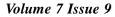
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## Estimation of copper in brass by iodo-potentiometric technique of analysis

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## ABSTRACT

The basic principle used for the estimation of copper in brass is iodometry. Many modifications were proposed to improve the results for quantitative estimation of copper in brass. In the present investigation, prime importance is given to redox potentials of the analyte solution and the measurements are carried out by iodo-potentiometric technique. All the potential measurements are recorded at their respective concentrations and buffer media. The out come of the investigation is that copper in brass can be estimated effectively at pH between 2.0 and 3.5 using NaOH and CH<sub>2</sub>COOH. A linear relationship is obtained for measured potentials  $E_1$ ,  $E_2$  and  $(E_1 - E_2)$ when plotted against the molar concentration of copper or weight of brass taken . The results are in conformity with the results of titrimetric data for the weight of brass samples. Identical results are observed for these two techniques. A calibration plot is obtained by plotting molar concentration (M) of copper in salt and brass with their corresponding measured values of potentials (E<sub>1</sub>) under the conditions of investigation. The determination of molar concentration of copper is feasible from the linear plot graph, if the potential (E<sub>1</sub>) of copper solution is known potentiometrically; and hence, its percentage. © 2008 Trade Science Inc. - INDIA

### INTRODUCTION

Iodide ion is a strong enough reducing agent that many oxidizing agents can react completely with the iodide ion resulting in many useful iodometric processes. The usual procedure involves the addition of an excess of iodide ion to the oxidizing agent analyte which produces iodine, which can be titrated with standard sodium thiosulfate solution. The iodine-thiosulfate reaction is quite fast and the equilibrium is far to the product side.

The basic reaction in the determination of copper in brass using the iodometric method<sup>[1-3]</sup> is represented by the equation:  $2Cu^{2+} + 4I^{-} \rightarrow 2CuI(s) + I$ , thiosulphate. The method has, however, certain difficulties<sup>[4,5]</sup> which are familiar to those who have used the method, and which limit its accuracy. The reaction involving cupric ion and iodide takes place quantitatively since the Cu<sup>+</sup> ion formed as result of the reduction is removed from the solution as a precipitate of cuprous iodide. The reaction involving copper (II) ion and iodide takes place quantitatively since the copper (D ion

This is a rapid, quantitative reaction in acidic solutions (pH = 2.0 - 3.5), if there is a large excess of iodide ion present and if the copper is in the form of a

The latter can be titrated with standard sodium

simple ion rather than a complex one.

dide takes place quantitatively since the copper (I) ion formed as result of the reduction is removed from the solution as a insoluble precipitate of copper (I) iodide.

### KEYWORDS

Brass; Iodometry; Redox; Analyte; Potential; Titrimetric.

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Copper (II) iodide is unstable since the iodide reduces  $Cu^{2+}$  to  $Cu^+$ . The method is, however, widely used on account of its speed but hardly to be classed in accuracy.

The amount of iodine liberated in the reaction between iodide ion and an oxidizing agent is a measure of the quantity of oxidizing agent originally present in the solution. The amount of standard sodium thiosulfate solution required to titrate the liberated iodine is then equivalent to the amount of oxidizing agent. The reaction between iodine and the thiosulfate ion is:

$$\mathbf{I}_2 + 2\mathbf{S}_2\mathbf{O}_3^{2\text{-}} \Leftrightarrow 2\mathbf{I}^{\text{-}} + \mathbf{S}_4\mathbf{O}_6^{-2}$$

As uasual<sup>[1-3]</sup>, the reaction proceeds quantitatively in acidic solutions (pH = 2.0 - 3.5). In strongly alkaline (pH > 4.0) or acidic solutions(pH < 2.0) the oxidation of the thiosulfate does not proceed by a single reaction. In the former, the thiosulfate ion is oxidized to sulfate as well as to the tetra -thionate. In the latter, the thiosulfuric acid formed undergoes an internal oxidation-reduction reaction to sulfurous acid and sulfur. Both of these reactions lead to errors since the stoichiometry of the reactions differs from that shown above for the thiosulfate as a reducing agent. The control of pH is clearly important. In many cases the liberated iodine is titrated in the mildly acidic solution employed for the reaction of a strong oxidizing agent and iodide ion. In these cases the titration of the liberated iodine must be completed quickly in order to eliminate undue exposure to the atmosphere since an acid medium constitutes an optimum condition for atmospheric oxidation of the excess iodide ion. The control of pH of copper solution is, therefore, clearly important.

A cursory survey of literature regarding the estimation of copper indicated that estimation based on the redox potentials of the analyte using potentiometric technique is not available for reference.

It is evident, sometimes, that certain difficulties<sup>[4,5]</sup> arise in the estimation of copper in salts and brass; and the attributed cause may be due to any of the following:

- I. the formation of an intermediate product of complexation during the reaction with the reagents and may also
- II. due to the variable pH conditions of the medium, for reasons.

Due to these, the reduction of  $Cu^{2+}$  to  $Cu^+$  is not quantitative. Hence, we have confined the investigation

to pH = 2.0 - 3.5 to minimize the side reactions.

This has prompted us to investigate /estimate the amount of copper present in a sample through redox potential measurements using potentiometric technique of analysis; and also corroborating this results simultaneously with titrimetric method of analysis maintaining a constant pH condition of the medium . We have attempted to correlate, logically, both of these results successfully.

### **EXPERIMENTAL**

### (i) Cell set up for potentiometric investigation

Potentiometry is an important tool used for quantitative analysis in analytical chemistry. In redox potentiometry, potential of an indicator electrode in equilibrium with redox ions to be determined is measured. Such redox titrations involve the transfer of electrons from the substance being oxidized to the substance being reduced.

### **Oxidized form + n electrons = reduced form**

For such reaction, the potential (E) acquired by the indicator electrode at  $25^{\circ}$ C is given by

 $E = E^0 +$ 

The potential is controlled by the ratio of these concentrations terms. It is possible to titrate two substances by the same titrant provided the standard potentials of the substances being titrated, and their oxidation or reduction products, differ by about 0.2 V.

The electrochemical cell set up desired for the investigation is

### Hg/Hg<sub>2</sub>Cl<sub>2</sub>/KCl (Sat<sup>d</sup>) // Redox analyte/Pt

(reference electrode) (Indicator electrode)

Therefore,  $\mathbf{E}_{\text{Indi}} = \mathbf{E}_{\text{cell}} + 0.2422$  volts

Where,  $E_{ref}$  is a saturated calomel electrode of constant potential, 0.2422 Volts at 25°C. The potentials ( $E_1$  and  $E_2$ ) of copper solutions are not measured under standard conditions.

### (ii) Procedure

In order to ascertain best conditions needed for quantitative precipitation of copper as cuprous iodide in brass, we have carried out the experiments at definite buffer media reagents and pH conditions<sup>6</sup>. The stock solutions of copper in brass is prepared by taking different weights of brass. The potential (E) measurements

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of copper solutions are taken with the requisite addition of the following reagents:

- (a) 4N NaOH
- (b) 1:1 Acetic acid
- (c) Standard Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution
- (d) Freshly prepared Starch
- (e) 5%  $NH_4CNS$  solution.

The copper solutions are treated with NaOH and  $CH_3COOH$  as reagents to maintain a definite pH of 2.0 - 3.5.

## (iii) Dissolution of the brass sample

The dissolution of each brass sample is achieved through the following procedure:

- I. Weigh 0.02 to 0.3g of clean and dry brass samples. Take each sample in a 250 ml conical flask and label the flasks.
- II. Add 6-8 drops of concentrated  $\text{HNO}_3$  into it. Vigorous reaction takes place giving cupric nitrate and liberates oxides of nitrogen is an undesirable product.
- III.Add 15 ml of distilled water and boil the solution in a hot plate.
- IV. Add about 2g of urea; boil for few minutes to expel of the brown fumes of  $NO_2$ , which is an undesirable product.

Wash the sides of the conical flask with about 10 ml of distilled water and the cool the copper solution to laboratory temperature. This is the solution containing certain molar concentration of  $Cu^{2+}$  ions.

## (iv) Potentiometric and iodometric data analysis

The potentiometric and volumetric titrations of the different copper solutions from brass samples are carried out in two-in-one cell as shown (figure 1) maintaining different PH conditions.

1. Pipette out 25 ml of the above copper solution into



Figure 1: A two in one cell set up for potentiometric and titrimetric analysis

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the cell.

- 2. Insert the Pt (indicator) and Calomel (reference) electrodes into the cell. Insert combined glass electrode to note the PH of the medium.
- 3. Use a magnetic bead stirrer to swirl the reacting solutions.
- 4. Record the overall potential(E) in millivolts and note the pH of the medium.
- 5. Add 4N NaOH solution drop by drop till a pale blue precipitate [Cu(OH)<sub>2</sub>)]is formed. Note the potential and pH.
- 6. Add acetic acid drop by drop till a definite desired pH (2.0 to 3.5) is obtained.
- 7. Add 5ml of 10% KI solution. Iodine is liberated. Record the steady potential after 10-15 minutes. This is the potential of the solution under the conditions of reaction of equation (1) and note as  $E_1$ .
- 8. Titrate with standard  $Na_2S_2O_3$  solution till the entire solution turns pale straw yellow.
- 9. Add 2ml of 5%  $NH_4CNS$  followed by the addition of 1ml of freshly prepared starch. The solution turns bluish black.
- 10. Titrate with standard  $Na_2S_2O_3$  solution till the sharp disappearance of blue color. Iodine is consumed completely. Note the Volume standard  $Na_2S_2O_3$ solution consumed.
- 11. Record the overall potential,  $(E_2)$  which accounts for the reaction of equation (2).

The potentials (mV) of copper solutions are recorded after the addition of each reagent. The corresponding pH of solutions are also noted using a digital pH meter. Particularly, the potential difference  $[E_1 - E_2]$ is of prime importance, since it is related to the amount of iodine liberated from an equivalent amount of copper present in the sample. The data obtained for copper in brass solutions are recorded in TABLES 1-2. Cupric ions in brass solutions can also be estimated simultaneously in the same two-in-cell by iodometric titration method. The titrimetric volumes of standard Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution consumed in each case are also recorded (TABLES 1 and 2).

## **RESULTS AND DISCUSSION**

Upon addition of excess iodide to a solution of Cu(II), a precipitate of CuI is formed along with  $I_2$ .

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	TABLE 1.1 outflohet it and the interit e data of copper in brass										
Sr.	8		Weight of BrassWeight of Brass= 0.052 g= 0.064 g			Weight of Brass = 0.102 g		Weight of Brass = 0.143 g		Weight of Brass = 0.157 g	
no.	Reagents	Potential In millivolts	pН	Potential In millivolts	pН	Potential In millivolts	pН	Potential In millivolts	pН	Potential In millivolts	pН
1	CuSO <sub>4</sub>	204	0.13	177	0.2	226	0.19	233	0.50	230	0.15
2	4N NaOH	191	5.37	167	5.03	215	4.48	206	4.4	210	4.4
3	1:1CH <sub>3</sub> COOH	200	3.87	178	3.94	212	3.63	219	4.5	218	3.8
4	10% KI (E <sub>1</sub> )	293	3.97	296	4.22	307	3.92	315	3.8	318	4.09
5	Addition of $Na_2S_2O_3$ till the disappearance of blue color (E <sub>2</sub> )	194	4.04	195	4.3	197	4.1	200	4.37	201	4.32
6	E <sub>1</sub> - E <sub>2</sub>	99		101		110		115	4.9	117	
7	Iodometric titration	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> :	=2.2 ml	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> =	= 2.8 ml	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	= 4.3ml	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> =	= 6.1ml	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> =	= 6.6 ml

TABLE 1 : Potentiometric and titrimetric data of copper in brass

	TABLE 2 : Po	tentiometric and titri	imetric data of coppe	er in brass	
titration	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> =2.2 ml	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> = 2.8 ml	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> = 4.3ml	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> = 6.1ml	Vol Na <sub>2</sub> S

Sr.		Weight of Brass = 0.200 g		8		8	Weight of Brass = 0.255 g		Weight of Brass = 0.302 g		Weight of Brass = 0. 311 g	
no.	Reagents	Potential In millivolts	pН	Potential In millivolts	рН	Potential In millivolts	pН	Potential In millivolts	pН	Potential In millivolts	pН	
1	CuSO <sub>4</sub>	213	0.47	213	0.45	220	0.05	207	0.28	217	0.20	
2	4N NaOH	147	4.28	212	4.30	209	4.05	176	4.07	185	3.8	
3	1:1CH <sub>3</sub> COOH	176	3.77	212	3.2	211	3.7	197	3.3	194	3.3	
4	10% KI (E <sub>1</sub> )	327	4.08	329	3.75	338	4.1	347	3.66	352	4.1	
5	Addition of Na2S2O3 till the disappearance of blue color (E <sub>2</sub> )	204	4.36	205	4.2	208	4.5	211	4.05	212	4.5	
6	E <sub>1</sub> - E <sub>2</sub>	123		124		130		136		140		
7	Iodometric titration	Vol $Na_2S_2O_3$	=2.2 ml	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> =	= 2.8 ml	Vol $Na_2S_2O_3$	= 4.3ml	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> =	= 6.1m	Vol Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> =	= 6.6 ml	

The copper(II) ions oxidise iodide ions to iodine, and in the process are themselves reduced to copper(I) iodide.

 $2Cu^{2+}(aq) + 4I^{-}(aq) \rightarrow 2CuI(s) + I_2(aq)$ 

### (a) The reaction of iodide ions with copper(II) ions

This is the reaction that liberates the iodine for titration: it is

$2Cu^{2+} + 2e^{-1}$	$\rightarrow 2Cu^{+}$	$E^{0}=+0.15V$
$I_2 + 2e^-$	$\rightarrow 2 I^{-}$	E <sup>0</sup> =+ 0.54V

 $2\mathrm{Cu}^{2+} + 4\mathrm{I}^{-} \rightarrow 2\mathrm{CuI} + \mathrm{I}_{2} \qquad \mathrm{E}_{1}^{0} = -0.39\mathrm{V} \tag{1}$ 

The potential data indicates that that the reaction is not spontaneous-but it does occur, and very quickly too. The value of  $E^{\circ}$  can be used to give the equilibrium constant for the reaction, and it comes to around  $10^{-7}$ . This is very small, and indicates little reaction. But this is under standard conditions. The factor that makes the reduction reaction possible is the fact that Cu<sup>+</sup> is not in solution at a concentration of 1 mol dm<sup>-3</sup>.

Copper(I) iodide is an extremely insoluble solid. The equilibrium constant for the reaction

 $Cu^+(aq) + I^-(aq) \rightarrow CuI(s)$ 

is around 10<sup>12</sup>. Thus as soon as any Cu<sup>+</sup> appears it

immediately precipitates, and this is sufficient to drive the reaction over to the right hand side. Indeed it is not hard to show that the equilibrium constant for the production of solid copper(I) iodide as distinct from copper(I) under standard conditions is of the order of 10<sup>5</sup>. This reaction is complete by anyone's standards, and certainly is complete enough for the reaction to be used in quantitative analysis.

The iodine that is liberated can be titrated in the usual manner with standard thiosulphate solution. The reaction involving copper (II) ion and iodide takes place quantitatively since the copper (I) ion formed as result of the reduction is removed from the solution as a insoluble precipitate of copper (I) iodide.

### (b) The reaction of sodium thiosulphate with iodine

The redox potential of the titration part of the reaction of sodium thiosulphate with iodine is as follows:

 $I_2 + 2e^- \rightarrow 2I^-$ ;  $E^o = +0.54$  Volts

 $S_4O_6^{2-} + 2e^- \rightarrow 2S_2O_3^{2-}$ ;  $E^0 = +$  0.09 Volts

The overall equation reaction by convention is:

$$I_2 + 2S_2O_3^2 \rightarrow 2I^- + S_4O_6^2; E_2^0 = +0.45mv$$
 (2)

The reaction is feasible under standard conditions.

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TABLE 3: Estimated percentage of copper in brass by iodo-potentiometric and titrimetric methods

	Weight of	Iodo	- potentiometric data for cop	per in brass	Iodometric analysis data for copper in brass			
S. no.	Weight of brass taken 'g'	Potentio- metric E <sub>1</sub> (mV)	Calculated molar concentration of copper, M (TABLE 4) by graph	Percentage of copper in brass	Volume of 0.232M Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> Solution,ml	Calculated molar concentration of copper, M	Percentage of copper in brass	
1	0.052	293	0.0204	62.32	2.1	0.0195	60.00	
2	0.064	296	0.0250	62.05	2.7	0.0251	62.20	
3	0.102	307	0.041	63.80	4.3	0.0399	62.14	
4	0.143	315	0.057	62.80	6.1	0.0566	62.87	
5	0.157	318	0.062	62.73	6.6	0.0612	61.87	
6	0.200	327	0.078	61.95	8.5	0.0788	62.65	
7	0.207	329	0.082	62.9	8.8	0.0817	62.66	
8	0.255	338	0.098	61.00	10.7	0.0993	61.85	
9	0.302	347	0.120	63.12	12.6	0.117	63.3	
10	0.311	352	0.124	63.30	13.2	0.123	62.43	

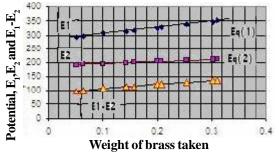


Figure 2: A plot of potentials,  $E_1$ ,  $E_2$  and  $E_1$ -  $E_2$  of copper solutions Vs Weight of brass samples taken

It has been found that iodine is adsorbed onto the surface of the copper(I) iodide precipitate and must be displaced to obtain correct results.  $NH_4CNS$  is usually added just before the end point is reached to displace the adsorbed iodine.

The pH of the copper solution is maintained at constant by adding requisite amount acetic acid drop by drop in a cell set up as shown in the figure 1. The overall potential,  $E_1$ , recorded for equation (1) under experimental conditions (pH =4.00) represents the reaction of cupric ions with iodide liberating iodine (figure 2), while potential contribution, if any, other than due to the reaction stated in equation 1, remains the same and the contribution may be minimum without causing any error for the investigation for copper solutions obtained from brass. Equation 2 represents the redox reaction between the liberated iodine and standard sodium thiosulphate; the overall potential ( $E_2$ ) established after the complete consumption of iodine at the end point of titration.

In the two–in-one cell set up (Figure 1) one can easily note both the potentials ( $E_1$  and  $E_2$ ) and also the volume of standard  $Na_2S_2O_3$  run down at the equivalence point of titration. The experimental data for dif-

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TABLE 4 : Potentiometric and	l titrimetric data for copper in
copper sulphate <sup>[6]</sup>	

Molar concentrations of copper solution, M	Potentials, E <sub>1</sub> of copper solutions from its salt
0	282 (intercept)
0.01	286
0.02	292
0.03	298
0.04	304
0.05	310
0.06	316
0.07	322
0.08	328
0.09	334
0.1	340

1 mV change of potential = 0.0018M

ferent weights of brass are noted in TABLES 1-4.

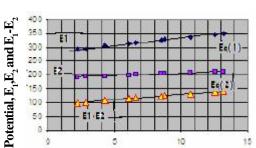
The decrease of potentials with the decrease of brass weights can be accounted due to decreasing amount of copper ions present in the copper solution for redox  $Cu^{2+}/Cu^+$  couple (equation 3).

$$E1 = E^{o}_{Cu}^{2+} / Cu^{+} + \frac{2.303RT}{F} \log \frac{[Cu+]}{Cu^{2+}}$$
(3)

Similarly, it is observed that the steady potential,  $E_2$ , decreases with the decrease of  $I_2$  which in turn depends on copper solutions containing different weights of brass . The equation (4) illustrates the behaviour of decrease of potential with iodine content in solution after the addition of KI.

$$E1 = E^{o}_{I_{2}}/_{I} - + \frac{2.303RT}{2F} \log \frac{[I^{-}]}{[I_{2}]}$$
(4)

It is reasonable to understand that the difference of the two overall potentials  $E_1$  and  $E_2$ , (equations 1 and 2); that is,  $[E_1 - E_2]$  for a particular concentration of copper ions under the conditions of investigation is related to the amount of I<sub>2</sub>liberated. Thus the stochiometry



Volume of sodium thiosulphate consumed Figure 3: A plot of potentials,  $E_1$ ,  $E_2$  and  $E_1$ -  $E_2$  of copper solutions Vs volume of standard sodium thiosulphate consumed at the end point

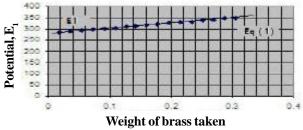


Figure 4 : A linear plot of potentials( $E_1$ ) of copper in brass Vs Weight of brass taken (TABLE 5) for investigation

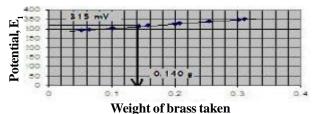
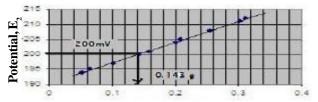


Figure 5: Determination of weight of brass from unknown potential,E, of copper solution of brass



Weight of brass taken Figure 6 : Determination of weight of brass from unknown potential,E, of copper solution of brass

involved in the iodometric reaction is 2 mols of  $Cu^{2+}$  ions  $\equiv$  2 moles of  $Na_2S_2O_3$  $\equiv$  1 mole of  $I_2 \equiv [E_1 - E_2]$ 

Figure 2 shows the linear variation of the potentials,  $E_1$ , which can be accounted due to the increased molar concentration of copper ions in the prepared solutions. Similar behaviour of linear increase in the potential values are observed for  $E_2$  and  $[E_1 - E_2]$  (figure

(5)

2) of brass solutions with the increase in brass weight or copper content in solution.

The data given in TABLE 1 -2 and the analysis of figure 2 clearly supports the stoichiometry (equation 5); which hints a quantitative relationship with the number of moles iodine liberated for stoichiometric moles of copper present in solution and in turn; is equal to [E<sub>1</sub>- $E_{2}$ ]. Hence,  $[E_{1} - E_{2}]$  data is useful to find out the amount of copper present in the brass solution sample. Since, the stoichiometry of equation (5) holds good, any one of the potential values ( $E_1$  or  $E_2$  or  $[E_1 - E_2]$  may be taken to determine molar concentration of copper graphically. The molar concentration of copper in brass is also obtained from the volumes of standard thiosulphate consumed at the end point of iodometric titration for the stoichiometric equivalent amount of copper ions. The results obtained from both of these techniques are identical.

During the investigation process, different weights of brass samples are taken (second column of TABLE 3) for estimation of copper and their respective potentials  $E_1$  and molar concentrations (M) copper in brass are found out by both iodo-potentiometric and titrimetric methods. It can be remarked that the percentage of copper present in brass samples are nearly identical from both these techniques. Therefore, iodo-potentiometric technique is a useful tool to analyse copper in brass.

The overall potential data  $E_1$  (equation 1) of copper solutions in brass (TABLE 3) and also the  $E_1$  data reported (TABLE 4) for copper in copper sulphate solutions in earlier communication<sup>6</sup> has very close similarity.

Surprisingly, all these potentials ( $E_1$ ) data fall in line when plotted with the their molar concentrations of copper (Figure 9). On analysis, analytical data indicated that 1mV change of potential ( $E_1$ )  $\equiv$  0.0018M of copper ions in solution. Based on this concept, the molar concent-rations of copper in brass for different weights are calculated, since it is found to vary linearly with the potential, $E_1$  values. The graph obtained is linear (figure 4) when potentials( $E_1$ ) of copper in brass is plotted against the weight of brass samples taken. The linear plot of graph(figure 3) indicate that any value out of these three is;  $E_1$ ,  $E_2$  or ( $E_1$ ,  $E_2$ ) may be taken to determine the weight of brass or molar concentration of copper in brass. This can be illustrated in figures 5-7.

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However, the prime importance is given for the quantitative relationship between the molar concentration of copper in brass than the weight of brass dissolved with their corresponding potential values . This has prompted us to plot molar concentration of copper in brass solution (TABLE 3, column 4) against the potentials, $E_1$ , under the conditions of investigation (figure 8).

Determination of molar concentration of copper in brass solution and percentage.

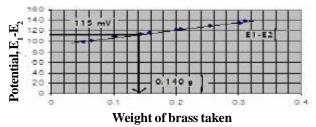


Figure 7: Determination of weight of brass from unknown potential,  $(E_1-E_2)$  of copper solution of brass

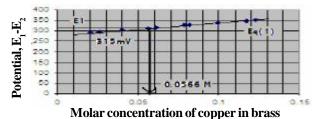


Figure 8: Determination of molar concentration of copper solution of brass from knowing the potential  $E_1$  of copper solution of brass

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The procedure to be followed is as given

- Dissolve the brass samples and collect the iodopotentiometric and titrimetric titration data as described elsewhere and find out potential change, 1mV = molar concentration of copper solutions (TABLE 4) or note the slope of linear plot obtained from the data of TABLE 4.
- 2. Plot graphs as stated:

(i) Potentials  $(E_1)$  Vs molar concentration of copper in brass samples .

(ii) Potentials  $(E_1)$  Vs Volume of standard thiosulphate consumed of copper in brass samples .

Dissolve a piece of brass(known weight) as usual and note its potential( $E_1$ ) and also find out the volume of standard thiosulphate (V ml) needed at the end point of titration.

- 4. Find out the molar concentration(M) of copper in brass for specific weighed sample from graph (Figure 9) and report its percentage.
- 5. Cross check the results with the molar concentration and percentage obtained from titrimetric procedure.

It is clear from figure 9 that the potentials,  $E_{1,}$  increases linearly with the increase of molar concentration of copper in salt and brass for the solutions prepared under the conditions of this investigation. Weigh any brass sample and subjected it to the measurements of the kind referred to in the present investigation and it is possible to report the molar concentration of copper

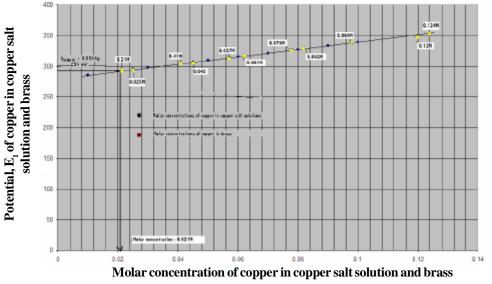


Figure 9: A plot of molar concentration of copper in salt and brass samples Vs measured potentials, E<sub>1</sub> (equation 1)

TABLE 5 : Data indicating the percentage of copper in brass for different weights of brass samples

		Iodo-j	potentiometric analysis			Iodometric titratio	
S. no	Weight of brass sample taken (g)	Potentials, E <sub>1</sub> (mV)	Molar concentration of copper estimated (M) 1mV change = 0.00180M (TABLE 4)	Estimated percentage of copper in brass sample	Volume of 0.232M Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> Solution, ml	Molar concentration of copper estimated (M)	Estimated percentage of copper in brass sample
		282 (At the				·	
	0.0	intercept point in the graph )	0		0	0	-
1	0.0091	284	0.0036	62.90	0.38	0.0035	61.56
2	0.0091	284	0.0072	62.84	0.38	0.0071	61.43
3	0.0172	288	0.0108	63.08	1.14	0.0105	61.32
4	0.0272	288	0.0108	63.00	1.14	0.0105	63.00
5	0.0454	290	0.0144	62.98	1.90	0.0176	61.58
5 #	0.0434	292	0.0204	62.32	2.1	0.0195	60.00
# 6	0.052	293 294	0.0204	63.07	2.1	0.0193	61.61
#	0.0544	294 296	0.0210	62.50	2.28	0.0251	62.30
# 7	0.004	290 298	0.0232	62.66	3.04	0.0231	61.15
8	0.073	298 300	0.0288	62.76	3.42	0.0281	61.41
8 9	0.082	300	0.0324	62.84	3.42	0.0317	61.62
9 10	0.091	302 304	0.0396	62.84 62.90	5.8 4.18	0.0335	61.02
10 #	0.100	304 307	0.0396	63.80	4.18	0.0385	62.14
# 11			0.041	62.96		0.0399	62.14 62.66
11 12	0.109	306	0.0432	62.96 63.00	4.56	0.0423	
	0.118	308	0.0408		4.94		61.65
13	0.127	310		63.07	5.32	0.0493	61.66
14	0.136	312	0.0540	63.0	5.7	0.0529	61.79
15	0.143	314	0.0576	63.98	6.08	0.0564	62.65
16	0.152	316	0.0612	63.96	6.46	0.0601	62.80
#	0.157	318	0.062	62.73	6.6	0.0612	61.87
17	0.161	318	0.0648	63.93	6.84	0.0635	62.65
18	0.170	320	0.0684	63.91	7.22	0.0671	62.70
19	0.179	322	0.0720	63.89	7.6	0.0705	62.56
20	0.188	324	0.0756	63.87	7.98	0.0741	62.61
21	0.197	326	0.0792	63.86	8.36	0.0776	62.57
#	0.200	327	0.078	61.95	8.5	0.0788	62.65
#	0.207	328	0.0828	63.50	8.74	0.0811	62.24
22	0.216	330	0.0864	63.54	9.12	0.0846	61.78
23	0.225	332	0.0900	63.54	9.5	0.0882	62.27
24	0.234	334	0.0936	63.43	9.88	0.0917	62.25
25	0.243	336	0.0972	63.54	10.26	0.0952	62.23
26	0.252	338	0.1008	63.54	10.64	0.0987	62,22
#	0.255	338	0.098	61.00	10.7	0.0993	61.85
27	0.261	340	0.1044	63.54	11.02	0.1023	62.26
28	0.270	342	0.1080	63.54	11.40	0.1058	62.24
29	0.279	344	0.1116	63.54	11.78	0.1093	62.23
30	0.288	346	0.1152	63.54	12.16	0.1129	62.27
31	0.297	348	0.1188	63.54	12.54	0.1164	62.26
#	0.302*	347	0.120	63.12	12.6	0.117	63.3
32	0.305	350	0.1224	63.75	12.98	0.1205	62.76
#	0.311*	352	0.124	63.30	13.2	0.123	62.43
33	0.314	352	0.1260	63.74	13.30	0.1234	62.42
34	0.321	354	0.1296	62.35	13.68	0.1270	62.85

ImV change of potential = 0.00180M . Note: Marked # Weight of brass taken (TABLE 3) for actual analysis

in brass solution(for 25 ml) from the measured  $E_1$  value for the weight brass taken, as shown in figure 9.

The percentage of copper in brass(dissolved in 25ml) can be determined thus;

Percentage of copper in brass = 
$$\frac{0.021 \text{ M} \times 63.54 \times 100}{40 \times 0.0544 \text{ g}} = 61.35$$

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