

## CHAPTER VI

### CONCLUSIONS AND FUTURE PERSPECTIVE

#### 6.1 Conclusions

The automated analytical methods have been successfully developed for the determinations of As(III), As(V) and Hg(II) by a combination instrument of an SI system and an electrochemical detection using Au-SPCEs. The summary of the optimized conditions for each method is shown in Table 6.1. Firstly, As(III) can be determined by using renewal Au-SPCE as the working electrode. Because the modified Au is difficult to remove, the Au can be modified as a thin layer. Therefore, a maximum volume of Au(III) solution for modification of Au is 0.3 mL that the modified Au can be completely cleaned out by 2.5 mL of the cleaning solution. Under the optimized condition, the detection limit of As(III) concentration was found to be  $0.1 \text{ mg L}^{-1}$ . Although the detection limit is insufficiency, this method requires a small sample volume of 0.3 mL. Secondly, the method for the determination of As(III) has been developed by using a long-lasting Au-SPCE. The cleaning potential of +0.4 V was used to remove the residual As, while the Au layer is still maintained on the SPCE. Due to its durability, the long-lasting Au-SPCE can be used for several times without the re-preparation of Au on the SPCE. The speciation between As(III) and As(V) has been achieved. The detection limit of As(III) concentration was improved to be as low as  $0.03 \text{ } \mu\text{g L}^{-1}$ , whilst the detection limit of As(V) concentration was  $2.3 \text{ } \mu\text{g L}^{-1}$ . Moreover, this method using long-lasting SPCE has been successfully applied to determine As(III) and As(V) in real water samples with excellent accuracy. Lastly, the SI-ASV using Au-SPCE has been developed for the determination of a highly toxic metal, Hg. The presence of  $\text{Cl}^-$  is a challenge because it has significant effect to the determination of Hg by electrochemical detection. The on-line medium exchange and a two-step deposition potential were applied to reduce the effect of interferences. The detection limit of Hg(II) concentration was found to be  $0.22 \text{ } \mu\text{g L}^{-1}$ . Using the proposed method, the determination of Hg(II) has been successfully applied in real samples with high  $\text{Cl}^-$  level such as sea water and table salt solution.





**Table 6.1** Comparison of the conditions used in each Chapter for the determinations of the metal ions by SI-ASV using Au-SPCE.

Conditions	Chapter IV	Chapter V	Chapter VI
Target analate (s)	As(III)	As(III) and As(V)	Hg(II)
Working electrode	Renewal Au-SPCE	Long-lasting Au-SPCE	Renewal Au-SPCE
Sample volume	0.3 mL	1.0 mL	0.9 mL
<b>Deposition step</b>			
- Concentration of Au(III) solution	5 mg L <sup>-1</sup>	5 mg L <sup>-1</sup>	0.5 mg L <sup>-1</sup>
- Volume of Au(III) solution	0.3 mL	9.2 mL	0.9 mL
- Deposition potential	One-step potential -0.5 V	One-step potential -0.5 V for As(III) detection (-1.5 V for total As detection)	Two-step potential -0.5 V for Au modification +0.2 V for Hg(II) deposition
- Type of supporting electrolyte solution	1 M HCl	1 M HCl	1 M HNO <sub>3</sub>
- Injection flow rate	10 μL s <sup>-1</sup>	10 μL s <sup>-1</sup>	10 μL s <sup>-1</sup>
<b>Voltammetry technique</b>	Linear sweep ASV	Linear sweep ASV	Square wave ASV
<b>Cleaning step</b>			
- Type of cleaning solution	1 M HCl	1 M HCl	1 M HCl and 1 M HNO <sub>3</sub>
- Volume of cleaning solution	2.5 mL	5.0 mL	2.5 mL of each solution

## 6.2 Future Perspective

SI system with electrochemical detection is composed of relatively small but robust instrument and it is a suitable choice for meeting the purpose of inventing a rapid and automatic method, especially, for water analysis. It would be intelligent automated system because the systems are under the computer-controlling that the operation conditions such as flow rates and injection volume can be easily adjusted for each procedure. The SI system can also be coupled with sample pretreatment or preparation components (for example packed column, SPE column, mixing chamber, etc.) to reduce tedious steps. Furthermore, an automated flow-based system can be miniaturized to integrate with a micro-channel flow-through detector like a microchip. Due to its flexibility and versatility, we believe that a wide variety of determinations based on SI system with electrochemical detection is being tried to invent and to develop by interested researchers around the world.

