

The preparation and assay of Aqueous Iodine Solution (Lugol's Solution)

- ▶ It contains 5% w/v ***Iodine*** (limits 4.75– 5.25% w/v) and 10% w/v ***Pot. Iodide*** (limits 9.5– 10.5% w/v).
- ▶ Used in pre-operative treatment of thyrotoxicosis.
- ▶ **Preparation:**

I₂ 50gm

KI 100gm

purified water sufficient to 1000ml

- *Dissolve KI in a little amount of water then add the I₂ and shake well until the I₂ is completely dissolved, then add sufficient water to 1000ml using volumetric flask.

- Assay: Dilute 25ml of the original sol. to 100ml with distilled water, then:

a) Assay of I_2 ,

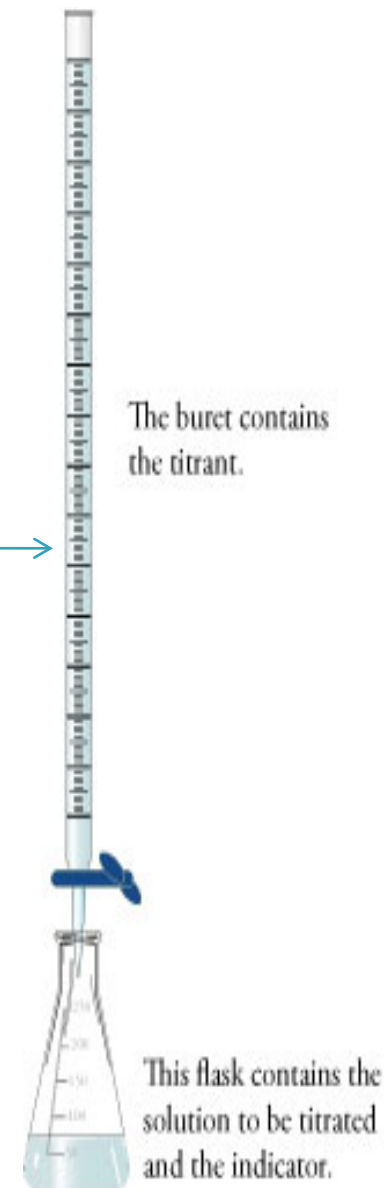
note: we must use stoppered flask

Each 1 ml of N/10 $Na_2S_2O_3 \equiv 0.01269$ gm I_2

\approx N/10 $Na_2S_2O_3$ 

dark brown, titrate  yellow then add 1 ml starch
the color change to dark blue complete tit.
until the end point colorless

10ml of unknown
10ml D.W 

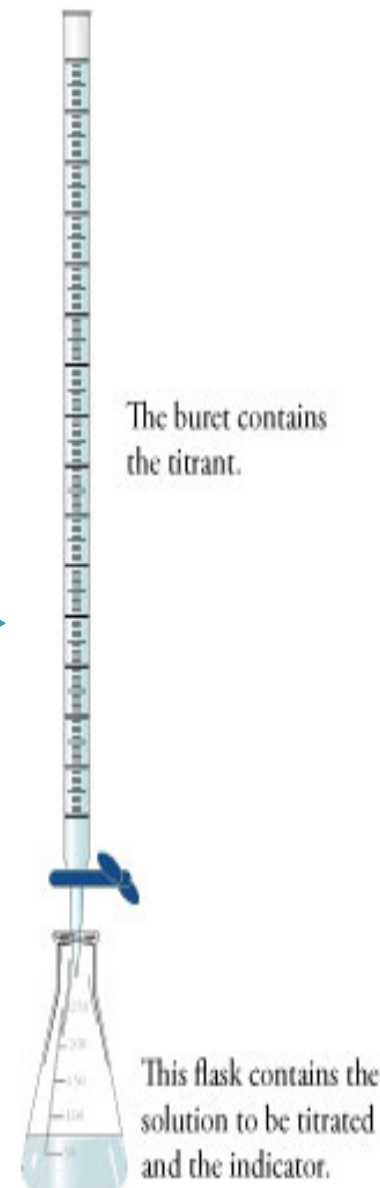


b) Assay of KI,

** titrate with M/20 KIO₃ (Pot. Iodate) until **dark brown** sol. change to **pale brown**, add 1ml of amaranth sol.; the color of sol. Change to **red** and then continue tit. Until the color of sol. change to **yellow -orange***

M/20 KIO₃ →

5ml of unknown
10ml D.W
20ml conc. HCl



- ▶ From the no. of ml. of M/20 KIO_3 required subtract $\frac{1}{4}$ no. of ml of N/10 $\text{Na}_2\text{S}_2\text{O}_3$ required in the assay of I_2
each 1ml of the remainder is \equiv 0.0166gm of KI

▶ Chemical principle for I₂ assay:

I₂ will oxidize chemically equivalent amount of sod. thiosulfate.



▶ Chemical principle for KI assay:

KIO₃ (pot. Iodate) is fairly strong oxidizing agent, it can quantitatively react with both iodide I⁻ and iodine I₂.

*If the conc. of HCl not exceed 1 N the reaction:



*In the presence of conc. HCl exceeding 4N, I₂ produced by the previous reaction is further oxidized to iodine monochloride (ICl):



* so the overall reaction between KIO₃ & KI in the presence of high conc. HCl >4N can be expressed by:



The presence of high conc. HCl is required to decrease :



stable cpd

- ▶ In this experiment we use amaranth indicator, it is a dye, dyes which are destroyed or changed in the presence of oxidizing titrant can be use as indicator.
- ▶ We can not use starch as indicator because starch_ iodide complex can not be formed in high acid conc. used in the iodate titration.

▶ ***Chemical factor. Homework***

Each 1 ml of 0.1 N $\text{Na}_2\text{S}_2\text{O}_3 \equiv 0.01269\text{gm}$ of Iodine

Each 1 ml of M/20 $\text{KIO}_3 \equiv 0.0166\text{gm}$ of KI

Each 1 ml of M/20 $\text{KIO}_3 \equiv 0.02538\text{gm}$ of iodine

▶ Calculation:

▶ **For I₂:**

<u>original sol.</u>	<u>Vol.</u>
25	100
X	10

 $\left. \vphantom{\begin{matrix} \text{original sol.} \\ 25 \\ X \end{matrix}} \right\} X = 2.5 \text{ ml of original sol.}$

$V' \times \text{ch.fact. } 0.01269 \times 100/2.5 = \%w/v \text{ of iodine in orig. sol.}$
 V' the corrected volume of sod. thiosulfate.

▶ **For KI:**

<u>original sol.</u>	<u>Vol.</u>
25	100
X	5

 $\left. \vphantom{\begin{matrix} \text{original sol.} \\ 25 \\ X \end{matrix}} \right\} X = 1.25 \text{ ml of original sol.}$

$$\begin{array}{c}
 \downarrow \\
 V_{\text{KIO}_3 \text{ M/20}} \text{ (req. for KI)} = V_{\text{KIO}_3 \text{ M/20 (used in the total assay)}} - \frac{1}{4} V'_{\text{Na}_2\text{S}_2\text{O}_3 \text{ 0.1N}}
 \end{array}$$

$\text{Vol. of KIO}_3 \text{ (req. for KI)} \times 0.0166 \times 100/1.25 = \%w/v \text{ of KI}$

- ▶ It is preferred to use iodate (KIO_3) of known M rather than of known N, it depends on the nature of reaction.
- ▶ KIO_3 sol. used in the titration of sol. containing both iodide and iodine.
- ▶ The equivalent of KIO_3 in its reaction with KI differs from its equivalent when react with iodine.