

Limit test for arsenic

BACKGROUND-

The IP prescribes limit test for arsenic as an impurity test for various drugs. The presence of arsenic in drugs even in traces is not desirable as it is toxic. The sample was dissolved in acids which converts the arsenic impurity into arsenious acid depending upon the valency state of arsenic present in the sample. The solution was treated with reducing agent (stannous chloride) to convert the pentavalent arsenic acid into trivalent arsenious acid. Arsenious acid undergoes reduction to form arsine gas and this gas reacts with mercuric chloride paper to give a yellow stain due to formation of mercuric arsenite.

Aim: Aim of this experiment is to carry out the limit tests for arsenic of the given samples.

REQUIREMENTS-

Sample: 10 gm Dextrose ($C_6H_{12}O_6$),

5gm $MgSO_4$,

5gm potassium bromide (KBr)

Apparatus: Analytical Balance,

Glass Rod,

Nessler's cylinder,

Filter paper,

Cotton,

Arsenic test apparatus (gutzeit's apparatus)

Chemicals: Dilute arsenic solution,

Stannated HCl,

Granulated Zinc,

Potassium Iodide,

Lead Acetate Solution,

Mercuric Chloride, Distilled water.

PROCEDURE-

Two sets of gutzeit's test apparatus was prepared for standard and test, cotton moistened with lead acetate solution, dried and kept inside the glass tube in such way that upper surface of the cotton should maintain a distance of 25mm to the top of the tube. Two rubber bungs are fitted, to sandwich the mercuric chloride paper of which one is fitted to the upper end and the other one just below it with paper between them. Both the bungs are been fixed by the use of spring clip in such way that gas evolved from the bottle must have to pass the 0.65cm internal circle of mercuric chloride paper.

Preparation of standard solution

The gutzeit's tube was to be filled with around 50ml of water to which 1ml of standard arsenic solution was added. To this 10ml of stannated HCl was added, 1g of potassium iodide and 10g of granulated zinc was added. Then the reaction was allowed to proceed for 40mins.

Preparation of test solution

Accurately weighed quantity of sample was taken and dissolved in 50ml of dissolved water which was previously placed in the gutzeit's apparatus. To the solution 10ml of stannated HCl, 1g of potassium iodide followed by 10gm of granulated zinc. Then the reaction was allowed to proceed for 40 mins.

Then the two solution's intensity was compared.

CONCLUSION-

The colour intensity observed in the test solution was less than the standard solution. Hence the given samples had passed the limit test for iron and those are of pharmaceutical grade.

REFERENCES-

1. Chatwal GR. Pharmaceutical inorganic chemistry (vol- 1), 2016.
2. Indian pharmacopoeia; 2016.