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.

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