**Answer to problem on sodium hydroxide solution**

1. The volume of 1.11 *N* hydrochloric acid solution used in titrating sodium hydroxide sample was for total alkali regardless of whether sodium carbonate is present as impurity or not; *i. e.,* the same volume of hydrochloric acid solution would be consumed if sodium carbonate was precipitated as barium carbonate (explain this fact by the related balanced chemical equations).

HCl NaOH

𝑁1 × 𝑉1 = 𝑁2 × 𝑉2 1.11 × 4.5 = 𝑁2 × 10

𝑁2 = 0.4995 the normality of the prepared sodium hydroxide solution

1. Any solid sodium hydroxide sample taken from the original container would contain sodium carbonate as impurity by 3% w/w. Thus, you have to calculate how much sodium hydroxide was used to prepare the

10 milliliters- sample. This is calculated from the volume of hydrochloric acid used for total alkali:

1.11 *N* HCl 1 *N* HCl

𝑁 × 𝑉 = 𝑁' × 𝑉' 1.11 × 4.5 = 1 × 𝑉'

𝑉' = 4.995 mL of 1 *N* hydrochloric acid solution would be required for total alkali (V3)

w𝑡 = 𝑉3 × 𝑐ℎ. f𝑎𝑐𝑡o𝑟 w𝑡 = 4.995 × 0.04

w𝑡 = 0.1998 g of total alkali (sodium hydroxide and the impurifying sodium carbonate) calculated as sodium hydroxide was used originally to prepare 10 mL of solution

3

100

× 0.1998 = 0.005994 g of sodium carbonate was present in the

sample used to prepare 10 mL of solution

1

w𝑡 = 𝑉2 × 𝑐ℎ. f𝑎𝑐𝑡o𝑟 0.005994 = 𝑉2 × 0.053

𝑉2 = 0.113 mL of 1 *N* hydrochloric acid solution was consumed by the amount of sodium carbonate present in the sample assayed

1.11 *N* HCl 1 *N* HCl

𝑁 × 𝑉 = 𝑁' × 𝑉'

1.11 × 𝑉 = 1 × 0.113

𝑉' = 0.1 mL of 1.11 *N* hydrochloric acid solution would be consumed by the amount of sodium carbonate present in the sample assayed

It is important to note that the percent w/w is related to solid samples. If you were asked to calculate the percent w/v, then this is related to the prepared solution as follows:

for total alkali: 0.1998

10

× 100 = 1.998 % w/𝑣

for sodium carbonate: 0.005994

10

× 100 = 0.05994 % w/𝑣

2



MgSO4. 7H2O

Mwt.=246.5gm/mole



* Contain not less than 99.5% MgSO4 calculated with reference of dried substance.
* Description: colorless crystal, or white crystal powder.
* Solubility: soluble at 20˚C in 1.5 parts of water, sparingly soluble in alcohol 96% i.e. practically insoluble in 96% ethanol.
* Uses: laxative agent.



* **Assay:**

***Each 1ml of M/20 EDTA≡0.012325gm MgSO4.7H2O***

***Note***:



*End point from pink to blue or bluish violet*

*M/20 EDTA*

10ml unknown 10ml buffer

0.1gm mordant black indicator



* The technique involves titrating *metal ions* with a *complexing agent* or *chelating agent (Ligand)* and is commonly referred to as ***complexometric titration***. This method represents the analytical application of a complexation reaction.
* In this type of titration we use ***metal ion indicator.***
* **Chemical principle**:
* It is type of complexometric titration.



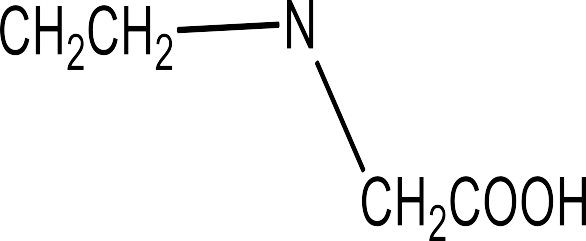
* Magnesium ions form relatively weak complex with (EDTA

;Ethylenediaminetetra-acetic acid).

* The stability of metal complex is pH dependent. Lower the pH of the solution, lesser would be the stability of complex (because more H+ ions are available to compete with the metal ions for ligand)
* But at pH 10 the reaction is rapid and quantitative .



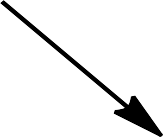
* NH3- NH4Cl buffer is used to maintain this pH.
* EDTA which is polyamine carboxylic acid is one of the most frequently used chelating agent:



EDTA



* EDTA form soluble, stable 1:1 chelates with metal ion.





* Because of the low water solubility of the free acid form of EDTA, the disodium salt (Disodium EDTA ) is used in preparation of std. sol.
* **Indicator: Metal Indicator**
* Metal indicators are used to detect the end point in complexometric titration.
* They are dyes which produce one color in the presence of metal ions and a different color when they are absent.
* The color changes are the result of displacement of the H⁺ ions by the metal.
* The metal indicator form 1:1 complex (MI) which

must be weaker than the Metal-EDTA complex.









* **Chemical factor:** it forms 1:1 chelate so; 1Mwt. MgSO4.7H2O≡ 1Mwt. EDTA

0.012325gm MgSO4.7H2O≡ 1ml of M/20 EDTA

* **Chemical principle:**
* At the onset of titration, the buffered reaction media contains the MI complex and free M:



* When the titrant (EDTA) is added to the system, it will progressively chelate the free metal ions in the solution, and then a competitive reaction will take place, 1:1metal ions and EDTA, since the MI complex is weaker than M\_EDTA.



* Finally, at the equivalent point, EDTA removes the last traces of metal ions from its colored complex with ind.(MI) to free metal ind.(I) of another color.



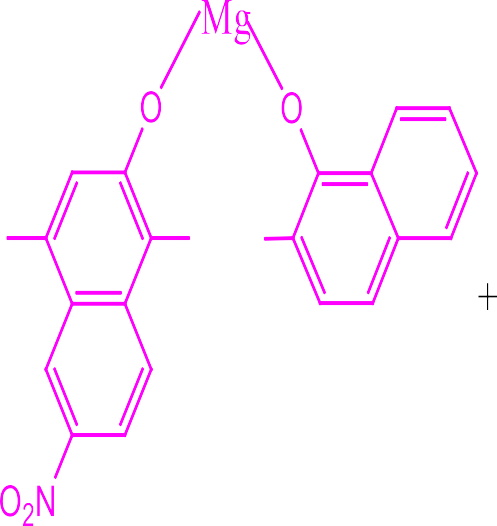




* **Mordant black indicator:** it has blue color at pH10 and most of its complexes are reddish.

***Note:*** In pH less than *6.3* and more than *11.5*, the indicator and its complexes are reddish,

so it is necessary to carry out the titration in the presence of ***buffer at pH 10***.









* **Calculation:**

***V ch. factor= gm wt. of MgSO4.7H2O wt 100/10= %w/v of unknown.***



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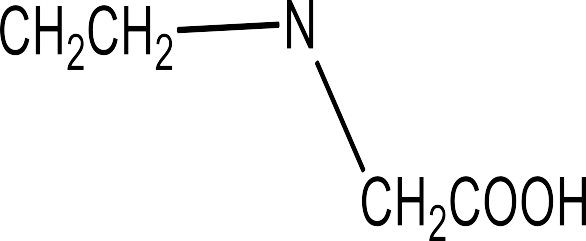
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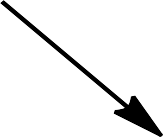
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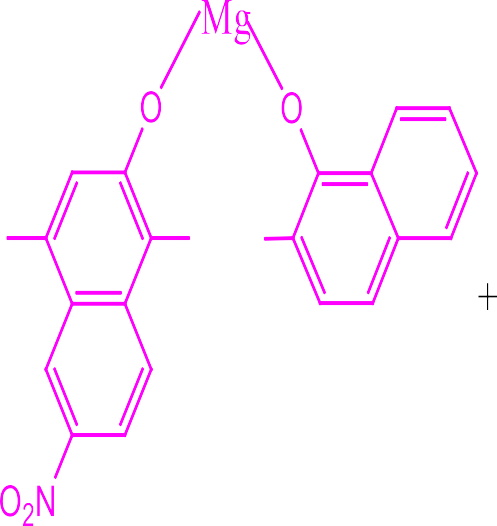




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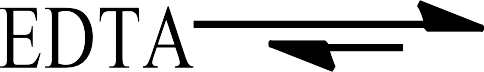
**ASSAY OF MAGNESIUM HYDROXIDE IN**

**ANTACID SUSPENSIONS (MAALOX)**



* Maalox suspension contains two active ingredients, aluminium hydroxide and magnesium hydroxide. These are both medicines called antacids. They are used to reduce the acidity of the stomach's juices.
* Magnesium ion is complexed with EDTA. The solution is titrated with standard EDTA solution to the first permanent appearance of violet color.
* Mordant black is used as the indicator.
* **Chemical principle**:







The overall reaction is given by :

MgI + Mg + EDTA Mg-EDTA + I

(colored metal indicator (original color

complex) of indicator)

* **Procedure**:

*note*: We must first dilute the Maalox suspension before assay:

1. Transfer 10mL unknown to 250-mL conical flask.
2. Add 5mL of pH-10 buffer.
3. Add 10ml D.W.
4. Add about 0.1gm of *Mordant Black indicator*.
5. Titrate with M/40 EDTA solution until the color

changes from pink to violet color.

* **Calculation:**
* Molecular Weight of Mg(OH)2 = 58.3
* mg of Mg(OH)2 Found = M ( EDTA ) x V ( EDTA )

in mL x Mwt. of Mg(OH)2

* **Complexometric titrations** use reagents containing ligands that form complexes with metal ions. The indicator is a substance that forms a colored complex with the ions, but this complex must be weaker than that formed with the reagent. The metal ion complexes preferentially with the reagent, until at the end- point no metal-indicator complex remains, and its colour disappears.

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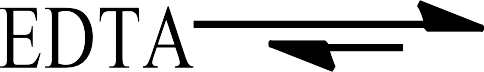
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Borax or Sod. borate or Sod. Tetraborate

Chemical formula Na2B4O7.10H2O Mwt.= 381.4 gm

* **Physical properties**: Borax is sod.tetraborate decahydrated, should be kept in well closed container, soluble in 20 parts of water.
* **Uses**; antiseptic and astringent in mouth paints.
* **Procedure**: 10mL of unknown

10mL of distilled water

2drops of methyl red indicator Titrate with *N*/2 HCl solution, the color of sol.

changes from yellow pink–orange(**1st end**

**point**).

Record the volume of *N*/2 HCl consumed.



###### Boil and cool the solution,

Add 4gm mannitol and 2drops of phenolphthalein ind. The color of sol. is pink

###### Now titrate with *N*/1 NaOH until the color of sol. changes from pink yellow pink(**2nd end point**).

Record the volume of *N*/1 NaOH consumed.



1st titration

2nd titration

*N*/2 HCl solution *N*/1 NaOH solution

10mL of unknown 10mL of D.W 2drops of methyl red indicator

Boil , cool and add 4gm mannitol and 2drops of ph.ph ind.

* **Chemical principle:**
* Borax is a salt of weak acid with strong base. Na2B4O7.10H2O + 2HCl 2NaCl+ 4H3BO3+5H2O
* Boric acid(H3BO3) will be liberated, it is very weak acid.
* Boil the solution to remove CO2 .
* Add mannitol because boric acid is weak acid to convert it into strong complex acid which is strong enough to be titrated directly with NaOH.
* Mannitol should be used in high conc. to prevent the hydrolysis of the complex.

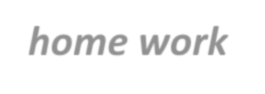
4H3BO3 + 4NaOH Mannitol 4NaBO2 + 8H2O

sod. metaborate

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* **Chemical factor: *home work***



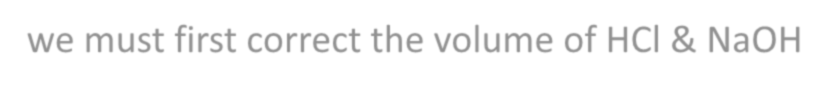
###### For borax with 0.5*N* HCl (0.09535 gm)

* For borax with 1*N* NaOH (0.09535 gm)

###### For sod. carbonate with 0.5*N* HCl (0.0265gm)

* For boric acid with 1*N* NaOH (0.06184gm)

###### Mwt. Boric acid=61.84 gm/mol Mwt. Na2CO3= 106gm/mol

* Calculations:

we must first correct the volume of HCl & NaOH

1. If we have **pure borax**, the vol. of *N*/2 HCl must be equal to the vol. of 1*N* NaOH.

V’of *N*/2 HCl = V’of 1*N* NaOH (the differences up to 0.3 mL) take the average of the two volumes & calculate the % w/v of Borax.

1. **Impure borax** may contain Na2CO3 or boric acid as impurities.
2. Mixture of **Borax & Na2CO3**

Na2CO3 is basic; it can react with HCl, so both borax and carbonate will react with HCl Na2CO3 + 2HCl 2NaCl + H2O + CO2

###### While NaOH react only with boric acid liberated

from borax. Then after vol. correction;

V1 of *N*/2 HCl > V2 of 1*N* NaOH

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liberated boric acid

H3BO3 + NaOH Mannitol NaBO2 + 2H2O

Liberated & free boric acid

So both the liberated and free boric acid react with NaOH,then

###### Correct the volumes and compare:

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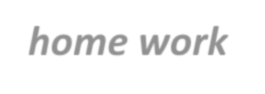
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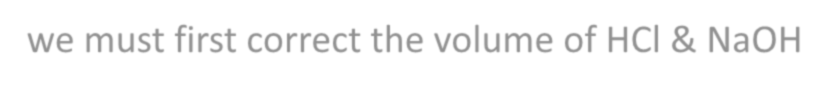
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## Assay of Chlorinated lime

Or bleaching powder Or Ca. Chlorohypochlorite

[Ca(OCl)Cl]

Assay of Chlorinated lime

[Ca(OCl)Cl]

### Contain not less than 30%w/w available chlorine.

* It is dull-white powder with characteristic odor, partially soluble in water and in 96% ethanol.

### Used as disinfectant.

**Procedure:**



The flask used in the titration is ***iodine flask***

≈0.1N **Na2S2O3**

10ml of unknown

1.5gm KI

5ml of 33%v/v HAC

***note:*** starch is added near the end point when

the color of the solution changed from brown to yellow.

\* **End point is colorless**

* Chemical principle:

Ca(OCl) Cl+2CH3COOH (CH3COO)2Ca+ HOCl+ HCl

hypoclorous acid

HOCl + HCl Cl2 +H2O

available chlorine

The *available chlorine* is the Cl2 which is liberated from the substance by the action of acid.

This liberated chlorine will displace an equivalent amount of I2 from KI.

Cl2+ 2KI 2KCl+ I2

The liberated I2 is titrated against sod. thiosulfate using starch mucilage as indicator.

I2 + 2Na2S2O3 2NaI + Na2S4O6

**Chemical factor**:

1ml of 0.1N Na2S2O3 ≡ 0.003545gm *available chlorine.*

Calculation:

Correct the sod. thiosulfate volume=V’

V’ X ch. factor= gm of *available chlorine* in 10ml. (Gm/10)X100= %w/v of *available chlorine.*

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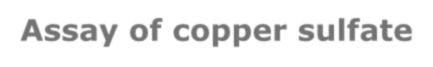
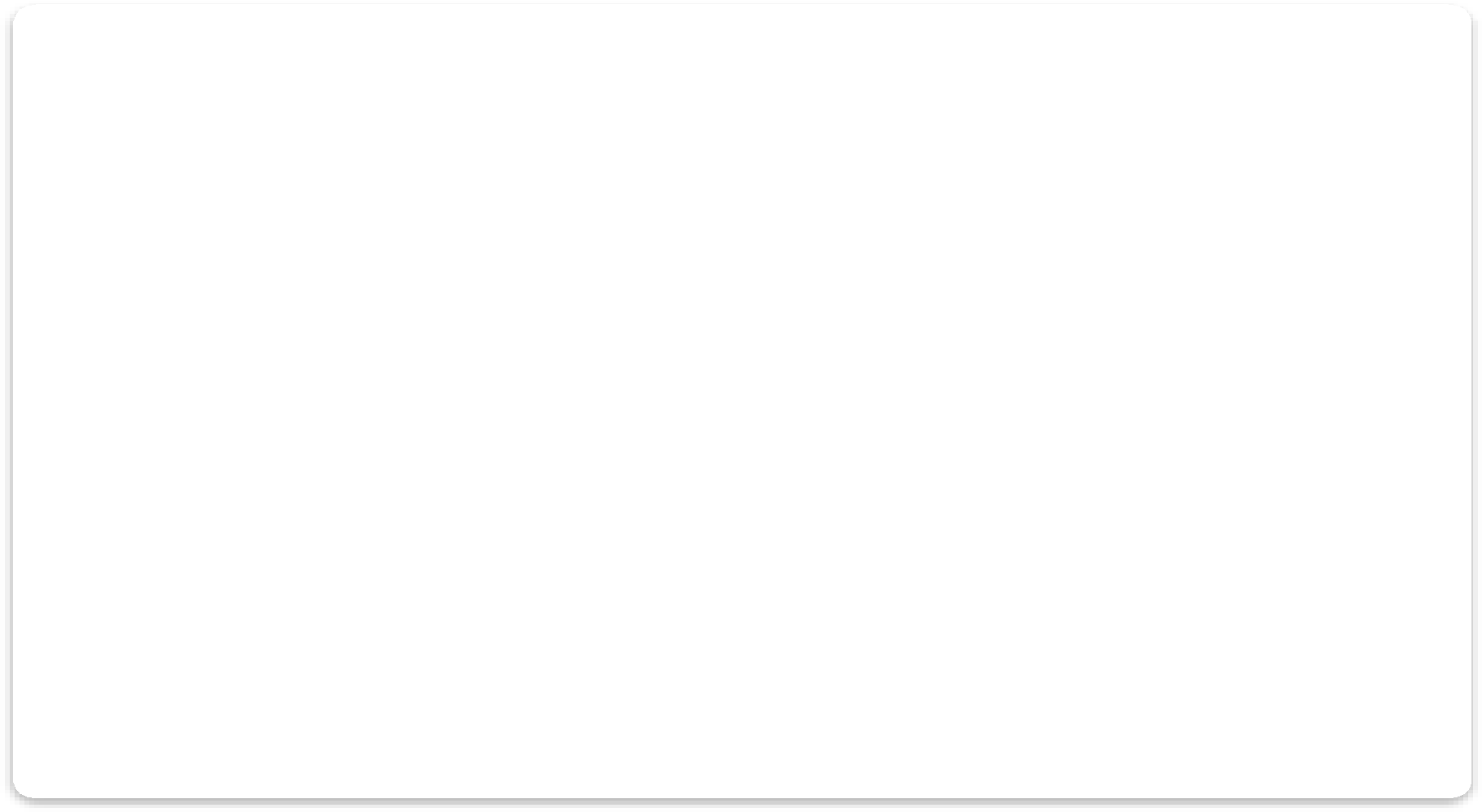
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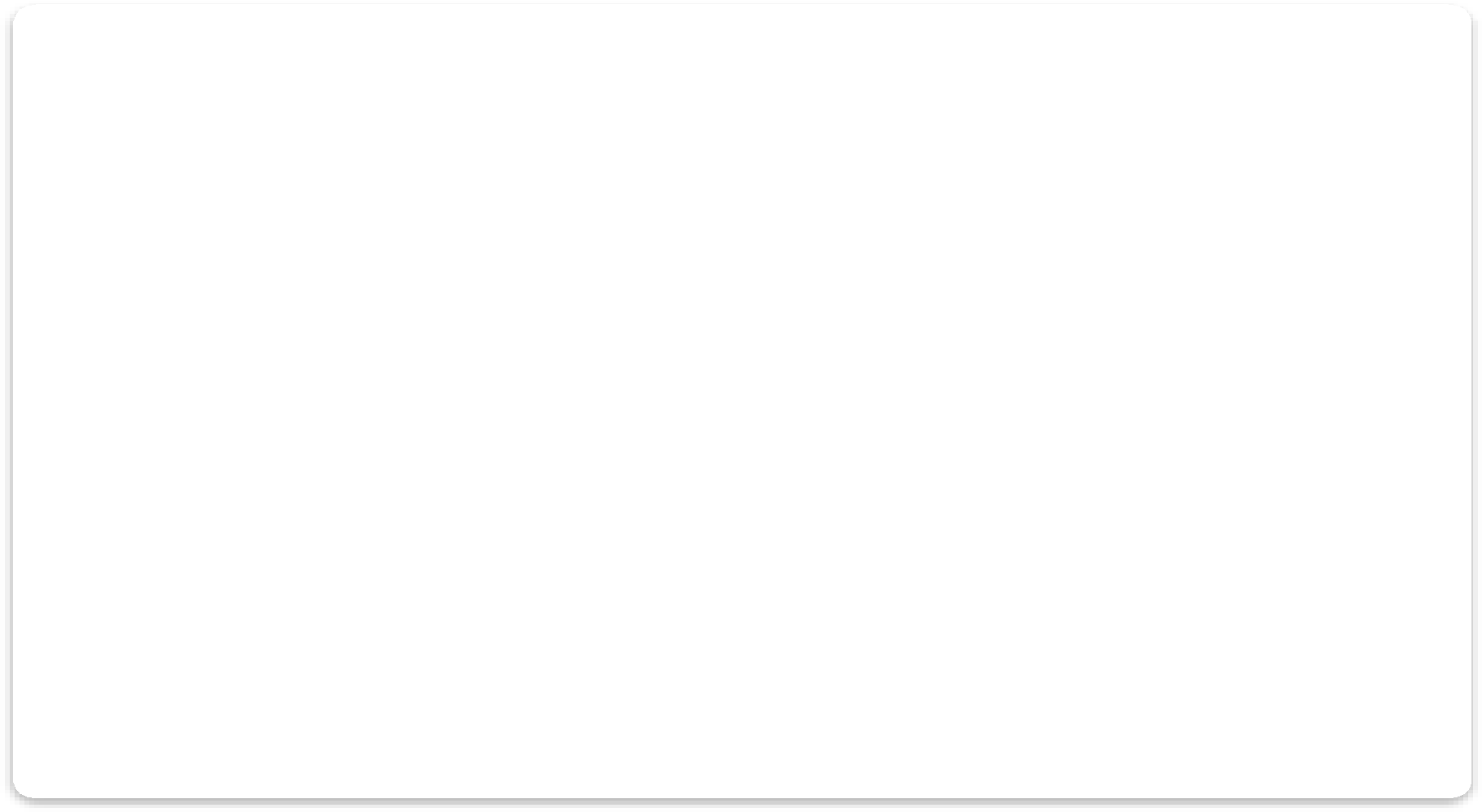


# Assay of copper sulfate

Cupric sulfate CuSO4.5H2O

***Mwt.=249.68 gm***

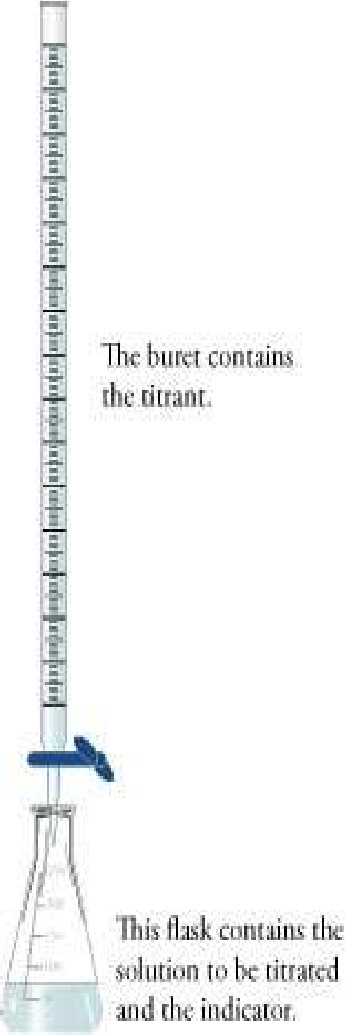
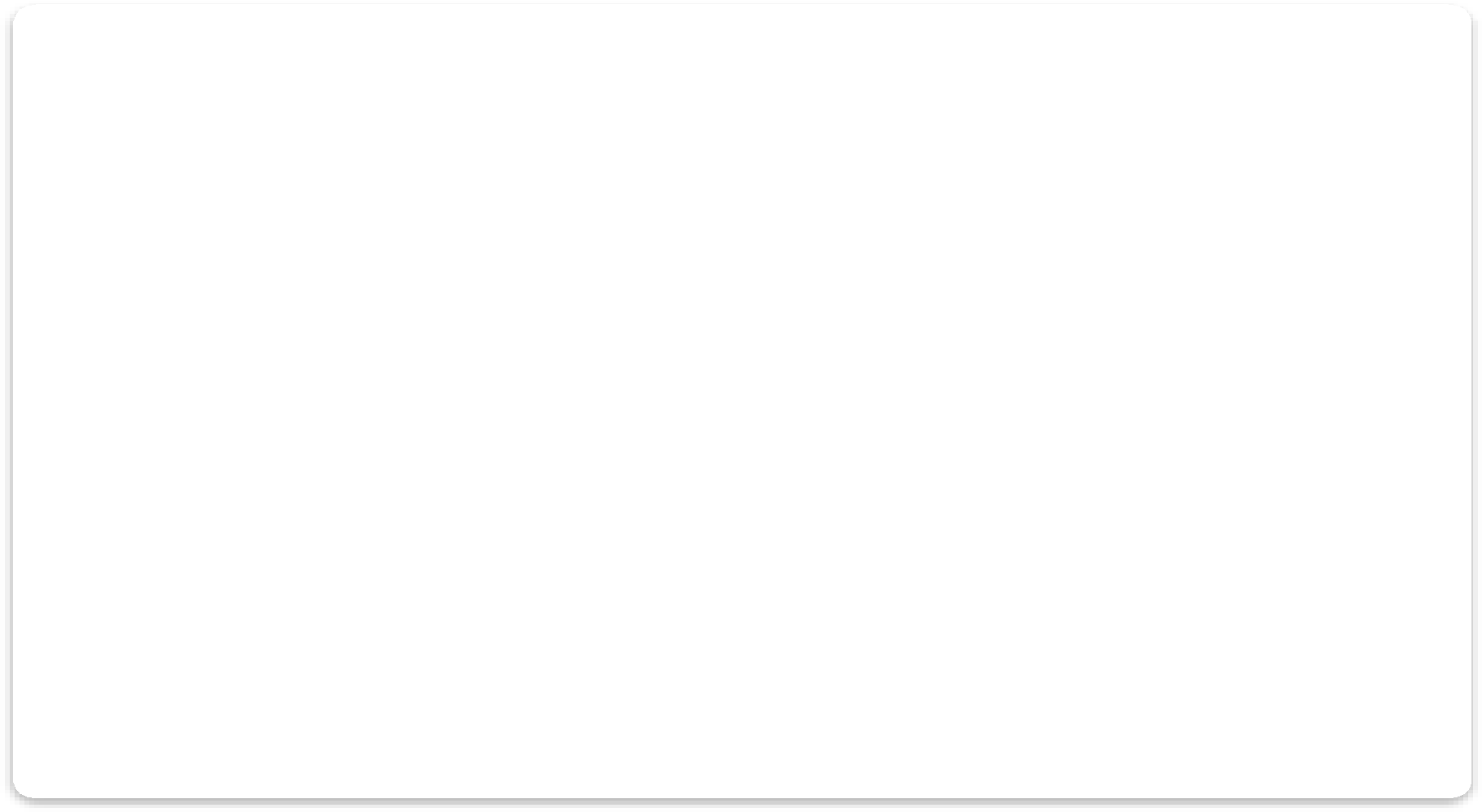
* + **Properties**: blue, fine prism or blue crystalline powder.



* + **Solubility**: soluble at 20˚c in 3parts of water and 3parts of glycerol, almost insoluble in 95% alcohol.

It contains not less than 98.5% and not more than 101%w/w CuSO4.5H2O.

* + **Uses**: as fungicide.
  + **Procedure:** the titration should be carried in stoppered flask.



During titration the color of the solution changed from ***brown*** to ***faint yellow*** then add1ml starch and 1gm KSCN and complete titration.

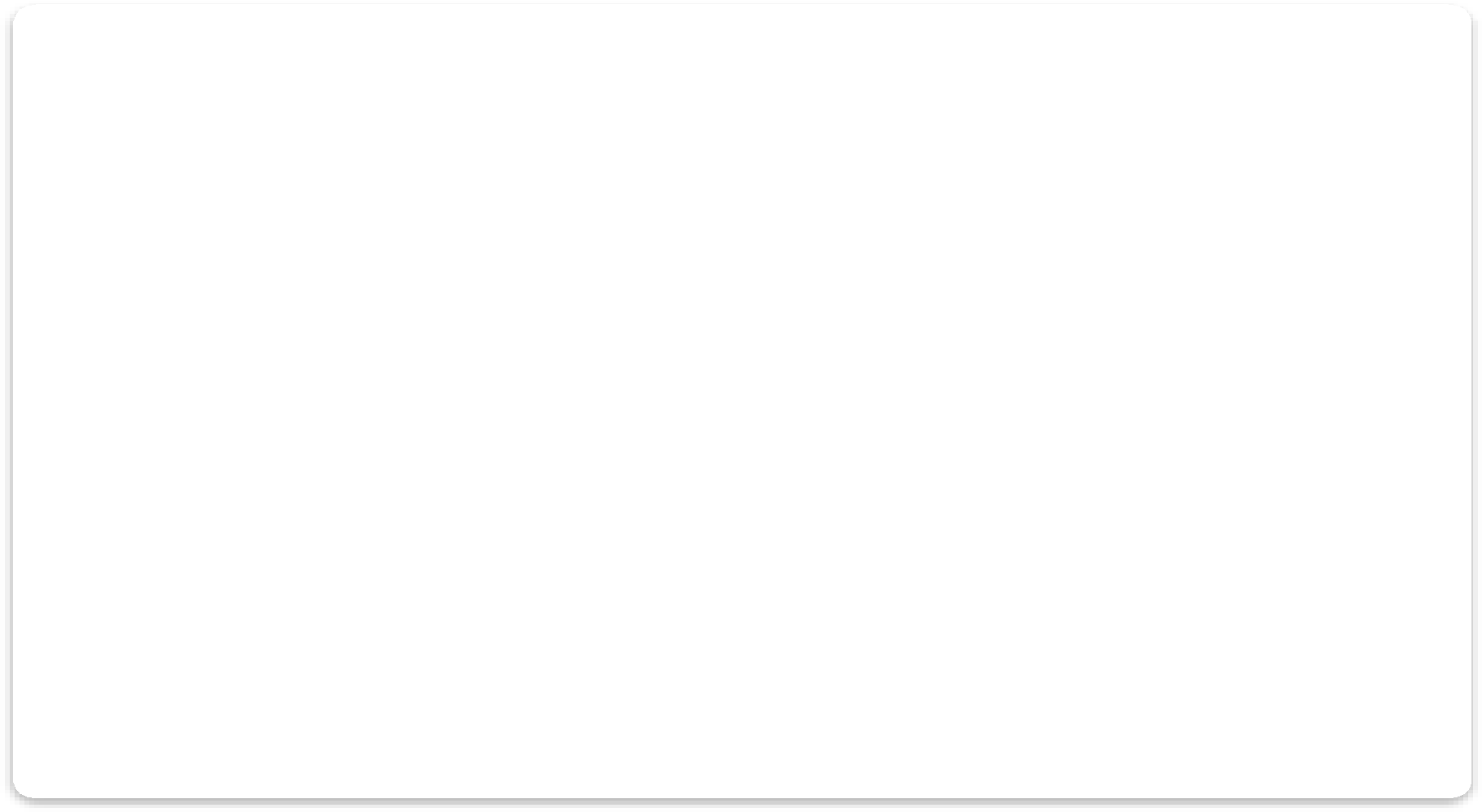
≈0.1N **Na2S2O3**

10ml unknown 30ml D.w.

1.5 gm KI

5ml acetic acid

* + **Chemical principle:**



* CuSO4 is treated with excess KI in acidic media.

oxidizing agent acidic media

2CuSO4+ 4KI 2CuI2+2K2SO4

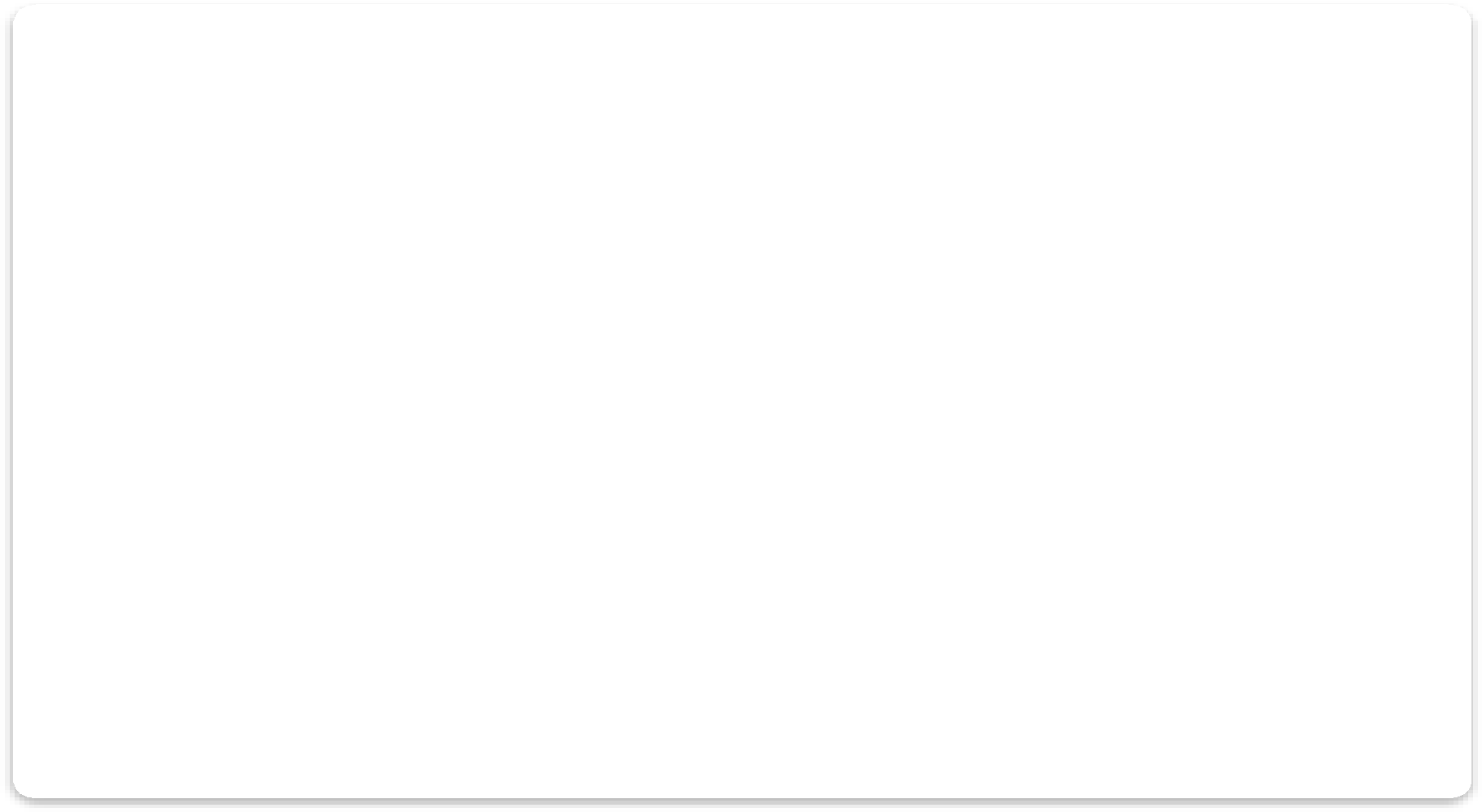
* The formed Cupric iodide(CuI2) is unstable.
* CuI2 will break into cuprous iodide(CuI or Cu2I2)

and I2.

2CuI2 2CuI(Cu2I2)↓ +I2

unstable yellow ppt.

* The liberated I2 is titrated with std. Na2S2O3 sol. I2 + 2Na2S2O3 2NaI + Na2S4O6
  + **Notes:**

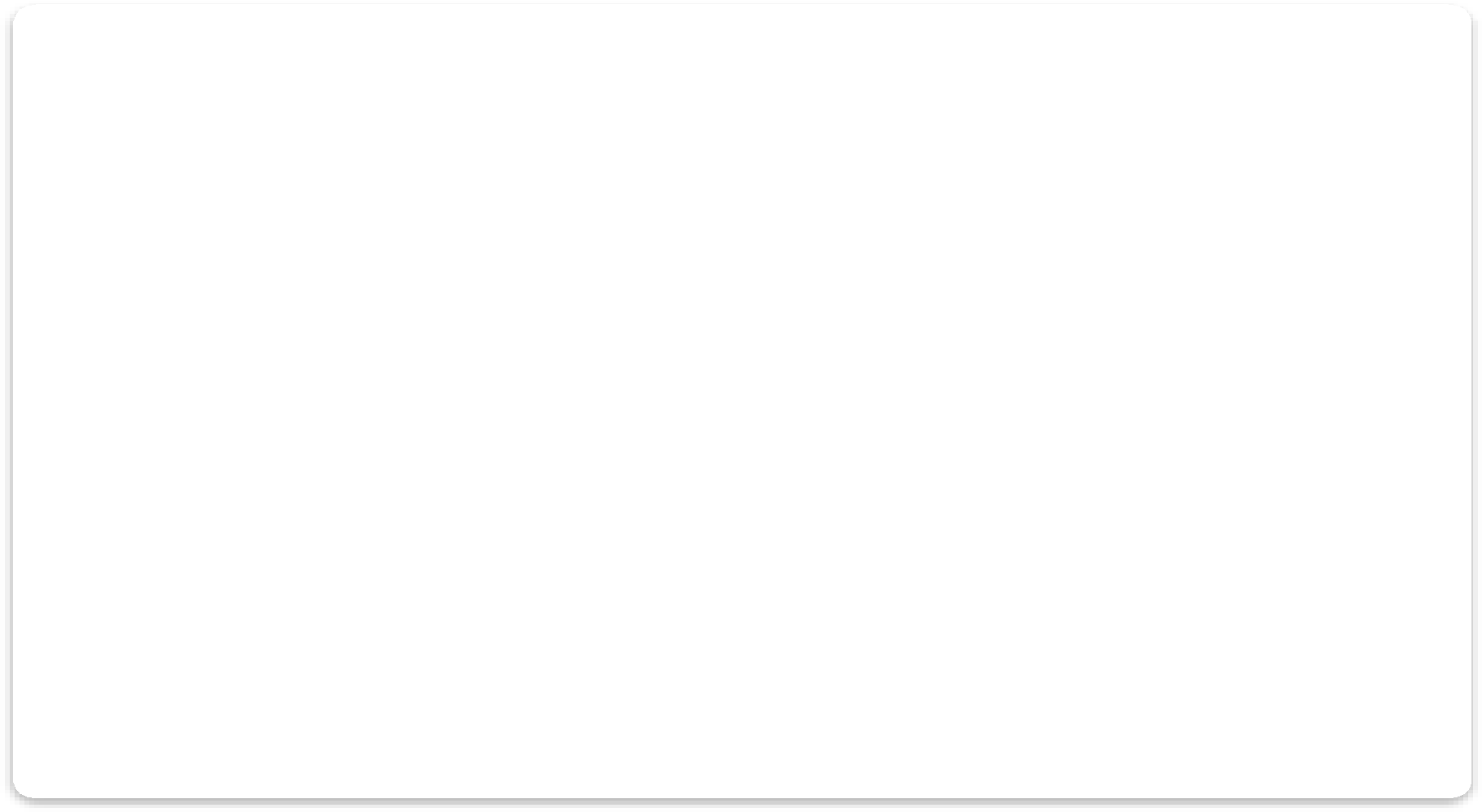


* 1. KI is added to liberate I2.
  2. Excess KI is added to dissolve I2.
  3. HAC is added to make the medium slightly acidic.
  4. In acidic media the liberated I2 is adsorbed by the ppted CuI.
  5. KSCN(pot. thiocyanate) is added near the end point.

KSCN+ CuI CuSCN+ KI

this KI will return to react with CuSO4 I2

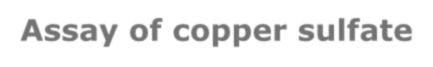
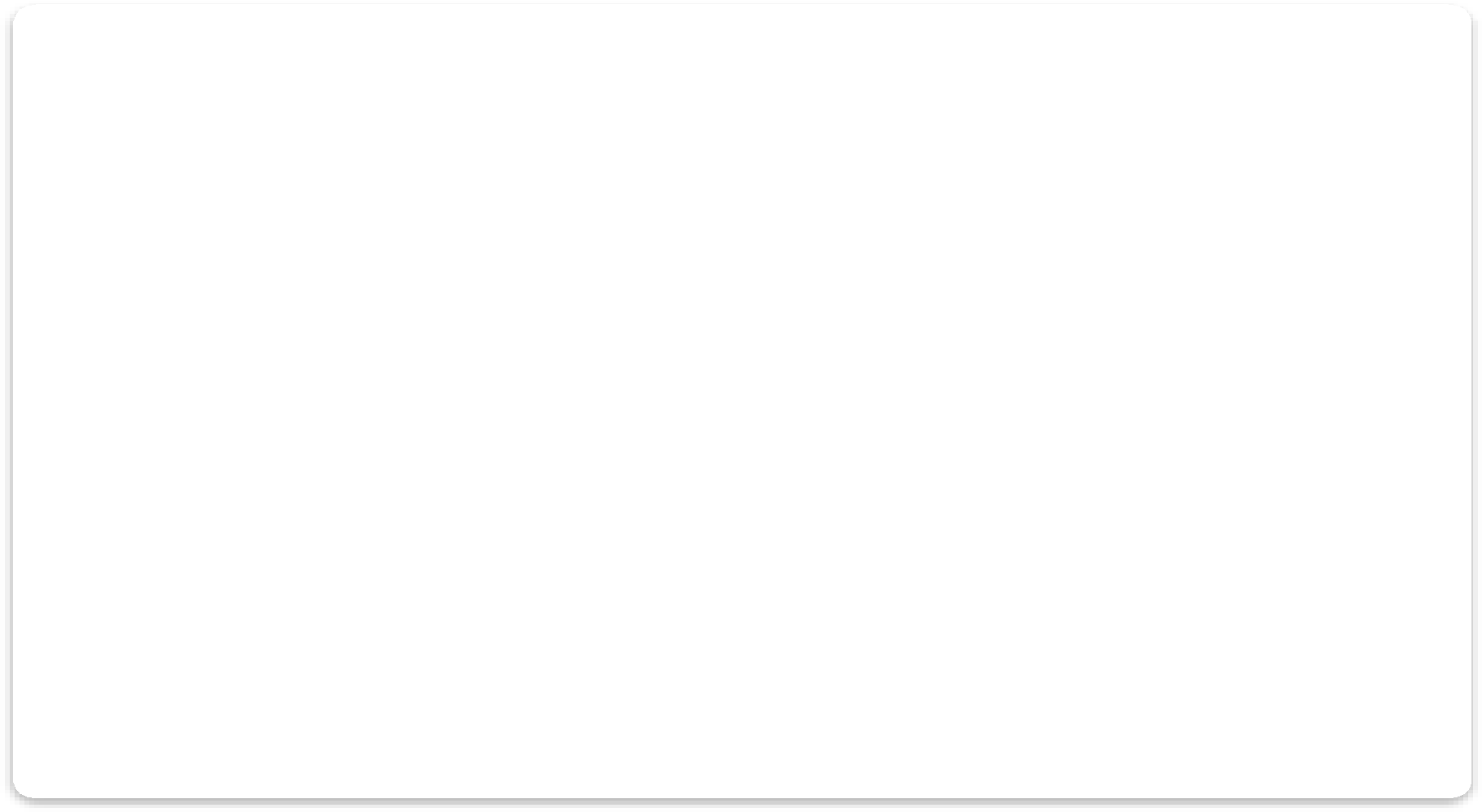
* + **Chemical factor:**



1ml of 0.1N Na2S2O3**≡** 0.02497gm CuSO4.5H2O

* + **Calculation:**
* correct the volume of Na2S2O3=V’
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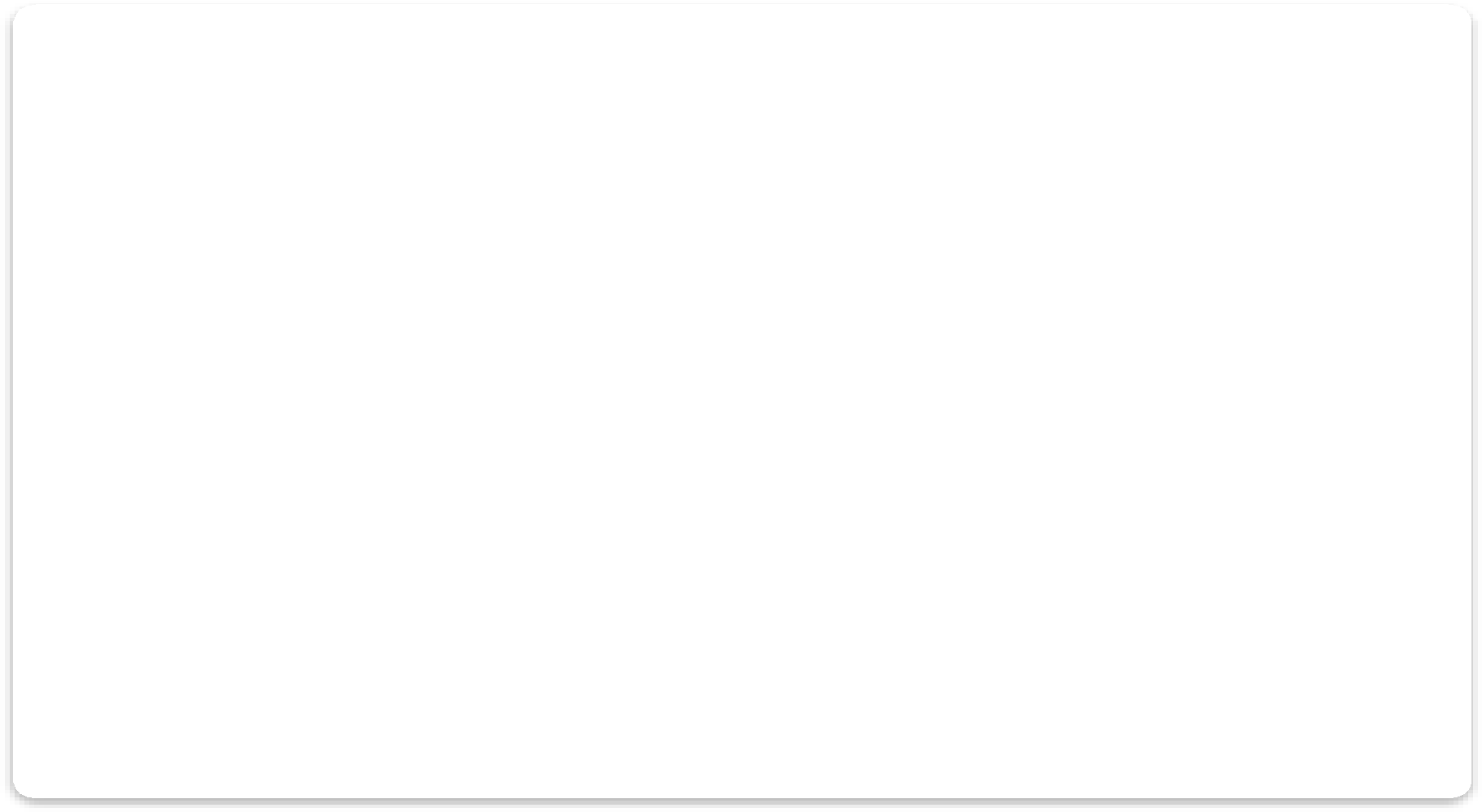


# Assay of copper sulfate

Cupric sulfate CuSO4.5H2O

***Mwt.=249.68 gm***

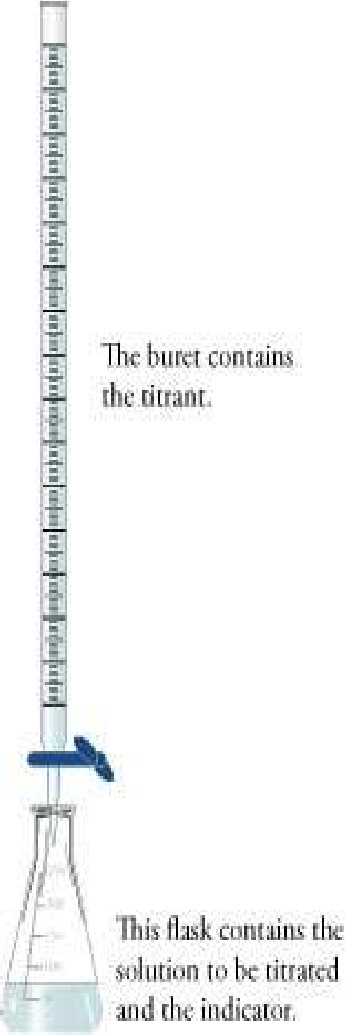
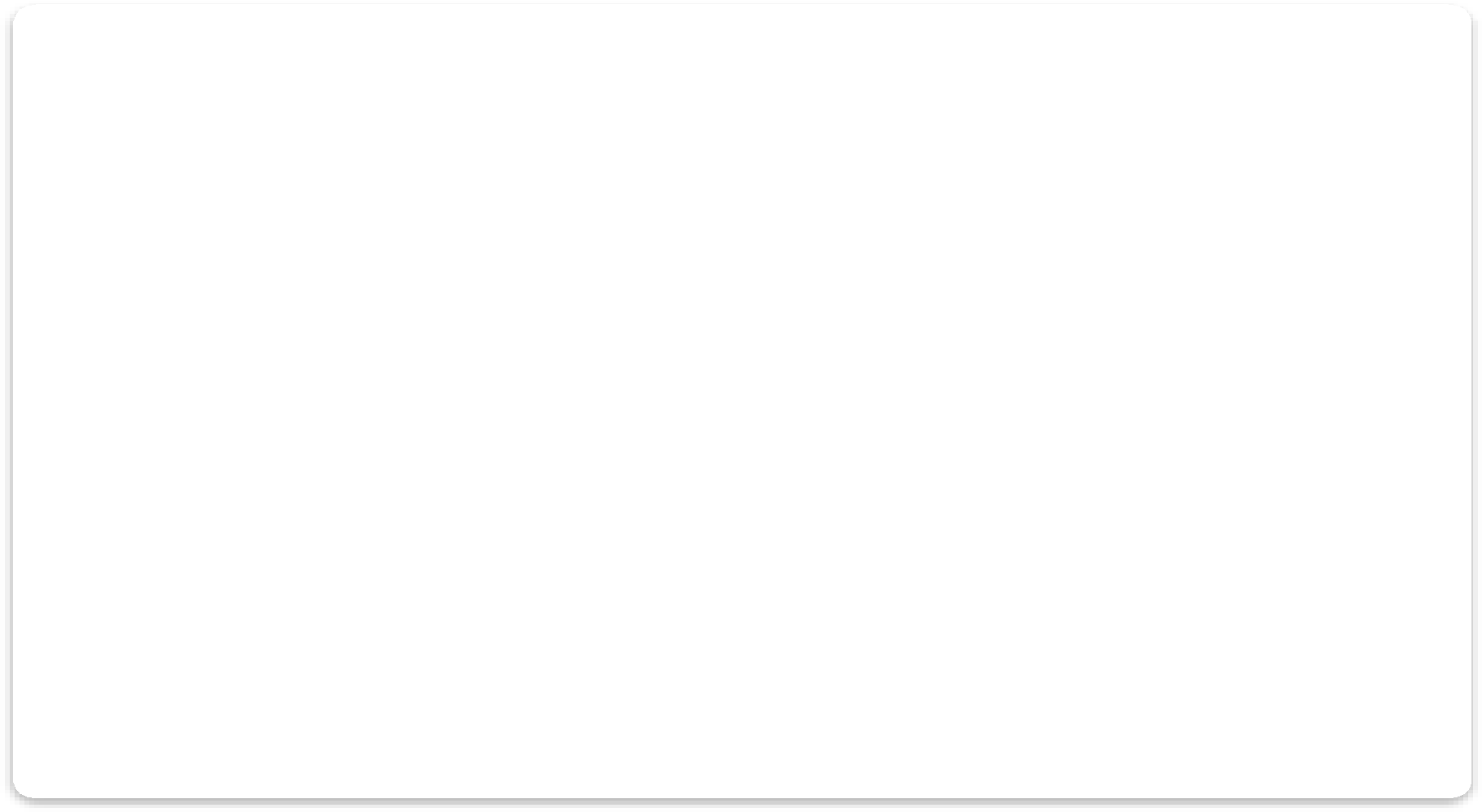
* + **Properties**: blue, fine prism or blue crystalline powder.



* + **Solubility**: soluble at 20˚c in 3parts of water and 3parts of glycerol, almost insoluble in 95% alcohol.

It contains not less than 98.5% and not more than 101%w/w CuSO4.5H2O.

* + **Uses**: as fungicide.
  + **Procedure:** the titration should be carried in stoppered flask.



During titration the color of the solution changed from ***brown*** to ***faint yellow*** then add1ml starch and 1gm KSCN and complete titration.

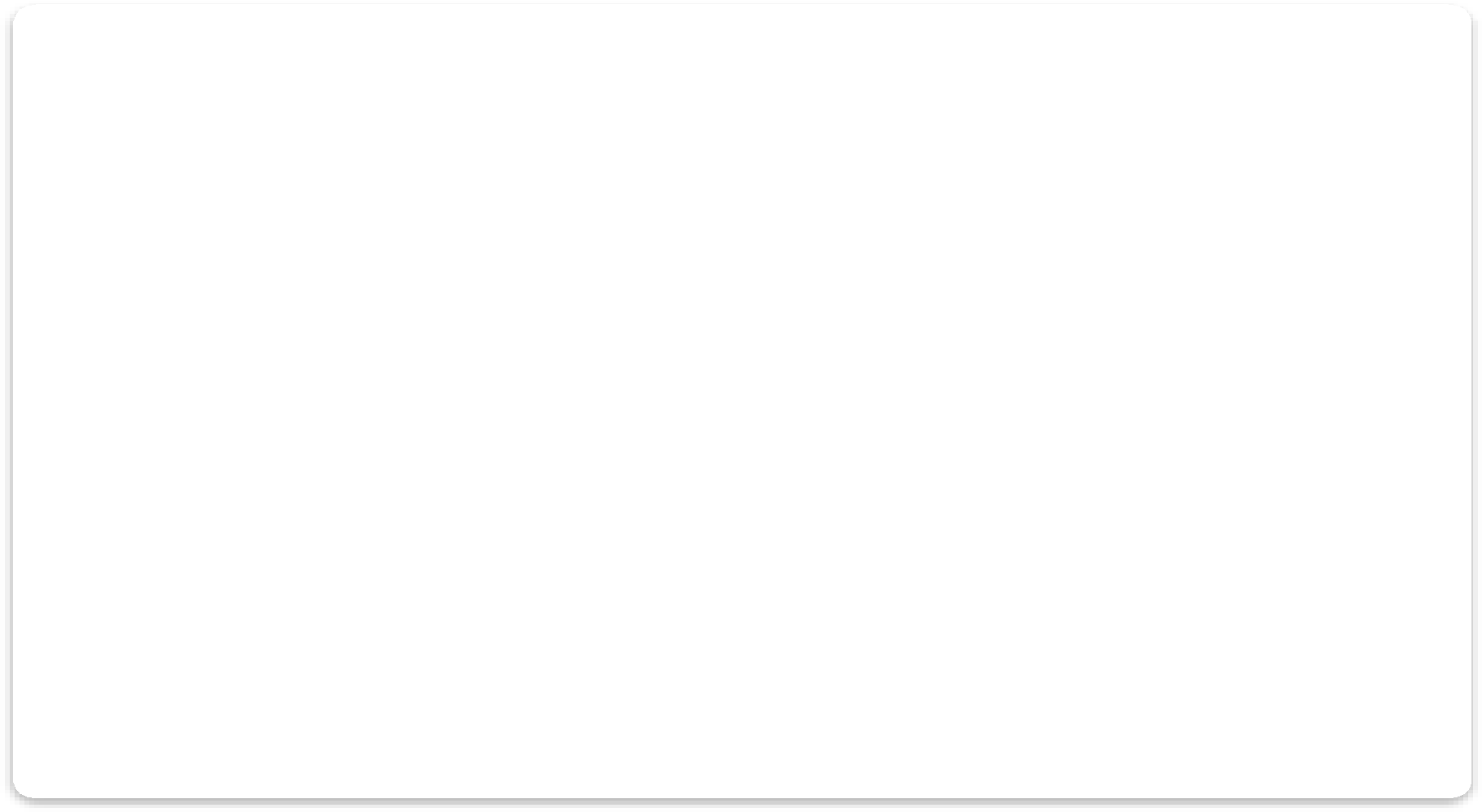
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10ml unknown 30ml D.w.

1.5 gm KI

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oxidizing agent acidic media

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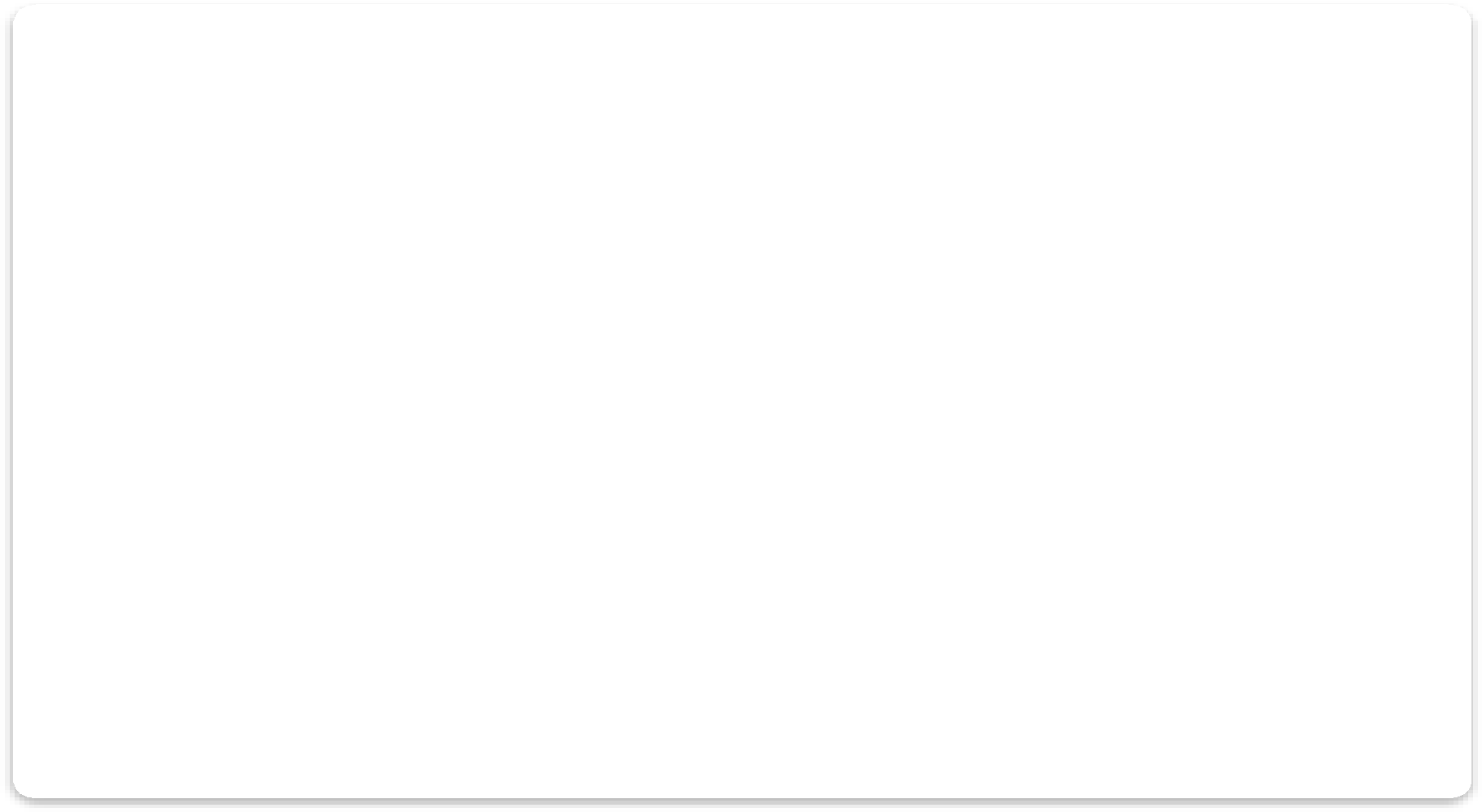
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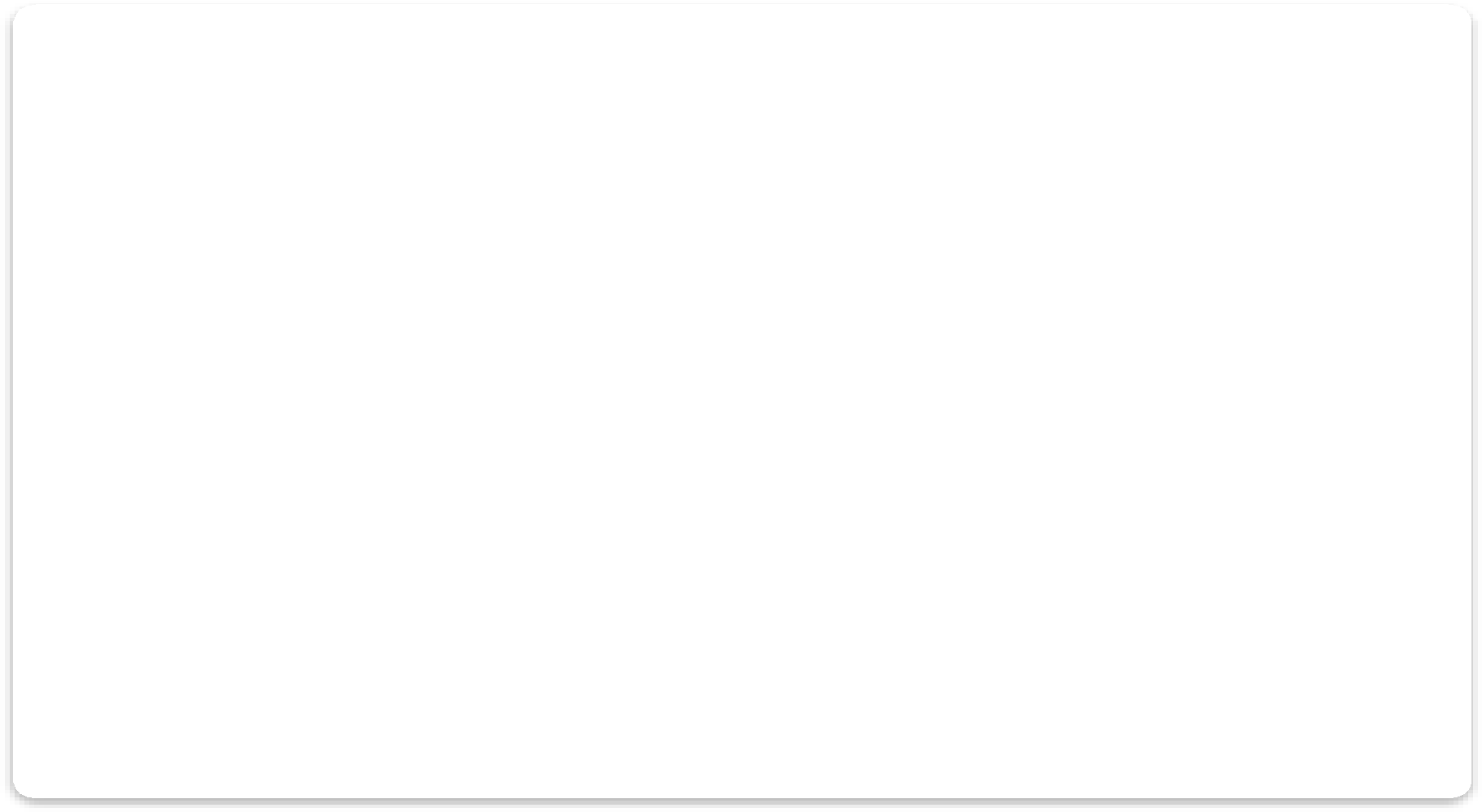


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KSCN+ CuI CuSCN+ KI

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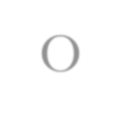
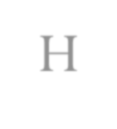
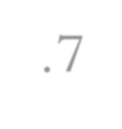
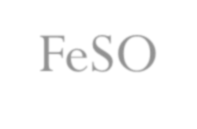
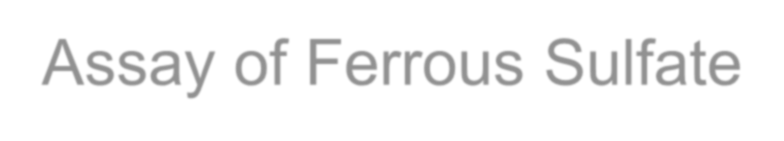
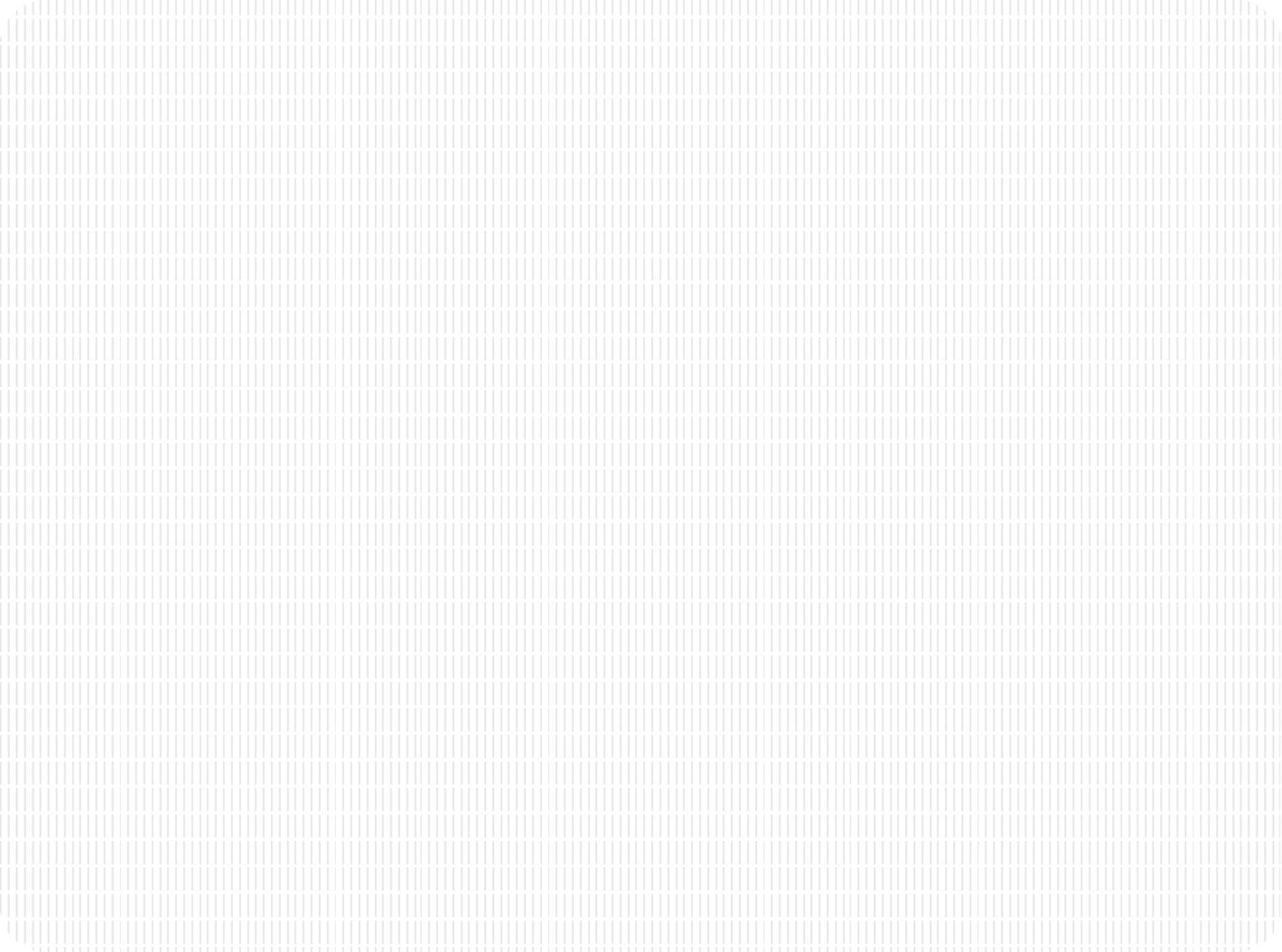
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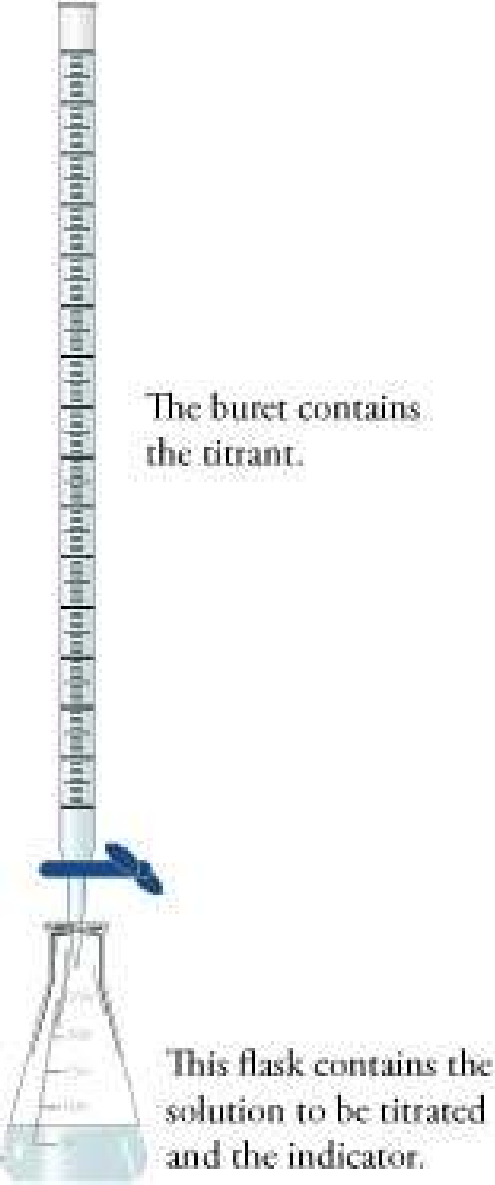


Assay of Ferrous Sulfate

FeSO4.7H2O

Mwt.=278gm/mole

* **Appearance**: The blue-green heptahydrate is the most common form of ferrous sulfate ,it is bluish green crystals, freely soluble in water.
* **Content**: Ferrous Sulfate (crystal) contains 98.0－ 104.0% of ferrous sulfate heptahydrate (FeSO4･ 7H2O)
* **Used** in the prevention and treatment of iron deficiency.
* **Procedure:**



*H2SO4 should be added to maintain a constant H ions concentration as explai*

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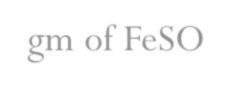
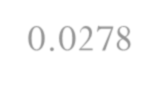
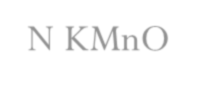
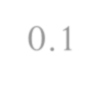
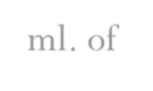
≈ 0.1N KMnO4

*Unknown powder*

*20ml of diluted H2SO4*

***End point: colorless pink***

* **Chemical principle:**



10FeSO4+2KMnO4+8H2SO4 2MnSO4+5Fe2(SO4)3+K2SO4+

8H2O

Or MnO4¯ +8 H++5Fe+2 Mn+2 +4H2O+5 Fe+3

* Ferrous sulfate is a ***reducing agent***.
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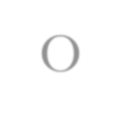
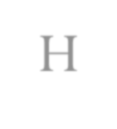
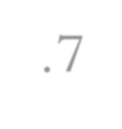
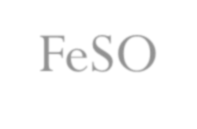
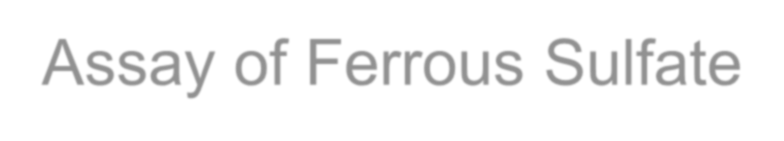
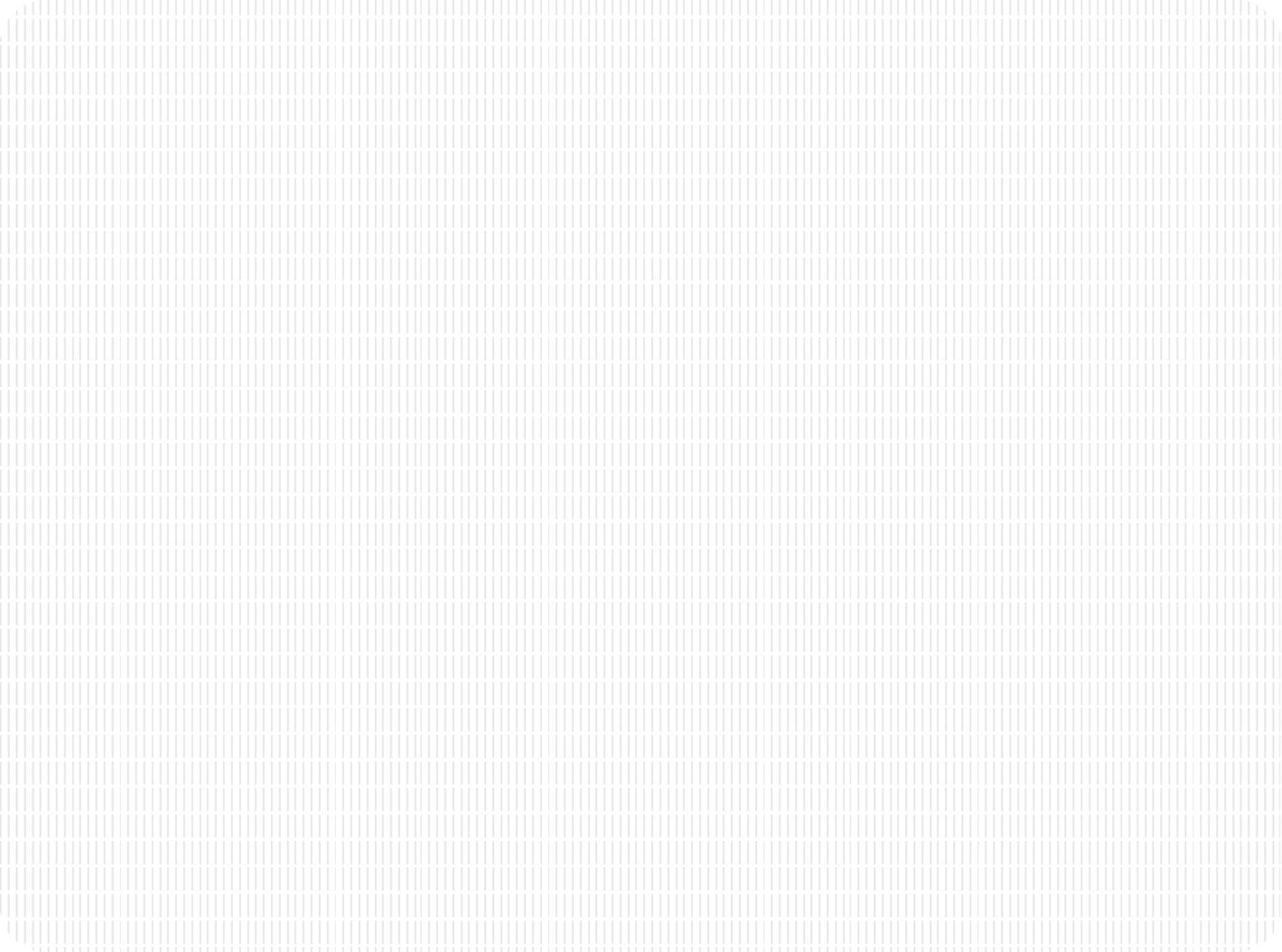
1ml. of 0.1N KMnO4 ≡ 0.0278gm of FeSO4

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Then multiply the corrected volume (V') by the Ch. Factor. to get the weight;

V' x ch. Factor =gm of FeSO4 in the unknown.

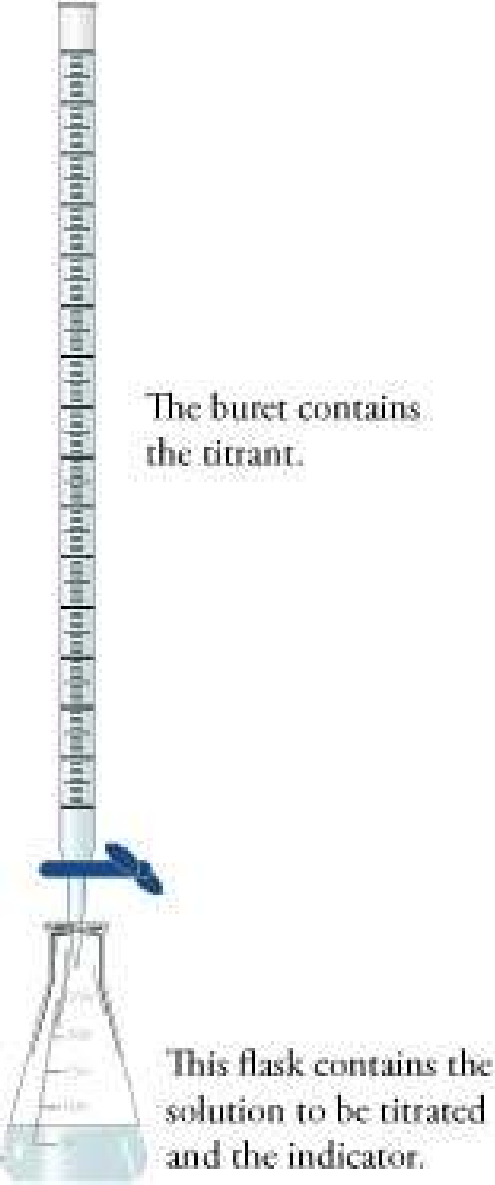


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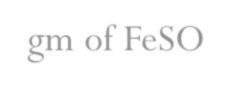
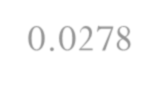
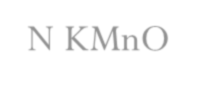
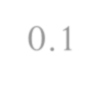
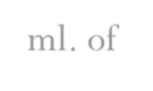
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Derive the ch. Factor from the previous chemical equation.

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Correct the volume of KMnO4 consumed=V'

Then multiply the corrected volume (V') by the Ch. Factor. to get the weight;

V' x ch. Factor =gm of FeSO4 in the unknown.



H2O2

*H2O2: Mwt.=34.02 gm/mole*

*Different H2O2 solution: 30%,27%w/w and 6%, 3%w/v*

###### H2O2 is slightly more viscous than water.

* Aqueous solution of H2O2 colorless and odorless. Rapidly decomposes when contact with organic matter and certain metals; Cu, Fe, Mn and if allowed to stay in alkaline solution. 2H2O2 → O2 + H2O
  + In acidic solution Fe2+ is oxidized to Fe3+ (H2O2 acting as an oxidizing agent):

2 Fe2+ + H2O2 + 2H + → 2 Fe3++ 2H2O

* + hydrogen peroxide acts as a reducing agent in its reaction with KMnO4 *in our experiment*.

2MnO4- + 5H2O2 + 6H+ → 2Mn2+ + 5O2 + 8H2O

* + **Uses:**

It is a ***disinfectant*** or ***antiseptic*** for treating wounds for its antimicrobial activity.

it is an effective ***cleaning agent*** prevents infection transmission in the hospital environment.

Procedure:

≈ 0.1N KMnO4

*10ml of unknown 25ml of D.W.*

*5ml of 50%v/v H2SO4*

***End point****: colorless pink*

* + **Notes:**

1. We make the media acidic **to;**
   * Prevent decomposition of H2O2
   * Prevent reaction of KMnO4 MnO2
   * Increase oxidizing power of KMnO4
2. Titration should be done slowly

#### Chemical principle:

2KMnO4+5H2 O2+3H2SO4 K2SO4 +2MnSO4+5O2+8H2O

**\**You must write the oxidation and reduction equations to calculate the eqwt. for both KMnO4 & H2O2*** *.*

#### Chemical factor:

According to the chemical equation; 2Mwt of KMnO4 ≡ 5Mwt of H2O2

*then complete the derivation of the Ch. Factor.*

The Chemical factor is equal to:

1ml of 0.1N KMnO4 ≡ 0.001701gm of H2O2

* + Calculation:
  + Correct the **volume** of KMnO4 NxV = *N’xV’* (corrected)
  + Multiply the corrected volume by the Ch. Factor. to get the **weight (wt.)** of H2O2
  + the **%w/v** of H2O2 = **wt**. /10 x 100
  + There is methods to calculate the concentration of hydrogen peroxide in commercial products:



#### 1. %W/V:

10ml of concentrated sol. was diluted to 200ml with

D.W. Then 20ml is taken from the dil. one and titrated with approximately 0.1N KMnO4

conc. diluted

10 200 x= 20X10**/**200=1ml of conc. sol. x 20 (original sol.)

Correct the volume of KMnO4= ***Vol. Vol.*** x ch. Factor =wt. of H2O2 in 1ml. ***Then calculate %w/v***



Assay of sodium hydroxide solution

NaOH solution

* + From B.p, NaOH solution contains not less than 97.5% w/w of total alkali (as NaOH) and not more than 2.5 % w/w Na2CO3.

#### Assay:

* 10 ml of unknown(bulb pipette).
* 25 ml of distilled water.
* Add 2.5 ml of barium chloride solution.
* Titrate with 1N HCl solution using 1-2 drops of phenolphthalein ind..
* The first end point from pink colorless(turbid)
* To the turbid sol. add 5 drops of Bromophenol Blue ind. and complete titration with 1N HCl .
* The second end point bluish violet yellowish green

1N HCl solution



10 ml of unknown 25ml of D.W

2.5 ml of BaCl2 solution

1-2 drops of ph.ph. ind. (1st titration)

5-6 drops of B.p.B ind. (2nd titration)

**Chemical principle:**

* + NaOH is strong base, absorbs CO2

2NaOH + CO2 Na2CO3 + H2O

* + both NaOH and Na2CO3 react with HCl NaOH + HCl NaCl + H2O

Na2CO3 + 2HCl 2NaCl + H2O + CO2

When we assay a sample, we do the assay for total alkalinity contributed to NaOH and Na2CO3.

* + Barium chloride (BaCl2) is added to precipitate all carbonate

BaCl2 + Na2CO3 BaCO3 + 2NaCl

soluble in alkaline insoluble in alkaline

* + ***1st titration***:

###### NaOH + HCl NaCl + H2O

Why HCl do not react with BaCO3? Why the end point is turbid?

##### 2nd titration:

###### 2HCl + BaCO3 BaCl2 + H2O + CO2

***definition of chemical factor***: the weight of substance that is chemically equivalent to 1ml of std. solution.

* + **Calculation of the *chemical factor*:**

###### From reaction of HCl with NaOH: 1Mwt of NaOH ≡ 1 Mwt HCl

1 Mwt of NaOH ≡ 1 eqwt HCL

###### 1 40 gm of NaOH ≡ 1 liter of 1*N* HCl 40/1000 gm NaOH ≡1ml of 1*N* HCl

0.04 gm of NaOH ≡ 1ml of 1*N* HCl of total alkalinity calculated as NaOH(chemical factor)

###### From reaction of 2HCl with Na2CO3

2Mwt of HCl ≡1Mwt of BaCO3 ≡1Mwt Na2CO3 1Mwt Na2CO3 ≡ 2Mwt of HCl

###### 1Mwt Na2CO3 ≡ 2 eqwt of HCl

½ Mwt Na2CO3 ≡ 1 eqwt of HCl 106/2 gm Na2CO3≡ 1liter of 1*N* HCL 53 gm Na2CO3 ≡ 1liter of 1*N* HCL 53/1000 gm Na2CO3 ≡ 1ml of 1*N* HCL

0.053 gm Na2CO3 ≡ 1ml of 1*N* HCL (chemical factor)

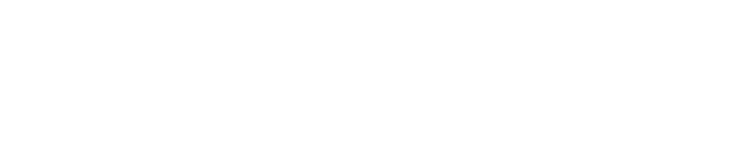
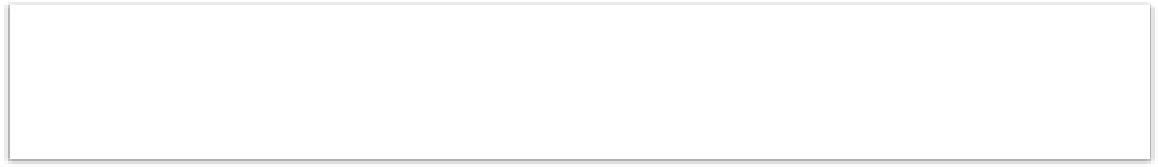
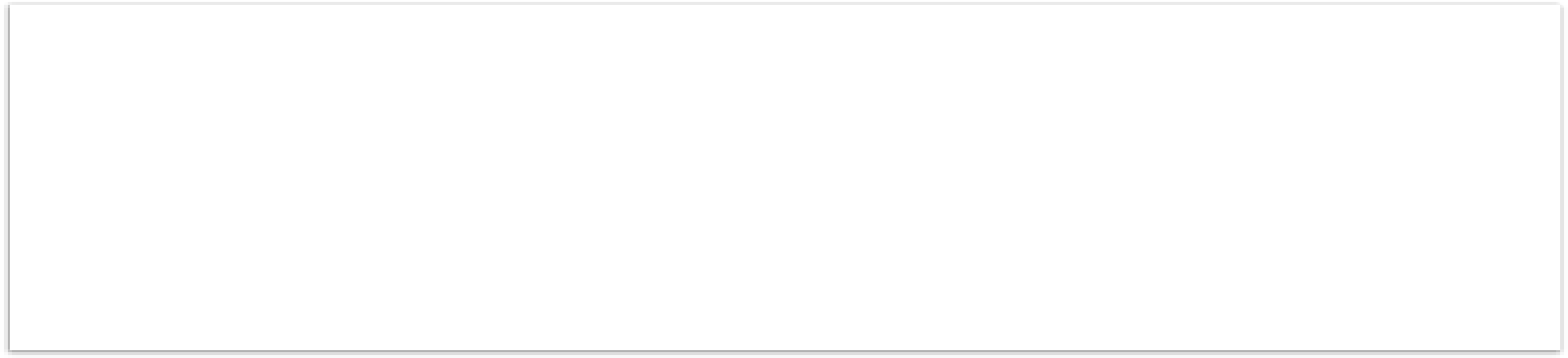
#### Calculations :

###### V1 is the of HCl consumed in the 1st titration V2 is the of HCl consumed in the 2nd titration V1+V2= V3 total HCl consumed.

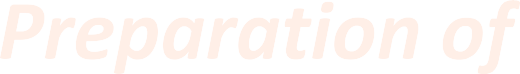
correct the V3 according to this equation: V x N= V’ x N’

###### Corrected V3 x 0.04= gm wt. of total alkali Then the % w/v of total alkali

Corrected V2 x 0.053= gm wt. of Na2CO3 Then the % w/v of Na2CO3 in the unknown



Redox Reaction



###### Dissolve 3.3 gm of solid KMnO4 in 1000ml. distilled water, in a flask.

* Heat the solution on a hotplate for 15min. at 70-80 ◦c.

###### Cool the solution to room temp. and allow stand for 2 days in a dark place.

* Filter the solution through the glass wool or asbestos before use.



* ***Filter the prepared soln. through glass wool as said before.***
* ***Rinse and Fill the burette with filtered KMnO4 soln.***
* ***Weigh accurately about 200mg sod. oxalate (Na2C2O4), previously dried at 110 ◦c to constant weight.***
* ***Dissolve it with 100ml of D.W. in a conical flask. Add 7ml. of conc. H2SO4.***
* ***Heat the soln. to about 70◦c.***
* ***Titrate against KMnO4 soln. until a permanent pale pink color appear (persist for 15 sec.).***
* ***Record the volume of KMnO4 consumed.***





###### Methods of analysis involve a change in valence of the reacting substances, thus there is always a transfer of electrons.

* Electrons lost by reducing agent are gained by oxidizing agent. The oxidizing agent will be reduced; gain of electrons and then decreasing in oxidation number, but the reducing agent will be oxidized; losing of electrons and then increasing in oxidation number.



###### We can calculate the eqwt. of the reactants:

Mwt.

###### Eq. wt =



no. of e- gained or lost by each molecule

*  ***Permanganate* ion** (MnO4‾ ) acts as an oxidizing agent in acidic media, being reduced to Mn⁺⁺:







* Oxalate ion (C2O4⁼)acts as reducing agent: C2O4⁼ 2CO2 + 2e‾



###### KMnO4 is widely used in volumetric analysis as a standard and oxidizing agent because:

1. It is strong oxidizing agent.

###### The intense color of KMnO4 solution is sufficient to signal the end point in most titrations thus it is used as self indicator.

* + On the other hand,

###### KMnO4 is a strong oxidizing agent, then it will oxidize any organic matter present in D.W., so heating for 15min. is to accelerate the oxidation of these organic matter.

1. KMnO4 soln. is allowed to stand for 2days to ensure

the completion of the decomposition reaction.

1. It is filtered through asbestos to remove all traces of manganese dioxide (MnO2) .
2. KMnO4 is unstable in the presence of direct sunlight, organic matter ex. Filter paper.
3. H2SO4 is added since KMnO4 is better oxidizing agent in acidic media, H2SO4 is added to keep [H⁺]ion conc. constant through out the titration.
4. Heat to about 70 ***◦***c because the oxidation of sod. oxalate is rapid enough if the temp. above 60 ◦c .
   * Sod. oxalate, not oxalic acid, is used because:
5. The salt can be obtained in very pure condition.
6. Free from water of crystallization.
7. It can be dried to 130 ◦c without decomposition.
8. Stable to air.

The chemical equation for the std. of pot. Permanganate:

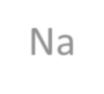
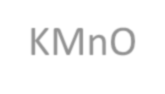
5Na2C2O4+ 2KMnO4+ 8H2SO4 2MnSO4+ 5Na2SO4



+ K2SO4 + 8H2O+ 10CO2

Calculations

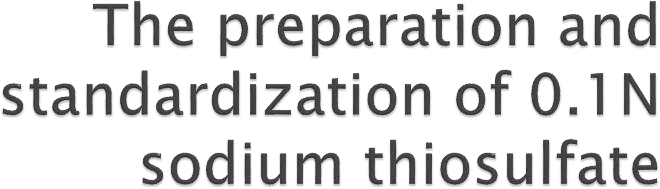
KMnO4 Na2C2O4



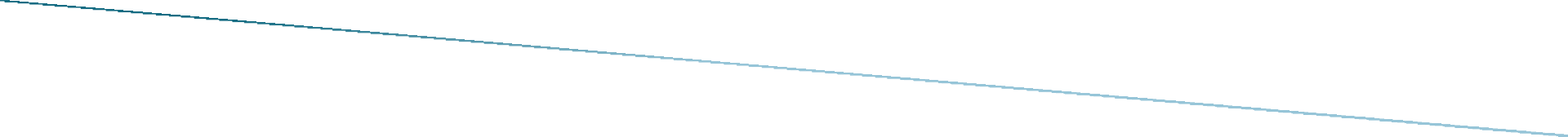
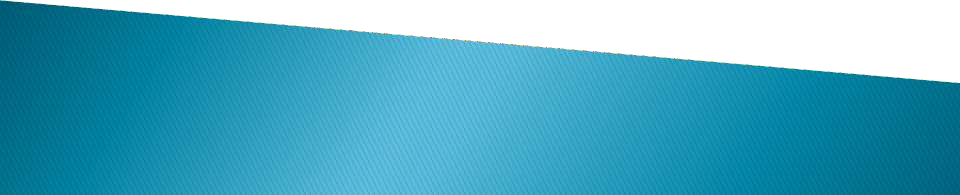
###### N X V = WT X 1000

eqwt

###### 67gm/mole



Na2S2O3.5H2O



**Preparation:**

Dissolve about 26gm sod. thiosulfate and 200mg sod. carbonate in 1000ml of recently boiled and cooled D.W.

**Standardization:**

Put into the stoppered flask the followings;

0.1-0.2 gm of potassium dichromate (K2Cr2O7) 100ml of D.W.

3gm potassium iodide (KI)

2gm sod. Bicarbonate( NaHCO3 ) 5ml conc. HCl

* Close the flask with the stopper and swirl to mix.
* Then keep the flask in dark place for 10min.



* Rinse the stopper and inner walls of

the flask with D.W.

* Titrate the liberated I2 with sod. thiosulfate the color change from brown to faint yellow.
* Add 1ml of starch indicator the color of

the solution change to dark blue.

* Complete titration until the color change to green-blue.

≈ 0.1N Na2S2O3

**End point: dark blue green-blue**



**Notes for preparation**:

* *Why do we use recently boiled and cooled water?*
  1. to destroy sulfur bacteria
  2. to expel CO2

Na2S2O3 +2H2CO3 2NaHCO3+H2S2O3

H2S2O3 H2SO3 + S⇓

thiosulfuric acid sulfar

SO2 +H2O

* *Na2CO3 is added to the solution as preservative*



**Chemical principle of standardization:**



* Pot. Dichromate is an oxidizing agent

K2Cr2O7 +6KI +14HCl 3I2+2CrCl3+8KCl+7H2O

* The liberated I2 is titrated against Na2S2O3

2Na2S2O3+ I2 Na2S4O6 +2NaI

sod. tetrathionate

* This indirect procedure is known as ***iodometry***.

**Notes for standardization**:

1. We use stoppered flask to prevent the loss of I2 .
2. KI is added to liberate I2.
3. Excess KI is added, Why?

I¯+ I2 I3¯ tri-iodide ion ***(soluble form of iodine)***

1. The standardization should be carried in acidic media and not in alkaline media:

because alkaline media I2 will react with OH¯ to give:

I2 + OH¯ HI+ IO¯ Hypoiodite ion 3 IO¯ IO3¯ +2I¯



These ions will oxidize the thiosulfate to:

IO¯ +IO3¯ +S2O3 SO4

thiosulfate sulfate

1. On the other hand in higher acidic media the excess KI will react with HCl to give HI, HI will be oxidized by atmospheric O2 to I2:

KI + HCl HI +KCl

excess atmospheric O2

HI I2

Sod. bicarbonate is added to react with formed HI:

NaHCO3+ HI NaI+ H2O+ CO2 ⇗



1. We put the flask in dark place for 10min.?
2. Starch ind. is added at the end (when the color of the solution change to yellowish green) or we can said the ind. is added when the iodine concentration is low because β-amylase which the soluble form of starch react :

β-amylase+I2+I¯ blue colour complex

the color change is reversible, the color being discharged (change to colorless) when iodine is reduced by Na2S2O3.

This reversibility is reduced when the iodine conc. is high for this reason starch ind. should be added until most of I2 has been reduced by Na2S2O3 .



1. The end point is a pale green and not colorless why?

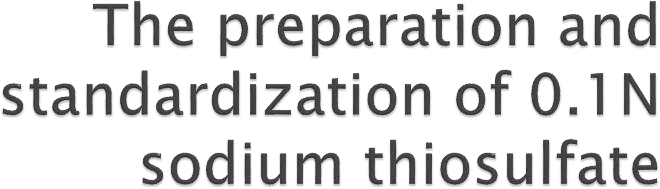


**Calculation:**

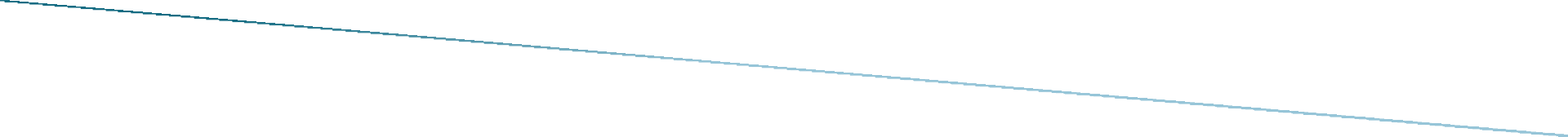
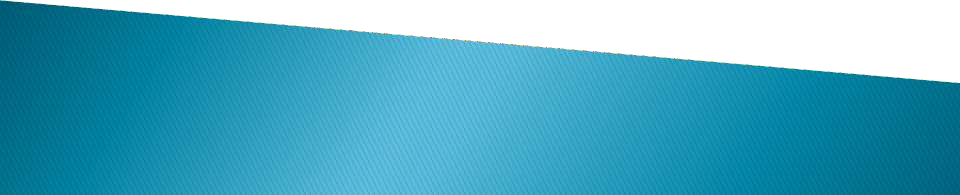
Na2S2O3 K2Cr2O7

N x V = (Wt/eqwt) x1000

eqwt= 49



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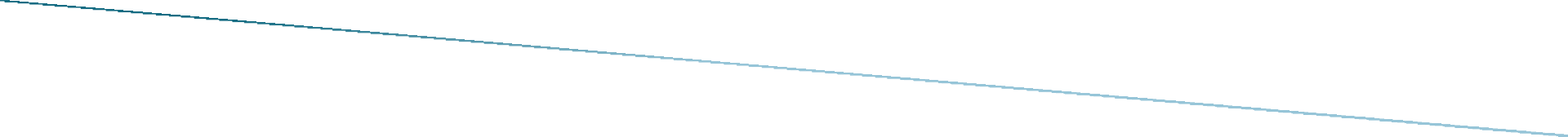
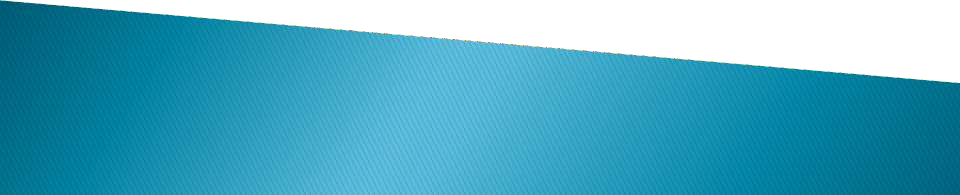


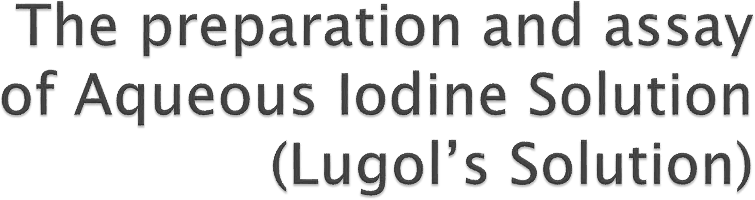
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Na2S2O3 K2Cr2O7

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* 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:`



1. **Assay of I2**,

***note***: *we must use stoppered flask* ***Each1ml of N/10 Na2S2O3≡0.01269 gm I2***

≈ N/10 Na2S2O3

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

until the end point colorless

10ml of unknown 10ml D.W



1. **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 KIO3 required subtract ¼ no. of ml of N/10 Na2S2O3 required in the assay of I2



***each 1ml of the remainder is≡ 0.0166gm of KI***

* Chemical principle for I2 assay:

I2 will oxidize chemically equivalent amount of sod. thiosulfate.

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stable cpd



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* ***Chemical factor***: **Homework**

Each1ml of 0.1N Na2S2O3≡0.01269gm of Iodine Each1ml of M/20 KIO3 ≡0.0166gm of KI Each1ml of M/20 KIO3 ≡0.02538gm of iodine



* + **Calculation:**
  + ***For I2:*** original sol. Vol.

***25 100 X= 2.5ml of original sol. X 10***

***V’ x ch.fact. 0.01269x 100/2.5 = %w/v of iodine in orig. sol.***

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***X 5***

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( req. for KI) in the total assay) o.1N

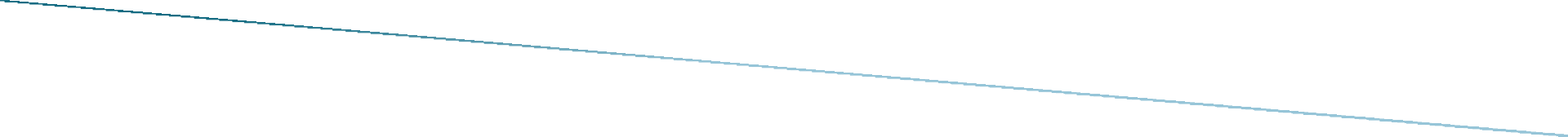
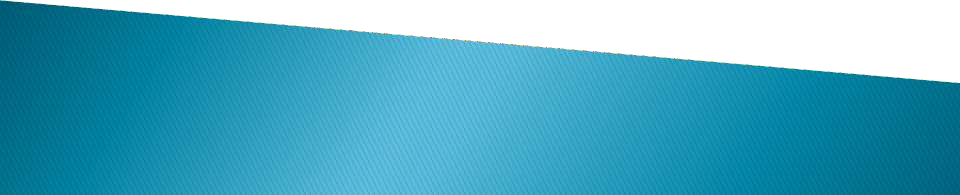
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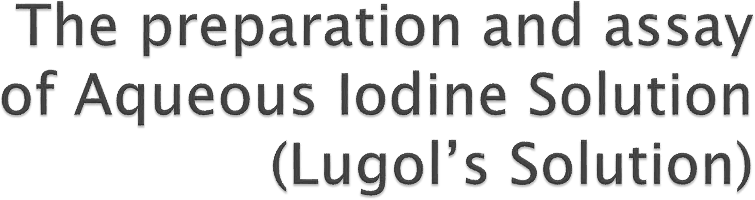


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