[TECHNOLOGY BRIEF]

Direct Injection of Orange Juice for the Rapid Detection of the Fungicide Carbendazim Using ACQUITY UPLC I-Class with Xevo TQ-S

GOAL

To rapidly determine carbendazim residue in orange juice, with no sample preparation, at regulated levels by taking advantage of the ultra-high sensitivity of the Xevo® TQ-S Mass Spectrometer combined with the ACQUITY UPLC® I-Class System.

BACKGROUND

Recent news about the presence of the fungicide carbendazim in orange juice (OJ) products has drawn widespread public attention on the safety of the fruit juice. Carbendazim is legally used in Brazil and many other countries for plant disease control, post-harvest food storage or transportation, and seed pre-plant treatment. However, it is not licensed for use on citrus fruits in the U.S. and is not permitted to be present at any concentration in OJ. The U.S. FDA has since responded by holding and testing all shipments of incoming OJ products from all foreign sources as of January 13, 2012.

Pesticide residues in fruit juices have always been an important food safety issue, especially taking into account the high consumption of juice by children. Many published methods are capable of analyzing fungicides, including carbendazim, in fruit juices in the low ppb range. However, intensive sample preparation is required in order to remove possible interferences and to enrich the analytes.

Detection of carbendazim residues in orange juice at concentrations below 10 parts per billion (ppb) is possible without sample preparation or pre-concentration by employing a simple 'dilute-and-shoot' procedure.

A recent and promising approach that requires no sample preparation has been achieved with Waters® Xevo TQ-S tandem quadrupole, where a direct injection, or dilute-and-shoot approach is employed. This approach is possible because of the ultra-high sensitivity and specificity of the Xevo TQ-S MS coupled with the ACQUITY UPLC I-Class System. The benefits of this dilute-and-shoot approach include a significant increase in analysis throughput and a reduction in method development time.

In this technology brief, we describe a simple dilute-and-shoot method that employs a Xevo TQ-S coupled with the ACQUITY UPLC I-Class System for the rapid analysis of carbendazim in orange juice.

THE SOLUTION

OJ products were diluted 100 times with solvent, filtered with a 0.45-µm nylon membrane syringe filter, and injected directly onto an ACQUITY UPLC I-Class System, coupled with the Xevo TQ-S. Typical reversed-phase conditions were used for the UPLC® separations with mobile phases A (10 mM ammonium acetate) and B (10 mM ammonium acetate in methanol). Two multiple reaction monitoring (MRM) transitions, 192.1 > 160.1 and 192.1 > 132.1 were used for quantification and confirmation, respectively.

The U.S. EPA assessment of carbendazim indicates that it has no health risk at 80 ppb in OJ. The U.S. FDA is testing import juice samples at much lower levels and will not allow samples that contain 10 ppb or higher of carbendazim entry through the U.S. border. The ultra-high sensitivity of this system allows comfortable detection of carbendazim in OJ below 10 ppb, with no sample preparation employed. Figure 1 shows the two MRM transitions monitored for a carbendazim standard equivalent to 10 ppb, a retail sample of blank OJ, the same OJ fortified at 10 ppb, and an additional retail sample of OJ found to contain carbendazim at 30 ppb. While in this study carbendazim concentrations were calculated using external standardization alone, the use of a stable isotope-labeled internal standard for quantitation is recommended to control for potential matrix variability.

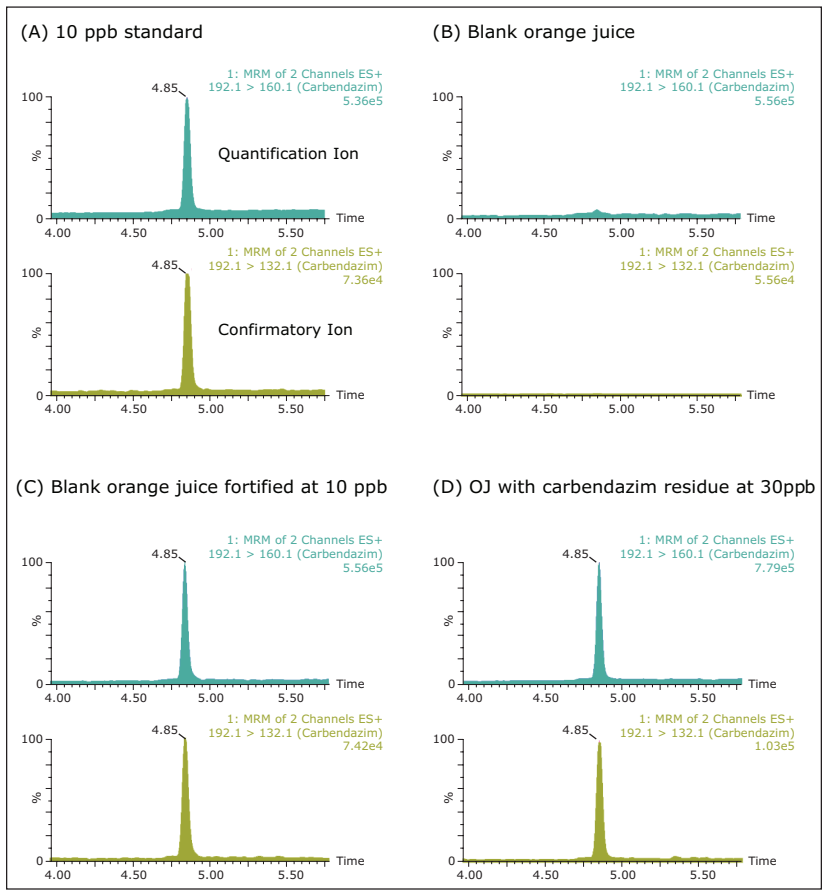

Figure 1. MRM transitions of (A) 10 ppb standard, (B) blank OJ, (C) the same OJ fortified at 10 ppb, and (D) an additional retail OJ sample with incurred carbendazim residue at 30 ppb.

SUMMARY

The ultra-high sensitivity of Xevo TQ-S Mass Spectrometer coupled with the ACQUITY UPLC I-Class System allows direct analysis of trace amounts of carbendazim in orange juice samples. The elimination of sample preparation not only results in high analysis throughput, but also simplifies the method development and optimization process, which is especially valuable to those who need to address versatile analytical requests in a short period of time.

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