CHAPTER 4
CONCLUSIONS

Flow-based techniques for the determination of some heavy metals in environmental samples were developed.

Automated on-line solvent extraction devices coupled with FAAS

An automated on-line solvent extraction with FAAS for some heavy metals in soil

The system employs solvent extraction of aqueous trace metals as DDC complexes into MIBK fed directly to FAAS. The quantitative extraction of the metal ions, Cu(II), Pb(II) and Ni(II) was possible under an acidic condition. Thus, these metals can be measured after digestion. However, the pH of sample must be adjusted for Cd(II) and Zn(II) determination. The pH of 1.0 was selected for Cd(II) and Zn(II) determination. The procedure is especially suitable for analysing sample with high salt and Fe contents. Such a sample normally cannot be analysed without extensive sample preparation. The proposed method was successively applied for heavy metal determination in soil reference materials and soil samples. The results obtained by the proposed method agreed well with the certified value for soil reference materials. For heavy metals in soil samples, the results obtained by using the proposed method agreed well with those obtained by using conventional standard additional FAAS. The advantages of this method are to minimise

tedious sample preparation and avoid contamination. In addition, this method is rapid resulting in increased of sample throughput.

Cr(III) and Cr(VI) speciation analysis in tap water and leachate samples

By using the system in **Figure 2.1**, it could be also an alternative method for Cr(III)/Cr(VI) speciation. The proposed method was successively applied to speciate chromium in tap water and leachate samples. The off-line oxidation of Cr(III) to Cr(VI) using $Na_2S_2O_8$ associated with $AgNO_3$ was required. The feature of its automation offers the system suitable for routine analysis. %Recoveries of Cr(III) and Cr(VI) with various ratios spiked into the samples were satisfactory.

On-line preconcentration and preseparation by using a column extraction procedure coupled with FAAS

FI on-line preconcentration for lead determination

FI on-line preconcentration was utilized for lead determination. The single standard calibration graph (0.4-1.6 μg Pb) was obtained. The proposed method was applied for the determination of lead in water samples collected from an industrial estate. It was found that the results agreed well with those obtained from ICP-AES (t=1.09). The detection limit was 0.2 μg Pb and %RSD of 3 was obtained (0.80 μg Pb(II), n=11). Concentration efficiency for 14.6 min^{-1} was established.

SI - column preconcentration for iron determination

The preliminary work for SI-column preconcentration for iron determination was performed. The calibration was made by using normal calibration graph. This work offers the automatic system, while suffers in rapidness caused from long analysis time. Further investigation should be studied to achieve better performance.

Preliminary study for SI-ASV for arsenic speciation

As the results from the preliminary study here, it indicates feasibility for arsenic speciation by using SI-ASV. Further investigation should be carried out in order to obtain the successful method for arsenic speciation in real samples.