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Diploma in Pharmacy 1st Year
Pharmaceutical Chemistry
Experiment

To perform the assay of sodium chloride by precipitation titration.

Aim:

To perform the assay of sodium chloride by precipitation titration.

Reference :

‘ Dr. Gupta G.D. , Dr. Sharma Shailish , Kaur Baljeet ’ “Practical Manual of Pharmaceutical Chemistry” Published by Nirali Prakashan, Page no 41 - 44

Apparatus and Material Required :

Burette, burette stand, conical flask, volumetric pipette, beaker, volumetric flask, funnel, glass rod, wash bottle, digital/analytical balance, ultrasonicator, silver nitrate ($AgNO_3$), sodium chloride (NaCl), and potassium chromate (K_2CrO_4)

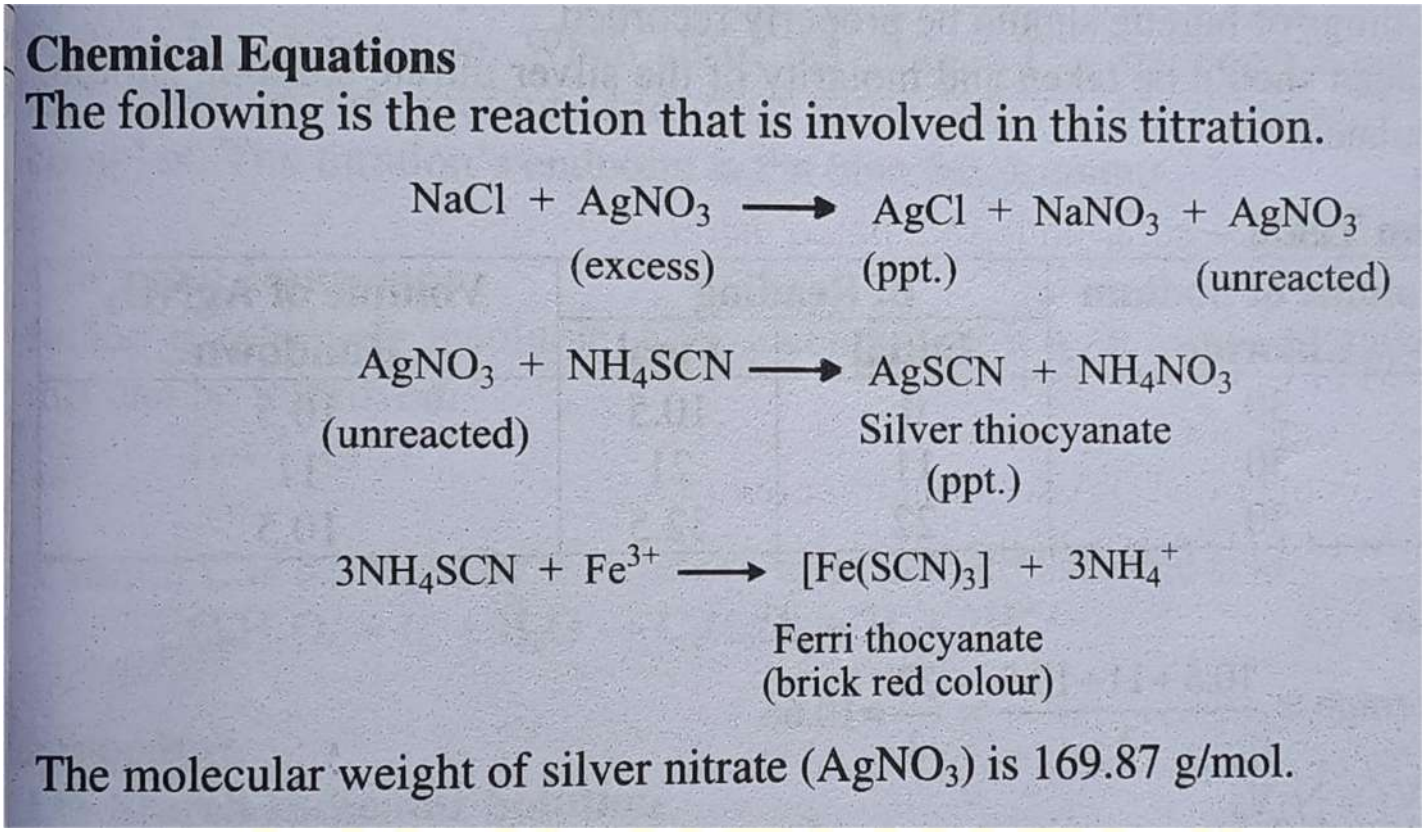
Theory:

- The principle of assay by Volhard's method is based on indirect volumetric precipitation titration. In this method, nitric acid solution is used to acidify NaC (or other chlorides) solution, and then in the presence of nitrobenzene, thin solution is treated with measured excess amount of standard solution of silver nitrate. Some moles of silver nitrate are consumed in the reaction with NaCl and the remaining unreacted silver nitrate is determined by reaction with NaCl and solution of amunguin thiocyanate. In this titration, solution of fewith a standard sulphate (ferric alum) is used as an indicator.

- Nitrobenzene forms an organic layer around the precipitate particles of silver chloride, thus, rendering it completely insoluble. This prevents saver chloride react with sulphate solutionocyanate. The nitric acid added prevents ferric ammonium sulphate solution (i.e., the indicator) to hydrolyse.

Chemical Equations

The following is the reaction that is involved in this titration.



Procedure:

Preparation of 0.1 N Silver Nitrate

16.99 gm of silver nitrate should be taken using a pipette and dissolved in 500 ml of distilled water in a volumetric flask and mix it properly. The volume is maintained to 1000 ml once it has completely dissolved.

Preparation of 0.5 N Sodium Chloride

05.84 gm of previously dried sodium chloride should be taken and dissolved in 500 ml of distilled water in a volumetric flask. It should be mixed properly. The volume should be maintained to 1000 ml once it has completely dissolved.

Preparation of Sodium Chloride Solution (Sample)

01 gm of sodium chloride should be taken and dissolved in 50 ml of distilled water in a volumetric flask. It should be mixed properly. The volume should be maintained to 100 ml once it has completely dissolved.

Procedure

1) All glassware should be cleaned and dried according to standard laboratory procedures.
2) The burette should be washed with distilled water before filling for the titration and again it should be washed with a portion of titrant solution. To ensure that all of the solution in the burette is the desired solution and not a polluted or diluted solution, pre-rinsing is required.
3) The unknown stock solution of titrant should be taken in a clean and dry beaker and then the burette should be filled using the funnel.
4) Air bubbles should be removed from the burette and adjust the reading to zero.
5) 10.00 ml of prepared sample solution of sodium chloride should be taken and poured into a conical flask.
6) 2 3 drops of potassium chromate solution should be added as an indicator.
7) The sample solution should be titrated with silver nitrate solution till the endpoint is obtained.
8) At the end of the reaction, a brick red colour indicates the actual endpoint of titration.
9) The titration should be repeated three times to obtain accurate results.
10) The readings of burette should be properly recorded.
11) Their mean should be taken and molarity of the silver nitrate solution should be calculated.

Observation Table

S. No.	Volume of Sodium Chloride	Burette Reading		Volume of AgNO, Rundown
		Initial	Final	
1	30	0	10.5	10.5
2	30	11	21	11
3	30	22	32.5	10.5

Calculation

Average = $\frac{10.5++11+10.5}{3}$ = 10.66

$N_1V_1 = N_2V_2$

$N_1 = 10.6$

$N_2 = N_1V_1/V_2$

$= 0.1 \times 10/10.6 = 0.0943$

%Purity = 0.02063 x V x N x 100 / NxW

Where,

$N_1 = 19m$

$N_2 = 0.094$

$V_2 = 10.66$

Equivalent factor = 0.02063

$$= \frac{0.02063 \times 10.66 \times 0.094}{1 \times 0.1}$$

%Purity = 20.6%

Result: The percentage purity of the sodium chloride (NaCl) sample was found to be 20.6%

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