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DETERMINATION OF ACEPHATE AND METHAMIDOPHOS RESIDUES IN SOIL
METHOD RM-12S-2

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## INTRODUCTION

The method of analysis described below determines both acephate and methamidophos in soil. This revision of RM-12S-1 describes a silica Sep Pak® cleanup procedure. Briefly, the method involves extraction with ethyl acetate, Sep Pak® cleanup and measurement by gas chromatography using a flame photometric detector.

## REAGENTS

Acetone: Pesticide Quality

Ethyl Acetate: Pesticide Quality

Ethyl Ether: Anhydrous, Reagent Grade

Methyl Alcohol: Pesticide Quality

Filter Paper: Whatman 2V

Sodium Sulfate: Anhydrous, granular

Acephate Reference Standard

Methamidophos Reference Standard

Silica Sep Pak® - Waters Associates, Inc., Milford, MA, Catalog No. 51900

## EQUIPMENT

Omni-Mixer with adaptor and shafts for use with pint and quart Mason jars or similar top drive blender

B-D Multifit syringe #2152 with Luer-lok, 10cc capacity

Gas Chromatograph equipped with a Melpar flame photometric detector (FPD). We used a Hewlett-Packard 5880 equipped with FPD in the phosphorus mode, an autosampler, and the following parameters:

| | |
|---|---|
| Column: | 1% Reoplex-400 Gas Chrom Q 100-120 mesh, 2' x 1/4" O.D., 2 mm I.D., glass. |
| Flow Rates: | Carrier gas (Nitrogen) -30 ml/min<br>Hydrogen -200 ml/min<br>Air -50 ml/min<br>Oxygen -20 ml/min |

Temperatures Injector - 200°C
Detector - 200°C
Column - Program from an initial temperature of 130°C to a final temperature of 200°C at 15° per minute and hold the final temperature for 2 minutes.

Retention Time: Acephate - 2.6 minutes (Figure 1)
Methamidophos - 1.5 minutes

## EXTRACTION

Transfer a 50 g sample to a quart Mason jar. (For recovery purposes, fortify a control sample with 1.0 ml of an acetone solution of the standards.) Add 100 ml of deionized water and blend on an Omni-Mixer for 5 minutes. Add 300 to 400 g of sodium sulfate with stirring to prevent caking. Quickly add 400 ml of ethyl acetate and blend on an Omni-Mixer for 5 minutes. Allow the solids to settle and decant the extract through filter paper containing sodium sulfate (Caution: Do not decant the water.) Repeat the extraction and filtration steps two more times using 200 ml portions of ethyl acetate. Rinse the filter with 50 ml of ethyl acetate, evaporate the combined filtrates to dryness on a rotary vacuum evaporator. Add 10.0 ml of acetone and proceed with the measurement. If necessary, evaporate 1.0 or 2.0 ml of the acetone extract in a round bottomed flask and proceed with cleanup.

## CLEANUP

Prewash the silica Sep Pak® with two 10 ml portions of ethyl ether. Add 5 ml ethyl ether to the round bottomed flask containing the evaporated acetone extract and mix well. Transfer onto the Sep Pak® and pass the ether through. Add another 5 ml ethyl ether to the round bottomed flask, mix, transfer and pass through. Add 5 ml of 10% (v/v) methyl alcohol in ethyl ether to the flask and mix well. Transfer the solvent onto the Sep Pak® and elute, collecting eluate. Add another 5 ml of 10% methyl alcohol in ethyl ether to the flask, mix, transfer and elute, collecting eluate. Evaporate the combined eluates to dryness and redissolve the sample in 1.0 or 2.0 ml (depending on volume of extract taken for cleanup) of acetone. Proceed with the measurement.

## MEASUREMENT

Transfer the solutions to be measured to vials for use on the automatic liquid sampler. Load the sample tray in the following order: standard, standard, fortified, solvent, check, standard, sample, sample, standard, sample, sample, standard... Set the syringe to deliver 3 µl. The standard vials contain 1.25 µg/ml acephate and 0.5 µg/ml methamidophos in acetone. See Figure 1 for a typical chromatogram.

## LIMIT OF DETECTABILITY

The limit of detectability of the method is 0.02 ppm acephate and 0.01 ppm methamidophos.

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