

Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pesticide Name: Kresoxim Methyl (BAS490 F)

MRID #: 443410-15

Matrix: Water

Analysis: GC/ECD

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Study Title

GLC Method for the Analysis of BAS 490 F
and Its Metabolite, BF 490-1, In Aquatic Media

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1 of 39

PR 86-5 DATA CONFIDENTIALITY CLAIM

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COMPANY BASF CORPORATION / AGRICULTURAL PRODUCTS GROUP

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GOOD LABORATORY PRACTICES STATEMENT

Document No. ADPEN-903-92-A92125M-002

This project is not a study as defined by 40 CFR Part 160, and therefore is not required to comply with FIFRA Good Laboratory Practices.

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**GLC METHOD FOR THE ANALYSIS OF BAS 490 F AND
ITS METABOLITE, BF 490-1, IN AQUATIC MEDIA**

BASF ANALYTICAL METHOD D9209

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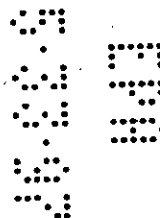


TABLE OF CONTENTS

	Page
STATEMENTS	
CONFIDENTIALITY STATEMENT.....	2
GOOD LABORATORY PRACTICES STATEMENT.....	3
INTRODUCTION	
1.0 INTRODUCTION	7
1.1 Scope	7
1.2 Source of the Method.....	7
1.3 Principle of the Method	7
2.0 SUMMARY.....	7
MATERIALS AND PROCEDURE	
3.0 EQUIPMENT.....	8
4.0 SAFETY.....	8
5.0 REAGENTS.....	9
6.0 INSTRUMENTATION.....	9
7.0 PREPARATION OF STANDARD SOLUTIONS.....	9
7.1 Stock Solutions	9
7.2 Standard Fortification Solutions	10
7.3 Standard Solutions for Gas Chromatography	10
8.0 SAMPLE WORKUP.....	10
8.1 Recovery Test.....	10
8.2 Preparation of Samples for Extraction.....	10
8.3 Extraction of Residue	10
9.0 CHROMATOGRAPHY.....	12
10.0 TIME REQUIRED FOR ANALYSIS.....	12
11.0 INTERFERENCES.....	12
11.1 Sample Matrices.....	12
11.2 Other Sources.....	12
12.0 CONFIRMATORY TECHNIQUES.....	13
13.0 METHOD OF CALCULATION.....	13
13.1 Standard Calibration Curve.....	13
13.2 Calculation of Equivalent Sample Weight.....	13
13.3 Determination of Sample Residues (ng).....	13
13.4 Determination of Sample Residues (ppb).....	14
13.5 Fortification Recoveries.....	14
RESULTS AND DISCUSSION	
14.0 RESULTS AND DISCUSSION	15
14.1 Test System/Test Substance	15
14.2 Quantitation.....	15
14.3 Accuracy and Precision	16
14.4 Limit of Detection and Quantitation.....	16
14.5 Conclusion.....	16

TABLE OF CONTENTS

Page

RESULTS AND DISCUSSION (Continued)

15.0	NOTES.....	16
16.0	RUGGEDNESS TESTING.....	17
17.0	LIMITATIONS.....	17
18.0	REFERENCES.....	17
19.0	CERTIFICATION	17

TABLES AND FIGURES

TABLES.....	18
FIGURES.....	32

33

1.0 INTRODUCTION

1.1 SCOPE

This method is used to determine the residues of BAS 490 F and BF 490-1 in aquatic media. An aquatic medium is extracted through a C18 solid phase extraction cartridge (SPE). The cartridge is extracted with methanol (MeOH). A sample of the extract is injected onto a GC/ECD. This gives the amount of BAS 490 F in the sample. An aliquot of the extract is then methylated and injected onto the GC/ECD. From this, a total amount of parent and hydrolysis product is determined. By subtracting the non-methylated result from the methylated one, an amount of, BF 490-1, can also be determined. BF 490-1 is a known aqueous hydrolysis product⁽¹⁾ of BAS 490 F.

1.2 SOURCE OF METHOD

The basic method for determining residues of BAS 490 F and BF 490-1 in water media was developed by BASF Corporation and modified by ADPEN Laboratories, Inc.

1.3 PRINCIPLE OF THE METHOD

An aliquot of aqueous medium is passed through a pre-conditioned C18 solid phase extraction column. BAS 490 F, the parent compound (a methyl ester) and its hydrolysis product, BF 490-1 (the corresponding acid), are eluted with 30 ml of methanol. The eluate is concentrated in a rotary evaporator to less than 10 ml at a water bath temperature in the range of 30 to 35°C. The sample is allowed to sit in the refrigerator prior to filtering with an Acrodisc syringe filter. An aliquot of the extract is injected onto a GC capillary column and analyzed for the parent compound using an electron capture detector. A 1 ml aliquot of the extract containing both the parent and hydrolysis product is methylated with TMS-diazomethane to convert the hydrolysis product to the parent. The resulting sample contains the "total" contribution of both the parent and derivatized hydrolysis product. It is then injected onto a GC capillary column and analyzed for the parent compound using an electron capture detector. The BF 490-1 contribution is determined by difference. A flow diagram of the method is shown in Figure 1. The structures of BAS 490 F and BF 490-1 are shown in Figure 2. Description of the reference substances can be found in Table I.

2.0 SUMMARY

BASF Analytical Method D9209 was validated for the analysis of aquatic media at a quantitation limit of 25 ppb for each compound. Procedural recoveries through the method for analyses of aquatic media fortified with BAS 490 F and BF 490-1 in the range of 25 ppb to 400 ppb were the following. For BAS 490 F, the procedural recoveries were $89 \pm 13\%$, $95 \pm 15\%$, $98 \pm 11\%$, and $102 \pm 11\%$ for BAS 490 F in

AAP, AAPSI, MAA, and 20X-AAP, respectively. For BF 490-1 the procedural recoveries were: $106 \pm 22\%$, $109 \pm 18\%$, $87 \pm 12\%$, and $97 \pm 18\%$ for BF 490-1 in AAP, AAPSI, MAA, and 20X-AAP respectively.

3.0 EQUIPMENT

Names of equipment manufacturers and brands are suggested. These may be substituted and equivalent equipment may be used.

- 3.1 Concentration tube, 50-ml, standard tapered joints, Corning or equivalent
- 3.2 Flasks, 24/40 standard tapered joints, 125 ml, flat bottoms
- 3.3 General laboratory glassware
- 3.4 N-Evap (Nitrogen Stream Evaporator): Organomation Assoc.
- 3.5 Rotary evaporator, Buchi Model RE 121 or equivalent, equipped with a heated water bath
- 3.6 Screw cap vials, 6 dram or 30 ml equivalent
- 3.7 Solid Phase Extraction, vacuum manifold, 24-port
- 3.8 Syringe Filter, Acrodisc® LC13 PVDF 0.45µ, Gelman
- 3.9 Syringe, gas tight, 5 ml, #1005, Hamilton, or 20-ml syringe
- 3.10 Volumetric flasks, miscellaneous sizes
- 3.11 Volumetric pipettes, type A, various sizes
- 3.12 Solid Phase Extraction (SPE) column, 500 mg, Octadecyl (C18), Varian
- 3.13 Reservoir, 20-ml, with connector for SPE column, Varian
- 3.14 Glass wool, silanized

4.0 SAFETY

All analysts must be familiar with the potential hazards of each of the reagents, solvents, and products used in this method before any laboratory work is done. Material Safety Data Sheets (MSDS), laboratory Safety Manual, product information, and other related materials should be consulted. Exposure to all chemicals should be reduced to the lowest possible level. Analysts should also be aware of OSHA regulations regarding the safe handling of the chemicals specified in this method. Disposal of all chemicals must be in compliance with local, state, and federal laws and regulations.

- 4.1 Trimethylsilyl (TMS) -diazomethane is a safer reagent and a substitute for diazomethane. Diazomethane is a very toxic and explosive yellow gas. Concentrated solutions may explode violently especially in the presence of impurities. Gaseous diazomethane may explode on heating above 90°C or on rough surfaces. Although TMS-diazomethane is safer, handle it with care, as it is an irritant and a highly flammable liquid. Avoid the use of glass stirrers or disposable pipettes. Keep refrigerated.
- 4.2 Acetone, acetonitrile, methanol, and n-propanol are flammable. Care should be taken to use these solvents in well ventilated areas away from ignition sources.
- 4.3 All open flask evaporations with an N-Evap should be done inside a hood.

5.0 REAGENTS

Names of chemical manufacturers and brands are suggested. These may be substituted and equivalent equipment may be used.

- 5.1 Acetonitrile, Burdick & Jackson, pesticide residue grade
- 5.2 Acetone, Burdick & Jackson, pesticide residue grade
- 5.3 Diazomethane, trimethylsilyl (TMS), 2.0M solution in hexane is recommended, Fluka, # 92738, or Aldrich Chemical Co. #36283-2
- 5.4 0.02M TMS-diazomethane in hexane, prepared from 2.0M solution of the reagent by taking a 1.0 ml aliquot and diluting to 100.0 ml with hexane. TMS-diazomethane is a safer reagent and a substitute for diazomethane. Handle with care due to the irritant and highly flammable nature of the liquid. See Section 4.0.
- 5.5 Methanol, Burdick & Jackson, pesticide residue grade
- 5.6 n-Propanol, Burdick & Jackson, pesticide residue grade
- 5.7 Water, deionized

6.0 INSTRUMENTATION

Names of instrument manufacturers are suggested, equivalent brands may be substituted.

- 6.1 Gas Chromatograph, Hewlett Packard 5890, Series II, or equivalent, equipped with electron capture detector operated according to conditions outlined in Table I.
- 6.2 Gas Chromatography Capillary Column, DB®-17, 30 meter column, 0.25um film thickness, 0.32 mm ID. J & W.
- 6.3 Inlet liner, 4mm ID., deactivated, tapered on one side, 5181-3316, Hewlett Packard.

7.0 PREPARATION OF STANDARD SOLUTIONS

All analytical standards shall be kept in the freezer. All standard solutions shall be kept in amber bottles and stored in a refrigerator. The details given for making dilutions are suggested. Concentrations and method of dilution may be modified if needed.

7.1 STOCK SOLUTIONS

Prepare a 0.10 mg/ml BAS 490 F stock solution by accurately weighing 0.0250 g of BAS 490 F standard into a 250 ml volumetric flask. Dissolve in acetonitrile and dilute to the mark. Prepare a 0.10 mg/ml BF 490-1 stock solution by accurately weighing 0.0250 g of BF 490-1 standard into a 250 ml volumetric flask. Dissolve in acetonitrile and dilute to the mark.

7.2 STANDARD FORTIFICATION SOLUTIONS

Prepare a 4.5 ng/ μ l mixed stock solution of BAS 490 F and the hydrolysis product, BF 490-1 from the 0.10 mg/ml stock solutions and dilute in deionized water. Make further dilutions in deionized water as needed. These solutions are used for fortification and shall be prepared fresh every two days.

7.3 STANDARD SOLUTIONS FOR GAS CHROMATOGRAPHY

These standards may be prepared either by serial dilution or from direct dilution of the BAS 490 F stock solution into methanol. The following concentrations are suggested: 1.0 ng/ μ L, 0.75 ng/ μ l, 0.375 ng/ μ l, 0.30 ng/ μ l, 0.15 ng/ μ l, 0.075 ng/ μ l, and 0.0375 ng/ μ l. Different preparation schemes may be used and additional standard concentrations may be prepared and used as needed.

8.0 SAMPLE WORKUP

8.1. RECOVERY TEST

The validity of the procedure should be demonstrated by recovery tests before analysis of unknown samples is attempted. An untreated sample (control) and two or three fortified samples shall also be processed with each set of samples analyzed. Typically, one of the fortification samples is run at the limit of quantitation. For each fortified sample, an appropriate volume of BAS 490 F and BF 490-1 mixed standard solution is added to a control medium sample. Fortifications are made into the sample after placing the sample on the C18 column.

8.2 PREPARATION OF SAMPLES FOR EXTRACTION

Each lot of SPE columns should be profiled to determine if the lot being used will provide adequate recovery of BAS 490 F and BF 490-1. Elution parameters may be modified if needed.

8.2.1 Keep all samples frozen until ready for analysis, thaw out sample and shake well to make homogeneous.

8.2.2 Connect a 20-ml reservoir to the C18 SPE column and condition by pulling 20 ml of n-propanol through the column using a vacuum of about 5 in. of mercury (Hg), followed by 20 ml of deionized water. Do not allow column to go dry at this step. The use of a one way stopcock to stop the flow is recommended.

8.3 EXTRACTION OF RESIDUE

8.3.1 Using a volumetric pipette, transfer 15 ml of the aqueous sample onto the reservoir on top of the conditioned C18 solid phase extraction column.

- 8.3.2 Using a vacuum of at least 3 in. of Hg., pull the sample through the cartridge. Do not save the eluant.
- 8.3.3 Dry the cartridge for 5 to 8 minutes with a vacuum of 5 in. Hg. to get rid of as much water as possible. Increase vacuum pressure several times during this time to force droplets of water through.
- 8.3.4 After removing the water from the cartridge, elute the sample with 30 ml of methanol and collect the eluate in an 6 dram or 30-ml vial.
- 8.3.5 Transfer the eluate quantitatively from the 6-dram vial into a 125 ml flat-bottom flask rinsing the vial with three small portions of methanol and transferring each to the flat-bottom flask.
- 8.3.6 Concentrate the eluate to approximately 7 ml, using a rotary evaporator at a water bath temperature in the range of 30 to 35°C.
- 8.3.7 Transfer quantitatively with methanol to a 10 ml volumetric flask or calibrated concentration tube and bring the extract to exactly 10.0 ml and mix. Refrigeration of the extract prior to filtration may be required, see Section 4.0. /S. 0.
- 8.3.8 Allow to come to room temperature if sample was refrigerated. Fit a 0.45µm Acrodisc syringe filter to a 5.0 ml gastight syringe and transfer quantitatively 5.0 ml of the extract to the syringe. Filter the extract through the syringe filter and into a 5.0 ml volumetric flask. Rinse the syringe with about 0.5 ml of methanol and pass through the filter and into the volumetric flask to dilute sample to the 5.0 ml mark.
- 8.3.9 Transfer approximately 1 ml of the extract into a GC vial and cap it. Save vial for injection together with the methylated samples on Section 8.3.13.
- 8.3.10 Take 1.0 ml of the extract from step 8.3.8 and place it in a 50-ml concentration tube. Add 10 ml of 0.02M TMS-diazomethane in hexane, add 5 ml of acetone, and mix well by swirling. Allow to sit for 30 minutes under a hood and then add 3 ml of MeOH. TMS-diazomethane is a safer reagent and a substitute for diazomethane, see Section 4.0. The sample size may be doubled in this step as long as the amount of reagent and solvents are doubled.
- 8.3.11 Take the methylated extract and blow it down with a gentle stream of nitrogen to just dryness using an N-Evap with a water bath temperature of approximately 45°C.
- 8.3.12 Add 3 ml of MeOH and repeat step 8.3.11 to remove traces of diazomethane. The sample should be colorless. If not, gently repeat step 8.3.11 only one more time. Dilute sample accurately to 1.0 ml or an appropriate volume with methanol.

8.3.13 Inject samples from 8.3.9 and 8.3.12 into a gas chromatograph equipped with an electron capture detector (ECD) as described in Section 9.0 below.

9.0 CHROMATOGRAPHY

- 9.1 The suggested chromatographic conditions are given in Table II. The chromatography should be checked for response whenever a new injection port liner, column, or instrument is used. Approximately 5 cm. of the inlet end of the GC column should be cut off if the peak shape or sensitivity deteriorates. The GC column should be conditioned with several injections of sample extracts and standards prior to injecting samples to be quantitated. See Figures 4 and 5 for typical chromatograms.
- 9.2 Change the deactivated glass injection port liner daily. The liner should contain a very small wad of silanized glass wool. Replace the septum daily or after approximately 100 injections have been made if HP 5181-1263 low-bleed red septa or equivalent is used.
- 9.3 Inject 1- μ l aliquots of the standards and samples. A small injection size is suggested to protect the capillary column, but 2- μ l injection size may be used if necessary. Calibrate the detector response and retention times by injections of the standard solutions throughout a set of analyses. Standards shall also be injected at the beginning and at the end of a set of analyses.
- 9.4 Analytical recoveries are determined as described in Section 13.5. Sample residues are determined as described in Section 13.1 through 13.4.

10.0 TIME REQUIRED FOR ANALYSIS

Analysis of a set of 10 media water samples requires about 7 man hours. GC analysis may be done overnight by autosampler. Data entry, integration and data report may take up to an additional 3 man hours.

11.0 INTERFERENCES

11.1 Sample Matrices

Baseline resolution was attained for BAS 490 F when using the DB-17 column. If interfering peaks from the matrix occur in the chromatogram, change the GC operating conditions or use an alternate GC column such as DB-1701 or DB-5 of the same length.

11.2 Other Sources

BAS 490 F is resolved from a background unknown peak with the DB-17 column. No interfering peaks from pesticides, solvents, or labware known to date.

12.0 CONFIRMATORY TECHNIQUES

No problems with interferences or questionable peak identity have been encountered to date. A GC column with different polarity, such as a DB-5, 30 meter column, may be used for confirmation if necessary.

13.0 METHODS OF CALCULATION

13.1 STANDARD CALIBRATION CURVE

Each standard should be injected at least twice in the analysis set. Standards are injected at the beginning, after every 1 to 3 samples and at the conclusion of the analysis. The peak height or peak area for each injected standard, for at least four concentration levels, is determined by manual measurement or computer integration using a chromatography data system. Regression analysis of peak height or peak area versus nanograms injected may be performed by a scientific calculator or a computer chromatography data system. This regression analysis gives an equation for a standard curve for calculation of sample concentration. The standard curve should have a correlation coefficient (r) of 0.9800 or better. A scientific calculator or a computer chromatography data system is used to calculate nanograms injected from the slope and intercept of the standard curve and the chromatographic peak height or area of each sample injection.

13.2 CALCULATION OF EQUIVALENT SAMPLE WEIGHT

The milligrams of sample injected must be determined to calculate ppb (Section 13.4). The equivalent sample weight in the final solution is calculated as follows:

$$\text{mg inj.} = \frac{(W)(V_i)}{(V_e)} \times \frac{(V_r) \times 1000}{(V_i)}$$

W = weight of sample extracted (g) (1 ml=1 g for clear water only)
 1000 = conversion factors (mg/g)
 V_a = aliquot volume (ml) (section 8.3.9 for parent or 8.3.10 for BF 490-1)
 V_e = volume of the extract (ml), prior to sample aliquoting (section 8.3.7)
 V_r = total volume of final injection soln. (ul)
 V_i = injection volume (ul)

13.3 DETERMINATION OF SAMPLE RESIDUES (NANOGRAMS)

The peak height or area from a sample injection (Section 13.1) and the slope and intercept of the standard curve (Section 13.1) are used to determine the nanograms of residue in each sample injection. This can be done by a chromatography data system, calculator or by graphing a standard curve of nanograms injected versus detector response. The next section shows how to calculate sample residues in parts-per-billion.

13.4 DETERMINATION OF SAMPLE RESIDUES (PPB)

Calculate the sample residue for each sample expressed in terms of parts-per-billion (ppb) using the following equation:

$$\text{ppb of BAS 490 F found} = \frac{(\text{ng of BAS 490 F found } ^1) \times 1000^3}{(\text{mg of sample injected } ^2)}$$

¹ Section 13.3

² Section 13.2

³ Conversion factor (ppb/ppm)

To calculate results for BF490-1, the BAS 490 F ppb found in the derivatized extract (total BAS 490 F) is subtracted from the BAS 490 F ppb found in the non-derivatized extract.

$$\text{ppb BF490-1} = (\text{ppb of BAS 490 F, total} - \text{ppb of BAS 490 F}) \times 0.9553$$

Where 0.9553 = the molecular weight correction factor for the conversion of BAS490F to BF490-1 analytes.

$$= \frac{\text{Gram Molecular Weight of BF 490-1 (289.34)}}{\text{Gram Molecular Weight of BAS 490 F (313.34)}}$$

13.5 FORTIFICATION RECOVERIES

The ppb of compound found in the final solution (Section 13.4) is divided by the amount of compound added to the control sample. This ratio times 100 is the percent recovery of the method at that level of fortification.

$$\% \text{ Recovery} = \frac{\text{ppb analyte found in injected solution}}{\text{ppb analyte added to control sample}} (100)$$

If the control sample shows a chromatographic response corresponding to the analyte(s) of interest, the ppb value corresponding to this control sample response should be subtracted from the ppb residues found in the fortified samples before the percent recovery calculation is made, i.e.:

$$\text{ppb found in recovery} = \text{ppb in fortified sample} - \text{ppb in control sample}$$

Because the residue determination of the hydrolysis product is calculated from the difference between parent residues and total residues, a lower or higher recovery of parent than 100% will affect the percent recovery of the hydrolysis product by increasing or decreasing the percent recovery value. An average recovery value between 70 and 120% for each set of analysis should be acceptable for this type of procedure.

14.0 RESULTS AND DISCUSSION

An on going validation of this method was conducted during the analysis of aquatic media samples (BASF Report # ER93033²⁰). Aquatic media were fortified at 25, 100, 200, 300, and 400 ppb with BAS 490 F and BF 490-1. Fortification samples were spiked with both the parent and hydrolysis product, or with the parent only prior to extraction.

14.1 TEST SYSTEM/TEST SUBSTANCE

The test system for this project, aquatic media, was obtained by BASF Corporation, Research Triangle Park, NC. from Malcolm Pirnie, Inc., Tarrytown, NY.

The aquatic media consisted of five algae (*Selenastrum capricornutum* a green algae, *Anabaena flos-aquae* a blue-green algae, *Navicula pelliculosa* a freshwater unicellular, non-motile diatom, *Skeletonema costatum* a marine diatom, and *Lemna gibba* a duckweed) mixed with four different nutrient medium (AAP, AAP/Si, MAA, and 20X-AAP). *Selenastrum capricornutum* and *Anabaena flos-aquae* were prepared with a composition of synthetic algal assay procedure nutrient medium (AAP). *Navicula pelliculosa* was prepared with a composition of AAP and 20 mg/L of silicon. *Lemna gibba* was prepared with a composition of 20X-AAP. *Skeletonema costatum* was prepared with a composition of synthetic marine algal assay nutrient medium (MAA). AAP consisted of a number of inorganic compounds including metals and salts of which the largest concentration was 11 mg/L of NaHCO_3 . MAA consisted of a number of inorganic compounds, $\text{Na}_2\text{EDTA} \cdot 2\text{H}_2\text{O}$, and a vitamin mix.

The control aquatic media was placed in sample bottles and then in an insulated container and shipped overnight from Malcolm Pirnie. The samples were received at ADPEN Laboratories, Inc. on 12/11/92, 1/26/93, and 3/5/93 and were stored frozen in ADPEN's freezer E6. Unique sample laboratory codes were assigned just prior to analysis. The laboratory sample code consists of year code, project code, and sample number (i.e., 93-BB100). Test and reference substances are listed in Table I.

14.2 QUANTITATION

Gas chromatographic quantitation was achieved by measuring peak heights. Results were calculated from a standard curve prepared by plotting detector response in peak height versus nanograms of compound injected. An equation for the fit of the standard curve was derived, and the correlation coefficient of the regression curve calculated for all analytical sets. (See Figure 3).

Integration and quantitation of peaks were done by computer using Hewlett-Packard ChemStation Chromatography Data System. Final results were computed for each set of samples by the use of a Lotus 1-2-3 spreadsheet. Statistical treatment of the data included determination of averages and standard deviations for the recovery data.

14.3 ACCURACY AND PRECISION

The accuracy is a measure of the difference between the determined and accepted true values. Accuracy is calculated as the range and average of relative errors. The error is defined as the nearness of the analytical measurement to its accepted value. The average relative error in aquatic media sample analyses as determined from recovery values ranging from 25 to 400 ppb was $4.7 \pm 14\%$ ($n = 128$) for BAS 490 F and $1.7 \pm 20\%$ ($n = 48$) for BF 490-1.

The standard deviation (square root of variance) is an estimate of precision. Precision is a measure of the scatter in repeated determinations from repeated chromatographic measurements. The precision (percent recovery $\pm$ standard deviation) for analyses of aquatic media fortified with BAS 490 F and BF 490-1 in the range of 25 ppb to 400 ppb were $89 \pm 13\%$, $95 \pm 15\%$, $98 \pm 11\%$, and $102 \pm 11\%$ for BAS 490 F in AAP, AAPSI, MAA, and 20X-AAP respectively and $106 \pm 22\%$, $109 \pm 18\%$, $87 \pm 12\%$, and $97 \pm 18\%$ for BF 490-1 in AAP, AAPSI, MAA, and 20X-AAP respectively. Individual results are summarized in Tables III and IV.

14.4 LIMIT OF DETECTION AND QUANTITATION

The limit of detection for BAS 490 F and BF 490-1 using a DB[®]-17 capillary column is 12.5 ppb, based on standards injected at half the quantitation limit. The limit of quantitation for BAS 490 F and BF 490-1 determined by this procedure, in the analysis of aquatic media samples is equal to 25 ppb for each compound.

14.5 CONCLUSION

Analytical Method D9209 is a valid and accurate method for determining residues of BAS 490 F and BF 490-1 in aquatic media.

15.0 NOTES

- 15.1 The aquatic media used for this validation contains a relative large number of inorganic salts and metals that are not removed by the C18 extraction column. These inorganic components in the media coat the GC glass insert and the inlet side of the capillary column causing chromatographic problems related to sensitivity. These salts also appear to cause the autosampler syringe plunger to stick. To correct these problems a filtration step was included in the method. For the aquatic media substrates used in the validation of this procedure these inorganic components could be observed as a colloidal suspension and/or larger particulates in the sample extract when kept in a refrigerator for at least one day. It is recommended that the samples be refrigerated overnight or for a day, be brought to room temperature, and then filtered as noted in Section 8.3.8.

16.0 RUGGEDNESS TESTING

This method has been used successfully at both BASF and ADPEN Laboratories, Inc. by at least three analysts.

17.0 LIMITATIONS

None known to date.

18.0 REFERENCES

1. Bleber, W. D., "Hydrolysis of Test Substance 242009 (BAS 490 F)," Draft Final Report.
2. Jackson, S. H. "Tier 1 Non-Target Aquatic Plant Toxicity Studies On BAS 490 F (242009)" BASF Report No. ER93033.

19.0 CERTIFICATION

I, the undersigned, hereby declare that the data referenced in this report was generated under my supervision according to the procedure described herein. The data and experimental results reported in this document are certified to be authentic accounts of the experiments conducted at ADPEN Laboratories, Inc.


Rolando Perez
Lab Manager/Technical Director
ADPEN Laboratories, Inc.

8/20/92
Date

TABLE I. Description of Reference Substances

Reference Substances:

1. Common Name:	Not available
2. Chemical Name:	methyl-(E)-methoxymino(α -(o-tolyl)oxy)-o-tolylacetate
3. Experimental Name:	BAS 490 F
4. CAS Number:	none
5. BASF Lot number:	CH397149-1
6. Purity:	99.6%
7. Date Received at ADPEN:	12/15/92
8. Storage Conditions at ADPEN:	Freezer, in the dark
9. ADPEN Code Number:	P-294
10. Expiration or Reassay Date: a/	12/94
11. Empirical formula:	C ₁₇ H ₁₉ NO ₄
12. Molecular weight:	313.34
13. Melting Point:	101°C
14. Appearance:	Colorless crystals
15. Odor:	None
16. Partitioning Coefficient: 1 g. Pow	3.4 (n-octanol/water)
17. Solubility (mg/liter at 20°C)	
Acetone	Not available
Acetonitrile	Not available
DMSO	Not available
Ethanol	Not available
n-Hexane	Not available
Methylene chloride	Not available
Water	2.0
Std. fat	17.2 g/kg

1. Common Name:	NA
2. Chemical Name:	(E)-methoxymino(α -(o-tolyl)oxy)-o-tolylacid
3. Experimental Name:	BF490-1 or BF490-A
4. CAS Number:	none
5. BASF Lot number:	LS1/9
6. Purity:	99.9%
7. Date Received at ADPEN:	12/15/92
8. Storage Conditions at ADPEN:	Freezer, in the dark
9. ADPEN Code Number:	P-293
10. Expiration or Reassay Date: a/	12/94
11. Empirical formula:	C ₁₇ H ₁₇ NO ₄
12. Molecular weight:	299.34
13. Solubility (mg/liter, water, 20°C)	Not available

Analytical standards supplied by:

Landwirtschaftliche
Versuchsstation der BASF
Produktsicherheit
Pflanzenschutz
Produktchemie
Gebäude U 444/GLP-Archiv von APS/U
Postfach 220
D-6703 Limburgerhof, Germany

a/ The purity of these materials has been determined under GLP's by BASF. BASF has archived an aliquot of these standards and has access to documentation relating to the synthesis and characterization of these compounds, including expiration dates / reassay dates. If no expiration or reassay date is available upon receipt of standards, ADPEN Laboratories, Inc. assigns two years after receipt of standard for the expiration date.

Table II. Suggested Gas Chromatographic Conditions

Gas Chromatograph: HP 5890 Series II

Initial Oven Temperature: 50° C
Initial Hold Time: 0.75 min.

Temperature Program:

	Rate:	Final Temp.:	Final Time:
Ramp 1	37° C/min.	280° C	2.0 min.
Ramp 2	40° C/min.	300° C	0.0 min.

Injector Temperature: 200° C
Injection Type: Splitless
Injection Volume: 1 or 2 µl, (1 µl is preferred)
Purge Valve: On Time: 1.0 min.
Off Time: 8.5 min.
Detector: Electron Capture

Detector Gases: Auxiliary: 5% Methane/Argon at 60 ml/min (Nitrogen may also be used)

GC Columns: DB®-17, 30 meters, 0.25 µm film thickness, 0.32 mm ID.

Carrier Gas: Helium, at 10 psi column back pressure with a regulated carrier source pressure of 40 psi.

Septum Purge: Helium, at 1.1 ml/min

Total Flow at Split Vent: Helium, at 150.0 ml/min

Minimum Response: 0.0375 ng of BAS 490 F

Approximate Retention Time: 8.3 min.

No needle residence time is specified. A fast injection type autosampler was used for the method validation. These are suggested conditions and may be modified if needed if it provides similar or better chromatography. Setting of carrier source pressure to 40 psi and column back pressure to 10 psi with above column will produce a set linear velocity, this linear velocity was not measured.

Table III. Summary Of Fortification Recoveries Of BAS 490 F And BF 490-1 In Aquatic Media Using Method D9209.

SUBSTRATE	FORTIFICATIONS									
	25 PPB		100 PPB		200 PPB		300 PPB		400 PPB	
	BAS 490 F	BF 490-1	BAS 490 F	BF 490-1	BAS 490 F	BF 490-1	BAS 490 F	BF 490-1	BAS 490 F	BF 490-1
AAP	61.6	128.9	67.7	97.3	88.2	148.0	96.1	68.0	99.7	68.1
AAP	64.3	123.8	98.0	90.7	93.7	92.2	99.0	107.1		
AAP	70.1	121.3	94.3	116.9	78.7	122.0	110.8	82.6		
AAP			110.0	96.8	95.0	89.2	78.7	119.7		
AAP			86.7	117.8	81.3	123.6	93.1			
AAP							86.9			
AAP							84.9			
AAP							84.4			
AAP							87.2			
AAP							80.0			
AAP							79.7			
AAP							75.8			
AAP							122.3			
AAP							96.0			
AAP							108.9			
AAP							102.2			
AAP							92.9			
AAP							80.2			
AAP							100.0			
AAP							90.6			
AAP							72.2			
AAP							77.2			
AAP							96.1			
AAP							101.1			
AVG. % REC	65.3	124.7	91.3	103.9	87.4	115.0	91.5	94.4	99.7	68.1
STD. DEV.	4.3	3.9	15.7	12.6	7.3	24.5	12.4	23.4	-	-
N =	3	3	5	5	5	5	24	4	1	1
AAP	BAS 490 F		Avg.		Std. Dev.		13.6		N = 38	
AAP	BF 490-1		Avg.		Std. Dev.		21.9		N = 18	

Table III. Summary Of Fortification Recoveries Of BAS 490 F And BF 490-1 In Aquatic Media Using Method D9209 (Continued).

SUBSTRATE	FORTIFICATIONS											
	25 PPB			100 PPB			200 PPB			300 PPB		
	BAS 490 F	BF 490-1		BAS 490 F	BF 490-1		BAS 490 F	BF 490-1		BAS 490 F	BF 490-1	
AAPSI	81.6	113.1		96.7	76.4		96.5	92.8		86.2	117.2	
AAPSI	83.2	110.1		143.3	85.7		105.7	113.4		103.3	122.6	
AAPSI	86.9	101.1		118.7	119.7		91.1	143.5		94.0	116.1	
AAPSI										93.6		
AAPSI										93.3		
AAPSI										90.8		
AAPSI										70.0		
AAPSI										73.9		
AAPSI										85.8		
AAPSI										79.4		
AAPSI										119.7		
AAPSI										89.3		
AAPSI										107.8		
AAPSI										95.6		
AAPSI										98.0		
AAPSI										94.0		
AAPSI										88.3		
AAPSI										88.7		
AAPSI										76.7		
AAPSI										72.8		
AAPSI										102.2		
AAPSI										110.6		
AAPSI										107.8		
AVG. % REC	83.9	108.1		119.6	93.9		97.8	116.6		92.2	118.6	
STD. DEV.	2.7	6.2		23.3	22.8		7.4	25.5		12.8	3.5	
N =	3	3		3	3		3	3		3	3	
AAPSI	BAS 490 F			Avg.			Std. Dev.			15.2 N = 32		
AAPSI	BF 490-1			Avg.			Std. Dev.			18.0 N = 12		
				109.3								

Table III. Summary Of Fortification Recoveries Of BAS 490 F And BF 490-1 In Aquatic Media Using Method D9209 (Continued).

SUBSTRATE	FORTIFICATIONS							
	25 PPB		100 PPB		200 PPB		300 PPB	
	BAS 490 F	BF 490-1	BAS 490 F	BF 490-1	BAS 490 F	BF 490-1	BAS 490 F	BF 490-1
MAA	106.1	83.3	99.7	102.3	110.0	89.0	106.1	81.7
MAA	110.7	86.1	99.3	91.7	93.0	101.3	91.8	80.1
MAA	105.3	89.9					88.3	
MAA							85.6	
MAA							70.0	
MAA							86.2	
MAA							113.9	
MAA							99.7	
MAA							79.4	
MAA							83.1	
MAA							89.0	
MAA							88.7	
MAA							99.8	
MAA							100.4	
MAA							98.7	
MAA							91.1	
MAA							101.1	
MAA							95.0	
MAA							106.7	
MAA							114.4	
MAA							113.9	
MAA							105.0	
AVG. % REC	107.4	86.4	99.5	97.0	101.5	95.2	95.7	70.9
STD. DEV.	2.9	3.3	0.3	7.5	12.0	8.7	11.7	15.3
N =	3	3	2	2	2	2	22	2
MAA	BAS 490 F		Avg.		Std. Dev.		N = 29	
MAA	BF 490-1		Avg.		Std. Dev.		N = 9	
			97.6		87.3		12.4	

Table III. Summary Of Fortification Recoveries Of BAS 490 F And BF 490-1 In Aquatic Media Using Method D9209 (Continued).

SUBSTRATE	FORTIFICATIONS							
	25 PPB		100 PPB		200 PPB		300 PPB	
	BAS 490 F	BF 490-1	BAS 490 F	BF 490-1	BAS 490 F	BF 490-1	BAS 490 F	BF 490-1
20X-AAP	113.1	78.9	83.0	122.0	92.7	120.0	91.1	91.1
20X-AAP	116.0	81.3	110.7	82.8	101.3	91.7	90.6	90.6
20X-AAP	117.3	81.0					89.1	89.1
20X-AAP							90.0	90.0
20X-AAP							95.3	95.3
20X-AAP							95.6	95.6
20X-AAP							90.3	90.3
20X-AAP							87.8	87.8
20X-AAP							102.0	102.0
20X-AAP							105.0	105.0
20X-AAP							104.2	104.2
20X-AAP							101.1	101.1
20X-AAP							98.3	98.3
20X-AAP							98.9	98.9
20X-AAP							103.3	103.3
20X-AAP							102.2	102.2
20X-AAP							120.6	120.6
20X-AAP							124.4	124.4
20X-AAP							125.0	125.0
20X-AAP							111.7	111.7
AVG. % REC	115.5	79.7	101.9	102.4	97.0	105.9	101.3	98.2
STD. DEV.	2.2	2.5	12.5	27.7	6.1	20.0	11.4	1.5
N =	3	3	2	2	2	2	20	2
20X-AAP	BAS 490 F		Avg.	102.3	Std. Dev.	10.9	N = 29	
20X-AAP	BF 490-1		Avg.	96.8	Std. Dev.	17.9	N = 8	

TABLE IV. Individual Recoveries of BAS 490 F and BF 490-1 In Aquatic Media.

[illegible]

Control sample used: 52

Sample Weight (g):

Low Dose and re-injected

$$\% \text{ PPA Found} = \frac{\text{PPA Found}}{\text{PPA Found} + \text{PPA Not Found}} \times 100$$

Corrected to convert BAS190°F to 84°F (0.9553 factor).

of H₂O₂ additions were added prior to extraction and were run concurrently with control samples.

Reaction volume was constant at 1 or 2.4. The con-

can produce a, perhaps even better, than the best of us.

patented drugs are put under scrutiny to help the public understand the value of the drugs and the quality of the information provided. The public is also being educated about the importance of the information provided by the public and the importance of the information provided by the public.

[illegible]

Control sample weight: 53
18.0
a/ Dried and re-lyophilized
b/ PBS Found-PPB Net = (No Found - Ig in control) $\times$ (mg/lyo) (mg lyoized)
c/ Corrected to convert DA-1007 to B5-100-1 (0.953 factor).
d/ Percent Recovery = (ppb Net/DA-1007) $\times$ 100
e/ Percent Recovery = (ppb Net/DA-1007) $\times$ 100
f/ The volume was constant at 1 or 2 μ l. The sample size was 15.0 grams. Although this table may have been rounded off for reporting purposes, but not for any further calculations. The PPB Net values were corrected for apparent moisture in the controls, values these residues were less than the limit of quantitation 7750 was assigned for these residues.

[illegible]

Control sample used: 54
Sample Waste (n):

^d PF9 Found=PF9 Net = ((mg Found - mg in control) x 1000)/(mg injected) of PF9 added to control BAS190F to BF490-1 (0.953 factor).

Net recovery = $[(\text{DPO Net})/(\text{DPO Added})] \times 100$
Reaction volume was constant at 1 or 2 μL . The sample size was 15.0 grams. Values in this table may have been rounded off for reporting purposes, but not for any further calculations. The DPO Net values were corrected for apparent residues in the control, although these residues were less than the limit of quantitation and were extrapolated.

Individual Records of BAS 490 F in Aquatic Media																
Media	No.	Lab Code	Vol. ml	Basal conc. ml	Aliquot	g	Vol/ml	Mg in BAS 490F	Peak Ht BAS 490F	fig Found BAS 490F	Found at BAS 490F	Final BAS 490F	PFB Added	Recor. % C Basal	Extrn. Date	Intn. Date
AAP	52	93-BB1R	10.0	1.0	1.5	2	1.0	3.00	0	0.0000	0.0	0.0	0.0	93.1	02/05/93	02/07/93
AAP	52	93-BB2R	10.0	1.0	1.5	2	2.0	1.50	76012	0.4180	279.3	279.3	300.0	88.9	02/05/93	02/07/93
AAP	52	93-BB3R	10.0	1.0	1.5	2	2.0	1.50	71599	0.3910	280.7	260.7	300.0	84.8	02/05/93	02/07/93
AAP	52	93-BB4R	10.0	1.0	1.5	2	1.0	3.00	0	0.0000	0.0	0.0	0.0	94.4	02/04/93	02/06/93
AAP	52	93-BB5R	10.0	1.0	1.5	2	1.0	3.00	143038	0.7640	254.7	254.7	300.0	84.8	02/04/93	02/06/93
AAP	52	93-BB102	10.0	1.0	1.5	2	1.0	3.00	142385	0.7600	253.3	253.3	300.0	84.8	02/04/93	02/06/93
AAP	52	93-BB103	10.0	1.0	1.5	2	1.0	3.00	0	0.0000	0.0	0.0	0.0	87.2	02/11/93	02/24/93
AAP	52	93-BB104	10.0	2.0	3.0	2	5.0	1.20	22340	0.3140	281.7	281.7	300.0	80.0	02/11/93	02/24/93
AAP	52	93-BB130	10.0	1.0	1.5	2	1.0	3.00	20598	0.2880	240.0	240.0	300.0	80.0	02/11/93	02/24/93
AAP	52	93-BB131	10.0	1.0	1.5	2	1.0	3.00	0	0.0000	0.0	0.0	0.0	79.7	02/25/93	03/09/93
AAP	52	93-BB132	10.0	4.0	6.0	2	10.0	1.20	23536	0.2870	239.2	239.2	300.0	75.8	02/25/93	03/09/93
AAP	52	93-BB167	10.0	4.0	6.0	2	10.0	1.20	22206	0.2730	227.5	227.5	300.0	75.8	02/25/93	03/09/93
AAP	52	93-BB187	10.0	1.0	1.5	1	1.0	1.50	0	0.0000	0.0	0.0	0.0	122.3	03/10/93	03/10/93
AAP	52	93-BB188	15.0	1.0	1.0	1	1.0	1.00	32822	0.3670	367.0	367.0	300.0	96.0	03/10/93	03/10/93
AAP	52	93-BB209	15.0	1.0	1.0	1	1.0	1.00	25274	0.2880	288.0	288.0	300.0	96.0	03/10/93	03/10/93
AAP	52	93-BB207	10.0	1.0	1.5	1	1.0	1.50	0	0.0000	0.0	0.0	0.0	108.8	03/03/93	03/20/93
AAP	52	93-BB208	25.0	1.0	0.8	1	1.0	0.60	47002	0.1960	328.7	328.7	300.0	102.2	03/03/93	03/20/93
AAP	52	93-BB209	25.0	1.0	0.8	1	1.0	0.60	44507	0.1840	306.7	306.7	300.0	102.2	03/03/93	03/20/93
AAP	53	93-BB167	10.0	5.0	7.5	1	5.0	1.50	563	0.0000	0.0	0.0	0.0	92.9	03/03/93	03/20/93
AAP	53	93-BB168	10.0	5.0	7.5	1	5.0	1.50	95198	0.4180	278.7	278.7	300.0	80.2	03/03/93	03/20/93
AAP	53	93-BB169	10.0	5.0	7.5	1	5.0	1.50	82874	0.3810	240.7	240.7	300.0	100.0	03/29/93	03/31/93
AAP	53	93-BB235	10.0	5.0	7.5	1	5.0	1.50	0	0.0000	0.0	0.0	0.0	90.8	03/29/93	03/31/93
AAP	53	93-BB236	25.0	5.0	3.0	1	5.0	0.60	40246	0.1800	300.0	300.0	300.0	100.0	03/29/93	03/31/93
AAP	53	93-BB23														

Sample Weight (g): 15.0

a/ PPB Found-PPB Net = (mg Found - ng in control) x MW Factor x 1000/(mg injected)

b/ Forifications were added prior to extraction and were run concurrently with control samples.

c/ Percent Recovery = (ppb Net/ppb Added) x 100

d/ Injection volume was constant at 1 or 2ul. The sample size was 15.0 g. Values in this table may have been rounded off for reporting purposes, but not for any further calculations.

The PPB Net values were corrected for apparent residues in the controls, although these residues were less than the limit of quantitation and were extrapolated.

TABLE IV. Individual Recoveries of BAS 490 F and BF 490-1 in Aquatic Media (Continued).

Media	Sample No.	Lab. Code	Vol. ml	Basal concn ml	g	Aliquot ml	Mg In BAS490F	Peak Rt. BAS490F	Found a/b	PPB	Found b/	PPB b/	Recovery %	Extraction Date	Injection Date
AAPSI	53	93-BB6R	10.0	1.0	1.5	2	1.0	3.00	0.0000	0.0	0.0	0.0	0.0	02/05/93	02/07/93
AAPSI	53	93-BB7R	10.0	1.0	1.5	2	2.0	1.50	76716	282.0	282.0	300.0	94.0	02/05/93	02/07/93
AAPSI	53	93-BB8R	10.0	1.0	1.5	2	2.0	1.50	76325	280.7	280.7	300.0	93.6	02/05/93	02/07/93
AAPSI	53	93-BB53	10.0	1.0	1.5	2	1.0	3.00	0.0000	0.0	0.0	0.0	0.0	02/04/93	02/06/93
AAPSI	53	93-BB54	10.0	1.0	1.5	2	1.0	3.00	156746	280.0	280.0	300.0	93.3	02/04/93	02/06/93
AAPSI	53	93-BB55	10.0	1.0	1.5	2	1.0	3.00	152678	272.3	272.3	300.0	90.8	02/04/93	02/06/93
AAPSI	53	93-BB107	10.0	1.0	1.5	2	1.0	3.00	0.0000	0.0	0.0	0.0	0.0	02/11/93	02/24/93
AAPSI	53	93-BB108	10.0	2.0	3.0	2	5.0	1.20	18115	210.0	210.0	300.0	70.0	02/11/93	02/24/93
AAPSI	53	93-BB135	10.0	1.0	1.5	2	1.0	3.00	19071	221.7	221.7	300.0	73.9	02/11/93	02/24/93
AAPSI	53	93-BB138	10.0	4.0	6.0	2	10.0	1.20	25657	257.5	257.5	300.0	85.8	02/25/93	03/09/93
AAPSI	53	93-BB137	10.0	4.0	6.0	2	10.0	1.20	23423	238.3	238.3	300.0	79.4	02/25/93	03/09/93
AAPSI	53	93-BB192	10.0	1.0	1.5	1	1.0	1.50	0.0000	0.0	0.0	0.0	0.0	03/10/93	03/10/93
AAPSI	53	93-BB193	15.0	1.0	1.0	1	1.0	1.00	31655	359.0	359.0	300.0	119.7	03/10/93	03/10/93
AAPSI	53	93-BB194	15.0	1.0	1.0	1	1.0	1.00	23393	268.0	268.0	300.0	89.3	03/10/93	03/10/93
AAPSI	53	93-BB212	10.0	1.0	1.5	1	1.0	1.50	0.0000	0.0	0.0	0.0	0.0	03/03/93	03/20/93
AAPSI	53	93-BB213	25.0	1.0	0.8	1	1.0	0.60	46612	323.3	323.3	300.0	107.8	03/03/93	03/20/93
AAPSI	53	93-BB214	25.0	1.0	0.8	1	1.0	0.60	41797	286.7	286.7	300.0	95.8	03/03/93	03/20/93
AAPSI	53	93-BB172	10.0	5.0	7.5	1	5.0	1.50	439	0.0	0.0	0.0	0.0	03/03/93	03/20/93
AAPSI	53	93-BB173	10.0	5.0	7.5	1	5.0	1.50	99999	294.0	294.0	300.0	98.0	03/03/93	03/20/93
AAPSI	53	93-BB174	10.0	5.0	7.5	1	5.0	1.50	96140	282.0	282.0	300.0	94.0	03/03/93	03/20/93
AAPSI	53	93-BB240	10.0	5.0	7.5	1	5.0	1.50	0.0000	0.0	0.0	0.0	0.0	03/29/93	03/31/93
AAPSI	53	93-BB241	25.0	5.0	3.0	1	5.0	0.60	36025	265.0	265.0	300.0	88.3	03/29/93	03/31/93
AAPSI	53	93-BB242	25.0	5.0	3.0	1	5.0	0.60	35564	260.0	260.0	300.0	86.7	03/29/93	03/31/93
AAPSI	53	93-BB260	10.0	5.0	7.5	1	5.0	1.50	205	0.0	0.0	0.0	0.0	04/07/93	04/12/93
AAPSI	53	93-BB261	10.0	2.0	3.0	1	5.0	0.60	37660	230.0	230.0	300.0	76.7	04/07/93	04/12/93
AAPSI	53	93-BB262	10.0	2.0	3.0	1	5.0	0.60	36341	218.3	218.3	300.0	72.8	04/07/93	04/12/93
AAPSI	53	93-BB304	10.0	2.0	3.0	1	5.0	0.60	0.0000	0.0	0.0	0.0	0.0	06/22/93	06/25/93
AAPSI	53	93-BB305	10.0	2.0	3.0	1	5.0	0.60	56258	306.7	306.7	300.0	102.2	06/22/93	06/25/93
AAPSI	53	93-BB308	10.0	2.0	3.0	1	5.0	0.60	59878	331.7	331.7	300.0	110.6	06/22/93	06/25/93

a) PPB Found-PPB Net = (mg Found - ng in control) x MW Factor x 1000/(mg injected)

b) Fortifications were added prior to extraction and were run concurrently with control samples.

c) Percent Recovery = (ppb Net/ppb Added) x 100

Infection volume was constant at 1 or 24. The sample size was 15.0 g. Values in this table may have been rounded off for reporting purposes, but not for any further calculations. The PPB Net values were corrected for apparent residues in the controls, although these residues were less than the limit of quantitation and were extrapolated.

15

TABLE IV. Individual Recoveries of BAS 490 F and BF 490-1 in Aquatic Media
(Continued).

Media No.	Sample	Lab Code	Vol. ml	Aliquot ml	Based on ml	Vol. ml	Vol. ml	Peak Ht	Found ng	Found at	Final	PPB b	Recon. % of added	Extrctn. Date	Injctn. Date
MAA 54	93-BB11R	10.0	1.0	1.5	2	1.0	3.00	0.0000	0.0	0.0	0.0	0.0	88.3	02/05/93	02/07/93
MAA 54	93-BB12R	10.0	4.0	6.0	2	10.0	1.20	59788	0.3180	265.0	265.0	300.0	88.3	02/05/93	02/07/93
MAA 54	93-BB13R	10.0	4.0	6.0	2	10.0	1.20	58085	0.3080	256.7	256.7	300.0	85.6	02/05/93	02/07/93
MAA 54	93-BB56	10.0	1.0	1.5	2	1.0	3.00	0.0000	0.0	0.0	0.0	0.0	70.0	02/04/93	02/06/93
MAA 54	93-BB59	10.0	1.0	1.5	2	1.0	3.00	118774	0.6300	210.0	210.0	300.0	89.2	02/04/93	02/06/93
MAA 54	93-BB80	10.0	1.0	1.5	2	1.0	3.00	145277	0.7760	258.7	258.7	300.0	89.2	02/04/93	02/06/93
MAA 54	93-BB112	10.0	1.0	1.5	2	1.0	3.00	0.0000	0.0	0.0	0.0	0.0	113.9	02/11/93	02/11/93
MAA 54	93-BB113	10.0	2.0	3.0	2	5.0	1.20	0.4100	0.0	341.7	341.7	300.0	99.7	02/11/93	02/11/93
MAA 54	93-BB114	10.0	2.0	3.0	2	5.0	1.20	0.3590	0.0	299.2	299.2	300.0	99.7	02/11/93	02/11/93
MAA 54	93-BB140	10.0	1.0	1.5	2	1.0	3.00	0.0000	0.0	0.0	0.0	0.0	78.4	02/25/93	03/09/93
MAA 54	93-BB141	10.0	4.0	6.0	2	10.0	1.20	23428	0.2860	238.3	238.3	300.0	83.1	02/25/93	03/09/93
MAA 54	93-BB142	10.0	4.0	6.0	2	10.0	1.20	24675	0.2990	249.2	249.2	300.0	83.1	02/25/93	03/09/93
MAA 54	93-BB197	10.0	1.0	1.5	1	1.0	1.50	0.0000	0.0	0.0	0.0	0.0	89.0	03/10/93	03/22/93
MAA 54	93-BB198	15.0	1.0	1.0	1	1.0	1.00	69458	0.2670	287.0	287.0	300.0	88.7	03/10/93	03/22/93
MAA 54	93-BB199	15.0	1.0	1.0	1	1.0	1.00	69429	0.2660	286.0	286.0	300.0	88.7	03/10/93	03/22/93
MAA 54	93-BB177	10.0	1.0	1.5	1	1.0	1.50	0.0000	0.0	0.0	0.0	0.0	99.6	03/03/93	03/20/93
MAA 54	93-BB178	10.0	1.0	1.5	1	1.0	1.50	101506	0.4480	298.7	298.7	300.0	100.4	03/17/93	03/19/93
MAA 54	93-BB179	10.0	1.0	1.5	1	1.0	1.50	102575	0.4520	301.3	301.3	300.0	98.7	03/17/93	03/19/93
MAA 54	93-BB217	10.0	1.0	1.5	1	1.0	1.50	0.0000	0.0	0.0	0.0	0.0	91.1	03/17/93	03/19/93
MAA 54	93-BB218	25.0	1.0	0.6	1	1.0	0.60	46016	0.1740	290.0	290.0	300.0	98.7	03/17/93	03/19/93
MAA 54	93-BB219	25.0	1.0	0.6	1	1.0	0.60	43839	0.1640	273.3	273.3	300.0	91.1	03/17/93	03/19/93
MAA 54	93-BB245	10.0	5.0	7.5	1	5.0	1.50	358	0.0000	0.0	0.0	0.0	101.1	03/29/93	03/31/93
MAA 54	93-BB248	25.0	5.0	3.0	1	5.0	0.60	40765	0.1820	303.3	303.3	300.0	95.0	03/29/93	03/31/93
MAA 54	93-BB247	25.0	5.0	3.0	1	5.0	0.60	38543	0.1710	285.0	285.0	300.0	106.7	04/07/93	04/12/93
MAA 54	93-BB265	10.0	5.0	7.5	1	5.0	1.50	1128	0.0000	0.0	0.0	0.0	105.0	04/07/93	04/12/93
MAA 54	93-BB286	10.0	2.0	3.0	1	5.0	0.60	49737	0.1920	320.0	320.0	300.0	106.7	04/07/93	04/12/93
MAA 54	93-BB287	10.0	2.0	3.0	1	5.0	0.60	49102	0.1890	315.0	315.0	300.0	105.0	04/07/93	04/12/93
MAA 54	93-BB309	10.0	2.0	3.0	1	5.0	0.60	550	0.0000	0.0	0.0	0.0	114.4	06/22/93	06/25/93
MAA 54	93-BB310	10.0	2.0	3.0	1	5.0	0.60	61533	0.2060	343.3	343.3	300.0	113.9	06/22/93	06/25/93
MAA 54	93-BB311	10.0	2.0	3.0	1	5.0	0.60	61215	0.2050	341.7	341.7	300.0	113.9	06/22/93	06/25/93

Sample Weight (g): 15.0
a) PPB Found-PPB Net = (ng Found - ng in control) x MW Factor x 1000/(mg injected)
b) Fortifications were added prior to extraction and were run concurrently with control samples.
c) Percent Recovery = (gpb Net/gpb Added) x 100
Injection volume was constant at 1 or 2 µl. The sample size was 15.0 g. Values in this table may have been rounded off for reporting purposes, but not for any further calculations.
The PPB Net values were corrected for apparent residues in the controls, although these residues were less than the limit of quantitation and were extrapolated.

TABLE IV. Individual Recoveries of BAS 490 F and BF 490-1 in Aquatic Media (Continued).

Sample	Lab. Code	Vol. (ml)	Basal concn (mg/ml)	Aliquot (g)	Peak Ht	Found (ng)	PPB Found	PPB Added	Recovery (%)	Extraction Date
20X-AAP	55 93-BB16R	10.0	1.0	1.5	2	0.0000	0.0	0.0	0.0	02/05/93 02/07/93
20X-AAP	55 93-BB17R	10.0	4.0	6.0	2	0.0000	273.3	300.0	91.1	02/05/93 02/07/93
20X-AAP	55 93-BB18R	10.0	4.0	6.0	2	0.0000	271.7	300.0	90.6	02/05/93 02/07/93
20X-AAP	55 93-BB63	10.0	1.0	1.5	2	0.0000	0.0	0.0	0.0	02/04/93 02/08/93
20X-AAP	55 93-BB64	10.0	1.0	1.5	2	0.0000	0.0	0.0	0.0	02/04/93 02/08/93
20X-AAP	55 93-BB65	10.0	1.0	1.5	2	0.0000	270.0	300.0	90.0	02/04/93 02/08/93
20X-AAP	55 93-BB117	10.0	1.0	1.5	2	0.0000	267.3	300.0	89.1	02/04/93 02/08/93
20X-AAP	55 93-BB118	10.0	2.0	3.0	2	0.0000	0.0	0.0	0.0	02/11/93 02/11/93
20X-AAP	55 93-BB119	10.0	2.0	3.0	2	0.0000	285.8	300.0	95.3	02/11/93 02/11/93
20X-AAP	55 93-BB145	10.0	1.0	1.5	2	0.0000	288.7	300.0	95.6	02/11/93 02/11/93
20X-AAP	55 93-BB146	10.0	4.0	6.0	2	0.0000	270.8	300.0	90.3	02/25/93 03/09/93
20X-AAP	55 93-BB147	10.0	4.0	6.0	2	0.0000	263.3	300.0	87.8	02/25/93 03/09/93
20X-AAP	55 93-BB202	10.0	1.0	1.5	2	0.0000	0.0	0.0	0.0	03/10/93 03/22/93
20X-AAP	55 93-BB203	15.0	1.0	1.0	1	0.0000	306.0	300.0	102.0	03/10/93 03/22/93
20X-AAP	55 93-BB204	15.0	1.0	1.0	1	0.0000	315.0	300.0	105.0	03/10/93 03/22/93
20X-AAP	55 93-BB182	10.0	5.0	7.5	1	0.0000	0.0	0.0	0.0	03/03/93 03/20/93
20X-AAP	55 93-BB183	10.0	5.0	7.5	1	0.0000	312.7	300.0	104.2	03/03/93 03/20/93
20X-AAP	55 93-BB184	10.0	5.0	7.5	1	0.0000	303.3	300.0	101.1	03/03/93 03/20/93
20X-AAP	55 93-BB222	10.0	1.0	1.5	1	0.0000	0.0	0.0	0.0	03/17/93 03/19/93
20X-AAP	55 93-BB223	25.0	1.0	0.6	1	0.0000	295.0	300.0	98.3	03/17/93 03/19/93
20X-AAP	55 93-BB224	25.0	1.0	0.6	1	0.0000	296.7	300.0	98.9	03/17/93 03/19/93
20X-AAP	55 93-BB250	10.0	5.0	7.5	1	0.0000	0.0	0.0	0.0	03/29/93 03/31/93
20X-AAP	55 93-BB251	25.0	5.0	3.0	1	0.0000	310.0	300.0	103.3	03/29/93 03/31/93
20X-AAP	55 93-BB252	25.0	5.0	3.0	1	0.0000	308.7	300.0	102.2	03/29/93 03/31/93
20X-AAP	55 93-BB270	10.0	5.0	7.5	1	0.0000	0.0	0.0	0.0	04/07/93 04/12/93
20X-AAP	55 93-BB271	10.0	2.0	3.0	1	0.0000	361.7	300.0	120.8	04/07/93 04/12/93
20X-AAP	55 93-BB272	10.0	2.0	3.0	1	0.0000	335.0	300.0	111.7	04/07/93 04/12/93
20X-AAP	55 93-BB314	10.0	2.0	3.0	1	0.0000	0.0	0.0	0.0	06/25/93 06/25/93
20X-AAP	55 93-BB315	10.0	2.0	3.0	1	0.0000	373.3	300.0	124.4	06/25/93 06/25/93
20X-AAP	55 93-BB316	10.0	2.0	3.0	1	0.0000	375.0	300.0	125.0	06/25/93 06/25/93

Sample Weight (g) = 15.0
 a) PPB Found/PPB Net = ((ng Found - ng in control) x MW Factor x 1000)/(mg injected)
 b) Fortifications were added prior to extraction and were run concurrently with control samples.
 c) Percent Recovery = (PPB Net/PPB Added) x 100
 d) Injection volume was constant at 1 or 2 µl. The sample size was 15.0 g. Values in this table may have been rounded off for reporting purposes, but not for any further calculations.
 The PPB Net values were corrected for apparent residues in the controls, although these residues were less than the limit of quantitation and were extrapolated.

Figure 1. FLOW CHART OF ANALYTICAL PROCEDURE

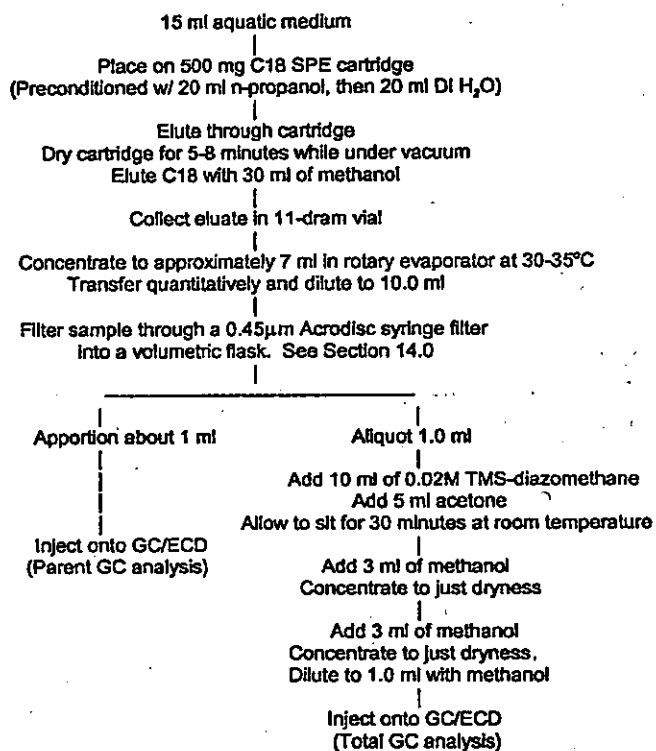
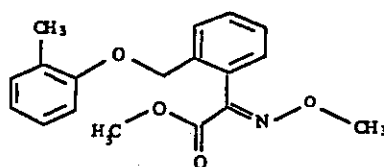
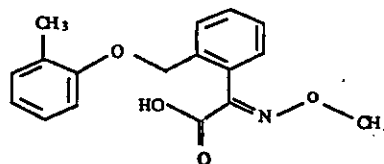


Figure 2. Structures Of The Test Substance And The Final Analytes



BAS 490 F



BF 490-1

Figure 3. Typical Standard Curves

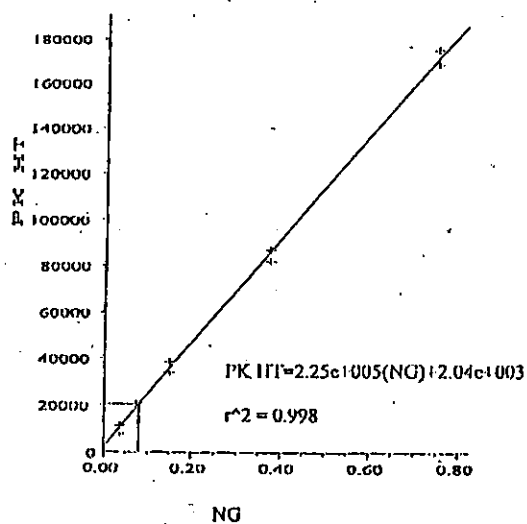
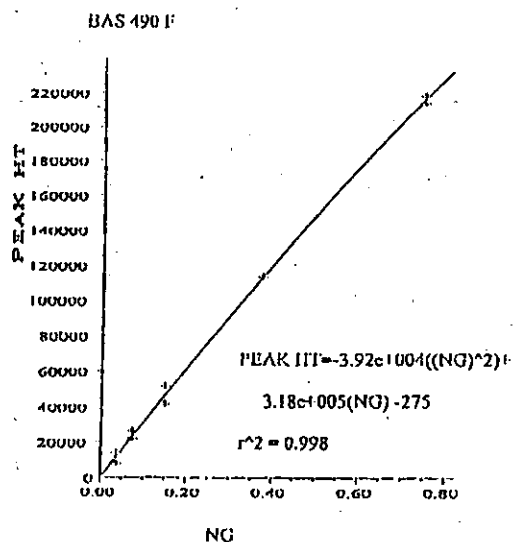
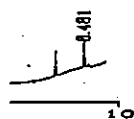
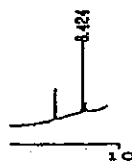


Figure 4. Typical Standard Chromatograms for the Determination of BAS 490 F and BF 490-1 in Aquatic Media.



0.0375 ng Std
BAS 490 F
Pk. Ht.: 7906
Rt.: 8.3 min.



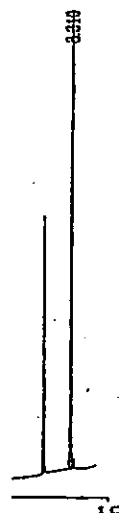
0.075 ng Std
BAS 490 F
Pk. Ht.: 21912



0.150 ng Std
BAS 490 F
Pk. Ht.: 41706



0.375 ng Std
BAS 490 F
Pk. Ht.: 112506



0.750 ng Std
BAS 490 F
Pk. Ht.: 213718

Figure 5. Typical Chromatograms for the Determination of BAS 490 F and BF 490-1 in Aquatic Media.

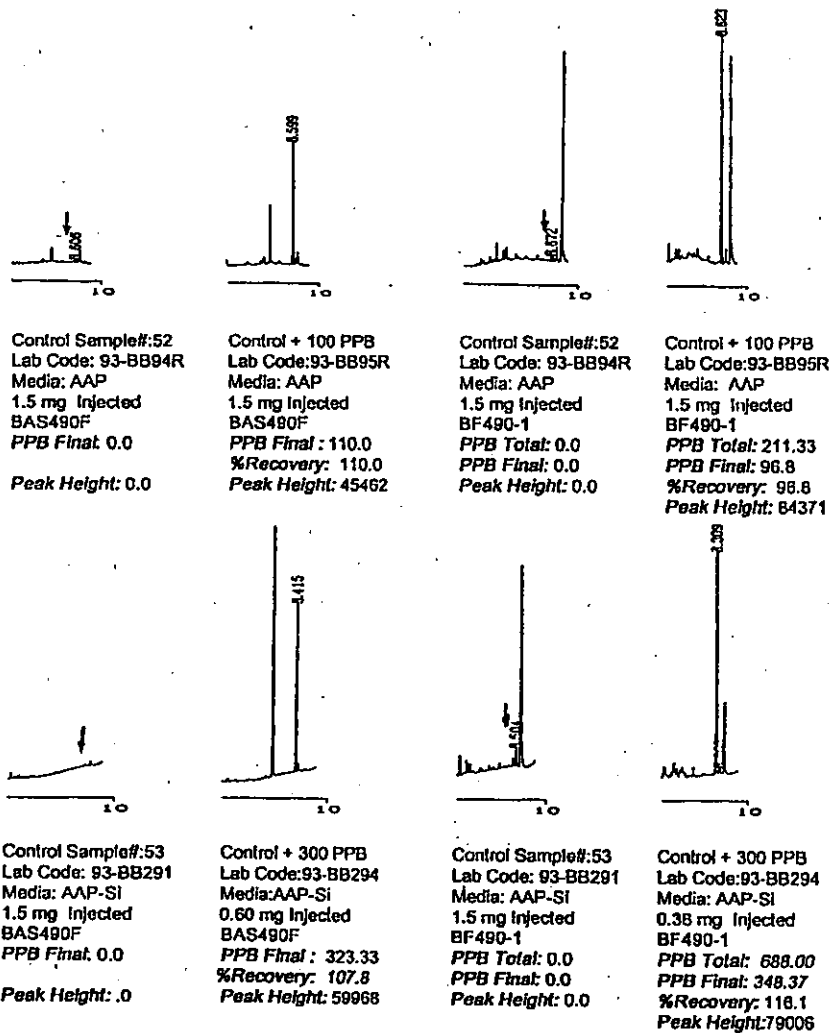


Figure 5. Typical Chromatograms for the Determination of BAS 490 F and BF 490-1 in Aquatic Media (continued).

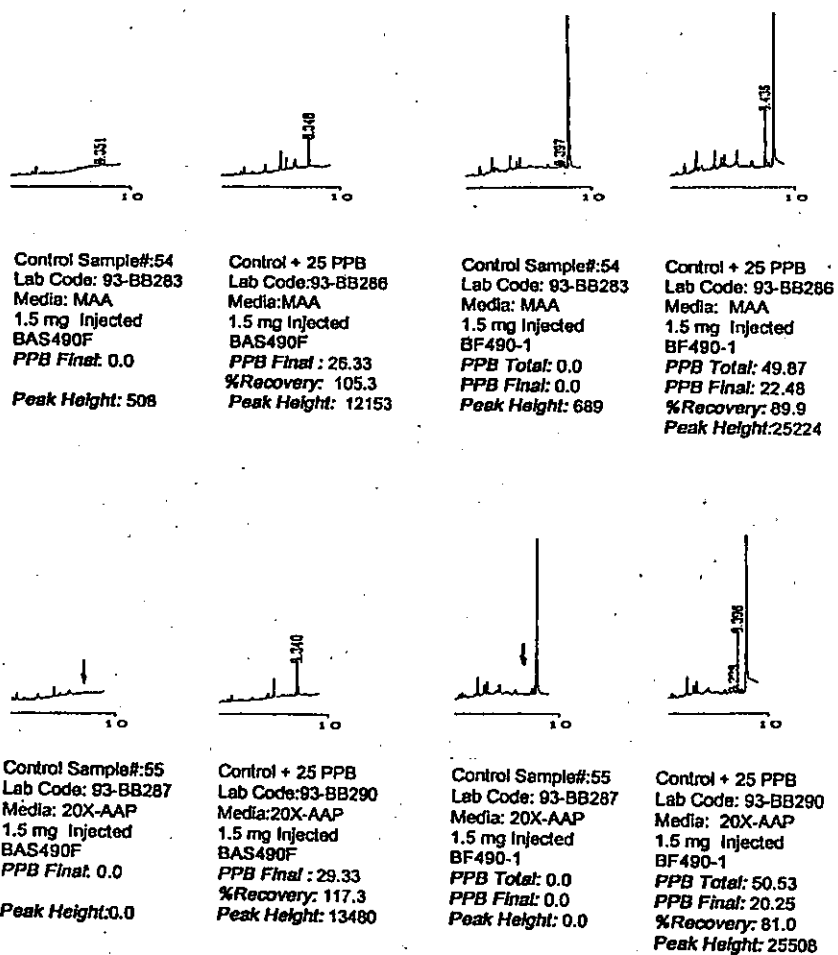
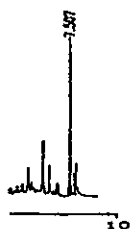


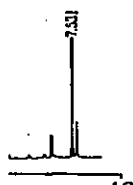
Figure 5. Typical Chromatograms for the Determination of BAS 490 F and BF 490-1 in Aquatic Media (continued).



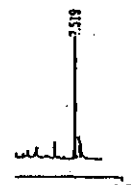
Sample#: 11
Lab Code: 93-BB26
Media: AAP
3.0 mg Injected
BAS490F
PPB Final: 296.33
Peak Height: 51785



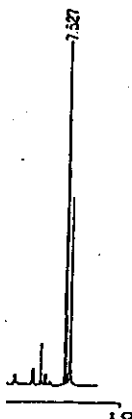
Sample#: 11
Lab Code: 93-BB26
Media: AAP
3.0 mg Injected
BF490F-1
PPB Total: 348.33
PPB Final: 49.68
Peak Height: 55875



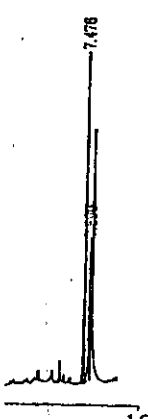
Sample#: 26
Lab Code: 93-BB29
Media: AAP
3.0 mg Injected
BAS490F
PPB Final: 243.0
Peak Height: 42906



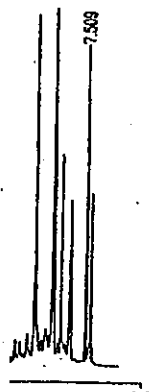
Sample#: 26
Lab Code: 93-BB29
Media: AAP
3.0 mg Injected
BF490-1
PPB Total: 260.67
PPB Final: <25.0
Peak Height: 43667



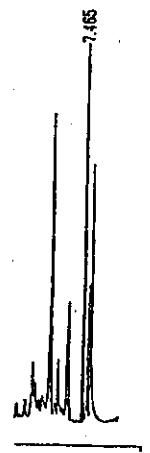
Sample#: 13
Lab Code: 93-BB44
Media: AAPSI
3.0 mg Injected
BAS490F
PPB Final: 274.67
Peak Height: 138413



Sample#: 13
Lab Code: 93-BB44
Media: AAPSI
3.0 mg Injected
BF490F-1
PPB Total: 255.00
PPB Final: <25.0
Peak Height: 134652



Sample#: 28
Lab Code: 93-BB47
Media: AAPSI
3.2 mg Injected
BAS490F
PPB Final: 236.25
Peak Height: 127577



Sample#: 28
Lab Code: 93-BB47
Media: AAPSI
3.2 mg Injected
BF490-1
PPB Total: 271.88
PPB Final: 34.03
Peak Height: 152731

Figure 5. Typical Chromatograms for the Determination of BAS 490 F and BF 490-1 In Aquatic Media (continued).

