

Determination of Imidacloprid Residues in Bee Pollen Using Xevo TQ-S

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GOAL

In this technology brief, the ability of Waters® Xevo® TQ-S coupled with the ACQUITY UPLC® I-Class System to detect trace levels of imidacloprid in bee pollen is demonstrated.

BACKGROUND

Analysis of complex environmental samples for trace levels of contaminants presents a challenge in achieving the necessary sensitivity of the instrument employed to detect sub-ng/g levels through often intense matrix effects. Monitoring honey bee populations for lethal and sub-lethal effects due to pesticide exposure requires reliably quantifying levels of pesticides in both bees and bee products. A local case in which imidacloprid pesticide was used to treat an invasive insect species prompted the monitoring of area honey bee populations. Honey bee samples from this area and their product, bee pollen, were subjected to analysis of imidacloprid residues. Both sample types are highly complex, with bee pollen containing on average 20% protein and 6% total lipid content,¹ as well as a plethora of vitamins, fatty acids, enzymes, and pigments. In the following work, a specialized extraction technique utilizing Waters' Sep-Pak® C₁₈ Cartridges to remove numerous matrix interferences and the highly sensitive Xevo TQ-S for reliable detection of imidacloprid in the pg/g range was employed.

Trace levels of imidacloprid pesticide were detected in complex bee pollen matrix using the ultra-sensitive ACQUITY UPLC with Xevo TQ-S.



Figure 1. ACQUITY UPLC with Xevo TQ-S.

THE SOLUTION

300 mg of bee pollen samples were homogenized in a mortar and pestle, then diluted 1:10 in Milli-Q water. 12 mL of 2% triethylamine in acetonitrile was added to the pollen slurry, vortex mixed, and then added to a QuEChERS AOAC tube 1 (6 mg MgSO₄ and 1.5 g sodium acetate). Upon centrifugation, the organic layer was removed and diluted to 15% water content and passed through a Sep-Pak C₁₈ 1-cc Cartridge under vacuum. The resulting extract was dried down to near dryness under a gentle stream of nitrogen, and reconstituted in four times the original volume in water.

The samples were then analyzed on an ACQUITY UPLC I-Class System, coupled to Xevo TQ-S. The MRM transition 256.2 → 175.2 was used for quantification, and 256.2 → 209.2 for confirmation. Extraction recovery and matrix suppression were assessed using store-bought organic bee pollen nutritional supplement spiked with 1 ng/g imidacloprid standard, and those results are displayed in Table 1. Both matrix-matched and solvent calibration curves were used for quantification of samples and recovery tests, respectively, and had an $r^2 > 0.99$. Method LOD and LOQ were determined by matrix-matched standards that exceeded a peak-to-peak signal-to-noise (S/N) of 3 and 10, respectively. Well conserved ion ratios were used as an additional criterion for identification.

Of the samples from the exposure area tested, imidacloprid residues were detected in three bee pollen samples, though below the LOQ in matrix, with the exception of one sample that contained 29.3 ng/g. MRMs of the aforementioned sample, one below the LOQ, and a matrix-matched 100 pg/g standard, which surpassed the peak-to-peak S/N ratio of 10, and was considered the method LOQ, as shown in Figure 2 a, b and c.

	1 ng/g spike	% RSD	Samples
Extraction recovery	95.57%	9.89	3
Matrix suppression	-62.51%	4.29	3

Table 1. Sample extraction and suppression results for imidacloprid in bee pollen. Despite intense matrix suppression effect, the sensitivity afforded by the Xevo TQ-S made it possible to detect and quantify imidacloprid present in matrix at the pg/g level.

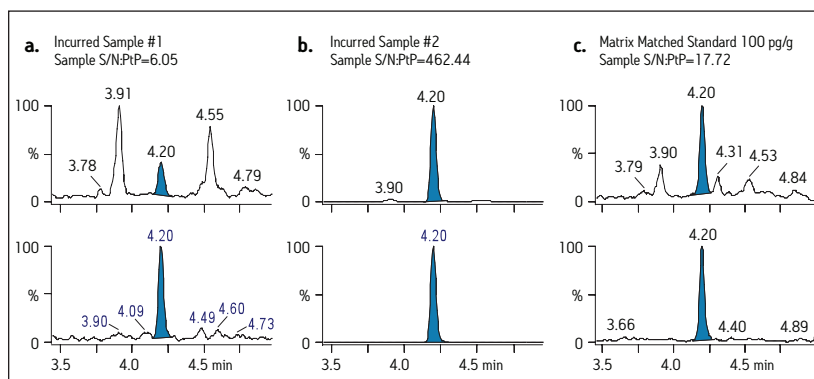


Figure 2. MRM chromatograms of quantification and confirmatory transitions for imidacloprid in bee pollen samples; a. sample containing imidacloprid below LOQ, b. above LOQ, c. matrix matched 100 pg/g standard.

SUMMARY

The detection of imidacloprid bee pollen was performed using the highly sensitive and robust Xevo TQ-S, which proved to be a powerful tool in solving this unique environmental monitoring challenge.

Reference

1. Muradian-Alameida LB, et al. Chemical composition and botanical evaluation of dried bee pollen pellets. JFCA. 2005;18: 105-111, 2005.

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