

Multi-Residue Pesticide Screening for Dried Botanicals

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GOAL

To demonstrate the performance and flexibility of the Xevo® TQ-S micro MS in a multi-residue pesticide screening analysis for botanicals.

BACKGROUND

Pesticides are widely used in farming, gardening, or public health management to prevent damage from pests, fungi, weeds, or disease transmission by insect bites. The application of pesticides is strictly regulated, and their residues in food and the environment are closely monitored. There are many multi-residue pesticide screening methods available for foods and vegetables, such as the AOAC Official Method 2007.01. However, there are limitations to these methods when applied to dried botanicals, especially resinous hemp-like dried botanicals, due to their complex sample matrix. More intensive sample cleanup may be needed during sample preparation for multi-residue pesticide screening in dried botanicals.

Tandem mass spectrometry (MS/MS), such as the triple quadrupole MS, is the detector of choice in the multi-residue pesticide screening analysis. This is because its high detection selectivity and sensitivity allows for the screening of hundreds of pesticides at low concentration levels (ppb) in a single analysis. Both LC-MS/MS and GC-MS/MS are commonly used for multi-residue pesticide screening since some pesticides are only amenable to either LC or GC. Two MS systems are required, one for the LC and one for the GC, which is a significant redundant investment for pesticide analysis labs.

Enables multi-residue pesticide analyses for both LC and GC amenable compounds on a single MS system.

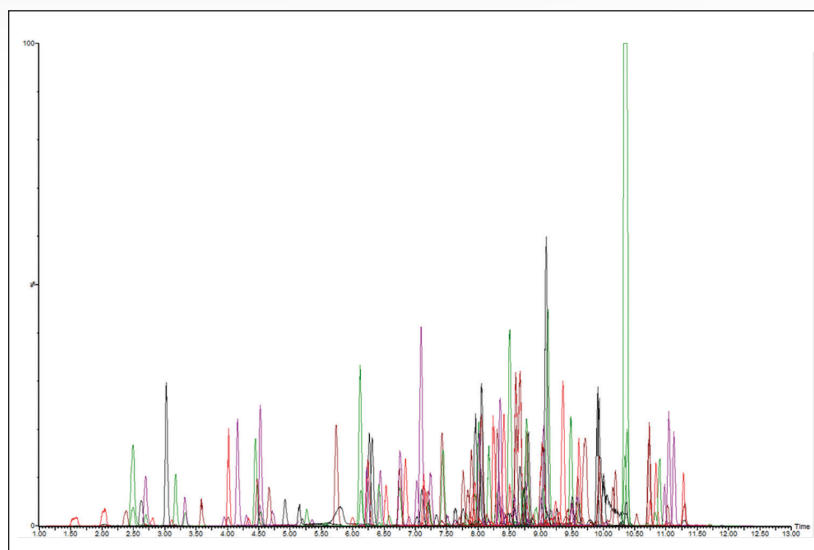


Figure 1. LC-MS/MS chromatograms overlay of 204 pesticides post-spiked in dried botanical extract. **System:** Xevo TQ-S micro ESI (positive and negative modes) coupled with the ACQUITY UPLC I-Class System; **Sample:** Dried botanical (hemp-like) extract spiked with the LC Multi-Residue Pesticide Standards Kit at 40 ppb level; **Column:** ACQUITY UPLC BEH C₁₈, 1.7 μm (2.1 x 100 mm); **Temp.:** 50 °C; **Mobile phase:** A: Water with 10 mM ammonium acetate (pH adjust to 5.0), B: Methanol with 10 mM ammonium acetate; **Flow rate:** 0.45 mL/min; **Gradient (%B):** Initial isocratic at 2% B for 0.25 min, then linear gradient to 99% B in 11.75 min, keep isocratic at 99% B for 1.50 min, then return to the initial condition of 2% B in 0.1 min. **Run time:** 15 min.

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THE SOLUTION

Waters® Xevo TQ-S micro tandem quadrupole mass spectrometer, equipped with the ACQUITY UPLC® I-Class System and Waters Atmospheric Pressure Gas Chromatography (APGC) provides a cost-effective solution for the multi-residue pesticide analysis. One Xevo TQ-S micro MS can be coupled to the UPLC® System or the APGC source, and analyze the LC amenable and the GC amenable compounds separately. The switch between the LC and the GC inlets is quick and easy, no more than a few minutes, since the MS system does not need to be vented.

In this technology brief, the ACQUITY UPLC I-Class System and the APGC source were coupled to the Xevo TQ-S micro separately, and the LC pesticides and the GC pesticides that were post-spiked in botanical extracts were analyzed. Waters' Quanpedia™ Database was used to automatically create the MS methods and the data processing methods for the hundreds of MRM transitions. The LC inlet method can also be automatically created by Quanpedia.

A modified QuEChERS method was used in this study for sample extraction. Basically, 2 grams of sample were extracted using acetonitrile in the presence of water and salt (DisQuE™ Quechers Pouch, Part no. [186006813](#)); then the extract was split into two portions. One was further cleaned using dispersive SPE which contained MgSO₄, PSA, C₁₈, and GCB (QuEChERS Cleanup Tube, Part no. [186008079](#)) and used for the LC-MS analysis. The other portion was further cleaned for the APGC-MS analysis.

Figure 1 shows an overlay of the 204 pesticide UPLC-MS/MS chromatograms. The pesticides (LC Multi-residue Pesticides Standards Kit, Part no. [186007574](#)) were post-spiked at 40 ppb level in the botanical extract. A series of botanical extract post-spiked at various levels (from 0.04 ppb to 40 ppb) were prepared and analyzed using UPLC-MS/MS. The limit of quantitation (LOQ) for these 204 pesticides was estimated at a signal-to-noise (S/N) level of 10. Not all pesticides can be detected at the same level, therefore the distribution of the LOQ values is shown in Figure 2. Nearly all the 204 pesticides (93%) have LOQ values of less than or equal to 10 ppb.

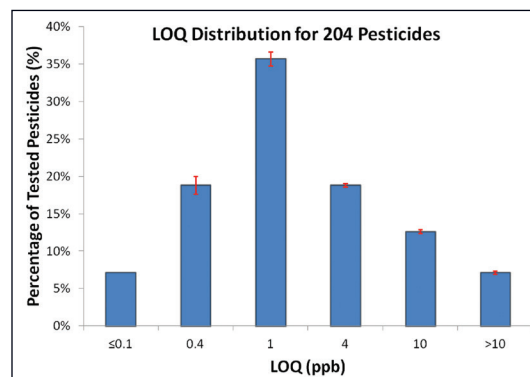


Figure 2. Distribution of the LOQ of 204 pesticides in specified LOQ range. The LOQs were obtained from UPLC-MS/MS analysis of post-spiked botanical extracts. Pesticides were spiked at levels from 0.04 ppb to 40 ppb. Experimental conditions are the same as those in Figure 1. Nearly all the pesticides (93%) have an LOQ of 10 ppb or less. (Data was an average of two repeat experiments.)

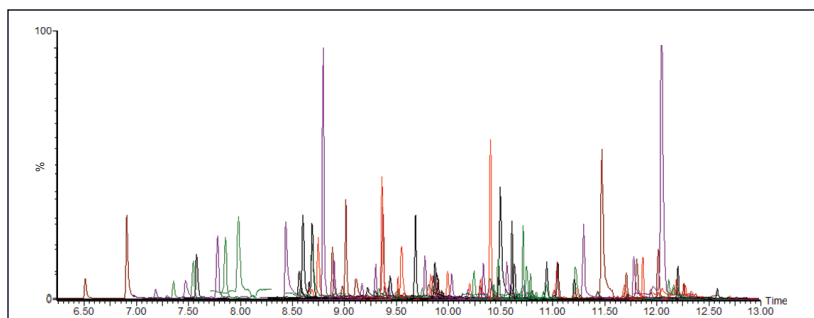


Figure 3. APGC-MS Chromatograms overlay of 135 pesticides post-spiked in dried botanical extract at 20 ppb. (System: Waters Xevo TQ-S micro coupled with APGC system; Sample: Dried botanical (hemp-like) extract spiked with GC multi-residue pesticide standards kit at 20 ppb level; Column: DB5 30 m x 0.25 mm x 0.25 μ m; Carrier gas: He; Temp. program: 35 °C for 1.20 min/ramp 25 °C/min to 320 °C hold for 3 min; Injector: MMI)

Figure 3 shows an overlay of the APGC-MS chromatograms of 135 pesticides, which were post-spiked into the GC portion of the botanical extract at a 20 ppb level.

SUMMARY

The Xevo TQ-S micro coupled to the ACQUITY UPLC I-Class System and APGC provides adequate sensitivity in multi-residue pesticide analyses, including LC and GC amenable compounds, for challenging resinous dried botanical extracts. The APGC capability of Xevo TQ-S micro provides additional cost effective benefits for the multi-residue pesticide screening.

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