Operation procedures for solid-phase extraction (HPLC) (Presep® Agri)

1. Conditioning of solid-phase extraction column: Feed 10 mL of acetonitrile and 20 mL of water (take care not to dry the packing material).
2. Adjust the sample solution to pH 4.0 with 0.1N nitric acid using a pH meter.
3. Feed the sample solution into the solid-phase extraction column. It is appropriate to feed the solution at a constant speed in a range from 10 to 20 mL/min.
4. After feeding the sample solution, feed air for some minutes to remove moisture from the cartridge.
5. Extraction: Extract with 5 mL of acetonitrile.
6. Analyze by HPLC.

Pretreatment for GC/MS analysis (Presep® Agri)

1. Conditioning of solid-phase extraction column: Feed 10 mL of dichloromethane, 5 mL of methanol and 20 mL of water in order (since dichloromethane does not mix with water, use methanol as an intermediate solvent).
2. Injection of test solution: Feed the sample solution into the solid-phase extraction column. It is appropriate to feed the solution at a constant speed in a range from 10 to 20 mL/min.
3. Dehydration: After feeding the sample solution, feed air for 15 minutes to dry the inside of the solid-phase extraction column.
4. Extraction: Extract with 5 mL of dichloromethane*. Fill an empty cartridge with filter with approx. 10 g of sodium sulfate, and feed the extract into the cartridge to remove water from the extract. Clean up sodium sulfate with 5 to 10 mL of dichloromethane, and collect dichloromethane with the extract in a glass test tube graduated in 1 mL increments.
5. Concentration: Add 2 to 3 mL of hexane to the extract, mix them, and concentrate the mixture to 1 mL under N2 gas flow.
6. GC/MS analysis

※ The extract can be obtained by using acetone. When using acetone, use acetone for conditioning in place of methanol.

Precautions for use

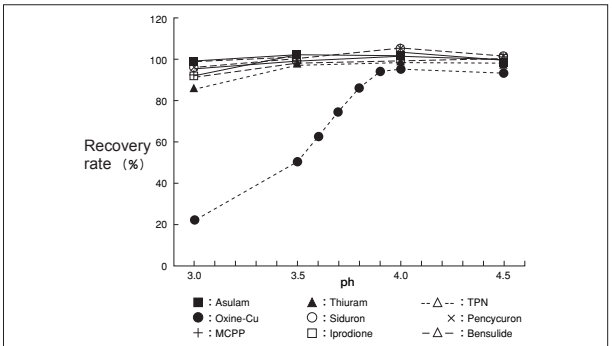
1. The conditioning is effective in removing components remaining in gel, reducing the interference from impurities, swelling the gel to increase the contact area between sample and gel and increasing the concentration efficiency. If the amount of acetonitrile is insufficient, the impurities extracted from the packing material may have an influence on extraction. If the amount of water is insufficient, the retention rate may be reduced under the influence of acetonitrile. If the packing material is dried, the recovery rate may be affected.
2. Changing the pH of test water will change the recovery rates of pesticides (oxine-copper and asulam).
3. Although the flow velocity does not have so significant influence on the recovery rate, it is desirable to feed the liquid at a constant flow velocity to minimize the variation.

If the velocity is constant in the specified range, the recovery rate will not be affected.

4. The dehydration operation can reduce the variation of quantity of water contained in the extract.
5. When injecting acetonitrile, keep the inside of the column processor at normal pressure. After injecting, slightly evacuate the column processor (to 1/10 or less of the test water injecting pressure) to recover the extract. When almost all extract has been recovered, further evacuate it to thoroughly recover the extract. If the flow velocity is too high, the repeatability may be reduced.
6. When treating a sample with a high solid content, filter the sample with an appropriate filter before solid-phase extraction operation.
7. The pesticide standard solution which is the basis of determination of recovery rate by HPLC method shall be diluted and prepared with 90% acetonitrile. If a standard solution prepared with 100% acetonitrile is used as the basis, positive errors in the recovery rates of oxine-copper and MCPP may be caused.

Conditions of solid-phase extraction which affect recovery rate

pH of test water and recovery rate



The pesticide standard solution was added to distilled water whose pH had been adjusted with 0.1N HNO_3, and the obtained solution was used as a sample solution.

Influence of amount of test water (containing 50 µg of each pesticide)

Amount of test water	200 mL	1000 mL
Pesticide	Recovery rate (%)	Recovery rate (%)
Asulam	93.1	93.2
Oxine-copper	102.0	93.7
MCPP	98.8	97.8
Thiuram	100.1	93.9
Siduron	99.1	96.8
Iprodione	99.3	94.7
TPN	100.4	95.0
Pencycuron	100.8	93.0
Bensulide	99.9	94.4

- The table shows the ratios regarding the recovery rate obtained with 500 mL of test water as 100%.
- Presep® Agri was used.