Detection, Monitoring, and Quantitation of Trace Sulfonamides in Pork Muscle Using the Agilent 6410A LC/MS/MS

Application Brief

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Sulfonamides are the one of the oldest groups of veterinary medicines in use today. All sulfonamide drugs are currently included in Annex 1 of the Council Regulation 2377/90. The existing EU maximum residue level (MRL) for all drugs of the sulfonamide group is 100 µg/kg in all food-producing species.

A variety of methods have been used to measure sulfonamide residue in biological materials, including thin-layer chromatography (TLC), high-performance liquid chromatography (HPLC), high-performance capillary electrophoresis (HPCE), gas chromatography (GC), and enzyme-linked immunosorbent assay (ELISA). Now, the LC/MS/MS method is used more widely.

In this study, a multiresidue analysis was performed to simultaneously determine sulfonamides in pork by the Agilent 6410A LC/MS/MS. This multiresidue analysis for sulfonamides can detect different kinds of sulfonamides within one run. Compared with the classic methods, this method can achieve greater sensitivity and be used for screening, confirmation, and quantification.

Highlights

- The pH of the mobile phase played an important role in the LC separation because the retention behaviors of the drugs were dependent on the ionization of the sulfonamides.
- Different kinds of sulfonamide drugs can be analyzed within one run.
- Using the RRLC can get all 14 compounds to elute within 10 minutes.
- High sensitivity easily meets the EU requirements.

Experimental

Sample Preparation

1. **Weigh** – 3-g samples of pork muscle were weighed directly into 50-mL polypropylene centrifuge tubes.
2. **Homogenize** – The samples were homogenized for 3 minutes with 10 mL acidified methanol.
3. **Centrifuge** – The samples were then centrifuged for 10 minutes.
3. **Extract** – 10 mL acidified methanol was extracted, filtered, and injected

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Instrument Settings

LC Conditions

LC	Agilent 1200 Series LC
Column	Agilent ZORBAX SB-C18 (2.1 × 50 mm, 1.8 µm)
Mobile phase	A: 0.1% TFA, B: Acetonitrile 0 min: 5% B 6 min: 23% B 9 min: 23% B 9.01 min: 90% B
Stop time	10 min
Column temperature	30 °C
Injection volume	1 µL
Flow rate	0.3 mL/min

MSD Conditions

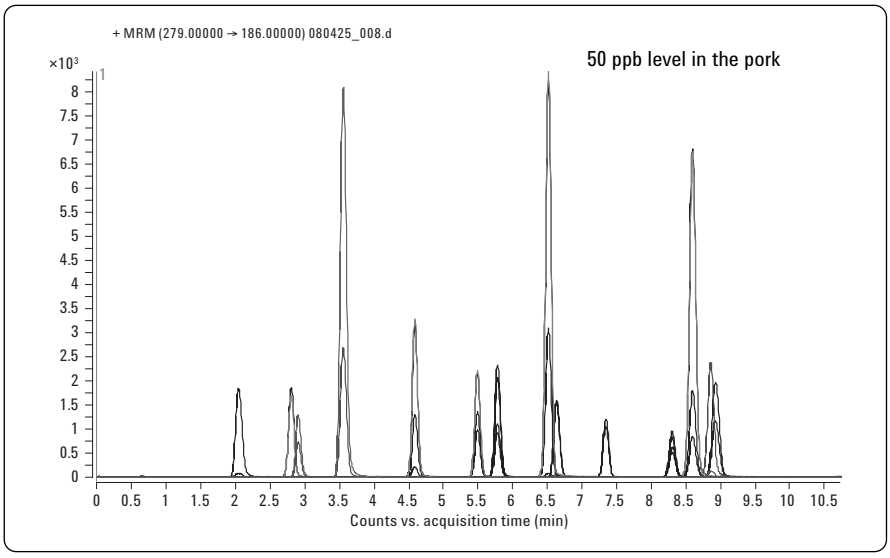
Ionization	ESI (positive)
Scan range	*m/z* 100 to 450
Drying gas	7 L/min at 350 °C
Nebulizer gas	30 psi

MRM setting

Compound	MRM	Frag	CE (V)
Sulfachloropyridazine (SCP)	285–156	100	15
	285–108		20
Sulfadiazine (SD)	251–156	120	10
	251–185		10
Sulfamethazine (SDM)	311–156	140	15
	311–218		15
Sulfamethoxypyridazine (SMP)	281–156	120	10
	281–215		15
Sulfamerazine (SM1)	265–156	120	15
	265–172		15
Sulfamethazin (SM2)	279–156	140	15
	279–204		15
Sulfalmethoxazole (SMZ)	254–156	120	15
	254–147		20
Sulfamonomethoxine (SMM)	281–156	120	10
	281–126		20
Sulfathiazole (ST)	256–156	120	15
	256–107		15
Sulfaquinoxaline (SQX)	301–156	140	15
	301–208		15
Sulfadoxine (SDM)	311–156	140	15
	311–108		20
Sulfaphenazole (SPP)	315–156	140	20
	315–160		20
Sulfaclozine	285–156	100	15
	285–131		20
Sulfafurazole (SIZ)	268–156	120	5
	268–113		10

Results

Good separation and response



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