Date 21-12-21

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Aim :-

Ram Kumar 21/12/21

To perform the limit test for sulphates of the given test substance.

Reference :-

Requirements :-

(a) Glasswares : Nessler's cylinder, measuring cylinder and glass rod.

(b) Chemicals :- Potassium Sulphate, test substance, hydrochloric acid, barium sulphate reagent and distilled water.

Principle :-

The principle involved in the limit test for sulphates is precipitation method and then comparison of test solution with the standard solution containing a known amount of sulphates. The sulphates are precipitated as barium sulphate by reacting with barium chloride in the presence of hydrochloric acid. The hydrochloric acid used prevents the reaction of other acid radicals with barium chloride.

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because in the presence of hydrochloric acid, only sulphates are precipitated.

$$SO_4^{2-} + BaCl_2 \xrightarrow{HCl} BaSO_4 + 2Cl^-$$

Due to the formation of precipitates, the solution appears turbid and the extent of turbidity depends on the amount of sulphates present. If the turbidity produced by the test is less than that of standard, it means that the sample contains sulphates within the prescribed limits.

Reagent Preparations:

- **Barium Sulphate Reagent:**

Dissolve 12g of barium chloride ($BaCl_2 \cdot 2H_2O$) in 1000 ml water to make 0.05M barium chloride solution. To 15 ml of the prepared solution, add 55 ml water, 20 ml alcohol, 5 ml of 0.0181 % w/v potassium sulphate (K_2SO_4) solution and makeup the volume upto 100 ml.

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- Standard Potassium Sulphate Solution:

Accurately weigh 0.1089 g of K_2SO_4 was taken and the volume was made up to 100 ml with water.

- Test sample:

(i) Sodium chloride: Dissolve 2g of sodium chloride in 15 ml of water.

(ii) Sodium bicarbonate: Dissolve 2g of sodium bicarbonate in 15 ml water.

Procedure:

Test	Standard	Interference / Reasons
1. Take 1ml of 25% w/v solution of barium chloride in Nesslers' cylinder.	Take 1ml of 25% w/v solution of barium chloride in Nesslers' cylinder.	The barium ions produced, react with sulphates to form precipitates (opalescence) of barium sulphate. $BaCl_2 + SO_4^{2-} \rightarrow BaSO_4 + 2Cl^-$
2. Add 1.5 ml of ethanolic sulphate standard solution (10 ppm of SO_4^{2-}).	Add 1.5ml of ethanolic sulphate standard solution (10 ppm of SO_4^{2-}).	The ethanolic solution of potassium sulphate increases the sensitivity of test and alcohol prevents

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			the super saturation of barium sulphate precipitates.
3.	Add 15 ml of test sample solution prepared as directed in individual monograph.	Add 15 ml of standard potassium sulphate solution prepared as directed in individual monograph.	It gives total sulphate ion present in the sample.
4.	Add 0.15 ml of 5M acetic acid.	Add 0.15 ml of 5M acetic acid	The acetic acid prevents the precipitation of various anions such as borate, oxalate, phosphates etc. present in the sample.
5.	Make up the volume with sulphate free water upto 50 ml.	Make up the volume with sulphate free water upto 50 ml.	Equal volume of opalescence are easily compared by viewing in black background.

Conclusion:

If opalescence produced in the standard is more than that of test, the sample complied the limit test of sulphate as per I.P. 1996.

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