supplied valid test and standard stain records. Compare colour intensity under the prescribed viewing conditions.

5. Report

State the relation to the standard and distinguish a classical teaching demonstration from a validated modern instrumental arsenic assay.

Observations and Record

Item	Required record	Observation
Test portion / standard	Mass; impurity amount in standard	_____
Conditions	Reagents, final amount and development time	_____
Comparison	Test versus standard response	_____

Worked Example

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

- Illustrative comparator arithmetic only: 1.0 g material at a stated 2 µg/g limit requires 2 µg arsenic in the comparison.
- If a supplied standard is 1 µg/mL, 2.0 mL contains 2 µg. This is a paper calculation, not an instruction to generate gas.

Result

The test response was ____ compared with the standard. Under valid matched conditions it ____ the prescribed limit; an invalid blank, standard or preparation requires repeat assessment.

Precautions

- Use the authorised closed demonstration or simulation only.
- Do not open, assemble or run a gas-generation experiment from a textbook description.
- Hazardous residues and detector materials are handled by authorised staff.
- Use the specific substance's prescribed limit; a teaching calculation does not set the permitted impurity concentration.

Viva Questions and Answers

1. What is compared?

The developed test stain against the prescribed standard stain.

2. Why are matched conditions needed?

Stain intensity depends on the controlled exposure and apparatus conditions.

3. Does this test identify every arsenic species separately?

No; the classical limit response is not a complete species-specific analysis.

Preparation of Aluminium Hydroxide

Experiment 6 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To prepare an aluminium hydroxide precipitate and explain precipitation, washing and yield basis.

Principle

Controlled addition of a suitable alkaline reagent to an aluminium-salt solution produces Al(OH)3. The hydroxide is amphoteric and can dissolve in sufficiently strong excess alkali. Fresh precipitate is hydrated; a wet gel mass cannot be assumed to be the mass of pure dry Al(OH)3.

Requirements

- Prescribed aluminium salt and alkaline reagent, water and test reagents for residual soluble salts.
- Beakers, balance, stirring equipment and suitable filtration apparatus.

Procedure

1. Calculate

Record the aluminium salt's hydrate form and assigned quantities. Calculate the ideal dry-product amount from the balanced reaction.

2. Prepare solutions

Dissolve the prescribed aluminium salt in water. Prepare or obtain the instructor-approved alkaline reagent.

3. Precipitate

Add the reagent gradually with stirring under the stated pH and temperature conditions. Avoid uncontrolled excess strong alkali.

4. Collect and wash

Separate the precipitate as prescribed and wash to remove soluble salts, checking the washings by the assigned test.

5. Finish

Retain as gel or dry under the prescribed method, depending on the exercise. Record appearance, recovered mass and its wet, dry or assay basis.

Observations and Record

Stage	Required observation	Record
Precipitation	Appearance and final condition	_____
Washings	Specified residual-salt test	_____
Recovered material	Wet / dry basis and mass	___ g

Worked Example

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

- Ideal reaction with ammonia solution: Al2(SO4)3 + 6 NH4OH → 2 Al(OH)3 + 3 (NH4)2SO4. NH4OH represents aqueous ammonia in this teaching equation.
- If 2.00 g Al2(SO4)3·18H2O, molar mass approximately 666.42 g/mol, reacts ideally: Al(OH)3 = (2.00/666.42) × 2 × 78.00 ≈ 0.468 g.
- This is an ideal dry Al(OH)3 yield. Do not calculate percentage yield from the uncorrected mass of a wet hydrated gel.

Result

The precipitate was described as ____. Recovered mass = ____ g on a ____ basis; washings met ____ test.

Precautions

- Avoid excess strong alkali because aluminium hydroxide is amphoteric.
- Wash under the prescribed method; visible whiteness does not prove absence of soluble salts.
- Use the stated hydrate and dry basis in yield calculations.

Viva Questions and Answers

1. What does amphoteric mean?

The material can react with both acids and suitable bases.

2. Why wash the precipitate?

To remove soluble salts and remaining reagents.

3. Why is wet-gel yield misleading?

The measured mass includes variable retained water.

Preparation of Potash Alum

Experiment 7 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

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

Principle

Potash alum is KAl(SO4)2·12H2O. 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.
- 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: $K_2SO_4 + Al_2(SO_4)_3 + 24\ H_2O \rightarrow 2\ KAl(SO_4)_2 \cdot 12H_2O$.
- Illustrative equimolar amounts: 1.743 g K_2SO_4 ≈ 0.010 mol and 6.664 g $Al_2(SO_4)_3 \cdot 18H_2O$ ≈ 0.010 mol. Theoretical alum = 0.020 × 474.39 ≈ 9.49 g.
- If 9.10 g crystals are recovered on the intended hydrate basis, yield = 9.10 ÷ 9.49 × 100 ≈ 95.9%.

Result

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

Precautions

- Use hydrate-corrected reactant masses.
- 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.

Preparation of Ferrous Sulphate

Experiment 8 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To prepare ferrous sulphate crystals and explain oxidation control and the heptahydrate yield basis.

Principle

Iron reacts with suitable dilute sulphuric acid to form $FeSO_4$ and hydrogen. Ferrous iron can oxidise to ferric iron on exposure. Crystallisation commonly gives $FeSO_4 \cdot 7H_2O$, so yield calculations must specify the hydrate.

Requirements

- Prescribed iron material, dilute sulphuric acid and water.
- Instructor-supervised ventilated reaction setup, beakers, filtration, cooling and balance.

Procedure

1. Calculate

Use the assigned iron amount and identify the limiting reactant. Write the target hydrate formula.

2. Observe reaction

Under the prescribed supervised procedure, react iron with the specified dilute acid. Keep the gas-evolving system unsealed and away from ignition sources.

3. Clarify

Separate residual solid iron or insoluble material as directed. Keep the solution under the prescribed conditions that limit oxidation.

4. Crystallise

Concentrate and cool by the assigned method to form crystals without uncontrolled overheating.

5. Collect and assess

Separate and surface-dry as prescribed. Record pale-green appearance, any yellow-brown material and the mass on the stated hydrate basis.

Observations and Record

Item	Observation / value	Record
Reaction	Gas evolution and completion	_____
Crystals	Colour and appearance	_____
Mass / yield	Hydrate basis	___ g / ___%

Worked Example

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

- Reaction: Fe + H_2SO_4 → $FeSO_4$ + H_2.
- Ideal example: 1.00 g Fe = 1.00/55.85 mol. Theoretical $FeSO_4 \cdot 7H_2O$ = (1.00/55.85) × 278.01 ≈ 4.98 g.
- Recovered 4.50 g on the heptahydrate basis gives approximately 90.4% yield. Surface water or a different hydrate invalidates this simple calculation.

Result

Crystals were obtained with appearance ____. Recovered mass = ____ g on the ____ hydrate basis; yield = ____%.

Precautions

- Hydrogen evolution requires the prescribed ventilation and absence of ignition sources.
- Do not seal the reacting vessel or use a direct flame.
- Limit oxidation and record discolouration instead of assuming all recovered material is Fe(II) salt.

Viva Questions and Answers

1. Why is hydrogen produced?

Iron displaces hydrogen from the suitable acid reaction.

2. Why can colour change on storage?

Ferrous material can oxidise and form ferric-related products.

3. Why use 278.01 g/mol in the example?

The yield is expressed as $FeSO_4 \cdot 7H_2O$, not anhydrous $FeSO_4$.

Magnesium Sulphate from Hydroxide or Carbonate

Experiment 9 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

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 $Mg(OH)_2$, $MgCO_3$ 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.

- Hydroxide: $Mg(OH)_2 + H_2SO_4 \rightarrow MgSO_4 + 2\ H_2O$. Carbonate: $MgCO_3 + H_2SO_4 \rightarrow MgSO_4 + CO_2 + H_2O$.
- For an ideal 2.00 g $Mg(OH)_2$ portion, theoretical $MgSO_4 \cdot 7H_2O = 2.00 \times 246.47/58.32 \approx 8.45$ 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.

Swelling Power of Bentonite

Experiment 10 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To assess bentonite swelling under the prescribed test conditions and report the correct volume basis.

Principle

Bentonite hydrates and expands in water. Swelling depends on mineral composition, particle handling, water quality and time. The monograph may specify a settled or swollen volume for a fixed mass rather than a normalised mL/g value; use the exact stated reporting basis.

Requirements

- Bentonite, prescribed water, balance and specified graduated cylinder.
- Timer and vibration-free standing area.

Procedure

1. Identify the method

Record the prescribed bentonite mass, cylinder capacity, water amount, addition sequence and reading time.

2. Disperse

Add small portions at the stated intervals to allow wetting. Do not dump a large dry lump into the water and call it a valid dispersion.

3. Stand

Keep under the prescribed conditions for the defined period. Avoid changing agitation or temperature during comparison.

4. Read

Read the stated swollen or settled volume at the defined endpoint. Distinguish material volume from the total liquid volume.

5. Report

Compare the specified volume result with the IP criterion supplied for the material. Calculate mL/g only if required or clearly label it as an additional derived value.

Observations and Record

Item	Condition / measurement	Record
Material / water	____ g / ____ mL	_____
Time / cylinder	_____	_____
Swollen volume	Defined endpoint	____ mL

Worked Example

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

- Illustrative measurement: 2.0 g bentonite occupies 28 mL under the stated method. Report 28 mL for 2.0 g when that is the prescribed basis.
- An additional derived swelling value is 28 ÷ 2.0 = 14 mL/g. It must not replace a criterion expressed as volume for a fixed portion without explanation.

Result

Swelling power = ____ mL for ____ g at ____ time, under the prescribed conditions. Comparison with the specified criterion: ____.

Precautions

- Use the stated water quality, addition sequence and standing time.
- A trapped dry clump is not a valid fully hydrated portion.
- Read the defined volume consistently and avoid vibration.

Viva Questions and Answers

1. Why add gradually?

It supports reproducible wetting and reduces dry lumps.

2. Why state the mass basis?

Volume for a fixed portion and volume per gram are different report formats.

3. Does swelling alone establish all bentonite quality attributes?

No; other identity and purity tests may also be required.

Acid-neutralising Capacity of Aluminium Hydroxide Gel

Experiment 11 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To calculate acid-neutralising capacity from added acid and a prescribed back-titration.

Principle

A known amount of acid reacts with the gel under defined conditions. Remaining acid is determined by back-titration to a specified endpoint. Acid consumed = acid added - acid remaining. For a monovalent hydrogen-ion basis, mmol of H+ corresponds numerically to mEq; express capacity per stated mass or dose volume.

Requirements

- Aluminium hydroxide gel, standard hydrochloric acid and standard sodium hydroxide.
- Calibrated pH measurement or specified endpoint system, burette, pipette, balance and timer.

Procedure

1. Record the basis

Homogenise as prescribed and measure the stated gel mass or volume. Record both titrant concentrations and the endpoint criterion.

2. Add known acid

Add the measured standard-acid quantity and mix under the method's temperature and reaction-time conditions.

3. Back-titrate

Titrate the remaining acid with standard base to the prescribed endpoint, not an arbitrary colour change.

4. Check controls

Run the prescribed blank or control and replicate preparation. Record any endpoint or dispersion difficulty.

5. Calculate

Use acid and base concentrations with consistent volumes. Apply the prescribed blank correction and express capacity on the stated gel basis.

Observations and Record

Quantity	Unit	Record
Gel portion	g or mL	____
Acid added	Concentration × volume	____
Base titre / endpoint	Concentration × volume; pH or indicator	____

Worked Example

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

- Illustrative 1.00 g gel: add 30.0 mL of 1.00 mol/L HCl; back-titrate with 15.0 mL of 0.500 mol/L NaOH.
- Acid added = 30.0 mmol; remaining acid = 7.50 mmol; consumed = 22.5 mmol H+ = 22.5 mEq. Capacity = 22.5 mEq/g.
- Using C in mol/L and V in mL gives mmol numerically: capacity = (CaVa - CbVb) ÷ mass. The method's blank and endpoint still govern the actual calculation.

Result

Acid-neutralising capacity = ____ mEq per ____ of gel, using endpoint ____ and reaction conditions ____.

Precautions

- Use the prescribed reaction time and endpoint; slow neutralisation can alter the result.
- Do not confuse measured wet-gel mass with dry Al(OH)3 mass.
- Do not infer a clinical dose from a classroom capacity calculation.

Viva Questions and Answers

1. Why use back-titration?

It measures the excess acid after the gel has reacted.

2. What is mEq on this basis?

For H+, one mmol has one mEq of charge.

3. Why define the endpoint?

Different endpoints can give different estimates of residual acid.

Potassium Iodate and Iodine in Potassium Iodide

Experiment 12 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To distinguish tests for free iodine and iodate impurity in the prescribed potassium iodide preparation.

Principle

Free iodine gives a characteristic colour with starch under the test conditions. In acid, iodate reacts with iodide to liberate iodine: $IO_3^- + 5\ I^- + 6\ H^+ \rightarrow 3\ I_2 + 3\ H_2O$. Separate observations before and after acidification help distinguish existing iodine from newly liberated iodine.

Requirements

- Potassium iodide test portion, fresh prescribed starch indicator and specified acid.
- Appropriate blanks and impurity standards, water and matched test vessels.

Procedure

1. Prepare the material

Dissolve the prescribed KI portion and inspect the initial appearance. Prepare separate aliquots for the tests.

2. Test free iodine

Apply the specified starch test to an aliquot without the iodate-liberation stage. Record any colour against its prescribed blank or comparator.

3. Test iodate

Use another aliquot and the prescribed acidification stage. Record newly developed iodine-starch colour at the specified time.

4. Review validity

Consider pre-existing iodine and reagent contamination. Use the method's separate controls rather than interpreting every blue colour as iodate.

5. Report

State the individual iodine and iodate observations and compare with the prescribed purity criteria.

Observations and Record

Aliquot / stage	Observed colour	Interpretation
Free-iodine test	_____	_____
Iodate test after acidification	_____	_____
Blank / standard	_____	Valid / invalid: ____

Worked Example

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

- Reaction ratio: 1 mol iodate releases 3 mol iodine in excess iodide and acid.
- In a separate validated iodometric calculation, those 3 mol iodine consume 6 mol thiosulphate. Thus 6 mol thiosulphate corresponds to 1 mol iodate.
- This ratio explains chemistry; the visual purity test itself must use its prescribed comparator and limit.

Result

Free-iodine test: ____. Iodate test: ____. Control validity and comparison with the prescribed purity criteria: ____.

Precautions

- Use separate aliquots and fresh specified indicator to reduce interpretation errors.
- Acidification conditions and reading time must match the method.
- Do not mistake pre-existing iodine for iodate-derived iodine.

Viva Questions and Answers

1. Why is acid needed in the iodate test?

The reaction that liberates iodine consumes hydrogen ions.

2. What does starch detect?

The iodine-containing complex that gives the characteristic colour.

3. Why test free iodine separately?

It may otherwise obscure the interpretation of an acidified iodate test.

Ammonium Chloride by Acid-base Titration

Experiment 13 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To standardise the stated titrant and calculate the content of the named compound from a prescribed titration.

Principle

In a formol teaching assay, formaldehyde converts ammonium species to hexamethylenetetramine and releases an equivalent amount of acid, which is titrated with standard sodium hydroxide. The formaldehyde reagent is neutralised or blank-corrected as required. This is a named teaching route; use the institution's prescribed acid-base assay if different.

Reaction and Calculation Basis

- Teaching reaction: $4\ NH_4Cl + 6\ HCHO \rightarrow C_6H_{12}N_4 + 4\ HCl + 6\ H_2O$.
- NH_4Cl molar mass ≈ 53.49 g/mol. For corrected volume V in mL and NaOH concentration C in mol/L, mass NH_4Cl in mg = V × C × 53.49.

Requirements

- Prescribed assay method, balance, burette, pipette, volumetric glassware and labelled test portion.
- Titrant: Approximately 0.1 mol/L sodium hydroxide.
- The primary standard or certified standard, prescribed medium and endpoint system named below.

Standardisation of the Titrant

1. Prepare the standard

Use certified potassium hydrogen phthalate, KHP, on its stated purity and drying basis. Accurately weigh and dissolve in the specified water amount.

2. Titrate

Titrate with NaOH using the prescribed phenolphthalein or potentiometric endpoint. Record blank correction and obtain the required concordant results.

3. Calculate

KHP reacts 1:1 with NaOH. C_{NaOH} = mass KHP × purity fraction ÷ (204.22 × NaOH volume in litres). Store and use the titrant as prescribed to limit carbon dioxide uptake.

Procedure

1. Check the method

Confirm that the laboratory uses the formol route and record the required reagent quantities, endpoint and test-portion basis.

2. Prepare the test

Accurately weigh and dissolve ammonium chloride in the prescribed water. The instructor provides the specified neutralised formaldehyde reagent.

3. Liberate titratable acid

Add the formaldehyde reagent and allow the prescribed reaction conditions. Run the specified matching blank.

4. Titrate

Titrate to the stated endpoint with standardised NaOH, recording initial and final readings.

5. Calculate

Use the blank-corrected volume and one mole NaOH per mole ammonium chloride under the valid reaction conditions.

Observations and Record

Record	Standardisation	Assay
Standard / test mass	____ g	____ g
Initial / final burette	____ / ____ mL	____ / ____ mL
Blank / corrected titre	____ / ____ mL	____ / ____ mL
Concentration / result	____ mol/L	____% on stated basis

Worked Example

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

- Standardisation example: 0.40844 g KHP (purity assumed 1.000) consumes 20.00 mL NaOH. C = (0.40844/204.22)/0.02000 = 0.1000 mol/L.
- For a 0.1000 g NH_4Cl test, corrected NaOH titre = 18.70 mL at 0.1000 mol/L. Mass found = 18.70 × 0.1000 × 53.49 = 100.03 mg.
- Assay ≈ 100.03/100.0 × 100 = 100.0%. A result near 100% is not automatically a pass without the prescribed criterion.

Result

Titrant concentration = ____ mol/L. Corrected titre = ____ mL. Calculated content = ____% on the ____ basis; prescribed criterion comparison: ____.

Precautions

- Formaldehyde requires the laboratory's ventilation and handling controls.
- A reagent blank is important; acidic reagent contamination otherwise biases the assay.
- Do not use a simple direct NaOH endpoint for ammonium chloride without the prescribed reaction or validated alternative method.
- Use the actual standardised concentration, correct for the prescribed blank and account for any aliquot or dilution. The monograph defines acceptance limits and the as-is, dried or hydrated basis.

Viva Questions and Answers

1. Why add formaldehyde in this method?

It makes ammonium content measurable through the corresponding released acid.

2. Why standardise NaOH?

Its effective concentration can change, including through carbon dioxide absorption.

3. What is the equivalence at 0.1 mol/L?

1 mL corresponds to approximately 5.349 mg NH_4Cl in this 1:1 method.

Ferrous Sulphate by Cerimetry

Experiment 14 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To standardise the stated titrant and calculate the content of the named compound from a prescribed titration.

Principle

Cerium(IV) oxidises Fe(II) to Fe(III) in the prescribed acidic medium. One mole of Ce(IV) accepts one electron from one mole of Fe(II). The hydrate formula and oxidation state of the test material must match the calculation.

Reaction and Calculation Basis

- $Fe^{2+} + Ce^{4+} \rightarrow Fe^{3+} + Ce^{3+}$.
- On the $FeSO_4 \cdot 7H_2O$ basis: mass in mg = V(mL) × C(mol/L) × 278.01.

Requirements

- Prescribed assay method, balance, burette, pipette, volumetric glassware and labelled test portion.
- Titrant: Approximately 0.1 mol/L cerium(IV) titrant.
- The primary standard or certified standard, prescribed medium and endpoint system named below.

Standardisation of the Titrant

1. Choose the standard

Use a certified Fe(II) standard or prescribed ferrous ammonium sulphate standard on its stated purity and hydrate basis.

2. Prepare

Prepare the standard in the specified acid medium, limiting oxidation during preparation.

3. Titrate and calculate

Titrate using the prescribed redox indicator or potentiometric endpoint. CCe(IV) = moles Fe(II) ÷ corrected titrant volume in litres. Obtain the required concordant results.

Procedure

1. Identify the material

Record whether the test is $FeSO_4 \cdot 7H_2O$ or another stated basis. Check appearance and oxidation concerns.

2. Dissolve promptly

Accurately weigh and dissolve in the prescribed acidic medium. Avoid unnecessary delay and exposure before titration.

3. Titrate

Use standardised Ce(IV) and the specified endpoint system. Record titre and any required blank.

4. Calculate

For heptahydrate use 278.01 g/mol and the 1:1 mole ratio. Apply any monograph-specific basis correction.

Observations and Record

Record	Standardisation	Assay
Standard / test mass	____ g	____ g
Initial / final burette	____ / ____ mL	____ / ____ mL
Blank / corrected titre	____ / ____ mL	____ / ____ mL
Concentration / result	____ mol/L	____% on stated basis

Worked Example

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

- Illustrative standard: 0.39214 g pure ferrous ammonium sulphate hexahydrate, approximately 392.14 g/mol, supplies 1.000 mmol Fe(II). A 10.00 mL titre gives 0.1000 mol/L Ce(IV).
- For 0.5000 g $FeSO_4 \cdot 7H_2O$, corrected titre 18.00 mL at 0.1000 mol/L gives 18.00 × 0.1000 × 278.01 = 500.42 mg.
- Assay = 500.42/500.0 × 100 ≈ 100.1% on the heptahydrate basis.

Result

Titrant concentration = ____ mol/L. Corrected titre = ____ mL. Calculated content = ____% on the ____ basis; prescribed criterion comparison: ____.

Precautions

- Fe(II) oxidation before titration can lower the measured ferrous content.
- Use the prescribed acidic medium and endpoint; do not choose an indicator solely by colour preference.
- Do not use anhydrous $FeSO_4$ mass in a heptahydrate calculation.
- Use the actual standardised concentration, correct for the prescribed blank and account for any aliquot or dilution. The monograph defines acceptance limits and the as-is, dried or hydrated basis.

Viva Questions and Answers

1. Why is the mole ratio 1:1?

Both changes involve one electron per reacting ion.

2. What oxidation change occurs?

Fe(II) becomes Fe(III), while Ce(IV) becomes Ce(III).

3. What is 1 mL of 0.1 mol/L equivalent to?

Approximately 27.801 mg $FeSO_4 \cdot 7H_2O$.

Copper Sulphate by Iodometry

Experiment 15 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To standardise the stated titrant and calculate the content of the named compound from a prescribed titration.

Principle

Cu(II) reacts with iodide to precipitate CuI and release iodine. The iodine is titrated with sodium thiosulphate. Combining the reaction ratios gives one mole of thiosulphate per mole of copper. Starch is added near the endpoint rather than into a strongly iodine-rich mixture.

Reaction and Calculation Basis

- $2\ Cu^{2+} + 4\ I^- \rightarrow 2\ CuI + I_2$.
- $I_2 + 2\ S_2O_3^{2-} \rightarrow 2\ I^- + S_4O_6^{2-}$.
- For $CuSO_4 \cdot 5H_2O$, mass in mg = V × C × 249.68.

Requirements

- Prescribed assay method, balance, burette, pipette, volumetric glassware and labelled test portion.
- Titrant: Approximately 0.1 mol/L sodium thiosulphate.
- The primary standard or certified standard, prescribed medium and endpoint system named below.

Standardisation of the Titrant

1. Use an iodate standard

Accurately weigh certified potassium iodate on its stated purity basis and dissolve as prescribed.

2. Liberate iodine

Add the specified excess iodide and acid in the approved iodometric flask. Carry out the prescribed blank and protect the reaction from unnecessary light and delay.

3. Titrate

Titrate liberated iodine with thiosulphate; add starch near the endpoint. One mole iodate consumes six moles thiosulphate. Cthio = 6 × moles iodate ÷ corrected volume in litres.

Procedure

1. Prepare the test

Accurately weigh $CuSO_4 \cdot 5H_2O$ and prepare in the monograph's medium. Control acidity and interferences as prescribed.

2. Release iodine

Add the stated excess iodide, mix and observe iodine colour with CuI precipitate.

3. Titrate promptly

Titrate with standardised thiosulphate until the prescribed near-endpoint stage, then add starch.

4. Complete the endpoint

Continue to disappearance of the iodine-starch colour. If the prescribed method includes a near-endpoint thiocyanate step to release adsorbed iodine, apply it only at that stage.

5. Calculate

Use corrected titre and the pentahydrate molecular mass. Distinguish a redox endpoint from simple disappearance of precipitate.

Observations and Record

Record	Standardisation	Assay
Standard / test mass	____ g	____ g
Initial / final burette	____ / ____ mL	____ / ____ mL
Blank / corrected titre	____ / ____ mL	____ / ____ mL
Concentration / result	____ mol/L	____% on stated basis

Worked Example

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

- Standard example: 0.07133 g KIO3, approximately 214.00 g/mol, gives 6 × 0.07133/214.00 mol thiosulphate equivalents. A 20.00 mL titre gives approximately 0.1000 mol/L.
- A 0.5000 g CuSO4·5H2O portion with corrected titre 20.00 mL at 0.1000 mol/L gives 20.00 × 0.1000 × 249.68 = 499.36 mg.
- Assay = 499.36/500.0 × 100 ≈ 99.87%.

Result

Titrant concentration = ____ mol/L. Corrected titre = ____ mL. Calculated content = ____% on the ____ basis; prescribed criterion comparison: ____.

Precautions

- Prevent iodine loss by unnecessary delay or open-vessel exposure.
- Add starch at the prescribed near-endpoint stage.
- Follow the specified acidity and handling of iodine adsorbed by CuI.
- Use the actual standardised concentration, correct for the prescribed blank and account for any aliquot or dilution. The monograph defines acceptance limits and the as-is, dried or hydrated basis.

Viva Questions and Answers

1. Why is iodometry indirect here?

The copper reaction releases iodine, and the iodine is what is titrated.

2. Why is the copper:thiosulphate ratio 1:1?

Two copper ions release one iodine molecule, which consumes two thiosulphate ions.

3. What is the pentahydrate factor at 0.1 mol/L?

Approximately 24.968 mg CuSO4·5H2O per mL.

Calcium Gluconate by Complexometry

Experiment 16 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To standardise the stated titrant and calculate the content of the named compound from a prescribed titration.

Principle

EDTA complexes calcium in a 1:1 mole ratio under the prescribed conditions. The endpoint indicator responds to the availability of calcium at the selected pH. Calcium gluconate monohydrate and anhydrous calcium gluconate have different formula masses.

Reaction and Calculation Basis

- Ca^{2+} + EDTA → calcium-EDTA complex; mole ratio 1:1.
- Monohydrate formula mass ≈ 448.39 g/mol. Mass in mg = V × C × 448.39.

Requirements

- Prescribed assay method, balance, burette, pipette, volumetric glassware and labelled test portion.
- Titrant: Approximately 0.05 mol/L EDTA.
- The primary standard or certified standard, prescribed medium and endpoint system named below.

Standardisation of the Titrant

1. Prepare calcium standard

Use a certified calcium standard or the prescribed calcium carbonate primary standard. For carbonate, dissolve using the specified acid treatment and handle carbon dioxide as directed.

2. Set conditions

Adjust to the specified complexometric pH and add the prescribed calcium endpoint indicator.

3. Titrate and calculate

Titrate with EDTA to the stated endpoint. CEDTA = moles calcium ÷ corrected EDTA volume in litres; obtain the required concordance.

Procedure

1. Confirm hydrate

Record the test-material formula and assay basis. Weigh and dissolve by the prescribed method.

2. Set pH

Establish the stated calcium-complexation conditions and add the specified indicator, such as the institution's prescribed murexide system.

3. Titrate

Use standardised EDTA and mix consistently. Recognise the defined indicator endpoint rather than an arbitrary faint colour.

4. Calculate

Use one mole EDTA per mole calcium gluconate and the formula mass for the stated hydrate. Apply any dilution or blank correction.

Observations and Record

Record	Standardisation	Assay
Standard / test mass	____ g	____ g
Initial / final burette	____ / ____ mL	____ / ____ mL
Blank / corrected titre	____ / ____ mL	____ / ____ mL
Concentration / result	____ mol/L	____% on stated basis

Worked Example

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

- Standardisation example: 0.10009 g pure CaCO3, 100.09 g/mol, gives 1.000 mmol calcium. A 20.00 mL EDTA titre gives 0.05000 mol/L.
- For 0.5000 g calcium gluconate monohydrate, corrected titre 22.30 mL at 0.05000 mol/L gives 22.30 × 0.05000 × 448.39 = 499.95 mg.
- Assay ≈ 99.99% on the monohydrate basis. Anhydrous material uses approximately 430.37 g/mol instead.

Result

Titrant concentration = ____ mol/L. Corrected titre = ____ mL. Calculated content = ____% on the ____ basis; prescribed criterion comparison: ____.

Precautions

- Use the prescribed pH and calcium-selective endpoint system.
- Other metal ions can interfere; the method's preparation and controls matter.
- Do not use the monohydrate factor for anhydrous material.
- Use the actual standardised concentration, correct for the prescribed blank and account for any aliquot or dilution. The monograph defines acceptance limits and the as-is, dried or hydrated basis.

Viva Questions and Answers

1. What is the calcium:EDTA ratio?

1:1 in the stated complexometric calculation.

2. Why control pH?

Complex stability and indicator behaviour depend on it.

3. What is the 0.05 mol/L monohydrate factor?

Approximately 22.4195 mg per mL.

Hydrogen Peroxide by Permanganometry

Experiment 17 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To standardise the stated titrant and calculate the content of the named compound from a prescribed titration.

Principle

In the prescribed acidic medium, permanganate oxidises hydrogen peroxide to oxygen while being reduced to Mn(II). Permanganate provides its own colour endpoint. The acidity and reaction conditions must suit the validated method; chloride-containing acid is not substituted for the prescribed sulphuric-acid medium.

Reaction and Calculation Basis

- $2\ MnO_4^- + 5\ H_2O_2 + 6\ H^+ \rightarrow 2\ Mn^{2+} + 5\ O_2 + 8\ H_2O$.
- H_2O_2 formula mass ≈ 34.014 g/mol. Mass in mg = V × C × (5/2) × 34.014.

Requirements

- Prescribed assay method, balance, burette, pipette, volumetric glassware and labelled test portion.
- Titrant: Approximately 0.02 mol/L potassium permanganate.
- The primary standard or certified standard, prescribed medium and endpoint system named below.

Standardisation of the Titrant

1. Use a primary standard

Accurately weigh certified sodium oxalate on the stated purity basis and dissolve in the prescribed acidic medium.

2. Establish reaction conditions

Apply the method's controlled temperature and addition conditions; oxalate-permanganate reaction rate matters.

3. Titrate

Titrate to the stated persistent faint-pink endpoint. Two moles permanganate react with five moles oxalate. C_{KMnO_4} = (2/5) × moles oxalate ÷ corrected titrant volume in litres.

Procedure

1. Prepare the peroxide

Accurately weigh or measure the prescribed test portion, retaining its mass or volume basis. Dilute as directed and account for any aliquot.

2. Acidify

Use the specified sulphuric-acid medium and reaction conditions. Do not substitute hydrochloric acid.

3. Titrate

Add standardised permanganate with mixing to the prescribed faint-pink endpoint and persistence time.

4. Calculate

Use the 5:2 peroxide-to-permanganate mole ratio. Express peroxide content on the stated w/w or w/v basis.

Observations and Record

Record	Standardisation	Assay
Standard / test mass	____ g	____ g
Initial / final burette	____ / ____ mL	____ / ____ mL
Blank / corrected titre	____ / ____ mL	____ / ____ mL
Concentration / result	____ mol/L	____% on stated basis

Worked Example

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

- Standardisation example: 0.06700 g $Na_2C_2O_4$, approximately 134.00 g/mol, is 0.5000 mmol oxalate. It reacts with 0.2000 mmol $KMnO_4$. A 10.00 mL titre gives 0.02000 mol/L.
- Peroxide factor: 1 mL of 0.02000 mol/L $KMnO_4$ corresponds to 1.7007 mg H_2O_2.
- If 1.000 g test solution consumes a corrected 17.64 mL, mass H_2O_2 ≈ 30.00 mg; content = 30.00/1000 × 100 = 3.000% w/w.

Result

Titrant concentration = ____ mol/L. Corrected titre = ____ mL. Calculated content = ____% on the ____ basis; prescribed criterion comparison: ____.

Precautions

- Use the prescribed acid and endpoint conditions.
- Avoid contamination that decomposes peroxide before assay.
- Record whether the test portion was weighed or measured; w/w and w/v are different bases.
- Use the actual standardised concentration, correct for the prescribed blank and account for any aliquot or dilution. The monograph defines acceptance limits and the as-is, dried or hydrated basis.

Viva Questions and Answers

1. Why is no separate colour indicator required?

A slight excess of permanganate gives the defined pink endpoint.

2. What is the reaction ratio?

Five moles H_2O_2 to two moles permanganate in the specified acidic reaction.

3. Why avoid an arbitrary acid substitution?

The substituted medium may undergo side reactions or change the valid assay conditions.

Sodium Benzoate by Non-aqueous Titration

Experiment 18 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To standardise the stated titrant and calculate the content of the named compound from a prescribed titration.

Principle

Sodium benzoate is titrated with perchloric acid in the prescribed non-aqueous medium. The medium supports measurement of a weak-base salt that may not give a satisfactory ordinary aqueous endpoint. One mole of sodium benzoate corresponds to one mole of acid under the stated method.

Reaction and Calculation Basis

- $C_6H_5COONa + HClO_4 \rightarrow C_6H_5COOH + NaClO_4$.
- Mass sodium benzoate in mg = V(mL) × C(mol/L) × 144.11.

Requirements

- Prescribed assay method, balance, burette, pipette, volumetric glassware and labelled test portion.
- Titrant: Approximately 0.1 mol/L perchloric acid in the prescribed non-aqueous system.
- The primary standard or certified standard, prescribed medium and endpoint system named below.

Standardisation of the Titrant

1. Use the standard

Use certified KHP on the stated purity and preparation basis, dissolved in the prescribed non-aqueous solvent.

2. Check the titrant

Use the instructor-provided or commercially standardised reagent. Students do not prepare concentrated perchloric-acid mixtures.

3. Titrate

Use the prescribed potentiometric or validated visual endpoint and solvent blank. $CHClO_4$ = moles KHP ÷ corrected volume in litres, with the method's temperature correction if required.

Procedure

1. Prepare the portion

Accurately weigh sodium benzoate on the stated assay basis. Use the specified dry non-aqueous solvent and dissolution method.

2. Set the endpoint

Use the prescribed electrode system or indicator; do not substitute an aqueous pH interpretation without validation.

3. Titrate

Titrate with the standardised reagent and run the specified solvent blank under matched conditions.

4. Calculate

Use the blank-corrected titre, actual acid concentration and 144.11 g/mol formula mass. Apply any specified temperature or basis correction.

Observations and Record

Record	Standardisation	Assay
Standard / test mass	____ g	____ g
Initial / final burette	____ / ____ mL	____ / ____ mL
Blank / corrected titre	____ / ____ mL	____ / ____ mL
Concentration / result	____ mol/L	____% on stated basis

Worked Example

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

- Illustrative KHP standard: 0.40844 g / 204.22 g/mol = 2.000 mmol. A corrected 20.00 mL titre gives 0.1000 mol/L acid.
- A 0.2000 g sodium benzoate portion gives a corrected 13.88 mL at 0.1000 mol/L. Mass found = 13.88 × 0.1000 × 144.11 ≈ 200.03 mg.
- Assay ≈ 100.0%. At 0.1 mol/L, each mL is equivalent to 14.411 mg sodium benzoate.

Result

Titrant concentration = ____ mol/L. Corrected titre = ____ mL. Calculated content = ____% on the ____ basis; prescribed criterion comparison: ____.

Precautions

- Perchloric-acid reagents require the laboratory's designated facilities and handling controls.
- Use the provided titrant and approved solvent system; do not heat or concentrate perchloric-acid mixtures.
- Water content and temperature can affect the non-aqueous method; follow its specified conditions.
- Use the actual standardised concentration, correct for the prescribed blank and account for any aliquot or dilution. The monograph defines acceptance limits and the as-is, dried or hydrated basis.

Viva Questions and Answers

1. Why use a non-aqueous medium?

It provides suitable acid-base behaviour and a measurable endpoint for the prescribed assay.

2. Why run a solvent blank?

The solvent or reagents may consume part of the titrant.

3. What is the stoichiometric ratio?

One mole acid per mole sodium benzoate under the stated method.

Sodium Chloride by Modified Volhard Method

Experiment 19 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To standardise the stated titrant and calculate the content of the named compound from a prescribed titration.

Principle

A known excess of silver nitrate precipitates chloride as AgCl. The remaining silver is back-titrated with thiocyanate in the prescribed acidic medium. Once silver is consumed, excess thiocyanate gives a ferric-thiocyanate colour. The modification prevents AgCl from interfering with the back-titration.

Reaction and Calculation Basis

- $Ag^+ + Cl^- \rightarrow AgCl$; $Ag^+ + SCN^- \rightarrow AgSCN$.
- If a matched blank is valid, NaCl mass in mg = (Vblank - Vtest) × CSCN × 58.44.
- General direct calculation: NaCl mass in mg = (CAgVAg - CSCNVSCN) × 58.44, for concentrations in mol/L and volumes in mL.

Requirements

- Prescribed assay method, balance, burette, pipette, volumetric glassware and labelled test portion.
- Titrant: Standard silver nitrate and approximately 0.1 mol/L ammonium thiocyanate.
- The primary standard or certified standard, prescribed medium and endpoint system named below.

Standardisation of the Titrant

1. Standardise silver nitrate

Use certified sodium chloride on its specified preparation basis. Titrate the known chloride amount with $AgNO_3$ by the laboratory's validated endpoint, such as potentiometric detection. CAg = moles NaCl ÷ corrected volume in litres.

2. Standardise thiocyanate

Take a measured standard $AgNO_3$ aliquot, establish the prescribed acidic conditions and ferric indicator, then titrate with thiocyanate to the defined endpoint.

3. Calculate

The Ag:SCN ratio is 1:1. CSCN = CAg × VAg ÷ corrected VSCN when the volumes share the same units. Obtain the required concordant results.

Procedure

1. Prepare the test

Accurately weigh and dissolve NaCl. Add the prescribed nitric-acid medium and a precisely measured excess of standard AgNO3.

2. Manage the precipitate

Apply the laboratory's approved modified-Volhard strategy to prevent AgCl consuming thiocyanate. Where the validated teaching route separates AgCl, filter quantitatively and retain the prescribed filtrate and washings.

3. Back-titrate

Add the prescribed ferric indicator and titrate excess silver with standard thiocyanate to the persistent defined reddish complex colour.

4. Run the blank

Use the same silver addition and relevant reagent conditions without chloride, following the method's blank procedure.

5. Calculate

Use either silver added minus residual silver, or the correctly matched blank-test titre difference. Keep both titrant concentrations when they differ.

Observations and Record

Record	Standardisation	Assay
Standard / test mass	____ g	____ g
Initial / final burette	____ / ____ mL	____ / ____ mL
Blank / corrected titre	____ / ____ mL	____ / ____ mL
Concentration / result	____ mol/L	____% on stated basis

Worked Example

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

- AgNO3 standardisation: 0.14610 g pure NaCl / 58.44 = 2.500 mmol; a 25.00 mL titre gives 0.1000 mol/L AgNO3.
- If 10.00 mL of 0.1000 mol/L AgNO3 consumes 10.00 mL thiocyanate, CSCN = 0.1000 mol/L.
- Matched-blank example: Vblank = 25.00 mL and Vtest = 8.00 mL at CSCN = 0.1000 mol/L. NaCl found = (25.00 - 8.00) × 0.1000 × 58.44 = 99.35 mg.
- For a 0.1000 g test portion, assay = 99.35%. Direct check: CAgVAg - CSCNVtest = 2.50 - 0.80 = 1.70 mmol chloride.

Result

Titrant concentration = ____ mol/L. Corrected titre = ____ mL. Calculated content = ____% on the ____ basis; prescribed criterion comparison: ____.

Precautions

- Follow the approved precipitate-protection or separation method; a casual endpoint reading in unprotected AgCl can be biased.
- Do not introduce an unapproved organic coating reagent.
- Keep silver-containing waste in the designated collection system.
- Use the actual standardised concentration, correct for the prescribed blank and account for any aliquot or dilution. The monograph defines acceptance limits and the as-is, dried or hydrated basis.

Viva Questions and Answers

1. Why is this a back-titration?

Chloride consumes part of a known silver excess, and the remaining silver is measured.

2. Why remove or protect AgCl?

AgCl can react with thiocyanate and cause extra titrant consumption.

3. What forms the endpoint colour?

A ferric-thiocyanate complex after the available silver has been consumed.

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