Analysis of Patulin in Fruit Juices and Extracts Using Liquid Chromatography Triple Quadrupole Mass Spectrometry

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ABSTRACT

Purpose: Patulin is a mycotoxin produced by different types of fungi as their secondary metabolite. The regulatory authorities have imposed restrictions on maximum patulin levels in fruits, apple juice and apple juice products. Patulin produces a wide range of adverse health effects. A rapid and sensitive LC/MS/MS method was developed and validated to analyze patulin in apples and apple juice products.

METHODS

Two different LC/MS/MS methods were tested using Thermo Scientific™ TSQ Fortis™ XE Triple Quadrupole Mass Spectrometer and Thermo Scientific™ Vanquish UHPLC System. Results: UPLC-MS/MS method for quantification of patulin was developed and tested.

INTRODUCTION

Patulin (5-hydroxy-1H-3-benzo[c]pyran-3-carboxylic acid, CAS# 715-35-9), is a metabolite produced as a mycotoxin by several fungal species and causes adverse health effects in humans. The maximum levels are regulated and are based on the toxicological characterization of the mycotoxin. The regulatory authorities have imposed restrictions on maximum patulin levels in different products, namely in apple juice. These limits are based on the tolerances for different food commodities (between 0.1 ppm and 1 ppm). The Commission Regulation (EU) No 1129/2011 set a limit for patulin in apple juice (maximal acceptable concentration (MAC) for patulin of 100 ppb (100 μg/kg) for the EU).

MATERIALS AND METHODS

Chemicals and Sample Preparation

Patulin was obtained from Sigma-Aldrich, all solvents and reagents were supplied from Fisher Scientific. The standard solution for the validation was prepared as 1, 5, 10, 50, 100 and 200 μg/kg to be diluted in water. A blank juice was used to prepare matrix-matched standards from 1 to 100 ppb. The procedure was conducted in triplicate.

LC methodology

The TSQ Fortis Mass Spectrometer was used for all the examples described in this work.

Mass Spectrometry

The TSQ Fortis Mass Spectrometer was used for all the examples described in this work.

LC method #1

Column: XBridge C18, 3.5 μm, 100 x 2.1 mm Flow rate: 0.3 mL/min; 1.0 mL/min Mobile phases: A: 0.1% formic acid in water Mobile phases: B: 0.1% formic acid in acetonitrile

LC method #2

Column: XBridge C18, 3.5 μm, 100 x 2.1 mm Flow rate: 0.3 mL/min; 0.5 mL/min Mobile phases: A: 0.1% formic acid in water Mobile phases: B: 0.1% formic acid in acetonitrile

Absolute Retention Times of Patulin: 2.8 min and 2.0 min

RESULTS

A linear relationship between the peak area and the concentration was observed in the range of 0.5-200 ppb. The linearity was found to be 0.999 in the 0.5-200 ppb concentration range in both neat and apple juice.

CONCLUSIONS

Method #1 (UPLC-MS/MS) quantitative method for patulin in apple juice products was developed using TSQ Fortis Triple Quadrupole Mass Spectrometer. Method #2 demonstrated better sensitivity, but required longer column equilibration than simpler method.

REFERENCES

2. LS 4991 (1958) Production of patulin in apple juice by Pseudomonas expansum (Lelio H. Lucifero, 1942).
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