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Chemical extraction of sulfachloropyridazine from muscle samples

PLOS One

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ABSTRACT

Analytical methodology for the detection of sulfachloropyridazine (SCP) in samples of feathers via LC-MS/MS was implemented based on techniques previously published by other authors:

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- 2- Renew JE, Huang CH. Simultaneous determination of fluoroquinolone, sulfonamide, and trimethoprim antibiotics in wastewater using tandem solid phase extraction and liquid chromatography–electrospray mass spectrometry. J Chromatogr A 2004; 1042: 113-21.
- 3- Shao B, Dong D, Wu y, Hu J, Meng J, Tu X, Xu S. Simultaneous determination of 17 Sulfonamide residues in porcine meat, kidney and liver by solid phase extraction and liquid chromatography-tandem mass spectrometry. Anal Chim Acta 2005; 546:174-81.
- 4- Pang G, Cao YZ, Zhang JJ, Jia GQ, Fan CL, Li XM, Liu YM, Li ZY, Shi YQ. Determination of sulfonamides in honey by liquid chromatography- tandem mass spectrometry. J AOAC Int 2005; 88:1304-11.
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- 6- Bedendo GC, Jardim IC, Carasek E. A simple hollow fiber renewal liquid membrane extraction method for analysis of sulphonamides in honey samples with determination by liquid chromatography-tandem mass spectrometry. J Chromatogr

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A 2010; 1217:6449-54.

7- Yu H, Tao Y, Chen D, Wang Y, Huang L, Peng D, et al. Development of a high-performance liquid chromatography method and a liquid chromatography-tandem mass spectrometry method with the pressurized liquid extraction for the quantification and confirmation of sulphonamides in the foods of animal origin. J Chromatogr B 2011; 879:2653-62.

The method is based mainly on a solid-liquid extraction with organic solvents. The analyte was concentrated using a water bath at 40-50°C under a mild nitrogen flow. For the instrumental analysis, a Symmetry C8 analytical column of 3.5µm and 2.1 x 100mm (Waters®) was fitted in an Agilent 1290 infinity series liquid-chromatograph equipment, coupled to an API 3200 (AB Sciex, Darmstadt, Germany) triple-quadrupole mass-spectrometer. The analytical data was then integrated using the Analyst® version 1.5 software package (SCIEX, Framingham, Massachusetts).

MATERIALS

MATERIALS

- 1.5 mL Eppendorf tubes Contributed by users
- Falcon Tube (50 mL) Fischer Scientific
- Sulfamethazine-(phenyl-13C6) hemihydrate Sigma Aldrich Catalog #32519
- Sulfachloropyridazine Sigma Aldrich Catalog #S9882
- 1 ml syringe Contributed by users Catalog #JD+01T2713
- 10 mL syringe Contributed by users Catalog #JD+10L2125-WEI
- Glass wool Merck Millipore Catalog #1040860250
- Millex Syringe Filter Merck Millipore Catalog #SLGVX13NK

STEP MATERIALS

- Sodium hydroxide Merck Millipore (EMD Millipore) Catalog #1064981000

Hydrochloric acid fuming 37% Merck Millipore (EMD Millipore) Catalog #1003172500
Ethyl Acetate J.T. Baker Catalog #9280-03
Ethyl Acetate J.T. Baker Catalog #9280-03
Formic Acid 98-100% Merck Millipore (EMD Millipore) Catalog #100264
Methanol J.T. Baker Catalog #9093-03
Water Merck Millipore (EMD Millipore) Catalog #1153334000
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Sodium hydroxide Merck Millipore (EMD Millipore) Catalog #1064981000
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SAFETY WARNINGS

Protect hands, eyes and face from organic solvents and reactivities during all the extraction steps with all the necessary materials.

BEFORE START INSTRUCTIONS

Muscle samples must be ground in a food processor before proceeding to extract the analytes to ensure their homogeneity of the samples.

Weigh 5 ± 0.05 g of muscle sample in a 50-mL polypropylen..

1

Fortifie the samples with the internal standard solution Sul...

2

Samples must be rested before extraction.

3

00:15:00

Add 15 mL of Water.

4

Water Merck Millipore (EMD Millipore) Catalog
#1153334000

Stirr the sample on a vortex-mixer.

5

00:10:00

Add 15 μ L of NaOH 1M to the mixture.

6

Sodium hydroxide Merck Millipore (EMD Millipore) Catalog
#1064981000

Stirr the sample again.

7

00:15:00

Sonicate the sample.

8

00:05:00

Adjust the pH of the sample to 7.8 – 8.0 with 10% v/v HCl.

9

Hydrochloric acid fuming 37% Merck Millipore (EMD Millipore) Catalog #1003172500

Add 10 mL of ethyl acetate.

10

Ethyl Acetate J.T. Baker Catalog #9280-03

Stirr on a vortex-mixer.

11

00:10:00

Centrifuge at 2,790 g

12

00:02:00

To dissolve the resulting gel, shook gently the sample by h...

13

00:10:00

Transferre the supernatant to a clean 50 mL polypropylene...

14

Ethyl Acetate J.T. Baker Catalog #9280-03

Evaporate resulting supernatant using a water bath at 40-5..

15

Reconstitute the sample with 500 μ L of a mixture of mobile...

16

Note

Mobile phase A: 0.1% formic acid in methanol (pH 2.9 ± 0.3).

Mobile phase B: 0.1% formic acid in water (pH 2.7 ± 0.2).

Formic Acid 98-100% Merck Millipore (EMD Millipore) Catalog #100264

Methanol J.T. Baker Catalog #9093-03

Water Merck Millipore (EMD Millipore) Catalog #1153334000

Stirr on a vortex-mixer the reconstituted solution.

17 00:05:00

Sonicate the reconstituted solution.

18 00:05:00

Transfer the reconstituted solution into an Eppendorf tube ...

19 00:10:00

Filter the sample through 13 mm millex filters with 0.22 μm ..

20