

Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pesticide Name: Pyridaben (BAS300 I)

MRID #: 441227-03

Matrix: Sediment

Analysis: GC/ECD

This method is provided to you by the Environmental Protection Agency's (EPA) Environmental Chemistry Laboratory (ECL). This method *is not* an EPA method but one which was submitted to EPA by the pesticide manufacturer to support product registration. EPA recognizes that the methods may be of some utility to state, tribal, and local authorities, but makes no claim of validity by posting these methods. Although the Agency reviews *all* Environmental Chemistry Methods submitted in support of pesticide registration, the ECL evaluates only about 30% of the currently available methods. Most methods perform satisfactorily but some, particularly the older methods, have deficiencies. Moreover, the print quality of the methods varies considerably because the methods originate from different sources. Therefore, the methods offered represent the best available copies.

If you have difficulties in downloading the method, or further questions concerning the methods, you may contact Elizabeth Flynt at 228-688-2410 or via e-mail at flynt.elizabeth@epa.gov.

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TITLE

Analytical Method Trial at Huntingdon Analytical Services for the Analysis of BAS 300 I in Pond Sediment

EPA GUIDELINE REQUIREMENT

Subdivision E, Series 72

441227-03

AUTHORS

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Study Completion Date: June 24, 1996

SPONSOR

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PERFORMING LABORATORY

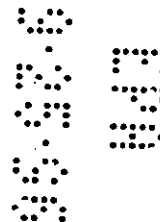
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BASF Protocol Number: 92162
BASF Report Number: A9606
Huntingdon Study Number: A008.036

BASF REGISTRATION DOCUMENT NO.:

96/5116

This report contains 26 pages.



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BASF Protocol Number 92162

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PR 86-5 DATA CONFIDENTIALITY CLAIM

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA 10 (d) (1) (A), (B) or (C).

Company: BASF Corporation, Agricultural Products

Company Agent: Rodney C. Akers Date: August 13, 1986

Title: Registration Scientist Signature: Rodney C. Akers

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Analytical Method Trial at Huntington Analytical
Services for the Analysis of BAS 300 I in Pond Sediment

BASF COMPLIANCE WITH GOOD LABORATORY PRACTICES

The study described in this report was conducted in compliance with the Good Laboratory Practice Standards as described in United States Environmental Protection Agency, Title 40 Code of Federal Regulations Part 160, Federal Register, issued 17 August 1989.

Jean Qiu

Jean Qiu
Study Director
BASF Corporation

6/24/96

Date

Jane Casale

Sponsor
BASF Corporation

6/24/96

Date

Rodney C. Akers

Submitter
BASF Corporation

August 13, 1996

Date

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Analytical Method Trial at Huntingdon Analytical
Services for the Analysis of BAS 300 I in Pond Sediment

HUNTINGDON COMPLIANCE WITH GOOD LABORATORY PRACTICES

The study described in this report was conducted in compliance with the Good Laboratory Practice Standards as described in United States Environmental Protection Agency, Title 40 Code of Federal Regulations Part 160, Federal Register, issued 17 August 1989.

Toreen A. Bixler
Toreen A. Bixler
Project Coordinator
Huntingdon Analytical Services

June 6, 1994
Date

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Analytical Method Trial at Huntingdon Analytical
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CERTIFICATE OF AUTHENTICITY

I hereby declare that the study was performed according to the procedures herein described, and that this report provides a correct and faithful record of the results obtained.

Huntingdon:

Toreen A Bixler June 6, 1996
Toreen A Bixler Date
- Project Coordinator

The following personnel were involved in the generation of data reported herein:

- Donna G. Besco, Analyst
- Gregory Bernard, Analyst
- Dennis C. Besco, Sample Custodian,

BASF:

Jean Qiu 6/24/96
Jean Qiu Date
Study Director

Analytical Method Trial at Huntingdon Analytical
Services for the Analysis of BAS 300 I in Pond Sediment

**HUNTINGDON QUALITY ASSURANCE AUDIT STATEMENT
AND STUDY INSPECTION DATES**

This report has been audited by the Huntingdon Quality Assurance Department. It is considered to be an accurate description of the procedures and practices employed during the course of the study and an accurate presentation of the raw data produced during the course of the study.

The following audits were made by the Quality Assurance Department during phases of the study described in this report. The dates on which audits were made and the dates on which the findings were reported to the Study Director and to Management are given below:

<u>Phase of Study</u>	<u>Date of Audit</u>	<u>Reported to</u>	
		<u>Study Director</u>	<u>Study Director Management</u>
Protocol Review	10/12/92	10/12/92	10/12/92
In-Process Inspection	10/12-15/92	10/21/92	10/21/92
Data Audit	10/21/92	10/21/92	10/21/92
Final Report Audit	04/11/94 06/06/96	04/11/94 06/06/96	04/11/94 06/06/96



John M. Isch
Quality Assurance Manager



Date

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Statement of the BASF Quality Assurance Unit

Report Number: A9606
Protocol Number: 92162

Type of Study: Analytical Method Trial

The Quality Assurance Unit of the testing facility at the APC has inspected/audited the protocol, study, raw data and the final report and reported its findings to the study director and management.

Date of Inspection	Reported to Study Director and Management
9/25/92 10/5/94	9/25/92 10/5/94



Signature of QAU

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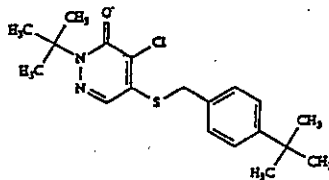
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Analytical Method Trial at Huntingdon Analytical
Services for the Analysis of BAS 300 I in Pond Sediment

TEST SUBSTANCE

The following standard, received from BASF Corporation, Agricultural Research Center, P.O.Box 13528, 26 Davis Dr., Research Triangle Park, NC 27709-3528, was utilized for this study:

Product Name: Pyridaben
Chemical Name: 2-~~tert~~-butyl-5-(4-~~tert~~-butylbenzylthio)-3(2H)-pyridazinone
CAS Number: 96489-71-3
Lot Number: 129S8604
Purity: 99.5%
Structure:



The analytical standard was stored at $<-5^{\circ}\text{C}$ when not in use. The standard calibration solutions were stored in a refrigerator (2°C - 10°C) when not in use.

SUMMARY

BASF Analytical Method No. D9201 entitled "GC Method for Residue Determinations of Pyridaben (BAS 300 I) in Soil and Pond Sediment" (Reference 1) has been successfully validated in pond sediment at Huntingdon Analytical Services (HAS). The method trial was performed using control pond sediment received from Toxikon Environmental Sciences, Jupiter, FL 33477. The sediment samples were collected September 14, 1992 from Tank #7 (Reference 2). The mean recovery for Pyridaben in pond sediment was 97.5% (n=8) with a standard deviation of $\pm 7.9\%$.

INTRODUCTION

This report contains the validation data of Pyridaben as determined by HAS. The study was initiated October 2, 1992, the experimental starting date was October 14, 1992 and data were collected up to October 16, 1992. This validation adheres to the guidelines set forth in BASF Study Number 92162 (HAS Study Number A008.036).

The original final report, original raw data including laboratory notebooks, chromatograms with corresponding data, as well as an exact copy of remaining raw data will be retained in the BASF archives at 26 Davis Drive, Research Triangle Park, NC 27709. Copies of original data, protocol and final report will be retained in the HAS archives for 3 years, after which time, said data will be discarded. Facility raw data will be retained by HAS.

SAMPLE IDENTIFICATION

The pond sediment sample was received at Huntingdon on September 25, 1992 and stored at $< -5^{\circ}\text{C}$ (Room 120) until analysis. It was given a unique eight digit sample number, where:

1. 20 - Agrichemical Group
2. 594 - Batch number
3. Next three numbers - Individual sample number

HAS notebook/queue numbers were assigned as follows:

1. First three numbers: HAS notebook number
2. Next two numbers: notebook page number
3. Next two numbers: unique sample number in each analytical set
4. The last letter A,B,C, etc. was added for computer identification so that the actual sample ID (1+2+3) was not overwritten if the sample had to be re-injected.

For example: 4040603A (See Figure 7)
404 - HAS notebook number
06 - notebook page number
03 - unique sample number
A - injection for quantitation of Pyridaben

METHOD OF ANALYSIS.

BASF Method D9201 was validated by fortifying duplicate control pond sediment samples with Pyridaben at 0.01, 0.02, 0.50 and 5.0 ppm. This was achieved by adding known amounts of Pyridaben standard solutions to the samples by Class A volumetric pipets. Duplicate control samples were also analyzed. The limit of quantitation was 0.01 ppm.

A brief description of the method follows. Pyridaben was extracted from pond sediment with methanol. The concentrated extract was acidified, and partitioned with dichloromethane (DCM). The residue was then subjected to Florisil column chromatography. Final quantitation was accomplished by capillary GLC using a ⁶³Ni electron capture detector. (See Figure 1)

QUANTITATION.

Gas chromatographic conditions utilized at HAS for quantitation of pyridaben are listed below:

Hewlett Packard 5890 Gas Chromatograph

Column:	J&W DB-5; 30 meter; 0.32 mm i.d.; 1.0 μ film thickness
Column temperature:	Initial temperature was 250°C for 0.5 minute; ramp to 300°C at 15° per minute. Hold for 10 minutes; then ramp to 325°C at 15° per minute and hold for 10 minutes. Equilibration time was 3 minutes.
Detector temperature:	325°C
Injector temperature:	270°C
Gas Flows:	
Carrier:	
Helium	Head pressure set to 18 psi; total flow at 107 mL/minute
Detector:	
Argon:Methane (95:5)	60 mL/minute
Injection volume:	1 μ L (splitless)

Gas chromatographic data were processed on a Perkin-Elmer computer using CLAS. The multi-level (linear regression) program was used to calculate the ppm found in control and fortified samples. Quattro Pro spreadsheets were used to summarize the data and to calculate recoveries from the fortified samples. Examples of the calculations used are shown in Figure 2.

RESULTS

Validation data of Pyridaben in pond sediment are presented in Table I. The mean recovery was 97.5% (n=8) with a standard deviation of $\pm 7.9\%$.

Quantitation levels of 0.01 ppm were achieved. Recoveries were corrected for residues in the controls.

A freezer storage stability study of BAS 300 I in pond sediment was conducted at Huntingdon. The test substance was found to stable over a twelve month period (Reference 3).

There were two (2) changes to the protocol:

1. Change in the analytical standard lot number.
2. To correct the EPA Guideline Requirement

Representative chromatograms of analytical standards, control and fortified pond sediment, as well as a typical linearity curve for Pyridaben, can be found in Appendix I. Chromatograms were reduced for reporting purposes.

REFERENCES

1. Nelson Delgado, "GC Method for the Determinations of Pyridaben (BAS 300 I) in Pond Sediment", BASF Method D9201, November 16, 1993, BASF Corporation, Research Triangle Park, NC 27709.
2. Gary M. Rand, Ph.D, Toxicon Environmental Sciences and Catherine M. Holmes, BASF Corporation, "Pyridaben (BAS 300 I): An Outdoor Aquatic Microcosm Study", May 26, 1995, BASF Report Number ER94066, BASF Corporation, Agricultural Products, P.O. Box 13528, Research Triangle Park, NC 27709-3528.
3. Torean A. Bixler, Huntingdon Analytical Services, "Freezer Storage Stability of BAS 300 I in Pond Sediment", December 7, 1994, BASF Report Number A9448, BASF Corporation, Agricultural Products Group, P.O. Box 13528, Research Triangle Park, NC 27709-3528

Figure 1. Flow Diagram for BASF Analytical Method No. D9201 as Applied to Pond Sediment

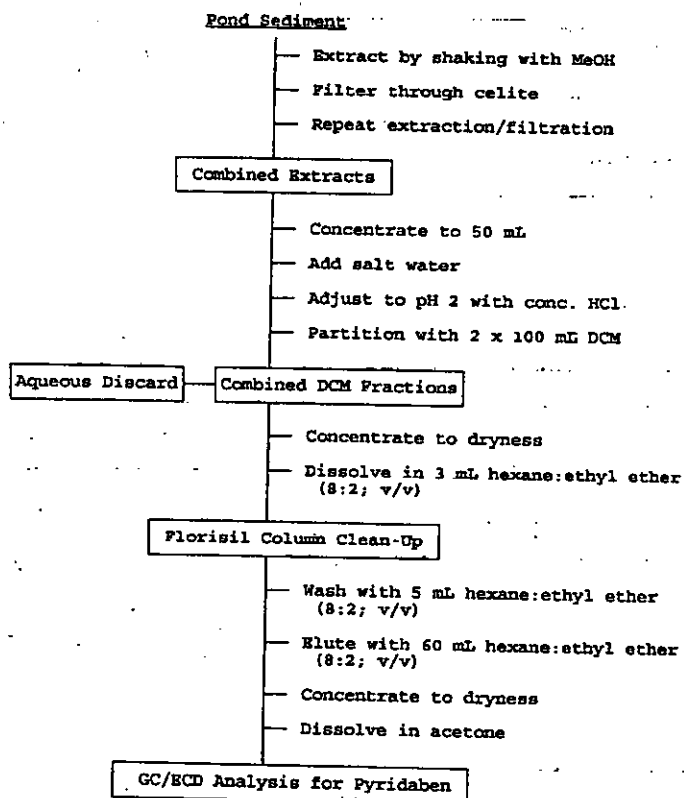


Figure 2. Typical Calculations for the Quantitation of Pyridaben in Pond Sediment

HAS Notebook/Queue Number: 4040603A; Pond Sediment fortified at 0.01 ppm with Pyridaben

PPM values were calculated using a multilevel calibration method that resides on a Perkin-Elmer LIMS/CLAS data acquisition system. The method constructed a weighted best fit line through identified calibration standards using the following equation:

$$y = cx + d \quad \text{where: } y = \mu\text{g/mL Found}$$

$c = \text{Slope } (1.380\text{E-}5)$
 $x = \text{Peak Height Response } (9728)$
 $d = y - \text{Intercept } (-1.140\text{E-}2)$

$$c = \frac{\sum \frac{1}{X_i} \sum Y_i - N \sum \left(\frac{Y_i}{X_i}\right)}{\sum \frac{1}{X_i} \sum X_i - N^2}$$

$$d = \frac{\left[\sum X_i \sum \left(\frac{Y_i}{X_i}\right) - N \sum Y_i\right]}{\sum \frac{1}{X_i} \sum X_i - N^2}$$

Four calibration standards were injected (in duplicate) throughout the analytical set from which the method was calibrated. The range of standards was 0.05 $\mu\text{g/mL}$ to 0.50 $\mu\text{g/mL}$, 0.05 ng to 0.50 ng injected (See Figures 2-5). Using the analyte response (peak height) and the regression standard curve, the calibration method in the data acquisition system computed a concentration (Conc; $\mu\text{g/mL}$) for that analyte in the sample.

$\mu\text{g/mL}$	Peak Height
0.05	3862, 4474
0.10	8264, 9003
0.25	19916, 19538
0.50	36692, 35271

$$y = (1.380\text{E-}5)(9728) + (-1.140\text{E-}2)$$
$$y = 0.1228$$

The calculated Conc was then multiplied by the final volume (F.V.; identified as standard weight on chromatogram report), multiplied by a dilution factor (D.F.), if necessary, and divided by the sample weight (50 g) to obtain a ppm concentration based on the following equation:

$$ppm = \frac{Conc \times F.V. \times D.F.}{Sample\ Wgt}$$

Where:

Conc = $\mu\text{g/mL}$ Found, (0.1228)
F.V. = 5.0 mL
Sample Wgt = 50.0 g

$$ppm = \frac{0.1228 \times 5 \times 1}{50}$$

$$ppm = 0.012$$

Recoveries were corrected for residues in controls as follows:

HAS Queue Number: 4040603A (Fortified Sample; see Figure 7)
4040601A (Control Sample; see Figure 6)

$$\begin{aligned} \text{Corrected ppm} &= \text{ppm Found in Fortified Sample} - \text{ppm Found in Control} \\ &= 0.012 - 0.002 \\ &= 0.010 \end{aligned}$$

$$\begin{aligned} \% \text{ Recovery} &= \frac{\text{Corrected ppm}}{\text{ppm Added}} \times 100 \\ &= \frac{0.010}{0.010} \times 100 \\ &= 100 \end{aligned}$$

TABLE I. Recoveries of Pyridaben in Pond Sediment

ppm Added 1)	Final Volume (mL)	Sample Wt. Injected (mg)	ng Found	ppm Net (Pyridaben) 2)	Percent Recovery 3)	HAS Notebook/ Queue No.	Extraction Date	Injection Date
0.00	5.0	10.0	0.02	0.002	-	4040601A	10/14/92	10/15/92
0.00	5.0	10.0	0.04	0.004	-	4040701A	10/16/92	10/15/92
0.01	5.0	10.0	0.11	0.011	110.0	4040602A	10/14/92	10/15/92
0.01	5.0	10.0	0.10	0.010	100.0	4040603A	10/14/92	10/15/92
0.02	5.0	10.0	0.19	0.019	95.0	4040604A	10/14/92	10/15/92
0.02	5.0	10.0	0.21	0.021	105.0	4040605A	10/14/92	10/15/92
0.50	100.0	0.50	0.23	0.466	93.2	4040702A	10/16/92	10/16/92
0.50	100.0	0.50	0.24	0.486	97.2	4040703A	10/16/92	10/16/92
5.0	1000.0	0.05	0.21	4.18	83.6	4040704A	10/16/92	10/16/92
5.0	1000.0	0.05	0.24	4.81	96.2	4040705A	10/16/92	10/16/92

1) Fortification solutions were added prior to extraction.

2) ppm Net = ng Found/mg Injected

3) Percent Recovery = (ppm Net/ppm Added) x 100

Injection volume was constant at 1 uL. Sample size was 50 grams.

Values for ng Found (Gross) and ppm Net have been rounded off for reporting purposes, but not for any further calculations. The ppm Net values were corrected for apparent residues in the controls, although these residues were less than the limit of quantitation and were extrapolated.

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APPENDIX I

REPRESENTATIVE CHROMATOGRAPHIC DATA

1942

1942



1942



REPRESENTATIVE CHROMATOGRAPHIC DATA

Figure 3. Typical Chromatogram of Analytical Standard 0.05 $\mu\text{g/mL}$;
1 μL injected = 0.05 ng of analyte

Figure 4. Typical Chromatogram of Analytical Standard 0.10 $\mu\text{g/mL}$;
1 μL injected = 0.10 ng of analyte

Figure 5. Typical Chromatogram of Analytical Standard 0.25 $\mu\text{g/mL}$;
1 μL injected = 0.25 ng of analyte

Figure 6. Typical Chromatogram of Analytical Standard 0.50 $\mu\text{g/mL}$;
1 μL injected = 0.50 ng of analyte

Figure 7. Representative Chromatogram of Control Pond Sediment
1 μL (10 mg) injected
<0.010 ppm Pyridaben Found

Figure 8. Representative Chromatogram of Control Pond Sediment
Fortified at 0.01 ppm with Pyridaben
1 μL (10mg) injected
0.010 ppm Pyridaben Recovered; 100.0% Recovery

Figure 9. Representative Chromatogram of Control Pond Sediment
Fortified at 5.0 ppm with Pyridaben
1 μL (0.05 mg) injected
4.81 ppm Pyridaben Recovered; 96.2% Recovery

Figure 10. Typical Pyridaben Linearity Curve

1941

1941

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1941

HAS 0.05 NG PYRIDASEN
MULTILEVEL EXTERNAL STANDARD

SAMPLE: S40-0001.01 INST: 10 VIAL: 03 SEQ NUMBER: 001
COLLECTION TIME: 14.04 DATE-TIME COLLECTED: 10/16/92 14:04:13
METHOD: 85038 / 85038 REV #: 00009 ANALYST: BASF1 DATE-TIME PROCESSED: 10/16/92 07:00:19
SAMPLE WT: 1.0000 STANDARD WT: 1.0000 DELTA/TIME FACTOR: 1.0000
NO. MINUTES NO. NAME PEAK HEIGHT WEIGHT
001 10.037 0 PYRIDASEN 2052 0.042

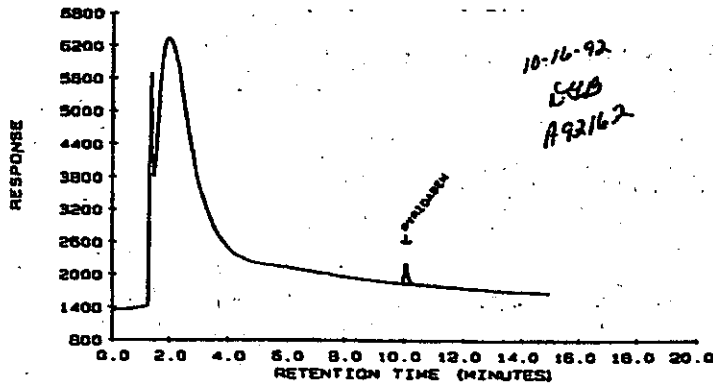


FIGURE 3

10571 Account of J. R. I. AM
20/10/1911

10571 Account of J. R. I. AM
20/10/1911



10571 Account of J. R. I. AM
20/10/1911

10571 Account of J. R. I. AM
20/10/1911

10571 Account of J. R. I. AM
20/10/1911



10571 Account of J. R. I. AM
20/10/1911

10571 Account of J. R. I. AM
20/10/1911



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HAS 0.10 NG PYRIDABEN
MULTILEVEL EXTERNAL STANDARD

SAMPLE: S404000.DT INET: 10 VIAL: PD SER NUMBER: 000
DATE-TIME COLLECTED: 10/16/92 15:14:59
COLLECTION TIME: 10.50 DATE-TIME PROCESSED: 10/16/92 07:33:48
METHOD: ECD30 / UR30S REV: 0.0000 ANALYST: BASF1 SAMP RATE: 0.12
SAMPLE WT: 1.0000 STANDARD WT: 1.0000 CALIBRATION FACTOR: 1.0000

PKT	PKT#	PKTNAME	PKTWT	PKTID
01	10.028	0 PYRIDABEN	0.100	0.100

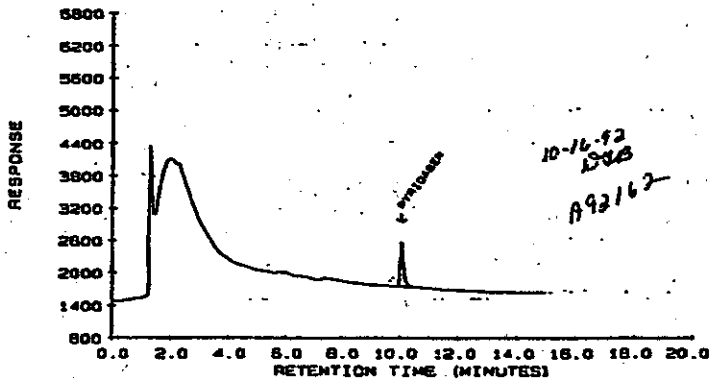
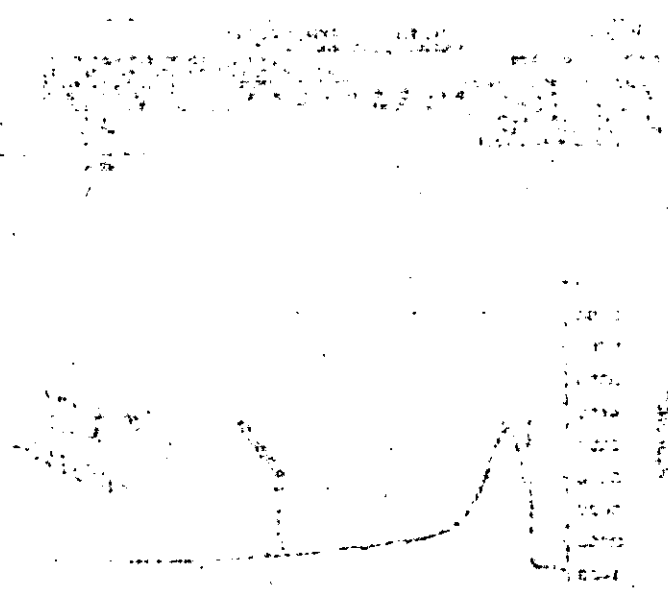


FIGURE 4

THE UNIVERSITY OF CHICAGO
LIBRARY



WAVELENGTH (microns)

TRANSMITTANCE

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HAS 0.25 NG PYRIDABEN
MULTILEVEL EXTERNAL STANDARD

SAMPLE: S4640003.01	INST: 10 VIAL #0 SEQ NUMBER: 005			
COLLECTION TIME : 14.54	DATE-TIME COLLECTED : 10/15/92 18:20:41			
METHOD: 88335 / 88035	REV: 01 00008 ANALYST: BASF1			
SAMPLE WT : 1.0000	STANDARD WT : 1.0000			
PEAK NO	RET. TIME (MIN)	COMPONENT	PEAK HEIGHT	PEAK AREA
002	10.034	N PYRIDABEN	19918	0.283
			20120	212.202

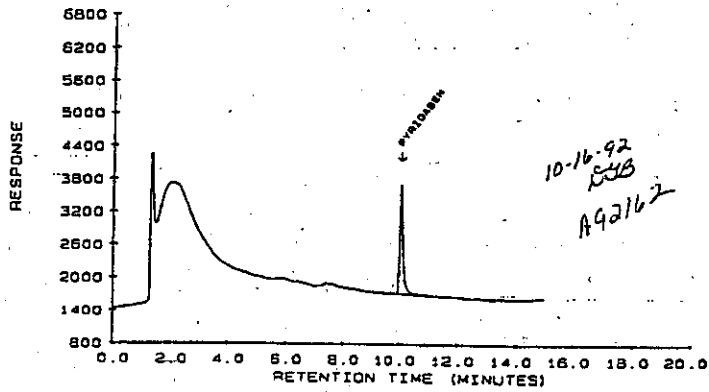


FIGURE 5

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HAS 0.50 NG PYRIDABEN
MULTILEVEL EXTERNAL STANDARD

SAMPLE: S4040504.01	INST: 10 VIAL: 70 SEQ NUMBER: 007
COLLECTION TIME: 14.04	DATE-TIME COLLECTED: 10/15/92 17:28:58
METHOD: 8503B / 8503B	REV #: 00000 ANALYST: BASF1
SAMPLE WT: 1.0000	STANDARD WT: 1.0000
IN BY GR COMPONENT	DILUTION FACTOR: 1.0000
NO MINUTES NO NAME	PEAK WEIGHT
001 10.027 N PYRIDABEN	36892 0.495
	36892 0.495

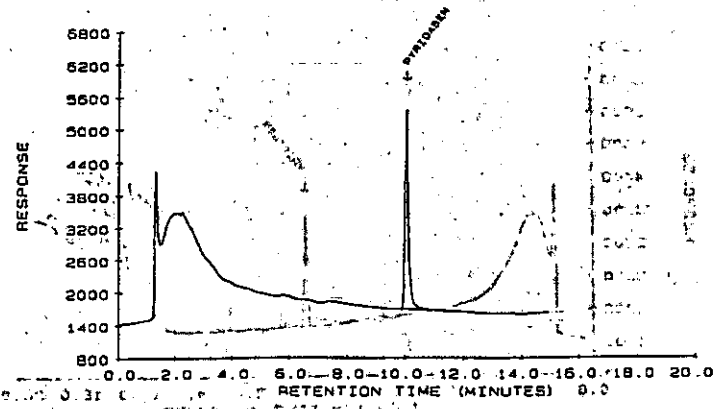


FIGURE 6

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HAS POND SEDIMENT CONT
PROCESSED BY GRAPHICAL ANALYSIS
MULTILEVEL EXTERNAL STANDARD

SAMPLE: 4040601A.D1 INST: 10 VIAL: 60 SEQ NUMBER: 002
COLLECTION TIME: 14.94 DATE-TIME COLLECTED: 10/15/92 14:41:18
METHOD: 85028 / 85028 REV: 01 00006 ANALYST: BASF DATE-TIME PROCESSED: 10/16/92 13:10:50
SAMPLE WT: 50.0000 STANDARD WT: 3.0000 DILUTION FACTOR: 1.0000
PK RT SR COMPONENT PEAK HEIGHT
NO MINUTES NO NAME
002 9.089 H PYRIDAZIN

PEAK	HEIGHT
002	3079
	191.402

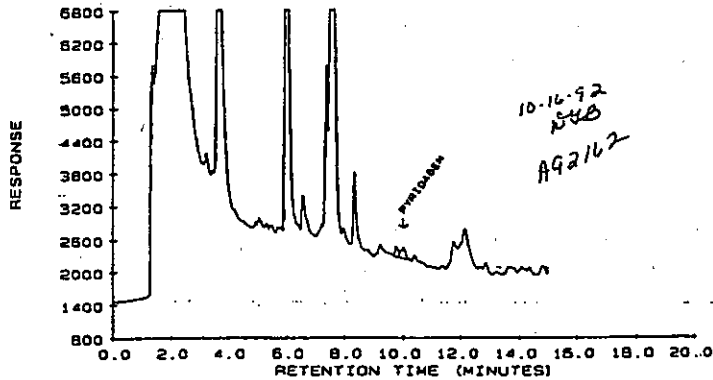


FIGURE 7

HAS

POND SOMNT 0.01 PPM
PROCESSED BY GRAPHICAL ANALYSIS
MULTILEVEL EXTERNAL STANDARD

SAMPLE: 40-0803A.01
INSTR: 10 VIAL/70 SEC NUMBER: 008
DATE-TIME COLLECTED: 10/15/92 18:53:50
COLLECTION TIME: 14.84 DATE-TIME PROCESSED: 10/15/92 13:13:50
METHOD: 83038 / 85038 REV #: 00008 ANALYST: BASF1 SAMP RATE: 3.13
SAMPLE WT: 50.0000 STANDARD WT: 5.0000 DILUTION FACTOR: 1.0000
PK RT CR COMPONENT
NO MINUTES NO NAME
009 10.025 M PYRIDASEN

PEAK	HEIGHT	WEIGHT
0728	0.012	
14113		436.512

10-16-92
BSB
A-92162

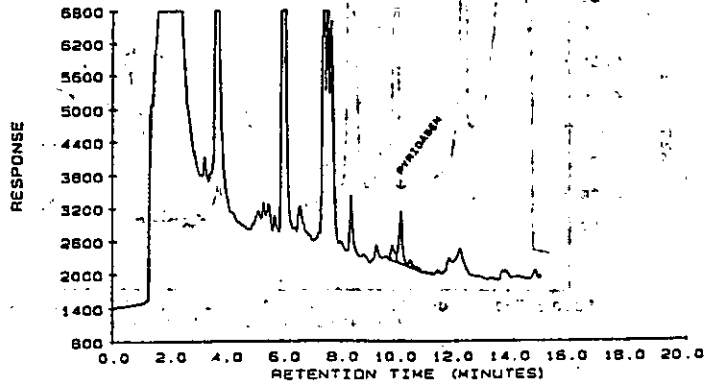


FIGURE 8

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HAS PONO SDMNT 5.0 PPM
MULTILEVEL EXTERNAL STANDARD

SAMPLE: 40407004 01	INSTR 10 VIAL 20 SEC NUMBER 010					
METHOD: 85038 / 85038	DATE-TIME COLLECTED 10/19/92 20:28:58					
REV #: 00008	DATE-TIME PROCESSED 10/19/92 07:22:24					
ANALYST: BASF	SAMP RATE: 2.13					
SAMPLE WT: 50.0000	STANDARD WT: 5.0000	DILUTION FACTOR: 200.0000				
PH	WT	GR	COMPONENT	WT	PEAK	HEIGHT
001	9.991	H	PYRIDAZEN		18853	4.812
					18853	4.812

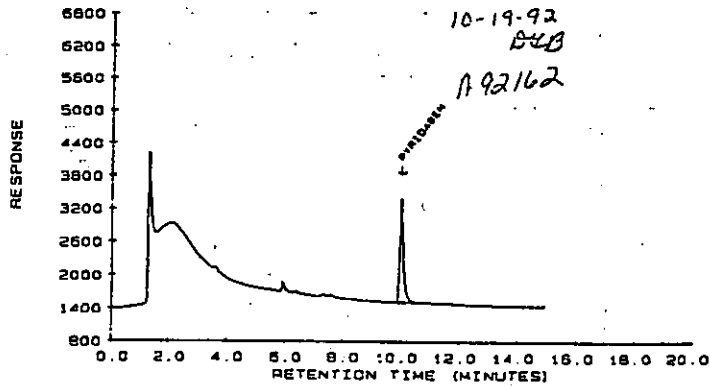
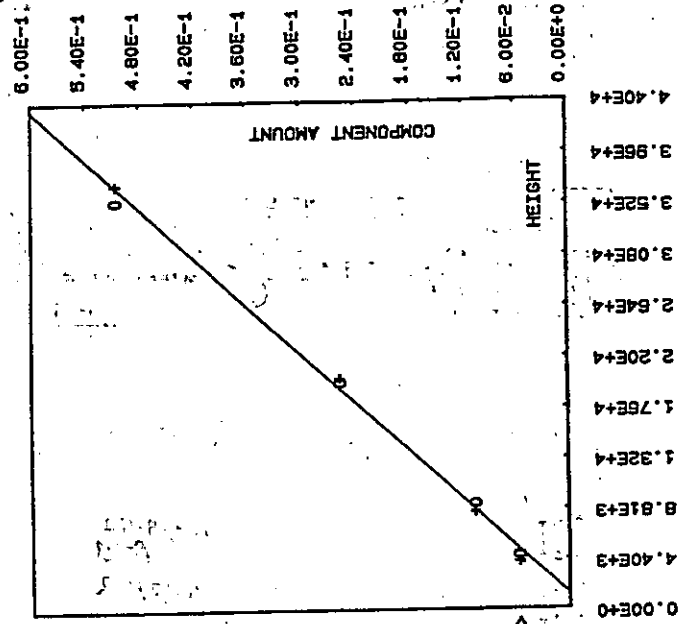


FIGURE 9

BASF Report Number A9606
 BASF Protocol Number 92162

HAS Study Number A008.036
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PERKIN-ELMER LIMS 2000
 METHOD B5036
 1ST ORDER EXTERNAL STANDARD

COMPONENT:
 PYRIDABEN

CONC = cx+ d

c = 1.380E-5
 d = -1.140E-2

CORR : 0.98832
 SD FIT : 0.00010

CURRENT LEVEL = A
 COMPONENT AMOUNT 0.5E-01

DATA FILE HEIGHT
 + S4040601.01 3882.0
 0 S4040605.01 4474.0

A 97167
 10-16-92
 JCB

FIGURE 10