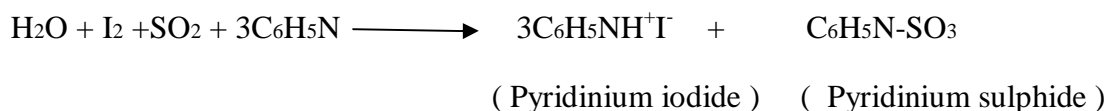


## KARL FISCHER TITRATION

( For determination of moisture content)

- Karl Fischer proposed a reagent which reacts quantitatively & selectively with water to measure a moisture content.
- Karl Fischer reagent consist of iodine,SO<sub>2</sub> in a mixture of anhydrous pyridine & anhydrous methanol
- Recently pyridine has been replaced by other amines particularly imidazole
- It involves a quantitative reaction of water with iodine and SO<sub>2</sub> in the presence of lower alcohol such as methanol & organic base such as pyridine



Reaction of iodine and SO<sub>2</sub> in the presence of pyridine and water forms pyridinium sulphide and pyridinium iodide.

In this method water react with iodine in quantitative amt which means that 1 mole of iodine react with 1 mole of water

-Reaction of pyridinium sulphide with methanol gives a complex.

These are 2 methods for the determination of moisture content

- Volumetric method
- Coulometric method

(Depending upon the iodine provided)

### Volumetric Method

In this method iodine required for reaction with water is previously dissolved in the water determination test standard (Karl Fischer reagent) & water content is determined by measuring the amount of iodine consume as a result of reaction with water in a sample.

-two methods of titration can be done to determine the end point of the reaction

- Amperimetric method- at constant voltage(change in current is measured)
- Potentiometric method – at constant current(change in potential is measured)

### Procedure for Karl Fischer

- Direct method
- Back titration method

## Direct method

- i) Take 25 ml of methanol for water determination in a dried titration flask & titrate with water determination test standard to the end point
- ii) Weigh accurately a quantity of sample (usually containing 10 to 50 mg of water) & transfer it quickly into titration flask & dissolve by stirring
- iii) Titrate the solution with water determination test standard to the end point under rigorous stirring.

## Calculation

$$\text{Water (moisture) present} = \frac{\text{Vol (ml) of KF reagent} \times f \text{ (mg / ml)} \times 100}{\text{wt of sample (mg)}}$$

F = no of mg of water of water correspondence to 1 ml of water determination test standard (need specific amt of Karl Fisher reagent) which reacts with specific amount of water.

- This is specific for different sample.

## 2) Back titration method

- Take 25 ml of methanol for water determination in dried titration flask & titrate with water determination test standard to the end point.
- Weigh accurately suitable quantity of sample & transfer the sample to titration vessel.
- Add an excess of and definite volume of water determination test standard
- Stir the content for 30 minutes protecting from atmospheric moisture.
- Titrate the solution with water-methanol standard solution from the burette.

## Calculation

$$\text{Water (moisture) present} = \frac{(\text{Vol of Karl Fischer reagent}) \times f^1 - \text{Volume of water-methanol solution consumed} \times f^1}{\text{wt of sample}} \times 100$$

$f^1$  = no of mg of water in 1 ml of water-methanol standard solution

## Coulometric Method

In this method, iodide are used instead of iodine & the time consumed in formation of iodine from iodide can be measured.

- In this, Karl Fischer reagent contain potassium iodide instead of iodine

- In this titration first iodine is produced by electrolysis of reagent containing iodide ion & then the water content in a sample is determined by measuring the quantity of electricity which is required for production of iodine.
- The titration shall consist of anodic & cathodic compartment separated by ceramic layer(diaphragm)
- The anionic compartment contain analyte solution which include SO<sub>2</sub>,KI & pyridine needed for chemical reaction , methanol or ethanol is used as solvent.
- In coulometric Karl Fischer titration when iodine is complete & the amt of water in sample is calculated by measuring the current needed for electrochemical generation of iodine from iodide.
- If ceramic layer is absent , the change of iodine is reversed , hence the diaphragm plays an role in titration cell.

**Anodic reaction** =  $2I^- \rightarrow I_2 + 2e^-$

**Cathodic reaction** =  $I_2 + 2e^- \rightarrow 2I^-$

*Iodine is a decolorizer which give sharp increase in a current.*

### Interfering Reaction of Karl Fischer Titration

- Karl Fischer water determination is only specific ,if no side reaction takes place with Karl Fischer reagent.
- this means that no water should be released in side reaction& the sample react with any of the component of a reagent.

Some of the interfering reactions are summarized below

-Carboxyl grp(aldehyde & ketones)- Aldehyde & Ketones can react with sulphurdioxide & water & water to form bisulphate complex .

Aldehyde & ketones can combine with methanol in Karl Fischer reagent to form acetols and ketols

### Prevention

Using methanol free Karl Fischer reagent or replacing methanol by higher alcohol

Eg- 2 –methoxy methanol

-By making use of different reaction rate & slowing down acetols and ketols formation [by carrying out titration at -10° C]



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