

Practical experience gained with testing the effect of sample processing on the stability of residues and uncertainty of the results

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Neither recovery tests nor proficiency tests provide information on the magnitude of variability and the bias of measured residues resulted from the inhomogeneity of processed sample material in terms of mass of test portion and the decomposition/evaporation of test substance during sample processing.

Significant reduction of concentration of some pesticide residues and substantial increase of the uncertainty of the results derived from the homogenization of sample materials have been reported in scientific papers long ago. Nevertheless, method validation results are still reported based on recovery tests only.

To demonstrate the potential consequences of sample processing, tomato, lettuce and maize were surface treated with a test mixture consisting of 20 pesticide residues in known concentration including those of stable (buprofezin and chlorpyrifos) unstable (chlorothalonil chlozolinate, dichlorvos, etridiazole) and unknown properties. To simulate the worst case scenario, the treated samples were mixed with untreated ones in 2:8 and 1:9 ratios, respectively. The samples were chopped with Stephan blender at ambient temperature as well as under cryogenic processing conditions. Maize was ground at ambient temperature only. One gram and 10 g test portions were withdrawn and analyzed with appropriate variants of QuEChERS method. The stability of analytes was determined based on the response ratios of residues of individual analyte and buprofezin. The ratios were also compared to those obtained with recovery tests conducted with spiked test portions.

In agreement with previous studies, the results indicated that cryogenic processing resulted in less loss of some residues and increased the reproducibility of the determination. Furthermore, it was found that the efficiency of sample homogenization depends on the equipment and the sample material, but independent from the analyte. Assuming that only one or a few types of chopper and grinder are used in a laboratory, the major variable in the determination of pesticide residues is the type of sample material (variety, maturity of fruits and vegetables). Consequently, the efficiency of sample processing should be regularly tested as part of the internal quality control of the analytical processes. The stability of analytes should be tested during the validation of the method at least on representative crops selected from the crop groups included in the DG SANTE Guidance document.