

COMPARISON OF ULTRA PERFORMANCE LIQUID CHROMATOGRAPHY AND HIGH PERFORMANCE LIQUID CHROMATOGRAPHY FOR THE DETERMINATION OF PRIORITY PESTICIDES IN BABY FOODS BY TANDEM QUADRUPOLE MASS SPECTROMETRY

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The European Union Baby Food Directive 2003/13/EC [1] designates pesticides as prohibited, in which case they are considered not to have been used if their residue does not exceed 0.003 mg kg^{-1} , or sets maximum residue limits (MRLs) between 0.004 and 0.008 mg kg^{-1} . This regulation has been difficult to enforce because existing multi-residue methods do not have sufficient limits of detection for these priority pesticides. Although many pesticides can be analysed readily by GC-MS/MS, others are more suited to LC-MS analysis. UPLC [2], using $1.7 \text{ }\mu\text{m}$ particles, has the potential to provide shorter run times and better chromatographic resolution than established HPLC methods.

A sample extraction and preparation method has been developed for LC-MS/MS analysis of seventeen of the priority pesticides and transformation products (e.g. metabolites) specified in baby food. Prior to LC-MS/MS analysis, co-extractives were removed from acetonitrile extracts using dispersive solid phase extraction with primary secondary amine (50 mg) sorbent.

Analysis by HPLC-MS/MS and UPLC-MS/MS gave linear calibrations over the concentration range 0.0005 to $0.01 \text{ }\mu\text{g ml}^{-1}$ (equivalent to $0.0005 - 0.01 \text{ mg kg}^{-1}$) for all seventeen pesticides. Extracts spiked with pesticides at $1 \text{ }\mu\text{g kg}^{-1}$,

yielded average recoveries in the range 85 - 119% with relative standard deviations less than 17%. The method employs electrospray ionisation in the positive mode, and several acquisition parameters including selection of MS/MS transitions, collision energy and cone voltage were optimized for best response. Appropriate selection of the cone voltage was critical to obtaining a sufficient response for two of the pesticides, disulfoton and terbufos.

The HPLC-MS/MS and UPLC-MS/MS multi-residue methods developed are simple, rapid and suitable for the screening of 17 priority pesticides in fruit-, potato- and cereal-based baby food at 1 $\mu\text{g kg}^{-1}$, and for quantification and confirmation at their respective MRLs. The major advantage of UPLC over HPLC is the speed of analysis and better peak shape for the targeted pesticides in the analyses of baby foods.

References

1. EC, 2003, Commission Directive 2003/13/EC of 10 February 2003 concerning cereal-based foods and baby foods for infants and young children, *Official Journal*, **L41/33**.
2. M.E. Swartz, *LC GC Europe*, June 2005, 5-11.