

Tests for Pharmaceutical Purity and Performance

Experiment 5 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

1. Swelling Power of Bentonite

Aim

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

Principle

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

Requirements

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

Procedure

1. Identify the method

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

2. Disperse

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

3. Stand

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

4. Read

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

5. Report

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

Observations and Record

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

Worked Example

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

- Illustrative measurement: 2.0 g bentonite occupies 28 mL under the stated method. Report 28 mL for 2.0 g when that is the prescribed basis.

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- An additional derived swelling value is $28 \div 2.0 = 14$ mL/g. It must not replace a criterion expressed as volume for a fixed portion without explanation.

Result

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

Precautions

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

Viva Questions and Answers

1. Why add gradually?

It supports reproducible wetting and reduces dry lumps.

2. Why state the mass basis?

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

3. Does swelling alone establish all bentonite quality attributes?

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

2. Acid-neutralising Capacity of Aluminium Hydroxide Gel

Aim

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

Principle

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

Requirements

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

Procedure

1. Record the basis

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

2. Add known acid

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

3. Back-titrate

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

4. Check controls

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

5. Calculate

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

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

Worked Example

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

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

Result

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

Precautions

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

Viva Questions and Answers**1. Why use back-titration?**

It measures the excess acid after the gel has reacted.

2. What is mEq on this basis?

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

3. Why define the endpoint?

Different endpoints can give different estimates of residual acid.

3. Ammonium in Potash Alum**Aim**

To determine ammonium-related impurity response by the assigned compendial test.

Principle

The prescribed ammonium test compares a sample response with a stated limit or standard.

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.

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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 assigned compendial test method and record test response versus standard or limit. 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	_____
test response versus standard or limit	_____
Calculated content and unit	_____

Worked Example

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

- State the method-specific comparison as meets / does not meet the assigned limit; do not invent a numerical content from a visual test.

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 prescribed ammonium test compares a sample response with a stated limit or standard.

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.

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4. Adsorption Power of Heavy Kaolin

Aim

To determine adsorption performance of heavy kaolin by the assigned adsorption test.

Principle

Adsorption removes an assigned test solute from solution; the remaining response indicates adsorption performance under defined conditions.

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 assigned adsorption test method and record residual test-solute signal. 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	_____
residual test-solute signal	_____
Calculated content and unit	_____

Worked Example

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

- Removal (%) = (initial amount – remaining amount) / initial amount × 100.
- Example: 100 units initially and 25 units remaining gives 75% removal, provided volumes and blanks are accounted for.

Result

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

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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?

Adsorption removes an assigned test solute from solution; the remaining response indicates adsorption performance under defined conditions.

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.

5. Iodate-related Purity Test in Potassium Iodide

Aim

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

Principle

Free iodine gives a characteristic colour with starch under the test conditions. In acid, iodate reacts with iodide to liberate iodine: $\text{IO}_3^- + 5 \text{I}^- + 6 \text{H}^+ \rightarrow 3 \text{I}_2 + 3 \text{H}_2\text{O}$. Separate observations before and after acidification help distinguish existing iodine from newly liberated iodine.

Requirements

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

Procedure

1. Prepare the material

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

2. Test free iodine

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

3. Test iodate

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

4. Review validity

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

5. Report

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

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Observations and Record

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

Worked Example

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

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

Result

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

Precautions

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

Viva Questions and Answers**1. Why is acid needed in the iodate test?**

The reaction that liberates iodine consumes hydrogen ions.

2. What does starch detect?

The iodine-containing complex that gives the characteristic colour.

3. Why test free iodine separately?

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

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