

Year 1 - Practical Notes

Pharmaceutical Chemistry

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Limit Test for Chlorides

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: $\text{Ag}^+ + \text{Cl}^- \rightarrow \text{AgCl}$. 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 $2.0 \times 20 = 40$ 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.

Limit Test for Sulphates

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: $\text{Ba}^{2+} + \text{SO}_4^{2-} \rightarrow \text{BaSO}_4$. 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.

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- 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.

Limit Test for Iron

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.

Limit Test for Heavy Metals

Experiment 4 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare a heavy-metal impurity response with the prescribed lead-equivalent standard.

Principle

The traditional sulphide method produces coloured metal sulphides under a prescribed pH. The response is compared with a lead standard and reported as lead-equivalent impurity; it is not an assay of each individual metal.

Requirements

- Assigned monograph, test material and lead standard
- Instructor-prepared sulphide reagent system and specified buffer
- Matched comparison tubes, pipettes and controlled ventilation

Procedure

1. Check the method

Identify the test mass, pretreatment, prescribed limit, buffer and reagent-development time.

2. Prepare test and standard

Prepare matched solutions of the test portion and the stated lead comparator. Include the required blank and sensitivity control.

3. Develop colour

Use the assigned buffered sulphide-reagent procedure under supervision; treat test and comparator equally.

4. Compare

At the prescribed time compare brown-to-dark colour under matched viewing conditions.

5. Record validity

Enter the response and state whether the blank and controls support a valid comparison.

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Observations and Record

Field	Record
Material / mass	_____
Lead-equivalent standard amount	_____
pH / development conditions	_____
Test versus comparator	_____
Control validity	_____

Worked Example

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

- Illustrative limit: 1.0 g material at an assigned 20 micrograms/g limit needs a comparator containing 20 micrograms lead equivalent.
- A 10 micrograms/mL standard supplies that amount in 2.0 mL; the actual monograph sets the limit.

Result

The test response was _____ relative to the comparator. Controls were _____. The material _____ the assigned limit.

Precautions

- Metal standards and sulphide reagents require designated ventilation, exposure controls and separate waste collection.
- Do not acidify sulphide waste outside the approved process.
- Do not interpret an invalid blank or poorly developed standard as a passing test.

Viva Questions and Answers**1. What does lead-equivalent mean?**

A response expressed relative to the prescribed lead comparator, rather than a separate amount for every metal.

2. Why control pH?

Sulphide formation and the response of different metals depend on pH.

3. Is this a modern elemental-impurity panel?

No; instrumental element-specific tests are separate methods.

Identification of Anions by Pharmacopoeial Tests

Experiment 5 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To identify assigned inorganic ions using characteristic and confirmatory reactions.

Principle

A screening reaction suggests an ion; a selective confirmation and appropriate controls strengthen identification. Colour, solubility, pH and interference affect reactions.

Characteristic Reactions

Ion	Expected teaching response
Chloride	Dilute nitric acid followed by silver nitrate gives white silver chloride; the precipitate dissolves in the prescribed ammonia test.
Sulphate	Acidified barium-reagent testing gives a white barium sulphate precipitate that persists in dilute mineral acid.
Carbonate	Dilute acid causes carbon dioxide effervescence; the gas gives the assigned limewater confirmation.
Phosphate	The assigned molybdate method gives a characteristic yellow response under its prescribed acidic conditions.

Requirements

- Assigned ion solutions, positive controls and reagent blank
- Prescribed reagents, test tubes, droppers and PPE

Procedure

1. Plan aliquots

Read each prescribed reaction and divide the unknown into separate labelled portions.

2. Screen

Observe initial colour, solubility and the assigned preliminary reaction.

3. Confirm

Perform the specified confirmation on a fresh portion; compare with positive and blank controls.

4. Record

Write the reagent, actual observation, inference and supporting reaction.

Pharmaceutical Chemistry

5. Conclude

Identify the ion only when the observed evidence and controls agree.

Observations and Record

Ion / reaction	Actual observation	Inference
Chloride	_____	_____
Sulphate	_____	_____
Carbonate	_____	_____
Phosphate	_____	_____

Result

The assigned ion was identified as _____ based on _____ and _____. Controls: _____.

Precautions

- Use clean equipment; sodium and chloride contamination are common.
- Use the approved ventilation for gas-evolving tests.
- Do not mix unknown reagents or infer identity from one nonspecific colour.

Viva Questions and Answers**1. Why use a fresh aliquot?**

Reagents from a previous test may interfere with the next reaction.

2. What is a confirmatory test?

A more selective reaction that supports the proposed identity.

3. Why include a blank?

It detects contamination and reagent background.

Identification of Cations by Pharmacopoeial Tests

Experiment 6 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To identify assigned inorganic ions using characteristic and confirmatory reactions.

Principle

A screening reaction suggests an ion; a selective confirmation and appropriate controls strengthen identification. Colour, solubility, pH and interference affect reactions.

Characteristic Reactions

Ion	Expected teaching response
Calcium	Oxalate testing gives a white calcium oxalate precipitate in the prescribed medium; a brick-red flame is supporting evidence.
Sodium	A strong yellow flame is supporting evidence but contamination is common; use the assigned monograph confirmation.
Potassium	A violet flame may be obscured by sodium; use the assigned confirmatory reagent, not flame colour alone.
Ammonium	The supervised alkaline test releases ammonia detected using moist indicator paper; never directly inhale evolved gas.
Copper	Copper(II) solutions commonly appear blue; prescribed ammonia addition produces a deep-blue complex.
Iron	Iron(III) gives the prescribed coloured thiocyanate response; oxidation state and interferences must be considered.

Requirements

- Assigned ion solutions, positive controls and reagent blank
- Prescribed reagents, test tubes, droppers and PPE

Procedure

1. Plan aliquots

Read each prescribed reaction and divide the unknown into separate labelled portions.

2. Screen

Observe initial colour, solubility and the assigned preliminary reaction.

Pharmaceutical Chemistry

3. Confirm

Perform the specified confirmation on a fresh portion; compare with positive and blank controls.

4. Record

Write the reagent, actual observation, inference and supporting reaction.

5. Conclude

Identify the ion only when the observed evidence and controls agree.

Observations and Record

Ion / reaction	Actual observation	Inference
Calcium	_____	_____
Sodium	_____	_____
Potassium	_____	_____
Ammonium	_____	_____
Copper	_____	_____
Iron	_____	_____

Result

The assigned ion was identified as _____ based on _____ and _____. Controls: _____.

Precautions

- Use clean equipment; sodium and chloride contamination are common.
- Use the approved ventilation for gas-evolving tests.
- Do not mix unknown reagents or infer identity from one nonspecific colour.

Viva Questions and Answers**1. Why use a fresh aliquot?**

Reagents from a previous test may interfere with the next reaction.

2. What is a confirmatory test?

A more selective reaction that supports the proposed identity.

3. Why include a blank?

It detects contamination and reagent background.

Preparation and Standardisation of Sodium Hydroxide

Experiment 7 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To prepare a nominal titrant and determine its actual strength.

Principle

Sodium hydroxide absorbs moisture and carbon dioxide, so a nominal solution is standardised against a suitable primary standard such as potassium hydrogen phthalate (KHP). One mole KHP reacts with one mole hydroxide.

Requirements

- Sodium hydroxide, suitable water and clean stoppered bottle
- Prescribed dried KHP, balance, burette and phenolphthalein

Procedure

1. Prepare nominal solution

Calculate the approximate mass required, dissolve carefully and cool before making to volume; use the assigned concentration.

2. Prepare primary standard

Weigh the prescribed KHP and dissolve it in the specified vehicle.

3. Titrate

Titrate with NaOH using the prescribed endpoint; repeat for concordant readings and apply any stated blank.

4. Calculate

Calculate the actual molarity and label concentration, factor, date and storage.

Observations and Record

Reading	Trial 1	Trial 2	Trial 3
Standard mass, g	_____	_____	_____
Initial burette, mL	_____	_____	_____
Final burette, mL	_____	_____	_____
Corrected titre, mL	_____	_____	_____

Worked Example

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

- $M(\text{NaOH}) = \text{mass KHP (g)} / [204.22 \text{ g/mol} \times \text{corrected titre (L)}]$.
- Example: 0.51055 g KHP and 25.00 mL NaOH gives 0.1000 mol/L.
- For nominal 0.1000 mol/L, factor = actual/nominal; here 1.000.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- NaOH is corrosive and dissolves exothermically; allow the solution to cool.
- Avoid carbon dioxide exposure and use the assigned storage closure.

Viva Questions and Answers

1. Why is weighing NaOH insufficient?

Absorbed moisture and carbonate change the effective hydroxide amount.

2. Why is KHP useful?

It is a suitable high-purity primary acid standard with a defined equivalent amount.

3. Why use concordant readings?

Agreement reduces random endpoint and reading error, but does not remove systematic errors.

Preparation and Standardisation of Potassium Permanganate

Experiment 8 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To prepare a nominal titrant and determine its actual strength.

Principle

Potassium permanganate is an oxidant and a self-indicator. In acidic oxalate standardisation, two permanganate ions react with five oxalate ions; one mole permanganate accepts five electrons.

Requirements

- Assigned potassium permanganate solution and primary-standard sodium oxalate
- Prescribed sulphuric-acid medium, controlled water bath and burette

Procedure

1. Prepare

Prepare the nominal permanganate solution and complete the assigned conditioning and clarification before standardisation.

2. Prepare standard

Weigh primary-standard oxalate and dissolve in the prescribed sulphuric-acid medium.

3. Control conditions

Use the method-specified warming; the oxalate reaction is slow at the beginning.

4. Titrate

Add titrant at the prescribed rate and determine the persistent pale-pink endpoint.

5. Repeat

Obtain concordant titres, calculate strength and label the protected storage bottle.

Observations and Record

Reading	Trial 1	Trial 2	Trial 3
Standard mass, g	_____	_____	_____
Initial burette, mL	_____	_____	_____
Final burette, mL	_____	_____	_____
Corrected titre, mL	_____	_____	_____

Worked Example

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

- $N(KMnO_4) = \text{mass sodium oxalate (g)} / [67.00 \text{ g/equivalent} \times \text{titre (L)}]$.
- Example: 0.16750 g sodium oxalate and 25.00 mL gives 0.1000 N, equivalent to 0.0200 M permanganate in this acidic reaction.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the specified acid; hydrochloric acid can introduce side reactions.
- Do not use organic filter paper in an improvised clarification process.
- Match temperature and endpoint conditions across repeats.

Viva Questions and Answers

1. Why is $KMnO_4$ a self-indicator?

A slight excess imparts the endpoint colour.

2. Why is 0.1 N not 0.1 M here?

Each permanganate ion accepts five electrons in the acidic reaction.

3. Why use concordant readings?

Agreement reduces random endpoint and reading error, but does not remove systematic errors.

Assay of Ferrous Sulphate by Redox Titration

Experiment 9 - 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

- $\text{Fe}^{2+} + \text{Ce}^{4+} \rightarrow \text{Fe}^{3+} + \text{Ce}^{3+}$.
- On the $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ basis: mass in mg = $V(\text{mL}) \times C(\text{mol/L}) \times 278.01$.

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. $C_{\text{Ce(IV)}} = \text{moles Fe(II)} \div \text{corrected titrant volume in litres}$. Obtain the required concordant results.

Procedure

1. Identify the material

Record whether the test is $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ 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.

Pharmaceutical Chemistry**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 FeSO₄·7H₂O, corrected titre 18.00 mL at 0.1000 mol/L gives $18.00 \times 0.1000 \times 278.01 = 500.42$ mg.
- Assay = $500.42/500.0 \times 100 \approx 100.1\%$ 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 FeSO₄ 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 FeSO₄·7H₂O.

Assay of Calcium Gluconate by Complexometric Titration

Experiment 10 - 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

- $\text{Ca}^{2+} + \text{EDTA} \rightarrow \text{calcium-EDTA complex}$; mole ratio 1:1.
- Monohydrate formula mass $\approx 448.39 \text{ g/mol}$. Mass in mg = $V \times C \times 448.39$.

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. $\text{CEDTA} = \text{moles calcium} \div \text{corrected EDTA volume in litres}$; 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.

Pharmaceutical Chemistry**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 CaCO_3 , 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 $22.30 \times 0.05000 \times 448.39 = 499.95$ mg.
- Assay $\approx 99.99\%$ 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.

Assay of Sodium Chloride by Modified Volhard Method

Experiment 11 - 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

- $\text{Ag}^+ + \text{Cl}^- \rightarrow \text{AgCl}$; $\text{Ag}^+ + \text{SCN}^- \rightarrow \text{AgSCN}$.
- If a matched blank is valid, NaCl mass in mg = $(V_{\text{blank}} - V_{\text{test}}) \times \text{CSCN} \times 58.44$.
- General direct calculation: NaCl mass in mg = $(C_{\text{Ag}}V_{\text{Ag}} - \text{CSCN}V_{\text{SCN}}) \times 58.44$, 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 AgNO₃ by the laboratory's validated endpoint, such as potentiometric detection. $C_{\text{Ag}} = \text{moles NaCl} \div \text{corrected volume in litres}$.

2. Standardise thiocyanate

Take a measured standard AgNO₃ 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. $\text{CSCN} = C_{\text{Ag}} \times V_{\text{Ag}} \div \text{corrected } V_{\text{SCN}}$ when the volumes share the same units. Obtain the required concordant results.

Pharmaceutical Chemistry**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: V_{blank} = 25.00 mL and V_{test} = 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: C_{AgVAg} - CSCNV_{test} = 2.50 - 0.80 = 1.70 mmol chloride.

Pharmaceutical Chemistry

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.

Assay of Ascorbic Acid by Iodine Titration

Experiment 12 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To determine ascorbic acid by reaction with standard iodine.

Principle

Ascorbic acid reduces iodine to iodide and is oxidised to dehydroascorbic acid. The mole ratio is 1:1. After the reducing substance is exhausted, excess iodine gives the starch endpoint.

Requirements

- Assigned ascorbic-acid preparation and freshly standardised iodine
- Prescribed acidic vehicle, starch reagent, burette and pipettes

Procedure

1. Weigh and dissolve

Prepare the assigned test amount promptly in the method-specified vehicle.

2. Titrate

Titrate with standard iodine under the stated conditions; use starch at the prescribed stage.

3. Identify endpoint

Record the prescribed persistent blue endpoint rather than a transient local colour.

4. Apply controls

Use the assigned blank and dilution factor; obtain concordant titres.

5. Calculate

Calculate iodine-equivalent ascorbic acid and express percentage on the specified basis.

Observations and Record

Field	Trial 1	Trial 2	Trial 3
Test mass, g	_____	_____	_____
Corrected titre, mL	_____	_____	_____
Iodine molarity	_____	_____	_____
Dilution factor	_____	_____	_____

Worked Example

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

- Mass ascorbic acid (mg) = corrected titre (mL) $\times$ iodine M $\times$ 176.12 $\times$ dilution factor.
- At 0.0500 M iodine (0.1000 N), each mL is equivalent to 8.806 mg ascorbic acid.
- Example: 20.00 mL represents 176.12 mg. If test mass is 180.00 mg without dilution, the assay is 97.84%.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Ascorbic acid oxidises during storage; prepare and assay as directed.
- Protect iodine solutions from unsuitable light exposure and volatilisation.
- Other reducing substances can interfere.

Viva Questions and Answers

1. Why is the reaction 1:1?

One mole ascorbic acid transfers two electrons, matching one mole iodine.

2. What gives the blue endpoint?

Iodine in the presence of starch after the reductant is consumed.

3. Can this identify ascorbic acid alone in any mixture?

No; other reductants may react with iodine.

Assay of Ibuprofen by Alkalimetry

Experiment 13 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To estimate ibuprofen by neutralising its carboxylic acid group.

Principle

Ibuprofen is a monoprotic acid. Under the prescribed solvent conditions one mole reacts with one mole hydroxide; a solvent blank may be needed.

Requirements

- Assigned ibuprofen specimen
- Standardised NaOH, prescribed alcoholic solvent, indicator or specified endpoint system

Procedure

1. Prepare

Weigh accurately and dissolve in the specified neutralised solvent.

2. Blank

Perform the prescribed solvent blank using the same volume and endpoint system.

3. Titrate

Titrate with standard alkali to the method-defined endpoint.

4. Repeat and calculate

Obtain concordant readings and subtract blank where required.

5. Report

State the assay on the assigned mass basis and compare with the prescribed specification.

Observations and Record

Item	Record
Test mass, g	_____
Titrant M / factor	_____
Test / blank titre, mL	_____
Dilution factor	_____
Percentage assay	_____

Worked Example

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

- Mass ibuprofen (mg) = corrected titre (mL) × NaOH M × 206.28 × dilution factor.
- 0.1000 M NaOH corresponds to 20.628 mg ibuprofen/mL.
- Example: 9.70 mL represents 200.09 mg. For a 205.00 mg test portion, the assay is 97.61%.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the specified solvent; poor dissolution can invalidate the endpoint.
- Keep alcohol away from ignition sources.
- Apply the blank and actual titrant strength.

Viva Questions and Answers

1. What group is titrated?

The carboxylic acid group.

2. Why is the mole ratio 1:1?

Ibuprofen has one titratable acidic proton in this assay.

3. What is the purpose of the blank?

To correct for solvent or reagent acid-base consumption.

Determination of Melting Point

Experiment 14 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To measure the assigned physical constant and interpret its limitations.

Principle

A crystalline solid melts over a range; impurities, heating rate and specimen packing can alter that range.

Requirements

- Assigned solid or liquid
- Calibrated apparatus, suitable thermometer and PPE

Procedure

1. Prepare capillary

Use a dry, finely powdered specimen and the instrument-specified capillary packing.

2. Set up

Check calibration with a suitable melting standard.

3. Heat

Approach the expected range and use the method-specified slow rate near melting.

4. Read

Record the first liquid formation and complete melting; repeat and compare the range.

Pharmaceutical Chemistry

Observations and Record

Observation	Trial 1	Trial 2
Onset / measured temperature, degrees C	_____	_____
Completion / repeated reading, degrees C	_____	_____
Pressure / calibration check	_____	_____

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Record onset and completion, not just one temperature.
- A depressed or broad range suggests possible impurity but is not an identity proof.

Viva Questions and Answers**1. Is the constant alone proof of purity?**

No; it is one part of identity and purity assessment.

2. Why control heating rate?

Fast heating creates thermal lag and inaccurate readings.

3. Why use repeats?

They help reveal inconsistent conditions or reading errors.

Determination of Boiling Point

Experiment 15 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To measure the assigned physical constant and interpret its limitations.

Principle

A liquid boils when its vapour pressure equals ambient pressure. Pressure, thermometer placement and the apparatus affect the measured temperature.

Requirements

- Assigned solid or liquid
- Calibrated apparatus, suitable thermometer and PPE

Procedure

1. Select apparatus

Use the approved micro-boiling or demonstration apparatus and record the method.

2. Check calibration

Verify the thermometer and note atmospheric pressure if required.

3. Observe

Heat safely under the prescribed setup and identify the instrument-defined boiling condition.

4. Record

Enter repeated temperatures and compare with the assigned pressure-corrected value.

Pharmaceutical Chemistry

Observations and Record

Observation	Trial 1	Trial 2
Onset / measured temperature, degrees C	_____	_____
Completion / repeated reading, degrees C	_____	_____
Pressure / calibration check	_____	_____

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use a controlled heat source appropriate to the liquid.
- Do not seal a heated boiling setup.
- Do not use direct flame for flammable liquids.

Viva Questions and Answers**1. Is the constant alone proof of purity?**

No; it is one part of identity and purity assessment.

2. Why control heating rate?

Fast heating creates thermal lag and inaccurate readings.

3. Why use repeats?

They help reveal inconsistent conditions or reading errors.

Preparation of Benzoic Acid from Benzamide

Experiment 16 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To prepare and characterise benzoic acid by supervised hydrolysis of benzamide.

Principle

Hydrolysis converts the amide group to a carboxylic-acid system. In an alkaline route, benzoate is formed and subsequent controlled acidification gives benzoic acid; purification uses its temperature-dependent solubility.

Requirements

- Instructor-assigned benzamide and approved hydrolysis reagents
- Controlled heating setup, filtration and recrystallisation equipment
- Balance, melting-point apparatus and assigned PPE

Procedure

1. Plan

Record the balanced transformation, limiting reactant and instructor-approved working quantities.

2. Hydrolyse

Carry out the assigned supervised hydrolysis method, documenting heating and completion checks.

3. Isolate

Cool and perform the prescribed controlled acidification of an alkaline reaction mixture; collect the precipitated product.

4. Purify

Recrystallise using the assigned suitable solvent, then collect and dry as instructed.

5. Characterise

Record appearance, dry mass, melting range and assigned identity tests.

6. Calculate

Use the limiting-reactant mole amount to calculate theoretical mass and percentage yield.

Observations and Record

Field	Actual record
Benzamide mass / molar amount	_____
Dry product mass	_____
Melting range	_____
Identity observation	_____
Theoretical mass / yield	_____

Worked Example

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

- Illustrative calculation only: 1.2113 g benzamide is 0.01000 mol using 121.13 g/mol. At a 1:1 conversion, theoretical benzoic acid is 1.2212 g using 122.12 g/mol.
- If dry isolated mass is 1.050 g, $\text{yield} = 1.050 / 1.2212 \times 100 = 85.98\%$.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Follow the approved heated-reagent method and ventilation.
- Add acid carefully to the cooled mixture under supervision.
- Yield must use dry purified product, not wet crystals.

Viva Questions and Answers

1. Why acidify a benzoate solution?

It converts soluble benzoate to the less soluble free acid.

2. Why recrystallise?

To separate the desired product from soluble and insoluble impurities.

3. Does high yield prove purity?

No; retained water or contaminants can inflate mass.

Picric Acid Topic - Theory and Supplied-Result Interpretation

Experiment 17 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To understand the syllabus topic and assess instructor-provided identity and yield data.

Principle

Picric acid is a nitro-substituted phenol with explosive hazards, especially when dry or in certain metal-salt forms. This activity uses diagrams and supplied results rather than preparation or handling.

Key Points

- Identify the phenolic and nitro functional groups on the supplied structural diagram.
- Relate strong electron-withdrawing groups to acidity at a conceptual level.
- Use only instructor-provided analytical data for the record.
- Discuss why a hazardous preparation is replaced with an educational simulation.

Practice Scenario

Hypothetical data supplied for calculation: theoretical mass 2.00 g; reported mass 1.50 g. Yield = 75.0%. These are worksheet values, not a preparation formula.

Activity Procedure

1. Review theory

Label the functional groups on the supplied structure; discuss substitution and acidity without a synthesis procedure.

2. Read supplied data

Identify the product name, stated theoretical mass, recorded mass and identity-test conclusion in the worksheet.

3. Calculate

Calculate yield from the supplied masses, keeping the data clearly hypothetical.

4. Interpret

Separate identity, purity and yield; state which conclusions the provided tests support.

Record and Observations

Data item	Worksheet record
Theoretical mass	_____
Reported product mass	_____
Identity / purity evidence	_____
Calculated yield	_____

Result

Complete the record using your activity, then state the findings, the action required and the reason for that action.

Precautions

- Do not prepare, dry or handle picric acid or its salts in this activity.
- Treat any existing hazardous container through the institution's trained hazardous-material process.

Viva Questions and Answers

1. What is the phenolic group?

A hydroxyl group attached directly to an aromatic ring.

2. Is yield proof of identity?

No; the supplied identity evidence must be assessed separately.

3. Why use a simulation?

It meets the theory and data-analysis objective without explosive-material preparation.

Identification and Purity Assessment of Aspirin

Experiment 18 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To assess the identity and assigned purity characteristics of aspirin.

Principle

Acetylsalicylic acid; an ester and carboxylic acid. Identity requires the prescribed characteristic test; purity is evaluated separately with specified limits.

Requirements

- Assigned monograph and labelled material
- Instructor-prepared reagents and controls
- Balance, melting-point setup or supplied instrumental data

Procedure

1. Review

Record official identity reactions and assigned purity limits.

2. Physical assessment

Record appearance, solubility under the assigned conditions and melting range if required.

3. Identity test

Assigned controlled hydrolysis followed by the prescribed salicylate confirmation.

4. Compare controls

Record the actual test response alongside the positive and blank controls.

5. Purity assessment

Compare the intact material with the assigned hydrolysed portion; an inappropriate ferric-colour response in intact aspirin can suggest free salicylate.

6. Conclude

State identity support and each purity-test finding separately.

Observations and Record

Check	Observation / result
Appearance / solubility	_____
Identity reaction	_____
Melting range / supplied spectrum	_____
Purity test and stated limit	_____
Conclusion	_____

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the prescribed test rather than improvised reagents.
- Observe corrosive-reagent and solvent precautions.
- Do not taste any pharmaceutical material.

Viva Questions and Answers

1. Is one colour test fully specific?

Often not; the complete assigned identity scheme and controls are required.

2. How do identity and purity differ?

Identity establishes what the material is; purity tests examine unwanted components or specified attributes.

3. Why record limits?

A measurement cannot be classified without the applicable criterion.

Identification and Purity Assessment of Caffeine

Experiment 19 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To assess the identity and assigned purity characteristics of caffeine.

Principle

A methylxanthine alkaloid. Identity requires the prescribed characteristic test; purity is evaluated separately with specified limits.

Requirements

- Assigned monograph and labelled material
- Instructor-prepared reagents and controls
- Balance, melting-point setup or supplied instrumental data

Procedure

1. Review

Record official identity reactions and assigned purity limits.

2. Physical assessment

Record appearance, solubility under the assigned conditions and melting range if required.

3. Identity test

Assigned pharmacopoeial identity or instructor-demonstrated colour reaction.

4. Compare controls

Record the actual test response alongside the positive and blank controls.

5. Purity assessment

Use the supplied spectral, melting and monograph identity data; avoid substituting appearance for identity.

6. Conclude

State identity support and each purity-test finding separately.

Observations and Record

Check	Observation / result
Appearance / solubility	_____
Identity reaction	_____
Melting range / supplied spectrum	_____
Purity test and stated limit	_____
Conclusion	_____

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the prescribed test rather than improvised reagents.
- Observe corrosive-reagent and solvent precautions.
- Do not taste any pharmaceutical material.

Viva Questions and Answers

1. Is one colour test fully specific?

Often not; the complete assigned identity scheme and controls are required.

2. How do identity and purity differ?

Identity establishes what the material is; purity tests examine unwanted components or specified attributes.

3. Why record limits?

A measurement cannot be classified without the applicable criterion.

Identification and Purity Assessment of Paracetamol

Experiment 20 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To assess the identity and assigned purity characteristics of paracetamol.

Principle

A phenolic acetanilide. Identity requires the prescribed characteristic test; purity is evaluated separately with specified limits.

Requirements

- Assigned monograph and labelled material
- Instructor-prepared reagents and controls
- Balance, melting-point setup or supplied instrumental data

Procedure

1. Review

Record official identity reactions and assigned purity limits.

2. Physical assessment

Record appearance, solubility under the assigned conditions and melting range if required.

3. Identity test

Assigned phenolic or hydrolysis-based identity reaction.

4. Compare controls

Record the actual test response alongside the positive and blank controls.

5. Purity assessment

Assess the assigned purity tests and melting range; a generic phenolic response is not specific enough alone.

6. Conclude

State identity support and each purity-test finding separately.

Observations and Record

Check	Observation / result
Appearance / solubility	_____
Identity reaction	_____
Melting range / supplied spectrum	_____
Purity test and stated limit	_____
Conclusion	_____

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the prescribed test rather than improvised reagents.
- Observe corrosive-reagent and solvent precautions.
- Do not taste any pharmaceutical material.

Viva Questions and Answers

1. Is one colour test fully specific?

Often not; the complete assigned identity scheme and controls are required.

2. How do identity and purity differ?

Identity establishes what the material is; purity tests examine unwanted components or specified attributes.

3. Why record limits?

A measurement cannot be classified without the applicable criterion.

Identification and Purity Assessment of Sulfanilamide

Experiment 21 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To assess the identity and assigned purity characteristics of sulfanilamide.

Principle

A sulphonamide with a primary aromatic amino group. Identity requires the prescribed characteristic test; purity is evaluated separately with specified limits.

Requirements

- Assigned monograph and labelled material
- Instructor-prepared reagents and controls
- Balance, melting-point setup or supplied instrumental data

Procedure

1. Review

Record official identity reactions and assigned purity limits.

2. Physical assessment

Record appearance, solubility under the assigned conditions and melting range if required.

3. Identity test

Assigned instructor-controlled diazotisation/coupling identity reaction.

4. Compare controls

Record the actual test response alongside the positive and blank controls.

5. Purity assessment

Read the specified colour response and controls; the aromatic amino group explains the assigned reaction.

6. Conclude

State identity support and each purity-test finding separately.

Observations and Record

Check	Observation / result
Appearance / solubility	_____
Identity reaction	_____
Melting range / supplied spectrum	_____
Purity test and stated limit	_____
Conclusion	_____

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the prescribed test rather than improvised reagents.
- Observe corrosive-reagent and solvent precautions.
- Do not taste any pharmaceutical material.

Viva Questions and Answers

1. Is one colour test fully specific?

Often not; the complete assigned identity scheme and controls are required.

2. How do identity and purity differ?

Identity establishes what the material is; purity tests examine unwanted components or specified attributes.

3. Why record limits?

A measurement cannot be classified without the applicable criterion.

Systematic Qualitative Analysis - Sodium Chloride

Experiment 22 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To analyse an assigned coded inorganic material and support its cation and anion identity.

Principle

Systematic analysis records preliminary observations, separate ion tests and confirmation. NaCl is the instructor-confirmed teaching identity for this exercise; students must record observed evidence.

Requirements

- Coded material, clean tubes and separate aliquots
- Assigned qualitative-analysis reagents, positive controls and blank

Procedure

1. Preliminary examination

Record appearance and assigned solubility tests. White crystalline, readily water-soluble teaching salt.

2. Plan aliquots

Prepare separate labelled portions for cation and anion analysis.

3. Perform prescribed reactions

Yellow flame supports sodium; nitric-acid/silver-nitrate testing with the prescribed confirmation supports chloride.

4. Check consistency

Compare with controls and resolve conflicting results before identifying the material.

5. Write report

State cation, anion, evidence and instructor confirmation.

Pharmaceutical Chemistry

Observations and Record

Stage	Observation	Inference
Appearance / solubility	_____	_____
Cation test	_____	_____
Cation confirmation	_____	_____
Anion test	_____	_____
Anion confirmation	_____	_____

Result

Cation identified: _____. Anion identified: _____. Supporting observations: _____. Instructor confirmation: _____.

Precautions

- Never taste the material or directly smell gases.
- Keep cation and anion aliquots separate.
- Use designated metal-containing waste where required.

Viva Questions and Answers**1. Which cation is expected here?**

Sodium.

2. Which anion is expected here?

Chloride.

3. Why is an unknown coded?

It makes the student base identification on evidence instead of the label.

Systematic Qualitative Analysis - Calcium Carbonate

Experiment 23 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To analyse an assigned coded inorganic material and support its cation and anion identity.

Principle

Systematic analysis records preliminary observations, separate ion tests and confirmation. CaCO_3 is the instructor-confirmed teaching identity for this exercise; students must record observed evidence.

Requirements

- Coded material, clean tubes and separate aliquots
- Assigned qualitative-analysis reagents, positive controls and blank

Procedure

1. Preliminary examination

Record appearance and assigned solubility tests. White material with poor water solubility.

2. Plan aliquots

Prepare separate labelled portions for cation and anion analysis.

3. Perform prescribed reactions

Controlled acid addition gives carbon dioxide; prescribed limewater confirmation supports carbonate. Calcium is confirmed on a separate prepared aliquot by oxalate testing.

4. Check consistency

Compare with controls and resolve conflicting results before identifying the material.

5. Write report

State cation, anion, evidence and instructor confirmation.

Pharmaceutical Chemistry

Observations and Record

Stage	Observation	Inference
Appearance / solubility	_____	_____
Cation test	_____	_____
Cation confirmation	_____	_____
Anion test	_____	_____
Anion confirmation	_____	_____

Result

Cation identified: _____. Anion identified: _____. Supporting observations: _____. Instructor confirmation: _____.

Precautions

- Never taste the material or directly smell gases.
- Keep cation and anion aliquots separate.
- Use designated metal-containing waste where required.

Viva Questions and Answers**1. Which cation is expected here?**

Calcium.

2. Which anion is expected here?

Carbonate.

3. Why is an unknown coded?

It makes the student base identification on evidence instead of the label.

Systematic Qualitative Analysis - Copper Sulphate

Experiment 24 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To analyse an assigned coded inorganic material and support its cation and anion identity.

Principle

Systematic analysis records preliminary observations, separate ion tests and confirmation. CuSO_4 is the instructor-confirmed teaching identity for this exercise; students must record observed evidence.

Requirements

- Coded material, clean tubes and separate aliquots
- Assigned qualitative-analysis reagents, positive controls and blank

Procedure

1. Preliminary examination

Record appearance and assigned solubility tests. Hydrated teaching material and its solution are commonly blue.

2. Plan aliquots

Prepare separate labelled portions for cation and anion analysis.

3. Perform prescribed reactions

The prescribed ammonia-complex response supports copper; an acidified barium test supports sulphate.

4. Check consistency

Compare with controls and resolve conflicting results before identifying the material.

5. Write report

State cation, anion, evidence and instructor confirmation.

Pharmaceutical Chemistry

Observations and Record

Stage	Observation	Inference
Appearance / solubility	_____	_____
Cation test	_____	_____
Cation confirmation	_____	_____
Anion test	_____	_____
Anion confirmation	_____	_____

Result

Cation identified: _____. Anion identified: _____. Supporting observations: _____. Instructor confirmation: _____.

Precautions

- Never taste the material or directly smell gases.
- Keep cation and anion aliquots separate.
- Use designated metal-containing waste where required.

Viva Questions and Answers**1. Which cation is expected here?**

Copper(II).

2. Which anion is expected here?

Sulphate.

3. Why is an unknown coded?

It makes the student base identification on evidence instead of the label.

Systematic Qualitative Analysis - Ammonium Chloride

Experiment 25 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To analyse an assigned coded inorganic material and support its cation and anion identity.

Principle

Systematic analysis records preliminary observations, separate ion tests and confirmation. NH_4Cl is the instructor-confirmed teaching identity for this exercise; students must record observed evidence.

Requirements

- Coded material, clean tubes and separate aliquots
- Assigned qualitative-analysis reagents, positive controls and blank

Procedure

1. Preliminary examination

Record appearance and assigned solubility tests. White crystalline, water-soluble teaching salt.

2. Plan aliquots

Prepare separate labelled portions for cation and anion analysis.

3. Perform prescribed reactions

The supervised alkaline/indicator test supports ammonium; silver-chloride testing with prescribed confirmation supports chloride.

4. Check consistency

Compare with controls and resolve conflicting results before identifying the material.

5. Write report

State cation, anion, evidence and instructor confirmation.

Observations and Record

Stage	Observation	Inference
Appearance / solubility	_____	_____
Cation test	_____	_____
Cation confirmation	_____	_____
Anion test	_____	_____
Anion confirmation	_____	_____

Result

Cation identified: _____. Anion identified: _____. Supporting observations: _____. Instructor confirmation: _____.

Precautions

- Never taste the material or directly smell gases.
- Keep cation and anion aliquots separate.
- Use designated metal-containing waste where required.

Viva Questions and Answers

1. Which cation is expected here?

Ammonium.

2. Which anion is expected here?

Chloride.

3. Why is an unknown coded?

It makes the student base identification on evidence instead of the label.

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