

## Analytical Method for Methyl Iodide (Agricultural Products)

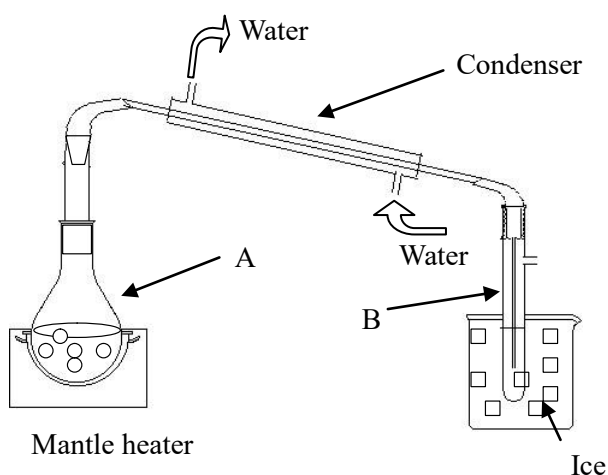
### 1. Analyte

Methyl iodide

### 2. Instruments

Gas chromatograph-electron capture detector (GC-ECD)

Distillation apparatus: Use apparatus shown in the figure below.



A: Distillation flask (500 mL recovery flask)  
B: Receiving vessel (30 mL test tube)

Figure. Schematic of distillation apparatus

### 3. Reagents

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

Reference standard of methyl iodide: Contains not less than 99% of methyl iodide.

### 4. Procedure

#### i) Grains, legumes, nuts, seeds, fruits and vegetables

Freeze sample, chop into the size finely smaller than 5 mm. Weigh 40.0 g of the chopped sample, put into a distillation flask, and add 200 mL of water and 5 mL of *n*-hexane. Add a few drops of defoaming silicone into the distillation flask. Connect the distillation flask and a receiving vessel with a condenser cooled at below 2°C by a low temperature circulator. Immerse the end of the condenser in liquid in the receiving vessel, and cool the receiving vessel in ice water. Before heating, add 4 mL of *n*-hexane to the receiving vessel, and cool in ice water. Heat the distillation flask for about 20 minutes with a mantle heater, and collect *n*-hexane and about 1 mL of water in the receiving vessel. Transfer the *n*-hexane layer collected in the receiving vessel into a 20 mL volumetric flask, add *n*-hexane to make exactly 20 mL, and use this solution as the test solution.

#### ii) Tea leaves

For tea leaves except powdered tea, freeze sample, and chop into the size finely smaller than 5 mm. Weigh 20.0 g of the chopped sample, put into a distillation flask, and add 200 mL of water and 5 mL of *n*-hexane. For powdered tea, weigh 20.0 g of sample, put into a distillation flask, and add 200 mL of water and 5 mL of *n*-hexane. Add a few drops of defoaming silicone to the distillation flask. Connect the distillation flask and a receiving vessel with a condenser cooled at below 2°C by a low temperature circulator. Immerse the end of the condenser in liquid in the receiving vessel, and cool the receiving vessel in ice water. Before heating, add 4 mL of *n*-hexane in the receiving vessel, and cool in ice water. Heat the distillation flask for about 20 minutes with a mantle heater, and collect *n*-hexane and about 1 mL of water in the receiving vessel. Transfer the *n*-hexane layer collected in the receiving vessel into a 10 mL volumetric flask, add *n*-hexane to make exactly 10 mL, and use this solution as the test solution.

#### **5. Calibration curve**

Prepare methyl iodide standard solutions of several concentrations (*n*-hexane). Inject 2 µL of each standard solution to GC-ECD, and make a calibration curve by peak-height or peak-area method. When the test solution is prepared following the above procedure, methyl iodide concentration in the test solution corresponding to the limit of quantification is 0.02 mg/L.

#### **6. Quantification**

Inject 2 µL of the test solution to GC-ECD and calculate the concentration of methyl iodide from the calibration curve made in 5.

#### **7. Confirmation**

Confirm using GC-ECD.

#### **8. Measurement conditions**

Example

Detector: ECD

Column A: 6% cyanopropylphenyl-methyl silicone, 0.32 mm in inside diameter, 30 m in length and 1.8 µm film thickness

Column B: Polyethylene glycol, 0.32 mm in inside diameter, 30 m in length and 0.5 µm in film thickness

Column temperature: 50°C (15 min)

Injection port temperature: 250°C

Detector temperature: 250°C

Carrier gas: Helium

Expected retention time: 4 min (Column A), 3 min (Column B)

#### **9. Limit of quantification**

0.01 mg/kg

## 10. Explanatory note

### 1) Outline of analytical method

The method consists of collection of methyl iodide from sample into a *n*-hexane layer with distillation under hermetic condition, and quantification and confirmation using GC-ECD.

### 2) Notes

- i) Because methyl iodide is highly volatile, keep samples in the frozen state, chop with a kitchen knife etc., and avoid the procedure that may generate heat. Carry out the entire procedure including sampling quickly.
- ii) In the distillation step, adjust heating to collect 5 mL of *n*-hexane and about 1 mL of water from the distillation flask into the receiving vessel for about 20 minutes.
- iii) Because methyl iodide is highly volatile and likely to react with components in samples, start the test procedure immediately after spiking of standard solution, without mixing and leaving the sample, in the recovery test. When acetone is used for preparing standard solutions, the measurement of methyl iodide will be interfered by acetone. Thus, use *n*-hexane for preparing standard solutions.

## 11. References

None

## 12. Type

C