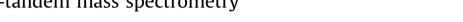
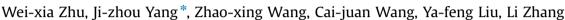
Contents lists available at ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta

Rapid determination of 88 veterinary drug residues in milk using automated TurborFlow online clean-up mode coupled to liquid chromatography-tandem mass spectrometry





Henan Entry-Exit Inspection and Quarantine Bureau of China, Zhengzhou 450003, China

ARTICLE INFO

Article history Received 9 July 2015 Received in revised form 12 October 2015 Accepted 14 October 2015 Available online 29 October 2015

Keywords: Veterinary drugs Multi-residue analysis Milk TurborFlow online solid phase extraction Automated online clean-up Mass spectrometry

ABSTRACT

A novel method based on TurborFlow online solid phase extraction (SPE) combined with liquid chromatography-tandem mass spectrometry (LC-MS/MS) has been established for simultaneous screening and confirmation of 88 wide-range veterinary drugs belonging to eight families (20 sulfonamides, 7 macrolides, 15 quinolones, 8 penicillins, 13 benzimidazoles, 4 tetracyclines, 2 sedatives, and 19 hormones) in milk. The preparation method consists of sample dilution and ultrasonic extraction, followed by an automated turbulent flow cyclone chromatography sample clean-up system. The detection was achieved in selected reaction monitoring mode (SRM). The total run time was within 39 min, including automated extraction, analytical chromatography and re-equilibration of the turboflow system. The optimization of different experimental parameters including extraction, purification, separation, and detection were evaluated separately in this study. The developed method was validated and good performing characteristics were obtained. The linear regression coefficients (R^2) of matrix-match calibration standard curves established for quantification were higher than 0.9930. The limits of detection (LOD) were in the range of 0.2–2.0 μ g/kg given by signal–noise ratio \geq 3 (S/N) and the limits of quantification (LOQ, S/N \ge 10) ranged between 0.5 µg/kg and 10 µg/kg. Average recoveries of spiked target compounds with different levels were between 63.1% and 117.4%, with percentage relative standard deviations (RSD) in the range of 3.3–17.6%. The results indicated that the developed method has great potential for the routine laboratory analysis of large numbers of samples on measuring different classes of compounds. In comparison to traditional procedures, the automated sample clean-up ensures rapid, effective, sensitive analyses of veterinary drugs in milk.

© 2015 Elsevier B.V. All rights reserved.

1. Introduction

Veterinary drugs (VDs) such as sulfonamides, macrolides, quinolones, and hormones are increasingly applied to animal husbandry as well as in the animal feed for the prevention and treatment of animal diseases purposefully [1]. These drugs are essential in protecting animals from diseases for large commercial interests. However, the illegal use of these drugs can cause residues in several animal-origin products such as milk, meat, liver and honey. Long terms expose on VDs residues might induce acute poisoning, allergic reaction, and long term antibiotic resistance due to their toxicity [2]. Therefore, European Union Council Directives (96/22/EC, 96/23/EC) [3,4], and the Ministry of Agriculture of the People's Republic of China announcement No 235 [5] have set regulations on monitoring programs and establishing specific

* Corresponding author. Fax: +86 371 55196568. E-mail address: jizhouyang@163.com (J.-z. Yang).

http://dx.doi.org/10.1016/j.talanta.2015.10.037 0039-9140/© 2015 Elsevier B.V. All rights reserved. maximum residue limits (MRLs) for the vast majority of VDs in various food to monitor the VDs residual level in the products. Owing to the high consumption of milk, the regulation of various VDs residues in milk is extremely strict with the lowest MRLs for consumer's protection. The rigorous regulation and the dramatically increased number of sample analyses require efficient, accurate and sensitive methods for simultaneous detection of multiclass veterinary drugs residues in milk.

The reported methods for measuring VDs residues mainly included immunoassay [6], capillary electrophoresis (CE) [7], high performance liquid chromatography with diode array detection (HPLC-DAD) [8] or fluorescence detection (FLD) [9], and liquid chromatography-tandem mass spectrometry (LC-MS/MS) [10,11], liquid chromatography coupled with high resolution mass spectrometry [12]. Immunoassay is widely used because it is guick, inexpensive, and selective while fulfilled with the basic requirements. Jiang, et al. [13] developed a multiplex flow-through immunoaffinity chromatography test for screening sulfonamide and quinolone residues in milk. However, the lack of structural





talanta

Table 1

The CAS and mass parameters of 88 veterinary drugs.

No.	Family	Compounds	CAS#	Product (m/z)	Daught ion (m/z)	CE (eV)	Tubelens
1	Sulfonamides	Sulfamonomethoxine	1220-83-3	281.1	156.0ª 126.1	16 19	76 76
2	Sulfonamides	Sulfamethoxypyridazine	80-35-3	281.1	126.1 156.0ª	19 16	76
3	Sulfonamides	Trimethoprim	738-70-5	291.2	126.1 123.0	19 28	76 101
					230.0 ^a	22	101
4	Sulfonamides	Benzenesulfonamide	526-08-9	315.0	131.0 158.1 ^a	45 29	95 95
5	Sulfonamides	Sulfaquinoxaline	59-40-5	301.0	92.0 156.0ª	31 16	108 108
6	Sulfonamides	Sulfapyridine	144-83-2	250.0	155.9 ^a	15	84
7	Sulfonamides	Sulfathiazole	72-14-0	256.1	184.1 92.1	17 27	84 98
8	Sulfonamides	Sulfisoxazole	127-69-5	268.0	156.0ª 113.1	14 18	98 95
					156.0 ^a	15	95
9	Sulfonamides	Benzenesulfonamide	729-99-7	268.0	113.1 156.0 ^ª	18 15	95 95
10	Sulfonamides	Sulfadimethoxine	122-11-2	311.2	108.1	22	93
11	Sulfonamides	Sulfadoxine	2447-57-6	311.2	156.0 ^a 108.1	15 21	93 93
12	Sulfonamides	Sulfamethazine	57-68-1	279.0	156.0ª 124.1	15 21	93 74
					186.0 ^a	16	74
13	Sulfonamides	Benzenesulfonamide	515-64-0	279.0	124.1 ^a 186.0	21 16	74 74
14	Sulfonamides	Sulfamerazine	127-79-7	265.1	156.0	15	87
15	Sulfonamides	Sulfamethoxazole	723-46-6	254.2	172.0 ^a 108.1	16 23	87 89
16	Sulfonamides	sulfachlorpyridazine	80-32-0	285.1	155.9ª 156.0	15 15	89 92
					129.9 ^a	20	92
17	Sulfonamides	Sulfaclozine	102-65-8	285.2	108.1 155.9 ^a	28 15	92 92
18	Sulfonamides	Succinylsulfathiazole	116-43-8	356.0	108.0 255.9ª	27 16	116 116
19	Sulfonamides	Sulfabenzamide	127-71-9	277.1	108.0	22	91
20	Sulfonamides	Benzenesulfonamide	144-82-1	271.0	155.9 ^ª 92.018	12 27	91 80
					155.9 ^a	13	80
21	Macrolides	Erythromycin	114-07-8	734.4	158.2 ^ª 576.2	29 18	136 136
22	Macrolides	Kitasamycin	1392-21-8	772.4	109.1 174.0 ^a	38 30	150 150
23	Macrolides	Spiramycin	8025-81-8	843.4	141.9	32	175
24	Macrolides	Clindamycin	18323-44-9	425.0	174.2 ^a 126.1 ^a	35 30	175 110
25	Macrolides	Tilmicosin	108050-54-0	869.4	377.1 132.0	18 39	110 183
					174.1 ^a	40	183
26	Macrolides	VlrgInlamycin	11006-76-1	526.1	354.9 ^ª 508.0	17 11	128 128
27	Macrolides	Tiamulin	55297-95-5	494.3	119.0 192.0ª	36 19	110 110
28	Quinolones	Enoxacin	74011-58-8	321.1	206.0 ^a	25	106
29	Quinolones	Fleroxacin	79660-72-3	370.0	303.1 269.0 ^a	15 25	106 111
	-				326.1	17	111
30	Quinolones	Marbofloxacin	115550-35-1	363.0	72.1.0 320.0 ^a	23 14	104 104
31	Quinolones	Orbifloxacin	113617-63-3	396.1	295.0 ^ª 352.0	23 16	111 111
32	Quinolones	Danofloxacin	112398-08-0	358.0	283.0	22	110
33	Quinolones	Ciprofloxacin	85721-33-1	332.1	314.0 ^a 288.0 ^a	17 17	110 115
	Quinolones	Difloxacin	98106-17-3	400.0	314.0 298.9ª	19 27	115 118
34	-				355.9	17	118
35	Quinolones	Pefloxacin	70458-92-3	334.2	233.0ª 290.0	25 16	105 105
36	Quinolones	Sparfloxacin	111542-93-9	393.1	291.9	24	117
37	Quinolones	Ofloxacin	82419-36-1	362.1	349.0 ^a 261.0 ^a	17 25	117 109
					318.0	17	109

Download English Version:

https://daneshyari.com/en/article/1243801

Download Persian Version:

https://daneshyari.com/article/1243801

Daneshyari.com