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A quick method for surveillance of 59 pesticide residues in fruits and vegetables using rapid three-dimensional gas chromatography (GC/MSD/μ-ECD/FPD) and LC/MS-MS

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ABSTRACT

This article describes a simple, quick and inexpensive method for determination of pesticides in fruits and vegetables. The method, known as the quick, easy, cheap, effective, rugged and safe (QuEChERS) method for pesticide residues, involves the extraction and simultaneous liquid-liquid partitioning formed by adding anhydrous magnesium sulfate ($MgSO_4$) plus sodium acetate (NaAc) followed by a simple cleanup step known as dispersive solid-phase extraction (dSPE). The extracts were analyzed by three-dimensional gas chromatography GC/MSD/μ-ECD/FPD in trace ion mode and liquid chromatography/tandem mass spectrometry (LC/MS-MS). Method sensitivity, linearity, repeatability and reproducibility, accuracy, matrix effects, and overall uncertainties have been studied for method validation according to the international norm ISO/IEC: 17025:2005 for both techniques. Identification, quantification and reporting with Total and Extracted ion chromatograms, μECD and DFPD were facilitated to a great extent by Deconvolution Reporting Software (DRS) for GC and Mass hunter software for LC. For all compounds LODs were 0.001 to 0.01 mg/kg and LOQs were 0.005 to 0.020 mg/kg. Correlation coefficients of the calibration curves were >0.991. To validate the effects of matrices, repeatability, reproducibility, recovery and overall uncertainty were calculated for twenty-four matrices at 0.020, 0.050 and 0.500 mg/kg. Recovery ranged between 75-107 % with RSD <17 % for repeatability and intermediate precision and UM of ± 13-22 %.

Supplementary Materials

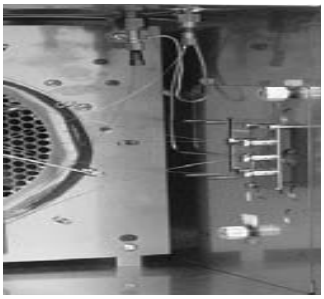

Figure S1. Configuration feature of 3-ways splitter.

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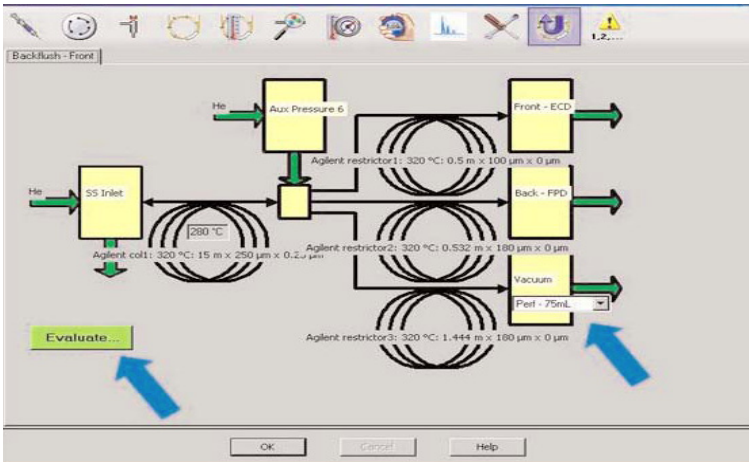

Figure S2. Configuration of back flush.

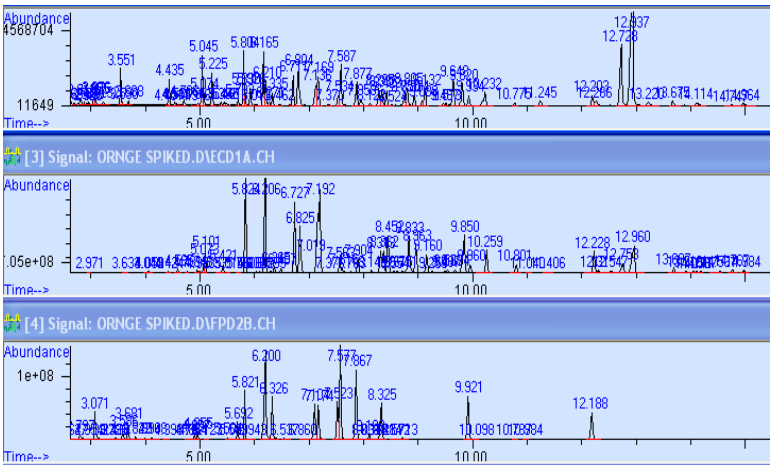

Figure S3. Chromatogram for multi signal configuration: Total ion chromatogram (GCMSD) along with DFPD (phosphorus or sulphur mode) and µECD data of orange spiked sample.

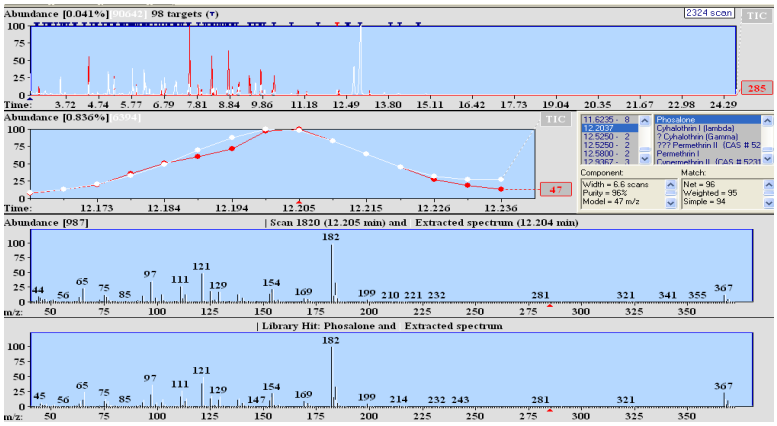

Figure S4. The screener software window of AMDIS for positive detection, identification and quantification of phosalone.