Part 7:
Identification and Test for Purity of Pharmaceuticals

CHAPTER-17

Experiment: 17

IDENTIFICATION AND TEST FOR PURITY OF ASPIRIN

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Chapter Id: ASU/NSP/EB/APHPC/2022/Ch-17

doi: https://doi.org/10.52458/9789391842611.nsp2022.eb.asu.ch17

Aim:

To perform and report identification test on the given sample of Aspirin as per I.P.

Requirements:

A. Glassware & Instruments:

1. Test tube
2. Capillary
3. Thiel's tube
4. Melting point apparatus

B. Chemicals & Reagents:

1. NaOH solution
2. Freshly prepared $FeCl_3$ solution
3. H_2SO_4
4. Alcohol
5. Distilled water (DW)
6. Chloroform
7. Ether

Theory:

Knowledge of acid/base chemistry, redox chemistry, and solubility are required for any process that can provide a qualitative determination of the ions present in a simple inorganic compound. In this regard, it is significantly easier to identify a single pure compound than a mixture. This experiment focuses solely on identifying simple compounds, defined as those containing only one cation and one anion.

Monograph of Aspirin:

Chemical formula: $C_9H_8O_4$

Molecular weight: 180.2

Organoleptic properties: Colourless crystals or white, crystalline powder; odourless or almost odourless.

Physical Properties:

- **Solubility:** Statements of solubility's are indicated by a descriptive terms and are intended to apply at 20°C to 30°C. The following table indicates the meaning of the terms used in statements of approximate solubility's.

Descriptive terms (Statement of approx. solubility)	**Approx. volume of solvent in ml per g of solute**
Very soluble	Less than 1
Freely soluble	From 1 to 10
Soluble	From 10 to 30
Sparingly soluble	From 30 to 100
Slightly soluble	From 100 to 1000
Very slightly soluble	From 1000 to 10,000
Insoluble/ practically insoluble	More than 10,000

- **Identification test:** These tests ensure that the examined item is consistent with the label on the container. If an article taken from a labelled container fails a prescribed identification test, the article may be mislabeled or substituted. These tests are not necessarily sufficient to establish proof of identity with absolute certainty.
- **Category:** The pharmaceutical category of a drug refers to its medicinal and pharmaceutical uses, such as pharmaceutical aid, analgesic, Antipyretic, and Anti-inflammatory. etc.

Procedure:

Physical Tests:

- **Organoleptic properties:** Observe the given drug critically for the following description. The drug is crystals or crystalline powder, colourless or white, odourless or almost odourless as per I.P.
- **Solubility:** Perform solubility test in the different solvents. The drug is sparingly soluble in water, freely soluble in alcohol, soluble in chloroform and in ether, slightly soluble in water.

ISBN: 978-93-91842-61-1

Identification Tests:

- **Test 1:** About 0.5 g of the drug is boiled for three minutes with 10 ml of NaOH solution; after cooling, 10 ml of diluted H_2SO_4 is added, producing a white crystalline precipitate with an AcOH odour. A deep violet hue is produced by filtering, dissolving the precipitate in about 2 ml of water, after adding $FeCl_3$ solution.
- **Test 2:** Add 3 ml of 95% alcohol and 3 ml of H_2SO_4 to the filtrate obtained in test A and warm; the odour of ethyl acetate is detectable.
- **Test 3:** Determination of Melting Point: The drug melts at approximately 142 degrees Celsius.

Observation:

Test	**Observation**	**Inference**
Physical Tests: a. Nature b. Colour c. Odour		
Solubility: a. Water b. Ethanol c. Chloroform d. Ether		
Identification Test: a. Test 1 b. Test 2 c. Test 3		
*If observation is as specified in the procedure, write "passes the test"; otherwise, write "fails the test."		

Results:

The given sample of Aspirin passes the tests……, (student must write tests number from observation table) and fails the tests……, (student must write tests number from observation table) for Identification as per I.P.

Viva Questions:

- Detail the physical characteristics of sodium chloride.
- What is the effect of storing aspirin in a humid environment?
- Aspirin's chemical name is:
- Write the functional group of aspirin.
- Describe two uses for aspirin.
- What are two brands of aspirin?
- What dosage forms does aspirin come in?
- What is the aspirin dosage?
- Describe the action mechanism of aspirin.
- List two official aspirin formulations.
- List four manufacturers of aspirin as a bulk drug.

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