

Limit Test for Heavy Metals

Experiment 4 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare a heavy-metal impurity response with the prescribed lead-equivalent standard.

Principle

The traditional sulphide method produces coloured metal sulphides under a prescribed pH. The response is compared with a lead standard and reported as lead-equivalent impurity; it is not an assay of each individual metal.

Requirements

- Assigned monograph, test material and lead standard
- Instructor-prepared sulphide reagent system and specified buffer
- Matched comparison tubes, pipettes and controlled ventilation

Procedure

1. Check the method

Identify the test mass, pretreatment, prescribed limit, buffer and reagent-development time.

2. Prepare test and standard

Prepare matched solutions of the test portion and the stated lead comparator. Include the required blank and sensitivity control.

3. Develop colour

Use the assigned buffered sulphide-reagent procedure under supervision; treat test and comparator equally.

4. Compare

At the prescribed time compare brown-to-dark colour under matched viewing conditions.

5. Record validity

Enter the response and state whether the blank and controls support a valid comparison.

Observations and Record

Field	Record
Material / mass	_____
Lead-equivalent standard amount	_____
pH / development conditions	_____
Test versus comparator	_____
Control validity	_____

Worked Example

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

- Illustrative limit: 1.0 g material at an assigned 20 micrograms/g limit needs a comparator containing 20 micrograms lead equivalent.
- A 10 micrograms/mL standard supplies that amount in 2.0 mL; the actual monograph sets the limit.

Result

The test response was _____ relative to the comparator. Controls were _____. The material _____ the assigned limit.

Precautions

- Metal standards and sulphide reagents require designated ventilation, exposure controls and separate waste collection.
- Do not acidify sulphide waste outside the approved process.
- Do not interpret an invalid blank or poorly developed standard as a passing test.

Viva Questions and Answers

1. What does lead-equivalent mean?

A response expressed relative to the prescribed lead comparator, rather than a separate amount for every metal.

2. Why control pH?

Sulphide formation and the response of different metals depend on pH.

3. Is this a modern elemental-impurity panel?

No; instrumental element-specific tests are separate methods.

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