Multi-class method for the determination of nitroimidazoles, nitrofurans, and chloramphenicol in chicken muscle and egg by dispersive-solid phase extraction and ultra-high performance liquid chromatography-tandem mass spectrometry

Zhiwen Zhang,1, Yuping Wu,1, Xiaowei Li, Yingyu Wang, Hui Li, Qin Fu, Yawen Shan, Tianhe Liu, Xi Xia,

⇑ Corresponding author.
E-mail address: xxia@cau.edu.cn (X. Xia).
1 The first two authors contributed equally to this work.

© 2016 Elsevier Ltd. All rights reserved.

1. Introduction
Nowadays, there is increasing concern about the health and safety impact of the extensive use of veterinary drugs in livestock husbandry, which can result in residues in edible animal products. In order to ensure human food safety, many countries set strict tolerance levels for these compounds and perform surveillance programs. Among the major groups of compounds to be monitored, some zero tolerance substances, like nitroimidazoles (NIIMs), nitrofurans (NFs), and chloramphenicol (CAP), are prohibited globally (European Commission, 2010) due to their carcinogenic and mutagenic potency (World Health Organisation, 1989, 1993) or the risk of causing aplastic anemia (Festing, Diamanti, & Turton, 2001). The European Commission has established the minimum required performance level (MRPL) at 0.3 μg/kg and 1 μg/kg for CAP and NFs (European Commission, 2003), respectively, and recommended value was 3 μg/kg for NIIMs (Zeleny, Schimmel, Ulberth, & Emons, 2009b). Consequently, methods to detect and quantify the residues of these compounds in animal tissues must be very sensitive as well as selective.

The most important NFs are furazolidone, furaltadone, nitrofurazone and nitrofurantoin, which are metabolized rapidly after their administration (Chu & Lopez, 2005). Their toxic metabolites, 3-amino-2-oxazolidinone (AOZ), 3-amino-5-morpholinomethyl-2-oxazolidinone (AMOZ), semicarbazide (SEM), and 1-aminohydantoin (AHD), are strongly bound to proteins and highly stable for long periods (McCacken, Blanchflower, Rowan, McCoy, & Kennedy, 1995). For this reason, the analysis of NFs is based on the determination of their main metabolites (Conneely et al., 2003; Leitner, Zöllner, & Lindner, 2001). The most frequently used NIIMs included metronidazole (MNZ), dimetridazole (DMZ), ronidazole (RNZ), and ipronidazole (IPZ). Their major metabolites, 2-hydroxymethyl-1-methyl-5-nitroimidazole (HMMNI), 1-(2-hydroxyethyl)-2-hydroxymethyl-5-nitroimidazole (MNZOH), 1-meth
Analytical methods have been described for the determination of NIMs (Cronly, Behan, Foley, & Malone, 2009; Gadaj et al., 2014; Mitrowska, Posyniak, & Zmudzki, 2010; Mohamed et al., 2008; Polzer, Stachel, & Gowiak, 2004; Zeleny, Harbeck, & Schimmel, 2009a), NFs (Barbosa, Freitas, Mourão, da Silveira, & Ramos, 2012; Bock, Stachel, & Gowiak, 2007; Khong et al., 2004; Lu et al., 2012; Rønning, Einarsen, & Asp, 2006; Santos et al., 2005). Although a number of analytical methods have been reported, a comprehensive method was developed to cover more analytes and obtain higher throughput. Applicability of the method was demonstrated in the analysis of 46 commercial samples from local markets.

2. Experimental

2.1. Materials and reagents

HPLC grade methanol (MeOH), acetonitrile (ACN), formic acid, hexane, and ethyl acetate (EtOAc) were obtained from Fisher Scientific Inc. (Pittsburgh, PA, USA). Hydrochloric acid was obtained from Alfa-Aesar (Ward Hill, MA, USA). 2-Nitrobenzaldehyde (2-NBA) and diatomaceous earth (Celite® 545) were purchased from Sigma-Aldrich (St. Louis, MO, USA). C18 (50 μm) was purchased from Bonna-Agela Technologies (Beijing, China). Water was purified using a Milli-Q Synthesis system from Millipore (Bedford, MA, USA). Syringe filter was purchased from Pall Corporation (Ann Arbor, MI, USA).

The analytical standards AOZ, AMOZ, AHD, SEM, AOZ-d5, AMOZ-d3, AHD-13C3, SEM (13C, 15N2), RNZ, MNZOH, HMMNI, IPZ, IPZOH, HMMNI-d3, and MNZOH-d3 were obtained from WitEga (Berlin, Germany). RNZ-d3 and DMZ-d3 were acquired from RIVM (Bilthoven, Netherlands). IPZ-d5, IPZOH-d5, and MNZ-d3 were provided by the EU Reference Laboratory for Residues of Veterinary Drugs (Berlin, Germany). CAP, MNZ, and DMZ were purchased from Sigma-Aldrich. CAP-d5 was purchased from Dr. Ehrenstorfer (Augsburg, Germany).

Standard stock solutions (1 mg/mL) were prepared by dissolving 10 mg of individual compounds in 10 mL of methanol with the exception of SEM (13C, 15N2), which was dissolved in water. Intermediate standard solutions of each compound (10 μg/mL) were prepared by dilutions with methanol from stock solutions. These solutions were stored at −20°C and were stable for at least 6 months. Mixed working standard solutions for spiking blanks (0.5, 0.1, 0.01 μg/mL) were obtained by appropriate dilutions with methanol. Working standard solutions of CAP (0.5, 0.1, 0.01 μg/mL) were prepared separately. Mixed working internal standard (IS) solution was prepared in methanol at 0.5 μg/mL. These solutions were stored at −20°C and were stable for at least 8 weeks.

2.2. Sample preparation

For routine testing samples, a previously homogenized sample (2.00 ± 0.02 g) was weighed into a 50 mL centrifuge tube. For spiked samples, 50 mL of working solutions at each concentration were added to blank samples. Eight μL of working IS solution (0.5 μg/mL) was spiked into each sample to obtain the concentration of 2.0 μg/kg. After vortexing, samples were allowed to stand for 30 min in the darkness. Ten mL of 0.2 M hydrochloric acid solution and 200 μL of 0.1 M 2-NBA freshly prepared in methanol were added and vortex-mixed for 1 min. The samples were placed in a shaker at 200 rpm and incubated overnight at 37°C. After cooling to room temperature, 4 g of C18 and 4 g of diatomaceous earth were added into the samples, and then 10 mL of hexane was added and shaken at 200 rpm for 5 min. The mixtures were centrifuged at 10,000 rpm for 5 min, and the aqueous layer and hexane layer were discarded. The samples were extracted with 15 mL of ethyl acetate by vortexing for 1 min and centrifuged at 10,000 rpm for 5 min. The supernatant was collected and the extraction was repeated with 15 mL of ethyl acetate. The combined extraction solutions were evaporated to dryness in a water bath at 40°C under nitrogen. The dried extract was reconstituted in 1.0 mL of 5% methanol in 0.1% formic acid, vortex-mixed for 1 min, and filtered through a 0.2 μm syringe filter into a glass LC vial.

2.3. Instrumental conditions

LC analyses were performed on a Waters Acquity ultraperformance liquid chromatography system with column oven temperature maintained at 40°C, using an Acquity BEH C18 column (50 mm × 2.1 mm i.d., 1.7 μm particle size) (Waters, Milford, MA, USA). The mobile phase was constituted by solvent A (0.1% formic acid in water) and solvent B (0.1% formic acid in acetonitrile). The injection volume was 5 μL. The flow rate was 0.4 mL/min with a linear gradient at the following conditions: 0–0.5 min, 98% A; 0.5–2 min, 98–85% A; 2–4.5 min, 85–60% A; 4.5–5.5 min, 60–1% A; 5.5–6 min, 1% A; 6–6.1 min, 1–98% A; 6.1–7.5 min, 98% A.
دانلود مقاله

http://daneshyari.com/article/1184757