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QuEChERS METHOD: A MODERN TECHNIQUE FOR ANALYSIS OF PESTICIDE RESIDUES IN FOOD

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Abstract:

Farmers use pesticides to manage pests in crops as it helps to increase agricultural productivity, but rampant abuse of pesticides results in pesticide residues in some component of the environment after its application, spillage or dumping, which may pose significant risks to human beings, environment and survey has shown that pesticides affect the nutritional values of food as well, so there is a growing need to monitor these micropollutants to control the quality of food. A number of analytical methods are designed to determine multiple pesticide residues however, few if any of these methods can simultaneously achieve high quality results for a wide range of pesticides, QuEChERS method for pesticide residue analysis provided high quality results in a fast, easy, an inexpensive approach. The objective of this paper is to give an overview of the benefit of this method for greater confidence in the safety of food supply.

Keywords: *pesticides, pesticide residue analysis, food, QuEChERS method*

Introduction:

Pesticides are widely used in agriculture to increase the yield, improve the quality and extend the storage life of food crops (Fernndez *et al.,* 2008) However, excessive use of pesticides finds way as pesticide residues in the environment and food hence these must be used properly, respecting the law, the environment and human health, since they are toxic and can cause contamination (Sanches *et al.,* 2003). Pesticide residues are the deposits of pesticide active ingredient, its metabolites or breakdown products present in some component of the environment after its application, spillage or dumping. Consumer exposure to pesticide residues in food is an issue that is of considerable concern to consumers, food producers, scientists and government agencies. Regulatory bodies world wide have set the maximum residue levels (MRLs) that limits the types and amounts of residues that can be legally present on foods. The determination of the residues of pesticides

in food and environmental samples is very important due to the risks that these compounds offer to human health, besides their persistence in the environment and their tendency to bioaccumulate (Prestes *et al.,* 2009). According to a recent survey, 70% of consumers purchase organic foods to avoid pesticides (Whole Foods Market 2005) Worthington reviewed 41 studies and reported that organic crops contained 27% more vitamin C, 21.1% more iron, 29.3% more magnesium, and 13.6% more phosphorus than did conventional crops (Worthington 2001) so there has been growing environmental concern, especially regarding the use and discharge of toxic substances. Pesticide residue analysis has become tremendously an important process in determining the safety of using certain pesticides.

Advantages of QuEChERS method over traditional methods of residue analysis:

Residue analysis provides a measure of the nature and level of any chemical contamination within the environment and of its persistence. A number of analytical methods designed to determine multiple pesticide residues have been developed in the time since this type of analysis became important some 40 years ago (Food and Drug Administration, 1999; Luke et al., 1975; Specht and Tilkes, 1980; Lee et al., 1991; Andersson and Pålsheden, 1991; Cook et al., 1999; General Inspectorate for Health Protection, 1996; Fillion et al., 2000; Sheridan and Meola, 1999; Lehotay, 2000) However, few if any of these methods can simultaneously achieve high quality results for a wide range of pesticides and the practical benefits desired by all laboratories. In 2003, **Qu**ick, **E**asy, **Ch**eap, **E**ffective, **R**ugged, and **S**afe, the QuEChERS method for pesticide residue analysis was introduced by (Anastassiades *et al.,* 2003), which provided high quality results in a fast, easy, an inexpensive approach, effective, rugged, low solvent consumption, wide pesticide range (Polar, pH-dependent compounds) and safe. Follow-up studies have further validated the method for greater than 200 pesticides (Lehotay *et al.,*(submitted) improved results for the remaining few problematic analytes (Lehotay *et al.,*(submitted) and tested it in fat-containing matrices (Lehotay *et al.,*(submitted).

The QuEChERS method has several advantages over most traditional methods of analysis in the following ways:

1) High recoveries (>85%) are achieved for a wide polarity and volatility range of pesticides, including notoriously difficult analytes.

2) Very accurate (true and precise) results are achieved because an internal standard (I.S.) is used to correct for commodity to commodity water content differences and volume fluctuations. 3) High sample throughput of about 10-20 pre-weighed samples in ≈30-40 min is possible

4) Solvent usage and waste is very small, and no chlorinated solvents are used.

5) A single person can perform the method without much training or technical skill

6) Very little glassware is used.

7) Method, it is quite rugged because extract cleanup is done to remove organic acids.

8) Very little bench space is needed thus the method can be done in a small mobile laboratory if needed.

9) The MeCN is added by dispenser to an unbreakable vessel that is immediately sealed, thus solvent exposure to the worker is minimal.

10) The reagent costs in the method are very inexpensive.

QuEChERS method for Analysis of pesticide residues in food:

The QuEChERS method commonly uses GC–MS and LC–MS/MS to cover the wide range of pesticides for analysis (Cunha *et al.,* 2007). It has been extensively validated for hundreds of pesticide residues in many types of foods, and has become Association of Analytical Communities (AOAC) Official Method 2007.01 (Lehotay *et al.,* 2007)

QuEChERS methods:

- ***Original Unbuffered Method:*** Fast and Easy Multiresidue Method Employing Acetonitrile Extraction/Partitioning and "Dispersive Solid-Phase Extraction" for the Determination of Pesticide Residues in Produce (Anastassiades *et al.,* 2003).

- ***European EN 15662 Method:*** 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 (EN 15662).
- ***Mini-Multiresidue Method:*** A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products (QuEChERS 2008).
- ***AOAC Official 2007.01 Method:*** Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate. (AOAC Official Method)

One can choose QuEChERS method based on the commodity and the compounds of interest.

General Procedure (common to all QuEChERS-based methods listed above)

Step 1: Sample preparation and extraction

Commodities are uniformly ground. Internal standards are also added at this point. Various salts, acids, and buffers may then be added to enhance extraction efficiency and protect sensitive analytes.

Step 2: Sample extract cleanup

A subsample of the modified solvent extract from Step 1 is cleaned up using dSPE. Small polypropylene centrifuge tubes are pre-filled with precise weights and proportions of bulk drying salts and SPE sorbent packings to remove excess water and unwanted contaminants from the sample extracts. After a brief agitation and centrifugation, the cleaned extracts are then prepared for analysis.

Step 3: Sample analysis

Samples may be pH adjusted or solvent-exchanged prior to analysis by either GC-MS or LC-MS.

Conclusions:

The QuEChERS method has several advantages over most traditional methods of analysis. High recoveries (greater than 85%) are achieved for a wide polarity and volatility range of pesticides, including notoriously difficult analytes. Very rugged because extract clean up is done to remove organic acids. The introduction of QuEChERS method has enabled us to gradually

expand the spectrum of targeted pesticides both, directly (broader analyte spectrum covered) and indirectly (more personnel available to operate novel GC & LC instruments) and contributed to the reduction of the large "grey area" of pesticides for which no residue data existed in the past. This fact is reflected by the increasing number of different pesticides tested in fruit and vegetables and greater confidence in the safety of food supply.

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