

ALTERNATIVE APPROACH TO MULTIRESIDUE DETERMINATION OF PESTICIDES IN FOOD BY GC-MS

Osmar D. Prestes, Caroline A. Friggi, Gisele L. Martins,
Martha B. Adaime, Renato Zanella*

Laboratório de Análises de Resíduos de Pesticidas (LARP), Universidade Federal de Santa Maria (UFSM), 97105-900 Santa Maria-RS, Brasil

*renatozanella@pq.cnpq.br

Many benefits have been achieved from the use of synthetic pesticides in agriculture, but in spite of the obvious advantages, the potential adverse impact on food consumers health must be considered. Monitoring programs have therefore been established to control pesticide residues in food of plant origin in order to ensure compliance with national and international law, and to reassure consumers that food crops are healthy. However, the great number of compounds registered for use on a diversity of plant cultivars makes pesticide residue analysis an on-going challenge for scientists [1].

In 2003, a miniaturized method, QuEChERS (quick, easy, cheap, effective, rugged and safe), has become an attractive alternative to classical sample preparation methods. The method is based on a single-step acetonitrile extraction and salting out by liquid-liquid partitioning from the water in the sample with MgSO₄ followed by a dispersive solid-phase extraction (D-SPE) clean-up. QuEChERS method was established for the determination of pesticide residues in fruits and vegetables by gas chromatography/mass spectrometry (GC/MS) and liquid chromatography/tandem mass spectrometry (LC/MS/MS) [2].

We investigated the suitability of the modified QuEChERS method for tomato, potato and milk. For the extraction step acetonitrile containing 1% acetic acid, sodium acetate, and magnesium sulphate were used. The cleanup using D-SPE extraction was carried out with C₁₈ and magnesium sulphate. For the milk samples we combined D-SPE with low temperature clean-up, because the main difficulty for the determination of pesticides in fatty matrices.

The method was validated considering the following parameters: linearity, limit of detection (LOD), limit of quantification (LOQ), matrix effect as well as precision and accuracy, evaluating the recovery at three different spike levels. The linear range used in the analytical curves was from 1.0 to 200 µg L⁻¹, with values of $r^2 > 0.99$. Recoveries showed values between 75 and 120% with RSD < 20%.

The developed and validated method has many advantages including speed and high sensitivity, which can be applied beneficially to the determination of multi-component pesticides contaminants in tomato, potato and milk.

[1] Walorczik, S. 2008. Application of gas chromatography/tandem quadrupole mass spectrometry to the multi-residue analysis of pesticides in green leafy vegetables, *Rapid Commun. Mass Spectrom.* 22: 3791-3801.

[2] Prestes, O. D., Friggi, C. A., Adaime, M. B., Zanella, R. 2009. QuEChERS – um método moderno de preparo de amostra para determinação multirresíduo de pesticidas em alimentos por métodos cromatográficos acoplados à espectrometria de massas, *Química Nova*, 32: 1620-1634.

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