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Comparison of three preparation procedures for determination of Organochlorine pesticides in food of animal origin (fat)

Dr Ivan Pecorelli, Mrs. Angela Paoloni, Mrs. SabrinaAlunni, Mr. Alessandro Pelliccia
Istituto Zooprofilattico Sperimentale dell'Umbria e delle Marche, Laboratorio contaminanti ambientali, Perugia

Residues of pesticides can be expected in food of animal origin, because of their application on plants used for feed production intended for animal consumption or treatment for stable hygiene. Usually the pesticide class investigated in fat of animal origin is Organochlorines.
A number of sample preparation techniques and methods of analyses, have been developed for the determination of pesticide residues in wide range of food of animal origin. Previously, analyses of pesticides from food of animal origin were performed with an accelerated solvent extraction (ASE) step by a solvent mixture of n-hexane and acetone with a suitable ratio (example 3:2 v/v), fat separation was then carried out with gel permeation chromatography (GPC) and a clean-up step by SPE silica columns was finally performed. Most of these procedures are time and solvent consuming and incurring in consequent cost of analyses. An alternative method may be QuEChERS, developed for the determination of pesticide residues in food of plant origin, but now used also for other matrices such as milk, eggs or food of animal origin in general.
The aim of this work was to evaluate the application of QuEChERS method, coupled with GC-MS/MS (QqQ) detection, in Multiple Reaction Monitoring (MRM), for the determination of Organochlorine pesticide residues in fatty animal matrices, alternatively, to classic preparation. The performance of two different types of dispersive solid phase extraction methods (d-SPE), with the same concentration of $MgSO_4$ (900 mg) and PSA (150 mg) and different concentration of C18, 150 mg and 400 mg respectively, were evaluated and compared to validation data obtained by classic ASE/GPC method.
Despite good results in terms of recovery and trueness, the ASE/GPC method is very complex and time consuming while classical QuEChERS (150 mg of C18) does not show good results due to the presence of co-extracted substances from matrix (6 molecules have low recovery and only 4 out of 25 show RSD below 20%).
High C18 (400 mg) QuEChERS method validation results, obtained from 5 replicates, showed mean recovery in the range of 70-120 %, and good RDS value below 15%, in accordance with performance described in Document N° SANTE/11945/2015, for all the 25 molecules analyzed. Only Hexachlorobenzene (HCB) has a mean recovery value of 60%.
The proposed method is now applied in laboratory for routine samples and will be tested in the next EUPT-AO on pig lard.