US EPA ARCHIVE DOCUMENT

ATTACHMENT 2 BB

Battelle Study Number SC900078
Zoecon Study Number 1538

419971- 01

FINAL REPORT

STUDY TITLE

METHOD DEVELOPMENT AND VALIDATION OF PROPETAMPHOS RESIDUE ANALYSIS IN FOOD COMMODITIES

DATA REQUIREMENTS

Environmental Protection Agency Guidelines
Subdivision O, Series 171
Section 171-4 (C)(5)

AUTHORS

Mark G. Schweitzer and Stephen J. Summer

STUDY COMPLETED ON

April 29, 1991

PERFORMED BY

Battelle
505 King Avenue
Columbus, OH 43201-2693

LABORATORY STUDY NUMBER

Battelle Study Number SC900078

SPONSOR

Zoecon Corporation
12200 Denton Drive
Dallas, TX 75234

Battelle Study Number SC900078
Zoecon Study Number 1538

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

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

Company: Zoecon Corporation

Company Agent: Kelly J. Parker

Title: Regulatory Specialist

Signature: Kelly J. Parker

Date: 21 August 1991

Battelle Study Number SC900078
Zoecon Study Number 1538

GOOD LABORATORY PRACTICES STATEMENT

This study was conducted according to the principles of Good Laboratory Practices and is in compliance with the United States Environmental Protection Agency's Federal Insecticide, Fungicide, and Rodenticide Act, Good Laboratory Practice Regulations, 40 CFR Part 160.

Mark G. Schweitzer, Ph.D.
Study Director

4/29/91
Date

SPONSOR CERTIFICATION OF GOOD LABORATORY PRACTICE

To the best of my knowledge, the Good Laboratory Practice Statement signed by the study director is truthful and accurate.

Sponsor
Janet Lephart

6 May 91
Date

Submitter
Kelly J. Parker

21 August 1991
Date

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QUALITY ASSURANCE STATEMENT

This study was inspected by the Quality Assurance Unit and reports were submitted to management and the study director as follows:

Phase	Inspection Date	Date Reported to Management
Protocol Review	10/03/90	N/R
Pre-Study Meeting	02/26/91	N/R
Sample Homogenization of Sample Matrix	02/27/91	N/R
Sample Extraction	02/28/91	N/R
Analysis of Sugar Sample Extracts	03/20/91	N/R
Study File Audit	04/17/91	04/26/91
Data Audit, Draft Final Report Audit	04/17/91	04/26/91
Final Report Audit	04/29/91	N/R

N/R = not required.

To the best of my knowledge, the methods described were the methods followed and the data presented accurately represent data generated during the study.

Sandra M. Anderson 29 April 1991
Quality Assurance Unit Date
Health and Environment Group

STUDY INFORMATION

BATTELLE STUDY NUMBER:	SC900078
STUDY TITLE:	Method Development and Validation of Propetamphos Residue Analysis in Food Commodities
TEST MATERIAL:	Propetamphos
STUDY DIRECTOR:	Mark G. Schweitzer
SPONSOR'S STUDY MONITOR:	Janet Lephart
TESTING FACILITY:	Battelle 505 King Avenue Columbus, OH 43201-2693
SPONSOR:	Zoecon Corporation 12200 Denton Drive Dallas, TX 75234
INITIATION DATE:	October 3, 1990
COMPLETION DATE:	April 29, 1991
STUDY PERSONNEL:	Mark G. Schweitzer Stephen J. Summer Robert W. Atkinson

Battelle Study Number SC900078
Zoecon Study Number 1538

STUDY CERTIFICATION STATEMENT

The data presented in this report accurately reflect the data generated during the study.

Mark G. Schweitzer, Ph.D.
Study Director

4/29/91
Date

Study Contact: Mark G. Schweitzer
Manager, Residue Chemistry Department
Battelle
505 King Avenue
Columbus, Ohio 43201
(614) 424-3616

SPONSOR CERTIFICATION

This report is an exact, unaltered copy of the report submitted by Battelle.

Sponsor Representative

30 April 91
Date

Submitter

Date

A. ABSTRACT

The purpose of this study was to develop and validate an analytical method for the determination of propetamphos residue in various food commodities. The actual commodities examined were apples, beer, bologna, bread, butter, flour, hamburger, lettuce, macaroni, milk, Rice Krispies®, and sugar. The method is basically two sub-methods; an analysis of propetamphos residue from non-liquid food commodities and propetamphos residue from liquid commodities. The non-liquid food commodities were homogenized in the presence of extraction solvent (either acetonitrile or acetone), filtered, centrifuged, dehydrated, filtered again, and analyzed by gas chromatography with a mass selective detector (GC/MSD). The liquid food commodities were adsorbed directly on an Extrelut column, eluted with hexane, filtered, and analyzed by GC/MSD.

In the validation of the method, untreated samples of each commodity were obtained from a local supermarket, were homogenized, and fortified in triplicate with propetamphos at concentrations of 0.1 and 0.3 ppm. The fortification recoveries are summarized in Table I. All recoveries fell in the range of 70-120% for each commodity. A typical sample set, consisting of two controls and six treated samples, can be completed by an analyst in approximately eight hours.

B. MATERIALS

Test Substance

The test substance was propetamphos (CAS Registry No. 31218-83-4). The propetamphos used in this study (lot number PK024, assay value 91.15%), was a liquid as provided by Zoecon. When not in use, the test substance was stored at approximately -20°C. Documentation of the stability of propetamphos is maintained by Zoecon.

Food Commodities

All of the food commodities were obtained from the Big Bear Supermarket, Fifth Avenue, Columbus, Ohio. Table II provides a summary of the commodities used in the validation study. When not in use, the food commodities were stored at approximately -20°C.

Reagents

1. Acetonitrile, Burdick and Jackson, distilled in glass, UV grade.
2. Acetone, Burdick and Jackson, distilled in glass, UV grade.
3. Hexane, Burdick and Jackson, distilled in glass, UV grade.
4. Anhydrous Sodium Sulfate, Baker, reagent grade.

Glassware

1. Buchner funnels, 76 mm.
2. Volumetric flasks, 500 mL, 200 mL, and 100 mL.
3. Vacuum flasks, 250 mL.
4. Extraction Bottles, Nalgene, wide-mouth, 8 ounce.
5. Glass sample vials, 4-dram and 1-dram, screw cap with teflon liners.
6. Glass GC Autosampler vials, Hewlett Packard, Crimptop, 11 mm.
7. 4 oz amber glass bottle with teflon-lined screw cap.

Equipment

1. Finnpipets, Cole-Parmer, 200-1000 μL and 0-200 μL adjustable (or equivalent).
2. Polytron Homogenizer, Brinkman Model PT 10/35 (or equivalent).
3. Sar Vac vacuum pump, Seargent Welch, Model 8803 (or equivalent).
4. Hobart Food Cutter, Model 8186 (or equivalent).
5. IEC Model K Centrifuge (or equivalent).

6. Extrelut QE columns, 20 mL, EM Science.
7. Glass fiber filters, Gelman Type A/E, 76 mm.
8. Disposable polypropylene plastic syringes (Catalog No. 201-640), Sunbrokers, 25 mL.
9. Filter Disks, Gelman Acrodisc, 0.2 micron, 25 mm.

Instrumentation

1. Hewlett Packard 5890 Series II gas chromatograph with splitless injector.
2. Hewlett Packard 7673 autoinjector.
3. Hewlett Packard 5971A mass selective detector.
4. Vectra 05/20 computer with VGA monitor and keyboard.
5. Windows 386 and Microsoft Chem Station software package (Revision A.00.00).
6. Hewlett Packard Laser Jet III Printer.

C. METHOD

Standard Preparation

Propetamphos Stock Solution. The propetamphos reference standard (0.050 g) was accurately weighed into a 1-dram vial. The propetamphos was diluted with 2 mL of hexane and quantitatively transferred using a pasteur pipet to a 500 mL volumetric flask. The 1-dram vial was used three more times with 2 mL portions of hexane; these rinses were added to the 500 mL volumetric flask. The solution was diluted to the mark with hexane. The final concentration was 100 µg/mL.

Propetamphos Working Stock Solution. The propetamphos stock solution (10 mL) was pipetted into a 100 mL volumetric flask and brought

up to the mark with hexane. The final concentration was 10 µg/mL or 10,000 ng/mL.

Propetamphos Standard Solutions. The appropriate volumes of the propetamphos working stock (10,000 ng/mL) was pipetted into 100 mL volumetric flasks to yield the final concentrations of 1, 5, 10, 25, 50, and 100 ng/mL. The following table shows the volumes to be used. The volumetrics were diluted to the mark with hexane.

mL of Working Stock	mL Diluted to	Solvent	Final Concentration
1.000	100	Hexane	100 ng/mL
0.500	100	Hexane	50 ng/mL
0.250	100	Hexane	25 ng/mL
0.100	100	Hexane	10 ng/mL
0.050	100	Hexane	5 ng/mL
0.010	100	Hexane	1 ng/mL

Fortification Procedure

Method recovery fortifications were prepared by adding an aliquot of a propetamphos stock solution to each sample matrix. Each 25 g solid sample (20 g liquid sample) was fortified with a stock solution of propetamphos in hexane. The stock solution used to fortify the samples was the propetamphos stock solution (100 µg/mL in hexane). For each sample, an appropriate aliquot of the stock solution was added to the pre-weighed sample using a calibrated automatic pipet. The sample was fortified immediately prior to extraction by evenly distributing the stock solution over the surface of the sample.

Sample Preparation For Non-Liquid Commodities

Homogenization. The entire sample matrix was homogenized with dry ice using a Hobart food cutter. The homogenized sample matrix was then placed into plastic bags which were loosely tied to allow CO_2 sublimation in the freezer.

Extraction Procedure. A 25-gram portion of the homogenized sample matrix was quantitatively weighed into a Nalgene, 8-ounce, wide-mouth bottle. For most non-liquid commodities, 60 mL of acetonitrile was added to the extraction bottle and the sample was homogenized with a Polytron for 1 minute. For foods high in sugar, such as sugar itself or Rice Krispies®, 60 mL of acetone was used as the extraction solvent. The extraction bottle was capped and centrifuged for 20 minutes at a speed control setting of 45 to 50 (approximately 3,000 xg). The extract was decanted into a Buchner funnel which had a 76 mm glass fiber filter in place. Using vacuum, the extract was filtered into a 250 mL vacuum flask. An additional 60 mL of acetonitrile (or acetone when appropriate) was added to the extraction bottle and re-extracted, centrifuged, and decanted. This procedure was repeated a third time and all three 60 mL portions were combined in the vacuum flask. The glass fiber filter was rinsed with a small portion of acetonitrile (or acetone when appropriate) into the vacuum flask. The entire extract from the vacuum flask was poured into a 200 mL volumetric flask and diluted to the mark with acetonitrile (or acetone when appropriate). The sample was returned to the original vacuum flask to which had been added ~10 grams of anhydrous sodium sulfate. A 10-20 mL aliquot of this dried sample was removed from the vacuum flask and filtered into a 4-dram vial, using a 0.2 μm syringe filter attached to a 25 mL solvent resistant plastic syringe. The samples were stored in the freezer at approximately -20°C until analyzed.

Sample Preparation For Liquid Commodities

Extraction Procedure. To ensure a homogenous sample, the liquid was shaken and a 20-gram portion of the sample was weighed into a Nalgene, 8-ounce, wide-mouth bottle. The sample was quantitatively transferred onto a dry Extrelut column and absorbed onto the column packing. The Extrelut column was allowed to dry for 30 minutes. The sample was eluted from the column directly into a 200 mL volumetric flask with two, 100 mL portions of hexane. A portion of the hexane was first added to the wide-mouth bottle to ensure quantitative removal of the sample. The hexane washings were added to the Extrelut column. The volumetric flask was diluted to the mark with hexane. A 10-20 mL aliquot of this sample was removed from the volumetric flask and filtered into a 4-dram vial using a 0.2 µm syringe filter attached to a 25 mL solvent resistant plastic syringe. Samples were stored in the freezer at approximately -20°C until time for analysis by gas chromatography.

Analysis

Chromatographic Analysis

Gas Chromatography Parameters

GC Column:	DB-5, 30 m x 0.25 mm capillary column, 0.10 µm film thickness (J&W Scientific, Folsom, California)
GC Detector:	Mass selective detector, SIM run monitoring m/z 138 as the major ion and m/z 194 as the qualifier ion.
Temperature Program:	Initial Temperature = 60°C Initial Time = 1.0 minutes Rate = 20°C/minutes

	Final Temperature = 280°C
	Final Time = 8.0 minutes
	Run Time = 20 minutes
Detector Temperature:	280°C
Injector Temperature:	250°C
Injector Mode:	Splitless injector (Grob)
	Purge: on at 0.75 minutes
	Liner: Splitless Silica for HP7673 Injection
Gas Flows:	Carrier gas (He): 60 cm/sec
	Column Head Pressure: 5.5 psi
Retention Time:	Carrier flows should be optimized for the instrument and should achieve a relative retention time of 10 minutes for the compound of interest.
Injection Volume:	2 μL

Chromatographic Analysis Procedure. The GC system was conditioned with triplicate injections of the matrix blank extracts prior to the analysis of each commodity to stabilize the system so that the propetamphos response remained constant. The calibration standards were injected in duplicate followed by the matrix extracts in duplicate. Quality control standards were injected no later than after every fifth sample to ensure chromatographic integrity. Calibration curves were run with each new matrix on each day of analysis.

Calculations. A calibration curve was constructed by plotting the peak area versus the actual propetamphos concentration for each standard. The concentration of propetamphos in the sample was calculated using the following equations:

$$\text{Propetamphos conc., } \mu g/g = \left(\frac{\text{Peak Area}}{M}\right)\left(\frac{\text{Final Volume}}{\text{Initial Sample Weight, g}}\right)$$

where M is the slope of the regression line produced from a linear regression analysis of the peak area versus propetamphos concentration. The regression analysis was constrained through the origin. LOTUS 1-2-3 (v. 2.2) was used to prepare data summaries.

Sample Identification

All samples were labelled with the Battelle study number, laboratory record book identification code, fortification level, date of preparation, and the analyst's initials.

D. VALIDATION RESULTS

Propetamphos was fortified into each of the homogenized food commodities at two levels, 0.1 and 0.3 ppm. The mean recoveries of all food matrices were within the 70-120% range with relative standard deviations ≤15% as shown in Table I. The use of a mass selective detector provided excellent sensitivity and specificity. No interferences were observed in any commodity, with the exception of Rice Krispies®. Rice Krispies® showed a low level interference with a measured concentration of approximately 0.02 ppm. All other control samples contained no detectable residues of propetamphos at a method sensitivity of 0.01 ppm. Sample chromatograms and detailed analytical data for each commodity can be found in the Appendices.

The method sensitivity was calculated based on the lowest detectable standard. The lowest detectable standard had a propetamphos concentration of 1 ng/mL. Assuming a sample size of 25 g, and a final

extract volume of 200 mL, the lowest detectable concentration (LDC) of propetamphos is calculated using the equation shown below:

$$LDC = \frac{(\text{Lowest Detectable Standard})(\text{Final Volume})}{(\text{Sample Weight})}$$

$$LDC = \frac{(1\ \text{ng/mL})(200\ \text{mL})}{25\ \text{g}}$$

$$= \frac{8\ \text{ng}}{\text{g}} = 0.008\ \mu\text{g/g} = 0.008\ \text{ppm}$$

E. CONCLUSIONS

The analytical method developed and validated in this study is a simple and accurate method for the analysis of propetamphos in food commodities. The use of a mass selective detector provided exceptional selectivity and sensitivity.

F. RECOMMENDATIONS

During the course of the study, it was determined that the use of the following recommendations prevent erosion in the method sensitivity:

1. Because this method has no "clean-up" procedures, it is recommended that the MSD source be placed on a routine maintenance and cleaning program after a total of approximately 100 sample injections.

2. As part of the routine maintenance of the source, the injector liner should be changed as well as the removal of the first 1-2 inches of the capillary column.

3. The use of ethylacetate should be avoided as it causes significant deterioration of the detector sensitivity.

4. A modified splitless injector liner (silica containing a glass wool plug) should be used with an autosampler.

5. All glassware used during the study, with the exception of the volumetric glassware should be dried/conditioned in a muffle oven prior to use. This eliminates the potential of carry over from sample to sample.

6. Whenever possible, the supplies such as reagents and instrumentation listed in this method (or their equivalent) should be used for this analysis.

6. ARCHIVE STATEMENT

Certified copies of the raw data will be forwarded to Zoecon on completion of the study. The final report, protocol, amendments, and deviations will be archived at Battelle and Zoecon according to their respective standard operating procedures. Battelle will maintain, but not forward the following information: personnel files, standard operating procedures, instrument log books, and any other data relative to this study.

TABLE I. SUMMARY OF FORTIFICATION RECOVERIES

	Fortification Level					
	0.1 ppm			0.3 ppm		
Commodity	Individual Recoveries, %	Mean Recovery, %	RSD,[1] %	Individual Recoveries, %	Mean Recovery, %	RSD, %
Apple	77.7 68.7 69.4	71.9	7.0	72.3 85.8 73.5	77.2	9.7
Beer	85.3 90.1 94.6	90.0	5.2	95.7 97.0 107.6	100.1	6.5
Bologna	72.7 80.3 88.0	80.3	9.5	88.3 93.8 92.4	91.5	3.1
Bread	89.8 78.7 77.4	81.9	8.3	84.4 91.7 90.5	88.8	4.4
Butter	91.8 106.6 99.2	99.2	7.4	106.7 105.7 111.2	107.9	2.7
Flour	74.0 74.5 83.2	77.3	6.7	70.2 87.2 86.9	81.5	11.9
Hamburger	77.6 100.3 102.0	93.3	14.6	82.3 85.0 81.4	82.9	2.3
Lettuce	100.8 82.8 84.5	89.4	11.1	95.5 93.3 92.3	93.7	1.3

[1] RSD = Relative standard deviation.

TABLE I. SUMMARY OF FORTIFICATION RECOVERIES (CONTINUED)

	Fortification Level					
	0.1 ppm			0.3 ppm		
Commodity	Individual Recoveries, %	Mean Recovery, %	RSD,[1] %	Individual Recoveries, %	Mean Recovery, %	RSD, %
Macaroni	77.4 70.5 80.8	76.3	6.9	69.8 73.1 76.5	73.1	4.6
Milk	98.9 112.0 107.8	106.2	6.3	106.2 108.8 112.2	109.0	2.8
Rice Krispies®[2]	94.0 112.1 109.0	105.1	9.2	116.1 110.7 125.3	117.4	6.3
Sugar	77.2 86.0 94.3	85.8	10.0	80.5 85.2 90.2	85.3	5.7

[1] RSD = Relative standard deviation.

[2] Recoveries for Rice Krispies® were corrected for the matrix blank. Correction to all other food matrices were not needed.

TABLE II. FOOD COMMODITY SUPPLIERS[1]

Commodity	Supplier/Manufacturer
Apples	(Red Delicious)
Beer	Anheuser-Busch (Bud Dry)
Bologna	Eckrich (All Beef)
White Bread	Wonder
Butter	Land-O-Lakes
Flour	Gold Medal
Hamburger	Big Bear (not less than 70% lean)
Lettuce	Top Fresh (Iceberg)
Macaroni	Meuller's (elbow macaroni)
Milk	Meyer Dairy (0.5% fat)
Rice Krispies®	Kellogg's
Sugar	Domino

[1] All commodities were purchased at the Big Bear Supermarket, Fifth Avenue, Columbus, Ohio.

APPENDIX 1

DETAILED ANALYTICAL DATA - APPLES

PROPETAMPHOS FOOD RESIDUE METHOD VALIDATION

DATA SUMMARY PAGE 1 OF 2

SC900078 LOTUS 123 V 2.2

ANALYST: S.J. SUMMER

INITIALS: DATE:

MATRIX: APPLES ANALYSIS DATE: MARCH 23,1991
STD CURVE ID #: 032391

CALIBRATION STANDARDS

SAMPLE DESCRIPTION	LRB NUMBER 45547-	PEAK AREA	AREA AVERAGE	CALC. CONC. NG/ML	PERCENT DEVIATION
1 NG/ML	4102	436	549	0.948352	5.164788
1 NG/ML	4103	662		1.439929	43.99291
5 NG/ML	4104	2302	2317	5.007125	0.142503
5 NG/ML	4105	2332		5.072378	1.447574
10 NG/ML	4106	4101	4092	8.920165	10.79834
10 NG/ML	4107	4083		8.881013	11.18986
25 NG/ML	4108	9797	9917	21.30964	14.76141
25 NG/ML	4109	10037		21.83167	12.67330
50 NG/ML	4110	22710	22146	49.39696	1.206071
50 NG/ML	4111	21582		46.94342	6.113140
100 NG/ML	4112	45518	46838	99.00709	0.992909
100 NG/ML	4113	48158		104.7494	4.749406

AVERAGE DEVIATION:9.436020

Regression Output:

Constant	0
Std Err of Y Est	1069.480
R Squared	0.996014
No. of Observations	12
Degrees of Freedom	11

X Coefficient(s) 459.7448
Std Err of Coef. 6.569515

THE CALCULATED CONCENTRATION OF PROPETAMPHOS IN THE SAMPLES IS DETERMINED BY THE FOLLOWING EQUATION:

PROPETAMPHOS CONC. (UG/ML) = (PEAK AREA)(X-COEFFICIENT)(MULTIPLIER)

MULTIPLIERS= 0.001 FOR QC STANDARD
0.008 FOR SAMPLE EXTRACTS

WHERE THE X-COEFFICIENT IS DETERMINED BY PERFORMING LINEAR REGRESSION ON THE ACTUAL STANDARD CONCENTRATION AND THE OBSERVED PEAK AREA. THE LINEAR REGRESSION IS CONSTRAINED THROUGH THE ORIGIN.

PROPETAMPHOS FOOD RESIDUE METHOD VALIDATION

DATA SUMMARY PAGE 2 OF 2
SC900078
INITIALS: DATE: MATRIX: APPLES

SAMPLE DESCRIPTION	LRB NUMBER 45547-	PEAK AREA	CONC. UG/G	EXPECTED CONC. UG/G	PERCENT RECOVERY	AVERAGE PERCENT RECOVERY
50 NG/ML STD.	4014	21961	0.047767	0.05	95.53559	89.31693
50 NG/ML STD.	4015	19102	0.041549	0.05	83.09826	
SOLVENT BLANK	4015	0	0	0	NA	
SOLVENT BLANK	4015	0	0	0	NA	
MATRIX BLANK	4014	0	0	0	NA	
MATRIX BLANK	4014	0	0	0	NA	
0.1 PPM SPIKE	4008	4049	0.070456	0.1	70.45647	77.49526
0.1 PPM SPIKE	4008	4881	0.084934	0.1	84.93406	
0.1 PPM SPIKE	4009	4077	0.070943	0.1	70.94369	68.67287
0.1 PPM SPIKE	4009	3816	0.066402	0.1	66.40204	
0.1 PPM SPIKE	4010	4039	0.070282	0.1	70.28246	69.41241
0.1 PPM SPIKE	4010	3935	0.068542	0.1	68.54236	
0.3 PPM SPIKE	4011	12349	0.214884	0.3	71.62813	72.29227
0.3 PPM SPIKE	4011	12578	0.218869	0.3	72.95640	
0.3 PPM SPIKE	4012	14463	0.251670	0.3	83.89000	85.34181
0.3 PPM SPIKE	4012	15136	0.263380	0.3	87.79362	
0.3 PPM SPIKE	4013	13781	0.239802	0.3	79.93419	73.53644
0.3 PPM SPIKE	4013	11575	0.201416	0.3	67.13868	

NA = NOT APPLICABLE

EXPECTED CONCENTRATION = (FORTIFICATION LEVEL, UG/G)(SAMPLE WT.)/(FINAL VOLUME)

SUMMARY OF RESULTS:

	AVERAGE RECOVERY	STANDARD DEVIATION	RELATIVE * STANDARD DEVIATION
0.1 PPM SPIKE	71.9	8.1	7.9
0.3 PPM SPIKE	77.2	7.5	9.7

* RELATIVE STANDARD DEVIATION = (STANDARD DEVIATION/AVERAGE) X 100

Page 1

Sequence Calibration Sample
File: D:\CHEMPC\DATA\APPLE\0901011.D
Operator: STEVE SUMMER
Date Acquired: 24 Mar 91 0:26 am
Method File Name: PROPET1.M
Sample Name: CAL STD 25 NG/ML EXT #45547-41-09
Misc Info: SC900078
Bottle Number: 9

34 3/28/91

PROPETAMPHOS (SC900078)
Calibration Last Updated: Sun Mar 24 00:20:25 1991
External Standard

Reference Window: 5 Percent
Non-Reference Window: 5 Percent
Correlation Window: 0.03 minutes
Default Multiplier: 1
Default Sample Amount: 0

Compound Information

*** Compound 1 ***

Name: PROPETAMPHOS ( )
Ret. Time 9.48 min., Extract & Integrate from 9.38 to 9.58 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 138			PROPET1.E
Q1 194	0	20	PROPET1.E

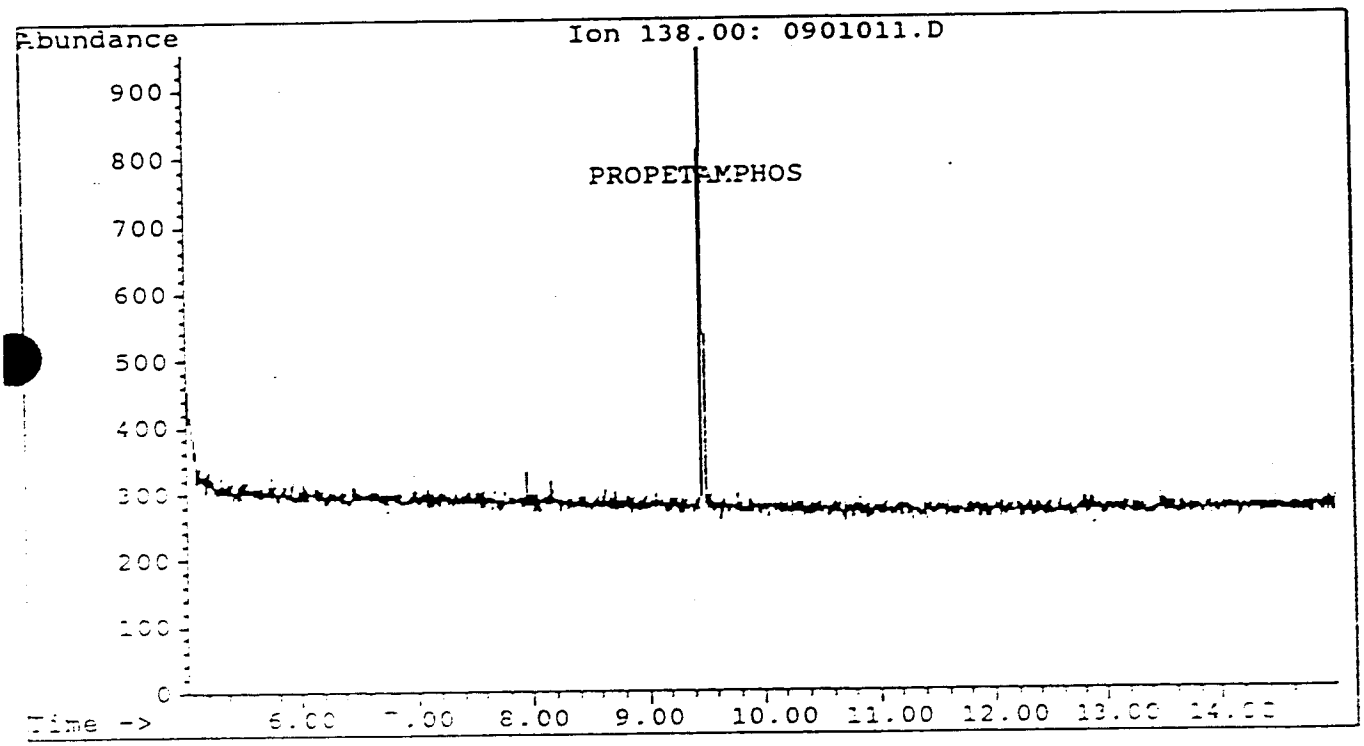
Lvl ID	Amt (UG/G)	Response
1	1	549
5	5	2317
10	10	4092
25	25	9917
50	50	28238
100	100	60049

Qualifier Peak Analysis ON
Curve Fit: Linear, Forced Through Origin

Battelle Study Number SC900078
Zoecon Study Number 1538

File: C:\CHEMPC\DATA\APPLE\0901011.D
Operator: STEVE SUMMER
Date Acquired: 24 Mar 91 0:26 am
Method File Name: PROPET1.M
Sample Name: CAL STD 25 NG/ML EXT #45547-41-09
Misc Info: SC900078
Bottle Number: 9

4/3/91



PROPETAMPHOS (SC900078)

Calibration Last Updated: Sun Mar 24 02:33:02 1991
External Standard Report - DETAILED

BA 3/28/91

Information from Data File:

File:	D:\CHEMPC\DATA\APPLE\1701019.D
Operator:	STEVE SUMMER
Date Acquired:	24 Mar 91 3:58 am
Method File Name:	PROPET1.M
Sample Name:	APPLE BLANK EXT #45547-40-14
Misc Info:	SC900078
Bottle Number:	17

Report Style:
Graphics: (Cn) Off
Compounds: (All) Single

Quantitation Settings:

Reference Peak Window:	5 Percent
Non-Reference Peak Window:	5 Percent
Correlation Window:	0.03 minutes
Default Multiplier:	0.008
Default Sample Amount:	0
Peak Type decoding:	* -> Time Reference Peak

Number of Compounds in Database: 1

Time Reference Peaks:

Pk#	Compound	Expected RT	Actual RT

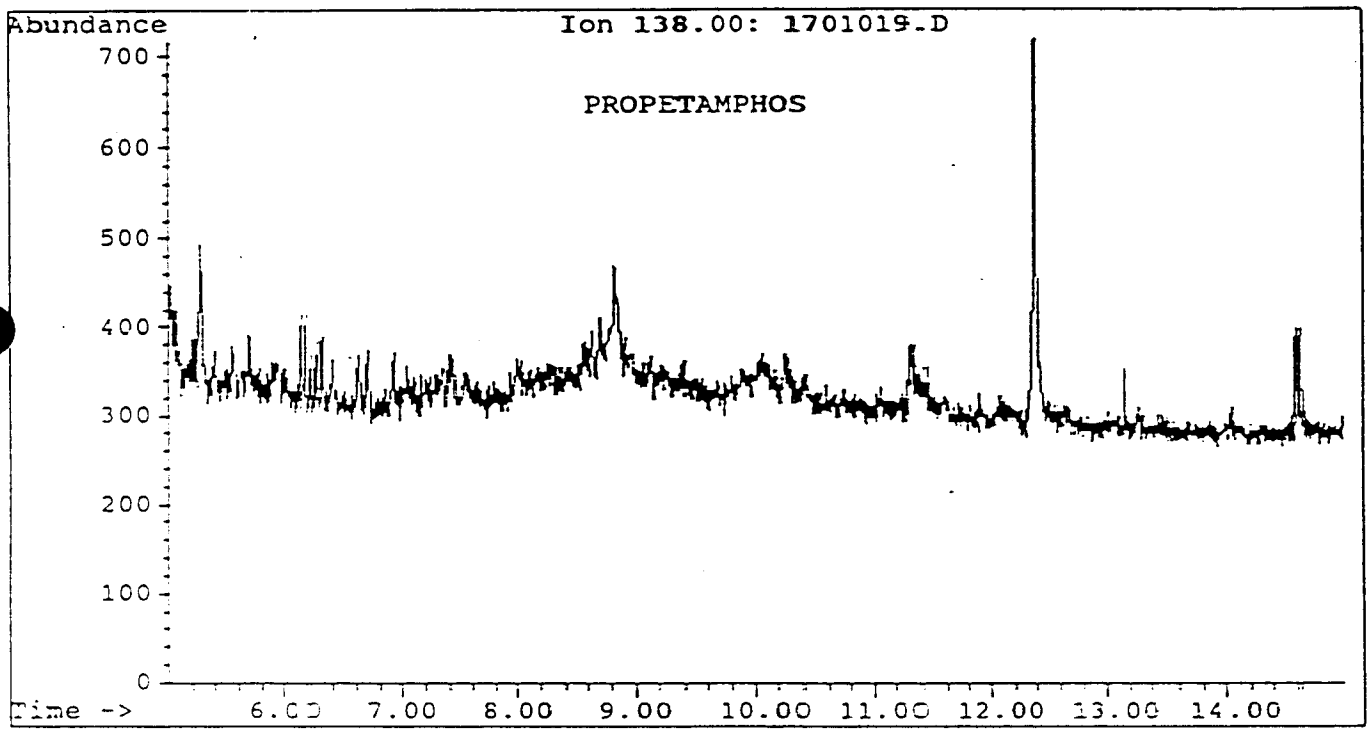
PROPETAMPHOS (SC900078)

File: D:\CHEMPC\DATA\APPLE\1701019.D
Operator: STEVE SUMMER
Date Acquired: 24 Mar 91 3:58 am 3/28/91
Method File Name: PROPET1.M
Sample Name: APPLE BLANK EXT #45547-40-14
Misc Info: SC900078
Bottle Number: 17

Compound: PROPETAMPHOS
Ret Time:
Amount:
Pk # and Type: 1
***** NOT FOUND ****

Battelle Study Number SC900078
Zoecon Study Number 1538

File: C:\CHEMPC\DATA\APPLE\1701019.D
Operator: STEVE SUMMER
Date Acquired: 24 Mar 91 3:58 am
Method File Name: PROPET1.M
Sample Name: APPLE BLANK EXT #45547-40-14
Misc Info: SC900078
Bottle Number: 17



PROPETAMPHOS (SC900078)

Calibration Last Updated: Sun Mar 24 02:33:02 1991
External Standard Report - DETAILED

BA 3/28/91

---

nformation from Data File:

File:	D:\CHEMPC\DATA\APPLE\1901021.D
Operator:	STEVE SUMMER
Date Acquired:	24 Mar 91 4:51 am
Method File Name:	PROPET1.M
Sample Name:	0.1 PPM APPLE SPIKE EXT #45547-40-08
Misc Info:	SC900078
Bottle Number:	19

---

eport Style:
Graphics: (On) Off
Compounds: (All) Single

---

uantitation Settings:

Reference Peak Window:	5 Percent
Non-Reference Peak Window:	5 Percent
Correlation Window:	0.03 minutes
Default Multiplier:	0.008
Default Sample Amount:	0
Peak Type decoding:	* -> Time Reference Peak

---

umber of Compounds in Database: 1

---

ime Reference Peaks:

Pk#	Compound	Expected RT	Actual RT

PROPETAMPHOS (SC900078)

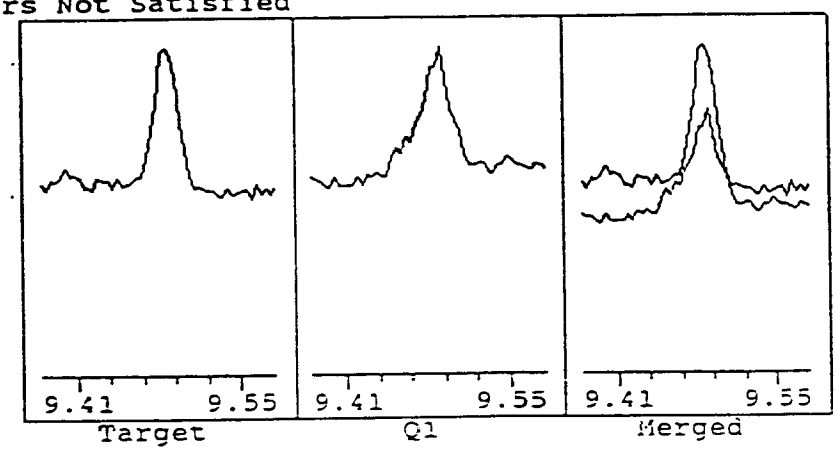
Battelle Study Number SC900078
Zoecon Study Number 1538

File: D:\CHEMPC\DATA\APPLE\1901021.D
Operator: STEVE SUMMER
Date Acquired: 24 Mar 91 4:51 am

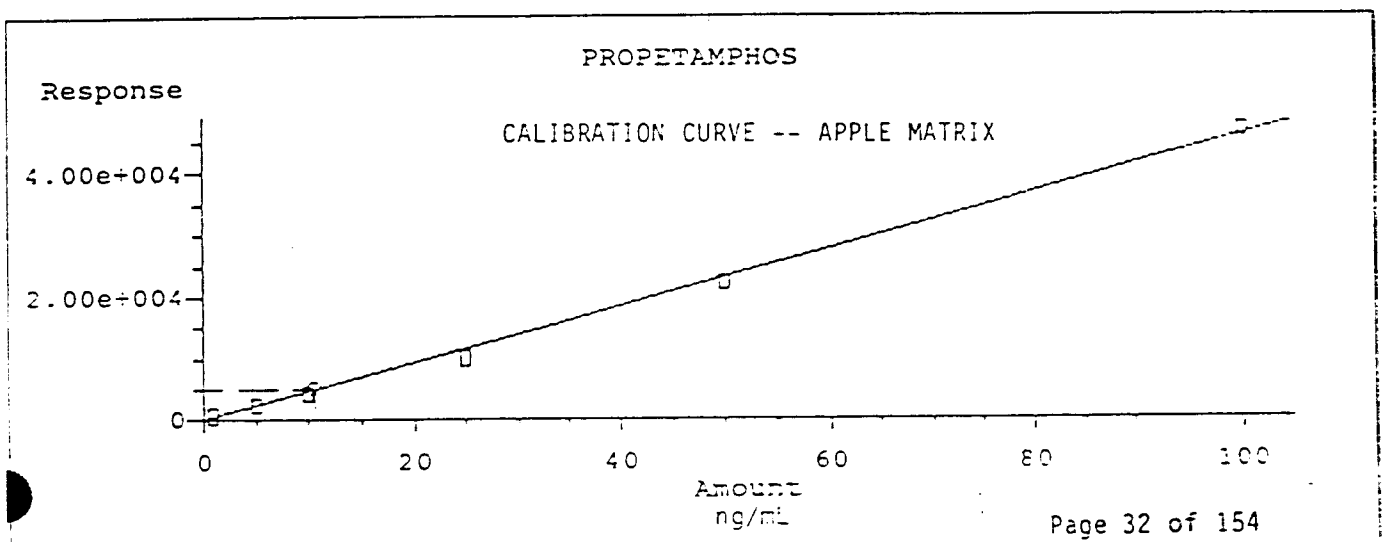
BA 3/28/91

Method File Name: PROPET1.M
Sample Name: 0.1 PPM APPLE SPIKE EXT #45547-40-08
Misc Info: SC900078
Bottle Number: 19

Compound: PROPETAMPHOS
Ret Time: 9.49
Amount: 0.08 UG/G
Pk # and Type: 1
Qualifiers Not Satisfied



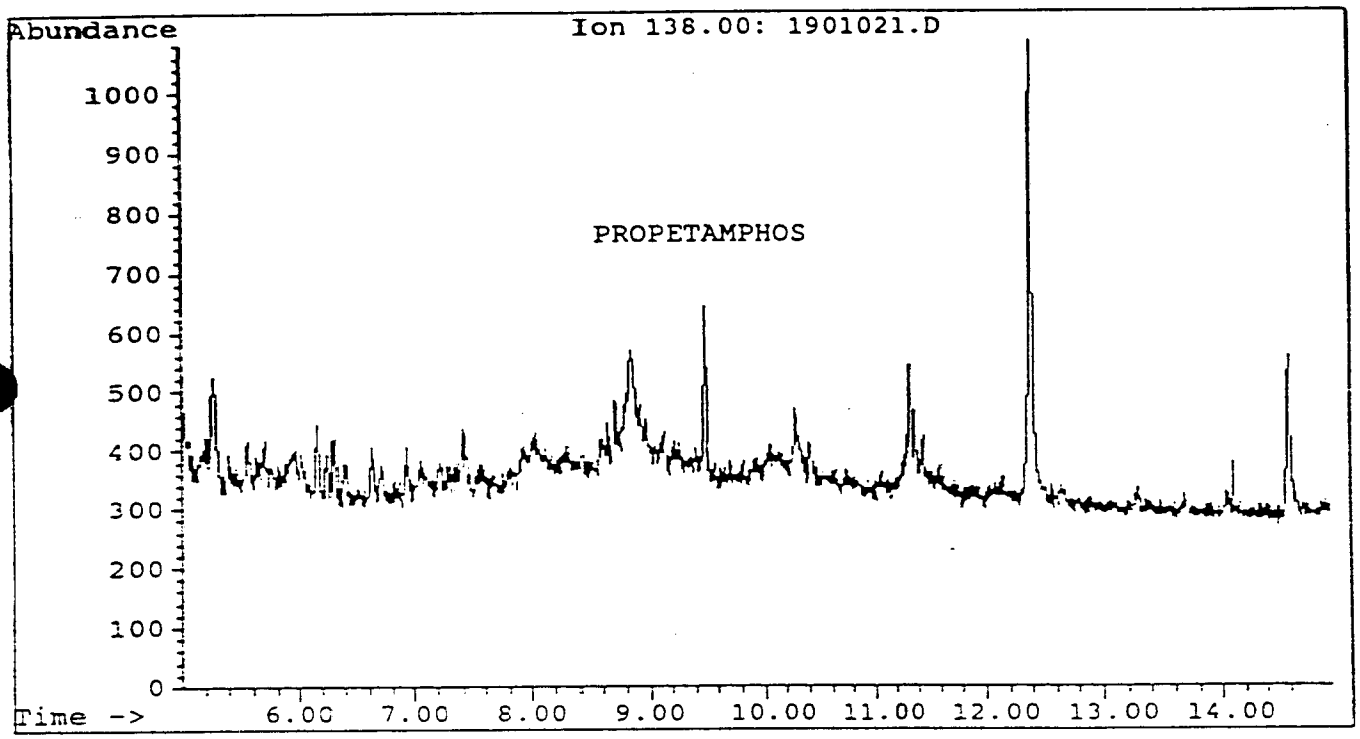
	Signal	Ratios	Limits	RT	Limits	Area	Integ Type
Tgt	138	100.0%		9.49	9.24	4881	PROPET1
Q1	194	86.0	0.0-0.0	9.49	to	4199	PROPET1
Q2	0				9.72		
Q3	0						



Response = 4.60e+002 * Amt
Corr Coef = 0.998 Curve Fit: Linear/Origin

File: C:\CHEMPC\DATA\APPLE\1901021.D
Operator: STEVE SUMMER
Date Acquired: 24 Mar 91 4:51 am
Method File Name: PROPET1.M
Sample Name: 0.1 PPM APPLE SPIKE EXT #45547-40-08
Misc Info: SC900078
Bottle Number: 19

4/3/91



APPENDIX 2

DETAILED ANALYTICAL DATA - BEER

PROPETAMPHOS FOOD RESIDUE METHOD VALIDATION

DATA SUMMARY PAGE 1 OF 2

SC900078 LOTUS 123 V 2.2

ANALYST: S.J. SUMMER

INITIALS: DATE:

MATRIX: BEER ANALYSIS DATE: MARCH 4, 1991
STD CURVE ID #: 030491

CALIBRATION STANDARDS

SAMPLE DESCRIPTION	LEB NUMBER 45547-	PEAK AREA	AREA AVERAGE	CALC. CONC. NG/ML	PERCENT DEVIATION
1 NG/ML	0802	429	429	0.762589	23.74102
1 NG/ML	0803	429		0.762589	23.74102
5 NG/ML	0804	2290	1836	4.070700	18.58555
5 NG/ML	0805	1382		2.456641	50.86727
10 NG/ML	0806	4726	4778	8.400930	15.99069
10 NG/ML	0807	4830		8.585801	14.14198
25 NG/ML	0808	12903	12454	22.93635	8.254583
25 NG/ML	0809	12005		21.34007	14.63971
50 NG/ML	0810	27638	25902	49.12926	1.741462
50 NG/ML	0811	24166		42.95744	14.08513
100 NG/ML	0812	56494	57906	100.4236	0.423652
100 NG/ML	0813	59318		105.4435	5.443551

AVERAGE DEVIATION:15.97133

Regression Output:

Constant	0
Std Err of Y Est	1777.833
R Squared	0.992855
No. of Observations	12
Degrees of Freedom	11
X Coefficient(s)	562.5567
Std Err of Coef.	10.91888

THE CALCULATED CONCENTRATION OF PROPETAMPHOS IN THE SAMPLES IS DETERMINED BY THE FOLLOWING EQUATION:

CONC. NG/ML = PEAK AREA/X-COEFFICIENT/MULTIPLIER

MULTIPLIERS = 1.000 FOR QC STANDARD
0.01 FOR SAMPLE EXTRACTS

WHERE THE X-COEFFICIENT IS DETERMINED BY PERFORMING LINEAR REGRESSION ON THE ACTUAL STANDARD CONCENTRATION AND THE OBSERVED PEAK AREA. THE LINEAR REGRESSION IS CONSTRAINED THROUGH THE ORIGIN.

PROPETAMPHOS FOOD RESIDUE METHOD VALIDATION

DATA SUMMARY PAGE 2 OF 2
SC900078
INITIALS: DATE: MATRIX: BEER

SAMPLE DESCRIPTION	LRB NUMBER 45547-	PEAK AREA	CONC. UG/G	EXPECTED CONC., UG/G	PERCENT RECOVERY	AVERAGE PERCENT RECOVERY
50 NG/ML STD.	0614	26591	0.047268	0.05	94.53624	92.12937
50 NG/ML STD.	0615	25237	0.044861	0.05	89.72250	
SOLVENT BLANK	0615	0	0	0	NA	
SOLVENT BLANK	0615	0	0	0	NA	
MATRIX BLANK	0614	0	0	0	NA	
MATRIX BLANK	0614	0	0	0	NA	
0.1 PPM SPIKE	0608	4636	0.082409	0.1	82.40946	85.33361
0.1 PPM SPIKE	0608	4965	0.088257	0.1	88.25776	
0.1 PPM SPIKE	0609	5193	0.092310	0.1	92.31069	90.12424
0.1 PPM SPIKE	0609	4947	0.087937	0.1	87.93780	
0.1 PPM SPIKE	0610	5248	0.093146	0.1	93.14616	94.61268
0.1 PPM SPIKE	0610	5405	0.096079	0.1	96.07920	
0.3 PPM SPIKE	0611	16591	0.286033	0.3	95.34446	95.73257
0.3 PPM SPIKE	0611	16222	0.288362	0.3	96.12067	
0.3 PPM SPIKE	0612	16086	0.285944	0.3	95.31483	96.99762
0.3 PPM SPIKE	0612	16654	0.296041	0.3	98.68042	
0.3 PPM SPIKE	0613	18181	0.321407	0.3	107.1358	107.5950
0.3 PPM SPIKE	0613	18236	0.324162	0.3	108.0542	

NA = NOT APPLICABLE

EXPECTED CONCENTRATION = (FORTIFICATION LEVEL, UG/G)(SAMPLE WT.)/(FINAL VOLUME)

SUMMARY OF RESULTS:

	AVERAGE RECOVERY	STANDARD DEVIATION	RELATIVE * STANDARD DEVIATION
0.1 PPM SPIKE	90.0	4.6	5.2
0.3 PPM SPIKE	100.1	6.5	6.5

* RELATIVE STANDARD DEVIATION = (STANDARD DEVIATION/AVERAGE) X 100

Sequence Calibration Sample
File: D:\CHEMPC\DATA\BEER\0901011.D
Operator: STEVE SUMMER
Date Acquired: 4 Mar 91 8:28 pm
Method File Name: PROPET1.M
Sample Name: CAL STD 25 NG/ML EXT #45547-08-09
Misc Info: SC900078
Bottle Number: 9

PROPETAMPHOS (SC900078)
Calibration Last Updated: Tue Mar 05 08:37:58 1991
External Standard

Reference Window: 5 Percent
Non-Reference Window: 5 Percent
Correlation Window: 0.03 minutes
Default Multiplier: 1
Default Sample Amount: 0

Compound Information

*** Compound 1 ***

Name: PROPETAMPHOS ( )
Ret. Time 9.55 min., Extract & Integrate from 9.45 to 9.65 min.

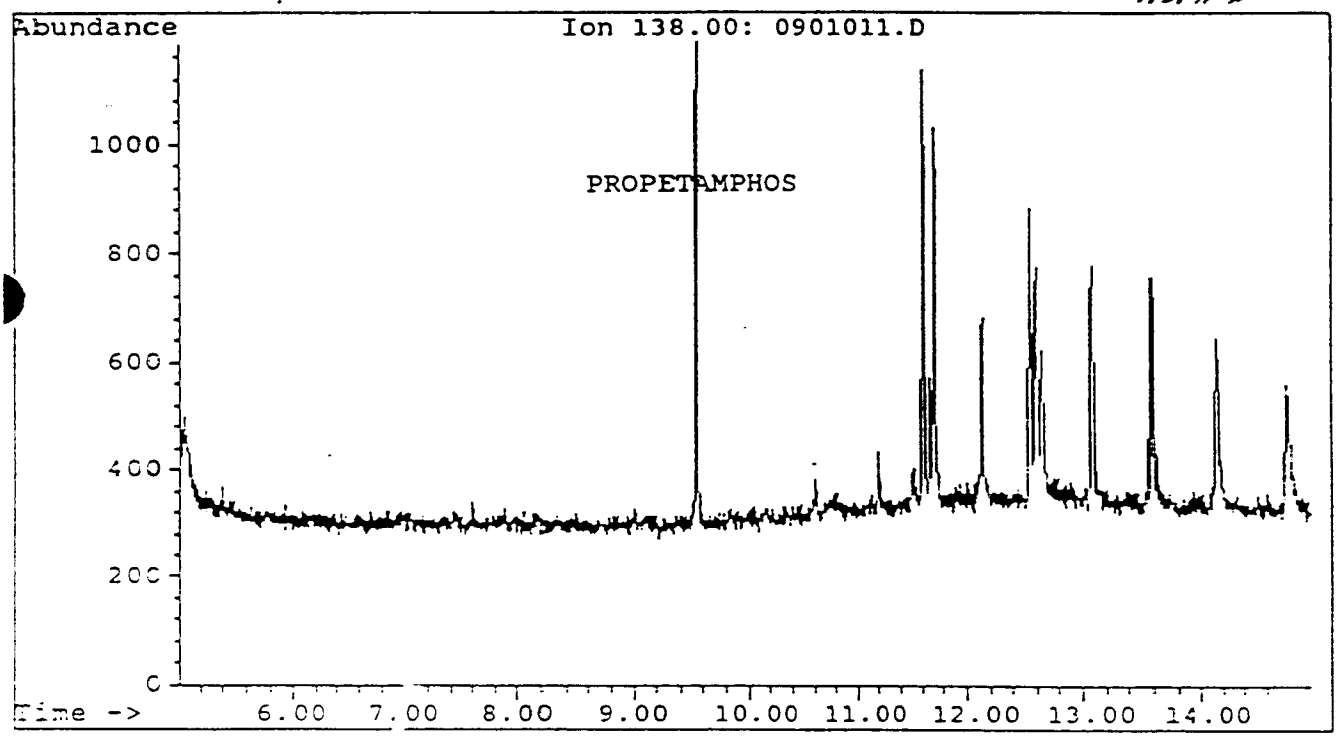
Signal		Rel Resp.	Pct. Unc.(rel)	Integration
Tgt	138			PROPET1.E
Q1	194	0	20	PROPET1.E

Lvl ID	Amt (UG/G)	Response
1	1	429
5	5	1836
10	10	4778
25	25	12454
50	50	26258
100	100	57942

Qualifier Peak Analysis ON
Curve Fit: Linear, Forced Through Origin

File: C:\CHEMPC\DATA\BEER\0901011.D
Operator: STEVE SUMMER
Date Acquired: 4 Mar 91 8:28 pm
Method File Name: PROPET1.M
Sample Name: CAL STD 25 NG/ML EXT #45547-08-09
Misc Info: SC900078
Bottle Number: 9

4/3/91



PROPETAMPHOS (SC900078)

Calibration Last Updated: Tue Mar 05 08:40:55 1991
External Standard Report - DETAILED

Information from Data File:
File: D:\CHEMPC\DATA\BEER\1701019.D
Operator: STEVE SUMMER
Date Acquired: 4 Mar 91 11:55 pm
Method File Name: PROPET1.M
Sample Name: BEER BLANK EXT #45547-06-14
Misc Info: SC900078
Bottle Number: 17

Report Style:
Graphics: (On) Off
Compounds: (All) Single

Quantitation Settings:
Reference Peak Window: 5 Percent
Non-Reference Peak Window: 5 Percent
Correlation Window: 0.03 minutes
Default Multiplier: 0.01
Default Sample Amount: 0
Peak Type decoding: * -> Time Reference Peak

Number of Compounds in Database: 1

Time Reference Peaks:

Pk#	Compound	Expected RT	Actual RT

PROPETAMPHOS (SC900078)

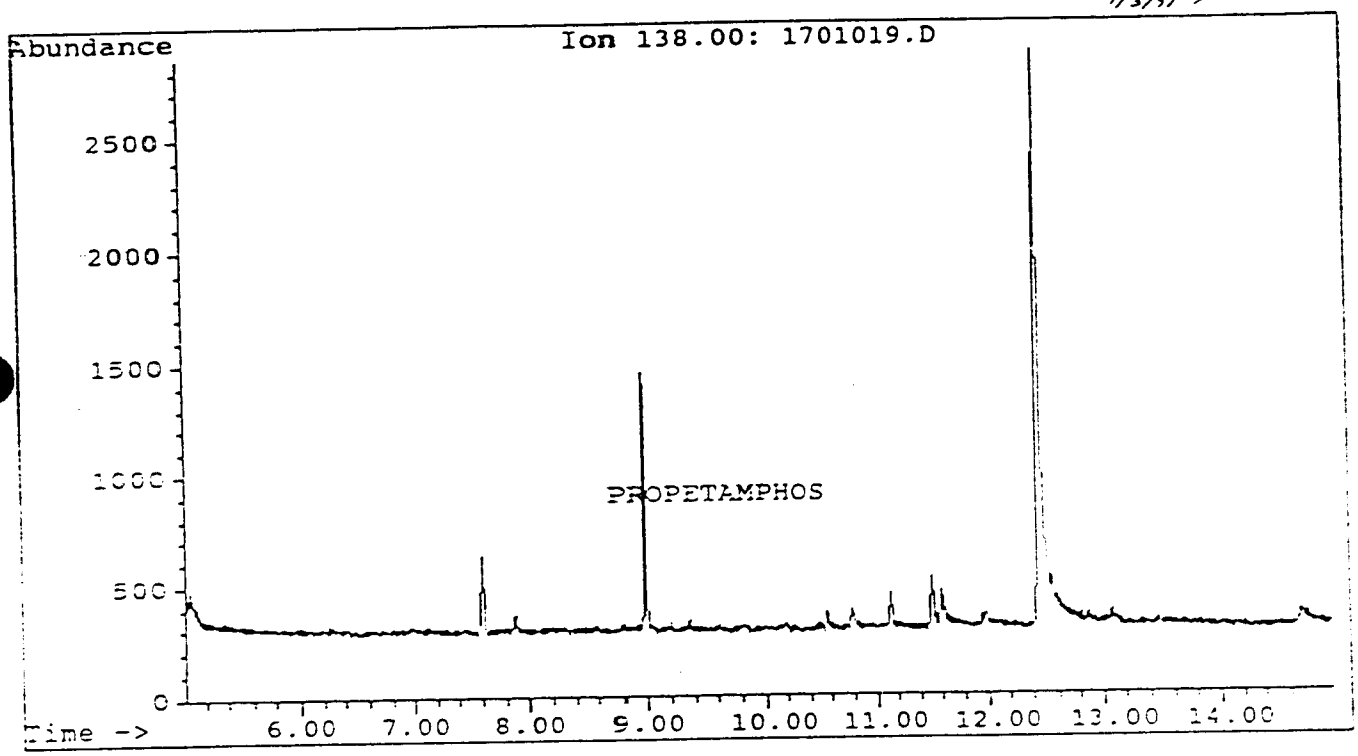
File: D:\CHEMPC\DATA\BEER\1701019.D
Operator: STEVE SUMMER
Date Acquired: 4 Mar 91 11:55 pm
Method File Name: PROPET1.M
Sample Name: BEER BLANK EXT #45547-06-14
Misc Info: SC900078
Bottle Number: 17

Compound: PROPETAMPHOS
Ret Time:
Amount:
Pk # and Type: 1
***** NOT FOUND ****

Battelle Study Number SC900078
Zoecon Study Number 1538

File: C:\CHEMPC\DATA\BEER\1701019.D
Operator: STEVE SUMMER
Date Acquired: 4 Mar 91 11:55 pm
Method File Name: PROPET1.M
Sample Name: BEER BLANK EXT #45547-06-14
Misc Info: SC900078
Bottle Number: 17

4/3/91



PROPETAMPHOS (SC900078)

Calibration Last Updated: Tue Mar 05 08:40:55 1991
External Standard Report - DETAILED

Information from Data File:
File: D:\CHEMPC\DATA\BEER\1901021.D
Operator: STEVE SUMMER
Date Acquired: 5 Mar 91 0:47 am
Method File Name: PROPET1.M
Sample Name: 0.1 PPM BEER SPIKE EXT #45547-06-08
Misc Info: SC900078
Bottle Number: 19

Report Style:
Graphics: (On) Off
Compounds: (All) Single

Quantitation Settings:
Reference Peak Window: 5 Percent
Non-Reference Peak Window: 5 Percent
Correlation Window: 0.03 minutes
Default Multiplier: 0.01
Default Sample Amount: 0
Peak Type decoding: * -> Time Reference Peak

Number of Compounds in Database: 1

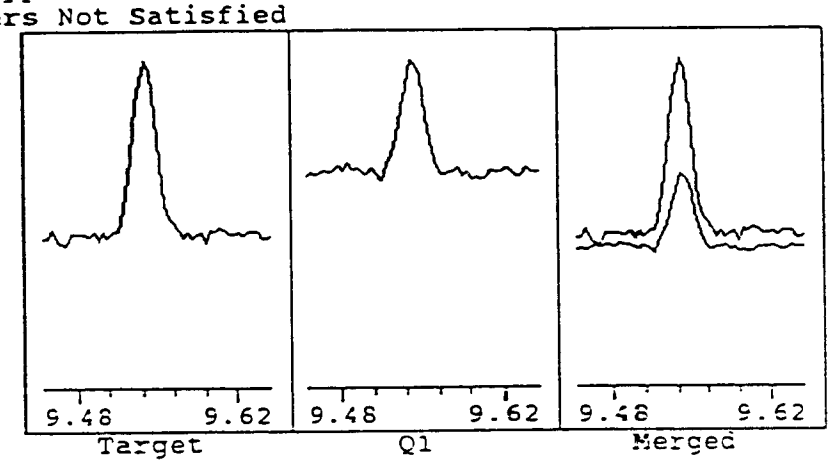
Time Reference Peaks:

Pk#	Compound	Expected RT	Actual RT

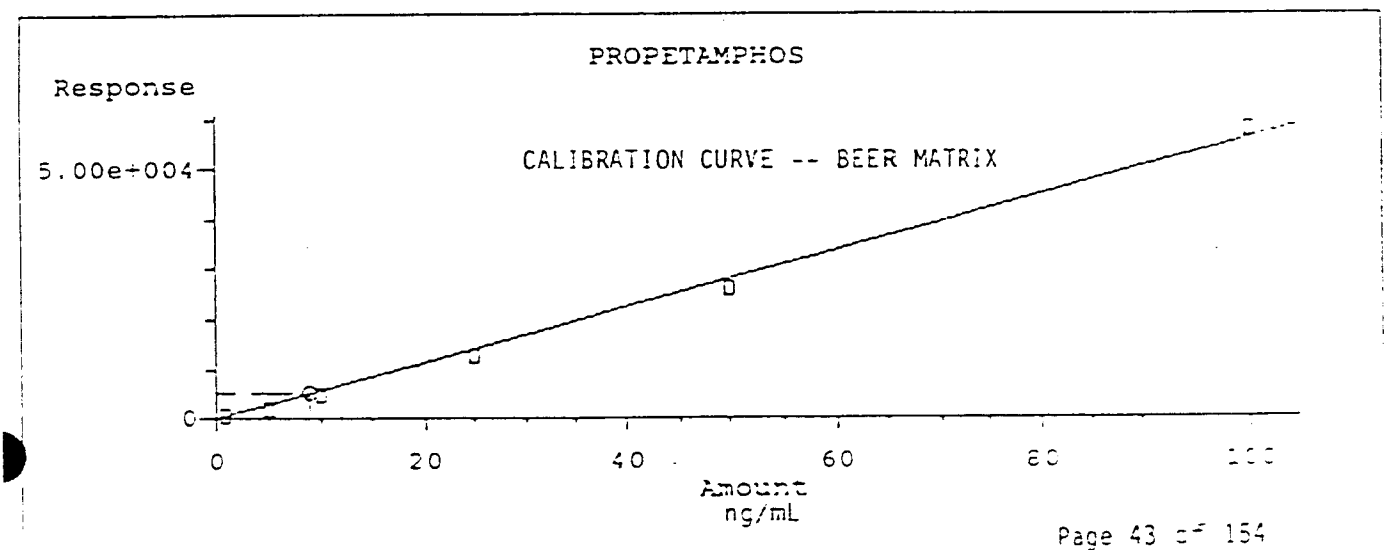
PROPETAMPHOS (SC900078)

File: D:\CHEMPC\DATA\BEER\1901021.D
Operator: STEVE SUMMER
Date Acquired: 5 Mar 91 0:47 am
Method File Name: PROPET1.M
Sample Name: 0.1 PPM BEER SPIKE EXT #45547-06-08
Misc Info: SC900078
Bottle Number: 19

Compound: PROPETAMPHOS
Ret Time: 9.54
Amount: 0.09 UG/G
Pk # and Type: 1
Qualifiers Not Satisfied



	Signal	Ratios	Limits	RT	Limits	Area	Integ Type
Tgt	138	100.0%		9.54	9.31	4965	PROPET1
Q1	194	42.0	0.0-0.0	9.54	to	2086	PROPET1
Q2	0				9.79		
Q3	0						

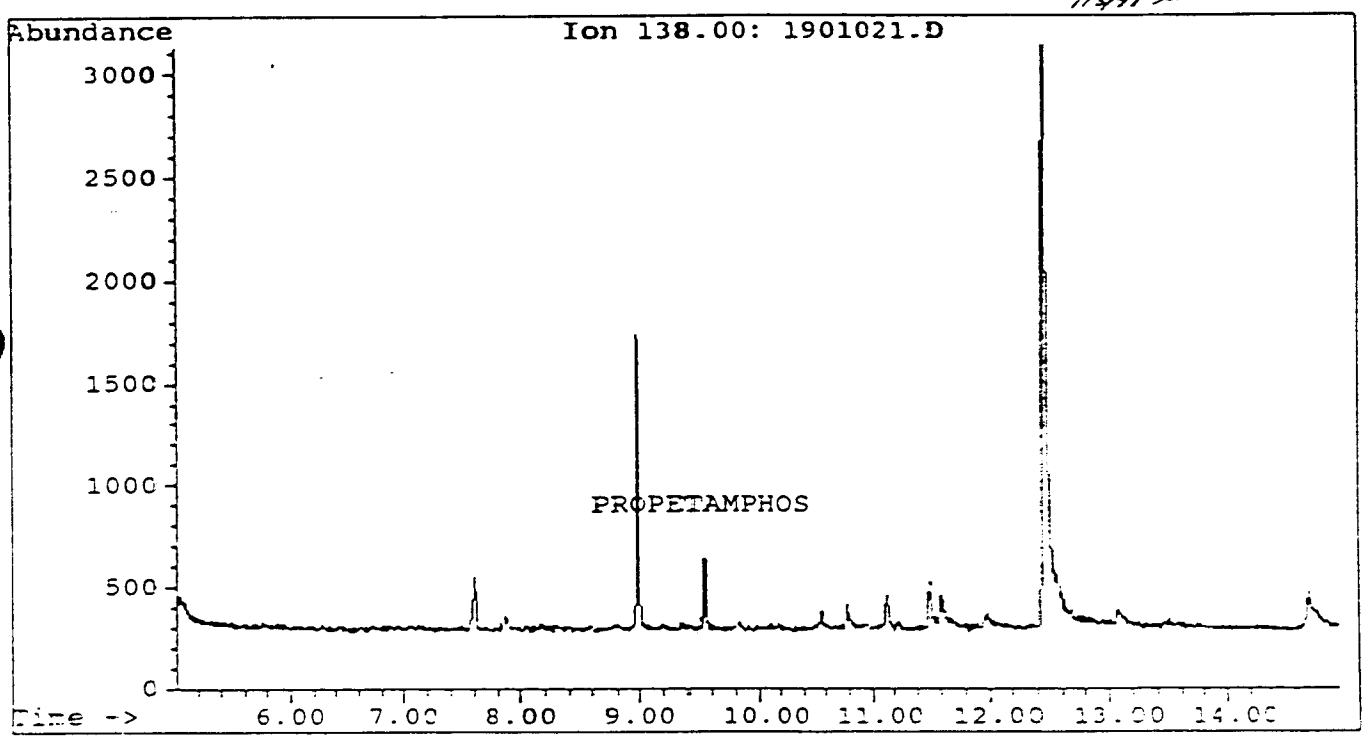


Response = 5.63e-002 * Amt
Corr Coef = 0.997 Curve Fit: Linear/Origin

Battelle Study Number SC900078
Zoecon Study Number 1538

File: C:\CHEMPC\DATA\BEER\1901021.D
Operator: STEVE SUMMER
Date Acquired: 5 Mar 91 0:47 am
Method File Name: PROPET1.M
Sample Name: 0.1 PPM BEER SPIKE EXT #45547-06-08
Misc Info: SC900078
Bottle Number: 19

4/3/91



APPENDIX 3

DETAILED ANALYTICAL DATA - BOLOGNA

PROPETAMPHOS FOOD RESIDUE METHOD VALIDATION

DATA SUMMARY PAGE 1 OF 2

SC900078 LOTUS 123 V-2.2

ANALYST: S.J. SUMMER

INITIALS: DATE:

MATRIX: BOLOGNA ANALYSIS DATE: MARCH 14,1991
STD CURVE ID #: 031491

CALIBRATION STANDARDS

SAMPLE DESCRIPTION	LEB NUMBER 45547-	PEAK AREA	AREA AVERAGE	CALC. CONC. NG/ML	PERCENT DEVIATION
1 NG/ML	2302	1309	901	2.414346	141.4346
1 NG/ML	2303	493		0.909299	9.070677
5 NG/ML	2304	2867	2849	5.287953	5.755062
5 NG/ML	2305	2831		5.221553	4.431079
10 NG/ML	2306	4909	4645	9.054259	9.457405
10 NG/ML	2307	4381		8.080405	19.19594
25 NG/ML	2308	11807	11455	21.77707	12.89171
25 NG/ML	2309	11103		20.47859	18.08560
50 NG/ML	2310	22389	22482	41.29472	17.41054
50 NG/ML	2311	22575		41.63776	16.72442
100 NG/ML	2312	58225	57123	107.3913	7.391374
100 NG/ML	2313	56021		103.3262	3.326271

AVERAGE DEVIATION:22.09517

Regression Output:

Constant	0
Std Err of Y Est	2579.314
R Squared	0.983974
No. of Observations	12
Degrees of Freedom	11
X Coefficient(s)	542.1757
Std Err of Coef.	15.84400

THE CALCULATED CONCENTRATION OF PROPETAMPHOS IN THE SAMPLES IS DETERMINED BY THE FOLLOWING EQUATION:

PROPETAMPHOS CONC. (UG/ML) = (PEAK AREA/X-COEFFICIENT)(MULTIPLIER)

MULTIPLIERS= 0.001 FOR QC STANDARD
0.008 FOR SAMPLE EXTRACTS

WHERE THE X-COEFFICIENT IS DETERMINED BY PERFORMING LINEAR REGRESSION ON THE ACTUAL STANDARD CONCENTRATION AND THE OBSERVED PEAK AREA. THE LINEAR REGRESSION IS CONSTRAINED THROUGH THE ORIGIN.

PROPETAMPHOS FOOD RESIDUE METHOD VALIDATION

DATA SUMMARY PAGE 2 OF 2
SC900078
INITIALS: DATE: MATRIX: BOLOGNA

SAMPLE DESCRIPTION	LRB NUMBER 45547-	PEAK AREA	CONC. UG/G	EXPECTED CONC. UG/G	PERCENT RECOVERY	AVERAGE PERCENT RECOVERY
50 NG/ML STD.	2314	35955	0.066316	0.05	132.6322	124.5149
50 NG/ML STD.	2315	31554	0.058198	0.05	116.3976	
SOLVENT BLANK	2215	0	0	0	NA	
SOLVENT BLANK	2215	0	0	0	NA	
MATRIX BLANK	2214	0	0	0	NA	
MATRIX BLANK	2214	497	0.007333	0	NA	
0.1 PPM SPIKE	2208	4943	0.072935	0.1	72.93575	72.71442
0.1 PPM SPIKE	2208	4913	0.072493	0.1	72.49309	
0.1 PPM SPIKE	2209	5489	0.080992	0.1	80.99218	80.28392
0.1 PPM SPIKE	2209	5393	0.079575	0.1	79.57567	
0.1 PPM SPIKE	2210	6067	0.089815	0.1	89.81589	87.99360
0.1 PPM SPIKE	2210	5840	0.086171	0.1	86.17131	
0.3 PPM SPIKE	2211	17156	0.253143	0.3	84.38100	88.34527
0.3 PPM SPIKE	2211	18768	0.276928	0.3	92.30954	
0.3 PPM SPIKE	2212	19073	0.281429	0.3	93.80967	93.80967 *
0.3 PPM SPIKE	2212	19073	0.281429	0.3	93.80967	
0.3 PPM SPIKE	2213	16680	0.246119	0.3	82.03961	92.37348
0.3 PPM SPIKE	2213	20882	0.308121	0.3	102.7071	

NA = NOT APPLICABLE

EXPECTED CONCENTRATION = (FORTIFICATION LEVEL, UG/G)(SAMPLE WT.)/(FINAL VOLUME)
*THE SECOND INJECTION FOR 2212 WAS A BAD INJECTION AND THE AREA WAS NOT USED IN THE AVERAGE. (AREA=33426)

SUMMARY OF RESULTS:

	AVERAGE RECOVERY	STANDARD DEVIATION	RELATIVE * STANDARD DEVIATION
0.1 PPM SPIKE	80.3	7.6	9.5
0.3 PPM SPIKE	91.5	2.8	3.1

* RELATIVE STANDARD DEVIATION = (STANDARD DEVIATION/AVERAGE) X 100

Page 1

Sequence Calibration Sample
File: D:\CHEMPC\DATA\BOLOGNA\0901011.D
Operator: STEVE SUMMER
Date Acquired: 14 Mar 91 1:15 pm
Method File Name: PROPET1.M
Sample Name: CAL STD 25 NG/ML EXT #45547-23-09
Misc Info: SC900078
Bottle Number: 9

E: 3/26/91

PROPETAMPHOS (SC900078)
Calibration Last Updated: Fri Mar 15 01:24:07 1991
External Standard

Reference Window: 5 Percent
Non-Reference Window: 5 Percent
Correlation Window: 0.03 minutes
Default Multiplier: 1
Default Sample Amount: 0

Compound Information

*** Compound 1 ***

Name: PROPETAMPHOS ( )
Ret. Time 9.70 min., Extract & Integrate from 9.60 to 9.80 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 138			PROPET1.E
Q1 194	0	20	PROPET1.E

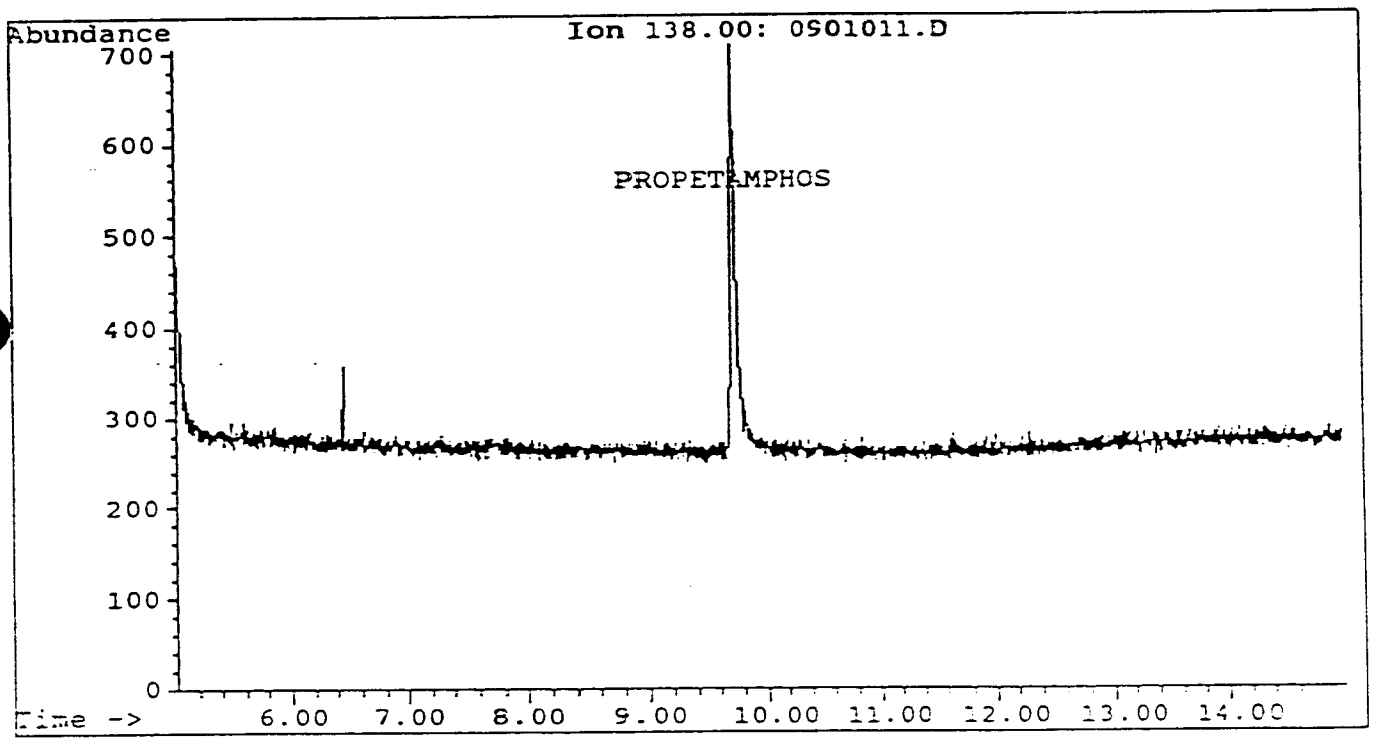
Lvl ID	Amt (UG/G)	Response
1	1	901
5	5	2849
10	10	4645
25	25	11455
50	50	25892
100	100	61389

Qualifier Peak Analysis ON
Curve Fit: Linear, Forced Through Origin

Battelle Study Number SC900078
Zoecon Study Number 1538

File: C:\CHEMPC\DATA\BOLOGNA\0901011.D
Operator: STEVE SUMMER
Date Acquired: 14 Mar 91 1:15 pm
Method File Name: PROPET1.M
Sample Name: CAL STD 25 NG/ML EXT #45547-23-09
Misc Info: SC900078
Bottle Number: 9

4/2/91



PROPETAMPHOS (SC900078)

Calibration Last Updated: Fri Mar 15 01:26:56 1991
External Standard Report - DETAILED

BA 3/26/91

Information from Data File:

File:	D:\CHEMPC\DATA\BOLOGNA\1701019.D
Operator:	STEVE SUMMER
Date Acquired:	14 Mar 91 4:46 pm
Method File Name:	PROPET1.M
Sample Name:	BOLOGNA BLANK EXT #45547-22-14
Misc Info:	SC900078
Bottle Number:	17

Report Style:
Graphics: (On) Off
Compounds: (All) Single

Quantitation Settings:

Reference Peak Window:	5 Percent
Non-Reference Peak Window:	5 Percent
Correlation Window:	0.03 minutes
Default Multiplier:	0.008
Default Sample Amount:	0
Peak Type decoding:	* -> Time Reference Peak

Number of Compounds in Database: 1

Time Reference Peaks:

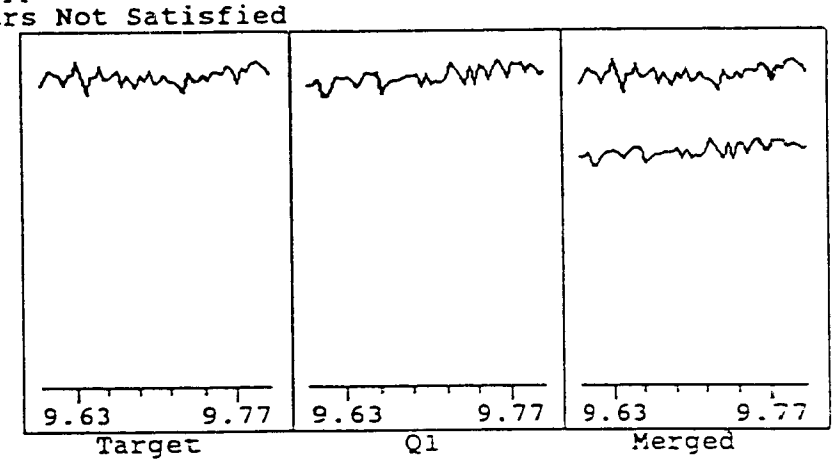
Pk#	Compound	Expected RT	Actual RT

Battelle Study Number SC900078
Zoecon Study Number 1538

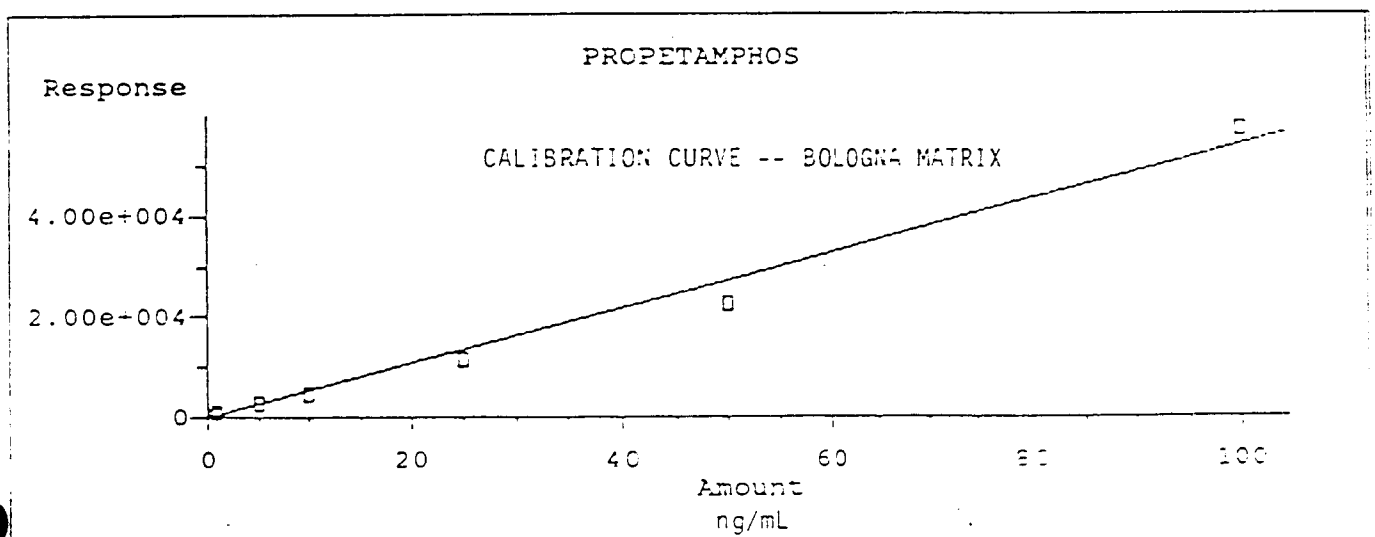
PROPETAMPHOS (SC900078)

File: D:\CHEMPC\DATA\BOLOGNA\1701019.D
Operator: STEVE SUMMER
Date Acquired: 14 Mar 91 4:46 pm 3/26/91
Method File Name: PROPET1.M
Sample Name: BOLOGNA BLANK EXT #45547-22-14
Misc Info: SC900078
Bottle Number: 17

Compound: PROPETAMPHOS
Ret Time: 9.65
Amount: 0.01 UG/G
Pk # and Type: 1
Qualifiers Not Satisfied



	Signal	Ratios	Limits	RT	Limits	Area	Integ Type
Tgt	138	100.0%		9.65	9.46	497	PROPET1
Q1	194	0.0	0.0-0.0	0.00	to	0	PROPET1
Q2	0				9.94		
Q3	0						

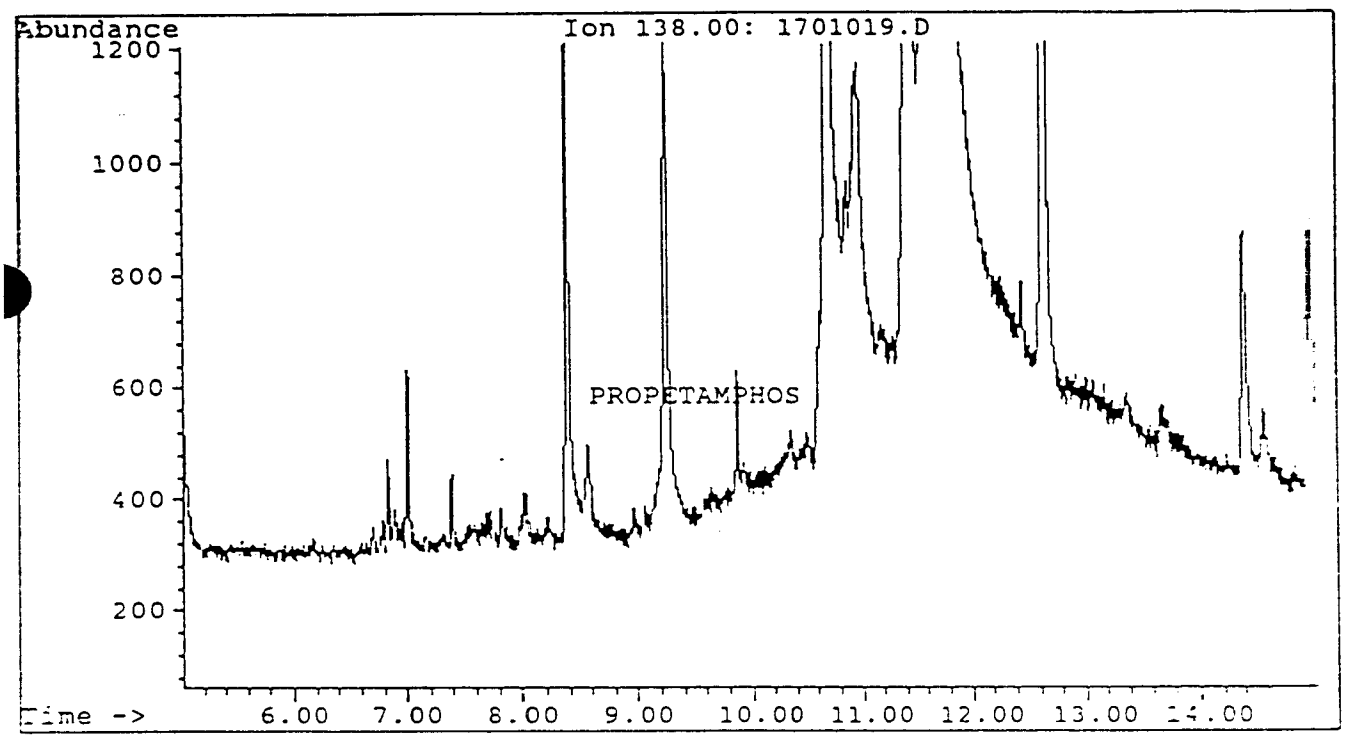


Response = 5.42e-002 * Amt
Corr Coef = 0.987 Curve Fit: Linear/Origin

Battelle Study Number SC900078
Zoecon Study Number 1538

File: C:\CHEMPC\DATA\BOLOGNA\1701019.D
Operator: STEVE SUMMER
Date Acquired: 14 Mar 91 4:46 pm
Method File Name: PROPET1.M
Sample Name: BOLOGNA BLANK EXT #45547-22-14
Misc Info: SC900078
Bottle Number: 17

4/2/91



PROPETAMPHOS (SC900078)

Calibration Last Updated: Fri Mar 15 01:26:56 1991
External Standard Report - DETAILED

BA 3/26/91

Information from Data File:

File:	D:\CHEMPC\DATA\BOLOGNA\1901021.D
Operator:	STEVE SUMMER
Date Acquired:	14 Mar 91 5:38 pm
Method File Name:	PROPET1.M
Sample Name:	0.1 PPM BOLOGNA SPIKE EXT #45547-22-08
Misc Info:	SC900078
Bottle Number:	19

Report Style:
Graphics: (On) Off
Compounds: (All) Single

Quantitation Settings:

Reference Peak Window:	5 Percent
Non-Reference Peak Window:	5 Percent
Correlation Window:	0.03 minutes
Default Multiplier:	0.008
Default Sample Amount:	0
Peak Type decoding:	* -> Time Reference Peak

Number of Compounds in Database: 1

Time Reference Peaks:

Pk#	Compound	Expected RT	Actual RT

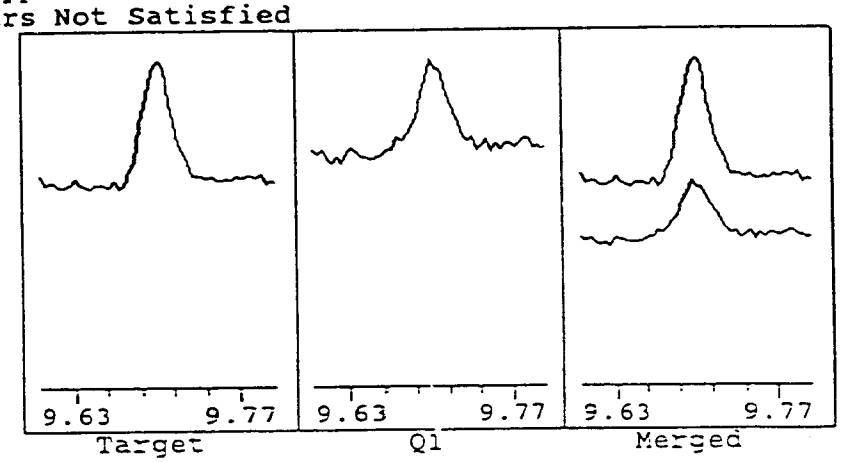
Battelle Study Number SC900078
Zoecon Study Number 1538

PROPETAMPHOS (SC900078)

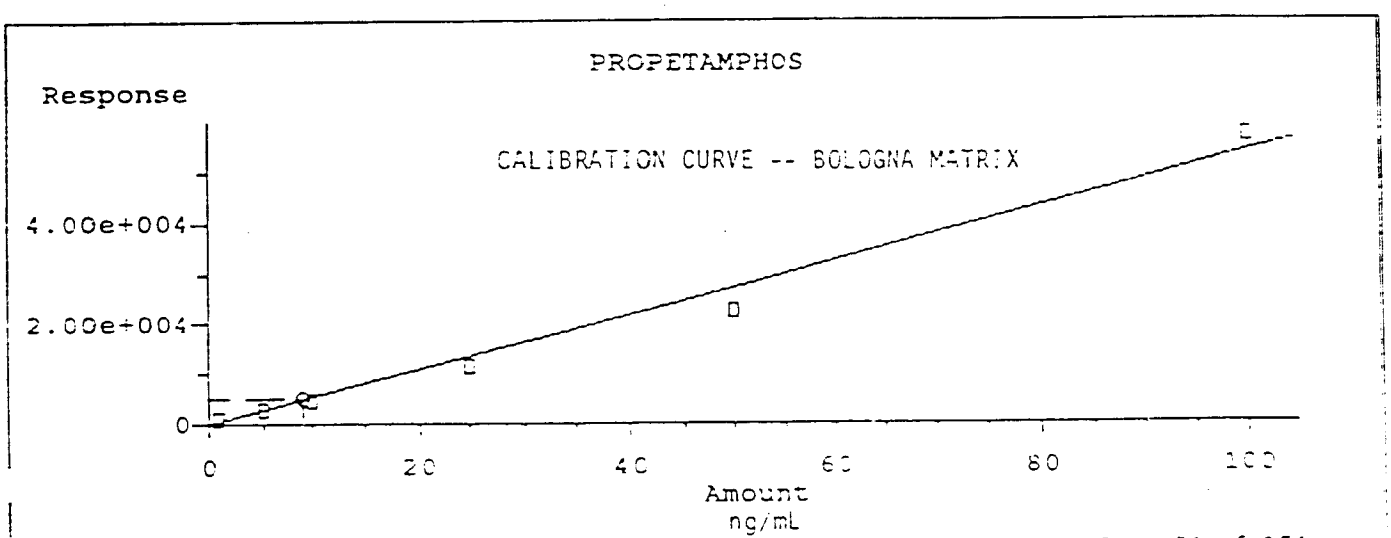
File: D:\CHEMPC\DATA\BOLOGNA\1901021.D
Operator: STEVE SUMMER
Date Acquired: 14 Mar 91 5:38 pm
Method File Name: PROPET1.M
Sample Name: 0.1 PPM BOLOGNA SPIKE EXT #45547-22-08
Misc Info: SC900078
Bottle Number: 19

BA 3/26/91

Compound: PROPETAMPHOS
Ret Time: 9.70
Amount: 0.07 UG/G
Pk # and Type: 1
Qualifiers Not Satisfied



	Signal	Ratios	Limits	RT	Limits	Area	Integ Type
Tgt	138	100.0%		9.70	9.46	4913	PROPET1
Q1	194	52.4	0.0-0.0	9.70	to	2574	PROPET1
Q2	0				9.94		
Q3	0						

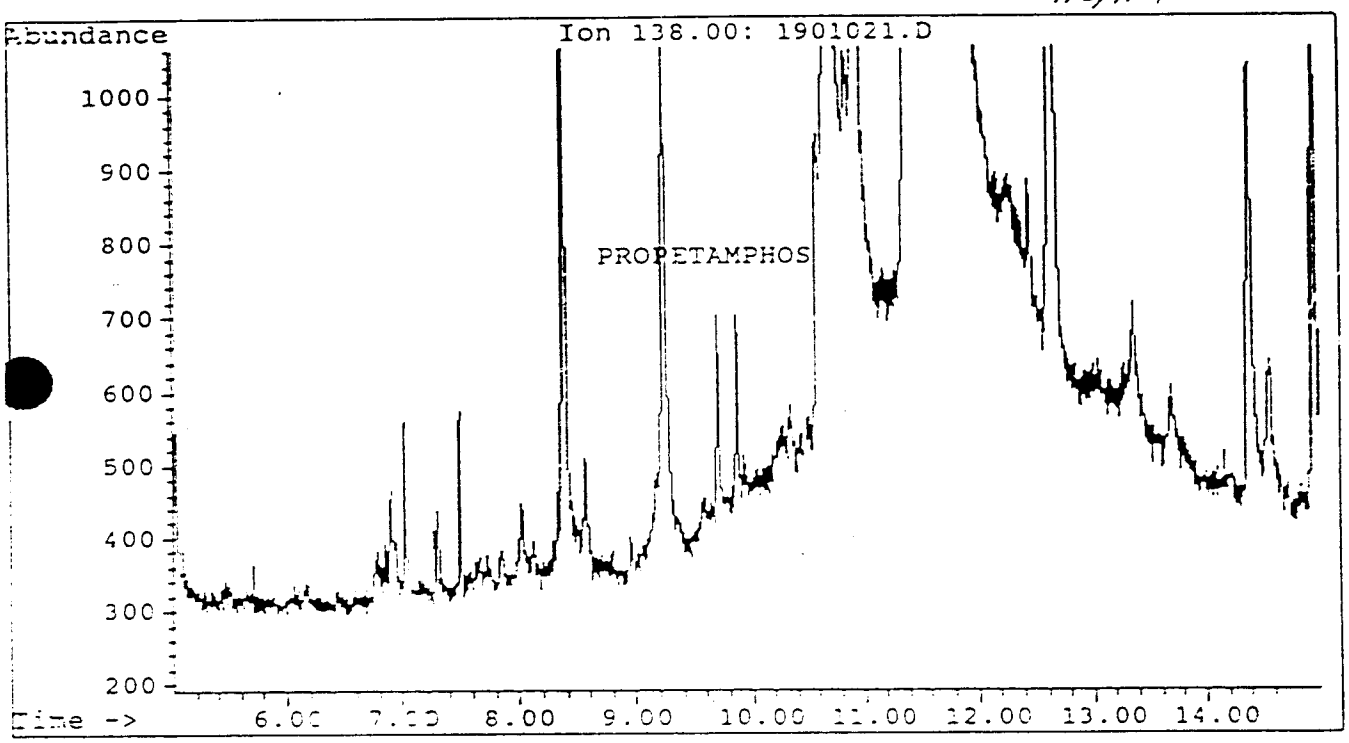


Response = 5.42e+002 * Amt
Corr Coef = 0.987 Curve Fit: Linear/Origin

Battelle Study Number SC900078
Zoecon Study Number 1538

File: C:\CHEMPC\DATA\BOLOGNA\1901021.D
Operator: STEVE SUMMER
Date Acquired: 14 Mar 91 5:38 pm
Method File Name: PROPET1.M
Sample Name: 0.1 PPM BOLOGNA SPIKE EXT #45547-22-08
Misc Info: SC900078
Bottle Number: 19

4/3/91



APPENDIX 4

DETAILED ANALYTICAL DATA - BREAD

PROPETAMPHOS FOOD RESIDUE METHOD VALIDATION

DATA SUMMARY PAGE 1 OF 2

SC900078 LOTUS 123 V 2.2

ANALYST: S.J. SUMMER

INITIALS: DATE:

MATRIX: BREAD ANALYSIS DATE: MARCH 15,1991
STD CURVE ID #: 031591

CALIBRATION STANDARDS

SAMPLE DESCRIPTION	LRB NUMBER 45547-	PEAK AREA	AREA AVERAGE	CALC. CONC. NG/ML	PERCENT DEVIATION
1 NG/ML	4402	786	783	0.889396	11.06030
1 NG/ML	4403	780		0.882607	11.73923
5 NG/ML	4404	2092	3115	2.367299	52.65601
5 NG/ML	4405	4146		4.691399	6.172013
10 NG/ML	4406	7097	7720	8.030598	19.69401
10 NG/ML	4407	8343		9.440507	5.594923
25 NG/ML	4408	19662	19581	22.27113	10.91546
25 NG/ML	4409	19480		22.04256	11.82975
50 NG/ML	4410	44002	43948	49.79038	0.419202
50 NG/ML	4411	43894		49.66818	0.663636
100 NG/ML	4412	90909	89301	102.8679	2.867926
100 NG/ML	4413	87691		99.22860	0.772396

AVERAGE DEVIATION:11.19882

Regression Output:

Constant	0
Std Err of Y Est	1607.938
R Squared	0.997571
No. of Observations	11
Degrees of Freedom	11
X Coefficient(s)	883.7448
Std Err of Coef.	9.877116

THE CALCULATED CONCENTRATION OF PROPETAMPHOS IN THE SAMPLES IS DETERMINED BY THE FOLLOWING EQUATION:

PROPETAMPHOS CONC. (UG/ML) = (PEAK AREA/A-COEFFICIENT) MULTIPLIER

MULTIPLIER= 0.001 FOR QC STANDARD
0.008 FOR SAMPLE EXTRACTS

WHERE THE A-COEFFICIENT IS DETERMINED BY PERFORMING LINEAR REGRESSION ON THE ACTUAL STANDARD CONCENTRATION AND THE OBSERVED PEAK AREA. THE LINEAR REGRESSION IS CONSTRAINED THROUGH THE ORIGIN.

PROPETAMPHOS FOOD RESIDUE METHOD VALIDATION

DATA SUMMARY PAGE 2 OF 2
SC900078
INITIALS: DATE: MATRIX: BREAD

SAMPLE DESCRIPTION	LAB NUMBER 45547-	PEAK AREA	CONC. UG/G	EXPECTED CONC. UG/G	PERCENT RECOVERY	AVERAGE PERCENT RECOVERY
50 NG/ML STD.	4414	47703	0.053978	0.05	107.9564	107.7697
50 NG/ML STD.	4415	47538	0.053791	0.05	107.5830	
SOLVENT BLANK	4315	0	0	0	NA	
SOLVENT BLANK	4315	0	0	0	NA	
MATRIX BLANK	4314	0	0	0	NA	
MATRIX BLANK	4314	0	0	0	NA	
0.1 PPM SPIKE	4306	10708	0.096932	0.1	96.93295	89.76346
0.1 PPM SPIKE	4308	9124	0.082593	0.1	82.59397	
0.1 PPM SPIKE	4309	11004	0.099612	0.1	99.61246	78.66523
0.1 PPM SPIKE	4309	6376	0.057718	0.1	57.71801	
0.1 PPM SPIKE	4310	9705	0.087889	0.1	87.88962	77.40243
0.1 PPM SPIKE	4310	7392	0.066915	0.1	66.91524	
0.3 PPM SPIKE	4311	27695	0.250705	0.3	83.56661	84.42199
0.3 PPM SPIKE	4311	28254	0.255756	0.3	85.25537	
0.3 PPM SPIKE	4312	30944	0.280117	0.3	93.37234	91.65239
0.3 PPM SPIKE	4312	29804	0.269797	0.3	89.93244	
0.3 PPM SPIKE	4313	30538	0.276177	0.3	92.05673	90.45899
0.3 PPM SPIKE	4313	29449	0.266552	0.3	88.85124	

NA = NOT APPLICABLE

EXPECTED CONCENTRATION = (FORTIFICATION LEVEL, UG/G SAMPLE WT.)/(FINAL VOLUME)

SUMMARY OF RESULTS:

	AVERAGE RECOVERY	STANDARD DEVIATION	RELATIVE * STANDARD DEVIATION
0.1 PPM SPIKE	81.9	6.8	8.3
0.3 PPM SPIKE	88.8	3.9	4.4

* RELATIVE STANDARD DEVIATION = (STANDARD DEVIATION/AVERAGE) X 100

Page 1

BJ 3/25/91

Sequence Calibration Sample
File: D:\CHEMPC\DATA\BREAD\0901011.D
Operator: STEVE SUMMER
Date Acquired: 15 Mar 91 6:28 am
Method File Name: PROPET1.M
Sample Name: CAL STD 25 NG/ML EXT #45547-44-09
Misc Info: SC900078
Bottle Number: 9

PROPETAMPHOS (SC900078)
Calibration Last Updated: Fri Mar 15 16:47:39 1991
External Standard

Reference Window: 5 Percent
Non-Reference Window: 5 Percent
Correlation Window: 0.03 minutes
Default Multiplier: 1
Default Sample Amount: 0

Compound Information

*** Compound 1 ***

Name: PROPETAMPHOS ( )
Ret. Time 9.62 min., Extract & Integrate from 9.52 to 9.72 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 138			PROPET1.E
Q1 194	0	20	PROPET1.E

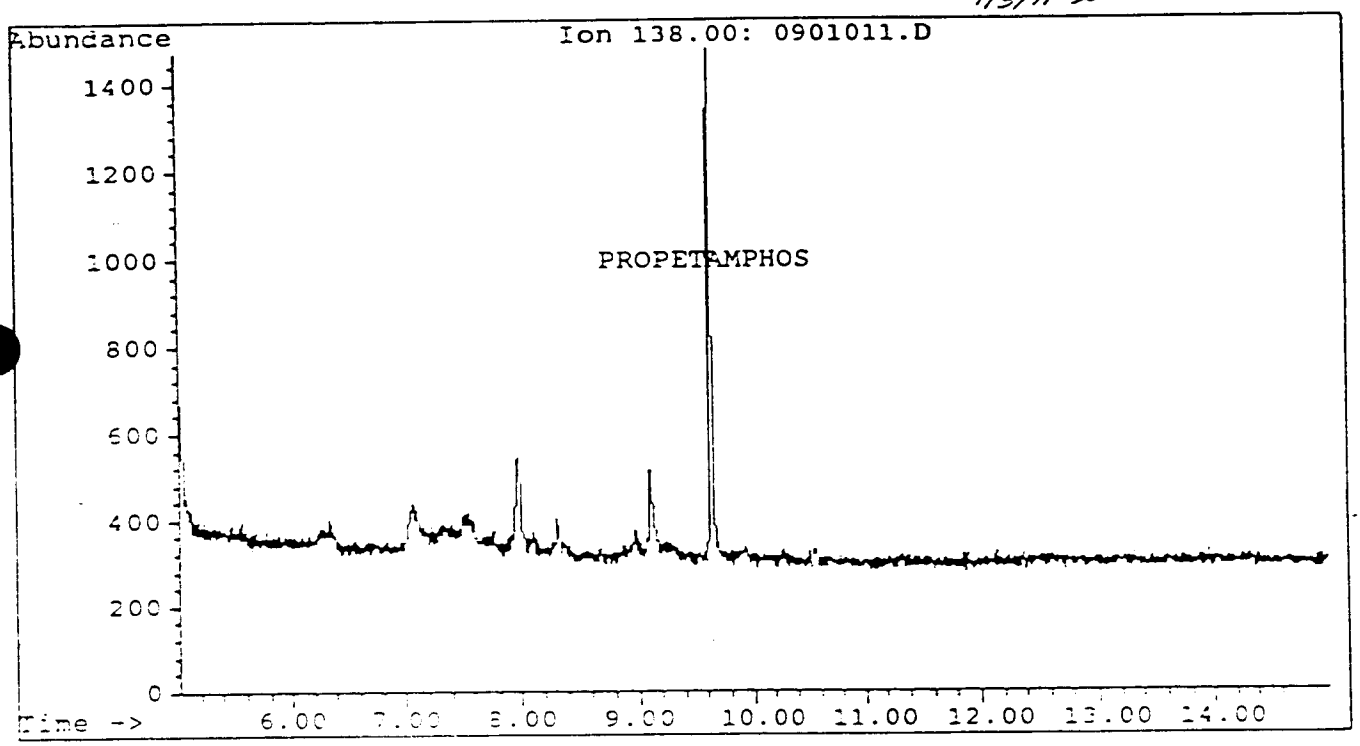
Lvl ID	Amt (UG/G)	Response
1	1	783
5	5	3119
10	10	7720
25	25	19581
50	50	43948
100	100	89300

Qualifier Peak Analysis ON
Curve Fit: Linear, Forced Through Origin

Battelle Study Number SC900078
Zoecon Study Number 1538

File: C:\CHEMPC\DATA\BREAD\0901011.D
Operator: STEVE SUMMER
Date Acquired: 15 Mar 91 6:28 am
Method File Name: PROPET1.M
Sample Name: CAL STD 25 NG/ML EXT #45547-44-09
Misc Info: SC900078
Bottle Number: 9

4/3/91



PROPETAMPHOS (SC900078) BA 3/25/91

Calibration Last Updated: Fri Mar 15 16:50:34 1991
External Standard Report - DETAILED

Information from Data File:

File:	D:\CHEMPC\DATA\BREAD\1701019.D
Operator:	STEVE SUMMER
Date Acquired:	15 Mar 91 9:57 am
Method File Name:	PROPET1.M
Sample Name:	BREAD BLANK EXT #45547-43-14
Misc Info:	SC900078
Bottle Number:	17

Report Style:
Graphics: (On) Off
Compounds: (All) Single

Quantitation Settings:

Reference Peak Window:	5 Percent
Non-Reference Peak Window:	5 Percent
Correlation Window:	0.03 minutes
Default Multiplier:	0.008
Default Sample Amount:	0
Peak Type decoding:	* -> Time Reference Peak

Number of Compounds in Database: 1

Time Reference Peaks:

Pk#	Compound	Expected RT	Actual RT

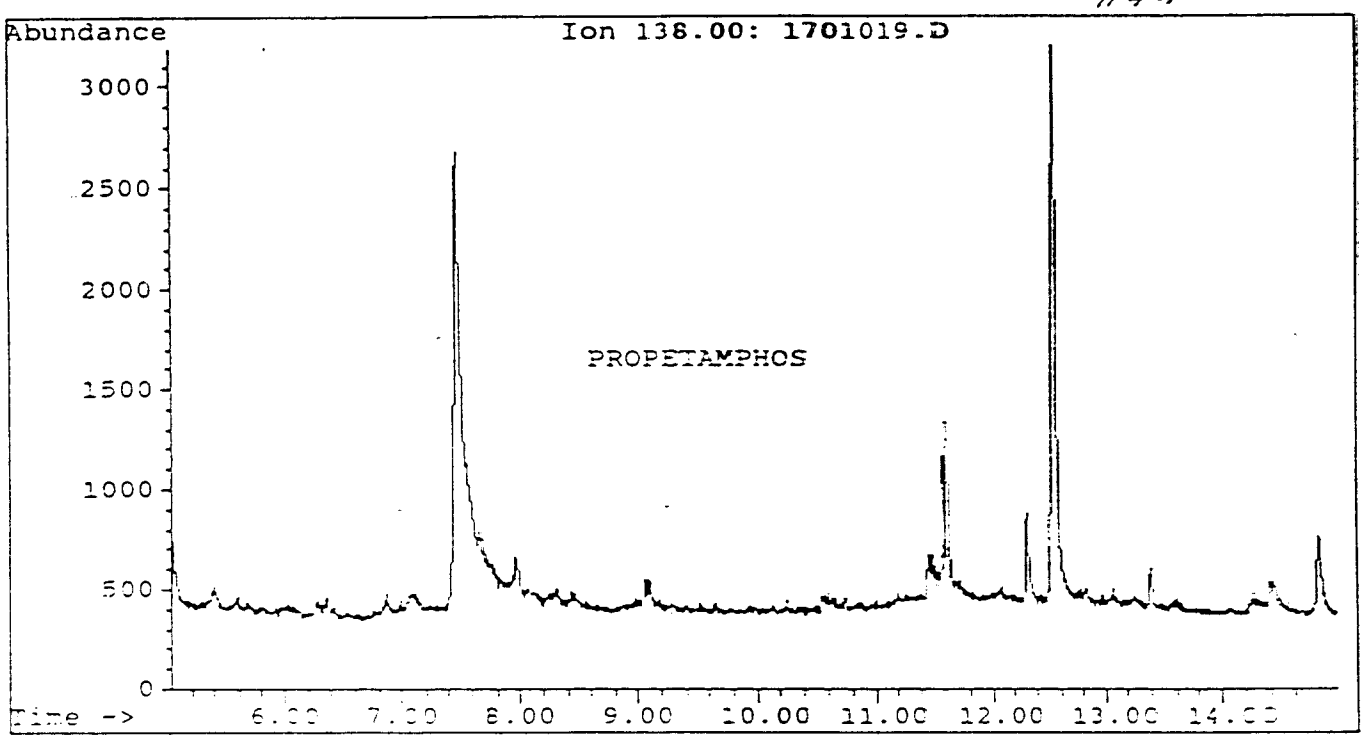
PROPETAMPHOS (SC900078) BA 3/25/91

File: D:\CHEMPC\DATA\BREAD\1701019.D
Operator: STEVE SUMMER
Date Acquired: 15 Mar 91 9:57 am
Method File Name: PROPET1.M
Sample Name: BREAD BLANK EXT #45547-43-14
Misc Info: SC900078
Bottle Number: 17

Compound: PROPETAMPHOS
Ret Time:
Amount:
Pk # and Type: 1
***** NOT FOUND ****

File: C:\CHEMPC\DATA\BREAD\1701019.D
Operator: STEVE SUMMER
Date Acquired: 15 Mar 91 9:57 am
Method File Name: PROPET1.M
Sample Name: BREAD BLANK EXT #45547-43-14
Misc Info: SC900078
Bottle Number: 17

4/3/91



PROPETAMPHOS (SC900078) GA 3/25/91

Calibration Last Updated: Fri Mar 15 16:50:34 1991
External Standard Report - DETAILED

Information from Data File:

File:	D:\CHEMPC\DATA\BREAD\1901021.D
Operator:	STEVE SUMMER
Date Acquired:	15 Mar 91 10:49 am
Method File Name:	PROPET1.M
Sample Name:	0.1 PPM BREAD SPIKE EXT #45547-43-08
Misc Info:	SC900078
Bottle Number:	19

Report Style:
Graphics: (On) Off
Compounds: (All) Single

Quantitation Settings:

Reference Peak Window:	5 Percent
Non-Reference Peak Window:	5 Percent
Correlation Window:	0.03 minutes
Default Multiplier:	0.008
Default Sample Amount:	0
Peak Type decoding:	* -> Time Reference Peak

Number of Compounds in Database: 1

Time Reference Peaks:

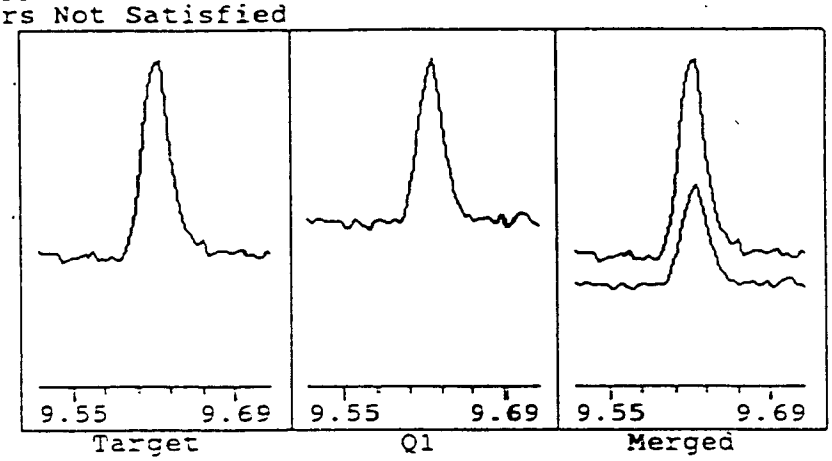
Pk#	Compound	Expected RT	Actual RT

PROPETAMPHOS (SC900078)

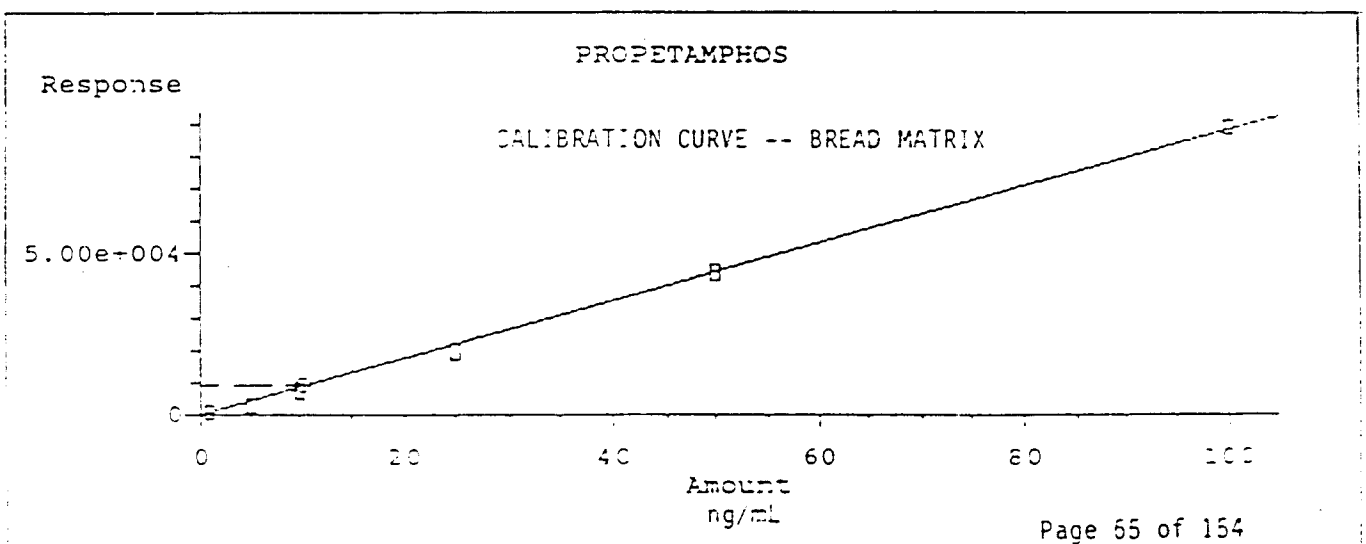
Battelle Study Number SC900078
Zoecon Study Number 1538

File: D:\CHEMPC\DATA\BREAD\1901021.D
Operator: STEVE SUMMER
Date Acquired: 15 Mar 91 10:49 am
Method File Name: PROPET1.M
Sample Name: 0.1 PPM BREAD SPIKE EXT #45547-43-08
Misc Info: SC900078
Bottle Number: 19

Compound: PROPETAMPHOS
Ret Time: 9.62
Amount: 0.08 UG/G
Pk # and Type: 1
Qualifiers Not Satisfied



	Signal	Ratios	Limits	RT	Limits	Area	Integ Type
Tgt	138	100.0%		9.62	9.38	9124	PROPET1
Q1	194	52.9	0.0-0.0	9.62	to	4829	PROPET1
Q2	0				9.86		
Q3	0						

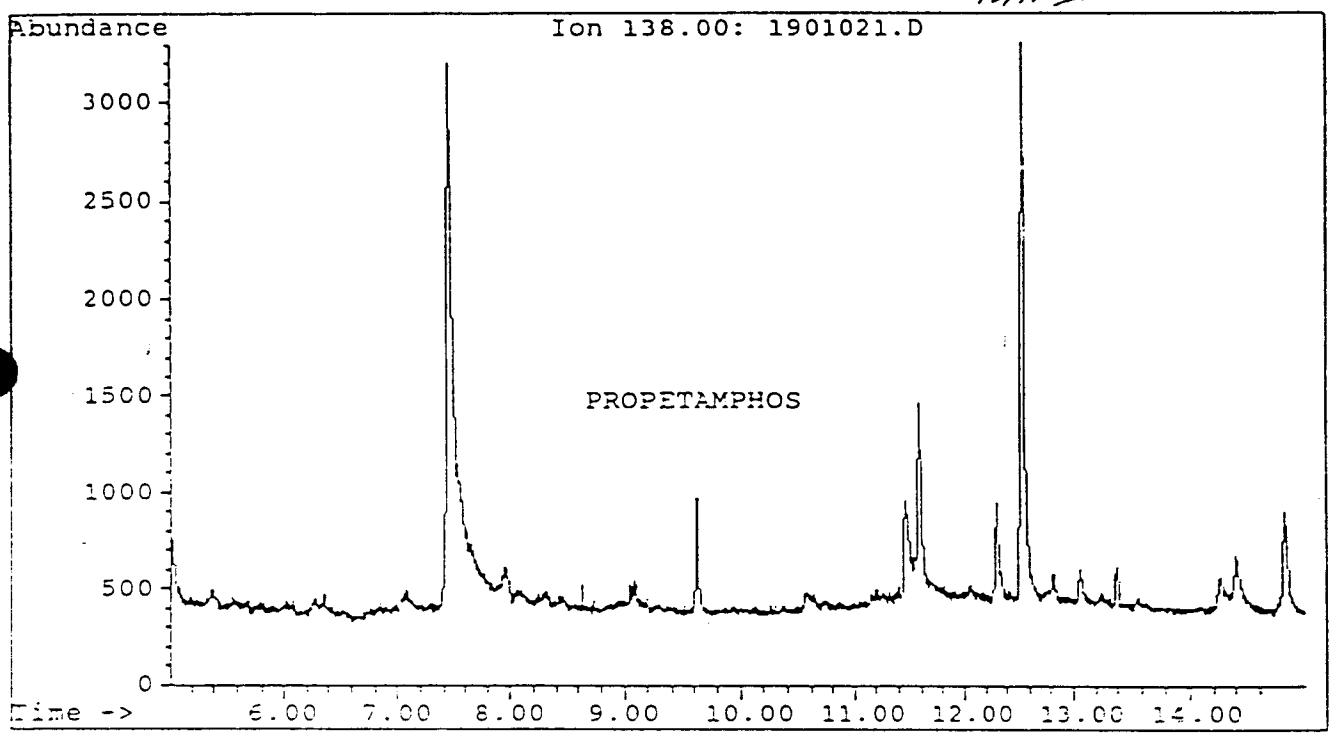


Response = 8.84e+002 * Amt
Corr Coef = 0.999 Curve Fit: Linear/Origin

Battelle Study Number SC900078
Zoecon Study Number 1538

File: C:\CHEMPC\DATA\BREAD\1901021.D
Operator: STEVE SUMMER
Date Acquired: 15 Mar 91 10:49 am
Method File Name: PROPET1.M
Sample Name: 0.1 PPM BREAD SPIKE EXT #45547-43-08
Misc Info: SC900078
Bottle Number: 19

4/3/91



APPENDIX 5

DETAILED ANALYTICAL DATA - BUTTER

PROPETAMPHOS FOOD RESIDUE METHOD VALIDATION

DATA SUMMARY PAGE 1 OF 2

SC900078 LOTUS 123 V 2.2

ANALYST: S.J. SUMMER

INITIALS: DATE:

MATRIX: BUTTER ANALYSIS DATE: MARCH 28,1991
STD CURVE ID #: 032891

CALIBRATION STANDARDS

SAMPLE DESCRIPTION	LRB NUMBER 45547-	PEAK AREA	AREA AVERAGE	CALC. CONC. NG/ML	PERCENT DEVIATION
1 NG/ML	6202	471	534	1.355283	35.52833
1 NG/ML	6203	597		1.717843	71.78432
5 NG/ML	6204	2352	1879	6.767784	35.35569
5 NG/ML	6205	1406		4.045707	19.08584
10 NG/ML	6206	2875	3165	8.272695	17.27304
10 NG/ML	6207	3455		9.941622	0.583776
25 NG/ML	6208	7360	7673	21.17810	15.28759
25 NG/ML	6209	7986		22.97939	8.082435
50 NG/ML	6210	15564	15698	44.78477	10.43044
50 NG/ML	6211	15832		45.55593	8.888124
100 NG/ML	6212	35904	35868	103.3123	3.312303
100 NG/ML	6213	35832		103.1051	3.105126

AVERAGE DEVIATION:19.05975

Regression Output:

Constant	0
Std Err of Y Est	1015.460
R Squared	0.993739
No. of Observations	12
Degrees of Freedom	11

X Coefficient(s) 347.5287
Std Err of Coef. 6.237686

THE CALCULATED CONCENTRATION OF PROPETAMPHOS IN THE SAMPLES IS DETERMINED BY THE FOLLOWING EQUATION:

PROPETAMPHOS CONC. (NG/ML) = (PEAK AREA X-COEFFICIENT)(MULTIPLIER)

MULTIPLIERS= 0.001 FOR QC STANDARD
0.008 FOR SAMPLE EXTRACTS

WHERE THE X-COEFFICIENT IS DETERMINED BY PERFORMING LINEAR REGRESSION ON THE ACTUAL STANDARD CONCENTRATION AND THE OBSERVED PEAK AREA. THE LINEAR REGRESSION IS CONSTRAINED THROUGH THE ORIGIN.

PROPETAMPHOS FOOD RESIDUE METHOD VALIDATION

DATA SUMMARY PAGE 2 OF 2
SC900078
INITIALS: DATE: MATRIX: BUTTER

SAMPLE DESCRIPTION	LAB NUMBER 45547-	PEAK AREA	CONC. UG/G	EXPECTED CONC. UG/G	PERCENT RECOVERY	AVERAGE PERCENT RECOVERY
25 NG/ML STD.	6214	10347	0.029773	0.025	119.0922	117.2104
25 NG/ML STD.	6215	10028	0.028832	0.025	115.3285	
SOLVENT BLANK	6215	0	0	0	NA	
SOLVENT BLANK	6215	0	0	0	NA	
MATRIX BLANK	6214	0	0	0	NA	
MATRIX BLANK	6214	0	0	0	NA	
0.1 PPM SPIKE	6208	3762	0.086600	0.1	86.60001	91.83699
0.1 PPM SPIKE	6208	4217	0.097073	0.1	97.07397	
0.1 PPM SPIKE	6209	4507	0.103749	0.1	103.7496	106.5580
0.1 PPM SPIKE	6209	4751	0.109366	0.1	109.3664	
0.1 PPM SPIKE	6210	4355	0.100342	0.1	100.3427	99.19178
0.1 PPM SPIKE	6210	4255	0.098040	0.1	98.04079	
0.3 PPM SPIKE	6211	13856	0.318960	0.3	106.3202	106.7345
0.3 PPM SPIKE	6211	13964	0.321446	0.3	107.1489	
0.3 PPM SPIKE	6212	13897	0.319904	0.3	106.6348	105.7293
0.3 PPM SPIKE	6212	13681	0.314471	0.3	104.8239	
0.3 PPM SPIKE	6213	14924	0.343083	0.3	114.3617	111.2157
0.3 PPM SPIKE	6213	14284	0.324209	0.3	108.0697	

NA = NOT APPLICABLE

EXPECTED CONCENTRATION = (FORTIFICATION LEVEL, UG/G)(SAMPLE WT.)/(FINAL VOLUME)

SUMMARY OF RESULTS

	AVERAGE RECOVERY	STANDARD DEVIATION	RELATIVE * STANDARD DEVIATION
0.1 PPM SPIKE	99.1	7.4	7.4
0.3 PPM SPIKE	107.9	2.9	2.7

* RELATIVE STANDARD DEVIATION = (STANDARD DEVIATION/AVERAGE) X 100

Page 1

DA 4/1/91

Sequence Calibration Sample
File: D:\CHEMPC\DATA\BUTTER1\0901011.D
Operator: STEVE SUMMER
Date Acquired: 28 Mar 91 9:41 pm
Method File Name: PROPET1.M
Sample Name: CAL STD 25 NG/ML EXT #45547-~~59~~-09 62
Misc Info: SC900078
Bottle Number: 9

DSA 4/2/91

PROPETAMPHOS (SC900078)
Calibration Last Updated: Thu Mar 28 21:34:55 1991
External Standard

Reference Window: 5 Percent
Non-Reference Window: 5 Percent
Correlation Window: 0.03 minutes
Default Multiplier: 1
Default Sample Amount: 0

Compound Information

*** Compound 1 ***

Name: PROPETAMPHOS ( )
Ret. Time 9.51 min., Extract & Integrate from 9.41 to 9.61 min.

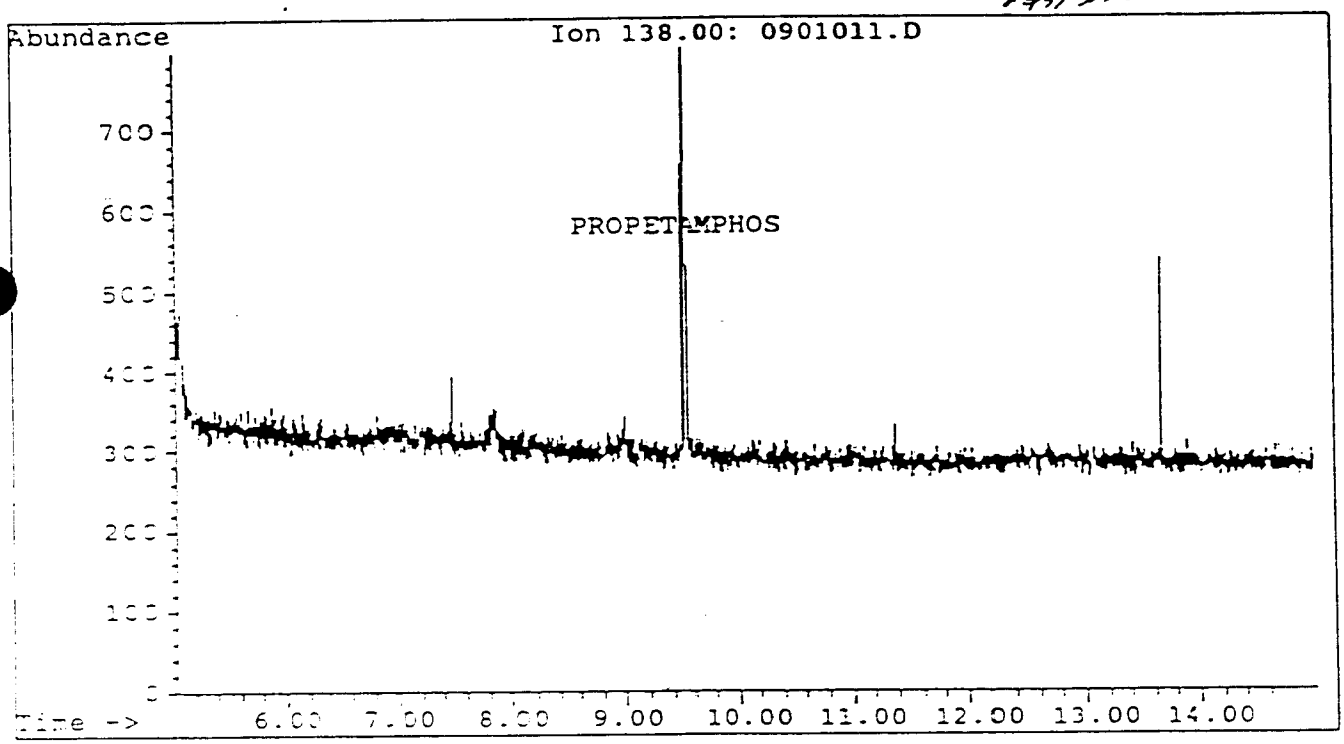
Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 138			PROPET1.E
Q1 194	0	20	PROPET1.E

Lvl ID	Amt (UG/G)	Response
1	1	534
5	5	1879
10	10	3165
25	25	7673
50	50	15728
100	100	36168

Qualifier Peak Analysis ON
Curve Fit: Linear, Forced Through Origin

Battelle Study Number SC900078
Zoecon Study Number 1538

File: D:\CHEMPC\DATA\BUTTER1\0901011.D
Operator: STEVE SUMMER
Date Acquired: 28 Mar 91 9:41 pm
Method File Name: PROPET1.M
Sample Name: CAL STD 25 NG/ML EXT #45547-~~58~~-09
Misc Info: SC900078
Bottle Number: 9



PROPETAMPHOS (SC900078) 4/1/91

Calibration Last Updated: Thu Mar 28 23:47:13 1991
External Standard Report - DETAILED

Information from Data File:
File: D:\CHEMPC\DATA\BUTTER1\1701004.D
Operator: STEVE SUMMER
Date Acquired: 29 Mar 91 10:46 am
Method File Name: PROPET1.M
Sample Name: BUTTER BLANK EXT #45547-~~58~~-14 61
Misc Info: SC900078
Bottle Number: 17 4/2/91

Report Style:
Graphics: (On) Off
Compounds: (All) Single

Quantitation Settings:
Reference Peak Window: 5 Percent
Non-Reference Peak Window: 5 Percent
Correlation Window: 0.03 minutes
Default Multiplier: 0.008
Default Sample Amount: 0
Peak Type decoding: * -> Time Reference Peak

Number of Compounds in Database: 1

Time Reference Peaks:

Pk#	Compound	Expected RT	Actual RT

PROPETAMPHOS (SC900078) 3: 4/1/91

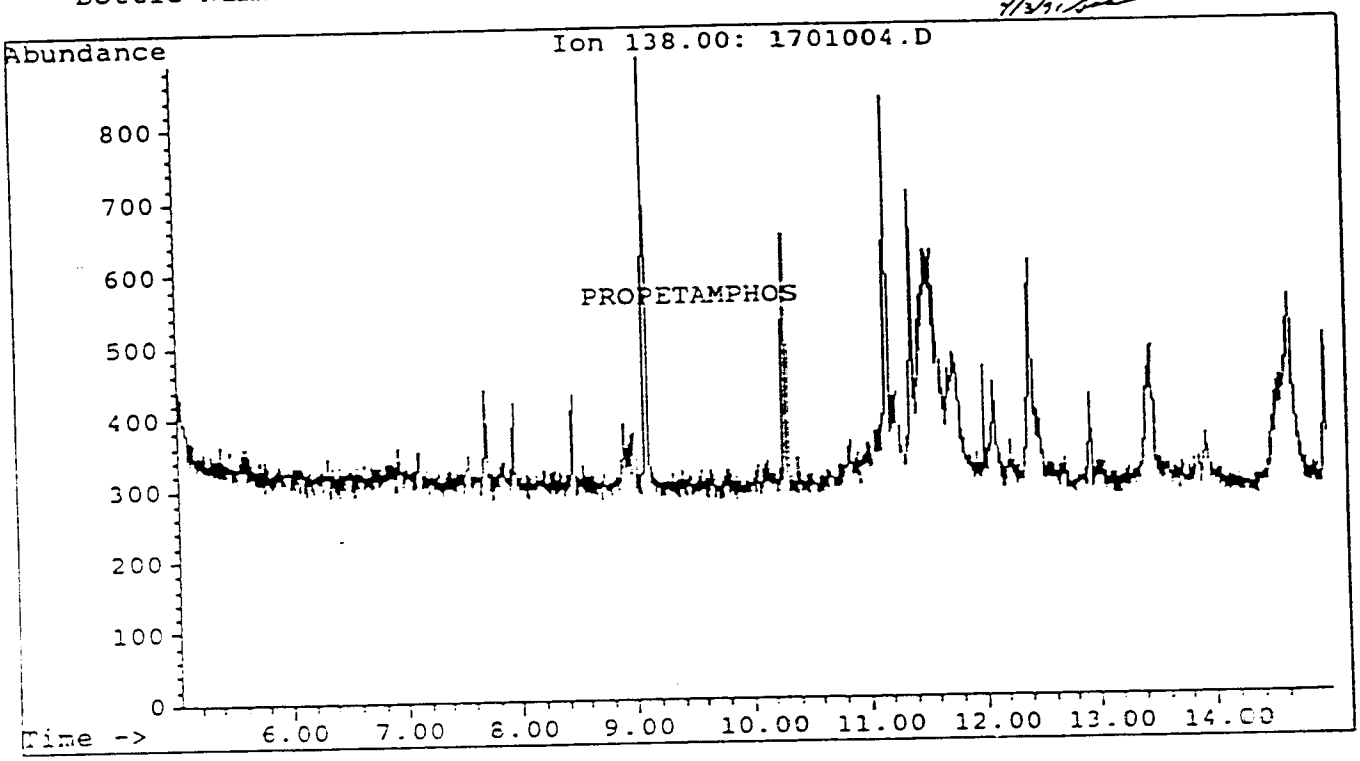
File: D:\CHEMPC\DATA\BUTTER1\1701004.D
Operator: STEVE SUMMER
Date Acquired: 29 Mar 91 10:46 am
Method File Name: PROPET1.M
Sample Name: BUTTER BLANK EXT #45547-~~58~~-14
Misc Info: SC900078 61
Bottle Number: 17 03: 4/2/91

Compound: PROPETAMPHOS
Ret Time:
Amount:
Pk # and Type: 1
***** NOT FOUND ****

Battelle Study Number SC900078
Zoecon Study Number 1538

File: D:\CHEMPC\DATA\BUTTER1\1701004.D
Operator: STEVE SUMMER
Date Acquired: 29 Mar 91 10:46 am
Method File Name: PROPET1.M
Sample Name: BUTTER BLANK EXT #45547-~~58~~-14 61
Misc Info: SC900078
Bottle Number: 17

4/3/91



PROPETAMPHOS (SC900078) BA 4/1/91

Calibration Last Updated: Thu Mar 28 23:47:13 1991
External Standard Report - DETAILED

Information from Data File:

File:	D:\CHEMPC\DATA\BUTTER1\1901006.D
Operator:	STEVE SUMMER
Date Acquired:	29 Mar 91 11:39 am
Method File Name:	PROPET1.M
Sample Name:	0.1 PPM BUTTER SPIKE EXT #45547-~~58~~-06 61
Misc Info:	SC900078
Bottle Number:	19

BA 4/2/91

Report Style:
Graphics: (On) Off
Compounds: (All) Single

Quantitation Settings:

Reference Peak Window:	5 Percent
Non-Reference Peak Window:	5 Percent
Correlation Window:	0.03 minutes
Default Multiplier:	0.008
Default Sample Amount:	0
Peak Type decoding:	* -> Time Reference Peak

Number of Compounds in Database: 1

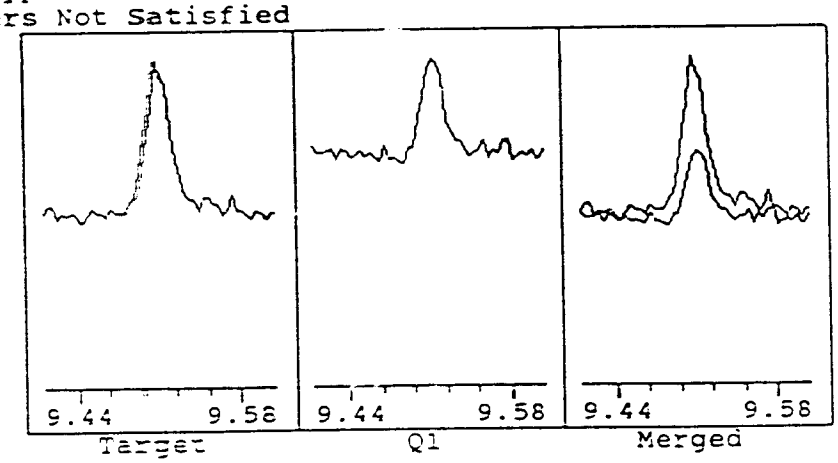
Time Reference Peaks:

Pk#	Compound	Expected RT	Actual RT

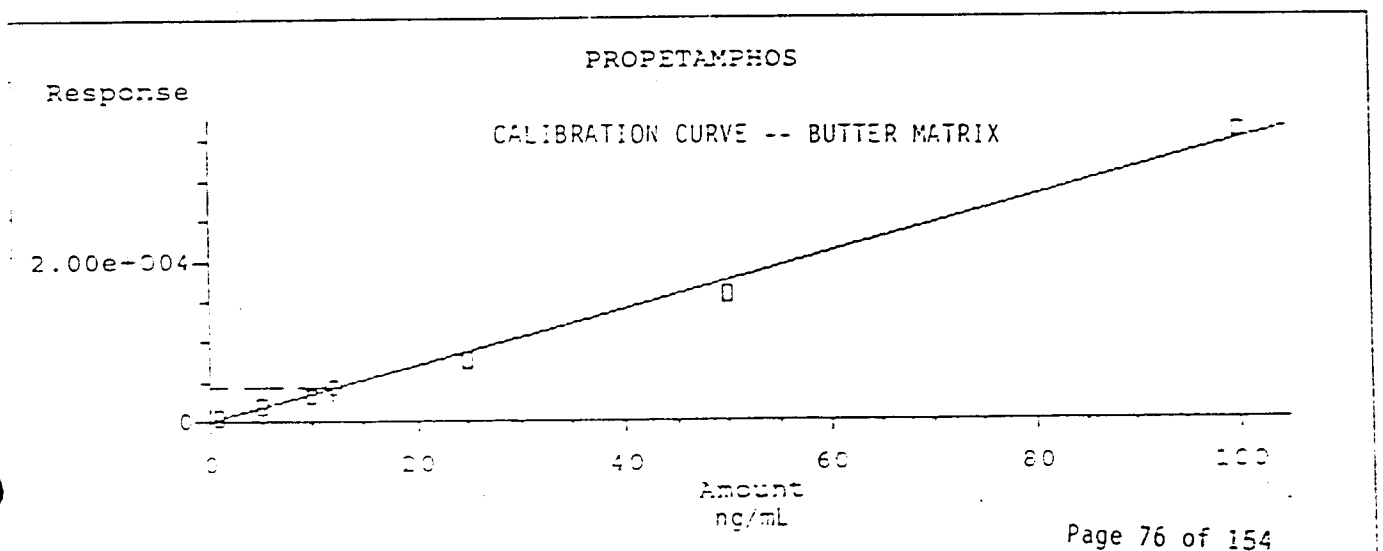
File: D:\CHEMPC\DATA\BUTTER1\1901006.D
Operator: STEVE SUMMER
Date Acquired: 29 Mar 91 11:39 am
Method File Name: PROPET1.M
Sample Name: 0.1 PPM BUTTER SPIKE EXT #45547-~~58~~-08 61
Misc Info: SC900078
Bottle Number: 19

4/2/91

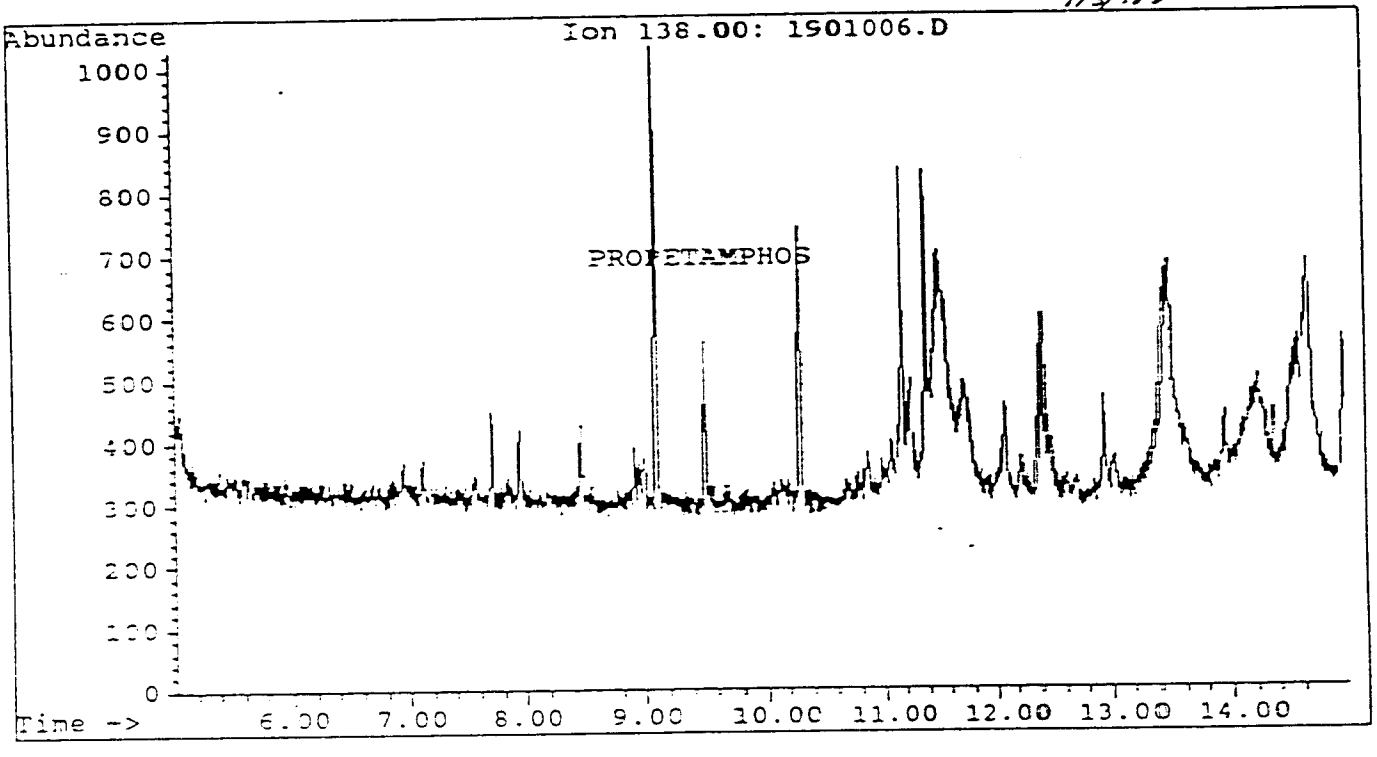
Compound: PROPETAMPHOS
Ret Time: 9.51
Amount: 0.10 UG/G
Pk # and Type: 1
Qualifiers Not Satisfied



	Signal	Ratios	Limits	RT	Limits	Area	Integ Type
Tgt	138	100.0%		9.51	9.27	4217	PROPET1
Q1	194	42.7	0.0-0.0	9.51	to	1801	PROPET1
Q2	0				9.75		
Q3	0						



Response = 3.48e-002 * Amt
Corr Coef = 0.995 Curve Fit: Linear/Origin

File: D:\CHEMPC\DATA\BUTTER1\1901006.D
Operator: STEVE SUMMER
Date Acquired: 29 Mar 91 11:39 am
Method File Name: PROPET1.M
Sample Name: 0.1 PPM BUTTER SPIKE EXT #45547-~~38~~-08
Misc Info: SC900078
Bottle Number: 19

4/3/91