

Accepted Manuscript

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PII: S0956-7135(18)30432-8

DOI: [10.1016/j.foodcont.2018.08.025](https://doi.org/10.1016/j.foodcont.2018.08.025)

Reference: JFCO 6289

To appear in: *Food Control*

Received Date: 29 May 2018

Revised Date: 21 August 2018

Accepted Date: 22 August 2018

Please cite this article as: Tu Q., Hickey M.E., Yang T., Gao S., Zhang Q., Qu Y., Du X., Wang J. & He L., A simple and rapid method for detecting the pesticide fipronil on egg shells and in liquid eggs by Raman microscopy, *Food Control* (2018), doi: 10.1016/j.foodcont.2018.08.025.

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A Simple and Rapid Method for Detecting the Pesticide Fipronil on Egg Shells and in Liquid Eggs by Raman Microscopy

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ABSTRACT: The pesticide fipronil was recently involved in a “poisonous eggs” scandal which resulted in substantial economic losses and public health risks. We report a simple and rapid method which can be implemented to detect fipronil on egg shells and in liquid eggs by Raman spectrometry. Fipronil rapidly crystallizes when solubilized in water-acetone solution (1:6, V/V, acetone/water). This process can be visually observed by naked eyes, suggesting a possible rapid and instrument-free screening action. Then, fipronil crystals were concentrated and recovered on a gold-coated glass slide for further identification and quantification by Raman microscopic analyses. A standard curve was established to quantify fipronil within the range of 1 to 500 mg/L ($r^2 \geq 0.997$), based on the unique fipronil Raman shift at 2256 cm^{-1} . Acetone based extraction recovered fipronil at three spiked levels of 5, 60 and 100 mg/kg on chicken egg shells and in liquid eggs from 59.91% to 81.72% and 85.97% to 152.46%, respectively. The limit of detection translated to the weight of liquid egg (0.32 mg/kg) and the surface area of an egg shell (0.065 mg/m^2). In conclusion, this method has demonstrated a strong capacity for the rapid (< 30 min) detection of fipronil on chicken egg shells and in liquid eggs.

Keywords: Fipronil, Detection, Raman spectroscopy, Chicken egg shells, Liquid eggs

1. Introduction

The European “poisonous eggs” incident was the largest European food scandal in 2017 and involved at least 40 countries (Reuters, 2017). Millions of eggs have been recalled across Europe

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