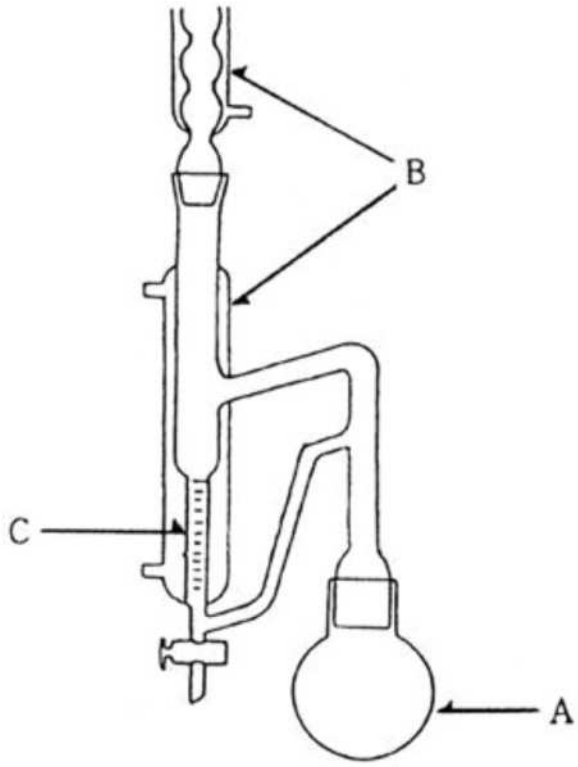
Analytical Method for Ethylene dibromide
(Targeted to Agricultural Products)

The target compound to be determined is ethylene dibromide.

1. Instrument

A gas chromatograph with an electron capture detector (GC-ECD), a gas chromatograph/mass spectrometer (GC/MS or GC/MS/MS) and Dean Stark distillation apparatus The Dean Stark distillation apparatus is roughly as shown in the following figure:

A: Distillation flask
B: Cooling tube
C: Distillation trap



2. Reagents and test solutions

In addition to the reagents and test solutions listed below, use those listed in Section C *Reagents/Test Solutions*, Etc., Part II *Food additives*.

Synthetic magnesium silicate (Florisil) for column chromatography: Heat florisil

(150-250 μm in particle size) at 130°C for 12 hours or longer. Cool down to room temperature in a desiccator.

Silicone for defoaming: Silicone produced for defoaming.

n-Hexane: Use a rotary vacuum evaporator on 300 mL of n-hexane to evaporate until 5 mL is left. For analysis, inject 5 μL of the solution into a GC-ECD. Peaks other than that of n-hexane in the resulting chromatogram should be as high as or lower than the peaks of γ-BHC at 2 x 10- 11g.

Water: Distilled water. If the distilled water is found to include any substance that may interfere with analysis of the target compositional substances from agricultural chemicals, wash with a solvent such as n-hexane before use.

3. Reference standard

Ethylene dibromide: This product contains not less than 99% of ethylene dibromide, and its boiling point is 131.5°C.

4. Procedure

a. Extraction

Homogenize about 1 kg of a test sample and transfer 100 g of the homogenized sample into a 1,000-ml distillation flask, and then add 200 ml of water and 10 ml of n-hexane to the flask. Add a few drops of defoaming silicone and boiling stones to the mixture and attach the flask to the Dean Stark distillation apparatus to be heated and refluxed for one hour. After cooling, remove most of the water in the distillation trap and filter the n-hexane layer into a 10-ml measuring flask through a liquid phase separation filter paper. Wash the trap with a small amount of n-hexane and filter the washings through the filter paper above. Combine the filtrate with the n-hexane layer to make exactly a 10-ml solution.

b. Clean-up

Transfer the solution obtained by the extraction described in 4-a to a 10-ml test tube with a glass stopper. Add approximately 1 g of florisil for column chromatography to the test tube and shake it vigorously, and then leave it to stand at room temperature for about 15 minutes. Collect about 5 ml of supernatant in another test tube with a glass stopper, which is used as the sample solution.

5. Determination

a. Qualitative tests

Perform qualitative tests under the following conditions. Test results obtained must be the same as those obtained for the reference standard.

Testing conditions

Column: A silicate glass capillary column (0.32 mm in inner diameter, length in 30 m) coated with 6% cyanopropylphenyl-methyl silicone for gas chromatography to a thickness of 1.8 μm.

Column temperature: Hold the column temperature at 50°C for two minute, followed by an increase of 5°C every minute until reaching 110°C, after which increase the temperature by 30°C every minute until reaching 260°C, and hold for five minutes.

Inlet temperature: 250°C

Detector: Operate at 300°C

Gas flow rate: Use helium as the carrier gas. Adjust the flow rate so that ethylene dibromide flows out in approximately eight minutes.

b. Quantitative tests

Determine the quantity from the test results obtained under the conditions described in 5-a using either the peak height or peak area method.

c. Confirmation tests

Perform gas chromatography/mass spectrometry under the conditions described in 5-a. Test results obtained must be the same as those obtained for the reference standard. Determine the quantity using either the peak height or peak area method, if required.

6. Limit of Quantification

0.0005mg/kg