EXPERIMENT NO -20

OBJECT: To perform limit test of heavy metals in given sample of sodium chloride.

REFERENCE:-

Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 58.

REQUIREMENTS

Chemical required: Thioglycolic acid, citric acid, Ammonia solution, ferric ammonium sulphate.

Apparatus required: Measuring cylinder, glass rod, pipette and Nessler's cylinder.

THEORY

Limit test of heavy metals is based on the reaction of metallic impurities with hydrogen sulfide in acidic medium to form brownish colour solution. Metals that response to this test are lead, mercury, bismuth, arsenic, antimony, tin, cadmium, silver, copper, and molybdenum. The metallic impurities in substances are expressed as parts of lead per million parts of the substance. The usual limit as per Indian Pharmacopoeia is 20

PROCEDURE STANDARD

Take 2 ml of standard lead solution and dilute to 25 ml with water. Adjust the pH between 3 to 4 by adding dilute acetic acid 'Sp' or dilute ammonia solution 'Sp. Dilute with water to 35 ml. Add freshly prepared 10 ml of hydrogen sulphide solution and dilute with water to 50 ml. Allow to stand for five minutes, view downwards over a white surface.

TEST

Weigh specific quantity of test substance, moisten with sulphuric acid and ignite on a low flame till completely charred. Add few drops of nitric acid and heat it. Allow to cool and add 4 ml of hydrochloric acid and evaporate to dryness. Moisten the residue with 10 ml of hydrochloric acid and digest for two minutes Neutralize with ammonia solution and make just acid with acetic acid. Adjust the pH between 3 to 4 and filter if necessary. Dilute with water to 35 ml. Add freshly prepared 10 ml of hydrogen sulphide solution. Dilute with water to 50 ml. Allow to stand for five minutes. View downwards over a white surface.

RESULT

Limit test for heavy metals was performed. The color produce in sample solution should not be greater than standard solution. If color produces in sample solution is less than the standard solution, the sample will pass the limit test of heavy metals and vice versa.