Original: Japanese
Provisional Translation

Analytical Method for Metaldehyde (Agricultural Products)

1. Analyte

Metaldehyde

2. Instrument

Gas chromatograph-mass spectrometer (GC-MS)

3. Reagents

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

Graphitized carbon black cartridge (250 mg): Polyethylene tube of 9 mm in inside diameter packed with 250 mg of graphitized carbon, or other cartridge with equal separation characteristics.

Porous diatomaceous earth cartridge (to hold 5 mL of solution): Polyethylene tube of 19–20 mm in inside diameter packed with porous diatomaceous earth prepared for column chromatography which can retain 5 mL of solution, or other cartridge with equal separation characteristics.

Reference standard of metaldehyde: Contains not less than 97% of metaldehyde. Melting point of the standard is 243–246°C (sealed tube method).

4. Procedure

1) Extraction

i) Grains, legumes, nuts and seeds

Add 20 mL of water to 10.0 g of sample and let stand for 2 hours. 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 as described above. Combine the resulting filtrates, and add acetone to make exactly 200 mL. Take 20 mL of aliquot of the extract, add 1 mL of water, and concentrate to about 3 mL at below 40°C.

ii) Fruits and vegetables

Add 100 mL of acetone to 20.0 g of 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 10 mL of aliquot of the extract, add 2 mL of water, and concentrate to about 3 mL at below 40°C.

iii) Tea leaves

Add 20 mL of water to 5.00 g of sample and let stand for 2 hours. 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 as described above. Combine the resulting filtrates, and add acetone to make exactly 200 mL. Take 40 mL of aliquot of the extract, and concentrate to about 4 mL at below 40°C.

2) Clean-up

Add 5 mL of ethyl acetate/*n*-hexane (1:9, v/v) to a graphitized carbon black cartridge (250 mg) and a synthetic magnesium silicate cartridge (900 mg), and discard the effluents. Transfer the extract obtained in 1) to a porous diatomaceous earth cartridge and let stand for 10 minutes. Connect the graphitized carbon black cartridge and the synthetic magnesium silicate cartridge under the porous diatomaceous earth cartridge. Elute with 30 mL of ethyl acetate/*n*-hexane (1:9, v/v), and concentrate the eluate to about 3 mL at below 40°C. Add *n*-hexane to the concentrated solution to make exactly 5 mL, and use this solution as the test solution.

5. Calibration curve

Prepare 0.002–0.04 mg/L metaldehyde standard solutions (*n*-hexane). Inject 1 µL of each standard solution to GC-MS, and make a calibration curve by peak-height or peak-area method.

6. Quantification

Inject 1 µL of the test solution to GC-MS and calculate the concentration of metaldehyde from the calibration curve made in **5**.

7. Confirmation

Confirm using GC-MS or GC-MS/MS.

8. Measurement conditions

GC-MS

Column: 6% cyanopropylphenyl-methyl silicone, 0.25 mm in inside diameter, 30 m in length and 1.4 µm in film thickness

Column temperature: 50°C (1 min) - 30°C/min heating - 260°C (3 min)

Injection port temperature: 150°C

Carrier gas: Helium

Ionization mode: EI (70 eV)

Major monitoring ions (*m/z*): 89, 45

Expected retention time: 6 min

9. Limit of quantification

0.01 mg/kg

10. Explanatory note

1) Outline of analytical method

The method consists of extraction of metaldehyde from sample with acetone, clean-up with a porous diatomaceous earth cartridge, a graphitized carbon black cartridge, and a synthetic magnesium silicate cartridge, and quantification and confirmation using GC-MS.

2) Notes

i) Because metaldehyde volatilizes readily, the volume of eluate after clean-up should leave about 3 mL during the concentration procedure.

ii) A lower injection port temperature improves the sensitivity of the instrument for metaldehyde, therefore, it is recommended that an injection port temperature of 150°C is used.

11. References

None

12. Type

C