Validation of a Method to Test for Pesticides Residues

Pauline Falzon1, Ramon Zammit2, David Spiteri2, Paula Grech Bonnici2, Anthony Serracino-Inglott1

1Department of Pharmacy, Faculty of Medicine and Surgery, University of Malta, Msida, Malta, 2Compliance and Quality, Water Services Corporation, Luqa, Malta.
pauline.falzon.15@um.edu.mt

INTRODUCTION

There is the need to develop a pesticide testing laboratory in Malta as samples are analysed abroad. Testing for pesticides residues in agricultural crops is a complex process because of the large number of pesticides used and the low concentrations allowed.

AIMS

To validate a multi-residue method in an ISO 17025 accredited laboratory intended for the routine analysis of pesticide residues in local agricultural crops to check for compliance with EU legislation on maximum residue levels, and to evaluate food safety and potential risks.

METHOD

Sub-portions of homogenized samples were treated according to the QuEChERS method that combines two stages: the extraction stage where the sample is extracted using acetonitrile and buffer salts, and the dispersive solid phase extraction (SPE) stage where the clean-up of the extract is carried out by the addition of primary secondary amine. The analyses of organophosphorus and organochlorine compounds was carried out by the instrumentation triple-stage quadrupole GC-MS/MS, and GC-ECD, respectively.

RESULTS

Area ratio versus pesticide concentration were fitted using linear regression and basing the regression on the ratio of the analyte response to the internal standard triphenyl phosphate to obtain the equation for GC-MS/MS calibration curves of the tested pesticides. Duplicate measurements allowed the precision of the calibration process to be evaluated, and assured that the tests run were valid and the results obtained were reliable. Good linearity and reproducibility of calibration curves were achieved. In GC-ECD, hexane detected the greatest number of pesticides (among the tested chemicals) indicated as peaks at the highest frequency intensity.

CONCLUSION

Lower levels of pesticides were detected in samples homogenized without dry ice. Losses of pesticides may have occurred through hydrolysis by free water, oxidation, formation of insoluble complexes, enzymatic degradation or degradation due to pH. A high percent recovery of a number of pesticides in local vegetables was achieved by an accredited national reference laboratory from an EU member state, using the same method without deviations to the process. The concentration of sorbitol at which pesticides showed the strongest positive linear correlation between the area ratio and analyte concentration, and the solvents most suitable for the detection and stability of a number of multiclass pesticide residues were determined. The method provides acceptable precision, accuracy, and linearity when applied to general crops. The method has a limitation for use in pigmented or fatty products.