CHAPTER V

DISCUSSION AND CONCLUSION

A 200 fold increase in the concentration of Cd, Cu and Zn, a 400 fold increase in the concentration of Pb (above the normal level) could be obtained by chelex-100. A 400 fold enrichment of the four elements could be attained by the reverse phase chromatography. The recovery yields of Cd, Cu, Pb and Zn through chelex-100 at pH7.6 with a flow rate of 1.0 $cm^3/_{min}$ were found to be nearly 100%. The recovery yields through the reverse phase chromatography technique were found to be lower. After correction of the chemical loss in the reverse phase chromatographic technique, the results of the two methods agree very well with each other. Both methods seem to be time consuming, but several samples could be run at the same time.

Both concentration procedures can be used to concentrate trace elements from much larger volumes of sea water than can be conveniently handled by conventional solvent extraction. In chelex-100 only the transition metals are adsorbed. Thus the presence of much higher concentration of alkali and alkaline earth ions do not interfere. In reverse phase chromatography, the ammonium pyrrolidine-dithiocarbamate forms insoluble complexes with most metals

except the alkalis and alkaline earths. The complexes are adsorbed on chromosorb w-DMCS after which can be eluted with MIBK, $CHCl_3$ etc. As MIBK is toxic and gives bad smell on heating, $CHCl_3$ can undergo only partial combustion in outer zones of the flame, the metal complexes in the organic solvent were stripped back to aqueous phase with 6M HNO_3.

The interferences of the major components in sea water with the determination of Cd, Cu, Pb and Zn by the atomic absorption spectrophotometric method are recapitulated in Table 5.1. It is obvious that the separation step is necessary, otherwise accurate analytical results could not be obtained.

Table 5.2 Summary of the extent of interference of some cationic and anionic species in the determination of Cd, Cu, Pb and Zn by the Atomic Absorption Spectrophotometer (14).

Element	Anionic Species								
	BO_3	SiO_3	NO_3	PO_4	SO_4	ClO_4	F	Cl	CH_3COO
Cd	-	-							
Cu	0	-							
Pb									
Zn	0	0	0	+	0	0	0	0	+

Element	Cationic Species									
	Na	K	Mg	Ca	Sr	Al	La	Ti	Mn	Fe
Cd										
Cu										
Pb										
Zn	+	+	+	+	++	+	+	+	+	++

APPENDIX I

MEASUREMENT OF DETECTION LIMITS

Table A-I Absorbance of aqueous and standard solution.

Number of Readings	Absorbance			
	Zn	Cd	Cu	Pb
1	0.001	0.001	0.002	0.000
2	0.015	0.009	0.020	0.009
3	0.003	0.001	0.003	0.000
4	0.013	0.010	0.020	0.009
5	0.002	0.001	0.002	0.001
6	0.012	0.009	0.019	0.008
7	0.001	0.000	0.001	0.001
8	0.010	0.010	0.020	0.009
9	0.000	0.001	0.001	0.001
10	0.011	0.011	0.020	0.009
11	0.000	0.001	0.000	0.001
12	0.014	0.010	0.020	0.009
13	0.002	0.001	0.001	0.001
14	0.016	0.009	0.019	0.009
15	0.003	0.001	0.002	0.001
16	0.017	0.010	0.020	0.009
17	0.004	0.002	0.001	0.001
18	0.017	0.011	0.019	0.009
19	0.004	0.002	0.001	0.001
20	0.017	0.010	0.019	0.009
21	0.004	0.002	0.001	0.002

Note: 1. Zn and Cd concentration 0.04 $\mu g/cm^3$

Pb concentration 0.80 $\mu g/cm^3$

Cu concentration 0.40 $\mu g/cm^3$

2. There are 11 readings for the blank and 10 for the standard solution. Each reading for the standard is "blanketed" by a pair of blank readings.

Calculation example (from the measurement of Zn).

Procedure:

1) Average the absorbance for the first two blank readings. (Absorbance 1 and 3) $(0.001)+(0.003) \div 2 = 0.002$

2) Subtract this value from the corresponding reading for the Zn solution (Absorbance 2).

3) Repeat for each pair of blank readings for each reading from the Zn solution in order.

4) Continue as indicated above until 10 values are obtained.

5) Table A-II shows these values as an example.

6) Calculate the standard deviation (S.D.) from

$$S.D = \pm\sqrt{\frac{\Sigma d^2}{N-1}}$$

$$= \pm\sqrt{\frac{1.803\times10^{-5}}{10-1}}$$

$$= \pm\ 1.415\times10^{-3}$$

Table A-II Mean and deviation.

Number	Absorbance X	Deviation $X - \bar{X}$	Deviation $(X-\bar{X})^2$
1	0.0130	+0.00085	7.225 - 07
2	0.0115	-0.00065	4.225 - 07
3	0.0105	-0.00165	2.723 - 06
4	0.0095	-0.00265	7.023 - 06
5	0.0110	-0.00115	1.323 - 06
6	0.0130	+0.00085	7.225 - 07
7	0.0135	+0.00135	1.823 - 06
8	0.0135	+0.00135	1.823 - 06
9	0.0130	+0.00085	7.225 - 07
10	0.0130	+0.00085	7.225 - 07
	$\bar{X}$ =0.01215		$\Sigma(X-\bar{X})^2$ = 1.803 - 05

7) The detection limit

$$D = 2 \times S \times \frac{C}{\bar{X}}$$

$$= 2 \times .001415 \times \frac{0.04}{0.01215}$$

$$= 0.009 \ \mu g/cm^3$$