

EXPERIMENT: 20

AIM:

TO PERFORM ASSAY OF ISONIAZID (REDOX TITRATION) BY BROMATOMETRY AND STANDARDIZATION OF TITRANT

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REQUIREMENTS

A. Glassware & Instruments

1. Pipette
2. Burette
3. Filter funnel
4. Conical flask
5. Graduated cylinders
6. Retort stand
7. Clamp

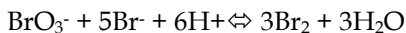
B. Chemicals & Reagents

1. Potassium Bromate
2. Isoniazid
3. Methyl orange or Methyl red
4. HCl
5. Water

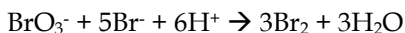
PRINCIPLE

Analysing Ionized IP Anti-tubercular drug: In the presence of HCl solution, this is determined by adding potassium bromide and titrating potassium bromate directly. As the titration continues, bromine is released and reacts with isoniazid as depicted below. An Azo-dye, methyl red solution, is used as an indicator and decolorizes by oxidation at the end point.

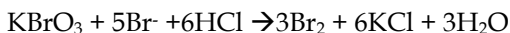
Potassium Bromate is a potent oxidising agent that is reduced easily to bromide in the presence of HCl, which is then oxidised by bromate to produce free bromine.



Therefore, the equivalent is $1/6$ moles, $\text{KBrO}_3/6$, or $167/6$, which is equivalent to 27.88. At the endpoint of titration, bromine is no longer present.



Therefore,



THEORY

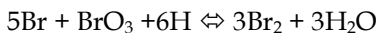
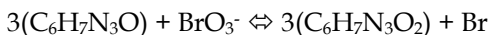
Bromate of Potassium (KBrO_3) is used as titrant. It is utilised as a primary standard and is a potent oxidizer.

Typically, potassium bromate is available in a highly pure form. The assay value of the analytical reagent is 99.9 percent. Since the substance can be dried at 120-150 degree C, is anhydrous, and is preserved indefinitely in aqueous solution, it can be used as a primary standard. The only disadvantage is that one-sixth of the relative molecular mass is a relatively small amount.

The reaction occurs in an acidic medium (typically 1M HCl is used).



Direct titration takes place



Br_2 + indicator (Methyl Orange/Methyl Red) $\rightarrow$ colourless

PROCEDURE

Preparation of 0.1N KBrO_3 solution

- Molecular weight of KBrO_3 = 167g/mol
- $$N = \frac{\text{weight} \times 1000}{\text{molecular weight} \times \text{volume taken}}$$
- $$\text{Weight} = \frac{0.1N \times \frac{167g}{\text{mol}} \times 250ml}{1000}$$
- Weight = 4.175g
- KBrO_3 is a standard substance and can be easily obtained in pure state by recrystallization from water.
- KBrO_3 is dried at 120-150 °C for 1-2 hr and then cooled in a desiccator.
- Dissolve 4.175 of KBrO_3 in sufficient quantity of water to produce 250ml.

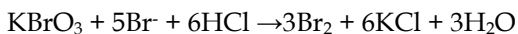
Preparation of 0.1N Isoniazid solution

- Molecular weight of **Isoniazid** = 137g/mol
- $$N = \frac{\text{weight} \times 1000}{\text{molecular weight} \times \text{volume taken}}$$
- $$\text{Weight} = \frac{0.1N \times \frac{137g}{\text{mol}} \times 250ml}{1000}$$
- Weight = 3.425g
- Dissolve 3.425g of Isoniazid in sufficient quantity of water to produce 250ml.

Assay of Isoniazid

1. Take the analyte solution (i.e. $C_6H_7N_3O$) in a conical flask at low temperature.
2. Add 1M HCl to the conical flask and make the solution acidic.
3. Add 2/3 drops of indicator i.e. methyl orange or methyl red so solution becomes red in acidic medium.
4. Titrate the above solution using standard $KBrO_3$ solution.
5. At the end point the solution becomes colourless from red colour.

REACTION



CALCULATION

Observation table

S No.	Volume of Isoniazid solution (ml)	Burette reading (ml)		Volume of Potassium bromate used (ml)
		Initial	Final	
1.				
2.				
3.				

Calculation and factor calculation

Factor calculation of Isoniazid

Molecular weight of Isoniazid = 137 g/mol

1N 1000ml Isoniazid = 137g

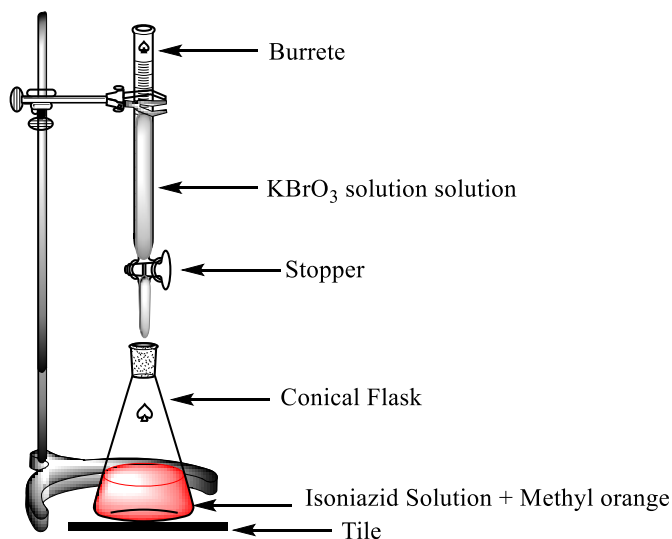
0.1 N 1ml Isoniazid = $137/10000 = 0.0137$ g

Percentage purity calculation of Isoniazid

% Isoniazid (by weight) =

$$\frac{\text{Observed normality (KBrO}_3\text{)} * \text{Volume of KBrO}_3\text{ (burette reading)} * \text{Factor} * 100}{\text{Theoretical normality (KBrO}_3\text{)} * \text{Weight (Isoniazid)}}$$

DIAGRAM



APPLICATION

In conjunction with other medications, isoniazid is used to treat active tuberculosis (TB) infections. Additionally, it is used alone to prevent active tuberculosis infections in individuals who may be infected with the bacteria (people with positive TB skin test). Isoniazid is an antibiotic that works by inhibiting bacterial growth.

This antibiotic is only used to treat bacterial infections. It is ineffective against viral infections (such as common cold, flu). Utilizing an antibiotic when it is not necessary can render it ineffective against future infections.

RESULT

Percentage purity of Isoniazid solution is