

Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pesticide Name: Indoxacarb

MRID #: 444773-15

Matrix: Water

Analysis: GC/ECD

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The EPA Good Laboratory Practice (GLP) requirements specified in 40 CFR Part 160 and the Council of the European Communities Concerning the Inclusion of Active Substance in Annex I are not applicable to analytical methods development. However, the methods development presented in this report was done under GLP expect that no protocol was written, no conduct audit was performed, and no QA audit of the study records was done. Analytical procedures, documentation, and archiving of the validation data followed Standard Operating Procedures.

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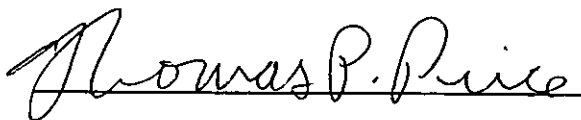


Frank K. Klemens
Section Research Chemist

12 June 97

Date

Company Representative



DuPont Registration Representative

Aug 18, 1997

Date

CERTIFICATION

**ENVIRONMENTAL CHEMISTRY METHOD FOR THE DETERMINATION OF
DPX-KN128/IN-KN127 RESIDUES IN WATER USING GC/ECD**

We, the undersigned, declare that the work described in this report was performed under our supervision, and that this report provides an accurate record of the procedures and results.

Report by:



Frank K. Klemens
Section Research Chemist

12 June 97

Date

Approved by:



Sidney S. Goldberg
Research Supervisor

12 - June - 1997

Date

Date Study Initiated:

May 31, 1994

Date Study Completed:

June 12, 1997

Sponsor:

E. I. du Pont de Nemours and Company
Wilmington, DE 19898
U.S.A

LIST OF ABBREVIATIONS AND SYMBOLS

CAS	<i>Chemical Abstracts Service</i>
CFR	Code of Federal Regulations
EPA	Environmental Protection Agency
GC-ECD	gas chromatography with electron capture detection
GLP	Good Laboratory Practice
LOQ	limit of quantitation
MDL	method detection limit
MSD	mass spectrometry detection
mL	milliliter
ng	nanogram
QA	Quality Assurance
RF	response factor
RSD	relative standard deviation
SD	standard deviation
SNR	signal-to-noise ratio
STD	standard
µg	microgram
µL	microliter

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ENVIRONMENTAL CHEMISTRY METHOD FOR THE DETERMINATION OF DPX-KN128/IN-KN127 RESIDUES IN WATER USING GC/ECD

Frank K. Klemens, Charles Ehlers, and Eric Harris

1.0 SUMMARY

An analytical method is described for determining the residues of DPX-KN128 combined with IN-KN127 in water. DPX-JW062 or DPX-MP062 Experimental Insecticides are formulated containing a mixture of DPX-KN128 and IN-KN127. DPX-JW062 Experimental Insecticide is a formulation containing a racemic (50:50) mixture of DPX-KN128 (insecticidally active enantiomer) and DPX-KN127 (insecticidally inactive enantiomer). DPX-MP062 Experimental Insecticide is a formulation containing a 75% of DPX-KN128 and 25% of DPX-KN127. Residues will be reported as the sum of DPX-KN128 and DPX-KN127 which elute in a single chromatographic peak. Henceforth in this study, DPX-KN128 combined with DPX-KN127 will be referred to as "KN128/KN127".

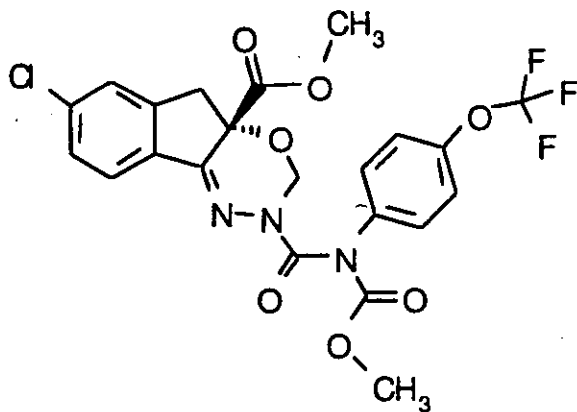
The method involved the analysis of toluene extracts of water samples using gas chromatography (GC) with an electron capture detector (ECD). Waters fortified at concentrations ranging from 0.050 to 2.0 ng/mL (part per billion or ppb) had recoveries ranging from 87-116% with an average recovery (\pm SD) of $100\% \pm 8.1\%$ with an RSD of 8.1 (N=24). The method limit of quantitation (LOQ) is 0.05 ppb with an estimate method detection limit (MDL) of 0.02 ppb. GC/MSD can be used as a confirmatory method using the GC column and conditions specified in this report.

2.0 INTRODUCTION

DPX-MP062 Experimental Insecticide is a postemergence insecticide developed for insect control in cotton, sweet corn, pome fruit, vegetables, grapes, and root crops production. DPX-MP062 Experimental Insecticide is formulated as a suspension concentration or a water-dispersible granule. DPX-MP062 Experimental Insecticide is effective for control of various insects, such as *Heliothis/Helicoverpa* spp., *Spodoptera exigua*, *S. littoralis*, *Pectinophera gossypiella* (pink bollworm), *Earias* spp. (spiny bollworm), *Plutella xylostella* (diamondback moth), *Tricoplusia ni* (cabbage looper), *Spodoptera exigua* (beet armyworm), *Pieris rapae* (imported cabbageworm), *Mamestra brassicae* (cabbage moth), and other insects.

The method involved the analysis of toluene extracts of water samples using gas chromatography (GC) with an electron capture detector (ECD). The method limit of LOQ is 0.05 ppb with an estimate MDL of 0.02 ppb. GC/MSD can be used as a confirmatory method using the GC column and conditions specified in this report.

The structure and chemical name of the active ingredient DPX-KN128 are shown below.



CAS Registry Number: 173584-44-6
 CAS Name (S)-methyl 7-chloro-2,5-dihydro-2-
 [[[(methoxycarbonyl)[4-(trifluoro-
 methoxy)phenyl]amino]carbonyl]=
 indeno[1,2-*e*][1,3,4]oxadiazine-4a(3*H*)-carboxylate

3.0 MATERIALS

3.1 *Equipment (Equivalent equipment and materials may be substituted unless otherwise specified; note any specifications in the following descriptions before making substitutions. Substitutions should only be made if equivalency/suitability has been verified with acceptable control and fortification recovery data.)*

Balance - AE163 balance, 0.0001 to 160.000g (Mettler, Hightstown, NJ).

Sample Bottles - 120-mL precleaned amber glass bottles and caps, #B7316-120 (Baxter, McGaw Hill Park, IL).

Sample Shaker - wrist-action shaker (Burrell, Pittsburgh, PA).

Micropipets - P.C.R. positive displacement pipets, 25 and 250 μ L (Tri-Continent Scientific, Grass Valley, CA).

Gas Chromatograph - Hewlett-Packard 5890 Series II Gas Chromatograph equipped with a 7673A autosampler, a pressure-programmable split/splitless inlet, and an ECD (Hewlett-Packard, Kennett Square, PA).

GC Inlet Liners - deactivated, packed, tapered liner, #5062-3587 (Hewlett-Packard, Kennett Square, PA).

Column - J&W Scientific 30 m x 0.25 mm DB-5 (1- μ film) column, #C4587-89 (Baxter, McGaw Hill Park, IL).

3.2 Reagents & Standards

Toluene - Mallinckrodt nanograde toluene, #8092-4*NY (Baxter, McGaw Hill Park, IL).

Acetonitrile - Burdick and Jackson UV grade acetonitrile, #015-4*DK (Baxter, McGaw Hill Park, IL).

DPX-JW062 - DPX-JW062 analytical standard, Lot 33, purity 98.6%, Stock #1340 (DuPont Agricultural Products, Global Technology Division, E. I. du Pont de Nemours and Company, Wilmington, DE).

3.3 *Safety & Health*

No unusually hazardous materials are used in this method. Each analyst must be acquainted with the potential hazards of the reagents, products, and solvents used in this method before commencing laboratory work. All appropriate material safety data sheets should be read and followed, and proper personal protective equipment should be used.

4.0 METHODS

4.1 *Principle of the Analytical Method*

The method involved the analysis of toluene extracts of water samples using GC with an ECD. 100 ± 5 mL of water are collected and are extracted with 10.0 mL of toluene. The sample is shaken for 10 minutes and the phases are allowed to separate. Exactly 1 mL of the toluene extract is transferred to a GC vial and analyzed by GC/ECD.

4.2 *Analytical Procedures*

4.2.1 *Glassware & Equipment Cleaning Procedure*

The effectiveness of any cleaning procedure used should be demonstrated by preparation and analysis of reagent blanks. In general, all glass- and plasticware should be washed in hot tap water with laboratory grade, non-phosphate detergent, rinsed several times with tap water, rinsed several times with deionized water, rinsed once with acetone, and allowed to fully dry before use. Care should be taken to avoid working with high levels of the analyte being monitored in the same laboratory where samples are being extracted and analyzed.

4.2.2 *Preparation & Stability of Reagents Solutions*

No preparation of any reagents was performed for this study. Reagents were used from a commercially available source. Manufacture recommend stability for these reagents was followed.

4.2.3 Standard Preparation and Stability

Stock Standard Solution

A stock standard solution is prepared by accurately weighing 10.0 mg of KN128/KN127 and dissolving in 50.0-mL acetonitrile to obtain a 200- $\mu\text{g}/\text{mL}$ solution. This stock standard solution is stable for several months when stored at 4°C.

Intermediate Standard Solutions

The stock standard solution is diluted serially (1.0 mL in 10.0-mL total) with acetonitrile to obtain 20.0 $\mu\text{g}/\text{mL}$, 2.0 $\mu\text{g}/\text{mL}$, and 0.20- $\mu\text{g}/\text{mL}$ intermediate standard solutions. This standard solution is stable for several months when stored at 4°C.

GC Calibration Standards

Standards for GC calibration are prepared using the intermediate standard solutions as described in Table 1. Aliquots of the intermediate standard solutions are dispensed using the micropipets. Standards for this study were stored in amber glassware in a freezer and are stable for several months, but standards can be stored in clear glassware in a freezer if needed.

KN128/KN127 concentration in intermediate standard solution ($\mu\text{g}/\text{mL}$)	Volume of intermediate standard solution (μL) diluted to 10.0 mL with toluene	KN128/KN127 concentration in GC standard (ng/mL)
0.2	25.0	0.50
2.0	12.5	2.5
2.0	50.0	10.0
20.0	25.0	50.0
20.0	100.0	200.0

4.2.4 Source of Samples

Water was sampled from four different sources and analyzed using this method (Bradenton, FL, Donna, TX, Elkhart, MD, and Madera, CA). There were no interferences in these water matrices.

4.2.5 Storage & Preparation of Samples

Sample Collection

100 mL of water samples are to be collected in 120-mL amber or clear glass bottles marked with a 100-mL graduation. The accuracy on collection should be approximately 100 ± 5 mL. Each sample bottle becomes an individual sample and cannot be split. A sufficient number of sample bottles must be prepared to account for replicate, retain, and fortification samples required by the protocol.

Storage

Water samples must be frozen from the time of sampling until extraction and analysis.

4.2.6 Sample Fortification Procedure

For each set of samples, spike two samples (controls, if available) with intermediate standard solutions to obtain concentrations approximately corresponding to the MDL and five times the MDL. Extract the spiked samples as specified above (Item 2).

For example, if the MDL is 0.020 ppb, two samples would be spiked, one at 0.020 and one at 0.10 ppb, with KN128/KN127. The 0.020-ppb sample is prepared by adding 10 μ L of the 0.20- μ g/mL intermediate standard solution (to obtain 0.002 μ g in 100 mL which is equivalent to 0.020 ppb), and the 0.10-ppb sample is prepared by adding 50 μ L of the 0.20- μ g/mL intermediate standard solution.

4.2.7 Analyte Extraction Procedure

The exterior of each sample bottle should be wiped clean and dry, and the total weight determined (including the bottle and cap). After recording the weight, add 10.0 mL of toluene to each sample, allow the samples to thaw, and shake for 10 minutes on a wrist-action shaker (samples oriented horizontally). After shaking, allow the phases to separate sufficiently to sample exactly 1 mL of the toluene extract. Transfer the aliquot of toluene to a GC vial. Empty the sample bottles, rinse with water and dry. Weigh the bottle and cap after drying. Calculate and record the sample weight - i.e., weight of bottle containing the sample - weight of the dry bottle and cap.

4.2.8 Analyte Purification Procedure

No further purification of the water sample was needed.

4.3 Instrumentation

4.3.1 GC Setup and Calibration

Set up the GC as specified in Table 2. Refer to manufacturer recommendations for setting detector gas flows (N_2 make-up gas). If pressure programming is not available, use a constant pressure of 30 psi for He carrier gas. Analyze standards to

determine the retention time of KN128/KN127 and to confirm the linearity of the GC method. See Table 1 for information on standard preparation.

Table 2. GC Conditions for KN128/KN127 Analysis	
injection volume = 5 μ L, splitless (inlet purge time = 2 min)	
injector temperature = 250°C	
detector temperature = 340°C	
signal range = 0	
Temperature program:	
initial temperature = 150°C	initial time = 1 min
heating rate = 15°C/min	
final temperature A = 330°C	initial time A = 9 min
Pressure program: (He carrier gas)	
initial pressure = 70 psi	initial time = 0.3 min
pressure program rate = 99 psi/min	
final pressure = 30 psi	final time = 40 min

4.3.2 Sample Analysis

The following sequence should be used when analyzing samples.

MDL standard, sample fortified at the MDL, sample 1, sample 2, sample 3, sample fortified at 5 x MDL, 5 x MDL standard, MDL standard, sample 4, etc.

MDL standard is a solution of KN128/KN127 in toluene with a concentration corresponding to the MDL. For example, if the MDL is 0.020 ppb, the MDL standard would be 0.2-ng/mL KN128/KN127.

The concentration of the MDL standard (ng/mL) is determined as follows:

$$C_{\text{MDLSTD}} = \text{MDL}(\text{ppb}) * 10$$

where 10 is the concentration factor for the extraction step.

Analyze the GC data to ensure proper identification and integration of the KN128/KN127 peak. Calculate the KN128/KN127 concentrations using the procedure described in the next section. Water samples with KN128/KN127 concentrations which are higher than the highest calibration standard should be diluted appropriately (i.e., to obtain a concentration lower than the highest standard) and reanalyzed.

4.4 Calculations

4.4.1 Response Factor (RF)

Quantitation of KN128/KN127 in the water samples is done using the average of the response factors of a standard analyzed before and after each set of three samples.

The response factor for KN128/KN127 is defined as,

$$\text{Response factor} = A_{\text{KN128/KN127}}/C_{\text{KN128/KN127}}$$

- $A_{\text{KN128/KN127}}$ = GC peak area for KN128/KN127.
- $C_{\text{KN128/KN127}}$ = concentration (ng/mL) of KN128/KN127 in the standard.

4.4.2 Quantitation of KN128/KN127 in Water

The following equation is used to calculate the concentration of KN128/KN127 in water samples,

$$\text{KN128/KN127 (ppb)} = (A_w/\text{Average RF})/10$$

- A_w = GC peak area for KN128/KN127 in the sample extract.
- Average RF [area/(ng/mL)] = average of the response factors of the standards analyzed before and after the sample (i.e., the MDL standard and 5X MDL standard).
- The divisor of the 10 accounts for the 10-fold concentration in the extraction procedure.

4.4.3 Recovery of KN128/KN127 from Fortified Samples

The following equation is used to calculate the % of KN128/KN127 recovered from fortified samples.

$$\% \text{ recovered} = [(A_F - A_S)/A_{\text{STD}}] \times 100$$

- A_F = KN128/KN127 peak area for fortified sample.
- A_S = KN128/KN127 peak area for unfortified sample.
- A_{STD} = KN128/KN127 peak area for the standard concentration corresponding to the spike level.

5.0 RESULTS AND DISCUSSION

5.1 Method Validation Results

5.1.1 Detector Response

A linear calibration curve for KN128/KN127 is shown in Figure 1. The concentration range was 0.5- to 200-ng/mL KN128/KN127 in toluene which corresponds to 0.05 to 2.0 ppb in water.

5.1.2 Controls

Water was sampled from four different sources and analyzed using this method. There were no interferences in the control water matrices.

5.1.3 Recoveries

The method was validated in the concentration range of 0.050 to 2.0 ppb by analyzing water from Bradenton, FL, Donna, TX, Elkhart, MD, and Madera, CA, fortified at 0.050, 0.30, and 2.0 ppb with KN128/KN127. The results of those analyses are shown in Table 3. The average recovery (\pm SD) for the four water types fortified at three concentrations was $100\% \pm 8.1$ with an RSD of 8.1 (N=24).

KN128/KN127 (ppb)	% Recovery			
	Bradenton, FL	Donna, TX	Elkhart, MD	Madera, CA
0.050	96	88	108	90
	94	92	116	96
0.30	87	96	103	97
	91	99	108	97
2.0	112	104	108	102
	98	109	109	109
average	96	98	109	99
standard deviation	8.6	7.7	4.2	6.4
RSD	8.9	7.9	3.8	6.5
Average for all samples	100			
Standard deviation for all samples	8.0			
RSD for all Samples	8.1			

5.1.4 Extraction Efficiency

Not applicable to this matrix.

5.1.5 Limit of Quantitation and Method Detection Limit

The LOQ was established by fortifying water samples with KN128/KN127 which resulted in a fortification level of 0.05 ng/mL. As can be seen in Table 3, the eight samples resulted in a range of recoveries of 88-116%. The average (\pm SD) for the

recoveries at this level was $98\% \pm 9.6\%$ with an RSD of 9.9%. The signal-to-noise ratio (SNR) at the 0.05 ng/mL level fell between 5-20. A LOQ of 0.05 ng/mL is justified based on the SNR and the recovery data at this level.

The MDL was determined using the method described below. Any water not containing KN128/KN127 or interferences should be suitable for the MDL determination.

The MDL was calculated from the standard deviation of the measured concentration of two aliquots each of Bradenton, FL, Donna, TX, Elkhart, MD, and Madera, CA, water samples fortified at 0.020 ppb with KN128/KN127. The data used to calculate the MDL are shown in Table 5. The MDL was determined as follows:

$$\text{MDL} = t(N-1, 1-\alpha=0.99) \times S_c$$

- t is the student t value for a one-tailed test at the 99% confidence level with $N-1$ degrees of freedom ($t = 3.0$ for eight replicates).
- S_c is the standard deviation of the measured concentrations for N samples.

For the calculated MDL to be valid, the proposed MDL (0.020 ppb) must be within a factor of three of the estimated MDL which was 0.020 ng/mL. The calculated MDL was 0.0081 ng/mL which meets this criterion.

Water Source	KN128/KN127 (ng/mL)
Bradenton, FL	0.020
	0.020
Donna, TX	0.016
	0.019
Elkhart, MD	0.021
	0.022
Madera, CA	0.026
	0.022
Average	0.021
Standard Deviation	0.0029
RSD	14

5.2

Timing

At least eight (8) to ten (10) samples can be easily extracted within 4 hours followed by an unattended overnight analysis.

5.3 *Modifications or Special Precautions*

The half-life for KN128/KN127 in water samples at room temperature is typically less than one week. It is important to extract samples within several hours of thawing.

The use of plasticware for the storage of toluene or for sample extractions is discouraged due to the extraction of plasticizers and additives which may interfere with KN128/KN127 in GC analyses.

5.4 *Method Ruggedness*

5.4.1 *KN128/KN127 Storage Stability in Water*

Samples of Bradenton, FL, Donna, TX, and Madera, CA, water fortified to 0.10 ng/mL with KN128/KN127 were analyzed after 30 days of storage in a freezer at approximately -10°C. The results of those analyses are shown in Table 4. The average recovery (\pm SD) was 99% \pm 7.2% with an RSD of 7.3%. These data indicate that KN128/KN127 are stable in water for at least 30 days when stored at approximately -10°C.

Water Source	% Recovery
Bradenton, FL	104
	105
	104
Donna, TX	93
	87
	90
Madera, CA	102
	99
	107

5.4.2 *Specificity/Potential Interferences*

Water was sampled from four different sources and analyzed using this method. Chromatograms for the four water samples are shown in Figures 2-5. There were no interferences in these water matrices. Chromatograms for a 0.5-ng/mL and a 10.0-ng/mL KN128/KN127-standard are shown in Figures 6 and 7. Chromatograms for Bradenton, FL, water samples fortified at 0.050 and 2.0 ppb with KN128/KN127 are shown in Figures 8 and 9.

Standards of bromacil and diuron, two potential interferences, were also analyzed. Both compounds eluted more than 9 minutes before KN128/KN127.

5.4.3 Confirmatory Method

GC with mass spectrometric detection can be used as a confirmatory method using the GC column and conditions specified in this method.

6.0 CONCLUSIONS

A toluene extraction/GC-ECD analysis method has been developed for the determination of KN128/KN127 in water. The method is suitable for measuring KN128/KN127 in water at levels exceeding 0.050 ppb. No interferences were detected by this method in water samples from four different sources. Waters fortified at concentrations ranging from 0.050 to 2.0 ng/mL (part per billion or ppb) had recoveries ranging from 87-116% with an average recovery (\pm SD) of 100% \pm 8.1% with an RSD of 8.1 (N=24). The method limit of quantitation (LOQ) is 0.05 ppb with an estimate method detection limit (MDL) of 0.02 ppb. GC/MSD can be used as a confirmatory method using the GC column and conditions specified in this report. The storage stability of KN128/KN127 in water was confirmed by recoveries of greater than 95% from three different types of water analyzed 30 days after fortifying to 0.10 ppb with KN128/KN127.

7.0 RETENTION OF RECORDS

The raw data for this study and the final report are retained in the GLP Archive located at either of the following locations:

Conoco Inc.
DuPont Corporate Center for Analytical Sciences
Ponca City Laboratories
1000 S. Pine
Ponca City, OK 74603-1267

E. I. du Pont de Nemours and Company
DuPont Agricultural Products
Global Technology Division
Experimental Station
Wilmington, DE 19880-0402

FIGURE 1
CALIBRATION CURVE FOR DPX-KN128/IN-KN127

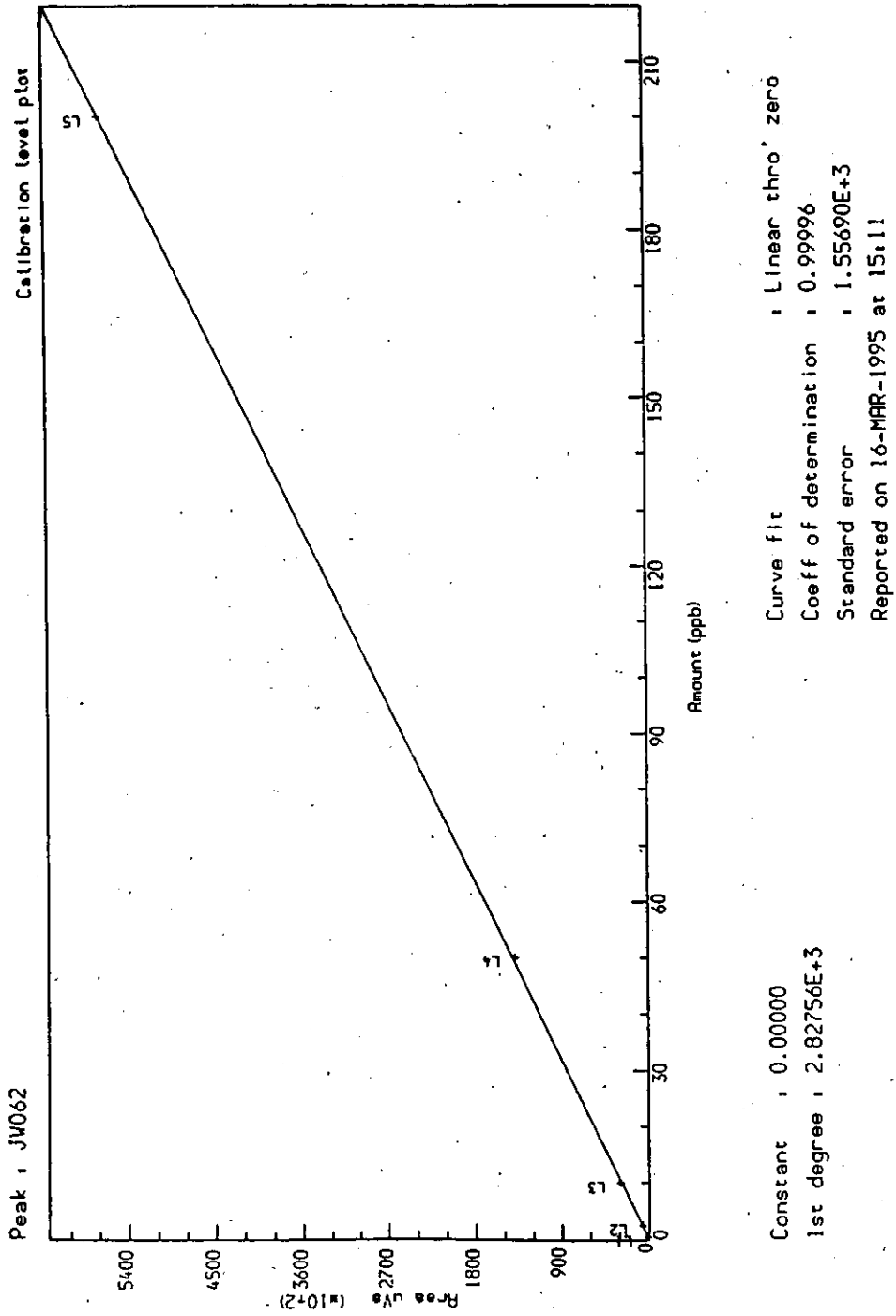


FIGURE 2
GAS CHROMATOGRAM FOR UNFORTIFIED BRADENTON, FL, WATER

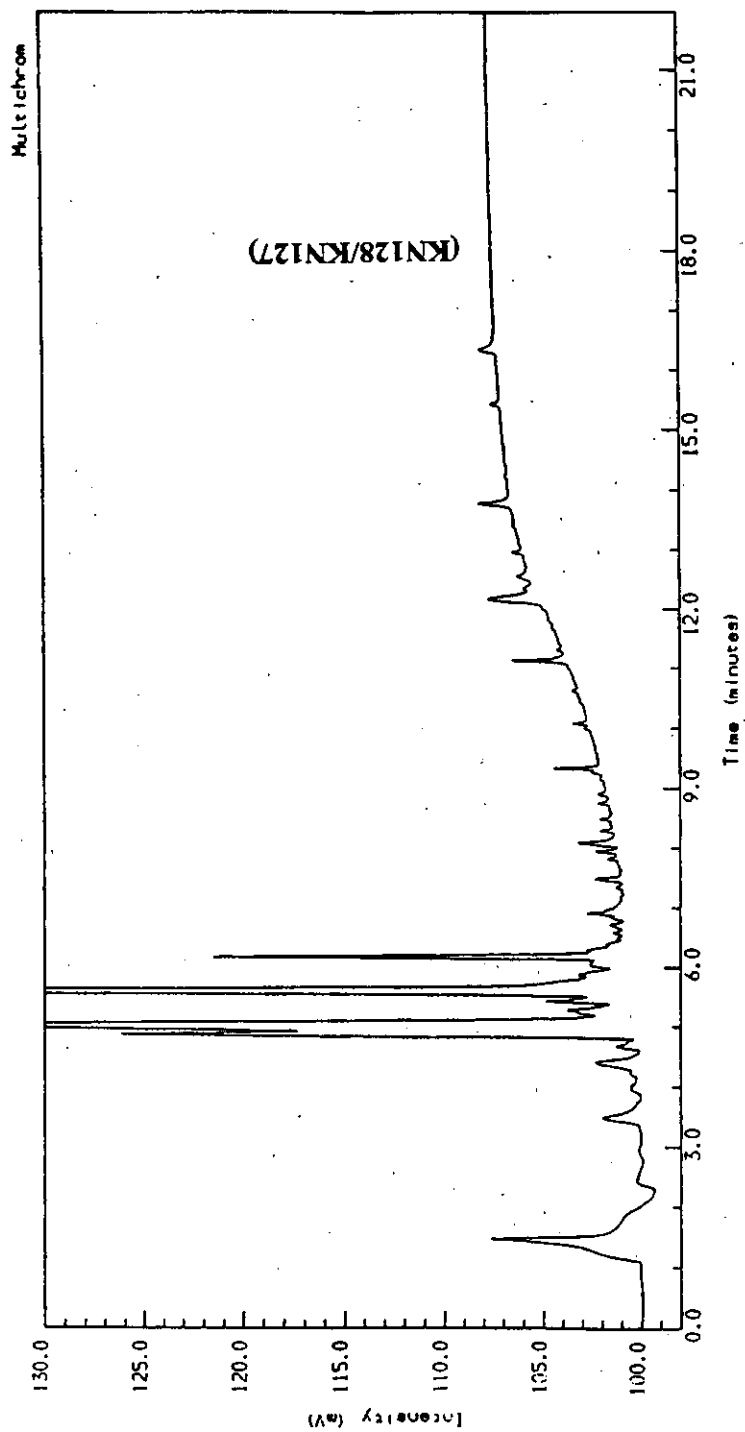


FIGURE 3
GAS CHROMATOGRAM FOR UNFORTIFIED DONNA, TX, WATER

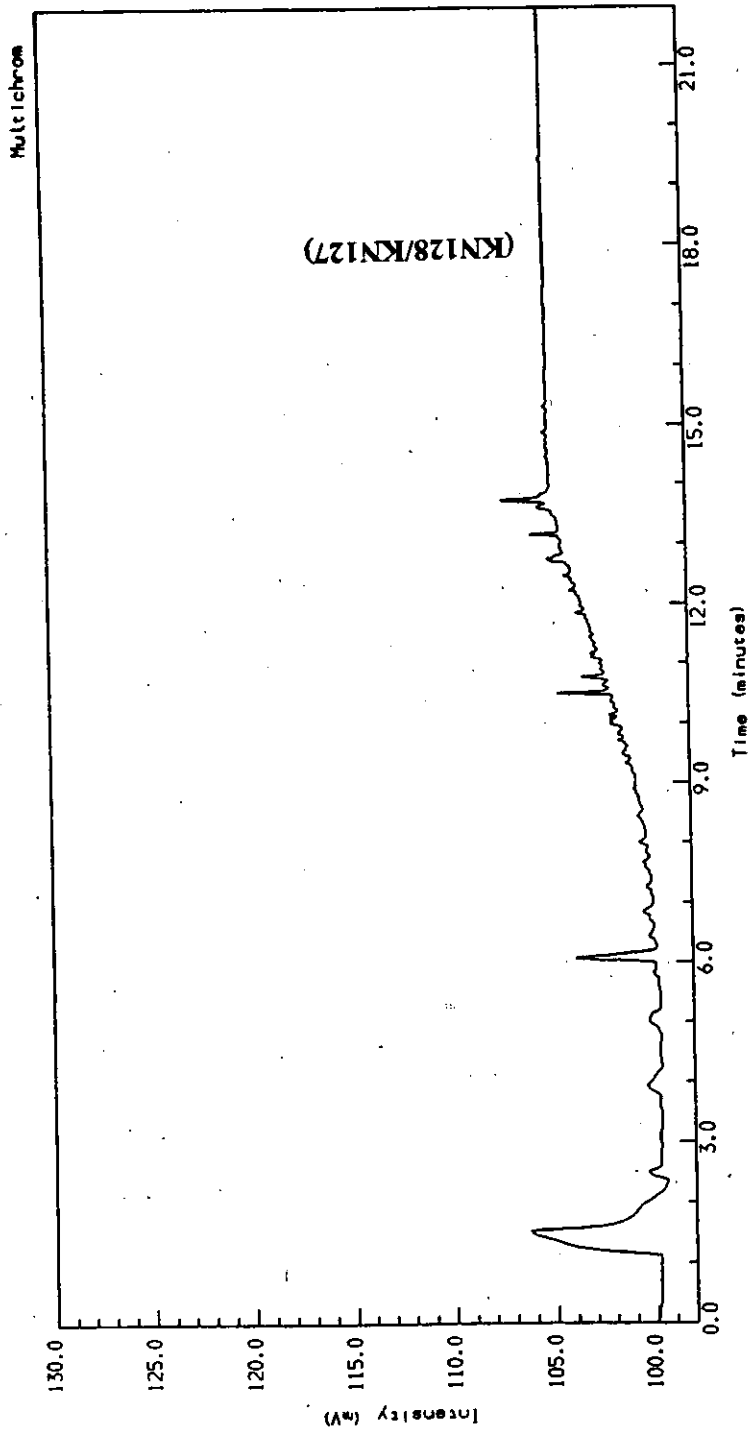


FIGURE 4
GAS CHROMATOGRAM FOR UNFORTIFIED ELKHART, MD, WATER

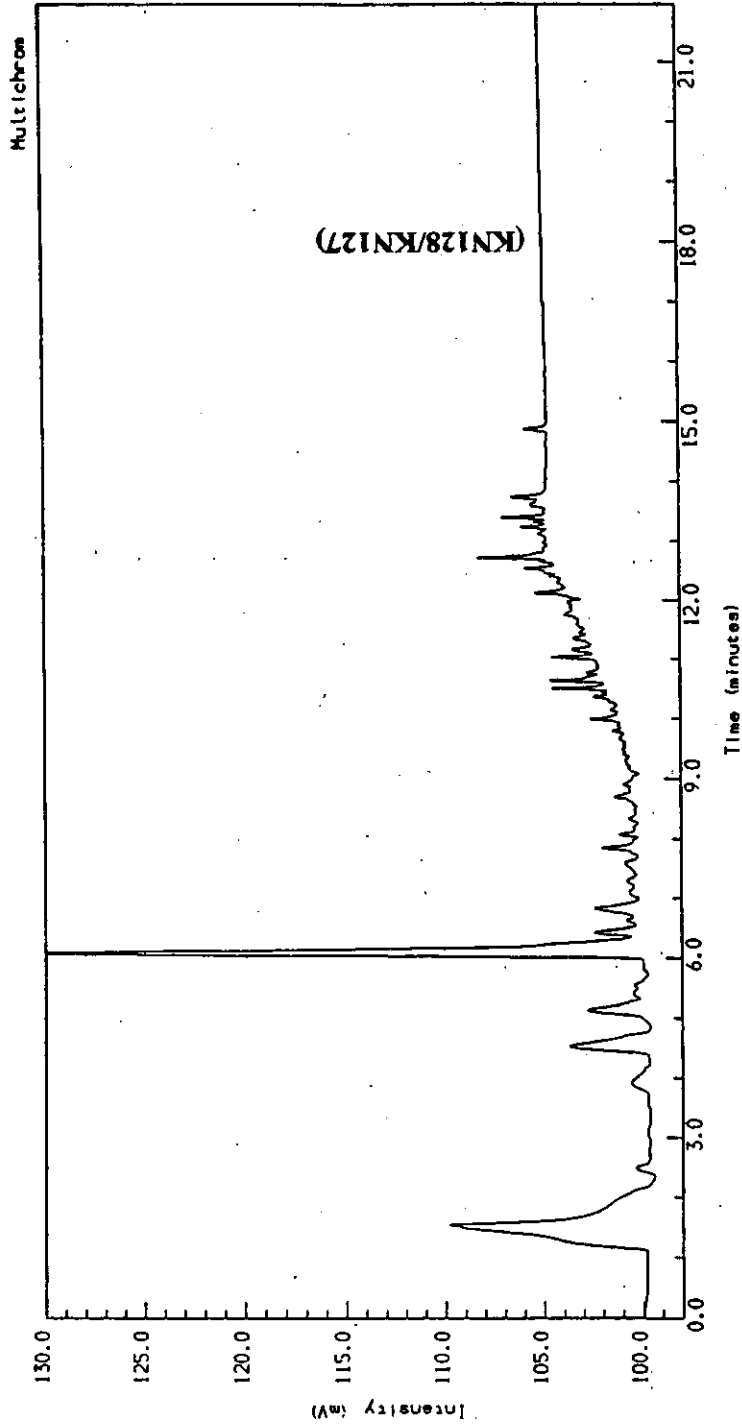


FIGURE 5.
GAS CHROMATOGRAM FOR UNFORTIFIED MADERA, CA, WATER

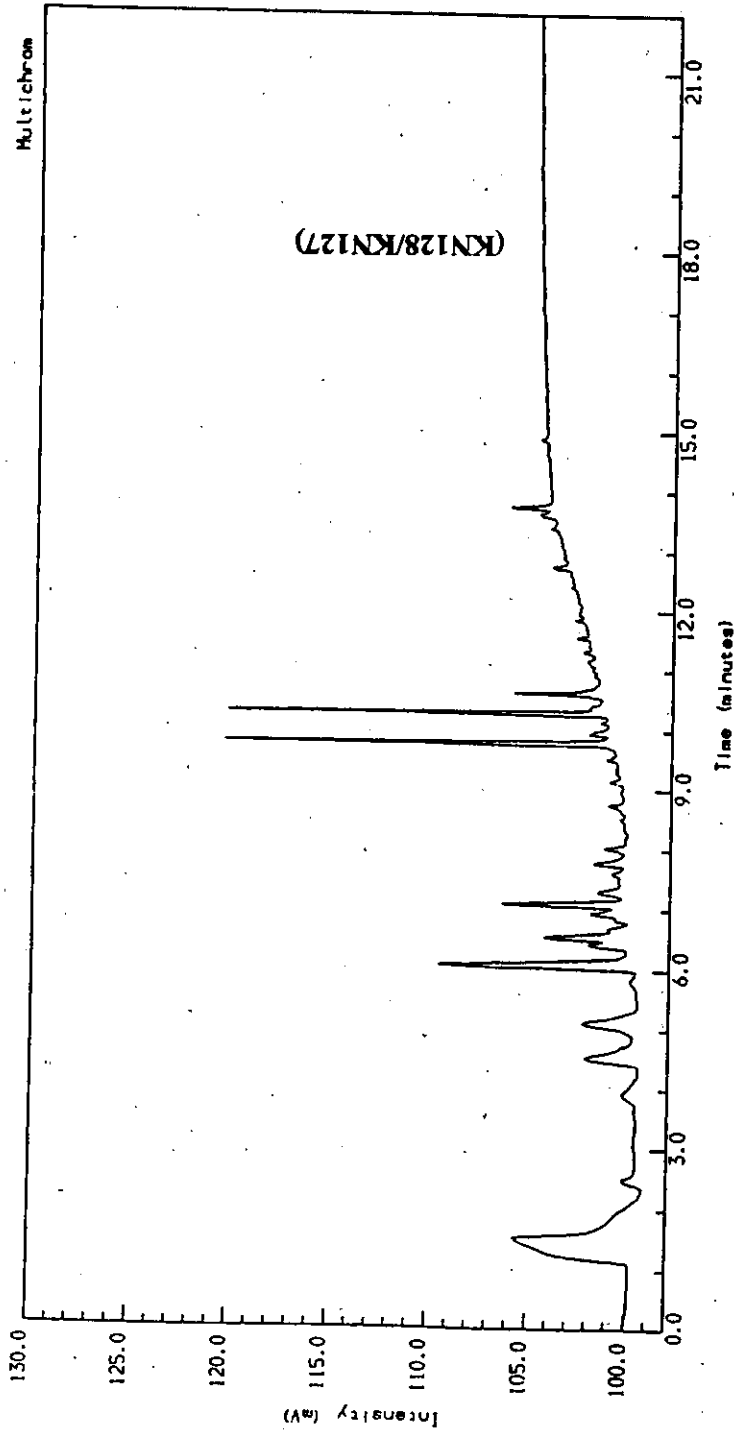
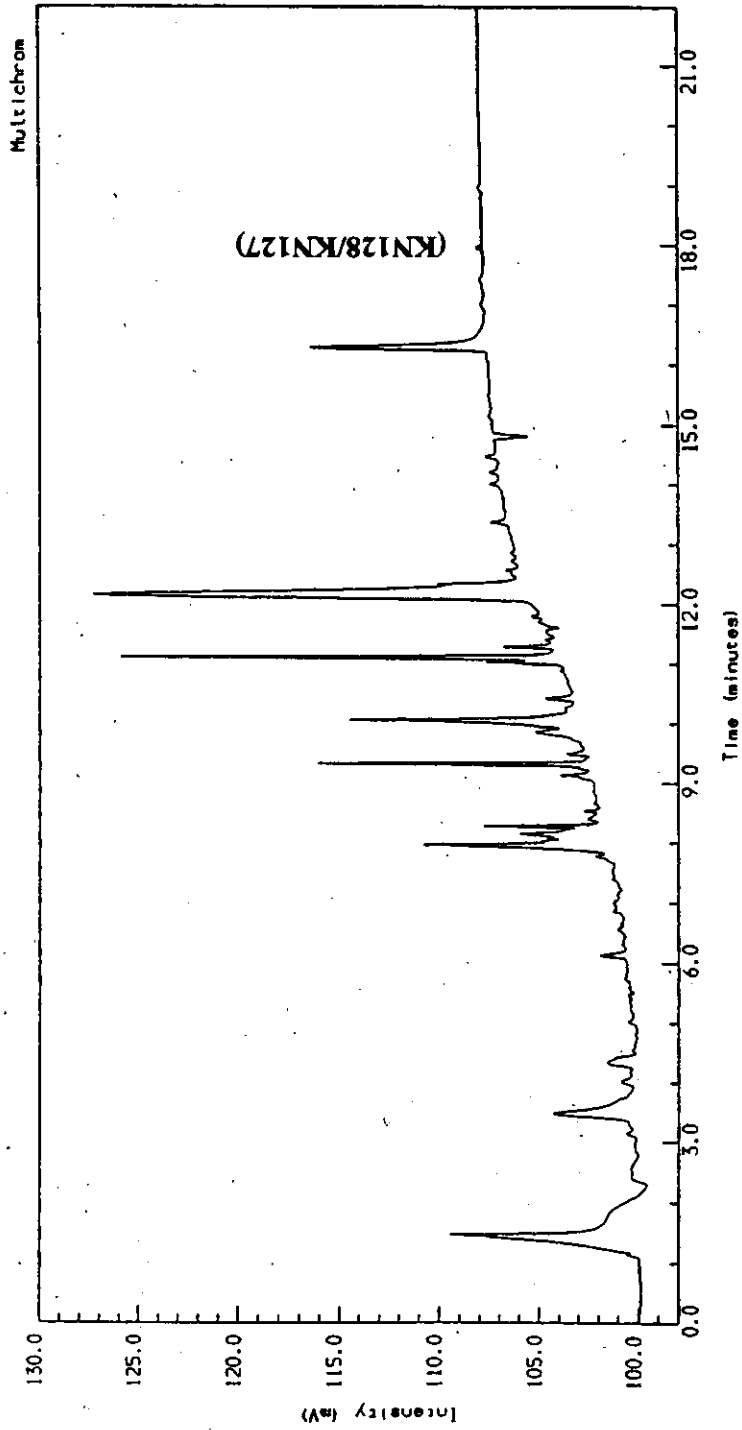
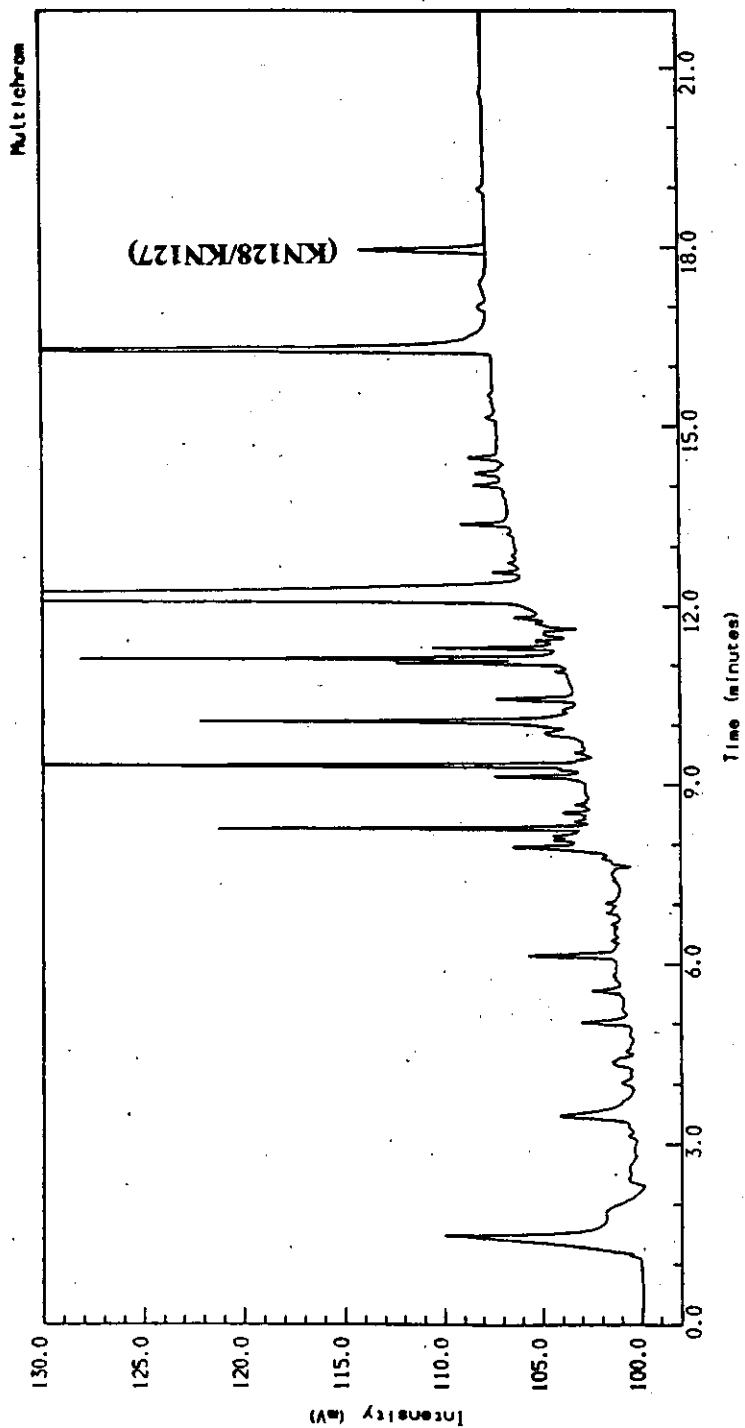


FIGURE 6
GAS CHROMATOGRAM FOR 0.5-PPB KN128/KN127 STANDARD IN TOLUENE .



24

FIGURE 7
GAS CHROMATOGRAM FOR 10.0-PPB KN128/KN127 STANDARD IN TOLUENE



25

FIGURE 8
GAS CHROMATOGRAM FOR BRADENTON, FL, WATER FORTIFIED TO 0.050-PPB
WITH KN128/KN127

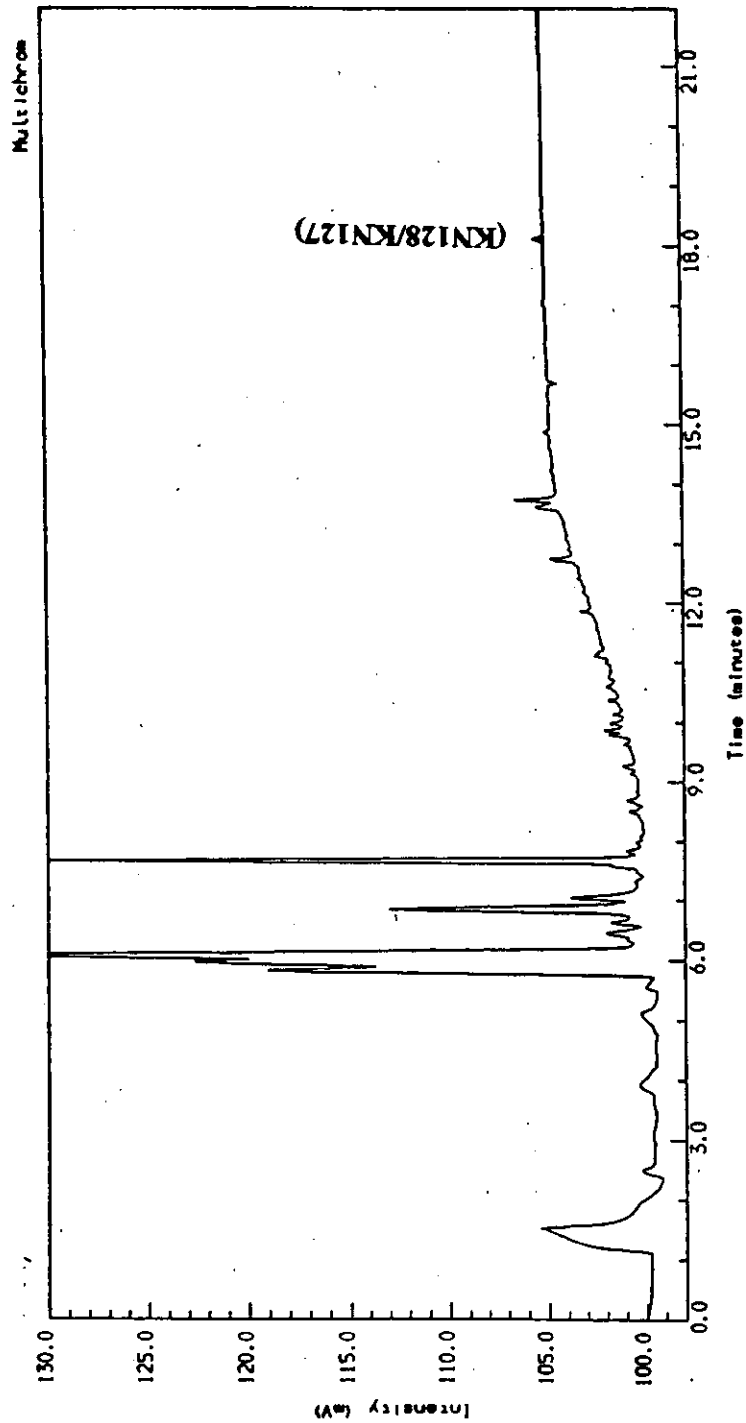


FIGURE 9
GAS CHROMATOGRAM FOR BRADENTON, FL, WATER FORTIFIED TO 2.0-PPB
WITH KN128/KN127

