

Accelerated LC/MS/MS for the Quantitation and Confirmation of Pesticides in Food and Water Samples

Recent regulations on food and environmental analysis require the screening for pesticides using confirmatory techniques, such as GC/MS and LC/MS/MS. With more than 1000 pesticides of more than 100 compound classes and their metabolites and degradation products being in use or present in the environment there is a demand for powerful and rapid analytical methods, which can detect very low concentrations of pesticides. Here we present a high-throughput LC/MS/MS method that combines the high sensitivity MRM screening with fast EPI confirmation and allows detection of low concentration pesticides, with LOD below 0.1 µg/L and direct injection of water samples.

The setup consisted of a reversed phase LC system using a polar modified column (Phenomenex Synergi-Fusion), a gradient of water/methanol with 5mM ammonium formate buffer, and a hybrid triple quadrupole linear ion trap mass spectrometer with Electrospray Ionization (ESI). Multiple Reaction Monitoring (MRM) and Enhanced Product Ion (EPI) scan were used to detect pesticides. This set-up allows the simultaneous screening, quantitation and confirmation of hundreds of pesticides in food and water samples with minimal or no sample preparation. Often further dilution of extracts is used to avoid matrix effects during the ionization process.

The developed LC/MS/MS method was used to monitor pesticides in various fruit and vegetable samples. Additionally, water samples were injected directly into LC/MS/MS and pesticides could be detected at concentration levels of 0.1 µg/L or below.

In one single experiment up to 600 MRM transitions were monitored without cross talk using traditional MRM and scheduled MRM (sMRM) functionality. The use of sMRM greatly enhances accuracy and reproducibility of LC/MS/MS detection of a large set of targeted analytes at low concentration levels. In the same experiment fast confirmatory EPI scans were triggered by intelligent software using Collision Energy Spread (CES) settings. In comparison to fixed Collision Energies CES was found to give more reproducible and richer MS/MS spectra and thus greatly enhancing the quality of library searching. Library searchable EPI spectra were generated at concentrations as low as 0.1 µg/L.

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