Chemical extraction of sulfachloropyridazine from feather 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 on a solid-liquid extraction with organic solvents. The clean up is carried out throught aromatic sulfonic acid (Bakerbond spe™) disposable extraction columns of 6 mL. The analyte is 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

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was then integrated using the Analyst® version 1.5 software package (SCIEX, Framingham, Massachusetts).

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Before start

Feather samples must be cryogenically treated with liquid nitrogen and then ground in an industrial Robot Coupe® R4 table-top cutter food processor (Burgundy, France) to ensure their homogeneity.

Materials

- ✓ 1.5 mL Eppendorf tubes by Contributed by users
- Falcon Tube (50 mL) by Fischer Scientific
- BAKERBOND spe™ Aromatic Sulfonic Acid (C6H5SO3H) Disposable Extraction Columns 7090-29 by J.T. Baker
- 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 2 ± 0.02 g of feather sample in a 50-mL polypropylene tube.
Step 1.

Fortifie each sample with the internal standard solution, Sulfamethazine-(phenyl-13C6) hemihydrate (SMZ-13C6) and the positive controls with the analyte Sulfamethazine .
Step 2.

Samples must be rested before extraction.
Step 3.

Add 40 mL of ethyl acetate.
Step 4.

REAGENTS
Ethyl Acetate 9280-03 by J.T. Baker

Stirr the samples on a vortex-mixer.

Step 5.

Sonicate the sample.

Step 6.

Centrifuge at 1,800 g

Step 7.

Filter the supernatant through glass wool, using a syringe of 10 mL. Pass the filtrate to a fresh 50-mL polypropylene tube.

Step 8.

Concentrate down to 15 mL using a water bath at 40-50 °C under a mild nitrogen flow.

Step 9.

Condition Aromatic sulfonic acid (Bakerbond spe™) disposable extraction columns with 6 mL of hexane and 6 mL of ethyl acetate.

Step 10.

REAGENTS

BAKERBOND spe™ Aromatic Sulfonic Acid (C6H5SO3H) Disposable Extraction Columns 7090-29 by J.T. Baker

Ethyl Acetate 9280-03 by J.T. Baker

n-Hexane 1037014000 by Merck Millipore

FIlter the samples through the columns.

Step 11.

Wash the column with 2 mL of water and 2 mL of methanol.

Step 12.

REAGENTS

Methanol 9093-03 by J.T. Baker

Water 1153334000 by Merck Millipore

Elute the column with 10 mL of a mixture of methanol and ammonia solution (97/3)

Step 13.

REAGENTS

Methanol 9093-03 by J.T. Baker

Ammonia solution 25% 1054322500 by Merck Millipore

Evaporate the samples using a water bath at 40-50°C under a mild nitrogen flow.

Step 14.

Reconstitute the sample with 300 µL of a mixture of mobile phase A and B (15/85)

Step 15.

REAGENTS

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 the reconstituted solution on a vortex-mixer.

Step 16.

Sonicate the reconstituted solution.

Step 17.

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

Step 18.

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

Step 19.

Warnings

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