CHEVRON CHEMICAL COMPANY
ORTHO AGRICULTURAL CHEMICALS DIVISION
DEVELOPMENT RESEARCH DEPARTMENT
RICHMOND, CALIFORNIA

DETERMINATION OF NALED
AND DDVP IN WATER
METHOD RM-3W-3

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INTRODUCTION

Naled (Phosphoric acid 1,2-dibromo-2,2-dichloroethyl dimethyl ester) is an insecticide which is rapidly converted by soil to dichlorvos (DDVP) in water. This method determines naled and DDVP in water and is a modification of method RM-3W-2. It consists of extraction with methylene chloride 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 dichloromethane.

Hexane - Pesticide grade.

Dichloromethane - 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.

Sodium Chloride.

APPARATUS

Rotary vacuum evaporators (water bath temperature not over 30°C).

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

Transfer a 500 mL aliquot of sample to a 1 liter separatory funnel (at this point in the procedure, fortify a control sample with 0.5 mL of an acetone solution containing 10.0 µg/ml naled and another control sample with 0.5 mL of an acetone solution containing 10.0 µg/ml DDVP for recovery purposes). Add 200 ml saturated sodium sulfate and swirl to mix. Add 50 g of NaCl and shake until dissolved. Extract with 2 x 100 mL methylene chloride (shake for about 30 seconds each time). Filter the extracts through anhydrous sodium sulfate into a 500-mL round bottom flask. Add 1 mL 1% dipropylphthalate as the keeper. Evaporate the extract just to dryness on a rotary evaporator (water bath temperature not over 30°C). Dissolve the residue in 5.0 mL hexane. Add a few grams of anhydrous sodium sulfate.

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

$$\text{PPB} = \frac{\text{Peak Area (sample)} \times 1.0\ \mu\text{g/mL} \times 5\ \text{mL} \times 1000}{\text{Mean Peak Area of Std.} \times \text{Sample Weight (500 g)}}$$

LIMIT OF DETECTION

The limit of detection is 1 ppb 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. Naled decomposes to DDVP and under the conditions stated the naled standard will 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%.

J. A. Duffy

J. A. DUFFY

S. Lee

S. LEE

Reviewed by: G. O. Breault

SL:gt

G. O. Breault
C. E. Crisp
Residue Laboratory (2)
R&M Files
R&D Files