CHAPTER V

CONCLUSIONS

Liquid-phase microextraction (LPME) followed by gas chromatography-electron capture detector (GC-ECD) was developed and applied for sample preparation and determination of trihalomethanes (THMs) and haloacetic acids (HAAs) in drinking water and tap water.

THMs were directly extracted from 10 mL water samples through extracting organic solvent immobilised in the pores and into a small volume (μL) of organic solvent present inside the porous hollow fiber. Experimental parameters such as types of hollow fiber membranes, extracting solvents, influence of sample volume, and extraction time were investigated. Polypropylene hollow fiber membrane, 1-octanol as extracting solvent, direct extraction mode at 35 °C for 30 min extraction were the optimal conditions for the analysis of THMs. Simple conditions such as extraction at room temperature, no stirring and no salt addition provided method detection limits in low μg/L and lower than the maximum contamination level (MCL) values of WHO, EU and US EPA regulations. The correlation coefficients (R^2) of THMs were greater than 0.9968 for the concentration range from 0.2 to 100 μg/L. The recoveries of spiked water samples THMs with 5 μg/L were between 98 and 105 % with relative standard deviations (RSD) less than 4 %. In comparison to the previous studies, i.e., headspace SPME [28] and headspace SDME [47], our method provided lower MDL.

In-situ derivatization followed by headspace LPME has been developed for the analysis of HAAs in water. The HAAs were directly derivatized in 10 mL water sample, followed by extraction and concentration with LPME in 10 mL headspace. 1-Octanol was the most suitable solvent for the analysis. The derivatization procedures such as extraction time, extraction temperature, methanol volume, salting out effect and stirring effect were optimized to achieve maximum sensitivity using the following condition: esterification for 60 min at 55 °C in 1 mL of methanol and 20 % Na_2SO_4 added water sample. The calibration curves were linear in the range studied for each analytes, with R^2 between 0.9904 and 0.9997. The method detection limits were in sub μg/L and lower than the MCL values of WHO and US EPA regulations. The relative standard deviations values obtained were satisfactory and ranged between 5 and 12 %

for all analytes. The major advantage of this procedure is that derivatization, extraction and preconcentration are combined into a single step. In addition, this method used acidic methanol as the derivatization agent instead of the hazardous diazomethane, and esterification was conducted in water instead of organic solvent. In comparison to the headspace method [23], our method showed lower MDLs, lower RSD and lower extraction temperature.

Application of these methods to water samples from Chulalongkorn University was performed. The results from survey water samples showed that the concentrations of THMs and HAAs were below the MCL values of WHO, EU and US EPA regulations.

In summary, it can be concluded that LPME has a great potential for the analysis of THMs and HAAs in drinking water. The major advantages of LPME in this work are that extraction and preconcentration are combined into a single step, only a few microliters of organic extractant per sample is used, the conditions for the analysis are simple, the extraction device is inexpensive and easy to make, and the method can provide high sample throughput.