CATHODIC STRIPPING VOLTAMMETRY OF THIOAMIDES ON A ROTATING SILVER DISK EL. (U) STATE UNIV OF NEW YORK AT BUFFALO DEPT OF CHEMISTRY M IWAMOTO ET AL. JAN 84

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**Authors:** Mizuho Iwamoto and R.A. Osteryoung

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9.2 borate buffer. Stripping peaks appeared at $\sim -0.76$ V vs SCE, approximately identical to that for $S^{2-}$, which, however, produces an oxidation wave at $-0.66$ V vs SCE. It is suggested that the insoluble compounds formed in the electrode surface are converted to $Ag_2S$. Linear cathodic stripping peak currents were found over a concentration range of $\sim 10^{-7}$ M to $10^{-5}$ M.
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by
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Cathodic stripping voltammetry at a rotating silver disc electrode has been applied for the determination of five thioamides; thioacetamide (TAA), thiourea (TU), thionicotinamide (TNA), ethionamide (ETNA), and thiosemicarbazide (TSC). Well defined oxidation waves limited by convective diffusion were found at -0.29, -0.12, -0.27, -0.15 and -0.105 V vs SCE for the five amides in a pH 9.7 borate buffer. Stripping peaks appeared at -0.76 V vs SCE, approximately identical to that for S²⁻, which, however, produces an oxidation wave at -0.66 V vs SCE. It is suggested that the insoluble compounds formed in the electrode surface are converted to Ag₂S. Linear cathodic stripping peak currents were found over a concentration range of 10⁻⁷ M to 10⁻⁵ M.
Introduction

Cathodic stripping voltammetry (CSV) has been employed for analysis of inorganic anions and organic materials which form sparingly soluble compounds with the electrode. Species which have been determined by CSV include halides, sulfide, etc. (1) and oxalate, succinate, dithizonate, diethyldithiophosphate, cysteine, ruheanic acid, thioanilide, dimercaptothiodiazide (7), thiourea (3) 2-mercaptohenzothiazole (4) diethyldithiocarbamate (5) and 2-mercaptothiazine N-oxide (6). Recently the CSV behavior of a wide range of organic sulphur compounds, flavins and porphyri.s were surveyed by Florence (7). Several extensive reviews covering CSV for the determination of biological compounds have appeared (8,9).

Most applications reported have been carried out at hanging mercury drop or mercury pool electrodes. Stripping voltammetry at solid electrodes has been performed on platinum (10), gold (11) and graphite or carbon (12).

Silver electrodes have been applied for the determination of trace amounts of halides (13) and thiocyanide (14). We have used a silver rotating disk electrode (AgRDE) for the determination of sulfide (15), with detection limits exceeding those of ion selective electrodes. Recently, the CSV of thiouracil on a AgRDE accompanied by complicated adsorption processes has been studied (16). The use of a AgRNF in CSV has the following advantages; 1) sparingly soluble compounds (similar to those on Hg) might be formed during anodic oxidation of solutes at silver electrodes; 2) rotating disk electrodes provide increased mass transport to the electrode surface and well defined hydrodynamic conditions compared
to a stationary electrode in a stirred solution (17,18); 3) solid electrodes for CSV are advantageous when compared to Hg which has both handling and toxicity problems.

The polarographic and voltammetric behavior of several thioamides on Hg, e.g. thiourea (3,19), thioacetamide (20), ethionamide (21) and other thioamides of pharmaceutical importance (22), have been studied, and some of these studies have involved CSV (3,21,22).

The purpose of the present work is to ascertain if CSV at a AgRnF can be utilized for compounds of biological importance. Five thioamides, thioacetamide (TAA), thiourea (TU), thionicotinamide (TNA), ethionamide (ETNA) and thiosemicarbazide (TSC), were investigated:

- Thioacetamide (TAA) $R = -CH_3$
- Thiourea (TU) $R = -NH_2$
- Thionicotinamide (TNA) $R = \text{structure}$
- Ethionamide (ETNA) $R = \text{structure}$
- Thiosemicarbazide (TSC) $R = -NHNH_2$
Experimental

Reagents

Thioacetamide (TAA), thionicotinamide (TNA), ethionamide (ETNA), and thiosemicarbazide (TSC) were obtained from Sigma Chemical Co., and thiourea (TU) from Fisher Scientific. Stock solutions of 1 x 10^{-2} M TAA, TSC and TU and 1 x 10^{-3} M TNA and ETNA were prepared with deionized water and were stored in a refrigerator. Possible changes in concentration from hydrolysis of the thioamides were monitored by measuring the UV spectra. Analytical grade Na_{2}B_{4}O_{7} was obtained from Fisher Scientific. 0.05 M buffer solutions were prepared and adjusted to be at least 100 times greater in concentration than the thioamides. The solution pH, adjusted to 9.2, was measured before and after each experiment.

Apparatus

A Pine Instrument's rotating silver disk electrode was mounted on a Pine Model ASR rotator. The geometrical surface area of the rotating disk was 0.442 cm^{2}. The electrode surface was polished prior to immersion in the electrolyte solution with 0.3 μm and 0.05 μm alumina (Dry Power, Type A, Fisher Scientific Co.) for at least 15 minutes on a Minimet Polisher (Ruehler Ltd.). The surface condition and preparation of the silver electrode were extremely important to the cathodic stripping peaks of the thioamides. The silver surface was polished with 45 μm, 15 μm and 6 μm diamond, and 1 μm, 0.3 μm and 0.05 μm alumina to remove scratches. This surface was examined through a microscope to ascertain if scratches were still noticeable. A satisfactory electrode surface was taken as one where the background current was about 10 μA/cm^{2} at approximately -1 V. If the background current had a value greater than 25 μA/cm^{2} and gave a hydrogen
evolution current which increased steeply at potentials more negative than -1.2 V, the possibility of electrode leakage at the disc was suspected. A small leak affects CSV of the thioamides for concentrations less than 10^-6 M.

An EG&G PARC Model 173 potentiostat/galvanostat equipped with a Model 179 digital coulometer and Model 175 universal programmer was used for voltammetric measurements. An Omnigraph Model 2000 X-Y recorder (Houston Instrument Co.) was used for recording voltammograms. All potentials were measured against a saturated calomel electrode (SCE). A large platinum counter-electrode was used.

**Procedure**

Ruffer solution poured into a 250 cm^3 pyrex glass electrolytic cell was deaerated by bubbling with argon for 30 minutes at a constant potential of -1.0V until a current less than 10 μA was obtained. After background voltammograms were obtained, the thioamide solution was added to the background solution with an Eppendorff pipet. The potential was then stepped to the deposition potential; the electrode was rotated during the deposition of thioamide. During deposition and stripping processes the solution was covered by an argon purge. The temperature was 25 ± 0.5°C.

The deposited film was cathodically stripped by means of a linear potential scan. The charge passed during deposition was measured by the coulometer and the charge passed during stripping was determined by integrating the recorder chart paper by cutting out the stripping current-potential (time) curve and weighing.
Results and Discussion

Cyclic Voltammograms at AgRDE

Cyclic voltammograms at a AgRDE for TAA, TU, TNA, ETNA and TSC at 
~10^{-4} \text{ M in pH 9.2 borate buffers are shown in Figs. 1-5.} The thioamides
give anodic currents (process 1 in Fig. 1-5) corresponding to the formation
of sparingly soluble silver compounds (see below) at half-wave potentials
(E_{1/2}) of: TAA)-0.29 \text{ V; TU} -0.12 \text{ V; TMA})-0.27 \text{ V; ETNA) } -0.15 \text{ V and TSC}
-0.105 \text{ V vs. SCE, respectively. Limiting currents plotted against the
square root of rotation rate (} \omega^{1/2} \text{) showed linear behavior (Fig. 6),
passing through the origin, and were proportional to concentration,
indicating control by convective diffusion of the thioamides from the bulk
solution. Figure 7 shows anodic current at less than limiting current
values plotted vs } \omega^{1/2} \text{ for TAA for various potentials. The convex
curves indicate chemical and/or electrochemical complications but were not
investigated further.

Thiourea produces a second wave at potentials more positive than
+0.15V (process 2 in Fig 2), which is due to oxidation of TU to the
disulfide (19). In the cases of ETNA and TNA, a spike "peak" appears prior
to the limiting current, which may correspond to nucleation of the Ag
compound on the electrode surface. The E_{1/2} of ETNA on anodic scan is
more positive than that on the reverse, cathodic scan, indicating that the
formation of a Ag-ETNA compound is inhibited by a strong adsorption of ETNA
on the Ag surface. TSC gives less-defined anodic waves with hysteresis,
suggesting more complicated electrode reactions.

All the thioamides studied show clear cathodic stripping peaks
suitable for determination of each thioamide (process 3 in Figs. 1-5) with
peak potentials ($E_p$) of -0.70 to -0.80 V. ETNA and TNA show a reduction current at about -1.0 V which is independent of the anodic deposition (process 4 in Fig. 3 and 4). The limiting reduction current of ETNA is proportional to its concentrations. These reduction currents may result from catalytic hydrogen evolution of the primary thioamide (22).

A comparison of the anodic charge ($Q_a$) passed during the deposition to cathodic charge ($Q_c$) consumed during stripping is shown in Table I. $Q_c$ for TAA, TU, TNA and FTNA are almost the same as $Q_a$ in $10^{-4}$ M solutions. The results indicate that at a high concentration of these four thioamides anodically deposited silver-thioamide compounds at a silver electrode do not dissolve chemically and are not removed mechanically during deposition; deposits are completely stripped during the cathodic scan. Contrary to this, $Q_c$ of TSC is $1/3$ of $Q_a$ even in $10^{-4}$ M solution. Hence, the Ag species deposited initially on the electrode, probably a Ag-TSC compound, appears soluble in the bulk solution.

Voltammograms of sulfide at the AgRDE were compared to those of thioamides (Fig. 8). Although anodic current corresponding to formation of Ag$_2$S appears at much more negative potential ($E_{1/2} = -0.66$ V) than those of the thioamides, its stripping peak potential is very close to that of the thioamides. For deposits of the same amount of silver-thioamide compounds, the stripping peak potentials are the same as for sulfide; for example, $E_{pC}$ for $Q_c$ of $4 \times 10^{-3}$ C is about -0.84 - 0.86 V for the thioamides and sulfide. This suggests the cathodic stripping process for these thioamides is similar to that of sulfide.

These preliminary studies indicate the possibility of cathodic stripping voltammetric analysis using deposition of these thioamides at
potentials more positive than -0.25 V for TAA, TNA and ETNA and -0.10 V for TII and TSC respectively.

Cathodic Stripping Voltammetry

As the pH decreases, the potential of Ag compound formation shifts to positive potentials close to that of silver oxide formation. TAA, TII and TNA gave sharp stripping peaks, in pH 4.6 acetate buffer, but the sensitivity decreased because of a decrease in the recovery of $Q_c$ and of the positive shift of the anodic waves. In very alkaline solutions, thioamides are unstable and tend to hydrolyze. Thus, a pH 9.2 borate buffer was chosen as the electrolyte for detailed study.

Fig. 9 shows the effect of the deposition potential ($E_d$) on the stripping peak current ($I_p$) in the range of $10^{-7}$ M TAA, TII, TNA, ETNA and $10^{-6}$ M TSC. At deposition potentials more positive than +0.1 V a decrease in the stripping peak current occurs as a result of overlapping oxide deposition. The optimum deposition potential ($E_d$) is -0.05 V for TAA, TNA and ETNA, and NV for TII and TSC. The plots of $I_p$ against time ($t_d$) for TII and TAA in the $10^{-6}$ - $10^{-7}$ M range are shown in Fig. 10. At concentrations of $\sim 10^{-7}$ M, TAA and TII show an induction period; other thioamides behave in a similar manner. At greater than $5 \times 10^{-6}$ M, the plots pass through the origin. Although the cathodic stripping charge ($Q_c$) is smaller than the anodic deposition charge ($Q_a$) calculated theoretically from the Levich equation at a concentration showing an induction period, $Q_c$ is almost the same as $Q_a$ at higher concentrations which display no induction period. This induction period may depend on the solubility and formation constant of the Ag compound and/or adsorption of thioamides. Thus, deposition times ($t_d$) greater than 600 s were employed
for CSV of the thioamides in concentration ranges less than $10^{-7}$ M.

$I_p$ is proportional to scan rate and to the square root of the rotation rate during deposition. In this work, scan rates of 50 mV/s and rotation rates of 2500 or 3000 rpm were employed.

Stripping peaks for the same amount of deposited silver-thioamide compounds are independent of electrode rotation during stripping, indicating that the cathodic stripping process of the Ag compounds is not controlled by diffusion of thioamides from the electrode surface and that the overall process for the stripping is irreversible.

Typical cathodic stripping voltammograms of TAA in the range of $10^{-7} - 10^{-6}$ M are shown in Figs 11. The stripping peaks for all the thioamides are very similar and reproducible. Plots of $I_p$ against concentration for TAA, TII, TIA and TNA are shown in Fig. 12. However, these calibration curves do not pass through the origin presumably because of the solubility of the Ag-thioamide compound, kinetics of film formation and other complicated anodic processes of thioamides on Ag electrodes. The analytical data for determination of TAA, TII, TNA, ETIA and TSC is listed in Table II. $I_p$ is proportional to $n_C$ at coverages corresponding to less than $5.5 \times 10^{-3}$ μC/cm², and the linearity in the plots of $I_p$ vs. $C$ is maintained to $5.0 \times 10^{-5}$ M at various deposition times. Thus, the application of the silver RPF for CSV permits thioamides to be determined in the range $5 \times 10^{-5} - 10^{-5}$ M.

An important interfering ion for the determination of sulphur containing organic molecules is chloride. Concentrations of Cl⁻ in the range of $10^{-5} - 10^{-4}$ M have no effect on the stripping peaks of $1 \times 10^{-6}$ M solutions of TAA and TII. The potential at which Cl⁻ forms AgCl
at the Ag electrode at pH 9.2 is +0.7 V, which is more positive than the deposition potential for the thioamides.

Suggested Mechanism for Anodic Deposition of Thioamides on Ag Electrode.

The mechanism for deposition of these thioamides appears different from that of thiol-type sulfur compounds like thiouracil. \( E_p \) values for the CSV of thiobarbituric acid and thinuracil (5) are about -0.25 V in pH 9.2 and -0.35 V at pH 8.3 in 10\(^{-5}\) M solutions, respectively, which are more positive than \( E_p \) for thioamides. The previous work (20-24) on anodic processes of the primary thioamides (thiourea, thioacetamide, thiosemicarbazide and ethionamide) on Hg concluded that HgS is produced in the anodic polarographic process of such compounds along with nitrile. \( E_p \) values for CSV of thioamides on Ag are very close to that of sulfide at both pH 9.2 and 4.6 (Table II). Solutions of the five thinamides and S\(^2-\) gave only one stripping peak at a given deposition time, but at deposition time shorter than 60 s the stripping peak split in two. It is suggested that Ag compounds formed initially on anodic deposition turn to AgS with increasing deposition time.

Some chemical reactions of five thioamides were examined and gave the following results: 1) at pH 9.2 the addition of thioamide solution to silver nitrate solution gave a dark gray-brown precipitate similar to that in sulfide solution for all thioamides, TAA, THI, TNA, ETNA and TSC; 2) the same treatment in neutral medium led to the same precipitate as pH 9.2 for TAA, TNA and ETNA but a white precipitate for TSC. As for THI, a white precipitate formed initially but turned black within several seconds. After the measurement of CSV for 10\(^{-4}\) M thioamide solutions, a dark gray
precipitate remained on the electrode surface.

TU and TSC form sparingly soluble 2:3 complexes in acidic solutions (26,27) and then the complexes decompose, more or less readily to silver sulphide with increasing silver ion concentration. The decomposition usually begins as the molar ratio of silver(I) to ligand approaches 1:1 with increasing silver ion concentration. An excess of ligand leads to the complex species \([\text{Ag(SR)}_n]^{3+}\) which are soluble in aqueous solution (27). Thus, it is assumed that at a Ag electrode the anodic reactions of the thioamides proceed to a final product of Ag$_2$S through silver-thioamide compound formation, i.e.

\[
\begin{align*}
2 \text{Ag}^+ & \rightarrow 2 \text{Ag}^+ + 2e^- \\
\text{RCNH}_{2} + \text{R-NC} = \text{NH} + \text{Ag}^+ \rightarrow \text{R-CN} = \text{NH} + \hat{\text{A}} \\
\text{Ag}^+ & \rightarrow \text{Ag}_2\text{S} + \text{RCN} + \hat{\text{H}}^+ \\
\text{or} \\
3\text{RCNH}_2 + 2 \text{Ag}^{2+} & \rightarrow \text{Ag}_2(\text{RCNH}_2)_3^{2+} \\
\text{S}^-\text{Ag}^+ & \rightarrow \text{S} \\
2 \text{R-C}=\text{NH} + \text{RCNH}_2 & \rightarrow 2\hat{\text{H}}^+ \\
\text{Ag}_2\text{S} & \rightarrow \text{RCN} + \text{RCNH}_2
\end{align*}
\]
The values of the diffusion coefficients (D) for TAA, TU, TNA and ETNA were calculated by using the anodic limiting currents (Table I) and the Levich equation, assuming n = 2 for the anodic reaction (Table III). The value of D for TU, $1.31 \times 10^{-5}$ cm$^2$ s$^{-1}$ is in good agreement with the published value of $1.33 \times 10^{-5}$ cm$^2$ s$^{-1}$ (28) and the values for other thioamides are quite reasonable considering the differences in molecular structure.

The reaction rate leading to Ag$_2$S formation appears to depend on the concentration of thioamide, the applied potential and deposition time. More work is needed to completely characterize the behavior of the anodic deposition of thioamides on Ag electrodes.
This work was supported in part by the Office of Naval Research.
References

Table I. Comparison of anodic charge ($Q_a$) and cathodic charge ($Q_c$).

$\omega = 2500$ rpm, scan rate = 50 mV/s

<table>
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<th></th>
<th>[C]/10^{-4} M</th>
<th>$I_d/10^{-4}$ A</th>
<th>$I_d/C$ AM^{-1}</th>
<th>$Q_a/10^{-3}$ C</th>
<th>$Q_c/10^{-3}$ C</th>
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<td>TAA</td>
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<td>1.00</td>
<td>1.00</td>
<td>3.43</td>
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<td>0.975</td>
<td>6.46</td>
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<td>2.10</td>
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<td>ETNA</td>
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<td>0.510</td>
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<td>6.40</td>
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<td>1.20</td>
<td>1.20</td>
<td>1.000</td>
<td>3.90</td>
<td>4.30</td>
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Table II. Cathodic Stripping Voltammetric Data for Determination of Thioamides

$t_d = 15 \text{s} \sim 900 \text{s}, \omega = 3600 \text{rpm}, \text{scan rate} = 50 \text{mV/s}$. Potentials are in V vs SCE.

<table>
<thead>
<tr>
<th>thioamide</th>
<th>$E_{1/2}^{a)}$ anodic waves</th>
<th>$E_d$</th>
<th>$E_p^{b)}$ stripping peak</th>
<th>linear range/M</th>
<th>$E_{1/2}^{a)}$</th>
<th>$E_p^{b)}$</th>
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<tbody>
<tr>
<td>TAA</td>
<td>-0.29</td>
<td>-0.05</td>
<td>-0.76</td>
<td>$5.1 \times 10^{-8} - 6.0 \times 10^{-5}$</td>
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<td>TU</td>
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<td>-0.78</td>
<td>$6.8 \times 10^{-8} - 6.0 \times 10^{-5}$</td>
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<td>-0.56</td>
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<td>TNA</td>
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<td>-0.76</td>
<td>$7.5 \times 10^{-8} - 6.0 \times 10^{-5}$</td>
<td>+0.03</td>
<td>-0.55</td>
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<tr>
<td>ETNA</td>
<td>-0.15 (-0.26)$^{c)}$</td>
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<td>-0.77</td>
<td>$9.0 \times 10^{-8} - 6.0 \times 10^{-5}$</td>
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<tr>
<td>TSC</td>
<td>-0.105</td>
<td>0</td>
<td>-0.78</td>
<td>$7.5 \times 10^{-7} - 1.0 \times 10^{-4}$</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

$S^{2-}$
-0.66
-0.76
-0.63
-0.53

$a)$ in $10^{-4}$ M solution

$b)$ at cathodic charge ($Q_c$) of 450 $\mu$C/cm$^2$

$c)$ on cathodic scan
Table III. Diffusion coefficients for thioamides in 0.05 M borate buffer.

<table>
<thead>
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<th>Thioamide</th>
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Figure Captions

1. RDE voltammogram (solid line) of $2 \times 10^{-4}$ M TAA; AgRDE; $\omega = 2500$ rpm; $\nu = 10$ mV s$^{-1}$; pH 9.2 borate buffer. Background in the absence of TAA (dashed line).

2. As Fig. 1; $2 \times 10^{-4}$ M TU.

3. As Fig. 1; $1.4 \times 10^{-4}$ TNA.

4. As Fig. 1; $1.35 \times 10^{-4}$ M ETNA.

5. As Fig. 1; $1.4 \times 10^{-4}$ M TSC.

6. $I_d - \omega^{1/2}$ plots of a) $4 \times 10^{-4}$ M TAA; b) $2.8 \times 10^{-4}$ M TNA; c) $1.99 \times 10^{-4}$ M ETNA; d) $4 \times 10^{-4}$ M TU and e) $1.0 \times 10^{-4}$ M TSC. $\nu = 10$ mV s$^{-1}$.

7. Dependence of anodic current of $4.0 \times 10^{-4}$ M TAA at various potentials vs square root of rotation rate. a) -0.1 V; b) -0.2 V; c) -0.25 V; d) -0.3 V; e) -0.35 V. $\omega = 2500$ rpm; $\nu = 10$ mV s$^{-1}$.

8. As Fig. 1; $1.24 \times 10^{-4}$ M Na$_2$S.

9. Effect of deposition potential ($E_d$) on stripping peak current ($I_p$) for thioamides. (-0-) $8.8 \times 10^{-7}$ M TAA; (-@-) $6.8 \times 10^{-7}$ M TU; (-@-) $7.6 \times 10^{-7}$ M TNA; (-@-) $7.3 \times 10^{-7}$ M ETNA; (-@-) $3.6 \times 10^{-6}$ M TSC. $t_d = 300$ s (180 s for TSC); $\omega = 3600$ rpm; $\nu = 50$ mV s$^{-1}$.

10. Plots of stripping peak currents ($I_p$) against deposition time ($t_d$) for: a) $5.4 \times 10^{-7}$ M TU; b) $8.0 \times 10^{-7}$ M TAA; c) $8.1 \times 10^{-7}$ M TU; d) $2.0 \times 10^{-6}$ M TAA; e) $3.0 \times 10^{-6}$ M TU; f) $5.2 \times 10^{-6}$ M TU; g) $6.1 \times 10^{-6}$ M TAA. $E_d = -0.05$ V for TAA; 0 V for TU; $\omega = 3600$ rpm; $\nu = 50$ mV s$^{-1}$.
11. Cathodic stripping peaks at various concentrations of TAA.
   a) 1.27; b) 2.54; c) 3.80; d) 5.06; e) 6.36; f) $8.83 \times 10^{-7}$ M.
   \[ t_d = 600 \text{ s}; \quad E_d = 0 \text{ V}; \quad \omega = 3600 \text{ rpm}; \quad \nu = 50 \text{ mV s}^{-1}. \]

12. Concentration dependence of stripping peak current ($I_p$) for
   a) TAA; b) TU and c) TNA.  \[ t_d = 900 \text{ s}; \quad E_d = -0.05 \text{ V for TAA and TNA}; 0 \text{ V for TU}; \quad \omega = 3600 \text{ rpm}; \quad \nu = 50 \text{ mV s}^{-1}. \]
Figure 3
Figure 4
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