

VEER NARMAD SOUTH GUJARAT UNIVERSITY
M.SC. CHEMISTRY
PHARMACEUTICAL AND ORGANIC CHEMISTRY
PRACTICALS-CPC-505

Drug Estimation

Time: 3¹/₂ hrs.

Marks:

Aim: Determination of percentage purity of the given sample of Isoniazide (C₆H₇ON₃) or the amount of Isoniazide in the given tablet.

Note: First note the weight of tablet and then powder it. Use this powder for estimation.

Reagents:

0.1N Brominating solution, 10% KI solution.

0.1N Na₂S₂O₃ solution, Conc. HCl and starch solution.

Assay:

Weight out accurately about 0.4 gm of the isoniazide sample and dissolve it in distilled water. Transfer it to 250 ml measuring flask and diluted it up to the mark with distilled water. Pipette out 25 ml of this diluted solution in a glass stopped bottle. Add 25 ml 0.1N brominating solution and cool it at 15⁰ C in ice bath. Add 5 ml (Conc.) HCl. Shake well and allow it to stand for 15 minutes in a bath maintained at about 15⁰ C. Add 10 ml 10% KI solution and titrate the liberated iodine against 0.1 N Na₂S₂O₃ solution using starch mucilage as an indicator.

Blank Titration:

Repeat the above operation with the same quantities of the same reagents omitting the isoniazide drug sample. The Difference between the above two titrations represents the amount of bromine solution required by the sample.

Standardization of bromine solution:

Place 25 ml 0.1 N brominating solution in a 250 ml conical flask. Add 10 ml 10% KI solution and 5 ml Conc. HCl and titrate against 0.1 N Na₂S₂O₃ solution using starch mucilage as an indicator. Calculate the exact normality of the brominating solution

Calculation: 1 ml 1.0 N brominating solution = 0.03429 gm of isoniazide.

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Time: 3¹/₂ hrs.

Marks:

Aim: Determination the amount of Glucose in the given solution (Iodometrically) in a measuring flask bearing your table number.

Process:

Diluted the given solution up to the mark with distilled water. Take 25 ml of the diluted solution in a glass Stoppard bottle. Add to this 5 ml of 15% sodium carbonate solution and 25 ml of iodine solution (persistent of yellow color) measured accurately. Stopper the bottle and keep it for 30 minutes in a dark place. Then acidify the mixture with 25ml of 1 M HCl and titrate excess of iodine against 0.1N sodium thiosulphate solution, using starch as an indicator.

Prepare 0.1 N sodium thiosulphate solution and standardize the given iodine solution.

Calculation:

1.0 ml of 0.1 N I₂ = 0.009 gm of glucose tabulated your results as under:

1. 0.1 N sodium thiosulphate required for 25 ml of iodine solution (Added) = _____ ml.
2. 0.1 N sodium thiosulphate required for 25 ml of diluted glucose solution = _____ ml.
(Back titration i.e. unused)
3. I₂ consumed in terms of 0.1 N sodium thiosulphate (used up) = _____ ml.
4. Amount of glucose in the given solution = _____ gm.

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Time: 3¹/₂ hrs.

Marks:

Carryout the assay of sodium chloride (NaCl) in the given solution in a measuring flask bearing your table number.

Diluted the given solution up to the mark with distilled water, Take 25 ml. of the diluted solution (equivalent to..... gm., weight of the given compound) in a conical flask. Add to it 40 ml. of 0.1 N Silver Nitrate solution. Shake vigorously for 1-2 minutes and titrate with 0.1 N Ammonium thiocyanate (NH₄SCN) using 2 ml of Ferric ammonium sulphate as an indicator.

Calculate the percentage purity.

Each ml of 0.1 N AgNO₃ = 0.005844 gm of NaCl.

$$\text{Percentage Purity} = \frac{[(N_1 \times V_1) - (N_2 \times V_2)] \times 0.005844 \times 100}{0.1 \times w}$$

Where V₁ = Volume of silver nitrate solution.
 N₁ = Normality of silver nitrate solution.
 V₂ = Volume of Ammonium thiocyanate solution.
 N₂ = Normality of Ammonium thiocyanate solution.
 W = Weight of the sample.

Note Before: Ask for the Weight of the sample (w) after showing your “Burette Reading ” to one of the examiner.

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Drug Estimation

Time: 3¹/₂ hrs.

Marks: 25

Aim: Determine the amount of analgin in the given solution.

Determine the given solution up to the mark. Take 25 ml diluted solution in a conical flask. Add 25 ml of distilled water and acidify the solution with 25 ml of 0.1 N HCl then immediately titrate the solution against 0.1 N I₂ solution using starch as an indicator. At the end point the color changes from colorless to blue.

Standardized of I₂ Solution:

Prepare exact 0.1 N solution of Na₂S₂O₃ solution. Take 25 ml iodine and titrate against 0.1 N (exact) Na₂S₂O₃ solution. Using starch indicator and calculate the exact normality of I₂ solution.

To prepare 0.1 N sodium thiosulphate solution.

Dissolved accurately weighed 6.205 gm of Na₂S₂O₃. 5H₂O in water and make up to 250 ml.

Calculation:

1.0 ml 0.1 N I₂ solution = 0.01757 gm of Analgin.

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