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Diploma in Pharmacy 1st Year

Pharmaceutical Chemistry

Experiment

To perform the identification test and test for purity of aspirin

Aim:

To perform the identification test and test for purity of aspirin

Reference :

‘ Dr. Gupta G.D. , Dr. Sharma Shailish , Kaur Baljeet ’ “Practical Manual of Pharmaceutical Chemistry” Published by Nirali Prakashan, Page no 70 - 72

Requirements:

Aspirin, test tube, water bath, sodium hydroxide, dilute sulphuric acid, ferric chloride, Nessler's cylinder, hydrogen sulphide solution, acetone, ferric ammonium sulphate solution, and standard salicylic acid solution

Procedure:

Identification Tests

- 1) 0.5g of aspirin should be boiled with 10 ml of sodium hydroxide solution for 3 minutes and then cooled. Excess of dilute sulphuric acid (10ml) should be added and precipitate is produced. White crystalline precipitate with acetic acid odour is produced. The precipitate should be dissolved in water and the ferric chloride Test solution should be added Deep violet colour is produced. (Presence of Phenolic - OH).
- 2) 0.5g of aspirin should be boiled with 10 ml of sodium hydroxide solution for 3 minutes and then cooled. Excess of dilute sulphuric acid (10ml) should be added and precipitate is produced. White crystalline precipitate with acetic acid odour is produced. 3ml of alcohol, 3ml of dilute Sulphuric acid should be added to the filtrate and then heated. The odour of ethyl acetate is detected.

Test for Purity

- 1) Test for Chloride: 0.25g of sample should be added to 75 ml of H₂O and is boiled for 5 minutes and then cooled. Sufficient amount of water should be added to restore the original volume and then filtered. The limit test for chlorides is passed in 25ml of the filtrate fraction.
- 2) Test for Sulphate: The limit test for sulphate should be passed using a 25ml sample of the filtrate obtained for the chlorides limit test.
- 3) Test for Heavy Metals: Not more than 10ppm determined by the following method. 2g of sample should be dissolved in 25ml of acetone and 1ml of H₂O and 10ml of Hydrogen sulphide solution should be added to this solution. Any colour produced is not darker than of a control made using 25 millilitres of acetone, 2 millilitres of standard lead solution, and 10 millilitres of hydrogen sulphide solution
- 4) Test for Salicylic Acid: Not more than 0.1% determined by the following method. 2.5g of sample should be dissolved in sufficient alcohol to produce 25ml (test solution), 48ml of water and 1ml of freshly prepared ferrous ammonium sulphate solution should be added in each of the two Nessler's cylinder. 1ml of 1N HCl and 2ml of ferric ammonium sulphate solution with sufficient H₂O should be diluted for preparing 100ml. 1ml of Std. Salicylic acid solution should be pipetted out in one cylinder while 1ml of the test solution should be pipetted out in another cylinder. The contents of the cylinder should be mixed. After 30 seconds of mixing the contents of the cylinders, the colour in the second cylinder is not more intense than in the cylinder containing the standard salicylic acid solution.

Result: The identification test and test for purity of aspirin were performed as per I.P.