

**Effect of Temperature on the Corrosion Inhibition of Trans-4-hydroxy-4'-  
stilbazole on Mild Steel in HCl Solution**

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**ABSTRACT**

The inhibition and the effect of temperature and concentration of trans-4-hydroxy-4'-stilbazole on the corrosion of mild steel in 1M HCl solution was investigated by weight loss experiments at temperatures ranging from 303 to 343 K. The studied inhibitor concentrations were between  $1 \times 10^{-7}$  M and  $1 \times 10^{-3}$  M. The percentage inhibition increased with the increase of the concentration of the inhibitor. The percentage inhibition reached about 94% at the concentration of  $1 \times 10^{-3}$  M and 303 K. On the other hand the percentage inhibition decreased with the increase of temperature. Using the Temkin adsorption isotherm, the thermodynamic parameters for the adsorption of this inhibitor on the metal surface were calculated. Trans-4-hydroxy-4'-stilbazole was found to be a potential corrosion inhibitor since it contained not only nitrogen and oxygen, but also phenyl and pyridine rings that are joined together with a double bond ( $-C=C-$ ) in conjugation with these rings.

**KEYWORDS:**

Corrosion, inhibitor, trans-4-hydroxy-4'-stilbazole, Temkin adsorption isotherm.

## INTRODUCTION

Organic compounds containing polar groups by which the molecule can become strongly or specifically adsorbed on the metal surface constitute most organic inhibitors [1,2]. These inhibitors, which include the organic N, P, S, and OH groups, are known to be similar to catalytic poisons, as they decrease the reaction rate at the metal/solution interface without, in general, being involved in the reaction considered. It is generally accepted that most organic inhibitors act via adsorption at the metal/solution interface. The mechanism by which an inhibitor decreases the corrosion current is achieved by interfering with some of the steps for the electrochemical process.

The corrosion inhibition of mild steel in aggressive acidic solutions has been widely investigated. In industries, hydrochloric acid solutions are often used in order to remove scale and salts from steel surfaces, and cleaning tanks and pipelines. This treatment may be prerequisite for coating by electroplating, galvanizing or painting techniques. The acid must be treated to prevent an extensive dissolution of the underlying metal. This treatment involves the addition of some organic inhibitors to the acid solution that adsorb at the metal/solution interface by displacing water molecules on the surface and forming a compact barrier film.

Many authors have used various nitrogen-containing compounds in their corrosion inhibition investigations. These compounds included quaternary ammonium salts [3-10], polyamino-benzoquinone polymers [11], azoles [12], substituted aniline-N-salicylidenes [13], amides [14], heterocyclic compounds [15,16], and cationic surfactants [17, 18]. Other authors worked on sulfur-containing inhibitors [19-24]. Other studies involved the effect of addition of some ions on the inhibition efficiency of some organic compounds. These ions included chromium [25], iodide [26], and chloride [27]. Structural effect of organic compounds as corrosion inhibitors was also studied [28,29]. In all these studies, the nitrogen atom(s) in the compounds showed to be able to absorb very well on the metal surface and form protective layer, which in

turns increased the corrosion inhibition with the increase in the concentration of the inhibitor, in some cases reaching 99% inhibition [5].

No studies have been reported on trans-4-hydroxy-4'-stilbazole inhibitor employed in this current work, in terms of studying the effect of temperature on the corrosion inhibition of mild steel in 1M HCl solution. Mild steel was chosen in our studies since high temperature aggressive acids are widely used in industries in connection to mild and low alloy steels.

In this work, trans-4-hydroxy-4'-stilbazole was prepared via a synthesis which produced a high percentage yield of this pure compound.

The aim of this work is to study, using weight loss measurements, the effect of temperature on the corrosion inhibition of mild steel in 1M HCl solution by trans-4-hydroxy-4'-stilbazole, a synthesized compound that was previously studied using electrochemical technique [30] and found to be a very efficient corrosion inhibitor, and to calculate the thermodynamic parameters. Hence, the output of this study is intended to be the building block or the nucleus for a new family or group of stilbazole derivatives in all studies of corrosion inhibitors.

## **EXPERIMENTAL DETAILS**

### *INSTRUMENTATION*

The experimental set-up consisted of a 250-mL round bottom flask fitted with a reflux condenser and a long glass rod on which the specimen was hooked and in turn immersed in a thermally controlled water bath [20, 31].

Analytical-grade hydrochloric acid (Ajax), 4-iodophenol, triethylamine, palladium acetate, tri(o-tolyl)phosphine, acetonitrile, dichloromethane, MgSO<sub>4</sub>, hexane, and pentane were obtained from Aldrich Chemical Company and used without further

purification. 4-Vinylpyridine was distilled under reduced pressure and stored in cold place.

### *SAMPLE PREPARATION*

Rectangular specimens (1 cm x 2.3 cm x 0.3 cm) cut from large sheet of 3 mm thick mild steel (IS 226 containing 0.18 % C, 0.6 % Mn, and 0.35 % Si) supplied by “Reliable Steel Traders”, Sharjah, UAE; were used for weight loss measurements. A 2-mm diameter hole was drilled close to the upper edge of the specimen and served to be hooked with a glass rod for immersion purposes. Prior to each experiment, the specimens were polished with 600 grade emery paper, rinsed with distilled water, degreased with acetone, dried, and finally weighed precisely on an accurate analytical balance.

### *SYNTHESIS OF TRANS-4-HYDROXY-4'-STILBAZOLE*

Several methods for the synthesis of trans-4-hydroxy-4'-stilbazole are available including the reaction of 4-picoline with 4-Hydroxy benzaldehyde in acetic anhydride [32,33] which, under such conditions, gave only tiny yields after long reactions. A better procedure was developed and which used Heck type vinylic addition reaction [34] catalyzed by palladium. Using this procedure we were able to obtain a yield of about 80% and quantities reaching up to 20 g in one single reaction, following the modified procedure set by Bruce et al. [35] and described in our previous work [30].

### *MEASURING PROCEDURE*

The flask was filled with 100 mL of 1M HCl solution either with or without trans-4-hydroxy-4'-stilbazole of various concentrations, then placed in water bath. As soon as the required working temperature was reached, the mild steel sample was immersed in the solution, and left there for exactly six hours, after which the sample was removed, rinsed with distilled deionized water, degreased with acetone, dried, and finally weighed precisely on an accurate analytical balance. This procedure was repeated

with all the samples with a variety of inhibitor concentrations ranging from  $1 \times 10^{-7}$  M up to  $1 \times 10^{-3}$  M; and at temperatures ranging from 303 K to 343 K.

## RESULTS

Weight loss corrosion tests were carried out on the mild steel in 1M HCl in the absence or presence of trans-4-hydroxy-4'-stilbazole over a period of 6 hours. Table 1 represents the corrosion rates [ $\text{mg.cm}^{-2}.\text{h}^{-1}$ ], and the percentage efficiencies for the studied inhibitor with concentrations varying from  $1 \times 10^{-7}$  M to  $1 \times 10^{-3}$  M at 303, 313, 323, 333, and 343 K, respectively. The percentage efficiency was calculated according to the following expression:

$$\% \text{ Inhibition} = \frac{W_{Uninh.} - W_{Inh.}}{W_{Uninh.}} \times 100 \quad (1)$$

Where:  $W_{Uninh.}$  = corrosion rate without inhibitor; and

$W_{Inh.}$  = corrosion rate with inhibitor.

Figures 1 and 2 show the plots of the corrosion rate of trans-4-hydroxy-4'-stilbazole as a function of concentration at 303, 313, 323, 333, and 343 K. At 303 K (Figure 1), the corrosion rate dropped from  $0.961 \text{ mg.cm}^{-2}.\text{h}^{-1}$  (1M HCl in the absence of the inhibitor) to  $0.950 \text{ mg.cm}^{-2}.\text{h}^{-1}$  when  $1 \times 10^{-7}$  M of trans-4-hydroxy-4'-stilbazole was present in the 1M HCl. This corrosion rate continued to decrease slightly to reach  $0.714 \text{ mg.cm}^{-2}.\text{h}^{-1}$  (26 % inhibition) at a concentration of  $1 \times 10^{-5}$  M, followed by a steep decrease to reach  $0.191 \text{ mg.cm}^{-2}.\text{h}^{-1}$  when the inhibitor's concentration was  $1 \times 10^{-4}$  M; and finally, at higher concentration ( $1 \times 10^{-3}$  M) the corrosion rate as initially decreased slightly to reach  $0.060 \text{ mg.cm}^{-2}.\text{h}^{-1}$  (94 % inhibition). At 313 K (Figure 1), the curve showed to have similar shape as that obtained at 303 K. At concentrations greater than  $1 \times 10^{-5}$  M, the corrosion rate decreased steeply and reached about  $0.112 \text{ mg.cm}^{-2}.\text{h}^{-1}$  (92 %) at  $1 \times 10^{-3}$  M.

At 323 K (Figure 2), when the concentration of the inhibitor was between  $1 \times 10^{-7}$  M and  $1 \times 10^{-5}$  M, it had very slight effect on the corrosion rate; whereas, at higher concentrations, the corrosion rate dropped from  $4.192 \text{ mg.cm}^{-2}.\text{h}^{-1}$  (at  $1 \times 10^{-5}$  M) down to 2.179 and  $0.543 \text{ mg.cm}^{-2}.\text{h}^{-1}$  at  $1 \times 10^{-4}$  M and  $1 \times 10^{-3}$  M, respectively.

At 333 K (Table 1), the presence of trans-4-hydroxy-4'-stilbazole acted as an accelerator between  $1 \times 10^{-7}$  M and  $1 \times 10^{-5}$  M, and at concentrations of  $1 \times 10^{-4}$  M and greater, it acted as inhibitor reaching percentage inhibitions of 27% and 81% at  $1 \times 10^{-4}$  M and  $1 \times 10^{-3}$  M, respectively. At 343 K, likewise, trans-4-hydroxy-4'-stilbazole acted as an accelerator between  $1 \times 10^{-7}$  M and  $1 \times 10^{-6}$  M, then inhibited the mild steel at  $1 \times 10^{-5}$  M and greater concentration, reaching a percentage inhibition of about 67 % at  $1 \times 10^{-3}$  M. It was noticed that a tenfold increase in the inhibitor's concentration, namely from  $1 \times 10^{-4}$  M to  $1 \times 10^{-3}$  M, had a very sizeable increase in the percentage inhibition as this increased from 27 % to 81 % at 333 K, and from 8.6 % to 67 % at 343 K.

Figure 3 shows the plots of the percent inhibition versus the concentration of the inhibitor at temperatures of 303, 313, 323, 333, and 343 K, respectively. This figure showed that the percent inhibition was not sufficiently affected by the increase of temperature (303 to 323K), especially when low concentrations ( $1 \times 10^{-7}$  to  $1 \times 10^{-5}$  M) of inhibitors were employed; whereas at higher concentrations (above  $1 \times 10^{-5}$  M), the presence of the inhibitor greatly increased the percent inhibition at all temperatures. At higher temperatures, it was noticed that at concentrations between  $1 \times 10^{-7}$  M to  $1 \times 10^{-4}$  M (343 K) and in some cases to  $1 \times 10^{-5}$  M (333 K) trans-4-hydroxy-4'-stilbazole acted as a corrosion accelerator.

The data obtained from the weight loss measurements were plotted in accordance to Arrhenius equation:

$$\ln \text{rate} = -\frac{E_a}{RT} + \text{const.} \quad (2)$$



Where:  $E_a$  = activation energy [ $\text{kcal.mol}^{-1}$ ],  $R$  = gas constant [ $\text{kcal.mol}^{-1}$ ],  
 $T$  = absolute temperature [K], and const. = constant

Figure 4 represents the Arrhenius plot of the corrosion of mild steel in 1M HCl solution ( $\ln$  corrosion rate as a function of  $1/T$ ) with or without the presence of trans-4-hydroxy-4'-stilbazole at concentrations ranging from  $1 \times 10^{-7}$  M to  $1 \times 10^{-3}$  M. From this figure, the slope ( $-\frac{E_a}{R}$ ) of each individual line was determined and used to calculate the activation energy according to equation 2, and taking  $R = 1.987 \times 10^{-3} \text{ kcal.mol}^{-1}$  (Table 2). The increase of concentration of trans-4-hydroxy-4'-stilbazole (from  $1 \times 10^{-7}$  M to  $1 \times 10^{-3}$  M), increased the activation energies for the corrosion of mild steel in 1M HCl (initially  $18.27 \text{ kcal.mol}^{-1}$ ) (Table 2).

Table 3 shows the surface coverage of various concentrations of trans-4-hydroxy-4'-stilbazole (from  $1 \times 10^{-7}$  M to  $1 \times 10^{-3}$  M) on mild steel surface as a function of temperature. These values were extracted from the corresponding percent efficiency values reported earlier in Table 1. The plot of surface coverage,  $\theta$ , against the natural logarithm of the concentration of the inhibitor,  $\ln C$ , for mild steel at various inhibitor temperatures is shown in Figure 5. After examining these data and adjusting them to different theoretical adsorption isotherms, it was concluded that the inhibitor was adsorbed on the mild steel surface according to Temkin isotherm:

$$-2a \theta = \ln K C \quad (3)$$

Where:  $a$  = molecular interaction constant,  
 $\theta$  = degree of coverage,  
 $K$  = equilibrium constant for the adsorption reaction, and  
 $C$  = concentration of the inhibitor.

The equilibrium constant for the adsorption reaction,  $K$ , is related to the standard free energy of adsorption [1] via equation 4:



$$K = \frac{1}{55.5} \exp\left(-\frac{\Delta G}{RT}\right) \quad (4)$$

Where: K = equilibrium constant for the adsorption reaction,  
55.5 = concentration of water [mol.L<sup>-1</sup>],  
ΔG = standard free energy [kcal.mol<sup>-1</sup>],  
R = gas constant [kcal.mol<sup>-1</sup>], and  
T = absolute temperature [K].

According to equation 3, the straight lines shown in Figure 5 will have the following slopes and intercepts:

$$\text{Slope} = -\frac{1}{2a} \quad (5)$$

$$\text{Intercept} = -\frac{1}{2a} \ln K \quad (6)$$

Combination of equations (5) and (6) leads to the following relationship:

$$\text{Intercept} = \text{Slope} \cdot (\ln K) \quad (7)$$

from which the equilibrium constant for the adsorption reaction, K, can be calculated:

$$K = e^{\left(\frac{\text{Intercept}}{\text{Slope}}\right)} \quad (8)$$

The free energy of adsorption of the inhibitor, ΔG, can be calculated from the results in Figure 5 used to calculate the equilibrium constant, K, and equation 4 at various temperatures (303 K to 323 K).

The enthalpy of adsorption, ΔH, for the inhibitor can be calculated from the following equation:

$$\Delta H = E_a - RT \quad (9)$$

The entropy,  $\Delta S$ , can be calculated at various temperatures for the inhibitor using the following equation :

$$\Delta G = \Delta H - T\Delta S \quad (10)$$

## DISCUSSION

The results in Table 2, show that the activation energy ( $E_a$ ) for the corrosion of mild steel in the presence of the inhibitor are higher compared to the activation energy in the absence of inhibitor at both  $1 \times 10^{-3}$  M and  $1 \times 10^{-4}$  M (about 27 vs. about 18 kcal.mol<sup>-1</sup>). Whereas, at lower concentration (from  $1 \times 10^{-5}$  to  $1 \times 10^{-7}$  M), the activation energy is very close to that in uninhibited HCl solution. This can be attributed to the fact that higher values of  $E_a$  in the presence of inhibitor compared to its absence are generally consistent with a physisorption, while unchanged or lower values of  $E_a$  in inhibited solution suggest charge sharing or transfer from the organic inhibitor to the metal surface to form coordinate covalent bonds (chemisorption).

The increase in the activation energies for the corrosion is attributed to a decrease in the adsorption of the inhibitor on the metal surface as the temperature increased; and subsequently, an increase in the corrosion rate will result due to the greater exposed area of the metal surface to the acid.

Tables 4 to 6 show the thermodynamic data obtained in the presence of the inhibitor at various temperatures. These thermodynamic quantities represent the algebraic sum of the values for adsorption and desorption. The negative value of  $\Delta G$  indicates the spontaneous adsorption of inhibitor on the surface of the mild steel. The free energy,  $\Delta G$ , varies from -11.54 kcal.mol<sup>-1</sup> at 303 K to -11.12 kcal.mol<sup>-1</sup> at 323 K. The adsorption process is believed to be exothermic and associated with a decrease in entropy ( $\Delta S$ ) of solute, while the opposite is true for the solvent [24]. The gain in

entropy which accompanies the substitutional adsorption process is attributed to the increase in the solvent entropy (Table 6). This agrees with the general suggestion that the values of  $\Delta G$  increase with the increase of inhibition efficiency [13,36] as the adsorption of organic compound is accompanied by desorption of water molecules off the surface.

The electron density group ( $-\text{OH}$ ) on the phenyl ring gives an extra inhibition effect. The high inhibition efficiency may be attributed to the preferred flat orientation of this compound on the metal surface. The interaction occurs between the delocalized  $\pi$ -electrons of the two rings, the double bond ( $-\text{C}=\text{C}-$ ), and the lone pair of electrons on N and O atoms with the positively charged metal surface. In addition, ( $-\text{C}=\text{C}-$ ) bond plays the major role in the stabilization of both sides of the Stilbazole structure.

These results agree with Fouada et al. [22] who suggested that the inhibition efficiency of organic compounds depends on many factors including their charge density, number of adsorption sites, heat of hydrogenation, mode of interaction with the metal surface, and formation of metallic complexes [37].

## CONCLUSION

Trans-4-hydroxy-4'-stilbazole was found to be a highly efficient inhibitor for mild steel in 1.0 M HCl solution, reaching about 94 % at  $1.0 \times 10^{-3}$  M and 303 K, a concentration considered to be very moderate.

Trans-4-hydroxy-4'-stilbazole is a potential corrosion inhibitor since it contains not only nitrogen and oxygen, but also phenyl and pyridine rings that are joined together with a double bond ( $-\text{C}=\text{C}-$ ) in conjugation with these rings. It was apparent from the molecular structure that this compound would be adsorbed onto the metal surface through the lone pair of electron of nitrogen and oxygen and pi electrons of the two aromatic rings (phenyl and pyridine) and the double bond that join them together.

The percentage of inhibition in the presence of this inhibitor was decreased with temperature which indicates that physical adsorption was the predominant inhibition mechanism because the quantity of adsorbed inhibitor decreases with increasing temperature.

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Concentration of Inhibitor	Temperature / K									
	303		313		323		333		343	
	Corr. Rate	% Effic- iency	Corr. Rate	% Effic- iency	Corr. Rate	% Effic- iency	Corr. Rate	% Effic- iency	Corr. Rate	% Effic- iency
1M HCl	0.961	—	1.394	—	4.671	—	12.225	—	26.280	—
1M HCl + $1 \times 10^{-7}$ M	0.950	1	1.381	0.9	4.648	0.5	14.057	-15	28.139	-7.1
1M HCl + $1 \times 10^{-6}$ M	0.916	5	1.343	3.7	4.570	2.2	12.733	-4	27.061	-3.0
1M HCl + $1 \times 10^{-5}$ M	0.714	26	1.203	14	4.192	10.3	12.714	-4	24.901	5.2
1M HCl + $1 \times 10^{-4}$ M	0.191	80	0.361	74	2.179	53.4	8.959	27	24.020	8.6
1M HCl + $1 \times 10^{-3}$ M	0.060	94	0.112	92	0.543	88.4	2.321	81	8.619	67

**Table 1.** Effect of concentration of trans-4-hydroxy-4'-stilbazole on the corrosion rate ( $\text{mg.cm}^{-2}.\text{h}^{-1}$ ) and percentage efficiency of mild steel in 1M HCl at various temperatures.

System	Activation Energy, $E_a$ (kcal.mol <sup>-1</sup> )				
	1x10 <sup>-3</sup> M	1x10 <sup>-4</sup> M	1x10 <sup>-5</sup> M	1x10 <sup>-6</sup> M	1x10 <sup>-7</sup> M
1M HCl	18.27	18.27	18.27	18.27	18.27
1M HCl + trans-4-hydroxy-4'-stilbazole	26.93	26.82	19.72	18.76	18.93

**Table 2.** The activation energy ( $E_a$ ) for the corrosion of mild steel in 1M HCl with and without trans-4-hydroxy-4'-stilbazole inhibitor at various concentrations.

	Temperature / K		
	303	313	323
Concentration of Inhibitor	Surface Coverage $\theta$	Surface Coverage $\theta$	Surface Coverage $\theta$
1M HCl + $1 \times 10^{-6}$ M	0.05	0.037	0.022
1M HCl + $1 \times 10^{-5}$ M	0.26	0.14	0.103
1M HCl + $1 \times 10^{-4}$ M	0.80	0.74	0.534
1M HCl + $1 \times 10^{-3}$ M	0.94	0.92	0.884

**Table 3.** Effect of concentration of trans-4-hydroxy-4'-stilbazole on surface coverage for mild steel in 1M HCl at various temperatures.

$\Delta H, \text{kcal.mol}^{-1}$				
303 K	313 K	323 K	333 K	343 K
26.33	26.31	26.29	26.27	26.25

**Table 4.** The enthalpy of adsorption ( $\Delta H$ ) for mild steel in 1M HCl in the presence of  $1 \times 10^{-3}$  M trans-4-hydroxy-4'-stilbazole inhibitor at various temperatures (303 K – 343 K).

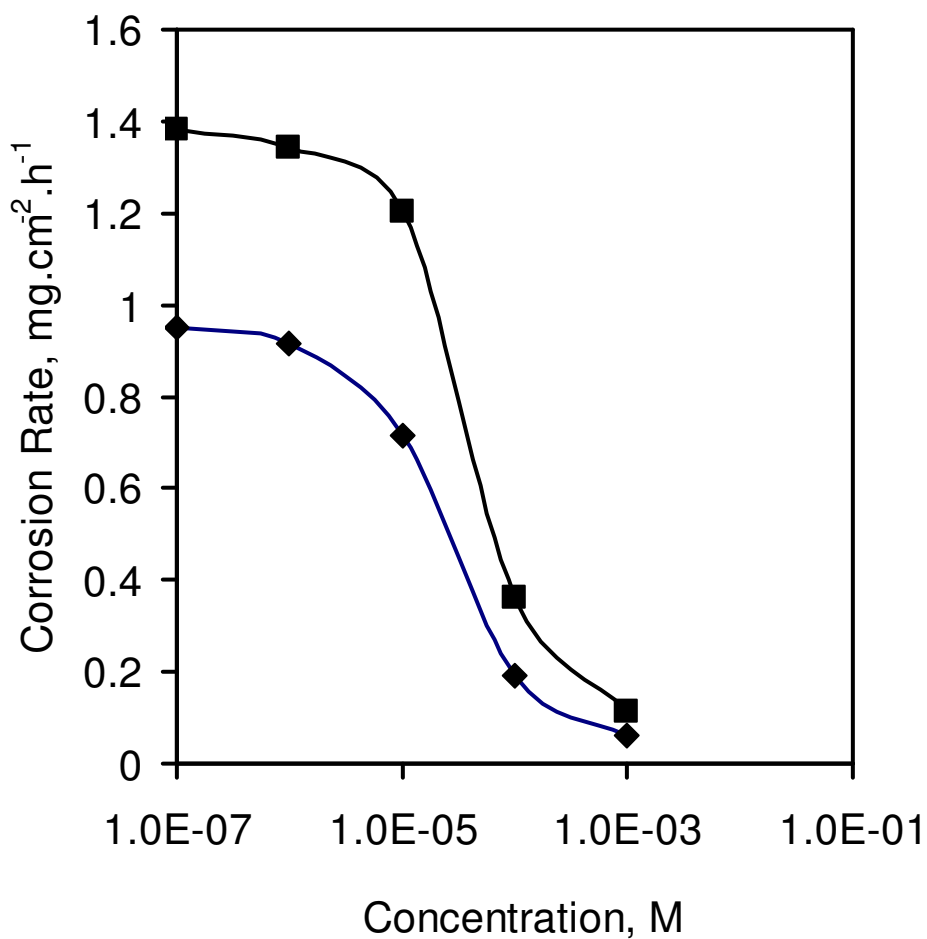
$\Delta G, \text{kcal.mol}^{-1}$		
303 K	313 K	323 K
-11.54	-11.76	-11.12

**Table 5.** The Free energy of adsorption ( $\Delta G_{\text{ads}}$ ) for mild steel in 1M HCl in the presence of trans-4-hydroxy-4'-stilbazole inhibitor at various temperatures (303 K – 323 K).

$\Delta S, \text{kcal. K}^{-1}.\text{mol}^{-1}$		
303 K	313 K	323 K
0.125	0.122	0.116

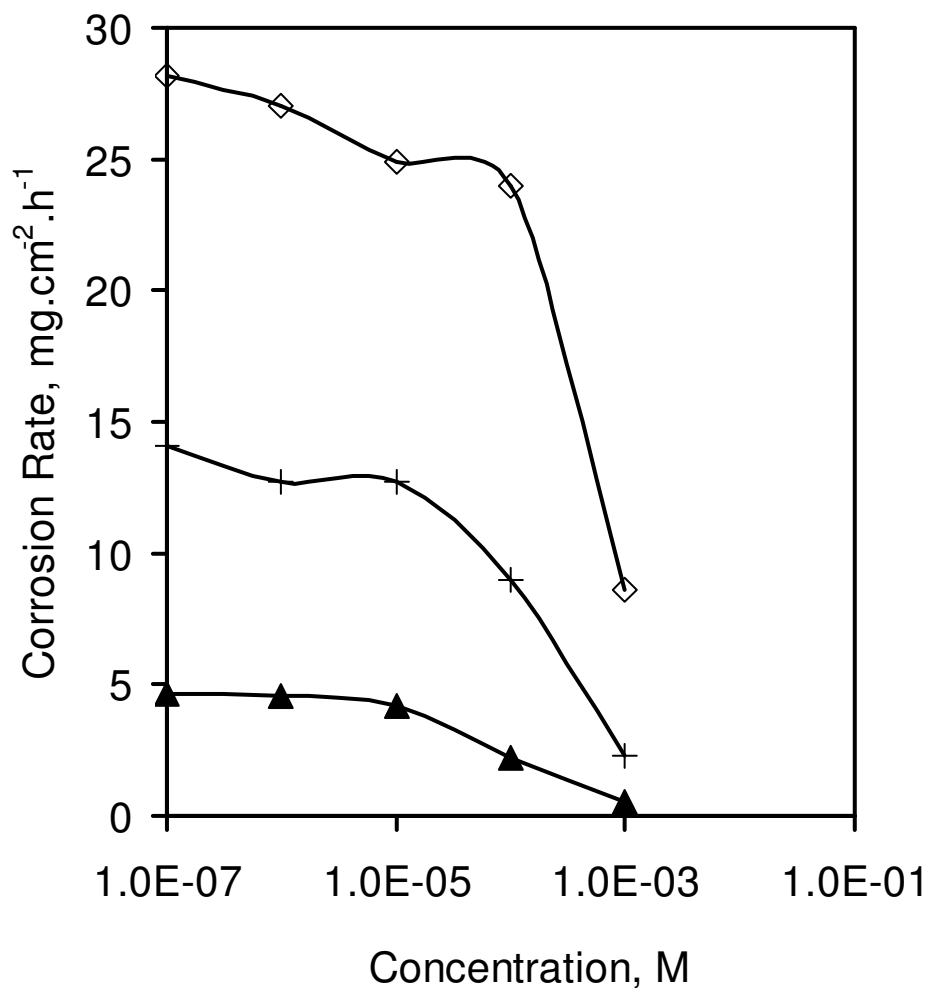
**Table 6.** The change in entropy ( $\Delta S$ ) for mild steel in 1M HCl in the presence of trans-4-hydroxy-4'-stilbazole inhibitor at various temperatures (303 K - 323 K).

**Figure 1**

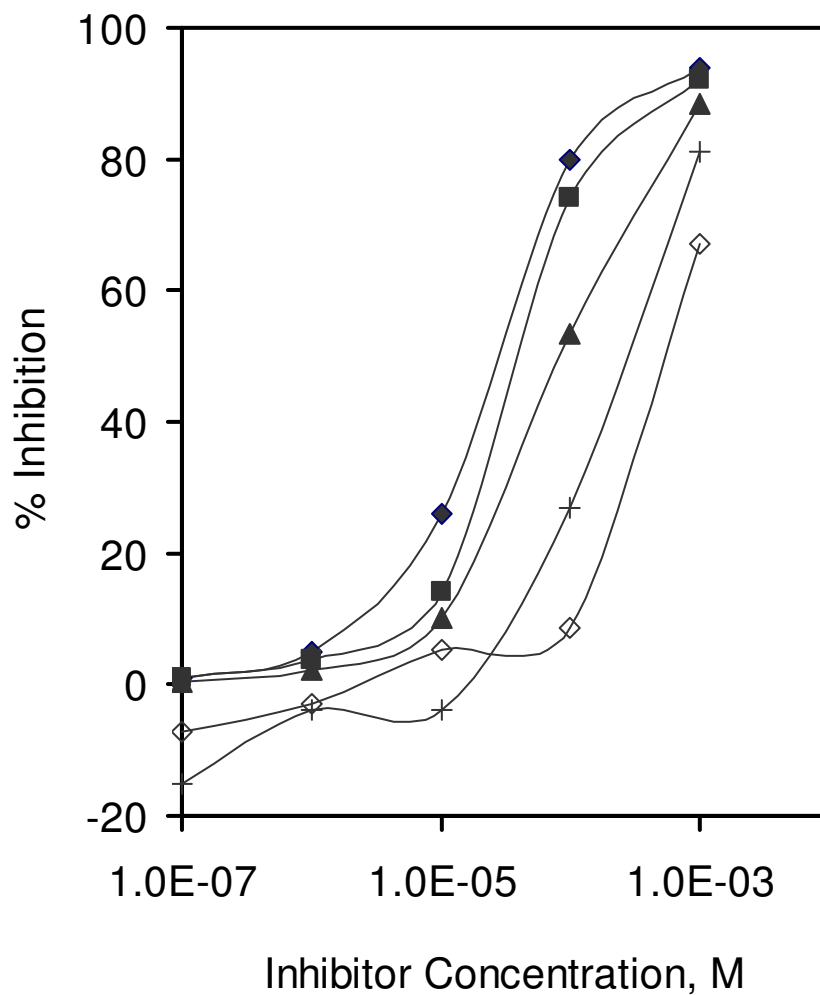




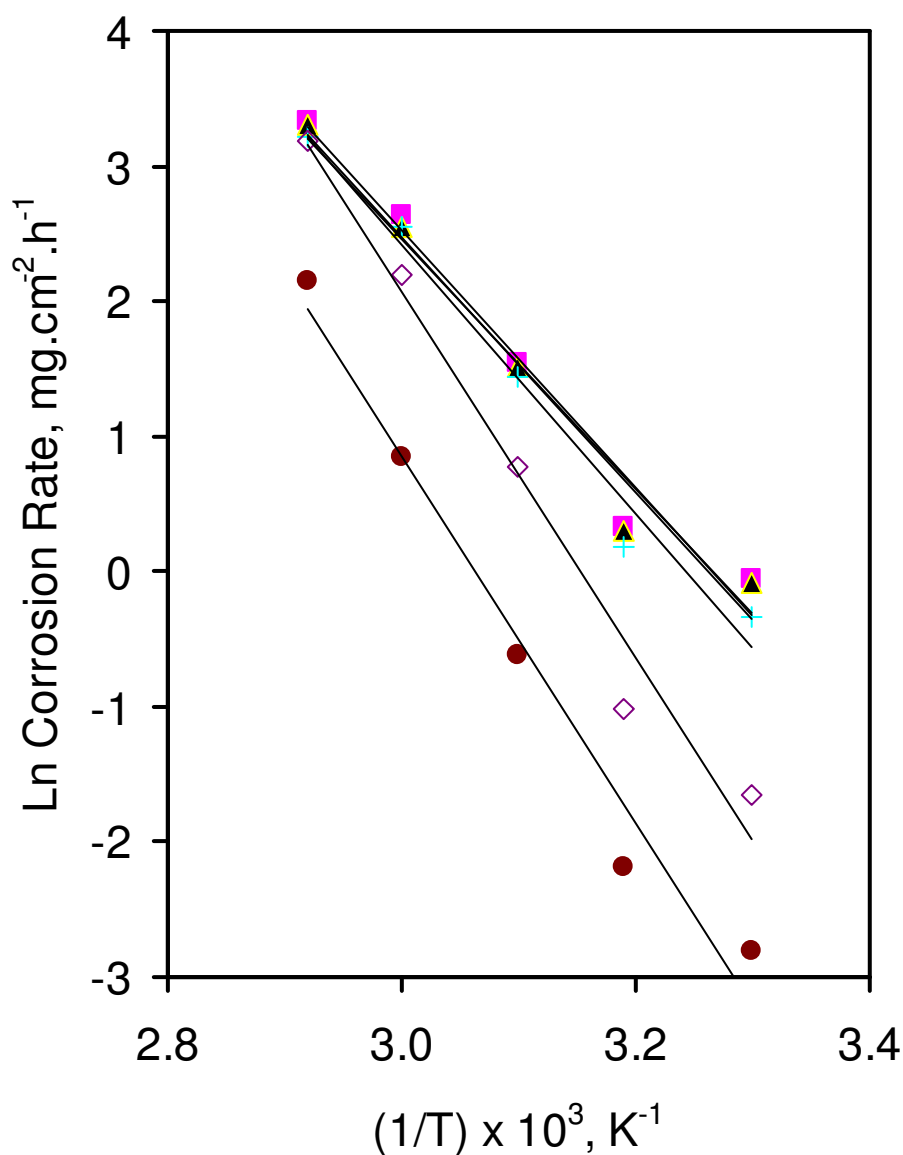
**Figure 2**



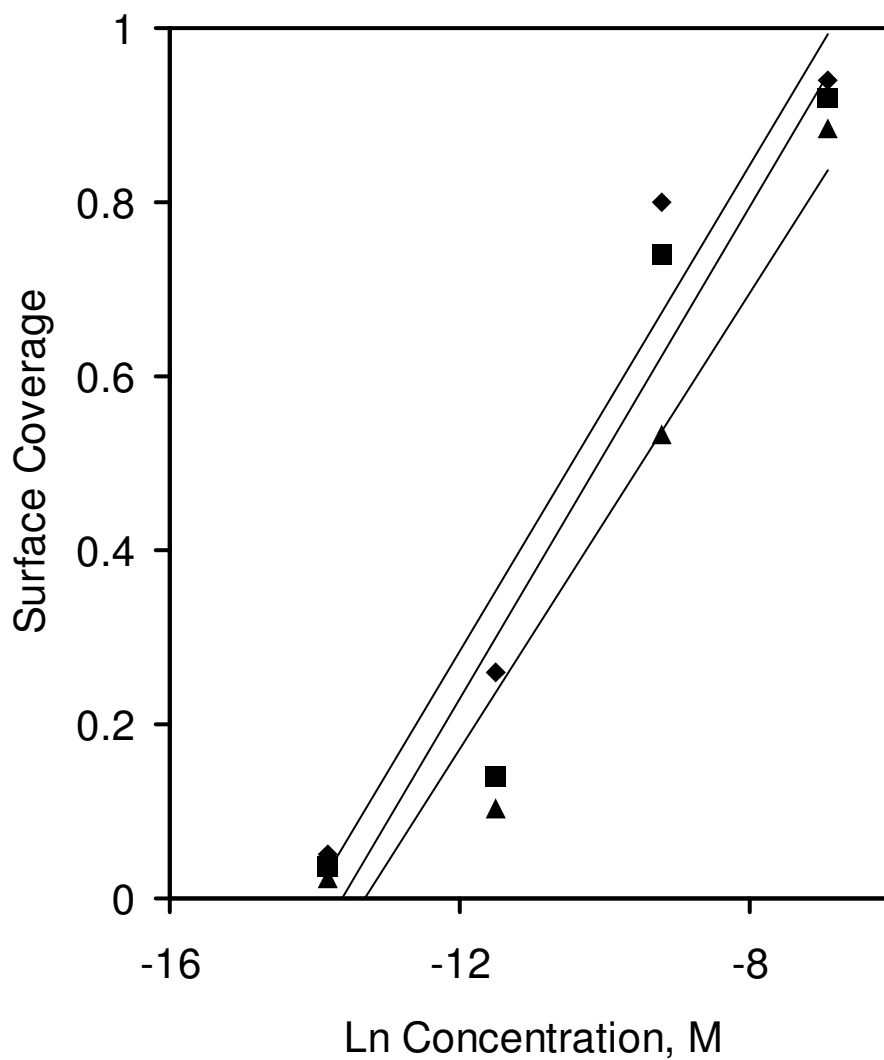
**Figure 3**



**Figure 4**



**Figure 5**



**Figure 1.** Effect of concentration of trans-4-hydroxy-4'-stilbazole on the corrosion rate ( $\text{mg.cm}^{-2}.\text{h}^{-1}$ ) of mild steel in 1M HCl at various temperatures.

◆ 303 K                      ■ 313 K

**Figure 2.** Effect of concentration of trans-4-hydroxy-4'-stilbazole on the corrosion rate ( $\text{mg.cm}^{-2}.\text{h}^{-1}$ ) of mild steel in 1M HCl at various temperatures.

▲ 323 K                      + 333 K                      ◇ 343 K

**Figure 3.** Effect of concentration of trans-4-hydroxy-4'-stilbazole on the percent inhibition of mild steel in 1M HCl at various temperatures.

◆ 303 K              ■ 313 K              ▲ 323 K              + 333 K              ◇ 343 K

**Figure 4.** Effect of temperature on the corrosion rate of mild steel in 1M HCl solution with and without the presence of various concentrations of trans-4-hydroxy-4'-stilbazole inhibitor.

◆ 1 M HCl                      ■  $1 \times 10^{-7}$  M                      ▲  $1 \times 10^{-6}$  M  
+  $1 \times 10^{-5}$  M                      ◇  $1 \times 10^{-4}$  M                      ●  $1 \times 10^{-3}$  M

**Figure 5.** Effect of concentration of trans-4-hydroxy-4'-stilbazole on the surface coverage of mild steel in 1M HCl at various temperatures.

◆ 303 K                      ■ 313 K                      ▲ 323 K