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

Requirements

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

Procedure

1. Identify conditions

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

2. Prepare the pair

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

3. Develop turbidity

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

4. Compare

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

5. Apply the modification

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

Modified Test: Matrix Preparation

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

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

Observations and Record

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

Worked Example

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

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

Result

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

Precautions

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

Viva Questions and Answers

1. What is the insoluble product?

Barium sulphate.

2. Why may acidification be important?

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

3. Does a darker starting colour mean more sulphate?

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

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