Pesticide Residue Analysis for Fruits and Vegetables

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ABSTRACT

The analysis of pesticide residues in food is crucial for ensuring food safety and protecting public health. This process involves detecting trace amounts of pesticides and their breakdown products, often in concentrations as low as ng/mL. Techniques such as the QuEChERS method are employed, which effectively extract residues from various food matrices like fruits and vegetables. After extraction and clean up steps, compounds are identified using advanced analytical instruments like LC-MS/MS. Adherence to validated methods and rigorous quality assurance protocols is essential to minimize deviations and ensure accuracy. By understanding these processes, analysts can contribute to mitigating health risks associated with pesticide exposure through informed agricultural practices and consumer awareness measures.

INTRODUCTION

Analysing pesticide residues involves identifying not just the main chemicals but also their by-products and breakdown substances. It is challenging to detect residues in very low concentrations, like less than a ng m/L, because it requires isolating, precisely identifying, and measuring such tiny amounts amid a lot of other substances. A trained chemist will know most of the procedures used in residue analysis. However, analysing concentrations ranging from gm/kg to mg/kg can be challenging,

requiring careful attention to detail. The lead analyst must have the right qualifications, skills, and expertise in residue analysis. Before analysing samples, they should follow validated methods and understand pesticide residue analysis concepts and requirements of Analytical Quality Assurance (AQA) systems. They must grasp the purpose of each step in the process and adhere strictly to the procedures to minimize any potential deviations.

TERMS RELATED PESTICIDE RESIDUE ANALYSIS

Pesticide: The substance used to destroy or control the spread of pests in agricultural commodities or animal feed

Pesticide Residue: The amount of insecticide left over after a lapse of time. It is measured in ppm.

Deposit: The amount of initially laid down insecticidal chemical on the surface.

Half-life: The time in which half of the amount of initial deposit is eliminated.

Hazard: Probability of being harmed due to use/exposure/handling of toxic substances.

Risk: The degree of physical, biochemical and histological changes acceptable in terms of usefulness of a chemical and its possible effects on public health.

Toxicity: Ability of a chemical to bring about changes in the biological system of the target animal.

Acute toxicity: Toxic effect produced by application of single dose of toxicant.

Chronic toxicity: Toxic effect produced by accumulation of small amounts of toxicants over a long period of time. Ex: Teratogenic and Mutagenic effects.

Safe waiting period: The period for which the pesticides continue to remain toxic.

Acceptable daily intake (ADI): Is the daily dose of a chemical which when given during an entire life time appears to be without appreciable risk on the basis of all facts known at that time.

Maximum Residue Limit (MRL): The concentration of a toxicant residue in or on the food when first offered for consumption.

Maximum Residual Limit (MRL) = Acceptable Daily intake (ADI)/day/man ÷ Contaminated food in mg.

QUECHERS METHOD: Quick Easy Cheap Effective Rugged and Safe

- ✓ The QuEChERS-method was developed by Michelangelo in the year 2001 for the analysis of veterinary drugs in animal tissues.
- ✓ After realizing its great potential in the extraction of polar and particularly basic compounds it was also tested on pesticide residue analysis in plant material with great success.
- ✓ Now QuEChERS is known as a multiclass, multiresidue method (MRM) for analysis of pesticides from high water content (80-95%) matrices.
- ✓ The QuEChERS method now published as AOAC method 2007.01 “Determination of Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate”.
- ✓ The preferred solvent acetonitrile because it has been shown to provide extraction of the broadest range of organic compounds without co-extraction of large amounts of lipophilic material.

- ✓ This method is validated for the analysis pesticide residue in dry products like cereals, dried fruits, tea and fatty foods like fish, meat.

STEPS IN PESTICIDE RESIDUES:

The following are the steps for determining pesticide residues (Lehotay 2006)

Sampling: Sampling vegetables and fruits involves using equipment like choppers, mincers, and grinders, ensuring homogenization. For moist foods, chopping or mincing is typical; dry ice may be used for volatile compounds. Mechanical equipment generates heat, affecting sample integrity, especially with fatty foods. Analysts choose methods based on food type and need for homogeneity.

Extraction: Extraction is the method of using a solvent to remove pesticide residues from a matrix. It aims to achieve high efficiency without altering the pesticides chemically, using cost-effective and easy-to-clean equipment. Efficiency depends on the chosen extraction technique and solvent.

Cleanup: Cleanup in analysis involves removing interfering substances, like moisture, pigments (chlorophyll, xanthophylls, anthocyanins), oils, fats, and waxes, from extracts containing pesticide residues. These substances, called co-extractives, are extracted along with pesticides during the initial solvent extraction from the substrate matrix.

Analysis: The final step in analysis involves identifying and quantifying compounds using suitable instrumentation, chosen based on analyte properties. Pesticides vary widely in physicochemical properties, requiring different techniques such as capillary gas chromatography (GC) or high-performance liquid chromatography (HPLC in reversed-phase mode). GC is ideal for volatile, thermally stable pesticides, while GC-MS is preferred for multi-residue analysis in fruits and vegetables due to its efficient separation and sensitive detection capabilities. LC-MS offers rapid analysis for compounds challenging to detect with traditional methods.

FLOW CHART OF QUECHERS METHOD

Fruits/Vegetable samples (2 kg) were homogenized with robot coupe blixer

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15±0.1g sample was taken in a 50 ml centrifuge tube

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30±0.1 ml acetonitrile was added to the above centrifuge tube

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The sample was homogenized at 14000-15000 rpm for 2-3 min using a Heidolph silent crusher (low volume homogenizer)

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3±0.1g sodium chloride was added to the tube and mixed by shaking gently

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Centrifuged for 3 min at 3000 rpm to separate organic layer

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The top organic layer of about 16 ml was taken into the 50 ml centrifuge tube containing 9±0.1 g anhydrous sodium sulphate to remove the moisture content

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8 ml of extract was taken into a 15 ml tube containing 0.4±0.01g PSA sorbent (for dispersive solid phase d-SPE clean-up) and 1.2±0.01 g anhydrous magnesium sulphate

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The sample tube was vortexed for 30 sec followed by centrifugation for 5 min at 5000 rpm

↓

1 ml extract was filtered by using a PTFE filter, into 2ml vial and is taken for injection into the LC-MS/MS

CONCLUSION

Today's market often contains fruits and vegetables laden with harmful chemicals, posing risks to both human health and the environment. Select for organic produce whenever possible to avoid these chemicals. Wash all produce thoroughly before consumption with water or a 2% salt solution to remove surface chemicals. Trim and peel where feasible to eliminate up to 50-70% of chemical residues. Cooking further reduces residue levels. Farmers should use insecticides judiciously to minimize chemical use and its impact.

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