



# Highly sensitive and simultaneous determination of sixteen sulphonamide antibiotics, four acetyled metabolites and trimethoprim in meat by rapid resolution liquid chromatography-tandem mass spectrometry

Hui Li<sup>a,b</sup>, Hanwen Sun<sup>a,\*</sup>, Jingxuan Zhang<sup>b</sup>, Kun Pang<sup>b</sup>

<sup>a</sup> College of Chemistry and Environmental Science, Hebei University, Key Laboratory of Analytical Science of Hebei Province, Wusi East Road 188, Baoding 071002, China

<sup>b</sup> Hebei Institute of Food Quality Supervision Inspection and Research, Shijiazhuang 050051, China

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## ABSTRACT

A novel multiresidue analysis method is developed for highly sensitive and simultaneous determination of 16 sulphonamides (SAs), 4 acetyled metabolites, and trimethoprim in pork and mutton by rapid resolution liquid chromatography-tandem mass spectrometry (RRLC-MS/MS). The sample was extracted with acetonitrile under ultrasonication incubation, followed by solid phase extraction (SPE). The calibration curves showed good linearity with correlation coefficient ( $r$ ) more than 0.998. The limit of quantification (LOQ) was 0.35–1.0  $\mu\text{g}/\text{kg}$ , which can ensure to detect studied drugs at the maximum residue level (MRL) of 10  $\mu\text{g}/\text{kg}$ . The mean recoveries at addition level of 1.0, 5.0 and 50  $\mu\text{g}/\text{kg}$  were in the range of 68.3–104% with the relative standard deviation (RSD) of 3.5–9.2%. The intra-day precision (as RSD) for six determinations at 50  $\mu\text{g}/\text{kg}$  spiked level within a day was in the range of 4.2–8.9%. The method is sensitive, accurate, convenient and rapid, and can be used for the qualitatively and quantitatively determination of multiresidue of the studied drugs in meat.

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## 1. Introduction

The residues of sulphonamides (SAs) in foods of animal origin are a major concern because they are harmful to the consumer's health, and could induce pathogens to develop resistance. SAs can be acetylated at the  $\text{N}^4$ -position.  $\text{N}^4$ -acetyl metabolite of SAs in food-producing animals may cause the renal toxicity as a result of precipitation in the kidney (Vree & Hekster, 1985), and may affect their excretion rates due to higher plasma protein binding than the parent compound (Vree, Hekster, Nouws, & Dorrestein, 1987). Trimethoprim is another antibiotic agent, which is often co-administered with sulfamethoxazole to enhance treatment against a variety of bacterial infections. In humans and animals, it can cause changes in the bone marrow and significant effects on some organ weights. To ensure food safety for consumers, the European Community, the U.S. Food and Drug Administration (FDA), and China Agriculture Department have laid down the maximum residue level (MRL) of 100  $\mu\text{g}/\text{kg}$  for SAs in foodstuffs of animal origin and 50  $\mu\text{g}/\text{kg}$  for trimethoprim in muscle, fat/skin, liver and kidney tissues of pigs (China Agriculture Ministry, 2002; European Union Regulation,

2010; Food and Drug Regulation, 1991, pp. 1478–1480). Korea set an MRL of 100  $\mu\text{g}/\text{kg}$  as sum of the 14 SAs in meat and milk (Korea Food Code, 2008). The Japan's Positive List System presents an MRL of 50  $\mu\text{g}/\text{kg}$  for sulfaquinolaxaline, 20  $\mu\text{g}/\text{kg}$  for sulfamerazine, 40  $\mu\text{g}/\text{kg}$  for sulfadimethoxyypyrimidine, and 10  $\mu\text{g}/\text{kg}$  for sulfamethazine. For other SAs the MRL takes 10  $\mu\text{g}/\text{kg}$  (Ministry of Health, Labour and Welfare of Japan, 2006). In order to better assess the occurrence of SAs in food, their metabolites should also be considered. These early conclusions evidenced the need for analytical methods capable to simultaneously determine parent compound as well as their metabolites at the low concentrations normally present in food. Therefore, it is urgently in need of developing rapid and effective method for the simultaneous determination of SAs residues and their metabolites in meats.

High-performance liquid chromatography (HPLC) as separation and analytical technology has been used widely to detect veterinary residues in food. A series of analytical methods were reported for the determination of SAs residues in food (See Table 1).

Analytical detection limits of HPLC-UV method are generally limited by significant signal interference associated with UV spectral overlaps with other food constituents. Recently, several HPLC-UV/DAD methods combined with effective extraction and cleanup were reported for the determination of residual SAs in food (Huang et al., 2012; Kowalski, Plenis, Oledzka, & Konieczna, 2011; Shi et al.,

\* Corresponding author. Tel.: +86 312 5079719; fax: +86 312 5079739.

E-mail address: hanwen@hbu.edu.cn (H. Sun).

**Table 1**  
References for the determination of SAs, acetylated metabolites and trimethoprim in food.

Analyte	Matrix	Extraction	Cleanup	Final method	LOD/recovery/RSD	Ref.	Ref. no.
6 SAs <sup>c</sup>	Shrimp, fish	Ultrasonication, 1% acetic acid	Molecularly imprinted polymers	LC–UV	8.4–10.9 µg/kg, 85.5–106.1%, 1.2–7.0%	Shi et al., 2011	1
7 SAs	Milk	Cloud point extraction, n-butyl alcohol		LC–UV	2.23–9.79 µg/L, 67.0–105.7%, 93–8.31%	Zhang et al., 2011	2
7 SAs	Poultry tissue	Ultrasonication, acetonitrile	C18 cartridges	MECC <sup>a</sup>	1.3–7.8 µg/kg, >77.2%, 9.5%, 11.2%	Kowalski et al., 2011	3
4 SAs	Milk, chicken muscle	Acetonitrile–water	Micro-solid phase extraction	LC	4.52–15.63 µg/kg, 43.11–83.81%	Huang et al., 2012	4
2 SAs, etc.	Porcine tissues	TwogramsC18, EDTA-Na <sub>2</sub> –oxalic acid	Matrix solid-phase dispersion	LC–DAD	LOQ: 7–34 µg/kg, 80.6–99.2%, <6.1%	Yu et al., 2012	5
6 SAs	Cattle meats	Acetonitrile		LC–FLD	8–15 µg/kg, 44.67–81%, <6%	Mor, Kocasari, Ozdemir, & Oz, 2012	6
6 SAs	Milk	Water, dichloromethane		PCSF <sup>b</sup> –MS	41–181 µg/kg	Dost, Jones, & Davidson, 2000	7
14 SAs	Milk, egg	Distilled water	Solid-phase extraction	LC–MS	1–10 µg/kg, 76–112%, <13%	Cavaliere et al., 2003	8
13 SAs	Raw meat, infant foods	Accelerated hot water extraction		LC–MS/MS	2.6 µg/kg, 70–101%	Gentili et al., 2004	9
16 SAs	Poultry/meat	Acetonitrile		LC–MS/MS	1.0–12.0 µg/kg, 75.4–97.3%, 3.48–14.09%	Pang et al., 2005	10
12 SAs	Pork meat	Pressurized liquid extraction, water	Oasis HLB cartridge	CE–MS/MS	<12.5 µg/kg, 76–98%, <14%)	Font et al., 2007	11
8 SAs	Pork	Ultrasonication, water	Solid-phase microextraction	LC–MS	16–39 µg/kg below 15%	Lu et al., 2007	12
17 SAs, etc.	Fish tissue	Ultrasonication, MeOH:CAN: formic acid		LC–MS/MS	5.65–24.0 µg/kg, <20%	Dasenaki & Thomaidis, 2010	13
5 SAs	Honey	Acetate buffer–octanol:pentanol	Hollow fiber renewal liquid membrane	LC–MS/MS	5.1–27.4 µg/kg, 80.9–103.1%, <15%	Bedendo et al., 2010	14
18 SAs	Muscles, livers, kidneys	Pressurized liquid extraction with acetonitrile	Hydrophilic–lipophilic balance cartridge	LC, LC–MS/MS	3 µg/kg, 71.1–118.3%, <13%	Yu et al., 2011	15
14 SAs	Marine products	Ultrasonication, acetonitrile	C18 powder	LC–PDA, LC–MS/MS	3–6 µg/kg, 51.8–89.7%, 5.6–8.6%	Won et al., 2011	16
13 SAs	Grass, carp tissues	C18-bonded silica	Matrix solid-phase dispersion	LC–MS/MS	0.75–3.0 µg/kg, 69.0–96.3%, ≤13.2%	Lu et al., 2011	17
6 SAs	Milk, milk powder	Hydrochloric acid (pH 2) –15% NaCl	C18-stir bar sorptive extraction	LC–MS/MS	2.7–31.5 µg/kg, 68–115%	Yu & Hu, 2012	18
SMM, <sup>d</sup> AcSMM, <sup>e</sup> SDM, <sup>f</sup> AcSDM <sup>g</sup>	Eggs	Ultrasonication saturated ammonium sulphate		HPLC–DAD	20, 30 µg/kg, >91%, within 4%.	Kishida, 2007	19
SMM, AcSMM, SDM, AcSDM	Chicken plasma, tissues, eggs	Ultrasonication, ethanol		HPLC–DAD	<30 µg/kg, >90% within 4%.	Kishida & Furusawa, 2005	20
SMM, AcSMM, SDM, AcSDM	Milk	Ultrasonication, ethanol–acetic Acid (97:3, v/v)		HPLC–DAD	LOQ: <25 µg/L, >81%, within 5%	Kishida & Furusawa, 2004	21
SDM, AcSDM	Chicken meat	Ultrasonication 20% (w/v) perchloric acid		HPLC–DAD	LOQ: ≤0.1 mg/kg, ≥84%, ≤6%	Furusawa, 2007	22
SDA, <sup>h</sup> TMP <sup>i</sup>	Broiler tissues	Ethyl acetate or dichloromethane	Hexane–chloroform in an acidic buffer	HPLC	15–100 µg/kg SDA, 20–25 µg/kg TMP	Dagorn & Delmas, 1994	23
SDA, TMP	Swine tissues	3 mL of 0.1 M acetic acid in water	SCX column	HPLC–DAD LC–MS/MS	15 µg/kg SDA, 20 µg/kg TMP	De Baere et al., 2000	24

<sup>a</sup> MECC — micellar electrokinetic capillary chromatography.

<sup>b</sup> PCSFC — packed column supercritical fluid chromatography.

<sup>c</sup> SAs — sulfonamides.

<sup>d</sup> SMM — sulfamonomethoxine.

<sup>e</sup> AcSMM — N4-acetyl sulfamonomethoxine.

<sup>f</sup> SDM — sulfadimethoxine.

<sup>g</sup> AcSDM — N4-acetyl sulfadimethoxine.

<sup>h</sup> SDA — sulfadiazine.

<sup>i</sup> TMP — trimethoprim.

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