

Anodic Stripping Voltammetric Determination of Thallium at a Mercury Film/Glassy Carbon Electrode: Optimization of the Method and Application to Environmental Waters

Abdoulkadri Ayouba Mahamane^{1,2,*}, Boubie Guel², Paul Louis Fabre³

¹Department of Chemistry, Faculty of Sciences, Abdou Moumouni University, Niamey, Niger

²Department of Chemistry, Faculty of Sciences, Joseph KI ZERBO University, Ouagadougou, Burkina Faso

³Chemical Engineering Laboratory, University of Toulouse, Toulouse, France

Email address:

kadayouba@gmail.com (Abdoulkadri Ayouba Mahamane)

*Corresponding author

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Abstract: This paper describes a procedure for the determination of thallium by differential pulse anodic stripping voltammetry (DPASV) using a mercury film deposited on glassy carbon as the working electrode. The procedure has been optimized using experimental design methodology. The following results were obtained: deposition potential: -1000 mV, deposition time: 4 min, speed of the rotating disc electrode: 1000 rpm, pulse amplitude 80 mV. The response of the electrode towards thallium ions was then verified by establishing the calibration curve, which showed a good correlation coefficient of 0.9973 and the error between 5 successive determinations did not exceed 1.25%. The calculated limit of detection (LOD) is equal to $2.2 \cdot 10^{-8} \text{ mol.L}^{-1}$. A certified standard of thallium at 1 mg.L^{-1} is determined by the standard addition method and the recovery rate obtained is 99.14%. The remarkable electroanalytical performances of the glassy carbon/mercury thin film electrode make it amenable to employ it successfully as an electrochemical sensor for the determination of traces of thallium in environmental samples. Measurements carried out on the waters from wells and boreholes in the village of Yamtenga reveal thallium levels above the standard for some sources of water. These waters are therefore not recommended for use as drinking as drinking water.

Keywords: Thallium, Mercury Film, Differential Pulse Anodic Stripping Voltammetry, Optimization, Natural Waters

1. Introduction

Thallium occurs in nature as an impurity in pyrites and blends. It is not usually found in natural waters. Its presence may be due to discharges from industries manufacturing insecticides and rat poison, photocells and lamps [1]. The determination of thallium is of primordial interest because despite its clinical, environmental and industrial applications [2], it is highly toxic for the biosphere. Its toxicity is even greater than that of mercury, cadmium, lead and copper [3]. Also, thallium is known to have mutagenic, teratogenic and carcinogenic effects [4]. The risks associated with this element require that its concentration be systematically measured. Unfortunately, it has not received as much attention from

researchers because it is difficult to measure using traditional techniques and there are few guidelines for this element. The American standard for drinking water is $2 \cdot 10^{-3} \text{ mg/L}$ [5].

Thallium has not been studied extensively. One of the reasons may be that classical analytical methods have poor sensitivity for thallium [6]. Preconcentration procedures, selective separation and purification methods are often needed for thallium determination because its concentration is too low to be determined [7]. This has pointed out the need for development of highly sensitive and reliable methods that would be able to determine ultra-trace thallium levels [8].

Some analytical techniques are available for the determination of trace amounts of Tl in environmental samples such as flame atomic absorption spectrometry (FAAS) [9], neutron activation [10], electrothermal vaporization inductively coupled plasma

mass spectrometry (ICP-MS) [11] and colorimetry [12], but all of these methods require one or more separation and preconcentration steps such as liquid-liquid extraction (LLE) [13], solid-phase extraction [14] and cloud point extraction [15].

Electrochemical techniques forming a very suitable approach were used to trace analysis in the solution. The most popular one, for environmental samples, is the anodic stripping voltammetry (ASV). This technique is very sensitive for many metals, it offers the capability of low cost multi-element analysis and it is suitable for automation. In particular, it is favourable for the determination of thallium [16, 17]. However, over the last 20 years, stripping analysis has been widely used in all the aquatic trace metal chemistry including drinking water [18]. For many metal ions (e.g. Pb, Cd, Cu, and Zn), differential pulse anodic stripping voltammetry (DPASV) at a mercury film electrode is used [19]. For DPASV, the pre-concentration step involves reduction of the metal ions followed by amalgamation with the mercury electrode [19]. In the trace range, however and for thallium analysis, the mercury film electrode (MFE) formed in situ during the plating stage on a specially prepared glassy carbon (GC) substrate electrode is required. For this purpose, the analyte is spiked with mercury (II) ion. During the plating step, the solution is stirred to speed up the mass transfer [19].

In this work, the field of mercury film is revisited with the goal of a simple, precise, and accurate determination of Tl^+ ions in an aqueous matrix using differential pulse anodic stripping voltammetry (DPASV). We first studied the optimal analytical parameters for the determination of trace amount of Tl^+ in aqueous matrix and after an application of this sensor on environmental waters was done.

2. Experimental

2.1. Electrodes, Instrumentation

The electrochemical experiments were carried out using a

Voltalab Analytical Radiometer running with Volta Master 4 Software. Differential Pulse Anodic Stripping Voltammetric (DPASV) curves were recorded in a three electrode cell set-up. The working electrode is an EDI 101 rotating disc having a glassy carbon tip (diameter 3 mm). A silver chloride Ag/AgCl, KCl sat was used as reference electrode and a platinum wire as auxiliary electrode.

2.2. Reagents

All the chemicals used were of analytical grade. Aqueous solutions were prepared with Milli-Q. The supporting electrolyte is perchloric acid $HClO_4$ (trace analysis). Mercury nitrate (purity > 99.99 %, Merck), thallium sulphate (purity > 99.995 %, Sigma Aldrich) dissolved in $0.1 \text{ mol.L}^{-1} HClO_4$, a certified standard of thallium 1 g.L^{-1} (Certipur, Merck) were used for preparation of standard solutions. Pure argon is used for degassing the solution.

2.3. Analytical Procedure

The mercury thin film was deposited on a carefully polished glassy carbon electrode (GCE) with abrasive papers of decreasing granulometry: 1200, 2400, 4000 keeping the working electrode at a potential of 0.2 V. The electrochemical determination of Tl^+ in aqueous solution using DPASV were performed with the following steps:

- 1) 19.9 mL of thallium solution prepared in the supporting electrolyte and $100 \mu\text{L}$ of $10^{-3} \text{ mol.L}^{-1} Hg^{2+}$ ions introduced into the electrochemical cell for optimization studies;
- 2) 9.9 mL supporting electrolyte, 10 mL water sample and $100 \mu\text{L}$ of $10^{-3} \text{ mol.L}^{-1} Hg^{2+}$ ions for real samples analysis.

After degassing with argon for ten minutes, the voltammogram is recorded by varying the potential towards the positive direction. The standard addition method was used to evaluate the content of thallium in water samples.

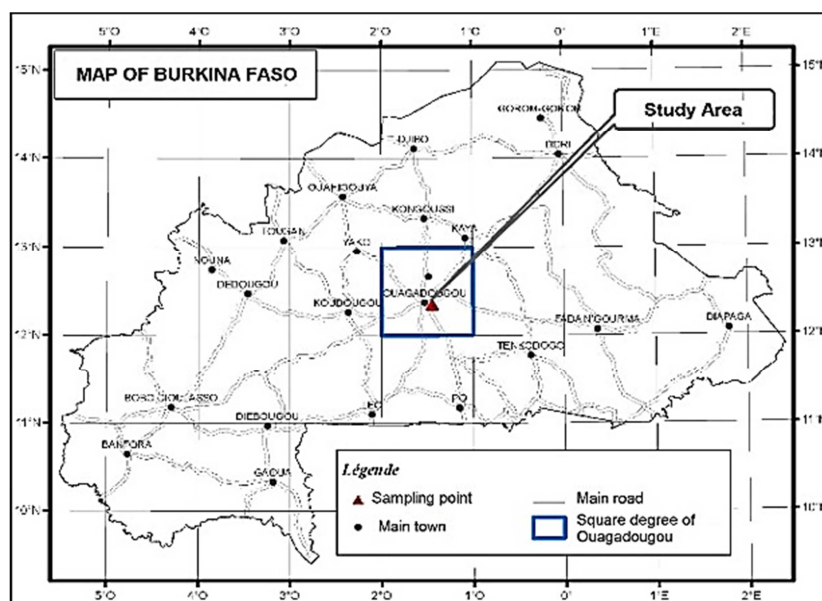


Figure 1. Localization of the study area and sampling sites on the map of Burkina Faso.

2.4. Real Samples

The real water samples were taken at eleven (11) water points, including nine (9) boreholes and two (2) wells in the village of Yamtenga. This village is located south-east of the city of Ouagadougou (Burkina Faso), with geographical coordinates in Universal Transverse Mercator (UTM): X = 669126, Y = 1363944. the aforementioned village belongs to the rural commune of Bogodogo, which is one of the districts of the commune of Ouagadougou. Sampling was done each three months on a period of one year. Samples are numbered as follows: B1, B3, B4, B5, B6, B7, B9, B10, B11 for boreholes water samples and W2, W8 for wells water samples (Figure 1).

3. Results and Discussion

3.1. Optimization of Analytical Parameters for Thallium Determination in Aqueous Solution

The electrochemical sensor for the determination of Tl^+ ions is implemented by immersing the GCE substrate/Hg film in the Tl^+ solution at the concentration C in the supporting electrolyte $HClO_4$ 0.1 mol.L⁻¹. The optimal conditions for the formation of the mercury film on the surface of the GCE were first determined: deposition potential equal to 0.2 V; deposition time equal to 4 min. The aim here is to optimize all the analytical parameters for determining the concentration of Tl^+ ions in an aqueous matrix. These analytical parameters are: deposition potential, deposition time, electrode rotation speed, pulse amplitude.

3.1.1. Deposition Potential

The deposition potential is a parameter that has a significant influence on the sensitivity of heavy metal determination [20-22]. The electrochemical reaction of deposition corresponding to the incorporation of Tl^+ ions into the mercury amalgam is:

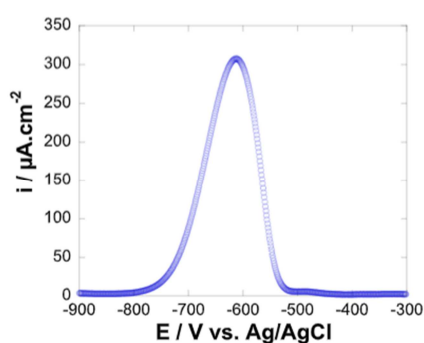
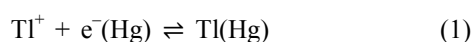
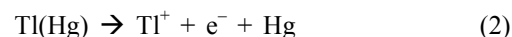


Figure 2. Voltammogram of 10^{-6} mol.L⁻¹ Tl^+ solution (deposition potential -900 mV, time of deposition 120 s, speed of the rotating electrode 500 revolution per minutes (rpm), amplitude of impulsions 40 mV).

This reaction is controlled by the diffusion of Tl^+ ions into the mercury amalgam. Following the recording of a voltammogram by differential pulse anodic stripping voltammetry of a standard solution of Tl^+ ions 10^{-6} mol.L⁻¹, a peak is observed at -650 mV

(Figure 2) which corresponds to the oxidation of Tl deposited on mercury according to the reaction:



It is then necessary to see which potential can lead to a maximum of deposition. At different deposition potentials between -600 mV and -1200 mV, the voltammetric peak was measured (Figure 3). The currents increase significantly for the first applied potentials. This increase becomes less significant for more negative values below -1000 mV. Indeed, the more negative the deposition potential, the greater the quantity of metal deposited on the mercury film until the latter is saturated [23]. The potential at which the highest peak was recorded, is -1000 mV. Below -1000 mV, the current decreases slightly. It would indeed be difficult to obtain a more consistent deposition of Tl below -1000 mV due to competition from the proton discharge. This potential of -1000 mV was chosen as the deposition potential for the rest of our study.

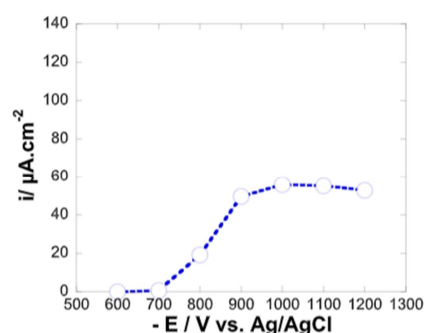


Figure 3. Influence of deposition potential ($[Tl^+] = 10^{-6}$ mol.L⁻¹, supporting electrolyte $HClO_4$ 0.1 mol.L⁻¹, deposition time 2 min, electrode rotation 1000 rpm, pulse amplitude 40 mV).

3.1.2. Deposition Time

The effect of the deposition time or pre-electrolysis time was studied by varying this time from 0 to 300 s. The results obtained are shown in Figure 4. We observe an increase in the intensity of the peak by prolonging the deposition times. This phenomenon has been noted by authors such as Ficher and van den Berg *et al.* [23]. A deposition time of 240 s was adopted for the rest of the work.

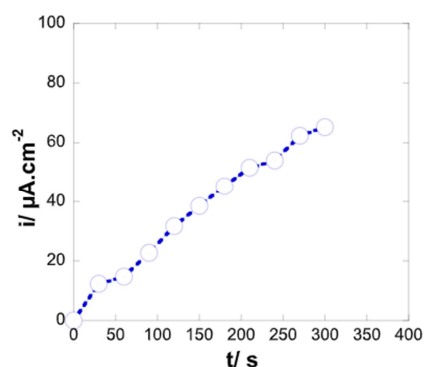


Figure 4. Variation of peak currents for different deposition times ($[Tl^+] = 10^{-6}$ mol.L⁻¹, supporting electrolyte $HClO_4$ 0.1 mol.L⁻¹, deposition potential -1000 mV, electrode rotation 1000 rpm, pulse amplitude 40 mV).

3.1.3. Electrode Rotation Speed

The rotating disc electrode is used to increase the sensitivity of the method because of the hydrodynamic flow of the electrolyte created under the electrode surface during the metal ion deposition step [24]. The influence of the electrode rotation speed between 100 and 1000 revolutions per minutes (rpm) was studied and the results are shown in Figure 5. A linear relationship between the peak current and the electrode rotation speed was obtained. This behavior reflects the importance of this parameter. In effect, during pre-electrolysis, the metal supply to the mercury film is ensured by diffusion and convection, which affects the yield of the metal deposit. For the rest of the work, it was adopted the highest value delivered by our speed controller, i.e. 1000 rpm.

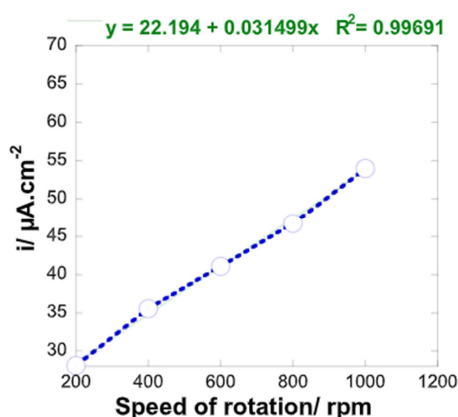


Figure 5. Influence of the electrode rotation speed on the peak currents ($[Tl^+] = 10^{-6} \text{ mol.L}^{-1}$, supporting electrolyte $HClO_4$ 0.1 mol.L^{-1} , deposition potential -1000 mV , deposition time 4 min , pulse amplitude 40 mV).

3.1.4. Pulse Amplitude

The effect of four pulse amplitudes on the peak currents was checked (Figure 6). For all four values, the linearity is well verified. Due to the low thallium contents to be investigated in natural matrices, the 80 mV amplitude, giving more intense and better resolved peaks, was chosen.

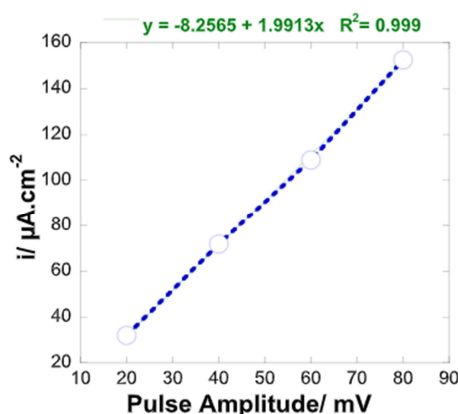


Figure 6. Influence of pulse amplitude ($[Tl^+] = 10^{-6} \text{ mol.L}^{-1}$, supporting electrolyte $HClO_4$ 0.1 mol.L^{-1} , deposition potential -1000 mV , deposition time 4 min , electrode rotation 1000 rpm).

3.2. Analytical Application of the Sensor

3.2.1. Performances of the Mercury Film-Based Sensor

The performance of the mercury film-based electrochemical sensor for the detection of thallium in water, was verified, by determining the accuracy, precision and detection limit of this sensor [25]. For accuracy, using the optimized parameters, the response of the electrode is verified by determining the concentrations of Tl^+ standard solutions by the standard addition method. An initial Tl^+ concentration of $5.10^{-7} \text{ mol.L}^{-1}$ was used and successive additions of $10 \mu\text{L}$ of $10^{-3} \text{ mol.L}^{-1} Tl^+$ were made. The results obtained are shown in Figure 7. In the range of concentrations explored, the calibration curve obtained is linear with a correlation coefficient of 0.9973 . The limit of detection (LOD) is calculated according to the following formula [20, 26]:

$$\text{LOD} = k.Sy/a \quad (3)$$

k is a constant which is 3 according to IUPAC, Sy is the residual standard deviation between measurements, a is the slope of the calibration curve. The calculated detection limit is equal to $2.2 \cdot 10^{-8} \text{ mol.L}^{-1}$ ($4.5 \mu\text{g.L}^{-1}$). A certified standard of thallium at 1 mg.L^{-1} is determined by the standard addition method. The recovery rate obtained is 99.14% . With regard to precision, the reproducibility between measurements was evaluated. The $i = f(E)$ curves were recorded 5 times for the same Tl^+ concentration, using the optimized parameters. The SRD (Standard Relative Deviation) for a constant Tl^+ concentration of $10^{-6} \text{ mol.L}^{-1}$ is 1.25% . Based on these results, the sensor appears to be reliable for the determination of Tl^+ ions in aqueous media.

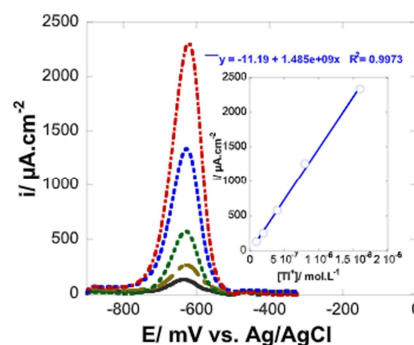


Figure 7. Calibration Curves and plot ($[Tl^+]$: $5 \cdot 10^{-7} - 2.5 \cdot 10^{-6} \text{ mol.L}^{-1}$, supporting electrolyte $HClO_4$ 0.1 mol.L^{-1} , deposition potential -1000 mV , deposition time 4 min , electrode rotation 1000 rpm , pulse amplitude 80 mV).

3.2.2. Comparison of Mercury Film-Based Sensor with Other Electrochemical Techniques for Tl^+ Determination in Aqueous Media

The performance of our sensor, for Tl^+ determination, is evaluated and compared with other electrochemical sensors as shown in table 1. The results show that our electrode has a good reproducibility (in term of % RSD) and high sensitivity (LOD) for determination of Tl^+ ions as other previously electrodes reported in the literature [27-32].

Table 1. Comparison of determination of thallium by electrochemical techniques.

Electrode	Technique	LOD ($\mu\text{g.L}^{-1}$)	RSD (%)	Reference
AuNPs-LS/Hg/GCE	DPASV*	28.56	14.27	[27]
Sb film/GCE	SWASV**	2	3.8	[28]
4-carboxybenzo-18-crown-6-SPCE/CNF	DPASV	10.9	2.8	[29]
T-MWCNTs/ITO/glass	DPASV	1.29	1.16	[30]
MeO/DPTU/GCE	SWASV	55.18	2.8	[31]
DCA-GCE	SWASV	4.08	1.57	[32]
MFE/GCE	DPASV	4.5	1.25	Present work

* Differential Pulse Anodic Stripping Voltammetry, ** Square Wave Anodic Stripping Voltammetry.

3.2.3. Real Samples

The analytical utility of this sensor is checked by applying it to the determination of thallium contents in real samples of borehole and well water from the village of Yamtenga. The thallium contents of all waters are grouped in the histogram of

Figure 8. The levels vary from $0.0732 \mu\text{g.L}^{-1}$ to $3.94 \mu\text{g.L}^{-1}$. The concentration peaks exceeding the American standard [5] were recorded at points W2, B5, W8 and B11. These waters are therefore not recommended for use as drinking water and remedial treatment should be considered for polluted waters.

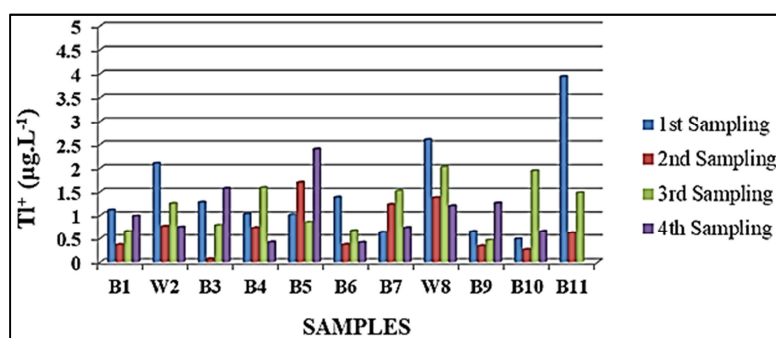


Figure 8. Spatial and temporal evolution of the thallium content of the analyzed water points.

4. Conclusion

The optimal analytical parameters for the determination of thallium on mercury film by anodic stripping voltammetry in aqueous media are: deposition potential -1000 mV, deposition time 240 s, electrode rotation speed 1000 rpm, pulse amplitude 80 mV. The thallium content of groundwater used as a source of drinking water by the rural population of the village of Yamtenga was analysed in order to assess the quality of this water. Thallium levels above the standards were found in some water points and declassify the groundwater as drinking water. It is therefore necessary to purify these waters in order to eliminate this metallic pollutant.

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