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DETERMINATION OF PESTICIDE RESIDUES BY CHROMATOGRAPHIC METHODS FOR FOOD SAFETY

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Actuality.In the process of growing oilseeds, leafy and stem vegetables use plant protection products, the active components of which are pesticides of different groups. The content of residual amounts of pesticides is controlled according to standardized methods. The methodology for measuring safety indicators is being actively developed today. In the process of modernization of methods for the study of pesticide residues, we can distinguish the stages of sample preparation and instrumental studies of plant extract. The analysis of xenobiotics uses the QuEChERS method (quick, easy, cheap, effective, rugged and safe). This method of laboratory control does not allow to remove some xenobiotics.

The purpose of the research. The purpose of this work is to establish the optimal conditions for the preparation of samples of crop products for the extraction of xenobiotics, to establish their qualitative and quantitative composition by chromatographic methods of laboratory control.

The materials and methods for investigation. Samples of crop products were used in the work: oilseeds (sunflower, soybean, flax), lettuce of different varieties, apples of different varieties. Several parallel laboratory samples were formed, of which

three samples were subjected to artificial enrichment with xenobiotics. Homogenization of samples was performed by grinding in a beaker of a laboratory mill-homogenizer at different temperatures. Chemicals of the qualification "ch.d.a" were used to obtain the plant extract: acetonitrile, methanol, acetone, n-hexane, toluene, isopropanol, acids (acetic, formic, trifluoroacetic, hydrochloric). These compounds were used as individual extractants, or in mixtures, including in a mixture with deionized water. Intensification of mass transfer during the extraction of analytes occurred when varying the ratio of raw material-extractant, under the action of temperature, stirring, ultrasonic waves. The separation of the phases of the extraction system was performed using a centrifuge. The obtained vegetable extract was subjected to purification from coextractive substances by methods of dispersion extraction or liquid-liquid extraction. The analysis of the content of xenobiotics was performed by high-performance liquid and gas chromatography with mass spectrometric detectors (HPLC / MS / MS and GC / MS).

The results and discussion. Homogenized sunflower seeds contain a significant amount of fat. The main component of homogenized samples of lettuce and apple fruit is water. For fine-grained homogenized samples of sunflower seeds, the optimal raw material-extractant ratio is 1:20, for pasty homogenized samples of apple fruits - 1:10, for liquid samples of homogenized lettuce - 1: 5. Analysis of the xenobiotic distribution parameter in the octane / water system (log Pow), reference data on the dielectric constant and dipole moment of solvents allowed to determine the extractants capable of dissolving and removing xenobiotics from raw materials. It was found that a mixture of acetonitrile and methanol (4: 1) should be used to extract benzimidazole derivatives and anilinopyrimidine derivatives, bipyridylium derivatives are best extracted with methanolic trifluoroacetic acid (9.5: 0.5).

Quantitative analysis of the content of xenobiotics in extracts obtained from samples artificially enriched with xenobiotics was performed. Xenobiotics were most completely removed from samples of crop products containing trace amounts of fat. The most difficult process of sample preparation is the process of obtaining a plant extract from sunflower seeds. The content of xenobiotics in extracts obtained

from samples artificially enriched in analytes is influenced by the temperature at which the process is performed and the duration of extraction.

Conclusions. The methodology of preparation of samples of crop products (oilseeds, lettuce leaves, apple fruits) for research by methods of chromatographic control of xenobiotics of three chemical groups of pesticides: benzimidazole derivatives, anilinopyrimidine derivatives, bipyridylium derivatives is developed in the work. The methodology includes the processes: homogenization of the sample, obtaining a plant extract, purification of the extract by solid-phase or liquid-liquid extraction, obtaining an extract of analytes. Based on the chemical composition of the sample matrix and the list of analytes, the optimal conditions of the variable component of the methodology are proposed, namely: obtaining plant extract under the action of selective solvents at homogenized raw material-solvent ratios from 1: 5 to 1:20 under conditions of constant mixing 200 rpm, or under the action of ultrasonic vibrations with a frequency of 37 kHz at a temperature of 4 ° C to 25 ° C for 5-25 minutes. The control of the qualitative and quantitative composition of plant extracts and analyte extracts was investigated by the methods of high-performance liquid and gas chromatography with mass-selective detectors.