# Assays of Inorganic Pharmaceutical Substances

Experiment 2 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

## 1. Ammonium Chloride by Acid-base Titration

### 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.

## 2. Ferrous Sulphate by Cerimetry

### 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 $18.00 \times 0.1000 \times 278.01 = 500.42$ 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 $FeSO_4$ 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 $FeSO_4 \cdot 7H_2O$.

## 3. Copper Sulphate by Iodometry

### 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 $KIO_3$, 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 $CuSO_4 \cdot 5H_2O$ 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 $CuSO_4 \cdot 5H_2O$ per mL.

## 4. Calcium Gluconate by Complexometry

### 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 $CaCO_3$, 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 ≈ 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.

## 5. Hydrogen Peroxide by Permanganometry

### 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

- $2\ MnO4^- + 5\ H2O2 + 6\ H^+ \rightarrow 2\ Mn^{2+} + 5\ O2 + 8\ H2O$.
- $H2O2$ 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. $CKMnO4$ = (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.

## 6. Sodium Benzoate by Non-aqueous Titration

### 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.

## 7. Sodium Chloride by Modified Volhard Method

### 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.

## 8. Potassium Iodide by Iodate Titration

### Aim

To determine potassium iodide content by the prescribed iodate method.

### Principle

The assigned iodate-iodide redox method has a method-specific reaction stoichiometry and endpoint.

### Requirements

- Assigned test material and the prescribed method reagents or standards.
- Calibrated volumetric glassware, appropriate instrument and record sheet.

### Procedure

#### 1. Prepare and identify the test

Confirm the analyte, method version, concentration basis and test-portion amount. Prepare the test solution as specified and record any dilution.

#### 2. Set the measurement basis

Check the instrument or standardised titrant. Prepare the specified blank, standard and control so that the test and comparison use the same basis.

### 3. Make the measurement

Apply the prescribed the prescribed iodate method method and record standardised iodate volume and assigned endpoint. Record replicate readings and any interfering appearance.

### 4. Calculate

Apply the stated equation with the correct blank correction, dilution and unit conversion. Show the substitution and retain the observed readings.

### 5. Assess the result

Compare replicate agreement and the assigned specification. Record a result with units and the limitations of the method.

## Observations and Record

| Measurement | Reading |
|---|---|
| Test portion / dilution | _____ |
| Blank / standard | _____ |
| standardised iodate volume and assigned endpoint | _____ |
| Calculated content and unit | _____ |

## Worked Example

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

- Analyte mass = blank-corrected titrant volume × official method factor × dilution factor.
- Illustration of factor use only: 12.0 mL × an assigned factor of 0.020 g/mL gives 0.240 g. Derive the actual factor from the official reaction, not this illustrative value.

## Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

## Precautions

- Use the exact method reagents and assigned endpoint; different methods are not interchangeable.
- Record the blank correction and the actual test portion rather than an assumed value.
- Hazardous reagent operations require the specified laboratory facilities and supervision.

## Viva Questions and Answers

### 1. What produces the analytical signal?

The assigned iodate-iodide redox method has a method-specific reaction stoichiometry and endpoint.

#### 2. Why include a blank or control?

It identifies contributions unrelated to the analyte or checks that the measurement system performed appropriately.

#### 3. What must accompany the result?

Units, method, dilution basis, relevant control performance and any limitation.

## 9. Barium Sulphate by Gravimetry

### Aim

To determine the assigned barium sulphate test quantity by the prescribed gravimetric method.

### Principle

A known chemical conversion and measured dry precipitate mass permit gravimetric determination when isolation and weighing are controlled.

### Requirements

- Assigned test material and the prescribed method reagents or standards.
- Calibrated volumetric glassware, appropriate instrument and record sheet.

### Procedure

#### 1. Prepare and identify the test

Confirm the analyte, method version, concentration basis and test-portion amount. Prepare the test solution as specified and record any dilution.

#### 2. Set the measurement basis

Check the instrument or standardised titrant. Prepare the specified blank, standard and control so that the test and comparison use the same basis.

#### 3. Make the measurement

Apply the prescribed the prescribed gravimetric method method and record constant-mass precipitate. Record replicate readings and any interfering appearance.

#### 4. Calculate

Apply the stated equation with the correct blank correction, dilution and unit conversion. Show the substitution and retain the observed readings.

#### 5. Assess the result

Compare replicate agreement and the assigned specification. Record a result with units and the limitations of the method.

### Observations and Record

| Measurement | Reading |
|---|---|
| Test portion / dilution | _____ |
| Blank / standard | _____ |
| constant-mass precipitate | _____ |
| Calculated content and unit | _____ |

### Worked Example

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

- Analyte mass = corrected precipitate mass × gravimetric factor. Recovery (%) = measured analyte mass / expected analyte mass × 100.
- Example: 0.490 g recovered from an expected 0.500 g gives 98.0% recovery.

### Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

### Precautions

- Use the exact method reagents and assigned endpoint; different methods are not interchangeable.
- Record the blank correction and the actual test portion rather than an assumed value.
- Hazardous reagent operations require the specified laboratory facilities and supervision.

### Viva Questions and Answers

#### 1. What produces the analytical signal?

A known chemical conversion and measured dry precipitate mass permit gravimetric determination when isolation and weighing are controlled.

#### 2. Why include a blank or control?

It identifies contributions unrelated to the analyte or checks that the measurement system performed appropriately.

#### 3. What must accompany the result?

Units, method, dilution basis, relevant control performance and any limitation.

## 10. Antimony-containing Pharmaceutical Salts

### Aim

To determine sodium antimony gluconate or antimony potassium tartrate by the explicitly assigned monograph assay.

## Principle

An assigned antimony assay measures content through the prescribed redox or other compendial reaction. The factor depends on the salt and hydration state.

## Requirements

- Assigned test material and the prescribed method reagents or standards.
- Calibrated volumetric glassware, appropriate instrument and record sheet.

## Procedure

### 1. Prepare and identify the test

Confirm the analyte, method version, concentration basis and test-portion amount. Prepare the test solution as specified and record any dilution.

### 2. Set the measurement basis

Check the instrument or standardised titrant. Prepare the specified blank, standard and control so that the test and comparison use the same basis.

### 3. Make the measurement

Apply the prescribed the explicitly assigned monograph assay method and record method-specific titration or instrumental reading. Record replicate readings and any interfering appearance.

### 4. Calculate

Apply the stated equation with the correct blank correction, dilution and unit conversion. Show the substitution and retain the observed readings.

### 5. Assess the result

Compare replicate agreement and the assigned specification. Record a result with units and the limitations of the method.

## Observations and Record

| Measurement | Reading |
|---|---|
| Test portion / dilution | _____ |
| Blank / standard | _____ |
| method-specific titration or instrumental reading | _____ |
| Calculated content and unit | _____ |

## Worked Example

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

- Content (%) = determined mass of the specified analyte / test-portion mass × 100.
- Illustration: 0.196 g determined in a 0.200 g portion gives 98.0%. Record the actual monograph basis and formula mass.

### Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

### Precautions

- Use the exact method reagents and assigned endpoint; different methods are not interchangeable.
- Record the blank correction and the actual test portion rather than an assumed value.
- Hazardous reagent operations require the specified laboratory facilities and supervision.

### Viva Questions and Answers

#### 1. What produces the analytical signal?

An assigned antimony assay measures content through the prescribed redox or other compendial reaction. The factor depends on the salt and hydration state.

#### 2. Why include a blank or control?

It identifies contributions unrelated to the analyte or checks that the measurement system performed appropriately.

#### 3. What must accompany the result?

Units, method, dilution basis, relevant control performance and any limitation.

First Hope

COMPLETE NOTES

Click here to visit our website

PHARMACY PRACTICAL NOTES

# Complete subject and semesters are available on FirstHope app only.

Scan or click here

Click here to get the app >

Available on Google Play

JOIN THE FIRSTHOPE COMMUNITY

## Join our Telegram channel

Click here to join Telegram >

Scan or click here

@FirstHopeNotes

### Instagram

@firsthope.official

Click here to follow >

### YouTube channel

FirstHope-Pharmacy / Live streams

Click here to watch >

### WhatsApp channel

Follow FirstHope on WhatsApp

Click here to join >

### FirstHope website

firsthope.co.in

Click here to visit >

**B.Pharm / D.Pharm / M.Pharm / Pharm.D**

Complete subjects and semesters are available in the FirstHope app.