Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Napropamide

MRID #: 450742-01

Matrix: Water

Analysis: GC/NPD

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450742-01

VOLUME 1

Study Title

Napropamide
Determination at ppb Concentrations in Water

Data Requirement

`Guideline Ref.: 160 Series`

Author

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Study Completed On

3-February-00

Performing Laboratory

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Laboratory Project ID

RRC 88-09

Report Number

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EPA
3 27 00

Study Number : RRC 88-09
Report Title : Napropamide
Determination at ppb Concentrations in Water

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Company	Zeneca Inc.	
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Study Number RRC 88-09

Report Title Napropaminde
Determination at ppb Concentrations in Water

Good Laboratory Practice Compliance Statement

I, the undersigned, declare that the objectives laid down for this study were achieved and that the data generated are valid. The report fully and accurately reflects the procedures used and the raw data generated in the above study.

This study does not meet the requirements of 40 CFR Part 160 because the study reports laboratory work not subject to GLP requirements. The laboratory work was completed prior to the publication of the GLP regulations. Although not conducted in strict compliance with current GLP regulations, the study was performed following appropriate EPA guidelines by using accepted laboratory methods and standard data management procedures.

The report is being re-issued to comply with PR Notice 86-5.

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Table of Contents

Page No.

Table of Contents (continued)

DETERMINATION OF NAPROPAMIDE AT PPB CONCENTRATIONS IN WATER

Report No. RRC 88-09

February 12, 1988

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I. SCOPE

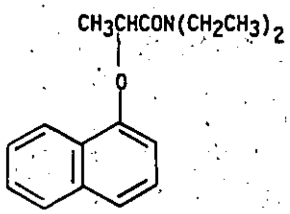
This method is intended for determining napropamide in water. Napropamide is the active ingredient in DEVRINOL®, selective herbicide manufactured by ICI Americas, Inc. The method has been validated for water concentrations of napropamide between 0.1 and 10 µg/L (ppb, w/w).

II. SUMMARY

A sample of napropamide-containing water is extracted directly with toluene. The toluene portion is analyzed for napropamide by gas chromatography with nitrogen-phosphorus detection.

III. INTRODUCTION

Napropamide is N,N-diethyl-2-(1-naphthenyloxy)propionamide (9CI). Napropamide has structure I shown below:



I

IV. APPARATUS AND REAGENTS

A. Apparatus

1. Gas Chromatograph. Hewlett-Packard Model 5880A, equipped with capillary splitless inlet, Hewlett-Packard Model 7673A automatic sampler, nitrogen-phosphorus detector, and electronic integrator or data acquisition system.
2. Injection Port Insert. Splitless insert, 2 mm i.d. by 77 mm, Hewlett-Packard Part No. 18740-80220.

3. Chromatographic Column. Bonded, fused-silica capillary, 12 m x 0.20 mm i.d., 0.33 micron film thickness, cross-linked methyl silicone, HP-1, Hewlett-Packard Part No. 19091-60312.

4. Pipets. Serological, capacity of 10.0 mL.

5. Bottles. Narrow-mouth, 2 oz., with poly-seal caps

6. Syringes. 10, 100, and 500 microliter capacities, Hamilton 701N, 710N, 750N or equivalent.

7. Separatory Funnel. 1000-mL capacity with PTFE stopcock and stopper.

8. Volumetric Flasks. Capacity of 100 mL.

9. Graduated Cylinder. 1000-mL capacity.

B. Reagents

1. Toluene. High purity, tested for the absence of interfering impurities.

2. Acetone. High purity, tested for the absence of interfering impurities.

3. Water. Deionized produced by Millipore Milli-Q or similar system.

4. Napropamide Standard. Available from ICI Americas, Inc., 1200 S. 47th St., Box No. 4023, Richmond, Calif. 94804-0023.

5. Calibration Solutions. Prepare a stock calibration solution by weighing 100.0 mg of napropamide standard into a 100-mL volumetric flask; dilute to volume with toluene. This stock solution contains 1000 µg/mL of napropamide. Calibration solutions of 1.0, 0.10 and 0.01 µg/mL are prepared by dilution of portions of the stock calibration solution with toluene.

6. Fortification Solution. Prepare a stock fortification solution by weighing 100.0 mg of napropamide standard into a 100-mL volumetric flask; dilute to volume with acetone. This stock solution contains 1000 µg/mL of napropamide. A fortification solution of 10 µg/mL is prepared by dilution of a portion of the stock calibration solution with acetone.

V. PROCEDURE

A. Extraction

1. Preparation of Fortified Samples

Fortify 1000-mL portions of water samples, measured with a graduated cylinder, with the acetone solution of napropamide. Add 10.0 µL of the 10 µg/mL acetone solution to produce 0.1 µg/L of napropamide in the fortified solution. Similarly, add 100 µL and 1000 µL of the acetone solution to produce fortifications of 1.0 µg/L and 10 µg/L, respectively.

2. Water Samples - Laboratory Extraction

Measure 1000-mL water sample in a graduated cylinder and transfer the contents into a separatory funnel. Add 10 mL toluene to the funnel containing the sample, stopper the funnel and shake it vigorously for approximately 2 minutes. Allow the phases to separate, drain and discard the aqueous (lower) phase. Drain the toluene layer into a 2 oz. narrow-mouth, bottle containing anhydrous sodium sulfate.

B. Gas Chromatographic Conditions

Follow the manufacturer's instructions for operation of the gas chromatograph and nitrogen-selective detector. Use these additional parameters for the analysis.

Inlet	Splitless insert
Oven initial temperature	100°C
Initial time	0.50 min
Temperature programming rate	30°C per min
Oven final temperature	220°C
Oven final time	10 min
Injector temperature	220°C
Detector temperature	300°C
Carrier gas	Helium
Carrier gas pressure	20 psi (140 kPa)
Carrier flow	1.7 mL through column, 65 mL vented

Injection size	4 µL
Quantitation	Peak area (external standard)

Under the above conditions the elution time of napropamide is 5.59 minutes. See Figure 1 for typical chromatograms.

C. Calibration

The gas chromatograph is calibrated using the napropamide calibration solutions specified in section IV.B.5. Calibrate the instrument using either the 1.00 or 0.10 µg/mL calibration solutions. Check for sensitivity by analyzing the 0.01 µg/mL napropamide calibration solution. Check for response linearity by analyzing the 1.00, and 0.10 µg/mL calibration solutions. Under the conditions specified, the instrument should exhibit a linear response within 10 %, i.e., the ratios of concentration over peak response for each calibration solution should agree to within 10 %.

D. Analysis of Samples

Inject the sample extracts using the same conditions used for calibration. The identity of the napropamide peak in the sample chromatogram is assigned based upon the coincidence of retention times (within 0.03 minutes) with those of the calibration chromatograms. If the response of a peak identified as an analyte exceeds that of the highest calibration solution, dilute the sample extract until its response is within the calibrated range. Reinject the calibration solution after every two to four sample injections and recalibrate as needed. Reinject the calibration solution at completion of the sample analysis.

VI. CALCULATIONS

A. Calibration Factors

Calculate a factor as follows:

$$F\ (\mu g/L) = \frac{\text{Conc. Calib.Solution } (\mu g/mL)}{\text{Extract Ratio}} \times 1000$$

$$\text{where Extract Ratio} = \frac{V_1}{V_2}$$

V_1 = Volume of aqueous sample, mL

V_2 = Volume of toluene, mL

Using the amounts specified above, i.e., V_1 = 1000 mL and V_2 = 10.0 mL, the extract ratio is 100. With various calibration solutions, the following factors were calculated:

Calibration Solution in µg/mL	Factor
1.00	10.0
0.10	1.0
0.01	0.10

B. Analyte in Sample

The concentration of napropamide in the original sample is calculated using external standard method as follows:

$$\text{Napropamide } (\mu g/L) = \text{Factor} \times \frac{\text{Sample Response}}{\text{Calibration Response}}$$

Response = peak area (counts) or peak height (cm)

Note for the above external standard calculations, equal volumes of both the extract and the calibration solutions are injected.

VII. DISCUSSION

A. Recoveries / Accuracy

Fortified water samples were prepared by the addition of acetone solution of 10 µg/mL napropamide to blank water samples. The samples were then analyzed by the method specified above. Recovery data are presented in Table 1. At fortification levels from 0.1 to 10.0 µg/L napropamide, the average recovery was 96 %. Typical chromatograms are shown in Figure 1.

B. Method Precision

The precision of the method depends on variations in sampling, extraction, and instrumental analysis. The variations in extraction and instrumental analysis can be evaluated from the data obtained during recoveries of fortified samples. The coefficient of variation, CV (CV = 100 x std dev / mean), for 9 recoveries was 5 %.

C. Interferences

This method is subject to interferences due to its low detection limit and use of nitrogen-phosphorus detector. Positive results should be confirmed by other means, preferably by GC/MS, MSD, or use of a second capillary column of greater polarity.

VIII. REFERENCES

1. WRC notebooks: 11235-11, 12, 13

M13:88-09

IX. SAFETY PRECAUTIONS

A. Toluene and Acetone

- Flammable
- Vapors harmful
- Avoid contact with skin and clothing
- Use with proper ventilation; avoid breathing vapor

B. Napropamide

- Avoid contact with skin and clothing
- Use with proper ventilation; avoid breathing vapor

TABLE 1

Recoveries of Napropamide from Water

LLV - 0.1 ppb

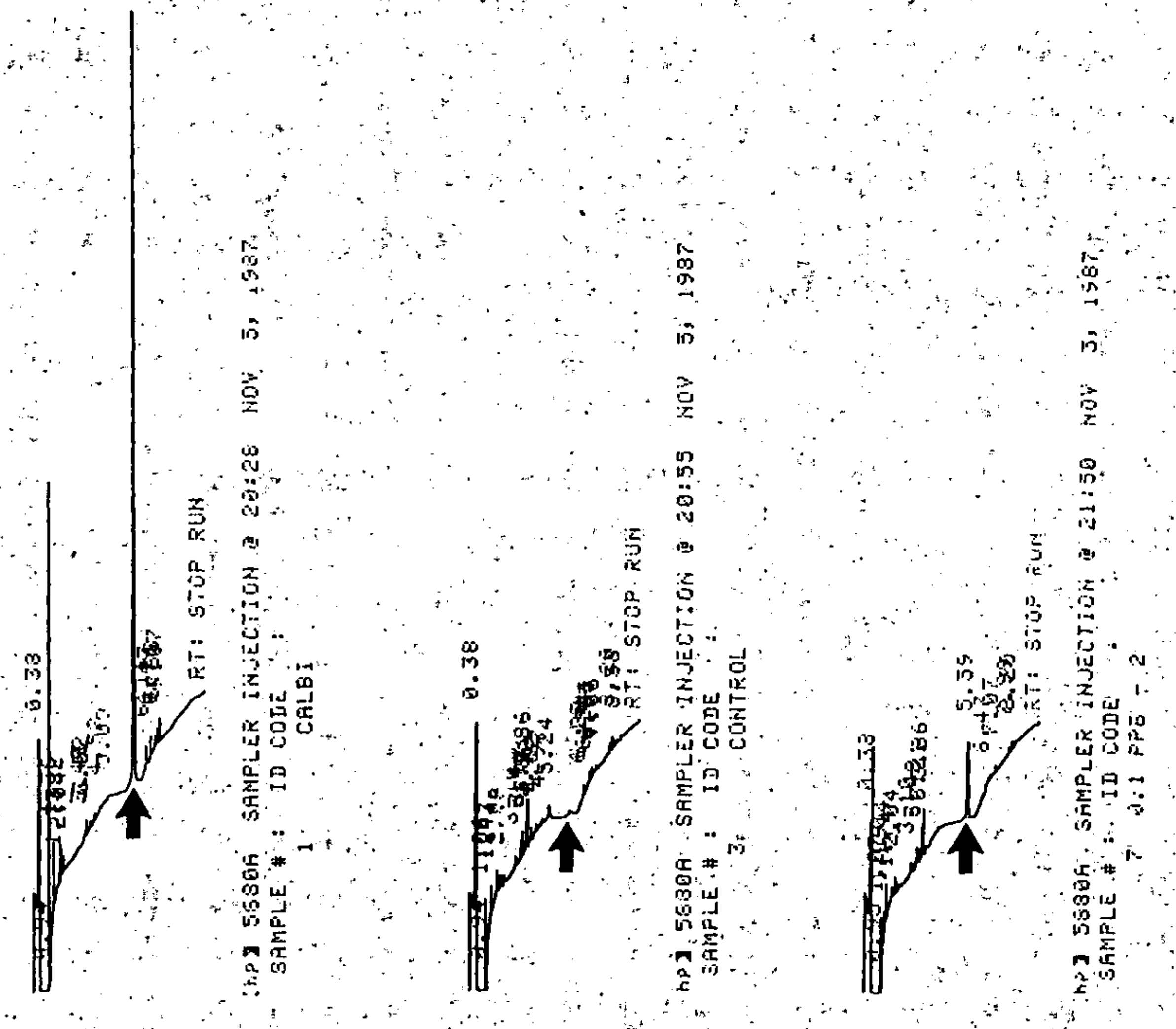
	% Recovery			
Sample No.	Run #1	Run #2	Run #3	Average
Fort @ 0.1 ppb - 1	102	105	109	105 (CV* 9.5)
Fort @ 0.1 ppb - 2	94	93	97	95 (CV 2.2)
Fort @ 0.1 ppb - 3	85	91	86	87 (CV 3.5)
Average	94 (CV 9.1)	96 (CV 7.9)	97 (CV 11.8)	96 (CV 9.4)
Fort @ 1.0 ppb - 1	93	89	99	94 (CV 5.3)
Fort @ 1.0 ppb - 2	93	92	108	98 (CV 9.2)
Fort @ 1.0 ppb - 3	90	95	86	93 (CV 3.1)
Average	92 (CV 1.9)	92 (CV 3.3)	101 (CV 6.6)	95 (CV 2.8)
Fort @ 10.0 ppb - 1	91	99	95	95 (CV 4.2)
Fort @ 10.0 ppb - 2	102	104	91	99 (CV 7.1)
Fort @ 10.0 ppb - 3	97	100	91	97 (CV 4.8)
Average	97 (CV 4.6)	101 (CV 7.1)	92 (CV 2.5)	97 (CV 2.1)

Average CV for 3 inj. over all levels = 5.4

Average CV for 3 rep. at each level = 4.8

*CV (coefficient of variation) = 100 x standard deviation/mean

FIGURE 1. Typical Chromatograms for Napropamide in Water



```
hp 5880A  SAMPLER INJECTION @ 20:28  NOV  5, 1987
 SAMPLE # :  ID CODE  :
        1        CALBI
```

```
hp 5880A  SAMPLER INJECTION @ 20:55  NOV  5, 1987
 SAMPLE # :  ID CODE  :
        3        CONTROL
```

```
hp 5880A  SAMPLER INJECTION @ 21:50  NOV  3, 1987
 SAMPLE # :  ID CODE  :
        7  0.1 PPB - 2
```

A. Calibration Solution
1.0 µg/mL

B. Water Blank Extract

C. Fortified Water Sample
0.1 µg/mL