CHAPTER III

INHIBITION OF CORROSION OF COPPER IN STAGNANT CONDITION
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In this chapter, the results obtained for the inhibition of corrosion of copper in stagnant condition in acidic and neutral or nearly neutral media are discussed and an attempt has been made to understand the results in light of various theories on mechanism of inhibition of corrosion of metals. For convenience of discussion the inhibitors are divided according to the polar groups associated with them as below:

3.1 Mercaptans
3.2 Sulphides
3.3 Azoles and thiazoline
3.4 Thioureas

The weight loss data are given on pages 202 to 250 for all the media employed in the present investigation. The order of the media is hydrochloric acid, acetic acid and its chloroderivatives, 3% sodium chloride. The results of the galvanostatic measurements are represented graphically and are given on pages 251-312. For convenience of the discussion some results are tabulated at proper places. The structures of the compounds studied as inhibitors are given at appropriate places.
3.1 **MERCAPTANS**

**RESULTS**

(A) *Hydrochloric acid:*

Weight loss results are given in tables 3.1 and 3.2 on pages 202 and 203.

The inhibitive power of mercaptans increased with increase in their concentrations in both the acid solutions at both the durations. There appeared to be much less regular behaviour in nature of inhibition with increase in the duration at a given concentration of the acid. The inhibitive power in general increased with increase in the concentration of acid at a given duration in the cases of butyl-mercaptan and dodecylmercaptan, while in the rest of the cases it mostly decreased. Excellent inhibition was observed in the case of dodecylmercaptan at its highest concentration.

**Galvanostatic Measurements (Fig. 1 on page 251):**

When the copper plate was made cathode in 0.5M HCl a sudden jump in potential was observed beyond the current density of $5.0 \times 10^{-5} \text{amp/cm}^2$. When the current density was increased beyond $10 \times 10^{-5} \text{amp/cm}^2$ the magnitude of the potential became gradual and slow. In the case of anode the polarisation was much less even at very high current density. This indicates that it is only the cathode which is predominantly polarized.
A sudden jump in the cathode polarisation was obtained at lower current densities in the cases of propyl, butyl- and dodecyl mercaptans. At higher current densities the cathodic polarisation becomes quite significant in all the above three cases. The polarisation of the anode, compared to blank was quite negligible in all mercaptans save propyl mercaptan.

(B) Acetic acid and Chloroacetic acids:

Ethyl mercaptan:

Weight loss results are given in table 3.3 on page 204.

The inhibitive power of ethyl mercaptan increased with increase in its concentration. It had some tendency to decrease inhibition with increase in the duration. Except in monochloroacetic acid the inhibition increased with increase in the concentration of acid at a given duration. In monochloroacetic acid the reverse trend prevailed. The inhibitive power in general increased with increase in chlorine content of acid.

Propyl mercaptan:

Results are given in table 3.4 on page 205.

The inhibitive power of propyl mercaptan increased with increase in its concentration except in 0.5M monochloro and dichloro acetic acids. It usually showed excellent inhibition at its highest concentration in acetic and trichloroacetic
acids. It mostly showed decrease in inhibition with increase in duration except in 0.1M trichloroacetic acid. The inhibitive power mostly increased with increase in the concentration of acid at a given duration.

**Butyl mercaptan:**

Results are given in table 3.5 on page 206.

The inhibitive power of butyl mercaptan usually increased with increase in its concentration except in 0.5M dichloroacetic acid where a maximum was observed at some intermediate concentration. In general its inhibition decreased with increase in the duration except in trichloroacetic acid where the reserve trend prevailed. Normally the inhibition at a given duration, increased with increase in concentration of the acid.

**Dodecyl mercaptan:**

Results are given in table 3.6 on page 207.

The inhibitive power of dodecyl mercaptan increased with increase in its concentration, exhibiting usually excellent inhibition at its highest concentration. There were not significant variations in its inhibitive power with increase in duration except at very low concentration. Normally there was increase in inhibition, at a given duration with increase in the concentration of the acid particularly so at higher concentrations. It exhibited very
excellent inhibition in 0.5M trichloracetic acid at higher concentrations.

**Galvanostatic Measurements:**

**Acetic acid** (Fig. 2 on page 252):

When copper plate is made cathode in 0.5M acetic acid, significant polarisation took place at the lower current densities. A difference of about -250 mV was observed when the current density was increased from $5.0 \times 10^{-5}$ to $20 \times 10^{-5}$ amp/cm$^2$. Compared to hydrochloric acid the magnitude of polarisation was usually low. Polarisation of anode was quite negligible compared to the cathode in 0.5M acetic acid and also compared to that in 0.5M hydrochloric acid.

Mercaptans in general, polarised both cathode and anode, the magnitude of cathode polarisation was always higher than the anode polarisation. It is interesting to note that all the mercaptans showed more than 95% inhibition, the polarisation of dodecyl mercaptan was invariably higher compared to anode in other mercaptans.

**Monochloracetic acid** (Fig. 3 on page 253):

In monochloracetic acid the extent of polarisation of cathode was higher than that in HCl and much higher than that in acetic acid.

Mercaptans in general polarise both cathode and anode to a greater extent than thioureas. In the case of
dodecylmercaptan particularly a very high jump was observed even at lowest current density applied, the magnitude of the jump being about 1100 mV.

**Dichloroacetic acid** (Fig. 4 on page 254):

When copper placed subject to polarisation in absence of inhibitor in 0.5M dichloroacetic acid, the increase polarisation was roughly linear at most of the current densities. The magnitude of the polarisation of cathode was of the same order as in monochloroacetic acid. The polarisation of anode was linear and much less.

Mercaptans in general, showed high polarisation of the cathode at most of the current densities, dodecylmercaptan showing highest polarisation at all current densities more particularly at higher current densities. Compared to the cathode polarisation of the anode, though high compared to the blank, was not much significant compared to the polarisation of the cathode.

**Trichloroacetic acid** (Fig. 5 on page 255):

Compared to all the acid solutions mentioned earlier the polarisation of the cathode in 0.5M trichloroacetic acid at most of the current densities was lower. Anode polarisation can be considered negligible compared to that of the cathode.
The behaviour of the mercaptans was practically of the same pattern as in dichloroacetic acid. However the polarisation of the anode is somewhat more in trichloroacetic acid.

(C) 3% SODIUM CHLORIDE:

Weight loss results are given in table 3.7 on page 208.

Among mercaptans only dodecyl mercaptan showed excellent inhibition at its highest concentrations in 3% sodium chloride solution.

Acceleration of corrosion or poor inhibition was observed at lower concentrations, the inhibitive power in general increasing with increase in concentrations.

Galvanostatic Measurements (Fig. 6 on page 253):

In absence of inhibitor i.e. blank, the cathodic polarisation of the copper increased somewhat at lower current densities. A sudden jump in potential was observed beyond a current density of $10.0 \times 10^{-5}$ amp/cm$^2$. Thereafter the polarisation became gradually higher with increase in current densities. The polarisation of anode was very slow and steady with increase in current density.

Mercaptans (Fig. 6 on page 256):

Both at lower and higher current densities, the
polarisation of the cathode was somewhat higher in the presence of mercaptans, in general, the extent of polarisation increasing with increase in chain length of the compound. The nature of anode polarisation was of the same pattern as the blank except at higher current densities in the case of dodecyl mercapta.

(D) Artificial Sea Water:

Weight loss results are given in table 3.8 on page 209. The trend of inhibition was same as in 3% sodium chloride solution. All mercaptans showed somewhat better inhibition compared to 3% sodium chloride solution.

Galvanostatic measurements (Fig. 7 on page 257):

The nature of polarisation of copper plates in artificial sea water was practically of the same nature as in 3% sodium chloride solution.

Towards the polarisation of cathode or anode the behaviour of artificial sea water and 3% sodium chloride were similar in nature except that the overall polarisation was somewhat low in artificial sea water. With dodecyl mercaptan, however, a sudden jump in cathode potential was observed at very low current density in artificial sea water, the anode also showing somewhat higher polarisation at the intermediate current density.
**MERCAPTANS**

\[
\begin{align*}
\text{Et}	ext{yl mercaptan} & : \text{CH}_3\cdot\text{CH}_2\cdot\text{SH} \\
\text{Propyl mercaptan} & : \text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{SH} \\
\text{Butyl mercaptan} & : \text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{SH} \\
\text{Dodecylmercaptan} & : \text{H}_3\text{C} \left(\text{CH}_2\right)_{10}\text{CH}_2\text{SH}
\end{align*}
\]

**SULPHIDES**

\[
\begin{align*}
\text{Di}	ext{ethyl sulphide} & : \text{H}_5\text{C}_2 \text{—S—C}_2\text{H}_5 \\
\text{Dibenzyl disulphide} & : \text{C}_6\text{H}_5\text{CH}_2 \text{—S—S—CH}_2\text{C}_6\text{H}_5
\end{align*}
\]
DISCUSSION

The inhibition action shown by organic compounds on the dissolution of metals is generally attributed to interaction by adsorption between inhibitor and metal surface, although some authors consider that this phenomenon forms only a first stage of inhibition process proper\(^ {10}\). The effective inhibiting action has been normally linked with the phenomenon of chemisorption which may range from simple adsorption to a formation of true physical barriers. The action of inhibitor is also attributed to an increase in the overvoltage of the proton discharge process which forms the partial cathodic reactions of the corrosion process, to an increase in the ohmic resistance due to presence of an inhibitor film at the metal solution interface or non specific adsorption\(^ {11}\).

Variation in inhibitive effects of substituents of an organic compound is attributed to consequent changes of solubility, electronic structure of the functional groups, the structure of the adsorbed film etc. The possibility of correlating structural characteristic with the inhibitive properties of organic compounds is justified by the fact that the metal inhibition interactions are based on chemisorption. The strength of the bond depends upon the nature of electron donor or acceptor.
A systematic approach to the mechanism of inhibition was attempted by Charles A. Mann(12). He studied along with the other properties of the organic compounds, the effect of length of aliphatic chain in case of amines on inhibition of iron in sulphuric acid solution. He concluded that inhibitor is attached to the cathodic area of the metal through the nitrogen atom. When attached, the cross sectional area projected down on the metal and the closeness of the packing of the inhibitor ions in a covering layer will determine the extent of inhibition (if this cover is penetrable by $H^+$ ion, corrosion will continue, though considerably lessened). The cross sectional area projected by the inhibitor may decide the extent of inhibition. The effect of length of aliphatic chain on inhibition has been explained by him as follows: If this chain stood perpendicular to the metal surface, they should all regardless of length, give the same protection for a definite amount of inhibitor. The longer chains are inclined to the surface, the projecting area then increasing with length of chain thus covering increased inhibiting value.

While working on mild steel in sulphuric acid Chiao and Mann(13) came to the conclusion that long chain aliphatic amines inhibit corrosion because of amines being adsorbed over the metallic surface. They assumed that at lower concentrations of the inhibitor, the adsorbed molecules
are quite far away from each other and have therefore considerable space to move about probably without even touching one another even though they may be tilted. The long hydrocarbon chain of large aliphatic amines can therefore be considered inclined towards the metal surface at lower concentrations. The angle of inclination increases (the effective molecular area including projected area decreases) with increasing amount of adsorption of inhibitor concentration in solution. The effective molecular area, including the projected area reaches a constant minimum value at its critical concentration. The critical concentration varies from one inhibitor to another. The more efficient the inhibitor, the lower will be its critical concentration.

Molecular area of the inhibitor projected onto the metallic surface has been considered as a structural parameters to account for the extent of inhibition by Ayers et al.\(^{14}\) who studied a series of pyridine derivatives. A correlation, although a slight one, was found between the values of the inhibition and those of the circular areas relating to the arrangement parallel to the surface.

Podobaev et al.\(^{38\#}\) however, obtained the results which were not agreeable with the above suggestion. They studied the inhibition exhibited by a series of derivatives of propargyl alcohol (OH-\(\text{CH}_2\text{C}≡\text{OH}\)), keeping the triple
bond in the molecule unchanged and substituting nitrogen containing heterocyclic nuclei in the hydroxyl group or esterifying it with fatty acid. Inspite of sizable increase in projected molecular area of these derivatives, the inhibiting efficiency was always low compared to the parent material, propargylalcohol.

An abstract of the results obtained for inhibition at lower and higher concentrations of the mercaptans in 0.5M acid and the neutral media at the shorter duration is given in table A| on page 102 for convenience. The discussion is therefore confined to these results only.

In the present investigation, it is observed that at a concentration of approximately 10⁻³M ethyl mercaptan, propyl mercaptan, butyl mercaptan and dodecyl mercaptan (with chain length having 2, 3, 4, and 12 carbon atoms respectively) give 97, 99, 96 and 99 % inhibition respectively in acetic acid; and their corresponding values in trichloroacetic acid are 99, 99, 99 and 99 %. In sodium chloride solution, on the other hand, these values are 36, 52, 61 and 98; and in artificial sea water the corresponding values are 60, 68, 73 and 99.

If the contention of Chi'ao and Mann is applicable to the present investigation we should get low inhibition in ethyl mercaptan compared to that with dodecyl mercaptan nearly at same concentration. The results suggest that this is not particularly so in the case of trichloroacetic acid. On the contrary, there is practically high acceleration of
### Table A

**Inhibition (%) in Various Media**

<table>
<thead>
<tr>
<th>Inhibitor</th>
<th>Concentration of Inhibitor (M)</th>
<th>Concentration of HCl (M)</th>
<th>Concentration of CH₃COOH</th>
<th>Concentration of CH₂ClCOOH</th>
<th>Concentration of CHCl₂COOH</th>
<th>Concentration of CCl₃COOH</th>
<th>Concentration of NaCl</th>
<th>Concentration of Sea Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethyl mercaptan</td>
<td>3.2x10⁻⁴</td>
<td>0.5</td>
<td>39</td>
<td>37</td>
<td>46</td>
<td>98</td>
<td>-91</td>
<td>-108</td>
</tr>
<tr>
<td></td>
<td>6.4x10⁻³</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>36</td>
<td>60</td>
</tr>
<tr>
<td>Propyl mercaptan</td>
<td>2.6x10⁻⁴</td>
<td>0.5</td>
<td>68</td>
<td>67</td>
<td>92</td>
<td>98</td>
<td>-10</td>
<td>-31</td>
</tr>
<tr>
<td></td>
<td>5.2x10⁻³</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>52</td>
<td>68</td>
</tr>
<tr>
<td>Butyl mercaptan</td>
<td>2.2x10⁻⁴</td>
<td>0.5</td>
<td>80</td>
<td>78</td>
<td>85</td>
<td>85</td>
<td>04</td>
<td>-18</td>
</tr>
<tr>
<td></td>
<td>4.4x10⁻³</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>61</td>
<td>73</td>
</tr>
<tr>
<td>Dodecyl mercaptan</td>
<td>4.9x10⁻⁵</td>
<td>0.5</td>
<td>42</td>
<td>39</td>
<td>33</td>
<td>09</td>
<td>-08</td>
<td>29</td>
</tr>
<tr>
<td></td>
<td>9.9x10⁻⁴</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>98</td>
<td>99</td>
</tr>
</tbody>
</table>

*Concentration units in Molar (M)*
corrosion in the neutral or nearly neutral (i.e. in 3% NaCl and artificial sea water) media at lower concentration. The acceleration suggests that not only there is little adsorption but there are some processes which induce the dissolution of metal.

According to Hackerman and Makrides (411) polarizability of sulphur atom is greater than that of nitrogen. Sulphur atom is less electronegative than nitrogen. Sulphur atom has two lone pairs of electrons while nitrogen has only one. Consequently, sulphur is better electron donor than nitrogen leading to greater tendency towards coordination. In other words, sulphur compounds are more strongly chemisorbed than the corresponding nitrogen compounds. They further state that within a series of sulphur compounds, for example mercaptans, the order of effectiveness as inhibitors should be methyl < ethyl < propyl < butyl < amyl.

As pointed out earlier, at a given concentration of a given mercaptan, there is wide variation in inhibition with the change in the nature of the medium (e.g. 98% in CCl₃COOH, 6% in HCl and acceleration up to 91% in 3% NaCl solution with ethyl mercaptan at a concentration of 3.2 x 10⁻⁴ M). While with dodecyl mercaptan, the inhibition is 98% or >98 in all the above three media at a concentration of 9.9 x 10⁻⁴ M. Thus only high polarizability does not seem to be
the criteria for inhibition. Interesting results have been obtained by Zucchi et al. (6) while working on inhibitory action of some organic sulphides on iron in \(1\text{N H}_2\text{SO}_4\) in 10 \% aqueous ethanol. The order of decreasing inhibition among the alkyl sulphides obtained by the above workers was as follows:

\[
\text{di-n-hexyl sulphide} \succ \text{di-n-butyl sulphide} \succ \text{di-n-octyl sulphide} \succ \text{di-ethyl sulphide} \succ \text{di-n-decyl sulphide}.
\]

In some cases (di-n-hexyl sulphide and di-n-decyl sulphide) they obtain acceleration of corrosion at lowest concentration (\(10^{-7} \text{M}\)). The stimulation at lower concentration has been presumed to be due to formation of organometallic compounds between sulphide molecules and ions of the metal under the conditions of low surface coverage. The inhibition at higher concentration has been explained as due to adsorption of the molecule on the metal surface through an unshared electron pair of the sulphur atom. Further, increase in the charge on the sulphur atom would increase the ability of the organic molecule to attach itself to the metal and will enhance the inhibitive power.

Confirmation of the importance of the electronic charge on the sulphur atom was obtained from a comparison of the inhibitory powers of alkyl sulphides where the inhibitory powers of alkyl sulphides were diethyl sulphide \(\prec\) di-butyl sulphide \(\prec\) di-hexyl sulphide. The reduction of
inhibitory power on going from di-n-hexyl to di-n-octyl and di-n-decyl sulphides cannot be interpreted, according to authors, on the basis of above hypothesis. This discrepancy has been attributed to the screening action of the carbon hydrocarbon chains, which are quite long in comparison with the sulphur atom and particularly with the position involved in the adsorption process. This explanation does not seem to hold for the results obtained in the present case with the mercaptans even when the adsorption, if it takes place, through the sulphur atom. The results of present investigation clearly show that dodecyl mercaptan invariably shows high inhibition in all the media at the given concentrations compared to the other mercaptans. The acceleration exhibited by mercaptans at lower concentrations is confined only to the neutral or nearly neutral media. Even in this case, the stimulation of corrosion decreases with increase in the length of chain.

According to Kolthoff et al. (415) reagents containing $-\text{SH}$ group are derivatives of $\text{H}_2\text{S}$. Such reagents can act in a way similar to $\text{H}_2\text{S}$ and can, therefore, form precipitates with metals like copper to form insoluble precipitate.

However, mercaptans cannot give rise to $\text{S}^{2-}$ ion like $\text{H}_2\text{S}$. The important property of mercaptans as reducing agents can lead to following possible mode of reactions:
The above reactions suggest that Cu^{++} ions are first reduced to Cu^{+} ions which may combine with the free mercaptans to produce the precipitates of Cu_{2}(RS)_{2}. A direct reaction can also be considered as a possibility, i.e.,

\[ 2RS^{-} + Cu^{++} \rightarrow Cu(RS)_{2} \]

Copper has a single 's' electron outside completed 'd' Shell\(^{(4f6)}\) (Configuration of copper: 1s\(^{2}\)2s\(^{2}\)2p\(^{6}\)3s\(^{2}\)3p\(^{6}\)d\(^{10}\)s\(^{1}\)). The filled 'd' shell is much less effective in shielding the s electron from the nuclear charge so that the first ionization potential of copper is higher than those of the alkalis. The second ionization potential of copper is much lower than those of alkali and account, in part, for the transition metal character shown by coloured ions and complexes in(II) oxidation states. Even in first oxidation state transition metal-like complexes, for example those with olefins, are formed. The Cu^{+} ion has the electronic structure 3d\(^{10}\) so that its compounds are diamagnetic and, except where colour results from the anion or charged transfer bands of the compounds are colourless. The relative
stabilities of Cu(I) and Cu(II) depend very strongly on the nature of anions or other ligand present. In aqueous solution the free Cu⁺ ion exists only in exceedingly low concentrations, and only cuprous compounds which are stable to H₂O are highly insoluble ones like CuCl. The dipositive state is the most important one for copper. Most cuprous compounds are readily oxidized to cupric compounds. There is a well defined aqueous chemistry of Cu²⁺ ion and a large number of salts of various anions exist in addition to wealth of complexes.

As pointed out earlier, sulphur has two lone pairs of electrons and has a tendency towards co-ordination. Consequently sulphur compounds can be chemisorbed by metal surface. Therefore sulphur atom in mercaptan should have ability for co-ordination and/or chemisorption.

It has been reported that mercaptans (thiols) by losing a proton co-ordinate more strongly especially to Pd(II), Pt(II), Cd(II) and Hg(II) [417].

Regarding the nature of reaction between mercaptans and copper little data are available. Judging from the above, it is most probable that for copper, it is Cu(II) state which is easily available in the solution for the reaction and for the mercaptans RS⁻ which is the species resulting from the dissociation of mercaptan RSH. The reaction between the above two species is likely to result in the formation of
the compound having the composition Cu(RS)_2.

In a qualitative analyses following experiments were carried out.

(I) 5 ml media (0.5 M HCl or 0.5 M CH_3COOH or 0.5 M CH_2ClCOOH or 0.5 M CHCl_2COOH or 0.5 M CCl_3COOH or 3 % NaCl or artificial seawater + 1 ml of 0.5 % CuCl_2 + 2 drops or 1.0 ml of 1 % reagent (mercaptan).

(II) 5 ml 0.5 M HCl (or 0.5 M CCl_3COOH) + 1ml 0.5 % CuCl_2 + 5 ml 1 % reagent (in fact in excess).

(III) Same experiment as in II above followed by filtration. The filtrate was evaporated in coming beakers and the residue treated with 2 ml of HNO_3 and evaporated to dryness. The residue was treated with ammonia.

The observations of the experiment were as below:

(I) In HCl, ethyl mercaptan gave no reaction. Propylmercaptan produced pale yellow precipitate while butyl and dodecyl mercaptans gave white precipitate. In rest of the media, ethyl mercaptan gave only a faint turbidity but no precipitate, the remaining compounds mostly giving pale yellow precipitates.
(II) Ethyl mercaptan gave slight turbidity in trichloroacetic acid, but gave no reaction in HCl. Propyl mercaptan gave milky solution with oily drops at the bottom in both the media while the remaining two mercaptans gave light yellow precipitate.

(III) The solution with reference to ethyl mercaptan and propyl mercaptan gave dark blue solution and that with butyl mercaptan light blue solution. With dodecyl mercaptan the solutions were mostly colourless.

Following qualitative inferences can be drawn from the above experiments.

Ethyl mercaptan has less tendency to form comparatively insoluble compound, in the media tested, with copper ions. Dodecyl mercaptan forms practically insoluble compound with copper ions. The remaining compounds exhibit intermediate behaviour. In other words, the solubility of the compound of a mercaptan with copper ions, if formed, decreases with increase in chain length.

The results show that inhibiting power of mercaptans in all the given media increases with increase in chain length of the mercaptans.

It has been reported that the solubility of an organic compound in water is decreased as its hydrophobic groups
(-CH₃, -C₂H₅ etc) increased in number or their molecular weight is increased (4-18). It may be expected that the intrinsic solubility of a metal complex precipitate will be decreased also as the molecular weight of the reagent is increased in this way. The effect is sometimes called weighting. Even though weighting criteria is more applicable to the chelates, it is quite probable that it is equally applicable in the case of alkyl mercaptan. Mercaptans, RSH, will dissociate in water as RS⁻ and H⁺. If the solution is made acidic its dissociation will be decreased leading to decrease in solubility. According to Kolthoff et al. (4-18) "as a rule, an increase in molecular weight brought about as described not only decreases the intrinsic solubility but also decreases the dissociation constant of the chelating agent HL and of the metal chelate ML so that the effect is doubly favoured". In the case of mercaptans therefore the dissociation of metal complex will decrease if the solution is made acidic. In turn, this may obviously lead to decrease in the solubility of the Cu-mercaptan complex.

From the above discussion one can arrive at the conclusion that mercaptans have a tendency to form an insoluble complex with copper ions. The solubility and the dissociation of the mercaptans as well as its metal complex will decrease with increase in the molecular weight of the mercaptan.
To explain the phenomenon of inhibition, mercaptans should be adsorbed, chemisorbed and/or form a mechanical barrier over the plate diminishing the easy access of the ions from the solution to the metal surface.

Even though there are some objections against the adsorption theory of inhibition, it would be wrong to deny the significance of physical adsorption as a first step to chemisorption or formation of protective films (4-19). The species that are adsorbed physically by means of electrostatic or weak van der Waals forces interact rapidly but are easily removed from the surface, for example by immersion of the metal in a solution free from the inhibitor. In order to see if the inhibition in the present investigation with mercaptans is simply due to physical adsorption following experiment was carried out.

The specimens were, at first, rotated (120 RPM having an velocity (angular) - meter per hour) for a duration of about 11/1 hours in a medium (3 % NaCl solution or 0.5 M HCl) in presence of an inhibitor, concentration of the inhibitor being that at which it gave maximum protection in the stagnant condition. The detailed procedure is given on page 8. Afterwards the treated specimens were taken out and then rotated in another medium in absence of the inhibitor (for 2 and 4 days in 3 % NaCl or 12 and 24 hours in 0.5 M HCl). The results with ethyl mercaptan there was acceleration (100 %) and with propyl mercaptan, butyl mercaptan and
dodecyl mercaptan there was inhibition up to 4%, 98% and 99% respectively in 0.5 M HCl; in 3% NaCl solution the corresponding inhibition was up to 86, 96, 97 and 99% respectively. These results show that inhibition, in NaCl, for all the mercaptans and for butyl and dodecyl mercaptans in HCl is not at least due to simple adsorption of the inhibitor by the metal surface. The plates, after the exposure, became somewhat red without formation of any visible film. The plates retained their luster. It is likely, therefore, that either the chemisorption of an inhibitor or formation of an extremely thin visible film of the inhibitor over the plate might be responsible for the inhibition. (An attempt was made to examine the pretreated plates by x-rays but the film, if formed, was, however, found to be transparent to the x-rays.)

As some of the pretreated plates gave excellent inhibition in 0.5 M hydrochloric acid, following experiment was carried out to test its resistance to vapours of hydrochloric acid.

Coupons immersed in waters containing 0.02% mercaptan for 2 days were exposed to hydrochloric acid vapour in a glass cupboard (details of the procedure is given on page 85). The experiment was concluded after nine days. Following observations were made.
Most of the plates were covered with a green powder at certain selected spots. At some places, heaps of green powder were also visible. After cleaning, the plates appeared to have been covered with a well-defined smooth, highly sticky reddish or reddish brown film which was difficultly removable even by rubbing vigorously with a rubber cork. In the case of dodecyl mercaptan, the major portion of the plate retained its original luster there being some localized spots or reddish patches.

In the places where lump of green powder accumulated, pits were observed below them after cleaning, the pits being filled with whitish mass which turn into green on keeping.

The above facts clearly show that the mercaptans have a distinct tendency to form a film. A question may be raised why no distinctly visible film is formed only when the treated specimens are exposed to the vapours of hydrochloric acid. A probable explanation is as follows:

When the plates are dipped in the solution containing inhibitor, following reactions may take place:

\[ \text{Cu} \rightarrow \text{Cu}^{++} + 2e^- \]

The Cu^{++} ions so formed at the anode will enter into the solution and migrate to the bulk of the solution, leading to little accumulation of the ions at the anode. This may when the plates are immersed in solution containing inhibitor, while a visible film is formed.
happen even if major portion of the surface of the specimen is covered with a thin film by chemisorption of the inhibitor. The Cu\(^{++}\) ions may then react with the inhibitor present in the solution to form either a soluble complex or to form a precipitate away from the surface of the plate. As an evidence of the above, in a number of cases particularly where very low inhibition was observed, accumulation of insoluble solid bluish or bluish green mass was observed at the bottom of the beaker in the stagnant condition.

These type of reactions, formation of precipitate or a soluble complex, lead to depletion of the concentration of the inhibitor. In turn, the tendency to form a thick film over the plate is minimised. Even in those cases where very high inhibition was observed the film was extremely thin and invisible. In the experiments where the solution was replaced by HCl and water vapours following processes can be expected, which can be considered responsible for formation of a visible film.

The plate is covered with a chemisorbed inhibitor. Hydrochloric acid and water vapours are condensed over the plate. Chloride ions of hydrochloric acid vapour will penetrate the chemisorbed layer (or a very thin film) at its weak spots and will form active anodes. Copper ions so formed at the anodes will migrate towards the cathodes i.e. the area
covered by the inhibitor but not penetrated by the chloride ions. Condensed hydrochloric acid vapours form a highly conducting layer over the plate particularly in the presence of moisture. This, therefore, facilitates the migration of Cu\(^{++}\) ions to the cathodic area. The continuous accumulation of Cu\(^{++}\) ions thus favours the following reaction in the forward direction: 

\[
\text{Cu}^{++} + 2\text{RS}^- \rightleftharpoons \text{Cu(RS)}_2
\]

As a result of continuous accumulation of copper ions and nearly completion of the reaction in the forward direction due to lack of diffusion or migration of copper ions, the surface develops a visible film. The anodes are visible over the plate in the form of pits filled with whitish material or greenish mass on standing. As stated earlier, the accumulation of greenish mass in the form of heaps over the plate support the above mechanism as they are the places where anodes are located. This green mass is probably hydrated CuCl\(_2\) as it dissolves very readily in water.

In the solution, formation of visible film is difficult because of high solubility of the copper salts formed due to corrosion. Thus the inhibition of copper by mercaptans in the media under consideration is principally due to formation of barrier film.

The support for film theory of inhibition comes from several authors\(^{420,421,422}\).
For mercaptans several authors have suggested that they are strongly adsorbed by copper ions possibly through sulphur which is responsible for inhibition (423, 424).

A specific suggestion that mercaptan can form a film on the copper surface in HCl acid is provided by Fuji and Kobayashi (172) wherein ethyl mercaptan dissolved in xylene formed a film on copper in 5% HCl.

A glance at the results show that the inhibitor efficiency of a given compound decreases with increase in the duration of the exposure. This is usually the case with most of the compounds, except with dodecyl mercaptan. Whenever the film formed over the copper plate is not quite uniform or if it is somewhat porous, it is bound to lead to decrease in inhibition with increase in duration. If the compound is able to form a very uniform, not penetrable film the protection may continue even if the film is thin.

The increase in inhibition with increase in duration observed in some cases (e.g. butyl mercaptan in 3% NaCl) may be due to blocking up or repair of the film at the weak spots, existing in the initial film.

In majority of the cases there is increase in inhibition, at a given concentration of the inhibitor, with increase in concentration of the media. It is observed, in general, that this tendency is more prevalent with mercaptan having longer chain and more so in trichloroacetic acid. The
possible effect that an acidic medium can have on mercaptan is to suppress the dissociation of its $H^+$ long of the later because they are also acids but weaker compared to that comprising the media.

The only explanation for an increase in inhibition with increase in concentration of the media can be attributed to the form of the inhibitor in which it is initially chemisorbed by the metal surface. An inhibitor of the type of mercaptan can undergo following types of reactions in an acid medium.

$$ RSH \rightleftharpoons RS^- + H^+ \quad (I) $$

$$ RSH + H^+ \rightarrow RSH_2^+ \quad (II) $$

Stronger the acid constituting the medium, the reaction (I) will be less favoured. Chances for reaction (II) are quite high because sulphur has two lone pairs of electrons which can attract proton from the medium. In other words, chemisorption of $RS^-$ may be more favoured in nearly neutral medium while chemisorption of $RSH$ or $RSH_2^+$ may be favoured in the acidic medium.

Several mechanisms have been proposed to account for inhibition by organic compounds in acidic media. Antropov (425) (who studied pyrimidine derivatives as inhibitors for acid corrosion of iron) the compounds are adsorbed on the metal surface in the form of cations and not as neutral
molecules. In this case, the nitrogen atom would be directed at the metallic surface. Some authors (426, 427) assumed the existence of cations in bulk of the acid solution and subsequent discharge with the formation of molecule at the interface with consequent chemisorption through an electron pair of the hetero-atom. For aliphatic compounds the scheme is

$$ R - NH_3^+ + e^- \rightarrow R NH_2 + \frac{1}{2}H_2 $$

The additive can act as transporter of proton catalyzing the electrode reaction.

According to Jofa (428) many organic compounds are of the cationic type and develop their inhibiting activity as cations, for example,

$$ R_3 - N + H^+ \rightarrow (R_3 NH)^+ $$

In the light of above explanation, the results of the present investigation can be explained as follows:

(i) $$ R - SH_2^+ + e^- \rightarrow RSH + \frac{1}{2}H_2 $$

(ii) $$ R - SH + H^+ \rightarrow (R - SH_2)^+ $$
At lower concentration of the acid there will be lower concentration of the inhibitor in the form of cation i.e., \( \text{RSH}_2^+ \). Its concentration would be increased at the higher concentration of the acid. These cations would be adsorbed either as such at the cathodic site or would be discharged with the formation of the molecules at the interface followed by chemisorption through a lone pair of sulphur. The results of polarization data reveal that inhibition is mostly governed by cathodic polarization with mercaptans.

Even though the results in acetic acid and trichloroacetic acid agree with the above, some discrepancy is observed in the case of dichloroacetic acid and, to a lesser extent, in monochloroacetic acid. It is difficult to account for this unusual behaviour. In the case of hydrochloric acid, the above explanation holds for butyl mercaptan and dodecyl mercaptan. For ethyl- and propyl-mercaptans some decrease is observed in HCl with its increase in strength. This discrepancy may be due to penetration of tiny chloride ions at the weak spots in the cover formed by the inhibitor.

With increase in concentration of an inhibitor mostly there is increase in inhibition in a given medium. This may apparently be due to the increased thickness of the protective film formed by the inhibitor over the metallic surface (429).
From polarization data it is apparent that inhibition is largely governed by the cathode polarization, greater the inhibition greater is the over all magnitude of the cathode polarization. In the case of sodium chloride solution and artificial sea water the magnitude of cathode polarization compared to the blank is fairly less which has been reflected in the lowering of the inhibition.

The change in steady state potential does not seem to reflect distinctly the nature of polarization. For example in the case of sodium chloride there is practically no shift of the steady state potential even on addition of dodecyl mercaptan even when it shows 99% inhibition.
3.2 SULPHIDES

RESULTS:

(A) Hydrochloric acid:

Weight loss results are given in table 3.9 on page 210.

The inhibitive power of diethyl sulphide and dibenzyl-disulphide increased with increase in their concentration, at both the duration in both the acid concentrations. The inhibitive power mostly decreased with increase in the duration except in dibenzyl disulphide in 1M acid. In both the sulphides, the inhibitive power usually decreased with increase in the concentration of the acid at a given duration. It is interesting to note that at lower concentrations diethyl sulphide increased corrosion.

Galvanostatic Measurements (Fig. 8 on page 258):

Incidently the pattern of polarisation of the copper plates in HCl solution containing sulphides the overall nature of polarisation was of the same pattern as the blank at higher current densities. At lower current densities however, the magnitude of polarisation was significantly high, more so when the plate was made cathode.

In the case of diethyl sulphide considerable anodic polarisation was observed at low current densities.
(B) Acetic acid and Chloro substituted acetic acid:

Diethyl sulphide:

Weight loss results are given in table 3.10 on page 211. The inhibitive power of diethyl sulphide increased with increase in its concentration showing mostly excellent inhibition in trichloroacetic acid. Its inhibitive power usually decreased with increase in duration except in trichloroacetic acid. At most of its concentrations there was an increase in its inhibition with increase in concentration of acid at a given duration; more so at higher concentration.

Dibenzyl disulphide:

Weight loss results are given in table 3.11 on page 212. At most of its concentrations the variation in the inhibitive power with increase in concentration of dibenzyl disulphide was not very significant, remaining mostly constant. In some cases however, a maximum in inhibition (for acetic acid) was observed at some intermediate concentration. The behaviour in inhibition with increase in duration is quite awkward, in 0.5 M trichloroacetic acid for example inhibition is excellent and constant at both the durations. The inhibitive power, at a given duration for a given acid increased with increase in chlorine content of the acid.
Galvanostatic Measurements (Fig. 9 on page 259):

Acetic acid:
Amongst two sulphides, dibenzyl disulphide showed fairly high cathodic polarisation compared to diethyl sulphide. Anodic polarisation showed same trend, the extent of polarisation being much less. It is worthwhile to note that inhibitive power of diethyl sulphide is higher \((i/2)\) than that of dibenzyl disulphide.

2-Monochloroacetic acid (Fig. 10 on page 260 b):
None of the sulphide showed any significant polarisation of the cathode. However some polarisation of the anode was observable.

Dichloroacetic acid (Fig. 11 on page 261):
Both sulphides showed a jump, when a current density was increased from \(5.0 \times 10^{-5}\) to \(10 \times 10^{-5}\) amp/cm\(^2\). In general, compared to polarisation of anode, the polarisation of cathode was more significant.

Trichloroacetic acid (Fig. 12 on page 262):
The behaviour of the diethyl sulphide and dibenzyl disulphide was practically of the same pattern as in dichloroacetic acid.
(c) 3% Sodium Chloride:
Weight loss results are given in table 3.12 on page 213.
Diethyl sulphide and dibenzyl disulphide can be considered as poor inhibitors, showing acceleration of corrosion at their lower concentrations; preferably at longer duration in 3% sodium chloride solution.

Galvanostatic Measurements (Fig. 13 on page 263):
The nature and extent of anode polarisation at all current densities and the cathode polarisation at lower current densities were of the same pattern as the blank in the case of diethyl sulphide and dibenzyl sulphide, both showing slightly high cathodic polarisation at higher current densities.

(D) Artificial Sea Water:
Weight loss results are given in table 3.13 on page 214.
Diethyl sulphide and dibenzyl disulphide afforded very poor inhibition in this media showing acceleration of corrosion at longer duration. At shorter duration diethyl sulphide show acceleration at its lowest concentration while dibenzyl disulphide showed slight acceleration (-3%) at its highest concentration.

Galvanostatic Measurements (Fig. 14 on page 264):
Diethyl sulphide and dibenzyl disulphide showed the same trend of cathode and anode polarisation in sea water as in sodium chloride except that overall magnitude of polarisation being somewhat low for the cathode at the higher current densities.
DISCUSSION

Diethyl sulphide and dibenzyl disulphide;

Only two sulphides have been studied viz., diethyl sulphide and dibenzyl disulphide. An abstract of the results of inhibition at lowest and highest concentration of these substances in different media are presented in Table A on page 126. The results show that there is an acceleration of corrosion in 0.5 M HCl and 3 % NaCl and also in artificial seawater at lower concentrations with diethyl sulphide while in the case of dibenzyl disulphide acceleration is observed in last two media only. Inhibition at a given concentration increases with increase in the strength of acid in the case of acetic acid and its chlorosubstituted derivatives at both the concentration of compounds except in diethyl sulphide at its higher concentration.

The sulphides can form cations in acidic media as follows:

\[
(C_2H_5)_2S + H^+ \rightarrow (C_2H_5)_2S^+ \\
C_6H_5 - CH_2 - S + 2H^+ \rightarrow C_6H_5 - CH_2 - SH^+ \\
C_6H_5 - CH_2 - S + 2H^+ \rightarrow C_6H_5 - CH_2 - SH^+ 
\]

If we assume the above to be correct, the results of inhibition can be explained, according to Jofa(428), if
### TABLE: A2

INHIBITION (%) IN VARIOUS MEDIA

<table>
<thead>
<tr>
<th>Inhibitor</th>
<th>Concentration of inhibitor (M)</th>
<th>0.5M HCl inhibition</th>
<th>0.5M CH3COOH inhibition</th>
<th>0.5M CH2ClCOOH inhibition</th>
<th>0.5M CCl2COOH inhibition</th>
<th>0.5M CCl3COOH inhibition</th>
<th>3% NaCl inhibition</th>
<th>Artificial Sea water inhibition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diethyl sulphide</td>
<td>2.2x10^-4</td>
<td>-100</td>
<td>1.1x10^-4</td>
<td>29</td>
<td>48</td>
<td>67</td>
<td>98</td>
<td>-52</td>
</tr>
<tr>
<td></td>
<td>4.4x10^-3</td>
<td>89</td>
<td>2.2x10^-3</td>
<td>99</td>
<td>81</td>
<td>82</td>
<td>99</td>
<td>24</td>
</tr>
<tr>
<td>Dibenzyl disulphide</td>
<td>6.1x10^-5</td>
<td>41</td>
<td>6.1x10^-5</td>
<td>56</td>
<td>81</td>
<td>91</td>
<td>99</td>
<td>-20</td>
</tr>
<tr>
<td></td>
<td>1.2x10^-3</td>
<td>79</td>
<td>1.2x10^-3</td>
<td>54</td>
<td>87</td>
<td>89</td>
<td>99</td>
<td>07</td>
</tr>
</tbody>
</table>
inhibition is supposed to be due to adsorption of cations of the organic molecules at the surface of the metal. In 5% sodium chloride and artificial sea water there are little chances to form cations of this type to any significant extent. Somewhat low inhibition at higher concentration of the substance in hydrochloric acid may be due to penetrating effect of very small chloride ions which can penetrate the film, if formed, by the inhibitor on the metal surface.

It would be interesting to consider the effect of increasing electron density over sulphur atoms. Ethyl radical is electron donating, while with benzyl radical the electron donating tendency will not be so prominent. In diethyl sulphide, two ethyl radicals are directly attached to sulphur atom which should increase the electron density over the sulphur atom. Therefore, compared to dibenzyl disulphide, diethyl sulphide should show quite high inhibition. The overall conclusion that can be drawn from the results is not in complete agreement with this.

When adsorbed or chemisorbed through sulphur, dibenzyl disulphide should show better coverage than diethyl sulphide and consequently better inhibition. It is apt to note that possibility of formation of multiple bonds can exist only with dibenzyl disulphide because two sulphur atoms are present in a molecule and that they are adjacent to each
other. It should therefore show better inhibition. The experimental results show that none of the above two factors seem to be quite fully justified.

Like mercaptans these two compounds also exhibited reddish brown film when the copper specimens, treated with 0.02 % solution of the compound, were exposed to hydrochloric acid vapours (Page 25). Therefore variations in inhibition may be essentially due to the nature of the film formed over the specimens by the inhibitors in a given medium.

According to Reid (430) following hydrolysis may take place with disulphides, producing the corresponding mercaptans and other products:

\[
2\text{RSSR} + 2\text{H}_2\text{O} \rightarrow 3\text{RSH} + \text{RSS}_2\text{H}
\]

\[
2\text{RSSR} + 3\text{H}_2\text{O} \rightarrow 5\text{RSH} + \text{RSS}_3\text{H}
\]

It is therefore, likely that in comparatively neutral media disulphide may undergo hydrolysis and lead to low inhibition.

The acceleration of corrosion in the chloride containing solutions at low concentration appears difficult to explain.

It is possible, however, that the organic sulphides may co-ordinate readily with copper ions (431) which can depolarize the anode leading to some acceleration.
It would be interesting to consider the extent of inhibition with increase in duration for a given acid strength and also for the effect of increasing strength of acid at a given duration for diethyl sulphide and dibenzyl disulphide. The results with diethyl sulphide show that there is a decrease in inhibition with increase in duration in almost all the media indicating that the protection afforded by the film over the plate decreases with time. This may, in part, be due to sluggishness of film repair at its weak spots with time. In the case of dibenzyl disulphide the behaviour of the film is not very uniform because it shows either decrease of inhibition (in HCl, monochloroacetic acid) or increase in inhibition or nearly constant inhibition with increase in duration.

In acetic acid and chlorosubstituted acetic acid the inhibitive power of diethyl sulphide increases, in general, with increase in concentration of acid at a given duration. While in rest of the media inhibition decreases. This indicates that the film formed in the organic acids studied above is better in concentrated acids compared to the dilute acid. In the solutions containing chloride ions the concentrations of penetrating ions (i.e., Cl' ions) increases with the strength of the media leading to depletion in inhibition.
The behaviour of the film produced by dibenzyl disulphide is not uniform for various media.

The results of polarization data show that inhibition exhibited by diethyl sulphide in HCl may be due to some polarization of the anode compared to the cathode polarization. In dichloro- and trichloro-acetic acid, of all the media, cathode is considerably polarized with both the sulphide indicating that cathode polarization is possibly the factor explaining inhibition.
3.3 **AZOLES:**

**RESULTS:**

(A) **Hydrochloric acid:**

Weight loss results are given in tables 3.14 to 3.17 on pages 215 to 218.

The inhibitive power of azoles increased with increase in concentration of the compound at both the duration in both the concentration of acid. In general the inhibitive power decreased at both the duration with increase in concentration of the acid except in the case of benzimidazole 2-mercaptobenzimidazole and benzotriazole where it increased. In 2-mercaptobenzothiazole the behaviour was slightly erratic. 2-mercaptobenzothiazole acted as an excellent inhibitor at its highest concentration in 0.5 M HCl.

**Thiazoline:**

2-Mercaptothiazoline:

Weight loss results are given in table 3.17 on page 218.

The inhibitive power of 2-mercaptothiazoline increased with increase in its concentration at both the duration in both the concentration of acid. It acted as good inhibitor at its highest concentration. Slight acceleration was observed at longer duration in 1 M HCl at lowest conc. of the compound.
Galvanostatic Measurements:
Azoles (Figs. 15 to 18 on pages 265 to 268): Compared to the blank it was only 2-mercaptopbenzothiazole, amongst the azoles, showed distinct cathodic and anodic polarisation at the higher current densities. Incidentally it was only 2-mercaptopbenzothiazole that gave highest inhibition.

2-Mercaptopthiazoline (Fig. 18 on page 268): 2-Mercaptopthiazoline has got nearly the same pattern of cathodic polarization at lower current densities as 2-mercaptopbenzothiazole. Compared to the anodic polarisation was not very significant.

(B) Acetic acid and Chlorosubstituted acetic acid: Benzimidazole weight loss results are given in table 3.13 on page 219. Benzimidazole was a poor inhibitor in chlorosubstituted acetic acid at both the duration at its higher concentrations. In acetic acid it gave poor inhibition at its lower concentrations while at its higher concentration good inhibition was observed at both the duration. The magnitude of its inhibition mostly increased with increase in its concentration. Its inhibitive power decreased with increase in the duration except in trichloroacetic acid where it increased somewhat in 0.1 M acid and behaved awkwardly in 0.5 M acid. In acetic acid its inhibition increased somewhat with increase in the acid concentration at a shorter duration, remaining nearly
constant at 10 days duration. In dichloroacetic acid it is increased at both durations. The pattern of inhibition with increase in the acid concentration in remaining two acids was somewhat erratic. Acceleration of the attack by the acid at lower concentration of the inhibitor was observed in dichloroacetic acid and in 0.5 M trichloroacetic acid.

2-Methylbenzimidazole:

Weight loss results are given in table 3.19 on page 220.

2-Methylbenzimidazole was a poor inhibitor at most of its concentrations. In acetic acid and dichloroacetic acid its inhibitive power increased with increase in its concentration. In remaining two acid solutions, maximum inhibition was observed at its intermediate concentration. Usually at higher concentration the inhibition was improved slightly with increase in the duration except in the case of 0.1 M acetic acid and 0.5 M monochloroacetic acid. Except in the cases of 0.5 acetic acid, 0.1 M monochloroacetic acid a fall in the inhibition was observed with increase in the concentration of the acid at both the durations.

There was a fall in the inhibition with increase in the concentration of the acid at its lower concentrations except in monochloro acetic acid where a rise was observed. At its higher concentrations there was some improvement in the inhibition particularly in trichloro acetic acid.
5-6 Dimethylbenzimidazole:

Weight loss results are given in table 3.20 on page 241.

5-6 Dimethylbenzimidazole was a poor inhibitor at its lower concentration in all the four acid solutions. Its inhibitive power increased with increase in its concentration except in trichloroacetic acid where a maximum inhibition was at some intermediate concentration. The effect of increase in duration was found erratic except in monochloroacetic acid where it decreased with increase in duration.

The inhibitive power decreased at a given duration with increase in the concentration of acetic acid, dichloroacetic acid (10 days) and trichloroacetic acid (2 days) solutions. In remaining acids there was either practically no change or some increase in the inhibition with increase in concentration of acid at a given duration.

2-Mercaptobenzimidazole:

Weight loss results are given in table 3.21 on page 242.

2-Mercaptobenzimidazole acted as an excellent inhibitor in 0.1 M acetic acid at all concentrations. It acted as a poor inhibitor at its lower concentrations in rest of the acid solutions. The inhibition increasing with increase in its concentrations.

Usually its inhibitive power decreased with increase in the duration except at higher concentrations in di- and trichloroacetic acids.
The behaviour of the compounds as inhibitor with increase in concentration of the acids at a given duration was somewhat awkward. However, in acetic acid the inhibitive power decreased at both the durations with increase in the concentration of the acid. It is interesting to note that there was tremendous increase in corrosion at lower concentration in 0.5 M dichloroacetic acid.

1,2,3 benzotriazole;

Weight loss results are given in table 3.22 on page 223. Benzotriazole acted as a poor inhibitor in monochloro and dichloroacetic acid except in 0.5 M dichloroacetic acid. Its inhibitive power decreased in acetic acid and increased in trichloroacetic acid with increase in the duration. In acetic acid the inhibitive power decreased while in trichloroacetic acid it increased with increase in concentration of acid at a given duration.

Sulphathiazole:

Weight loss results are given in table 3.23 on page 224. In mono- and dichloroacetic acids sulphathiazole acted as a poor inhibitor. In rest of the acids the inhibition increased with increase in concentration of sulphathiazole.

2-mercaptobenzothiazole:

Weight loss results are given in table 3.24 on page 225.
The inhibitive power of 2-mercaptobenzothiazole increased with increase in its concentration, showing mostly excellent inhibition at its higher concentrations. In 0.5 M acid solutions the inhibitive power decreased with increase in the duration particularly at its lower concentration. In 0.1 M acid solutions, however, there appeared no such regularity.

With increase in concentration of acid at a given duration, there appeared a fall in inhibition.

2-mercaptobenzothiazole:

Weight loss results are given in table 3.25 on page 226.

Inhibitive power of 2-mercaptobenzothiazole increased with increase in its concentration. Usually a fall in inhibition was observed at lower concentrations of the compound with increase in duration, the inhibition appearing nearly constant at its higher concentrations. At lower concentrations again the trend was to decrease inhibition at a given duration with increase in concentration of the acid. It exhibited excellent inhibition in trichloroacetic acid at higher concentrations.

Galvanostatic Measurements:

1. Acetic acid (Figs. 19-21 on pages 269-271):

None of the azoles showed significant cathode polarisation. At higher current densities, polarisation of
the cathode was less than that of the blank in some of the azoles, for example of dimethyl benzimidazole, 2-methyl-benzimidazole.

The polarisation of the anode was somewhat higher compared to the blank with all azoles. It is interesting to note that there is much variation in the inhibitive power of azoles in acetic acid solution.

2-mercaptothiazoline (Fig. 21 on page 271):

Behaviour of 2-mercaptothiazoline was very similar to that of azoles in general.

- Monoiodoacetic acid (Fig. 22 on page 272):

In this medium sulphathiazole showed both anodic and cathodic polarization, the extent of polarization is small. 2-mercaptobenzothiazole and 2-mercaptothiazoline showed same behaviour where both the electrodes are polarised to some extent.

- Dichloroacetic acid (Fig. 23 on page 273):

In the case of 2-mercaptobenzothiazole anode and cathode both were polarized compared to that in 0.5 M dichloroacetic acid. The extent of cathodic polarization is considerable. Same behaviour was observed in 2-mercaptothiazoline.

- Trichloroacetic acid (Fig. 24 on page 274):

Compared to blank the polarization of both anode and
cathode was distinctly high in with 2-mercaptobenzothiazole in 0.5 M trichloroacetic acid solution.

Same trend was observed in the case of 2-mercaptothiazoline.

(C) 3% Sodium chloride:

Weight loss results are given in tables 3.26 to 3.28 on pages 227 to 229.

Almost all azoles showed excellent inhibition at their higher concentrations, in 3% sodium chloride. The inhibitive power increased with increase in its concentration. Acceleration of corrosion was observed at the lower concentration of 5-6 dimethyl benzimidazole, 2-mercaptobenzimidazole in sodium chloride solution.

Of the four derivatives of benzimidazole, studied (only for sodium chloride solution) 2-hydroxymethyl benzimidazole showed excellent inhibition at its highest concentration; in general, there was acceleration of corrosion at their lower concentrations.

Galvanostatic Measurements:

Azoles (Figs. 25 to 28 on pages 275 to 278):

The overall nature of cathodic polarisation in the presence of most of azoles was of the same pattern as the blank. Of course the polarisation at the lower current densities was somewhat higher. It is interesting to note
that the polarization of the cathode in the presence of
2-mercaptobenzimidazole was lowered compared to the blank at
the intermediate current density. The polarization of the
anode was practically of the same pattern as that of blank
in all the cases.

2-mercaptothiazoline (Fig. 28 on page 218):

2-mercaptothiazoline showed nearly same pattern of
cathodic polarization except at the intermediate current
density. Anode polarization was quite significant at
all current densities.

(D) Artificial Sea Water:

Weight loss results are given in tables 3.29 and 3.30
on pages 230 to 231.

In artificial sea water almost all azoles gave excellent
inhibition. Inhibition increased with increase in
concentration of azoles at both the duration.

2-mercaptothiazoline afforded excellent inhibition at
its higher concentration but at lower concentration it
showed some acceleration at shorter duration.

Galvanostatic Measurement (Figs. 29 to 31 on pages 279 to 287):

Azoles also show somewhat higher cathode polarization
in artificial sea water at lower current densities, nearly
same trend prevailing at higher current density compared to
that in sodium chloride solution. The nature of anode
polarization remained practically same in sea water compared
to that in 3% sodium chloride solution.
**AZOLES**

Benzimidazole  
2-methylbenzimidazole  
5-6 dimethylbenzimidazole  

2-mercaptobenzimidazole  
Benzotriazole  
2-mercaptobenzothiazole  

Sulphathiazole  
2-hydroxyethylbenzimidazole  

2-hydroxyethylbenzimidazole  
2-hydroxybenzylbenzimidazole  

2-mercaptomethylbenzimidazole  
2-mercaptothiazoline
DISCUSSION

Benzimidazoles:

Eight benzimidazoles have been studied in 3% sodium chloride solution because of their high inhibitive tendency, at proper concentration in such system. Only four benzimidazoles have been studied in acid media because they exhibited poor inhibition in such media. In general, the inhibitive power increases with increase in concentration of benzimidazoles in a given medium. Most of these compounds, except the parent material i.e. benzimidazole, show an acceleration of corrosion at their lower concentration in 3% sodium chloride solution; this tendency is not so prevalent in the acid media. A summary of the results of inhibition at two concentrations (viz. lower and highest concentrations) of the compound in 0.5 M acid media and in 3% sodium chloride, and artificial sea water at the shorter duration (arbitrarily selected) is given in table A3 on page 142-143.

First we will consider the nature of inhibition in the acid media.

According to Mann(369) the cross sectional area of the ion projected down on the metal determines the effectiveness of the inhibitor. If benzimidazoles are arranged in the order of increasing coverage we should have roughly the following sequence:
## Table A3

### Inhibition (%) in Various Media

<table>
<thead>
<tr>
<th>Inhibitor</th>
<th>Concentration</th>
<th>0.5M HCl</th>
<th>0.5M CH₃COOH</th>
<th>0.5M CH₂ClCOOH</th>
<th>0.5M CHCl₂COOH</th>
<th>0.5M CCl₃COOH</th>
<th>3% NaCl</th>
<th>Artificial Sea water</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(M)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2 days</td>
<td>3 days</td>
<td>4 days</td>
<td>5 days</td>
<td>6 days</td>
<td>7 days</td>
<td>8 days</td>
<td>9 days</td>
</tr>
<tr>
<td>Benzimidazole</td>
<td>8.4 x 10⁻⁵</td>
<td>0.08</td>
<td>0.54</td>
<td>-0.05</td>
<td>0.39</td>
<td>-0.18</td>
<td>0.84</td>
<td>0.92</td>
</tr>
<tr>
<td></td>
<td>1.7 x 10⁻³</td>
<td>0.52</td>
<td>0.86</td>
<td>0.39</td>
<td>0.74</td>
<td>0.42</td>
<td>0.98</td>
<td>0.99</td>
</tr>
<tr>
<td>2-Methylbenzimidazole</td>
<td>7.6 x 10⁻⁵</td>
<td>0.12</td>
<td>-0.02</td>
<td>0.19</td>
<td>0.12</td>
<td>0.11</td>
<td>0.18</td>
<td>0.36</td>
</tr>
<tr>
<td></td>
<td>1.5 x 10⁻³</td>
<td>0.42</td>
<td>0.36</td>
<td>0.31</td>
<td>0.33</td>
<td>0.17</td>
<td>0.97</td>
<td>0.98</td>
</tr>
<tr>
<td>5-6 Dimethylbenzimidazole</td>
<td>6.8 x 10⁻⁵</td>
<td>-0.05</td>
<td>0.14</td>
<td>0.31</td>
<td>0.23</td>
<td>0.49</td>
<td>-0.08</td>
<td>0.17</td>
</tr>
<tr>
<td></td>
<td>1.4 x 10⁻³</td>
<td>0.19</td>
<td>0.33</td>
<td>0.41</td>
<td>0.44</td>
<td>0.45</td>
<td>0.98</td>
<td>0.99</td>
</tr>
<tr>
<td>2-Mercaptobenzimidazole</td>
<td>6.7 x 10⁻⁵</td>
<td>0.33</td>
<td>0.04</td>
<td>0.46</td>
<td>-0.21</td>
<td>0.42</td>
<td>-0.17</td>
<td>0.12</td>
</tr>
<tr>
<td></td>
<td>1.3 x 10⁻³</td>
<td>0.32</td>
<td>0.94</td>
<td>0.68</td>
<td>0.76</td>
<td>0.88</td>
<td>0.96</td>
<td>0.99</td>
</tr>
<tr>
<td>2-Hydroxymethylbenzimidazole</td>
<td>6.8 x 10⁻⁵</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-39</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>1.4 x 10⁻³</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>97</td>
<td>-</td>
</tr>
<tr>
<td>2-Hydroxyethylbenzimidazole</td>
<td>6.2 x 10⁻⁵</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-43</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>1.2 x 10⁻³</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>66</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td>7</td>
<td>8</td>
<td>9</td>
</tr>
<tr>
<td>---</td>
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<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>1-2-hydroxybenzylbenzimidazole</td>
<td>4.5x10^-5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-17</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>8.9x10^-4</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>92</td>
<td>-</td>
</tr>
<tr>
<td>2-mercaptomethylbenzimidazole</td>
<td>6.1x10^-5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-99</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>1.2x10^-3</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>32</td>
<td>-</td>
</tr>
<tr>
<td>Benzo triazole</td>
<td>8.4x10^-5</td>
<td>11</td>
<td>06</td>
<td>44</td>
<td>52</td>
<td>54</td>
<td>18</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>1.7x10^-3</td>
<td>27</td>
<td>62</td>
<td>25</td>
<td>90</td>
<td>94</td>
<td>99</td>
<td>99</td>
</tr>
<tr>
<td>Sulphathiazole</td>
<td>3.9x10^-5</td>
<td>15</td>
<td>31</td>
<td>39</td>
<td>41</td>
<td>42</td>
<td>-68</td>
<td>00</td>
</tr>
<tr>
<td></td>
<td>7.8x10^-4</td>
<td>53</td>
<td>84</td>
<td>48</td>
<td>44</td>
<td>49</td>
<td>93</td>
<td>96</td>
</tr>
<tr>
<td>2-mercaptobenzothiazole</td>
<td>6.0x10^-5</td>
<td>38</td>
<td>35</td>
<td>45</td>
<td>68</td>
<td>37</td>
<td>-23</td>
<td>34</td>
</tr>
<tr>
<td></td>
<td>1.2x10^-3</td>
<td>98</td>
<td>99</td>
<td>83</td>
<td>77</td>
<td>99</td>
<td>99</td>
<td>99</td>
</tr>
<tr>
<td>2-mercaptothiazoline</td>
<td>8.4x10^-4</td>
<td>44</td>
<td>41</td>
<td>27</td>
<td>45</td>
<td>05</td>
<td>36</td>
<td>-49</td>
</tr>
<tr>
<td></td>
<td>1.7x10^-3</td>
<td>91</td>
<td>86</td>
<td>92</td>
<td>94</td>
<td>99</td>
<td>97</td>
<td>99</td>
</tr>
</tbody>
</table>
The figures below the compounds represent the maximum inhibition exhibited by the compound at the given concentration in the media mentioned on the left. Considering the overall behaviour, the approximate sequence of increasing inhibition will be

\[
\begin{align*}
\text{Benzimidazole} & < 2\text{-mercaptobenzimidazole} < 2\text{-methylbenzimidazole} < 5\text{-6 dimethylbenzimidazole}
\end{align*}
\]

This order is not in agreement with the order mentioned above. In other words, the concept of area projected by inhibitor down on the metal does not seem to be applicable for benzimidazoles for copper in acid media studied in the present investigation. This conclusion is in agreement with the observations made by Podobaev (387) who compared the results of measurements of inhibition exhibited by derivatives of propargyl alcohol, H0-CH2-C=CH, keeping the triple bond in molecule unchanged.
Benzimidazoles are characterised by having two atoms of nitrogen in the smaller ring of the molecule. The benzimidazole system is highly aromatic. Therefore, if the electron density over the nitrogen atom is altered by introduction of substituents in the ring there should also be a corresponding change in the inhibitive power depending upon the electron donating or electron withdrawing capacity of the substituents. By increasing the electron density over nitrogen atom in pyridine, Ayers and Hackerman were able to get following sequence of increasing inhibition:

\[
\text{Pyridine} < 3\text{-picoline} < 2\text{-picoline} = 4\text{-picoline}
\]

If we consider the results of the present investigation we should get the following observations. If electron donating group, for example \(-\text{CH}_3\), is introduced in the imidazole ring there should be an increase in inhibition in a given media because of an increase of the electron density over the nitrogen atom. \(-\text{SH}\) group, like \(-\text{CH}_3\) group, is also electron donating group. Results of the present investigation do not agree with the above idea; the inhibitive powers of benzimidazole, 2-methylbenzimidazole and 2-mercapto benzimidazole nearly at same molarity \((1.5 \pm 0.2 \times 10^{-3})\) are respectively 52, 42 and 82 \% in 0.5 M \(\text{HCl}\) and 86, 36 and 94 \% in 0.5 M \(\text{CH}_3\text{COOH}\) while in 3 \% \(\text{NaCl}\) these values are 98, 97 and 96 \%.

The above facts suggest that, for benzimidazole, some other criteria is required to explain the extent of inhibition.
In a qualitative analysis it was observed that 2-mercaptobenzimidazole gave yellowish precipitate in HCl and in trichloroacetic acid and orange precipitate in rest of the three acids. The remaining compounds, however, gave no precipitates. 2-mercaptobenzimidazole invariably gave very high inhibition in all the five acid media while the remaining compounds gave comparatively low inhibition. It appears therefore that the products of corrosion form an insoluble compound only with 2-mercaptobenzimidazole, suggesting that film, if formed with 2-mercaptobenzimidazole on copper surface, is more resistant compared to that formed by the remaining three compounds.

An experiment similar to that in mercaptans was also carried out for benzimidazoles wherein the copper plates were dipped in 0.02% solutions of the inhibitor in distilled water for 3 days and subsequently exposed to hydrochloric acid vapours for 9 days. (Detailed description of experiment is given on page 115). It was observed that plates were covered with reddish or reddish brown, adherent film after cleaning. As described earlier (Page 115), the film may be due to accumulation of too much excess of copper ions to form a visible film. It is concluded, therefore, that the inhibition observed at higher concentration of the inhibitor in acid media may essentially be due to formation of a film over the metal surface. It is the nature
of this film which essentially decides the extent of inhibition. It is important to note that in some cases where poor inhibition was observed the plates immersed in acid media in the stagnant conditions exhibited an ash coloured loose film. The protective power of this film appears to be much less because of its high looseness and probably porous character.

We will now consider the results obtained in 3% sodium chloride solution. In sodium chloride system the order of increasing inhibitive power at higher concentrations of all eight benzimidazoles is given below:

2-mercaptomethylbenzimidazole ≤ 2-OH-ethylbenzimidazole ≤ 2-OH-benzylbenzimidazole ≤ 2-mercaptobenzimidazole ≤ 2-OH-methyl benzimidazole ≤ 2-methylbenzimidazole ≤ 5-6 dimethyl benzimidazole ≤ benzimidazole.

At lower concentration the order of increasing inhibitive power (or the order of decreasing acceleration) is as follows:

2-mercaptomethylbenzimidazole ≤ 2-OH-ethylbenzimidazole ≤ 2-OH-methyl benzimidazole ≤ 2-OH-benzylbenzimidazole = 2-mercaptobenzimidazole ≤ 5-6 dimethylbenzimidazole ≤ 2-methylbenzimidazole ≤ benzimidazole.

In general, both the orders can be considered to be roughly similar.

In a qualitative analysis it was observed that with copper ions in 3% sodium chloride solution benzimidazole
gave red precipitate, 2-methylbenzimidazole gave white precipitate, 5-6 dimethylbenzimidazole gave dirty green precipitate and 2-mercaptobenzimidazole gave yellow precipitate, while 2-hydroxymethylbenzimidazole, 2-hydroxyethylbenzimidazole and 2-hydroxybenzylbenzimidazole gave yellowish green, green and gray green precipitate respectively.

All these compounds except 2-hydroxyethylbenzimidazole and 2-mercaptomethylbenzimidazole gave high inhibition (more than 90%) while the later two compounds gave comparatively low inhibition.

These substances, therefore, appear to have a tendency to form insoluble compounds with copper ions, preferably complex or co-ordination compounds.

Imidazole is a basic compound having a tendency to form salts with acids. This basic property results from the ability of ring nitrogen to accept the proton.

\[
\begin{align*}
&\text{Imidazole} + \text{H}^+ &\leftrightarrow &\text{Imidazolium}^+ \\
&\text{N} & &\text{N}^+ \\
&\text{CH} & &\text{NH} \\
&\text{H} & &\text{H}
\end{align*}
\]

A co-ordination compound can result between copper ions and benzimidazole in which a lone pair of electrons
from one nitrogen atom and the covalent link formed by a replacement of hydrogen atom from -NH group of benzimidazole. This type of complexation has been confirmed by Prajapati(15).

Several workers have reported the preparation and infrared spectra of metal chelates of imidazole derivatives(433, 434).

A typical mode of co-ordination of benzimidazole with copper can be represented as below where it is presumed that two molecules of benzimidazole combine with one copper ion:

According to Goodame and Haines(435) the actual composition of the complex is \[
\left[ \text{Cu(BMA)}_4 \right]^{-}\]
in which weak coordination of anion is suggested. This type of co-ordination facility is available in all the derivatives of benzimidazole. It is important to note that introduction of various groups in 2-position of benzimidazole may change the electron density over the nitrogen atoms responsible for
co-ordination. It is further likely that these groups may act as steric hinderence to co-ordination. In addition to the above, the stability of the metal ligand bond will be dependent upon the nature of 2-substituted group of benzimidazole. We will discuss these factors in the light of the results obtained for inhibition of corrosion in 3% sodium chloride system.

Introduction of methyl radical at the 2-position will increase the electron density over nitrogen atom. The inhibition exhibited by 2-methylbenzimidazole is nearly same as that of benzimidazole suggesting that change in electron density does not substantially alter the inhibitive power of benzimidazole.

Introduction of bulky groups at 2-position as in 2-hydroxybenzylbenzimidazole or 2-hydroxymethylbenzimidazole does not also seem to profoundly change the inhibitive power of benzimidazole as their inhibitive powers are 92 and 97% respectively. Thus steric hinderence does not seem to play a prominent role.

We will now consider the effect of stability of the complex formed between benzimidazole derivatives and copper ions.

Vermilyea et al. (436) report that the inhibitor effectiveness is the function of the acidity of the inhibitor,
The acidity being expressed as proton level \( J \)

\[ J = 0.059 \left( \frac{1.744}{K_a} - \log K_a \right) \]

where \( K_a \) is the acid dissociation constant of the inhibitor.

All the strong inhibitors have \( J \) values between -0.44 to -0.69, while none of the inhibitors with very low or very high \( J \) value are effective. The above limits of \( J \) correspond to \( -\log K_a \) (i.e. \( pK_a \)) values between 5.7 to 9.9. The average \( pK_a \) values \( (pK_{av}) \) of the inhibitor, along with \( pK_1 \) and \( pK_2 \) as available from literature are given in the following table (437, 15, 438).

**TABLE :** \( pK \) values (at 25\( ^\circ \) C) and Inhibition

<table>
<thead>
<tr>
<th>Inhibitor</th>
<th>( pK_1 )</th>
<th>( pK_2 )</th>
<th>( pK_{av} )</th>
<th>Maximum inhibition (%) in 3 % NaCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzimidazole</td>
<td>5.532</td>
<td>12.3</td>
<td>8.9</td>
<td>98</td>
</tr>
<tr>
<td>2-methyl</td>
<td>6.15</td>
<td>11.48</td>
<td>8.8</td>
<td>97</td>
</tr>
<tr>
<td>5-6 dimethyl</td>
<td>5.96</td>
<td>12.52</td>
<td>9.2</td>
<td>98</td>
</tr>
<tr>
<td>2-OH methyl</td>
<td>5.40</td>
<td>11.55</td>
<td>8.5</td>
<td>97</td>
</tr>
<tr>
<td>2-OH ethyl</td>
<td>5.26</td>
<td>—</td>
<td>—</td>
<td>66</td>
</tr>
<tr>
<td>2-OH benzyl</td>
<td>4.74</td>
<td>—</td>
<td>—</td>
<td>92</td>
</tr>
<tr>
<td>2-mercapto</td>
<td>9.2</td>
<td>11.0</td>
<td>10.1</td>
<td>96</td>
</tr>
</tbody>
</table>

From the above table we can say that except for the two inhibitors whose values of \( pK_2 \) are not available from the literature, the average value of \( pK_a \) vary between 8.5 and
10.1, i.e. they are nearer to the highest values $pK_{av}$ fixed for strong inhibition. Even in the two cases where values of $pK_2$ are not available from literature the values of $pK_{av}$ will be nearer to the higher values observed for the inhibitors because the basic strength of compounds having 2-ethyl, 2-isopropyl groups etc., exhibit the same strength as does the 2-methyl derivatives(439).

Eventhough the majority of the results at higher concentration agree with the views expressed by Vermelia et al., it completely fails at lower concentration of the inhibitor where benzimidazole gives 84% inhibition while 2-hydroxymethylbenzimidazole shows acceleration upto 39%. 2-OH ethyl benzimidazole with a likely value of $pK_{av}$ in the range 5.7 to 9.9 gives much less inhibition compared to the rest.

It is reported that greater the basic strength of a ligand (i.e. greater the value of $pK_a$) greater will be the stability constant of the metal complexes and hence greater would be the inhibitor effectiveness(440).

Table below gives the stability constant of copper complexes between Cu(II) ion and the compound acting as an inhibitor(15, 438).
TABLE: Stability Constants and Inhibition

<table>
<thead>
<tr>
<th>Inhibitor</th>
<th>Log stability constant</th>
<th>% inhibition in 3 % NaCl</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-mercaptobenzothiazole</td>
<td>12.8</td>
<td>99</td>
</tr>
<tr>
<td>2-mercaptobenzimidazole</td>
<td>11.9</td>
<td>96</td>
</tr>
<tr>
<td>Benzotriazole</td>
<td>8.7</td>
<td>99</td>
</tr>
<tr>
<td>2-hydroxymethylbenzimidazole</td>
<td>15.54</td>
<td>97</td>
</tr>
<tr>
<td>2-hydroxyethylbenzimidazole</td>
<td>16.2</td>
<td>66</td>
</tr>
</tbody>
</table>

The above data at a first glance suggest that there is no well established relation between the stability of the complex of the compound with Cu(II) ions in 3 % sodium chloride solution.

All the above factors like electron density over nitrogen atom and stability of metal complex cannot account fully for the inhibitive powers exhibited by benzimidazole and its derivatives in the neutral solutions.

As stated earlier (Page 146), benzimidazole and its derivatives have a tendency to form a film over the copper plate. Perhaps it is the nature of the film which may account for the variation in inhibitive powers in sodium chloride system.

As it is in the case of benzotriazole it is likely that benzimidazole may form a polymer film particularly in the case of neutral solution. Evidences indicating formation of a simple but porous film by benzimidazole in slight acid
solution (pH 5.0) giving poor inhibition and strongly adherent polymeric film in neutral solution (3% sodium chloride) giving rise to high inhibition are available. According to Tobe et al. (237) benzotriazole gives better inhibition than benzimidazole in acidic solutions because former forms a protective film of copper complex polymer in acid solutions whereas latter does not.

2-mercaptobenzimidazole, for example, can form a copper complex as follows:

\[
\begin{array}{c}
\text{\textbf{N}} \\
\text{C-S-Cu-S-C}
\end{array}
\]

The polymeric chain could be built up in the complex film formed on the metal surface by alternate copper atoms and 2-mercaptobenzimidazole (2MBMA) molecule as below:

\[
\begin{array}{c}
\text{\textbf{N}} \\
\text{Cu-S-C}
\end{array}
\]

\[
\begin{array}{c}
\text{\textbf{N}} \\
\text{Cu-S-C}
\end{array}
\]
It is also possible that if the molecule is firmly bonded to the surface by several bonds, instead of simple bonding, it may give better inhibition. For example, 2-mercaptobenzothiazole may be attached to the surface of the copper, which is partly oxidized, as follows(441):
The nature of inhibition under such conditions would be dependent upon structure of the oxide film and geometry of the attached molecule. Compounds like 2-mercaptobenzimidazole, 2-hydroxymethylbenzimidazole can combine with partially oxidized copper in a manner depicted above. The strength of such bonds may decide the extent of inhibition.

As an evidence to the above, following experiment was carried out. Air was continuously passed through the solution containing 3% NaCl and inhibitor at a 75°C (detail on page 84). It was observed that with most of the benzimidazoles the inhibitive power was high and same as in the stagnant condition. The inhibitive power of 2-hydroxyethylbenzimidazole significantly increased (89%) in this experiment compared to that in the stagnant condition (66%).

This may be explained on the basis that at high temperature in the presence of air the oxidation of the copper surface may have been accelerated.

Low inhibition exhibited by 2-mercaptomethylbenzimidazole is primarily due to its high instability in air. The compound undergoes decomposition and the solution, if kept overnight, becomes dark. Even during the experiment on inhibition, the solution becomes dark on standing.

It is pertinent to note at this stage that fairly good inhibition is exhibited by benzimidazole and 5-6 dimethyl-
benzimidazole at the higher concentrations in 0.5 M CH$_3$COOH, even when none of these two compounds gives insoluble precipitate with copper ions in this media. It is apt to note that these compounds give 98% inhibition in 3% NaCl and quite poor inhibition in comparatively stronger acid solutions, e.g. 0.5 M HCl or 0.5 M CCl$_3$COOH. The intermediate behaviour of these compounds in acetic acid medium can be assigned to the intermediate solubility of the copper complexes in the medium. In other words, in sodium chloride we get precipitates with copper ions while in stronger acid media we get soluble compound and in acetic acid medium the compound may be just soluble.

Derivatives of benzimidazoles, particularly those containing -SH or -OH group in 2-position lead to acceleration of corrosion at their lower concentration in 3% sodium chloride solution. As benzimidazole molecule is chemically stable, resistant to drastic treatment with acids as well as bases and it is not readily attacked by oxidizing agent(442), it can be presumed, therefore, that it is only that portion of the molecules of benzimidazole derivatives which is not incorporated in either of the rings that is likely to undergo oxidation or reduction. It is equivalent to saying that -OH or -SH group present in the side chain that is likely to undergo some reactions at the local cathodes or anodes. It is likely that these groups may
undergo oxidation at the local anodes and may cause acceleration of corrosion. SH groups are powerful reducing agents. They can be oxidized even by aqueous solutions of cupric chloride, the reaction being formation of disulphide (\(M^{+3}\)).

\[
2RSH + 2CuCl_2 \rightarrow R-S-S-R + 2CuCl + 2HCl
\]

The above reaction may lead to depolarization, if any, of local anodes leading to acceleration. In a similar way, OH groups can also undergo some oxidation, though to a lesser extent than the SH group. This is evident from 99% acceleration by 2-mercaptop benzimidazole at a lower concentration and 2-hydroxymethylbenzimidazole which gives 39% acceleration nearly at the same concentration. The results reveal that it is only at the lower concentration at which acceleration of corrosion takes place; while inhibition is observed at the higher concentration. This type of behaviour appear to be similar to that with thiourea which is discussed on page 195.

Benzimidazole, 2-mercaptop benzimidazole, 2-methylbenzimidazole and 5-6 dimethyl benzimidazole show increase in inhibition with increase in concentration in a given medium. This may probably be due to formation of increasingly compact film over the metal surface with increase in the concentration of the substance. Except
benzimidazole, the compactness of the film appears to increase somewhat with increase in the duration, except in 1 M hydrochloric acid with 2-methylbenzimidazole and 5-6 dimethylbenzimidazole.

In acetic acid it can be said, in general, that inhibition decreases with increase in concentration of acid at a given duration while in chloro-substituted acetic acid, there is no definite trend regarding increase of decrease in inhibition with the increasing concentration of acid at a given duration.

In general, regarding the extent of inhibition with increase in duration in a given medium, it can be said that in monochloroacetic acid the inhibition mostly tends to decrease while in rest of the acids there appears to be no definite regularity. It can be presumed that the nature and extent of inhibition which can be said to depend upon the nature and compactness of the film is difficult to define for acetic acid and chloro-substituted acetic acids. This may probably due to relative tendencies of the acetate ion and the species or the form of inhibitor molecule in the acetate medium to combine with the copper ion, it being reported that the acetate anion $\text{[AC]^-}$ coordinate to a metal in one of the following Schemes:\text{[4H]}.
Usual bridging structure (i.e. structure III) of the acetate ion was found in Cu₂(AC)₄·2H₂O (M+5). In solution, however, it is difficult to assign a specific structure. Due to this discrepancy regarding assignment of the exact nature of the coordination compound or complex formed by acetate ions with copper ions and that produced by the inhibitors with copper ions and their relative stability or formation etc., it is apparently difficult to describe the variation of inhibition of these compounds in acetic and its chlorosubstituted acids as all these compounds, if formed, are quite soluble in these acid media.

In the case of sodium chloride solution the inhibitive power of a given substance, at a given concentration remains either nearly constant or improved a little with increase in the duration. While in the case of sea water it
remains practically constant except in sulphathiazole and 2-mercaptotothiazoline. This indicates that practically there is little weakening of the inhibitor film formed over the surface once formed.

**Benzotriazole:**

The inhibitive power of benzotriazole increases with increase in its concentration in all the seven media. Its inhibitive power is extremely less in acetic acid, hydrochloric acid and 3% sodium chloride at its low concentration \((8.4 \times 10^{-5} \text{M})\). However, the inhibitive power increases with increase in the chlorine content of acetic acid. At higher concentration \((1.7 \times 10^{-3} \text{M})\), again, its inhibitive power is much low in hydrochloric acid and monochloro acetic acid showing nearly excellent inhibition in trichloroacetic acid and in neutral media.

There is no significant cathode or anode polarization of copper electrode in any of the acidic or neutral media in presence of benzotriazole.

Several theories have been proposed to account for the inhibitory action of benzotriazole. According to Tadashi (446) benzotriazole undergoes following ionization reaction in solution:

\[
\text{C}_6\text{H}_4\text{N}_2\text{NH} \rightleftharpoons \text{C}_6\text{H}_4\text{N}_2\text{N}^- + \text{H}^+
\]

It is the anion which is thought to be present in most
solutions. The high inhibition, e.g. 99 and 96 % respectively in 3 % NaCl and trichloro acetic acid cannot be explained on the basis of the above assumptions because in trichloroacetic acid the effective concentration of the anion will be negligible compared to that in sodium chloride solution. The suggestion(447) that benzotriazole exists as a neutral molecule can also be ruled out from the above argument because the experimental evidences are not in confirmation with this(307).

Studies on infrared and ultraviolet spectra of copper treated with benzotriazole led Morito et al.(235) to the conclusion that in 3 % sodium chloride solution a film is formed which consists of benzotriazole cuprous salt formed by the substitution of a H atom in the NH group with Cu(I) ion. The above authors, in an another communication(448), suggest that in sodium chloride solution following reaction takes place to the right hand side, while in acid media the reaction goes to the left side:

\[ \text{Cu}^+ + \text{BTA} \rightleftharpoons \text{BTA} - \text{Cu(I)} + \text{H}^+ \]

They further state that in deaerated solution BTA - Cu(I) film was formed on copper but at pH > 3 the film is not formed because of reversal of above equation. In otherwords, the corrosion reaction of copper was much more markedly inhibited in nearly neutral solution than in acid solution.
The data in present investigation again do not confirm the above mechanism as an inhibition up to 96% is observed in trichloroacetic acid.

In a qualitative analysis in which inhibitor solution was added to various media containing low concentration of copper ions (1 ml of 0.5% CuCl₂ in 0.5 M concentration of medium) it was observed that green precipitates were formed only in 3% NaCl and in artificial sea water while in the rest of the acidic media no precipitate was observed, the solution, however, turning green in acetic acid. In nearly neutral solutions well defined precipitates of Cu(C₆H₄N₃)₂ is formed by Cu(II) ions (449). This indicates that the compound of copper(II) with benzo triazole is formed in an insoluble form only in the neutral media. As indicated earlier, very high inhibition (99%) in neutral media may be definitely attributed to the formation of a film. In rest of the media even though no precipitate is formed there are chances that a compound may be formed at the surface of the film after chemisorption of the inhibitor through the nitrogen atom by the copper surface.

In an experiment similar to that described earlier (page 65) the specimen treated with water containing inhibitor and subsequently exposed to HCl vapour it was observed that a brownish-red, strongly adherent film was observed on the plates after cleaning.
In acid media, there seems to form extremely thin invisible film which can account for the inhibition which is of the order of 96%. Several authors (204, 229, 277) suggested that a film in which benzotriazole is a constituent along with copper is formed over the surface which protects the underneath metal. According to Cotton (277) a strong bond is formed between benzotriazole and copper surface which is similar to chemical combination occurring in the insoluble complexes with copper ions. He considers that the liable hydrogen and two nitrogen atoms are required for the bonding between copper and benzotriazole. Infrared spectroscopy data indicated that cuprous-benzotriazole was polymeric, so a linear structure is suggested. In this structure, the copper is bonded using sp orbital with a covalent bond formed by the replacement of the hydrogen atom from the NH group and a co-ordinate bond with a lone pair of electrons from one of the nitrogen atoms. A chain is therefore built up of alternate benzotriazole molecules and copper atoms as shown below.
Morito et al. (235) studied the infrared spectra of Cu(I) benzotriazole and concluded that NH frequencies at 3400 cm\(^{-1}\) disappear due to complexation through the NH group and one of the nitrogen atoms of the ring. The complex formed by cupric ions with benzotriazole was insoluble and uncharged. The metal ligand ratio is 1:2.

A probable mode of protection is also by forming a possible polymeric compound with copper(II) and benzotriazole over the copper surface (15). A possible structure can be represented as follows:
The low inhibition in HCl compared to that in sodium chloride by benzotriazole even at high concentration can be attributed mainly to some reduction of benzotriazole in HCl. The reduction undergone by benzotriazole in acid solution has been proposed as below(450):
In hydrochloric acid, chloride ions are highly penetrating which may penetrate the film formed by the chemisorbed benzotriazole which might have escaped the reduction. In trichloro acetic acid, however, the reduction is possible, but due to the absence of penetrating chloride ions, good inhibition is observed. It is difficult, however, to explain high inhibition (54%) in trichloroacetic acid compared to that in 3% sodium chloride (18%) at a low concentration (3.4 x 10^{-5} M) of benzotriazole.

**Thiazoles**

The inhibitive power of sulphathiazole is of high magnitude only in the neutral or nearly neutral solutions. In chlorosubstituted acetic acid and HCl its inhibitive power is much lower, the inhibitive power in CH$_3$COOH being intermediate. The inhibitive power thus appears to depend
upon the acidity of the solution, higher the acidity, in general, lower the inhibition.

In a qualitative analysis it was observed that distinct precipitates were obtained in neutral solution and no precipitates in hydrochloric acid and in remaining acid solutions only faint turbidity was obtained. This indicates that the precipitates of sulpathiazole with Cu(II) ions are less soluble in neutral media and apparently more soluble in acid media. Sulpathiazole contains -NH₂ group which increases its solubility in the acid media and consequently that of its copper complex. The probable structure of Cu(II) sulpathiazole can be represented as below:

\[
\begin{array}{c}
\text{HC} - \text{N} \\
\text{HC} \quad \text{C} - \text{NO}_2 \quad \text{SO}_2 \\
\text{S} \quad \ldots \quad \text{Cu/2}
\end{array}
\]

2-mercaptobenzothiazole gives excellent protection in all the acid media and in 3% sodium chloride and artificial sea water. It is thought that an extremely adherent film is formed between copper ions and 2-MBT at the surface of the metal. This film prevents any copper entering into solution.

In aqueous solution, it ionizes as follows:
The ionic species form insoluble salts with metal ions such as copper, protective coating consisting mainly of the insoluble copper salts of 2-mercaptobenzothiazole. However, a more plausible structure for the protective coating would take into account the fact that 2-mercaptobenzothiazole possesses not only an ionized sulphur group but also a ring nitrogen and ring sulphur, both capable of forming coordinate bonds with the metal or metal ion. A possible mode of attachment of 2-mercaptobenzothiazole with the partially oxidized surface of copper can be depicted as below:**
The precise geometry would depend upon the structure of the oxide film and the geometry of the 2-mercaptobenzothiazole molecule. The molecule under such condition would be firmly bonded to the surface by several bonds. A similar type of bonding to the unoxidized metal surface would also be possible.

The infrared spectra of Cu(2-MBT)$_2$ complex indicates the covalent bonding between copper ion and 2-MBT through the sulphur from -SH group and co-ordinate link through ring sulphur, which is situated in the thiazole ring. The structure of the Cu(2-MBT)$_2$ complex is shown below:

![Diagram of Cu(2-MBT)$_2$ complex]

It may also be pointed out that the film formed on the copper plate consists of Cu(II) (2-MBT)$_2$ or Cu(I)-2MBT. Thermodynamically the favourable reaction is Cu(II). Therefore, the inhibition afforded by 2-MBT may be principally due to Cu(MBT)$_2$ complex. It is also suggested
that the blockening effect of the film is due to polymeric form of the complex which can act as physical barrier as well as interphase between the metal plate and corroding medium. The polymeric form of the complex film can be represented as below:

\[
\begin{align*}
\text{Cu} - \ 2\text{-MBT} & \quad \text{Cu} \quad 2\text{MBT} - \text{Cu} \\
\text{Cu} - 2\text{MBT} & \quad \text{Cu} \quad 2\text{MBT} - \text{Cu}
\end{align*}
\]

2-Mercaptothiazoline

2-Mercaptothiazoline is found effective in hydrochloric acid and acetic acid. It is an excellent inhibitor in trichloroacetic acid but is less effective in monochloro- and dichloroacetic acid solutions. In salt solution and artificial sea water it gives excellent inhibition. In a qualitative experiment it was observed that 2-mercaptotiazoline gave pale yellow precipitates with copper ions in acetic acid and chlorosubstituted acetic acid solutions. 2-mercaptotiazoline has a chelate forming tendency with copper ions. The probable copper-mercaptotiazoline chelate which forms a protective layer on the metal surface, has the following structure:
A glance at the structures of 2-mercaptobenzothiazole and 2-mercaptothiazoline will show that the mode of complex formation of both these substances will be similar with Cu(II) ions.

Even though both 2-mercaptobenzothiazole and 2-mercaptothiazoline give nearly excellent inhibition in most of the media, 2-mercaptobenzothiazole can be considered somewhat superior because it gives excellent inhibition comparatively at the lower concentration. This type of low inhibitive power of 2-mercaptothiazoline may partially be due to somewhat higher solubility of the substance itself, and the nature of the film formed over the copper surface when in solution. With 2-mercaptobenzothiazole the film formed over copper plate in acid or neutral media is quite thin, sticky and smooth while that with 2-mercaptothiazoline the film is neither very smooth nor thin particularly in hydrochloric acid solution. In a qualitative experiment it was observed that in hydrochloric acid and neutral media 2-mercaptothiazoline gave yellow precipitate while in acetic acid and its chlorosubstituted acids mostly yellowish colloidal solutions were obtained with Cu(II)
ions. On the contrary 2-mercaptobenzothiazole gave distinct precipitate.

**Galvanostatic Measurements**

The results of galvanostatic measurements show that

(i) In 0.5 M HCl only 2-mercaptobenzothiazole polarises cathode slightly. The steady state potential evidently becomes more negative in presence of the above compound.

(ii) In 0.5 M acetic acid some anodic polarization is observed compared to the cathode polarization, the polarization being somewhat awkward. The polarization is somewhat more apparent at lower current densities.

(iii) In 0.5 M monochloracetic acid, cathode is significantly polarized both in presence and absence of inhibitors. Anode polarization though slight is evident compared to the cathode polarization. 2-mercaptobenzothiazole polarises both cathode and anode particularly at higher current densities.

(iv) In 3% sodium chloride solution only 2-mercaptothiazoline shows some significant anode polarization. It also makes steady state potential distinctly more negative.

(v) In artificial sea water polarization data do not reflect much significant behaviour.
The behaviour of benzotriazole towards cathodic or anodic reaction is subject to some controversy. According to Cotton (277) it significantly interferes with cathodic oxygen reaction. It does not greatly affect cathodic hydrogen evolution but its main effect is upon the anodic reaction. While according to Morito et al. (448) benzotriazole inhibited mainly the anodic reaction, and in the cathodic regions, the retardation of hydrogen evolution was more effective than that of oxygen reaction. Dugdale and Cotton (233) showed that significant differences in cathodic behaviour were found between treated and untreated samples, with smaller differences in anodic behaviour. According to Bonora et al. (229) benzotriazole at a concentration of $10^{-2}$ M for copper in 0.1 N NaCl solution showed a marked inhibition effect both on anode and cathode polarization. According to Poling (420) anodic processes are inhibited by high electrolyte resistance of the thick surface film formed by benzotriazole. He further deduced that as well as acting as a physical barrier at the anode, the film inhibited the cathodic hydrogen evolution much more effectively than the oxygen reduction reaction. The present results show that in nearly neutral solution, for example artificial sea water, some cathode polarization and indication of film formation is observed with benzotriazole.
In 0.5 M hydrochloric acid as well as in 0.5 M acetic acid neither cathode nor anode is significantly polarized. Benzotriazole does not also seem to shift even steady state potential in neutral or acidic media.

2-mercaptobenzothiazole is generally believed to be an anodic inhibitor for copper (391). According to Bosworts (209) addition of 2-mercaptobenzothiazole in acetic acid produces a high cathodic polarization at low current densities.

The present investigation show that in neutral media like 3% sodium chloride solution and even in HCl and CH₃COOH, it is only the cathode which is significantly polarized compared to the anode particularly at lower current densities.
3.4. THIOUREAS

RESULTS

(A) Hydrochloric acid:

Weight loss results in tables 3.31 to 3.35 on pages 232 to 236.

In general the inhibition increased with increase in the concentration of thiourea or its derivatives in both the acid solutions (0.5 and 1 M) at both the durations (2 and 4 days). It was further observed that the inhibition usually increased with increase in duration in a given acid solution at the higher concentrations of the compound added. In the case of thiourea some discrepancy appeared where a maximum in inhibition was observed at some intermediate concentration and also that in 0.5 M HCl some lowering of inhibition at higher concentrations. In a very narrow sense the inhibitive power of thiourea and its derivatives decreased with increase in concentration of the acid, more so at intermediate and higher concentrations. In the case of N-N'-diethyl thiourea, the behaviour was erratic. In the cases of thiourea (4 days duration) and allyl thiourea (2 days), the trend was somewhat reverse. Excellent inhibition was observed at the highest concentration only in the case of phenyl thiourea; and occasionally in
m-chlorophenyl thiourea. Perceptible acceleration of corrosion was observed at the lower concentration with N-N'-diethylthiourea at four days durations in 0.5 M HCl.

Galvanostatic Measurements (Figs. 32 to 35 on page 282 to 285):

In the presence of thiourea and its derivatives instead of obtaining a jump when the cathode was polarised a steady increase in polarisation was observed with increase in current density. The magnitude of polarisation was quite high compared to the blank only at the higher current densities, more particularly in the cases of phenyl thiourea, diphenylthiourea, m-chlorophenyl thiourea; the magnitude of m-chlorophenyl thiourea and N-N'-dibutyl thiourea. In the cases of phenylthiourea and m-chlorophenyl thiourea the magnitude of polarization of the anode was high at the higher current densities. It is interesting to note that thiourea showing same high inhibition (98%) as m-chlorophenylthiourea do not show such high polarisation.

(B) Acetic acid and Chlorosubstituted acetic acid:

Thiourea:

Weight loss results are given in table 3.36 on page 237. Thiourea mostly acted as poor inhibitor except in trichloroacetic acid where it had good or effective inhibition in 0.1 M acid solution and excellent inhibition at the intermediate concentration in 0.5 M acid. It is
interesting to note that maximum inhibition by thiourea at any duration in any acid solution was observed at its intermediate concentration. Acceleration of corrosion, at highest concentration of thiourea was observed in acetic acid solution and to some extent in monochloroacetic acid. In general there was a fall in inhibition with increase in duration except in trichloroacetic acid, where some increase was observed in 0.1 M acid solution. With increase in concentration of acid some increase in inhibition was observed in di- and trichloroacetic acid at shorter duration while in monochloroacetic acid there was some fall at both the durations.

Phenylthiourea:

Weight loss results are given in table 3.37 on page 238. Phenylthiourea acted as an excellent or good inhibitor in trichloroacetic acid at most of its concentrations. In general its inhibitive power fall with increase in duration in mono and dichloroacetic acids. In 0.1 M acetic acid the behaviour is somewhat erratic. With increase in concentration of the acid, the inhibitive power of phenyl thiourea in general, decreased the extent of the decrease decreasing with increase in chlorine content of the acid. It is interesting to note that even though there was variation the inhibitive power was not significantly change with
increase in concentration of phenyl thiourea.

Diphenylthiourea:

Weight loss results are given in table 3.38 on page 139.

Except in trichloroacetic acid, diphenylthiourea mostly acted as poor or fair inhibitor. Its behaviour with increase in its concentration was somewhat erratic. Its inhibitive power generally decreased with increase in duration except in 0.1 M dichloro and trichloroacetic acid. In trichloroacetic acid inhibitive power increased somewhat and remained nearly constant in 0.1 and 0.5 M acids respectively. Except in acetic acid there was an increase in inhibition with increase in concentration of acid at a given concentration of diphenylthiourea at a given duration. Excellent or good inhibition was observed only in 0.5 M trichloroacetic acid.

m-Chlorophenylthiourea:

Weight loss results are given in table 3.39 on page 140.

The inhibitive power of m-chlorophenylthiourea increased with increase in its concentration. At lower concentrations, in monochloroacetic acid it accelerated corrosion. Significant decrease in inhibition with increase in duration was observed in mono- and dichloroacetic acids, and also in 0.5 M trichloroacetic acid (lower concentration). In acetic and monochloroacetic acids the
inhibitive power decreased with increase in concentration of the acid at a given duration. In dichloroacetic acid the situation is practically reverse. In trichloroacetic acid however the increase and decrease were observed respectively for one and two days duration.

**p-methoxyphenyl thiourea:**

Weight loss results are given in table 3.40 on page 241.

The inhibitive power of p-methoxyphenyl thiourea mostly increased with increase in its concentration. It acted as an excellent inhibitor at most of its concentrations in trichloroacetic acid. In acetic acid its inhibitive power was fairly good at its higher concentrations. In remaining two acids its inhibitive power was mostly fair and poor frequently. Acceleration of corrosion was observed in monochloroacetic acid at longer duration. With increase in duration the compound usually showed decrease in inhibition, except in 0.1 M trichloroacetic acid where the reverse trend prevailed to a minor extent. The variation in inhibition with increase in the concentration in acid at a given duration was somewhat erratic.

**p-nitrophenyl thiourea:**

Weight loss results are given in table 3.41 on page 242.

The inhibitive power of p-nitrophenyl thiourea increased with increase in its concentration, showing
acceleration of corrosion at lower concentration in 0.5 M trichloroacetic acid. The inhibitive power usually decreased with increase in the duration except in 0.1 M trichloroacetic acid where it acted as an excellent inhibitor, more so at 2 days duration. The compound exhibited low inhibition with increase in acid concentration at a given duration.

N-N'-di-o-tolyl thiourea:

Weight loss results are given in table 3.42 on page 43.

The inhibitive power of N-N'-di-o-tolyl thiourea increased with increase in its concentration in 0.1 M acid solutions, showing excellent inhibition in 0.1 M acetic acid and trichloroacetic at higher concentration except at longer duration in acetic acid. In 0.5 M acid solutions a maximum inhibition was observed at the intermediate concentration. In acetic and monochloroacetic acids the inhibitive power decreased with increase in the duration, the trend being opposite in dichloroacetic acid. In acetic and dichloroacetic acid the inhibition mostly decreased with increase in the concentration of the acid, at a given duration the reverse trend prevailing in monochloroacetic acid.

N-N'-di-p-tolyl thiourea:

Weight loss results are given in table 3.43 on page 44.

The inhibitive power of N-N'-di-p-tolyl thiourea increased with increase in its concentration except acetic acid
where maximum inhibition was observed at its intermediate concentration. In acetic and monochloroacetic acid, the inhibitive power decreased with increase in the duration; while in trichloroacetic acid it showed reverse trend. In acetic and trichloroacetic acids the inhibitive power decreased with increase in concentration of the acid for a given duration while in remaining two acids the inhibition increased.

**N-N'-diethylationurea:**

Weight loss results are given in table 3.44 on page 245.

In trichloroacetic acid, N-N'-diethylthiourea acted as a good inhibitor in dilute acid and very excellent inhibitor in concentrated acid. In rest of the acids a maximum in the inhibition was observed at some intermediate concentration. In general, this derivative of thiourea acted as a poor inhibitor. Its inhibitive power usually decreased with increase in the duration except in dilute dichloro and trichloroacetic acid.

In general the inhibitive power at a given duration increased with increase in the concentration of acid.

**N-N'-dibutylthiourea:**

Weight loss results are given in table 3.45 on page 246.

The inhibitive power of N-N'-dibutylthiourea increased with increase in its concentration. In general there appeared
to be no distinct feature regarding the change in inhibition with increase in duration as well as the conc. of acid. In monochloroacetic acid however some decrease was observed with increase in durations while in acetic acid increasing inhibition was observed with increase in concentration of acid. This derivative of thiourea showed increased inhibition with increase in chlorine content of the acid. In trichloroacetic acid the compound acted as an excellent inhibitor at all its concentrations.

Galvanostatic Measurements:

Acetic acid (Figs. 36 to 39 on pages 186 to 189):
In presence of thiourea and its derivatives polarisation of cathode was usually high when the plate was made cathode. Significant cathode polarisation took place particularly in the presence of N-N'-dibutylthiourea. Even though the polarisation of the anode in the presence of thiourea and its derivatives was high than that in blank, the magnitude of polarisation was mostly not significant.

Monochloroacetic acid (Figs. 40 to 44 on pages 190 to 194):
Compared to the blank the polarisation of the cathode was always somewhat higher, same was the case with the polarisation of the anode.

Dichloroacetic acid (Figs. 45 to 49 on pages 195 to 199):
Most of the thioureas showed higher polarisation of
the cathode at higher current densities. The polarisation of
the anode was also somewhat higher compared to the blank
but much less compared to the polarisation of the cathode.

Trichloroacetic acid (Figs. 50 to 54 on pages 300 to 304):
Thioureas in general showed significant polarisation
of the cathode only at high current densities. Anode also
showed some polarisation at most of the current densities
though low compared to the cathode polarisation.

(C) 3% Sodium chloride:
Weight loss results are given in tables 3.46 and
3.47 on pages 247 and 248.

The inhibitive power of most of the thiourea
derivatives increased with increase in their concentrations
in 3% sodium chloride system, except N-N'-diethyl thiourea
and N-N'-dibutylthiourea. Excellent inhibition was observed
with phenyl thiourea and m-chlorophenyl thiourea at their
highest concentration.

Thiourea simply accelerated corrosion in this systems,
the extent increasing with increase in its concentration.
N-N'-diethylthiourea and N-N'-dibutylthiourea also showed
acceleration of corrosion particularly at their higher
concentration in sodium chloride solution.

Most of thiourea derivatives can be considered as
poor inhibitors.
Galvanostatic Measurements (Figs. 55 to 58 on pages 305 to 308. The polarisation in the presence of thiourea was nearly of the same patern as that of blank in the case of cathode. In the rest of the cases the polarisation of the cathode increased continuously at the lower current densities, becoming nearly steady at higher current densities. The polarisation of anode was nearly of the same patern in almost all thioureas except m-chlorophenylthiourea which showed somewhat high polarisation at very high current densities.

(D) Artificial Sea Water:

Weight loss results are given in tables 3.48 and 3.49 on pages 249 and 250.

Thiourea accelerated corrosion in artificial sea water; the magnitude of acceleration with increase in its concentration is very small. In the case of phenylthiourea and m-chlorophenylthiourea the acceleration of corrosion was observed at lower concentration of the compounds but good or excellent inhibition was afforded by these two compounds at thier higher concentrations. Diethylthiourea showed somewhat awkward behaviour where acceleration of corrosion at its lowest and highest concentration was obtained. Except phenylthiourea and m-chlorophenylthiourea, all derivatives were found poor inhibitors for copper in artificial sea water.
Mostly in all these compounds inhibitive power decreased with increase in duration.

Galvanostatic Measurements (Figs. 59 to 62 on pages 309 to 312):

The addition of thiourea or its derivatives to artificial sea water did not substantially differ in the nature of cathode or anode polarisation compared to the corresponding behaviour in 3% sodium chloride except that in some cases the polarisation of cathode in artificial sea water was somewhat less than that in 3% sodium chloride.
THIOUREAS

\[
\begin{align*}
H_2N\text{C=NH}_2 & \quad \text{Thiourea} \\
\text{C=S} & \quad \text{Phenylthiourea} \\
\text{Cl} & \quad \text{m-chlorophenyl thiourea} \\
\text{C=S} & \quad \text{p-methoxyphenyl thiourea} \\
\text{NO}_2 & \quad \text{p-nitrophenyl thiourea} \\
\text{H}_3C & \quad \text{N-N' di-o-tolyl thiourea} \\
\text{C}_4H_9\text{N} & \quad \text{N-N' di-p-tolyl thiourea} \\
\text{CH}_3 & \quad \text{N-methyl thiourea} \\
\text{CH}=\text{CH} & \quad \text{N-allyl thiourea}
\end{align*}
\]
DISCUSSION

The inhibitive power of thiourea and its derivatives is not very regular in different media. In some media, for example in NaCl solution, thiourea accelerates corrosion while in trichloroacetic acid solution it exhibits high inhibition. Number of workers have studied thiourea and its derivatives as corrosion inhibitors for different metals in different media. Variety of mechanisms have been proposed to explain the inhibition exhibited by these compounds. According to Hackeman \((411)\) variations in inhibition effectiveness of the substituents of organic group in thiourea are attributed to consequent changes of solubility, electronic structure of the functional group and the structure of adsorbed film. According to Maxted et al. \((452)\) chemisorption is more or less reversible. Consequently the extent of chemisorption of these compounds should be a function of their solubility.

The possibility of correlating structural characteristics with the inhibitive properties of organic compounds is justified by the fact that the metal inhibition interactions are based on chemisorption. A bond of Lewis acid-base type \((382)\) is formed generally with the inhibitor as the electron donor and the metal as an electron acceptor, the strength of the bond depending upon the characteristics of both the adsorbent and adsorbate.
In thiourea and its derivatives two polar functions having atoms of nitrogen and sulphur are present. It is agreed upon, in general, that the polar function is the reaction centre for the establishment of the chemisorption process. In the case of thiourea and its derivatives both nitrogen and sulphur can act as reaction centres.

A number of workers have studied thiourea and related compounds for their co-ordination properties. The electronic structures I, II and III each contributing roughly an equal amount.

Thiourea and its derivatives have been reported to form co-ordination compounds with number of transition metal ions(453). Looking at the overall superiority of sulphur containing organic compounds over the nitrogen containing organic compounds due to low solubility of sulphur compound and lower electronegativity of sulphur atom compared to nitrogen atom and presence of two lone pairs of electron in sulphur compared to one in nitrogen, it becomes obvious that the sulphur atom of thiourea may...
co-ordinate more towards formation of co-ordination. However, this cannot rule out, some possible contribution by nitrogen in complex formation.

From the studies of infrared spectra we can find out whether co-ordination with the metal ions occurs through sulphur or nitrogen atom. Most of the transition metal ions form M-S bonds. Copper(I) has been reported to form M-S bond with thiourea(454). Some of the substituted thioureas can also form M-N bonds with metal ions. Thus M-N bond formation has been observed by Lane and his colleagues(455) with copper(I) using methyl thiourea as ligand.

An abstract of the results obtained on inhibition by thiourea and its derivatives (at their lowest and highest concentrations only) in various media is given in TableA on page 191.

The results, in general, show that the acceleration of corrosion is confined mostly to the neutral media while inhibition to the acidic media. It is interesting to note that extremely high inhibition ( > 95%) is observed either in the neutral media or mostly in the media containing strong acids.

Acceleration of corrosion by thiourea is observed in the neutral media (at both the concentrations) and at higher concentration of thiourea in acetic acid.
<table>
<thead>
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<th>Inhibitor</th>
<th>Concentration</th>
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<th>Concentration</th>
<th>Concentration</th>
<th>Concentration</th>
<th>Concentration</th>
<th>3%</th>
<th>Artificial Sea water</th>
</tr>
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<td>Conc. (M)</td>
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<td>17</td>
<td>21</td>
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<td>-336</td>
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</table>
The acceleration of corrosion by thiourea has been attributed to $H_2S$ produced by cathodic reaction of thiourea\(^{(456)}\).

$$H_2N - C - NH_2 + 6H + 2H^+ \rightarrow NH_4^+ + CH_3NH_3^+ + H_2S$$

This reaction is accompanied by some minor side reactions where $CH_4$ is produced. $H_2S$ so produced acts as an anodic depolarizer. The reduction of thiourea implies that the cathode reaction is also depolarized to some extent. The overall result of depolarisation is to accelerate corrosion. When thiourea is present in small concentration, the adsorbed layer is incomplete and dissolution of the metal will be partially inhibited. As $H_2S$ depolarizes anode, there is overall acceleration of corrosion. This mechanisms has been challenged by Antropov\(^{(457)}\). He observed that very high coefficient of inhibition in HCl solutions, saturated with $H_2S$. The present investigation show that no acceleration is observed in hydrochloric acid while some acceleration is observed in acetic acid only at a higher concentration of thiourea. According to Makrides and Hackerman at higher concentration of thiourea adsorption becomes extensive which decreases the rate of anodic reaction considerably. The results of the present investigation are not in agreement with this view because
there is a continuous increase in acceleration of corrosion with increase in the concentration of thiourea in sodium chloride solution.

In an experiment, varying quantity of sodium sulphide (Na$_2$S) was added to 0.5 M HCl or 3% sodium chloride solution. Copper plates were immersed for 2 and 15 days respectively in the HCl and NaCl solutions. It was observed that in HCl there was inhibition up to 30% (added Na$_2$S up to 2.6x10$^{-3}$M) while in the case of NaCl acceleration up to 50% was observed. If we compare these results with those obtained for thiourea in the present investigation high inhibition up to 94% in HCl and 33% acceleration in NaCl cannot be attributed to H$_2$S. High inhibition in HCl can be explained only if we assume that thiourea is adsorbed by the copper surface at all the concentrations, the magnitude of adsorption increasing with increase in its concentration. Again in the case of sodium chloride, acceleration (approximately 330%) cannot be assigned to H$_2$S. It is probable that reaction products at the local anode (Cu$^{2+}$) are removed not principally by H$_2$S, but by thiourea itself. It was observed that an aqueous solution of thiourea gives white precipitate with copper ions in 3% sodium chloride solution. This suggests that most of the copper ions are removed as insoluble products at the local anodes; the remaining copper ions may migrate to the cathode.

A probable mechanism to explain acceleration of corrosion of
copper in 3% sodium chloride is suggested below. Thiourea, a highly soluble substance, is probably not adsorbed, or adsorbed to a negligible extent over the copper surface. This leaves cathodic and anodic area nearly intact. Anodic products (Cu\(^{2+}\)) form insoluble precipitate, thus removing the corrosion products from the anodic site, a process of depolarization.

At the cathode hydrogen ions are produced due to cathodic reduction of hydrogen ions leaving hydroxyl ions at the sites i.e. formation of NaOH at the cathode. This NaOH forms Cu(OH)\(_2\) by combining with copper ions that may have possibly migrated to the cathode. This copper hydroxide is essentially CuO.xH\(_2\)O which is black in colour. It is interesting to note that whenever acceleration is observed in NaCl solution, the copper plate is usually covered with a black film. This mechanism appears to be more probable because the acceleration of corrosion increases with increase in concentration of thiourea.

It is pertinent to note that above mechanism is applicable strictly to thiourea only which is highly soluble in the aqueous media. Other derivatives of thiourea are not so soluble in aqueous media; in fact their solutions were prepared in ethyl alcohol. The acceleration exhibited by them at their lower concentration and inhibition at higher concentration can be explained on the lines suggested
by Hackerman and Makridas. Acceleration observed at higher concentration of thiourea and inhibition at lower concentration in acetic acid solution still remains unexplained.

We will now consider the effect of substituted thiourea, on inhibition. As pointed out earlier, the electron density over the sulphur atom acting as a reaction centre and polarizability of the function decide strength of adsorption bond in the chemisorption process. The order of increasing inhibitive power (or decreasing acceleration of corrosion) at the higher concentration of the compound in the neutral and acidic media is as follows:

**Hydrochloric acid**

Diethylthiourea < di-p-tolylthiourea < di-o-tolylthiourea < diphenylthiourea < dibutylthiourea < thiourea < m-chlorophenylthiourea = phenylthiourea

3% Sodium chloride

Thiourea < dibutylthiourea < diethylthiourea < di-p-tolylthiourea < di-o-tolylthiourea < diphenylthiourea < m-chlorophenylthiourea < phenylthiourea.

The adsorption of thiourea takes place probably through sulphur atom. Substitution of various radicals or groups in thiourea takes place on the nitrogen atom. If the substitutents are electron withdrawing (e.g., phenyl ring)
or electron repelling (e.g., methyl group) the change of electron density takes place over atom or atoms of nitrogen but not over the sulphur atom. If the adsorption is to take place through sulphur atom the change of electron density should not significantly affect the inhibitive powers. The order of increasing inhibition for some thiourea derivatives in HCl is given below:

Methyl thiourea < Thiourea < m-Chlorophenyl thiourea

This order suggests that increase or decrease of the inhibition is not in accordance with increase or decrease of electron density over nitrogen atom as suggested by the earlier view. This order also suggests that chemisorption, if it takes place, does not seem to take place through the nitrogen atom over which these groups have been substituted.

Let us consider the derivatives in which substitution is carried out over both the nitrogen atoms. The order of increasing inhibition in HCl for some selected compounds is as follows:

Diethyl thiourea < diphenyl thiourea
< dibutyl thiourea < Thiourea

If chemisorption takes place through nitrogen, we should have the order
diphenyl thiourea $\leftarrow$ thiourea $\leftarrow$ diethyl thiourea $\leftarrow$ dibutyl thiourea

In any case the idea of electron density does not seem to applicable for these derivatives for copper in the acid media.

In acetic and chloro substituted acetic acids also the expected order is not obtained except in trichloro acetic acid where we get the order:

diphenyl thiourea $\leftarrow$ thiourea $\leftarrow$ diethyl thiourea $=$ dibutyl thiourea

It is likely that this may be an accident.

According to Mann (412) and Hackerman (414) the cross sectional area of the ion projected down on the metal and the closeness of packing of the ion in covering layer determine the effectiveness of the inhibitor.

A co-relation although a slight one, was obtained by Hackerman between the values of the inhibition and circular areas relating to the arrangement parallel to surface. If the area projected is high the inhibition should also be correspondingly high. Let us consider the results obtained with thiourea, diethylthiourea and diphenylthiourea in hydrochloric acid. The order of increasing inhibitive power, in general, is as follows:

diethylthiourea $\leftarrow$ diphenylthiourea $\leftarrow$ thiourea

In the case of the remaining acids, the orders obtained not only differ from the above but also differ
among themselves. Thus the value of the projected molecular area does not appear to be a decisive factor for the inhibition. This is in agreement with the work carried out by Podobaev(387) cited earlier. It is interesting to point out at this stage the effect of two similar compounds on the extent of inhibition, \( N'N'\text{-di-p-tolyl thiourea} \) and \( N'N'\text{-di-o-tolyl thiourea} \) exhibit respectively high difference in inhibition in acetic acid and dichloroacetic acid; while they show roughly same inhibition in hydrochloric acid and trichloroacetic acids. These results again do not support the earlier view because both the compounds have equal geometrical area, same molecular weight and the same structural formula with only one difference: that is, the location of the methyl radical in the ring.

From the above discussion it is apparent that inhibition exhibited by thiourea and its derivatives involves some other mechanism. As with mercaptans and azoles, it was observed that specimens treated with 0.02 % compound in water when exposed to HCl vapours showed the presence of reddish or reddish brown film on the metal surface, after cleaning. This explains that it is perhaps the nature of the film which appears to play some role in the inhibition exhibited by the above compounds.

We will now consider the behaviour of the inhibitor with increase in duration. In the case of hydrochloric acid,
most of the inhibitors show increase in inhibition with increase in duration. Of course, large size substances do show constant inhibition indicating that film, if formed, does not lose its protective character; on the contrary, some film repair appears to take place on standing. In acetic and monochloroacetic acid the film appears to decrease its protective character with time as majority of the substances show decrease in inhibition, more particularly in monochloroacetic acid, with increase in duration. In trichloroacetic acid and to a some lesser extent in dichloroacetic acid the film appears to maintain its protective character with increase in duration. In neutral media on the other hand there appears to be no definite trend.

The effect of increasing the strength of acid does not again seem to have a regular pattern of inhibition. Thus in hydrochloric acid and to somewhat lesser extent in acetic acid and to a lesser extent in monochloroacetic acid the inhibitive power declines with increase in the acid strength. Of course in hydrochloric acid nearly constant values of inhibition and in monochloroacetic acid an increase in inhibition is observed with many substances. In dichloroacetic acid the inhibitive power usually increases with increase in strength of the acid. In trichloroacetic acid no definite trend appears to prevail.

The results of Galvanostatic measurements reveal that almost all the thioureas polarize cathode to some extent.
Very high polarization is observed mostly in trichloroacetic acid, while in rest of the acids, the magnitude of cathodic polarization, in general, decreases with decrease in the chlorine content of the acetic acid. High cathodic polarization in hydrochloric acid is observed with dibutylthiourea and m-chlorophenyl thiourea. It is interesting to note that majority of the thiourea do not show any significant polarization either of cathode or anode in sodium chloride solution even when there is about 98% or more inhibition (phenyl thiourea and m-chlorophenylthiourea). It is equally surprising that some cathode polarization is observed with dibutylthiourea even when it shows very high acceleration of corrosion. It is pertinent to note that high cathode polarization does not necessarily mean high corrosion inhibition (phenyl thiourea in dichloroacetic acid - 65%, Dibutylthiourea in HCl - 88%).

Shift of the steady state potential with most of the inhibitors, particularly with the acid media, is in the cathodic direction indicating polarization of the local anode while polarization with the impressed current show usually cathodic behaviour.
### TABLE 3.1

**INHIBITION OF CORROSION OF COPPER IN HYDROCHLORIC ACID SOLUTION**

Temperature: 32±2°C  
Coupon: 3x6 cms

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<th>Loss Inhibition (%)</th>
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<td>(mg)</td>
<td>(mdd)</td>
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### TABLE 3.2

**INHIBITION OF CORROSION OF COPPER IN HYDROCHLORIC ACID SOLUTION**

**Temperature**: 32±2°C  
**Coupon**: 3x6 cms

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<th>Loss Inhibition (mdd)</th>
<th>Inhibition (%)</th>
<th>Loss Inhibition (mg)</th>
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### Table 3.3

#### Inhibition of Corrosion of Copper in Acetic Acid and Chlorosubstituted Acetic Acid Solutions

- **Temperature:** 32±2°C
- **Coupon:** 3x6 cm²
- **Inhibitor:** Ethyl mercaptan

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TABLE 3.4
INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS

Temperature: 32±2°C  
Coupon: 3x6 cms

Inhibitor: Propyl mercaptan

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### Table 3.5

**Inhibition of Corrosion of Copper in Acetic Acid and Chlorosubstituted Acetic Acid Solutions**

**Temperature:** 32±2°C  
**Coupons:** 3x6 cms  
**Inhibitor:** Butyl mercaptan

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**INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS**

**Temperature:** 32±2°C  
**Coupon:** 3x6 cms  
**Inhibitor:** Dodecyl mercaptan

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### INHIBITION OF CORROSION OF COPPER IN 3\% SODIUM CHLORIDE SOLUTION

**Temperature :** 32±2°C  
**Coupon :** 3x6 cms

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**INHIBITION OF CORROSION OF COPPER IN HYDROCHLORIC ACID SOLUTION**

**Temperature**: 32±2°C  
**Coupon**: 3x6 cms

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**Note**: All concentrations are in Molar (M).
TABLE 3.10

INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND
CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS

Temperature: 32 ± 2°C Coupon: 3x6 cms
Inhibitor: Diethyl sulphide

<table>
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<th>5 days</th>
<th>10 days</th>
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<td>8.8x10⁻¹</td>
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<th>5 days</th>
<th>10 days</th>
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<td>Loss 10 days (mg)</td>
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<td>Loss 5 days (mg)</td>
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**TABLE 3.11**

**INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS**

**Temperature:** 32±2°C  
**Coupon:** 3x6 cms  
**Inhibitor:** Dibenzyl disulphide

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<th>Duration: 5 days</th>
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<th>Loss 10 days (mg)</th>
<th>Inhibition 10 days (%)</th>
<th>Loss 5 days (mg)</th>
<th>Inhibition 5 days (%)</th>
<th>Loss 10 days (mg)</th>
<th>Inhibition 10 days (%)</th>
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<table>
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<td>93</td>
<td>2.0</td>
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<td>2.4x10^-4</td>
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<td>94</td>
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<td>4.9x10^-4</td>
<td>1.6</td>
<td>95</td>
<td>1.8</td>
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<tr>
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<td>95</td>
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### TABLE 3.12

**INHIBITION OF CORROSION OF COPPER IN 3% SODIUM CHLORIDE SOLUTION**

**Temperature:** 32±2°C  
**Coupon:** 3x6 cms

<table>
<thead>
<tr>
<th>Concentration of Inhibitor (M)/Duration</th>
<th>Loss (mg)</th>
<th>% Inhibition (mm)</th>
<th>Loss (mg)</th>
<th>% Inhibition (mm)</th>
</tr>
</thead>
<tbody>
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<td>0.0</td>
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<td>69.5</td>
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<td>57.8</td>
<td>5.33</td>
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<tr>
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</table>

**Diethyl sulphide**

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<tr>
<th>Concentration of Inhibitor (M)/Duration</th>
<th>Loss (mg)</th>
<th>% Inhibition (mm)</th>
<th>Loss (mg)</th>
<th>% Inhibition (mm)</th>
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<td>56.4</td>
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**Dibenzyl disulphide**
### TABLE 3.13

**INHIBITION OF CORROSION OF COPPER IN ARTIFICIAL SEA WATER**

Temperature: 32±2°C  
Coupon: 3x6 cms

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<th>Loss (mg)</th>
<th>Loss (mg)</th>
<th>Inhibition (%)</th>
<th>Loss (mg)</th>
<th>Loss (mg)</th>
<th>Inhibition (%)</th>
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<td>15 days</td>
<td>30 days</td>
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<tr>
<td>Diethyl sulphide</td>
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</tr>
<tr>
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<td>6.03</td>
<td>-</td>
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<td>80.3</td>
<td>7.43</td>
<td>-24</td>
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<tr>
<td>2 (10^{-4})</td>
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<td>-08</td>
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<td>7.41</td>
<td>-23</td>
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<td>18</td>
<td>77.5</td>
<td>7.18</td>
<td>-19</td>
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<td>70.5</td>
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<tr>
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<td>5.17</td>
<td>18</td>
<td>64.3</td>
<td>5.95</td>
<td>01</td>
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<tr>
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<td>4.19</td>
<td>34</td>
<td>70.6</td>
<td>6.54</td>
<td>-09</td>
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<td>78.1</td>
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<td>-20</td>
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<tr>
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TABLE: 3.14

INHIBITION OF CORROSION OF COPPER IN HYDROCHLORIC ACID SOLUTION

Temperature: 32±2°C  
Coupon: 3x6 cms

<table>
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<tr>
<th>Concentration of Inhibitor (M)/Duration</th>
<th>Loss of Inhibitor (mg)</th>
<th>Loss of Inhibitor (mg/days)</th>
<th>Inhibition (%)</th>
<th>Loss of Inhibitor (mg)</th>
<th>Loss of Inhibitor (mg/days)</th>
<th>Inhibition (%)</th>
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<tr>
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### TABLE 3.15

**INHIBITION OF CORROSION OF COPPER IN HYDROCHLORIC ACID SOLUTION**

Temperature: 32±2°C  
Coupon: 3x6 cms

<table>
<thead>
<tr>
<th>Concentration of Inhibitor (M)</th>
<th>Loss (mg)</th>
<th>Loss (mdd)</th>
<th>Inhibition (%)</th>
<th>Loss (mg)</th>
<th>Loss (mdd)</th>
<th>Inhibition (%)</th>
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**5:6 dimethylbenzimidazole**

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**1M Hydrochloric acid**

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<th>Loss (mg)</th>
<th>Loss (mdd)</th>
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**2-mercaptobenzimidazole**

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<th>Loss (mg)</th>
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TABLE : 3.16

INHIBITION OF CORROSION OF COPPER IN HYDROCHLORIC ACID SOLUTION

Temperature : 32±2°C                Coupon : 3x6 cms

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<th>Loss (mdd)</th>
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<th>Loss (mg)</th>
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1:2:3 benzotriazole

0.5M Hydrochloric acid

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<th>Loss (mg)</th>
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1M Hydrochloric acid

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Sulphathiazole

0.5M Hydrochloric acid

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1M Hydrochloric acid

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<th>Loss (mg)</th>
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### TABLE 3.17

INHIBITION OF CORROSION OF COPPER IN HYDROCHLORIC ACID SOLUTION

**Temperature:** 32±2°C  
**Coupon:** 3x6 cms

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<th>Loss (mgd)</th>
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### TABLE: 3.18

**INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS**

Temperature: 32±2°C  
Coupon: 3x6 cms  
Inhibitor: Benzimidazole

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### Table 3.19

INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS

Temperature: 32±2°C  
Coupon: 3x6 cms  
Inhibitor: 2-methylbenzimidazole

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TABLE 3.20

INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS

Temperature: 32±2°C  Coupon: 3x6 cm

Inhibitor: 5-6 dimethylbenzimidazole

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INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS

Temperature: 32±2°C  Coupon: 3x6 cm²

Inhibitor: 2-mercaptobenzimidazole

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TABLE : 3.22

INHIBITION OF COHESION OF COPPER IN ACETIC ACID AND
CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS

Temperature: 32±2°C  Coupon: 3x6 eras
Inhibitor: 1,2,3 benzo triazole

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### TABLE 3.23

**INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS**

**Temperature:** 32±2°C  **Coupon:** 3x6 cms  **Inhibitor:** Sulphathiazole

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# Table 3.24

**Inhibition of Corrosion of Copper in Acetic Acid and Chlorosubstituted Acetic Acid Solutions**

**Temperature:** 32±2°C  
**Coupon:** 3x6 cms  
**Inhibitor:** 2-mercaptobenzothiazole

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### TABLE: 3.25

**INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS**

Temperature: 32±2°C  
Coupon: 3x6 cms  
Inhibitor: 2-mercaptotiazoline

<table>
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<tr>
<th>Concentration of Inhibitor (M)</th>
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<th>Loss Inhibition (mg) 10 days</th>
<th>%</th>
<th>Loss Inhibition (mg) 5 days</th>
<th>%</th>
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<th>%</th>
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<th>Loss Inhibition (mg)</th>
<th>%</th>
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<th>%</th>
<th>Loss Inhibition (mg)</th>
<th>%</th>
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<th>%</th>
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<th>%</th>
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<th>Loss Inhibition (mg)</th>
<th>%</th>
<th>Loss Inhibition (mg)</th>
<th>%</th>
<th>Loss Inhibition (mg)</th>
<th>%</th>
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### TABLE 3.26

**INHIBITION OF CORROSION OF COPPER IN 3% SODIUM CHLORIDE SOLUTION**

**Temperature : 32±2°C**

<table>
<thead>
<tr>
<th>Concentration of Inhibitor (M)</th>
<th>Loss (mg)</th>
<th>Loss (mdd)</th>
<th>Inhibition (%)</th>
<th>Loss (mg)</th>
<th>Loss (mdd)</th>
<th>Inhibition (%)</th>
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<td>30 days</td>
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TABLE: 3.27

INHIBITION OF CORROSION OF COPPER IN 3% SODIUM CHLORIDE SOLUTION

Temperature: 32±2°C  Coupon: 3x6 cms

<table>
<thead>
<tr>
<th>Concentration of Inhibitor (M)/Duration</th>
<th>Loss (mg)</th>
<th>Loss (mdd)</th>
<th>Inhibition (%) 15 days</th>
<th>Loss (mg)</th>
<th>Loss (mdd)</th>
<th>Inhibition (%) 30 days</th>
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2-hydroxymethylbenzimidazole

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<th>Loss (mdd)</th>
<th>Inhibition (%) 15 days</th>
<th>Loss (mg)</th>
<th>Loss (mdd)</th>
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2-hydroxyethylbenzimidazole

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<th>Loss (mdd)</th>
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<th>Loss (mg)</th>
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<td>0.31</td>
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<tr>
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2-hydroxybenzylbenzimidazole

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<th>Loss (mg)</th>
<th>Loss (mdd)</th>
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2-mercaptomethylbenzimidazole
**TABLE 3.28**

INHIBITION OF CORROSION OF COPPER IN 3% SODIUM CHLORIDE SOLUTION

Temperature: 32±2°C  
Coupon: 3x6 cms

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<th>Loss (mg)</th>
<th>Loss (mdd) Inhibition (%)</th>
<th>Loss (mg)</th>
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**1:2:3 benzotriazole**

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**Sulphathiazole**

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**2-mercaptobenzothiazole**

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**2-mercaptothiazoline**
### TABLE: 3.29

**INHIBITION OF CORROSION OF COPPER IN ARTIFICIAL SEA WATER**

**Temperature**: 32±2°C  
**Coupon**: 3x6 cms

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### TABLE 3.30

**INHIBITION OF CORROSION OF COPPER IN ARTIFICIAL SEA WATER**

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<th>Inhibition (%)</th>
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<th>Loss (mmd)</th>
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### TABLE 3.33

INHIBITION OF CORROSION OF COPPER IN HYDROCHLORIC ACID SOLUTIONS

Temperature: 32±2°C  
Coupon: 3x6 cms

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<th>Loss Inhibition (%)</th>
<th>Loss (mg)</th>
<th>Loss Inhibition (%)</th>
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**0.5M Hydrochloric acid**

| **N-N'-di-o-tolylthiourea**            |           |                     |           |                     |
| 0.0                                    | 76.0      | 106                 | -         | 402                 |
| 7.8x10⁻⁴                               | 58.2      | 80.8                | 23        | 325                 |
| 1.6x10⁻⁴                               | 55.5      | 77.7                | 27        | 316                 |
| 3.1x10⁻⁴                               | 48.5      | 67.3                | 36        | 228                 |
| 7.8x10⁻³                               | 32.4      | 44.9                | 46        | 136                 |
| 1.6x10⁻³                               | 24.6      | 34.0                | 68        | 77.3                |

**1M Hydrochloric acid**

| **N-N'-di-o-tolylthiourea**            |           |                     |           |                     |
| 7.8x10⁻⁴                               | 58.0      | 80.5                | 11        | 360                 |
| 1.6x10⁻⁴                               | 49.4      | 68.5                | 24        | 268                 |
| 3.1x10⁻⁴                               | 34.8      | 48.3                | 47        | 156                 |
| 7.8x10⁻³                               | 30.2      | 41.9                | 55        | 124                 |
| 1.6x10⁻³                               | 23.8      | 33.0                | 64        | 79                  |

**N-N'-di-p-tolylthiourea**

**0.5M Hydrochloric acid**

| **N-N'-di-p-tolylthiourea**            |           |                     |           |                     |
| 7.8x10⁻⁴                               | 87.1      | 121                 | -         | 318                 |
| 1.6x10⁻⁴                               | 82.0      | 114                 | -         | 275                 |
| 3.1x10⁻⁴                               | 60.0      | 83                  | 21        | 255                 |
| 7.8x10⁻³                               | 38.4      | 53                  | 50        | 214                 |
| 1.6x10⁻³                               | 31.5      | 43.7                | 59        | 172                 |

**1M Hydrochloric acid**
### TABLE: 3.34

**INHIBITION OF CORROSION OF COPPER IN HYDROCHLORIC ACID SOLUTION**

Temperature: 32±2°C  
Coupon: 3x6 cms

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## TABLE: 3.36
INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS

Temperature: 32±2°C  Coupon: 3x6 cms  Inhibitor: Thiourea

<table>
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<tr>
<th>Concentration (M)/Duration</th>
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### Table 3.38

**Inhibition of Corrosion of Copper in Acetic Acid and Chlorosubstituted Acetic Acid Solutions**

**Temperature:** 32±2°C  
**Coupon:** 3x6 cms  
**Inhibitor:** Diphenylthiourea

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<th>Loss Inhibition (mg)</th>
<th>Loss Inhibition (mg)</th>
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**Duration:** 1 day  2 days  1 day  2 days
**TABLE 3.39**

**INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS**

Temperature: 32±2°C  
Coupon: 3x6 cms  
Inhibitor: m-chlorophenylthiourea

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<th>Loss Inhibition (mg)</th>
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</table>
### TABLE: 3.40

INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS

Temperature: 32±2°C  Coupon: 3x6 cms

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<thead>
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<tr>
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<tr>
<td>4.4×10⁻⁴</td>
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<tr>
<td>1.1×10⁻³</td>
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### TABLE 3.41

INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS

**Temperature:** 32±2°C  
**Coupon:** 3x6 cms  
**Inhibitor:** p-nitrophenylthiourea

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<th>Concentration of Inhibitor (M)</th>
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<th>5 days</th>
<th>10 days</th>
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<td>0.5M CH₃COOH</td>
<td>0.5M CHCl₂COOH</td>
</tr>
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</tr>
<tr>
<td></td>
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<td>0.7x10⁻⁴</td>
<td>0.1x10⁻⁴</td>
<td>0.1x10⁻⁴</td>
</tr>
<tr>
<td></td>
<td>2.0x10⁻⁴</td>
<td>4.1x10⁻⁴</td>
<td>2.0x10⁻⁴</td>
<td>2.0x10⁻⁴</td>
</tr>
<tr>
<td></td>
<td>4.1x10⁻⁴</td>
<td>0.7x10⁻⁴</td>
<td>4.1x10⁻⁴</td>
<td>0.7x10⁻⁴</td>
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<tr>
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<td>0.1x10⁻³</td>
<td>1.0x10⁻³</td>
<td>0.1x10⁻³</td>
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</tbody>
</table>

**Duration:**

- 0.1M CH₃COOH
  - 5 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³
  - 10 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³

- 0.1M CHCl₂COOH
  - 5 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³
  - 10 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³

- 0.5M CH₃COOH
  - 5 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³
  - 10 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³

- 0.5M CHCl₂COOH
  - 5 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³
  - 10 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³

**Concen. of** | **Loss (mg)** | **Loss (mg)** | **Loss (mg)** | **Loss (mg)** |
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<td>5 days</td>
<td>10 days</td>
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<td>68.5</td>
<td>48.0</td>
<td>76.0</td>
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<tr>
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<td>20.0</td>
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<td>1.4</td>
<td>1.9</td>
<td>20.0</td>
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**Duration:**

- 0.1M CH₃COOH
  - 1 day: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³
  - 2 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³

- 0.5M CH₃COOH
  - 1 day: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³
  - 2 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³

- 0.1M CHCl₂COOH
  - 1 day: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³
  - 2 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³

- 0.5M CHCl₂COOH
  - 1 day: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³
  - 2 days: 0.0, 5.1x10⁻⁵, 1.0x10⁻⁴, 2.0x10⁻⁴, 4.1x10⁻⁴, 1.0x10⁻³
### TABLE: 3.42

**INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS**

**Temperature:** 32±2°C  
**Coupon:** 3x6 cms  
**Inhibitor:** N-N’ di-o-tolylthiourea

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<th>Loss Inhibition (%)</th>
<th>Loss Inhibition (mg)</th>
<th>Loss Inhibition (%)</th>
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**0.1M CH₃COOH**  
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**0.5M CH₃COOH**  
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### INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS

Temperature: 32±2°C  
Coupon: 3x6 cm²  
Inhibitor: N-N′ di-p-tolyliurea

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<td>Loss Inhibition (mg)</td>
<td>Inhibition (%)</td>
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### Table 3.44

**Inhibition of Corrosion of Copper in Acetic Acid and Chlorosubstituted Acetic Acid Solutions**

**Temperature:** 32±2°C  
**Copper:** 3x6 cms

**Inhibitor:** N-N' diethylthiourea

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| **0.5M CH₃COOH**       |           |        |         |        |         |
| 0.0                    |           |        |         |        |         |
| 7.6x10⁻⁵               | 46.0      | -      | 84.5    | -      | 62.0    | 98.0    |
| 1.4x10⁻⁴               | 29.8      | 37     | 64.3    | 24     | 33.4    | 60.2    |
| 2.8x10⁻⁴               | 24.0      | 48     | 51.5    | 39     | 29.4    | 52.9    |
| 5.6x10⁻⁴               | 25.0      | 46     | 48.7    | 42     | 26.0    | 58.7    |
| 1.4x10⁻³               | 25.6      | 44     | 61.9    | 27     | 37.2    | 61.2    |

| **0.1M CHCl₂COOH**     |           |        |         |        |         |
| 0.0                    |           |        |         |        |         |
| 7.6x10⁻⁵               | 56.0      | -      | 123     | -      | 78.0    | 175     |
| 1.4x10⁻⁴               | 28.7      | 49     | 57.1    | 57     | 36.2    | 54      |
| 2.8x10⁻⁴               | 24.7      | 56     | 50.0    | 62     | 17.3    | 78      |
| 5.6x10⁻⁴               | 26.2      | 53     | 51.4    | 61     | 16.2    | 79      |
| 1.4x10⁻³               | 41.0      | 27     | 75.7    | 54     | 23.4    | 70      |

| **0.5M CHCl₂COOH**     |           |        |         |        |         |
| 0.0                    |           |        |         |        |         |
| 7.6x10⁻⁵               | 14.6      | 50     | 18.8    | 89     | 2.9     | 99      |
| 1.4x10⁻⁴               | 10.7      | 63     | 18.4    | 89     | 2.4     | 99      |
| 2.8x10⁻⁴               | 8.7       | 70     | 17.3    | 90     | 2.0     | 99      |
| 5.6x10⁻⁴               | 6.1       | 79     | 11.5    | 93     | 0.6     | >99     |
| 1.4x10⁻³               | 0.8       | 97     | 4.6     | 97     | 0.2     | >99     |

**Duration:**
- **1 day**
- **2 days**
- **1 day**
- **2 days**

**Notes:**
- The inhibition values are given in milligrams (mg).
- The inhibition percentage (%) is calculated based on the initial weight of the copper sample.
- The duration of the inhibition tests is given in days.
INHIBITION OF CORROSION OF COPPER IN ACETIC ACID AND CHLOROSUBSTITUTED ACETIC ACID SOLUTIONS

Temperature: 32±2°C  
Coupon: 3x6 cms  
Inhibitor: N-N' dibutylthiourea

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<th>Loss Inhibition (mg)</th>
<th>Loss Inhibition (%)</th>
<th>Duration</th>
<th>Loss Inhibition (mg)</th>
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<th>Duration</th>
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### Table 3.47

**Inhibition of Corrosion of Copper in 3% Sodium Chloride Solution**

Temperature: 32±2°C  
Coupon: 3x6 cms

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<th>30 days</th>
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TABLE 3.48

INHIBITION OF CORROSION OF COPPER IN ARTIFICIAL SEA WATER

Temperature: 32±2°C Coupon: 3x6 cms

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<tr>
<th>Concentration of Inhibitor (M)/Duration</th>
<th>Loss (mg)</th>
<th>Loss (mdd)</th>
<th>Inhibition (%)</th>
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<th>Inhibition (%)</th>
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TABLE: 3.49

INHIBITION OF CORROSION OF COPPER IN ARTIFICIAL SEA WATER

Temperature: 32±2°C  Coupon: 3x6 cms

<table>
<thead>
<tr>
<th>Concentration of Inhibitor (M)/Duration</th>
<th>Loss (mg)</th>
<th>Loss (mdd)</th>
<th>Inhibition (%)</th>
<th>Loss (mg)</th>
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<th>Inhibition (%)</th>
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<td>4.26</td>
<td>29</td>
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</table>
Influence of current densities on cathode and anode potential

- **AA'** 0.5M HCl (Blank)
- **BB'** $6.4 \times 10^{-3}$ M ethyl mercaptan
- **CC'** $5.2 \times 10^{-3}$ M propyl mercaptan
- **DD'** $4.4 \times 10^{-3}$ M butyl mercaptan
- **EE'** $9.9 \times 10^{-4}$ M dodecyl mercaptan
Influence of current densities on cathode and anode potential

-AA' 0.5 M CH₃COOH
-BB' 3.2x10⁻³ M ethyl mercaptan
-CC' 2.6x10⁻³ M propyl mercaptan
-DD' 2.2x10⁻³ M butyl mercaptan
-EE' 4.0x10⁻⁴ M dodecyl mercaptan
FIG: 3

POTENTIAL in mV (VS. S.C.E.)

Log C, D, Amp/Cm²

● AA' 0.5 M CH₂ClCOOH (Blank)

× BB' 3.2x10⁻³ M Ethyl mercaptan

○ CC' 2.6x10⁻³ M Propyl mercaptan

△ DD' 8.8x10⁻⁴ M Butyl mercaptan

□ EE' 4.0x10⁻⁴ M Dodecyl mercaptan
Influence of current density on anode and cathode potential

AA' 0.5 M CHCl₂COOH (Blank)
0.5 M CHCl₂COOH containing
BB' 3.2x10⁻³ M Ethyl mercaptan
CC' 2.6x10⁻³ M Propyl mercaptan
DD' 4.4x10⁻⁴ M Butyl mercaptan
EE' 4.0x10⁻⁴ M Dodecyl mercaptan

Fig. 4

POTENTIAL in mV (V.S.S.C.E.)

Log C.D. Amp/Cm²
FIG. 5

Effect of C.M. on cathode and anode potential

- A: 0.5 M CCl₃COOH (Blank) 7.5 M CCl₃COOH containing
- B: 3.2 x 10⁻³ M Ethyl mercaptan
- C: 2.6 x 10⁻³ M Prityl mercaptan
- D: 2.2 x 10⁻³ M Butyl mercaptan
- E: 4.0 x 10⁻⁴ M Dodecyl mercaptan

POTENTIAL in mV (VS. S.C.E.)

Log C.D. Amp / Cm²

Potential in mV (VS. S.C.E.)

-1400
-1200
-800
-400
0
+400
+200
-5.0
-4.0
-3.0
-2.0
Influence of current density on cathode and anode potential.
Influence of current density on anode and cathode potential

- AA': Artificial sea water (Blank)
- BB': 2.6x10^{-3}M propyl mercaptan
- CC': 2.2x10^{-3}M butyl mercaptan
- DD': 9.9x10^{-4}M dodecyl mercaptan
**FIG: 6**

Influence of current densities on cathode and anode potential.

- **AB** - 0.5 M HCl (Blank)
- **BC** - 0.5 M HCl containing 4.4 x 10^{-3} M diethyl sulphide
- **CD** - 1.2 x 10^{-3} M dibenzyl disulphide
Influence of current densities on cathode and anode potential

- **AA'**: 0.5M CH$_3$COOH (blank)
- **C**: 0.5M CH$_3$COOH containing
- **BB'**: 2.4x10$^{-4}$ M Dibenzyldisulphide
- **CC'**: 2.2x10$^{-3}$ M diethyl sulphide
Effect of current density on cathode and anode potential.
FIG. 11

POTENTIAL in mV (VS. S.C.E.)

Log C.D. Amp/Cm²

- AA': 0.5M CHCl₂COOH (Blank)
- BB': 8.8x10⁻⁴M diethyl sulphide
- CC': 1.2x10⁻⁴M dibenzyl disulphide

-5.0 -4.0 -3.0 -2.0
-5.0 -4.0 -3.0 -2.0
-100 +100 +200 +300
FIG. 12.

Effect of C.D. on cathode and anode potential

- AA': 0.5M CCl₃COOH (Blank)
- BB': 2.2x10⁻³M diethyl sulphide
- CC': 1.2x10⁻³M dibenzyl disulphide

POTENTIAL in mV (VS. S.C.E.)

Log C.D. Amp / Cm²

Potential vs. C.D. and Ampere density
Influence of current density on cathode and anode potential

- AA' 3% NaCl (Blank)
- BB' 2.2x10^{-3} M diethyl sulphide
- CC' 1.2x10^{-3} M dibenzyl disulphide
FIG: 14

Influence of current density on cathode and potential

- AA' Artificial sea water
- BB' 2.2x10^-3 diethyl sulphide
- CC' 2.4x10^-4 M dibenzyl disulphide

POTENTIAL IN mV (VS.S.C.E.)

Log C.D. Amp/Cm²
Influence of current densities on cathode and anode potential

- AA' 0.5M HCl (Blank)
- BB' 1.7x10^-3M Benzimidazole
- CC' 1.5x10^-3M 2-methyl benzimidazole
- DD' 1.4x10^-3M 5-6 dimethyl benzimidazole
Influence of current densities on cathode and anode potential

- AA' 0.5 M HCl (Blank)
- BB' 1.7x10^{-3} M benzimidazole
- CC' 1.3x10^{-3} M 2-mercaptobenzimidazole
- DD' 1.2x10^{-3} M 2-mercapto benzo thiazole
FIG: 17
Influence of current densities on cathode and anode potential

- **AA'** 0.5M HCl (Blank)
- 0.5M HCl containing
- **X** - BB' 1.7x10^{-3}M Benzimidazole
- **O** - CC' 1.3x10^{-3}M 2-mercaptobenzimidazole
- **△** - DD' 1.7x10^{-3}M Benzo triazole
Influence of current densities on cathode and anode potential
Influence of current densities on cathode and anode potential

- AA' 0.5M CH₃COOH (Blank)
- BB' 1.7x10⁻³M Benzimidazole
- CC' 1.5x10⁻³M 2-methyl benzimidazole
- DD' 1.4x10⁻³M 5-6 dimethyl benzimidazole
- EE' 1.7x10⁻³M Benzotriazole
Influence of current densities on cathode and anode potential

- $0.5\text{M }\text{CH}_3\text{COOH}$ (Blank)
- $0.5\text{M }\text{CH}_3\text{COOH}$ containing
- $1.7\times10^{-3}\text{M Benzimidazole}$
- $1.3\times10^{-3}\text{M 2-mercaptobenzimidazole}$
- $1.2\times10^{-3}\text{M 2-mercaptobenzothiazole}$

![Graph showing potential vs. log C.D. Amp/Cm² for different solutions and current densities.](image-url)
Effect of current densities on cathode and anode potential

- AA' 0.5 M CH₃COOH (Blank)
- 0.5 M CH₃COOH containing
  - BB' 7.8 x 10⁻⁴ M sulphathiazole
- CC' 1.2 x 10⁻³ M 2-MBT
- DD' 1.7 x 10⁻³ M 2-mercaptothiazoline

<table>
<thead>
<tr>
<th>POTENTIAL IN mV (VS. S.C.E.)</th>
<th>Log C.D. Amp/Cm²</th>
</tr>
</thead>
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<td>-200</td>
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<tr>
<td>-400</td>
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<td>+200</td>
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FIG. 21
Effect of current density on cathode and anode potential.

- **AA**: 0.5M CH₂ClCOOH (Blank)
- **BB**: 7.8x10⁻⁴M sulphathiazole
- **CC**: 1.2x10⁻³M 2-mercaptobenzothiazole
- **DD**: 1.7x10⁻³M 2-mercaptothiazoline

Log C.D. Amp/CM²

Potential in mV (VS, S.C.E.)
Influence of current density on anode and cathode potential

Figure 23

POTENTIAL in mV (VS. S.C.E.)

-1000
-800
-600
-400
-200
+200
+400

Log C.D. Amp / Cm²

B
A
C

A': 0.5 M CHCl₂COOH

B': 1.2x10⁻⁷ M 2-mercaptobenzothiazole

C': 1.7x10⁻⁷ M 2-mercaptothiazoline
Influence of current densities on anode and cathode potential

- AA' 0.5 M CCl₃COOH
  0.5 M CCl₃COOH containing
- BB' 1.2x10⁻³ M 2-mercaptobenzothiazole
- CC' 1.7x10⁻³ M 2-mercaptothiazoline

Log C.D. Amp / Cm²

Potential in mv (VS. S.C.E.)
Influence of current densities on cathode and anode potential

FIG: 25

POTENTIAL in mV (VS. S.C.E.)

-1600
-1200
-800
-400

3% NaCl (Blank)
3% NaCl containing
1.5x10^-3 M Benzimidazole
1.5x10^-3 M 2-methyl benzimidazole
1.4x10^-3 M 5-6 dimethyl benzimidazole

Log C.D. Amp/Cm²

+200
0
-5.0
-4.0
-3.0
-2.0
Influence of current densities on cathode and anode potential

\begin{align*}
\text{POTENTIAL in mV (VS. S.C.E.)} \\
\end{align*}
Influence of current densities on cathode and anode potential
Influence of current density on cathode and anode potential.
Influence of current densities on cathode and anode potential

- **AA'** Artificial sea water (Blank)

- **BB'** $1.7 \times 10^{-3}$M Benzimidazole
- **CC'** $1.5 \times 10^{-3}$M 2-methyl benzimidazole
- **DD'** $1.4 \times 10^{-3}$M 5-6 dimethyl benzimidazole
- **EE'** $1.7 \times 10^{-3}$M Benzotriazole

**Log C.D. Amp / Cm²**

**Potential in mV (vs. S.C.E.)**
Influence of current densities on cathode and anode potential

- AA' Artificial sea water (Blank)
- BB' 1.7x10^{-3} M Benzimidazole
- CC' 1.3x10^{-3} M 2-mercaptobenzimidazole
- DD' 1.2x10^{-3} M 2-mercaptobenzothiazole
Influence of current densities on cathode and anode potential

- **AA'** Artificial sea water (Blank)
- **BB'** 7.8x10^{-4} M sulphathiazole
- **CC'** 1.2x10^{-3} M 2-MBT
- **DD'** 1.7x10^{-3} M 2-mercaptothiazoline

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The diagram shows the relationship between potential (in mV) and logarithm of current density (Amp/Cm²) for different conditions.
Influence of current densities on cathode and anode potential

- AA' 0.5M HCl (Blank)
- BB' 2.6x10⁻³M Thiourea
- CC' 1.3x10⁻³M Phenyl thiourea
- DD' 8.8x10⁻⁴M Dipheryl thiourea
- EE' 1.1x10⁻³M m-chloropheryl thiourea
Influence of current densities on cathode and anode potential

- **AA'** 0.5M HCl (Blank)
- **BB'** 2.6x10^{-3}M thiourea
- **CC'** 1.6x10^{-3}M N-N'-di-p-tolyl thiourea
- **DD'** 1.6x10^{-3}M N-N'-di-p-tolyl thiourea
FIG: 34

Influence of current density on cathode and anode potential

- **AA'** 0.5M HCl (Blank)
- **BB'** 4.4x10^{-4}M N-methyl thiourea
- **CC'** 3.4x10^{-3}M N-allyl thiourea
Influence of current density on cathode and anode potential

0.5M HCl containing

- BB' 2.6x10^{-3}M Thiourea
- CC' 1.4x10^{-3}M N-N' diethyl thiourea
- DD' 2.1x10^{-3}M N-N' dibutyl thiourea
Influence of current densities on cathode and anode potential

- AA': 0.5M CH$_3$COOH (Blank)
- 0.5 M CH$_3$COOH containing
- BB': 2.6x$10^{-4}$ M Thiourea
- CC': 1.3x$10^{-4}$ M Phenyl thiourea
- DD': 1.1x$10^{-3}$ M m-chlorophenyl thiourea
Influence of current density on cathode and anode potential

- **AA'** 0.5M CH₃COOH (Blank)
- 0.5M CH₃COOH containing
- **BB'** 1.1 x 10⁻³ M p-methoxy phenyl thiourea
- **CC'** 1.0 x 10⁻³ M p-nitro phenyl thiourea
- **DD'** 1.8 x 10⁻⁴ M Diphenyl thiourea

**Figure 3**

**Log C.D. Amp/Cm²**

**POTENTIAL in mV (V.S.C.E.)**

-800  -600  -400  -200  +200  +100  0  -5.0  -4.0  -3.0  -2.0
Effect of current densities on cathode and anode potential

- **AA**: 0.5 M CH$_3$COOH (Blank)
- **BB**: 2.6x$10^{-4}$ M Thiourea
- **CC**: 1.6x$10^{-4}$ M N,N'-dio-tolyl thiourea
- **DD**: 7.8x$10^{-5}$ M N,N'-di-p-tolyl thiourea

**Log C.D. Amp/Cm$^2$** vs **Potential in mV (V.S.S.C.E.)**
Influence of current densities on cathode and anode potential

FIG. 39

- AA' 0.5M CH₃COOH (Blank)
- BB' 2.6x10⁻⁴M Thiourea
- CC' 2.8x10⁻⁴M N-N'-diethyl thiourea
- DD' 1.1x10⁻³M N-N'-dibutyl thiourea

Log C.D. Amp / Cm²
Effect of current density on cathode and anode potential

- FIG: 40

- POTENTIAL in mV (VS. S.C.E.)

- Log C.D. Amp / Cm²

- ● - AA' 0.5M CH₂ClCOOH (Blank)
- × - BB' 1.1x10⁻³ M Thiourea
- ○ - CC' 1.3x10⁻³ M Phenylthiourea
- △ - DD' 3.5x10⁻⁴ M Diphenylthiourea
Effect of current density on cathode and anode potential

- **A**: 0.5M CH₂ClCOOH (Blank)
- **BB**: 1.1x10⁻³M Thiourea
- **CC**: 1.3x10⁻³M Phenylthiourea
- **DD**: 1.1x10⁻³M m-chlorophenyl thiourea
FIG. II
Effect of current density on cathode and anode potential

POTENTIAL in mV (VS. S.C.E.)

-1000
-800
-600
-400
-200

Log C.D. Amp/Cm²

-4.0
-3.0
-2.0

+100

+200

-5.0

A

A'

B

B'

C

○ - AA' 0.5M CH₂ClCOOH (Blank)
× - BB' 1.1x10⁻³M p-methoxy phenylthiourea
○ - CC' 1.0x10⁻³M p-nitrophenyliothiourea

0.5M CH₂ClCOOH containing
FIG: 43

Effect of current density on cathode and anode potential

- AA': 0.5 M CH₂ClCOOH (Blank)
- BB': 1.1 x 10⁻³ M Thiourea
- CC': 1.6 x 10⁻⁴ M N-N' di-o-tolyl thiourea
- DD': 7.8 x 10⁻⁴ M N-N' di-p-tolyl thiourea

Log CD Amp/Cm²
Effect of current density on cathode and anode potential

- **A**: 0.5M CH₂ClCOOH (Blank)
- **B**: 0.5M CH₂Cl₆COOH containing
- **C**: 1.1x10⁻³M Thiourea
- **D**: 5.6x10⁻⁴M N-N' diethyl thiourea
- **E**: 1.1x10⁻³M N-N' dibutyl thiourea
Effect of C.D. on cathode and anode potential

- AA' 0.5 M CHCl₂COOH (Blank)  
- 0.5 M CHCl₂COOH containing  
   - x-BB' 1.1x10⁻³ M Thiourea  
   - ○-CC' 1.3x10⁻³ M Phenyl thiourea  
   - △-DD' 8.8x10⁻⁴ M Dipheryl thiourea

Log C.D. Amp / Cm²
FIG: 46

effect of C.D. on cathode and anode
Potential

POTENTIAL in mV (VS. S. C. E.)

-1000
-800
-600
-400
-200
0
+100
+200

-4.0
-3.0
-2.0
0
-5.0

- AA' 0.5M CHCl₂COOH
- BB' 1.1x10⁻³M Thiourea
- CC' 1.3x10⁻³M Phenyl thiourea
- DD' 2.2x10⁻⁴M m-chlorophenyl thiourea

Log C.D. Amp / Cm²
Effect of C.D. on cathode and anode potential

- AA' 0.5M CHCl₂COOH (Blank)
- BB' 2.2x10⁻⁴M p-methoxyphenyl thiourea
- CC' 1.0x10⁻⁳M p-nitrophenyl thiourea
Effect of current density on cathode and anode potential

- AA' 0.5M CHCl₂COOH
- BB' 1.1x10⁻³M Thioxamidene
- CC' 1.6x10⁻⁴M N-N' di-o-tolyl thione
- DD' 7.8x10⁻⁴M N-N' di-p-tolyl thione

Log C.D. Amp/Cm²
**Effect of C.D. on cathode and anode potential**

- **AA'** 0.5M CHCl₂COOH (Blank)
- BB' 1.1x10⁻³M Thiourea
- CC' 5.6x10⁻⁴M N-N' diethyl thiourea
- DD' 1.1x10⁻³M N-N' dibutyl thiourea

Log C.D. Amp/Cm²
Effect of C.D. on cathode and anode potential

- AA': 0.5M CCl₃COOH (Blank)
- BB': 2.6x10⁻⁴ M Thiourea
- CC': 5.3x10⁻¹ M Phenyl thiourea
- DD': 8.8x10⁻⁵ M Dipheryl thiourea

Log C.D. Amp / Cm²
Effect of C.D. on cathode and anode potential
Effect of C.D. on cathode and anode potential

- AA, 0.5M CCl₃COOH (Blank)
- BB, 4.4x10⁻⁴M p-methoxy phenyl thiourea
- CC, 1.0x10⁻³M p-nitrophenyl thiourea
FIG. 53

Effect of C.D. on cathode and anode potential

- AA: 0.5M CCl₃COOH (Blank)
0.5M CCl₃COOH containing
- BB: 2.6x10⁻⁴M Thiourea
- CC: 7.8x10⁻⁴M N-N' di-o-tolyl thiourea
- DD: 7.8x10⁻⁴M N-N' di-p-tolyl thiourea

POTENTIAL in mV (VS. S.C.E.)

Log C.D. Amp/Cm²
FIG. 5

Effect of C.D. on cathode and anode potentials

- AA: 0.5M CCl₃COOH (Blank)
- BB: 2.6x10⁻¹M Thiourea
- CC: 1.4x10⁻³M N-N' diethyl thiourea
- DD: 1.1x10⁻³M N-N' dibutyl thiourea

POTENTIAL in mV (VS. S.C.E.)

Log C.D. Amp/Cm²

-5.0 -4.0 -3.0 -2.0
+200 +100
Influence of current density on cathode and anode potentials

- AA' 3% NaCl (Blank)
- BB' 2.6x10^{-3} M Thiourea
- CC' 1.3x10^{-3} M phenylthiourea
- DD' 8.8x10^{-4} M diphenylthiourea
Influence of current densities on cathode and anode potential

- $AA'$ 3% NaCl (Blank)
- $BB'$ 2.6x$10^{-3}$M Thiourea
- $CC'$ 1.3x$10^{-3}$M Phenyl thiourea
- $DD'$ 1.1x$10^{-3}$M m-chlorophenyl thiourea
Influence of current densities on cathode and anode potential

- AA' 3% NaCl (Blank)
- BB' 2.6 x 10^{-3} M Thiourea
- CC' 7.8 x 10^{-4} M N-N' di-o-tolyl thiourea
- DD' 7.8 x 10^{-4} M N-N' di-p-tolyl thiourea
Influence of current density on cathode and anode potential

- **A** - 3% NaCl (Blank)
- **BB** - 2.6x10^{-3} M Thiourea
- **CC** - 1.4x10^{-4} M N-N diethyl thiourea
- **DD** - 1.1x10^{-3} M N-N dibutyl thiourea
Influence of current density on cathode and anode potential
FIG: 60

Influence of current density on cathode and anode potential

- **AA'** Artificial sea water (Blank)
- **BB'** 2.6x10^-4 M Thiourea
- **CC'** 1.3x10^-3 M Phenyl thiourea
- **DD'** 1.1x10^-3 M m-chlorophenyl thiourea

POTENTIAL IN MV (VS. S.C.E.)

Log C.D. Amp/Cm²
Influence of current density on cathode and anode potential

- **AA**' Artificial sea water (Blank)
- **BB**' Artificial sea water containing $2.6 \times 10^{-4}$ M thiourea
- **CC**' 7.8$ \times 10^{-4}$ M N-N'-di-o-tolyl thiourea
- **DD**' 7.8$ \times 10^{-4}$ M N-N'-di-p-tolyl thiourea
Influence of current density on cathode and anode potential

- AA' Artificial sea water (Blank)
- BB' 2.6x10^-4 M Thiourea
- CC' 5.6x10^-4 M N-N' diethyl thiourea
- DD' 1.1x10^-3 M N-N' dibutyl thiourea