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LCMS-8050

Application News

SGLC-LC/MS-031EN

Analysis of 22 benzimidazole veterinary drug residues in pork and pork liver

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User Benefits

- Established an effective, fast and simple sample preparation method for analysis of benzimidazole drug residues in pork and pork liver.
- Realized simultaneous quantitative analysis of 22 benzimidazole drug residues.
- SHIMSEN QuEChERS provides dSPE sorbent in centrifuge tube for enhanced laboratory efficiency and throughput.

■ Introduction

Benzimidazole veterinary drugs are one of the traditional antimicrobial agents widely used in the treatment of diseases in livestock and poultry. The residues of benzimidazole veterinary drugs in meat products from livestock and poultry can pose potential health hazards to humans. Therefore, the detection of benzimidazole veterinary drug residues in meat from livestock and poultry becomes particularly important. In this application, we present a complete workflow, from sample preparation using SHIMSEN QuEChERS, to sample analysis using Shim-pack Velox SP-C18 column on Shimadzu LCMS-8050.

Table 1. LCMS conditions

UHPLC condition:	
LC system:	Shimadzu Nexera™ LC-30
Column:	Shim-pack Velox SP-C18, 1.8 µm, 100 × 2.1 mm *1
Column Temp.:	40 °C
Flow rate:	0.25 mL/min
Mobile phase A:	0.1% formic acid aqueous solution
Mobile phase B:	acetonitrile
Gradient program:	14% B (0 min) → 25%B (2 min) → 90% B (2.5 min) → 90% B (3.5 min) → 14% B (3.51 min) → 14% B (5 min)
Injection volume:	5 µL
MS conditions:	
Interface:	Heated ESI (Positive or Negative)
Interface temp:	300 °C
Collision gas:	Ar
Nebulizing gas:	N2, 3 L/min
Heating gas flow:	N2, 10 L/min
DL temperature:	250 °C
Drying gas flow:	N2, 10 L/min
Heat block temp:	400 °C
MS mode:	MRM

*1 P/N: 227-32001-02

■ Experimental

Materials:

SHIMSEN QuEChERS dSPE tube (P/N: 380-00188)

Filter and vial:
SHIMSEN Disc HPTFE syringe filter (P/N: 380-00341);
LabTotal Vial (P/N: 227-34001-01)

Sample Preparation:

Weigh 5.0 g of the sample, add 5 mL of water, manually shake for 1 min, add 10 mL of acetonitrile, manually shake for 1 min, add 4 g of anhydrous sodium sulfate and 1 g of sodium chloride, manually shake for 1 min, centrifuge at 8000 rpm for 2 min, and 2 mL of the supernatant was transferred into SHIMSEN QuEChERs dSPE tube (100 mg C18, 100 mg PSA, 100 mg Al-N, and 500 mg anhydrous sodium sulfate), vortex for 1 min, centrifuge at 8000 rpm for 2 min, take 1 mL of the supernatant into a nitrogen evaporation tube, blow dry with nitrogen at 35°C, add 1 mL of 50% methanol water solution, vortex to redissolve, then filtered the solution through a 0.22 µm syringe filter for LC-MS/MS analysis. Figure 1 shows the simplified sample preparation workflow.

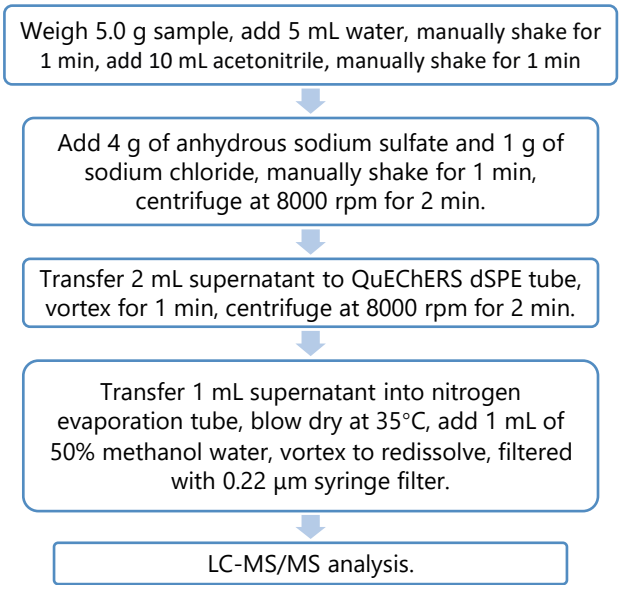

Figure 1. QuEChERS sample preparation workflow

Table 2. List of MRM used for each veterinary drug.

No.	Compounds	Precursor ion (m/z)	Quaatifyingion (m/z)	Q1 Pre Bias	CE	Q3 Pre Bias
1	5-hydroxythiabenzimidazole	217.80	191.10*	-11	-26	-18
		217.80	147.10	-11	-33	-13
2	carbendazim	192.00	160.10*	-10	-19	-30
		192.00	132.15	-14	-29	-11
3	albendazole-2-aminosulfone	239.90	133.15*	-10	-30	-29
		239.90	198.10	-10	-20	-11
4	thiabendazole	202.00	175.10*	-15	-26	-29
		202.00	131.15	-11	-32	-11
5	albendazole sulfoxide	282.00	240.10*	-11	-14	-24
		282.00	208.05	-11	-25	-20
6	hydroxylbenzimidazole	298.00	160.00*	-11	-35	-14
		297.90	266.10	-12	-24	-26
7	aminomethylimidazole	238.00	105.05*	-10	-26	-16
		238.00	77.10	-10	-36	-11
8	2-aminoflubendazole	256.00	123.05*	-10	-27	-20
		256.00	95.05	-10	-40	-14
9	conbendazole	303.00	217.05*	-11	-27	-18
		303.00	261.05	-12	-18	-25
10	albendazole sulfone	298.00	159.00*	-11	-37	-27
		298.00	223.80	-11	-27	-21
11	oxfendazole	315.90	159.05*	-10	-32	-14
		315.90	191.10	-10	-22	-18
12	oxibendazol	250.10	218.15*	-10	-20	-21
		250.10	176.15	-10	-28	-16
13	fenbendazole Sulfone	331.80	300.05*	-10	-23	-30
		331.80	159.05	-10	-38	-14
14	mebendazole	296.00	264.10*	-11	-22	-26
		296.00	105.05	-11	-35	-16
15	albendazole	266.00	234.10*	-11	-20	-14
		266.00	191.05	-10	-34	-17
16	dibenzimidazole	248.10	216.15*	-10	-20	-20
		248.10	173.15	-13	-32	-28
17	flubendazole	314.00	282.05*	-12	-24	-28
		314.00	123.10	-12	-36	-11
18	fenbendazole	300.00	268.00*	-11	-22	-27
		300.00	159.05	-11	-35	-14
19	triclabendazole sulfoxide	376.70	358.90*	-14	-19	-22
		377.00	152.00	-11	-37	-26
20	trichlorobenzimidazole sulfone	392.80	242.00*	-10	-40	-23
		392.80	167.10	-15	-41	-15
21	febantel	447.20	383.05*	-11	-20	-17
		447.10	311.90	-13	-24	-13
22	triclabendazole	358.80	343.90*	-11	-27	-21
		358.80	274.00	-11	-38	-17

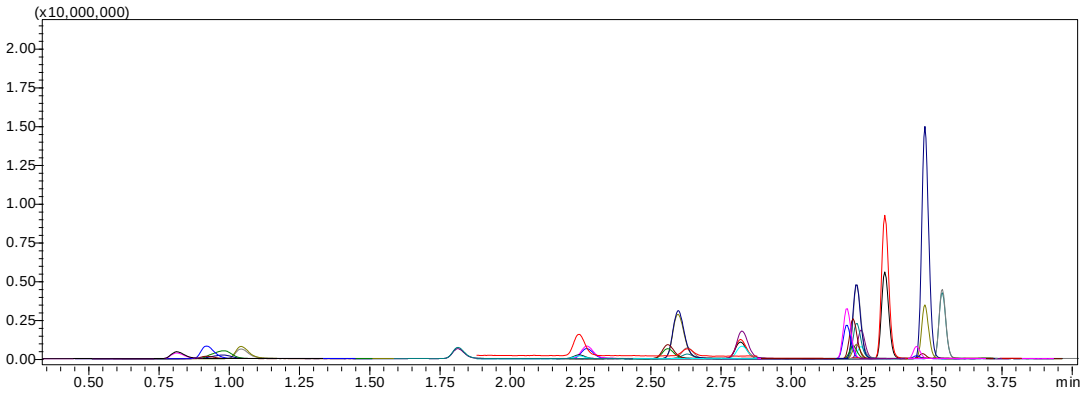

Figure 2. MRM chromatogram of 22 benzimidazole veterinary drug standards in pork and pork liver(concentration: 10 μg/L).

Table 3. List of recovery and %RSD for each veterinary drug.

No.	Compounds	Pork（Spiked amount：10.0 μg/kg）		Pork liver（Spiked amount：10.0 μg/kg）	
		Recovery（%）	RSD(%)	Recovery（%）	RSD(%)
1	5-hydroxythiabenzimidazole	80.93	8.81	78.40	2.28
2	carbendazim	91.29	3.37	94.85	3.80
3	albendazole-2-aminosulfone	79.88	1.90	80.53	0.75
4	thiabendazole	94.06	0.51	99.70	3.30
5	albendazole sulfoxide	94.08	1.41	90.49	2.09
6	hydroxylbenzimidazole	104.08	1.80	101.22	12.19
7	aminomethylimidazole	75.78	0.76	79.14	9.09
8	2-aminoflubendazole	81.29	0.51	77.93	2.20
9	conbendazole	103.83	0.98	100.68	4.87
10	albendazole sulfone	103.68	0.98	100.80	1.85
11	oxfendazole	95.75	0.81	97.61	17.44
12	oxibendazol	96.31	0.55	101.06	19.84
13	fenbendazole Sulfone	106.14	1.93	104.19	4.21
14	mebendazole	103.96	4.38	101.54	4.62
15	albendazole	100.46	3.85	99.47	5.69
16	dibenzimidazole	94.10	3.87	95.11	5.47
17	flubendazole	110.01	5.46	105.58	6.08
18	fenbendazole	103.38	7.16	109.30	14.34
19	triclabendazole sulfoxide	91.68	6.11	105.64	8.34
20	trichlorobenzimidazole sulfone	104.57	8.21	112.29	15.82
21	febantel	104.44	4.90	109.55	8.25
22	triclabendazole	93.96	5.37	103.44	6.17

■ Results and Discussion

Pork and pork liver matrix was spiked with a standard solution of 22 benzimidazole veterinary drugs to a final concentration of 10 μg/kg. QuEChERS method sample preparation was performed according to Figure 1. Figure 2 shows the MRM chromatogram of the 22 benzimidazole veterinary drugs standards in pork and pork liver. Three independent experiment was performed to determine average recovery and %RSD. Results shows all the compounds having a good recovery rate: The spiked recovery rate for pork at a concentration of 10.0 μg/kg was 75.78%-110.01%, with an %RSD of 0.51%-8.81%; for pork liver at the same spiked concentration, the recovery rate was 77.93%-112.29%, with an %RSD of 0.75%-19.84%. Recovery and %RSD for all the compounds are shown in Table 3.

■ Conclusion

This study presents a method for the determination of 22 benzimidazole veterinary drug residues in pork and pork liver. Shimadzu SHIMSEN QuEChERS products were used for clean-up, followed by analysis using Shim-pack Velox SP-C18 column on Shimadzu LCMS-8050. The recovery and reproducibility was determined using 10 μg/kg spiked pork and pork liver blank sample. The results shows a high recovery rate and a good reproducibility. This method is suitable for the simultaneous determination of 22 benzimidazole drug residues in samples such as pork and pork liver.

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