

Analytical Method for Flumethrin (Animal Products)

1. Analyte

Flumethrin

2. Application

Animal products

3. Instrument

Liquid chromatograph-tandem mass spectrometer (LC-MS/MS)

4. Reagents

Use the reagents listed in Section 3 of the General Rules, except the following.

Reference standard of flumethrin: Contains not less than 98% of flumethrin.

5. Procedure

1) Extraction

i) Except for honey

Add 100 mL of acetone to 10.0 g of the sample, homogenize, and filter with suction. Add 50 mL of acetone to the residue on the filter paper, homogenize, and filter as described above. Combine the resulting filtrates and add acetone to make exactly 200 mL. Take exactly a 2 mL aliquot of the solution, add 30 mL of *n*-hexane, and extract with shaking twice with 30 mL each of acetonitrile saturated with *n*-hexane. Combine the extracts, concentrate at below 40°C, and remove the solvent. Dissolve the residue in 10 mL of acetonitrile and water (7:3) mixed solution.

ii) Honey

Dissolve 10.0 g of the sample in 20 mL of water. Add 100 mL of acetone to the mixture, homogenize, and filter with suction. Add 10 mL of water and 50 mL of acetone to the residue on the filter paper, homogenize, and filter as described above. Combine the resulting filtrates and add acetone to make exactly 200 mL. Take exactly a 4 mL aliquot of the solution, concentrate to approximately 0.5 mL at below 40°C, and add 10 mL of acetonitrile and water (7:3) mixed solution.

2) Clean-up

Inject 5 mL each of acetonitrile and water into an octadecylsilanized silica gel cartridge (500 mg) sequentially and discard each effluent. Transfer the solution obtained in 1) to the cartridge, and discard the effluent. Then, inject 5 mL of acetonitrile and water (9:1) mixed solution, collect the eluate, add acetonitrile and water (9:1) mixed solution to make exactly 5 mL, and use this as the test solution.

6. Calibration curve

Prepare flumethrin standard solutions (in acetonitrile and water [9:1] mixed solution) of several concentrations, inject each solution into LC-MS/MS respectively, and make calibration curves by peak-height or peak-area method. When the test solution is prepared following the above procedure, the concentration of flumethrin in the test solution corresponding to 0.01 mg/kg (0.005 mg/kg for honey) in the sample is 0.0002 mg/L.

7. Quantification

Inject the test solution into LC-MS/MS and calculate the concentration of flumethrin from the calibration curve prepared in 6.

8. Confirmation

Confirm using LC-MS/MS.

9. Measurement conditions

(Example)

Column: Octadecylsilanized silica gel, 2.0 mm inside diameter, 150 mm in length and 5 µm in particle diameter

Column temperature: 40°C

Mobile phase: Linear gradient from a mixed solution of 4 mmol/L ammonium acetate solution and methanol (3:7) to (0:10) in 5 min, and hold for 10 min at (0:10).

Ionization mode: ESI (+)

Major monitoring ions (*m/z*): Precursor ion 527, product ions 267, 239

Injection volume: 10 µL

Expected retention time: 8 min

10. Limit of quantification

0.01 mg/kg (0.005 mg/kg for honey)

11. Explanatory note

1) Outline of analytical method

The method consists of extracting flumethrin from sample with acetone, defatting by acetonitrile/hexane partitioning (omitted for honey), cleaning up with an octadecylsilanized silica gel cartridge, and quantifying and confirming using LC-MS/MS.

2) Notes

- i) When the analytical method for flumethrin using LC-MS/MS was developed, the following monitoring ions were used:
 - for quantitative ions (*m/z*): precursor ion 527, product ion 267
 - for qualitative ions (*m/z*): precursor ion 527, product ion 239
- ii) Food items used to develop the analytical method: cattle muscle, cattle fat, cattle liver, cattle milk, chicken muscle, chicken egg and buckwheat honey.

12. Reference

None

13. Type

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