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Detection of Nitrogen Sulphur and Halogens

Experiment 2 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To interpret Lassaigne elemental tests using instructor-prepared fusion extracts and appropriate controls.

Principle

Sodium-fusion analysis converts suitable covalently bound elements into water-soluble ionic forms. Nitrogen is detected through the iron-cyanide colour sequence, sulphur through a sulphide reaction and halogens through silver-halide formation after the prescribed removal of interfering ions.

Requirements

- Teacher-prepared and labelled Lassaigne extracts, positive and negative controls.
- Prepared ferrous reagent, nitroprusside reagent, silver nitrate and instructor-treated extract for the halogen branch.

Procedure

1. Obtain the extract

Record the teacher-prepared extract identifier. Hot fusion, sodium handling and extract treatment are performed only by trained staff in the designated setup.

2. Nitrogen branch

Use a separate extract portion for the instructor's iron-reagent sequence. Observe the Prussian-blue result against known positive and negative controls; record an inconclusive or failed-control result as such.

3. Sulphur branch

On a new portion add the assigned fresh nitroprusside reagent. A violet/purple response supports sulphide in the prepared extract; observe promptly as colour can fade.

4. Halogen branch

Use only the extract portion prepared by the instructor for this branch. Add the specified silver nitrate reagent and observe white, cream or yellow silver-halide precipitate. Further prescribed solubility comparison can help distinguish halides.

5. Consider combinations

When both nitrogen and sulphur occur, the fusion chemistry may produce a thiocyanate response. Interpret with the teacher's method; an unsuitable fusion can give a false negative.

6. Record elements

List detected elements separately from functional-group conclusions. A nitrogen-positive result alone does not distinguish an amine from an amide or nitro compound.

Observations and Record

Branch	Supporting observation	Record / limitation
Nitrogen	Prussian-blue response	_____
Sulphur	Violet nitroprusside response	_____
Halogens	AgCl white; AgBr cream; AgI yellow	_____

Worked Example

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

- Positive nitrogen with negative sulphur/halogen narrows the classes but does not identify a specific molecule. Combine it with the functional tests and physical constant.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Do not prepare or acidify sodium-fusion extracts independently; some treatments can release hazardous vapours.
- Silver-containing residues are disposed of through the prescribed waste system; do not store reactive test mixtures.

Viva Questions and Answers

1. Why use fusion before these tests?

The original covalent forms are not generally detected by ordinary ionic reagents.

2. Why remove halogen-test interference?

Other fusion ions may form misleading silver precipitates.

3. Does a negative nitrogen test always prove absence?

No. Extract preparation, sensitivity and controls must be satisfactory.

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