CHAPTER 4
CONCLUSION AND SUGGESTION FOR FURTHERWORK

4.1 Conclusion

A reverse flow injection method in combination with UV irradiation system for speciation of selenium(IV) and (VI) as selenite and selenate in water has been developed by spectrophotometric detection. It is based on selenite oxidation of iodide to triiodide in a weak acid medium, resulting in a 1:1 ion-association complex of triiodide with a selective complexing agent, rhodamine B, in the presence of poly(vinyl alcohol). The reaction is very sensitive, reproducible and rapid. The method involved the injection of small amounts of rhodamine B into the three-line FI system. The purple-coloured complex is measured spectrophotometrically with a maximum absorption at 590 nm. The complex is formed from selenite; therefore, a prereduction step is necessary to reduce selenate to selenite for the determination of selenate. In this study, selenate is reduced on-line to selenite by UV irradiation system using UV lamp. On-line reduction in a closed system prevents losses of selenium during the prereduction step. Unlike other conventional methods, UV irradiation for the reduction of selenate to selenite is a simpler, cleaner and rapid method, since it does not require any additional reducing agent.

Various analytical parameters, such as acidity, flow rate and reagent concentration, etc., were optimized by univariate method to achieve the best sensitivity, selectivity, the least possible consumption of reagents and the most rapid analysis. This method has proven to be reliable. The proposed FI method is applicable over a broad linear range 0.0-1.0 ppm of selenite and selenate.

Working calibration curves were established over the range 0-0.5 ppm selenite and selenate, with the correlation coefficients of 0.9996 and 0.9960 for selenite and selenate respectively. The relative standard deviation for replicate injections are 1.21% and 2.37% for 0.2 ppm of selenite and selenate (n = 10). The method was very reproducible with the relative standard deviation of 1.83% and 4.43% for 0.2 ppm of selenite and selenate respectively (n = 10). The method was sensitive with the detection limit of 0.005 ppm selenite and 0.01 ppm selenate. High recoveries of spiked water samples was obtained (100.1% and 98.06 % for selenite and selenate respectively). The proposed FI method is free of interferences from most common ions such as sodium, potassium, magnesium, calcium, and etc. The proposed flow injection spectrophotometric method for speciation of selenite and selenate has been carried out under optimum conditions. The proposed method has been applied to the determination of selenite and selenate in spring water samples collected from different locations in Chiang Mai Province. The amounts of selenite and selenate were found in all samples, which these two contents were below the standard value of drinking water in term of selenium (0.01 ppm), which means that the waters can be safely supplied and used to drink. By comparing with hydride generation ICP-AES method, the results obtained by both FIA and ICP-AES are in good agreement. The proposed FI-spectrometric procedure has been successfully applied to the determination of selenite and selenate in water samples.

The advantages of the proposed FI-spectrophotometric method is that this method is simple, highly sensitive, rapid (2 species can be determined within 3 min) and safety because the system do not contain toxic solvents. The proposed method have good repeatability, reproducibility, accuracy and selectivity. Additionally, it requires low reagent and sample consumption and inexpensive instrumentation compared with complicated instrumental techniques. The proposed method can replace other conventional methods and

requires few preliminary treatments. It can be applicable to routine analysis of large numbers of water samples of widely varying compositions. The method can also be extended to the determination of selenium in other environmental matrices.

4.2 Suggestion for further work

(a) The proposed FI manifold can be improved for continuous monitoring. A flow injection system can be equipped with a computer or timer to automatically control the measurement and then evaluate data. An autosampler device can be used to obtain accurate and reproducible data.

(b) The FI system should be standardized using standard selenium solution daily to ensure that reproducible results are obtained.

(c) With slight modification the, FI-manifold could be used to determine a wide range of analytes.

(d) An on-line preconcentration and separation by the FI system with a microcolumn can be used to make adequate concentration for instrumental used in case of samples containing trace amounts of selenium.