Original: Japanese
Provisional translation

Analytical Method for Propyrisulfuron (Fishery Products)

1. Analyte

Propyrisulfuron

2. Application

Fishery 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 propyrisulfuron: Contains not less than 95% of propyrisulfuron.

5. Procedure

1) Extraction

Add 70 mL of acetone and *n*-hexane (1:2, v/v), 6 mL of 1 mol/L hydrochloric acid and 8 g of sodium chloride to 10.0 g of the sample and homogenize. Centrifuge at 3,000 rpm for 5 min and collect the organic layer. Add 30 mL of acetone and *n*-hexane (1:2, v/v) to the residue and aqueous layer, homogenize, centrifuge as described above, and collect the organic layer. Combine the resulting organic layers and add acetone and *n*-hexane (1:2, v/v) to make exactly 100 mL. Take exactly a 10 mL aliquot of the solution, concentrate at below 40℃, and remove the solvent. Add 20 mL of *n*-hexane to the residue, extract with shaking twice with 20 mL each of acetonitrile saturated with *n*-hexane, and combine the extracts.

2) Clean-up

i) Ethylenediamine-*N*-propylsilanized silica gel column chromatography

Inject 10 mL each of acetone and acetonitrile into an ethylenediamine-*N*-propylsilanized silica gel cartridge (500 mg) sequentially and discard each effluent. Transfer the solution obtained in 1) to the cartridge, add 5 mL of acetonitrile and 10 mL of acetone sequentially, and discard each effluent. Then, add 10 mL of 1 vol% formic acid-acetone solution, concentrate the eluate at below 40℃, and remove the solvent. Dissolve the residue in 10 mL of acetonitrile and water (3:7, v/v).

ii) Octadecylsilanized silica gel column chromatography

Inject 10 mL each of acetonitrile and water into an octadecylsilanized silica gel cartridge (500 mg) sequentially and discard each effluent. Transfer the solution obtained in i) to the cartridge and discard the effluent. Then, add 10 mL of acetonitrile and water (3:2, v/v), collect the eluate, add acetonitrile and water (3:2, v/v) to make exactly 10 mL, and use this solution as the test solution.

6. Calibration curve

Prepare stock standard solutions by dissolving the propyrisulfuron reference standard in acetone. Dilute the stock standard solutions with acetonitrile and water (3:2, v/v) and prepare standard solutions of several concentrations. Inject each solution into LC-MS/MS and make calibration curves by peak-height or peak-area method. When the test solution is prepared following the above procedure, the concentration of propyrisulfuron in the test solution corresponding to 0.01 mg/kg in the sample results in 0.001 mg/L.

7. Quantification

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

8. Confirmation

Confirm using LC-MS/MS.

9. Measurement conditions

(Example)

Column: Octadecylsilanized silica gel, 2.1 mm inside diameter, 100 mm in length and 3 μm in particle diameter

Column temperature: 40°C

Mobile phase: Initially 0.01 vol% acetic acid and 0.01 vol% acetic acid-acetonitrile solution (7:3, v/v) for 0.5 min, followed by a linear gradient to (1:4, v/v) in 2.5 min, and hold for 6 min.

Ionization mode: ESI (+)

Major monitoring ions (*m/z*): Precursor ion 456, product ions 261, 196

Injection volume: 5 μL

Expected retention time: 6 min

10. Limit of quantification

0.01 mg/kg

11. Explanatory note

1) Outline of analytical method

The method consists of extraction of propyrisulfuron from the sample with acetone and *n*-hexane (1:2, v/v) with a saturated amount of sodium chloride under acidic condition using hydrochloric acid, defatting by acetonitrile/hexane partitioning, clean-up with an ethylenediamine-*N*-propylsilanized silica gel cartridge and an octadecylsilanized silica gel cartridge, and quantification and confirmation using LC-MS/MS.

2) Notes

i) For the centrifuge used during the development of this analytical method, 3,000 rpm corresponds to approximately 1,710 × g.

ii) Columns with an inner diameter of 12-13 mm may also be used for the ethylenediamine *N*-propylsilanized silica gel cartridge (500 mg) and the octadecylsilanized silica gel cartridge

(500 mg).

iii) The matrix may cause fluctuations in the elution conditions of the octadecylsilanized silica gel cartridge.

iv) When the analytical method for propyrisulfuron using LC-MS/MS was developed, the following monitoring ions were used:

for quantitative ions (*m/z*): precursor ion 456, product ion 261

for qualitative ions (*m/z*): precursor ion 456, product ion 196

v) Food items used to develop the analytical method: eel and *Corbicula* (freshwater clam)

12. References

None

13. Type

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