Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Naled

MRID #: 404941-01

Matrix: Soil

Analysis: GC/NPD

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DETERMINATION OF NALED
AND DICHLORVOS IN SOIL SEDIMENT
METHOD RM-3S-1

FILE NO.: 740.01/DIBROM
DATE: OCTOBER 27, 1987

INTRODUCTION

Naled (Phosphoric acid 1,2-dibromo-2,2-dichloroethyl dimethyl ester) is an insecticide which is rapidly converted by soil to dichlorvos (DDVP). This method has been developed to determine naled and dichlorvos in soil sediment. The method consists of extraction with hexane and measurement by gas chromatography with nitrogen/phosphorus detection.

REAGENTS

Acetone - Pesticide grade for rinsing glassware.

DDVP - Analytical standard. Stock solution prepared in hexane.
Reference standards prepared by dilution with hexane.
Fortifying solution (10 µg/ml) prepared by dilution with acetone.

Dipropylphthalate - Eastman Kodak. Prepare a 1% solution in hexane.

Filter Paper - Whatman No. 42, 18.5 cm, or equivalent.

Hexane - Pesticide grade.

Naled - Analytical standard. Stock solution prepared in methanol.
Reference standards prepared by dilution with hexane.
Fortifying solution (10 µg/ml) prepared by dilution with acetone.

Sodium Sulfate - Anhydrous reagent grade.

Saturated sodium sulfate solution.

APPARATUS

Rotary vacuum evaporators with a maximum water bath temperature of 30°C.

Reciprocating shaker.

Gas Chromatograph

Hewlett-Packard 5890 equipped with NP detector and automatic sampler and recording integrator.

Column: 30m x 530µ 50% phenylmethylsilicone wide bore capillary (1 µ thickness)

Column Oven Temperature:

Initial:	125°C with no hold
Rate:	15°C/min
Final:	210°C hold 3 min

Detector Temperature: 275°C
Injection Temperature: 150°C
Carrier Gas: Helium at 20 mL/min
Detector Makeup Gas: Helium at 10 mL/min
Air: 80 mL/min
Injection size: 1-2 µL
Retention Times: DDVP 2.33 min (Fig. 1)
Naled 5.68 min (Fig. 2)

EXTRACTION

The sediment sample is suction filtered and about 10-g sample taken for moisture determination. Transfer a 50-g sample to a pint amber bottle. Two control samples are fortified at this point with 0.5 ml of an acetone solution of either naled or DDVP standard for recovery purposes. Add 20 ml saturated sodium sulfate and swirl to mix. Add 100 ml hexane and shake with a reciprocating shaker for 45 min. to one hour. Decant and measure 50 ml of the hexane extract and pass through anhydrous sodium sulfate in a glass funnel into a 250 ml round bottom flask. Rinse the sodium sulfate with about 20 ml of hexane into the round bottom flask Add 1 ml 1% dipropylphthalate as keeper and evaporate the filtrate just to dryness on a rotary vacuum evaporator using a water bath temperature of no higher than 30°C being careful not to evaporate any longer than is necessary. Add 3.0 ml of hexane and proceed with the measurement.

MEASUREMENT

Transfer approximately 0.5 mL of the solutions to be measured to amber vials for use on the autosampler. Load the sample tray in the following order: DDVP standard, naled standard, DDVP standard, naled standard, sample, DDVP standard, sample, naled standard, etc. alternating samples and standards. The standard vials contain 1.0 µg/mL naled or DDVP in hexane. The integrator is programmed to measure peak area or height.

CALCULATION

$$\text{PPM} = \frac{\text{Peak Area (sample)} \times 1.0\ \mu\text{g/mL} \times 3\ \text{mL} \times 2}{\text{Mean Peak Area of Std.} \times \text{Sample Weight (50 g)}}$$

LIMIT OF DETECTION

The limit of detection is 0.01 ppm for both naled and DDVP.

NOTES

1. Naled is thermally labile and so the injection port temperature is maintained as low as is practically possible [illegible] decomposes to DDVP and under the conditions stated the naled [illegible] give a DDVP peak which has less than 10% of the peak area of the naled peak. This is equivalent to less than 5% decomposition of naled, which is acceptable.

2. The relative standard deviation for the 1.0 µg/mL reference standards injected with the samples should be less than 7%.

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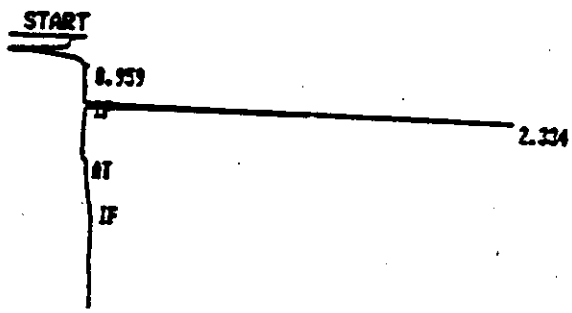
R&D Files

Figure 1

RUN PARAMETERS
ZERO = 10
ATT 2^ = 2
CHT SP = 0.5
AR REJ = 0
THRSH = 2
PK WD = 0.04

TIMETABLE EVENTS
2.150 INTG 0 = 0
5.400 ATT 2^ = 1
5.400 INTG 0 = 0



STOP

RUN# 1412 NOV 28, 1987 13:45:53

DDVP Std SAMPLE# 3

DIBROM

ESTD

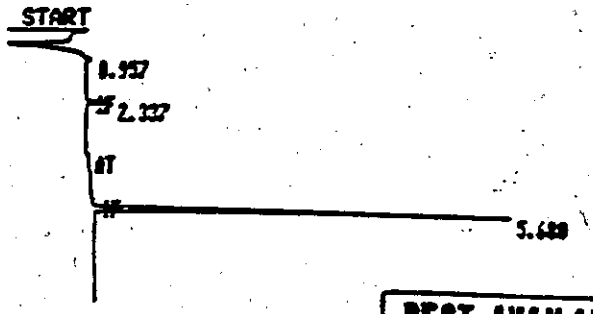
RT	TYPE	AREA	WIDTH	HEIGHT
2.334	BB	83760	.048	54968

1.0 µg/mL DDVP Standard

Figure 2

RUN PARAMETERS
ZERO = 10
ATT 2^ = 2
CHT SP = 0.5
AR REJ = 0
THRSH = 2
PK WD = 0.04

TIMETABLE EVENTS
2.150 INTG 0 = 0
5.400 ATT 2^ = 1
5.400 INTG 0 = 0



RUN# 1413 NOV 28, 1987 13:57:33
Naled Std SAMPLE# 4

DIBROM

ESTD

RT	TYPE	AREA	WIDTH	HEIGHT
2.337	PB	[illegible]	.041	[illegible]
5.688	PV	42893	.349	14443

1.0 µg/mL Naled Standard