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Analytical Methods

Quantitative analysis of lincomycin and narasin in poultry, milk and eggs by liquid chromatography–tandem mass spectrometry

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ABSTRACT

This study presents the simultaneous extraction and determination of lincomycin (LCM) and narasin (NAR) by using liquid chromatography–electrospray ionisation tandem mass spectrometry (LC–MS/MS) on samples from poultry, milk and eggs ($n = 196$). The homogenised samples are extracted with acetonitrile and the extract is further cleaned using C_{18} solid-phase extraction cartridges. The recoveries of the analytes in different matrices were found ranging from 90% to 101% and 85% to 95% for LCM and NAR, respectively. The corresponding limits of detection were 0.6 and 1.5 ng g^{-1} for LCM and NAR, respectively. As a result of monitoring, NAR was not detected in any samples and LCM was detected in one egg with a concentration of 25 ng g^{-1}. The method was relatively simple to perform and therefore could be used for food safety surveillance activities.

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1. Introduction

Veterinary drugs have become a necessary part of the livestock production and play an important role in the maintenance of animal health, mainly for the prevention of disease, the curing of infection and also increasing the productive capacity of animals. However, the use of veterinary drugs may induce the presence of drug residues in animal food products if appropriate withdrawal times are not respected, or through the use of contaminate feed (Carretero, Blasco, & Picó, 2008; Marazuelaa & Bogialli, 2009).

The Korea Food and Drug Administration (KFDA) which regulates the use of veterinary drugs in food producing animals in Korea, established new regulation for 44 veterinary drugs in food and food-producing animal tissues and they have set out maximum residues levels (MRLs) for them in 2008 (Table 1) (Korean Food and Drug Administration, 2008). The regulations include lincomycin (LCM) and narasin (NAR) which are belong to feed additives antibiotics. In Korea, feed additives are the most used for veterinary drugs and the portion is about 37% of total use. Owing to the widespread using of these drugs on farms, withdrawal times have been established for these substances, but there is a risk that residues will be present in animal products intended for human consumption. Therefore, suitable methods must be available for monitoring the presence of veterinary drugs in animal food products from improper use or from feed cross contamination.

LCM, which is a member of the lincomaside group, is a commonly administered antibiotic used to control post-weaning diarrhoea. LCM is effective against most Gram-positive organisms, including staphylococci and some streptococci, by inhibiting protein synthesis. NAR is a member of the ionophore antibiotic, which is the most successful anticoccidial agent in use today, due primarily to their effectiveness in preventing coccidiosis in different animal species and because they cause a little or insignificant drug resistance problems. In addition to their anticoccidial activity, the ionophores have growth properties because they improve feed efficiency and increase the rate of weight gain by selectively inhibiting some bacteria, including the methane producers, which are less efficient as feed conversion.

Several methods have been reported for the determination of the LCM in animal tissues, such as using GC with derivatisation (Luo, Yin, Ang, Rushing, & Thompson, 1996), and HPLC with electrochemical detection in milk and animal tissues (Moats, 1991). Recently, LC–MS/MS with electrospray ionisation (ESI) has been used to determine the LCM in meat and milk (Cristina, Juan, Jordi, & Guillermina, 2010). Methods have also been reported for the determination of the NAR in the edible tissues of chicken using HPLC (Ward, Moran, Turner, & Coleman, 2005). To detect the substances in low concentrations and to confirm the presence of the analytes, liquid chromatography linked to mass spectrometric

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Table 1
Maximum residual levels (MRLs) of veterinary drugs in livestock.

Veterinary drugs	Republic of Korea (μg kg^{-1})[a]	EU (μg kg^{-1})	Codex (μg kg^{-1})[c]
Lincomycin	Bovine muscle 100 Bovine liver 500 Bovine fat 50 Bovine kidney 1000 Milk 150 Egg 50 Swine muscle 200 Swine liver 500 Swine fat 100 Swine kidney 1500 Fish 100 Poultry muscle 200 Poultry liver 500 Poultry fat 100 Poultry kidney 500 Crustacean 100	All food producing species[b]: Muscle 100 Fat 50 Liver 500 Kidney 1500 Milk 150 Egg 50	Cattle milk 150 Chicken muscle 200 Chicken liver 500 Chicken kidney 500 Chicken fat 100 Pig muscle 200 Pig liver 500 Pig kidney 1500 Pig fat 100
Narasin	Poultry muscle 100 Poultry liver 300 Poultry fat 500 Poultry kidney 300 Egg N.D.	Provisional MRL[d]: Chicken muscle 50 Chicken liver 50 Chicken kidney 50 Chicken fat 50	Chicken muscle 15 Chicken liver 50 Chicken kidney 15 Chicken fat 50

[a] Korean Food Code (2008).
[b] Commission Regulation 37/2010 and amendments.
[c] Codex Alimentarius Commission: maximum residue limits for veterinary drugs in foods (2009).
[d] Codex Alimentarius Commission E: Codex committee on residues of veterinary drugs in foods (2007).

detection has been developed in bovine, porcine or chicken muscles using LC–MS/MS (Yamada, Kozono, Ohmori, Morimatsu, & Kitayama, 2006), in milk (Thompsona, Nootb, & Kendalla, 2011), in egg (Dubois, Pieeret, & Delahaut, 2004) and in eggs and broiler meat (Rokka & Peltonen, 2006). These methods generally involve solvent extraction of the residues followed by a clean-up stage or target a single class of veterinary drugs. Actually, a way to improve cost-effectiveness of method is to perform different class determination therefore generic sample preparation procedures are necessary to simultaneously extract a different range of veterinary drugs. However, the methods have limitations for their application to the simultaneous determination of the LCM and NAR residues in animal food products regarding the analysis time and the throughput of samples per working day, which are important factors.

The aim of this study was to present a rapid and simultaneous method of determining the presence of two drugs, LCM and NAR, from different chemical groups, which could be used to examine variety food matrices. The above method was validated and was used to surveillance monitoring of targeted chemical residues in a large number of poultry, milk and eggs.

2. Material and methods

2.1. Standards and reagents

Standard materials of lincomycin hydrochloride (>90%) and narasin (from *Streptomyces auriofacien*, approx. 97%) were purchased from Sigma–Aldrich. Ammonium acetate (anhydrous, 98%) and sodium phosphate monobasic monohydrate ASC reagent were also obtained from Sigma–Aldrich, and sodium phosphate dibasic dodecahydrate was obtained from Riedel-de-Haen. HPLC grade acetonitrile, hexane and methanol were obtained from Burdick & Jackson. Acetone and sodium hydroxide were obtained from Wako pure chemical Industries, Ltd. (Japan). Formic acid, 98%, was obtained from Flucka. All chemicals used were of analytical grade unless stated otherwise. Distilled, deionised water was generated in-house with the Milli-Q system (Milli-Pore; resistance ⩾ 18.2 MΩ). The solid phase extraction (SPE) columns were C_{18} cartridges (Waters, 500 mg, Milford, MA).

2.2. Preparation of standards and solutions

Stock standard solutions (100 μg mL^{-1}) of LCM and NAR were prepared by weighing and dissolving each antibiotic in methanol. The stock solutions were stored in the dark at 4 °C, and were used for less than 1 month. Dilutions into methanol were carried out down to 10, 1 or 0.1 μg mL^{-1} and below. Standard working mixtures, at various concentrations, were prepared daily by appropriate dilution of aliquots of the stock solutions in methanol. A working standard mixture containing 0.1 μg mL^{-1} of each compound was prepared in methanol for use as a spiking solution. 0.2 M phosphate buffer solution (pH 7.2) was prepared by mixing 28 mL of 0.2 M sodium phosphate monobasic monohydrate and 72 mL 0.2 M sodium phosphate dibasic dodecahydrate.

2.3. Preparation of samples and sample extraction

A total 196 samples were analysed, including 65 of poultry muscle, 66 of milk and 66 of eggs. All of them were collected from Korean local markets in various districts. Every sample was individually labelled with a code that made it fully traceable back to the farm where it was produced.

The minced poultry muscle tissues, milk and whole eggs (excluding shells) were homogenised by a blender and then stored at −20 °C until analysis. However, the milk and eggs were analysed fresh, which gave no significant difference regarding recovery or precision.

The sample extraction procedure described in the Korea Food Code method was used as a basis (Korea Food and Drug Administration, 2009).

2.3.1. Milk

0.2 M phosphate buffer solution (3 mL) and 1 M sodium hydroxide (120 μL) were added to each 15 mL polypropylene tube containing 2 g blended milk. The tubes were capped and vortex-mixed for 10 s and then acetonitrile (6 mL) was added to each tube. The tubes were tightly sealed and shaken sideways vigorously and centrifuged at approximately 1650*g* at 4 °C for 5 min. The samples were placed for 30 min at −20 °C and the supernatant solution

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