Determination of Carbendazim in Orange Juice by QuEChERS and LC/MS/MS Detection

INTRODUCTION

Carbendazim is a broad-spectrum fungicide with a planar structure that is widely used to control fungi and molds in crops and fruits. It is permitted for use in the European Union (EU) and many other countries, but it is not registered for use in the United States. A beverage manufacturer reported the detection of carbendazim in its orange juice products in early 2016, motivating the UCT Pharma and the rejection limit of carbendazim at 0.15 kg in imported orange products.

Orange juice is a complex matrix that contains organic acids, sugars, orange oil, and carotenoids. In this study, the QuEChERS (acronym for Quick, Easy, Cheap, Effective, Rugged) method was used to extract carotenoids. In this study, the QuEChERS (acronym for Quick, Easy, Cheap, Effective, Rugged) method was used to extract carotenoids.

EXPERIMENTAL

Apparatus

Graphitized carbon black (GCB) is often used to clean up pigmented samples, however it also adsorbs co-extractives. Because GCB adversely affects the recovery of carbendazim concentrations, it is not a suitable cleanup method. In this study, dispersive solid phase extraction (dSPE) was used to clean up the orange juice samples. In this study, dispersive solid phase extraction (dSPE) was used to clean up the orange juice samples.

Carbendazim concentrations are listed in Table 3. Among the six samples tested, only Sample 3 was detected with a positive result at 5.3 ng/mL.

INSTRUMENTAL

LC: Thermo Acqua equipped with PDA, auto-sampler, Guard column: Restek C18, 2.1*10 mm

INSTRUMENTAL

Analytical column: Synergi RP-C18, 2.1*100 mm, 3 µm, 120 Å

Column temperature: ambient (about 20°C)

Injection volume: 5 µL at 16°C

Flow rate: 200 µL/min

Gradient program:

Time (min) %A %B 0 98 2 0 100 20 0 100 60 0 100 90 0 100

MS/MS: Thermo TSQ Vantage triple quadrupole

Internal standard: Triphenylphosphine (TPP)

Signal modulation: 3500 V

Product ion: 132.0780, 133.0780

Injection volume: 200 µL/min

Instrumental parameters:

Table 1: SRM transitions

Table 2: Recovery and Reproducibility Data

RESULTS

Matrix matched calibration

Table 2: Recovery and Reproducibility Data

Application to real samples

The chromatograms of Sample 3 and Sample 3 fortified with 50 ng/mL of carbendazim are shown in Figure 1. The detected concentration was far below the EU's maximum allowable level of 200 ng/mL in orange juice.

CONCLUSIONS

A simple, fast, inexpensive, and effective method using QuEChERS extraction and dSPE cleanup was developed for the extraction and concentration of carbendazim in orange juice. The method is suitable for the detection of carbendazim in orange juice with a detection limit of 0.15 kg. The method is suitable for the detection of carbendazim in orange juice with a detection limit of 0.15 kg.