Cover Sheet for

Environmental Chemistry Method

Pesticide Name: **Cyazofamid**

MRID#: **454090-21**

Matrix: **Water**

Analysis: **HPLC/UV**

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

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

STUDY TITLE

ANALYTICAL PROCEDURE FOR THE DETERMINATION OF IKF-916 RESIDUES IN WATER

NOTE: CYAZOFAMID (CAS #120116-88-3) is 4chloro-2-cyano-*N*,*N*-dimethyl-5-(4-methylphenyl)- 1*H*-imidazole-1-sulfonamide. Other identifier is IKF-916.

DOCUMENT NUMBER

011140-1

DATA REQUIREMENT

EPA Pesticide Guideline
860.1340 (171-4)

AUTHOR

N. P. Jordan
R. G. Kenyon

DATE

December 1, 1999

PERFORMING LABORATORY
Department of Residue Analysis
Ricerca, Inc.
7528 Auburn Road, P.O. Box 1000
Painesville, OH 44077-1000

STUDY SPONSOR
ISHIHARA SANGYO KAISHA, LTD.
3-15, Edobori 1-Chome, Nishi-ku
Osaka, Shiga 550-0002, Japan

SUBMITTED BY
ISK BIOSCIENCES CORPORATION
7470 Auburn Road, Suite A
Concord, Ohio 44077

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS[1]

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

This statement supersedes all other markings of confidentiality.

COMPANY: ISHIHARA SANGYO KAISHA, LTD.

COMPANY AGENT: Jerry R. Lucietta

Typed Name

Agent for ISHIHARA SANGYO KAISHA, LTD.
Manager, Regulatory Affairs
ISK Biosciences Corporation

Title

Jerry R. Lucietta

Signature

March 30, 2001

Date

[1]We submitted this material to the United States Environmental Protection Agency specifically under provisions contained in FIFRA as amended, and thereby consent to use and disclosure of this material by EPA according to FIFRA. Notwithstanding the wording of our marking (either as "Property of ISK BIOSCIENCES CORPORATION" or "Property of ISHIHARA SANGYO KAISHA, LTD."), this marking by itself conveys no supplemental claims of confidentiality under FIFRA Sections 10(c) or 10(b). In submitting this material to the EPA according to the method and format requirements contained in PR Notice 86-5, we do not waive any protection or right involving this material that would have been claimed by ISK BIOSCIENCES CORPORATION or by ISHIHARA SANGYO KAISHA, LTD. or agents of these companies anywhere else in the world, if this material had not been submitted to the EPA.

COMPLIANCE STATEMENT

ISK Biosciences Corporation

This study was conducted and reported in compliance with Good Laboratory Practice Regulations set forth in Title 40, Part 160 of the Code of Federal Regulations of the United States of America.

Michael A. Peplowski — March 30, 2001

Michael A. Peplowski
Manager, Product Registration
ISK BIOSCIENCES CORPORATION, APPLICANT

Date

REFERENCE TO COMPLIANCE STATEMENT WITHIN THE REPORT

A GOOD LABORATORY PRACTICES compliance statement

with the signatures of

the STUDY DIRECTOR and the STUDY SPONSOR

is found on page 3 of the report.

Ricerca, Inc.

REPORT

Study Title:

Analytical Procedure for the Determination of IKF-916 Residues in Water

Document Number: 011140-1
Ricerca Project Identification Number: 011140

Data Requirement:

OPPTS Series 860.1340

Study Completed:

December 1, 1999

Authors:

N. P. Jordan
R. G. Kenyon

Performing Laboratory:
Department of Residue Analysis
Ricerca, Inc.
7528 Auburn Road
P. O. Box 1000
Painesville OH 44077-1000

Study Sponsor:
Ishihara Sangyo Kaisha, Ltd.
3-15, Edobori 1-chome, Nishi-ku
Osaka, 550-0002
JAPAN

STATEMENT OF NO DATA CONFIDENTIALITY CLAIM

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

Company: Ishihara Sangyo Kaisha, Ltd.

Company Agent: S. Ogyu

Title: General Manager

Signature **Date**

These data are the property of Ishihara Sangyo Kaisha, Ltd. and, as such, are considered confidential for all purposes other than compliance with FIFRA Section 10. Submission of these data in compliance with FIFRA does not constitute a waiver of any right to confidentiality that may exist under any other statute or in any other country.

COMPLIANCE STATEMENT

The study reported herein, "Analytical Procedure for the Determination of IKF-916 Residues in Water", Ricerca Document Number 011140-1, was conducted and reported in compliance with the Good Laboratory Practice Regulations set forth in Title 40, Part 160 of the Code of Federal Regulations of the United States of America

J.E. Jablonski

12-1-99

J. E. Jablonski, Ph.D.
Study Director
Department of Residue Analysis
Ricerca, Inc.

Date

S. Ogyu

December 7, 1999

S. Ogyu
Sponsor Representative
Ishihara Sangyo Kaisha, Ltd.

Date

FLAGGING STATEMENT

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APPROVALS

Study Title:	Analytical Procedure for the Determination of IKF-916 Residues in Water
Study Number:	011140
Document Number:	011140-1
Performing Laboratory:	Department of Residue Analysis Ricerca, Inc. 7528 Auburn Road P. O. Box 1000 Painesville OH 44077-1000

N. P. Jordan — 11-30-99
N. P. Jordan, Research Chemist I — **Date**
Department of Residue Analysis

R. G. Kenyon — 11/30/99
R. G. Kenyon, Research Chemist I — **Date**
Department of Residue Analysis

J. E. Jablonski — 12.1.99
J. E. Jablonski, Ph.D., Study Director — **Date**
Department of Residue Analysis

Jerome L. Wiedmann — 11-30-99
J. L. Wiedmann, Ph.D., Section Head — **Date**
Department of Residue Analysis

D L Ballee — 11/30/99
D. L. Ballee, Director — **Date**
Department of Residue Analysis

R A Baxter — 12-1-99
R. A. Baxter, Vice President — **Date**
Ricerca, Inc.

QUALITY ASSURANCE STATEMENT

The Ricerca Quality Assurance Unit has performed inspections on the study, "Analytical Procedure for the Determination of IKF-916 Residues in Water", Ricerca Document Number 011140-1, and reported findings to the Study Director and to Management on the dates listed below:

Dates of Inspection	Dates Reported to Study Director	Dates Reported to Management
11/9/99	11/16/99	11/17/99
11/17/99	11/18/99	11/29/99

Margaret Hardy
Quality Assurance Auditor

11/30/99
Date

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ABSTRACT

An analytical method for the determination of IKF-916 residues in water was developed. The method used reversed phase HPLC with absorbance detection at 280 nm to quantitate IKF-916 in sample extracts. Sample preparation consisted of typical residue procedures including filtration, extraction, and concentration.

Each water type (tap water, pond water) was analyzed in sets of 12 consisting of duplicate controls, five 0.1 ppb fortifications and five 1 ppb fortifications.

Mean recoveries with relative standard deviations (RSD) and ranges for the two water types at the two fortification levels are shown below.

Water Type	**Mean Recovery , RSD (%)**	**Range**
Tap Water	Mean = 105%, RSD = 7.0%	95 - 118
Pond Water	Mean =101%, RSD = 8.4%	88 - 116

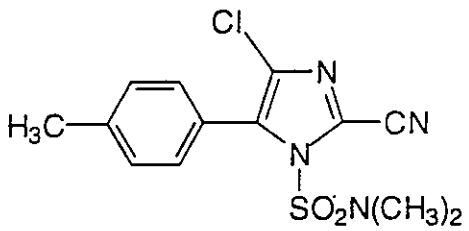
These results support the proposed IKF-916 limit of quantitation (LOQ) of 0.1 ppb. The mean recoveries and relative standard deviations are in the acceptable ranges and indicate the method is precise and accurate. Calibration curves generated from external standards in the range of 0.025 to 1.000 µg/mL showed good linearity with coefficients of determination routinely higher than 0.999. No control samples were found to have background interferences at or near the retention time of IKF-916 with responses equal to or greater than the 0.025 µg/mL calibration standard (equivalent to the limit of detection).

INTRODUCTION

A residue method for the determination of IKF-916 in drinking and surface water was developed and evaluated in this study. Method performance was discussed in terms of EU method parameters including accuracy, precision, limit of quantitation, specificity and linearity.

TEST/REFERENCE SUBSTANCE

The structure, CAS registry number, and chemical name for IKF-916 are listed below.



Chemical names: 4-chloro-2-cyano-*N*,*N*-dimethyl-5-*p*-tolylimidazole-1-sulfonamide (IUPAC-J and IUPAC)

4-chloro-2-cyano-*N*,*N*-dimethyl-5-(4-methylphenyl)-1*H*-imidazole-1-sulfonamide (CA-J and CA)

CAS No.: 120116-88-3
Lot Number: 9704-1
Purity: 99.1% (Date of Certificate: July 15, 1997)

OBJECTIVE

The objective of this study was to develop and evaluate an analytical method for the determination of IKF-916 residues in drinking and surface water. Specific criteria under evaluation were method accuracy, precision (repeatability), limit of quantitation, specificity and linearity. The proposed method limit of quantitation was 0.1 ppb, and was to be achieved by demonstrating the successful recovery of IKF-916 from 0.1-ppb fortifications.

CONDUCT OF STUDY

SPONSOR

Ishihara Sangyo Kaisha, Ltd.
3-15, Edobori 1-chome, Nishi-ku
Osaka, 550-0002
JAPAN

STUDY DIRECTOR

Joseph E. Jablonski
Department of Residue Analysis
Ricerca, Inc.
7528 Auburn Road
P.O. Box 1000
Painesville OH 44077-1000
Business Phone: (440) 357-3761

SCHEDULE OF EVENTS

Study Initiation Date:	October 1, 1999
Experimental Start Date:	November 8, 1999
Experimental Termination Date:	November 10, 1999
Study Completion:	December 1, 1999

RETENTION OF DATA

Raw data supporting this study and the final report are stored in the Ricerca, Inc. Archives at the above address.

This study was conducted by Ricerca, Inc., 7528 Auburn Road, Painesville, Ohio 44077, Department of Residue Analysis, in accordance with protocol 011140-0 (Appendix A).

Personnel involved in the study were:

R. C. Beckwith, Research Chemist II
W. J. Debevc, Associate Chemist
K. L. Furlong, Associate Chemist
N. P. Jordan, Research Chemist I
R. G. Kenyon, Research Chemist I

J. E. Jablonski, Senior Scientist
J. L. Wiedmann, Section Head

MATERIALS AND METHODS

SAMPLE PROCUREMENT AND RECEIPT

Control pond water was obtained from a pond located in Leroy Township, OH. Control tap water was obtained from a laboratory at the Ricerca, Inc. site. The pond water was maintained in a cooler with ice prior to analysis.

The characteristics of the pond water sample are:

pH: 7.03
total hardness: 79.3 mg/L
DOC: 13 mg/L

SAMPLE IDENTIFICATION

At the time of receipt at Ricerca (pond water) or at the time of use (tap water), control samples were assigned a Ricerca laboratory code number consisting of the year and a consecutive number. Sample identification numbers are listed below.

Sample Matrix	Control Sample Identification Number
Pond Water	99-0496
Tap Water	99-0497

At the time of analysis, each aliquot of water was assigned a unique sample ID.

The sample ID and corresponding sample description were recorded on Operations Form and Sample List (flowsheet).

Appendix D contains sample history information (dates of extraction and analysis).

ANALYTICAL PROCEDURE SUMMARY

A detailed analytical method is provided in Appendix B. A summary of the method is provided here.

An aliquot of the water sample was acidified to approximately pH 3. For pond water samples, the sample was filtered through a bed of Celite. The sample was pulled onto a C_{18} solid phase extraction column using vacuum. The column was eluted with acetonitrile followed by water. The acetonitrile/water was evaporated to 0.2 – 0.3 mL, and the sample residue was diluted with 30/70 acetonitrile/water for HPLC quantitation. All samples were quantified by reversed-phase HPLC with UV absorbance detection at 280nm. Figure 1 contains a method flow diagram. Preparation of a set of 12 samples for HPLC analysis required approximately 8 hours.

CALCULATION OF RESIDUES

IKF-916 residues were quantified using linear multi-point calibration curves generated from the injection of IKF-916 external standards. Detailed sample calculations are found in Appendix C.

RESULTS AND DISCUSSION

A residue method for the determination of IKF-916 in water was developed, and the analytical results in relation to the EU method reporting requirements are discussed below. A set consisting of 2 controls, five samples fortified at the LOQ (0.1 ppb) and five samples fortified at 10 times LOQ (1 ppb) of duplicate controls were run for each water type.

ACCURACY

Accuracy is defined as the nearness of the measured residue value to the true value. The overall IKF-916 mean recovery calculated from all fortifications is an indication of method accuracy. The table below summarizes recovery data for each water type.

Water Type	Fortification Levels (ppb)	Mean Recovery (%)
Tap Water	0.1 , 1.00	105
Pond Water	0.1 , 1.00	101

The mean recoveries were calculated from 5 values at 0.1 ppb and 5 values at 1 ppb for each water type. Recoveries of IKF-916 from individual fortified samples are shown in Tables 1 and 2, for tap water and pond water, respectively. Overall mean recoveries of IKF-916 were 105% (tap water) and 101% (pond water). This indicates a high degree of accuracy for the method.

PRECISION AND REPEATABILITY

The short-term precision afforded by an analytical method is given by the variability obtained by a single analyst working in one laboratory with the same equipment on a set of replicate fortified samples. The relative standard deviation (RSD) calculated for a set of recoveries under these conditions is a measurement of repeatability. The range of recovery values is also an indicator of method precision. The table below lists the RSD of the mean recovery for each matrix, and the range of recoveries. Values are taken from Tables 1and 2 which show individual sample recoveries for tap water and pond water, respectively.

Water Type	RSD of Mean Recovery (%)	Range
Tap Water	7.0	95 - 118
Pond Water	8.4	88 - 116

RSD values were less than 20% for each water type, which indicates good precision.

LIMIT OF QUANTITATION

The target method limit of quantitation (LOQ) or limit of determination (LDM) was 0.1 ppb. A practical assessment of method LOQ is given by measuring the accuracy and precision of recoveries obtained from replicate fortifications at the proposed LOQ. The mean recovery and RSD of five 0.1 ppb tap water fortifications was 106% and 8.6%, respectively. For pond water, the mean recovery and RSD of five 0.1 ppb fortifications was 105% and 8.1%, respectively. These results support an LOQ of 0.1 ppb for IKF-916 in water.

SPECIFICITY

Specificity for IKF-916 in the sample extracts was good. No control samples were found to have background interferences at or near the retention time of

IKF-916 with responses equal to or greater than the 0.025 µg/mL calibration standard. The response of the 0.025 µg/mL calibration standard was used as the limit of detection. Representative chromatograms from each water type are shown in Appendix C.

LINEARITY

Linearity of calibration curves was very good in the concentration range 0.025 to 1.00 µg/mL IKF-916. The coefficient of determination (R^2) for all chromatographic runs was 0.999 or better. A first order linear fit with 1/X weighting was used for every calibration curve. See Figure C-1 for an example of typical detector linearity for IKF-916.

CONCLUSIONS

The data shown in this report demonstrate that the analytical method for the determination of IKF-916 in water provides a specific, accurate and precise procedure. A method limit of quantitation of 0.1 ppb was demonstrated for IKF-916.

11/30/99/rgk

Figure 1: Flow Diagram for IKF-916 Water Method

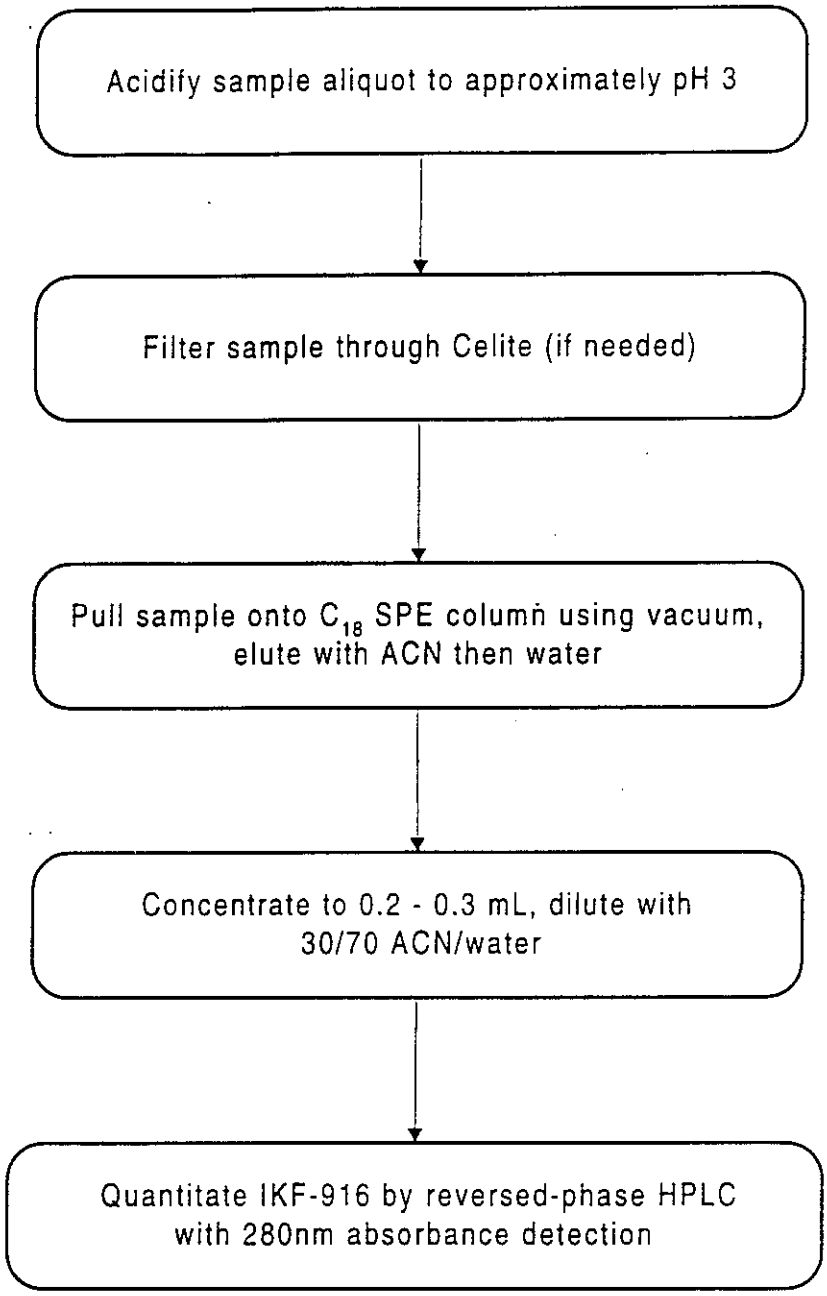


Table 1: Recovery of IKF-916 from Fortified Tap Water

Sample ID	IKF-916 Fortification (ppb)	Net Recovered (ppb)	Percent Recovery
TWC1	Control	< 0.025	---
TWC2	Control	< 0.025	---
TWLOQ1	0.100	0.103	103
TWLOQ2	0.100	0.118	118
TWLOQ3	0.100	0.112	112
TWLOQ4	0.100	0.101	101
TWLOQ5	0.100	0.095	95
TW10XLOQ1	1.00	1.007	101
TW10XLOQ2	1.00	1.029	103
TW10XLOQ3	1.00	1.015	102
TW10XLOQ4	1.00	1.136	114
TW10XLOQ5	1.00	0.992	99
		Mean	**105**
		SD	**7.3**
		RSD	**7.0**

Limit of detection = 0.025 ppb
Limit of quantitation = 0.1 ppb
SD = Standard Deviation
RSD = Relative Standard Deviation = Standard Deviation/Mean Recovery x 100

Table 2: Recovery of IKF-916 from Fortified Pond Water

Sample ID	IKF-916 Fortification (ppb)	Net Recovered (ppb)	Percent Recovery
PWC1	Control	< 0.025	---
PWC2	Control	< 0.025	---
PWLOQ1	0.100	0.101	101
PWLOQ2	0.100	0.105	105
PWLOQ3	0.100	0.093	93
PWLOQ4	0.100	0.116	116
PWLOQ5	0.100	0.108	108
PW10XLOQ1	1.00	1.100	110
PW10XLOQ2	1.00	0.977	98
PW10XLOQ3	1.00	0.978	98
PW10XLOQ4	1.00	0.881	88
PW10XLOQ5	1.00	0.947	95
		Mean	**101**
		SD	**8.5**
		RSD	**8.4**

Limit of detection = 0.025 ppb
Limit of quantitation = 0.1 ppb
SD = Standard Deviation
RSD = Relative Standard Deviation = Standard Deviation/Mean Recovery × 100

APPENDIX A

Protocol and Amendments

Ricerca, Inc.

PROTOCOL

Study Title:

Analytical Procedure for the Determination of Residues of IKF-916 and Its Metabolites (CCIM, CCIM-AM, CTCA and CCBA) in Water

Document Number: 011140-0

Performing Laboratory:
Department of Residue Analysis
Ricerca, Inc.
7528 Auburn Road
P. O. Box 1000
Painesville OH 44077-1000

Study Sponsor:
Ishihara Sangyo Kaisha, Ltd.
3-15, Edobori, 1 chome, Nishi-ku
Osaka, 550-0002
JAPAN

Protocol:

Analytical Procedure for the Determination of Residues of IKF-916 and Its Metabolites (CCIM, CCIM-AM, CTCA and CCBA) in Water

Document Number: 011140-0

DISTRIBUTION

Originals:

Ishihara Sangyo Kaisha, Ltd./S. Ogyu
Ricerca, Inc. Archivist

Copies:

Study Director/J.E. Jablonski (1-1.1)
Residue Analysis Files (2)
Quality Assurance Files (3)

PROTOCOL APPROVAL

Study Title: Analytical Procedure for the Determination of Residues of IKF-916 and Its Metabolites (CCIM, CCIM-AM, CTCA and CCBA) in Water

Document Number: 011140-0

Performing Laboratory: Department of Residue Analysis
Ricerca, Inc.
7528 Auburn Road
P. O. Box 1000
Painesville OH 44077-1000

J.E. Jablonski, Study Director — Date: 10.1.99
Department of Residue Analysis

J.L. Wiedmann, Section Head — Date: 9-30-99
Department of Residue Analysis

D. L. Bailee, Director — Date: 10/1/99
Department of Residue Analysis

R. A. Baxter, Vice President — Date: 10/1/99
Ricerca, Inc.

S. Ogyu, General Manager — Date: 10/5/99
Safety Science Research Laboratory
Sponsor Representative

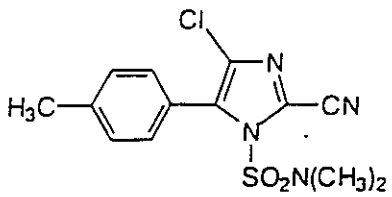
TABLE OF CONTENTS

INTRODUCTION

This protocol covers the development of an analytical procedure for the analysis of IKF-916 and its metabolites (CCIM, CCIM-AM, CTCA and CCBA) in drinking (tap or ground) water and surface (river or pond) water. The development of a method for water will be necessary for European Registration, and provides justification for the test system.

TEST AND REFERENCE SUBSTANCES

- IKF-916



Chemical names:

(IUPAC-J and IUPAC)

4-chloro-2-cyano-*N*,*N*-dimethyl-5-*p*-tolylimidazole-1-sulfonamide

(CA-J and CA)

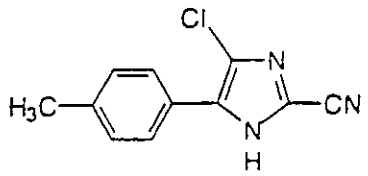
4-chloro-2-cyano-*N*,*N*-dimethyl-5-(4-methylphenyl)-1*H*-imidazole-1-sulfonamide

CAS No.: 120116-88-3

Lot Number: 9704-1

Purity: 99.1%

- CCIM



Chemical names:

4-chloro-5-*p*-tolylimidazole-2-carbonitrile (IUPAC and IUPAC-J)

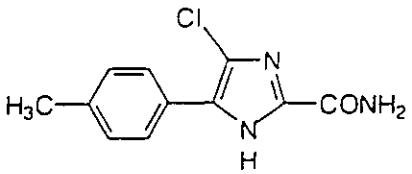
4-chloro-5-(4-methylphenyl)-1*H*-imidazole-2-carbonitrile (CA and CA-J)

CAS No.: 120118-14-1

Lot Number: 9506

Purity: 99.7%

- CCIM-AM



Chemical names:

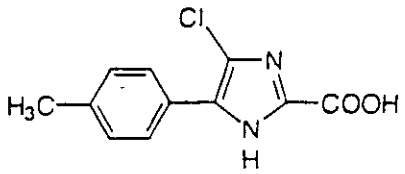
4-chloro-5-*p*-tolylimidazole-2-carboxamide (IUPAC and IUPAC-J)

4-chloro-5-(4-methylphenyl)-1*H*-imidazole-2-carboxamide (CA and CA-J)

Lot Number: 9804

Purity: 98.7%

- CTCA



Chemical names:

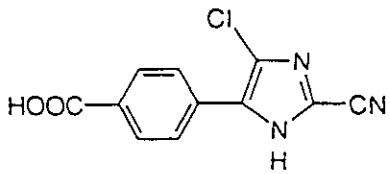
4-chloro-5-*p*-tolylimidazole-2-carboxylic acid (IUPAC-J)

4-chloro-5-(4-methylphenyl)-1*H*-imidazole-2-carboxylic acid (CA-J)

Lot Number: 9804

Purity: 99.3%

- CCBA



Chemical names:

4-(4-chloro-2-cyanoimidazol-5-yl)benzoic acid (IUPAC-J)

4-(4-chloro-2-cyano-1*H*-imidazol-5-yl)benzoic acid (CA-J)

Lot Number: 9907

Purity: 98.1%

The stability, characterization, retention and disposal of the test and reference substances are the responsibility of the Sponsor.

OBJECTIVE

The objective of this study is to develop a method for the determination of IKF-916 and its metabolites (CCIM, CCIM-AM, CTCA and CCBA) in drinking (tap or ground) water and surface (river or pond) water with a target limit of quantitation (LOQ) of 0.1 ppb.

IDENTIFICATION OF THE TEST FACILITY

The study will be conducted at Ricerca, Inc., Department of Residue Analysis, 7528 Auburn Road, Painesville, Ohio 44077.

PROPOSED STARTING AND COMPLETION DATES

It is proposed that this study be conducted from September 1999 through November 1999. The actual starting and completion dates will be included in the final report.

VALIDATION OF THE ANALYTICAL PROCEDURE

Control samples of drinking (tap or ground) water and surface (river or pond) water will be amended by the addition of standard solutions of IKF-916 and its metabolites prior to the extraction step. Fortifications will be within the approximate range of 0.1 to 10 ppb. For each water type, a sample set will consist of a minimum of 5 samples fortified at the LOQ, 5 samples fortified at 10 times LOQ and 2 control samples. The amended samples will be processed through the analytical procedure developed to demonstrate the recovery of IKF-916 and its metabolites from amended water and to demonstrate control of bias. Reagent blanks will also be included as needed. The analytical method will be developed during the course of the study. Necessary modifications to the method will be documented, and a complete analytical procedure will be detailed in the final report. The final report will include only method validation data after the method has been developed.

SAMPLE PROCUREMENT AND RECEIPT

Samples will be obtained from local sources (laboratory, well, river or pond) and/or previous Ricerca studies. Upon arrival, samples will be issued a Ricerca, Inc. laboratory code and will be stored frozen.

SAMPLE IDENTIFICATION

Prior to analysis, the sample to be analyzed will be assigned a unique laboratory reference number. The reference number will be constructed to distinguish each laboratory sample that is processed through the analytical procedure. Each laboratory sample reference number and corresponding sample description will be recorded on operations and data collection forms. Chromatographic data will be identified by the laboratory sample reference number, or cross-referenced to the number.

DATA RECORDKEEPING

All chromatograms generated in support of method validation analyses will be properly labeled in accordance with the appropriate SOP. Method validation chromatograms and Operations and Data Collection forms (flowsheets) will be verified, approved and maintained in folders in the project activity file. Should the need arise, other raw data will be documented in a research notebook or on observation forms and will be signed, dated and verified. Exact copies of appropriate pages of the research notebook or the observation forms will be placed in the project activity file. Other relevant information will be placed in the project activity file in memo form.

Upon completion of the study, the project activity file will be submitted to Ricerca, Inc. Corporate Archives, 7528 Auburn Road, Painesville, Ohio 44077, for permanent storage.

GLP STATEMENT

The described study will be conducted in accordance with the "Good Laboratory Practice Standards," set forth in 40 CFR Part 160 of the Federal Register.

SAFETY AND HEALTH

- Laboratory personnel will practice good sanitation and health habits.
- Any health condition of laboratory personnel that may be considered to adversely affect the study will be reported to the Study Director.

- Any injury to laboratory personnel obtained during this study, which may be considered related to the test material, will be reported to the Study Director.

- Every reasonable precaution shall be taken to prevent inadvertent exposure of personnel and the environment to the test agent.

AMENDMENTS TO PROTOCOL

All protocol amendments will be expressed in writing, signed and dated by the Study Director.

Amendments usually will be issued prior to initiation of a protocol change. However, when a change is required without sufficient time to issue a written amendment, that change may be effected verbally by the Study Director and followed with a written amendment as soon as possible. In this case, the effective date of the written amendment will be the date of the verbal change. Changes made in this manner will be supported by written documentation in the activity file (observation form, letter, memo, etc.). Copies of the signed amendments will be appended to all distributed protocols.

REPORT

The Study Director and/or his designee at the conclusion of the study will prepare a final report. The report will accurately describe, but will not be limited to, the following:

- Name and address of the facility performing the study and the dates on which the study was initiated and completed.

- Objective as stated in the approved protocol and any changes to the original protocol.

- Reference and/or a detailed description of all methods used.

- Data generated while conducting the study and any transformations, calculations or operations performed on the data.

- Purity of analytical standards.

- Method validation results, observations, deviations from the protocol and/or standard operating procedures, and representative chromatograms.

- Name and signature of the Study Director.
- Statistical methods employed for analysis of the data, if necessary, will include the mean and standard deviation and may be applied to recovery data and controls.
- Locations where raw data and the final report are to be stored.
- The signed and dated statement by Ricerca Quality Assurance Unit regarding dates of study inspections and dates findings were reported.
- The signed and dated statement by the Study Director indicating that the study was conducted in compliance with GLP Standards 40 CFR Part 160.
- Additions or corrections to the report shall be in the form of an amendment by the Study Director. The amendment shall clearly identify that part of the report that is being altered and the reasons for the alterations. The amendment will be reviewed, signed and dated by the Ricerca Quality Assurance Unit, and signed and dated by the Study Director.

TEST FACILITY

Ricerca, Inc.
Department of Residue Analysis
7528 Auburn Road
Painesville OH 44077

STUDY DIRECTOR

J. E. Jablonski
Ricerca, Inc.
Department of Residue Analysis
7528 Auburn Road
Painesville OH 44077
Business Phone: (440) 357-3761

ASSOCIATE INVESTIGATOR

J. L. Wiedmann
Ricerca, Inc.
Department of Residue Analysis
7528 Auburn Road
Painesville OH 44077
Business Phone: (440) 357-3764

SPONSOR

Ishihara Sangyo Kaisha, Ltd.
3-15, Edobori, 1 chome, Nishi-ku
Osaka, 550-0002
JAPAN

Ricerca, Inc.

PROTOCOL AMENDMENT FORM

Ricerca Protocol 011140-0
Analytical Procedure for IKF-916 and Metabolites in Water

PROTOCOL AMENDMENT NO.: 1

DESCRIPTION OF AMENDMENT:

Page 10 Report
Sentence 1. . . . prepare a final report.

Change to:
. . . prepare two final reports. One report will provide the procedure for IKF-916 and the second report will provide the procedure for IKF-916, CCIM, CCIM-AM, CTCA and CCBA.

REASON FOR AMENDMENT:

The sponsor requested that separate reports be issued.

EFFECT ON THE STUDY:

None

EFFECTIVE DATE: October 20, 1999

APPROVAL SIGNATURES:

J. E. Jablonski — 11.5.99
Study Director — Date
Joe E. Jablonski

Jerome L Wiedmann — 11-5-99
Ricerca Management — Date
Jerome L. Wiedmann

S. Ogyu — Nov 9. 1999
Sponsor — Date
S. Ogyu
Ishihara Sangyo Kaisha, Ltd.

Ricerca, Inc.

PROTOCOL AMENDMENT FORM

Ricerca Protocol 011140-0
Analytical Procedure for IKF-916 and Metabolites in Water

PROTOCOL AMENDMENT NO.: 2

DESCRIPTION OF AMENDMENT:

Sample Procurement and Receipt

Sentence 2. . . . will be stored frozen.

Change to:

. . . will be stored refrigerated, frozen or kept cold by packing with ice. The pond water will have pH and total hardness determined by Ricerca, Inc. DOC (dissolved organic carbon) will be determined on the pond water by Galbraith Laboratories, Inc. 2323 Sycamore Drive, Knoxville, Tennessee 37921.

REASONS FOR AMENDMENT:

1. Preferred storage conditions are cold but not frozen for the pond water.
2. Water characterization is added to better define its method capability.

EFFECT ON THE STUDY:

1. None
2. Will enhance the value of the method to users.

EFFECTIVE DATE: November 5, 1999

APPROVAL SIGNATURES:

J. E. Jablonski — 11.5.99
Study Director — Date
Joe E. Jablonski

Jerome L. Wiedmann — 11-5-99
Ricerca Management — Date
Jerome L. Wiedmann

S. Ogyu — Nov 9, 1999
Sponsor — Date
S. Ogyu
Ishihara Sangyo Kaisha, Ltd.

APPENDIX B

Detailed Analytical Method

CHEMICALS

Acetic acid, glacial, Fisher TraceMetal Grade

Acetonitrile, Burdick & Jackson, High Purity

Celite 545®, Fisher Scientific, #C212-500

Hydrochloric acid, concentrated, Fisher ACS certified PLUS

IKF-916 Analytical Standard, Lot No. 9704-1, Purity 99.1%

Water, Burdick & Jackson, High Purity

(Chemicals with equivalent purity from alternate sources may be substituted.)

REAGENTS

(Quantities may be adjusted proportionately to make more or less reagent)

30/70 acetonitrile/water (v/v): add 30 mL of acetonitrile to 70 mL of water

0.5 N Hydrochloric Acid solution: dilute 4.13 mL concentrated HCl to 100 mL using HPLC water

HPLC Mobile Phase: 50/50 acetonitrile/water + 0.5% acetic acid

To prepare, add 10 mL glacial acetic acid to a mixture of 1 liter acetonitrile and 1 liter water

EQUIPMENT

Analytical electronic balance with 0.1-mg readability

Büchner funnels, 110 mm

Disposable syringes, 3-mL, B-D* Brand #390585

Extraction columns, BAKERBOND spe ™ C_{18}, 6 mL, 1000 mg per column, J. T. Baker, #7020-07

Filter paper, Whatman® 934-AH, 90 mm

Filters, syringe tip, 0.45 µm PTFE, Gelman Acrodisc #4422 (13 mm) or #4219 (25 mm)

Glassware: Assorted beakers, Erlenmeyer flasks with side-arms, graduated cylinder, culture tubes, graduated centrifuge tubes, pipets, volumetric flasks, etc.

pH paper, colorpHast® pH 0-14, EM-Reagents

Reservoirs for sep-pak cartridges, 20 mL, Supelco #57021

Stir bars

Turbo Vap® LV Evaporator, Zymark

Vacuum manifold for solid phase extraction, Supelco

Visiprep Large Volume Sampler for 6 mL SPE tubes, Supelco, #57275 modified with adapters for easy connect/disconnect (female Luer adapter #58721, male Luer to female 1/4-28, PEEK adapter #55072)

NOTE: Appropriate substitution for certain items are left to the analyst's discretion.

INSTRUMENTATION

High-performance liquid chromatograph (HPLC) with data system Model 620 quaternary pump

Waters Wisp 714 autosampler

Waters MS-600 controller and quaternary pump

Waters model 486 absorbance detector

PE-Nelson Turbochrom chromatography data system

Systec Model CH-1448 temperature controller

Systec Goldenfoil heating element

Helium for degassing mobile phases

HPLC column: The column listed below is recommended.

Ultracarb 3 ODS (20), 150 mm x 3.2 mm, Phenomenex No. 00F-0205-RO

HPLC guard column: SecurityGuard cartridge, C_{18} (ODS, Octadecyl), 4 mm L × 3 mm ID, Phenomenex No. AJO-4287

(Alternate HPLC Systems and/or columns may be used.)

STANDARD PREPARATION

Weigh 0.0500 g of IKF-916 analytical standard into a small vial. Quantitatively transfer the neat standard with rinses of acetonitrile into a Class A 100-mL volumetric flask. Dilute to the mark with acetonitrile to produce a stock standard with a concentration of 500 µg/mL IKF-916. Transfer 20 mL of the stock standard into a Class A 100-mL volumetric flask. Dilute to the mark with acetonitrile to produce a 100-µg/mL IKF-916 standard solution.

Prepare working standards from the stock standard described above as follows. Transfer 10 mL of the 100-µg/mL standard into a Class A 100-mL volumetric flask. Dilute to the mark with acetonitrile to produce a 10-µg/mL standard. Transfer 10 mL of the 10-µg/mL standard to a Class A 100-mL volumetric flask. Dilute to the mark with acetonitrile to produce a 1-µg/mL fortification standard. Transfer 20 mL of the 1-µg/mL fortification standard to a Class A 100-mL volumetric flask. Dilute to the mark with acetonitrile to produce a 0.2-µg/mL fortification standard.

Prepare IKF-916 calibration standards of concentration 0.025, 0.05, 0.10, 0.25, 0.50 and 1.00 µg/mL with 30/70 acetonitrile/water (see Reagents).

The following table shows volumes of standards and sample dilution solvent needed to prepare 20 mL of each calibration standard for IKF-916. Each calibration standard was prepared in a 30 mL amber vial.

Calibration Standard Concentration	Volume of Standard Used to Prepare	Volume of 30/70 ACN/H_2O
1.00 µg/mL	2.0 mL of 10 µg/mL*	18.0 mL
0.50µg/mL	1.0 mL of 10 µg/mL*	19.0 mL
0.25 µg/mL	0.5 mL of 10 µg/mL*	19.5 mL
0.10 µg/mL	2.0 mL of 1.00 µg/mL	18.0 mL
0.05 µg/mL	2.0 mL of 0.50 µg/mL	18.0 mL
0.025 µg/mL	2.0 mL of 0.25 µg/mL	18.0 mL

*Prepare a 10 µg/mL working standard as follows. Transfer 10 mL of the 100 µg/mL standard solution prepared above to a 100 mL class A volumetric flask. Dilute to the mark with 20 mL of acetonitrile and 70 mL of water.

NOTE: Recommended expiration interval for the calibration standards is one month. All standard solutions are stored in freezers.

SAMPLE EXTRACTION

1. Measure a 1 liter aliquot of the water sample into a beaker. Add a stir bar.
2. Add HCl to reach pH 3. Use a dilute HCl solution (0.5 N). Stir while adding the acid. Use pH paper to determine the pH.
3. Fortify with IKF-916.
4. For dirty samples (pond or river water): Add 1 scoop (≈ 7 grams) of Celite 545® to the beaker of sample and stir. Prepare a bed of Celite (15 – 20 grams) on a glass fiber filter paper in a Büchner funnel. Filter sample through the bed of Celite into a side-arm flask.
5. Setup the solid phase extraction manifold.
6. Condition BAKERBOND C_{18} columns with 5 mL acetonitrile then 5 mL water.
7. Pull sample onto column using Visiprep Large Volume Sampler.
8. Rinse sample container with 10 mL HPLC water. Pull onto column.
9. Elute column with 10 mL acetonitrile into a small beaker or vial using vacuum. Stop the solvent front at the top of the packing.
10. Elute with 3 mL water into the same container as the previous step. Allow column to pull dry.
11. Quantitatively transfer column eluate to a culture tube with rinses of acetonitrile.
12. Concentrate sample to approximately 3 mL in a TurboVap at 38°C using additions of acetonitrile to drive off the water.
13. Transfer to 10 mL graduated centrifuge tube with acetonitrile. Concentrate to approximately 0.2 – 0.3 mL.

14. Dilute with 30/70 acetonitrile/water (v/v). Filter through 0.45 µm PTFE filter. Load into amber HPLC vials.

HPLC QUANTITATION

Instrument Setup

Analytical column: Ultracarb 3 ODS 20; 150 mm × 3.2 mm
Guard column: SecurityGuard cartridge, C_{18} (ODS, Octadecyl), 4 mm L × 3 mm ID
Mobile Phase: 50/50 acetonitrile/water (v/v) + 0.5% acetic acid
Flow Rate: 0.5 mL/min
Column Temp: 40 ° C
Injection Volume: 100 µL

Detection: UV absorbance
Detector: Waters
Wavelength: 280 nm
Sensitivity: 0.01 AUFS

Instrument Calibration

Under the instrumental conditions described above the approximate retention time of IKF-916 is approximately 14 minutes.

The normal range of calibration standards is listed below.

ID	Concentration
LEV1	0.025 µg/mL
LEV2	0.05 µg/mL
LEV3	0.10 µg/mL
LEV4	0.25 µg/mL
LEV5	0.50 µg/mL
LEV6	1.00 µg/mL

APPENDIX C

Representative Chromatograms and Sample Calculations

EXPLANATION OF CHROMATOGRAMS

Figures C-1 through C-19 show the chromatograms and calibration data (as a representative set) generated for the analysis of IKF-916 in pond water.

Figure C-1 shows the calibration curve generated by the Turbochrom data system from the injection of the 6 calibration standards shown in Figures C-2 through C-7. The coefficient of determination for this linear calibration plot was 0.999906. The pond water control sample chromatograms, PWC1 and PWC2, are shown in Figures C-8 and C-9, respectively. There was some very small interference peaks near the retention time of 14.2 minutes of IKF-916 in these controls. The UV 280 nm responses of these peaks were well below the response of the 0.025 µg/mL calibration standard injected with the sample set. Therefore, the background peaks were considered to be non-detects. Figures C-10 through C-14 show chromatograms of the 0.1 ppb fortified samples PWLOQ1, PQLOQ2, PWLOQ3, PWLOQ4 and PWLOQ5, respectively. The controls and 0.01 ppb fortified samples were diluted to a volume of 1 mL for HPLC analysis. The dilution volume is listed as the 'multiplier' on the Turbochrom report. Figures C-15 through C-19 show chromatograms of the 1 ppb fortified samples PW10XLOQ1, PW10XLOQ2, PW10XLOQ3, PW10XLOQ4 and PW10XLOQ5, respectively. The 1 ppb fortifications were diluted to a volume of 2 mL for HPLC analysis. The 'divisor' listed on the Turbochrom report is the sample volume, which was 1000 mL.

Figures C-20 through C-23 show representative chromatograms from the validation of tap water. Figure C-20 shows the 0.10 µg/mL calibration standard injected with the tap water set. The IKF-916 retention time was about 14.3 minutes. Analysis of tap water was conducted using the same chromatographic conditions used for analysis of pond water. Figure C-21 shows a representative tap water control sample, TWC1. Figures C-22 and C-23 show chromatograms from representative 0.1 ppb and 1 ppb IKF-916 fortified tap water samples, respectively.

SAMPLE CALCULATIONS

Residues of IKF-916 were calculated by electronic integration of the detector signal. The analog voltage measured by the absorbance detector was digitized in a PE-Nelson 900 Series interface and sent to the data system. The computer data system used to integrate the signal and calculate results was Perkin-Elmer Turbochrom Client Server version 6.1. Recovery calculations were performed using functions in Microsoft Excel ™.

The equation used to quantitate residues is shown below.

$$\text{Gross residue ppb} = \frac{(D - I)}{S} * \frac{V}{W} * \frac{1000mL}{L}$$

Where: S = Slope (µV-sec/µg/mL)
D = Detector Response,(Peak Area, µV-sec)
I = Intercept (µV -sec)
V = Volume for HPLC or Multiplier (mL)
W = Sample Aliquot Volume (mL)

For pond water sample PWLOQ1 (see Figure C-10, page 54), the residue was calculated as follows:

$$\text{Gross Residue ppb} = \frac{46076 - (-572.581867)}{463454.800624} * \frac{1}{1000} * 1000$$

$$= 0.1007\ ppb$$

$$= 0.101\ ppb\ (rounded)$$

$$\%\ Recovery = \frac{(ppb\ found - ppb\ control) * 100}{ppb\ added}$$

$$\%\ \text{Recovery} = \frac{(0.101 - 0) * 100}{0.1} = 101\ \%$$

The calibration curve coefficients were calculated by the Turbochrom data system using the peak area responses of the IKF-916 external calibration standards injected with the samples.

The limit of detection (LOD) was 0.025 ppb. The LOD was based on the residue equivalent to the lowest level calibration standard injected (0.025 µg/mL), and a concentration factor of 1. For water samples, 1000 mL aliquots of sample were diluted in 1 mL of solvent for HPLC analysis. The LOD is then calculated as:

$$0.025\ \text{ppb} = 0.025\ \mu\text{g/mL} \times \frac{1\ \text{mL}}{1000\ \text{mL}} \times \frac{1000\ \text{mL}}{\text{L}}$$

Peaks at the retention time of IKF-916 in control samples with area counts equal to or greater than the area of the 0.025 µg/mL calibration standard were

considered to be detections, and the residues were quantified. No peaks were found in any control sample which met this criteria.

LIST OF FIGURES

Figure C - 1: Turbochrom Calibration Curve Plot for IKF-916, Pond Water Validation Set

Fit Analysis Output For Method File: \\Hemlock\tccs_data\Residue\Proj11140\WAT-01\916-1108w.mth
Component Name : IKF-916
Date : 11/29/99 10:05:29 AM

Curve Parameters:

Curve #1 : 1st Order
Weighting Factor = 1/x R-Squared = 0.999906
Calibration Curve : Y = (-572.581867) +(463454.800624) X

Curve #1 :

Level Name	Observed X-Value	Calculated X-Value	Delta	%Diff.	Observed Y-Value	Calculated Y-Value	Delta	%Diff.
lev1	0.025000	0.025326	-3.263e-04	-1.288	11165.000000	11013.788	151.212	1.373
lev2	0.050000	0.048435	0.001565	3.230	21875.000000	22600.158	-725.158	-3.209
lev3	0.100000	0.100591	-5.914e-04	-0.588	46047.000000	45772.898	274.102	0.599
lev4	0.250000	0.252863	-0.002863	-1.132	116618.000000	115291.118	1326.882	1.151
lev5	0.500000	0.503092	-0.003092	-0.615	232588.000000	231154.818	1433.182	0.620
lev6	1.000000	0.994692	0.005308	0.534	460422.000000	462882.219	-2460.219	-0.531

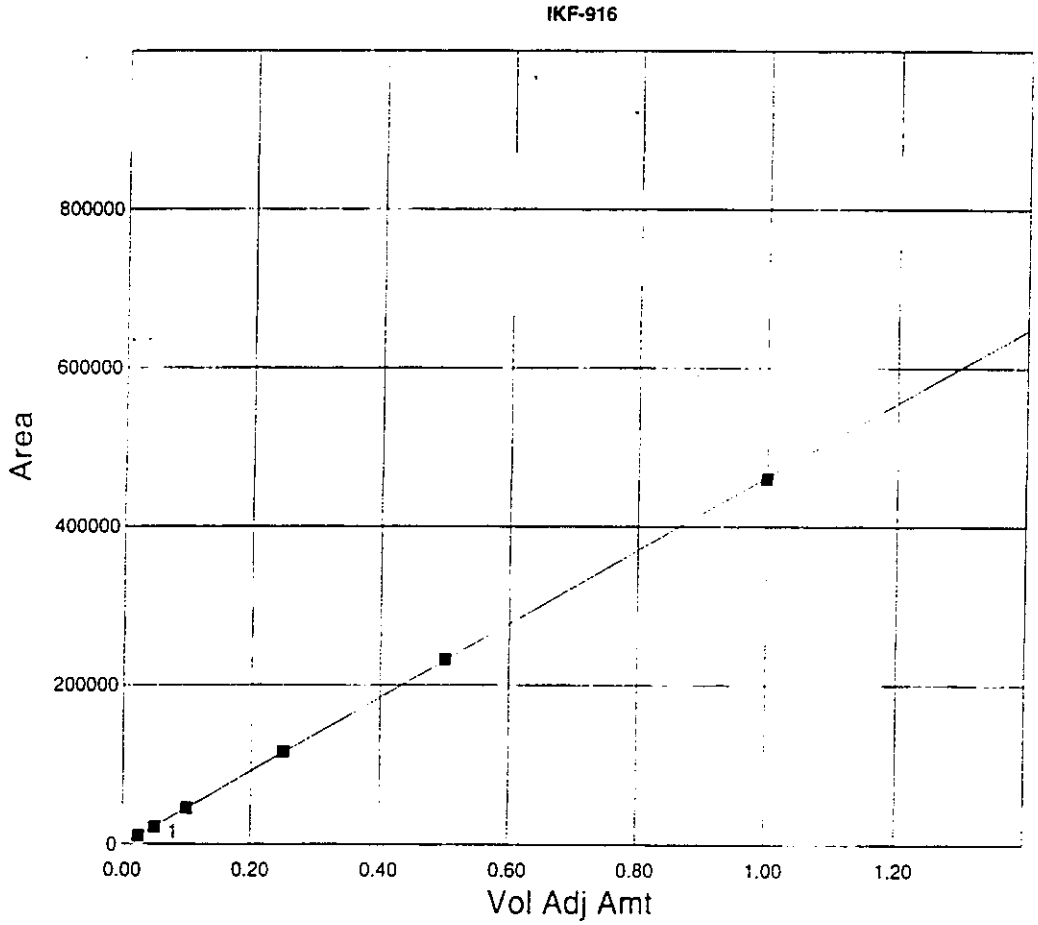


Figure C - 2: IKF-916 Calibration Standard, 0.025 µg/mL

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Sample Name	: 0.025 ug/mL	Data Acquisition Time	: 11/08/99 03:54:30 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 1		

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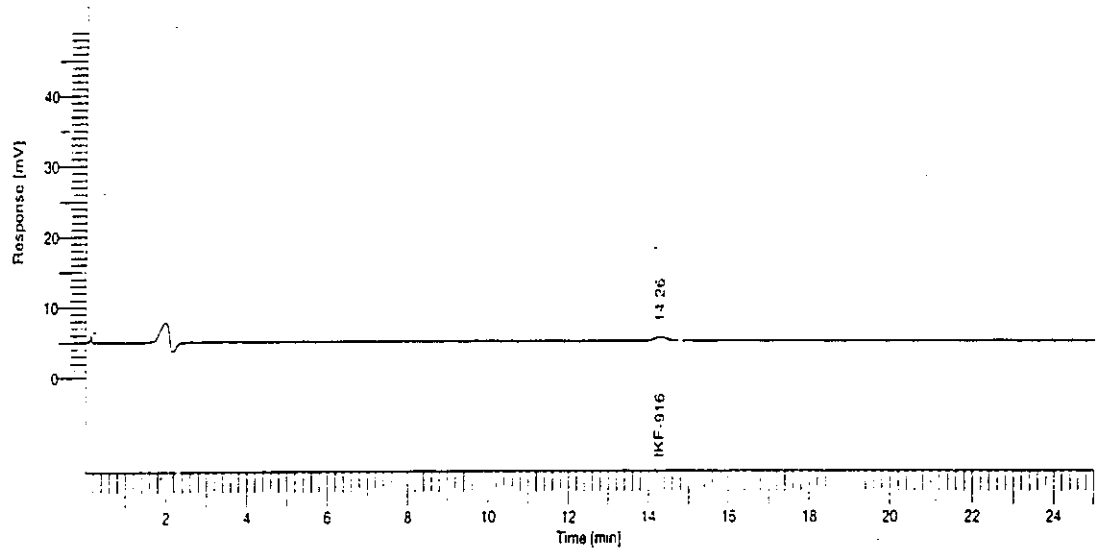

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.264	11165.00	BB	1.0	1.00	25.3263

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 3: IKF-916 Calibration Standard, 0.05 µg/mL

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:00:36 AM
Sample Name	: 0.05 ug/mL	Data Acquisition Time	: 11/08/99 05:50:45 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 4		

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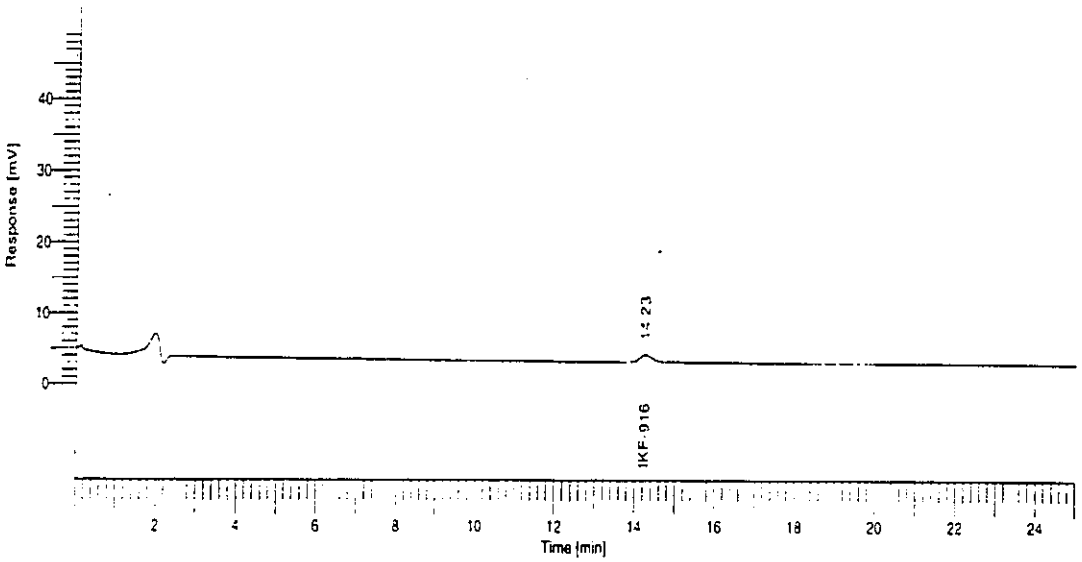

IKF-916

Peak #	Component Name	Time [min]	Area [µV-s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.231	21875.00	BB	1.0	1.00	48.4353

PROJECT: 011140
INSTRUMENT: HO-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 4: IKF-916 Calibration Standard, 0.10 µg/mL

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:00:47 AM
Sample Name	: 0.1 ug/mL	Data Acquisition Time	: 11/08/99 07:47:00 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 7		

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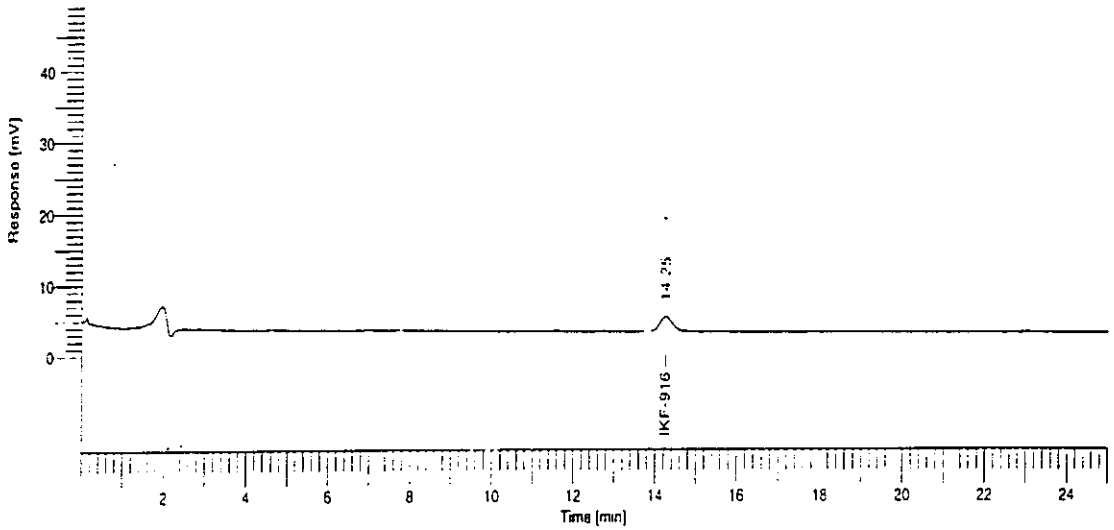

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.250	46047.00	BB	1.0	1.00	100.5914

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 5: IKF-916 Calibration Standard, 0.25 µg/mL

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:01:00 AM
Sample Name	: 0.25 ug/mL	Data Acquisition Time	: 11/08/99 10:22:00 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 11		

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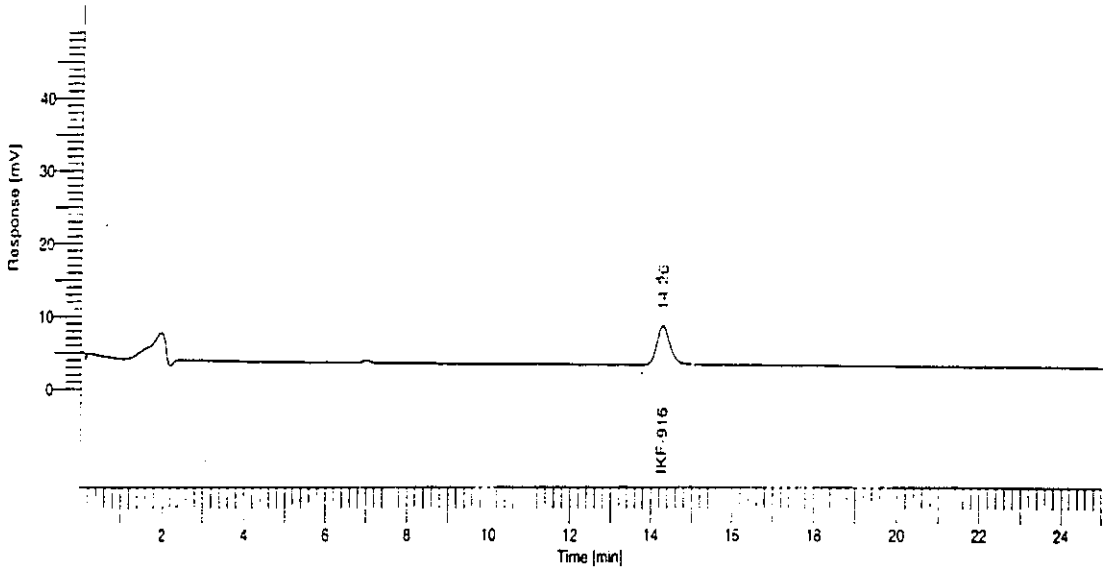

IKF-916

Peak #	Component Name	Time [min]	Area [µV-s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.257	116618.00	BB	1.0	1.00	252.8630

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 6: IKF-916 Calibration Standard, 0.5 µg/mL

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:01:11 AM
Sample Name	: 0.5 ug/mL	Data Acquisition Time	: 11/09/99 12:18:14 AM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 14		

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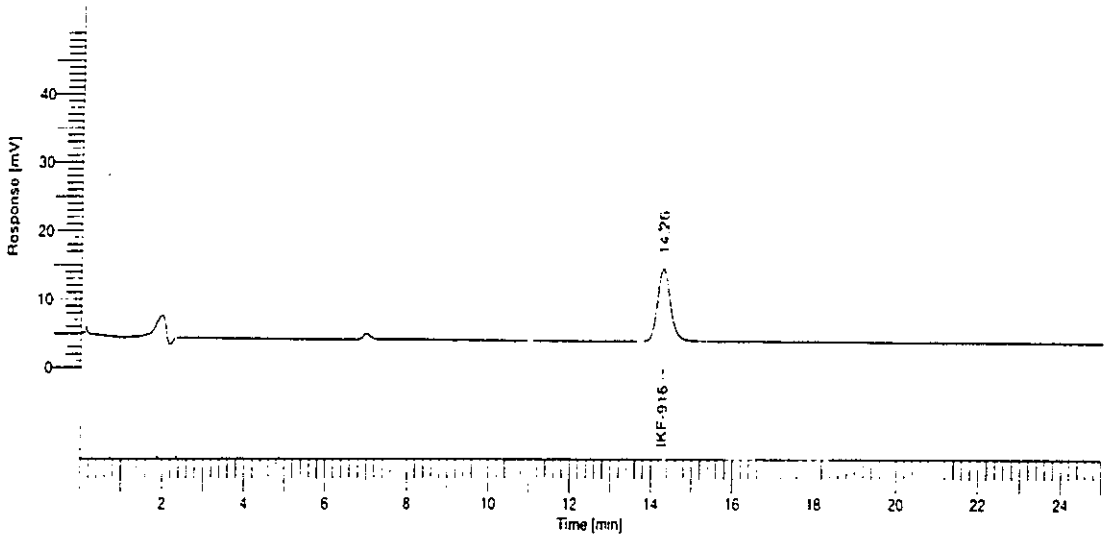

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.263	232588.00	*BB	1.0	1.00	503.0924

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 7: IKF-916 Calibration Standard, 1.0 µg/mL

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:01:25 AM
Sample Name	: 1.0 ug/mL	Data Acquisition Time	: 11/09/99 02:53:14 AM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 18		

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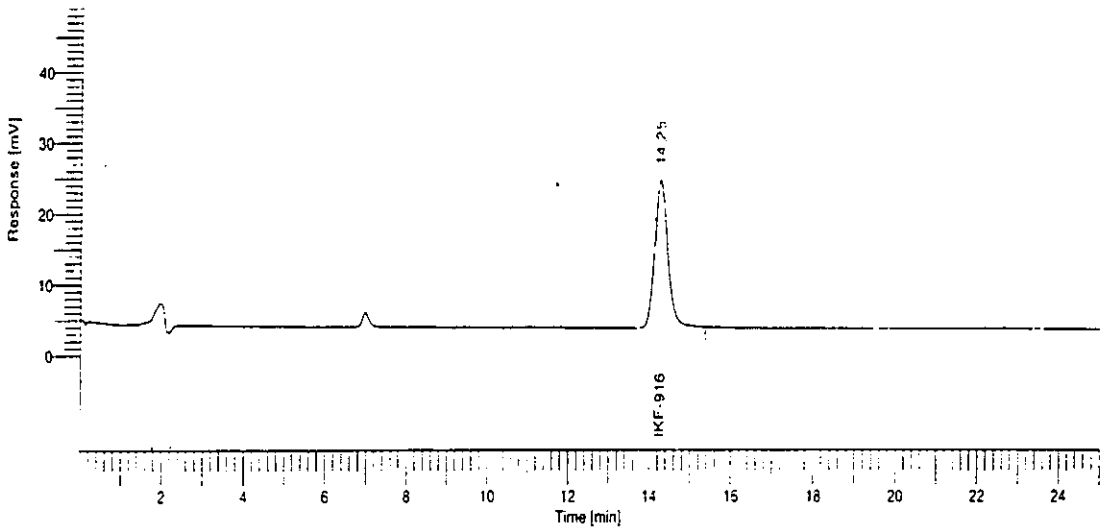

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.250	460422.00	*BB	1.0	1.00	994.6916

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 8: Control Pond Water Sample PWC1

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:00:28 AM
Sample Name	: PWC1	Data Acquisition Time	: 11/08/99 04:33:15 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 2		

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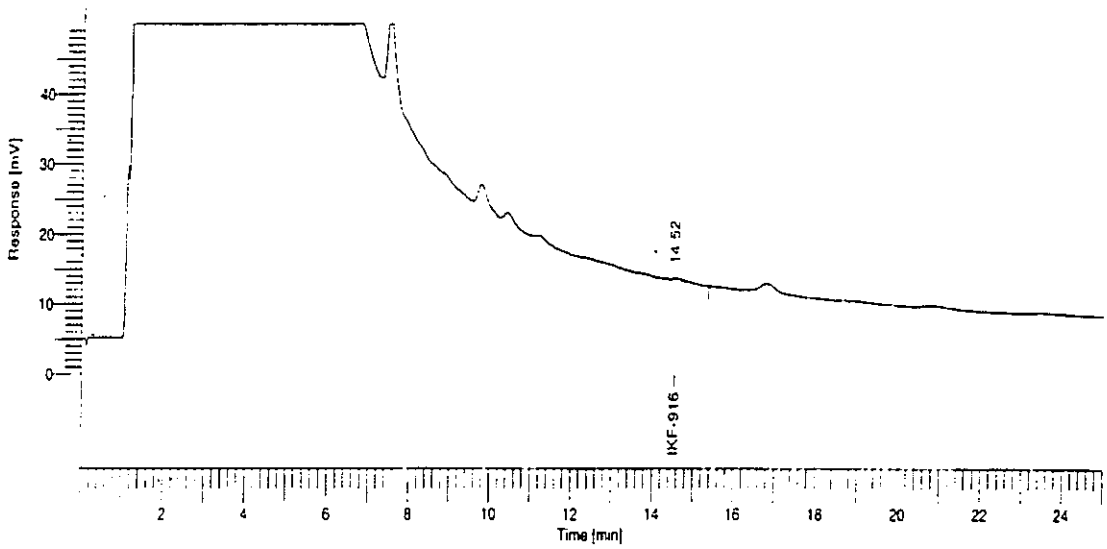

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.524	5171.00	*BB	1.0	1e+03	0.0124

Peak is not IKF-916 based on retention time
Response is lower than response of 0.025 µg/mL Standard
JKJ 11/29/99

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 9: Control Pond Water Sample PWC2

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:00:32 AM
Sample Name	: PWC2	Data Acquisition Time	: 11/08/99 05:12:01 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 3		

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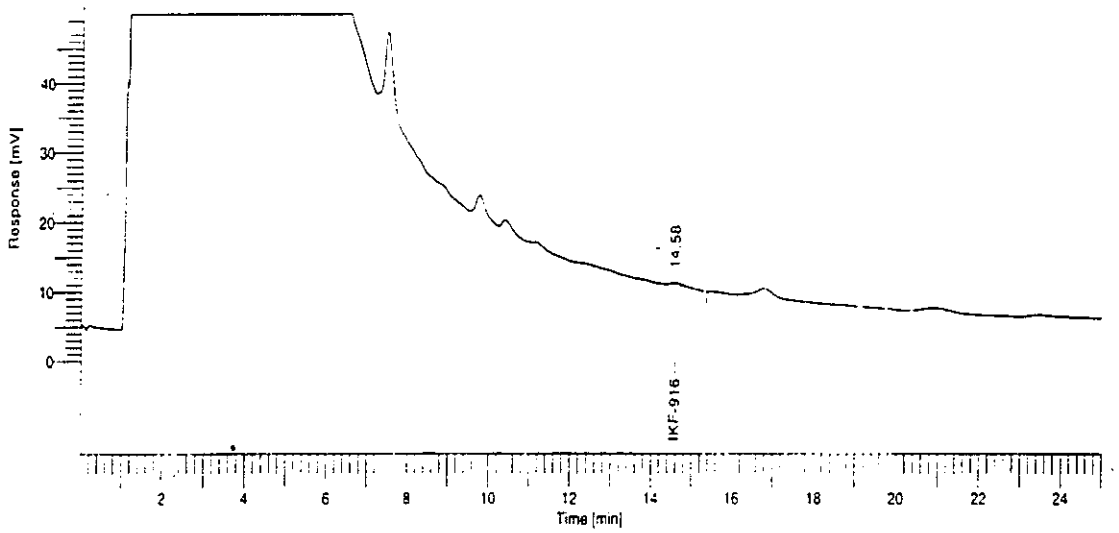

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.576	6533.00	*BB	1.0	1e+03	0.0153

Peak is not IKF-916 based on retention time
Response is lower than response of 0.025 µg/mL Standard.
JJ 11/29/99

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 10: IKF-916 Fortified (0.1 ppb) Pond Water Sample PWLOQ1

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:00:39 AM
Sample Name	: PWLOQ1	Data Acquisition Time	: 11/08/99 06:29:30 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 5		

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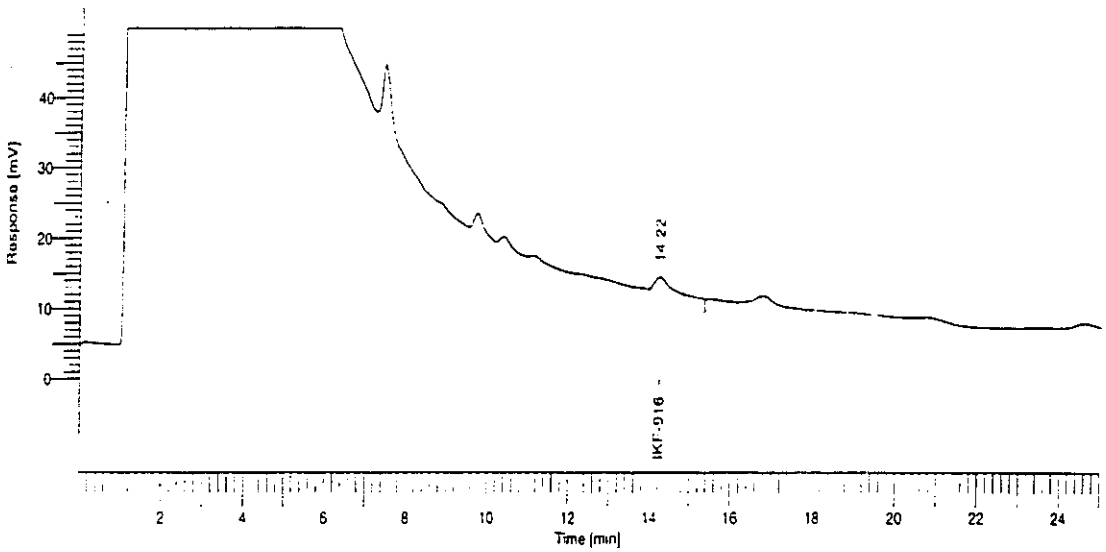

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.222	46076.00	*BB	1.0	1e+03	0.1007

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 11: IKF-916 Fortified (0.1 ppb) Pond Water Sample PWLOQ2

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:00:43 AM
Sample Name	: PWLOQ2	Data Acquisition Time	: 11/08/99 07:08:15 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 6		

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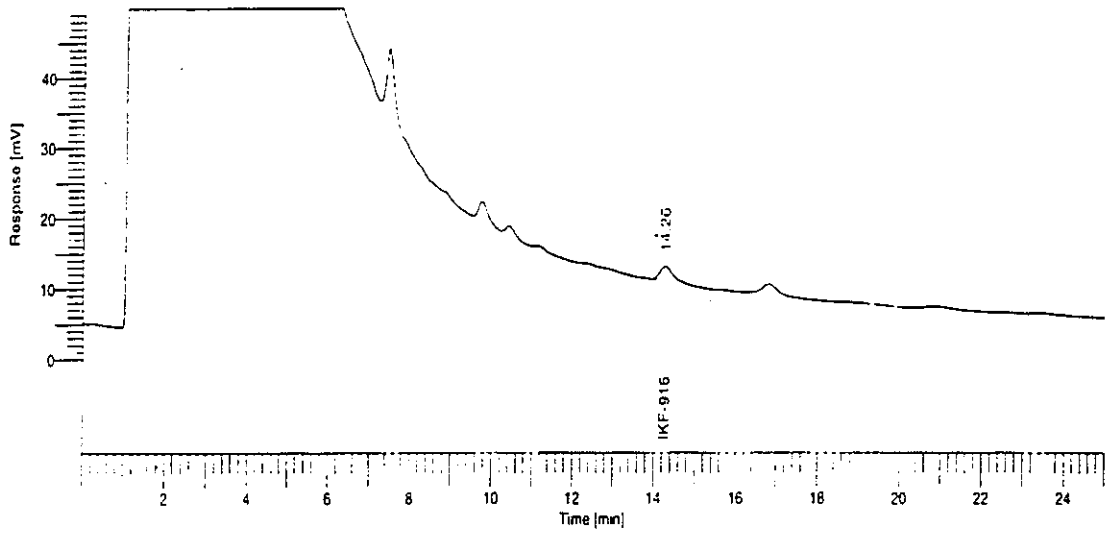

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.258	48042.50	*BB	1.0	1e+03	0.1049

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 12: IKF-916 Fortified (0.1 ppb) Pond Water Sample PWLOQ3

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:00:50 AM
Sample Name	: PWLOQ3	Data Acquisition Time	: 11/08/99 08:25:45 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 8		

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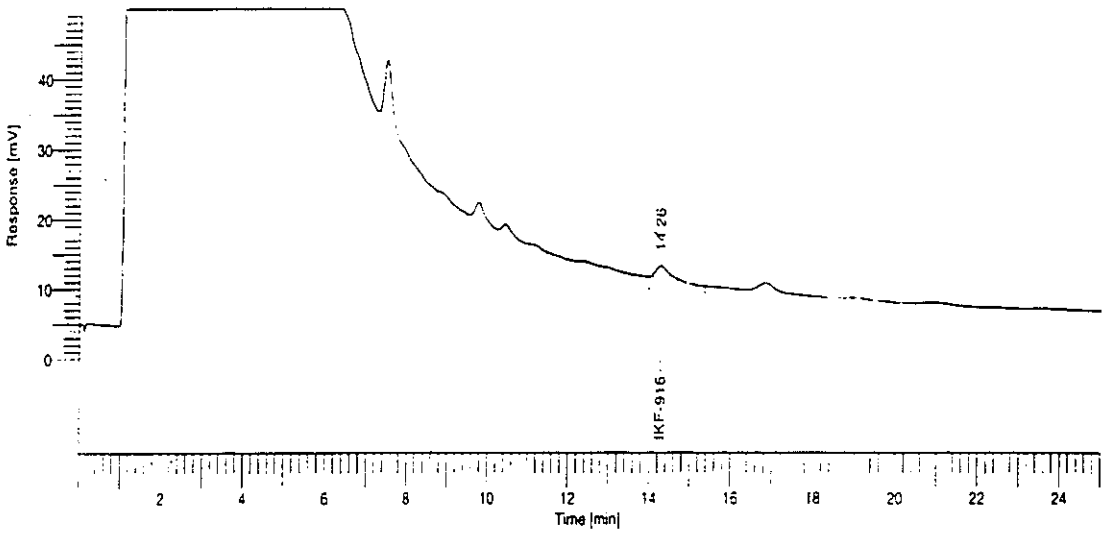

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.263	42586.00	*BB	1.0	1e+03	0.0931

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 13: IKF-916 Fortified (0.1 ppb) Pond Water Sample PWLOQ4

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:00:53 AM
Sample Name	: PWLOQ4	Data Acquisition Time	: 11/08/99 09:04:30 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 9		

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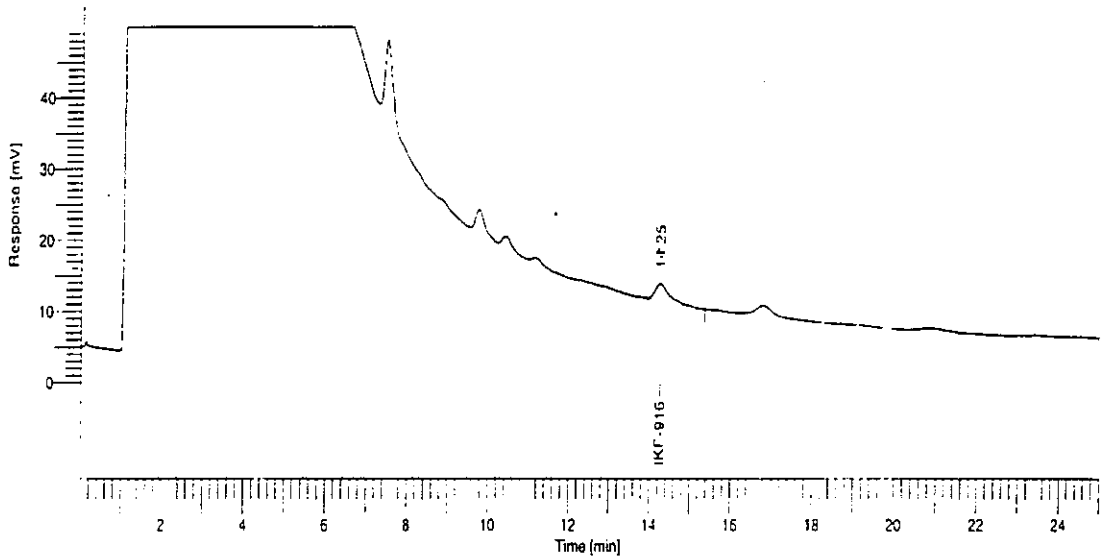

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.247	53201.00	*BB	1.0	1e+03	0.1160

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 14: IKF-916 Fortified (0.1 ppb) Pond Water Sample PWLOQ5

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:00:56 AM
Sample Name	: PWLOQ5	Data Acquisition Time	: 11/08/99 09:43:15 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 10		

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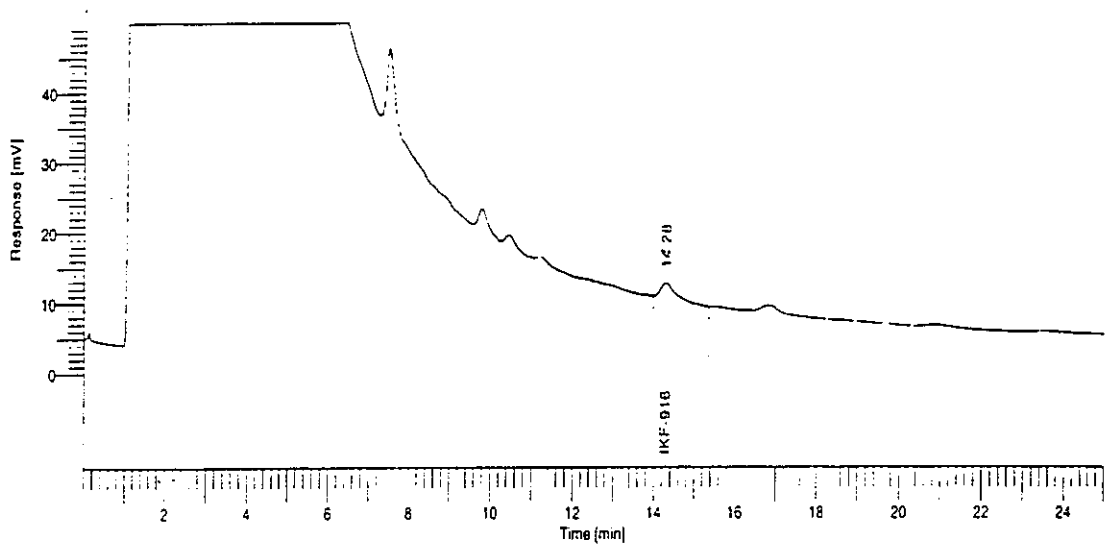

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.275	49296.00	*BB	1.0	1e+03	0.1076

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 15: IKF-916 Fortified (1 ppb) Pond Water Sample PW10XLOQ1

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:01:04 AM
Sample Name	: PW10XLOQ1	Data Acquisition Time	: 11/08/99 11:00:44 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 12		

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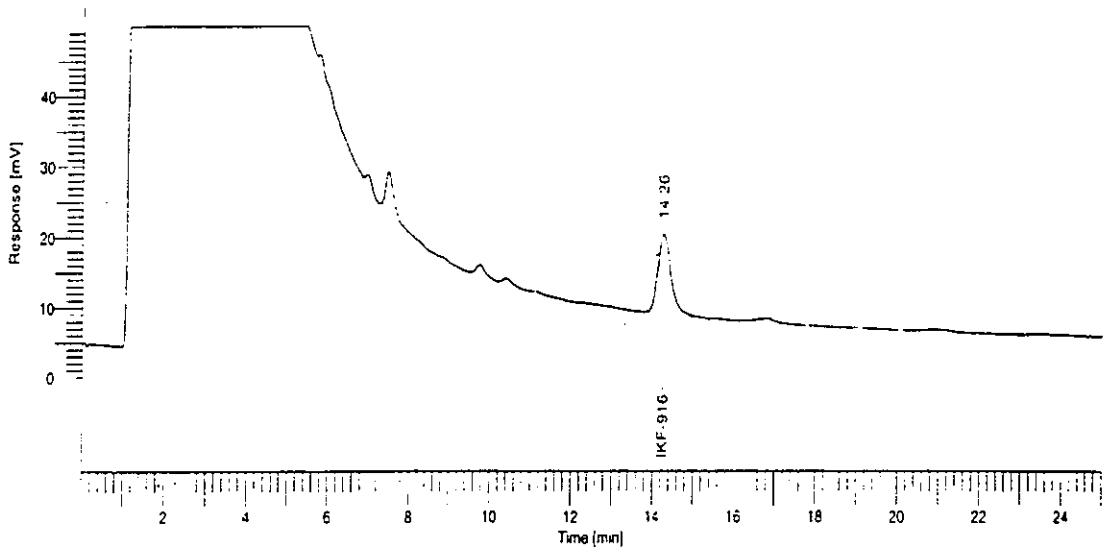

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.256	254269.50	*BB	2.0	1e+03	1.0997

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 16: IKF-916 Fortified (1 ppb) Pond Water Sample PW10XLOQ2

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:01:07 AM
Sample Name	: PW10XLOQ2	Data Acquisition Time	: 11/08/99 11:39:29 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 13		

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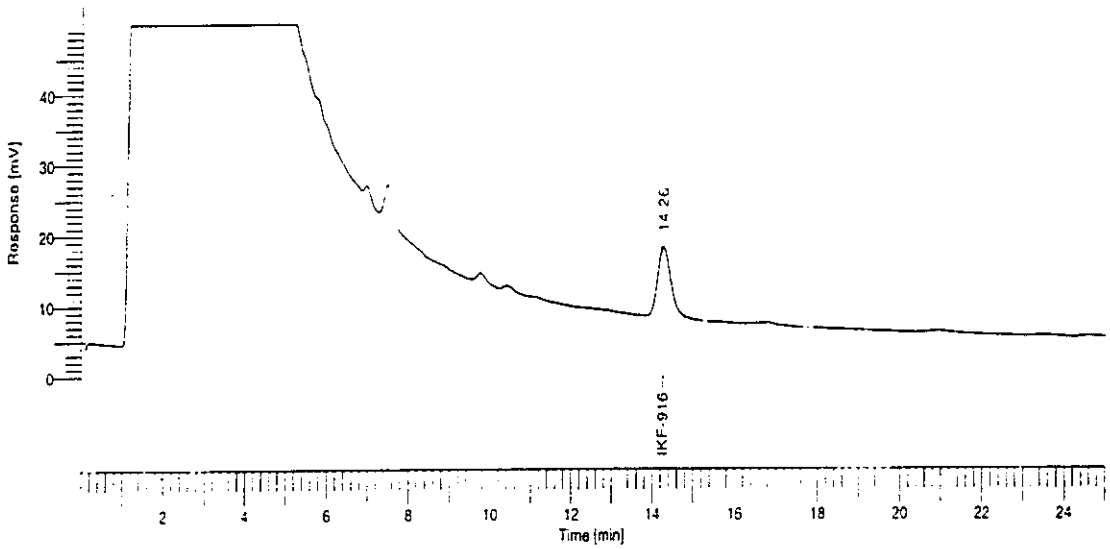

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.256	225828.50	*BB	2.0	1e+03	0.9770

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 17: IKF-916 Fortified (1 ppb) Pond Water Sample PW10XLOQ3

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:01:14 AM
Sample Name	: PW10XLOQ3	Data Acquisition Time	: 11/09/99 12:56:59 AM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 15		

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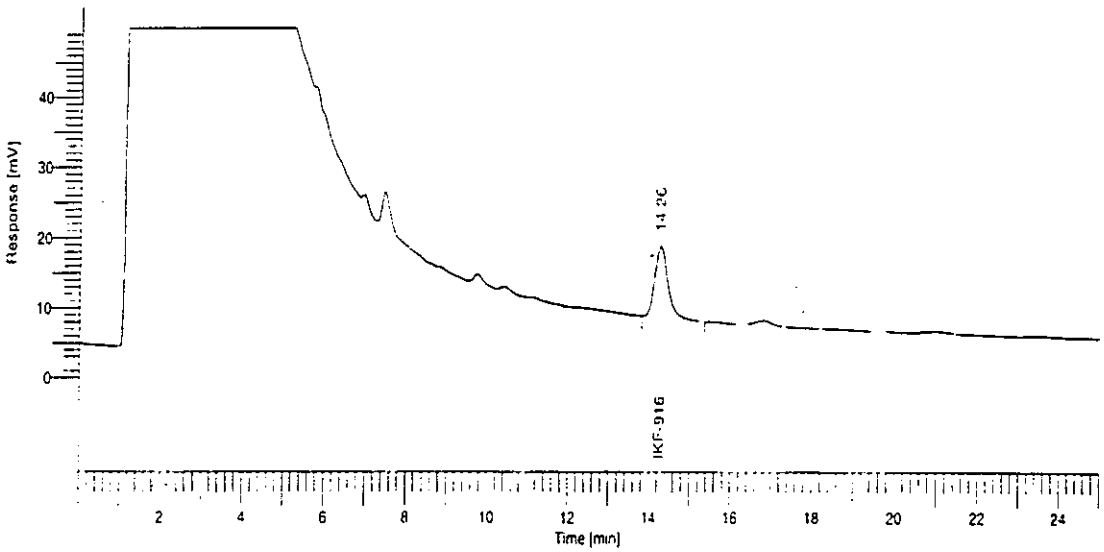

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.262	226040.00	*BB	2.0	1e+03	0.9779

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 18: IKF-916 Fortified (1 ppb) Pond Water Sample PW10XLOQ4

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:01:17 AM
Sample Name	: PW10XLOQ4	Data Acquisition Time	: 11/09/99 01:35:44 AM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 16		

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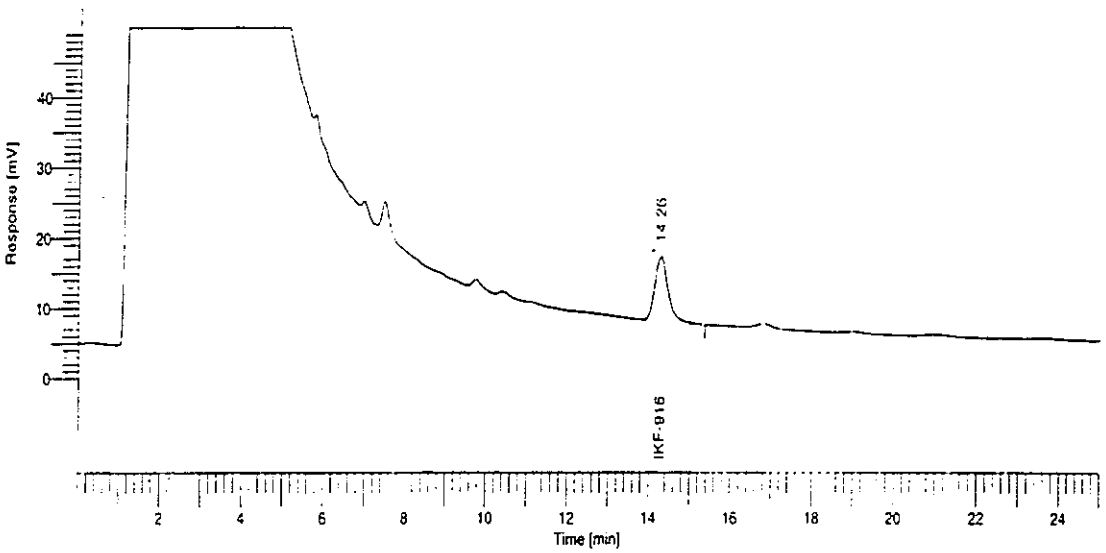

IKF-916

Peak #	Component Name	Time [min]	Area [µV-s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.261	203641.00	*BB	2.0	1e+03	0.8813

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 19: IKF-916 Fortified (1 ppb) Pond Water Sample PW10XLOQ5

Software Version	: 6.1.0.2:G07	Date	: 11/29/99 10:01:21 AM
Sample Name	: PW10XLOQ5	Data Acquisition Time	: 11/09/99 02:14:29 AM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 17		

Result File : \\hemlock\Tccs_Data\Residue\Proj11140\WAT-01\1108a017.rst
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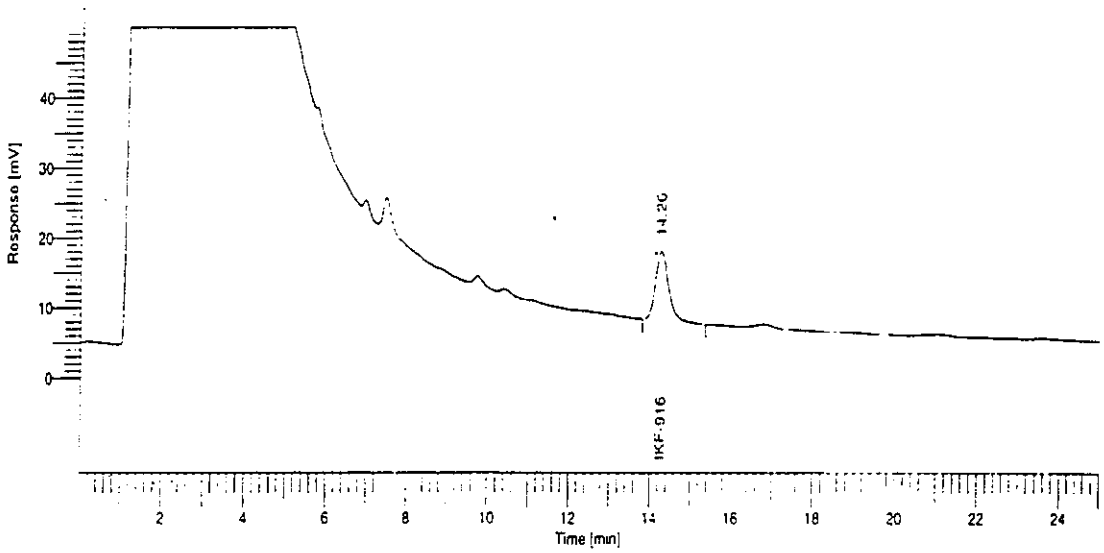

IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.259	218913.00	*BB	2.0	1e+03	0.9472

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN:Luna, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 20: IKF-916 Calibration Standard, 0.10 µg/mL

Software Version	: 6.1.0.2:G07	Date	: 11/10/99 07:21:05 AM
Sample Name	: 0.1 ug/mL	Data Acquisition Time	: 11/09/99 08:00:55 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 7		

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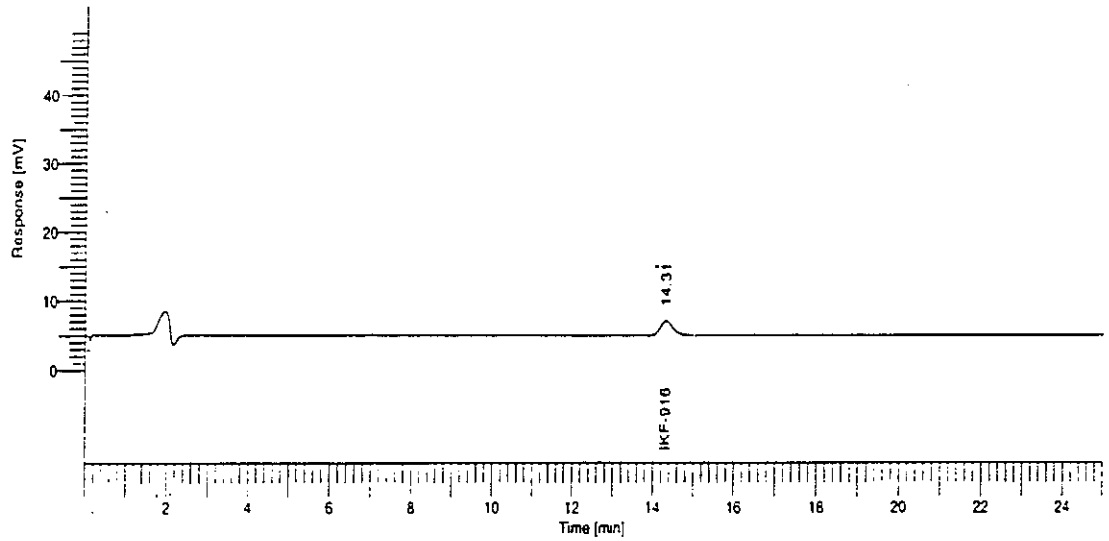


IKF-916

Peak #	Component Name	Time [min]	Area [µV·s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.307	45677.00	BB	1.0	1.00	98.5615

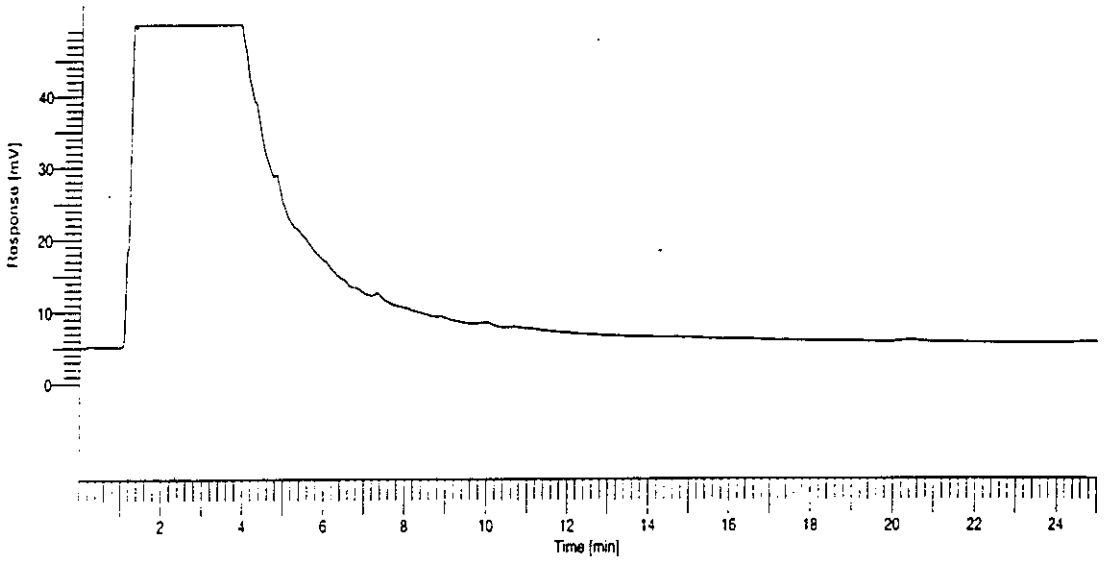
PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN: Ultracarb ODS 20, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 21: Control Tap Water Sample TWC1

Software Version	: 6.1.0.2:G07	Date	: 11/10/99 07:20:47 AM
Sample Name	: TWC1	Data Acquisition Time	: 11/09/99 04:47:10 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 2		

Result File : \\hemlock\Tccs_Data\Residue\Proj11140\WAT-01\1109a002.rst
Sequence File : \\hemlock\Tccs_Data\Residue\Proj11140\WAT-01\916-1109w.seq



IKF-916

No peaks available to report

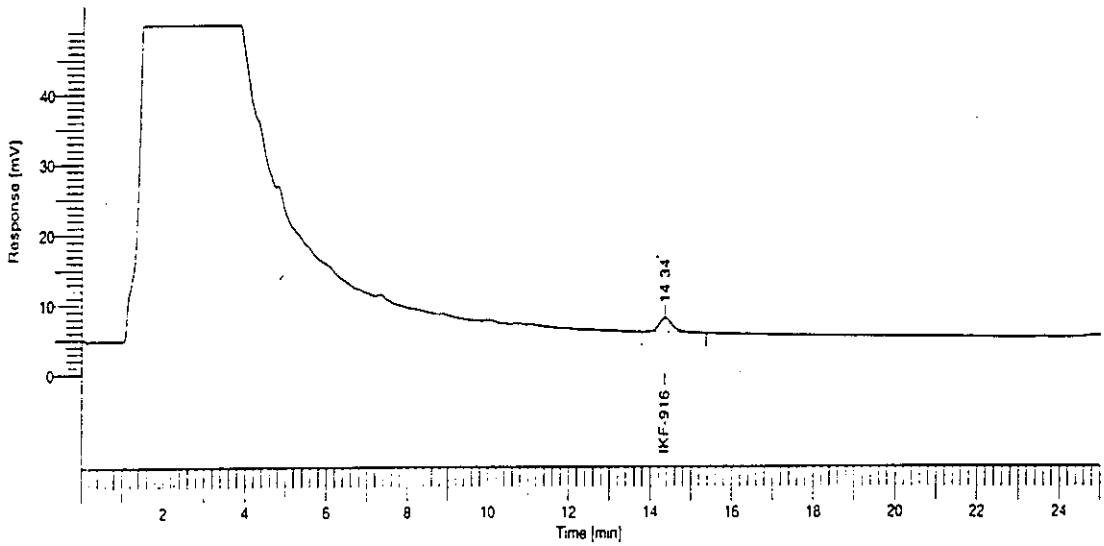
PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN: Ultracarb ODS 20, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 22: IKF-916 Fortified (0.1 ppb) Tap Water Sample TWLOQ1

Software Version	: 6.1.0.2:G07	Date	: 11/10/99 07:20:58 AM
Sample Name	: TWLOQ1	Data Acquisition Time	: 11/09/99 06:43:25 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 5		

Result File : \\hemlock\Tccs_Data\Residue\Proj11140\WAT-01\1109a005.rst
Sequence File : \\hemlock\Tccs_Data\Residue\Proj11140\WAT-01\916-1109w.seq



IKF-916

Peak #	Component Name	Time [min]	Area [µV-s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.337	47609.00	*BB	1.0	1e+03	0.1027

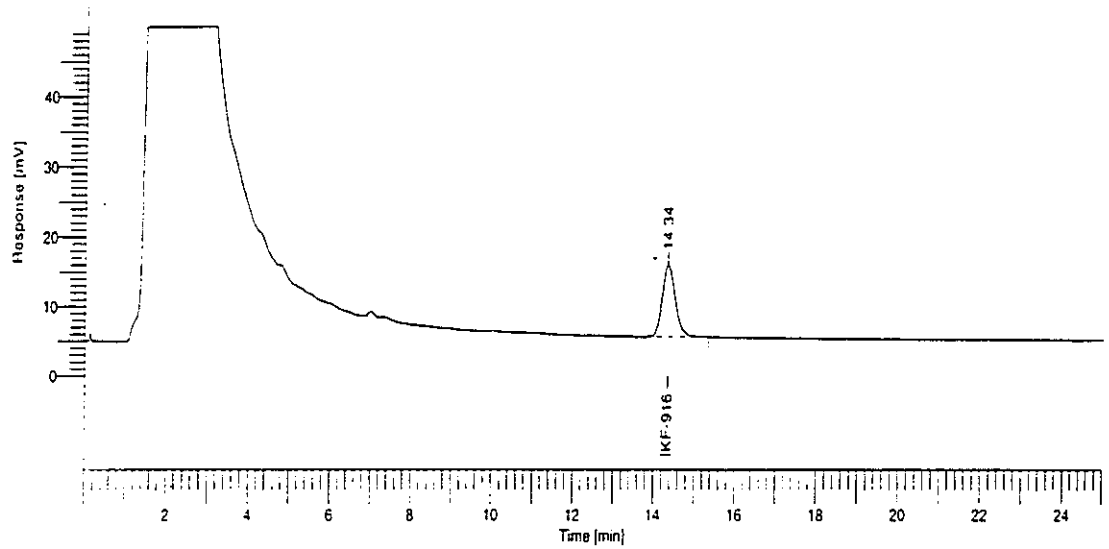
PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN: Ultracarb ODS 20, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

Figure C - 23: IKF-916 Fortified (1 ppb) Tap Water Sample TW10XLOQ1

Software Version	: 6.1.0.2:G07	Date	: 11/10/99 07:21:21 AM
Sample Name	: TW10XLOQ1	Data Acquisition Time	: 11/09/99 11:14:40 PM
Instrument Name	: RA-HPLC-WAT-01	Channel	: A
Rack/Vial	: 0/0	Operator	: jablonski_j
Sample Amount	: 1.000000	Dilution Factor	: 1.000000
Cycle	: 12		

Result File : \\hemlock\Tccs_Data\Residue\Proj11140\WAT-01\1109a012.rst
Sequence File : \\hemlock\Tccs_Data\Residue\Proj11140\WAT-01\916-1109w.seq



IKF-916

Peak #	Component Name	Time [min]	Area [µV-s]	BL	Multiplier	Divisor	Results ppb
1	IKF-916	14.339	233788.00	*BB	2.0	1e+03	1.0070

PROJECT: 011140
INSTRUMENT: HQ-150-C
COLUMN: Ultracarb ODS 20, C18(2) , 150 x 3.2mm, 3um particle size
GUARD COLUMN: Security Guard, C18
MOBILE PHASE: 50/50 Acetonitrile/Water + 0.5% acetic acid
FLOW RATE: 0.5 mL/min, INJ.VOL.: 100 ul, COL. TEMP.: 40 c
DETECTOR: Waters 486 MS absorbance detector, wavelength=280 nm,sensitivity=0.01 AUFS,filter=1.0

ANALYSTS: Joe Jablonski, Nancy Jordan, Bill Debevc

APPENDIX D

Sample History

Table D - 1: Sample History – Pond Water (Significant Dates)

Sample ID	Extract Date	Analysis Date
PWC1	11/8/99	11/8/99
PWC2	11/8/99	11/8/99
PWLOQ1	11/8/99	11/8/99
PWLOQ2	11/8/99	11/8/99
PWLOQ3	11/8/99	11/8/99
PWLOQ4	11/8/99	11/8/99
PWLOQ5	11/8/99	11/8/99
PW10XLOQ1	11/8/99	11/8/99
PW10XLOQ2	11/8/99	11/8/99
PW10XLOQ3	11/8/99	11/9/99
PW10XLOQ4	11/8/99	11/9/99
PW10XLOQ5	11/8/99.	11/9/99

Table D - 2: Sample History – Tap Water (Significant Dates)

Sample ID	Extract Date	Analysis Date
TWC1	11/9/99	11/9/99
TWC2	11/9/99	11/9/99
TWLOQ1	11/9/99	11/9/99
TWLOQ2	11/9/99	11/9/99
TWLOQ3	11/9/99	11/9/99
TWLOQ4	11/9/99	11/9/99
TWLOQ5	11/9/99	11/9/99
TW10XLOQ1	11/9/99	11/9/99
TW10XLOQ2	11/9/99	11/9/99
TW10XLOQ3	11/9/99	11/10/99
TW10XLOQ4	11/9/99	11/10/99
TW10XLOQ5	11/9/99	11/10/99