

Simultaneous determination of 200 pesticide residues in honey using gas chromatography-tandem mass spectrometry in conjunction with streamlined quantification approach

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Abstract

A sensitive, accurate and reliable multi-class GC-MS/MS assay protocol for quantification and confirmation of 200 common agricultural pesticides in honey was developed and validated according to EU guidelines. A modified extraction procedure, based on QuEChERS method (quick, easy, cheap, effective, rugged and safe) was employed. Mass spectrophotometric conditions were individually optimized for each analyte to achieve maximum sensitivity and selectivity in MRM mode. The use of at least two reactions for each compound allowed simultaneous identification and quantification in a single run. The pesticides under investigation were separated in less than 31 min using the ultra-inert capillary column (DB-35MS). For all analytes, neat standard calibration curves in conjunction with correction for matrix effect were successfully employed. The detection limits of the assay ranged from 1.00 to 3.00 ng mL⁻¹ for the studied pesticides. The developed assay was linear over concentration range of 10.00–500.00 ng mL⁻¹, with correlation coefficient of more than 0.996. At the LOQ, 81% of the studied pesticides were efficiently recovered in the range of 70.00–120.00%, with CV% less than 15.00% while 99.3% compounds had mean percentage recovery of 60.00–140.00%, with CV% less than 21.00% (N = 18, over three different days). The proposed assay was successfully applied for the analysis of the studied pesticide residues in one PT sample and 64 commercial honey samples collected over 1 year from different districts around Egypt. Results revealed that only one honey sample out of the 64 analyzed samples was contaminated with tau-Fluvalinate (10.00 g kg⁻¹). This wide scope assay protocol is applicable for monitoring pesticide residues in honey by national regulatory authorities and accredited labs; that should help ensure safety of such widely used product.

Journal of Chromatography A 2015, November