Implementation of Waters Pesticide Screening Application Solution for the Detection of an Incurred Residue in Orange Juice

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This is an Application Brief and does not contain a detailed Experimental section.

Abstract

This application brief demonstrates the efficacy of high resolution separations and accurate mass QTof MS for screening QuEChERS extracts of orange juice samples for pesticides at or below regulatory limits in juice samples.

Benefits

Pesticide screening using the ACQUITY UPLC I-Class System/Xevo G2-S QTof provides a comprehensive approach to identify and quantify incurred pesticide residues in juice samples.

Introduction

The accurate detection of pesticides is crucial to ensure the safety of food products. The detection of carbendazim (a fungicide banned from use on oranges in the U.S.) in orange juice samples in 2012 provides evidence of the requirement for rapid screening of food and beverage samples. In addition, the sensitivity required must be below the U.S. FDA action limit of 10 µg/kg for carbendazim. Waters Pesticide Screening Application Solution employing Xevo G2-S QTof in combination with the ACQUITY UPLC I-Class and the UNIFI Scientific Information System is designed for rapid screening of food commodities.

The excellent chromatographic resolution offered by the ACQUITY UPLC I-Class System ensures the best separation of matrix components from analytes of interest. The Xevo G2-S QTof delivers stable mass accuracy, excellent in-spectrum linear dynamic range, MS^E capability, and isotopic fit. These capabilities ensure accurate and reliable routine residue detection with both precursor and fragment ions acquired from a single injection.
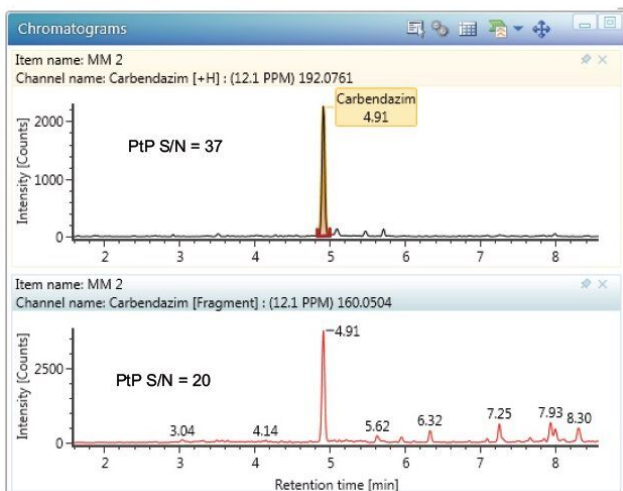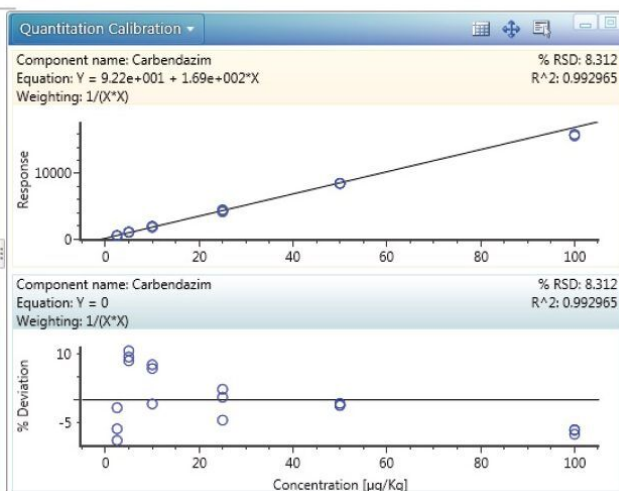
Figure 1. (A) XIC of carbendazim precursor (top) and fragment (bottom) ion for 5 mg/kg standard in matrix (which is half the 10 mg/kg FDA action limit for carbendazim). (B) Matrix matched calibration curve and residuals plot for carbendazim in orange juice showing good linearity with an r^2 value > 0.99.

In combination with the new UNIFI scientific information system, confident identification and quantification of pesticide residues is made possible. In this technology brief, we demonstrate the screening capabilities of this application solution for the detection of an incurred residue of carbendazim in orange juice.

Results and Discussion

Six different orange juice samples were subjected to QuEChERS extraction using Waters DisQuE Dispersive SPE. The resulting organic extracts were dried down and reconstituted in water. Six matrix matched calibrants ranging from 2.5 to 100.0 µg/kg containing 57 pesticides were prepared from a known blank juice sample (previously analyzed and found to be free from these pesticides). The standards and samples were analyzed using the method and column included with Waters Pesticide Screening Application Solution. This methodology utilizes an ACQUITY UPLC BEH C_{18} 2.1 x 100 mm Column with a water/methanol/10 mM ammonium acetate gradient. Precursor and fragment ions were acquired in a single injection using MS^E functionality. The resulting data were processed against compounds from Waters' Screening Library, which contains over 2000 compounds.

The extracted ion chromatograms for the precursor ion (upper) and fragment ion (lower) of the matrix

matched calibration standard at 5 µg/kg are shown in Figure 1A. The high signal-to-noise ratio at half the action limit, shown in Figure 1A, enabled accurate quantification of carbendazim at low levels in matrix. The matrix matched calibration curve for carbendazim is shown in Figure 1B. Of the six orange juice samples tested, carbendazim was detected with fragment ion confirmation in one sample. A screen shot of the results from the detection of this residue is shown in Figure 2. The extracted ion chromatograms for precursor and fragment ions for the sample are shown in Figures 2A and 2B. The low- and high-energy spectra are shown in Figures 2C and 2D, respectively. The precursor ion and fragment ions show excellent mass accuracy with 0.4 and 0.3 ppm mass errors, respectively.
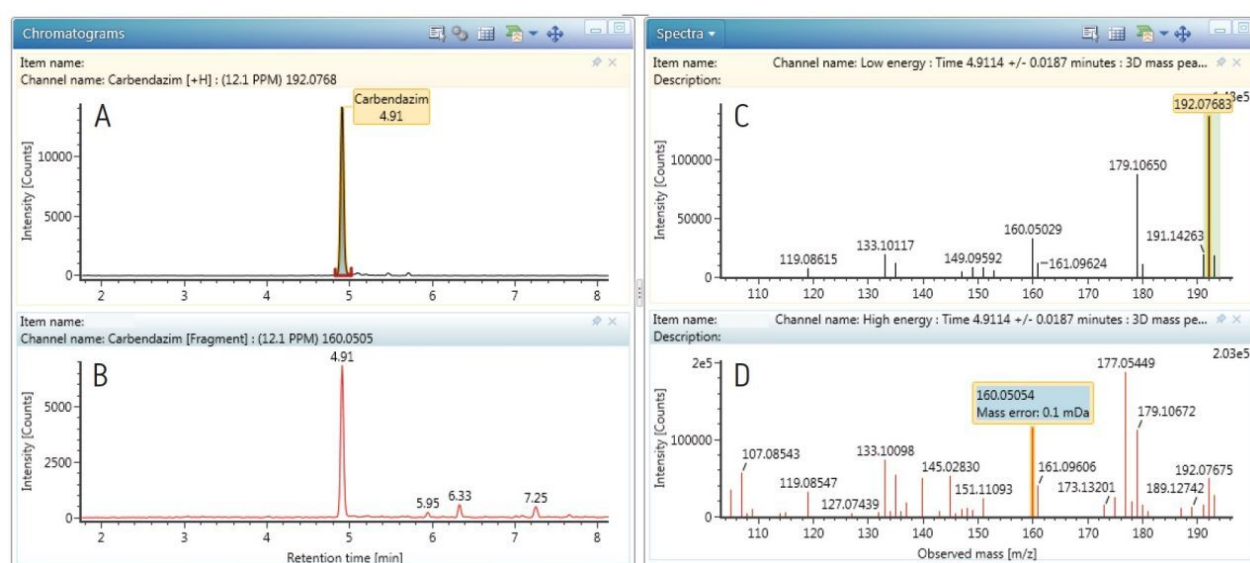

Figure 2. The positive identification of carbendazim in an orange juice sample at 18.2 mg/kg following QuEChERS extraction. The precursor and fragment ions extracted chromatograms are shown in panels A and B, respectively. The low- and high-energy MS^E spectra for carbendazim are shown in panels C and D, respectively. The identified precursor and fragment ions of carbendazim are shown. Mass accuracy for both the precursor and fragment ion are below 1 ppm, ensuring accurate and reliable identification.

Conclusion

Waters Pesticide Screening Application Solution successfully identified and quantified carbendazim residues in orange juice extracts below the FDA action limit for orange juice imports. The application of a comprehensive database containing molecular formulae, retention time, and exact mass fragment ion information provides increased confidence in identifications and helps to reduce the number of false

detections.

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