Original: Japanese

Provisional Translation

Analytical Method for Oxadiargyl (Agricultural Products)

1. Analyte

Oxadiargyl

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 oxadiargyl: Contains not less than 95% of oxadiargyl.

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 acetonitrile to the sample, homogenize, and filter with suction. Add 50 mL of acetonitrile to the residue on the filter paper, homogenize, and filter as described above. Combine the resulting filtrates, and add acetonitrile to make exactly 200 mL. Take a 10 mL aliquot of the extract, add 10 mL of water, and concentrate to about 10 mL at below 40°C.

2) Clean-up

i) 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 effluents. Transfer the extract obtained in 1) to the cartridge, add 10 mL of acetonitrile/water (1:1, v/v), and discard the effluent. Elute with 10 mL of acetonitrile/water (7:3, v/v), concentrate the eluate at below 40°C, and remove the solvent. Dissolve the residue in 5 mL of *n*-hexane.

ii) Synthetic magnesium silicate column chromatography

Add 10 mL of *n*-hexane to a synthetic magnesium silicate cartridge (910 mg), and discard the effluent. Transfer the solution obtained in i) to the cartridge, add 5 mL of *n*-hexane and discard the effluent. Elute with 10 mL of ethyl acetate/*n*-hexane (1:9, v/v), concentrate the eluate at below 40°C, and remove the solvent. Dissolve the residue in water/methanol (1:4, v/v) to make exactly 10 mL for fruits and vegetables, exactly 5 mL for grains, legumes, nuts and seeds, and exactly 2.5 mL for tea leaves, and use this solution as the test solution.

5. Calibration curve

Prepare oxadiargyl standard solutions (water/methanol (1:4, v/v)) of several concentrations. Inject each standard solution to LC-MS, and make a calibration curve by peak-height or

peak-area method. When the test solution is prepared following the above procedure, the sample containing 0.01 mg/kg of oxadiargyl gives the test solution of 0.001 mg/L in concentration.

6. Quantification

Inject the test solution to LC-MS, and calculate the concentration of oxadiargyl from the calibration curve made in **5**.

7. Confirmation

Confirm using LC-MS.

8. Measurement conditions

Example

Column: Octadecylsilanized silica gel, 2.1 mm in inside diameter, 150 mm in length and 5 µm in particle diameter

Column temperature: 40°C

Mobile phase: 5 mmol/L ammonium acetate/5 mmol/L ammonium acetate-methanol solution (1:4, v/v)

Ionization mode: ESI (+)

Major monitoring ions (*m/z*): 343, 341

Injection volume: 5 µL

Expected retention time: 6 min

9. Limit of quantification

0.01 mg/kg

10. Explanatory notes

1) Outline of analytical method

The method consists of extraction of oxadiargyl from sample with acetonitrile, clean-up with an octadecylsilanized silica gel cartridge and a synthetic magnesium silicate cartridge, and quantification and confirmation using LC-MS.

2) Notes

i) If the clean-up is insufficient, perform an additional clean-up using a graphitized carbon black cartridge (500 mg).

Outline of procedure: Transfer the eluate obtained from the octadecylsilanized silica gel cartridge to a graphitized carbon black cartridge (rinsed by 10 mL of acetonitrile), elute with 10 mL of acetonitrile, collect the total eluate, concentrate at below 40°C and remove the solvent. Dissolve the residue in 5 mL of *n*-hexane, and then clean-up using a synthetic magnesium silicate column chromatography.

ii) When the analytical method for oxadiargyl using LC-MS was developed, the following monitoring ions were used:

for quantification (*m/z*): 341

for confirmation (*m/z*): 343

11. References

None

12. Type

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