**Quaternary aryl phosphonium salts as corrosion inhibitors for iron in HCl**

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**Abstract**

Cathodic and anodic Tafel extrapolation data for the inhibition of iron corroding via hydrogen evolution in deaerated 1 mol dm-3 HCl at 22 oC are obtained for five synthesised phosphonium salts, 4-tolyltriphenyl-phosphonium chloride (TTPPC), 4-phenoltriphenylphosphonium chloride (PTPPC), 4-anilinetriphenylphosphonium bromide (ATPPB), 4-benzyl alcoholtriphenylphosphonium chloride (BATPPC), 4-hlorophenyltriphenylphosphonium bromide (CPTPPB) and two commercially available phosphonium salts, tetraphenylphosphonium bromide (TPPB) and methyltriphenylphosphonium bromide (MTPPB). The inhibitor concentrations ranged from 1 10-3 to 1 10-7 mol dm-3. The inhibition of iron corrosion in 1 mol dm-3 HCl at 22 oC was found to be in the order TPPB > BATPPC > CPTPPB > PTPPC > MTPPB > TTPPC > ATPPB.

**Keywords**: polarisation, Tafel region, voltammetry.

(Approx. 6.000 words, 10 tables, 12 figures, 4 reactions/equations and 34 references).

**Highlights**

**** The preparation of quaternary aryl phosphonium salts is described.

 Possible resonance structure of some quaternary aryl phosphonium salts are presented.

 Mechanistic aspects of corrosion inhibition are discussed.

 Electrochemical and classical techniques are illustrated by data.

**1. Introduction**

Corrosion of metals is a serious problem in many industries, including civil services such as water and sewage supplies. Inhibitors are frequently used in order to prevent or minimize the corrosion. Corrosion increases running costs and reduces plant efficiency, availability, and product quality.

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

The inhibition of iron corrosion in aqueous acidic solutions has been widely investigated. In many industries, hydrochloric acid solutions are used to remove scale and salts from steel surfaces, 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. The treatment involves addition of some organic inhibitors to the acid solution that are adsorbed at the metal/solution interface by displacing water molecules on the surface and forming a compact barrier film.

Many authors have synthesized and used various phosphorus‐containing compounds in their corrosion inhibition investigations. These compounds have included imidazoline double quaternary phosphonium salt [2-9], P,​P'-​bis (triphenylphosphonio) methyl benzophenone dibromide [10], 4-​vinylbenzyl triphenyl phosphonium chloride [11], and aromatic phosphonium salts [12].

In such studies, the phosphorus atom(s) in the compounds were shown to be able to strongly adsorb on the metal surface and form a protective layer which, in turn, increased the corrosion inhibition at higher inhibitor concentration.

In this work, The synthesis, including the preparation of the tris(triphenylphosphine)nickel (Ni[P(C6H5)3]3) catalyst and subsequent preparation of the quaternary aryl phosphonium salts, is considered. In most cases, tetraphenylphosphonium chloride was used as a reference for comparison purposes as its structure can be considered the parent of a family of phosphonium salts. Electrochemical techniques are used to determine inhibitor properties of the synthesized phosphonium salts and two commercially available phosphonium salts.

**2. Experimental details**

**2.1** **Preparation of quaternary aryl phosphonium salts**

The synthesis of quaternary aryl phosphonium compounds involved the use of a nickel (0) complex of triphenylphosphine, tris(triphenylphosphine) nickel, as shown in Scheme 1.



Scheme 1. Structure of tris(triphenylphosphine)nickel, Ni[P(C6H5)3]3

As shown in reaction (1) [13], the nickel catalyst (2) undergoes oxidative addition in the presence of aryl halide (1) to form the arylnickel complex (3). Oxidative addition has also been reported for the synthesis of other arylnickel (0, II) complexes [14,15].



(1)

The arylnickel complex (3) formed in reaction (1) reacts with triphenylphosphine to give the phosphonium salt (4) and regenerate the nickel catalyst (2) via reaction (2).



(2)

The overall reaction can be summarized as:



(3)

The preparation of tris(triphenylphosphine) nickel has been described in the literature [16] (yield 70%). It was reported to be extremely oxygen sensitive and tends to trap solvent when precipitated from solution. It was also reported that the compound decomposes in solution over several hours; even under rigorous oxygen-free conditions and on standing the deep-red solution darkens and a black precipitate was formed. From the information above, it was decided that isolation of the compound would be impractical and thus preparation of tris(triphenylphosphine)nickel and its subsequent reaction with aryl halides was carried out *in-situ*, under nitrogen inert gas blanket.

### 2.2 Preparation of tris(triphenylphosphine)nickel catalysts

Anhydrous nickel chloride (0.0071 mol, 0.99 g) and triphenylphosphine (0.0304 mol, 8.0 g) were placed into a 1000 cm3 quickfit conical flask and refluxed in acetonitrile (240 cm3) for one hour. The reaction mixture was allowed to cool to room temperature then zinc (0.0117 mol, 0.76 g) was added. The mixture was stirred under nitrogen overnight. A red-brown solid formed and was subsequently used *in-situ* for the preparation of the phosphonium salt.

### 2.3 Preparation of quaternary aryl phosphonium salts [17]

Ethanol (200 cm3), triphenylphosphine (0.05 mol, 13.06 g) and p-aryl halides (0.05 mol) were introduced to Ni[P(C6H5)3]3 (0.005 mol, 4.23 g) whilst the apparatus was purged with nitrogen gas. The boiling mixture was stirred for 24 hours. The solution was filtered and the filtrate volume was reduced using a rotary evaporator (under reduced pressure). The remaining solids were then dissolved in water and the solution was filtered to remove any residues. The resulting solution was treated with diethyl ether to remove any non-ionic organics (mainly unreacted triphenylphosphine). The aqueous solution was evaporated to dryness. The crude phosphonium salts were recrystallized from water (with the exception of 4-anilinetriphenylphosphonium, which was recrystallized from methanol and dried in a vacuum oven at 70 oC overnight). Table 1 lists the prepared phosphonium salts which were evaluated by electrochemical techniques.

FAB-MS (fast atom bombardment-mass spectroscopy) on a Joel JMS-DX303 GC/Mass Spectrometer, IR (infra-red), on a Mattson Instrument Research Series 1 FTIR Spectrometer and 1H NMR (nuclear magnetic resonance), on a Joel GSX 270 MHz Fourier Transform NMR Spectrometer, spectra were obtained for each compound. Melting points were obtained for each compound and the yields are shown in Table 2.

**2.4 Working electrode preparation**

Iron metal (purity 99.5%) used was obtained, in sheet form, from Goodfellow and included. The 4 mm diameter working electrode material was punched from sheet material. The working electrode surface was prepared initially with silicon carbide (corundum), grades 200 to 1200 and followed by polishing with progressively finer grades of alumina slurry (grades 5 m down to 1 m) with absolute alcohol to achieve a reproducible surface finish and rinsed with absolute alcohol before use.

**2.5 Electrolyte preparation**

1M HCl stock solution was made up in 2 dm3 volumes from concentrated HCl (Fisons). Inhibited solutions were prepared by dissolution of the phosphonium salts (Table 1) to produce 500 cm-3 of 110-3 mol.dm-3 solutions. Subsequent dilution by a factor of 10 allowed a range of solutions from 110-3 to 110-7 mol dm-3 to be prepared. All studies was carried out at a temperature of 22 oC using solutions which had been deoxygenated with a fast stream of nitrogen gas for 25 minutes prior to experimentation.

**2.6 Equipment**

Tafel extrapolation data for the inhibition of iron in deaerated 1 mol dm-3 HCl at 22 oC are considered for the five synthesized and two commercially available phosphonium salts solutions at concentrations ranging from 110-3 to 110-7 mol dm-3.

All electrochemical measurements were performed using an EG & G potentiostat / galvanostat, model 273A, interfaced (using the computer's RS232 port) by a National Instruments GPIB-PCII interface board, running NI-488.2 software, to a Viglen PC, model Genie Executive 3SX25 (386SX with a clock speed of 25 MHz and a maths co-processor).

A three electrode cell was used, with a working electrode consisting of an iron disc of circular configuration of 4 mm diameter (geometric surface area 0.1257 cm2); a saturated calomel reference electrode (SCE); and a platinum foil counter electrode with a surface area of 3.4 cm2.

Tafel extrapolation measurements were performed over a polarisation range of 200 mV each side of the corrosion potential, *Ecorr*. at a rate of 0.050 mV s-1, and an initial delay of 7 minutes was applied to the experiments. Experiments were usually performed in triplicate to ensure reproducibility of the results.

## 3. Results and discussion

## 3.1 Tafel extrapolation data for iron in HCl

Tafel extrapolation data for the inhibition of iron in deaerated 1 mol dm-3 HCl at 22 oC were considered for the five synthesized and two commercially available phosphonium salts solutions at concentrations ranging from 110-3 to 110-7 mol dm-3.

Each of the compounds tested by electrochemical techniques will be displayed as a table showing the mean values for corrosion current density (*jcor*), inhibitor efficiency (%) and corrosion potential (*Ecorr.*) versus the saturated calomel reference electrode (SCE) together with anodic and cathodic Tafel slopes for each of the inhibitor concentrations tested. Also, the effects of each compound are shown via a figure representing the Tafel experimental run that most closely depicts the mean of the duplicate or triplicate experimental results. Each figure has an insert showing the values of corrosion current density and inhibitor efficiency for each inhibitor concentration tested.

### 3.2 4-Anilinetriphenylphosphonium bromide (ATPPB)

High inhibition is achieved at the highest inhibitor concentration of 110-3 mol.dm-3 with inhibition at 91.7% (Figure 1), for a more extensive summary see Table 3. However, inhibition drops rapidly as inhibitor concentration decreases while below 110-5 mol.dm-3, the corrosion rate is increased compared to the uninhibited iron. The value of corrosion potential moves in a more noble direction as the concentration of inhibitor increases. This is indicative of anodic inhibitor. This behaviour at high inhibitor concentrations is explained by Hoar and Holliday [17] and by West [18] for a predominately blocking mode of inhibition. At high surface coverages (near monolayer) any gaps due to irregular packing, will be small and because of the relative size of the hydrated hydrogen and metal ions, anodic inhibition will always predominate. The cathodic Tafel slopes remain near the value of those obtained for uninhibited conditions until the concentration drops below 110-5 mol dm-3 when an increase is noted. For the anodic Tafel slopes, the values remain near 100 mV per decade with the exception of the highest concentration which is somewhat higher at 136 mV per decade.

The first step of inhibition would be expected to be the protonation of the aniline group to form an anilinium ion which can be electrostatically adsorbed. This was seen by Banerjee and Malhotra [19] who studied the corrosion of mild steel in HCl at 30 oC inhibited by aniline. This may help elucidate the reasons for corrosion acceleration at inhibitor concentrations of 110-5 mol dm-3 and below; the increase in cathodic Tafel slopes could indicate cathodic depolarisation as the protonated anilinium ion may catalyse the cathodic reaction. This is similar to the mechanism suggested by Davolio and Soragni [20] who reported corrosion acceleration of carbon steel in 1 M sulphuric acid at 30 oC in the presence of phenylmethylsulphoxide. A catalytic effect has been observed for other nitrogen-based inhibitors on the hydrogen evolution reaction for zinc and zinc alloy corrosion in HCl. Similar behaviour has been observed for protonated quinolines [21] and amines [22]. Foroulis [23] observed stimulation of copper corrosion for aniline and *n*-butylamine in 4.2 mol dm-3 sulphuric acid and aniline and pyridine in 2.6 mol dm-3 perchloric acid at 50 oC.

### 3.3 4-Benzyl alcoholtriphenylphosphonium chloride (BATPPC)

At the highest concentrations of 110-3 and 110-4 mol dm-3 (Figure 2), this compound exhibits high inhibition with values of 94.7% and 89.5% respectively as shown in Table 4 which summarises the results for this inhibitor. However, inhibition drops rapidly as the inhibitor concentration decreases. Between the concentrations of 110-5 and 110-6 mol dm-3 an inflection is observed. The most likely explanation for this effect is a change in the mode or configuration (or both) of adsorption. This has been observed previously by Hackerman *et al* [24] who studied homopiperazine adsorption on iron in 6 mol dm-3 HCl at 25 oC. A change in configuration might present itself as the adsorption of a single phenyl ring at high inhibitor concentrations where packing is compacted but, at low inhibitor concentrations, the possibility of a second phenyl ring adsorbing is maybe due to the low covering of inhibitor molecules at the metal surface. This could be initiated through the aliphatic alcohol group via the lone-pair on the oxygen atom or protonation of the alcohol group. The corrosion potential is polarised in the noble direction compared to uninhibited values. Inhibition is anodic at high inhibitor concentrations indicating a predominately blocking mode of inhibition. The anodic Tafel slopes for iron dissolution as ferrous ions remained close to those obtained for uninhibited conditions until the concentration drops below 110-5 mol dm-3 when deviations are noted. The values of cathodic Tafel slopes for hydrogen evolution remained near -145 mV per decade with the exception of the 110-6 mol dm-3 solution which gave somewhat higher values of -172 mV per decade.

**3.4 4-Chlorophenyltriphenylphosphonium bromide (CPTPPB)**

The results of Tafel analysis are shown in Table 5 and Figure 3 for this inhibitor. At the highest concentrations of 110-3 and 110-4 mol dm-3, this compound exhibited a high inhibition with values of 95.6% and 92.7%, respectively being achieved. However, the degree of inhibition dropped rapidly as the inhibitor concentration decreased. A promotion of corrosion was achieved at 110-6 mol m-3 (% = -149.4%) and 110-7 mol.dm-3 (% = -157.8%), as previously encountered at low inhibitor concentrations [25-27]. The corrosion potential remained almost constant over the entire range of inhibitor concentration near uninhibited acid values indicating mixed inhibition. The absence of anodic inhibition at high inhibitor concentrations would suggest that blocking is not the important inhibition mode. The cathodic Tafel slopes exhibited a decrease with inhibitor concentration. This may indicate a change in cathodic mechanism with the increase of inhibitor concentration. However, the anodic Tafel slopes showed the opposite effect, suggesting that the anodic reaction became more difficult.

### 3.5 Methyltriphenylphosphonium bromide (MTPPB)

The plot of inhibitor efficiency against inhibitor concentration (Figure 4 and Table 6) showed two steps at 110-3 to 110-4 mol dm-3 and also at 110-5 to 110-6 mol dm-3. Acceleration was achieved at inhibitor concentration of 110-7 mol dm-3 (% = -33.7%). The corrosion potential is polarised 20 mV noble compared to uninhibited values for the highest concentration and shifts to approximately 15 mV basic compared to uninhibited values at concentrations < 110-4 mol dm-3. The anodic Tafel slopes remained near the value of that obtained for uninhibited conditions with the exception of the 110-3 mol dm-3 concentration which is 50 mV per decade higher. The cathodic Tafel slopes values appear to oscillate around the value obtained for that in uninhibited conditions. The only close literature comparison appears to be due to Allen *et al* [28], who reported an inhibitor efficiency of 98.9% at an inhibitor concentration of 110-3 mol dm-3 for carbon-steel in 5.7 mol dm-3 HCl at 80 oC using weight loss and solution analysis techniques.

### 3.6 4-Phenoltriphenylphosphonium chloride (PTPPC)

Good inhibition was observed at the two highest concentrations with inhibitor efficiencies of 95.0 and 89.1% as illustrated in Table 7 and Figure 5. Below these concentrations, however, inhibitor efficiency drops to approximately zero. The two highest concentrations exhibit corrosion potentials near those obtained for uninhibited solutions to within 1 mV. Concentrations that have inhibitor efficiencies approximately zero are polarised at less than 10 mV more noble compared to the uninhibited value. The anodic and cathodic slopes for all concentrations, except 110-3 mol dm-3, show large deviations compared to uninhibited values.



Scheme 2. Resonance structures of 4-phenoltriphenylphosphonium chloride (PTPPC).

Scheme 2 shows the possible resonance structures of 4-phenoltriphenylphosphonium chloride and indicates the initiation of electrostatic adsorption or the protonated phenol group, stabilized by similar resonance structures to the above examples that can offer an alternative initiation step. The resonance structures shown in Scheme 2 could indicate the loss of inhibition at concentrations of 110-5 mol dm-3 and below. Cathodic splitting, as explained by Horner *et al.* [29] in reaction (4), could produce a phosphine containing the phenol ring, Ph2Ph(OH)P, by attack of the phosphorus-carbon bond. The subsequent phosphine might accelerate corrosion, nullifying the effect of the phosphonium parent, probably through interaction with the corrosion intermediate. The Tafel slopes have increased significantly above the values for inhibited concentrations. An alternative reason for acceleration of corrosion might arise via an analogy to the 4-aniline system. The phenol group can protonate similar to the aniline group, although much less readily. The subsequent protonated phenol ion may then catalyse the cathodic reaction through cathodic depolarisation.

****** (4)

### 3.7 Tetraphenylphosphonium bromide (TPPB)

Inhibition is achieved over the entire concentration range as shown in Table 8 and Figure 6. The corrosion potential shows little variation throughout the concentration range but all values are approximately 15-20 mV more noble than that in uninhibited solution. The cathodic slopes show only minor variations but remain slightly smaller when compared to the uninhibited solution. However, the anodic slope is 20 mV lower than uninhibited solutions at the lower concentrations and rise rapidly to 201 mV per decade at the highest concentration. The only literature comparison is attributable to Pagetti *et al.* [30] and Troquet and Pagetti [31] who reported an inhibitor efficiency of 75% at an inhibitor level of 110-3 mol dm-3 for Armco iron in 1 mol dm-3 HCl at 20 oC using Tafel analysis. The only secondary inhibitor for TPPB is triphenylphosphine [31-33] due to cathodic splitting.

### 3.8 4-Tolyltriphenylphosphonium chloride (TTPPC)

From Table 9 and Figure 7, this compound exhibits a high degree of inhibition with a value of 96.4% being achieved at maximum concentration. However, the degree of inhibition drops rapidly as the inhibitor concentration decreases, until acceleration occurs at 110-6 mol dm-3 (% = -193.6%) and 110-7 mol dm-3 (% = -150.2%). The two concentrations that exhibited high inhibitions have corrosion potentials near uninhibited ones but the lower concentrations have slightly more noble values. The cathodic Tafel slopes for the three lower concentrations are similar to uninhibited one and show a decrease at higher concentrations. The anodic Tafel slopes remain near those for uninhibited conditions, with the exception of the highest concentration which is 84 mV per decade higher. Adsorption of the para-substituted ring is likely to be enhanced due to the hyperconjugation effect (see below) previously proposed by Ayers and Hackerman [34] who studied the effect of methyl substitution on the pyridine ring using 2-, 3- and 4-picoline inhibition of iron in 6.08 mol dm-3 HCl at 35 oC.



Scheme 3. Resonance structures of 4-tolyltriphenylphosphonium chloride (TTPPC).

A comparison was made among the seven inhibitors and the results are summarised in Table 10. TPPB gives the best overall inhibition throughout the concentration range tested, as shown in Figure 8 and Table 10, although TTPPC gives the highest individual inhibition of 96.4% at the 110-3 mol dm-3 level. BATPPC was the only other inhibitor to function over the entire concentration range studied. The other five inhibitors accelerated the corrosion rate of iron in 1 mol dm-3 HCl at low concentration levels. These five did give some degree of corrosion protection at the two highest concentration levels (% = 75 - 97%).

ATPPB produces a linear relationship between corrosion inhibition (Figure 8) and current density (Figure 9) *vs.* inhibitor concentration despite this compound accelerating corrosion at low inhibitor levels. TPPB also shows a similar relationship but in this case inhibition is observed over the entire concentration range. MTPPB exhibits the lowest inhibition at the highest concentrations but shows a lower rate of decrease in inhibition compared to the synthesized phosphonium salts. This would seem to indicate that the 4-substituted phenyl compounds exhibit greater structural effects than replacement of a benzene ring. The two features noted above, namely very good inhibition at high inhibitor concentration and a rapid loss of inhibition, in some cases leading to corrosion acceleration are most prominent for the para-substituted compounds. This infers that surface coverage and molecular packing are important in the nature of these inhibitors at the iron surface. For BATPPC, the percentage inhibition increased, fell and rose once more to give 94% inhibition at 110-3 mol dm-3. This may be due to this compound undergoing a change in molecular configuration as inhibitor concentration varies. At high inhibitor concentrations, where packing is compacted, singularly chemisorbed phenyl ring is the predominant configuration. At low inhibitor concentrations, the possibility of a second phenyl ring adsorbing might due to the low covering of inhibitor molecules at the metal surface. Acceleration might be achieved via a catalytic pathway involving the para-substituted ring or a decomposition product that is repelled by the layer of phosphonium ions at high inhibitor concentrations. ATPPB is the poorest inhibitor of the compounds tested and acceleration of corrosion is achieved at inhibitor concentrations of 110-5 mol dm-3 and below. The order of inhibition is TPPB > BATPPC > CPTPPB > PTPPC > MTPPB > TTPPC > ATPPB for iron in 1M HCl at 22 oC, by Tafel extrapolation.

Figure 10 shows a Hoar-Holliday plot [17], of corrosion potential against the logarithm of corrosion current density. At corrosion current densities < 150 A cm-2, the inhibitors can be divided into two groups. The first consists of CPTPPB, TTPPC and PTPPC where *Ecor*. is within 5 mV of the uninhibited value. The second group comprises ATPPB, BATPPC, TPPB and MTPPB which are polarised approximately 20 mV more noble. ATPPB appears to shows a nearly linear relationship between corrosion potential and corrosion current density. The shape and features of the curve show a similarity for this compound with those shown in Figures 7 and 8. For ATPPB, the mode of inhibition and acceleration are due to changes in the electrical double layer. From the inclusion of an aniline group to the phosphonium salt another positive centre is created due to protonation from electrolyte; counter ions must be present to neutralise this charge. At high concentrations, the adsorption of the positive phosphonium ions causes the charge of the double layer to decreases, which will decrease corrosion. At lower concentrations, the majority of the phosphonium ion species is more widely distributed about the metal surface during adsorption. Adsorption of the bromide, counter ion, is more likely as these are smaller and can exert greater influence over surface charge, thus corrosion increases. The linear relationship between potential and corrosion current density indicates that adsorption is mainly through electrostatic adsorption. Chemisorption of a phenyl ring in the phosphonium ion is not indicated to a significant degree. BATPPC shows linearity in Figure 10, despite the reversal in the direction of change in corrosion current density as the inhibitor concentration is change between 110-5 and 110-6 mol dm-3. MTPPB is only the compound that demonstrates significant cathodic characteristics as the corrosion potential is more basic than in the uninhibited solution.

As general rule, the cathodic Tafel slopes for all the phosphonium salts tested are shown in Figure 11. The dotted line indicates the Tafel slope of uninhibited acid. At high concentrations the majority of the data points are lower than the dotted line, then move to within 10 mV of the dotted line. A similar trend is shown for the anodic Tafel slopes in Figure 12, with the exception that the data points at the highest concentration are all larger than the dotted line. There are two exceptions in both cases, PTTPC shows values that are significantly higher than any other at the lower concentrations; TPPB shows the opposite effect.

**4. Conclusions**

The order of inhibition for iron in 1 mol dm-3 HCl at 22 oC is TPPB > BATPPC > CPTPPB > PTPPC > MTPPB > TTPPC > ATPPB.

Another noticeable trend is that Tafel extrapolation measurements show that the two highest concentrations impart high inhibition (% = 75-97%), which is followed by a rapid fall at 110-5 mol dm-3, indicating a different inhibition mechanism or mode between high and low inhibitor concentration.

CPTPPB, TTPPC and PTPPC showed mixed inhibition, while ATPPB, BATPPC, TPPB and MTPPB primarily exhibited anodic inhibition.

Generally, the cathodic Tafel slopes at high inhibitor concentrations showed that the majority of the data points are lower in values than measured for uninhibited acid at high inhibitor concentrations, then shift to values within 10 mV of those measured for uninhibited acid at lower inhibitor concentrations.

**List of symbols**

***Symbol Meaning Units***

*ba* Anodic Tafel slope V

*bc* Cathodic Tafel slope V

*c* Molar concentration mol dm-3

*Ecor* Corrosion potential V

*j* Current density A cm-2

*jcor* Corrosion current density A cm-2

***Greek***

%**Inhibitor efficiencyDimensionless

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|  |  |
| --- | --- |
| **Phosphonium salt** | **Structure** |
| 4-tolyltriphenyl-phosphonium chloride (TTPPC)  *(Synthesized)* |  |
| 4-phenoltriphenylphosphonium chloride (PTPPC)  *(Synthesized)* |  |
| 4-anilinetriphenylphosphonium bromide (ATPPB)  *(Synthesized)* |  |
| 4-benzyl alcoholtriphenylphosphonium chloride (BATPPC)  *(Synthesised)* |  |
| 4-Chlorophenyltriphenylphosphonium bromide (CPTPPB)  *(Synthesised)* |  |
| Tetraphenylphosphonium bromide (TPPB)  *(Commercially available)* |  |
| Methyltriphenylphosphonium bromide (MTPPB)  *(Commercially available)* |  |

Table 1. Synthesized and commercially available phosphonium salts evaluated by electrochemical techniques in this paper.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | | Melting point / oC | Yield | | Molar mass  / g mol-1 |
| R | X | / g | / % |
| CH3 | Cl | 277-8 | 10.23 | 52.61 | 388.88 |
| OH | Cl | 296-9 | 3.47 | 17.76 | 390.85 |
| CH2OH | Cl | 235-6 | 12.33 | 60.91 | 404.88 |
| Cl | Br | 265-7 | 11.74 | 51.75 | 453.75 |
| NH2 | Br | 338-40 | 14.49 | 66.72 | 434.32 |

Table 2. Melting point and yield data for synthesised phosphonium salts.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| *c*  / mol dm-3 | *jcor*/ A cm-2 | *%* | *Ecor vs.* SCE / V | *ba*  / V | *bc* / V |
| 1 10-3 | 45 | 91.7 | -0.496 | 0.136 | -0.143 |
| 1 10-4 | 245 | 54.7 | -0.515 | 0.101 | -0.139 |
| 1 10-5 | 681 | -25.9 | -0.519 | 0.100 | -0.145 |
| 1 10-6 | 1004 | -85.7 | -0.528 | 0.099 | -0.155 |
| 1 10-7 | 1558 | -188.1 | -0.534 | 0.101 | -0.177 |
| 0 | 541 | - | -0.526 | 0.086 | -0.149 |

Table 3. Tafel extrapolation data for 4-anilinetriphenylphosphonium bromide (ATPPB) for the inhibition of iron in 1 mol dm-3 HCl at 22 oC.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| *c* / mol dm-3 | *jcor*/ A cm-2 | *%* | *Ecor vs.* SCE / V | *ba* / V | *bc*  / V |
| 1 10-3 | 28 | 94.7 | -0.497 | 0.108 | -0.154 |
| 1 10-4 | 57 | 89.5 | -0.508 | 0.098 | -0.151 |
| 1 10-5 | 446 | 17.6 | -0.525 | 0.095 | -0.139 |
| 1 10-6 | 319 | 40.9 | -0.523 | 0.114 | -0.172 |
| 1 10-7 | 512 | 3.9 | -0.526 | 0.070 | -0.142 |
| 0 | 541 | - | -0.526 | 0.086 | -0.149 |

Table 4. Tafel extrapolation data for 4-benzylalcoholtriphenylphosphonium chloride (BATPPC) for the inhibition of iron in 1 mol dm-3 HCl at 22 oC.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| *c*  / mol dm-3 | *jcor* / A cm-2 | % | *Ecor vs.* SCE / V | *ba* / V | *bc* / V |
| 1 10-3 | 24 | 95.6 | -0.526 | 0.126 | -0.113 |
| 1 10-4 | 39 | 92.7 | -0.524 | 0.108 | -0.118 |
| 1 10-5 | 455 | 15.8 | -0.516 | 0.072 | -0.130 |
| 1 10-6 | 1349 | -149.4 | -0.530 | 0.112 | -0.151 |
| 1 10-7 | 1394 | -157.8 | -0.531 | 0.078 | -0.145 |
| 0 | 541 | - | -0.526 | 0.086 | -0.149 |

Table 5. Tafel extrapolation data for 4-chlorophenyltriphenylphosphonium bromide (CPTPPB) for the inhibition of iron in 1 mol dm-3 HCl at 22 oC.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| *c*  / mol dm-3 | *jcor* / A cm-2 | *%* | *Ecor* *vs.* SCE / V | *ba*  / V | *bc* / V |
| 1 10-3 | 111 | 79.4 | -0.503 | 0.139 | -0.173 |
| 1 10-4 | 130 | 76.0 | -0.542 | 0.080 | -0.144 |
| 1 10-5 | 449 | 17.0 | -0.542 | 0.082 | -0.163 |
| 1 10-6 | 518 | 4.3 | -0.540 | 0.090 | -0.143 |
| 1 10-7 | 723 | -33.7 | -0.542 | 0.090 | -0.153 |
| 0 | 541 | - | -0.526 | 0.086 | -0.149 |

Table 6. Tafel extrapolation data for methyltriphenylphosphonium bromide (MTPPB) Inhibition of iron in 1 mol dm-3 HCl at 22 oC.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| *c*  / mol dm-3 | *jcor* / A cm-2 | *%* | *Ecor* *vs.* SCE / V | *ba*  / V | *bc* / V |
| 1 10-3 | 27 | 95.0 | -0.525 | 0.070 | -0.141 |
| 1 10-4 | 59 | 89.1 | -0.525 | 0.117 | -0.124 |
| 1 10-5 | 535 | 1.0 | -0.512 | 0.213 | -0.205 |
| 1 10-6 | 544 | -0.7 | -0.512 | 0.172 | -0.203 |
| 1 10-7 | 532 | 1.6 | -0.516 | 0.207 | -0.174 |
| 0 | 541 | - | -0.526 | 0.086 | -0.149 |

Table 7. Tafel extrapolation data for 4-phenoltriphenylphosphonium chloride (PTPPC) for the inhibition of iron in 1 mol dm-3 HCl at 22 oC.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| *c*  / mol dm-3 | *jcor* / A cm-2 | % | *Ecor* *vs.* SCE / V | *ba* / V | *bc* / V |
| 1 10-3 | 43 | 92.0 | -0.499 | 0.201 | -0.134 |
| 1 10-4 | 86 | 84.1 | -0.511 | 0.122 | -0.140 |
| 1 10-5 | 102 | 81.2 | -0.508 | 0.066 | -0.123 |
| 1 10-6 | 129 | 76.1 | -0.503 | 0.068 | -0.119 |
| 1 10-7 | 195 | 63.8 | -0.504 | 0.070 | -0.127 |
| 0 | 541 | - | -0.526 | 0.086 | -0.149 |

Table 8. Tafel extrapolation data for tetraphenylphosphonium bromide (TPPB) inhibition of iron in 1 mol dm-3 HCl at 22 oC.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| *c* / mol dm-3 | *jcor*/ A cm-2 | *%* | *Ecor vs.* SCE / V | *ba* / V | *bc* / V |
| 1 10-3 | 19 | 96.4 | -0.529 | 0.170 | -0.125 |
| 1 10-4 | 96 | 82.2 | -0.522 | 0.093 | -0.130 |
| 1 10-5 | 354 | 34.4 | -0.516 | 0.089 | -0.150 |
| 1 10-6 | 1588 | -193.6 | -0.504 | 0.101 | -0.143 |
| 1 10-7 | 1353 | -150.2 | -0.518 | 0.108 | -0.148 |
| 0 | 541 | - | -0.526 | 0.086 | -0.149 |

Table 9. Tafel extrapolation data for 4-tolyltriphenylphosphonium chloride (TTPPC) inhibition of iron in 1 mol dm-3 HCl at 22 oC.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| *c* / mol dm-3 | Inhibitor efficiency, *%* | | | | | | |
| ATPPB | BATPPC | CPTPPB | MTPPB | PTPPC | TPPB | TTPPC |
| 110-3 | 91.7 | 94.7 | 95.6 | 79.4 | 95.0 | 92.0 | 96.4 |
| 110-4 | 54.7 | 89.5 | 92.7 | 76.0 | 89.1 | 84.1 | 82.2 |
| 110-5 | -25.9 | 17.6 | 15.8 | 17.0 | 1.0 | 81.2 | 34.4 |
| 110-6 | -85.7 | 40.9 | -149.5 | 4.3 | -0.7 | 76.1 | -193.7 |
| 110-7 | -188.1 | 3.9 | -157.8 | -33.7 | 1.6 | 63.8 | -150.2 |

Table 10. Inhibitor efficiency for iron in 1 mol dm-3 HCl at 22 oC in the presence of various phosphonium salts.

**Figures captions**

Figure 1. Tafel curves for 4-anilinetriphenylphosphonium bromide (ATPPB) inhibition of iron in 1 mol dm-3 HCl at 22 oC.

Figure 2. Tafel curves for 4-benzylalcoholtriphenylphosphonium chloride (BATPPC) inhibition of iron in 1 mol dm-3 HCl at 22 oC.

Figure 3. Tafel curves for 4-chlorophenyltriphenylphosphonium bromide (CPTPPB) inhibition of iron in 1 mol dm-3 HCl at 22 oC.

Figure 4. Tafel curves for methyltriphenylphosphonium bromide (MTPPB) inhibition of iron in 1 mol dm-3 HCl at 22 oC.

Figure 5. Tafel curves for 4-phenoltriphenylphosphonium chloride (PTPPC) inhibition of iron in 1 mol dm-3 HCl at 22 oC.

Figure 6. Tafel curves for tetraphenylphosphonium bromide (TPPB) inhibition of iron in 1 mol dm-3 HCl at 22 oC.

Figure 7. Tafel curves for 4-tolyltriphenylphosphonium chloride (TTPPC) inhibition of iron in 1 mol dm-3 HCl at 22 oC.

Figure 8. Variation in inhibitor efficiency with inhibitor concentration determined by Tafel extrapolation for iron in 1 mol dm-3 HCl at 22 oC inhibited by various phosphonium salts.

Figure 9. Variation in corrosion current density with inhibitor concentration determined by Tafel extrapolation for iron in 1 mol dm-3 HCl at 22 oC inhibited by various phosphonium salts.

Figure 10. Variation in corrosion potential with corrosion current determined by Tafel extrapolation for iron in 1 mol dm-3 HCl at 22 oC in the presence of various phosphonium salts.

Figure 11. Variation in anodic Tafel slope with inhibitor concentration determined by Tafel extrapolation for iron in 1 mol dm-3 HCl at 22 oC inhibited by various phosphonium salts (Uninhibited data are shown by dotted lines).

Figure 12. Variation in cathodic Tafel slope with inhibitor concentration determined by Tafel extrapolation for iron in 1 mol dm-3 HCl at 22 oC inhibited by various phosphonium salts (Uninhibited data are shown by dotted lines).

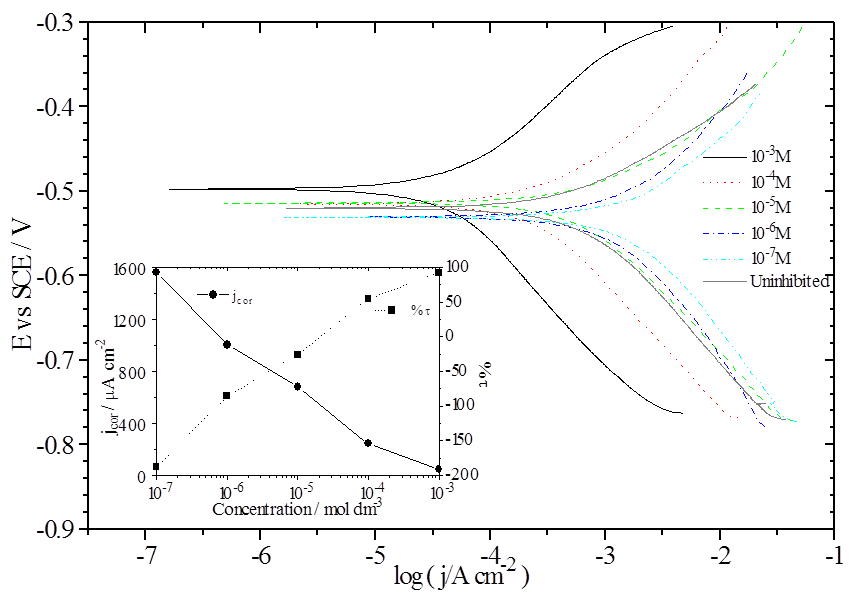


Figure 1.

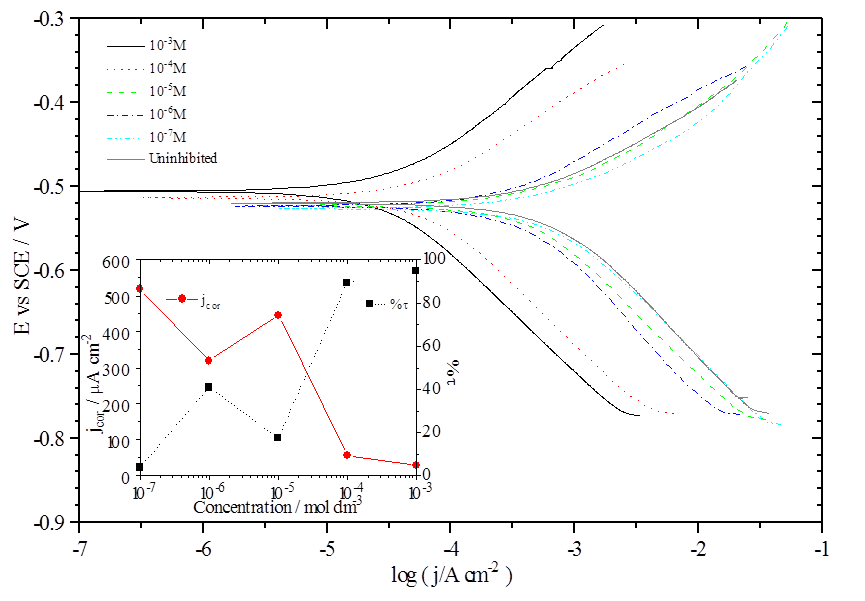


Figure 2.

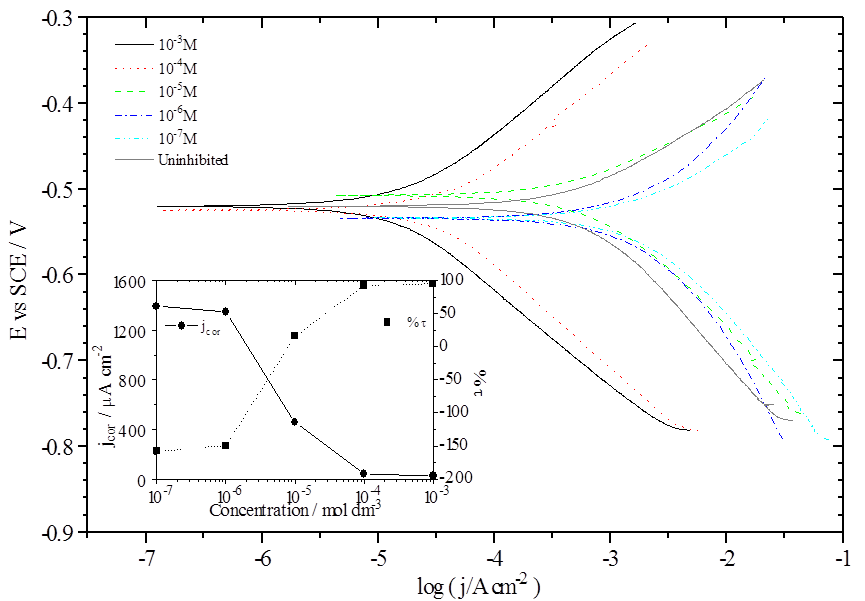


Figure 3.

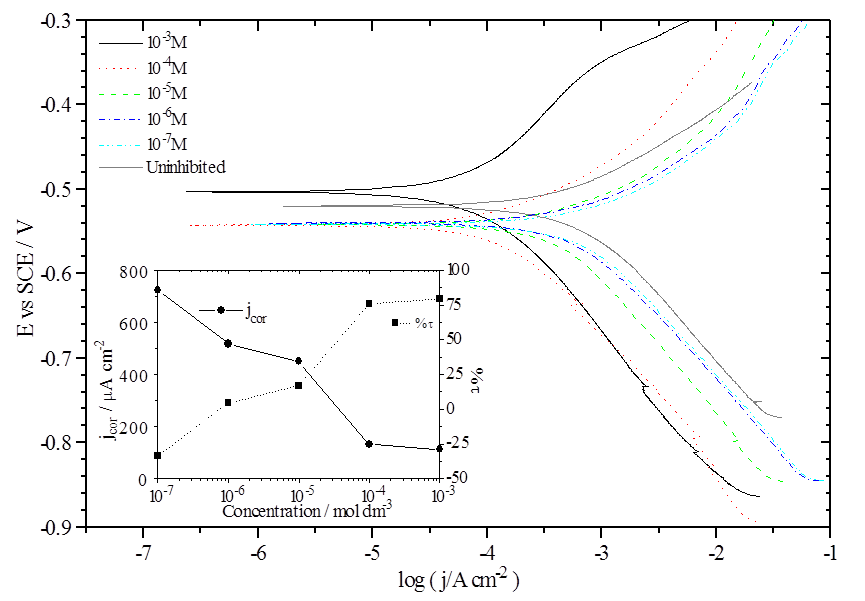


Figure 4.

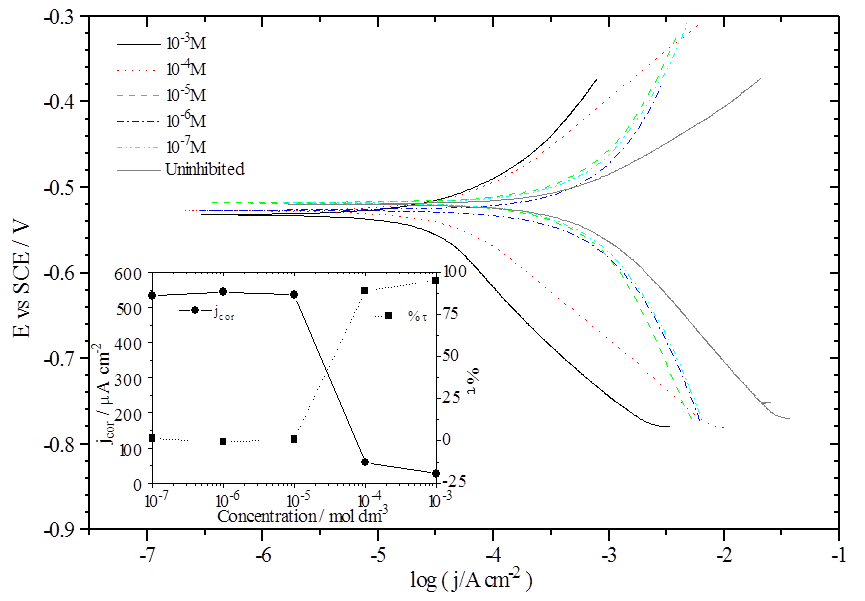


Figure 5.

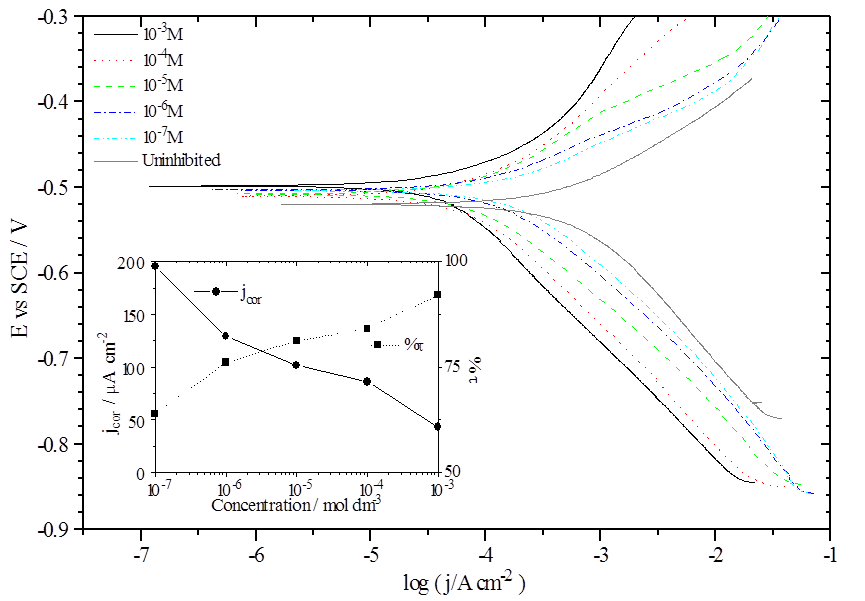


Figure 6.

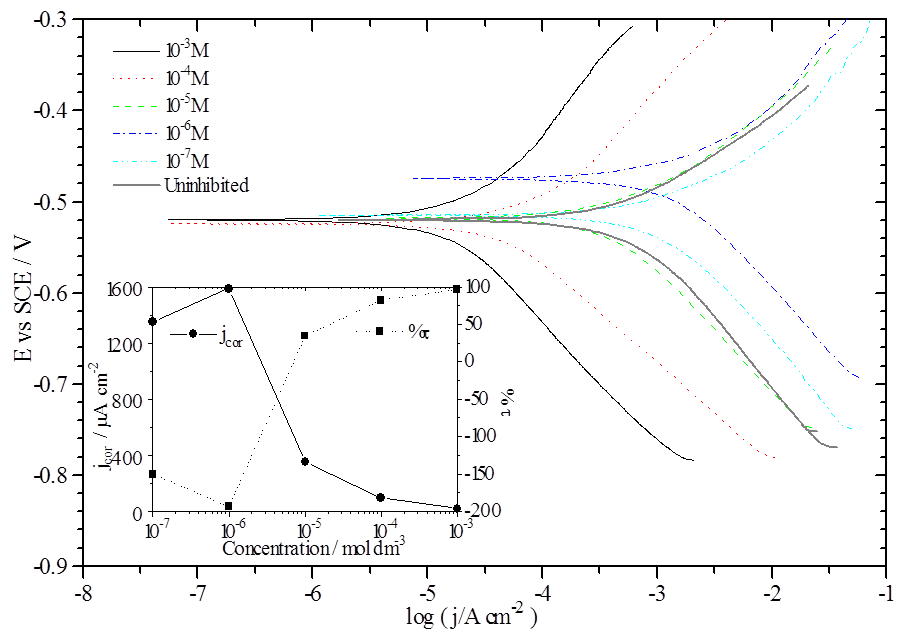


Figure 7.

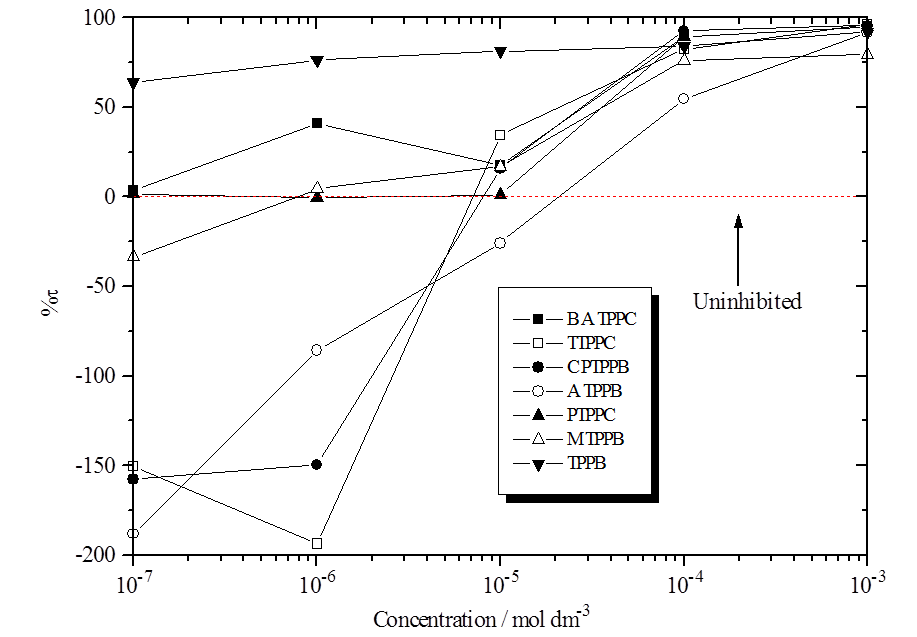


Figure 8.

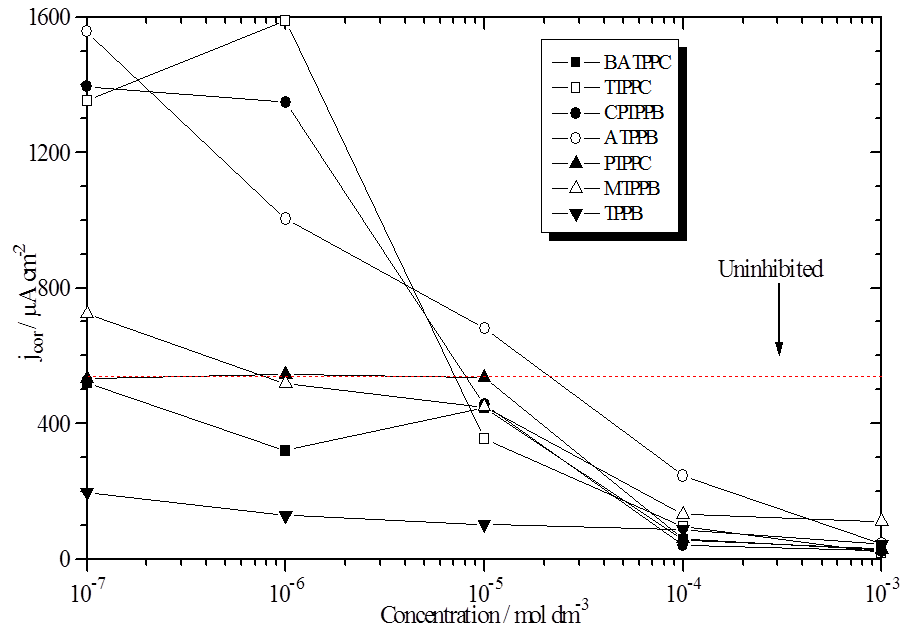


Figure 9.

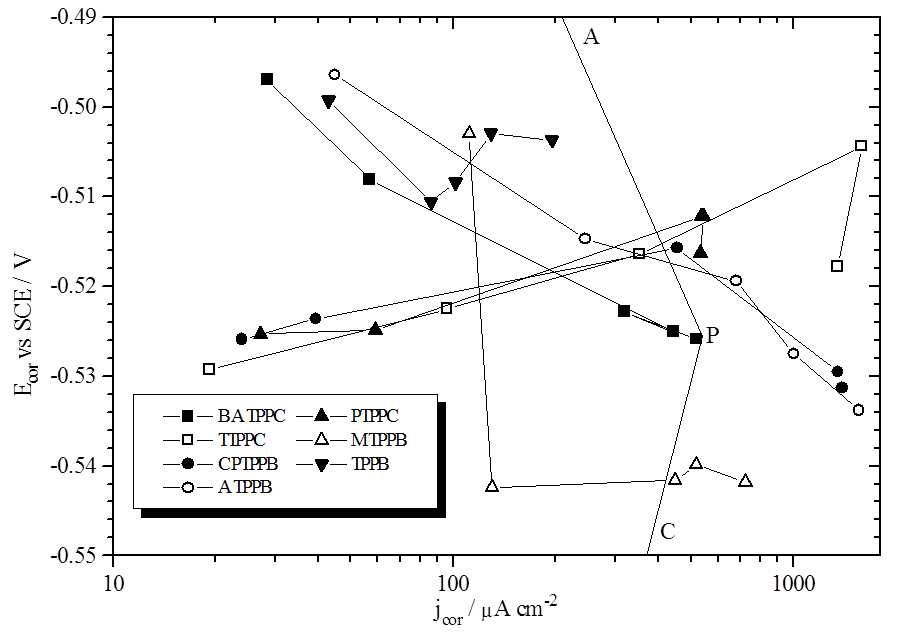


Figure 10.

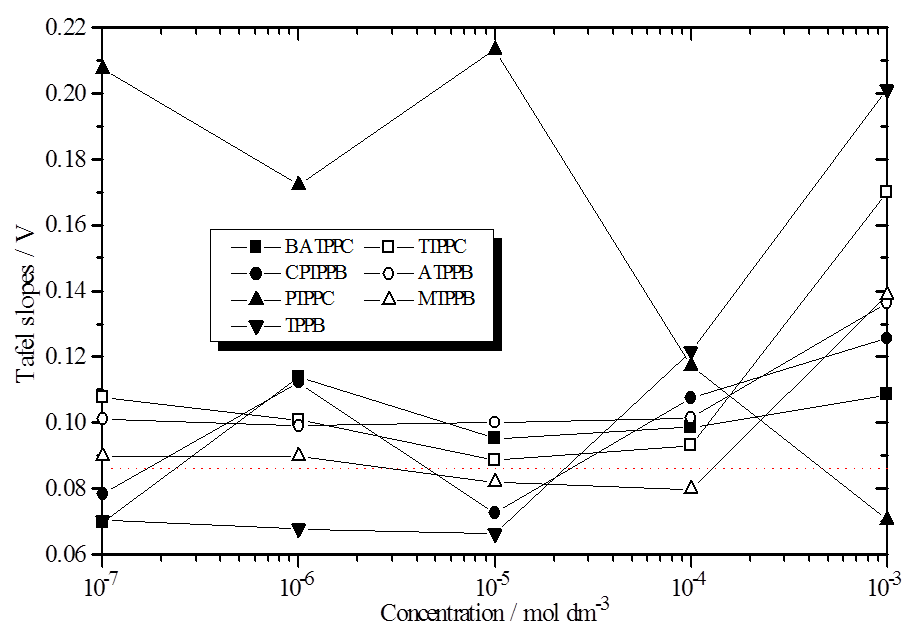


Figure 11.

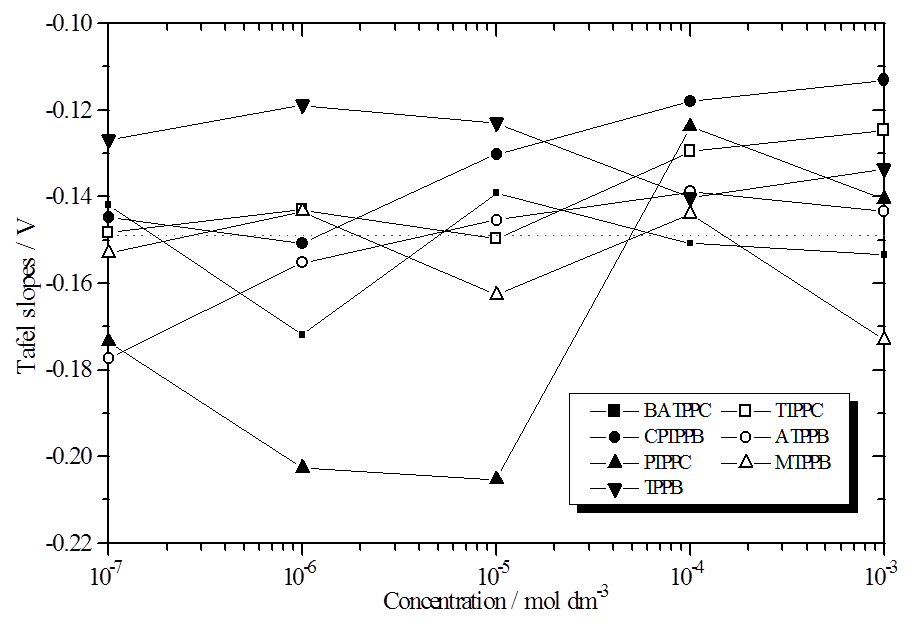


Figure 12.