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Ash Values and Extractive Values

Experiment 10 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To calculate total ash, selected ash fractions and water- or alcohol-soluble extractive values for a prescribed crude drug.

Principle

Ash tests assess the residue remaining after controlled incineration. Total ash includes physiological mineral residue and extraneous inorganic matter; acid-insoluble ash can indicate siliceous contamination. Extractive value measures residue recovered under a defined solvent and extraction method, not the amount of one specific active constituent.

Requirements

- Representative powdered drug; prescribed water, alcohol and acid reagents.
- Tared crucibles, furnace, desiccator and balance.
- Extraction flask, volumetric glassware, filter and tared evaporating dish.

Procedure

1. Determine total ash

Weigh the prescribed drug portion in a tared crucible. Incinerate gradually at the method's temperature to the required endpoint, cool in a desiccator and weigh.

2. Determine ash fractions

Treat the total ash with the specified acid for acid-insoluble residue, or water for the water-soluble ash determination. Collect and incinerate the relevant residue as directed.

3. Extract

Weigh the drug portion and use the prescribed solvent strength, solvent volume, time and agitation. Prepare a separate water and alcohol test where required.

4. Measure an aliquot

Filter as prescribed and transfer a known aliquot to a tared dish. Evaporate and dry the residue under the stated conditions.

5. Calculate

Express ash as $\text{residue mass} \div \text{drug mass} \times 100$. For extractive value, account for total extract volume and the aliquot volume; state the solvent and basis.

Introduction to Pharmacognosy

Observations and Record

Test	Starting portion / residue	Calculated value
Total / acid-insoluble ash	___ g / ___ g	___% / ___%
Water-soluble ash	Total ash minus insoluble residue	___%
Extractive	Drug ___ g; residue ___ g; volumes ___	___%

Worked Example

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

- Total ash example: 0.10 g ash from 2.00 g drug gives 5.0%. Acid-insoluble residue 0.02 g gives 1.0% on the original drug basis.
- If water-insoluble residue is 0.03 g from 0.10 g total ash, water-soluble ash is $0.07 \div 2.00 \times 100 = 3.5\%$.
- Extractive (%) = $100 \times \text{residue mass} \times \text{total extract volume} \div (\text{aliquot volume} \times \text{drug mass})$.
- Example: 4.0 g drug, 100 mL extract, 25 mL aliquot, 0.080 g dry residue: $100 \times 0.080 \times 100 \div (25 \times 4.0) = 8.0\%$.

Result

Total ash ___%; acid-insoluble ash ___%; water-soluble ash ___%; ___-soluble extractive ___%. Conditions and basis are recorded.

Precautions

- Avoid flaming, loss of ash and weighing a hot crucible.
- Use the stated solvent strength and extraction conditions; changing them changes the extractive value.
- Apply the drug-specific specification rather than an invented universal limit.

Viva Questions and Answers

1. What can acid-insoluble ash suggest?

The presence of siliceous material such as sand, interpreted with the method and specification.

2. Is extractive value an assay of one active ingredient?

No; it measures total extracted residue under defined conditions.

3. Why correct for the aliquot?

Only part of the extract was evaporated, so its residue must be scaled to the total volume.

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