Chapter 4

Conclusion

This study showed that Gas Chromatography - Mass Spectrometry can be used as a method for qualitative and quantitative analysis of TCDD residue in water. The optimum conditions for Gas Chromatography - Mass Spectrometry equipped with HP-5MS of 30 m X 0.25 mm i.d., film thickness 0.25 μm were as follow : carrier gas flow rate 1 ml/ minute, inlet temperatures 260 $^{\circ}$C, interface temperature 300 $^{\circ}$C. The column temperature was programmed at the initial temperature of 100°C, held for 3 minutes, ramped up 220 $^{\circ}$C at a rate of 30 $^{\circ}$C / min held for 2 minutes, then ramped up to 240 $^{\circ}$C at a rate of 5 $^{\circ}$C / min and held for 5 minutes before ramped up to 300 $^{\circ}$C at a rate of 20 $^{\circ}$C / min and finally held for 5 minutes. This column program could be used to analyze the TCDD with high precision, i.e. with RSD of less than 4 %. The results showed a linear dynamic range of 0.5 – 100.0 ng / mL with the correlation coefficiency of 0.9977. The detection limit of 16.9 ng / L was obtained for TCDD.

Since the TCDD residue concentration in water was very low, pre-concentration was necessary. In this study pre-concentration of TCDD was by the Amberlite XAD-2 resin adsorption. Amberlite XAD-2 resin was conditioned before used with sequencing series of methanol - water. The slurry of the conditioned XAD-2 resin was then filled into a column for water pre – concentration.

Traditional extraction of dioxin is a very expensive and time-consuming process due to the consumation of a lot of hazardous solvents. To overcome these disadvantages, a more economical, minimization of manual labour and faster extraction method, i.e. ultrasonic extraction, has been developed. In this ultrasonic extraction the energy transmitted through the bath water into the vials disrupts XAD-2 particles and ensured good contact between the particles and the solvent. The parameters affected the extraction efficiency were studied. The results found that, toluene-acetone mixture (1:1, v/v) was the best

extraction solvent and the optimum extraction time was 20 minutes. A 60 mL of the solvent was sufficient for the extraction and double extraction (2 X 30 mL) of toluene-acetone mixtures provided the highest extraction efficiency (93 %) with a good relative standard deviation (4 %). The advantages of XAD-2 column and ultrasonic extraction over the use of conventional technique are, more economical, minimization of manual labour, faster extraction and reduction of the amount of solvent and high extraction efficiency.

The analysis results of the two water samples from Songkhla Municipal Landfill showed the concentration of TCDD residue in water of 1.62×10^{4} pg / L and 2.52×10^{3} pg / L. The other from HatYai Regional Water Supply showed that concentration of TCDD residue in water less than 1.69×10^{3} pg / L. Dinkuns *et al.*, (1995) reported the US EPA water quality criterion of 1.3×10^{-2} pg / L. Therefore, the results were showed a much higher concentration than this quality criterion.

The techniques of Method 8290 for the analysis of dioxins was based on High Resolution Gas Chromatography – High Resolution Mass Spectrometry employing electron ionization. It provided procedures for the detection and quantitative measurement of dioxins in variety of environmental matrices and at part - per trillion to part - per quadrillion concentrations (US EPA, 1994). In this study it was shown that High Resolution Gas Chromatography – Low Resolution Mass Spectrometry is also a reliable technique which can be used the qualitative and quantitative analysis of dioxin residue in water. The XAD-2 sample preparation is relatively simple and its good adsorption and makes it more economical than conventional technique.