

VALIDATION PROCEDURE OF PESTICIDES SUCH AS α - BHC, γ - BHC, β - BHC, DDE, DDD AND DDT IN FRUITS AND VEGETABLES GROWN IN AZERBAIJAN

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Fruits and vegetables are important part of healthy ration because of the presence of significant amount of nutrients and minerals in them. Almost all fruits and vegetables contain much kind of vitamins such as B group vitamins, carotenes (α , β carotenes, lutein), vitamin C, etc. However, at the same time they can also turn out to be the source of toxic substances such as pesticides. Pesticides are unhealthy residues, which are commonly used in agriculture field to protect the plants, crops from different insects and diseases. The use of pesticides have increased because they have quick action, decrease toxins produced by forth infecting organisms and are less labour intensive than other insects, herbicides and diseases control methods.

Approximately 30 - 40 years ago, in soviet period, pesticides such as α - BHC, γ - BHC, β - BHC, DDE, DDD and DDT are very intensively used in cotton plant fields in Azerbaijan territory. Then using of these pesticides was prohibited. But despite this pesticides above mentioned can remain in long time in soil. Therefore investigation and controlling of MRL (maximum residual level) was very important in Azerbaijan for protection of health of consumers.

In food testing laboratory were carried out validation of pesticides, α - BHC, γ - BHC, β - BHC, DDE, DDD and DDT in fruits and vegetables using the method BS EN 15662:2008 -Foods of plant origin, Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and clean-up by dispersive SPE, QuEChERS-method. As a matrix was used apple without any pesticides including above mentioned substances. Calibration standard solutions were prepared at five concentration levels 10, 20, 50, 100, 500 ppb for each pesticide. The validation plan is two analysts in same and different periods (for estimation repeatability and reproducibility) carried out 10 times testing together and separately on spiked sample in three concentrations (10 ppb, 100 ppb and 500 ppb) using GC-MS equipment. The recovery of analysis for each pesticide in three concentrations was 76.23 – 125%. The CV_R were among 9,37 – 26%. For monitoring the validity of the method, the laboratory participated into proficiency test the results are within the acceptable Z score. As a result, recoveries, RSD_r , and RSD_R values, calibration curve, specificity, LOG, trueness (bias), matrix effect for each pesticide were within the acceptable range according to the criteria specified in SANCO/12571/2013 19 November 2013 rev. 0. During the validation procedure, expanded uncertainty for each analysed pesticides was estimated in different concentrations. There were varies between from 0,199 ppb to 0,787 ppb.

[1] BS EN 15662:2008 -Foods of plant origin, Determination of pesticide residues using GC-MS and/or LC-MS/MS

[2] SANCO/12571/2013 19 November 2013 rev. 0