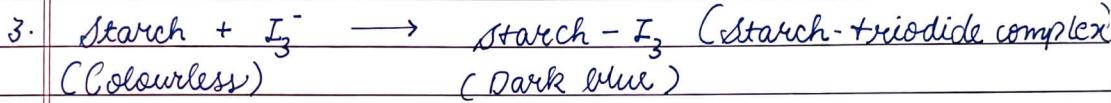
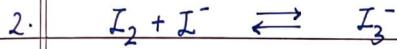
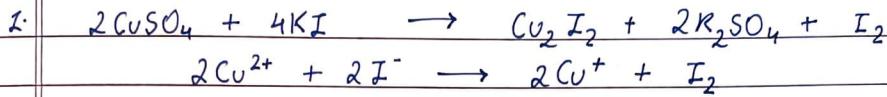


- Aim: Estimation of Cu in brass sample using standard sodium thiosulfate solution (Iodometric titration)

- Theory:



- Procedure:

1. 0.5g of brass is dissolved in 10mL conc. HNO_3 & dilute it upto 100mL with distilled water in standard measuring flask.

2. Pipette 10mL of this diluted alloy solution (test solⁿ) into a conical flask then add a few drops of dil. NH_4OH solⁿ until a slight permanent ppt. ^{remain}

3. The ppt is redissolved by adding acetic acid drop by drop till the solⁿ becomes clear.

4. Then about 5mL of a 5% KI solⁿ is added & titrate the free liberated iodine with 0.05N $\text{Na}_2\text{S}_2\text{O}_3$ solution from the burette using 1% starch solⁿ

Observation Table -

Sr No.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Difference (mL)	Concurrent Reading (mL)
1	0.0	9.2	9.2	
2	0.0	8.2	8.2	8.2
3	0.0	8.2	8.2	
4	0.0	8.2	8.2	

Calculation -

$$1000 \text{ mL IN } \text{Na}_2\text{S}_2\text{O}_3 = 63.57 \text{ g of Cu} = 249.57 \text{ g } \text{CuSO}_4 \cdot 5\text{H}_2\text{O}$$

$$\therefore 1 \text{ mL IN } \text{Na}_2\text{S}_2\text{O}_3 = 0.06357 \text{ g of Cu} = 0.24957 \text{ g } \text{CuSO}_4 \cdot 5\text{H}_2\text{O}$$

$$A \text{ mL of } 0.05 \text{ N } \text{Na}_2\text{S}_2\text{O}_3 = 8.2 \text{ mL}$$

$$\therefore \text{Quantity of Cu in given soln} = A \times 0.05 \times 0.06357 \times 100 / 10$$

$$= B = \underline{\underline{0.26 \text{ g}}}$$

$$\% \text{ of Cu in the alloy} = X \times 100 / W = 52\%$$

B = quantity of Cu.

W = weight of alloy taken = 0.5g

A : Constant Burette Reading -

as an indicator.

5. The end point is marked with the disappearance of blue colour.
6. Repeat the titration 3-4 times.

- Observations :

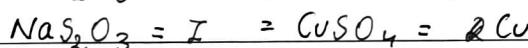
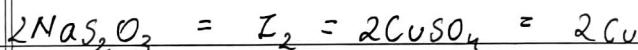
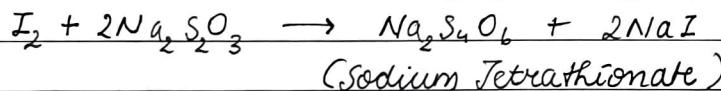
Burette - 0.05N $\text{Na}_2\text{S}_2\text{O}_3$ solution

Flask - 10mL dil. alloy solⁿ + NH_4OH (dropwise till slight permanent ppt) + HAC (till ppt dissolves) + 5% KI.

Indicator - Starch solⁿ (2-3 drop)

End pt - disappearance of blue colour

- Equations :



- Results :

1. 10mL of diluted solⁿ required = 8.2mL of 0.05N $\text{Na}_2\text{S}_2\text{O}_3$
2. Quantity of Cu in the given solⁿ = 0.26g
3. % of Cu in alloy = 52%

- Aim : To determine hardness of water by EDTA method.
- Apparatus : Burette, pipette, flask, test tube
- Preparation of standard solutions / reagents :

A. Standard hard water (0.01M CaCl_2) -

Weigh accurately 0.5g of dried CaCO_3 , transfer to 500ml volumetric flask, add about 100ml of water. Further add 1N HCl solution drop-wise till effervescence ceases and the solution become clear. Make up the solution to the mark with distilled water and shake the flask well for uniform concentration.

B. EDTA solution (0.01M EDTA) -

Dissolve 4g of EDTA crystals along with 0.1g MgCl_2 in little distilled water in 1L standard flask and make it upto the mark with distilled water. Shake the flask for uniform concentration.

If the solution is turbid, add a few drops of 0.1N NaOH solution to the solution clear.

C. Indicator (EBT solution) -

Dissolve 0.5g of Eriochrome Black-T in 100ml of ethanol / methanol. Date the bottle. Solutions older than 6 weeks should not be used.

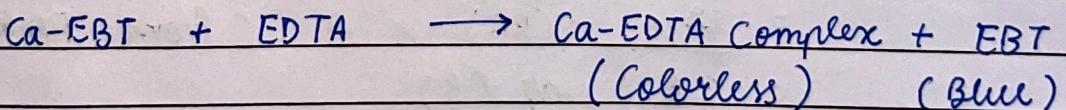
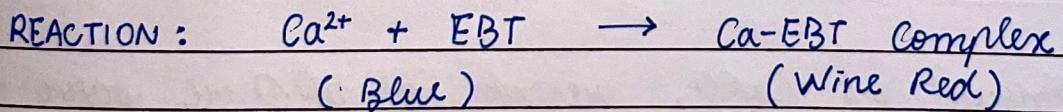
D. Buffer solution (pH 10) :

Add 6.75g of NH_4Cl to 5ml of conc. ammonia solution and dilute with distilled water to one liter. pH will be slightly above 10.

• Procedure :

PART I - Standardisation of EDTA solution

Take 10mL of 0.1M CaCl_2 solⁿ in a conical flask & $\frac{1}{2}$ test tubl of buffer solⁿ. Then add 2-3 drops of indicator solution. Titrate carefully against EDTA solution to the end point where the color changes from wine-red to pure blue. The titration should be carried out ~~not~~ slowly near the end point with constant stirring. No tinge of red color should remain in the solution. Repeat the titration with four other aliquots of the Ca^{2+} solⁿ. Calculate the molarity of the EDTA solⁿ & the CaCO_3 equivalent of EDTA solⁿ.



PART 1

STANDARDISATION OF EDTA SOLⁿ

Observation :

Burette : 0.01M EDTA solⁿ

Flask : 10 mL 0.01M CaCl_2 solⁿ + $\frac{1}{2}$ T.T of buffer solⁿ

Indicator : Eriochrome Black-T (EBT) solⁿ (2-3 drops)

End pt : Wine Red to Blue

Observation Table :

Sr. no.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Differences (mL)
1	0	9.0	9.0
2	0	8.7	8.7
3	0	8.7	8.7

Concurrent reading : 8.7 mL

Calculation 1 :

$$1 \text{ mole of } \text{Ca}^{2+} = 2 \text{ mole of Na EDTA}$$

Hence,

$$\text{Molarity of EDTA soln} \times \text{Vol. of EDTA soln} = \text{Molarity of } \text{Ca}^{2+} \text{ soln} \times \text{Vol. of } \text{Ca}^{2+} \text{ soln}$$

$$\Rightarrow M_2 \times BR = 0.01 \times 10 \quad BR - \text{Burette Reading}$$

$$\bullet M_2 = \frac{0.1}{8.7} = \frac{0.1}{8.7} = 0.0114 \text{ M}$$

$$\text{Molarity of EDTA soln} = 0.0114 \text{ M}$$

$$1.0 \text{ mL of EDTA soln} = \text{Molarity of EDTA} \times 100 \rightarrow \text{Mwt of } \text{CaCO}_3$$

$$\text{Eq. of } \text{CaCO}_3 = 1.14 \text{ mg of } \text{CaCO}_3 (\text{A})$$

PART-2 Estimation of Hardness of Water

Take 50mL of given water sample in a conical flask. To this sample add $\frac{1}{4}$ test tube of buffer soln & 2-3 drops of indicator soln. Titrate with EDTA soln from burette. End pt. is marked by a color change from wine red to pure blue. Repeat the titration with 4 other aliquots. Calculate the total hardness of the water as ppm of CaCO_3 .

- Results :

The given order sample has total hardness of 282.72 ppm of CaCO_3 .

PART 2

Estimation of Hardness of Water

Burette : 0.0114 M EDTA soln

Flask : 50 mL water sample + $\frac{1}{4}$ TT Buffer soln

Indicator : Eriochrome Black-T-solution (2-3 drops)

End pt. : Wine Red to Pure Blue

Observation Table :

Sr no.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Differences
1	10.0	12.3	12.3
2	10.0	12.4	12.4
3	0	12.4	12.4
4	0	12.4	12.4

Reactions : $\text{Ca}^{2+} + \text{EBT} \rightarrow \text{Ca-EBT Complex}$
(Blue) \rightarrow (Wine Red)

$$\text{Ca-EBT} + \text{EDTA} \longrightarrow \text{Ca-EDTA Complex} + \text{EBT} \\ (\text{Colourless}) \quad (\text{Blue})$$

Calculations :

$$\begin{aligned}
 1) \text{ Amount of } \text{CaCO}_3 \text{ present in } 50.0 \text{ mL water sample.} \\
 &= \frac{12.4}{1000} \text{ (BR)} \times A \text{ (from PART - 1)} \\
 &= \underline{9.57} \text{ mg (B)}
 \end{aligned}$$

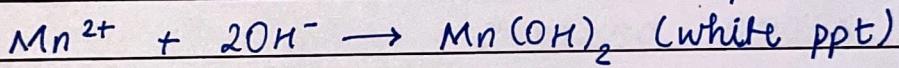
2) Total hardness of given water sample as ppm of CaCO_3

$$= \frac{B \times 1000}{50.0} = \frac{9.57 \times 1000}{50}$$

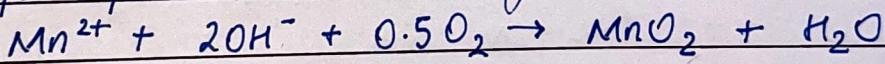
$$= \underline{\underline{282.72}} \text{ ppm} (= \text{Mg/L})$$

- Obj: To determine dissolved oxygen (DO) in waste water.
- Apparatus Required: Conical flask, Burette, test tubes
- Chemical Required: 0.005N $\text{Na}_2\text{S}_2\text{O}_3$ soln, alk. KI soln, MnSO_4 soln, starch soln as indicator.
- Reactions:

If no oxygen is present, a pure precipitate is formed when MnSO_4 and alkali- iodide reagent ($\text{NaOH} + \text{KI}$) are added to the sample



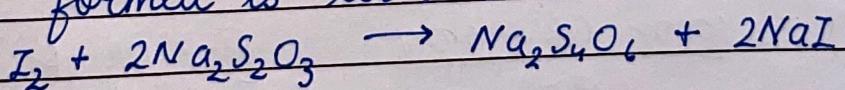
If sample has some oxygen, Mn^{2+} is oxidised to Mn^{4+} & precipitates brown hydrated oxide.



(Brown hydrated ppt)

MnO_2 oxidises iodide to iodine in the presence of acid : $\text{MnO}_2 + 2\text{KI} + \text{H}_2\text{SO}_4 \rightarrow \text{Mn}^{2+} + \text{I}_2 + \text{K}_2\text{SO}_4 + 2\text{H}_2\text{O}$

Iodine formed is titrated with thiosulfate soln



- Procedure :

1. Take 50mL of given water sample in a conical flask.
2. Add 2mL each of alkaline KI solⁿ and MnSO₄ solⁿ.
3. Shake the flask vigorously. Brown ppt. will be produced.
4. Now add carefully 2mL of conc. H₂SO₄ solⁿ and shake.
5. Brownish solⁿ will be liberated, Iodine (I₂) will be produced.
6. Quickly add 2mL of freshly prepared starch solution (indicator) which gives blue colour.
7. Titrate ~~so~~ slowly against standard 0.005N Na₂S₂O₃ solutions till the blue color just disappears.
8. Repeat the titration 4 times.

- Results :

1. Volume of 0.005N Na₂S₂O₃ solⁿ required for 50mL of given water sample = 12.0 mL
2. Dissolved oxygen in the given water sample = 9.6 mg/L

Burette : 0.005N $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ solⁿ

Flask : 50mL water sample + 2mL alkaline KI solⁿ + 2mL of MnSO_4 solⁿ + 2mL of conc. : K_2SO_4

Indicator: 2mL of starch solⁿ

Color change: Blue to colourless

Observation Table :

Sr no.	Initial Burette Reading (mL)	Final Burette Reading (mL)	Differences (mL)
1	0	12.1	12.1
2	0	12.0	12.0
3	0	12.0	12.0

Calculation : 1000 mL requires 8mg of dissolved oxygen
1mL $\text{Na}_2\text{S}_2\text{O}_3$ = 8mg of dissolved oxygen
1mL $\text{Na}_2\text{S}_2\text{O}_3$ = 0.04mg of dissolved oxygen

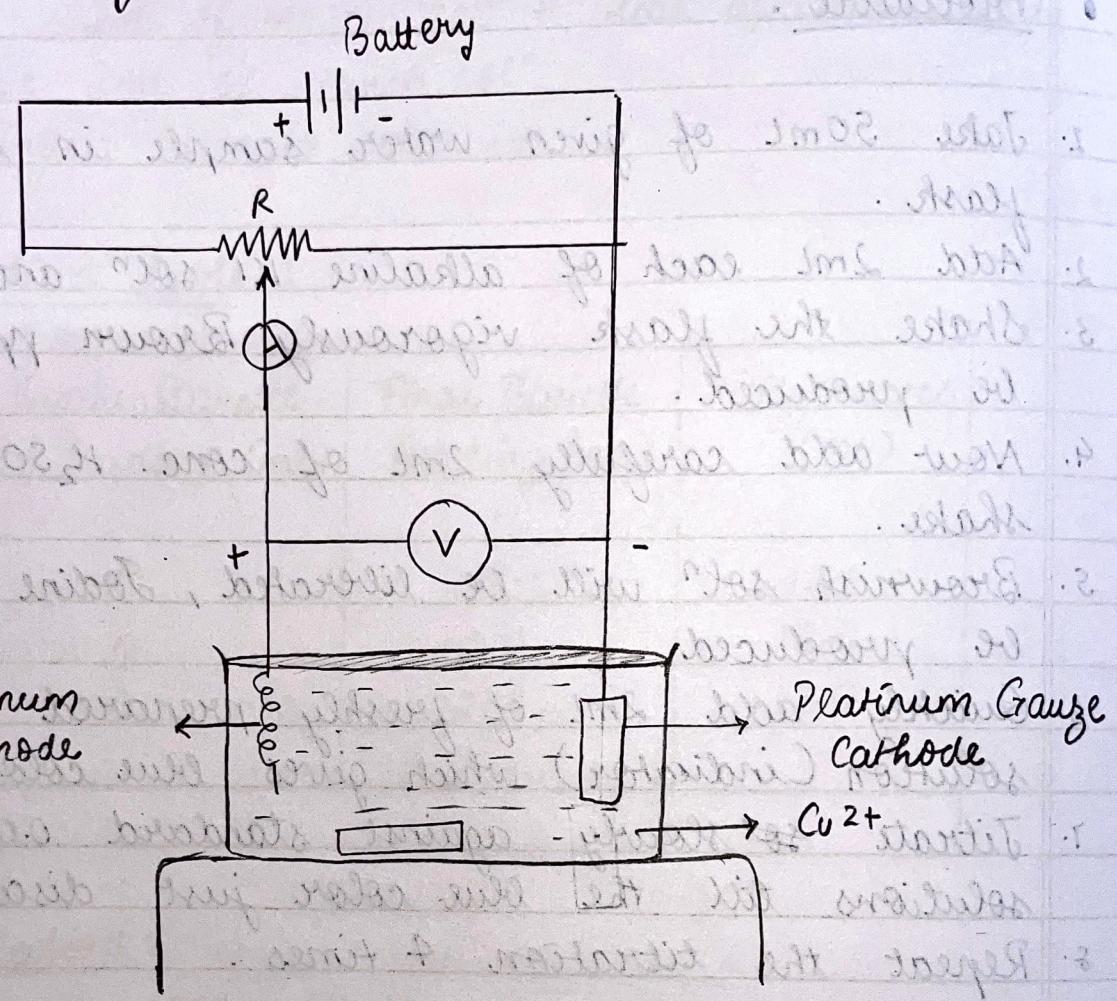
xmL 0.005N $\text{Na}_2\text{S}_2\text{O}_3$ = $x \times 0.04$ mg of dissolved oxygen

Sample taken :

$$(\text{mg/L}) = \frac{1000 \times \text{BR} \times 0.04}{50} \quad [\therefore \text{BR of } \text{Na}_2\text{S}_2\text{O}_3 = \text{I}_2 \text{ liberated}]$$

- Aim: To study electro-deposition of Cu on cathode
 - Apparatus Required: Electrolytic cell, battery, rheostat, ammeter, $CuSO_4$ solⁿ & other solutions.
 - Procedure:
1. Check the gauge electrode given to you. Consult the instructor if it needs any pretreatment. If necessary give the treatment as it is to be given for spiral platinum anode. Dry it in oven & weight accurately.
 2. Clean the platinum spiral in 1:1 KNO_3 solⁿ for 3-4 minutes. Wash with tap water and then distilled water.
 3. Take exactly 150mL of 2% copper sulphate solⁿ with a burette into 250 mL beaker. The given solⁿ already contains 5mL conc H_2SO_4 & conc. HNO_3 which acts as a depolarizer. Add about 50mL of distilled water.
 4. Arrange the circuit as shown in the circuit diagram. Be sure that the gauge cathode is connected to the negative terminal & platinum spiral anode is connected to the +ve terminal of the power source, as in the circuit diagram. Complete the circuit by plugging in the switch key & raise the beaker containing the electrolyte until the cathode is completely immersed in the

Circuit Diagram



R - resistor , A - ammeter , V - voltmeter

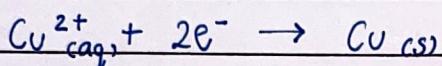
electrolyte. Adjust the rheostat such that the current reading, on the ammeter is about 0.2A. Be sure that the electrodes are not short-circuited & that the magnetic, paddle does not hit either of the electrodes.

5. Stir the solution vigorously with magnetic stirrer. Continue the electrolysis until blue color of solⁿ has entirely disappeared (This will take about 45 mins). Reduce the current to 0.1A. By adjusting rheostat, add 1.0g of urea and continue the electrolysis for another 5 mins. Test the completion of copper deposition by taking a drop test solⁿ with glass rod on a filter paper & placing drop of conc. NH_3 solⁿ close to it. Where the boundaries of the two solⁿ meet, a blue color will be formed if the solⁿ contains copper, if no such blue color is observed; it is an indication that all Cu^{2+} ions reduced to Cu^{\equiv} & deposited on the cathode.
6. To stop electrolysis, turn off magnetic stirrer. Remove the support under beaker and slowly lower the beaker with one hand, while washing the exposed portion of the cathode with distilled water. As soon as the cathode is completely out of solⁿ, cut off the current. Remove the cathode, wash it thoroughly with distilled water & then dip it in a beaker of acetone. Place it on a watch glass & keep it in an electric oven for 2-3 mins

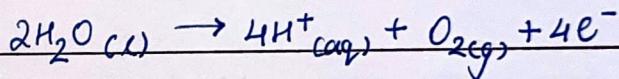
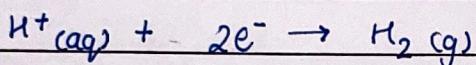
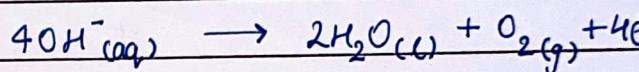
Cool the electrode to room temperature in desiccators and then weight accurately.

- Reactions :

At Cathode



At Anode



- Results :

- 150 mL of the given stock soln contains 0.44 g of Cu.
- Amount of Cu in 1L of the given stock soln = 2.93 g

Observations :

1. Wt. of platinum gauze electrode = 12.24g (W₁)
2. Wt. of platinum + Cu deposited = 12.68g (W₂)
3. Wt. of Cu deposited = W₂ - W₁ = 0.44g (W₃)

4. Current employed = 0.1A

5. Duration of electrolysis = 2700s

6. Vol. of stock copper soln taken 150mL

Calculation:

Wt. of Cu present in 150mL of given = 0.44g (W₃)

Stock soln of CuSO₄ →

$$\frac{W_3 \times 1000}{150} = \frac{0.44 \times 1000}{150} = \underline{\underline{2.93g}}$$

- Aim: To titrimetrically determine 1-Ascorbic acid (Vitamin C)
- Reagents: Freshly boiled & cooled water, 0.1N Iodine soln, 1N sulfuric acid, 1% Starch soln.
- Procedure:

BLANK ESTIMATION -

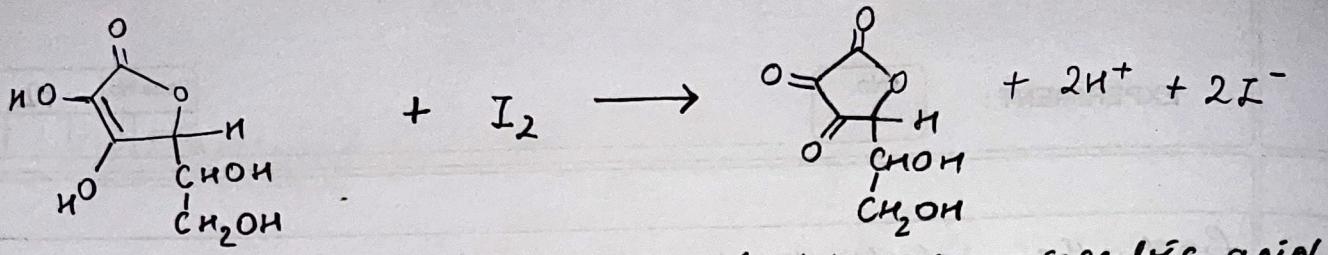
Take blank solution & titrate against 0.1N I_2 soln. End pt. marked by appearance of blue color. Let the volume of titrant consumed be B .

ACTUAL ESTIMATION -

Dissolve the given sample (120g of Ascorbic acid) in 50mL of freshly boiled & cooled water. Then, add 10mL of 1N sulfuric acid, and 3mL of 1% starch soln. Titrate against 0.1N I_2 soln until a persistent blue color is obtained. Let the volume of titrant consumed be V . Then, calculate the percentage of ascorbic acid in the given sample.

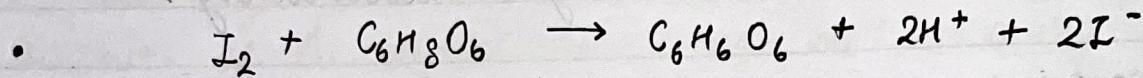
• Result:

Percentage purity of given sample of 1-Ascorbic acid = $\frac{D}{120} \times 100 = 73.33\%$.

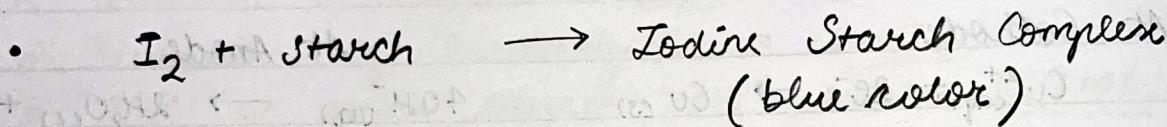


L-Ascorbic acid

L-dehydroxy ascorbic acid



(Vitamin C) (oxidized
Vitamin C)



Observation :

Used sample = Ascorbic acid

BLANK				ACTUAL			
IBR	FBR	Diff	Concurrent	IBR	FBR	Diff	Concurrent
0	0.1	0.1		0	10	10	
0	0.1	0.1	0.1	0	10.1	10.1	
0	0.1	0.1		0	10.1	10.1	
0	0.1	0.1		0	10.1	10.1	10.1

CALCULATION:

1000 mL 1N I_2 soln = 88g L-ascorbic acid

1mL 0.1N I_2 soln = 8.8 mg "

$$(B-A) \text{ mL } 0.1 \text{ N } I_2 \text{ solution} = 8.8 \times (B-A) \text{ mg L-ascorbic acid}$$

D = 88 mg of L-ascorbic acid

$$\text{Now } \% \text{ purity of L-ascorbic acid} = \frac{100 \times D}{\text{wt sample}} = \underline{\underline{73.33\%}}$$