# 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.

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

### Result

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

### Precautions

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

### Viva Questions and Answers

#### 1. Why add gradually?

It supports reproducible wetting and reduces dry lumps.

#### 2. Why state the mass basis?

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

#### 3. Does swelling alone establish all bentonite quality attributes?

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

## 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.

### Observations and Record

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

### Worked Example

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

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

### Result

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

### Precautions

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

### Viva Questions and Answers

#### 1. Why use back-titration?

It measures the excess acid after the gel has reacted.

#### 2. What is mEq on this basis?

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

#### 3. Why define the endpoint?

Different endpoints can give different estimates of residual acid.

## 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.

### 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.

## 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.

### 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: $IO_3^- + 5\ I^- + 6\ H^+ \rightarrow 3\ I_2 + 3\ H_2O$. Separate observations before and after acidification help distinguish existing iodine from newly liberated iodine.

### Requirements

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

### Procedure

#### 1. Prepare the material

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

#### 2. Test free iodine

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

#### 3. Test iodate

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

#### 4. Review validity

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

#### 5. Report

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

### Observations and Record

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

### Worked Example

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

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

### Result

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

### Precautions

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

### Viva Questions and Answers

#### 1. Why is acid needed in the iodate test?

The reaction that liberates iodine consumes hydrogen ions.

#### 2. What does starch detect?

The iodine-containing complex that gives the characteristic colour.

#### 3. Why test free iodine separately?

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

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