MRID 42288001

SOP # Meth-32

Revision Original Date 07/91

DETERMINATION OF METHOMYL RESIDUES IN SOIL

References:

Morse Laboratories Method No.: DUP-89AM-001 (Revision 1, 10-19-89), entitled "Determination of Methomyl Residues in Soil."

Principle:

Methomyl is extracted from the sample by first wetting the soil with deionized water and then extracting with ethyl acetate. Quantitation and detection is by use of a high performance liquid chromatograph equipped with a variable wavelength, ultraviolet detector. The quantitation limit is 0.02 ppm.

Note: During all analyses, equivalent apparatus, solvents, glassware, or techniques (such as sample concentration) may be substituted for those specified in the method. In the event an equivalent piece of equipment or an equivalent technique is used, its use will be documented in the study records.

Reagents/Materials:

1. Acetonitrile, water; HPLC grade (Fisher Scientific, Fair Lawn, New Jersey).

2. Ethyl acetate; for organic residue analysis (J.T. Baker Chemical Company, Phillipsburg, New Jersey).

3. Deionized water (100%); generated at Morse Laboratories by Polymetrics deionized water supply system.

4. Filter paper; Qualitative #4 18.5 cm (Whatman International Ltd., Maidstone, England).

5. Acetic acid; Glacial, reagent (VWR Scientific, EM Science, Norwood, Ohio).

6. Methomyl; analytical grade (E.I. du Pont de Nemours and Company, Inc., Wilmington, Delaware).

Prepare:

1. Methomyl analytical standards: stock solutions at 1000 ug/mL in acetonitrile. Dilute for fortification standards in acetonitrile. Dilute for HPLC standards in mobile phase.

2. Acetic acid:acetonitrile:HPLC grade H_2O (1:14:85) by volume

Apparatus/Equipment:

1. 500 mL glass-stoppered Erlenmeyer flasks

2. Plastic funnels

3. 500 mL evaporating flasks

4. Steambath evaporator with vacuum

5. Wrist Action Shaker (Burrell Corporation, Pittsburgh, PA).

6. Buchi rotary evaporator (Brinkmann Instruments, Inc., Westbury, NY).

7. Vortex Mixer (VWR Scientific, Bridgeport, NJ).

8. Usual laboratory glassware and support equipment.

Sample Preparation/Extraction:

See "Preparation of Soil Cores for Methomyl Analysis", July 5, 1991.

1. Weigh a 25.0 g representative sample into a 500 mL Erlenmeyer flask. Fortify as necessary.

2. Thoroughly wet the sample with 25 mL of deionized water. Add 100 mL of ethyl acetate. Stopper the flask securely and shake via a wrist action shaker at high speed for 15 minutes. (Morse Labs shaker SH-03-XX agitation knob set at 2.5).

3. Decant the ethyl acetate (upper layer) through Whatman #4 filter paper into a 500 mL evaporating flask. Do not filter the water into the evaporating flask.

4. Repeat the extraction and filtration steps two more times, each time adding 100 mL of ethyl acetate. STOPPING POINT.

5. Evaporate the combined ethyl acetate extract at 35 ± 2°C using a rotary evaporator to approximately 3 mL.

6. Transfer the concentrated extract to a properly labeled test tube using approximately 3 mL of ethyl acetate for rinses and transfers. STOPPING POINT.

7. Evaporate the sample extract to approximately 0.2 mL on a steambath evaporator. Take the extract to dryness using a gentle stream of nitrogen.

8. Dissolve the sample residue in the test tube by adding 2.5 mL of 85:14:1 water:acetonitrile:acetic acid reagent. Vortex each sample approximately 5 seconds. Store the sample extracts in a freezer prior to HPLC analysis.

HPLC Conditions:

Instrument: Spectra Physics SP8700XR Isocratic Pump.

Detector: Spectra Physics SP8450 UV/VIS detector, at 233 nm.

Column: DuPont Zorbax RX 4.6 mm i.d. x 25 cm.

Mobile Phase: 15:85 acetonitrile:water, flow rate of 0.8 mL/min.

Temperature: 35°C using FIAtron CH-30 HPLC column heater with a FIAtron TC-50 temperature controller (FIAtron Systems, Inc., Oconomowoc, Wisconsin).

Injector Volume: 20 uL

Approximate Retention Time: 12.5 minutes

Note: Historically, the column and conditions stated in the method have been satisfactory for the matrix being analyzed. The specific column packing, mobile phase, column temperature and flow rate listed are typical conditions for this analysis. Specific conditions for each HPLC run will be noted on each chromatogram and will not otherwise be documented.

Determination and Calculation of Results:

Refer to Morse Laboratories, Inc. SOP # CALC-DUP-10

REVIEWED BY [signature]
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