

CHAPTER 4

4. Conclusion

The present trend in the development of pesticide residue analysis is towards multiclass and multiresidue methodology with adequate recovery characteristic (over 80% as a rule, but not less than 70%), good reproducibility and has low detection limits. In addition, it must be capable of determining as many pesticides as possible in the most rapid and accurate manner.

In this study, an attempt was made to develop a multiresidue method that would combine the analyses of organophosphorus (OP), organochlorine (OC), N-methylcarbamates (NMC), phenylureas and several other pesticides into a single procedure. Different classes of pesticides are extracted and cleaned up at the same time. No fractional collection is required. The final extract was injected into the LC-API-MS system.

This method has several advantages over other analytical methods. The API-MS detection which was performed in the SIM mode can combine both positive and negative modes of operation (PCI/NCI) in a single run. It can be switched to and fro from PCI to NCI mode. No derivatization step is required as compared to the current official methods for the determination of polar, non-volatile and thermolabile compounds such as NMCs and phenylureas. Furthermore, the use of a mass spectrometer as the detector gives a high degree of confirmation of molecular

identity as compared to methods based on fluorescence or ultraviolet detectors. It is also very time consuming to carry out the analysis of each pesticide or each group of pesticide with the official methods such as GC-NPD, GC-ECD and HPLC-UV. The NPD detector most commonly used for nitrogen and phosphorus containing pesticides and ECD detector for halogenated compounds. HPLC is the favoured technique for determination of N-methylcarbamate pesticides since many of them lack the thermal stability necessary for GC determination.

Extraction with 5 % (v/v) ethanol in ethyl acetate resulted in a multiresidue extraction. The polar pesticides could be quantitatively extracted. This solvent mixture has sufficient polarity to extract highly polar compounds such as methamidophos and dimethoate and yet is not miscible with water, thus minimising co-extracted water and avoid labour intensive steps such as liquid-liquid partitioning (LLP) and salting out. Residual water in the solvent extract can be removed by using anhydrous sodium sulphate. After using the appropriate clean up steps, matrix co-extractives were removed without loss of the more volatile or the polar pesticides.

The recoveries of 11 pesticides in this multiclass and multiresidue analytical method, their correlation coefficients and limits of detection (LOD) are presented in Tables (17), (16) and (15) respectively. This method also shows acceptable repeatability, reproducibility (Table 19), and accuracy (Table 18).

The FIA-API-MS has been demonstrated to be useful for the determination of various pesticide formulations. Quick analysis can be performed without any sample

pre-treatment process. This technique can be used for quantitative determination as well as for the positive identification of the active ingredient in the pesticide formulations and also shows potential usefulness in clinical toxicology to analyse the pesticide that has inadvertently entered the human body.