Analytical Method for Penthiopyrad (Agricultural Products)

1. Analyte

Penthiopyrad

2. Instrument

Liquid chromatograph-mass spectrometer (LC-MS)

3. Reagents

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

Reference standard of penthiopyrad: Contains not less than 99% of penthiopyrad.

4. Procedure

1) Extraction

For fruits and vegetables, weigh 20.0 g of sample. For grains, legumes, nuts and seeds, weigh 10.0 g of sample, for tea leaves, weigh 5.00 g of sample, add 20 mL of water and let stand for 30 minutes.

Add 100 mL of acetone, homogenize, and filter with suction. Add 50 mL of acetone to the residue on the filter paper, homogenize, and filter with suction. Combine the resulting filtrates, and add acetone to make exactly 200 mL.

2) Clean-up

i) Graphitized carbon black column chromatography

Add 5 mL of acetone to a graphitized carbon black cartridge (500 mg), and discard the effluent. Transfer the extract (5 mL for fruits and vegetables; 10 mL for grains, legumes, nuts and seeds; 5 mL for tea leaves) obtained in 1) to the cartridge, and elute with 15 mL of acetone. Combine the eluates, add 10 mL of water, and concentrate to about 10 mL at below 40°C.

ii) Octadecylsilanized silica gel column chromatography

Add 5 mL each of acetonitrile and water to an octadecylsilanized silica gel cartridge (1,000 mg) sequentially, and discard the effluent. Transfer the solution obtained in i) to the cartridge, add 10 mL of acetonitrile/water (2:3, v/v), and discard the effluent. Elute with 10 mL of acetonitrile/water (7:3, v/v), add acetonitrile/water (7:3, v/v) to the eluate to make exactly 10 mL, and use this solution as the test solution.

5. Calibration curve

Prepare 0.0005–0.01 mg/L penthiopyrad standard solutions (acetonitrile/water (7:3, v/v)). Inject 10 µL of each standard solution to LC-MS, and make a calibration curve by peak-height or peak-area method.

6. Quantification

Inject 10 µL of the test solution to LC-MS and calculate the concentration of penthiopyrad

from the calibration curve made in **5**.

7. Confirmation

Confirm using LC-MS or LC-MS/MS.

8. Measurement conditions

Example

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

Column temperature: 40°C

Mobile phase: acetonitrile/2 mmol/L ammonium acetate solution (3:2, v/v)

Ionization mode: ESI (−)

Major monitoring ion (*m/z*): 358

Expected retention time: 8 min

9. Limit of quantification

0.01 mg/kg

10. Explanatory note

1) Outline of analytical method

The method consists of extraction of penthiopyrad from sample with acetone, clean-up with a graphitized carbon black cartridge and an octadecylsilanized silica gel cartridge, quantification using LC-MS, and confirmation using LC-MS or LC-MS/MS.

2) Notes

i) It has been confirmed that "Multi-residue Method I for Agricultural Chemicals by LC-MS (Agricultural Products)" was applicable to penthiopyrad in summer orange; accordingly, the multi-residue method can be used under the condition that the performance is verified before use.

ii) When LC-MS (ESI(+)) is used, the major monitoring ion (*m/z*) is 360.

iii) When LC-MS/MS (ESI(−)) is used, the major monitoring ions are as follows:

precursor ion (*m/z*): 358

product ion (*m/z*): 149, 109

11. References

None

12. Type

C