Original: Japanese

Provisional Translation

Analytical Method for Acetamiprid (Agricultural Products)

1. Analyte

Acetamiprid

2. Instruments

Gas chromatograph-flame thermionic detector (GC-FTD)

Gas chromatograph-nitrogen phosphorus detector (GC-NPD)

Gas chromatograph-mass spectrometer (GC-MS)

3. Reagents

Use the reagents listed in Section 3 of the General Rules.

4. Reference standard

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

5. Procedure

1) Extraction

i) Fruits, vegetables and powdered tea

For fruits and vegetables, weigh about 1 kg of sample accurately, add an appropriate quantity of water (if necessary), homogenize, and then take the sample equivalent to 20.0 g. For powered tea, weigh 5.00 g of sample, add 20 mL of water, and let stand for 2 hours.

Add 100 mL of acetone to the sample, homogenize for 3 minutes, and filter through a filter paper, covered with a 1-cm-thick layer of diatomaceous earth, with suction into a vacuum rotary evaporator flask. Collect the residue on the filter paper, add 50 mL of acetone, homogenize for 3 minutes, treat as described above, combine the filtrate in the vacuum rotary evaporator flask, and concentrate to about 30 mL at below 40°C.

Transfer the concentrated filtrate to a 300 mL separating funnel containing 100 mL of 10% sodium chloride solution. Wash the vacuum rotary evaporator flask with 100 mL of ethyl acetate, and transfer the washing to the separating funnel. Shake the separating funnel vigorously for 5 minutes with a shaker, let stand, and transfer the ethyl acetate layer to a 300 mL conical flask. Add 50 mL of ethyl acetate to the aqueous layer, treat as described above, and combine the ethyl acetate layer in the conical flask. Add an appropriate quantity of anhydrous sodium sulfate to the ethyl acetate layer, let stand for 15 minutes with occasional shaking, and filter into a vacuum rotary evaporator flask. Wash the conical flask with 20 mL of ethyl acetate, and wash the residue on the filter paper with the washing. Repeat this step one more time. Combine the washings in the vacuum rotary evaporator flask, and remove ethyl acetate at below 40°C. Add 10 mL of n-hexane to the residue, and remove *n*-hexane at below 40°C. Dissolve the residue in 2 mL of acetone/*n*-hexane (3:7, v/v).

ii) Tea leaves except for powdered tea

Immerse 9.00 g of sample in 540 mL of water at 100°C, let stand for 5 minutes at room temperature, filter, cool, and transfer 360 mL of the filtrate to a 500 mL conical flask. Add 2 mL of saturated lead acetate solution to the filtrate, and let stand for 1 hour at room temperature, filter through a filter paper, covered with a 1-cm-thick layer of diatomaceous earth, with suction, and transfer the filtrate to a 1,000 mL separating funnel. Wash the conical flask with 50 mL of water, wash the residue on the filter paper with the washing, and transfer the washing to the separating funnel. Add 25 g of sodium chloride and 100 mL of ethyl acetate to the separating funnel, shake vigorously for 5 minutes with a shaker, let stand, and transfer the ethyl acetate layer to a 300 mL conical flask. Add 100 mL of ethyl acetate to the aqueous layer, treat as described above, and combine the ethyl acetate layer in the conical flask. Add an appropriate quantity of anhydrous sodium sulfate to the ethyl acetate layer, let stand for 15 minutes with occasional shaking, and filter into a vacuum rotary evaporator flask. Wash the conical flask with 20 mL of ethyl acetate, and wash the residue on the filter paper with the washing. Repeat this step one more time. Combine the washings in the vacuum rotary evaporator flask, and remove ethyl acetate at below 40°C. Add 10 mL of n-hexane to the residue, and remove *n*-hexane at below 40°C. Dissolve the residue in 2 mL of acetone/*n*-hexane (3:7, v/v).

2) Clean-up

Place 5 g of synthetic magnesium silicate for column chromatography suspended in acetone/*n*-hexane (3:7, v/v), and then about 5 g of anhydrous sodium sulfate in a chromatographic tube of 15 mm in inside diameter and 300 mm in length, and let flow out acetone/*n*-hexane (3:7, v/v) to the extent that only a small quantity of acetone/*n*-hexane (3:7, v/v) remains on the top of the column. Transfer the solution obtained in 1) to the column, add 50 mL of acetone/*n*-hexane (3:7, v/v), and discard the effluent. Elute with 150 mL of acetone/*n*-hexane (1:1, v/v), collect the eluate to a vacuum rotary evaporator flask, and remove acetone and *n*-hexane at below 40°C. Dissolve the residue in acetone to make exactly 5 mL, and use this solution as the test solution.

6. Measurement

1) Qualification

Perform the test under the measurement conditions described below. The result shall agree with that obtained using the reference standard.

Measurement conditions

Column: Silicate glass capillary 0.53 mm in inside diameter, 30 m in length coated with 5% phenyl-methyl silicone for gas chromatography 1.5 μm in film thickness

Column temperature: 60°C (2 min) - 20°C/min heating - 160°C (1 min) - 10°C/min heating - 200°C (1 min) - 3°C/min heating - 230°C (1 min) - 5°C/min heating - 260°C (15 min)

Injection port temperature: 260°C

Detector temperature: 260°C

Carrier gas and flow rate: Helium. Adjust the flow rate to elute acetamiprid at about 33 minutes. Optimize the flow rate of air and hydrogen.

2) Quantification

Quantify using peak-height or peak-area method, on the basis of the result obtained using the measurement conditions described in 1).

3) Confirmation

Perform gas chromatography-mass spectrometry using the measurement conditions described in 1). The result shall agree with that obtained using the reference standard. When necessary, quantify using peak-height or peak-area method.

7. Limit of quantification

0.01 mg/kg

8. Explanatory note

None

9. References

None

10. Type

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