

ADVANCES ON PESTICIDE RESIDUE ANALYSIS USING CHROMATOGRAPHY-MASS SPECTROMETRY IN PERU

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Abstract:

The agricultural export is one of the most important economical activities in constant growth in Peru that produce economical incomes and employments. Amount exported in 2008 was US\$ 2,597 millions representing an increase of 31.7% compared with 2007; the main commodities exported were asparagus, sweet pepper, citric fruit and coffee; and the most important destiny countries were United States (31%), Spain (12%) and Netherlands (10%) [1]. However; it has been attenuated by rejections mainly due to presence of pesticide residues at levels greater than maximal residues limit (MRL) laying down by the national or international food safety authorities. During 2008, 13% of rejection of Peruvian shipment in USA was due to pesticide residues [2]. For this reason, the Peruvian National Agricultural Health Service-SENASA implemented in 2006 the laboratory Toxic Residues and Agricultural Product Control Center for official analysis of pesticide residue in food. The aim of this study was to verify the performance of a multi-residue pesticide analytical method in sweet pepper, asparagus, grape and oranges using QuEChERS method [3] and detection by Liquid Chromatography coupled to Tandem-Mass Spectrometry (LC-MS/MS) and Gas Chromatography coupled to Mass Spectrometry (GC-MS). A representative portion of the sample (15 g) was subjected to extraction with acetonitrile containing acetic acid, followed by the addition of salts (MgSO₄ and NaOAc), to facilitate the removal of a significant amount of polar components of the matrix and then perform dispersive solid-phase extraction with a mixture of magnesium sulfate, primary secondary amine (PSA) and graphitized carbon black (GCB). The performance parameters measured were recovery (%), precision (RSD%), linearity and LOD. The recovery and precision were assessed at levels of 10, 20 and 50 ug/kg for LC-MS/MS and at level of 100 ug/kg for GC-MS, and linearity with matrix-matched calibration in 5 levels, 5 to 250 ug/kg for LC-MS/MS and 20 to 1000 ug/kg for GC-MS. Preliminary results for the 150 pesticides assessed in this study ranging from 50 to 120% for recovery, 3.9 to 26% for precision (%RSD) and 2 to 60 ug/kg for LOD. The recoveries (%) and the precisions (%RSD) were found satisfactory for most pesticides in according with the criteria of Codex [4]. The range of the analytical curve was linear and the correlation coefficients were mostly >0.99. The chromatograms also showed good selectivity. The results show that the method QuEChERS is adequate for official control and could be used for the national program of monitoring pesticides residues.

[1] Evolución de las exportaciones peruanas Enero – Diciembre 2008.

http://www.siicex.gob.pe/siicex/portal5ES.asp?_page_=458.42200

[2] Import Refusal Reports for OASIS.

http://www.accessdata.fda.gov/scripts/ImportRefusals/ir_index.cfm

[3] Lehotay, S., 2007. Determination of Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate: Collaborative Study. Journal of AOAC International. 90:485-520.

[4] Guidelines on Good Laboratory Practice in Residue Analysis. CAC/GL 40-1993, rev.1-2003

