Chemical extraction of sulfachloropyridazine from muscle samples

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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:

1- Hindle R. A validated atmospheric pressure chemical ionization method for analysing sulphonamides in pork muscle. 2013. Available at http://www.youngin.com/application/0411-0045EN-E.pdf. Accessed October 12, 2017.

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.

5- Stubbings G, Bigwood T. The development and validation of a multi-class liquid chromatography tandem mass spectrometry (LC– MS/MS) procedure for the determination of veterinary drug residues in animal tissue using a QuEChERS (QUick, Easy, CHeap, Effective, Rugged and Safe) approach. Anal Chim Acta 2009; 637:68-78.

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 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).

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Before start

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

Materials

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

Protocol

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

Step 1.

Fortifie the samples with the internal standard solution Sulfamethazine-(phenyl-13C6) hemihydrate (SMZ-13C6) and the possitive controls with the analyte Sulfamethazine

Step 2.

Samples must be rested before extraction.

Step 3.

Add 15 mL of Water.

Step 4.

READAGENTS

Water 1153334000 by Merck Millipore

Stirr the sample on a vortex-mixer.

Step 5.

Add 15 µL of NaOH 1M to the mixture.

Step 6.

READAGENTS

Sodium hydroxide 1064981000 by Merck Millipore

Stirr the sample again.

Step 7.

Sonicate the sample.

Step 8.

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

Step 9.

READAGENTS

Hydrochloric acid fuming 37% 1003172500 by Merck Millipore

Add 10 mL of ethyl acetate.

Step 10.

READAGENTS

Ethyl Acetate 9280-03 by J.T. Baker

Stirr on a vortex-mixer.

Step 11.

Centrifuge at 2,790 g

Step 12.

To dissolve the resulting gel, shook gently the sample by hand and centrifuge it again at 2,790 g

Step 13.

Transferre the supernatant to a clean 50 mL polypropylene tube and repeat the extraction with 10 mL of Ethyl acetate two more times (step 10, 11, 12 and 13 until have 30 mL of extraction solvent in the new tube)

Step 14.

READENTS

Ethyl Acetate 9280-03 by J.T. Baker

Evaporate resulting supernatant using a water bath at 40-50°C under a mild nitrogen flow

Step 15.

Reconstitute the sample with 500 µL of a mixture of mobile phase A and B (15/85).

Step 16.

READENTS

Formic Acid 98-100% 100264 by Merck Millipore

Methanol 9093-03 by J.T. Baker

Water 1153334000 by Merck Millipore

NOTES

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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).

Stirr on a vortex-mixer the reconstituted solution.

Step 17.

Sonicate the reconstituted solution.

Step 18.

Transfer the reconstituted solution into an Eppendorf tube and centrifuged at 17,000 g

Step 19.

Filter the sample through 13 mm millex filters with 0.22 µm polyvinylidene fluoride (PVDF) membranes and transfer into a glass vials.

Step 20.

Warnings

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