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HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361

OFFICE OF
PREVENTION, PESTICIDES, AND
TOXIC SUBSTANCES

MEMORANDUM

DATE: 11-OCT-2001

SUBJECT: PP# 0E06185. Multiresidue Method (MRM) Testing of Diflufenzopyr and its Metabolite M1. MRID# 44307415. Barcode D278337. Chemical 005107 and 005108. Case 293208. Submission S590359.

FROM: Jennifer R. Tyler, Chemist
Registration Action Branch 1 (RAB1)
Health Effects Division (HED) (7509C)

THRU: G. Jeffrey Herndon, Branch Senior Scientist
RAB1/HED (7509C)

TO: F.D. Griffith, Jr., Ph.D., Chief
Analytical Chemistry Laboratory (ACL)
Biological Economical and Analysis Division (BEAD) (7503C)

Attached are completed forms from the MRM testing of diflufenzopyr and its metabolite M1.

Title	"Multiresidue Methodology Testing for SAN-835H and Phthalazinone:M1" Fred A. Claussen, M.A., Author (11/12/96)
Chemicals	Diflufenzopyr* and M1**
Type	Herbicide
Protocol	C
Company	Valent U.S.A. Corporation
Performing Laboratory	EPL Bio-Analytical Services, Inc. P.O. Box 109, 395 N. Memorial Parkway Harristown, IL 62537
MRID#	443074-15
40 CFR Reference	180.549

Attachment 1: Multiresidue Testing Forms for Diflufenzopyr and M1.

If you have any questions, please do not hesitate to call.

cc (without attachment): J. Tyler, Robert Forrest/Shaja Brothers (RD-7505C)
RDI: G. Herndon (10/11/01); RAB1 Chemist Team (10/11/01); G. Kramer (10/11/01)
J. Tyler:806W:CM#2:(703)305-5564:7509C:RABI

* 2-(1-[([3,5-difluorophenylamino] carbonyl)hydrazono]ethyl)-3-pyridinecarboxylic acid
** 8-methylpyrido[2,3-d]pyridazin-5(6H)-one

1.

ATTACHMENT 1

(Not Available Electronically)

REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical Phthalazinone:M1 on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: Phthalazinone:M1
Alternative Names:
8-methyl-5(6H)-pyrido[2,3-d]pyridazinone
Reference Standard: Phthalazinone:M1, lot number: RS-M1-051895
Molecular Formula: $C_8H_7N_3O$
Structure:

Comments: All retention times were measured from the time of injection, not from the solvent front.

RESULTS FOR DG MODULE (SECTION 302): DGI Level I
Standard reference material dissolved in: Acetone
Brief details about GLC system used:
Column: HP-1 (Hewlett-Packard) at 190°C

Length:	30 m
id:	0.53 mm
Film Thickness:	0.88 μm
Carrier gas:	He
Flow rate:	15 mL/min
Makeup gas:	P5
Flow rate:	50 mL/min

Retention time (relative to chlorpyrifos) of p,p'-DDT: 3.09

Detector: ^{63}Ni ECD
Temperature: 350°
Other Conditions: None
Detector response to 0.15 ng chlorpyrifos:50% FSD
(under computer)

Behavior of Phthalazinone:M1:
Retention time (relative to chlorpyrifos): 0.35
ng required for 50% FSD: 47

Information submitted by: Fred Claussen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
Date: June 9, 1997

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REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical methylated SAN-835H on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: Methylated SAN-835H
Alternative Names: Not Applicable
Reference Standard: SAN-835H, lot number RS-835-070296
Molecular Formula: Unknown
Structure: Unknown

Comments: SAN-835H derivatized using 1M TBAH in MeOH according to PAM Volume I, Section 402C1b, pp. 402-21 through 402-22. Florisil cleanup was not used. Several peaks were observed in the GC/ECD chromatogram. The most prominent peak was used to obtain the results below. Only one other peak (methyl peak 1) in the chromatogram yielded a peak height >5% FSD. The relative retention time to chlorpyrifos for methyl peak 1 was 5.22. A methylated product could not be verified by GC/MS. All retention times were measured from the time of injection, not from the solvent front.

RESULTS FOR DG MODULE (SECTION 302): DG1 Level I
Standard reference material dissolved in: Hexane
Brief details about GLC system used:
Column: HP-1 (Hewlett-Packard) at 190°C

Length:	30 m
id:	0.53 mm
Film Thickness:	0.88 μm
Carrier gas:	He
Flow rate:	15 mL/min
Makeup gas:	P5
Flow rate:	50 mL/min

Retention time (relative to chlorpyrifos) of p,p'-DDT: 3.09

Detector: ^{63}Ni ECD
Temperature: 350°
Other Conditions: None
Detector response to 0.15 ng chlorpyrifos:50% FSD
(marker compound)

Behavior of methylated SAN-835H:
Retention time (relative to chlorpyrifos): 3.30
ng required for 50% FSD: 51 (SAN-835H equivalent)

Information submitted by: Fred Claussen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
Date: June 9, 1997

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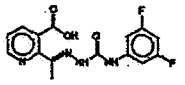
REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical SAN-835H on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: SAN-835H (BAS 654 H)
Alternative Names: Diflufenzopyr
(2-(methyl((3,5-difluorophenylamino)carbonyl)hydrazono)methyl)-3-pyridine-carboxylic acid)
Reference Standard: SAN-835H, lot number RS-835-070296
Molecular Formula: $C_{15}H_{12}N_4O_3F_2$
Structure:



Comments: Compound did not chromatograph on Level I system as described below. SAN-835H degraded to M1 upon injection into the gas chromatograph. Three additional peaks other than M1 were observed in the chromatogram (Unknowns #1, 2 and 3) The relative retention times to chlorpyrifos were 0.26, 0.46 and 5.34 for Unknowns #1, 2 and 3, respectively. All retention times were measured from the time of injection, not from the solvent front.

RESULTS FOR DG MODULE (SECTION 302): DG13 Level I
Standard reference material dissolved in: Acetone
Brief details about GLC system used:
Column: DB-17 (J&W Scientific) at 200°C

Length:	30 m
id:	0.53 mm
Film Thickness:	1.0 μm
Carrier gas:	He
Flow rate:	25 mL/min
Makeup gas:	P5
Flow rate:	50 mL/min

Retention time (relative to chlorpyrifos) of p,p'-DDT: 3.47
Detector: ^{63}Ni ECD
Temperature: 350°
Other Conditions: None
Detector response to 0.15 ng chlorpyrifos:50% FSD
(reader comment)

Behavior of SAN-835H:
Retention time (relative to chlorpyrifos): Could not be determined.
ng required for 50% FSD: Could not be determined.

Information submitted by: Fred Claussen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
Date: June 9, 1997

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REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical Phthalazinone:M1 on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: Phthalazinone:M1
Alternative Names:
8-methyl-5(6H)-pyrido[2,3-d]pyridazinone
Reference Standard: Phthalazinone:M1, lot number RS-M1-051895
Molecular Formula: $C_7H_5N_3O$
Structure:

Comments: All retention times were measured from the time of injection, not from the solvent front.

RESULTS FOR DG MODULE (SECTION 302): DG13 Level I
Standard reference material dissolved in: Acetone
Brief details about GLC system used:
Column: DB-17 (J&W Scientific) at 200°C

Length:	30 m
id:	0.53 mm
Film Thickness:	1.0 µm
Carrier gas:	He
Flow rate:	25 mL/min
Makeup gas:	P5
Flow rate:	50 mL/min

Retention time (relative to chlorpyrifos) of p,p'-DDT: 3.47

Detector: ^{63}Ni ECD
Temperature: 350°
Other Conditions: None
Detector response to 0.15 ng chlorpyrifos:50% FSD

Behavior of Phthalazinone:M1:
Retention time (relative to chlorpyrifos): 0.42
ng required for 50% FSD: 105

Information submitted by: Fred Claussen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
Date: June 9, 1997

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REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical methylated SAN-835H on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: Methylated SAN-835H
Alternative Names: Not Applicable
Reference Standard: SAN-835H, lot number RS-835-070296
Molecular Formula: Unknown
Structure: Unknown

Comments: SAN-835H derivatized using 1M TBAH in MeOH according to PAM Volume I. Section 402C1b, pp. 402-21 through 402-22. Florisil cleanup was not used. Several peaks were observed in the GC/ECD chromatogram. The most prominent peak was used to obtain the results below. Two additional peaks yielded peak heights >5% FSD (methyl peaks 1 and 2). Relative retention times to chlorpyrifos were 5.77 and 6.83 for peaks 1 and 2, respectively. A methylated product could not be verified by GC/MS. All retention times were measured from the time of injection, not from the solvent front.

RESULTS FOR DG MODULE (SECTION 302): DG13 Level I
Standard reference material dissolved in: Hexane
Brief details about GLC system used:
Column: DB-17 (J&W Scientific) at 200°C

Length:	30 m
id:	0.53 mm
Film Thickness:	1.0 μm
Carrier gas:	He
Flow rate:	25 mL/min
Makeup gas:	P5
Flow rate:	50 mL/min

Retention time (relative to chlorpyrifos) of p,p'-DDT: 3.47

Detector: ^{63}Ni ECD
Temperature: 350°
Other Conditions: None
Detector response to 0.15 ng chlorpyrifos:50% FSD
(another compound)

Behavior of methylated SAN-835H:
Retention time (relative to chlorpyrifos): 5.23
ng required for 50% FSD: 123 (SAN-835H equivalent)

Information submitted by: Fred Claussen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
Date: June 9, 1997

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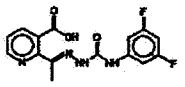
REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical SAN-835H on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: SAN-835H (BAS 654 H)
Alternative Names: Diflufenzopyr
(2-(methyl(((3,5-difluorophenyl)amino)carbonyl)hydrazono)methyl)-3-pyridine-carboxylic acid)
Reference Standard: SAN-835H, lot number RS-835-070296
Molecular Formula: $C_{15}H_{12}N_4O_3F_2$
Structure:



Comments: Compound did not chromatograph on Level I system as described below. SAN-835H degraded to M1 upon injection into the gas chromatograph. Three additional peaks were observed in the chromatogram which yielded peak heights >5% FSD (unknowns 1,2 and 3). Relative retention times to chlorpyrifos were 1.66, 1.09 and 0.69 for unknown peaks 1, 2 and 3, respectively. Unknown peak 1 was also observed in the M1 chromatogram. All retention times were measured from the time of injection, not from the solvent front.

RESULTS FOR DG MODULE (SECTION 302): DG18 Level I
Standard reference material dissolved in: Acetone
Brief details about GLC system used:
Column: DB-225 (J&W Scientific) at 195°C

Length:	30 m
id:	0.53 mm
Film Thickness:	1.0 μm
Carrier gas:	He
Flow rate:	20 mL/min
Makeup gas:	P5
Flow rate:	50 mL/min

Retention time (relative to chlorpyrifos) of ethion: 3.90
Detector: ^{63}Ni ECD
Temperature: 350°
Other Conditions: None
Detector response to 0.15 ng chlorpyrifos:50% FSD
(marker compound)

Behavior of SAN-835H:
Retention time (relative to chlorpyrifos): Could not be determined.
ng required for 50% FSD: Could not be determined.

Information submitted by: Fred Claussen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
Date: June 9, 1997

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REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical Phthalazinone:M1 on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: Phthalazinone:M1
Alternative Names:
8-methyl-5(6H)-pyrido[2,3-d]pyridazinone
Reference Standard: Phthalazinone:M1, lot number RS-M1-051895
Molecular Formula: $C_9H_8N_2O$
Structure:

Comments: An additional peak (unknown #1) yielding a peak response >5% FSD was observed near 9 minutes. The relative retention time to chlorpyrifos for this peak was 1.66 minutes. This peak was not observed in the chromatography obtained on other systems. The nature and source of the compound producing this peak are not known. All retention times were measured from the time of injection, not from the solvent front.

RESULTS FOR DG MODULE (SECTION 302): DG18 Level I
Standard reference material dissolved in: Acetone
Brief details about GLC system used:
Column: DB-225 (J&W Scientific) at 195°C

Length:	30 m
id:	0.53 mm
Film Thickness:	1.0 μm
Carrier gas:	He
Flow rate:	20 mL/min
Makeup gas:	P5
Flow rate:	50 mL/min

Retention time (relative to chlorpyrifos) of ethion: 3.90
Detector: ^{63}Ni ECD
Temperature: 350°
Other Conditions: None
Detector response to 0.15 ng chlorpyrifos: 50% FSD

Behavior of Phthalazinone:M1:
Retention time (relative to chlorpyrifos): 0.68
ng required for 50% FSD: 105

Information submitted by: Fred Clausen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
Date: June 9, 1997

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REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical methylated SAN-835H on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: Methylated SAN-835H
Alternative Names: Not Applicable
Reference Standard: SAN-835H, lot number RS-835-070296
Molecular Formula: Unknown
Structure: Unknown

Comments: SAN-835H derivatized using 1M TBAH in MeOH according to PAM Volume I. Section 402C1b, pp. 402-21 through 402-22. Florisil cleanup was not used. No difference between reagent blank and methylated standard chromatographic profiles was observed. Therefore, it was concluded that the reaction products observed in the chromatograms from modules DG1 and DG13 did not elute using the Level I GC parameters listed below.

RESULTS FOR DG MODULE (SECTION 302): DG18 Level I
Standard reference material dissolved in: Hexane
Brief details about GLC system used:
Column: DB-225 (J&W Scientific) at 195°C

Length:	30 m
id:	0.53 mm
Film Thickness:	1.0 μm
Carrier gas:	He
Flow rate:	20 mL/min
Makeup gas:	P5
Flow rate:	50 mL/min

Retention time (relative to chlorpyrifos) of ethion: 3.90

Detector: ^{63}Ni ECD
Temperature: 350°
Other Conditions: None
Detector response to 0.15 ng chlorpyrifos:50% FSD
(reader compound)

Behavior of methylated SAN-835H:
Retention time (relative to chlorpyrifos): Could not be determined.
ng required for 50% FSD: Could not be determined.

Information submitted by: Fred Claussen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
Date: June 9, 1997

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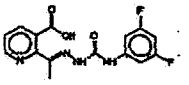
REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical SAN-835H on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: SAN-835H (BAS 654 H)
Alternative Names: Diflufenzopyr
(2-(methyl(((3,5-difluorophenylamino)carbonyl)hydrazono)methyl)-3-pyridine-carboxylic acid)
Reference Standard: SAN-835H, lot number RS-835-070296
Molecular Formula: $C_{15}H_{12}N_4O_3F_2$
Structure:



Comments: Compound did not chromatograph on Level I system as described below. SAN-835H degraded to M1 upon injection into the gas chromatograph. All retention times were measured from the time of injection, not from the solvent front.

RESULTS FOR DG MODULE (SECTION 302): DG5 Level I
Standard reference material dissolved in: Acetone
Brief details about GLC system used:
Column: HP-1 (Hewlett-Packard) at 185°C

Length:	30 m
id:	0.53 mm
Film Thickness:	0.88 μm
Carrier gas:	He
Flow rate:	15 mL/min
Makeup gas:	N_2
Flow rate:	~10 mL/min

Retention time (relative to chlorpyrifos) of ethion: 2.56

Detector: N/P
Temperature: 300°
Other Conditions: H_2 N/P flow rate at 4.7 mL/min. Air N/P flow rate at about 140 mL/min.
Detector response to 1.5 ng chlorpyrifos:50% FSD
(reader comprised)

Behavior of SAN-835H:
Retention time (relative to chlorpyrifos): Could not be determined.
ng required for 50% FSD: Could not be determined.

Information submitted by: Fred Claassen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537

Phone: (217) 963-2143
Date: June 9, 1997

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REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical Phthalazinone:M1 on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: Phthalazinone:M1
Alternative Names:
8-methyl-5(6H)-pyrido[2,3-d]pyridazinone
Reference Standard: Phthalazinone:M1, lot number RS-M1-051895
Molecular Formula: $C_8H_7N_3O$
Structure:

Comments: All retention times were measured from the time of injection, not from the solvent front.

RESULTS FOR DG MODULE (SECTION 302): DG5 Level I
Standard reference material dissolved in: Acetone
Brief details about GLC system used:
Column: HP-1 (Hewlett-Packard) at 185°C

Length:	30 m
id:	0.53 mm
Film Thickness:	0.88 μm
Carrier gas:	He
Flow rate:	15 mL/min
Makeup gas:	N_2
Flow rate:	~10 mL/min

Retention time (relative to chlorpyrifos) of ethion: 2.56

Detector: N/P
Temperature: 300°
Other Conditions: H_2 N/P flow rate at 4.7 mL/min. Air N/P flow rate at about 140 mL/min.
Detector response to 1.5 ng chlorpyrifos:50% FSD
(reactive component)

Behavior of Phthalazinone:M1:
Retention time (relative to chlorpyrifos): 0.33
ng required for 50% FSD: 13

Information submitted by: Fred Clausen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
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REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical methylated SAN-835H on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: Methylated SAN-835H
Alternative Names: Not Applicable
Reference Standard: SAN-835H, lot number RS-835-070296
Molecular Formula: Unknown
Structure: Unknown

Comments: SAN-835H derivatized using 1M TBAH in MeOH according to PAM Volume I, Section 402C1b, pp. 402-21 through 402-22. Florisil cleanup was not used. All retention times were measured from the time of injection, not from the solvent front. Three peaks were observed in the standard chromatogram that did not appear in the reagent blank. The peak at 13.38 min is reported below since it was also reported for the ECD system. However, a peak at 1.18 min (rt_r=0.30) yielded at response of approximately 50% FSD. This peak was not observed using the ECD system (see DGI Level I).

RESULTS FOR DG MODULE (SECTION 302): DG5 Level I
Standard reference material dissolved in: Hexane
Brief details about GLC system used:
Column: HP-1 (Hewlett-Packard) at 185°C

Length:	30 m
id:	0.53 mm
Film Thickness:	0.88 μm
Carrier gas:	He
Flow rate:	15 mL/min
Makeup gas:	N_2
Flow rate:	~10 mL/min

Retention time (relative to chlorpyrifos) of ethion: 2.56

Detector: N/P
Temperature: 300°
Other Conditions: H_2 N/P flow rate at 4.7 mL/min. Air N/P flow rate at about 140 mL/min.
Detector response to 1.5 ng chlorpyrifos:50% FSD
(analyte component)

Behavior of methylated SAN-835H:
Retention time (relative to chlorpyrifos): 3.41
ng required for 50% FSD: Could not be determined. Injection of approximately 1000 ng (SAN-835H equivalent) resulted in a peak response <50% FSD.

Information submitted by: Fred Claussen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
Date: June 9, 1997

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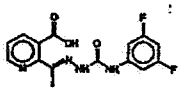
REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical SAN-835H on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: SAN-835H (BAS 654 H)
Alternative Names: Diflufenzopyr
(2-(methyl(((3,5-difluorophenylamino)carbonyl)hydrazono)methyl)-3-pyridine-carboxylic acid)
Reference Standard: SAN-835H, lot number RS-835-070296
Molecular Formula: $C_{15}H_{12}N_4O_3F_2$
Structure:



Comments: Compound did not chromatograph on Level I system as described below. SAN-835H degraded to M1 upon injection into the gas chromatograph. All retention times were measured from the time of injection, not from the solvent front.

RESULTS FOR DG MODULE (SECTION 302): DG17 Level I
Standard reference material dissolved in: Acetone
Brief details about GLC system used:
Column: DB-17 (J&W Scientific) at 190°C

Length:	30 m
id:	0.53 mm
Film Thickness:	1.0 μm
Carrier gas:	He
Flow rate:	40 mL/min
Makeup gas:	N_2
Flow rate:	~10 mL/min

Retention time (relative to chlorpyrifos) of ethion: 3.33

Detector: N/P
Temperature: 300°
Other Conditions: H_2 N/P flow rate at 4.7 mL/min. Air N/P flow rate at about 140 mL/min.
Detector response to 1.5 ng chlorpyrifos: 50% FSD
(under evaluation)

Behavior of SAN-835H:
Retention time (relative to chlorpyrifos): Could not be determined.
ng required for 50% FSD: Could not be determined.

Information submitted by: Fred Claussen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
Date: June 9, 1997

REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical Phthalazinone:M1 on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: Phthalazinone:M1
Alternative Names:
8-methyl-5(6H)-pyrido[2,3-d]pyridazinone
Reference Standard: Phthalazinone:M1, lot number RS-M1-051895
Molecular Formula: $C_9H_9N_3O$
Structure:

Comments: All retention times were measured from the time of injection, not from the solvent front.

RESULTS FOR DG MODULE (SECTION 302): DG17 Level I
Standard reference material dissolved in: Acetone
Brief details about GLC system used:
Column: DB-17 (J&W Scientific) at 190°C

Length:	30 m
id:	0.53 mm
Film Thickness:	1.0 µm
Carrier gas:	He
Flow rate:	40 mL/min
Makeup gas:	N_2
Flow rate:	~10 mL/min

Retention time (relative to chlorpyrifos) of ethion: 3.33

Detector: N/P
Temperature: 300°
Other Conditions: H_2 N/P flow rate at 4.7 mL/min. Air N/P flow rate at about 140 mL/min.
Detector response to 1.5 ng chlorpyrifos: 50% FSD
(reader comment)

Behavior of Phthalazinone:M1:
Retention time (relative to chlorpyrifos): 0.40
ng required for 50% FSD: 14

Information submitted by: Fred Claussen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
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Phone: (217) 963-2143
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REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical methylated SAN-835H on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: Methylated SAN-835H
Alternative Names: Not Applicable
Reference Standard: SAN-835H, lot number RS-835-070296
Molecular Formula: Unknown
Structure: Unknown

Comments: SAN-835H derivatized using 1M TBAH in MeOH according to PAM Volume I. Section 402C1b, pp. 402-21 through 402-22. Florisil cleanup was not used. All retention times were measured from the time of injection, not from the solvent front. As with other GC systems, multiple peaks were observed in the standard chromatogram that did not appear in the reagent blank. The peak reported below appears to be the same peak appearing on the equivalent ECD system. The presence of a very large, broad peak near 2 min indicates the possibility of multiple products formed during the reaction with TBAH. A sharper peak (methyl peak 1) emerging from this broad peak had a rrt_1 of 0.31 and yielded a response roughly equivalent to 75% FSD. This peak was not observed for the equivalent ECD system.

RESULTS FOR DG MODULE (SECTION 302): DG17 Level I
Standard reference material dissolved in: Hexane
Brief details about GLC system used:
Column: DB-17 (J&W Scientific) at 190°C

Length:	30 m
id:	0.53 mm
Film Thickness:	1.0 μm
Carrier gas:	He
Flow rate:	40 mL/min
Makeup gas:	N_2
Flow rate:	~10 mL/min

Retention time (relative to chlorpyrifos) of ethion: 3.33

Detector: N/P
Temperature: 300°
Other Conditions: H_2 N/P flow rate at 4.7 mL/min. Air N/P flow rate at about 140 mL/min.
Detector response to 1.5 ng chlorpyrifos:50% FSD
(marker compound)

Behavior of methylated SAN-835H:
Retention time (relative to chlorpyrifos): 5.72
ng required for 50% FSD: Could not be determined. Injection of approximately 1000 ng (SAN-835H equivalent) resulted in a peak response <50% FSD.

Information submitted by: Fred Claussen
Address: EPL-Bio-Analytical Services, Inc.
P.O. Box 109
Harristown, IL 62537
Phone: (217) 963-2143
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