

Automated On-line Solid-Phase Extraction Methods in Interlaboratory Studies of Polar Pesticides in Water Samples

D. Barceló† and M. F. Alpendurada**

The use of automated on-line solid-phase extraction followed by liquid chromatography diode array or mass spectrometric detection for the determination of polar pesticides in water matrices is discussed. Emphasis is given to the participation in interlaboratory exercises organized in Europe by the Aquachek and the Measuring and Testing program of the European Union. Criteria of the coefficient of variation in interlaboratory exercises for polar pesticides in water is given. © 1997 John Wiley & Sons, Inc. Lab Robotics and Automation 9: 165–174, 1997.

INTRODUCTION

The increasing requirements to demonstrate comparability of analytical data in environmental monitoring demand some external assessment of the quality of the results provided by individual laboratories. One way of doing this is by assessment of the performance in interlaboratory comparisons using centrally distributed samples [1]. Within the European Union, the former BCR (now Measuring and Testing) organizes different types of interlaboratory studies in environmental and other matrices. Interlaboratory tests provide a means of detecting and guarding against undiscovered sources of errors. In this re-

spect, the three major sources of errors detected in interlaboratory studies were classified [2] as these:

1. sample pretreatment, e.g., extraction, preconcentration, and separation;
2. final measurement, e.g., calibration errors, spectral interferences, and coelution of peaks; and
3. the laboratory itself, e.g., training and educational levels of researchers, technicians, and management.

For the analysis of pesticides in water, the sample pretreatment consist most of the time of extracting traces of pesticides from the aqueous media, concentrating these traces and removing from the matrix other components that have been coextracted and concentrated and that may interfere in the chromatographic analysis (cleanup). Sample pretreatment is still the weakest link and the time-determining step in the whole analytical procedure, accounting for about two-thirds of the total analysis time and being the primary source of errors and discrepancies between laboratories. The development of more rapid and reliable strategies requires removal of intermediary steps such as transfer, evaporation, and derivatization techniques that maximize sample throughput. Automated devices that couple the sample pretreatment using solid-phase extraction (SPE) and the chromatographic separation on-line were introduced in the last few years (OSP-2, Prospekt, . . .). Such automated on-line SPE systems offer the possibility of a complete automation and avoid manipulation of samples [3]. In this respect, the use of such

*Laboratory of Hydrology, Faculty of Pharmacy, University of Porto, Rua Anibal Cunha, 164, 4000 Porto, Portugal.

†Author to whom correspondence should be addressed. Permanent address: Department of Environmental Chemistry, CID-CSIC, c/Jordi Girona, 18-26, 08034 Barcelona, Spain.