THE AUTOMATED CLEAN-UP OF PCBs AND DIOXINS BY SOLID PHASE EXTRACTION COUPLED TO HPLC (ASPEC)

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INTRODUCTION

The clean-up of PCBs and dioxins prior to identification and subsequent quantification by GC/MS is essential due to the often varied and high quantity of potential interferences.
Existing methodologies (1, 2) are very time consuming and labour intensive, especially in the packaging of columns, or in the column chromatography itself.
Solid phase extraction (SPE) has been widely used in the analysis of water and mycotoxins (3,4) with low level of constituents.
In this study, automated solid phase extraction has been investigated and optimised for the clean-up of PCBs and dioxins from various matrices. Recoveries and limitations of the process are discussed.

EXPERIMENTAL

Soil samples (25g) and foliage (10g) samples were spiked with reference internal standards and Soxhlet extracted with toluene for 18 hours. The samples were then concentrated by rotary evaporation and solvent exchanged to hexane 1(ml) and placed in the ASPEC sample pack for processing. A second set of standards were added at this stage to establish extraction efficiency. The SPE columns were prepared as follows:

Silica: 1g of kieselgel 70-230 mesh (Merck)
Florisil: 1g of florisil 60-100 mesh (BDH) - activated to 600°C for 24 hours.

All SPE columns were stored in a desiccator for a maximum of 24 hours before use. The samples are processed according to the schematic Table 1. The methodology allows internal standard to be added at any part of the sequence without any change to the hardware or software.

RESULTS AND DISCUSSION

The results of the clean-up efficiencies of PCBs from various matrices are shown in Table 2, whereas the efficiencies of the clean-up for PCDD and PCDF analyses are shown in Table 2A.
The efficiency of clean-up are consistent for each matrix at about 95%. However, plasma recovery efficiencies were lower at 75% which is probably due to the greater difficulty encountered in the extraction of PCBs from the matrix rather than the clean-up.
Table 3 illustrates the detected level of blank samples which were run in conjunction with the samples in order to check contamination in the ASPEC system. The results indicate that the ASPEC system is not susceptible to the problems of carry-over under normal operating conditions.

CONCLUSIONS AND LIMITATIONS

The ASPEC SPE system is capable of performing the automated clean-up of up to fifteen samples under operating conditions, and can be used for the batch clean-up of samples that are routinely monitored.
Under adverse conditions of high oil content, the ASPEC system may not be suitable or where high chlorophyll level in foliage are expected. The ASPEC may be further improved if coupled to an SFE system where the selected extraction of PCBs and dioxins may be possible.
Although no contamination from sample to sample was encountered, care must be taken when processing samples from different matrices, i.e. flyash and paper samples. The software for the ASPEC/HPLC control will be reported.

REFERENCES

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TABLE 1

SCHEMATIC OF PROCEDURE

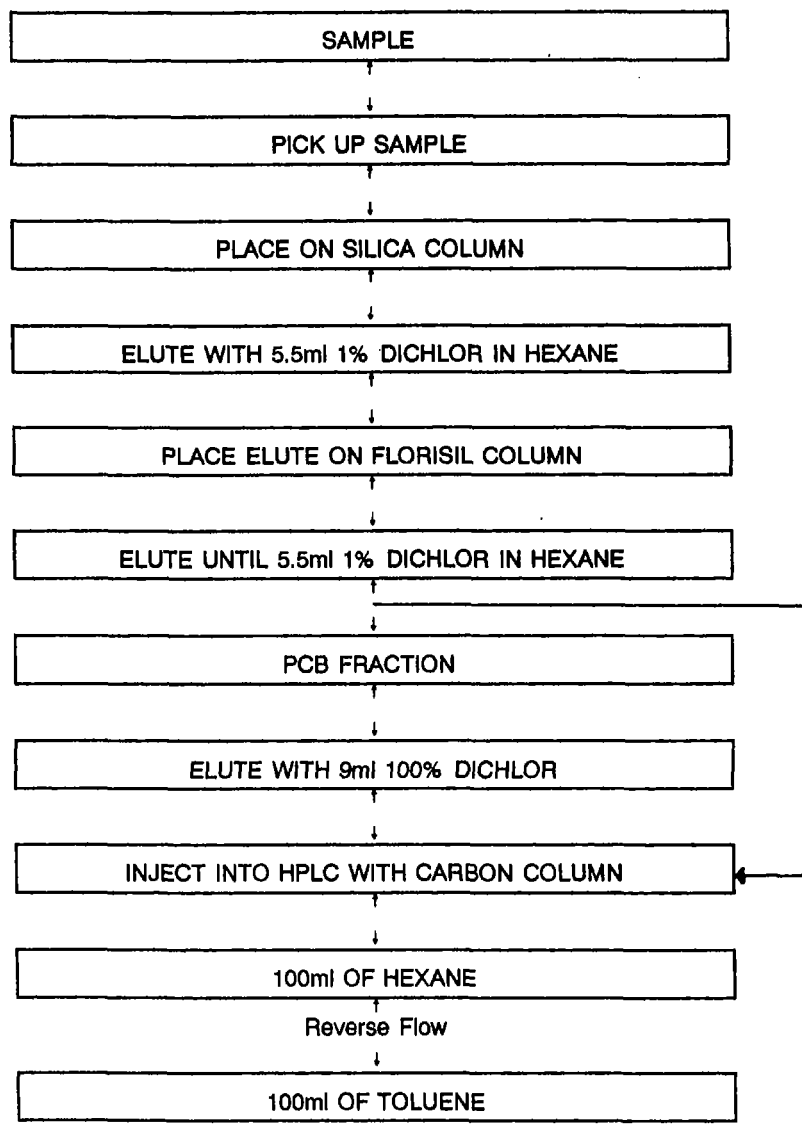

TABLE 2

(a) PCB Results

MATRIX	MEAN µg/g	STANDARD DEVIATION µg/g	RECOVERY %	STANDARD DEVIATION %
SOIL	5.46	0.21	96.4	5.7
GRASS	4.50	0.11	93.0	8.7
PLASMA	0.32	0.02	72.1	5.6

(b) Dioxin Results (TEQ)

MATRIX	MEAN ng/g	STANDARD DEVIATION ng/g	RECOVERY %	STANDARD DEVIATION %
SOIL	0.4	0.02	95	7.4
GRASS	0.6	0.03	97	6.2

TABLE 3

BLANK LEVELS FOR PCBs

	LEVEL µg/g
BLANK	ND
SAMPLE	2.16
BLANK	ND
SAMPLE	2.14
BLANK	ND
SAMPLE	2.10
BLANK	ND
SAMPLE	2.17
BLANK	ND

ND – Not detected

Detection Limit for PCBs is <0.01